Catalytic Asymmetric C–H Arylation of \( \eta^6 \)-AreneChromium Complexes: Facile Access to Planar-Chiral Phosphines

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Supporting Information

ABSTRACT: A catalytic asymmetric direct C–H arylation of \( \eta^6 \)-arene chromium complexes to obtain planar-chiral compounds is reported. The use of the hemilabile ligand \( \text{H}_8 \text{BINAP}(O) \) is key to providing high enantioselectivity in this transformation. We show that this methodology opens the door to the synthesis of a variety of planar-chiral chromium derivatives which can be easily transformed into planar chiral mono- or diphosphines. Mechanistic studies, including synthesis and characterization of Pd and Ag complexes and their detection in the reaction mixture, suggest a Pd-catalyzed/Ag-promoted catalytic system where Ag carries out the C–H activation step.

KEYWORDS: asymmetric catalysis, C–H activation, (arene)-chromium complexes, planar-chirality, chiral ligands

1. INTRODUCTION

Unsymmetrically substituted metallocenes and \( \eta^6 \)-arene chromium complexes are two notable families of planar-chiral transition-metal organometallic complexes, and they have extensively been applied as stoichiometric auxiliaries and/or starting materials for asymmetric synthesis of biologically interesting substances.1,2 Derivatives of ferrocene have been extensively investigated as optically active ligands in catalysis.3 In contrast, the use of (arene)chromium complexes as chiral ligands has been significantly less explored with only a small selection of such ligands reported to date (Figure 1). In spite of this, a variety of (arene)Cr(CO)\(_3\) based chiral ligands have been successfully used in asymmetric C–C bond formation reactions, such as cross-coupling reactions,4 addition of dialkylzinc to benzaldehyde,4,5 1,4-additions to Michael acceptors,4,5 hydrovinylations,4,5 1,2-additions of phenylboroxines to imines5 and nucleophilic substitutions.6b,8 Such ligands have also been employed in asymmetric reductions,8b,9 hydroaminations,8b hydroborations,10 and hydroisilylations.11

Formation of optically active (arene)chromium species is usually accomplished by means of either resolution of racemates,12 or asymmetric synthesis via diastereoselective complexation,13 diasteree- or enantioselective deprotonation/electrophile quenching14 and nucleophilic addition/hydride abstraction sequences15 (Scheme 1a–c). All these methods employ stoichiometric chiral reagents or auxiliaries. Additionally, many of these processes are hampered by the use of sensitive organometallic reagents, poor functional group compatibility, and/or low atom economy. Conversely, the

more attractive access to nonracemic (arene)chromium complexes via asymmetric catalysis has been significantly less explored. Uemura first reported the desymmetrization of o-...
Scheme 1. Synthesis of Planar-Chiral (Arene)Cr(CO)₃ Complexes

2. RESULTS AND DISCUSSION

2.1. Optimization of the Conditions of the Direct Asymmetric C–H Arylation. We have recently demonstrated that (fluoroarene)Cr(CO)₃ complexes such as 1a can undergo Pd-catalyzed direct arylation affording excellent yields of ortho-substituted biaryls with high regioselectivity. A combination of stoichiometric and kinetic mechanistic studies showed that PPh₃ ligated Ag(I)-carboxylates are responsible for the C–H activation step. With this in mind, we envisaged that the process could be rendered asymmetric in the presence of a chiral phosphine ligand suitable for distinguishing between the two enantiotopic C–H bonds of a prochiral complex. Furthermore, the presence of a C–F bond in the aromatic core should allow for the subsequent easy transformation of the resulting products into chiral arylphosphine derivatives via phosphinolysis.

