

SUPPORTING INFORMATION

P-Arylation of Secondary Phosphine Oxides Catalyzed by Nickel Supported Nanoparticles

Elżbieta Łastawiecka,^a Anna Flis,^a Marek Stankevič,^a Magdalena Greluk^b Grzegorz Słowik,^b and Wojciech Gac^b

^a. Department of Organic Chemistry, Faculty of Chemistry, Maria Curie-Skłodowska University,
Gliniana St. 33, Lublin 20-614, Poland.

^b. Department of Chemical Technology, Faculty of Chemistry, Maria Curie-Skłodowska University,
3 M. Curie-Skłodowska Sq., 20-031 Lublin, Poland.

Table of Contents

General Experimental	S3
Synthesis of Substrates (Secoundary Phosphine Oxides: 1a , 5a , 5b , 5c and 5d)	S3
General experimental procedure for the coupling reduction of aryl halides and secondary phosphine oxides.	S4
The analitical data for products 3a-3s' and 6a-6f	S4
¹ H NMR ¹³ C NMR and ³¹ P NMR spectra of 3a-3s' and 6a-6f	S14

General remarks

All reactions were performed under an argon atmosphere using Schlenk techniques or in a 10mL glass reaction tubes with crimp cap. Only dry solvents were used, bouth a Ni/Al₂O₃ and a Ni/CeO₂ are air and moisture stable, and were used without any protection, K₂CO₃ and other base was commercially available and used as received. Solvents for chromatography were commercially available and used as received without further purification.

The NMR spectra was recorded with Bruker Ascend (500 MHz), spectrometer in CDCl₃ as a solvent at room temperature unless otherwise noted. Chemical shifts (δ ppm) are given in ppm relative to residual CHCl₃. The following abbreviations are used in reporting NMR data: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad). Coupling constants (J) are in Hz. High-resolution mass spectrometry analyses were obtained on Shimadzu LCMS IT-TOF spectrometer. The FT-IR (ATR) spectra were recorded by means of a Nicolet 6700 (Thermo Scientific) spectrometer equipped with Smart Orbit accessory in the range of 400 - 4000 cm⁻¹. The resolution 2 cm⁻¹ and maximum source aperture were used. Interferograms of 128 scans were averaged for each spectrum. Thin layer chromatography (TLC) was performed with precoated silica gel plates and visualized by potassium permanganate (KMnO₄) stain. The reaction mixtures were purified by column chromatography over silica gel (60–240 mesh). Melting points were determined in a capillary tube.

Synthesis of Substrates (Secoundary Phosphine Oxides)

Diphenylphosphine oxide Ph₂P(O)H (**1a**) was synthesized according to literature procedure.¹ Scale: 50 mL of aqueous HCl (1 M) and 9.2 g (41.7 mmol) of chlorodiphenylphosphine, yield: 7.5 g (89 %) of colourless crystals of Ph₂P(O)H.

The diarylphosphine oxides (Ar₂P(O)H) **5a**, **5b**, **5c** and **5d** were synthesized according to reported procedure from (BuO)₂P(O)H with corensponding ArMgBr in THF.² Properties matched previously reported values.

(*rac*)-*t*-Butyl(phenyl)phosphane oxide (**5e**) was synthesized according to a modified literature procedure.³ A solution of *t*-BuMgBr (12.3 mL, 12.2 mmol) was added dropwise to PPhCl₂ (1.5 mL, 1.1 mmol) in dry THF (20 mL) cooled to -78 °C, under nitrogen. After the addition, the mixture was allowed to warm to room temperature and stirred for 18 h. The mixture was cooled to 0 °C, poured into ice and treated with HCl (0.5 M). After the addition, the mixture was allowed to warm to room temperature and stirred for 24 h, the progress of the reaction was followed by ³¹P NMR spectroscopy. After this time, the saturated NaHCO₃ solution was added and the aqueous layer was extracted with CHCl₃ (3 x 50 mL). The combined organic layers were dried (MgSO₄), filtered and concentrated under reduced pressure. The residue was purified by flash chromatography using chloroform/methanol 15:1 as eluent, (1.55 g, 76%). All data for (*rac*)-*t*-Butyl(phenyl)phosphane oxide (**5e**) are in according with previously reported.

¹ M. M. Kabat, L. M. Garofalo, A. R. Daniewski, S. D. Hutchings, W. Liu, M. Okabe, R. Radinov, Y. Zhou, *J. Org. Chem.* 2001, **66**, 6141.

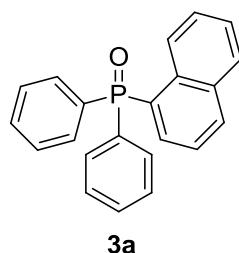
² A. L. Casalnuovo, T. V. RajanBabu, T. A. Ayers, T. H. Warren, *J. Am. Chem. Soc.*, 1994, **116**, 9869.

³ R.K. Haynes, T.-L.Au-Yeung, W.-K. Chan, W.-L. Lam, Z.-Y. Li, L.-L. Yeung, A.S.C. Chan, P. Li, M. Koen, C.R. Mitchell, S.C. Vonwiller, *Eur. J. Org. Chem.* 2000, 3205.

General experimental procedure for the coupling reduction of aryl halides and secondary phosphine oxides.

To a 10mL glass reaction tubes equipped with magnetic stirrer were placed a secondary phosphine oxide (0.5 mmol), aryl halides (0.55 mmol, 1.1 equiv), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv), Ni10/CeO₂ (2.9 mg, 1 mol % of Ni) and dry acetonitrile (0.5 mL; $c_i = 1$ mol/L). The mixture was stirred under a flow of argon for 1 min and then heated at 100 °C for 18 h before cooling to room temperature. The solvent was then removed under vacuum and the resulting residue was dissolved in CHCl₃. The crude obtained was filtered to remove base and the resulting filtrate was purified by silica-gel column chromatography using mixtures of chloroform and methanol (CHCl₃/MeOH 15/1).

The analytical data for products 3a-3s' and 6a-6f



1-Naphthyl(diphenyl)phosphine oxide (3a)

The compound **3a** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 86%. White crystal, **mp** = 184.9–185.2 °C (lit. 183.8–184.7 °C)⁴; **R_f** = 0.55 (CHCl₃/MeOH = 15: 1); **¹H NMR** (500 MHz, CDCl₃) δ: 7.29 – 7.36 (m, 1 H) 7.37 – 7.42 (m, 1 H) 7.42 – 7.53 (m, 6 H) 7.53 – 7.58 (m, 2 H) 7.67 – 7.74 (m, 4 H) 7.67 – 7.75 (m, 4 H) 7.90 (dd, $J_{P-H} = 8.20$, 1.0 Hz, 1 H) 8.03 (d, $J_{P-H} = 8.2$ Hz, 1 H) 8.61 (d, $J_{P-H} = 8.5$ Hz, 1 H); **¹³C NMR** (126 MHz, CDCl₃) δ: 124.2 (d, $J_{P-C} = 14.5$ Hz,) 126.5 (s) 127.4 (s) 127.6 (d, $J_{P-C} = 5.5$ Hz,) 128.9 (d, $J_{P-C} = 102.0$ Hz) 128.6 (d, $J_{P-C} = 11.8$ Hz) 128.8 (d, $J_{P-C} = 1.2$ Hz) 131.9 (d, $J_{P-C} = 2.7$ Hz) 132.08 (d, $J_{P-C} = 10$ Hz) 132.8 (d, $J_{P-C} = 104.5$ Hz) 133.3 (d, $J_{P-C} = 2.7$ Hz) 133.7 (d, $J_{P-C} = 8.0$ Hz) 133.8 (d, $J_{P-C} = 12.0$ Hz) 133.9 (d, $J_{P-C} = 9.1$ Hz); **³¹P NMR** (202 MHz, CDCl₃) δ: 32.3; **FT-IR (ATR)** $\nu(cm^{-1})$: 480, 501, 515, 546, 663, 694, 719, 737, 750, 777, 802, 829, 984, 1072, 1117, 1144, 1192, 1433, 1504, 3055; **HRMS (ESI)**: m/z = 329.1080 [C₂₂H₁₇OP+H]⁺, m/z (teor.) = 329.1090, diff. = -3.04 ppm.

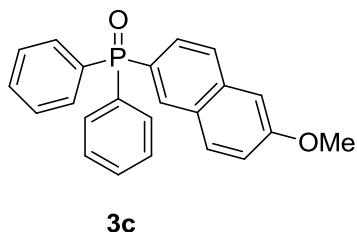
2-Naphthyl(diphenyl)phosphine oxide (3b)

The compound **3b** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 2-Bromonaphthalene (**2b**, 0.077 mL, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 73%. White crystal, **mp** = 133.7–134.2 °C (lit. 184–185 °C)⁵; **R_f** = 0.48 (CHCl₃/MeOH = 15: 1); **¹H NMR** (500 MHz, CDCl₃) δ: 7.46 – 7.51 (m, 4 H) 7.53 – 7.62 (m, 4 H) 7.66 (ddd, $J=9.9$, 8.4, 1.6 Hz, 1 H) 7.71 – 7.77 (m, 4 H) 7.89 (d, $J=8.2$ Hz, 2 H) 7.92 (dd, $J=8.5$, 2.8 Hz, 1 H) 8.31 (dd, $J=14.0$, 0.8 Hz, 1 H); **¹³C NMR** (126 MHz, CDCl₃) δ: 126.9 (d, $J_{P-C} = 10.0$ Hz) 127.0 (s) 127.8 (s) 128.3 (s) 128.4 (s) 128.6 (d, $J_{P-C} =$

⁴ M. Stankevič, J. Pisklak, K. Włodarczyk, *Tetrahedron*, 2016, **72**, 810.

⁵ A. Jutand, A. Mosleh, *J. Org. Chem.*, 1997, **62**, 261.

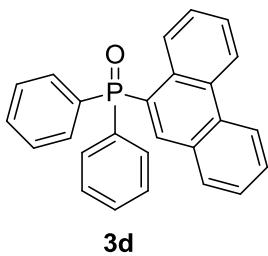
11.8 Hz) 129.0 (s) 129.6 (d, $J_{P-C} = 104.5$ Hz) 132.0 (d, $J_{P-C} = 2.7$ Hz) 132.2 (d, $J_{P-C} = 10.0$ Hz) 132.4 (d, $J_{P-C} = 13.6$ Hz) 133.0 (s) 134.0 (d, $J_{P-C} = 9.1$ Hz) 134.7 (d, $J_{P-C} = 1.8$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ: 29.1; FT-IR (ATR) ν (cm⁻¹): 507, 530, 544, 604, 642, 700, 723, 752, 820, 831, 1078, 1086, 1103, 1192, 1439; HRMS (ESI): m/z = 329.1082 [C₂₂H₁₇OP+H]⁺, m/z (teor.) = 329.1090, diff. = -2.43 ppm.



(6-Methoxy-2-naphthyl)(diphenyl)phosphine oxide (3c)

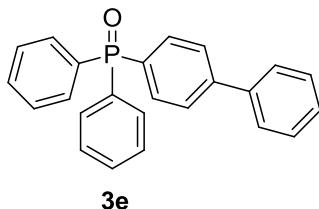
The compound **3c** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 2-bromo-6-methoxynaphthalene (**2c**, 130.4 mg, 0.55 mmol, 1.1 equiv to **1a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100

°C for 72 h. Yield: 72%. White crystal, **mp** = 125.7-126.8 °C; **R_f** = 0.45 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ: 3.91 (t, $J=2.9$ Hz, 3 H) 7.15 (br. s., 1 H) 7.19 (dd, $J=9.1, 1.3$ Hz, 1 H) 7.43 - 7.49 (m, 4 H) 7.50 - 7.56 (m, 2 H) 7.61 (t, $J=9.1$ Hz, 1 H) 7.68 - 7.81 (m, 6 H) 8.19 (d, $J=13.9$ Hz, 1 H); ^{13}C NMR (126 MHz, CDCl₃) δ: 55.4 (s) 105.7 (s) 119.9 (s) 126.8 (d, $J_{P-C} = 106.6$ Hz) 127.1 (d, $J_{P-C} = 11.8$ Hz) 127.6 (d, $J_{P-C} = 10.9$ Hz) 127.9 (d, $J_{P-C} = 13.6$ Hz) 128.5 (d, $J_{P-C} = 11.8$ Hz) 130.5 (s) 131.9 (d, $J_{P-C} = 2.7$ Hz) 132.1 (d, $J_{P-C} = 10.0$ Hz) 132.8 (d, $J_{P-C} = 104.5$ Hz) 133.7 (d, $J_{P-C} = 9.1$ Hz) 136.3 (d, $J_{P-C} = 1.8$ Hz) 159.5 (s); ^{31}P NMR (202 MHz, CDCl₃) δ: 29.3; FT-IR (ATR) ν (cm⁻¹): 816, 852, 891, 1030, 1065, 1119, 1209, 1262, 1331, 1387, 1406, 1496, 1587, 1624, 2966, 3008; HRMS (ESI): m/z = 359.1198 [C₂₃H₁₉O₂P+H]⁺, m/z (teor.) = 359.1195, diff. = 0.84 ppm.



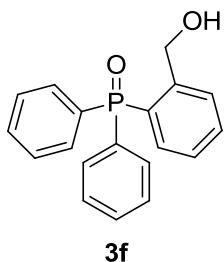
9-Phenanthryl(diphenyl)phosphine oxide (3d)

The compound **3d** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 9-bromophenanthrene (**2d**, 141.4 mg, 0.55 mmol, 1.1 equiv to **1a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 34%. White crystal, **mp** = 199.5-200.5 °C; **R_f** = 0.63 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ: 7.48 - 7.54 (m, 5 H) 7.57 - 7.62 (m, 3 H) 7.64 - 7.70 (m, 2 H) 7.72 - 7.81 (m, 6 H) 8.66 (d, $J=8.2$ Hz, 1 H) 8.73 (dd, $J=13.1, 8.4$ Hz, 2 H); ^{13}C NMR (126 MHz, CDCl₃) δ: 122.6 (s) 123.0 (s) 127.0 (s) 127.2 (d, $J_{P-C} = 9.1$ Hz) 127.8 (d, $J_{P-C} = 102.2$ Hz) 128.7 (d, $J_{P-C} = 12.7$ Hz) 128.7 (d, $J_{P-C} = 5.5$ Hz) 129.1 (s) 129.7 (d, $J_{P-C} = 14.5$ Hz) 130.1 (s) 130.7 (d, $J_{P-C} = 8.2$ Hz) 130.9 (d, $J_{P-C} = 8.2$ Hz) 132.0 (d, $J_{P-C} = 2.7$ Hz) 132.2 (d, $J_{P-C} = 10.0$ Hz) 132.6 (d, $J_{P-C} = 104.6$ Hz) 136.9 (d, $J_{P-C} = 11.8$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ: 32.7; FT-IR (ATR) ν (cm⁻¹): 428, 436, 501, 513, 530, 569, 607, 694, 719, 758, 958, 1103, 1157, 1184, 1435; HRMS (ESI): m/z = 379.1239 [C₂₆H₁₉OP+H]⁺, m/z (teor.) = 379.1246, diff. = -1.85 ppm.



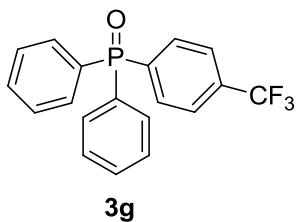
Biphenyl-4-yl(diphenyl)phosphine oxide (3e)

The compound **3e** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 4-bromobiphenyl (**2e**, 128.2 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 48%. White crystal, **mp** = 121.3–122.1 °C. R_f = 0.5 (CHCl₃/MeOH = 15: 1); ¹H NMR (500 MHz, CDCl₃) δ: 7.38 – 7.43 (m, 1 H) 7.45 – 7.52 (m, 6 H) 7.55 – 7.60 (m, 2 H) 7.60 – 7.63 (m, 2 H) 7.68 – 7.79 (m, 8 H); ¹³C NMR (126 MHz, CDCl₃) δ: 127.2 (d, $J_{P-C} = 12.7$ Hz) 127.3 (s) 128.2 (s) 128.6 (d, $J_{P-C} = 12.7$ Hz) 129.0 (s) 131.0 (d, $J_{P-C} = 105.0$ Hz) 132.0 (d, $J_{P-C} = 2.7$ Hz) 132.1 (d, $J_{P-C} = 10.0$ Hz) 132.5 (d, $J_{P-C} = 104.5$ Hz) 132.6 (d, $J_{P-C} = 10.5$ Hz) 139.9 (s) 144.8 (d, $J_{P-C} = 2.7$ Hz); ³¹P NMR (202 MHz, CDCl₃) δ: 29.2; FT-IR (ATR) $\nu(cm^{-1})$: 528, 548, 563, 658, 694, 729, 752, 764, 1119, 1192, 1390, 1439, 1483, 1599; HRMS (ESI): m/z = 355.1239 [C₂₄H₁₉OP+H]⁺, m/z (teor.) = 355.1246, diff. = -1.97 ppm.



(2-(Hydroxymethyl)phenyl)(diphenyl)phosphine oxide (3f)

The compound **3f** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 2-bromobenzyl alcohol (**2f**, 102.3 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 75%. Colorless crystal, **mp** = 152.9–154.1 °C (lit. 152–153 °C)⁶; R_f = 0.45 (CHCl₃/MeOH = 15: 1); ¹H NMR (500 MHz, CDCl₃) δ: 4.61 (s, 2 H) 7.06 (ddd, $J=14.1, 7.6, 0.9$ Hz, 1 H) 7.29 (tdd, $J=7.6, 7.6, 2.7, 1.4$ Hz, 2 H) 7.49 – 7.54 (m, 5 H) 7.54 – 7.58 (m, 1 H) 7.59 – 7.67 (m, 6 H); ¹³C NMR (126 MHz, CDCl₃) 64.7 (d, $J_{P-C} = 5.5$ Hz) 127.2 (d, $J_{P-C} = 12.7$ Hz) 128.7 (d, $J_{P-C} = 11.8$ Hz) 131.2 (d, $J_{P-C} = 101.7$ Hz) 131.7 (d, $J_{P-C} = 104.5$ Hz) 131.6 (d, $J_{P-C} = 10.0$ Hz) 132.1 (d, $J_{P-C} = 10.0$ Hz) 132.3 (d, $J_{P-C} = 2.7$ Hz) 132.8 (d, $J_{P-C} = 2.7$ Hz) 133.7 (d, $J_{P-C} = 12.7$ Hz) 146.7 (d, $J_{P-C} = 7.3$ Hz); ³¹P NMR (202 MHz, CDCl₃) δ: 34.9; FT-IR (ATR) $\nu(cm^{-1})$: 538, 712, 750, 1028, 1053, 1080, 1115, 1174, 1437, 3338; HRMS (ESI): m/z = 331.0866 [C₁₉H₁₇O₂P+Na]⁺, m/z (teor.) = 331.0858, diff. = 2.42 ppm.



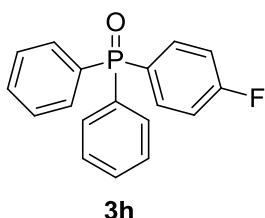
Diphenyl[4-(trifluoromethyl)phenyl]phosphine oxide (3g)

The compound **3g** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 1-bromo-4-(trifluoromethyl)benzene (**2g**, 123.8 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 47%. Colorless oil (lit. 88–89 °C)⁷; R_f = 0.52 (CHCl₃/MeOH = 15: 1); ¹H NMR (500 MHz, CDCl₃) δ: 7.47 – 7.54 (m, 4 H) 7.56 – 7.62 (m, 2 H) 7.65 – 7.71 (m, 4 H) 7.74 (dd, $J=8.0, 1.7$ Hz, 2 H) 7.84 (dd, $J=11.4, 8.2$ Hz, 2 H); ¹³C NMR (126 MHz, CDCl₃) δ: 123.6 (d, $J_{C-F} = 272.5$ Hz,) 125.2 – 125.5 (m) 128.7 (d, $J_{P-C} = 12.7$ Hz) 131.24 (s) 132.04 (d, $J_{P-C} = 10.0$ Hz) 132.4 (d, $J_{P-C} = 2.7$ Hz,) 132.6 (d, $J_{P-C} = 10.0$ Hz) 133.7 (dd,

⁶ M.B. Tollefson, J. J. Li, P. Beak, *J. Am. Chem. Soc.*, 1996, **118**, 9052.

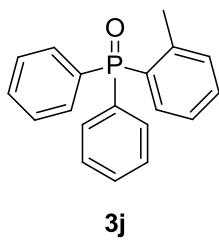
⁷ O. Berger, C. Petit, E. L. Deal, J.-L. Montchamp, *Adv. Synth. Catal.*, 2013, **355**, 1361.

$J_{F-C,P-C}=32.8, 2.8$ Hz) 137.2 (d, $J_{P-C}=100.5$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ : 28.0; FT-IR (ATR) ν (cm⁻¹): 538, 559, 692, 714, 833, 1016, 1063, 1117, 1165, 1190, 1327, 1437; HRMS (ESI): m/z = 347.0807 [C₁₉H₁₄F₃OP+H]⁺, m/z (teor.) = 347.0807, diff. = 0.00 ppm.



4-Fluorophenyl(diphenyl)phosphine oxide (3h)

The compound **3a** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 1-bromo-4-fluorobenzene (**2h**, 0.064 mL, 0.55 mmol, 1.1 equiv to **1a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (14.5 mg, 5 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 64%. White crystal, **mp** = 136.2-137.5 °C (lit. 133.6-135.5 °C)⁸; **R_f** = 0.57 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ : 7.14 - 7.21 (m, 2 H) 7.46 - 7.52 (m, 4 H) 7.55 - 7.60 (m, 2 H) 7.65 - 7.72 (m, 6 H); ^{13}C NMR (126 MHz, CDCl₃) δ : 115.9 (dd, $J=21.7, 13.2$ Hz) 128.6 (dd, $J=106.8, 3.5$ Hz) 128.6 (d, $J=11.8$ Hz) 132.4 (d, $J=105.0$ Hz) 132.0 (d, $J=10.0$ Hz) 132.1 (d, $J=2.9$ Hz) 134.6 (dd, $J=11.4, 8.6$ Hz) 165.1 (dd, $J=253.5, 3.3$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ : 28.4; FT-IR (ATR) ν (cm⁻¹): 521, 544, 696, 727, 752, 829, 843, 1093, 1120, 1182, 1228, 1439, 1496, 1593; HRMS (ESI): m/z = 297.0825 [C₁₈H₁₄FOP+H]⁺, m/z (teor.) = 297.0839, diff. = -4.71 ppm.

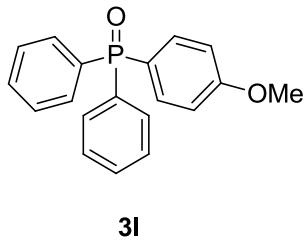


Diphenyl(o-tolyl)phosphine oxide (3j)

The compound **3j** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 2-iodotoluene (**4j**, 0.07 mL, 0.55 mmol, 1.1 equiv to **1a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 36%. White crystal, **mp** = 121.8-122.3 °C (lit. 121.5-122.9 °C)⁹; **R_f** = 0.68 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ : 2.46 (s, 3 H) 7.04 (ddd, $J=14.0, 7.7, 1.3$ Hz, 1 H) 7.11 - 7.17 (m, 1 H) 7.27 - 7.31 (m, 1 H) 7.43 (tt, $J=7.5, 1.5$ Hz, 1 H) 7.45 - 7.51 (m, 4 H) 7.53 - 7.58 (m, 2 H) 7.64 - 7.70 (m, 4 H); ^{13}C NMR (126 MHz, CDCl₃) δ : 21.7 (d, $J_{P-C}=5.4$ Hz) 125.2 (d, $J_{P-C}=12.7$ Hz) 128.6 (d, $J_{P-C}=12.7$ Hz) 130.7 (d, $J_{P-C}=103.7$ Hz) 131.8 (d, $J_{P-C}=2.7$ Hz) 131.9 (d, $J_{P-C}=10.0$ Hz) 132.1 (d, $J_{P-C}=1.8$ Hz) 132.7 (d, $J_{P-C}=103.6$ Hz) 133.5 (d, $J_{P-C}=12.7$ Hz) 143.3 (d, $J_{P-C}=8.2$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ : 31.9; FT-IR (ATR) ν (cm⁻¹): 505, 548, 706, 725, 764, 1120, 1136, 1180, 1435, 3500; HRMS (ESI): m/z = 293.1099 [C₁₉H₁₇OP+H]⁺, m/z (teor.) = 293.1090, diff. = 3.07 ppm.

⁸ T. Fu, H. Qiao, Z. Peng, G. Hu, X. Wu, Y. Gao, Y. Zhao, *Org. Biomol. Chem.*, 2014, **12**, 2895.

⁹ M. Stankevič, A. Włodarczyk, *Tetrahedron*, 2013, **69**, 73.



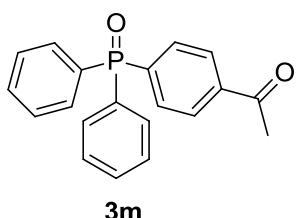
(4-Methoxyphenyl)diphenylphosphine oxide (**3l**)

The compound **3l** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 4-iodoanisole (**4l**, 128.7 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 42%. White crystal, $mp = 107.6\text{-}108.7$ °C $R_f = 0.42$ ($CHCl_3/MeOH = 15: 1$); 1H

NMR (500 MHz, $CDCl_3$) δ : 3.84 (s, 3 H) 6.95 - 6.98 (m, 2 H) 7.43 - 7.48 (m, 4 H) 7.51 - 7.55 (m, 2 H) 7.56 - 7.61 (m, 2 H) 7.63 - 7.69 (m, 4 H); ^{13}C **NMR** (126 MHz, $CDCl_3$) δ : 55.3 (s) 114.1 (d, $J_{P-C}=12.7$ Hz) 123.4 (d, $J_{P-C}=110.8$ Hz) 128.4 (d, $J_{P-C}=11.8$ Hz) 131.8 (d, $J_{P-C}=2.7$ Hz) 132.0 (d, $J_{P-C}=10.0$ Hz) 132.9 (d, $J_{P-C}=104.5$ Hz) 134.0 (d, $J_{P-C}=10.9$ Hz) 162.5 (d, $J_{P-C}=2.7$ Hz); ^{31}P **NMR** (202 MHz, $CDCl_3$) δ : 29.2; FT-IR (ATR) $\nu(cm^{-1})$: 536, 702, 727, 1020, 1120, 1176, 1192, 1256, 1437, 1504, 1599; **HRMS (ESI)**: $m/z = 309.1047$ [$C_{19}H_{17}O_2P+H]^+$, m/z (teor.) = 309.1039, diff. = 2.59 ppm.

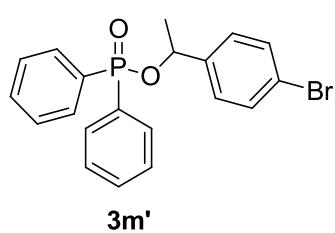
The coupling reduction of 4-bromoacetophenone (**2m**) and diphenylphosphine oxide (**1a**)

The compounds **3m** and **3m'** were prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 4-bromoacetophenone (**2m**, 109.5 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). The products **3m** and **3m'** separated by column chromatography in 23% and 49% yield, respectively.



1-(4-(Diphenylphosphoryl)phenyl)ethan-1-one (**3m**)

Yield: 23%. $mp = 165.8\text{-}169.2$ °C (lit. 170-172 °C)¹⁰; $R_f = 0.38$ ($CHCl_3/MeOH = 15: 1$); 1H **NMR** (500 MHz, $CDCl_3$) δ : 2.65 (s, 3 H) 7.47 - 7.53 (m, 4 H) 7.57 - 7.62 (m, 2 H) 7.65 - 7.71 (m, 4 H) 7.81 (dd, $J=11.3, 8.5$ Hz, 2 H) 8.04 (dd, $J=8.5, 2.5$ Hz, 2 H); ^{13}C **NMR** (126 MHz, $CDCl_3$) δ : 26.8 (s) 128.1 (d, $J_{P-C}=11.8$ Hz) 128.7 (d, $J_{P-C}=12.7$ Hz) 131.8 (d, $J_{P-C}=104.5$ Hz) 132.0 (d, $J_{P-C}=10.0$ Hz) 132.3 (d, $J_{P-C}=2.7$ Hz) 132.4 (d, $J_{P-C}=10.0$ Hz) 137.7 (d, $J_{P-C}=100.8$ Hz) 139.5 (d, $J_{P-C}=2.7$ Hz) 197.5 (s); ^{31}P **NMR** (202 MHz, $CDCl_3$) δ : 28.4; FT-IR (ATR) $\nu(cm^{-1})$: 538, 557, 623, 700, 731, 1119, 1196, 1263, 1392, 1437, 1687; **HRMS (ESI)**: $m/z = 321.1032$ [$C_{20}H_{17}O_2P+H]^+$, m/z (teor.) = 321.1039, diff. = -2.18 ppm.

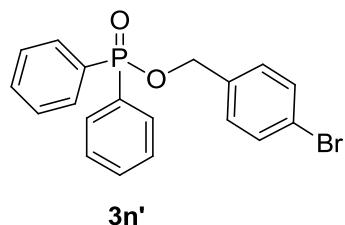


1-(4-Bromophenyl)ethyl diphenylphosphinate (**3m'**)

Yield: 49%. Colorless oil. $R_f = 0.62$ ($CHCl_3/MeOH = 15: 1$); 1H **NMR** (500 MHz, $CDCl_3$) δ : 1.65 (d, $J=6.6$ Hz, 3 H) 5.49 (dd, $J=9.1, 6.3$ Hz, 1 H) 7.17 - 7.21 (m, 2 H) 7.33 - 7.38 (m, 2 H) 7.41 - 7.45 (m, 2 H) 7.45 - 7.50 (m, 3 H) 7.52 - 7.57 (m, 1 H) 7.64 - 7.69 (m, 2 H) 7.82 - 7.88 (m, 2 H); ^{13}C **NMR** (126 MHz, $CDCl_3$) δ : 24.9 (d, $J_{P-C}=3.6$ Hz) 73.7 (d, $J_{P-C}=5.4$ Hz) 121.8 (s) 127.7 (s) 128.4 (d, $J_{P-C}=13.6$ Hz) 128.5 (d, $J_{P-C}=12.7$ Hz) 130.3 (d, $J_{P-C}=126.3$ Hz) 131.5 (d, $J_{P-C}=10.0$ Hz) 131.6 (s) 131.8 (d, $J_{P-C}=10.0$ Hz) 131.9 (d, $J_{P-C}=3.6$ Hz) 132.1 (d, $J_{P-C}=2.7$ Hz) 132.2 (d, $J_{P-C}=2.7$ Hz) 132.7

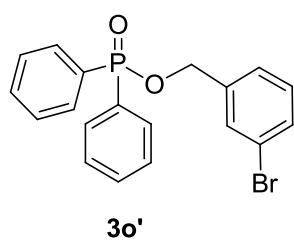
¹⁰ O. M. Bukachuk, M. G. Nikula, V. N. Belenkov, M. I. Shevchuk, *J. Gen. Chem. USSR (Engl. Transl.)*, 1986, **56**, 343, 298.

(s) 141.1 (d, $J_{P-C}=4.5$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ: 31.4; FT-IR (ATR) $\nu(cm^{-1})$: 540, 628, 729, 958, 1009, 1070, 1113, 1130, 1228, 1439, 1489; HRMS (ESI): m/z = 423.0123 [C₂₀H₁₈BrO₂P+Na]⁺, m/z (teor.) = 423.0120, diff. = 0.71 ppm.



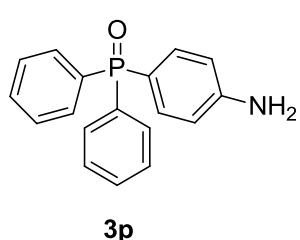
4-Bromobenzyl diphenylphosphinate (3n')

The compound **3n'** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 4-bromobenzaldehyde (**2n**, 101.8 mg, 0.55 mmol, 1.1 equiv to **1a**) and K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 71%. Colorless oil.¹¹ R_f = 0.65 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ: 5.03 (d, $J=7.3$ Hz, 2 H) 7.25 (d, $J=8.2$ Hz, 2 H) 7.44 - 7.51 (m, 6 H) 7.52 - 7.58 (m, 2 H) 7.83 (dd, $J=12.3, 7.9$ Hz, 4 H); ^{13}C NMR (126 MHz, CDCl₃) δ: 65.6 (d, $J_{P-C}=5.5$ Hz) 122.4 (s) 128.6 (d, $J_{P-C}=13.6$ Hz) 129.6 (s) 131.0 (d, $J_{P-C}=103.2$ Hz) 131.6 (s) 131.7 (s) 132.4 (d, $J_{P-C}=2.7$ Hz) 135.4 (d, $J_{P-C}=7.3$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ: 32.8; FT-IR (ATR) $\nu(cm^{-1})$: 532, 555, 696, 731, 752, 798, 1009, 1070, 1113, 1130, 1227, 1439, 1489; HRMS (ESI): m/z = 387.0155 [C₂₀H₁₈BrO₂P+H]⁺, m/z (teor.) = 387.0144, diff. = 2.84 ppm.



3-Bromobenzyl diphenylphosphinate (3o')

The compound **3o'** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 3-bromobenzaldehyde (**2o**, 0.064 mL, 0.55 mmol, 1.1 equiv to **1a**) and K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 87%. White crystal, mp = 63.3-64.8 °C; R_f = 0.65 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ: 5.04 (d, $J=6.9$ Hz, 2 H) 7.22 (t, $J=7.8$ Hz, 1 H) 7.29 - 7.32 (m, 1 H) 7.43 - 7.51 (m, 6 H) 7.53 - 7.58 (m, 2 H) 7.81 - 7.88 (m, 4 H); ^{13}C NMR (126 MHz, CDCl₃) δ: 65.4 (d, $J_{P-C}=5.5$ Hz) 122.5 (s) 126.4 (s) 128.6 (d, $J_{P-C}=13.6$ Hz) 130.1 (s) 131.1 (d, $J_{P-C}=136.6$ Hz) 130.8 (s) 131.4 (s) 131.7 (d, $J_{P-C}=10.0$ Hz) 132.4 (d, $J_{P-C}=2.7$ Hz) 138.6 (d, $J_{P-C}=7.3$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ: 32.8; FT-IR (ATR) $\nu(cm^{-1})$: 526, 553, 669, 692, 731, 756, 785, 854, 1024, 1072, 1130, 1219, 1371, 1439, 1591; HRMS (ESI): m/z = 387.0133 [C₁₉H₁₆BrO₂P+H]⁺, m/z (teor.) = 387.0144, diff. = -2.84 ppm.



