Selective Extraction of Beryllium from Soil

Robert W. Smithwick III, Derek H. Bowman Jr. and Thomas J. Oatts

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Robert.Smithwickiii@cns.doe.gov
Analytical Chemistry Organization
Y-12 National Security Complex
Oak Ridge, TN

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- For soil samples, a method will be described that will more selectively extract and measure the anthropogenic Be.
- For surface-wipe and breathing-zone samples, the same method will apply but these samples contain much less soil.

Beryllium in the Soil at the Hanford Site

- The Hanford-WA site (586 mi²) has 290 inactive and 1200 active buildings. Some of these buildings may have native Be from sandy topsoil windblown into them or may have Be contamination from past activities.

Beryllium in the Soil at the Hanford Site

- The Hanford-WA site (586 mi^2) has 290 inactive and 1200 active buildings. Some of these buildings may have native Be from sandy topsoil windblown into them or may have Be contamination from past activities.
- A method[1,2] was described in 2015 by Scott Seydel and Charles Davis to distinguish between native Be and anthropogenic Be. Their method is based on the ratios of native Be to other native metals present in Hanford soil.

[1] S. A. Seydel and C. B. Davis, "The Hanford Metal Ratio Process Development", (2015). https://bhsc.llnl.gov/documents/10_15_BHSC.../Hanford_Metal_Ratio1_Seydel.pdf

[2] C. B. Davis and S. A. Seydel, "The Hanford Metal Ratio Study", (2015). https://bhsc.llnl.gov/documents/10_15_BHSC.../Hanford_Metal_Ratio2_Davis.pdf

Speciation of Be-Containing Particulate

- Four classes of Be compounds can be extracted[3] from Be-containing particulate in the following sequence:

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- Alternatively, the first three classes of Be compounds can be extracted collectively in a single step with ABF solution.

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Selective Extraction of Beryllium from Soil

- In past work[5], Be has been completely extracted from 0.50 g of soil with 50 mL of 3% ABF at 90 ºC for 40 hrs., (no mixing).

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- Sample extracts were filtered and analyzed by ICP-OES.
- After being extracted with 5% ABF, the soil was digested in mixed acid by a two-step microwave method[6]. The residual Be was measured in the final solution by ICP-OES.

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[6] M. A. Wilson et al., *Commun. Soil Sci. Plan.*, 37, 2006, 513-524.

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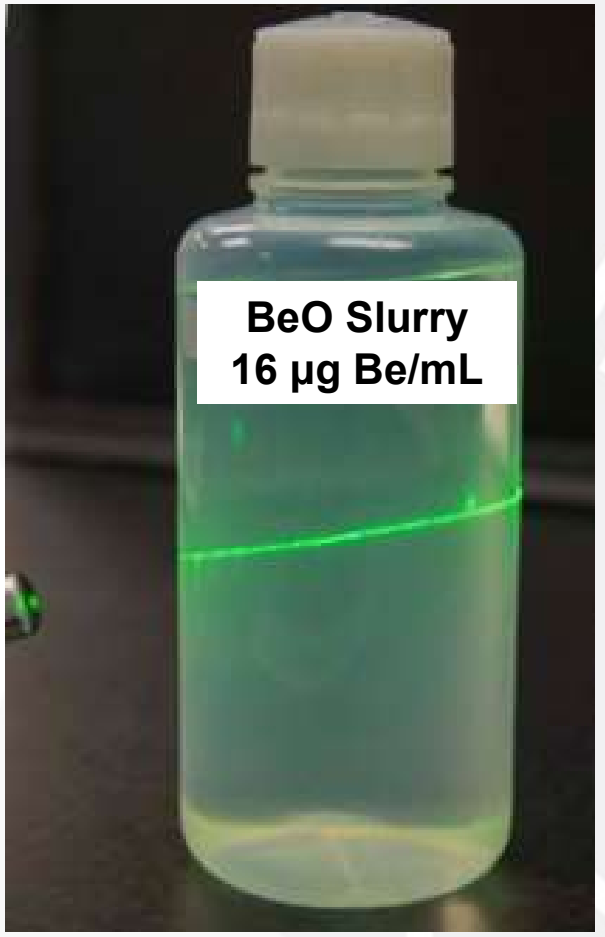

