National Aeronautics and Space Administration



Preliminary testing of NASA's Molecular Adsorber Coating technology for future missions to Mars

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Abstract



The Molecular Adsorber Coating (MAC) is a sprayable coatings technology that was developed at NASA Goddard Space Flight Center (GSFC). The coating is comprised of highly porous, zeolite materials that help capture outgassed molecular contaminants on spaceflight applications. The adsorptive capabilities of the coating can alleviate molecular contamination concerns on or near sensitive surfaces and instruments within a spacecraft. This paper will discuss the preliminary testing of NASA's MAC technology for use on future missions to Mars. The study involves evaluating the coating's molecular adsorption properties in simulated test conditions, which include the vacuum environment of space and the Martian atmosphere. MAC adsorption testing was performed using a commonly used plasticizer called dioctyl phthalate (DOP) as the test contaminant.

 Keywords: molecular adsorber coating, molecular adsorbers, getters, MAC, zeolite, coatings technology, outgassing, molecular contamination, contamination control, vacuum chamber, Martian environment, Mars, CO2, carbon dioxide, dioctyl phthalate, DOP, plasticizer

Presentation Outline



INTRODUCTION	Mars ExplorationMolecular Adsorber Coatings
APPROACH	 Test Background Sample Fabrication Sample Conditioning Test Configuration Contaminant Source Test Run Summary Chamber Background Run Contaminant Baseline Runs Sample Test Runs Test Parameter Comparison
TEST METHODS	 Test Analysis Summary Gravimetric Analysis Method Chemical Analysis Methods
RESULTS & DISCUSSION	Gravimetric Analysis ResultsChemical Analysis Results
SUMMARY	ConclusionsFuture Work



IMAGE CREDIT: NASA/JPL CAL-TECH



Introduction

- Mars Exploration
- Molecular Adsorber Coatings

Mars Exploration

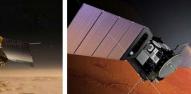


For many decades, NASA and its international partners have been at the forefront of Mars exploration through a series of missions, which have included orbiters and landers





MAVEN



ExoMars 2016 (ESA/Roscosmos)

Orbiter

Mars Reconnaissance Mars Express (ESA)











Mars 2020

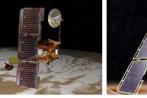
ExoMars Rover (ESA) Mission)

InSight (Discovery

Mars Science Laboratory - Curiosity



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2001 Mars Odyssey

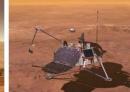
Mars Climate Orbiter Mars Global Surveyor Mars Observer











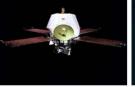
Mars Phoenix

- Opportunity

Mars Exploration Rover Mars Exploration Rover Mars Polar - Spirit

Lander/Deep Space 2





Mariner 6&7



Mariner 3 & 4



Pathfinder

Viking 1&2



Mariner 8 & 9

IMAGE CREDIT: NASA

Mars Exploration





IMAGE CREDIT: NASA/KSC

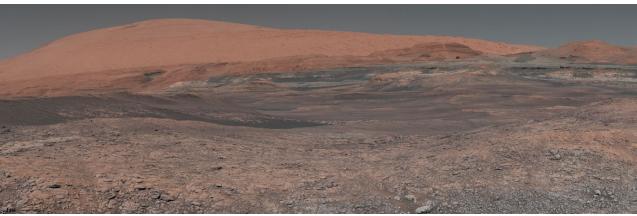
- Mars is the 4th planet from the sun
- It is often referred to as the "Red Planet"
- The Mars Exploration Program (MEP) is led by NASA
- The program's science goals are to:
 - Determine the potential for prior habitability and biological life
 - Understand the processes and history of climate on Mars
 - Study the geological origins and evolution of Mars
 - Pave the way towards human exploration in the future

Mars Exploration



- Highly sensitive, mobile laboratories abroad the rovers perform experiments, which include trace organic analysis of collected samples
- However, cross-contamination can interfere with the scientific findings because the presence of molecular contaminants can disguise potential signs of life or show false positives
- Maintaining cleanliness of the spacecraft through all the phases of the mission has become one of the most challenging aspects on upcoming Mars missions (i.e. Mars 2020 and ExoMars rovers)
 - Thus, there is a <u>current need</u> to explore innovative contamination control mitigation methods





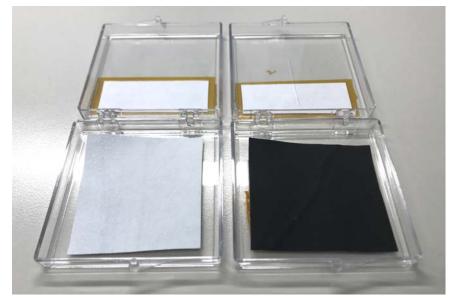
Images of Mount Sharp on Mars taken by the Mast Camera on NASA's Curiosity Rover

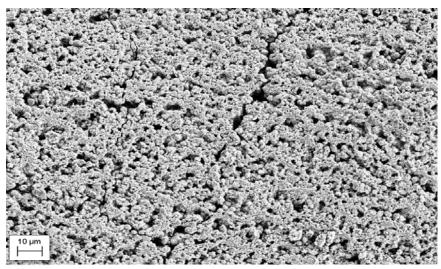
IMAGE CREDIT: NASA/JPL CAL-TECH

Molecular Adsorber Coatings



- MAC is an existing zeolite-based, sprayable coatings technology developed by NASA GSFC
- Used to mitigate molecular contamination concerns for various spaceflight applications
- Designed to passively entrap outgassed species that may otherwise deposit on critical instruments and components, and degrade the performance and the lifetime of NASA missions
 - These outgassed contaminants may originate from commonly used materials on the spacecraft, such as adhesives, lubricants, epoxies, and potting compounds
- Has been ground tested and flight qualified at some of the representative spaceflight conditions, which include high vacuum pressures and moderate temperature ranges
- Several NASA missions, such as ICON and JWST, have found practical applications for MAC





Molecular Adsorber Coatings



NASA Mission	Ionospheric Connection Explorer (ICON)	James Webb Space Telescope (JWST)
Туре	Flight Application	Ground Application
Description	MAC plates were installed within ICON's contamination sensitive Far Ultraviolet (FUV) instrument to reduce the effects of on-orbit material outgassing	MAC samples have been extensively used as an effective contamination getter during vacuum chamber testing of JWST's critical flight and optical ground support hardware



IMAGE CREDIT: NASA/UC BERKLEY

Reference: Abraham, Nithin S., Hasegawa, Mark M., and Secunda, Mark S., "Application of the Molecular Adsorber Coating technology on the lonospheric Connection Explorer program", Proc. SPIE 9952, Systems Contamination: Prediction, Control, and Performance 2016, 99520D (September 2016)

IMAGE CREDIT: NASA/CHRIS GUNN

Reference: Abraham, Nithin S., Hasegawa, Mark M., Wooldridge, Eve M., and Henderson-Nelson, Kelly A., "The use of the Molecular Adsorber Coating technology to mitigate vacuum chamber contamination during Pathfinder testing for the James Webb Space Telescope", Proc. SPIE 9952, Systems Contamination: Prediction, Control, and Performance 2016, 99520C (September 2016)



Approach

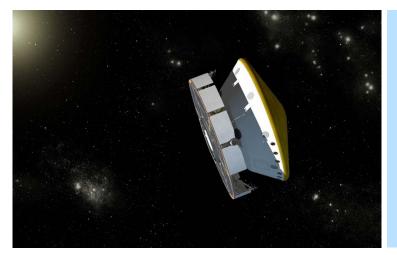
- Test Background
- Sample Fabrication
- Sample Conditioning
- Test Configuration
- Contaminant Source
- Test Run Summary
- Chamber Background Run
- Contaminant Baseline Runs
- Sample Test Runs
- Test Parameter Comparison

