Suzuki Cross-Coupling Reactions: Synthesis of Unsymmetrical Biaryls in the Organic Laboratory

Christopher S. Callam and Todd L. Lowary*
Department of Chemistry, The Ohio State University, Columbus, OH 43210-1106; *lowary.2@osu.edu

The cross-coupling reaction of aryl boronic acids with aryl halides and related derivatives (e.g., triflates) provides a convenient and efficient synthetic methodology for the formation of biaryl compounds (1). The method was first reported in 1981 by Miyaura, Yanagi, and Suzuki using the conditions shown in Scheme I (2).

![Scheme I](image)

This reaction, commonly known as the Suzuki cross coupling, is now in widespread use throughout the organic synthetic community and has found much application in the area of natural product synthesis. New methodology continues to emerge and there are numerous examples in the recent literature of new highly active palladium cocatalysts (3) and the development of asymmetric biaryl cross couplings (4).

The Suzuki reaction is by far the most versatile and useful synthetic reaction for the assembly of biaryl systems. Some of the molecules this reaction has been used to synthesize are korupensamine A (5) and hippadine (6) (Scheme II). These two natural products make use of the Suzuki reaction as the key step in the formation of the C–C sp²–sp² bonds indicated.

![Scheme II](image)

Despite the popularity of this reaction, transition-metal-catalyzed processes are seldom encountered in the undergraduate curriculum. With this in mind, we have designed a laboratory experiment that exposes students to this area, allowing them to learn about the use of organometallic chemistry in the modern synthetic laboratory. The coupling reaction performed in this experiment allows students to extend their knowledge of organometallic compounds from the common organolithium and Grignard reagents to the less familiar world of organotransition metal chemistry. For example, the palladium-catalyzed C–C bond-forming reaction done in this laboratory differs significantly from the formation of these bonds via a Grignard reaction in that the transformation is catalytic in the metal and can be carried out in aqueous media. This reaction also tolerates a wide range of functionality (e.g., carbonyl groups) that would be incompatible with a Grignard reagent.

The series of molecules prepared in this experiment are substituted biaryl alcohols (4), which can be synthesized in two straightforward steps from commercially available aryl halides (1) and aryl boronic acids (2) as outlined in Scheme III. The experiment is adapted from an Organic Synthesis preparation (7). For all possible combinations of the coupling partners, both the product alcohols (4) and the aldehyde or ketone intermediates (3) are readily crystallizable solids.

![Scheme III](image)

The Experiment

This experiment is commonly performed in an honors organic chemistry lab course, which is taught in parallel with the sophomore organic chemistry lecture. This introductory course runs for a total of 20 weeks over two quarters. The experiment described here is carried out in the first half of the second quarter and requires two 4-hour laboratory periods. In the first period, each student is given (or chooses) two unknown cross-coupling partners and carries out the palladium-catalyzed reaction. In the second period, the product is recrystallized and then reduced with sodium borohydride. Purification of the alcohol by recrystallization is also done in
the second period. The structural identity of the product is then determined by obtaining its melting point, IR spectrum, and ¹H NMR spectrum. Over a two-year period, involving a total of 30 students, all successfully completed this experiment with overall yields between 50 and 80%.

The procedure as outlined in the Supplemental Material uses 1 g of the aryl halide, which affords approximately 0.6–0.8 g of the biaryl alcohol depending on which substrates are used. All the steps can be carried out with equipment standard in an undergraduate teaching laboratory. Although we performed evaporation steps using a rotary evaporator, this manipulation can also be done by simple distillation or with the use of a steam bath.

This laboratory allows for the discussion of the mechanistic details of palladium-catalyzed cross-coupling reactions, which involve (i) oxidative addition, (ii) transmetalation, and (iii) reductive elimination. Discussion can also turn toward the role of triphenylphosphine in the reaction and why aqueous sodium carbonate is needed for this reaction to proceed in a catalytic fashion. The second reaction allows the students to reinforce concepts learned in the organic chemistry lecture and reviews the mechanistic details of metal hydride reductions. Finally, many biaryl compounds (e.g., korupensamine A, Scheme II) are chiral owing to restricted rotation about the C–C bond that joins the two aromatic rings. Introducing the concept of atropisomerism is therefore possible in the context of this laboratory (8).

