Acid-alkaline extraction experiment

Phenolic compounds undergo similar salt formation with sodium hydroxide solution. Hence, a sodium hydroxide solution can be used to extract a carboxylic acid or phenolic compound from its solution in an organic solvent, or conversely, an organic solvent can be used to remove organic impurities from a carboxylic acid or phenol dissolved in aqueous sodium hydroxide. Aqueous solutions of sodium bicarbonate likewise convert carboxylic acids to their sodium salts, but are not sufficiently alkaline to form salts of phenolic compounds. This provides an elegant method for the separation of a carboxylic acid and a phenolic compound. First the acid may be removed from solution in an organic solvent by means of extraction with sodium bicarbonate solution, and then the phenol, with sodium hydroxide solution. Inorganic acids are regularly removed from organic solvents by extraction with sodium hydroxide, sodium carbonate, or sodium carbonate solutions. Dilute hydrochloric acid is often used in the extraction of basic substances from mixtures or in the removal of basic impurities. The dilute acid converts the base such as ammonia or an organic amine (R3N) into the water-soluble chloride salt (R3NH+, Cl-). Conversely, organic impurities may be removed from amines by extraction from a dilute acid solution with organic solvents. Sodium salts of carboxylic acids and phenolic compounds are readily reconverted to the parent compounds by treatment with sulphuric or phosphoric acid. The chlorides of amines revert to the original amine upon addition of sodium hydroxide solution.

ETHER IS HIGHLY FLAMMABLE, WHEN THE SEPARATION IS BEING CARRIED OUT, ALL NEARBY FLAMES MUST BE EXTINGUISHED.

OBJECTIVES:

1. To perform an acid-alkaline extraction.
2. To recover benzoic acid and p-dichlorobenzene from its mixture using acid-alkaline extraction method.
3. To determine the percentage recovery of benzoic acid and p-dichlorobenzene.

APPARATUS AND MATERIALS: Separatory funnel (250ml), Buchner funnel, benzoic acid, p-dichlorobenzene, ether, 10% NaOH, concentrated HCl, distilled water, anhydrous CaCl2

PROCEDURE:

The supplied ether solution (40ml was taken) which contained the benzoic acid/p-dichlorobenzene mixture (1g) was poured into a small separatory funnel.

Freshly prepared 10% NaOH (20ml) solution was added.

The funnel was stoppered, shaked well, inverted occasionally and any surplus pressure was released through the tap.

The mixture was allowed to stand.

The stopper was removed and the lower aqueous layer was run into a conical flask.

The residue in the funnel was shaked with another portion of 10% NaOH (10ml) and the lower water layer was run off into the same conical flask.

To the solution in the conical flask, drop wise concentrated HCl was added until no precipitation occurred.

The ether solution was washed with water (30ml).

The lower layer was run off and discarded.

Three or four granules of anhydrous CaCl2 were added to the ether solution and the mixture was shaked occasionally until no turbidity remains.

The ether solution was decanted into clean dry small conical flask.

A boiling chip or two were added and was heated gently on the hot-plate until approximately 15-20ml of ether remains.

Then the ether solution was decanted into a tarred watch glass and was set aside to allow the rest of the ether to evaporate, preferably in the funnel hood.

Meanwhile, the precipitated benzoic acid was filtered using a Buchner funnel and flask.

Then, it was washed with a 5ml portion of cold distilled water.

A current of air was drawn through the product for about 5 minutes to remove as much water as possible.

The crystals were ensured are completely dried by pressing in a folded filter paper.

The weight and melting point of the recovered benzoic acid was determined.

As soon as all the ether had evaporated, the recovered p-dichlorobenzene was weighed and its melting point was determined. (Pure p-dichlorobenzene melts at 530C).

RESULTS & CALCULATIONS:

Initial weight of benzoic acid = 1. 0000g

Initial weight of p-dichlorobenzene = 1. 0002g

Weight of beaker = 106. 5879g

Weight of 2 filter papers = 1. 6153g

Weight of 2 filter papers + weight of benzoic acid recovered = 2. 5437g

Weight of beaker + weight of p-dichlorobenzene recovered = 106. 7491g

Weight of benzoic acid recovered = 0. 9284g

Weight of p-dichlorobenzene recovered = 0. 3392g

Melting point of benzoic acid recovered = 123ËšC

Melting point of p-dichlorobenzene recovered = 56ËšC

Percent Recovery of benzoic acid

= 0. 9284g / 1. 0000g x 100%

= 92. 84%

Percent Recovery of p-dichlorobenzene

= 0. 3392g / 1. 0002g x 100%

= 33. 91%

Relative Accuracy of Melting Point of benzoic acid
= 123ËšC / 122ËšC x 100%

= 100. 81%

Relative Accuracy of Melting Point of p-dichlorobenzene
= 56ËšC / 53ËšC x 100%

= 105. 66%

DISCUSSION:

A vital feature when choosing a solvent system for extraction is to pick out two immiscible solvents. Some common liquid/liquid extraction solvent pairs are water-dichloromethane, water-ether and water-hexane. In this experiment, to dissolve in ether (diethyl ether), (C2H5)2O the benzoic acid and p-dichlorobenzene were used. Most extraction includes water because it is highly polar and immiscible with most organic solvents. In addition, the compound that is to be extracted should be soluble in organic solvent but not soluble in water. The volatility of the solvent is very significant. This is because solvent have low boiling point like ether (very volatile) which can used to drying the isolated material very fast.