Test Background



 A preliminary study was performed to evaluate the molecular adsorption properties of MAC in two relevant environments for future Mars exploration missions

	Phase A	Phase B	
Simulated Environment	Vacuum of Space	Mars Atmosphere	
Test Pressure Range	High Vacuum (10 ⁻⁷ Torr)	Low Vacuum (7 Torr)	
Test Gas Purge	-	Carbon Dioxide	



- Phase A simulates the voyage of the spacecraft in space to Mars
- The cruise period can typically vary depending on the launch conditions, such as the orbit between Earth and Mars, as well as, the propulsion technology that is available
- The average journey to Mars from Earth is about 9 months

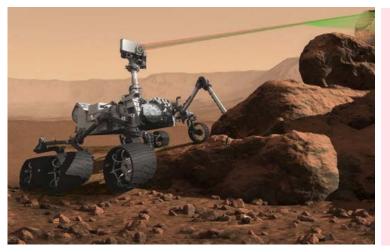
IMAGE CREDIT: NASA/JPL-CALTECH

Test Background



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Test Pressure Range	High Vacuum (10 ⁻⁷ Torr)	Low Vacuum (7 Torr)
Test Gas Purge	-	Carbon Dioxide



- Phase B simulates the contact of the spacecraft in the Martian atmosphere
- The atmospheric pressure on the surface of Mars can vary from about 3 to 7 Torr depending on seasonal variations
- The Martian atmosphere is comprised of about 96% carbon dioxide with trace levels of argon, nitrogen, and oxygen

IMAGE CREDIT: NASA/JPL-CALTECH

Sample Fabrication

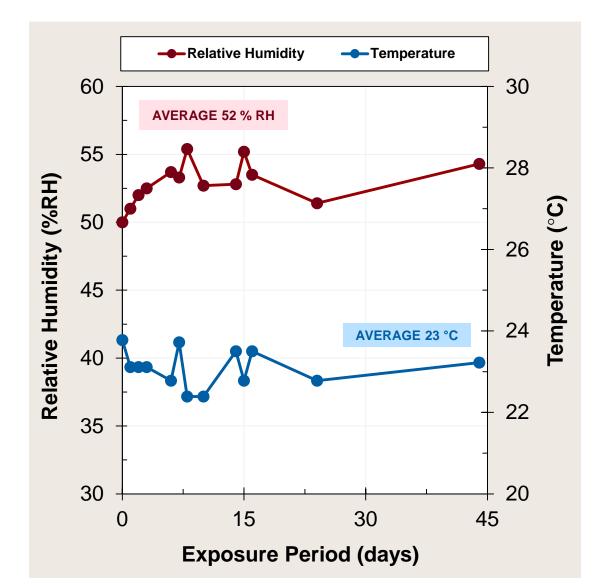


- NASA GSFC fabricated a total of 26 aluminum foil samples
- Samples were coated with the white version of the MAC technology
- Samples were divided into two batches with two thickness variations
 - Note that 1 mil (or 1 thousandth of an inch) is equivalent to 25.4 microns
- Coating area per sample was approximately 3.9 cm²

Batch ID	Number of Samples	МАС Туре	Average Coating Thickness
Ι	13	MAC-W	7.2 mils ± 1.0
II	13	MAC-W	5.3 mils ± 1.0

Sample Conditioning

- ATLO refers to the Assembly, Test, and Launch Operations phases of a NASA Mars mission
- During ATLO, flight hardware will be exposed to a relative humidity (RH) and temperature controlled environment until launch
- Similarly, the MAC samples were conditioned in a RH and temperature controlled laboratory for a duration of **approximately 45 days**
- This sample conditioning period simulates the expected exposure of the spacecraft components to ATLO conditions
- This period also helps evaluate any impact of moisture and trace levels of ambient, offgassed species in the room to MAC's adsorption capabilities while not in use



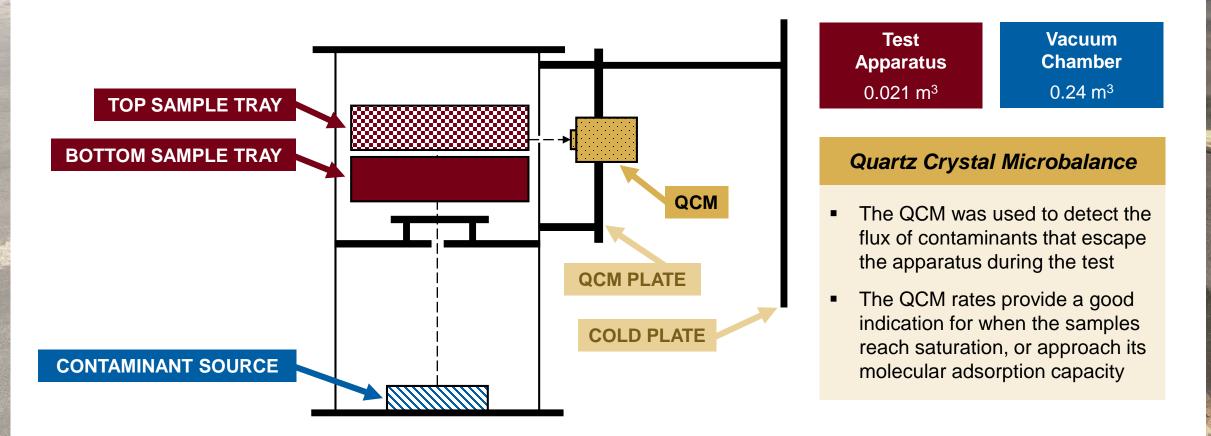




Test Configuration



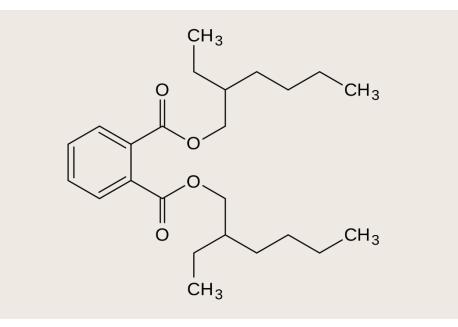
- Previous experiments have studied the molecular adsorption properties of MAC
- This experiment is performed by saturating MAC samples with a known contaminant source at a specified temperature within the confines of a test apparatus in a vacuum chamber



Contaminant Source



Chemical Name	Dioctyl phthalate		
Chemical Formula	$C_{24}H_{38}O_4$		
Molecular Weight	390.56 g/mol		
Purity	≥ 99.5 %		
Vendor	Sigma Aldrich		

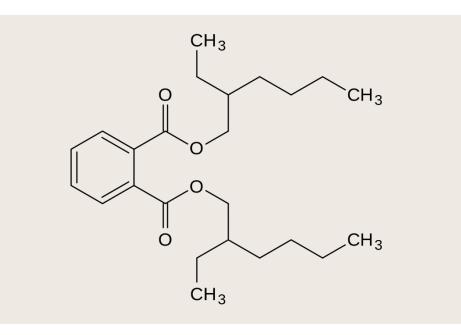


- Previous experiments have studied the molecular adsorption capacity of MAC using representative outgassed spaceflight contaminants
 - Simple, long-chain hydrocarbons
 - Stearyl Alcohol
 - Complex, silicone-based compounds
 - DC-704 Diffusion Pump Oil
- Plasticizers are also found in spaceflight applications, specifically on rinses of scavenger plates and cold fingers of vacuum chambers where spacecraft components are tested
- Therefore, a common plasticizer called **Dioctyl** phthalate (DOP) was selected as the contaminant source for these preliminary test efforts

