Contamination Control and Knowledge

During Construction of New Curation Facilities at NASA Johnson Space Center.

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ASTROMATERIALS RESEARCH AND EXPLORATION SCIENCE
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Introduction

Beginning in early 2019, NASA Johnson Space Center (JSC) Astromaterials Curation Office will commence construction of:

- The curation facility for NASA's OSIRIS-REx returned material,
- A curation facility for a subset of JAXA's Hayabusa2 returned material,
- Advanced curation ISO 7 laboratories, and
- Precision cleaning ISO 5/6 cleanroom and advanced cleaning ISO 6 laboratories.

The ultimate goal of curation is to ensure all samples remain preserved as much as possible in a pristine state to enable science investigations in the indefinite future. In that context, Contamination Control and Knowledge (CCK) plays an important role. To face the upcoming challenges of organic-rich samples, the Curation Office is developing a comprehensive plan to monitor and understand contamination and its sources in curation cleanrooms. CCK serves three goals:

1) Make sure that it is possible to keep working in the curation laboratories, or decide when it is necessary to secure the samples in storage;
2) Establish a list of all contaminants found even temporarily in existing ARES cleanrooms that might impact future scientific discoveries, and
3) Gather best practices for future construction work.

Why is Contamination Control and Knowledge important?

Contamination Control and Knowledge is a mandatory part of any clean process.
A cleanroom is not enough to ensure cleanliness, as contaminants come from multiple sources (see figure 1) and it can occur in various forms:

- particulate,
- organic,
- abiotic or biotic,
- molecular, etc.

Even though cleanrooms are kept at positive pressure in accordance to international standard ISO 14644-4, the immediate surrounding environment can have an impact on them, by eddies against the airflow, and ingress of staff [1].
A CCK plan must use both qualitative and quantitative methods. Quantitative methods are usually quicker and will inform on the usability of the cleanroom (e.g airborne particle load). Qualitative methods identify specific contaminants, and can help find the source of contamination.

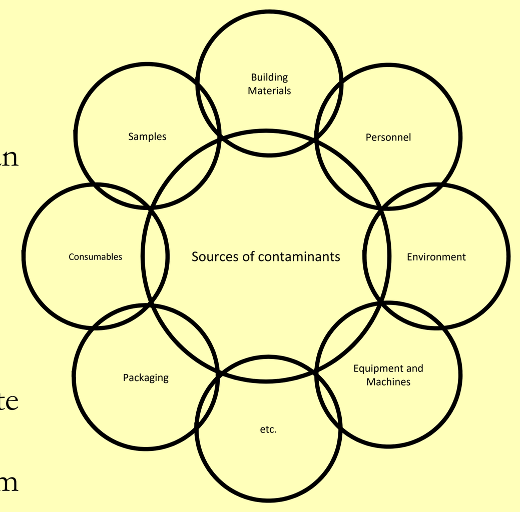

Figure 1: Sources of contamination in a cleanroom.

Goal 1: Is the cleanroom clean enough?

Astromaterials are weekly or even daily taken out of storage and handled in curation cleanrooms. Even though they are usually kept in inert gas environment, it is important to make sure the cleanrooms are functioning properly.
Cleanrooms at JSC are routinely monitored for

- Quantitative airborne particulate load (>0.3μm),
- Room to-room differential pressure,
- Temperature and humidity, and
- Air changes per hour.

For ISO 5 and above, handheld particle counting instruments are typically set-up weekly for particle channels at 0.3, 0.5 (see figure 2), 0.7, 1.0, 5.0, and 10.0 μm. A full ISO audit is conducted annually or bi-annually.
Curation facilities surfaces (floors, walls, etc.) are being routinely cleaned, and work stations are frequently wiped when in use.

