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1-YEAR FINAL RESULTS REPORT

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"Analysis of Volatile Organic Compounds on Mir Station"

Submitted by:
Peter T. Palmer, Ph.D., Principal Investigator
Department of Chemistry & Biochemistry
San Francisco State University
San Francisco, CA 94132-1722
Phone: (415) 338-7717
Email: palmerp@lewis.sfsu.edu

Warren Belisle, Co-Investigator
Analytical Chemistry Laboratory
Lockheed-Martin Technical Services
NASA Ames Research Center, MS 239-11
Moffett Field, CA 94035-1000
Phone: (415) 604-5252
Email: wbelisle@mail.arc.nasa.gov

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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 were collected onto cartridges using the US Solid Sorbent Air Samples (SSAS). Grab samples were collected using US Grab Sample Containers (GSC). Samples were 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 were employed to ensure reliable, high quality data. Analyses were 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) [2]. Significant effort has been 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 have provided detailed information on the types and concentrations of VOCs in the Mir environment. Moreover, the demonstration of new technology and comparison against proven methods has yielded 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 fully 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) 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 [3-7]. 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. Note that Palmer prepared a final report on Mir 19 samples and forwarded a copy on to the JSC Toxicology Lab [8].

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 Table

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

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.

C. Tables, Graphs, Figures Index

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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. Final 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 JSC Toxicology Lab provided the following information on these samples.

Table 2. Description of Mir 21 samples analyzed

JSC ID	GSC S/N	Sample Type	Sampling Location	Sampling Time & Date	Additional Sample Information
AA01241	GSC21400	GSC	Core Module	15:00, 7-Mar-96	14.20 psia initial pressure repressurized from 6.47 to 18.50 psia
AA01243	GSC21403	GSC	Core Module	14:15, 21-Mar-96	13.70 psia initial pressure repressurized from 5.58 to 17.09

					psia
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 psia original pressure 0.100 L humidified air added
SSAS tube # 3	GSC21255	SSAS	Core Module	14:00, 21-Mar-96 to 14:00, 22-Mar-96	1.329 L sample volume 14.52 psia 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 autosetup 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 matches 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 is 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 Mir GSC and SSAS samples is complex. It should be noted that these two different types of samples require different sets of equations to compute VOC concentrations. It is important to distinguish between *the Mir sample taken for analysis* and *the original Mir sample*, as the GSCs were first analyzed as is by the JSC Toxicology Lab and then repressurized (diluted) to permit the additional analyses here. The details of these calculations are outlined below.

VOC concentrations were calculated for both GSC and SSAS samples using a proportional relationship between intensities and concentrations for samples and standards and additional correction factors. The details of this calculation are shown below.

given a proportional relationship between intensity and concentration:

$$\frac{\text{(ppmv of VOC in sample)}}{\text{(ppm of VOC in standard)}} = \frac{\text{(intensity of quan ion for VOC in sample)}}{\text{(intensity of quan ion for VOC in standard)}}$$

we can compute concentration of VOC in sample analyzed:

*ppmv of VOC in sample analyzed = (intensity ratio) * (correction factor for intensity) * (correction factor for volume)*

where:

intensity ratio = (intensity of quan ion in sample) / (intensity of quan ion in standard)
correction factor for intensity = (t value for quan ion in sample) / (t value for quan ion in standard)
correction factor for volume = (volume of standard) / (volume of sample)

The intensity ratio was computed by dividing the quan ion intensity of a VOC in the sample by the quan ion intensity of the 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 a number of VOCs identified in the Mir samples), this intensity ratio was calculated relative to m/z 92 from toluene (an exception to this was d5-chlorobenzene, for which the response of chlorobenzene was used). Correction factors for the intensity of the VOC quan ion relative to that of toluene (i.e., t values [9]) were not available for any of the VOCs identified in the Mir samples and hence were not employed in these calculations. The correction factor for the volumes was 0.5 as the volumes of standards and samples were 50 and 100 mL, respectively.

