Experiment #1:Solubility Tests and Recrystallization.

Results:

Table 1. Solubilities of anthracene, benzoic acid, phthalic acid, resorcinol and sodium naphthionate in four solvents; ethanol, n-hexane(ligroin), toluene and distilled water. The solubilities of each solute in each solvent were also tested at low (room) temperature as well as high(boiling) temperature.

	Solutes →				
Solvents ↓	Anthracene	Benzoic acid	Phthalic acid	Resorcinol	Sodium Napthionate
Ethanol	Insoluble at low and high temps.	Insoluble at low and high temp.	Insoluble at low temp. Very small amount dissolved at high temp.	Insoluble at low and high temp.	Insoluble at low and high temp.
Ligroin	Insoluble at low temp. Partially soluble at high temp.	Insoluble at both low and high temp.	Insoluble at low temp. Partially insoluble at high temp.	Insoluble at low temp. Partially soluble at high temp.	Insoluble at low temp. Partially soluble at high temp.
Toluene	Insoluble at low temp. Partially soluble at high temp.	Insoluble at low temp. Fully soluble at high temp.	Insoluble at low temp. Partially soluble at high temp.	Insoluble at low temp. Partially soluble at high temp.	Insoluble at low and high temp.
Water	Insoluble at low temp. Partially soluble at high temp.	Insoluble at low temp. Partially soluble at high temp.	Insoluble at low temp. Partially soluble at high temp.	Fully soluble at low temp.	Insoluble at low temp. Partially soluble at high temp.

Note: Temp. is shorthand for temperature.

Table 2. Visual observations of solutes in each solvent at low and high temperatures.

Solutes ↓	Ethanol	Ligroin	Toluene	Water
Anthracene (small, white, fine crystals)	Unchanged at low and high temp.	White precipitate at high temp.	White precipitate at high temp.	White precipitate at high temp.
Benzoic acid (opaque, white medium sized crystals.)	Unchanged at low and high temp.	Mostly unchanged. Formed white clumps in solution.	Fully dissolved at high temp.	Unchanged at low and high temp.
Phthalic acid (dense,white powder)	Unchanged at low and high temp.	Unchanged at low and high temp.	White powder remained after being partially dissolved at high temp.	White powder remained after being partially dissolved at high temp.
Resorcinol (Large white crystals.)	Unchanged at low and high temp.	Crystals remained after partial dissolution at high temp.	Unchanged at low and high temp.	Fully soluble at low temp. No precipitate.
Sodium Napthionate (large crystals with purple colour)	Unchanged at low temp. but crystals remained after partial dissolution at high temp.	Crystals remained after partial dissolution at high temp.	Unchanged at low and high temp.	Crystals remained after partial dissolution at high temp.

Table 3. Experimental and literature IR peaks for salicylic acid sample in KBr pellet.

Functional group	Experimental IR Peaks(cm-1)	Literature IR Peaks(cm-1)
OH(phenol)	3600-3100	3600-3300
OH(carboxylic acid)	3270-2450	3300-2550
C-H	3170-2800	3150-2990
C--O(carboxylic acid)	1578.7	1670
C--O(aromatic)	1484, 1444.2	1610, 1490

Note: (-) Hyphen represents a single bond between the 2 atoms. (--) represents a double bond between the 2 atoms.

Calculations:

Percent recovery = (mass of recovered material (actual) / mass of starting material (theoretical)) (100)

Actual mass = 0.4144 g
Starting mass = 1.01 g

Percent recovery = (0.4144 g / 1.01 g) (100)
Percent recovery = 41.029 %
Percent recovery = 41.0 % 3 sf.

Discussion.

In table 1 it is seen that the solubilities of each solute in each respective solvent vary widely. It can also be noted that temperature changes also had varying degrees of effectiveness in causing a solute to dissolve.

