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A Preliminary Study on the Use of a NASA Developed Coatings Technology for Protecting Natural Science Collections from Molecular Contaminants

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Abstract

Many museum conservators and collection managers are faced with the challenge of molecular contaminants that can promote the degradation of specimens on display in exhibits or in cabinets at storage facilities. This has prompted the need to explore innovative techniques to alleviate the presence of chemical species that originate from atmospheric off-gassing of materials or cross-contamination among collection items. For example, the Smithsonian Institution's National Museum of Natural History (NMNH) has tackled this problem for many years, specifically targeting contaminants, such as mercury vapor, at its Museum Support Center (MSC) storage facility in Suitland, Maryland.

Similarly, the presence of molecular contaminants poses a significant threat for NASA science and exploration missions. The deposition of chemical species on sensitive surfaces can degrade the performance and operational lifetime of satellites, telescopes, and instruments. As a result, a sprayable zeolite-based coatings technology was designed to passively capture molecular contaminants and reduce the risks associated with material outgassing in vacuum environments for aerospace applications. This technology, called the Molecular Adsorber Coating (MAC), was developed at NASA Goddard Space Flight Center (GSFC). MAC has been extensively used during thermal vacuum chamber testing of various spaceflight hardware and components, such as for the James Webb Space Telescope (JWST). The coating is also planned to fly aboard upcoming NASA missions to address on-orbit outgassing concerns within instrument and laser cavities.

Recently, the MAC technology was evaluated as a possible solution for protecting the Smithsonian Institution's natural science specimens, specifically its mineral ore and botany collections at the MSC storage facility. The initial year-long study between NASA GSFC and NMNH involved investigating the effectiveness of the MAC technology in capturing molecular contaminants that are present within the collections and storage cabinets at ambient, non-vacuum conditions. The work included sample fabrication, installation and retrieval efforts, testing efforts and associated challenges, preliminary findings, and future plans for the multi-year project.

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- Impact of Molecular Contamination on NASA Missions
- Molecular Adsorber Coatings
- Current NASA Applications of MAC
- Sources of Off-gassing from Collection Items
- Contamination Mitigation Methods

- The presence of molecular contaminants can pose a significant threat to NASA science and exploration missions
- Sources can originate from commonly used spacecraft materials that outgas (or release molecules) during vacuum testing or during spaceflight operations
 - e.g. adhesives, lubricants, epoxies, potting compounds
- Molecular contaminants can deposit on critical surfaces, such as optics, electronics, laser systems, detectors, baffles, solar arrays, thermal coatings, and vacuum chambers
- This can degrade the performance and operational lifetime of satellites, telescopes, and instruments



JWST in the High Bay Cleanroom at GSFC



ATLAS instrument on ICESat-2 emitting laser pulses

- The Molecular Adsorber Coating (MAC) is a sprayable zeolite-based coatings technology that was developed at NASA Goddard Space Flight Center (GSFC)
- MAC has a highly porous structure that is designed to passively capture molecular contaminants
- As a ground tested and flight qualified coating, MAC is effective at trapping high molecular weight chemical species at representative spaceflight conditions, such as under high vacuum pressures and moderate temperature ranges
 - e.g. hydrocarbons, silicones, plasticizers, and other outgassed constituents from common spaceflight materials



White and black colored variations of the MAC technology



Development of MAC at NASA GSFC



Scanning Electron Microscope (SEM) image illustrating porous structure

Current NASA applications include its use:

- During vacuum testing of critical hardware and components to mitigate the risk of molecular contaminants from the chamber environment
- Within instrument and laser cavities to address on-orbit outgassing concerns

Example: James Webb Space Telescope (JWST)

MAC samples were installed in Chamber A at NASA Johnson Space Center (JSC) in Houston, TX during several cryogenic thermal vacuum (TVAC) test efforts of JWST's flight hardware and optical ground support equipment (OGSE) from 2014 to 2017



Installation efforts for the custom-fabricated MAC samples in the plenum located just below the chamber. Chamber A is the largest high vacuum, cryogenic optical test chamber in the world. It has a diameter of 55 feet and is 90 feet tall.

E CREDIT: NASA/CHRIS GUNN

Some NMNH specimens and objects are known to off-gas as a function of materials that are:

- Inherent in the specimens and objects
 - e.g. Mercury vapor from mercury ores



Mineral ores, such as cinnabar, at the National Museum of Natural History (NMNH)

Some NMNH specimens and objects are known to off-gas as a function of materials that are:

- Deliberately applied to specimens and objects over time
 - e.g. Mercury salts applied to botanical specimens to reduce potential for mold and pests



Botanical specimens with mercury salt stains on mounted sheet paper and labels

Some NMNH specimens and objects are known to **off-gas** as a function of materials that are:

- Acquired by absorption or adsorption from other collections, storage furniture and supplies, and chemicals used in building systems and building maintenance
 - e.g. Mercury off-gassed from cabinet surfaces after initial contamination from mercury ores, and organic acids from poor quality storage materials



