Microwave Assisted Regioselective Addition of P(O)-H Bonds to Alkenes Without Solvent or Catalyst.

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SUPPLEMENTARY INFORMATION

General considerations	S2
Experimental descriptions	S2-S16
Selected NMR spectra	S17-S34

General considerations: Addition reactions were carried out under an atmosphere of nitrogen. Diethyl ether, dichloromethane, and hexane were dried using a Grubbs-type solvent purification system. Ethyl acetate and THF were dried by distillation from CaH_2 and Na/benzophenone respectively. Nitrogen was purified by passage activated analytical columns from Chromatography Research Supplies. The remaining chemicals were obtained from Aldrich and used as received. For control reactions, the radical inhibitors were removed by distillation. All yields are based upon isolated material. The purity of the products was determined by ^1H NMR spectroscopy and GC unless specified otherwise. Spectroscopic data of known compounds were compared with literature values or authentic samples. Elemental analyses were performed by Midwest Microlabs. ^1H and ^{13}C chemical shifts were determined by reference to residual ^1H and ^{13}C solvent resonances. All coupling constants are given in Hertz. ^{31}P NMR spectra were referenced to external H_3PO_4 (0 ppm). Quantitative ^{31}P NMR spectra were collected using an inverse-gated decoupling sequence with a recycle delay of 30 s. Microwave catalyzed reactions were carried out in 10 mL reactor vials using a CEM Discover microwave reactor using an IR temperature sensor. The hold time for each run was 0 s, and the listed time is the total time of irradiation. The specific microwave power levels used in the preparations are included in each experimental description.

General procedure for the microwave assisted hydrophosphinylation reactions. A new microwave reactor vial (10 mL) was charged in air with the phosphine oxide or DOPO and a new magnetic stirring bar. The vial was evacuated and refilled with nitrogen before the addition of the alkene. When the alkene was a solid, it was added

prior to evacuation. After thoroughly mixing the reagents (stirring), the vial was placed in the microwave reactor and irradiated for 60 minutes at the desired temperature. The maximum power setting of 50 watts was maintained until the desired temperature was reached. The power was then reduced for the remainder of the reaction to maintain the temperature. The reaction was cooled to room temperature, analyzed, and purified when needed by trituration with an appropriate solvent or by column chromatography.

Synthesis of 2-(diphenylphosphinyl)ethyl-phosphonic acid, diethyl ester (Table 1

entry 1).¹ The general method was followed with HP(O)Ph₂ (0.050 g, 0.25 mmol) and vinyl diethylphosphonate (38.0 μ L, 0.25 mmol). The maximum power setting (50 watts) was maintained until a temperature of 120 °C was achieved (78 s). The power was then reduced to 10 watts for the remainder of the reaction to maintain 120 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.88 g (97%) of the title compound (96 % pure). Trituration of the residue with ether/hexane (1:1; 2 mL) afforded 0.82 g (90%, 98% pure) of the title compound. ¹H NMR (CDCl₃, 19 °C): δ 7.73 (m, 4H, Ar-H), 7.50 (m, 6H, Ar-H), 4.02 (m, 4H, -OCH₂-), 2.47 (m, 2H, -CH₂-), 1.91 (m, 2H, -CH₂-), 1.25 (t, 6H, J = 7.05, -CH₃). ¹³C{¹H} NMR (CDCl₃, 19 °C): δ 132.0 (d, J = 2.6, Ar-C), 131.8 (dd, J = 97.0, 3.4, quat), 130.8 (d, J = 9.1, Ar-C), 128.8 (d, J = 11.5, Ar-C), 61.9 (d, J = 6.3, -OCH₂-), 23.0 (dd, J = 69.0, 3.6, -CH₂-), 17.7 (d, J = 139.9, -CH₂-), 16.4 (d, J = 6.0, -CH₃). ³¹P{¹H} NMR (CDCl₃, 19 °C): δ 31.3 (d, J = 66.3, -P(O)-), 29.4 (d, J = 66.3, -P(O)-).

Synthesis of 3-(diphenylphosphinyl)-propanoic acid, butyl ester (Table 1, entry 2).²

The general method was followed with HP(O)Ph₂ (0.050 g, 0.25 mmol) and butyl acrylate (35.5 μ L, 0.25 mmol). The maximum power setting (50 watts) was maintained until a temperature of 120 °C was achieved (120 s). The power was then reduced to 13 watts for the remainder of the reaction to maintain 120 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.078 g (96%) of the title compound (94% pure). Purification of the crude residue by column chromatography (silica gel, ether/THF 5:1) afforded 0.068 g (83%, 98% pure) of the title compound. ¹H NMR (CDCl₃, 19 °C): δ 7.79-7.72 (m, 4H, Ar-H), 7.54-7.45 (m, 6H, Ar-H), 4.03 (t, 2H, J = 6.7, -OCH₂-), 2.62 (m, 4H, -CH₂-), 1.56 (m, 2H, -CH₂-), 1.32 (m, 2H, -CH₂-), 0.90 (t, 3H, J = 7.3, -CH₃). ¹³C{¹H} (CDCl₃, 19 °C): δ 172.4 (d, J = 17.1, C=O), 132.1 (d, J = 99.9, quat), 131.9 (d, J = 2.6, Ar-C), 130.7 (d, J = 9.4, Ar-C), 128.7 (d, J = 11.7, Ar-C), 64.8 (s, -OCH₂-), 30.4 (s, -CH₂-), 26.4 (d, J = 2.0, -CH₂-), 24.9 (d, J = 72.9, -CH₂-), 19.0 (s, -CH₂-), 13.6 (s, -CH₃). ³¹P{¹H} (CDCl₃, 19 °C): δ 30.7 (s).

Synthesis of 3-(diphenylphosphinyl)-2-methyl-propanoic acid, methyl ester (Table 1,

entry 3).³ The general method was followed with HP(O)Ph₂ (0.050 g, 0.25 mmol) and methyl methacrylate (26.5 μ L, 0.25 mmol). The maximum power setting (50 watts) was maintained until a temperature of 120 °C was achieved (145 s). The power was then reduced to 10 watts for the remainder of the reaction to maintain 120 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.073 g (98%) of the title compound (87% pure). Purification of the crude residue by column chromatography (silica gel, THF/hexane 1:1) afforded 0.064 g (78%, 98% pure) of the title compound. ¹H

NMR (CDCl₃, 19 °C): δ 7.79 – 7.72 (m, 4H, Ar-H), 7.55 – 7.43 (m, 6H, Ar-H), 3.50 (s, 3H, -OCH₃), 2.93 (m, 2H, -CH₂-), 2.33 (m, 1H, -CH-), 1.29 (d, 3H, J = 7.0, -CH₃).

¹³C{¹H} NMR (CDCl₃, 19 °C): δ 175.7 (d, J = 10.0, C=O), 133.3 (d, J = 95.3, quat), 132.0 (d, J = 94.8, quat), 131.7 (d, J = 3.0, Ar-H), 130.8 (d, J = 9.3, Ar-C), 130.4 (d, J = 9.3, Ar-C), 128.5 (d, J = 11.9, Ar-C), 128.4 (d, J = 11.8, Ar-C), 51.8 (s, -OCH₃), 33.5 (d, J = 2.6, -CH-), 32.9 (d, J = 71.7, -CH₂-), 19.1 (d, J = 7.2, -CH₃). ³¹P{¹H} NMR (CDCl₃, 19 °C): δ 29.3 (s).

Synthesis of 3-(diphenylphosphinyl)-propanenitrile (Table 1, entry 4).³ The general method was followed with HP(O)Ph₂ (0.050 g, 0.25 mmol) and acrylonitrile (16.3 μ L, 0.25 mmol). The maximum power setting (50 watts) was maintained until a temperature of 120 °C was achieved (79 s). The power was then reduced to 13 watts for the remainder of the reaction to maintain 120 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.62 g (98%) of the title compound (95% pure). Trituration of the residue with ether (1 mL) afforded 0.057 g (90%, 98% pure) of the title compound. ¹H NMR (CDCl₃, 19 °C): δ 7.74 (m, 4H, Ar-H), 7.55 (m, 6H, Ar-H), 2.64 (m, 4H, -CH₂-). ¹³C{¹H} NMR (CDCl₃, 19 °C): δ 132.6 (d, J = 2.8, Ar-C), 130.9 (d, J = 101.2, Ar-C), 130.7 (d, J = 9.7, Ar-C), 129.0 (d, J = 11.8, Ar-C), 118.6 (d, J = 18.3, -CN), 26.2 (d, J = 69.9, -CH₂-), 10.4 (d, J = 1.0, -CH₂-). ³¹P{¹H} NMR (CDCl₃, 19 °C): δ 28.7 (s).

Synthesis of 4-(diphenylphosphinyl)-2-butanone (Table 1, entry 5).⁴ The general method was followed with HP(O)Ph₂ (0.050 g, 0.25 mmol) and methyl vinyl ketone (20.6 μ L, 0.25 mmol). The maximum power setting (50 watts) was maintained until a

temperature of 120 °C was achieved (130 s). The power was then reduced to 20 watts for the remainder of the reaction to maintain 120 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.65 g (96 %) of the title compound (97 % pure).

Trituration of the residue with ether/hexane (1:1; 2 mL) afforded 0.058 g (87 %, 99 % pure) of the title compound. ^1H NMR (CDCl_3 , 19 °C): δ 7.67 – 7.61 (m, 4H, Ar-H), 7.45 – 7.35 (m, 6H, Ar-H), 2.67 (m, 2H, $-\text{CH}_2-$), 2.44 (m, 2H, $-\text{CH}_2-$) 2.02 (s, 3H, $-\text{CH}_3$).

$^{13}\text{C}\{^1\text{H}\}$ (CDCl_3 , 19 °C): δ 206.2 (d, J = 13.6, C=O), 132.3 (d, J = 99.5, quat), 131.9 (d, J = 2.6, Ar-C), 130.7 (d, J = 9.6, Ar-C), 128.7 (d, J = 11.7, Ar-C), 35.2 (d, J = 2.4, $-\text{CH}_2-$), 29.7 (s, $-\text{CH}_3$), 23.2 (d, J = 73.8, $-\text{CH}_2-$). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 31.6 (s).

Synthesis of bis{2-(diphenylphosphinyl)ethyl}sulfone (Table 1, entry 6). The general method was followed with HP(O)Ph_2 (0.050 g, 0.25 mmol) and divinylsulfone (12.4 μL , 12.5 mmol). The maximum power setting (50 watts) was maintained until a temperature of 120 °C was achieved (110 s). The power was then reduced to 22 watts for the remainder of the reaction to maintain (120 °C). After 60 minutes, the reaction was cooled to room temperature to afford 0.064 g (99 %) of the title compound (97 % pure).

Trituration of the residue with ether (2 mL) afforded 0.062 g (95 %, 99% pure) of the title compound. Anal. Calcd for $\text{C}_{28}\text{H}_{28}\text{O}_4\text{P}_2\text{S}$: C, 64.36; H, 5.40. Found: C, 64.22; H, 5.38.

^1H NMR (CDCl_3 , 19 °C): δ 7.79 – 7.65 (m, 8H, Ar-H), 7.57 – 7.45 (m, 12H, Ar-H), 3.19 (m, 4H, $-\text{CH}_2-$), 2.72 (m, 4H, $-\text{CH}_2-$). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 132.5 (d, J = 2.7, Ar-C), 130.9 (d, J = 101.9, Ar-C), 130.6 (d, J = 9.6, Ar-C), 129.0 (d, J = 12.0, Ar-C), 45.9 (s, $-\text{CH}_2-$), 22.2 (d, J = 68.4, $-\text{CH}_2-$). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 29.2 (s).

