THE QUEST FOR A trans-CHELATING DIPHOSPHINE MOLYBDENUM COMPLEX

Erin Chang, David Chen, Shrey Dalwadi, CJ Faulhaber, Serena Huang, Brian Katat, Joo Un Lee, Ethan Liu, Sydney Mullin, Jeremy Rasmussen, Beyer White, Claudia Zhang

Advisor: Dr. Mary-Ann Pearsall
Assistant: Sarah Costa

ABSTRACT
In the modern world, catalysts are essential in the synthesis of many compounds. Often, these are organometallic complexes, compounds with a central metal atom and bonded ligands. Before these catalysts can be implemented commercially, it is necessary for them to be synthesized and analyzed in a laboratory setting to fully understand their formation, structure, and properties.

This study focuses on the synthesis of an octahedral trans-chelating diphosphine molybdenum complex, an organometallic complex with a central molybdenum atom, two phosphines linked by a carbon chain on opposite sides of the molecule, and carbonyl groups on the remaining peripheral atoms. We varied the lengths of carbon chain that were used to determine the range of carbon chain length that allowed for the isomerization of the cis-substituted molecule to the trans-isomer.

The production of the molybdenum compound comprised of the initial formation of a cis-molybdenum-dipiperidine complex followed by the replacement of the piperidines with a specific diphosphine, then thermal isomerization of the cis-complex into the desired trans-configuration. The structures of the intermediates and products were analyzed by nuclear magnetic resonance (NMR) and infrared (IR) spectroscopy, which yielded valuable insight into the identity of the reaction products and the extent of isomerization.

The study was a successful proof of concept and determined that the minimum hydrocarbon chain length for trans-isomerization is 5 carbons.

INTRODUCTION
Organometallic complexes are compounds containing bonds between metal and carbon atoms. The simplest of these complexes are those containing solely carbonyl groups (CO), such as molybdenum hexacarbonyl (Mo(CO)_6). In molybdenum hexacarbonyl, the carbonyl groups function as ligands, compounds with a lone or unshared electron pair. A ligand acts as a Lewis base, readily donating its lone pair of electrons to an accepting atom. This study focused on a set of ligands, bis-diphenylphosphino alkanes. We attempted to synthesize novel trans-chelating molybdenum complexes by replacing the carbonyl groups with various diphosphine groups in an effort to better study the structure and function of similar catalysts.
In this experiment, our starting molecule had six carbonyl groups (CO), while our final product had four such groups. The diphosphine ligands consist of two phosphorus atoms, each bonded to two phenyl groups connected by a hydrocarbon chain. The bonds between the phosphorus atoms and molybdenum atoms are donor covalent bonds. The goal was to produce a molybdenum complex with a trans-chelating diphosphine group. In the cis-isomer of our various forms of molybdenum tetracarbonyl, the phosphorus atoms of the diphosphine chains were bonded adjacent to one another on the central molybdenum atom, while in the trans-isomer they were bonded opposite of one another (Fig. 1). The trans-isomer is generally more stable than the cis-isomer, mostly due to the lack of steric hindrance and the presence of larger ligands in the axial positions (i.e. the trans-isomer is energetically favored). The chelate effect explains why the two ends of the diphosphine bond to the same molecule. Namely, acting as a bidentate ligand to one metal complex is more entropically favorable than if the chain were to bond to two molecules. The primary independent variable in our research was the length of the hydrocarbon chain: we conducted syntheses with chains that were two (ethane), four (butane), five (pentane and dimethylpentane), six (hexane), eight (octane), and twelve (dodecane) carbons long. The purpose of varying the hydrocarbon chains was to determine the range of lengths that could form the trans-isomer; for example, some may be too short to stretch into the trans-position. Overall, our research aimed at providing a proof of concept as to whether such a trans-isomer might possibly be synthesized as well as which lengths of the hydrocarbon chain could amply stretch to accommodate the trans-orientation.

