National Aeronautics and Space Administration

Using NASA's Molecular Adsorber Coating technology during thermal vacuum testing to protect critical laser flight optics on the ATLAS instrument

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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 was designed to address molecular contamination concerns on or near sensitive surfaces and instruments within the spacecraft for flight or ground-based applications in vacuum conditions. This paper will discuss the use of NASA's MAC technology to isolate and protect the critical laser flight optics of the Advanced Topographic Laser Altimeter System (ATLAS) instrument on the Ice, Cloud, and land Elevation Satellite-2 (ICESat-2). MAC was strategically used during thermal vacuum (TVAC) testing efforts to reduce the risk of contaminating the laser optical components from non-baked items and other unknown outgassing sources from the chamber environment. This paper summarizes the design and implementation efforts, and the chemical analysis of the MAC samples that were used during two recent TVAC tests for the ICESat-2/ATLAS mission.

- **Keywords:** *molecular adsorber coating, molecular adsorbers, getters, MAC, zeolite, coatings technology, outgassing, molecular contamination, vacuum contamination, thermal vacuum, TVAC, ICESat-2, ATLAS, vacuum chambers, chamber facility, flight optics, laser optics, optical components*

Presentation Outline

INTRODUCTION	▪ ATLAS Instrument ▪ Molecular Adsorber Coatings
APPROACH	▪ Purpose ▪ Sample Fabrication & Exposure ▪ Sample Location ▪ Sample Configuration ▪ Temperature Profile
TEST METHODS	▪ Solvent Rinse Methods ▪ Chemical Analysis Methods
RESULTS & DISCUSSION	▪ Solvent Rinse Results ▪ Molecular Adsorption Capacity ▪ Chemical Analysis Results ▪ Comparison to Contamination Monitoring Methods
SUMMARY	▪ Conclusions ▪ Future Work

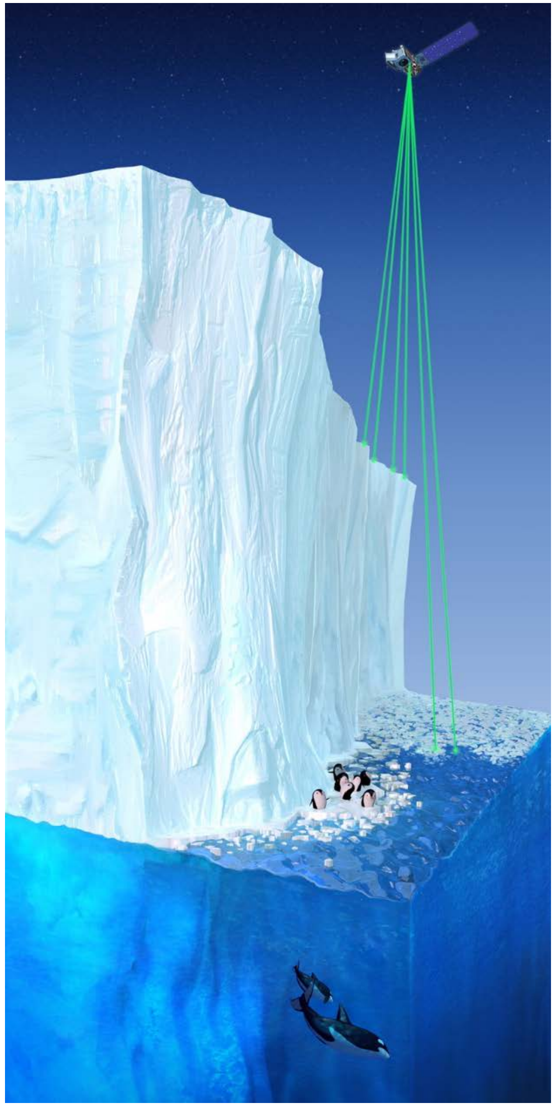


IMAGE CREDIT: NASA/GSFC

Introduction

- ATLAS Instrument
- Molecular Adsorber Coatings

ATLAS Instrument

- **ICESat-2**
  - Ice, Cloud and land Elevation Satellite-2
  - NASA mission that will study the **cryosphere** to investigate the changes in the Earth's frozen and icy regions due to the warming climate
- **ATLAS**
  - Advanced Topographic Laser Altimeter System
  - Built by **NASA Goddard Space Flight Center**
  - Sole instrument on ICESat-2 spacecraft that will measure the height of:
    - *Glaciers*
    - *Ice sheets*
    - *Sea ice*
    - *Rain forests*
    - *Deserts*
    - *Urban areas*

ICESat-2
ICE, CLOUD, AND LAND ELEVATION SATELLITE-2

Advanced Topographic Laser Altimeter System

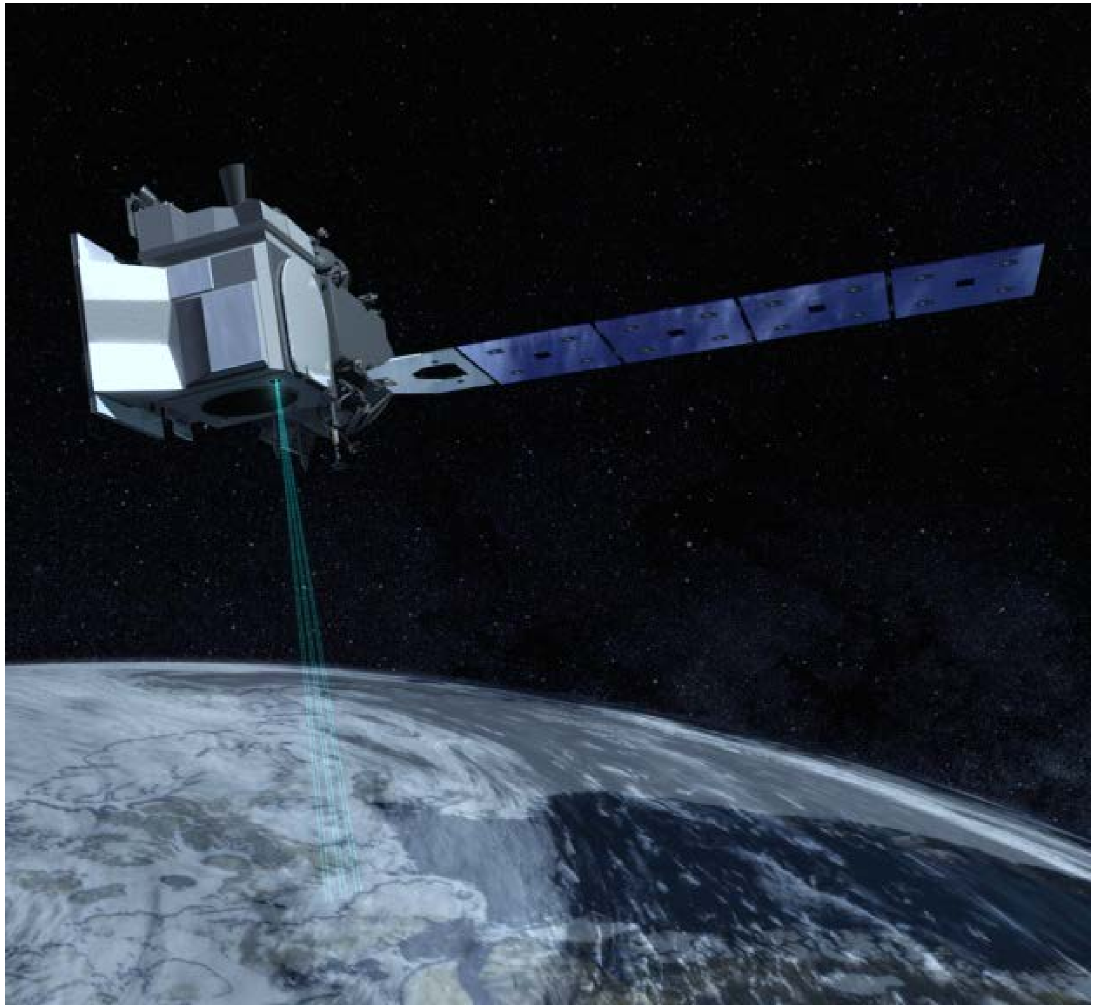


IMAGE CREDIT: NASA/GSFC

ATLAS Instrument

- **Instrument's three main tasks are to:**
  - Send laser pulses of **visible green light** to Earth
  - Catch returning photons using its precisely aligned beryllium telescope
  - Record travel time of each returned photon to calculate distance between the spacecraft and Earth

Wavelength	532 nm
Beams	Single laser split into 6 beams & arranged into 3 pairs
Travel Rate	10,000 pulses/sec
Measurement	Taken every 0.7 m along ground path
Photons Sent	About 200 trillion
Photons Return	About a dozen

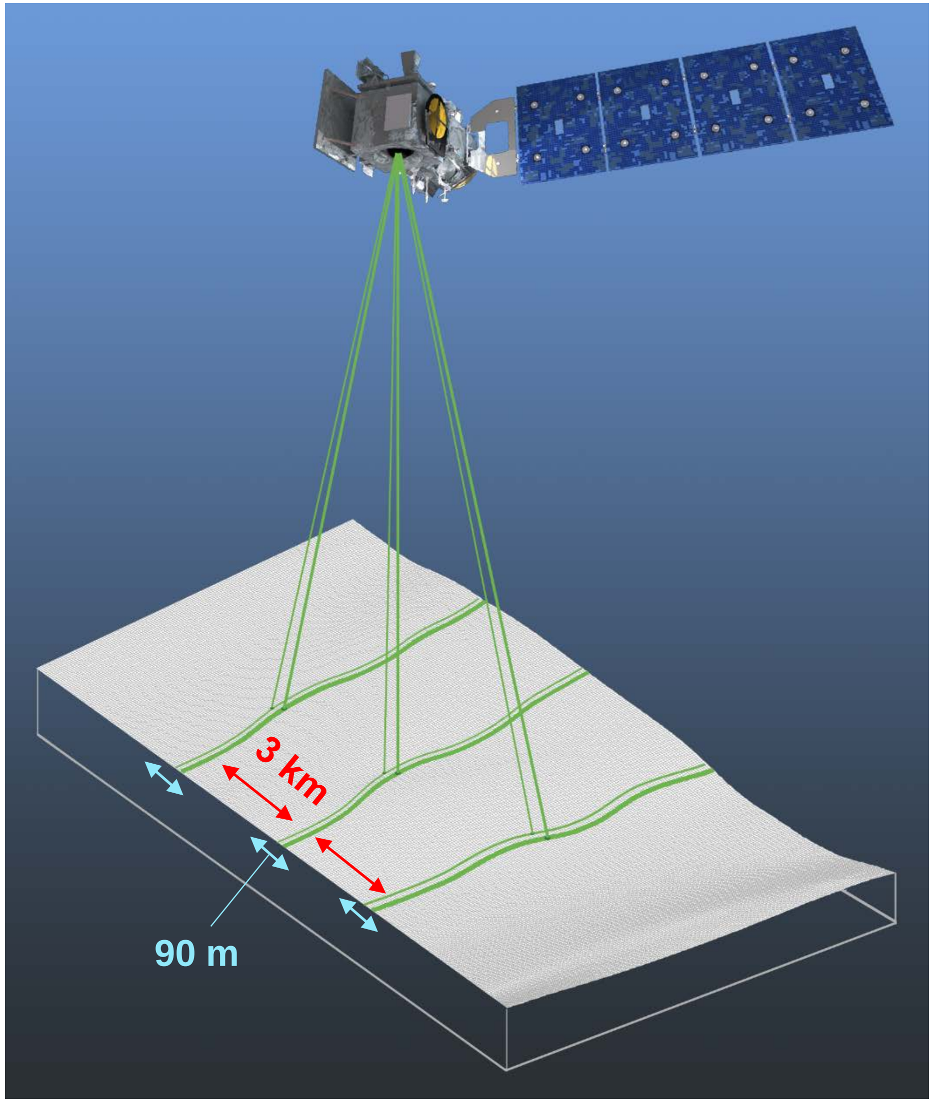


IMAGE CREDIT: NASA/GSFC

ATLAS Instrument

Prior to sending the laser beam down to Earth:

- Photons must first travel through a **series of critical optical components**, such as lenses and mirrors, along the instrument's optical bench in order to:
  - Align the laser and the telescope
  - Check the wavelength of the laser
  - Start the timing mechanism
  - Determine the size of the ground footprint
  - Split the single laser into 6 beams

Therefore, protecting the critical laser flight optics on the ATLAS instrument is important to the successful operation of the satellite

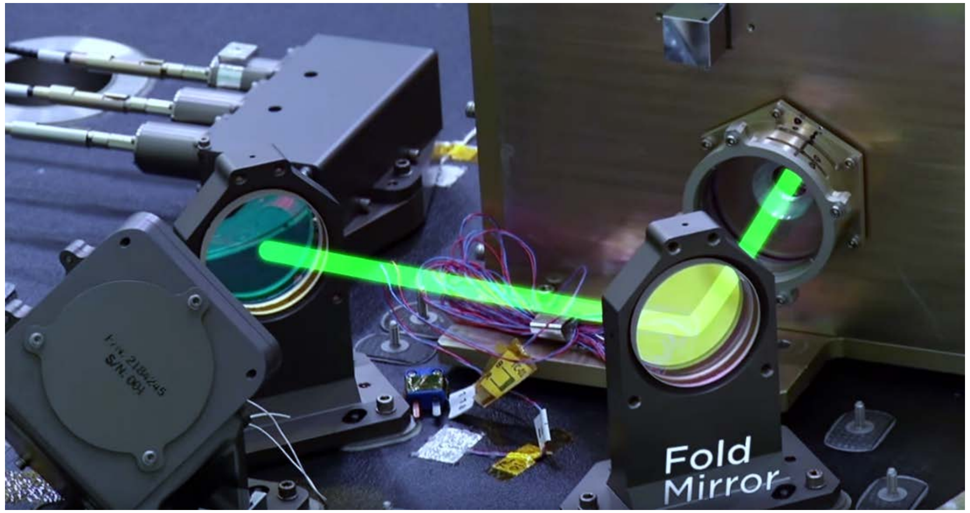


IMAGE CREDIT: NASA/GSFC

Shown in the above images is an animated green laser beam making several turns as it travels through the laser optical components on the optical bench of the ATLAS instrument.