Contaminant Source



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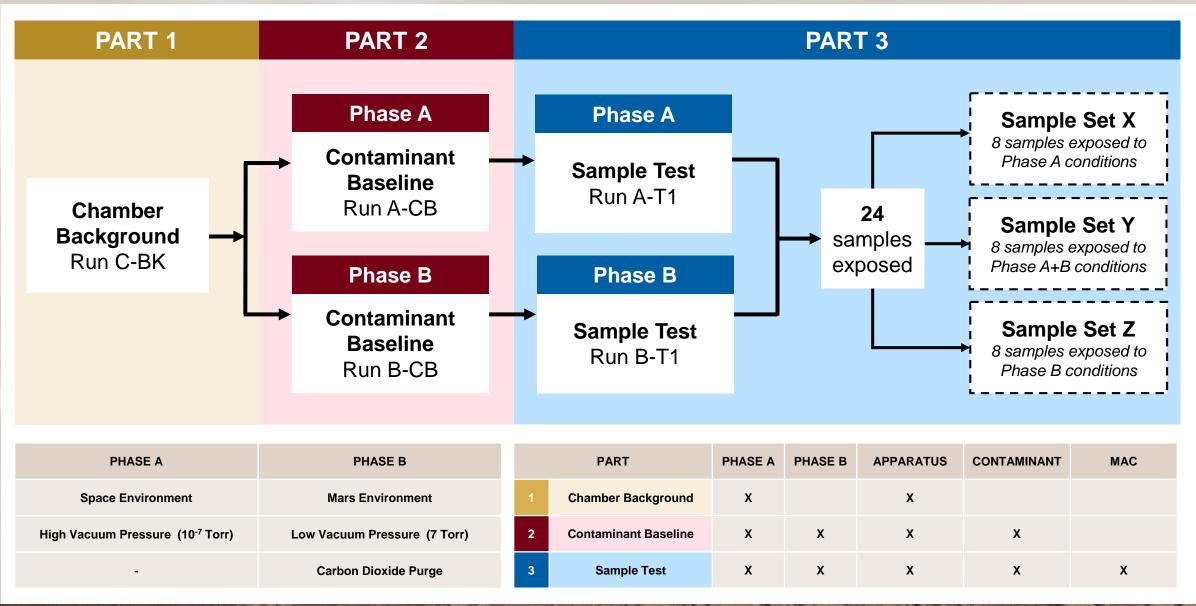
Dioctyl phthalate (DOP)

- Single component, high molecular weight plasticizer that is an ester of phthalic acid
- Commonly found in polymers, resins, elastomers, cosmetics and pesticides
- It appears as a colorless, odorless, oily non-volatile liquid
- Other chemical name synonyms include:
 - Bis(2-ethylhexyl) phthalate
 - DEHP
 - Diethylhexyl phthalate
 - Phthalic acid bis(2-ethylhexyl ester)

Test Run Summary



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Part 1. Chamber Background Run

NASA

Phase A Conditions

- Performed vacuum bake-out of test apparatus at various temperatures
- Established final background conditions based on QCM response rates of the vacuum chamber

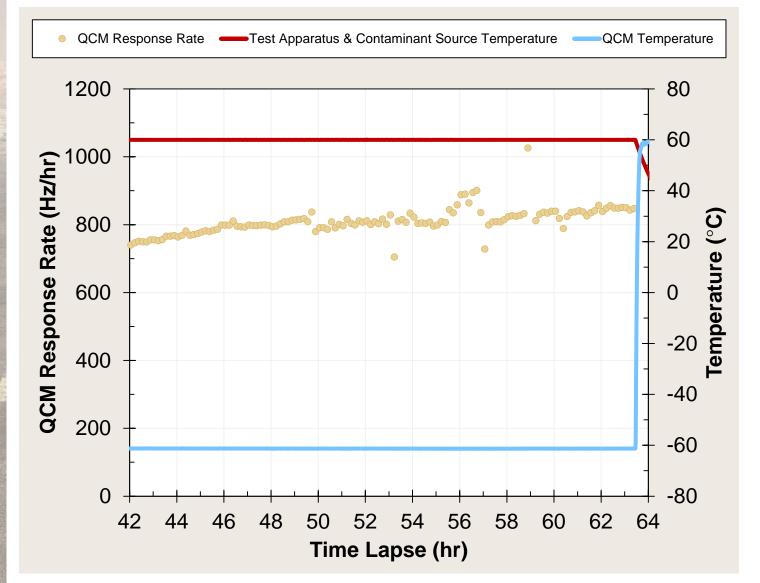
Test Duration	125 hours
Chamber Pressure	2.0 x 10 ⁻⁷ Torr
Number of Samples	-
Contaminant Mass	-
Contaminant Temperature	-
Apparatus Temperature	25, 45, 70 °C
Cold Plate Temperature	- 170 °C
QCM Temperature	- 60 °C
QCM Stabilization Rate	25 Hz/hr



MAGE CREDIT: NASA/GSFC

Part 2. Contaminant Baseline Runs





Phase A Conditions

- Established QCM response rates of DOP at various temperatures in the absence of any MAC samples
- Criteria for DOP temperature selection:
 - ✓ Provides an accelerated rate
 - ✓ Results in minimal maintenance of the QCM throughout the length of the test

Test Duration	65 hours
Chamber Pressure	2.4 x 10 ⁻⁷ Torr
Number of Samples	-
Contaminant Mass	2.5 g
Contaminant Temperature	60 °C
Apparatus Temperature	60 °C
Cold Plate Temperature	- 170 °C
QCM Temperature	- 60 °C
QCM Stabilization Rate	840 Hz/hr

Part 2. Contaminant Baseline Runs



CHAMBER PUMPED DOWN TO ROUGH VACUUM

Phase B Conditions

Attempted to establish QCM response rates of DOP at various temperatures in the absence of any MAC samples; however, many challenges were experienced!

PUMP VALVE PARTIALLY CLOSED AND THROTTLED WHILE PURGING CO₂ INTO CHAMBER

CHALLENGE 1.

Occurrence of snow and ice build-up on the cold surfaces of the test components

CHALLENGE 2.

Difficultly reaching and maintaining temperatures of at least 60 °C

CHALLENGE 3.

Little to no detection of QCM rate deposition from either the contaminant source or the chamber background

CHAMBER ISOLATED WHEN STEADY PRESSURE OF 7 TORR WAS ACHIEVED Due to the presence of CO₂ in the system, which condensed on the cold surfaces

Therefore, QCM and cold plate temperatures were increased to 25 and 0 °C, respectively

Likely due to the dominating convective heat transfer forces, and some limitations with heater power on the apparatus

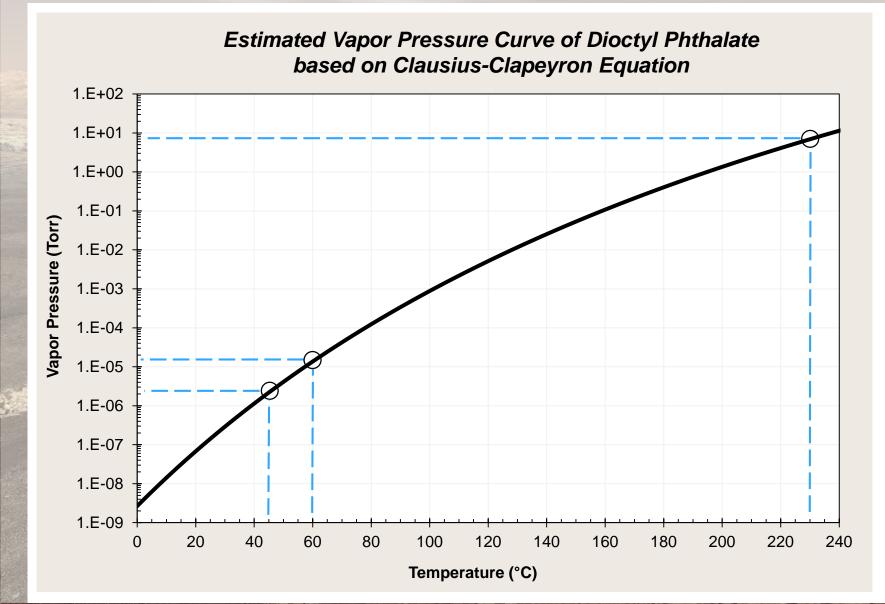
Test apparatus and contaminant source temperatures were decreased to 45 °C

