Boude Moore LIGO Internal Report 88-2 6/7/88

VACUUM TEST FACILITY DESCRIPTION AND EXPERIMENT PLANS

1.0 INTRODUCTION

This report is a brief description of the Vacuum Test Facility (VTF) and how it will be used. The VTF is an instrument for the precise and accurate measurement of gas flows. The pressures on both sides of an orifice are observed; from these the gas flow and pump speed are found by iteration. The VTF includes roughing and high vacuum pumps, mass spectrometers and pressure standards, and a liquid nitrogen trap. It is installed on a mobile cart.

In a very general sense the purpose of the VTF is to minimize the construction and operating costs of the LIGO vacuum systems, consistent with the performance requirements. Towards this end, the outgassing rates of candidate materials will be measured and the performance of pumps evaluated.

The VTF and the test chambers will be described, followed by a discussion of test objectives and then test procedures.

2.0 DESCRIPTION OF VACUUM TEST FACILITY (VTF)

The specialized functions of the VTF are performed by separate subsystems. These are labeled Sections A through E on Fig. 1.

2.1 Pumping Subsystem - Section A

Roughing Pumps - Section A1

Initial pumpdown of the VTF or test chambers is performed with the roughing subsystem, Section A1 of Fig. 1. This subsystem includes a small (1.2 cfm) mechanical pump, 50 liter/second turbopump, gages, vent valve in event of loss of power, and isolation valves. No measurable amounts of backstreaming oil will be observed in the system provided that the manufacturer's operating procedures are employed.

This pump can also be used during bakeout.

Low Pressure Pump - Section A2

When a total pressure of $\leq 1 \times 10^{-6}$ torr is reached, the 400 liter/second ion pump may be started. This startup policy is the same as planned for the LIGO, and is intended to avoid argon gas bursts from the ion pumps. During measurements the roughing pumps are isolated by valves, so that the full gas load flows to the ion pump. The ion pump is free of vibration, an important feature for both the VTF and the LIGO.

2.2 Measurement Subsystem - Section B

Mounted directly on the ion pump are two sections of tubing; each is approximately 6 inches diameter by 6 inches long. The two sections are separated by an orifice plate which also serves as the copper sealing gasket. Each tube section has three instruments installed symmetrically so that they read identical pressures.

The orifice is a 3/4 inch diameter hole. This was sized as a compromise to provide both a reasonably high pumping speed (33 L/s for air @ 300 K) and also a large (factor of 10) pressure difference.

This orifice plate nearly closes the end of the upper tube section; the orifice area is only 1.66% of the end area. Thus the gas molecules, moving at random, have only a small chance of striking the orifice, and so a nearly uniform pressure exists in the upper tube. The nonuniformities are calculated to be about 1/4%.

In the lower tube section, in contrast, there is a definite pressure gradient. The pressure at the pump flange is slightly less than the pressure at the gage location; the difference varies with the pump speed (which varies with pressure and gas species), but is calculated to be less than 1% for all pressures and species. The pressure of gas returning through the orifice is also calculated to be less than that at the gage location, but by nearly 5%.

These pressure differences have been calculated by numerical methods (Ref. 1), and their effects will be included in the estimates of gas flow and pump speed determined from the analyses of experimental data. Since the corrections are small, the remaining error from the pressure gradients is assumed to be negligible.

The spinning rotor gage (SRG) in each tube section provides a stable and accurate pressure reference, traceable to NBS. Pressure measurement error is specified by the manufacturer to be less than $\pm$ 1% of the measured value at pressures above 10^{-6} torr. Its drift is less than 1% per year.

The mass spectrometer (MS) has a mass range of 1–200 AMU. The Faraday cup provides stable pressure measurements down to 10^{-10} torr, while the electron multiplier extends the range to 10^{-12} torr. Up to 100 measurements per second can be taken, and provisions have been made to acquire and log data at this rate when necessary. The MS's will be used for direct measurement of pressures, and will be calibrated against the SRG.

The ionization gage (IG) provides measurements of total pressure. It can also be used in conjunction with the MS to extend the threshold of measurement to extremely low partial pressures. For example, when checking for oil backstreaming, an IG filament can be cleaned by heating, turned off to accumulate gas for a noted time interval, and then flashed on again. The resultant surge of pressure can be resolved by the MS.

