Design and Fabrication of a Hydrogen Detection Calibration System

Contract NAS8-11999

FINAL REPORT

September 8, 1967

Submitted to: George C. Marshall Space Flight Center
Huntsville, Alabama 35812

Submitted by: Aero Vac Corp.
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Troy, New York 12181

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SCOPE OF WORK AND GENERAL RESULTS

Scope of Work

The scope of work included the design, fabrication, testing, delivery and installation of a Hydrogen Detection Calibration System which would meet or exceed all specifications set forth in the subject contract under Al "Statement of Work".

Results of Contract Effort

Aero Vac Corp. delivered and installed at G.M.S.F.C., Huntsville, Alabama, during the period July 21 thru July 25, the equipment specified in the contract "Statement of Work". Included in this report are pertinent results of the engineering study required to meet all the contract specifications.

DETAIL RESULTS

Engineering activities were directed to the design of three major sub-components and the interfacing of each to complete the Hydrogen Calibration System. These sub-components were:

1. Instrumentation and Gas Admittance Control System
2. Vacuum Chamber and Pumping System
3. Environmental Cooling and Heating System

Of primary concern was that in each of the sub-components either pure hydrogen, or in some modes of operation, hydrogen/oxygen mixtures would deliberately be introduced. In the event of accidental leakage into the test facility, hydrogen/air mixtures could be introduced into the Environmental Heating and Cooling System. Thus, "Design of Safety" was of prime importance.

Safety

Preliminary work was devoted to obtaining design criteria for equipment operating in hydrogen/oxygen environments and also thermodynamic, corrosive, and gas handling data of hydrogen/oxygen mixtures.

Within the confines of the subject vessel, two processes may occur in the ignition of a flammable mixture. Once a flammable mixture is ignited, the resulting flame, if not extinguished, will either attach itself to the ignition source or propagate from it. If it propagates from the source, the propagation rate will be either subsonic (deflagration) or supersonic (detonation) relative to the unburned gas. If it is subsonic, the pressure will equalize at the speed of sound throughout the enclosure in which combustion is taking

place so that the pressure drop across the flame (reaction) front will be relatively small. If the rate is supersonic, the rate of pressure equalization will be less than the propagation rate and there will be an appreciable pressure drop across the flame front. Moreover, with most combustible air mixtures, at ordinary temperatures, the ratio of the peak to initial pressure within the enclosure will seldom exceed 8:1 in the former (deflagration), but may be more than 40:1 in the latter (detonation) case. With a sufficiently powerful ignition source, the detonation may occur immediately upon ignition, even in the open. However, the ignition energy required to initiate a detonation is usually many orders of magnitude greater than that required to initiate a deflagration.

Based on the above and other information obtained in references 1,2,3,4, and 5, the vacuum chamber was designed to withstand an overpressure of 37 atmospheres. This pressure corresponds to the maximum measured overpressure of a reflected shock wave if a mixture of 62% hydrogen and 38% oxygen initially at one atmosphere pressure is ignited and detonation occurs.

To prevent the buildup of pressure within the vacuum vessel, a rupture disk was chosen. References 4 and 6 and consultation with one of the foremost manufacturers of rupture disks were utilized to correctly size the disk.

Radiographs were taken of the pressure vessel girth weld and the circumferential weld of the head to cylinder. The radiographs are being forwarded under separate cover. Inspection using NAV SHIPS Specification 250-1500, as a requirement for allowable porosity, revealed no faults sufficient for rejection.

The need to prevent the overpressurization of the vessel was dictated by the allowable pressure in the mechanical vacuum pump. While the reservoir of the pump will withstand approximately 200 psi, it is reported that the shaft oil seals will be damaged at approximately 35 to 40 psi. Accordingly, the burst specifications on the disk were set at 18-20 psig. In order to minimize the direct confrontation of the pump to a detonation wave, three (3) 90° bends were used in the vacuum piping.

The choice of vacuum pump fluid was based on the need for oxidation resistance, low flammability and lubricity. The choice of Cellulube #220*, in part, was based on its being certified (#194574 Bu Ships) as an approved fluid for Aircraft Carrier catapult systems.

All motors are explosion proof; however, no manufacturer provides an explosion proof motor for operation in a hydrogen/oxygen environment which conforms to the applicable code requirements. Therefore, a source of gaseous nitrogen is introduced into the sealed electrical control box and thence purges the explosion proof motors by passing through the "liquitite" conduit which distributes power conductors to the motors, heaters and the liquid nitrogen solenoid valve.

