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

1. INTRODUCTION

1.1. ATLAS Instrument

The Ice, Cloud and land Elevation Satellite-2 (ICESat-2) is a NASA mission that will study the cryosphere to investigate the changes in the Earth's frozen and icy regions due to the warming climate. The spacecraft will carry a photon counting instrument called the Advanced Topographic Laser Altimeter System (ATLAS) that will measure the height of glaciers, ice sheets and sea ice, as well as, other ecosystems, which may include rain forests, deserts, and urban areas. During its orbit, this sole instrument built by NASA Goddard Space Flight Center (GSFC) has three main tasks to accomplish. First, ATLAS will generate a laser beam at a wavelength of 532 nm, which appears as green visible light on the electromagnetic spectrum. As illustrated in *Figure 1*, this single laser beam is split into six beams that are arranged in three pairs. *Figure 2* shows how each pair is separated by 3.3 km and has a width of 90 m. The laser also fires at a rapid rate of 10,000 pulses per second with the instrument taking measurements every 0.7 m along the satellite's ground path. [1-8]

Of the 200 trillion photons, or particles of light, that are rapidly-fired down at Earth, ATLAS is also tasked with collecting the dozen returning photons using its precisely aligned beryllium telescope. Lastly, the instrument will measure the travel times of each returned photon to calculate the distance between the spacecraft and Earth. However, before sending the beams to Earth, the photons must first travel through a series of optical components, such as lenses and mirrors, along the instrument's optical bench. The purpose of this critical pathway is multifold. It includes aligning the laser and the telescope, checking the wavelength of the laser, starting the timing mechanism, determining the size of the ground footprint, and finally, splitting the single laser into six beams. Therefore, protecting the critical laser optics on the ATLAS instrument is imperative to the successful operation of the satellite. [1-8]

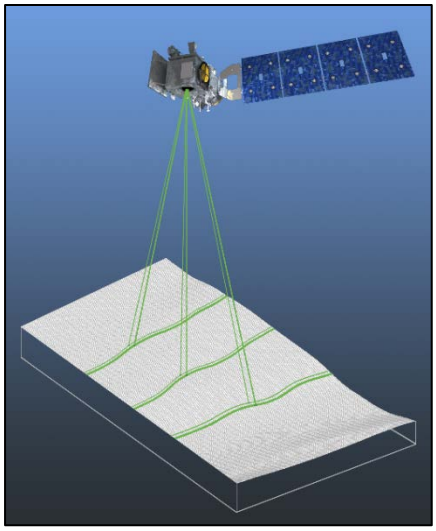


IMAGE CREDIT: *NASA/GSFC*

Figure 1. Illustration of ATLAS Laser Beam Pairs

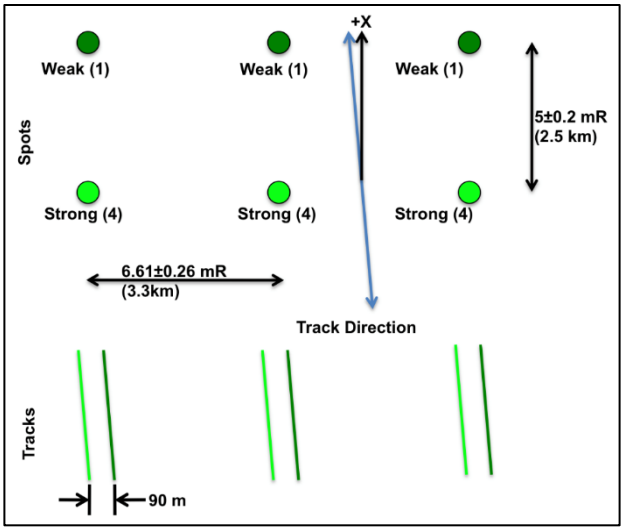


IMAGE CREDIT: *NASA/GSFC*

Figure 2. Illustration of ATLAS Laser Beam Pattern

Many have investigated the catastrophic impacts of contamination on laser induced optical damage and performance degradation in spaceflight laser systems.[9] Molecular contaminants, such as silicones and aromatic hydrocarbons, are particularly known to damage laser optics.[9] Consequently, it is important to protect the optical components from potential molecular contaminants throughout the various phases of a NASA mission. Although molecular contamination is often times inevitable, the risk of exposure to it can be alleviated with various contamination control mitigation methods, particularly during the Integration and Test (I&T) phases of the project. One such method is the strategic placement of Molecular Adsorber Coating (MAC) samples during thermal vacuum (TVAC) testing of flight hardware in vacuum test chambers.

