# 4.1

# Environmental Monitoring
# Air Quality

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**SUMMARY**

Air pollutants were quantified during the Phase II (30 day), Phase IIa (60 day), and Phase III (90 day) tests. Measurements from the Phase II test demonstrated a generally stable and safe atmosphere; however, measurements of ammonia and formaldehyde were incomplete. Near day 10 a large amount of methane entered the atmosphere and Freon® 113 was unusually high most of the time. There were periodic "bursts" of ethanol and isopropanol imposed on a steady state level of methanol. The Phase IIa test, which was the first opportunity to measure formaldehyde, was plagued with excess formaldehyde offgassing from various materials in the test chamber. This led to mucosal irritation in one crewmember. Methanol was unusually high, and at one point carbon monoxide had accumulated nearly to its long-term spacecraft maximum allowable concentration (SMAC). In contrast to the Phase II test where an accidental release of methane occurred, methane accumulated steadily throughout the Phase IIa test. Ammonia levels in the Phase IIa test quickly reached a low, steady-state concentration. Except for formaldehyde, all contaminants met standards for acceptable air quality. The Phase III test demonstrated much improved control of formaldehyde even though it exceeded its long-term SMAC late in the test. Ammonia accumulated steadily during the 90 days, reaching approximately 1/8 of its long-term SMAC. During the final days of the test, the air was characterized by rather rapid rises in irritant compounds and methylcyclosiloxanes. The trace contaminant control system (TCCS) suffered degraded performance during this time, and this is the likely cause of the increases in concentrations. Even though air quality standards were exceeded for irritants late in the test, there were no reports from the crew that the air was causing symptoms.

*Introduction*

The pollutants present in the atmosphere of a sealed environment represent the summation of many interacting dynamic processes. Those processes can be roughly separated into pollutant sources and pollutant sinks. This simple division, however, masks the complexity inherent in the behavior of each of the sources and sinks. Some examples will illustrate this point.

Air revitalization systems are necessarily thought of as sinks for air pollutants; however, there are examples where such systems have been the source of serious spacecraft pollution, or have converted relatively non-toxic pollutants to hazardous pollutants. Humans are generally regarded as pollutant sources; however, inhaled air is "scrubbed" of many pollutants by the human respiratory system before being exhaled into the vehicle atmosphere. Materials can be the source of offgassing of trace contaminants released from their molecular structures; on-the-other-hand, materials can provide surfaces for the condensation and absorption of less volatile air pollutants. The task of understanding and controlling the sources and utilizing the sinks to produce a healthy, respirable atmosphere in a sealed environment is not a simple one. A summary of circumstances that have lead to potentially unhealthy levels of air pollution during ground-based or on-orbit operations are given in the introductory subsections below with a perspective on how they relate to the Advanced Human Life Support and Enclosed System Study.

***Materials Offgassing as a Source of Pollution***

All polymeric materials release volatile substances that have been trapped in the polymeric matrix or can be formed as a result of slow decomposition of the material. All non-metallic materials are screened for offgassing rates before being accepted for use inside space vehicles and modules. In addition, the aggregate of offgassing produced in a module is estimated from the sum of offgassing from all components (in Spacelabs) or is tested after the module has been configured for flight (Spacehab and ISS modules). If uncured materials are present, this can produce a dramatic effect on the rate of offgassing into the module's atmosphere. For example, an initial test of the Node 1 module for the ISS gave an offgassing rate of 0.3 T units/day; however, a subsequent test conducted after further curing of adhesives used in the module gave a rate of only 0.02 T units/day. The major components contributing to Node 1 offgassing were methanol and propenal. As we will show later in this chapter, careful attention to materials offgassing can preclude serious problems with air pollution, even in ground-based tests such as the Advanced Human Life Support and Enclosed System Study.

***Systems Leaks as a Source of Air Pollution***

Chemicals are an integral part of many systems that comprise sealed environments, especially in heat-exchange loops. Perhaps the most notorious system leak occurred during the NASA/Mir Program when the Mir heat-exchange loops repeatedly leaked an aqueous solution of ethylene glycol. At times the magnitude of the leak was sufficient to elicit symptoms of respiratory irritation in crewmembers. Ethylene glycol condenses on cool surfaces and does not readily evaporate, hence, its spread throughout the station took place on a time-scale of weeks to months. Leaks of Freon® from refrigerator coolant loops have also been observed during space operations aboard Mir; however, most Freon® is very low in toxicity and has relatively high exposure limits. The Closed Environment Chamber used chilled water from facilities supplies for thermal control; therefore, the risk of systems leaks involving potentially toxic compounds was much less than was experienced on the ageing Mir space station.