Hydrogen/oxygen mixtures have an Auto Ignition temperature of 400° C. In order to stay below this temperature, the box containing the electrical heater elements in the Environmental Cooling and Heating System has a temperature sensitive explosion proof switch set at 500° F. In the event of blower drive belt breakage, the loss of circulating air over the heaters will result in a temperature rise causing the temperature detector switch to disconnect power to the heater elements.

*Cellulube is a trade name of the Stauffer Chemical Company.

Operating Instructions

See Appendix

Installation Test Results

The cognizant M.S.F.C. personnel witnessed the performance of the Environmental Heating and Cooling System through the full temperature excursion from -120° C to +77° C. Also, instruction was given to M.S.F.C. personnel in the proper calibration procedure for gas flowraters, and demonstrations of dynamic and static gas admittance procedures were exhibited.

EXPLOSIVE TEST AND RESULTS

To assure complete safety to personnel while working in the proximity of the equipment, an explosive test was performed at the Wyle Laboratories (Huntsville, Alabama) facility.

Test Objective

To determine the ability of the vacuum vessel to maintain its integrity in the event that during normal use an accidental explosion occurs.

Test Procedure

1. With the vessel at atmospheric pressure, note reading of bourdon tube pressure gauge.
2. Ready all strain gauge instrumentation. Obtain base line data. Note: Zero will shift as the vessel is evacuated, but will be of no significance since back-filling will return readings to their original readings.
3. With foreline valve open and gas admittance valve closed, start vacuum pump. Pump for the total amount of time as determined in "Preparation for Test 3 d."
4. Close foreline valve tight.
5. Stop vacuum pump.
6. While observing bourdon tube pressure gauge on foreline, admit from a regulated supply through the gas admittance valve a sufficient quantity of hydrogen to raise the pressure to 18.5" Hg vacuum.

7. Remove hydrogen source from test area.

8. Again, while observing pressure gauge, admit from a regulated supply through the gas admittance valve a sufficient quantity of oxygen to raise the pressure to 0.0" Hg (in other words, one atmosphere). Note: The mixture in the volume is now 62% H_2 by volume in oxygen at a total pressure of 0.0" Hg.

9. Close gas admittance valve tight.

10. Remove oxygen source from test area.

11. Start recorders and "Fastax" camera.

12. Ignite mixture.

13. Provide Aero Vac with all records and film from camera for evaluation.

Explosive Test Results

As evidenced by the enclosed affidavit, no defects or other detrimental effects of the explosion could be found.

The 400 foot of film, taken by Fastax camera at a rate of 6000 frames per second, is forwarded under separate cover. Viewing of this film shows approximately 10 to 12 frames of conditions at initiation and during explosion. The film shows the test success.

CERTIFICATION TEST REPORT

WYLE LABORATORIES HUNTSVILLE FACILITY
1800 Governors Drive West, Huntsville, Alabama 35800

Aero-Vac Corporation
P. O. Box 448
Troy, New York

WYLE JOB NO. 41129
CUSTOMER P.O. NO. 13983
CONTRACT NAS 11999
MANUFACTURER Aero-Vac Corporation
DATE 31 July 1967

1. SPECIMEN Hydrogen Calibration System Vacuum Chamber
2. PART NO. N/A 3. SERIAL NO. 683-135
4. EQUIPMENT USED

APPARATUS	MFG.	MODEL NO.	ACCURACY	DATE OF LAST CALIB.
See Attached List				

5. REQUIREMENTS

To determine the ability of the vacuum vessel to maintain its integrity in the event that, during normal use, an accidental explosion occurs.

6. PROCEDURES AND RESULTS

The vacuum chamber was pumped down to a pressure of less than 100 microns. The chamber was then back-filled with a pre-determined mixture of gaseous hydrogen and oxygen. Four strain gauges were mounted on the outside of the vacuum chamber for the purpose of measuring strain during the explosion. A high-speed movie camera (6000 frames per second) was used during the test to observe any structural failure during the explosion.

Upon completion of the test, the chamber was visually inspected. There was no evidence of damage or permanent deformation detected during the above inspection except for the burst disc that ruptured as anticipated.

STATE OF ALABAMA)
COUNTY OF MADISON) ss

William W. Holbrook, being duly sworn, deposes and says: That the information contained in this report is the result of complete and carefully conducted tests and is to the best of his knowledge true and correct in all respects.

SEAL

William W. Holbrook

SUBSCRIBED and sworn to before me this 1 day of August, 1967

Notary Public in and for the County of Madison, State of Alabama.