ATLAS Instrument

POTENTIAL PROBLEM

Catastrophic impacts of contamination result in laser induced optical damage and performance degradation in spaceflight laser systems

Molecular contaminants, such as silicones and aromatic hydrocarbons, are particularly known to damage laser optics

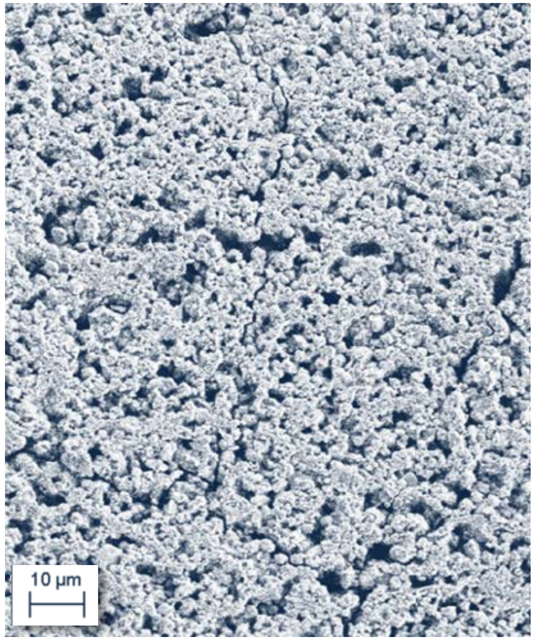
PROPOSED MITIGATION

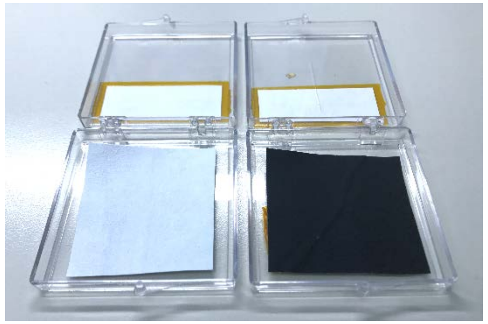
Consequently, it is important to reduce the risk of exposure from potential molecular contaminants throughout the various phases of a NASA mission, particularly during the I&T phases of the project

A contamination control mitigation method that was proposed was the **strategic placement of MAC samples** during TVAC testing of ATLAS hardware in vacuum test chambers

Molecular Adsorber Coatings

- **Molecular Adsorber Coating (MAC)**
  - Sprayable coatings technology
  - Developed by NASA Goddard Space Flight Center
  - Addresses molecular outgassing concerns
  - Comprised of zeolite-based, porous materials
  - Passively captures molecular contaminants
  - Has low outgassing materials
  - Effective in trapping high molecular weight chemical species, such as hydrocarbons, silicones, plasticizers, and other outgassed constituents from common spaceflight materials
  - Used in **on-orbit spaceflight applications**, particularly on or near sensitive surfaces and components on the spacecraft, such as instrument cavities, electronics boxes, and detectors





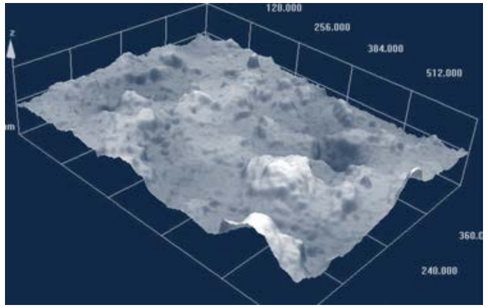


IMAGE CREDIT: NASA/GSFC

Ionospheric Connection Explorer (ICON): MAC plates were installed in the Far Ultraviolet (FUV) instrument to address on-orbit material outgassing concerns

Molecular Adsorber Coatings

- MAC has also been extensively used in **ground-based applications** as a passive getter material during TVAC testing to mitigate the risk of molecular contamination on many NASA missions

NASA MISSION		TVAC PURPOSE	YEAR
JWST	James Webb Space Telescope	Used during testing of optical ground support equipment (OGSE), thermal pathfinder (TPF), and optical telescope element and integrated science (OTIS) instruments	2017 2016 2015 2014
GOLD	Global-scale Observations of the Limb and Disk	Used during instrument level testing	2017
GEDI	Global Ecosystem Dynamics Investigation Lidar	Used during component level testing	2016
NICER	Neutron star Interior Composition Explorer	Used during component level testing	2015
MMS	Magnetosphere Multiscale Mission	Used during component level testing	2014

Approach

- Purpose
- Sample Fabrication & Exposure
- Sample Location
- Sample Configuration
- Temperature Profile

Purpose

- MAC samples were deployed during two TVAC tests to **isolate and protect the critical laser flight optics** on the ATLAS instrument from outgassing sources
  - Potential sources may originate from commonly used spaceflight materials and components, TVAC test set-up and chamber environment, or from other unknowns
  - Time-temperature bake-outs were performed on most items prior to the tests; however, it does not completely eliminate the possibility of outgassing, especially from materials comprised of **silicones or elastomers**
  - Potential sources may also contribute to **molecular reflection** that would not be indicative of the on-orbit flight case due to the confined space and warm walls of the chamber
  - Location of **existing facility scavenger cold plates** would not isolate critical optical components from all possible outgassing sources

POTENTIAL OUTGASSING SOURCES

- Staking Compounds
- Adhesives
- Epoxies
- Cables & Wires
- Isolator Systems
- Batteries
- Non-Baked Items
- Unknown Residues

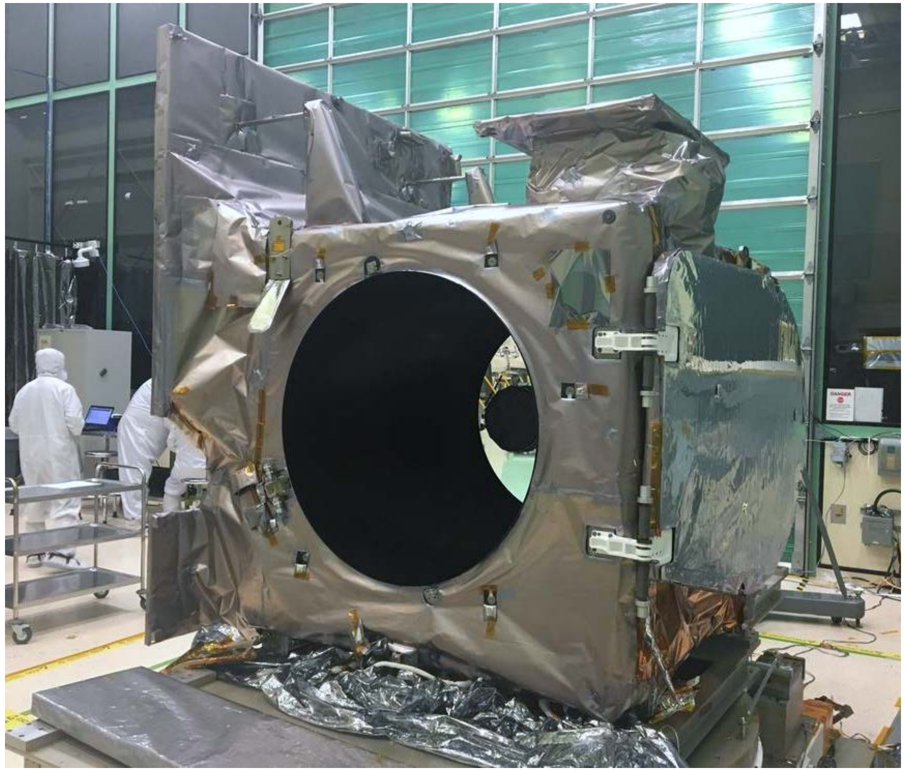


IMAGE CREDIT: NASA/GSFC

Sample Fabrication & Exposure

- NASA GSFC custom-fabricated samples coated with the white version of MAC

COATING TYPE	MAC-W
SUBSTRATE	Aluminum Alloy
COATING THICKNESS	6.2 mils
COATING AREA	95.2 cm^2

IMAGE CREDIT: NASA/GSFC

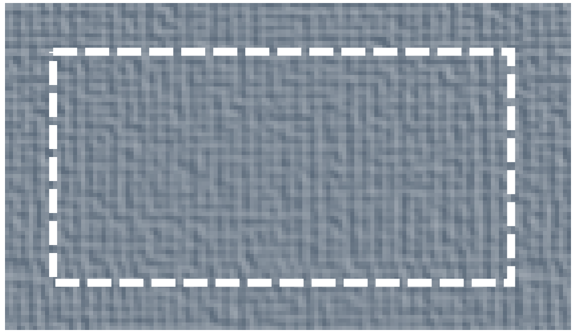
- One sample was exposed during each of the ATLAS TVAC tests (2 samples used total)

TVAC TEST ID	CHAMBER ID	SAMPLE INSTALLATION	EXPOSURE TIME
TVAC A	X	Mid 2017	~ 50 days
TVAC B	Y	Late 2017	~ 30 days

Sample Location

- For each TVAC test, a MAC sample was installed in a **strategic location** that would best isolate and protect the critical laser flight optics during testing in the chamber facility

- The location of the MAC sample is identified by the **white dashed rectangle** as shown in the ATLAS thermal instrument model



