Exploring Space through Sample Return Missions:

Planetary Protection and Contamination Control and Knowledge

Aurore Hutzler

Horizon 2061 – 11-13 September 2019 - Toulouse

Geologist by training in analytical geochemistry

Ph.D. in France on meteorite terrestrial ages

Post-doc in Austria on the ideal curation facility for astromaterials

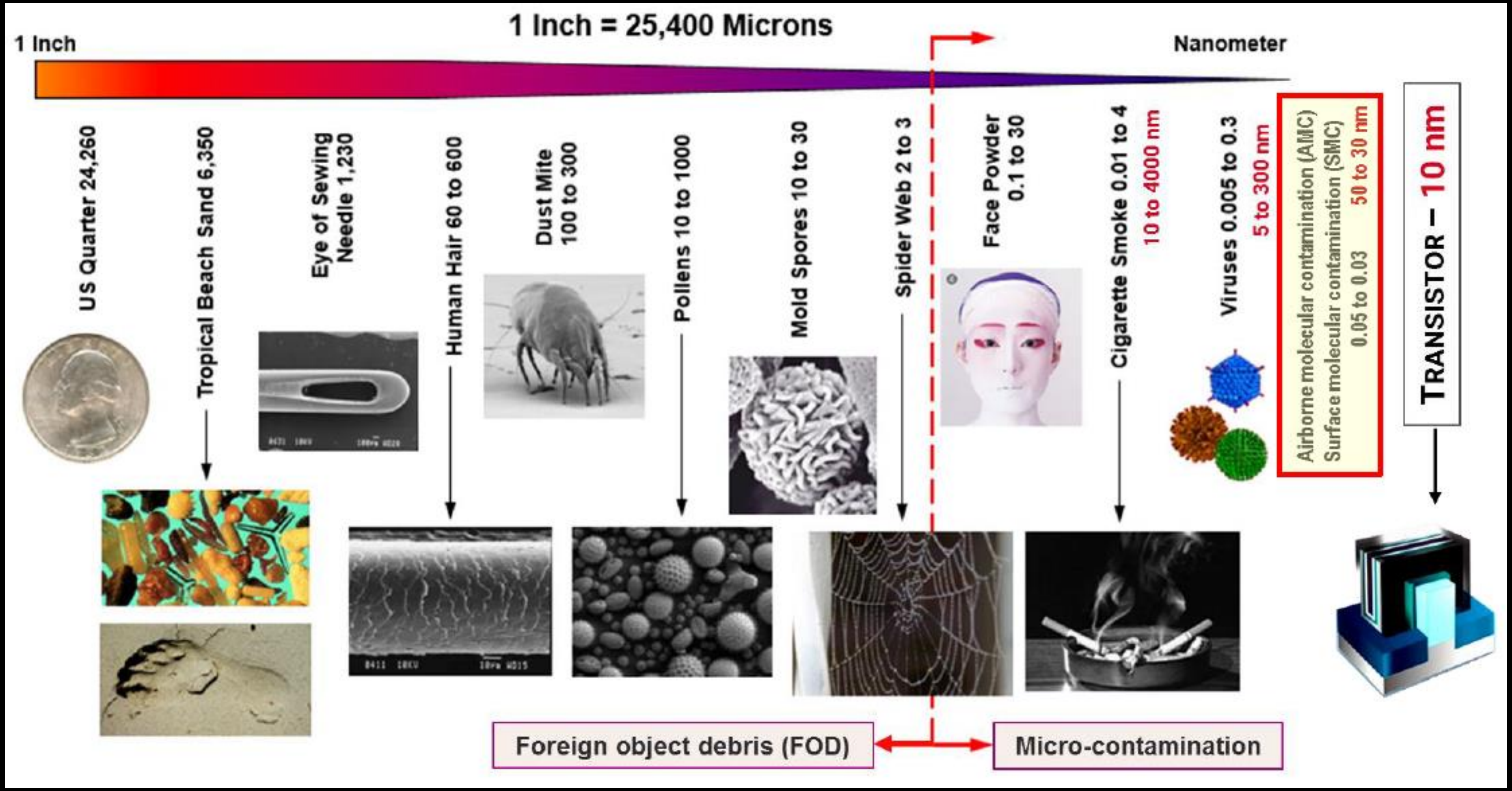
Now Visiting Scientist at the Curation Office, NASA JSC (Houston, Texas) on contamination control and knowledge and material suitability.

Outline

- What is contamination?
- Why do we need to worry about contamination?
- Where does it come from?
- How to deal with contamination during a mission lifetime?
- What we currently do at the Curation Office
  - The example of organic contamination
  - The example of human exploration of Mars

Setting the stage

A **contaminant** is defined as a substance or compound that will affect the pristine state of a sample.



Setting the stage

Contamination Control (CC) is the series of actions to lower the level of contamination that could affect samples.

Contamination Knowledge (CK) is a series of actions to monitor and record levels of contamination that could affect samples.

Planetary Protection (PP) “is the term given to the practice of protecting solar system bodies (i.e., planets, moons, comets, and asteroids) from contamination by Earth life, and protecting Earth from possible life forms that may be returned from other solar system bodies” (NASA PP website): in short, it is a concern about biological contamination.

Why do we limit contamination?

In the past 7 years, NASA Curation Office had requests to do the following:

- **Stable isotope studies** – H, He, Li, C, N, O, Ne, Mg, Si, Cl, Ar, Ca, Ti, V, Fe, Ni, Cu, Zn, Se, Kr, Rb, Sr, Mo, Ru, Cd, In, Sn, Xe, Ba, Hf, W, Os (32)
- **Radiogenic isotope studies** – K-Ca, K-Ar, Ar-Ar, Rb-Sr, Cs-Ba, Sm-Nd, Sm-Nd, Lu-Hf, Hf-W, Re-Os, Pb-Pb, Th-Pb, U-Pb, U-Th/He, U-Th disequil (15)
- **Organics** – amino acids, soluble organics, perchlorates, life toxicity (ppt)
- Highly and moderately **siderophile elements** – low ppb levels
- **Volatile elements** – ppm level measurements of H, OH, H_2O, F, Cl, Br

ELSEVIER
Geochimica et Cosmochimica Acta 172 (2016) 357–369
Acta
www.elsevier.com/locate/gca

The origin of amino acids in lunar regolith samples

Jamie E. Elsila[a,*], Michael P. Callahan[a,1], Jason P. Dworkin[a], Daniel P. Glavin[a], Hannah L. McLain[a,b], Sarah K. Noble[a,2], Everett K. Gibson Jr.[c]

