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180-DAY PRELIMINARY SCIENCE REPORT

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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 Experiment

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. Previous Mission Experience

Palmer and the JSC Toxicology Lab have signed a Memorandum of Understanding (MOU) which documents 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's mission experience includes a set of "practice" samples from the Mir 19 mission, whose analyses enabled the testing of analytical methods and instruments, and the garnering of practical experience on space station air samples. A preliminary report on Mir 19 samples has been generated and forwarded onto the JSC Toxicology Lab for review [7].

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/Functional Objectives Table

Table 1. Sessions/Functional Objectives Table

mission	session			scheduled	actual	scheduled	actual	samples/	
	name	FO#	HW#	day	day	subjects	subjects	parameters	method
Mir 21	collect GSC sample	FO1	HW1	7-Mar-96	7-Mar-96	N/A	N/A	GSC21400	see below
Mir 21	collect GSC sample	FO1	HW1	21-Mar-96	21-Mar-96	N/A	N/A	GSC21403	see below
Mir 21	collect SSAS sample	FO2	HW2	7-Mar-96	7-Mar-96	N/A	N/A	SSAS #2	see below
Mir 21	collect SSAS sample	FO2	HW2	21-Mar-96	21-Mar-96	N/A	N/A	SSAS #3	see below

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

No anomalies were reported on the specific subset of Mir 21 samples analyzed by Palmer.

B. Completeness/Quality of Data

A number of GSC and SSAS samples were collected on the Mir 21 mission and sent to the JSC Toxicology Lab for analysis. A subset of these samples which included 2 GSC and 2 SSAS samples were forwarded to Palmer for analysis.

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IV. DISCUSSION

A. Status of Data Analysis

All samples were analyzed via conventional GC/MS methods [1]. Although the Mir 21 samples were originally intended for analysis in Warren Belisle's laboratories at NASA Ames Research Center, the equipment he planned to use for these analyses was not yet installed and operational at the time at which the samples were received. Hence, the samples were analyzed in Palmer's laboratory at San Francisco State University.

Samples were not analyzed via DSITMS techniques, which are still under development in Palmer's labs. The sample volumes required for these analyses were deemed too large and the techniques too immature for application to Mir samples at this time. As this research proceeds, these methods will be applied to samples from later Mir missions.

B. Preliminary Research Findings

1. Introduction

This report documents the analysis of volatile organic compounds (VOCs) in 4 air samples collected during the Mir 21 mission. Two types of samples were collected from the core module of Mir station: 2 grab samples using GSCs and time-averaged samples using the SSAS unit. The following information on these samples was provided by the JSC Toxicology Lab.

Table 2. Description of Mir 21 samples analyzed

<u>JSC ID</u>	<u>GSC S/N</u>	<u>Sample Type</u>	<u>Sampling Location</u>	<u>Sampling Time & Date</u>	<u>Additional Sample Information</u>
AA01241	GSC21400	GSC	Core Module	15:00, 7-Mar-96	14.20 psi initial pressure repressurized from 6.47 to 18.50 psi
AA01243	GSC21403	GSC	Core Module	14:15, 21-Mar-96	13.70 psi initial pressure repressurized from 5.58 to 17.09 psi
SSAS tube # 2	GSC21256	SSAS	Core Module	15:00, 7-Mar-96 to 15:00, 8-Mar-96	1.329 L sample volume 14.56 psi original pressure 0.100 L humidified air added
SSAS tube # 3	GSC 21255	SSAS	Core Module	14:00, 21-Mar-96 to 14:00, 22-Mar-96	1.329 L sample volume 14.52 psi original pressure 0.100 L humidified air added

2. Experimental

Mir samples and standards were analyzed using a modified form of the EPA TO-14 method [1]. Commercial Tekmar AeroCAN, AeroTRAP, and cryofocusing equipment (Cincinnati, OH) was employed to concentrate and isolate the VOCs from the bulk of the air sample as follows. 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 a moisture control module, and cryofocused onto the head of the GC column. Injection was achieved by flash heating the head of the column. The various parameters (i.e., temperatures, flowrates, and timing) used within the various operational stages (i.e., sample transfer, cryotrap cooldown, desorb, cryofocus inject, etc.) of the Tekmar equipment are too numerous for the scope of this report and hence only the most critical parameters are documented in Table 3.

