Synthesis of radical precursor 2-chloropyrimidine

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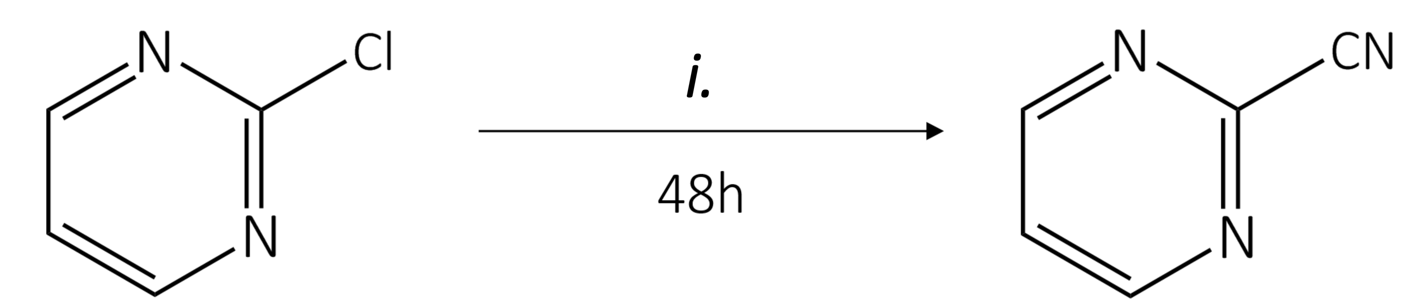
Introduction

Organic spintronics give rise to new field of technology that investigates organic semiconductors, as tools for electronics such as memory devices, or diodes. Organic semiconductors have lower production costs and are much more flexible than their inorganic counterparts. The objective of research is to synthesize 2-chloropyrimidine, the starting material for a series of reactions that produces a radical molecule. When it is arranged in a stack and a delocalized spin is transmitted through the structure, the radical molecule creates the core of the organic semiconductor.

Methods

Four trials of 2-chloropryimidine synthesis are performed. The reaction is a mixture of 1,4-Diazabicyclo[2,2,2]octane, potassium cyanide, and 2-chloropryimidine under nitrogen. The solvent system is dimethyl sulfoxide and water, which is dropped into the mixture at a precise dropping rate of two drops per minute. After dropping, the mixture is set to stir for a minimum of 48 hours. Multiple extractions are performed with diethyl ether and hexanes, subsequently with water. The solvent is removed with a rotary evaporator. Nuclear magnetic resonance spectroscopy and thin layer chromatography are used to determine product purity. The thin layer chromatography solvent system was developed according to observed R_f values throughout trials.

Synthesis



Reaction Conditions. ***i.*** 3.58g KCN, 1.07g 1,4-diazabicyclo[2,2,2] octane
Purification Steps. Extract seven times 90% diethyl ether 10% hexanes, three times H_2O , dry with $MgSO_4$, Rotor evaporation

