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"Analysis of Volatile Organic Compounds on Mir Station"

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I. INTRODUCTION

The goal of this research is the characterization of volatile organic compounds (VOCs) in air samples from Mir Space Station using new technology based on ion trap mass spectrometry (ITMS). Twenty-four hour time-averaged samples will be collected onto cartridges using the US Solid Sorbent Air Samples (SSAS). Grab samples will be collected using US Grab Sample Containers (GSC). Samples will be transferred from Mir via the Space Shuttle, forwarded to the Toxicology Laboratory at NASA Johnson Space Center (JSC) for analysis and sample subdivision, and then sent on to San Francisco State University (SFSU) for the purposes of this work. Standard operating procedures, quality control samples, and confirmatory experiments will be employed to ensure reliable, high quality data. Analyses will be performed via a modified form of EPA-approved gas chromatography/mass spectrometry (GC/MS) methods [1] and new techniques based on direct sampling ion trap mass spectrometry (DSITMS). Significant effort will be put into developing, testing, and demonstrating DSITMS techniques with the requisite sensitivity, selectivity, and speed for real-time monitoring of trace-level contaminants in air. The results of this research will provide detailed information on the types and concentrations of VOCs in the Mir environment. Moreover, the demonstration of new technology and comparison against proven methods will yield valuable information on the feasibility of its use for monitoring air quality in advanced life support systems.

A. Hypotheses

1. The types and concentrations of VOCs in the Mir Station atmosphere are not well characterized. Detailed information on the types and concentrations of VOCs in the Mir Station atmosphere is required to assess the toxicological risks of long-term human exposure to this environment.
2. Detailed information on the types and concentrations of VOCs in the Mir Station atmosphere can be obtained through the use of the same proven methods employed for previous US space missions. These methods, based on the use of both cartridges and grab samples for sampling, and GC/MS for analysis, represent the most reliable, cost-effective means for characterizing this environment.
3. New technology based on DSITMS shows excellent promise for on-line, real-time monitoring of VOCs. Demonstration of this technology on air samples collected from Mir Station can provide a means for intercomparison with conventional GC/MS methods and provide a measure of its potential for environmental health assessments and advanced life support applications.

B. Objectives of Investigation

1. Characterize Mir Station atmosphere via proven sampling and analysis strategies.
2. Demonstrate the use of DSITMS for direct monitoring of VOCs in air samples collected from Mir Station.
3. Document the types and concentrations of VOCs on Mir Station and analyze results in collaboration with other science investigators to further the goals of the NASA Research Announcement (NRA).

C. Background / History of Project

Palmer and the JSC Toxicology Lab have signed a Memorandum or Understanding (MOU) that details the nature of their collaborative research on characterization of VOCs in Mir air samples. The JSC Toxicology Lab has extensive experience on space station air analysis and flown SSAS and GSC units on a variety of prior NASA missions ranging from the Apollo through the Shuttle programs [2-6]. Palmer and Belisle have additional experience analyzing for VOCs in air samples collected in various locations in the San Francisco Bay Area [7], boreal ecosystems [8], and bioregenerative life support chambers [9,10].

Palmer's mission experience includes a set of "practice" samples from the Mir 19 mission [11]. These samples enabled the testing of analytical methods and instrumentation, and the garnering of practical

experience on space station air samples. Analytical results on these samples, that is compounds tentatively identified and their concentrations, were reviewed with researchers at the JSC Toxicology Lab and generally showed good agreement.

Palmer and Belisle's mission experience also includes two sets of samples from the Mir 21 mission. The first set was analyzed via GC/MS methods in Palmer's labs. These results are documented in a final report that was recently submitted to NASA and the JSC Toxicology Lab [12]. The second set was received later and analyzed by GC/MS methods in Belisle's labs and DSITMS methods in Palmer's labs. These results will be documented in a separate report.

II. METHODS/RESEARCH OPERATIONS

A. List and Description of All Functional Objectives

FO1. Collect instantaneous air samples using GSCs.
FO2. Collect 24-hour time-averaged samples using the SSAS unit.

