

Isolation, Extraction, and Identification of Fluorescent Unknown in Friedel-Crafts Acylation of Biphenyl

Hagemann, Nicholai (School: Sand Creek High School)

Acylation of biphenyl with acetyl chloride is a commonly used Friedel-Crafts reaction. Side products observed between the reactant and the acetylating agent are often due to incorrect stoichiometric quantities. Fluorescent complexes were observed and made the focus of this project, while a lack of interest and funding in these byproducts by traditional industries was the motivation. Isolating, extracting, and identifying these develops a methodology to separate compounds with comparable properties. The procedure, proven reproducible, was scaled up to yield a quantity of product sufficient for analysis. Thin-layer chromatography (TLC) determined the product's transition into a mobile phase could be favored in polar liquids. Including a slightly less polar solvent increased the difference in retardation factors. The efficient solvent system, repurposed to column chromatography, was run through a pipet and buret. Fractions were analyzed qualitatively via TLC to assess which contained a single compound. Crystals, collected from creating an oversaturated solution, were transported to a regulated research institution, which had the technical equipment necessary for quantitative analysis. Nuclear magnetic resonance (NMR) indicated fourteen carbon atoms with eight hydrogen atoms. Infrared spectroscopy (IR) exhibited the possibility of the presence of chlorine. Additionally, gas chromatography to mass spectroscopy (GC-MS) characterized possible fragmentation for biphenyl and acetyl chloride derivatives, labeling a possible molecular mass as 279 amu. Collective interpretation alludes to the unknown containing [1,1'-Biphenyl]-4,4'-dicarbonyl dichloride. Recrystallization and X-ray crystallography were considered in plans to continue experimentation.

Awards Won:

