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Application of Molecular Adsorber Coatings in Chamber A for the James Webb Space Telescope

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Abstract



As a coating made of highly porous zeolite materials, the Molecular Adsorber Coating (MAC) was developed to capture outgassed molecular contaminants, such as hydrocarbons and silicones. For spaceflight applications, the adsorptive capabilities of the coating can alleviate on-orbit outgassing concerns on or near sensitive surfaces and instruments within the spacecraft. Similarly, this sprayable paint technology has proven to be significantly beneficial for ground-based space applications, in particular, for vacuum chamber environments.

This presentation describes the application of the MAC technology for the James Webb Space Telescope (JWST) at NASA Johnson Space Center (JSC). The coating was used as a mitigation tool to entrap outgassed contaminants, specifically silicone-based diffusion pump oil, from within JSC's cryogenic optical vacuum chamber test facility called Chamber A. This presentation summarizes the background, fabrication, installation, chemical analysis test results, and future plans for the MAC technology, which was effectively used to protect the JWST test equipment from vacuum chamber contamination.

Key words: Molecular Adsorber Coating, zeolite, molecular adsorber, adsorber, adsorption, outgassing, molecular contamination, spaceflight applications, vacuum applications, James Webb Space Telescope, JWST, Chamber A, DC-704, DC704, diffusion pump oil, silicones, sprayable paint technology, coatings, getters, passive getter

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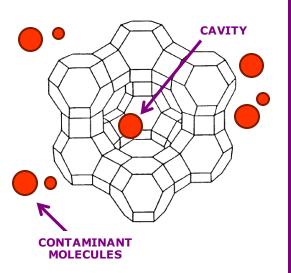


Molecular Adsorber Coating

Molecular Adsorber Coating

- The Molecular Adsorber Coating (MAC) was developed by NASA Goddard Space Flight Center (GSFC)
- MAC is a sprayable, zeolite-based and highly porous coatings technology that was designed to **passively** capture outgassed contaminants
- MAC is available in white and black variations
 - White Molecular Adsorber Coating, GSFC MAC-W
 - Black Molecular Adsorber Coating, GSFC MAC-B
- Examples of molecular contaminants include high molecular weight chemical species, such as:
 - Hydrocarbons
 - Phthalates
 - Palmitates
 - Esters
 - Silicones

- Sources of molecular contaminants are products of outgassing from materials found within the spacecraft, such as:
 - Plastics
 - Adhesives
 - Lubricants
 - Epoxies
 - Potting Compounds





Molecular Adsorber Coating



- MAC was designed to be used as a contamination control mitigation method to address material outgassing concerns on or near sensitive surfaces and instruments:
 - Inside instrument cavities, electronics boxes, detectors, and baffles
 - Near components such as telescopes, cameras, lasers, mirrors, and optics
- MAC reduces the risk of on-orbit molecular contamination from degrading the performance of spaceflight hardware
- Through GSFC's Internal Research and Development (IRAD) program, significant MAC testing and demonstration efforts were performed in relevant environments (i.e. vacuum) for spaceflight applications
 - Adsorptive Capabilities
 - Thermal/Optical Properties
 - Adhesion Performance
 - Thermal Stability
 - Particulate Characteristics

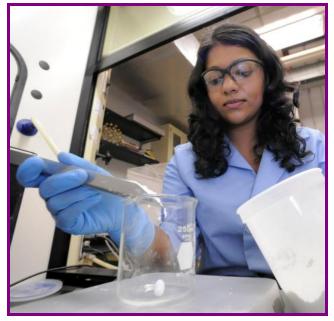


Photo Credit: NASA/Pat Izzo

Molecular Adsorber Coating



















MAC is planned for use on the following NASA missions as an **on-orbit passive getter** material to mitigate outgassing concerns within the spacecraft or instrument:

FLIGHT-BASED SPACE APPLICATIONS:

- **ICON**, Ionospheric Connection Explorer -- 2017
- **GEDI**, Global Ecosystem Dynamics Investigation Lidar -- 2018
- MAC has been used on the following NASA missions as a Ground Support Equipment (GSE) passive getter material during vacuum chamber tests to mitigate outgassing concerns:

GROUND-BASED SPACE APPLICATIONS:

- **JWST**, James Webb Space Telescope *-- 2014, 2015, 2016, 2017*
- **GOLD**, Global-scale Observations of the Limb and Disk -- 2017
- **ICESat-2**, Ice, Cloud, and Land Elevation Satellite-2, -- 2017
- ATLAS, Advanced Topographic Laser Altimeter System -- 2016
- **GEDI**, Global Ecosystem Dynamics Investigation Lidar -- 2016
- NICER, Neutron star Interior Composition Explorer -- 2015
- MMS, Magnetosphere Multiscale Mission -- 2014

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- Chamber A was originally built in 1965 as part of the Space Environment Simulation Laboratory at NASA Johnson Space Center (JSC) in Houston, Texas
- This vacuum chamber has a diameter of 55 feet, is 90 feet tall, and has an interior volume of 400,000 cubic feet
- It is best known for space environmental testing of the space capsules and equipment for NASA's **Apollo missions** with and without the mission crew