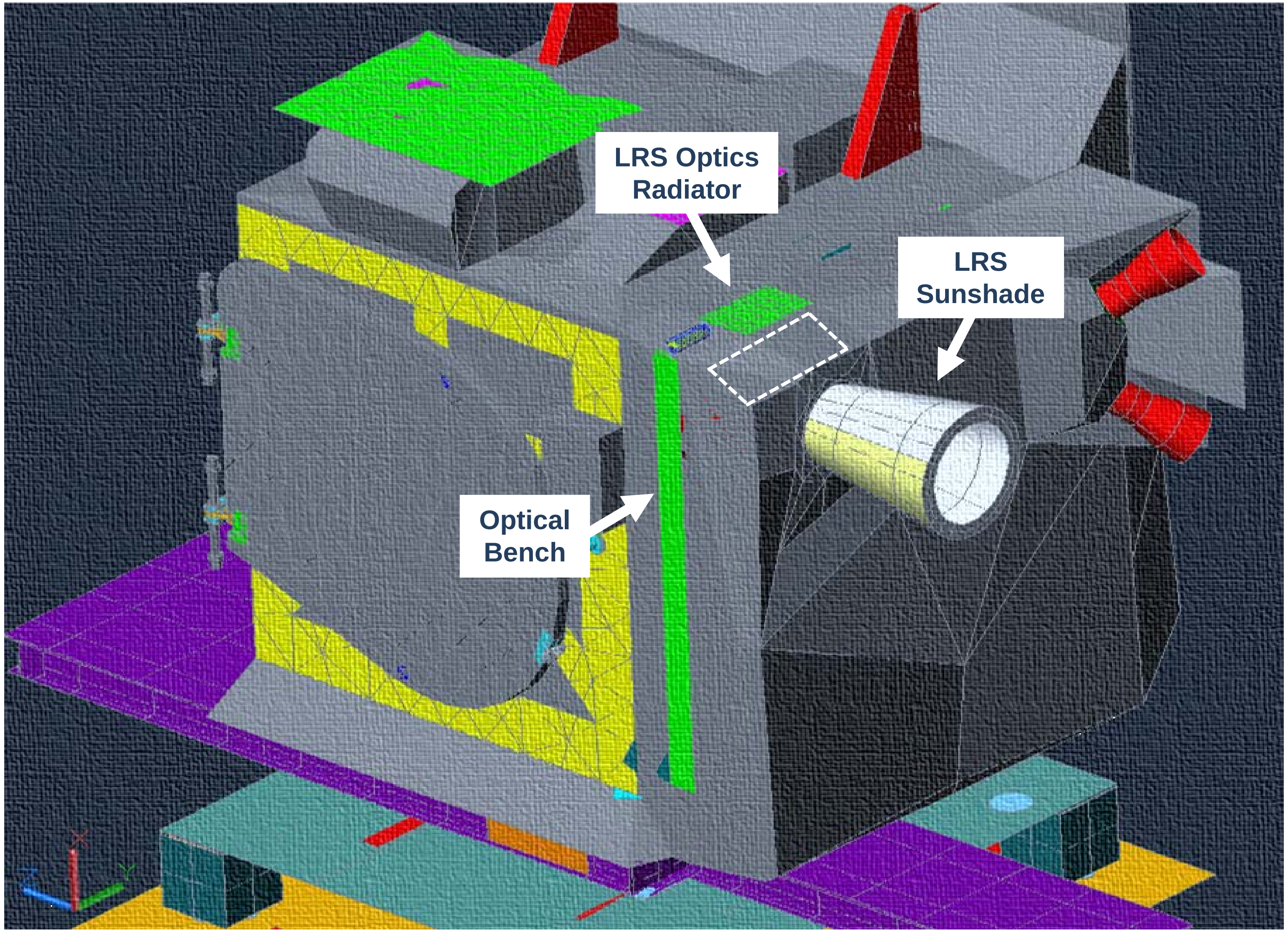


IMAGE CREDIT: NASA/GSFC

Sample Location

- The critical pathway for the transmit optics components on the optical bench is housed below the **blanket vent** *(these components direct the laser from its source on the instrument)*

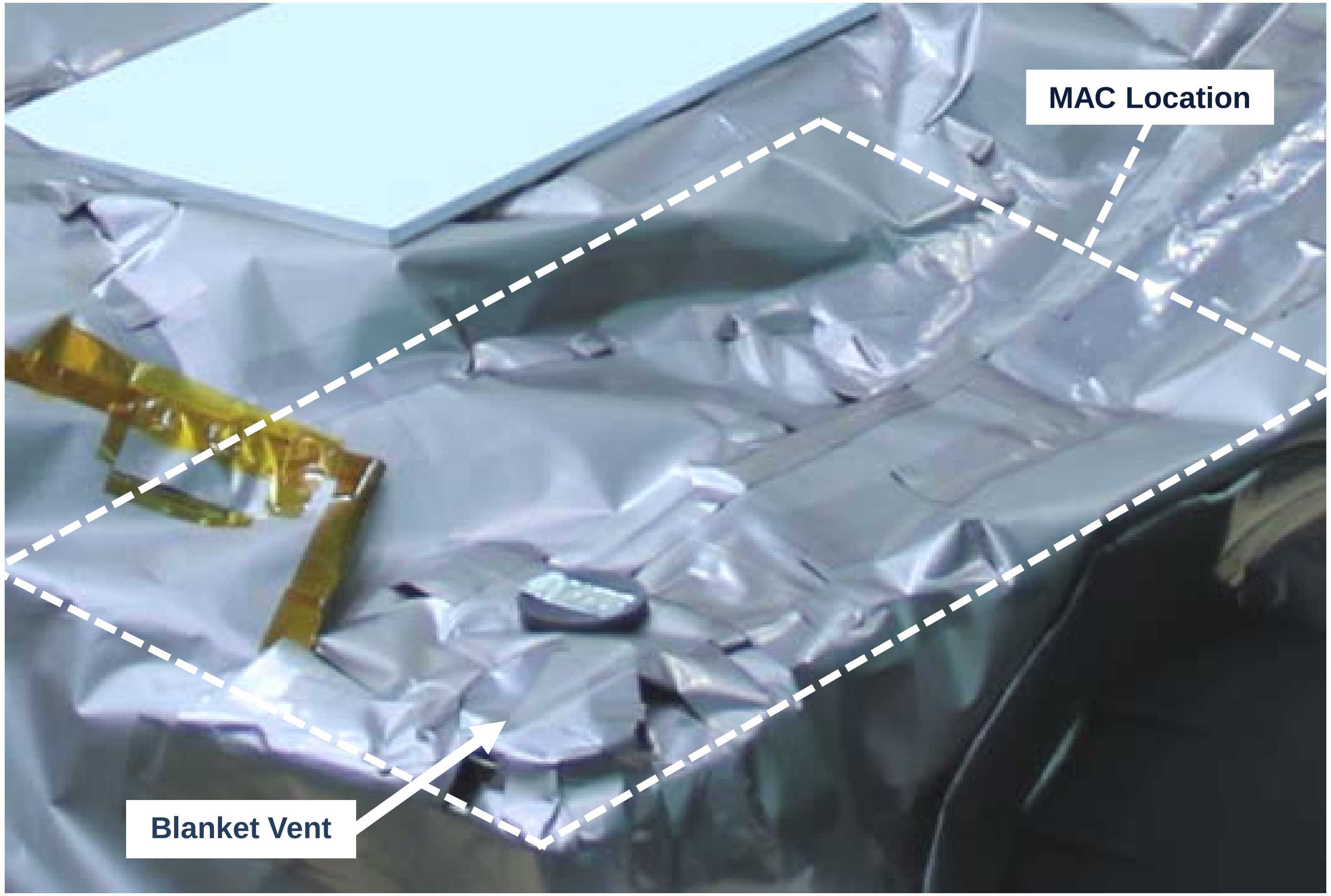
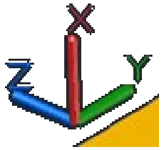
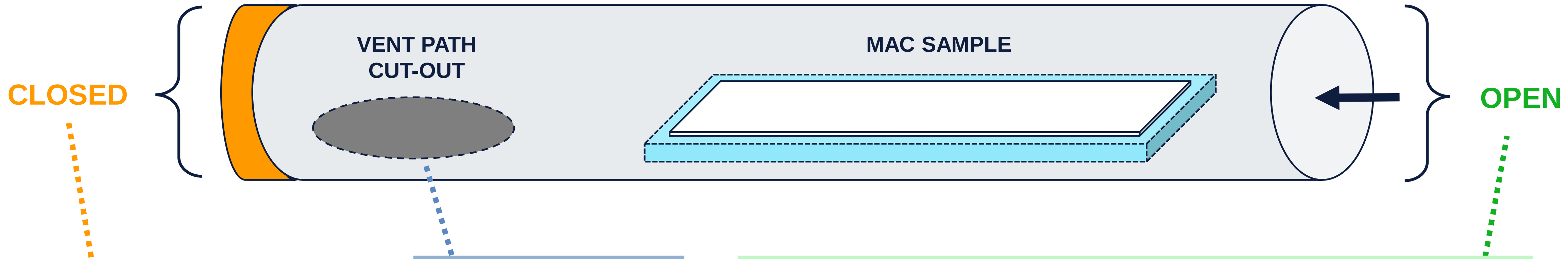


IMAGE CREDIT: NASA/GSFC



Sample Configuration

- An oval shaped **blanket channel** was constructed using VDA SLI material
- The purpose of the fabricated channel is ***to restrict the amount of outgassed species from the test environment*** that may contaminate the critical transmit optics along the vent path



This side was closed off with Kapton tape to direct the entrance of any contaminants through the open side

MAC may also capture outgassed species that ascend from the vent path

Outgassed species that enter the channel will pass MAC, and likely strike its surface; at that point, MAC will passively capture the contaminant before it can continue on towards the vent path. The same is expected of species that ascend from the vent path.

Sample Configuration

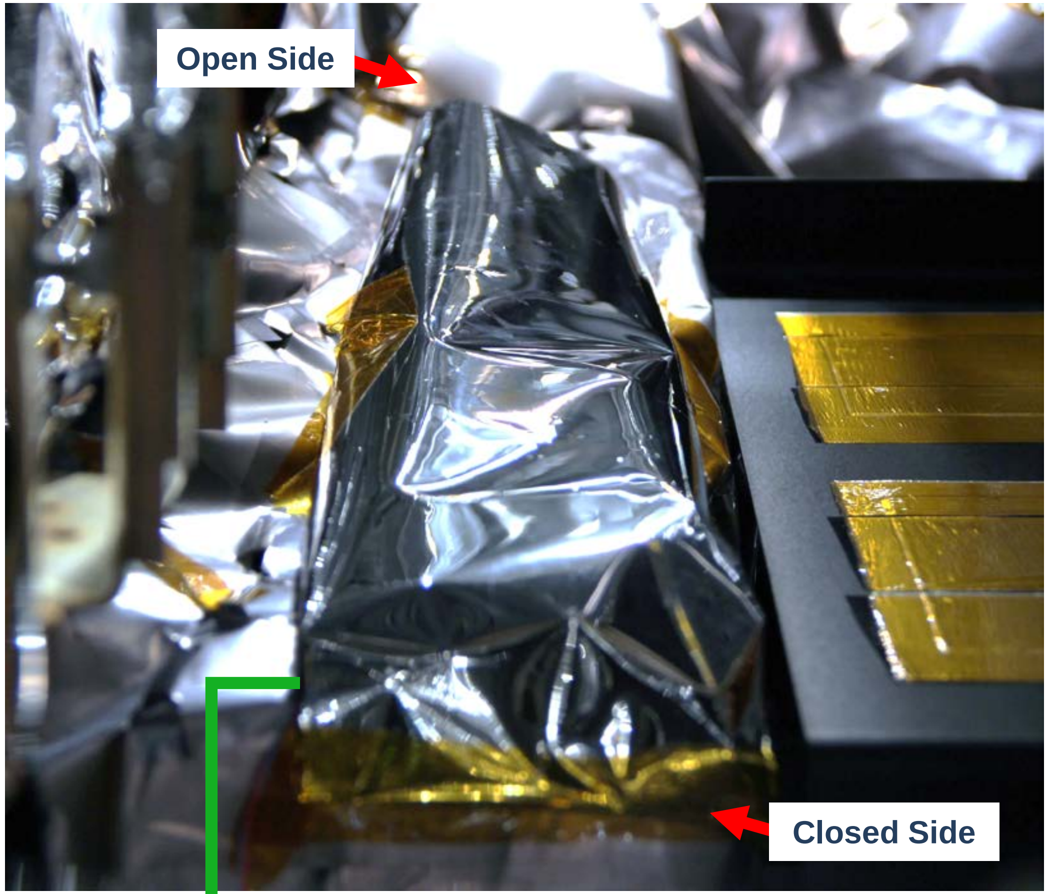
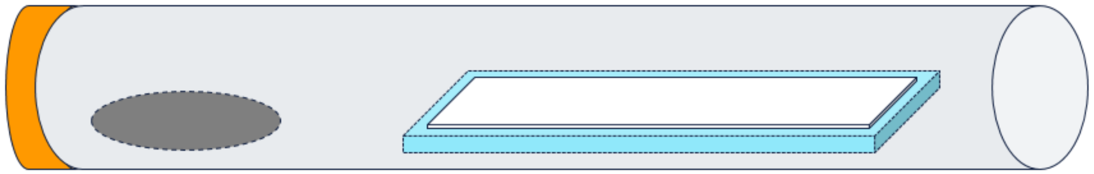


