Pb-Resin: New Approaches, Challenges, and Troubleshooting

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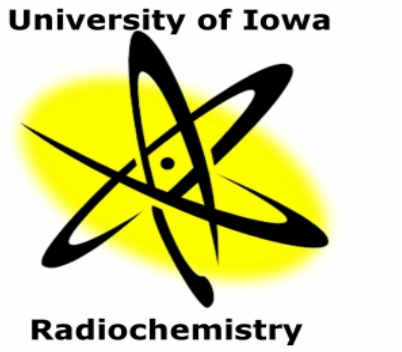

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Older Methods with Pb-Resin

- Two similar methods
  - ASTM D7538
  - Eichrom Pb-210 and Pb-210 in Water OTW01 (Pre 5/1/2014)
- Iron Hydroxide preconcentration
- Load on Pb-Resin or Sr-Resin in 1 M HNO_3
- Strip Pb with 20 mL H_2O
- H_2SO_4 precip
- Gas Flow

Rationale for ASTM D7538

- Highly respected organization
- Highly regarded and widely used methods
- Validated by interlab comparisons
- Very similar to Eichrom method

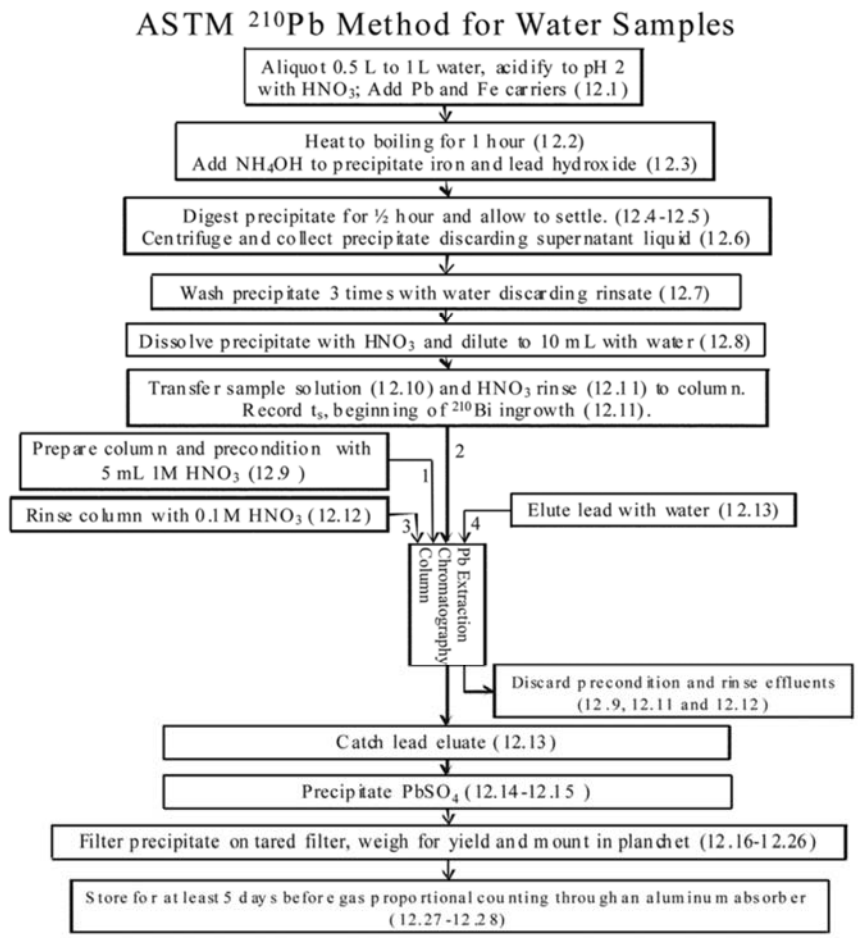


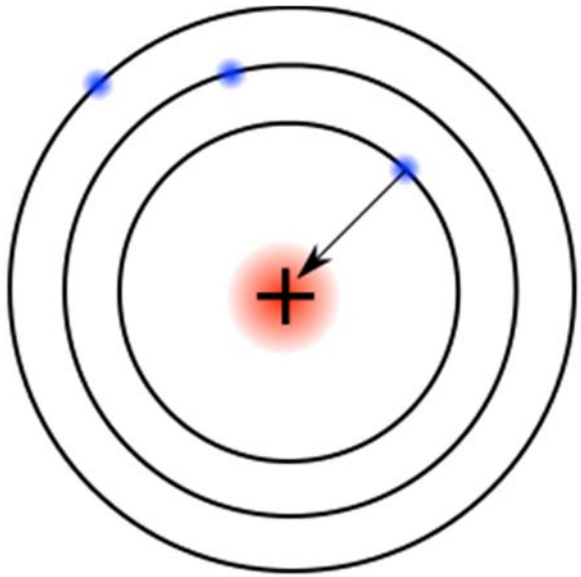
FIG. 1 ^{210}Pb Method for Water Samples

Problems with ^{210}Pb Methods

- Recovery based on mass
  - Unknown quantities of stable Pb in samples
  - Drifting analytical balances
  - Must have exceptionally pure separations
  - Affected by humidity
- Stable Pb carrier
  - How much background is in Pb carrier?
  - Requires more resin
  - Requires H_2SO_4

