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Modules for Introducing Organometallic Reactions: A Bridge between Organic and Inorganic Chemistry

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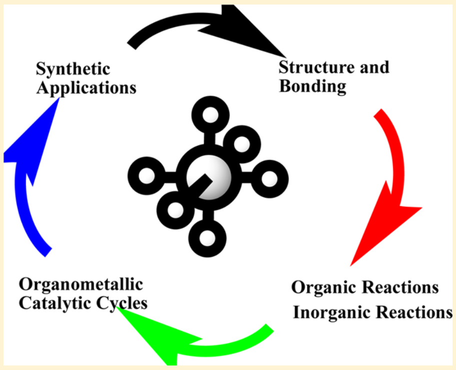
Ⓢ *Supporting Information*

ABSTRACT: Transition metal organometallic reactions have become increasingly important in the synthesis of organic molecules. A new approach has been developed to introduce organometallic chemistry, along with organic and inorganic chemistry, at the foundational level. This change highlights applications of organometallic chemistry that have dramatically evolved in recent decades. With this development, a larger number of students have been reached than would typically be exposed to organometallic reactions. Modules are described that introduce transition metal organometallic compounds and reactions by analogy with thematically similar topics in general chemistry and organic chemistry. These topics are periodically reinforced by incorporation into organic synthesis problems.



KEYWORDS: *First Year Undergraduate/General, Second Year Undergraduate, Organic Chemistry, Inorganic Chemistry, Catalysis, Organometallics, Synthesis, Curriculum*

■ INTRODUCTION

Transition metal organometallic reactions have become increasingly important in the synthesis of organic molecules.[1] Underscoring this crucial role are three Nobel Prizes in the past dozen years awarded for palladium-catalyzed cross-coupling in 2010,[2] olefin metathesis in 2005,[3] and asymmetric catalysis in 2001.[4] However, in the undergraduate chemistry curriculum, these two dynamic areas remain worlds apart: transition metal reactions are found in advanced inorganic courses, whereas organic chemistry courses rarely venture beyond Grignard reagents and hydrogenation reactions. On the basis of a recent survey of the material tested in the American Chemical Society (ACS) Organic Chemistry final exam,[5] there appears to be a trend to increase the representation of organometallic reactions, but this trend is not well supported by most introductory undergraduate organic chemistry texts.

Many students hoping to study organic chemistry arrive in graduate school with little understanding of transition metal organometallic reactions. Not all students take advanced inorganic chemistry, and in any case, such courses often relegate the treatment of organometallic chemistry to a chapter on ligands and binding and a chapter on reactions and/or industrially important catalytic cycles. While a survey of inorganic faculty members reveals that the number of advanced inorganic chemistry courses that cover organometallic chemistry is increasing,[6] only about 50% of inorganic chemistry courses cover metal carbonyl compounds or metal alkyl complexes in detail. This situation indicates that many students will still graduate with little to no understanding of organometallic reaction mechanisms or catalytic cycles.

A survey of this journal reveals only a few noteworthy articles in recent decades on ways to introduce organometallic chemistry to undergraduates.[7−11] A larger number of exercises are available on the Virtual Inorganic Electronic Resource (VIPEr), which provides a resource for faculty members attempting to increase the coverage of this content.[12−16] Most focus on the ability to name individual mechanistic steps and count electrons. Although these are key foundational topics in organometallic chemistry, the dominant approach in these exercises does not push students to use these reactions in ways that have become increasingly prevalent in the larger arena of chemistry, such as organic synthesis, polymer chemistry, or industrial processes. In addition, one could also choose to implement laboratory exercises to reinforce students' understanding of these topics.

To introduce all chemistry majors to organometallic reactions more coherently, a series of modules were designed to develop the ideas of structure and reactivity of organotransition metal compounds and the application of these ideas to synthesis. By combining these topics with the development of an understanding of chemical structure and organic reactivity, students can become proficient in bridging the realms of organic and inorganic chemistry. These modules have been incorporated in a first-year introductory course on structure in chemistry and the following first-year and second-year sequence of foundational-level courses on reactivity in organic, biological, and inorganic chemistry.[17,18] However, these themes of organometallic reactivity could also

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easily be introduced in a more traditional sequence of first-year undergraduate general chemistry and second-year undergraduate organic chemistry courses.

