

## Cryostat Parts and Notes

Notes updated 1/01. Designed by Steve Andrews, 650-723-0386. See article about cryostat: Steven S. Andrews and Steven G. Boxer, "A liquid nitrogen immersion cryostat for optical measurements" *Review of Scientific Instruments* 71:3567-3569, 2000.

These notes refer to two cryostats that were machined by the Varian machine shop at Stanford University around August 1999, and then welded and brazed at the HEPL tube shop. All measurements are in inches. Costs of most raw materials are given below, but I don't know either the machining or welding costs. For price comparison a ST-100 Janis cold finger cryostat is \$4315.

### Figures

- A. Full size drawing of design made before machining. Some measurements and minor design changes were changed during machining (in particular the design of the cap, parts 19-23).
- B. Measurements of all parts, from finished machined parts.
- C. Color picture of cryostat for publication, in 1/2 scale. The cap is shown very nearly as it was machined. The only part of this figure that is incorrect is that the fill and vent tubes, parts 9 and 12, are actually 90° away from each other, rather than the 180° as shown.

### Parts

Numbers below refer to part numbers, shown on attached figures. All the measurements here, except where noted, were taken from finished machined parts.

1. Inner N2 tube. 1.5" O.D., 0.016" wall, 13.61" long, stainless (type 321). Polished on outside. No holes or other machining. Purchased from TW Metals. \$135 for 4 feet.
2. Outer N2 tube. 2.5" O.D., 0.028" wall, 7.96" long, stainless (type 321). Polished on outside. No holes or other machining. Purchased from TW Metals. \$129 for 3 feet.
3. Radiation shield. 2.75" O.D., 0.028" wall, 10.52" long, aluminum (type 6061-T4). Polished on inside and outside. Top of tube has 4 bolt holes (0.10" diameter, center of holes 0.105" below top of tube, countersunk), so tube can be bolted to

- piece #5, with 1-72, 1/4" flathead bolts. Has two 1" diameter holes near bottom, at window positions, which puts the hole center 1.559" above bottom of tube. Holes are at 45° angle offset from bolt holes on top. Purchased from TW Metals. \$185 for 12 feet. (Excess tube was given to Varian machine shop.)
4. Janis cryostat shroud. Purchased from Janis. Didn't require any machining or modifications. All quartz windows are used for UV/Vis work; 2 windows are calcium fluoride for infrared work. I.D. is 3.00". \$720 each.
    - 4a. Upper portion of Janis cryostat shroud, called instrument skirt. The vacuum valve and pressure release valve are included and are standard. Of the four ports, we didn't get any of them with electrical connections, but got one port on each instrument skirt with a 1/8" NPT thread for a pressure gauge (thermocouple gauge purchased from physics store). I.D. is 2.75". Purchased from Janis. \$500 each.
    - 4b. Not identified in figures: two 3" tri-clover O-rings and clamps are required.
  5. Top of N2 jacket. Diameter is appropriate for I.D. of radiation shield (#3), 2.687". Total thickness is 0.250". Has two 0.250" through holes drilled for filling tube (#12) and vent tube (#9), 90° apart from each other. If the filling tube is in the front, the vent tube is on the left (in figure A, the vent port is on the right and the filling tube is in back). Holes are not tilted. Also has threaded 1-72 holes to attach to radiation shield (#3), at 45° angle from other holes.
  6. Top of cryostat. Tri-clover type cap, stainless. Purchased from physics store. Has large central hole to match outside of chimney (#7), and holes for filling and vent tubes (#12, 9, 10). Holes are not tilted.
  7. Outside of chimney. 1.75" O.D., 0.035" wall, 1.963" long, stainless. Polished on inside and outside. No holes or machining required. Purchased from TW Metals. \$112 for 1 foot.
  8. Quick-flange weld fitting. Stainless, 40 mm. Purchased from physics store. Has outside machined so it attaches to 1.75" tube (#7).
  9. Lower vent tube. 0.25" O.D., 0.010" wall, about 3.34" long, stainless. Purchased from physics store. No holes or machining required.
  10. Upper vent tube. 0.3125" O.D., 0.020" wall, or thicker, about 0.7" long, stainless. Purchased from physics store. No holes or machining required.
  11. Female 1/4" NPT weld fitting. Standard size comes designed for 1/4" tube, so it was drilled out for 5/16" tube (#10). Also, back (the left side, in figure A) was machined down to leave some space between the fitting and the chimney. Purchased from Sunnyvale Valve and Fitting. \$12.90 each.

