***t*-BuOK-Mediated Hydrophosphination of Functionalized Alkenes: A Novel Synthesis of Chiral P,N- and P,P-Ligands**

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General: All reactions were carried out with magnetic stirring and, if air or moisture sensitive, in flame dried glassware under argon. Syringes which were used to transfer reagents and solvents were purged with argon prior to use. Melting points were determined on a Büchi B-540 apparatus and are uncorrected. NMR spectra were recorded at room temperature in CDCl_3 on Bruker ARX 200, AC 300. Chemical shifts are given relative to the deuterated solvent peak: (δ_{H} : 7.27, δ_{C} : 77.0 ppm). For ^{31}P NMR, 85 % phosphoric acid was used as an external standard. Optical rotations were measured on a Perkin Elmer 241 polarimeter. IR spectra were recorded on a Nicolet 510 FT-IR spectrometer. Electron impact (EI) mass spectra were recorded on a Varian CD 7A. Enantiomeric excess were determined by HPLC analysis (chiral OD-H, AD, OJ and OB columns, Daicel Chemical Industries with *n*-heptane/2-propanol as mobile phase and detection by a diod array UV/Vis detector) or by GC analysis (Chiralsil-L-Val column with hydrogen as carrier gas). Racemic compounds were used to choose the operating conditions for the resolution of the two enantiomers. Column chromatography was carried out on silica gel 60 (70-230 mesh, ASTM). Hydrogenations were performed in 100 mL stainless steel autoclaves or a schenk tube with a hydrogen filled balloon for the reaction under 1 bar pressure.

Solvents

Solvents were dried according to standard methods by distillation over drying agents as stated below and were stored under argon: DMSO (CaH_2), THF (Na/benzophenone), triethylamine (KOH), CH_2Cl_2 and toluene (Na).

Reagents

- Reagents of >98 % purity were used as obtained.
- *n*-Butyllithium was used as 1.5 M solution in hexane.
- *t*-Butyllithium was used as 1.5 M solution in pentane.
- 1.7 M ZnBr_2 solution was prepared by drying ZnBr_2 (30.5 g, 0.14 mol) under vacuum at 120 °C for 5 h. After cooling to rt, dry THF (80 mL) was added and stirring was continued until the salt was dissolved.
- Diisopropylamine was distilled from CaH_2 .

(1*R*,4*R*)-1,7,7-Trimethylbicyclo[2.2.1]hept-2-en-2-yl trifluoromethanesulfonate (12)¹

A solution of (+)-camphor (**10**) (10 mmol, 1.52 g) in THF (15 mL) was added to a solution of LDA (10 mmol) in THF (40 mL) at $-78\text{ }^{\circ}\text{C}$ and stirred for 1 h. A solution of *N*-phenyltrifluoromethanesulfonimide ($\text{ Tf}_2\text{NPh}$) (10.7 mmol, 3.82 g) in THF (20 mL) was then added and the reaction mixture was stirred at $0\text{ }^{\circ}\text{C}$ for 14 h. The reaction mixture was added with 30 mL of saturated ammonium chloride and extracted with Et_2O . The organic phase was washed with water, brined and dried (MgSO_4). The residue was purified by chromatography on silica gel (pentane) to give **12** (2.56 g, 90 %) as a colourless liquid.

$[\alpha]_{\text{D}}^{23}$: +8.63 (*c* 1.07, CHCl_3).

$^1\text{H NMR}$ (300 MHz, CDCl_3): δ 5.59 (d, $J = 3.9\text{ Hz}$, 1H), 2.37 (t, $J = 3.9\text{ Hz}$, 1H), 1.90-1.80 (m, 1H), 1.65-1.54 (m, 1H), 1.30-1.22 (m, 1H), 1.12-1.03 (m, 1H), 0.95 (s, 3H), 0.85 (s, 3H), 0.71 (s, 3H).

$^{13}\text{C NMR}$ (75 MHz, CDCl_3): δ 155.6, 118.9 (q, $J = 318.0\text{ Hz}$), 118.0, 57.3, 54.2, 50.5, 31.2, 25.7, 20.0, 19.3, 9.8.

IR (KBr, cm^{-1}): 1623, 1423, 1391, 1212, 1142, 1111.

