

**The Synthesis and Spectroscopic Analysis of Complex
Structural Organic Compounds**

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Abstract

Organic chemistry, the study of carbon-containing compounds, is foundational to many scientific advancements, particularly in developing pharmaceuticals and sustainable chemical processes. Within this field, imines—compounds featuring a carbon-nitrogen double bond—hold significant relevance due to their versatile applications in organic synthesis, medicinal chemistry, and catalysis. This paper details a research project to deepen undergraduate engagement with imine chemistry and spectroscopic techniques by developing laboratory experiments reinforcing key organic concepts and methods. The project involved synthesizing an α -diimine compound using various anilines and sulfanilamide. This helps simulate a common approach in drug development. Challenges encountered during synthesis and analysis were addressed through iterative experimentation and informed problem-solving. These experiences contributed to the design of educational laboratory modules and highlighted the real-world importance of imine compounds in pharmaceutical research. This work reflects the author's growing understanding of the chemical principles that underpin modern medicine and the value of laboratory research in preparing for a healthcare career.

Acknowledgements

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List of Abbreviations and Important Tables

Abbreviation	Definition
^1H	Proton (NMR)
^{13}C	Carbon (NMR)
ACS	American Chemical Society
Ar-BIAN	1,2-Bis[(aryl)imino]acenaphthenes
CDCl_3	Deuterated Chloroform
COSY	Correlation Spectroscopy
Dep-BIAN	1,2-Bis[(2,6-diethylphenyl)imino]acenaphthene
Dpp-BIAN	1,2-Bis[(2,6-diisopropylphenyl)imino]acenaphthene
IR	Infrared
Mes-BIAN	1,2-Bis[(2,4,6-trimethylphenyl)imino]acenaphthene
NMR	Nuclear Magnetic Resonance
PPE	Personal Protective Equipment
PPM	Parts Per Million

Abbreviation	Definition
Sulf-BIAN	4-{[(1E,2E)-2-[(4-sulfamoylphenyl)imino]-1,2-dihydroacenaphthylen-1-ylidene]amino}benzene-1-sulfonamide
ZnCl ₂	Zinc Chloride

NMR Abbreviations and Splitting Patterns

Abbreviation	Definition
¹ H	Proton (NMR)
¹³ C	Carbon (NMR)
s	Singlet
d	Doublet
t	Triplet
q	Quartet
hept	Heptet
m	Multiplet - Generalized Term for more than a Triplet

Introduction

Organic chemistry is the study of compounds containing carbon—their structure, properties, composition, reactions, and synthesis. This branch of chemistry has been instrumental in research that led to the development of life-saving pharmaceuticals, advanced materials, and sustainable chemical processes. A solid grasp of both fundamental and advanced concepts in organic chemistry can pave the way for deeper scientific inquiry that could yield significant scientific advancements.

An area of continued interest within the field is the synthesis and analysis of imines, which are compounds characterized by a carbon-nitrogen double bond. Imines play a crucial role in organic synthesis, medicinal chemistry, and catalytic systems. Typically introduced in the second semester of undergraduate organic chemistry, they are considered foundational knowledge for further study and application.

This paper discusses:

1) The process and experimentation that was conducted, including challenges that emerged and resolution, which led to the development of a series of laboratory experiments that can be integrated into an undergraduate organic chemistry course (The laboratory projects will be discussed in a separate paper to be published in *The Journal of Chemical Education*);

2) The application of the laboratory project methodology to synthesize an α -diimine compound with sulfanilamide to model a common practice used in drug development.

As someone who aspires to be a medical doctor, this project has enhanced my scientific knowledge and helped me appreciate the pharmaceutical relevance of imines and their potential applications in drug development.

Imines

Imines, also known as Schiff bases, are organic compounds characterized by a carbon-nitrogen double bond (C=N). They are widely utilized in pharmaceuticals, catalysis, and materials science. Typically, imines are synthesized through a reaction between a primary amine and a carbonyl group (such as an aldehyde or ketone) under acidic conditions. Modern approaches have enhanced these methods by integrating principles of green chemistry, such as solvent-free reactions, microwave-assisted synthesis, and the use of recyclable catalysts. The significance of imines lies in their broad reactivity and functional flexibility, making them a key player in the interdisciplinary field of organic chemistry. They are essential intermediates in organic synthesis and versatile ligands and scaffolds in drug development and industrial chemistry. Their extensive reactivity and functional versatility enable them to engage in various critical chemical transformations, including reductive aminations, cycloadditions, and nucleophilic additions. In asymmetric synthesis, imines often act as electrophilic partners in the formation of chiral centers, rendering them indispensable for constructing complex, biologically active molecules.

Moreover, in organometallic chemistry, imines function as versatile ligands, as the nitrogen readily binds to different transition metals. Similar to their roles in organic chemistry and biological systems, imines in electrochemical chemistry serve as intermediates for synthesizing various compounds. Imines can also contribute to photochemical oxidation and reactive coupling reactions in this field. From an industrial perspective, they are valuable intermediates for synthesizing various materials, including pharmaceuticals, agrochemicals, and polymers. They serve as precursors for constructing heterocyclic compounds and for pivotal

transformations, such as reductive amination, a cornerstone of medicinal chemistry for generating complex amine derivatives.

Sulfur-Containing Drugs

Sulfur-containing drugs, specifically sulfonamides, commonly referred to as sulfa drugs, represent a historically significant and chemically diverse class of pharmaceutical compounds. First introduced in the 1930s, sulfa drugs were the earliest antimicrobial agents and the first antibiotics used to treat human bacterial infections. The first widely used sulfonamide, prontosil, demonstrated efficacy against streptococcal infections, laying the foundation for developing numerous sulfonamide derivatives. Their discovery and clinical success marked the beginning of the antibiotic era, catalyzing modern medicinal chemistry and the development of pharmaceuticals.

The core of the sulfonamides consists of a sulfonamide functional group ($-\text{SO}_2\text{NH}_2$) attached to an aromatic ring. This group is vital for the drug's mechanism of action, involving the competitive inhibition of the bacterial enzyme dihydropteroate synthase (DHPS), which is crucial in the folate synthesis pathway. By mimicking para-aminobenzoic acid (PABA), sulfonamides disrupt the production of dihydrofolic acid, thereby inhibiting bacterial growth. The toxicity was advantageous. Because humans obtain folate through their diet, they do not synthesize it endogenously.

With the initial success, the clinical use of Sulfa drugs declined due to the emergence of bacterial resistance and adverse hypersensitivity reactions. Common side effects, such as skin rashes, were fine, but severe reactions could manifest as Stevens-Johnson syndrome (SJS), a rare skin and mucous membrane disorder. It's usually a reaction to medication that starts with flu-like

symptoms, followed by a painful rash that spreads and blisters. Then the top layer of affected skin dies, sheds, and begins to heal after several days. Another severe reaction would manifest as toxic epidermal necrosis (TEN), which is a severe form of Stevens-Johnson syndrome (SJS). Both SJS and TEN are life-threatening conditions. Besides their antimicrobial applications, sulfonamide derivatives have been adapted for various therapeutic uses, including diuretics (e.g., furosemide), antidiabetic agents (e.g., sulfonylureas), anticonvulsants (e.g., zonisamide), and anti-inflammatory drugs (e.g., sulfasalazine). These diverse applications underscore the versatility of the sulfonamide moiety in drug design.

Ar-BIANs

Ar-BIANs or 1,2-Bis[(aryl)imino]acenaphthenes are a class of redox-active α -diimine ligands that have garnered significant attention in coordination chemistry due to their unique electronic properties and structural versatility. These ligands are synthesized through the condensation of acenaphthenequinone with arylamines, resulting in a rigid, planar framework that facilitates effective π -conjugation and electron delocalization.

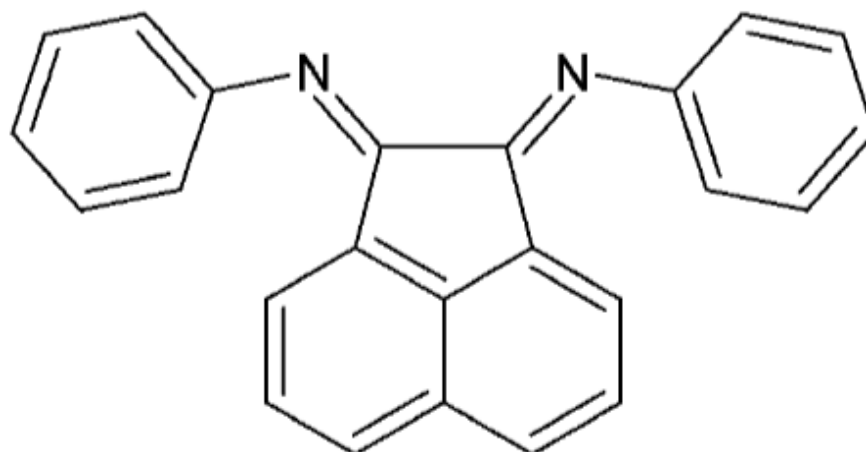


Figure 1

Ar-BIAN ligands have been extensively studied for their ability to stabilize unusual oxidation states and coordination geometries in metal complexes. However, the specific properties of Ar-BIANs that are important to this research are that when disubstituted with aniline derivatives featuring side groups at the 2- and 6-positions, they produce stable and safe molecules. 1,2-Bis[(aryl)imino]acenaphthenes possess several chemical characteristics discussed in organic chemistry. Ar-BIANs, as imines, undergo imine formation when disubstituted with anilines. Additionally, due to their various chemical characteristics, which will be discussed later in this thesis, Ar-BIANs are excellent for spectroscopic techniques.

