

**1 Purpose**

This procedure is used to remove CF3I from the bubble chamber volume. This procedure is written for the “dirty” mechanical prototype system.

**2 Prerequisites**

It is assumed that the system is in the “Compressed” state valve position. It is assumed that the hydraulic system is in the normal operating state

**3 Introductory notes**

The differential pressure across the bubble chamber and quartz jar must be limited to 15 psid in either direction. No objectionable differential pressure will be developed as long as the stop plate is within its free travel range. ZT4 free travel range is -2.1” to +1.0”

The general procedure is to isolate and depressurize the glycol side of the system to just below the saturation pressure of the CF3I, then distill the CF3I into an external container. Then, glycol is sucked out of the outer vessel to the de-gassing tank.

**4 Procedure**

1. Determine the temperature of the bubble chamber by looking at TE30 thru TE33. Determine the saturation pressure of the CF3I at this temperature by looking at the saturation table.

	Saturation	Saturation	Saturation
Temperature	Pressure	Pressure	Pressure
Celcius	Mpa	psia	psig
-22.5	0.101	14.70	0.00
0	0.228	33.04	18.34
5	0.269	38.97	24.27
10	0.315	45.67	30.97
15	0.367	53.21	38.51
20	0.425	61.66	46.96
25	0.490	71.08	56.38
30	0.562	81.54	66.84

2. Change the hydraulic pump piston position set point to ZT-6 = 7.90”. This will transfer some glycol into the diaphragm tank. The diaphragm tank will be full at WT-9 = 90 lbs, so if it reaches this level stop the pump. Stop the hydraulic pump in manual when ZT-6 = 7.90”. Close MV-25 to isolate the hydraulic pump. This is necessary because PSV-34 has leakage from the outer vessel side of the pump

to the diaphragm tank. If MV-25 is left open, glycol will leave the outer pressure vessel (at 35 to 50 psig) and end up in the diaphragm tank (at about 10 or 12 psig).

3. Reduce PV10/PIT7 setting to a value that will reduce the glycol pressure to about 5 psig below the saturation pressure of the CF3I. For example, if the temperature of the bubble chamber is 20 C, the saturation pressure is 47 psig. We would want the glycol pressure to be 42 psig. The PV10/PIT7 setting should be  $42 \text{ psig} / 4 = 10.0 \text{ psig}$ . The piston will travel towards 0.0" on ZT-6 as the CF3I in the bubble chamber boils and starts occupying the expansion volume. Note: Use caution to limit the differential pressure PT83-PT3 if the bubble chamber stop plate, ZT4, gets outside the range  $\pm 1.0$ ".
4. The glycol cylinder should have moved 8", accepting  $8" \times 0.2 \text{ L/in} = 1.6 \text{ Liters}$  of glycol. The CF3I should have boiled 1.6 liters of gas and ZT-4 movement should be  $1.6 \text{ L} \times (1" / 2 \text{ L}) = 0.8 \text{ inches}$  upward.
5. Fine tune the expansion volume and ZT4 position to 0.0" by opening MV-25 and running the glycol pump. Running the pump at negative values will withdraw liquid from the outer pressure vessel and increase ZT4 position. Running the pump at positive values will add glycol to the pressure vessel and decrease the ZT4 position. When done, stop the pump and close MV-25.
6. Note that CF3I can be an irritant. Do not allow the CF3I gas to blow directly on skin or face. Wear safety glasses and a glove. Remove the cap and VCR gasket on the outlet of MV-83. Crack open MV-83 to allow a bit of water then CF3I gas to escape. If more than 10 ml of liquid water starts to come out, temporarily close MV-83, allow more CF3I to boil, and repeat until you consistently get just CF3I gas.
7. The CF3I needs to be recovered to prevent release indoors. Obtain a clean, high purity, 10 gallon (37.9 liter) Alloy Products Corporation vessel and record it's starting empty mass. [Reference tare = 15.0 kg].
8. Place the APC vessel in an empty container that will later serve as an open fluid bath. The APC vessel will need to be secured to prevent it from floating when the water is added. Do not add water now or the APC vessel would start cooling prematurely.
9. Connect a clean Teflon transfer line between the CF3I storage container gas port and MV-83. A fine particulate filter and pressure gage should be part of the transfer line. The transfer line also will need a tee to a vacuum pump for line evacuation. Enclose the transfer line to prevent light exposure to the CF3I.

