X.4 An Introduction to Vacuum Line Techniques

1 Safety

Gas cylinders

High pressure gas cylinders store a dangerous amount of energy; always treat them with respect. Cylinders must never be allowed to fall over. Make sure yourself that they are securely fixed, either by being chained to the bench, or (small cylinders only) supported in a trolley or stand. Check the outlet pressure on the regulator before opening the outlet tap to fill any vessel; ensure that the pressure is low enough to be supported safely.

Liquid nitrogen

Minor spills onto bare skin are of no consequence, but spills onto clothing in close contact with skin can cause unpleasant burns. If such a spill occurs (e.g. onto socks or cuffs) remove the article as quickly as possible.

Vacuum systems

All vacuum systems, particularly glass ones, are dangerous. You must wear safety glasses throughout this experiment.

2 Purpose

In this experiment you will learn:

to construct vacuum systems from standard components,

the uses of different types of pressure gauge,

how to locate leaks in vacuum apparatus,

how to use gas cylinders safely,

about pumping speed, and how pipes affect it,

to use liquid nitrogen and freezing mixtures.

As part of the practical you will determine how the vapour pressure of a volatile solvent depends on temperature.

3 Apparatus

Equipment

Check the label by the apparatus to determine the number of the set you are using. The suggested layouts shown later in this script are different for the different sets. In the figures at the end of the instructions A = air admittance valve, P = Pirani gauge, V = gas volume, D = diaphragm gauge. Identify the following components, which should be part of the kit for your experiment:

Vacuum pump A two-stage rotary vane pump is provided. Examine the cutaway model, which is near the emergency exit from the laboratory on the south side of the building, to understand how the pump works.

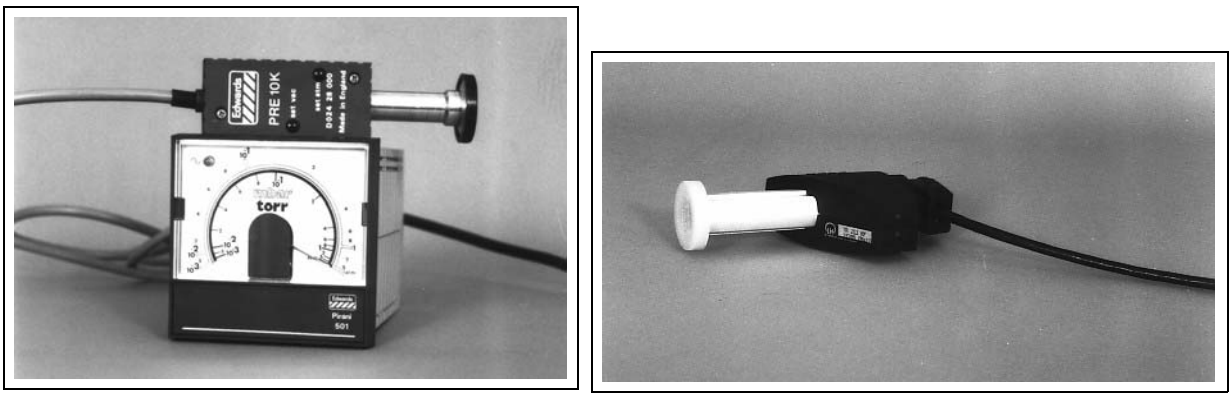


Fig. 1. Pirani Head / gauge set 1 (left); Pirani head set 2 (right)

Pirani gauge This measures pressures between 10^{-3} mbar and 1000 mbar by determining the thermal conductivity of the gas. (What is a 'bar' in the scientific sense? See Appendix B).

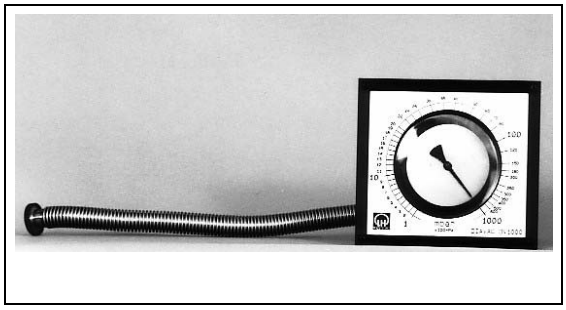


Fig. 2. Diaphragm gauge, set 1 (left). Diaphragm gauge, set 2 (right).

Diaphragm gauge Measures pressures between 1 and 1000 mbar, through the pressure distortion of a diaphragm.

