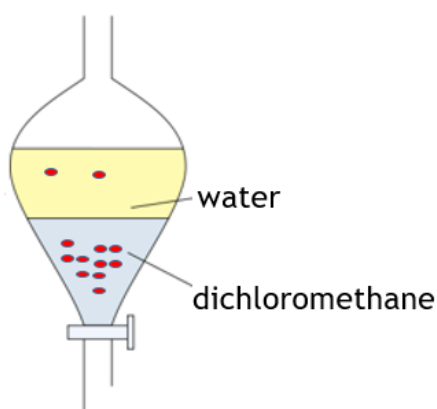


4.9 Key Steps in Laboratory Synthesis Answers

1. a) For a distillation set up, the condenser is placed horizontally and for heating under reflux, the condenser is placed vertically.
b) Reaction mixtures are heated under reflux because it allows prolonged heating without the escape of volatile compounds.
c) A sample of the liquid compound could be placed in a round bottom flask with anti-bumping granules and the distillation apparatus set up. When the compound begins to distil its boiling point will be indicated on the thermometer. This can be compared against a literature value (obtained from a reliable source) and if they match then it indicates that the liquid compound is the desired substance.
d) The water inlet should be at the bottom of the condenser and the water outlet should be at the top of the condenser.
e) Anti-bumping granules are added to ensure that the reaction mixture does not boil too violently.
2. Vacuum filtration is much faster than normal filtration and thus saves time in the laboratory.
3. a) The solvent must be immiscible with water.
The product must not react with the solvent.
The product must be more soluble in the solvent than in water.
b) A diagram that shows the dichloromethane as the lower layer (as it is more dense).
Remember that the equilibrium constant is given by $\frac{[\text{product}]}{[\text{reactant}]}$. This indicates that there should be 5 times more solute in the dichloromethane layer than in the water layer.



4. a) The ether layer forms the upper layer as it is less dense.
- b) A partition coefficient of 12, means that the ratio of solute is distributed as 12 parts in the ether layer and 1 part in the water layer. To find the mass of solute in the ether layer:
- $$(12 / 13) \times 1.1\text{g}$$
- $$\underline{1.02\text{g}}$$
- c) Many organic compounds are more soluble in ether as it is a non-polar solvent and these compounds are often non-polar (from Higher Chemistry, like dissolves in like).

5. a) $n = m / \text{GFM}$

$$n = 2.36 / 118$$

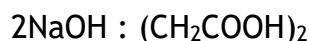
$$n = \underline{0.02 \text{ moles}}$$

- b) NaOH:

$$n = c \times v$$

$$n = 1 \times 0.0348 \text{ (34.8 cm}^3\text{)}$$

$$n = 0.0348 \text{ moles}$$



$$2 : 1$$

$$0.0348 \text{ moles} : 0.0174 \text{ moles IN THE AQUEOUS LAYER}$$

At the start there were 0.02 moles, therefore the number of moles in the diethyl ether layer:

$$0.02\text{moles} - 0.0174\text{moles} = \underline{0.0026\text{moles}}$$

- c) $(0.0026 / 0.0174) = \underline{0.149}$

- d) The extraction process could be carried out twice using two portions of 50cm³ diethyl ether or indeed carrying out four extractions using 25cm³ portions of diethyl ether.

