

# Revisiting Elimination Reactions in the Pentanol and Bromopentane Series

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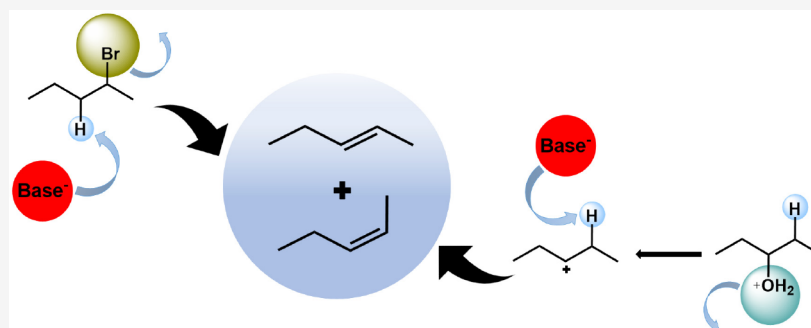
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**ABSTRACT:** While elimination reactions are ubiquitous in organic chemistry textbooks, their implementation in undergraduate laboratories still suffers from a lack of reliable and easy setup procedures. This paper describes an easy-to-implement set of elimination experiments in the pentanol and bromopentane series using a modified Hickman distillation head. These experiments expose the students to elimination reactions under acidic and basic conditions while analyzing the reaction mixture to determine the stereochemistry of the final products. Additionally, the new safe and green design of these two experiments makes them attractive to implement in large enrollment organic chemistry teaching laboratories in addition to the experiments already available in such settings.

**KEYWORDS:** *Organic Chemistry, Elimination Reactions, Third-Year Undergraduate, Laboratory Instruction, Gas Chromatography, Nucleophilic Substitution, Analytical Chemistry*

## INTRODUCTION AND BACKGROUND

Substitution and elimination reactions are two fundamental reactions taught in undergraduate organic chemistry.<sup>1–5</sup> Both reactions are studied substantially in theory, but nucleophilic substitution reactions have garnered more usage in laboratory settings as compared to elimination reactions.<sup>6–9</sup> The major problems associated with performing the elimination experiments are the number of products observed and the low boiling points of the formed products.<sup>10–19</sup> In this paper, we will show an easy to implement protocol for the acid catalyzed elimination of the pentanol series and 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU) catalyzed elimination of the bromopentane series using gas chromatography (GC) and proton and carbon nuclear magnetic resonance (<sup>1</sup>H and <sup>13</sup>C NMR) as analytical tools. We will also show a detailed investigation of the effects of the position of either the hydroxyl or the bromine group on the elimination products under these conditions. This study aimed to reinforce the basic principles of S<sub>N</sub>1/E1 and S<sub>N</sub>2/E2 reactions for third-year undergraduate students.

We have previously published on the nucleophilic substitution reactions of the pentanol series.<sup>20</sup> Therein, we demonstrated how <sup>1</sup>H NMR spectroscopy could be used as an inexpensive and readily available method to analyze the reaction

products for the nucleophilic substitution reactions of 1-, 2-, and 3-pentanol and 2-methyl-2-butanol. The results of that work prompted us to consider a similar approach for elimination reactions.

For this study, we looked at two elimination reactions: the dehydration in the pentanol series under acidic conditions and the dehydrohalogenation in the bromopentane series under basic conditions in an undergraduate laboratory setup (Scheme 1). For the alcohol elimination reactions, students used a 50% aqueous sulfuric acid solution at 95 °C, while for the elimination reactions in the bromopentane series, the students used 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU), a bulky non-nucleophilic base at 85 °C. In all cases, the students analyzed the outcome of their reaction mixtures using <sup>1</sup>H NMR, <sup>13</sup>C NMR, or GC when required. These analytical tools allowed the students

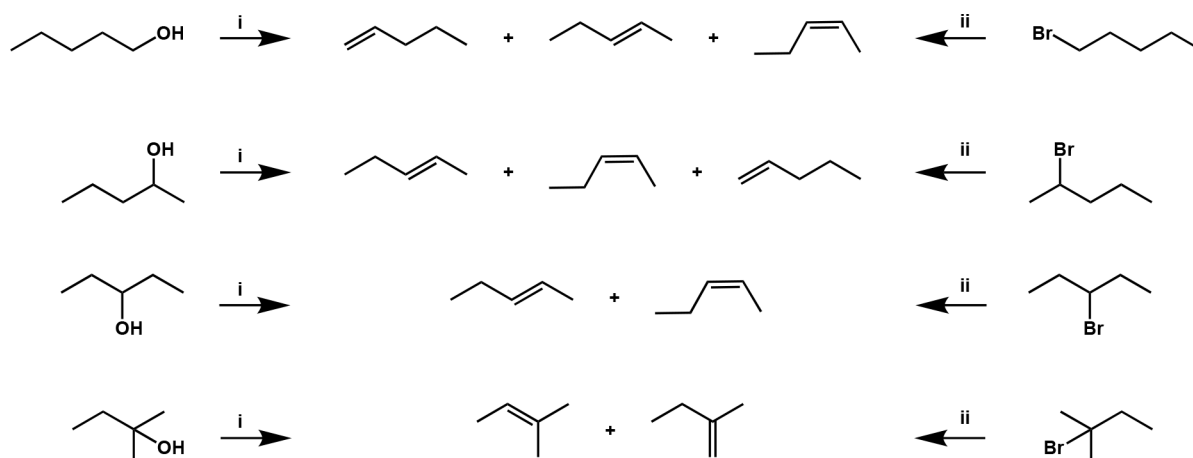
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**Scheme 1.** Expected Reaction Products for the Elimination Reaction in the Pentanol Series under Acidic Conditions (i) 50% Aqueous  $\text{H}_2\text{SO}_4$ , 95 °C, and in the Bromopentane Series under Basic Conditions (ii) DBU, 85 °C



to identify the formed compounds and, more importantly, their stereochemistry, an essential feature of such reactions.