### *Experiment and Payload Leaks Cause Pollution*

Experiments and payloads generally contain smaller volumes of chemicals than systems; however, some of the chemicals are highly toxic. Certain experiments use strong bases, which can cause permanent eye damage if they were to escape containment, and others use strong fixatives, which can cause severe eye and upper airway irritation. For example, paraformaldehyde fixative used in the Fundamental Biology Investigation-1 leaked past several containment barriers during the Mir-18 flight, but caused no apparent effect on crew health. The cause of the leak was failure to adequately control the heat-sealing process used for the containment bags. Other, less serious leaks have been observed from Shuttle payload experiments. The experiments conducted during the Closed Environment Living Study generally did not involve toxic chemicals that could escape into the atmosphere; however, an "experiment" conducted near the end of the 90-day test did contribute substantially to air pollution. Addition of food processing activity and waste disposal processes will add new risks to air quality.

### *Accumulation of Human Metabolites*

Carbon dioxide is the major anthropogenic pollutant present in sealed environments. A major subsystem of the air revitalization system of space vehicles is dedicated to removal of this single compound. Failure to control this pollutant can quickly lead to physiological effects on the crew. To improve resource utilization, regenerable carbon dioxide removal systems have been developed; however, the sophistication of these systems can leave them more vulnerable to failure than the traditional, non-regenerable lithium hydroxide-based filtering systems. Periodically, levels of carbon dioxide spike up on the Shuttle if the lithium hydroxide filters are not changed on schedule. At times on Mir the level of carbon dioxide slightly exceeded the U.S. standard of 5.3 mmHg. There were no known effects on crew health. Since different types of carbon dioxide removal systems and different modes of operation of the systems were to be used in the Closed Environment Living Experiment, we expected that there might be some excursions in carbon dioxide concentrations.

### *Utility Chemicals Causing Air Pollution*

Utility chemicals include such diverse items as hardware cleaners, degreasers, glues, personal hygiene materials, medications, and anti-fogging solutions. Problems with such chemicals in the air are rare; however, water-soluble compounds such as alcohols will be removed from the air through the humidity condensate and can end up polluting the water if the humidity condensate is being recovered for purification. For this reason, the use of alcohol-based hand cleaners and alcohol-containing hygiene wipes are strictly controlled on the ISS, but do not need strict control on the Shuttle where humidity condensate is not recovered. Volatile components of utility chemical formulations tend to appear periodically in air samples over a broad range of concentrations. Several major pollutants (e.g. 2-propanol) in the Closed Environment Living Chamber atmosphere exhibited this characteristic.

### *Propellant Entry as a Source of Air Pollution*

Perhaps the most toxic air pollution event in human space flight experience occurred as a result of propellant entering the habitable volume of the vehicle. At the conclusion of the Apollo-Soyuz Program in July 1975, the descending Apollo capsule was equilibrating its low internal pressure with the increasing, outside, atmospheric pressure at the same time thrusters were firing. This resulted in nitrogen tetroxide being pulled into the capsule causing illness and even unconsciousness in the crew. Modern vehicles are designed so that this cannot happen; however, there is a small risk that propellants could lodge on a crewmember's extra vehicular activity (EVA) suit and be brought into the habitable volume through the airlock. Propellant entry will, of course, not be an issue for the ground-based Closed Environment Living Experiment.

### *Combustion as a Source of Air Pollution*

The highest environmental health risk in modern space vehicles results from the possibility that a fire could occur inside the cabin. Aboard the Shuttle there have been experiences involving wiring shorts, pyrolysis of electronic components, and motor burn-out that have resulted in concern about toxic combustion products in the atmosphere. Perhaps the worst was the production of formaldehyde from Delrin® polymer that burned as a result of a seriously overheated motor in the refrigerator-freezer on STS-40. Aboard Mir there were at least two major pollution events resulting from fire or pyrolysis of materials. The solid fuel oxygen generator caused a spectacular fire that nearly resulted in abandonment of the Mir space station, and the "BMP" trace contaminant removal system produced large amounts of carbon monoxide when it overheated, apparently due to improper operation. As expected, wiring fires and other combustion events proved to be a very small risk during the study; however, incineration of waste material during the Phase III test demonstrated that high-temperature operations pose significant air quality risks.

### *Microbiological Metabolites as Air Pollutants*

Microbes pose a threat to crew health not only from their ability to cause infectious disease but also because they can produce noxious air pollutants. The best example of this occurred during STS-55 when urine and other waste materials were being put in the contingency waste container and disposed of by squeezing the container contents into space. The crew reported that the odors generated by doing this were unbearable. Air samples and subsequent ground-based testing revealed that microbes had metabolized the contents into methyl sulfides, which penetrated the walls of the bag and created a noxious odor. Waste management is a major concern for air quality management in long-term space flight and in simulations such as the LMLSTP.

