October 25, 1963

THE HYDROGEN PURGING TECHNIQUE

H. G. Worstell
Los Alamos Scientific Laboratory

The PHERMEX machine at Los Alamos has just been put into operation with the first complete evacuation started in June. A design, construction, and testing program has been going on for about 3-1/2 years. The PHERMEX cavity assembly consists of seven sections whose inside diameter is 15 ft. and outside diameter of flanges and stiffening rings is 16 ft. There are three hard vacuum rf sections which are separated from each other by two soft vacuum coil sections. Each of the three hard vacuum sections have a 1/4 in. thick by 15 ft. diameter water-cooled copper bulkhead at their ends. Each hard vacuum section has its own pumping box, two observation windows, three probe ports, and six 18 in. diameter tuning slug and anode loop nozzles. There is a 17 x 24 in. manhole in each pumping box to provide cavity access. The distance between copper bulkheads is 102 in. A steel water jacket encases the hard vacuum sections to control cavity temperature at or near 70°F.

The four soft vacuum sections consist of two 20 in. thick coil sections and two heads. Each of these four sections contain water-cooled beam confining coils. The heads have a 24 in. diameter manhole and a 24 in. diameter nozzle located on the beam axis.

The complete cavity assembly is approximately 38 ft. long and although relatively lightly constructed weighs about 100 tons. The total evacuated volume is 6130 ft.^{3} of which 4700 ft.^{3} is operated at a hard vacuum. We avoided the use of elastomer seals wherever possible. We have approximately 150 ft. of metal seals and 82 ft. of elastomer seals in the hard vacuum. The soft vacuum system contains about 400 ft. of elastomer seals, but there is no outgas problem here.

We actually have three separate systems, soft, roughing and hard. A 6 in. diameter aluminum manifold connects the four soft vacuum sections in parallel. The rough vacuum manifold is fabricated from 12 in. diameter aluminum pipe and connects the three hard vacuum sections in parallel. We do not use a hard vacuum manifold. Three 12 in. pneumatic gate valves are used to isolate the hard vacuum system from the roughing manifold.

Two 6 in. pneumatic valves are located between the rough and soft vacuum systems to provide isolation of these two systems and pressure equalization for initial pump-down. Three 12 in. aluminum rupture diaphragms separate the three hard vacuum sections from the soft vacuum manifold to provide protection for the six relatively fragile copper bulkheads. These diaphragms will rupture any time a pressure differential on the bulkhead reaches 1/4 psi.

Our mechanical pumping consists of two Kinney KD 310 pumps located at either end of the soft vacuum manifold and two batteries of roughing pumps each of which connect into the roughing manifold between the three rf cavities.

Each battery has three pumps in series, a 3500 cfm blower which is backed by a 900 cfm blower which in turn is backed by a 105 cfm staged rotary piston pump.

Six 1000 ℓ/sec vac-ion pumps comprise the hard vacuum pumping. Two of these pumps are connected directly to each of the rf section pumping boxes with no manifolding or valves. We believe this system is one of, if not the largest, hard vacuum volume that has been evacuated with ion pumps.

Briefly the pump-down sequence is as follows:

1. With all phasing valves open, the two KD 310 and the two 105 cfm rough pumps are started and the complete cavity system is exhausted to approximately 1000 microns.

2. The two 6 in. phasing valves are then closed isolating the soft system from the rough system. The four blower pumps are started at this point and evacuate the hard vacuum system to the starting pressure of the ion pumps.

3. As soon as the ion pumps have started, the three 12 in. phasing valves are closed, the rough pumps turned off and blown to air. The two KD 310 pumps are valved off from the soft vacuum manifold and blown to air. A small Welch pump, connected into one of the coil sections, maintains the soft vacuum system at about 70 microns during the remainder of the evacuated period.

It is common knowledge among ion pump users that these pumps are rather fussy about the kind of gas they will digest. Their pumping speed for the noble gases, water vapor, most hydrocarbons, and outgas material may be reduced to nearly zero. As the system is pumped down, the ion pump will, in effect, differentially pump the gases for which it has an appetite and leave the distasteful components. This process continues until a system base pressure is reached.