My Commission expires Oct. 14, 1968

TEST BY John H. Kennedy

PROJ. ENGINEER Glenn Dishner

CUSTOMER WITNESS

QAR DCAS

QUALITY CONTROL

WYLE LABORATORIES, INC.

INSTRUMENTATION

DATE 7-19-67 JOB NO. 41129 TEST AREA REVETMENT

TECHNICIAN J. REESE CUSTOMER AERO V. TYPE TEST EXPLOSION

No.	Instrument	Manufacturer	Model No.	Serial No.	Wyle or Gov't No.	Range	Accuracy	Calibration	
								On	Due
1	VISICORDER	HONEYWELL	1508		81218	0-2Kc	± 5%	3-9-67	PRIOR TO USE
2	MAG. TAPE RECORDER	P.I	PI-214		91519	0-5Kc	± .5DB	PRIOR TO	USE
3	BALANCE UNIT	WYLE	6		N/A	0-2Kc	± 2%	3-8-67	PRIOR TO USE
5	D.C. AMPLIFIER	E/I	A20B		93709	0-20Kc	± 2%	5-1-67	11-1-67
6	D.C. AMPLIFIER	E/I	A20B		93710	0-20Kc	± 2%	5-1-67	11-1-67
7	D.C. AMPLIFIER	E/I	A20B		93704	0-20Kc	± 2%	5-1-67	11-1-67
8	DC. AMPLIFIER	E/I	A20B		93705	0-20Kc	± 2%	5-1-67	11-1-67
9	STRAIN GAGE	BUDD	C9-144		N/A	1500 MICROSTRAIN	± 2%	PRIOR TO	USE
10	STRAIN GAGE	DUDD	C9-144		N/A	1500	± 2%	PRIOR TO	USE
11	STRAIN GAGE	DUDD	C9-144		N/A	1500	± 2%	PRIOR TO	USE
12	STRAIN GAGE	DUDD	C9-144		N/A	1500	± 2%	PRIOR TO	USE
13	OSCILLOSCOPE	H/P	321A		7-200-626		± 1%	5-18-67	8-18-67
14	DECADE RESISTOR	G.R.C.	41662		90483	100KΩ	± .1%	5-24-67	11-24-67
	Note: Due to the A.C. ignition system supplied by Aero-Vac the strain gauge readings recorded on the oscillograph records were distorted beyond normal reduction capabilities								

INSTRUMENT TEST ENGINEER D. DeLucchi CHECKED & RECEIVED BY John Kinisch

W-456

REFERENCES

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2. Michael G. Zabetakis, "Flammability Characteristics of Combustible Gases and Vapors", Bulletin 627, 1965, Bureau of Mines, United States Department of Interior.

3. D. L. Ward, D. G. Pearce, D. J. Merrett, "Liquid Hydrogen Explosions in Closed Vessels", Atomic Energy Research Establishment, Harwell, England.

4. Merl D. Creech, "Combustion Explosions in Pressure Vessels Protected with Rupture Disks", Transactions of the A.S.M.E. October 1941.

5. A.S.M.E. Boiler and Pressure Vessel Code Unfired Pressure Vessels - Section VIII.

6. Benjamin Block, "Emergency Venting for Tanks and Reactors", Chem. Eng., Jan 22, 1966, pp 111-116.

APPENDIX

OPERATING INSTRUCTIONS

Hydrogen Calibration System

Model S.O. 1326

July 1967

AERO VAC CORP. TROY, NEW YORK A/C 518 274-5850

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1.0 GENERAL DESCRIPTION

The Hydrogen Detection Calibration System consists of a vacuum chamber, a mechanical vacuum pump and valving, an environmental heating and cooling system and a gas admittance, flow measurement and quantitative gas calibration system.

The equipment is capable of admitting and measuring hydrogen, air and oxygen in quantities of 100 ppm (parts per million) to 15% hydrogen in air or oxygen over a pressure range of 0.1 torr to 760 torr in the vacuum chamber.

The environmental heating and cooling system is capable of controlling the vacuum chamber temperature through the complete range from -120° C to +77° C.

All motors and electrical control boxes are explosion proof and further purged with nitrogen gas in order to preclude explosions in the event that explosive mixtures of hydrogen and oxygen are present in the test area. The mechanical vacuum pump is charged with a special oil which has the property of being compatible with hydrogen and oxygen. A further precaution is taken by purging and venting the pump exhaust with gaseous nitrogen.