In the laboratory for which the experiment was designed, the goal of the experiment was for the students to determine which set of substrates they started with and the structure of the final product. This can be easily achieved by allowing the students to do some library research on the physical properties (melting points) of the possible coupling products and through the use of IR and NMR spectroscopy.

Hazards
Palladium acetate and the boronic acids are irritants. Students will also use concentrated hydrochloric acid, which is corrosive, and flammable solvents (hexanes, methanol, ethyl acetate). The use of gloves should be encouraged and where possible all transformations should be carried out in a fume hood. Because flammable solvents are employed, all heating steps should be done using an oil bath, steam bath, or heating mantle, not a Bunsen burner.

Acknowledgments
This work was supported by The Ohio State University and The National Science Foundation (CHE-9875163).

Supplemental Material
Notes for the instructor and for students are available in this issue of JCE Online.

Literature Cited
Suzuki cross coupling reactions: synthesis of unsymmetrical biaryls in the organic laboratory

Christopher S. Callam and Todd L. Lowary
Department of Chemistry, The Ohio State University
100 West 18th Avenue, Columbus, Ohio 43210

Notes for the Student

In the following document, notes preceded by "Ins" are notes for the instructor and can be found in the "Notes for the Instructor" document. Notes for the students are found at the end of this document.

CAUTION!

Aryl boronic acids and palladium acetate are irritants. Hydrochloric Acid is corrosive. Wear gloves and use caution in all steps of the laboratory experiment.

Introduction

In 1981 Suzuki and coworkers developed an efficient methodology for the synthesis of sp²-sp² carbon-carbon bonds between two aromatic rings (Scheme 1). The palladium (0) catalyzed coupling of aryl boronic acids with aryl halides (known as the Suzuki cross coupling reaction) represents one of the most efficient and simple methods for carbon-carbon bond formation in organic chemistry. The popularity of this and other palladium (0) catalyzed reactions has grown over the last twenty years and are now routine in the organic laboratory. The Suzuki reaction is by far the most versatile and useful synthetic reaction for the assembly of
biaryl systems. Some of the molecules this reaction has been used to synthesize are Korupensamine A, an anti-malarial agent, and Hippadine, an alkaloid from *Crinum amaryllidaceae* which shown biological activity (Scheme 2). Unlike the more familiar metal promoted organic reactions (*e.g.*, Grignard and organolithium reactions) the Suzuki cross coupling is catalytic in the metal and can be carried out in an aqueous environment. Furthermore, this reaction will tolerate a wide range of functional groups. For example, in this experiment you will synthesize a C–C bond in the presence of a carbonyl group in an aqueous environment. Such a reaction would be impossible with a Grignard reagent.

![Scheme 2]

The molecule prepared in the experiment is a biaryl alcohol (4, Scheme 3). The goal of the experiment is two fold. The first goal is to perform the cross coupling reaction from two unknown coupling partners and isolate the product (3). Upon isolation of (3) the carbonyl group will be
reduced with sodium borohydride to yield the biaryl alcohol (4) with an unknown substitution pattern. Both the intermediate (3) and final product (4) are solids. The second goal is to determine the structure of the product from its IR spectrum, NMR spectrum and melting point. In turn, you can determine the starting materials you were given. In the first period, you will perform the palladium catalyzed cross coupling reaction and corresponding work up to obtain a crude intermediate (3). In the second period, you will purify the intermediate (3) by recrystallization, reduce it with sodium borohydride and purify the resulting alcohol (4).

**Experimental – First Lab Period**

To a 100 mL three necked round bottomed flask equipped with a magnetic stir bar, condenser, and a nitrogen inlet balloon add the aryl halide (1.00 g, 5.02 mmol), the aryl boronic acid (0.692 g, 5.68 mmol), and n-propanol (10 mL). Stir the mixture for 15 min allowing complete dissolution of all solids. To the solution add palladium acetate (3.6 mg, 16.0 μmol), triphenylphosphine (12.8 mg, 48.8 μmol), 2M aqueous sodium carbonate (3.25 mL, 6.48 mmol), and deionized water (2.0 mL). Heat the solution at reflux under a nitrogen environment until complete (~ 1 h). The reaction progress can be monitored by TLC (4:1, hexanes:ethyl acetate) (Ins 2).