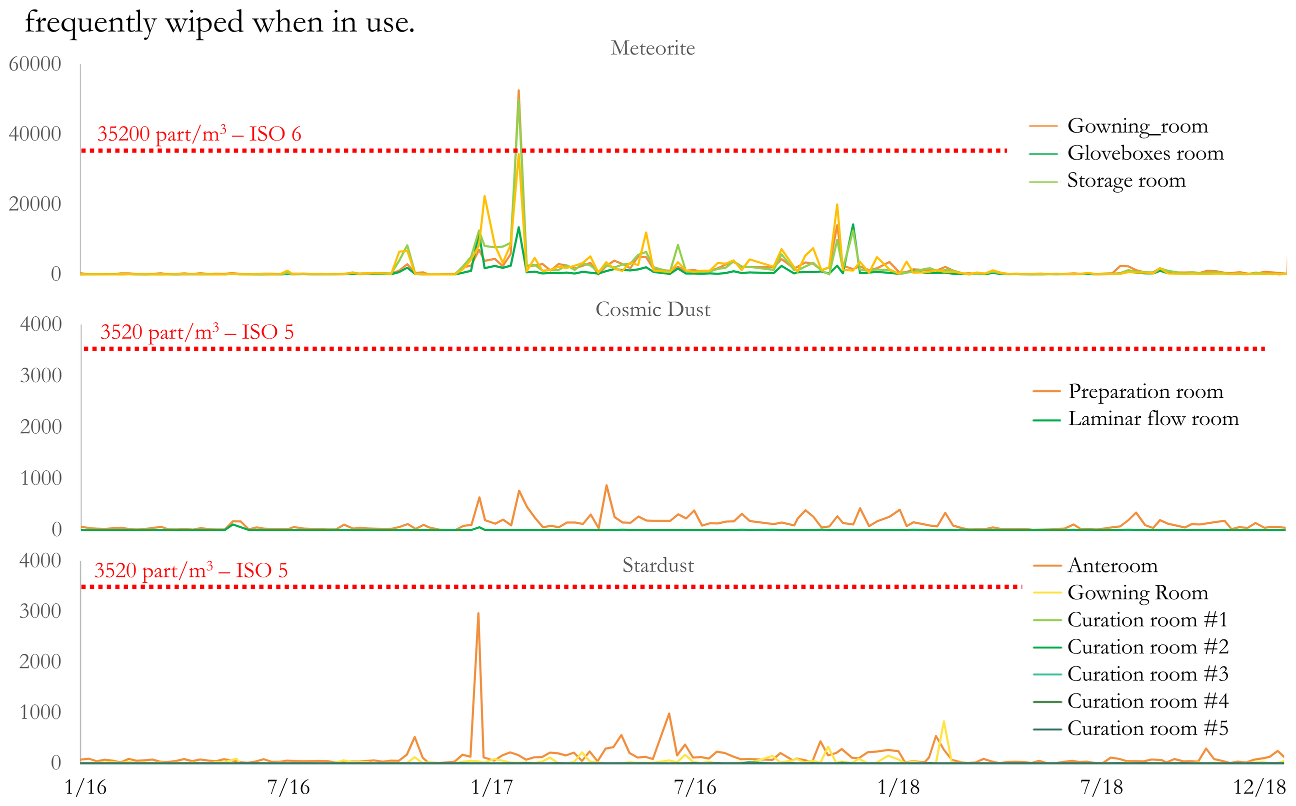

Figure 2: >0.5 μm airborne particle load in impacted cleanrooms, from January 2016 to December 2018. Plots are color coded from less clean (orange and yellow) to cleanest areas (green).

Goal 2: What is and has been in the cleanroom?

In preparation for the more stringent organic contamination requirements for OSIRIS-REx [2] and Hayabusa2, the Curation Office conducted in 2014 an organic contamination baseline study [3] that provided a long-term historical perspective on curation cleanroom affected by organics.

The CCK plan aims at expending this knowledge, and at getting more comprehensive results. To that effect, it encompasses various methods to quantify organic and inorganic molecular contamination, and to characterize particles. Even though microbial contamination is being studied in several cleanrooms [4], it is not within the scope of this poster.

Airborne Molecular Contamination (AMC). AMC occurs when contaminants are in molecular form without forming particle aggregates in the airflow. Contaminants can be in the form of trace inorganic, organic and biological species. Even though AMCs should be evacuated out of the cleanroom by the airflow, they can interact with surfaces, and deposit to become Surface Molecular Contamination (SMC), posing a threat to the integrity of samples.

For short-term events, AMC tests are performed using 200mm silicon wafers (24 to 72h exposure) and air sampling tubes (6h pumping) provided by Balazs NanoAnalysis, to measure molecular organics (AMC-MC) and molecular metals (AMC-MM). Wafers for AMC-MM are analyzed for 35 elements by Vapor Phase decomposition Inductively Coupled Plasma Mass Spectrometry (VPD ICP-MS). Tubes and wafers for organics are analyzed by Thermal desorption-gas chromatography mass spectrometry (TD-GC-MS). A baseline for background contamination of at-rest cleanrooms has been acquired in early January 2019. Figure 3 shows the sets.