Next, the concentrations of VOCs were converted from ppmv to mg/m^3 which are the standard units used for reporting VOC concentrations in space environments. The details of this conversion are shown below.

given standard equation for converting from units of ppmv to mg/m^3 [10]:

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

using the universal gas law:

$$PV = nRT$$

$$n/V = moles/L = P/RT$$

and given values for R, T, and P:

$$R = 0.0821\ L\ atm/mole\ K$$

$$P = \text{pressure of Mir sample in atm} = (P \text{ of sample in psia}) * (1\ atm/14.696\ psia)$$

$$T = \text{temperature of Mir sample} = 298\ K \text{ (assumption)}$$

we can combine terms and simplify the equation to get:

$$mg/m^3 \text{ of sample analyzed} = (ppmv) * (P \text{ of sample in psia}/360) * (mw \text{ in } g/mole)$$

Unlike units of ppmv, units of mg/m^3 are dependent on the pressure and temperature of the air sample and hence must reflect the pressure of the Mir sample and temperature of the Mir environment. Pressures of GSC and SSAS samples were measured by the JSC Toxicology Lab and are reported in Table 2. Although the temperature of the Mir environment was not recorded at the time the samples were collected, it is a fairly safe assumption that the temperature onboard Mir was approximately 298 K. Moreover, since this temperature should rarely vary by more than 5 degrees Kelvin onboard Mir (representing a relative error of 5/298 or 1.7%) the dependence of concentration on temperature is insignificant compared to other sources of error in computing VOC concentrations.

For GSC samples, an additional dilution factor must be applied as shown below.

defining the dilution factor for GSC samples as:

$$\text{dilution factor} = (\text{psia of sample after repressurization}) / (\text{psia of sample before repressurization})$$

finally, we can compute concentration of the VOC in the original GSC sample:

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

This reflects the fact that the samples were first drawn off the GSC by the JSC Toxicology Lab for analysis and then repressurized to permit these analyses.

For SSAS samples, the calculation must take into account the volume of the stainless steel canister the sample was desorbed into and the volume of sample collected.

first we must compute mg of VOC in sample analyzed:

$$mg \text{ of VOC in sample } a = (mg/m^3 \text{ of VOC in sample analyzed}) * (\text{corrected vol of canister in } m^3)$$

where:

*corrected vol of canister in m^3 = (vol of canister in m^3) * (14.696 psia) / (canister P in psia after desorption into GSC)*

finally, we can compute concentration of the VOC in the original SSAS sample:

mg/m^3 of original sample = (mg of VOC in sample analyzed) / (m^3 of sample collected)

The concentration of a VOC in the sample analyzed represents the mg of VOC normalized to a 1 m3 sample volume. This number must be first multiplied by the corrected volume of the stainless steel canister which the SSAS sample was desorbed into to obtain the mg of VOC in the sample analyzed. This corrected volume represents a measurement of volume of the canister performed by the JSC toxicology Lab corrected for the actual pressure of the canister. Finally, the mg of VOC in the sample analyzed must be divided by the actual volume sampled to obtain the original concentration of the VOC in the sample collected on Mir.

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, and 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 be attributed to the 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. As a result of this malfunction, quantitation was performed using the one remaining trustworthy data file for the gas standard (TO14STD4). Results from this analysis and relevant information on the VOCs in this gas standard 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.

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 that were difficult to separate chromatographically.