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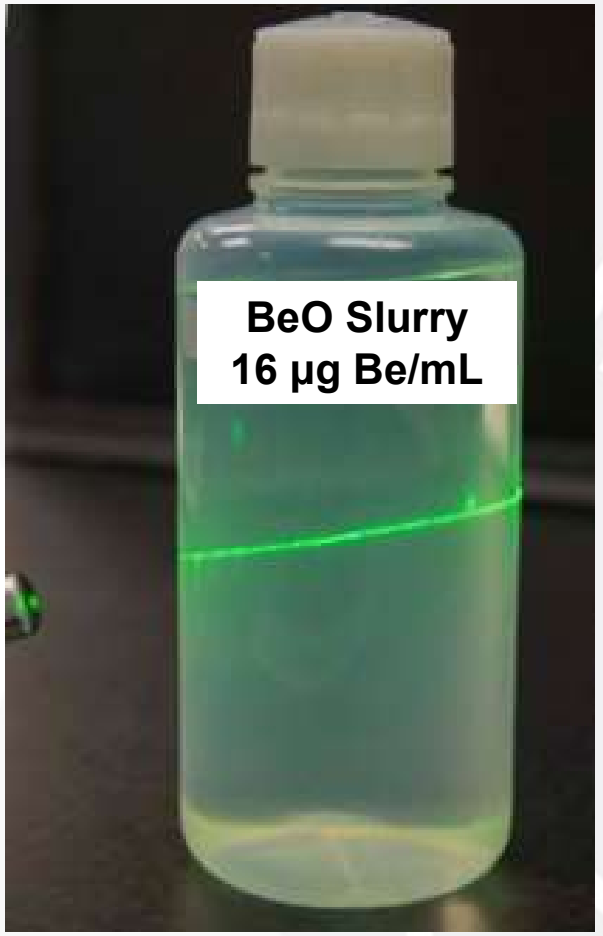

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- Through a clear Teflon bottle, scattered light from laser pointer shows a uniform dispersion of BeO particles in the slurry.
- Slurry was shaken immediately prior to pipetting a set of three 10-µL aliquots.
- For 36 aliquots, the mean ±2σ (95% interval), was 0.164 ±0.016 µg Be/aliquot.

