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# Comparison of Beryllium Samples taken on Ghost Wipes™ versus the new (smaller) Beryllium Wipe™

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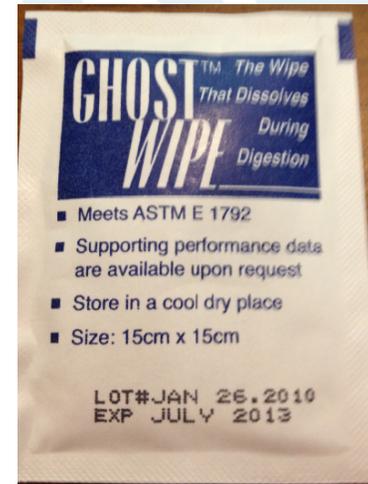
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# Disclaimers

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- Mention of commercial products in this presentation does not imply endorsement by the authors, SRNS, SRNL, SRS, or the U.S. Department of Energy (DOE).
- The speaker is an employee of a Department of Energy (DOE) contractor and is not a spokesperson for DOE itself.

# Face Off



# Trace Beryllium Determination in Polyvinyl Alcohol Wipes by Extraction and Fluorescence Detection: Interlaboratory Analysis<sup>1</sup>

## In 2011

- Involved the use of smaller 5cm X 5cm polyvinyl alcohol wipes
- Use of the smaller wipes (in addition to other parameters studied) was credited with improved sample recovery and lower standard deviation than previous studies with the larger wipes.
- It was noted in the study that these wipes were effective in taking 100cm<sup>2</sup> surface samples.

Table 1. ILS summary data from dry PVA wipes; number of laboratories n=8 unless otherwise indicated.

Certified spike level (µg Be)	Mean ± standard deviation (µg Be)	RSD (%)	Recovery (%)
<0.01 (media blank)	0.0025 ± 0.0030 (n=4)	120	---
0.030 ± 0.005	0.0291 ± 0.00461	15.8	97
0.16 ± 0.02	0.144 ± 0.0164	11.4	90
0.32 ± 0.02	0.295 ± 0.0169	5.73	92.2
1.8 ± 0.1	1.71 ± 0.0779	4.56	95
2.8 ± 0.1	2.76 ± 0.283	10.3	98.6
5.6 ± 0.1	5.29 ± 0.538	10.2	94.5

1. Ashley, K, Wise, TJ, Marlow, D, Agrawal, A, Cronin, JP, Adams, L, Ashley, E, and Lee, PA, (2011), Trace Beryllium Determination in Polyvinyl Alcohol Wipes by Extraction and Fluorescence Detection: Interlaboratory Analysis, *Anal. Methods*, 2011,3, 1906-1909

## Why Consider a “Beryllium Wipe™”?

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### **ASTM D7707-11 Standard Specification for Wipe Sampling Materials for Beryllium in Surface Dust was published in 2011**

#### **Standard Requirements for Wipes Included:**

- **Mean background beryllium content per wipe  $<0.0005 \mu\text{g}$**
- **Fully wet upon removal from package**
- **Moisture content: Coefficient of variation for a random sampling of the lot is no greater than 25% across individually packaged wipes and within and between multi-packaged wipes.**
- **Mean beryllium recoveries from wipes spiked with certified reference materials shall be 100+/-20% at the 95% confidence level**

# Why Consider a “Beryllium Wipe™”?, continued

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## Standard Requirements for Wipes, continued

- **Size of Wipes:**

- Before moistening, mean thickness between 0.005-0.05mm
- Before moistening, coefficient of variation in the mass of a lot  $\leq 10\%$
- Between 200cm<sup>2</sup>-400cm<sup>2</sup> with dimensions of 10-20 cm on each side

**OR**

- Between 16cm<sup>2</sup>-38cm<sup>2</sup> with dimensions of 4-8 cm on each side

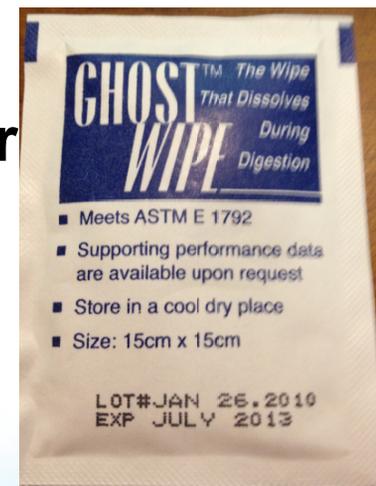
NOTE: Larger wipes are intended for use on larger areas (example 1000 cm<sup>2</sup>) and small on smaller surfaces (example 100cm<sup>2</sup>)

- **Sufficiently rugged and capable of wiping a surface of up to:**

- 2000 cm<sup>2</sup> (larger wipes)
- 500 cm<sup>2</sup> (smaller wipes)

## History

- Savannah River Site has used Ghost Wipes™ (Environmental Express) for over eight years to sample surfaces for beryllium.
- These wipes meet requirements specified in ASTM E1792 (Standard Specification for Wipe Sampling Materials for Lead in Surface Dust).
- Specifications for this standard are similar to those of D7707.
- There are some differences in the two standards.



