Supporting Information for

Neoteric solvents for exploratory catalysis: hydrophosphination catalysis with CHEM21 solvents

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Contents

General considerations

Air- and moisture-sensitive reactions were carried out under a dry N_2 atmosphere using an M. Braun glovebox or standard Schlenk techniques. Diphenylphosphine was synthesized according to a modified literature procedure.[1] All other reagents were acquired from commercial sources and dried by conventional means as necessary. Ethanol and Cyrene were degassed and freeze-pump-thawed. 2-methyltetrahydrofuran (2-MeTHF) and cyclopentylmethyl ether (CPME) were distilled from sodium -benzophenone ketyl. Ethyl acetate and DMSO were dried over calcium hydride and then distilled. Heptane was degassed, stirred over sodium, and filtered through basic alumina. All solvents were stored over 3Å or 4Å molecular sieves under inert atmosphere prior to use.

General procedure for catalytic experiments

In an N2 filled glovebox, 0.38 mmol of Ph2PH and 0.38 mmol of unsaturated substrate was added to a shell vial containing 0.019 mmol of catalyst (and an internal standard where applicable). 400 μL of solvent was added to the shell vial and the contents were mixed via pipette. The resulting solution was added to an NMR tube (with an external standard where applicable) with a disposable NMR tube cap which was subsequently wrapped with parafilm and wiped with bleach. Initial 31P{1H} NMR (and 1H NMR where applicable) spectra were obtained before placing the tube in a chamber containing a Rexim G23 UV-A (9 W) lamp. 31P{1H} spectra were collected periodically to determine reaction progress. Conversions were determined by integration of 31P{1H} NMR spectra to those of staring materials. An external standard (sealed capillary) of PPh3 was used. In reactions with DMSO-d_6, 1,3,5-trimethoxybenzene was used as an internal standard.

Note on NMR characterization

The hydrophosphination products described in this work are well characterized in the literature and have been isolated as pure substances.[1-13] Therefore, $^{31}P\{^1H\}$ NMR is used as the primary means of characterization for these reactions. As an article of convenience and in recognition of the cost, proteo-solvents were used for this reactivity, except in the case of DMSO-d_6. An external standard (sealed capillary) of PPh_3 in $CDCl_3$ was added to each reaction as a chemical shift reference and integration standard. However, some chemical shifts deviate slightly different due to the high variety of solvents in this study.

Conversions were determined by integration of $^{31}P\{^1H\}$ NMR resonances to starting materials and confirmed with an external standard of PPh_3. Common NMR resonances (ppm) include Ph_2PH = -41, PPh_3 = -6, $[Ph_2P]_2$ = -14.[9, 14]

Photo and spectral output of lamp

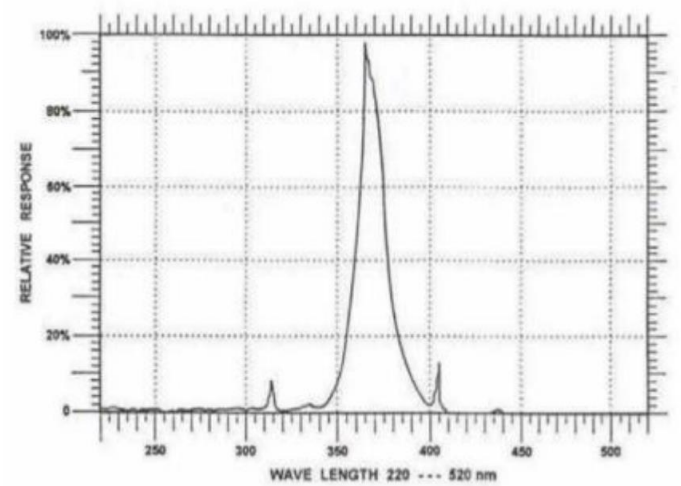


Figure S1. Spectral distribution of Rexim G23 UV-A (9W) lamp provided by manufacturer.

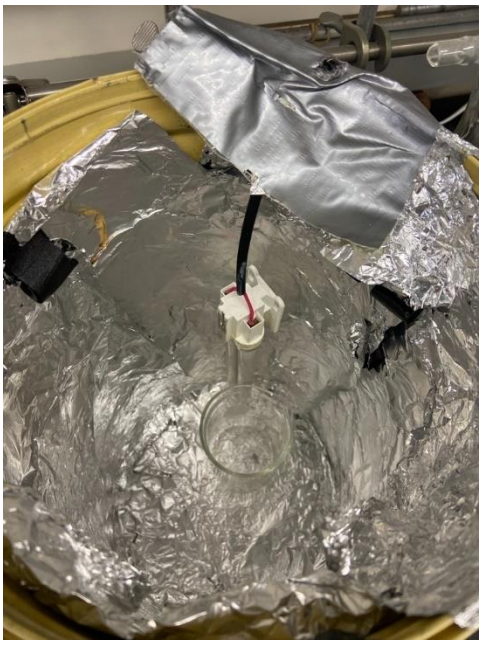


Figure S2. Photo of the photoreactor containing the Rexim G23 UV-A (9W) lamp.

Dielectric constant of solvent effect on hydrophosphination reactivity

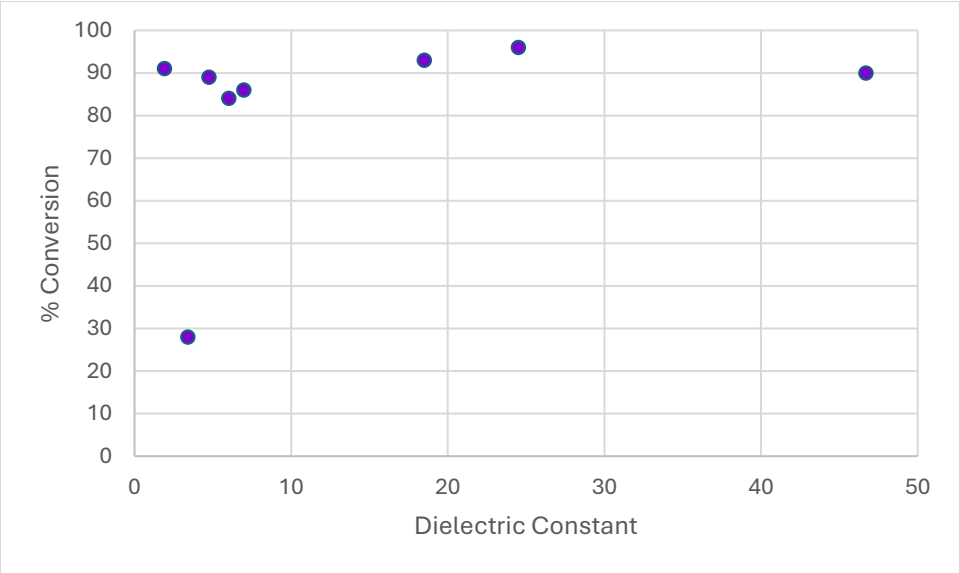
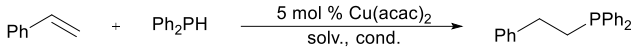


Figure S3. Plot of solvent dielectric constant versus conversion to product.

solvent	dielectric constant	conversion (%)
EtOH	24.5	96
EtOAc	6.02	84
DMSO	46.7	90
heptane	1.92	91
2-MeTHF	6.97	86
Cyrene	3.4	28
CPME	4.76	89
MEK	18.51	93

Table S1. Solvent dielectric constant and conversion to products under conditions described in text.

NMR Spectra of hydrophosphination with 5 mol % $Cu(acac)_2$



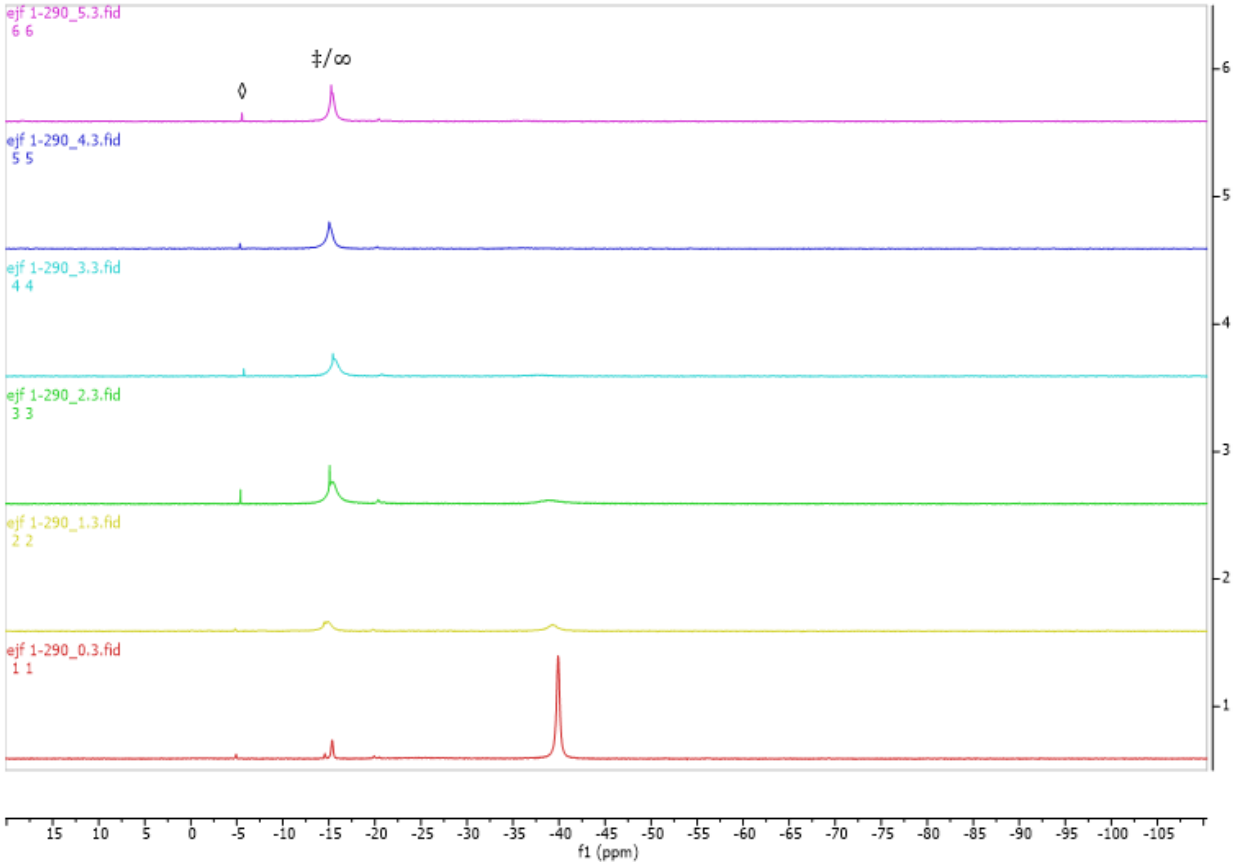


Figure S4. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

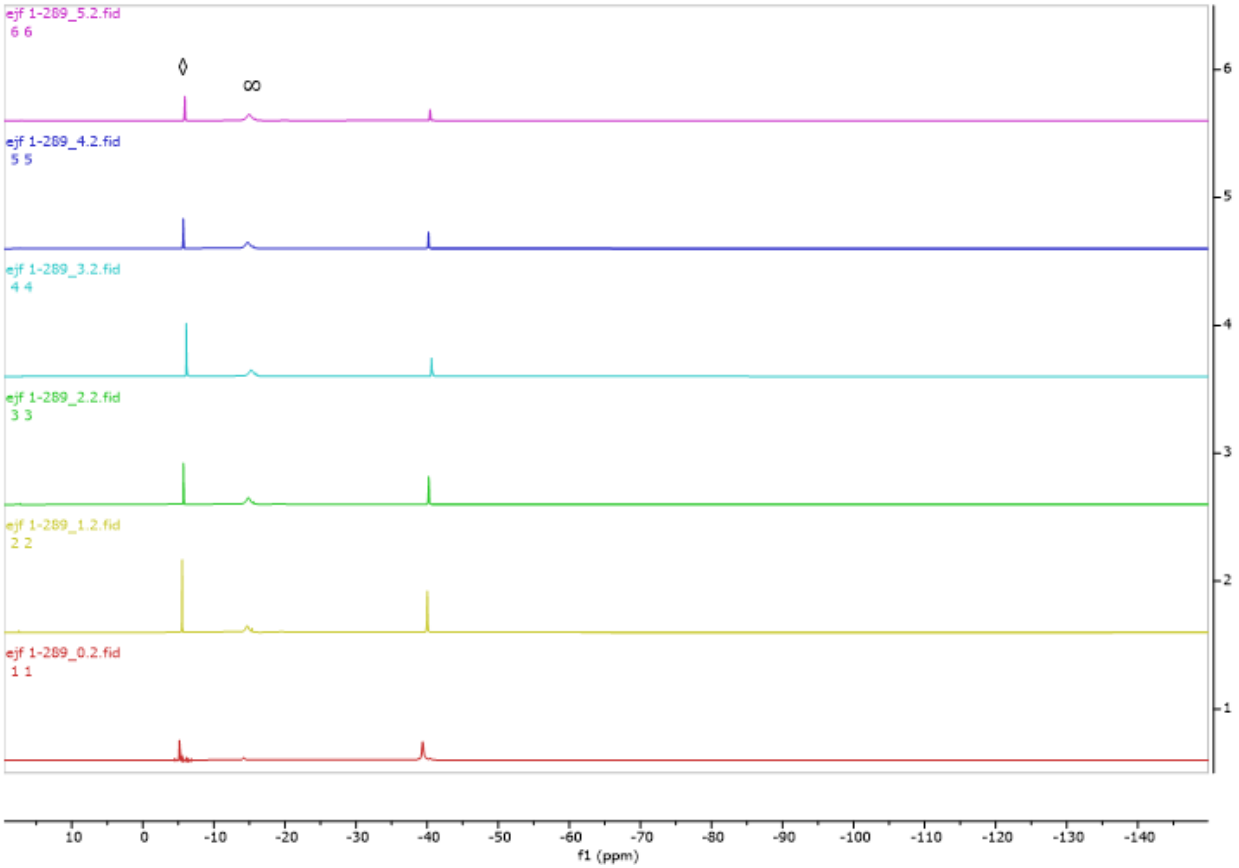


Figure S5. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

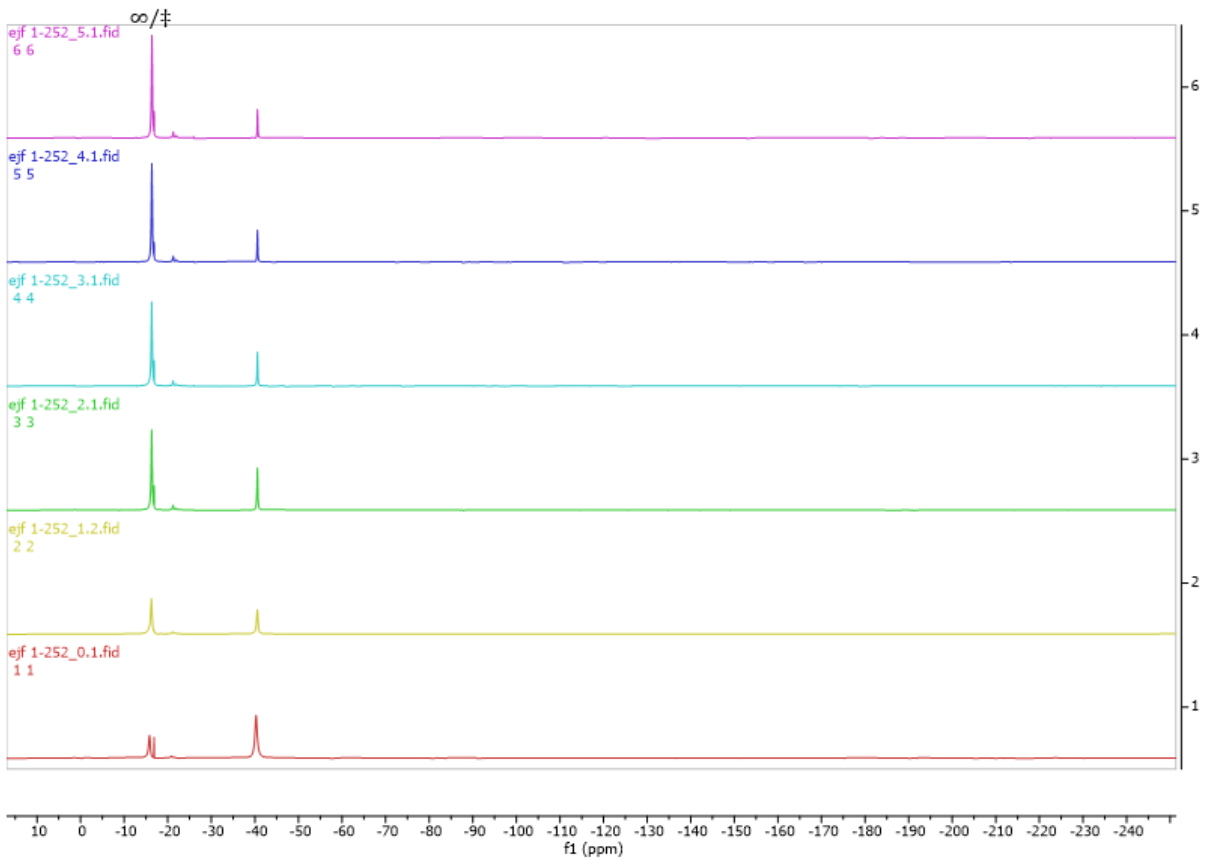


Figure S6. Stacked $^{31}P\{^{1}H\}$ NMR (DMSO-d_6, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

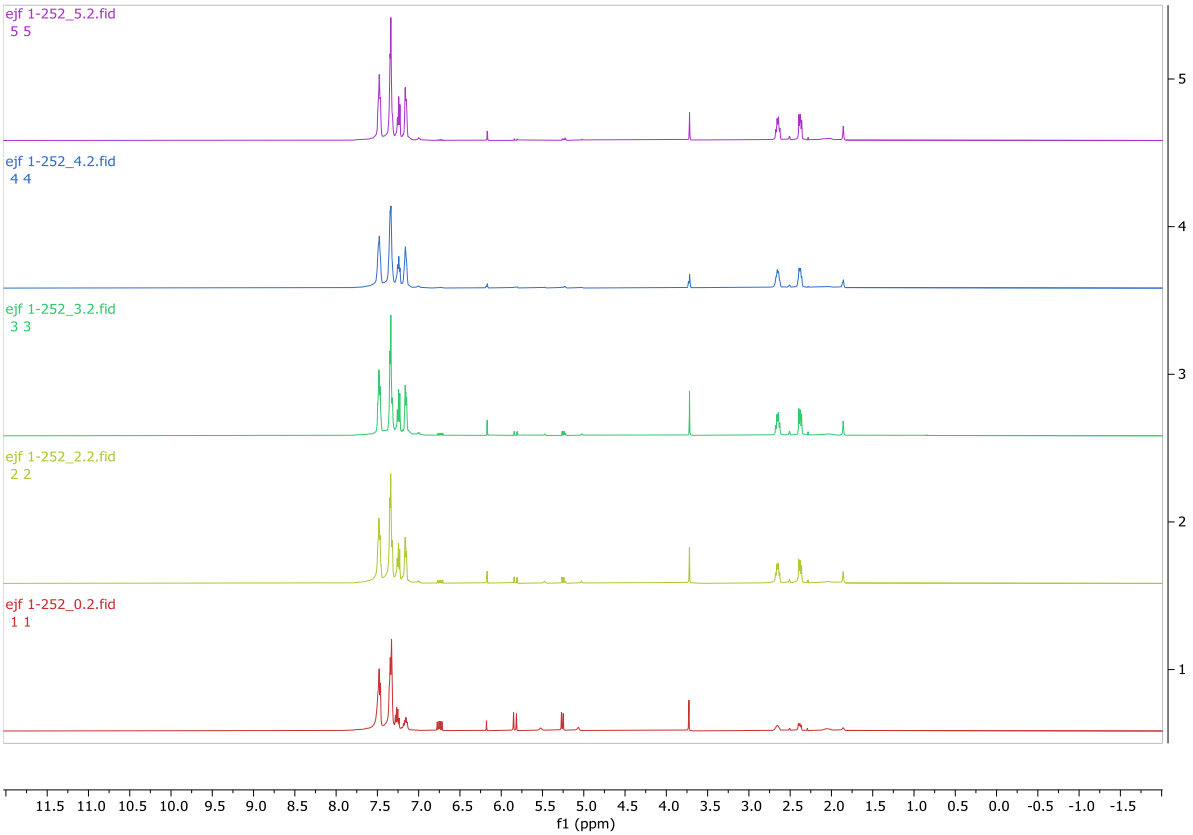


Figure S7. Stacked ^{1}H NMR (DMSO-d_6, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