Standard fittings All the components can be connected using rubber O-rings on O-ring carriers and clamps (Fig 3); talk to a demonstrator if you are in any doubt about how to connect the components. There are two standard sizes of orifice in the ends of the pieces where the O-ring carriers fit; 10 mm and 16 mm. Some of the O-ring carriers are made as adapters with a 10 mm section on one side and a 16 mm section on the other; use these to join pieces of different standard.

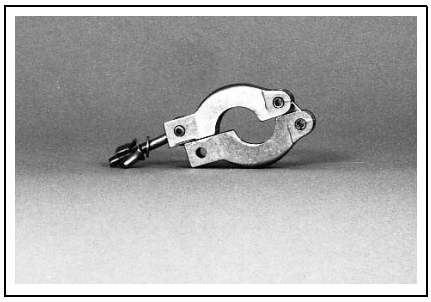


Fig 3. The type of clamp to be used in the experiment.

4 Procedure

The rotary pump contains a considerable amount of oil. When you turn the rotary pump off, if a vacuum remains in the part of the apparatus directly connected to the pump, this oil will be sucked back and may damage some components. It is important, therefore, that when you turn off the pump you immediately relieve any vacuum that may exist in the apparatus. If in any doubt about how to do this, consult a technician or demonstrator. Do not forget to do this, or your experiment may be disrupted as you wait for the apparatus to be cleaned. If you do inadvertently get oil into the apparatus, see the technician without delay.

As a first step, clamp the 10 cm straight tube on the inlet of the pump. By mounting components on this tube rather than directly on the pump, you can raise all the fittings clear of the top of the pump, and you will find it much easier to assemble the components.

Ultimate vacuum

1. Examine the different components, and ensure you understand how they can be connected.

2. To measure the ultimate vacuum of the pump (that is, the lowest pressure which the pump is capable of delivering), fit the Pirani gauge directly on top of the pump. Turn on the pump and gauge, and record the pressure when it becomes steady (do not wait for more than two or three minutes). It should be in the 10^{-2} mbar range or better. If it is not, consult a demonstrator.

3. Once you have measured the ultimate vacuum, turn the pump off and immediately remove the Pirani gauge, to prevent any danger of oil being sucked up. Enter the pressure below.

Ultimate vacuum pressure:

Leak detection

1. Construct a vacuum system using the small vacuum components, including the small gas volume (see below), and the Pirani gauge, but not the diaphragm gauge. Mount the small volume vertically, so that its weight does not bend any fitting. A suitable layout is suggested in setup 1. (Check that you are referring to the correct figure for the number of the set you are using before starting to put the apparatus together.) Be sure that all O-rings are seated evenly between the smooth metal surfaces, and are pulled tight by the clamps. The clamps need to be tightened only enough to make them secure; do not over-tighten.

Fig 4. The "small volume".

2. Turn the pump on, close the air admittance valve, open connecting taps and measure the pressure. The Pirani reading is likely to be significantly higher than the ultimate vacuum of the pump, because one component has a deliberate leak.

3. To locate the leak, use a wash-bottle to squirt SMALL quantities of propanone (acetone) onto likely places, while watching the Pirani gauge. (Do not forget that

acetone is highly flammable and you are working with electrical equipment, so use only very small amounts.) It is best to start from the lowest point and work up, otherwise you may be confused by solvent dripping down onto components underneath the one you are testing. Be careful not to get acetone near the plastic mounting of the Pirani gauge. Acetone has a much higher thermal conductivity than air (Appendix A explains why), so you will get a higher pressure reading when the solvent leaks in.

Likely places for a leak are O-ring joints and seams or welds of the metal parts.

4. When you have found the leak, close the tap, admit air, dismantle the system and take the leaky component to the service room for a vacuum-tight replacement, which you will need later.

Pumping speed

1. To measure the speed of the pump (defined as the volume of gas removed per second), connect the large gas volume (full of air) to the system with the diaphragm gauge, as shown in setup 2.

2. Start timing using the stopwatch supplied when you open the tap to the pump, and record the pressure reading on the diaphragm gauge at regular intervals for a total time of about 7 minutes.

ⓘ Choose the length of interval between readings that you feel will give you the best results, bearing in mind how rapidly the readings are changing.

3. Complete the table on the next page as you work.

4. At the end of the measurement period, open the air leak, then turn off the pump. Work out the average pumping speed (the volume removed per second) during each time interval. Use your results to answer the following question:

Does the pumping speed depend on the pressure?