B. List and Description of All Hardware Items Used

HW1. GSC - Grab sample container capable of collecting an instantaneous air sample.
HW2. SSAS - Solid Sorbent Air Sampler capable of collecting up to 7 time-averaged air samples.

C. Sessions Table

Table 1. Sessions/Functional Objectives Table

mission	session name	FO#	HW#	scheduled day	actual day	scheduled subjects	actual subjects	samples/ parameters	method
Mir 22	collect GSC sample	FO1	HW1	N/A	11-Nov-96	-	-	AA01466	*
Mir 22	collect GSC sample	FO1	HW1	N/A	11-Nov-96	-	-	AA01467	*
Mir 22	collect SSAS sample	FO2	HW2	N/A	N/A	-	-	AA01657	*
Mir 22	collect SSAS sample	FO2	HW2	N/A	N/A	-	-	AA01613	*
Mir 22	collect SSAS sample	FO2	HW2	N/A	N/A	-	-	AA01661	*
Mir 22	collect SSAS sample	FO2	HW2	N/A	N/A	-	-	AA01617	*

Notes:
N/A refers to the fact that this information was not available
***** denotes information on the sampling methods provided below in section D

D. Discussion of Method/Protocol

Specific methods to delineate the steps involved with unstowing the sampling devices, collecting air samples, restowing the sampling devices, and transferring the sampling devices to and from the Shuttle have been documented by the JSC Toxicology Lab. The methods used to collect air samples using these devices are briefly described here. For GSCs, this involves recording the date, time, and location; opening a valve on the GSC to begin collection of the air sample; and then closing the valve once the GSC pressure has reached ambient pressure. For the SSAS unit, this involves recording the date, time, and location, switching the valve on the SSAS unit from a "park" location to one of the 7 sampling tube locations, and turning on the sampling pump to begin collection. After the desired sampling interval is complete (usually ~24 hours), the date and time are again recorded, the sampling pump turned off, and the valve is switched back to the "park" location.

III. RESULTS

A. List of Pre-, In-, and Post-Flight Anomalies

None to report.

B. Completeness/Quality of Data

A number of GSC and SSAS samples were collected on the Mir 22 mission and returned to the JSC Toxicology Lab for analysis. A subset of these samples that included 2 GSC (samples AA01466 and AA01467) and 4 SSAS samples (samples AA01657, AA01613, AA01661, and AA01617) were forwarded to Palmer and Belisle in late July 1997. These samples were analyzed by GC/MS and DSITMS methods. The data quality appears to be excellent and meets the expected performance requirements of each method.

C. Tables, Graphs, Figures Index

Table 1. Sessions/Functional Objectives Table
Table 2. Collection and processing information on Mir 22 samples
Table 3. Results from GC/MS analysis of sample AA01466
Table 4. Results from GC/MS analysis of sample AA01467
Table 5. Results from GC/MS analysis of sample AA01657
Table 6. Results from GC/MS analysis of sample AA01613
Table 7. Results from GC/MS analysis of sample AA01661
Table 8. Results from GC/MS analysis of sample AA01617
Table 9. Summary of GC/MS results on Mir 22 samples
Table 10. Comparison of GC/MS and DSITMS results

IV. DISCUSSION

A. Status of Data Analysis

Analysis of the samples using a modified form of standard EPA GC/MS methods [1] was successful. A copy of GC/MS results from was provided to the JSC Toxicology Lab for comment. DSITMS analyses of these samples were recently completed and the results are reported here for the first time.

B. Preliminary Research Findings

1. Introduction

Two types of samples were collected from Mir station: grab samples using GSCs and time-averaged samples using the SSAS unit. Information describing where and when these samples were collected and how they were processed was provided by the JSC Toxicology Lab and is shown in Table 2.