Synthesis of 2-(diphenylphosphinylolethyl)-ethylsulfone (Table 1, entry 7). The general method was followed with HP(O)Ph_2 (0.050 g, 0.25 mmol) and ethylvinylsulfone (25.8 mL, 25 mmol). The maximum power setting (50 watts) was maintained until a temperature of 120 °C was achieved (137 s). The power was then reduced to 17 watts for the remainder of the reaction to maintain 120 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.077g (97%) of the title compound (95 % pure). Trituration of the residue with ether/hexane (1:1; 2 mL) afforded 0.65 g, (82 %, 99 % pure) of the title compound. Anal. Calcd for $\text{C}_{16}\text{H}_{19}\text{O}_3\text{PS}$: C, 59.61; H, 5.94. Found: C, 59.07; H, 5.95. (^1H NMR (CDCl_3 , 25 °C): δ 7.79 – 7.72 (m, 4H, Ar-H), 7.58 – 7.47 (m, 6H, Ar-H), 3.19 (m, 2H, $-\text{CH}_2-$), 3.02 (q, 2H, $J = 7.5$, $-\text{CH}_2-$), 2.75 (m, 2H, $-\text{CH}_2-$), 1.37 (t, 3H, $J = 7.50$, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 132.4 (d, $J = 2.9$, Ar-C), 131.1 (d, $J = 101.5$, Ar-C), 130.6 (d, $J = 9.4$, Ar-C), 129.0 (d, $J = 11.9$, Ar-C), 47.5 (s, $-\text{CH}_2-$), 44.7 (d, $J = 2.0$, $-\text{CH}_2-$), 21.9 (d, $J = 69.1$, $-\text{CH}_2-$), 6.5 (s, $-\text{CH}_3$). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 29.5 (s).

Synthesis of 2-[2-diphenylphosphinyl]ethyl]-pyridine (Table 1, entry 8).⁵ The general method was followed with HP(O)Ph_2 (0.050 g, 0.25 mmol) and 2-vinylpyridine (26.7 μL , 0.25 mmol). The maximum power setting of 50 watts was maintained until the desired temperature was reached (118 s). The power was then reduced to 11 watts for the remainder of the reaction to maintain the temperature. After 60 minutes, the reaction was cooled to room temperature to afford 0.074 g (98%) of the title compound (96% pure). Trituration with ether:hexane (1:1, 1 mL) afforded 0.067 g (88%, 98% pure) of the title compound. ^1H NMR (CDCl_3 , 19 °C): δ 8.49 (d, 1H, $J = 4.2$, Ar-H), 7.82 – 7.75 (m, 4H,

Ar-H), 7.56-7.42 (m, 7H, Ar-H), 7.14 (d, 1H, $J = 7.8$, Ar-H), 7.09 (m, 1H, Ar-H), 3.12 (m, 2H, -CH₂-), 2.78 (m, 2H, -CH₂-). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl₃, 19 °C): δ 160.2 (d, $J = 14.6$, Ar-C), 149.2 (s, Ar-C), 136.5 (s, Ar-C), 132.8 (d, $J = 98.6$, Ar-C), 131.7 (d, $J = 3$, Ar-C), 130.8 (d, $J = 9.3$, Ar-C), 128.6 (d, $J = 11.6$, Ar-C), 123.1 (s, Ar-C), 121.5 (s, Ar-C), 29.6 (d, $J = 2.0$, -CH₂-), 29.1 (d, $J = 73.4$, -CH₂-). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl₃, 19 °C): δ 31.3 (s).

Synthesis of 6H-Dibenz[c,e][1,2]oxaphosphorin-6-(propanoic acid, butyl ester)-6-

oxide (Table 2 entry 1). The general method was followed with DOPO (0.050 g, 0.23 mmol) and butylacrylate (33.2 μL , 0.23 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (130 °C) was reached (181 s). The power was then reduced to 22 watts for the remainder of the reaction to maintain 130 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.077 g (98%) of the title compound (95% pure). Purification by column chromatography (silica ether/THF 4:1) afforded 0.63 g (80%, 98% pure). Anal. Calcd for C₁₉H₂₁O₄P: C, 66.27; H, 6.15. Found: C, 66.15; H, 6.06. ^1H NMR (CDCl₃, 19 °C): δ 7.99-7.85 (m, 3H, Ar-H), 7.72 (t, 1H, $J = 7.6$, Ar-H), 7.53 (dt, 1H, $J = 7.3, 2.6$, Ar-H), 7.39 (t, 1H, $J = 7.6$, Ar-H), 7.28-7.21 (m, 2H, Ar-H), 4.03 (t, 2H, $J = 6.6$, -OCH₂-), 2.63 (m, 2H, -CH₂-), 2.40 (m, 2H, -CH₂-), 1.55 (m, 2H, -CH₂-), 1.32 (m, 2H, -CH₂-), 0.90 (t, 3H, $J = 7.3$, -CH₂-). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl₃, 19 °C): δ 171.7 (d, $J = 18.0$, C=O), 148.9 (d, $J = 8.1$, quat), 135.7 (d, $J = 6.1$, quat), 133.4 (d, $J = 2.3$, Ar-C), 130.6 (s, Ar-C), 130.0 (d, $J = 11.0$, Ar-C), 128.5 (d, $J = 13.3$, Ar-C), 125.1 (s, Ar-C), 124.7 (s, Ar-C), 123.9 (d, $J = 9.5$, Ar-C), 123.8 (d, $J = 123.9$, quat), 122.0 (d, $J = 11.0$, Ar-C), 120.4 (d, $J = 6.1$, Ar-C), 64.9 (s, -OCH₂-), 30.4 (s,

-CH₂-), 26.6 (d, J = 2.9, -CH₂-), 23.9 (d, J = 99.3, -CH₂-), 18.9 (s, -CH₂-), 13.6 (s, -CH₃).

³¹P {¹H} (CDCl₃, 19 °C): δ 35.7 (s).

Synthesis of 6H-Dibenz[c,e][1,2]oxaphosphorin-6-(2-methyl-propanoic acid, methyl ester)-6-oxide (Table 2 entry 2). The general method was followed with DOPO (0.050

g, 0.23 mmol) and methyl methacrylate (15.2 μL, 0.23 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (130 °C) was reached (200 s). The power was reduced to watts for the remainder of the reaction to maintain 130 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.70 g (97%) of the title compound as a colorless solid (90% pure). Purification by column chromatography (silica THF/hexane 1/1) afforded 0.058 g (81%, 98% pure) of the title compound. Analysis by ³¹P NMR spectroscopy of the crude reaction mixture as well as the product obtained from the chromatography revealed a diastereomeric ratio of 1:1.1.