Cool the reaction to room temperature, add water (7 mL), and stir the mixture open to the air for 5 min (Stu 1, Ins 3). Dilute the reaction with ethyl acetate (10 mL) and transfer it to a separatory funnel. Separate the two layers and re-extract the aqueous layer with ethyl acetate (10 mL). Combine the organic extracts and wash them with a 5% sodium carbonate solution (2 x 10 mL) and brine (2 x 10 mL) sequentially (Stu 2, Ins 4). Transfer the organic phase to a 125 mL Erlenmeyer flask equipped with a magnetic stir bar and add activated charcoal (0.50 g) and sodium sulfate (1 g). Stir this mixture for 10 min.

Filter the solution through a 1 cm bed of Celite using a Buchner funnel into a 125 mL filter flask. After filtration, rinse the Celite with several portions of ethyl acetate. Concentrate the resulting pale yellow filtrate under reduced pressure to yield the biaryl product as a solid.
Experimental – Second Lab Period

Slurry the crude intermediate (3) in hexanes (5 mL) while warming to reflux. Add methanol (2 mL) to clarify the solution. Upon dissolution of the solid, remove the heat source and allow the solution to cool to induce crystal formation. Isolate the crystals by vacuum filtration and wash them with cold hexanes. Dry isolated crystals by suction filtration to afford the purified biphenyl adduct as a solid.

To a 50 mL Erlenmeyer flask equipped with a magnetic stir bar, add 20 mL of methanol and the biaryl adduct (3) (400 mg, 2.04 mmol). Stir the mixture at room temperature for 5 min (Stu 3, Ins 4). To the mixture, add dropwise over the course of 5 min a solution of sodium borohydride (0.09 g, 2.4 mmol) dissolved in 2 mL of water. Stir the reaction mixture at room temperature for 20 min and then pour it into a 50 mL beaker containing 10 mL of cold water and 1 mL of conc. HCl. Filter the mixture using vacuum filtration to yield the crude biaryl alcohol (Stu 4, Ins 5). Recrystallize the crude product from petroleum ether to yield the purified biaryl alcohol.

Notes for Students

Stu 1 Upon cooling the reaction, the mixture darkens and forms a thin black emulsion on top of the solution.

Stu 2 During the extractions in the work up, the thin black emulsion is taken with the organic layer each time until the final wash with brine when it is discarded.

Stu 3 The solids do not dissolve readily in the methanol until the addition of the sodium borohydride.

Stu 4 The crude solids should be thoroughly dried by suction filtration to remove any trace water prior to recrystallization.
Suzuki cross coupling reactions: synthesis of unsymmetrical biaryls in the organic laboratory

Christopher S. Callam and Todd L. Lowary
Department of Chemistry, The Ohio State University
100 West 18th Avenue, Columbus, Ohio 43210

Notes for the Instructor

Background

The palladium catalyzed cross coupling of aryl halides with organoboron species has become one of the most synthetically useful and widely used reactions for the formation of \(sp^2\)–\(sp^2\) carbon-carbon bonds. When the organoboron partner is an aryl boronic acid derivative, the process is known as the Sukuzi cross coupling reaction, a transformation first reported by Suzuki and coworkers in 1981.\(^1\) Since that time, various modifications have been made to the reaction conditions and it is now adaptable to most substrates. The increased availability of the aryl boronic acids and aryl halides now enables this reaction to be done in the undergraduate laboratory.

An organic laboratory experiment, which involves the coupling of an aryl halide (1) and an aryl boronic acid (2) using palladium (0) as the catalyst, is shown in Scheme 1. The product of the coupling (3) can be isolated as a crystalline material. Subsequent reduction with sodium borohydride yields a biaryl alcohol derivative (4) with varying functionality depending on the

\[
\begin{align*}
\text{O} & \quad \text{R} \\
\text{Br} & \quad \text{R'} \\
\text{1} & \quad \text{2} \\
\text{R} &= \text{H, CH}_3 & \text{R'} &= \text{H, CH}_3, \text{OCH}_3 \\
\text{Br} & \quad \text{R} \\
\text{R'} & \quad \text{R'} \\
\text{1} & \quad \text{2} \\
\text{1} & \quad \text{2} \\
\end{align*}
\]

\[\text{Pd(OAc)}_2, \text{PPh}_3 \quad \text{aq. Na}_2\text{CO}_3 \quad n-\text{PrOH} \quad \text{NaBH}_4 \quad \text{CH}_3\text{OH} \quad \text{H}_2\text{O} \]

Scheme 1
substrates used in the initial coupling reaction. The laboratory involves first giving the students (or allowing them to choose) an unknown boronic acid and aryl halide and having them carry out the coupling reaction and reduction. Second, the students determine the structure of the product by measuring its melting point, infrared spectrum and \(^1\)H-NMR spectrum.

