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Beryllium Surface Wipes – Where We Are Today (and yes, size does matter!)

What the Lab Folk Would Like You to Know

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History of Beryllium Surface Wipes

- In the beginning (at least for DOE), 10 CFR 850
  - 0.2 μg per 100 cm^2
- Over the next several years, some key issues:
  - Wet versus dry
  - Wetting agents
  - Wipe size
  - Wipe composition

Questionnaire – March 2004

Site	Media	Dry/Wet and Wetting Agent	Collection Method
1	Ghost Wipe®	Water	NIOSH 9100/ASTM D6966
2	Ghost Wipe®	Wet	(1)
3, 4	Ghost Wipe®	Alcohol	HUD (1995)/OSHA 125G
5	Ghost Wipe®	Organic	ASTM D6966
6	6 x 6 Gauze	Methanol	EPA 6010
7	Whatman® 41/541 or linen cloth	Dry and wet (water)	NIOSH 9100 (mod)
8	Whatman® 50 smear tab	Dry	In-House
9	Smear Tab	Water	ASTM E1728
10	Whatman® 41	Dry and wet (water)	NIOSH 9100 (mod)
11	Ghost Wipe®, Whatman® 41, or smear tab	Dry and wet (water)	NIOSH 9100
12	Ghost Wipe®	Water	(1)
13	Whatman® 41, Ghost Wipe®	Dry and wet (water)	(1)
14	Ghost Wipe®	Water	NIOSH 9100
15	Smear Tabs, Whatman® 41, Ghost Wipe®	Water	OSHA 125G
16	Whatman® 41	Dry	In-House

(1) means method not identified

Source: Brisson, Ekechukwu, Ashley, and Jahn, Opportunities for Standardization of Beryllium Sampling and Analysis, *J. ASTM Int.*, 2006, Vol. 3, No. 1

History of Surface Wipe Standards Within ASTM

- ASTM E1728, wipe sampling for settled dust samples for lead
  - First approved in 1994, current version 2016 - individually packaged wet wipes
- ASTM E1792, specification for wipe sampling materials for lead
  - First approved in 1994, current version 2016
  - Size range between 200 cm^2 and 625 cm^2
- ASTM D6966, wipe sampling for settled dust samples for metals
  - Modeled after E1728 and E1792
  - First approved in 2003, current version 2013 (minor revisions pending now)
- ASTM D7296, dry wipe sampling for beryllium and compounds
  - One of the earliest standards with BHSC/SAS involvement
  - First approved in 2006, current version 2012 (minor revisions pending now)
  - Modeled after D6966
  - Intended solely for applications where a wetting agent would damage the surface
- For all of the above, background level shall be $\leq$0.1 of target metal concentration
- For E1728 and D6966, wetting agent not specified

Development of ASTM D7707 (small-size wipe)

- Originally approved in 2011, re-approved without change in 2016
- Direct result of BHSC/SAS discussions
- Details on following slides
- Environmental Express offered a small-size Ghost Wipe® that meets D7707 criteria
- Sales were not sufficient to continue offering it
- SRS has had recent discussion with supplier about whether these could be made available again
  - If not, SRS will have to go to something else which may impact our laboratory reporting limit

Development of ASTM D7707 (small-size wipe)

ASTM D7707

- *defines a wipe as* a disposable towelette that is premoistened with an acceptable agent and subsequently used to collect a sample of settled dust on a surface for beryllium analysis.

- is intended for use by manufacturers and suppliers to evaluate the performance of beryllium wipe sampling materials and by users of wipes to compare the performance of candidate wipes for the sampling of beryllium in surface dust.

Development of ASTM D7707 (small-size wipe)

Standard Requirements:

- Clean and free of imperfections that would affect performance
- Established expiration date, marked on each package (not less than one year from manufacture date)
- Background beryllium less than 0.0005 µg
- Sufficiently rugged (large wipe capable to wipe a 2000 cm^2 ; small wipe, a 500 cm2 of vinyl or urethane-coated vinyl tile surface without tearing)
- Fully wetted upon removal from package with a moisture content such that the coefficient of variation for a random sampling of the lot of wipes be no greater than 25 % across individually packaged wipes, and 25 % within and between multi-packaged wipes
- Large wipes are 200 - 400 $cm^{2,}$ with the mean dimension of a side or diameter no smaller than 10 cm or larger than 20 cm
- Small wipes are 16 - 38 $cm^{2,}$ with the mean dimension of a side or diameter no smaller than 4.0 cm or larger than 8.0 cm