It is expected that the pressure measurement error will be 5% of the reading, or less. This reflects uncertainties in the reference standard itself, extrapolation to lower pressures, and drift between calibrations. There are other uncertainties involved in the outgassing measurements such as the background level to be subtracted, stability of the background and substitution/displacement of gases on the surfaces.

It has been previously agreed (with Rai Weiss and Bill Althouse) that a 20% error in the measurement of outgassing rates is a reasonable target. It is believed that this target will be met with this VTF design.

2.3 Trap for Volatiles - Section C

A liquid nitrogen trap is mounted directly above the measurement section. This can be cooled to remove water and oils from the gas flow, so that only non-volatiles such as hydrogen enter the measurement section. Thus the AMU 2 peak can be attributed entirely to hydrogen, with no contribution from water or hydrocarbons.

2.4 Manifold - Section D

The manifold is a length of 6 inch tubing with several 1 1/2 inch ports. Multiple test chambers can be attached to it. Isolation valves allow individual test chambers to be pumped down from atmosphere without disturbing the VTF or other chambers under test.

The manifold can be readily extended if necessary, to accommodate even more test chambers.

2.5 Test Chambers - Section E

Only one test chamber is shown, but ports are provided for several others. The first chambers being built are described in Part III, below.

Outgassing tests on materials such as wire insulation can be done in a chamber attached to any of the ports. The chamber would be procured to match the required tests.

2.6 General VTF Characteristics

In addition to the specialized functions described above, the VTF must have some general characteristics in order to be an accurate instrument for the measurement of gas flow. The leak rate should be immeasurably small, avoiding corrections and uncertainties as to the source of the gas flow. The outgassing background should be much less than the gas flow to be measured.

These desirable characteristics can only be attained through a period of leak checking and vacuum bakeout.

3.0 TEST CHAMBERS

The four test chambers now being built will be made from 304L stainless steel, specially processed for low hydrogen content (tentatively 1 ppm—more samples are being measured). This steel is yellow in color, no doubt due to chrome oxide formed on the surface during the final annealing at the mill. It has a #1 hot rolled finish; this is the least costly but roughest grade, approximately .002 inches peak to valley observed through a microscope by Alex Abramovici. Most vacuum chambers are made from #4 cold rolled steel. The cold worked #4 may have a lower hydrogen diffusion coefficient, based on measurements of ferritic steels (Ref. 5 and 6).

The chambers will be 1 foot internal diameter and 4 feet internal length. They will be electron beam welded to avoid hydrogen in the weld, so that accurate measurement of the bulk outgassing can be made. The outgassing area is approximately 13,000 cm^2, after the 1.5 inch diameter connection port is cut out.

The chambers will be cleaned before the ends are welded on, using different procedures for each chamber. These surface cleaning procedures are detailed in Table I. They include:

1. No cleaning
2. Oakite 33 and hot water
3. Oakite 33 and steam
4. Oakite 33 and cold water

Oakite 33 was recommended by JPL (Ref. 2). The choice of hot vs. cold water is discussed below; see 4.0, Test Objectives.

Large chambers, such as 4 foot diameter by 20 feet long, may be tested in the future. These will require a 2 1/2 inch valve and connection to the manifold. Also a larger mechanical pump would be needed temporarily during initial pumpdown. There is a port available for this temporary connection.

4.0 TEST OBJECTIVES

4.1 Hydrogen Outgassing

The hydrogen outgassing due to diffusion out of the bulk 304L stainless steel can be reduced at low cost by high-temperature annealing at the steel mill. A sample of specially processed steel has been obtained and its hydrogen outgassing rate will be measured on the VTF. Since it may be acceptable to operate the LIGO above the ultimate design pressure for a few years, there is a possibility that the LIGO bakeout can be deleted with a considerable cost avoidance. This test was the first identified, and has been a major factor in the VTF design.

The allowable hydrogen partial pressure in the LIGO is 10^{-8} torr $\cdot$ L/s $\cdot$ cm^2. The design target is a hydrogen outgassing rate of 10^{-12} torr$\cdot$L/s$\cdot$cm^2. With the test chamber outgassing area of 13,000 cm^2, the corresponding gas load would be 1.3×10^{-8} torr $\cdot$ L/s. The orifice conductance for hydrogen is 125 L/s at 300K. Thus at the target outgassing rate, the pressure in the upper measurement tube would be 1.3×10^{-10} torr and in the lower section would be in the mid 10^{-11} range, depending on pump speed. This is the pressure increment caused by gas flow from the test chamber, and would be added to the background due to VTF outgassing.

