

10 %  $\text{Na}_2\text{S}_2\text{O}_3$  was added to remove the excess bleach and  $\text{I}_2$ . This was done until the brown color had mostly dissipated. 3M  $\text{HCl}$  was added to the solution until it had pH of 4. The round bottom flask was then placed in an ice bath to initiate crystallization and the solid formed was collected using vacuum filtration. The mass, melting point, and  $^1\text{H}$  NMR was obtained for analysis.

### Results and Discussion:

(see attached NMR data)

*This data must be included in the Exp.*

The initial mass of vanillin was 1.98 g. The recovered amount of product was about 0.355 g, thus the percent yield was 17.9%. The low percent yield can be attributed to product lost during both vacuum filtrations or in the transferring of solution between glass ware. In addition to these experimental and calculated values, the melting point range of the compound was obtained. Through use of the melting point apparatus, the range for the product was found to be 147-151 degrees Celsius. The known literature value is relatively higher than the value we obtained. The range in the melting point value occurs because of impurities present. The melting point is a physical property that can be used to identify a compound by comparing an experimentally obtained value to a known literature value. The value obtained from the product of this experiment is relatively consistent with the known literature value, so it can be determined through this that the proper product was created from this experiment.

$^1\text{H}$  NMR (Nuclear Magnetic Resonance) data was also used to analyze the product of this experiment. The first peak found at approximately 10.8 ppm with an integration of about 1 represents the proton in the alcohol group attached to the aromatic ring. This was also determined to be the alcohol peak because of its small rounded shape, however, in our NMR data, it is relatively small size for an alcohol peak. The next singlet peak at approximately 9.8 ppm with an integration of 1 is representative of the proton attached to double-bonded oxygen (the acetyl group). It is located at the correct ppm for this particular structure. The next singlet at about 7.9 ppm with an integration of 1 represents the proton attached to a carbon next to the iodide on the aromatic ring. The proton associated with this peak undergoes a shift due to its close proximity to the iodide, thus it has a larger ppm value. The next singlet peak with an integration of 1 found at about 7.5 ppm represents the proton attached to a carbon between the acetyl group and the oxygen attached to a methyl. This peak also undergoes a shift due to its location between two oxygens. The last peak in the NMR data is a singlet at approximately 3.8 ppm represents the three protons attached to a carbon attached to an oxygen. The oxygen accounts for the shift that occurs for this peak. All peaks are singlets because none of the proton groups have adjacent groups with which to interact. The subsequent peak at about 3 ppm is water and the peak at about 2.5 is the DMSO. The NMR data further confirms the structure of the product.

The accuracy of the experiment could be improved with a better percent yield. With a better percent yield, the concentration of the product would be more accurate. The

*Aldehyde group.*

*phenolic group.*

*methoxy group*