# Differences between the Standards: A Side by Side Comparison

## ASTM E1792

- Lead background  $<1.0\mu\text{g}$  (no requirement for other metals)
- Size: 1 size
  - Between  $200\text{cm}^2 - 625\text{cm}^2$  and no more than 10cm-25cm length
- Ruggedness: When used on a smooth surface vinyl composite floor with a minimum of 95% revealing no holes or tears



## ASTM D7707

- Beryllium background  $<0.0005\mu\text{g}$  (no requirement for other metals)
- Size: 2 sizes
  - Smaller  $16\text{cm}^2 - 20\text{cm}^2$  with dimensions of 4cm-8cm length
  - Larger:  $200\text{cm}^2 - 400\text{cm}^2$  with dimensions of 10cm-20cm length
- Ruggedness: When used on a  $2000\text{cm}^2$  area (large) or  $500\text{cm}^2$  area (small) of a vinyl or urethane-coated vinyl tile surface without tearing



## History, continued



- There are many brands of wipes that meet ASTM E1792.
- Savannah River Site selected Ghost Wipes™ for beryllium sampling as well as lead and other metals surface wiping.
- The wipes meet the standard requirements for beryllium sampling for most parameters
- The unknown was “Are Beryllium background levels  $<0.0005\mu\text{g}$ ”. This could not be verified by the ICP-ES used in the IH laboratory.
- Ghost Wipes™ consistently showed a Be background of  $<0.02\mu\text{g}$ , the ICP-ES detection limit

## History, continued

- **New Limitations with the Ghost Wipe™ were noted when lower detection limits were considered.**
- **The ability to use ICP-ES for a lower detection limit had been exhausted.**
- **An ICP-MS dedicated to IH work was not in the budget.**
- **Optical Fluorescence made lower detection limits possible and was budget-friendly, but larger volumes of ammonium bifluoride were required to submerge the Ghost Wipe™ resulting in a minimum dilution factor of 100 and negating the detection limit advantage.**



## History, continued



- After ASTM D7707 was published, Environmental Express began offering two sizes of wipes that meet the newer standard and were targeted for beryllium sampling.
- SRS had an interest in trying out the newer wipes because:
  - Certified to meet D7707 standards
  - “Free” of beryllium.
- The smaller wipe was selected because:
  - Potential to lower the detection limit
  - Introduction of less wipe residue into analytical equipment (ICP-ES)
  - Less tendency to over-saturate wipes when sampling
- No other suppliers are currently offering the smaller size.
- Some SRS IH have begun using Beryllium Wipes™ for sampling
- Everyone will discontinue the use of Ghost Wipe™ for beryllium sampling starting October 1<sup>st</sup> (except in specific cases, later mentioned).

## ICP-ES Evaluation, current status

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- **Wipe material was spiked at the typical mid-point of the beryllium calibration range**
  - **1.0 ug/ml Be at the instrument**
- **Method Detection Limits were calculated based on the spiked material.**
- **Media blanks were also analyzed.**
- **Data is preliminary. MDLs are properly determined by measuring blanks or samples at/near the reporting limit. Time constraints limited the amount of data that could be collected at the time of this presentation.**
- **It is expected that this preliminary data will mimic actual MDL measurements.**
- **Other polyvinyl alcohol wipes , with diminished size and background Be levels minimized, would be expected to give the similar results.**

# Method Detection Limit Simulation, Ghost Wipes™

## Ghost Wipe™ Method Detection Limit (Taylor/EPA)

Wavelength =		313.042
Standard Conc. Used =		1.00
Units		ug
Media		Ghost Wipe™
Replicate Number <sup>(1)</sup> below	Date	Measured Values
	8/2/2012	0.8290
	8/2/2012	0.8779
	8/2/2012	0.7257
	8/2/2012	0.7382
	8/9/2012	1.0638
	8/9/2012	1.0590
	8/17/2012	0.8649
	8/17/2012	0.8257
	8/20/2012	1.0091
	8/20/2012	1.0141
	8/20/2012	0.9532
	8/20/2012	0.9851
	8/27/2012	0.9612
	8/27/2012	0.9594
	8/28/2012	1.0620
	8/28/2012	1.1146
Recovery, % =		<b>94.0</b>
<sup>(4,5)</sup> Lower Limit of Detection (LLD) =		0.3077
<sup>(4,5)</sup> Lower Limit of Quantitation (LLQ) =		1.54
<sup>(4,5)</sup> Lower Limit of Detection (LLD) =	Simulation if spiked at 0.01	0.003
<sup>(4,5)</sup> Lower Limit of Quantitation (LLQ) =	Simulation if spiked at 0.01	<b>0.0154</b>