Table 2. Collection and processing information on Mir 22 samples

JSC ID	GSC S/N	Sample type	Sampling location	Sampling time and date	Sample processing information
AA01466	1032-21037	GSC	base block	15:29, 11-Nov-96	initial pressure 13.70 psia repressurized from 7.90 to 13.82 psia 350 mL canister
AA01467	1040-21045	GSC	Kvant-2	15:32, 11-Nov-96	initial pressure 13.70 psia repressurized from 7.90 to 15.02 psia 350 mL canister
AA01657	21256-0013	SSAS Tube #2	base block	09:42, 16-Oct-96- 11:30, 17-Oct-96 (1548 min)	sample volume 1.07 L initial pressure 14.37 psia 0.100 L humidified air added 500 mL canister
AA01613	21502-0011	SSAS Tube #3	Peroda	16:47, 11-Nov-96- 14:30, 12-Nov-96 (1303 min)	sample volume 0.956 L initial pressure 14.70 psia 0.100 L humidified air added 500 mL canister
AA01661	21499-0013	SSAS Tube #6	N/A	N/A	method blank initial pressure 14.27 psia 0.100 L humidified air added 500 mL canister
AA01617	21487-0011	SSAS Tube #7	N/A	N/A	method blank initial pressure 14.24 psia 0.100 L humidified air added 500 mL canister

2. Experimental

GC/MS: Mir samples and standards were analyzed using a modified form of the EPA TO-14 method [1]. The most salient experimental details on analytical methods employed are summarized here. Detailed information on preparing standards, tuning the GC/MS instrument, and quality assurance/quality control (QA/QC) procedures are beyond the scope of this report and are documented elsewhere [13].

An Entech 7000 concentrator was used to isolate the VOCs from the bulk of the air sample. Samples were pulled out of a stainless steel canister and passed through a cryogenically cooled internal trap. VOCs were desorbed from this internal trap, passed through moisture control and carbon dioxide removal modules, and cryofocused onto the head of the GC column. Injection was achieved by flash heating the head of the column. A Varian Saturn 3 gas chromatograph/ion trap mass spectrometer/data system was used to separate and detect individual VOCs. A 150 m, 0.23 mm ID Petrocol column was used to effect the separation.

A number of QA/QC procedures were implemented for these analyses. Blanks were analyzed to ensure that the sample path through both the Entech unit and GC/MS instrument were not contaminated and background ion counts were acceptable. Canisters used to prepare the working gas standards were cleaned with humidified, purified air and their cleanliness proofed by GC/MS analysis prior to use. Mass spectrometer response was tuned to meet EPA criteria for bromofluorobenzene. Identification of specific VOCs in the Mir samples was achieved via a combination of both a retention time match to previously analyzed standards and library searching of experimental spectra against the NIST library of approximately 60,000 reference mass spectra. A Supelco TO-14 100 per-billion by volume (ppbv) gas standard containing 37 common VOCs was used to prepare a series of working standards. Neat stock solutions for 29 other VOCs were prepared and used to generate an additional series of working standards. Five point calibration

curves were established for each target compound. Internal standards were employed in standards and samples to correct for variations in instrument response. Varian EnviroPro software was employed to automate VOC identification and quantitation. The results were entered into Microsoft Excel spreadsheets and VOC concentrations in the original Mir samples were computed using sample data provided by the JSC Toxicology Lab. Replicate sample analyses were not performed due to limited sample volumes and the desire to perform subsequent DSITMS analyses on the same samples.

DSITMS: Mir samples and standards were analyzed using a variation of a proposed EPA method [14,15]. Again, only the most salient experimental details are summarized here. More detailed information on the preparation of gas standards, tuning the ion trap instrument, and acquiring DSITMS data are beyond the scope of this report and are documented elsewhere [15-21].

The sample introduction system employed for these DSITMS analyses is referred to as the Continuous Air Monitor (CAM). This is a prototype inlet system developed by Scientific Instrument Services based on a design originated by researchers at Oak Ridge National Laboratories [22]. The CAM mixes an air sample into a helium stream, and then passes the combined stream is then passed through an open-split interface prior to introduction into a Teledyne 3DQ Discovery ion trap mass spectrometer. All experiments employed the ion trap in conventional MS scan mode to detect as many VOCs in the samples in a single acquisition. Although separate MS/MS scan functions would permit more selective detection of individual VOCs, the very limited sample volumes and the CAM's high rate of sample precluded their use here. A low mass cutoff of m/z 60 was employed to eject relatively large background ion signals below this value from the trap. A scan range of m/z 60 to 220 was used to detect major VOC ions expected to be present in these samples. The CAM was operated in a continuous mode of operation, where the inlet was challenged with zero-grade air for 60 seconds followed by the sample or a gas standard. The CAM sample consumption coupled with typical sample volumes of 200 mL allowed for approximately 10 seconds of data acquisition for each sample.

