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# Space Station Freedom Environmental Control and Life Support System Phase III Simplified Integrated Test Detailed Report

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# NASA

# ACKNOWLEDGMENTS

# TABLE OF CONTENTS

# LIST OF ILLUSTRATIONS

# LIST OF ILLUSTRATIONS (Concluded)

# LIST OF TABLES

# LIST OF ACRONYMS AND ABBREVIATIONS

AR          Air revitalization

BE          Boeing environmental

CCA         Coolant control assembly

CMIF        Core Module Integration Facility

CMS         Core Module Simulator

cc          Cubic centimeter

ccm         Cubic centimeter per minute

CFM         Cubic feet per minute

EEF         End-use equipment facility

ECLSS       Environmental control and life support system

°F          Fahrenheit degrees

ft          Feet

FID         Flame ionization detector

FCA         Fluids control assembly

4BMS        Four-bed molecular sieve

GC/MS       Gas chromatography/mass spectrometer

h           Hour

in          Inch

K           Kelvin degrees

kg          Kilogram

MSFC        Marshall Space Flight Center

MCT         Metabolic control test

| | |
|---|---|
| MP | Materials and Processes |
| m | Meters |
| mm | Millimeters |
| mmHg | Millimeters of mercury |
| min | Minute |
| NASA | National Aeronautics and Space Administration |
| p | Partial pressure |
| ppm | Parts per million |
| Pa | Pascal |
| PDU | Performance diagnostic units |
| KOH | Potassium hydroxide |
| lbm | Pound mass |
| psi | Pounds per square inch |
| psia | Pounds per square inch absolute |
| psid | Pounds per square inch difference |
| psig | Pounds per square inch gauge |
| PCA | Pressure control assembly |
| QC | Quality control |
| RPD | Relative percent difference |
| s | Second |
| SIT | Simplified integrated test |
| S.S. Freedom | Space Station Freedom |
| SMAC | Spacecraft maximum allowable concentration |

| slpm | Standard liters per minute |
|------|---------------------------|
| SFE | Static feed electrolyzer |
| SCATS | Systems and Components Automated Test System |
| THCS | Temperature and humidity control system |
| TC | Total carbon |
| TIC | Total inorganic carbon |
| TOC | Total organic carbon |
| TCCS | Trace contaminant control subsystem |
| WRT | Water recovery test |

TECHNICAL MEMORANDUM

# SPACE STATION FREEDOM ENVIRONMENTAL CONTROL AND LIFE SUPPORT SYSTEM PHASE III SIMPLIFIED INTEGRATED TEST DETAILED REPORT

## 1.0 INTRODUCTION

The Core Module Integration Facility (CMIF), in building 4755 of Marshall Space Flight Center (MSFC), is the National Aeronautics and Space Administration (NASA) and Boeing test area for the development of the Space Station Freedom (S.S. Freedom) environmental control and life support system (ECLSS). The current CMIF layout is shown in Figure 1.

Testing of individual ECLS subsystems has been conducted in the subsystem test area of the CMIF prior to integrated system testing. This bench testing is referred to as phase I ECLSS testing. Integrated system testing is conducted in the Core Module Simulator (CMS). A test control room is located next to the CMS. Also located next to the CMS is a facility for generating waste water for use in water recovery system tests. This facility is referred to as the end-use equipment facility (EEF).

Currently, the CMIF is also the location for Boeing's comparative test program which is assessing alternative ECLS subsystems to their proposed S.S. Freedom baseline design. This test area is shown in Figure 1.

Since 1987, MSFC-phased testing of the ECLSS has been pursued at the CMIF. In 1989 Boeing began their ECLSS development testing (Fig. 2). Phase I consisted of the independent testing of individual subsystems that are being considered for use in the S.S. Freedom. Integrated testing of subsystems was conducted by MSFC in phase II and continues to be conducted in phase III. A summary of the overall ECLSS development test program is given in Reference 1, and the results of phase II testing are given in References 2 and 3.

An overview of MSFC phase III testing is shown in Figure 3. Requirements for phase III testing are given in References 4 and 5. Phase II used a set of available hardware to prove the subsystem integration capability of typical equipment focusing on oxygen recovery. Phase III utilizes predevelopment equipment of the S.S. Freedom baseline subsystems version and is more comprehensive than previous testing because it includes all water recovery subsystems as well as the air revitalization (AR) system. The independent testing of the AR subsystems in the CMS began the phase III activities. The simplified integrated test (SIT) of these subsystems was conducted July 31 to August 11, 1989, and is described in detail in this report.

The phase III SIT was similar to the phase II SIT in that both were tests of integrated AR subsystems. There were three major differences in the phase II SIT and phase III SIT. First, in the phase III SIT, the Bosch carbon dioxide reduction subsystem, which is a baseline subsystem for the Boeing S.S. Freedom ECLSS, replaced the Sabatier carbon dioxide reduction subsystem that was used in the phase II SIT. This was the first time the Bosch design had been successfully

2
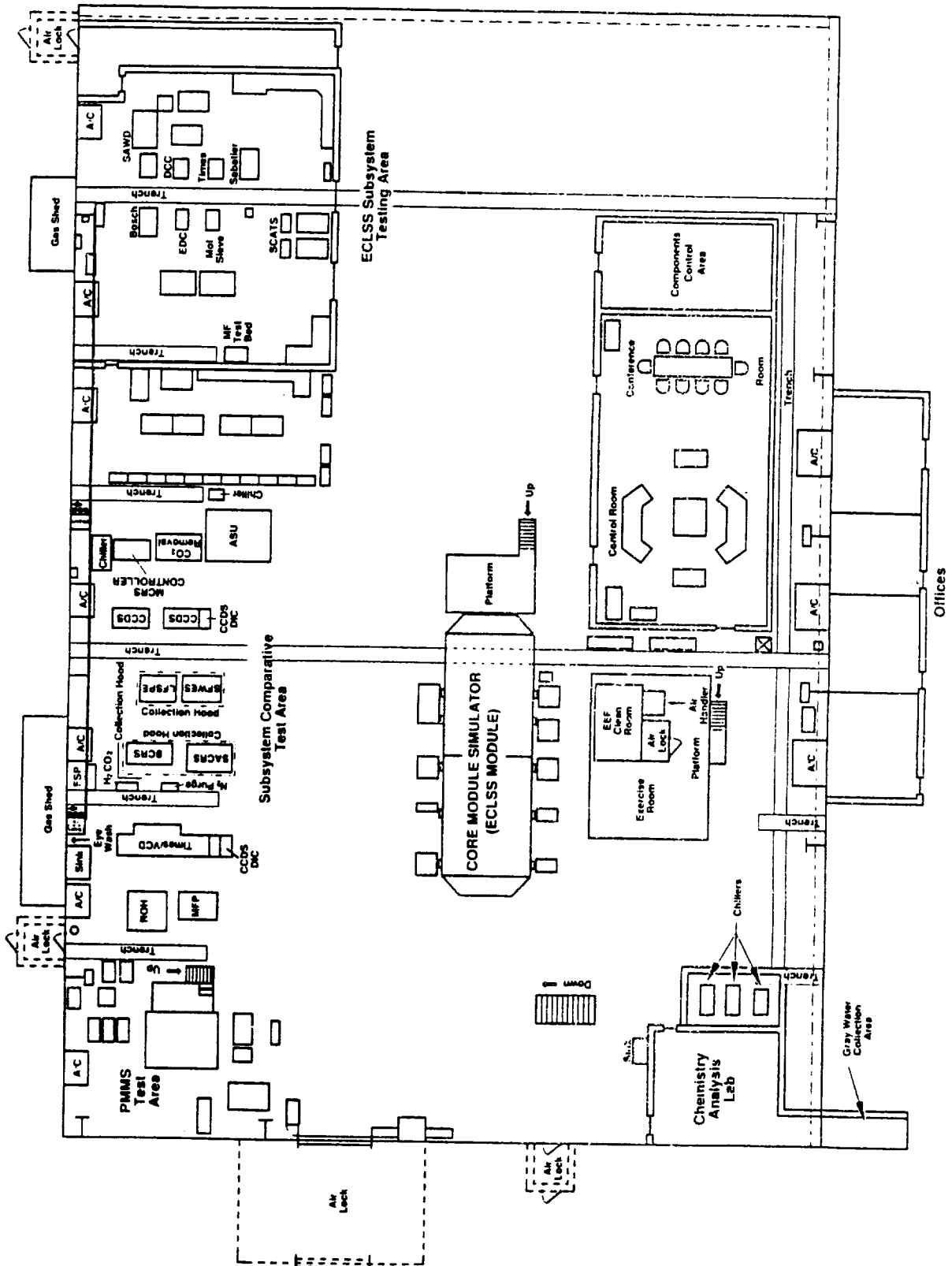


Figure 1. Core Module Integration Facility.
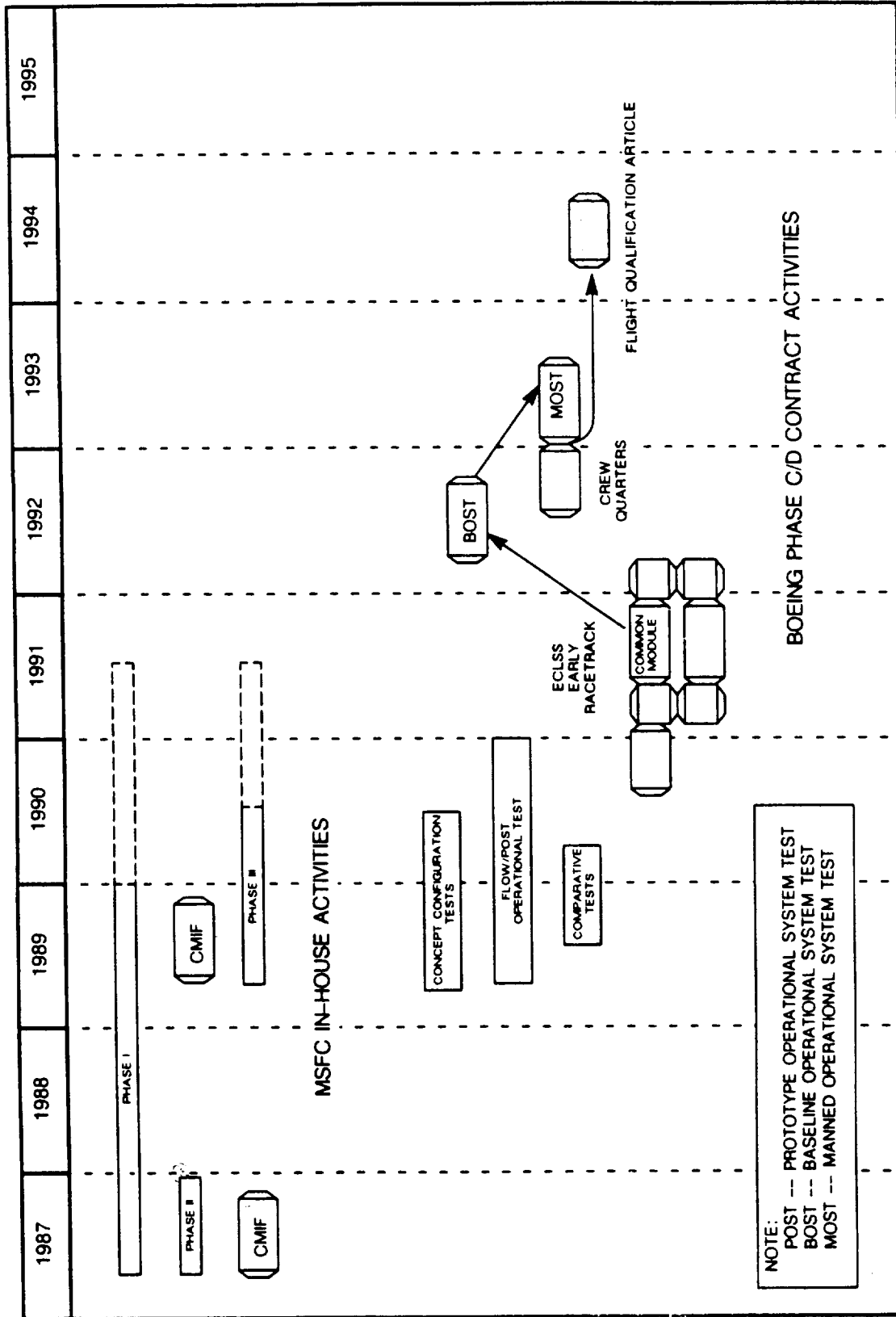
Figure 2. ECLSS development test program.
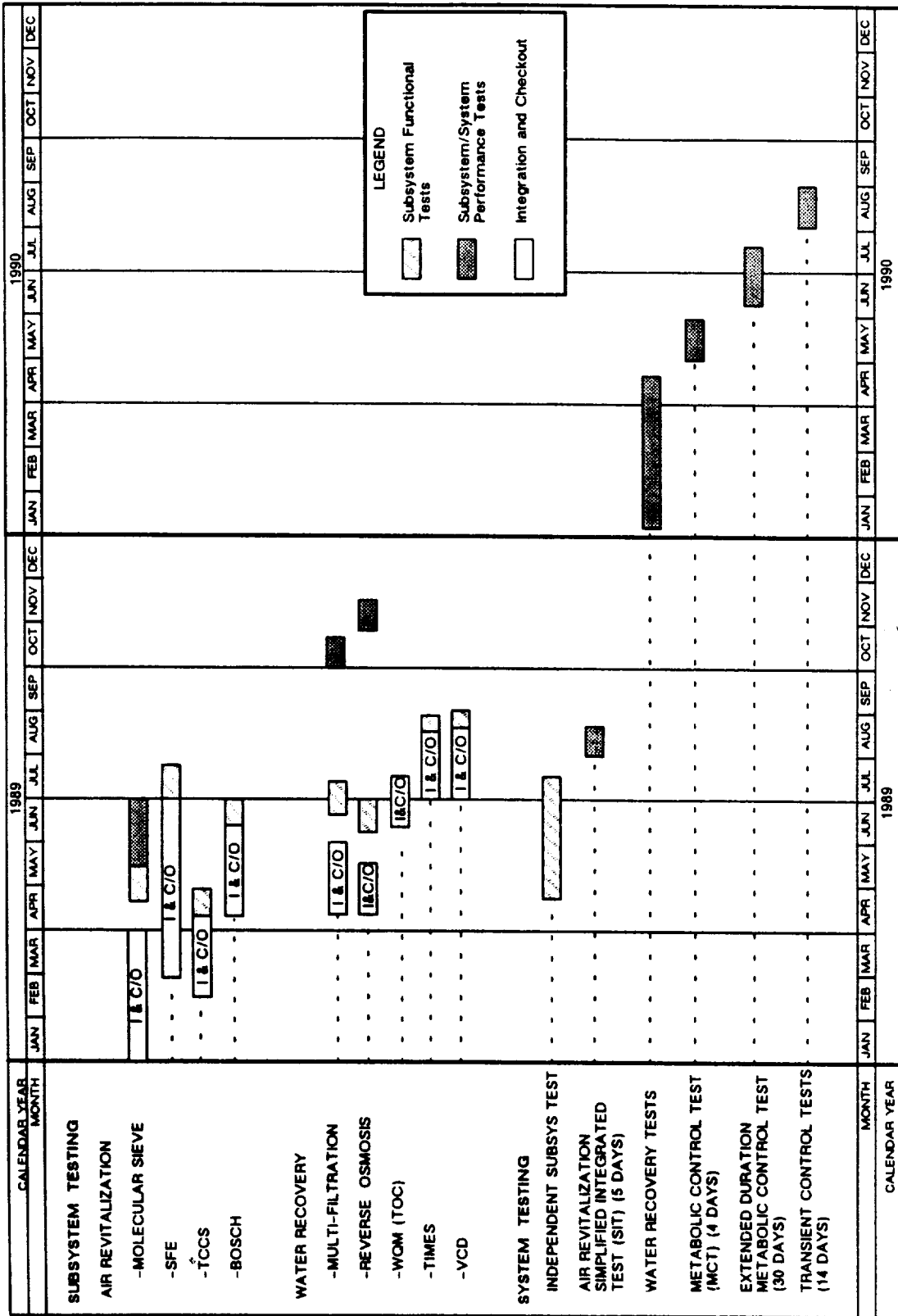
Figure 3. ECLSS phase III test schedule.

CALENDAR YEAR / MONTH

1989

1990

JAN | FEB | MAR | APR | MAY | JUN | JUL | AUG | SEP | OCT | NOV | DEC

**SUBSYSTEM TESTING**

AIR REVITALIZATION

- MOLECULAR SIEVE — I & C/O
- SFE — I & C/O
- TCCS — I & C/O
- BOSCH — I & C/O

WATER RECOVERY

- MULTI-FILTRATION — I & C/O
- REVERSE OSMOSIS — I&C/O
- WQM (TOC) — I&C/O
- TIMES — I & C/O
- VCD — I & C/O

**SYSTEM TESTING**

INDEPENDENT SUBSYS TEST

AIR REVITALIZATION SIMPLIFIED INTEGRATED TEST (SIT) (5 DAYS)

WATER RECOVERY TESTS

METABOLIC CONTROL TEST (MCT) (4 DAYS)

EXTENDED DURATION METABOLIC CONTROL TEST (30 DAYS)

TRANSIENT CONTROL TESTS (14 DAYS)

MONTH

CALENDAR YEAR

LEGEND

Subsystem Functional Tests

Subsystem/System Performance Tests

Integration and Checkout

integrated with other oxygen recovery subsystems in ECLSS testing by NASA. The major test objectives of SIT were to prepare for later fully integrated oxygen with water recovery testing in the metabolic simulation testing to follow, as such to identify integration problems and focus on determining areas where further testing is needed to prove the integration concept is viable.

Secondly, the trace contaminant control system (TCCS) was integrated and monitored differently in phase III than in phase II. Direct integration with the Bosch was implemented to process product gases of the carbon dioxide reduction design.

Finally, the phase III SIT subsystems ran completely integrated for approximately 5 of the 11 days, whereas the phase II SIT ran completely integrated for 2 days.

## 2.0  SYSTEM DESIGN/PERFORMANCE

The SIT configuration utilized four air revitalization subsystems, baselined by Boeing, located inside the module simulator at MSFC. These four subsystems were the static feed electrolyzer (SFE) oxygen generation subsystem, the four-bed molecular sieve (4BMS) carbon dioxide removal subsystem, the Bosch, and the TCCS. A facility simulator of the temperature and humidity control system (THCS) was also located inside the module to provide ventilation and sensible and latent heat removal inside the module. Located outside the module and inside a control room were performance diagnostic units (PDU), used to monitor the SFE and Bosch, and the facility-provided Systems and Components Automated Test System (SCATS) computer for data acquisition and management. Other facility items, such as bottled gases for system and subsystem purging and power supply services, were also provided outside the module.

Figure 4 shows a block diagram of subsystems and fluid interchanges with the phase III SIT. Each subsystem is capable of handling the metabolic requirements of a three-man crew. The SFE generated oxygen and hydrogen from facility-provided water, the 4BMS removed carbon dioxide ($CO_2$) from the module atmosphere, and the Bosch took the $CO_2$ and reacted it with hydrogen ($H_2$) to produce water ($H_2O$) and carbon (C). Any gases not reacted in the Bosch are then vented to the TCCS.

The 4BMS subsystem removes carbon dioxide from the cabin atmosphere via an adsorption process. The air is pulled from the cabin downstream of the condensing heat exchanger in the THCS and is returned just upstream of the condensing heat exchanger. Carbon dioxide adsorbed out of the air by the 4BMS is fed to an accumulator and then flows to the Bosch.

The SFE subsystem electrolyzes water into gaseous hydrogen and oxygen. During this test, deionized water was provided by the facility for the electrolysis process. All oxygen produced by the SFE was vented outside the simulated module. The hydrogen produced was fed to the Bosch subsystem.

The Bosch takes in hydrogen from the SFE and carbon dioxide from the 4BMS and produces water and carbon in a high temperature chemical reaction. Carbon produced by the Bosch is formed in the reactor catalyst beds inside the subsystem and the water vapor is condensed, collected, and stored.

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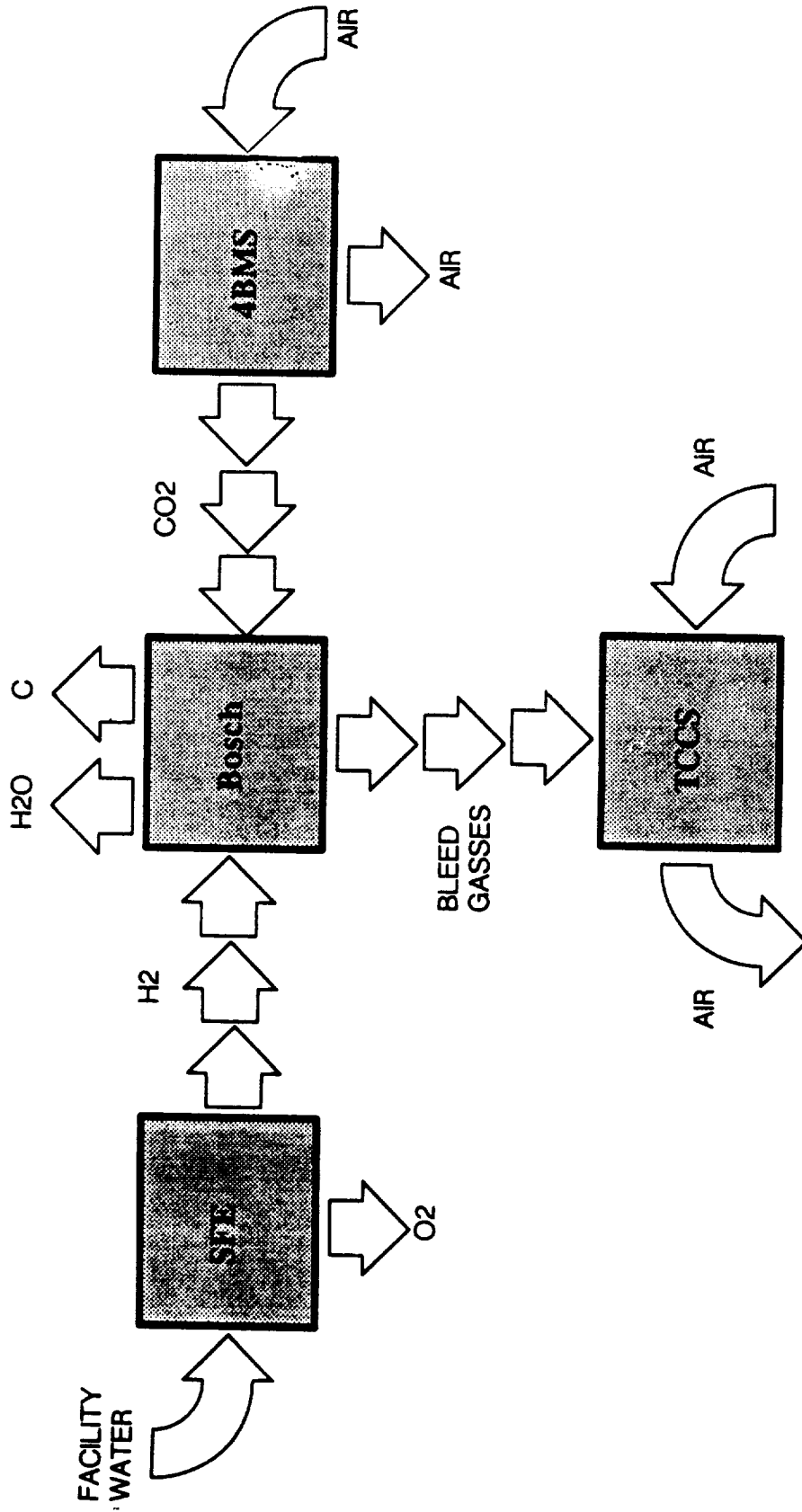
Figure 4. Phase III simplified integration test (SIT) elements.

The TCCS is designed to remove contaminants from the module atmosphere. Inlet air is withdrawn downstream of the condensing heat exchanger in the THCS and is returned to the cabin directly via a vent in the top of the subsystem.

The THCS is built from "off-the-shelf" components and is designed to remove latent and sensible heat via a condensing heat exchanger. An axial fan is located upstream from the heat exchanger to circulate air. A variable damper is located upstream of the heat exchanger to control temperature. All components of the THCS are located below the subfloor of the module simulator. The air intake duct of the THCS is located below the subfloor and the cabin supply ducts are located along the ceiling.

A picture of the subsystems inside the module simulator is shown in Figure 5 and the general layout of each subsystem location inside the module is shown in Figure 6.

A detailed schematic of the SIT configuration is shown in Figure 7. All elements inside the bold black line were located inside the module. A total of 160 sensors were used to monitor the SIT. Of these measurements, 48 were part of facility operations, 20 were located in the integration hardware between subsystems, and 92 were internal to the subsystems.

All the ECLSS subsystems used in this test were "pre-prototype" hardware. No effort was made to simulate higher system management functions, such as thermal control. The door to the module simulator was left open throughout the test to allow easy access for any necessary equipment repairs or replacements. No attempt was made during this test to simulate flight-like conditions. This represents the first time S.S. Freedom AR subsystems, baselined for S.S. Freedom, have been operated in an integrated fashion. This test was simply a first attempt to study how these subsystems functioned when integrated together. A complete description of events which occurred during the test are given in a report published by MSFC's Test Laboratory [6].

# 3.0  SUBSYSTEM DESIGN/PERFORMANCE

## 3.1  Four-Bed Molecular Sieve

### 3.1.1  Detailed Description

The 4BMS, shown in Figure 8, was used to remove $CO_2$ from the module simulator air and concentrate it for processing by the Bosch. Cabin air with $CO_2$ flows through a sorbent material and the $CO_2$ is selectively adsorbed. Adsorption is the physical trapping of individual molecules in voids in the sorbent structure and does not result in a chemical or physical change in the sorbent itself (distinct from the process of absorption which involves a chemical reaction or a physical change or both in the sorbent material). In addition to the molecular size, the polarity of the molecules and the vapor pressure are important factors in the adsorption.

For the 4BMS, the $CO_2$ sorbent material used is a synthetic zeolite which was selected for its superior ability to adsorb $CO_2$. The designation for the $CO_2$ sorbent is Zeolite 5A. Due to the affinity of Zeolite 5A for water vapor, it is necessary to dry the air before passing it through the $CO_2$ sorbent beds. The desiccants used to do this are silica gel and another type of zeolite,

7
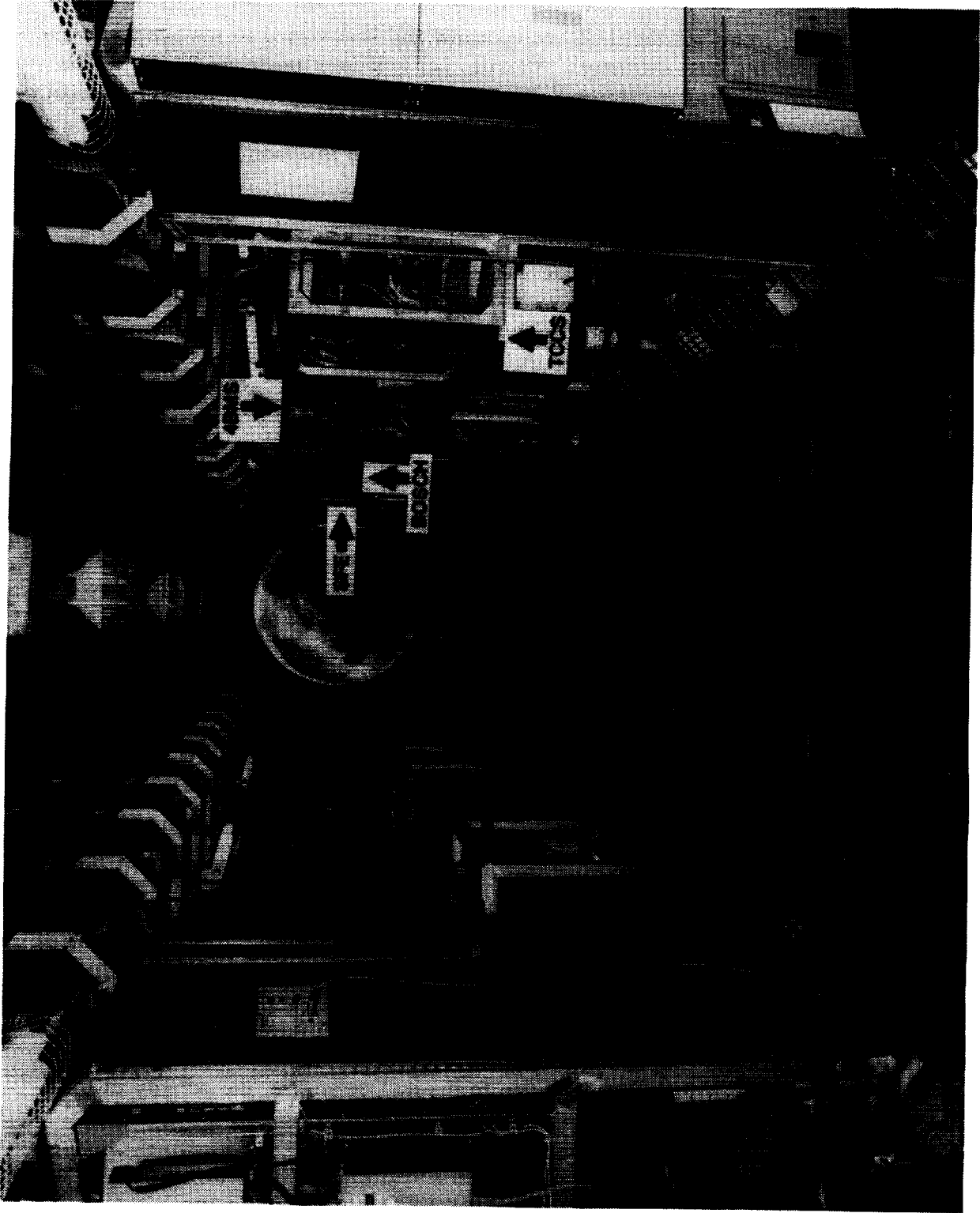
Figure 5. Interior of module simulator.