To achieve the necessary background levels, the VTF will be baked out until the hydrogen pressure above the orifice is small compared to 10^{-10} torr, preferably 10^{-11} torr or less. The pressure below the orifice would then be in the 10^{-12} range.

4.2 Water Vapor Outgassing

A second specific objective is to measure the water vapor outgassing of the candidate steels for periods of several years. Data in the literature are unfortunately limited to less than 100 hours outgassing time. This is surprising, but common vacuum practice is to drive the water off the surface with a mild bake (100–150 C for 24 hours). It would be desirable, in order to minimize LIGO costs, to let the water pump away with no bakeout, or at least only a low-temperature bake. However, this water outgassing (unlike the hydrogen outgassing) will be a function of the surface cleaning process. There is some evidence (Ref. 3) that cold water washing increases the outgassing only slightly. On the other hand, a hot water wash may cause a large increase ($\times$ 3) in the quantity outgassed, and also a large

increase in the outgassing time (desorption energy increase from 22 kcal/mole to 25) (Ref. 4). It is thus desirable to compare cleaning procedures.

It may also become necessary to measure the outgassing at temperatures higher than 300K. There is a considerable increase in H_2O outgassing rate at 50 C (Ref. 4), which suggests the possibility of using air conditioning to heat the LIGO beam tube.

If this 50 C bake is not adequate, then a higher-temperature bake of short LIGO sections, one at a time, should be considered as a backup. This leads to a test to show that heating a test chamber in sections, one after another, is an effective bakeout procedure for water vapor.

The H_2O partial pressure allowable in LIGO is 9×10^{-10} torr, compared to 10^{-8} torr for hydrogen. Also the ion pump speed for water is only half of its speed for hydrogen. Thus the design target for water outgassing rate is 1/22 of the hydrogen target, or 4.5×10^{-14} torr·L/s·cm^2. With the 13,000 cm^2 test chamber area, the gas load is 6×10^{-10} torr·L/s. The orifice speed for water is calculated as 42 L/s, leading to a partial pressure of 1.4×10^{-11} torr. The background H_2O should thus be 10^{-12} torr or less. However, this should be achieved by the bakeout long before reaching the hydrogen target.

4.3 Duty Cycle Effect

The duration of the contemplated experiments could be several years. There is an obvious practical advantage to conducting several tests in parallel, keeping the chambers at vacuum, but measuring their outgassing sequentially. However, there is a possibility that the pressure rise in the valved-off chambers would affect the outgassing rate, especially for water at higher pressures. It is proposed to measure the effect of this pumping duty cycle by closing off a chamber for increasing periods of time. The results will also confirm the LIGO design which assumes outgassing is independent of pump size and spacing.

4.4 Gas Bursts

Bursts of gas from ion pumps or other sources could interfere with LIGO operation. Bursts have been observed by Kay (Ref. 7) due to glow discharge burial of argon which migrates to a small pore, where it builds up pressure to a fracture. A search for bursts is proposed.

If a gas burst occurs, the pressure will rise nearly instantaneously, and then be exponentially pumped back down. The volume of the pump and tube below the orifice is 38.3 liters. The ion pump speed for argon at 10^{-10} torr is rated at about 25 L/s, while that of the orifice is 28 L/s. The combined speed of 53 L/s will pump down this volume at a rate of 1.66 seconds per decade (factor of 10 in pressure). The sampling rate of the MS is not yet known exactly, but is reported to be more than 100/second by the vendor.

Although argon is the primary candidate for gas bursts, other gases might be considered. As an upper limit, the pumpdown rate for hydrogen is .256 seconds/decade at 10^{-10} torr.

The detectable gas burst quantity varies directly with the local pressure (or partial pressure). For example, if the total pressure were 10^{-11} torr and a burst causing a 10% change could be detected, then there would be 1.24×10^9 molecules in the burst. This compares to a total of 1.5×10^{18} molecules in a 4 km LIGO tube at 10^{-8} torr.

Even more sensitivity could be obtained by monitoring the argon peak. Since the background of argon is very low, nearly zero, a burst of only 1×10^7 molecules could be seen.