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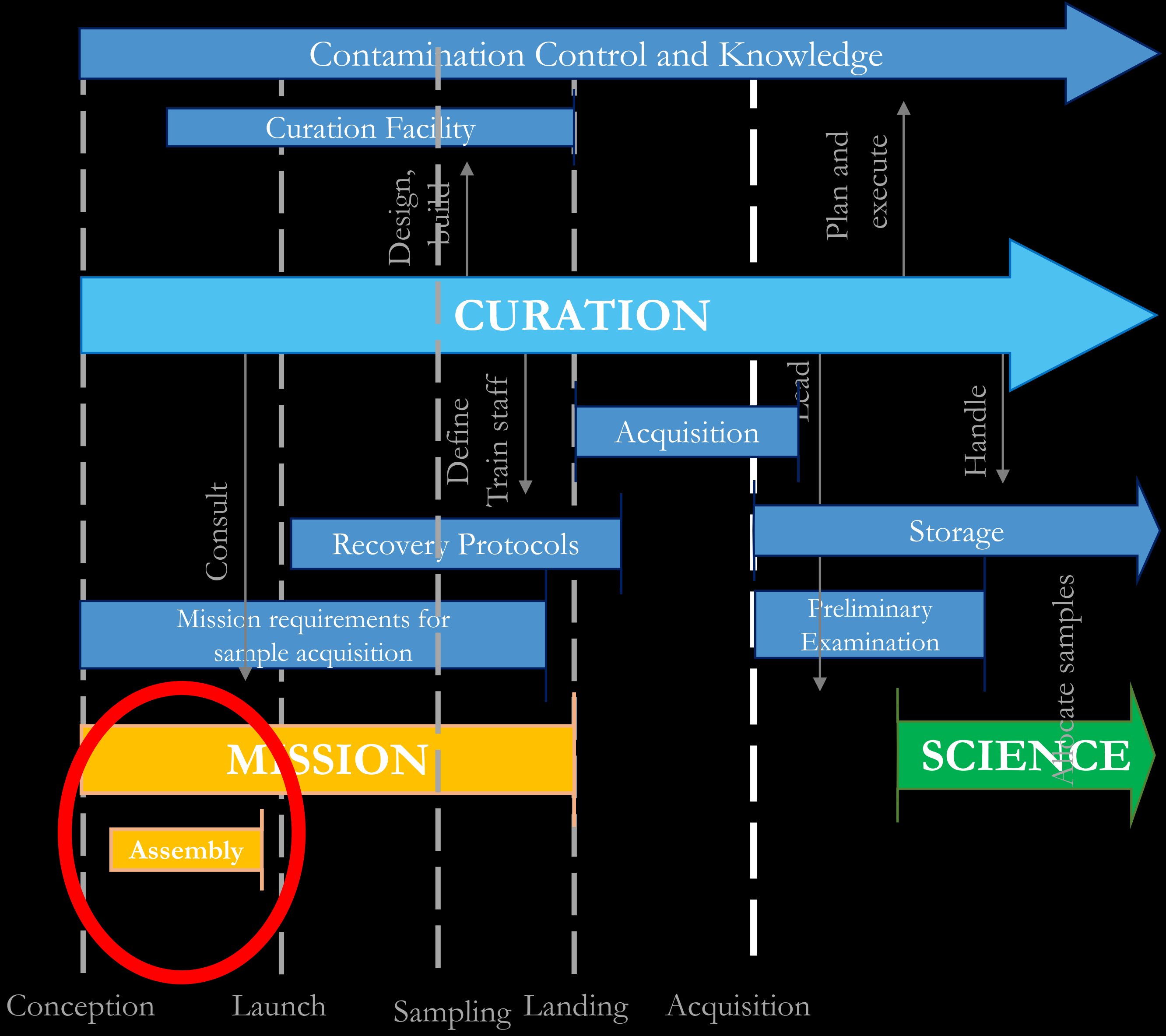
Proceedings of Lunar and Planetary Science, Volume 22, pp. 449-458
Lunar and Planetary Institute, Houston, 1992

Xylan: A Potential Contaminant for Lunar Samples and Antarctic Meteorites

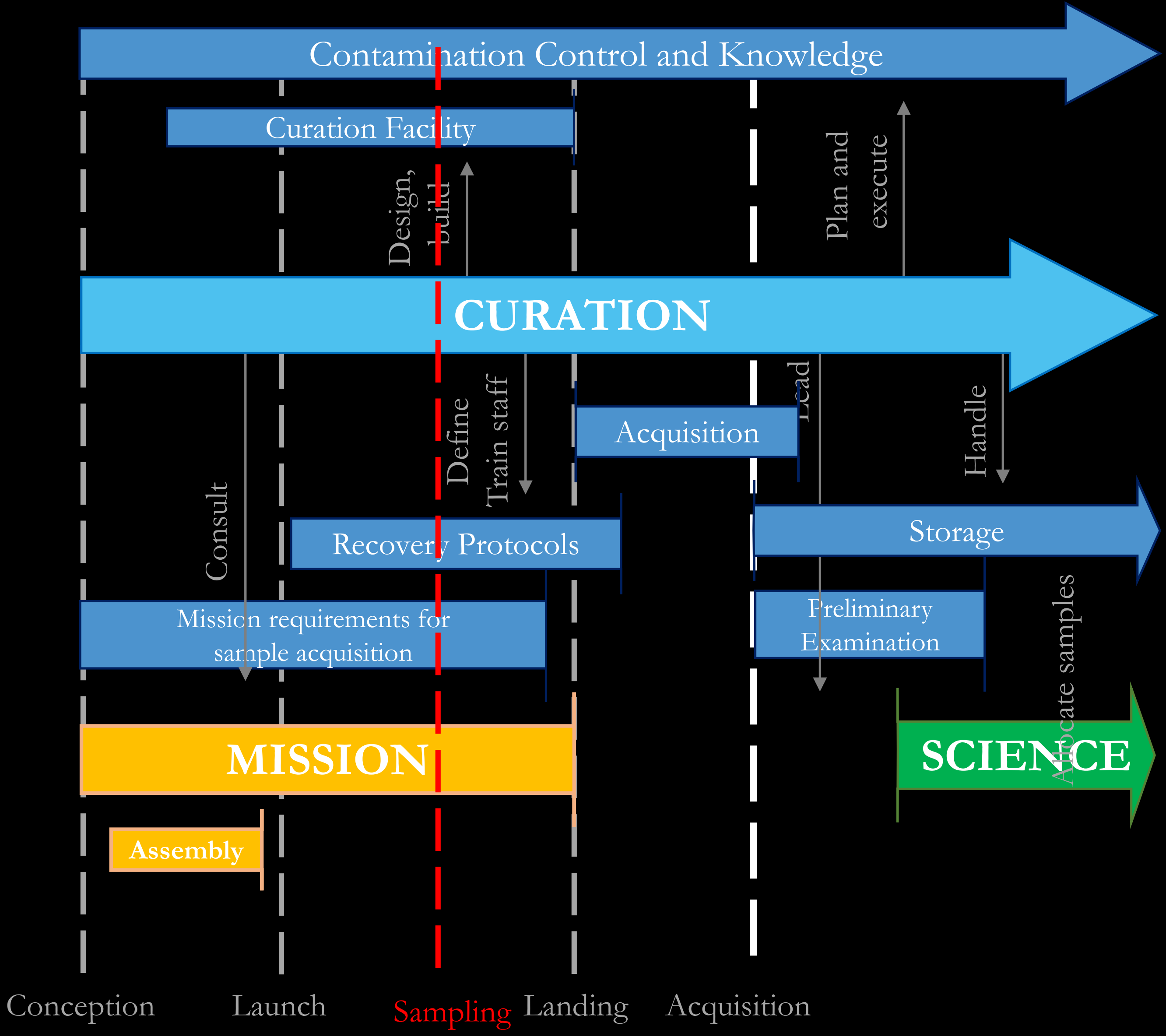
I. P. Wright*, S. S. Russell*, S. R. Boyd*,†, C. Meyer‡, and C. T. Pillinger*