Nuclear Magnetic Resonance Spectroscopy

Nuclear Magnetic Resonance Spectroscopy, also known as NMR Spectroscopy, is one of the most powerful and widely used analytical techniques for characterizing molecules, usually organic compounds. Since its development in the mid-20th century, NMR has become

indispensable in both academic research and industrial applications, ranging from analyzing small molecules to studying complex biomolecules. At its core, NMR spectroscopy uses the magnetic properties of specific atomic nuclei. When placed in a strong external magnetic field, nuclei such as ^1H , ^{13}C , ^{19}F , or ^{31}P , which have a non-zero nuclear spin, absorb radiofrequency radiation at specific resonant frequencies. These frequencies are influenced by the electronic environment surrounding the nucleus, which allows NMR to provide detailed information about molecular structure, connectivity, and dynamics.

The most common technique used in organic chemistry is ^1H NMR, which analyzes the hydrogen atoms in a compound. Peaks in a ^1H NMR spectrum correspond to different hydrogen environments, and their chemical shift (δ), splitting patterns (J-coupling), and integration give insight into the number of hydrogens, their neighboring atoms, and their relative positions. At the same time, ^{13}C NMR is used to gather the carbon skeleton structure, where chemical shifts reveal the nature of carbon atoms, such as methyl, methylene, carbonyl, or aromatic carbons. With significant advances in 2-Dimensional NMR, techniques such as COSY (correlation spectroscopy) and HMBC (Heteronuclear Multiple Bond Correlation) have extended the capability of NMR to elucidate larger and more complex structures by correlating nuclei through spin-spin couplings across one or more bonds. Besides structural determination, NMR can be used for various analytical methods. NMR can be used to investigate and explore reaction mechanisms, conformational analysis, purity and identity of compounds, and intermolecular interactions. Making NMR is a valuable analytical tool for characterizing molecules, as well as in drug discovery and development.

Infrared Spectroscopy

Infrared Spectroscopy, also known as IR spectroscopy, is a fundamental analytical technique used in organic chemistry to identify and characterize chemical compounds by measuring the vibrational frequencies of the chemical bonds. Each type of bond absorbs infrared radiation at characteristic frequencies, corresponding to specific vibrational modes, such as stretching and bending. When infrared radiation is passed through a sample, bonds absorb energy at characteristic wavenumbers (typically measured in cm^{-1}), producing an absorption spectrum that serves as a molecular fingerprint.

The bonds of each functional group absorb infrared light at specific frequencies, allowing chemists to deduce structural features quickly. For example, O–H stretching in alcohols appears broadly around $3200\text{--}3600\text{ cm}^{-1}$, while C=O stretching in carbonyl compounds is typically sharp and intense near 1700 cm^{-1} . Aromatic C=C stretches, nitrile groups, and amines also exhibit unique vibrational signatures. By analyzing these absorption bands, chemists can identify unknown compounds, track the progress of reactions, and confirm the presence of specific functional groups.

A few advantages of IR spectroscopy are its speed, simplicity, and minimal sample preparation. However, one key benefit is that, unlike NMR, it can analyze gases. IR spectroscopy is often used in conjunction with other spectroscopic techniques, such as NMR and mass spectrometry, to gain a comprehensive understanding of a compound's identity and purity. With several technological advances, including NMR and Fourier-transform infrared (FTIR) spectroscopy, now standard in most labs, the resolution and sensitivity of IR measurements have been enhanced, enabling rapid and precise analysis. In synthetic organic chemistry, IR spectroscopy is routinely used to verify the formation of target products by confirming the

presence (or absence) of key functional groups. It also plays a critical role in quality control, polymer characterization, environmental analysis, and materials science.

Lab Project - The Synthesis and NMR Analysis of Complex Structural Imines

Introduction/Background

As discussed previously, organic chemistry is a cornerstone of both chemistry and science. Furthermore, several topics in organic chemistry, such as imines, are essential for undergraduates to learn due to their significance in chemistry and their inclusion on the MCAT syllabus. With this in mind, this segment of the research aimed to create a series of teaching laboratory experiments that would enable students to perform reaction chemistry and synthesize imines through safe, quick, small-scale reactions, yielding high amounts of pure products.

Supporting these efforts, advancements in NMR spectroscopy, particularly the increasing resolution and accessibility of benchtop NMR spectrometers, offer a valuable analytical tool for undergraduate labs. These lower-cost, low-maintenance instruments make spectroscopy more feasible for smaller institutions. For example, in 2019, the Chemistry department at Sarah Lawrence College acquired a Nanalysis 60 Pro NMR benchtop spectrometer. Research conducted by Nicholle Chew (2019) using an NMR benchtop spectrometer to analyze various Ar-BIANs revealed initial findings on the use of Ar-BIANs disubstituted with anilines featuring multiple side groups, primarily at the 2- and 6-positions, in this organic chemistry lab project. In addition to having positive initial data from Nicholle Chew (2019) and conducting a chemical synthesis reaction related to imines, we also aimed to create several experiments optimized for low-field benchtop NMR.

Building on that work and aiming to fill a gap in the literature, since most NMR-based experiments are designed for high-field instruments, we created several experiments specifically optimized for low-field benchtop NMR. These multi-week experiments guide students through the synthesis of imines, beginning with examples published by El-Ayaan^{1,2} *et al.* and progressing to related compounds with the same structural backbone. By performing both acid-catalyzed and Lewis acid-catalyzed reactions, students learn to compare synthesis pathways and apply NMR to characterize the products, thereby deepening their understanding of both organic chemistry and spectroscopic analysis.

Learning Goals

The learning goals for the organic chemistry lab project are related to Imines and imine formation and NMR spectroscopy. Some of these goals were:

Imines and Imine Formation

- What are imines?
- Describe imine formation and its importance
- Be able to synthesize imines
 - By Brønsted-Lowry-acid catalyzed reactions
 - By Lewis-acid catalyzed reactions
- Explain the mechanism behind the synthesis

NMR Spectroscopy

- Describe the basics of NMR spectroscopy
- Set up NMR samples

- Operate an NMR benchtop spectrometer
 - Run ^1H and ^{13}C Experiments
- Understand how to analyze and interpret NMR spectra.
- Discuss the differences between low-field and high-field spectrometers and their spectra.

Hazards and Safety

Hazards

During the Lab Project students will use: hot glassware; acetic acid; zinc chloride (ZnCl_2); hexane; potassium carbonate; and various anilines. Some of the chemical hazards include:

- Acetic acid is a corrosive liquid that can cause severe skin burns and eye damage; it is also harmful if inhaled.
- Zinc chloride (ZnCl_2) is caustic and can irritate the skin, eyes, and respiratory tract.
- Hexane is a highly flammable liquid and vapor, and prolonged or repeated exposure may cause organ damage.
- Anilines are toxic and may be absorbed through the skin, resulting in harmful systemic effects.
- Potassium carbonate is an alkaline, non-combustible substance that can irritate the skin, eyes, and respiratory tract. It should be handled with protective equipment and stored away from moisture and acids.
- Many anilines are classified as potential carcinogens and should be handled with caution.

All waste materials, including reaction residues and solvents, must be collected and disposed of per institutional hazardous waste disposal guidelines. The safety data sheets for all the chemicals used in all the experiments are included in the supporting materials.

Safety

The safety protocol discussed is a generic protocol, where students must wear standard protective laboratory eyewear and appropriate chemical-resistant gloves throughout the procedure to prevent direct contact with hazardous materials. Lab coats and closed-toe shoes must also be worn at all times to minimize the risk of contamination and skin exposure. All manipulations involving hot glassware, volatile solvents, corrosive reagents, or toxic compounds must be carried out in a properly functioning, certified chemical fume hood to ensure adequate ventilation and to limit inhalation of harmful vapors. Bench-top work should be minimized for substances that produce flammable or toxic fumes. Careful attention must be given to the handling and transfer of reagents to prevent spills and accidental releases. Before beginning the experiment, students should be trained in the location and proper use of safety equipment, including eyewash stations, safety showers, and fire extinguishers. Adherence to these safety protocols is critical to maintaining a safe working environment and protecting all participants from potential chemical hazards.

Chemical Scheme of Various 1,2-bis[(aryl)imino]acenaphthenes

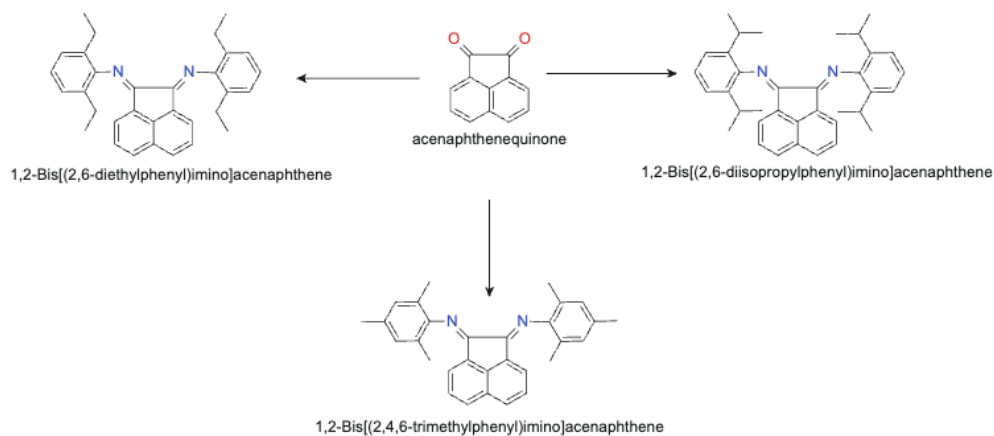


Figure 2

Experimental Overview

The lab project comprises six experiments, including three acid-catalyzed reactions and three Lewis acid-catalyzed reactions. Each experiment focuses on the chemical reaction between Acenaphthenequinone and each one of the three primary amines. By optimizing several experiments, a lab experiment unfolds over three to four detailed lab sessions, each lasting between 2.5 and 3.5 hours, and is complemented by an overnight ^{13}C NMR experiment. Students are to engage in these experiments in pairs.

