Beryllium Colorimetric Detection for Real Time Monitoring of Laboratory Environments

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Background

- Beryllium (Be) has exceptional material properties (3 times lighter than Al, 6 times stiffer than steel)
- Used extensively in the aerospace, computer, electronics and nuclear industries
- Be is also a toxic metal that poses extreme risks to human health
  - Triggers an cell-mediated immunoresponse in 1-6% of exposed individuals – Chronic Be Disease (CBD)
  - Class A Carcinogen

Introduction

- Be can be present in laboratory working areas and potentially in soils at DOE facilities that have participated in open-air testing of nuclear weapons components.
- Nuclear Stockpile Stewardship Program requires testing of nuclear stockpile.
  - Dual Axis Radiographic Hydrotesting Facility (DARHT) program proposes to contain test shots in an 8' vessel .
  - Advanced Hydrotesting Facility (AHF) program also proposes to contain shots.

Introduction

- Off-Site Source Recovery Project (OSRP) Disposal Team is developing disposal options for sealed radioactive sources that contained Be in contact with actinides (^{239}Pu, ^{238}Pu, ^{241}Am); need to study the fate and transport of Be resulting from disposal of sealed radioactive sources.
- Before any of these research activities can be initiated we need to ensure that we can perform Be activities safely and minimize human exposure.

Be Safe Handling

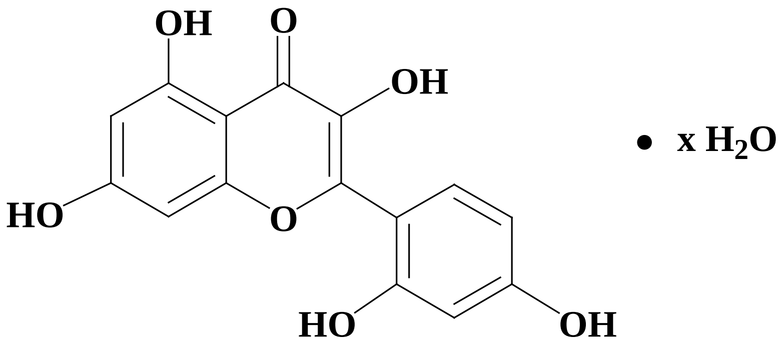
- Laboratory designated for Be activities
- Respirator trained personnel handle solid Be
  - Currently installing a dedicated glove-box
- ESH-5 performs air and surface monitoring on a routine basis
- Colorimetric technique for detecting Be on work surfaces

Test Requirements

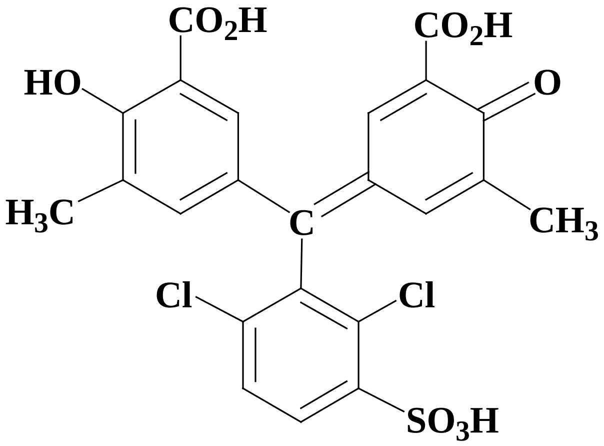
- Easy - straightforward
- Fast - need results ASAP
- Cost effective - current technique OSHA ID-125G (ICP-AES) ~ $250/sample
- Low detection limits
  - 10 CFR 850 (DOE, CBDPP), <3 μg/100 cm^2 during non-operational periods, <0.2 μg/100 cm^2 for release

Colorimetric Reagents

- Morin hydrate (Szczepaniak and Jedrusiak, 1970)



- Chrome Azurol S (Mordberg and Filkova, 1974)



Colorimetric Method

- 1% Chrome Azurol S
- 5% EDTA
- Be contam-inated solution
- Ammonia (1M)/Ammonia Chloride (1M) pH 10 buffer solution
- Detection verified for limit of 0.2 µg Be/100 cm^2

Colorimetric Swipe

- Swipes performed similarly to swipes performed by IH's for the OSHA ID 125-G technique.
- Water or acid can be used to wet the pretreated filter.
- The surface area swiped must be accurately recorded.

