Tips for Using Vacuum Pumps on Vacuum Manifolds

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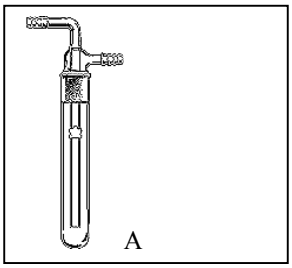
I. Tips for Component Selection

A. Glass Cold Trap



1.Cold Trap Selection. Two piece glass cold traps (A) are easier to clean than one piece traps(B). The reason is two-piece traps permit easy removal of the tube containing condensed vapors. The tube can be removed without disconnecting the trap body from the rubber hose. A two-piece glass cold trap with an inner tubing of 25 mm O.D. makes a good multiple purpose trap. (Kontes 926035-0025). The Kontes trap has serrated hose connections accepting ½" ID hose. Keep a spare tube available to make it easy to clean trap without waiting to clean the dirty tube. (Kontes 926252-0025). Before installing the clean tube, place inner tubing of the trap in a beaker and rinse off condensate with alcohol or acetone. A one-piece trap(B) is more time consuming to clean since it requires disconnecting the whole trap from the hose.

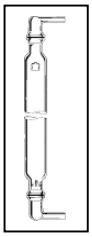
2. When and why to Clean Cold Trap. Cold traps should preferably be cleaned at the end of each day's use Daily cleaning keeps the trap efficiency high by maximizing glass surface area and prevents accidental ingestion by the pump the following day of trapped chemicals re-evaporating over night when the trap warms up.

If you run out of time at the end of the day to clean trap, be sure to shut off the pump and release the vacuum between the pump and trap. The reason is dry ice slurry or liquid nitrogen traps normally will not stay completely cold through the whole night. If the pump is open to the trap overnight, you will sublime condensate out of the trap and into the pump.

B. Acid Neutralization Trap

1.When to use an Acid Neutralization Trap. Use an acid neutralization trap between the pump and the cold trap when drying or distilling samples containing acid, particularly strong acids. The sample may be acidic or the acid may be present in the precipitate if the sample was washed with acids during preparation.

2. Constructing an inexpensive Acid Neutralization Trap.



An acid trap can be constructed by installing potassium hydroxide pellets in a Drying Tower (Kontes 655910-0000). The pellets are easily added and removed from the joint on top of the tower. PLEASE BE SURE TO PUT GLASS WOOL on either side of the potassium hydroxide pellets to prevent the pellets from being drawn into the pump or accidentally migrate into the vacuum hose. Replace potassium hydroxide once you see the shape of the pellets start breaking-up.

C. Dial Vacuum Gauge

1. Why Monitor Vacuum Level. A vacuum gauge is a critical tool for telling you if your vacuum system is leak-tight. Leaky systems cause a high vacuum pump to exhaust more oil, run noisier, run hotter, requiring more frequent oil additions and shortens the service interval of the pump.

2. Vacuum Gauge Options. Absolute pressure gauges are available –McLeod Gauge, manometers, electronic gauges – but can be expensive and some do contain mercury. A dial gauge is an inexpensive tool for giving you

"working" vs "not working" information about your vacuum manifold. A dial gauge is inexpensive because it only gives the vacuum level below atmospheric pressure. Welch offers a dial gauge/regulator kit, part no. 1421, which comes with a dial gauge, vacuum release valve, 3/8" ID hose, and connecting hardware. For example, if a manifold is opened to a vacuum pump and the dial gauge doesn't show 29"Hg or better, the vacuum manifold likely has a leak. Location of the leak comes from systematically checking each connection and looking for a change in vacuum level on the gauge.

D. Vacuum Hose & Hose reducers

1.Why is hose important? Poor hose is a very common reason for a leaky manifold. Using a leak tight vacuum manifold, the drying time is shortened and the service life of the pump is extended.

2.Vacuum Hose Options. Welch recommends red rubber vacuum hose with hose clamps to connect the manifold to the trap(s) and to the vacuum pump. Rubber hose holds up better over time and the vacuum manifold remains leak-tight. Avoid using Tygon tubing since it will become brittle over time leading to leaks. Tygon hose dimensions change as plasticizer in the Tygon is removed by solvent vapors. Change vacuum hosing every year.

3. Hose Reducers. Be sure to have the proper sized reducer to make connection from manifold to trap to pump. Leak at reduction points will cause the pump to run at higher pressures than need be resulting in a shorten service interval for the vacuum pump.

II. Tips for Operation & Maintenance

A. Vacuum Manifold.

1. Grease lubricated stopcocks. Visually inspect the grease on lubricated stopcocks when turned. Verify that the grease moves smoothly. If you see air streaks in the grease as the stopcock is turned, use a heat gun to warm grease to get grease to flow smoother. After done with drying the sample, clean with solvent and re-grease the affected stopcocks.

2. Teflon valves. Regularly visually inspect o-rings to insure in good condition. If you feel a change in the when the valve is turned, clean out glass valve with solvent and replace o-ring.

B. Cold Trap.

1. Maintaining a Cold Trap. Keep slurry topped-up with crushed dry ice. An isopropyl and crushed dry ice slurry works best to give maximum refrigeration with minimal foaming when dry ice is added to warm alcohol. Add dry ice slowly to warm alcohol and stir.

2.Cleaning Trap. Remove trap at end of day and set in fume hood to warm up. Have a spare tube available for using the following day. With a two-piece trap, cleaning the tube is easy to do. You simply remove the tube containing the condensate and put into a fume hood to warm up overnight. In the morning, you can properly dispose of the liquid left behind. With a one-piece trap, the process is similar to a two-piece trap. The main difference is you need to disconnect the glass tubing on the trap from the vacuum hose to clean the trap.