Using either aqueous sulfuric acid or DBU, the two first sets of elimination reactions provided an inexpensive and easy method to demonstrate the stereoselectivity of the elimination reaction in the undergraduate teaching laboratory. The use of water as solvent for the acid catalyzed elimination reactions and DBU as the solvent for base catalyzed elimination reaction provided a safe and green approach toward these experiments with a very attractive E-factor (g waste per g product).<sup>21</sup> Indeed, such experiments while yielding an appreciable amount of product did result in a minimal amount of waste either after the reaction or after the products' analyses. Moreover, we have found it important to incorporate E-factor calculations into the development of new laboratory experiments to eliminate redundant or unnecessary reaction steps, thus dramatically reducing the amount of waste generated per experiment. Furthermore, since the procedure and setup for the dehydrohalogenation and dehydration experiments were similar, instructors could use a mix of haloalkanes and alcohols to show the different products formed for each reaction. Additionally, we have expanded on the previous work on secondary alkyl halides to include 3-bromopentane, which is essential to further illustrate the behavior of all secondary substrates with bases.<sup>15,16</sup>

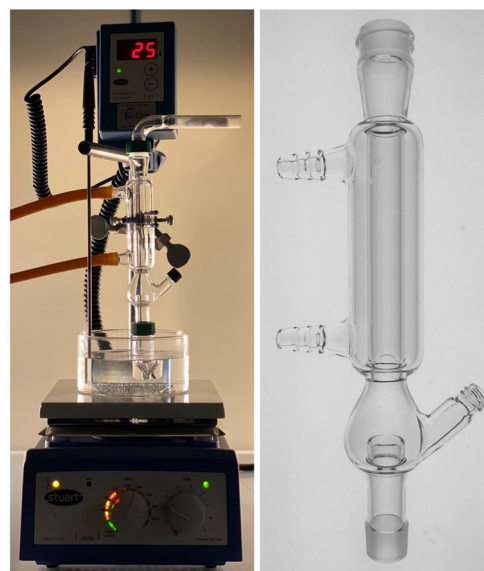
### ■ PEDAGOGICAL GOALS

For the elimination reactions under acidic conditions or using DBU, in the Fall 2021 semester, third-year undergraduate students performed the elimination experiments under the supervision of teaching assistants (TAs) and a senior instructor. The first batch of students performed the elimination reactions for the pentanol series under acidic conditions. The students were paired up, and 1-, 2-, or 3-pentanol or 2-methyl-2-butanol were assigned to each group randomly. Likewise, the second batch of students performed the elimination reactions under basic conditions for the bromopentane series. Once more, the students were paired up and randomly assigned 1-, 2-, or 3-bromopentane or 2-bromo-2-methylbutane. At the end of the experiment, the students collected their products, prepared their NMR and GC samples, and submitted them for analysis. The GC,  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectroscopy data were shared with the students for interpretation and analysis of the reactions outcomes. These experiments allowed the third year students to

confirm and reinforce their knowledge of the elimination reaction, especially when using DBU as a base. While students are usually familiar with eliminations using sodium methoxide and ethoxide as well as potassium *tert*-butoxide, they are less familiar with the elimination using DBU. This experiment was found to be useful by the students to reaffirm their knowledge of the factors influencing the formation of the Saytzeff or the Hoffman product, and after the careful analysis of their assessments in the form of a laboratory report, it was found that, by the third year, most if not all students had a good grasp on the factors influencing the elimination reactions, their outcome, and stereochemistry.

### ■ EXPERIMENTAL DESCRIPTION

Detailed instructions with notes are available in the [Supporting Information](#).

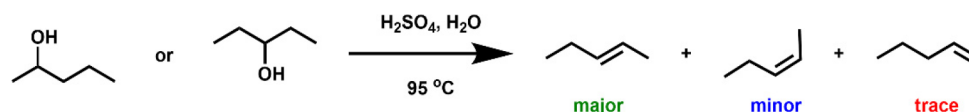


**Figure 1.** Distillation setup (left) and modified Hickman distillation head (right) (see the [Supporting Information](#) for specifications).

Table 1. Reaction and Analyses E-Factors Calculated for the Alcohol and Alkyl Halide Elimination Reactions

experiment	reaction E-factor <sup>a</sup>	analysis E-factor
Pentanol series dehydration (H <sub>2</sub> SO <sub>4</sub> )	2-pentanol: 15.8 (3.38)	GC: 4.38
	3-pentanol: 9.20 (3.38)	<sup>1</sup> H NMR: 4.39
	2-methyl-2-butanol: 8 (3.38)	<sup>13</sup> C NMR: 4.39
Bromopentane series dehydrohalogenation (DBU)	2-bromopentane: 19.94 (3)	GC: 2.96
	3-bromopentane: 13.14 (3)	<sup>1</sup> H NMR: 2.97
	2-bromo-2-methylbutane: 11.26 (3)	<sup>13</sup> C NMR: 2.97

<sup>a</sup>Between bracket is the calculated optimal E-factor using quantitative yields; otherwise, the E-factors given are those of the average reaction yields.