The first period of the laboratory consists of performing the palladium-catalyzed Suzuki cross coupling reaction followed by standard work up as described in the Notes to Students. In common with other Pd (0) catalyzed C–C bond forming reactions, the catalytic cycle for the Suzuki cross coupling reaction involves 1.) oxidative addition, 2.) transmetallation, and 3.) reductive elimination (Scheme 2).²

![Scheme 2](image)

Oxidative addition of an aryl halide to a palladium (0) complex affords a stable sigma palladium (II) complex. Often, the oxidative addition is the rate determining step of the catalytic cycle, and the relative reactivity of the aryl halides (Ar-X) decreases in the order of
In the second step of the catalytic cycle, transmetallation occurs between the sigma palladium aryl complex and the aryl boronic acid. This process yields an intermediate palladium (II) species, which subsequently undergoes the final step of the catalytic cycle, reductive elimination, to yield the product. In addition to C–C bond formation, reductive elimination regenerates the palladium (0) catalyst. This reaction requires the presence of a base such as sodium carbonate. In the absence of an anionic base, transmetallation between organopalladium (II) halides and organoboron compounds does not occur readily because of the low nucleophilicity of the organic group on the boron atom. However, the nucleophilicity of this group can be increased by quaternization of the boron with anionic bases giving the corresponding “ate” complexes. Palladium (0) catalysts or their precursors can be used for the cross coupling reactions. Pd(OAc)$_2$ and PdCl$_2$ are excellent choices due to the fact that they are air stable and readily reduced to the active Pd(0) complexes by the phospine used for the cross coupling reaction. In this experiment, Pd(OAc)$_2$ is employed.

In the second lab period, the crude biaryl product (3) is purified by recrystallization and the aldehyde or ketone is subsequently reduced with sodium borohydride. This reaction illustrates the reduction of aldehydes and ketones to the corresponding primary and secondary alcohols, a topic covered in all undergraduate organic chemistry courses. After performing the reduction, (4) can be isolated by recrystallization. Following isolation of the final product the students take its melting point, infrared spectrum, and $^1$H NMR spectrum. Armed with this information, they are easily able to assign the structure of their products and in turn the starting aryl halide and aryl boronic acid.

**Notes for the Instructor**

Ins 1 All commercial reagents and solvents were used as supplied. Benzeneboronic acid, 4-bromobenzaldehyde, and 4-bromoacetophenone were purchased from Acros Chemicals. 4-Methyl-phenylboronic acid and 4-methoxy-phenylboronic acid were purchased from Lancaster Chemicals. $^1$H NMR spectra were recorded on Bruker NMR Spectrometers at
200, 250, and 300 MHz with an internal (CH$_3$)$_3$Si (δ 0, CDCl$_3$) for aldehyde and ketone derivatives. All $^1$H NMR spectra on the alcohols were obtained in acetone-$d_6$ and referenced to the residual solvent peak (δ 2.05). IR spectra were recorded as thin films on a Perkin Elmer 1600 Series FTIR. TLC was conducted on 0.1 mm silica gel on aluminum support, and the plates were visualized by UV fluorescence quenching. Melting points were determined on a Mel-Temp capillary melting point apparatus and are uncorrected.

**Ins 2** *Characteristic of all biphenyl ketone syntheses:* As the reaction solution was refluxing, a color change was observed, which could be used to monitor the reaction progress. During the reflux the mixture changed from light yellow (5 min) to orange (10 min), to red (20 min) and finally dark red-black (complete).

*Characteristic of all of biphenyl aldehyde syntheses:* As the reaction solution was refluxing, there was no sequential change in color observed. The reaction mixture simply went from light to dark brown (complete).

**Ins 3** Upon cooling the solution, it darkened forming a thin black emulsion on top. During the extractions in the work up, this emulsion was taken with the organic layer each time until the final wash at which point it was discarded.

**Ins 4** Initially, the solids did not dissolve completely in the methanol. Upon addition of the sodium borohydride, the solution became homogeneous.

**Ins 5** The crude solids should be dried thoroughly by suction filtration to remove any trace water prior to recrystallization.