4.5 Ion Pump Speed

The speed of the ion pump is established as a byproduct of the gas flow calculation for each gas species. The conventional analysis assumes that a uniform pressure exists both above and below the orifice.

$$Q = S_o\,(P_u - P_d)$$

$$S_p = \frac{Q}{P_d}$$

where:

S_p = speed of pump, L/s
Q_p = gas throughput to pump, torr· L/s downstream of the orifice
P_d = pressure downstream of the orifice, torr, as measured by a gage
S_o = orifice speed, L/s
P_u = pressure upstream of orifice, torr, as measured by a gage

Implicit in these equations is the assumption that the measured pressure, at the instrument, is the same as that on the pump and on the orifice. This is nearly true for the volume upstream of the orifice. However, the volume downstream has pronounced pressure gradients. This requires some small corrections to the data to obtain accurate measurements of flow and pump speed.

It should be noted that pressure is difficult to define when a gradient exists. The calculations for the correction factors were carried out in flux units. In the equations below, the pressure is defined as that uniform pressure which produces the calculated flux.

$$Q = S_o(K_u P_u - K_o P_d)$$

$$S_p = \frac{Q}{K_p P_d}$$

$$K_o = f(S_p)$$

$$K_p = g(S_p)$$

where: the definitions above

K_u = pressure on upstream side of orifice/P_u, the upstream gage pressure
K_o = pressure on downstream side of orifice/P_d, the downstream gage pressure
K_p = pressure on pump opening/P_d, the downstream gage pressure
f & g = non-linear functions which have been found numerically

The correction factors, K_o and K_p have been numerically defined as a function of S_p. K_u is a constant, since the orifice speed does not vary (for a particular gas species).

It is necessary to solve this set of equations for each observation,in order to find the gas flow and pump speed.

A record of the VTF ion pump speed for hydrogen and for water vapor will be made, at various pressures and quantities pumped.

4.6 Air Leak Measurement vs. Hydrogen Pressure

In the LIGO, the hydrogen partial pressure may limit the threshold of detection of air leaks. This possible problem was discussed in LIGO Internal Report 88-1, 4/28/88.

In the VTF a small air leak can be deliberately introduced through a variable valve and then the hydrogen partial pressure can be increased from a separate source (a calibrated leak). The extent of the interference can then be directly observed.

4.7 Future VTF Objectives

TBD.

5.0 TEST PROCEDURES

5.1 Hydrogen Outgassing Test—Chamber #1—No Surface Cleaning

Assume the VTF is under vacuum and baked to a hydrogen background less than 10^{-10} torr. Calibration of gages is complete. Background is recorded.

Vent the manifold and roughing pumps to atmosphere with dry nitrogen. Open the test chamber isolation valve, and pump down.

As soon as practical, open the valve to the measurement section and begin measurements. Close the valve to the roughing pumps.

Record hydrogen pressure at intervals close enough to define the change with time. Cool the LN_2 trap whenever there is uncertainty as to the hydrogen fraction in the AMU 2 peak. It is expected that the hydrogen outgassing will follow a function of the form

$$Q = Q_o\left(\frac{t_o}{t}\right)^{1/2} \qquad torr \cdot L/s \cdot cm^2$$

where:

$Q_o = 1.5 \times 10^{-10}$ torr $\cdot$ L/s $\cdot$ cm^2 if the initial concentration is 1 ppm by weight and if the diffusion coefficent is 5×10^{-14} cm^2/s;

$t_o = 1$ hr.

t is measured from 6:00 A.M. PST March 29, 1988, when the final anneal was complete.

Recheck the hydrogen background monthly. To do this, isolate the test chamber and allow the hydrogen to come to equilibrium. (The time to reach equilibrium is an indication of the sticking time of H_2 on the chamber and instrument surfaces.) Record background pressure.

After the background check, compare the accumulated hydrogen in the test chamber with the static rate measurements. Reopen the test chamber and record pumpdown transient. Integrate the pressure to find the amount of hydrogen accumulated while the test chamber was closed. Compute the average outgassing rate and compare with the previous direct measurement. This method greatly increases the sensitivity, but adds some uncertainty due to the changing pressures. (A changing pressure at a single point in time cannot be measured as accurately as a static pressure which can be sampled many times.)

5.2 Water Outgassing Test

Prepare for test and pumpdown as for hydrogen (in most cases, the same operation).

Record water vapor pressure as done for hydrogen, except that more frequent measurements will be needed, because the water outgassing varies as 1/t starting from the time of pumpdown. The nominal water vapor outgassing would be:

$$Q = Q_o\left(\frac{t_o}{t}\right) \quad torr \cdot L/s \cdot cm^2$$

where:

$Q_o = 1.4 \times 10^{-7}$ torr $\cdot$ L/s $\cdot$ cm^2

$t_o = 1$ hr.