1.2. Molecular Adsorber Coatings

MAC is a sprayable coatings technology that was developed at NASA GSFC to address outgassing concerns for flight or ground-based space applications. The coating is comprised of zeolite-based, porous materials that passively capture outgassed contaminants within its crystalline structure. MAC also has low outgassing properties due to its composition of inorganic materials. Past research studies have shown that the coating is effective in trapping high molecular weight chemical species, such as hydrocarbons, silicones, plasticizers, and other outgassed constituents from common spaceflight materials. The MAC technology can be used on-orbit, particularly on or near sensitive surfaces and components on the spacecraft, such as instrument cavities, electronics boxes, and detectors. For example, MAC plates were installed in the Far Ultraviolet (FUV) instrument of NASA's Ionospheric Connection Explorer (ICON) mission to address on-orbit material outgassing within the instrument. [10-14]

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. For example, MAC panels were installed throughout the Chamber A test facility at NASA Johnson Space Center (JSC) during the four major cryogenic vacuum tests for the James Webb Space Telescope (JWST) from 2014 to 2017. The coating samples were affixed along various locations in the chamber to passively capture the residual contaminants from the chamber environment during the testing of JWST's optical ground support equipment (OGSE), thermal pathfinder (TPF), and optical telescope element and integrated science (OTIS) instruments. MAC samples were also used during smaller component-level vacuum testing, such as on the Magnetosphere Multiscale Mission (MMS) in 2014, the Neutron star Interior Composition Explorer (NICER) in 2015, the Global Ecosystem Dynamics Investigation lidar (GEDI) in 2016, as well as, during instrument-level testing for the Global-scale Observations of the Limb and Disk (GOLD) mission in 2017. [13-16]

Similarly, the MAC technology was proposed for use during two recent ATLAS vacuum chamber tests. The purpose of using MAC during the TVAC tests was to isolate and protect the critical laser flight optics on the ATLAS instrument from any potential outgassing sources. These sources may originate from commonly used spaceflight materials and components, such as staking compounds, adhesives, epoxies, cables, wires, isolator systems, and batteries. Other outgassing sources also include non-baked items and unknown residual contaminants from the TVAC test set-up or the chamber environment. Time-temperature bake-outs were performed on most items prior to the tests; however, this method does not completely eliminate outgassing, especially from materials comprised of silicones or elastomers. These potential sources may contribute to molecular reflection that would not be indicative of the on-orbit flight case due to the confined chamber and the warm walls of the chamber. Lastly, the location of existing facility scavenger cold plates would not isolate the critical optical components from these outgassing sources.

2. APPROACH

2.1. Sample Fabrication

NASA GSFC custom-made samples from aluminum alloy substrates. *Figure 3* shows the samples that were coated with the white version of the MAC technology. The average total coating thickness on the samples was 0.16 mm, or 6.2 mils. The coating area per sample was approximately 95.2 cm^2. [17]

IMAGE CREDIT: *NASA/GSFC*

Figure 3. White MAC Samples for ATLAS TVAC Chamber Tests

2.2. Sample Exposure

Two of the MAC samples shown in *Figure 3* were used during vacuum chamber tests with the ATLAS instrument. A summary of the TVAC exposure periods is shown in *Table 1*. The first sample was installed for TVAC A in mid-2017. The sample was exposed to the chamber facility for approximately 50 days. The second sample was installed in a different vacuum chamber for TVAC B in late 2017. This sample was exposed to the chamber environment for a shorter duration of approximately 30 days.

TVAC Test ID	Chamber ID	Sample Installation	Exposure Period
A	X	Mid 2017	~ 50 days
B	Y	Late 2017	~ 30 days

Table 1. Summary of TVAC Chamber Test Exposure Periods

2.3. 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. As shown in *Figure 4*, this target location is between the Laser Reference System (LRS) Sunshade and the LRS Optics Radiator of the ATLAS instrument. More specifically, the sample was installed near a blanket vent as shown in *Figure 5*. This proposed location is important because the critical pathway for the transmit optics components on the optical bench is housed below the blanket vent. These components are responsible for directing the laser from its source on the instrument.