2.0 SAFETY PROVISIONS

2.1 Vacuum Chamber

The vacuum chamber was designed to withstand an internal explosive pressure of 500 psi. Further protection from overpressure due to a reflected shock from a detonation, as opposed to an explosion, is provided by a 20 psig rupture disc installed in a four inch vent line on the top of the chamber. This line should be vented out of the test building during installation in a manner prescribed by the cognizant safety officer at the installation site.

2.2 Mechanical Vacuum Pump

Since it is not possible to purchase an explosion proof vacuum pump, certain features were designed and incorporated into the mechanical vacuum pump system. Foremost, the foreline is so constructed that a shock wave must traverse three 90° bends prior to entering the pump casing; however, after and if the shock traverses the first 90° bend, the shock front is then in line with a 1-1/2" diameter, 20 psig rupture disc. Further protection is offered by a boiler plate enclosure on three sides and the top of the pump.

The mechanical pump is charged with Cellulube #220*. This fluid, Cellulube #220, should be used as a make-up for oil losses. This fluid is approved by the U.S. Bureau of Mines. The fluid is an approved lubricant for the pump, has an auto-ignition temperature (ASTM D286-30) of 1150° F and a Fire Point, Cleveland open cup (ASTM D92-56) of 655° F. CAUTION: Use only Cellulube #220 in the mechanical pump.

*Cellulube is a trade name of the Stauffer Chemical Company

2.3 Electrical

All motors are explosion proof; however, no manufacturer provides an explosion proof motor for operation in a hydrogen-oxygen environment which conforms to the applicable code requirements. Therefore, a source of gaseous nitrogen is introduced into the sealed electrical control box, and thence purges the explosion proof motors by passing through the liquitite conduit which distributes power conductors to the motors, heaters and liquid nitrogen solenoid valve.

The box containing the electrical heater elements has a temperature sensitive, explosion proof switch factory preset at 500° F. In the event of blower drive belt breakage, the loss of circulating air over the heaters will result in a temperature rise causing the temperature detector switch to disconnect power to the heater elements.

2.4 Instrument Console

Operating personnel may perform their work functions from this enclosure. The rear of the enclosure is covered by a shield to protect the instrumentation plumbing from damage.

3.0 CHAMBER AND PUMPING SYSTEM

3.1 Chamber Description

3.1.1 The chamber is a 20" O.D. by 24" long horizontally oriented vessel with a removable work table located internally.

3.1.2 Electrical feedthrus are located on a flanged penetration on the left side of the chamber. Eight pins rated at 5 amps each are provided.

3.1.3 The gas admittance line and chamber pressure measuring line are located on a flanged penetration on the right side of the chamber. The gas admittance line is centrally located and terminates at the flange while the pressure measuring line extends five inches beyond the flange into the chamber.

3.1.4 Access to the chamber is provided by a full opening hinged door. The hinge is removable so that the front insulated enclosure may be attached when it is planned to operate the chamber at temperatures above or below room ambient.

3.1.5 The access door can be sealed by either of two means.

3.1.5.1 When test operation is planned to extend below -130° F (-54.5° C), a gold wire O-ring is used. The gold wire O-ring is formed from 99.99% purity gold, full annealed, with a cut length of 66-23/32" and a cross sectional diameter of .025". The joint is formed by butt welding the cut ends by ordinary acetylene or hydrogen torch methods.

3.1.5.2 In order to effect a vacuum tight seal when the gold wire of section 3.1.5.1 is used, a bolt torque of 40 foot pounds should be used on the 24 chamber door flange. The suggested procedure to insure uniform closure is to torque each 7th bolt starting in either a clockwise or counter clockwise direction from any arbitrary bolt assigned number one. Lubricate nut face and nut threads; apply torque to nut to insure proper bolt stress level. Bolts and nuts are special high strength material (A-286) to withstand force of accidental explosion and to match the thermal expansion characteristics of the type 304L flange material.

3.1.5.3 For operation between -54.5° C and +77° C, a silicone O-ring is provided.

3.1.5.4 Lubrication of the silicone O-ring is recommended in extremely light quantities with Cellulube #220 (the mechanical pump oil). Place only a few drops in the palm of the hand. Then draw the O-ring through the palm of the hand.

3.1.6 The rupture disc and vacuum support located in the 4" nominal diameter pipe will need replacement only in the event of an accidental overpressure by admitting gas from the calibration source or explosion of the gas mixture. Spare vacuum support and rupture disc ordering information are located in Section 9.0.

