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Sr Resin Shelf-Life

Madeleine Eddy, PhD.

65th RRMCM, Atlanta, GA

1 November 2022

Déjà vu?

Eichrom Resin Shelf-life

Daniel McAlister, Jill Bryant,
Sarah Tejchma, Ed Rush

Radiobioassay & Radiochemical Measurements Conference



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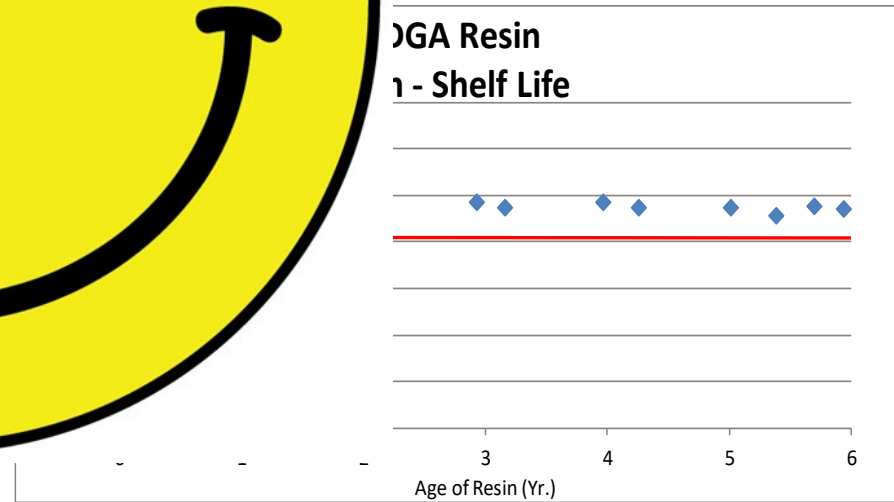
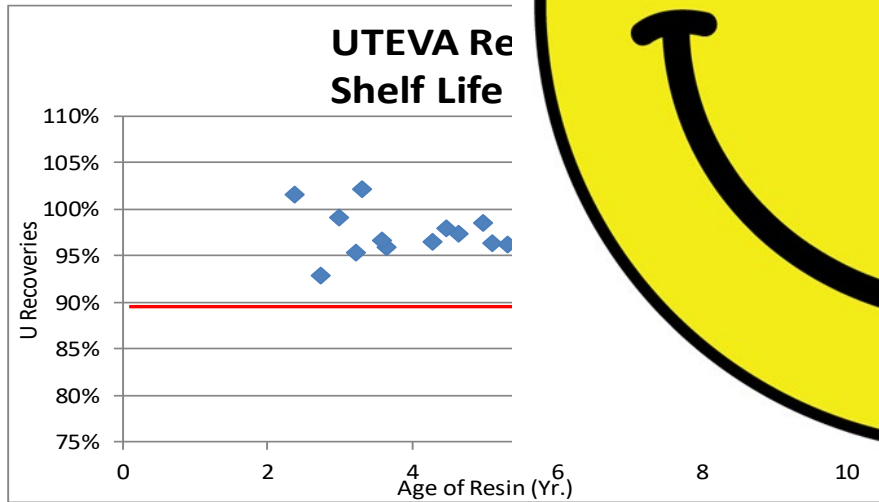
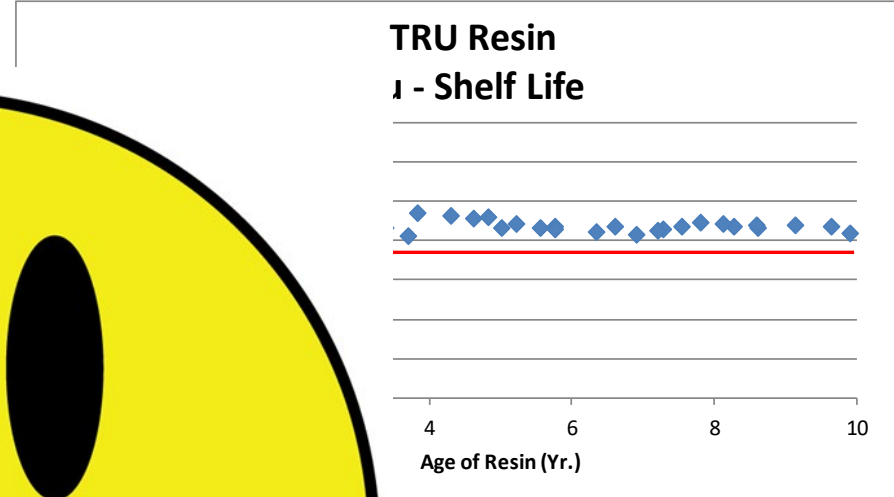
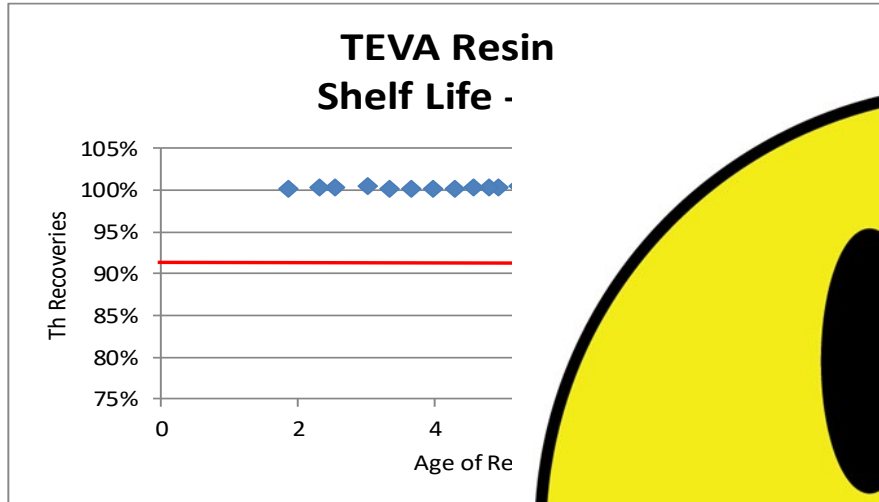


RRMC 2014
Knoxville, Tennessee USA
October 27 - October 31, 2014

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2014 Shelf-Life Study



Sr Resin Shelf-Life QC

- All lots of bulk Sr resin are analyzed every six months for two years after production
- QC procedure represents conditions Sr resin is commonly used under
- For several years (2013-2017) no failures were observed
- To date, **NO** failures observed within the first year
- Recent cluster of failures observed sporadically for 2018-2020 lots
- *But what about columns and cartridges???*