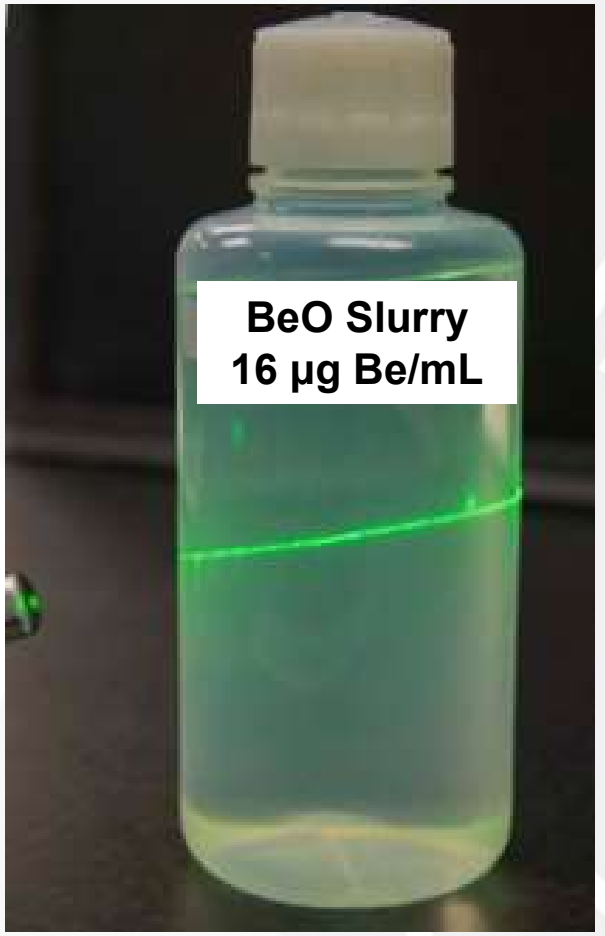

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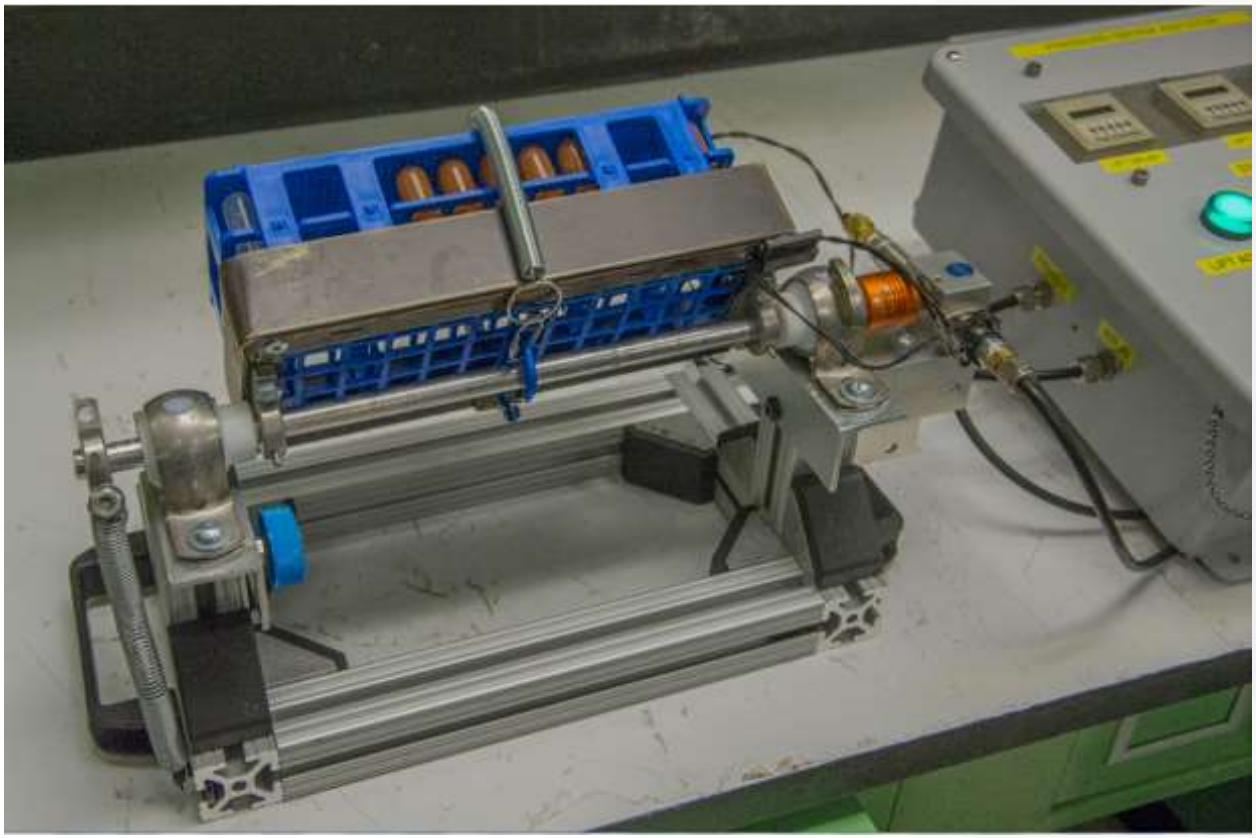
Table 1. Results for Vertical Tubes Without Sample Mixing.

60 °C	µg Be Extracted with ABF from 0.25 g Soil	µg Be Dissolved from BeO Spike	µg Be Extracted from Soil plus BeO Spike	µg Be Left in Soil after ABF Extraction
TN Red Dirt #1	0.088 (28%)	0.159	0.222 (90%)	0.230* (72%)
Hanford-1 Soil	0.063 (19%)	0.159	0.193 (87%)	0.265 (81%)
Hanford-2 Soil	0.080 (22%)	0.169	0.207 (83%)	0.288 (78%)
Hanford-3 Soil	0.058 (16%)	0.163	0.187 (85%)	0.309 (84%)
Averages	~0.07 µg (20%)	~0.16 µg	~0.20 µg (87%)	~0.28 µg (80%)

Pneumatic Rocker

- Tubes (10 mL) were rocked at 6 cycles/min. for 60 min. in an oven containing the preheated air-driven rocker at 60 ºC.



