Sublimation

A compound is said to sublime if it passes from the solid state directly into a gaseous form without first becoming a liquid. This property is usefully employed in reverse, as a method of purification. Essentially, the compound is allowed to become a gas and is then solidified on a cool surface. Because the cooling compound does not pass through the liquid phase, the material is not washed from that cool surface. Hence the solid collects on that surface in a form that is free from non-sublimable impurities.

Generally, the technique of sublimation is an excellent method for growing crystals. If the process is slow then the quality of the crystal formation is usually better. Iodine is a fine example of a sublimable material. It readily sublimes from a sun-warmed glass surface to another part of the glass vessel that is slightly cooler.

Most sublimations are carried out at low pressure, although this is sometimes for convenience rather than absolute necessity. It is important that the sample is free of solvent, otherwise the solvent will distil onto the cold surface and wash any sublimed material back into the flask. This usually means that the sample is dried on a vacuum line at, or below, room temperature before the cold probe is inserted into the flask.

Choose a probe of length such that there is only a small, but distinct distance between the sample and the probe. A shorter distance will mean that the heating bath need not be quite so hot for the material to solidify on the probe. A stirrer bar should not be used in the flask as this might cause the sample to spatter onto the probe.

A temperature gradient is necessary between the sample (hotter) and the collecting probe (colder). There is a relatively limited choice for generating the cold temperature: running water, ice , dry ice/acetone or liquid nitrogen. The common choice is between a water-cooled probe and a dry ice/acetone probe. The warmer temperature is usually generated by a water or oil bath.

Air-sensitive compounds may easily be handled, as the manipulations can take place on a vacuum line. At the end of the sublimation, it will be necessary to allow the probe to warm to room temperature under nitrogen before removing to another 3-neck flask previously filled with nitrogen. Caution: letting a cold surface come into contact with an open nitrogen system can cause solvent from other reactions to condense. Choose a line that no-one else is using. The 3-neck flask will be necessary to allow the probe to sit in one neck, nitrogen to pass into a second and a spatula to scrape off the solid from the probe through the third. The transfer should take place with the probe at room temperature to discourage the condensation of atmospheric moisture on to the outer layer of the sample.