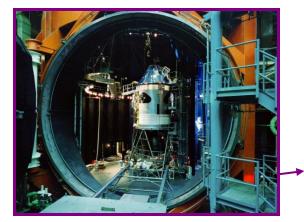


Photo Credit: NASA

Apollo command and service module 2TV-1 in Chamber A for a full mission duration vacuum test in 1968



Photo Credit: NASA



- Chamber A has experienced significant upgrades to accommodate the arrival and testing of the James Webb Space Telescope (JWST) in a space simulation environment
- These recent upgrades include but are not limited to the following:
 - Liquid Helium Shroud capable of reaching cryogenic temperatures as low as -262°C to simulate the extremely cold environment that the telescope will be exposed to
 - Clean Room retrofitted to the test facility
 - Pumping Systems consists of ultraclean hydrocarbon-free high vacuum pumping systems

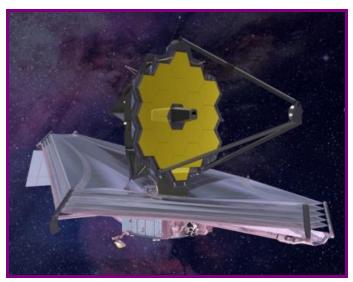


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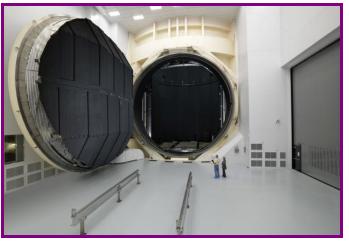


Photo Credit: NASA/Robert Markowitz & Bill Stafford



- Chamber A is categorized as one of the largest high vacuum, cryogenic optical test chambers in the world
- The JWST Pathfinder model has been used for practicing ambient and vacuum testing that will be performed on the flight telescope
- Optical Ground Support Equipment (OGSE) has also been used during these testing efforts
- Shown in this photograph is the full-scale JWST Pathfinder model entering the recently upgraded Chamber A for cryogenic vacuum testing in 2015

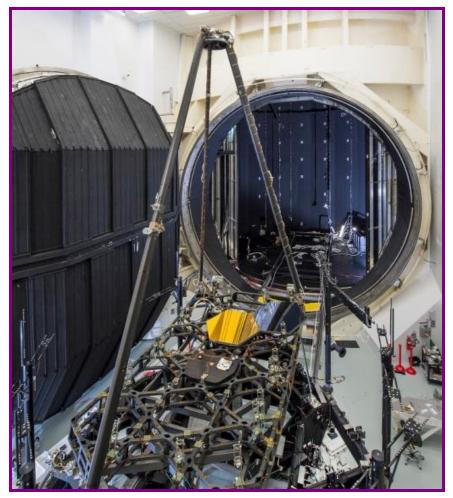
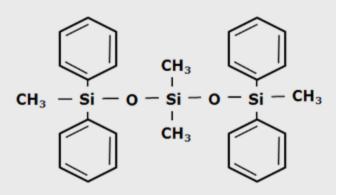


Photo Credit: NASA/Chris Gunn



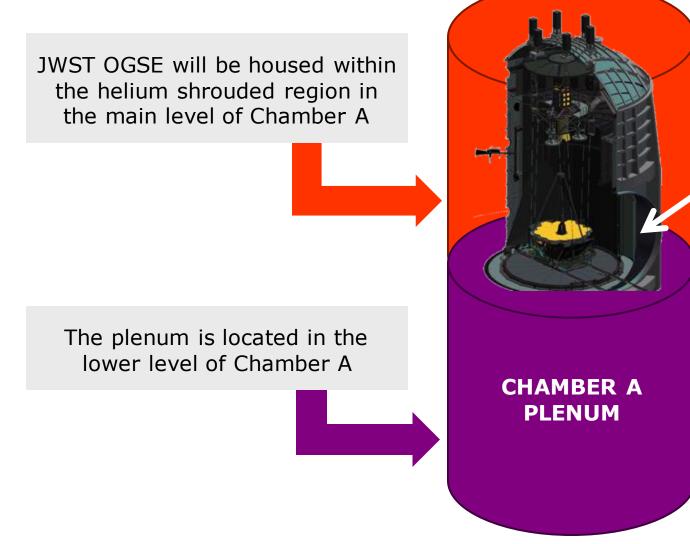
- Due to Chamber A's history prior to JWST, molecular contaminants, such as silicones, still remain within the chamber plenum
- In particular, one of the main contaminant sources was the residual silicone from **Dow Corning® 704**
 - Also known as DC-704
 - Single component, silicone-based diffusion pump oil
 - Commonly used for high vacuum systems
 - Designed to work well with diffusion pumps due to its properties
 - Low vapor pressure
 - Low volatility
 - Frequently used in Chamber A for Apollo mission testing



Molecular Structure of DC-704 TETRAMETHYL TETRAPHENYL TRISILOXANE







CRYOGENIC HELIUM SHROUD



- Silicone-based contaminants are known to outgas at ambient temperatures, and are extremely difficult to remove
- If not properly mitigated, the outgassing effects of DC-704 can accumulate on contamination sensitive surfaces during vacuum testing