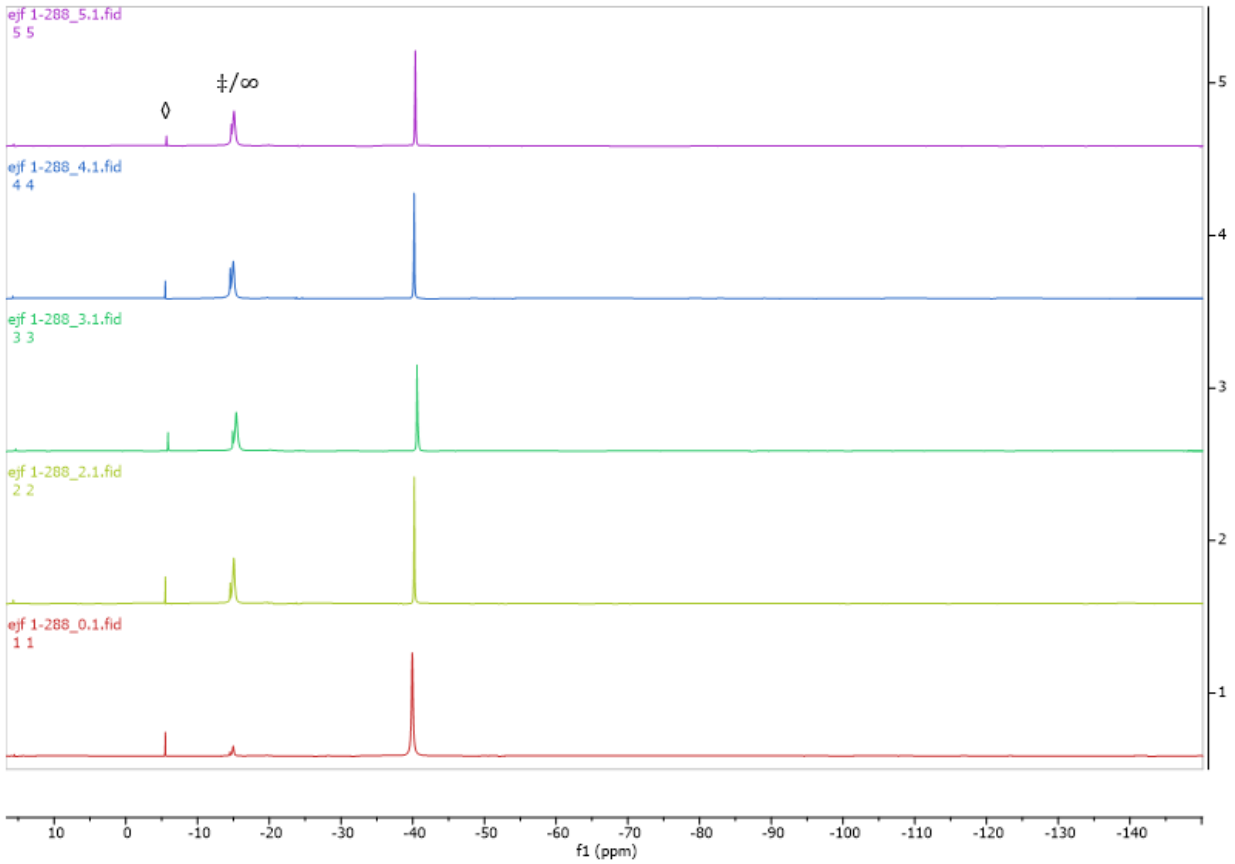


Figure S8. Stacked $^{31}P\{^{1}H\}$ NMR (heptane, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

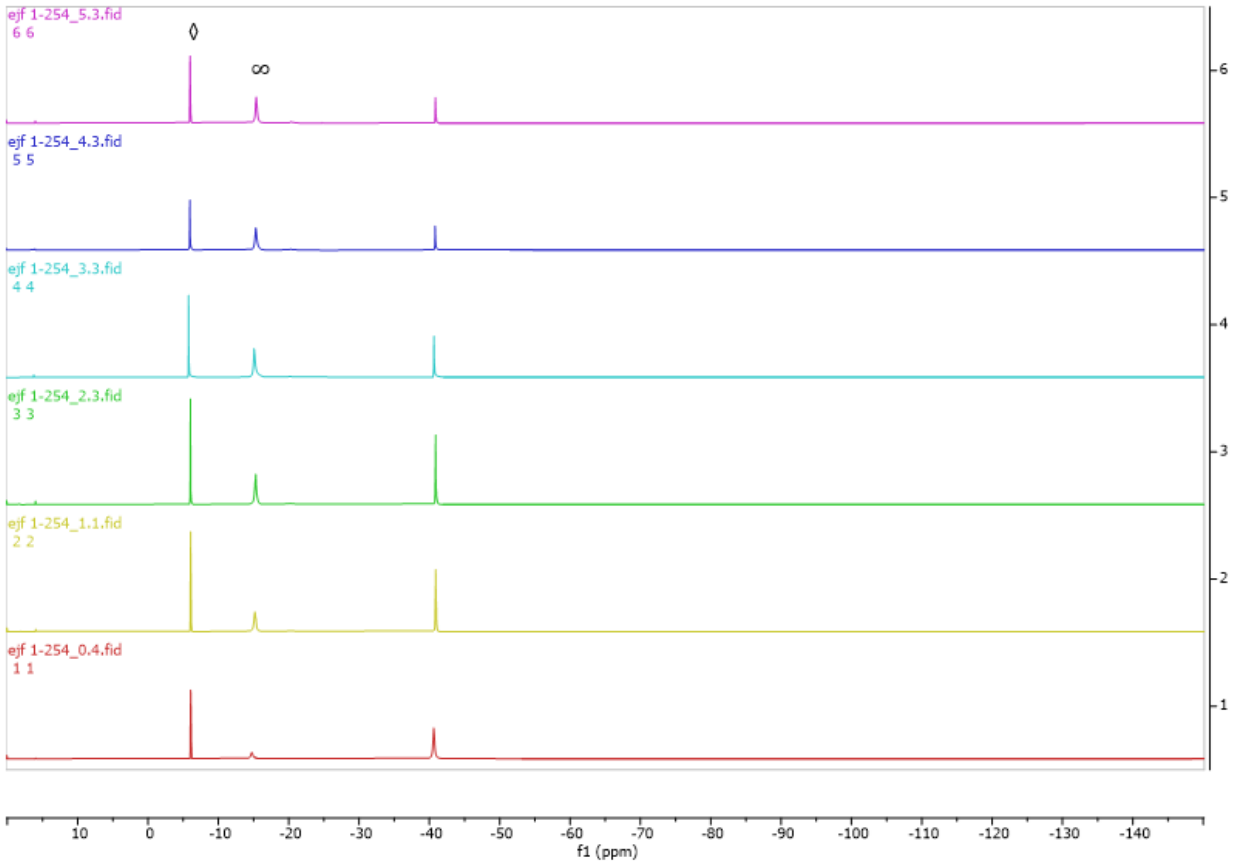


Figure S9. Stacked $^{31}P\{^{1}H\}$ NMR (2-MeTHF, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

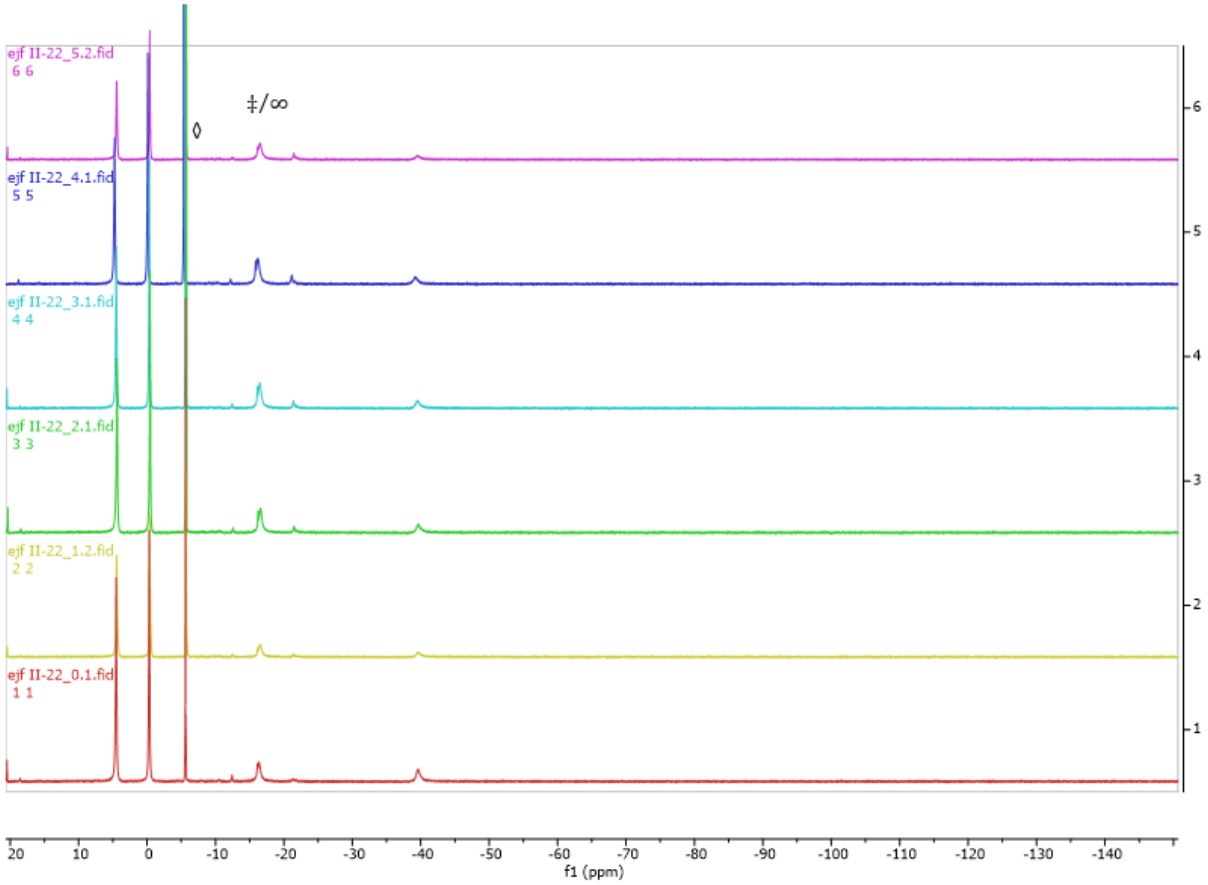


Figure S10. Stacked $^{31}P\{^{1}H\}$ NMR (Cyrene, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

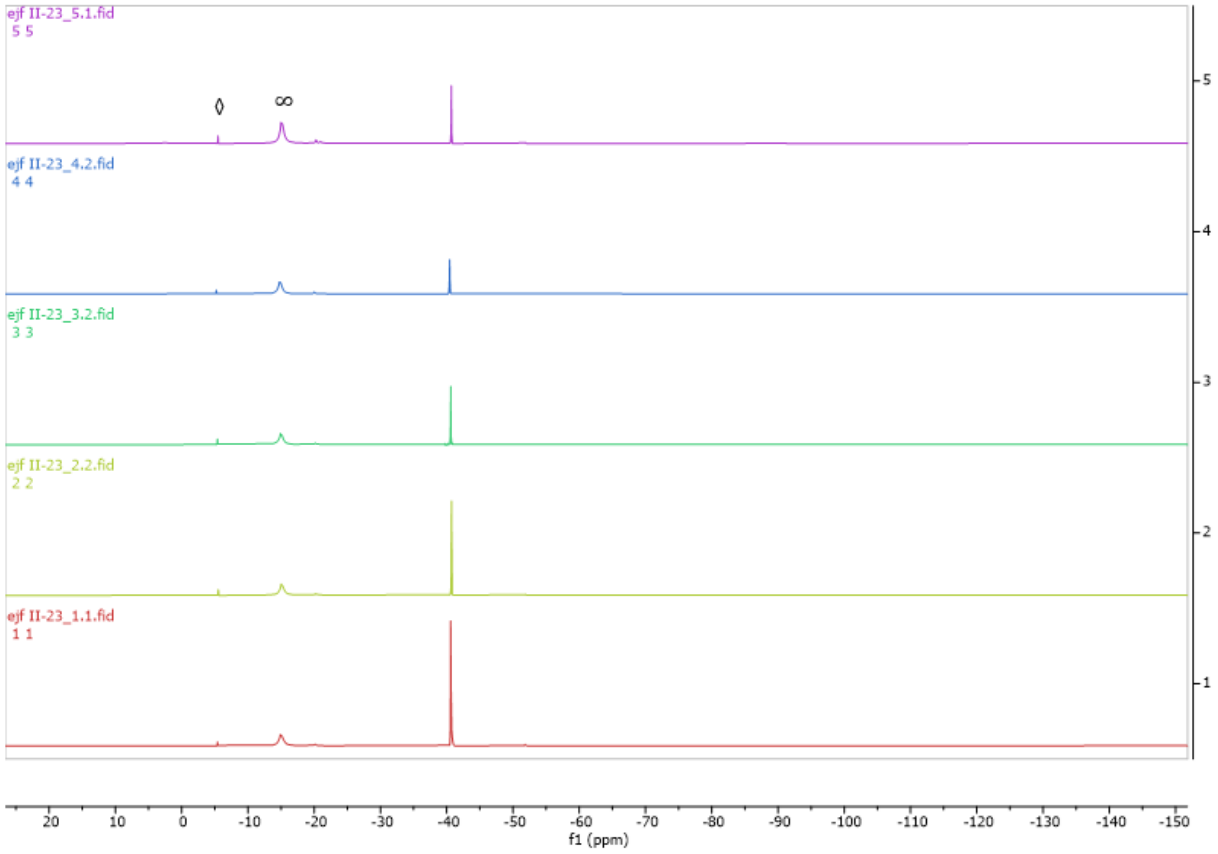


Figure S11. Stacked $^{31}P\{^{1}H\}$ NMR (CPME, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

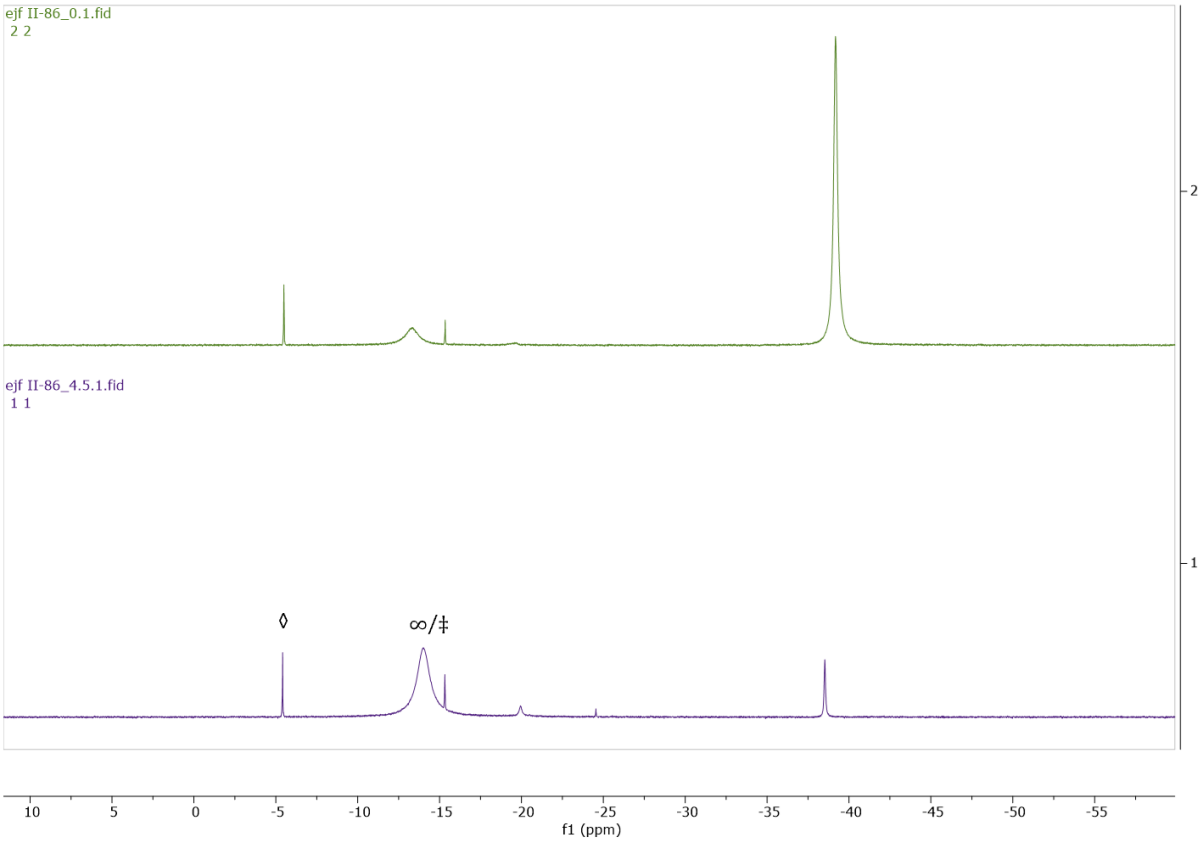
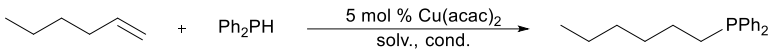


Figure S12. Stacked $^{31}P\{^{1}H\}$ NMR (MEK, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.



