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Research-Focused Approach for Introducing Undergraduate Students to Aromatic Organic Synthesis at a Community College

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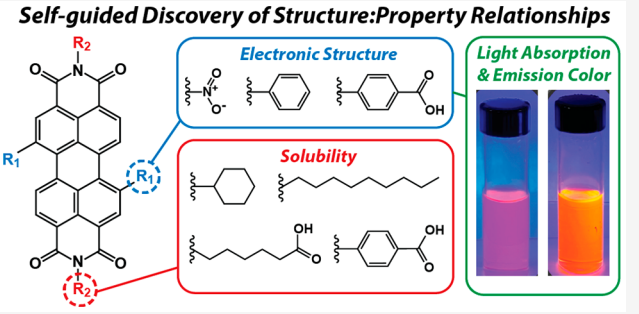
ABSTRACT: The addition of research-focused experiences to undergraduate chemistry laboratory courses has been shown to bolster student learning, enhance student retention in STEM, and improve student self-identity as scientists. In the area of synthetic organic chemistry, the preparation of libraries of compounds with novel optical and electronic properties can provide a natural motivational goal for research-focused exercises that can be undertaken by individual students or collectively as a class. However, integrating such experiences into a community college teaching laboratory setting can face challenges imposed by the cost of supplies, limited laboratory space, and access to characterization facilities. To address these challenges, we have devised a sequence of inquiry-driven, research-focused laboratory exercises that can be readily integrated into an organic chemistry laboratory course with minimal cost. This sequence consists of a multistep synthesis of perylenediimide dyes that introduces students to advanced synthetic techniques, such as organometallic coupling reactions, column purification, and reactions performed under inert atmosphere. This high-yield, three-part synthesis can be easily varied by individual students or small groups within a class to form a broad library of compounds with potential utility for applications in light harvesting, molecular electronics, catalysis, and medicine. We describe the design of low-cost workstations for chemical synthesis under inert atmosphere and provide auxiliary lesson plans that can be used to expand the scope of a laboratory course beyond synthetic organic chemistry by introducing students to concepts in molecular spectroscopy.



KEYWORDS: *Second-Year Undergraduate, Organic Chemistry, Laboratory Instruction, Inquiry-Based/Discovery Learning, Aromatic Compounds, Dyes/Pigments, Synthesis*

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INTRODUCTION

Course-based undergraduate research experiences (CUREs) hold several benefits over nonresearch-based methods for undergraduate scientific instruction as they have been shown to improve student learning outcomes, enhance student retention in STEM degree programs, and encourage students to self-identify as scientists.[1−5] CUREs aid students in building key problem solving skills, develop their ability to work collectively in groups, and foster the creative application of core scientific concepts to real-world problems.[6] Unlike other research opportunities open to undergraduates, such as industrial research internships or volunteer work in academic laboratories, CUREs do not require students to dedicate time to research outside of a classroom setting. This makes CUREs particularly effective at engaging students from nontraditional backgrounds who are subject to financial or familial obligations that limit their interaction with science to classes within their degree program.

In the area of synthetic organic chemistry, research to form libraries of new compounds offers an attractive topic for CUREs.[7−11] Given the flexibility of chemical synthesis, organic compounds can exhibit a range of chemical behaviors that stem from small variations in starting material and functionalization paths, which can allow students to propose and test hypotheses for creating compounds with a desired set of properties. Within the framework of building a chemical library, individual students or small groups can each work to design, prepare, and characterize a unique set of compounds. As each student group produces a unique compound, this can foster student ownership of achievement and self-confidence that reinforce their propensity to self-identify as scientists. The preparation of a library of materials in the absence of predefined synthetic pathways can also be used to teach students skills relating to literature searching, reading papers, and developing procedures.

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However, implementing research-focused experiences, like those found in CUREs, in laboratory courses at a community college poses several challenges. Courses can be constrained by budgetary concerns that can limit the scope of compounds that can be explored due to the need for expensive glassware, instrumentation, and starting materials. The teaching laboratory space within a community college may also be shared among multiple sections of different courses, making it difficult to setup complicated glassware or synthetic equipment needed for long-term, advanced reactions. Moreover, access to characterization facilities for mass spectrometry (MS) and nuclear magnetic resonance (NMR) that can assess the quality and structure of synthesized compounds can be limited at a community college.

To address these concerns, we have developed an inquiry-based laboratory course focused on building a library of perylenediimide (PDI) dyes (Figure 1), stable chemical compounds with a long history of use as industrial inks and pigments due to their high degree of coloration and photostability.[12−15] These materials have remained at the forefront of ongoing research efforts due to their potential use as photocatalysts,[16−18] photodynamic therapy agents,[19−21] and light-harvesting components of high-efficiency solar cells that employ singlet fission.[22−27] PDI syntheses include several key organic transformations that make them attractive instructional tools for introducing students to retrosynthetic analysis and methods for general organic reactions, such as acid anhydride-to-imide conversion, electrophilic aromatic substitution, and metal-catalyzed couplings. Important for instructional classrooms, PDI syntheses can give highly colored, air-stable, solid products in high yield from low-cost, commercially available anhydride, amine, Suzuki catalyst, and boronic acid starting materials. As deeply colored, fluorescent dyes, PDI reaction products separated by column chromatography can be readily screened and identified via anticipated colorimetric changes.

