

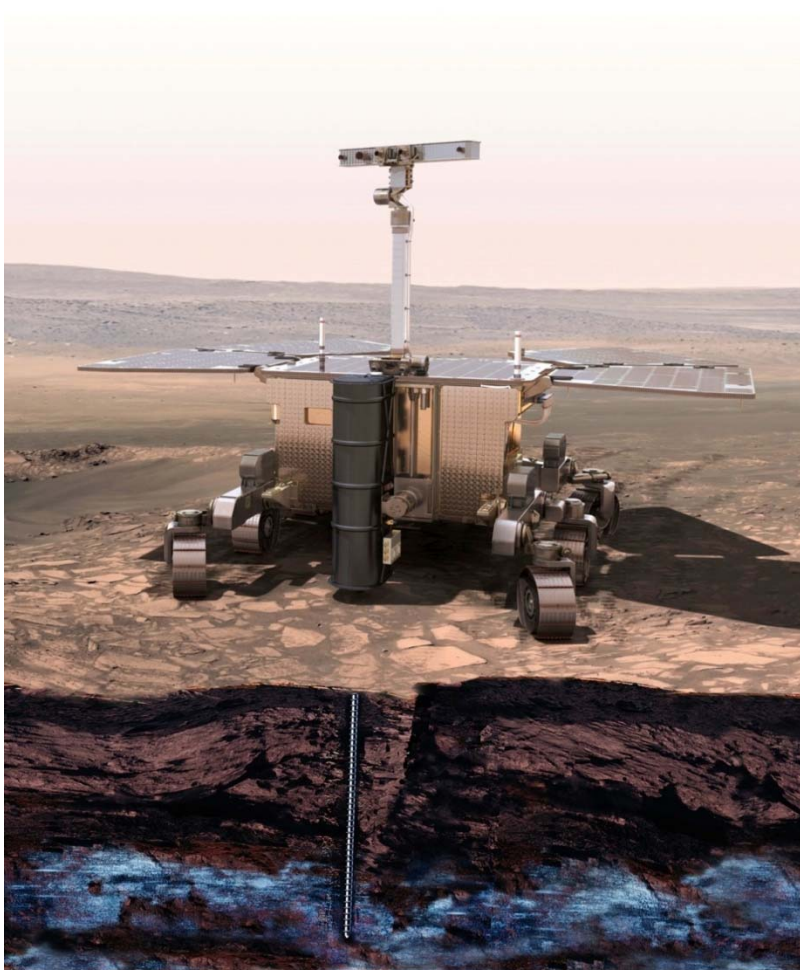
# Developing the cleanliness requirements for an organic-detection instrument

## Mars Organic Molecule Analyzer – Mass Spectrometer (MOMA-MS)

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# 2018 ExoMars Rover



- **Science:** Search for signs of past and present life on Mars
- **Launch:** May, 2018 on Proton-M
- **EDL:** February, 2019
- **Nominal Mission:** 218 sols
- **Mass:** 310 kg
- **Science Payload Mass:** 26 kg
- **Key Payload Elements:**
  - 2 m sampling drill
  - Remote (Mast) instruments
  - Contact (Drill) instruments
  - Laboratory instruments with sample crusher and delivery carousel
    - RLS (Raman Laser Spectrometer)
    - MicrOMEGA (imaging IR spectrometer)
    - MOMA (pyro-GC/MS, LDMS)