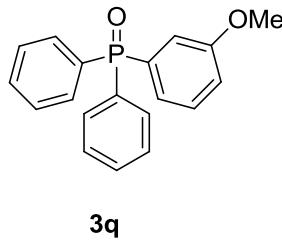
(4-Aminophenyl)diphenylphosphine oxide (3p)

The compound **3p** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 4-iodoaniline (**4p**, 120.5 mg, 0.55 mmol, 1.1 equiv to **1a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **1a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 32%. White crystal, mp = 245.1-246.6 °C (lit. 245.5-246.5 °C)¹²; R_f = 0.25 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ: 3.48 - 4.38 (br.s, 2 H) 6.68 (d, $J=6.9$ Hz, 2 H) 7.36 - 7.49 (m, 6 H) 7.49 - 7.56 (m, 2 H) 7.67 (dd, $J=12.0, 7.6$ Hz, 4 H); ^{13}C NMR (126 MHz, CDCl₃) δ: 114.3 (d, $J_{P-C}=12.7$ Hz) 118.7 - 120.6 (m) 128.4 (d, $J_{P-C}=11.8$ Hz) 131.6 (d, $J_{P-C}=1.8$ Hz)

¹¹ B. Xiong, G. Wang, C. Zhou, Y. Liu, P. Zhang, K. Tang, *J. Org. Chem.*, 2018, **83**, 993.

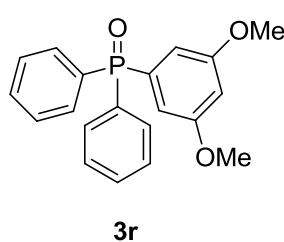
¹² C.M. Whitaker, K. L. Kott, R. J. McMahon, *J. Org. Chem.*, 1995, **60**, 3499.

132.1 (d, $J_{P-C}=10.0$ Hz) 133.4 (d, $J_{P-C}=104.5$ Hz) 133.8 (d, $J_{P-C}=10.9$ Hz) 149.9 (s); **^{31}P NMR** (202 MHz, $CDCl_3$) δ : 29.6; **FT-IR (ATR)** $\nu(cm^{-1})$: 505, 523, 542, 696, 710, 729, 1119, 1161, 1437, 1508, 1599, 1637, 3217, 3338, 3396; **HRMS (ESI)**: m/z = 294.1039 [$C_{18}H_{16}NOP+H$]⁺, m/z (teor.) = 294.1042, diff. = -1.02 ppm.



(3-Methoxyphenyl)diphenylphosphine oxide (3q)

The compound **3p** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 3-iodoanisole (**4q**, 128.7 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 37%. White crystal, **mp** = 105.0-105.6 °C (lit. 106.1 - 107.7 °C)¹³; **R_f** = 0.41 ($CHCl_3/MeOH = 15: 1$); **1H NMR** (500 MHz, $CDCl_3$) δ : 3.81 (s, 3 H) 7.09 (dd, $J=8.2, 1.6$ Hz, 1 H) 7.16 (dd, $J=11.7, 7.6$ Hz, 1 H) 7.31 (d, $J=13.2$ Hz, 1 H) 7.38 (td, $J=7.9, 3.8$ Hz, 1 H) 7.53 - 7.60 (m, 2 H) 7.69 (dd, $J=12.0, 7.9$ Hz, 4 H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ : 55.4 (s) 116.8 (d, $J_{P-C}=10.9$ Hz) 118.2 (d, $J_{P-C}=2.7$ Hz) 124.4 (d, $J_{P-C}=10.9$ Hz) 128.5 (d, $J_{P-C}=11.8$ Hz) 129.7 (d, $J_{P-C}=14.5$ Hz) 132.0 (d, $J_{P-C}=2.7$ Hz) 132.1 (d, $J_{P-C}=10.0$ Hz) 132.8 (s) 133.2 - 134.5 (m) 159.6 (d, $J_{P-C}=14.5$ Hz); **^{31}P NMR** (202 MHz, $CDCl_3$) δ : 29.5; **FT-IR (ATR)** $\nu(cm^{-1})$: 496, 519, 538, 592, 694, 723, 750, 1039, 1119, 1190, 1238, 1286, 1437, 1471, 1483, 1576, 1593, 3051; **HRMS (ESI)**: m/z = 309.1041 [$C_{19}H_{17}O_2P+H$]⁺, m/z (teor.) = 309.1039, diff. = 0.65 ppm.



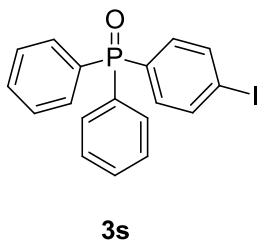
3,5-Dimethoxyphenyl(diphenyl)phosphine oxide (3r)

The compound **3r** was prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 1-iodo-3,5-dimethoxybenzene (**4r**, 145.2 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). Yield: 27%. White crystal, **mp** = 80.2-80.9 °C; **R_f** = 0.48 ($CHCl_3/MeOH = 15: 1$); **1H NMR** (500 MHz, $CDCl_3$) δ : 3.78 (s, 6 H) 6.61 (t, $J=2.0$ Hz, 1 H) 6.80 (dd, $J=13.1, 2.4$ Hz, 2 H) 7.45 - 7.50 (m, 4 H) 7.54 (m, 1 H) 7.65 - 7.71 (m, 4 H); **^{13}C NMR** (126 MHz, $CDCl_3$) δ : 55.6 (s) 104.1 (d, $J_{P-C}=2.7$ Hz) 109.8 (d, $J_{P-C}=10.9$ Hz) 128.5 (d, $J_{P-C}=12.7$ Hz) 132.3 (d, $J_{P-C}=104.5$ Hz) 132.0 (d, $J_{P-C}=2.7$ Hz) 132.1 (d, $J_{P-C}=9.1$ Hz) 134.5 (d, $J_{P-C}=103.5$ Hz) 160.8 (d, $J_{P-C}=18.2$ Hz); **^{31}P NMR** (202 MHz, $CDCl_3$) δ : 29.9; **FT-IR (ATR)** $\nu(cm^{-1})$: 519, 550, 698, 725, 837, 1041, 1063, 1119, 1167, 1207, 1288, 1340, 1417, 1439, 1456, 1589, 3410, 3452; **HRMS (ESI)**: m/z = 339.1137 [$C_{20}H_{19}O_3P+H$]⁺, m/z (teor.) = 339.1145, diff. = -2.36 ppm.

The coupling reduction of 1,4-diiodobenzene (**4s**) and diphenylphosphine oxide (**1a**)

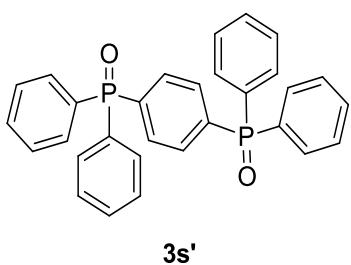
The compounds **3s** and **3s'** were prepared according to the general procedure from diphenylphosphine oxide (**1a**) (101 mg, 0.5 mmol), 1,4-diiodobenzene (**4s**, 181.4 mg, 0.55 mmol, 1.1 equiv to **1a**), K_2CO_3 (207 mg, 1.5 mmol, 3 equiv to **1a**) and $Ni10/CeO_2$ (2.9 mg, 1 mol % of Ni to **1a**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L). The procedure afforded the mixture of **3s**, **3s'** and triphenylphosphine oxide in 13%, 17% and 8% yields, respectively.

¹³ X. Zhang, H. Liu, X. Hu, G. Tang, J. Zhu, Y. Zhao, *Org. Lett.*, 2011, **13**, 3478.



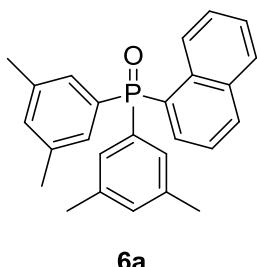
(4-Iodophenyl)diphenylphosphine oxide (3s)¹⁴

Yield: 13%; White solid. **mp** = 157.6–158.0 °C; **R_f** = 0.58 (CHCl₃/MeOH = 15: 1); **¹H NMR** (500 MHz, CDCl₃) δ: 7.41 (dd, *J*=11.3, 8.2 Hz, 2 H) 7.47 – 7.53 (m, 4 H) 7.56 – 7.62 (m, 2 H) 7.67 (dd, *J*=11.8, 7.7 Hz, 4 H) 7.85 (d, *J*=6.6 Hz, 2 H); **¹³C NMR** (126 MHz, CDCl₃) δ: 99.7 (d, *J_{P-C}*=2.7 Hz) 128.7 (d, *J_{P-C}*=12.7 Hz) 132.0 (d, *J_{P-C}*=105.4 Hz) 132.3 (d, *J_{P-C}*=103.4 Hz) 132.1 (d, *J_{P-C}*=10.0 Hz) 132.2 (d, *J_{P-C}*=1.8 Hz) 133.5 (d, *J_{P-C}*=10.0 Hz) 137.8 (d, *J_{P-C}*=11.8 Hz); **³¹P NMR** (202 MHz, CDCl₃) δ: 28.8; **FT-IR (ATR)** *v*(cm⁻¹): 540, 696, 731, 808, 1005, 1055, 1068, 1117, 1192, 1379, 1437, 1570, 2920; **HRMS (ESI)**: m/z = 404.9911 [C₁₈H₁₄IOP+H]⁺, m/z (teor.) = 404.9900, diff. = 2.72 ppm.



1,4-Phenylenebis(diphenylphosphine) dioxide (3s')

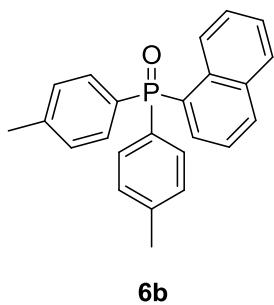
Yield: 17%; White solid. **mp** = 296.6–297.6 °C; (lit. 300)¹³ **R_f** = 0.36 (CHCl₃/MeOH = 15: 1); **¹H NMR** (500 MHz, CDCl₃) δ: 7.47 – 7.53 (m, 15 H) 7.55 – 7.60 (m, 7 H) 7.68 (dd, *J*=12.0, 7.6 Hz, 14 H) 7.74 – 7.81 (m, 7 H); **¹³C NMR** (126 MHz, CDCl₃) δ: 128.7 (d, *J_{P-C}*=12.7 Hz) 131.7 (d, *J_{P-C}*=106.3 Hz) 132.0 (d, *J_{P-C}*=10.9 Hz) 132.1 (d, *J_{P-C}*=4.5 Hz) 132.2 (d, *J_{P-C}*=21.8 Hz) 137.0 (d, *J_{P-C}*=100.8 Hz); **³¹P NMR** (202 MHz, CDCl₃) δ: 28.6; **FT-IR (ATR)** *v*(cm⁻¹): 519, 542, 559, 575, 692, 719, 1120, 1165, 1192, 1439; **HRMS (ESI)**: m/z = 479.1337 [C₃₀H₂₄O₂P₂+H]⁺, m/z (teor.) = 479.1324, diff. = 2.71 ppm.



Bis(3,5-xylyl)(1-naphthyl)phosphine oxide (6a)

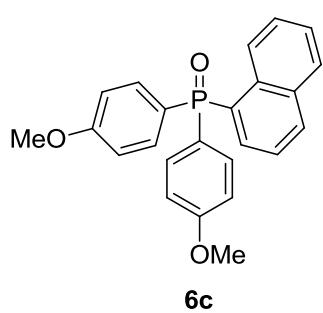
The compound **6a** was prepared according to the general procedure from bis(xylyl)phosphine oxide (**5a**) (129 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **5a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **5a**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **5a**) in acetonitrile (0.5 mL; *c_i* = 1 mol/L). Yield: 45%. White crystal, **mp** = 201.5–201.9 °C; **R_f** = 0.63 (CHCl₃/MeOH = 15: 1); **¹H NMR** (500 MHz, CDCl₃) δ: 2.32 (s, 12 H) 7.18 (d, *J*=0.6 Hz, 2 H) 7.29 – 7.35 (m, 5 H) 7.40 (ddd, *J*=8.2, 6.9, 2.5 Hz, 1 H) 7.44 – 7.53 (m, 2 H) 7.90 (d, *J*=7.9 Hz, 1 H) 8.02 (d, *J*=7.9 Hz, 1 H) 8.63 (d, *J*=8.5 Hz, 1 H); **¹³C NMR** (126 MHz, CDCl₃) δ: 21.4 (s) 124.1 (d, *J_{P-C}*=14.5 Hz) 126.4 (s) 127.2 (s) 127.8 (d, *J_{P-C}*=6.4 Hz) 128.6 (s) 129.4 (d, *J_{P-C}*=100.8 Hz) 129.6 (d, *J_{P-C}*=10.0 Hz) 132.6 (d, *J_{P-C}*=103.5 Hz) 133.0 (d, *J_{P-C}*=2.7 Hz) 133.6 (d, *J_{P-C}*=2.7 Hz) 133.7 (d, *J_{P-C}*=11.8 Hz) 133.8 (d, *J_{P-C}*=4.5 Hz) 133.9 (d, *J_{P-C}*=5.4 Hz) 138.2 (d, *J_{P-C}*=13.6 Hz); **³¹P NMR** (202 MHz, CDCl₃) δ: 32.8; **FT-IR (ATR)** *v*(cm⁻¹): 436, 471, 577, 694, 773, 806, 850, 870, 984, 1120, 1151, 1188, 1273, 1450; **HRMS (ESI)**: m/z = 385.1727 [C₂₆H₂₅OP+H]⁺, m/z (teor.) = 385.1716, diff. = 2.86 ppm.

¹⁴ T. Ghosh, P. Maity, D. Kundu, B. C. Ranu *New J. Chem.*, 2016, **40**, 9556.



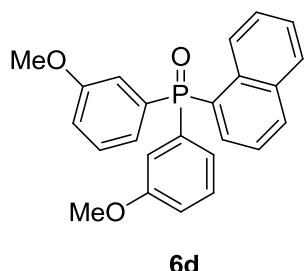
(1-Naphthyl)bis(*p*-tolyl)phosphine oxide (**6b**)

The compound **6b** was prepared according to the general procedure from bis(*p*-tolyl)phosphine oxide (**5b**) (115 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **5b**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **5b**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **5b**) in acetonitrile (0.5 mL; $c_i = 1 \text{ mol/L}$). Yield: 52%. White crystal, **mp** = 169.6–170.0 °C; **R_f** = 0.5 (CHCl₃/MeOH = 15: 1); ¹**H NMR** (500 MHz, CDCl₃) δ: 2.41 (s, 6 H) 7.25 – 7.28 (m, 4 H) 7.29 – 7.40 (m, 2 H) 7.42 – 7.51 (m, 2 H) 7.55 – 7.61 (m, 4 H) 7.86 – 7.92 (m, 1 H) 8.01 (d, $J=8.2 \text{ Hz}$, 1 H) 8.63 (d, $J=8.5 \text{ Hz}$, 1 H); ¹³**C NMR** (126 MHz, CDCl₃) δ: 21.6 (s) 124.1 (d, $J_{P-C}=13.6 \text{ Hz}$) 126.4 (s) 127.2 (s) 127.7 (d, $J_{P-C}=5.5 \text{ Hz}$) 128.7 (s) 129.5 (d, $J_{P-C}=101.7 \text{ Hz}$) 129.3 (d, $J_{P-C}=12.7 \text{ Hz}$) 129.8 (m, $J_{P-C}=107.2 \text{ Hz}$) 132.1 (d, $J_{P-C}=10.0 \text{ Hz}$) 133.1 (d, $J_{P-C}=2.7 \text{ Hz}$) 133.7 (d, $J_{P-C}=11.8 \text{ Hz}$) 133.8 (d, $J_{P-C}=8.2 \text{ Hz}$) 133.9 (d, $J_{P-C}=9.1 \text{ Hz}$) 142.3 (d, $J_{P-C}=2.7 \text{ Hz}$); ³¹**P NMR** (202 MHz, CDCl₃) δ: 32.5; FT-IR (ATR) $\nu(\text{cm}^{-1})$: 478, 503, 519, 532, 544, 550, 619, 660, 675, 777, 812, 1115, 1149, 1184; HRMS (ESI): m/z = 357.1393 [C₂₄H₂₁OP C₁₁H₂₃OP+H]⁺, m/z (teor.) = 357.1403, diff. = -2.80 ppm.



Bis(4-methoxyphenyl)(1-naphthyl)phosphine oxide (**6c**)

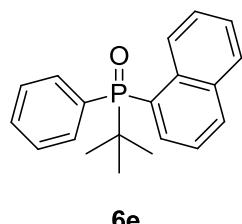
The compound **6c** was prepared according to the general procedure from bis(*p*-anisyl)phosphine oxide (**5c**) (131 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **5c**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **5c**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **5c**) in acetonitrile (0.5 mL; $c_i = 1 \text{ mol/L}$). Yield: 8%. Light yellow solid, **mp** = 59.1–62.9 °C; **R_f** = 0.35 (CHCl₃/MeOH = 15: 1); ¹**H NMR** (500 MHz, CDCl₃) δ: 3.86 (s, 6 H) 6.98 (dd, $J=8.8, 1.9 \text{ Hz}$, 4 H) 7.29 – 7.35 (m, 1 H) 7.37 – 7.42 (m, 1 H) 7.47 (d, $J=8.5 \text{ Hz}$, 1 H) 7.49 – 7.53 (m, 1 H) 7.60 (dd, $J=11.5, 8.7 \text{ Hz}$, 4 H) 7.90 (d, $J=7.9 \text{ Hz}$, 1 H) 8.02 (d, $J=8.2 \text{ Hz}$, 1 H) 8.62 (d, $J=8.5 \text{ Hz}$, 1 H); ¹³**C NMR** (126 MHz, CDCl₃) δ: 55.3 (s) 114.1 (d, $J_{P-C}=13.6 \text{ Hz}$) 124.3 (d, $J_{P-C}=111.7 \text{ Hz}$) 124.1 (d, $J_{P-C}=14.5 \text{ Hz}$) 126.4 (s) 127.2 (s) 127.7 (d, $J_{P-C}=6.4 \text{ Hz}$) 128.7 (s) 129.7 (d, $J_{P-C}=102.6 \text{ Hz}$) 133.1 (d, $J_{P-C}=2.7 \text{ Hz}$) 133.7 (d, $J_{P-C}=11.8 \text{ Hz}$) 133.8 (d, $J_{P-C}=8.2 \text{ Hz}$) 133.9 (d, $J_{P-C}=10.9 \text{ Hz}$) 162.4 (d, $J_{P-C}=2.7 \text{ Hz}$); ³¹**P NMR** (202 MHz, CDCl₃) δ: 32.3; FT-IR (ATR) $\nu(\text{cm}^{-1})$: 513, 542, 783, 802, 816, 825, 1018, 1119, 1178, 1194, 1257, 1296, 1439, 1462, 1502, 1568, 1595; HRMS (ESI): m/z = 389.1287 [C₂₄H₂₁O₃P+H]⁺, m/z (teor.) = 389.1301, diff. = -3.60 ppm.



Bis(3-methoxyphenyl)(1-naphthyl)phosphine oxide (**6d**)

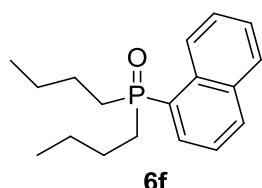
The compound **6d** was prepared according to the general procedure from bis(*m*-anisyl)phosphine oxide (**5d**) (131 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **5d**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **5d**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **5d**) in acetonitrile (0.5 mL; $c_i = 1 \text{ mol/L}$). Yield: 44%. White crystal, **mp** = 129.9–131.6 °C; **R_f** = 0.5 (CHCl₃/MeOH = 15: 1); ¹**H NMR** (500 MHz, CDCl₃) δ: 3.80 (s, 6 H) 7.06 – 7.16 (m, 4 H) 7.29 – 7.41 (m, 6 H) 7.44 – 7.52 (m, 2 H) 7.89 (d, $J=8.2 \text{ Hz}$, 1 H) 8.01 (d, $J=8.2 \text{ Hz}$, 1 H) 8.62 (d, $J=8.5 \text{ Hz}$, 1 H); ¹³**C NMR** (126 MHz, CDCl₃) δ: 55.4 (s) 116.7 (d, $J_{P-C}=10.9 \text{ Hz}$) 118.2 (d, $J_{P-C}=2.7 \text{ Hz}$) 124.1 (d, $J_{P-C}=14.5 \text{ Hz}$) 124.3 (d, $J_{P-C}=10.0 \text{ Hz}$) 126.5 (s) 127.4 (s) 127.6 (d, $J_{P-C}=5.4 \text{ Hz}$) 128.8 (d, $J_{P-C}=102.8 \text{ Hz}$) 128.8 (s) 129.7 (d, $J_{P-C}=14.5 \text{ Hz}$) 133.3 (d, $J_{P-C}=2.7 \text{ Hz}$) 134.0 (d, $J_{P-C}=14.5 \text{ Hz}$)

$J_{P-C} = 103.5$ Hz) 133.7 (d, $J_{P-C} = 8.0$ Hz) 133.8 (d, $J_{P-C} = 11.8$ Hz) 133.9 (d, $J_{P-C} = 9.1$ Hz) 159.7 (d, $J_{P-C} = 15.4$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ : 32.7; FT-IR (ATR) ν (cm⁻¹): 490, 501, 528, 696, 775, 789, 1034, 1099, 1147, 1186, 1240, 1288, 1423, 1477, 1574; HRMS (ESI): m/z = 389.1309 [C₂₄H₂₁O₃P+H]⁺, m/z (teor.) = 389.1301, diff. = 2.06 ppm.



tert-Butyl(1-naphthyl)phenylphosphine oxide (6e)

The compound **6e** was prepared according to the general procedure from *tert*-butyl(phenyl)phosphine oxide (**5e**) (64 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **5e**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **5e**) and Ni10/CeO₂ (2.9 mg, 1 mol % of Ni to **5e**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 25%. White crystal, mp = 142.6-143.2 °C (lit. 137-138 °C)¹⁵; R_f = 0.64 (CHCl₃/MeOH = 15: 1); 1H NMR (500 MHz, CDCl₃) δ : 1.42 (d, $J = 14.8$ Hz, 9 H) 7.43 - 7.54 (m, 6 H) 7.84 - 7.93 (m, 3 H) 7.94 - 8.03 (m, 2 H) 8.97 - 9.02 (m, 1 H); ^{13}C NMR (126 MHz, CDCl₃) δ : 26.2 (s) 35.0 (d, $J_{P-C} = 70.8$ Hz) 123.6 (d, $J_{P-C} = 12.7$ Hz) 126.2 (s) 127.0 (s) 127.2 (d, $J_{P-C} = 89.0$ Hz) 128.2 (d, $J_{P-C} = 3.5$ Hz) 128.2 (d, $J_{P-C} = 11.8$ Hz) 128.7 (s) 131.3 (d, $J_{P-C} = 2.7$ Hz) 132.3 (d, $J_{P-C} = 8.2$ Hz) 132.4 (d, $J_{P-C} = 10.9$ Hz) 132.8 (d, $J_{P-C} = 89.9$ Hz) 132.8 (d, $J_{P-C} = 2.7$ Hz) 134.2 (d, $J_{P-C} = 9.1$ Hz) 134.8 (d, $J_{P-C} = 7.3$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ : 44.5; FT-IR (ATR) ν (cm⁻¹): 447, 492, 513, 532, 548, 613, 636, 669, 704, 754, 777, 804, 823, 1101, 1155, 1174, 1439; HRMS (ESI): m/z = 309.1400 [C₂₀H₂₁OP+H]⁺, m/z (teor.) = 309.1403, diff. = -0.97 ppm.

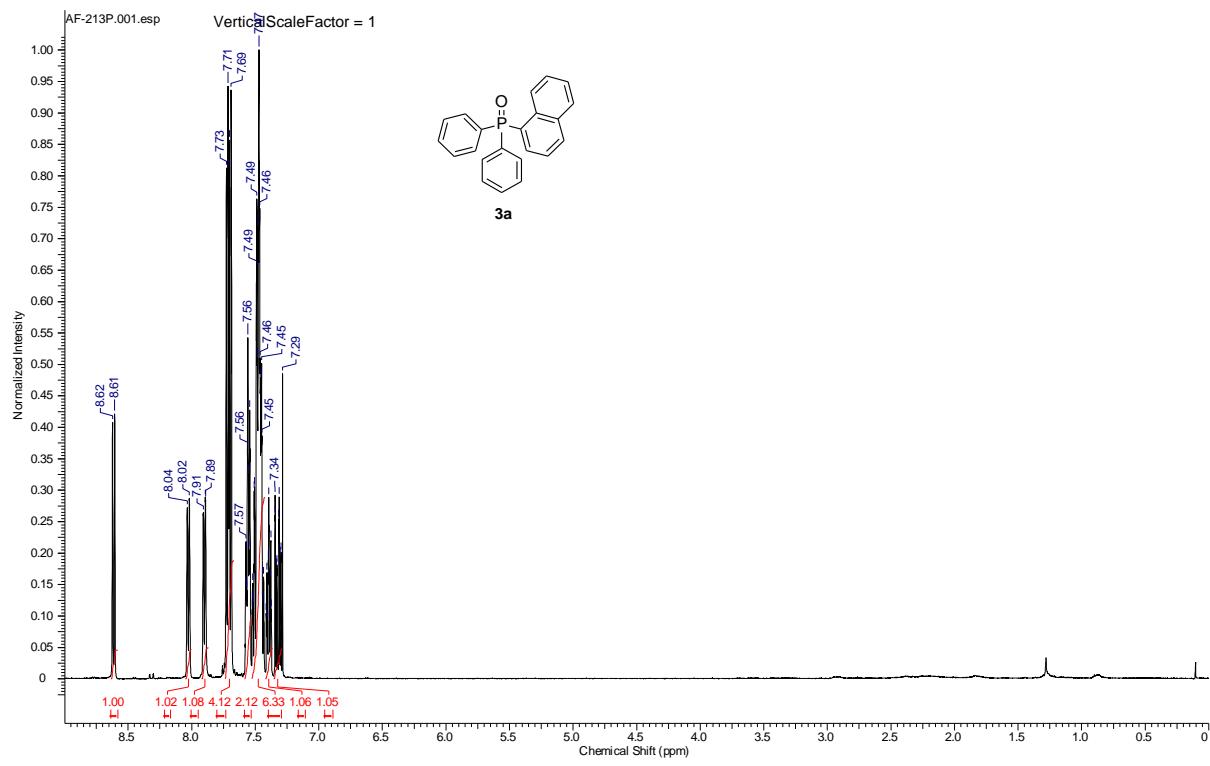


Di(*n*-butyl(1-naphthyl))phosphine oxide (6f)

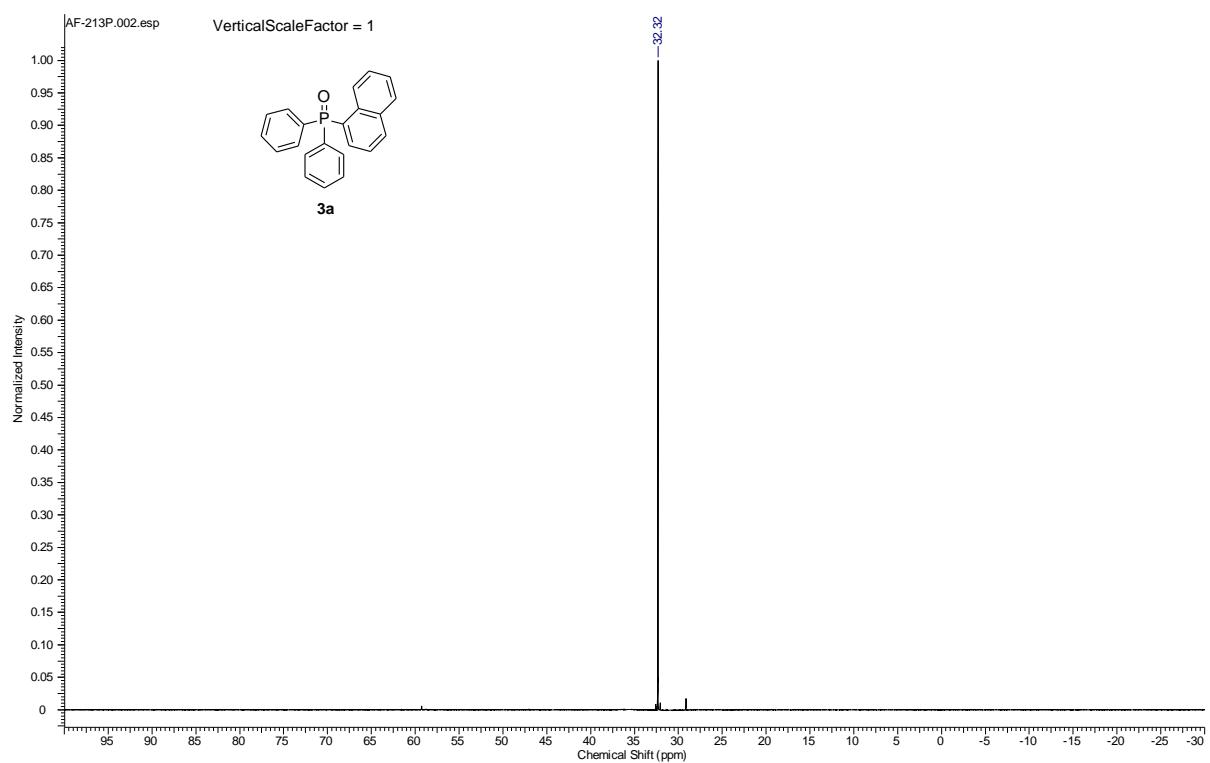
The compound **6f** was prepared according to the general procedure from di(*n*-butylphosphine oxide (**5f**) (81 mg, 0.5 mmol), 1-bromonaphthalene (**2a**, 0.077 mL, 0.55 mmol, 1.1 equiv to **1a**), K₂CO₃ (207 mg, 1.5 mmol, 3 equiv to **5f**) and Ni10/CeO₂ (2.9 mg, 5 mol % of Ni to **5f**) in acetonitrile (0.5 mL; $c_i = 1$ mol/L), the reaction mixture was heated at 100 °C for 72 h. Yield: 5%, yellow oil; R_f = 0.5 (hexan/*i*-PrOH = 6: 1); 1H NMR (500 MHz, CDCl₃) δ : ppm 0.82 - 0.89 (m, 6 H) 1.32 - 1.43 (m, 4 H) 1.43 - 1.52 (m, 2 H) 1.58 - 1.70 (m, 2 H) 2.12 - 2.22 (m, 4 H) 7.53 - 7.66 (m, 3 H) 7.94 (d, $J = 7.9$ Hz, 1 H) 7.97 - 8.05 (m, 2 H) 8.61 (d, $J = 8.2$ Hz, 1 H); ^{13}C NMR (126 MHz, CDCl₃) δ : 13.6 (s) 23.8 (d, $J = 3.6$ Hz) 24.1 (d, $J = 14.5$ Hz) 30.5 (d, $J = 68.1$ Hz) 124.7 (d, $J = 12.7$ Hz) 125.7 (d, $J = 3.6$ Hz) 126.2 (s) 127.2 (s) 128.7 (d, $J = 87.2$ Hz) 129.3 (s) 132.2 (d, $J = 7.3$ Hz) 132.6 (d, $J = 3.6$ Hz) 133.3 (d, $J = 9.1$ Hz) 133.8 (d, $J = 9.1$ Hz); ^{31}P NMR (202 MHz, CDCl₃) δ : 42.5; FT-IR (ATR) ν (cm⁻¹): 777, 1159, 1209, 1466, 1506, 2870, 2956; HRMS (ESI): m/z = 289.1714 [C₁₈H₂₅OP+H]⁺, m/z (teor.) = 289.1716, diff. = -0.69 ppm.

¹⁵ J. R. Corfield, N. J. De'Ath , S. Trippett *J. Chem. Soc. (C)*, 1971, 1930.

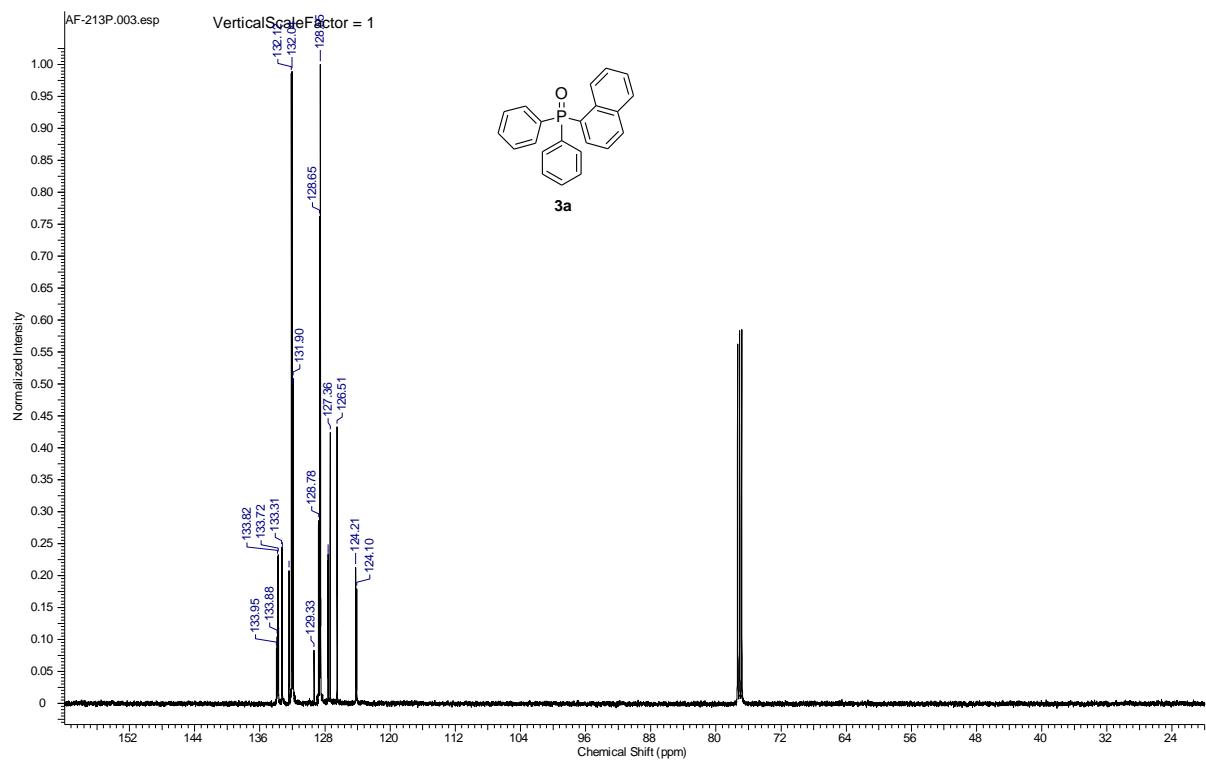
^1H NMR, ^{13}C NMR and ^{31}P NMR spectra of 3a-3s' and 6a-6f.