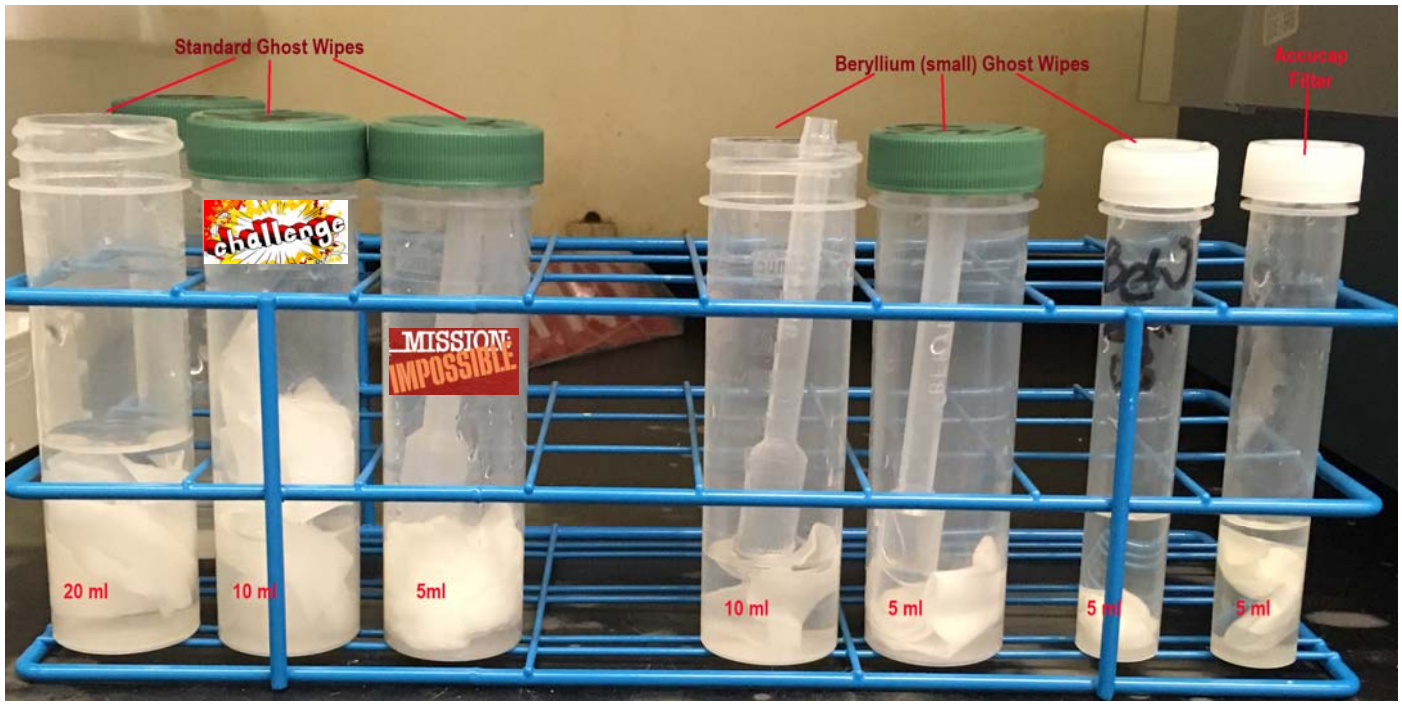
- Dry Wipes (without moistening agent) shall
  - Have a mean thickness of at least 0.005 cm, but no greater than 0.05 cm
  - The coefficient of variation in mass of a dry wipe lot no greater than 10 %

Development of ASTM D7707 (small-size wipe)

- Background beryllium less than 0.0005 µg
- Mean beryllium wipe recoveries(spiked with a Certified Reference Material) within 20% of the reference value at a 95 % confidence level
- Collection efficiency tested on vinyl tile test surface using a solid beryllium-containing reference material
- Mass of beryllium-containing material loaded is 0.50 +/- 0.05 g for the large wipe and 0.050 +/- 0.005 g for the small wipe. (95% of the individual wipes to obtain at least a 75% collection efficiency against the known beryllium mass loaded, utilizing the appropriate surface area for the wipe size
- Wrapped individually to dispense one wipes one at a time
- Each package or container marked with supplier's name, date of manufacture, lot number, expiration date, and "Meets ASTM D7707 for Large Wipes" or "Meets ASTM D7707 for Small Wipes," as appropriate
- General packaging states that the material "Meets ASTM D7707 for Large Wipes" or "Meets ASTM D7707 for Small Wipes," as appropriate, and supporting performance data are available upon request.