Scheme 2. Elimination Reaction of 2-Pentanol and 3-Pentanol under Acidic Conditions Yields *trans*-2-Pentene as Major Product, *cis*-2-Pentene as Minor Product, and Traces of 1-Pentene

### General Instructions for the Reactions under Acidic Conditions Using H<sub>2</sub>SO<sub>4</sub> and Basic Conditions Using DBU

Students were paired in groups and given a vial containing the alcohol or the alkyl halide (premeasured 1 mL). The students ran the elimination reaction and recovered the reaction products using a modified Hickman distillation head as part of their distillation setup (Figure 1).

In the elimination under acidic conditions, the students mixed their alcohol (1 mL) and a cold sulfuric acid solution (50%, 2 mL) in a conical vial and heated the reaction mixture using an oil bath previously adjusted to 95 °C. For the reaction under basic conditions, the students mixed DBU (1 mL) and their alkyl halide (1 mL) in a conical vial and heated the reaction using an oil bath previously adjusted to 85 °C. In both reactions, after a few minutes, bubbles appeared, and the alkenes' mixture began collecting in the Hickman distillation head. At the end of each reaction, the students collected the alkenes' mixture in a preweighed vial, calculated the reaction yield, and prepared an NMR sample (CDCl<sub>3</sub>) and a GC sample (MTBE).

### GC Samples Preparation and Parameters

Full detailed descriptions and instructions of the GC parameters used in all experiments are available in the Supporting Information.

### E-Factor Calculations (g Waste per g Product)

This laboratory experiment was designed with two specific goals in mind: the isolation of highly volatile olefins in large quantity and quality, all while minimizing the waste generated during the laboratory period. The E-factor calculation was used by the class instructor to develop this new experiment while adding a new waste minimization dimension to the experimental design. We further differentiated between the reaction E-factor calculated using the following formula, E-factor = Total waste (g)/Total product (g), and the analysis E-factor calculated using the total mass of reagents used during the laboratory experiments (Table 1). All of these E-factors, however, did not account for the cleaning solvents as this amount might differ depending on the laboratory glassware used and wash solvents available to the students.

### HAZARDS

Standard safety precautions and proper PPE should be used when performing these reactions, and all reactions should be performed inside a well-ventilated fume hood. DBU, is a strong

base, while concentrated sulfuric acid is corrosive, and therefore, both should be handled with care. Reagents 1-, 2-, and 3-pentanol, 2-methyl-2-butanol, 1-, 2-, and 3-bromopentane, 2-bromo-2-methylbutane, and methyl *tert*-butyl ether (MTBE) are flammable and should be handled in a well-vented environment. CDCl<sub>3</sub> is toxic and an irritant and should be handled with care. Students, teaching assistants, and instructors handling these chemicals should exercise caution.