# ExoMars with MOMA Enables Critical Mars Science!

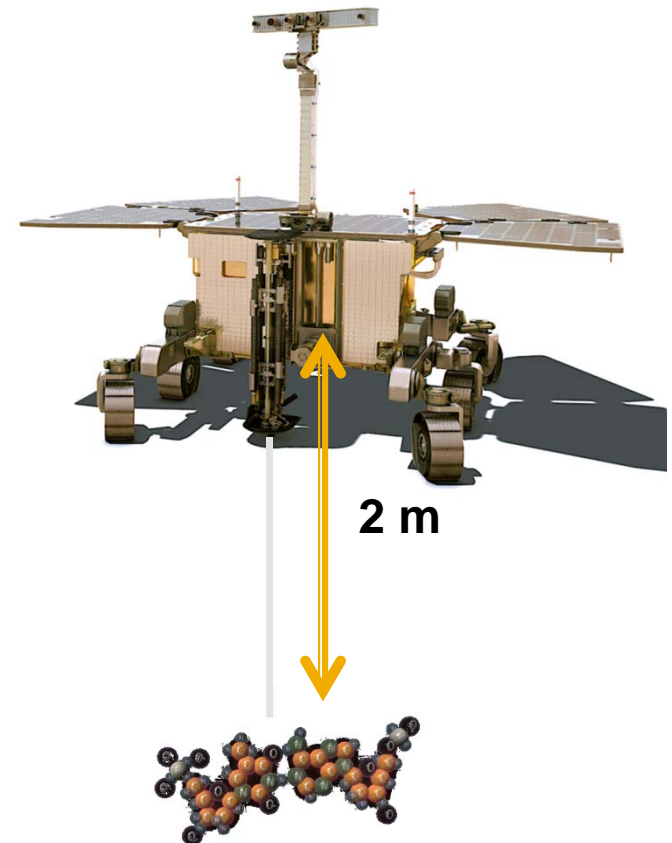
## Search for signs of past or present life?



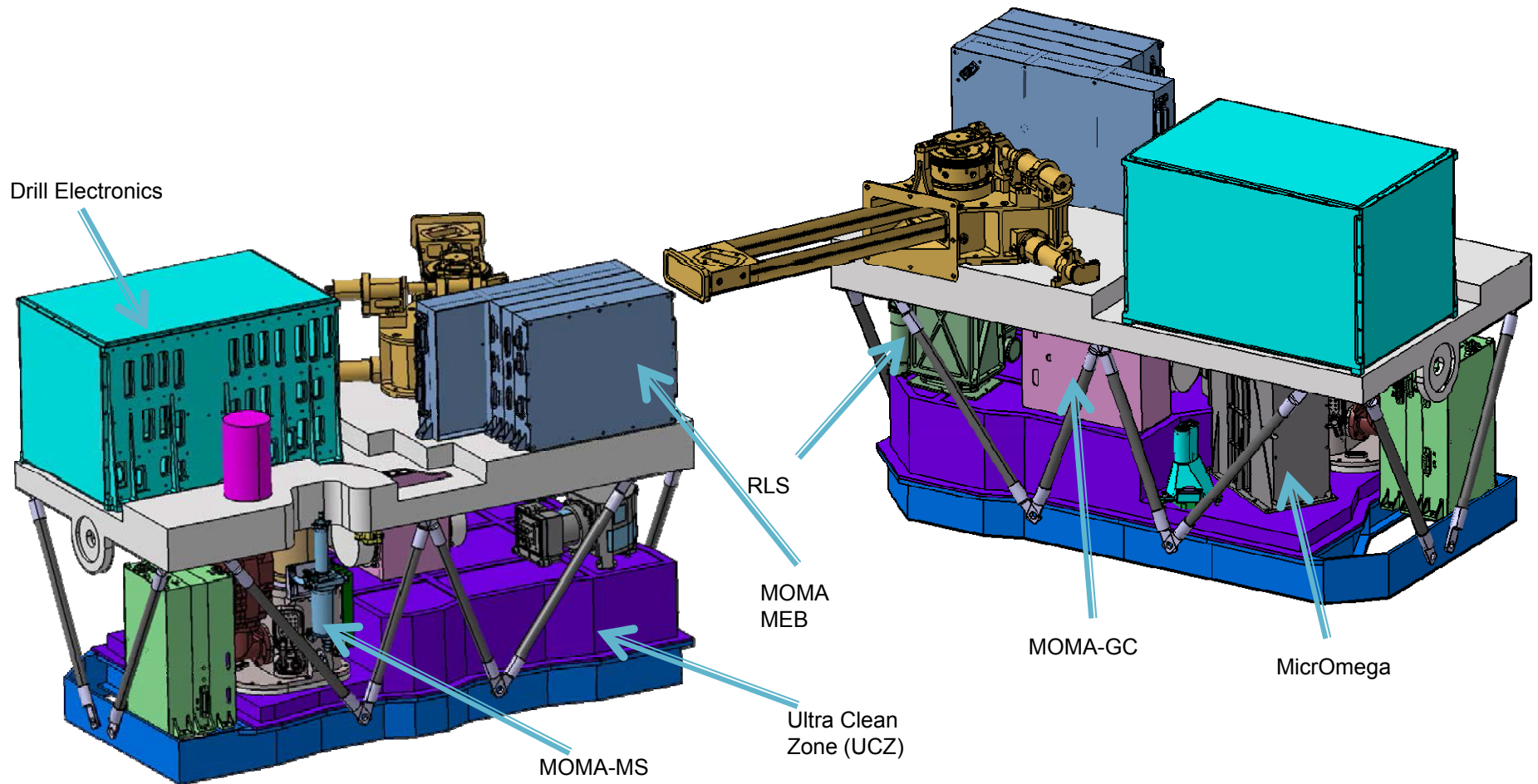
- Complex organics with *nonrandom, repeating structures* (e.g., biopolymers)
- Organics do not exist in isolation: potential mixture of abiotic/meteoritic and biogenic
- *Chirality* (handedness) as a biomarker