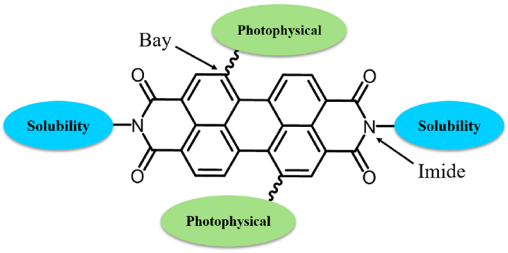


Figure 1. PDI dye with points of functionalization.

Moreover, as the electronic structure of PDIs is set by their chemical structure, synthesis of distinct PDI dyes allows students to connect how their structure affects the colors of light they absorb and emit. Advanced structural analysis tools, such as NMR, MS, and infrared spectroscopy, can further enhance a course by allowing students to determine the structure of novel PDIs.

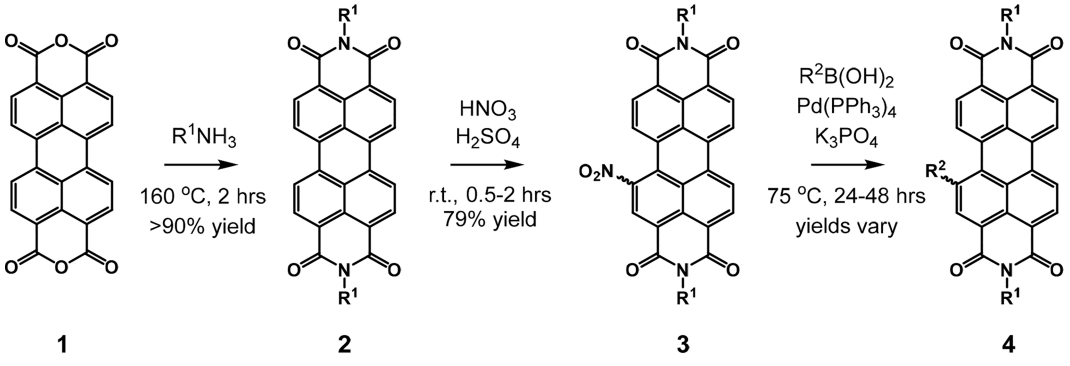
In this report, we describe a research-focused laboratory course developed at Austin Community College (ACC) that resulted in the synthesis of a broad array of PDIs with distinct functional groups. This course can be easily extended to produce an expansive PDI library that contains several novel compounds. We present a modular, three-step synthetic pathway (Scheme 1) that undergraduate students in the course's initial cohort used to create PDIs featuring different chemical substitutions. We describe the general reaction scheme, starting materials, reagents, and general yields for compounds synthesized by students. We also detail low-cost, mobile glassware workstations that allow reactions to be conducted under inert atmosphere within an undergraduate teaching lab. These workstations can be readily implemented in teaching facilities shared among several classes that otherwise preclude use of stationary setups for inert atmosphere chemistry, such as Schlenk lines and in-house nitrogen or argon gas lines. We provide lesson plans for how these reactions can be adapted to explore how PDI molecular properties, such as their coloration, solubility, and emission quantum yield, change as a function of molecular structure. This course provides a basis for a laboratory class that goes beyond the prescriptive lab manuals and teaching approaches endemic to traditional community college lab courses by allowing hypothesis-based chemical research to be undertaken by undergraduate students.

METHODS

General Procedures

Conversion of Perylenetetracarboxylic Dianhydride (PTCDA) to Nitrogen-Substituted Imides. PTCDA, a bright red powdery solid, was suspended in a large excess of commercially available, reasonably priced amines. The suspension was refluxed for 2 h, and most PTCDA dissolved with heat and the color deepened as the reaction progressed. As the heat was removed, a precipitate formed and was filtered once cool. The deep red-to-purple precipitates were dried in a hood for several days or warmed in an oven for an hour. The Supporting Information details variations in this general method used to create PDIs with specific amine substitutions.

Scheme 1. General Synthetic Pathway Used for PDI Library Creation



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J. Chem. Educ. XXXX, XXX, XXX−XXX

Nitration of PDI Bay Positions. PDIs were dissolved in dichloromethane (DCM) and cooled to 0 °C using an ice bath. Equal parts nitric acid and sulfuric acid were slowly and carefully mixed in a separate beaker and then added dropwise to the cooled PDI solution. Following the acid mixture addition, the ice bath was removed, and the reaction stirred for 30−90 min. Varying the reaction times gave single versus double nitration of the PDI. The single and double addition products could be separated using silica gel flash chromatography, eluting with DCM and chloroform ($CHCl_3$).