QA/QC procedures were implemented insofar as the limited sample volumes and compromises inherent to these DSITMS analyses allowed. Samples were transferred into previously cleaned and proofed Tedlar bags just prior to analysis. Corrected mass spectra for each sample was obtained by subtracting an average background response from an average sample response. As only a few VOCs were present above the approximately 50 ppbv DSITMS detection limit in the samples analyzed, the corrected mass spectra were relatively simple and contained only a few major peaks. VOCs in each sample were tentatively identified by the presence of characteristic ions. Intermediate gas standards were prepared in a static dilution bottles from neat liquids of the pure compound. Working gas standards were prepared in Tedlar bags via dilutions into known volumes with gas-tight syringes. A four-point calibration curve was established for each VOC via working standards prepared and analyzed on the same day as the samples. Teledyne Sequel version 1.0 was used for data analysis. Excel version 5.0 was used to generate calibration curves, compute standard deviations and least squares fits.

3. Results

GC/MS: Results from the GC/MS analyses of samples AA01466, AA01467, AA01657, AA01613, AA01661, and AA01617 ,are shown in Tables 3, 4, 5, 6, 7, and 8, respectively. The "nd" in these tables refers to a VOC that was not detected. Concentrations in this report are given in units of mg/m^3 which are the standard units used by both the JSC Toxicology Lab and EPA for reported toxic air contaminant concentrations. There is a large amount of information in these tables and some discussion of their content is necessary to facilitate their interpretation. A summary of the VOCs identified in these samples, their possible sources, range of concentrations, and 7-day SMACs [23,24] are given in Table 9.

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insert Table 7 here from separate Excel document (filename Mir22-AA01661)

insert Table 8 here from separate Excel document (filename Mir22-AA01617)

Table 9. Summary of GC/MS results on Mir 22 samples

VOC	possible source	concentration (mg/m^3)	7-day SMAC (mg/m^3)
halogenated hydrocarbons:			
dichlorofluoromethane (CFC12)	refrigerant	nd-1.75	490
trichlorofluoromethane (CFC11)	refrigerant	nd-0.43	560
trichlorotrifluoroethane (CFC113)	refrigerant	0.04-0.79	400
dichloromethane (methylene chloride)	solvent	0.07-0.80	50
bromotrifluoromethane (HCFC1301)	refrigerant	nd-0.21	11000
dichlorofluoroethane (CFC22)	refrigerant	nd-0.18	350
1,2-dichloroethane	solvent	nd-0.05	2
1,1,1-trichloroethane	solvent	nd-0.21	160
perfluoro-1,3-dimethylcyclohexane	refrigerant	0.06-11.95	N/A
aromatic hydrocarbons:			
toluene	fuel	0.11-0.34	60
ethylbenzene	fuel	0.01-0.04	130
m-and p-xylene	fuel	0.01-0.09	220
o-xylene	fuel	0.02-0.17	220
aliphatic & cyclic hydrocarbons:			
cyclopentene	?	nd-0.01	170
methyl-cyclopentene	?	nd-0.02	N/A
dimethyl-cyclopentane	?	nd-0.01	N/A
methyl-hexane	?	nd-0.02	N/A
methyl-ethyl-hexane	?	nd-0.01	N/A
methyl-cyclohexane	?	nd-0.02	60
isoprene	plant emission	nd-0.05	560
limonene	plant emission	nd-0.14	560
alpha-terpinene	plant emission	nd-0.02	N/A
oxygenated hydrocarbons:			
2-propanol (isopropyl alcohol)	human metabolite	nd-0.05	150
butanol (butyl alcohol)	human metabolite	nd-0.02	80
2-ethyl-1-hexanol	human metabolite	nd-0.11	N/A
butanal	human metabolite	nd-0.05	120
butenal (crotonaldehyde)	human metabolite	nd-0.05	120
pentanal (valeraldehyde)	human metabolite	nd-0.10	110
hexanal (hexaldehyde)	human metabolite	nd-0.07	N/A
heptanal (heptaldehyde)	human metabolite	nd-0.07	N/A
2-butanone (MEK)	human metabolite	nd-0.42	30
2-heptanone	human metabolite	nd-0.08	N/A
siloxanes & miscellaneous compounds:			
hexamethylcyclotrisiloxane	plastic offgas	nd-1.73	230
octamethylcyclotetrasiloxane	plastic offgas	0.21-0.99	1000
carbon disulfide	?	nd-0.04	16

