

Implications of BeO Reference Material: Spiked Filter Round Robin Results

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BeO Spiked Filter Round Robin

The SAS BeO/Digestion working group has been pursuing two primary goals:

1. A high-fired beryllium oxide certified reference material.
2. Development of a BeO spiked filter round robin for lab method efficiency determination.

We claimed victory on item 1 with the development and production of NIST SRM® 1877, “Beryllium oxide powder”.

Moved on to item 2.

- Became possible with High Purity Standards stepping up and volunteering to prepare and distribute round robin samples.

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- Collect data on existing lab preparation and analysis methods used at this time.
- Utilizes NIST SRM® 1877 as spiking material.
- Provide a blind quality control study for labs to use as method effectiveness information.
- Test of methods needed to produce BeO spiked filters for future studies.
- Provide lab data for solid form of beryllium known to be difficult to digest by some methods.

BeO Spiked Filter Round Robin

- Formal request for participation sent out via e-mail to all BH&SC members.
- Study composed of volunteer laboratories, i.e. study *not funded!*
- Filter samples prepared by chemists from High Purity Standards, Charleston, SC under direction of Amy Adams, Inorganic Lab Manager.
- Project coordinated by Amy Adams and Tom Oatts.
- Labs provided 5 filters, in duplicate, spiked at 0.5 μg –25 μg , and two blanks. (set contains 12 filters for analysis)

BeO Spiked Filter Round Robin

- 27 different labs volunteered
- 36 sets of samples distributed
- 7 labs requested multiple sets to check different methods
- Lab breakdown:
 - 4 unique commercial labs
 - 3 unique military (DoD) labs
 - 4 foreign labs (UK, France, Canada)
 - 16 government labs (DOE, CDC/NIOSH, OSHA)
- 34 labs submitted data

Lab participant listing – part 1

Name of Laboratory	Location
ALS Datachem	Salt Lake City, Utah
B&W Pantex	Amarillo, Texas
Berylliant, Inc.	Tucson, Arizona
Bureau Veritas North America	Novi, Michigan
Center for Disease Control / National Institute for Occupational Safety and Health (CDC/NIOSH)	Cincinnati, Ohio
Comprehensive Industrial Hygiene Laboratory Navy and Marine Corps Public Health Center	San Diego, California
Fluor Hanford	Richland, Washington
Forensic Analytical Laboratories	Hayward, California
Galson Laboratories	East Syracuse, New York
Health and Safety Laboratory	Buxton, Derbyshire, England
Health Physics Analytical Laboratory	Abingdon, Oxon, England
Institut National de Recherche et de Sécurité (INRS)	Vandoeuvre-les-Nancy, France
Institut de Recherche Robert Sauvé et de Sécurité en Travail (IRSST)	Montréal, Quebec, Canada

Lab participant listing – part 2

Name of Laboratory	Location
Los Alamos National Laboratory	Los Alamos, New Mexico
Lawrence Livermore National Laboratory	Livermore, California
Lawrence Livermore National Laboratory / Environmental Monitoring Radiochemistry Laboratory (EMRL)	Livermore, California
Navy & Marine Corps Public Health Center Laboratory	Norfolk, Virginia
National Security Technologies, LLC	Mercury, Nevada
Oak Ridge National Laboratory	Oak Ridge, Tennessee
Occupational Safety and Health Administration (OSHA)	Salt Lake City, Utah
Savannah River Nuclear Solutions, LLC (non-rad IH lab)	Aiken, South Carolina
Savannah River Nuclear Solutions, LLC (rad IH lab)	Aiken, South Carolina
Savannah River National Laboratory	Aiken, South Carolina
U.S. Air Force School of Aerospace Medicine (USAFSAM/OEHTA)	Brooks City-Base, Texas
United States Enrichment Corporation, Inc	Piketon, Ohio
Wisconsin Occupational Health Laboratory	Madison, Wisconsin
Y-12 National Security Complex	Oak Ridge, Tennessee

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- Labs asked to follow normal analytical methods for beryllium filters.
- Data report designed to collect preparation and analysis method details for method comparison purposes and categorization.
- Sample sets included safe handling information, data reporting requirements, and Material Safety Data Sheet.
- Data to be compiled into a report for distribution to volunteer labs and BH&SC members.
- Intend to publish data in peer-reviewed journal