Anal. Calcd for C₁₇H₁₇O₄P: C, 64.56; H, 5.42. Found: C, 64.61; H, 5.78. ¹H NMR (CDCl₃, 19 °C, both diastereomers): δ 7.98-7.84 (m, 3H, Ar-H), 7.69 (t, 1H, J = 7.7, Ar-H), 7.51 (dd, 1H, J = 7.4, 2.8, Ar-H), 7.38 (t, 1H, J = 8.2, Ar-H), 7.28-7.17 (m, 2H, Ar-H), 3.58 (s, 3H, -OMe, one diastereomer), 3.57 (s, 3H, -OMe, second diastereomer), 2.98 (m, 1H, -CH- or -CH₂-), 2.59 (m, 1H, -CH- or -CH₂-), 2.13 (m, 1H, -CH- or -CH₂-), 1.36 (d, 3H, J = 7.6, -CH₃, one diastereomer), 1.33 (d, 3H, J = 7.6, -CH₃, second diastereomer). ¹³C {¹H} NMR (CDCl₃, 19 °C, both diastereomers): δ 175.4 (d, J = 12.3, C=O), 175.1 (d, J = 10.11, C=O), 148.9 (d, J = 8.5, quat), 148.8 (d, J = 8.2, quat), 135.6 (d, J = 6.6, quat), 135.4 (d, J = 6.1, quat), 133.3 (d, J = 2.3, Ar-C), 133.2 (d, J = 2.2, Ar-C), 130.6 (s, Ar-C), 130.5 (s, Ar-C), 130.1 (d, J = 11.2, Ar-C), 130.1 (d, J = 10.6, Ar-C),

128.4 (d, $J = 13.3$, Ar-C), 128.3 (d, $J = 13.4$, Ar-C), 125.1 (s, Ar-C), 125.0 (s, Ar-C), 124.4 (d, $J = 121.1$, Ar-C), 124.3 (d, $J = 120.7$, Ar-C), 123.8 (d, $J = 9.7$, Ar-C), 123.7 (d, $J = 9.3$, Ar-C), 122.2 (d, $J = 11.1$, quat), 121.9 (d, $J = 10.9$, quat), 120.4 (d, $J = 6.2$, Ar-C), 120.3 (d, $J = 6.3$, Ar-C), 52.0 (s, -OMe), 51.9 (s, -OMe), 33.7 (d, $J = 3.6$, -CH-), 33.6 (d, $J = 3.0$, -CH-), 32.2 (d, $J = 97.4$, -CH₂-), 31.7 (d, $J = 96.5$, -CH₂-), 18.99 (d, $J = 9.2$, -Me), 18.6 (d, $J = 8.4$, -Me). ³¹P {¹H} (CDCl₃, 19 °C): δ 35.4 (s, one diastereomer), 34.4 (s, second diastereomer).

Synthesis of 6H-Dibenz[c,e][1,2]oxaphosphorin, 6-propanenitrile, 6-oxide (Table 2,

entry 3).⁶ The general method was followed with DOPO (0.050 g, 0.23 mmol) and acrylonitrile (15.2 μ L, 0.23 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (130 °C) was reached (110 s). The power was reduced to 32 watts for the remainder of the reaction to maintain 130 °C. After 60 minutes, the reaction was cooled to room temperature to afford 0.061 g (99%) of the title compound (96 % pure). Trituration with ether (1 mL) afforded 0.055 g (89%, 98% pure) of the title compound. ¹H NMR (CDCl₃, 25 °C): δ 8.03-7.86 (m, 3H, Ar-H), 7.77 (t, 1H, $J = 7.7$, Ar-H), 7.77 (td, 1H, $J = 7.5, 3.0$, Ar-H), 7.43 (t, 1H, $J = 7.7$, Ar-H), 7.32-7.26 (m, 2H, Ar-H), 2.71 (m, 2H, -CH₂-) 2.38 (m, 2H, -CH₂-). ¹³C {¹H} NMR (CDCl₃, 19 °C): δ 148.7 (d, $J = 8.5$, quat), 136.0 (d, $J = 6.5$, quat), 134.1 (d, $J = 2.4$, Ar-C), 131.0 (s, Ar-C), 130.1 (d, $J = 10.7$, Ar-C), 128.8 (d, $J = 13.4$, Ar-C), 125.3 (s, Ar-C), 125.1 (s, Ar-C), 124.4 (d, $J = 130.7$, quat), 124.2 (d, $J = 9.9$, Ar-C), 121.9 (d, $J = 10.8$, quat), 120.4 (d, $J = 6.3$, Ar-C), 117.9 (d, $J = 17.7$, quat), 24.7 (d, $J = 97.2$, -CH₂-), 10.6 (d, $J = 2.9$, -CH₂-). ³¹P {¹H} (CDCl₃, 19 °C): δ 31.9 (s).

Synthesis of 4-(6-oxido-6H-dibenz[c,e][1,2]oxaphosphorin-6-yl)-2-butanone (Table 2

entry 4). The general method was followed with DOPO (0.050 g, 0.23 mmol) and methyl vinyl ketone (19.3 μ L, 0.23 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (130 $^{\circ}$ C) was reached (107 s). The power was then reduced to 23 watts for the remainder of the reaction to maintain the temperature. After 60 minutes, the reaction was cooled to room temperature to afford 0.64 g (95%) of the title compound as a colorless solid (97% pure). Recrystallization from ether/hexane (1:6) afforded 0.055 g (83%, 99% pure) of the title compound. Anal. Calcd for $C_{16}H_{15}O_3P$: C, 67.13; H, 5.28. Found: C, 67.09; H, 5.10. 1H NMR ($CDCl_3$, 19 $^{\circ}$ C): δ 8.00-7.85 (m, 3H, Ar-H), 7.72 (t, 1H, $J = 7.9$, Ar-H), 7.53 (m, 1H, Ar-H), 7.39 (t, 1H, $J = 7.8$, Ar-H), 7.27 (t, 1H, $J = 7.6$, Ar-H), 7.22 (d, 1H, $J = 7.9$, Ar-H), 2.90 - 2.77 (m, 2H, -CH₂-), 2.40 - 2.23 (m, 2H, -CH₂-), 2.16 (s, 3H, -CH₃). $^{13}C\{^1H\}$ NMR ($CDCl_3$, 19 $^{\circ}$ C): δ 205.4 (d, $J = 14.0$, -C=O), 148.9 (d, $J = 8.0$, quat), 135.6 (d, $J = 6.2$, quat), 133.4 (d, $J = 2.3$, Ar-C), 130.6 (s, Ar-C), 129.9 (d, $J = 10.9$, Ar-C), 128.5 (d, $J = 13.1$, Ar-C), 125.2 (s, Ar-C), 124.7 (s, Ar-C), 124.2 (d, $J = 121.0$, Ar-C), 123.9 (d, $J = 9.58$, Ar-C), 122.2 (d, $J = 10.9$, Ar-C), 120.4 (d, $J = 6.0$, Ar-C), 35.2 (d, $J = 3.2$, -CH₂-), 29.6 (s, -CH₃), 22.2 (d, $J = 95.3$, -CH₂-). $^{31}P\{^1H\}$ NMR ($CDCl_3$, 19 $^{\circ}$ C): δ 37.0 (s).

