JoVE: Science Education

Proper Operation of Vacuum Based Equipment

--Manuscript Draft--

Manuscript Number:	10377
Full Title:	Proper Operation of Vacuum Based Equipment
Article Type:	Manuscript
Section/Category:	Manuscript Submission
Corresponding Author:	Robert Rioux UNITED STATES
Corresponding Author Secondary Information:	
Corresponding Author's Institution:	
Corresponding Author's Secondary Institution:	
First Author:	Robert Rioux
First Author Secondary Information:	
Order of Authors:	Robert Rioux
Order of Authors Secondary Information:	

PI Name: Robert M Rioux & Ajay Sathe & Zhifeng Chen

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Overview:
Certain laboratory procedures require the use of reduced pressure or vacuum. 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 oven, degassing equipment and freeze drying. Besides, vacuum generated a pressure difference compared to atmosphere, which is used for filtration, pipette. Ultra-high vacuum removes air to achieve chemical inertness, which is used for electron beam welding, maintaining the 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 lab instrumentation such as inside a mass spectrometer and could achieve vacuum levels of 10^{-10} Torr. It works by rapid spinning to collide with air or vapor molecules to impact momentum toward the direction of exhaust. The high vacuum levels bring the pump suitable for lots of ultra-high vacuum application. However, air atmosphere is too dense for turbomolecular pump to work and therefore 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 could achieve vacuum of 10^{-3} Torr. This meets most of the general lab applications and they are easy to operate. The oil was 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.

Dry scroll pump is one of the most common dry pump technologies seen in the lab and could achieve the vacuum level of 10^{-1} Torr. Some newer models could even achieve vacuum level of 10^{-3} Torr, same as oil pump. Dry scroll pump works with two interleaved spiral scrolls moving eccentrically and compressing air and vapor towards exhaust. This pump doesn't need the oil and also pump with a faster rate, which is attractive to some applications like glove box. However, tip seals are needed to keep vapors in correct channel but these tip seals are wear parts and needed periodic maintenance.

Water aspirators, which are also called water jet pump, are usually attached to the lab sink faucet and could achieve vacuum level of 10-15 Torr. It works by utilizing fast flow water to create vacuum in the side arm. With low cost, it was historically popular to achieve deep vacuum. However, lots of water are 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 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

    1.1. Safety glasses, lab coats, and face shields must be utilized when working with or near vacuum apparatus.

    1.2. 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

    2.1. 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.

    2.2. Check the glass and tubing regularly for defects/cuts as these can easily crack/break under vacuum.

    2.3. 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.

    2.4. 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
    3.1. 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.

    3.2. The traps also help to prevent vapors/odors from being emitted into the exhaust of the pump.

    3.3. 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.

    3.4. The system MUST be under vacuum before, during, and after using liquid nitrogen as a coolant; otherwise, liquid oxygen can condense in the trap and cause an explosion. Once under vacuum, submerge the trap in a Dewar flask containing liquid nitrogen. When

done, remove the Dewar flask from the trap, and slowly open the system to atmospheric pressure.

3.5. 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

4.1. 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

5.1. Glassware larger than 250 mL that is utilized with vacuum equipment must be wrapped with tape, netting, or plastic coating to minimize flying debris in case of an explosion. This includes traps and rotary evaporators.

Summary:

Vacuum work can result in an implosion and the possible hazards of flying glass, splattering chemicals, and fire. All vacuum operations must be set up and operated with careful consideration of the potential risks.

References:

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