$t =$ time from start pumpdown

Recheck the water background as for hydrogen, except that the background should go to zero (below measureable levels). Record the water transient and integrate to find the quantity which was held on the VTF surfaces. Water may sorb on the manifold surface while it is at atmospheric pressure before pumpdown, even though dry nitrogen was used for backfill. It may be appropriate to bake the manifold lightly.

After the background check, measure the accumulated water in the isolated test chamber, as for the hydrogen. However it is expected that considerably more care will be needed because most of the water will be on the surfaces rather than in the gas phase. It will be necessary to add the quantity which sorbs on the clean VTF surfaces; this is presumed to be the same as that which was desorbed during the previous background check.

5.3 Pumping Duty Cycle Test-Water Vapor

Repeat the accumulation procedures (above) but gradually increase the accumulation time. Monitor the outgassing results for any change in rates with accumulation time.

5.4 Surface Cleaning Tests

In the event that multiple tests can be performed simultaneously (e.g., if the results of the Duty Cycle Testing show that outgassing measurements were valid for accumulation times of greater than, say, 1 day), then pump down Chamber #2. When the time between measurements increases sufficiently (to, say, one/day) then pump down Chamber #3. Similarly, pump down Chamber #4.

Measure hydrogen and water outgassing as above. Compare the rates and total quantities for the four surface treatments.

5.5 Temperature Effect Test

If water outgassing is too high, suggesting that an unbaked LIGO system is not feasible, then proceed with mild baking procedures to find the minimum bakeout that would give acceptable results.

Select a chamber, and raise its temperature 10 C. Measure the new outgassing rates, and continue until a new trendline is established. Extrapolate to find how long a time to reach the target rates.

Repeat, raising the temperature in 10 C steps until a minimum satisfactory bakeout temperature is found. (This result may vary with the surface cleaning procedure previously used.)

5.6 Sectional Bake Test

This test is to investigate the possibility that hot molecules (leaving the surface being baked) striking a cold surface might adsorb with unusually high energy and stick for a long time, leading to higher final outgassing rates. If earlier results indicate that this test is necessary, demonstrate that a vacuum chamber can be baked in small sections, one after the other, to achieve the desired water outgassing rate.

Select a chamber. Bake in sections (suggest 10 sections), one at a time, at TBD temperature and for TBD time. Evaluate outgassing rate achieved.

5.7 Ion Pump Speed Tests

These are conducted automatically as an integral part of the gas flow measurements. However, the results will have to be correlated to determine the speed as a function of pressure, gas species, quantity previously pumped of various species, and pump bakeout history.

This test may be repeated with a getter installed in the pump. This increases the hydrogen pump speed from 440 L/s to 1220 L/s at 10^{-8} torr.

5.8 Gas Burst Tests

Procedure TBD.

5.9 Air Leak Measurement

Assume the VTF is pumped down and the instruments are calibrated. Admit an air leak through the variable valve, and adjust until it is barely detectable. Record mass scan. Increase until the signal to noise ratio is approximately 2. Record mass scan. Admit hydrogen from calibrated leak and observe effect on air signature. Increase hydrogen pressure to 10^{-4} torr, or until the air can no longer be seen. Record mass scan.