Table 3. Selected Tekmar parameters used for analysis of Mir 21 samples

leak checking of system prior to analysis
standby temperatures of 200 °C (transfer line, valve oven, etc.)
sample volume of 100 mL at a flow of 50 mL/min
standard volume of 50 mL at a flow of 50 mL/min
cryotrapping temperature of -165 °C
desorb temperature of 200 °C for 5 min

A Finnigan Magnum gas chromatograph/mass spectrometer/data system (San Jose, CA) was used to separate and detect individual VOCs. Selected GC parameters are shown in Table 4. The mass spectrometer was tuned prior to data acquisition using Finnigan's autotune procedure. This automatically adjusts filament emission current, multiplier gain, and AGC target value, and performs a mass calibration. Mass spectrometer acquisition parameters are shown in Table 5.

Table 4. Selected GC parameters used for analysis of Mir 21 samples

column:	25 m, 0.25 mm ID J&W DB-5 column (Folsom, CA)
He flow:	1 mL/min
T program:	starting at 50 °C for 10 min ramping to 250 °C at 5°C/min holding at 250 °C for 10 min total program time of 60 min
GC transfer line:	250 °C

Table 5. Selected MS parameters used for analysis of Mir 21 samples

ion trap manifold temp:	220 °C
emission current:	100 uA
multiplier high voltage:	2400 V
AGC target valve:	65000
ionization mode:	70 eV EI
mass range:	50 to 400 daltons
scan rate:	1 scan/sec

Identification of specific VOCs in the Mir samples was achieved via a combination of both retention time match to a known standard and library searching of mass spectral data. Retention time information for a number of VOCs was obtained from the analysis of several gas standards. Library searching was accomplished by matching experimental mass spectra against the NIST library of approximately 60,000 reference mass spectra. It should be noted that the lack of standards for each tentatively identified VOC and the inability of library searching to provide conclusive information resulted in identifications that were unavoidably ambiguous. In these cases, manual interpretation of mass spectral data was used to provide a tentative identification. Despite these problems, a high degree of confidence can be placed in all but a few of the identifications.

The most accurate method for determining target compound concentrations utilizes isotopically-labeled internal standards. This becomes rather expensive, time-consuming, as well as problematic when the large number of VOCs of interest in space station air samples are considered. Usually, a less accurate method involving multipoint calibration curves for each target compound of interest and a surrogate to correct for short-term drift in instrument response is used. While this method is currently employed by the JSC Toxicology Lab for analysis of Mir air samples and was intended for use by Palmer for the Mir 21 samples, the associated gas standards and surrogate standards were not available at the time these samples were analyzed. Hence, an alternate method involving ratioing VOC intensities in the Mir samples to those in a TO-14 gas standard of known concentration from Scott Specialty Gases (Fremont, CA). It should be noted that this compromise is based on valid assumptions and should result in only slightly less accurate concentrations than the desired methods.

Calculating concentrations of individual VOCs in the original Mir GSC and SSAS samples is rather complex and takes into account a number of correction factors. First, the concentration of each VOC in the Mir sample taken for

analysis was computed by calculating an intensity ratio representing the quan ion intensity of the specific VOC in the sample divided by quan ion intensity of that same VOC in a gas standard of known concentration. Where a gas standard for a specific VOC was not available (which was the case for many of the compounds identified in the Mir sample), the intensity ratio was calculated relative to toluene, except for d5 chlorobenzene for which the response of chlorobenzene was used. This intensity ratio was then multiplied by the concentration of the VOC in the gas standard. Correction factors for the intensity of the VOC quan ion relative to that of toluene (i.e., t values) were not available for any of the VOCs [8] and hence were not used in these calculations. The correction factor for the volume was 0.5 as the volumes of samples and standards were 100 and 50 mL, respectively. The results of this calculation is the concentration of the specific VOC *in the Mir sample taken for analysis*, which is not the same as the Mir sample for reasons which are outlined below.

Calculation of VOC concentration in Mir sample taken for analysis:

$$\text{ppmv of VOC} = (\text{intensity ratio}) * (\text{ppmv of standard}) * (\text{correction factor for intensity}) * (\text{correction factor for volume})$$

where:

$$\text{intensity ratio} = (\text{response of quan ion in sample}) / (\text{response of quan ion in standard})$$

$$\text{correction factor for intensity} = (\text{t value for quan ion in sample}) / (\text{t value for quan ion in standard})$$

$$\text{correction factor for volume} = (\text{volume of standard}) / (\text{volume of sample})$$

Next, the concentration of VOCs were converted from ppmv to mg/m³ [9], which are the standard units used for reporting VOC concentrations in space environments. These units are usually expressed at standard temperature and pressure and hence the appropriate correction factors must be applied as shown below.