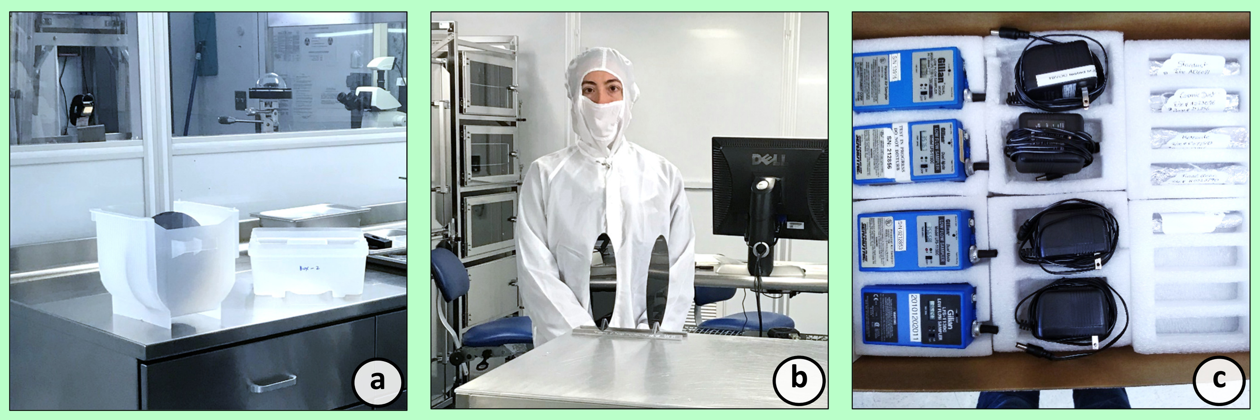

Figure 3: Setup for Balazs NanoAnalysis during baseline contamination acquisition (January 2019). (a) shows the wafer exposed for inorganic analysis in Stardust cleanroom. (b) shows the two wafers on an aluminum stand for organics analysis. (c) is the air sampling setup, with the pumps on the left, and the absorbing tubes wrapped on the right.

For a long-term accumulation, we use the contamination knowledge witness plates designed for CCK during ATLO of OSIRIS-REx. Plates are equipped with four precision cleaned silicon wafers and four high-purity baked-out aluminum foils. Wafers are used to characterize particles and are described in the next section. Al foils are analyzed at NASA Goddard Space Center by solvent extraction and pyrolysis GC-MS.

Airborne Particles characterization. Silicon wafers are imaged and analyzed in-house by SEM/EDX for size, texture, and bulk elemental abundances of particles.

Figure 4 shows the contamination knowledge plates assembled and ready to be deployed, and individually the silicon wafers (approximately 13x13mm) and Al foils (approximately 15x46mm).

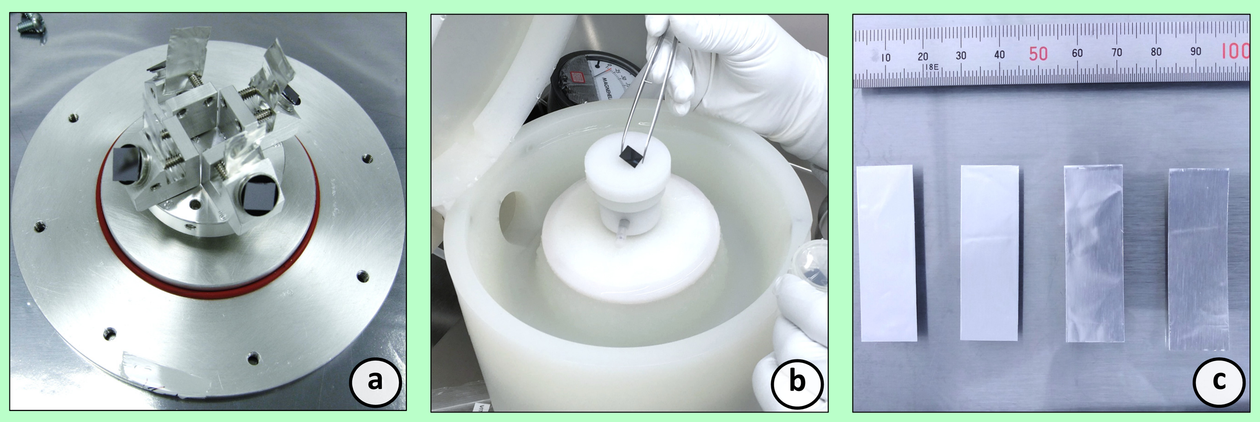

Figure 4: (a) Contamination knowledge plates equipped of precision cleaned silicon wafers mounted on SEM sample holders to collect particles and high-purity aluminum foils for organic NVR analysis (courtesy of K. Righter OSIRIS-REx curation lead). (b) is the silicon wafers being precision cleaning in Genesis ISO 4 cleanroom. (c) Al foils cut to 15x46mm.