Mitigation Method 1	Mitigation Method 2					
Many cleaning efforts were performed by the JWST Contamination Control team to remove DC-704 from the plenum	MAC was proposed to be placed at strategic locations around the chamber to capture the remaining DC-704 during the vacuum tests					
 These cleaning efforts were successful in reducing the silicone levels significantly However, there was still some residual DC-704 	MAC adds an extra level of precaution by cost effectively lowering the contamination risk and preventing harmful outgassed species originating from within the chamber environment from migrating and depositing onto sensitive surfaces					

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Fabrication & Installation

- MAC Barn Door Panels
- MAC Plenum Samples

Fabrication & Installation

MAC was used as a mitigation technique to capture molecular contamination (i.e. DC-704) within the Chamber A vacuum test facility during the three Pathfinder tests and is also, currently planned for use during the final cryogenic vacuum test of JWST at NASA JSC this summer

Test Category		Test Name	Acronym		
	1	Optical Ground Support Equipment Test 1	OGSE-1		
PATHFINDER	2	2 Optical Ground Support Equipment Test 2			
	3	Thermal Pathfinder Test	TPF		
JWST	4	Optical Telescope Element and Integrated Science Cryo-Vacuum Test	OTIS CV		

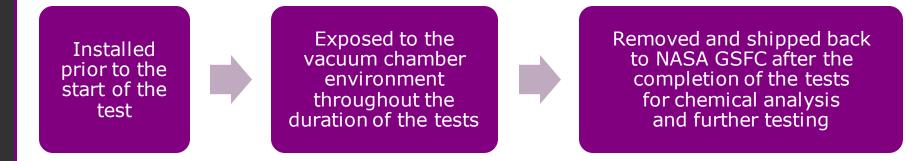
ю	MAY	JUN	JUL	AUG	SEP	ост	NOV
201	OGSE-1 Test lasted about ~ 30 days				OGSE-2 Test lasted about		
l6	МАҮ	JUN	JUL	AUG	SEP	ост	NOV
2016					TPF Test lasted about ~ 50 days		
N	ΜΑΥ	JUN	JUL	AUG	SEP	ост	NOV
2017			OTIS CV Test will last about	~ 90 days			

Fabrication & Installation

- NASA GSFC custom designed and fabricated over 200 MAC samples in various sizes and substrate materials from 2014 to 2017
- A set of MAC samples was created for each location in Chamber A
 - These two strategic locations were selected to capture vacuum chamber contamination and prevent them from entering the chamber environment where the test equipment is housed

	SAMPLE SET	PLACEMENT LOCATION
1	MAC Barn Door Panels	Main Level of Chamber A
2	MAC Plenum Samples	Plenum of Chamber A

For each of the tests, the two sets of MAC samples were:



Fabrication & Installation

 Below is the timeline of the fabrication and installation of the MAC samples for the JWST tests in Chamber A:

	LI	EGEND	
Fabrication	Installation	Test	OGSE-1
Fabrication	Installation	Test	OGSE-2
Fabrication	Installation	Test	TPF
Fabrication	Installation	Test	OTIS CV

	JAN	FEB	MAR	APR	ΜΑΥ	JUN	JUL	AUG	SEP	ост	NOV	DEC
2014												

	JAN	FEB	MAR	APR	ΜΑΥ	JUN	JUL	AUG	SEP	ост	NOV	DEC
2015												

	JAN	FEB	MAR	APR	ΜΑΥ	JUN	JUL	AUG	SEP	ост	NOV	DEC
2016												

	JAN	FEB	MAR	APR	ΜΑΥ	JUN	JUL	AUG	SEP	ост	NOV	DEC
2017												

MAC Barn Door Panels





Photo Credit: NASA/Chris Gunn

Base perimeter along barn door on external wall of cryogenic helium shroud

Main Level of Chamber A

- "Barn Door" Cryogenic Helium Shroud
 - During testing, the cryogenic helium shroud reaches temperatures as cold as -241°C and as warm as room temperature
 - The internal wall of the shroud is painted with a black thermal/optical coating
 - The external wall of the shroud is made of an aluminum finish

Proposed MAC Location

- MAC samples were placed against the base of the external wall on the shroud to cover some of the exposed gaps near the perimeter along the barn doors of Chamber A
 - This proposed MAC location helps capture vacuum chamber contaminants that may have migrated from the plenum and prevent them from depositing on the sensitive JWST test equipment housed internal to the cryogenic helium shroud

NASA

MAC Barn Door Panels

Subs	Substrate Information									
Thickness	0.0625 inch									
Material	6061-T6 Aluminum Alloy									
Height	6 inch									
Width	11 - 46 inch (varies)									
Border Edge	0.50 - 0.75 inch (varies)									

6 inch by 12 inch black MAC and white MAC barn door panels



Photo Credit: NASA/Chris Gunn

Installation of a white MAC barn door panel on the external wall of the cryogenic helium shroud covering the gap along the base perimeter of Chamber A

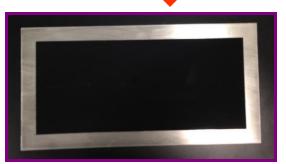


Photo Credit: NASA

Photo Credit: NASA

A border was implemented on samples to reduce possible coating damage due to handling and installation activities