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- Volunteer period: 02/09/2009 – 02/20/2009
- Samples prepared: 02/16/2009 – 02/20/2009
- Samples shipped: 03/02/2009 – 03/06/2009
- Deadline for data submission: 04/03/2009



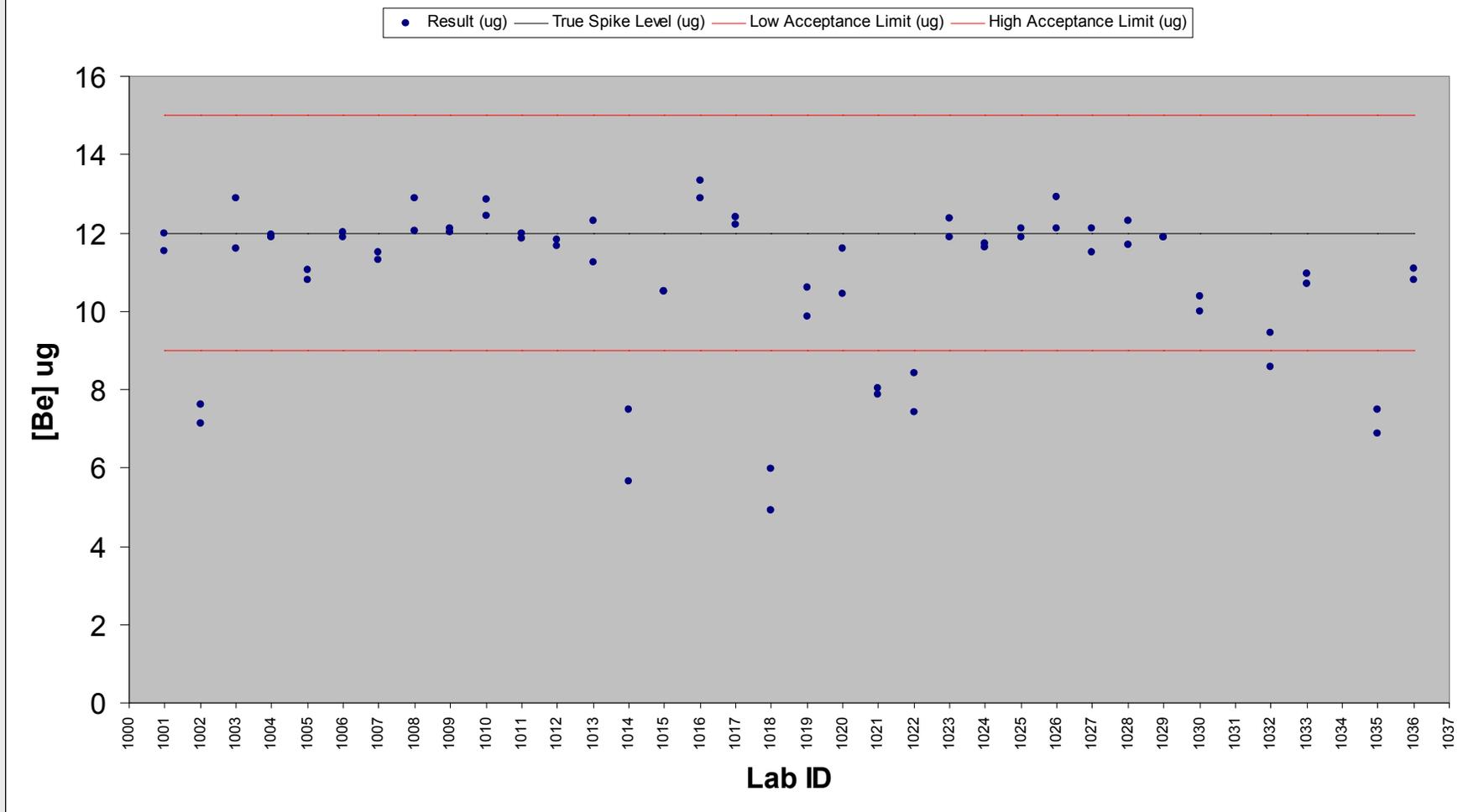
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- Data submitted by 34 of the 36 lab participants.
- Full statistical assessment performed on data set.
- Defined performance evaluation acceptance limits at 75%-125%, similar to existing BePAT limits.
- Pooled data using common sample preparation reagents and like sample prep methods.