Goal 3: Best practice and mitigating contamination

Analysis of AMC and particles as described in Goal 2 will enable identifying unexpected contaminants that could pose unacceptable science risks, and mitigating them if possible. Short-term analysis will allow for a faster response. Even though materials to be used in the new cleanrooms have been chosen for their low outgassing properties [5], materials being used for the construction around the cleanrooms are not controlled. If the source of an unexpected contaminant is unclear, it will be necessary to track it. A protocol under consideration would be to outgas samples of building materials in an emission chamber, and to collect AMCs in solvent tubes for analysis through desorption in a GC-MS [6].

If it is not possible to cancel the production of the contamination (mandatory materials for safety, materials without a valid alternative, etc.), the pathway to the cleanroom should be investigated. Mitigation possibilities would be to revisit staff behavior and gowning apparel, or to clean the surfaces of the cleanroom more often or with more efficient procedures, to remove as much contaminant as possible from the cleanroom. To investigate the efficiency of contamination removal, we are developing surface contamination analyses. We envision two methods of studying surface contamination. First, collecting particles on the surface using carbon tape, and analyzing them using an SEM. Second, wiping or rinsing the surface using an appropriate solvent, which will be then analyzed for organics or trace metals.

Baseline 2019

As described in Goal 2, a baseline has been acquired in the three critical curation facilities in January 2019. Below is an overview of the most important compounds found in the cleanrooms.

Compounds in air (ng/L)	Stardust	Cosmic Dust	Meteorite
Low Boilers C7 - C10	8.2	17.3	19.3
Medium Boilers >C10 - C20	20	39.8	57.5
High Boilers >C20	*	*	*
Sum >=C7	28.2	57.1	76.8
Benzaldehyde	0.9	0.8	0.8
Butoxy ethanol	0	0.2	0.5
C10-C14 Hydrocarbon	0.4	1.5	1.5
C12 Hydrocarbon + Naphthalene	0.2	0.4	0.5
C3 Benzene + Cyclo(Me2SiO)4	*	1.2	1.1
C3-C4 Benzene	0.1	1.5	2
C6-C9 Hydrocarbons	1.8	3.5	3.9
Cyclo(Me2SiO)3	0.3	0.4	0.5
Cyclo(Me2SiO)4	0.6	*	*
Cyclo(Me2SiO)5	13.2	25	27
Cyclo(Me2SiO)6	0.3	0.7	0.7
Ethyl hexanal	*	0.5	*
Limonene	0.4	2.2	2.9
m,p-Xylene	0.3	0.5	0.5
Nonanal	*	0.4	0.4
Tetrachloroethylene	0.4	0.8	0.3
Toluene	0.8	1.2	1.3
TXIB	0.4	3.3	4.3

Figure 5: Main organic compounds from 6h air sampling, by TD-GC-MS. Detection Limit is 0.1 ng/L. *= the analyte was not found at or about the detection limit.

Organic contamination is measured directly in the air with an adsorbent tube, and indirectly by deposition on wafers. Air sampling is sensitive for the whole range of hydrocarbons (C7 to C28), while wafers won't show light hydrocarbons (below C12) as they tend to not deposit on surfaces.

Overall, the three cleanrooms are low in organic contamination. Meteorite shows a 53% lower contamination level compared to the 1998 study [3]. This shows that it is possible to mitigate contaminants by changing procedures.
Hydrocarbons, benzene, toluene and xylenes are common contaminants of Houston air, entering the laboratories through HVAC. Other compounds are probably from outgassing flooring, paints and wet wipes, as well as gloves, bags and other plastics. TXIB is a plasticizer.