Electric Rocker

- Tubes (10 mL) were rocked at 40 cycles/min. for 60 min. in an oven containing the pre-heated Infinity Rocker Pro at 70ºC.

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Infinity Rocker Pro (12 VDC) is rated to 40 ºC.
Next Rocker Pro (24 VDC) is rated to 60 ºC.

Selective Extraction of Beryllium from Soil

Table 2. Results for Pneumatic Rocker.

60 °C	µg Be Extracted with ABF from 0.25 g Soil	µg Be Dissolved from BeO Spike	µg Be Extracted from Soil plus BeO Spike	µg Be Left in Soil after ABF Extraction
TN Red Dirt #1	0.160 (50%)	0.167	0.321 (98%)	0.158* (50%)
Hanford Soil #1	0.157 (49%)	0.166	0.315 (98%)	0.166 (51%)
Hanford Soil #2	0.174 (49%)	0.164	0.319 (94%)	0.178 (51%)
Hanford Soil #3	0.146 (42%)	0.150	0.307 (104%)	0.202 (58%)

Be with rocking	~0.16 µg (47%)	~0.16 µg	~0.31 µg (97%)	~0.18 µg (53%)
Without rocking	~0.07 µg (20%)	~0.16 µg	~0.20 µg (87%)	~0.28 µg (80%)

Selective Extraction of Beryllium from Soil

- Without mixing, ~0.2 µg Be was extracted from the BeO-spiked soils. This average is 87% of the total Be (for the soil and the BeO spike) dissolved in separate tubes. This low Be recovery was due to limited reaction of ABF within the unmixed mud or sand at the bottom of the tube.

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- With rocking, ~0.3 µg Be was extracted from the BeO-spiked soils. This average is 97% of the total Be (for the soil and the BeO spike) dissolved in separate tubes. This improvement with rocking is significant because BeO-spiked soil simulates BeO-contaminated soil.

Selective Extraction of Beryllium from Soil

Tables 2 & 3. Results for Pneumatic and Electric Rockers.

