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NMR Kinetics of the Diels-Alder Reactions of 9-Substituted Anthracenes: Development of an Organic Chemistry Lab Experience

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Abstract / Introduction

NMR spectroscopy plays an integral role in the teaching of organic chemistry. With the integration of conceptual understanding and problem solving, deciphering spectrums of an unknown chemical structure can provide many necessary challenges¹.

The Diels-Alder reaction ($[4\pi + 2\pi]$ cycloaddition reaction) is one of the most important reactions in synthetic organic chemistry as it allows for the formation of two C-C bonds and displays good regio- and stereoselectivity². The kinetics of the reaction between maleic anhydride and anthracene and two of its derivatives (9-methylantracene & 9-anthracenecarboxaldehyde) were the main focus of this experiment.

Figure 1: Reaction between maleic anhydride and anthracene.

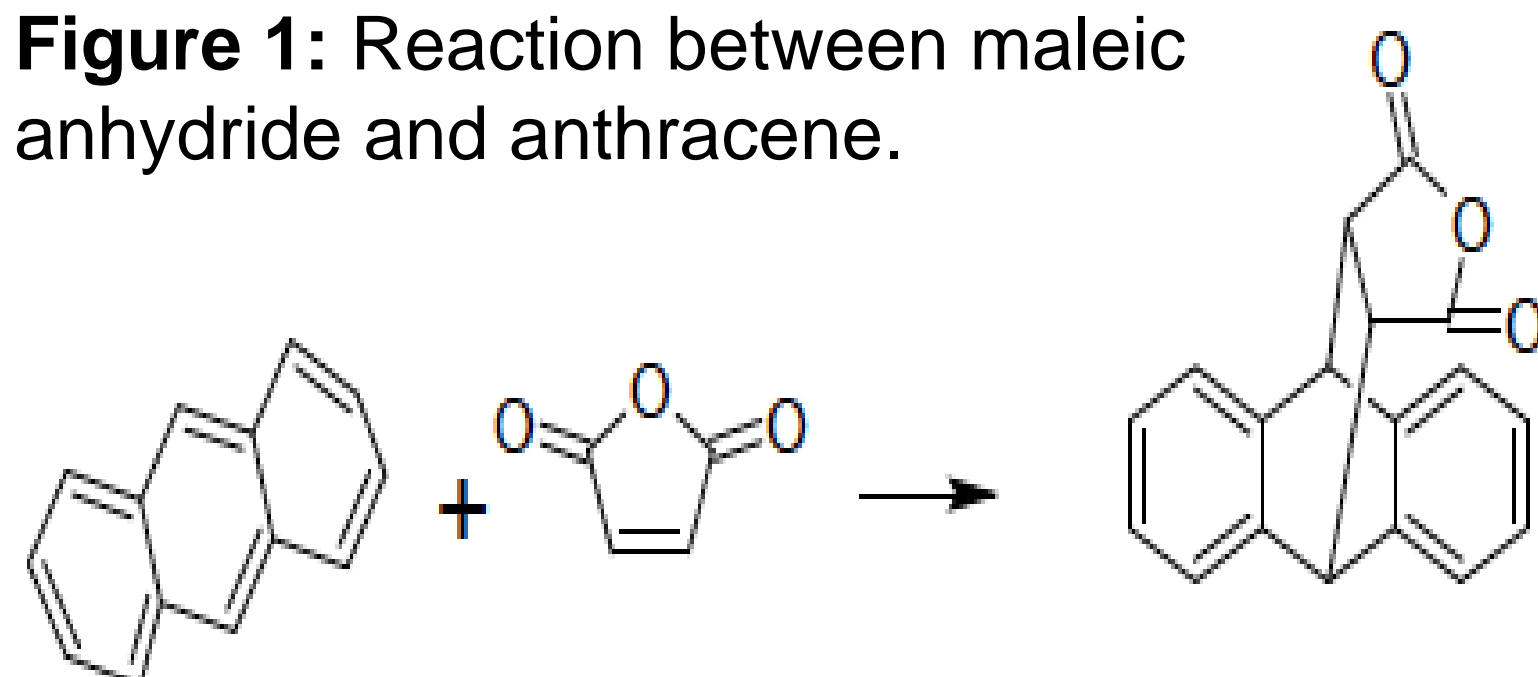


Figure 2: Reaction between maleic anhydride and 9-methylantracene

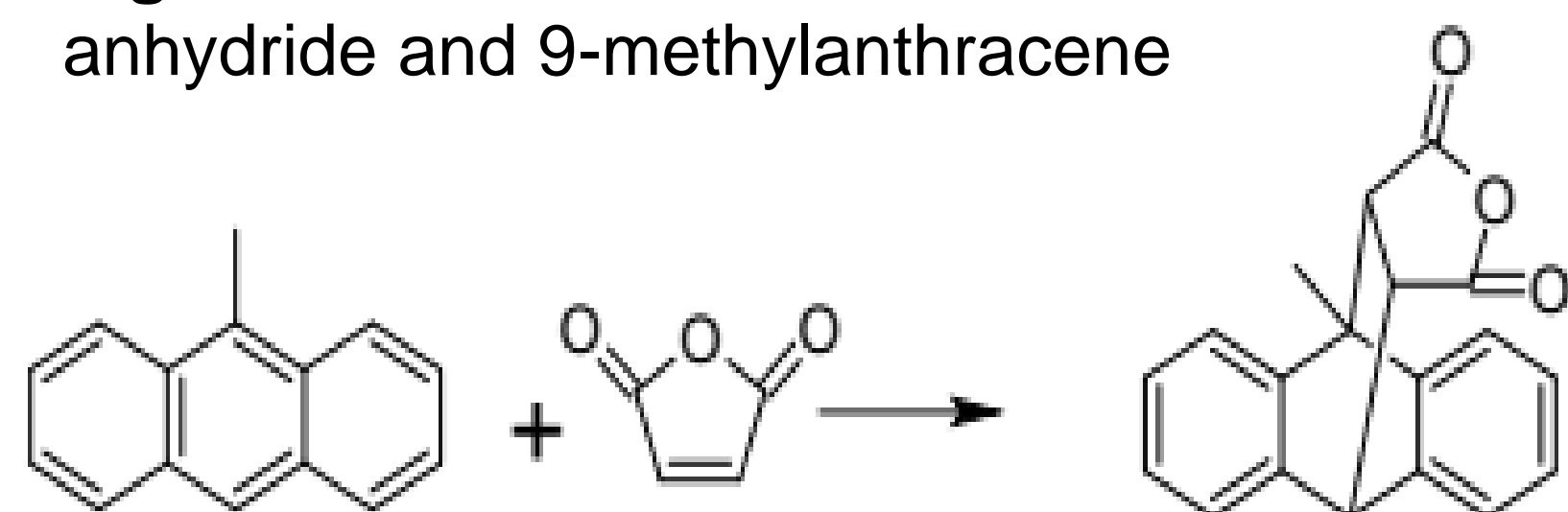
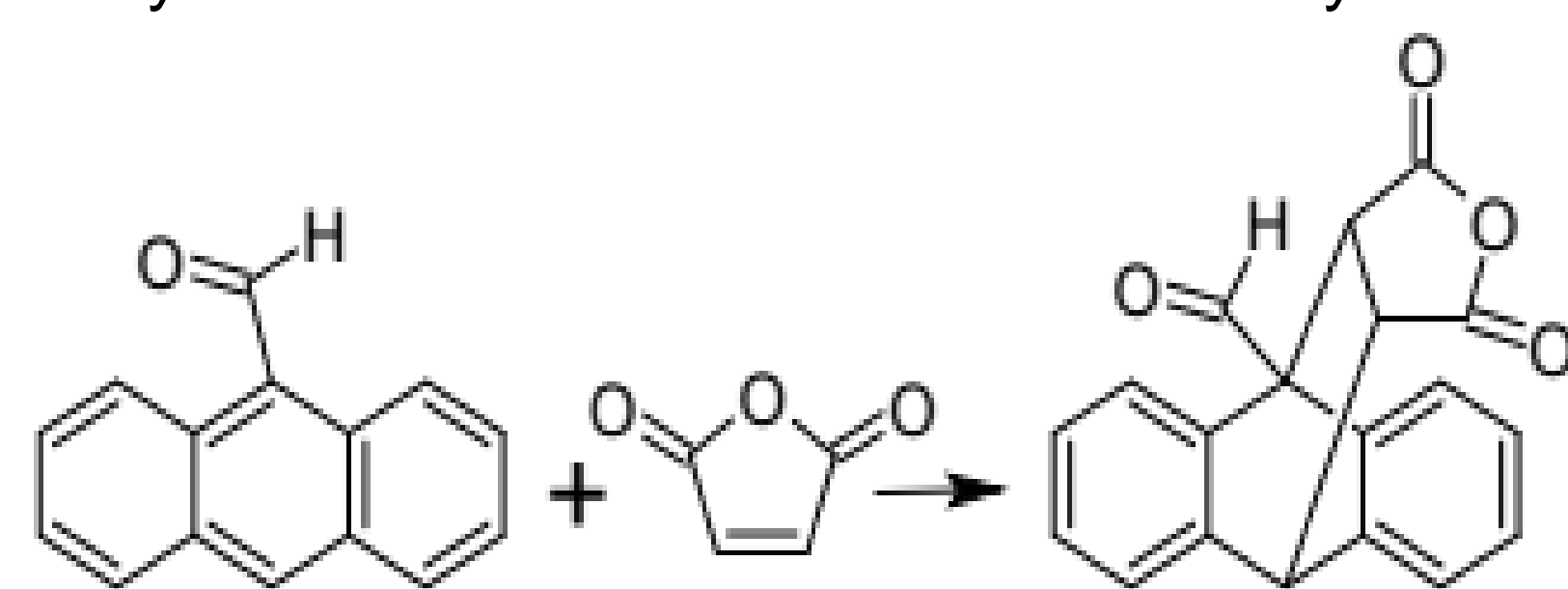