Synthesis of Bis-(6-oxido-6H-dibenz[c,e][1,2]oxaphosphorin-6-yl)-sulfone (Table 2

entry 5). The general method was followed with DOPO (0.050 g, 0.23 mmol) and divinyl sulfone (11.6 μ L, 0.115 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (130 $^{\circ}$ C) was reached (172 s). The power was

50 watts for the remainder of the reaction to maintain the temperature. After 60 minutes, the reaction was cooled to room temperature to afford 0.63 g (99%) of the title compound as a colorless solid (98 % pure). Trituration with ether (2 mL) afforded 0.58 g (91%, 99% pure) of the title compound. Analysis of the $^{31}\text{P}\{^1\text{H}\}$ NMR spectrum revealed a 1:1 mixture of diastereomers with nearly identical chemical shifts. Anal. Calcd for $\text{C}_{28}\text{H}_{24}\text{O}_6\text{P}_2\text{S}$: C, 61.09; H, 4.39. Found: C, 60.81; H, 4.70. ^1H NMR (CDCl_3 , 19 °C): δ 8.03 – 7.85 (m, 6H, Ar-H), 7.77 (t, 2H, $J = 7.8$, Ar-H), 7.57 (m, 2H, Ar-H), 7.42 (t, 2H, $J = 7.6$, Ar-H), 7.30 (t, 2H, $J = 7.60$, Ar-H), 7.23 (d, 2H, $J = 8.0$, Ar-H), 3.38 – 3.21 (m, 4H, $-\text{CH}_2-$), 2.55 – 2.42 (m, 4H, $-\text{CH}_2-$). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 148.6 (d, $J = 8.2$, quat), 135.9 (d, $J = 6.7$, quat), 134.2 (d, $J = 2.3$, Ar-C), 131.1 (s, Ar-C), 130.1 (d, $J = 10.9$, Ar-C), 129.0 (d, $J = 13.7$, Ar-C), 125.4 (s, Ar-C), 125.2 (s, Ar-C), 124.2 (d, $J = 9.9$, Ar-C), 122.8 (d, $J = 124.8$, Ar-C), 121.8 (d, $J = 11.2$, Ar-C), 120.4 (d, $J = 6.4$, Ar-C), 45.9 (d, $J = 2.8$, $-\text{CH}_2-$), 21.1 (d, $J = 95.5$, $-\text{CH}_2-$). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 32.14 (s, one diastereomer), 32.13 (s, second diastereomer).

Synthesis of 2-[2-(6-oxido-6H-dibenz[c,e][1,2]oxaphosphorin-6-yl)ethyl]-pyridine

(Table 2 entry 6). The general method was followed with DOPO (0.050 g, 0.23 mmol) and 2-vinylpyridine (24.9 μL , 0.23 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (140 °C) was reached (130 s). The power was then reduced to 11 watts for the remainder of the reaction to maintain the temperature. After 60 minutes, the reaction was cooled to room temperature to afford 0.72 g, (97 %) of the title compound as a colorless solid (92 % pure). Trituration with ether (1 mL) afforded 0.055 g (75%, 98 % pure) of the title compound. Anal. Calcd for $\text{C}_{19}\text{H}_{16}\text{NO}_2\text{P}$:

C, 71.02; H, 5.02. Found: C, 70.85; H, 5.69. ^1H NMR (CDCl_3 , 19 °C): δ 8.44 (d, 1H, J = 4.35, Ar-H), 7.97 – 7.83 (m, 3H, Ar-H), 7.68 (t, 1H, J = 7.6, Ar-H) 7.56 (td, 1H, J = 7.7, 2.1, Ar-H), 7.48 (td, 1H, J = 7.4, 3.0, Ar-H), 7.35 (t, 1H, J = 7.7, Ar-H), 7.24 (t, 1H, J = 7.6, Ar-H), 7.16 – 7.08 (m, 3H, Ar-H), 3.12 (m, 2H, $-\text{CH}_2-$), 2.56 (m, 2H, $-\text{CH}_2-$).

$^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 159.4 (d, J = 15.3, Ar-C), 149.1 (s, Ar-C), 149.1 (d, J = 8.4, Ar-C), 136.5 (s, Ar-C), 135.6 (d, J = 5.9, Ar-C), 133.2 (d, J = 2.3, Ar-C), 130.5 (s, Ar-C), 130.1 (d, J = 11.0, Ar-C), 128.4 (d, J = 13.4, Ar-C), 125.1 (s, Ar-C), 124.5 (s, Ar-C), 124.3 (d, J = 114.7, Ar-C), 123.8 (d, J = 9.4, Ar-C), 122.8 (s, Ar-C), 122.1 (d, J = 10.6, Ar-C), 121.5 (s, Ar-C), 120.4 (d, J = 6.4, Ar-C), 29.9, (d, J = 3.31, $-\text{CH}_2-$), 28.0 (d, J = 96.9, $-\text{CH}_2-$). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 36.8 (s).