## MATERIALS AND METHODS

*Volatile Organic Compounds*

Air samples were acquired periodically in 500 ml, passivated canisters that had been evacuated, proofed for cleanliness, and spiked with 3 surrogate standards (C13-acetone, fluorobenzene-D5, and chlorobenzene-D5). The samples were analyzed by gas chromatography (GC) and GC/mass spectrometry (MS) according to work instructions (WI) 003 and 004, respectively, in the Johnson Space Center (JSC ) Toxicology Laboratory. The Toxicology Laboratory is ISO 9000 certified.

During the Phase II test, formaldehyde was monitored using Fourier Transform Infrared (FTIR) spectroscopy of grab sample canister (GSC) contents. The major limitation of this method is that its detection limit is near 2 mg/m3, which is well above the long-term exposure limit for exposure to this irritant. For tests IIa and III, formaldehyde badge samples were obtained periodically, most often from chamber level 1, with nominal sampling durations of 24 hours. This improved the formaldehyde detection limit by approximately 100-fold. The diffusion-controlled, badge samples were analyzed by the chromotrophic acid colorimetric method according to WI-006 in the JSC Toxicology Laboratory.

During parts of the Phase IIa test, when formaldehyde became a crew health issue, the badge measurements were confirmed with two active sampling methods. In the first method, formaldehyde was trapped in impingers containing a 1% sodium bisulfite solution, and the solution was subsequently analyzed by a chromotrophic acid colorimetric method. In the second method (EPA TO-11), formaldehyde was reacted with dinitrophenylhydrazine, which was coated onto silica gel beads in a tube. The tubes were extracted with acetonitrile, and the solution was analyzed by high-pressure liquid chromatography.

Ammonia was monitored during Phases IIa and III with an Interscan Model 2900, which used an electrochemical cell to detect ammonia. The instrument was calibrated with a gas permeation source at 4 mg/m$^3$.

***Toxicological Assessment of Mixtures of Pollutants***

The mixture of pollutants present in the atmosphere was assessed according to methods applied to spacecraft atmospheres. The average toxicity index for each toxicological group (Tgrp) was calculated for groups of "n" toxicants found at their respective concentrations (Cn) for 30 to 90 days and causing similar toxic effects or targeting the same organ system (e.g. respiratory system irritants, cardiotoxicants, carcinogens, etc). The equation below was used with 180-day spacecraft maximum allowable concentrations (SMACs):

$$Tgrp = C_1/SMAC_1 + C_2/SMAC_2 + .... + C_n/SMAC_n$$

The atmosphere was considered acceptable if each Tgrp value was <1.0. Certain SMACs have been set lower because of the effects of space flight (e.g. immune effects, hematological effects, etc.), hence, for the Earth-based application in this study, a few of the SMACs may be lower than necessary to fully protect crew health.

**Findings**

*Phase II 30-Day Test*

Even though the atmosphere throughout the test was acceptable for human respiration based on the T-value calculations from nine GSC samples, several