Addition of Aryl Groups to PDI Bay Positions via Suzuki Coupling. PDIs prepared with nitro groups at their bay positions were combined with distinct substituted arylboronic acids and palladium catalyst in tetrahydrofuran (THF). The THF was stored over sieves and not dried further prior to use. The reactions were heated to 75 °C and typically required 48−72 h of reaction time. The THF was evaporated, and the residual crude was purified by flash chromatography with $CHCl_3$. The products tended to be dark red-maroon solids that fluoresce when dissolved in $CHCl_3$, which made the columns easy to monitor with a UV blacklight.

RESULTS

PDI dyes were identified as a platform for chemical exploration due to their chemical stability, ease of modular chemical functionalization, high degree of coloration and luminescence, and low cost of starting materials.[14,15] Students participating in our course worked with instructors to develop synthetic approaches that could be readily varied to allow small, independent groups of students to prepare chemically distinct PDIs. To ensure students received training in key organic synthesis techniques, instructors guided students toward approaches involving reactions commonly taught in introductory organic chemistry courses, such as electrophilic aromatic synthesis, organometallic coupling reactions, and amide/imide formation. The benefits of these reactions in a large classroom setting are chiefly their relative ease and speed, as well as their usage in modern chemistry lab settings.

Class workflow started with students choosing imide- and bay-substitutions they wanted to achieve following a short discussion and literature search aimed at having students uncover how different functional groups impact PDI properties. Both the PDI bay and imide positions were identified as key points for chemical attachment as they allow for exploration of complementary PDI properties (Figure 1). Functional groups added to a PDI's bay positions can directly interact with the molecule's central π-system.[14,15] As such, placement of electron donating/withdrawing groups at these positions was used to explore their influence on the PDI HOMO/LUMO energy gap, as evidenced via changes in absorption and emission line shapes. In contrast, functional groups attached to the imide positions negligibly impact PDI spectroscopic properties as the PDI HOMO and LUMO both contain nodes at these points.[14,15] Rather, functional groups placed at the imide positions alter PDI solubility. Imide substitution was used by students to explore how different functional groups impart solubility by, for example, adding branched vs linear alkyl chains of different lengths, rigid vs flexible rings, and groups of differing polarity (Figure 2 and Table 1).

To identify viable synthetic routes for PDI preparation, students conducted independent literature reviews to find procedures that would support their synthetic goals. Literature

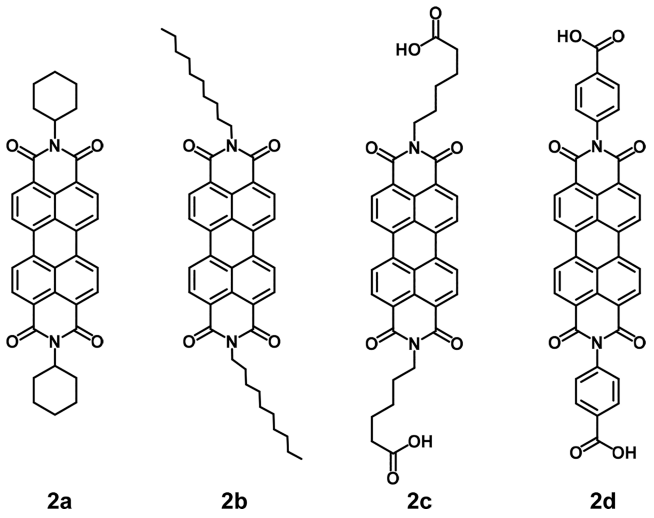

Figure 2. Representative imide substitutions employed by undergraduates to explore PDI solubility.

Table 1. PDI Solubility in Distinct Solvents (*n* = insoluble; *y* = soluble)[a]

sample	toluene	THF	DCM	$CHCl_3$	DMSO	MeCN
1	n	n	n	n	y	n
2a	n	n	y	y	y	n
2b	n	n	y	y	y	n
2c	n	n	n	n	y (slightly)	n
2d	n	n	n	n	y (slightly)	n

[a]Functionalization at both the imide and bay positions can be used to alter solubility. Solubility was assessed by adding 0.01 mmol (∼5 mg) of each PDI to 10 mL of solvent and noting visual dissolution of PDI powder. We note this assessment can be made more quantitative by using UV−vis absorption spectroscopy to measure absorption spectra of PDI solutions as PDI aggregates are known to show distinct changes in their vibronic absorption profiles upon aggregation.[28−31].

searches through SciFinder were performed using both keywords and structure searches. The large number of synthetic routes students found suggested a range of reactions that could lead to successful PDI synthesis. To implement these syntheses, solutions were developed to address three key challenges: (1) creation of workstations for performing chemistry under inert atmosphere, (2) allowing for prolonged heating times of reaction flasks in a shared laboratory setting, and (3) use of specialty starting materials followed by purification of products via silica gel chromatography.

