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# **Progress Report**

# **BH&SC Analytical Subcommittee**

**Mike Brisson (SRS), Chairman**

**October 20, 2004**

**WSRC-MS-2004-00733S**

# Subcommittee Charter

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- Key goals:
  - Improve quality and consistency of methodologies
  - Foster communications among analytical and IH communities
  - Develop technical guidance and offer assistance to others

# Current Subcommittee Membership

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## DOE Sites Represented:

- DOE-HQ (EH-52)
- Hanford
- KCP
- LANL
- LLNL
- NTS
- ORNL
- Paducah GDP
- Pantex
- Rocky Flats
- Sandia (NM)
- SRS
- Y-12

**Cross-Section of IH, Analytical Chemists,  
Lab Managers, and Researchers**

## Other Agencies Represented:

- AWE-Aldermaston
- Dept. of Defense (Army and Navy)
- IRSST (Quebec)
- NIOSH (Cincinnati and Morgantown)
- OSHA (Salt Lake City)

## Opportunities (partial list):

- Brush-Wellman
- AIHA
- PNNL

# Subcommittee Activities

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- Regular conference calls since 12/03
  - Every 2-3 weeks
  - Typically 15-20 participants each call
- Subcommittee meetings in tandem with full BH&SC meetings

# Subcommittee Working Groups

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- Sampling (co-leads: David Weitzman, Steve Jahn)
- Digestion (lead: Tom Oatts, Y-12)
- Analysis/Reporting (lead: Kevin Ashley, NIOSH)
- Accreditation (lead: Melecita Archuleta, Sandia)  A 3D-style icon with the word "New" in yellow and orange, tilted upwards.
- Future Needs (lead: Amy Ekechukwu, SRS)

# Major Accomplishments since April

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- Baseline questionnaire compilation **ASTM Topic**
- Progress on standard protocols **ASTM Topic**
- Progress toward BeO SRM
- Study plan for different wipes
- Round robin in progress for LANL-developed field analyzer (“BeFinder”) **ASTM Topic**
- Draft analytical guidance for Release Criteria Technical Standard
- Radiological beryllium analytical capabilities
- ASTM Beryllium Symposium (April 2005)

# Questionnaire Summary - Background

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- Total responses – 17 (U.S., U.K., Canada)
- Four have processes producing BeO
  - Firing temp. unknown or classified
- Five handle rad-contaminated samples
- Annual sample volumes:
  - Air: 25,000 (range 37 to 12,000); AWE does nearly as many as all others combined
  - Wipes: 96,000 (range 3 to 33,250)
- U.S. and Canadian labs are AIHA accredited
  - Exception: SRS Rad IH lab is “equivalent” but is pursuing accreditation
  - AWE has HSE approval

# Questionnaire Summary – Wipe Sampling

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- Media
  - Most use Ghost Wipes
  - Whatman 41, ECM, Smear Tabs also used
  - 12 use wet wipes; 6 use dry (H<sub>2</sub>O is dominant wetting agent but alcohols also used)
- Sampling Plans: split between overall strategy guidance and investigation-specific plans
- No consensus on collection methods
  - HUD, NIOSH, ASTM, EPA, OSHA
- Nearly unanimous call for BeO SRM for use in PAT testing

# Questionnaire Summary - Digestion

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- Energy systems: hotplate, hotblock, microwave (OV and CV)
- Wide variety of reagents/combinations
  - $\text{HNO}_3$ ,  $\text{H}_2\text{O}_2$ ,  $\text{HCl}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{HClO}_4$ ,  $\text{HF}$
- Final sample volumes
  - Air filters: Range 5-50 mL, avg. 21 mL
  - Wipes: Range 5-100 mL, avg. 40 mL
  - Affects reporting limits
- Most sites have done recovery studies
- Wide variety of digestion and storage vessel materials

# Questionnaire Summary – Air Analysis

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- Wide variety of methods
  - NIOSH 7300 (7 total, 2 with mods), EPA (3), OSHA (2), in-house methods (3)
- Reporting limits vary widely
  - Differences in units ( $\mu\text{g}/\text{filter}$ ,  $\mu\text{g}/\text{sample}$ ,  $\mu\text{g}/\text{L}$ , ppb) make evaluation difficult
- Variety of batch sizes, QC, blanks, and spikes
- Instrumentation: ICP-ES (11), ICP-MS (3 primary, 2 secondary), AA (1)
- Emission lines (ICP-ES): 313.107 nm, 313.042 nm, 234.861 nm