ⓘ From $PV = nRT$ you can show that $k\,dt = -V_o\,dp/p$ where k is the pumping speed, p is the average pressure during the measurement, t is time and V_o is the volume of the vessel. dp and dt are the corresponding changes in pressure and time. Hint: you may have to discount the first few readings.

Time /s	Pressure / mbar	Pumping speed

You can verify your answer to the question by examining the cutaway model of a rotary pump, knowing that the rotation rate of the blades is constant.

5. Measure the dimensions of the large vessel to estimate its volume and thus get a numerical value for the pumping speed.

Pumping speed .....

Effect of pipes, use of gas cylinders

At low pressures (below about 5 mbar) the pumping speed can be limited by the effect of pipes used to connect parts of a system. To investigate this you will fill a fixed volume with air and see how long it takes to pump it out either directly or through a selection of tubes of different dimensions.

1. Reconstruct the vacuum system as shown in setup 3.

ⓘ A common mistake in this section of the experiment is to place the Pirani gauge on the *pump* side of the different tubes supplied. If you do this you will get meaningless results, so check carefully that you have put together the apparatus as suggested in the diagrams which follow. Try not to bend the tubes out of shape (more than they already are!) since an irregularly bent tube will give poorer results than one with smooth curves.

2. Admit air to the system, including the small volume, then evacuate it, using a stopwatch to determine how long it takes for the pressure to drop from 5 mbar to 1 mbar (Pirani).

3. Once you have taken a measurement, allow air to re-fill the volume, then repeat the measurement. Take at least four measurements.

4. Repeat the experiment, connecting the small volume in turn to each of the sections of tubing provided. Record the results in the table below; calculate an average time for each tube.

Tube length	Tube internal diameter	Time for p to fall from 5 to 1 mbar
Direct connection		

5. You can assume that the pumping speed (inversely related to the time) is proportional to powers of the length and diameter; estimate these powers

Dependence on length .....

Dependence on internal diameter .....

ⓘ You might guess that the time required to evacuate the volume will depend upon the viscosity of the gas and on other factors. For example, water adheres strongly to most metal surfaces, and desorbs (detaches itself) only rather slowly.

Attempt to determine the extent of this effect by repeating your measurements using dry nitrogen, according to the following procedure.

1. Evacuate the small gas volume, close its tap, shut the main tap to admit air to the rest of the system and remove the volume.

2. Take it to the nitrogen cylinder, fit it onto the outlet and fill it to a pressure slightly above atmospheric with nitrogen, following the instructions given nearby.

3. Bring it back to your bench and connect it to the system using what you judge to be a sensible choice from the set of tubing.

4. Evacuate the rest of the system, then use a stopwatch to determine how long it takes after you open the tap to the gas bottle for the pressure to drop from 5 mbar to 1 mbar (Pirani).

5. Repeat, with all the operations being carried out by the second member of the pair.

Dimensions of tube chosen:

Time 1:

Time 2:

Do your measurements show a (statistically) significant difference between the time required for nitrogen and that required for air?

Vapour pressure

1. Modify the system as in setup 4, using the metal cone to adapt to the glass tube.

2. Clean the metal cone and matching glass cone if necessary with solvent and tissues; apply vacuum grease (Glisseal) evenly to the glass and push them gently together.

3. Evacuate the system and check that it is free from leaks.

4. Ask the technician for some liquid nitrogen in an insulating plastic cup or Dewar (wear safety glasses if using a Dewar - Dewars can implode!).

5. Prepare a freezing mixture of ice and salt in a ratio of about 3:1 in another cup and an ice/water mixture in a third. Ice is available from the technician.

6. Shut the tap. Admit air to the system, remove the glass tube and pipette about 1cm^3 of a volatile liquid (use methanol, ethanol or acetone) into it.

7. Replace the glass tube on the vacuum system, close the air admittance valve and freeze the solvent using liquid nitrogen.

8. When it is completely frozen (a minute or two) open the tap to evacuate the air; check that you get a good vacuum with the solvent still being cooled. When the vacuum is good shut the tap to seal the system.

9. With the tap shut, remove the liquid nitrogen and let the solvent warm up. Now successively equilibrate the solvent with

(a) the ice-salt freezing mixture,
(b) ice and water

(c) water at three well-spread temperatures between 0°C and 20°C.

10. Record the temperature and pressure each time. You will need to stir the coolant and wait a few minutes each time for equilibration, when the pressure stabilises.