■ DISCUSSION

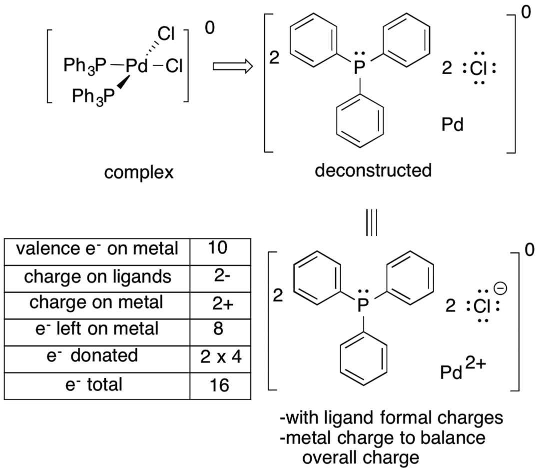
The implementation of modules on the structure and reactivity of organometallic compounds challenges students to develop an understanding of a wider range of modern reactions as well as the application of these concepts in cross-disciplinary situations. These modules are implemented periodically over the first 2 years of chemistry. Students are first introduced to structural aspects of organotransition metal chemistry in the first semester of general/introductory chemistry. Once students have attained an understanding of coordination complex geometry, isomers, and electron counting, reaction steps are presented in concert with related organic reactions, culminating in the development of complete catalytic cycles. An outline of the topics and sequencing for these modules is provided in the Supporting Information.

The delivery of organometallic chemistry aims to exploit a spiral approach in which concepts learned at one point in time are reinforced and built upon later.[19] Rather than developing one comprehensive unit for a second semester undergraduate organic chemistry course, this approach involves several short modules to be introduced within a four semesters' sequence, each time increasing the depth and building on previous knowledge.

Introductory Modules

The first modules introduce students to structural aspects of organometallic chemistry including topics of geometry, isomers, and denticity. It is not uncommon to see a unit on coordination chemistry in general chemistry textbooks. These topics could also fit easily into the first semester of an "atoms first" general chemistry. The development of Lewis structures is extended to introduce a basic tool in transition metal organometallics. Rather than leaving the rudiments of the eighteen-electron rule for an advanced inorganic class, this relatively simple concept is introduced in the first semester of introductory chemistry. Students adapt to this task quickly, especially when provided with an electron-counting table that they can fill in (Figure 1).

In a follow-up unit, students are introduced to coordination compounds in the context of Lewis acid−base complexes. Students explore organic and inorganic reactions using curved arrow notation (electron pushing). Arrow notation is conceptually useful so that students can keep track of bond-breaking and bond-making steps, yet it is not widely used in most inorganic courses.[20] At this point, students can begin to explore other related topics, such as Hard−Soft Acid−Base Theory.



valence e⁻ on metal	10
charge on ligands	2-
charge on metal	2+
e⁻ left on metal	8
e⁻ donated	2 x 4
e⁻ total	16

Figure 1. Electron counting in coordination complexes.

Modules on Reaction Mechanisms

Building upon students' understanding of the structure and geometry of coordination complexes, modules on key mechanistic steps in organotransition metal reactions are introduced. These major classes (with their complementary reverse reactions) are association/dissociation, oxidative addition/reductive elimination, and insertion/elimination. The most common transition metal organometallic reactions are presented alongside analogous reactions from organic chemistry in these modules. Examples of all modules are provided in the Supporting Information.

Ligand substitution in coordination complexes can occur via a spectrum of pathways with two limiting cases: either a dissociative mechanism (dissociation prior to association) or an associative mechanism (association prior to dissociation). This class of reactions is naturally discussed in close proximity to aliphatic nucleophilic substitutions (S_N1 and S_N2, Figure 2).

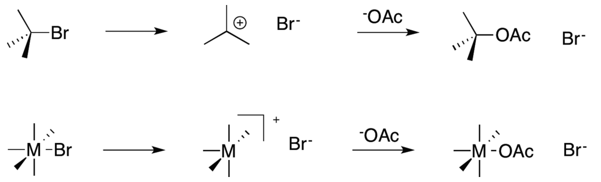


Figure 2. Comparison of unimolecular substitution in alkyl halides (S_N1) and coordination complexes (dissociative ligand exchange).

Because these different kinds of reactions have some similar characteristics, including similar rate laws in limiting cases, they serve to reinforce some ideas about kinetics. In addition, they help to illustrate the role of structural factors in influencing mechanistic pathways.