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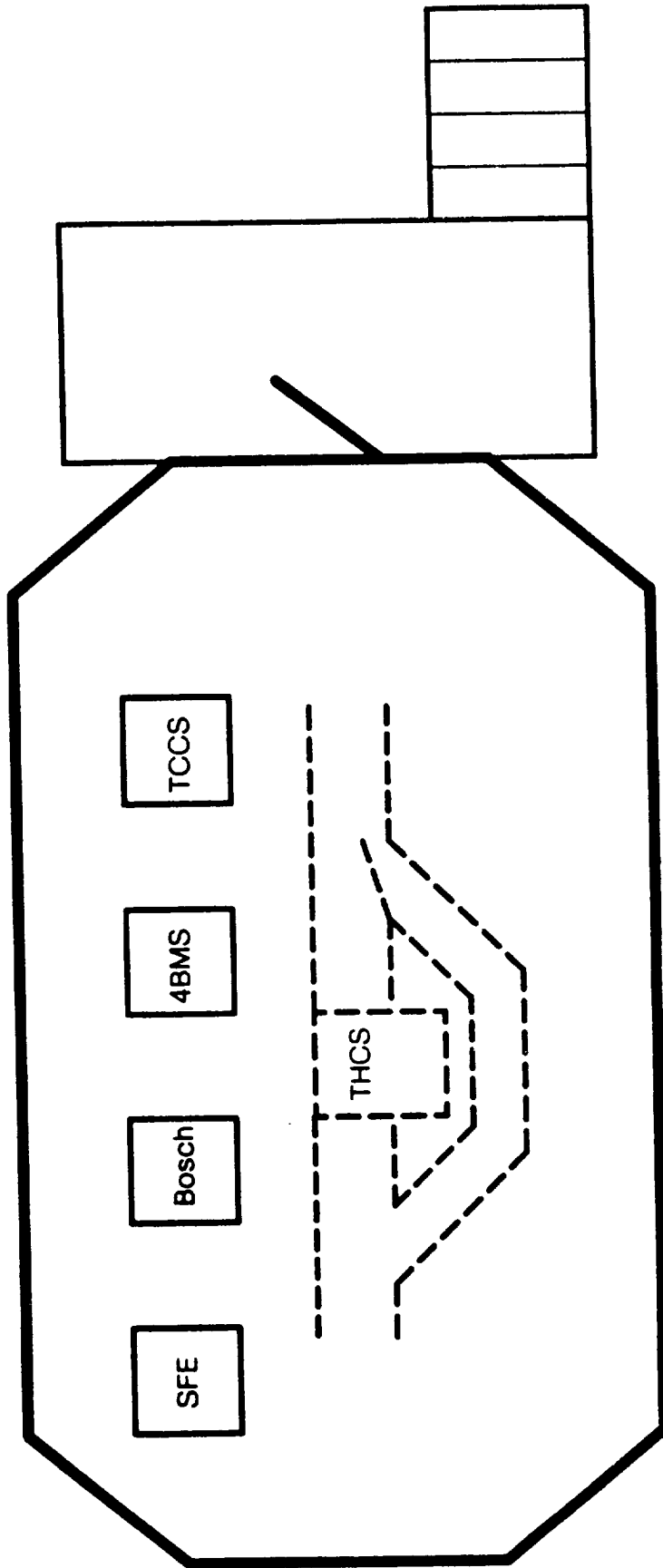
Figure 6. Module simulator diagram.
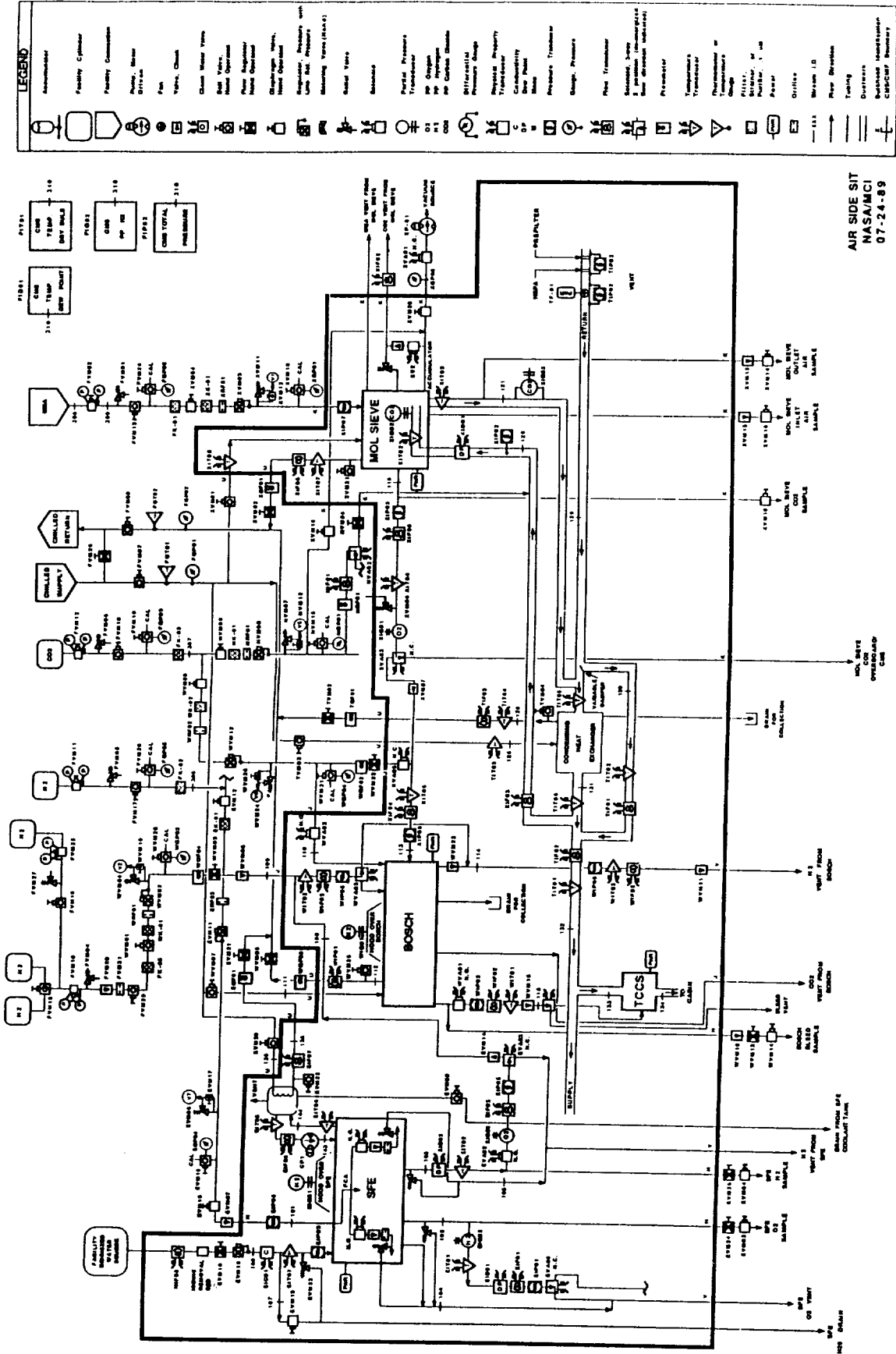
Figure 7. Phase III SIT schematic.
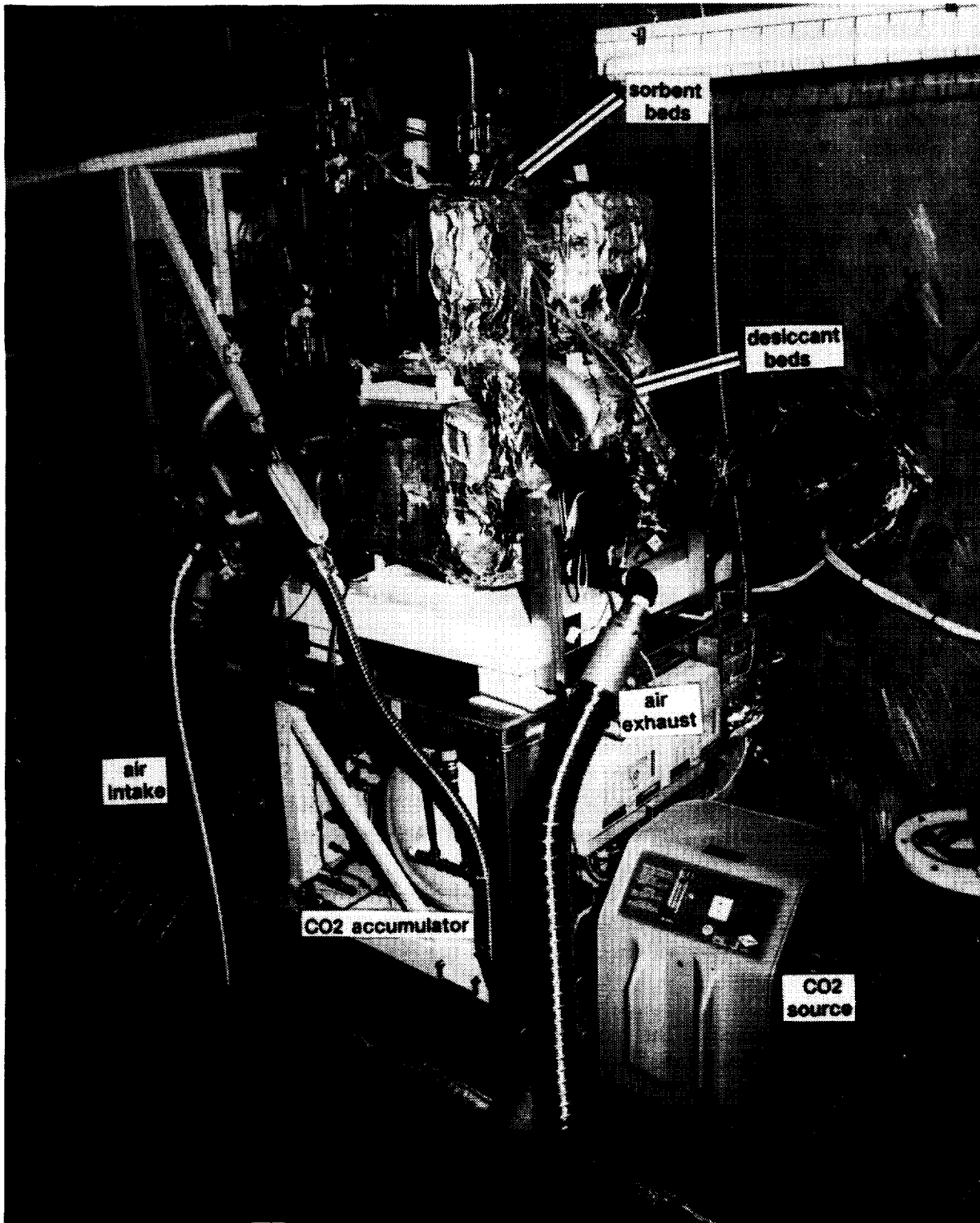
Figure 8. Four-bed molecular sieve.

designated Zeolite 13X. The sorbents are in the form of pellets approximately one-eighth of an inch long and one-sixteenth of an inch in diameter. During operation, the sorbents alternately adsorb and desorb and water vapor and $CO_2$, which requires two beds each of desiccant and $CO_2$ sorbent to perform $CO_2$ removal in an essentially continuous manner.

A schematic of the 4BMS and its instrumentation is shown in Figure 9. The flow path of the air through the 4BMS takes it first through a desiccant bed. The first layer is silica gel, which can adsorb water vapor readily at higher relative humidities, but its capacity decreases at relative humidities less than 50 percent. The second layer is Zeolite 13X, which has a higher capacity than silica gel at relative humidities less than 35 percent. By utilizing a two-layer bed arrangement of this configuration essentially all of the water vapor is removed efficiently. The adsorption process results in a temperature rise in the air stream. There is also a temperature rise across the blower. The blower is located downstream of the desiccant bed so that the temperature rise does not cause a drop in the relative humidity of the incoming air. Downstream of the blower, the precooler reduces the temperature of the air stream to temperatures more conducive to $CO_2$ adsorption (from about 361 to 294 K). The dry, cool air then flows through a $CO_2$ sorbent bed, cooling the bed (heated during the previous desorb half-cycle) to a temperature where much of the $CO_2$ is removed from the air stream. The air is next directed through the second desiccant bed to desorb the water that was adsorbed during the previous half-cycle. The outlet air is recycled to the inlet at the beginning of each half cycle in order to improve performance. After 11 minutes the desorbing desiccant bed has heated (due to residual heat in the now adsorbing $CO_2$ sorbent bed) enough to begin desorbing water vapor so the recycle valve switches to end recycle. The outlet air has the same average moisture content as the inlet air.

While one $CO_2$ sorbent bed is adsorbing $CO_2$, the other is desorbing $CO_2$ for storage in the accumulator tank prior to delivery to the Bosch. During desorption, the residual air in the canister is pumped back to the duct upstream of the blower. This is done for 2 min. The evacuated bed is then isolated, then the heater in the bed comes on to raise the temperature to about 477 K (400 °F) which, in combination with the pressure which is reduced to about 0.5 psia, releases the $CO_2$ from the zeolite where it is transported to the $CO_2$ accumulator. After about 55 min, the desorption is complete and the next half-cycle begins (see Fig. 10 for the operational modes).

The 4BMS consists of modified Skylab (utilized Skylab hardware for canisters and heaters, five-way valves) hardware and commercially available components (blower, vacuum pumps, $CO_2$ holding tank, controller, two-way valves). The unit is not optimized with regard to weight, volume, or power usage.

The 4BMS was installed in the module simulator so that it could be operated independently (and vent $CO_2$) or integrated with the Bosch for $CO_2$ reduction. During operation, air from the THC heat exchanger was ducted directly to the inlet of the 4BMS. This duct contained a flowmeter, a port for injection of $CO_2$, and a connection to a $CO_2$ partial pressure sensor. The $CO_2$ supply line contained a flowmeter and a metering valve for regulating the $CO_2$ flow. As the air flowed through the subsystem, temperature measurements were made at various locations (e.g., inlet upstream and downstream of the precooler, downstream of the sorbent beds). The $CO_2$ partial pressure ($pCO_2$) was measured again at the air exit. The air exited into the volume of the simulator.
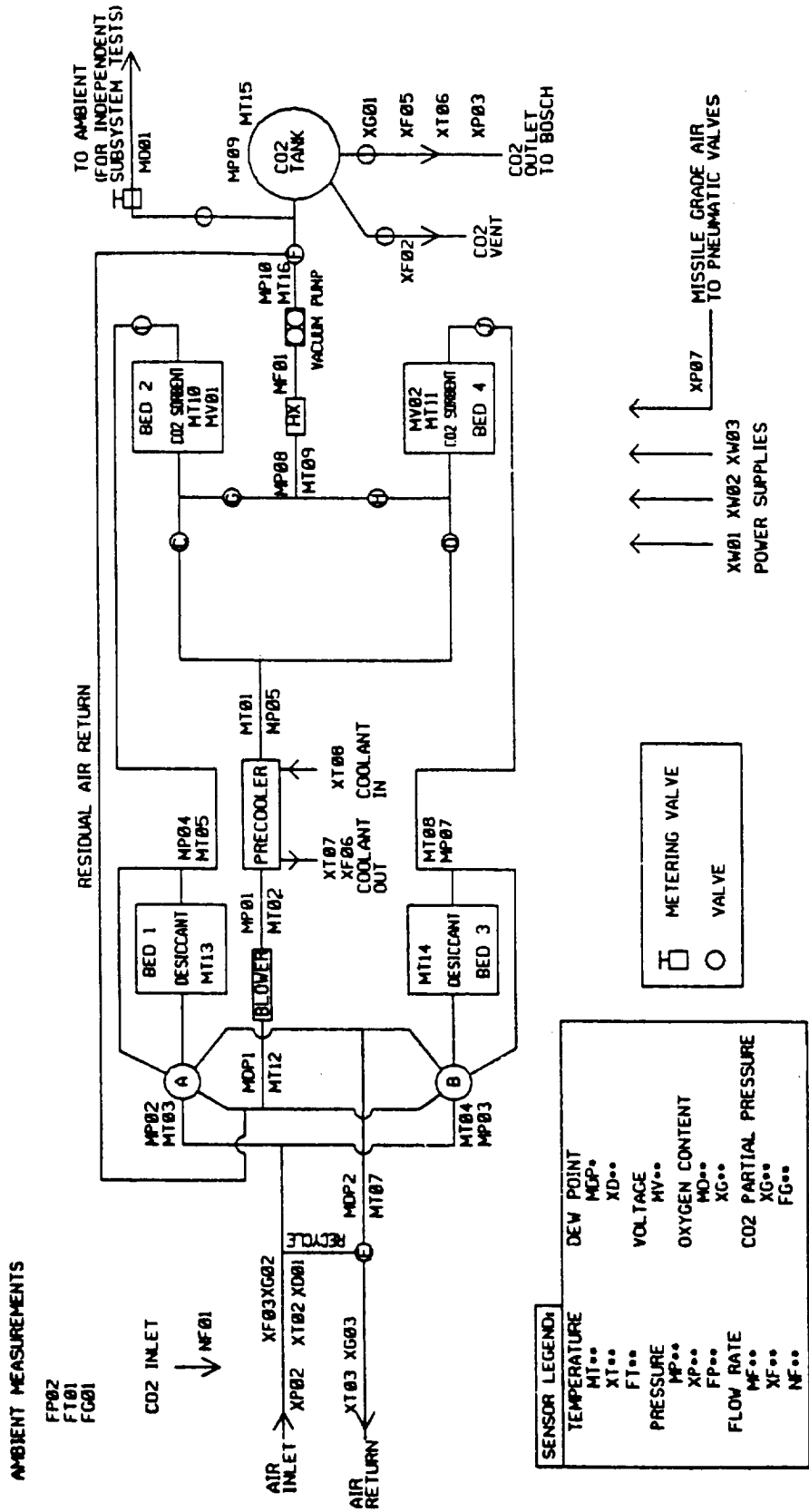
Figure 9. 4BMS schematic.

13

FOUR BED MOLECULAR SIEVE

| MINUTES | 0 | 7 | 11 | 55 | 62 | 66 | 110 |
|---|---|---|---|---|---|---|---|
| CYCLE MODE | 1A | 1B | 2 | 3A | 3B | 4 | |

DESICCANT

| | BED 1 | ←——— ADSORB ———→ | ←——— DESORB ———→ |
| | BED 3 | ←——— DESORB ———→ | ←——— ADSORB ———→ |

CO2 SORBENT

| | BED 2 | △ ← DESORB ———→ | ←——— ADSORB ———→ |
| | HEATER | ON | ON RECYCLE | ON | OFF | OFF | OFF |
| | BED 4 | ←——— ADSORB ———→ | △ ← DESORB ———→ |
| | HEATER | OFF | OFF | OFF | ON RECYCLE | ON | ON |

| COMPONENT DESCRIPTION | POSITION | | | | | |
|---|---|---|---|---|---|---|
| VALVE A | 2 | 2 | 2 | 1 | 1 | 1 |
| VALVE B | 1 | 1 | 1 | 2 | 2 | 2 |
| VALVE C | 4 | 4 | 4 | 3 | 3 | 3 |
| VALVE D | 3 | 3 | 3 | 4 | 4 | 4 |
| VALVE E | 6 | 6 | 5 | 6 | 6 | 5 |
| VALVE F | 7 | 8 | 8 | 7 | 8 | 8 |
| VALVE G | 3 | 3 | 3 | 4 | 4 | 4 |
| VALVE H | 4 | 4 | 4 | 3 | 3 | 3 |
| VALVE I | 4 | 4 | 4 | 3 | 3 | 3 |
| VALVE J | 3 | 3 | 3 | 4 | 4 | 4 |
| CO2 COMPRESSOR | ON | ON | ON | ON | ON | ON |
| CO2 TANK | – | CHARGING TANK | | – | CHARGING TANK | |

| VALVE | POSITION | DESCRIPTION |
|---|---|---|
| A,B | 1 | DESICCANT BED TO AIR RETURN |
| A,B | 2 | INLET AIR TO DESICCANT BED THEN BLOWER |
| C,D,G,H,I,J | 3 | OPEN |
| C,D,G,H,I,J | 4 | CLOSED |
| E | 5 | OUTLET TO CABIN |
| E | 6 | RECYCLE OUTLET TO INLET |
| F | 7 | RESIDUAL AIR REMOVAL |
| F | 8 | CO2 TO HOLDING TANK |

△ = RESIDUAL AIR REMOVAL MODE

Figure 10. 4BMS operating modes.

14

The desorbed $CO_2$ was pumped to an accumulator from which the $CO_2$ could be regulated to the Bosch or vented. An $O_2$ sensor in the $CO_2$ outlet line was used to measure the percentage of $O_2$ in the $CO_2$ (an indication of the amount of air present in the $CO_2$). An operational safety constraint was imposed to assure 2 percent or less $O_2$ in the accumulator.

The two-way valves, recycle valve, and $CO_2$ outlet flow control valve are pneumatic-type and pressurized nitrogen was supplied to actuate them.

### 3.1.2  4BMS Performance Analysis

During the phase III SIT, the 4BMS operated for 235 h (including 148 h 39 min when it was integrated with the other AR subsystems). Air leakage in the subsystem caused the 4BMS to be periodically deintegrated from the other air revitalization subsystems for troubleshooting and leak repair.

During the SIT, several facility and internal subsystem sensors did not function properly. The loss of these measurements limits the ability to analyze the performance of the 4BMS. However, since later tests are to repeat this condition, the sensors were not replaced in the interest of beginning the following water recovery testing early. The average $CO_2$ removal efficiency was expected to be between 1.8 and 2.13 kg/day (3.96 and 4.69 lbm/day). While it can be determined that the 4BMS was removing $CO_2$ (because the inlet partial pressure of $CO_2$ was higher than the outlet), the actual $CO_2$ removal efficiency cannot be calculated because of the loss of several important sensors. This is discussed further in Section 3.1.3.

Some of the key measurements made during this test are shown in Figures 11 through 14. These are discussed below.

MP08 – Desorbing Bed 2 or 4 Presssure: the pressure in the desorbing $CO_2$ sorbent bed usually ranged from 1 to 2 psia. Figure 11 shows pressure spikes as high as 14 psia. These occurred during pumpdown portion of modes 1 and 3. Otherwise the pressure peaked early during modes 2 and 4 when the increasing temperature desorbed most of the $CO_2$.

XG01 – Figure 12 shows the steady increase in the $O_2$ percentage in the $CO_2$ outlet. The downward spikes indicate times when the $O_2$ content reached 2 percent. The $CO_2$ accumulator was then backfilled with pure facility $CO_2$ as discussed in Section 3.1.2.

XG02 – As seen in Figure 13, the inlet partial pressure ($pCO_2$) was maintained at 3 mmHg. This is the maximum 90-day operational $pCO_2$ for S.S. Freedom.

XG03 – The outlet $pCO_2$ ranged from about 1 to 1.5 mmHg (Fig. 14). This is higher than expected, since previous testing yielded outlet $pCO_2$ levels as low as 0.4 mmHg. The increased outlet $CO_2$ concentration was probably caused by leakage in the subsystem. This leakage may have caused the bed 2 or 4 inlet dew point to be higher than expected. The increased moisture in the air was adsorbed on the sorbent material, displacing some $CO_2$ that otherwise would have been adsorbed. This contributed to the 4BMS's degraded performance.
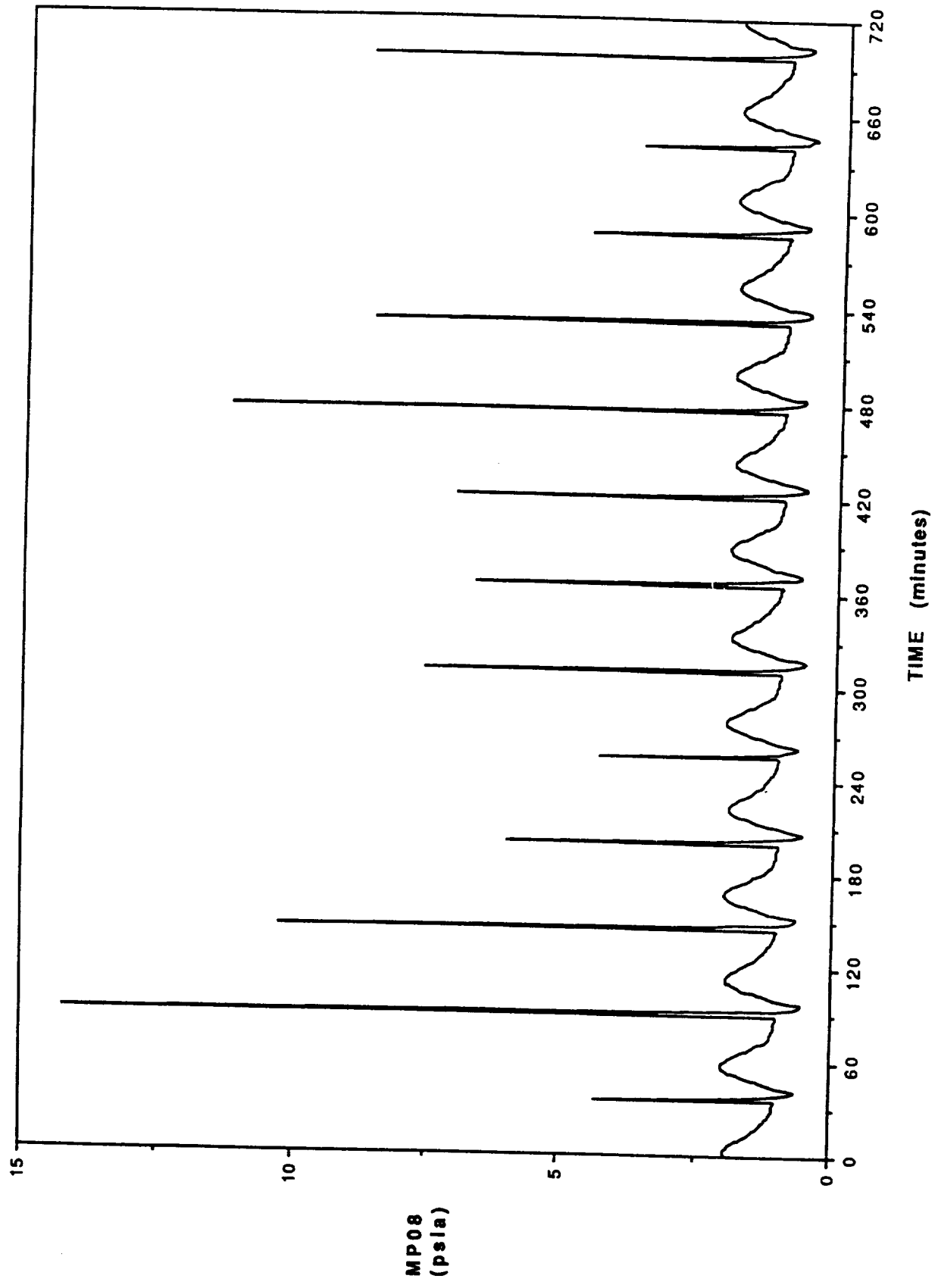
Figure 11. 4BMS sorbent bed 2 or 4 pressure (MP08) during day 5.

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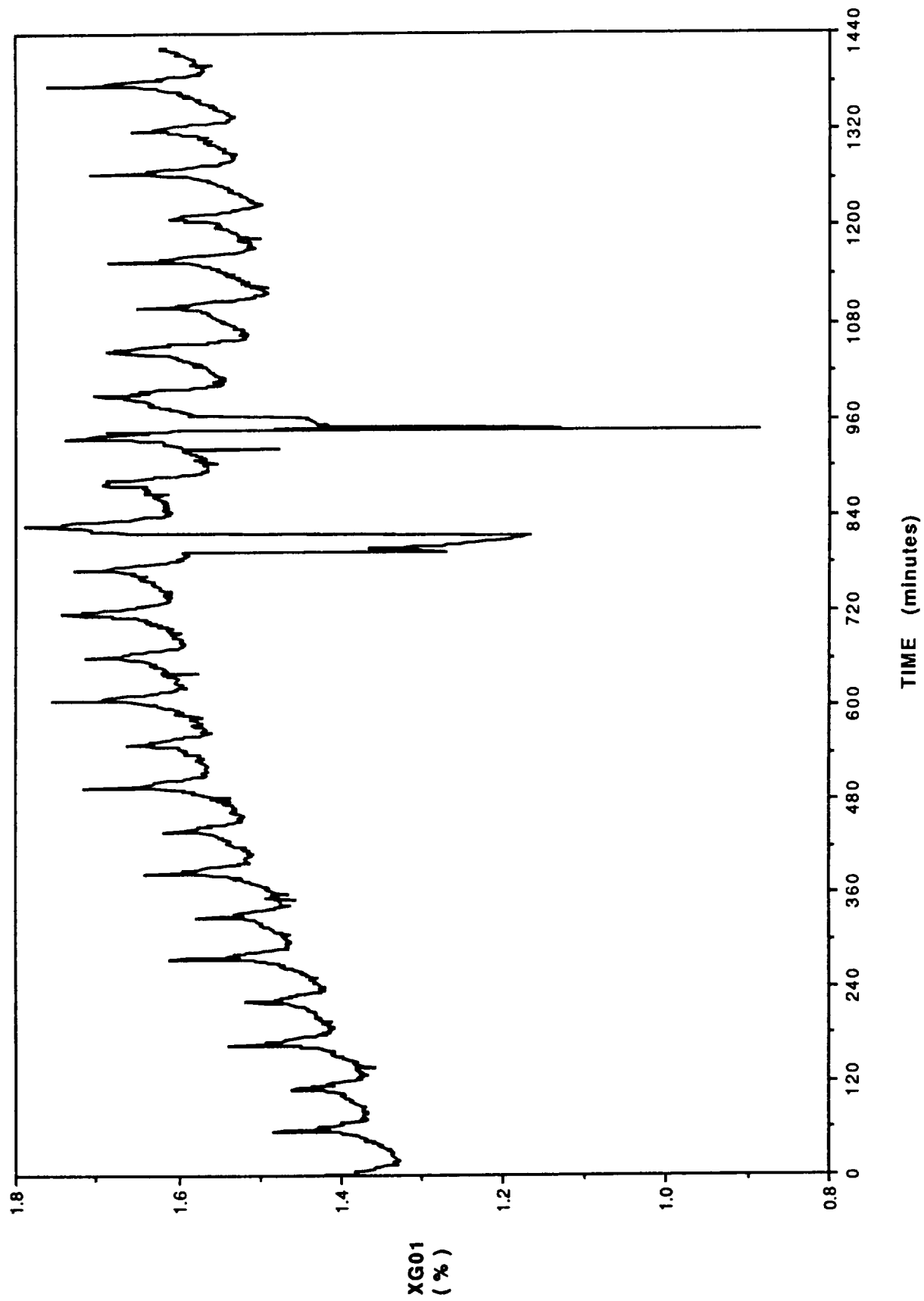
Figure 12. 4BMS percent $O_2$ in $CO_2$ product (XG01) during day 5.

