

TECHNICAL TENSION BETWEEN ACHIEVING PARTICULATE AND MOLECULAR ORGANIC ENVIRONMENTAL CLEANLINESS: DATA FROM ASTROMATERIAL CURATION LABORATORIES.

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Introduction: NASA Johnson Space Center operates clean curation facilities for Apollo lunar, Antarctic meteorite, stratospheric cosmic dust, Stardust comet and Genesis solar wind samples. Each of these collections is curated separately due to unique requirements. The purpose of this abstract is to highlight the technical tensions between providing particulate cleanliness and molecular cleanliness, illustrated using data from curation laboratories. Strict control of three components are required for curating samples cleanly: a clean environment; clean containers and tools that touch samples; and use of non-shedding materials of cleanable chemistry and smooth surface finish. This abstract focuses on environmental cleanliness and the technical tension between achieving particulate and molecular cleanliness. An environment in which a sample is manipulated or stored can be a room, an enclosed glovebox (or robotic isolation chamber) or an individual sample container.

Room Environment: Maintaining a particle-clean room is usually accomplished using HEPA (high efficiency particulate air) or ULPA (ultra low penetration air) filtered air maintained at higher pressure than the less-clean environment. Cleanrooms can be operated by diluting room air with filtered clean air (typically ISO class 6 or greater)[1]. The cleanest rooms are designed with laminar flow to control particle paths (typically ISO class 5 or less). Judicious use of cleanable materials and strict protocols can greatly improve cleanliness. JSC operates several astromaterial curation cleanrooms (Table 1).

Airborne Molecular Contamination Levels in Genesis Laboratory: Molecular contamination levels were measured in Genesis Lab air by exposing organic-free polished silicon wafers to lab air for 24 hours. The weight of organic species (with carbon chain length >7) sticking to a polished silicon wafer after exposure are presented in ng/cm² as yearly averages for 2001 through 2011 in Fig. 1. In general, the amount of airborne molecular contaminants decreased as activity levels decreased (year 2001-2004). Levels increased after repairs from flooding in 2010. Airborne species derived from RTV sealing compounds, used in the 54 ULPA fan filter units, and plasticizers are measured as a variety of cyclic siloxanes. Table 2 gives an example of other species detected. Observation of caprolactam is noteworthy. This nitrogen-containing species is an

offgas product of nylon bags used to protect samples and supplies from particulate contamination.

Glovebox and Storage Cabinet Environments: Glovebox and nitrogen storage cabinets are purged continually with clean nitrogen. Thus, cleanliness depends upon an enclosure which is made of cleanable, low offgassing materials and of cleanable design and also upon pure purge gas. Enclosures using point-of-use (POU) filter/purifiers provide pure purge gas to samples. Genesis storage enclosure POU purifier/filters supply nitrogen with < 1 ppb H₂O, O₂, CO₂, CO and retain particles > 3 nm.

Gloveboxes are often used with inert purge gas to prevent unwanted chemical reactions to samples. However, any sample or hardware manipulation within a glovebox or robotic enclosure will generate particles abraded from the samples and tools inside the enclosure (particularly for rocky fines). Often a glovebox is assumed to be particle-clean. In fact, unless the particles are actively removed, this is not so.

If standard HEPA particle filtration technology is incorporated into the glovebox to remove particles, it is likely molecular contaminants are increased (mainly because filter sealants offgas cyclic siloxanes and other products).

Organic contamination was measured inside several curation stainless steel gloveboxes which are purged with pure nitrogen. These gloveboxes do not have active particle removal. Molecular contamination was measured as species adsorbed on a polished silicon wafer (witness plate) as described for Genesis laboratory air. An example of molecular species observed in one glovebox is shown in Table 3. Sources of organic contamination include offgassing from gloves and plastic sample bags. Offgas products from two glove materials are provided in Table 4.

Heat sealed bags are commonly used to protect samples and tools from particulate contamination; however, heat sealing produces molecular contaminants which can be absorbed on samples (Table 5). A combined heat sealing test of Teflon®, nylon, and polyethylene inside of nitrogen-filled glove box produced caprolactam (1300 ng/cm² from nylon), and lesser amounts of N,N-dibutylformamide, dibutyl phthalate, cyclo(Me₂SiO)₈, N,N-dibutylacetamide.

Summary: Techniques used to reduce particulate contamination or control atmospheric reactions may

increase molecular contamination. Some examples are given for planning purposes.

Table 1. JSC sample curation cleanrooms “at rest”.

| ISO Class | Collection | Air Flow |
|-----------|------------------------|--------------------------|
| 4 | Genesis solar wind | Laminar, vertical flow |
| 5 | Cosmic Dust, Stardust | Laminar, horizontal flow |
| 5 | Stardust | Dilution |
| 6 | Lunar | HEPA filtered supply |
| 6 | Space-exposed hardware | Dilution |
| 6-7 | Meteorite | HEPA filtered supply |

Figure 1. Genesis Laboratory molecular contamination levels 2000-2011. Vertical axis is ng organics/cm².

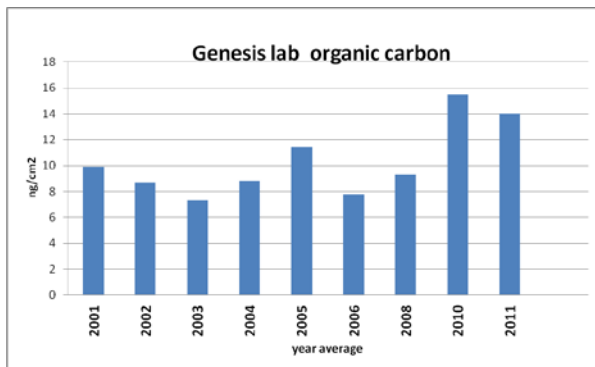


Table 2. Example of organics detected in Genesis lab air. These are semiquantitated results with detection limit 0.1 ng/cm² (TD-GC-MS, Balazs 2003).

| Species | ng/cm ² | Use |
|-----------------------------|--------------------|---------------------------------|
| 2-(2-butoxy ethoxy) ethanol | 0.5 | Solvent used in paint, coatings |
| Di-isobutyl phthalate | 0.4 | Plasticizer |
| TXIB | 0.4 | Plasticizer |

Table 3. Example of molecular contaminants in a glovebox gas as measured by witness wafer. Semi-quantitated results with detection limit of <0.1 ng/cm² (TD-GC-MS, Balazs, 2000).

| Species | ng/cm ² | Use |
|-----------------------|--------------------|---|
| TXIB | 0.2 | Plasticizer |
| N,N-dibutyl formamide | 1 | Unknown source. Species detected in enclosures containing tools & bags and gloved with neoprene |
| Diethylphthalate | 0.2 | Plasticizer |

Table 4. Offgas products from glovebox glove materials (GC-MS, JSC, 2000)

| Glove Material | Species |
|----------------|--|
| Neoprene | Carbonyl sulfide, propanone, acetaldehyde, butanal |
| Viton | Carbonyl sulfide, methanol, carbon disulfide, acetaldehyde |

Table 5. Gas extraction at temperature of melting, DSC, TX/GC/MS (JSC 1997)

| BagMaterial | Melting T, C | Species Offgassed |
|--------------|--------------|--|
| Teflon | 268 | none |
| Nylon | 221 | Caprolactam, 1-butoxy-2-propanol |
| Polyethylene | 110 | 1-pentene, pentane, heptane, 3-methylene-heptane |

References:

- [1] ISO-14644-1, Class of Air Cleanliness, Institute of environmental Sciences and Technology
- [2] Balazs Analytical Services, Fremont, CA.