MS (EI, 70 ev): 284 (M^+ , 22), 151 (20), 123 (100), 95 (38), 81 (31), 55 (24).

(1*R*,5*S*)-6,6-Dimethylbicyclo[3.1.1]hept-2-en-2-yltrifluoromethanesulfonate (14)¹

A solution of (+)-nopinone (**13**) (1.80 g, 13 mmol) in THF (20 mL) was added to a solution of LDA (13 mmol) in THF (50 mL) at $-78\text{ }^{\circ}\text{C}$ and stirred for 1 h. A solution of $\text{ Tf}_2\text{NPh}$ (5.00 g, 14 mmol) in THF (20 mL) was then added and the reaction mixture was stirred at $0\text{ }^{\circ}\text{C}$ for 14 h. The residue was purified by flash chromatography (pentane) to give **14** (3.23 g, 92 %) as a colourless liquid.

$[\alpha]_{\text{D}}^{26}$: +23.5 (*c* 0.545, CHCl_3).

$^1\text{H NMR}$ (300 MHz, CDCl_3): δ 5.46 (m, 1H), 2.50-2.40 (m, 1H), 2.35-2.00 (m, 4H), 1.35-1.20 (m, 4H), 0.86 (s, 3H).

^{13}C NMR (75 MHz, CDCl_3): δ 155.4, 118.9 (q, $J = 315.0$ Hz), 111.8, 46.7, 40.5, 40.1, 32.1, 28.6, 25.9, 21.2.

IR (KBr, cm^{-1}): 1667, 1421, 1247, 1208, 1143, 1063, 1042.

Synthesis of di(2-furyl)phosphine oxide (18)

To a solution of bis(2-furyl)phosphine chloride² (1.2 g, 6.0 mmol) in CH_2Cl_2 (20 mL) was added H_2O (10 mL) at 0 °C. The reaction mixture was stirred at 0 °C for 0.5 h. CH_2Cl_2 and H_2O were added, and the resulting solution was washed with brine, dried (MgSO_4) and concentrated *in vacuo*. The crude product **18** showed the presence of the expected product ($\delta = -17.0$ ppm in ^{31}P NMR and GC MS; mass peak at 182) and was used in the next step without purification.

Synthesis of di(2-furyl)(1*R*,4*R*)-1,7,7-trimethylbicyclo[2.2.1]hept-2-en-2-yl)phosphine oxide (8b)

Alkenyll triflate **12** (341 mg, 1.2 mmol), difurylphosphine oxide (**18**) (255 mg, 1.4 mmol), *i*- Pr_2NEt (0.6 mL, 3 mmol) in toluene (8 mL). $\text{Pd}(\text{OAc})_2$ (13.5 mg, 60 μmol), dppb (25.6 mg, 60 μmol) in toluene (2 mL) were added and the reaction mixture was stirred at 60 °C for 15 min. 30 % H_2O_2 was added and the reaction mixture was stirred at 25 °C for 15 min. The mixture was diluted with CH_2Cl_2 (30 mL) and washed with water and brine. The solution was dried over MgSO_4 , filtered and the solvent was removed *in vacuo*. Purification by flash chromatography (20% Et_2O in CH_2Cl_2) furnished **8b** (220 mg, 58 %) as a yellow foam.

^1H NMR (300 MHz, CDCl_3): δ 7.64-7.61 (m, 2H), 7.09 (ddd, $J = 3.3, 2.1, 0.6$ Hz, 1H), 6.98 (ddd, $J = 3.3, 2.1, 0.6$ Hz, 1H), 6.63 (dd, $J = 12.9, 3.3$ Hz, 1H), 6.47-6.42 (m, 2H), 2.44-2.40 (m, 1H), 1.88-1.80 (m, 1H), 1.55-1.46 (m, 1H), 1.23-1.10 (m, 1H), 1.04-0.94 (m, 4H), 0.78 (s, 3H), 0.71 (s, 3H).