Dpp-BIAN

The experimental overview first discusses the synthesis of 1,2-bis[(2,6-diisopropylphenyl)imino]acenaphthene, Dpp-BIAN, by a Brønsted-Lowry-acid catalyzed reaction, as first reported by El-Ayaan^{1,2} *et al.* The original procedure was already good. However, we chose to optimize the Brønsted-Lowry acid-catalyzed Dpp-BIAN synthesis

because it is simple, produces high yields, and requires fewer total lab sessions compared to the alternative Lewis-acid-catalyzed reactions. The Dpp-BIAN Brønsted-Lowry acid-catalyzed reaction synthesis was the most optimized; we modified procedures from the original literature protocol for the reaction. We also reduced the amount of reagents used to get a yield of 1.5 grams. We also used acetic acid as the sole solvent, rather than acetonitrile, to minimize costs and also because acetonitrile is considered more hazardous than acetic acid.

One week before each laboratory session, we read a detailed lab procedure document outlining the specific experiment we conducted. This document includes information about the objectives, required materials, safety protocols, and step-by-step methodologies, which students must review thoroughly. This preparatory phase is crucial for helping us grasp the essential concepts involved in the experiment.

On the first day of the laboratory session, we meticulously reviewed the lab procedure, ensuring that we fully understood each step. We wore all our PPE properly, including safety goggles, gloves, and lab coats, to prioritize safety throughout the experiment. During this initial session, we began by accurately measuring and recording the quantities of reagents required for the experiment. For the Dpp-BIAN synthesis, we carefully measured 1.35 g of acenaphthenequinone and added it to a clean, dry 500 mL round-bottom flask. Then we measured and added 78 mL of acetic acid. Once the reagents were combined, we placed the flask under a fume hood and used a heating mantle with a stirrer to reflux, maintaining a constant temperature of 110 degrees Celsius for 30 minutes. This allowed acenaphthenequinone to fully dissolve in the acetic acid; ergo, a more effective reaction. After the initial reflux period, we added 3 mL of 2,6-diisopropylaniline to the round-bottom flask, ensuring it continued to reflux with the preheated reactants. The flask remained under the fume hood as we continued the reflux

process for an additional 1.5 hours, again maintaining the temperature at 110 degrees Celsius. This extended heating allowed for further reaction and formation of the desired product.

Once 1.5 hours had elapsed, we carefully turned off the heat source and let the mixture cool gradually to room temperature. This cooling phase was crucial for the subsequent steps in the experimental procedure, which took place over the following week. Throughout the process, we took detailed notes and made observations to see what we could optimize or change. Taking notes also fostered a deeper understanding of the chemical reactions and techniques involved in the synthesis.

On the second day of the Dpp-BIAN synthesis, we washed our mixture with hexane to remove the acetic acid through a Buchner filter funnel. Then we allowed air to pass through the Buchner funnel to speed up the drying process. Once dried, we calculated our yields. We prepared an NMR sample by adding 0.05 g of Dpp-BIAN to an NMR tube, and then we added 0.750 mL of CDCl_3 . After we made our sample, we ran a 64 scan ^1H NMR for around five minutes and saved the spectra. Then we ran a ^{13}C NMR experiment overnight.

The following day, we saved our ^{13}C NMR spectrum and then conducted a 1.5-hour-long COSY (Correlation Spectroscopy) experiment, a two-dimensional proton NMR technique. This method allowed us to identify correlations between protons coupled through chemical bonds, providing valuable insights into the sample's molecular structure. We then cleaned up the lab and set up another experiment, usually the Mes-BIAN, Lewis acid-catalyzed reaction.

Mes-BIAN

The following experiment we did was the 1,2-Bis[(2,4,6-trimethylphenyl)imino]acenaphthene, Mes-BIAN, Lewis acid-catalyzed reaction.

In our first session, we measured and added 18 g of acenaphthenequinone, 45.9 g of anhydrous zinc chloride, 31.5 mL of 2,4,6-trimethylphenylaniline, and 300 mL of acetic acid. Then, we added it to a 500 mL round-bottom flask with a stir bar. The mixture was then stirred and refluxed at 80°C for 1 hour. After one hour, the solution was allowed to sit for at least 12 hours for recrystallization. During the second session, we extracted the product using vacuum filtration with a Buchner filter funnel. First, we washed the product with acetic acid for 10 washes, and then we washed it with diethyl ether until the acetic acid odor was faint. Using the Buchner filter funnel to pass air through the intermediate, we allowed it to dry.

During the next session, we ground the solid intermediate in a mortar and pestle and then weighed it to get the yield. After gathering the yield, we then measured out 12 g of the zinc Mes-BIAN complex intermediate, 100 g of K_2CO_3 , and 250 mL of water. Then we added it to a 500 mL round-bottom flask with a stirrer. Continuing, we heated and refluxed for 3 hours at 100 degrees Celsius. After the 3 hours of reflux, the product was left to cool for at least 24 hours.

Next, we used gravity filtration to remove zinc carbonate, excess potassium carbonate, and water. The product was left to dry overnight. The next day, we boiled 300 mL of ethanol in a 500 mL round-bottom flask, added the dried product to the boiling ethanol, and waited until the solid in the boiling solution fully dissolved. Once fully dissolved, while still warm, we poured the solution through gravity filtration. We kept the ethanol solution; the solid $ZnCl_2$ was removed. We then put the ethanol solution in a flask and placed it under a fume hood to evaporate to about a quarter of its original volume, which we expected to take 2-3 days.

However, when we returned midway through the third day, all the ethanol had already evaporated, and crystals had formed. We removed the crystals from the flask, ground them in a mortar and pestle, and then weighed the product to determine the yield. We prepared an NMR

sample by adding 0.05 g of Mes-BIAN to an NMR tube and then adding 0.750 mL of CDCl₃. After we made our sample, we ran a 64 scan ¹H NMR for around five minutes and saved the spectra. We then ran a ¹³C NMR experiment overnight. The following day, we saved our ¹³C NMR spectrum and then conducted a 1.5-hour-long COSY (Correlation Spectroscopy) experiment, a two-dimensional proton NMR technique.

Dep-BIAN

The experiment we did was similar to a Dpp-BIAN experiment, but modified by swapping 2,6-diisopropylaniline for 2,6-diethylaniline. This modification allowed us to synthesize [(2,6-diethylphenyl)imino]acenaphthene, Dep-BIAN.

In the first session, we accurately measured and recorded the quantities of reagents required for the experiment. For the Dep-BIAN synthesis, we carefully measured 0.45 g of acenaphthenequinone and added it to a clean, dry 250 mL round-bottom flask. Then we measured and added 26 mL of acetic acid. Once the reagents were combined, we placed the flask under a fume hood and used a heating mantle with a stirrer to reflux, maintaining a constant temperature of 110 degrees Celsius for 30 minutes. This allowed acenaphthenequinone to fully dissolve in the acetic acid, in a more effective reaction. After the initial reflux period, however, instead of adding 2,6-diisopropylaniline, we added 1.2 mL of 2,6-diethylaniline to the round-bottom flask, ensuring it continued to reflux with the preheated reactants. The flask remained under the fume hood as we continued the reflux process for an additional 1.5 hours, again maintaining the temperature at 110 degrees Celsius. This extended heating allowed for further reaction and formation of the desired product.

Once 1.5 hours had elapsed, we carefully turned off the heat source and let the mixture cool gradually to room temperature. This cooling phase was crucial for the subsequent steps in the experimental procedure, which took place over the following week. Throughout the process, we took detailed notes and made observations to see what we could optimize or change. Taking notes also fostered a deeper understanding of the chemical reactions and techniques involved in the synthesis.

On the second day of the Dep-BIAN experiment, we washed our mixture with hexane to remove the acetic acid through a Buchner filter funnel. Then we allowed air to pass through the Buchner funnel to speed up the drying process. Once dried, we calculated our yields. We prepared an NMR sample by adding 0.05 g of Dep-BIAN to an NMR tube and then adding 0.750 mL of CDCl_3 . After we made our sample, we ran a 64 scan ^1H NMR for around five minutes and saved the spectra. Then we ran a ^{13}C NMR experiment overnight.

The following day, we saved our ^{13}C NMR spectrum and then conducted a 1.5-hour-long COSY (Correlation Spectroscopy) experiment, a two-dimensional proton NMR technique.