To address the first of these challenges, performing chemistry under inert atmosphere, we devised mobile workstations that could be easily moved into and out of fume hoods (Figure 3). Unlike permanent fixtures for inert atmosphere chemistry, such as Schlenk lines, the mobile nature of these workstations allows them to be easily removed from fume hoods and stored at the end of a laboratory period, which can allow benchtops and fume hoods in teaching laboratory space to be used by multiple classes throughout the day. Our mobile workstation design employs common laboratory parts, such as round-bottom flasks, 19/24 connectors, condensers, and rubber lines taken from a standard organic glassware kit. The inert atmosphere setup uses a doubled balloon Ar air source, which students were able to fill from a single Ar tank using inexpensive gas adaptors. A single Schlenk adapter piece (a three-neck valve or Flushing Adapter) is added to the setup

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J. Chem. Educ. XXXX, XXX, XXX−XXX

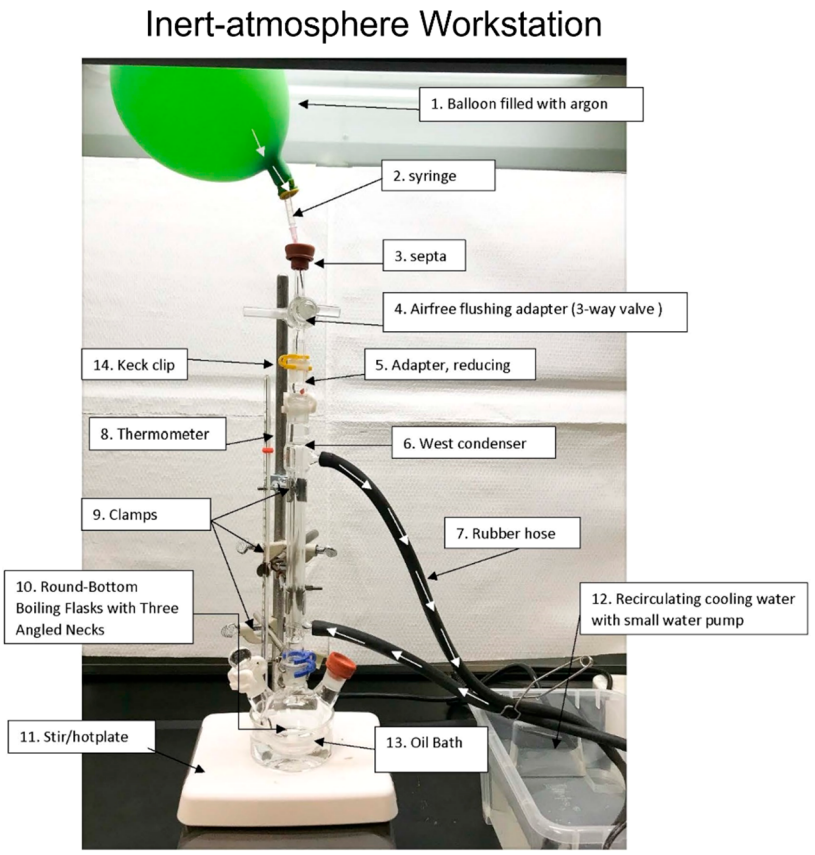

Figure 3. Representative mobile workstation employed by students for organic synthesis under inert atmosphere conditions.

to allow for use of vacuum and inert argon gas to be introduced to the vessel without the need for a large Schlenk manifold. A standard Bunsen burner was used to flame-dry the setup under vacuum applied by house vacuum lines, which yielded dry glassware within about 10−20 s of applied high heat. After cooling to room temperature, the glassware was filled with Argon by turning the Schlenk valve to the argon balloon gas source. Upon completion of these measures, the workstation is self-contained and can be moved. This can allow laboratories with limited fume hood space or gas lines to move mobile stations to an alternative benchtop for synthesis.

The overall cost of each mobile workstation is ∼$150, which is more economical than Schlenk manifolds, which can cost thousands of dollars. Indeed, the low cost of these workstations allowed us to provide each student in a class of 16 with their own independent workstation. In addition, the physical footprint of each workstation is small (2 sq ft), which allowed pairs of students to work side-by-side in 4′ wide hoods. Importantly, reactions carried out using these workstations showed product purities (+90% yields) that paralleled reaction yields obtained using other inert atmosphere methods.

The second challenge, achieving prolonged heating times and extended reflux of materials, was safely addressed by developing a mobile, water recycling pump system that was employed in conjunction with a silicon oil heating bath (Figure 3). Like the inert atmosphere workstations, materials for the condenser portion of the mobile workstation were taken from a standard organic glassware kit ($389). This setup uses a small, inexpensive water pump (Homasy 80 GPH [300L/H, 4W] Submersible Water Pump - $8.99) to recirculate the water within the condenser, which is ideal for teaching laboratories with limited fume hoods or that lack cooling water lines. Rubber hoses were used to connect the condenser to the water pump, which in turn was immersed in a plastic 4 qt Tupperware water reservoir. We found that students sharing a fume hood could share water sources, reducing clutter, the potential for flooding, and equipment costs. The use of a thermometer within the oil bath to tune the reflux temperature allowed us to streamline the inert atmosphere glassware setup and permitted students to practice safe heating procedures. Typically, introducing a thermometer into a three-neck flask held under an inert atmosphere engenders some risk as layers of parafilm and stoppers are typically used to create an airtight seal around the neck of the reaction flask, yet such seals can fail if not properly created by novice students. In contrast, a thermometer within the oil bath eliminates the need for these sealing steps and provides comparable control over the temperature of reaction mixtures.