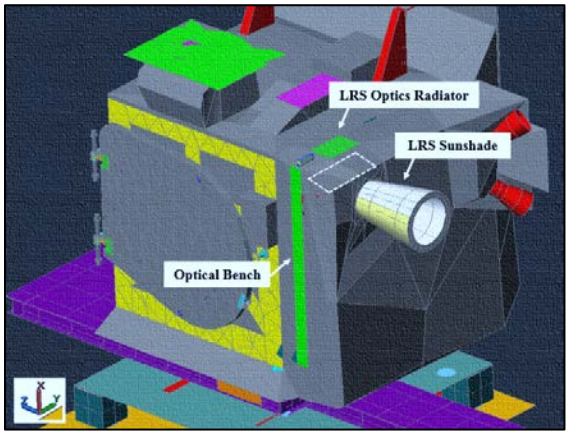


IMAGE CREDIT: *NASA/GSFC*

Figure 4. Thermal Model of ATLAS Instrument

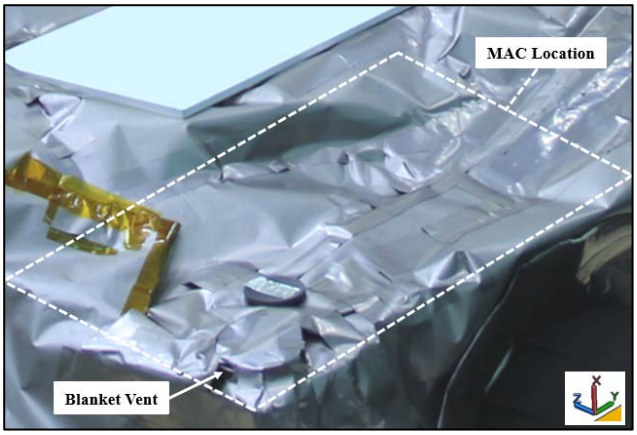


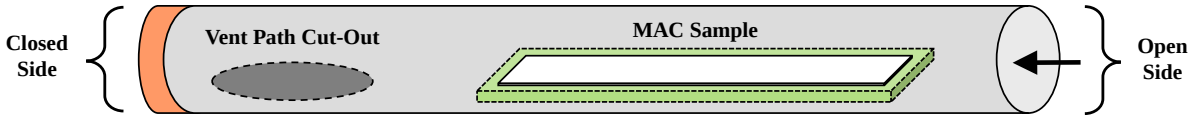
IMAGE CREDIT: *NASA/GSFC*

Figure 5. Location of MAC Sample during TVAC Tests

2.4. Sample Configuration

Prior to MAC installation efforts, an oval shaped channel was constructed using a sheet of Vapor Deposited Aluminum (VDA) Single Layer Insulation (SLI) blanket material. The purpose of the channel is to restrict the amount of outgassed molecular species from the test environment that may contaminate the critical transmit optics along the vent path. As illustrated in *Figure 6*, an appropriately sized hole was cut-out on the bottom of the channel to place over the vent at the proposed installation location. *Figure 6* also illustrates the placement of the MAC sample inside the constructed blanket channel.

The side closer to the vent cut-out was closed off with Kapton tape as depicted in *Figure 6*. This directs the entrance of any potential contaminants through the open side of the narrow oval shaped blanket piece. The outgassed species that enter the channel will pass the MAC sample and likely strike the surface of the coating. At that point, the coating will passively capture the contaminant before it can continue on. The same is expected for any outgassed species that may ascend from the vent path, as well. *Figure 7* shows the fabricated channel with the MAC sample installed in the proposed location prior to the start of the TVAC tests.



NOT DRAWN TO SCALE

Figure 6. Design of Blanket Channel with MAC Sample

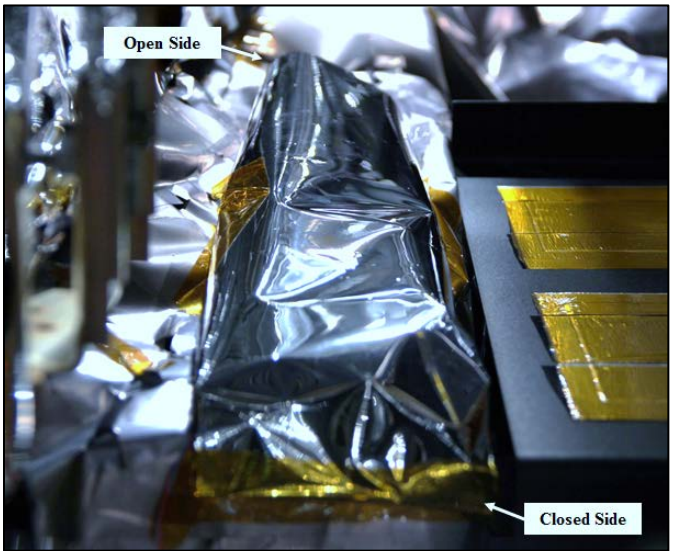


IMAGE CREDIT: *NASA/GSFC*

Figure 7. Installation of Blanket Channel with MAC Sample

2.5. Temperature Profile

Figure 8 shows the thermocouple that was attached to monitor the temperature profile of the sample throughout the duration of the tests. As illustrated in *Figure 9*, the MAC sample reached temperatures between -78 and 36 °C during the second TVAC test. The sample from TVAC A was also exposed to similar temperature cycles during vacuum testing.