3. Trapping sulfur compounds and DMF. Please be aware that when sulfur compounds and DMF are ingested by a pump the compounds rapidly mix with the pump oil. The chemical mix then experiences heat and shear forces inside the pump. The result is the oil tends to discolor quickly and the oil viscosity increases. The high oil viscosity becomes particularly apparent when the oil cools down after the pump is turned off making it hard to change the oil. High grade synthetic oils(Welch Gold Oil 8995G-11) do hold up better than oil commonly used with high vacuum pumps, but there is no substitute for good trap maintenance - keep trap full of dry ice during day and clean the trap at the end of the day.

4. Start-up of a cold trap. Prior to turning on the vacuum pump, first load trap with crushed dry ice to create slurry before turning on pump. Wait 5 to 10 minutes while trap cools before turning on the pump. This pause will allow a refreezing of trapped chemicals and improve the traps efficiency when pumping on the manifold. This step will length the time interval between oil changes.

C. Acid Neutralization Trap.

1.Location of Trap. Be sure the acid neutralization trap is located between the pump and the cold trap. This allows the cold trap to condense the solvents and not enter the acid trap and reduce its effectiveness.

2. Changing charge. When you see the potassium hydroxide pellets start to crumble and lose shape, it is time to change the charge in the Drying Tower.

D. Vacuum Pump Maintenance tips

1. Reason Most Pumps Fail: The most common reason for pump failure is loss of lubricity of the pump oil. Pump oil maintenance is the key to extending pump service life.
2. When to change pump oil. Pump oil should be changed if you see the oil discolor, the oil level rise, or you suspect that chemicals were ingested by the pump due to low refrigerant level in the trap. Welch recommends an oil maintenance program be developed for the pump. First start with fresh oil and record the date. Monitor the oil condition daily for the first week and then weekly there after. Once a change in the oil condition occurs, change it. This process gives you an estimated oil change interval for the application. When high concentrations of organic solvents and/or acids are pumped, it is not uncommon to change the oil at least once a month, sometimes more often.
3. How to change pump oil. Change the pump oil when the pump is still warm so that the oil drains easily – similar to what you do for a car oil change. If contaminated oil is allowed to cool, the oil is harder to drain completely out of the pump and possibly problem will occur when trying to restart the pump. A good way to get the contaminated oil out of the pump mechanism is to add 20-30 milliliters of oil into the inlet and turn the pump on with exhaust port open and drain closed. Drain the flushed oil. Repeat until oil comes out "clear."
4. How full to keep oil level. Keep oil level at full mark.
5. Pump location. Locate vacuum pump with good ventilation around. Do not position motor against wall or cabinet door to prevent the pump from overheating and the thermo-protection of the pump from turning on.
6. Venting Exhaust of Pump. Exhaust vapors should be vented in a fume hood. A common approach is to run vacuum hose from the exhaust port of the pump directly into the fume hood. This prevents build up of organic vapors around the pump that can lead to a hazardous situation without proper ventilation.
7. Exhaust Oil Filter and Oil Mist Recycler. If you are using an exhaust oil filter, check it at least twice a month to see if it has become saturated. Saturation occurs if oil droplets are on the side of the filter. Replace the element as needed. An Oil Mist Recycler is recommended when operating the vacuum pump above 1 Torr(1mm Hg).
8. Releasing vacuum at pump inlet when pump turned off. Good vacuum technique is to release the vacuum at the pump's inlet after the pump is turned off. This is particularly important in "chemical" applications where oil contamination can increase oil viscosity affecting the pump's antisuckback feature.
9. Oil selection. Use specified oil for the vacuum pump. For corrosive or high solvent applications, Welch Gold Vacuum Pump oil(part no. 8995G-11) has excellent resistance to chemical attack and is very stable under hot pump conditions.

III. Tips on Pumps
A.Sizing a pump for application. Too small a pump for an application can lead to premature failure if the chamber under evacuation is larger than what the pump can handle. A useful rule of thumb is "Use a pump with a free air displacement 1 to 1.5 times the volume of the chamber evacuated." (i.e. for a one cubic foot chamber use a 1 to 1.5 cubic foot per minute(CFM) pump).

Bigger pumps don't have shorter service intervals on small chambers compared to a properly sized pump. In chemical applications, a larger pump is detrimental when a trap is used. The reason is the bigger pump with its higher pumping speed is more efficient at subliming off condensed material from cold traps than a small pump. For vacuum manifolds, evacuation of the glassware is inhibited because of small tubing and small bore holes in stopcocks. The effective pumping speed of a 200 L/min becomes equivalent to a 30 L/min pump in this situation. The result is there is effectively no apparent difference in drying times between a 200L/min pump and a 30L/min pump.

B. Using a pump for both a rotary evaporator and a manifold. A rotary evaporator is sometimes linked via a stopcock to a vacuum manifold. If a two-stage rotary vane high vacuum pump is used as the vacuum source for the manifold, be sure to use a vacuum distillation controller with solenoid valve to regulate the vacuum level in the rotary evaporator. A controller is particularly important when volatile compounds(b.p. <100 deg C) are distilled or stripped. Two stage vacuum pump without a controller will give too deep of a vacuum resulting in excessive foaming or bumping leading to rapid contamination of pump. Welch GEM Rotary Evaporator System(8890A-70) is a cost effective solution which works well. The GEM system offers sufficient pumping speed(31 L/min), vacuum to 0.1 mm Hg, vacuum regulation and a rugged gear pumping mechanism for heavy vapor loads coming off a rotary evaporator.