**Ins 6** Analytical data:

**1-biphenyl-4-yl-ethanone.** (0.82 g, 83%) as a light brown solid; mp 119-120 °C (lit. 120-121°C)$^1$; R$_f$ 0.76 (4:1, hexanes:ethyl acetate). $^1$H NMR (CDCl$_3$) δ$_H$ 8.04 (d, 2 H, J 8 Hz), 7.70 (d, 2 H, J 8 Hz), 7.66-7.61 (m, 2 H), 7.52-7.38 (m, 3 H), 2.65 (s, 3 H). IR (thin film) 1681, 1602, 1403, 1358, 1264, 765, 690 cm$^{-1}$.

**1-biphenyl-4-yl-ethanol.** (0.38 g, 95%) as a shiny gray crystalline solid; mp 95-97 °C (lit. 96-98 °C)$^2$; R$_f$ 0.50 (4:1, hexanes:ethyl acetate). $^1$H NMR (acetone-$d_6$) δ$_H$ 7.67-7.34
(m, 9 H), 4.89 (q, 1 H, J 6.4 Hz), 1.43 (d, 3 H, J 6.4 Hz). IR (thin film) 3327, 2970, 1403, 1073, 1005, 896, 835 cm$^{-1}$.

**biphenyl-4-carbaldehyde.** (0.71 g, 71%) as a light brown solid; mp 60-61 °C (lit. 60-63 °C)$^3$; $R_f$ 0.82 (4:1, hexanes:ethyl acetate). $^1$H NMR (CDCl$_3$) $\delta_H$ 10.1 (s, 1 H), 7.97 (d, 2 H, J 9 Hz), 7.77 (d, 2 H, J 9 Hz), 7.65 (d, 2 H, J 9 Hz), 7.55-7.35 (m, 3 H). IR (thin film) 2342, 1696, 1604, 1216, 834, 764 cm$^{-1}$.

**4-phenyl-benzyl alcohol.** (0.37 g, 93%) as a shiny white crystalline solid; mp 104-105 °C (lit. 105-107 °C)$^4$; $R_f$ 0.32 (4:1, hexanes:ethyl acetate). $^1$H NMR (acetone–$d_6$) $\delta_H$ 7.67-7.34 (m, 9 H), 4.69 (d, 2 H, J 6 Hz), 4.18 (t, 1 H, J 6 Hz). IR (thin film) 3233, 2922, 1405, 1040, 998 cm$^{-1}$.

**1-(4'-methyl-biphenyl-4-yl) ethanone.** (0.89 g, 85%) as an off white solid powder; mp 119-120 °C (lit. 122 °C)$^5$; $R_f$ 0.90 (4:1, hexanes:ethyl acetate). $^1$H NMR (CDCl$_3$) $\delta_H$ 8.02 (d, 2 H, J 10 Hz), 7.67 (d, 2 H, J 10 Hz), 7.53 (d, 2 H, J 10 Hz), 7.30 (d, 2 H, J 10 Hz), 2.64 (s, 3 H), 2.42 (s, 3 H). IR (thin film) 3045, 1678, 1603, 1398, 1360, 1266, 1134, 847 cm$^{-1}$.

**1-(4'-methyl-biphenyl-4-yl)-ethanol.** (0.37 g, 93%) as a fluffy white solid; mp 95-96 °C (lit. 99-100 °C)$^6$; $R_f$ 0.70 (4:1, hexanes:ethyl acetate). $^1$H NMR (acetone–$d_6$) $\delta_H$ 7.61-7.24 (m, 8 H), 4.88 (dq, J 4 Hz, 6 Hz 1 H), 4.14 (d, 1 H, J 4 Hz), 2.36 (s, 3 H), 1.43 (d, 3 H, J 6 Hz). IR (thin film) 3371, 2966, 1911, 1497, 1394, 1290, 1084, 1008 cm$^{-1}$.

**4-(4'-methylphenyl)-benzaldehyde.** (0.62 g, 61%) as a white solid; mp 105-107 °C (lit. 107 °C)$^7$; $R_f$ 0.90 (4:1, hexanes:ethyl acetate). $^1$H NMR (CDCl$_3$) $\delta_H$ 10.0 (s, 1 H), 7.94 (d, 2 H, J 8 Hz), 7.64 (d, 2 H, J 8 Hz), 7.54 (d, 2 H, J 8 Hz), 7.29 (d, 2 H, J 8 Hz), 2.42 (s, 3 H). IR (thin film) 2837, 1701, 1602, 1391, 1139 cm$^{-1}$.