^1H NMR spectra of 1-naphthyl(diphenyl)phosphine oxide (**3a**) (CDCl_3 , 500 MHz).

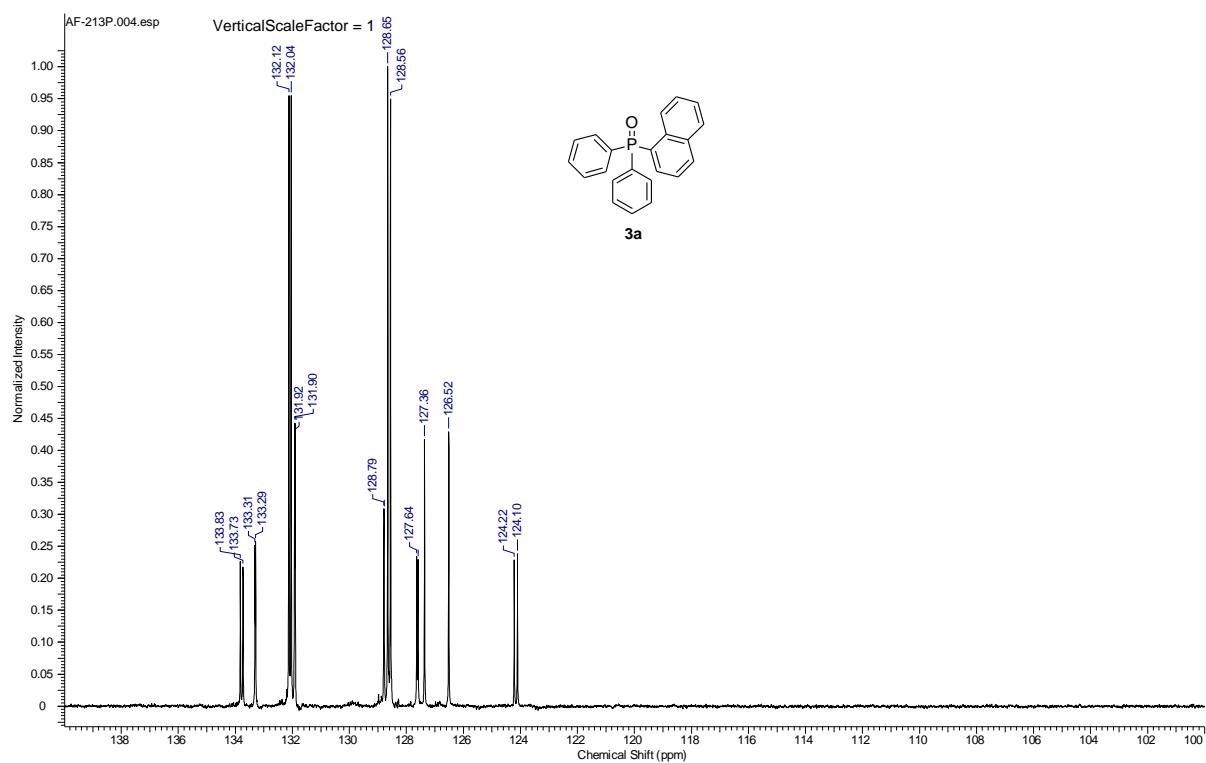


^{31}P NMR spectra of 1-naphthyl(diphenyl)phosphine oxide (**3a**) (CDCl_3 , 202 MHz).



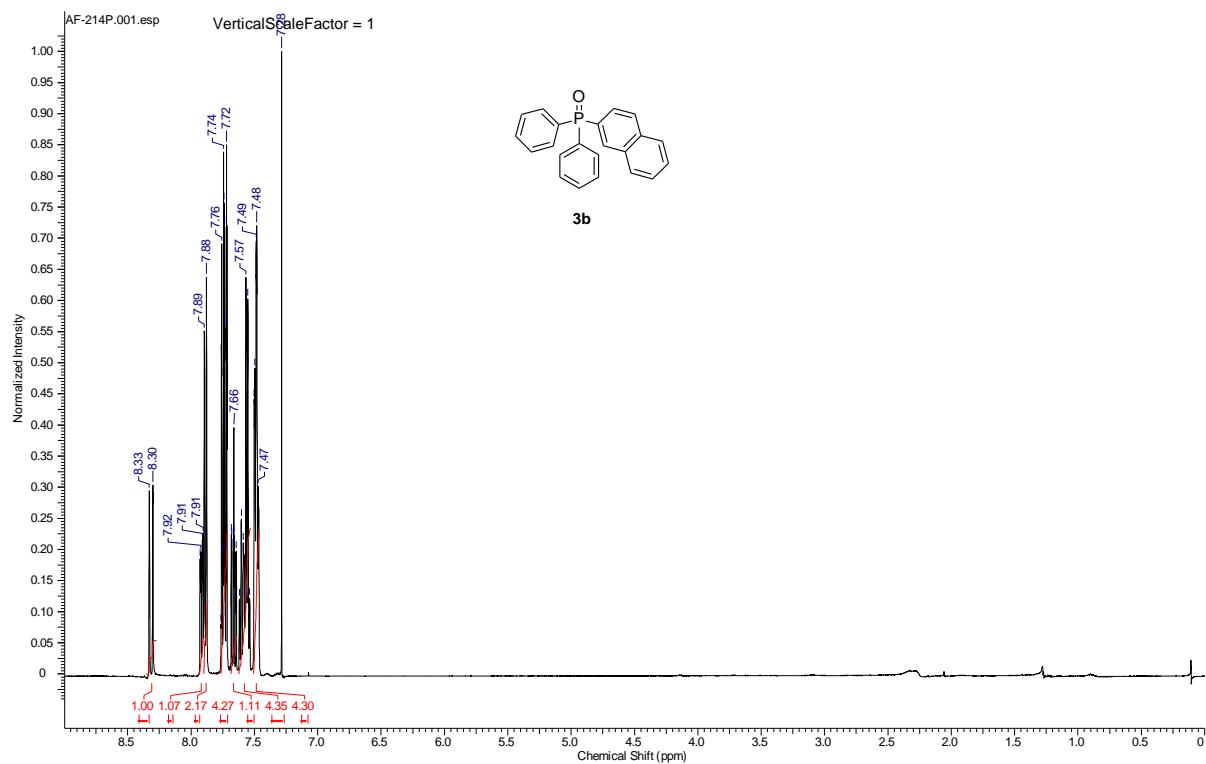
E:\FTP\NMR\ELA LASTAWIECKA\AF-213P\AF-213P.003.esp

³¹C NMR spectra of 1-naphthyl(diphenyl)phosphine oxide (**3a**) (CDCl₃, 126 MHz).



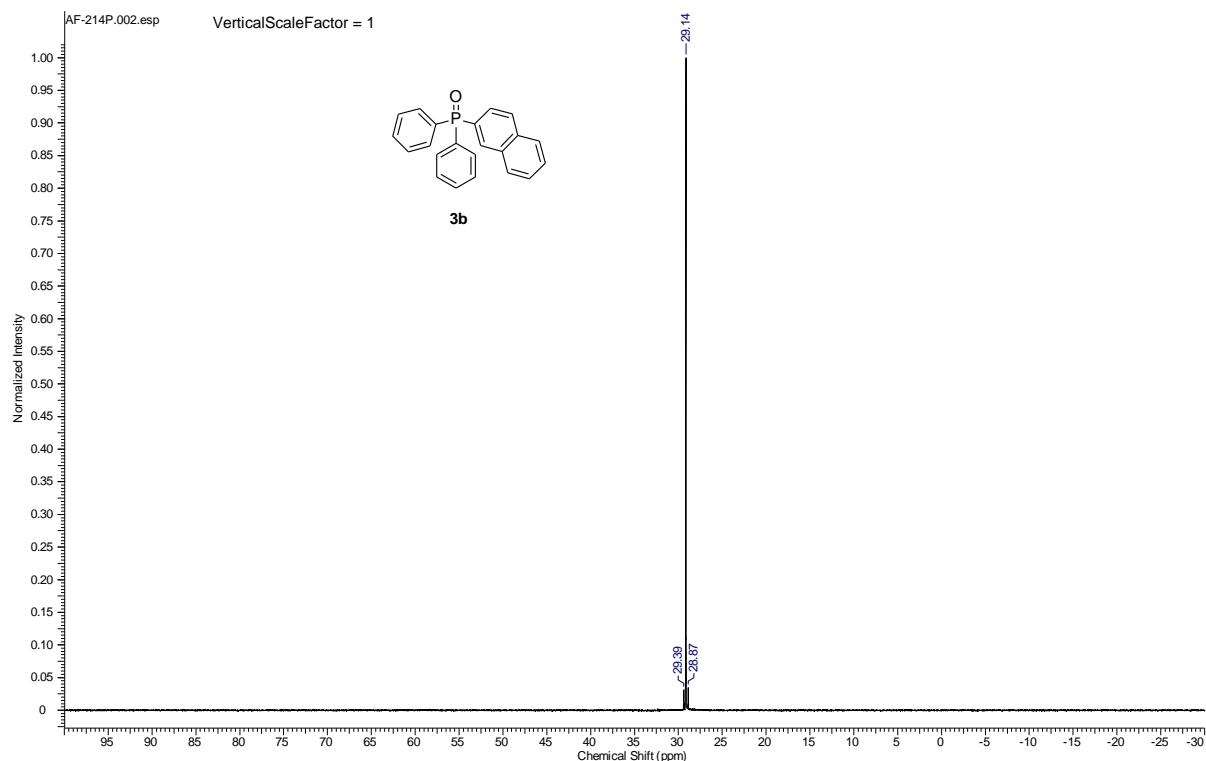
E:\FTP\NMR\ELA LASTAWIECKA\AF-213P\AF-213P.004.esp

DEPT 135 spectra of 1-naphthyl(diphenyl)phosphine oxide (**3a**) (CDCl₃, 126 MHz).



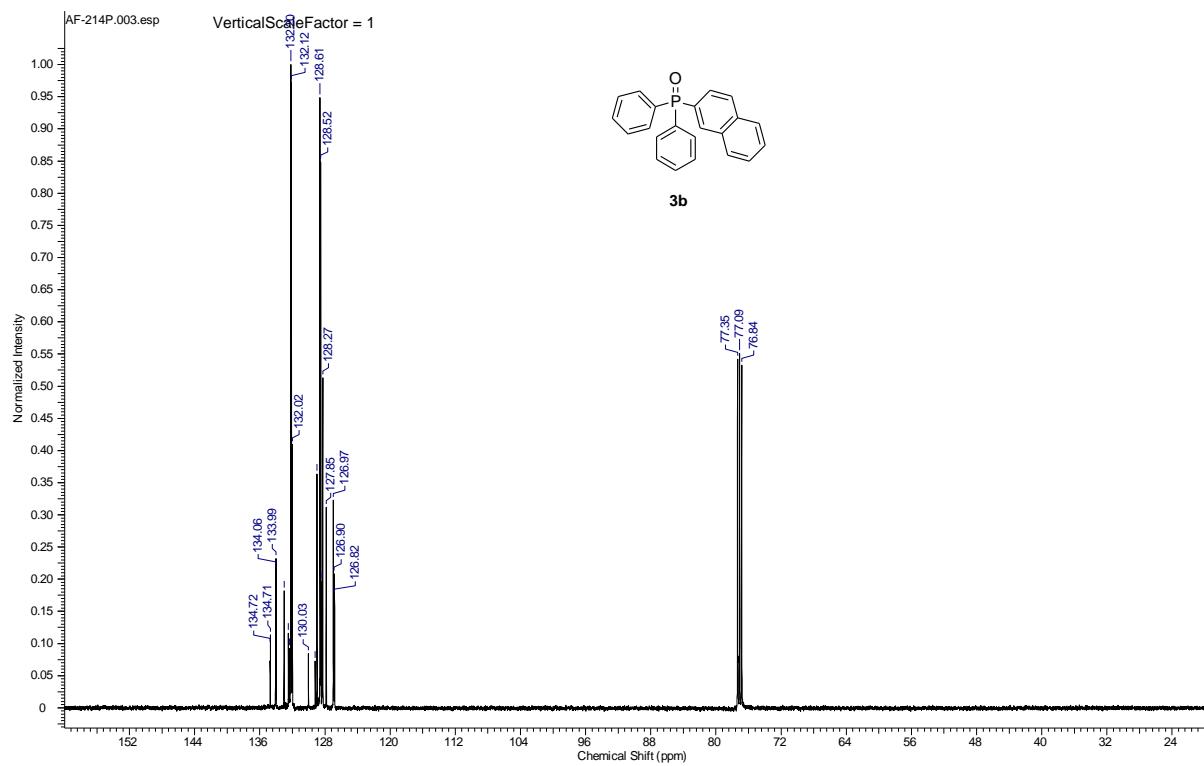
E:\FTP\NMR\ELA LASTAWIECKA\AF-214P\AF-214P.001.esp

¹H NMR spectra of 2-naphthyl(diphenyl)phosphine oxide (**3b**) (CDCl₃, 500 MHz).

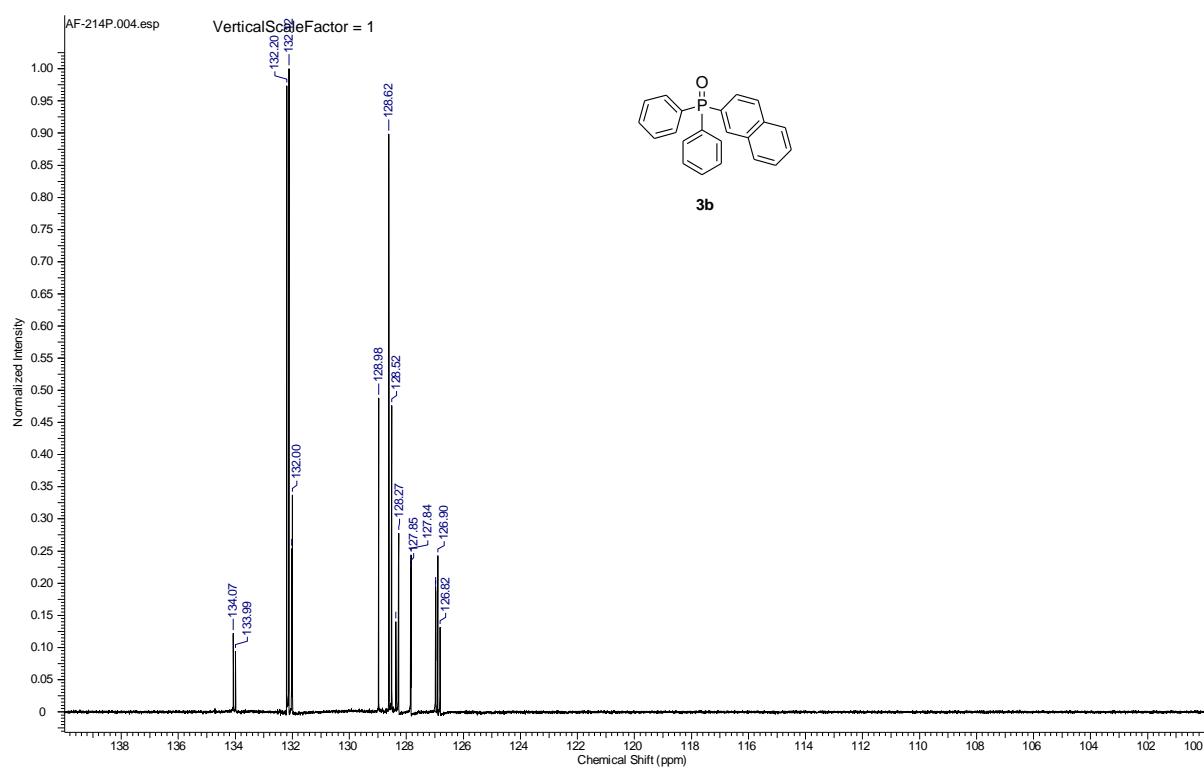


E:\FTP\NMR\ELA LASTAWIECKA\AF-214P\AF-214P.002.esp

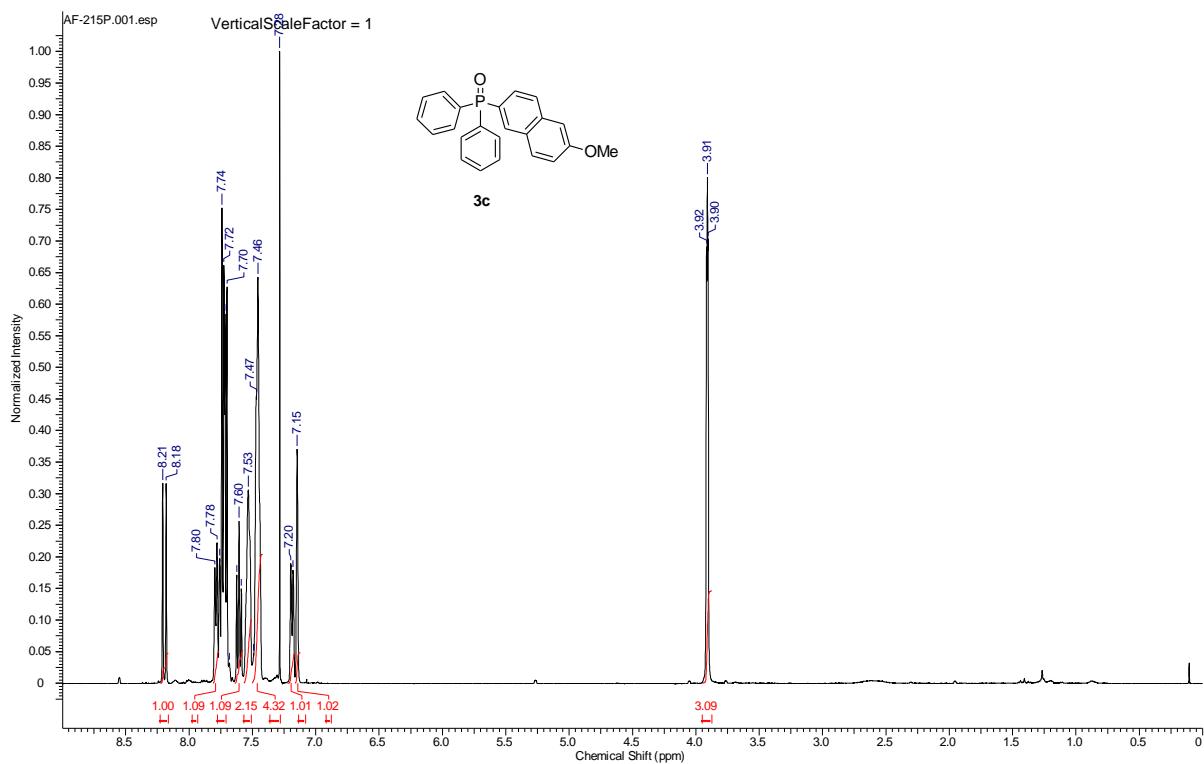
³¹P NMR spectra of 2-naphthyl(diphenyl)phosphine oxide (**3b**) (CDCl₃, 202 MHz).



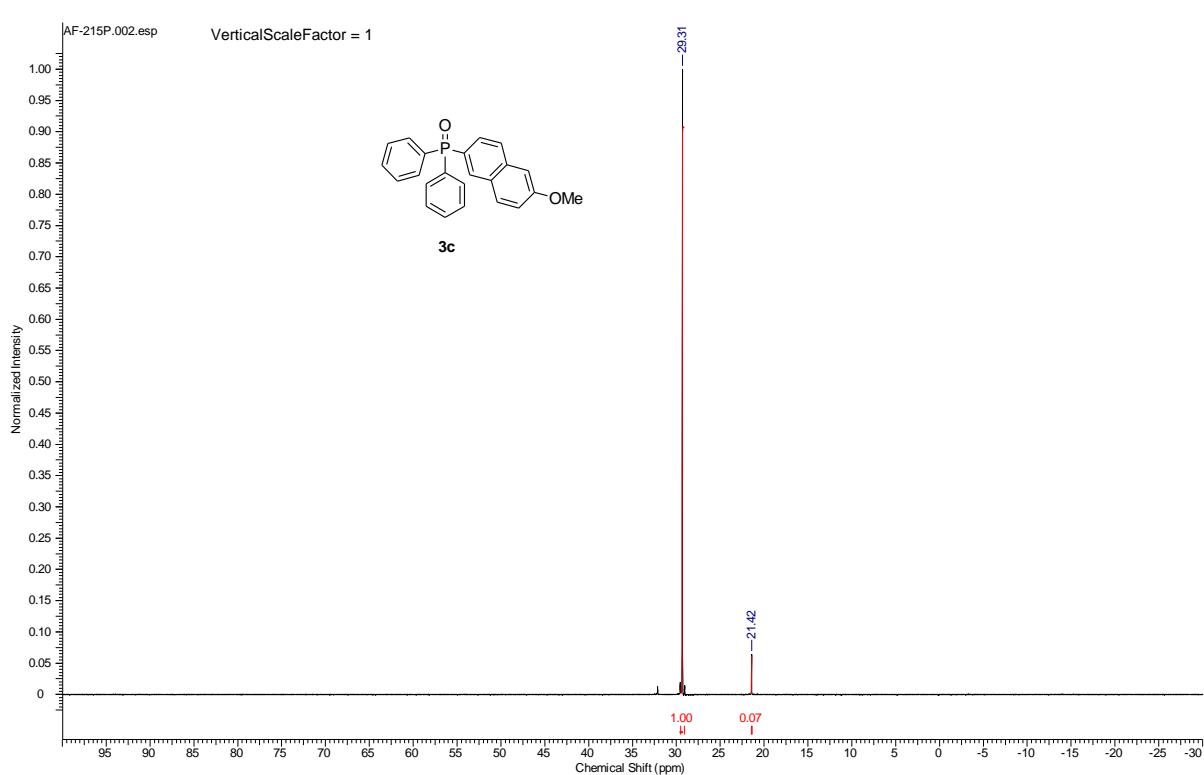
³¹C NMR spectra of 2-naphthyl(diphenyl)phosphine oxide (**3b**) (CDCl₃, 126 MHz).



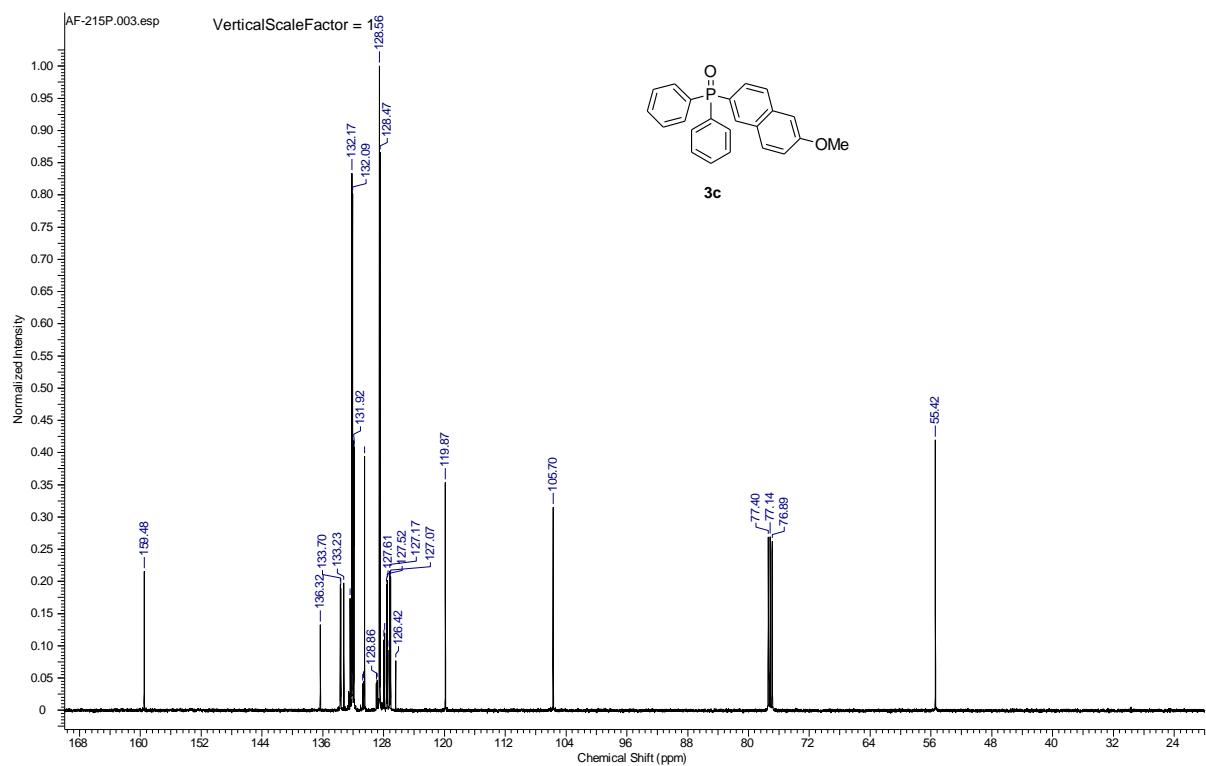
DEPT-135 spectra of 2-naphthyl(diphenyl)phosphine oxide (2b) (CDCl_3 , 126 MHz)



¹H NMR spectra of (6-methoxy-2-naphthyl)(diphenyl)phosphine oxide (**3c**) (CDCl₃, 500 MHz).

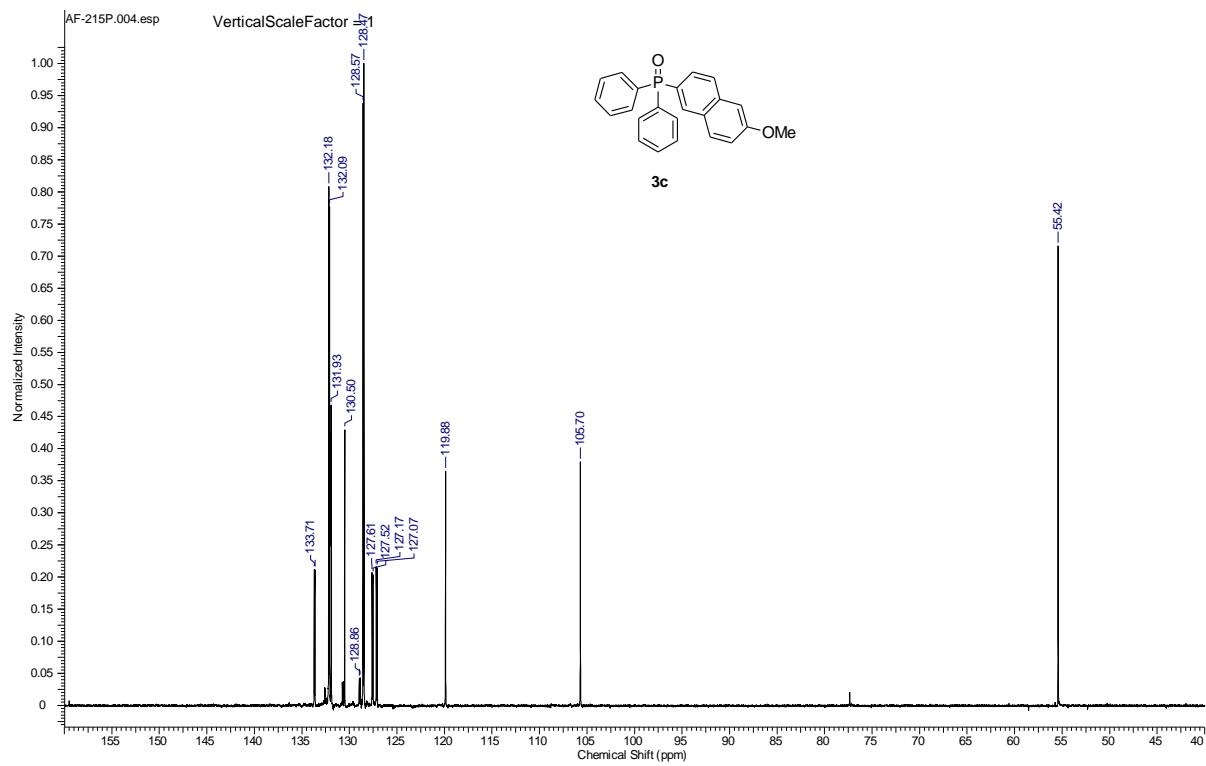


³¹P NMR spectra of bis(6-methoxy-2-naphthyl)(diphenyl)phosphine oxide (**3c**) (CDCl₃, 202 MHz).



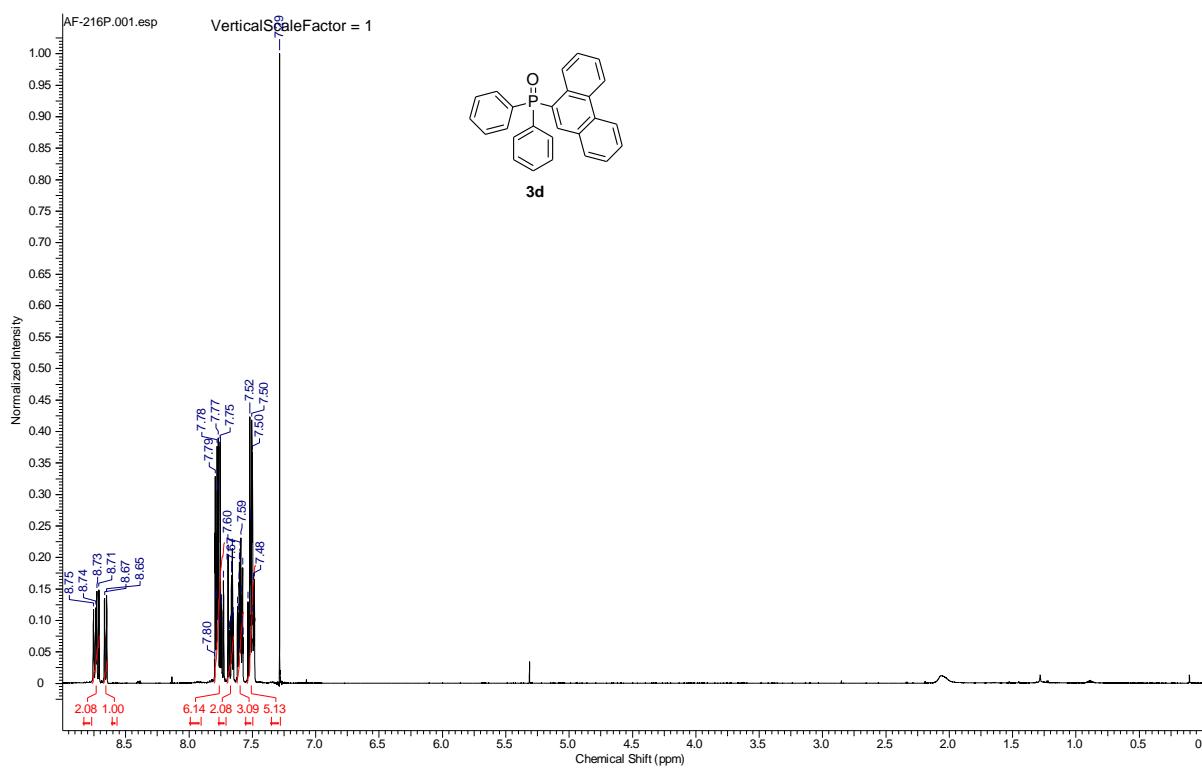
E:\FTP\NMR\ELA LASTAWIECKA\AF-215P\AF-215P.003.esp

³¹C NMR spectra of (6-methoxy-2-naphthyl)(diphenyl)phosphine oxide (**3c**) (CDCl₃, 126 MHz).

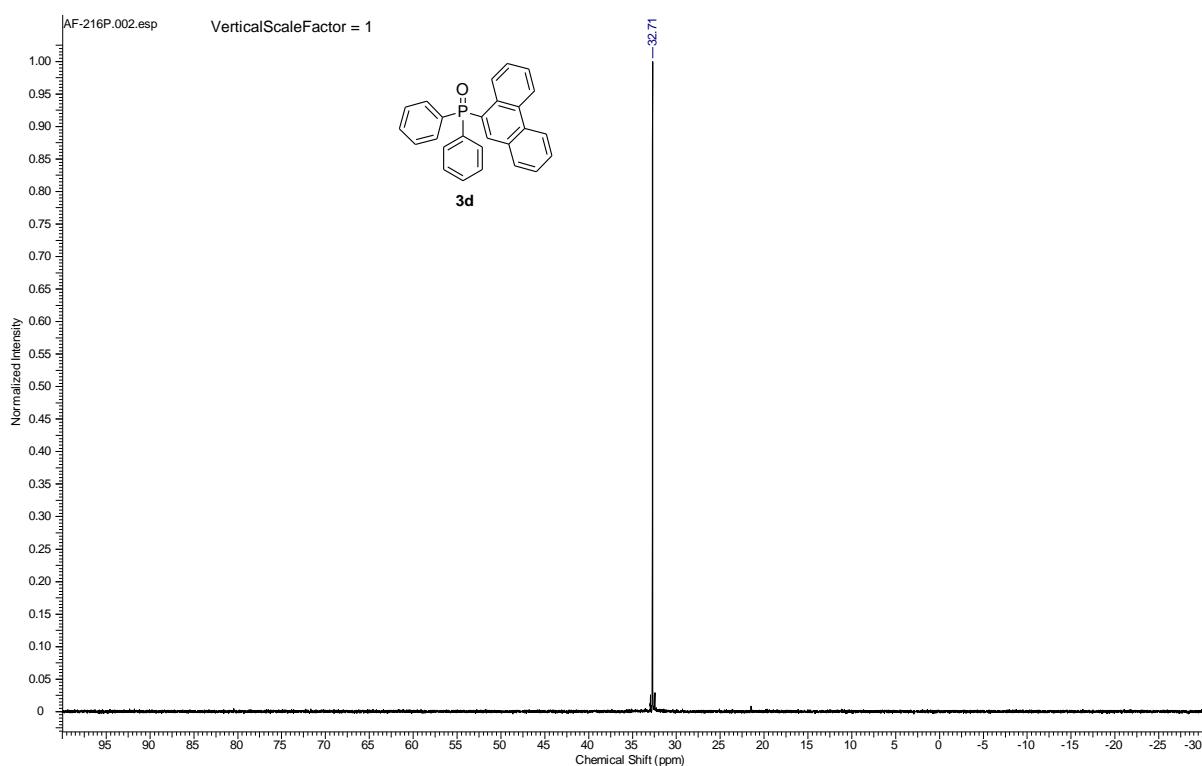


E:\FTP\NMR\ELA LASTAWIECKA\AF-215P\AF-215P.004.esp

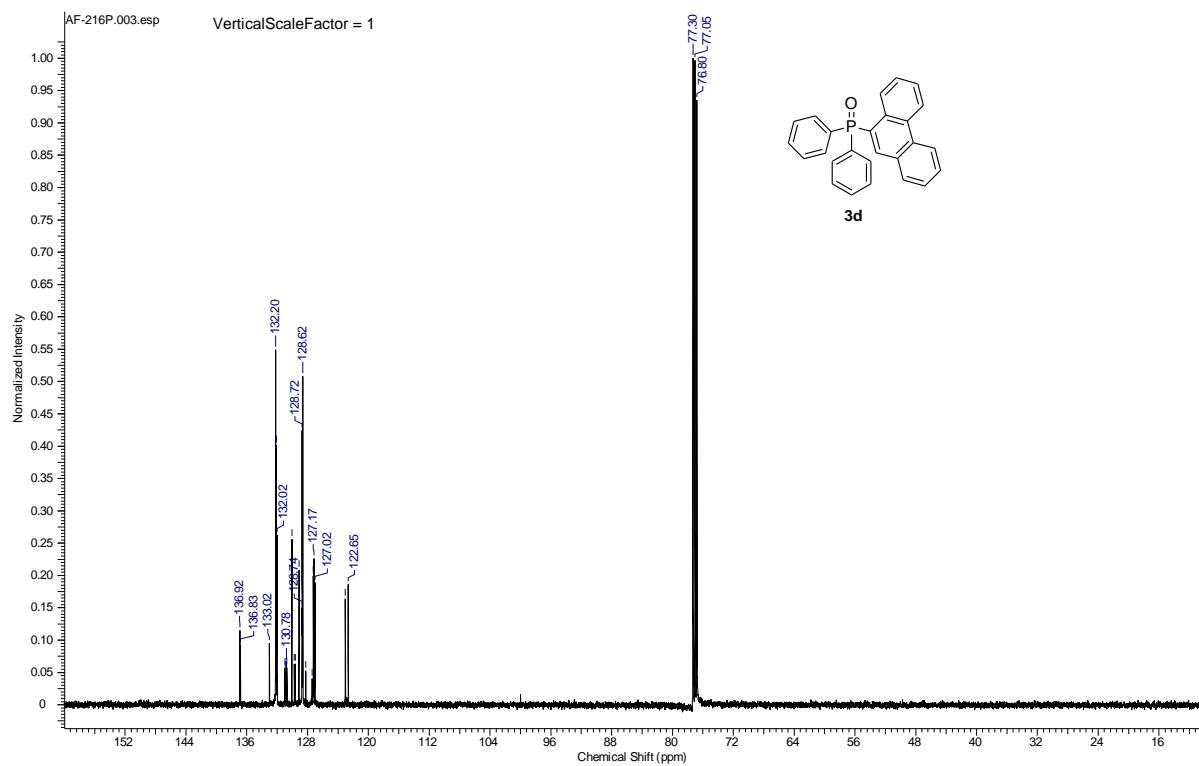
DEPT 135 spectra of (6-methoxy-2-naphthyl)(diphenyl)phosphine oxide (**3c**) (CDCl₃, 126 MHz).