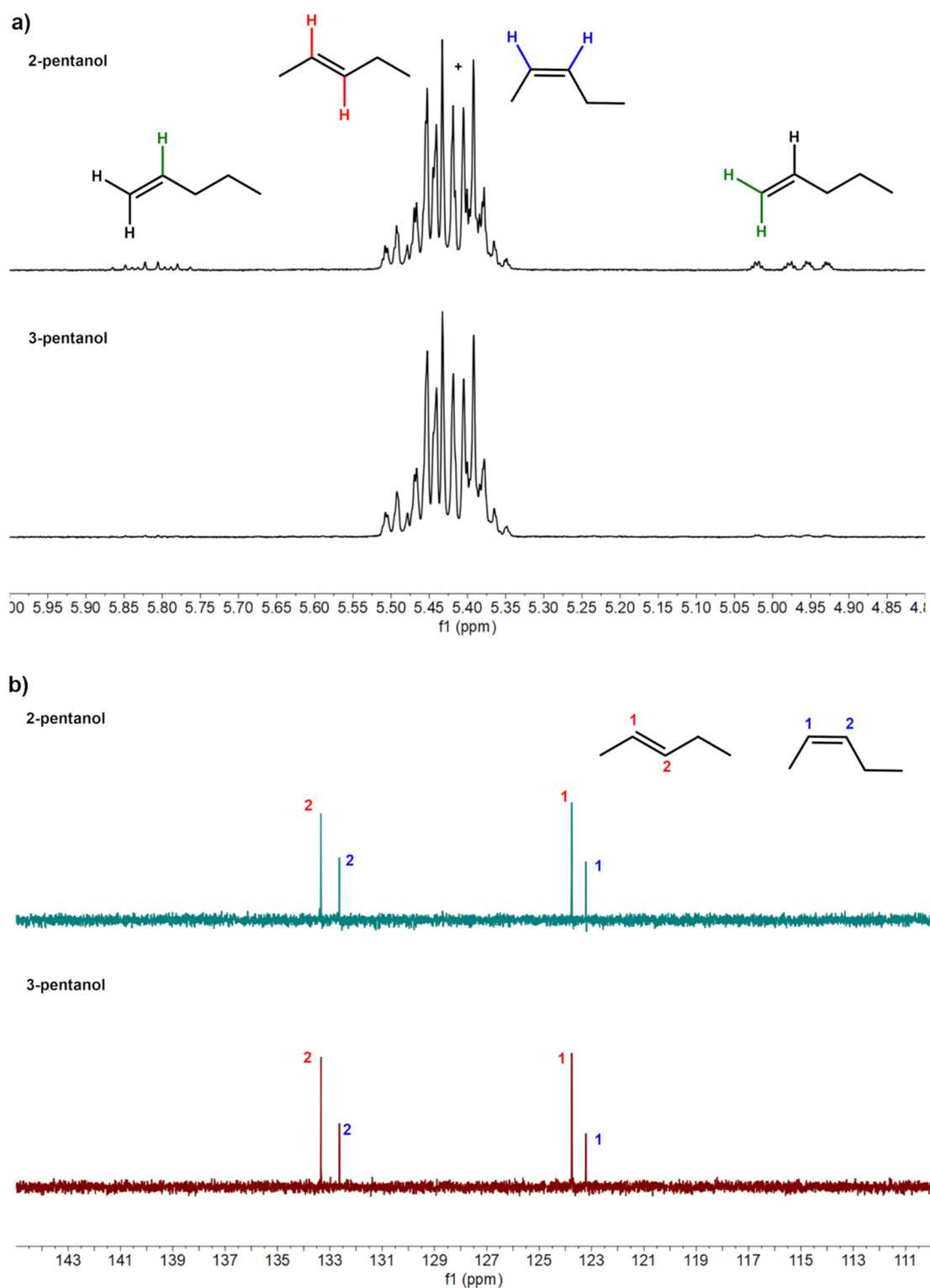
### RESULTS AND DISCUSSION

This section describes the findings from a set of elimination experiments run under acidic and basic conditions. Students used a series of alcohols to demonstrate the elimination reactions under acidic conditions, while alkyl halides were used for the elimination reactions under basic conditions. These experiments provided a large pool of data for students and instructors to use as a teaching tool to understand the different competing mechanisms under diverse reaction conditions. Furthermore, these new elimination experiments added to the collection of experimental data that instructors could readily use to set up elimination reactions in an undergraduate lab setting economically and inexpensively. Additionally, since all elimination reactions yielded a mixture of products, <sup>1</sup>H and <sup>13</sup>C NMR as well as GC efficiently identified these mixtures as evident from the obtained data (Figures S17–S42).

While elimination reactions compete with nucleophilic substitution reactions, the reaction conditions can be modified to favor elimination over substitution. (1) Steric hindrance around the alpha carbons, (2) a strong base, especially if it is a poor nucleophile, and (3) high temperature are some of the factors that promote elimination reactions. For the sake of uniformity, we kept the temperature of the reactions fixed at 95 °C for acid-mediated dehydration of alcohols and 85 °C for DBU-mediated reactions.<sup>15,16</sup>

### Elimination Reactions under Acidic Conditions

Due to the absence of an active nucleophilic species, acid-catalyzed dehydration of alcohols yields mainly the alkenes. Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) or sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) can be used for performing elimination reactions under acidic conditions.<sup>17</sup> Haloacids (HX) are not helpful as they tend to favor substitution over elimination. HSO<sub>4</sub><sup>-</sup> and H<sub>2</sub>PO<sub>4</sub><sup>-</sup> ions are very poor nucleophiles due to resonance stabilization, whereas halide anions are very good nucleophiles. Students used sulfuric acid



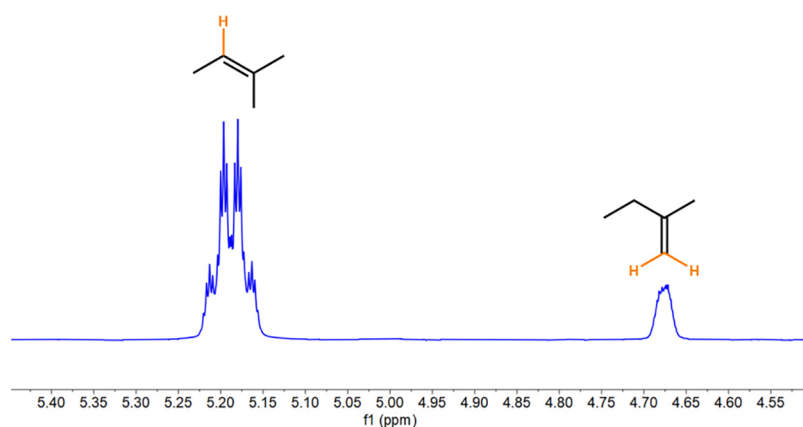
**Figure 2.** (a) Truncated  $^1\text{H}$  NMR spectra of the elimination reaction products of 2-pentanol and 3-pentanol. The major product in both reactions is a mixture of *cis*- and *trans*-2-pentene (m, 5.32–5.52 ppm). A small amount of 1-pentene was also observed (m, 5.73–5.85 ppm; m, 4.93–5.04 ppm). (b) Truncated  $^{13}\text{C}$  NMR spectra of elimination reaction products of 2-pentanol and 3-pentanol. The major product in both reactions is a mixture of *cis*- and *trans*-2-pentene.

( $\text{H}_2\text{SO}_4$ ) as the preferred acid in the reactions because of its availability.