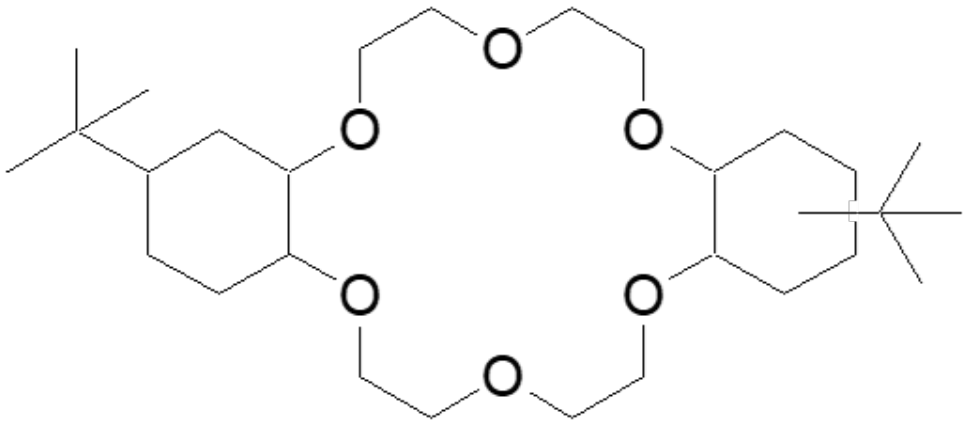
Lot	6 mo.	12 mo.	18 mo.	24 mo.
17A	Green	Green	Green	Green
17B	Green	Green	Green	Green
17D	Green	Green	Green	Green
17F	Green	Green	Green	Green
17G	Green	Green	Green	Green
17H	Green	Green	Green	Green
18A	Green	Green	Green	Red
18C	Green	Green	Green	Green
18D	Green	Green	Green	Green
18E	Green	Green	Green	Green
18G	Green	Green	Green	Red
18H	Green	Green	Green	Green
19B	Green	Green	Green	Red
19C	Green	Green	Green	Green
19E	Green	Green	Green	Red
19G	Green	Green	Green	Red
19I	Green	Green	Green	Green
19L	Green	Green	Green	Green
20B	Green	Green	Red	Red
20E	Green	Green	Red	Red
20F	Green	Green	Green	Green
20H	Green	Green	Green	Green



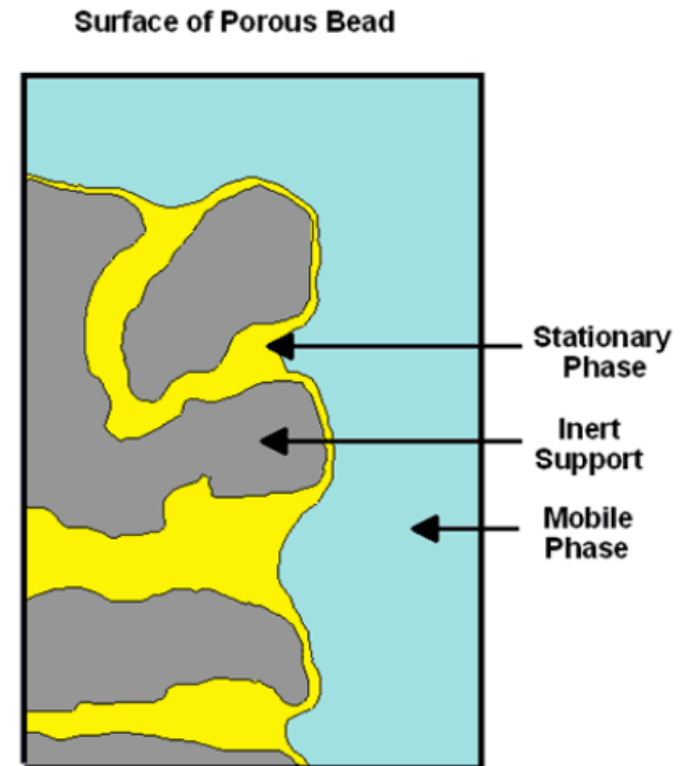
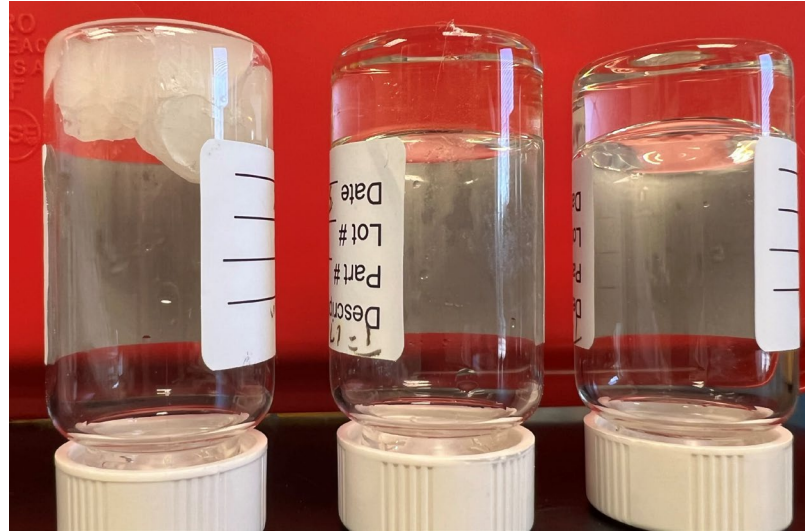
What is Sr resin?

Inert polymeric support impregnated with “crown” extractant and octanol diluent

- Sr resin has a high affinity for Sr(II) and Pb(II)
- Octanol is necessary to aid in phase transfer