The third challenge, that of costly starting materials and expensive, time-consuming purification, was addressed via use of a streamlined, three-step synthetic strategy that could be easily modified to afford several distinct PDI compounds (Scheme 1). To prepare imide-functionalized PDIs, a low-cost reagent, PTCDA, was reacted with amines to yield products of reasonably high purity (>90%). These high yields were obtained by dissolving PTCDA in an amine selected for N-attachment, meaning that this amine served as both the solvent for the reaction as well as its sole reagent. By using this "flood" of amine together with high heat, N-attachment progressed quickly, within 2 h, allowing students to complete this reaction in a single lab period. Reaction progress can be tracked by fluorescence and coloration changes. Within 30 min of the N-

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J. Chem. Educ. XXXX, XXX, XXX−XXX

functionalization step, the reaction flask can be observed to give off yellow and green fluorescence when illuminated by a UV flashlight. Due to the high yield of the N-attachment reactions, the filtered crude product from this step can be directly reacted in subsequent steps to yield further functionalized PDIs.

To prepare PDIs for organometallic coupling, a nitro group was substituted at their bay position. Though less common than using a halogenated intermediate for coupling, Suzuki couplings via these nitroarenes are well-researched.[32−35] Halogenation via liquid bromine yielded single, double, triple, and quadruple substituted products that were very difficult to separate. In contrast, reaction with a nitro group was found to yield only single and double substituted products whose production could be easily monitored by thin layer chromatography (TLC). Nitration was also found to complete in 30−90 min, less time than halogenation reactions, and has the added benefit of involving less-toxic reagents.

Purification of core-substituted mono- and double-NO_2 PDIs was accomplished within a single column, which gave distinct bands using DCM. Verified by TLC, the fluorescent bands are separated by flash chromatography and the purified products were concentrated all within 2 h, the time of a typical lab class period. Dried and purified products **3a** and **3b** were used to perform organometallic coupling via a Suzuki reaction in a subsequent class period. Students completed the setup for Suzuki coupling within a few hours and the reaction was allowed to progress for 48 h. Space for this reaction to progress is minimal and three to four reactions can be allowed to run in a single hood over a weekend. The final workup for the third product is a multistep process that varies in detail for different synthesized PDIs (see the Supporting Information for details). These variations challenge students to consider the solubility, polarity, and phase selectivity of their synthesized product in designing schemes for its purification.

Following each key step in the synthesis of their chosen PDI dyes, students characterized their synthesized compounds using a combination of UV−visible absorption, fluorescence, and FT-IR spectroscopy. Representative spectra obtained by students are highlighted in the Supporting Information. Students began obtaining spectra in the early weeks of the course, beginning with the starting material, PCTDA, and their amines of choice for the O-to-N functionalization step. This gave students the opportunity to familiarize themselves with a compact absorption/emission spectrometer used for data acquisition (Vernier fluorescence/UV−vis spectrophotometer, $2999). In the first 4 weeks of the course, a focus was placed on introducing students to spectroscopic equipment and data analysis by having students produce graphs of spectra of starting materials using spreadsheet software such as Excel or Google Sheets. In the second 4 weeks of the course, these skills were cemented through assigned mini laboratories. Students were asked to test the solubility of their products in several solvents and UV−vis absorption spectra were taken of these products in each solvent to assess aggregation and interrogate solvatochromic shifts in absorption and emission spectra.

Infrared spectra of samples embedded in solid KBr discs formed under pressure were recorded using a 500 model Buck Scientific IR spectrophotometer. Students analyzed spectral features of each starting material and product and matched those peaks to standard signals presented by common organic functional group. Prior to measuring spectra, students were asked to predict the spectra of starting materials and synthesized products using their knowledge of infrared absorption and spectra found in the literature. Students then made comparisons between their predicted and measured spectra, analyzing the experimental data for emerging functional group peaks and impurities. Any impurities or unexpected results served as a learning point for students. These teachable moments allowed students to troubleshoot their synthesis, question their own peak assignments and make revisions, and/or consider rerunning spectra to validate their conclusions. Infrared analysis was found to be particularly useful in monitoring O-to-N functionalization reactions as they give notable changes in IR spectra, in particular the movement of stretches in the carbonyl region from 1725−1780 to 1640−1700 cm^{-1} upon N-functionalization and the appearance of C−H stretching transitions associated with functional groups added to the imide position near 2800−3100 cm^{-1} (see the Supporting Information).