The surface of Mars is bathed in ultraviolet and cosmic radiation, potentially leading over time to the degradation of complex organics in the uppermost surface layers.

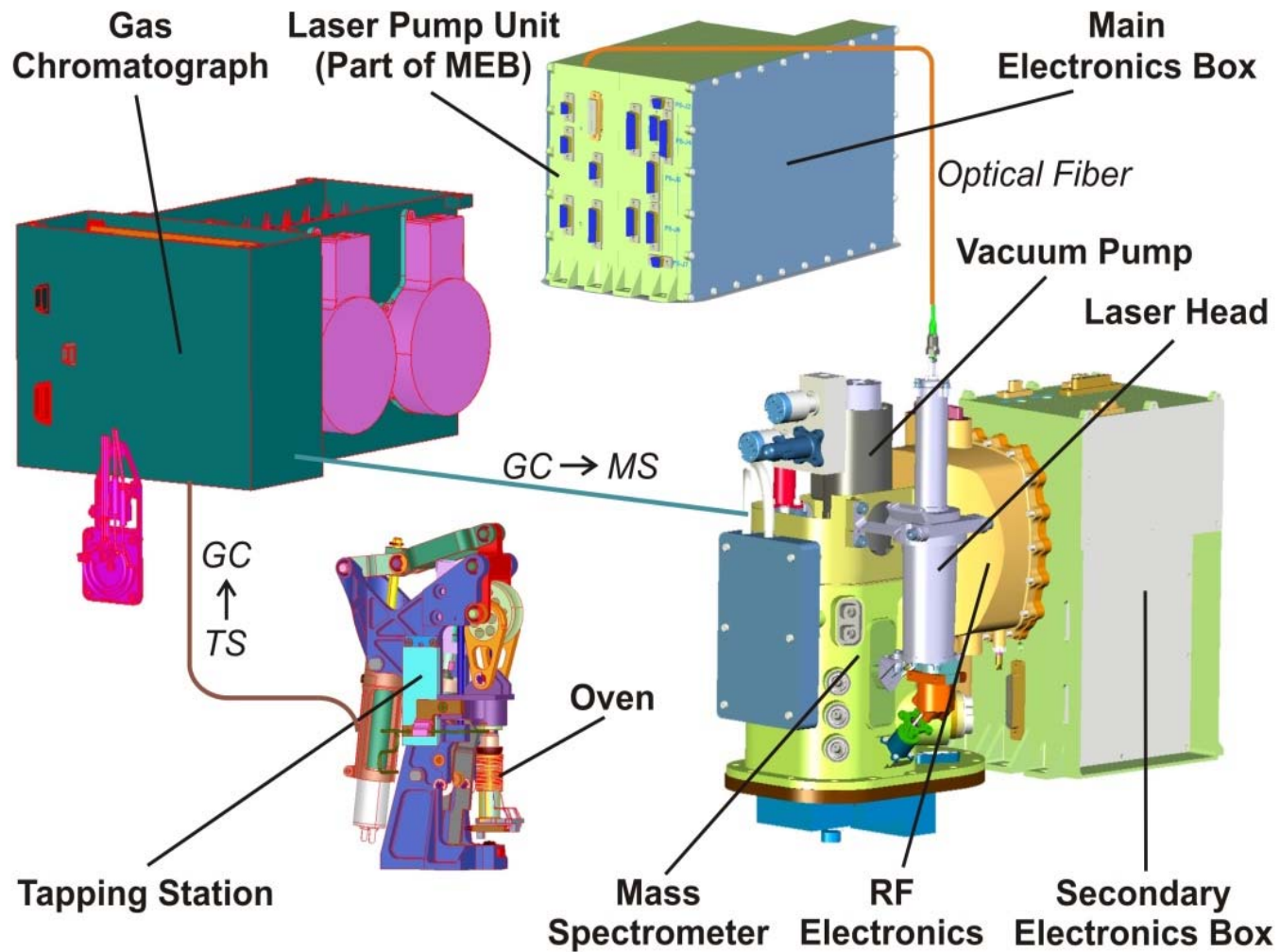
MOMA provides both *pyrolysis/gas chromatography and laser desorption MS* analysis of samples from as deep as 2 meters, potentially revealing a gradient of organics!