4,4'(5')-di(t-butylcyclohexano)18-crown-6



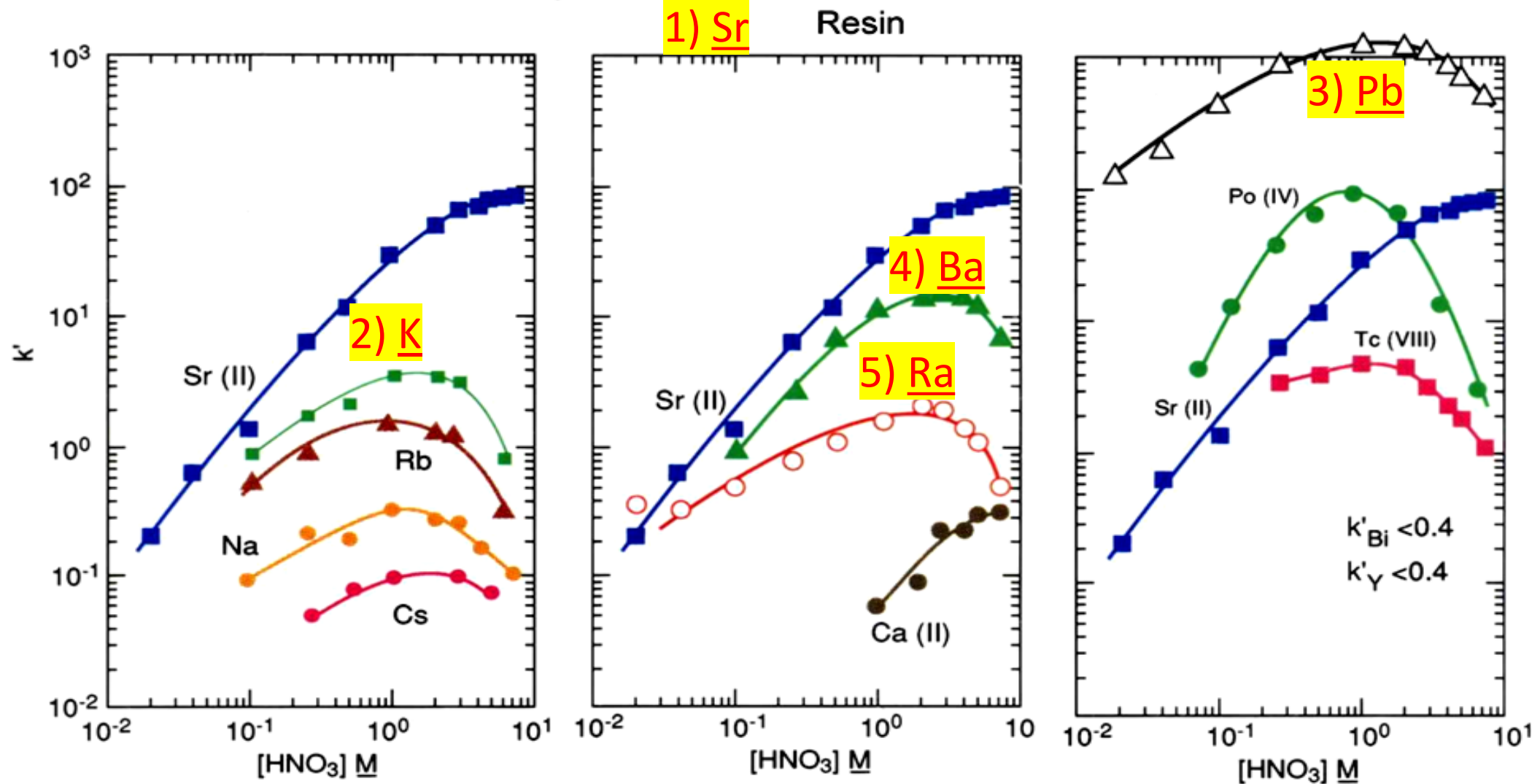
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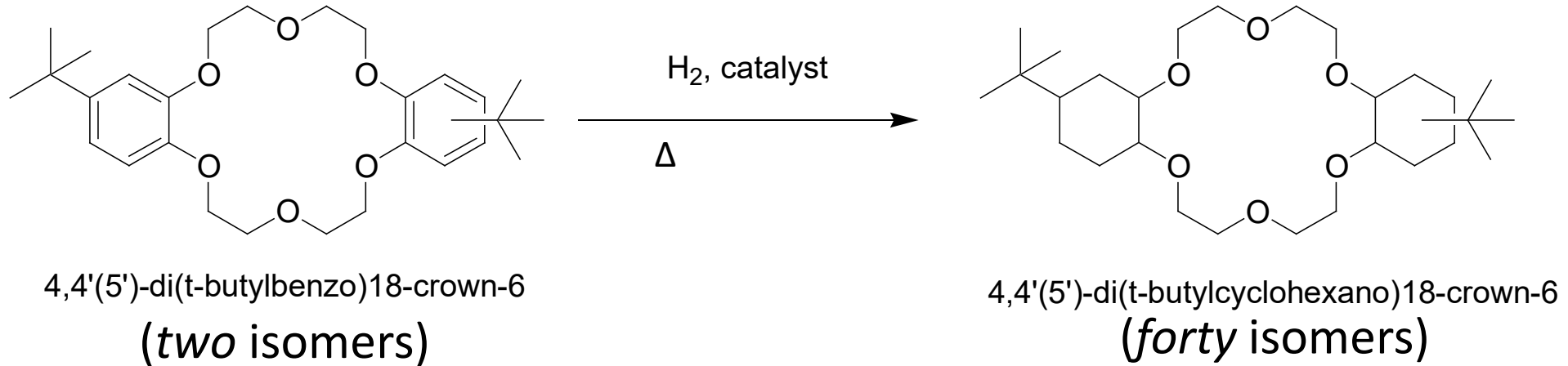
What is Sr resin?

Eichrom T-Shirt Game
Day 1

Acid dependency of k' for various ions at 23-25°C.









Sr Resin – Crown Production

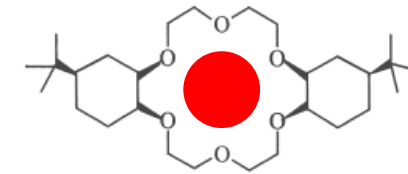


- Crown production on 200-600 g scale
- Each lot of SR resin is a unique crown blend
- Multiple lots of Sr resin may use the same support and/or octanol

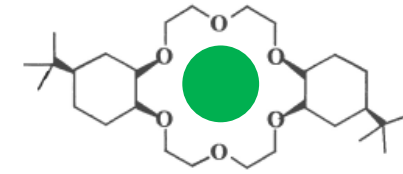
Crown Isomers

Table 1. Predicted D_{Sr} at 0.1 M ligand concentration for the n-octanol:1 M nitric acid system.

4(z),4'(z) <i>cis-syn-cis</i> 	9.50±1.6	4(z),5'(z) <i>cis-syn-cis</i>	3.00±0.5
4(z),4'(e) <i>cis-syn-cis</i> 	5.79±1.0	4(z),5'(z) <i>cis-anti-cis</i>	2.77±0.5
4(e),4'(e) <i>cis-syn-cis</i> 	4.93±0.8	4(e),5'(e) <i>cis-anti-cis</i>	2.52±0.4
4(z),5'(e) <i>cis-syn-cis</i> 	4.57±0.8	4(e),4'(e) <i>cis-anti-cis</i>	0.052±0.009
4(e),5'(e) <i>cis-syn-cis</i> 	3.67±0.6	4(z),4'(z) <i>cis-anti-cis</i>	0.064±0.010
4(z),4'(e) <i>cis-anti-cis</i> 	3.21±0.5	4(z),5'(e) <i>cis-anti-cis</i>	0.022±0.004



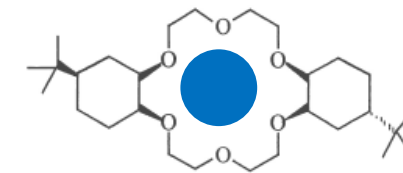
4(z),4'(z)-cis-syn-cis



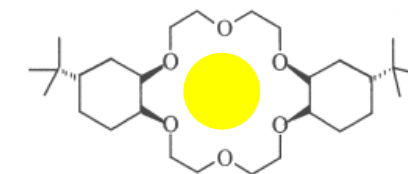
4(z),5'(z)-cis-syn-cis



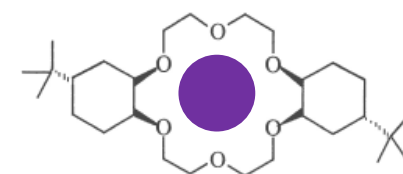
4(z),4'(e)-cis-syn-cis



4(z),5'(e)-cis-syn-cis



4(e),4'(e)-cis-syn-cis



4(e),5'(e)-cis-syn-cis

Isomer mixture will determine
Sr resin extraction power...
but does it impact shelf-life?