3.2 Pumping System Description

3.2.1 The vacuum chamber is pumped by a two stage rotary mechanical pump which has a speed of 15 cfm (7 liters/sec) at a pressure of 760 torr. It is charged with Cellulube #220

principally for its oxidation resistance, and high fire and auto ignition qualities (see Section 2.2). Since mixtures of hydrogen and oxygen are to be pumped, the pump exhaust must be vented outside of the test area as prescribed by the cognizant safety officer at the site. The volume above the reservoir of oil in the pump is purged with gaseous nitrogen to dilute the hydrogen and oxygen mixture to proportions that are not explosive. A blast shield encloses the pump on three sides and the top. The open side is away from personnel and the instrumentation console.

3.2.2 Two valves are provided in a parallel arrangement between the mechanical pump and the chamber. The larger valve is provided for rapid evacuation and dynamic testing (as described in Section 7.0), while the smaller valve has a speed limiting orifice inserted in the ball. The orifice is .093 inches in diameter and provides a pumping speed ratio of 10:1.

3.2.3 Protection from overpressure to the mechanical pump is provided by a rupture disc and rupture disc support in the 1-1/2" section of the foreline between the valving and the chamber. Spare vacuum support and rupture disc ordering information are located in Section 9.0.

4.0 ENVIRONMENTAL HEATING AND COOLING

4.1 General Description

The environmental enclosure insulation is contained within thin sheets of stainless steel which are formed to enclose the vacuum chamber. Flow splitters are attached to the inner surface to cause the cooling or heating medium to re-circulate from a blower, through a heater, through the chamber/insulation interspace and return to the blower. All insulation is non-hygroscopic.

4.1.1 Cooling of the chamber is effected by a controlled injection of liquid nitrogen (LN_2) through a spray nozzle into a diffusing duct immediately downstream of the blower discharge. The LN_2 changes to the gas phase giving up latent heat and sensible heat to effect the cooling. Approximately 170 pounds of LN_2 are required to cool the chamber (and associated ducting and insulation) from 30° C ambient to -120° C. The holding requirement at -120° C is approximately 1 pound per minute.

4.1.2 Heating of the chamber is effected by an array of four (4) 2000 watt, 220V heaters wired in a series parallel arrangement to yield a total of 2000 watts of heat. The blower recirculates room air through the same flow paths as in the cooling cycle.

4.1.3 Control of the chamber temperature is provided continuously over the temperature range of -120° C to +77° C by a "read through" type temperature controller with thermocouple sensing. Changing of the "Cooling" mode to "Heating" mode is effected by switch operation on the control panel.

4.1.4 Control accuracy as measured during pre-delivery tests was as follows:

- Low Set Point -120° C - Less than ± 1° C

- High Set Point +77° C - Less than ± 1° C

Further data indicated a difference between gas inlet and gas outlet temperature of 6° C which is indicative of the temperature uniformity of the vacuum chamber.

5.0 CONTROL SYSTEM

5.1 Mechanical Pump

The mechanical pump motor is controlled by a pair of oil-tight pushbuttons on the electrical control panel. The red illuminated pushbutton indicates a pump "running"condition. Overload protection is provided by current sensitive heaters in the motor starter contactor. In the event an overload condition occurs, the hermetic and purged contactor/control box must be opened and the motor starter reset button operated.

Venting of the vacuum chamber or gas nitrogen purging to void the vacuum system of water vapor is accomplished by operation of either valve located on the right rear of the control panel. The gas nitrogen supply to this valving system should be regulated to a pressure of 3 to 5 psig.

5.2 Blower Motor

The blower motor is controlled similarly to the mechanical pump motor and also with the red illuminated pushbutton indicating a running condition. (Note that blower drive belt breakage is not indicated except by an overtemperature condition when in the "Heating" mode.)

5.3 Heating and Cooling System

The desired temperature is set on the temperature controller. The Heat or Cool switch is placed in the position corresponding to above or below ambient and the cycle initiated by actuation of the "Blower Start" pushbutton.

5.4 The "Heat/Cool" switch has an intermediate position which interrupts power to both the LN_2 solenoid valve and power to the heaters, yet maintaining blower operation. This can enable the user to effect a cooldown of the chamber from the +77° C set point to ambient temperature rapidly without the use of liquid nitrogen by removal of the front insulated enclosure.

5.5 All fuses are contained within the gas purged hermetic control enclosure.

6.0 INSTRUMENTATION SYSTEM

6.1 Instrumentation System General

The gas measurement system incorporates the pressure gauges, flow meters, calibration equipment and control valving to enable the user to precisely measure and admit as few as 100 ppm of hydrogen in air or oxygen or up to 15% hydrogen in air or oxygen at pressures from 0.1 torr to 760 torr (atmospheric pressure).