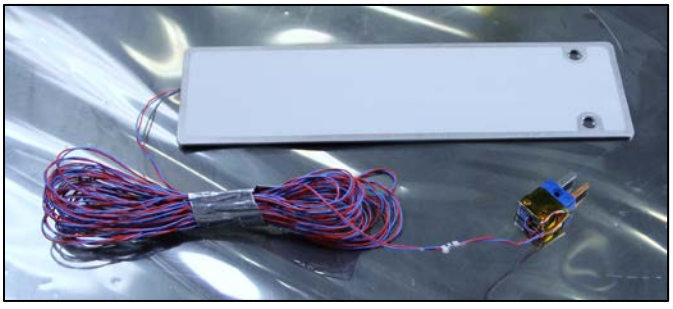


IMAGE CREDIT: *NASA/GSFC*

Figure 8. MAC Sample with Thermocouple

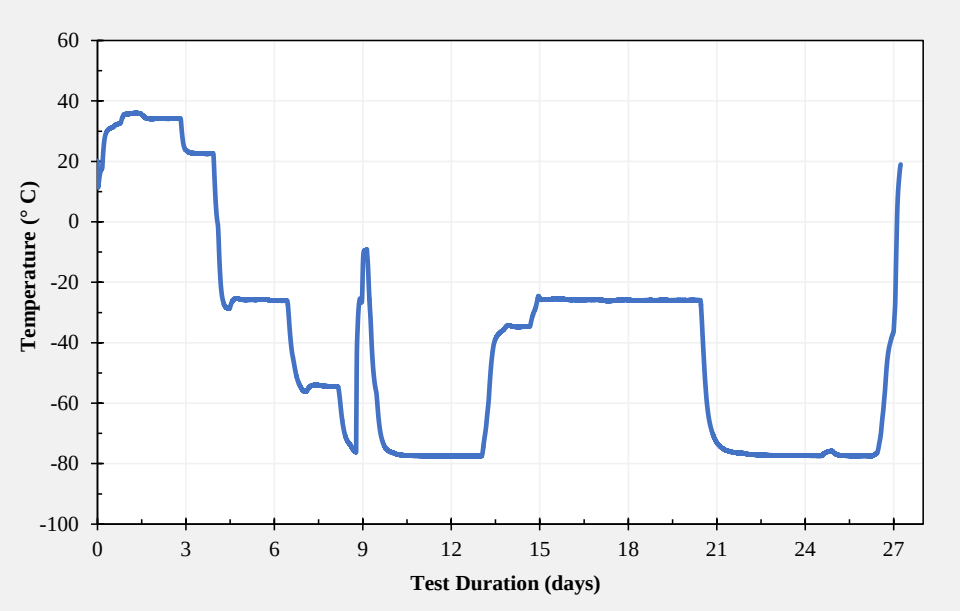


Figure 9. Temperature Profile of MAC Sample during TVAC B Test

3. TEST METHODS

3.1. Solvent Rinse Methods

At the end of each TVAC test, the blanket channel was carefully removed from its installed location. The contaminated MAC sample inside the channel was immediately sealed, and submitted for analysis. A rinse analysis was performed on the samples by directly rinsing the surface of the coating with an organic solvent of choice. This method was used to extract contaminants that were adsorbed on the coating, but only those that can be dissolved with the selected solvent. Previous studies have demonstrated that chloroform is an effective solvent that can dissolve common chemical species of interest, such as various hydrocarbons and silicones. These studies have also shown that multiple rinses of the coating surface removes additional contaminants. However, the dissolved species were observed to decrease with each consecutive rinse. [15-16]

As described in *Table 2*, three MAC samples were analyzed using the proposed solvent rinse method. All samples were coated at the same time in March 2016, and were kept sealed in polyethylene bagging material when not in use. Two of the samples are designated as contaminated because they were exposed to the two chamber facilities during the TVAC tests. A control sample with no exposure to a vacuum test facility was also used to establish a baseline reference for comparison purposes. This control sample will provide insight into the residual contaminants that the coating may have collected 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

Table 2. Summary of Analyzed MAC Samples

The MAC samples were rinsed four times with optima-grade chloroform as shown in *Table 3*. First, the surface of the coating was rinsed with a single pass of the solvent. The rinsate from each sample was collected and allowed to evaporate to dryness in a pre-weighed dish. Next, the surface of the sample was rinsed three times with chloroform. The purpose of this multiple rinse was to dissolve any remaining contaminants in the coating. The rinsate of this triple solvent pass was also collected in another pre-weighed dish and allowed to completely dry. The dishes for each rinse type were then weighed to determine the remaining amount of Non-Volatile Residue (NVR).

Rinse Type	Total Rinses	Rinse Number	Rinse Solvent
Single	1	Rinse 1	Chloroform
Triple	3	Rinses 2, 3, 4	Chloroform

Table 3. Summary of Solvent Rinses on MAC Samples

3.2. Chemical Analysis Methods

The sample rinsates were also evaluated using two chemical analysis methods to obtain a general approximation of the types and relative amounts of contaminants in the NVR. First, Fourier Transform Infrared Spectroscopy (FTIR) was performed on the sample rinsates using a Thermo Fisher Scientific Nicolet 6700 instrument. Second, pyrolysis-Gas Chromatography/Mass Spectrometry (GC/MS) was performed using a Shimadzu Scientific Instruments QP2010 Ultra and GL Sciences Optic-4 Inlet instrument. For pyrolysis, the collected NVR was placed in a micro-vial inside a liner and heated in the GC outlet at a high rate of 30 °C per second to an elevated temperature of 600 °C. The volatile and semi-volatile chemical species that evolved from this thermal decomposition phase were introduced to the GC column interface with the MS. The non-volatile chemical species remained in the micro-vial to avoid inlet contamination of the instrument.

4. RESULTS & DISCUSSION

4.1. Solvent Rinse Results

Figure 10 illustrates the NVR results from the single and triple pass solvent rinses. The error bars shown in the plot are associated with the weighing uncertainty of ± 0.04 on the laboratory balance that was used for the gravimetric measurements. The NVR difference between the initial rinse of the TVAC A sample and the control sample shows that the amount collected on the TVAC exposed MAC is relatively low at roughly 0.21 mg. In contrast, the NVR difference between the initial rinse of the TVAC B sample and the control sample shows that the amount collected on the TVAC exposed MAC was about 6.2 times greater at 1.3 mg. Similar trends are observed for the triple pass rinse for both TVAC exposed samples. Furthermore, the decreasing mass observed in the triple pass rinse suggests that most of the contaminants were removed in the first initial rinse.[18-19]