Likely attributed to DOP not reaching its equilibrium vapor pressure

Therefore, since vapor pressure is a good indication of a liquid's evaporation rate, DOP may not easily evaporate at Phase B conditions

Part 2. Contaminant Baseline Runs





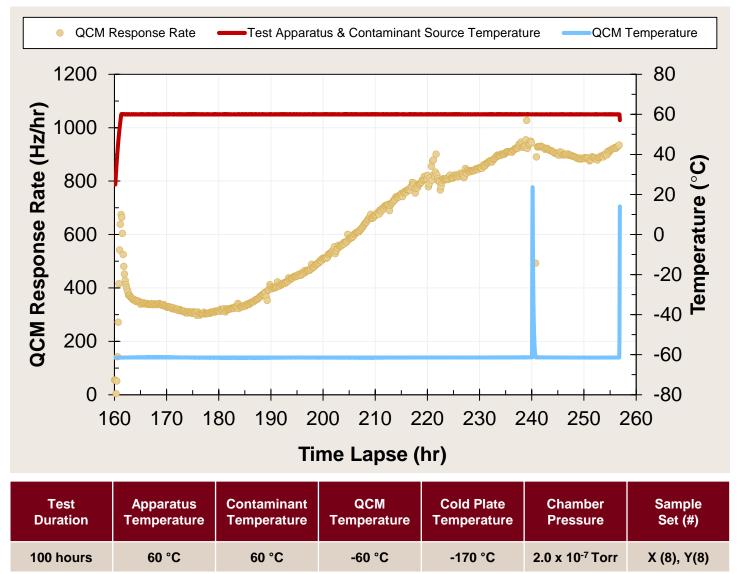
Vapor pressure of DOP at 45 °C was estimated at approximately 2.2 x 10⁻⁶ Torr

Estimated vapor pressure was calculated using the **Clausius-Clapeyron Equation**, where: ²²

 $ln \frac{P_1}{P_2} = \frac{H_{vap}}{R} \left(\frac{1}{T_2} - \frac{1}{T_1} \right)$

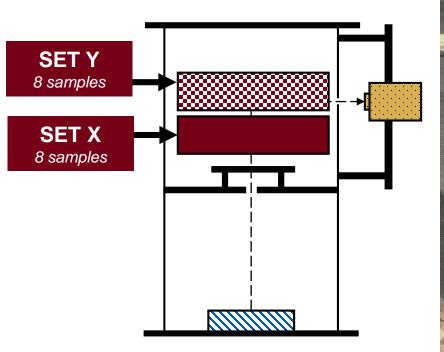
- Gas constant, R, is 8.3145 J/mol-K
- Enthalpy of vaporization, H_{vap}, based on Perry and Weber data at 125 °C is 107.6 kJ/mol as referenced on NIST Chemistry WebBook ¹⁹⁻²⁰
- Vapor pressure of DOP at 25 °C per the Hazardous Substances Data Bank (HSDB) is 1.42 x 10⁻⁷ mmHg as referenced in the NIH Pub Chem Open Chemistry Database ²¹





Phase A Conditions

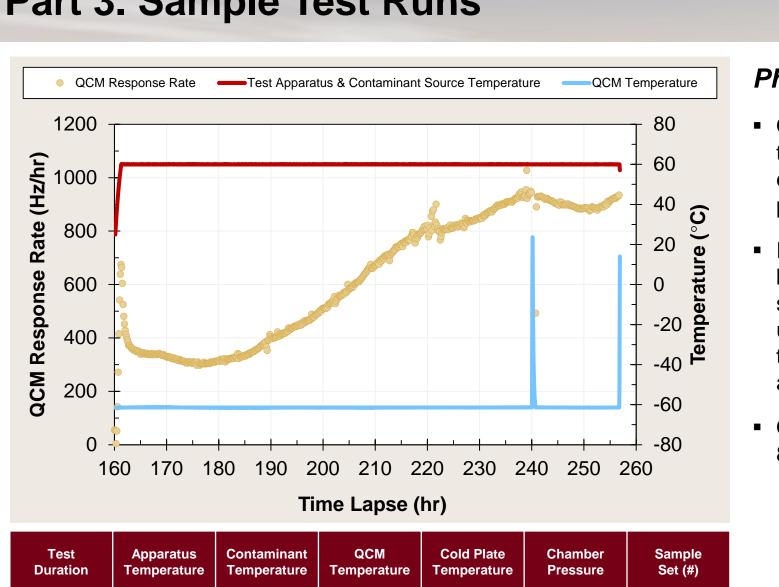
- DOP from contaminant baseline runs was replenished to avoid depletion during sample test runs
- Total of 16 MAC samples
 - Sample Set X and Set Y



100 hours

60 °C

60 °C



-60 °C

-170 °C

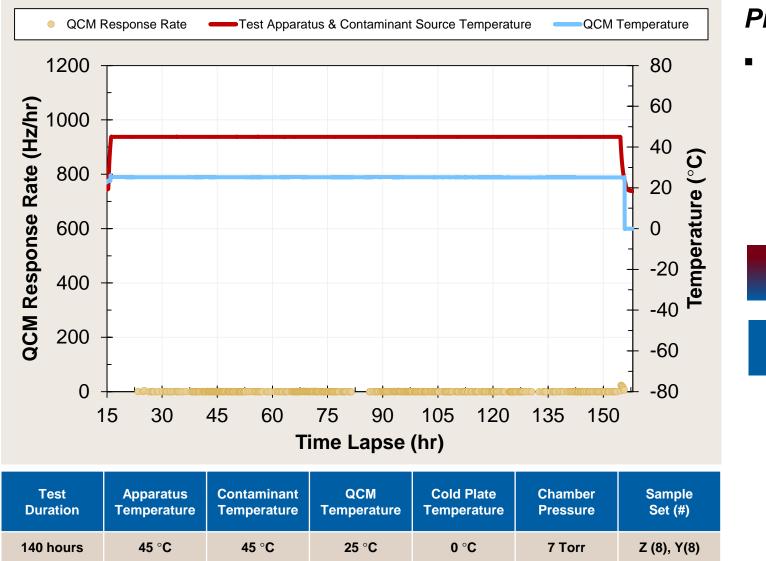
Phase A Conditions

- QCM rate initially drops from 650 to 300 Hz/hr, which is attributed to the entrapment of outgassed DOP onto the pores of MAC
- Rate gradually increases towards its baseline at 840 Hz/hr, which is due to the surface adsorption sites on MAC filling up, or reaching its saturation with DOP; thus, more species are exiting the test apparatus and depositing onto the QCM
- QCM rate appears to level off between 870 and 940 Hz/hr
 - The increased shift from the baseline may be due to excessive DOP build-up on the QCM crystal

2.0 x 10⁻⁷ Torr

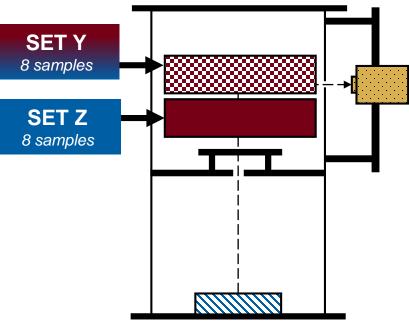
X (8), Y(8)