# Analytical Laboratory Drawer (ALD) Architecture



# MOMA Instrument Modules



# Requirements

# COSPAR Category IV requirements for Mars

Category IV for Mars is subdivided into IVa, IVb, and IVc:

Category IVa. Lander systems not carrying instruments for the investigations of extant Martian life are restricted to a biological burden no greater than Viking lander pre-sterilization levels

Category IVb. For lander systems designed to investigate extant Martian life, all of the requirements of Category IVa apply, along with the following requirement:

The entire landed system must be sterilized at least to Viking post-sterilization biological burden levels, or to levels of biological burden reduction driven by the nature and sensitivity of the particular life-detection experiments, whichever are more stringent

OR

The subsystems which are involved in the acquisition, delivery, and analysis of samples used for life detection must be sterilized to these levels, and a method of preventing recontamination of the sterilized subsystems and the contamination of the material to be analyzed is in place.

Definition of “Special Region”

- A Special Region is defined as a region within which terrestrial organisms are likely to propagate, OR a region which is interpreted to have a high potential for the existence of extant Martian life forms.
- Given current understanding, this is apply to regions where liquid water is present or may occur. Specific examples include but are not limited to:
  - Subsurface access in an area and to a depth where the presence of liquid water is probable
  - Penetrations into the polar caps
  - Areas of hydrothermal activity.

<http://cosparhq.cnes.fr/Scistr/Pppolicy.htm>

# Initial Derivation

- The core “Level 1” science requirement for cleanliness:  
*The maximum organic contamination level per gram of martian sample shall be less than 50 ng*
- Derivation of cleanliness requirements documented by M. Giuliani in “EXOMARS RM End of Life Cleanliness Analysis Report” (2013)
  - Includes contamination budget from hardware receiving through end of mission
  - Evaluates physical transfer of molecular contaminants from surfaces to sample through contact
  - Evaluates organic contribution from particles
  - Molecular transfer by offgassing



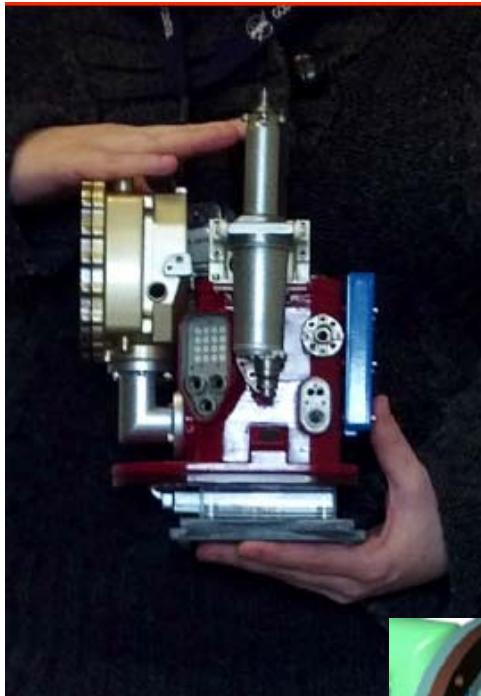
# Derived Requirements (at Delivery)

