Observations

Table 1. Solubility and crystalline forms of the anthracene, benzoic acid, phthalic acid, resorcinol and sodium naphthionate in ligroin, toluene, ethanol, and water.

Organic Compound	Physical Characters	Solvents			
		Ligroin	**Toluene**	**Ethanol**	**Water**
Anthracene	Solubility in cold	Insoluble	Insoluble	Insoluble	Insoluble
	Solubility in hot	Insoluble	Soluble	Insoluble	Insoluble
	Crystal	None	Needles	None	None
Benzoic Acid	Solubility in cold	Insoluble	Soluble	Insoluble	Slightly soluble
	Solubility in hot	Insoluble	Slightly soluble	Insoluble	Soluble
	Crystal	Needles	None	None	Amorphous
Phthalic Acid	Solubility in cold	Insoluble	Insoluble	Insoluble	Slightly soluble
	Solubility in hot	Insoluble	Insoluble	Slightly soluble	Slightly soluble
	Crystal	None	None	None	None
Resorcinol	Solubility in cold	Insoluble	Soluble	Insoluble	Slightly soluble
	Solubility in hot	Insoluble	Insoluble	Insoluble	Soluble
	Crystal	None	None	None	None
Sodium Naphthionate	Solubility in cold	Insoluble	Insoluble	Insoluble	Slightly soluble
	Solubility in hot	Insoluble	Soluble	Insoluble	Soluble
	Crystal	None	Granular	None	None

Table 2. Experimental IR peaks compared to the literature IR peaks for salicylic acid.

Functional Group	Experimental peak(cm^{-1})	Literature peak (cm^{-1})
C=O (carboxylic acid)	1659	1675
O-H (alcohol)	3700-3300	3700-3400
O-H (carboxylic acid)	3400-2250	3400-2500
C=C (aromatic)	1612.6 and 1483.9	1600 and 1475
C-H	3200-2750	3200-2800

Theoretical yield of salicylic acid
1g Salicylic acid + Alizarin Yellow R (0.2%)
1.00g x 0.002 = 0.002g Alizarin Yellow R
Thus, the purified sample must be: 1.00-0.002 = 0.998g of pure salicylic acid

Actual yield of salicylic acid
Sample used in IR spectrum: 0.002g
Pure salicylic acid collected: 0.1281g

$$Percent\ Recovery = \frac{mass\ of\ recovered\ material\ (g)(actual)}{mass\ of\ starting\ material\ (g)\ (theoretical)} \times 100$$

$$Percent\ Recovery = \frac{0.1281g}{0.998g} \times 100$$

$$Percent\ Recovery = \ 12.8\%$$

Discussion:

The experimental peak values of the functional groups of salicylic acid are very close to the literature peak values. For the C=O functional group, the experimental peak value was only 16cm^{-1} less than the literature peak. The literature range value for the O-H (carboxylic acid) functional group was 3400-2500cm^{-1} and 3400-2250cm^{-1} for the experimental value, which is also very similar. The literature range value for the O-H (alcohol) functional group was 3700-3300cm^{-1} and 3700-3400 cm^{-1} for the experimental value, which is also very similar when compared. The C=C (aromatic) literature peaks were located at 1600/1475 cm^{-1} and 1612.6/1483.9 cm^{-1} for the experimental peak. The C-H functional group experimental value ranged between 3200-2750 cm^{-1} and for the literature value it ranged between 3200-2800 cm^{-1}. Thus, due to the fact that the experimental peak values were very similar to the literature peak value of salicylic acid, it is evident that the sample prepared is pure. The fingerprint region of the IR spectrum represents the bending vibrations within the salicylic acid. It is an important region because every compound generates its own unique pattern of peaks. When comparing two IR spectrums of compounds, if all peaks in the fingerprint region match, the compound is expected to be identical in structure. The fingerprint region of the

experimental IR spectrum does match the literature fingerprint region, thus, further supporting the idea that the sample is identical and pure.

The process of recrystallization involves dissolution of the solid in an appropriate solvent at a high temperature and the formation of the crystals when cooled, so that any impurities remain in solution. (Tipson, 1950) Thus, a “good” recrystallization solvent is one in which the solid is soluble at high temperatures and insoluble at low temperatures. It will dissolve a large amount of the impure compound at temperatures near the boiling point of the solvent. Small amount of compound being purified should remain in solution at low temperatures. Low solubility at low temperatures minimizes the amount of purified compound that will be lost during recrystallization. Furthermore, it is often stated that "like dissolves like" meaning that compounds having similar structural features will be soluble in one another. (Carleton University, 2015) Thus, in the laboratory, it is predicted that the non-polar compounds will dissolve in non-polar solvents and polar solutes will dissolve in polar compounds.