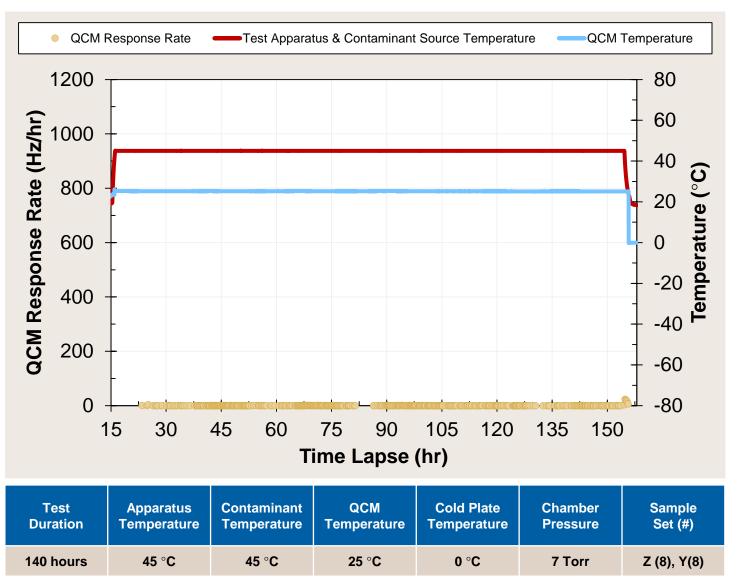




Phase B Conditions

- Total of 16 MAC samples
 - Set Z (new samples)
 - Set Y previously contaminated during Phase A sample test run





Phase B Conditions

- QCM detected little to no deposition of DOP or chamber background species, where ≤ 1 Hz/hr are likely invalid readings
- This made it challenging to determine when MAC will become saturated with DOP, if even possible!
- Thus, test was concluded with an additional 40 hrs more than Phase A

Measurement Period	Contaminant Mass	Mass Loss	
Pre-Phase A	3.08 g	0.74 a	
Post-Phase B	2.34 g	0.74 g	

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Test Parameter Comparison



Part	PART 1		PART 2		PART 3	
Test Description	Chamber Background		Contaminant Baseline		Sample Tests	
Phase	Α	В	Α	В	Α	В
Test Duration	125 hours	-	65 hours	-	100 hours	140 hours
Chamber Pressure	2.0 x 10 ⁻⁷ Torr	-	2.4 x 10 ⁻⁷ Torr	7 Torr	2.0 x 10 ⁻⁷ Torr	7 Torr
Number of Samples	-	-	-	-	16	16
Contaminant Mass -			2.5 g		3.1 g	
Contaminant Temperature	-	-	60 °C	45 °C	60 °C	45 °C
Apparatus Temperature	25, 45, 70 °C	-	60 °C	45 °C	60 °C	45 °C
Cold Plate Temperature	- 170 °C	-	- 170 °C	0°C	- 170 °C	0°C
QCM Temperature	- 60 °C	-	- 60 °C	25 °C	- 60 °C	25 °C
QCM Stabilization Rate	25 Hz/hr	-	840 Hz/hr	-	870-940 Hz/hr	≤ 1 Hz/hr

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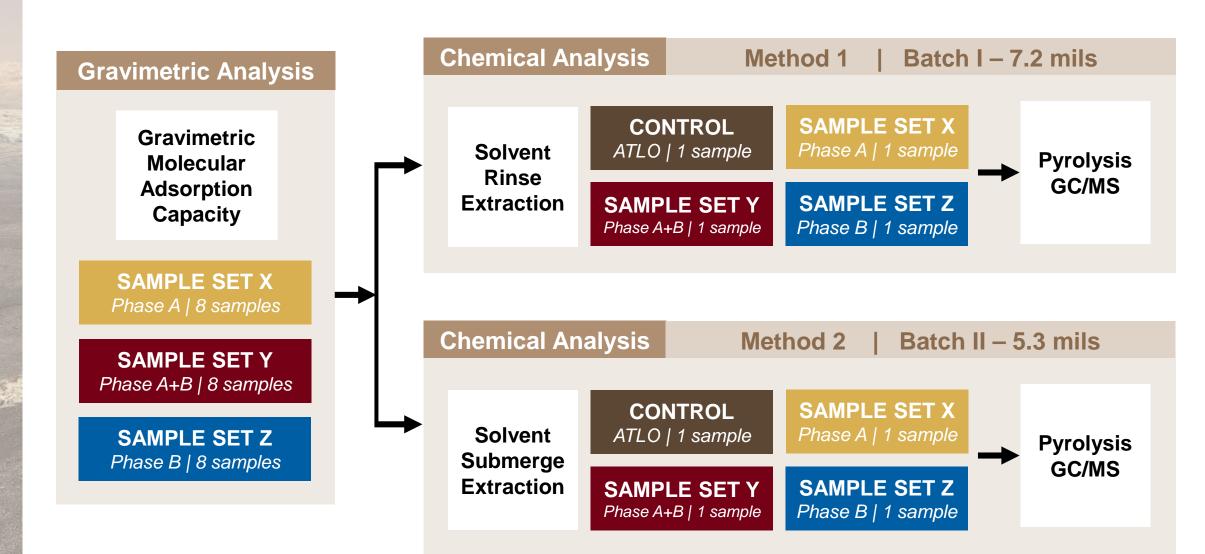


Test Methods

- Test Analysis Summary
- Gravimetric Analysis Method
- Chemical Analysis Methods

Test Analysis Summary





Gravimetric Analysis Method





IMAGE CREDIT: NASA / PAT IZZO

- Molecular Adsorption Capacity is defined as the measure of MAC's capability to adsorb or entrap outgassed materials, or molecular contaminants
- Also referred to as molecular capacitance (units: g/cm²)
- Dependent on various parameters, such as coating thickness, surface area coverage, type of contaminants, duration of exposure to contaminant, and other test conditions
- Calculated based on mass changes in the coating that are attributed to the entrapment of outgassed species
- Pre-exposure and post-exposure gravimetric measurements were performed in a nitrogen purged dry glove box at 25 °C and ≤ 5 %RH
- Samples were also exposed to vacuum for 48 to 72 hours prior to performing mass measurements to release any moisture
- A water absorption correction factor was incorporated to the calculated value to account for any errors associated with moisture

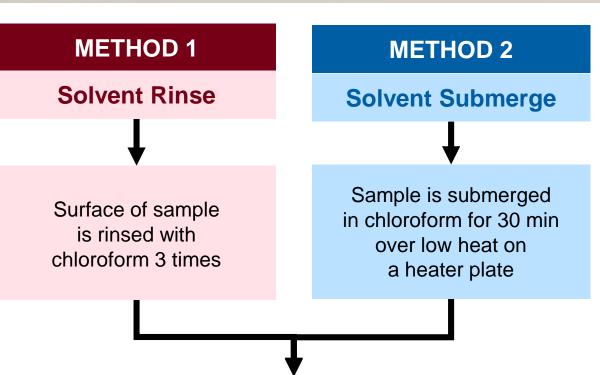
Chemical Analysis Methods



- 2 solvent extraction methods were performed:
 - 6 contaminated samples (3 per method)
 - 2 control samples (1 per method)

Chemical Analysis Objectives:

- Extract and verify the DOP entrapped in MAC
- Extract other adsorbed chemical species (if any), but only those that can be dissolved with chloroform
 - Based on previous studies, these solvent extraction methods are not expected to remove all of the adsorbed contaminants from MAC
- Compare results to calculated gravimetric molecular adsorption capacity



- Remaining Non-Volatile Residue (NVR) is weighed and analyzed qualitatively to provide a general estimation of the relative amounts and types of chemical species
- Pyrolysis-Gas Chromatography/Mass Spectrometry (GC/MS) is performed on NVR from both methods