Synthesis of 2-diphenylphosphinyl-1-phenyl-1-(6-oxido-6H-

dibenz[c,e][1,2]oxaphosphorin-6-yl)ethane (Table 3 entry 1). The general method was followed with (*E*)-1-phenyl-2-(diphenylphosphinyl)ethene (0.047 g, 0.15 mmol) and DOPO (0.033 g, 0.15 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (140 °C) was reached (378 s). The power was reduced to 40 watts for the remainder of the reaction to maintain 140 °C. After 60 minutes, the reactor vial was cooled to room temperature to afford 0.079 g, (98%) of the title compound (90% pure, 1:1.7 mixture of diastereomers). The residue was triturated with a EtOAc/ether (1:1) to afford 0.055 g (68%) of the title compound (98% pure) as a 1:1.7 mixture of diastereomers. Anal. Calcd for $\text{C}_{32}\text{H}_{26}\text{O}_3\text{P}_2$: C, 73.84; H, 5.03. Found: C, 73.97; H, 5.08. ^1H NMR (CDCl_3 , 19 °C): δ 7.98 (m, 1H, Ar-H), 7.84 – 7.0 (m, 18H, Ar-H), 6.97 – 6.78 (m, 4H, Ar-H), 3.84 – 3.65 (m, 1H, $-\text{CH}-$, or $-\text{CH}_2-$), 3.26 – 3.10 (m, 1H, $-\text{CH}-$, or $-\text{CH}_2-$

), 3.07 – 2.93 (m, 1H, -CH-, or -CH₂-). ¹³C{¹H} NMR (CDCl₃, 19 °C, low frequency region): δ 40.0 (dd, J = 85.3, 3.6, -CH- both diastereomers), 28.3 (d, J = 76.7, -CH₂- major diastereomer), 28.2 (d, J = 69.3, -CH₂- minor diastereomer). ³¹P{¹H} NMR (CDCl₃, 19 °C): δ 35.1 (d, J = 51.4, major diastereomer), 34.9 (d, J = 56.4, minor diastereomer), 27.9 (d, J = 51.4, major diastereomer), 27.8 (d, J = 56.4, minor diastereomer).

Synthesis of 2-diphenylphosphinyl-2-(6-oxido-6H-dibenz[c,e][1,2]oxaphosphorin-6-yl)heptane (Table 3 entry 2). The general method was followed with (*E*)-1-

(diphenylphosphinyl)-1-heptene (0.037 g, 0.12 mmol) and DOPO (0.27 g, 0.12 mmol).

The maximum power setting of 50 watts was maintained until the desired temperature (140 °C) was reached (367 s). The power was then reduced to 35 watts for the remainder of the reaction to maintain the temperature. After stirring for 60 minutes, the reaction was cooled to room temperature, triturated with ether/hexane (1:5, 1 mL) to afford 0.051 g (80 %) of the title compound (98 % pure, diastereomeric ratio = 1:2.7). Anal. Calcd for C₃₁H₃₂O₃P₂: C, 72.36; H, 6.27. Found: C, 71.98; H, 6.13. ¹H NMR (CDCl₃, 19 °C, major diastereomer): δ 7.93 – 7.89 (m, 1H, Ar-H), 7.82 – 7.78 (m, 2H, Ar-H), 7.70 – 7.65 (m, 3H, Ar-H), 7.54 – 7.33 (m, 10H, Ar-H), 7.24 – 7.17 (m, 2H, Ar-H), 2.82 – 2.72 (m, 1H, -CH- or -CH₂-), 2.53 – 2.41 (m, 2H, -CH- or -CH₂-), 1.84 (m, 2H, -CH₂-), 1.34 – 1.24 (m, 2H, -CH₂-), 1.12 – 1.02 (m, 2H, -CH₂-), 0.97 – 0.88 (m, 2H, -CH₂-), 0.72 (t, J = 7.4, -CH₃). ¹³C{¹H} NMR (CDCl₃, 19 °C, low frequency region, major diastereomer): δ 32.2 (d, J = 94.7, -CH-), 31.7 (s, -CH₂-), 28.8 (d, J = 4.2, -CH₂-), 26.9 (d, J = 69.4, -CH₂-), 26.9 (d, J = 3.9, -CH₂-), 22.2 (s, CH₂-), 13.9 (s, -CH₃). ³¹P{¹H} NMR (CDCl₃, 19 °C,

both diastereomers): δ 39.9 (d, $J = 54.2$, $-\text{P}(\text{O})(\text{O}^\wedge\text{C})$, minor diastereomer), 39.4 (d, $J = 55.7$, $-\text{P}(\text{O})(\text{O}^\wedge\text{C})$, major diastereomer), 29.9 (d, $J = 55.7$, $-\text{P}(\text{O})\text{Ph}_2$, major diastereomer), 29.2 (d, $J = 54.2$, $-\text{P}(\text{O})\text{Ph}_2$, minor diastereomer).

Synthesis of 2-diphenylphosphinyl-1-(4-tolyl)-1-(6-oxido-6H-

dibenz[c,e][1,2]oxaphosphorin-6-yl)ethane (Table 3 entry 3). The general method was followed with (*E*)-1-(4-tolyl)-2-(diphenylphosphinyl)ethene (0.026 g, 0.082 mmol) DOPO (0.018 g, 0.082 mmol). The maximum power setting of 50 watts was maintained until the desired temperature (140 °C) was reached (301 s). The power was then reduced to 36 watts for the remainder of the reaction to maintain the temperature. The reaction was cooled to room temperature to afford 0.043 g, (97 %) of the title compound as a colorless solid (92 % pure). Trituration of the residue with ether/EtOAc (10:1, 1 mL) afforded 0.031 g (71 %, 98 % pure). Analysis of the $^{31}\text{P}\{^1\text{H}\}$ NMR revealed a mixture of diastereomers (1:1.9). Anal. Calcd for $\text{C}_{33}\text{H}_{28}\text{O}_3\text{P}_2$: C, 74.15; H, 5.28. Found: C, 74.31; H, 5.63. ^1H NMR (CDCl_3 , 19 °C, both diastereomers): δ 7.99 – 6.64 (m, 22H, Ar-H), 3.72 (m, $-\text{CH}-$ or $-\text{CH}_2-$), 3.20 (m, $-\text{CH}-$ or $-\text{CH}_2-$), 3.00 (m, $-\text{CH}-$ or $-\text{CH}_2-$), 2.13 (s, 3H, $-\text{CH}_3$). $^{13}\text{C}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C, both diastereomers, low frequency region): δ 39.6 (dd, $J = 90.3, 4.1$, $-\text{CH}-$, minor diastereomer), 39.3 (dd, $J = 89.4, 3.7$, $-\text{CH}-$, major diastereomer), 28.7 (d, $J = 69.7$, $-\text{CH}_2-$, major diastereomer), 28.6 (dd, $J = 70.2, 4.8$, $-\text{CH}_2-$, minor diastereomer). $^{31}\text{P}\{^1\text{H}\}$ NMR (CDCl_3 , 19 °C): δ 35.0 (d, $J = 57.2$, $-\text{P}(\text{O})(\text{O}^\wedge\text{C})$, major diastereomer), 35.0 (d, $J = 52.5$, $-\text{P}(\text{O})(\text{O}^\wedge\text{C})$, minor diastereomer), 27.8 (d, $J = 52.5$, $-\text{P}(\text{O})\text{Ph}_2$, minor diastereomer), 27.7 (d, $J = 57.2$, $-\text{P}(\text{O})\text{Ph}_2$, major diastereomer).