Figure 3: Reaction between maleic anhydride and 9-anthracenecarboxaldehyde

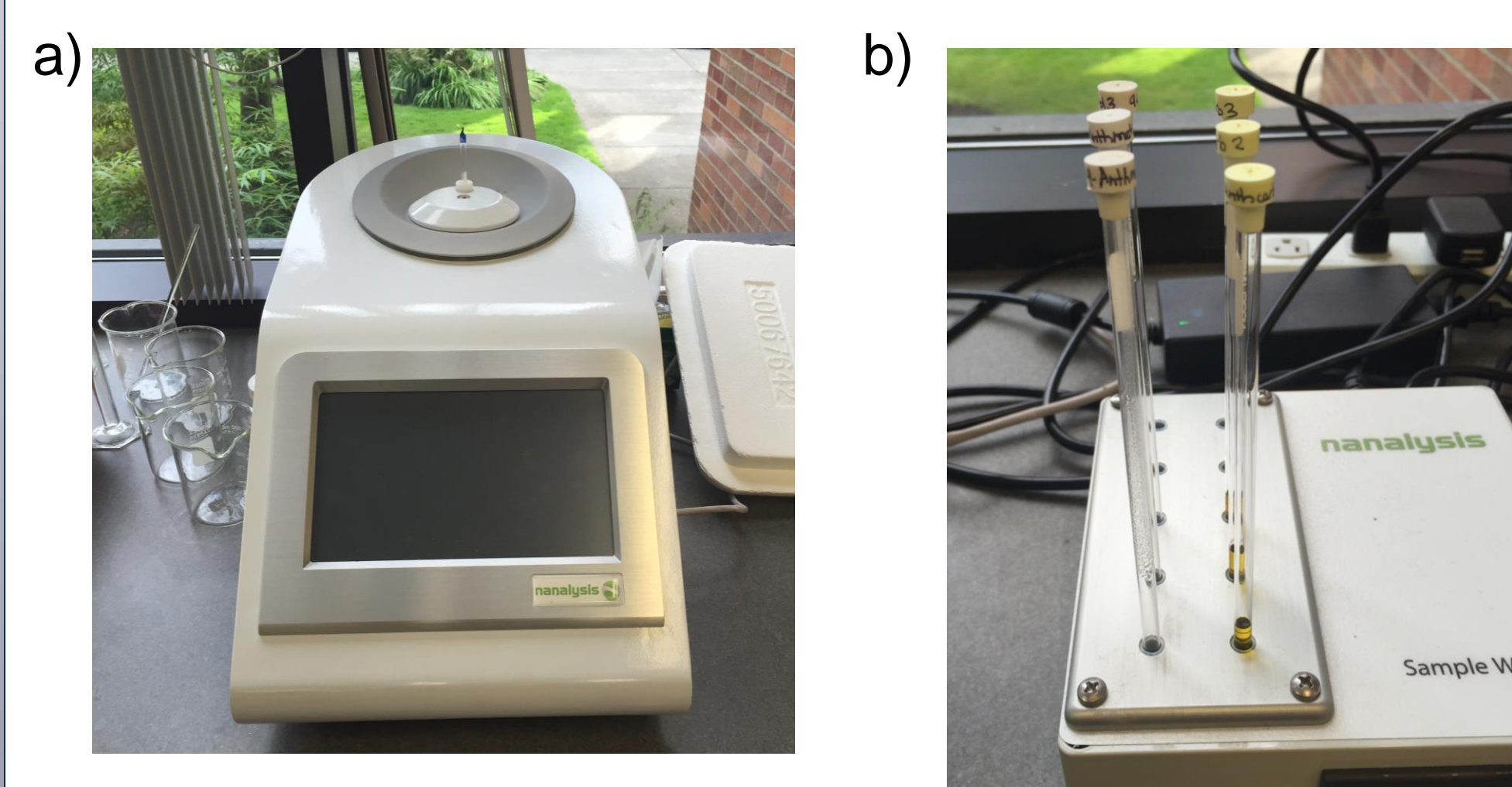


Abstract / Introduction

An internal standard was utilized in this experiment to provide for more accurate recording of the proton integrations³. A 0.1 M concentration of 4-chlorobenzaldehyde was added into the solution prior to placement into the NMR. This was chosen due to its protons not interfering with the most important peaks in the resulting scans.

Methods

Figure 4: a) 60 MHz benchtop HNMR b) NMR tubes and sample warmer.



A 1 M solution was created of each reactant with chloroform-D as the solvent. Solutions were filtered prior to the acquisition of the spectra. The representative peak of the internal standard revealed itself at the position farthest downfield. An initial scan was necessary as soon as the two solutions mixed together. Additional scans were performed as time allowed with special attention paid to the rate of the reaction. For example, the reaction with 9-methylantracene is quite rapid so more frequent scans were required. The other two required approximately 1-2 scans per day. The results from our reactions compare favorably to the literature.⁴