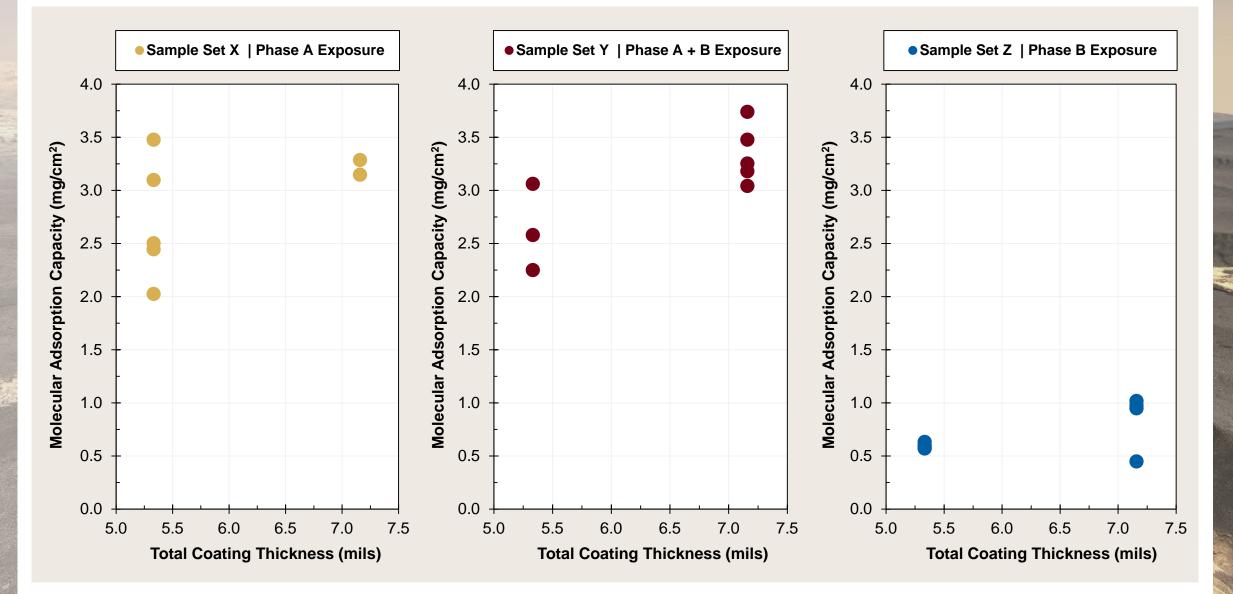


Results & Discussion

- Gravimetric Analysis Results
- Chemical Analysis Results

Gravimetric Analysis Results

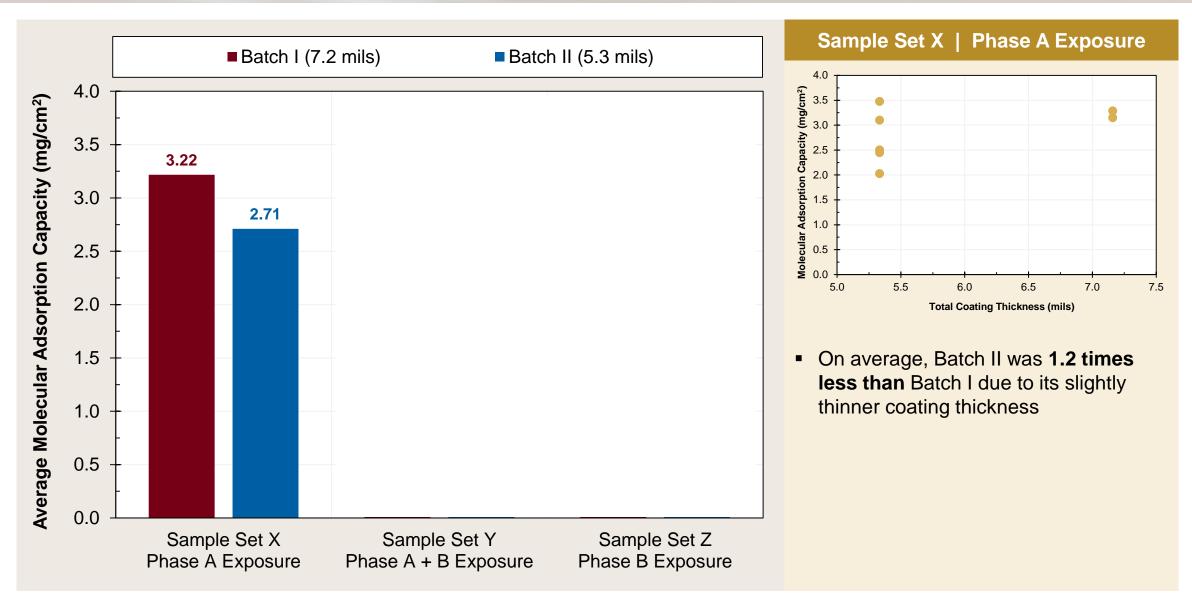




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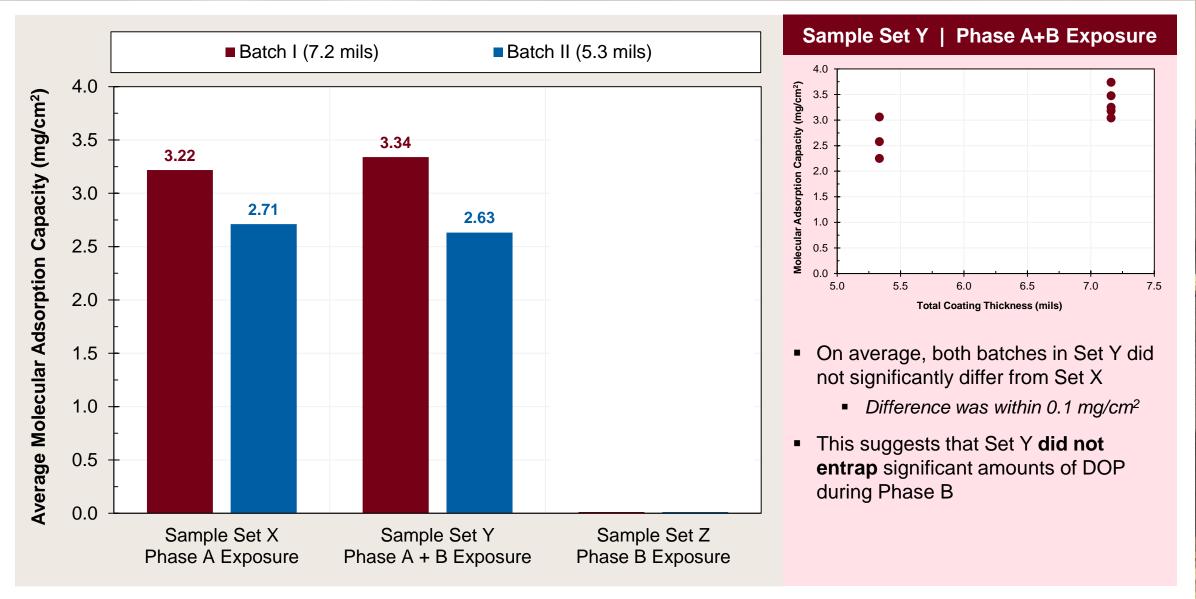
Gravimetric Analysis Results