# Questionnaire Summary – Wipe Analysis

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- Wide variety of methods
  - NIOSH 7300 (7 total, 1 with mods), EPA (4), OSHA (2), in-house methods (2)
- Reporting limits vary widely
  - Differences in units ( $\mu\text{g}$ ,  $\mu\text{g}/\text{sample}$ ,  $\mu\text{g}/\text{wipe}$ , ppb,  $\mu\text{g}/\text{L}$ ) make evaluation difficult
- Variety of batch sizes, QC, blanks, and spikes
- Instrumentation: ICP-ES (8), ICP-MS (1 primary, 2 secondary), AA (1)
- Emission lines (ICP-ES): same lines but more use 313.042
- Elemental interferences (ICP-ES)
  - Most common: Fe (5); Cr and V (4); Al, Ca, Cu, Mg, Mn, Ti (3); 23 others mentioned by one or two sites
  - Depends on line(s) used

# Standard Protocols Status

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## Sampling Methods

- Guidance: use wet wipes unless they cannot be used for technical reasons
- Wet Wipes – ASTM D6966 – published
- Dry Wipes: ASTM method to be developed
  - Y-12 and NTS methods as templates
- Bulk Sampling – ASTM Micro-vacuum method being balloted
  - Alternative method using disposable sampling chamber under discussion
- Skin Sampling – ASTM method proposed
  - Issue for BH&SC: What are the objectives?

## Sample Preparation

- ASTM method to be developed
- Specific reagent(s) not yet identified, but hoping to avoid use of  $\text{HClO}_4$  or HF

# Standard Protocols Status (cont.)

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## Analysis Methods

- ICP-ES: ASTM method being balloted
- ICP-MS: Evaluate applicability of ASTM D5673
- AA: No standard method yet proposed
- Fluorometric: Data being generated to support an ASTM standard method

## Reporting Limits

- Subcommittee evaluating options

# Be Oxide SRM Status

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- Evaluating BeO from several suppliers
  - All appear based on UOX125
  - Want SRM to be commercially available
- Estimated two-year effort
  - Year 1: Establish SRM pedigree on selected material
  - Year 2: Establish proficiency samples based on BeO SRM
  - Issue for BH&SC: What matrix or matrices should be used for proficiency samples?
- Request for FY05 funding is pending
- LLNL is offering material for round-robin
  - Lead-free soil containing BeO with nominal 0.5  $\mu\text{g/g}$  available Be
- LANL has developed a BeO slurry that will be tested at three DOE sites

# Study Plan for Surface Sampling

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- Would use  $\text{BeSO}_4$  solution
- Would test smooth, slightly rough, and porous surfaces
- Would test several methods
  - Whatman 541 (wet and dry)
  - Ghost Wipes
  - Dry linen paper
  - Kansas City method
  - Vacuum method
  - Pressure controlled method
- Study not yet funded

# BeFinder Round Robin

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- Field-portable analyzer
- Uses 1%  $\text{NH}_4\text{HF}_2$  at room temperature
- Round-robin involving six sites
- Potential issues so far:
  - Recovery on Ghost Wipes
  - Recovery of  $\text{BeO}$  (particle size?)

# Analytical Guidance (Tech Std.)

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First draft submitted to DOE – Outline:

- Purpose of analysis and DQO
- Standard methods
  - Performance-based (alternate methods should demonstrate equivalent or superior performance)
- QA/QC
- Communication between IH and lab
- Field instrumentation
- Lessons learned

# Radiological Beryllium Labs

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- Y-12 can do U-contaminated samples
- Need identified for lab to handle Pu-contaminated Be samples
- SRS has established capability
  - Equivalent under 10 CFR 850 but pursuing accreditation
  - Discussions in progress with interested sites

# Beryllium Symposia

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## ASTM

- April 21-22, 2005 – Reno, NV
- Organizers: K. Ashley, K. Creek, M. Brisson, A. Ekechukwu
- Analytical Subcommittee co-sponsoring
- For all interested parties (IH, analytical, operations, vendors, etc.)
- Abstract deadline has passed

## ACS

- March 13-17, 2005 – San Diego, CA
- One-day session on IH analyses in nuclear environments (Be, Pb, etc.)
- Organizers: A. Ekechukwu, M. Brisson
- Abstract deadline: 11/12/04

## AIHA

- Roundtable proposed for 2006

# Other Open Action Items

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- Develop performance criteria for field analyzers (awaiting funding)
- Resolve shipping issues
- Make AIHA accreditation more “value-added”
  - What evaluators should look for
  - How the post-BeO process should look
  - Input to AIHA policy manual
- Lexicon of standard terminology