6.2 Chamber pressure is measured by a precision bourdon tube pressure gauge in the range of 760 torr to 10 torr. For lower pressures, an electronic capacitance type manometer measures pressure in the range of 100 torr to 10 microns full scale on the most sensitive range.

6.3 Hydrogen flow is measured with visual indicating type flowrators. Three flowrators are used to cover the range of hydrogen gas requirements.

6.4 Oxygen or air flow is measured with visual indicating type flowrators. Separate flow circuits are used for each of the two gases, each with a single flowrator in the circuit.

6.5 Calibration of the flowrators is performed by use of a volumetric displacement device. By observing the displacement of a mercury sealed piston and measuring the time interval required for this displacement, the volume per unit time, or flow rate, is determined. Two different sizes are used for calibrating the three hydrogen flowrators while identical calibrators are used for the oxygen and air flowrators.

7.0 OPERATING PROCEDURE

7.1 Gas source requirements to cover the entire range from 100 ppm to 15% hydrogen in air or oxygen are listed below. The impurity content in the case of items one, two and three is stated so that the overall accuracy of the calibration system is sufficient to determine the mixture content in the chamber to ± 3%. Similarly the special mixed gases of items four and five have percentage tolerances in order to meet the stated chamber mixture content analysis of ± 3%.

Item	Gas
1	100% Hydrogen - max impurity 25 ppm
2	100% Air - max impurity 1 ppm of H_2
3	100% Oxygen - max impurity 1 ppm of H_2
4	.4% ± .01% Hydrogen in air
5	.4% ± .01% Hydrogen in oxygen

The premixed gases of item 4 and 5 are to be used when the mixture ratio of hydrogen in oxygen or air is to be tested in the range from 100 ppm to 4000 ppm. When higher percentages of hydrogen in oxygen or air are desired, then the 100% hydrogen source (item one) is used.

7.2 Two modes of operation are required to cover the range of chamber test pressures. The following table shows the minimum and maximum operating conditions over the range of pressures and range of mixture ratios.

	Mode	Chamber Pressure (torr)	Hydrogen	Air or Oxygen
I	Static	20 to 760	100 ppm to 4000 ppm	Balance
II	Static	20 to 760	4000 ppm to 15%	Balance
III	Static or Dynamic	0.1 to 20	100 ppm to 4000 ppm	Balance
IV	Static or Dynamic	0.1 to 20	4000 ppm to 15%	Balance

The static mode is used in the interest of conserving gas and to minimize the number of flowrators to cover the range of flow which would be required. Both static or dynamic test conditions may be used in Mode III or IV. However, outgassing rates at these pressures and the attendent problem of maintaining a stable chamber pressure indicate that a dynamic condition is more suitable.

7.3 Test Procedure for Setting Static Condition

7.3.1 If the chamber temperature condition is to be at any temperature other than room ambient, the chamber should be pumped to its ultimate pressure. Further, it is advisable that all flow paths through the flowrators be opened and to the pressure gauges P1 and P2. CAUTION: Never open the valves to the calibrators unless P1 and P2 show greater than atmospheric pressure. Specifically, the following valves should be open to evacuate all the plumbing downstream of the gas admittance valves (V1, V16, V17).

OPEN - V3, V4, V5, V7, V8, V9, V10, V20, V22 (red handles)

OPEN - V13, V15, V19, V21 (green handl s)

OPEN - V11 (black handle)

DO NOT OPEN - V2, V6, V12, V18, V23

7.3.2 Start blower and set temperature control for desired temperature.

7.3.3 After chamber temperature has stabilized, the gas test conditions may be set.

7.3.4 An example, assuming a test condition follows:

$$P_{chamber} = 500 \text{ torr}$$

Mixture 15% H_2; 85% O_2

From the table of 7.2 this defines a static test. At this point, the oxygen/air side of the control console and the hydrogen side should be isolated from each other and the chamber. This is done by closing V13, V15, V21 and V11.

Since a flowrator is not required in a static mode, V3, V4, V5, V7, V8 and V22 can be closed.

Calculate, as follows, the pressure to which the chamber must be raised with hydrogen (assume local ambient barometer of 760 torr):

$$ppH_2 = \text{mixture \% } H_2 \text{ x chamber test pressure}$$

$$ppH_2 = .15 \times 500 = 75 \text{ torr}$$

This indicates that hydrogen should be admitted until the chamber pressure is increased from its low ultimate up to a pressure of 75 torr and then air or oxygen added to raise the pressure to 500 torr. The procedure is to relieve all control from PR2

(overboard dump regulator), increase pressure on PR1, open throttle valve V1 until a positive pressure is read on P1. Then, while observing chamber pressure on the electronic manometer, increase the pressure to 75 torr (75 mm) by use of V11.