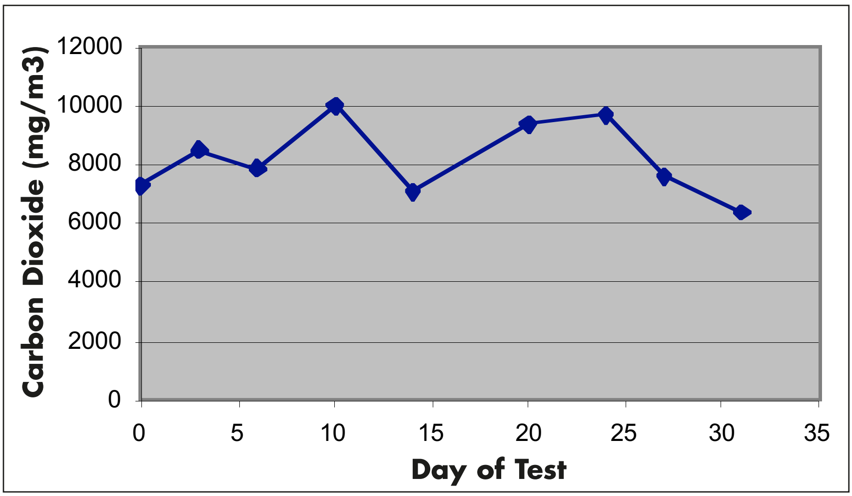


***Figure 4.1-1b*** *Carbon Dioxide in the 30-day test*

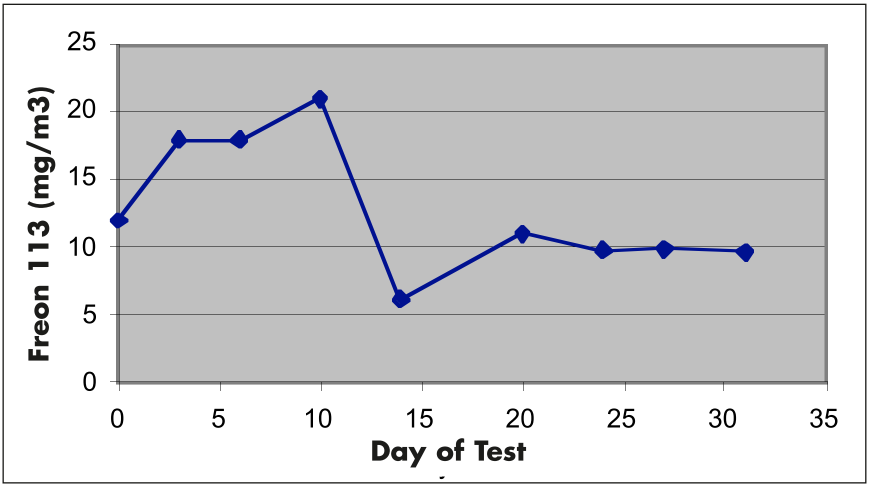


***Figure 4.1-2*** *Freon® 113 in the 30-day test*

atmospheric anomalies occurred during the test. On test day, six the carbon dioxide reduction system failed due to flooding of the methane/water separator. After replacement of the faulty, low-level water sensor, a methane leak was detected from one of the separator fittings and this was replaced. This occurred over a period of approximately three days and caused unusually high levels of methane and, to a

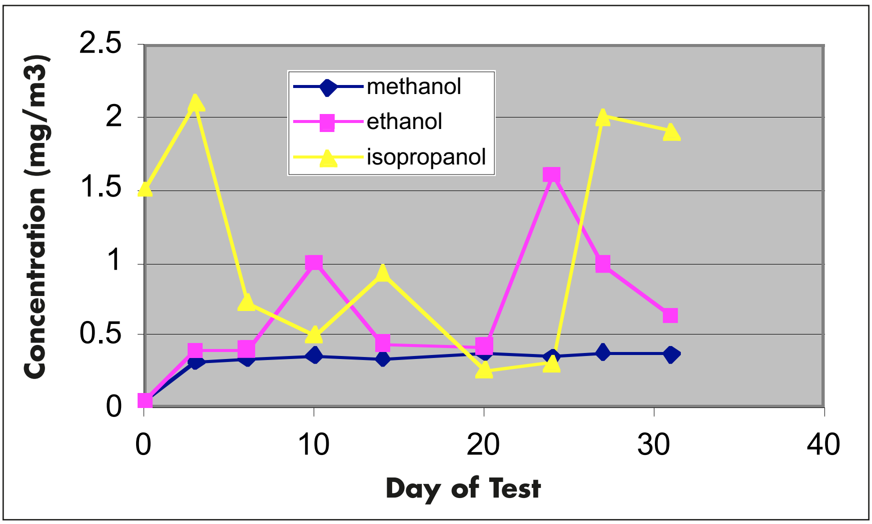


***Figure 4.1-3*** *Alcohols in 30-Day test*

lesser extent, carbon dioxide in the day 10 sample. The methane concentration slowly decayed throughout the remaining 20 days of the test.

The concentration of carbon monoxide was somewhat higher than that typically observed in space vehicles and the level of Freon® 113 was much higher than typically observed in space vehicles. The carbon monoxide concentrations increased from trace to approximately 4 mg/m3 by day 10 and stayed near that level until the end of the test. The Freon® 113 concentrations were relatively high before the test began (12 mg/m$^3$) and increased through day 10 to about 20 mg/m$^3$, after which they stabilized at about 10 mg/m$^3$. This compound probably originated from the pre-test cleaning of electronic components of hardware.

Some of the low-molecular-weight alcohols exhibited interesting behavior. Ethanol and isopropanol concentrations varied from about 0.3 to 2 mg/m$^3$ during the test. The variation was undoubtedly due to the use of these alcohols in the hand wipes and sterilizing pads. This is in contrast to methanol, which maintained a steady state concentration of about 0.35 mg/m$^3$ throughout the test. Methanol originates primarily from hardware offgassing and one would expect the continuous rate of production and the rate of removal to result in a nearly uniform concentration.

As noted in the methods section, formaldehyde was measured during the 30-day test using FTIR spectroscopy on aliquots taken from the GSCs. This resulted in a method that was relatively insensitive to formaldehyde and led to concentrations that were consistently reported as less than the method detection limit. The method was replaced by a much more sensitive badge-sampling method, and this change proved to be a fortuitous improvement, as the 60-day test demonstrated.

*Phase IIa 60-Day Test*

The dynamics of air pollutants during the Phase IIa test were much different than during the Phase II testing. From an air-quality perspective the 60-day test can be summarized as a learning experience about the importance of controlling materials offgassing.