Shelf-Life Experiment

Goal: Correlate resin failures to physical parameters ex: crown isomer mix, octanol loss, or storage methods

Plan: Perform standard shelf-life QC test on all available lots of pre-packed columns and cartridges of Sr Resin

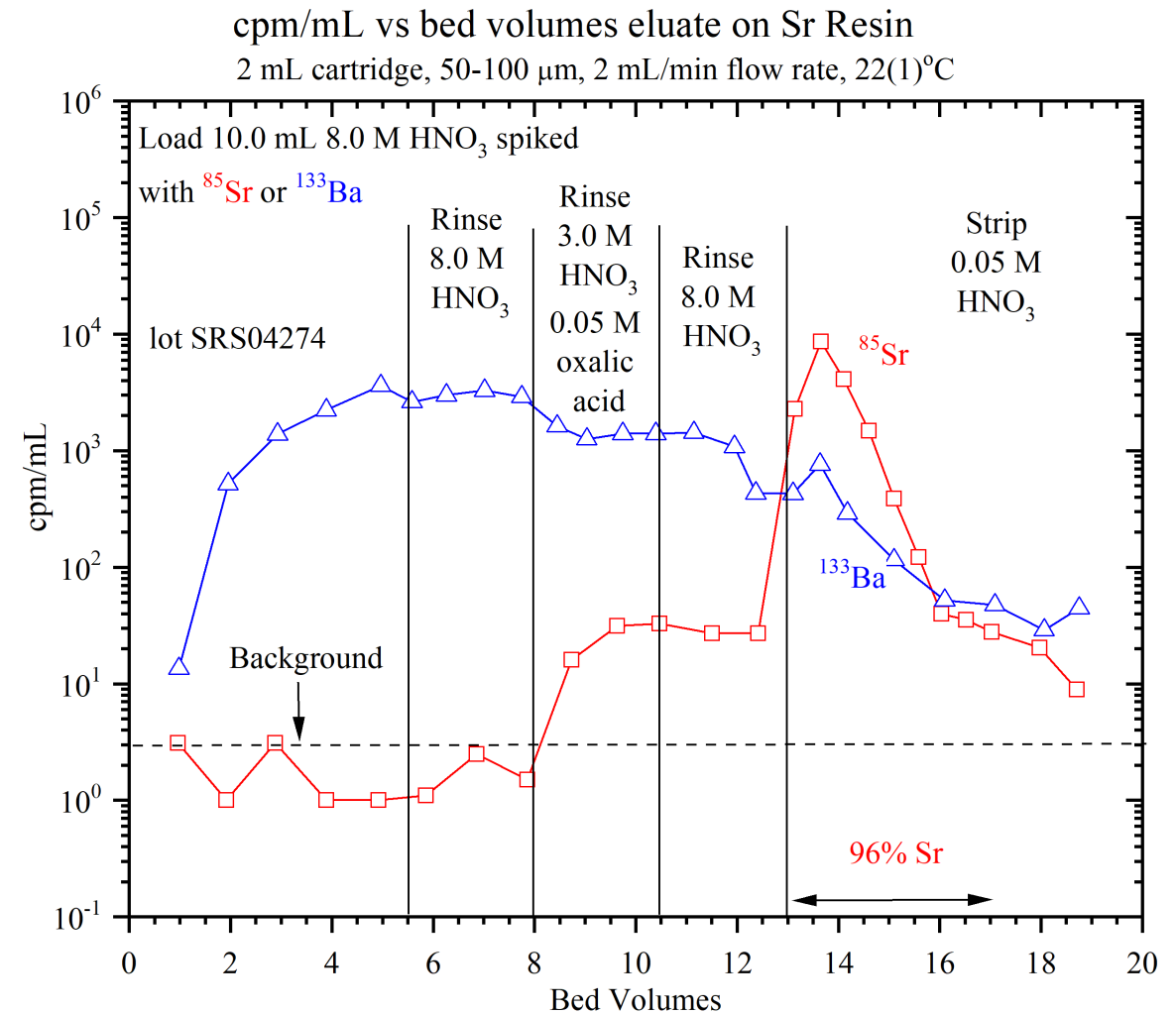
Measure:

% Sr in strip

If > 85% then pass

% Ba in strip

If < 0.75% then pass



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Shelf-Life Experiment: Columns

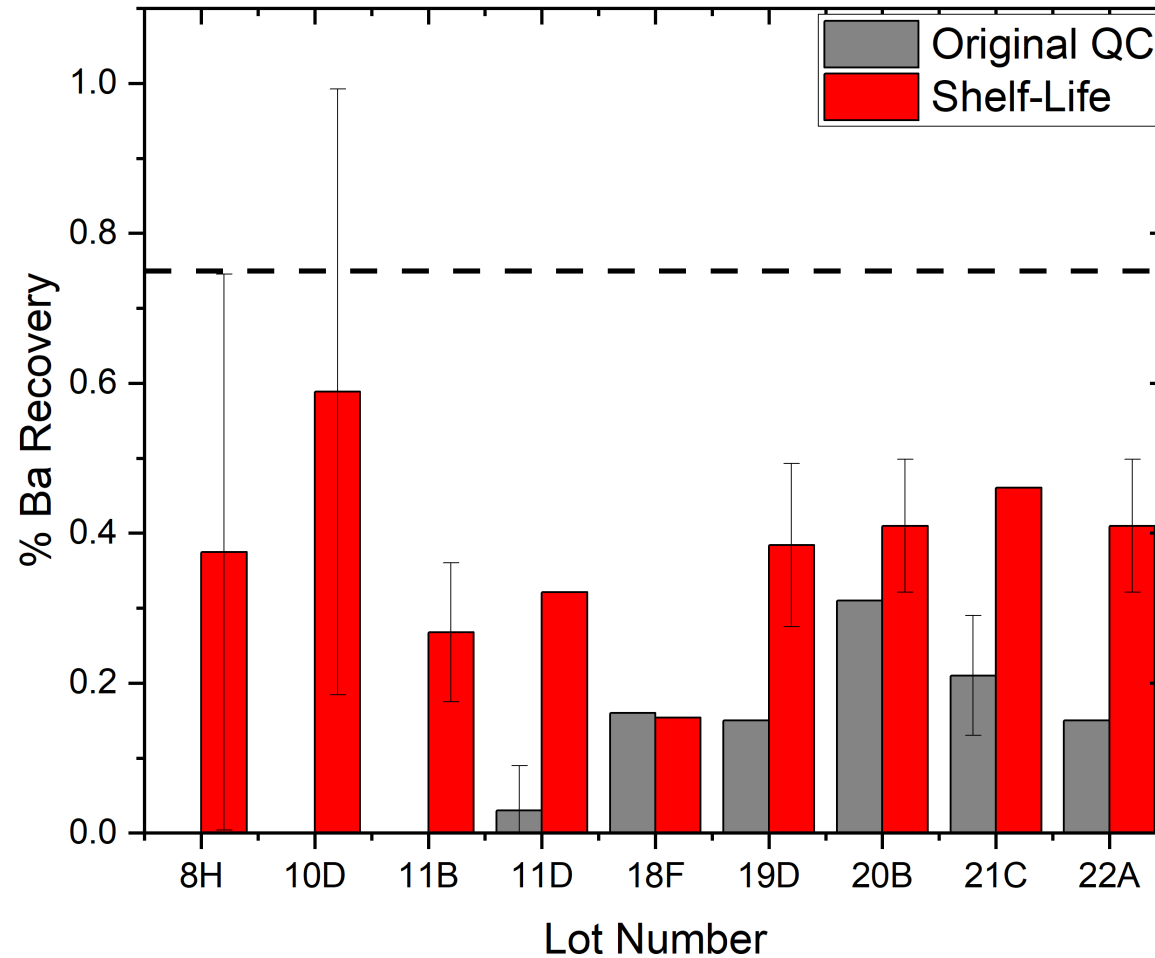
Columns:

- Nine total lots
- 2008-2022
- 100-150 μm particle size
- Manually packed in 2 mL columns
- Stored in 0.05 M HNO_3
- Gravity flow elution



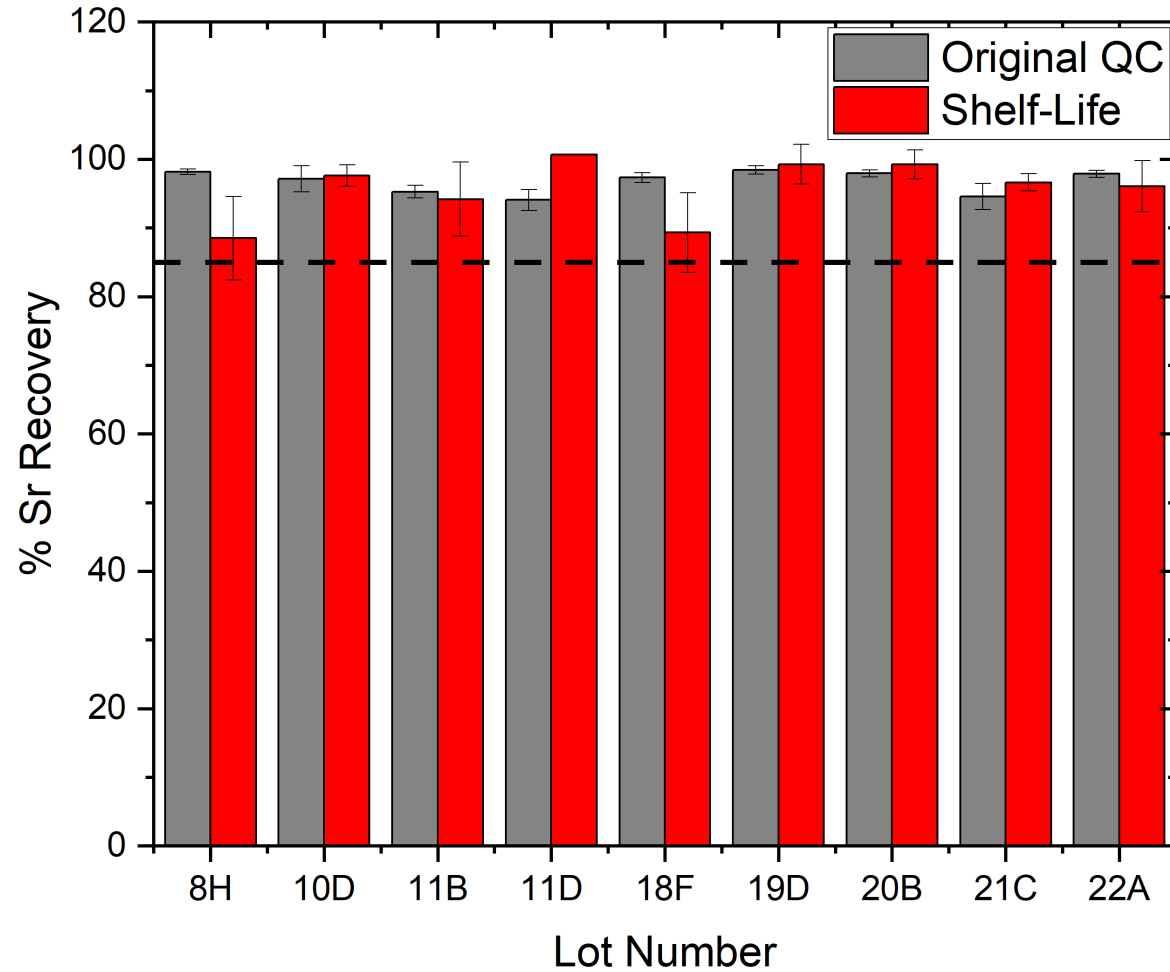
Columns – Barium Contamination in Strip Fraction

% Ba Recovery in Strip Fraction for 2 mL Columns



Columns – Strontium Recovery

% Sr Recovery in Strip Fraction for 2 mL Columns



Shelf-Life Experiment: Cartridges

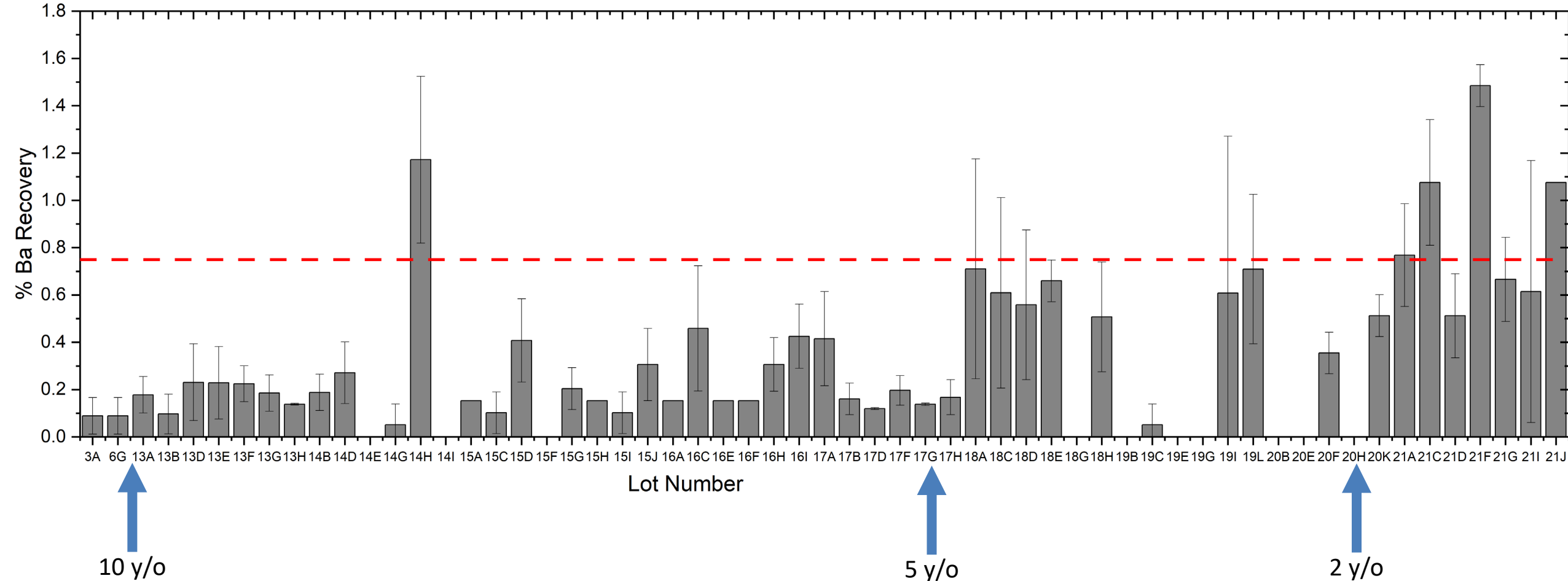
Cartridges:

- 59 total lots
- Multiple lots from 2013-2021
- One lot from 2003 and 2006
- 50-100 μm particle size
- Automatically packed in 2 mL cartridges
- Stored dry
- Vacuum box assisted elution at approx. 1 mL/min