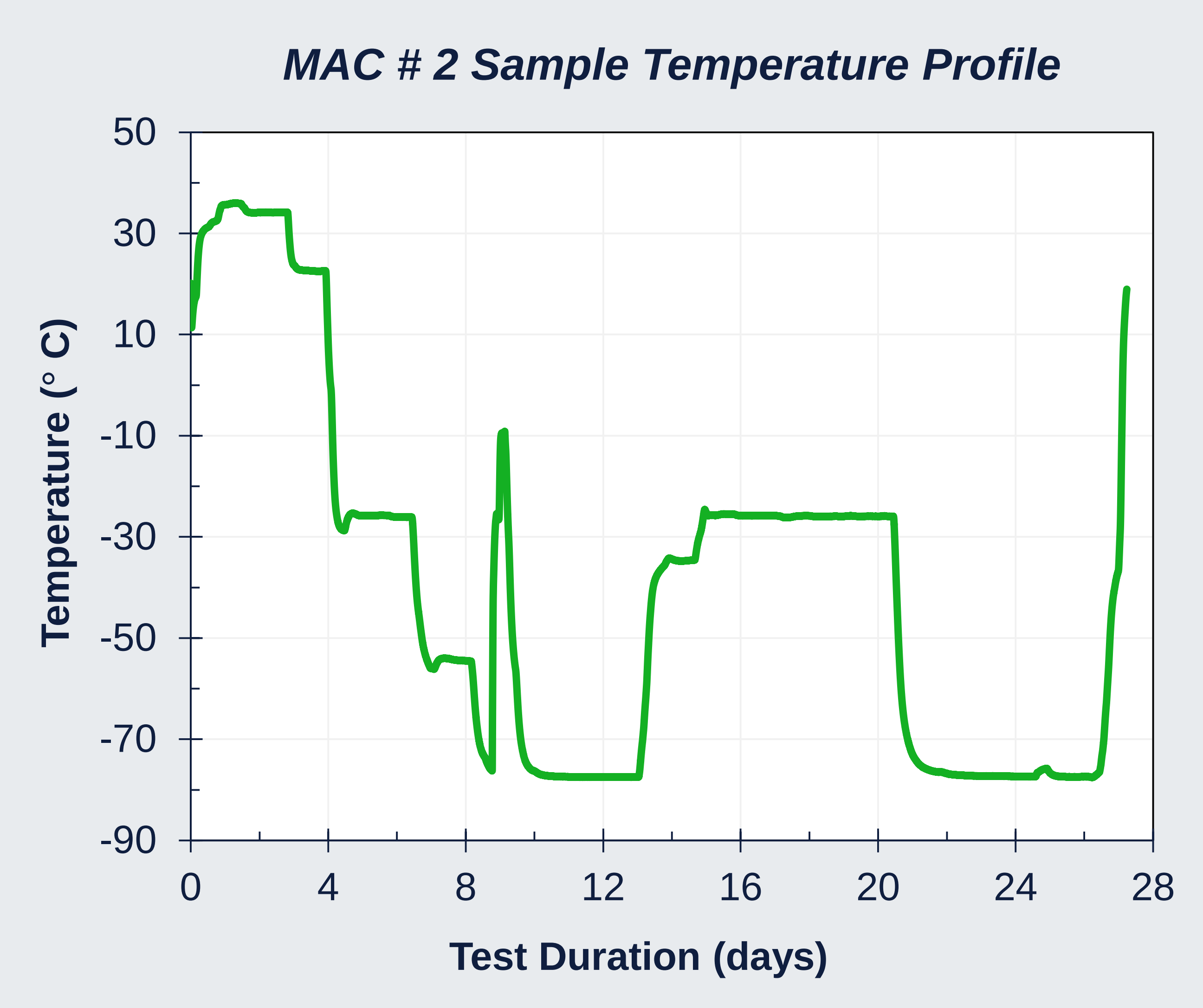
IMAGE CREDIT: NASA/GSFC

TEST ID	SAMPLE ID	SAMPLE INSTALLATION	EXPOSURE TIME
TVAC A	MAC # 1	Mid-2017	~ 50 days
TVAC B	MAC # 2	Late-2017	~ 30 days



Shown is the blanket channel (with the MAC sample) installed prior to the start of TVAC B

Temperature Profile



Maximum Temperature: + 36 °C

Minimum Temperature: - 78 °C

- A thermocouple was attached to monitor sample temperatures throughout the duration of the tests
- MAC # 1 from TVAC A was exposed to a similar temperature profile

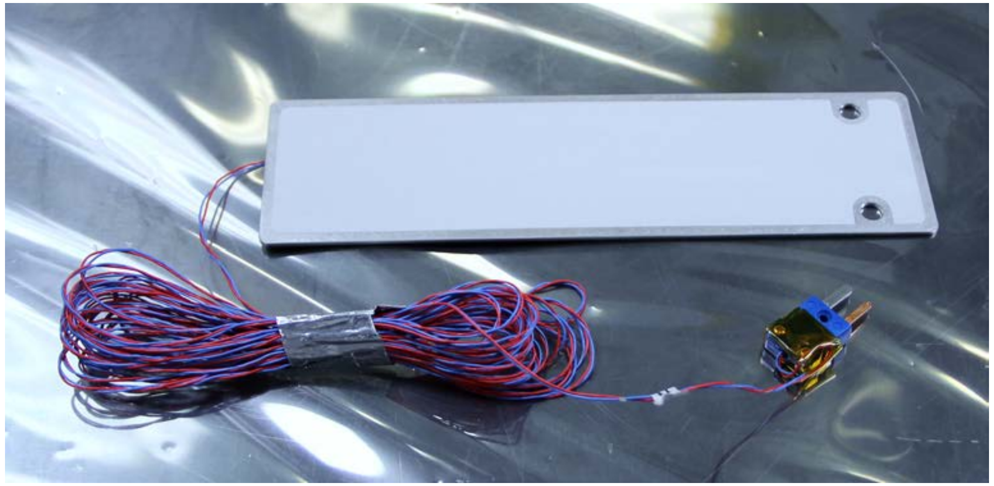


IMAGE CREDIT: NASA/GSFC

Test Methods

- Solvent Rinse Methods
- Chemical Analysis Methods

Solvent Rinse Methods

- The surface of the coating was directly rinsed **4 times** with optima grade **chloroform**
- This method was used **to extract contaminants** that were adsorbed on MAC – but only those that can be dissolved with the selected organic solvent
- Previous solvent rinse test efforts have demonstrated that:
  - Chloroform is effective in dissolving **common species of interest**, such as hydrocarbons and silicones
  - Multiple solvent rinses remove additional contaminants from MAC
  - Dissolved species from multiple rinses decrease with each consecutive rinse

MAC Control Sample

- Coated at the same time as the other two
- Not exposed to any chamber facility
- Establishes a baseline reference
- Provides insight into the adsorbed residual contaminants due to handling or from exposure to **offgassed species** that are present in ambient, non-vacuum environments, such as a laboratory

SAMPLE ID	TVAC EXPOSURE	SAMPLE CONDITION	ANALYSIS DATE
MAC # 0	No TVAC	Control	August 2017
MAC # 1	TVAC A	Contaminated	August 2017
MAC # 2	TVAC B	Contaminated	March 2018

Solvent Rinse Methods

RINSE TYPE	TOTAL RINSES	RINSE NUMBER	RINSE SOLVENT
Single	1	Rinse 1	Chloroform
Triple	3	Rinses 2, 3, 4	Chloroform

DETERMINING THE MASS OF THE NON-VOLATILE RESIDUE FROM THE SOLVENT RINSES

Rinse sample 1x with solvent → **Collect 1x rinsate in pre-weighed dish** → **Rinse sample 3x with solvent** → **Collect 3x rinsate in another pre-weighed dish** → **Allow both to evaporate to dryness** → **Record mass of dishes to determine NVR**

Purpose of this multiple rinse (3x) is to dissolve any remaining contaminants in MAC

Chemical Analysis Methods

- NVR from the samples were also evaluated using two chemical analysis methods to obtain a **general approximation** of the types and relative amounts of contaminants in the NVR

METHOD	FTIR ANALYSIS	PYROLYSIS GC/MS ANALYSIS
	Fourier Transform Infrared Spectroscopy	Pyrolysis-Gas Chromatography/Mass Spectrometry
INSTRUMENT	Thermo Fisher Scientific Nicolet 6700	Shimadzu Scientific Instruments QP2010 Ultra and GL Sciences Optic-4 Inlet

PYROLYSIS

Collected NVR was placed in a micro-vial inside a liner and heated in GC outlet at a high rate of 30 °C/s to an elevated temperature of 600 °C

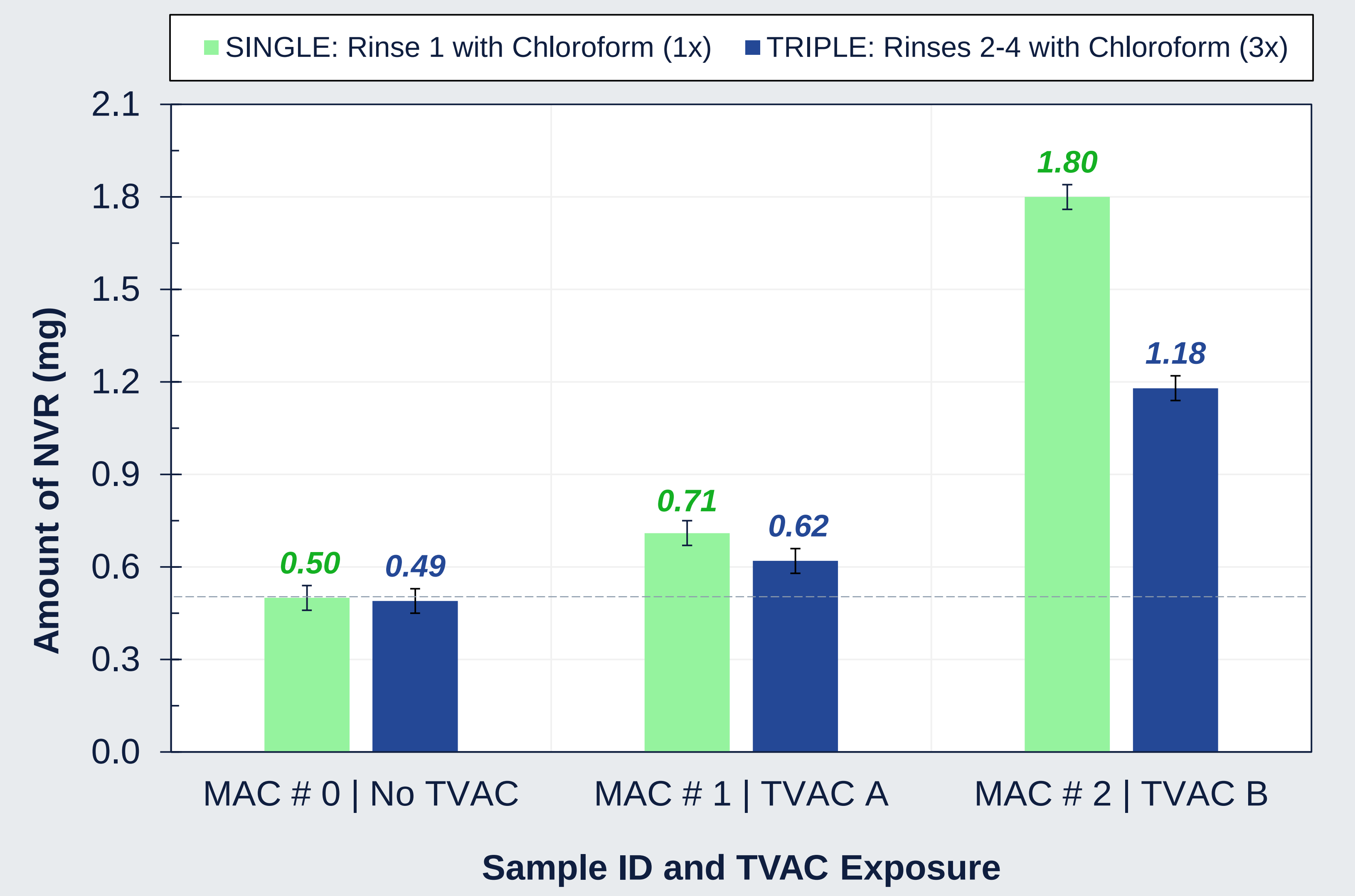
→ Next, volatile and semi-volatile chemical species that evolved from this thermal decomposition phase were introduced to GC column interface with MS

→ Non-volatile chemical species remained in micro-vial to avoid inlet contamination of instrument

Results & Discussion

- Solvent Rinse Results
- Molecular Adsorption Capacity
- Chemical Analysis Results
- Comparison to Contamination Monitoring Methods