We started our investigation testing similar conditions to those reported for the (nonasymmetric) ortho-arylation of (fluorobenzene)Cr(CO)₃ (1a) complexes as a benchmark for reactivity. Under these conditions good reactivity was observed with 41% of racemic monoarylated product 3aa and 31% of bisarylated 4aa formed (Table 1, entry 1). We then replaced Pd(PPh₃)₄ with Pd(dba)₂ and (S)-BINAP (L₁), but under these conditions no reactivity was observed (entry 2). We had previously observed that the use of the highly hindered amine TMP (2,2,6,6-tetramethylpiperidine) as an additive enhances the reactivity of η⁶-coordinated arenes toward C–H arylation. Gratifyingly, the use of this additive promoted the reaction, forming monoarylated chromium complex 3aa in 28% yield and a low but promising enantioselectivity (38:62 er) (entry 3). A screen of Pd(II) sources revealed that Pd(CH₃CN)₄(BF₄)₂ gave similar yield to Pd(dba)₂ with a slightly higher enantiomeric ratio (entry 4). Given that the carboxylic acid is involved in the C–H activation step, we expected the enantioselectivity of the process to be heavily influenced by the nature of the carboxylate. Indeed, carboxylic acid screening showed that dicyclohexylacetic acid enhanced the reactivity and, importantly, the enantioselectivity (er: 18:82−16:84, entries 5 and 6). Conversely, reaction in the absence of the carboxylic acid gave a low yield of arylation and the enantiomeric ratio dropped to 55:45 (entry 7). Reactivity was completely shut down in the absence of silver carbonate (entry 8), whereas in the absence of K₂CO₃ a slightly lower yield and enantioselectivity were obtained (entry 9). Unreacted starting material was observed in all reactions. In some cases, free arene(s) was detected due to decomplexation...
of starting material and/or product and/or bisarylated byproduct.

We then set out to explore a variety of chiral ligands (Table 2). In comparison with BINAP (L1), the more sterically hindered 3,5-xyllyl-BINAP (L2) gave a similar arylation yield but significantly lower enantioselectivity (Table 2, entries 1 and 2). On exploring the effect of the bite angle, we found that SegPhos (L3), with its smaller bite angle than L1, decreased the enantiomeric ratio while keeping similar reactivity; DIOP (L4), which has a larger bite angle than L1, also gave very low enantioselectivity (entries 3 and 4), suggesting that bite angles similar to that of L1 were ideal for this transformation. Good conversion but low ee was observed when the monophosphine MeO-MOP (L5) was used (entry 5). Phosphoramide MonoPhos (L6) and the P,N-ligand PPFA (L7) both completely inhibited the reaction (entries 6 and 7), while H8-BINAP (L8) led to similar reactivity to its unsaturated counterpart BINAP (L1) but with slightly higher ee (entry 8). Finally, we tested BINAP(O) (L9), a ligand that has been rarely used in asymmetric catalysis, which contains both a strong and a weak donor atom, providing it the ability to act as a di- or monodentate ligand. Interestingly, in the presence of this hemilabile ligand, the yield and enantioselectivity of the process increased up to 47% and 14:86 er, respectively (entry 9).

| Entry | Conditions | Yield 3aa (%) | ee 3aa (%) | Yield 4aa (%) | ee 4aa (%) |
|-------|------------|---------------|------------|---------------|------------|
| 1 †   | Pd(PPh3)3, 1-AdCO2H | 41            | –          | 31            |            |
| 2     | Pd(dba)2, 1-AdCO2H  | 0             | –          | 0             |            |
| 3     | Pd(dba)2, 1-AdCO2H, 2 equiv TMP | 28          | 38:62      | 9             |            |
| 4     | Pd(CH3CN)2(BF4)2, 1-AdCO2H, 2 equiv TMP | 28          | 36:64      | 7             |            |
| 5     | Pd(CH3CN)2(BF4)2, Cy2CHCO2H, 2 equiv TMP | 39          | 18:82      | 33            |            |
| 6 ‡   | Pd(CH3CN)2(BF4)2, Cy2CHCO2H, 2 equiv TMP | 43          | 16:84      | 39            |            |
| 7 ‡   | Pd(CH3CN)2(BF4)2, 2 equiv TMP | 24          | 55:45      | 2             |            |
| 8 ‡   | Pd(CH3CN)2(BF4)2, Cy2CHCO2H, 2 equiv TMP | 0           | –          | 0             |            |
| 9 ‡   | Pd(CH3CN)2(BF4)2, Cy2CHCO2H, 2 equiv TMP | 40          | 19:81      | 27            |            |

Table 2. Screening of Chiral Ligands in the Asymmetric C–H Arylation of Complex 1a with 2a

**All hydrogen atoms are omitted for clarity.**

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### 2.2. Synthesis and Reactivity of H8-BINAP(O) and H8-BINAP Derivatives

As the best results of reactivity and selectivity were found when H8-BINAP (L8) and BINAP(O) (L9) were used as chiral ligands, we decided to synthesize H8-BINAP(O) and other partially hydrogenated BINAP variants to test them as chiral ligands in the C–H asymmetric arylation of chromium complexes.