Cartridges – Barium Contamination

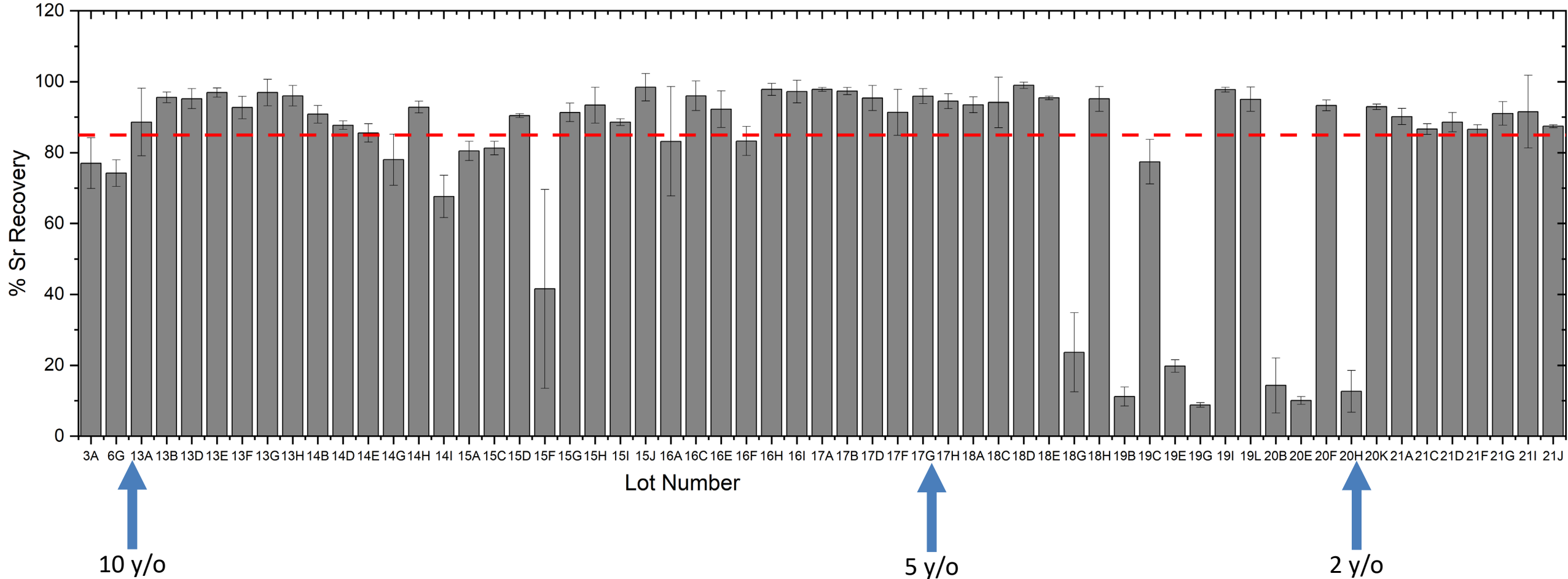
% Ba Recovery in Strip Fraction for 2 mL Cartridges



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Cartridges – Strontium Recovery

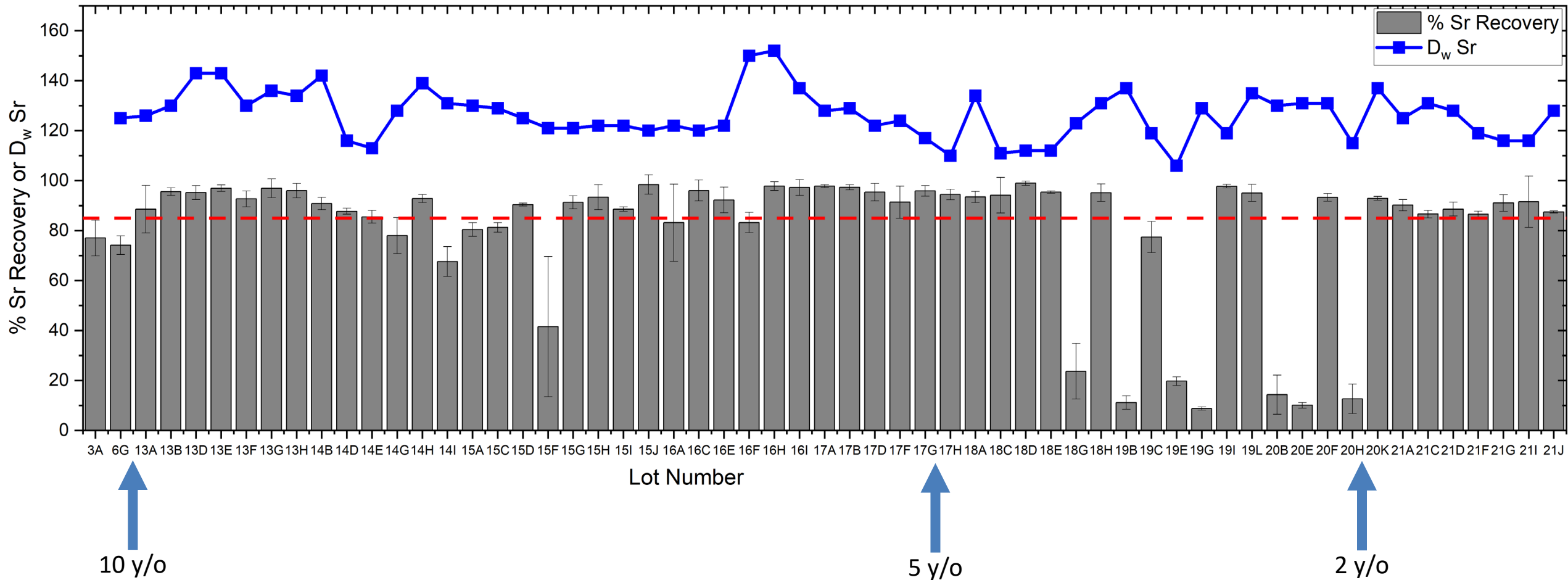
% Sr Recovery in Strip Fraction for 2 mL Cartridges



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Cartridges – Sr Recovery vs. Uptake