^{13}C NMR (75 MHz, CDCl_3): δ 152.1 (d, $J = 9.0$ Hz), 147.8 (d, $J = 10.8$ Hz), 146.7-146.6 (m, 1H), 145.8 (d, $J = 10.8$ Hz), 139.3 (d, $J = 117.2$ Hz), 121.2-120.8 (m), 110.0-109.8 (m), 57.4

(d, $J = 5.5$ Hz), 55.8 (d, $J = 9.3$ Hz), 52.4 (d, $J = 13.7$ Hz), 30.7, 22.7 (d, $J = 3.2$ Hz), 18.3, 18.0, 10.7.

^{31}P NMR (81 MHz, CDCl_3): δ 0.3.

IR (KBr, cm^{-1}): 1553, 1461, 1368, 1208, 1133, 1007, 911, 883.

EI (70 eV): 317 ($[\text{M}+\text{H}]^+$, 18.7), 316 (M^+ , 96), 301 (77), 273 (100).

$\text{C}_{18}\text{H}_{21}\text{O}_3\text{P}$ HRMS: Calcd.: 316.1221.
Found: 316.1228.

Synthesis of Ir-BARF complex-1³

To a two-necked flask fitted with a reflux condensor was added the P,N-ligand **1** (0.1 mmol), $[\text{Ir}(\text{cod})\text{Cl}]_2$ (33.6 mg, 0.05 mmol) and CH_2Cl_2 (5 mL) were heated to reflux at 45 °C for 1 h, until ^{31}P NMR indicated that the ligand was consumed. After cooling to 25 °C, $\text{Na}[\text{BARF}]$ (130 mg, 0.15 mmol) was added, followed by H_2O (5 mL) and the resulting two-phase mixture was stirred vigorously for 30 min. The residue was purified by column chromatography (50% CH_2Cl_2 in pentane) to afford Ir-1 (136 mg, 88 %) as an orange solid.

Mp: 173-177 °C.

^1H NMR (300 MHz, CDCl_3): δ 8.44-8.40 (m, 1H), 7.68-7.04 (m, 25 H), 4.78 (dd, $J = 23.0$, 11.3 Hz, 1H), 4.44-4.36 (m, 1H), 4.10-4.00 (m, 1H), 3.85-3.76 (m, 1H), 3.26-3.16 (m, 1H), 2.12-1.96 (m, 10H), 2.92-1.02 (m, 7H), 1.09 (s, 3H), 0.98 (s, 3H).

^{13}C NMR (75 MHz, CDCl_3): δ 163.5-161.1 (m), 151.7, 139.7, 135.2, 134.6 (d, $J = 12.6$ Hz), 133.6 (d, $J = 9.3$ Hz), 132.1, 131.8, 130.3-128.6 (m), 126.7, 123.5, 123.1, 122.8, 117.8 (d, $J = 3.8$ Hz), 93.9 (d, $J = 8.8$ Hz), 92.5 (d, $J = 14.6$ Hz), 66.4, 63.6, 61.5 (d, $J = 7.4$ Hz), 51.0, 49.0 (d, $J = 8.7$ Hz), 46.9 (d, $J = 3.8$ Hz), 46.2, 45.9, 37.3, 34.0 (d, $J = 15.2$ Hz), 28.7, 28.2, 26.7, 22.5, 20.7, 14.2.

^{31}P NMR (81 MHz, CDCl_3): δ 18.9.

$\text{C}_{67}\text{H}_{54}\text{BF}_4\text{IrNP}$	Calcd.: C, 51.48	H, 3.48	N, 0.90
	Found: C, 51.55	H, 3.39	N, 0.84

Synthesis of Ir-BARF complex-3

To a two-necked flask fitted with a reflux condensor was added the P,N-ligand **3** (98.8 mg, 0.22 mmol), [Ir(cod)Cl]₂ (74 mg, 0.11 mmol) and CH₂Cl₂ (10 mL) were heated to reflux at 45 °C for 1 h, until ³¹P NMR indicated that the ligand was consumed. After cooling to 25 °C, Na[BARF] (297 mg, 0.34 mmol) was added, followed by H₂O (10 mL) and the resulting two-phase mixture was stirred vigorously for 30 min. The residue was purified by column chromatography (50% CH₂Cl₂ in pentane) to afford Ir-**3** (312 mg, 88 %) as an orange solid.

Mp: 165-169 °C.