Not shown: For use with N2 jacket in vacuum mode, some fittings are required: female vacuum fitting (#11) attaches to male tee, and then to small size quick-flange fitting. Other branch of the tee is closed with a stopper during use. Purchased from physics store.

12. Fill tube. 0.25" O.D., 0.010" wall, about 3.34" long, stainless. Purchased from physics store. Top is flush with top of cryostat, after welding. In figure A, the fill tube is on the back, which is how it was machined. In figure C, the fill tube is on the left for clarity. No holes or machining required.
13. Window assembly. Machined from a single piece of stainless. Holds 2 windows, which are 1" diameter and between 2 (0.078") and 4 mm (0.197") thick. Windows are sealed with indium seals. Through holes for windows are 0.75". The inside of the piece is flush with the inside of the inner N2 tube (#1), except at the windows. The outside of the piece is flush with the outside of the outer N2 tube (#2). In the sides, away from the windows, four 0.378" holes were drilled on each side to allow N2 in the jacket to be within and below the window assembly and to minimize the thermal mass of the window assembly (see figures). The bottom is capped by both an inner cap (#16) and an outer cap (#17). The window assembly is symmetric top to bottom, left to right, and front to back.
14. Rings to hold windows (#15). Stainless. Through hole is 0.75". Has 6 allen head bolts, size 2-56. Purchased bolts from physics store. Rings were made for both 2 mm thick windows (#14a) and for 4 mm thick windows (#14b). See suggested modifications, below.
15. Windows. Size is 1" diameter or 25 mm diameter (both are standard and work well), with thicknesses between 2 mm and 4 mm. For IR use, we used uncoated Cleartran (from Janos) for the older cryostat. Broadband anti-reflection coated Cleartran for use at 5 microns also works well (2 mm thick from Spectral Systems, \$175 each). For UV-Vis-NIR, quartz (CVI Laser) has worked well, although care needs to be taken to minimize strain during installation and during cool-down.
16. Inner cap for window assembly (#13), stainless. 0.125" thick. Cap is round.
17. Outer cap for window assembly (#13), stainless. Should have been made of plate stock to minimize vacuum leaks, but was made of bar stock. Initial material thickness was 0.50" and outside is flush with outside of window assembly. Cap is round, covering the entire bottom of the window assembly. Has a tapped hole part way into the bottom for a 3/8" 6-32 nylon bolt. This is not used due to concern about outgassing from the nylon, and because it doesn't seem to be necessary.

18. Bottom of radiation shield, aluminum. 2.75" diameter, 0.10" thick. Both sides were polished. Has a hole through center for a 3/8" 6-32 nylon bolt. Got bolts from physics store, which were shortened to 3/8".
19. Sample mount top. Quickflange fitting, stainless, 40 mm, already purchased from physics store. Has 1/4" NPT center hole, angled 1/8" NPT hole, and angled 0.375" hole for a weld fitting. The last hole was angled diagonally as well so a tube down this hole won't hit the sample holder rod (#21). See suggested modifications, below.
20. Ultratorr male connector, 1/4" O.D. tube to 1/4" male NPT, stainless. Has inside bored through to give 0.26" hole. Got from Sunnyvale Valve and Fitting. \$12.60 each.
21. Sample holder rod. 1/4" O.D. black fiberglass tube, purchased from Tap Plastics. Initial length about 3 feet, final length about 2 feet. \$2.50 each.
22. Pressure relief valve. 1/8" NPT, 10 psi, stainless, Circle Seal type. Purchased from Tempresco Inc. \$49.50 each.
- 22a. Extension for pressure relief valve. This is not shown on figure A, but was found to be necessary and is included on figure C.
23. Fill port. 1/4" Swage male reducer, stainless, purchased from physics store. Capped with swage cap, also purchased from physics store. Bored through to give 0.25" hole. As mentioned above (#19), this was angled diagonally so that a funnel inserted here won't hit the sample holder rod (#21).
- 23a. Extension for fill port. As with #22a, this is not shown on figure A, but was found to be necessary and is included on figure C.