Why Size of Wipe Matters

- Wipe samples are digested or extracted in acid solutions
- The bigger or bulkier the wipe, the more acid solution is needed
- The more acid solution, the higher the dilution factor
- The higher the dilution factor, the higher the laboratory reporting limit



Why Size of Wipe Matters

Additional Parameters		Typical Pb Wipe	Beryllium Ghostipe™	Whatman 541™	Partial Ghostipe™ (cut)	37MM Glass Fiber Filters*
Volume ABF	Digestion Tube capacity (ml)	50	15-50	15	15-50	15
	Extraction Volume (minimum to submerge with no gel formation)	30	10	5	10	5
[2]ICP-ES	[5]LLQ @ instrument *(ppb)*	1	1	1	1	1
	[5]LLQ Final *(µg/wipe)*	0.03	**0.01**	**0.005**	**0.01**	**0.005**
[3]ICP-MS	[5]LLQ @ instrument *(ppb)*	0.2	0.2	0.2	0.2	0.2
	[5]LLQ Final *(µg/wipe)*	0.006	**0.006**	**0.006**	**0.006**	**0.006**
[4]Optical Fluorescence	[5]LLQ @ instrument *(ppb)*	0.2	0.2	0.2	0.2	0.2
	[5]LLQ Final *(µg/wipe)*	0.03	**0.01**	0.005	**0.01**	**0.005**

1) Glass fibers may compete with beryllium for fluoride dissolution (not typically an issue except when competing actinide

2) ICP-ES values provided assume ion exchange interference removal prior to analysis.

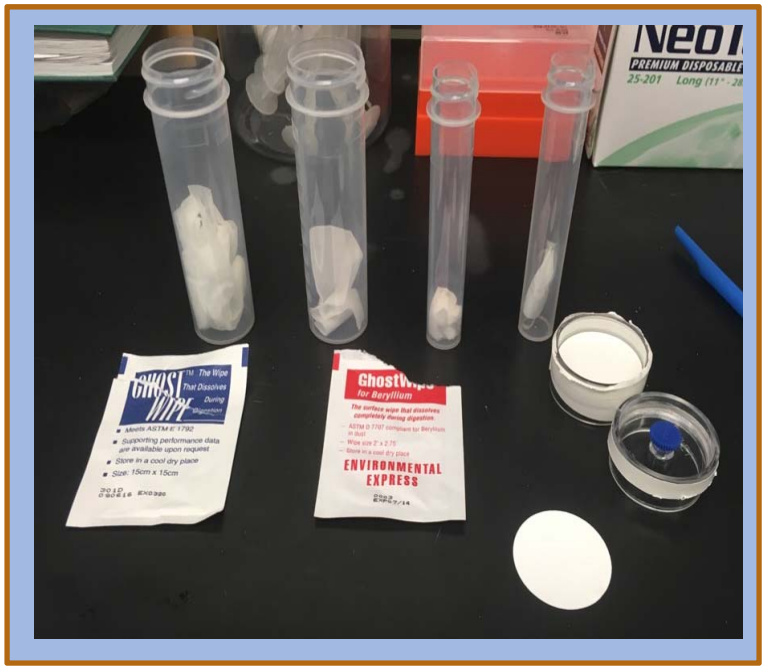
3) ICP-MS includes a total dilution of 30 to reduce concentration of co-existing metals in the solution

4) Optical Fluorescence includes a final dilution of 5X to prep for measurement.

5) Typical LLQ are provided for comparison but can vary greatly even using the same instrument.

Why Size of Wipe Matters

Wipe size impacts analytical detection values significantly.



Increased volume required to submerge larger wipes can completely counter any benefit of using more sensitive laboratory equipment.

Additional Challenges: Inconsistent Liquid Volume

Moisture is desired in a wipe, but moisture content varies

Liquid content dilutes ammonium fluoride concentration, potentially reducing the effectiveness of the dissolution.

Additional Challenges: Compatibility

High nitrocellulose content in some (larger) wipes will interact with ammonium bifluoride, causing a gel formation.

Analyzed portion not representative of entire sample

Non-ABF digestions systems also negatively impacted by larger sized nitrocellulose wipes.

Residual solids require additional filtration or lead to obstruction in instrument components.

Shorter Consumable Shelf Life = Increased CO$T

Larger wipes increase reaction with oxidizers used during digestion (nitric acid)

Savannah River Site Experience

2011:

- ASTM D7707 standard approved.
- Environmental Express offers a small-sized surface wipe, specific for beryllium analysis, specifically in response to the ASTM standard language.
- Savannah River Site implemented use of these smaller wipes immediately.