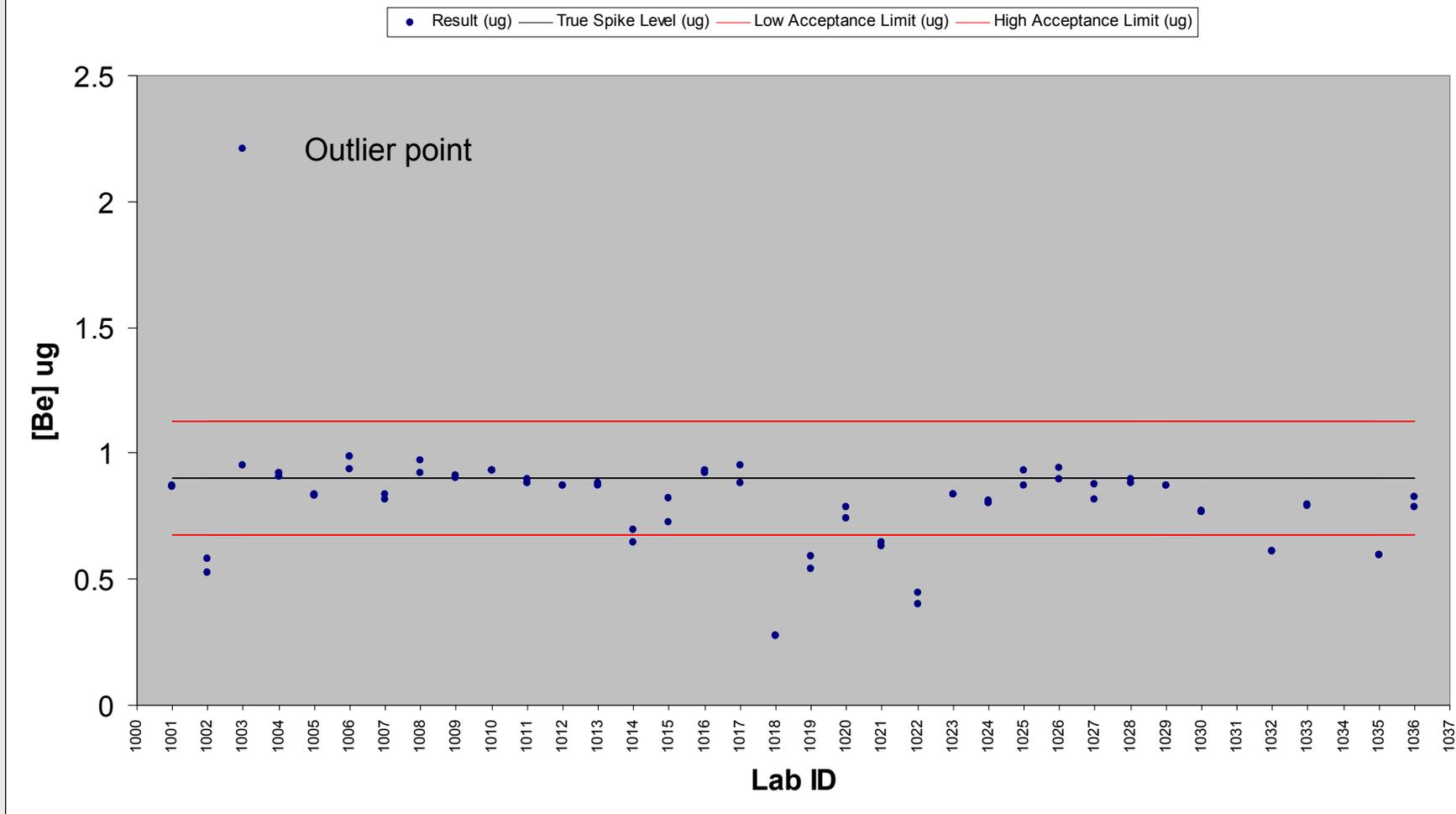
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BeO Round Robin Sample 1 Data