Table 4 shows the cumulative NVR and NVR per coating area for the total four rinses. The results indicate that the cumulative NVR for the second sample is about 3 times greater than the control sample, and about 2.3 times greater than the sample that was exposed during the first test. The results suggest that the exposed sample from TVAC B captured the most contaminants, even though, it was exposed for a shorter duration. Some possible reasons for this may include differences in the test facility, the chamber size, the temperature variations, and the sample handling procedures. [18-19]

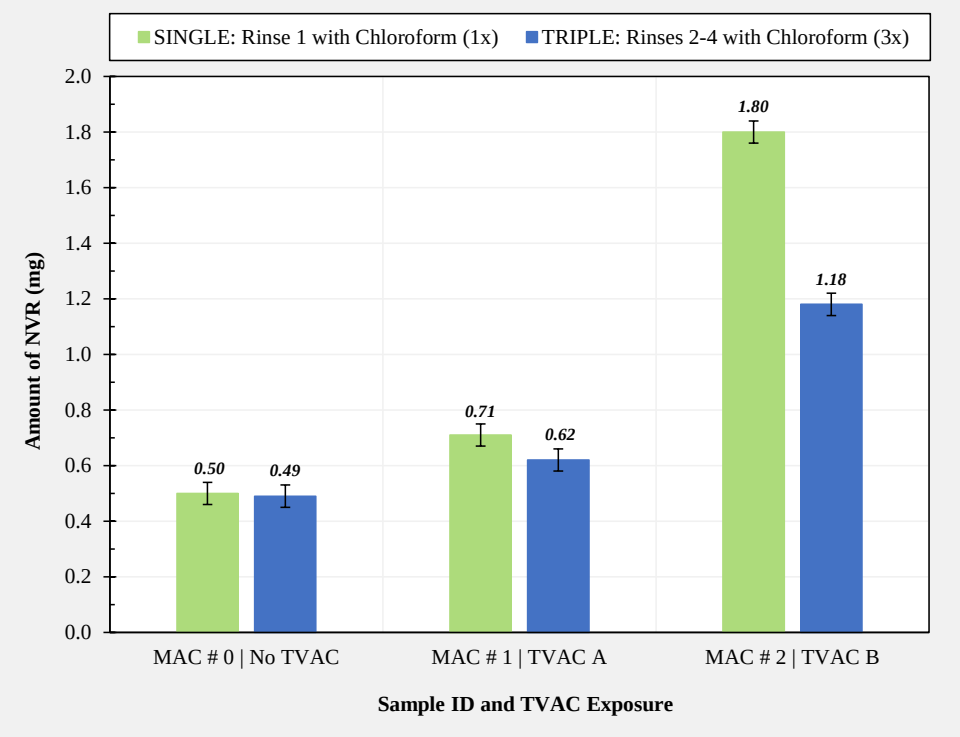


Figure 10. Amount of NVR per Solvent Rinse Type

Sample ID	Cumulative NVR	Cumulative NVR per Coating Area
MAC # 0	0.99 mg ± 0.04	10.40 µg/cm^2 ± 0.44
MAC # 1	1.33 mg ± 0.04	13.97 µg/cm^2 ± 0.44
MAC # 2	2.98 mg ± 0.04	31.29 µg/cm^2 ± 0.44

Table 4. Cumulative NVR Mass and Cumulative NVR per Coating Area

4.2. Molecular Adsorption Capacity

The cumulative NVR per coating area of the exposed samples were compared to past experimental data of MAC samples that were fully saturated with a contaminant source at 45 °C for durations between 88 and 160 hours in vacuum conditions. The test contaminant is stearyl alcohol, which is an eighteen-chain hydrocarbon that is representative of the outgassed species that are commonly found in spaceflight applications. The experimental data shown in *Figure 11* demonstrate that the vacuum molecular adsorption capacity of MAC is directly proportional to the total coating thickness. [10-16]

The MAC samples that were used for the two TVAC tests measured a coating thickness of 6.2 mils and a coating area of 95.2 cm^2. Therefore, from *Figure 11*, the molecular adsorption capacity per sample is approximately 2.4 mg/cm^2. This suggests that the samples have the possibility of collecting up to 228 mg of molecular contaminants in vacuum conditions. However, the estimated maximum mass assumes that the majority of the chemical species collected are long-chain hydrocarbons similar to stearyl alcohol.