Results and Discussion

Each of the three experiments was completed to determine how long it would take and to see if the different 1,2-bis[(aryl)imino]acenaphthenes met our expectations for a good NMR spectrum. We primarily optimized the Dpp-BIAN acid-catalyzed reaction because it was a simple synthesis experiment with an easy way to purify the product and produced high yields. However, we were not able to fully optimize the other experiments, such as Mes-BIAN's Lewis-acid-catalyzed experiment and Dep-BIAN's Brønsted-Lowry acid-catalyzed experiment.

This paper focuses on Dpp-BIAN and discusses both Mes-BIAN and Dep-BIAN results, data, and changes in the experiments.

Dpp-BIAN

The Dpp-BIAN experiment is considered the best across the lab project because it is less hazardous, simpler, has a shorter duration, and leads to a favorable price-to-yield ratio.

It is important to note that we modified the literature protocol for the acid-catalyzed reaction by using acetic acid as the sole solvent, instead of acetonitrile. This modification has two main benefits:

1) It made the process become safer, as acetonitrile in the body can be hydrolyzed to produce cyanide, which is highly toxic and deadly;

2) It made the experiment cost-effective which helps in achieving a more favorable yield-to-cost ratio.

Even with these changes, we could add all the reagents, except the aniline, to a round-bottom flask. We let it reflux for 30 minutes, allowing the acenaphthenequinone to dissolve fully in the acetic acid. After these 30 minutes, we added the 2,6-diisopropylaniline; the acetic acid serves as both the solvent and the catalyst. Within a few steps, over 35 minutes, we combined all the reagents in a round-bottom flask, facilitating the reaction. The synthesis reaction only took 1.5 hours, allowing the Dpp-BIAN acid-catalyzed reaction to be completed within one lab session. Within two lab sessions, we were able to obtain a fully purified product to analyze. When we compared this Dpp-BIAN experiment to other lab experiments in the lab project, the other lab experiments took longer, usually requiring three to four lab sessions. By reducing the target yield from the original protocol of 3.15 g for the acid-catalyzed reaction to

ideally get 1.5 g of the imine, the amount of reagents needed is reduced. Reducing the amount of reagents and products helps reduce costs, as there are fewer reagents to purchase and fewer products to dispose of at the end of the year. Besides being more straightforward and quicker, our new modified procedure has a favorable yield-to-cost ratio. The original method, using acetonitrile, yields 77%, while our procedure yields an average of 60%. However, four liters of lab-grade acetonitrile costs \$765, whereas the cost of acetic acid is \$302 for the same volume. Thus, it is more cost-effective to use acetic acid as the solvent and catalyst, since a 10% difference in yield results in a \$300 savings.

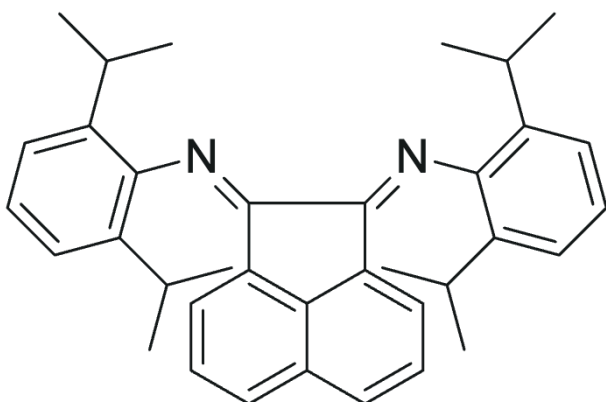


Figure 3

The Dpp-BIAN is a great molecule for teaching several organic chemistry topics. With a quick and straightforward reaction, we were able to obtain high yields of a very stable imine at a reasonable cost, producing enough Dpp-BIAN material for use in NMR analysis, as well as for any other analytical techniques the instructor may want to use, such as IR spectroscopy, melting

point, and possibly mass spectrometry. As an imine, Dpp-BIAN is excellent for teaching analytical techniques, especially NMR Spectroscopy.

The figure shows a ^1H spectrum collected from our Nanalysis 60 Pro, a 60 MHz benchtop spectrometer.

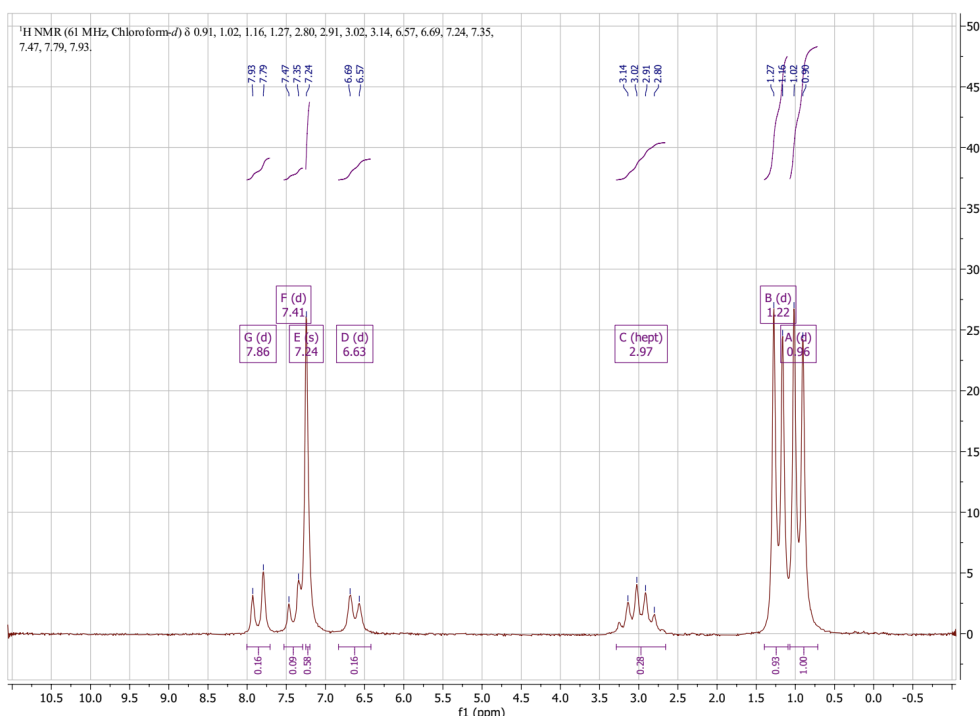


Figure 4

The proton 64 scan spectrum was collected in under five minutes using our Nanalysis 60 Pro benchtop spectrometer. We obtained excellent signals and apparent peak splitting, which was due to the time at which the spectrum was acquired. This allowed us to see why Dpp-BIAN is well-suited for NMR Spectroscopy.

The Dpp-BIAN spectrum displays a variety of signals that utilize the entire spectrum. We saw both low and high signals, indicating the presence of aliphatic and aromatic groups within

the molecules. In the aliphatic region, we identified a diisopropyl splitting pattern: two doublets in the 1.0-1.3 ppm range and a multiplet signal that, upon resolution, reveals a heptet splitting pattern in the 3.0 ppm range. When analyzing the two doublets and examining a three-dimensional model of Dpp-BIAN, it can be observed that the signals are influenced by steric hindrance. Students in organic chemistry typically do not experience steric hindrance around a sigma bond, as simple organic molecules that exhibit this characteristic are rare. In contrast, molecules with symmetry due to unrestricted free rotation about single bonds are far more common.

In the aromatic region, we identified the naphthene signal moiety, which consists of two doublets at 6.65 ppm and 7.87 ppm, and a triplet at 4.39 ppm. Additionally, the few hydrogens on the benzene ring of the amine group gave rise to a singlet at 7.27 ppm. In this aromatic region, we saw an overlap between the triplet of naphthene and the singlet of the amine group. Even with the overlapping signals, the Dpp-BIAN proton spectra were good, clear, and easy to identify, determining the compound's characteristics.

With such a clear NMR spectrum, Dpp-BIAN, noted for its distinct NMR spectral features, serves as an exemplary model compound in chemical education. The molecule provides identifiable signals within the aromatic and aliphatic regions, with a notable diisopropyl splitting pattern attributable to steric hindrance. This characteristic and other spectral features within a single molecule offer an invaluable tool for elucidating key NMR spectroscopy principles. Its structural simplicity combined with spectral complexity facilitates student engagement in identifying and analyzing spectral characteristics, thereby enhancing the instructional efficacy of sort NMR exercises.