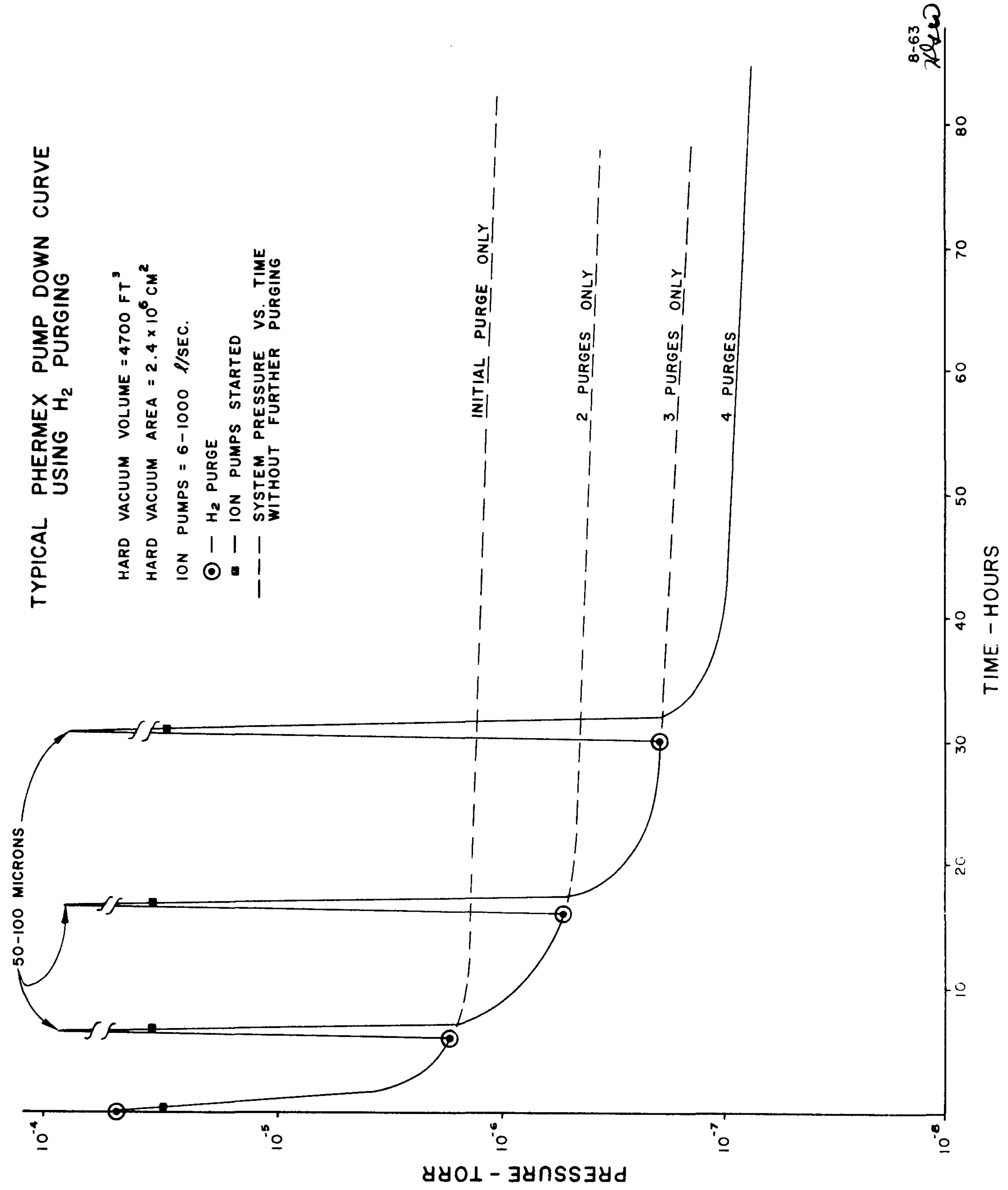
Some three years ago we were faced with this dilemma in conjunction with the prototype of our final machine. At that time the largest ion pumps were considerably smaller than are commercially available today. We not only encountered a great deal of trouble in starting the pumps on this prototype (1400 $ft.^3$), but also found that it took as long as two weeks to reach 3 or 4 x 10^{-6} Torr.