Visible dark stains of mercury contamination on cabinet surfaces

PAGE 9

Mitigation methods to alleviate the presence of contaminants include:

- Enclosing specimens in vapor impermeable *Marvelseal* bags
- Cleaning storage furniture with *mercury chelating agents* and *alcohol wipes*
- Installing various scavengers routinely used in museum conservation



Cleaning to remove visible mercury stains from the storage cabinets



Mercury ores enclosed in vapor impermeable Marvelseal bags

PAGE 10

- Purpose of Collaboration
- Objectives of Experiment
- Sample Fabrication Effort
- Sample Installation Effort
- Sample Retrieval Efforts
- Cabinet Descriptions
- Project Timeline

Interagency Collaboration between GSFC and NMNH

 Purpose: to study the use of MAC at ambient (non-vacuum) conditions to protect natural science collection items

Initial Year-Long Experiment at MSC

- Objective: to explore the use of MAC for mitigating off-gassed contaminants that are present within the storage cabinets
 - Residual contamination from mercury vapor
 - Other likely contaminants
 - e.g. organic and inorganic compounds

Past NMNH surveys have shown an abundance of other **organic and inorganic** contaminants inside collection storage cabinets

Museum Support Center (MSC)

is a Smithsonian Institution museum collections storage facility in Suitland, Maryland. The facility houses more than 54 million collection items in over 600,000 square feet of storage space.



GSFC and NMNH project team at MSC

Sample Fabrication

- GSFC custom-fabricated MAC samples in different size varieties
- Sample dimensions were 2" by 2" and 3" by 3"
- Magnets were affixed to the back side of each sample for easy installation onto the cabinet doors

Sample Installation

- MAC samples were installed in 3 cabinets at MSC
 - 2 "contaminated" cabinets and 1 "control" cabinet
- 37 samples were installed per cabinet; total 111 samples

Sample Retrieval

- MAC samples were retrieved at 1, 4, 8, and 12 month intervals
- 27 samples were removed from the cabinets for each of the 1, 4, and 8 month exposure periods
- Remaining 30 samples were removed from the cabinets for the 12 month exposure period







MSC Cabinet Descriptions

- Contaminated mineral science cabinet
- Contains mercury ore specimens that are enclosed in Marvelseal bags
- Cabinets were previously cleaned
- Scavenger sheets are present
- Contaminated botany cabinet
- Contains botanical specimens from 2 a collector known to have used mercuric chloride solutions
 - Control empty cabinet
- 3 Has never been used to store collection items

Cabinet 1: Mineral Science



Cabinet 3: Control



Cabinet 2: Botany





- Protocols Prior to Installation or Retrieval
- Mercury Vapor Concentration Measurements
- Protocols During Installation or Retrieval



GSFC Industrial Hygienist wearing a respirator and using a Jerome mercury vapor analyzer prior to a sample retrieval effort

Prior to the installation and/or retrieval of MAC samples

- Arizona Instrument's Jerome® J405 Gold Film Mercury Vapor Analyzer was used to measure the concentration of mercury vapor in each cabinet
- Initial concentration measurements were performed with the cabinet doors slightly cracked open
- Final concentration measurements were performed after leaving the cabinet doors **fully open** for a short duration, or until the concentrations dropped to an acceptable level
- Mercury vapor was present in both Cabinet 1 (Mineral Science) and Cabinet 2 (Botany), where the initial measurements were higher than the final measurements
- Highest levels of mercury vapor were detected in Cabinet 2 (Botany)
- No detectable mercury vapor in Cabinet 3 (Control)





Cabinet 1: Mineral Science



Sample Month Exposure

Cabinet 2: Botany

Cabinet 3: Control



GSFC Measurement (Cracked Open)

NMNH Measurement (Cracked Open)

GSFC Measurement (Fully Open)

NMNH Measurement (Fully Open)

ACGIH® TLV 8 hr TWA: American Conference of Governmental Industrial Hygienists Threshold Limit Value 8 Hour Time Weighted Average

OSHA PEL 8 hr TWA: Occupational Safety and Health Administration Permissible Exposure Limit 8 Hour Time Weighted Average

US Department of Labor, Annotated OSHA Z-2 Table for Mercury (Z37.8-1971)

Regulatory Limits	OSHA PEL ACC & 8 hour TWA	100 µg/m³
Recommended Limits	ACGIH [®] 2018 TLV 8 hour TWA	25 μg/m ³ elemental and inorganic
	NIOSH REL 10 hour TWA	50 µg/m³

Abbreviations

- ACC Acceptable Ceiling Concentration
- ACGIH American Conference of Governmental Industrial Hygienists
- OSHA Occupational Safety and Health Administration
- NIOSH National Institute for Occupational Safety and Health
- PEL Permissible Exposure Limit
- REL Recommended Exposure Limit
- TLV Threshold Limit Value
- TWA Time Weighted Average