Solvent Rinse Results



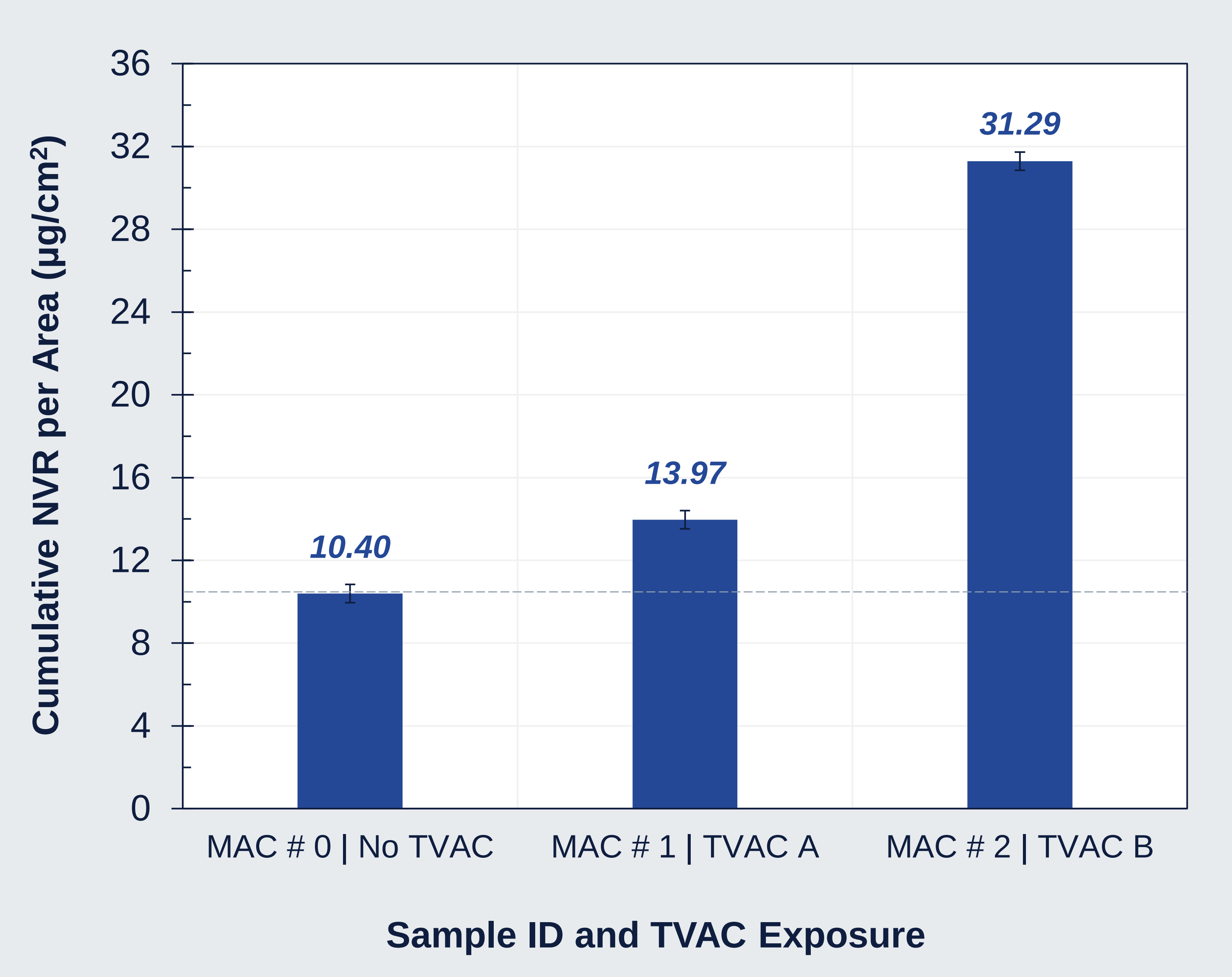
The error bars are associated with the weighing uncertainty of ± 0.04 on the laboratory balance that was used for the gravimetric measurements

SINGLE RINSE	
TEST ID	**ADJUSTED NVR**
TVAC A	0.21 mg
TVAC B	1.30 mg

The control sample was subtracted for the adjusted NVR shown

- Amount collected during TVAC B was about **6.2 times greater** than TVAC A
- Similar trends were observed for 3x rinse runs of exposed TVAC samples
- Decreasing NVR for 3x rinse suggests that most of the contaminants were removed in the initial 1x rinse

Solvent Rinse Results

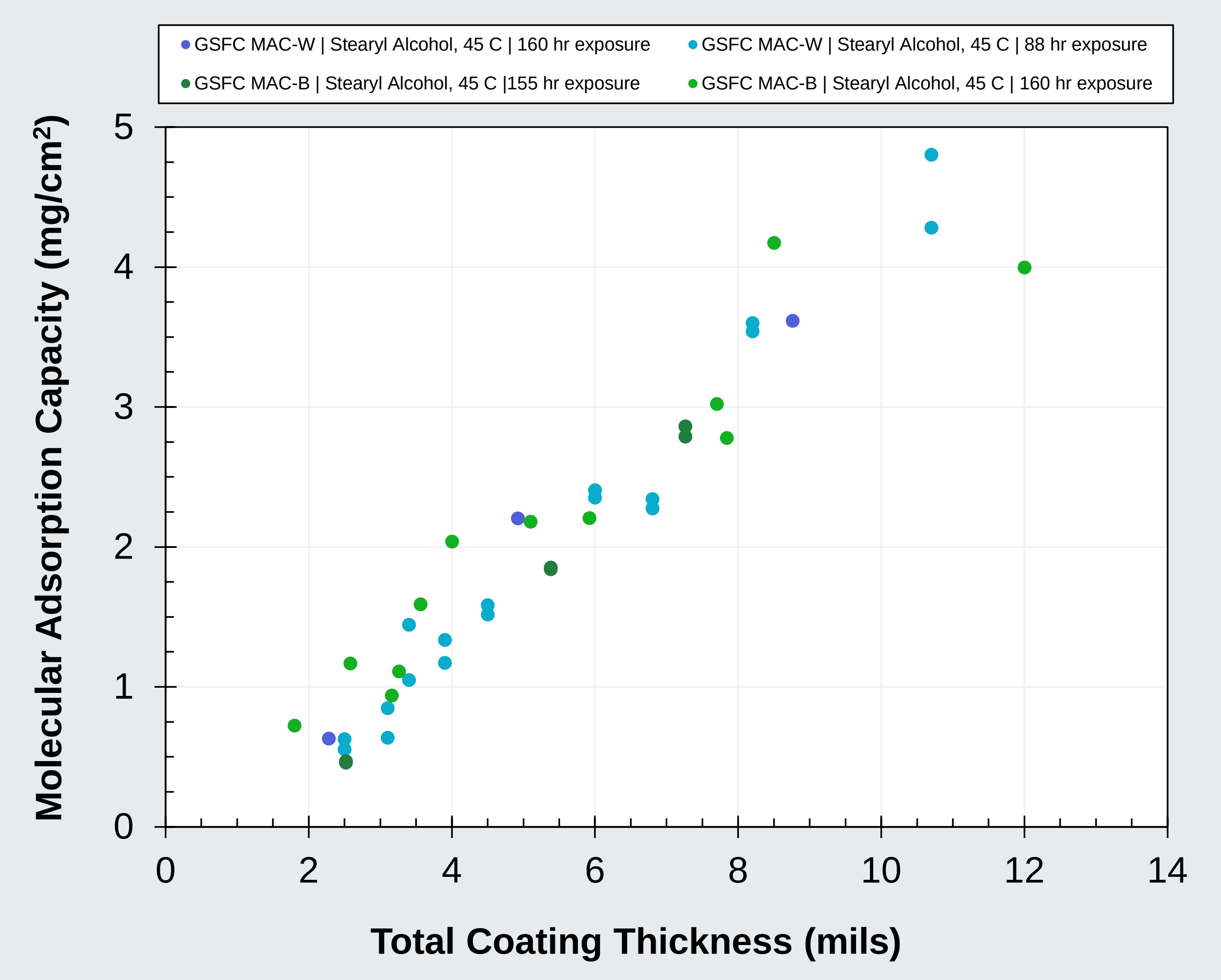


SAMPLE ID	CUMULATIVE NVR
MAC # 0	0.99 mg ± 0.04
MAC # 1	1.33 mg ± 0.04
MAC # 2	2.98 mg ± 0.04

- Sample exposed during **TVAC B captured the most contaminants**, even though, it was exposed for a shorter test duration
- Some possible reasons for this may include differences in the test facility, chamber size, temperature variations, and sample handling procedures
- MAC # 2 results are about:
  - 3 times greater than control sample
  - 2.3 times greater than MAC #1

Molecular Adsorption Capacity

- Past studies have involved saturating MAC samples with **stearyl alcohol** as the test contaminant source under vacuum conditions
- Stearyl alcohol is an 18-chain hydrocarbon that is representative of outgassed species that are commonly found in spaceflight applications
- The experimental data shown suggests that the molecular adsorption capacity of MAC is **directly proportional** to coating thickness

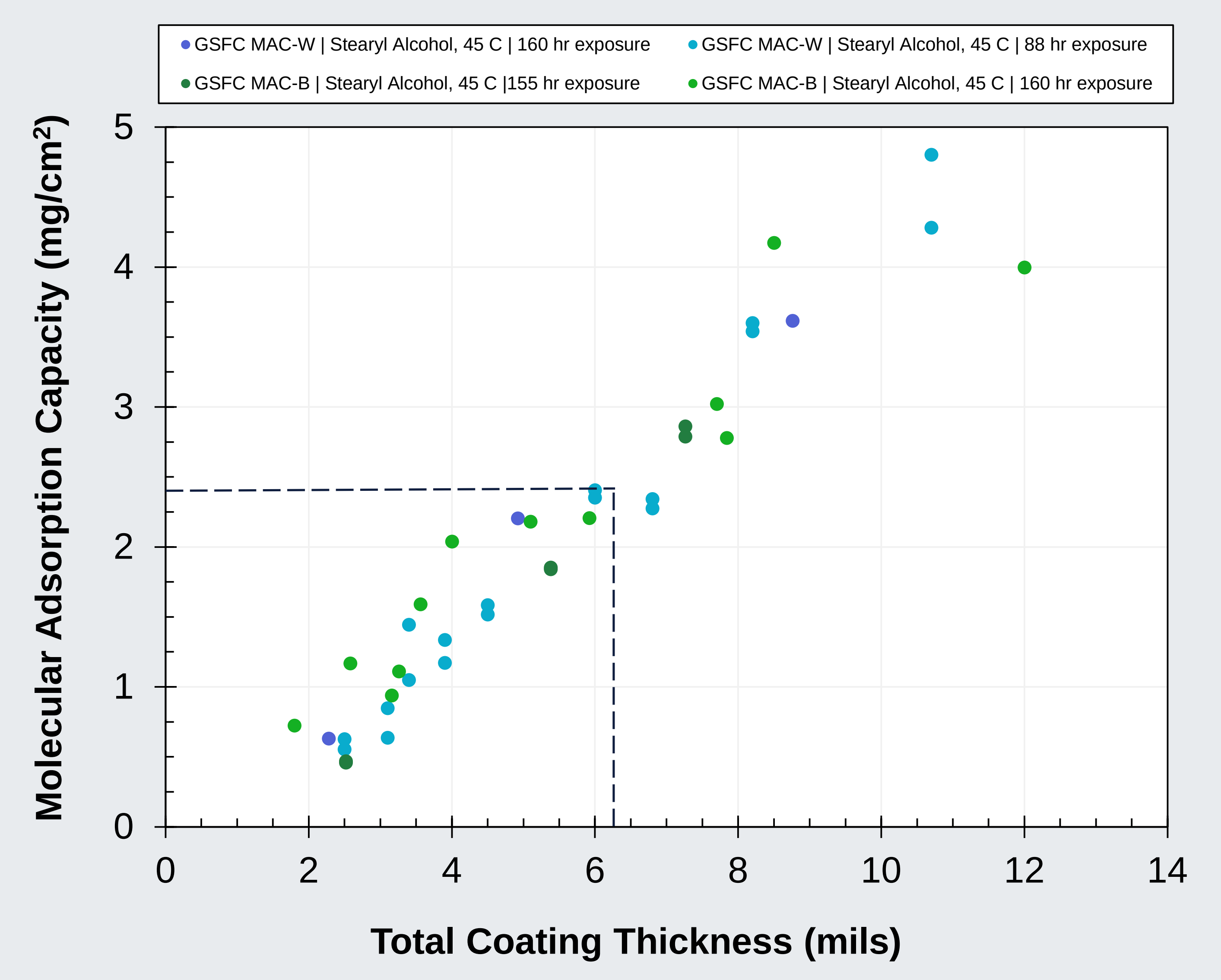


Molecular Adsorption Capacity

- The predicted vacuum molecular adsorption properties of the MAC samples that were used during the ATLAS TVAC tests are shown below:

Coating Thickness	**6.2 mils**
Coating Area	**95.2 cm^2**
Estimated Molecular Adsorption Capacity in Vacuum	**2.4 mg/cm^2**
Estimated Maximum Mass	**~ 228 mg**

However, this assumes that the majority of the species collected during the tests are long-chain hydrocarbons similar to stearyl alcohol