Sources of contamination for SRM



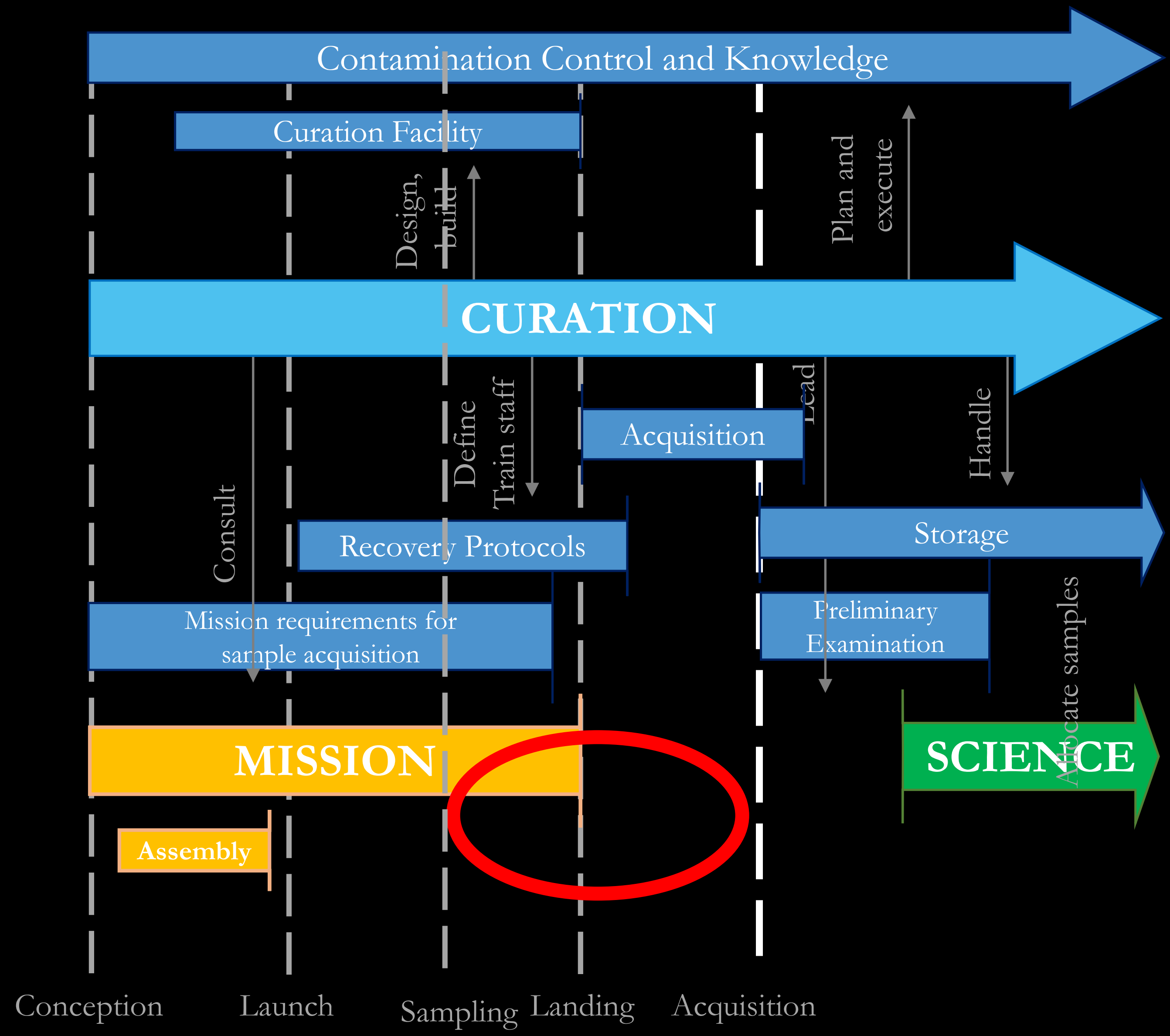
During spacecraft assembly

Sources of contamination for SRM



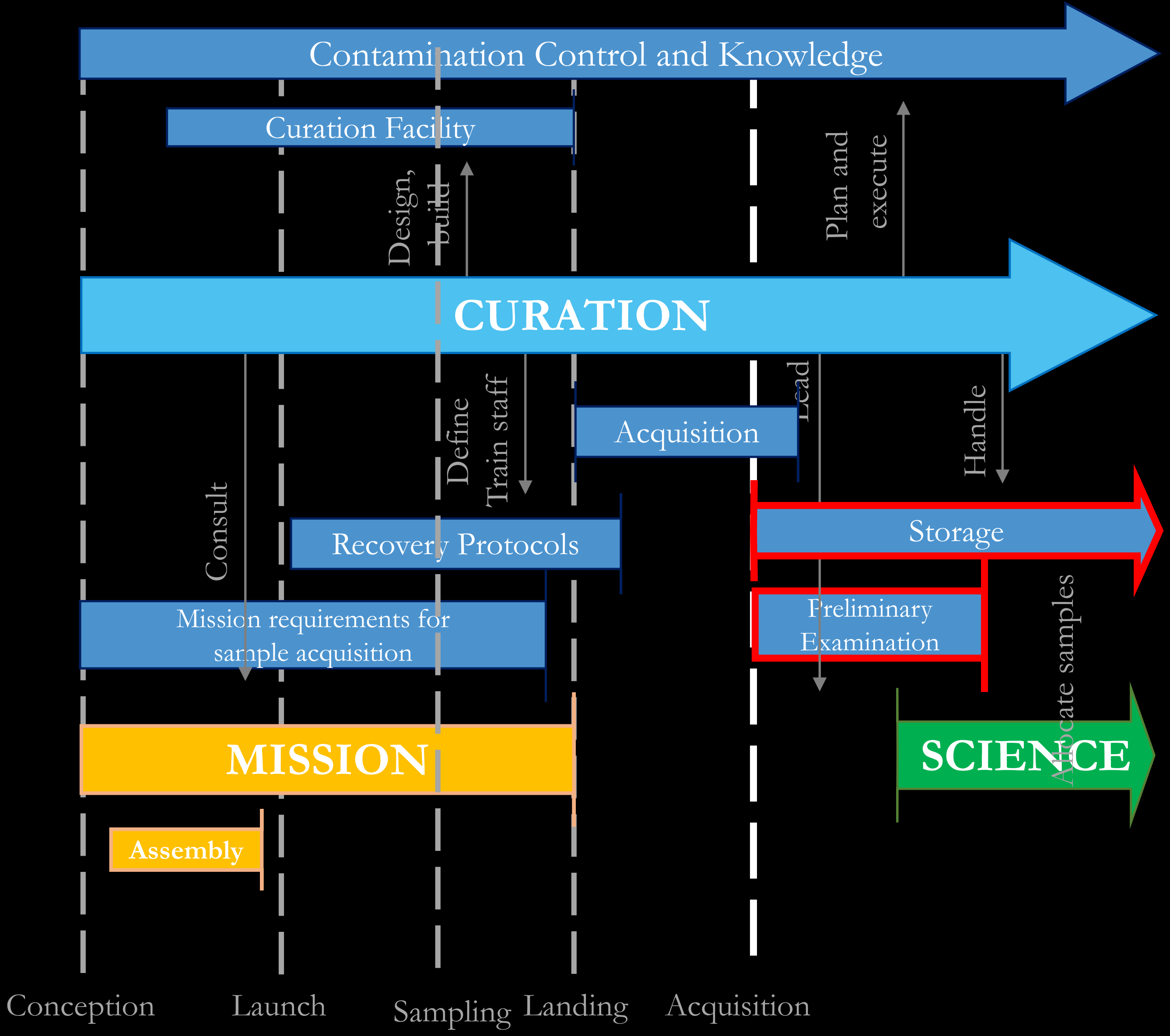
During sampling

Sources of contamination for SRM

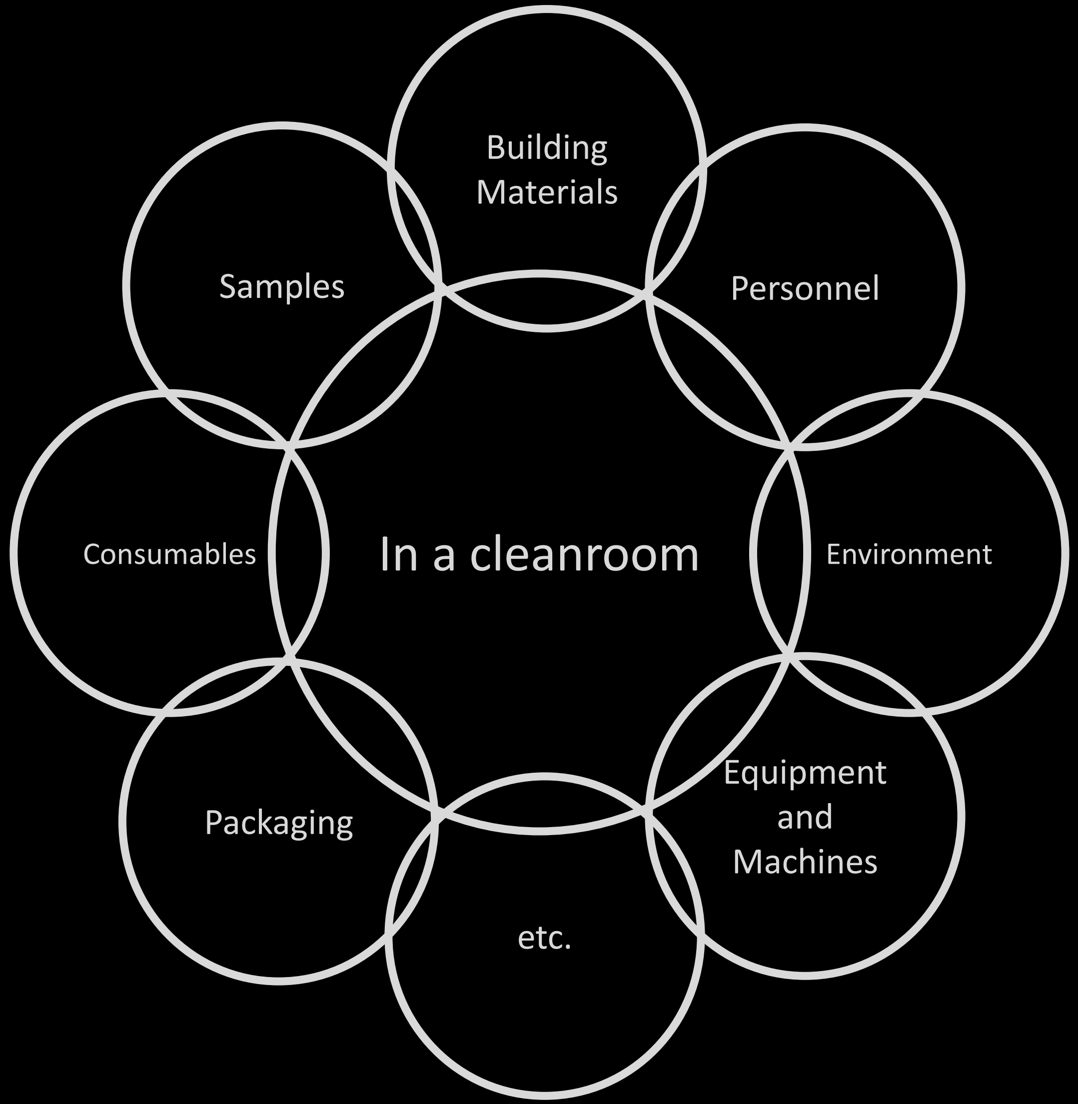


During journey and landing

Sources of contamination for SRM



During storage and handling



How to: before acquisition

Requirements defined and adapted to samples; spacecraft designed accordingly (CC).

Spacecraft assembled and tested in cleanrooms.

Need for collection and curation of A LOT of CK samples starting during spacecraft assembly (Mars 2020).

Recovery procedures even for non-nominal landing.

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Need for collection and curation of A LOT of CK samples starting during spacecraft assembly (Mars 2020).

Recovery procedures even for non-nominal landing.

How to: after landing (contamination control)

All curation activities are in cleanrooms (ISO 7 to ISO 4) with up-to-date gowning and housekeeping protocols.

Limited number of materials in cleanrooms and in contact of the samples (glass, stainless steel, aluminium, specific plastics).

Tools are precision cleaned (Level 50, IEST-STD-CC1246E).

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1.5 Preferred Products

The following list of materials are preferred in the construction of the cleanroom and laboratory. All construction materials shall be submitted to the NASA curator for review. Any deviation from the following list shall be at the sole discretion of the NASA curator.

a. Metals:

1. Stainless Steel 300 series: preferably grade 304, 304L, 316, or 316L; passivated and #4 Finish or better; or electropolished.
2. Aluminum with Clear Anodized: MIL-A-8625 Type 2 or type 3. Preferably grade: 6061-T6 and 6063-T6.