Similarly, because S_N2 reactions represent one mode of mechanism for oxidative addition, it is useful to introduce oxidative addition/reductive elimination as a related topic (Figure 3). Because of the rich variety of oxidative addition mechanisms, these reactions could alternatively be discussed with radical reactions, for example, or with reactions controlled by orbital symmetry (pericyclic reactions and cycloadditions). However, students seem to grasp easily the concept that there are two different types of oxidative addition available: those that look just like aliphatic nucleophilic substitutions and those that must be different for lack of a leaving group.

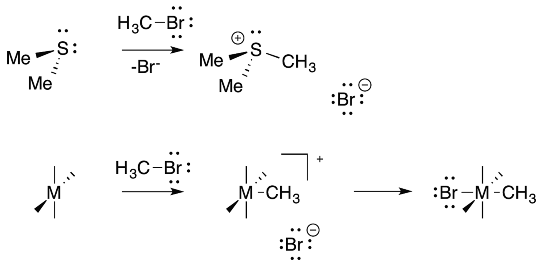


Figure 3. Nucleophilic substitution and oxidative addition.

The third major class of organotransition metal reactions, insertion/elimination, has close analogies in both carbonyl addition and electrophilic addition to alkenes, depending on the case. It is easiest to draw analogies between the addition of complex hydrides and Grignard reagents to organic carbonyls and 1,1-insertion (or migratory insertion) involving coordinated carbon monoxide (Figure 4). This discussion can be extended to describe 1,2-insertions (or beta-insertion) to coordinated alkenes.

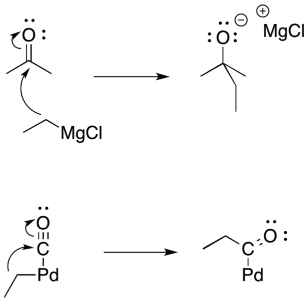


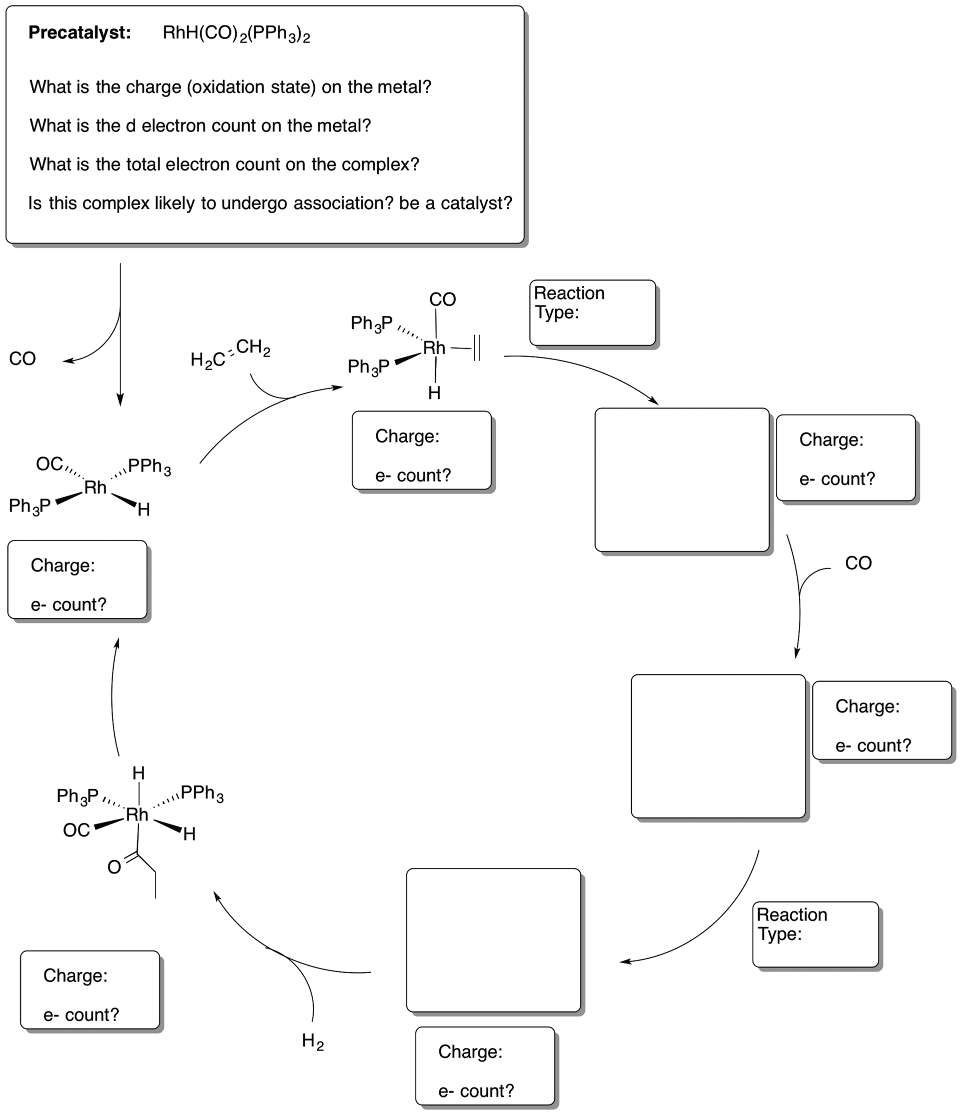
Figure 4. Nucleophilic addition to carbonyl and migratory insertion.