Compounds on wafers (ng/cm²)	Stardust	Cosmic Dust	Meteorite
Low Boilers C7 - C10	1.5	3.5	1.6
Medium Boilers >C10 - C20	11	17.2	19.1
High Boilers >C20	2.6	1	3.4
Sum >=C7	15.1	21.7	24.1
C6-C9 Hydrocarbons	0.5	1.7	0.6
Cyclohexanol, 3,5-dimethyl- + Unk.(m/z:97)	1.1	0.8	0.1
Dibutyl phthalate	0.5	*	3.9
Diisobutyl phthalate	0.3	0.4	0.4
Mono(2-ethylhexyl)phthalate	*	*	0.5
Phthalic anhydride	0.2	0.7	0.2
Siloxane	*	3	0.6
TXIB	3.7	4.2	4.7
Unk.(m/z:41,55,67,77,97,105,110)	*	0.7	0.3
Unk.(m/z:43,55,71,77,105,193,277)	*	0.3	0.5
Unk.(m/z:43,56,71,105,155,193,207,277)	*	2.5	*
Unk.(m/z:43,56,71,77,105,123,193,277)	*	*	3.5

Figure 6: Main organic compounds from wafers exposed for 72h, by TD-GC-MS, SEMI MF 1982-1103 Method-B. Detection Limit is 0.1 ng/cm². *= the analyte was not found at or about the detection limit.

Element (1E10 atoms/cm²)	DL	Stardust	Cosmic Dust	Meteorite
Barium (Ba)	0.001	*	*	0.002
Boron (B)	0.5	86	76	130
Calcium (Ca)	0.1	0.2		0.4
Copper (Cu)	0.01	*	*	0.01
Iron (Fe)	0.05	*	*	0.17
Magnesium (Mg)	0.05	*	*	0.36
Potassium (K)	0.05	0.12		0.15
Sodium (Na)	0.05	0.45	0.13	0.29
Tin (Sn)	0.005	0.008	*	*
Zinc (Zn)	0.05	0.08	0.09	0.05

Figure 7: Main inorganic compounds from wafers exposed for 72h, by VDP-ICP-MS. DL= Detection Limit. *= the analyte was not found at or about the detection limit

Inorganic contamination is kept at a minimum, especially for both ISO 5 cleanrooms (Stardust and Cosmic Dust).
Calcium, Potassium and Sodium are from human contamination. Boron is from HEPA filters. Tin and Zinc are usually from electrical systems and soldering.

Existing and future curation facilities

Lunar	Meteorite	Cosmic Dust	Genesis	Stardust	Hayabusa	Hayabusa2	OSIRIS-REx	Precision Cleaning facilities	Adv. Curation facilities	Adv. Cleaning Facilities	Moon, Comet, Phobos, Mars
1969	1977	1981	2004	2006	2012	2020	2023	2020	2020	2020	
Lunar rocks and soils	Antarctic meteorites	Cosmic Dust from Earth's stratosphere	Solar wind samples	Cometary and Interstellar particles	Asteroid Itokawa (subset)	Asteroid Ryugu (subset)	Asteroid Bennu				
ISO 6	ISO 6	ISO 5	ISO 4	ISO 5	ISO 5	ISO 5	ISO 5	ISO 5/6	ISO 6/7	ISO 6	


Figure 8: the Curation Office at JSC is in charge of seven collections, and a number of support laboratories. Cleanrooms impacted by the construction are in orange. The blue scale shows existing laboratories (white), upcoming facilities for planned return missions and new capacities (light blue), and future missions (dark blue).

Conclusion and future work

The Contamination Control and Knowledge plan encompasses a range of tests to reach all three goals. It should enable the Curation Office to more accurately track contamination, its sources, and its vectors. Most of the protocols are developed in-house, using the expertise and the high-resolution instruments available at ARES, and leveraging outside resources when necessary and appropriate. Understanding these processes should lead to an reevaluation of the current contamination mitigation protocols, e.g. housekeeping, cleanroom gowning apparel, gloves and staff behavior in the Curation Office.

The baseline acquired with Balazs NanoAnalysis sets shows that our clean rooms have low levels on contamination, whether organic or inorganic, and that there is no unknown contaminant sources.

References: [1] IEST Recommended Practice: IEST-RP-CC003.3. [2] Dworkin et al. (2018) Space Sci Rev, 214: 19. [3] Calaway et al. (2014). NASA/TP-2014-217393. [4] Davis et al. (2019) *LPSC L:2280*. [5] Calaway et al. (2019) *LPSC L*:1448. [6] Kataoka H. et al. (2012) *InTech*: 161-184.

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