10. Pull a vacuum on the transfer line and APC vessel. Confirm that the transfer line and vessel is clean and leak free by achieving an ultimate pressure equal to the pump ultimate pressure with no observable rate of rise with the vacuum pump isolated. Backfill with CF3I gas from MV-83 to a positive pressure of 10 psig. Close MV-83 and watch the pressure gage to verify that pressure does not bleed down during a few minutes time. Use liquid Snoop at the connections on the transfer line to look for leaks at positive pressure. When it is verified that no leaks exist, proceed to the next step.
11. Repeat an evacuation and backfill to slightly positive pressure with CF3I at least 2 more times. Close the APC vessel gas port valve. Leave MV-83 open.
12. Fill the water bath around the APC vessel. Connect a chiller to the fluid bath. Cool the APC vessel and water bath to just above freezing, approximately 5 C.
13. Record PT83 pressure with the APC port valve closed.
14. Open the APC gas port valve to allow CF3I gas to flow from the bubble chamber into the APC vessel. As CF3I is condensed into the APC vessel, more CF3I will boil to maintain the pressure in the bubble chamber. The pressure may vary by as much as 10 psig as the pressure reduces and then more boiling occurs.
15. Optionally run the chiller for the bubble chamber at a set point of 40 C to warm the upper part of the expansion volume and gaseous CF3I. As the glycol warms, it will expand and may decrease the expansion volume bellow ZT-4 position. Open MV-25 and run the glycol pump as necessary to keep the bellows positive, between +0.0" and +0.5".
16. As an option, record the mass of the APC vessel lifted out of the water bath at an interval of 1 or more hours to determine the condensation transfer rate. The rate was about 1 kg per hour during the Dec. 29, 2009 operation.
17. The APC vessel gas port valve can be closed to stop the gas transfer procedure as necessary.
18. When PT-83 pressure decreases, there will be no more liquid CF3I being available to boil. ZT-4 will start to decrease, (approximately 0.8") as ZT6, the glycol cylinder moves from 0.0" to 8.0". Open MV-25 and run the glycol pump to keep ZT-4 between +0.0" and +0.5".
19. When PT-83 decreases to nearly the saturation pressure in the APC vessel (24 psig at 5 C), close the gas port valve on the APC vessel because no more CF3I can be recovered. Stop the glycol pump and close MV-25 and MV-27.

20. Run the vacuum pump on the transfer line to further decrease PT83 pressure to a value of around atmospheric pressure. As the pressure decreases, the expansion volume ZT-4 will decrease lowering the outer pressure vessel glycol pressure as well. As necessary, run the glycol pump to keep the differential pressure across the bellows/jar to less than 12 psid. Isolate the vacuum pump and stop the glycol pump (close MV-25 & MV-27) when pressures are reduced.
21. Record the initial liquid level in the glycol de-gassing tank on LI-11. A volume of 70 gallons needs to be available to accept the contents of the outer pressure vessel system.
22. Close (or check closed) MV-48 on the gas side of the de-gassing system. Close MV-49 on the liquid side of the de-gassing tank. Open MV-79 and start the vacuum pump to evacuate the de-gassing tank. This step can be started earlier in the procedure.
23. Close (or check closed) MV-30, MV-32, MV-37, MV-38, and MV-50. Open MV-39. Open MV-26 and MV-36 on the liquid drain line. Open MV-7, MV-3, MV-33, and MV-31 to connect the high point volumes together.
24. Open MV-49 to pull a soft vacuum on the outer glycol vessel and bubble chamber. The bubble chamber may expand. Keep an eye on the differential pressure across the bellows/jar and stop or make changes to limit the differential pressure to +/- 12 psid. Open MV-20 to allow air to enter the glycol volume. The glycol level should drop as liquid is sucked into the de-gassing tank via the drain line.
25. The net volume of the outer vessel is about 70 gallons of glycol. Record the de-gassing tank liquid level, LI-11 versus time to get an idea of liquid transfer rate. The rate was 0.5 gpm (5 gallons every ten minutes) on Jan. 4, 2010.
26. When the liquid is drained, bubbles will be seen passing in the liquid filter, F-58. Close MV-39, MV-7, MV-3, MV-33, and MV-31 then open each one alone one at a time for about 1 minute to create a single air backfilling path. Leave MV-31 open and then open MV-27 to allow fluid to be sucked down the recompression hose. A significant increase in air bubbles passing through F-58 will be seen when the hose is emptied. Close MV-27 and MV-31.
27. Close MV-36, MV-26 and MV-49 on the drain line. Close MV-20.
28. Disconnect the compressed air line. Leave MV-61 the air tank drain open.
29. Close (or check closed) MV-83 and disconnect the gas transfer line between the bubble chamber and APC vessel. Remove the APC vessel from the water bath and weigh its mass to determine the amount of CF3I transferred.

30. Close isolation valves at the outer pressure vessel. Close MV-5, MV-6, MV-12, MV-53, and MV-28.
31. Record final pressures, positions, levels, weights, configuration etc. in the log book.
32. Use the vacuum in the de-gassing tank and appropriate valve line ups to suck glycol and air into the de-gassing tank as hoses are disconnected from the outer vessel.
33. The END.