This novel reaction is important because transition metal complexes are key catalysts in many chemical reactions. For example, acetic acid is a substance used in polymers found in paint and adhesives, ester production, and as a solvent. It is an extremely important compound in our daily lives, but it would be impossible to generate without transition metal catalysts: a rhodium catalyst is used at low temperatures to generate mass amounts of the substance. This is merely one example of the importance of transition metal catalysts - there are thousands of reactions only possible due to the presence of these compounds. In order to use a catalyst efficiently, it is...
essential to understand the structure of these catalysts, learn more about their nature in reactions, and determine the most effective way to synthesize such compounds (economic viability). This experiment sought to build a model synthetic approach for future research by demonstrating the structural viability for other potential catalytic compounds: the molybdenum compound generated typically does not function as an effective catalyst, but it mirrors the structure of other compounds that are extremely important in the synthesis of a number of different materials.

The process for synthesizing the final product consisted of three main sections: (a) replacing two carbonyl groups with piperidine \((C_3H_7N)\), (b) replacing the piperidine with various diphosphine groups, and (c) using thermal isomerization in an attempt to create a trans-chelated diphosphine molybdenum complex.

A total of seven experiments were run, each with a different diphosphine groups: 1,2-bis(diphenylphosphino)ethane (DPPE), 1,4-bis(diphenylphosphino)butane (DPPB), 1,5-bis(diphenylphosphino)pentane (DPPP), 3,3-dimethylpentane (DPPDm), 1,6-bis(diphenylphosphino)hexane (DPPH), 1,8-bis(diphenylphosphino)octane (DPOO), 1,12-bis(diphenylphosphino)dodecane (DPPDD) (Fig. 2). These diphosphine groups differed only in the length of the carbon chain that connected the two phosphorus atoms, with the exception of 1,5-(diphenylphosphino) and 3,3-dimethylpentane, which had an additional dimethyl group. The initial starting material, Mo(CO)\(_6\), had two of its carbonyl groups replaced with piperidine, a very weak ligand due to its lack of unfilled nonbonding orbitals. The piperidine ligands were used to combat two issues: ensuring the formation of a cis-isomer and preventing the final diphosphine from replacing too many or too few of the initial carbonyl groups (our study focused on disubstitution). This transition was conducted at a low temperature in order to replace the piperidine while preventing isomerization from cis- to trans- at that step, which helped us avoid getting a mixture of products (Fig. 3). The initial formation of the cis-isomer resulted due to the fact that the diphosphine groups connected by shorter hydrocarbon chains were more likely to be stable in the cis-transfiguration. Finally, the cis-isomer was heated at 110°C to attempt the transformation into the trans-isomer. The trans-form is expected to be more stable because in the trans-isomer, the large ligands have more room and are less likely to interact with each other intermolecularly. However, the deciding factor of trans- vs. cis- was the length of hydrocarbon chains connecting the diphosphine groups. Subsequently, the second goal of the experiment was to determine the ideal range of hydrocarbon chain to use.
Figure 2: Ligands utilized in the experiment

1,5-bis(diphenylphosphino)pentane
1,5-bis(trihexylphosphino)pentane
1,6-bis(diphenylphosphino)hexane
1,8-bis(diphenylphosphino)octane
1,12-bis(diphenylphosphino)tetradecane
Figure 3: Reaction scheme for synthesis of Mo(CO)$_4$(trans-ligand)

Data analysis relied on two techniques: infrared (IR) spectroscopy and nuclear magnetic resonance (NMR) spectroscopy. The primary method implemented was IR spectroscopy, which relies on the fact that various functional groups within molecules vibrate at unique frequencies, especially CO groups.\(^5\) In other words, the frequency of electromagnetic radiation absorbed by a molecule is characteristic of both the type and strength of the bonds in the molecule as well as the number and orientation of its CO groups.\(^6\) The more symmetric these CO groups are, the fewer absorptions are seen in the IR data because all the groups stretch and bend the same way. In the data analysis of IR spectroscopy results, the trans-isomer of Mo(CO)$_4$(PR$_3$)$_2$ is predicted to have fewer peaks than the cis-isomer.