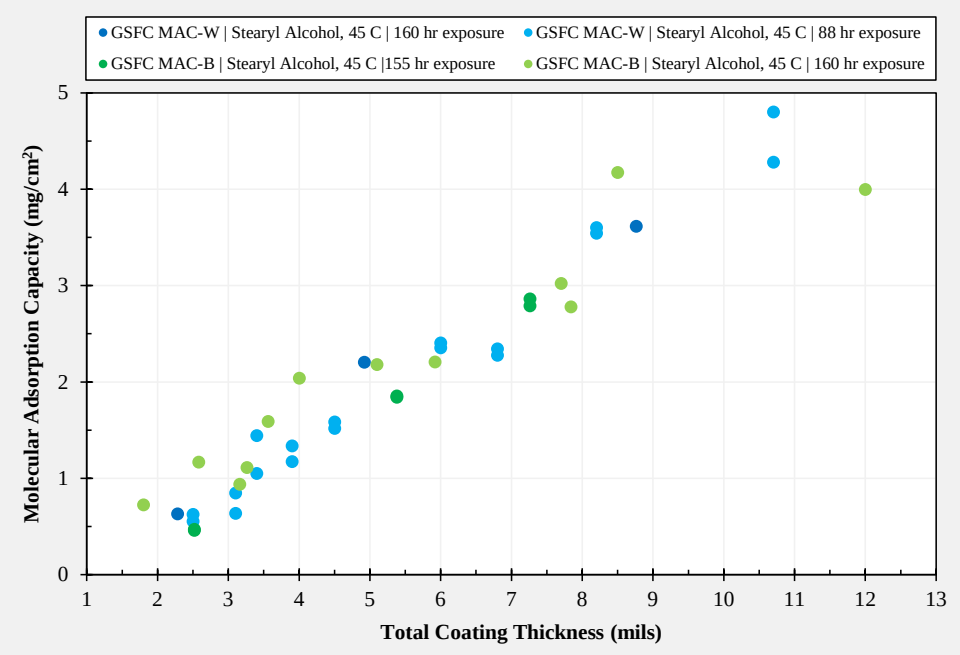


Figure 11. Experimental Molecular Adsorption Capacity as a Function of Coating Thickness in Vacuum Conditions

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. Some considerations include that not all of the adsorbed contaminants will be removed with multiple solvent rinses, or will be long-chain hydrocarbons similar to the experiment contaminant. The saturation ratio for the first TVAC sample and the second TVAC sample are 0.6 and 1.3 percent, respectively. This suggests that the samples 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.

4.3. Chemical Analysis Results

Figure 12 illustrates the percent distribution of chemical species from the pyrolysis-GC/MS analysis for the single solvent rinse of the control MAC sample. The results indicate that 99 percent of the single rinse from the control sample 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. The remaining 1 percent of miscellaneous chemical species are likely room environmental compounds. The triple rinse of the control sample also showed similar results. [18-19]

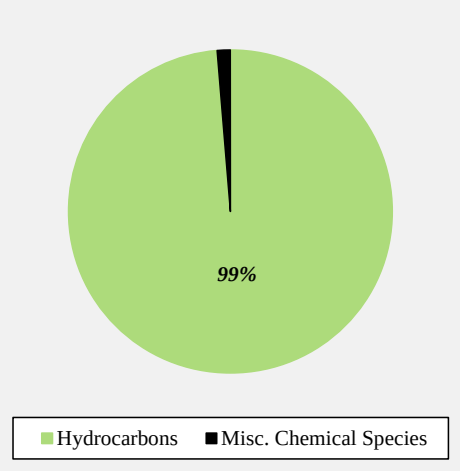


Figure 12. Percent Distribution of Chemical Species from Initial Rinse of Control Sample

Figure 13 demonstrates the percent distribution of the identified chemical species from the pyrolysis-GC/MS analysis of the single rinse for the TVAC exposed MAC samples. The most abundant type of chemical species that were collected on the coating during both TVAC tests were hydrocarbons at 90 percent and 79 percent for the first sample and the second sample, respectively. For TVAC A, the second most abundant species at 8 percent were silicones, such as methyl phenyl silicones and methyl silicones. These silicone-based species are commonly sourced from lubricants, elastomers, and adhesives. The remaining least abundant species at 2 percent from the first sample consisted of plasticizers, such as phthalate-based species, as well as, other miscellaneous chemical constituents, such as palmitate-based species. [18-19]