17
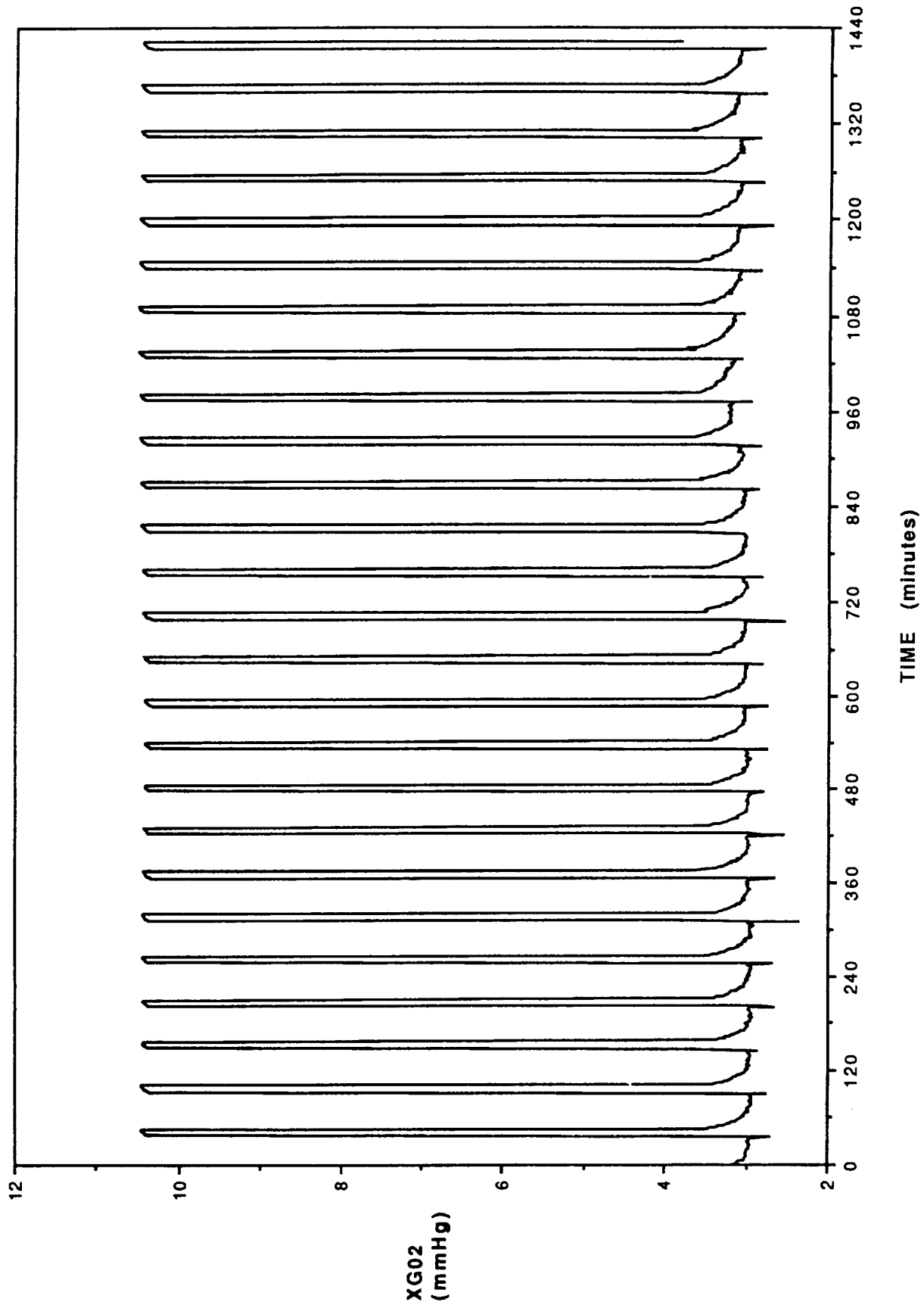
Figure 13. 4BMS inlet air partial pressure of $CO_2$ (XG02) during day 5.

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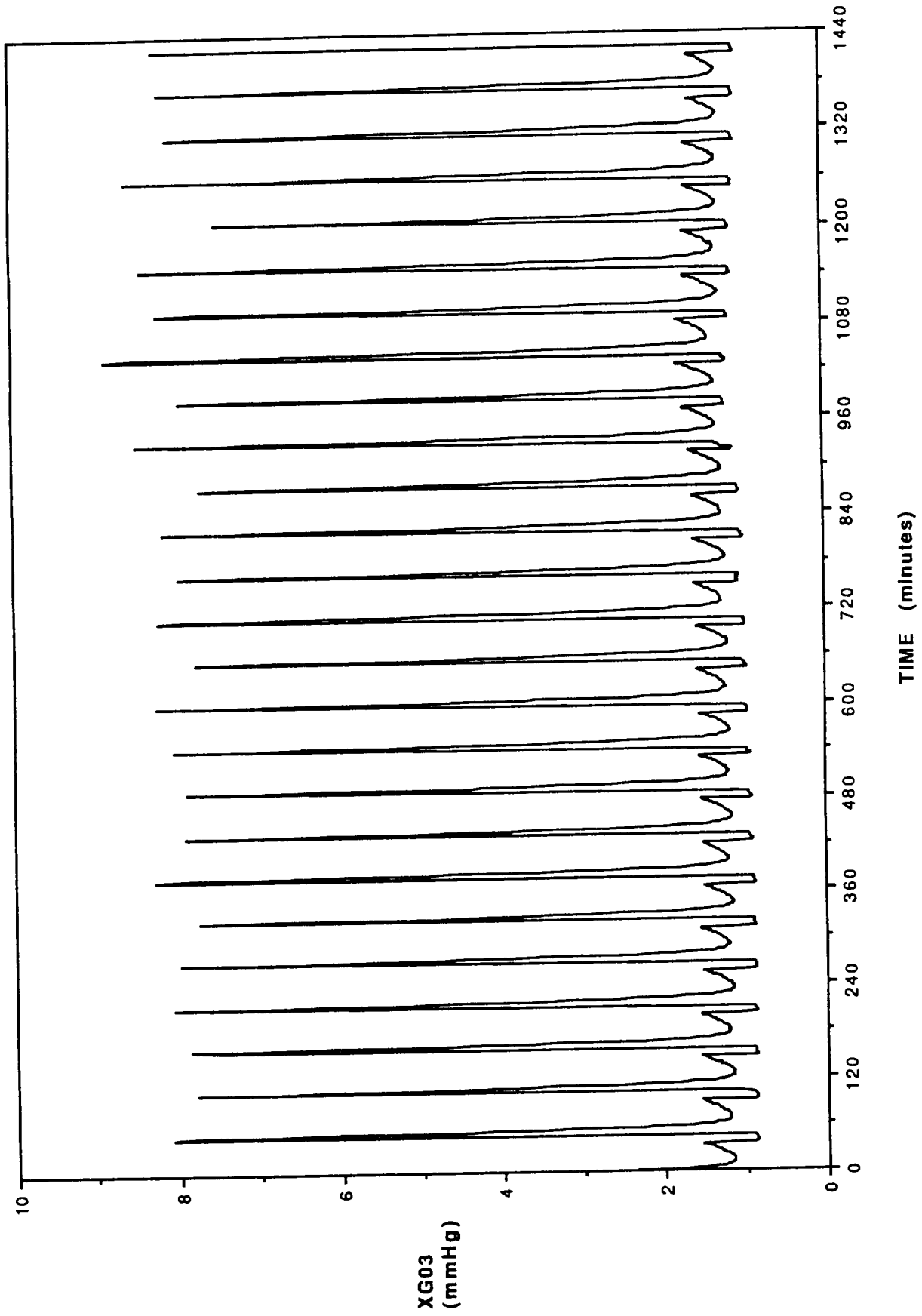
Figure 14. 4BMS outlet air partial pressure of $CO_2$ (XG03) during day 5.

19

### 3.1.3 Discussion of Results

The results of this test show that the 4BMS does perform its intended function during integrated operation. However, excessive air leakage at various locations resulted in lowered performance.

The composition of the outlet $CO_2$ was roughly 84-percent $CO_2$, 13-percent $N_2$, and 3-percent $O_2$, with no detectable quantities of any other compound. The outlet $CO_2$ concentration is expected to be greater than 97 percent. The increased air content was due to the subsystem leakage.

As mentioned earlier, it was discovered during the test that several facility and internal subsystem sensors were not functioning properly [6]. In order to calculate the subsystem's $CO_2$ removal rate, reliable measurements of inlet air flow (XF03), $CO_2$ injection flow rate (FF01), $CO_2$ outlet flow (XF05), and the $CO_2$ outlet pressure (XP03), among others, are necessary. Reliable values of these measurements were not available. An evaluation of the 4BMS's performance is not offered for this reason. Due to the uncertainties indicated here these tests will be repeated at a later date.

### 3.1.4 Recommendations/Lessons Learned

It is recommended that for future phase III testing, a complete leak test be performed on the subsystem prior to integrated operation. 4BMS leakage will be a critical design issue in the flight design, as such provisions should be made to helium leak check all connectors.

## 3.2 Static Feed Electrolyzer

### 3.2.1 Detailed Description

The SFE subsystem was used to generate oxygen at a three-man metabolic rate and hydrogen for use by the Bosch. A schematic of the SFE is shown in Figure 15. The SFE consists of five major components: (1) the electrolysis module, (2) the fluids control assembly (FCA), (3) the pressure control assembly (PCA), (4) the coolant control assembly (CCA), and (5) the feed water tank. A photograph of the SFE sitting inside the simulated module is shown in Figure 16.

The electrolysis of deionized facility water to oxygen and hydrogen takes place in the electrolysis module which consists of 12 cells stacked together between 2 insulation plates and 2 end plates. Each cell contains a water compartment, an oxygen compartment, and a hydrogen compartment. The water and hydrogen compartments are separated by a water feed membrane, while the two gas compartments are separated by the electrolyte matrix/electrode assembly. The electrolyte is aqueous potassium hydroxide (KOH).

The reactions which occur in the cells are:

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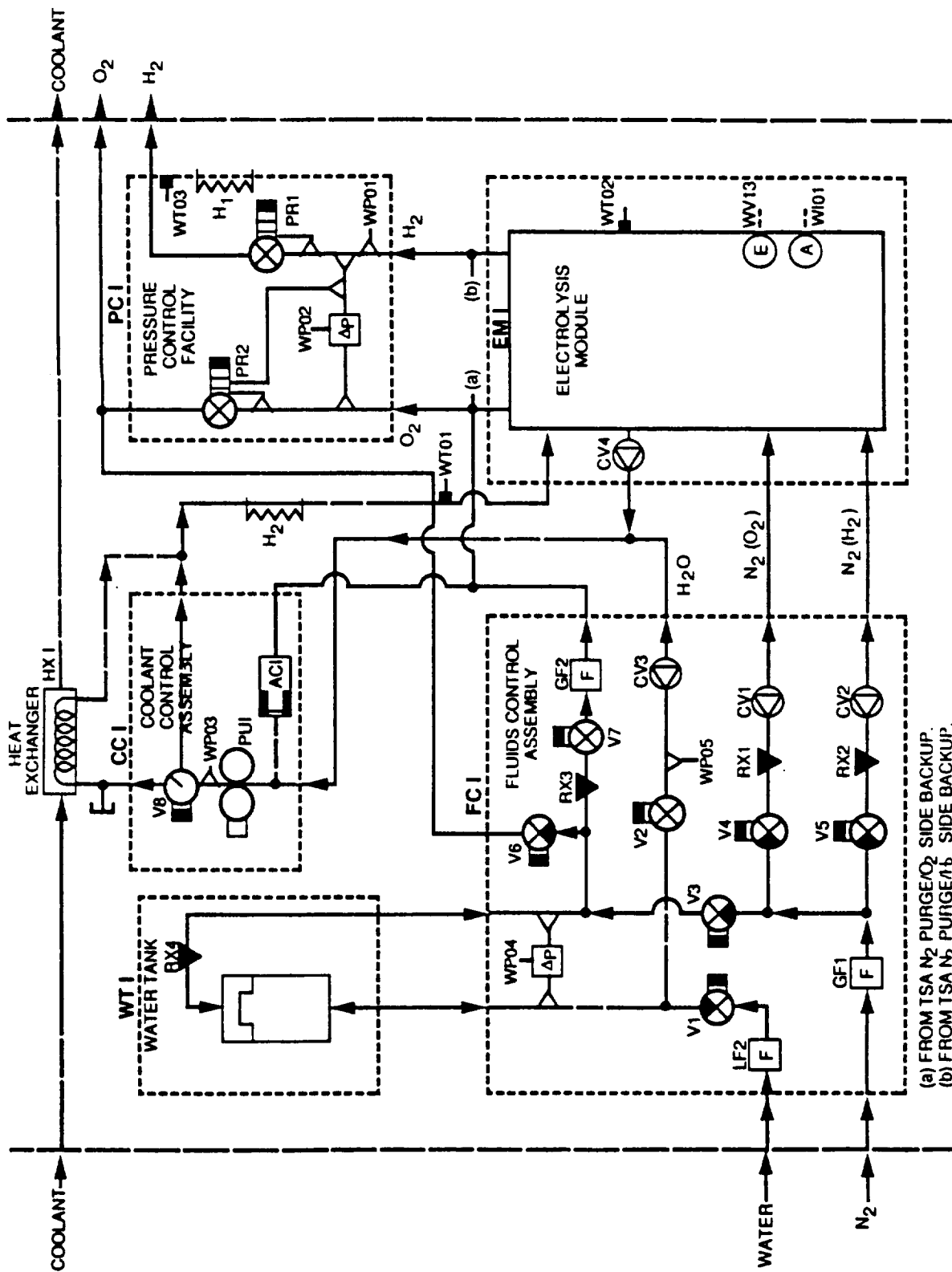
Figure 15. Static feed electrolyzer subsystem.

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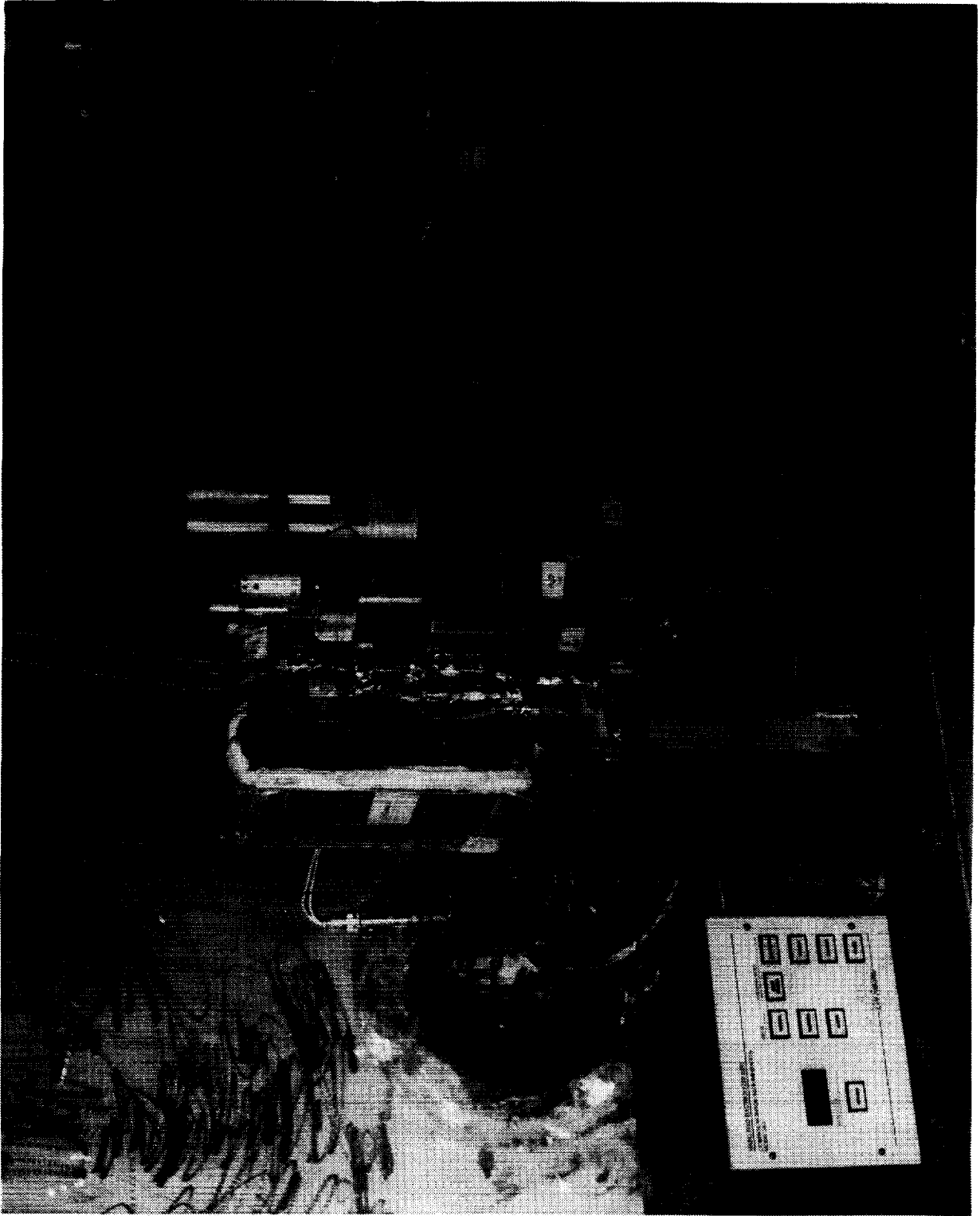
Figure 16. SFE subsystem.

Cathode:   $2e- + 2(H_2O) \rightarrow H_2 + 2(OH-)$

Anode:   $2(OH-) \rightarrow H_2O + 1/2(O_2) + 2e-$
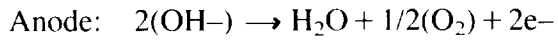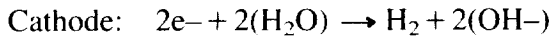
The resulting overall reaction is:

Electrical Energy $+ H_2O \rightarrow H_2 + 1/2(O_2) + Heat$.
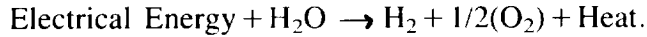
Before power is applied to the module, the water feed cavity and the electrolyte matrix contain equal concentrations of KOH electrolyte. As power is applied to the electrodes, water is electrolyzed from the electrolyte matrix resulting in a KOH concentration increase and a water vapor pressure decrease in the matrix. As the water pressure in the electrolyte matrix drops below that in the water feed cavity, water diffuses from the water feed cavity through the hydrogen cavity into the electrolyte matrix in an attempt to reestablish the initial equilibrium. As water diffuses from the water feed cavity, it is statically replenished from the feed water tank. The processes of electrolysis, diffusion, and the static replenishment of feed water occur continually as long as power is applied to the cell electrodes. The electrolysis module is equipped with the voltage, current, and temperature sensors required to monitor its performance.

The FCA consists of seven valves which are mounted on two motor-driven cams. The cams are driven to the required positions to open and close the valves to control the purge gas and feed water flows and water tank fills. During normal mode operations, valves V2 and V7 (Fig. 16) are open. V2 permits the flow of water from the feed water tank, WT1, while V7 allows the air side of the tank to be pressurized with product oxygen. The water tank is refilled every three hours. During the tank fill sequence, V2 and V7 are closed. V6 opens briefly to vent the air side of the tank to ambient pressure, and V1 opens to refill the tank from an external water supply. When V6 and V1 close, V3 opens and facility nitrogen flows in to repressurize the tank. Upon completion of the fill sequence, V3 closes and V2 and V7 reopen. The time required for a tank fill is approximately 4 to 5 min. During this fill time, the SFE continues to generate oxygen and hydrogen at the required rates. A nitrogen purge is included in the startup and shutdown sequences. During the purge, V4 and V5 open to allow nitrogen flow through the subsystem oxygen and hydrogen passages. The FCA is instrumented with valve position indicators and pressure sensors necessary for monitoring its operation.

The PCA consist of two motor-driven regulators, an absolute pressure sensor, and a differential pressure sensor. Regulator PR1 controls the hydrogen production pressure, while PR2 controls the oxygen to hydrogen differential pressure. PR1 and PR2 also control the pressurization and depressurization of the SFE during startups and shutdowns. The PCA is equipped with feedback valve position indicators which, with the pressure and differential pressure sensors, provide for monitoring of its operation.

The CCA consists of a motor, a pump, an accumulator, and a motor-operated diverter valve. The diverter valve controls the ratio of flow through the heat exchanger HX1 to flow

through the bypass in order to control the temperature of the feed water. The accumulator AC1 accommodates thermal expansion and contraction of the feed water. The CCA is instrumented with pressure and temperature sensors and the valve position indicator required to monitor its performance.

The WT1 is a metal bellows tank which supplies static replenishment to the electrolysis module. The tank is refilled every three hours as previously explained. The sensors which provide for monitoring of the tank are included in the FCA.

The startup, shutdown, and other mode transition sequences are software controlled. Any out of range sensor reading will cause an automatic shutdown of the subsystem.

## 3.2.2  SFE Performance Analysis

The SFE-III was delivered to MSFC through Boeing Aerospace in the fall of 1986 and was set up for phase II testing which began in spring of 1987. Over a period of approximately 5 months, a total of 248 h of testing was completed. Following the phase II testing, the SFE-III was integrated into the phase III test hardware.

During the phase III testing, the SFE-III ran for a total of 266 h in the normal mode and experienced only one shutdown. The SFE was integrated with the Bosch-II for a total of 257 h, or approximately 97 percent of its operating time. During that time, the SFE performed its function of delivering oxygen to the atmosphere and hydrogen to the Bosch $CO_2$ reduction subsystem. The paragraphs below analyze specific experiences during that test time.

During the first day of testing it was noticed that the condensate trap in the SFE $H_2$ line was approximately one-third full of liquid. The trap had been checked and found empty during the startup procedure. It is not known when or how the liquid was expelled from the cell stack. Since the subsystem sensors indicated that the SFE was functioning normally, it was decided to continue the test. The trap was subsequently drained, and the liquid was analyzed. It proved to be 24.9-percent KOH. A potential source of the liquid could be leaky feed cores which, in the SFE vintage design, were constructed with an epoxy prepreg edge seal. This type of seal has been known to degrade during exposure to electrolyte with time (usually over a period of several years). A new feed core design is now used on all subsequent electrolyzers. This core uses a new edge seal which is resistant to the electrolyte.

The SFE is integrated with the Bosch by configuring a three-way valve, EVA03, to send the SFE product hydrogen to the Bosch hydrogen inlet rather than to the vent line. During initial attempts to integrate the two subsystems, when EVA03 was switched to its integrated position, the hydrogen flow dropped below zero, as measured by flow sensor EIF03. At the same time the hydrogen concentration in the SFE hood, measured by EIG01, increased. Leak check of the line between the SFE and the Bosch showed that the dew point sensor connection was loose. Once this connector was tightened, the SFE and the Bosch were successfully integrated.

24

During the fourth day of testing the SFE was inadvertently shutdown due to facility work which was not related to the SIT. When subsystem power was restored, the SFE transitioned to the shutdown mode which is normal procedure. During the restart, the transition to normal mode was nominal and no liquid was accumulated in the $H_2$ condensate trap.

For the first few days of testing, the SFE cell voltages ran slightly higher than expected, though they never reached the warning level. High voltages generally indicate that the cells are being dried out. This can be prevented by increasing the cell stack operating temperature in order to increase the rate of diffusion of feed water to the cell matrices. On the fifth day, the temperature set point for the water inlet to the cell stack was increased from $141 \pm 2$ °F to $143 \pm 2$ °F. This served to reduce the cell voltages to nominal levels. However, increasing the ETT1 set point also caused the oxygen outlet temperature, ETT2, to increase so that it repeatedly bounced in and out of its warning range. This was an intermittent nuisance for the remainder of the test. This nuisance could have been corrected by making a set point change for T2. The high warning and high alarm on T2 should have been increased in conjunction with the increase of the T1 control points. However, since ETT2 remained well below the alarm level, the SFE performance was not affected.

### 3.2.3  Discussion of Results

All subsystem measurements indicated that the SFE was performing nominally at a three-man level throughout the SIT. However, the three-man operating level was not verified by the integrated system/subsystem instrumentation downstream of the SFE or by the water intake measurement.

The weight of the facility deionized water tank was recorded after each subsystem tank fill. Subsystem tank fills occurred automatically at 3.06-h intervals. The SFE water intake was averaged over 84 such intervals to yield a water consumption rate of 2.766 kg/day (6.097 lbm/day). The water content of the product oxygen and hydrogen was approximately 0.0095 and 0.01 kg/day (0.021 and 0.022 lbm/day), respectively, as indicated by the sample analyses. The water electrolysis rate, therefore, was approximately 2.75 kg/day (6.054 lbm/day) which is 97.5 percent of the three-man rate.

The average flow in the oxygen outlet line was approximately 1.165 slpm as indicated by EIF01. Adjusting for the water and hydrogen content of the oxygen stream yields an oxygen production rate of 2.206 kg/day (4.853 lbm/day). This is 87.9 percent of the three-man rate or 90.2 percent of the rate which would correspond to the water consumption rate. Similar calculations for the hydrogen stream indicate a hydrogen production rate of 0.246 kg/day (0.541 lbm/day) which is only 78.4 percent of the three-man rate and only 80.4 percent of the amount indicated by the water consumption rate.

The hydrogen sensor in the SFE hood showed a positive reading for most of the test. This indicates a possible hydrogen leak which would account for some of the discrepancy in the inlet and outlet flow measurements. The leak is most likely from interconnecting lines between the SFE and the Bosch because the SFE subsystem had been successfully helium leak checked to a level of $1 \times 10^{-7}$ cc/s. It is not possible to quantify the leakage.

Other possible sources of inaccuracies include flow sensor calibration, interpretation of flow sensor output, and measurement of the water content of the product gases. In any event, an accurate mass balance cannot be done with the available data (Fig. 17). This will be performed in future tests.

### 3.2.4 Recommendations/Lessons Learned

Prior to the SIT, the SFE was last tested in the phase II metabolic control test (MCT) in 1987. During that test, recovered hygiene water was used as SFE feedstock. Following the MCT, the SFE, still containing the poor quality hygiene water, was stored for 2 years. No precautions were taken to prepare the SFE for this long period of storage. It is possible that this storage period and the out-of-limits water quality adversely affected the SFE voltage during the SIT, thus requiring a slight increase in operating temperature. Appropriate post-test procedures should be developed for flushing the SFE should it be tested with out-of-limits hygiene or other waters.

In order to determine actual subsystem performance, it is necessary to do an accurate mass balance. This cannot be done without accurate flow measurements and accurate sample analyses. For future testing it is imperative that flow sensors be accurately calibrated and that their output be correctly interpreted. Greater care must also be taken to assure reliable sample analyses. Future tests are planned in which the errors will be remedied.

A thorough leak check of the subsystem, as well as of the facility, should be performed prior to testing. Removal from or addition to the subsystems or facility of any component should warrant a repeat leak check.

### 3.3 Bosch Carbon Dioxide Reduction Subsystem

### 3.3.1 Detailed Description

The Bosch II, shown in Figure 18 as it was situated inside the CMS and schematically in Figure 19, is the second Bosch built by Life Systems Inc. and was designed to reduce an equivalent of a three-man rate of metabolic $CO_2$ production, or 3.0 kg/day (6.6 lbm/day). $CO_2$ is fed to the subsystem where it is mixed with recycled gas and pumped downstream by a variable-speed compressor (C01). A slipstream of the resulting gas mixture is directed through a gas composition controller (CG1) which measures and compares its thermal conductivity to a set point and then meters in the appropriate amount of feed hydrogen through a variable orifice valve. Excess feed hydrogen is vented through an internal relief valve (RV2). Downstream of the compressor, two 4-way valves (V5 and V6) direct the gas flow to one of the two identical reactors. Upon entering the reactor assembly, the gas is preheated through a regenerative heat exchanger, then flows through the packed bed of iron (steel wool) catalyst. At a 922 K (1,200 °F) bed temperature, the $CO_2$ and $H_2$ are converted to solid carbon (which is deposited on the iron catalyst) and water vapor. Figure 20 is a photo of the typical solid carbon product. Approximately 6 percent of the reactants are converted per pass, requiring around a 15:1 recycle ratio for complete conversion. The reaction products, consisting of unreacted $CO_2$ and $H_2$, intermediate and side reaction

26

Figure 17. SFE mass balance.

H2O in = 2.765 kg/day (6.097 lb/day)

SFE

LEAKAGE = ?

O2 = 2.200 kg/day (4.852 lb/day)
H2O = 0.010 kg/day (0.021 lb/day)
H2 = 0.001 kg/day (0.001 lb/day)

LEAKAGE = ?

