

Using a separatory funnel:

A separatory funnel is the device which is most commonly used to perform liquid-liquid extractions. A variety of shapes and sizes is available but the method of operation does not vary. The funnels have a tap at the bottom through which the contents may be drained.

Glass taps are lubricated with a minimum of grease. A plastic screw is provided to keep the tap in place. Teflon taps do not require grease and are held in place by a nut and washer.

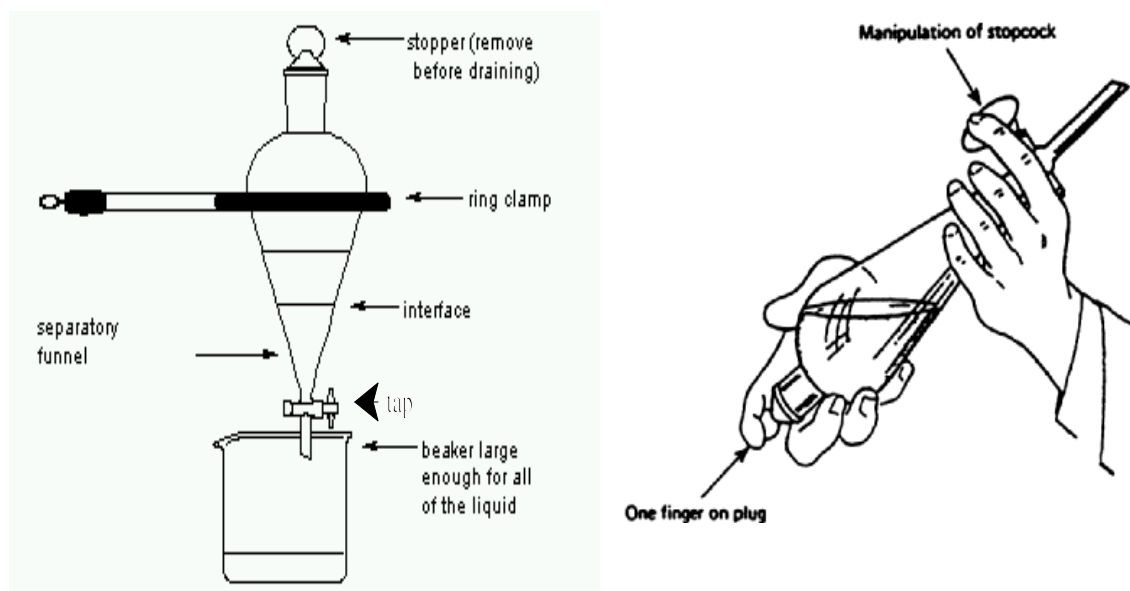


Figure: A liquid-liquid extraction showing the correct use of a separatory funnel.

To perform the extraction, the two liquid phases are added to the separatory funnel, making certain that the tap is closed! The funnel should not be filled to more than three quarters of its height to allow for mixing of the two layers. The stopper is inserted and the separatory funnel held as shown in the diagram.

This method allows manipulation of the tap and at the same time holds the stopper and tap tightly in place. The solvents are then shaken vigorously for 20 - 30 seconds. The shaking process greatly increases the surface area in contact between the two liquids and will allow for equilibrium to be established more rapidly. The separatory funnel must be vented often during the shaking process to relieve excess vapour pressure. The funnel is vented by opening the tap when the funnel is inverted. The stem of the funnel should be pointed into a fumehood during the venting process to avoid spraying anyone with the contents of the funnel. Venting is of particular importance in instances where sodium bicarbonate is used to extract acidic solutions because of the CO_2 released in large quantities.

At the end of the shaking period the funnel is vented a final time and supported on a ring clamp allowing the layers to separate (see Figure). The liquids are separated by drawing off the lower layer; proceed very slowly as the interface approaches the tap.

Most common applications of liquid-liquid extraction invariably involve an aqueous phase. Occasionally, the presence of solute may cause a change in the expected relative densities of the two liquids. The respective layers can be identified by dropwise addition of water to the mixture in the separatory funnel and noting in which layer the drops are miscible.

The most useful hint is to not throw either fraction away until you are sure you know where your product is!