Colorimetric Swipe Validation

- Positive Be detection occurs when 2 µg of Be is collected on a filter.
- To achieve detection limit of 0.2 µg/100 cm^2, swipe 30.5 x 30.5 cm^2; to achieve 3 µg/100cm^2, swipe 8 x 8 cm^2

1 µg Be on filter

2 µg Be on filter

Interferences

- Metals commonly found in environments where test will be employed (individual elements, metal mixtures, metallic surfaces)
- Machining fluids (cutting oils, solvents)
- Cleaning fluids (Fantastic)
- Oxidized paint

Interference Elements

- Have characterized the suitability of the technique in the presence of Fe, Al, Ca, Mg, Cu, Ni, Cr, Pb, UO_2 all at concentrations 100 times that of the minimum detectable Be concentration.
- Considered metal mixtures at concentrations that may compete with Be for Chrome Azurol S binding.

Interference Results

205µg M^{2+} | 205µg M^{2+} + 2µg Be^{2+} | 205µg M^{2+} | 205µg M^{2+} + 2µg Be^{2+}

Ca^{2+}

Mg^{2+}

Cd^{2+}

Ni^{2+}

Interference Results

205µg M^{2+} | 205µg M^{2+} + 2µg Be^{2+} | 205µg M^{2+} | 205µg M^{2+} + 2µg Be^{2+}

Cr^{2+}

Pb^{2+}

Cu^{2+}

Fe^{2+}

Interference Results - Uranium

UO_2^{2+} (no Be present):

100 µg

160 µg

205 µg

UO_2^{2+} (with and without Be present):

205 µg UO_2

205 µg UO_2
+ 2 µg Be

UO_2^{2+} (with and without Be present):

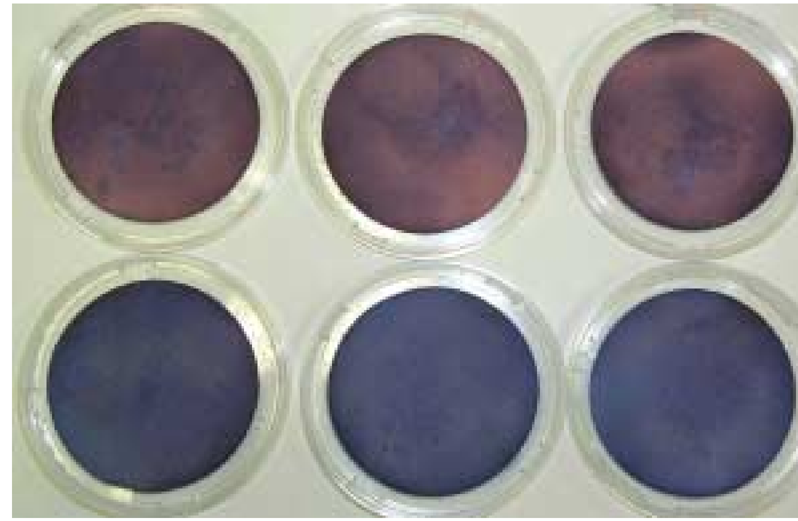
10% EDTA

205 µg UO_2

205 µg UO_2
+ 2 µg Be

Interference Results - Aluminum

Al^{3+} (no Be present):
1) 40 μg
2) 100 μg
3) 160 μg
4) 205 μg



Al^{3+} (with and without Be present):
10% EDTA
205 μg Al^{3+}, top row
205 μg Al^{3+} + 2 μg Be, bottom row

Al^{3+} (with and without Be present):
5% EDTA
205 μg Al^{3+}, top row
205 μg Al^{3+} + 2 μg Be, bottom row

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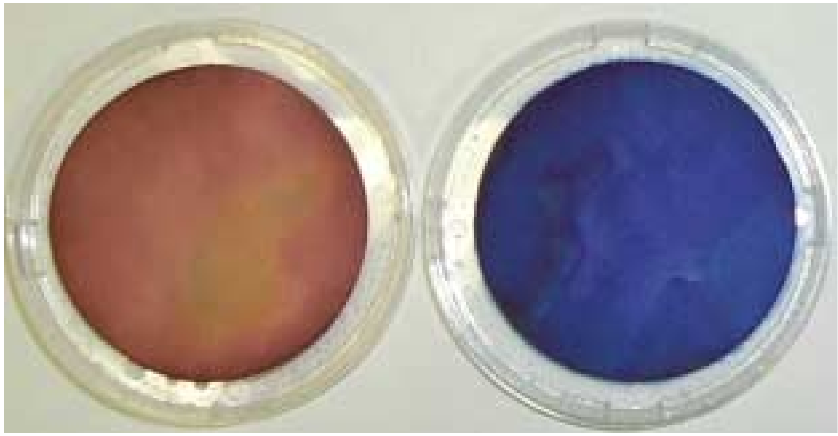
Mixtures of Metals