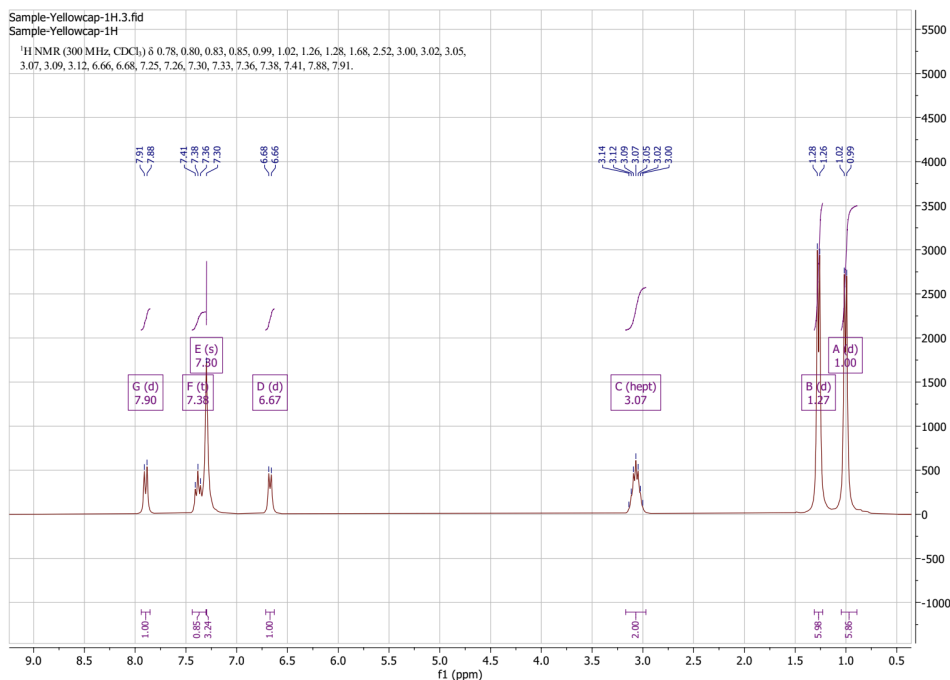


Figure 5

A comparative spectral analysis of Dpp-BIAN at various magnetic field strengths highlights its value in educational environments. The proton NMR spectra of the compound, recorded using both 60 MHz and 300 MHz spectrometers, vividly reflect how magnetic field strength affects spectral resolution and peak distribution. For example, the 60 MHz spectrum shows significant signal overlap, which is markedly different from the sharper resolution found in the 300 MHz spectrum, as shown in Figure 3 (or possibly Figure 4). At 300 MHz, the enhanced spectral resolution is evident through clearer peaks and well-defined splitting patterns, with signals further apart, effectively reducing the overlap seen in lower-field spectra.

We used CCNY's 300 MHz spectrometer to acquire several of these 300 MHz spectra. While using the 300 MHz, we noticed that the time required to acquire both proton and carbon spectra was significantly shorter on the 300 MHz than on our 60 MHz benchtop spectrometer. The stronger magnetic field allowed for the shorter duration.

Based on these findings and changes, incorporating Dpp-BIAN's spectra into laboratory curricula provides a dynamic platform for students to engage with the fundamental concepts of NMR spectroscopy. Comparative spectral analyses, facilitated by varying magnetic field strengths, elucidate key spectral differences and reinforce core theoretical principles. This immersive, hands-on approach bridges the gap between abstract theoretical knowledge and tangible practical application, thereby solidifying Dpp-BIAN's utility as a model in chemical education.

Mes-BIAN

The Mes-BIAN experiment took significantly longer to complete than the Dpp-BIAN, primarily due to the Lewis-acid-catalyzed reaction. However, using a Lewis acid, specifically zinc chloride (ZnCl_2), substantially enhances the reaction between 2,4,6-trimethylaniline and acenaphthenequinone, resulting in a greater reaction than traditional Brønsted-Lowry acid-catalyzed processes. This method holds value in organic synthesis, where Brønsted-Lowry acids protonate substrate molecules, facilitating various reaction pathways. In contrast, zinc chloride does not donate protons; instead, the zinc dication, Zn^{2+} , forms a coordinate covalent bond with the nitrogen atom of 2,4,6-trimethylaniline, by accepting its lone pairs of electrons. This strong bonding alters the reaction mechanism: instead of the trivial deprotonation step, additional reactions are required to remove the zinc chloride from the final product. This distinction emphasizes the difference between imine formation from Brønsted-Lowry acid-catalyzed versus Lewis-acid-catalyzed reactions.

Though it exemplifies Lewis-acid-catalyzed reactions, the experiment may take too long to use in a regular teaching laboratory. However, it may still be beneficial in teaching students

about the various possible reactions that can synthesize imines. After doing both the Dpp-BIAN and Mes-BIAN reactions, students can see and discuss the similarities and differences between an acid-catalyzed reaction and a Lewis-acid reaction.

After removing the ZnCl_2 , the final yield was 68%, while the intermediate yield was 67%. When we compare that to the published yield in El-Ayaan *et al.*^{1,2}, the final yield was 70% while the intermediate yield was 90%. This may indicate that our reaction had several errors. This is one area where we could optimize the experiment. We noticed that when you increase the scale of the reaction, the yields become inconsistent. By conducting smaller-scale reactions, we can increase yields while minimizing the errors we or students might make. Another area we wanted to improve was reducing the time needed for the three-hour reaction with K_2CO_3 . If the scale were smaller, we could decrease the time since there are fewer products to purify. This idea needs to be further tested in other experiments.

When you compare Mes-BIAN, the proton spectrum is less exciting than Dpp-BIAN.

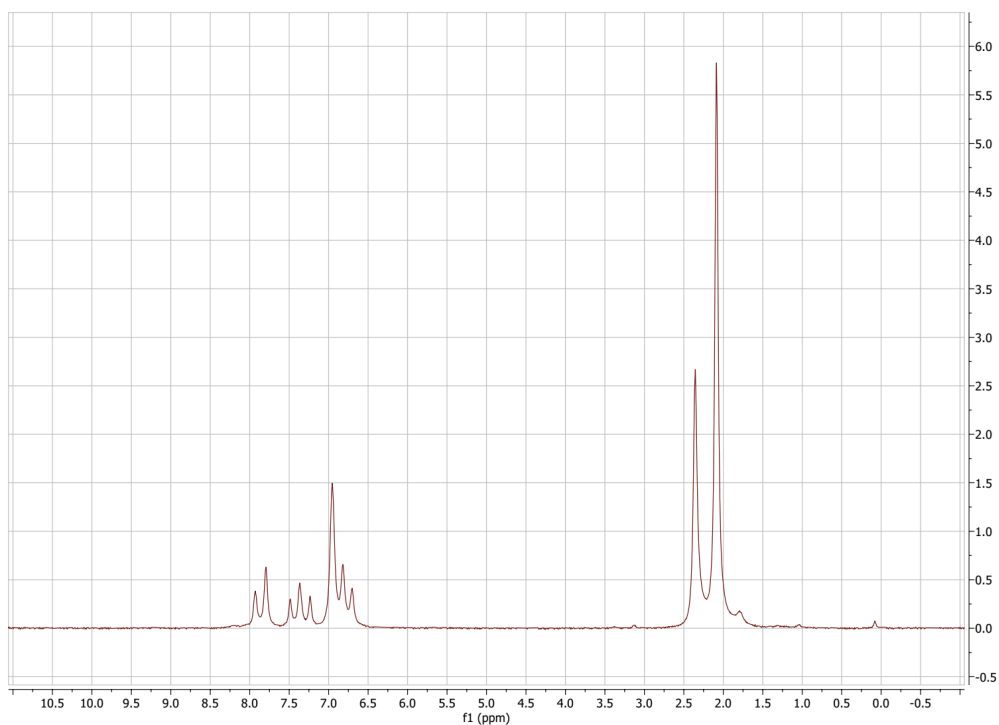
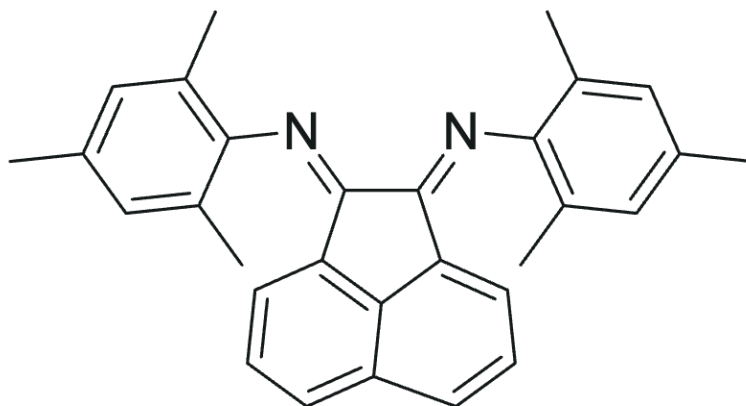


Figure 6

The Mes-BIAN ^1H NMR spectrum had 64 scans and was estimated to have an acquisition time of around 5 minutes. The obtained spectrum had clear signals and fully-resolved splitting patterns. Similar to Dpp-BIAN, the Mes-BIAN spectrum displays a variety of signals that utilize the entire spectrum. We could see both low and high signals, indicating the presence of aliphatic and aromatic groups within the molecules. We can identify two singlets in the 2.2-2.4 ppm range in the aliphatic region. When analyzing the two singlets and examining a three-dimensional model of Mes-BIAN, we can observe that the methyl groups at the 2- and the 6-positions of the phenyl group give the same signal, indicating that there is free rotation of the 2,4,6-trimethylphenyl group. When we say the spectrum is not as exciting as Dpp-BIAN, the molecule exhibits free rotation about the N-C bonds, as expected from a knowledge of basic organic chemistry.

In the aromatic region, we can identify the naphthene signal moiety, which consists of two doublets at 6.65 ppm and 7.87 ppm, and a triplet at 4.39 ppm. Additionally, the few

hydrogens on the benzene ring of the amine group give rise to a singlet at 7.0 ppm. In this aromatic region, we can identify an overlap between the naphthene's first doublet and the aniline group's singlet. Even with the overlapping signals, the Mes-BIAN proton spectra are good, clear, and easy to identify, determining which signal and splitting go with each hydrogen.



When you look at the high field ^1H NMR spectrum of Mes-BIAN, the molecule, the analysis becomes more interesting due to the increase in signal strength and the greater resolution of peaks.