**4'-methyl-(biphenyl-4-yl)-methanol.** (0.36 g, 90%) as a crystalline white solid; mp 100-101 °C; $R_f$ 0.88 (4:1, hexanes:ethyl acetate). $^1$H NMR (acetone–$d_6$) $\delta_H$ 7.67-7.24 (m, 8 H).
8 H), 4.66 (d, 2 H, J 5 Hz), 4.18 (t, 1 H, J 5 Hz), 2.36 (s, 3 H). IR (thin film) 3232, 2860, 1500, 1396, 1140, 1049, 1012 cm⁻¹.

1-(4'-methoxy-biphenyl-4-yl)-ethanone. (0.77 g, 73%) as a shiny crystalline brown solid; mp 150-151 °C (lit. 153-154 °C); Rₜ 0.80 (4:1, hexanes:ethyl acetate). ¹H NMR (CDCl₃) δH 8.00 (d, 2 H, J 9 Hz), 7.66 (d, 2 H, J 9 Hz), 7.57 (d, 2 H, J 9 Hz), 7.01 (d, 2 H, J 9 Hz), 3.87 (s, 3 H), 2.63 (s, 3 H). IR (thin film) 2341, 1675, 1602, 1400, 1032 cm⁻¹.

1-(4'-methoxy-biphenyl-4-yl)-ethanol. (0.33 g, 84%) as a light gray solid; 120-120.5 °C (lit. 120-122 °C); Rₜ 0.73 (4:1, hexanes:ethyl acetate). ¹H NMR (acetone-d₆) δH 7.61-7.42 (m, 6 H), 7.04-6.99 (d, 2 H, J 3 Hz), 4.88 (dq, J 2.5 Hz, 7 Hz, 1 H), 4.14 (d, 1 H, J 2.5 Hz), 3.83 (s, 3 H), 1.43 (d, 3 H, J 7 Hz). IR (thin film) 3322, 2951, 1608, 1459, 1265, 1182, 1038 cm⁻¹.

4'-methoxy-biphenyl-4-carbaldehyde. (0.63 g, 62%) as an off white solid; mp 102-104 °C (lit. 105-106 °C); Rₜ 0.67 (4:1, hexanes:ethyl acetate). ¹H NMR (CDCl₃) δH 10.0 (s, 1 H), 7.92 (d, 2 H, J 9 Hz), 7.73 (d, 2 H, J 9 Hz), 7.59 (d, 2 H, J 9 Hz), 7.01 (d, 2 H, J 9 Hz), 3.87 (s, 3 H). IR (thin film) 2941, 2839, 1680, 1601, 1495, 1296, 1188, 1034 cm⁻¹.

4'-methoxy-biphenyl-4-yl-methanol. (0.32 g, 80%) as an white crystalline solid; mp 158-159 °C (lit. 162-163 °C); Rₜ 0.56 (4:1, hexanes:ethyl acetate). ¹H NMR (acetone-d₆) δH 7.60-7.40 (m, 6 H), 7.03-6.98 (m, 2 H), 4.65 (s, 2 H), 3.84 (s, 3 H). IR (thin film) 3220, 2838, 1607, 1500, 1447, 1400, 1272, 1182, 1037, 1012 cm⁻¹.
<table>
<thead>
<tr>
<th>Name</th>
<th>Structure</th>
<th>mp</th>
</tr>
</thead>
<tbody>
<tr>
<td>1-biphenyl-4-yl-ethanone</td>
<td><img src="image" alt="Structure" /></td>
<td>120-121°C</td>
</tr>
<tr>
<td>1-biphenyl-4-yl-ethanol</td>
<td><img src="image" alt="Structure" /></td>
<td>96-98 °C</td>
</tr>
<tr>
<td>biphenyl-4-carboxaldehyde</td>
<td><img src="image" alt="Structure" /></td>
<td>60-63 °C</td>
</tr>
<tr>
<td>4-phenyl-benzyl alcohol</td>
<td><img src="image" alt="Structure" /></td>
<td>104-105 °C</td>
</tr>
<tr>
<td>1-(4′-methyl-biphenyl-4-yl) ethanone</td>
<td><img src="image" alt="Structure" /></td>
<td>119-120 °C</td>
</tr>
<tr>
<td>1-(4′-methyl-biphenyl-4-yl)-ethanol</td>
<td><img src="image" alt="Structure" /></td>
<td>95-96 °C</td>
</tr>
<tr>
<td>4-(4′-methylphenyl)-benzaldehyde</td>
<td><img src="image" alt="Structure" /></td>
<td>107 °C</td>
</tr>
<tr>
<td>4′-methyl-(biphenyl-4-yl)-methanol</td>
<td><img src="image" alt="Structure" /></td>
<td>100-101 °C</td>
</tr>
<tr>
<td>1-(4′-methoxy-biphenyl-4-yl)-ethanone</td>
<td><img src="image" alt="Structure" /></td>
<td>153-154 °C</td>
</tr>
<tr>
<td>1-(4′-methoxy-biphenyl-4-yl)-ethanol</td>
<td><img src="image" alt="Structure" /></td>
<td>120-122 °C</td>
</tr>
<tr>
<td>4′-methoxy-biphenyl-4-carboxaldehyde</td>
<td><img src="image" alt="Structure" /></td>
<td>105-106 °C</td>
</tr>
<tr>
<td>4′-methoxy-biphenyl-4-yl-methanol</td>
<td><img src="image" alt="Structure" /></td>
<td>162-163 °C</td>
</tr>
</tbody>
</table>
**Approximate quantities of chemicals for 10 students**