¹H NMR (200 MHz, CDCl₃): δ 8.43-8.39 (m, 1H), 7.68-7.04 (m, 27H), 4.77 (dd, *J* = 23.0, 11.0, 1H), 4.44-4.36 (m, 1H), 4.10-4.00 (m, 1H), 3.84-3.76 (m, 1H), 3.26-3.16 (m, 1H), 2.60-1.84 (m, 11H), 1.72-1.16 (m, 6H), 1.09 (s, 3H), 0.98 (s, 3H).

¹³C NMR (75 MHz, CDCl₃): δ 165.2-162.8 (m), 153.4, 141.4, 137.9 (d, *J* = 53 Hz), 136.9, 136.4 (d, *J* = 12.5 Hz), 135.3 (d, *J* = 9.4 Hz), 133.8 (d, *J* = 1.7 Hz), 133.5 (d, *J* = 2.1 Hz), 132.1-130.3 (m), 128.4, 126.6, 125.2-124.6 (m), 121.2, 119.6-119.5 (m), 95.6 (d, *J* = 8.7 Hz), 94.3 (d, *J* = 14.9 Hz), 68.2, 65.3, 63-3 (d, *J* = 7.5 Hz), 52.8, 50.7 (d, *J* = 8.5 Hz), 48.6 (d, *J* = 3.8 Hz), 47.9, 47.6, 39.1 (d, *J* = 3.6 Hz), 36.3, 35.9, 35.6 (d, *J* = 7.3 Hz), 30.5, 29.9, 28.9, 28.5 (d, *J* = 1.7 Hz), 24.3, 22.4.

³¹P NMR (81 MHz, CDCl₃): δ 18.9.

MS (FAB): *m/z* = 751 ([M+H]⁺, 100), 666 (14).

C₃₉H₄₄IrNP HRMS: Calcd.: 750.2634.

Found: 750.2841.

Synthesis of (*S*)-1,2-diphenylpropane (**21**)⁴

Ir-**3** (6.5 mg, 4 μ mol, 1 mol%) and *E*-1,2-diphenylpropene (77 mg, 0.4 mmol) in toluene (2 mL) were added into the autoclave. The autoclave was sealed and pressurized to 50 bar H₂ and the mixture was stirred at rt for 2 h. After the crude product was passed through a short column (pentane), (*S*)-**21** was obtained in quantitative yield, 95 % *ee* as a colourless oil.

HPLC (Chiralcel OJ, *n*-heptane/*i*-PrOH 99/1, 0.5 mL/min, 215 nm): *t*_r/min = 13.1 (*R*), 16.1 (*S*).

¹H NMR (300 MHz, CDCl₃): δ 7.22-6.96 (m, 10H), 2.98-2.82 (m, 2H), 2.74-2.64 (m, 1H), 1.16 (d, *J* = 6.9 Hz, 3H).

¹³C NMR (75 MHz, CDCl₃): δ 147.4, 141.2, 129.6, 128.7, 128.5, 127.4, 126.4, 126.2, 45.4, 42.3, 21.5.

Synthesis of (*S*)-2-(4-methoxyphenyl)-1-phenylpropane (**22**)

Ir-**3** (3.3 mg, 2 μ mol, 1 mol%) and *E*-1-phenyl-2-(4-methoxyphenyl)-1-propene (45 mg, 0.2 mmol) in CH₂Cl₂ (1 mL) were added into the autoclave. The autoclave was sealed and pressurized to 50 bar H₂ and the mixture was stirred at rt for 2 h. After the crude product was passed through a short column (pentane), (*S*)-**22** was obtained in quantitative yield, 95.2 % *ee* as a colourless oil.

HPLC (Chiralcel OJ, *n*-heptane/*i*-PrOH 95/5, 0.5 mL/min, 215 nm): *t*_r/min = 13.1 (*R*), 16.1 (*S*).

¹H NMR (300 MHz, CDCl₃): δ 7.18-6.96 (m, 7H), 6.76-6.72 (m, 2H), 3.70 (s, 3H), 2.92-2.78 (m, 2H), 2.70-2.62 (m, 1H), 1.13 (d, *J* = 6.9 Hz, 3H).