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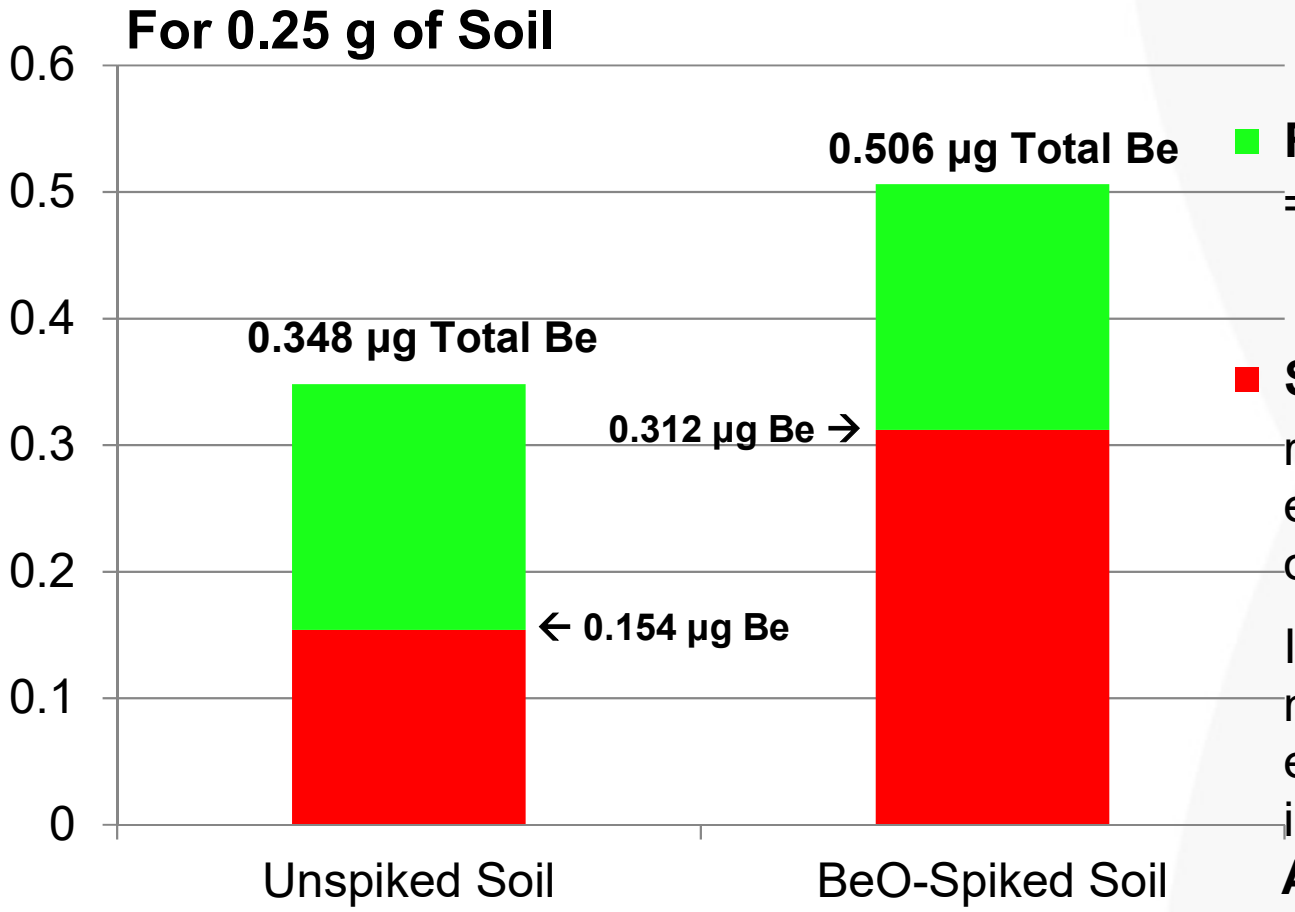
70 ºC				
TN Red Dirt #1	0.146 (49%)	0.169	0.295 (94%)	0.152 (51%)
Hanford Soil #1	0.136 (36%)	0.164	0.291 (97%)	0.237 (64%)
Hanford Soil #2	0.172 (43%)	0.168	0.333 (98%)	0.227 (57%)
Hanford Soil #3	0.144 (39%)	0.170	0.315 (100%)	0.232 (61%)
Averages	0.154 µg (44%)	0.164 µg	0.312 µg (98%)	0.194 µg (56%)

Selective Extraction of Beryllium from Soil

60;70 ºC	µg Be Extracted with ABF from 0.25 g Soil	µg Be Dissolved from BeO Spike	µg Be Extracted from Soil plus BeO Spike	µg Be Left in Soil after ABF Extraction
Averages	0.154 µg (44%)	0.164 µg	0.312 µg (98%)	0.194 µg (56%)



Residual native Be
= 0.194 µg Be

Selectively Extracted Be
requires one analysis and excludes the Residual Be on the basis of chemistry.

If the Residual Be is also measured (on the ABF-extracted soil), one can improve the estimate of **Anthropogenic Be...**