Conversion of units of concentration from ppmv to mg/m³:

$$\text{mg/m}^3 = (\text{ppmv}/10^6) * (\text{moles/L}) * (\text{mw in g/mole}) * (10^3 \text{ mg/g}) * (10^3 \text{ L} / 1 \text{ m}^3)$$

given universal gas law of PV = nRT, moles/L = n/V = P/RT:

$$\text{mg/m}^3 = (\text{ppmv}/10^6) * (P/RT) * (\text{mw in g/mole}) * (10^3 \text{ mg/g}) * (10^3 \text{ L} / 1 \text{ m}^3)$$

given R = 0.0821 L atm/mole K and assuming P = 1.00 atm and T = 298 K, we can simplify this equation to get:

$$\text{mg/m}^3 = (\text{ppmv}) * (\text{mw in g/mole}) * (0.0409)$$

For GSC samples, a number of correction factors must be applied as outlined below. A correction factor for temperature would normally represent the ratio of standard temperature (298 K or 25 °C) to the temperature onboard Mir. In this case, temperatures onboard Mir were not known, and since this correction factor would be fairly close to unity, it was not used. The correction factor for pressure represents the ratio of Mir Station pressure, which in this case is represented by the ambient pressure of the GSC, to standard pressure (1 atm or 14.696 psi). A dilution factor was applied to take into account the fact that these samples were analyzed by the Toxicology Lab at JSC and then diluted to a higher pressure.

Calculation of concentration in original GSC sample:

$$\text{mg/m}^3 = (\text{mg/m}^3 \text{ of sample analyzed}) * (\text{T factor}) * (\text{P factor}) * (\text{dilution factor})$$

where:

$$\text{T factor} = 298 \text{ K} / (\text{T onboard Mir in K})$$

$$\text{P factor} = 14.696 \text{ psia} / (\text{P onboard Mir in psia})$$

$$\text{dilution factor} = (\text{canister P after dilution}) / (\text{canister P before dilution})$$

For SSAS samples, the calculation of the concentration of a VOC in the sample analyzed represents the mg of VOC in a 1 m³ sample volume. This number must be first multiplied by the volume analyzed to get the mg of VOC in that sample. Next, this number must be divided by the actual volume sampled to compute the original concentration of the VOC on Mir. A volume correction factor is applied in this step to reflect the factor that the original Mir sample was desorbed

from a SSAS tube into a GSC which already contained 100 mL of humidified air. A pressure correction factor is also applied to reflect the ration of the final canister pressure after desorption of VOCs from the SSAS tube into the GSC was complete to standard pressure.

Calculation of concentration in original SSAS sample:

mg of VOC in sample analyzed = (mg/m³ of VOC in sample analyzed) * (corrected vol of canister in m³)

where:

corrected vol of canister in m³ = 14.696 psi * (vol of canister in m³ at STP) / (canister P after desorption into GSC)

now calculate concentration of in original SSAS sample:

mg/m³ = (mg of VOC in sample analyzed / m³ of sample collected) * (T factor) * (P factor)

where:

T factor = 298 K / T onboard Mir in K

P factor = 14.696 psia / (P onboard Mir in psia)

3. Results

Replicate analyses of the gas standards and samples were performed as outlined in Table 6.

Table 6. Sequence of analyses of Mir samples and standards

Date	JSC Sample ID	SFSU data file	Description
7/29/96	AA01241	AA01241A	100 mL of Mir Sample
7/29/96	AA01241	AA01241B	100 mL of Mir Sample
7/29/96	AA01243	AA01243A	100 mL of Mir Sample
7/29/96	AA01243	AA01243B	100 mL of Mir Sample
7/29/96	SSAS Tube #3	SSMIR21A	100 mL of Mir Sample
7/29/96	SSAS Tube #3	SSMIR21B	100 mL of Mir Sample
7/29/96	SSAS Tube #2	SSMIR21D	100 mL of Mir Sample
7/29/96	N/A	TO14STD2	50 mL of TO-14 gas standard
7/30/96	N/A	TO14STD3	50 mL of TO-14 gas standard
7/30/96	N/A	TO14STD4	50 mL of TO-14 gas standard
7/30/96	SSAS Tube #2	SSMIR21E	100 mL of Mir Sample
7/30/96	SSAS Tube #2	SSMIR21F	100 mL of Mir Sample

A malfunction of the Tekmar equipment appeared during the acquisition of the SSMIR21B data file and continued through the SSMIR21D, TO14STD2, TO14STD3 runs. Quan ion intensities during these runs were several orders of magnitude lower than expected. The cause was most likely a blockage of the sample lines in the Tekmar unit which could have been caused by high water and/or carbon dioxide content in the samples. The resulting data files were judged unreliable and discarded from further interpretation. This problem can be avoided in future Mir sample analyses by reducing the volume of sample analyzed and using an Entech air concentrator which includes an improved water/carbon dioxide removal subsystem.