¹³C NMR (75 MHz, CDCl₃): δ 158.2, 141.3, 139.5, 129.6, 128.5, 128.3, 126.2, 114.1, 55.6, 45.7, 41.4, 21.8.

Synthesis of (*S*)-3-phenylbutanoate (23a**)**

Ir-3 (7.3 mg, 4.5 μ mol, 1 mol%) and ethyl *trans*- β -methylcinnamate (**23**) (85 mg, 0.45 mmol) in toluene (1 mL) were added into the autoclave. The autoclave was sealed and pressurized to 50 bar H₂, and the mixture was stirred at rt for 2 h. After the crude product passed through the short column (pentane), (*S*)-**23a** was obtained in 58 % *ee* as a pale yellow oil.

HPLC (Chiralcel OB, *n*-heptane/*i*-PrOH 99.5/0.5, 0.5 mL/min, 215 nm): 13.3 (*R*), 15.2 (*S*) min.

¹H NMR (300 MHz, CDCl₃): δ 7.24-7.08 (m, 5H), 3.99 (q, *J* = 7.2 Hz, 2H), 3.25-3.14 (m, 1H), 2.56-2.40 (m, 2H), 1.22 (d, *J* = 7.2 Hz, 3H), 1.10 (t, *J* = 7.2 Hz, 3H).

¹³C NMR (75 MHz, CDCl₃): δ 172.8, 146.1, 128.9, 127.1, 126.8, 60.6, 43.4, 36.9, 22.2, 14.6.

Synthesis of 3-phenyl-2-methylallyl alcohol (24a**)**

Ir-3 (7.0 mg, 4.3 μ mol, 1 mol%) and *trans*-2-methyl-3-phenyl-2-propen-1-ol (**24**) (64 mg, 0.43 mmol) in toluene (0.5 mL) were added to the autoclave. The autoclave was sealed and pressurized to 50 bar H₂ and the mixture was stirred at rt for 16 h. After the crude product passed through the short column (33% Et₂O in pentane), **24a** was obtained in 69 % *ee* as a colourless oil.

HPLC (Chiralcel OD-H, *n*-heptane/*i*-PrOH 95/5, 0.5 mL/min, 215 nm): 16.5/18.9 min.

¹H NMR (300 MHz, CDCl₃): δ 7.24-7.06 (m, 5H), 3.48-3.36 (m, 2H), 2.68 (dd, *J* = 13.5, 6.2 Hz, 1H), 2.35 (dd, *J* = 13.5, 8.1 Hz, 1H), 1.94-1.80 (m, 1H), 1.31 (br s, 1H), 0.85 (d, *J* = 6.6 Hz, 3H).

¹³C NMR (75 MHz, CDCl₃): δ 141.0, 129.5, 128.6, 126.3, 68.1, 40.1, 38.2, 16.8.

For the hydrogenation of 3-phenyl-2-methylallyl acetate (**25**) was hydrolyzed (MeOH/K₂CO₃) to 3-phenyl-2-methylallyl alcohol (**24a**, 80 % *ee*).

Rhodium-catalyzed asymmetric hydroboration of styrene

A mixture of [Rh(cod)₂]BF₄ (8.1 mg, 0.020 mmol, 1 mol %) and diphosphane **20** (12.0 mg, 0.024 mmol, 1.2 mol %) in dry THF (5 mL) was stirred for 10 min at RT in a 10 mL Schlenk tube under argon. Styrene (2 mmol, 0.23 mL) was added to the resulting orange solution. The homogeneous mixture was cooled to -20 °C and stirred at this temperature for 15 min before adding freshly distilled catecholborane (2.4 mmol, 0.26 mL). The reaction was stirred at 0 °C for 16 h and quenched by the addition of EtOH (2 mL). Aqueous NaOH (2 M, 2 mL) and 30 % H₂O₂ were added subsequently and the mixture was warmed up to rt, over a period of 2 h under vigorous stirring. The mixture was then extracted with Et₂O. The organic phase was washed with NaOH (1M), brined and dried (MgSO₄). The residue was purified by flash chromatography (20% Et₂O in pentane) to give **28** (72 %, 176 mg) as a colourless liquid.

HPLC (Chiralcel OD-H, *n*-heptane/*i*-PrOH 98/2, 0.4 mL/min, 215 nm): *t*_r/min = 35.5 (*R*), 43.7 (*S*).