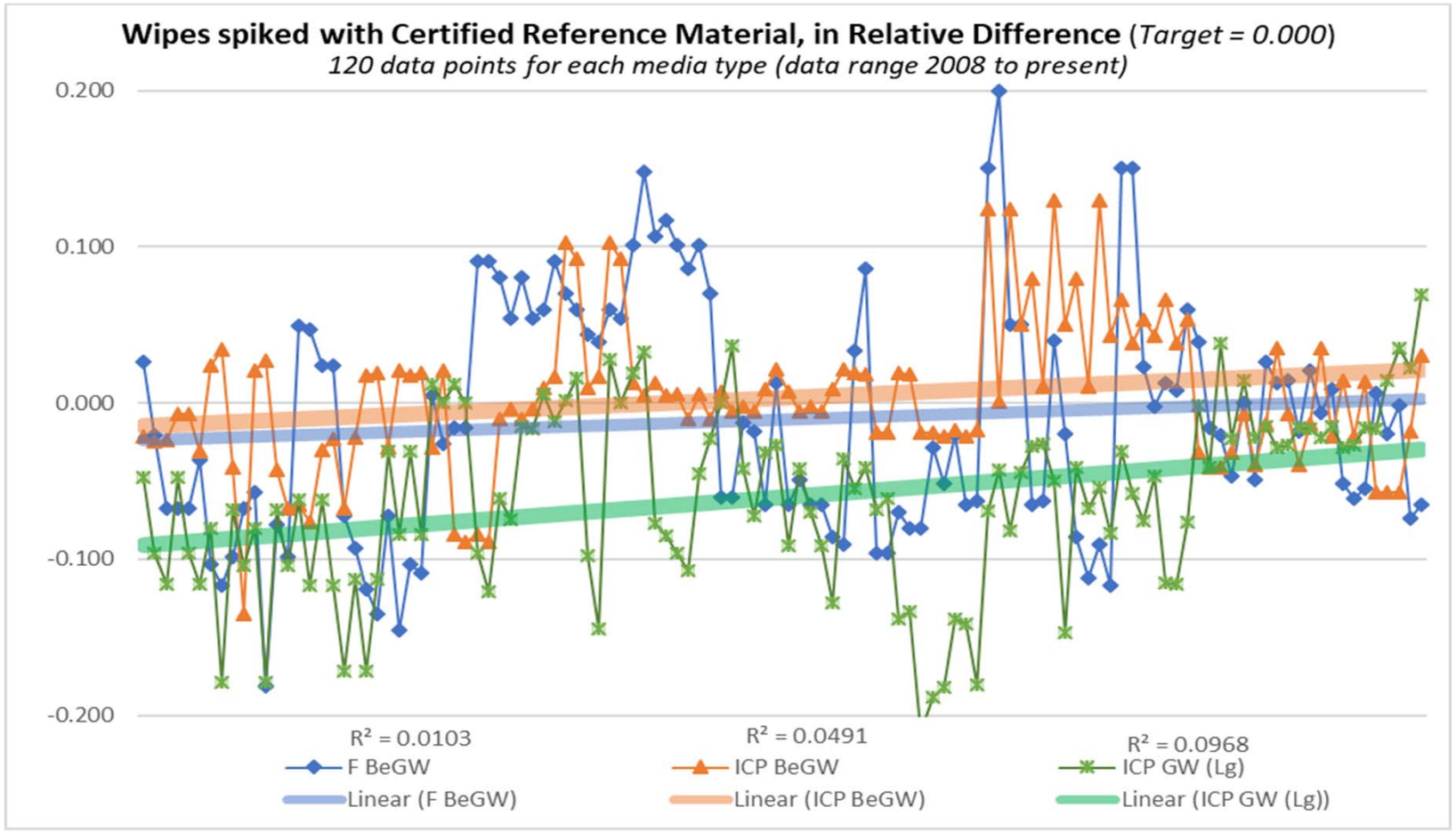
2014

- Environmental Express discontinued production of smaller beryllium wipes
- Savannah River Site continued using these beryllium wipes from the surplus stock, extending the expiration date (validation based upon continued blank performance, physical examination of package integrity, and consultation with manufacturer)
- This was to be an interim measure until a new wipe material was identified that fulfilled analytical capability and packaging specified by the Industrial Hygiene customer.

2018

- SRS Industrial Hygiene requires the wet wipes to be individually packaged and to meet the requirements of ASTM D7707
- The surplus of expired beryllium wipes has diminished significantly.
- Laboratory in the process of determining LLQ impact to beryllium, utilizing optical fluorescence.
- If acceptable LLQ can not be met, purchase and setup of additional laboratory equipment (ICP, etc.) may be necessary (IH ICP in the radiological laboratory is near “end of use”, no longer covered by instrument vendor).

Savannah River Site Experience: Spiked Media Comparison



Opportunity

Environmental Express is willing to periodically manufacture batches of beryllium wipes if a minimum purchasing commitment can be obtained.

Savannah River Site has expressed interest in purchasing, but has a small beryllium scope, the minimum quantitiy is much greater than would be required.

Interested in purchasing the smaller beryllium wipe?

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