Experiment 10 “Synthesis and Observation of Liquid Crystals”

The purpose of this experiment was to synthesize cholesteryl benzoate by reacting cholesterol with benzoyl chloride and attempt to quantify some of its properties. Cholesteryl benzoate is a liquid crystal so at certain temperatures it appears to take a state of matter between solid and liquid. The molecule also has unique appearance when observed under a polarized microscope.

The amount of cholesteryl benzoate obtained was about 0.380g or 7.74e-4 mol. Since 0.974g or 2.52e-3 mol of cholesterol was used, the %yield is calculated to be 30.7%. The melting point in the literature for both cholesteryl benzoate and cholesterol was stated to be about 148°C (*Cholesterol; Cholesteryl benzoate*). The experiment yielded multiple melting points. At about 98.5-104°C the solid crystals were observed to transition into the liquid crystal phase and then at 143°C the liquid crystals “melted” again into the full liquid stage. Including reasonable room for human error, the final product can be assumed to be fairly pure because its final melting point from liquid crystal to liquid is close to the melting point provided by the literature.

The IR spectra of cholesterol has a clear broad peak that represents the OH group at 3427cm-1 and a group of peaks from 2848 to 2930cm-1 which represent the C-H bonds. The IR spectra of the product has expected peaks for the C-H bonds from 2847 to 2926cm-1 which is similar to the cholesterol but it also has a low, broad peak at about 3406cm-1. This peak is similar to that of an OH group or N-H bond, neither of which are part of the expected product. This peak may have been caused by the pyridine somehow reacting to produce an N-H bond. It is more likely that the peak is due to the OH group on unreacted cholesterol that remained in the product. It is also possible that some ethanol that was used to clean the instrument mixed with the product and provided a strong enough signal to produce the unexpected peak. Even though there is an extra peak in the IR spectra, it is particularly small and since the melting point was close to the expected temperature, the peak can probably be ignored. As a result, the product is relatively pure but not completely pure.

The observations from the melting point apparatus are outlined in the following table.

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| Temperature (°C) | Observation |
| 143 | Color changes from clear to cloudy white (beginning of liquid crystal phase) |
| 131 | Appears to be royal blue in color (full liquid crystal phase) |
| 123 | Color fades back to cloudy white |
| 104 | Solid starts to form |
| 98.5 | Color changes to white (full crystal phase)  The crystal started forming up the sides of the capillary |

Using the microscope without the polarized lens, the liquid appeared to be clear with many black lines without any order to them (these may have been scratches on the slide itself). After adding the polarized filter, the product was still clear but the black scratches disappeared. The sample still appeared to be a clear liquid but slowly started to transition to have a crystalline like appearance that was extremely colorful. Different areas of the sample had different color schemes, one area was mostly pink/red, another had different shades of blue etc.

References

“Cholesterol.” *National Center for Biotechnology Information. PubChem Compound Database*, U.S. National Library of Medicine, https://pubchem.ncbi.nlm.nih.gov/compound/Cholesterol.

“Cholesteryl Benzoate.” *Cholesteryl Benzoate CAS#: 604-32-0*, https://www.chemicalbook.com/ProductChemicalPropertiesCB6751799\_EN.htm.