¹H NMR (300 MHz, CDCl₃): δ 7.20-7.09 (m, 5H), 4.66 (q, *J* = 6.5 Hz, 1H), 2.80 (br s, 1H), 1.30 (d, *J* = 6.5 Hz, 3H) ppm.

¹³C NMR (75 MHz, CDCl₃): δ 146.4, 128.8, 127.7, 125.9, 70.6, 25.6 ppm.

References:

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Data for the x-ray crystallography analyses of 17a, 17e and 19**Data related to the aminophosphine oxide 17a**

Empirical formular	C ₂₇ H ₃₀ NOP
Formular weight	415.49
Temperature	295 (2)K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	P2 ₁ (No.4)
Unit cell dimensions	a = 9.438(2) Å b = 12.063(14) Å β = 103.508 (13)° c = 10.701(9) Å
Volume	1184.6 (3) Å ³
Z	2
Density (calculated)	1.165 Mg/m ³
Absorption coefficient	0.134 mm ⁻¹
F(000)	444
Crystal size	0.27 x 0.43 x 0.53 mm
Theta range for data collection	6.80 to 23.99 deg
Index ranges	-10≤h≤10, -13≤k≤13, -12≤l≤12
Reflections collected	4489
Independent reflections	3611 [R (int) = 0.0161]
Absorption correction	
Max. and min. transmission	0.9969 and 0.7934
Refinement method	full-matrix least-squares on F ²
Data/restraints/parameters	2373/274/1
Goodness-of-fit on F ²	1.101
Final R indices [I>2sigma(I)]	R1 = 0.0387, wR2 = 0.0447
R indices (all data)	R1 = 0.0970, wR2 = 0.1022
Absolute structure parameter	-0.04 (10)
Largest diff. Peak and hole	0.280 and -0.136 Å ³

Data related to the aminophosphine oxide 17e

Empirical formular	C ₃₂ H ₃₂ NOP
Formular weight	477.56
Temperature	295 (2)K
Wavelength	0.71073 Å
Crystal system	triclinic
Space group	P2 ₁ (No.2)
Unit cell dimensions	a = 10.856(2) Å α = 105.86 (2)° B = 11.450(2) Å β = 92.166 (15)° C = 12.027(2) Å γ = 113.13 (2)°
Volume	1304.6 (4) Å ³
Z	2
Density (calculated)	1.216 Mg/m ³
Absorption coefficient	0.130 mm ⁻¹
F(000)	508
Crystal size	0.20 x 0.33 x 0.53 mm
Theta range for data collection	2.95 to 23.98 deg
Index ranges	-12≤h≤12, -13≤k≤12, 0≤l≤13
Max. and min. transmission	0.9996 and 0.9760
Refinement method	full-matrix least-squares on F ²
Data/restraints/parameters	2465/318/0
Goodness-of-fit on F ²	1.045
Final R indices [I>2sigma(I)]	R1 = 0.0509, wR2 = 0.1054
R indices (all data)	R1 = 0.0792, wR2 = 0.1207
Absolute structure parameter	
Largest diff. Peak and hole	0.176 and -0.219 Å ³

Data related 1,2-diphosphine oxide (19)

FIGURE 1. X-ray structure of *trans*-19.

Empirical formular	$C_{30}H_{32}O_4P_2$
Formular weight	518.50
Temperature	295 (2)K
Wavelength	0.71073 Å
Crystal system	orthorhombic
Space group	$P2_12_12_1$ (No.19)
Unit cell dimensions	$a = 8.105 (4) \text{ Å}$ $b = 8.729 (3) \text{ Å}$ $c = 37.158 (12) \text{ Å}$
Volume	$2628.9 (17) \text{ Å}^3$
Z	4
Density (calculated)	1.310 Mg/m^3
Absorption coefficient	0.200 mm^{-1}
F(000)	1096
Crystal size	0.23 x 0.43 x 0.57 mm
Theta range for data collection	2.57 to 23.98 deg
Index ranges	$-9 \leq h \leq 9, -9 \leq k \leq 9, -42 \leq l \leq 42$
Reflections collected	4774
Independent reflections	4082 [R (int) = 0.0126]