Additional items that might be needed:

Vacuum pump to pump on N2 jacket if the cryostat is to be used in vacuum mode. This pump needs to not mind high flow rates for extended periods of time (>50 SCFH at the beginning), but a good vacuum is not important. At the FEL, we use a diaphragm pump. The quieter, the better.

Maybe a turbo pump, so the cryostats can be pumped out conveniently. However, the diffusion pump may be fine.

### Suggested changes

The sample mount top (#19) and ultratorr connector could be improved. A better design would have had a smooth center hole in the top, and a weld fitting ultratorr connector. This would have taken less space on the cap and been easier to build.

The bolt connecting parts #17 and #18 has never been used since it hasn't been necessary. It could be left as an option, or removed which would simplify the two parts.

Two sets of window holding rings were made, #14a and #14b. However, only the ones designed for 2 mm thick windows (#14a) have been used, and the others don't seem useful.

Windows are held on with 2-56 size bolts, which is relevant to parts #13 and #14. This is sufficient for a skilled or careful user, but novices tend to break the bolts, causing major permanent damage. Size 3-56 bolts might be better.

Initially, we tried to weld the cryostats together, but then found that it worked much better to braze most connections.

## He insert notes

The cryostat could be converted to a He immersion cryostat by removing the inside windows which connect the inner N<sub>2</sub> region to the high vacuum. Then a He insert tube is dropped in the inner N<sub>2</sub> tube and clamped, the high vacuum is re-established, and the conversion is complete.

He insert parts include:

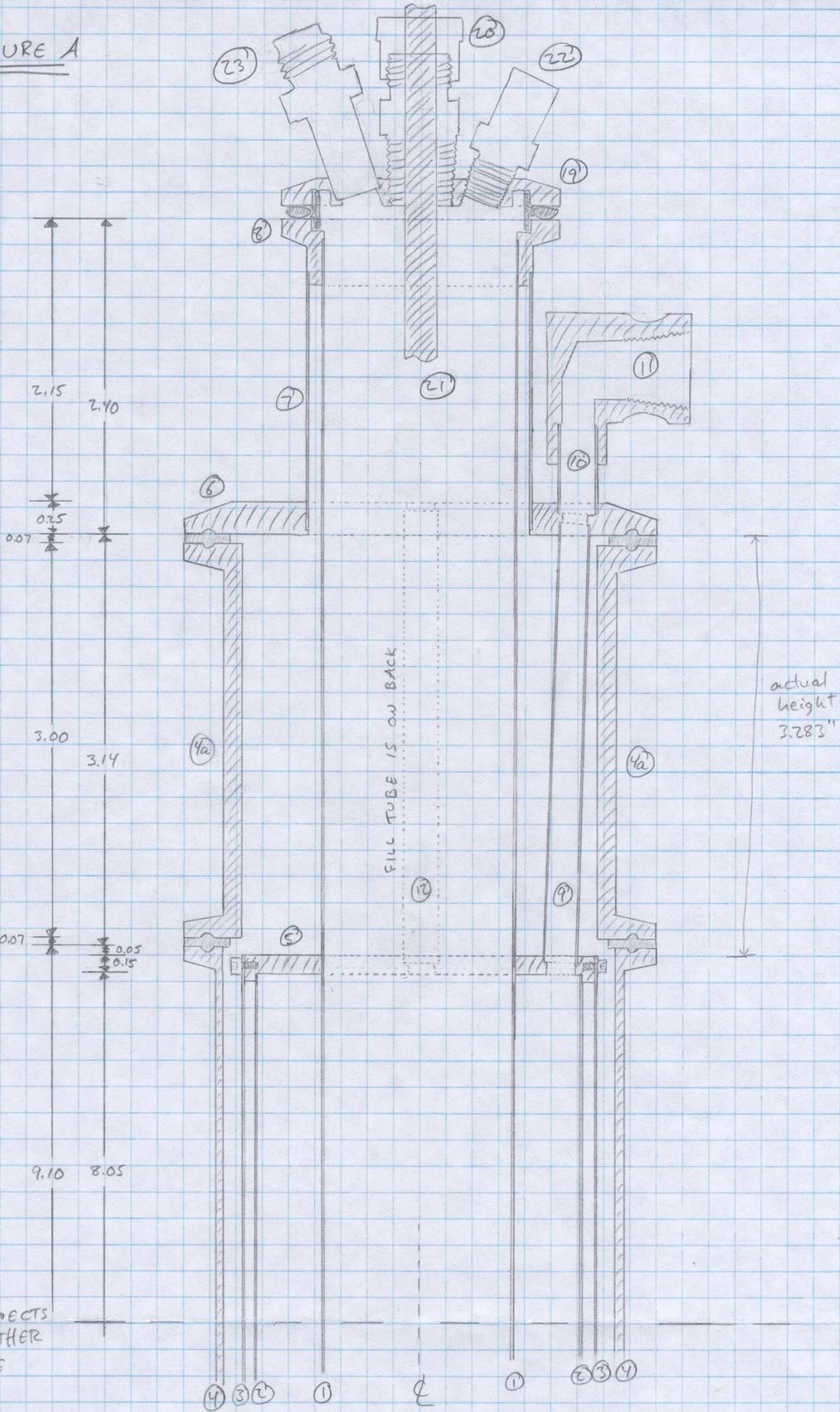
Top piece is 40 mm quickflange to 40 mm quickflange spacer. Inside is a 1" tube for He and a 1.25" tube as a radiation shield. In the sides of the spacer (not shown in diagram) are two ports into the He region; one is for evacuation of vapor and the other is connected to a 1/8" tube that goes to the very bottom allowing liquid nitrogen to be sucked out without opening the system. Also, a third port could be added as well, into the high vacuum region. With this port, the N<sub>2</sub> windows could be left in place and the inner and outer high vacuums would be separate.