- Particulate [*Level 50 (0.2 ppm) at BOL*]
  - UCZ/Sample Path: Level 1 (0.0004 ppm)
  - Analytical Laboratory Drawer (exterior): Level 500 (2400 ppm)
- Molecular [*Level A/100 (10 ng/cm<sup>2</sup>) at BOL*]
  - UCZ/Sample Path: Level A/1000 (1 ng/cm<sup>2</sup>)
  - Analytical Laboratory Drawer (exterior): Level A (1000 ng/cm<sup>2</sup>)
  - Outgassing:  $5.82 \times 10^{-19}$  g/cm<sup>2</sup>-s [ $<2$  ng over 9 months]
- Bioburden
  - UCZ/Sample Path:  $<0.03$  spores/m<sup>2</sup>
  - Analytical Laboratory Drawer (exterior):  $<1000$  spores/m<sup>2</sup>

# NASA/GSFC Verification Limits

- Surface Particles
  - Manual Microscope – count of particles on filter from solvent rinse
    - Limit of Detection:  $0.0002 \text{ mm}^2 (\pm 0.025 \text{ mm}^2)$
- Surface Molecular
  - Gravimetric – delta mass of evaporated rinse solvent
    - Limit of Detection:  $10,000 \text{ ng} (\pm 39,000 \text{ ng})$  [standard deviation of >100 “blank” samples over 6 years]
- Estimated area of MOMA-MS critical surfaces (used for normalization)
  - UCZ interface (Base):  $170 \text{ cm}^2$
  - Aperture Valve Assembly:  $108 \text{ cm}^2$
  - GPS Sample Path:  $148 \text{ cm}^2$  (~118  $\text{cm}^2$  is exhaust)
- Volatile Molecular (Organics)
  - Quartz Crystal Microbalance (QCM) – total outgassing measurement of MOMA-MS
    - Deposition rate of  $1 \text{ Hz/Hr} = 5.44 \times 10^{-13} \text{ g/cm}^2\text{-s}$  [converted to source outgassing by ratio of areas]
    - Limit of Detection:  $2.4 \text{ ng} (\pm 5 \text{ ng})$  [15MHz QCM:  $S=1.97 \text{ ng/cm}^2/\text{Hz}$ ]
    - Requires concurrent thermal stability of  $\pm 0.1^\circ\text{C}$
  - Sorption Tube – used for verification of tubing and sample pathway; flows neutral gas through system as carrier, with collection in sorption tube at exhaust
    - Limit of Detection:  $1 \text{ ng} (\pm 0.3 \text{ ng})$

# Bioburden Verification



- Swab external surface:
  - Just prior to delivery
  - **<1000 spores/m<sup>2</sup>** at delivery
- Swab base and protective bowl
  - Just prior to closeout before DHMR
  - Test for **<300 spores/m<sup>2</sup> before** 4-D DHMR to achieve <0.03 spores/m<sup>2</sup>
  - < 3 spores for total internal volume (11 swab samples of 25 cm<sup>2</sup> each)
- NOTE: The approved swabs for bioburden sampling are flocked and shed fibers.

*{A single swab has a scaling factor to 1111 spores/m<sup>2</sup>.  
Batching of swab samples allows the scaling factor to be  
divided by the number of swabs in the batch.}*

# Handling Microbially Reduced (Sanitized) Hardware

- The sanitized surfaces must be protected from the external environment by biofilters (HEPA equivalent) or covers/caps.
- The bioburden reduced surfaces may only be exposed in an Aseptic ISO 5 environment
- Preparation of aseptic work environment:
  - Wipe all surfaces with 70% IPA wetted, sterile wipes
  - Inspect for visible cleanliness with bright illumination
  - Illuminate with UVC for 15 minutes (optional)
  - Requires sterile gowning, gloves, and tools

# Estimated Exposure Limit for Sanitized Surfaces

- Assumed limitation for the probability of viable spore exposure is  $< 10^{-3}$
- The theoretical fallout capacity for particles 0.3 microns and larger can be defined by the particle distribution allocated in ISO 14644 air classifications as:
  - ISO 3 = 0.005/m<sup>2</sup>-s
  - ISO 4 = 0.05/m<sup>2</sup>-s
  - ISO 5 = 0.5/m<sup>2</sup>-s
- Assumes all particles are single spore carriers
- Aseptic conditions are assumed to decrease the viability of spores by 3-4 decades