1100
3003
5005

1.4 Prohibited Materials

The following list of materials is prohibited in the construction of the cleanroom and laboratory. See Finish Schedule in the Construction Documents for information on which rooms in Project Scope are subject to these material restrictions and review. For all applicable rooms, all construction materials shall be submitted to the NASA Curator for review. Any deviation from the following list shall be at the sole discretion of the Curator.

a. Particulate shedding materials are prohibited and should be avoided to reduce cleanroom particulate load. All materials should meet ISO Class Cleanroom standards per designated area.

1. Materials that might erode, crack, or flake.
2. Materials with thin films that might erode, crack, or flake.
3. Most foams.

How to: after landing (contamination control)

All curation activities are in cleanrooms (ISO 7 to ISO 4) with up-to-date gowning and housekeeping protocols.

Limited number of materials in cleanrooms and in contact of the samples (glass, stainless steel, aluminium, specific plastics).

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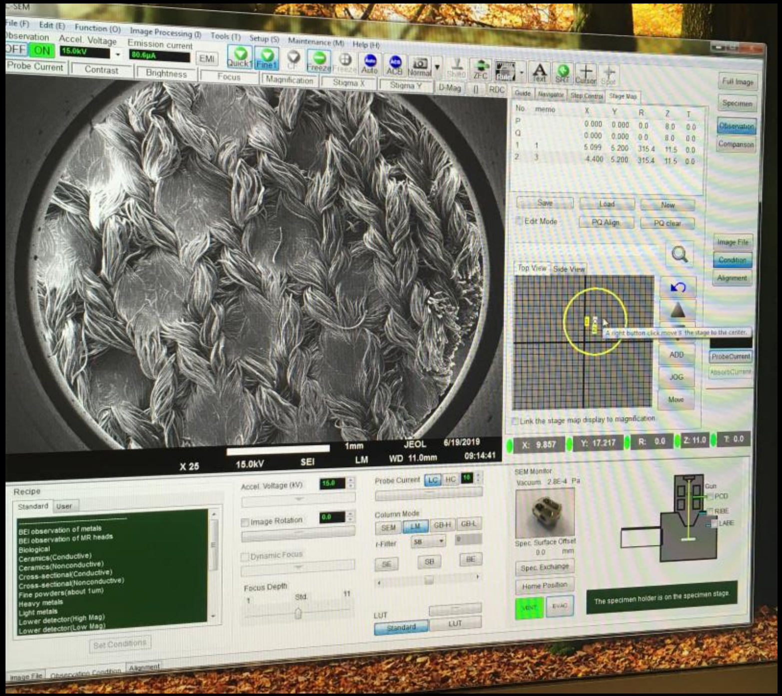
How to: after landing (contamination knowledge)

Routine CK measurements

- Weekly particle counts in labs.
- Daily monitoring of the UPW system.
- The O_2 + H_2O in the lunar cabinets is measured 4x/hour
- Elemental and isotopic composition of each batch of N_2 is measured

Periodically

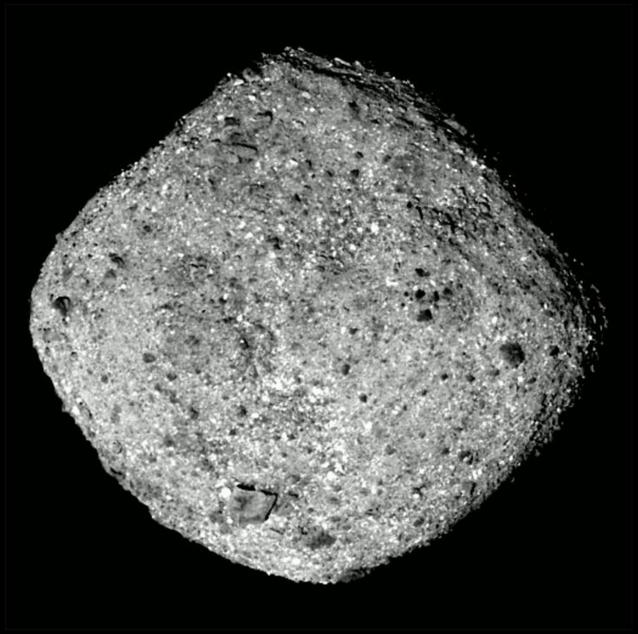
- composition of particles when the cabinets are cleaned.
- total organic hydrocarbons in the labs.

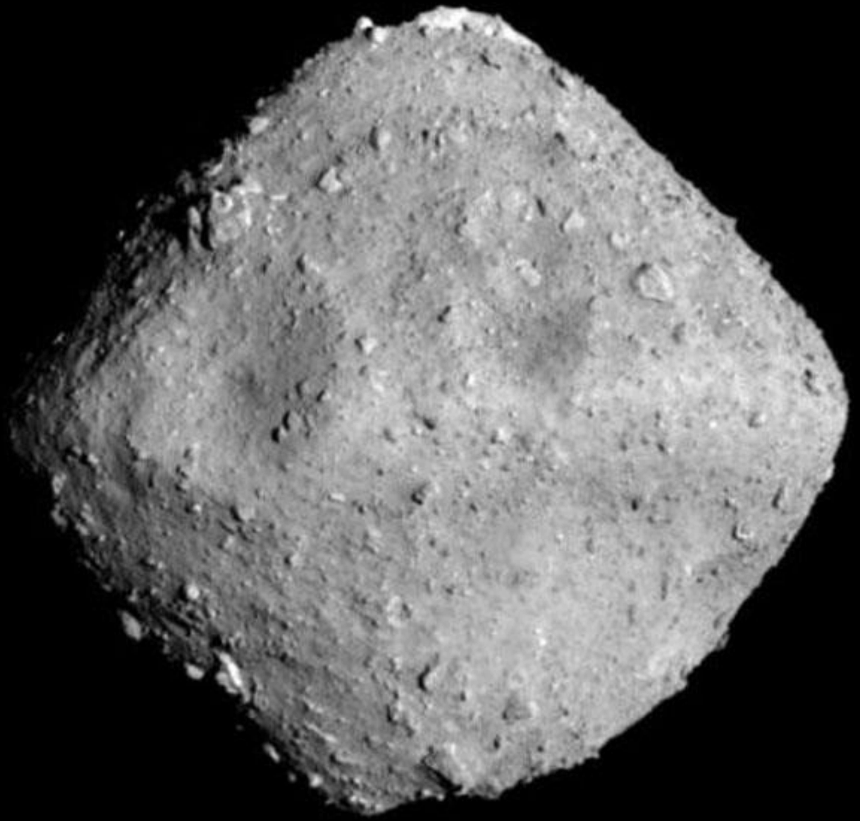


Contamination Knowledge is time sensitive
More is better!

Moving forward: organic contamination

No technical requirements for current collections, except airborne particle load.