A hydrogen purging technique was developed at this time which proved to be very effective in the elimination of the undesirable residual gases. Not only did this purging technique assist greatly in starting the ion pumps, but made possible the attainment of system pressures that were lower by a factor of 10 in a fraction of the previously required pumping time. Purging proved to be so successful that one of the design requirements for the final machine vacuum system was that it must be capable of being purged with ease.

The sequence shown in Fig. 1 illustrates the procedure we now employ in purging the hard vacuum sections. It is a plot of the average data we obtained for several pump-down periods during the early stages of PHERMEX operation.

Briefly the technique is applied as follows:

1. At the end of the roughing cycle and just prior to starting the ion pumps, H_2 is admitted to the system with the three 12 in. phasing valves closed to bring the system pressure up to about 100 microns. This provides a


TYPICAL PHERMEX PUMP DOWN CURVE USING H2 PURGING
HARD VACUUM VOLUME = 4700 FT³
HARD VACUUM AREA = 2.4 × 10⁶ CM²
ION PUMPS = 6-1000 ℓ/SEC.
⊙ — H2 PURGE
■ — ION PUMPS STARTED
- - - SYSTEM PRESSURE VS. TIME WITHOUT FURTHER PURGING
50-100 MICRONS
INITIAL PURGE ONLY
2 PURGES ONLY
3 PURGES ONLY
4 PURGES
PRESSURE - TORR
10⁻⁴
10⁻⁵
10⁻⁶
10⁻⁷
10⁻⁸
TIME - HOURS
10
20
30
40
50
60
70
80
8-63

residual gas dilution of about 10^5.

2. The three phasing valves are opened to the rough manifold and the rough pumps are allowed to exhaust the system to the starting pressure of the ion pumps and the pumps are started. The three phasing valves are closed and the rough system blown to air.

3. At some time later the ion pumps are turned off, the rough pumps started, H_2 again is admitted to the system, the system exhausted and the ion pumps restarted as in Steps 1 and 2.

The system may be purged as frequently and as many times as desired. Each successive purge of course will have less effect than its predecessor.

Generally speaking, either the volume being pumped or the surface area and outgas rate will determine the minimum size of ion pumps for a given system.

If the system is large, such as PHERMEX, the pumps may heat excessively before the pressure can be reduced to a safe operating point. This will also happen in the case of a smaller system if the ion pump starting pressure is too high. We find that we must pump down to about 8×10^{-6} Torr within 20 minutes after starting the pumps or a shutdown to allow the pumps to cool is necessary. The low starting pressure provided by our rough pumps plus H_2 purging has pretty well eliminated this problem in our case. These ion pumps might start at 10^{-2} Torr, as the manufacturer claims, on a smaller system, but in our case we have been unsuccessful in our attempts to start at pressures above 5×10^{-4} Torr.

The determining factor which fixes the size of pumps for hard vacuum systems is usually the surface area being pumped and its outgas rate. We have a large surface area but fortunately a very low outgas rate and by purging with H_2 we have been able to eliminate the undesirable residual noble gas constituents from air and mitigate substantially the slower pumping effect of system outgas materials. Recent data definitely indicates that the outgas components are many times more difficult to ion pump than the noble gases left from pumping down on air.

We are well pleased with the performance of the ion pumps and have had no down time to date due to pump trouble. The estimated lifetime of the pumping elements in our service is 12 to 14 years. They have proven to be very reliable and require no attention once the system pressure has been reduced to some safe pressure. It is common practice to allow the system to operate for several days at a time without so much as bothering to check the pressure. A protection circuit is built into the power supplies such that if the system pressure should happen to rise to about 5×10^{-6} Torr the pumps simply turn themselves off. These pumps provide a very clean vacuum and are compact and easy to install. Two men installed our six pumps and had them ready to start in about 5 hours.

The ion pumps are sometimes tempermental and can be downright obstinate during the starting period. Once the operator becomes familiar with the pump's starting characteristics and treats them with the proper care and respect, he will encounter little trouble in starting. If the system being evacuated is large, such as PHERMEX, it is essential that the roughing pumps be capable of providing a relatively low starting pressure for the ion pumps. There is no doubt that the performance of the ion pumps in our system would be something less than satisfactory without the benefit of system purging.

FEATHERSTONE: How long does it take to pump down before you can turn on the vac ions?