At the bottom of the insert is a window assembly which holds a pair of 1/2" windows. This assembly is capped on the bottom. The current idea is that the windows would be held in on the edges with 1/16" wide surfaces on both the window assembly and the rings, leaving a 3/8" visible region.

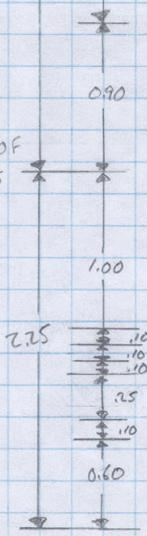
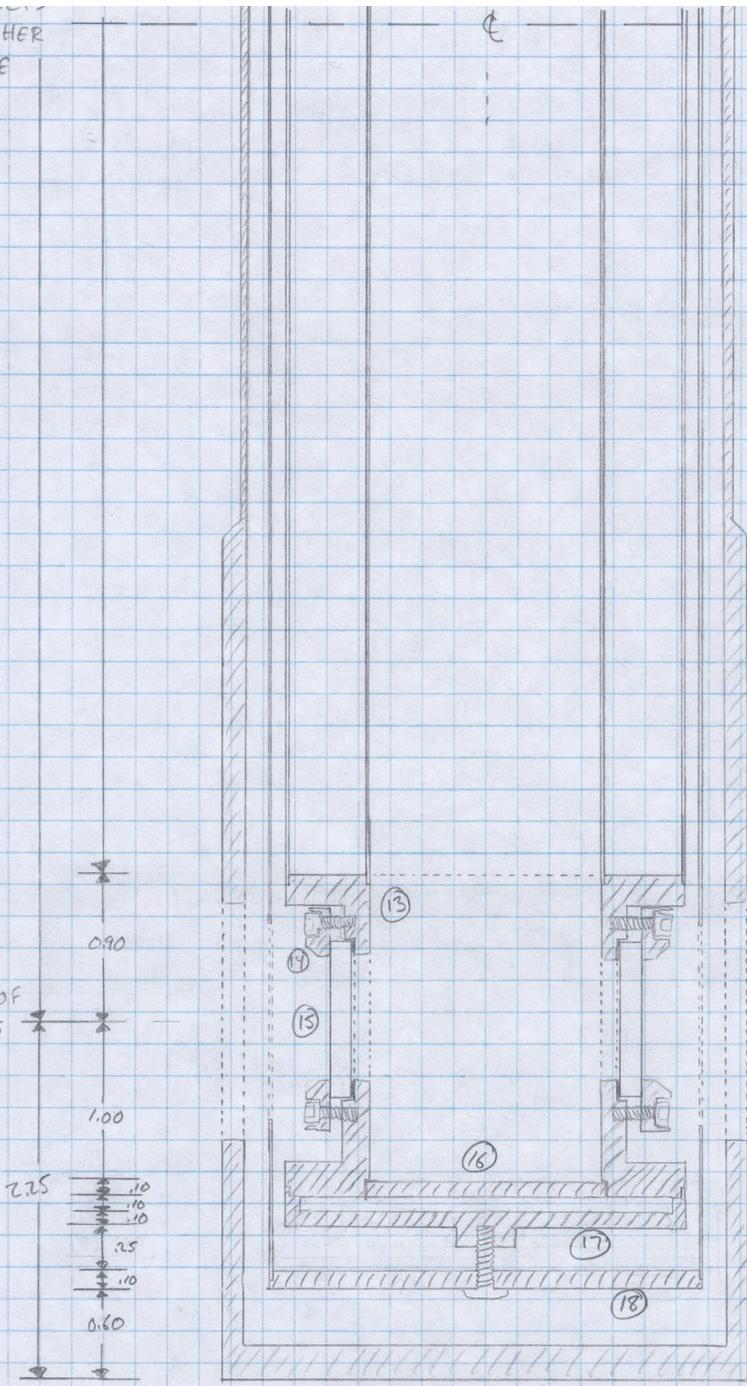
The sample mount is the exact same one that is used for N<sub>2</sub> immersion. Something important here is to insulate the bottom of the top piece with some sort of foam.