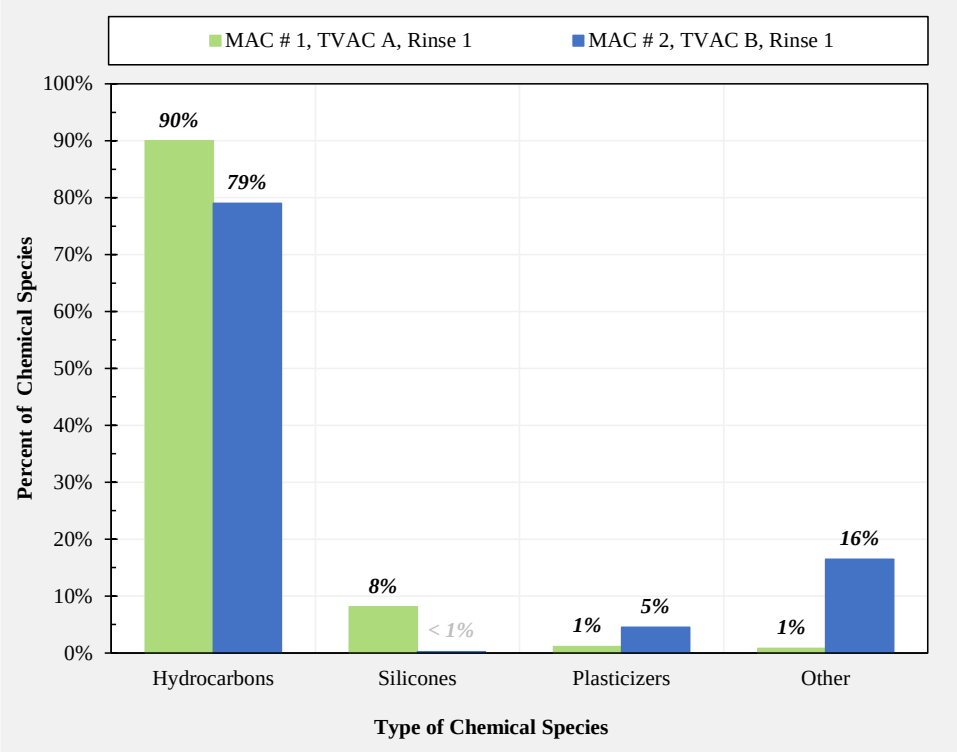


Figure 13. Percent Distribution of Chemical Species from Initial Rinse of TVAC Exposed Samples

In comparison, the second most abundant species for the sample exposed during TVAC B were common handling contaminants at 16 percent as shown in *Figure 13*. These contaminants consisted of mostly palmitate-based species, such as isopropyl palmitate. This compound is commonly found in cosmetic products, such as emollients, moisturizers, and thickening agents, as well as, lubricants, rubber, and latex. Other handling contaminants included methyl palmitate, butyl palmitate, and isopropyl myristate. In addition, phthalate-based plasticizers, such as di(2-ethylhexyl) phthalate, were also detected in the single rinse of the second sample at 5 percent. The least abundant species identified in the second sample were silicone-based compounds at 0.2 percent. The triple rinse of both TVAC exposed samples also showed similar trends to the single rinse results. [18-20]