Molecular Adsorption Capacity

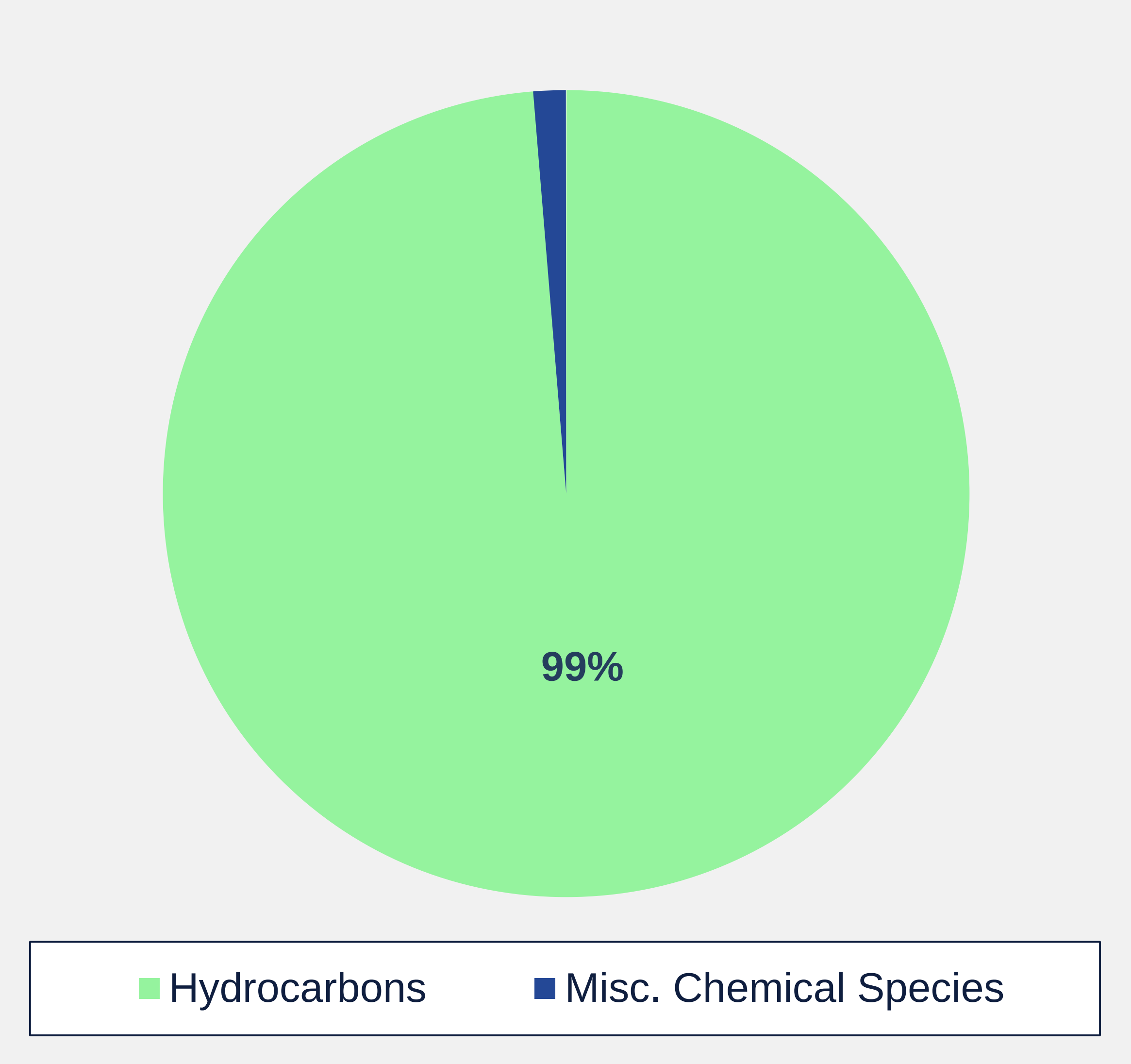
- The **saturation ratio** is defined as the ratio of the cumulative NVR per coating area to the estimated experimental molecular adsorption capacity (in vacuum conditions)
- The results suggest that the MAC samples that were deployed during the TVAC tests were **not significantly contaminated** with outgassed species **to complete saturation of the pores in the coating**
- Nevertheless, the chemical species that were collected during TVAC were isolated from further contaminating the critical laser flight optics on the ATLAS instrument

SAMPLE	ESTIMATED SATURATION RATIO
MAC # 1	0.6 %
MAC # 2	1.3 %

SOME CONSIDERATIONS

- Not all of the adsorbed contaminants will be removed with multiple solvent rinses
- Not all of the adsorbed contaminants will be long-chain hydrocarbons similar to the experiment contaminant

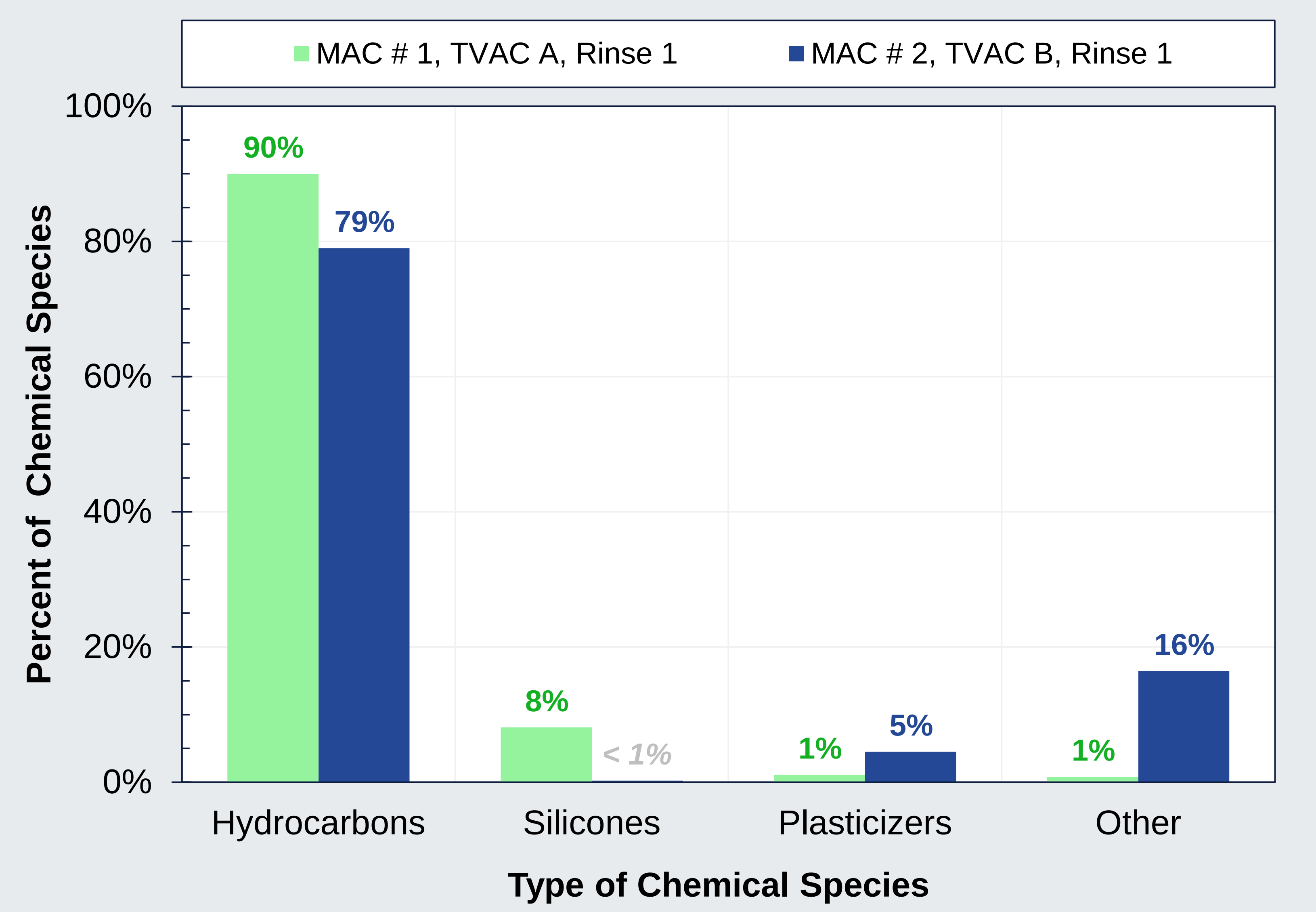
Chemical Analysis Results



MAC # 0 Control Sample

- Shown is the percent distribution of chemical species for the single solvent rinse of the control sample
- Results indicate that 99% is comprised of **primarily hydrocarbons** that may have been adsorbed from exposure to the ambient environment during its storage period or due to sample handling
- Remaining 1% of miscellaneous species are likely room environmental compounds
- Similar trends were observed for 3x rinse of control sample (as well as the two vacuum exposed samples)

Chemical Analysis Results



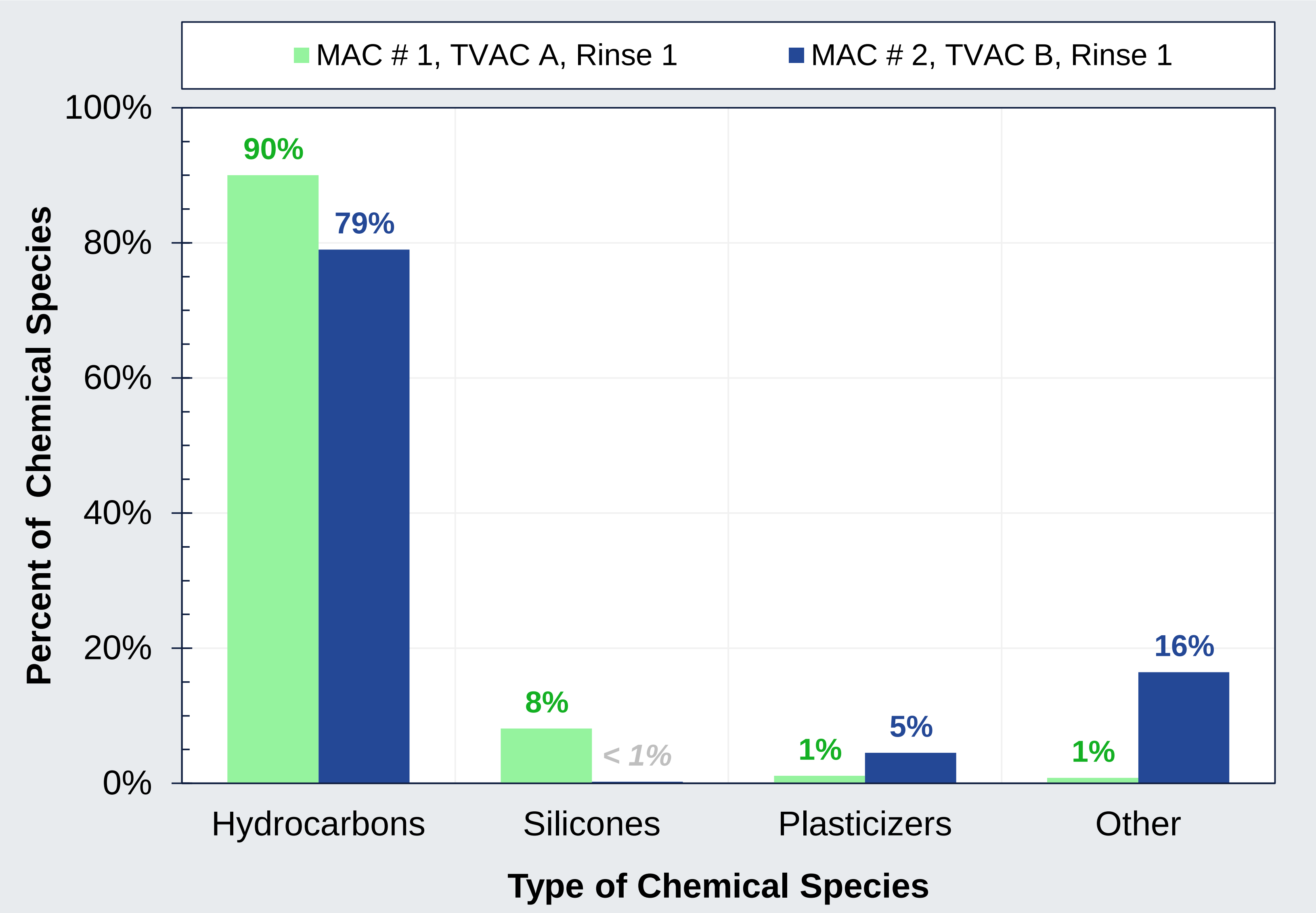
Both TVAC Tests

- Most abundant type of chemical species were **hydrocarbons**

TVAC A

- Second most abundant species at 8% were **silicones**, such as **methyl phenyl silicones** and **methyl silicones**, that are commonly sourced from lubricants, elastomers, and adhesives
- Remaining least abundant species at 2% consisted of **plasticizers**, such as **phthalate-based species**, and **other** miscellaneous chemical constituents, such as **palmitate-based species**