MAC Barn Door Panels



- Fabricated a total of **122** (~ 107 ft²) MAC barn door panels for placement at the main level of Chamber A (i.e. barn doors) during each of the tests, including the last test
- Installed a total of **78** (~70 ft²) of these samples for the first three tests
 - This number <u>excludes</u> the samples to be installed during the last test (i.e. OTIS CV)

	CHAMBER A	TESTS	FABRIC		INSTALLATION			
#	Test Name	Installation Date	Sample Quantity	Coating Area (ft ²)	Sample Quantity	Coating Area (ft ²)		
1	OGSE-1	May 2015	65	57	29	29		
2	OGSE-2	September 2015	60	57	24	21		
3	TPF	September 2016	29	25	25	20		
4	OTIS CV	July 2017 *	28	25	TBD	TBD		

* Tentatively Planned Date

TBD = TO BE DETERMINED

MAC Plenum Samples



Lower Level of Chamber A

Plenum of Chamber A

- Encompasses a large volume
- Located beneath the chamber
- Classified as a confined space area
- During testing, the plenum contamination (i.e. DC-704) may migrate towards the main level cryogenic helium shroud, and ultimately find a path into the chamber where the sensitive test equipment is placed



 MAC samples were placed against the walls of the plenum to capture contamination at its source



Photo Credit: NASA/Chris Gunn



As shown, NASA GSFC installed MAC samples on the walls of the plenum in the Chamber A test facility prior to the start of the tests

MAC Plenum Samples



 Variety of flexible substrates were explored for the plenum samples

Aluminum Foil

- Used primarily during OGSE-1 tests
- At times, handling was a challenge due to the low tear resistance of the material

Kapton

Limited use during OGSE-1 tests

Aluminum Laminate Materials

- Used primarily during OGSE-2 & TPF
- NepTape® 1026
 - Typically used in industry as a second shield in multi-shielded Local Area Network (LAN) coaxial cables
 - Its layered structure allows the material to exhibit a higher tear resistance, and consequently is more flexible and easier to handle than aluminum foil coated MAC samples



Photo Credit: NASA/Chris Gunn

A thin border less than 0.25 inch was implemented on the plenum samples to avoid direct contact with the coating during handling and installation. This border provided a location to adhere Kapton tape to during its placement on the plenum wall.

Construction of NepTape® 1026

1 mil Aluminum Foil

0.92 mil Polyester Film

1 mil Aluminum Foil

MAC Plenum Samples



- Fabricated a total of 81 (~ 140 ft²) MAC plenum samples for placement at the plenum of Chamber A (i.e. plenum walls) during each of the tests, including the last test
- Installed a total of **59** (~102 ft²) of these samples for the first three tests
 - This number <u>excludes</u> the samples to be installed during the last test (i.e. OTIS CV)

	CHAMBER A	TESTS	FABRIC		INSTALLATION		
#	Test Name	Installation Date	Sample Quantity	Coating Area (ft ²)	Sample Quantity	Coating Area (ft ²)	
1	OGSE-1	May 2015	33	60	26	38	
2	OGSE-2	September 2015	33		7	22	
3	TPF	September 2016	26	42	26	42	
4	OTIS CV	July 2017 *	22	38	TBD	TBD	

* Tentatively Planned Date

TBD = TO BE DETERMINED

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Chemical Analysis

- Vacuum Desorption Bake-Out Method
- Sample Solvent Rinse Method

Chemical Analysis



Chemical analysis was performed on some of the contaminated MAC samples from the three Chamber A tests (i.e. OGSE-1, OGSE-2, and TPF) using the following two methods:

	Method Name	Method Description	Chemical Analysis	
1	Vacuum Desorption Bake-Out Method (VDBM)	Adsorbed contaminants on the coating are removed via high temperature desorption in vacuum	FTIR* Pyrolysis GC/MS**	
2	Sample Solvent Rinse Method (SSRM)	Adsorbed contaminants on the coating are removed via a solvent rinse of the coating surface	FTIR Pyrolysis GC/MS	

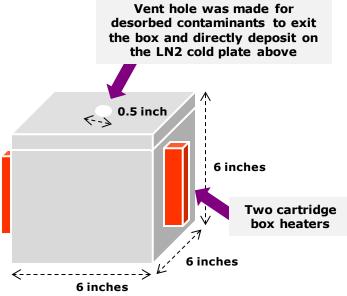
* Fourier Transform Infrared Spectroscopy (FTIR)

** Pyrolysis-Gas Chromatography/Mass Spectrometry (GC/MS)



Vacuum Desorption Bake-Out Method

- In industry, a common practice used to regenerate microporous materials, such as zeolites, involves high temperature vacuum bake-outs between 175 - 315°C
- Performed on a single aluminum foil white MAC sample that had been deployed in the Chamber A plenum environment during OGSE-1 (May 2015)
 - Small piece (4 inches by 3.5 inches) was cut from Sample PW 13
 - Total coating thickness of 7 mils
- Constructed a bake-out box
 - Substrate Type: 6061-T6 aluminum alloy
 - Substrate Thickness: 0.050 inches
 - Dimensions of 216 cubic inch