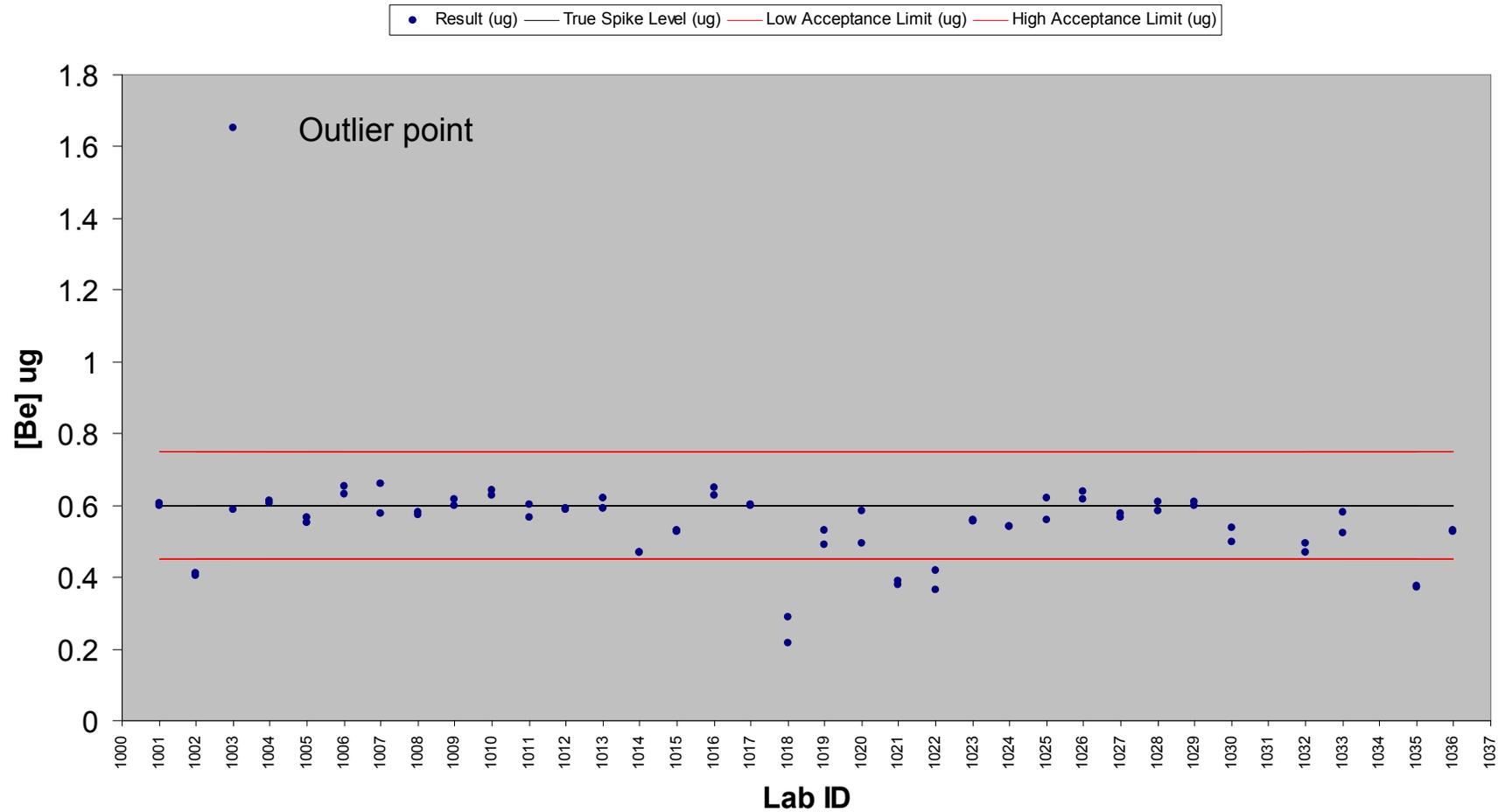
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BeO Round Robin Sample 2 Data