H2 = 0.245 kg/day (0.541 lb/day)
H2O = 0.010 kg/day (0.022 lb/day)
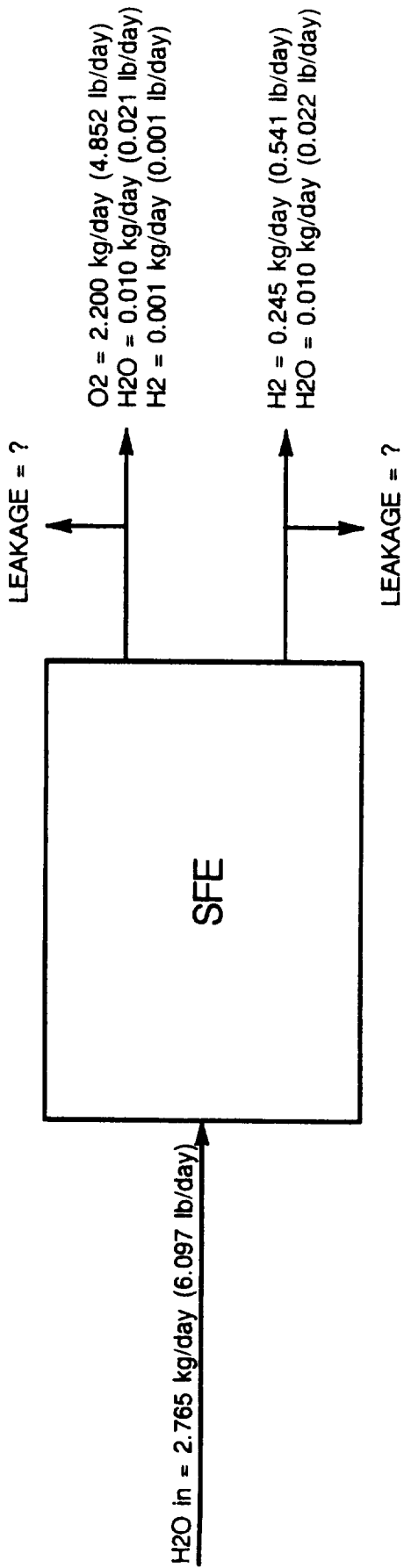
NOTE: ONLY 89% OF THE INLET FLOW
CAN BE ACCOUNTED FOR IN
THE OUTLET FLOW.
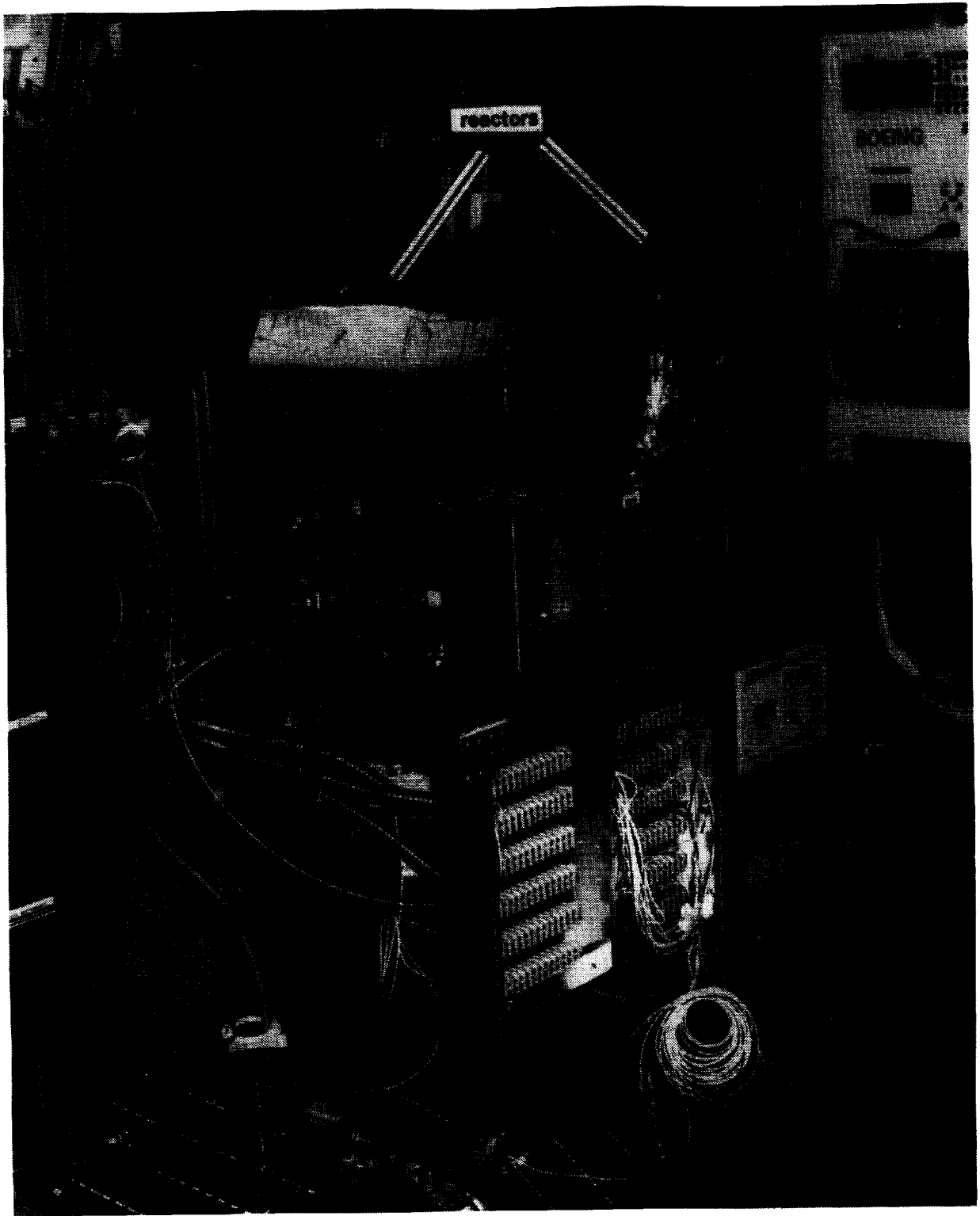
Figure 18. Bosch subsystem.

28
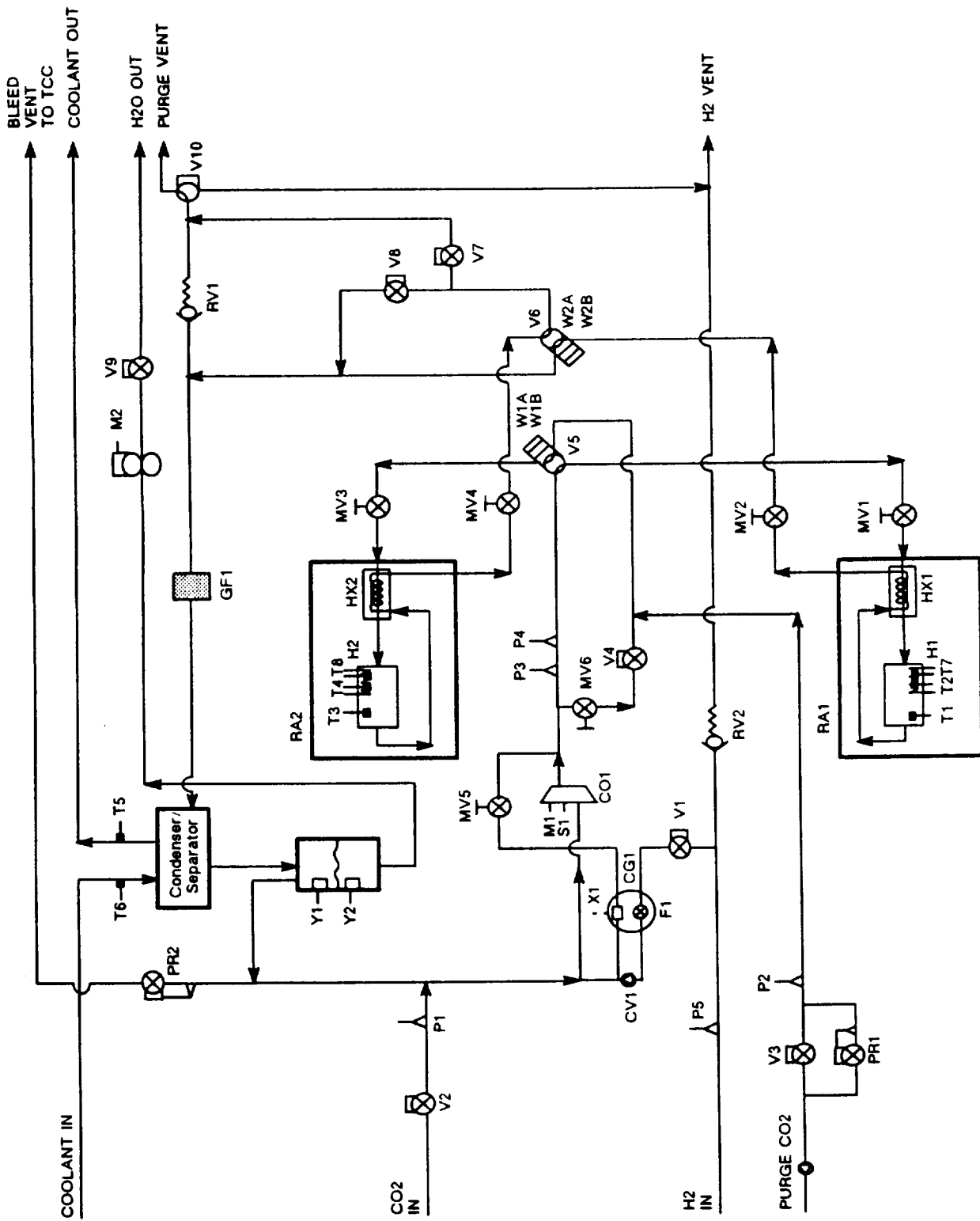
Figure 19. Bosch II subsystem schematic.

29
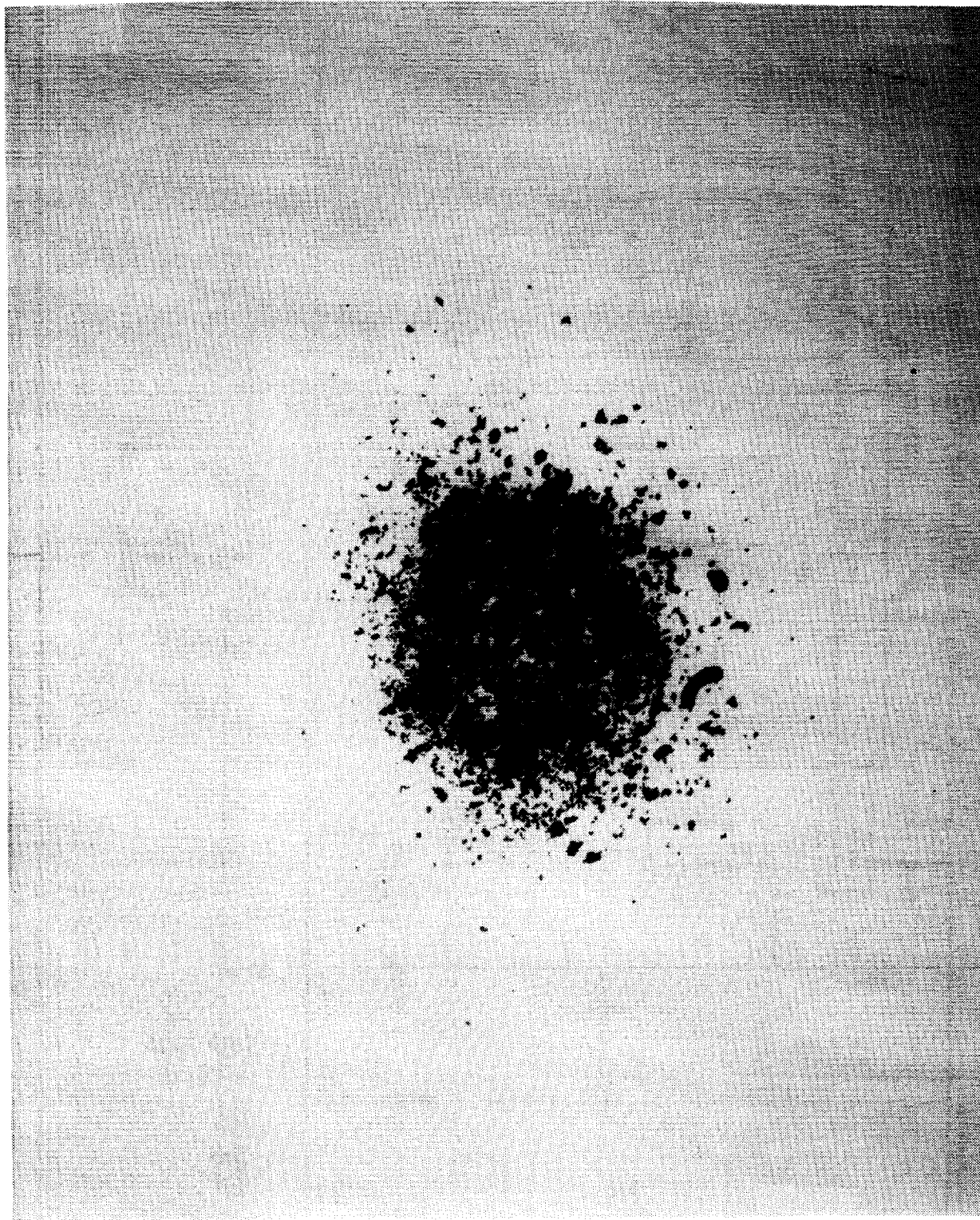
Figure 20. Bosch carbon product.

products CO and $CH_4$, product water vapor, and contaminant $N_2$, exit the reactor assembly through the regenerative heat exchanger to the liquid-cooled condenser/separator. Condensed product water collects in an accumulator which is periodically emptied through valve V9. Two liquid level sensors (Y1 and Y2) indicate when the accumulator is full or empty and signal the pump motor (M2) to turn on or off. If the tank does not reach the full level within every hour, a water dump will automatically be initiated. The remaining dry gas mixture is recycled and mixed with fresh inlet reactants. Typically, inlet $CO_2$ from the $CO_2$ removal subsystem will contain a percentage of contaminant air. Because the inert nitrogen does not react, it causes a pressure buildup in the Bosch. As a result, it is necessary to bleed off a portion of the recycle mixture through a back pressure regulator (PR2) set at the desired maximum reactor inlet pressure. When the reactor cartridge becomes full of carbon as indicated by differential pressure, between P1 and P3, the subsystem automatically switches over to the alternate reactor. The full cartridge is removed from the reactor assembly once cooled (Fig. 21), and a fresh one installed. Facility $CO_2$ is used to purge the reactors prior to startup and following switchover or shutdown.

## 3.3.2 Bosch Performance Analysis

The Bosch II was delivered to MSFC through Boeing Aerospace in the fall of 1987 and was set up for independent bench testing. Approximately 1,300 h of testing over a 20-month period was completed on the unit prior to the SIT. Several full cartridge runs were performed with pure reactants and different man rates of feed. The unit was then modified to accommodate testing with impure $CO_2$ (containing $N_2$ from the 4BMS) by adding a bleed vent along with a back pressure regulator and modifying the appropriate control set points. Additional testing following the modification included runs with various levels of $N_2$ mixed with the reactant $CO_2$. A total of 37 anomalies associated with the subsystem were logged during the bench testing. These anomalies included numerous heater burn-outs; failures of thermocouples and other sensors; electrical noise problems resulting in random valve movement; failures of controller cards; leakage from the compressor, reactors, fittings, and valves; a compressor coupling mechanical failure; and numerous lessons learned regarding proper control sequencing and software, particularly in regard to implementing the modifications to accommodate inerts in the $CO_2$ feed. All of the anomalies applicable to the Bosch III comparative test unit have been tracked and some design changes made to ensure that the same problems would not recur on the development and flight unit. All of the anomalies except four were resolved prior to the SIT. These resolutions resulted in many improvements to the Bosch II, including electrical, heater, software, and reactor seal surface redesigns.

In addition to serving to develop the Bosch hardware technology, the independent testing that was performed supplied valuable performance data, especially in the areas of transients over a full cartridge and operation with inerts. For instance, it was discovered that the maximum rate of $CO_2$ reduction declines over the life of a cartridge as carbon is deposited on the catalyst. Also, data gathered on cartridge delta pressure trends was used to determine the appropriate switchover point. Bleed gas flowrate and composition were measured and used to provide preliminary estimates of the resulting load to the trace contaminant control subsystem catalytic oxidizer. Finally, data from analyses of Bosch product water samples generated during testing with inerts showed the presence of relatively high concentrations of ammonia, however, a correlation between the amount of ammonia produced given a percentage $N_2$ and $CO_2$ has not been established. Where possible, such data as discussed has been factored into the ongoing development of an analytical model for the Bosch and used in system impact studies.

Figure 21. Bosch cartridge.

As previously mentioned, there were four open anomalies on the Bosch going into the SIT. These were as follows:

(1) Random valve movement, presumably due to electrical noise. Although redesign of the four-way valve circuitry now prevents these valves from moving inadvertently, the two-way valves are still susceptible to noise voltage. Since actuation is rapid there have been no noted adverse effects from these slight movements, which is why it was decided to proceed into the SIT with this anomaly still open.

(2) Compressor stills. Short "hiccups" in compressor operation have been evident as long as the valve clicking. More recently, complete stalls have occurred requiring manual intervention to correct. It is not known whether the short "hiccups" are related to the longer stalls. This anomaly was also not considered a "show stopper" going into the SIT since it was felt it could be corrected if it occurred.

(3) Purge pressure creeping up into alarm levels during normal processing mode. This anomaly occurred during the independent/performance test run on the Bosch just prior to the SIT. To get through the test a fitting had to be cracked periodically to relieve pressure accumulating in the purge line. Following the test troubleshooting was performed to see if the inlet purge valve V3 was leaking through. It apparently was not, and it was decided to proceed into the SIT after adding a hand valve in the purge line to relieve pressure if necessary. This will be investigated further in future tests.

(4) High methane production from the Bosch reaction. Gas samples of the bleed taken during the independent/performance test showed a 50-percent concentration of methane, which is much higher than expected. Prior to the test, the reactor heaters had been redesigned to accommodate thicker thermocouples in hopes of resolving the ongoing problem of thermocouples failing in the high temperature environment of the reactors. Since the new thermocouples are imbedded within the heaters instead of laid along the outside surface, they now read a higher temperature than before and thus control the heater to a now lower actual temperature. The resulting bed temperatures read during the independent/performance test were 150 to 200 $\pm$ F lower than in previous testing. It is suspected that the lower temperature favors the exothermic side reaction more than before, thereby producing more methane. There was also a discrepancy between the amounts of carbon and water produced which also can be attributed to the lower reaction temperature and explained by carbon going to methane via the side reaction $C + 2H_2 = CH_4$. Prior to the SIT, insulation was added around the outside reactor canisters in hopes of reducing heat loss and raising the bed temperatures and reducing the amount of methane produced. Plans are to perform further tests, prior to the next integrated test, to assure proper reaction temperatures are achieved.

Of the four open anomalies going into the SIT, two were not repeated during the test. No compressor stalls were noted. It is possible that the compressor "hiccups" may have occurred, but since personnel were only in the test chamber for short periods of time, they may have not been noticed. Secondly, the purge pressure anomaly did not recur. No increases in the purge line pressure during normal processing were recorded.

Two of the previous anomalies were still evident in the SIT. Audible valve clicking was noted by personnel in the chamber throughout the test. Again, subsystem operation was apparently

unaffected. Also, although the added reactor insulation served to raise the bed temperature by approximately 40 °F, there was still a high methane percentage in the bleed gas (45 percent) and a discrepancy between the carbon and water production rates. One additional anomaly is being tracked as a result of a post-test leak check. It was discovered that the socket head cap screws which seal the reactor back flange were loose following the test. It is not known at what point during the test they became loose, but it is probable that there was some leakage of recycle loop gases as a result. This problem has occurred in prior bench testing. It is suspected that the cause is thermal expansion due to different materials used for the flange and the screws rather than a vibration problem. Solutions to the problem are presently in work.

### 3.3.3  Discussion of Results

The Bosch subsystem ran a total of 273 h in normal mode during the test with no shutdowns. It ran integrated with the 4BMS and SFE subsystems for 154 h, or approximately 56 percent of its operating time. This test represented the longest continuous run on the Bosch II as well as the first time it was integrated with other air revitalization subsystems.

Processing was started on Bosch reactor No. 1 and the subsystem was run independently off facility reactant gases until it was integrated with the SFE after 6 h. Following integration it was evident that there was a severe hydrogen leak between the two subsystems as the inlet $H_2$ flowrate to the Bosch dropped from 2,700 ccm to 1,000 ccm. The source of the leak was not found until the following morning at the hydrogen inlet to the gas composition controller within the Bosch. After tightening the loose fitting causing the leak, the $H_2$ inlet flowrate read about 2,200 ccm, or about 2.8-man rate. Because of the system instrumentation problem, integration of the Bosch with the 4BMS was not attempted for another 26 h after this point.

Upon integrating the 4BMS with the Bosch, it became apparent that there was insufficient regulation between the two subsystems. Too much initial $CO_2$ flow from the accumulator caused internal pressures within the Bosch to rise and sent it into an automatic reactor switchover. After 1 h, the 4BMS and Bosch were deintegrated and a metering valve was added in the interface to better regulate the $CO_2$ flowrate. The subsystems were then reintegrated and the flowrate adjusted to the maximum level that could be processed with the available $H_2$ that the Bosch was receiving. Because it was found to be difficult to maintain a constant flowrate when the upstream $CO_2$ accumulator pressure was cycling, a pressure regulator was also added just upstream of the metering valve after the Bosch had completed its reactor switchover. At that point, it was decided that the test would be continued until the new reactor cartridge was full of carbon and switchover back to reactor No. 1 had been completed. This was expected to take another five days at a three-man rate of operation. It actually took an additional eight days because only a two-man rate of $CO_2$ reduction was achieved. This will be discussed in more detail in a later section. The Bosch ran the remainder of the test integrated with the SFE except for one electrolyzer shutdown that lasted about 5 h. Integration with the 4BMS was intermittent for the remainder of the test because of air leakage problems with that subsystem which caused the percent $O_2$ in the $CO_2$ to rise to an unacceptable level (less than 2 percent). The first bleed flow from the Bosch began approximately 14 h after switchover and was routed to the TCCS. The bleed was diverted to vent a number of times during the remainder of the test because the unexpectedly high amount of methane in it was causing the TCCS catalytic oxidizer temperature to rise above desired levels. Samples of the Bosch bleed were taken three times during the test.

34

Because of the discrepancy between the total amounts of water and carbon produced by the Bosch reaction, it was difficult to tell when the reactor cartridge should have been full and switchover initiated. The reactor differential pressure which signals switchover at 10 psid became somewhat erratic and rose slightly, but not enough to trigger a switchover. Finally, because it was believed from the water production that the carbon cartridge should have been overfilled, the delta pressure set point was lowered and the compressor speed raised to force initiation of switchover. After a successful switchover the subsystem was shutdown.

Figure 22 gives an overall mass balance around the Bosch subsystem for the test. The flowrates, shown in kg/day, are averages over the length of operation on the second reactor cartridge.

Because there was no good instrument from which its flowrate could be determined, the $CO_2$ flow into the Bosch during the test was unknown. The $H_2$ inlet flowrate was measured by two sources. The facility inlet $H_2$ flowmeter, WF03, averages 0.258 kg/day (0.569 lbm/day) (converted from a volumetric reading in slpm). In addition, the subsystem also has an internal $H_2$ inlet flowmeter. This flowrate in past testing has usually matched well with $CO_2$ fed and products formed. For the test it averages 0.229 kg/day (0.505 lbm/day), or a 2.5-man level. Figure 23 compares these two as-read flow readings over an excerpted time slice of the test. The changes in each reading are consistent with each other, although their absolute values are somewhat different. The difference may have been due either to a leak between the sensors or simply error associated with one or both readings. There may also have been a hydrogen leak upstream of WF03 as well, since the flowrate measured by both sensors was lower than the expected rate of 0.313 kg/day (0.69 lbm/day). Had this flowrate been achieved there would have been excess hydrogen, since the Bosch does not use all the $H_2$ generated when the electrolyzer produces $O_2$ at a three-man rate. Instead, the Bosch took in all that was available and the $CO_2$ flow had to be metered down accordingly.

The average carbon production rate was calculated by measuring the final weight of the cartridge, subtracting the initial weight, and dividing by the number of days of operation on reactor No. 2. The result was an average net deposition rate of 0.386 kg/day (0.85 lbm/day), or a 1.42-man level equivalent.

Figure 24 is a plot of water production for the test, determined by hourly volumetric measurements of water dumped. For reactor No. 2 only, the total water produced was divided by time to get an average rate of 1.63 kg/day (3.6 lbm/day), or a two-man level.

The flowrates of constituents in the bleed were determined by first estimating an average bleed flow for reactor No. 2 from a plot of flowmeter WF02. This was calculated as approximately 220 ccm. Next, an average bleed composition was calculated from the results of analyses of the three gas samples (Table 1) and normalized to a 100 percent total. These volume percentages were multiplied by the average total flowrate to get the volumetric flowrates of each constituent, which were then translated into the mass flows given in Figure 22. For the most part, the total bleed flowrate and percentages were consistent with expected performance. The exception is the high percentage of methane as previously discussed.

A complete mass balance cannot be performed around the Bosch since the inlet $CO_2$ rate was unknown. However, a balance around $H_2$ shows that the $H_2$ out in the product water and bleed
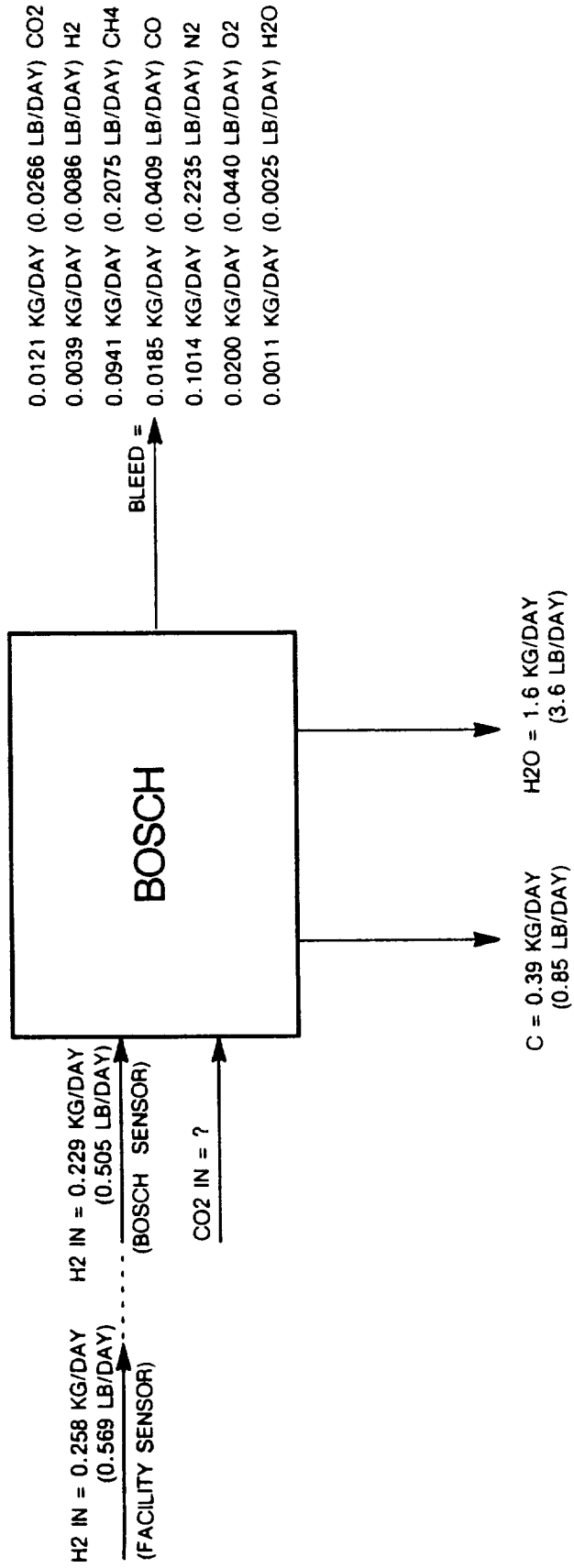
Figure 22. Bosch mass balance.

H2 IN = 0.258 KG/DAY
(0.569 LB/DAY)
(FACILITY SENSOR)

H2 IN = 0.229 KG/DAY
(0.505 LB/DAY)
(BOSCH SENSOR)

CO2 IN = ?

BOSCH

BLEED =

0.0121 KG/DAY (0.0266 LB/DAY) CO2
0.0039 KG/DAY (0.0086 LB/DAY) H2
0.0941 KG/DAY (0.2075 LB/DAY) CH4
0.0185 KG/DAY (0.0409 LB/DAY) CO
0.1014 KG/DAY (0.2235 LB/DAY) N2
0.0200 KG/DAY (0.0440 LB/DAY) O2
0.0011 KG/DAY (0.0025 LB/DAY) H2O

H2O = 1.6 KG/DAY
(3.6 LB/DAY)

C = 0.39 KG/DAY
(0.85 LB/DAY)

36
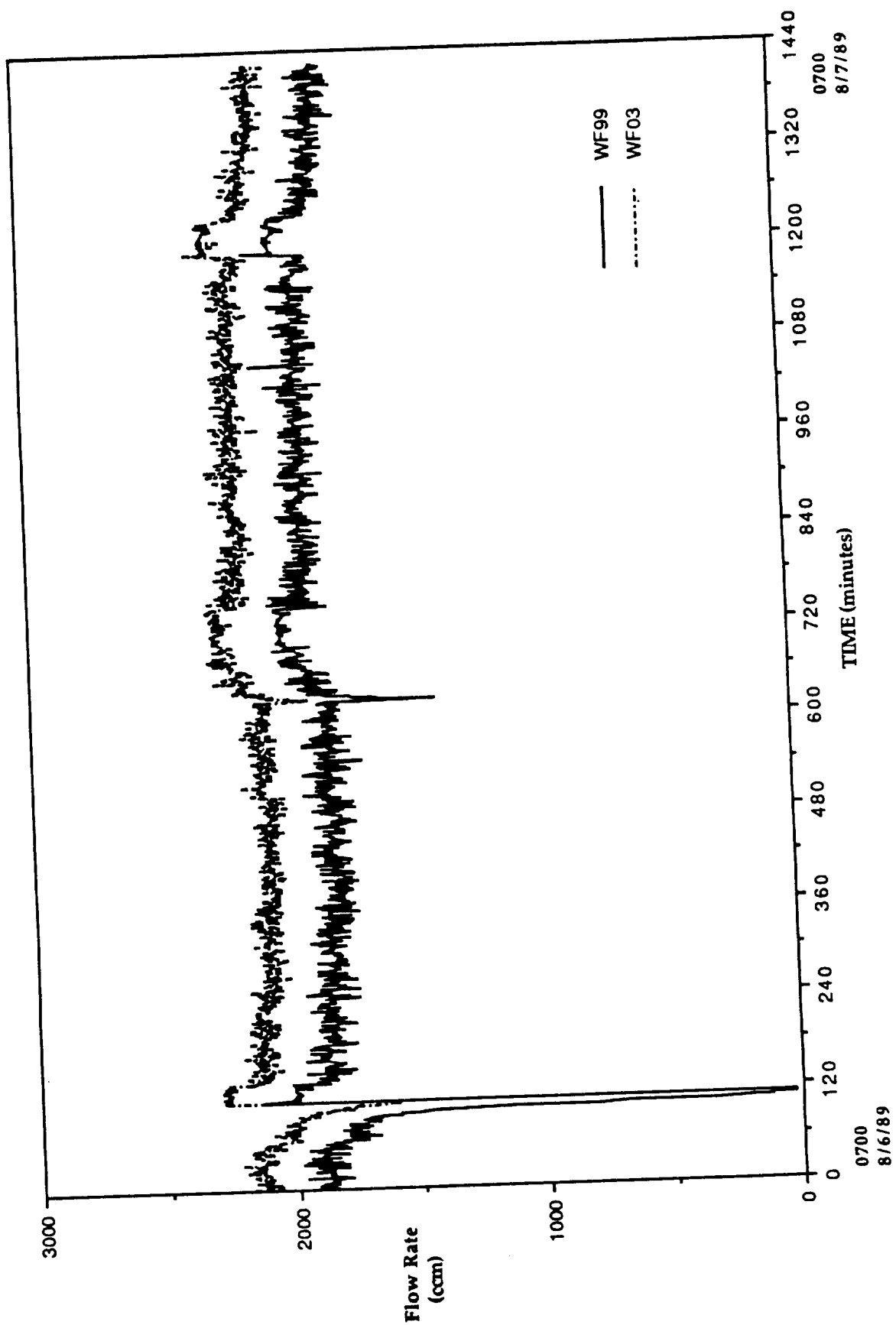
Figure 23. Bosch $H_2$ feed flow (WF99) and $H_2$ inlet flow (WF03).