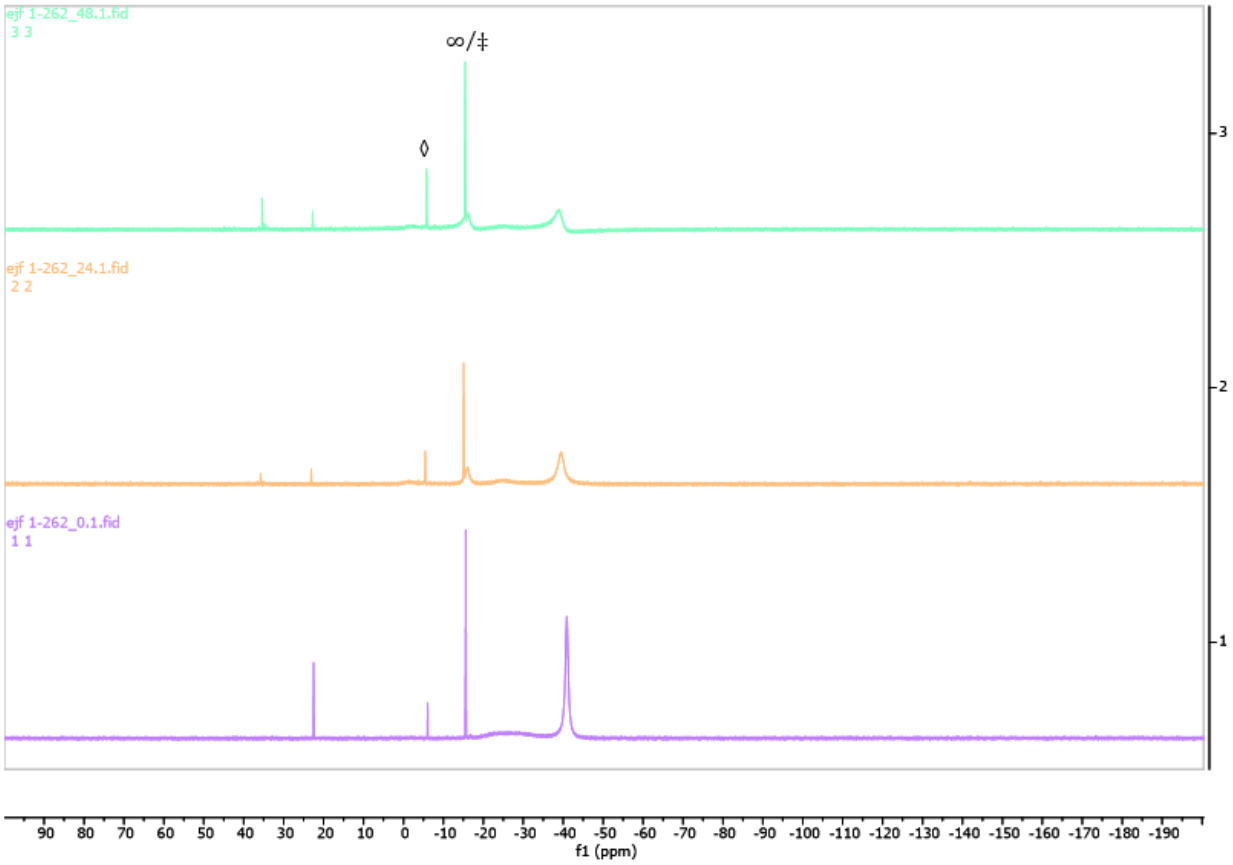


Figure S13. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

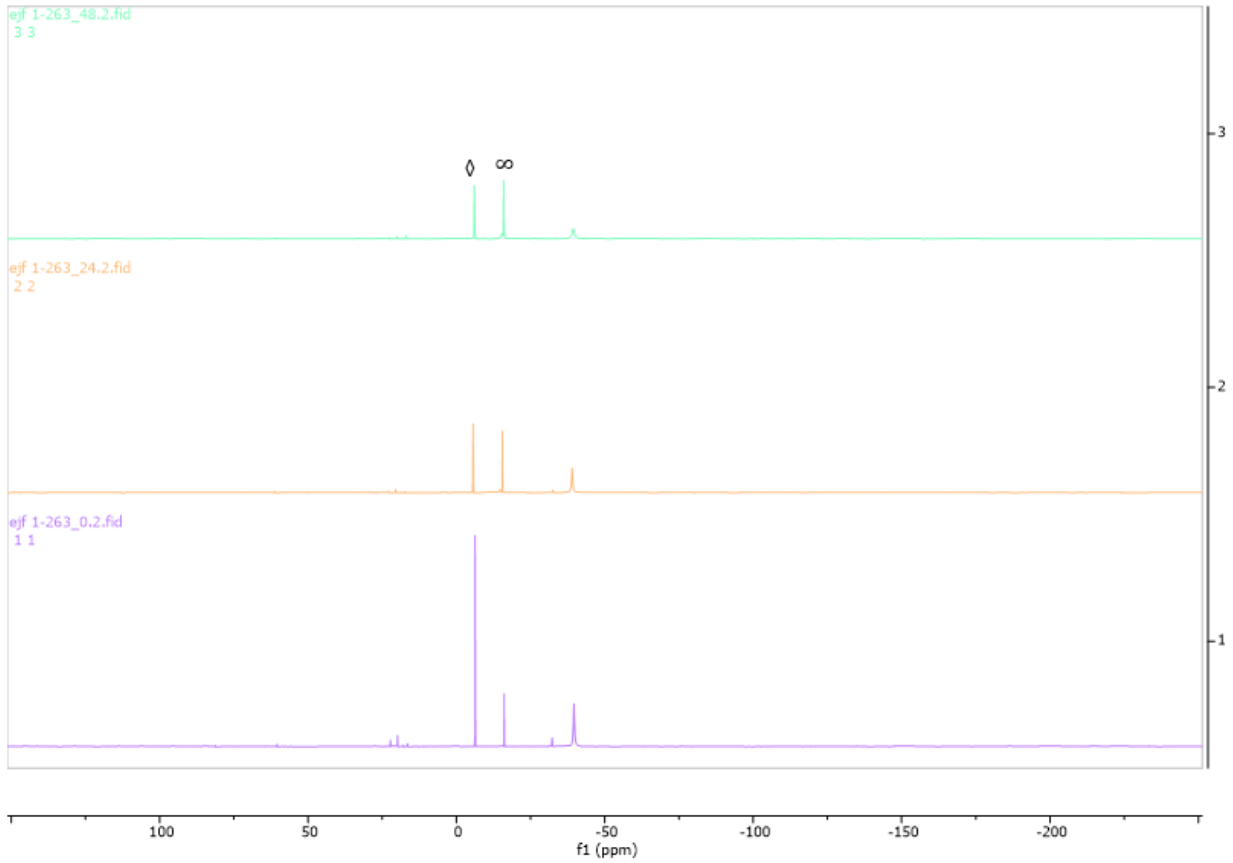


Figure S14. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

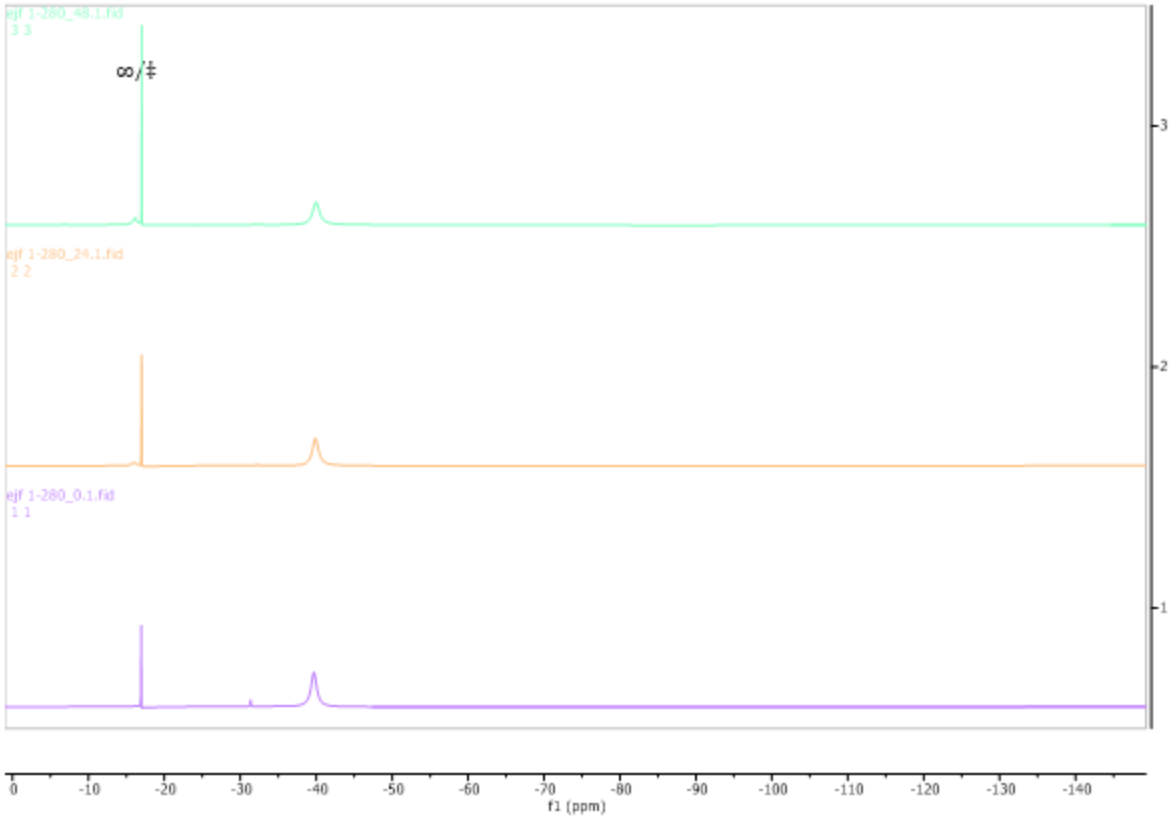


Figure S15. Stacked $^{31}P\{^{1}H\}$ NMR (DMSO-d_6, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

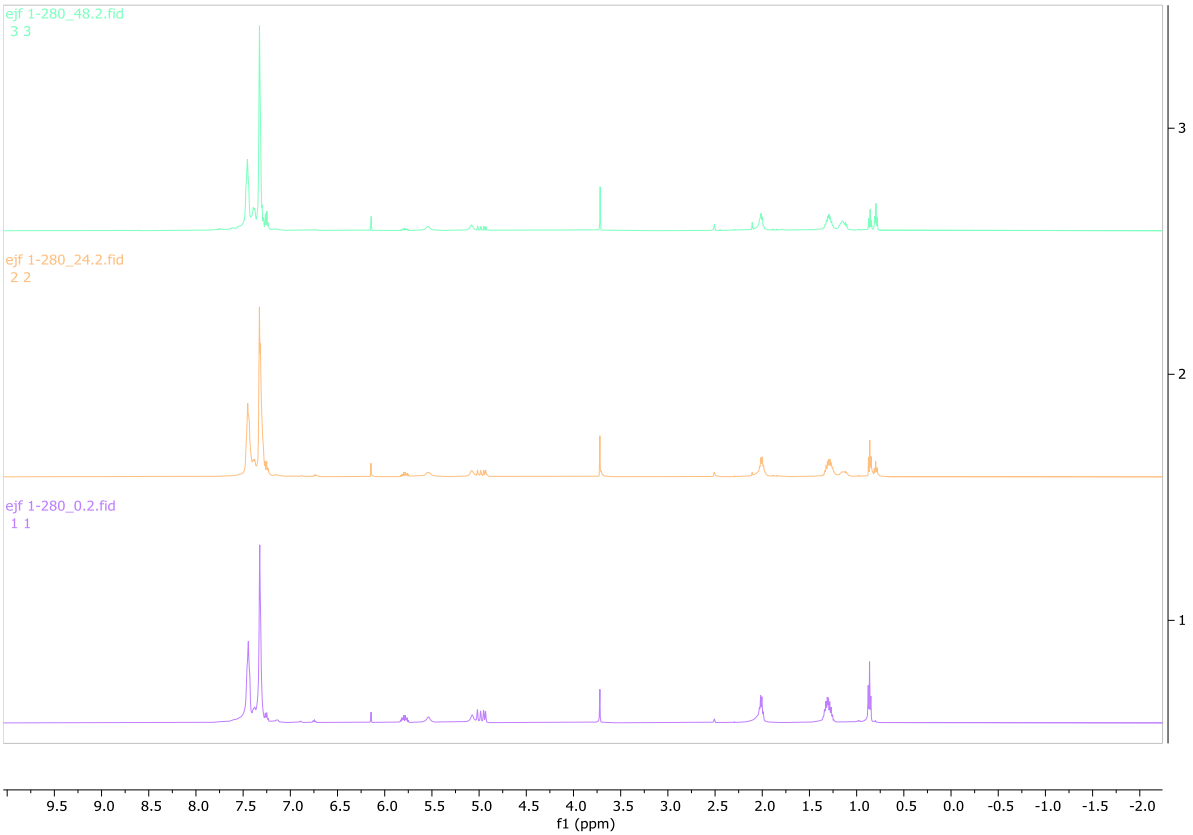


Figure S16. Stacked ^{1}H NMR (DMSO-d_6, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

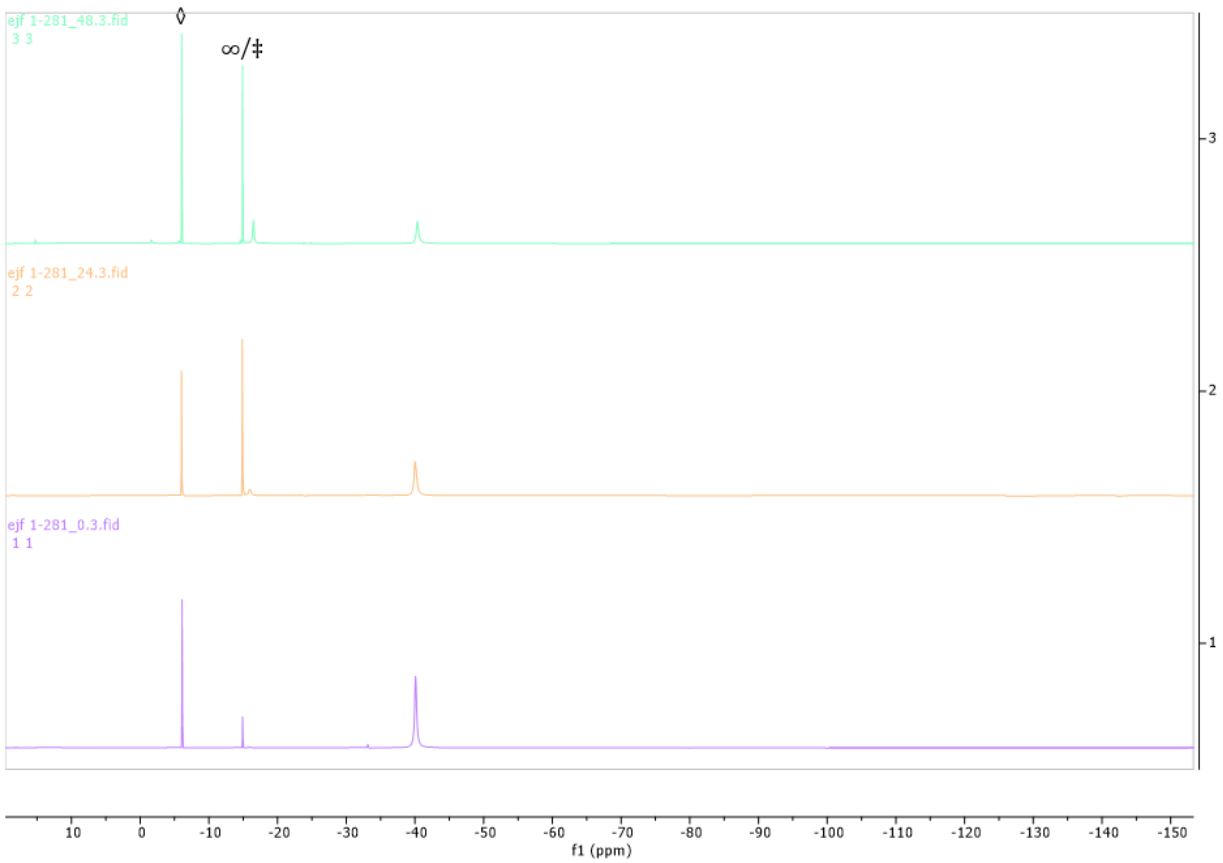


Figure S17. Stacked $^{31}P\{^{1}H\}$ NMR (heptane, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

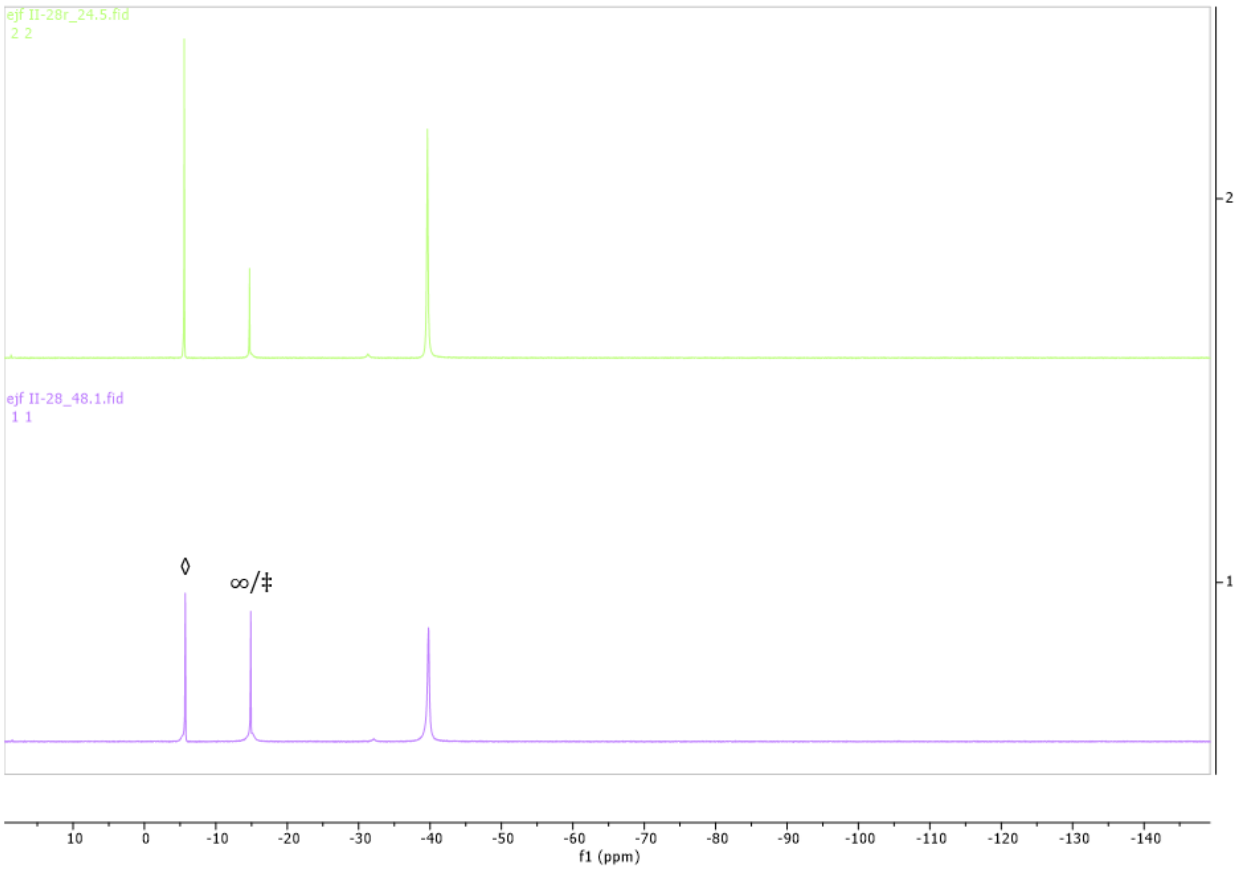


Figure S18. Stacked $^{31}P\{^{1}H\}$ NMR (2-MeTHF, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

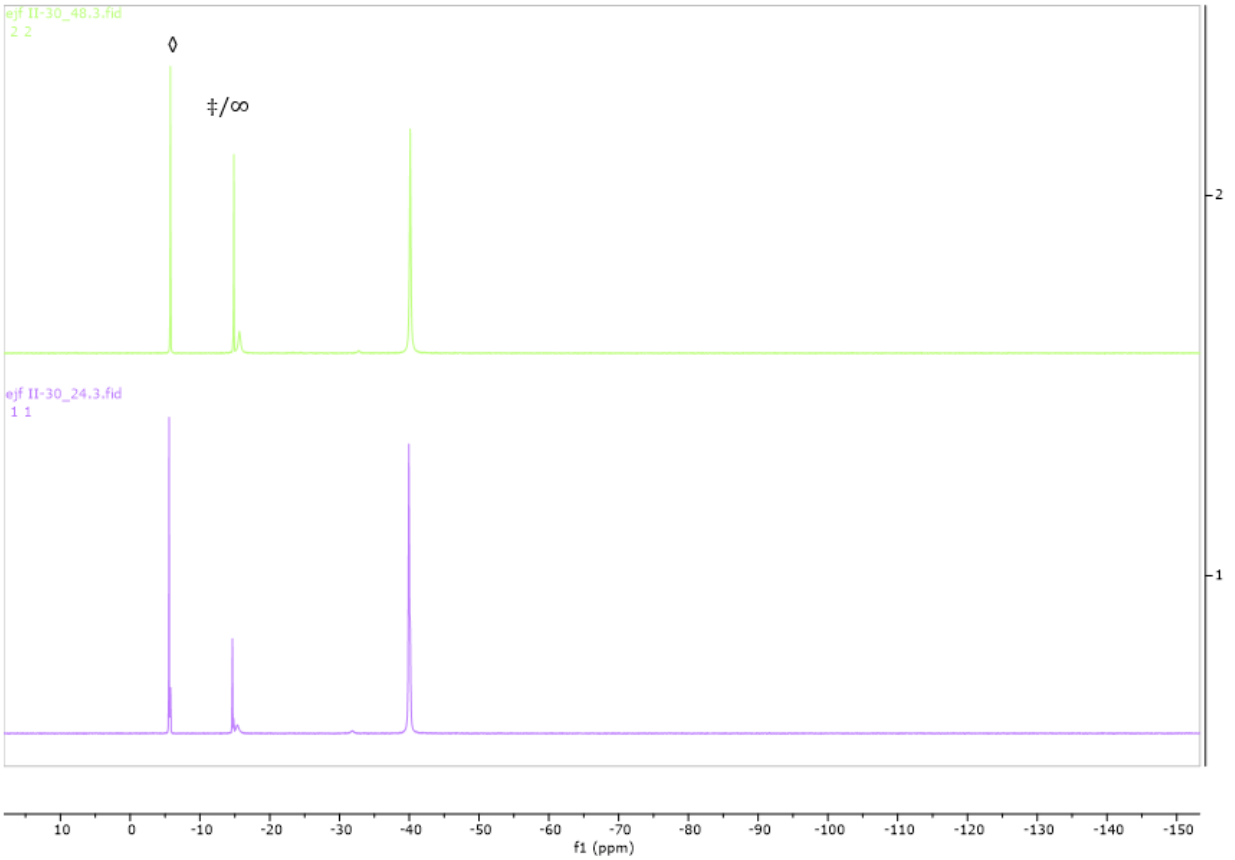


Figure S19. Stacked $^{31}P\{^{1}H\}$ NMR (CPME, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

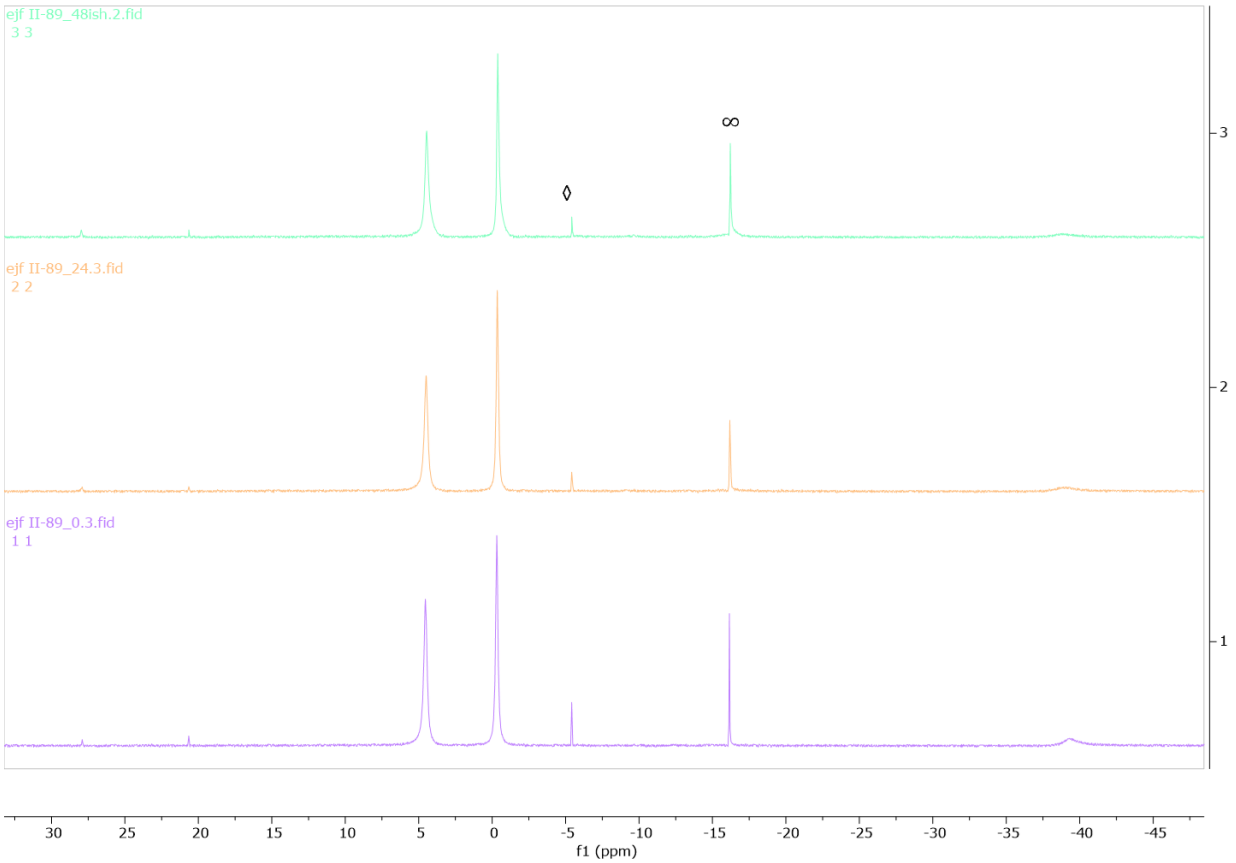


Figure S20. Stacked $^{31}P\{^{1}H\}$ NMR (Cyrene, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