Source: https://www.osha.gov/dsg/annotated-pels/tablez-2.html

During the installation and/or retrieval of MAC samples

- Respirators with mercury vapor absorbing cartridges were worn by all personnel working in or near the cabinets
- Personnel and area air monitoring samples were also collected using a Gilian GilAir Plus sampling pump
- Results indicate that airborne concentrations of mercury were significantly below the regulatory and recommended limits



GSFC engineer wearing a respirator and an air sampling pump during a MAC sample retrieval effort at MSC

- TD-GC/MS Overview
- Sample Collection Temperature Runs
- Test Method Advantages
- Internal Reference Standards

 MAC samples were evaluated using a sample introduction technique called Thermal Desorption-Gas Chromatography/Mass Spectrometry (TD-GC/MS)

Coating samples are placed in a heated thermal desorption sampling chamber to release the adsorbed chemical species

Regeneration temperature range of commercially available zeolites is typically between 175 to 315 °C

Volatile and semi-volatile compounds are collected in glass sample tubes containing Tenax-TA adsorbent traps

 Tenax-TA (60/80 mesh) adsorbents are made of porous, polymer resin (2,6-diphenylene oxide) and have a maximum temperature limit of 350 °C

Sample tubes are analyzed using a Shimadzu GC/MS QP2010 Ultra, in which the volatile compounds are desorbed using thermal energy (heat), then enter into a carrier gas stream (Helium) and lastly, are ionized and separated by mass



A sample tube with the released compounds is ready for placement in the sample holder on the GC/MS

PAGE 21

TD-GC/MS Sample Collection Temperature Runs

	RUN A	RUN B
HEATER PLATE	110 °C	250 °C
CHAMBER WALL	90 °C	220 °C
COLLECTION RANGE	20 to 110 °C	110 to 250 °C
PURPOSE	Captures the lower molecular weight (or boiling point) species	Captures the higher molecular weight (or boiling point) species

TD-GC/MS Method Advantages

- Provides better repeatability, recovery, detection, and quantification
- Can analyze a wide range of organic compounds (extremely volatile to semi-volatile)
- Can detect trace compounds that solvent extraction may not







TD-GC/MS testing of a 2" by 2" MAC sample using the Scientific Instrument Services (SIS) heated thermal desorption sampling chamber

- Internal reference standards of known concentrations were spiked into the adsorbent traps
- This provides a semi-quantitative estimate of concentrations for each unknown compound
- Response factors for all unknown compounds are assumed to be the same as the standard reference compound
- Simple peak area comparisons can be made between the reference and the unknown compounds

STANDARD	PURPOSE	
Benzene-d6	Confirms the detection of highly volatile compounds	
Toluene-d8	Provides a quantification peak	
Naphthalene-d8	Separates the volatile and semi-volatile compounds	
The internal reference standard methodology uses standards that are not found in nature (e.g. deuterated)		

VOLATILE SEMI-VOLATILE B-d6 T-d8 N-d8



TIME (min)



GSFC engineers analyzing the results of the TD-GC/MS test method

- Temperature Run Comparisons
- Percent Normalized GC/MS Peak Area
- Approximate Concentrations per Category
- TD-GC/MS Summary
- Additional Test Method Results

Comparison between Run A (110 °C) and Run B (250 °C)

- Same peaks are present for both temperature runs
- 250 °C runs have a greater relative abundance of species that are also present in the 110 °C runs
- 250 °C runs produce additional peaks for species that are not present in the 110 °C runs
- 250 °C runs result in a greater total concentration of identified chemical species than the 110 °C runs





Percent Normalized GC/MS Peak Area (%) at Run B Conditions







PAGE 28



TD-GC/MS Summary

 Preliminary results show the presence of several organic compounds, but do not show the presence of any residual mercury compounds

Additional Test Method Results

 Test methods, such as solvent rinse extraction and x-ray fluorescence (XRF) analysis, were also performed; however, results did not show any elemental or organic/inorganic mercury



Shimadzu GC/MS QP2010 Ultra used for testing efforts at GSFC

Closing Remarks

- Conclusions
- Future Work
- Acknowledgements
- Reference Materials

Conclusions & Future Work

Conclusions

- MAC collected off-gassed contaminants during its exposure within the collection storage cabinets at MSC
 - Various hydrocarbons
 - Compounds containing oxygen (e.g. alcohols, aldehydes, carboxylic acids, epoxies, esters, ethers, ketones, and phenols)
 - Compounds containing silicon, nitrogen, phosphorus, sulfur and chlorine

Future Work

- Further interpret and analyze the TD-GC/MS data to identify the specific chemical species within each category and approximate concentrations
- Perform residual gas analyzer (RGA) experiments to detect mercury (or other unique compounds) under vacuum conditions
- Perform coating variation studies to target off-gassed contaminants of interest
- Run similar exposure experiments at museum exhibits or additional storage cabinets for the purposes of collecting hydrocarbons and other residual species of interest



MAC samples installed in Cabinet 3 (Control) for the initial year-long experiment at MSC

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Reference Materials

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