Discussion:

In this experiment, we practiced the concepts of oxidation and reduction reactions. We split up the two reactions so that we could accomplish the reactions more efficiently. I was primarily responsible for the oxidation reactions, though we made sure to check in with the other reactions. For the oxidation reaction, we attempted to collect 50 mg of 9-flurenol, which was a very light, white, sticky substance. As it was very hard to get a precise amount, we collected around 0.048 g of it, which was directly kept in a 10 mL RBF. Using the conversion of 20 drops = 1 mL, we calculated that we needed 3 drops (0.12 mL) of glacial acetic acid, along with 8 drops (0.4 mL) of NaClO (initially, also this is most known as commercial bleach). After mixing the solution for 5 minutes, we took a TLC plate analysis which revealed that the reaction had not fully taken place. After repeatedly adding 8 drops of NaClO (4 times, so a total of 32 drops), we were able to obtain an almost fully reacted solution as indicated by our TLC plates. This solution was kept in a separatory funnel along with 2 mL of hexane that was initially poured into the 10 mL RBF to get out as much of the solution as possible. After shaking the funnel and extracting the organic layer (bottom), 2 more mL was kept in the extraction funnel. The organic layer was extracted once more, isolating the hexane layer. This hexane layer was put in another flask, of which a little more than 2 mL of water was added alongside 2.823 g in total of our drying agent (NaCO3 in the lab manual, but we used MgSO4). At this point, we had gotten a little confused between the two reactions and accidentally vacuum filtrated this dried solution. What was left in the Buchner funnel was the solid MgSO4 while our vacuum chamber contained the aqueous solution we wanted, but only for a few seconds. The boiling point of hexane is 68(^0)C, but under the pressure made in the vacuum chamber, the temperature did not need to be that low. This resulted in a solid crystal remaining at the bottom of our vacuum chamber. The vacuum chamber was rinsed with an excess amount of hexane and then put in an 100 mL RBF in an attempt to proceed by normal methods. This solution was then rotary evaporated, chilled, and the resulting product used for IR. The product might have not been as dry as we had hoped for as the percent yield was above 100. We do know, however, that our reaction produced the correct product as shown by our IR spectra, which also indicated that some characteristics of excess hexane.

The reduction reaction was much quicker and went much smoother. As instructed, we collected a fifth of designated chemicals given by the lab manual. In turn, about 1 g of 9-flurenone and 6 mL of ethanol were added to a 100 mL beaker. The hardest part of this reaction was making sure that the dropwise addition of the reducing agent fully reacted and was never too much. The reducing agent was composed of .04 g CH3NaO, 0.1 g NaBH4, and 2 mL of CH3OH. Following the full addition of the reducing agent, the reaction mixture was let to sit for 20 minutes. During this reaction, it was noticed that some yellow crystals were visible. However, these disappeared during the last 5 minutes of the reaction, creating a clearer solution, followed by a milky white solution. Preceding this step, a little more than 50 mL of water was added to the solution. The solution was then neutralized (by the addition of excess HCl to acquire a slightly acidic solution), vacuum filtrated, and washed with cold water. This resulted in a higher yield than expected, though we did not fully understand why. The product produced was a sticky, white, large but light mass, much like what was used for our oxidation reaction. Our IR analysis also indicated the expected product was formed. However, the broad peaks also indicated that the product was not pure. This was most likely due to residual inorganic salt which may have been created though the reaction of residual NaBH4 and HCl.

Conclusion:

In conclusion, the oxidation and reduction of 9-flurenone and 9-flurenol was very much attainable. For the oxidation reaction, the rate of the reaction was greatly increased through the addition of more oxidizing reagent. To begin the oxidation reaction, .048 g of 9-flurenol was obtained along with acetone and our oxidizing agent (commercial bleach). Once reacted completely, the product was extracted and used for IR. The IR spectrum indicated our product was not pure, but was the expected product, as indicated by the sharp peaks in between 1500 – 1600 cm-1 (benzene ring stretches) along with a sharp (unmarked) dip in between 1500 – 1700 cm-1 (carbonyl group stretch). The reduction of 9-flurenone was relatively shorter than the oxidation. A sample of the reactant was taken alongside some warm ethanol in a 100 mL beaker. The reducing agent was then gradually added (dropwise), and the reaction mixture was left alone. Change in the reaction mixture was visible as the color and consistency changed. Following the presumed full reaction of the solution, the product was precipitated out and then used for an IR analysis. The IR spectrum showed that the expected product was produced, as indicated by the minor (unmarked) stretches seen between the 1500-1700 cm-1 region (benzene ring stretches) alongside the broad peaks in the range of 3000-3500 cm-1 (hydroxyl stretches). This lab was a success as both the expected products, 9-flurenol and 9-flurenone for reduction and oxidation (respectively) were produced, though they did seem to be quite impure as shown by the IR analysis and our quite large percent yields.