Control reactions. The general procedure was followed using acrylonitrile, methyl vinyl ketone, and HP(O)Ph_2 as model substrates. The alkenes were purified by distillation and used in the synthetic approach. Analysis of the crude reaction mixtures did not reveal a significant difference between the bulk and distilled material. Since the reactions did not generate much pressure under the listed conditions, reactions were carried out without the pressure sensor (16 guage S.S. needle). No significant difference was observed.

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Current Data Parameters
NAME ras102804.001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041028
Time 20.06
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4132.231 Hz
FIDRES 0.252211 Hz
AQ 1.9825140 sec
RG 256
DM 121.000 usec
DE 172.86 usec
TE 300.0 K
D1 1.00000000 sec
P1 10.00 usec
SF01 300.1350731 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 8192
SF 300.1333595 MHz
WDW EM
SSB 0
LB 0.25 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.041 ppm
F1 2413.49 Hz
F2P -0.113 ppm
F2 -33.99 Hz
PPMCM 0.40773 ppm/cm
HZCM 122.37392 Hz/cm

Current Data Parameters
NAME ras102804.002
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041028
Time 20.08
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 23
DS 2
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864820 sec
RG 16384
DW 12.000 usec
DE 17.14 usec
TE 300.0 K
D12 0.0000200 sec
DL6 21.00 dB
D1 1.00000000 sec
CPDPRG waltz16
P31 105.00 usec
D11 0.0300000 sec
DL5 21.00 dB
P1 4.00 usec
SFO1 121.5022852 MHz
NUCLEUS 31P

F2 - Processing parameters
SI 32768
SF 121.4963165 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.600 ppm
F1 26802.06 Hz
F2P -122.346 ppm
F2 -14864.60 Hz
PPMCM 17.14730 ppm/cm
HZCM 2083.33325 Hz/cm

Current Data Parameters

NAME ras110204.001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20041102
Time 11.42
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 9
DS 0
SWH 4132.231 Hz
FIDRES 0.252211 Hz
AQ 1.9825140 sec
RG 1024
DW 121.000 usec
DE 172.86 usec
TE 300.0 K
D1 30.0000000 sec
P1 10.00 usec
SFO1 300.1350731 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 8192
SF 300.1333656 MHz
WDW EM
SSB 0
LB 0.25 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.233 ppm
F1 2470.87 Hz
F2P -0.119 ppm
F2 -35.57 Hz
PPMCM 0.41755 ppm/cm
HZCM 125.32177 Hz/cm

Current Data Parameters
NAME ras110104.004
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041102
Time 7.10
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 24
DS 2
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864820 sec
RG 32768
DW 12.000 usec
DE 17.14 usec
TE 300.0 K
D12 0.0000200 sec
DL6 21.00 dB
D1 1.00000000 sec
CPDPRG waltz16
P31 105.00 usec
D11 0.0300000 sec
DL5 21.00 dB
P1 4.00 usec
SF01 121.5022852 MHz
NUCLEUS 31P

F2 - Processing parameters
SI 32768
SF 121.4963165 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 220.600 ppm
F1 26802.06 Hz
F2P -122.346 ppm
F2 -14864.60 Hz
PPMCM 17.14730 ppm/cm
HZCM 2083.33325 Hz/cm

Current Data Parameters

NAME ras110104.002
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20041101
Time 21.12
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgdc
TD 65536
SOLVENT CDCl₃
NS 10000
DS 4
SWH 20000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6384500 sec
RG 45500
DW 25.000 usec
DE 35.71 usec
TE 300.0 K
D12 0.0000200 sec
DL5 19.00 dB
CPDPRG waltz16
P31 90.00 usec
D1 1.00000000 sec
P1 5.50 usec
SF01 75.4767000 MHz
NUCLEUS ¹³C
D11 0.0300000 sec

F2 - Processing parameters

SI 32768
SF 75.4685989 MHz
WDW EM
SSB 0
LB 0.50 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 239.850 ppm
F1 18101.12 Hz
F2P -25.161 ppm
F2 -1898.88 Hz
PPMCM 13.25054 ppm/cm
HZCM 1000.00000 Hz/cm

Current Data Parameters
NAME ras091504.001
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040915
Time 7.18
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 9
DS 0
SWH 4132.231 Hz
FIDRES 0.252211 Hz
AQ 1.9825140 sec
RG 1024
DW 121.000 usec
DE 172.86 usec
TE 300.0 K
D1 1.00000000 sec
P1 10.00 usec
SF01 300.1350731 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 8192
SF 300.1333889 MHz
WDW EM
SSB 0
LB 0.25 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
F1P 8.555 ppm
F1 2567.54 Hz
F2P 0.529 ppm
F2 158.68 Hz
PPMCM 0.40130 ppm/cm
HZCM 120.44296 Hz/cm

Current Data Parameters

NAME ras091504.003
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20040915
Time 9.09
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 19
DS 2
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864820 sec
RG 32768
DW 12.000 usec
DE 17.14 usec
TE 300.0 K
D12 0.0000200 sec
DL6 21.00 dB
D1 1.00000000 sec
CPDPRG waltz16
P31 105.00 usec
D11 0.0300000 sec
DL5 21.00 dB
P1 4.00 usec
SF01 121.5022852 MHz
NUCLEUS 31P

F2 - Processing parameters

SI 32768
SF 121.4963165 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters

CX 20.00 cm
F1P 220.600 ppm
F1 26802.06 Hz
F2P -122.346 ppm
F2 -14864.60 Hz
PPMCM 17.14730 ppm/cm
HZCM 2083.33325 Hz/cm

Current Data Parameters
 NAME ras091504.002
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20040915
 Time 7.48
 INSTRUM arx300
 PROBHD 5 mm Multinu
 PULPROG zgdc
 TD 65536
 SOLVENT CDC13
 NS 2283
 DS 4
 SWH 20000.000 Hz
 FIDRES 0.305176 Hz
 AQ 1.6384500 sec
 RG 45500
 DW 25.000 usec
 DE 35.71 usec
 TE 300.0 K
 D12 0.0000200 sec
 DL5 19.00 dB
 CPDPRG waltz16
 P31 90.00 usec
 D1 1.00000000 sec
 P1 5.50 usec
 SF01 75.4767000 MHz
 NUCLEUS 13C
 D11 0.0300000 sec