Results / Discussion

Figure 5: Maleic anhydride with Anthracene

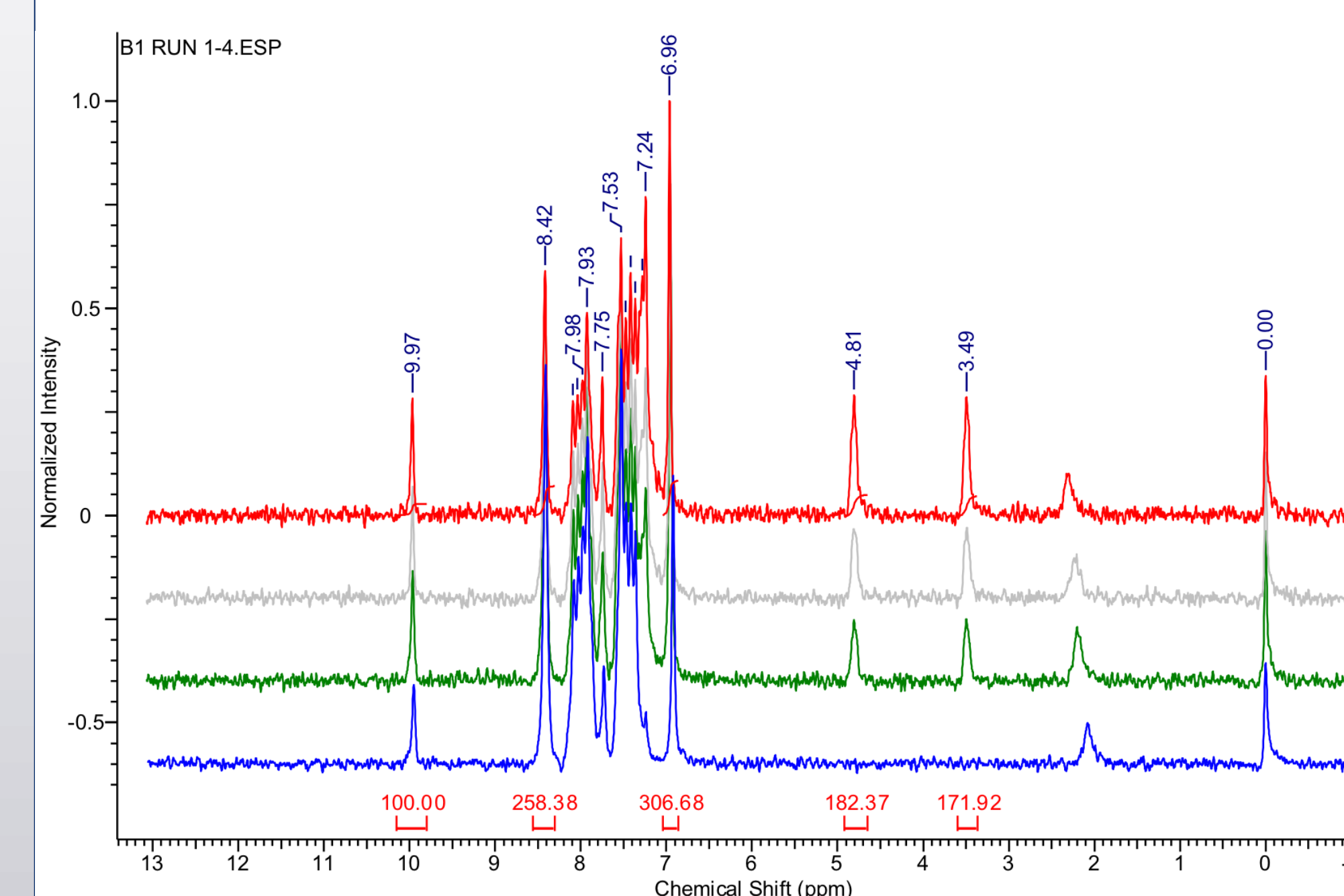


Figure 6: Maleic anhydride with 9-methylantracene

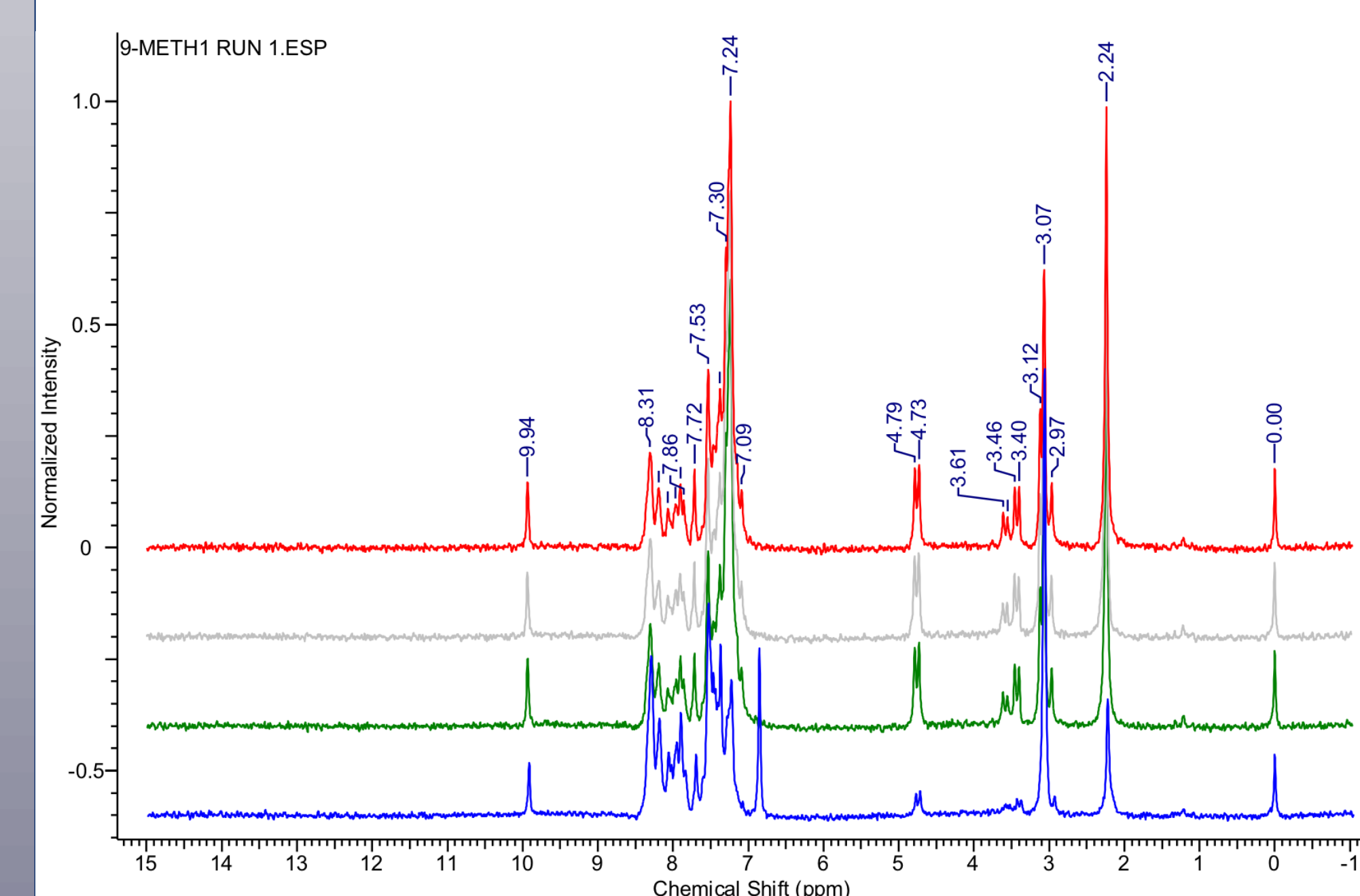
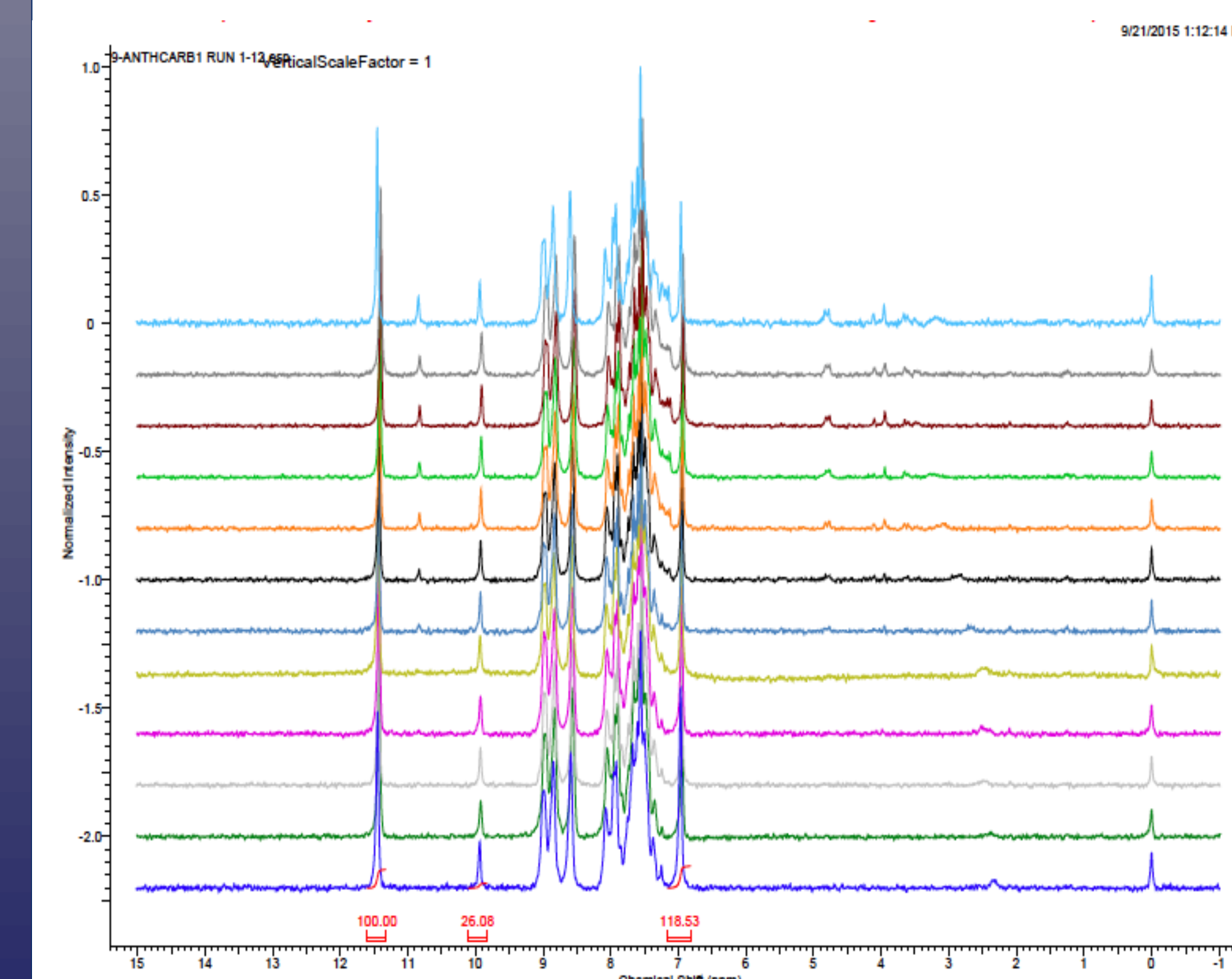