Utilizing facilities at the University of Texas at Austin (UT), ACC students were able to characterize reaction products using ^1H NMR and ^{13}C NMR as well as MS. NMR facilities at UT were available for students to run ^1H NMR and ^{13}C NMR, with 400−600 mHz Varian spectrometers. Many students could use the same small bottle of deuterated $CHCl_3$ for preparing samples, thereby reducing cost. NMR tubes were recycled after each submission, which further reduced the overhead cost of sample submission. The NMR facilities manager sent students their data via email within a few days of sample submission. Reading and interpreting NMR data was done using UT licensed Mnova software on students' personal computers or using a shared laboratory laptop. MS was performed using electrospray ionization in negative ion mode (ESI−). Students prepared samples in autosampler vials (Waters Clear 12 × 32 mm^2 screw neck auto sampler vial) that were then submitted to UT's MS facility. Measurements were performed by a facility attendant and students received mass reports and full ion chromatographs of their samples for confirmation of products and purity within 48−72 h. We note that if access to NMR or MS facilities is limited, these sections of the course can be omitted. As PDIs are highly colored and fluorescent, UV−vis and emission spectroscopy can be used to confirm successful synthesis of the central PDI chromophore while infrared absorption spectroscopy can be used to infer functional group transformations.

■ DISCUSSION AND OUTCOMES

The curriculum we report was developed and implemented at ACC in spring 2019 as a second-semester honors organic chemistry laboratory course, Chemistry Honors 2125. This class met for a 3 h lab period once each week over a 16 week semester. Data presented below was compiled over two student cohorts (spring 2019 and spring 2020), during which 17 student participants successfully synthesized more than 25 unique PDI products (Scheme 2). Our course was organized into four units that each emphasized a distinct set of learning objectives: (1) Scientific Reasoning and Independent Inquiry, (2) General Laboratory Techniques, (3) Organic Synthesis, and (4) Chemical Characterization. Each unit lasted roughly 4 weeks and featured distinct sets of in-class and out-of-class activities designed to help students develop specific skills relating to the course's objectives (Figure 4).

In the first four week unit, "Scientific Reasoning & Independent Inquiry", students focused on understanding the scientific motivation for the potential uses of PDIs, learned

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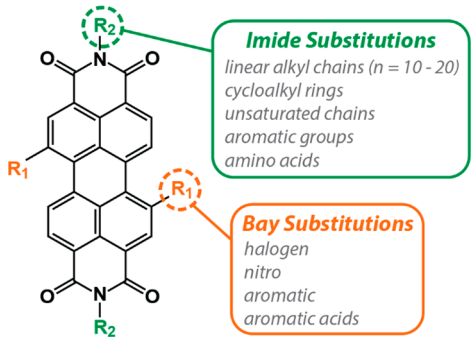
J. Chem. Educ. XXXX, XXX, XXX−XXX

Scheme 2. PDI Substitution Patterns Explored by Honors 2025 Students



how to read scientific literature and access online resources, and were introduced to scientific notetaking, with an emphasis placed on reporting out the results of their research work. Central to the course's success was teaching students how to read, access, and understand scientific articles. Students were given reference articles[14,15,26] with background about PDIs and their uses in energy harvesting applications as well as synthetic details for their preparation during the first day of class. These articles were later dissected by the class during discussion sessions that were designed to work through the key scientific principles presented in the articles and to demystify commonly used jargon. Student learning was assessed using short quizzes structured around concepts contained in these articles. Students also received instruction in how to access and search online databases such as SciFinder and Reaxys. Following their introduction to online databases, students were given literature search challenges to assess their ability to employ these tools. Students were given a mixture of known and novel compounds and were asked to find background literature on these compounds. For known compounds, students were tasked with identifying reported synthetic routes, relevant spectra, and commercial availability and cost of reagents for their preparation.

In parallel with these activities, students also received training in creating a digital scientific notebook, which functioned as a living document that could be accessed by each member of the class. Notebooks were expected to contain a detailed record of all reaction conditions, experimental notes, yields, and relevant photos. The known compounds assigned for database searches outlined above were also used to teach students how to convert preparative methods reported in the literature into stepwise procedures that could be performed in lab. Using these reports as a guide, students were asked to propose in their notebooks a step-by-step procedure that could be used at ACC for their preparation and purification. These procedures were shared with fellow members of the class who were expected to provide critical feedback on safety and feasibility. Following revision, these procedures were evaluated by course instructors.

The second four week unit focused on introducing students to "General Laboratory Techniques" that they would need to successfully setup, manage, and characterize chemical reactions to be performed over the duration of the course. Each week, one or more new synthetic, purification, or characterization techniques were presented through mini-laboratories and class demonstrations. Students were given small, low-stakes opportunities to use synthetic and spectroscopic equipment in lab. Mini-laboratories included testing the solubility of starting materials and products, performing serial dilutions of sample PDI compounds to characterize their optical extinction, and running thin film chromatography. Interactive stations were setup across the laboratory that allowed students to learn in small groups how to prepare samples for NMR, mass spectrometry, and IR spectroscopy, perform flame-drying techniques, operate a rotary evaporator, and perform flash

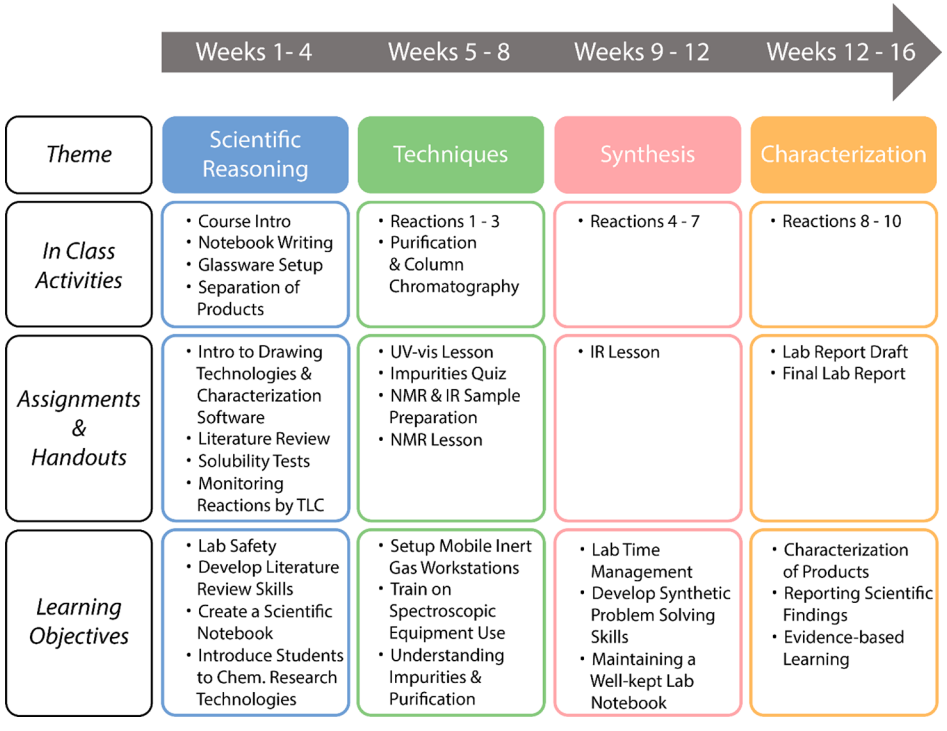


Figure 4. Chemistry Honors 2125 course structure.

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J. Chem. Educ. XXXX, XXX, XXX−XXX

column chromatography. Students were also asked to consider potential impurities in the reactions they were planning and to select the best techniques for identifying possible impurities and quantifying reaction yields. To assess student knowledge of techniques such as flame-drying and rotary evaporation, students were observed performing these techniques by an instructor. Once a student successfully demonstrated the safe operation of these techniques, they were cleared to perform them in lab.

The next section of the course focused on "Organic Synthesis" and allowed students to achieve their independent research goals by preforming reactions in lab. Students worked independently to develop their own timeline for completion of a series of PDI products. Notebook preparation and calculations became vital prework for successful completion of reactions. Prior to conducting any synthesis work, procedures proposed by students were evaluated and graded by instructors. Throughout these weeks, students managed air-free, organic glassware setups, executed their proposed reactions, and purified their crude materials. Students learned how to monitor reaction progress and purification including TLC and solubility tests. In addition, learning objectives of laboratory safety and organization were consistently integrated into the daily class time through PPE checks, notebook checks, and monitoring proper storage of materials and reagents by students. Students encountered optimization and time management lessons within the context of their research.

The last 4 weeks of class focused on "Chemical Characterization" of product compounds obtained by students. During these weeks, students concentrated on the collection and analysis of spectroscopic data (^{1}H NMR, ^{13}C NMR, UV−vis absorbance/emission, and infrared spectra) and MS data that could confirm the successful preparation of target compounds and provide some measure of their properties, such as molar extinction coefficients, optical absorption lineshapes, and emission quantum yields. Students were assessed on their ability to record and analyze spectroscopic data for structure determination. By the start of this unit, most students had successfully completed the synthesis of one or more products. If characterization of these products was complete, students were allowed to continue to attempt synthesis of additional compounds intended to display new chemical or optical properties. During this last four week period, students also composed a final laboratory report that summarized their synthetic outcomes and the properties of compounds they had obtained.

Skills obtained by students who completed Chemistry Honors 2125 include the ability to read and search scientific literature, skills in maintaining scientific notebooks and composing written reports of research outcomes, the ability to conduct independent lab work and perform upper-level organic synthesis, and skills in characterizing compounds using spectroscopic and MS methods. Below, we provide two example lessons used to build student skills in two of these areas, organic synthesis and spectroscopic characterization of compounds via ^{1}H NMR analysis. These lesson plans are intended to provide an overview of the general flow of a class session and provide a window into how students might respond to a given learning objective or lesson plan. Both lesson plans are written to appeal to students engaging in each of these tasks for the first time.

Organic Synthesis Lesson Plan

In one synthetic-focused class period, students set up their first reaction performed under inert atmosphere conditions. The main objectives of this lab class were to give students practical knowledge of organic reflux techniques as well as setup an air-free reaction chamber for the first time. This first synthesis day had students constantly working throughout the period and required that all students come to class with a complete and thorough notebook prep and a clear idea of their goals for the day.

Before beginning their reactions, students were given a short demonstration of a method for flame-drying glassware and were shown an inert atmosphere glassware setup that would become their goal to assemble. Instructors also provided a "notebook check" for completion of prelab assignments that outlined tasks associated with glassware assembly and setup of an inert atmosphere chemical reaction. Either this demo or notebook check assignment could be moved to a prior class day to allow for more time in lab for assembly of synthetic setups by students. Students in the laboratory at ACC share hood spaces between two people. They were reminded to maintain communication with their partner when applying an open flame to their reflux chambers. Instructors took time to check the general progress of the class in creating their inert atmosphere chambers and reminded students that they could refer to their notes from the demo session on how to stepwise prepare their stations. Pairs of students worked cooperatively to assemble their glassware and add to them starting materials and reagents. Each hood shared a single Tupperware container to supply water to the two students' condensers. Instructors checked each student setup for watertight junctions to avoid water dripping into the oil baths (a splash and burn hazard). When students had added all their materials, they were instructed to take photographs of their setup to include in their notebook. Students timed their reactions individually for 2 h. At the end of this period, round-bottom flasks were lifted from heat and the reactions were cooled and worked up. Filtering and washing took minimal time and students had the remainder of class to clean their workstations. Subsequent reactions took less time for students to setup, perform, and clean, but preparation prior to class consistently proved key to successful completion of their goals in the class period. Students were encouraged to take copious notes and photos of their reaction setups as their reactions progressed. Students submitted a completed lab notebook page online to a shared Google Drive folder.

^{1}H NMR Lesson Plan

For a week in which reaction product characterization was a focus, students used their knowledge of ^{1}H NMR to predict the peaks and splitting of hydrogen signals they would see in an experimental spectrum. This lesson required that students sketch their anticipated products, label hydrogens and lines of symmetry within the molecule, and identify key organic functional groups that may shield, deshield, or fragment their spectral peaks. Students were then asked to draw the spectra they predict and compare it to one found in the literature prior to coming to class. In class, students prepped samples for ^{1}H NMR to be run at UT's facilities. Samples were run by a UT student worker or facilities manager and the data was emailed to students. Students then used Mnova software to integrate and assign peaks in their experimental NMR spectra. They compared their predictions, values from the literature, and

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J. Chem. Educ. XXXX, XXX, XXX−XXX

spectra taken experimentally with their own samples. Students saw the effect of residual solvent peaks as well as impurities in their "real" ^{1}H NMR spectra. This allowed for a fruitful discussion about impurities and the assignment of ^{1}H NMR peaks. Students were encouraged to theorize about how they might obtain better spectral resolution and what constitutes thorough reporting of ^{1}H NMR spectra in the literature.

SUMMARY AND CONCLUSIONS

CUREs serve a key role in introducing students to research and can provide them with a foundational experience that can serve as a basis for their future success in STEM fields. We have developed an inquiry-based, research-focused course that introduces students to methods in modern organic synthesis by having them engage with a modular, three-step synthesis for preparing a library of PDI dyes. These materials were chosen as a basis for an inquiry-based course due to their relevance in applications relating to light harvesting, medicine, and catalysis on top of the low-cost of starting reagents for their synthesis, their ability to be prepared in only a few steps using safe procedures, and their synthetic modularity, which allowed each student to prepare a unique set of compounds over a semester-long project. The high-yielding synthetic process we developed was successfully pioneered in a laboratory course at ACC, which presented unique challenges in terms of shared use of teaching laboratory space and limited resources for equipment that were overcome through the creation of low-cost, mobile workstations for performing chemical synthesis under an inert atmosphere. Through partnership with UT, ACC students were exposed to several advanced characterization techniques such as NMR and mass spectrometry. Our course develops students' independent research skills, providing them with abilities such as critical thinking, knowledge of chemical synthesis, research writing, and time management—each of which are vital to success in upper-level chemistry coursework and the STEM workforce.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available at https://pubs.acs.org/doi/10.1021/acs.jchemed.2c00662.

Discussions of synthetic procedures and preparation of specific compounds, schematics of inert atmosphere workstations used for PDI synthesis, parts used to assemble inert atmosphere workstations, IR spectra, NMR spectra, absorption and emission spectra, and mass spectra, tables of parts used to assemble inert atmosphere workstations, and comparison of measured ^{1}H NMR and high-frequency IR transitions to literature reports (PDF)

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Notes

The authors declare no competing financial interest.

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J. Chem. Educ. XXXX, XXX, XXX−XXX