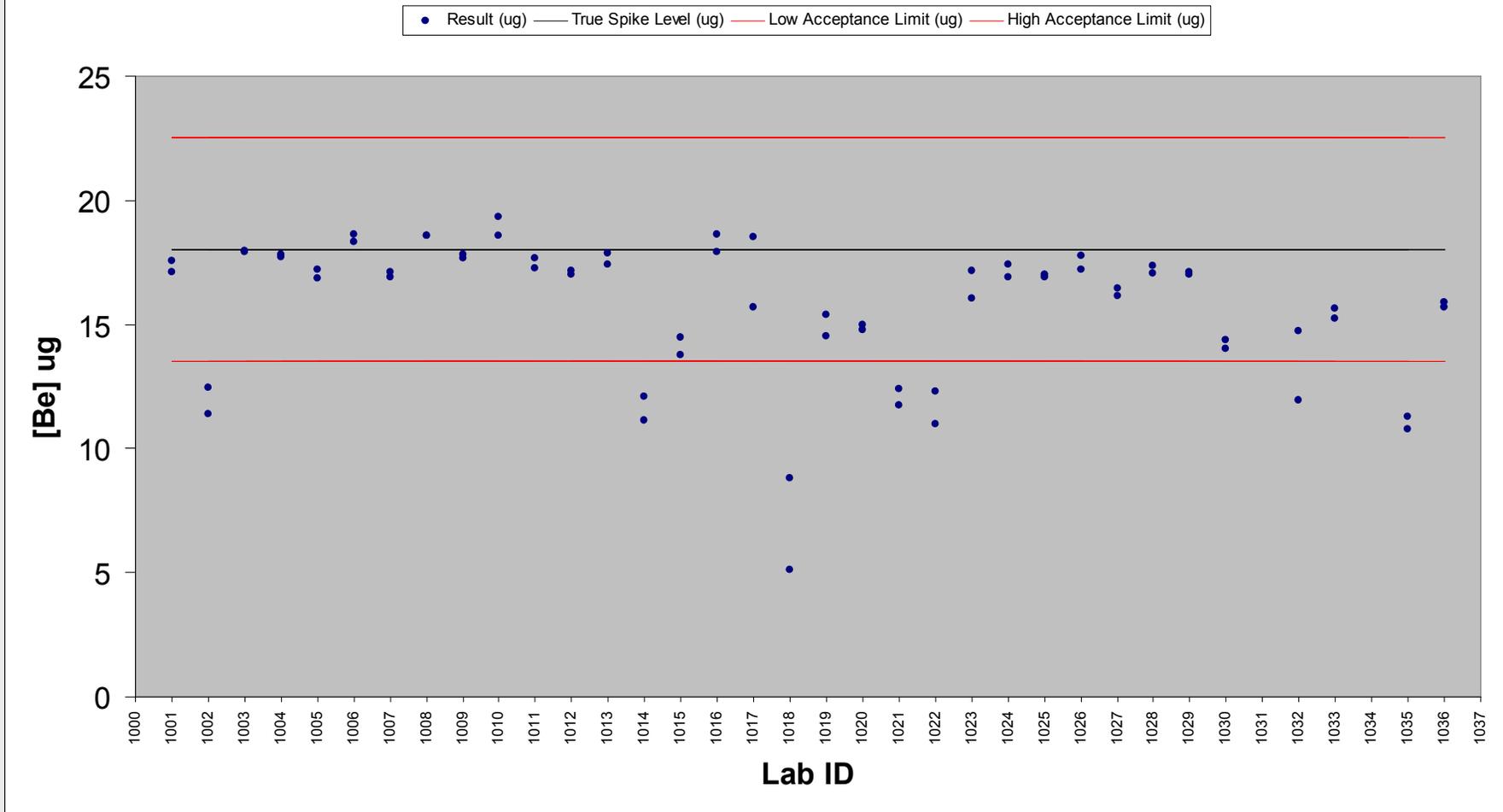
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BeO Round Robin Sample 3 Data