The idea on use is that the He region could be filled with N<sub>2</sub> initially, and the sample is immersed, and the region is clamped. Using the side port attached to a tube, the liquid N<sub>2</sub> is pumped out (but the sample stays frozen and frost-free). Using the 1/4" Swage fitting on top, He is added. Once it is filled, the He tube could either be removed, or it could continually flow in more He. Using the vapor side port, He could be pumped on to make it colder and to get to the lambda point.

FIGURE A



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CENTER OF WINDOWS

13

14

15

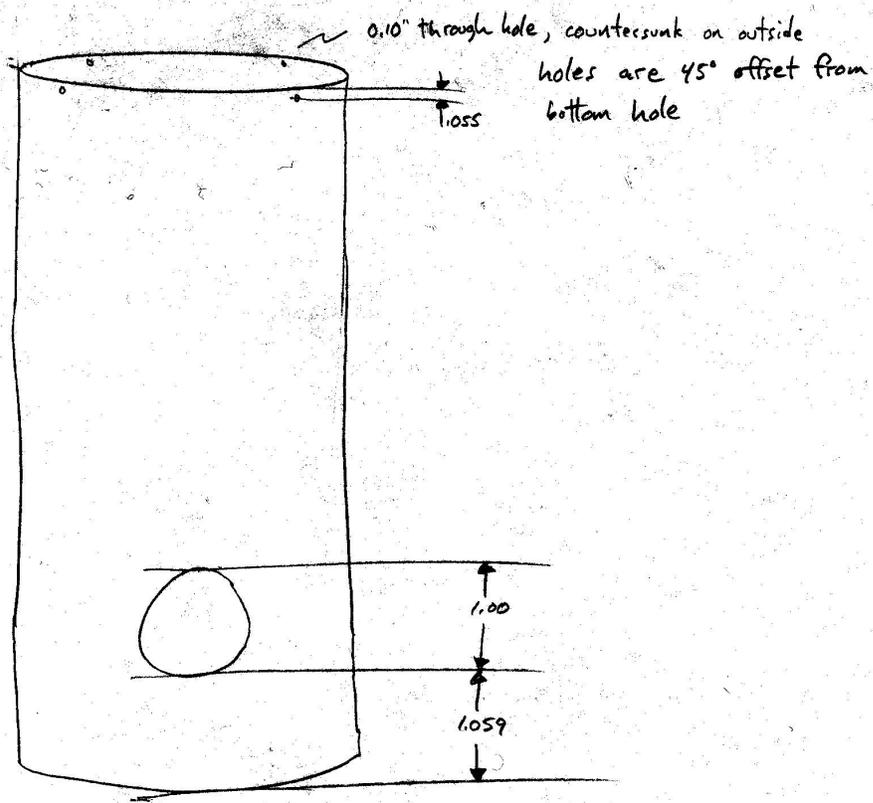
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17