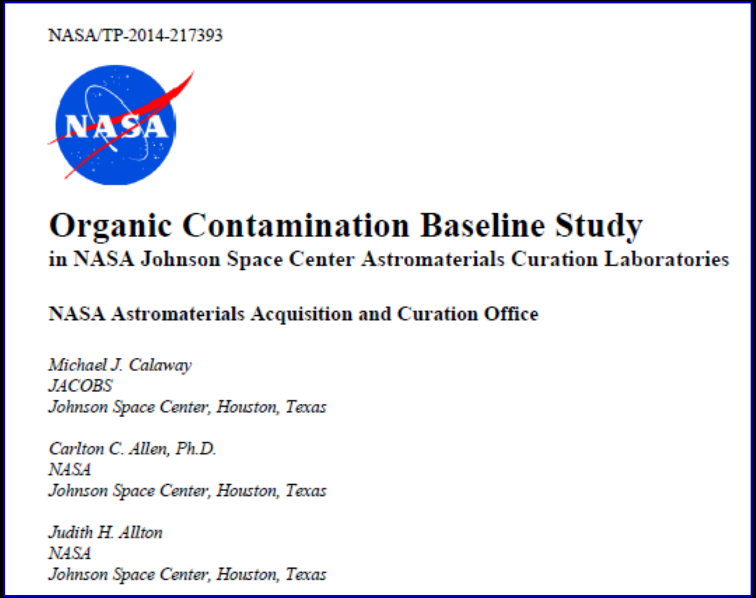
Carbonaceous regolith upcoming!

Moving forward: organic contamination

A concerted approach:

- Based on background knowledge
- Low-organic curation facilities construction
- Quantification of organic contaminants in current cleanrooms
- Quantification of biological contaminants in current cleanrooms, and mitigation plans
- Advanced cleaning

Calaway et al., 2014
NASA/TSP-2014-217393


NASA/TP-2014-217393

Organic Contamination Baseline Study
in NASA Johnson Space Center Astromaterials Curation Laboratories

NASA Astromaterials Acquisition and Curation Office

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Moving forward: organic contamination

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Element
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Ethyl hexanal
Heptamethylnonane + Methylnaphthalene
Limonene
m,p-Xylene
Nonanal
o-Xylene
Tetrachloroethylene
Toluene
TXIB

Outside air

Cleanroom materials

Cleaning agents

Silicone

Unidentified sources

DL: 0.1 ; Unit ng/L

Moving forward: organic contamination

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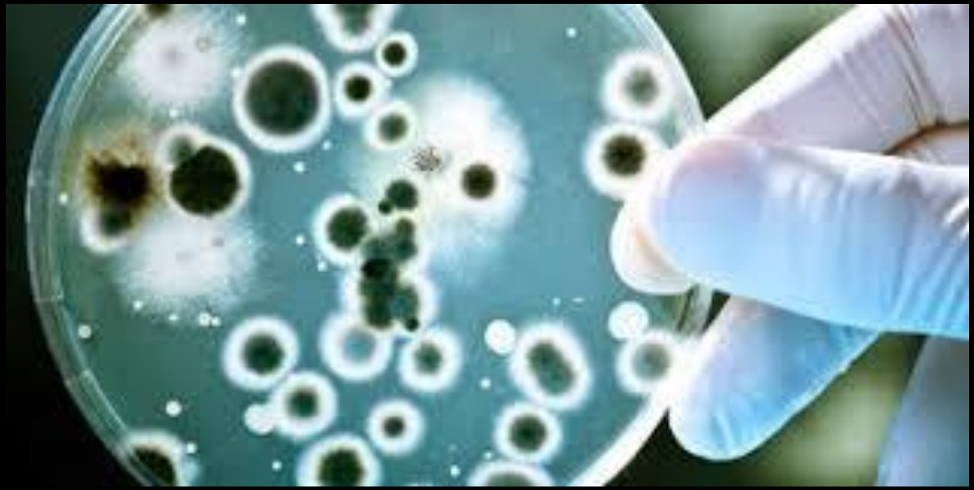
- Advanced cleaning



Moving forward: human exploration of Mars

- Current PP requirements
  - based on Viking-era robotic missions
  - Static through innovation
  - unachievable for human exploration…and we do not have yet human exploration PP requirements
- Lack of knowledge of current state-of-the-art of spacesuit contamination, needed to even start mitigation plans (filters, mechanical pressured suits, etc)
- (lack of knowledge of impact of Martian dust on humans)

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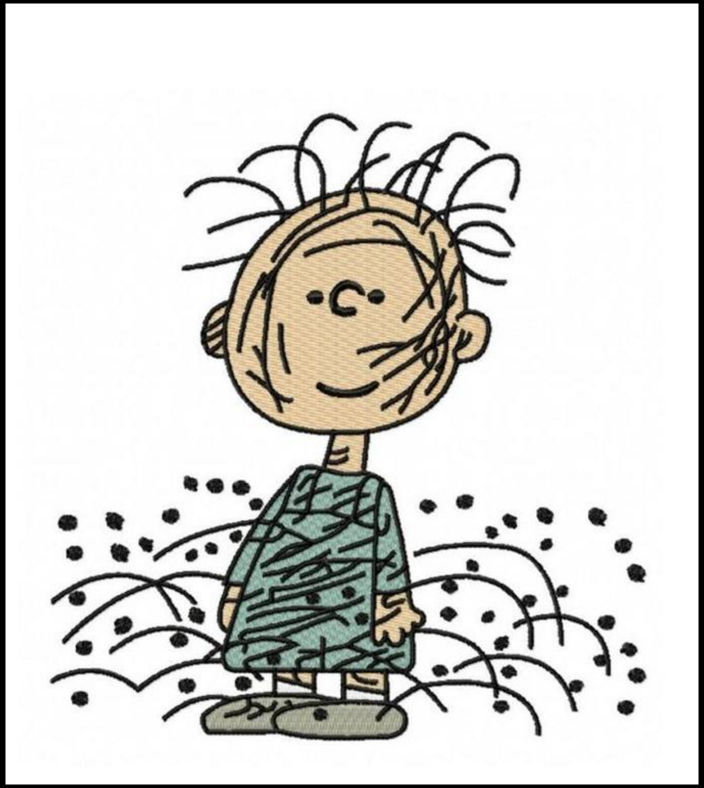

Final thoughts

- CCK has been adapted to the curation needs for the past 50 years, and successful at keeping the samples pristine.
- Organic and biological contamination are an upcoming concern for future sample return missions.
- The Curation Office has been preparing for these challenges for a few years, but more work needs to happen, with a multidisciplinary approach.
- Timeline still works, but shouldn’t wait too long.

AMC-MM and AMC-MC measurements

200mm silicon wafers and air sampling tubes provided by Balazs NanoAnalysis.

72h exposure; AMC-MC analyzed by Thermal Desorption-Gas Chromatography Mass Spectrometry (TD-GC-MS), protocol SEMI MF 1982-0714.

6h active air sampling; AMC-MC analyzed by Thermal Desorption-Gas Chromatography Mass Spectrometry (TD-GC-MS).