Beyond these three basic reaction classes, students are also introduced to redox reactions in both organic reactions and organotransition metal reactions. Finally, olefin metathesis is covered in the context of reactions under control of orbital symmetry. This topic is covered shortly after a section on alkene oxidation reactions including ozonolysis and osmium tetroxide-mediated dihydroxylation. Coverage of this additional reaction is justified because of its ubiquitous use in the synthesis of complex organic molecules, chemical feedstocks, and polymers.

Catalytic Cycles

A true understanding of transition metal chemistry allows students to employ a series of organometallic reactions in a catalytic cycle. Introducing catalytic cycles provides an opportunity to push students to incorporate the key mechanistic steps developed in previous units.

Hydroformylation is a great example of a reaction that produces a dramatic change in molecular architecture (Figure 5). This is an important example in homogeneous catalysis, leading to over 7 million tons of products per year.[21] The hydroformylation of long-chain alkenes also has interesting



Precatalyst: $RhH(CO)_2(PPh_3)_2$

What is the charge (oxidation state) on the metal?

What is the d electron count on the metal?

What is the total electron count on the complex?

Is this complex likely to undergo association? be a catalyst?

Figure 5. Exercise in which students develop a catalytic cycle for hydroformylation.

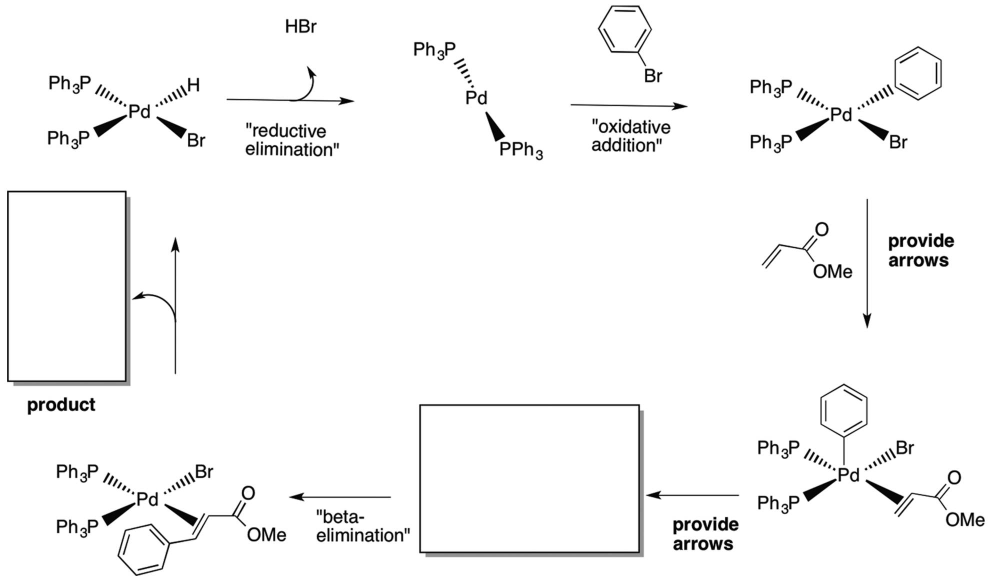


Figure 6. Exercise in which students develop a catalytic cycle for the Heck reaction.