Using the modified Hickman distillation head, the students were able to distill the highly volatile elimination products (bp below  $40^\circ\text{C}$ ) directly without using carrier solvents. This

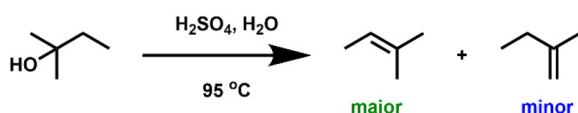
technique made the reaction simple and avoided introducing solvents and washing and drying steps when collecting the elimination products. The students were able to analyze the distillate directly and use methods such as GC and NMR without solvent removal or additional purification steps.





**Figure 3.** Truncated  $^1\text{H}$  NMR spectrum of 2-methyl-2-butanol elimination reaction under acidic conditions. 2-Methyl-2-butene (m, 5.15–5.22 ppm) and 2-methyl-1-butene (m, 4.65–4.69 ppm) were the two observed products, with the former being the major product.

**Scheme 3. Elimination Reaction of 2-Methyl-2-butanol under Acidic Conditions Yields 2-Methyl-2-butene as the Major Product and 2-Methyl-1-butene as the Minor Product**



Reactions of primary alcohols can lead to the formation of alkenes following the E2 elimination mechanism, as primary carbocations are highly unstable for E1 eliminations. 1-Pentanol, under our reaction conditions, yielded dipentyl ether as a major reaction product. The triplet peak at 3.35 ppm in the  $^1\text{H}$  NMR spectrum of the reaction mixture corresponds to the protons on the C1 carbon of the ether (Figure S17). The peak at 71 ppm in the  $^{13}\text{C}$  NMR spectrum of the same reaction mixture corresponds to the C1 carbon of the dipentyl ether. A byproduct described by the preliminary analyses as a sulfate derivative (either 1-pentyl sulfate or dipentyl sulfate) was also observed, but this matter is still under thorough investigation. In the case of primary alcohols, it has been shown that elimination requires high temperature and pressure or even a catalyst,<sup>22</sup> while under our conditions,  $\text{S}_{\text{N}}2$  was favored (Figure S17). This information can be used to teach the students about the two competing reaction mechanisms. Only when the reaction mixture was allowed to reflux did the students observe the formation of trace amounts of 1-pentene and 2-pentene (*cis* and *trans*) (Figure S17).

To demonstrate the elimination reactions of secondary alcohols, students used 2- and 3-pentanol. Elimination reactions of secondary alcohols favor the E1 pathway. In the experiments with 2- and 3-pentanol (Scheme 2), students observed a trace amount of 1-pentene in the reactions. Peaks at 4.90–5.00 and 5.72–5.87 ppm correspond to 1-pentene in reactions of 2-pentanol. Peaks between 5.32 and 5.52 ppm correspond to a mixture of *cis*- and *trans*-2-pentene in the reaction mixtures of 2-pentanol and 3-pentanol (Figure 2).  $^{13}\text{C}$  NMR spectroscopy of the reaction mixtures confirmed these observations and showed that *trans*-2-pentene was the major product (Figure 2), further corroborated by the GC analysis. GC analysis also revealed the ratio of *cis* and *trans* forms to be approximately 1:1.9 in the case of 2-pentanol and 1:2.6 in the case of 3-pentanol. These results are as previously described in the literature since *trans* products are more stable for steric reasons.<sup>1–5</sup> Another feature of this

reaction was the isolation of pure olefin mixtures in good yields, ranging from 33% for 2-pentanol to 55% for 3-pentanol.

Reactions of 2-methyl-2-butanol (*tert*-pentanol) under acidic conditions yielded 2-methyl-2-butene as the major product and 2-methyl-1-butene as the minor product (Figure 3) in agreement with Saytzeff's rule (Scheme 3). Peaks between 4.65 and 4.69 ppm correspond to the alkenyl protons of 2-methyl-1-butene, and peaks between 5.15 and 5.22 ppm correspond to the alkenyl proton of 2-methyl-2-butene.<sup>23</sup> The ratio of the two products was found to be 9:1 and could readily be determined by  $^1\text{H}$  NMR and confirmed by GC, while the average yield for this reaction was around 63%.