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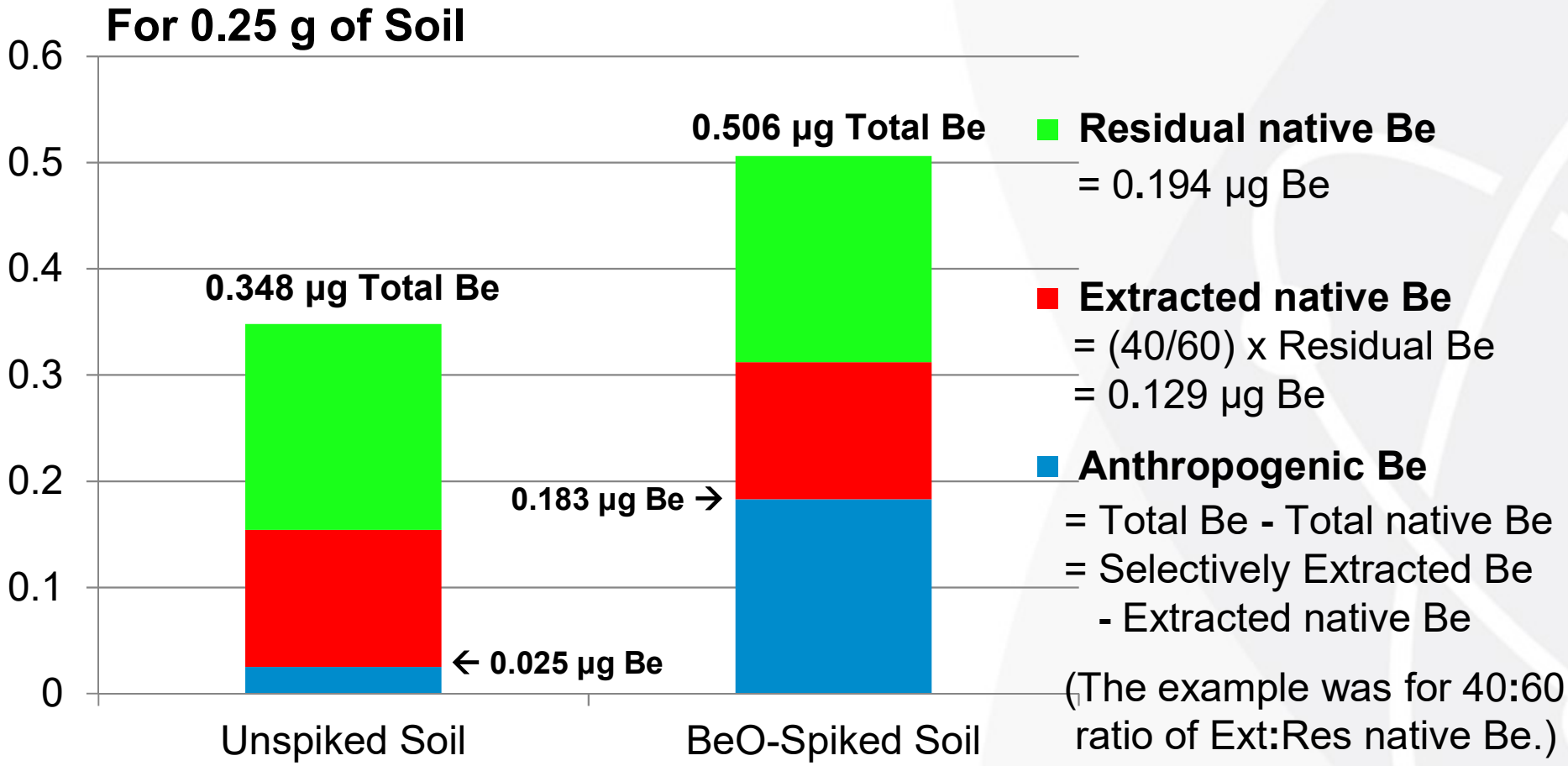

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- Hanford-area soils were used in the method development but different soils (i.e.TN Red Dirt) should give similar results.
- For cleanup activities, the proposed method more selectively extracts and measures the anthropogenic Be in the sample.
- For 0.25-g soil samples, Be was selectively extracted into 9 mL of 5% ABF solution at 70 ºC for 60 min. with rocking.

Summary -Cont'd

- For surface-wipe and breathing-zone samples that have low amounts of soil, a lower concentration of ABF could be used to extract Be. Also, sample rocking should not be necessary.

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- With surface smears, the selective-extraction method could be valuable if its use prevented buildings, containing native soil, from falsely being designated as Be contaminated.
- The decision to measure and accept selectively extracted Be, rather than total Be, will require oversight approval.

Acknowledgments

- Darrin Mann (Y-12) and Rikki Martz (Y-12) performed the MW digestions, and Craig Hanzelka (Y-12) built the pneumatic rocker and its controller.
- Johnafred Thomas (Y-12 Director of Quality Technical Services), Khris Olsen (retired from PNNL), and Kelley Paup-Lefferts (at USDA-NRCS, Pasco MLRA Soil Survey Office) each provided soil samples from outside of the Hanford DOE reservation.