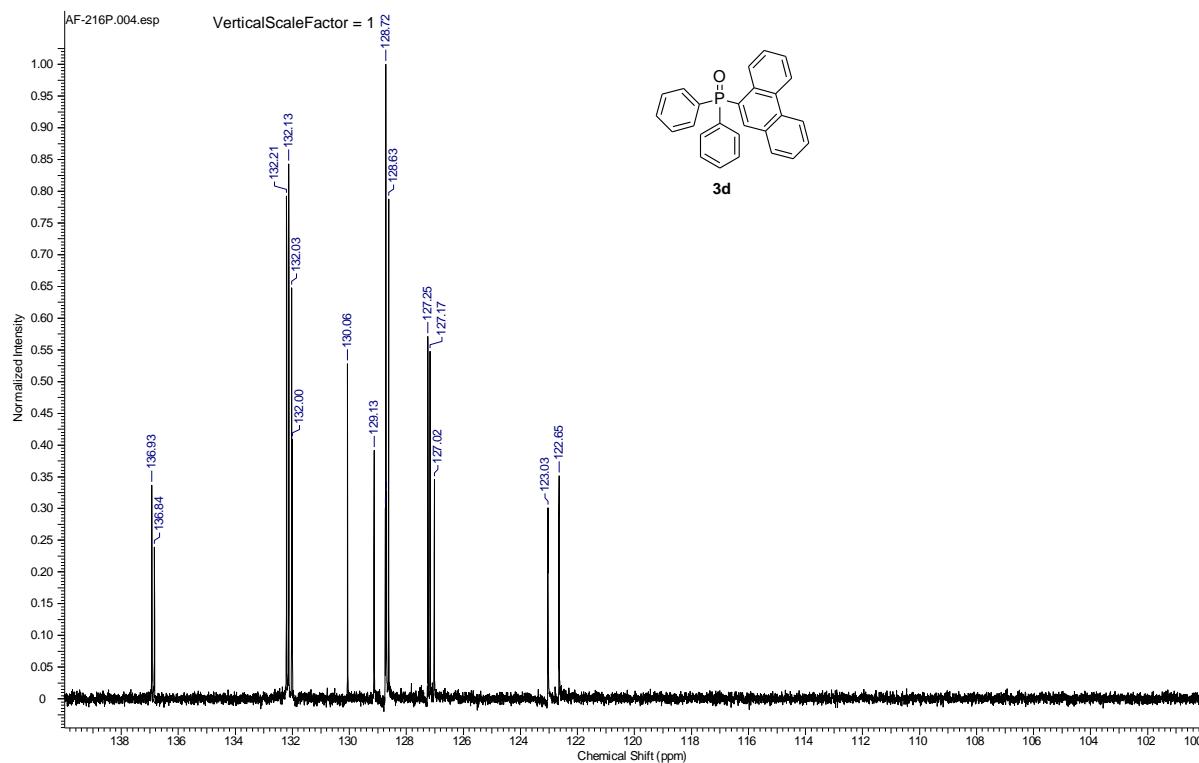
¹H NMR spectra of 9-phenanthryl(diphenyl)phosphine oxide (**3d**) (CDCl₃, 500 MHz).



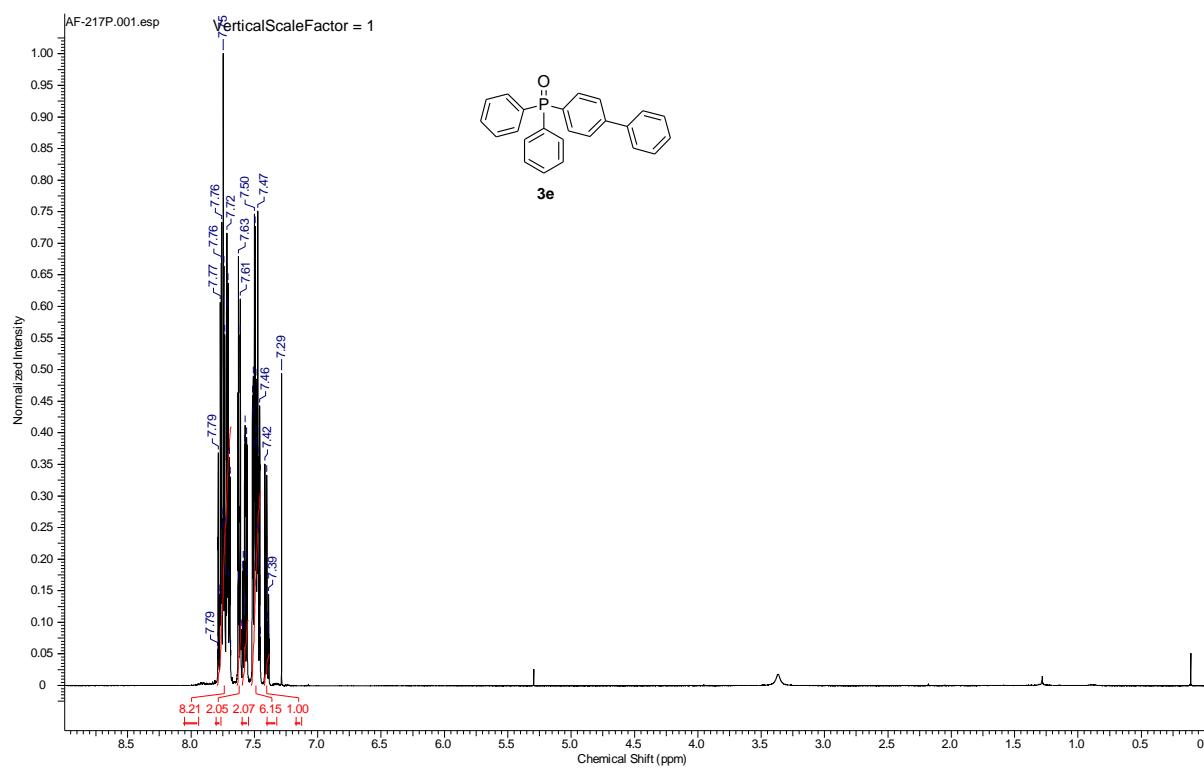
³¹P NMR spectra of 9-phenanthryl(diphenyl)phosphine oxide (**3d**) (CDCl₃, 202 MHz).



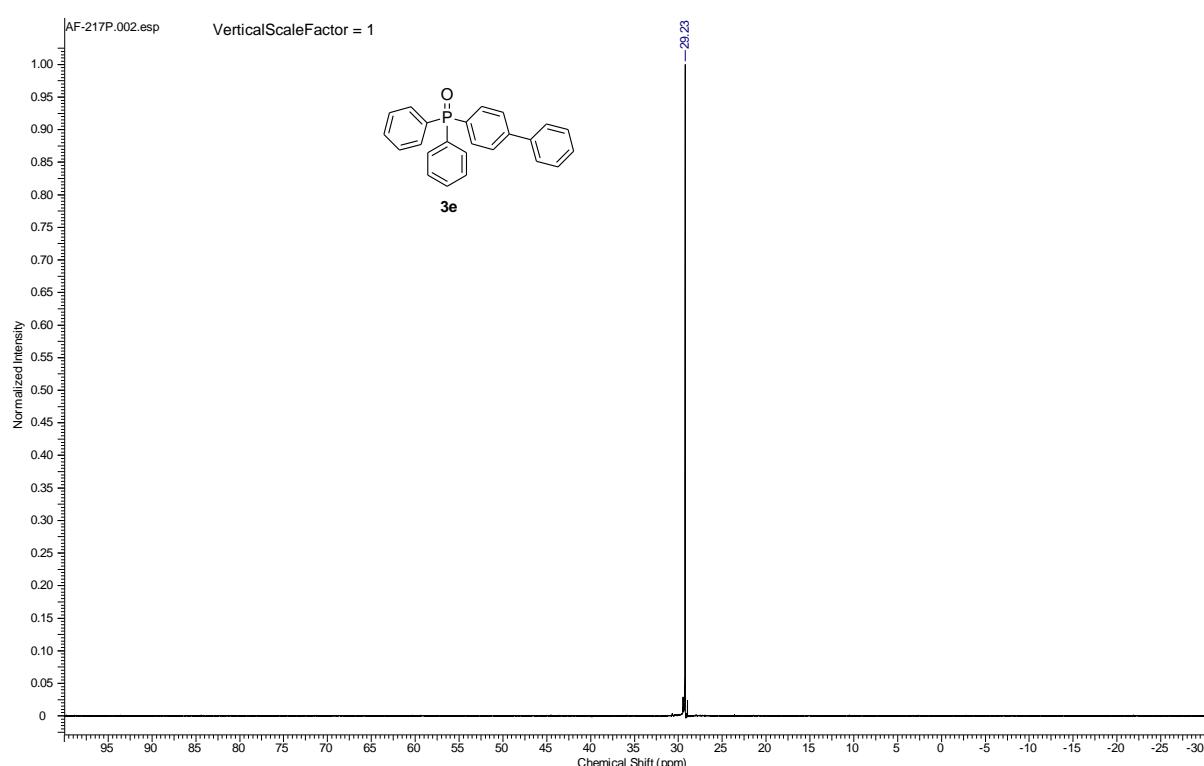
³¹C NMR spectra of 9-phenanthryl(diphenyl)phosphine oxide (**3d**) (CDCl₃, 126 MHz).



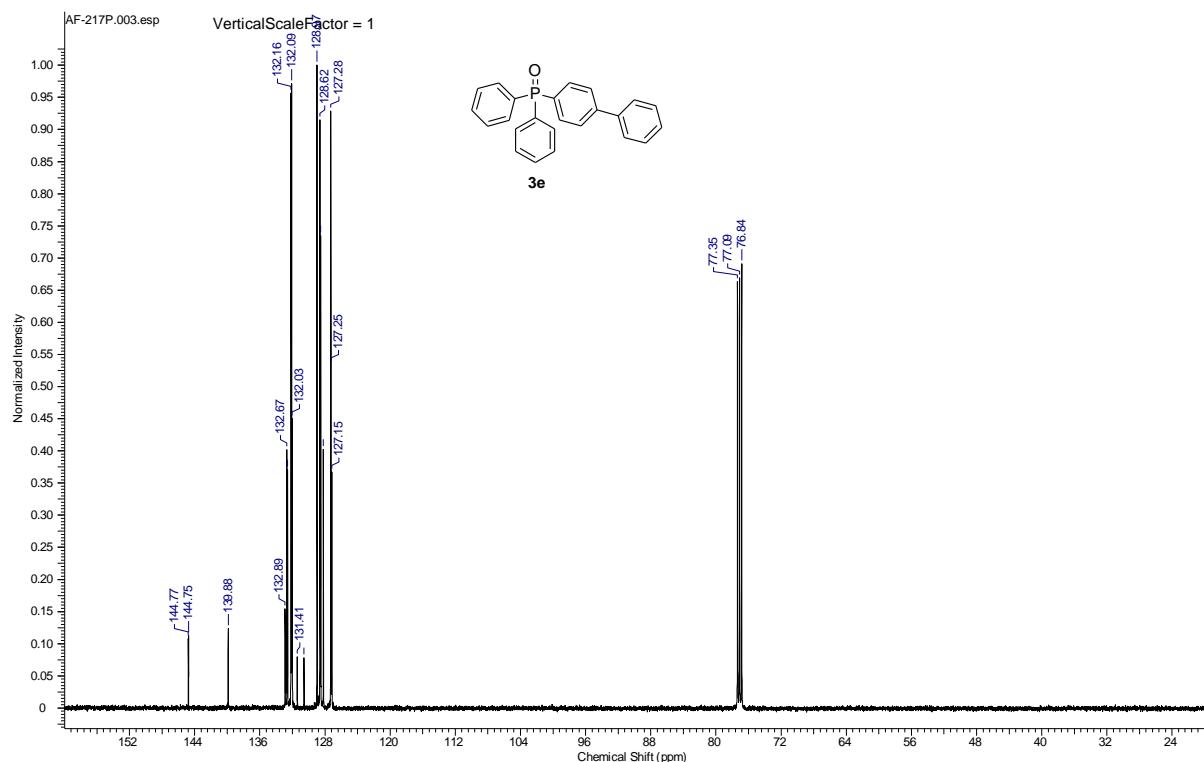
DEPT-135 spectra of 9-phenanthryl(dibenzyl)phosphine oxide (2d) (CDCl_3 , 126 MHz)



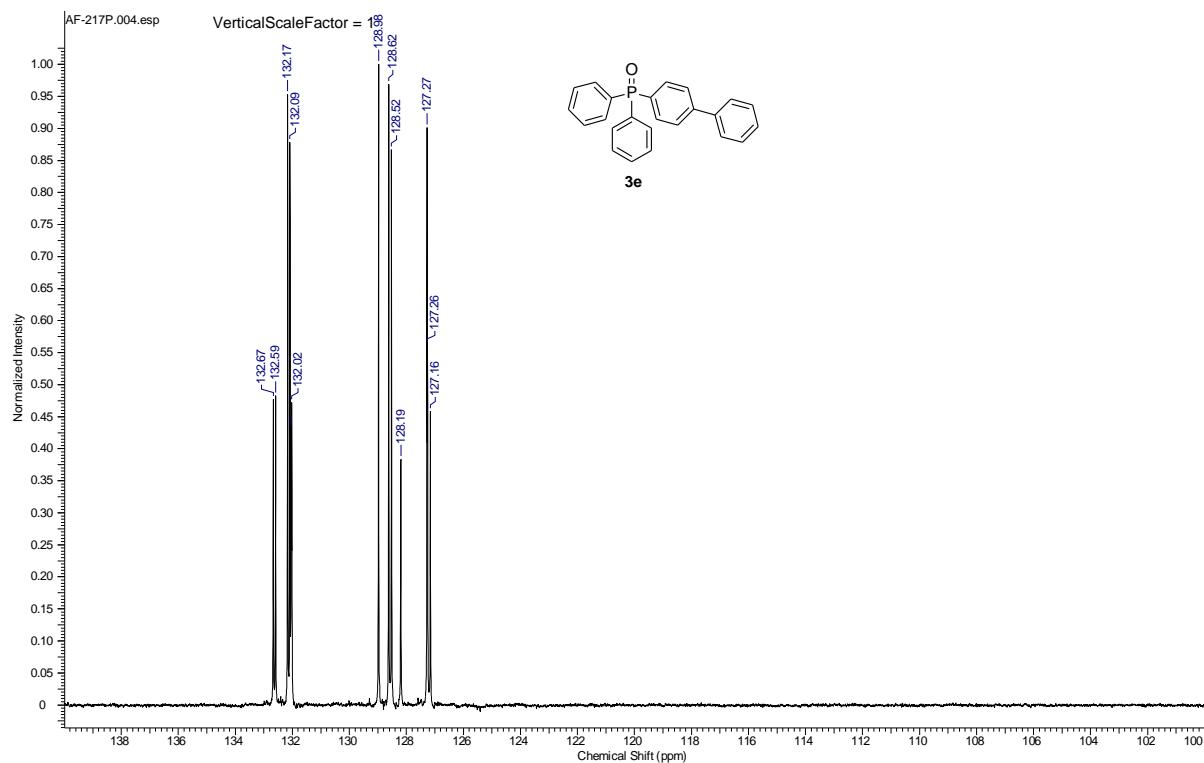
¹H NMR spectra of biphenyl-4-yl(diphenyl)phosphine oxide (**3e**) (CDCl₃, 500 MHz).



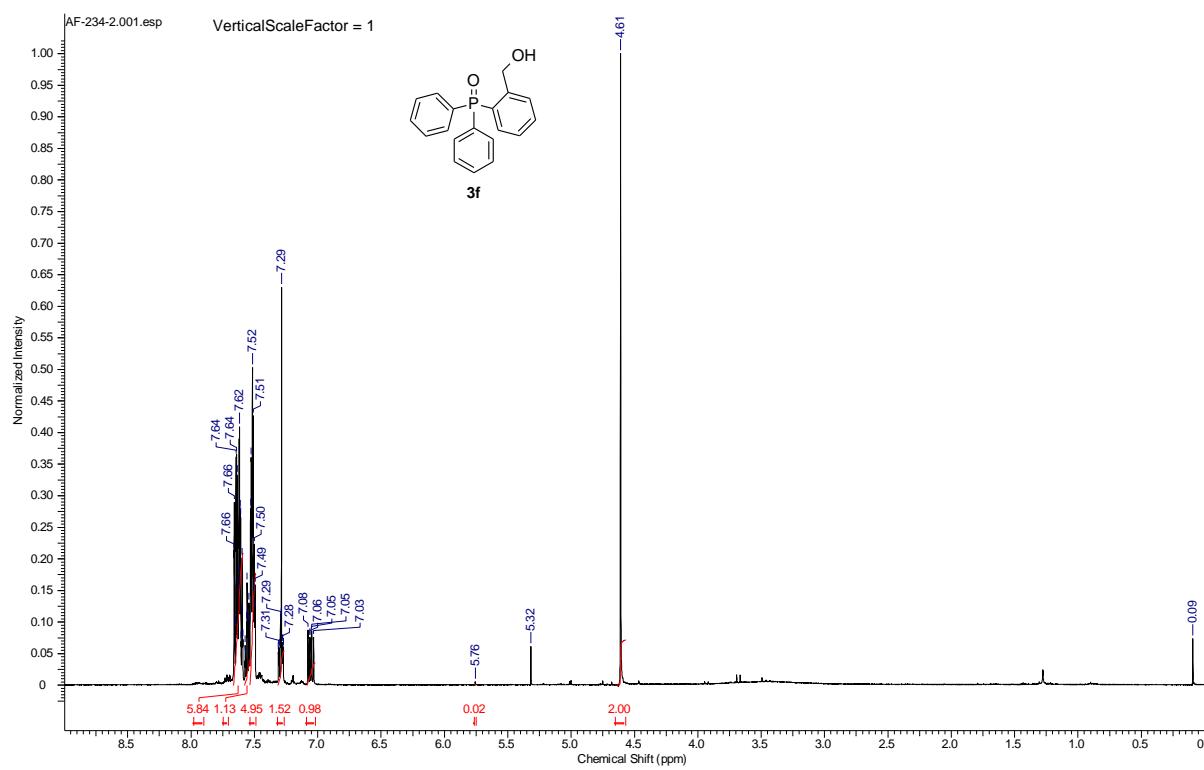
³¹P NMR spectra of biphenyl-4-yl(diphenyl)phosphine oxide (**3e**) (CDCl₃, 202 MHz).



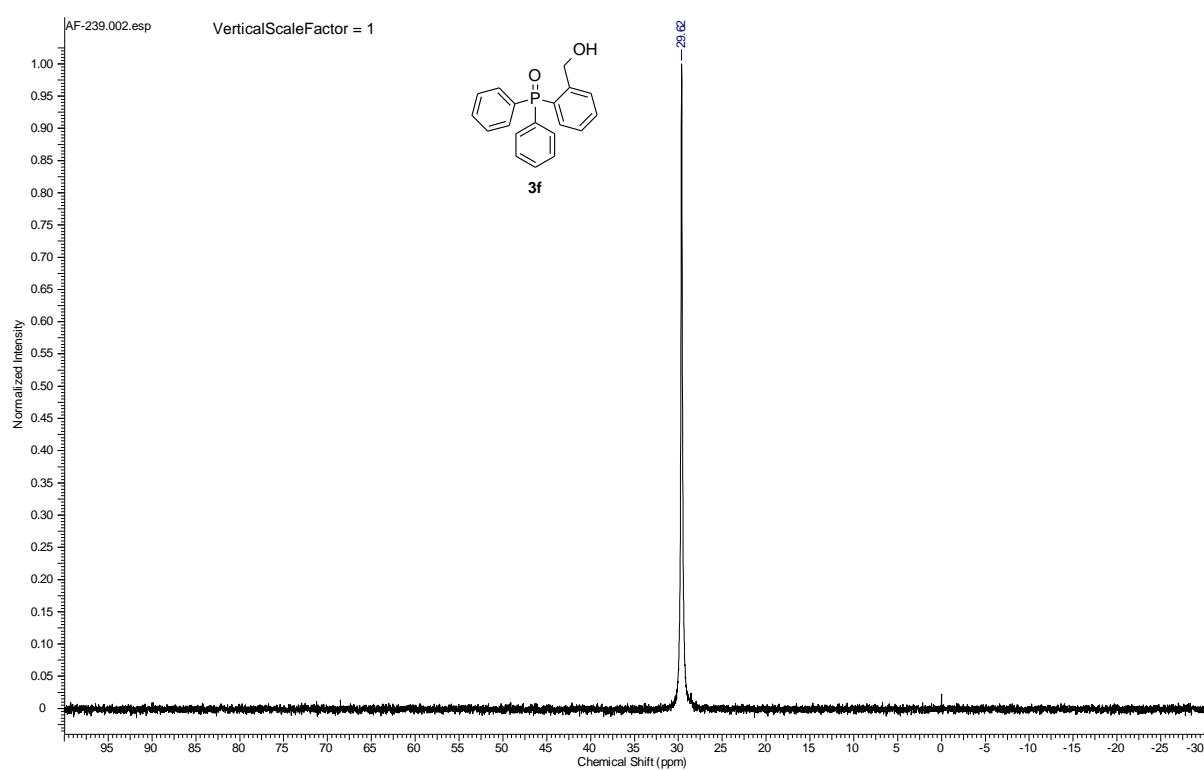
³¹C NMR spectra of biphenyl-4-yl(diphenyl)phosphine oxide (**3e**) (CDCl₃, 126 MHz).



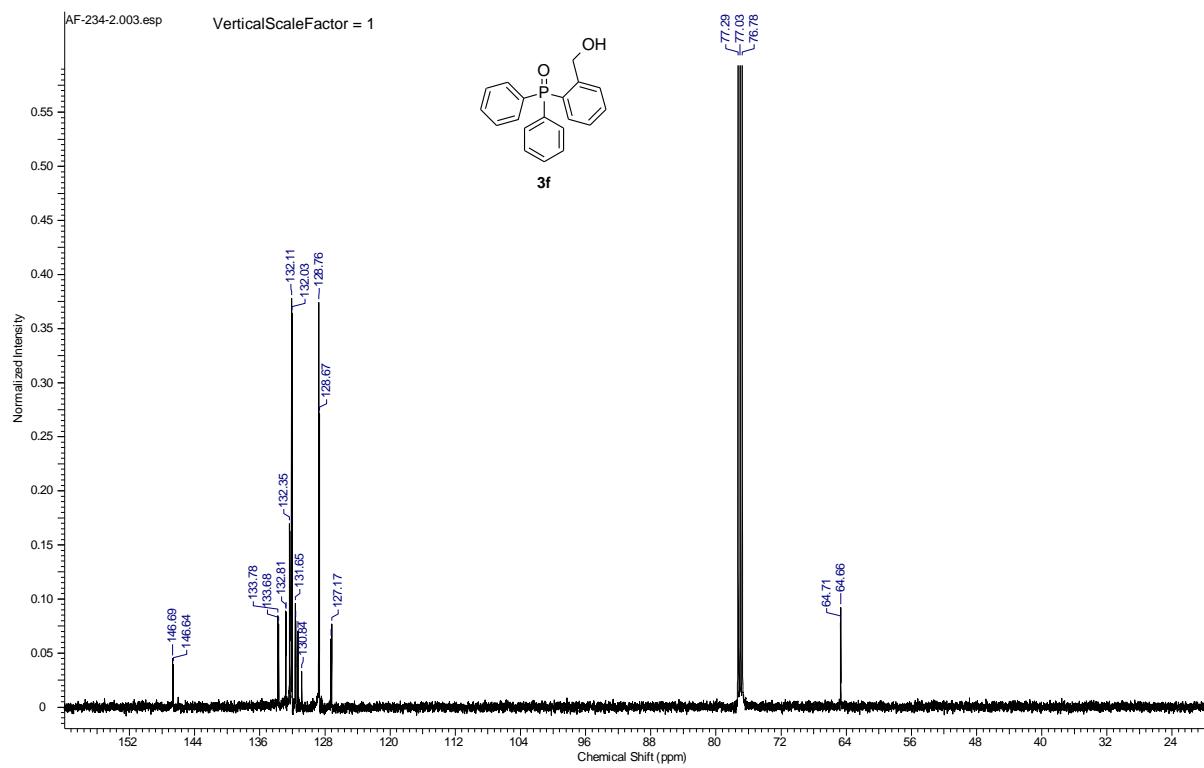
DEPT-135 spectra of bis(biphenyl-4-yl)diphenylphosphine oxide (3a) (CDCl_3 , 126 MHz)



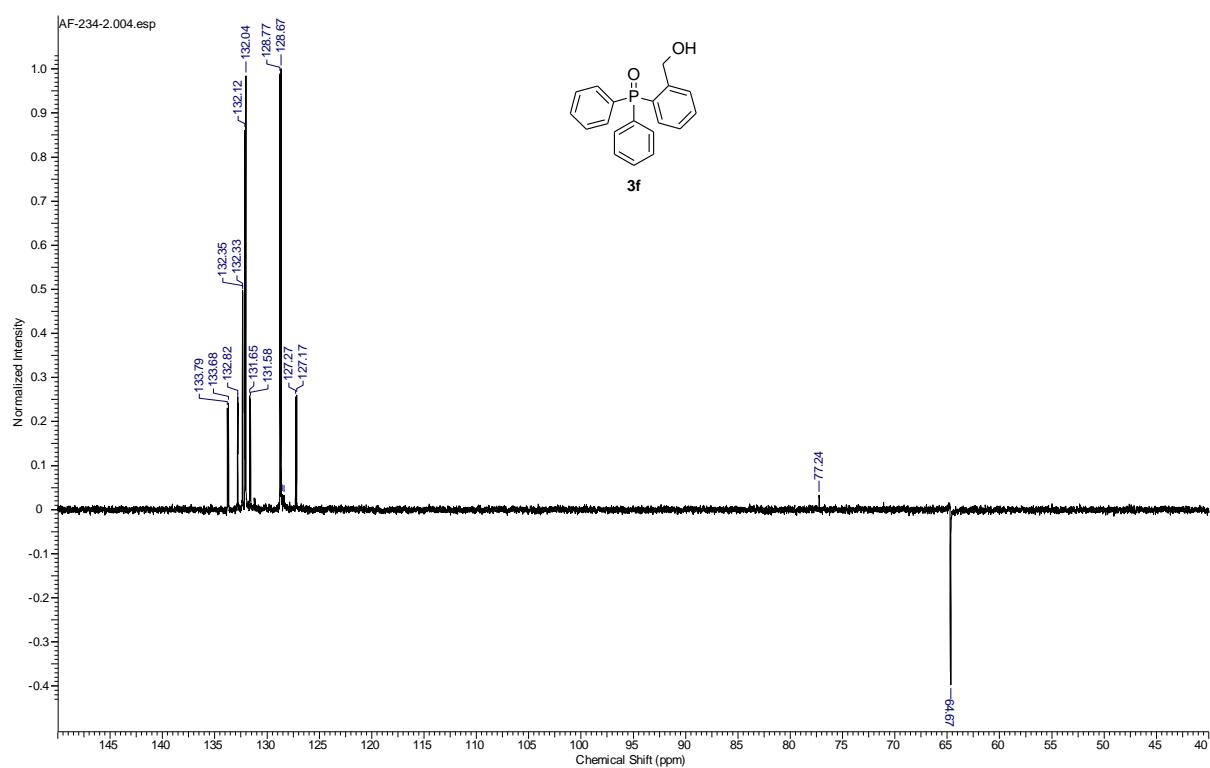
¹H NMR spectra of (2-(hydroxymethyl)phenyl)(diphenyl)phosphine oxide (**3f**) (CDCl₃, 500 MHz).



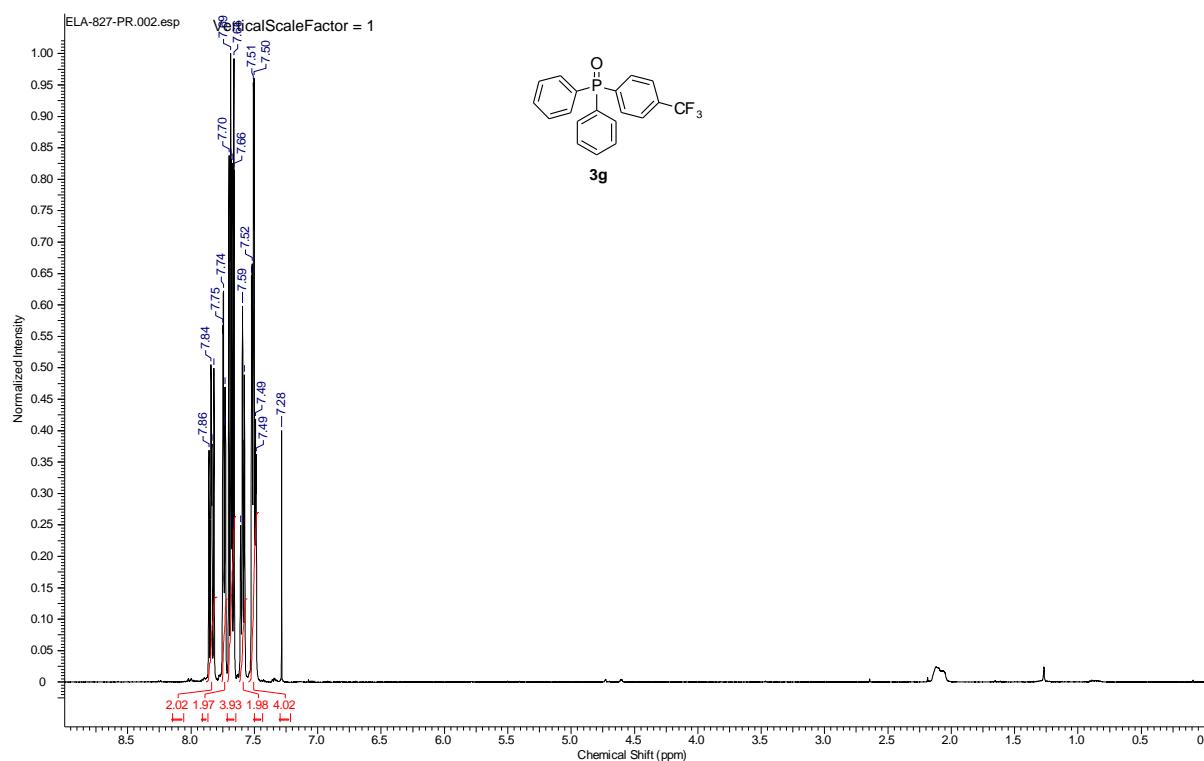
³¹P NMR spectra of (2-(hydroxymethyl)phenyl)(diphenyl)phosphine oxide (**3f**) (CDCl₃, 202 MHz).



³¹C NMR spectra of (2-(hydroxymethyl)phenyl)(diphenyl)phosphine oxide (**3f**) (CDCl₃, 126 MHz).