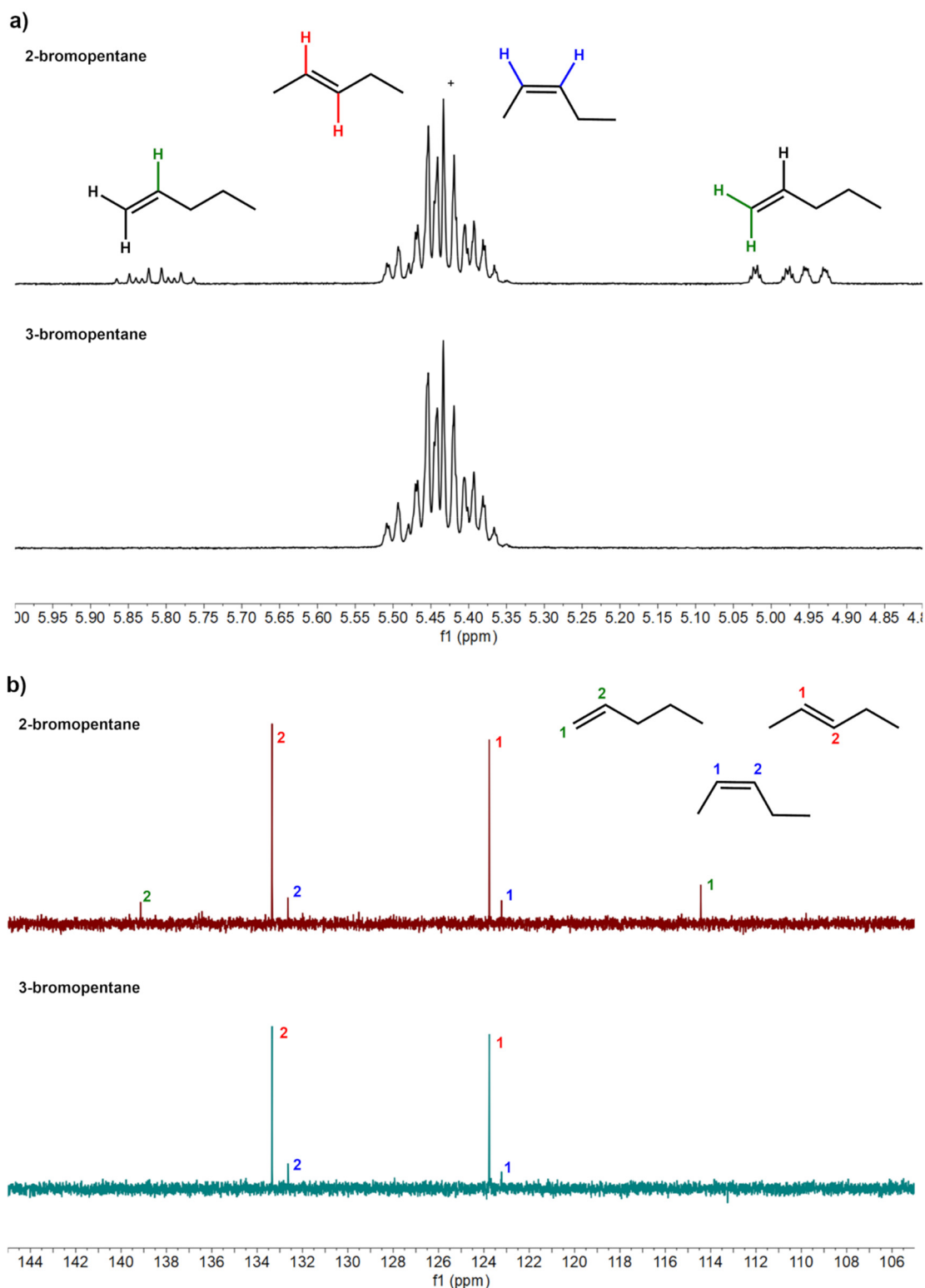
With these four experiments, instructors could readily demonstrate (1) the competition between the substitution and elimination reactions, (2) the stability of the *trans*-isomer over the *cis*-isomer, and (3) the preferred formation of the more substituted alkene over the less substituted alkene, all essential features of the elimination reactions. Additionally, this method exposed the students to the distillation and isolation of highly volatile compounds without using a carrier solvent.

On a side note, in this work, the students focused primarily on elimination products' identification and stereochemistry, while the side reactions were only investigated when no elimination products were isolated or observed (Figure S17).

**Elimination Reactions under Basic Conditions**

Bases are some of the widely used reagents for elimination reactions as acids can lead to side reactions with the formed alkenes. Using the bromopentane series as our substrate for the base-mediated elimination reactions, we investigated the DBU-mediated elimination reactions.<sup>15,16</sup>

In the presence of strong bases, elimination reactions compete with substitution reactions; however, 1,8-diazabicyclo[5.4.0]-undec-7-ene, more commonly known as DBU, is a strong but non-nucleophilic base. Additionally, we used DBU in our reactions as it served a dual purpose of reagent and solvent. Reactions of primary substrates with strong bases usually favor substitution reactions unless a non-nucleophilic base like DBU is used. We expected to observe some 1-pentene formation in the reaction of 1-bromopentane with DBU; however, students did not collect or observe any alkene from the reaction mixture in the Hickman distillation head as the reaction medium was gelatinized under the reaction conditions. Further  $^1\text{H}$  NMR analysis of the reaction mixture showed only traces of 1- and *cis*- and *trans*-2-pentene (Figure S30).

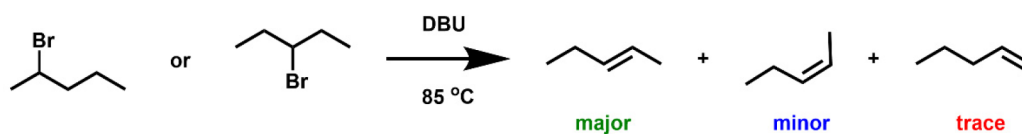


**Figure 4.** (a) Truncated  $^1\text{H}$  NMR spectra of the DBU-mediated elimination reaction products of 2-bromopentane and 3-bromopentane. The major product in both reactions is a mixture of *cis*- and *trans*-2-pentene (m, 5.35–5.52 ppm). In 2-bromopentane, a trace amount of 1-pentene was also observed (m, 5.74–5.83 ppm; m, 4.90–5.03 ppm). (b) Truncated  $^{13}\text{C}$  NMR spectra of elimination reaction products of 2-pentanol and 3-pentanol. The major product in both reactions is a mixture of *cis*- and *trans*-2-pentene.