Quantitation was performed for the one intact data file for the gas standard (TO14STD4). Results from this analysis and relevant information on the VOCs in this gas standards are shown in Table 7. Replicate analyses of the two GSC samples were performed (data files AA01241A and AA01241B for JSC sample ID AA01241, AA01243A and AA01243B for JSC sample ID AA01243). Qualitative and quantitative results from these samples are shown in Table 8. Replicate analyses of the two SSAS samples were performed (data files SSMIR21A for SSAS tube # 3, and SSMIR21E and SSMIR21F for SSAS tube #2). Qualitative and quantitative results from these samples are shown in Table 9.

There is a large amount of information in these tables and some discussion of their content is necessary to facilitate their interpretation. The most useful information for the purposes of the astronauts, toxicologists, and program managers is presented in the second and third columns, which list the IUPAC and common name for the specific VOC tentatively identified, and the last column, which lists the concentration of that VOC in the Mir sample. The other columns detail information which is more likely to be of interest to the JSC Toxicology Lab for intercomparison and evaluation of results. Only a minor commentary on these results is presented here for discussion purposes. More detailed evaluation of

these results will be presented in the 1-year report, which will include critical feedback from the JSC Toxicology Lab and intercomparison of results.

Retention times between replicate runs generally agreed within a few seconds with the exception of some early eluting VOCs. Reproducibilities of quan ion counts between runs were generally less than 20% RSD (relative standard deviation), which is acceptable for this type of analysis and surprisingly low given the fact that an internal standard was not employed. The exceptions to this were some of the early eluting compounds which were difficult to separate chromatographically.

The compounds identified represent fairly common VOCs representing halocarbons, aromatic hydrocarbons, and siloxanes. Some of the identifications are tentative at this point due to the lack of pure standards and are hence denoted by question mark in the first column in Tables 8 and 9. Note that many of the CFCs identified in the GSC samples were not identified in the SSAS samples. This is due to the particular sorbent agent used in the SSAS unit, which is intended to trap relatively higher molecular weight compounds in the C6-C12 range. Likewise, many of the hydrocarbons identified in the SSAS samples were not identified in the GSC samples. This may be due to the fact that larger sample volumes associated with the SSAS unit provided the lower detection limits required to identify these VOCs. While space maximum allowable concentrations (SMACs) were not available for the identified compounds [10], most SMACs fall in the 0.1 to 1000 mg/m³ range. As nearly all the VOCs were present at concentrations much less than 1 mg/m³, these concentrations seem to be well below the SMACs.

C. Conclusions

The analyses of these first set of Mir samples provided valuable experience as well as information on the types and concentrations of VOCs in the Mir environment. Most of the compounds in the Mir samples identified by library searching and manual interpretation common air contaminants (i.e., halocarbons, siloxanes, and aromatic compounds). The calculated concentrations were lower than SMACs where available.

There were a number of known limitations to the methods employed. The Tekmar equipment was not functioning properly and this prevented replicate analyses of some of the samples and standards. The chromatographic separation was hindered by the lack of a high resolution column (greater than 25 m in length) and a cryocooling capability on the GC. Gas standards required for retention time confirmation and quantitation of each tentatively identified compounds were not available at the time these analyses were performed and hence indirect quantitation was employed in some cases. Analysis of the Mir 21 samples enabled the identification and correction of these problems prior to analysis of samples from future Mir missions. It should be noted that Warren Belisle's laboratories possesses a Entech thermal desorption system (Encino, CA) which has several advantages over the Tekmar modules used in these analyses, including inlets for samples, standards, and surrogates and a carbon dioxide management system. The GC will include both a cryocooling capability to effect improved separation of low boiling compounds and a 150 m Petrocol column for better overall separation efficiency. Calibration curves will be established for target compounds using a combination of commercial and custom gas standards. Surrogates will be used for daily quantitation. This equipment has been already been successfully employed to the analysis of Mir 22 samples.

Mir 21 samples were not analyzed via DSITMS techniques, which are still under development in Palmer's labs. Recent results indicated detection limits on the order of 50 ppbv for several chlorofluorocarbons using MS, selected ion monitoring, and MS/MS modes [11]. Current work focusing on monoterpenes indicate that although the various isomers cannot be differentiated from one another via MS/MS, they can be selectively monitoring as a compound class at detection limits on the order of 50 ppbv [12,13]. Additional research, development, and testing of new MS methods for various VOCs and air sampling systems will proceed over the course of this contract. Application of these methods to VOCs in Mir samples will be performed at a later date.

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