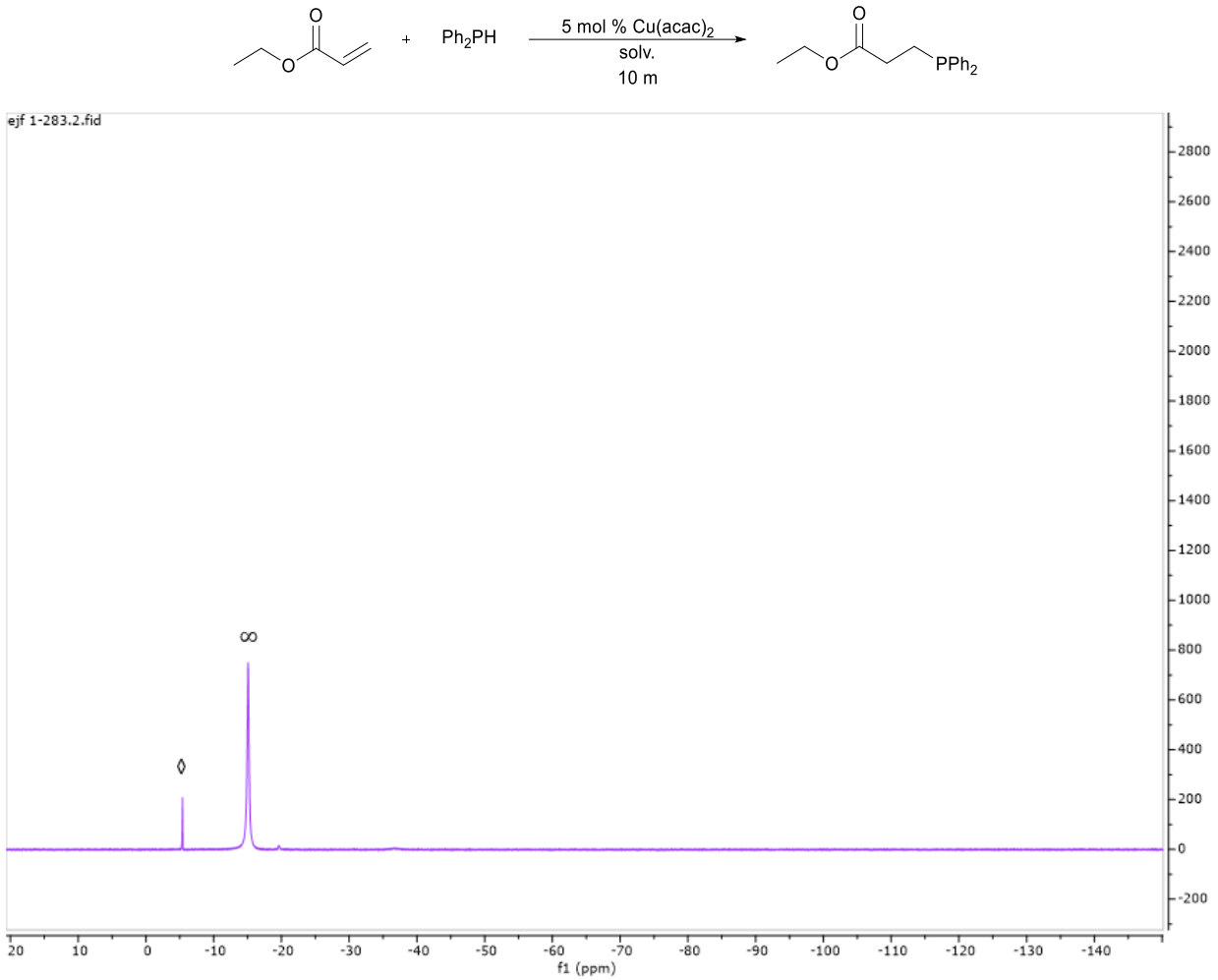


Figure S21. $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

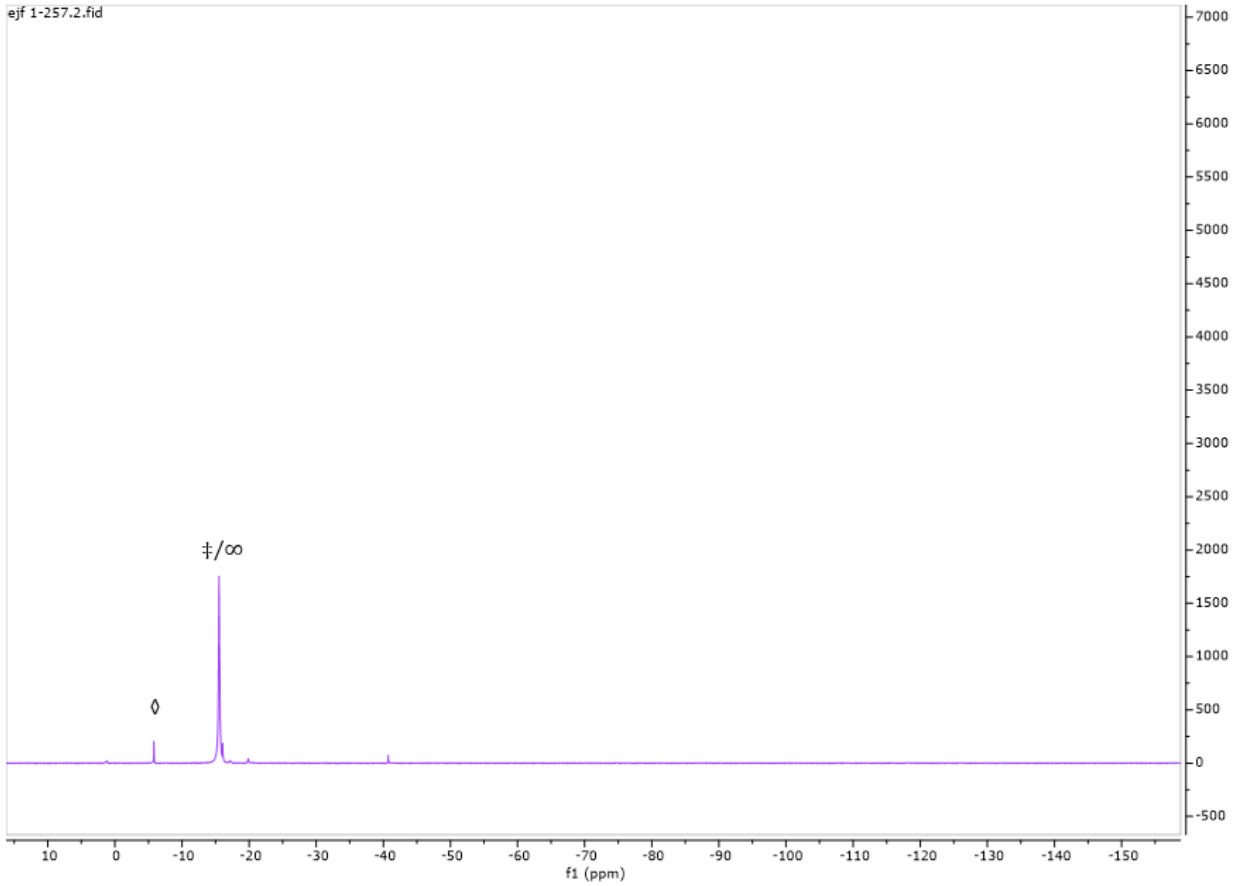


Figure S22. $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

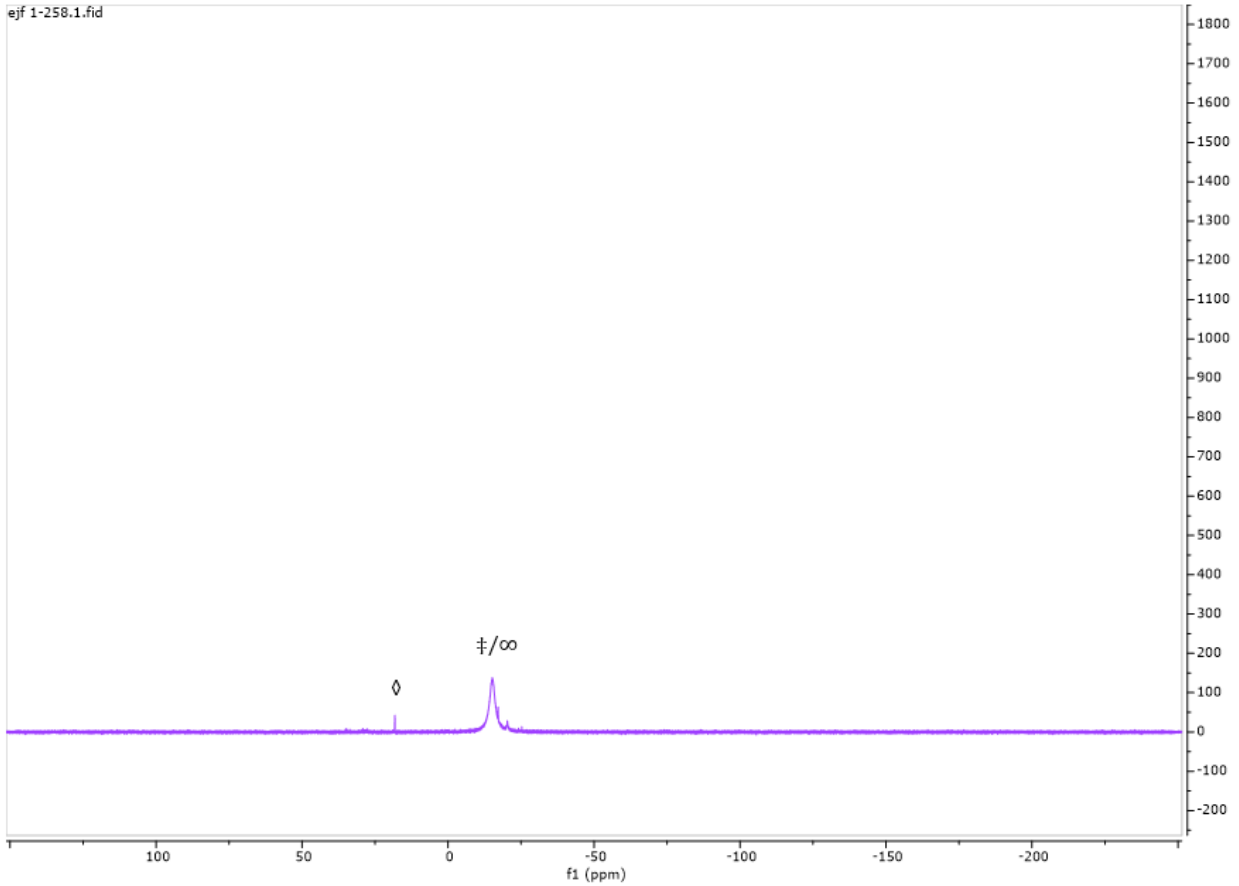


Figure S23. $^{31}P\{^{1}H\}$ NMR (DMSO-d_6, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

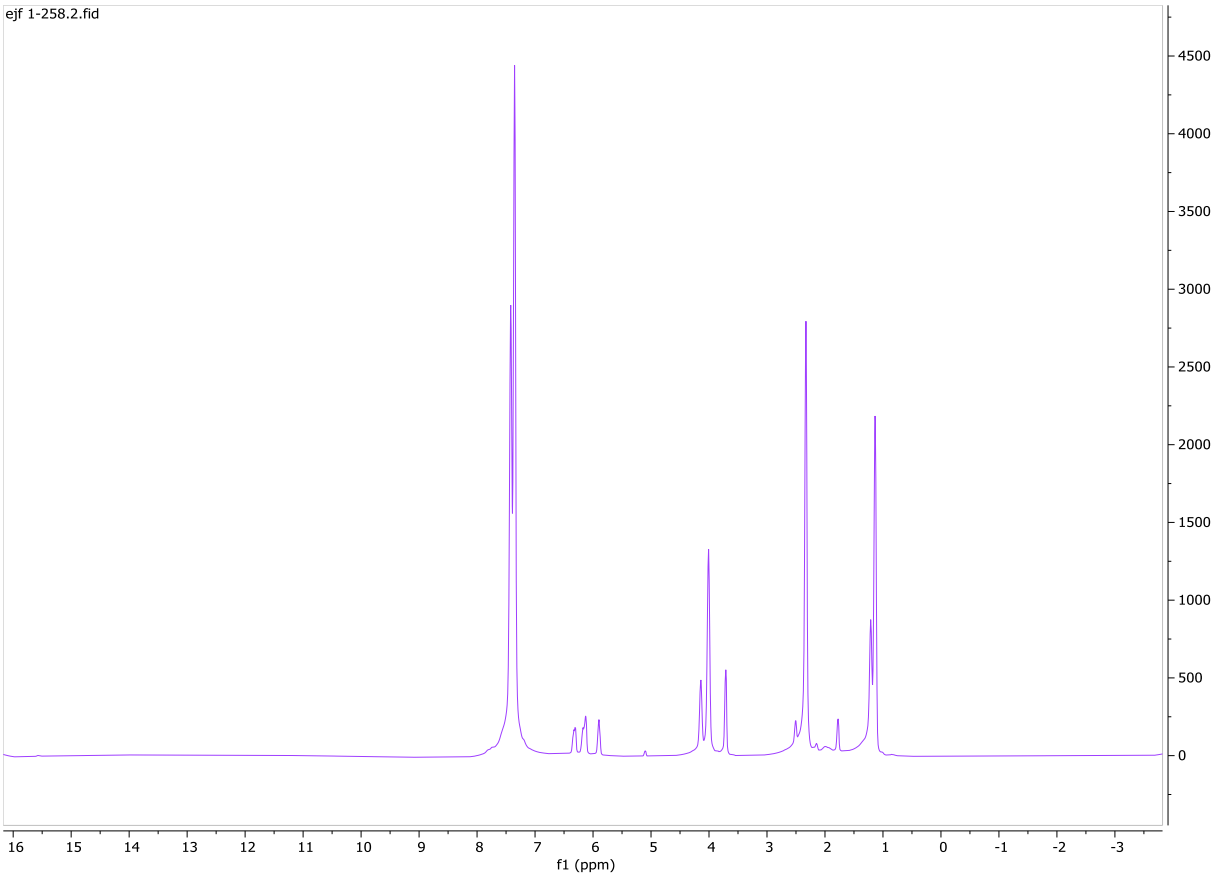


Figure S24. ^{1}H NMR (DMSO-d_6, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

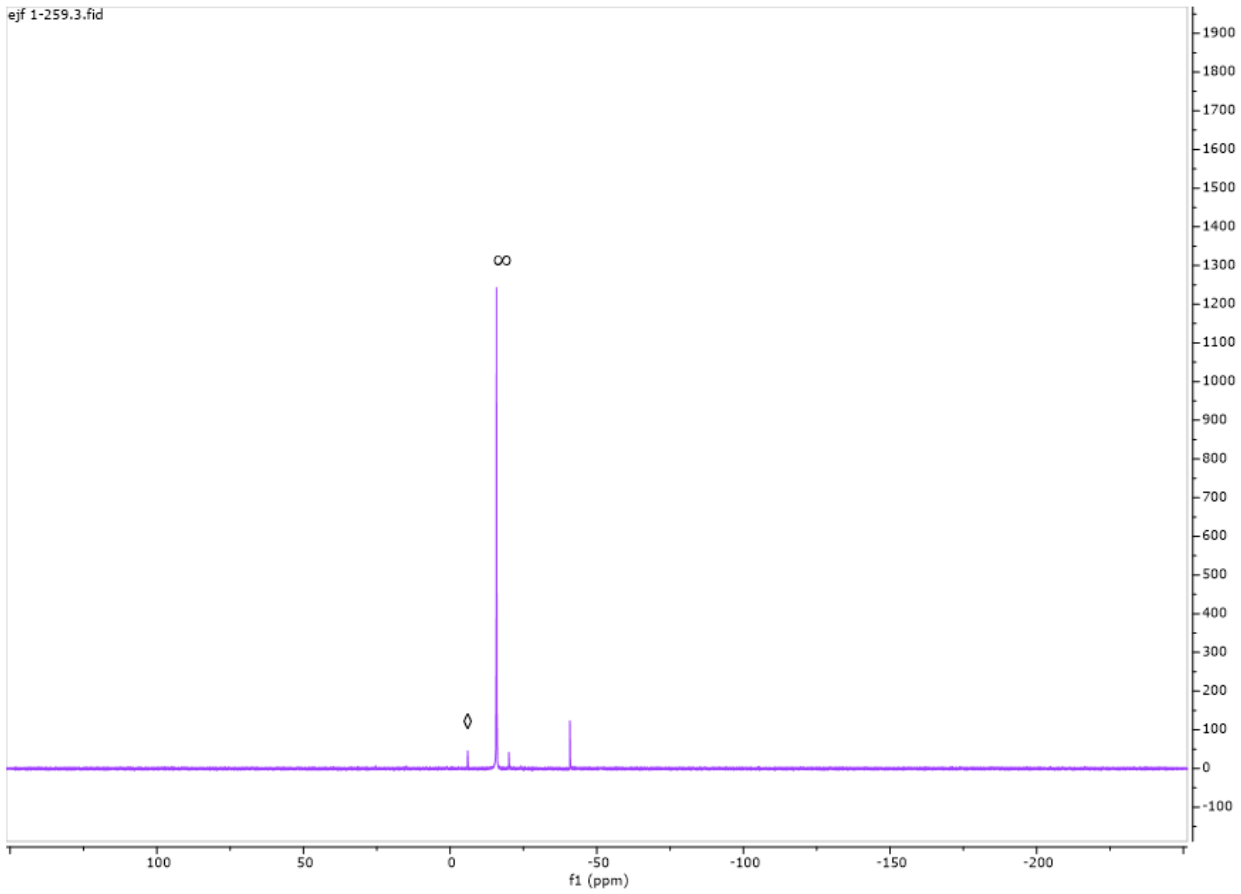


Figure S25. $^{31}P\{^{1}H\}$ NMR (heptane, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

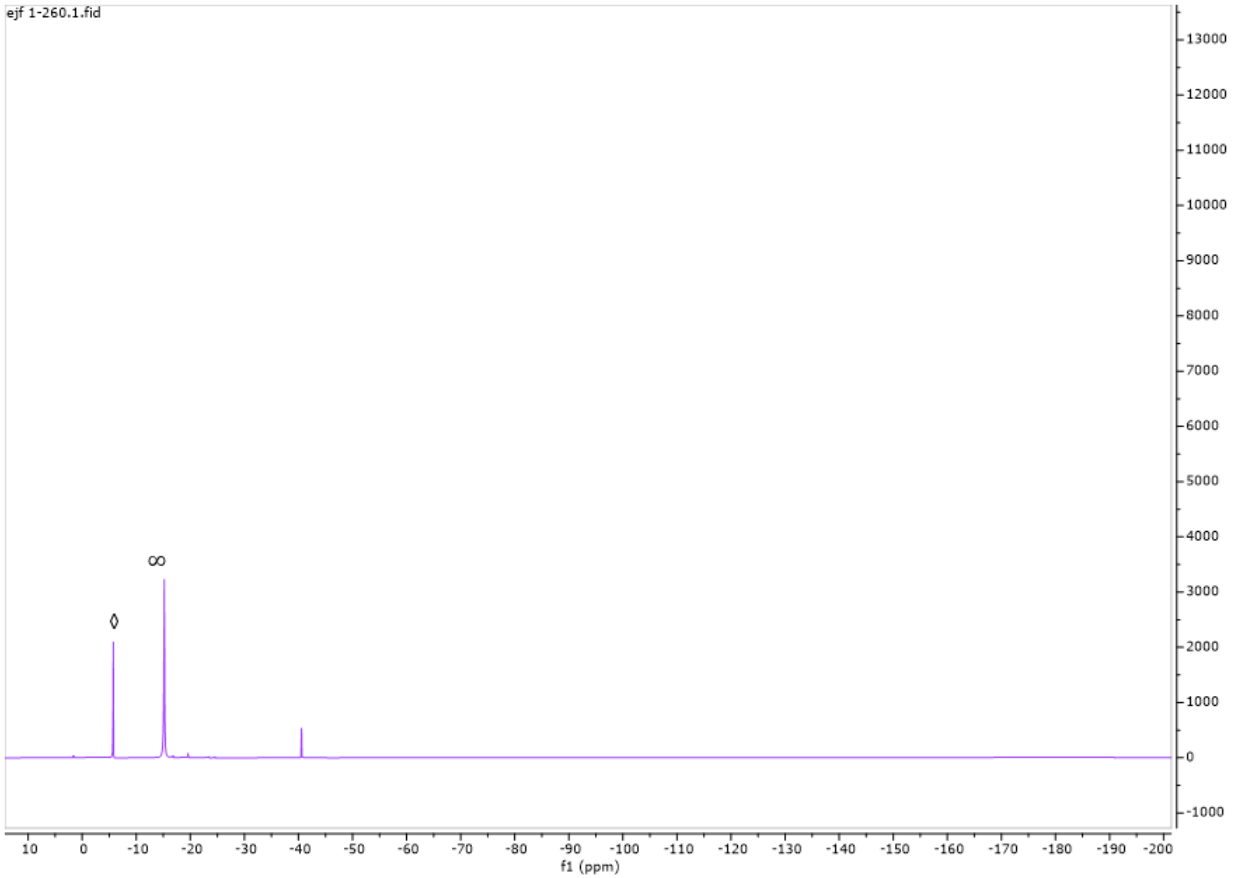


Figure S26. $^{31}P\{^{1}H\}$ NMR (2-MeTHF, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

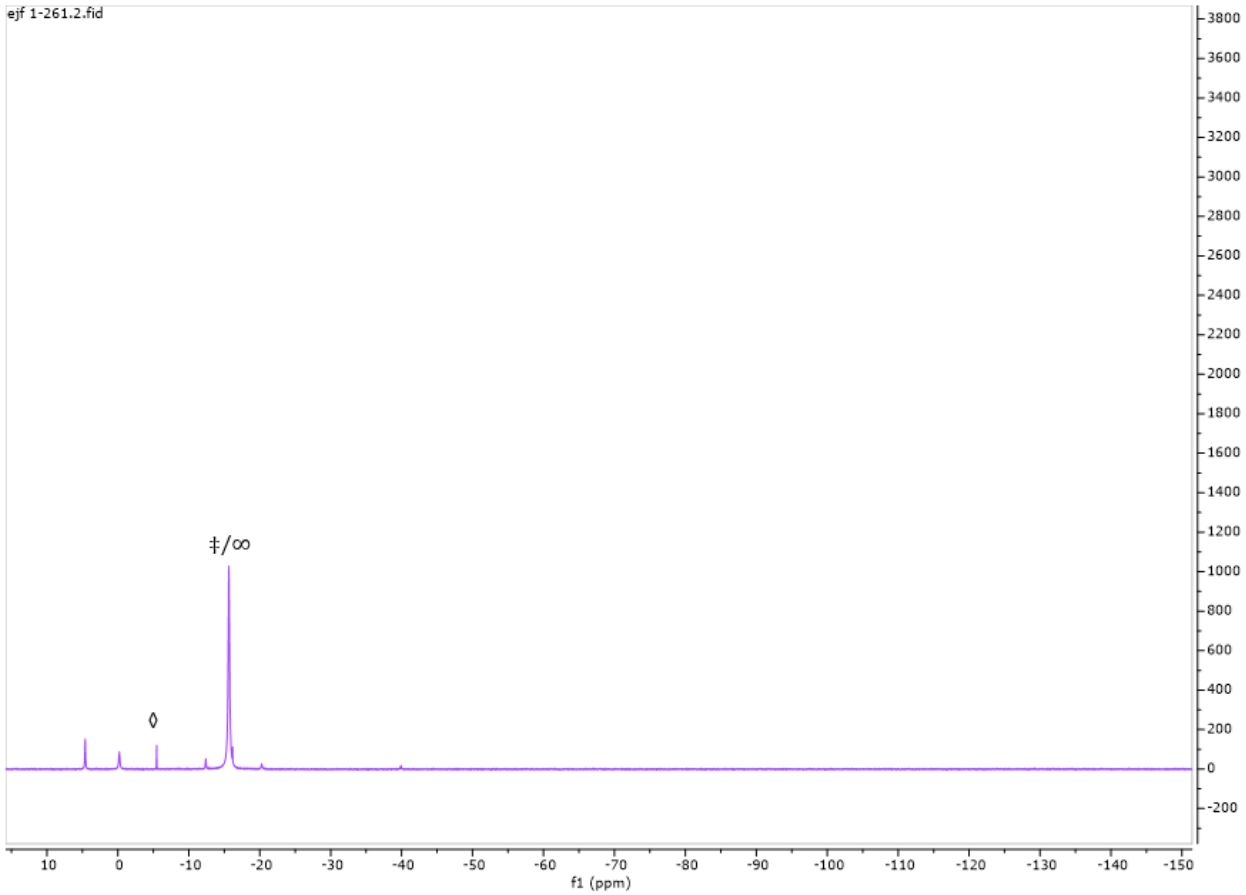


Figure S27. $^{31}P\{^{1}H\}$ NMR (Cyrene, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