Reactions of 2-bromopentane yielded the expected products of the reaction with DBU. The  $^1\text{H}$  NMR of the reaction mixture showed the presence of 1-pentene and *cis*- and *trans*-2-pentene in an approximately 1:9 ratio.  $^{13}\text{C}$  NMR and GC analysis confirmed the presence of the products and showed *trans*-2-

pentene as the primary product, as described in the literature.<sup>24</sup> 3-Bromopentane reaction, on the other hand, provided only a mixture of *cis*- and *trans*-2-pentene as the product, as seen from the peaks between 5.32 and 5.55 ppm in the  $^1\text{H}$  NMR of the reaction mixture (Figure 4). GC analysis of the reaction mixture

Scheme 4. Elimination Reaction of 2-Bromopentane with a Non-nucleophilic Base, DBU, Yields *trans*-2-Pentene as the Major Product and *cis*-2-Pentene and 1-Pentene as the Minor Products<sup>a</sup>



<sup>a</sup>1-Pentene was not observed in the reaction of 3-bromopentane.

Scheme 5. Elimination Reaction of 2-Methyl-2-bromobutane with a Non-nucleophilic Base, DBU, Yields 2-Methyl-2-butene as the Major Product and 2-Methyl-1-butene as the Minor Product



showed the presence of the *cis*- and *trans*-2-pentene in a ratio of 1:7.3. The results of the two secondary haloalkanes showed that the E2 elimination pathway was favored in the reactions facilitated by a non-nucleophilic strong base (Scheme 4). In 2-bromopentane, the proton on the C1 carbon is available for the incoming base facilitating 1-pentene as the product, while the elimination of the proton from C3 carbon yields the more substituted Saytzeff product. In the case of 3-bromopentane, only the C2 proton is available for elimination, yielding 2-pentene as the only product of the reaction.

The reaction of the tertiary substrate, 2-bromo-2-methylbutane, yielded results similar to those of the elimination reaction under acidic conditions (Scheme 5). <sup>1</sup>H NMR analysis showed that 2-methyl-2-pentene, 5.16–5.21 ppm, was produced as the major product and 2-methyl-1-pentene, 4.65–4.68 ppm, was the minor product as per the Saytzeff rule (Figure 5). The ratio of the two products observed was 4:1, which is only slightly different from the ratio observed under acidic conditions.

To further drive the elimination reaction stereochemistry and outcome concepts home, instructors can simply use a summary bar graph of the students' findings to share with the class. Indeed, Figure 6 readily displayed the outcome of each reaction and the product distribution, showing that in all cases the Saytzeff products and the *trans* products were favored under the used reaction conditions.

## STUDENT RESPONSE

As seen in the Supporting Information, all students were able to perform these reactions in the laboratory setting using the modified Hickman distillation head. This reaction implementation allowed the students to isolate highly volatile compounds and analyze them without the help of a carrier solvent or additional purification or extraction steps, dramatically lowering the chemical waste per laboratory section and experiment. Moreover, with these new experiments in hand, the instructor could readily compare the outcome of the elimination reaction using sulfuric acid and DBU to the ubiquitous elimination reactions using alkoxides. As summarized in Figure 7, the average laboratory report scores were high (Students: 34; Mean: 87.9), showing an overall strong understanding of the elimination reaction mechanism and stereochemistry as well as the factors influencing the formation of the Saytzeff and Hoffman products. Most of the students were able to accurately predict the outcome of each one of their reactions using their knowledge of the E1 and E2 eliminations, perform the reaction, isolate their olefins, and run the necessary analyses to identify them. The interpretation of the GC results was rather straightforward; however, in certain cases, they did struggle with the NMR spectra interpretation and assignments, even when supplied with the FIDs and trained on the NMR spectral analysis software. Students commonly struggled with properly assigning the proton chemical shifts to their structures or calculating products' ratios from their integrations especially when signals overlapped. Finding the signals to integrate and use for the product ratio's calculation proved challenging at times, and discussing such matters with the class instructor or teaching assistants proved essential for the proper calculations. This observation further reinforced the belief in exposing students to more NMR spectroscopy of complex mixtures as opposed to solely relying on given GC ratios. We have found that pairing