$^{31}$P NMR spectroscopy was used to probe the phosphorus atoms in the molecules. This allowed us to assess the purity of the diphosphine groups, confirm if the phosphine ligand was attached to the molybdenum complex, and determine whether a cis or trans-isomer was generated.\(^7\) We used two varieties of NMR spectroscopy: $^{31}$P and $^1$H NMR. $^1$H NMR uses the fact that non-chemically identical hydrogens split neighboring atoms in different ways, resulting in a variety of peaks that give both the number of hydrogen on the atom through the integration of the curve as well as the number of neighboring hydrogen through the splitting of the peak (it will be split into one more peak than the number of non-chemically identical neighboring hydrogen).\(^8\) $^{31}$P NMR is a similar process that is slightly easier to analyze since there are fewer phosphorus atoms.\(^9\) Chemical shift is used to monitor changes in phosphorus environments.
METHODS AND PROCEDURES

A. Replacement of carbonyl groups with piperidine

The first step of the experiment was to synthesize a molybdenum tetracarbonyl piperidine, Mo(CO)$_4$(pip)$_2$, isomer in the cis shape. 40 mL of dry toluene and 10 mL of piperidine were added to one gram (3.79 mmol) of molybdenum hexacarbonyl, Mo(CO)$_6$, under N$_2$ gas. The mixture was heated at reflux at 110°C and stirred for 2 hours (Fig. 4). The solid Mo(CO)$_6$ was completely dissolved to give a yellow-orange solution. By the end, the solution increased in opacity because yellow Mo(CO)$_4$(pip)$_2$ precipitated under heated conditions and was filtered. Then, the precipitate was washed with 20 mL of petroleum ether, and the mixture was cooled in an ice bath for 15 minutes. Afterwards, the mixture was re-filtered. Lastly, we took an IR of the product (peaks: 1934.12 cm$^{-1}$, 1888.58 cm$^{-1}$, shoulder at 1865.94 cm$^{-1}$, and 1819.31 cm$^{-1}$).

B. Replacement of piperidine groups to form cis- isomer

0.5 grams (1.32 mmol) Mo(CO)$_4$(pip)$_2$ (as prepared in A) was dissolved in 20 mL CH$_2$Cl$_2$ under N$_2$. 0.75g (2.86 mmol) of the ligand being tested was added to the mixture, which was then subjected to reflux for 15 minutes (Fig. 4). The reaction solution was filtered using vacuum filtration after being cooled to room temperature. A rotary evaporator was used to reduce the volume of the filtrate to 8 mL and 15 mL of methanol was added to the filtrate. Methanol, a polar compound, causes the formation of a precipitate when combined with the nonpolar filtrate. The solution was cooled in a freezer (<0°C) for approximately 45 hours, allowing for the crystallization of the product. The product was collected using vacuum filtration. We finally obtained the product’s IR spectrum in dichloromethane and its NMR spectrum in chloroform. The IR spectrum presented two peaks, indicating that the products were cis-. Prior to creating the solution for use in the NMR, products that were gel-like in formation, more specifically ligands 1,6 (DPPH) and greater, were subject to evaporation in the rotary evaporator to minimize the amount of remaining solvent. The NMR spectrum was also obtained to be later compared to the NMR spectrum of the product to be created in part C.

