



Sublimation

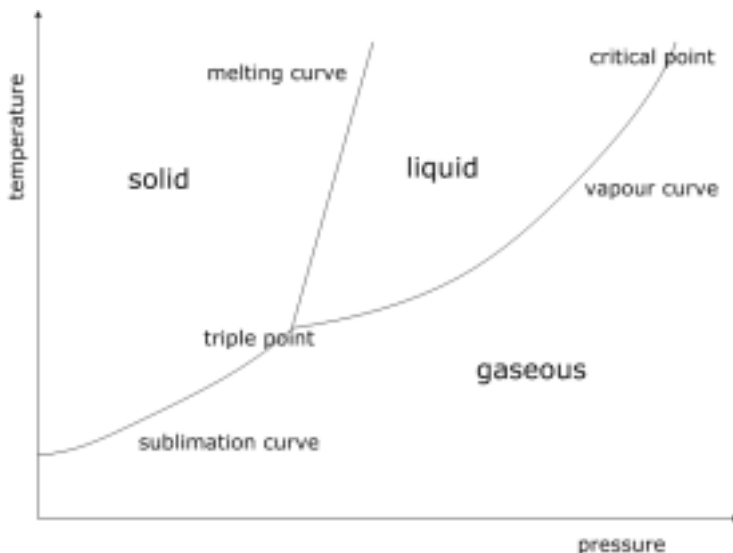
Sublimation is the term used to describe the transition from solid phase to gas phase, without going through the liquid phase. In principle, this unique behaviour is possible with any substance or mixture which has a melting temperature at a specific pressure below a certain point, known as the Triple point. The unique property of sublimation allows products to be separated, similar to that of evaporation, where liquids are separated from dissolved solids. Sublimation gives very pure products, without the need of additional solvents, an advantage over conventional methods.

Theoretical background

The diagram below shows equilibrium curves of different aggregates and their physical phases, as a function of temperature and pressure. The triple point is the point where, for a specific substance, the solid, liquid and gaseous phase are all in equilibrium, at a specific temperature and pressure. Vapour pressure of solid material depending on the temperature is displayed graphically by the sublimation pressure curve. It lies below triple point. If a solid material is heated below the pressure of the according triple point, it changes directly into the gaseous phase without previous liquefaction.

This phenomenon is called sublimation. The reverse effect can also occur as well. Vapour can condense into crystals directly, by passing the liquid phase. Sublimation occurs more commonly than just in the laboratory, in fact each solid which has a smell indicates sublimation properties.

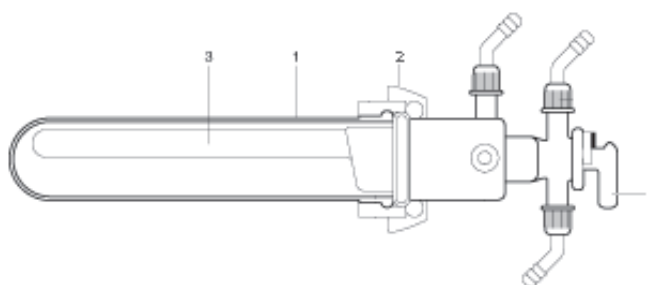
Because sublimation is pressure dependant, it is possible to set conditions in which the substance falls below its triple point. Thus even solid materials which melt at normal pressure, can sublime at a reduced pressure (vacuum sublimation).



Method

The BÜCHI Glass Oven B-585 with sublimation accessory allows substances to be sublimated and purified product deposited on the cold finger.

First, the sample is placed in the drying flask (1) and distributed evenly along the base. Up to 10 g of sample may be sublimated. The sublimation accessory is then inserted into the oven, held by flange screws (2). Water is then pumped through the cold finger (3), to cool it. The stopcock (4), is for evacuation and aeration of the oven. The oven is then heated until sublimation occurs and the product deposited on the cold finger.



Characteristics

Sublimation usually gives a much better purity than crystallisation, and a higher degree of purity can be achieved by repeating the process. In addition, sublimation with the Glass Oven B-585 also permits the purification of small quantities.

Example of application:

Sublimation of a mixture of 1 g (L) tartaric acid and 1 g naphthalene

Process parameter

Vacuum:	25 mbar
Temperature:	80 °C
Time:	60 min.
Result:	Sublimate of naphthalene 0.95 g



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