**Scheme 3. Synthesis and ORTEP Plot of (R)-H8-BINAP(O) Ligand (L10)**

**All hydrogen atoms are omitted for clarity.**
confirmed by X-ray diffraction analysis (Scheme 3). With the aim of investigating the influence on the modification of electronic and steric properties of the H₈-BINAP(O) ligand on the reactivity and enantioselectivity of the reaction, we synthesized a range of H₈-BINAP derivatives (L11−L18) following similar experimental procedures to those reported for their BINAP analogues (see Supporting Information).

Gratifyingly, when H₈-BINAP(O) (L10) was tested as the ligand, high reactivity and enantioselectivity were observed for the arylation of 1a (Table 3, entry 1). We then tested ligands in which the weaker donor atom of the mono-oxidized H₈-BINAP(O) ligand on the reactivity and enantioselectivity of the reaction, we synthesized a range of H₈-BINAP derivatives (L11−L18) following similar experimental procedures to those reported for their BINAP analogues (see Supporting Information).

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Table 3. Screening of H₈-BINAP Derivatives in the Asymmetric C–H Arylation of Complex 1a with 2a

| Entry | Ligand | Yield 3aa (%)a | er 3aa b | Yield 4aa (%)b |
|-------|--------|----------------|---------|----------------|
| 1     | (S)-L10| 42             | 7.93    | 27             |
| 2     | (S)-L11| 33             | 46.54   | 16             |
| 3     | (S)-L12| 27             | 46.54   | 7              |
| 4     | (S)-L13| 37             | 27.73   | 22             |
| 5     | (S)-L14| 33             | 50.50   | 19             |
| 6     | (S)-L15| 46             | 10.90   | 24             |
| 7     | (S)-L16| 37             | 20.80   | 20             |
| 8     | (S)-L17| 37             | 27.73   | 22             |
| 9     | (S)-L18| 40             | 18.82   | 22             |

Determined by ¹H NMR spectroscopy using 1,3,5-trimethoxybenzene as internal standard. b Determined by HPLC (Chiralpak IB hexane/isopropyl alcohol).

in which the weaker donor atom of the mono-oxidized H₈-BINAP(O) was substituted by different functional groups. When the chiral ligand contained an ester group (L11) reactivity decreased while the product obtained was almost racemic (entry 2). A similar result was observed with a ligand bearing an alcohol substituent (L12) (entry 3). Interestingly, the more coordinating amide-derivative (L13) yielded 37% of product 3aa with a moderate 27:73 er (entry 4). These results demonstrate that the oxidized phosphine is essential to achieve high enantioselectivity. To evaluate the influence of the phosphine fragment, we tested the bis-oxidized H₈-BINAP ligand (L14). This led to a racemic product in similar yield to that obtained in the absence of chiral ligand (29% of 3aa and 12% of 4aa), indicating that the phosphine moiety is necessary for coordination to the metal center. Once we had established that the combination of phosphine−phosphine oxide is the most adequate for achieving high reactivity and enantioselectivity, we decided to investigate the influence of the substituents in both of these groups. A ligand containing the P(O)(4-methoxyphenyl), fragment (L15) gave analogous results to H₈-BINAP(O) ligand (L10) in terms of both reactivity and er (entry 6). However, when the phenyl groups were replaced by 3,5-dimethylphenyl groups (L16), both reactivity and enantioselectivity decreased (entry 7). The same scenario, but with even lower er, was observed when the aryl groups of the oxidized phosphine were replaced by alkylic cyclopentyl fragments (L17) (entry 8). Finally, replacing the phenyl groups in the PPh₃ fragment with the more electron-donating 4-methoxyphenyl substituents (L18), led to only a moderate enantiomeric ratio (entry 9).