37
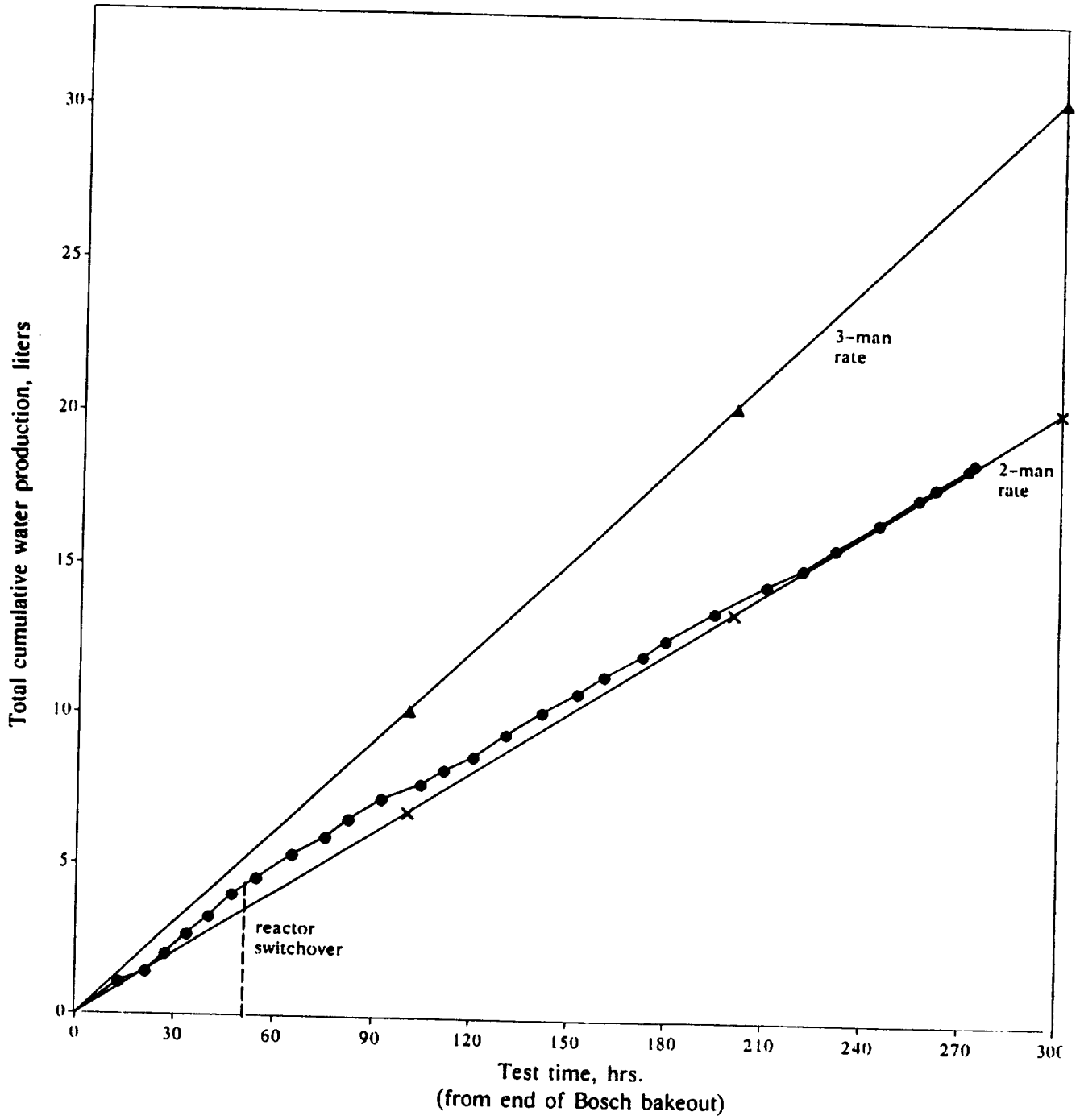
Figure 24. Bosch water production during SIT.

38

Table 1. Bosch bleed analysis.

| ANALYTES | UNITS | SAMPLED AUGUST 4, 1989 | | | | SAMPLED AUGUST 7, 1989 | | | SAMPLED AUGUST 8, 1989 | |
|---|---|---|---|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 10:00 | 10:45 | 11:45 | 12:15 | 12:30 | 12:49 | 13:47 | 13:10 | 13:36 |
| | | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED |
| $CO_2$ | % VOL | | | .9 | 1.5 | 2.0 | | | 3.1 | |
| $H_2$ | % VOL | | | 11.0 | 13.8 | 14.8 | | | 18.2 | |
| $N_2$ | % VOL | | | 39.6 | 37.1 | 33.6 | | | 16.3 | |
| $O_2$ | % VOL | | | 5.4 | 6.9 | 7.2 | | | .9 | |
| $H_2O$ VAPOR | % VOL | .85 | | | | | | .35 | | |
| $CH_4$ | % VOL | | | 42.1 | 40.9 | 46.7 | | | 3.8 | |
| CO | % VOL | | | ND | <.2 | 3.8 | | | 6.6 | |
| | | | | | | | | | | |
| AMMONIA | ppm | | 3.6 | | | | 1.3 | | | .7 |
| TRACE ORGANICS | ppm | | | ND | | | | | | |
| | | | | | | | | | | |
| LAB | | BE | BE | MP | BE | BE | BE | BE | BE | BE |

BE – BOEING ENVIRONMENTAL LAB
MP – MATERIALS AND PROCESS LAB
ND – NONE DETECTED

5-9021-0-220

39

constituents containing hydrogen accounts for over 91 percent of the $H_2$ fed in. It is possible that the remaining hydrogen can be accounted for in the instrumentation and sample analysis error, or leakage, if the leak found at the back of the reactor was present during part of the test. Because of the large amount of methane formed and lost in the bleed, all of the $H_2$ fed did not go to product water (2.5-man rate of $H_2$ feed versus 2-man rate of $H_2O$ produced). Even less carbon was deposited than water formed on a man-level basis. A total of 17.9 man-days of $H_2O$ was produced on cartridge No. 2 while only 12.8 man-days of carbon had accumulated. In testing prior to the heater thermocouple improvements these totals corresponded much more closely.

If a mass balance is performed around carbon and the assumption made that none was lost through any leakage of $CO_2$ or other carbon-containing compounds, an estimate can be backed out for the average amount of $CO_2$ fed. This result is approximately 1.71 kg/day (3.78 lbm/day) $CO_2$, or a 1.72-man level. If it is then assumed that all of the $N_2$ detected in the bleed came from air in the feed $CO_2$, the average volume percentage of air would have been around 10.5 percent. Sample results from 4BMS $CO_2$ analyses averaged some 18-percent air which is higher, but the Bosch feed was alternated between 4BMS $CO_2$ and facility $CO_2$ which contained no air. At any rate, these percentages are at least four times higher than the S.S. Freedom specification of 2.5 percent maximum air in $CO_2$. Any air contamination impacts Bosch efficiency, resulting in loss of reactants through the bleed and increasing operating pressure.

Since the Bosch was the one subsystem in the test which was integrated with all three of the other subsystems, some discussion is called for concerning what was learned about the performance of the interfaces.

As discussed previously, since the $CO_2$ is held in an accumulator whose pressure cycles from $1.38 \times 10^5$ to $2.76 \times 10^5$ Pa (20 to 40 psia), regulation for constant pressure and flowrate was found to be needed for the Bosch. Since the Bosch can only regulate the $H_2$ feed for a given amount of $CO_2$, any excess $CO_2$ over the maximum amount of $H_2$ available causes Bosch internal pressures to rise and the reaction to cease. A significant pressure rise will initiate automatic reactor switchover. Figure 25 is a plot of $H_2$ flowrate metered by the Bosch as it adjusted to the inlet $CO_2$ before and after the proper pressure regulator was installed. The cycles before correspond to the 55-min cycles of the 4BMS and are a result of varying pressure from the $CO_2$ accumulator. After the regulator was installed, these cycles were dampened out and the Bosch operated at a more steady rate.

As mentioned in the discussion of the mass balance, normally the Bosch is fed more hydrogen from the SFE than it needs to reduce a three-man rate of $CO_2$. The excess $H_2$ would then be expected to build in pressure at the interface until it is relieved to vent through the Bosch via RV2 (Fig. 19). Since the $H_2$ flowrate to the Bosch was less than what was expected, there was no excess and this interplay could not be observed. There were several instances during the test when either the $CO_2$ bottle was changed or the $CO_2$ was sampled that the inlet $H_2$ pressure spiked enough to cause some venting, but for the most part the vent flow was zero. In all other respects the SFE/Bosch interface worked well. No flow regulation is needed because the Bosch handles variations internally. The SFE delivers $H_2$ at a pressure regulated by the downstream components and its output was sufficient to handle the back pressure from the Bosch.

40
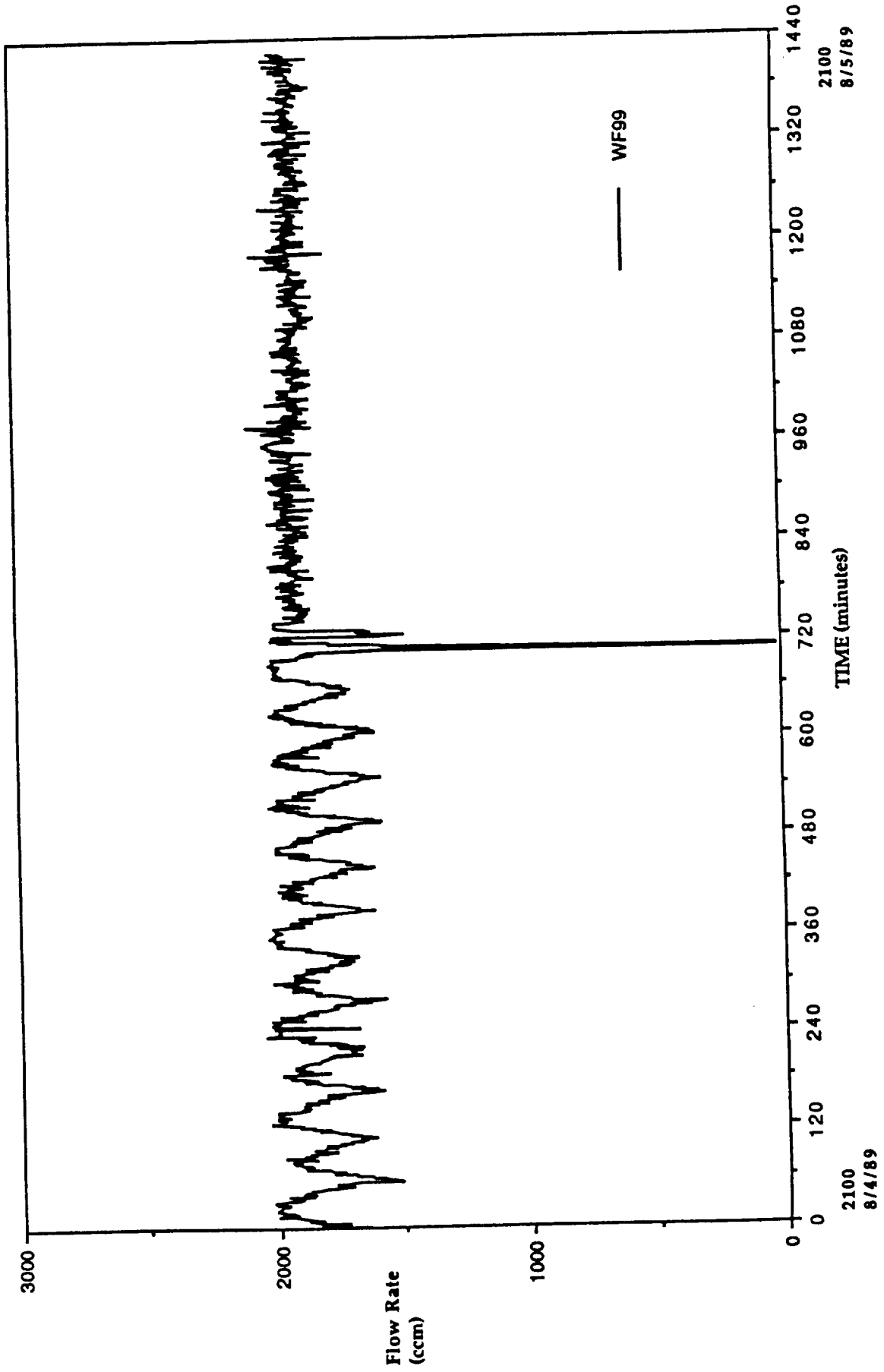
Figure 25. Bosch $H_2$ feed flow (WF99).

41

There were also no problems encountered controlling the interface to the TCCS for the Bosch bleed, and none were expected. A three-way valve was used to divert the bleed to vent when necessary.

Table 2 gives the results of the laboratory analysis performed on the Bosch product water samples taken during the test. The pH value of 7.2 is consistent with past water samples and is fairly neutral as expected.

Of the metals analyzed, all were below detectable limits except for calcium, which still had a low concentration of 0.15 ppm. These results are also consistent with past water sampling as there has never been any metal detected over 0.27 ppm (zinc in December 1987 bench test).

The amount of total solids present in the water for this test was an improvement over past testing. Typically, solid concentrations between 10 and 20 ppm have been previously detected, but during the SIT there was less than 10 ppm.

Anions were also very low as expected. One surprising result of the analysis was the relatively low ammonia concentration compared with past samples. Since previous testing with nitrogen in the feed $CO_2$ resulted in ammonia concentrations in the water between 1,000 and 1,250 ppm, it was expected that the result from the SIT would be at least that high, considering the rate of air leakage from the 4BMS. However, only 92.6 ppm was detected. All that can be concluded at this point is that more testing is needed before a determination can be made as to what level of ammonia can typically be expected.

### 3.3.4 Recommendations/Lessons Learned

The SIT was considered an overall success for the Bosch. There were no automatic shutdowns during the 11-day test, and the unit performed well with the feed rates it was given. However, as a result of the test and follow-up analysis, there are several recommendations to be made regarding future testing.

First, for the subsystem, the heater set point should be raised enough that the reactor bed temperatures are restored to 922 K (1,200 °F) for the present iron catalyst. An alternative would be to change to a nickel catalyst being considered pending some results from comparative testing of the Bosch III. If a nickel catalyst is used for future testing, the current bed temperature would probably be adequate and no heater changes would have to be made.

For the system, all interfaces to the Bosch should be leak checked with helium prior to another integrated test. All flowmeter calibrations should be verified in-house instead of relying on manufacturer's calibration curves. In some cases the calibrations were off even though they had supposedly been performed using the correct gas and operating pressure.

One final recommendation for the flight system design stems from what was learned about the 4BMS/Bosch interface. If there is a loss of delivered $CO_2$ from the 4BMS for any reason, the Bosch can adjust by automatically closing off the inlet $H_2$ through the gas composition controller. However, if there is a loss or sudden decrease of feed $H_2$ from the SFE, the current Bosch design

Table 2. Bosch $CO_2$ reduction water analyses.

| | | SAMPLED AUGUST 4, 1989 | | | | SAMPLED AUGUST 6 1989 |
|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 02:23 - 14:30 | 14:30 - 19:00 | 19:55 | 21:06 | 21:31 |
| ANALYTES | UNITS | BOSCH WATER | BOSCH WATER | BOSCH WATER | BOSCH WATER | BOSCH WATER |
| pH | | | | 7.2 | | |
| TOTAL SOLIDS | ppm | | | | <10 | |
| TOTAL DISSOLVED SOLIDS | ppm | | | | <10 | |
| TOTAL SUSPENDED SOLIDS | ppm | | | | <10 | |
| TURBIDITY | NTU | | | 9.6 | | |
| DISSOLVED $O_2$ | ppm | | | 2.02 | | |
| BARIUM | ppm | <.01 | | | | |
| CADMIUM | ppm | <.03 | | | | |
| CALCIUM | ppm | .15 | | | | |
| CHROMIUM | ppm | <.06 | | | | |
| IRON | ppm | <.05 | | | | |
| MAGNESIUM | ppm | <.01 | | | | |
| MANGANESE | ppm | <.01 | | | | |
| MOLYBOENUM | ppm | <.09 | | | | |
| NICKEL | ppm | <.11 | | | | |
| SILVER | ppm | <.14 | | | | |
| ZINC | ppm | <.02 | | | | |
| AMMONIA | ppm | | | | | 92.60 |
| CHLORIDE | ppm | | | | | .10 |
| FLORIDE | ppm | | | | | .27 |
| NITRATE | ppm | | | | | <.120 |
| PHOSPHATE | ppm | | | | | <.190 |
| POTASSIUM | ppm | | | | | 1.03 |
| SULFATE | ppm | | | | | <.190 |
| HETEROTROPHS | CFU/100ml | | 10.000 | | | |
| GRAM NEGATIVE | CFU/100ml | | 7.500 | | | |
| GRAM POSITIVE | CFU/100ml | | 100 | | | |
| YEAST AND MOLD | CFU/100ml | | <100 | | | |
| LAB | | BE | BE | BE | BE | BE |

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43

has no means of reducing the inlet $CO_2$ accordingly. This has the result of stopping the reduction reaction and raising internal pressures within the subsystem, either sending it into automatic switch-over or causing it to vent the $CO_2$ through the bleed to the TCCS and ultimately back into the cabin. To avoid this, the air revitalization system design should incorporate the proper instrumentation and controls which will redirect the $CO_2$ flow away from the Bosch if there is a loss of $H_2$ inlet pressure.


## 3.4 Trace Contaminant Control Subsystem (TCCS)

### 3.4.1 Detailed Description

The TCCS removes trace gas contaminants from the module atmosphere by circulating air through a series of packed sorbent beds and a high temperature catalytic oxidizer (Fig. 26). Located in either a high-flow or low-flow branch of the TCCS, each trace contaminant removal subassembly targets a range of contaminants to provide an efficient contamination control approach. Table 3 details some subassembly feature while Figure 27 illustrates the location of each sub-assembly in the TCCS.

The high-flow branch consists of a vane axial fan which draws 0.011 m$^3$/s (24 cfm) through a fixed charcoal bed. The charcoal bed contains 21.8 kg (48 lbm) of phosphoric acid-impregnated activated carbon. This bed functions as a removal device for high molar volume contaminants and for ammonia [7].

Approximately 0.0015 m$^3$/s (3.2 cfm) of the high-flow branch is drawn into the low-flow branch by a centrifugal blower, processed, and mixed with the air in the high-flow branch before exhausting to the test chamber atmosphere. This branch contains a regenerable charcoal bed, a lithium hydroxide (LiOH) presorbent bed, a high-temperature catalytic oxidizer, and a LiOH postsorbent bed. The regenerable bed was operated empty since the present S.S. Freedom TCCS design concept does not include a regenerable bed. If it was operated with the regenerable bed, the bed's function is to remove low molar volume contaminants. The presorbent bed contains 0.91 kg (2.0 lbm) of LiOH sorbent which removes acidic gases and potential catalyst poisons from the air before it enters the catalytic oxidizer. The catalytic oxidizer then oxidizes any contaminants which have not been removed by the fixed charcoal bed and presorbent bed. This assembly consists of a five-pass crosscurrent flow plate fin heat exchanger and an electrically heated catalyst canister. Air enters the heat exchanger where it is preheated by exiting air. The air is heated to the final operating temperature by two 70 watt electric heaters located in the catalyst canister. Heated air flows through a bed of 0.5 percent palladium on 3.18 mm (0.125 in) diameter alumina (Al2O3) spheres. Contaminants which are not easily removed by the preceding removal assemblies are oxidized in the canister. Air containing oxidation products flows out of the catalytic oxidizer assembly and into a LiOH postsorbent bed which contains 1.4 kg (3.0 lbm) of LiOH. The oxidation products are removed in this bed and clean air flows back to the high flow branch and is then exhausted to the cabin atmosphere [8].

The SIT was the first time that the TCCS had been integrated with other ECLSS subsystems. The air inlet to the fixed charcoal bed was interfaced with the temperature and humidity control system. This interface reduced the relative humidity of the inlet air to enhance contaminant

44
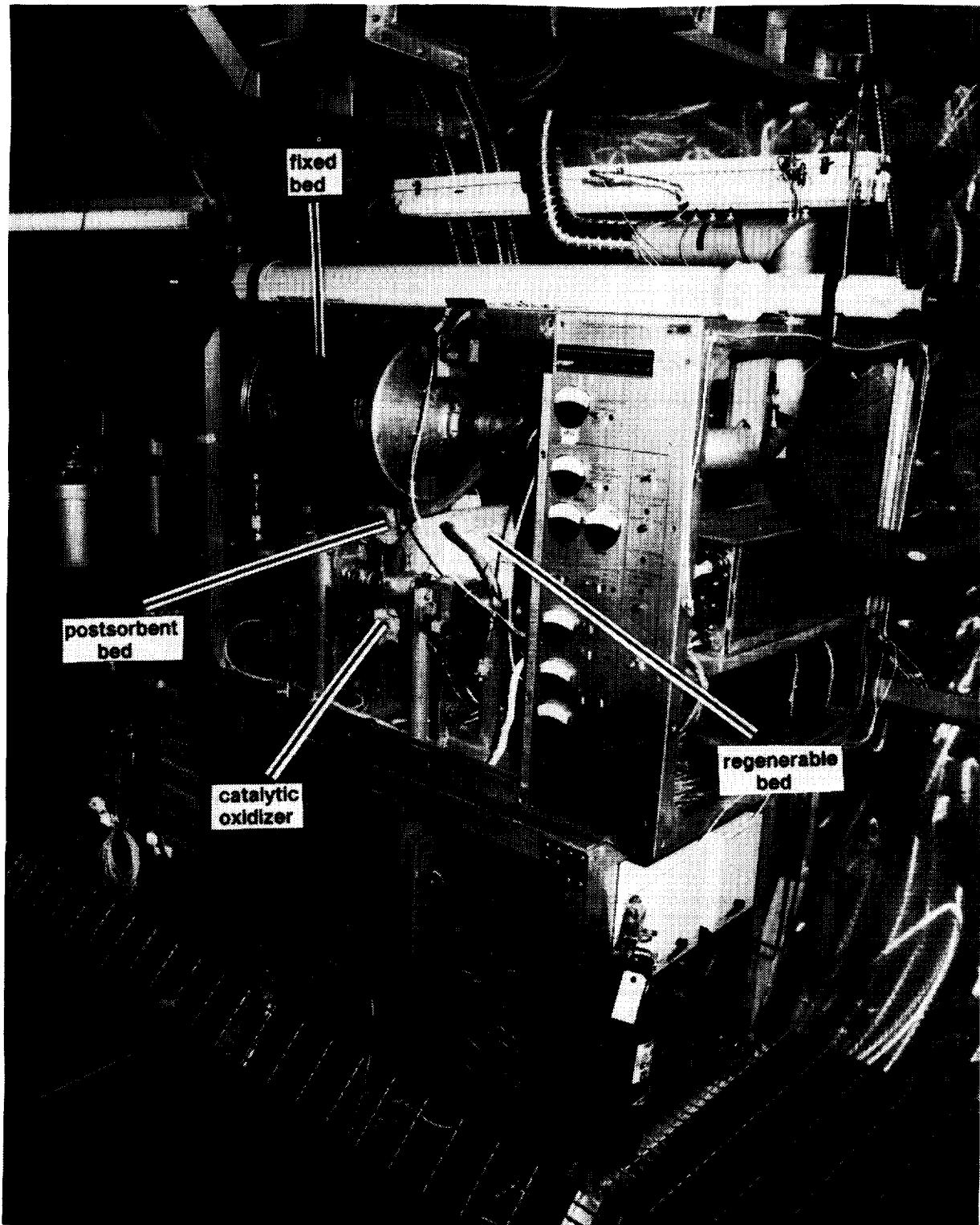
Figure 26. Trace contaminant control system.

Table 3. TCCS subassembly characteristics.

| COMPONENT | FLOW (m³/s) | LENGTH (m) | DIAMETER (m) | ΔP (Pa) | MASS (kg) |
|---|---|---|---|---|---|
| Fixed Bed | 0.0165 | 0.381 | 0.330 | 623 | 30.4 |
| Presorbent Bed | 0.00212 | 0.152 | 0.127 | 237 | 1.59 |
| Postsorbent Bed | 0.00212 | 0.222 | 0.127 | 386 | 2.04 |
| Catalytic Oxidizer | 0.00212 | 0.0914 | 0.0558 | 1490 | 14.1 |
| Axial Fan (11500 RPM/55W) | 0.0165 | 0.146 | 0.114 | 672 | 1.35 |
| Centrifugal Blower (22500 RPM/90W) | 0.00423 | -- | -- | 4230 | 1.02 |
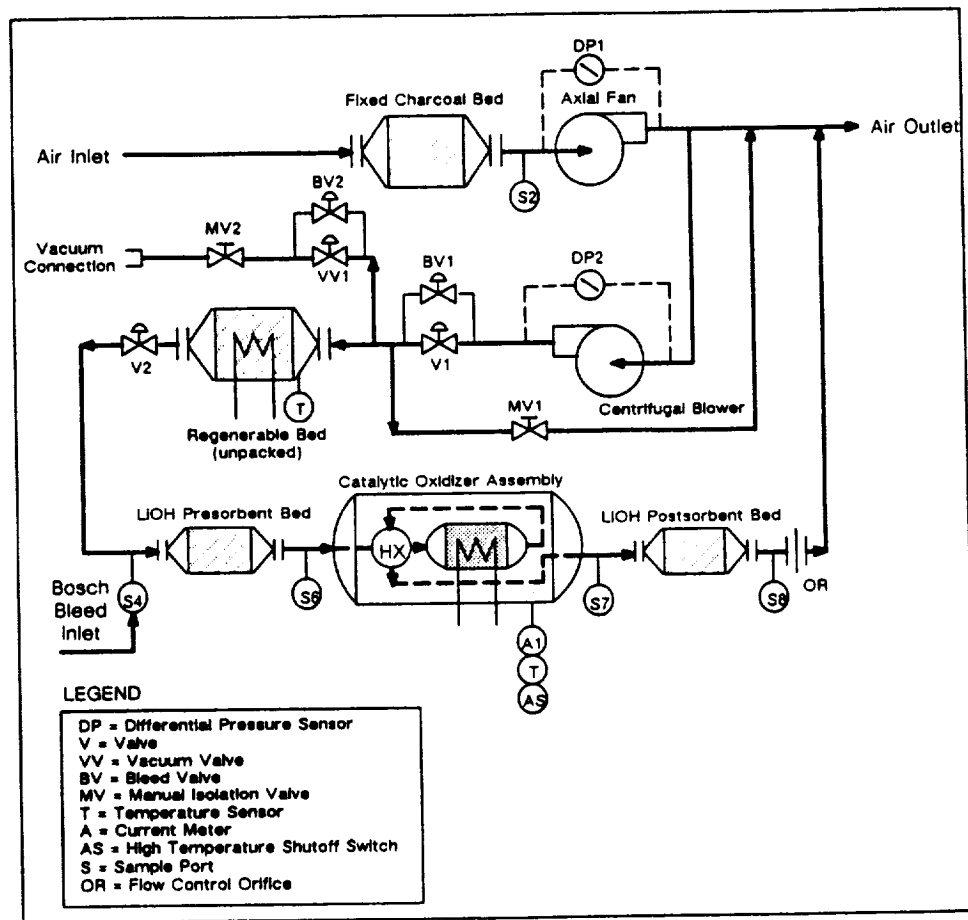


Figure 27. TCCS schematic.

removal and avoid contaminant displacement by water vapor. The major interface was with the Bosch carbon dioxide reduction subsystem. The Bosch process produces a bleed stream of reactants, inerts, and reaction products such as methane, carbon monoxide, hydrogen, nitrogen, oxygen, and carbon dioxide. Methane, carbon monoxide, and hydrogen are particularly important since they are major atmosphere constituents generated from material offgassing and crew metabolic processes. The bleed stream was introduced through a port just before the presorbent bed to obtain optimum oxidation of these contaminants. The TCCS exhaust was interfaced directly with the test chamber atmosphere.

TCCS instrumentation provides simple subsystem status information on electrical current, differential pressure, and catalytic oxidizer temperature. Table 4 documents the major instrumentation measurements and their nominal values. Differential pressure measurements are taken across the axial fan and centrifugal blower to determine whether they are operating properly. Most of the other data provided relates to the catalytic oxidizer operating conditions. The catalytic oxidizer heater current is monitored and is used as a control input to an overcurrent cutoff switch which shuts down the heater current in the event of a power surge. Likewise, the catalytic oxidizer temperature is monitored by a thermocouple probe which monitors the catalytic oxidizer external canister temperature. This temperature reading also serves as a control input for an overtemperature cutoff switch which shuts down the heater current. The temperature limit is required to protect the catalyst from extremely high temperatures which may result in its thermal degradation. A flow measurement was added in the low flow branch to aid in conducting mass and energy balances on the catalytic oxidizer assembly. Figure 27 shows the location of the major instrument readings.

### 3.4.2 TCCS Performance Analysis

The TCCS operated with few complications during the entire test. The major adjustment made during the test was to integrate and deintegrate the TCCS and Bosch bleed stream as necessary. Also, the catalytic oxidizer thermocouple probe which provides temperature readings would vibrate loose from its contact point and required minor adjustment periodically. These anomalies will be corrected by modifying the TCCS.

### 3.4.3 Discussion of Results

Design documentation for the TCCS shows that the subsystem should operate at 0.017 $m^3$/s (35 cfm) in the high flow branch and 0.00201 $m^3$/s (4.25 cfm) in the low flow branch. These flow rates produce a pressure drop of 669 Pa (0.097 psi) and 4,206 Pa (0.61 psi) across the vane axial fan and centrifugal blower, respectively. The catalytic oxidizer external canister temperature reaches 616 K (650 °F) which corresponds to an air temperature of 672 K (750 °F). Figure 28 shows the expected catalytic oxidizer operating point. At this temperature approximately 9 percent of the methane and 100 percent of the carbon monoxide and hydrogen will be oxidized per pass.