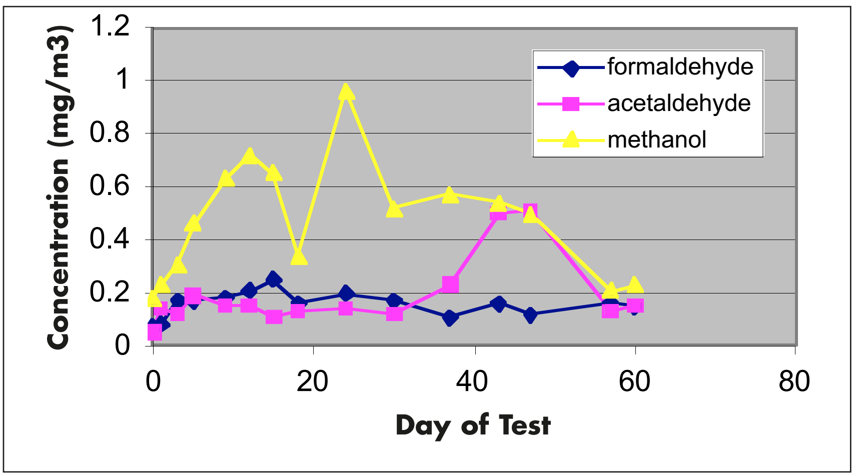


***Figure 4.1-4*** *Selected Airborne Pollutants During the 60-Day test*

Steady-state concentrations were not achieved for methanol, acetaldehyde, and formaldehyde until the last few days of the study. Formaldehyde was of particular concern because the measured values increased to 0.25 mg/m³ by day 15, whereas the long-term SMAC is only 0.05 mg/m³ (8).

The accuracy of the badge method was confirmed by comparing it to an impinger method and an U. S. Environmental Protection Agency (EPA) method. The day 27 badge result from level 1 was 0.17 mg/m³, the coincident impinger sample was 0.17 mg/m³, and the average of four EPA-type samples was 0.18 mg/m³. A number of materials inside the chamber quickly underwent offgas testing to determine their rate of formaldehyde production. Most materials did not offgas detectable levels of formaldehyde; however, the poster murals were found to release measurable amounts of formaldehyde and were removed from the chamber on day 17. The airborne formaldehyde dropped from its high of 0.25 mg/m³ on day 15 to 0.16 mg/m³ on day 18.

Three compounds, coming primarily from anthropogenic sources, showed very different concentration profiles. During this test the primary methane source was

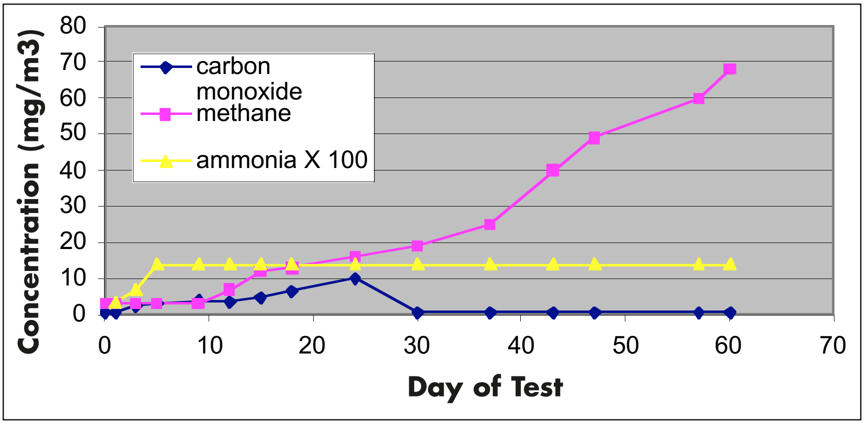


***Figure 4.1-5*** *Anthropogenic Pollutants in the 60-Day test.*

the human occupants; there was no evidence of a system leak such as that seen during the Phase II test. Methane concentrations increased steadily with time as the test progressed. Carbon monoxide also exhibited this behavior until day 30 when an abrupt drop in the concentration occurred. After this time, carbon monoxide was never found above a trace amount (about 0.5 mg/m³). Ammonia concentrations reached a steady state level of 0.14 mg/m³ by day 5 of the test and did not change from this level in the remaining 55 days.

*Phase III 90-Day Test*

Air pollutants were better controlled during most of this test than during the Phase IIa test; however, there was evidence that a new source of air contamination