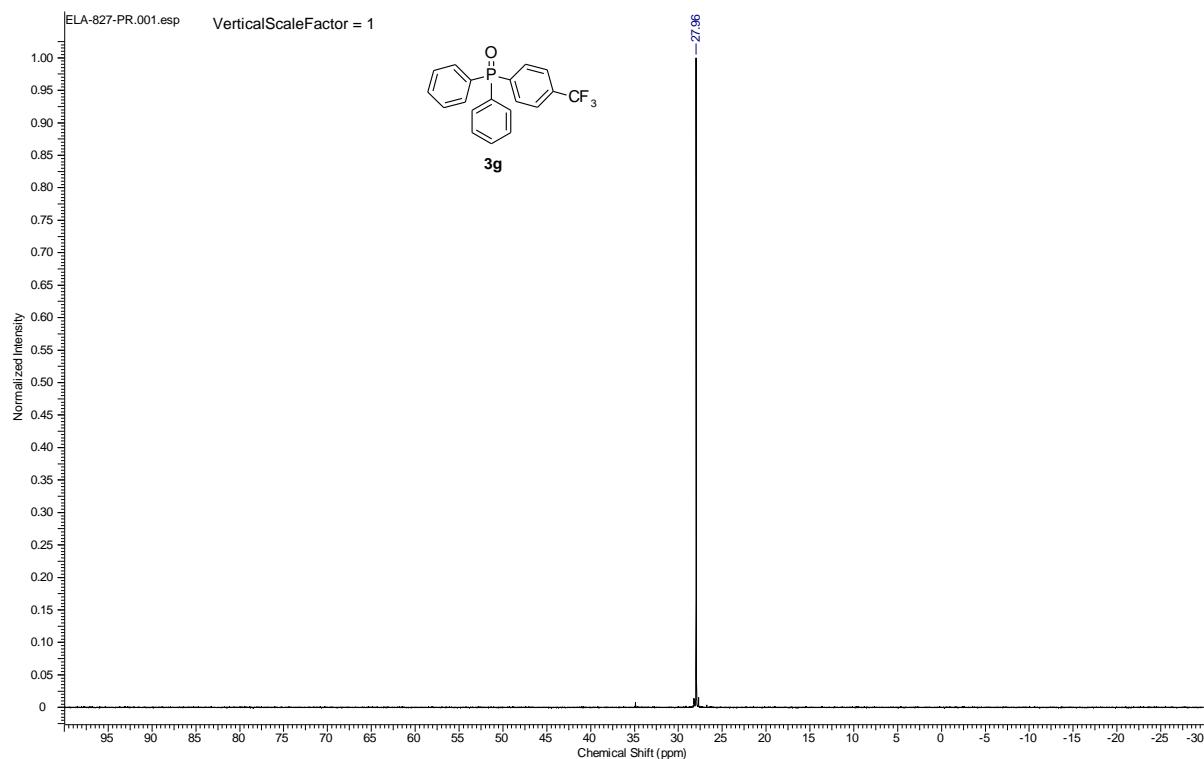


DEPT 135 spectra of (2-(hydroxymethyl)phenyl)(diphenyl)phosphine oxide (**3f**) (CDCl₃, 126 MHz).



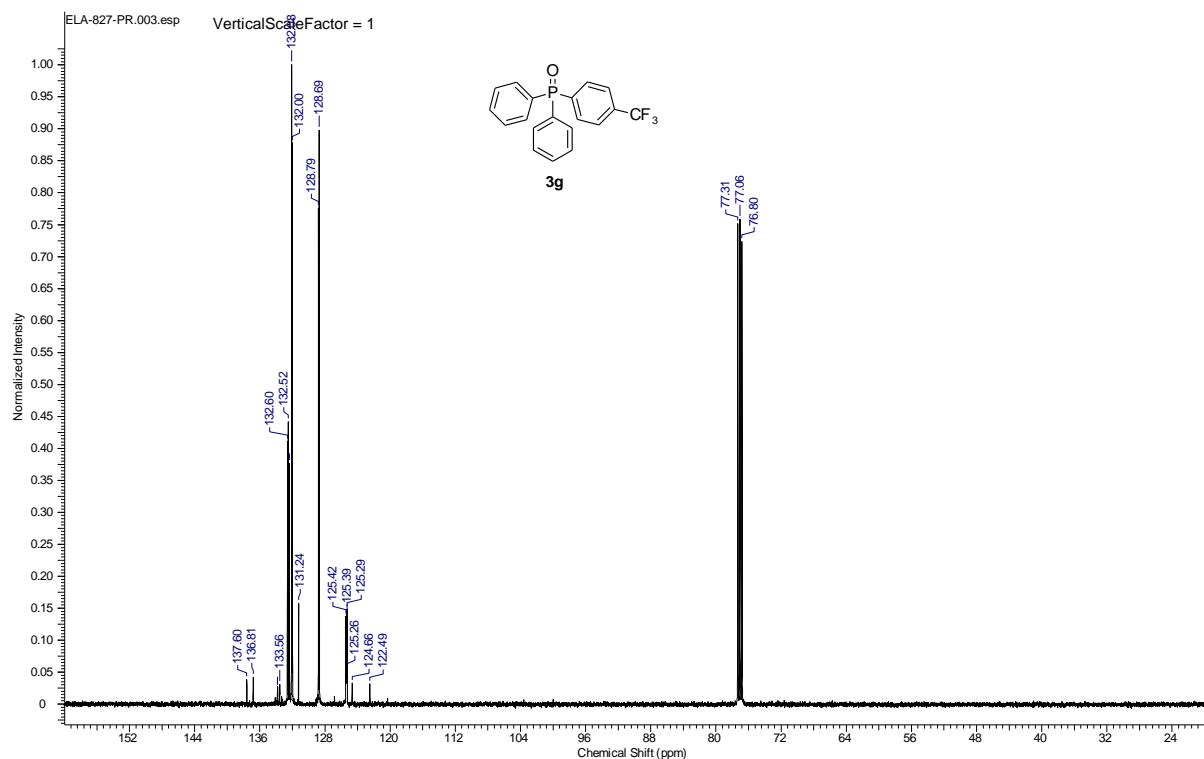
E:\FTP\NMR\ELA LASTAWIECKA\ELA-827-PR\ELA-827-PR.002.esp

¹H NMR spectra of diphenyl[4-(trifluoromethyl)phenyl]phosphine oxide (**3g**) (CDCl₃, 500 MHz).



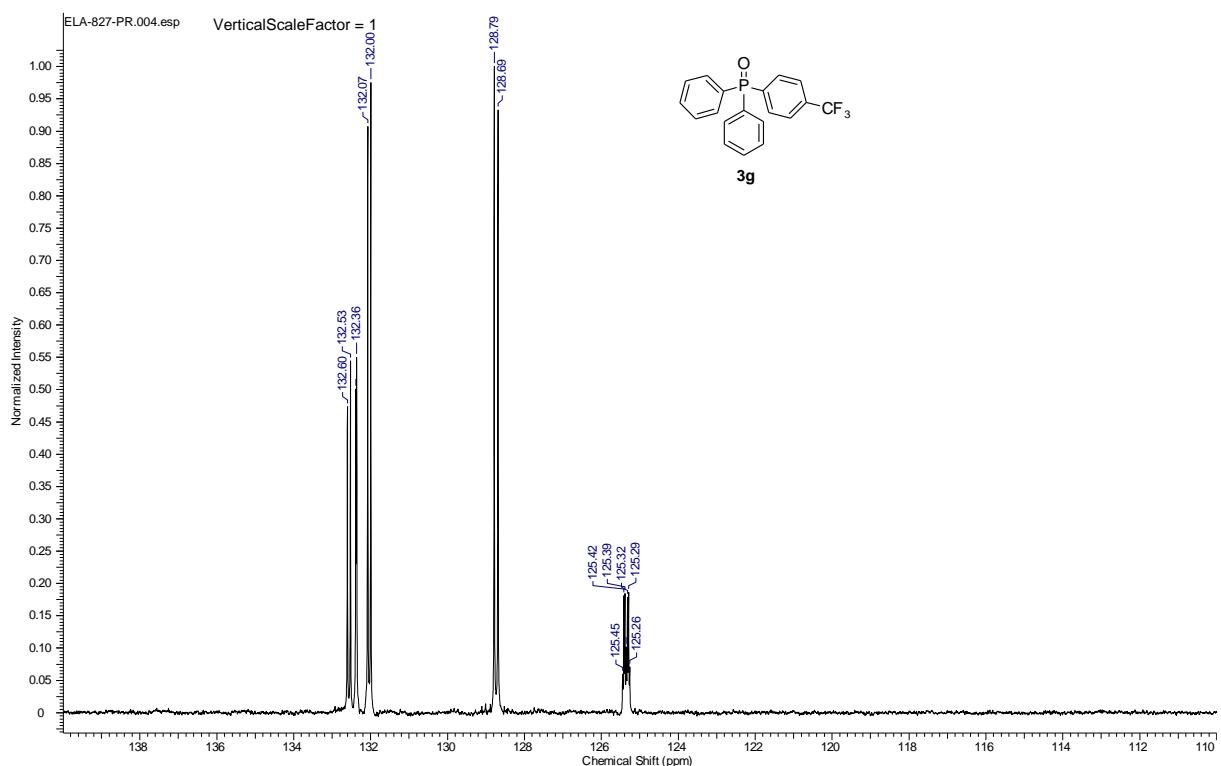
E:\FTP\NMR\ELA LASTAWIECKA\ELA-827-PR\ELA-827-PR.001.esp

³¹P NMR spectra of diphenyl[4-(trifluoromethyl)phenyl]phosphine oxide (**3g**) (CDCl₃, 202 MHz).

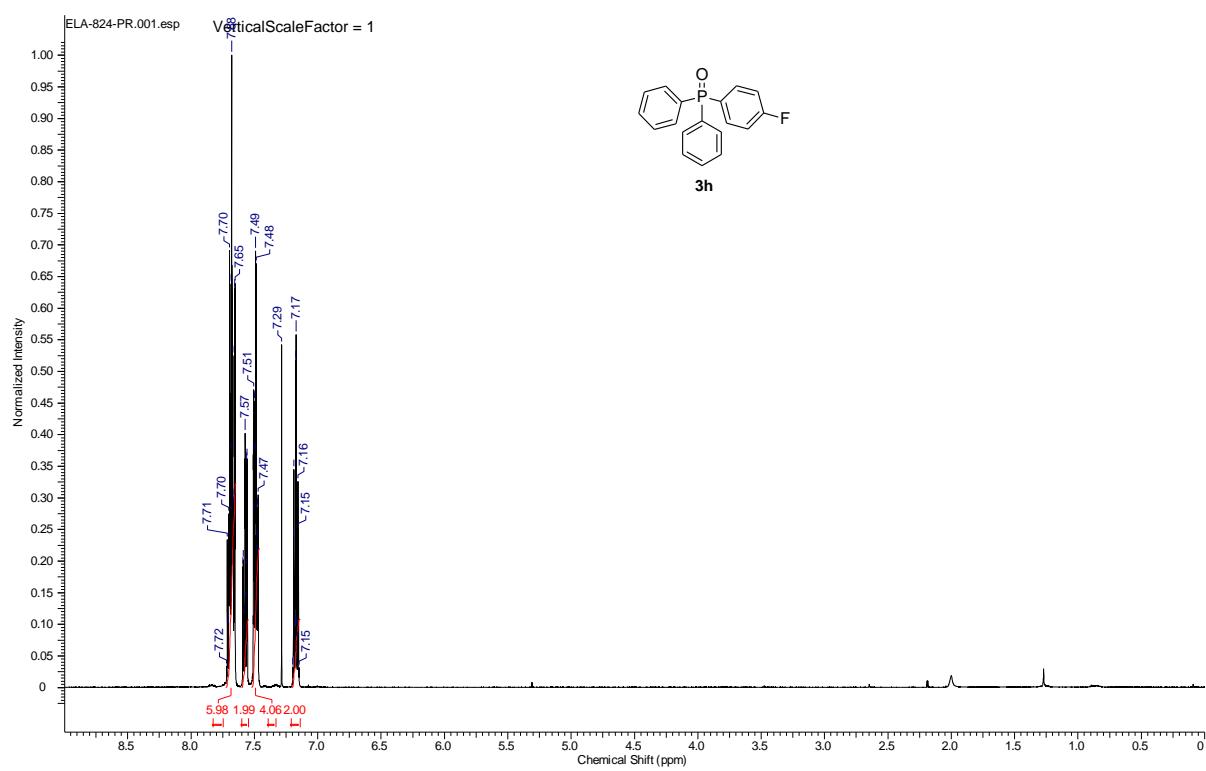


E:\FTP\NMR\ELA LASTAWIECKA\ELA-827-PR\ELA-827-PR.003.esp

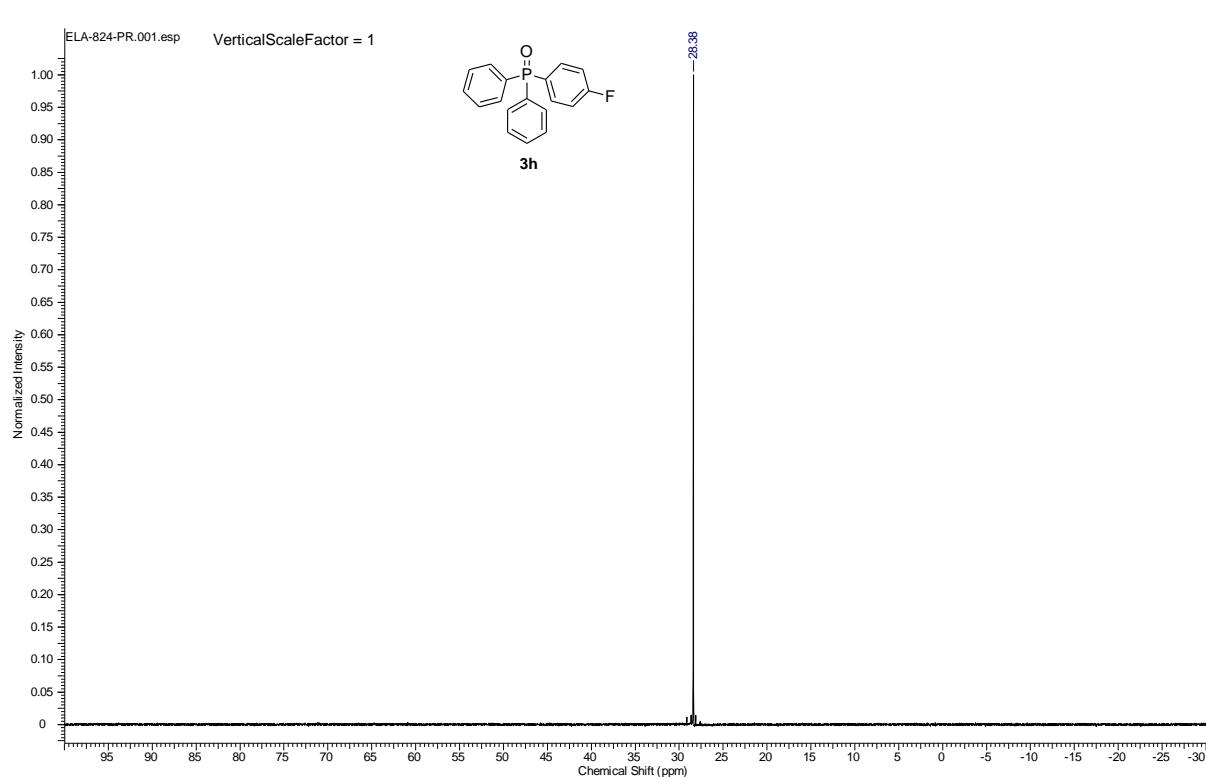
^{31}C NMR spectra of diphenyl[4-(trifluoromethyl)phenyl]phosphine oxide (**3g**) (CDCl_3 , 126 MHz).



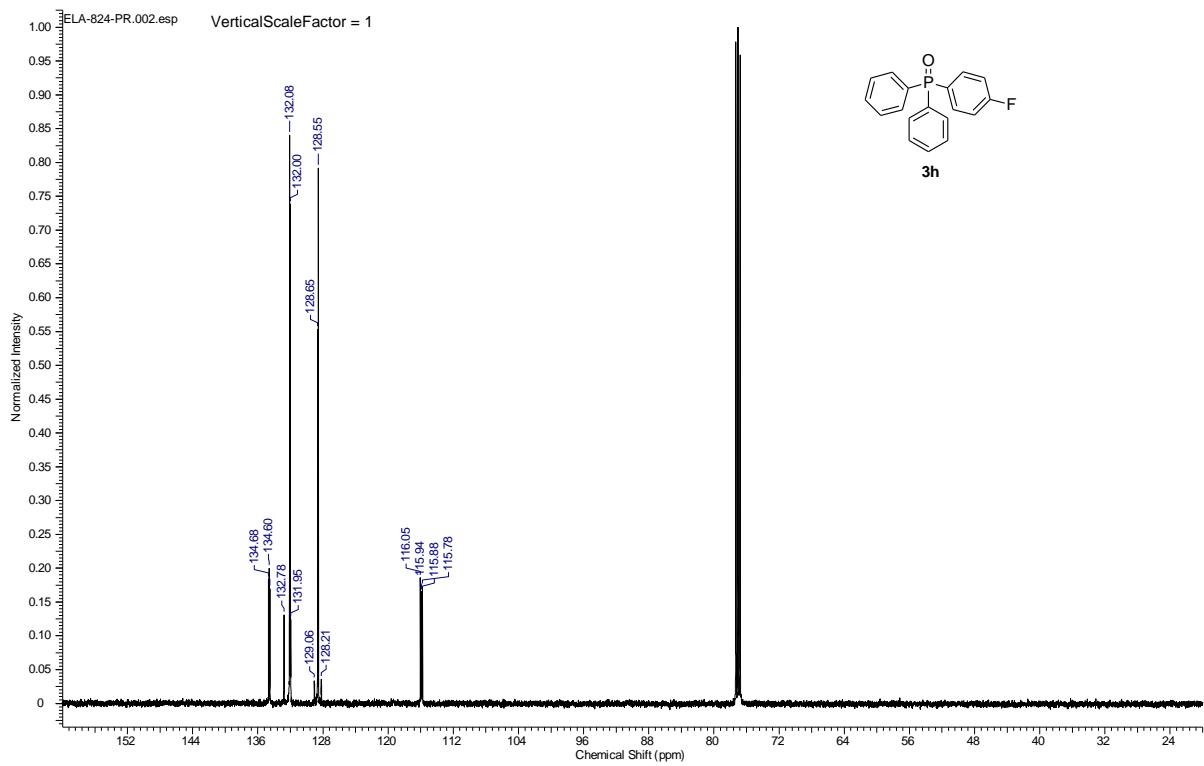
DEPT 135 spectra of diphenyl[4-(trifluoromethyl)phenyl]phosphine oxide (**3g**) (CDCl_3 , 126 MHz).



¹H NMR spectra of 4-fluorophenyl(diphenyl)phosphine oxide (**3h**) (CDCl₃, 500 MHz).

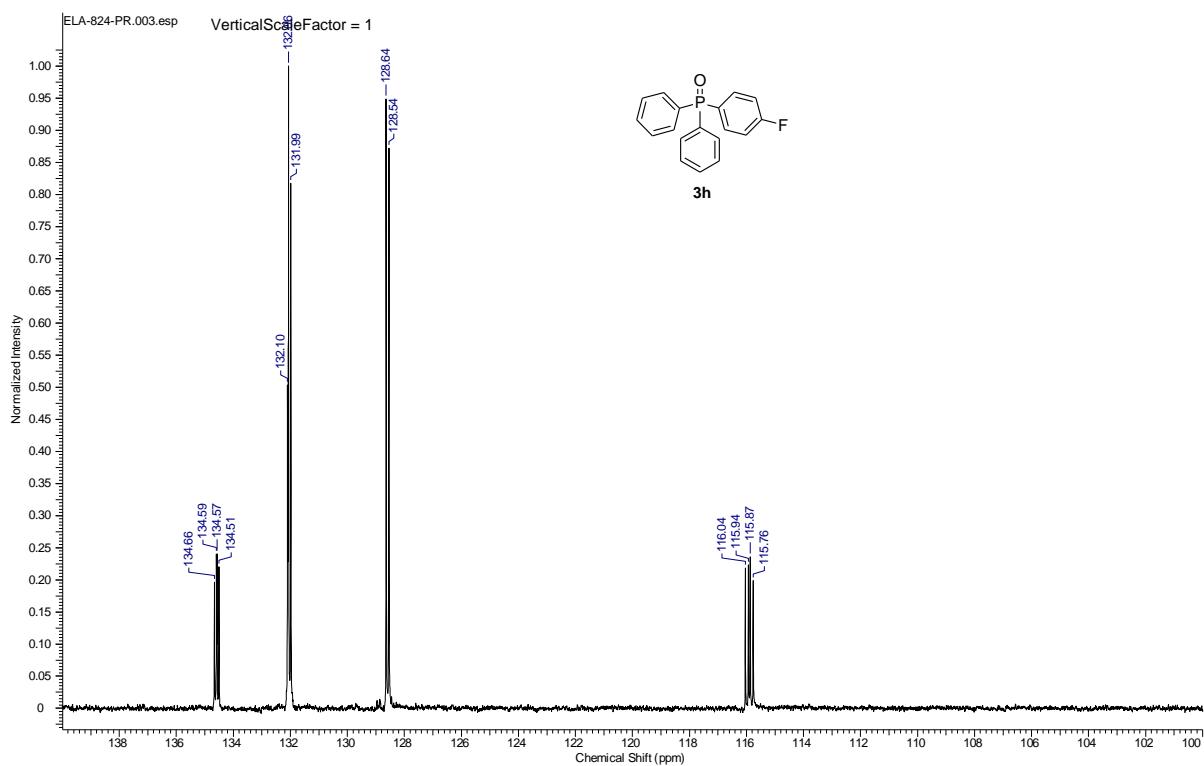


³¹P NMR spectra of 4-fluorophenyl(diphenyl)phosphine oxide (**3h**) (CDCl₃, 202 MHz).



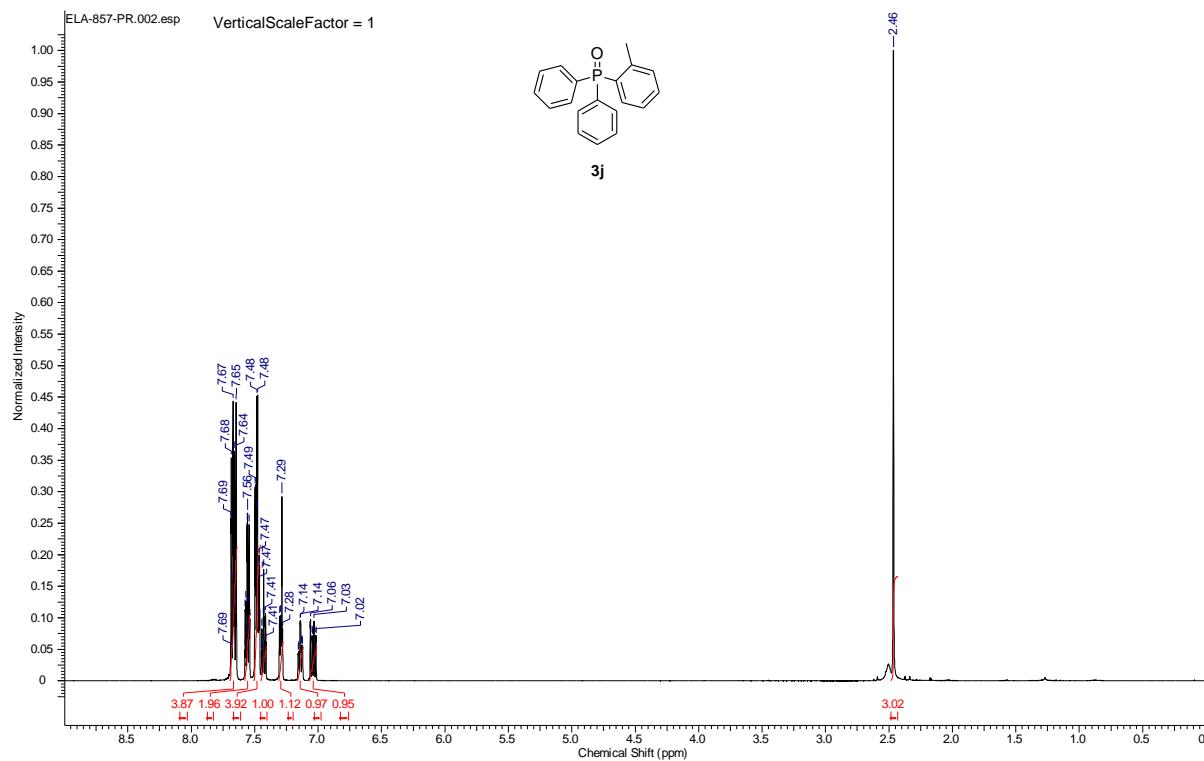
G:\NI-SPRZeGANIA\SI\ELA-824-PR\ELA-824-PR.002.esp

³¹C NMR spectra of 4-fluorophenyl(diphenyl)phosphine oxide (**3h**) (CDCl₃, 126 MHz).



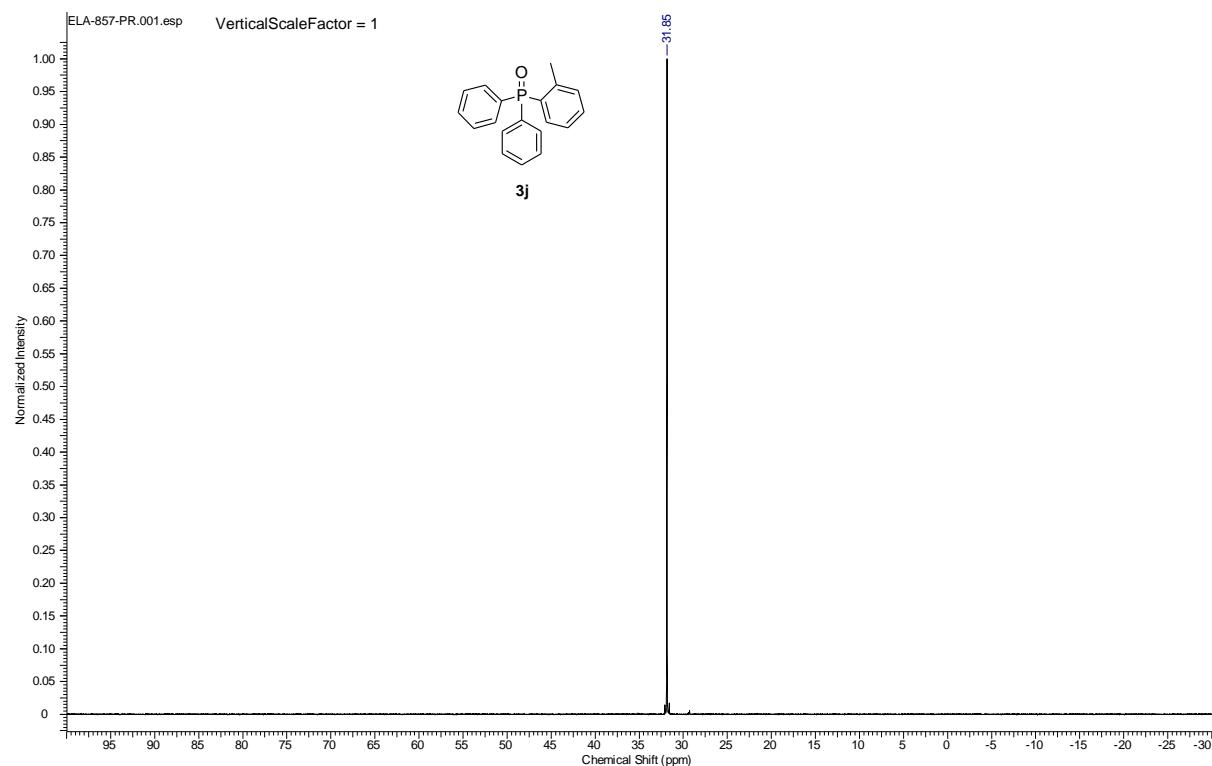
G:\NI-SPRZeGANIA\SI\ELA-824-PR\ELA-824-PR.003.esp

DEPT 135 spectra of 4-fluorophenyl(diphenyl)phosphine oxide (**3h**) (CDCl₃, 126 MHz).

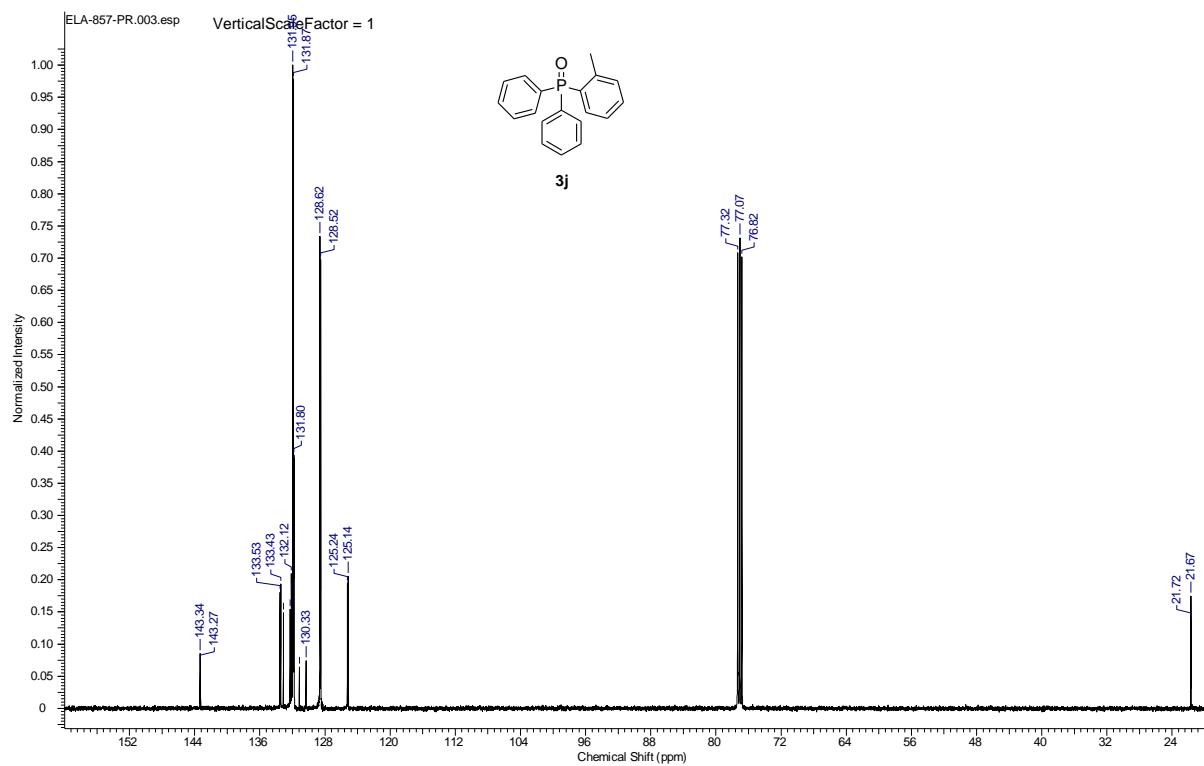


G:\NI-SPRZ\GANIA\SI\ELA-857-PR\ELA-857-PR.002.esp

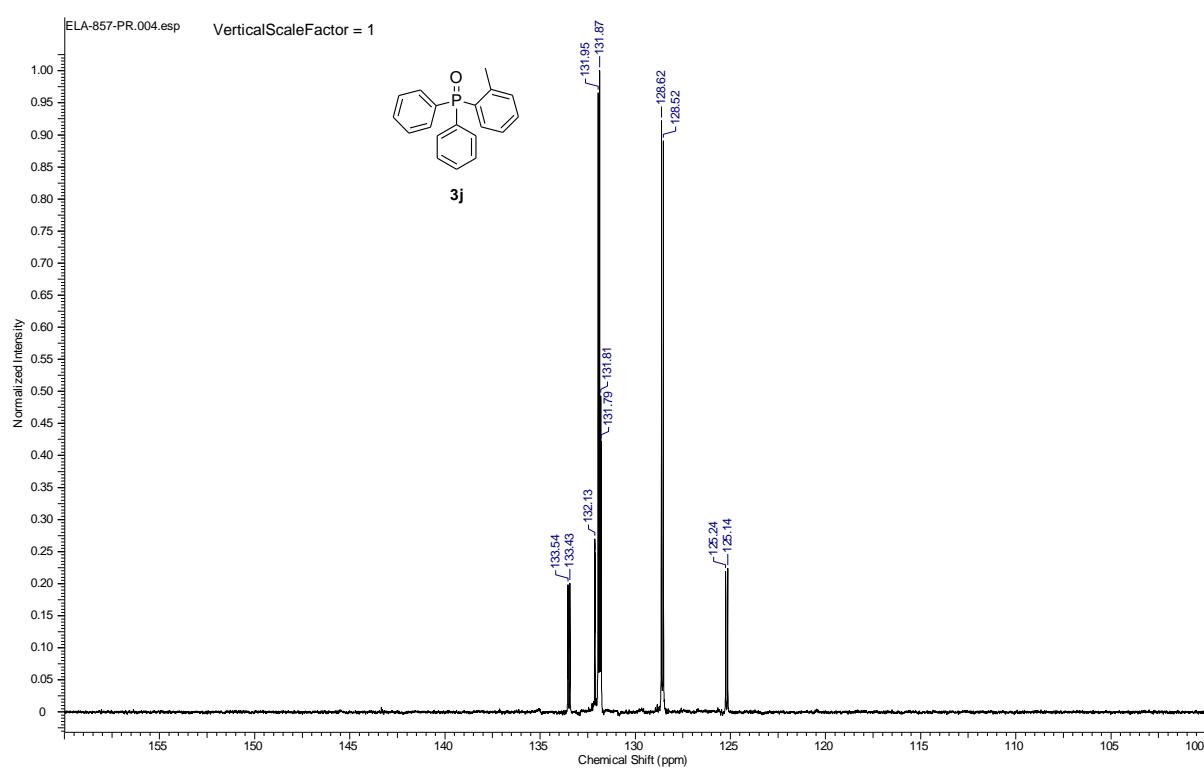
¹H NMR spectra of diphenyl(*o*-tolyl)phosphine oxide (**3j**) (CDCl₃, 500 MHz).