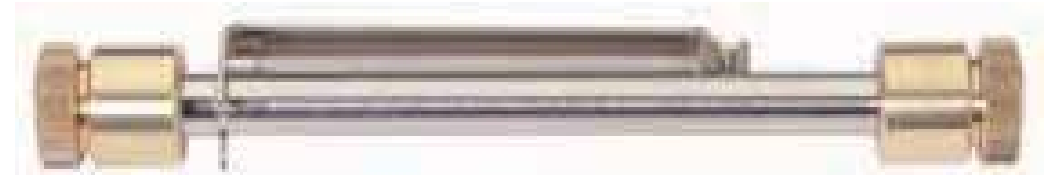
# What is a “Conservative” Area?

- Contamination Control
  - More concerned with normalized results
  - Assumes representative sampling – multiple samples are averaged
- Understating area is conservative
  - When full surface is rinse sampled
- Planetary Protection
  - Total biological load is most important
  - Multiple samples are batched (summed) as a single value
- Overstating area is conservative
  - Sample value is multiplied by the area for total load

# A Different Approach to Measuring Molecular Organic Contamination (MOC)

**Primary Objective:** Measure the volatile organic content of the gas stream to  $< 50$  ppb

- How?
  - Sorption tube connected to MS exhaust
  - Use flight-like GC gas flow of 2-3 sccm
  - Minimum sample size = 0.3 liters (1.8-2.3 hours)
  - All plumbing at  $135^{\circ}\text{C}$ ; Ultraclean Zone at  $50^{\circ}\text{C}$
- What?
  - Adsorbent resin – Tenax TA
  - Thermal desorption tube
- When?
  - During integrated testing



# Estimation of Minimum Thermal Desorption Sample Size

- Density of methane (STP): 0.72 g/l
- Background HC limit: 50 ppb<sub>v</sub> (methane equivalent)
- Allowable HC content in sample gas: 36 ng/l  
*[=density \* limit]*
- GC flow rate: 2-3 sccm
- Limit of detection: 1 ng (target 10 ng for certainty)
- Minimum collection time: 1.8-2.3 Hours  
*[=LOD/allowable/flow]*



# Thermal Desorption Allocations

- Oven (pyrolysis) < 30 ng total  
(minimum of 6 volume changes)
- GPS/MS MOC < 30 ng/L
- GC MOC < 15 ng/L  
*{Note: 1 L is the approximate volume of 6 GC runs}*
- pUCZ or UCZ < 30 ng/L  
*{Note: trend monitoring of the UCZ can inform the cruise outgassing model}*

# Current Status

- UCZ/Sample Path
  - Particles  $< 2_{(\pm 0.9)}$  ppm (Level 100) plus CO<sub>2</sub> snow cleaning
  - NVR  $< 100_{(\pm 140)}$  ng/cm<sup>2</sup> plus CO<sub>2</sub> snow cleaning
  - Volatile Organics  $< 50_{(\pm 0.01)}$  ppb<sub>v</sub>
  - Bioburden  $< 0.03_{(\pm 0.01)}$  spores/m<sup>2</sup> (after DHMR)
- Outgassing requirement replaced with a list of approved materials and quantity (13 materials currently identified)
  - All organic materials shall be subjected to vacuum bake-out of 75°C for 360 hours or equivalent
  - Outgassing characteristics defined by VBQC testing and COMOVA model inputs

# Summary

- Trace organic analysis, by robotic exploration, adds significant levels of complexity - contaminants can easily mask “signs of life” or present false positives
- Bioburden can be considered “one more form” of contamination, but has an entirely different emphasis
- Verification methods for bioburden and contamination are not always compatible
- Maintenance of sanitized surfaces requires Aseptic ISO 5 environmental conditions (and limited exposure times)
- The increasing sensitivity of space flight instrumentation is narrowing the margin for contamination impacts, resulting in extrapolated levels that are immeasurable by “standard” methods

# Acknowledgements

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