Solvent chosen:

T (K)					
P					

11. Theory (Appendix A) shows that a plot of lnp against 1/T(K) should be a straight line of slope $-H_{vap}/R$. Prepare a suitable plot, and calculate the enthalpy of vaporisation.

Clearing up

1. With the tap still shut, condense and freeze the solvent in liquid nitrogen again.

2. When it is well frozen, admit air and remove the glass tube.

3. Turn off the pump, open the tap, then dismantle the whole system and leave all the separate parts in the plastic tray.

4. Wait for the solvent to melt, then pour it into the appropriate residue bottle. Wipe the grease off the glass and metal cones.

5. Ask a demonstrator to check your results and apparatus and, if all is well, sign you off.

6. Add your results to the cumulative results file (unless they are obviously wrong!)

Chemical Properties, Hazards and Emergency Treatment

Acetone (propanone)

Colourless aromatic liquid. Highly flammable, so presents a considerable fire risk. Harmful if swallowed, or if breathed in over a prolonged period. Skin contact will lead to removal of natural grease and drying of the skin. Skin or eye contact: wash off with water. If swallowed: wash out the mouth with water, call for medical help.

Ethanol (ethyl alcohol)

Colourless aromatic liquid. Flammable. Harmful if consumed in quantity. Skin or eye contact: wash off with water. If swallowed: wash out the mouth with water.

Methanol (methyl alcohol)

Colourless aromatic liquid. Very flammable, so methanol presents a considerable fire risk. Very harmful if swallowed - methanol can cause blindness. Harmful if absorbed through the skin or inhaled. Skin or eye contact: wash well with water. If swallowed: wash out the mouth with water. If the quantity swallowed is substantial, seek immediate medical help.

Appendix A Questions and answers

This experiment may have raised some questions in your mind, both practical and theoretical.

Why is it better to use nitrogen rather than air to measure pumping speed?

Air has a very variable water content. Water adsorbs strongly on (sticks onto) most surfaces, and the time it takes to pump off (outgas) may be long.

Why must air be pumped away before the vapour pressure is measured?

We want the pressure to be that of the vapour alone. When a vessel is open to the atmosphere the total pressure $p_{vap} + p_{air}$ is equal to the atmospheric pressure. In a fixed volume both pressures would vary with temperature and would be impossible to separate.

How does thermal conductivity depend upon pressure?

This is bookwork. Briefly, thermal conductivity is independent of pressure above about 10 mbar because changes in the number of particles present and the distance they can travel carrying heat (mean free path, proportional to 1/p) effectively cancel out. At low pressures the distance molecules can travel becomes fixed, because it is limited by the size of the container; the conductivity is then proportional to the number of particles per unit volume which in turn is proportional to pressure.

Why is the thermal conductivity of acetone higher than that of air?

The amount of heat each molecule can carry depends on how many ways it has of storing energy. An atom has only translational energy. A diatomic molecule also has two rotations and one vibration. A non-linear polyatomic molecule has three translations, three rotations and many ($3n$-6, where n is the number of atoms) vibrations, each of which can store and carry an amount of energy of order kT (in the classical limit). For details of this see the discussion of Equipartition in any physical chemistry text.

How does vapour pressure depend upon temperature?

More bookwork! Thermodynamic theory gives the Clausius-Clapeyron equation, whose integrated form is

$$\ln p = - H_{vap} / RT + \text{const}$$

Thus a plot of ln p against 1/T has slope $-H_{vap}/R$ giving the enthalpy of vaporisation. The normal boiling point, at which the vapour pressure is one atmosphere, can be read off the graph and fixes the value of the constant.

What limits the ultimate vacuum of the rotary pump?

The limiting vacuum is set by leakage through the pump from the exhaust (at atmospheric pressure) to the inlet, caused by mechanical imperfections and solubility of air in the oil sealant. In a two-stage mechanical pump (as used here) the inlet of one stage is the exhaust of the next, greatly improving the ultimate vacuum.

