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Proper Operation of Vacuum Based Equipment

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Overview

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Vacuum is required for a number of laboratory procedures. This is most routinely achieved in the laboratory by the use of vacuum pumps. In addition to working at low pressures, vacuum pumps can also be used to enable rapid changing of the atmospheres in a reactor or flask by evacuation and backfilling.

Principles

Vacuum is useful for a variety of purposes in the lab. For example, vacuum lowers the boiling point of liquids and promotes the vaporing process, which is used for vacuum ovens, degassing equipment, and freeze drying. Besides, vacuum generated a pressure difference compared to atmosphere, which is used for filtration and pipettes. Ultra-high vacuum removes air to achieve chemical inertness, which is used for electron beam welding, maintaining a clean surface and chemical or physical vapor deposition. A vacuum pump is a device that helps evacuate a sealed chamber in order to attain a pressure lower than atmospheric pressure. The most commonly utilized pumps in the laboratory are turbomolecular pumps, oil pumps, dry scroll pumps, or water aspirators.

Turbomolecular pumps are often used in laboratory instrumentation, such as inside a mass spectrometer, and can achieve vacuum levels of 10^{-10} Torr. These work by rapid spinning to collide with air or vapor molecules to impact momentum toward the direction of exhaust. The high vacuum levels have a pump suitable for lots of ultra-high vacuum applications. However, air is too dense for a turbomolecular pump to work, and therefore these pumps need a secondary pump to drop the atmosphere pressure down to 1 Torr to enable the turbomolecular pump to work.

Oil pumps are most often used in the lab and typically achieve a vacuum of 10^{-3} Torr. This meets most of the general lab applications, and they are easy to operate. Oil is used to lubricate and seal the pump, which helps to achieve deep vacuum. However, the use of oil also brings the problem of oil change and waste oil disposal.

The dry scroll pump, which has the ability to achieve an ultimate vacuum level of 10^{-3} Torr, is one of the most common dry pump technologies used in the laboratory setting. The dry scroll pump works with two interleaved spiral scrolls moving eccentrically and compressing air and vapor towards exhaust. This pump doesn't need oil, and also pumps with a faster rate, which is attractive for some applications like a glove box. However, tip seals are needed to keep vapors in the correct channel, but these tip seals are wear parts and needed periodic maintenance.

Water aspirators, which are also called water jet pumps, are usually attached to the lab sink faucet and could achieve a vacuum level of 10-15 Torr. These work by utilizing fast flowing water to create vacuum in the side arm. Due to their low costs, these were historically popular to achieve deep vacuum. However, water is wasted and the vacuum level is not high.

The choice of the type of pump is dictated by the end application and the quality of the vacuum ultimately required. Irrespective of the pump used, the generation of vacuum leads to the possibility of implosion or explosion hazards. The following protocols are outlined to minimize the risks associated with the use of vacuum equipment and to ensure safe working conditions.

Procedure

1. Use of Personal Protective Equipment

1. Safety glasses, lab coats, and face shields must be utilized when working with or near a vacuum apparatus.
2. A blast shield must be utilized to prevent flying glass or debris resulting from a sudden change in pressure.

2. Use of Proper Tubing and Equipment

1. Always use tubing, glassware, and other equipment that is rated for use with vacuum. Improper use can result in material failure and cause explosion/implosions.
2. Check the glass and tubing regularly for defects/cuts, as these can easily crack/break under vacuum.
3. The exhaust of the vacuum pump must be connected to a fume hood or a scrubbed building exhaust. This is particularly crucial if the vacuum is utilized on a system utilizing corrosive or toxic chemicals.
4. Depending on the experiment and extent of vacuum involved, a guard or protective barrier between the operator and vessel under vacuum should be employed. These can be the same type of barriers used to isolate operators from high-pressure equipment.

3. Traps

1. Always use a trap between the vacuum source (pump) and the apparatus that utilizes the vacuum. The trap protects the expensive vacuum source from damage in case of accidental leaks or material back flow into the vacuum line.

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2. The traps also help to prevent vapors/odors from being emitted into the exhaust of the pump.
3. The traps are usually cryostatted using dry ice or liquid-nitrogen baths. Extreme care must be taken while utilizing such cryogenic temperatures, and proper PPE must be used to transfer the coolants in and out of the traps.
4. Since the use of cryogenic fluids (*i.e.*, liquid nitrogen) for cooling purposes can lead to the liquefaction of oxygen, vacuum systems **must** be under vacuum before and during operation. After achieving the desired vacuum level, the dewar containing the oil trap can be subsequently filled with liquid nitrogen. After completion of the experiment requiring vacuum, while still under vacuum, remove the Dewar flask from the liquid nitrogen filled trap, let the trap warm to room temperature, and slowly open the system to atmospheric pressure.
5. The Dewar flasks themselves are under vacuum and must be handled with utmost precaution as they can instantaneously implode. Always use proper PPE when transporting or working with Dewar flasks.

4. Bleed lines

1. The vacuum lines must be slowly bled before disconnecting from the traps and vacuum source. A sudden change in the pressure stresses the materials and can cause premature fracture and explosions.

5. Glassware coating

1. Glassware larger than 250 mL that is utilized with vacuum equipment must be shrouded with either tape, netting, or a plastic coating to reduce the chance of flying debris, in case of an explosion. This includes traps, dewars, rotary evaporators, and any other glassware maintained under vacuum.

Applications and Summary

Operations requiring vacuum have multiple hazards associated with them. Vessel implosion can lead to flying glass and other materials, the release of chemicals to the working environment, and potentially fire due to the condensation of liquid oxygen. Vacuum operations should be set up properly and only operated after potential risks have been identified and properly mitigated.

References

1. NRC (National Research Council). *Prudent Practices in the Laboratory. Handling and Management of Chemical Hazards*. National Academy Press: Washington, DC, 2011.
2. Laboratory Vacuum Pump Buyers' Guide, 2012.

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