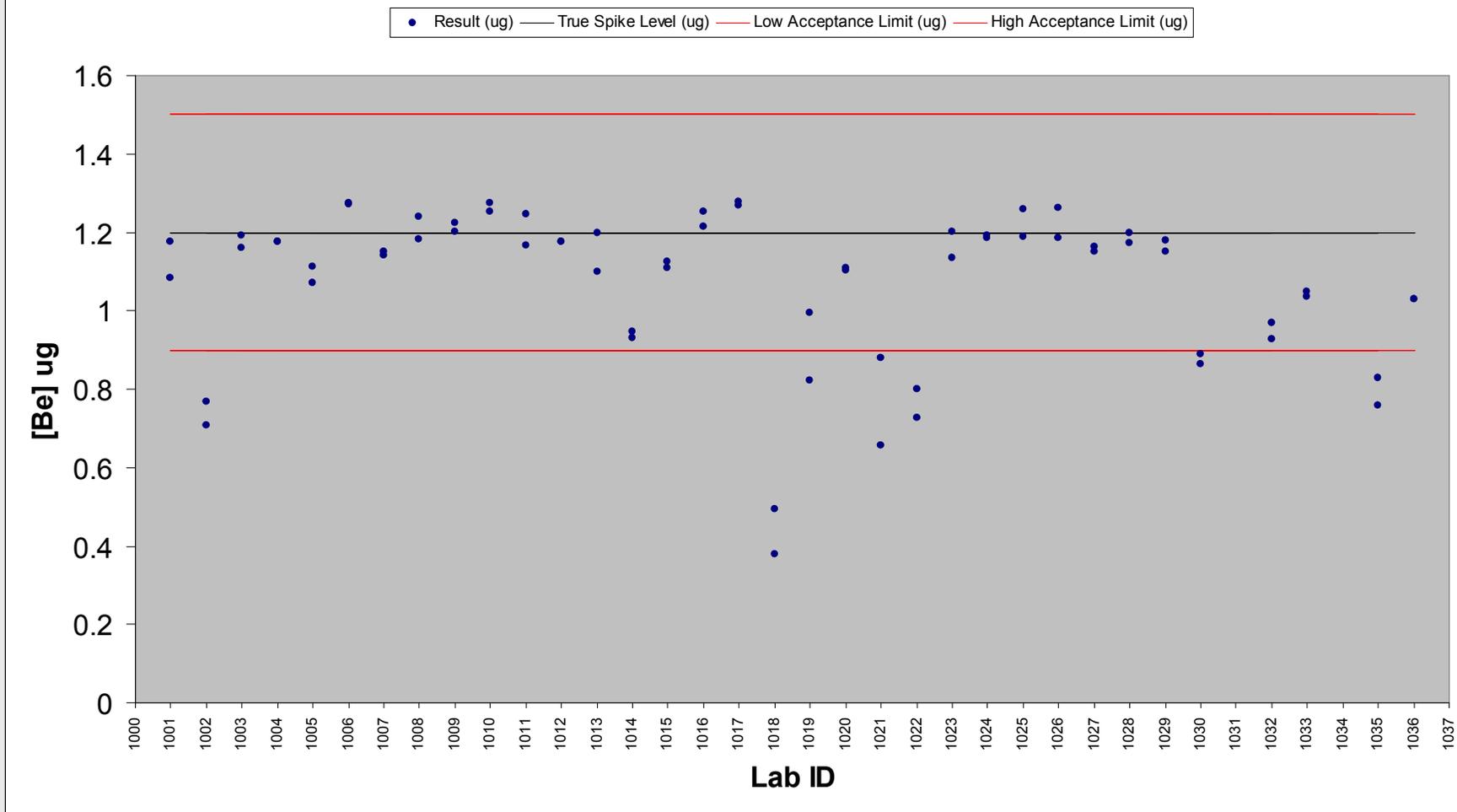
BeO Spiked Filter Round Robin

BeO Round Robin Sample 4 Data