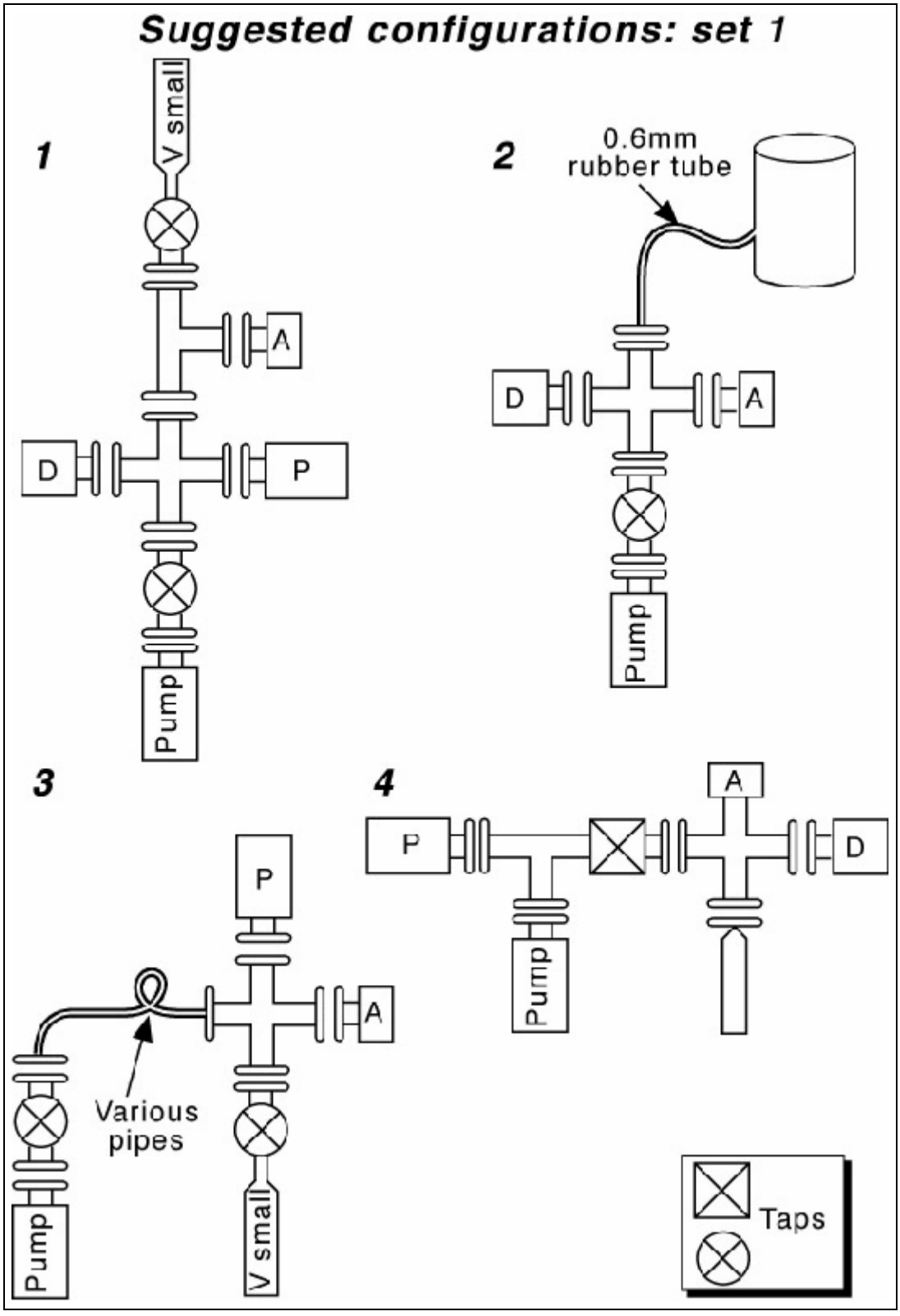
How are better vacua obtained?