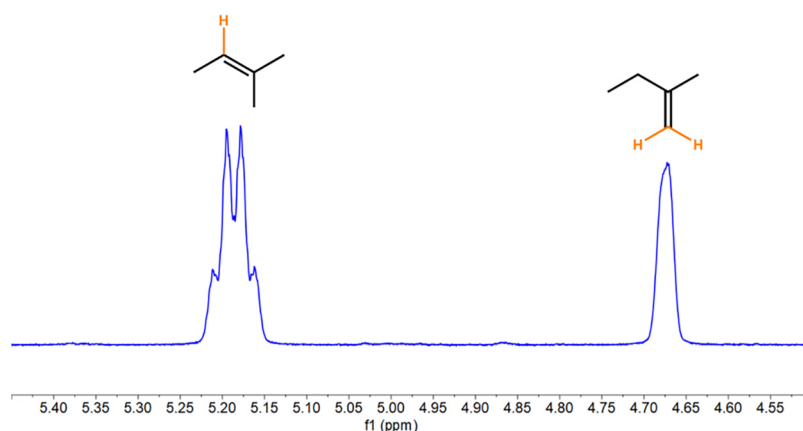
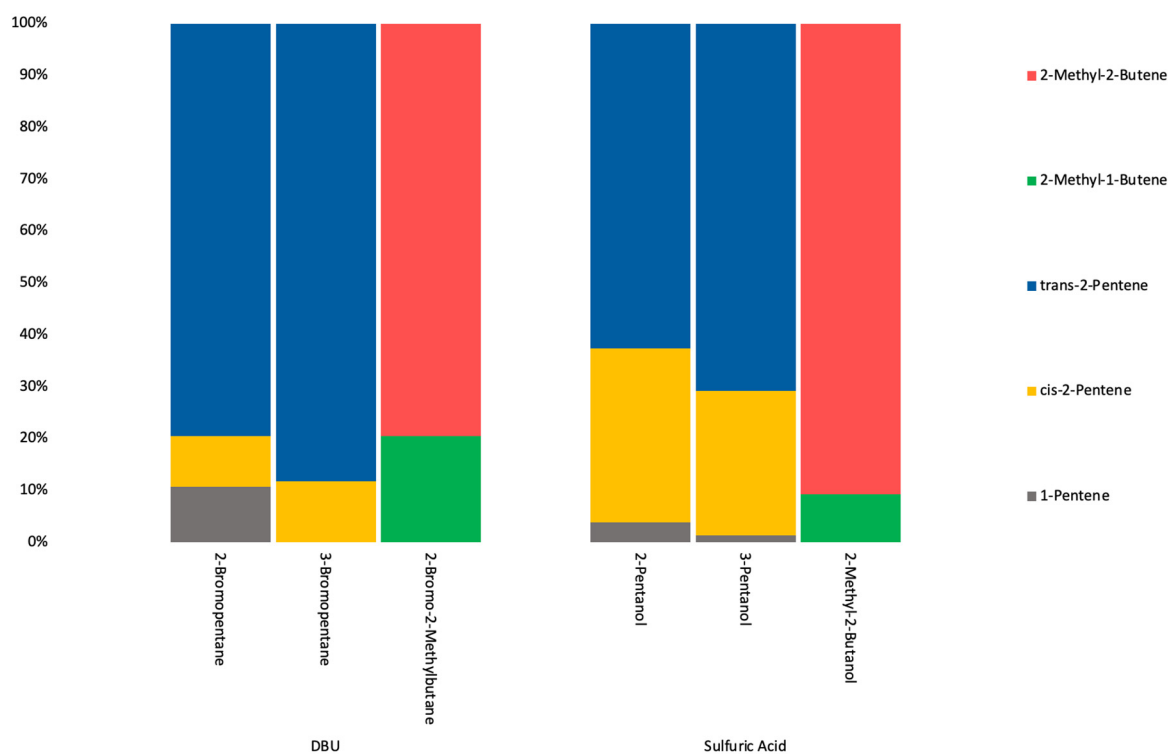
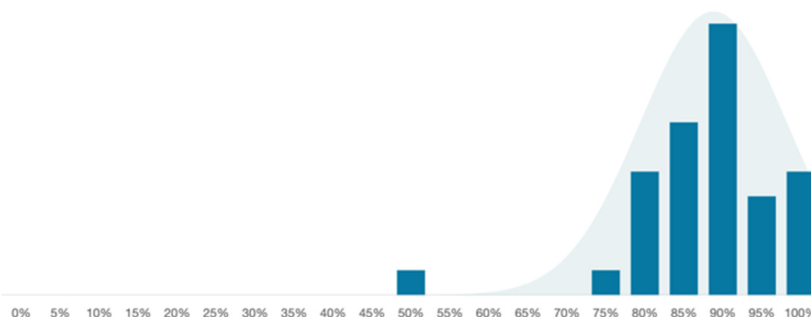


Figure 5. <sup>1</sup>H NMR spectrum of 2-methyl-2-bromobutane elimination reaction products under basic conditions. 2-Methylbut-2-ene (m, 5.16–5.21 ppm) and 2-methylbut-1-ene (m, 4.65–4.68 ppm) were the two observed products, with the former being the major.



**Figure 6.** Comparison of relative elimination product ratios in the pentane series using DBU and sulfuric acid.



**Figure 7.** Class assessment (laboratory report) scores are given as an average out of 100%. It should be noted that grading in each cohort was performed by a single grader (Students: 34; Mean: 87.9; Median: 90; Std Dev: 9.3).

both techniques is now essential for the understanding of reactions' outcomes, especially when reaction GC standards or starting materials are inaccessible.

## CONCLUSION

The distillation and isolation of the alkenes resulting from the acid- and base-mediated elimination reactions in the pentanol and bromopentane series were demonstrated in an undergraduate laboratory setting. When using sulfuric acid and DBU, students could readily collect their products, record their spectra, analyze them, and correctly interpret the reactions' outcome using either NMR or GC analytical methods. At the same time, most of the results were in accordance with the rules and mechanisms described in most organic chemistry manuals. Moreover, the use of the modified Hickman distillation head dramatically minimized the waste generated during these laboratory experiments, especially when dealing with low boiling point products.

## ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available at <https://pubs.acs.org/doi/10.1021/acs.jchemed.2c01052>.

Lab report guidelines, questions, and marking scheme (PDF)

Experimental procedures, detailed NMR spectra, and GC graphs (PDF)

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## Notes

The authors declare no competing financial interest.

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