The target compounds for these analyses included 37 VOCs in the TO-14 standard plus an additional 29 VOCs. This latter set of 29 VOCs encompassed fluorocarbons, siloxanes, terpenes, and oxygenated hydrocarbons that were either identified in samples from prior Mir missions and toxic and/or potential contaminants. Out of the total of 66 target compounds, only 35 VOCs were identified in any one of the six samples, and only 8 VOCs were identified above detection limits in all the samples. Most of these are common air contaminants that can be categorized into specific compound classes shown in Table 9 and are not unexpected for the Mir environment. Although d5-chlorobenzene was also identified in the samples, it was added to the canisters prior to their use by the JSC Toxicology Lab as a surrogate standard and was not actually present in the Mir environment. The presence of siloxanes in the Mir samples may be somewhat questionable. A NASA report suggests that these compounds may have a common system source and hence may not actually be present in the Mir samples [25]. Clearly, further work on the source and stability of siloxanes in canisters is warranted.

The differences in VOC concentrations between the various samples is understandable given their different sampling locations, times, and methods (GSC vs. SSAS). Finally, it should be noted that VOC concentrations were several orders of magnitude below their SMACs, indicating the air samples were fairly clean.

DSITMS: The scope of these analyses was limited due to a number of inherent characteristics of both the samples and the DSITMS methods employed here. The following three points should be noted prior to discussion of DSITMS results. 1) VOC concentrations in the Mir samples were very low to begin with. The highest concentration VOC detected via GC/MS analyses was perfluoro-1,3-dimethyl-cyclohexane at approximately 12 mg/m^3 in sample AA01467. This concentration is equivalent to 730 ppbv. 2) The samples were subjected to two repressurizations (and hence dilutions) subsequent to successive GC/MS analyses at the JSC Toxicology Lab and NASA Ames. Hence, the VOC concentrations in *the samples subjected to DSITMS analyses* were lower than the VOC concentrations in *the original Mir samples*. Following up on GC/MS data in the example given above and factoring in the two dilution factors, the concentration of perfluoro-1,3-dimethyl-cyclohexane in sample AA01467 taken for DSITMS analysis can be approximated. This concentration is roughly 160 ppbv. 3) The detection limit for the DSITMS methods employed here was on the order of 50 ppbv.

Samples AA01657, AA01613, AA01661, and AA01617 were collected using the SSAS. These samples had inherently low VOC concentrations to begin with, and subsequent dilutions resulted in VOC concentrations that were well below the DSITMS detection limit. Hence, it was not surprising that DSITMS data did not indicate any VOCs in these samples. DSITMS data indicated the presence of perfluoro-1,3-dimethyl-cyclohexane in samples AA01466 and AA01467. Mass spectra from DSITMS analysis of perfluoro-1,3-dimethyl-cyclohexane gas standards included the following ions listed in order of decreasing intensity: m/z 69, 181, 231, 131, 281, and 100. Mass spectra from DSITMS analyses of these samples included ions at m/z 69, 181, 131, and 100 (note that ions at m/z 231 and 281 were not detected as the scan range for these acquisitions were m/z 60 to 220). This identification is supported by prior GC/MS results indicating this VOC as the major component in each of these samples. DSITMS data on the same two samples also indicate the presence of sulfur hexafluoride. Inspection of the NIST reference mass spectrum for this compound showed m/z 127 as the base peak. This hypothesis is further supported by GC/MS results on Mir 19 and 21 samples, which also indicated the presence of this VOC [11,12]. The presence of few other ions at reasonable intensities in the DSITMS mass spectra rules out the possibility of this ion being organic in nature. Although this ion is also the base peak for iodine, this VOC is unlikely to be present in appreciable concentrations in the Mir air samples.