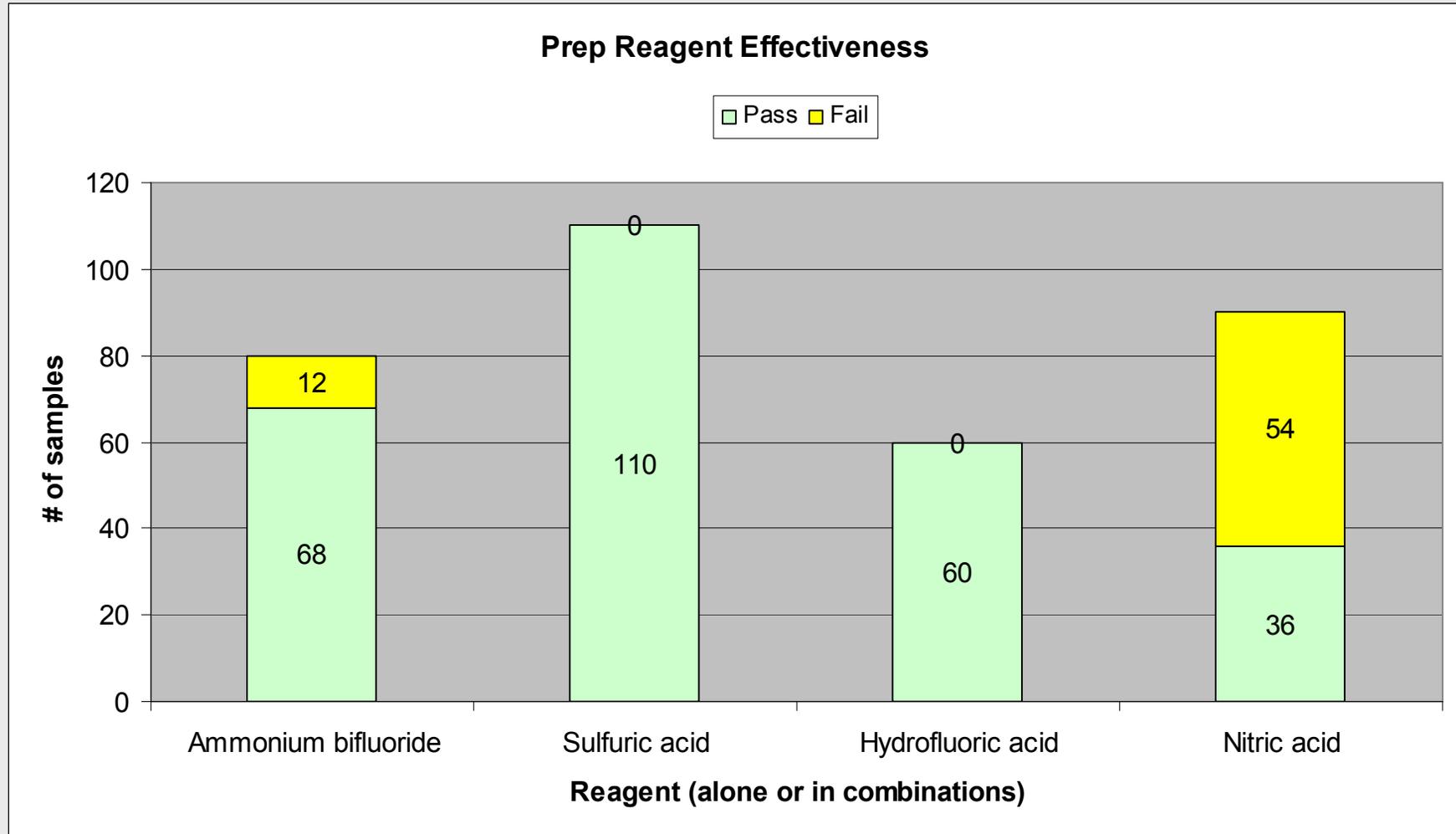


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BeO Round Robin Sample 5 Data