Both the benzoic acid and p-dichlorobenzene are able to dissolve in the ether solution according to the theory ‘ like dissolve in like’ which organic compounds are soluble in organic solvent. These solvent does not respond with the ether solution, but they just dissolve in it. Then, sodium hydroxide,

NaOH is added into the ether to react with benzoic acid to form sodium salt of benzoic acid and water.

Two layers of solution are formed which upper layer is organic solvent while the lower layer is aqueous layer. The sodium salt of benzoic acid (sodium benzoate) is dissolve in the aqueous layer which runs off into a conical flask. The second time of addition of NaOH into the solvent is used to make sure that all the benzoic acid is reacted completely with sodium hydroxide. This reaction caused the similar two layers are formed and the aqueous layer is transferred into the same conical flask by using the same technique. The reaction between benzoic acid and sodium hydroxide produces a lot of vapor after few times of shaking. Thus, the surplus pressure produced from the reaction is being released through the tap of the separatory funnel for several times to reduce the pressure inside it.

In the separatory funnel, water is added to wash the solvent. Later, two or three granular calcium chloride was added into the organic solvent after the aqueous layer was run off. The purpose of adding of calcium chloride is to get rid of residual water in the organic solvent. The calcium chloride in the granular form will be preferable. The calcium chloride is known as drying agent in the organic solvent which is not dissolved in the solvent but dries the solvent. The calcium chloride forms a cluster together with the water droplets as it solidified them. In another words, the calcium chloride reacts with water to form hydrates which is their preferred form when water is available. The chemical reaction between granular calcium chloride and water:

CaCl2 (s) + H2O (l) CaCl2. H2O (s)

Heavier hydrate sinks to the bottom of the funnel and it is easier to be removed when larger size of hydrate is formed in the solvent. An excess drying agent was used to ensure that all the water in solvent is removed. If the water remains in the materials collected, it could obstruct with the analysis.

The upper layer containing p-dichlorobenzene in ether is run off into a conical flask. Then, the conical flask is added with two or three boiling chips and is being heated on a hot plate to evaporate the ether. The boiling chips are small, insoluble, and porous stones made of calcium carbonate or silicon carbide. There are a lot of pores inside the boiling chips which provide cavities to trap air and to provide spaces respectively to enable bubbles of solvent to be form. It releases tiny bubbles which can prevent boiling over when boiling chips are heated. Over boiling of solvent will cause lost of solution which may lead to inexact result to be obtained. Boiling chips are never added to a hot solution because it will cause immediate boiling over of solution. If the ether in the conical flask is left 20ml, the solution is left aside in the fume hood. The ether is not continuously heated as the crystal of p-dichlorobenzene will melt and hence the crystal cannot be recovered. The white p-dichlorobenzene is formed in the crystal form after all the ether evaporates. The weight of recovered p-dichlorobenzene is about 0. 3392g with melting point of 560C. In addition, the percent recovery and relative accuracy of melting point for p-dichlorobenzene are 33. 91% and 105. 66%. The percent recovery of p-dichlorobenzene is very low which only has 33. 91% might be because to the lost of product in the experiment. Furthermore,

the relative accuracy of melting point is more than 100% because the product is not a pure p-dichlorobenzene due to some impurities exist in the product.

Next, the sodium benzoate in aqueous layer collected in the conical flask is added with hydrochloric acid, HCl. The white precipitate is formed from the reaction. The neutralization take place when sodium benzoate and HCl reacts with each other to produce precipitate of benzoic acid as the main product (sodium chloride salt and water are the side product).

The white precipitate is washed with cold water during filtration to minimize the solubility of benzoic acid in the water. The weight of benzoic acid recovered and its melting point are 0. 9284g and 1230C. The percent recovery of benzoic acid is 92. 84% while the relative accuracy of melting point of benzoic acid is 100. 81%. Similarly, the product was lost during the experiment was conducted. Most probably some of the products were dissolved in the cold distilled water during filtration. Then, the relative accuracy of melting point for benzoic acid is more than 100% due to the existence of impurities in the products.

CONCLUSION

In the acid-alkaline extraction, the benzoic acid and the p-dichlorobenzene is mixed by adding into ether. Then the NaOH is added to the mixture and the reaction occurs to separate the benzoic acid and p-dichlorobenzene. Thus, the extraction is done. From the experiment, we cannot obtain 100% of recovered from the extraction. This is due to the unavoidable mistakes done during the experiment. The recovery percentage for the benzoic acid is 92.

84% and for p-dichlorobenzene the recovery percentage is 33. 91%. The relative accuracy of melting point for benzoic acid and p-dichlorobenzene are 100. 81% and 105. 66%.