The hydrogen side of the control console should now be isolated by closing V1, V9 and V20.

The next step is similar to that described for admitting hydrogen. If oxygen is chosen as the balance of the mixture, increase PR3, open V16 and V13 until P2 shows a positive pressure. Then open V21 and V11 until the chamber pressure indicates 500 torr, as measured by the precision bourdon tube pressure gauge. If air had been chosen, the valving used would be PR4, V17, V21 and V11.

The mixture ratio is now 15% H_2 in oxygen (or air) at 500 torr.

The above procedure would be similar if the 300 ppm of H_2 in oxygen were required at a pressure of 20 torr except the .4% mixture of H_2 in oxygen would be required as the test gas on the hydrogen side of the system and account must be made of the quantity of O_2 in the mixture which is being admitted.

$$\text{Press}_{(\text{final mix})} \times \frac{\text{Parts } H_2}{\text{Parts final mix}} = \text{pp}H_{2\,(\text{final mix})}$$

$$\text{pp}H_{2\,(\text{final mix})} \times \frac{\text{Parts Premix}}{\text{Parts } H_2} = \text{pp Premix}_{(\text{final mix})}$$

$$20 \text{ torr} \times \frac{300}{10^6} = 20 \times 3 \times 10^{-4} = 60 \times 10^{-4}$$

$$60 \times 10^{-4} \times \frac{10^6}{4000} = \frac{6}{4} = 1.5 \text{ torr}$$

Thus, admit from the base pressure, the premix .4% H_2 in O_2 up to a chamber pressure of 1.5 torr (1500 microns) and then add the balance of O_2 to a final chamber pressure of 20 torr.

7.3.5 The method of setting up a dynamic test is similar except flowrators and a pumping speed chart are utilized.

Assume test pressure = 2 torr and a mixture of 15% H_2 in O_2 is desired. Start with the system pumped to the ultimate and with all instrument lines evacuated as before.

From the pumping speed chart, it is determined that at a pressure of 2 torr the total gas flow possible is in the order of 12.5 torr liters/sec.

$$\dot{q}_{H_2} = \%\ H_2 \text{ in mix} \times q_T = .15 \times 12.5 = 1.825 \text{ torr liters/sec}$$

$$\text{Volumetric rate}_{H_2} = \frac{\text{torr liters}}{\text{sec}} \frac{1}{\text{Pl (torr)}} \times \frac{1000 \text{ cc}}{\text{liter}} \times \frac{60 \text{ sec}}{\text{min}}$$

$$\text{Volumetric rate}_{H_2} = \frac{1.825 \times 6 \times 10^4}{\text{Pl}} \text{ cc/min}$$

P1 is set by operation of the overboard dump pressure regulator which passes all the flow overboard until the desired mixture ratio has been set. A value of 16 to 18 psia will give satisfactory performance. The value of 18 psia must be converted to its equivalent in "torr".

$$P1\ (torr) = \frac{lb}{in^2} \times \frac{"Hg\ in^2}{lb} \times 25.4\ \frac{mm}{in}$$

$$P1\ (torr) = \frac{18}{.4912} \times 25.4 = 930\ torr$$

Now the flow rate to set into a flowrator in the hydrogen system can be established.

$$\text{Volumetric rate}_{H_2} = \frac{1.825 \times 6 \times 10^4}{930} = 117.6\ cc/min$$

At this point, examination of the calibration curves of flowrators F1, F2 and F3 show that flowrator F1 will be suitable. From the curve 117.6 cc/min = 129.5 mm (float position at center of spherical float).

At this time, close all valves except those to route the flow through flowrator F1 (including V9) and isolate the oxygen side of the control console from the hydrogen side by closing V21.

Increase pressure on PR-1, gradually open V1 and set P1 at 18 psia using PR-2 and the flow rate at 117.6 cc/min with V1.

Do not open V11 at this point.

Now the oxygen flow rate must be set.

If 12.5 torr liters/sec x .15 is the hydrogen flow rate, the balance is oxygen. Thus,

$$\dot{q}O_2 = 12.5 - 1.825 = 10.675 \text{ torr liters/sec}$$

and

$$\text{Volumetric rate}_{O_2} = \frac{10.675 \times 6 \times 10^4}{P_2} \text{ cc/min}$$

Since P2 will equilibrate with P1 (except for slight pressure drop in measuring line), P2 will be 930 torr as was P1. Thus,

$$\text{Volumetric rate}_{O_2} = \frac{10.675 \times 6 \times 10^4}{930} = 685.5 \text{ cc/min}$$

At this point, examination of the curve for flowrator F4 indicates a set point of 41.5 mm will yield 685.5 cc/min of oxygen.