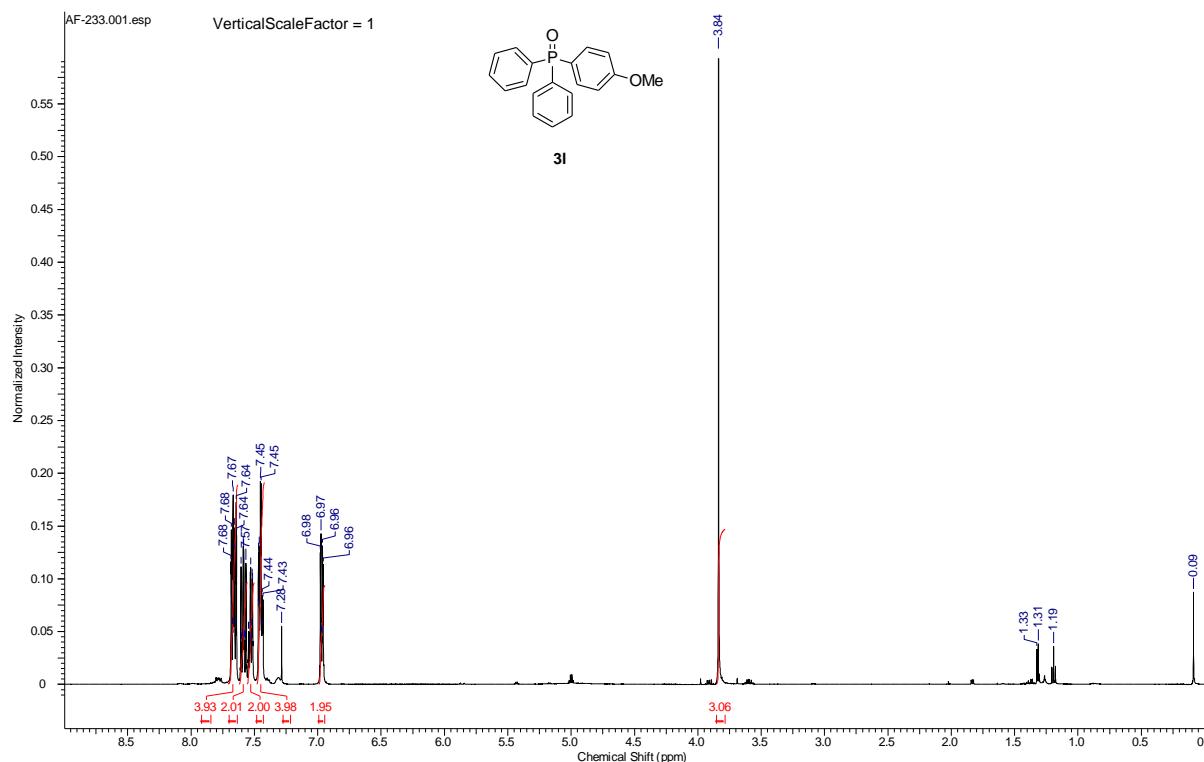
³¹P NMR spectra of diphenyl(*o*-tolyl)phosphine oxide (**3j**) (CDCl₃, 202 MHz).



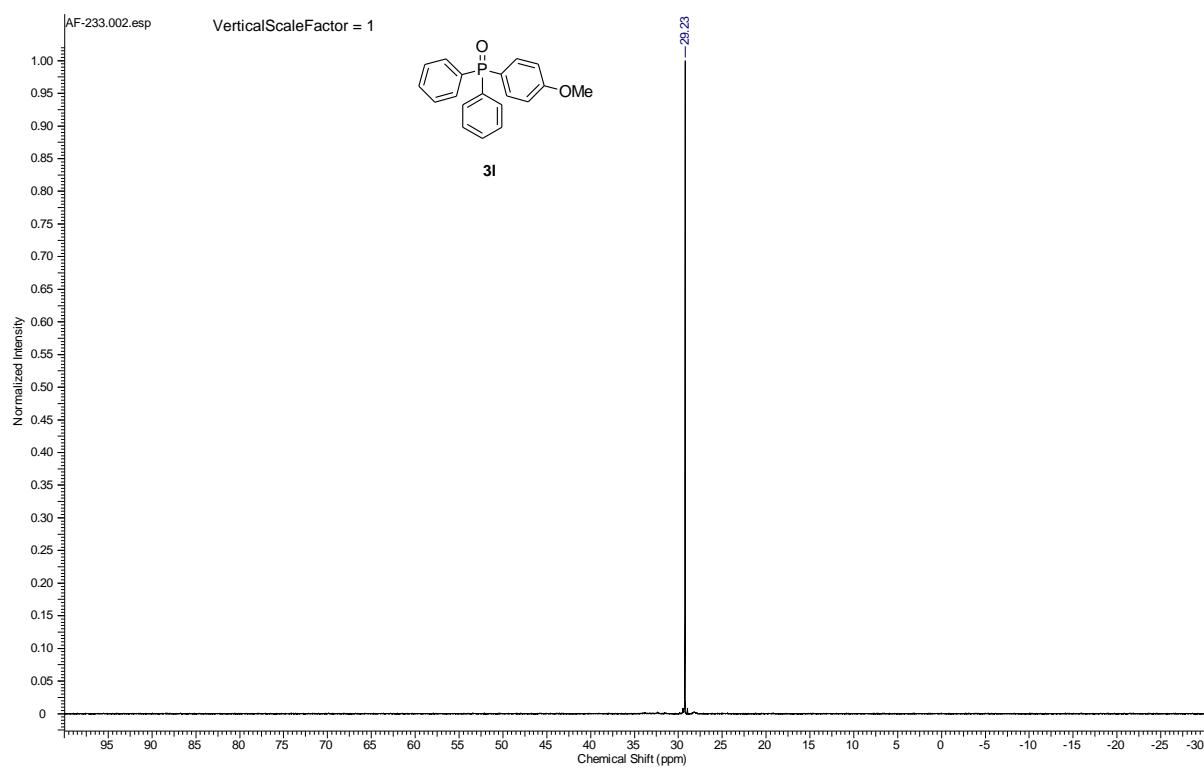
^{31}C NMR spectra of diphenyl(*o*-tolyl)phosphine oxide (**3j**) (CDCl_3 , 126 MHz).



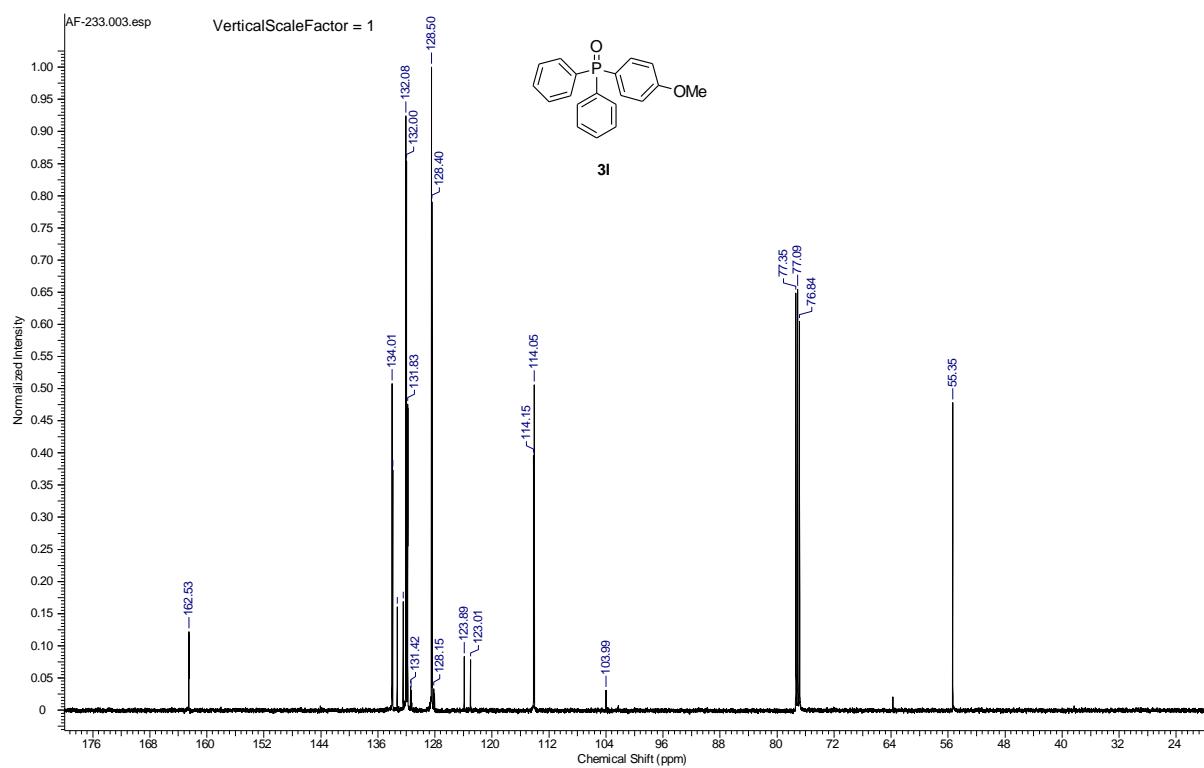
DEPT 135 spectra of diphenyl(*o*-tolyl)phosphine oxide (**3j**) (CDCl_3 , 126 MHz).



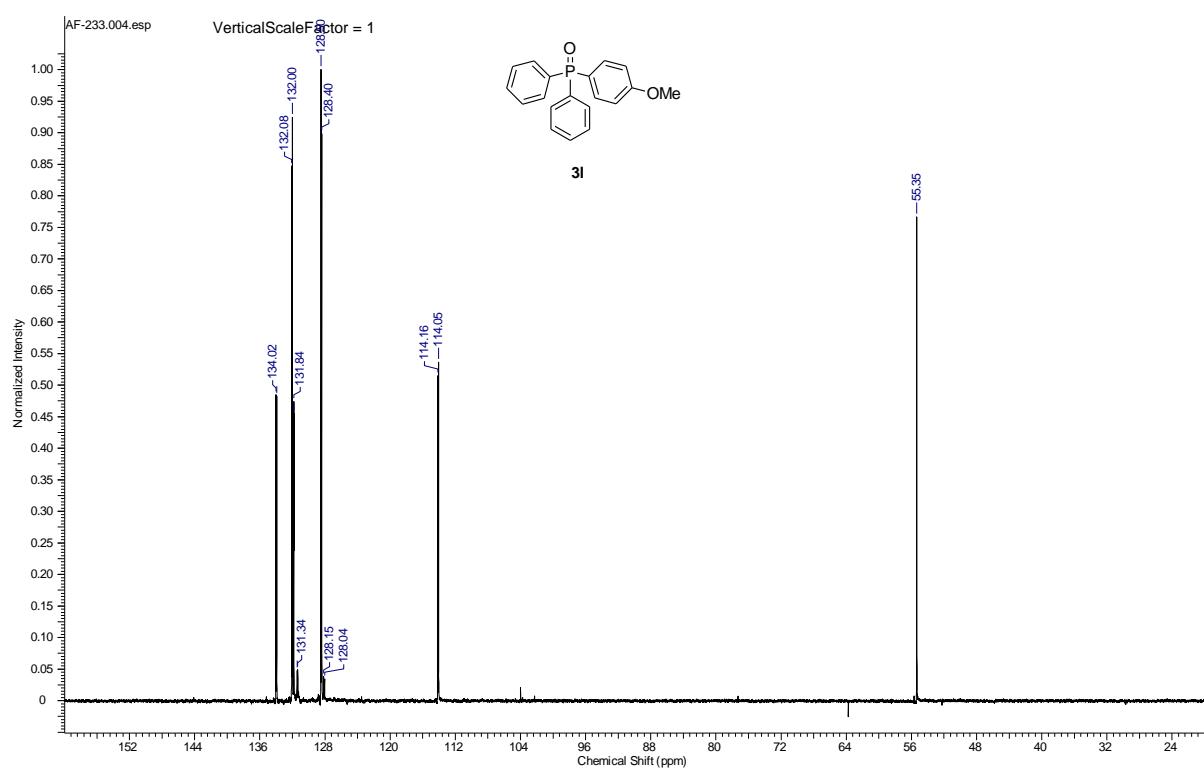
¹H NMR spectra of (4-methoxyphenyl)diphenylphosphine oxide (**3I**) (CDCl_3 , 500 MHz).



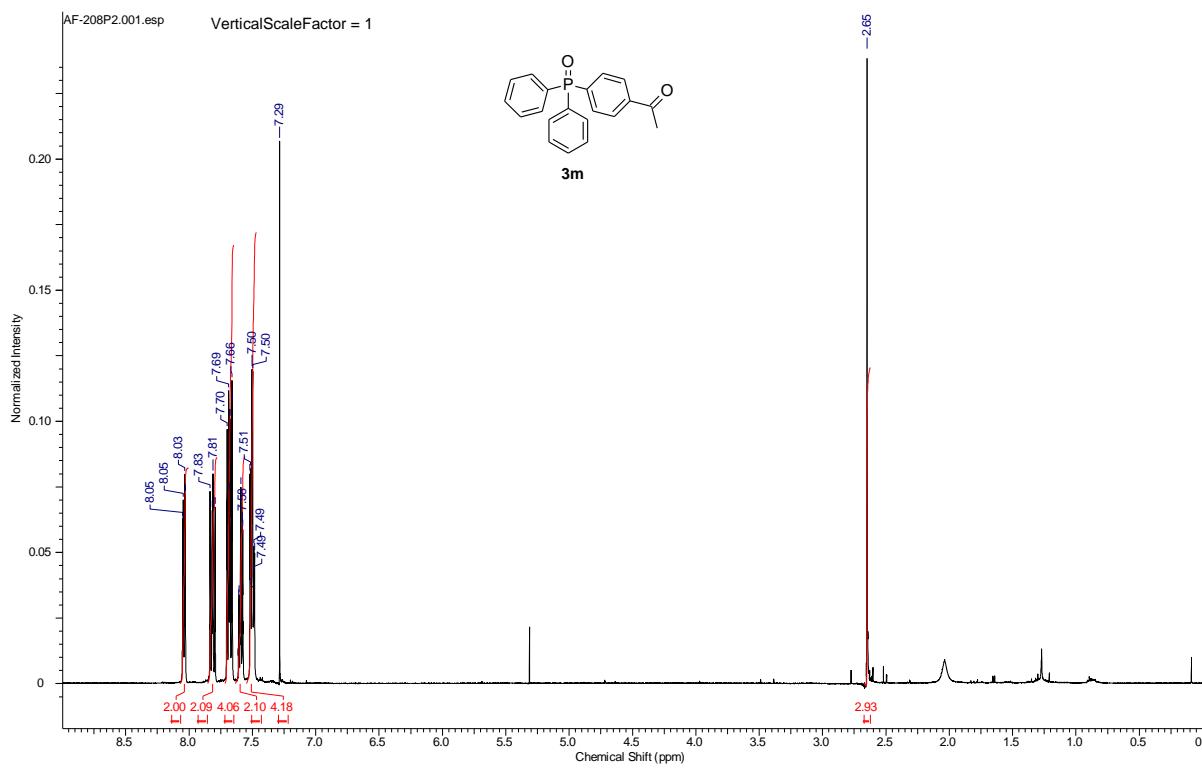
³¹P NMR spectra of (4-methoxyphenyl)diphenylphosphine oxide (**3I**) (CDCl_3 , 202 MHz).



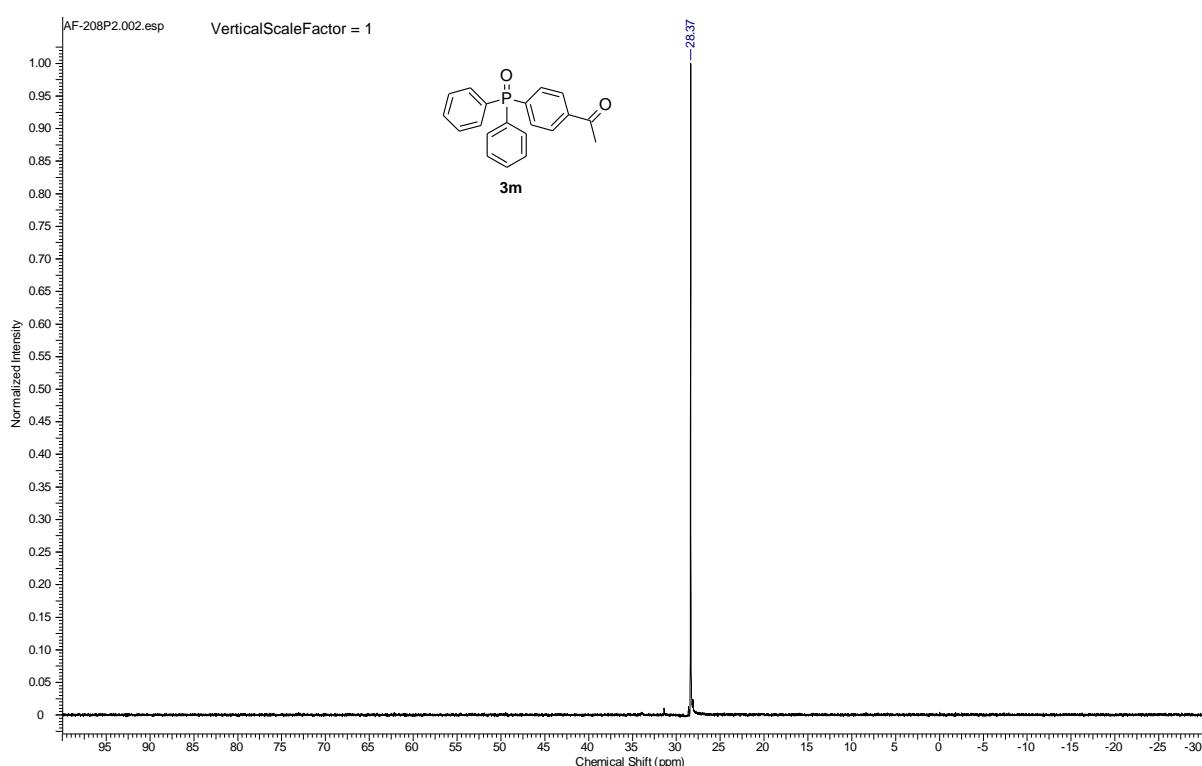
^{13}C NMR spectra of (4-methoxyphenyl)diphenylphosphine oxide (**3I**) (CDCl_3 , 126 MHz).



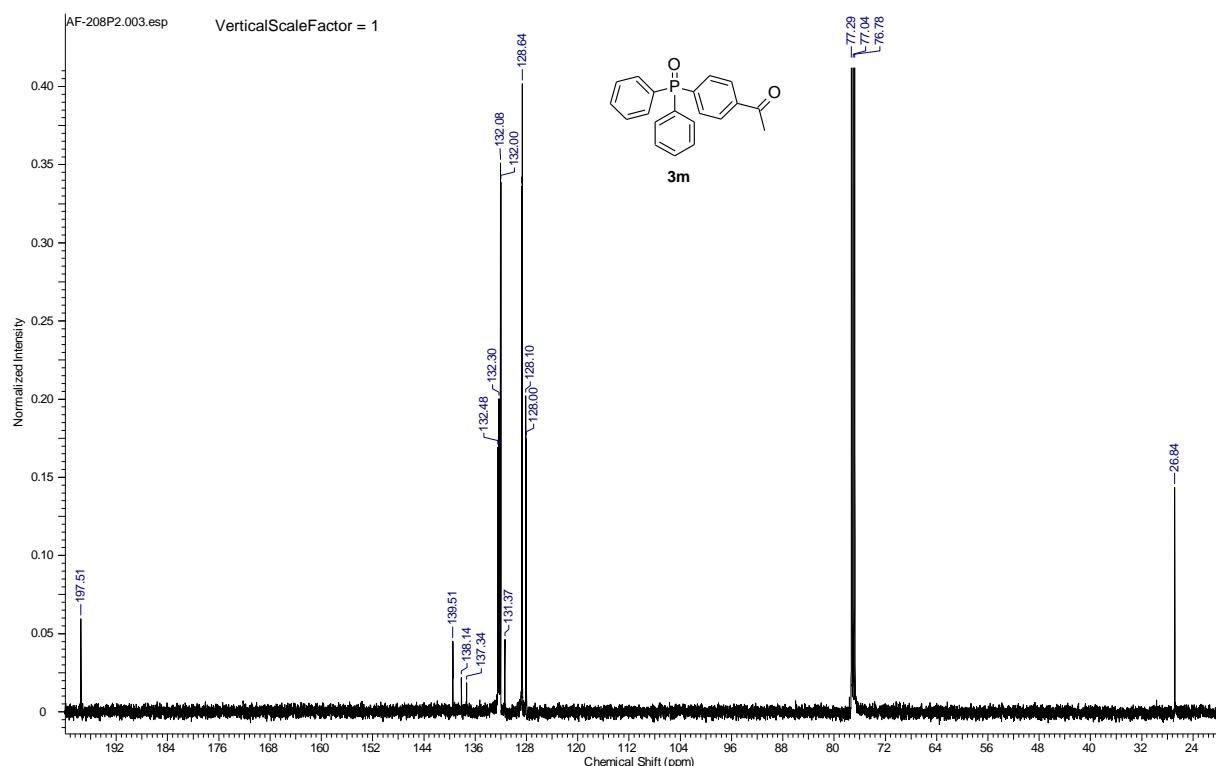
DEPT 135 spectra of (4-methoxyphenyl)diphenylphosphine oxide (**3I**) (CDCl_3 , 126 MHz).



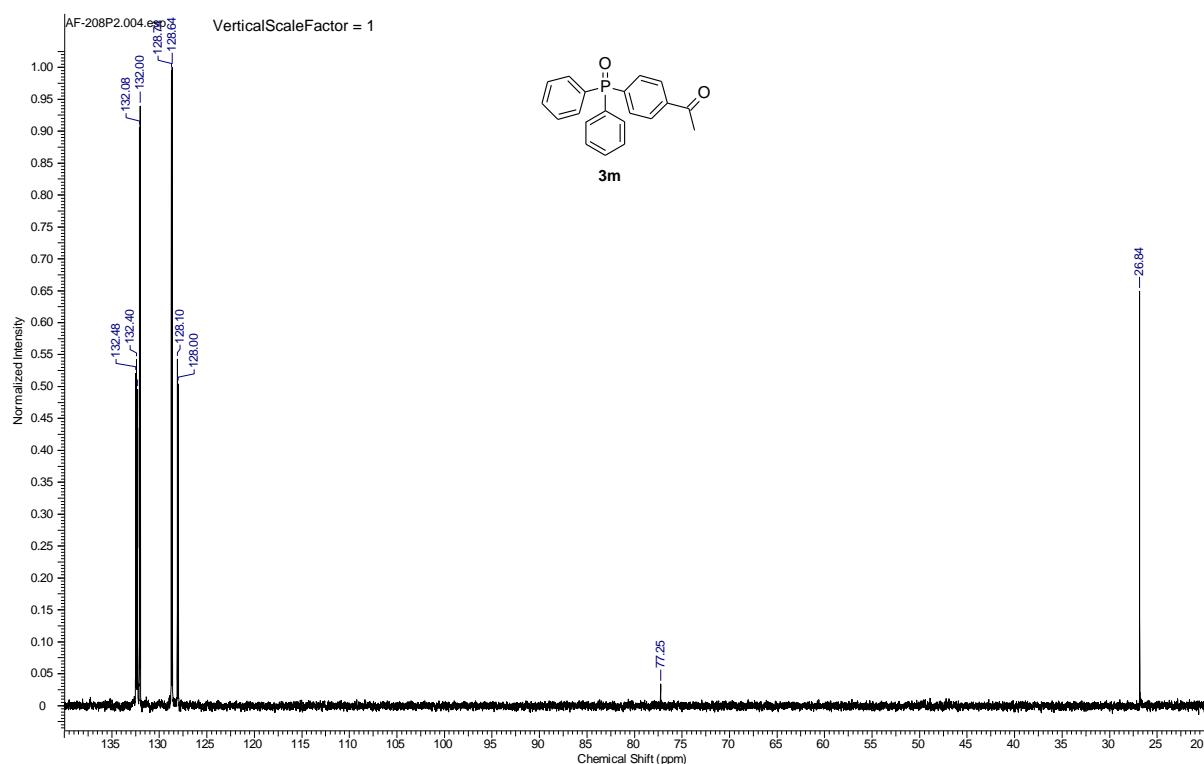
¹H NMR spectra of bis1-(4-(diphenylphosphoryl)phenyl)ethan-1-one (**3m**) (CDCl₃, 500 MHz).



³¹P NMR spectra of 1-(4-(diphenylphosphoryl)phenyl)ethan-1-one (**3m**) (CDCl₃, 202 MHz).

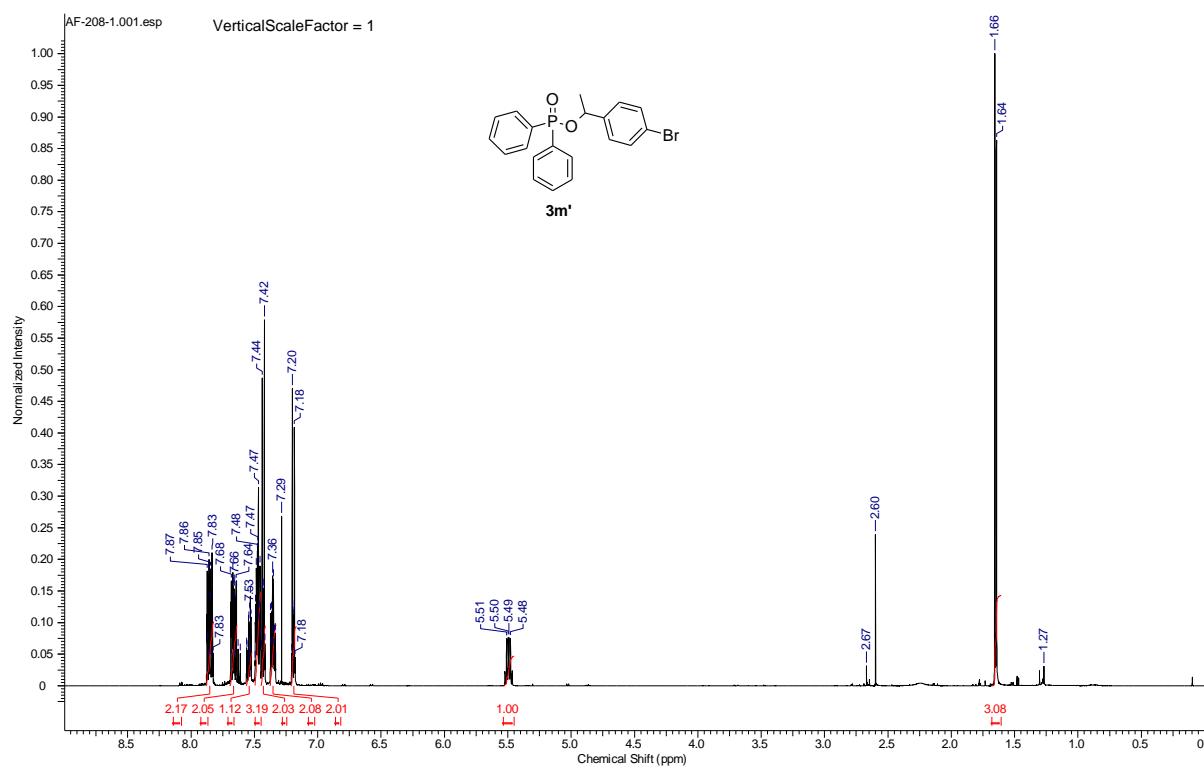


^{31}C NMR spectra of 1-(4-(diphenylphosphoryl)phenyl)ethan-1-one (**3m**) (CDCl_3 , 126 MHz).

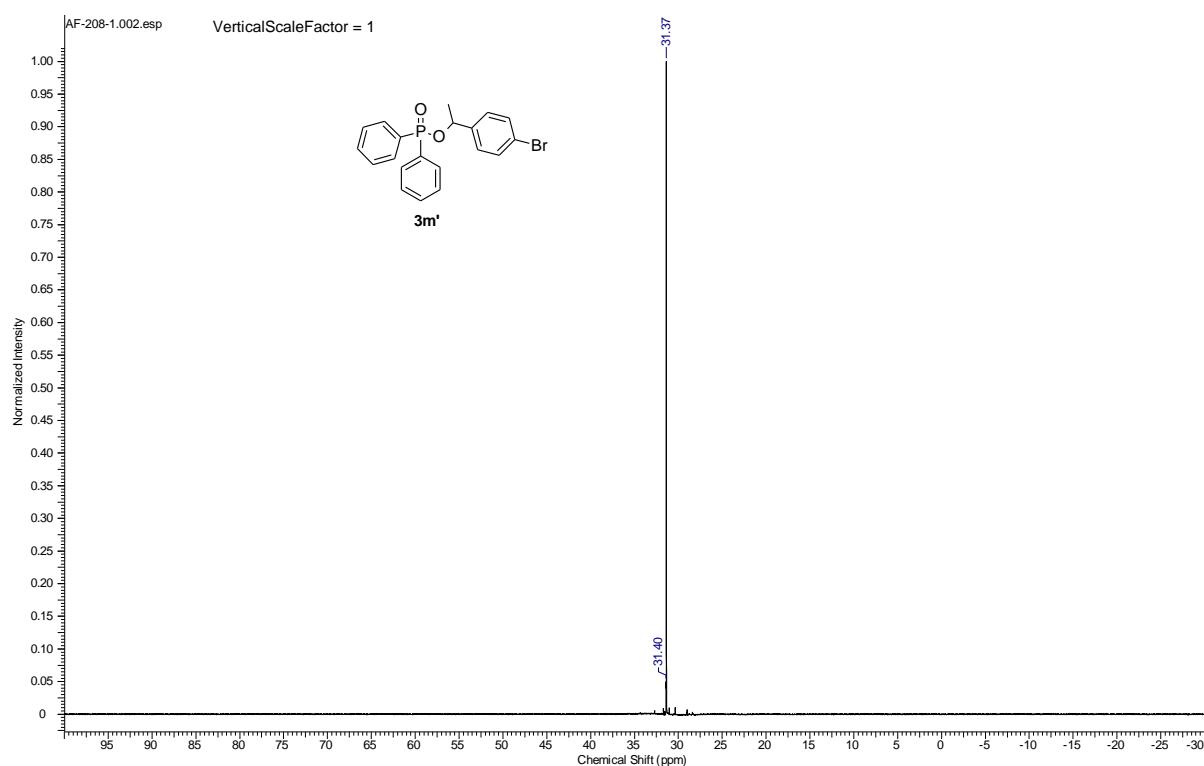


G:\NI\SPRZ\GANIA\S\AF-208P2\AF-208P2.004.esp

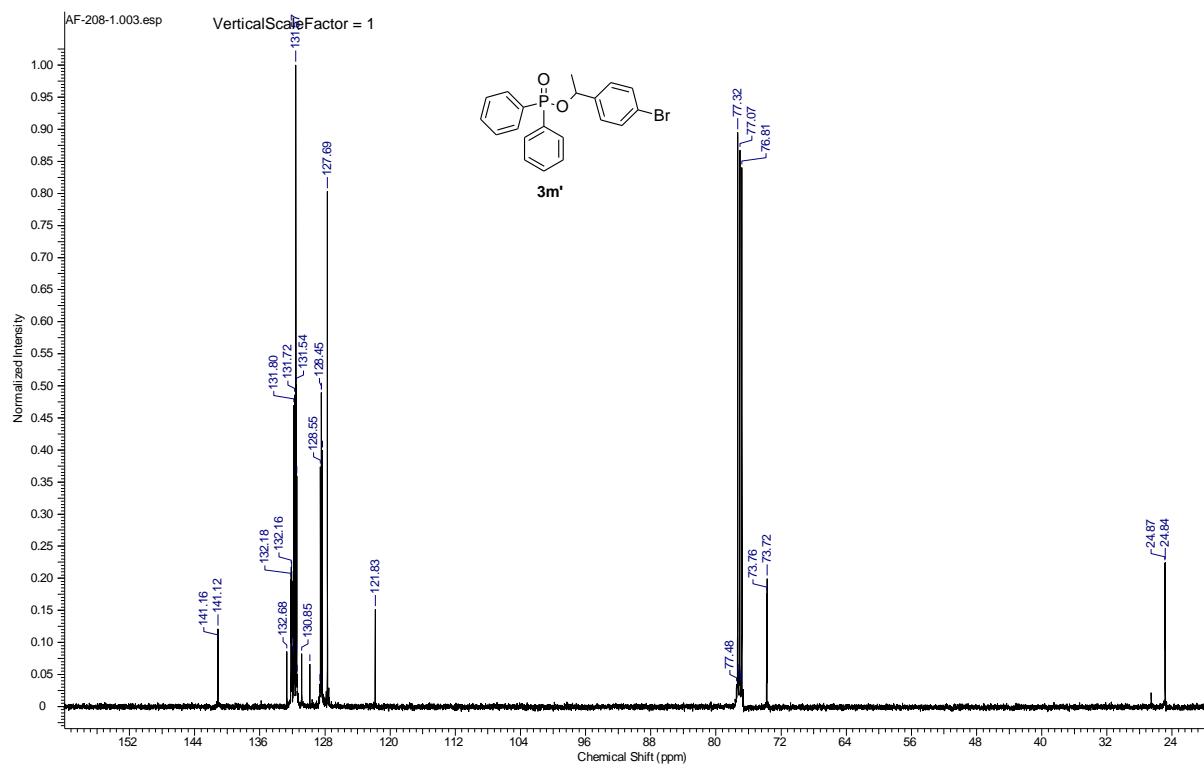
DEPT 135 spectra of 1-(4-(diphenylphosphoryl)phenyl)ethan-1-one (**3m**) (CDCl_3 , 126 MHz).



¹H NMR spectra of 1-(4-bromophenyl)ethyl diphenylphosphinate (**3m'**) (CDCl₃, 500 MHz).

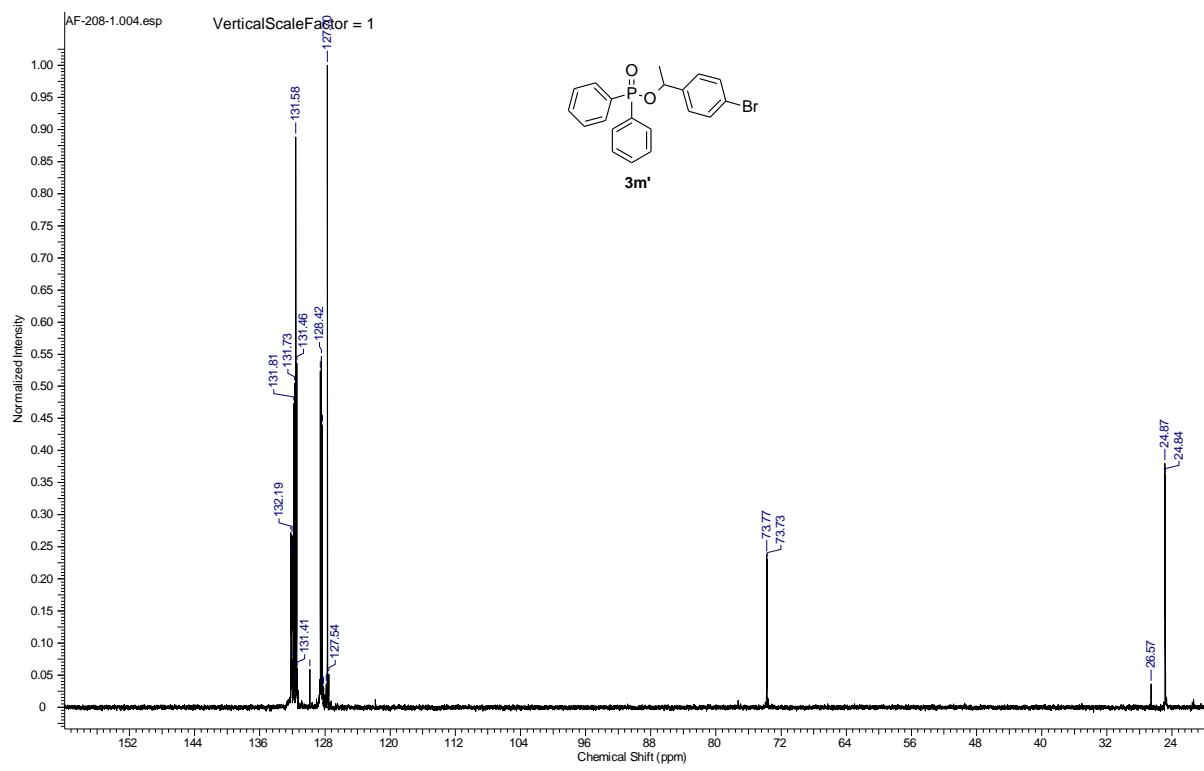


³¹P NMR spectra of 1-(4-bromophenyl)ethyl diphenylphosphinate (**3m'**) (CDCl₃, 202 MHz).