First, the difference in solubility of the solutes in solvents are due to the independent polarity of each compound being used in the experiment. It is rudimentary knowledge that substance of similar polarity are able to dissolve into each other. This is referenced often by the phrase “like dissolves like.”. This trend can be seen in an extent in the results obtained in table 1.

For example, anthracene is a fully non polar, organic compound. It is composed of 3 phenyl rings and has no polar bonds. As such it is expected to dissolve in nonpolar solvents and be insoluble in polar solvents. This is seen in table 1, as it is insoluble in ethanol but partially soluble in ligroin, toluene and water when raised to high temperatures. Its solubility in ligroin and toluene was expected, as they were both completely nonpolar solvents. It also experienced small amounts of dissolution when heated in water. Its partial solubility in hot water could potentially be for two reasons. The first is that the heated environment provided enough energy for the electronegative oxygen atoms to break the bonds found in anthracene’s phenyl group, or it the water was contaminated with one of the nonpolar solvents. As anthracene and water are the farthest apart among the compounds tested in terms of polarity, it is an unexpected result to have anthracene dissolve in water.

Furthermore, molecules that are amphipathic(have both polar and nonpolar groups), are able to experience dissolution in solvents of varying polarity. This is seen in table 1 where phthalic acid and benzoic acid are able to partially dissolve in both toluene and water at high temperatures. This is expected, and is the result of each solute having both polar and nonpolar components in their structure, which allows them to be at least partially soluble in both polar and nonpolar solvents.

It is also seen that in table 1, raising the temperature of a solution usually had a significant impact on the solubility of the solute compound. This is because by raising the temperature of the system(the system being the test tube containing both solvent and solute), the amount of energy present within the system increased proportionally as well. And by doing this, the bonds of the solute were able to absorb energy, thus becoming more unstable, and making it easier for the solvent to break their intramolecular bonds. This lead to the previously insoluble solute to either partially, or fully dissolving in the solvent at high temperatures.

This can also be related to the concept of solvent power[1]. Which states that a solvent's ability to break apart a solute to dissolve it, is impacted by the boiling point of said solvent. The effect of this factor can be seen in table 1, and the differing solvent abilities of water and ethanol. Water is more polar than ethanol, and also has a higher boiling point. As such it has a greater solvent power, with the result of said power being observed in table. For example, in the dissolution of phthalic acid, water was able to partially dissolve the solute at high temperatures, whereas ethanol was completely not able to.

When comparing the IR values of the experimental process to the literature values found on the SDBS in table 3, it is apparent that there are a number of differences between the two sets of values. Although they are not all greatly different in magnitude, the frequency of there being significant difference between the sets of values allows one to come to the conclusion that the experimental salicylic acid sample was not completely purified, and still contained impurities.

OH(phenol) - Both the experimental and literature values started at the same point of 3600, but the literature value extended far past the lower limit of the experimental of 3100, and instead stopped at 2300.

OH(carboxylic acid) - Both the experimental and literature values were very similar in terms of range on the spectrum. Lit. range extended from 3300-2550, and the exp. Range extended from 3270 - 2450.

CH - Was overall quite similar in range between the two spectrums, although it was significantly shorter in the lit. spectrum. Exp. range extended from 3170 - 2800, and the lit. range extended from 3150 - 2990.

C--O(carboxylic acid) - Varied by significant amounts. Exp. peak was found at 1578.8, and lit. peak was found at 1670.

C--O(aromatic) The 2 exp. Peaks varied quite significantly from the lit. values. It is noted that the second peak of the lit. values was very close in value to the first peak in the exp. values. Exp. peaks were found at 1484 and 1444.2, and lit. values were found at 1610 and 1490.

It is also important to note that due to the nature of the IR spectrum process performed, the salicylic acid spectrum wasn't affected by anything other than its own impurities, as the IR spectrum was observed using a KBr pellet. KBr does not have a spectrum and thus does not affect the spectrum of the substance being observed.