WORSTELL: Generally, around three hours. It depends on how long you had it up to air and what you were doing while you had it open. It's getting better all the time. In February, we completed and shipped the first cavity section to Los Alamos and began a program of anode loop orientation. We had to pump this unit down to 1 x 10^{-6} Torr and then let it up to air to rotate our loops. We had to go through quite a program to get the right angle. So we pumped that thing down 21 times in 31 days and we had to get to 1 x 10^{-6} before we could get the high level rf into it.

HUBBARD: At what pressure do you turn on the Vacion pumps?

WORSTELL: That's a good question. Actually we let the rough pumps go down to their base pressure which is in the 10^{-5} scale. The Roots blowers will go as low as 5 x 10^{-5}. Then we bake out the ion pumps and keep roughing. We find that an hour or two spent in extra roughing will save several hours on the other end. Our criterion was to pump from atmosphere to at least 1 x 10^{-6} Torr within 16 hours. We are actually doing it now in about 10 hours.

HUBBARD: Do you bake the Heraeus pumps?

WORSTELL: No.

YOUNG: What vacuum do you have right now?

WORSTELL: About three weeks ago it was 3×10^{-8} Torr and was still going down, so I would expect that the middle cavity is going to get on the 10^{-9} scale, although we have never had it under vacuum for more than two weeks at a time until this last pump down.

QUESTION: When you shut down do you use dry gas or just let it go up to air?

WORSTELL: We would like to let it up with dry nitrogen, but it takes 32 bottles to fill the cavity. Even being in Los Alamos where the humidity is very low, we still get water vapor. We also feel that the nature of the first gas that hits the walls determines 90% of the problem, so we let in two bottles of dry nitrogen per cavity and then blow the rest with ambient air.

LIVDAHL: Have you tried purging with nitrogen instead of hydrogen?

WORSTELL: I have tried purging the tank with trapped air to try it out. It worked just as well, but it takes about three times as long to pump all the way down. It's just a matter of time. However, we do need the hydrogen to start the ion pumps with this large a system and such a small pumping capacity.

GOULD: At your base pressure of 3×10^{-8} Torr have you made any gas analysis of the residual gas in the chamber?

WORSTELL: No.

FEATHERSTONE: What is your surface area?

WORSTELL: The surface area of the hard vacuum chamber is about 2.4×10^{6} cm^{2} of very clean copper. It has never been baked out. The cavities were very dirty as they sat for months outside in all kinds of weather during fabrication. We sandblasted the inside with really fine dust, almost like chalk powder, that did not take any copper off. We had a piece of copper sandblasted for 15 minutes and it didn't affect the copper. It gets all the junk out and then we tumble the tank sections and passivate the surface with chromium trioxide. This surface works well for both the vacuum and the rf.

FEATHERSTONE: You passivate it?

WORSTELL: Yes, after sandblasting the surface is very active. If you put a drop of water on it, it oxidizes instantly. We get kind of a dull finish with it and it's not smooth.

Earlier I mentioned outgassing rates. You can calculate these rather easily when you have a system like this. After the cavities had been under vacuum for a while we turned off the pumps for three and a half hours one day. The pressure was then 5×10^{-8} Torr and it came up to 1×10^{-6}. This gives an outgas rate of 4.7×10^{-12} Torr-ℓ/sec/cm^2. This is about 2 orders of magnitude lower than most people quote for stainless steel. The outgassing surface is 99.9% copper. If you look at the literature you will find that the outgassing rates published go anywhere from 2.5×10^{-5} to 5×10^{-9} for copper. As you can see, we have a much cleaner system than the samples used in tests.

GOULD: This agrees fairly well with the outgassing data on our linac. Our design figure was 8×10^{-9} Torr-ℓ/sec/cm^2, but this includes all the organics that exist in our design. After three years of pumping, we're running at the rate of about 5×10^{-11} Torr-ℓ/sec/cm^2 and we have a lot more organics than you do. The published figures for copper have never come anywhere near that.

WORSTELL: No. they haven't. I was looking at some of B. Dayton's data. One number was 2.3×10^{-6} and this was supposedly a baked sample, and the next number was one that was 2×10^{-8} and this was copper that had been exposed to a 100% humidity atmosphere at 95° for 24 hours before it was put in there.