**First Lab period**

<table>
<thead>
<tr>
<th>Chemical</th>
<th>CAS Registry #</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Phenylboronic acid</td>
<td>[98-80-6]</td>
<td>3 g</td>
</tr>
<tr>
<td>4-Methylphenylboronic acid</td>
<td>[5720-05-8]</td>
<td>3 g</td>
</tr>
<tr>
<td>4-Methoxyphenylboronic acid</td>
<td>[5720-07-0]</td>
<td>3 g</td>
</tr>
<tr>
<td>4-Bromoacetophenone</td>
<td>[99-90-1]</td>
<td>5 g</td>
</tr>
<tr>
<td>4-Bromobenzaldehyde</td>
<td>[1122-91-4]</td>
<td>5 g</td>
</tr>
<tr>
<td>*-Propanol</td>
<td>[71-23-8]</td>
<td>100 mL</td>
</tr>
<tr>
<td>Triphenylphosphine</td>
<td>[603-35-0]</td>
<td>0.150 g</td>
</tr>
<tr>
<td>Palladium(II) acetate</td>
<td>[3375-31-3]</td>
<td>0.05 g</td>
</tr>
<tr>
<td>Sodium carbonate (2M aq.)</td>
<td>[497-19-8]</td>
<td>35 mL</td>
</tr>
<tr>
<td>Activated charcoal</td>
<td>[64365-11-3]</td>
<td>10 g</td>
</tr>
<tr>
<td>Silica</td>
<td>[112926-00-8]</td>
<td>20 g</td>
</tr>
<tr>
<td>Sodium chloride (sat. aq.)</td>
<td>[7647-14-5]</td>
<td>200 mL</td>
</tr>
<tr>
<td>Ethyl acetate</td>
<td>[141-78-6]</td>
<td>200 mL</td>
</tr>
<tr>
<td>Sodium carbonate (5% aq)</td>
<td>[497-19-8]</td>
<td>200 mL</td>
</tr>
<tr>
<td>Celite</td>
<td>[61790-53-2]</td>
<td>25 g</td>
</tr>
<tr>
<td>Sodium sulfate</td>
<td>[7757-82-6]</td>
<td>15 g</td>
</tr>
</tbody>
</table>

**Second Lab Period**

<table>
<thead>
<tr>
<th>Chemical</th>
<th>CAS Registry #</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium borohydride</td>
<td>[16940-66-2]</td>
<td>1.00 g</td>
</tr>
<tr>
<td>Methanol</td>
<td>[67-56-1]</td>
<td>220 mL</td>
</tr>
<tr>
<td>Hydrochloric acid (conc.)</td>
<td>[7647-01-0]</td>
<td>10 mL</td>
</tr>
<tr>
<td>Hexanes</td>
<td>[110-54-3]</td>
<td>50 mL</td>
</tr>
</tbody>
</table>

*Assuming equal distribution of unknowns.
References


11 Unreported Compound