6.0 REFERENCES

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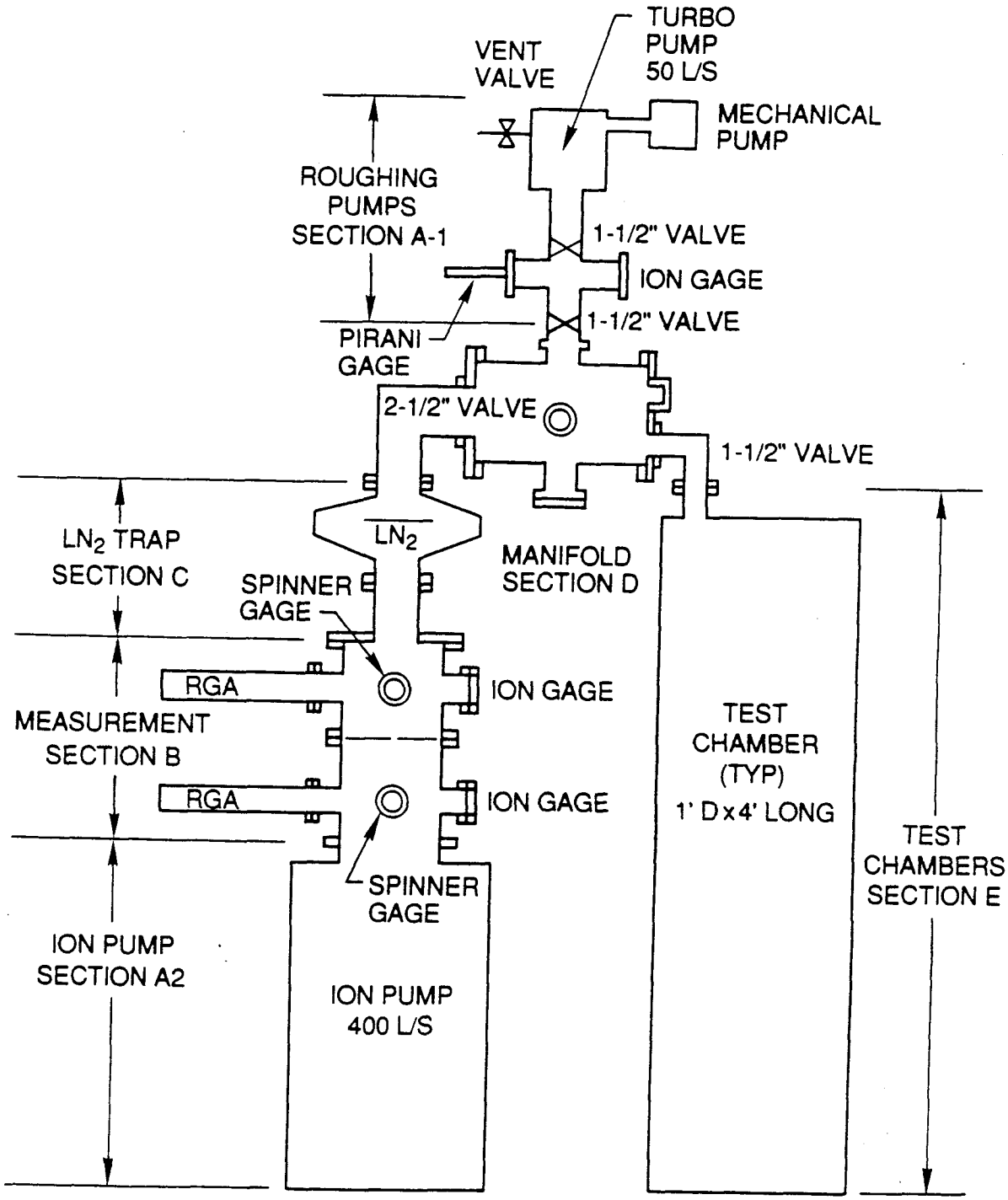
TABLE I: CLEANING PROCEDURE

1. Clean 4 cylinders (4 ft. long x 1 ft. dia.) after electron beam welding but before ends are welded on. Clean inside only. Clean 8 ends, inside only. Do not clean 4 ports, these are pre-baked. Each cylinder and pair of ends has a different cleaning procedure.
2. Caltech representative to witness cleaning.
3. Use soft water for all rinses, and for steam.
4. Record water and steam temperatures.
5. Oakite 33. Use 20% solution, at temperature specified below. Brush on, let set for 1/2 hour, and rinse with water at specified temperature.
6. Cylinder 1 - No (or minimal) cleaning. Vacuum and spot clean as required for clean appearance. Record what was done.
7. Cylinder 2 - Hot water (130–140 F) rinse, hot Oakite brush and set, hot water rinse.
8. Cylinder 3 - Steam rinse, brush Oakite (130–140 F), set, steam rinse.
9. Cylinder 4 - Cold water rinse, cold Oakite brush and set, cold water rinse.


TURBO
PUMP
50 L/S
VENT
VALVE
MECHANICAL
PUMP
ROUGHING
PUMPS
SECTION A-1
1-1/2" VALVE
ION GAGE
1-1/2" VALVE
PIRANI
GAGE
2-1/2" VALVE
1-1/2" VALVE
LN2 TRAP
SECTION C
LN2
MANIFOLD
SECTION D
SPINNER
GAGE
RGA
ION GAGE
MEASUREMENT
SECTION B
TEST
CHAMBER
(TYP)
1' D x 4' LONG
RGA
ION GAGE
TEST
CHAMBERS
SECTION E
SPINNER
GAGE
ION PUMP
SECTION A2
ION PUMP
400 L/S