Vacuum Desorption Bake-Out Method

- Baseline run without the MAC plenum sample was performed to determine the chamber background contamination that would deposit on the Liquid Nitrogen (LN2) cold plate
- Test configuration was jacketed with aluminum foil during testing to limit the chamber background deposition on the cold plate

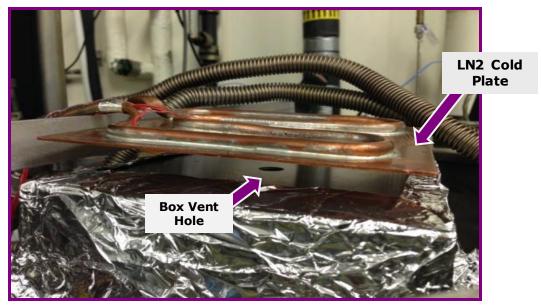
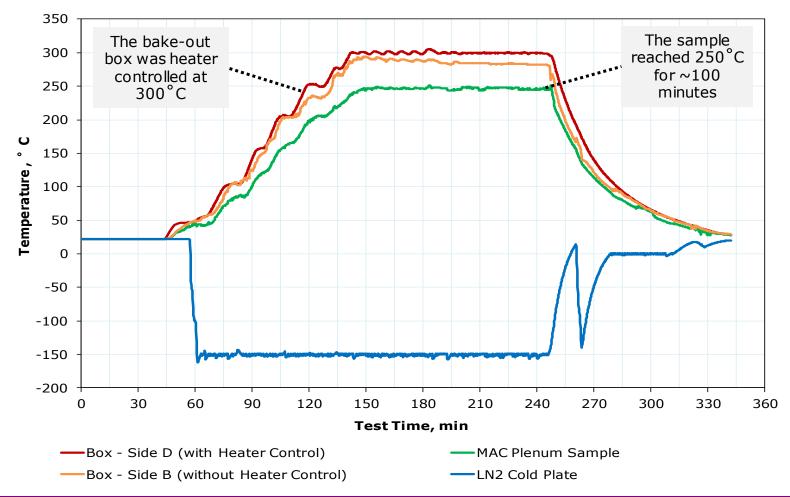


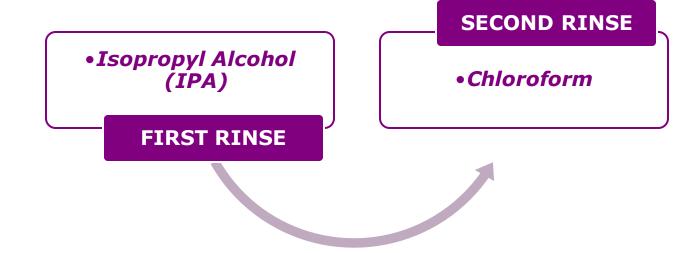
Photo Credit: NASA

Temperature Profile for Vacuum Desorption Test

The MAC plenum sample PW 13 was tested inside the constructed bake-out Box for a duration of 5 hours in high vacuum around 3.0×10^{-5} Torr



 The cold plate was immediately rinsed twice with two solvents after repressing the vacuum chamber



- The two rinsates were then transferred to separate pre-weighed dishes and allowed to evaporate to dryness
- The remaining Non-Volatile Residue (NVR) was weighed and analyzed using two chemical analysis techniques:
 - Fourier Transform Infrared Spectroscopy (FTIR)
 - Pyrolysis-Gas Chromatography /Mass Spectrometry (GC/MS)

- Results demonstrate that chloroform removed over 5.5 times the amount removed from the initial rinse with IPA
 - This suggests that IPA does not sufficiently remove the cold plate contaminants that were collected from the sample bake-out at 250°C
- IPA rinse results show DC-704 diffusion pump oil as the most prevalent NVR species
 - This suggests that most of the DC-704 from the cold plate was removed from the initial rinse
- Chemical species found during the chamber background and from the solvent itself were subtracted from the results shown below:

Rinse	Solvent	NVR (mg)	GC/MS Analysis
First	Isopropyl Alcohol	3.58 ± 0.04	 DC-704 diffusion pump oil (80%) Hydrocarbons (20%)
Second	Chloroform	19.78 ± 0.04	Hydrocarbons (97%)Other (3%)