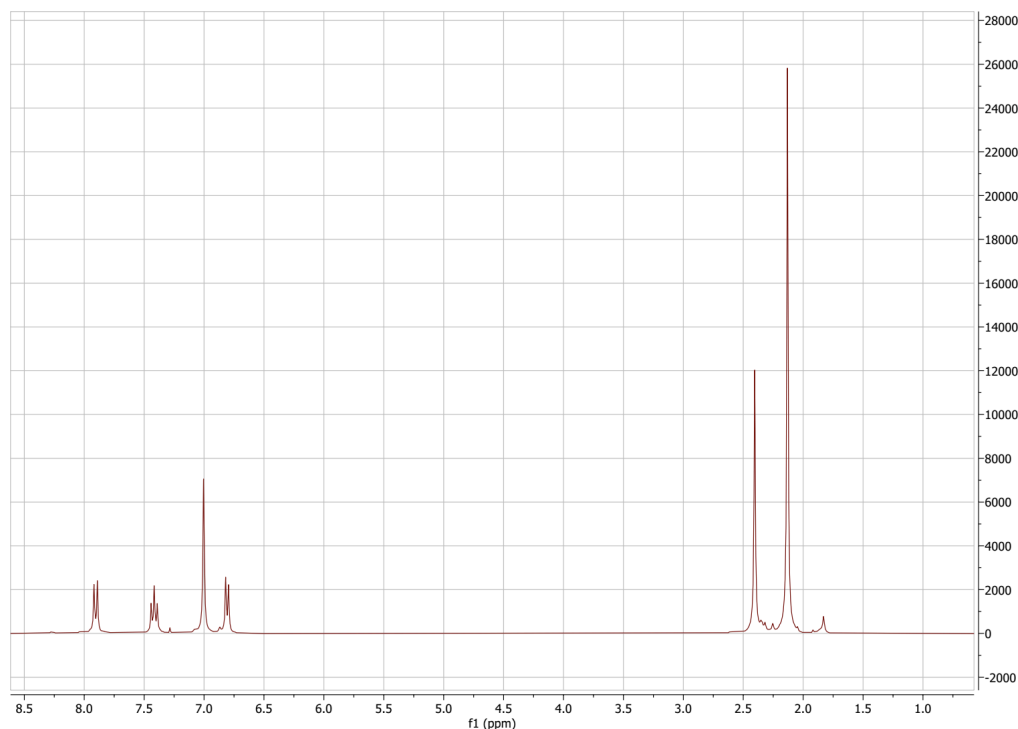


Figure 7

The 300 MHz ¹H NMR spectrum shows better resolution and more defined peaks. Like Dpp-BIAN, you can see how the singlet merging with the first doublet has been resolved in the high-field spectra, indicating that the signals are very close in lower-field spectrometers. Meanwhile, the stronger magnetic field spreads the peaks apart in a high field, clarifying the signals.

Mes-BIAN is a good model compound. Its synthesis illustrates the use of Lewis-acid catalysis and it gives a good ¹H NMR spectrum, which allows for easy identification of signals, splitting patterns, and the possible structural elucidation of the molecule. However, due to the time needed to synthesize the molecule, it isn't the best option for a teaching lab. Enhancing optimization or considering changes in the procedure, such as reducing the scale, could help expedite the overall experiment, particularly the removal of ZnCl₂. We still recommend

conducting the experiment, possibly as a project or independent study exercise as it would require more time, or a student-access to the lab outside of class time.

Dep-BIAN

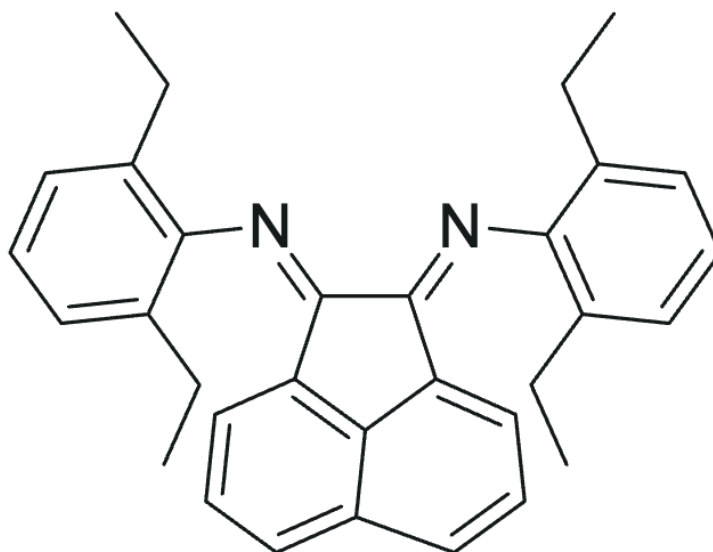


Figure 8

The Dep-BIAN experiment was the last experiment we decided to do and include in this work. Initially, we used the 2,6-diethylaniline to understand which positions on an aniline with side chains would change the physical properties when reacted with glyoxal to form an imine. This experiment sparked the idea of using 2,6-diethylaniline and reacting it with acenaphthenequinone to form an Ar-BIAN. We decided to use our modified Dpp-BIAN procedure to synthesize this molecule. We could barely optimize the experiment(s) because it was the last experiment we decided to add to the lab project. However, the results of our modified Dpp-BIAN experiment looked very promising, so we added it to the lab project.

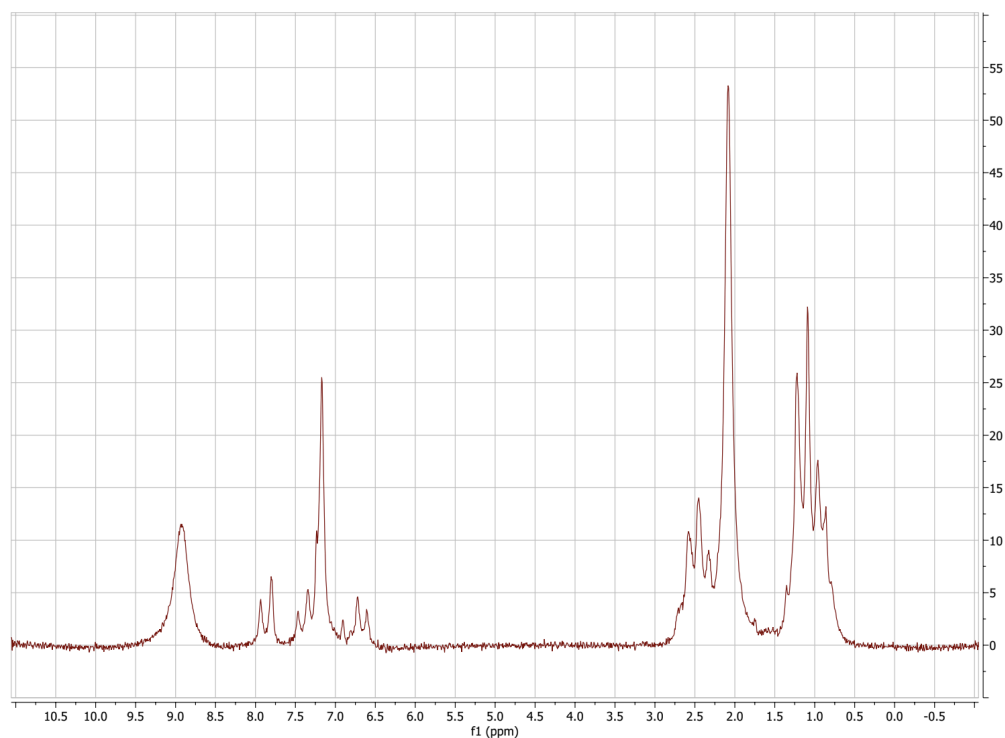


Figure 9

This is a ¹H NMR spectrum of Dep-BIAN, 64 scans, for around five minutes. The spectrum doesn't look as clear and defined as Dpp-BIAN and Mes-BIAN. However, we are still able to see distinct signals and splitting patterns. Just like Dpp-BIAN and Mes-BIAN, Dep-BIAN utilizes the entire ppm range; there are signals in the aromatic and aliphatic regions. The aliphatic region is messy, but we can see glimpses of a typical ethyl splitting pattern. While in the aromatic region, we can see the naphthene moiety, the two doublets, and then the triplet with a shoulder peak off the singlet of the aniline. Due to the noise, poor splitting, and the broad peak at 9.0 ppm, we can say that our Dep-BIAN still has a significant amount of acetic acid

present. This also tells us that more hexane washes are needed to remove the acetic acid. This error and observation indicate that other improvements and refinements could be made with the experiment and the procedure. Nonetheless, this experiment still allows students to learn about imines by synthesizing not-so-common Ar-BIAN, and use NMR spectroscopy to analyze and characterize the molecular structure.

The lab project also includes other experiments using either Lewis-Acid catalysis or Brønsted-Lowry acid-catalysis to synthesize these three α -diimine compounds. By providing these experiments, students can conduct subsequent NMR spectroscopic analysis that reveals distinct spectra for these different imines, reflecting the diverse chemical environments of the nuclei within each molecule. Despite variations in catalytic methodologies, the structural integrity of the bis(imino) compounds is consistently confirmed through identical NMR spectra derived from various catalytic conditions. This comprehensive experimental suite deepens students' understanding of synthetic and analytical techniques, underscoring the robustness of molecular structures across diverse chemical environments.