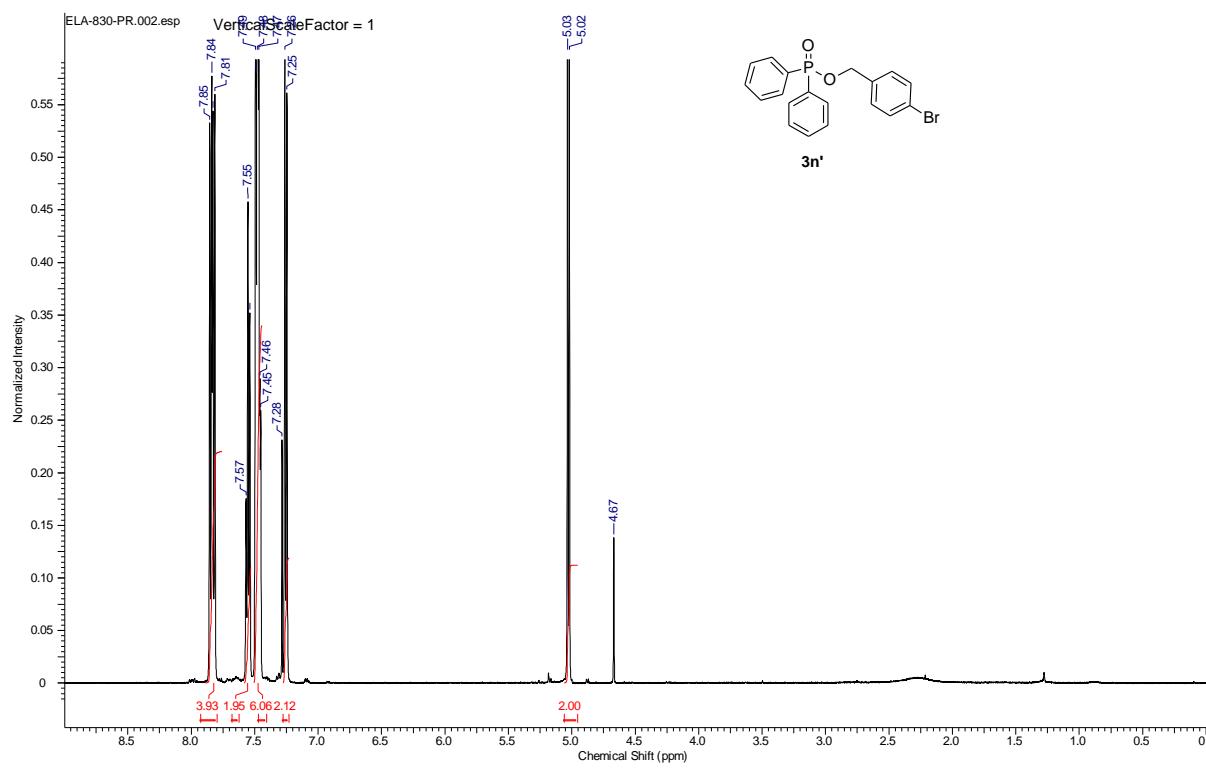
E:\FTP\NMR\ELA LASTAWIECKA\AF-208\1AF-208-1.003.esp

³¹C NMR spectra of 1-(4-bromophenyl)ethyl diphenylphosphinate (**3m'**) (CDCl₃, 126 MHz).

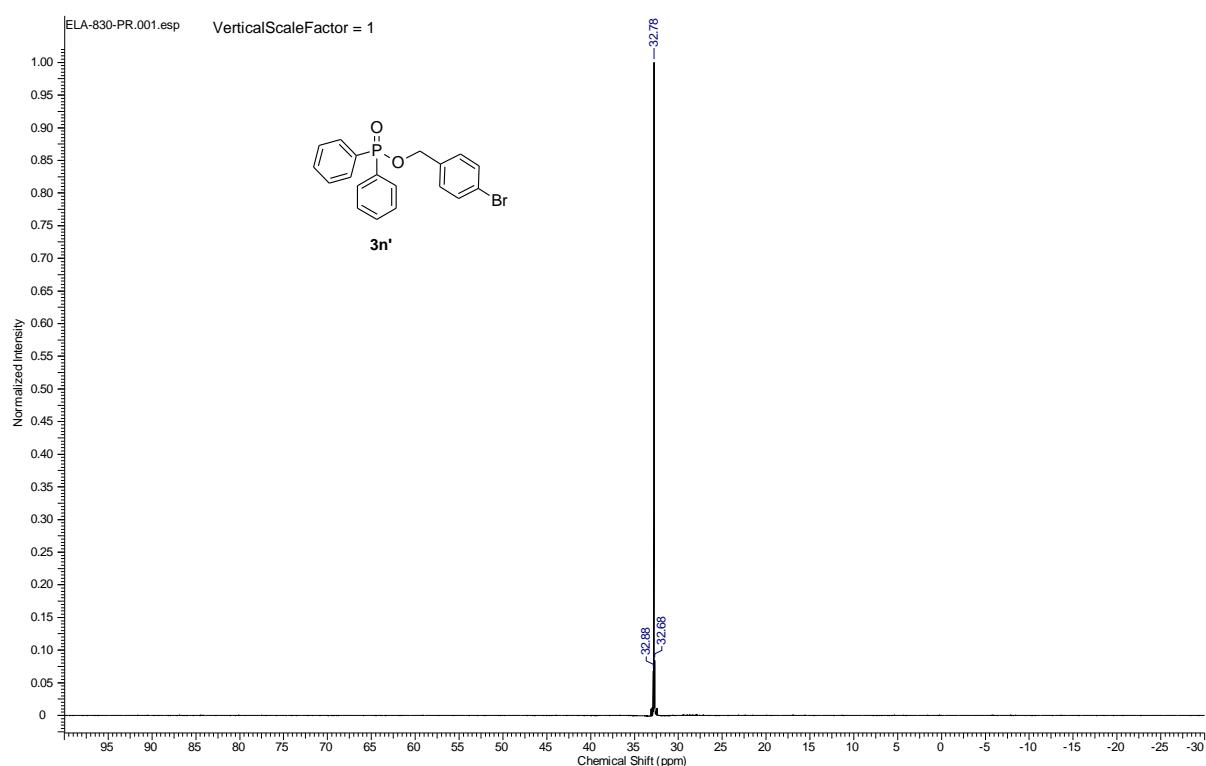


E:\FTP\NMR\ELA LASTAWIECKA\AF-208\1AF-208-1.004.esp

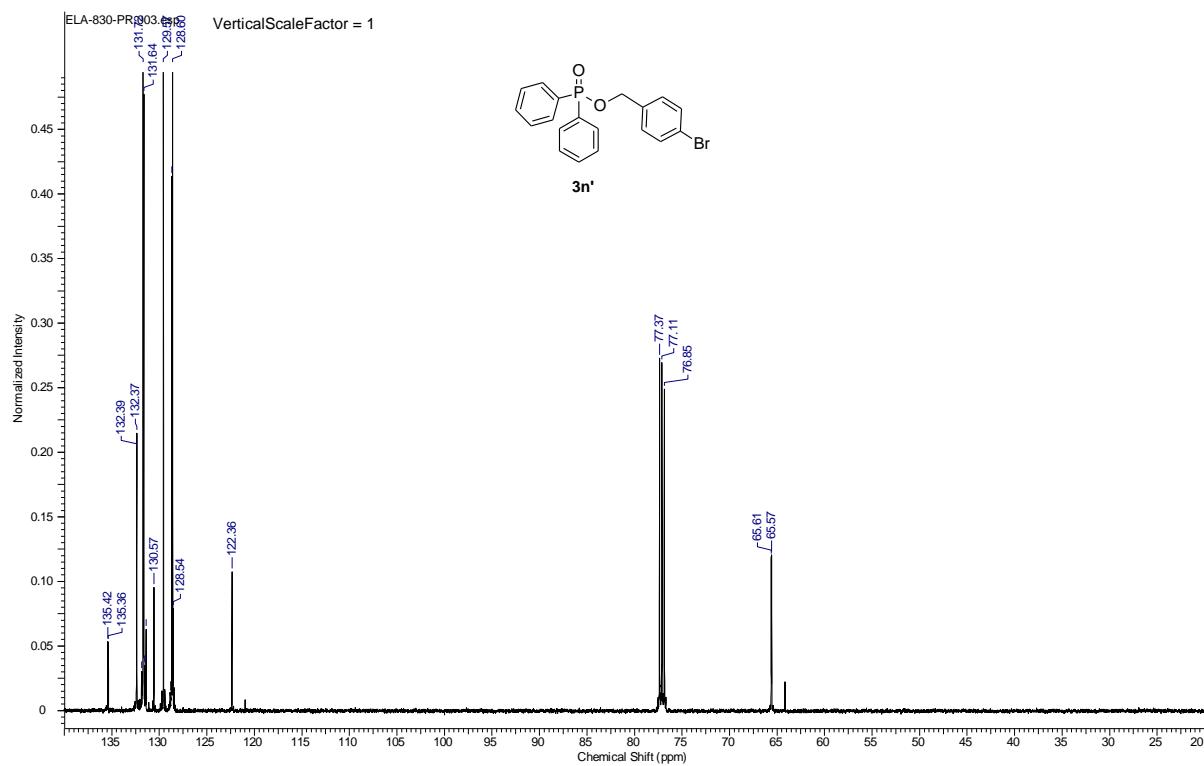
DEPT 135 spectra of 1-(4-bromophenyl)ethyl diphenylphosphinate (**3m'**) (CDCl₃, 126 MHz).



¹H NMR spectra of 4-bromobenzyl diphenylphosphinate (**3n'**) (CDCl₃, 500 MHz).

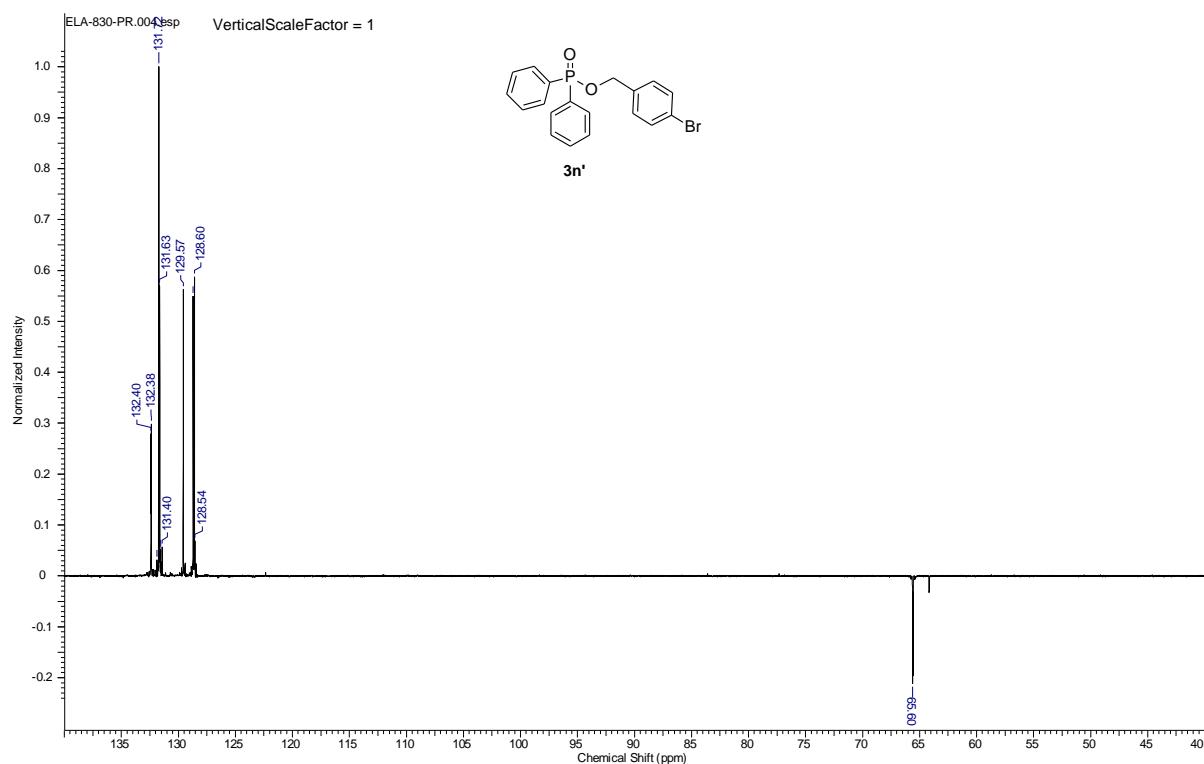


³¹P NMR spectra of 4-bromobenzyl diphenylphosphinate (**3n'**) (CDCl₃, 202 MHz).



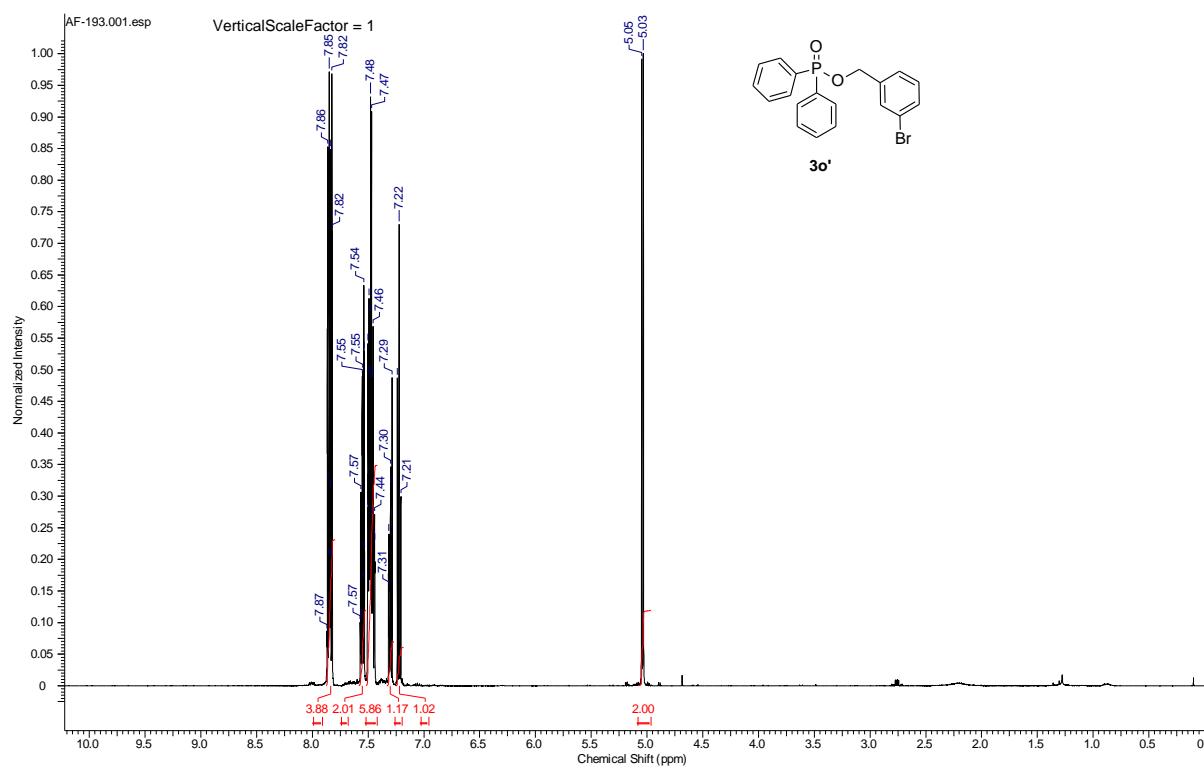
E:\FTP\NMR\ELA LASTAWIECKA\ELA-830-PR\ELA-830-PR.003.esp

³¹C NMR spectra of 4-bromobenzyl diphenylphosphinate (**3n'**) (CDCl_3 , 126 MHz).



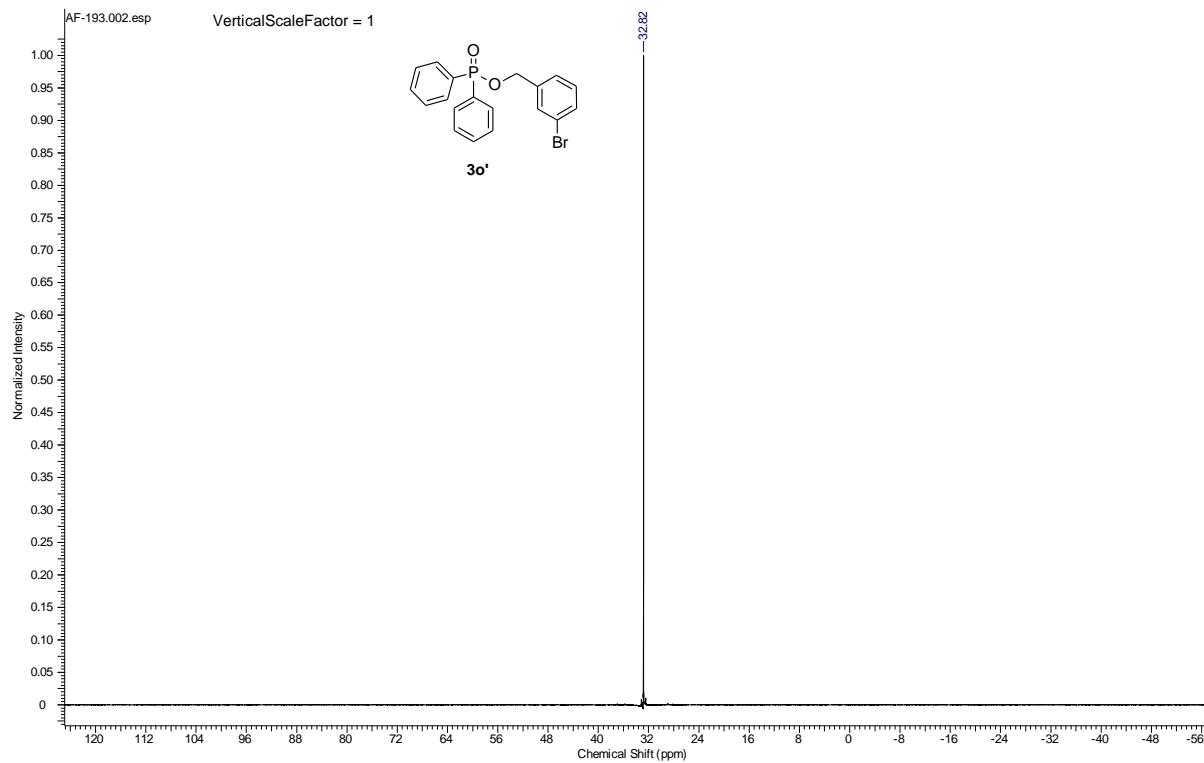
E:\FTP\NMR\ELA LASTAWIECKA\ELA-830-PR\ELA-830-PR.004.esp

DEPT 135 spectra of 4-bromobenzyl diphenylphosphinate (**3n'**) (CDCl_3 , 126 MHz).



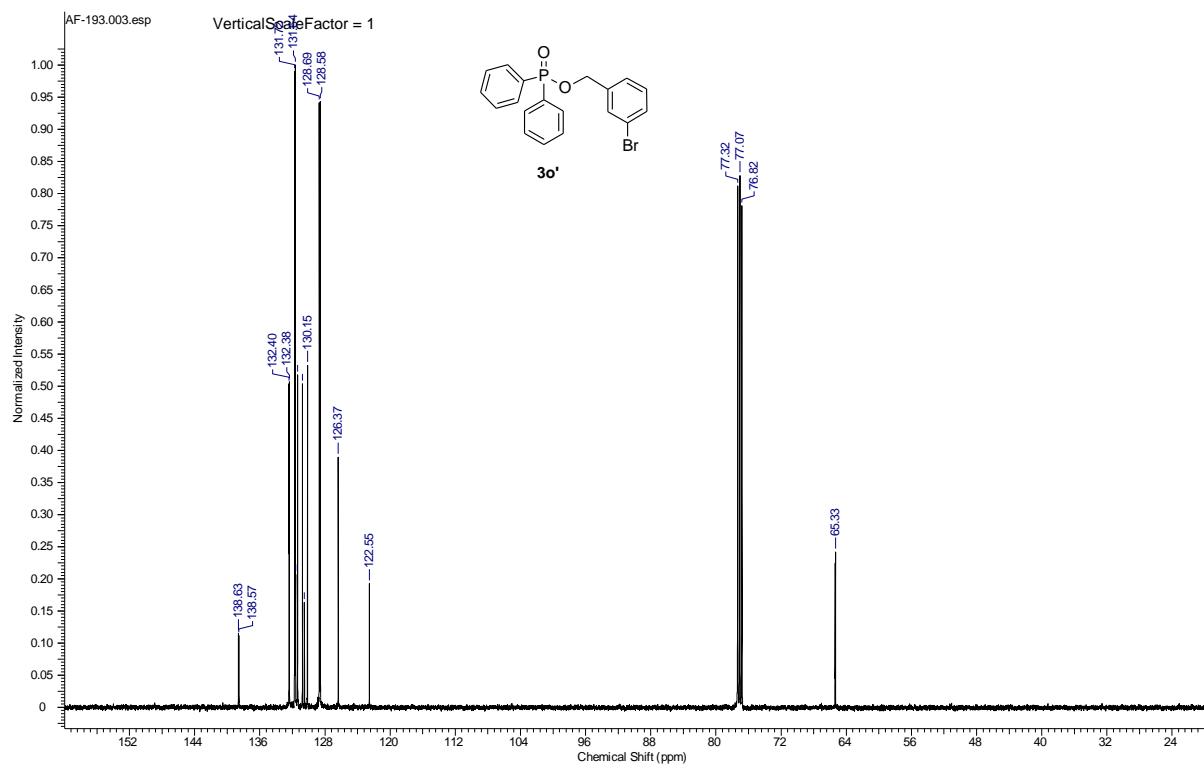
G:\NI-SPRZ\GANIA\SI\AF-193\AF-193.001.esp

¹H NMR spectra of 3-bromobenzyl diphenylphosphinate (**3o'**) (CDCl₃, 500 MHz).



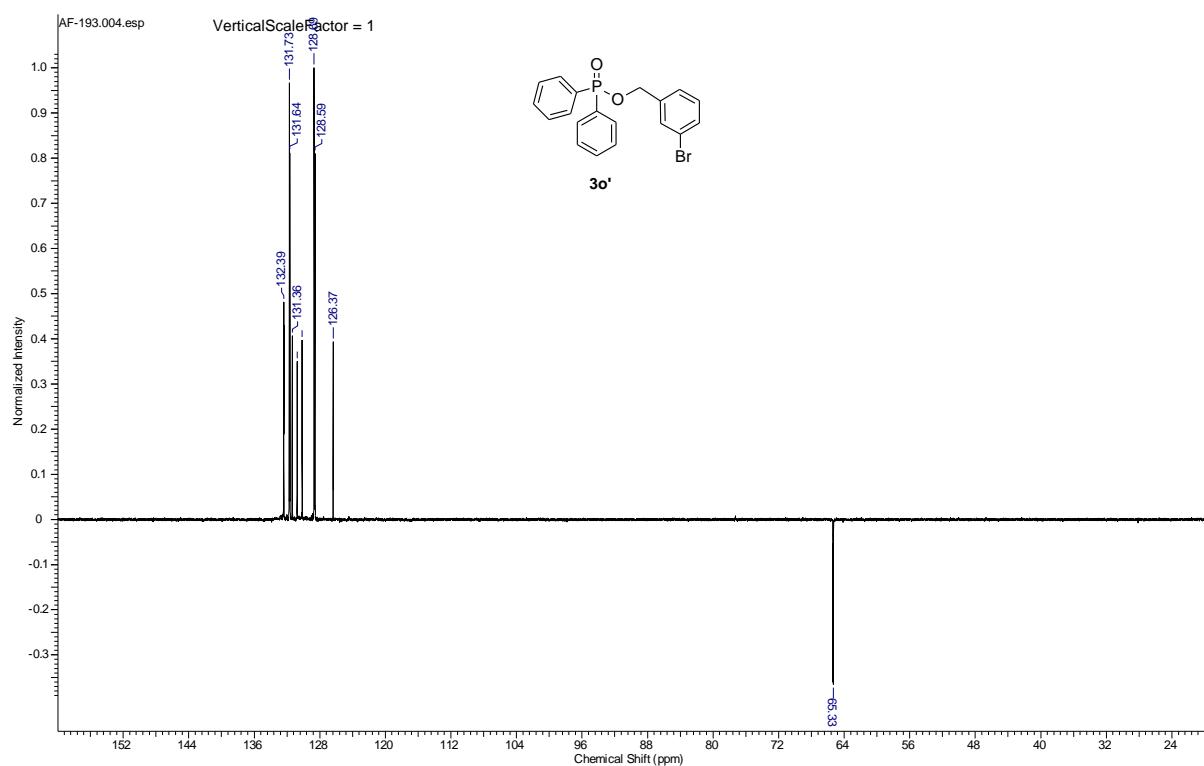
E:\FTP\NMR\ELA LASTOWIECKA\AF-193\AF-193.002.esp

³¹P NMR spectra of 3-bromobenzyl diphenylphosphinate (**3o'**) (CDCl₃, 202 MHz).



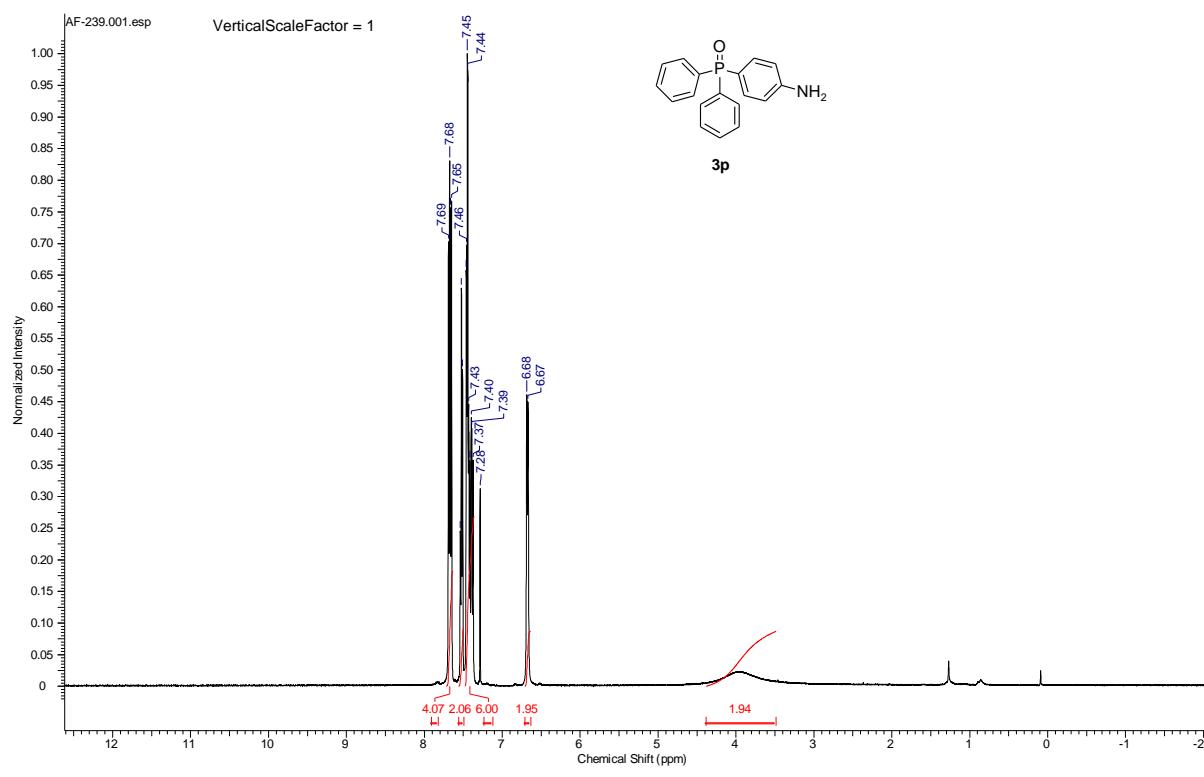
E:\FTP\NMR\ELA LASTAWIECKA\AF-193\AF-193.003.esp

³¹C NMR spectra of 3-bromobenzyl diphenylphosphinate (**3o'**) (CDCl₃, 126 MHz).

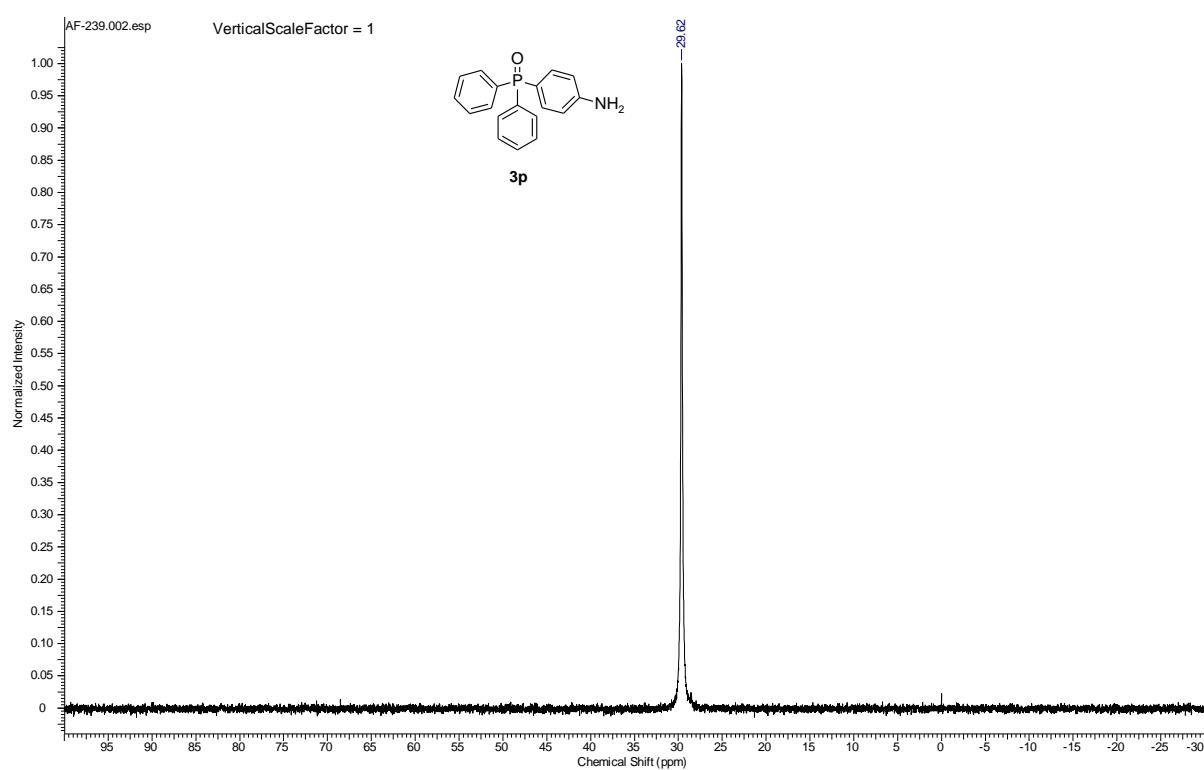


E:\FTP\NMR\ELA LASTAWIECKA\AF-193\AF-193.004.esp

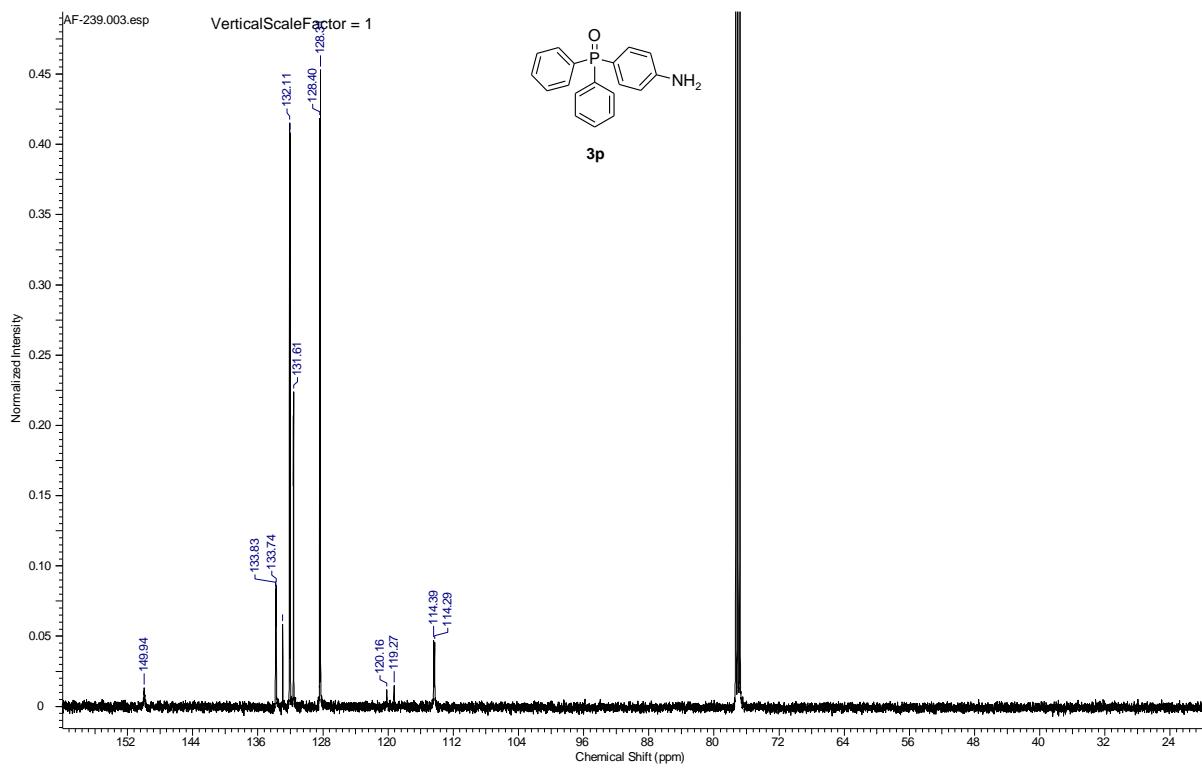
DEPT 135 spectra of 3-bromobenzyl diphenylphosphinate (**3o'**) (CDCl₃, 126 MHz).