2.3. Enantioselective C–H Activation of (η⁶-Fluoroarene)Cr(CO)₃ Complexes. With the optimized conditions for the asymmetric arylation reaction, we set out to explore the generality of the methodology with respect to the fluoroarene chromium complex (Scheme 4a). Unsubstituted fluoroarene chromium derivatives showed good reactivity and er (3aa,ab). The presence of a methyl substituent on the fluoroarene did not produce a significant difference in terms of yield or enantiomeric ratio (3bb); however, when the substituent was an ester or a methoxy group a slight decrease in the enantiomeric ratio was observed (3bc,db). Interestingly, when the fluoroarene core contains a masked aldehyde in the form of a 1,3-dioxane group, the enantioenriched product was obtained in a 97:3 enantiomeric ratio (3eb). Different protecting groups for the aldehyde gave similar results of yield and enantioselectivity to 1,3-dioxane (3ff,gb,gc). The absolute configuration of the planar-chiral products could be unambiguously confirmed by X-ray diffraction analysis of a monocrystal of enantioenriched 3aa, showing that when (R)-L10 is used as a chiral ligand, the major isomer obtained is (S)-3aa (Figure 2).

Then, we turned our attention to the effect of substitution at the iodoarene coupling partner (Scheme 4b). A variety of functional groups at the para- and meta-positions of iodoarenes, including electron-donating and electron-withdrawing substituents, were tolerated, allowing the corresponding chiral biaryl chromium complexes 3 in moderate yield and high enantioselectivities. The reaction is compatible with carbonyl functionalities such as ester, ketone, and aldehydes (3ea,ek,el). Nitrogen-containing substituents, such as nitro (3ed) and cyano (3ec), can be present on the iodoarene. The reaction is also compatible with Br and Cl substituents (3eg,em), which would allow for further functionalization via cross-couplings, CF₃ substituents (3ef) and 3,5-disubstituted iodoarenes (3eo). For p-substituted iodoarenes, higher er values are obtained with electron-poor arenes (3ea–3eg) than with electron-rich derivatives (3ei). However, similar enantioselectivities were observed for m-substituted iodoarenes regardless of their electronic properties (3ek–3en). On the other hand, ortho-substituted iodoarenes showed low reactivity (3ap,aq).

Analysis over time of the reaction of 1e with 2b shows that at low conversions the product 3eb is formed in 92:8 er. However, the er increases steadily throughout the reaction, in parallel to the formation of bisarylation product 4eb, with er reaching 98:2 when 27% of bisarylated 4eb has been formed (see SI). This suggests that the observed er values in Scheme 4 are the result of an asymmetric C–H arylation step compounded with a kinetic resolution of the product.
2.4. Mechanistic Studies. 2.4.1. Role of Ag(I) Salts in Asymmetric Arylation of (Fluoroarene)Cr(CO)₃ Complexes. Recent studies by our group on Pd-catalyzed direct functionalization of aryl C−H bonds in the presence of silver salts revealed that phosphine ligated silver carboxylate can metalate the C−H bonds in arenes bound to a Cr(CO)₃ fragment, the resulting arylsilver(I) complex is then proposed to transfer its aryl moiety to a palladium intermediate. Similar conclusions were also drawn by Sanford and Hartwig, in the case of thiophenes and (poly)fluoroarenes, and we have recently exploited this activation mode to develop the first direct α−arylation of benzo[b]thiophenes and thiophenes at room temperature.

As this reaction did not proceed in the absence of silver (Table 1, entry 8), we hypothesized that in this case the silver salt would also be involved in the C−H bond cleavage step. To test this hypothesis, we studied the H/D exchange of 1e with 10 equiv of D₂O in the presence of different combinations of additives (Table 4). No deuteration was observed when 1e was submitted to the standard reaction conditions in the absence of the silver salt (Table 4, entry 1). Deutered complex d₁-1e was not detected when the reaction was carried out only in the presence of Ag₂CO₃ (entry 2), or in combination with the chiral ligand L₁₀ with or without the carboxylic acid (entries 3−4). Importantly, in entries 2−4, the silver salt was appreciably insoluble in toluene. On the other hand, addition of TMP to Ag₂CO₃ led to formation of 32% of d₁-1e (entry 5). This is consistent with a higher solubility of the Ag-salt either through coordination or increased solvent polarity. Addition of 5.5 mol % of L₁₀ led to an increased H/D exchange of 54% (entry 6), consistent with an enhanced rate of C−H activation of the Ag-L₁₀ complex. Similarly enhanced H/D exchange was obtained when both L₁₀ and the carboxylate were added in the presence of TMP (entry 7). Interestingly, ¹H and ³¹P NMR analysis of the reaction mixture in entry 7 revealed the presence of a Ag-L₁₀ complex. These results are consistent submitted to the standard reaction conditions in the absence of the silver salt (Table 4, entry 1). Deutered complex d₁-1e was not detected when the reaction was carried out only in the presence of Ag₂CO₃ (entry 2), or in combination with the chiral ligand L₁₀ with or without the carboxylic acid (entries 3−4). Importantl
with a silver(I)-mediated C–H activation step in operation under the present reaction conditions that likely involves a coordinated L10 ligand.

