

Producing pure substances is a very important process, especially in the pharmaceutical industry. Separating insoluble solids from solvents is easily achieved using a simple technique called filtration. Soluble solids again are easily separated from solvents by evaporation, and chromatography is used to separate a mixture of liquids. But what happens when we have two soluble solids that we want to separate? In this lesson we will look at recrystallisation, a commonly used process in the pharmaceutical industry.

What is recrystallisation?

Recrystallisation is a very important purification technique, purifying substances by removing unwanted by-products. It is also used to manufacture the correct crystal size and shape of a material. These factors can have a very significant impact on how a medicine acts when taken by a patient. The same principles and techniques of recrystallisation can be applied both on a laboratory and industry scale.

What are the principles behind recrystallisation?

The process depends on two principles, the fact that substances tend to be more soluble in a hot solvent than in cold solvent, and that each solute tends to behave as though it were alone in the solvent.

How do I know what solvent to use?

Using the correct solvent is a very important part of the process. The solute must be insoluble in the solvent at room temperature, and as the temperature of the solvent increases, the solubility of the solute also increases. It is also important that the impurities present are soluble in the solvent at room temperature and insoluble at higher temperatures.

An excellent substance for showing this process is benzoic acid ($\text{C}_6\text{H}_5\text{COOH}$). A molecular crystal rather than an ionic crystal, shown by its low melting point of 122°C , benzoic acid is used as an anti-microbial agent and is found in toothpastes, mouthwashes, cosmetics and deodorants.

Impure benzoic acid contains the impurities phthalic acid and benzylbenzoate. If the impure sample is dissolved in a minimal volume of hot solvent, in this case boiling water – and filtered to remove insoluble impurities, the resulting solution will contain dissolved benzoic acid as well as dissolved impurities.

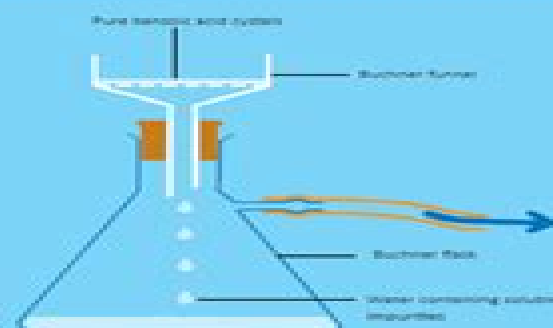
Upon cooling, the benzoic acid crystals come out of solution as its solubility in the solvent decreases. The impurities will remain in solution and can be filtered off.

It is very important when carrying out this experiment that rubber gloves are worn, because organic substances can be absorbed through the skin, and as well, goggles should be worn to protect your eyes from hot liquids which may splash out from the flask.

Fig.1 Filtration to remove insoluble impurities



Fig.2 After cooling, the remaining solution is filtered off



How can we check the purity of our substance?

A pure substance has a fixed melting point while an impure substance melts over a wide range of temperatures and at a lower temperature than the pure substance. The melting points of almost all substances are available in tables.

To check the melting point place a few crystals of the sample on an aluminum block which has a thermometer inserted. Use a hot plate to heat the block slowly. Watch the crystals carefully and note the temperature at which they begin to melt and the temperature at which they have all melted. Compare this value with the value found in the table of pure substance. Another method of checking the melting point of crystals is to use a narrow capillary tube in an oil bath. The capillary tube has one end open and the other end sealed. The

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