The TCCS performance during the SIT exceeded the expected performance for methane oxidation, 67.3 percent per pass, but fell short of expectations for carbon monoxide oxidation which achieved only 83.1 percent per pass. Sample analysis results illustrating these efficiencies are summarized in Table 5. The increase in methane conversion was accompanied by an increase in

Table 4. TCCS process measurements.

| MEASUREMENT | TEST ID NUMBER | UNITS | NOMINAL VALUE |
|---|---|---|---|
| Catalyic Oxidizer Heater Current | JI01 | Amps | 1.3 |
| Catalyic Oxidizer Cannister Temp | JT01 | Fahrenheit | 680 |
| Centrifugal Blower Differential Pressure | JP01 | in. $H_2O$ | 17 |
| Axial Fan Differential Pressure | JP02 | in. $H_2O$ | 3.5 |
| Low Flow Branch Flow Rate | JF01 | $ft^3/min$ | 3.2 |
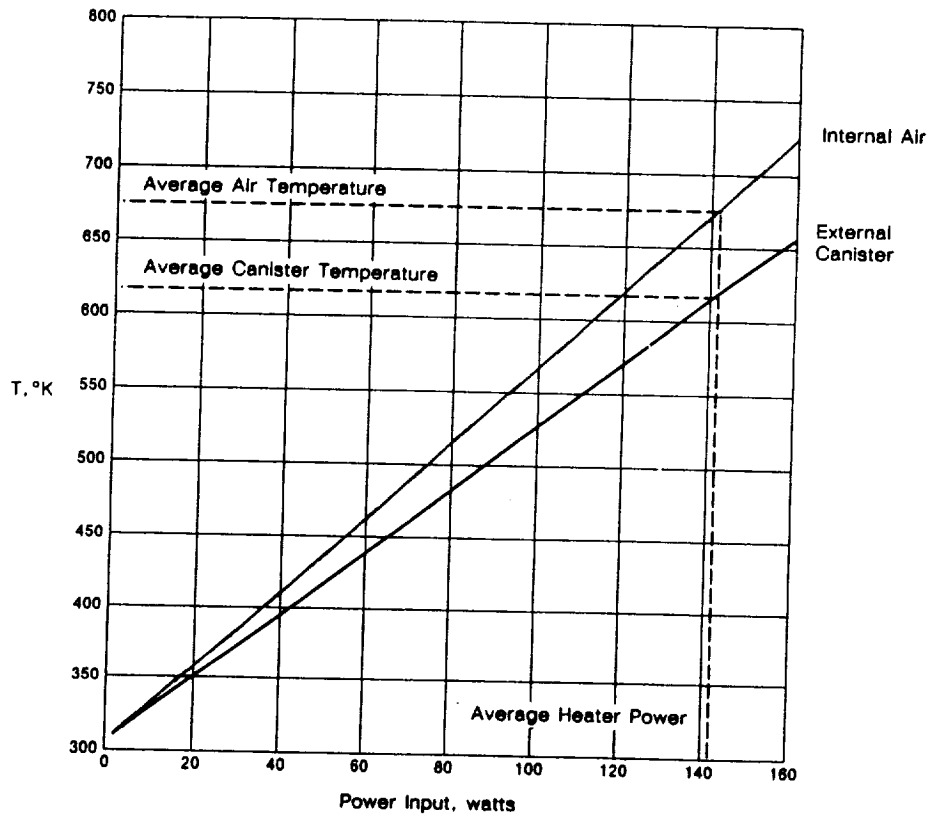


Figure 28. Catalytic oxidizer performance at 4.2 CFM air flowrate.

48

Table 5. Summary of TCCS sample analysis results.

| SAMPLE IDENTIFICATION NUMBER | CAT. OXIDIZER SAMPLE LOCATION | | CONCENTRATION | | CONVERSION | |
|---|---|---|---|---|---|---|
| | Inlet | Outlet | $CH_4$ | CO | $CH_4$ | CO |
| JS6-EH-0935-[8-3-89] *<br>JS7-EH-0945-[8-3-89] * | X | X | 30.21 ppm<br>0.00 ppm | 0.00 ppm<br>0.00 ppm | – | – |
| JS6-EH-1102-[8-3-89]<br>JS7-EH-1105-[8-3-89] | X | X | 4543.9 ppm<br>956.1 ppm | 171.6 ppm<br>26.1 ppm | 0.790 | 0.848 |
| JS6-BAC-0657-[8-4-89]<br>JS7-BAC-0659-[8-4-89] | X | X | 0.13 vol%<br>0.05 vol% | <0.2 vol%<br><0.2 vol% | 0.615 | – |
| JS6-BAC-1232-[8-4-89]<br>JS7-BAC-1236-[8-4-89] | X | X | 0.17 vol%<br>0.04 vol% | <0.2 vol%<br><0.2 vol% | 0.765 | – |
| JS6-EH-1632-[8-4-89]<br>JS7-EH-1642-[8-4-89] | X | X | 3204.13 ppm<br>694.59 ppm | 147.12 ppm<br>24.28 ppm | 0.783 | 0.835 |
| JS6-EH-0818-[8-5-89]<br>JS7-EH-0826-[8-5-89] | X | X | 2693.05 ppm<br>835.23 ppm | 135.46 ppm<br>23.18 ppm | 0.690 | 0.829 |
| JS6-BAC-1524-[8-7-89]<br>JS7-BAC-1525-[8-7-89] | X | X | 0.16 vol%<br>0.07 vol% | <0.2 vol%<br><0.2 vol% | 0.562 | – |
| JS6-BAC-1734-[8-8-89] *<br>JS7-BAC-1737-[8-8-89] * | X | X | <0.4 vol%<br><0.4 vol% | <0.2 vol%<br><0.2 vol% | – | – |
| JS6-EH-1057-[8-10-89]<br>JS7-EH-1101-[8-10-89] | X | X | 2735.94 ppm<br>470.10 ppm | 141.35 ppm<br>26.44 ppm | 0.828 | 0.813 |
| JS6-EH-0915-[7-31-89]<br>JS7-EH-0919-[7-31-89] | X | X | 25.0 ppm<br>16.2 ppm | –<br>– | 0.352 | – |
| AVERAGE CONVERSION | | | | | 0.673 | 0.831 |

* Background samples not included in performance evaluation.

49

the catalytic oxidizer operating temperature. The external canister temperature averaged 650 K (710 °F) during the time the TCCS was integrated with the Bosch. This corresponds to an air temperature of 711 K (820 °F). Figure 29 shows the actual catalytic oxidizer operating point. Differential pressures for both the axial fan and centrifugal blower were low, having values of 607 Pa (0.088 psi) and 4,109 Pa (0.596 psi), respectively. The lower differential pressure reading for the centrifugal blower was accompanied by a low branch flow rate which averaged 0.0015 m³/s (3.2 cfm). This flow rate is lower than expected but is consistent with the lower blower differential pressure.

Contaminants introduced into the TCCS through the Bosch bleed stream are an excellent performance challenge for the TCCS catalytic oxidizer assembly. The contaminants of interest are methane, carbon monoxide, and hydrogen. Expected oxidation efficiencies for these contaminants are 9 percent for methane and 100 percent for both carbon monoxide and hydrogen. Results of gas samples taken of the catalytic oxidizer assembly influent and effluent, shown in Table 5, show an average oxidation efficiency of 67.3 percent for methane and 83.1 percent for carbon monoxide. Hydrogen oxidation efficiency could not be determined from the sample analysis because the inlet and outlet concentrations fell below the sample analysis detector sensitivity.

Improved methane oxidation efficiency was obtained because the catalytic oxidizer temperature was much higher than expected. Figure 30 shows the expected and actual methane oxidation efficiencies as a function of operating temperature. The average temperature of 711 K (820 °F) corresponds directly to 67.3-percent methane conversion.

Poor carbon monoxide oxidation efficiency was not expected. Design literature shows that carbon monoxide oxidation efficiency is 100 percent per pass regardless of the operating conditions. The temperature experienced during the SIT should not result in poor conversion. Post-test material balances were conducted to assess this result. During the analysis, two cases were considered. The first case assumes 83.1-percent carbon monoxide oxidation overall while the second case assumes 100-percent oxidation of the feed carbon monoxide and incomplete methane oxidation with 93-percent oxidation of the carbon monoxide produced by this reaction. The second case results in an 83.1-percent apparent overall carbon monoxide oxidation efficiency and the first case also supports the test results. The second case is the more realistic since carbon monoxide oxidation should be very close to complete. Any carbon monoxide produced within the reactor would be more sensitive to residence time and may not be completely oxidized resulting in apparently low carbon monoxide oxidation efficiency [9]. Additional testing will be required to determine the actual mechanism of this result.

Post-test analysis predicting the subsystem's capability to control contaminant concentrations to levels below the spacecraft maximum allowable concentration (SMAC) indicates that the conditions of the SIT would result in a methane concentration at 23 percent of its SMAC of 1,771 mg/m³ and a carbon monoxide concentration at 76 percent of its SMAC of 28.60 mg/m³ in a closed test chamber at the end of the SIT. This performance is shown in Figure 31.
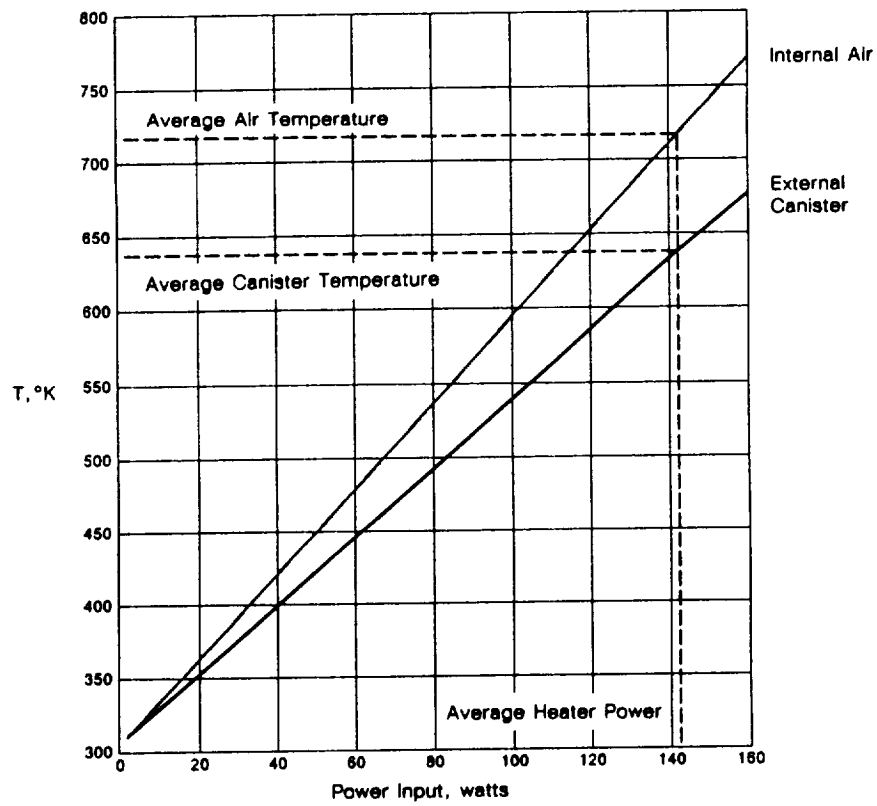
Figure 29. Catalytic oxidizer performance at 3.2 CFM air flowrate.
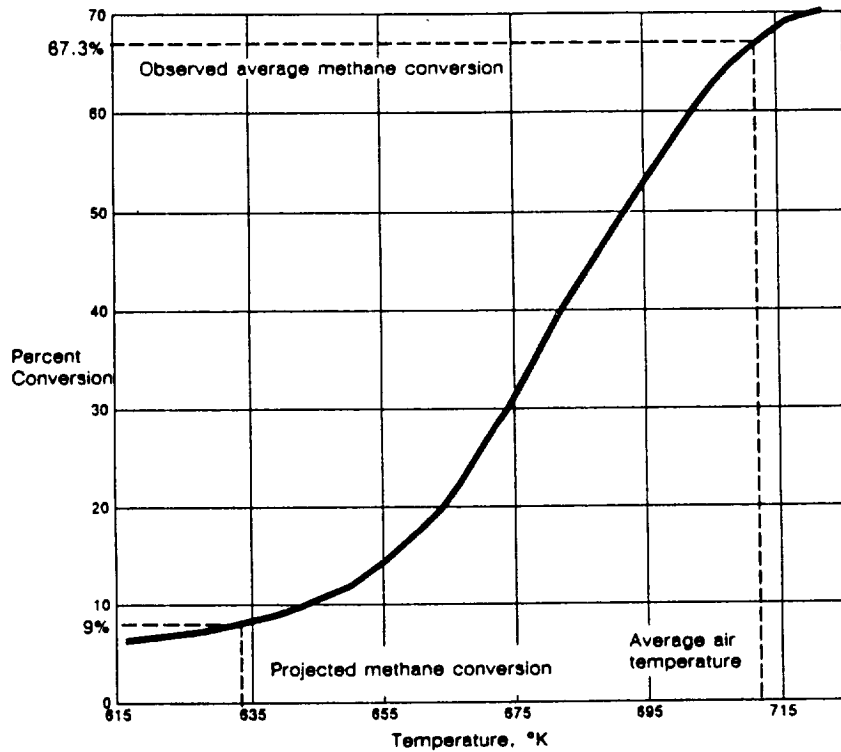


Figure 30. Catalytic oxidizer methane conversion performance.

51
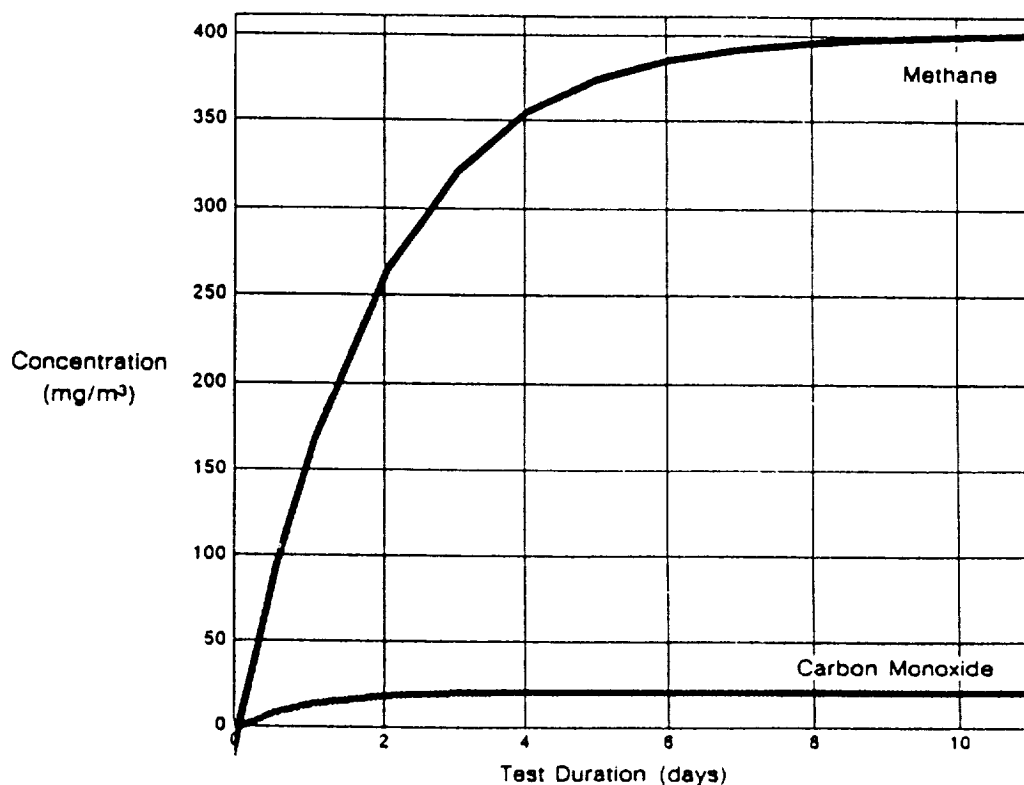
Figure 31. Projected test chamber methane and carbon monoxide.

During the SIT, the catalytic oxidizer was at a much higher temperature than expected. After achieving integration with the Bosch bleed stream, the external canister temperature rose to as high as 700 K (800 °F) which corresponds to an air temperature of 755 K (900 °F). The average external canister temperature was 650 K (710 °F) which corresponds to an air temperature of 711 K (820 °F). This temperature rise results from the additional energy released by the exothermic oxidation reactions occurring in the catalytic oxidizer canister. A post-test analysis of the energy released during these reactions predicts an air temperature of 703 K (806 °F) if the design temperature of 633 K (680 °F) is reached by the heater [10]. It must be noted that the analysis assumes energy losses through conduction and radiation routes are minimal.

The high temperatures experienced during the SIT required some precautions to protect the catalyst from thermal degradation. Temperatures above 811 K (1,000 °F) may cause thermal degradation of the catalyst resulting in reduced oxidation efficiencies. Since the TCCS flow rate in the low flow branch is controlled by a fixed orifice plate, the temperature in the catalytic oxidizer could not be regulated by controlling the flow rate. Therefore, when the temperature approached 700 K (800 °F), the Bosch bleed stream was diverted to an external duct for a period of time to allow the catalytic oxidizer to cool. This technique is not desirable, especially when the Bosch bleed is continuous and must interface with the TCCS in an uninterrupted manner to achieve efficient contamination control. Modifications to the TCCS to allow flow rate regulation will eliminate the need to deintegrate the Bosch bleed stream from the TCCS in future testing.

### 3.4.4 Recommendations/Lessons Learned

The SIT demonstrated that the TCCS can be successfully integrated with other ECLSS subsystems and can operate continuously for extended periods of time with minimal adjustment. The oxidation efficiencies obtained in the test indicate that additional testing is required to understand and validate the TCCS removal capabilities for methane and carbon monoxide and to study efficiency sensitivity to air flow rate, catalytic oxidizer temperature, and air contaminant concentration. Also, some refurbishment of the catalytic oxidizer temperature probe and capability to control the flow rate in the low flow branch are necessary to obtain optimum performance data.

# 4.0   AIR AND WATER SAMPLING RESULTS

## 4.1   Introduction

During the phase III SIT, gas and water samples were taken to evaluate subsystem and system level performance. Gas samples were taken from the inlet and outlet of each subsystem in addition to background atmospheric samples taken within the building and module simulator. Background air samples were taken to determine if organic contaminants were present in the atmosphere which could contaminate other gas and water samples taken. Water samples were taken from the Bosch $CO_2$ reduction subsystem and temperature and humidity control unit. See Table 6 for a list of the subsystems sampled and their respective sample points.

The majority of all gas and water samples were taken by an assigned Boeing laboratory analyst. A few selected TCCS gas and Bosch product water samples were taken by other civil service personnel who were familiar with gas sampling techniques. The on-line gas samples were collected in 5- or 15-liter sampling bags in addition to a few TCCS gas samples which were collected using a metal evacuated cylinder. After the samples were collected for each day, they were taken to the Boeing Environmental (BE) Laboratory and/or the MSFC Materials and Processes (MP) Laboratory for analysis. Table 7 shows the sample schedule implemented by each laboratory and the analytes determined for each sample point. All subsystem sample ports were located outside the simulated module except for the TCCS and Bosch product water ports which were located inside the module.

The initial intent of the ECLSS Branch was to have two additional independent laboratories analyzing gas samples in parallel with the BE laboratory. Because of contracting problems during negotiations, the independent laboratories could not begin until after the phase III SIT. As an expedient solution, the MP Laboratory was requested to help in providing analytical services for analyzing gas samples.

## 4.2   Sampling Technique

All gas samples analyzed by the BE Laboratory and taken prior to August 8, 1989, were collected in 5- or 15-liter capacity, five-layer sampling bags purchased from Calibrated Instruments Inc. Gas samples taken on August 8, 1989, were collected using Tedlar film sample bags. A few gas samples analyzed by the MP Laboratory collected from the TCCS were taken using a metal

Table 6. Subsystem sample points.

| SUBSYSTEM | SAMPLE POINTS |
|---|---|
| **WATER** | |
| BOSCH | 1.) PRODUCT WATER |
| TEMPERATURE AND HUMIDITY COMDENSATE (THC) | 1.) CONDENSATE |
| **AIR** | |
| BOSCH | 1.) REACTOR INLET<br>2.) BOSCH BLEED |
| STATIC FEED ELECTROLYSIS (SFF) | 1.) PRODUCT $O_2$<br>2.) PRODUCT $H_2$ |
| MOLECULAR SIEVE (MS) | 1.) INLET AIR<br>2.) OUTLET AIR<br>3.) $CO_2$ PRODUCT TO STORAGE |
| TRACE CONTAMINANT CONTROL (TCC) | 1.) PORT #6 (INLET TO CATALYTIC OXIDIZER)<br>2.) PORT #7 (OUTLET OF CATALYTIC OXIDIZER) |
| MODULE ATMOSPHERE | 1.) INSIDE MODULE |
| 4755 BUILDING ATMOSPHERE | 1.) OUTSIDE MODULE |

Table 7. Sampling schedule.

**TEST DAY**

| SAMPLE POINT | 7/31 SAMPLE GROUP* | | 8/3 SAMPLE GROUP | | 8/4 SAMPLE GROUP | | 8/5 SAMPLE GROUP | | 8/6 SAMPLE GROUP | | 8/7 SAMPLE GROUP | | 8/8 SAMPLE GROUP | | 8/10 SAMPLE GROUP | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM |
| **BOSCH** | | | | | | | | | | | | | | | | |
| PRODUCT WATER | | | | | I | | | | II | | | | | | | |
| REACTOR INLET | | | IV | | V | | | | | | V | | V | | | |
| BLEED GAS | | | IV | | IV | | | | | | IV | | IV | | | |
| **SFE** | | | | | | | | | | | | | | | | |
| PRODUCT O₂ | | | V | IV | V | IV | | | | | V | | V | | | |
| PRODUCT H₂ | | | V | IV | V | IV | | | | | V | | V | | | |
| **MS** | | | | | | | | | | | | | | | | |
| INLET AIR | | | V | VII | V | | | | | | V | | V | | | |
| OUTLET AIR | | | V | VII | V | | | | | | V | | V | | | |
| CO₂ PRODUCT | | | V | VII | V | | | | | | V | | V | | | |
| **TCCS** | | | | | | | | | | | | | | | | |
| PORT #6 | IV | VII | IV | VII VII | IV | VII VII | | VIII | | VIII | IV | | IV | | VIII | |
| PORT #7 | IV | VII | IV | VII VII | IV | VII VII | | VIII | | VIII | IV | | IV | | VIII | |
| **THC** | | | | | | | | | | | | | | | | |
| CONDENSATE | | | III | | III | | | | | | | | III | | | |
| **BUILDING** | | | IV | VII | | | | | | | IV | | IV | | | |
| **MODULE** | | | IV | VII | | | | | | | IV | | IV | | | |
| LAB ➝ | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM | BE | PM |

BE – BOEING ENVIRONMENTAL LABORATORY
MP – MATERIALS AND PROCESS LABORATORY
*SAMPLE GROUPS ARE DEFINED ON THE FOLLOWING PAGE

5-8350-0-220

Table 7. Sampling schedule (continued)

# Sample Groups

## Water Samples

I.   pH, Total Solids, Total Dissolved Solids, Total Suspended Solids, Turbidity, Dissolved $O_2$ Metals – Cd, Ba, Ca, Cr, Fe, Mg, Mn, Mo, Ni, Ag, Zn Microbiologicals – Heterotrophs, Gram Negative, Gram Positive, Total Yeast and Molds

II.  Ions – $NH_4^+$, $Cl^-$, $PO_4^{3-}$, $Fl^-$, $NO_3^{2-}$, $SO_4^{2-}$, $K^+$

III. Ph, Conductivity, Total Phenols, Volatile Organics, Base Neutral/Acid Extractables

## Gas Samples

IV.   $CO_2$, $CO$, $CH_4$, $H_2$, $N_2$, $O_2$

V.    $CO_2$, $CO$, $CH_4$, $H_2$, $N_2$, $O_2$, $H_2O$ Vapor

VI.   $CO_2$, $CO$, $CH_4$, $H_2$, $N_2$, $O_2$, $H_2O$ Vapor, Ammonia

VII.  $CO_2$, $CO$, $CH_4$, $H_2$, $N_2$, $O_2$, Trace Organics

VIII. $CO$, $CH_4$, $H_2$

evacuated sample cylinder. Both a 15-liter, five-layer sampling bag and a sample cylinder are shown in Figure 32. The sampling technique used to sample the SFE, 4BMS, TCCS, and Bosch subsystems required connecting a plastic tube to the sample port, purging the sample port and tubing to remove contaminants, attaching the sample bag, and filling the bag. The sample bag had a built-in valve with a hose bib connection which was opened or closed by twisting the bib. After the bag was connected to the plastic tubing, it was quickly twirled to open the valve and allow the flow of gas into the bag. Prior to sample collection, the tubing and sample ports were purged from 15 seconds to 5 minutes depending on the flow of gas through the line being sampled. The purge times used prior to sample collection are listed below for each subsystem.

| Subsystem | Purge Time |
|---|---|
| 4BMS | 30 s |
| SFE | 5 min |
| TCCS | 15 s |
| Bosch | |
| Bleed Gas Line | 3 min |
| Inlet Feed Line | 30 s |

Because the gas pressure was low at the 4BMS inlet an outlet gas lines, a Teflon diaphragm pump had to be used to purge and fill the sample bags.

The analysis for water vapor in gas was done on-line by passing the sampled gas through a midget impinger filled with indicator silica gel. A wet test meter was connected to the outlet of the impinger to measure the volume of gas passing through the impinger. The impinger filled with silica gel, a desiccant which will absorb water vapor, was dried and weighed prior to the analysis. The impinger was connected to the sample port and 0.5 to 2 ft$^3$ of gas was passed through the impinger. The impinger was taken back to the laboratory and weighed. The difference in weight was contributed to water vapor present in the gas stream.

Ammonia in gas was collected on-line by passing the sampled gas through a midget impinger filled with 0.1 normal water-sulfuric acid solution. A wet test meter was connected to the outlet of the impinger to measure the volume of gas passed through the impinger. The ammonia in the gas is converted to ammonium ($NH_4+$) and the concentration determined by ion chromatography.

Some of the TCCS gas samples were collected using evacuated gas cylinders. The technique used to fill the cylinder required connecting metal tubing to the sample port, purging through the port and tubing, connecting the cylinder, purging through the cylinder three times to remove contaminants, and collecting a gas sample for analysis. Initially the cylinder was under vacuum and the gas was allowed to flow into the cylinder by opening the inlet valve. Once the cylinder was full of gas, the outlet valve was opened and the gas allowed to purge through the cylinder for 3 minutes. After this period of purging, both inlet and outlet valves were closed and then reopened to continue purging for 1 minute. This process was repeated again before a gas sample was taken for analysis. Each gas sample collected in gas cylinders followed this purging sequence.

Bosch water was collected in a graduated cylinder as the water was pumped from the subsystem water tank. The cylinder was used to measure the volume of water produced so that mass
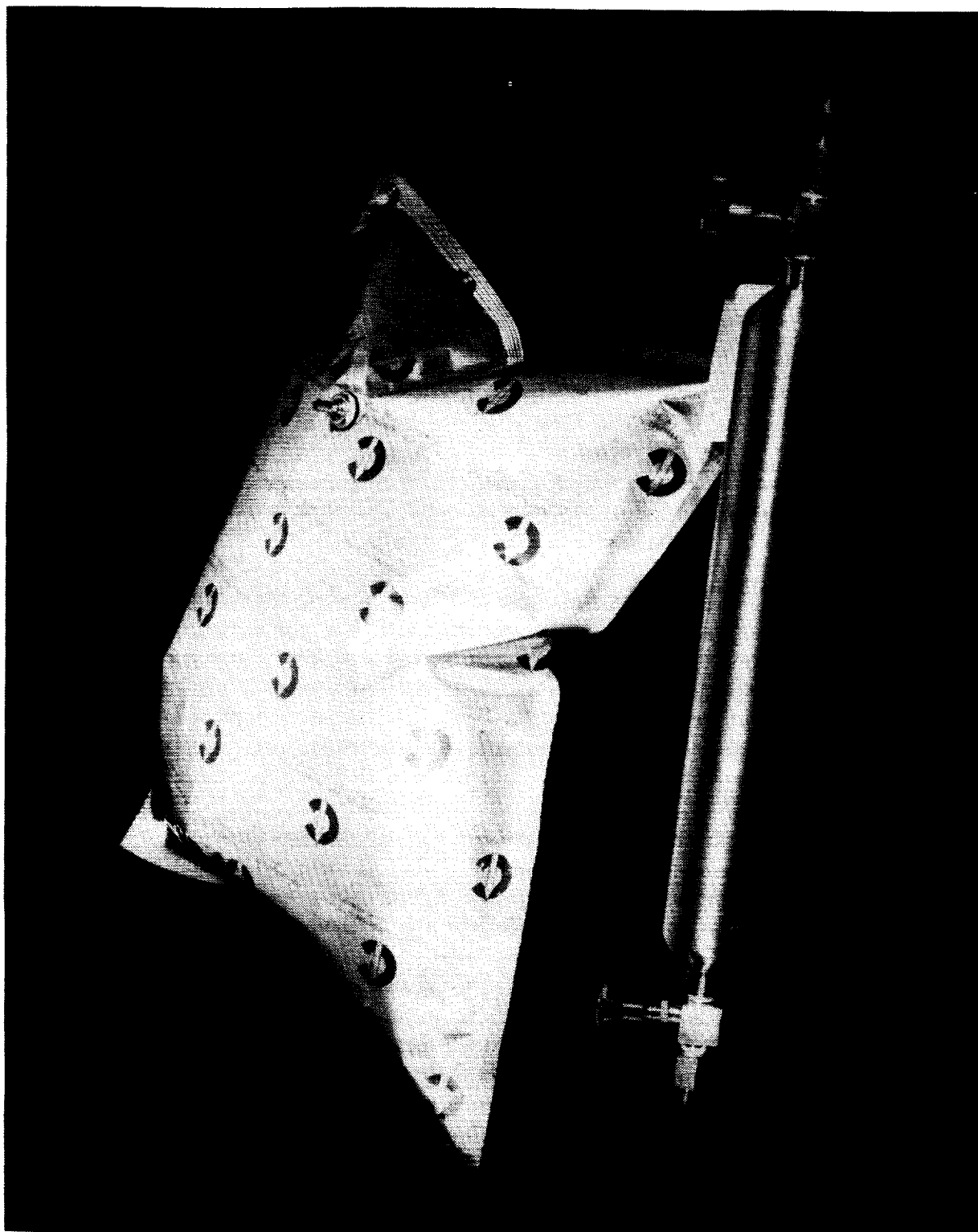
Figure 32. Gas sampling devices – bag and cylinder.

balance calculations could be made. Once the volume was measured, the water was either poured into the proper sample container and refrigerated or collected in another container and archived.

Humidity condensate samples were collected as condensate was produced. Condensate water was abundant and samples could be taken at any time.