A comparison of DSITMS to GC/MS results is given in Table 10. Perfluoro-1,3-dimethyl-cyclohexane concentrations were computed from a DSITMS calibration curve, which plotted m/z 69 intensity versus concentration for 4 different gas standards. GC/MS and DSITMS concentrations for perfluoro-1,3-dimethyl-cyclohexane differ by a factor of two to three. A probable reason for this is that the m/z 69 ion used to

quantify perfluoro-1,3-dimethyl-cyclohexane is also the base peak for perfluoropropane. This latter VOC was identified at relatively high concentrations in GC/MS results from the Mir 19 and 21 missions [11,12]. Although it was not indicated in the GC/MS results reported here, it is possible that it is was not adequately cryotrapped on the Entech system due to its high volatility and/or improper cryotrapping parameters. Sulfur hexafluoride concentrations were estimated using the m/z 127 intensities from the same calibration curve, as sulfur hexafluoride gas standards were not available at the time these samples were analyzed and appropriate response factors were unknown. Sulfur hexafluoride was not detected in GC/MS analyses but was indicated in DSITMS data. This is due to the unavoidable compromise and inherent limitations in the GC/MS method, which was designed for determination of a wide range of VOCs, and hence failed to adequately resolve and detect sulfur hexafluoride.

Table 10. Comparison of GC/MS and DSITMS results

VOC	GC/MS concentration (mg/m^3)	DSITMS concentration (mg/m^3)
sample AA01466:		
perfluoro-1,3-dimethyl-cyclohexane	8.19	22
sulfur hexafluoride	nd	26
sample AA01467:		
perfluoro-1,3-dimethyl-cyclohexane	11.95	22
sulfur hexafluoride	nd	26

C. Conclusions

GC/MS analyses of the Mir 22 samples met the expected performance requirements. Results showed common VOCs normally found in air samples, including halogenated hydrocarbons, aromatic hydrocarbons, aliphatic and cyclic hydrocarbons, oxygenated hydrocarbons, and siloxanes. All of these VOCs were present at concentrations several orders of magnitude below their SMACs. In fact, these results show air quality on board Mir station to be better than most indoor environments, insofar that there were fewer VOCs detected and those that were found were present at relatively low concentrations.

DSITMS analyses of the Mir 22 samples, although limited in scope, were likewise successful and identified the one VOC present at concentrations exceeding the 50 ppbv detection limit. it should be noted that the DSITMS results did not indicate any false positives (identifying a VOC that was not indicated in GC/MS data) or false negatives (not identifying a VOC that was indicated in GC/MS data, assuming the 50 ppbv DSITMS detection limit is taken into account). Furthermore, DSITMS was shown to be capable of monitoring permanent gases as well as VOCs, which is a “blind spot” in GC/MS analyses.

Comparing the two methods, GC/MS is clearly capable of detecting a wide range of VOCs at far lower concentrations than DSITMS In defense of the DSITMS methods, these are still under development and offer a number of potential advantages over GC/MS. Although detection limits are currently several orders of magnitude higher than GC/MS, this is a function of the relative amounts of samples entering the detector, which are on the order of 10-100 mL for GC/MS and 10-100 μL for DSITMS. Recent work using alternate sample introduction systems for DSITMS show promise in reducing detection limits by as much as three orders of magnitude. Moreover, given that all but a few of the target VOCs have SMACs in the ppmv range [24,25], it is apparent that DSITMS techniques employed here are more than adequate for monitoring most VOCs at concentrations well below their SMACs. Finally, the instrumentation for DSITMS is far simpler and would ultimately requires much less space, weight, and power on a space platform.

Future research will focus on developing GC/MS methods to separate and identify nitrogen and sulfur containing VOCs, developing more sensitive DSITMS sample introduction systems (i.e., CAM, membrane

inlet, and glow discharge source). If possible, additional Mir samples will be reserved exclusively for DSITMS analysis to enable the use of MS/MS scan modes for more selective identification of particular VOCs.

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