Reaction of silver carboxylate 8 with 1 equiv of (R)-H8-BINAP(O) ((R)-L10) in CH2Cl2 at room temperature afforded the phosphine-ligated Ag(I) carboxylate 9 in 96% yield (eq 1). Its structure was confirmed by X-ray diffraction analysis (Figure 3). 1H and 31P NMR analysis of 9 matched with those observed in the reaction mixture of Table 4, entry 7, confirming the presence of the Ag-L10 complex. Furthermore, 31P NMR analysis of the reaction of 1e with 2i under our standard conditions, after 2 h (Figure 4a), clearly showed resonances corresponding to the (R)-H8-BINAP(O)-ligated silver carboxylate 9, strongly suggesting its participation in the catalytic cycle (Figure 4b).

Interestingly, another smaller set of signals, presumably corresponding to a PdL* compound, were present in the 31P NMR spectrum of the reaction mixture (Figure 4a). We speculated that these could correspond to Pd-complexes 11 or 12 (Scheme 5). Complex 11 was synthesized via the Buchwald-type palladium derivative 10,41 which underwent smooth oxidative addition with 4-iodoanisole to give 11 (Scheme 5). The structure of 11 was confirmed by single-crystal X-ray diffraction analysis (Figure 5). 12 was prepared from 11 by reaction with AgO2CCHCy2 (8). Comparison of the 31P NMR of both 11 and 12 with those observed in the analysis of the catalytic reaction mixture, revealed that the small set of signals in the latter corresponded to 12 (Figure 4c). This analysis highlights that the ligand L10 can coordinate to both Ag and Pd in the reaction. While qualitative analysis suggests that the majority of L10 would be coordinated to Ag, it cannot be discarded that it also plays a role in the reactivity of the Pd-species.

2.4.2. Proposed Catalytic Cycle. From the experiments above and previous work in the field,27 we propose the bimetallic catalytic cycle outlined in Scheme 6 to be in

Figure 3. ORTEP plot of (R)-H8-BINAP(O)-ligated silver carboxylate 9. Selected bonds and angles: P(1)–Ag(1), 2.330(2) Å; P(2)–Ag(1), 3.463(2) Å; O(1)–Ag(1), 3.270(4) Å; O(2)–Ag(1), 2.797(5) Å; O(3)–Ag(1), 2.107(5) Å; O(3)–Ag(1)–P(1), 123.35°. All hydrogen atoms omitted for clarity.

Figure 4. 31P NMR spectra in CDCl3 of (a) reaction mixture of 1e with 2i under asymmetric catalytic conditions after 2 h; (b) (R)-H8-BINAP(O)-ligated silver carboxylate 9; (c) L*PdAr-carboxylate 12.

Scheme 5. Synthesis of H8-BINAP(O)-Ligated Palladium Complexes

Figure 5. ORTEP plot of (R)-H8-BINAP(O)-ligated IArPd complex 11. Selected bonds and angles: I–Pd, 2.6352(9) Å; Pd–P(1), 2.282(2) Å; Pd–O(1), 2.234(6) Å; Pd–C(45), 1.980(10) Å; P(1)–Pd–I, 167.66(6)°; O(1)–Pd–I, 93.88(17)°; O(1)–Pd–P(1), 87.32(18)°; C(45)–Pd–I, 85.8(3)°; C(45)–Pd–P(1), 94.1(3)°; C(45)–Pd–O(1), 174.7(3)°. All hydrogen atoms are omitted for clarity.
operation. In this mechanism, a Pd(0)-L10 complex A undergoes oxidative addition to B, which after transmetalation affords arylcarboxylate-Pd derivative C, structurally related to complex 12. In a parallel catalytic cycle, silver carboxylate E carries out C–H activation on η6-coordinated arene I to form arylsilver intermediate F, presumably by a carboxylate assisted CMD mechanism.37 Transmetalation from silver intermediate F to the palladium-arylcarboxylate C38 would form D, which would in turn release the product 3 after reductive elimination. We propose that L10 is coordinated to both Ag and Pd, throughout the process. Two possible enantiodetermining steps must thus be considered: an enantioselective C–H activation by complex E, followed by a fast transmetalation with C, or alternatively a fast reversible C–H activation, followed by a rate and enantioselectivity determining transmetalation. However, further experiments will be necessary to understand this process fully, which is further complicated by the low solubility of some of the species in toluene.

