Particulate Generation in a Tritium System

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Outline

• Description of Events
• Analysis of Material
• Sources of material contamination
• System Restoration/Modifications
• Contaminant Minimization and Control
• Lessons Learned
An Unpleasant Surprise

• Let down filter on compressor became plugged after 10 years of operation.
• Tritium processing interrupted when other filters (flow orifices) became plugged approximately two weeks later
• A fine particulate was found widespread in the facility which required mitigation

We were surprised at this revelation and probably should not have been. This is the tenth anniversary of these events. Perhaps we can benefit from a repeat of the story.
Typical Compressor System

- N2 SUPPLY
- CONTAM N2 TANKS
- NI VAC
- P-EVAC PUMP A
- N2 EVAC HDR A
- MIX TKs & TCAP
- Expansion Vol

Components:
- 5 Micron Filter
- 11 Micron Filter
- 10 Mil Orifice
- New Future 5 Micron Filter
- Exist. 5 Micron Filters
- Exist. 11 Micron Filter
- Exist. 5 Micron Filter
Compressor Filter Debris

High-Pressure Fitting Removed from Let-Down Line

As-Removed Flow Orifice

Stereomicrograph of Filter Debris – Broken Loose Outside Compressor System

New Flow Orifice
Filter Debris Analysis

SEM Photomicrograph

X-ray Spectrum
Analysis of Particulates from Compressor Let Down Filter

<table>
<thead>
<tr>
<th>Substance</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum</td>
<td>1.2 wt%</td>
</tr>
<tr>
<td>Calcium</td>
<td>1.5 wt%</td>
</tr>
<tr>
<td>Iron</td>
<td>0.8 wt%</td>
</tr>
<tr>
<td>Silicon</td>
<td>3.1 wt%</td>
</tr>
<tr>
<td>Fluoride</td>
<td>24 wt%</td>
</tr>
<tr>
<td>Ammonium</td>
<td>11 wt%</td>
</tr>
<tr>
<td>Chloride</td>
<td>0.1 wt%</td>
</tr>
<tr>
<td>SubTotal</td>
<td>41.7 wt%</td>
</tr>
<tr>
<td>Dirt/corrosion</td>
<td>3 wt%</td>
</tr>
<tr>
<td>Degraded Teflon</td>
<td>55.7%</td>
</tr>
<tr>
<td>Total</td>
<td>~100%</td>
</tr>
</tbody>
</table>

Analytical Methods:
- ICP—ES on an aqua-regia dissolution
- IC following deionized water dissolution
- IR
- X-ray Diffraction
Analysis of Particulates from Compressor Let Down Filter

X-ray diffraction

- (NH₄)₃SiF₆ - Ammonium Silicon Fluoride
  Cryptohalite, syn - (NH₄)₂SiF₆
- (NH₄)₃AlF₆ - Ammonium Aluminum Fluoride

Intensity (Counts)

Two-Theta (deg)
Formation of Ammonium Fluorosilicate

$$\text{NT}_3(g) + \text{T}_2\text{O}(g) \rightarrow \text{NT}_4\text{OT}$$

$$[-\text{CF}_2-\text{CF}_2-]_n + \beta \rightarrow \text{F}_2(g) \text{ and TF}$$

$$\text{F}_2 + \text{T}_2\text{O} \rightarrow \text{TF}(g)$$

$$\text{TF} + \text{SiO}_2 \rightarrow \text{SiF}_4(g) \text{ [or Si}_2\text{F}_6(g)] + \text{T}_2\text{O}$$

$$\text{SiF}_4(g) + 2\text{TF}(g) \rightarrow \text{T}_2\text{SiF}_6$$

$$\text{T}_2\text{SiF}_6 + \text{NT}_4\text{OT} \rightarrow (\text{NT}_4)_2\text{SiF}_6$$

(This material had been observed in an event many years earlier in another facility. Limited documentation on that event.)
In Process Filters

- Particulate matter found on multiple filters and flow orifices within the process facility
- First examination in 10 years of service. No noted performance change in over 10 years of service in many locations.
- Particulate found is same as that on compressor filters, but in less quantity
- Particulate found in various amounts on several other flow orifices. The variability was never fully understood.
Example Flow Orifice

Typical Appearance of a New Flow Orifice

Inlet Side

Inlet  →  Outlet

Inlet Side
Flow Orifice – Plugged

Photomicrograph of Debris
Flow Orifice Material Analysis

X-Ray Spectrum
SEM images of lint free cloth used to collect particles. Particle sizes ½ to 10-20 microns.

X-ray spectrum of particles
Sources of Contaminants

– Compressor poppets, valve packing, valve seats, etc.
  • Teflon and other polymers used in construction
    – Degrade through normal wear
    – Degrade through exposure to tritium
– Materials introduced during maintenance
  • Krytox and Swak (lubricants)
  • Freon or Vertrel (cleaning agents)
  • Dust and dirt from line break activities
3rd Stage Poppet

Poppet Assembly

Glass-Filled Teflon® Seat, approximately 0.08 grams

As-Received

Post-Service (Average 1-2 Year Life)
Restoration and Modifications to Compressors

- New inter-stage filter cartridges installed
- Poppets replaced on 3rd stage
- Filters installed on letdown lines
- New filter cartridges installed on N₂ lines
- Poppet and filter cartridge surveillance initiated based on compressor run time
Contaminant Minimization and Control

• Contaminant Minimization: minimize contaminants introduced into the system
  – Use alternate materials which do not degrade in tritium service
  – Where alternates are not available, minimize use and tritium exposure to materials used

• Contaminant Control
  – Install additional filters
  – Periodically inspect filters
Lessons Learned

• Wear and tear of components happens
• Tritium degradation of materials happens
• Design to protect critical systems
• Implement appropriate protective measures (e.g., filters)
• Probably cannot eliminate particulate; can better control and minimize it
• Consideration should be given to particulate flow, migration and accumulation in the design of the facility
Thank You

Questions?