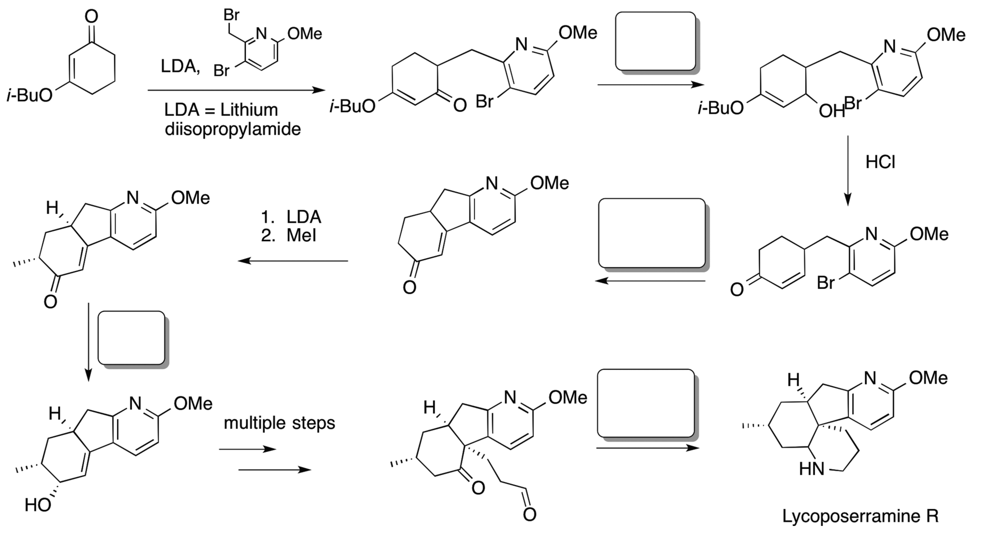


Figure 7. Exercise exemplifying the use of the Heck reaction in the synthesis of a natural product.[23]

practical applications, providing products that can be used in the synthesis of detergents and other commodities. Although the overall cycle might seem relatively complicated, students can see that the mechanism is a combination of the simpler steps that they have already learned. Furthermore, applying the 18-electron rule can help them understand the order of the steps. Being able to comprehend such an elegant homologation, as well as others, can be very motivating for students.

Other transformations of industrial importance, such as the Dupont hydrocyanation and the Monsanto acetic acid processes, are all within reach of students who have mastered the three essential classes of organometallic reactions. At this point, students can also be introduced to homogeneous and heterogeneous catalysis. Hydrogenation is a familiar reaction in all organic chemistry courses; however, students often are not exposed to the subtleties involved in these two different mechanisms. Students can begin to understand the stereochemical preference for syn addition in heterogeneous catalysis and the difference in reactivity between the reactivity of Crabtree's catalyst and Wilkinson's catalyst.

Use in Synthesis

The modules on reactivity culminate in the application to the production of pharmaceuticals and other valuable commodities. As organotransition metal reactions have become indispensible methods for synthetic organic chemists, students need to be presented with these tools.

Students are introduced to these reactions in the lecture course rather than the laboratory so that students understand the mechanism as well as application. "Roadmap problems" are frequently employed in which students fill in missing reagents or products along a synthetic route leading to the total synthesis of a natural product as described in the primary literature.[22] These exercises effectively underscore the utility of synthesis. In addition, they provide practice with previously learned reactions.

These syntheses frequently incorporate organometallic reactions, such as the Heck reaction (Figure 6), Stille couplings, or Suzuki reactions. Seeing these reactions used in context (Figure 7) helps students to appreciate the central role of transition metal chemistry in modern synthesis.

Recently, introductory organic chemistry laboratory experiments have been published that incorporate these organometallic catalyzed reactions.[24] Although this approach is laudable, it does not provide the breadth or depth of understanding that can be achieved through concerted integration of the material. These laboratory approaches would be enhanced if instructors also instituted the use of organometallic modules throughout general and organic chemistry.

