

Total Alpha Emitting Radium with Filters

By

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Planchettes versus Filters

- EPA Method 903, SM 7500 Ra B, ASTM D2460
- Quick method overview
- Discuss the differences
- Present single operator test
- Present multi-lab data
- Present added benefits
- Summary of findings

Quick Method Summary

- Take a large water sample usually 1 liter
- Perform a gross precipitation $\text{PbSO}_4\text{-BaSO}_4$
- Re-dissolve with EDTA
- Perform a second precipitation of only Barium and Radium using acetic acid (actinides, lead, and Po are not precipitated)
- Place on Planchettes (Filters) for Alpha counting.

Planchettes



Filters



Filter Close-Up



Precipitate on Filters



A Single Operator Test

Ra 226 Spiked at 5.18 pCi/L

- Planchette%Recoveries

95.22

82.95

96.61

98.07

97.69

92.27

97.32

- Average 94.30

- RSTDEV 5.39

- Filter % Recoveries

90.16

96.50

94.64

101.43

96.41

99.84

103.32

- Average 97.47

- RSTDEV 4.45

Study Design

- Performed this with input from ASTM D19.04
- 3 Commercial Labs
- Receive 2 samples of spiked drinking water
- Run each sample in triplicate as per method with planchettes
- Run each sample in triplicate as per method with filters
- Report in triplicate

Multi-Lab Study

Ra 226 spiked at 32.7 pCi/L

- Planchette % Recoveries

Lab 1 Rep1	99.1
Lab 1 Rep2	96.9
Lab 1 Rep3	100.0
Lab 2 Rep1	97.9
Lab 2 Rep2	99.9
Lab 2 Rep3	98.1
Lab 3 Rep1	87.5
Lab 3 Rep2	86.6
Lab 3 Rep3	108.1

- Average
- RSTDEV

97.1
6.56

- Filter % Recoveries

Lab 1 Rep1	92.5
Lab 1 Rep2	96.6
Lab 1 Rep3	95.6
Lab 2 Rep1	100.2
Lab 2 Rep2	89.9
Lab 2 Rep3	95.3
Lab 3 Rep1	104.0
Lab 3 Rep2	105.9
Lab 3 Rep3	93.8

- Average
 - RSTDEV
- 97.1
5.31

Multi-Lab Study

Ra 226 spiked at 3.57 pCi/L

- Planchette % Recoveries

Lab 1 Rep1	104.3
Lab 1 Rep2	82.4
Lab 1 Rep3	76.5
Lab 2 Rep1	92.8
Lab 2 Rep2	92.0
Lab 2 Rep3	99.8
Lab 3 Rep1	102.0
Lab 3 Rep2	105.7
Lab 3 Rep3	93.4

- Average 94.3
- RSTDEV 9.91

- Filter % Recoveries

Lab 1 Rep1	99.2
Lab 1 Rep2	93.6
Lab 1 Rep3	106.0
Lab 2 Rep1	102.3
Lab 2 Rep2	99.0
Lab 2 Rep3	109.1
Lab 3 Rep1	114.1
Lab 3 Rep2	99.0
Lab 3 Rep3	108.5

- Average 103.4
- RSTDEV 6.44

Multi-Lab Study

BaSO₄ Recoveries/Chemical Yields

- Planchettes Lab 1

89.07%

89.62%

85.25%

85.25%

92.35%

90.71%

91.26%

83.06%

- Average 88.3%

- RSTDEV 3.4

- Filters Lab 1

97.81%

98.91%

98.91%

100.55%

101.99%

96.72%

99.45%

95.63%

- Average 98.8%

- RSTDEV 2.0

Multi-Lab Study

BaSO₄ Recoveries/Chemical Yields

- Planchettes Lab 2

88.7%
78.0%
83.6%
100.0%
90.4%
78.0%
88.1%
88.1 %

- Average 86.9%
- RSTDEV 7.2

- Filters Lab 2

88.1%
94.4%
94.4%
93.2%
96.6%
97.5%
100.0%
97.7%

- Average 95.2%
- STD 3.6

Multi-Lab Study

BaSO₄ Recoveries/Chemical Yields

- Planchettes Lab 3

100%

85%

59%

95%

73%

73%

56%

94%

- Average 79.4%

- RSTDEV 16.7

- Filters Lab 3

89%

88%

96%

81%

100%

82%

82%

72%

- Average 86.3%

- RSTDEV 9.0

Study Instructions

- Reprecipitate barium sulfate (BaSO_4) by dropwise addition of acetic acid then add 3 drops more. Record the time. Set up a clean appropriate vacuum filter apparatus with pre-weighed Eichrom[®] Resolve[®] PTFE Filter 47 mm Diameter 3um PTFE laminate, part number RP-50-47PTFE3. Turn on vacuum and add 2-5 mL ethyl alcohol to filter to open pores in Eichrom[®] filters. Vortex sample gently, and empty into apparatus. Add about 2 mL de-ionized water into vial, vortex again and add to filter. Rinse vial sides, and then bottom about 3 times with de-ionized water to make sure all precipitate has been removed. Rinse the filter apparatus 3 times with deionized water, add about 5 mL ethyl alcohol to finish rinsing and assist drying the filter, then quickly remove filter from vacuum assembly.
- Dry filter at 45C, cool to room temperature in dessicator, and weigh the precipitate to determine the chemical yield.
- Count the filter collected precipitate in an appropriate alpha-particle counter, recording the time. Reserve the filters for additional measurements, if desired.
- Measure background count rate of the detector by counting a clean unused filter for at least as long as the precipitate was counted. Note: Lot testing background of filters is recommended.

Summary of Findings

- 1) Very Similar Results – Recoveries Improved
- 2) Chemical yields improved significantly – this leads to slightly improved detection limits.
Combined average improvement of 9% also the improved standard deviation
- 3) Improved efficiencies factors due to improved geometry. On the order of 5% -15% improvement in signal response due to geometry.
- 4) Expectation is that MDL//MDC improved.

Thank You

- Questions?



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