72h exposure; AMC-MM analyzed by Vapor Phase Decomposition Inductively Coupled Plasma Mass Spectrometry (VPD ICP-MS); 35 elements (Al, As, B, Ba, Be, Ca, Cd, Ce, Co, Cr, Cu, Fe, Ga, Ge, Hf, In, K, La, Li, Mg, Mn, Mo, Na, Ni, Pb, Sb, Sn, Sr, Ta, Ti, W, V, Y, Zn and Zr).

AMC Baseline – Air-sampled organics

January 2019

		ISO 5	ISO 5	ISO 6	ISO 6
Element (ng/L; DL:0.1)	Blank	Stardust	Cosmic Dust	Meteorite	Final Clean
Low Boilers C7 - C10	*	8.2	17.3	19.3	19.2
Medium Boilers >C10 - C20	*	20	39.8	57.5	44.7
High Boilers >C20	*	*	*	*	*
Sum >=C7	*	28.2	57.1	76.8	63.9

Potential contamination sources:

- Stardust: Vaseline oil (gears), silicon-based aerogel
- Cosmic Dust: silicone oil (collectors), lexan (polycarbonate), hexane (oil thinner)
- Meteorites/Final Clean: nylon and Teflon heat sealing

AMC Baseline – Air-sampled organics

January 2019

Element (ng/L; DL:0.1)	Blank	Stardust (ISO 5)	Cosmic Dust (ISO 5)	Meteorite (ISO 6)	Final Clean (ISO 6)
Low Boilers C7 - C10	*	8.2	17.3	19.3	19.2
Medium Boilers >C10 - C20	*	20	39.8	57.5	44.7
High Boilers >C20	*	*	*	*	*
Sum >=C7	*	28.2	57.1	76.8	63.9

1998 Study of Organics in Air

Hydrocarbons	Control Blank (ng/L)	Meteorite Cleanroom TD-GC-MS Results (ng/L)	Mars Glovebox TD-GC-MS Results (ng/L)
Low boilers C6-C10	10	63	16
Medium boilers >C10-C20	2	93	11
High boilers >C20	*	1	*
Sum >= C6	12	157	27

Only comparable study

AMC Baseline – Air-sampled organics

Element	Blank	Stardust	Cosmic Dust	Meteorite	Final Clean
2-(2-Ethoxyethoxy)ethanol	*	*	*	0.3	*
Alpha.Pinene	*	0.1	0.2	0.3	0.3
Benzaldehyde	*	**0.9**	**0.8**	**0.8**	**0.9**
Benzene	*	0.3	0.2	0.3	0.3
Butoxy ethanol	*	*	0.2	0.5	0.4
C10-C14 Hydrocarbon	*	**0.4**	**1.5**	**1.5**	**1.8**
C12 Hydrocarbon + Naphthalene	*	0.2	**0.4**	**0.5**	**0.5**
C3 Benzene + Cyclo(Me2SiO)4	*	*	**1.2**	**1.1**	**1.3**
C3-C4 Benzene	*	0.1	**1.5**	**2**	**2.7**
C6-C9 Hydrocarbons	*	**1.8**	**3.5**	**3.9**	**4.1**
Cyclo(Me2SiO)3	*	**0.3**	**0.4**	**0.5**	**0.5**
Cyclo(Me2SiO)4	*	**0.6**	*	*	*
Cyclo(Me2SiO)5	*	**13.2**	**25**	**27**	**28.4**
Cyclo(Me2SiO)6	*	**0.3**	**0.7**	**0.7**	**0.8**
Ethyl hexanal	*	*	**0.5**	*	**0.5**
Heptamethylnonane + Methylnaphthalene	*	*	*	0.3	0.3
Limonene	*	0.4	**2.2**	**2.9**	**3.4**
m,p-Xylene	*	0.3	0.5	0.5	0.6
Nonanal	*	*	0.4	0.4	0.5
o-Xylene	*	0.1	0.2	0.2	0.3
Tetrachloroethylene	*	0.4	0.8	0.3	0.2
Toluene	*	**0.8**	**1.2**	**1.3**	**1.3**
TXIB	*	0.4	**3.3**	**4.3**	**3.6**

Cleaning agents (or insecticide)

DL: 0.1 ; Unit ng/L

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C10-C14 Hydrocarbon	*	**0.4**	**1.5**	**1.5**	**1.8**
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o-Xylene	*	0.1	0.2	0.2	0.3
Tetrachloroethylene	*	0.4	0.8	0.3	0.2
Toluene	*	**0.8**	**1.2**	**1.3**	**1.3**
TXIB	*	0.4	**3.3**	**4.3**	**3.6**

Cleaning agents (or insecticide)

Outside air

DL: 0.1 ; Unit ng/L

AMC Baseline – Air-sampled organics

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Benzaldehyde	*	**0.9**	**0.8**	**0.8**	**0.9**
Benzene	*	0.3	0.2	0.3	0.3
Butoxy ethanol	*	*	0.2	0.5	0.4
C10-C14 Hydrocarbon	*	**0.4**	**1.5**	**1.5**	**1.8**
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o-Xylene	*	0.1	0.2	0.2	0.3
Tetrachloroethylene	*	0.4	0.8	0.3	0.2
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Outside air

Cleanroom materials (plasticizer, sealants…)

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Benzene	*	0.3	0.2	0.3	0.3
Butoxy ethanol	*	*	0.2	0.5	0.4
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Cleaning agents (or insecticide)

Outside air

Cleanroom materials (plasticizer, sealants…)

Silicone (samples collecting plates or deodorants

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Limonene	*	**0.4**	**2.2**	**2.9**	**3.4**
m,p-Xylene	*	0.3	0.5	0.5	0.6
Nonanal	*	*	0.4	0.4	0.5
o-Xylene	*	0.1	0.2	0.2	0.3
Tetrachloroethylene	*	0.4	0.8	0.3	0.2
Toluene	*	**0.8**	**1.2**	**1.3**	**1.3**
TXIB	*	0.4	**3.3**	**4.3**	**3.6**

DL: 0.1 ; Unit ng/L

Unidentified sources

Cleaning agents (or insecticide)

Outside air

Cleanroom materials (plasticizer, sealants…)

Silicone (samples collecting plates or deodorants

AMC Baseline – Organics on wafers

January 2019		ISO 5	ISO 5	ISO 6	ISO 6
Compound (ng/cm2; DL=0.1)	Blank	Stardust	Cosmic Dust	Meteorite	Final Clean
Low Boilers C7 - C10	*	1.5	3.5	1.6	1.2
Medium Boilers >C10 - C20	*	11	17.2	19.1	22.9
High Boilers >C20	*	2.6	1	3.4	3.8
Sum >=C7	*	15.1	21.7	24.1	27.9

Potential contamination sources:

- Stardust: Vaseline oil (gears), silicon-based aerogel
- Cosmic Dust: silicone oil (collectors), lexan (polycarbonate), hexane (oil thinner)
- Meteorites/Final Clean: nylon and Teflon heat sealing