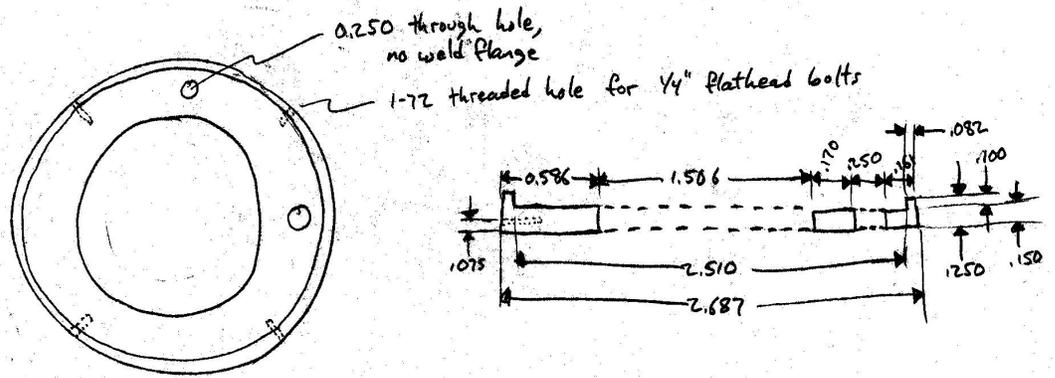
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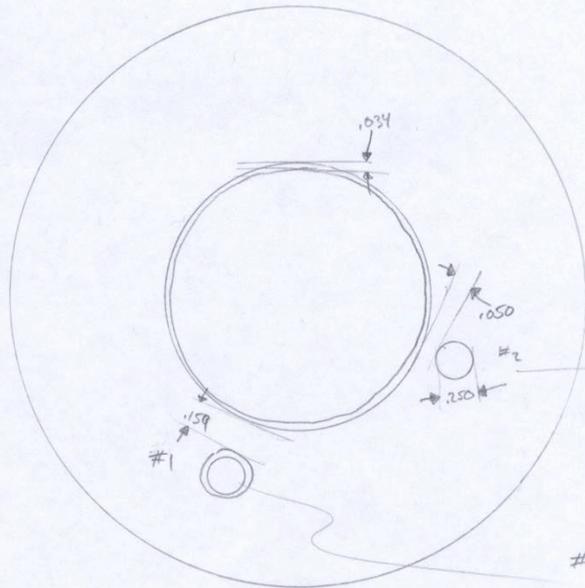


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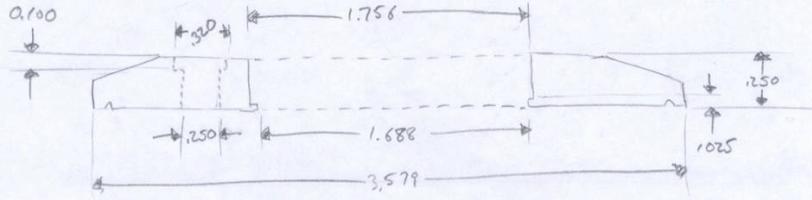
90° between through holes  
 threaded holes offset 45° from through holes.

6

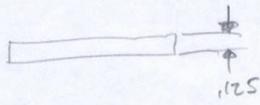
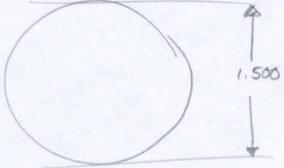