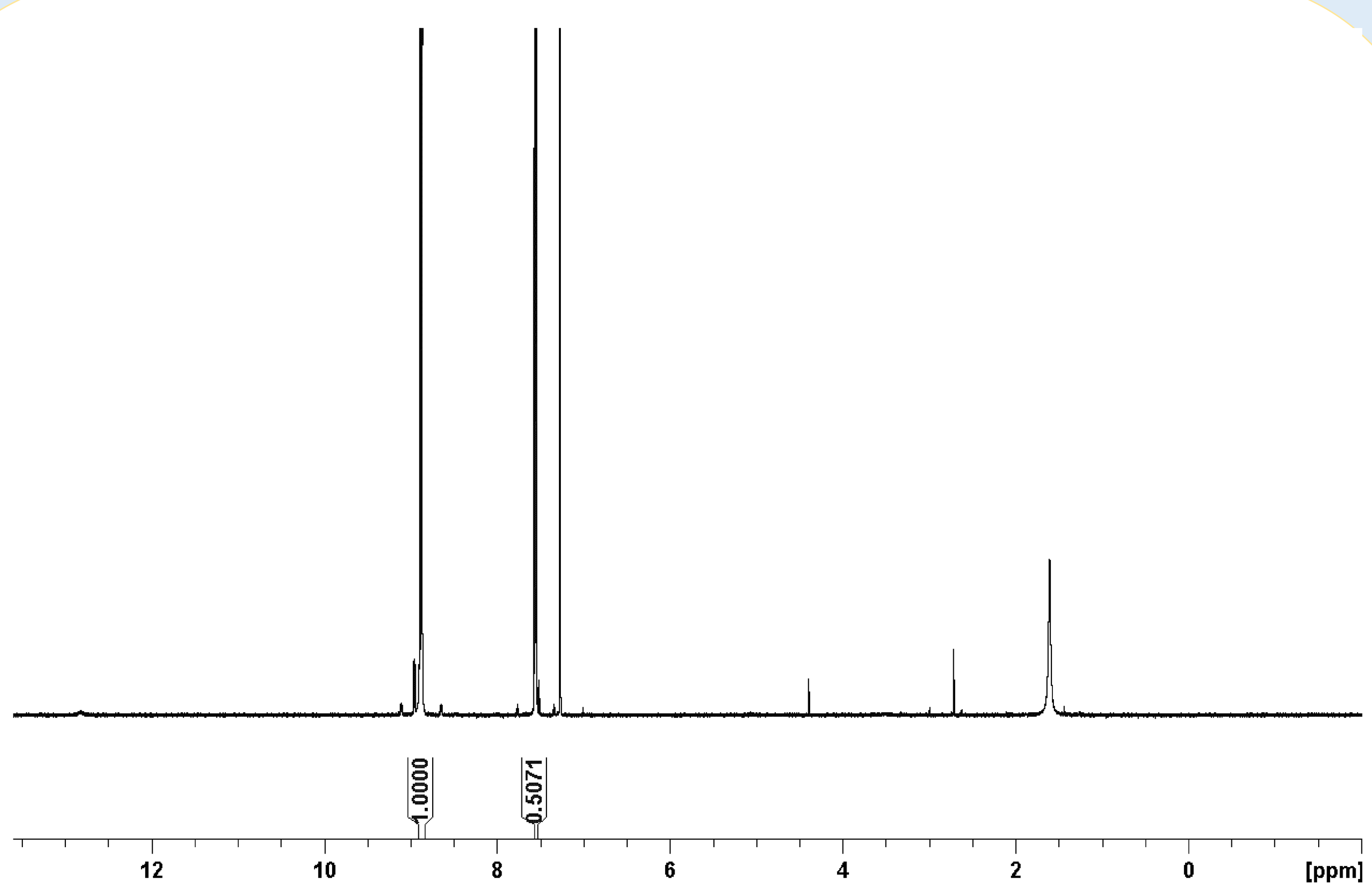
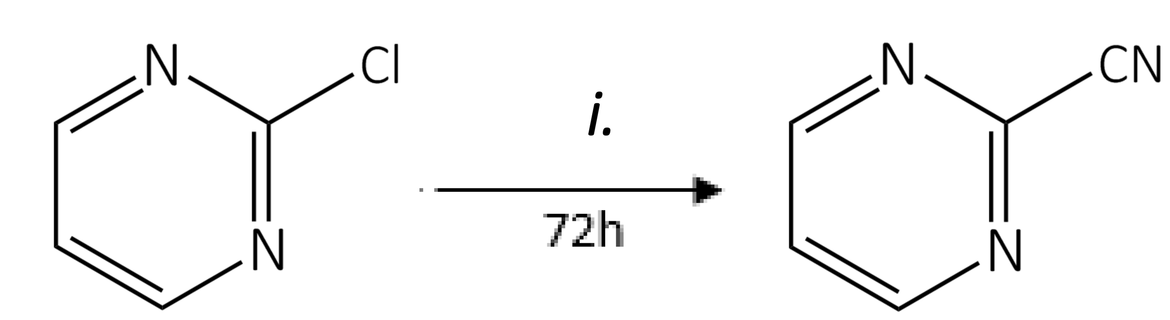


Figure 1: ^{1}H NMR (CCl_4) of 2-cyanopryimidine. The proton NMR shows a pure sample and successful synthesis of the compound. One triplet and one doublet appear on the spectrum: unsuccessful synthesis trials display two triplets and two doublets, the second set of peaks representing 2-chloropyrimidine remaining in the final product.

Results

The procedure to synthesize 2-cyanopyrimidine is successful. Nuclear magnetic resonance techniques displayed in Figure 1 show a pure sample of 2-chloropyrimidine. A pure sample is obtained from two of four samples, with yields of 57.7% and 68.8%. One flaw of the current methodology is failure to remove inorganic material, which causes 2-cyanopyrimidine's to change in consistency and color. One trial is completed with the silica plug as an additional purification step. The silica plug is successful in preventing color and consistency change of the sample. Another determining factor in the yield of the reaction is the reaction time. The sample left to react for an addition 24 hours produced a greater yield by 11.1%. Additional trials must be carried out to verify to correlational relationship between reaction time and yield.
Two proposed modifications to the current methodology are longer stirring time by 24 hours and a silica plug purification step.

Proposed Modifications



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Future Work

Investigation of coloration and consistency of large scale trials of 2-chloropyrimidine synthesis with the modified procedure is a logical next step. 2-chloropyrimidine serves as the starting material for subsequent reactions that will ultimately produce the radical molecule used in an organic semiconductor, so future work involves continuing with the extended procedure to make the radical molecule.

Conclusions

2-chloropyrimidine is the intermediate to the active molecule in an organic semiconductor. Both small-scale and large-scale synthesis in the laboratory have acceptable yields and are organically pure, therefore, synthesis is expected to function on an industrial scale. Industrial manufacturing of 2-cyanopyrimidine facilitates mass production of organic semiconductors, whose fabrication has a lower impact on the environment than its inorganic counterparts, and operate faster. Organic semiconductors are important in order to preserve natural resources.

Literature Cited and Contact

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