C. Isomerization of cis- to trans-

To see if the cis- compound could be converted to trans-, 0.5g (0.68 mmol) of Mo(CO)$_4$(pip)$_2$ + the ligand was dissolved in 10 mL of dry toluene under N$_2$, which gave a yellow or brown solution. This solution was then heated at reflux for 30 minutes (Fig. 4) and allowed to cool to room temperature. During the heating process, the solution may darken. The toluene was then evaporated off until a brownish off-white residue appears. In order to purify the product, the residue was dissolved in 15 mL of CH$_2$Cl$_2$, and the insoluble material was removed using a Büchner flask set up. Finally, an IR and NMR spectrum were completed in order to analyze whether the ligand was capable of conversion to a trans- product.
RESULTS AND DISCUSSION

Table 1: Qualitative Results of Products based on Diphenylphosphine Groups

<table>
<thead>
<tr>
<th>Ligand</th>
<th>cis-Mo(CO)$_4$(pip)</th>
<th>cis- product</th>
<th>trans- product</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2 (DPPE)</td>
<td>Yellow solid with thick texture. Although shade of yellow slightly varies from group to group, the texture of the solid remains consistent.</td>
<td>Yellow, crystalline</td>
<td>Yellow, crystalline</td>
</tr>
<tr>
<td>1,5 (DPPP)</td>
<td>Yellow-green powder with a very fine texture.</td>
<td>Brown solid when dry, but insoluble in toluene after reflux.</td>
<td></td>
</tr>
<tr>
<td>1,5 dimethyl (DPPMP)</td>
<td>Thick, yellow oil</td>
<td>Thick, yellow oil; slight brown impurities</td>
<td></td>
</tr>
</tbody>
</table>

Figure 4: General Set-Up for Experimental Procedures
Qualitatively, the products containing diphosphine ligands varied in their texture and color. Generally, the cis- products were yellow and particularly bright, with slight variation. The texture of the products varied much more. The cis- products containing DPPE, DPPB, and DPPP all had a solid structure, whether crystalline or powder-like. All other cis- products were to some extent viscous and had oily or gel-like properties. After the attempted isomerization to trans-, the color became much more varied. The complexes containing DPPB, DPPP, and DPPO became brown after heating. The remaining products maintained their yellow color, but tended to decrease in their brightness. All samples that had a solid or oily nature respectively maintained those same properties after the isomerization.

Many cis- products were a viscous oil (Fig. 5), suggesting that the product had great affinity for the dichloromethane solvent. This is attributable to the relatively long hydrocarbon chain and thus its greater London Dispersion Forces, the primary intermolecular force of the hydrocarbon chain. Furthermore, its long chain means that complex compounds can get “tangled” together, resulting in an oil-like product rather than a crystalline solid. After using the rotary evaporator to remove any remaining solvent, the product had a more crystalline appearance. It was still impossible to isolate to obtain an accurate mass measurement, so from this point onward there were no more yield calculations.

An IR spectroscopy was performed on a sample of Mo(CO)$_6$ to determine its structure. Because Mo(CO)$_6$ can only vibrate in one fashion, it was expected that one major peak would be created and observed through the experimental procedure. Mo(CO)$_6$ possesses six carbon atoms triple bonded to an oxygen and single bonded to a central molybdenum metal, which typically yields results in the 2100-1750 cm$^{-1}$ range$^3$. The expected results were supported by the IR with one distinct peak at 1981.54 cm$^{-1}$ (Fig. 6).

![Figure 5: Mo(CO)$_6$(cis-DPPO) is an oily, yellow substance after isolation from solvent.](image)

<table>
<thead>
<tr>
<th></th>
<th>Product</th>
<th>Description</th>
<th>Quality After Isomerization</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,6 (DPPH)</td>
<td>Bright yellow gel. Fairly viscous and stuck to the vial.</td>
<td>Viscous, yellow oil with many brown impurities</td>
<td></td>
</tr>
<tr>
<td>1,8 (DPPO)</td>
<td>Bright yellow, oily substance</td>
<td>Dark brown, very viscous, oil-like</td>
<td></td>
</tr>
<tr>
<td>1,12 (DPPDD)</td>
<td>Very viscous oil, fluorescent yellow</td>
<td>Very little change in qualities of product, maintained a yellow color but duller, and acute viscosity</td>
<td></td>
</tr>
</tbody>
</table>
The next step in the process was to initiate the di-substitution process of piperidine. A new IR spectrum was predicted due to the asymmetrical nature of the product and the presence of three distinct peaks, as a result of the asymmetry of cis geometry. The three peaks at 1934.12 cm\(^{-1}\), 1888.58 cm\(^{-1}\), and 1819.31 cm\(^{-1}\) (Fig. 7) confirm a successful disubstitution process within the compound. Peaks at 2072.61 cm\(^{-1}\) and 2012.98 cm\(^{-1}\) can be ignored as they appear due to minor impurities in the solution used for IR spectroscopy.