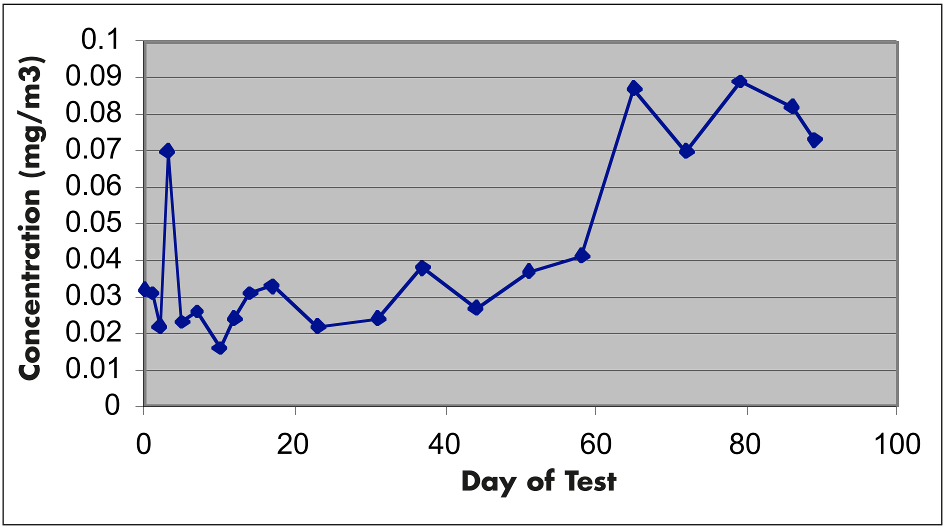


***Figure 4.1-6*** *Formaldehyde in 90-Day test*

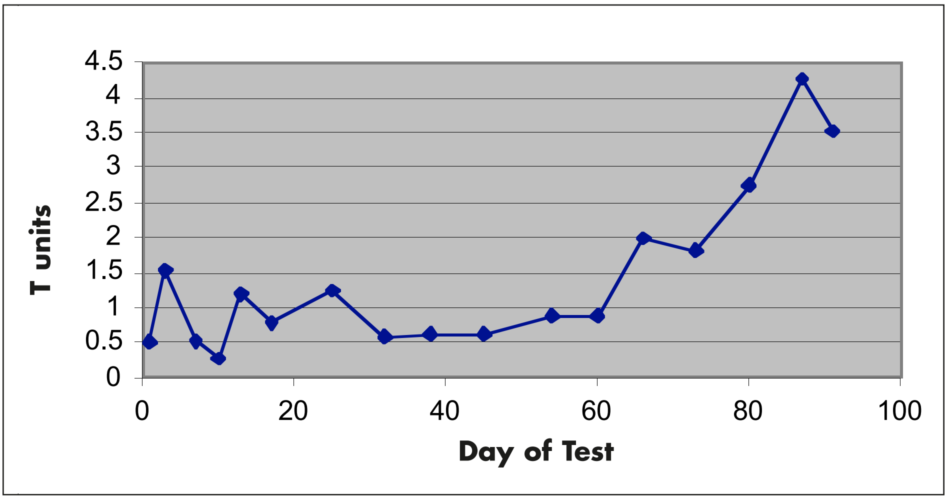


***Figure 4.1-7*** *T Value for Irritants in the 90-Day test*

was introduced late in the test and this caused a large increase in the concentration of respiratory irritants. Separate from this was a slight increase in formaldehyde toward the end of the test, but this was apparently due to an anomaly in a catalyst bed rather than excessive offgassing of materials as found in the 60-day test. The formaldehyde profiles are shown in Figure 4.1-6 and the Tgrp for the irritants is shown in Figure 4.1-7. Another distinct difference between the 60-day test and the 90-day test was the accumulation of ammonia during the latter test. The abrupt increase of common pollutants near the end of the test is shown in Figure 4.1-9.