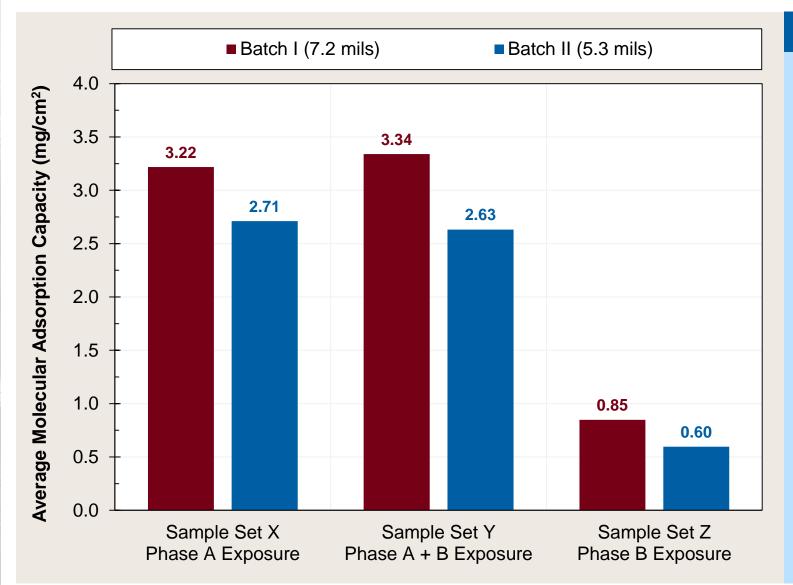
Gravimetric Analysis Results



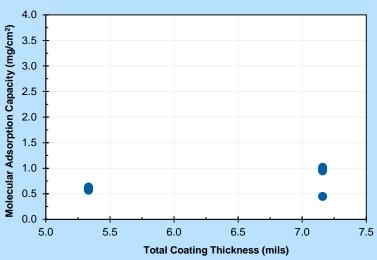


Gravimetric Analysis Results



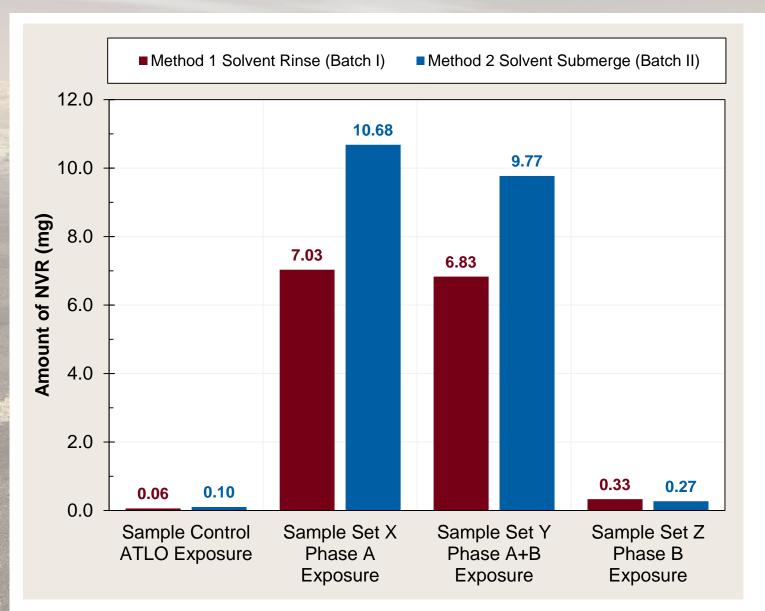


Sample Set Z | Phase B Exposure



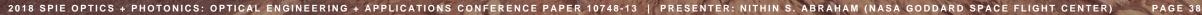
- On average, Set Z was significantly lower by a factor of 4 when compared to Set X and Y
- This suggests that it was unlikely that MAC approached saturation, and that very limited outgassing of DOP occurred in the low vacuum conditions of Phase B
- DOP most likely did not reach its vapor pressure at Phase B conditions; Regardless, some limited adsorption did occur within the allocated time frame of the test, possibly originating from the lingering DOP species that existed in the test apparatus from the previous Phase A exposure

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Set	Phase	Method 1	Method 2
X	Α	117 x	107 x
Y	A+B	114 x	98 x
Z	В	6 x	3 x

- Method 2 resulted in higher amounts of NVR collection than Method 1
- Control (simulated ATLO exposed) samples had the least amount of NVR
- All contaminated samples collected greater amounts of NVR when compared to control samples
- Phase A appears to have the most amount of adsorbed species, whereas Phase B has the least amount of adsorbed species of the contaminated samples
- These trends are consistent with results achieved from the gravimetrically derived molecular adsorption capacity values





METHOD 1 SOLVENT RINSE METHOD 2 SOLVENT SUBMERGE Batch I (Samples I-6, I-8, I-10) - Chemical Analysis, METHOD 1 - Batch II (Samples II-3, II-5, II-10) - Chemical Analysis, METHOD 2 Batch I (Samples I-6, I-8, I-10) - Gravimetric Analysis Batch II (Samples II-3, II-5, II-10) - Gravimetric Analysis 4.0 4.0 Contaminant Adsorption (mg/cm²) - 3.48 Contaminant Adsorption (mg/cm²) 3.5 3.25 3.5 3.06 3.0 3.0 2.5 2.5 2.72 2.49 2.0 1.79 2.0 1.5 1.5 0.95 1.0 1.0 0.60 0.5 0.5 0.08 0.07 0.0 0.0 Sample Set X Sample Set Y Sample Set Z Sample Set X Sample Set Y Sample Set Z Phase B Exposure Phase B Exposure Phase A Exposure Phase A + B Exposure Phase A Exposure Phase A + B Exposure

- 3 samples with similar gravimetric values from each batch were compared to solvent extraction methods
- Solvent submerge method was closer to gravimetric values than solvent rinse methods for all phases
 - Difference between Method 1 and gravimetric values ranged from 0.9 to 1.5 mg/cm²
 - Difference between Method 2 and gravimetric values was slightly less, ranging from 0.5 to 0.8 mg/cm²

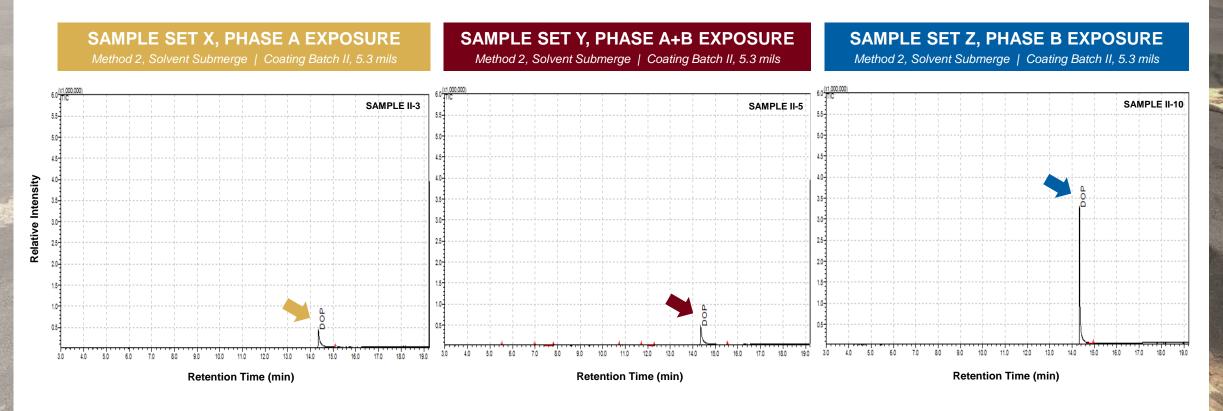


- Pyrolysis GC/MS results for all 6 contaminated samples from any phase exposure, regardless of the method used indicate that the majority of NVR consisted of DOP
- There were no other discernable peaks from the baseline noise

		METHOD 1 Solvent Rinse		METHOD 2 Solvent Submerge	
		Batch I (7.2 mils)		Ba	atch II (5.3 mils)
Exposure	Sample Set	Sample	Chemical Species	Sample	Chemical Species
ATLO	Control	I-14	No detectable organics	II-14	No detectable organics
Phase A	Х	I-6	~ 100 % DOP	II-3	~ 100 % DOP
Phase A + B	Y	I-8	~ 100 % DOP	II-5	~ 100 % DOP
Phase B	Z	I-10	~ 100 % DOP	II-10	~ 100 % DOP