#2 through hole, 0.250" diameter, no weld flange

#1 through hole, bottom is 0.250" diameter, top is 0.320" diameter

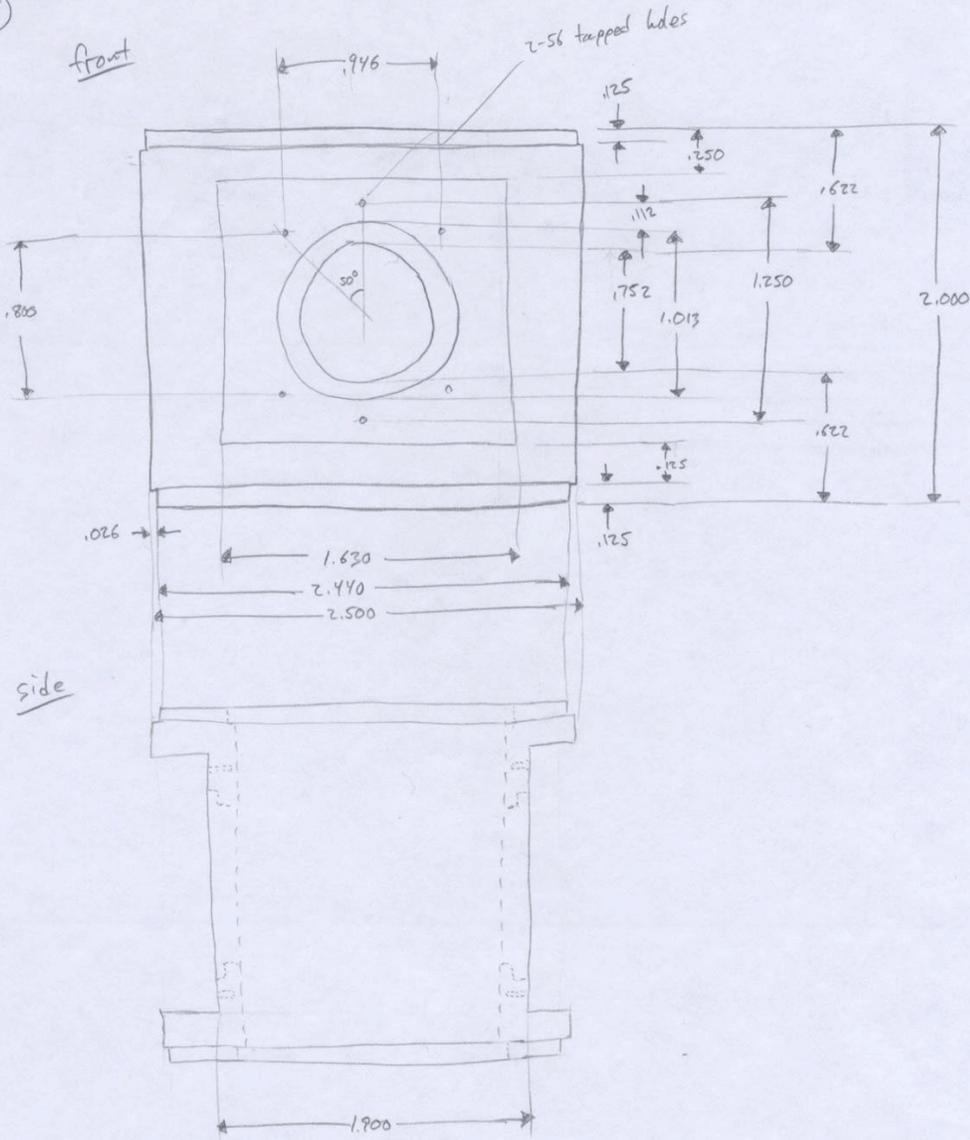


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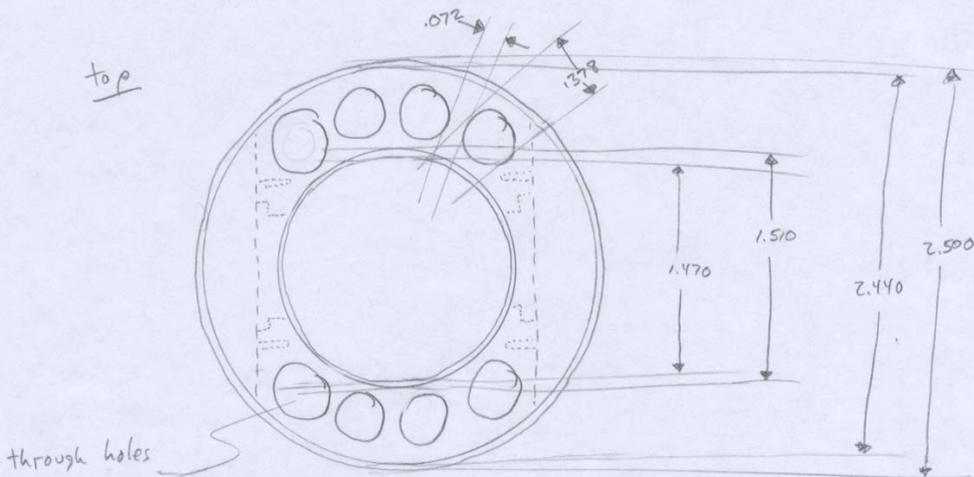
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front