# Method Detection Limit Simulation, Beryllium Wipes™

Beryllium Wipes™ Method Detection Limit (Taylor/EPA)		
Wavelength =		313.042
Standard Conc. Used =		1.00
Units		ug
Media		Be Wipe
Replicate Number (1) below	Date	Measured Values
	9/4/2012	0.9787
	9/4/2012	0.9752
	9/5/2012	0.9761
	9/5/2012	0.9928
	9/6/2012	0.993
	9/6/2012	0.9687
	9/12/2012	0.9585
	9/12/2012	0.8651
	9/13/2012	1.0206
	9/13/2012	1.0274
	9/17/2012	0.9568
	9/17/2012	0.9326
	9/17/2012	0.9352
	9/17/2012	0.9241
	9/19/2012	0.9697
	9/19/2012	0.977
Recovery, %=		<b>97</b>
(4,5)Lower Limit of Detection (LLD) =		0.1015
(4,5,6)Lower Limit of Quantitation (LLQ) =		0.51
(4,5)Lower Limit of Detection (LLD) =	Simulation if spiked at 0.01	0.001
(4,5,6)Lower Limit of Quantitation (LLQ) =	Simulation if spiked at 0.01	<b>0.0051</b>

## A Comparison of Blank Values

Rep	GW	BeW
1	0.003636	0.0039
2	0.003654	0.0015
3	0.002566	0.001884
4	0.003318	0.0016
5	0.001904	0.0030
6	0.00285	0.001662
7	0.00346	0.0026
Average	<b>0.00306</b>	<b>0.00231</b>
Std Dev.	<b>0.00065</b>	<b>0.00089</b>

### No Statistically Significant Difference Noted

#### Qualifications

- ICP-ES Blank Values much less than typical detection limit of 0.02 $\mu$ g
  - Only 7 replicates available for Beryllium Wipes™

## Field Experience with the Smaller Wipes

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Field IH are now testing the smaller wipes on “real world” sample environments.

### Current Sampling Practice:

- Wipe across 100cm<sup>2</sup>
- Fold in half
- Repeat wipe
- Fold in half
- Repeat third time
- Load into container for transport to lab

## Field Experience with the Smaller Wipes, continued

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### Observations:

- **Material used for both wipes was the same and there were no issues with ruggedness**
- **No differences were seen in wipe packaging and wetness**
- **Most differences and adjustments were in regard to using the smaller sized wipe**
  - **Small Beryllium Wipes™ are ~15% smaller than traditional Ghost Wipes™**
  - **The Smaller Sized wipe resulted in a final folded wipe to be 1X1.5” (Ghost Wipes™ are ~2.5X3” after folding twice)**

# Field Experience with the Smaller Wipe, continued



## Field Experience with the Smaller Wipes, continued

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**Concerns raised about more finger/hand contact with beryllium contaminated surfaces**

- **Cross Contamination**
- **Increased risk of exposure**
  - **Field observations noted that personnel performing the sampling change gloves after each “wipe” of the surface, regardless of wipe size.**
- **Some surfaces are very rough/abrasive with high levels of debris**
- **In some instances, larger, less “defined” surface areas are wiped.**
  - **The larger Beryllium Wipe™ or the Ghost Wipe™ may still be used in cases where the surface area may not be suitable for the small wipes. D7707 allows for the use of both sizes.**

# Summary

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- **Beginning Oct 1<sup>st</sup>, all field IH will begin using the smaller Beryllium Wipe™ for routine surface sampling.**
- **In circumstances where smaller wipes are not practical, the option for use of the larger wipes is still available.**
- **IH techs and laboratory personnel will continue to provide feedback regarding use of the smaller wipes**
- **Feedback will be provided to the wipe supplier(s) if ideas for improvement are noted.**
- **Only the Beryllium Wipe™ was evaluated/implemented in this study because currently no other wipe is available that proclaims it is “ASTM D7707 compliant”.**

## Next in Line...

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- **Additional MDL testing of the smaller wipes on the ICP-ES**
- **Optical Fluorescence Testing using the smaller wipes**
- **Expected revision of Reporting limits, possibly for both ICP-ES and Optical Fluorescence.**
- **Comparison of Ghost Wipes™ with the larger Beryllium Wipes™**

# And The Winner Is?