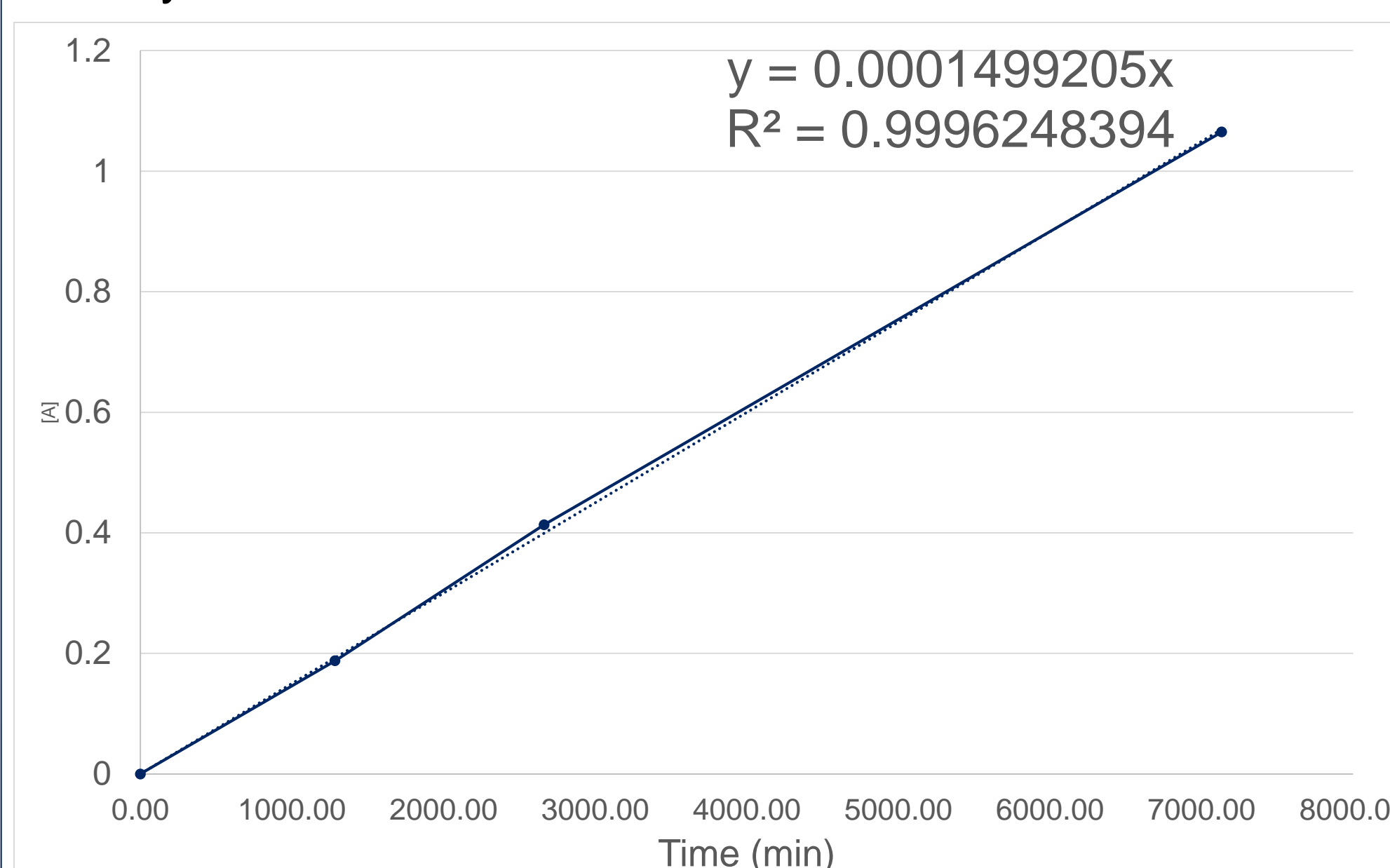


Figure 7: Maleic anhydride with 9-anthracenecarboxaldehyde



Results / Discussion

Figure 8: A representative plot of the 2nd order kinetics observed with the reaction of anthracene and maleic anhydride.



The differences in reactions rates were very obvious from observation and data collection. The addition of the methyl group significantly increased the rate, while the aldehyde significantly decreased the rate. Unfortunately, due to length of the reaction with the aldehyde substituent, not enough data was collected to be useable for this experiment. We are currently in the process of writing a lab to be performed by the Organic Chemistry II class in the spring.

References

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