After the synthesis of Mo(CO)\(_4\)(pip)\(_2\), the experiments split into different directions, allowing each pair to experiment with a unique diphosphine ligand. Since the purpose of the experiment was to form a trans- configuration of the molecule, 7 different diphosphine groups were tested on the basis that a certain length would be required to synthesize an intramolecular linkage. Based on this prediction, groups were assigned various diphosphine groups, ranging from 1,2 to 1,12.

To determine the purity of each of these diphosphine groups, separate NMRs were obtained. The phosphorus NMRs yielded information about the purity of the ligands in question. It was essential to know the purity of these groups so that the designated 1.40 mmol of the ligand could be calculated and then added. Analyzing the NMR spectra, it was concluded that while
some diphosphine groups were 100% pure, others needed to be adjusted due to impurities (Table 2). Figure 8 contains a sample NMR spectra to test for purity.

Table 2: Purity of Diphenylphosphine Groups

<table>
<thead>
<tr>
<th>Ligand</th>
<th>Purity (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1,2</td>
<td>100</td>
</tr>
<tr>
<td>1,4</td>
<td>92.7</td>
</tr>
<tr>
<td>1,5</td>
<td>100</td>
</tr>
<tr>
<td>1,5 dimethyl</td>
<td>38.51</td>
</tr>
<tr>
<td>1,6</td>
<td>80.74</td>
</tr>
<tr>
<td>1,8</td>
<td>55.6</td>
</tr>
<tr>
<td>1,12</td>
<td>100</td>
</tr>
</tbody>
</table>

Figure 8: $^{31}$P NMR of DPPB. The tall peak just at about -18 ppm corresponds to the desired ligand, while the small peak at about 32.5 ppm corresponds to impurities. The percent purity was calculated with the relative integrations of the peaks.

cis- Isomer Synthesis

Compared to the IR spectrum of Mo(CO)$_4$(pip)$_2$, the peaks of the IR spectra of the diphosphine-substituted cis- isomer were slightly different in intensity and wavenumber, indicating the formation of a new molecule. The IR spectra after reaction still had three peaks, supporting the retention of cis- conformation. Figure 9 is the IR spectrum of Mo(CO)$_4$(cis-DPPO), an example of all the IR spectra of cis-substituted products for all diphosphines used in this experiment.

The NMR spectra of the cis- products also confirmed that the reactions were successful for each ligand. Figure 10 is the NMR spectra of Mo(CO)$_4$(cis-DPPO), an example of the spectra that can serve as a model for all the cis-diphosphine substituted products. The tallest peak of each spectra, which is generally around 26 ppm, corresponds with the new cis- product. That this shows the cis product is supported by the fact that the chemical shift is very similar to the literature chemical shift of Mo(CO)$_4$(cis-DPPP), 25.89 ppm, reported by Özer et al.$^{11}$ The chemical shifts of the cis-substituted isomers for each diphosphine ligand should be relatively similar to 25.89 ppm, because the only difference between each diphosphine is the length of the carbon chain. The NMRs of all the products are all within 5 ppm of the related literature value, demonstrating that replacement of the piperidine synthesis of all the cis-diphosphine isomers was successful.

The second tallest peak is a result of excess diphosphine reagent (corresponds to the peak pure diphosphine ligands). Figure 11 shows an example NMR of the DPPO sample before any
One can see that the chemical shift of the pure DPPO matches with the rightmost peak in Figure 9.