Close V15, increase pressure on PR-3, open V16 and raise P2 above atmospheric pressure. At this point open V21 and using throttle valve V16, adjust flow on F4 to set point of 41.5 mm.

Thus the mixture ratio is now set, but all of the mixture is being dumped overboard. At this point throttle valve V11 is gradually opened, while observing the electronic manometer until the chamber pressure is increased to 2 torr (2000 microns).

Calibration of the device within the chamber can now be effected.

7.3.6 Calibration of the flowrators

A calibration curve of the flowrators should be made before any testing is initiated, and also on a periodic basis to insure no leaks have developed in the system to negate the prior calibration. A calibration curve may be made in the following manner.

7.3.6.1 Two sizes of volumetric calibrators are provided for the hydrogen gas admittance system. Calibrator C1 should be used for F1 and C2 for F2 and F3. The vacuum pump need not be used during this operation for all flow will be passed through the overboard dump regulator and thence out of the test area.

7.3.6.2 For calibrating F1, initiate a flow circuit such that all gas from V1 goes through F1 and that P1 can be measured. Choose increments of 10 mm deflection of the spherical float. Set PR-2 such that P1 measures 18 psia. For each set point on F1, obtain a volumetric flow rate(cubic centimeter/minute). For F1 this is accomplished by simultaneous "closure" of V4 and "opening" of V2. Use the mercury seal ring or other convenient datum on the piston in the volumetric rate meter. Measure the volume as a function of time over the largest volume to minimize error.

7.3.6.3 All other flowrators may be calibrated in a similar manner using C2 in conjunction with F2 and F3, C3 in conjunction with F4 and C4 in conjunction with F5.

7.3.6.4 WARNING: Gas flow must be stopped to the calibrator before the top of the piston travel is reached, or the mercury seal will be blown out of the top of the bore tube.

7.3.6.5 Venting of the calibration gas from the volumetric device is accomplished by opening the toggle valve at the base of the calibrator. In the case of C1 and C2, the gas is vented to the downstream side of PR-2 since this gas (hydrogen) could represent a hazard to the room environment. Calibrators C3 and C4 are vented similarly but to the room environment because of the non-hazardous nature of the gas.

8.0 MAINTENANCE

8.1 Mechanical Pump

Periodic checks should be made of the oil level through the view windows in the oil reservoir. If oil is required, use only Cellulube 220 as make-up for loss. Water vapor will contaminate the mechanical pump and cause loss in ability to reach low ultimate vacuum pressures. It is recommended, therefore, that either the pump be operated on "gas ballast" for 12 to 16 hours before a test or better to be operated continuously when not being used with a gas nitrogen purge set at approximately 300 microns. The purge pressure is set by opening the purge valve on the right rear of the control panel. Gas nitrogen should be supplied at 2 to 5 psig.

8.2 Vacuum Chamber

The vacuum chamber interior should be cleaned periodically with acetone and methanol to remove oils and water vapor from the surfaces.

8.3 Follow manufacturers' recommendations with respect to other equipment supplied. Maintenance and operating instructions are under separate cover.

9.0 SPARE PARTS

9.1 It is recommended that the folowing spare parts be inventoried:

9.1.1 Gasket - copper 1-1/2" I.D., Varian #953-5014

9.1.2 O-ring - silicone, Parker #2-388

9.1.3 Mechanical Pump Oil - Cellulube #220, Stauffer Chemical Company

9.1.4 Rupture Disc - Size 1-1/2" 18-20 psig burst pressure, Fike, Blue Springs, Mo.

9.1.5 Rupture Disc - Size 4" 18-20 psig burst pressure, Fike, Blue Springs, Mo.

9.1.6 Vacuum Support - Size 1-1/2" teflon coated, Fike, Blue Springs, Mo.

9.1.7 Vacuum Support - Size 4" teflon coated, Fike, Blue Springs, Mo.

9.1.8 Bolts - Machine 1/4-28 x 1-1/4 type 18-8 stainless steel

9.1.9 Nuts - Hex 1/4-28

9.1.10 Bolt - Machining, Mercury Air Products #MHH3001-624-56-S

9.1.11 Nut - Hex, Mercury Air Products #MN105-H-S