Chemical Analysis Results

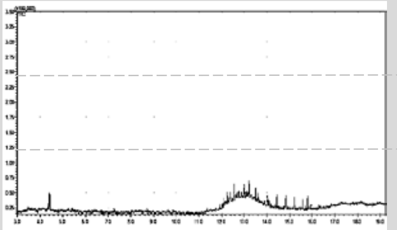


TVAC B

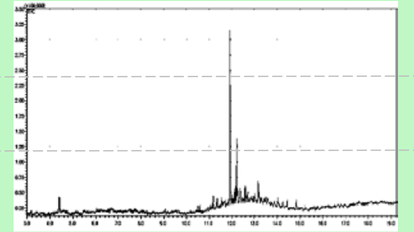
- Second most abundant species were "other" at 16%, specifically handling contaminants, which consisted of mostly **palmitate-based species**, such as **isopropyl palmitate**, which are commonly found in cosmetics (i.e. emollients, moisturizers, thickening agents), lubricants, rubber, and latex
  - *"Other" also included methyl palmitate, butyl palmitate, and isopropyl myristate*
- Third most abundant species at 5% include **phthalate-based plasticizers**, such as **di(2-ethylhexyl) phthalate**
- Least abundant species identified were silicone-based compounds at 0.2%

Chemical Analysis Results

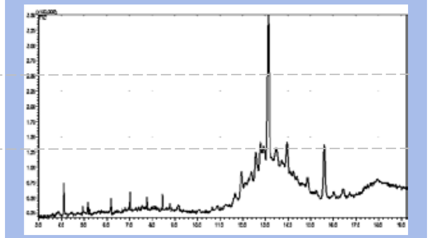
MAC # 0 Rinse 1

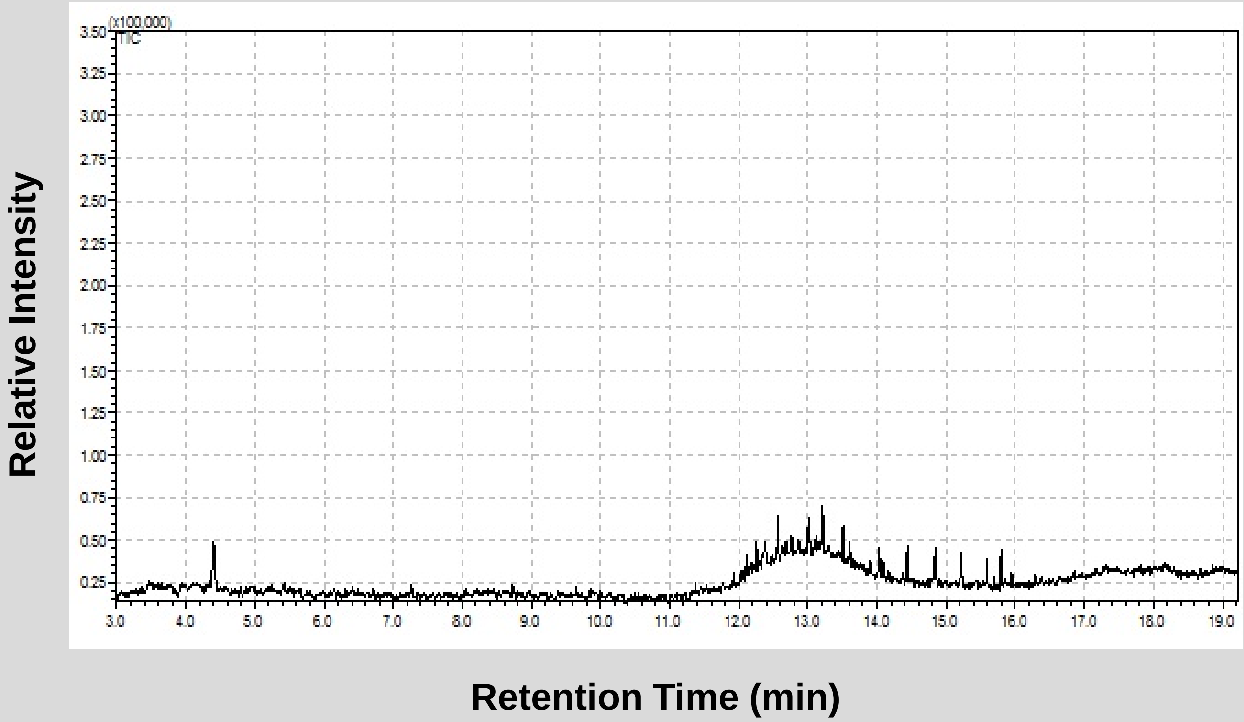


MAC # 1 Rinse 1



MAC # 2 Rinse 1



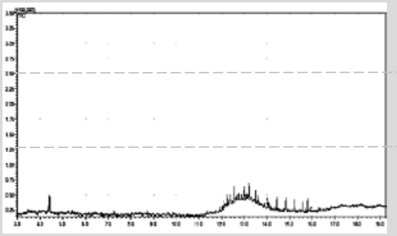


MAC Control Sample

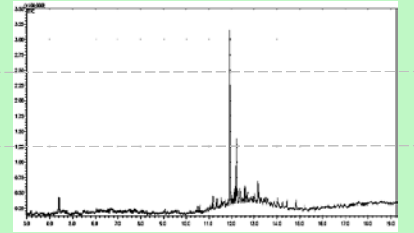
- GC/MS plot shows the **lowest** relative intensity, or compound abundance, of identified species
- Mostly hydrocarbon peaks

Chemical Analysis Results

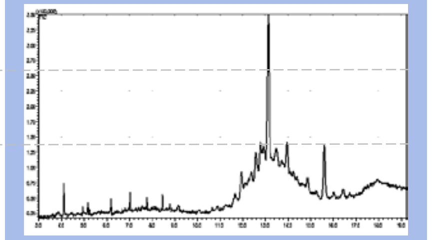
MAC # 0   Rinse 1

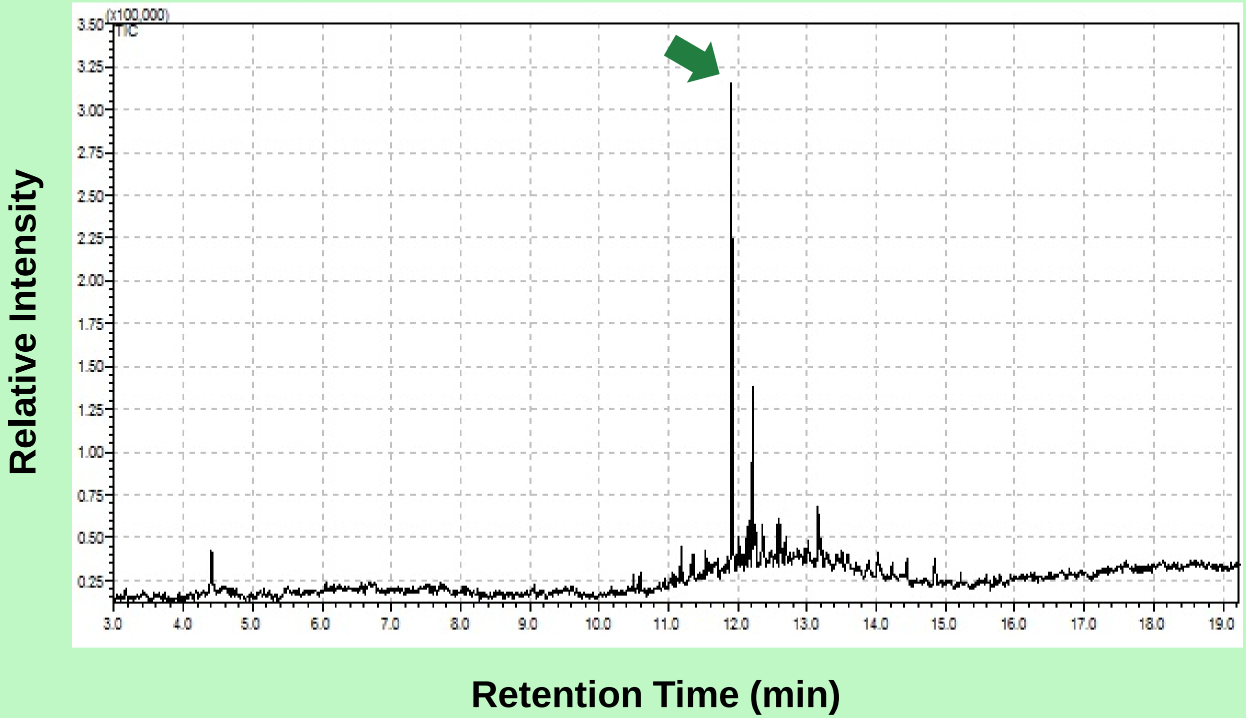


MAC # 1   Rinse 1



MAC # 2   Rinse 1



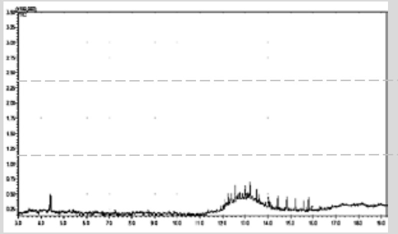


MAC # 1 from TVAC A

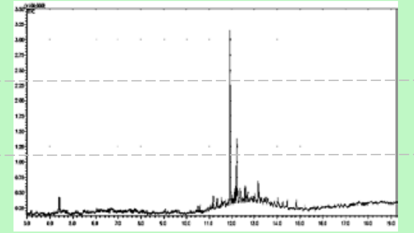
- GC/MS plot shows additional peaks at a greater relative intensity when compared to the control sample
- Mostly hydrocarbon peaks
- Highest intensity peak present for **silicones**, such as **methyl phenol silicone** at around 11.9 min

Chemical Analysis Results

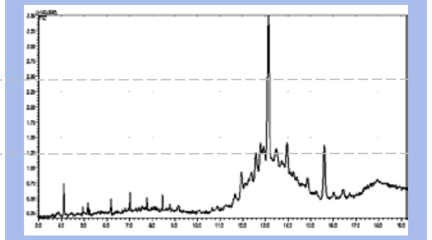
MAC # 0 Rinse 1

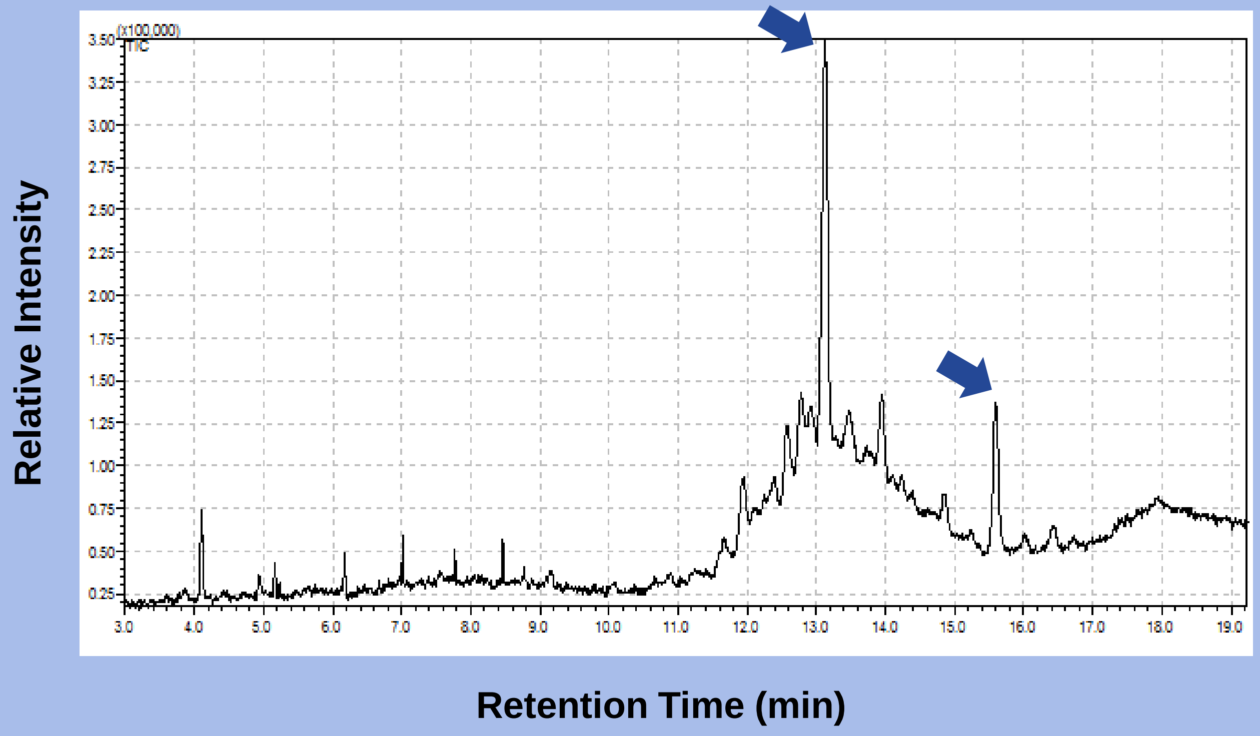


MAC # 1 Rinse 1



MAC # 2 Rinse 1





MAC # 2 from TVAC B

- GC/MS plot shows the greatest relative intensity of species when compared to control sample and first TVAC sample
- Again, mostly hydrocarbon peaks
- Many high intensity peaks present for **palmitate-based species**, such as **isopropyl palmitate** at around 13.2 min
- High intensity peaks also present for **plasticizers**, such as **di(2-ethylhexyl) phthalate** at around 15.6 min