2.5. Synthesis and Derivatization of Enantioenriched Planar-Chiral (Arene)Chromium Tricarbonyl Phosphines. The presence of a C–F bond in the aromatic core of the chiral (arene)chromium complexes allows easy functionalization via a variety of nucleophilic aromatic substitutions,12 including phosphination reactions, to obtain arylphosphine derivatives easily.

Nelson and co-workers investigated the effectiveness of optically active arylmonoamine chromium complexes as ligands in asymmetric catalysis in the Pd(II)-catalyzed alkylation of allylic acetates under Trost’s conditions,8 thus demonstrating that chromium-complexed arylphosphines provide chiral equivalents of triarylphosphine ligands that are ubiquitous in late transition-metal chemistry and catalysis. To test the applicability of our approach for the synthesis of new planar-chiral phosphines we carried out the asymmetric arylation of 4-iodobenzonitrile (Scheme 6, 3ec). Reaction of the aldehyde derivative 3ec with potassium diphenylphosphide resulted in nucleophilic aromatic substitution to afford the chiral planar triarylphosphine (Scheme 7a). The protected aldehyde on 13 provides an ideal handle for further derivatization and tuning of electronic properties of this chiral phosphine. Accordingly, treatment of 13 under acidic conditions revealed the aldehyde to obtain 14, which could then be reduced by NaBH4 to the alcohol derivative 15 (Scheme 7b). Both the aldehyde in 14 and the alcohol in 15 could then be easily transformed into a variety of functionalities. Over the past few decades, chiral diphosphines have proven to be among the most useful and versatile ligands for metal-catalyzed asymmetric reactions and the design and preparation of such diphosphines remains as active an area of research as ever.35 The synthesis of C4-symmetric diphosphine ligands has long received the most attention, due perhaps to the relative ease of obtaining these molecules.44 However, studies have showed that C3 symmetry is not a necessary condition for obtaining highly enantioselective ligands.45 Our functionalized planar chiral phosphines, such as 14 and 15, are ideal starting points for the synthesis of novel classes of planar chiral diphosphines. Reaction of the aldehyde derivative 14 with p-(diphenylphosphino)benzylamine in the presence of a catalytic amount of acid afforded the diphosphine-imine derivative 16 in quantitative yield. This compound can be reduced by benzyltriphenylphosphonium tetrahydroborate to give the corresponding chiral amine-diphosphine derivative 17, providing a novel class of bidentate chiral phosphines (Scheme 7b).

3. CONCLUSIONS

In conclusion, we have developed the first protocol for catalytic direct C–H asymmetric arylation of (η6-arene)chromiumtricarbonyl complexes to afford enantioenriched planar-chiral products in one step. The development of this methodology required the synthesis of a new family of H4-BINAP derivatives, finding that H4-BINAP(O) was the most suitable chiral ligand for the reaction. Optimized catalytic conditions were applied to a variety of iodoarenes and (fluoroarene)Cr(CO)3 complexes affording the corresponding chiral products in good yield and excellent enantioselectivity. Mechanistic studies suggest that the reaction proceeds through a Pd/Ag bimetallic double catalytic cycle where the C–H
activation is carried out by Ag. These enantioenriched arylcomplexes can be used for the synthesis of chiral planar monodentate phosphines and a new class of chiral planar bidentate phosphines. The application of these new chiral ligands to asymmetric catalysis is currently under investigation.

**ASSOCIATED CONTENT**

Supporting Information

The Supporting Information is available free of charge on the ACS Publications website at DOI: 10.1021/acscatal.9b00918.

Experimental procedures and characterization data (PDF)
- Data for C₄₄H₄₀OP₂ (CIF)
- Data for C₅₈H₅₈AgO₂P₂C₆H₁₀O, 0.78(H₂O) (CIF)
- Data for C₁₇H₁₆CrF₂O₅ (CIF)
- Data for C₃₂H₂₄CrNO₅P, C₅H₁₂ (CIF)
- Data for C₃₅H₃₆O₇P₄, CH₂Cl₂ (CIF)

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Notes

The authors declare no competing financial interest.

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