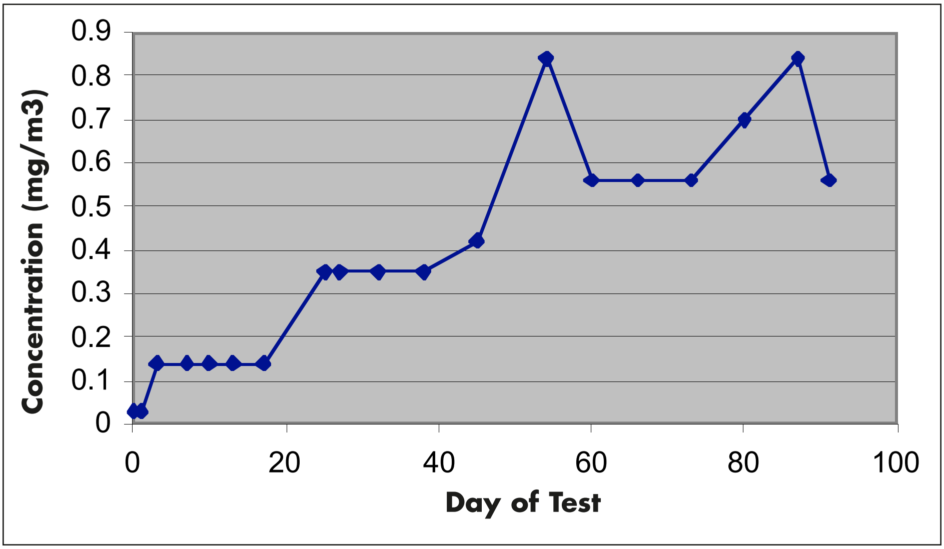


***Figure 4.1-8*** *Ammonia in the 90-Day test*

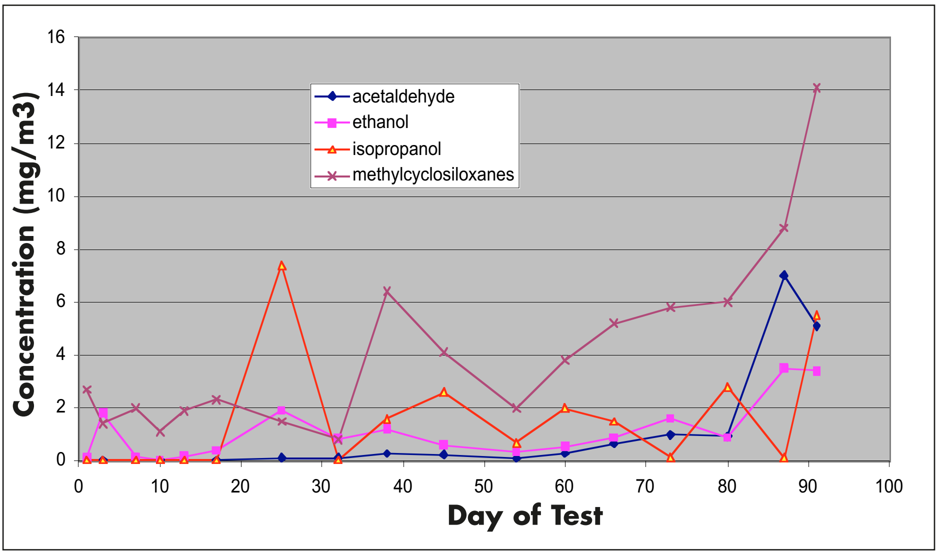


***Figure 4.1-9*** *Major Pollutants in the 90-Day test*

## Discussion

*Phase II 30-Day Test*

Even though the air quality seemed to be acceptable during this test, there were important limitations to the methods used to measure pollutants. Specifically, the FTIR method of quantifying formaldehyde from aliquots of the GSC samples proved to be too insensitive to provide useful information. Formaldehyde concentrations during this test may have been comparable to those measured during the Phase IIa test because the materials used in both tests were similar. Had we recognized the importance of measuring low concentrations of formaldehyde, we would have been better prepared to conduct the Phase IIa test in an uneventful manner.

The total T-values, with the contribution from carbon dioxide removed because it acts independently of other pollutants, and formaldehyde and ammonia not quantified, ranged from 0.32 to 0.58 during the 30-day test. This suggests that the trace pollutants were collectively quite stable during the test and that the atmosphere was easily within acceptable limits for human respiration. Given these low T-values, there was no need to separate the compounds according to toxicological groups.

*Phase IIa 60-Day Test*

Pollutant levels during this test were significantly higher than those typically encountered in space flight or during the Phase II test. In part this was due to excess offgassing from polymeric materials that had not received adequate testing for their offgassing properties. This led to concentrations of formaldehyde well above accepted limits and resulted in symptoms being reported in one crewmember. A concerted effort was mounted to identify the source(s) of the formaldehyde, with limited success during the test. Removal of murals on day 17 reduced the formaldehyde concentrations somewhat, but these items apparently were only one of the sources of formaldehyde.

The search for other sources of formaldehyde included evaluations after the study and a "bake out" study after the crew left the chamber. This bake out study demonstrated that the equilibrium between formaldehyde sources and removal processes was shifted to produce higher airborne concentrations as the chamber temperature increased. Post-test analyses by the Crew and Thermal Systems Division also indicated that the melamine foam acoustic tiles and carpeting were important sources of formaldehyde. A 40 g sample of the foam reached an equilibrium concentration of 0.5 $mg/m^3$ inside a 10 L bell jar. These tiles were removed from the test chamber and replaced with solamide tiles for the Phase III test.

One crewmember reported eye and upper-airway irritation as the formaldehyde concentrations climbed to their peak of 0.25 $mg/m^3$ on day 15 of the test. These symptoms should be expected at this level of formaldehyde, but not in every crewmember. There is a population of persons who are much more sensitive to the irritant properties of formaldehyde than the general population. The SMAC of 0.05 $mg/m^3$ was set to protect even sensitive individuals (8). In contrast, the Threshold Limit Valve (TLV®) of 0.3 ppm (0.4 $mg/m^3$) was set to protect the majority of workers, with the understanding that "the recommended formaldehyde 0.3 ppm ceiling TLV® will not protect that portion of the workforce reported to be responsive to low ambient concentrations of this chemical."