## 4.3  Comparison of Laboratory Results

Table 8 lists the results of gas samples analyzed by both the MP and BE laboratories which were believed to have the same analyte concentrations. These samples were taken in serial order usually within minutes of each other. Because the gas samples were not collected at the same time or split from an initially filled sample bag, the samples could not be classified as true duplicate samples. The results of each set of analytes determined ($CO_2$, $CO$, $CH_4$, $H_2$, $O_2$, $N_2$) was expected to be the same, but analyte concentrations could have varied. The laboratory results which showed the largest differences are circled in Table 8. As a measure of precision, the relative percent difference (RPD) is listed below for those results. For an explanation of RPD see Section 4.5.

| Analyte | Units | Result | | RPD |
|---|---|---|---|---|
| $CO_2$ | % Vol | 3.6 | 0.7 | 133.3 |
| | % Vol | 0.04 | 0.6 | 175.0 |
| | % Vol | 0.41 | 0.1 | 121.6 |
| | % Vol | 89.6 | 71.7 | 22.2 |
| $O_2$ | % Vol | 9.5 | 6.4 | 39.0 |
| $N_2$ | % Vol | 29.0 | 46.9 | 47.2 |
| | % Vol | 33.6 | 23.0 | 37.5 |
| | % Vol | 11.5 | 22.5 | 64.7 |
| LAB | | BE | MP | |

For real duplicate samples analyzed by separate laboratories, an RPD value of less than 20 is an acceptable goal. Other result comparisons listed in Table 8 give a calculated RPD of less than five RPD.

## 4.4  Results

The results for each subsystem including environmental air samples are given in Tables 9 through 15. The results are listed in chronological order, and the laboratory which performed the analysis is given at the bottom of each page. Detection limits and methods of analyses are given in Tables 16 through 18.

There were a number of parameters which were initially requested in the sample requirements for which the BE laboratory could not determine. This was due to either inadequte

Table 8. Laboratory comparison.

**SAMPLES TAKEN AUGUST 3, 1989**

| SAMPLE POINT | UNITS | CO2 | | CO | | CH4 | | H2 | | O2 | | N2 | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **BOSCH** | | | | | | | | | | | | | |
| REACTOR INLET | % VOL | 3.5 | .7 | 5.9 | ND | 33.3 | 32.1 | 23.1 | 14.4 | 3.4 | 4.8 | 29.0 | 46.9 |
| **SFE** | | | | | | | | | | | | | |
| PRODUCT O2 | % VOL | — | .2 | — | ND | — | ND | — | ND | 85.7 | 82.4 | 15.6 | 16.4 |
| PRODUCT H2 | % VOL | <.04 | .6 | <.2 | ND | <.04 | ND | 81.2 | 69.0 | 9.5 | 6.4 | 33.6 | 23.0 |
| **MS** | | | | | | | | | | | | | |
| INLET AIR | % VOL | .41 | .1 | ND | ND | <.04 | .0030 | <.4 | ND | 22.1 | 20.9 | 78.8 | 72.9 |
| OUTLET AIR | % VOL | .15 | .1 | .2 | ND | <.04 | .0035 | <.4 | ND | 22.2 | 22.1 | 78.8 | 77.8 |
| CO2 PRODUCT | % VOL | 89.6 | 71.7 | <.2 | .0007 | <.04 | .0014 | <.4 | ND | 4.8 | 3.1 | 11.5 | 22.5 |
| **TCCS** | | | | | | | | | | | | | |
| PORT #6 | % VOL | .09 | <.1 | <.2 | ND | <.04 | ND | <.4 | ND | 22.4 | 20.7 | 78.8 | 78.2 |
| PORT #7 | % VOL | .11 | <.1 | <.2 | ND | <.04 | .0089 | <.4 | ND | 22.3 | 21.4 | 77.7 | 77.6 |
| BUILDING | % VOL | .08 | <.1 | <.2 | ND | <.04 | ND | <.4 | ND | 22.0 | 20.2 | 78.7 | 78.8 |
| MODULE | % VOL | .08 | <.1 | <.2 | ND | <.04 | ND | <.4 | ND | 22.2 | 20.3 | 78.5 | 78.7 |
| **LAB** | | BE | MP | BE | MP* | BE | MP* | BE | MP | BE | MP | BE | MP |

BE – BOEING ENVIRONMENTAL LABORATORY
MP – MATERIALS AND PROCESS LABORATORY
*RESULTS FOR CH4 AND CO DETERMINED BY THE MP LAB WERE CONVERTED FROM ppb TO % VOLUME.
IN SOME CASES THE NUMBER WAS TRUNCATED AND ROUNDED OFF.

5-8348-0-220

60

Table 8. Laboratory comparison (continued)

**SAMPLES TAKEN JULY 31, 1989**

| SAMPLE POINT | UNITS | CO$_2$ | | CO | | CH$_4$ | | H$_2$ | | O$_2$ | | N$_2$ | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TCSS | | | | | | | | | | | | | |
| PORT #6 | % VOL | .06 | <.1 | <.2 | ND | <.04 | .0025 | <.4 | ND | 22.1 | 20.5 | 78.1 | 78.5 |
| PORT #7 | % VOL | .06 | <.1 | <.2 | ND | <.04 | .0016 | <.4 | ND | 22.2 | 20.4 | 78.7 | 78.5 |
| | | | | | | | | | | | | | |
| LAB | | BE | MP | BE | MP | BE | MP | BE | MP | BE | MP | BE | MP |

**SAMPLES TAKEN JULY 31, 1989**

| SAMPLE POINT | UNITS | CO$_2$ | | CO | | CH$_4$ | | H$_2$ | | O$_2$ | | N$_2$ | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| BOSCH | | | | | | | | | | | | | |
| BLEED GAS | % VOL | 1.5 | .9 | <.2 | ND | 40.9 | 42.1 | 13.8 | 11.0 | 6.9 | 5.4 | 37.1 | 39.6 |
| TCCS | | | | | | | | | | | | | |
| PORT #6 | % VOL | .09 | <.1 | .2 | .01716 | .17 | .45439 | <.4 | ND | 22.4 | 21.1 | 78.2 | 77.4 |
| PORT #7 | % VOL | .18 | .1 | <.2 | .00261 | <.04 | .09561 | <.4 | ND | 22.2 | 19.7 | 78.2 | 79.2 |
| | | | | | | | | | | | | | |
| LAB | | BE | MP | BE | MP* | BE | MP* | BE | MP | BE | MP | BE | MP |

BE – BOEING ENVIRONMENTAL LABORATORY
MP – MATERIALS AND PROCESS LABORATORY
*RESULTS FOR CH$_4$ AND CO DETERMINED BY THE MP LAB WERE CONVERTED FROM ppb TO % VOLUME.

61

Table 9. Molecular sieve gas analyses.

| ANALYTES | UNITS* | 14:05 PRODUCT CO₂ | 14:06 PRODUCT CO₂ | 14:55 INLET | 14:56 INLET | 14:58 OUTLET | 14:59 OUTLET | 15:05 OUTLET | 15:12 INLET | 16:00 PRODUCT CO₂ | 13:20 PRODUCT CO₂ | 13:25 PRODUCT CO₂ | 13:45 INLET | 13:45 INLET | 13:55 OUTLET | 13:58 OUTLET |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | SAMPLED AUGUST 3, 1989 | | | | | | | SAMPLED AUGUST 4, 1989 | | | | | |
| $CO_2$ | % VOL | 71.7 | 89.6 | .1 | .41 | .1 | .15 | | | .37 | 83.6 | | .4 | | .16 | |
| $H_2$ | % VOL | ND | <.4 | ND | <.4 | ND | <.4 | | | <.4 | <.4 | | <.4 | | <.4 | |
| $N_2$ | % VOL | 22.5 | 11.5 | 77.9 | 78.8 | 77.8 | 78.8 | | | | 29.4 | | 77.5 | | 77.5 | |
| $O_2$ | % VOL | 4.8 | 3.1 | 20.9 | 22.1 | 21.1 | 22.2 | | | | 4.6 | | 22.5 | | 22.5 | |
| $H_2O$ VAPOR | % VOL | | | | | | | 1.9 | .2 | .99 | | 1.6 | | 1.0 | | .19 |
| $CH_4$ | % VOL | .0014 | <.04 | .0031 | <.04 | .0035 | <.04 | | | <.04 | <.04 | | <.04 | | <.04 | |
| CO | % VOL | .0007 | <.2 | ND | 0 | ND | .2 | | | | <.2 | | <.2 | | <.2 | |
| | | | | | | | | | | | | | | | | |
| TRACE ORGANICS | | | | | | | | | | | | | | | | |
| FREON 113 | ppm | 2.1 | | 1.0 | | .8 | | | | | | | | | | |
| ACETONE | ppm | .7 | | .6 | | .6 | | | | | | | | | | |
| 2-PROPANOL | ppm | 22.2 | | 24.1 | | .4 | | | | | | | | | | |
| METHYLENE CHLORIDE | ppm | ND | | ND | | .5 | | | | | | | | | | |
| TOLUENE | ppm | ND | | .4 | | .1 | | | | | | | | | | |
| TRANS-1,2 DIMETHYLCYCLOHEXANE | ppm | .5 | | ND | | ND | | | | | | | | | | |
| 1-BUTANOL | ppm | .7 | | ND | | ND | | | | | | | | | | |
| | | | | | | | | | | | | | | | | |
| LAB | | MP | BE | MP | BE | MP | BE | BE | BE | BE | BE | BE | BE | BE | BE | BE |

* RESULTS FOR CH₂ AND CO DETERMINED BY THE MP LAB WERE CONVERTED FROM ppb TO % VOLUME. IN SOME CASES THE NUMBER WAS TRUNCATED AND ROUNDED OFF.

BE – BOEING ENVIRONMENTAL LAB
MP – MATERIALS AND PROCESS LAB
ND – NONE DETECTED

5-8116-0-205

62

Table 9. Molecular sieve gas analyses (continued)

| TIME OF DAY | | SAMPLED AUGUST 7, 1989 | | | | | | | SAMPLED AUGUST 8, 1989 | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | 11:42 | 11:45 | 12:00 | 12:04 | 12:05 | 12:15 | 15:40 | 16:00 | 16:02 | 16:15 | 16:27 | 16:45 | 16:49 |
| ANALYTES | UNITS | PRODUCT $CO_2$ | PRODUCT $CO_2$ | INLET | INLET | OUTLET | OUTLET | PRODUCT $CO_2$ | INLET | OUTLET | INLET | OUTLET | PRODUCT $CO_2$ | PRODUCT $CO_2$ |
| $CO_2$ | % VOL | 77.9 | | 3.72 | | .25 | | 77.3 | .37 | .12 | | | 86.2 | |
| $H_2$ | % VOL | <.4 | | <.4 | | <.4 | | 1.0 | <.4 | <.4 | | | 3.6 | |
| $N_2$ | % VOL | 22.5 | | 74.6 | | 77.4 | | 9.8 | 77.9 | 78.0 | | | 5.6 | |
| $O_2$ | % VOL | 6.6 | | 21.7 | | 22.5 | | 2.3 | 22.4 | 22.5 | | | 1.7 | 3.2 |
| $H_2O$ VAPOR | % VOL | | .96 | | 1.4 | | .66 | | | | .11 | .54 | | |
| $CH_4$ | % VOL | <.04 | | <.04 | | <.04 | | 3.9 | <.04 | <.04 | | | 5.55 | |
| $CO$ | % VOL | <.2 | | <.2 | | <.2 | | 5.9 | <.2 | <.2 | | | <.2 | |
| | | | | | | | | | | | | | | |
| LAB | | BE | BE | BE | BE | BE | BE | BE | BE | BE | BE | BE | BE | BE |

BE – BOEING ENVIRONMENTAL LAB

5-8117-0-205

63

Table 10. Static feed electrolysis gas analyses.

| ANALYTES* | UNITS | SAMPLED AUGUST 3, 1989 | | | | | SAMPLED AUGUST 4, 1989 | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 12:35 | 12:37 | 13:17 | 13:15 | 13:16 | 15:05 | 15:06 | 15:30 | 15:45 |
| | | $O_2$ LINE | $O_2$ LINE | $H_2$ LINE | $H_2$ LINE** | $H_2$ LINE* | $O_2$ LINE | $O_2$ LINE | $H_2$ LINE | $H_2$ LINE** |
| $CO_2$ | % VOL | .2 | | | .6 | < .04 | | | | < .04 |
| $H_2$ | % VOL | ND | | | 69.0 | 81.2 | | | | 78.7 |
| $N_2$ | % VOL | 16.4 | 15.6 | | 23.0 | 33.6 | 25.6 | | | 41.0 |
| $O_2$ | % VOL | 82.4 | 85.7 | | 6.4 | 9.5 | 75.6 | | | 10.9 |
| $H_2O$ VAPOR | % VOL | | | .4 | | | | .54 | .42 | |
| $CH_4$ | % VOL | ND | | | ND | < .04 | | | | < .04 |
| CO | % VOL | ND | | | ND | < .2 | | | | < .2 |
| TRACE ORGANICS | | | | | | | | | | |
| ACETONE | ppm | .2 | | | ND | | | | | |
| LAB | | MP | BE | BE | MP | BE | BE | BE | BE | BE |

*THE BE LAB COULD NOT DETERMINE $CO_2$, CO, AND $H_2$ IN THE $O_2$ LINE BECAUSE OF INSTRUMENT LIMITATIONS.
**SEE SECTION 4.6 FOR EXPLANATION OF RESULTS

BE - BOEING ENVIRONMENTAL LAB
MP - MATERIALS AND PROCESS LAB
ND - NONE DETECTED

5-8121-0-205

Table 10. Static feed electrolysis gas analysis (continued)

| TIME SAMPLE TAKEN | | SAMPLED AUGUST 7, 1989 | | | | SAMPLED AUGUST 8, 1989 | | |
| --- | --- | --- | --- | --- | --- | --- | --- | --- |
| | | 10:49 | 10:50 | 11:15 | 11:17 | 14:25 | 15:30 | 15:35 |
| ANALYTES* | UNITS | $O_2$ LINE | $O_2$ LINE | $H_2$ LINE** | $H_2$ LINE | $O_2$ LINE | $H_2$ LINE** | $H_2$ LINE |
| $CO_2$ | % VOL | | | .02 | | | <.04 | |
| $H_2$ | % VOL | | | 72.4 | | | 90.2 | |
| $N_2$ | % VOL | 27.4 | | 60.6 | | | 1.2 | |
| $O_2$ | % VOL | 73.4 | .94 | 16.5 | .54 | .75 | .7 | .41 |
| $H_2O$ VAPOR | % VOL | | | <.04 | | | <.04 | |
| $CH_4$ | % VOL | | | | | | | |
| CO | % VOL | | | <.2 | | | <.2 | |
| LAB | | BE | BE | BE | BE | BE | BE | BE |

* THE BE LAB COULD NOT DETERMINE $CO_2$, CO, AND $H_2$ IN THE $O_2$ LINE BECAUSE OF INSTRUMENT LIMITATIONS

** SEE SECTION 4.6 FOR EXPLANATION OF RESULTS.

BE – BOEING ENVIRONMENTAL LAB

5-8122-0-205

Table 11. Bosch gas analyses.

| TIME SAMPLE TAKEN | | SAMPLED 8/2/89 | SAMPLED AUGUST 3, 1989 | | SAMPLED AUGUST 4, 1989 | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| ANALYTES | UNITS | NR | 11:33 | 11:32 | 10:00 | 10:45 | 11:45 | 12:15 | 13:50 | 15:50 |
| | | PURGE LINE | REACTOR INLET | REACTOR INLET | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | REACTOR INLET | REACTOR INLET |
| $CO_2$ | % VOL | 95.1 | 3.5 | .7 | | | .9 | 1.5 | 5.2 | |
| $H_2$ | % VOL | <.4 | 23.1 | 14.4 | | | 11.0 | 13.8 | 20.3 | |
| $N_2$ | % VOL | 6.7 | 29.0 | 46.9 | | | 39.6 | 37.1 | 35.5 | |
| $O_2$ | % VOL | 1.8 | 3.4 | 4.8 | | | 5.4 | 6.9 | 5.4 | |
| $H_2O$ VAPOR | % VOL | | | | .85 | | | | | 1.7 |
| $CH_4$ | % VOL | <.04 | 33.3 | 32.1 | | | 42.1 | 40.9 | 32.1 | |
| CO | % VOL | <.2 | 5.9 | ND | | | ND | <.2 | 8.9 | |
| AMMONIA | ppm | | | | | 3.6 | | | | |
| TRACE ORGANICS | ppm | | | ND | | | ND | | | |
| LAB | | BE | BE | MP | BE | BE | MP | BE | BE | BE |

BE – BOEING ENVIRONMENTAL LAB
MP – MATERIALS AND PROCESS LAB
ND – NONE DETECTED

5-8118-0-205

66

Table 11. Bosch gas analyses (continued)

| TIME SAMPLE TAKEN | | SAMPLED AUGUST 7, 1989 | | | | | SAMPLED AUGUST 8, 1989 | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | 12:30 | 12:49 | 13:47 | 15:28 | 15:29 | 13:10 | 13:36 | 17:11 | 17:15 |
| ANALYTES | UNITS | BOSCH BLEED | BOSCH BLEED | BOSCH BLEED | REACTOR INLET | REACTOR INLET | BOSCH BLEED | BOSCH BLEED | REACTOR INLET | REACTOR INLET |
| $CO_2$ | % VOL | 2.0 | | | 2.4 | | 3.1 | | 96.7 | |
| $H_2$ | % VOL | 14.8 | | | 21.7 | | 18.2 | | 19.2 | |
| $N_2$ | % VOL | 33.6 | | | 21.6 | | 16.3 | | 11.7 | |
| $O_2$ | % VOL | 7.2 | | | 2.4 | | .9 | | 1.0 | |
| $H_2O$ VAPOR | % VOL | | | .35 | | 1.5 | | | | 2.1 |
| $CH_4$ | % VOL | 46.7 | | | 46.2 | | 53.8 | | 25.7 | |
| $CO$ | % VOL | 3.8 | | | 5.9 | | 6.6 | | 0 | |
| | | | | | | | | | | |
| AMMONIA | ppm | | 1.3 | | | | | .7 | | |
| | | | | | | | | | | |
| LAB | | BE | BE | BE | BE | BE | BE | BE | BE | BE |

BE – BOEING ENVIRONMENTAL LAB

5-8119-0-205

67

Table 12. Bosch $CO_2$ reduction water analyses.

| ANALYTES | UNITS | SAMPLED AUGUST 4, 1989 | | | | SAMPLED AUGUST 6 1989 |
|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 02:23 - 14:30 | 14:30 - 19:00 | 19:55 | 21:06 | 21:31 |
| | | BOSCH WATER | BOSCH WATER | BOSCH WATER | BOSCH WATER | BOSCH WATER |
| pH | | | | 7.2 | | |
| TOTAL SOLIDS | ppm | | | | <10 | |
| TOTAL DISSOLVED SOLIDS | ppm | | | | <10 | |
| TOTAL SUSPENDED SOLIDS | ppm | | | | <10 | |
| TURBIDITY | NTU | | | 9.6 | | |
| DISSOLVED $O_2$ | ppm | | | 2.02 | | |
| BARIUM | ppm | <.01 | | | | |
| CADMIUM | ppm | <.03 | | | | |
| CALCIUM | ppm | .15 | | | | |
| CHROMIUM | ppm | <.06 | | | | |
| IRON | ppm | <.05 | | | | |
| MAGNESIUM | ppm | <.01 | | | | |
| MANGANESE | ppm | <.01 | | | | |
| MOLYBOENUM | ppm | .09 | | | | |
| NICKEL | ppm | <.11 | | | | |
| SILVER | ppm | <.14 | | | | |
| ZINC | ppm | <.02 | | | | |
| AMMONIA | ppm | | | | | 92.60 |
| CHLORIDE | ppm | | | | | .10 |
| FLORIDE | ppm | | | | | .27 |
| NITRATE | ppm | | | | | <.120 |
| PHOSPHATE | ppm | | | | | <.190 |
| POTASSIUM | ppm | | | | | 1.03 |
| SULFATE | ppm | | | | | <.190 |
| HETEROTROPHS | CFU/100ml | | 10,000 | | | |
| GRAM NEGATIVE | CFU/100ml | | 7,500 | | | |
| GRAM POSITIVE | CFU/100ml | | 100 | | | |
| YEAST AND MOLD | CFU/100ml | | <100 | | | |
| LAB | | BE | BE | BE | BE | BE |

BE - BOEING ENVIRONMENTAL LABORATORY
CFU - COLONY FORMING UNITS

5-8123-0-205

68

Table 13. TCCS analyses.

| ANALYTES | UNITS* | SAMPLED JULY 31, 1989 | | | | SAMPLED AUGUST 3, 1989 | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| TIME OF SAMPLE TAKEN | | 09:14 | 09:15 | 09:18 | 09:19 | 09:35 | 09:45 | 11:02 | 11:02 | 11:05 | 11:05 |
| | | PORT 6 | PORT 6 | PORT 7 | PORT 7 | PORT 6 | PORT 7 | PORT 6 | PORT 6 | PORT 7 | PORT 7 |
| $H_2$ | % VOL | <.4 | ND | <.4 | ND | ND | ND | ND | <.4 | ND | <.4 |
| $CH_4$ | % VOL | <.04 | .00250 | <.04 | .00162 | .003021 | ND | | <.04 | .00892 | <.04 |
| CO | % VOL | <.2 | ND | <.2 | ND | ND | ND | ND | <.2 | ND | <.2 |
| $CO_2$ | % VOL | .06 | <.1 | .06 | <.1 | | | <.1 | .09 | <.1 | .11 |
| $N_2$ | % VOL | 78.1 | 78.5 | 78.7 | 78.5 | | | 78.2 | 78.7 | 77.6 | 77.7 |
| $O_2$ | % VOL | 22.1 | 20.5 | 22.2 | 20.4 | | | 20.7 | 22.4 | 21.4 | 22.3 |
| TRACE ORGANICS | | | | | | | | | | | |
| FREON 113 | ppm | | .2 | | .3 | | | ND | | ND | |
| ACETONE | ppm | | .1 | | .4 | | | ND | | .2 | |
| 2-PROPANOL | ppm | | ND | | .3 | | | ND | | ND | |
| TRANS-1,2 DIMETHYLCYCLOHEXANE | ppm | | ND | | .1 | | | ND | | ND | |
| TOLUENE | ppm | | ND | | .1 | | | ND | | ND | |
| BUTYL ACETATE | ppm | | ND | | .1 | | | ND | | ND | |
| O-XYLENE | ppm | | ND | | .1 | | | ND | | ND | |
| TRICHLOROETHYLENE | ppm | | ND | | ND | | | ND | | ND | |
| LAB | | BE | MP | BE | MP | MP | MP | MP | BE | MP | BE |

*METHANE RESULTS REPORTED BY THE MP LAB WERE CONVERTED FROM PPM TO % VOLUME.

BE – BOEING ENVIRONMENTAL LAB
MP – MATERIALS AND PROCESS LAB
ND – NONE DETECTED

5-8113-0-205

Table 13. TCCS analyses (continued)

| ANALYTES | UNITS* | 06:57 PORT 6 | 06:59 PORT 7 | 12:32 PORT 6 | 12:32 PORT 6 | 12:34 PORT 7 | 12:36 PORT 7 | 16:32 PORT 6 | 16:42 PORT 7 |
|---|---|---|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | | | | | | | | |
| | | | | SAMPLED AUGUST 4, 1989 | | | | | |
| $H_2$ | % VOL | <.4 | <.4 | ND | <.4 | ND | <.4 | ND | ND |
| $CH_4$ | % VOL | .13 | .05 | .45439 | .17 | .09561 | <.04 | .320413 | .069459 |
| CO | % VOL | <.2 | <.2 | .01716 | .2 | .00261 | <.2 | .014712 | .002428 |
| $CO_2$ | % VOL | .08 | .18 | <.1 | .09 | .1 | .18 | | |
| $N_2$ | % VOL | 78.1 | 78.4 | 77.4 | 78.2 | 79.2 | 78.2 | | |
| $O_2$ | % VOL | 22.2 | 22.2 | 21.1 | 22.4 | 19.7 | 22.2 | | |
| TRACE ORGANICS | | | | | | | | | |
| FREON 113 | ppm | | | 2.0 | | 0.8 | | | |
| ACETONE | ppm | | | .4 | | 0.4 | | | |
| 2-PROPANOL | ppm | | | ND | | ND | | | |
| TRANS-1,2 DIMETHYLCYCLOHEXANE | ppm | | | ND | | ND | | | |
| TOLUENE | ppm | | | .2 | | ND | | | |
| BUTYL ACETATE | ppm | | | ND | | ND | | | |
| O-XYLENE | ppm | | | ND | | ND | | | |
| TRICHLOROETHYLENE | ppm | | | .5 | | ND | | | |
| LAB | | BE | BE | MP | BE | MP | BE | MP | MP |

*METHANE AND CARBON MONOXIDE REPORTED BY THE MP LAB WERE CONVERTED FROM PPM TO % VOLUME.

ALL NUMBERS WERE RETAINED.

BE – BOEING ENVIRONMENTAL LAB

MP – MATERIALS AND PROCESS LAB

ND – NONE DETECTED

5-8114-0-205

Table 13. TCCS analyses (continued)

| ANALYTES | UNITS* | SAMPLED AUGUST 5, 1989 | | SAMPLED AUGUST 6, 1989 | | SAMPLED AUGUST 7, 1989 | | SAMPLED AUGUST 8, 1989 | | SAMPLED AUGUST 10, 1989 | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 08:18 | 08:26 | 08:15 | 08:28 | 15:24 | 15:25 | 17:34 | 17:37 | 10:57 | 11:01 |
| | | PORT 6 | PORT 7 | PORT 6 | PORT 7 | PORT 6 | PORT 7 | PORT 6 | PORT 7 | PORT 6 | PORT 7 |
| $H_2$ | % VOL | <.3 | ND | ND | ND | <.4 | <.4 | <.4 | <.4 | ND | ND |
| $CH_4$ | % VOL | .269305 | .083523 | .019921 | .014449 | .16 | .07 | <.04 | <.04 | .273594 | .047010 |
| CO | % VOL | .013546 | .002318 | .002457 | .001343 | <.2 | <.2 | <.2 | <.2 | .014135 | .002644 |
| $CO_2$ | % VOL | | | | | .09 | .23 | .08 | .09 | | |
| $N_2$ | % VOL | | | | | 77.2 | 77.6 | 77.9 | 77.7 | | |
| $O_2$ | % VOL | | | | | 22.4 | 22.3 | 22.5 | 22.5 | | |
| LAB | | MP | MP | MP | MP | BE | BE | BE | BE | MP | MP |

* METHANE AND CARBON MONOXIDE RESULTS REPORTED BY
THE MP LAB WERE CONVERTED FROM ppm TO % VOLUME.

BE – BOEING AEROSPACE LAB
MP – MATERIALS AND PROCESS LAB
ND – NONE DETECTED

5-8115-0-205

71

Table 14. Environmental gas samples.

| ANALYTES | UNITS | SAMPLED AUGUST 3, 1989 | | | | SAMPLED AUGUST 7, 1989 | | SAMPLED AUGUST 8, 1989 | |
|---|---|---|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 15:50 | 15:51 | 16:10 | 16:10 | 14:03 | 14:18 | 13:15 | 13:30 |
| | | BUILDING ATMOS. | BUILDING ATMOS. | MODULE ATMOS. | MODULE ATMOS. | BUILDING ATMOS. | MODULE ATMOS. | BUILDING ATMOS. | MODULE ATMOS. |
| $N_2$ | % VOL | 78.7 | 78.7 | 78.5 | 78.7 | 77.0 | 77.4 | 77.0 | 76.9 |
| $O_2$ | % VOL | 22.0 | 20.2 | 22.2 | 20.3 | 22.5 | 22.5 | 22.3 | 22.4 |
| $CO_2$ | % VOL | .08 | <.1 | <.08 | <.1 | .04 | .13 | .18 | .05 |
| $H_2$ | % VOL | <.4 | ND | <.4 | ND | <.4 | <.4 | <.4 | <.4 |
| $CH_4$ | % VOL | <.04 | ND | <.04 | ND | <.04 | <.04 | <.04 | <.04 |
| CO | % VOL | <.2 | ND | <.2 | ND | <.2 | <.2 | <.2 | <.2 |
| TRACE ORGANICS | | | | | | | | | |
| FREON 113 | ppm | | .9 | | .7 | | | | |
| ACETONE | ppm | | .3 | | .2 | | | | |
| 2-PROPANOL | ppm | | .5 | | .4 | | | | |
| | | | | | | | | | |
| | | | | | | | | | |
| LAB | | BE | MP | BE | MP | BE | BE | BE | BE |

BE – BOEING ENVIRONMENTAL LAB
MP – MATERIALS AND PROCESS LAB
ND – NONE DETECTED

5-8125-0-205

Table 15. Temperature and humidity control analyses.

| ANALYTES | UNITS | SAMPLED AUGUST 3, 1989 | | | | | SAMPLED AUGUST 4, 1989 | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| TIME SAMPLE TAKEN | | 14:35 | 14:40 | 14:50 | 15:10 | 15:20 | 13:30 | 14:00 | 14:40 | 14:47 |
| | | | | | THC CONDENSATE | | | THC CONDENSATE | | |
| BNA's | | | | | | | | | | |
| PHENOL | ppb | | | | 20 | | | 20 E | | |
| OTHER: (ALL UNITS ppb)[1] | | | | | | | | | | |
| 2 - BUTOXY ETHANOL | | | | | 20 E | | | 200 E | | |
| OCTAMETHYL – | | | | | | | | 40 E | | |
| CYCLOTETRASILOXANE | | | | | | | | 40 E | | |
| BENZYL ALCOHOL | | | | | 30 E | | | 120 E | | |
| 2 - PHENYL - 2 - PROPANOL | | | | | | | | 40 E | | |
| 2 - ETHYLHEXANOIC ACID | | | | | 180 E | | | 40 E | | |
| 2 - (2 - BUTOXYETHOXY) ETHANOL | | | | | | | | 40 E | | |
| DECANOIC ACID | | | | | | | | 40 E | | |
| DODECANOIC ACID | | | | | | | | 80 E | | |
| UNKNOWN ALCOHOL | | | | | 170 E | | | 40 E | | |
| (2 - ETHOXYETHOXY ETHANOL) | | | | | | | | | | |
| UNKNOWN ALKYL CARBOXYLIC ACID | | | | | 40 E | | | 160 E | | |
| UNKNOWN HYDROCARBON | | | | | | | | 40 E | | |
| LAB | | | | | BE | | | BE | | |

BE – BOEING ENVIRONMENTAL LAB
BNA's – BASE NEUTRAL EXTRACTABLES
E – ESTIMATE ONLY – ESTIMATES ARE BASED ON THE ION CURRENT OF AN INTERNAL STANDARD.