F2 - Processing parameters
 SI 32768
 SF 75.4685995 MHz
 WDW EM
 SSB 0
 LB 0.50 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 208.263 ppm
 F1 15717.34 Hz
 F2P 17.555 ppm
 F2 1324.81 Hz
 PPMCM 9.53544 ppm/cm
 HZCM 719.62616 Hz/cm

Current Data Parameters

NAME ras102704.008
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters

Date_ 20041027
Time 15.49
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 8
DS 0
SWH 4132.231 Hz
FIDRES 0.252211 Hz
AQ 1.9825140 sec
RG 1430
DW 121.000 usec
DE 172.86 usec
TE 300.0 K
D1 1.00000000 sec
P1 10.00 usec
SF01 300.1350731 MHz
NUCLEUS 1H

F2 - Processing parameters

SI 8192
SF 300.1333641 MHz
WDW EM
SSB 0
LB 0.25 Hz
GB 0
PC 1.00

1D NMR plot parameters

CX 20.00 cm
F1P 8.969 ppm
F1 2692.01 Hz
F2P 1.348 ppm
F2 404.65 Hz
PPMCM 0.38106 ppm/cm
HZCM 114.36813 Hz/cm

Current Data Parameters
NAME ras102704.010
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041027
Time 19.39
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 60
DS 2
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864820 sec
RG 32768
DW 12.000 usec
DE 17.14 usec
TE 300.0 K
D12 0.0000200 sec
DL6 21.00 dB
D1 1.00000000 sec
CPDPRG waltz16
P31 105.00 usec
D11 0.0300000 sec
DL5 21.00 dB
P1 4.00 usec
SF01 121.5022852 MHz
NUCLEUS 31P

F2 - Processing parameters
SI 32768
SF 121.4935392 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 243.464 ppm
F1 29579.31 Hz
F2P -99.490 ppm
F2 -12087.35 Hz
PPMCM 17.14769 ppm/cm
HZCM 2083.33325 Hz/cm

Current Data Parameters
 NAME ras102604.002
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041026
 Time 21.29
 INSTRUM arx300
 PROBHD 5 mm Multinu
 PULPROG zgdc
 TD 65536
 SOLVENT CDCl3
 NS 13434
 DS 4
 SWH 20000.000 Hz
 FIDRES 0.305176 Hz
 AQ 1.6384500 sec
 RG 45500
 DW 25.000 usec
 DE 35.71 usec
 TE 300.0 K
 D12 0.0000200 sec
 DL5 19.00 dB
 CPDPRG waltz16
 P31 90.00 usec
 D1 1.00000000 sec
 P1 5.50 usec
 SF01 75.4767000 MHz
 NUCLEUS 13C
 D11 0.0300000 sec

F2 - Processing parameters
 SI 32768
 SF 75.4685989 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00

1D NMR plot parameters
 CX 20.00 cm
 F1P 165.857 ppm
 F1 12517.01 Hz
 F2P 16.324 ppm
 F2 1231.96 Hz
 PPMCM 7.47665 ppm/cm
 HZCM 564.25238 Hz/cm

Current Data Parameters
NAME ras120604.011
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041207
Time 22.05
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgpg30
TD 65536
SOLVENT Aceton
NS 24
DS 2
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864820 sec
RG 32768
DM 12.000 usec
DE 17.14 usec
TE 300.0 K
D12 0.0000200 sec
DL6 21.00 dB
D1 1.00000000 sec
CPOPRG waltz16
P31 105.00 usec
D11 0.0300000 sec
DL5 21.00 dB
P1 4.00 usec
SF01 121.5022852 MHz
NUCLEUS 31P

F2 - Processing parameters
SI 32768
SF 121.4963165 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 43.782 ppm
F1 5319.39 Hz
F2P 20.827 ppm
F2 2530.36 Hz
PPMCM 1.14778 ppm/cm
HZCM 139.45149 Hz/cm

Current Data Parameters
NAME ras100104.005
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041001
Time 16.17
INSTRUM arx300
PROBHD 5 mm Multinu
PULPROG zgig
TD 65536
SOLVENT Aceton
NS 483
DS 2
SWH 41666.668 Hz
FIDRES 0.635783 Hz
AQ 0.7864820 sec
RG 32768
DW 12.000 usec
DE 17.14 usec
TE 300.0 K
D11 0.0300000 sec
DL5 21.00 dB
D1 30.0000000 sec
P1 4.00 usec
SF01 121.5022852 MHz
NUCLEUS 31P
CPDPRG waltz16
P31 105.00 usec

F2 - Processing parameters
SI 32768
SF 121.4963165 MHz
WDW EM
SSB 0
LB 0.10 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
F1P 39.144 ppm
F1 4755.81 Hz
F2P 23.850 ppm
F2 2897.70 Hz
PPMCM 0.76468 ppm/cm
HZCM 92.90553 Hz/cm

*Impurity in the crude reaction mixture. The crude material is used for the diastereomeric ratio information due to differences in solubility between the diastereomers.

Current Data Parameters
 NAME ras121504.016
 EXPNO 1
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20041216
 Time 7.47
 INSTRUM arx300
 PROBHD 5 mm Multinu
 PULPROG zgig
 TD 65536
 SOLVENT Aceton
 NS 94
 DS 2
 SWH 41666.668 Hz
 FIDRES 0.635783 Hz
 AQ 0.7864820 sec
 RG 32768
 DW 12.000 usec
 DE 17.14 usec
 TE 300.0 K
 D11 0.0300000 sec
 DL5 21.00 dB
 D1 30.00000000 sec
 P1 4.00 usec
 SFO1 121.5022852 MHz
 NUCLEUS 31P
 CPDPRG waltz16
 P31 105.00 usec

F2 - Processing parameters
 S1 32768
 SF 121.4963165 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.40

1D NMR plot parameters
 CX 20.00 cm
 F1P 39.762 ppm
 F1 4830.94 Hz
 F2P 24.648 ppm
 F2 2994.60 Hz
 PPMCM 0.75572 ppm/cm
 HZCM 91.81709 Hz/cm