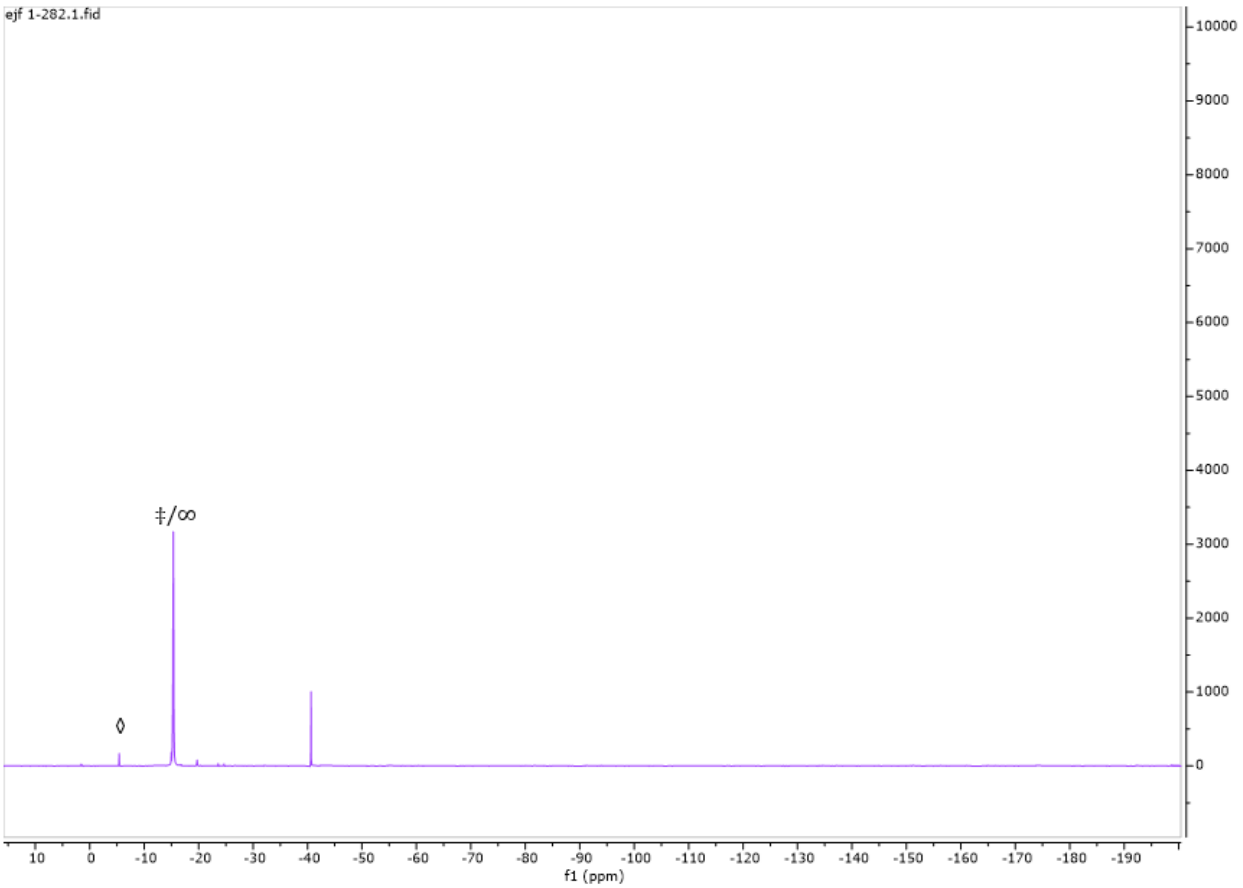


Figure S28. $^{31}P\{^{1}H\}$ NMR (CPME, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

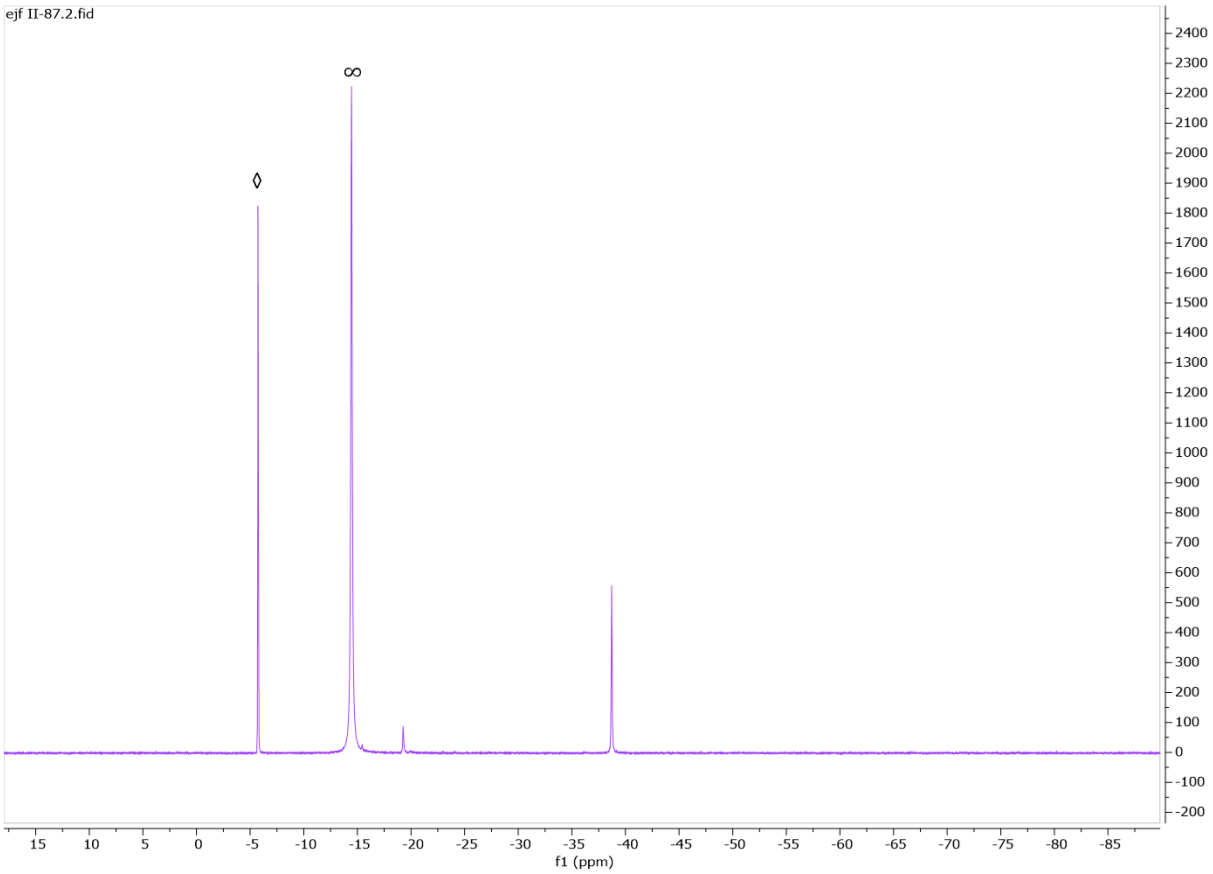


Figure S29. $^{31}P\{^{1}H\}$ NMR (MEK, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

NMR Spectra of hydrophosphination with 1 mol % $Cu(acac)_2$

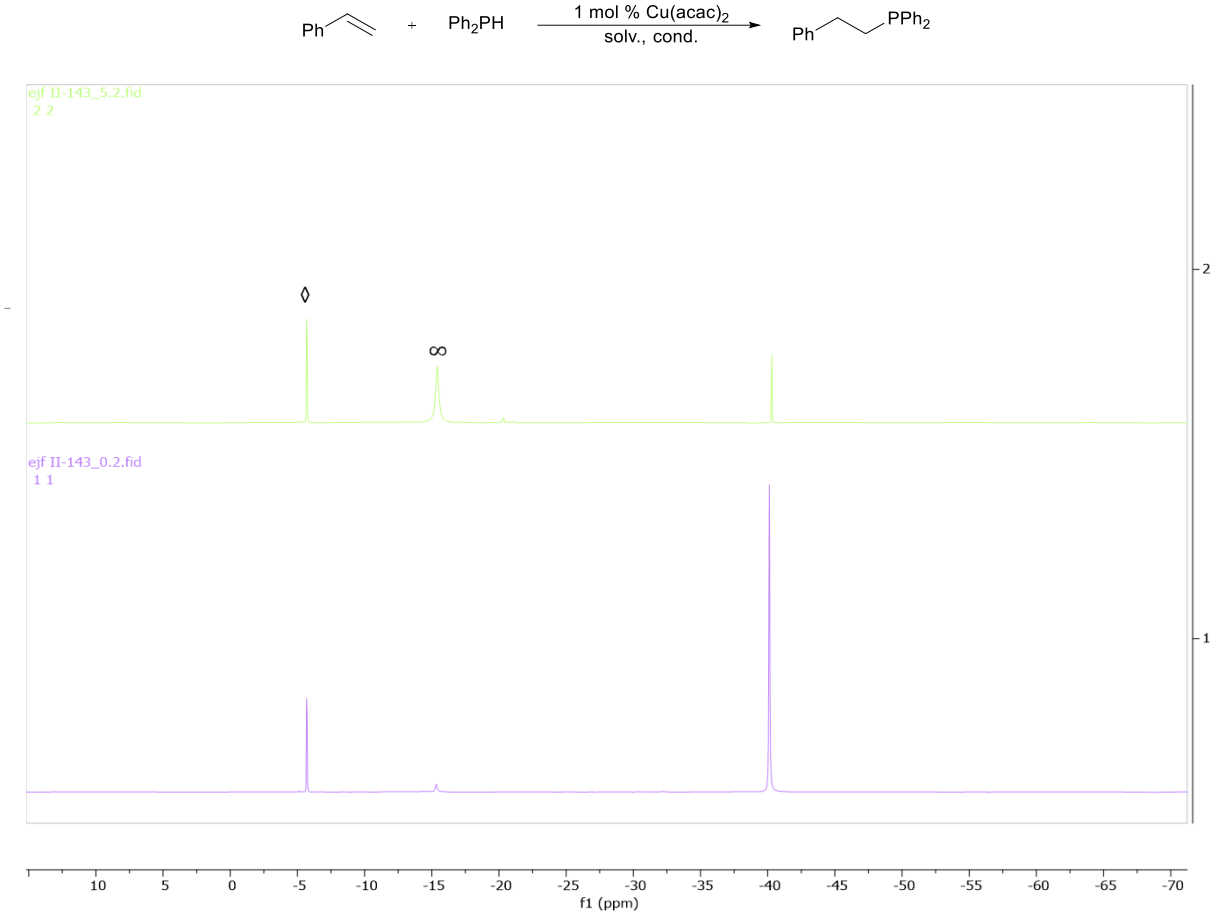
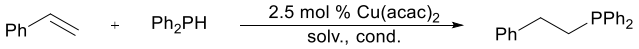


Figure 30. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

NMR Spectra of hydrophosphination with 2.5 mol % $Cu(acac)_2$



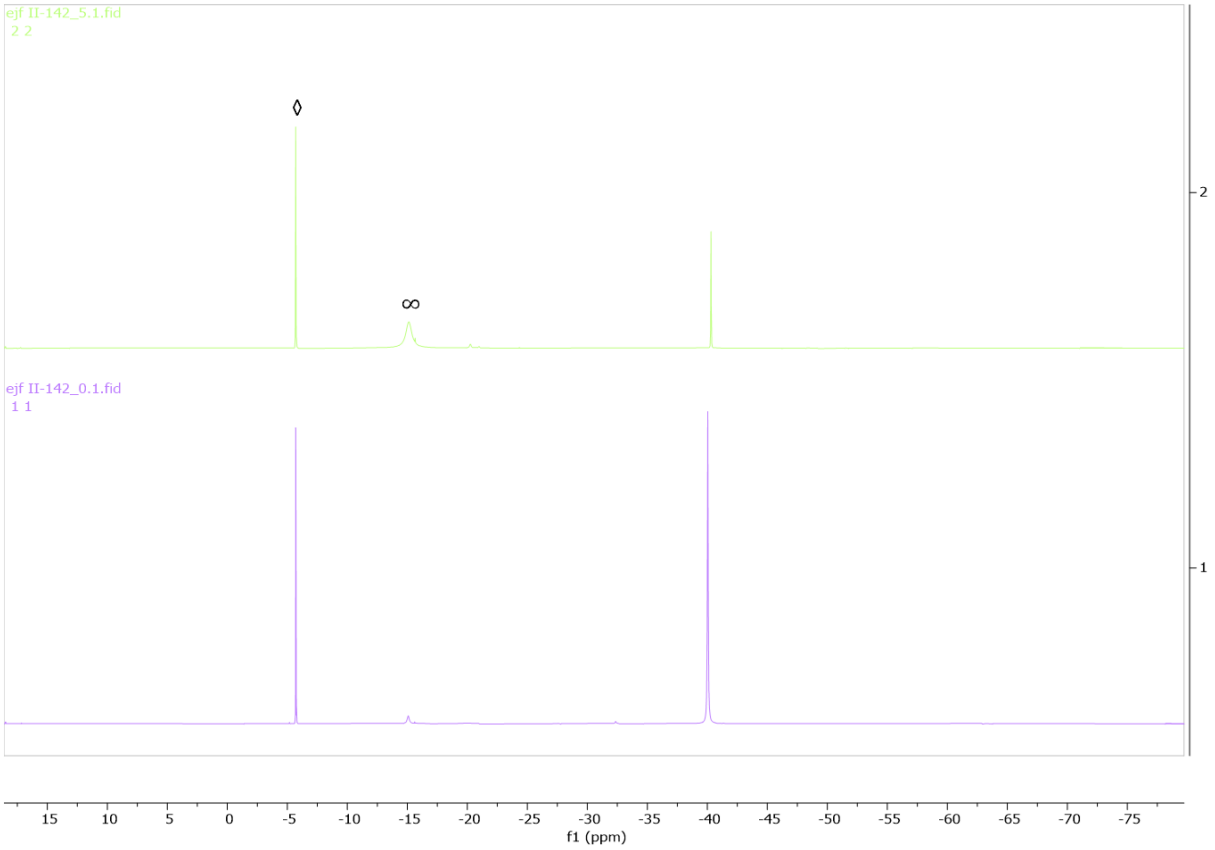


Figure 31. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative copper-catalyzed hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

NMR Spectra of hydrophosphination with 5 mol % LiOEt

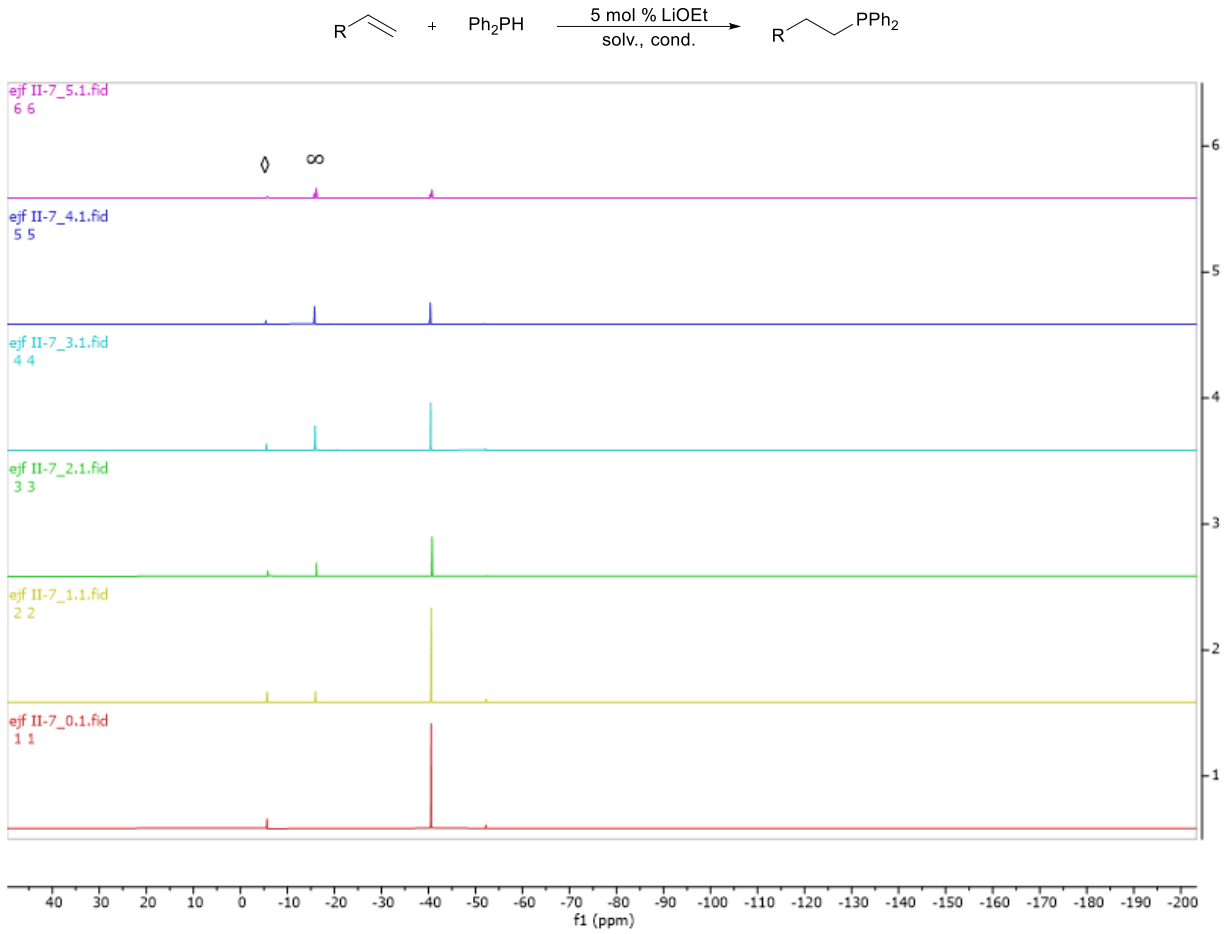


Figure S32. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative LiOEt-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

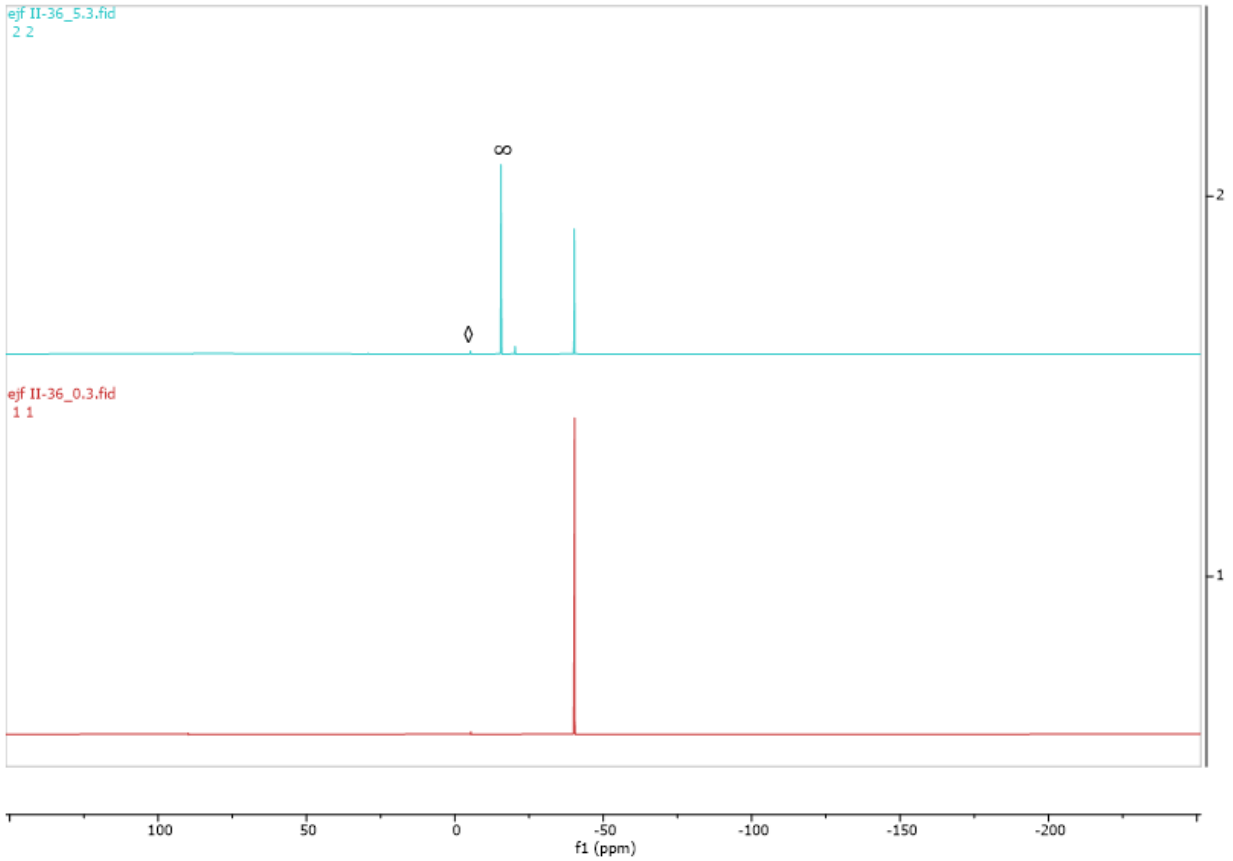


Figure S33. Stacked 31P{1H} NMR (EtOAc, 500 MHz) spectra of a representative LiOEt-catalyzed hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