5-8126-0-205

Table 15. Temperature and humidity control analyses (continued)

| TIME SAMPLE TAKEN | | 07:45 | 07:50 | 08:00 | 08:05 | SAMPLED AUGUST 8, 1989 — 12:50 | 14:13 | 14:19 | 16:10 |
|---|---|---|---|---|---|---|---|---|---|
| ANALYTES | UNITS | THC CONDENSATE | THC CONDENSATE | THC CONDENSATE | THC CONDENSATE | THC CONDENSATE | THC CONDENSATE | THC CONDENSATE | THC CONDENSATE |
| pH | | | | 6.8 | | | 6.5 | | |
| CONDUCTIVITY | UM HOS | | | 58.8 | | | 54.2 | | |
| TOTAL PHENOLS | ppb | .17 | | | | .16 | | | |
| VOLATILE ORGANICS | ppb | | | | | | | | |
| ACETONE | ppb | | | | 157 | | | | 1000 |
| TOLUENE | ppb | | | | TR | | | | |
| ETHYL BENZENE | ppb | | SEE NEXT PAGE | | TR | | | SEE NEXT PAGE | |
| OTHER[1] | | | | | | | | | |
| ETHANOL | ppb | | | | 100 E | | | | 20 E |
| 2 - PROPANOL | ppb | | | | 100 E | | | | |
| 2 - ETHYL - 1 - HEXANOL | ppb | | | | 50 E | | | | |
| | | | | | | | | | |
| LAB | | BE | | BE | BE | BE | BE | | BE |

BE – BOEING ENVIRONMENTAL LAB
E – ESTIMATE ONLY – ESTIMATES ARE BASED ON THE ION CURRENT OF AN INTERNAL STANDARD.
TR – TRACE

74

5-8128-0-205

Table 16. MP analytical methods/gas samples.

| ANALYTE | REFERENCE | METHOD TYPE* | DETN. LMTS. |
|---|---|---|---|
| $N_2$ | IN HOUSE | MS | 0.5% |
| $O_2$ | | MS | 0.5% |
| $CO_2$ | | MS | 0.5% |
| $H_2$ | | MS | 0.5% |
| $CH_4$ | | MS–(GC/CR) | 0.5%–(0.5-1 ppm) |
| CO | | GC | 0.5%/0.5-1 ppm |
| **TRACE ORGANICS** | | | **RANGE** |
| FREON 113 | IN HOUSE | GC/FID | 0.2 – 1 ppm |
| METHYLENE CHLORIDE | | GC/FID | |
| TRICHLOROETHYLENE | | GC/FID | |
| ACETONE | | GC/FID | |
| TRANS 1, 2, DIMETHYLCYCLOHEXANE | | GC/FID | |
| TOLUENE | | GC/FID | |
| O-XYLENE | | GC/FID | |
| BUTYL ACETATE | | GC/FID | |
| 2–PROPANOL | | GC/FID | |
| 1–BUTANOL | | GC/FID | |
| | | GC/FID | |

ACRONYMS:
MS – MASS SPECTROSCOPY
GC/CR – GAS CHROMATOGRAPHY CATALYTIC REACTOR DETECTOR
GC/FID – GAS CHROMATOGRAPHY FLAME IONIZATION DETECTOR

Table 17. BE analytical methods/gas samples.

| ANALYTE | REFERENCE | METHOD TYPE [1] | DETN. LMTS. |
|---|---|---|---|
| **ORGANICS** | | | |
| $N_2$ | IN HOUSE | GC/TCD | .2% (2000 ppm) |
| $O_2$ | ↓ | ↓ | .2% (2000 ppm) |
| $CO_2$ | | | .2% (2000 ppm) |
| $H_2$ | | | .4% (4000 ppm) |
| $CH_4$ | | | .04% (400 ppm) |
| $H_2O$ VAPOR | | GRAVAMETRIC | (22–88) ppm[2] |

1. GC/TCD – GAS CHROMATRGRAPHY WITH THERMAL
   CONDUCTIVITY DETECTOR

2. THE RANGE GIVEN REPRESENTS THE DETECTION LIMITS FOR 14 TO 57 LITERS
   OF GAS SAMPLED.

manpower, lack of equipment, or lack of analytical method and expertise. The following is a list of the additional parameters requested which the BE laboratory could not determine.

| Parameter | Sample Description |
|---|---|
| Alkalinity (carbonates) | Bosch Product Water |
| Formic Acid | Bosch Product Water |
| Formaldehyde | Bosch Product Water |
| Total Phenols | Bosch Product Water |
| Total Keljdal Nitrogen | Bosch Product Water |
| Total Organic Carbon (TOC) | Bosch Product Water |
| Total Inorganic Carbon (TIC) | Bosch Product Water |
| Total Carbon (TC) | Bosch Product Water |
| NO, $NO_2$ | Bosch Bleed |
| TOC, TIC, TC, $CO_2$, CO, $H_2$ | Humidity Condensate SFE $O_2$ Line |
| Trace Organics | Bosch, TCCS, MS, module and building gas samples |

The organic volatiles and base/neutral extractables reported by the BE laboratory for humidity condensate (Table 12) were submitted for informational purposes only. The compounds listed are only those compounds which were found to be greater than the detection limit. These compounds, determined by gas chromatography/mass spectrometer (GC/MS) detection, were acquired by a new chemist in training and the GC/MS was reported to be improperly configured. The compounds reported in Table 12 should be considered as minimal values only. Other organic compounds not listed in this report were reported as below the method detection limit and could have possibly been present if holding time and GC/MS configuration had been in compliance.

76

Table 18. BE analytical methods/water samples.

| ANALYTE | REFERENCE[2] | | METHOD TYPE[1] | DETN. LMTS. |
|---|---|---|---|---|
| **PHYSICALS** | | | | |
| pH | EPA | 150.1 | ELECTROMETRIC | 1-14 pH UNITS |
| COLOR | EPA | 110.2 | COLORIMETRIC | 1 PT/CO |
| TURBIDITY | EPA | 180.1 | SPECTROMETRIC | 0.5 NTU |
| CONDUCTIVITY | EPA | 120.1 | ELECTROMETRIC | 0.1 umhos/cm |
| DISSOLVED O | SM | 10030 | ELECTROMETRIC | |
| TOTAL DIS. SOLIDS | EPA | 160.2 | GRAVIMETRIC | 10 ppm[4] |
| TOTAL SOLIDS | EPA | 160.3 | GRAVIMETRIC | 10 ppm[4] |
| TOTAL SUSP. SOLIDS | EPA | 160.1 | GRAVIMETRIC | 10 ppm[4] |
| **ELEMENTS** | | | | |
| BARIUM | EPA | 200.7 | ICP | 0.002 pm |
| CADMIUM | EPA | 200.7 | ICP | 0.010 pm |
| CALCIUM | EPA | 200.7 | ICP | 0.001 pm |
| CHROMIUM | EPA | 200.7 | ICP | 0.010 pm |
| IRON | EPA | 200.7 | ICP | 0.020 pm |
| MAGNESIUM | EPA | 200.7 | ICP | 0.005 pm |
| MAGANESE | EPA | 200.7 | ICP | 0.001 pm |
| MOLYBDENUM | EPA | 200.7 | ICP | 0.020 pm |
| SILVER | EPA | 200.7 | ICP | 0.030 pm |
| ZINC | EPA | 200.7 | ICP | 0.010 pm |
| **IONS** | | | | |
| AMMONIA | EPA | 300.7 | IC | 0.090 pm |
| CHLORIDE | EPA | 300.0 | IC | 0.075 pm |
| FLUORIDE | EPA | 300.0 | IC | 0.05 pm |
| NITRATE | EPA | 300.0 | IC | 0.25 pm |
| PHOSPHATE | EPA | 300.0 | IC | 0.38 pm |
| POTASSIUM | EPA | 300.7 | IC | 0.050 pm |
| SULFATE | EPA | 300.0 | IC | 0.38 pm |

Table 18. BE analytical methods/water samples (continued)

| ANALYTE | REFERENCE[2] | METHOD TYPE[1] | DETN. LMTS. |
|---|---|---|---|
| **ORGANICS** | | | |
| VOLATILES (39 COMPOUNDS) | EPA 624 | GC/MS | (5-150) ppb[3] |
| BASE NEUTRAL ACID EXTRACTABLES (51 COMPOUNDS) | EPA 625 | GC/MS | 20 ppb |
| TOTAL PHENOLS | HACH | SPECTROMETRIC | 20 ppb |
| **MICROBIOLOGICALS** | | | |
| HETEROTROPHS BY PCA | SM 16th ed | MEMBRANE FILTRATION | 0 CFU/100 ml[4] |
| GRAM NEGATIVE BY MAC | SM 16th ed | MEMBRANCE FILTRATION | 0 CFU/100 ml[4] |
| GRAM POSITION BY (PEA) | SM 16th ed | MEMBRANE FILTRATION | 0 CFU/100 ml[4] |
| YEAST AND MOLD | SM 16th ed | MEMBRANE FILTRATION | 0 CFU/100 ml[4] |

1.  IC    – ION CHROMATOGRAPHY
    ICP   – INDUCTIVELY COUPLED PLASMA EMMISION SPECTROSCOPY
    GC/MS – GAS CHROMATOGRAPHY WITH MASS SPECTROMETER DETECTOR

2.  EPA – ENVIRONMENTAL PROTECTION AGENCY
    SM  – STANDARD METHODS

3.  THE DETECTION LIMITS DETERMINED FOR MOST OF THE VOLATILE ORGANICS RANGED FROM 5 TO 150 ppb.  TO SEE ACTUAL DETECTION LIMITS SEE APPENDIX.

4.  DETECTION LIMITS ARE DEPENDENT ON SAMPLE VOLUME.

## 4.5 Quality Control

Quality control (QC) is a program which continually monitors the reliability (accuracy and precision) of results reported and signals when an analysis is out of control. Out of control indicates that a problem has occurred in the laboratory relative to instrument operation, analytical procedure, analyst error etc., which will yield incorrect or erroneous results. Implementing QC in the laboratory is time consuming and expensive, but is necessary to insure reliable results. The following is a general discussion of the QC data submitted by the BE laboratory for air and water analyses. The MP laboratory did not submit QC data.

For gas analyses, the only QC submitted by the BE laboratory was calibration verification control charts. Once instrument calibration was completed, the calibration was verified using purchased gas standards which were either from a different lot number or different manufacture from the standards initially used to calibrate the instrument. The concentrations of the calibration verification standards were mid range on the calibration curve. Calibration was verified and considered acceptable if the percent recovery for the calibration verification standards were within 90 to 110 percent of the known concentration. The following table gives the low and high percent recoveries of the standards used to verify calibration. The numbers given are estimates taken from the calibration verification control charts. The control charts indicate that the instrument used to evaluate gas samples was correctly calibrated. The calibration verification control charts do not indicate accuracy or precision for the method used, but they do indicate proper instrument calibration. After instrument calibration and calibration verification, the calibration was checked every eight hours using a check standard to detemine if instrument drift was occurring. If the check standard varied greater than ± 10 percent from the known value, the instrument had to be recalibrated and calibration verified before gas samples could be analyzed.

| Gas | Calibration Verification (Low–High) % Recovery |
|---|---|
| $CO_2$ | 100 – 106 |
| CO | 97 – 103 |
| $H_2$ | 97 – 105 |
| $CH_4$ | 98 – 104 |
| $N_2$ | 98 – 99 |
| $O_2$ | 97 – 111 |

The BE laboratory submitted calibration verification, accuracy, and precision control charts for analytes (metals, ions, total phenols, and TOC) determined in water samples. In general, calibration verification control charts indicated that instrumentation was properly calibrated. All recoveries were within ± 10 percent except verification standards determined for cadmium and zinc which demonstrated one out of control condition each. This indicated that instrument recalibration was necessary before further water analyses could be initiated.

The accuracy control charts plot the percent recoveries of matrix spike determinations. These charts are used to indicate matrix interferences and out of control conditions. An out of control condition for accuracy exists when the percent recovery for any one matrix spike exceeds the

upper and lower control limits (±3 standard deviation) from the mean percent recovery. The accuracy control charts indicate that the BE laboratory systems were never out of control. As an indication of accuracy, the following table lists the accuracy range in percent recovery for ±2 standard deviation from the mean value. The BE laboratory did not identify which matrix spikes were run with the water samples tested.

| Analyte | Accuracy % Recovery |
|---|---|
| Barium | 88.5 – 117.4 |
| Cadmium | 75.2 – 108.4 |
| Calcium | 88.3 – 126.9 |
| Chromium | 65.0 – 101.8 |
| Copper | 82.6 – 109.2 |
| Iron | 74.3 – 116.3 |
| Magnesium | 90.3 – 114.9 |
| Manganese | 88.9 – 110.2 |
| Molybdenum | 66.5 – 97.5 |
| Nickel | 79.9 – 106.1 |
| Potassium | 79.8 – 129.4 |
| Silver | 66.8 – 114.0 |
| Sodium | 56.4 – 142.6 |
| Zinc | 84.2 – 118.2 |
| Ammonia | 51.6 – 111.9 |
| Total Phenols | 46.1 – 145.9 |

The concentration of matrix spikes determined should be considered when interpreting accuracy data. For example, if a method of analysis detected 7.5 ppm out of a known 10 ppm standard, 75 percent of the true value is recovered with a difference of only 2.5 ppm. If 75 ppm is detected out of a known 100 ppm standard, 75 percent of the true value is again recovered, but the difference is 25 ppm, a much larger difference. The percent recovery of 75 percent is the same for both determinations, but the actual differences between the determined and known values vary greatly. As analyte concentrations approach the detection limit, the relative uncertainty of the result increases. Therefore, uncertainty near the detection limit should be considered when evaluating data.

Precision for matrix spike duplicates was determined and expressed as RPD. This is a measure of the mutual agreement between two samples assumed to have the same concentration of analyte spiked. RPD is defined in the following equation:

$$RPD = |X1 - X2|/X1 + X2 * 200$$

where:

$X1$ = First duplicate result

$X2$ = Second duplicate result

Precision control charts were plotted against the upper control limit ($+3$ standard deviation) to indicate out of control conditions. These charts indicated that the BE laboratory was always in control. Precision for each analyte is listed in the following table. The range given represents $+2$ standard deviation from the mean RPD.

| Analyte | Precision RPD |
|---|---|
| Barium | 5.8 |
| Calcium | 8.2 |
| Cadmium | 7.8 |
| Chromium | 12.1 |
| Copper | 11.1 |
| Iron | 8.0 |
| Magnesiuum | 5.8 |
| Manganese | 7.0 |
| Molybdenum | 11.6 |
| Nickel | 11.3 |
| Silver | 10.6 |
| Zinc | 15.9 |
| TOC | 19.2 |

## 4.6 Discussion of Results

Gas results from the SFE $H_2$ line samples indicated possible sample contamination either during or after sample collection. Oxygen and nitrogen concentrations were found in a 1 to 4 ratio in the $H_2$ line samples which is the same ratio found in air. Because the SFE subsystem does not produce nitrogen or oxygen in the hydrogen product, contamination is suspected. Hydrogen may also be diffusing through the sample gas bag therefore increasing the concentrations of the other gases detected. Although hydrogen leakage could be occurring, this would not account for the oxygen to nitrogen ratios of 1 to 4. In addition, nitrogen was also detected in the SFE $O_2$ line samples in significant concentrations. Because the SFE subsystem does not produce nitrogen in the oxygen product, contamination is again suspected.

In two separate cases, gas sampling bags deflated to the extent that either the entire sample was lost or a portion of the analysis could not be completed. An SFE $O_2$ gas bag deflated entirely prior to analysis. The cause of this problem could be the result of a defective bag or not fully closing the inlet valve to the sample bag.

Continued improvement in analytical methods and detection techniques is needed in many areas. Measuring water vapor by using silica gel is a technique which the BE laboratory used for the first time during the SIT. The concentration of water vapor determined in the product $CO_2$ line of the 4BMS subsystem was higher than expected. Because the product $CO_2$ gas is passed through a desiccant prior to exiting the subsystem, water vapor should not be present.

Because of limitations associated with the flame ionization detector (FID) used to detect gases, low concentrations of hydrogen could not be determined in high concentrations of oxygen. The SFE $O_2$ line samples could not be analyzed for low concentrations of hydrogen. In order to detect low concentrations of hydrogen, large volumes of the gas sample would have to be injected on the gas chromatograph which could damage the FID.

Matrix effects or analyte loss could account for the low mean percent recovery of ammonia, chromium, and molybdenum in Bosch water matrix spikes. Because of the volatility of ammonia, quick sample turn around time is required to prevent sample loss. Matrix effects may account for the low chromium and molybdenum spike recoveries, but these effects are presently unknown.

## 4.7 Recommendations/Lessons Learned

The gas sampling techniques used for the SIT should be verified to eliminate concerns of sample bag leakage, diffusion, and the introduction of contaminants (air) during the sample process. In addition, gas leakage and diffusion need to be studied as a function of gas type and storage time prior to analysis.

The gravimetric method used to measure water vapor in a gas should be verified so that accuracy and precision can be determined. Currently, the validity of this method is questionable until its reliability can be determined.

An alternate analytical method of detection needs to be defined for analyzing low concentrations of hydrogen (ppm range) in high concentrations of oxygen. Because of instrument limitations, the BE laboratory could not analyze for hydrogen in the SFE $O_2$ stream samples taken during the SIT.

In general, analytical laboratory expertise, instrumentation, and manpower is needed to further accomplish the goals of future integrated subsystem tests. All new analytical methods and techniques should be verified prior to actual sample analyses so that accuracy and precision can be defined.

## 5.0 CONCLUSIONS/LESSONS LEARNED

There were several important lessons learned and conclusions drawn from the phase III SIT. First, the integration of the Bosch $CO_2$ reduction subsystem in the air revitalization system requires careful consideration of sensitive operational interfaces. In particular, the $CO_2$ supplied from the

4BMS must be controlled to a relatively constant supply pressure and flow rate. The $CO_2$ collected by the 4BMS should be relatively pure (98 percent or better) if the required operational interfaces with the 4BMS and TCCS are to be maintained.

Second, perhaps more attention should be paid to the deterioration of each subsystem as a function of time. Leakage problems seriously affected the entire test (see reference 5 for a detailed discussion). The 4BMS used in the phase III SIT was four years old. Minor leakage problems had occurred during phase II testing, however they can become a major factor as subsystems age.

Finally, several instruments, particularly flowmeters, gave unexpected results which were believed to have been erroneous. Most of the instrumentation used was calibrated for a pure gas and most of the gases produced by the subsystems are not pure, therfore inaccurate readings are likely caused by calibration errors. For example, the $H_2$ flowmeter for the SFE indicated a conflicting amount of $H_2$ was being produced for the amount of water being used by the subsystem. The unexpected reading from the flowrate could have been caused by a slight amount of water vapor mixed with the $H_2$ in the line.

The next integrated tests of these same air revitalization subsystems will be the metabolic control test, the extended metabolic control test, and the transient control test. The lessons learned and conclusions drawn from the simplified integrated test have given some insight for better integrated operation during these future tests.

# REFERENCES

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3. "Space Station CMIF Extended Duration Metabolic Control Test Final Report." NASA TM-100362, March 1989.

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# PHASE III SIT INSTRUMENTATION

## FOUR-BED MOLECULAR SIEVE INSTRUMENTATION

| NUM | DESCRIPTION | ENGU | NUM | DESCRIPTION | ENGU |
|---|---|---|---|---|---|
| MD01 | POWER | DISCR | MT07 | REC AIR OUT TEMP | DEGF |
| MD02 | HOLD | DISCR | MT08 | DES BED 3 INLET | DEGF |
| MD03 | RUN | DISCR | MT09 | DES BED 2/4 OUT | DEGF |
| MD04 | MODE 1A | DISCR | MT10 | DES BED 2 HTR | DEGF |
| MD05 | MODE 1B | DISCR | MT11 | DES BED 4 HTR | DEGF |
| MD07 | MODE 2 | DISCR | MT12 | BLOWER INLET | DEGF |
| MD08 | MODE 3A | DISCR | MT13 | BED 1 TEMP | DEGF |
| MD09 | MODE 3B | DISCR | MT14 | BED 3 TEMP | DEGF |
| MD11 | MODE 4 | DISCR | MT16 | DWNSTR VAC PUMP | DEGF |
| MD12 | CO2 TANK HI PRES | DISCR | XT03 | AIR RETURN TEMP | DEGF |
| MD13 | SB HI TEMP WARN | DISCR | XD01 | MOL S AIR IN DEW | DEGF |
| MT02 | BLOWER OUT TEMP | DEGF | MDP1 | ADSORB DES BD DP | DEGF |
| MT03 | AD BED 1 INLET | DEGF | MDP2 | INLET AIR DEW | DEGF |
| MT04 | AD BED 3 INLET | DEGF | XF06 | COOLANT FLOW | GPM |
| MT05 | DES BED 1 INLET | DEGF | XT08 | COOLANT IN TEMP | DEGF |
| XT02 | AIR IN TEMP | DEGF | XT07 | COOLANT OUT TEMP | DEGF |
| MV01 | SB BED 2 VOLTS | VLTS | FG01 | CMS CO2 CONC | MMHG |
| MV02 | SB BED 4 VOLTS | VLTS | XG02 | INLET AIR PCO2 | MMHG |
| MP09 | CO2 HOLD TANK | PSIA | XG03 | OUTLET AIR PCO2 | MMHG |
| MP01 | BLOWER OUTLET | PSIG | XF02 | CO2 TANK VENT FL | #/HR |
| MP02 | BED 1 IN PRESS | PSIG | XF03 | INLET AIR FLOW | #/HR |
| MP03 | BED 3 IN PRES | PSIG | XF05 | CO2 OUT FLOW | #/HR |
| MP04 | PURGE BED 1 | PSIG | MF01 | CO2 FLOW BED 2/4 | #/HR |
| MP05 | SORB BED 2/4 IN | PSIG | FP02 | CMS PRESSURE | PSIA |
| MP07 | PURGE DES BED 3 | PSIG | XP03 | MS CO2 OUT PRESS | PSIA |
| MP08 | DESORB BED 2/4 | PSIA | XP07 | MS GN2 PRESS | PSIG |
| MP10 | DNSTRM VAC PUMP | PSIA | MO01 | O2 UPSTRM CO2 TK | % |
| MP11 | PRESS AIR INLET | PSIG | XG01 | MOL S O2 IN CO2 | % |
| XP02 | MS AIR IN PRES | PSIA | MF01 | CO2 FLOW BED 2/4 | #/HR |
| XT06 | MS CO2 OUT TEMP | DEGF | | | |
| *FG99 | COMB GAS | %LEL | | | |
| *FG98 | COMB GAS | %LEL | | | |
| *FG97 | COMB GAS | %LEL | | | |
| *FG96 | COMB GAS | %LEL | | | |
| *XW01 | MS HEATER | WTTS | | | |
| *XW02 | MS RELAY PWR | WTTS | | | |
| *XW03 | MS AC POWER | WTTS | | | |

* Denotes subsystem internal measurement.

# STATIC FEED ELECTROLYSIS INSTRUMENTATION

| NUM | DESCRIPTION | ENGU | NUM | DESCRIPTION | ENGU |
|---|---|---|---|---|---|
| EP03 | SFE H2O IN PRES | PSIG | EF06 | SFE COOLANT FLOW | GPM |
| ET07 | SFE H2O IN TEMP | DEGF | ET05 | SFE COOLANT TEMP | DEGF |
| HF08 | SFE DI H2O FLOW | CC/M | ET06 | SFE COOL OUT TEM | DEGF |
| EC01 | SFE H2O IN COND | MMHG | EE07 | SFE CHIL H2O FLW | GPM |
| EP01 | SFE O2 VENT PRES | PSIG | EP06 | SFE GN2 IN PRESS | PSIG |
| EP02 | SFE O2 PRO ACC P | PSIG | *EW01 | FCA MOTOR 1 VPI | % |
| EF01 | SFE O2 VENT FLOW | SLPM | *ETT2 | O2 OUTLET TEMP | DEGF |
| ET01 | SFE O2 OUT TEMP | DEGF | *ETT3 | PCA TEMP | DEGF |
| ED01 | SPE O2 OUT DP | DEGF | *EX02 | SFE STATUS | |
| EG02 | SFE H2 IN O2 VNT | PPM | *EV13 | MODULE VOLTAGE | VLTS |
| EP05 | SFE H2 OUT PRESS | PSIA | *EI01 | MODULE CURRENT | AMPS |
| EF03 | SFE H2 OUT FLOW | SLPM | *ETT1 | COOLANT TEMP | DEGF |
| ET02 | SFE H2 OUT TEMP | DEGF | *EPP1 | H2 PRESS | PSIG |
| ED02 | SFE H2 OUT DP | DEGF | *EPP2 | H2-O2 DELTA P | PSID |
| EG03 | SFE O2 IN H2 OUT | % | *EPP3 | CCA OUTLET | PSID |
| EG01 | SFE HOOD H2 CONC | % | *EPP4 | H2O TNK DELTA P | PSID |

# TCCS INSTRUMENTATION

| NUM | DESCRIPTION | ENGU | NUM | DESCRIPTION | EGNU |
|---|---|---|---|---|---|
| JI01 | CAT OXIDIZER I | AMPS | JF01 | TCCS FLOW | CFM |
| JT01 | CAT OXIDIZER T | DEGF | | | |
| JP01 | RGEN BED DIFF P | "H2O | | | |
| JP02 | FIXED BED DIFF P | "H2O | | | |
| JD01 | FIXED BED FAN | | | | |
| JD02 | CAT OXID HTR | | | | |
| JD03 | CAT OXID OVR/TEM | | | | |
| JD04 | CAT OXID OVR/I | | | | |
| JD05 | +15 VCD MON | | | | |
| JD06 | -15 VCD MON | | | | |

* Denotes subsystem internal measurement.

# BOSCH SUBSYSTEM INSTRUMENTATION

| NUM | DESCRIPTION | ENGU | NUM | DESCRIPTION | ENGU |
|---|---|---|---|---|---|
| *WR02 | REACTOR 2 IN VPI | | *WV06 | REAC OUT SEL VAL | |
| *WR03 | REACTOR1 OUT VPI | | *WV07 | BYPASS RET VALVE | |
| *WR04 | REACTOR2 OUT VPI | | *WV08 | RECYC LOOP VALVE | |
| *WL01 | COND LEV ID FULL | | *WV09 | WATER DRAIN VALV | |
| *WL02 | COND LEV ID EMPT | | *WV10 | VENT SELECT VALV | |
| *WD02 | GAS COMPOSITION | | *WT07 | TOTAL OPER TIME | HRS |
| *WD03 | REACTOR 1 HEATER | | *WT08 | TIME SINCE S/D | HRS |
| *WD04 | REACTOR 2 HEATER | | *WT09 | TIME OF LAST S/D | HRS |
| *WD05 | COMPRESSOR MOTOR | | *WT10 | TIME IN NORMAL | HRS |
| *WD06 | WATER PUMP | | *WT11 | TIME IN SHUTDOWN | HRS |
| *WA01 | COMP SPEED ACT. | | *WT13 | REAC1 T CONT ON | HRS |
| *WV01 | H2 REACT. VALVE | | *WT14 | TIME REAC1 HT ON | HRS |
| *WV02 | CO2 REACT. VALVE | | *WT15 | REAC2 T CONT ON | HRS |
| *WV03 | PURGE VALVE | | *WT16 | TIME REAC2 HT ON | HRS |
| *WV04 | BYPASS CONT VALV | | *WT17 | TIME COMPRES ON | HRS |
| *WV05 | REAC IN SEL VALV | | *WT18 | TIME H2O PUMP ON | HRS |
| WT02 | BOSCH H2 IN TEMP | DEGF | *WF99 | H2 FEED GAS FLOW | CC/M |
| XT05 | MS-TSA CO2 TEMP | DEGF | *WH01 | RECYC H2 COMP | % |
| WT01 | BOSCH BLEED TEMP | DEGF | *WD01 | EXT. SHUT DOWN | |
| WT03 | BCH H2 VENT TEMP | DEGF | *WP01 | INLET PRESSURE | PSIA |
| WP06 | BOSCH H2 IN PRES | PSIA | *WP99 | PURGE PRESSURE | PSIA |
| XP06 | MS-TSA CO2 PRESS | PSIA | *WP03 | REACTOR PRESSURE | PSIA |
| WP02 | BOSCH-TCCS PRESS | PSIA | *WP04 | REACTOR PRESSURE | PSIA |
| WP05 | BCH H2 OUT PRESS | PSIA | *WP98 | H2 INLET PRESS. | PSIA |
| WF03 | BOSCH H2 IN FLOW | SLMP | *WW99 | COMPRESSOR SPEED | RPM |
| XF04 | MOL S TSA CO2 FL | SLPM | *WT01 | REACTOR #1 TEMP | DEGF |
| WF02 | BOSCH-TCCS FL | SLPM | *WT98 | REACTOR #1 HTR T | DEGF |
| WF01 | BOSCH COOLANT FL | GPM | *WT97 | REACTOR #2 TEMP | DEGF |
| WF06 | BCH H2 OUT FLOW | SLPM | *WT04 | REACTOR #2 HTR T | DEGF |
| WG01 | BCH HOOD H2 CONC | % | *WT05 | COOLANT OUT TEMP | DEGF |
| | | | *WT06 | COOLANT IN TEMP | DEGF |
| | | | *WR01 | REACTOR 1 IN VPI | |

* Denotes subsystem internal measurement.

# PHASE III SIT FACILITY INSTRUMENTATION

| NUM | DESCRIPTION | ENGU | NUM | DESCRIPTION | ENGU |
|---|---|---|---|---|---|
| TT01 | SUPPLY AIR TEMP | DEGF | FD01 | CMS DEW POINT | DEGF |
| TF02 | SUPPLY AIR FLOW | CFM | FT01 | CMS DRY BULB | DEGF |
| TT02 | BY-PASS TEMP | DEGF | FP02 | CMS PRESSURE | PSIA |
| TF01 | BYPASS FLOW | CFM | FG03 | CMS H2 SENSOR | |
| TT05 | THCS CHX OUT TEM | DEGF | | | |
| TT06 | THCS CHX IN TEMP | DEGF | | | |
| TT03 | CHW IN TEMP | DEGF | | | |
| TT04 | CHW OUT TEMP | DEGF | | | |
| TF03 | CHILLED RETURN | GPM | | | |
| TP02 | FAN DELTA | "H2O | | | |
| TP03 | FILTER DELTA | "H2O | | | |

\* Denotes subsystem internal measurement.

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**16. Abstract**

This report is a description of the phase III simplified integrated test (SIT) conducted at the Marshall Space Flight Center (MSFC) Core Module Integration Facility (CMIF) in 1989. This was the first test in the phase III series integrated environmental control and life support systems (ECLSS) tests. The basic goal of the SIT was to achieve full integration of the baseline air revitalization (AR) subsystems for Space Station Freedom. Included in this report is a description of the SIT configuration, a performance analysis of each subsystem, results from air and water sampling, and a discussion of lessons learned from the test. Also included in this report is a full description of the preprototype ECLSS hardware used in the test.

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