Comparison to Contamination Monitoring Methods

- MAC results were compared against typical contamination control monitoring methods, which are often used during spaceflight vacuum chamber testing

Monitoring Method	Witness Foil	Scavenger Plate	Cold Finger
Description	Witness Sample	Cold panel cooled by LN2	Small cylindrical device cooled by LN2
Purpose	Used to collect NVR representative of what may be found on the hardware	Used to collect the majority of the outgassed contamination throughout the TVAC test	Used to collect residual outgassing during the last several hours of the TVAC test
NVR Rinse	Chloroform	Isopropyl Alcohol	Isopropyl Alcohol
Chemical Analysis	FTIR & GC/MS	FTIR & GC/MS	FTIR & GC/MS

Comparison to Contamination Monitoring Methods

- Comparison provides **additional verification** of the identified contaminant species from the MAC samples that were installed for the two TVAC tests

	TVAC A			TVAC B				
	MAC Sample	**TVAC Witness Foil**	**TVAC Scavenger Plate**	**MAC Sample**	**TVAC Witness Foil**	**TVAC Bake-Out Scavenger Plate**	**TVAC Bake-Out Cold Finger**	**Post TVAC Chamber Cert Cold Finger**
	Chloroform	*Chloroform*	*Isopropyl Alcohol*	*Chloroform*	*Chloroform*	*Isopropyl Alcohol*	*Isopropyl Alcohol*	*Isopropyl Alcohol*
Hydrocarbons	X	X	X	X		X	X	X
Silicones	X	X	X	X	X	X	X	
Plasticizers	X	X	X	X	X	X	X	X
Other	X	X		X	X	X	X	X

Comparison to Contamination Monitoring Methods

- A solvent rinse NVR comparison was made between the witness foils and the MAC samples given that they are **both passive collection methods**
- Witness foils were deployed during the start of each test and placed in a subject location near the ATLAS instrument in the chamber facility; however, the MAC samples and witness foils **were <u>not</u> installed in the same location**

TEST ID	WITNESS FOIL *Rinse 1 with Chloroform*	MAC SAMPLE *Rinse 1 with Chloroform*	
TVAC A	**0.10 µg/cm²** ± 0.02	**2.20 µg/cm²** ± 0.44	**22x**
TVAC B	**0.07 µg/cm²** ± 0.02	**13.65 µg/cm²** ± 0.44	**195x**

For MAC, the control sample was subtracted for the adjusted NVR per coating area

- The significantly larger relative amounts that were collected on MAC **<u>do not</u>** provide conclusive evidence that condensation would have taken place on the instrument due to **physical and chemical differences between the coating and the hardware surface**
- However, results **<u>do</u>** suggest that MAC may serve as a **better method for both mitigation and indication of contaminant threats**
- Unlike other typical monitoring methods, contaminants that are passively captured within the coating during TVAC are **less likely to be released during warm-up activities to ambient pressures**

Summary

- Conclusions
- Future Work

Conclusions

- The use of the MAC technology during ATLAS TVAC testing was effective in protecting the critical laser flight optics
- The identified chemical species that were captured by MAC isolated the transmit optics components from potential molecular contamination
- The continued use of the MAC technology as both a **mitigator and indicator** for outgassed molecular contaminants is recommended during vacuum chamber tests of spaceflight hardware for future NASA missions

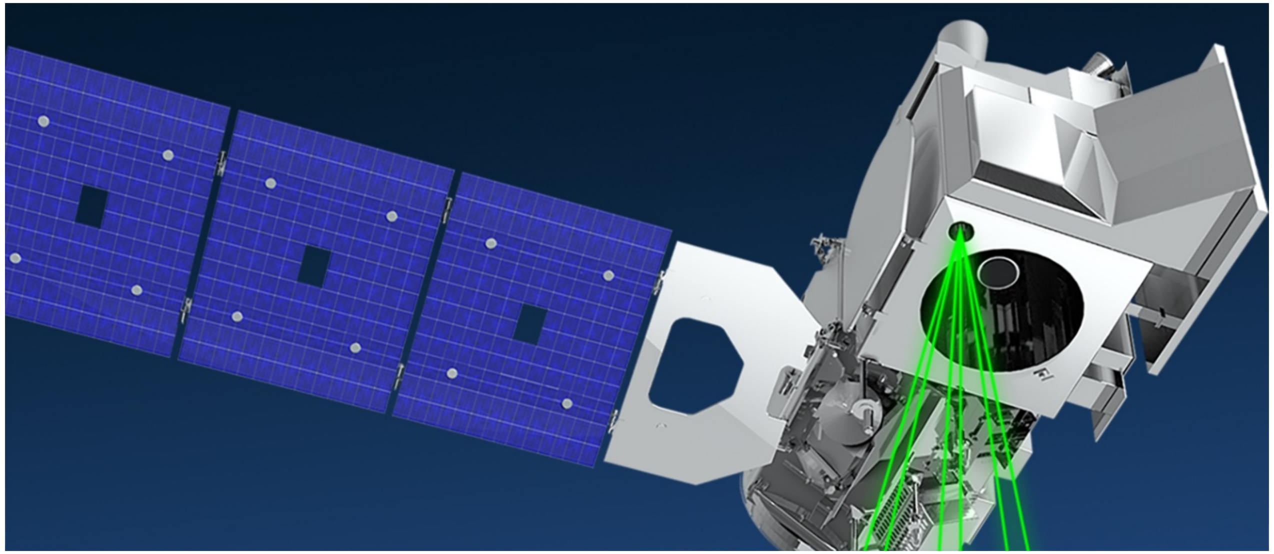
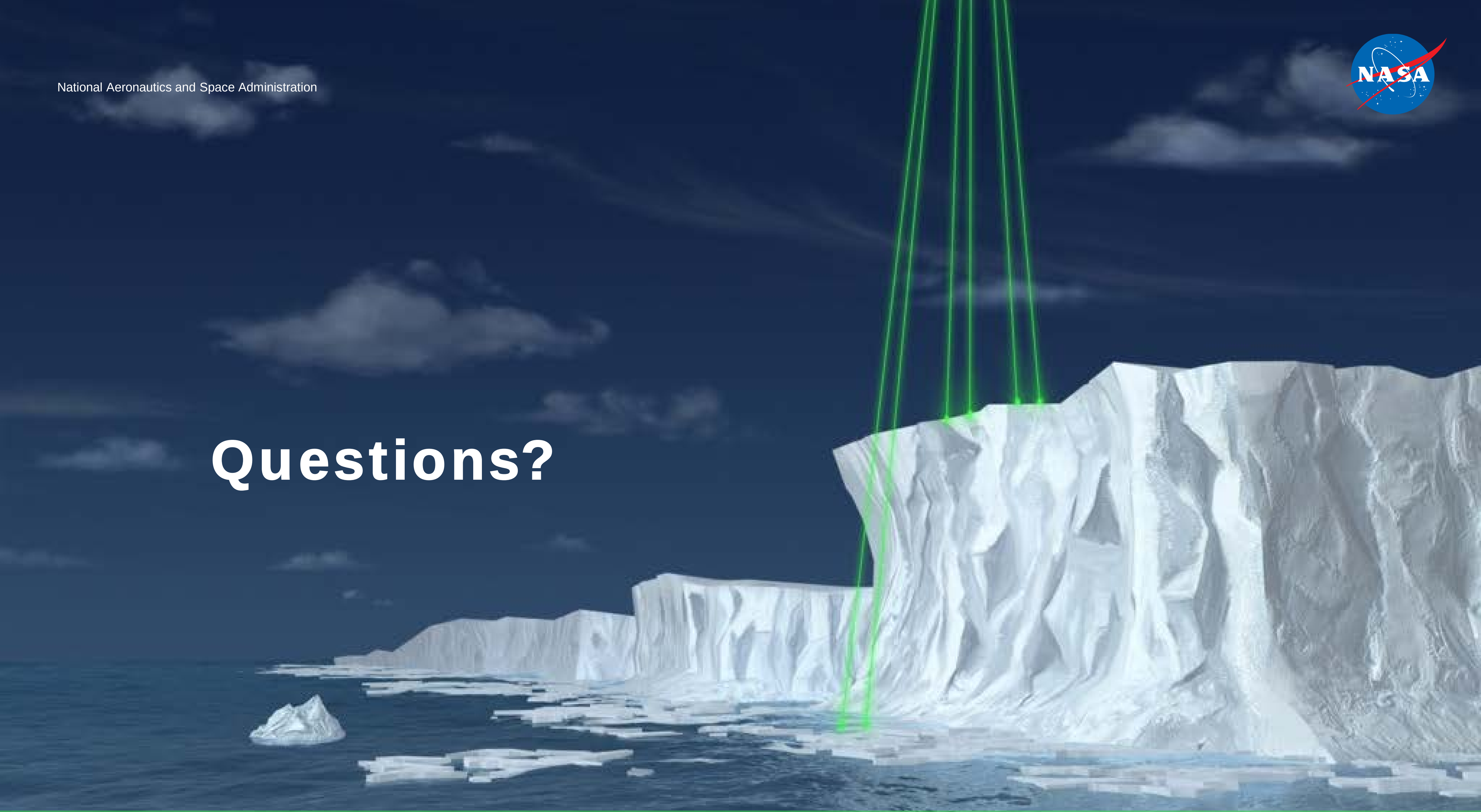


IMAGE CREDIT: NASA/GSFC

FUTURE WORK

- **Explore alternative methods of identifying the collected contaminants on MAC, such as via thermal desorption techniques or the use of other organic solvents**



Questions?

Acronyms

ATLAS	Advanced Topographic Laser Altimeter System
FTIR	Fourier Transform Infrared Spectroscopy
FUV	Far Ultraviolet
GC/MS	Gas Chromatography/Mass Spectrometry
GEDI	Global Ecosystem Dynamics Investigation Lidar
GOLD	Global-scale Observations of the Limb and Disk
GSFC	Goddard Space Flight Center
I&T	Integration and Test
ICESat-2	Ice, Cloud, and Land Elevation Satellite 2
ICON	Ionospheric Connection Explorer
JWST	James Webb Space Telescope
LN2	Liquid Nitrogen
LRS	Laser Reference System
MAC	Molecular Adsorber Coating
MAC-W	White Molecular Adsorber Coating
MMS	Magnetosphere Multiscale Mission
NASA	National Aeronautics and Space Administration
NICER	Neutron star Interior Composition Explorer
NVR	Non-Volatile Residue
OGSE	Optical Ground Support Equipment
OTIS	Optical Telescope Element And Integrated Science
SGT	Stinger Ghaffarian Technologies
SLI	Single Layer Installation
TPF	Thermal Pathfinder
TVAC	Thermal Vacuum
VDA	Vapor Deposited Aluminum

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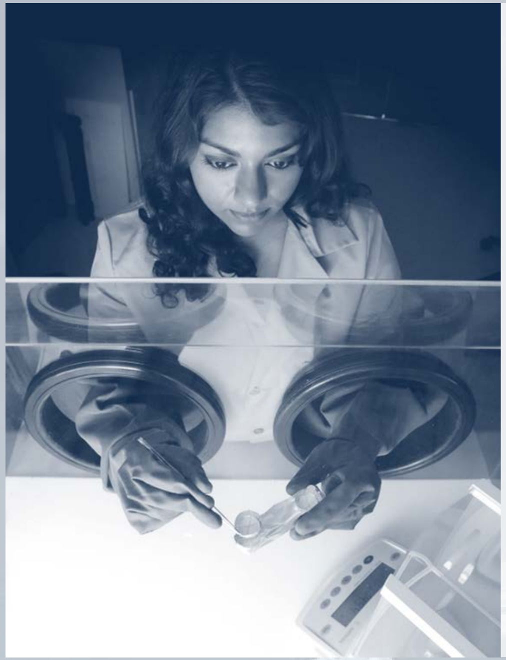

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