There were a number of adjustments in the trace contaminant control devices throughout the 60-day test. Normally, methanol is generated at a fairly constant rate from materials offgassing. The large changes in methanol concentration suggest that changes in the trace contaminant control devices caused most of these concentration changes. On the other hand, the drop on day 18, as depicted in Figure 4.1-4, may be from removal of materials on day 17 in an attempt to reduce offgassing of formaldehyde.

Carbon monoxide increased steadily during the first 24 days of the test because there was no removal mechanism as shown in Figure 4.1-5. The measurement on day 24 was 10 $mg/m^3$, which is just below the long-term SMAC of 11 $mg/m^3$ (10 ppm) for this compound. On day 25 the high temperature catalytic bed was started and this caused a dramatic drop in concentration. This action seemed to have no measurable effect on the steadily rising methane concentrations; however, methane is known to be more difficult to oxidize than carbon monoxide.

The total T-values for all measured pollutants except carbon dioxide and formaldehyde ranged from 0.15 (pretest) to 1.84 (day 12). The T-values reached much higher numbers than during the Phase II test. Four of the T-values (days 5, 12, 24, and 37) were significantly above 1, and these were broken down into toxicity groups to determine if any single group exceeded a value of 1. The following groups were identified and ranges found: irritants without formaldehyde (0.38-0.46), neurotoxicants (0.11 to 1.05), respiratory system injury (0.26 to 0.55), hepatotoxicants (0 to 0.71), gonad toxicants (0.11 to 0.55), immunotoxicants (0 to 0.12), carcinogens (0 to 0.23), and cardiotoxicants (0.05 to 0.95). The only unacceptable value was for neurotoxicants, which was due to the one relatively high value of carbon monoxide found on day 24. Since long-term SMACs were used to calculate the T values, and the exposure was no more than a few days, there was an extremely low risk of any neurotoxicity.

*Phase III 90-Day Test*

Until day 80 the total T-values, without carbon dioxide and formaldehyde, ranged from 0.06 to 1.89, which was comparable to the Phase IIa result. The remarkable increase in acetaldehyde and ethanol late in the test can be attributed in part to fermentation processes such as the baking of bread. These processes are known to produce large amounts of ethanol and metabolic products such as acetaldehyde. The cause of the increase in concentration of the methylcyclosiloxanes is unknown.

The slight increase in formaldehyde concentrations after day 60 has been attributed to incomplete oxidation of methanol in a catalytic bed (12). This cause was determined after the 90-day test by evaluating the performance of the catalyst bed. Under test conditions of 200 deg C, approximately half the input methanol was reacted, but 2/3 of the reacted methanol was converted to formaldehyde rather than water and carbon dioxide. Further investigation suggested that the catalyst had been poisoned by organic sulfur compounds (12). The highest formaldehyde levels reached (0.09 $mg/m^3$) were still well below those expected to elicit symptoms in most individuals.

The cause of the increase in ammonia during the test (see Figure 4.1-8) was due to venting of the bioreactor head gas and headspace above the waste-water tanks directly into the TCCS beginning on day 21 (10). Apparently, the ammonia-conversion catalyst in the TCCS was not fully capable of converting the additional load of ammonia. Hence, the ammonia concentration began to increase at this time and had not reached a steady-state concentration by the end of the 90-day test.

## SIGNIFICANCE

The findings reported here underscore the need for comprehensive air quality analyses to determine whether preventative measures to limit pollution have been effective, to ascertain if the ARS is capable of dealing with the pollutant load on a sustained basis, to detect any new sources of air pollution, and to judge whether the air has been acceptable for crew health. These goals can be achieved only in a test chamber or space vehicle due to the complex interactions between the sources and sinks. Such interactions will only be made more complex as food preparation and waste processing systems are integrated into habitats.

During the LMLSTP the analyses were retrospective, yet they still provided valuable insight into the dynamic changes that were occurring in the chamber. NASA is on the threshold of being able to analyze spacecraft air for trace pollutants on a near-real time basis, and this will further enhance the value of air quality assessments. Future research should focus on understanding the risks that specific air pollutants pose to crew health, and then developing analyzers capable of addressing those risks using a minimum of resources. That research must be conducted in realistic, ground-based environments before analytical hardware is flown in space vehicles, which one can only hope will be headed to Mars in the not-too-distant future.

**Acronyms**

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| ARS | Air Revitalization System |
| EPA | U.S. Environmental Protection Agency |
| FTIR | Fourier Transform Infrared |
| GC | Gas Chromatography |
| GSC | Grab Sample Canister |
| ISS | International Space Station |
| JSC | Johnson Space Center |
| LMLST | Lunar Mars Life Support Test |
| MS | Mass Spectrometer |
| SMAC | Spacecraft Maximum Allowable Concentration |
| TCCS | Trace Contaminate Control System |
| TLV® | Threshold Limit Value |
| Tgrp | Toxicological Group |
| WI | Work Instruction |

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