Left:

Pb: 100 µg
Fe: 40 µg
Cd: 30 µg
Zn: 30 µg
Mn: 20 µg

Right:

Pb: 100 µg
Fe: 40 µg
Cd: 30 µg
Zn: 30 µg
Mn: 20 µg
Be: 10 µg

Left:

Fe: 20 µg
Mg: 20 µg
Pb: 20 µg
Tl: 10 µg
Ni: 5 µg

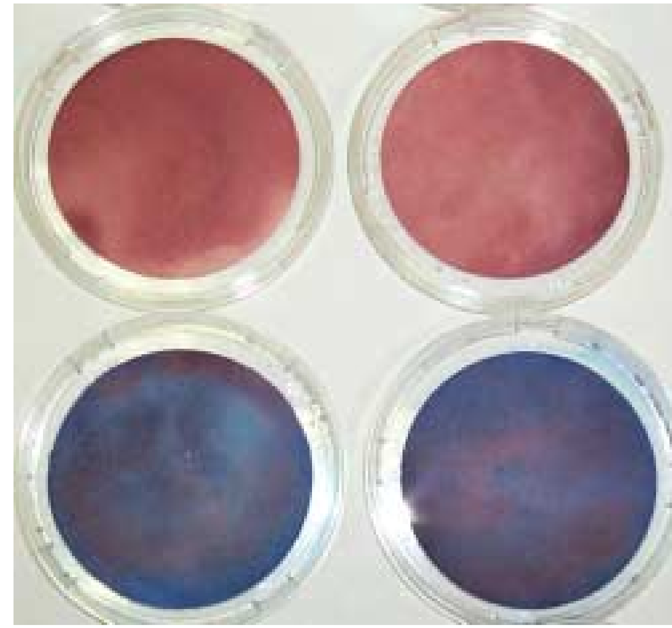
Right:

Fe: 20 µg
Mg: 20 µg
Pb: 20 µg
Tl: 10 µg
Ni: 5 µg
Be: 2 µg

Other Potential Interferences

Oxidized paint on exterior door

Unpainted hood

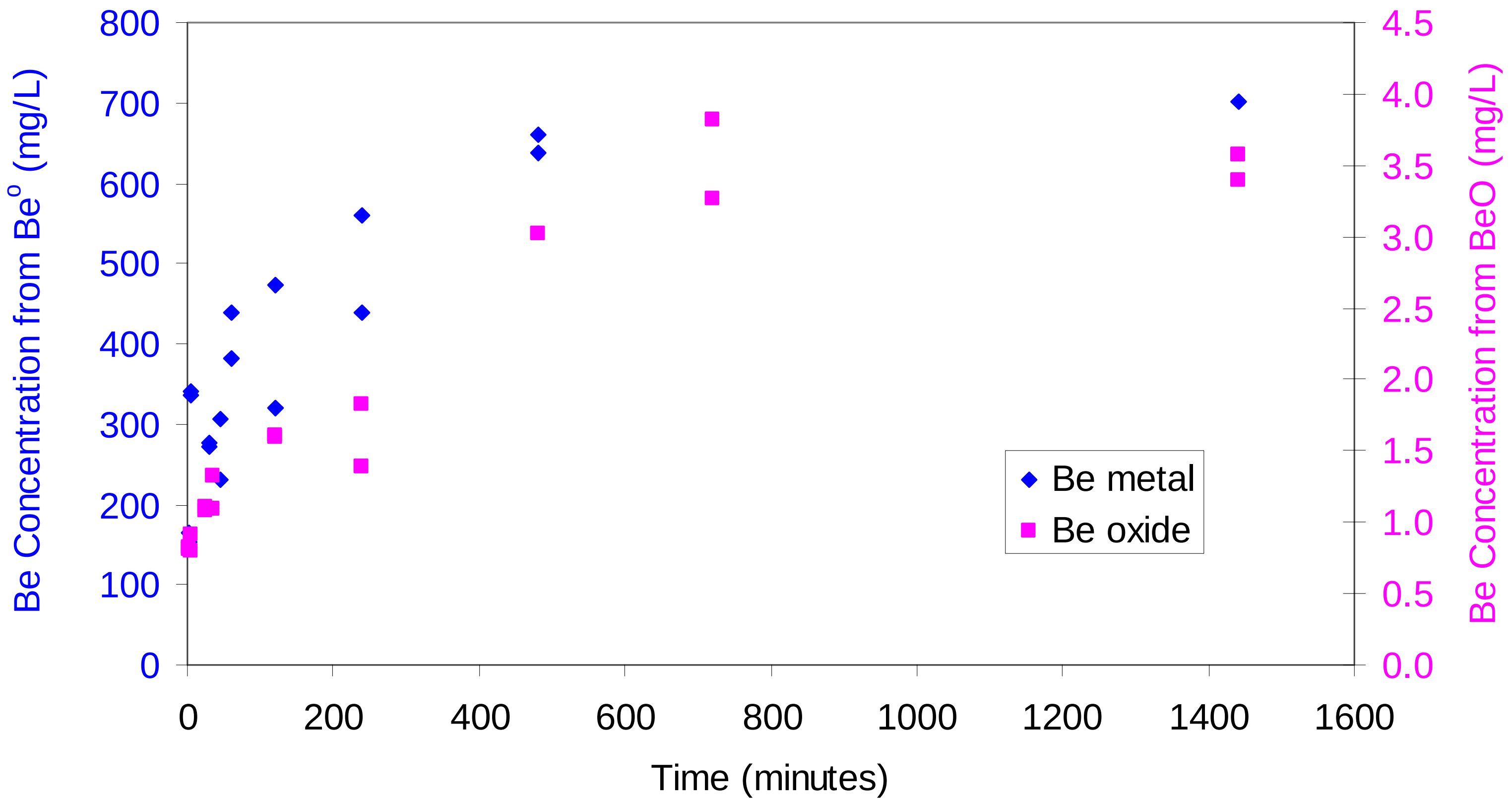


- Machine shop fluids (top)
- Machine shop fluids mixed with 2 µg Be

Solid Beryllium

- Solid forms of Be evaluated included $Be(OH)_2$, $BeCl_2$, $BeSO_4$, Be^o and BeO.
- Kinetic batch results confirmed that $Be(OH)_2$, $BeCl_2$, $BeSO_4$ were completely soluble in 2% HCL within 2 minutes of contact.
- Be^o and BeO were more resistant to solubilization.

Kinetic Batch Tests

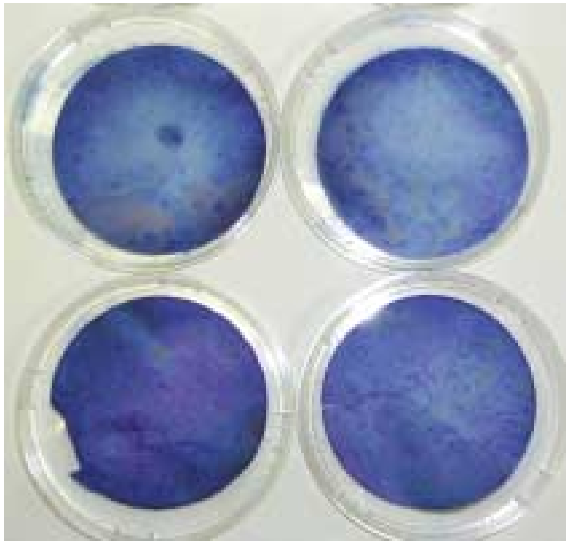


Practical Application – Std. Curve

0 μg Be 2 μg Be 6 μg Be

9 μg Be 19 μg Be

Practical Application



Swipes of a Be glovebag taken immediately after a Be activity

Swipes of a known contamination area in a Be machine shop

Conclusions

- Met our objectives
  - Easy – don’t need to hire a technician, anyone can interpret results
  - Fast – 30 minutes start to finish
  - Cost effective
  - Low detection limit
- Routine housekeeping
- Protection of Be workers