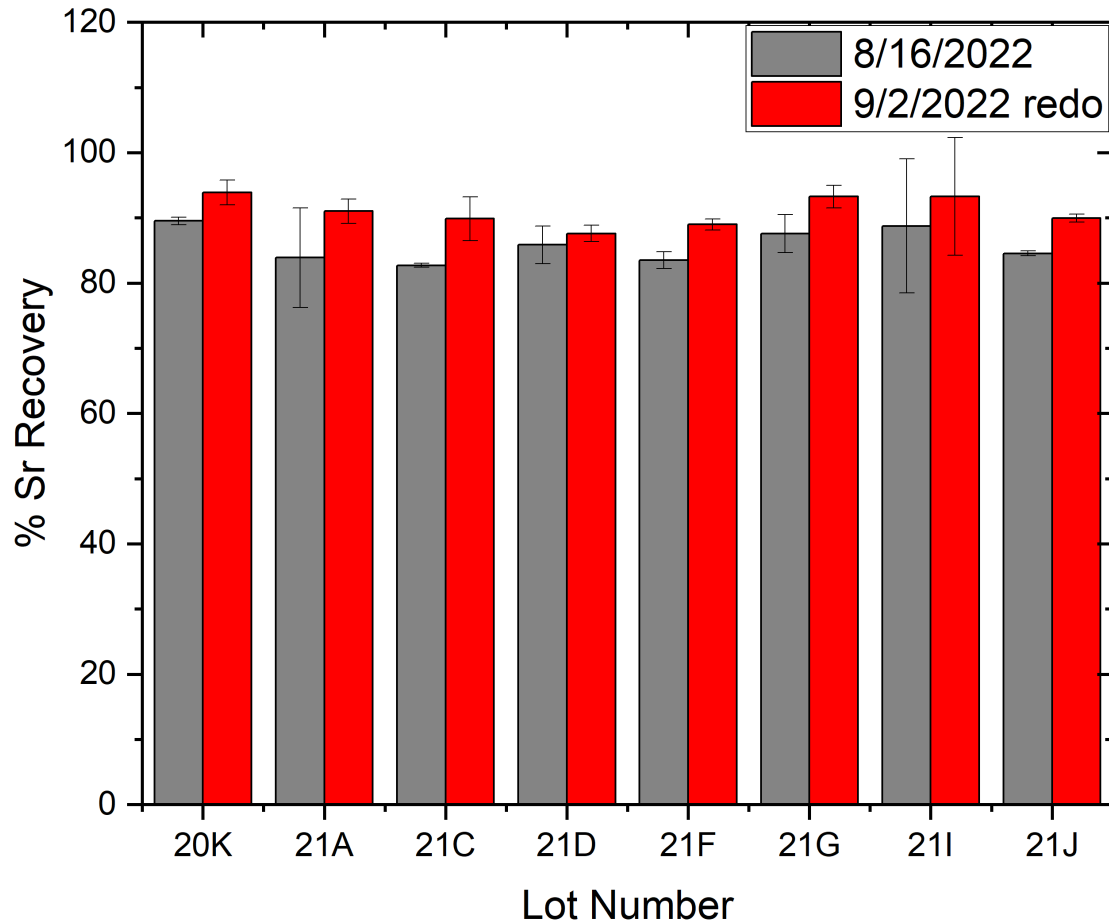
Sr Recovery vs. Uptake for S-grade Sr Resin



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Sample reruns:

% Sr Recovery in Strip Fraction for 2 mL Cartridges

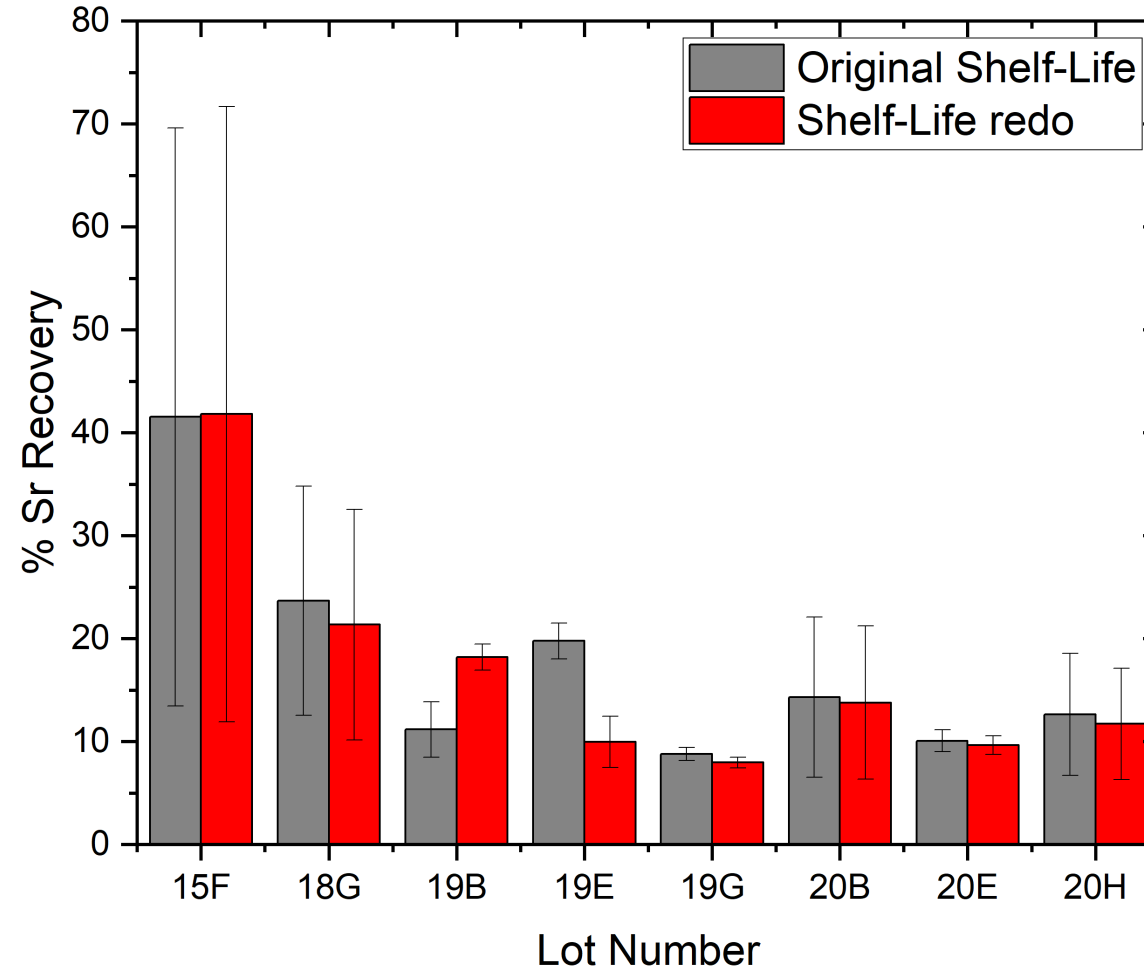


- Samples stored in 0.05 M HNO₃
- Marginal improvement in rerun
- Could storage in acid help to better prepare the resin for extraction?
- **Could rerunning lots that failed after storage in 0.05 M HNO₃ lead to a passing performance?**

*NOTE: reusing cartridges could lead to inaccurate results due to residual metals on resin, but in this case the mass balance for the redo did not exceed 100%