**Figure 9:** IR Spectroscopy of Mo(CO)$_4$(cis-DPPO). Significant peaks at 1901.70 cm$^{-1}$, 1873.33 cm$^{-1}$, and 1842.72 cm$^{-1}$. The peak at 2018.86 cm$^{-1}$ is a metal carbon overtone and is not significant to the analysis.

**Figure 10:** $^{31}$P Spectrum of Mo(CO)$_4$(cis-DPPO). The leftmost peak at about 25.60 ppm corresponds to the cis isomer, while the peak to the right at about -16.20 ppm corresponds to excess DPPO.
Figure 11: $^{31}$P Control Spectra of DPPO sample before any reactions. The tall peak is the pure DPPO. The chemical shift of that peak (-15.23 ppm) is very similar to that of the shorter peak in Fig. 10, indicating that there was excess DPPO mixed in with the Mo(CO)$_4$(cis-DPPO) product.

trans-Isomer Synthesis

The trans- isomers were characterized by one major peak in the IR spectra compared to three in the cis- analysis, reflecting the complexes’ symmetry. None of the experiments showed a complete disappearance of the peaks due to the presence of cis complexes, which appear as shoulders or smaller peaks at the same wavenumbers as on the cis- spectra. The experiments that used DPPE and DPPB experienced no change in their IR spectra because the trans- isomer could not be formed due to the shorter chains. All other ligands (DPPP, DPPPdm, DPPH, DPPO, DPPDD) produced a trans- isomer in some capacity, showing this shift, however, no experiments completely eliminated the cis- product, as depicted by the remaining shoulder peak (Fig. 12).

Figure 12: (left) IR spectrum of cis-DPPH substituted isomer with three distinct peaks at 1916.08 cm$^{-1}$, 1900.50 cm$^{-1}$, 1875.97 cm$^{-1}$ and (right) IR spectrum post isomerization with only a single main peak, but also a shoulder of the remaining cis- isomer at 1916.60 cm$^{-1}$, very similar to the first peak in the cis- spectra.
In analysis using NMR spectroscopy, the *trans*- isomer was characterized with a major peak downfield from the peak associated with the *cis*- isomer in the NMR spectra of the *cis*- sample. Similar to the IR analysis, the complexes with DPPE and DPPB did not seem to have isomerized to a *trans*- isomer due to their lack of the aforementioned chemical shift. All other experiments did show a new downfield peak, confirming that to some extent the *trans*-isomer was created. The surprising aspects of these spectra were the prominence of the remaining *cis*- isomer peaks (Fig. 13). This demonstrates that not all of the *cis*- product isomerized which was unexpected because the *trans*- isomer was predicted to be more thermodynamically favorable. Of the experiments that did successfully synthesize the *trans*- isomer, most had a ratio of 65% *trans*- and 35% *cis*-

Figure 13: NMR spectrum of Mo(CO)$_4$(trans-DPPO) showing a new *trans*- peak at 38.9 ppm, a retained *cis*- peak at 26.0 ppm, and an unreacted portion of the original DPPO ligand at -16 ppm.

In the case of the DPPP experiment, the percentage of the complex sample that isomerized was closer to 30% *trans*-, while 70% remained the *cis*- isomer (Fig. 14). This inverse ratio was understood as a difficulty in the relatively short carbon chain to reach opposite sides of the molecule in the *trans*- isomer. In comparison, the alternate DPPP chain containing two extra methyl groups was predicted to have a much harder time at forming the *trans*- isomer because of the protruding carbon atoms, and to behave similarly to the DPPP ligand. However, it much more closely resembled the other successful experiments using longer chains that produced a comparatively large amount of the *trans*- isomer (Fig. 14). This may be due to a formation of a dimer molecule, with one ligand connecting to two complexes, as seen in Figure 15. This would cause a peak in the NMR spectrum with a chemical shift such that was interpreted in the *trans* isomer.
Figure 14: (left) NMR spectrum for DPPP with a trans- peak at 39.06 ppm, a cis- peak at 25.82 ppm, and a peak at -16.21 ppm from excess diphosphine DPPP ligand. (right) NMR spectrum for DPPPdm with a trans- peak at 39.46 ppm, a cis- peak at 26.91 ppm, and unreacted ligand at 14.51 ppm.