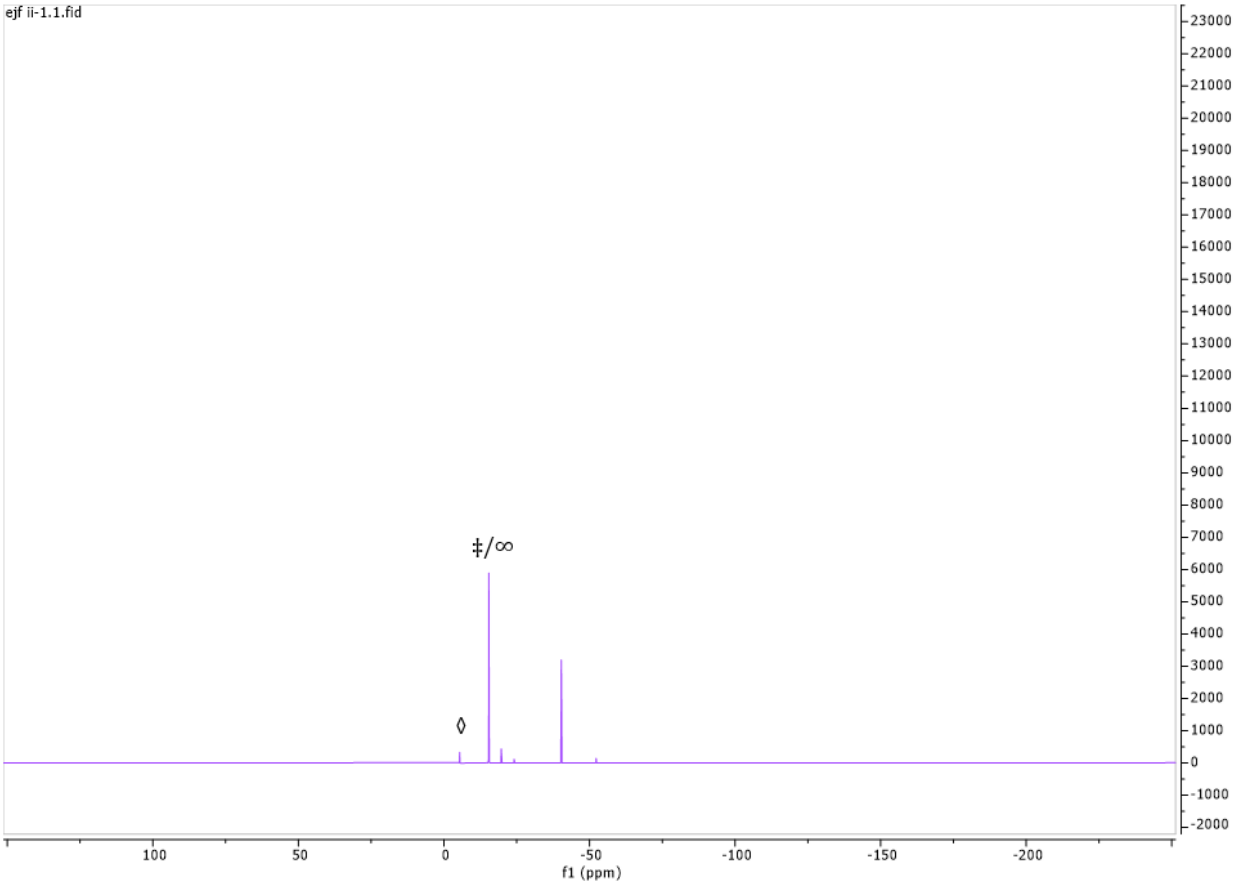


Figure S34. $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative LiOEt-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard

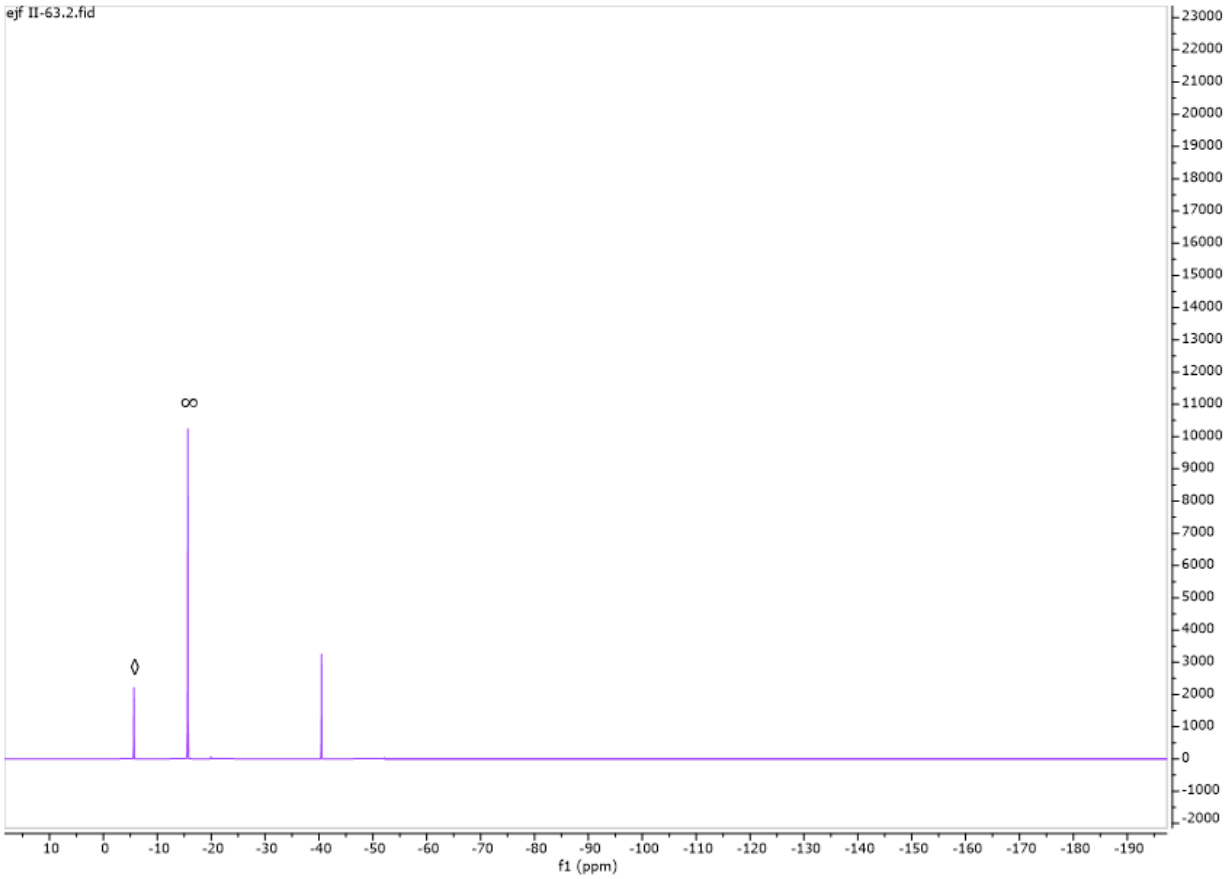


Figure S35. $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative LiOEt-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard

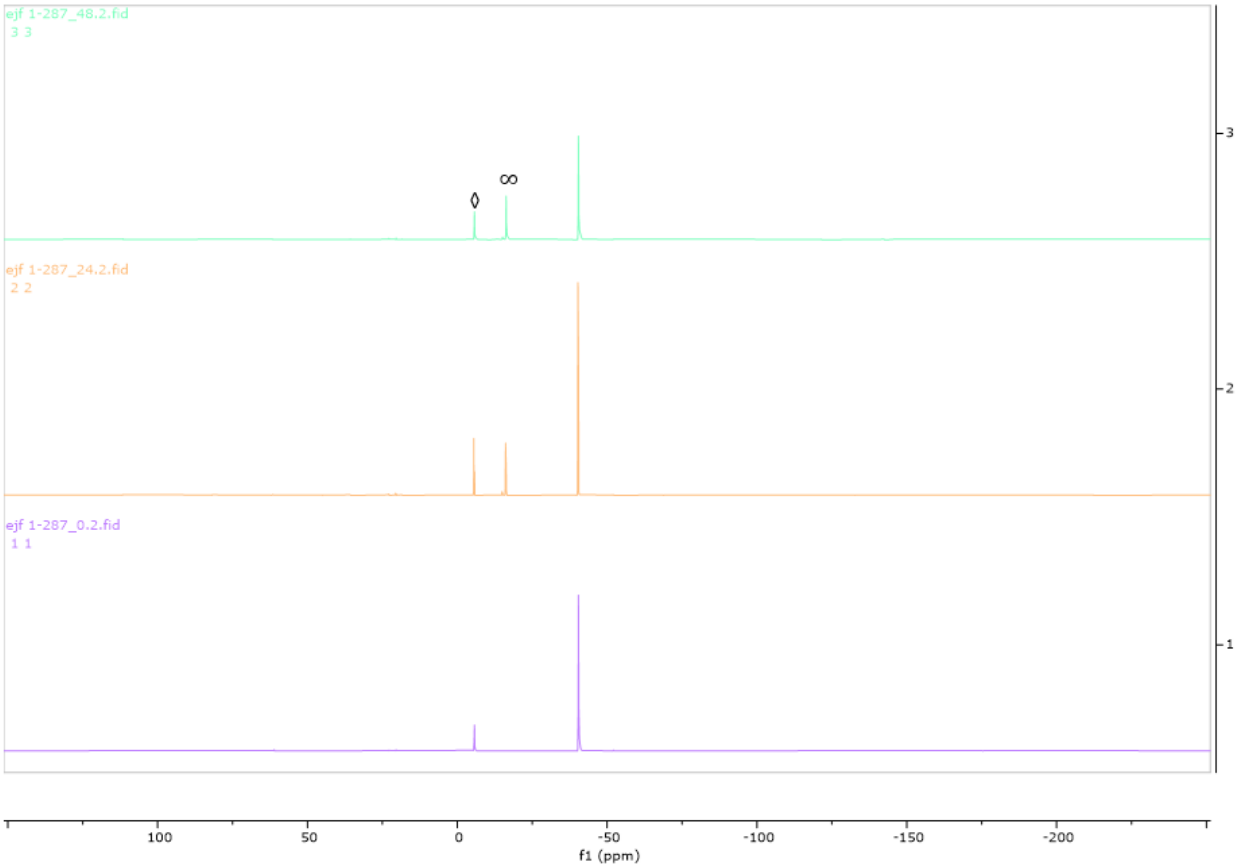


Figure S36. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative LiOEt-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

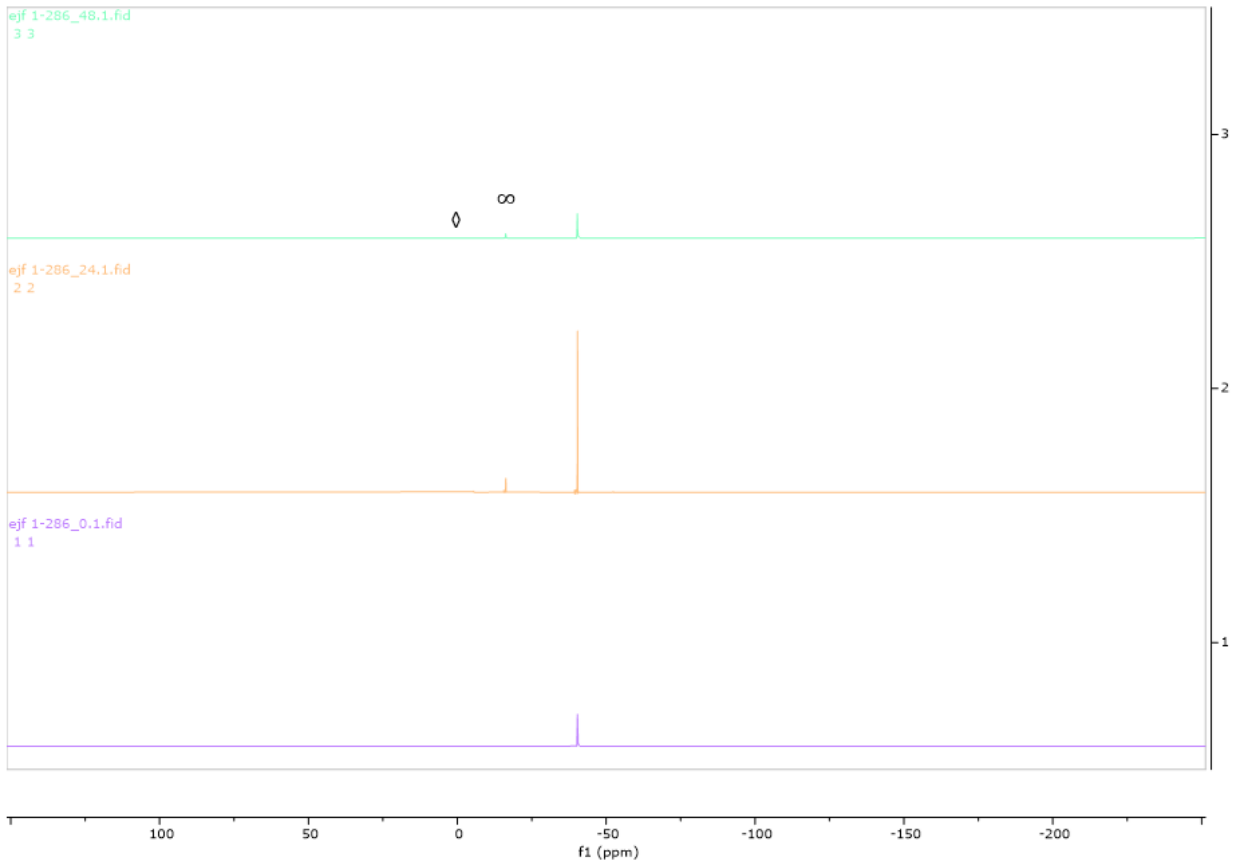


Figure S37. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative LiOEt-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24, 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

NMR Spectra of hydrophosphination with 5 mol % NaOEt

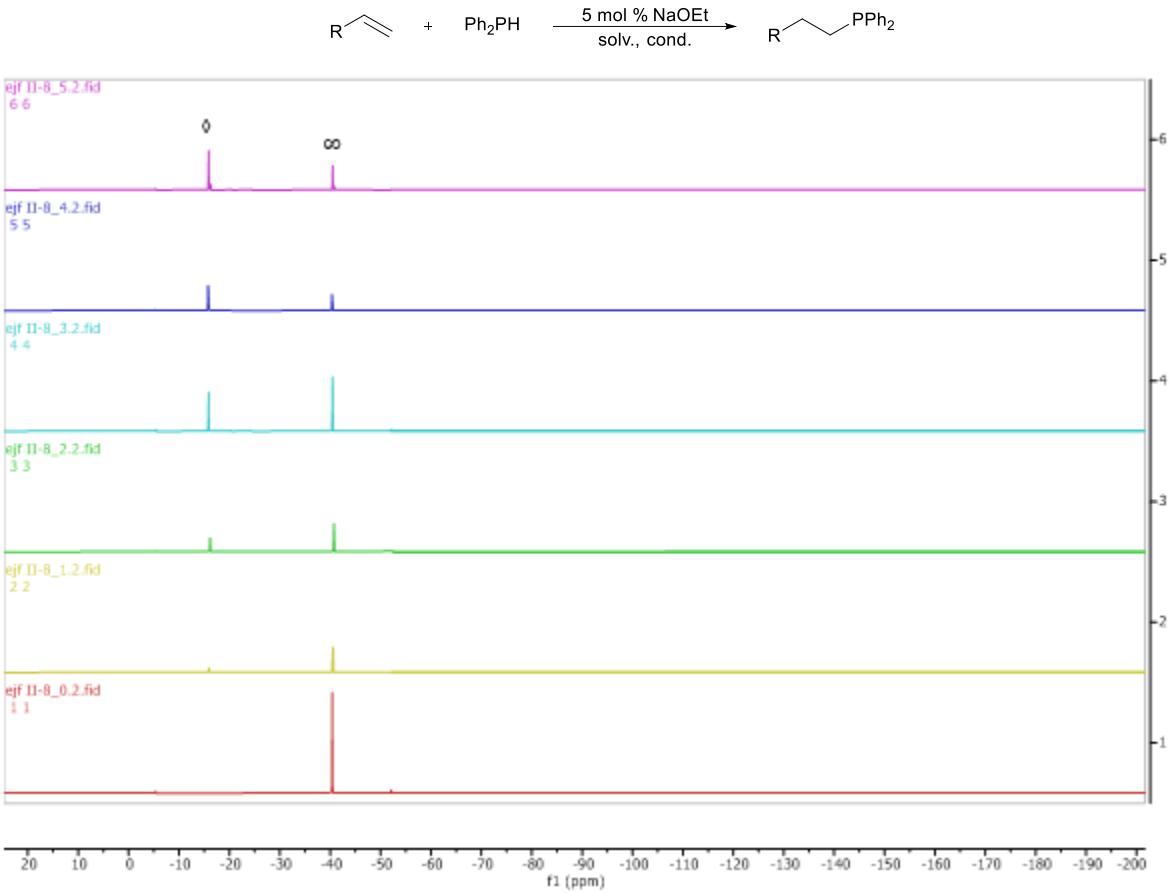


Figure S38. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative NaOEt-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

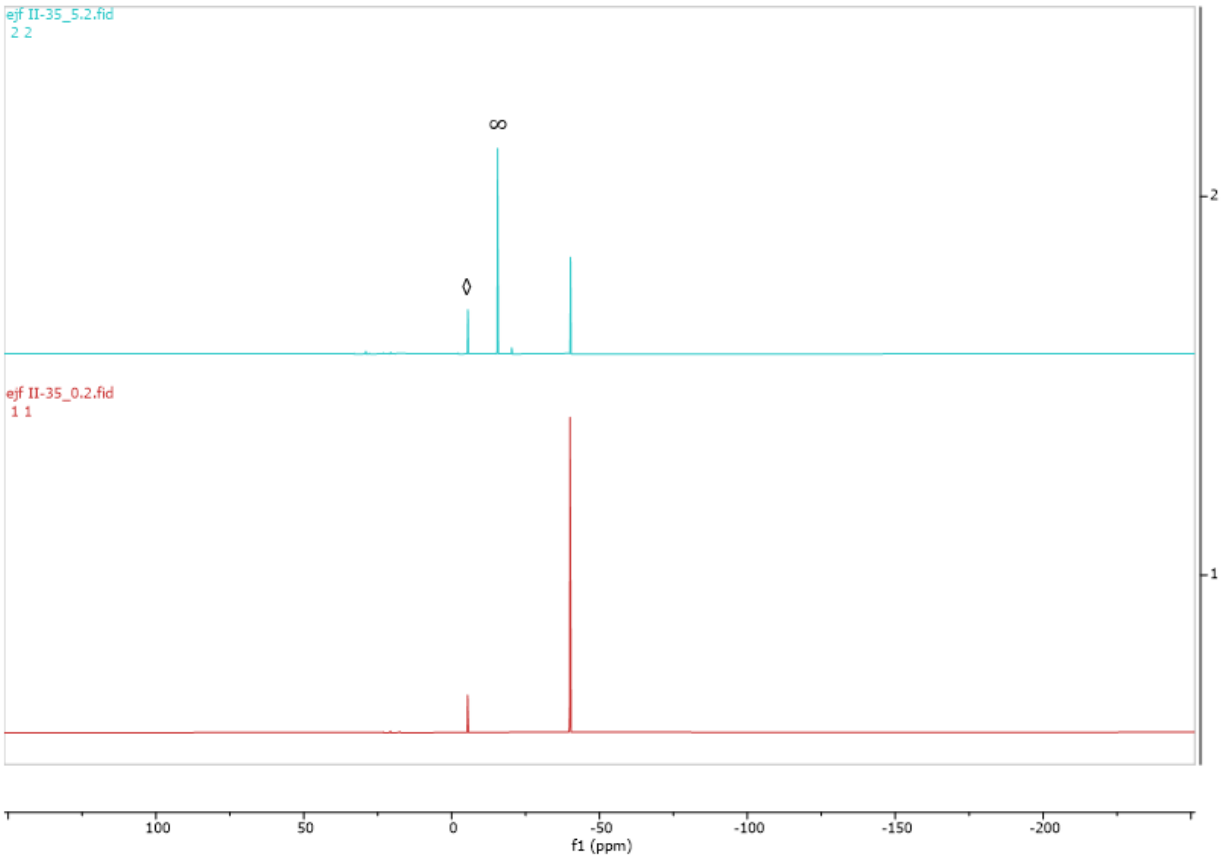


Figure S39. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative NaOEt-catalyzed hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