There is a large amount of information in these tables and some discussion of their content is necessary to facilitate their interpretation by nonspecialists. 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 that is more likely to be of interest to the JSC Toxicology Lab for intercomparison and evaluation of results. The compounds identified in the samples represent fairly common VOCs plus a few surprises. Sulfur hexafluoride is a relatively inert, nontoxic compound whose presence may be attributed to its use in metabolic experiments on board Mir. Perfluoropropane and perfluorodimethylcyclohexane were among the highest concentration contaminants identified in the samples. These are also inert and nontoxic compounds whose presence may have been due to their use as refrigerants in Mir subsystems. CFC12 and CFC11 are common coolants that may have been brought on board Mir from Earth through unavoidable contamination of the Shuttle during docking procedures. Likewise, benzene, toluene, ethylbenzene, xylene isomers, and various aromatic hydrocarbons are ubiquitous air contaminants that may have been transferred to Mir from Earth via the Shuttle. Isoprene and limonene can be attributed to plant experiments on Mir. D5-fluorobenzene, D5-chlorobenzene, and 1-bromo-2-fluorobenzene are surrogate standards that were dosed into the GSC and SSAS units prior to sampling and were not actually present in the Mir samples. Siloxane polymers are used in scotch tape, lubricants, and other common materials. Note that scotch tape was used to label the Mir samples. It is possible that trace levels of these polymers contaminated the sample inlets during handling procedures and subsequent GC/MS analyses resulted in the breakdown of these polymers to produce the hexamethylcyclotrisiloxane and octamethylcyclotetrasiloxane peaks identified in the Mir samples. It should be noted that analyses of blanks and standards did not yield similar levels of these compounds. Further work will be performed to test this hypothesis. Some of the identifications were tentative due to the lack of pure standards to provide retention time confirmation and reference mass spectra. Such tentative identifications are denoted by question mark in the first column in Tables 8 and 9. While space maximum allowable concentrations (SMACs) were not available for all VOCs identified in the samples, most SMACs fall in the 0.1 to 1000 mg/m^3 range [11]. As nearly all the VOCs were present at concentrations far less than 1 mg/m^3, the VOC concentrations in the Mir samples are all well below the SMACs. Caveats should be noted in intercomparing results between GSC and SSAS samples. The particular sorbent agent used in the SSAS is intended to trap higher molecular weight compounds in the C6-C12 range. Hence, SSAS results do not show many of the CFCs identified in the GSC samples. Sample volumes are inherently larger in SSAS samples. Hence, more VOCs were identified in the SSAS compared to the GSC samples.

C. Conclusions

GC/MS analyses of Mir 21 samples provided detailed information on the types and concentrations of VOCs on board Mir. Most of the VOCs represent common air contaminants (i.e., halocarbons, siloxanes, and aromatic compounds). Concentrations were lower than SMACs where available. DSITMS analyses of these samples were not performed due to limited sample volumes. Research and development of DSITMS methods will continue over the course of this investigation. Recent results show detection limits on the order of 50 ppbv for several chlorofluorocarbons using MS, selected ion monitoring, and MS/MS modes [12]. MS mode can be used to survey the types and approximate concentrations of VOCs in air samples. MS/MS mode is used to confirm the presence of specific VOCs and provide more accurate concentration data. It is important to understand that MS/MS is not a panacea. Studies of monoterpenes indicate that the various isomers cannot be differentiated from one another via MS/MS [13]. However, they can be selectively monitored as a compound class at detection limits on the order of 50 ppbv [14]. Moreover, their chemistry

and toxicology is similar enough such that differentiation of these isomers may be unnecessary. Finally, it should be noted that application of DSITMS methods to VOCs in Mir 22 samples is in progress and preliminary results compare well with proven GC/MS methods.

D. Investigation Results

The GC/MS methods employed in these analyses, which were based on the use of an ion trap mass spectrometer, provided more than adequate sensitivity to determine VOCs at levels well below their SMACs. The results of the GC/MS analyses have provided information on the types and concentrations of VOCs on Mir. Results have been found to agree with those obtained with the JSC Toxicology Lab. The air quality on board Mir space station was found to be nominal, given the Mir 21 sample set analyzed, the specific VOCs that were the focus of these analyses, and available SMACs. Additionally, it can be stated that VOC concentrations on board Mir were found to be lower than most typical indoor environments on Earth.

E. Investigation Applications

The GC/MS methods used in this work are commonly employed for the analysis of VOCs in air for a variety of applications. The use of an ion trap mass spectrometer in these methods provides greater sensitivity and hence requires smaller sample volumes for analysis. The new DSITMS methods are appropriate for a host of applications that require in-situ, on-line, real-time air quality data. These include process control, fence-post monitoring, stack monitoring, engine exhaust analysis, and human breath analysis.

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