Using Dpp-BIAN as a representative α -diimine model compound offers a valuable platform for introducing key concepts in organic chemistry, including ligand design, spectroscopic analysis, and structure–property relationships. Mes-BIAN and Dep-BIAN serve as complementary examples, though their syntheses require further optimization to improve reaction time and product yield. Incorporating all three compounds—featuring methyl, ethyl, and isopropyl substituents—into the laboratory curriculum allows students to observe and interpret distinct NMR splitting patterns associated with common alkyl groups. This comparative approach enhances student understanding of proton coupling, symmetry, and the influence of molecular structure on spectral features.

We wanted to create an engaging learning environment that enhances students' understanding and retention of organic chemistry principles by integrating hands-on experiments with theoretical instruction. Ultimately, the goal would be to establish a cohesive program that complements the existing curriculum and fosters students' critical thinking and problem-solving skills.

Sulfanilamide

Introduction/Background

Sulfur-containing drugs, specifically sulfonamides, commonly referred to as sulfa drugs, represent a historically significant and chemically diverse class of pharmaceutical compounds. Sulfa drugs were the earliest antimicrobial agents and the first antibiotics to treat human bacterial infections. Sulfanilamide was one of these early sulfa antibiotics. Sulfanilamide declined due to the emergence of bacterial resistance and adverse hypersensitivity reactions. However, it is still being used as a possible treatment for yeast infections.

Sulfanilamide has the core of the sulfonamides, which consists of a sulfonamide functional group ($-\text{SO}_2\text{NH}_2$) attached to an aromatic ring. The sulfonamide group is connected to an aniline; this structure has modified the scaffold into hundreds of derivatives with various pharmacological activities.

To deepen our understanding of α -diimine compounds, we sought to explore their potential in modeling strategies relevant to drug discovery and pharmaceutical research. To expand the scope of our study, we attempted the condensation of sulfanilamide with acenaphthenequinone to form a novel α -diimine derivative.

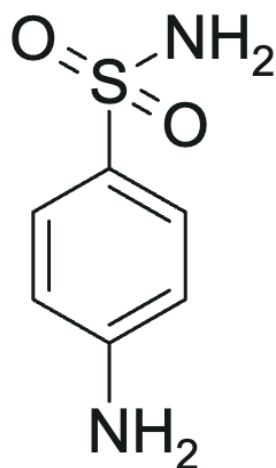


Figure 10

Unlike typical arylamines such as 2,6-diisopropylaniline, 2,4,6-trimethylphenylaniline, and 2,6-diethylaniline, sulfanilamide introduces unique synthetic challenges. These arise primarily from the strongly electron-withdrawing sulfonamide group, which can significantly reduce the nucleophilicity of the amine and hinder imine formation under standard conditions. Through several experiments and many failed experiments, we started to use the Mes-BIAN procedure because of the electron-withdrawing group.

Experimental Overview

In our first session, we measured and added 18 g of acenaphthenequinone, 45.9 g of anhydrous zinc chloride, 13.0 g of sulfanilamide, and 300 mL of acetic acid. Then, we added them to a 500 mL round-bottom flask with a stir bar. The mixture was stirred and refluxed at

80°C for 1 hour. After one hour, the solution should sit for at least 12 hours for recrystallization. We extracted the product using vacuum filtration with a Buchner filter funnel during the second session. First, we washed the product with acetic acid for 10 washes, and then we washed it with diethyl ether until the acetic acid smell was faint. Using the Buchner filter funnel to pass air through the intermediate, we allowed it to dry.

During the next session, we ground the solid in a mortar and pestle and then weighed it to get the yield. After gathering the yield, we measured 12 g of the zinc Sulf-BIAN intermediate, 100 g of K₂CO₃, and 250 mL of water. Then, we added them to a 500 mL round-bottom flask with a stirrer. We heated and refluxed the mixture for 3 hours at 100 degrees Celsius. After heating, the product was left to cool for at least 24 hours.

Next, we used gravity filtration to remove zinc carbonate, excess potassium carbonate, and water. The product was left to dry overnight. The next day, we boiled 300 mL of THF in a 500 mL round-bottom flask, added the dried product to the boiling THF, and waited until the solid in the boiling solution fully dissolved. Once fully dissolved, while still warm, we poured the solution through gravity filtration. We kept the ethanol solution; the solid ZnCl₂ was removed. We then placed the THF solution in a flask and put it under a fume hood to evaporate to about a quarter of its original volume, taking around 2-3 days. However, when we returned around two and a half days later, there was no THF, and crystals had formed (the whole procedure will be included in the supplemental materials). We removed the crystals from the flask, ground them in a mortar and pestle, and then weighed the product to determine the yield. We prepared an NMR sample by adding 0.05 g of Mes-BIAN to an NMR tube and then adding 0.750 mL of Deuterated DMSO. After making our sample, we ran a 64-scan ¹H NMR for around five minutes and saved the spectra.

Results and Discussion

To clarify, we could not obtain the IR spectrum of the Sulf-BIAN because our IR spectrometer was not working correctly. We plan to obtain an IR spectrum of this compound sometime in the future.

The Sulfanilamide BIAN derivative is very interesting. We initially tried to do the reaction using Brønsted-Lowry acid-catalysis; however, due to the presence of the sulfonamide functional group, the reaction would not work as desired, yielding mixtures of different products instead. When we did a ^1H NMR of the product, we got a lot of noise and random signals.

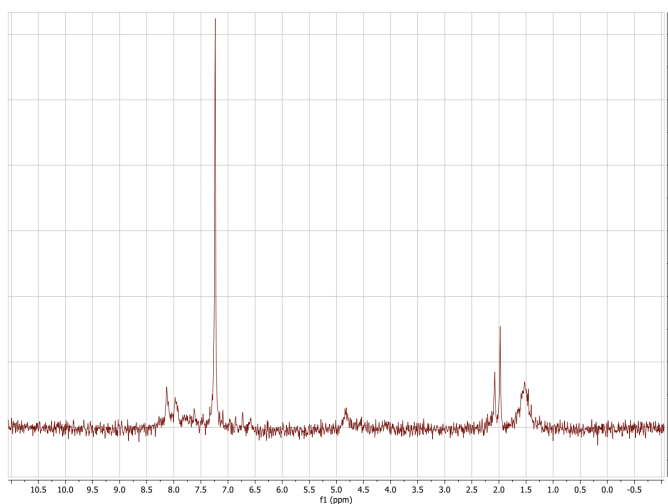


Figure 11

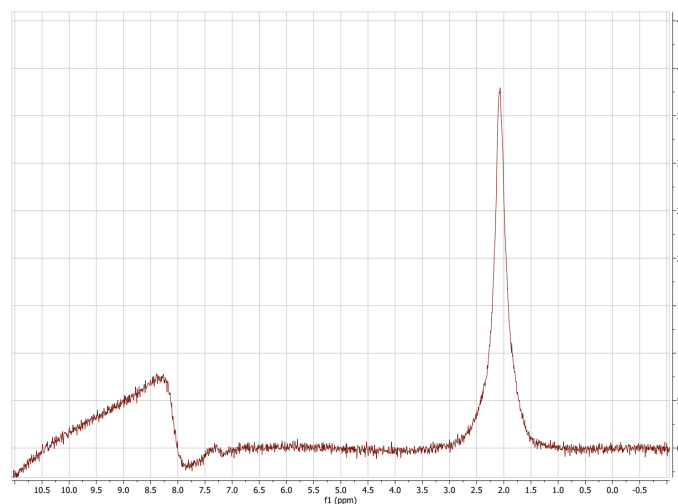


Figure 12

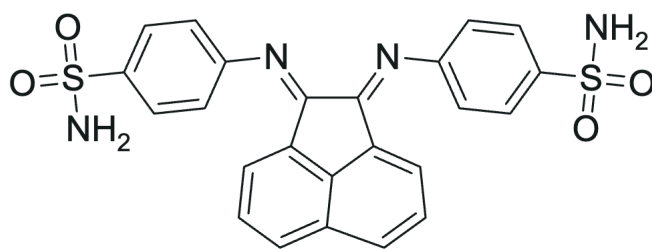


Figure 12

Repetition of the same experiment would give product mixtures with different signals in their NMR spectra. Moreover, the signals we were expecting were not present in these NMR spectra. Therefore, we decided to use the Lewis-acid catalyzed reaction, using the same procedure as used for the Mes-BIAN experiment. The first reaction of synthesizing the zinc intermediate went well; we created a solid, hopefully stable product. Using deuterated DMSO as the NMR solvent, we obtained the ^1H NMR spectrum shown below:

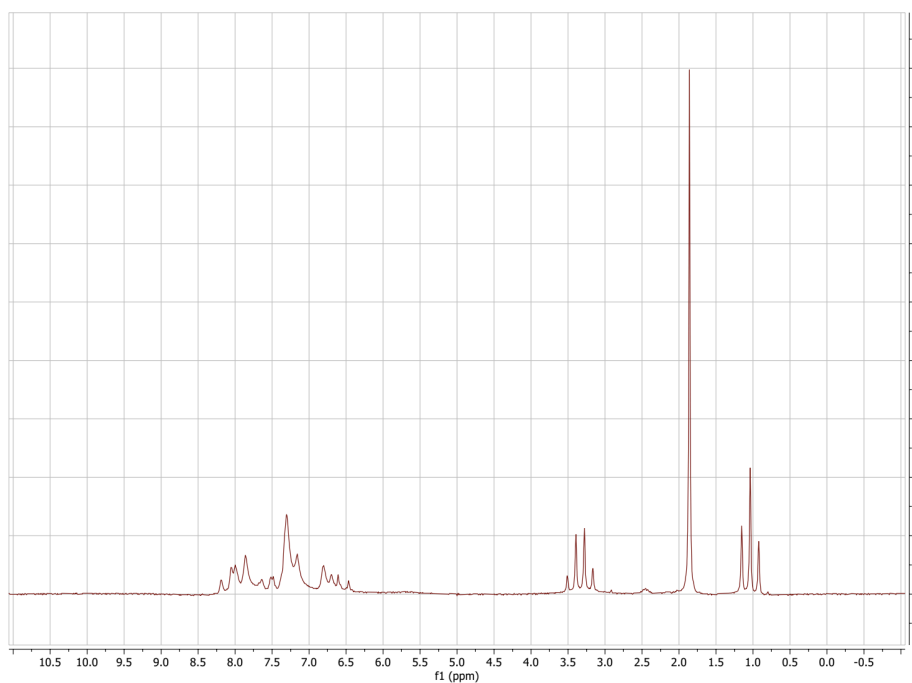


Figure 13

This ^1H NMR resembles the spectrum expected for sulfanilamide BIAN more closely. If we ignore the triplet and quartet from the excess diethyl ether, we observe a single singlet signal

in the aliphatic region. In the aromatic region, several signals appear without notable splitting patterns. The singlet signal originates from the amine group on the sulfonamide functional group, while other hydrogens are not bonded to a ring, meaning the remaining signals are all in the aromatic region. According to the molecule's diagram, we should see a singlet in the aliphatic region from the amine group, followed by two doublets from the hydrogens bonded to the sulfanilamide, and then a doublet, triplet, doublet from the naphthene moiety. As mentioned before, we see the amine peak, while all the signals overlap in the aromatic region.

In addition to the normal ^1H NMR, we did a D_2O ^1H NMR experiment to see if the amine peak would disappear or diminish due to deuterium exchange. However, while

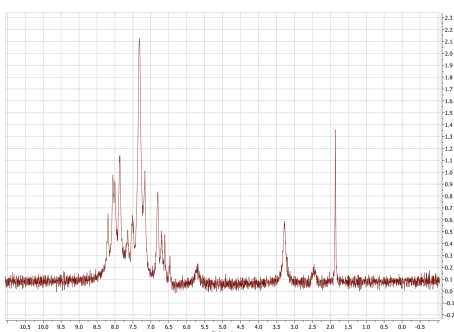


Figure 14

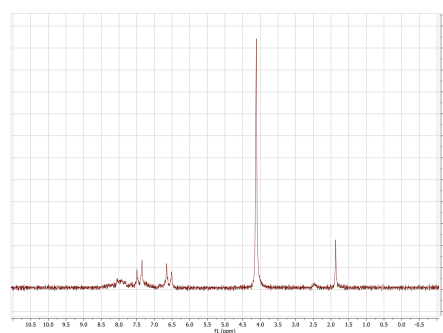


Figure 15

experimenting, the amine signal wasn't diminishing as expected. Instead, it appeared that the entire molecule was breaking apart. We determined that the addition of deuterium oxide (a form of water) was reversing the imine-formation reaction to reform the sulfanilamide and acenaphthenequinone starting materials.

As you can see, the NMR signals have a significant change. The control spectrum has also changed from the initial spectrum, indicating that with the evaporation of diethyl ether. Also, the spectrum shows that the molecule interacts with the water, with a water peak that is slowly growing. The diagram on the left shows 0 mL of D_2O and features several signals in the aromatic region. However, after adding 3 mL, the spectrum on the right reveals that all the peaks

drastically crash while the water peak is taller than before. The spectrum on the right is the sulfanilamide ^1H NMR spectrum, but with a water peak.



Figure 16

This image shows the NMR tube after we added 3 mL of D₂O to the Sulfanilamide BIAN. We can see that the acenaphthene is not soluble in water, so it just sinks to the bottom. In contrast, sulfanilamide is soluble in water and other common solvents. Even though the experiment didn't go as we expected, it created a model of how azo-dyes and sulfanilamide

would interact when treated in the 1930s. Since the azo-dyes were not soluble in water, they would just pass through the digestive system. However, when ingested, the azo-dyes hydrolysed to form the water-soluble sulfanilamide which was absorbed into the bloodstream.

In addition, this experiment also modeled how, when pharmaceutical companies develop drugs, they use different ligands to add to an existing medicine so they can attach other active ingredients to make the medication treat more symptoms.

Conclusion and Future Work

Conclusion

The outcome of this project has profoundly deepened my understanding of imines, their formation mechanisms, and the principles of NMR spectroscopy, while also intimately connecting me with concepts relevant to medicine and drug development. This lab project is meticulously designed to engage students actively in the synthesis and analysis of imines, utilizing NMR spectroscopy as a key analytical tool.

Among the various experiments, the acid-catalyzed synthesis of Dpp-BIAN stands out as the most effective. This particular reaction offers an exemplary molecular model for educational purposes. The process is not only safe and straightforward but also time-efficient, resulting in a stable, solid product that students can characterize using NMR techniques. Dpp-BIAN features an intricate molecular structure that encompasses both aliphatic and aromatic proton environments, along with significant steric hindrance surrounding a sigma bond. This complexity empowers students to employ a wide array of NMR analytical tools, thereby developing essential skills in interpreting various chemical environments, recognizing splitting patterns in the spectra, and effectively correlating molecular structure with spectroscopic data.

Another experiment, the synthesis of Mes-BIAN, proves to be a valuable addition to the lab project. While it necessitates a longer reaction time due to the use of Lewis-acid catalysis, it affords students exposure to a distinct pathway for imine formation. This alternative approach not only broadens their comprehension of reaction mechanisms but also enhances their insights into diverse synthetic strategies. Furthermore, optimizing the Mes-BIAN experiment to improve its reaction time and yield could significantly enhance its effectiveness. Achieving higher yields would instill greater confidence and engagement among students, as they would be able to witness more tangible, successful outcomes during their analyses. This, in turn, would enrich the overall educational experience. Although shorter experiments may be more practical for teaching labs, incorporating molecules with different substituted side groups offers an additional benefit: it allows students to recognize and analyze varied analytical patterns in NMR spectroscopy. This comprehensive approach ultimately fosters a more robust understanding of the theoretical and practical aspects of imine chemistry and analytical techniques.

Lastly, Dep-BIAN represents an innovative experimental approach designed specifically for undergraduate students, allowing them to work with ethyl groups substituted at the 2- and 6-positions of the molecular framework instead of using isopropyl groups. By opting for ethyl substituents, which are less sterically hindered compared to isopropyl counterparts, this variant creates a more accessible and manageable system for students in laboratory settings, all while preserving the fundamental structural integrity required for effective experimentation. While the Brønsted-Lowry acid-catalyzed reaction has shown promising results, further investigation is necessary to determine whether employing a Lewis-acid-catalyzed reaction might yield higher efficiency. This exploration could also focus on maintaining the brevity and simplicity of the experiment, which is paramount in an educational environment tailored for

undergraduates. By optimizing the reaction conditions, educators can enhance the learning experience while ensuring that students achieve meaningful results in a timely manner.

Producing what is believed to be a novel compound was exciting. It gave us insight into creating and researching novel compounds, as in pharmaceutical research. Having failed and adapting your experiment just to figure out what was wrong was a very beneficial learning experience.

Future Work

For the lab projectatory , we need to finalize the manuscript, ensuring that it includes a comprehensive introduction, methodology, results, and discussion sections. Additionally, we should focus on polishing the supplemental materials, which may include detailed data sets, graphs, charts, and any relevant additional information that supports our findings. It's crucial to review all figures and citations for accuracy and clarity before submission.

Some recommendations we received at the ACS Spring National Conference included researching how effective the lab experiments are in teaching students about the different materials. We can create surveys or questionnaires to see how different students feel about their understanding of the various topics. Then, after the lab, we can give the same survey or questionnaire to see if there is any difference. Another recommendation received at the ACS Spring National Conference is to implement high-field NMR spectrometers and spectroscopy a bit more to give students a better representation of what is going on in industry and academia.

In the context of the Dpp-BIAN experiment, further investigations into the consistency and reliability of the yield when implementing our modified procedure are essential. A deeper

understanding of these factors will provide valuable insights into the efficacy of our methods and help optimize outcomes.

For the Mes-BIAN experiment, more experiments need to be conducted to test different procedures. By doing more experiments, we will be able to optimize the experiment more to see what can be changed and what should be kept the same. All we need to do is do the experiments, analyze the results, and make observations. Similarly, for Dep-BIAN, we need to do the experiments on both acid-catalyzed and Lewis-acid-catalyzed reactions to see which experiment produces the best results in a reasonable amount of time.

Lastly, regarding Sulfanilamide BIAN, it would be beneficial to conduct comprehensive research into its efficacy as an antibiotic. This could involve reviewing historical data on its effectiveness, examining current scientific literature, and potentially undertaking new clinical trials to assess its therapeutic outcomes. Given the evolution of bacterial resistance over the years, understanding whether Sulfanilamide still maintains its potency against various pathogens is crucial. Such research could not only reaffirm its role in modern medicine but also guide clinical decisions and inform patient treatment plans. Moreover, other research can be done on compounds that could form imines, such as curcumin, and see if any structural changes affect the compounds' antioxidant, anti-inflammatory, and anti-protein-aggregating abilities.

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