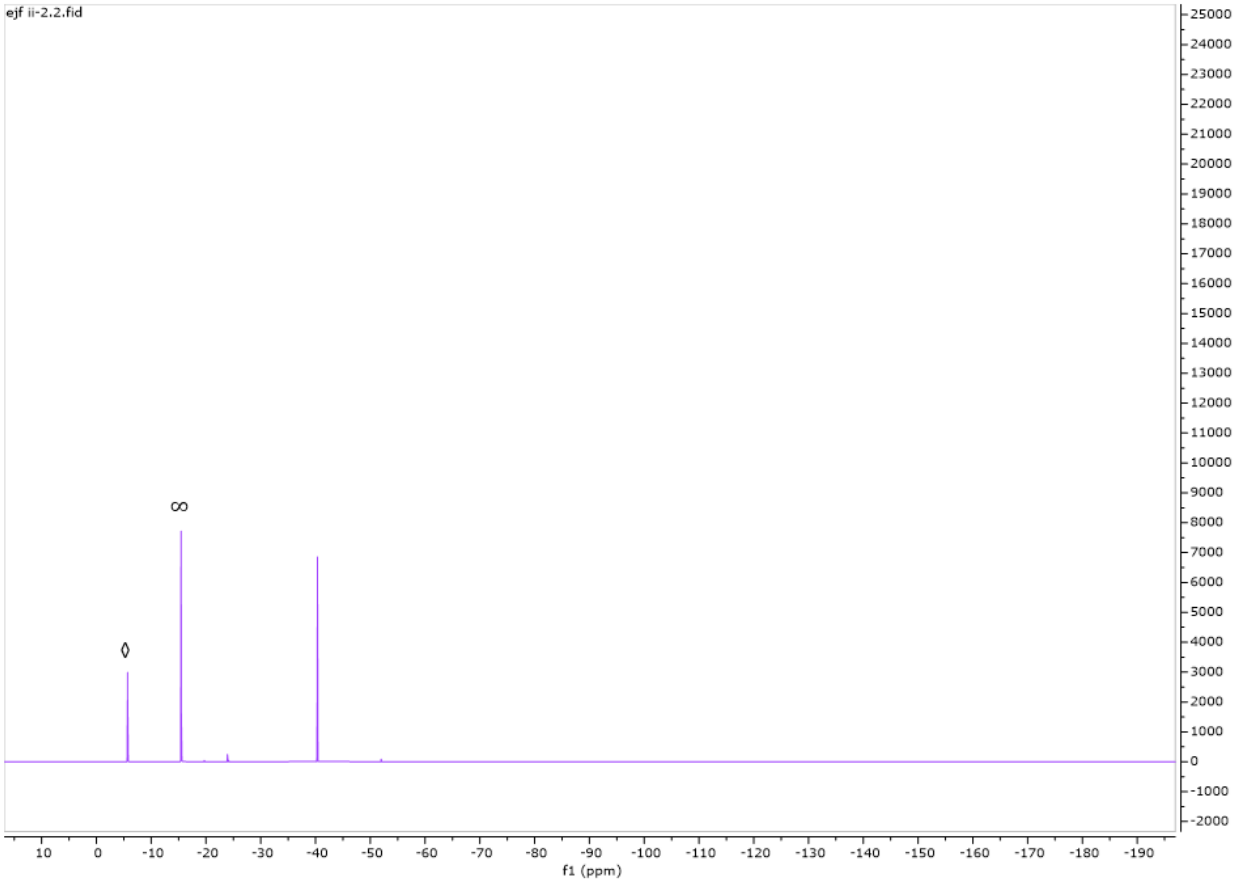


Figure S40. $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative NaOEt-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

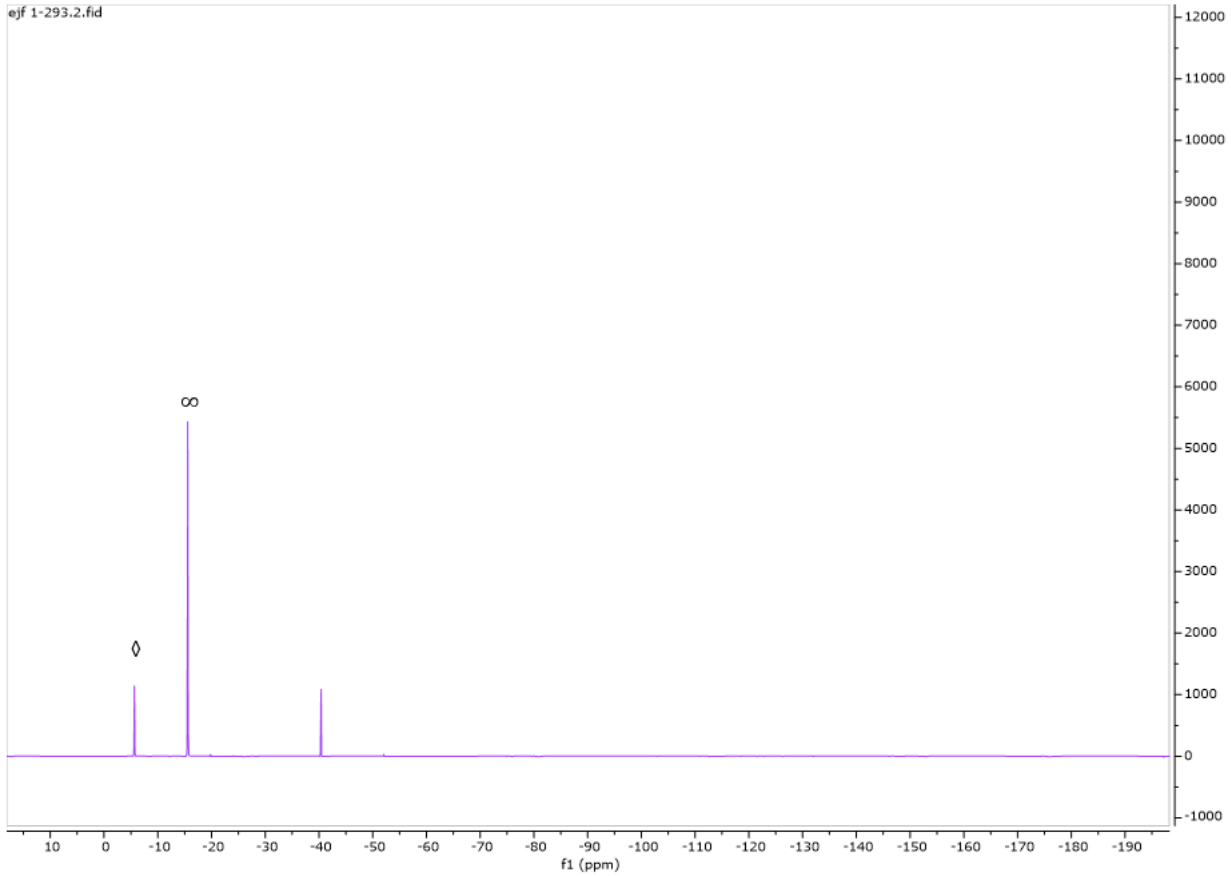


Figure S41. $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative NaOEt-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

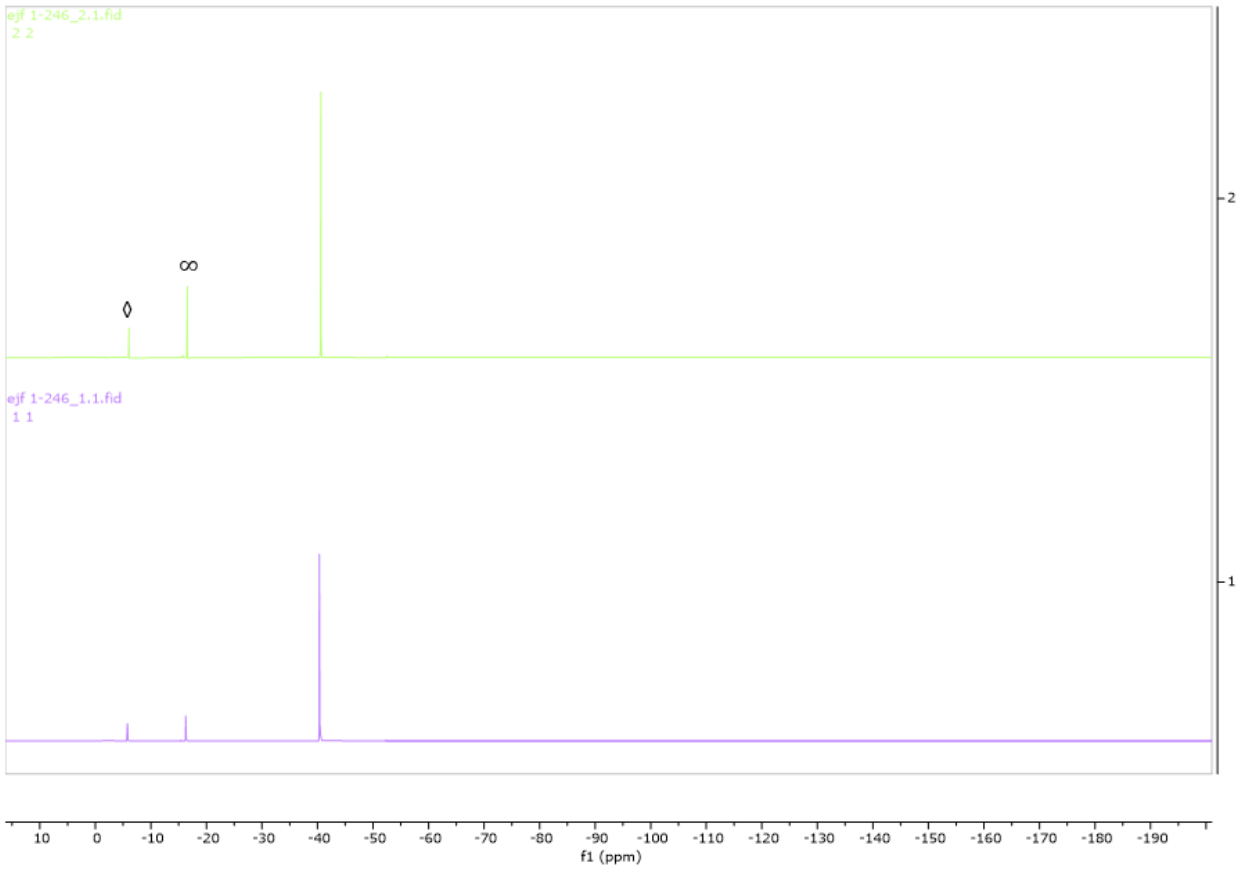


Figure S42. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative NaOEt-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

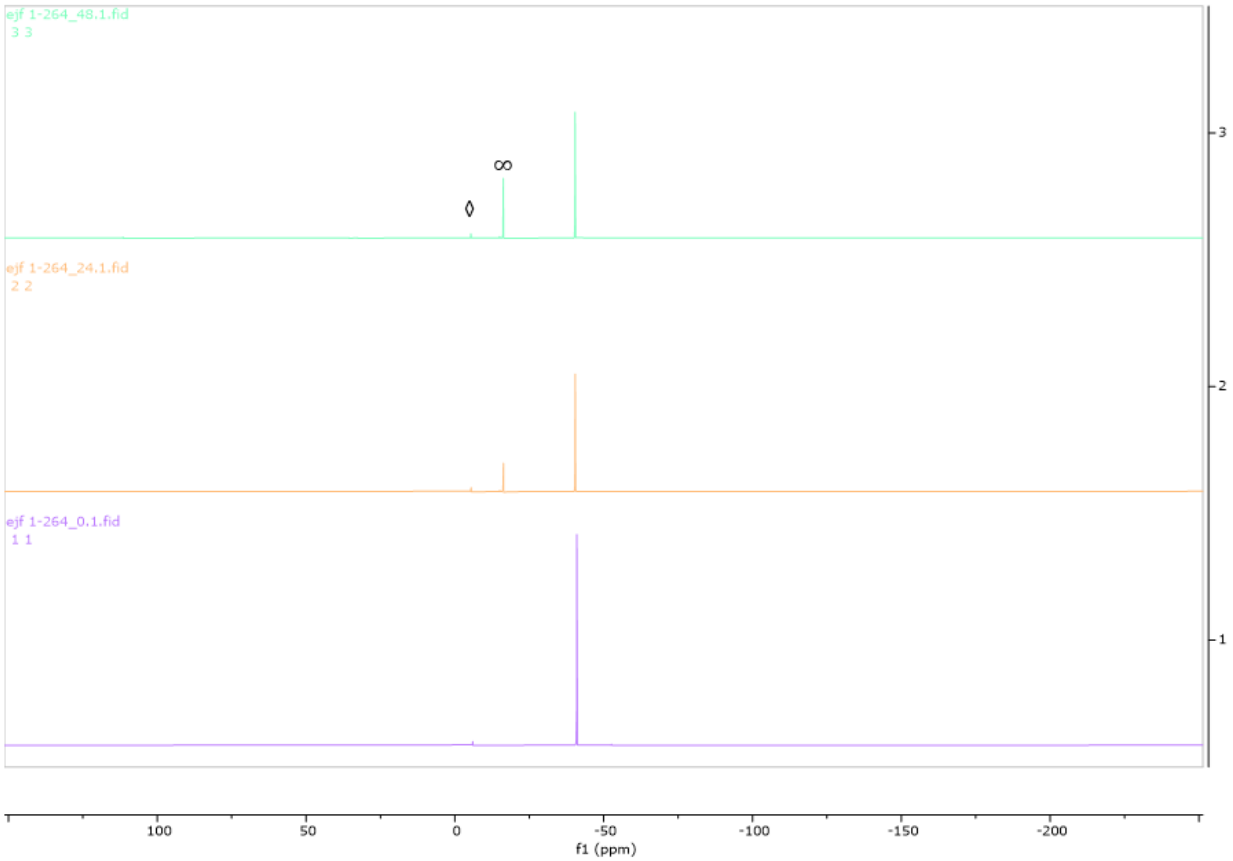


Figure S43. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative NaOEt-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

NMR Spectra of hydrophosphination with 5 mol % KOEt

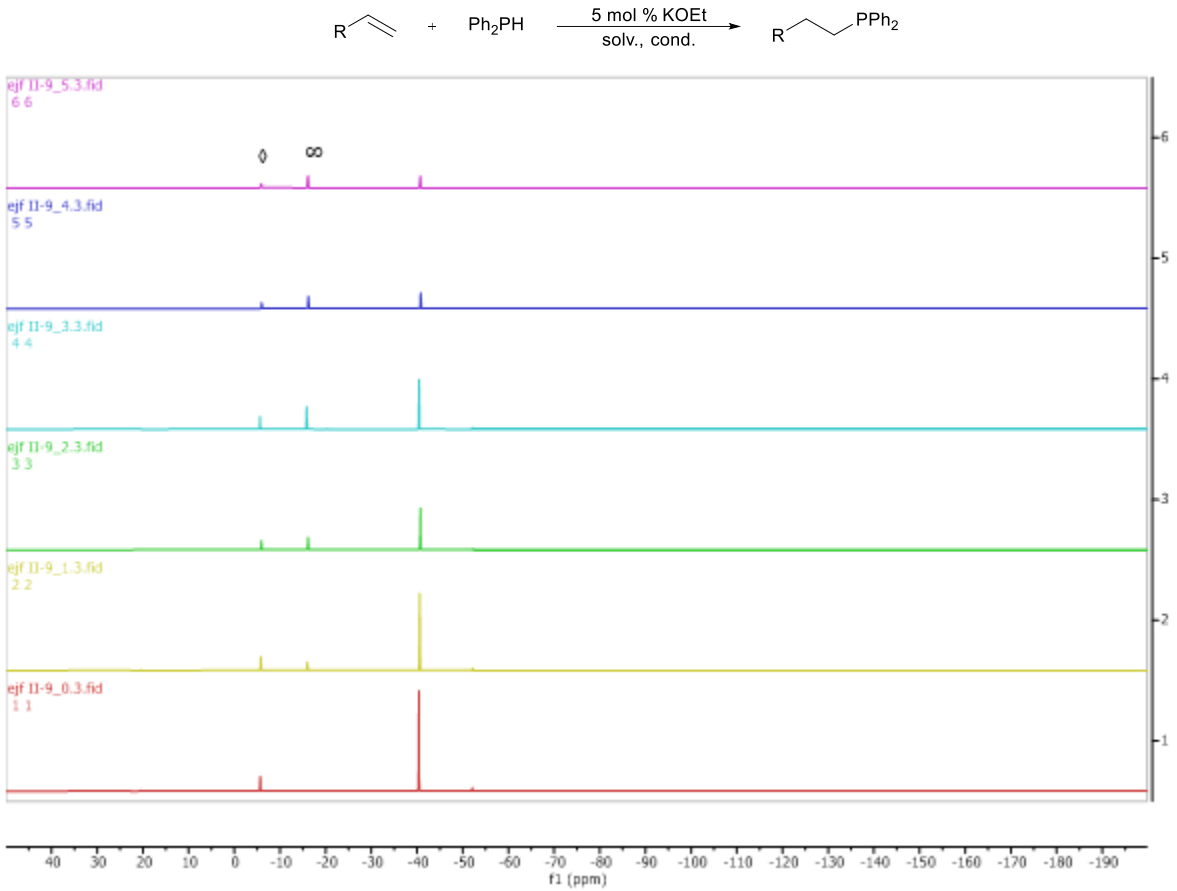


Figure S44. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative KOEt-catalyzed hydrophosphination of styrene and diphenylphosphine at 0, 1, 2, 3, 4, and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

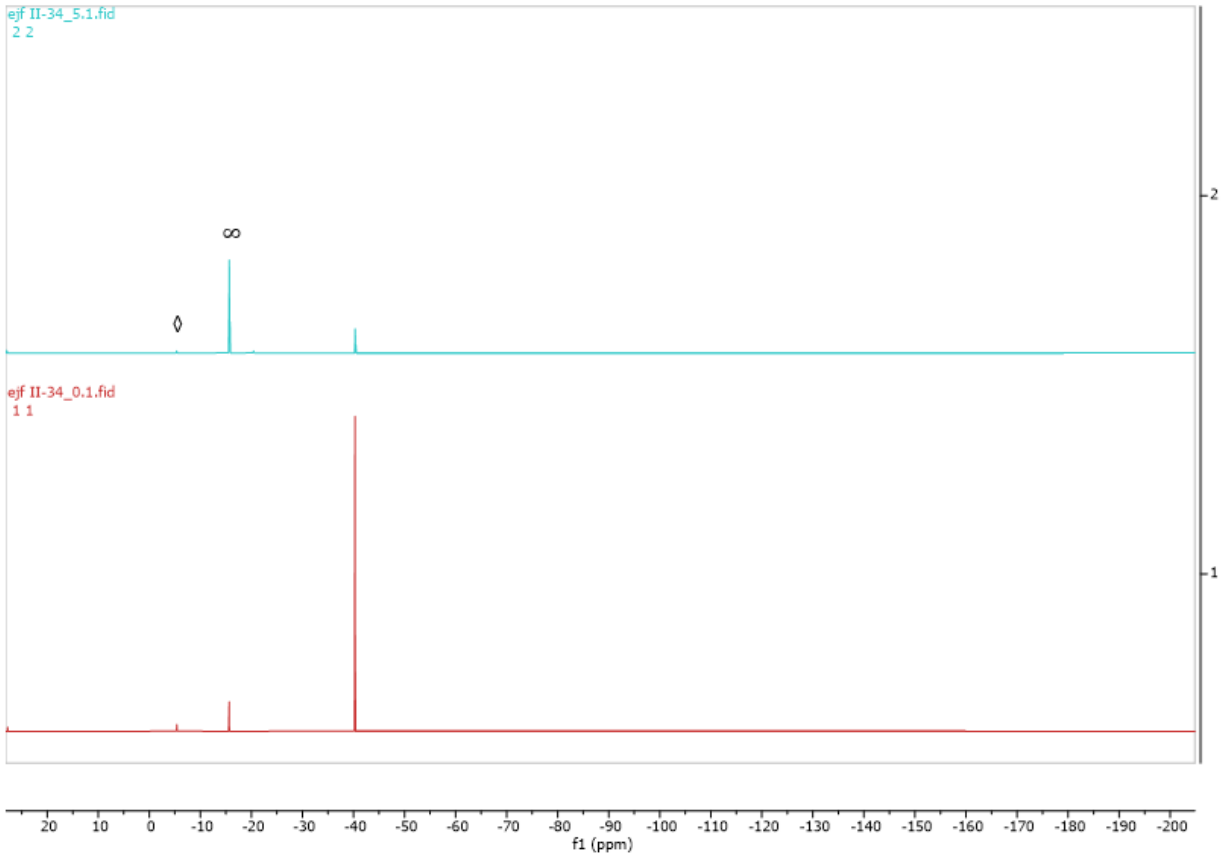


Figure S45. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative KOEt-catalyzed hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

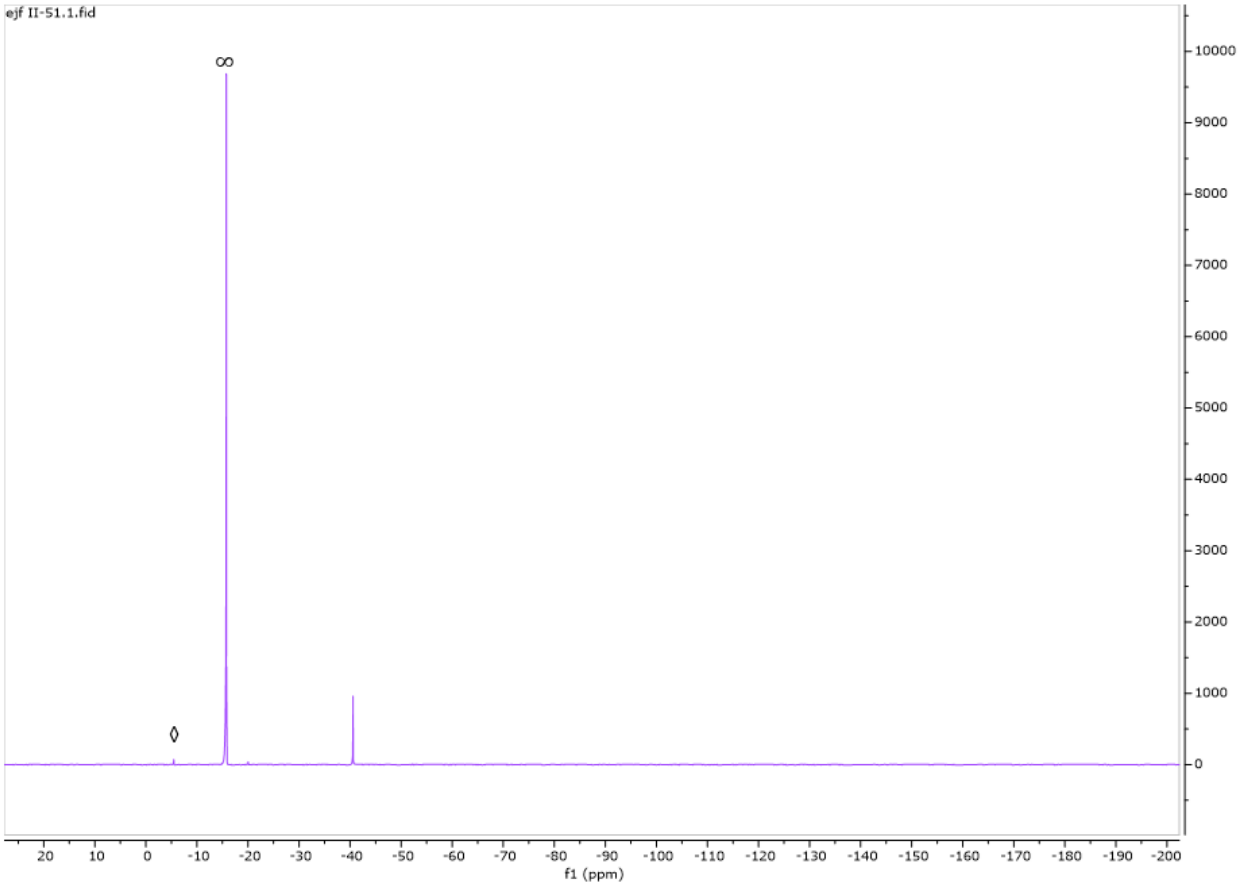


Figure S46. $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative KOEt-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