Water is a polar molecule, thus it will dissolve compounds that are polar as well. When the solutes were added, resorcinol, sodium naphthionate and benzoic acid were soluble in hot and slightly soluble in cold. Phthalic acid was slightly soluble in hot and cold. All of these compounds are very polar (contain –OH bonds), thus, it was expected that they would be soluble with water due to the fact that “like dissolves like”. Furthermore, anthracene was insoluble in water at both temperatures. This is because anthracene has a very weak polarity and does not dissolve well with polar molecules. Additionally, crystals were formed between water and benzoic acid. Thus, benzoic acid can be purified by recrystallization from water because of its high solubility in hot water and poor solubility in cold water.

Toluene is a molecule with a very weak polarity. Anthracene, a non-polar compound, was slightly soluble in hot and insoluble in cold baths. It also produced needle-like crystals. This result was expected due to the fact that each of them are non-polar, thus, they are able to dissolve with one another. Furthermore, because the solution

was soluble in hot and not in cold, the compound was able to be purified by recrystallization from toluene. Sodium naphthionate was also soluble in the hot and insoluble in the cold, thus, granular-like crystals formed. Benzoic acid was soluble in cold, but it was not soluble in hot, thus, no crystals formed. Phthalic acid was insoluble in both hot and cold temperatures and no crystal was produced. Resorcinol was soluble in cold but not hot temperatures, therefore, no crystal was produced.

Ligroin is a non-polar solvent. None of the solutes were soluble in the solvent at both hot and cold temperatures. Nevertheless, needle-like crystals formed when benzoic acid was cooled with ligroin. Thus, although it was recorded that benzoic acid was insoluble at a hot temperature in ligroin; the results may not be accurate. The experimenter may not have left the solution in the hot bath long enough to observe the dissolution.

Ethanol is a primary alcohol, thus it is polar and should dissolve any compound with a compound that is polar. However, all of the compounds were insoluble in the hot and cold solution and no crystals were formed. This may be due to errors performed in the laboratory. The solvent may have also been heated past its melting point, thus, melting the solid rather than dissolving it.

Due to the fact that the results achieved were not as expected, it is suggested that a mixture of two solvents, a solvent pair, should be used when the experiment is repeated. Sometimes, a single suitable solvent for recrystallization cannot be found. A useful solvent pair consists of two miscible solvents having different solvent powers. The first technique involves a “good” solvent that has a lower boiling point than the “poor” solvent. An example of this could be ethanol (B.P 78^{o}C) and water (B.P. 100^{o}C). The second technique can be utilized if the “poor” solvent is less volatile than the “good” solvent. (Carleton University, 2015)

After eliminating Alzarin Yellow R, which is 0.2% of the crude sample, 0.998g of the pure Salicylic acid was expected to be left behind. However, as a result, only 0.1281g

was recovered which yielded a low percent recovery of 12.2%. This could be due to multiple sources of error present in the laboratory. The recovery of recrystallization is always expected to be less than 100% due to the fact that, even at very low temperatures, the desired compound has some finite solubility in the recrystallization solvent and is thus lost when solvent and soluble impurities are removed. Thus, any unnecessary prolonged contact with recrystallization solvent, especially if the solvent is not ice-cold will result in loss of product. If too much solvent is used, the desired product will stay dissolved in solution and the percent recovery will be lower than it should be. Furthermore, to obtain optimal results, a minimum of near-boiling solvent should be used for the recrystallization, and a minimum of ice-cold solvent should be used for the rinse. Occasionally crystals will not form, even though the saturation point has been reached. Failure of crystallization to occur after the solution has cooled somewhat could be due to too much solvent has been used or that the solution is supersaturated. Some loss of sample may have resulted from transferring solids from 3 one container to another. For example, in the second filtration step, material was lost on filter paper. In the hot filtration step, material may have crystallized in filter paper.

Conclusion

The purpose of this experiment was to examine the solubility of the five different organic compounds in various solvents. It was predicted that a "good" recrystallization solvent is one in which the solid has a very high solubility at high temperatures and a very low solubility at low temperatures. This was supported when anthracene and toluene, water and benzoic acid, and toluene and sodium naphthionate were mixed together and formed crystals. Furthermore, by recrystallization and the use of decolorizing charcoal, salicylic acid would be purified. The percent recovery after the salicylic acid came to a value of 12.8%. The experimental peak values of the C=O, O-H (alcohol), O-H (carboxylic acid), C=C, and C-H functional groups were 1659cm^{-1}, 3400-2250cm^{-1}, 3700-3300cm^{-1}, 1612.6/1483.9cm^{-1} and 3200-2750 cm^{-1} respectively. These values were very similar to the literature peak value, thus supporting the idea that the sample is pure.

References

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