Assessment

Student comprehension of organometallic chemistry was primarily assessed through short quizzes on specific topics conducted during class time. Representative data are summarized in Table 1; examples of quizzes can be found in the Supporting Information. Overall, students demonstrated a reasonable level of proficiency with the selected topics: ligand binding, geometry, and electron counting in the first semester course, CHEM 125 (Structure and Properties in Chemistry); insertion and elimination reactions in the second semester course, CHEM 250 (Reactivity in Organic, Inorganic and Biochemistry I); and catalytic cycles in the third semester course, CHEM 251 (Reactivity in Organic, Inorganic and Biochemistry II). Insertion and elimination reactions proved a greater challenge for students than did simple structural considerations in organometallic complexes. Nevertheless, students were able to consolidate their knowledge by the third semester, when they were asked to demonstrate an understanding of catalytic cycles. These students successfully bridged the gap between organic and inorganic chemistry.

Table 1. Results from Class Quizzes on Selected Organometallic Topics

Quiz Topic	Course	Sample Size (Ave.)	Mean Score (Ave.)	Standard Deviation (Ave.)
Geometry, ligands, e^--count	CHEM 125[a]	18.4	77.0	17.7
Insertion and elimination	CHEM 250[b]	24.3	65	17.3
Catalytic cycles	CHEM 251[c]	19.7	75.7	13.3

[a]Structure and properties; this material could be inserted into General Chemistry I or II. Total students: 129. [b]Reactivity I; this material could be inserted into Organic Chemistry I. Total students: 73. [c]Reactivity II; this material could be inserted into Organic Chemistry II. Total students: 59.

Although the courses listed in Table 1 do not correspond to those in a typical chemistry curriculum, for the purposes of this article they can be thought of as corresponding to General Chemistry I, Organic Chemistry I, and Organic Chemistry II. A typical unit on coordination chemistry, found in most general chemistry textbooks, could readily be extended to include organometallic bonding. Likewise, coverage of alkene addition and elimination reactions in Organic Chemistry I closely parallel beta-insertion and -elimination, whereas migratory insertion is more similar to addition to carbonyls in Organic Chemistry II. Catalytic cycles could be introduced toward the end on an Organic Chemistry II course. In each case, the introduction of new exercises in each course could reap benefits in enhanced comprehension of an increasingly important area of chemistry.

Class quizzes do not provide an external measure of student performance; that information is better assessed via ACS standard exams. Because our courses do not correspond to specific ACS exam subject areas, we worked with the ACS Exams Institute to develop three hybrid tests comprised of questions from different subject exams. For organometallic topics, the relevant questions were drawn from the ACS Organic Exam and the ACS Inorganic Exam. Student scores on individual questions were compared with the difficulty index reported from nationally normed data; the difficulty index is the fraction of students who answered an individual question correctly. Results are compiled in Table 2. The questions are described only vaguely in order to preserve the integrity of the ACS exams. These tests were administered at the end of each of the Reactivity courses: CHEM 250, CHEM 251, and CHEM 315 (Reactivity in Organic, Inorganic and Biochemistry III). In a more traditional curriculum, the additional exposure to organometallics in CHEM 315 might be replicated in an undergraduate inorganic chemistry course.

Table 2. ACS Exam Question Results (from Fall 2012 through Spring 2014)[a]

Question Topics	CSB/SJU Ave % Correct	National Ave % Correct
CHEM 250 (n = 413)		
Electron counting in a complex	63	65
Identify organometallic reaction type	32	81
Identifying dative bonds	76	60
CHEM 251 (n = 205)		
Identify oxidation state, coordination number	49	41
Electron counting in a complex	52	65
Identify organometallic reaction type	77	81
CHEM 315 (n = 79)		
Predict reactant required in organometallic reaction	76	55
Predict product of organometallic catalysis	80	82

[a]At least one section of each course was taught each semester, except CHEM 315, which was not taught in Fall 2013.

Overall, the results in Table 2 suggest that CSB/SJU students performed at a level comparable to the national pool, despite encountering these concepts at an earlier stage in their college careers than their counterparts nationally. A moderate weakness in identifying organometallic reactions in CHEM 250 mirrors the previous results on the class quizzes. However, as indicated by the quiz results, students were able to improve their performance by the following semester. That eventual mastery of the topic is also suggested by the results of the ACS exam questions.

ACS exam questions were supplemented by additional questions contributed by the course instructors. This practice was motivated partly to maintain exam lengths at approximately 70 questions, making the test-taking experience as similar as possible to an ACS organic exam. In addition, these supplementary questions increased the breadth of organometallic coverage. Results from these questions are summarized in Table 3. Overall, these questions indicate an acceptable level of retention of the material by the end of the semester.