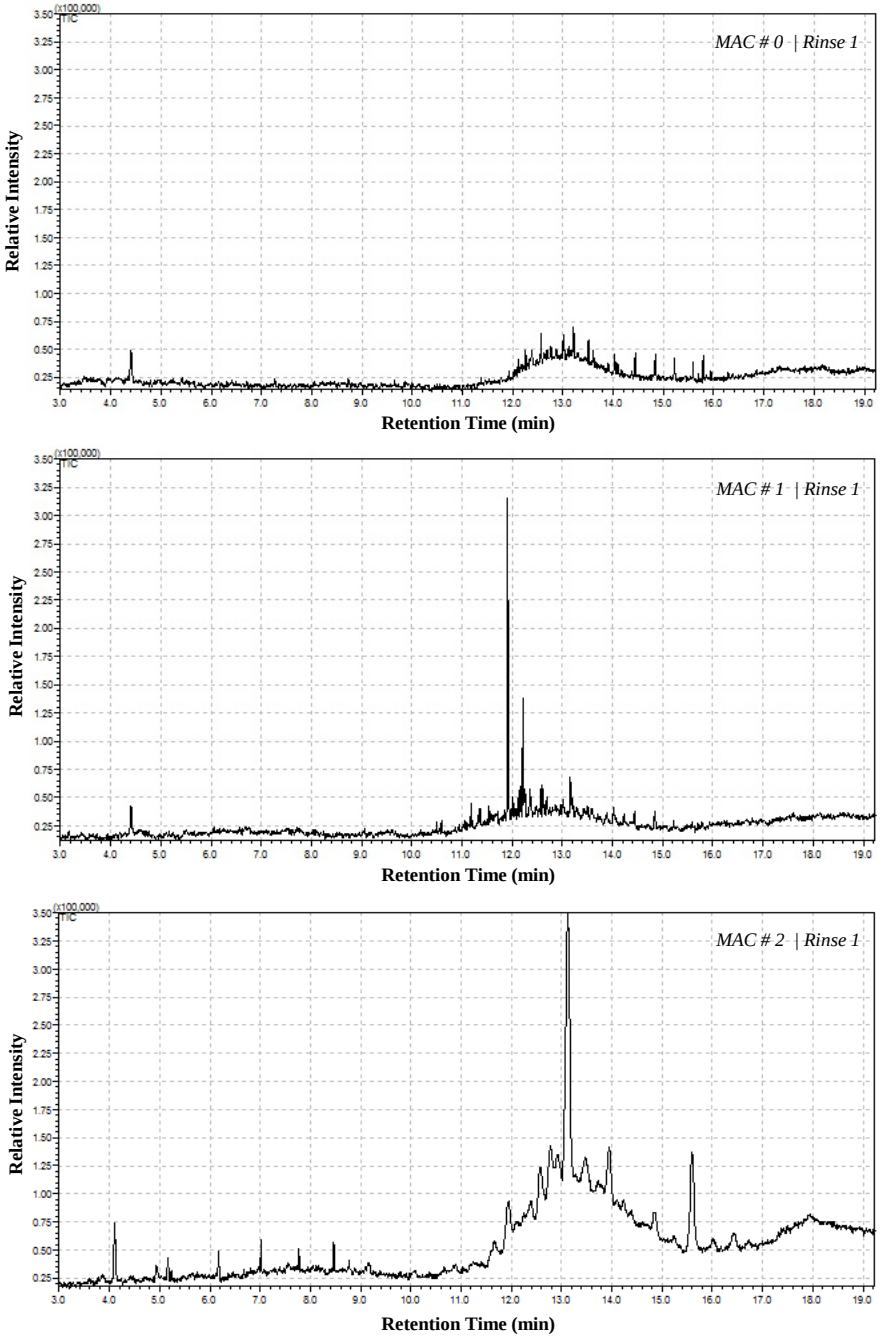


Figure 14. Comparison of GC/MS Plots for Initial Rinse of MAC Samples

Figure 14 illustrates the comparison of the GC/MS plots for the single rinse on the samples. The plots show that the relative intensity, or compound abundance, of identified species for the exposed samples are greater than the control sample. For example, the highest intensity peak for silicones was observed on the exposed sample from TVAC A. In particular, the second plot in *Figure 14* for the TVAC A sample shows a peak for methyl phenol silicone at around 11.9 min. Similarly, the third plot in *Figure 14* for the TVAC B sample shows many peaks for palmitate-based species, such as isopropyl palmitate at around 13.2 min, and plasticizers, such as di(2-ethylhexyl) phthalate at around 15.6 min.

4.4. Comparison to Contamination Monitoring Methods

The solvent rinse NVR and chemical analysis results for MAC were compared against typical contamination control monitoring methods, which are often used during spaceflight vacuum chamber testing. These methods include witness foils, scavenger plates, and cold fingers. A witness foil was deployed during each test and placed in a subject location near the ATLAS instrument in the chamber facility to collect NVR representative of what may be found on the hardware. The coating and foil samples, however, were not installed in the same location. Additionally present in each TVAC chamber was a scavenger plate and a cold finger. A scavenger plate is a cold panel cooled by liquid nitrogen (LN2) that is used to collect the majority of the outgassed contamination throughout the TVAC test. A cold finger is a small cylindrical device also cooled by LN2 that is used to collect the residual outgassing during the last several hours of the test. [21-22]

A chemical analysis comparison of the type of NVR collected by the witness foils, scavenger plates, and cold fingers can provide confirmation of the contaminant species identified from the coating samples that were installed for the two TVAC tests. Following TVAC, the witness foils were tested in a similar method, in which they were rinsed with chloroform and analyzed using FTIR and GC/MS methods. The analysis of the witness foils showed similar chemical species to the MAC samples, such as hydrocarbons, silicones, plasticizers, and other lesser abundant species of mostly organic acid derived constituents. Furthermore, rinses with isopropyl alcohol of the facility scavenger plates and cold fingers from the TVAC tests and post-TVAC bake-out runs show predominately hydrocarbons, silicones, and plasticizers, as well. These results provide additional verification of the identified species on the MAC samples. [18-19, 23-27]

A solvent rinse NVR comparison was made between the witness foils and the MAC samples given that they are both passive collection methods. *Table 5* shows a summary of the single rinse NVR per surface area between the two witness foils and the two coating samples. For TVAC A, the NVR from the coating was about 22 times greater than the witness foil. Similarly, the results from TVAC B show that the NVR measured from the coating was about 195 times greater than the witness foil. [18-19, 23-27]

TVAC Test ID	**Witness Foil** *Rinse 1 with Chloroform*	**MAC Sample** *Rinse 1 with Chloroform*
TVAC A	0.10 μg/cm^2 ± 0.02	2.20 μg/cm^2 ± 0.44
TVAC B	0.07 μg/cm^2 ± 0.02	13.65 μg/cm^2 ± 0.44

Table 5. Single Rinse NVR per Surface Area of Witness Foils and MAC Samples

The significantly larger relative amounts that were collected on the MAC samples as compared to the witness foils do not provide conclusive evidence that condensation would have taken place on the instrument due to the physical and chemical differences between the coating and the hardware surface. The results, however, do suggest that the coating may serve as a better method for both mitigation and indication of contaminant threats near spaceflight hardware during vacuum chamber tests. Unlike other typical monitoring methods, contaminants that are passively captured within the coating during TVAC testing are less likely to be released during warm-up activities to ambient pressures. [18-19, 23-27]

5. CONCLUSIONS

In conclusion, the use of the MAC technology during ATLAS TVAC testing was effective in protecting the critical laser flight optics on the instrument. The identified chemical species that were captured by the coating isolated the transmit optics components from potential molecular contamination. As a result, 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. Future work may include exploring alternative methods of identifying the collected contaminants on the coating, such as via thermal desorption techniques or the use of other organic solvents.

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Thermal Coatings	Alfred Wong	SGT, Inc	Code 546
	Mark Hasegawa	NASA GSFC	Code 546
Contamination Control	David Hughes	NASA GSFC	Code 546
	BonnieBlue Valentin-Santiago	SGT, Inc	Code 546
	Brandon Stergiou	SGT, Inc	Code 546
	Dong-Shiun Lin	SGT, Inc	Code 546
Thermal Engineering	Deepak Patel	NASA GSFC	Code 545
	Matthew Garrison	NASA GSFC	Code 545
	Heather Bradshaw	NASA GSFC	Code 545

Table 6. Project Team Member Acknowledgements

NASA National Aeronautics and Space Administration
GSFC Goddard Space Flight Center
SGT, Inc Stinger Ghaffarian Technologies, Incorporated
Code 541 Materials Engineering Branch
Code 545 Thermal Engineering Branch
Code 546 Contamination and Coatings Engineering Branch

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