Introducing, Pb-203

- Pb-203
  - Cyclotron produced
  - EC, screaming gammas
  - $t_{1/2}$ = 52 hours
    - "massless" tracer



- **Can measure on the column!**
- Do not need to consider endogenous Pb
- Drifting analytical balances no longer a problem
- May be able to use smaller quantities of resin per sample?
  - more samples

Experimental Conditions

- Fracking flowback fluid, river water, and tap water were utilized
- Each matrix was analyzed under the following conditions: Pb-203 tracer/stable Pb carrier, Pb-203 tracer only, and stable Pb carrier only
- Matrix/condition pairs were analyzed in triplicate in accordance with ASTM D7538

Pb-203 Tracer Recovery

Matrix	Condition
Fracking Flowback Fluid	Pb-203 Tracer Only
	Stable Lead Carrier & Pb-203 Tracer
River Water	Pb-203 Tracer Only
	Stable Lead Carrier & Pb-203 Tracer
Tap Water	Pb-203 Tracer Only
	Stable Lead Carrier & Pb-203 Tracer

- Three replicates of each matrix/condition group were analyzed.

Stable Lead Carrier Recovery

Matrix	Condition
Fracking Flowback Fluid	Stable Lead Carrier Only
	Stable Lead Carrier & Pb-203 Tracer
River Water	Stable Lead Carrier Only
	Stable Lead Carrier & Pb-203 Tracer
Tap Water	Stable Lead Carrier Only
	Stable Lead Carrier & Pb-203 Tracer

- Three replicates of each matrix/condition group were analyzed.

Stable Lead Carrier Recovery

Matrix	Condition
Fracking Flowback Fluid	Stable Lead Carrier & Pb-203 Tracer
River Water	Stable Lead Carrier & Pb-203 Tracer
Tap Water	Stable Lead Carrier & Pb-203 Tracer

- Three replicates of each matrix/condition group were analyzed.

Recovery Discussion

- Recoveries vary wildly across samples
  - Large standard deviations
  - Likely due to high lead concentrations in matrix
- The Pb-203 was measureable on the column post elution step
  - Lead was loaded and retained on the resin, but not eluted

Ulowa Recovery

- Soil Recoveries
  - 81 samples
  - Average: 75% ± 9 %
  - Range: 49% - 105%
- Note, these were prepared by Eichrom Pb in Soil Method

- Water Recoveries
  - 8 samples
  - Average: 76% ± 10 %
  - Range: 62% - 91%
- Note, these values were prepared by ASTM D7538 on flowback/produced fluids from the Niobrara formation

Hypotheses About Recovery Issues

- pH seems a likely cause of the variation

Water Source	pH
UI Chemistry Lab	7
SHL at UI	5

  - UI Chemistry Lab uses house deionized water
  - SHL uses house deionized water that is then run through a Millipore purification system
  - Incomplete rinsing of loading solutions could lead to lowered pH in an un-buffered system
- Stable lead carrier could actually be necessary for effective elution
- Same operator, minimize sources of variation

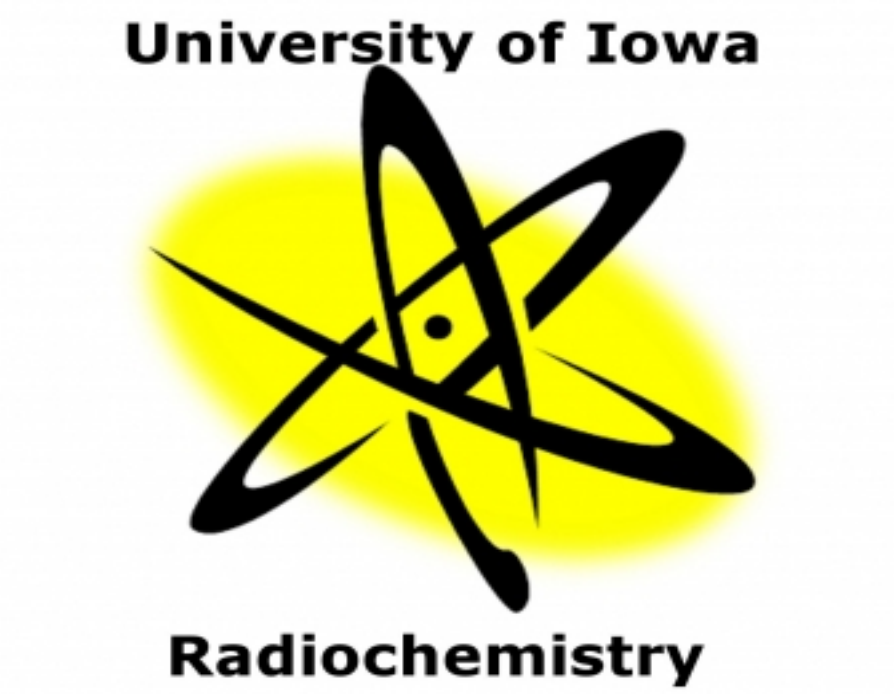
Immediate Plans

- Repeat experiment using buffered eluent solution recommended by current Eichrom method
- We would like to open it up to suggestions on method and experimental improvements

Future Directions

- Revision of ASTM method D7538
- Place a caution on Eichrom method for those that want to use water
- Possible develop single sample Pb-Po prep for alpha spec by SR Resin?

Thanks! Questions?



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