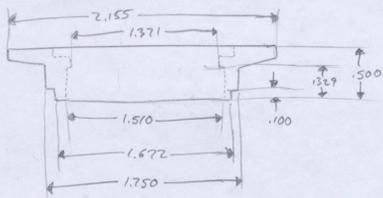


side

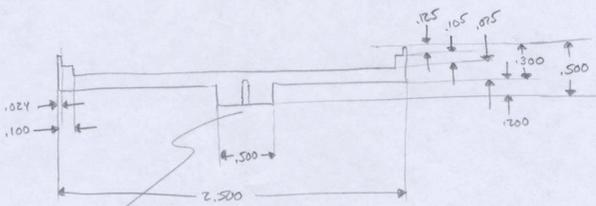
top



16

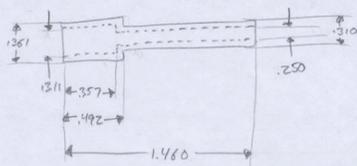


17

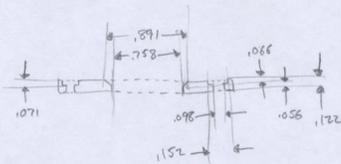
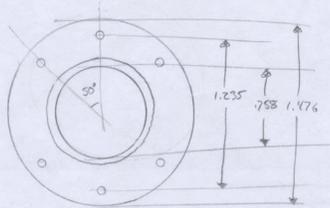


6-32 threaded hole in center

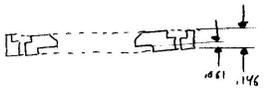
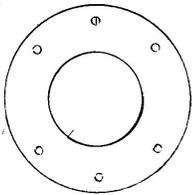
23a



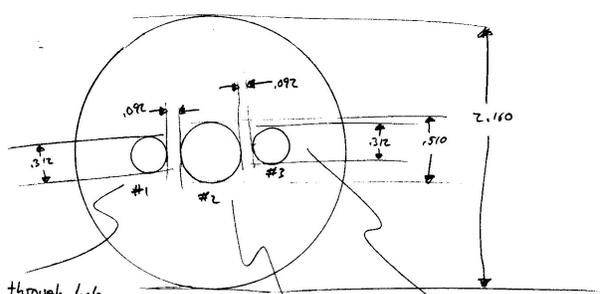
14a



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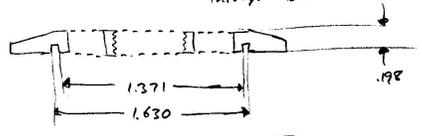
19



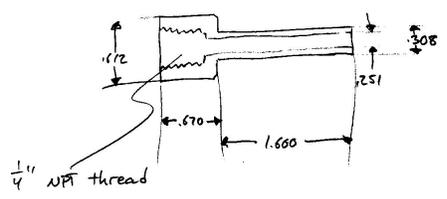
#1) .312" through hole, tilted on two axes, so the axis of the hole is in this direction: ↘

#2) 1/4" NPT threaded through hole

#3) .312" through hole, not tilted.

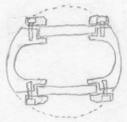
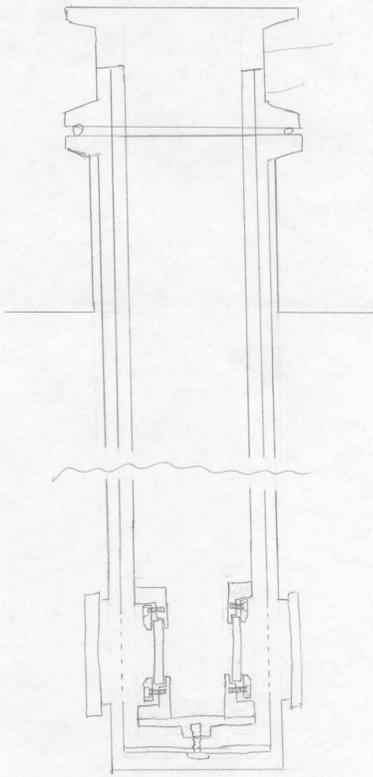


22a



1/4" NPT thread

the insert





Sample chamber  
fill port

Sample chamber 10 psi  
pressure relief valve

Jacket  
fill port

Jacket vent/  
evacuation port

Guard vacuum  
port

Sample chamber

Cooling jacket

Sample