NASA

Sample Solvent Rinse Method

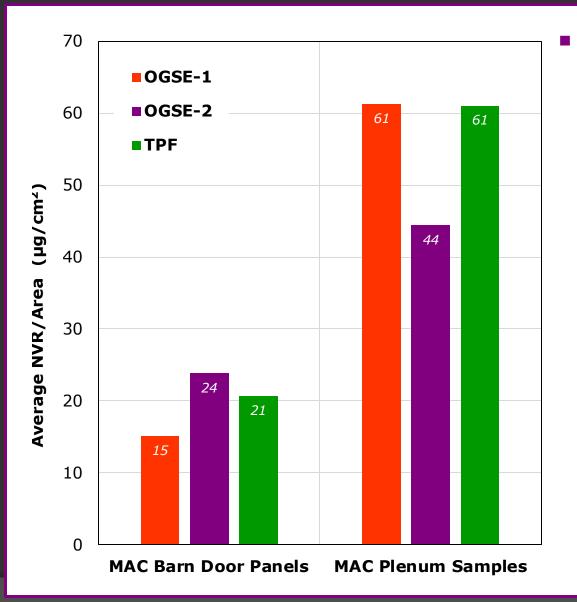
- Involves directly rinsing the MAC surface with a solvent
 - This is a destructive test that damages the coating surface
- Qualitatively provides a general approximation of the chemical species that are bound to the surface of the coating and can be dissolved using the selected solvent
 - It does not remove all the contaminants that are entrapped on the porous structure of MAC
 - It does not provide a complete representation to quantitatively assess the exact amounts and types of contaminants that were collected on MAC
- Analysis techniques are similar to VDBM:
 - Rinsates from the samples were collected and allowed to evaporate to dryness in separate pre-weighed dishes
 - Remaining NVR was weighed and analyzed using FTIR and pyrolysis-GC/MS

TEST	SAMPLES		
OGSE-1	13		
OGSE-2	8		
TPF	18		

Selected MAC barn door panels and MAC plenum samples that were contaminated during each of the three completed cryogenic vacuum tests were rinsed with **CHLOROFORM**

 Below is a summary of the NVR results for a single rinse of chloroform on the MAC samples exposed to the main level (i.e. barn doors) and lower level (i.e. plenum) of Chamber A during the three JWST cryogenic vacuum tests

	MAC Barn Door Panels		MAC Plenum Samples			
Chamber A Test Name	Samples Analyzed	Range of NVR/Area	Average NVR/Area	Samples Analyzed	Range of NVR/Area	Average NVR/Area
OGSE-1 Installed May 2015	7	6 to 20 µg/cm ²	15.16 ± 0.09 μg/cm ²	6	21 to 101 µg/cm ²	61.27 ± 0.09 μg/cm ²
OGSE-2 Installed September 2015	4	14 to 31 µg/cm ²	23.80 ± 0.09 µg/cm ²	4	22 to 59 µg/cm ²	44.40 ± 0.44 μg/cm ²
TPF Installed September 2016	9	11 to 46 µg/cm ²	20.71 ± 0.09 µg/cm ²	9	29 to 84 µg/cm ²	61.06 ± 0.03 µg/cm ²



NVR results for a single rinse of chloroform were greater for the MAC plenum samples when compared to the MAC barn door panels

OGSE-1

On average, the plenum samples collected about **4 times** more than the barn door samples for the first test

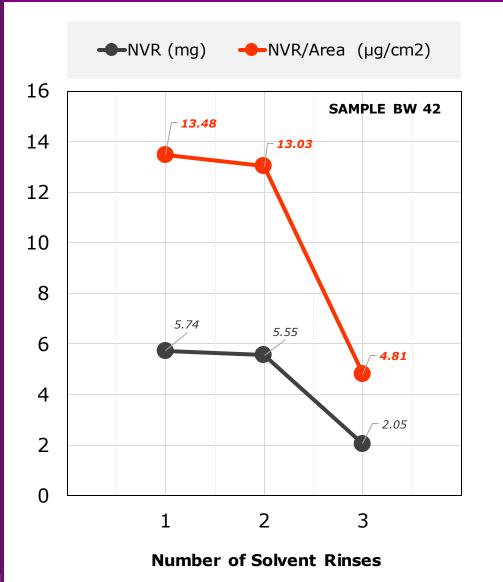
OGSE-2

On average, the plenum samples collected about **2 times** more than the barn door samples for the second test

TPF

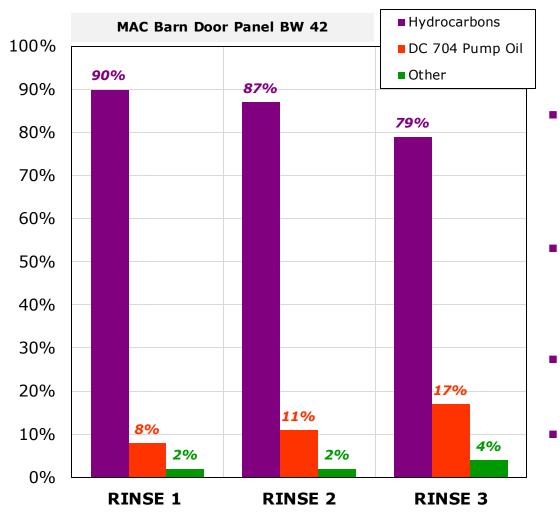
On average, the plenum samples collected about **3 times** more than the barn door samples for the third test





- One MAC barn door panel from OGSE-1 was rinsed 3 times to determine how much additional NVR is removed with multiple rinses of chloroform
- Results confirmed that a single solvent rinse **does not** fully remove the chemically adsorbed contaminants
 - Two consecutive rinses remove the same amount of NVR each time
 - A third rinse showed a
 64% reduction in NVR than the first two rinses

Multiple Solvent Rinse Results on MAC Barn Door Panel from OGSE-1



The "Other" category consists of:

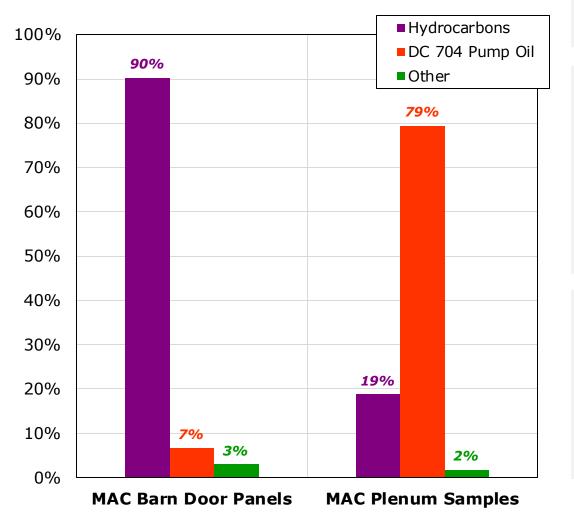
- Di(2-ethylhexyl) phthalate (plasticizer)
- Methyl phenyl silicone
- DC 705 pump oil
- The significance of this method is to **qualitatively** determine the types and relative amounts of chemical species that were detected from a solvent rinse of the coating surface
- Each consecutive rinse displays the <u>same</u> chemical species:
 - Hydrocarbons

DC-704

- With each repeated rinse, there is a gradual **reduction of hydrocarbons** from 90% to 79%
- With each repeated rinse, there is a gradual **increase of DC-704** present from 8% to 17%



Average Single Solvent Rinse Results from the OGSE-1 Test in May 2015



The "Other" category consists of:

- Di(2-ethylhexyl) phthalate (plasticizer)
- Methyl phenyl silicone
- DC 705 pump oil

MAC BARN DOOR PANELS

- High levels of hydrocarbons
- Low levels of DC-704

As expected, results show the migration of DC-704 to the main level of Chamber A near the helium shroud and barn door

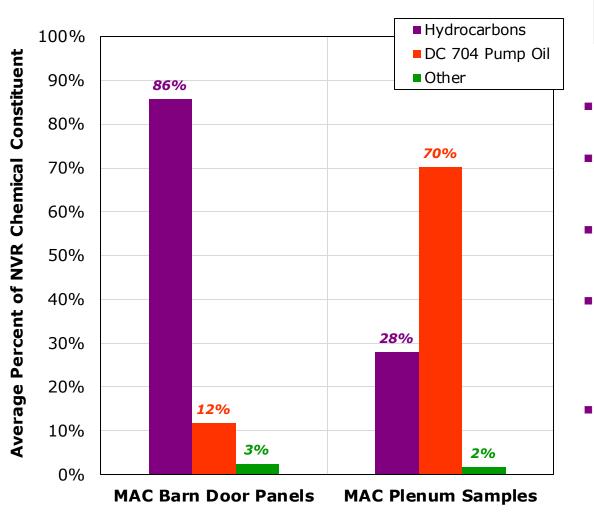
MAC PLENUM SAMPLES

- High levels of DC-704
- Low levels of hydrocarbons

This is predictable considering that the plenum is the source of the silicone contamination

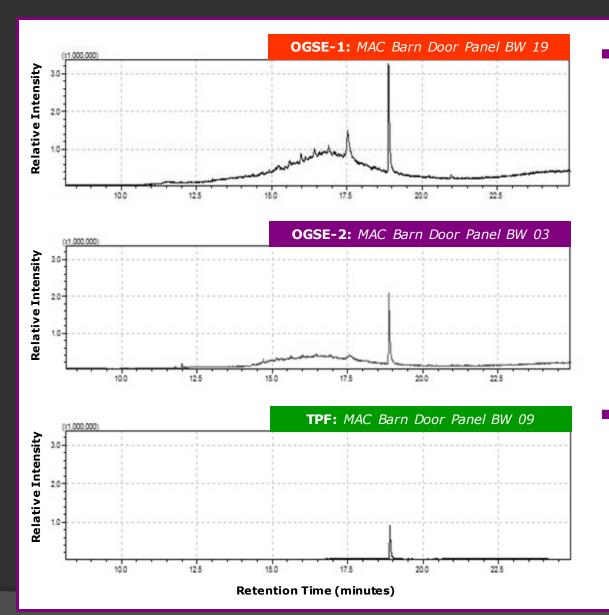


Average Single Solvent Rinse Results from the OGSE-2 Test in September 2015



The "Other" category consists of:

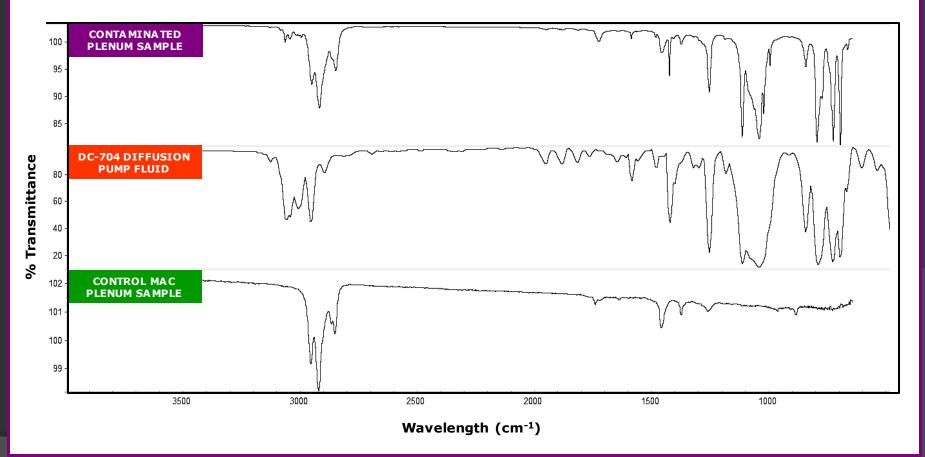
- Di(2-ethylhexyl) phthalate (plasticizer)
- Methyl phenyl silicone
- DC 705 pump oil
- Same trends were observed for the MAC samples from OGSE-2
- The main contaminant adsorbed by the MAC barn door panels were hydrocarbons
- The main contaminant adsorbed by the MAC plenum samples was DC-704 pump oil
- There was a reduction of about 12% of DC-704 adsorbed for the OGSE-2 MAC plenum samples compared to the OGSE-1 MAC plenum samples
- The relative amounts of hydrocarbons adsorbed on the OGSE-2 MAC barn door panels were slightly lower by about 5%



- A comparison of the chemical analysis of the MAC barn door panels from the three tests illustrate that the **relative intensity** (or compound abundance) of DC-704 diffusion pump oil and hydrocarbons that are detected and/or rinsed from the contaminated coating is **decreasing** with each consecutive Chamber A test
- Similar decreasing trends of the contaminants were observed for the MAC plenum samples from each of the three tests

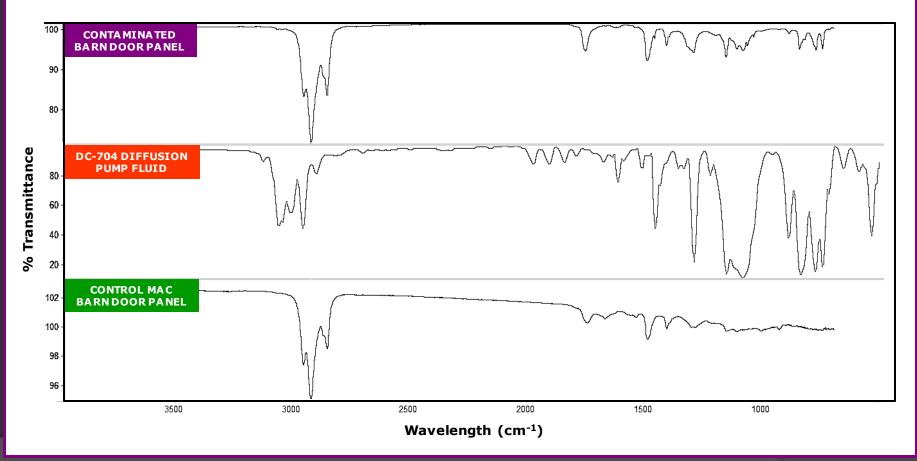
OGSE-1: FTIR Spectra Comparison for MAC Plenum Sample

 The spectrum of the contaminated MAC plenum sample (PW 14) has a very similar resemblance (or match) to the spectrum of DC-704, particularly in the 500 to 2000 cm⁻¹ wavelength range



OGSE-1: FTIR Spectra Comparison for MAC Barn Door Panel

 The spectrum of the contaminated MAC barn door panel (BW 41) does not have a similar resemblance (or match) to the spectrum of DC-704, particularly in the 500 to 2000 cm⁻¹ wavelength range



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Conclusions

Closing Remarks



Mitigator and Indicator

- MAC can serve not only as a contaminant risk mitigator, but also as a contaminant indicator by identifying the molecular contamination risks in the chamber that may not be collected on post-vacuum witness foils
 - For instance, molecular species that strike the coating surface are captured and less likely to be released during warm-up to ambient conditions
- Results from laboratory testing and chemical analysis methods have proven that MAC continues to be effective in removing molecular contamination, in particular the outgassed silicone-based diffusion pump oil (DC-704)
 - As a result, this reduces the risk of molecular contamination transfer from the vacuum chamber to the test equipment

Coating Particulation

 Particulation related anomalies from MAC were <u>not</u> observed during post-test chamber inspections

Future Plans



- Continued use of MAC is planned for the final cryogenic vacuum testing of JWST in Chamber A at NASA JSC this summer
- Future work includes:
 - Fine tuning the chemical analysis methods for determining the amount of contaminants adsorbed on the coating, such as:
 - Exploring different solvents for rinsing
 - Improving vacuum desorption tests for greater test efficiency
 - Investigating other tear resistant substrates
 - Performing more analysis on future samples
 - Continuing to expand upon the benefits of using MAC for vacuum chamber applications



Photo Credit: NASA/Chris Gunn

JWST sits inside Chamber A at NASA JSC for the final OTIS Cryo-Vacuum Test

Acknowledgements





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