Instructors also solicited students' opinions about their learning progress. At the end of each semester of the Reactivity

Table 3. Supplemental Final Exam Questions

Question Topics	Ave. % Correct
CHEM 250 (n = 413)	
Mode of metal-alkene binding	81
CHEM 251 (n = 205)	
Identify organometallic reaction type (2 questions)	89
Predict product of catalytic cycle (2 questions)	85

sequence (CHEM 250, CHEM 251, CHEM 315), students completed a SALG survey (Student Assessment of Learning Gains)[25] using a five point Likert scale to rate their learning progress in broad areas of each course. Areas with relevance to organometallic chemistry are summarized in Table 4. Additionally, students in all three courses were asked during spring 2014 how their study of organometallic chemistry helped them increase their understanding of specific topics, such as coordination complexes or catalytic cycles (Table 5). The average responses in both cases were typically between 3 (moderate gain) and 4 (good gain). Significantly, overall evaluations of these learning gains increased from the first to the third semester of Reactivity.

Table 4. Student Assessment of Learning Gains in Areas of General Relevance to Organometallic Chemistry (from Fall 2013 and Spring 2014)[a]

Topic Areas	Score[b]
CHEM 250 (n = 171)	
How to recognize Lewis acids/bases and electrophiles/nucleophiles and apply these principles in the reactivity of transition metal complexes, organic carbonyls and biochemical reactions:	4.1
The use of molecular orbital and ligand field theory to understand properties and reactivity	3.5
CHEM 251 (n = 70)	
The use of transition metals to catalyze organic reactions	4.1
CHEM 315 (n = 34)	
Understanding mechanisms and regiochemical and stereochemical outcomes of pericyclic reactions, as well as the role of pericyclic reactions in organic oxidations, olefin methathesis, and biochemical processes	3.9

[a] Survey question: As a result of your work in this course, what gains did you make in your understanding of each of the following? [b] Scale: 1 = no gains; 2 = a little gain; 3 = moderate gain; 4 = good gain; 5 = great gain.

Table 5. Student Assessment of Learning Gains on Specific Topics as Attributed to the Study of Organometallic Chemistry (from Spring 2014)[a]

Topic Areas	CHEM 250 (n = 136)[b]	CHEM 251 (n = 15)[b]	CHEM 315 (n = 21)[b,c]
Electron counting	3.4	3.7	4.0
Catalytic cycles	3.0	4.2	4.2
Organic synthesis[d]	3.1	4.2	4.4
Metal–ligand binding	3	4	4.1

[a] Survey question: How much did the study of organometallic chemistry help you understand the following? [b] Scale: 1 = no gains; 2 = a little gain; 3 = moderate gain; 4 = good gain; 5 = great gain. [c] The CHEM 315 survey replaced the term "organometallic" with "bioinorganic" in the survey question. [d] For CHEM 315, this question was replaced with "reduction or oxidation of substrate."

■ CONCLUSION

Knowledge of organotransition metal reactions has become essential to organic and inorganic chemists alike. In order to prepare students for graduate school or the workplace, these topics need to be presented in a context in which they can be repeated and seen in new variations. Interspersing these reactions with those of organic chemistry can help to deepen students' understanding of organometallic reactions by building on natural connections between the two fields. Allowing students to see related reactions in organic and inorganic contexts builds in a short-term spiral approach in which students have two chances to "get" a concept within the same semester. Because the organometallic concepts are introduced in short modules in different semesters, a long-term spiral is built that allows students to return to these concepts and consolidate their knowledge. Results from in-class quizzes, ACS exam questions, and student surveys all suggest a reasonable comprehension of organometallics can be reached even by first-year and second-year students. By presenting these concepts at a foundational level, students are able to build on these ideas as they encounter them again in in-depth courses.

The series of modules presented here was used to introduce the ideas of coordination compounds to students in their first two years of classes. The topics develop from simple structural concepts to different reaction steps and eventually to catalytic cycles and the application to organic synthesis and polymer synthesis. These modules can be used to introduce the topics of organotransition metal complexes into lower division chemistry courses. The approach could also complement a growing library of organometallic laboratory experiments for undergraduates, allowing instructors to select appropriate activities for their students to build this knowledge base semester after semester.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Overview of the organotransition metal chemistry within a traditional curriculum and within the CSB/SJU curriculum and examples of the problems and sample quizzes. This material is available via the Internet at http://pubs.acs.org.

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Notes

The authors declare no competing financial interest.

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