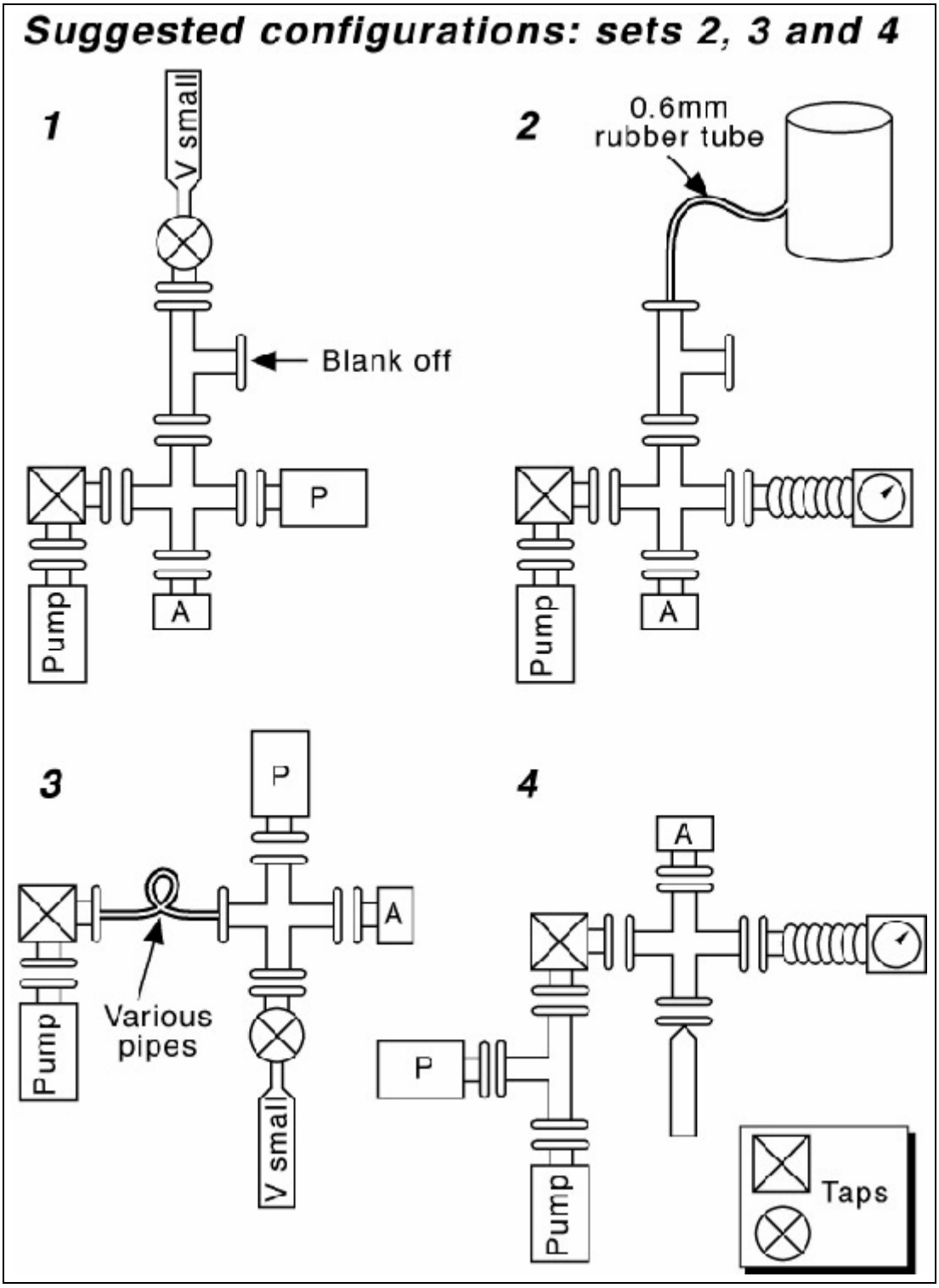
High vacuum (down to about 10^{-8} mbar) is achieved either with turbomolecular pumps (multiblade fans, turning extremely fast), or using diffusion pumps, in which gas is entrained by a rapid flow of vapour and droplets of a relatively involatile fluid such as mercury (see cutaway model). Both sorts of pump must be exhausted by a rotary vane mechanical pump.

For ultrahigh vacuum (below 10^{-8} mbar) down to the best vacua obtainable on earth (about 10^{-15} mbar) pumping usually involves the removal of gas-phase molecules by fixing them onto solid surfaces; the main techniques are cryopumping (condensing at liquid He temperature), ion pumping (ionisation, acceleration, shooting into surface or burial by sputtered deposits) or titanium sublimation pumping (burial under continuously depositing metal).

Appendix B Units of pressure

1 atmosphere = 101.3 kPa
= 1.013×10^5 N m^{-2}
= 760 Torr (1 Torr is the pressure produced by a column of mercury 1mm high)
$\approx$ 1 bar ($\equiv 10^5$ Pa)


Suggested configurations: set 1
1
V small
A
D
P
Pump
2
0.6mm
rubber tube
D
A
Pump
3
P
A
Various
pipes
Pump
V small
4
A
P
D
Pump
Taps


Suggested configurations: sets 2, 3 and 4
1
V small
Blank off
P
Pump
A
2
0.6mm
rubber tube
Pump
A
3
P
A
Various
pipes
Pump
V small
4
A
P
Pump
Taps