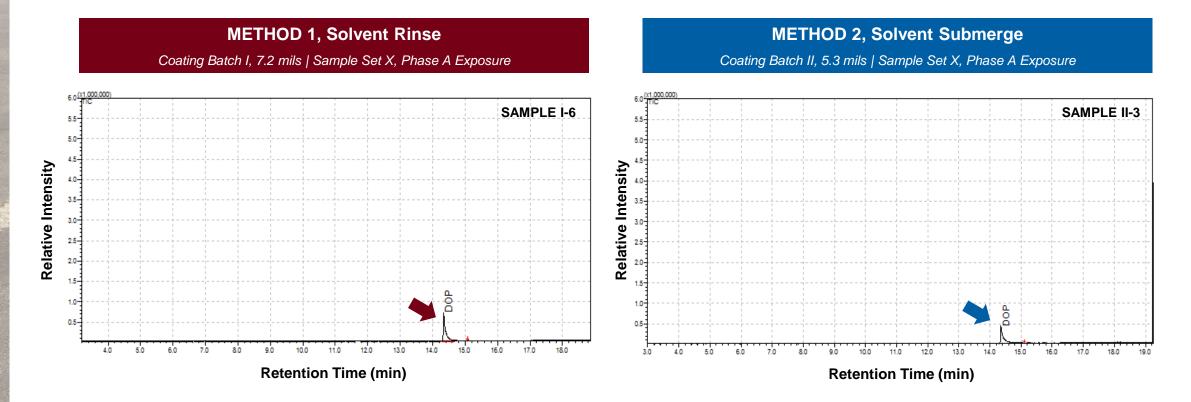


- Shown are the GC/MS plots for the three contaminated samples that were analyzed using Method 2: Solvent Submerge Extraction
 - The relative intensity, or compound abundance, of DOP is the largest in the Phase B exposed sample
 - This is most likely attributed to the low amount of NVR compared to the samples from Sets X and Y



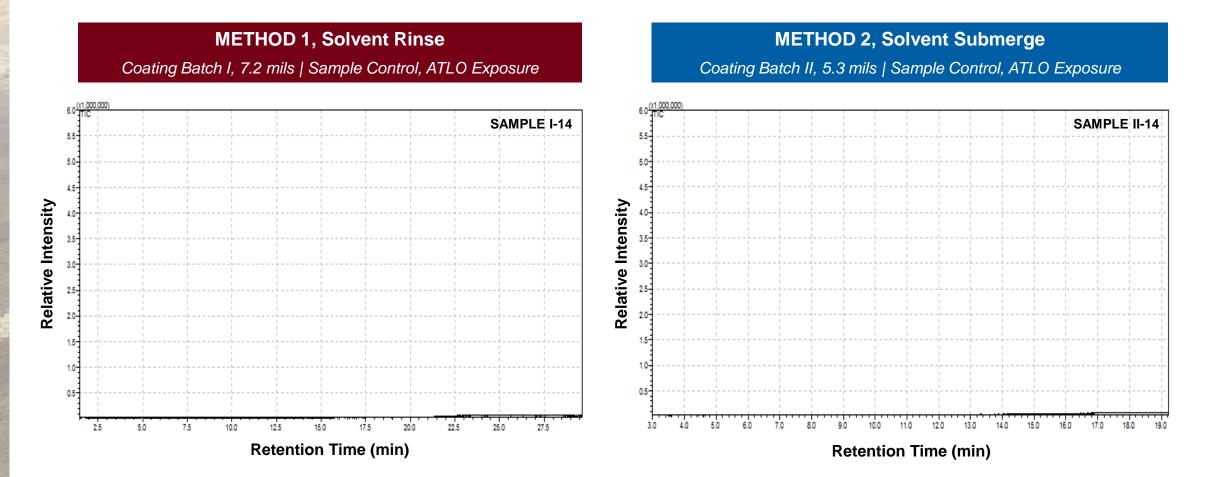


- Similar trends were observed for the samples that were analyzed using Method 1: Solvent Rinse Extraction
- Shown are GC/MS plots that illustrate the similarities in the detection of the DOP peak in two Phase A exposed samples using both solvent extraction methods





Lastly, no detectable organics were present on the control samples for both methods





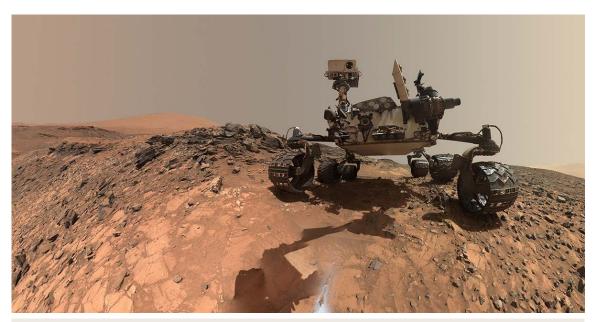
Summary

- Conclusions
- Future Work

Conclusions

NASA

- Preliminary test results confirm that NASA's MAC technology is effective at adsorbing high molecular weight plasticizers, such as DOP, in simulated spaceflight conditions that are representative of the vacuum environment of space, and the Martian atmosphere
- These results support the use of the MAC technology for applications that:
 - Require the reduction of harmful outgassed molecular species within critical hardware components
 - Need to meet challenging molecular contamination requirements



Self-portrait of NASA's Curiosity Mars rover near a rock target called "Buckskin" on Mount Sharp IMAGE CREDIT: NASA/JPL CAL-TECH/MSSS

- Explore other contaminants of interest, such as low molecular weight chemical species
 Berform additional tests in low vacuum carbo
 - Perform additional tests in low vacuum carbon dioxide purged systems
 - Tailor MAC as needed for mission specific applications

FUTURE

National Aeronautics and Space Administration

With the start



ANY QUESTIONS?

IMAGE CREDIT: NASA/JPL CAL-TECH | MARS 2020 ROVER ARTIST'S CONCEPT | LINK: HTTPS://WWW.NASA.GOV/IMAGE-FEATURE/JPL/PIA21635/NASA-S-MARS-2020-ROVER-ARTIST-S-CONCEPT

Acronyms



ATLO	Assembly, Test, and Launch Operations
CAL-TECH	California Institute of Technology
• CO ₂	Carbon Dioxide
 DEHP 	Diethylhexyl Phthalate
 DOP 	Dioctyl Phthalate
 EDGE 	Edge Space Systems
FUV	Far Ultraviolet
 GC/MS 	Gas Chromatography/Mass Spectrometry
 GSFC 	Goddard Space Flight Center
 HSDB 	Hazardous Substances Data Bank
 ICON 	Ionospheric Connection Explorer
■ JPL	Jet Propulsion Laboratory
 JWST 	James Webb Space Telescope
 KSC 	Kennedy Space Center
 MAC 	Molecular Adsorber Coating
 MAC-W 	White Molecular Adsorber Coating
 MEP 	Mars Exploration Program
 MSSS 	Malin Space Science Systems
 NASA 	National Aeronautics and Space Administration
■ NIH	National Institutes of Health
 NIST 	National Institute of Standards and Technology
 NVR 	Non-Volatile Residue
 QCM 	Quartz Crystal Microbalance
■ RH	Relative Humidity
 SGT 	Stinger Ghaffarian Technologies
 UC 	University of California

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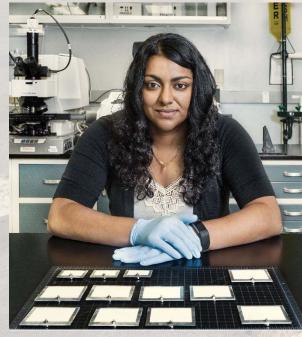
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IMAGE CREDIT: NASA / CHRIS GUNN