Absorption correction	
Max. and min. transmission	0.9952 and 0.7830
Refinement method	full-matrix least-squares on F ²
Data/restraints/parameters	2541/328/0
Goodness-of-fit on F ²	1.020
Final R indices [I>2sigma(I)]	R1 = 0.0420, wR2 = 0.1061
R indices (all data)	R1 = 0.0487, wR2 = 0.1128
Absolute structure parameter	-0.01 (11)
Largest diff. Peak and hole	0.333 and -0.278 Å ³

Current Data Parameters
 USER: knoc
 NAME: 111_1b089
 EXPNO: 10
 PROCNO: 1

F2 - Acquisition Parameters
 Date_: 20020529
 Time: 7.32
 INSTRUM: spect
 PROBHD: 5 mm Multiv
 PULPROG: zg30
 TD: 32768
 SOLVENT: CDCl3
 NS: 8
 DS: 2
 SWH: 7575.758 Hz
 FIDRES: 0.23194 Hz
 AQ: 2.1627381 sec
 RG: 512
 DM: 66.000 usec
 DE: 82.50 usec
 TE: 300.0 K
 HL1: 1 dB
 D1: 1.00000000 sec
 P1: 12.20 usec
 SF01: 600.053503 MHz
 NUCLEUS: 1H

F2 - Processing parameters
 SI: 32768
 SF: 600.0500152 MHz
 WDW: EM
 SSB: 0
 LB: 0.30 Hz
 GB: 0
 PC: 4.00

1D NMR plot parameters
 CX: 23.00 cm
 F1P: 8.478 ppm
 F1: 5082.68 Hz
 F2P: -0.240 ppm
 F2: -144.24 Hz
 PPMCM: 0.37873 ppm/c
 HZCM: 227.25749 Hz/cm

Brucker AMX600
 AK: Knoche1

Arbeitskreis Knochel
User Katja Tappe
Bruker ARX 300

Current Data Parameters
USCA TAP
NAME 111_tub61
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20021018

Time 16.58
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgpg30
TD 299320
SOLVENT CDCl3
NS 65536
DS 2048
OS 6
SWH 19230.770 Hz
FIDRES 0.293438 Hz
AQ 1.7039860 sec
RG 16384
DN 26.000 usec
DE 32.50 usec
TE 300.0 K
D12 0.0002000 sec
DL6 23.00 dB
D1 1.50000000 sec
CPDPRG waitz16
P31 110.00 usec
D11 0.0300000 sec
DL5 20.00 dB
P1 6.20 usec
SFO1 75.4762456 MHz
NUCLEUS 13C

F2 - Processing Parameters
SI 65536
SF 75.4677190 MHz
WDW EM
SSB 0
LB 2.00 Hz
GB 0
PC 1.00

10 NMR plot parameters
CX 21.50 cm
F1P 240.379 ppm
F1 18140.87 Hz
F2P -14.442 ppm
F2 -1089.90 Hz
PPMCM 11.85214 ppm/cm
HZCM 894.45433 Hz/cm

STANDARD P31 OBSERVE
Pulse Sequence: s2pul1
Date: May 28 2002
Solvent: CDCl3
Temp: 23.0 C / 296.1 K
Mercury-200 "felix"
PULSE SEQUENCE
Pulse 39.5 degrees
Acq. time 1.260 sec
Width 30000.0 Hz
84 repetitions
OBSERVE P31, 80.9517971 MHz
DECOUPLE H1, 199.9773163 MHz
Power 36 db
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 262144
Total time 1 min, 54 sec

STANDARD P31 OBSERVE
(triphenylphosphate = -10ppm)
Pulse Sequence: s2pul1
Date: Nov 12 2002
Solvent: CDCl3
Temp: 23.0 C / 296.1 K
Mercury-200 "edward"
PULSE SEQUENCE
Pulse: 48.3 degrees
Acq. time: 1.280 sec
Width: 50000.0 Hz
64 repetitions
OBSERVE P31, 80.9549618 MHz
DECOUPLE H1, 199.9851442 MHz
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Time: processing 1.0 Hz
File size: 262144
Total time: 1 min, 54 sec