AMC Baseline – Organics on wafers

Compound (DL: 0.1 ; Unit ng/cm²)	Blank	Stardust	Cosmic Dust	Meteorite	Final Clean
1,2-Benzenedicarboxylic acid, diheptyl ester	*	*	*	*	0.4
Alkyl ester	*	0.2	0.1	0.1	*
Alkyl ketone	*	0.2	*	*	*
Benzoic acid	*	*	0.5	0.2	0.1
Benzyl butyl phthalate	*	*	*	*	0.3
Bis(2-ethylhexyl) phthalate	*	*	*	*	*
C12 Hydrocarbon + Unknown(m/z:43,71,89,155)	*	0.4	0.3	*	*
C6-C9 Hydrocarbons	*	0.5	1.7	0.6	0.4
Cyclo(Me2SiO)8	*	0.1	0.1	*	*
Cyclo(Me2SiO)9	*	0.1	0.1	0.1	0.2
Cyclohexanol, 3,5-dimethyl- + Unknown(m/z:97)	*	1.1	0.8	0.1	0.3
Dibutyl phthalate	*	0.5	*	3.9	3.8
Diethylene glycol dibutyl ether	*	0.1	0.2	0.1	0.2
Diisobutyl phthalate	*	0.3	0.4	0.4	0.5
Dodecanenitrile	*	0.1	*	0.2	*
Heptyl octyl phthalate	*	*	*	*	0.4
Methacrylic acid	*	0.3	*	*	*
Mono(2-ethylhexyl)phthalate	*	*	*	0.5	0.6
Phenol	*	0.1	0.2	0.2	*
Phthalic anhydride	*	0.2	0.7	0.2	0.3
Possible Alkyl ester + Pentane, 1-iodo-	*	0.3	0.3	0.1	*
Siloxane	*	*	3	0.6	0.6
Tri(2-chloroethyl) phosphate	*	*	*	*	1.4
TXIB	*	3.7	4.2	4.7	6.6
Unknown(m/z:41,50,76,87,104,132,176,193)	*	0.2	*	*	*
Unknown(m/z:41,51,67,77,85,95,105,123,189)	*	*	0.3	0.1	*
Unknown(m/z:41,55,67,77,97,105,110)	*	*	0.7	0.3	0.2
Unknown(m/z:43,55,71,77,105,193,277)	*	*	0.3	0.5	0.5
Unknown(m/z:43,56,71,105,155,193,207,277)	*	*	2.5	*	*
Unknown(m/z:43,56,71,77,105,123,193,277)	*	*	*	3.5	3.5
Unknown(m/z:43,57,90,83,97,112,149,163,239,256)	*	*	*	0.2	*

Cleanroom materials (plasticizer, sealants…)

AMC Baseline – Organics on wafers

Compound / DL: 0.1; Unit: ng/cm²	Blank	Stardust	Cosmic Dust	Meteorite	Final Clean
1,2-Benzenedicarboxylic acid, diheptyl ester	*	*	*	*	0.4
Alkyl ester	*	0.2	0.1	0.1	*
Alkyl ketone	*	0.2	*	*	*
Benzoic acid	*	*	0.5	0.2	0.1
Benzyl butyl phthalate	*	*	*	*	0.3
Bis(2-ethylhexyl) phthalate	*	*	*	*	*
C12 Hydrocarbon + Unknown(m/z:43,71,89,155)	*	0.4	0.3	*	*
C6-C9 Hydrocarbons	*	0.5	1.7	0.6	0.4
Cyclo(Me2SiO)8	*	0.1	0.1	*	*
Cyclo(Me2SiO)9	*	0.1	0.1	0.1	0.2
Cyclohexanol, 3,5-dimethyl- + Unknown(m/z:97)	*	1.1	0.8	0.1	0.3
Dibutyl phthalate	*	0.5	*	3.9	3.8
Diethylene glycol dibutyl ether	*	0.1	0.2	0.1	0.2
Diisobutyl phthalate	*	0.3	0.4	0.4	0.5
Dodecanenitrile	*	0.1	*	0.2	*
Heptyl octyl phthalate	*	*	*	*	0.4
Methacrylic acid	*	0.3	*	*	*
Mono(2-ethylhexyl)phthalate	*	*	*	0.5	0.6
Phenol	*	0.1	0.2	0.2	*
Phthalic anhydride	*	0.2	0.7	0.2	0.3
Possible Alkyl ester + Pentane, 1-iodo-	*	0.3	0.3	0.1	*
Siloxane	*	*	3	0.6	0.6
Tri(2-chloroethyl) phosphate	*	*	*	*	1.4
TXIB	*	3.7	4.2	4.7	6.6
Unknown(m/z:41,50,76,87,104,132,176,193)	*	0.2	*	*	*
Unknown(m/z:41,51,67,77,85,95,105,123,189)	*	*	0.3	0.1	*
Unknown(m/z:41,55,67,77,97,105,110)	*	*	0.7	0.3	0.2
Unknown(m/z:43,55,71,77,105,193,277)	*	*	0.3	0.5	0.5
Unknown(m/z:43,56,71,105,155,193,207,277)	*	*	2.5	*	*
Unknown(m/z:43,56,71,77,105,123,193,277)	*	*	*	3.5	3.5
Unknown(m/z:43,57,90,83,97,112,149,163,239,256)	*	*	*	0.2	*

Unidentified sources

Cleanroom materials (plasticizer, sealants…)

AMC Baseline – Metals on wafers

Compound (1E10 atoms/cm²)	DL	blank	Stardust	Cosmic Dust	Meteorite	Final Clean	CICG
Barium (Ba)	0.001	*	*	*	0.002	*	*
Boron (B)	0.5	2.8	86	76	130	77	160
Calcium (Ca)	0.1	*	0.2	*	0.4	2.4	0.5
Chromium (Cr)	0.01	*	*	*	*	0.14	*
Cobalt (Co)	0.005	*	*	*	*	*	0.005
Copper (Cu)	0.01	*	*	*	0.01	*	*
Iron (Fe)	0.05	*	*	*	0.17	0.68	*
Magnesium (Mg)	0.05	*	*	*	0.36	0.1	0.06
Nickel (Ni)	0.05	*	*	*	*	0.05	*
Potassium (K)	0.05	*	0.12	*	0.15	*	1.6
Sodium (Na)	0.05	0.08	0.45	0.13	0.29	0.47	0.3
Tin (Sn)	0.005	*	0.008	*	*	*	1.6
Zinc (Zn)	0.05	*	0.08	0.09	0.05	0.19	*

Boron from HEPA filters

Ca, Mg, K and Na: Human contamination

Fe, Cr: Stainless steel

Tin, Zn: brass in instruments, or solder, or electrical systems

Summary

Strong baseline, showing that our cleanrooms are doing their job, and pointing to a few minor sources of contamination (housekeeping, outside air).

Next steps:

Expose Al foil for AMC-MC, and archive every month for long-term results.

Track sources of contamination; perform material testing if necessary.