In relation to the “fingerprint” portion of the spectrum, which extends form 1600 cm-1 to 600 cm-1, were very similar between the experimental and literature spectrums.

Lastly, to restate a previous conclusion, due to the number of differences between the experimental and literature spectrums it is clear that the salicylic acid sample still contained a significant number of impurities. Although it is still worthwhile to note that there are still a number of close and similar values between the experimental and literature ranges and peaks.

The experimental process was split into two distinct parts. The first pertained to the solubility testing of a varied set of solutes in an equally varied set of solvents. Each solute had its solubility tested in each solvent in a trial format at room temperature before being placed in a steam bath.

The second portion of the experiment pertained to the purification and recrystallization of salicylic acid. The experimental process utilized the knowledge of the differing solubilities of the impurities found in the salicylic acid, and designed the steps in a format to remove both insoluble and soluble impurities. The more pure the salicylic sample, the more similar its experimental IR spectrum is to the accepted literature IR spectrum.

The notable key reagent in this experiment is the activated charcoal. It was used during the recrystallization portion of the experiment and acted to purify the liquid salicylic acid by binding to soluble impurities and precipitating out of solution, leaving behind a purified salicylic sample. This allowed the purification process to be much less intensive and relatively simple.

Conclusion:

The purpose of this experiment was to examine the solubilities of different compounds in solvents of differing polarity and structure. And to purify salicylic acid through a recrystallization process.

Table 1. displays the solubilities for the all the solutes in each solvent at both low and high temperatures.
Table 2. Describes the physical characteristics of each organic substance in each solvent at low and high temperatures.
Table 3. Displayed the experimental and literature values for the IR spectrum of salicylic acid. Compared the experimental values of peaks and ranges of the funcitonal groups found in salicylic acid with the literature values found in the IR spectrum of salicylic acid.

The percent recovery of salicylic acid was found to be 41.0 %

The purpose of the experiment was fulfilled.

Experimental error: In the solubility testing portion of the experiment it is seen that some organic substances that should be insoluble in a solvent actually experienced small amounts of dissolution.An example of this namely being the dissolution of anthracene in water. As it was explained in the discussion section of this report, anthracene should be completely insoluble in water, however partial solubility was observed. This would most likely be due to the contamination of apparatus used to scoop each solute into its respective test tube. Seeing as how there were many groups using the same set of compounds, some individuals may have used the incorrect scoops to place the solutes in each tube. This leads to a scoop potentially having multiple different solute samples on it, which would then lead to confusing results as seen here. And while it was advised to wipe off each scoop with a material provided, it would be more effective to fully clean and wash each scoop after each use. This change would lead to a slightly longer experimental process, but ultimately severely decrease the probability of contamination occurring.

In the salicylic portion of the experiment, it was observed that the salicylic sample synthesized still contained a significant number of impurities, as well as having a low yield. The errors leading to these results are different. The first, being that the experimental design did not specify a structured method of determining how much activated charcoal should be added solution. This is very important as the activated charcoal was needed to remove the soluble impurities found in the solution. It failed to account for the amount of water being added, and did not describe if or how the mass of charcoal would need to be increased if a relatively large amount of water was added to the solution. This is a crucial factor as the water can serve to dilute the activated charcoal and decrease its ability, and as a result its effectiveness, to bind to the impurities in the solution. An improvement to the experimental design would be to develop a set of values that measure the amount of water and the corresponding amount of charcoal that should be used to ensure maximum purification of the salicylic acid solution.

To increase percent recovery, two changes can be made to increase recovery. The first would be the use of apparatus that allows more efficient removal of sample would prove beneficial. As this would minimize the amount of sample that is inevitably left behind as it changes containers multiple times. And secondly, limiting the number of times the sample solution and solid sample, are transferred. As this would minimize the number of times that a small amount of sample could be left behind in the previous container.

References:

1. CHEM 2203/2204 Laboratory Manual. 2017-2018.C.A.White and G.W. Buchanan. Department of Chemistry.