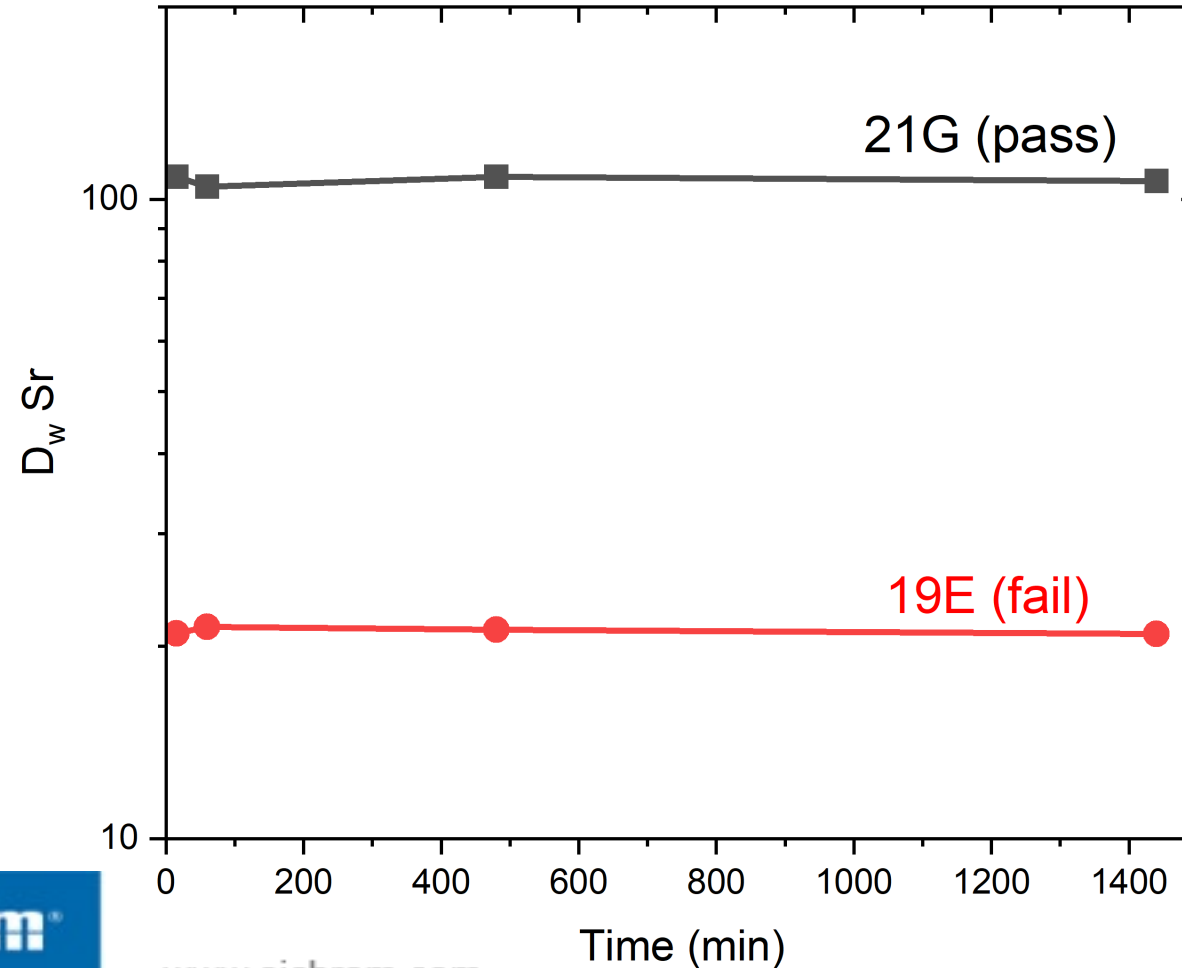
Failed lots rerun

% Sr Recovery in Strip Fraction for 2 mL Cartridges



Do failing lots have a kinetic issue?

Kinetics of Sr Uptake on S-grade Sr Resin
0.25 g resin, 60 ppm Sr, 3 M HNO₃



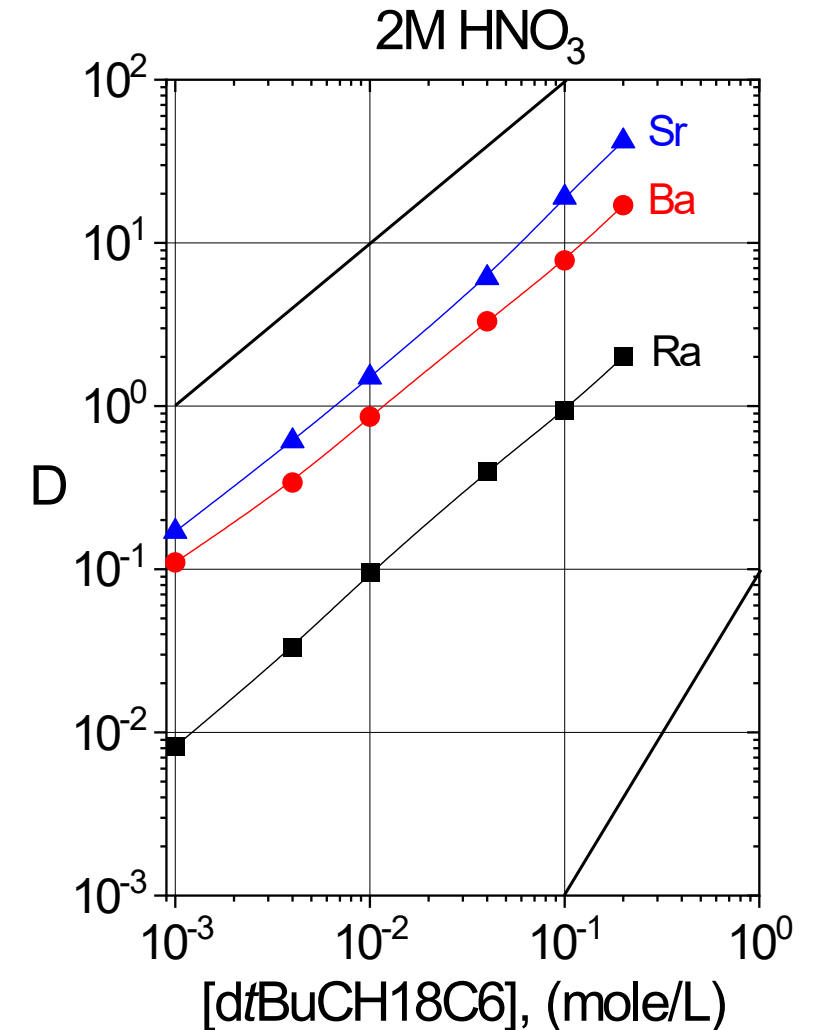
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Do failing lots have greater octanol loss?

Procedure for testing mass loss:

- Empty contents of 3 cartridges into 5 mL column
- Strip crown/octanol using 125 mL 100% ethanol
- Dry resin and record solid mass
- Dry crown/octanol *gently* and record liquid mass
- Dissolve crown/octanol in octanol
 - Measure D-Sr from 2 M HNO₃
 - Use calibration curve to calculate [crown]



Resin Mass Loss

Lot	Pass/Fail	Solid mass	Liquid mass	Crown in liquid	%mass loss
19B	Fail	1.21	0.49	-	12.7
19E	Fail	1.27	0.48	-	10.6
19G	Fail	1.25	0.52	0.13	9.3
19C	Borderline	1.17	0.57	0.28	11.1
13F	Pass	1.20	0.47	0.34	14.8
21G	Pass	1.18	0.65	-	6.4
22A	Pass	1.18	0.64	0.29	6.5
theoretical	-	1.20	0.75	0.40	-

Conclusions

- Sr resin failures are a physical issue
- **Sr resin has at least a one-year shelf life**
- Still some sporadic lot failures after 1.5-2 years
- All lots showed some octanol loss
- Failed lots had more difficult-to-remove crown
- Storage in dilute acid appears to improve shelf-life



Future Work

- Continue monitoring
- Expand studies to Pb resin
- Evaluate failed lots Pb uptake
- Investigate alternate conditions for production to reduce octanol loss
- Add more QC testing
 - Crown/octanol stripping?
 - Dilute acid storage?



Questions?



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