Figure 15: Possible structure of DPPdm showing dimerization of two complexes

CONCLUSION AND FUTURE WORK

The purpose of this experiment was to synthesize a novel trans-chelating molybdenum complex and to determine the minimum diphosphine carbon chain length that is necessary to produce this compound. Based on the results, the 1,5 dimethyl, 1,6, 1,8, and 1,12 ligands all showed signs of this transformation by forming a gel-like, viscous liquid product. Such appearance was due to the tangling of the molecule caused by its long hydrocarbon chain. Unlike its counterparts, 1,5 was solid after isomerization; however, upon reviewing its spectra, the product was proven to have achieved the trans- conformation. Meanwhile, the products of 1,2 and 1,4 hydrocarbons showed expected results, portraying no signs of formation of the trans-isomer, as validated by IR spectroscopy and NMR tests. These compounds, unlike most trans-products, produced a brown solid instead of a gel.
Through analysis of the spectra, two particular trends were identified. For one, the phosphorus NMR spectra for the *trans*- isomer had peaks further downfield than those of the *cis*- isomer. The *cis*- isomers appeared at approximately 25 ppm in the NMR spectrum, while the *trans*- isomers observed to occur around 39 ppm. This signifies that they are experiencing more electron withdrawal. In addition, both NMR results contained an additional major peak, mostly due to the excess of reactant present in the test tube. On the other hand, the IR spectroscopy graphs showed their own trends. In the *trans*- isomer graphs, there was one major peak at around 2020 cm\(^{-1}\), while the *cis*- contained three distinct peaks. The primary peaks also contained shoulders, acknowledging the impurities in the compound.

Additionally, the NMR results showed that the *trans*- and *cis*- isomers coexisted in products with ligands that contained hydrocarbon chains long enough to support the *trans*- configuration. For the *trans*- 1,5 dimethyl, 1,6, 1,8, and 1,12 and *cis*- molecules of the final product, the ratio of *trans*- to *cis*- was approximately 65 to 35. This is a particularly interesting result, as the *trans*- isomer is supposedly more favorable, suggesting that all of the compound would be converted to this isomer. The first possibility is that the initial reactants with the *cis*- isomer resulted in an incomplete reaction, meaning we did not heat the product under reflux conditions long enough. In addition, these results could also suggest an equilibrium state between the *cis*- and *trans*- isomers. Future research would involve attempting to determine which of these conditions is the case. To test the first hypothesis, we would repeat the same experimental procedure with a longer reflux time to investigate whether the reaction goes to completion. As for the second hypothesis, we would isolate the *trans*- isomer by thin layer chromatography and place the isolated *trans*- isomer in reflux to observe whether the *cis*- isomer is formed in the final product. Interestingly, the *trans*-1,5 molecules yielded different results from the rest of the ligands, with the final product only containing only 35% *trans*- and 65% *cis*-.

We hypothesize that this is because the 5-carbon chain in the 1,5 ligand is just long enough to form the *trans* structure, but not long enough to form *trans* isomers in excess. However, the 1,5 dimethyl ligand that also has a 5-carbon chain was able to produce approximately 65% *trans*, similar to other longer ligands. A possible explanation could be because the 1,5 dimethyl is forming a more thermodynamically favored structure. This is referring to the dimerization of the 1,5 dimethyl molecules, resulting in the ends of the two diphosphine chains to be attached to two different molybdenum complexes. This hypothesis can be supported with future research by analyzing the 1,5 dimethyl product with mass spectrum, thus measuring the size and mass of the molecule.
APPENDIX

1,2

1,4:

1,5:
1,5 dimethyl:

1,6:
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