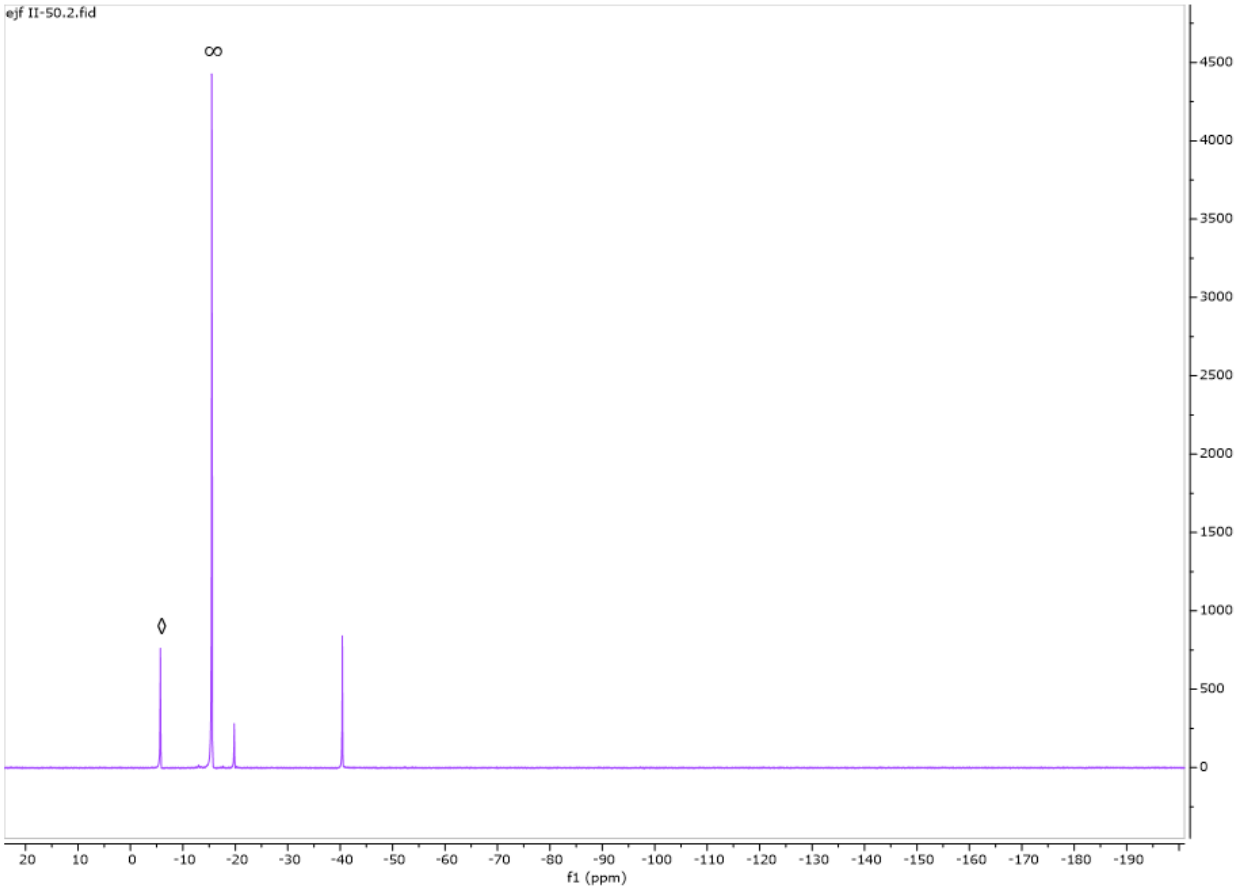


Figure S47. $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative KOEt-catalyzed hydrophosphination of ethyl acrylate and diphenylphosphine after 10 min. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

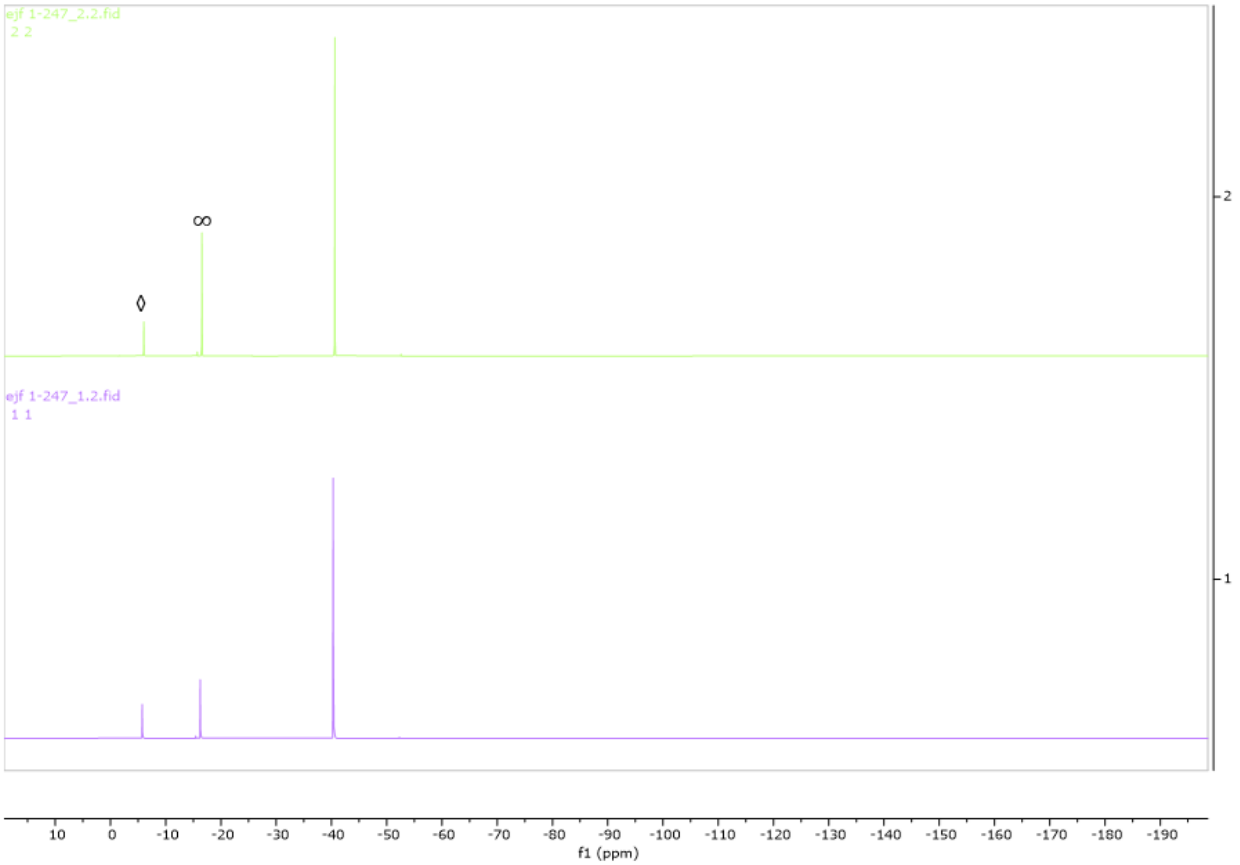


Figure S48. Stacked $^{31}P\{^{1}H\}$ NMR (EtOAc, 500 MHz) spectra of a representative KOEt-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

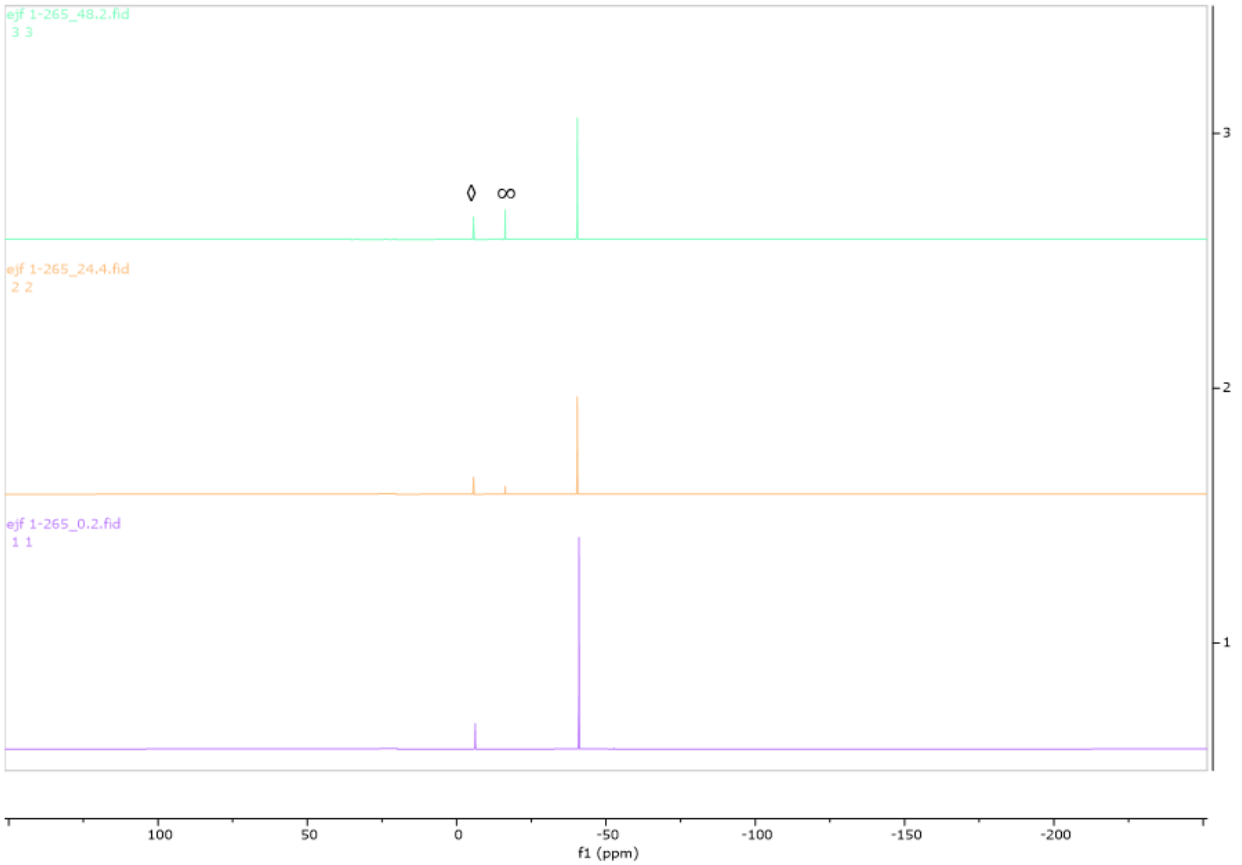


Figure S49. Stacked $^{31}P\{^{1}H\}$ NMR (EtOH, 500 MHz) spectra of a representative KOEt-catalyzed hydrophosphination of 1-hexene and diphenylphosphine at 0, 24 and 48 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

Catalyst-free control data

entry	solvent	Conversion (%) initial	5 h
1	EtOH	0	60
2	EtOAc	0	33
3	DMSO	0	63
4	heptane	0	44
5	2-MeTHF	0	26
6	Cyrene	0	23
7	CPME	0	32

Table S2. Conversions of control reactions with irradiation at 360 nm. Reaction conditions: Diphenylphosphine (0.38 mmol), styrene (0.38 mmol), solvent (400 μL).

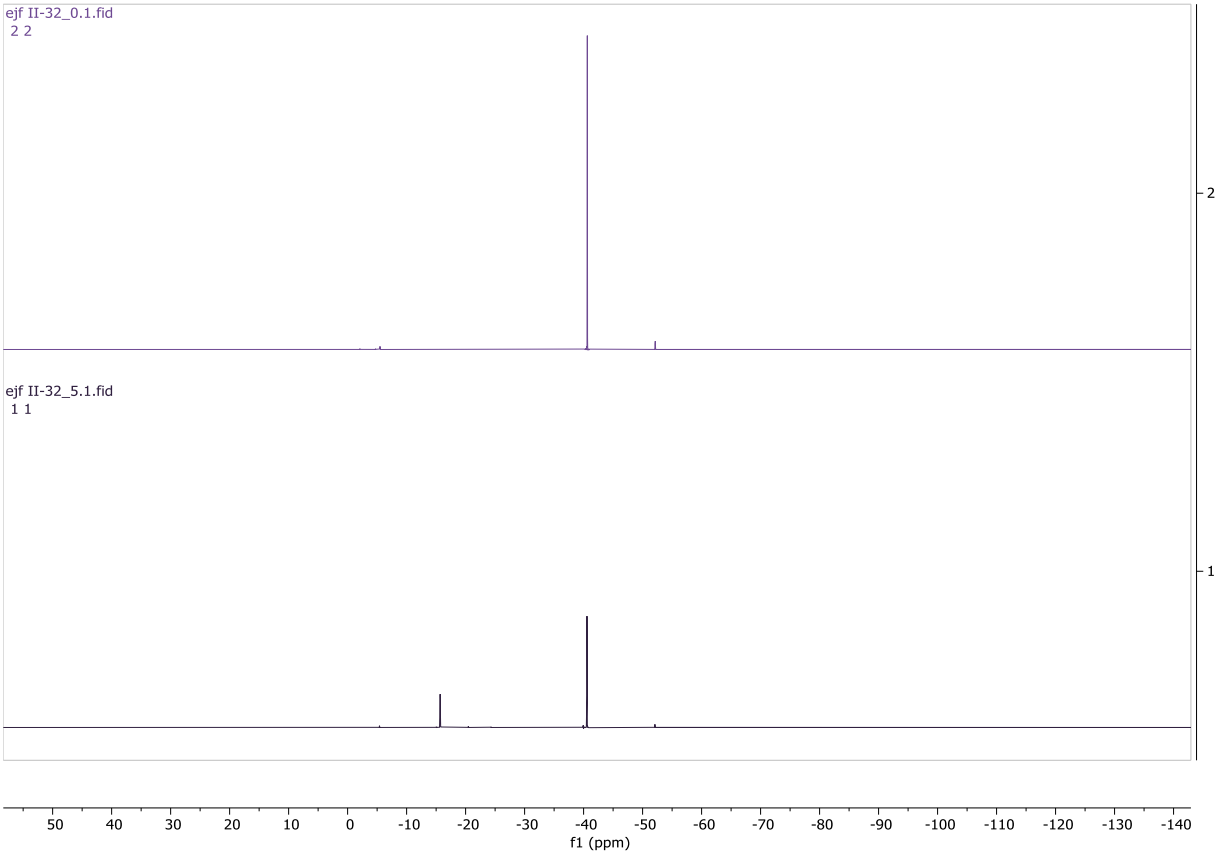


Figure S50. Stacked $^{31}P\{^{1}H\}$ NMR (2-MeTHF, 500 MHz) spectra of a representative control hydrophosphination of styrene and diphenylphosphine at 0 and 5 h intervals with irradiation at 360 nm. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

NMR Spectra of hydrophosphination reactions with 2-MeTHF

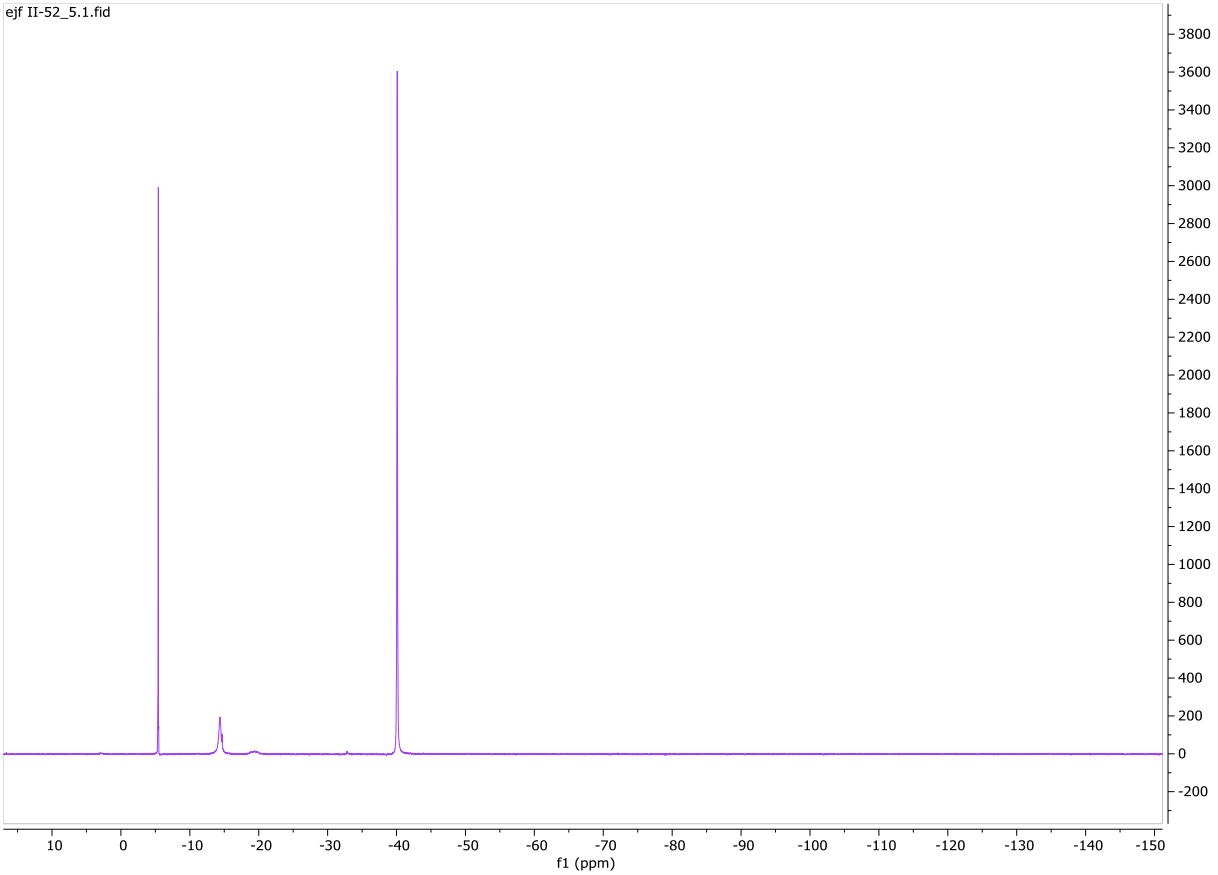


Figure S51. $^{31}P\{^{1}H\}$ NMR (2-MeTHF, 500 MHz) spectra of a representative hydrophosphination of styrene (0.38 mmol) and diphenylphosphine after 2 h at 90 °C under N_2. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

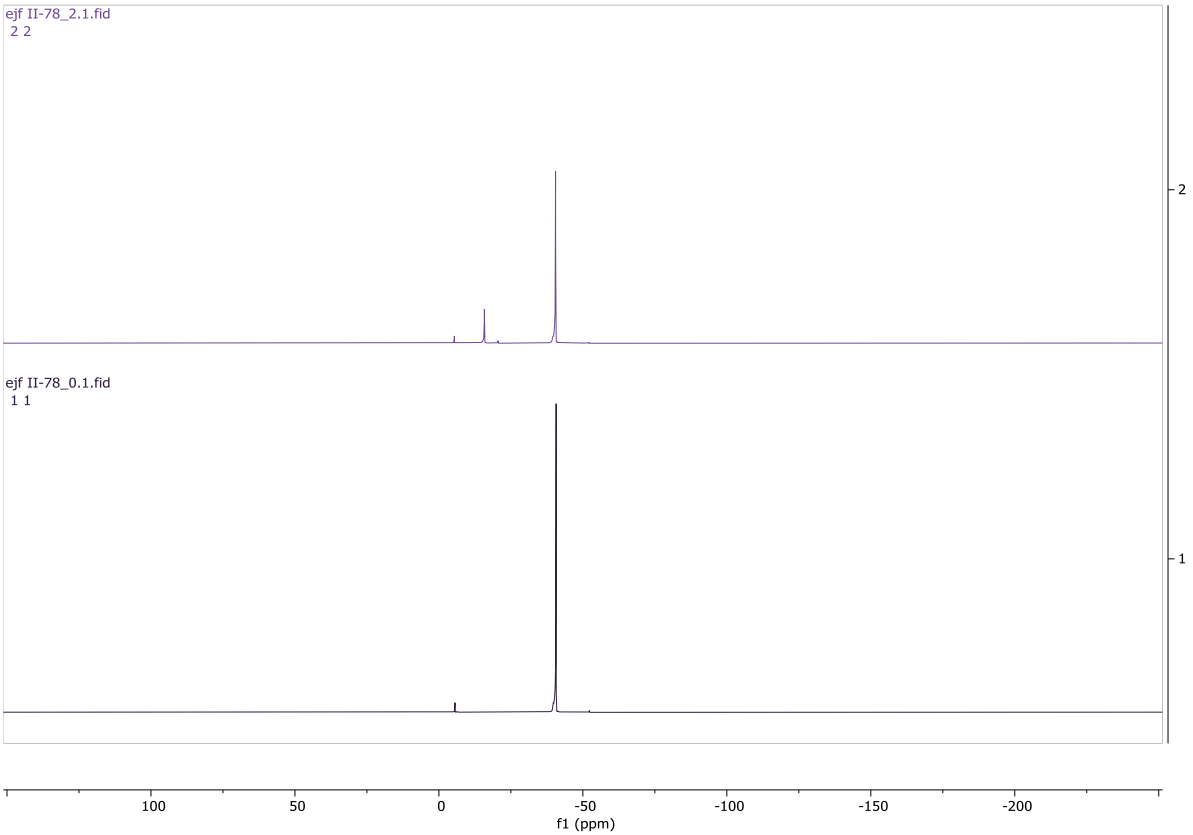


Figure S52. $^{31}P\{^{1}H\}$ NMR (2-MeTHF, 500 MHz) spectra of a representative hydrophosphination of styrene and diphenylphosphine (0.5 g) after 2 h at 90 °C under Ar. Legend: ‡ = $[Ph_2P]_2$, ∞ = hydrophosphination product, and ◊ = external standard.

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