Discussion:

We used TPCP along with styrene to perform this Diels-Alder reaction. The TPCP acted as our 4 carbon diene because it contains the necessary s-cis conformation. Alongside this, we used styrene as our dienophile, which contributes 2 carbons. The combination of these two (under heat) should have caused a reaction producing a 6 member ring. This has the possibility of resulting in either an endo (faster) or exo (more stable) conformation. To differentiate what we got when the reaction was complete, we had to do a melting point analysis and an NMR scan. Before that, however, there were some complications with the reaction itself that may have skewed the results. At the beginning of the procedure, when collecting the TPCP and the styrene into a test tube, we did not get all the TPCP (powder) off the sides of the container using the styrene (aqueous), therefore resulting in a lesser reacting yield. Beyond this, there were not many complications. We then monitored the reaction by taking TLC plate analysis 30 minutes apart. The chemicals used on the TLC plate included normal TPCP (dissolved in ethyl acetate), the reaction solution (also dissolved in ethyl acetate), and a mixture of the two. The TLC plate analysis took some time as we did it incorrectly twice before getting it correct. This was due to the fast evaporation of the mobile phase (5% ethyl acetate + 95% pentane) which we did not account for. After taking two TLC plate analyses, we stopped the reaction, cooled the test tube, and then tried to isolate our desired product. This was done through multiple washes of the ethanol (more than 5 mL was used) into a vacuum filtration contraption. A fine tan (very light purple, not too white, but just barely purple) powder was collected. This product was then taken for melting point analysis and NMR analysis. The impurity of this product can be seen by the broad range of the melting point. Upon analysis of the NMR, it is clear that there are many aromatic rings due to the multiplet that appeared in the 6 – 8 ppm region. This does not provide us with any information as both the endo and exo configurations have multiple aromatic rings. Finally, when analyzing the X-ray crystallography provided by the CCDC, we can tell that the major product formed would be the endo version of the molecule (this can be told by the recognition that the carbonyl is pointing in the opposite direction of the phenol on the 5th carbon). The bond angle for most carbons attached to the outer phenol groups are about the same, except on the 2nd and 3rd carbons (the ones with the double bond), which have an angle of about 123 degrees, which is larger than the other dihedral angles of about 117 degrees. The dihedral angle of the two hydrogens attached to carbons 5 and 6 is 12.7 degrees.

Conclusion:

In conclusion, our Diels-Alder reaction using TPCP as our diene and styrene as our dienophile resulted in the formation of the endo conformation of 1,2,3,4,5-Pentaphenylbicyclohept-2-en-7-one. We did not result in a very pure product as the melting point range was broad and was not in the range of expected values. This, however, was expected as the best way to really analyze the product was through an X-ray crystallography. The crystallography showed us that the dihedral angles of the molecules were: alpha = 118.181 degrees, beta = 94.775 degrees, and gamma = 99.292 degrees. The Karplus equation gives us a mathematical relation between a J value and the dihedral angle between two neighboring hydrogens. By the Karplus correlation, we can see that there would be different J values all correlated with what we can expect from the dihedral angles, proving that this product was in fact what we achieved to extract.