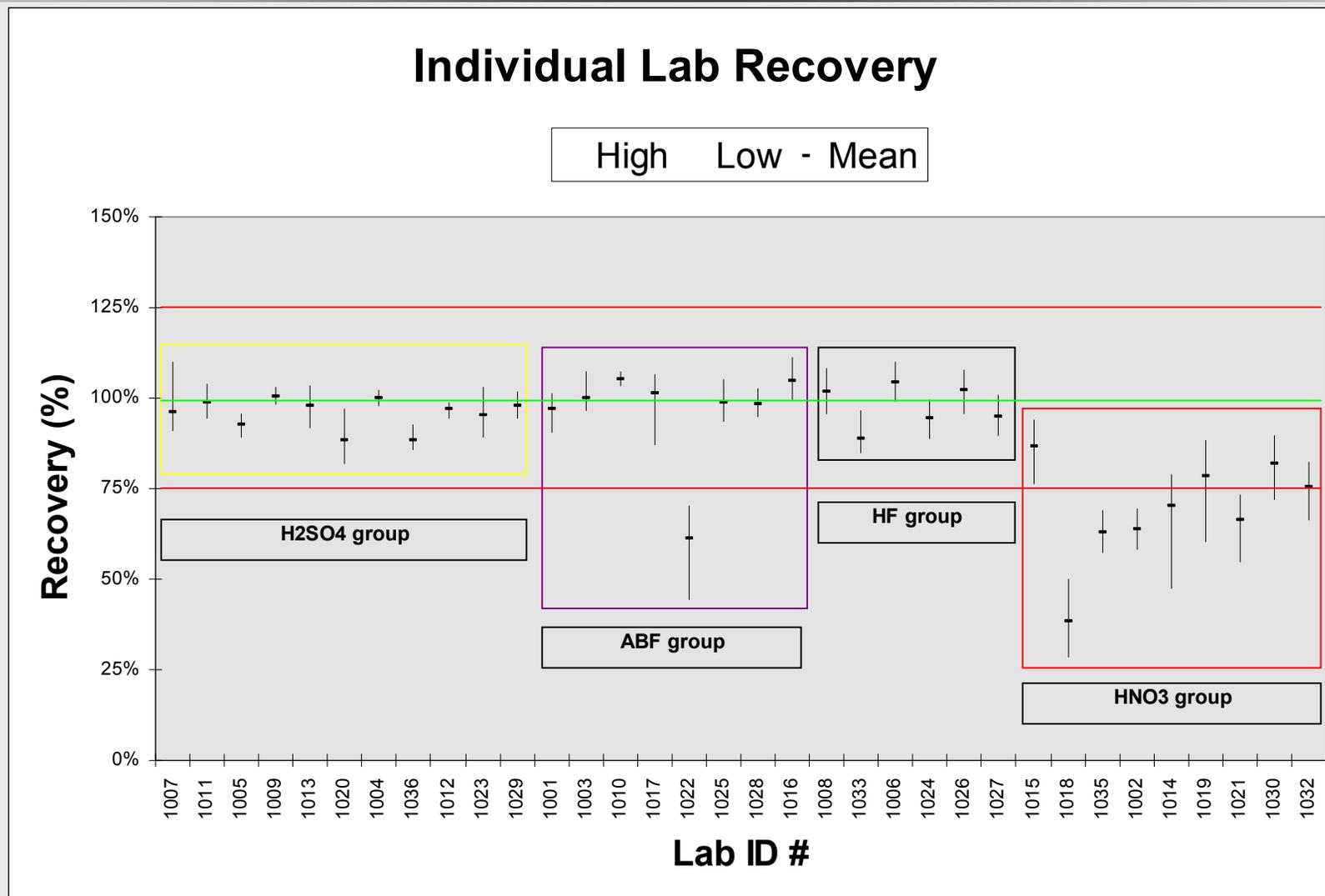
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BeO Spiked Filter Round Robin

Reagents used in prep	Pass	Fail	Total
ABF + HNO ₃ [Group 1]	10	0	10
ABF only [Group 1]	58	12	70
H ₂ SO ₄ + HCl [Group 2]	20	0	20
H ₂ SO ₄ + HNO ₃ [Group 2]	40	0	40
H ₂ SO ₄ + HNO ₃ + HCl [Group 2]	20	0	20
H ₂ SO ₄ + HNO ₃ + HClO ₄ [Group 2]	10	0	10
H ₂ SO ₄ only [Group 2]	20	0	20
HF + HNO ₃ [Group 3]	10	0	10
HF + HNO ₃ + HCl [Group 3]	10	0	10
HF + HNO ₃ + HCl + HClO ₄ [Group 3]	30	0	30
HF only [Group 3]	10	0	10
HNO ₃ + HCl [Group 4]	26	34	60
HNO ₃ only [Group 4]	10	20	30
totals	274	66	340

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Conclusions

- Sample preparation methods employing sulfuric acid, hydrofluoric acid, or ammonium bifluoride are very effective in solubilizing beryllium, in the form of beryllium oxide.
- Methods employing nitric acid, or combinations of nitric, hydrochloric, and/or perchloric acids are not completely effective in solubilizing BeO.
- Techniques designed for multi-element determinations on air filters using nitric acid alone, or with combinations of HNO₃, HCl, or HClO₃, do not solubilize BeO quantitatively.

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