¹H NMR spectra of (4-aminophenyl)diphenylphosphine oxide (**3p**) (CDCl₃, 500 MHz).

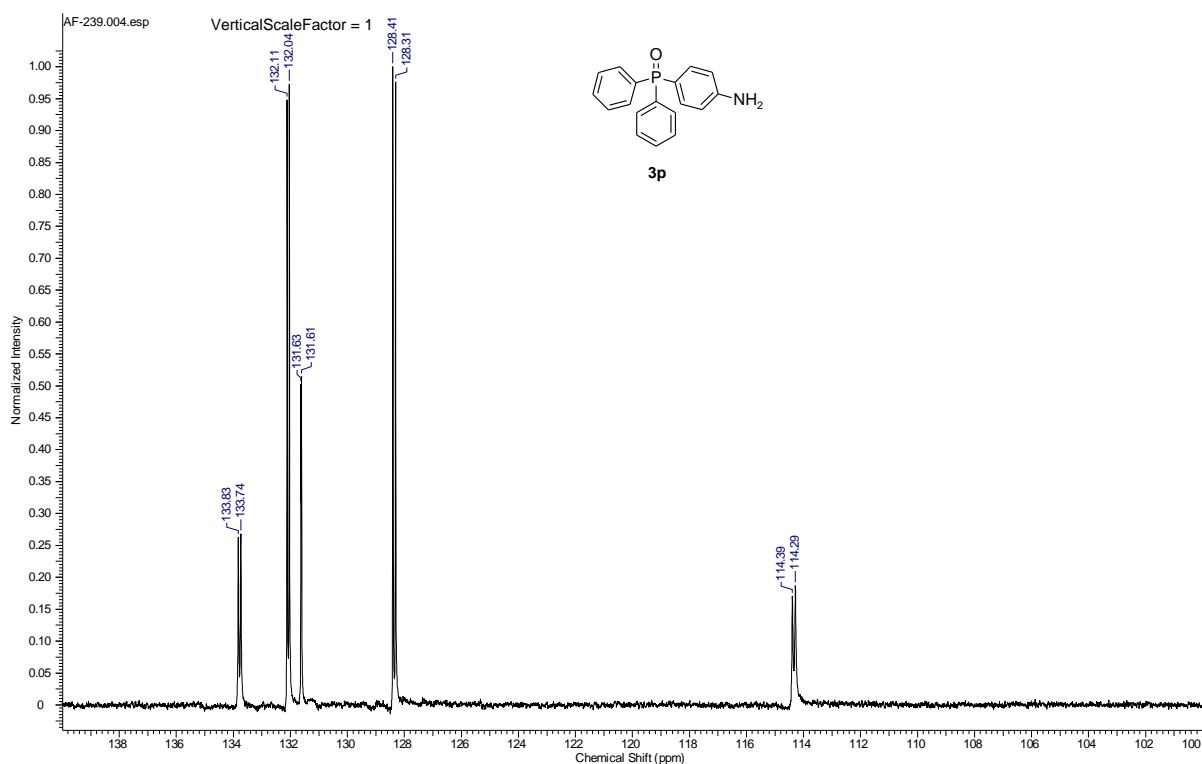


³¹P NMR spectra of (4-aminophenyl)diphenylphosphine oxide (**3p**) (CDCl₃, 202 MHz).



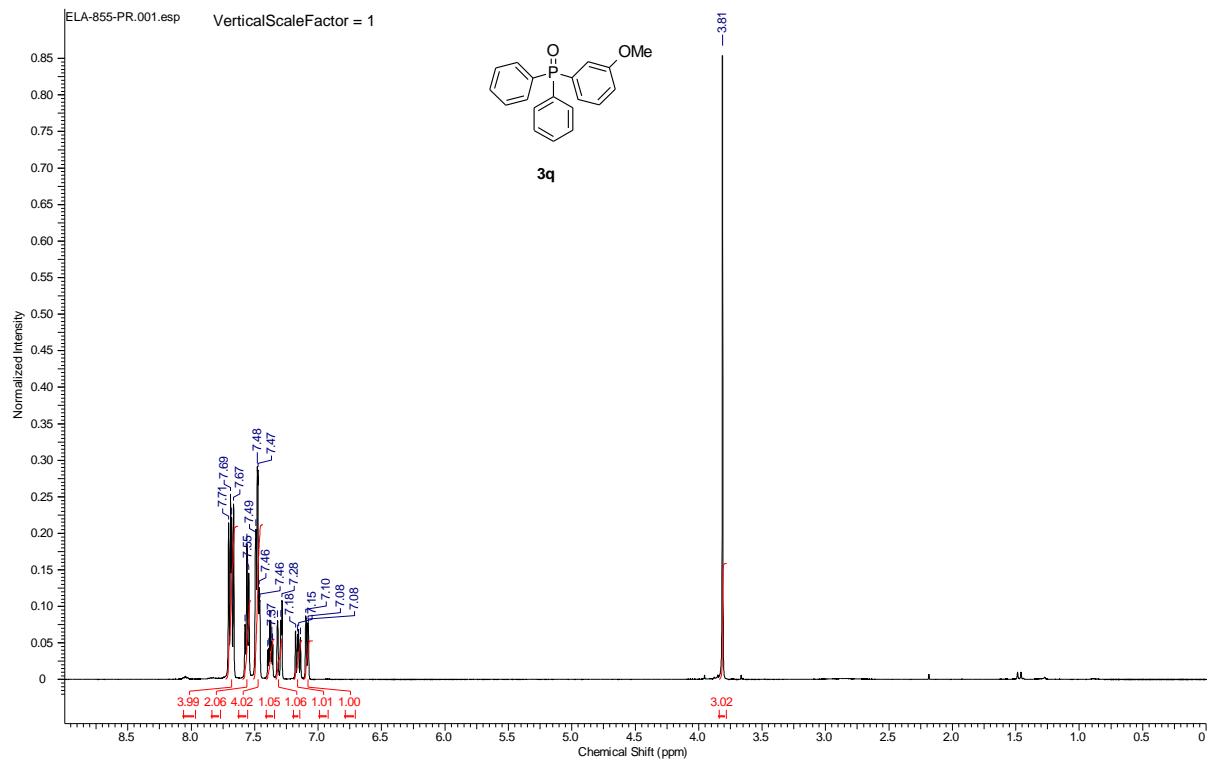
E:\FTP\NMR\ELA LASTAWIECKA\AF-239\AF-239.003.esp

³¹C NMR spectra of (4-aminophenyl)diphenylphosphine oxide (**3p**) (CDCl₃, 126 MHz).

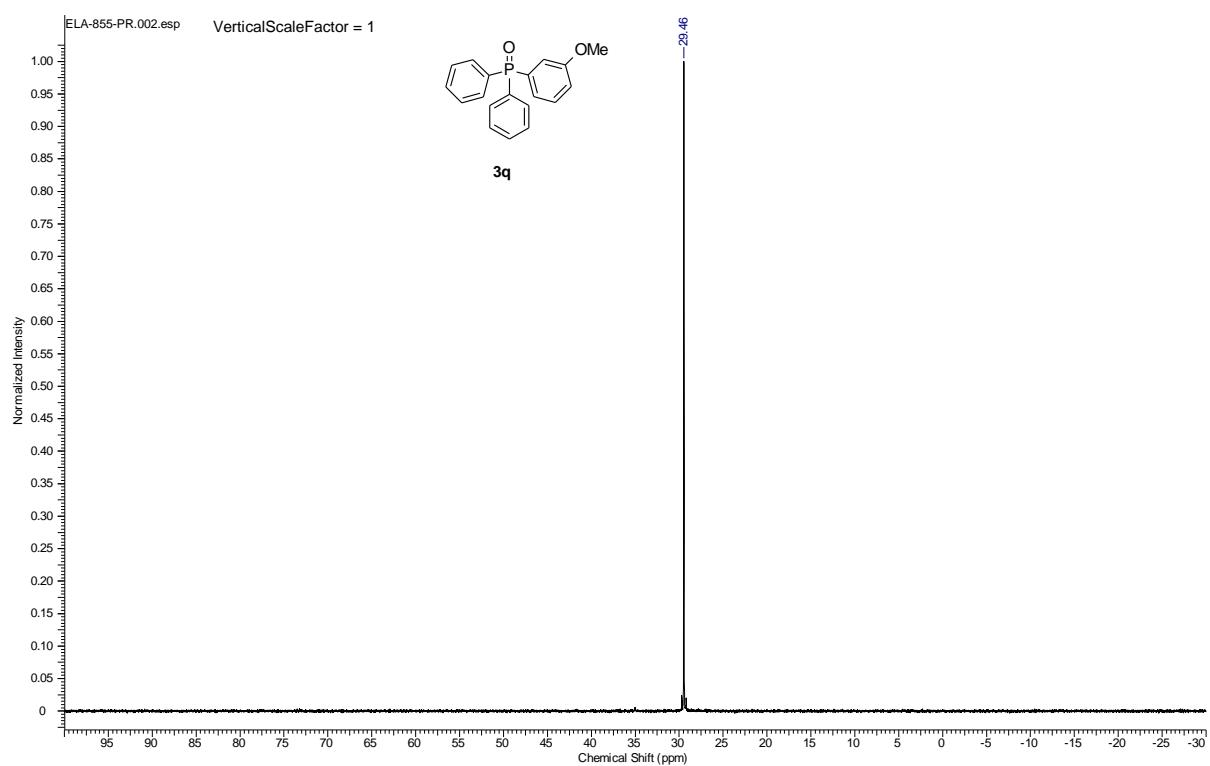


E:\FTP\NMR\ELA LASTAWIECKA\AF-239\AF-239.004.esp

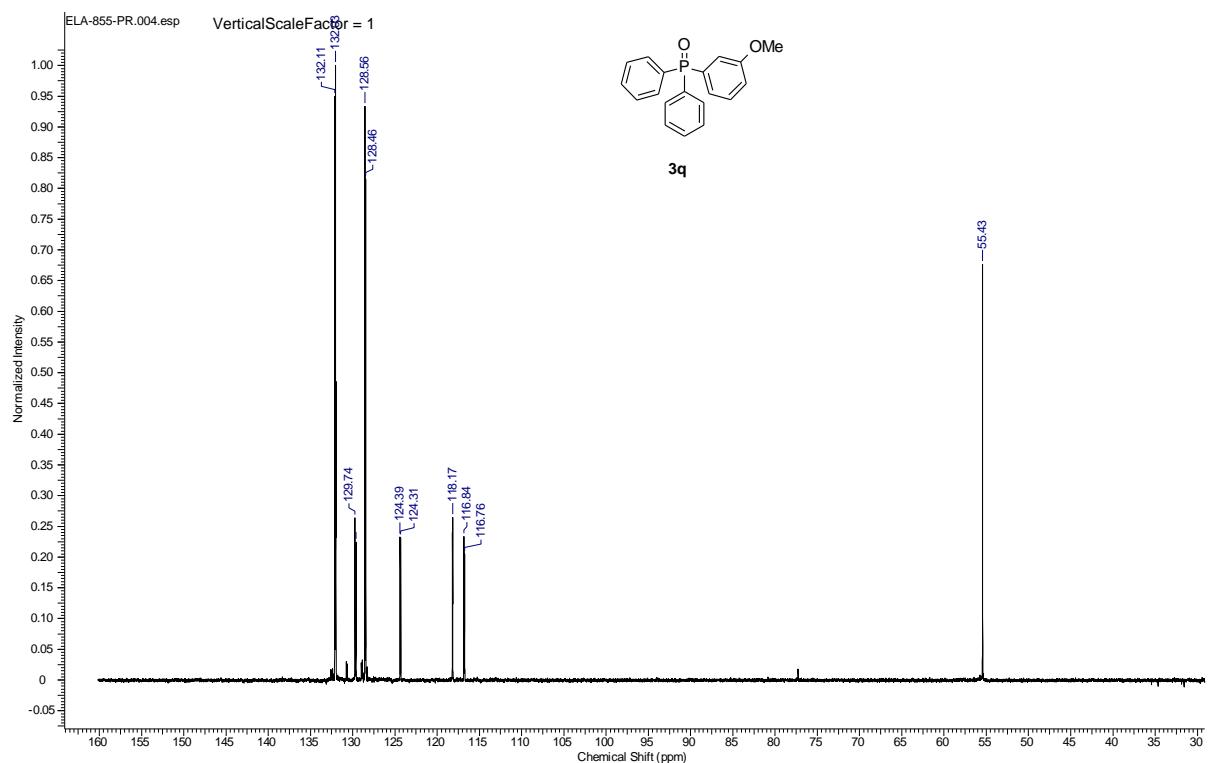
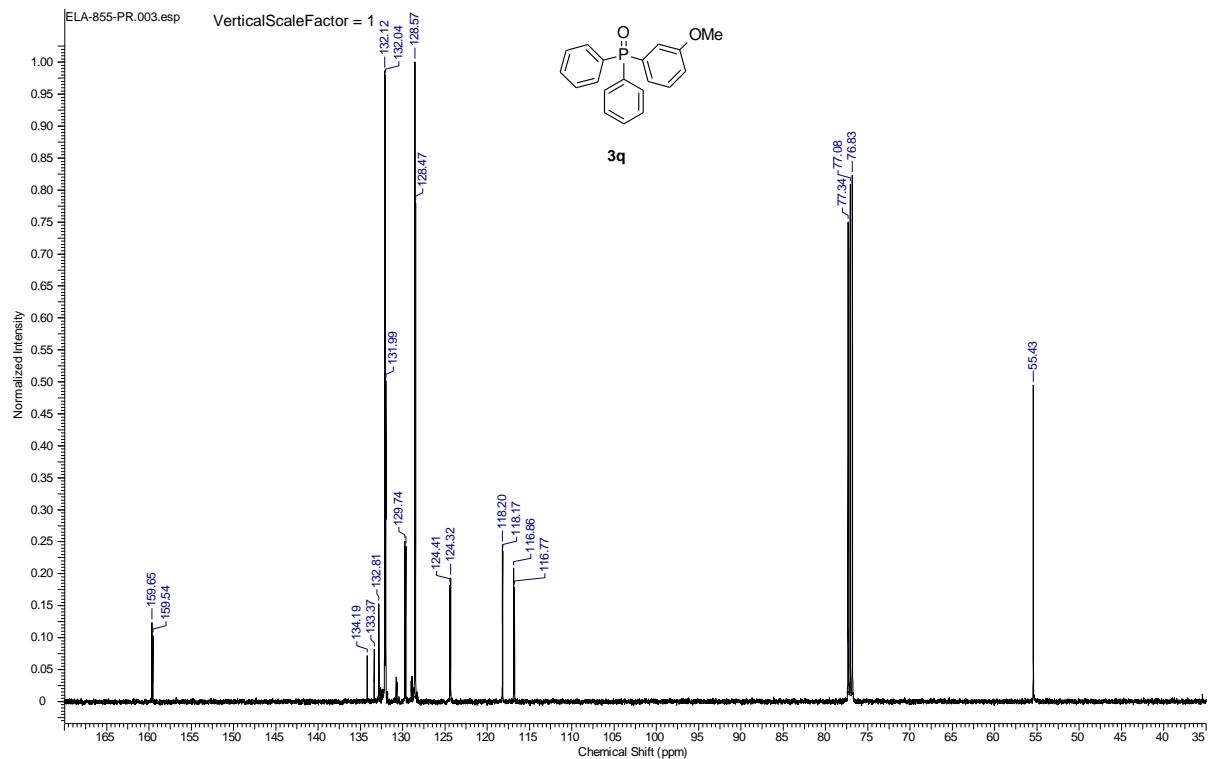
DEPT 135 spectra of (4-aminophenyl)diphenylphosphine oxide (**3p**) (CDCl₃, 126 MHz).



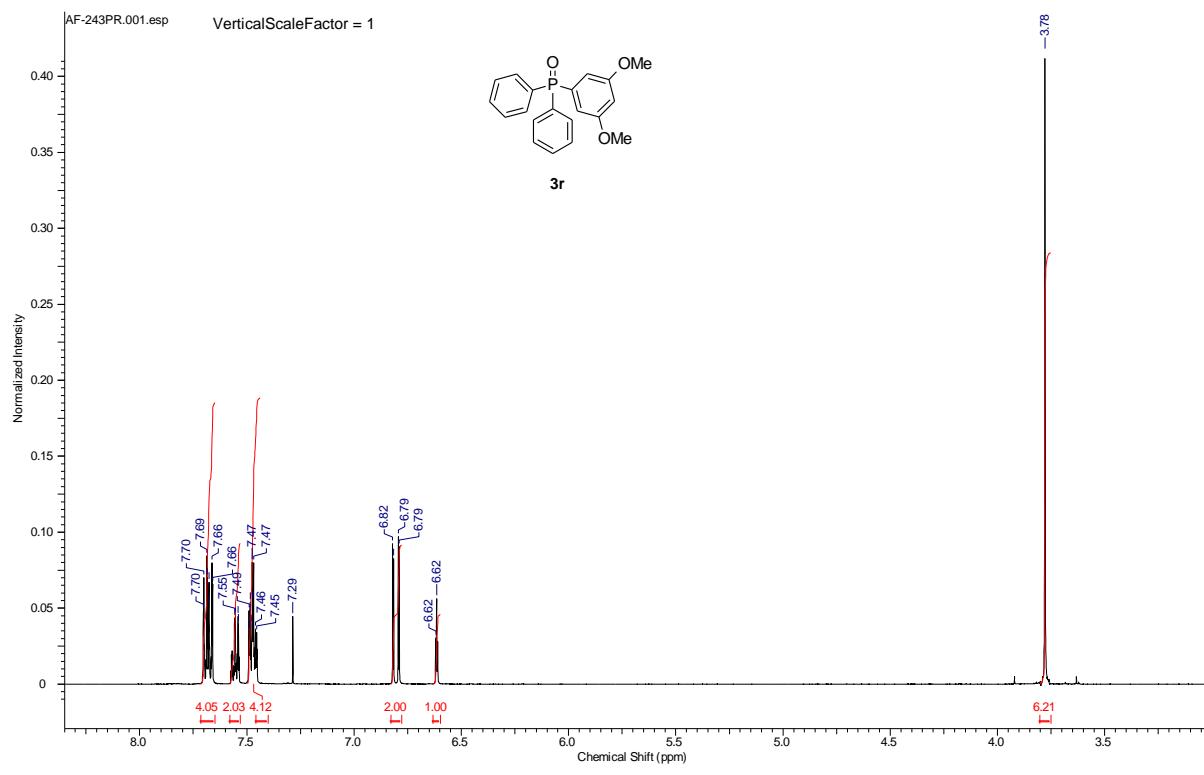
¹H NMR spectra of (3-methoxyphenyl)diphenylphosphine oxide (**3q**) (CDCl₃, 500 MHz).



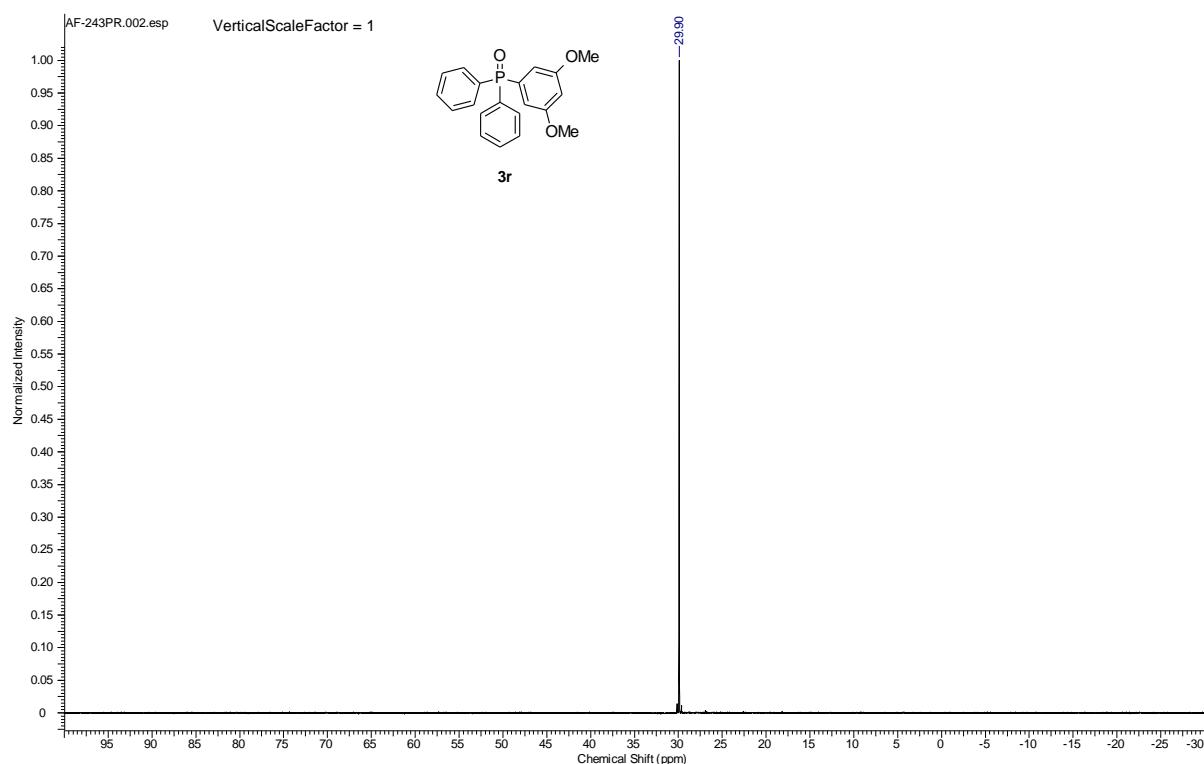
³¹P NMR spectra of (3-methoxyphenyl)diphenylphosphine oxide (**3q**) (CDCl₃, 202 MHz).



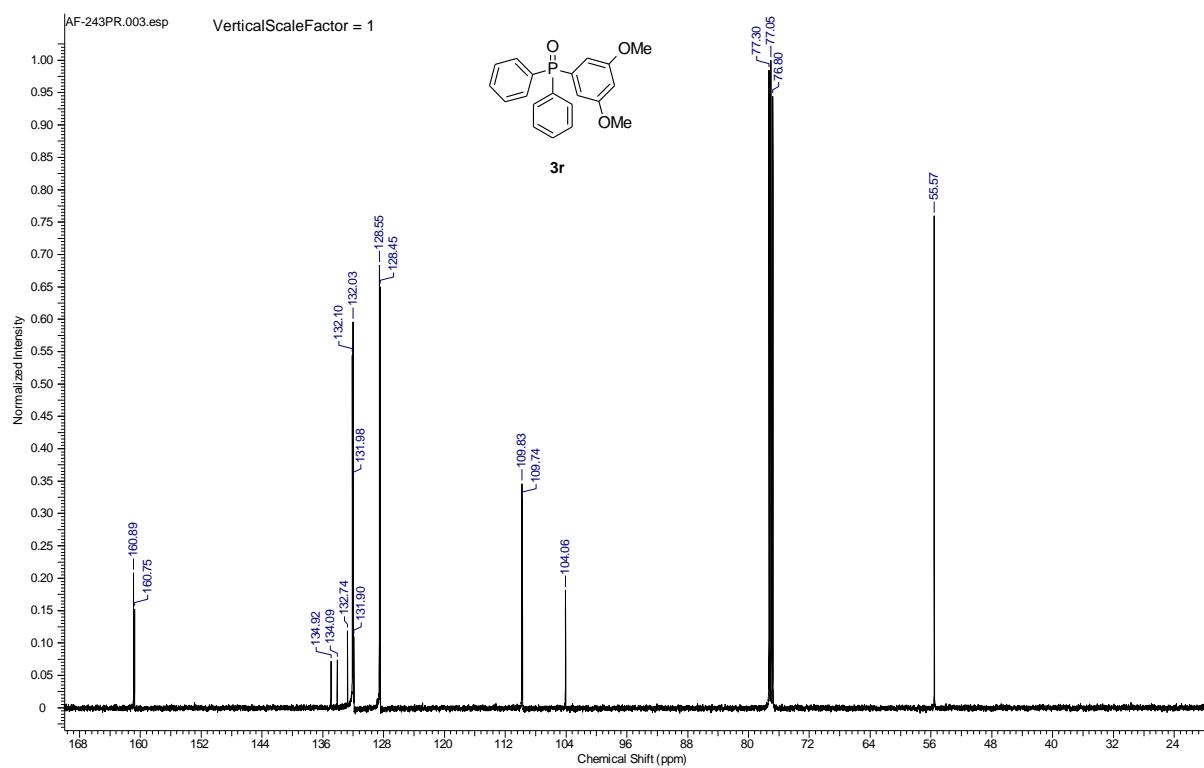
DEPT 135 spectra of (3-methoxyphenyl)diphenylphosphine oxide (**3q**) (CDCl₃, 126 MHz).



¹H NMR spectra of 3,5-dimethoxyphenyl(diphenyl)phosphine oxide (**3r**) (CDCl₃, 500 MHz).

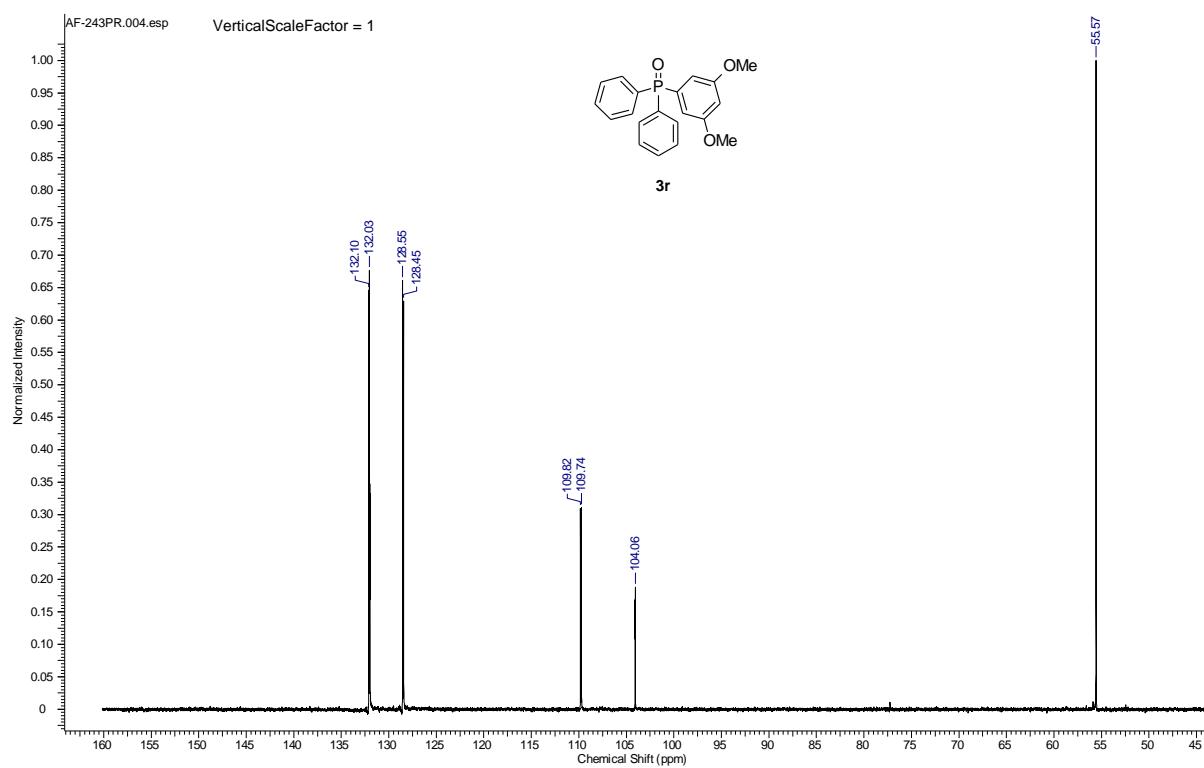


³¹P NMR spectra of 3,5-dimethoxyphenyl(diphenyl)phosphine oxide (**3r**) (CDCl₃, 202 MHz).



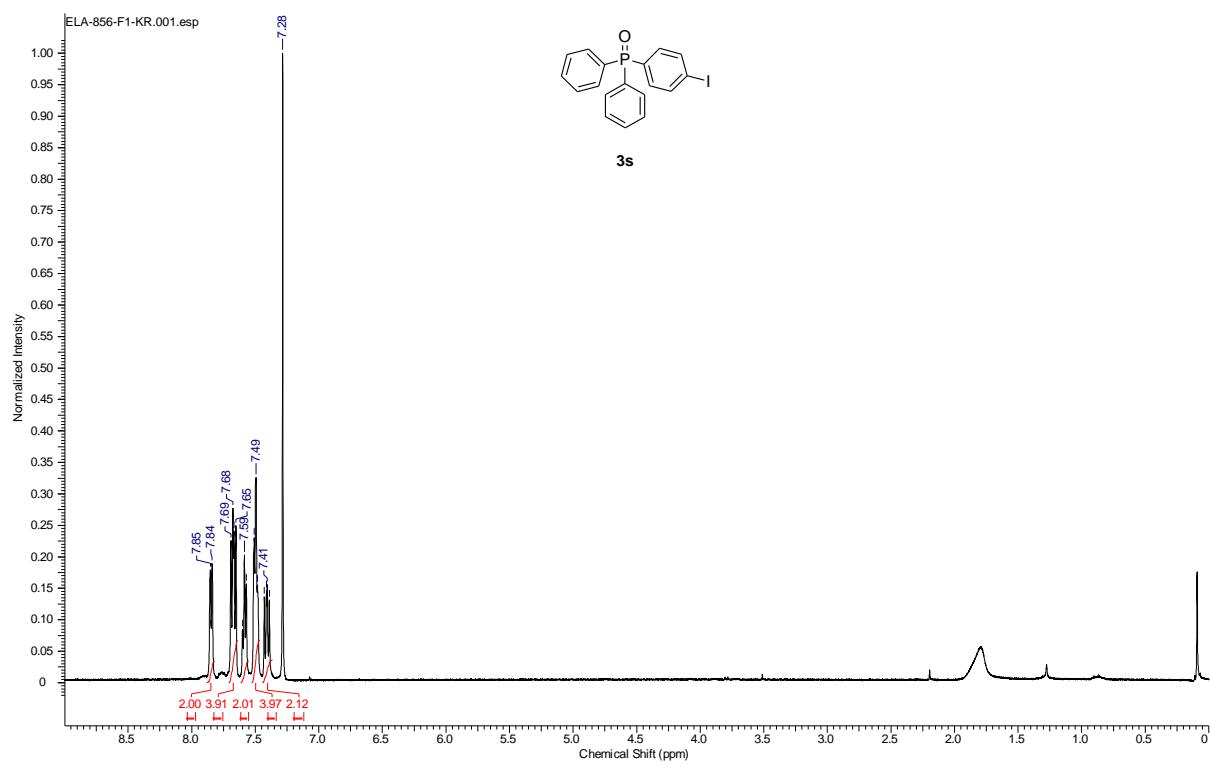
E:\FTP\NMR\ELA LASTAWIECKA\AF-243PR\AF-243PR.003.esp

³¹C NMR spectra of 3,5-dimethoxyphenyl(diphenyl)phosphine oxide (3r**) (CDCl₃, 126 MHz).**



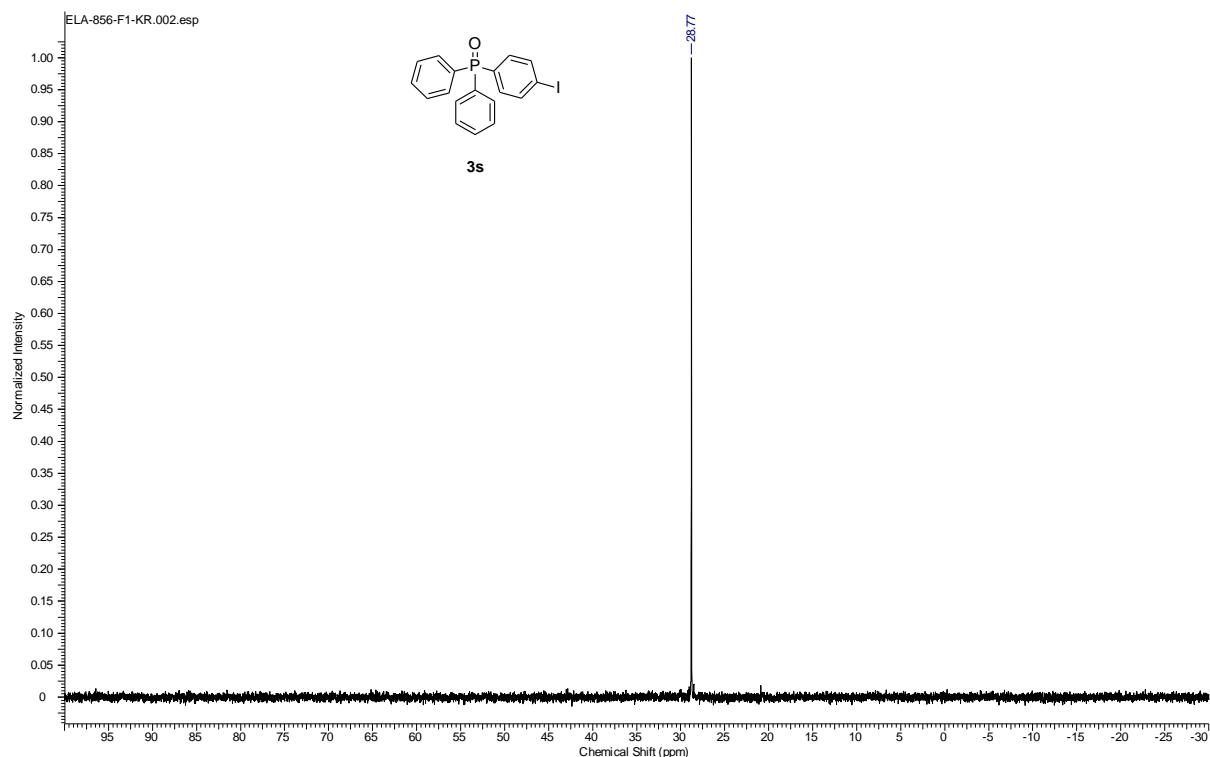
E:\FTP\NMR\ELA LASTAWIECKA\AF-243PR\AF-243PR.004.esp

DEPT 135 spectra of 3,5-dimethoxyphenyl(diphenyl)phosphine oxide (3r**) (CDCl₃, 126 MHz).**



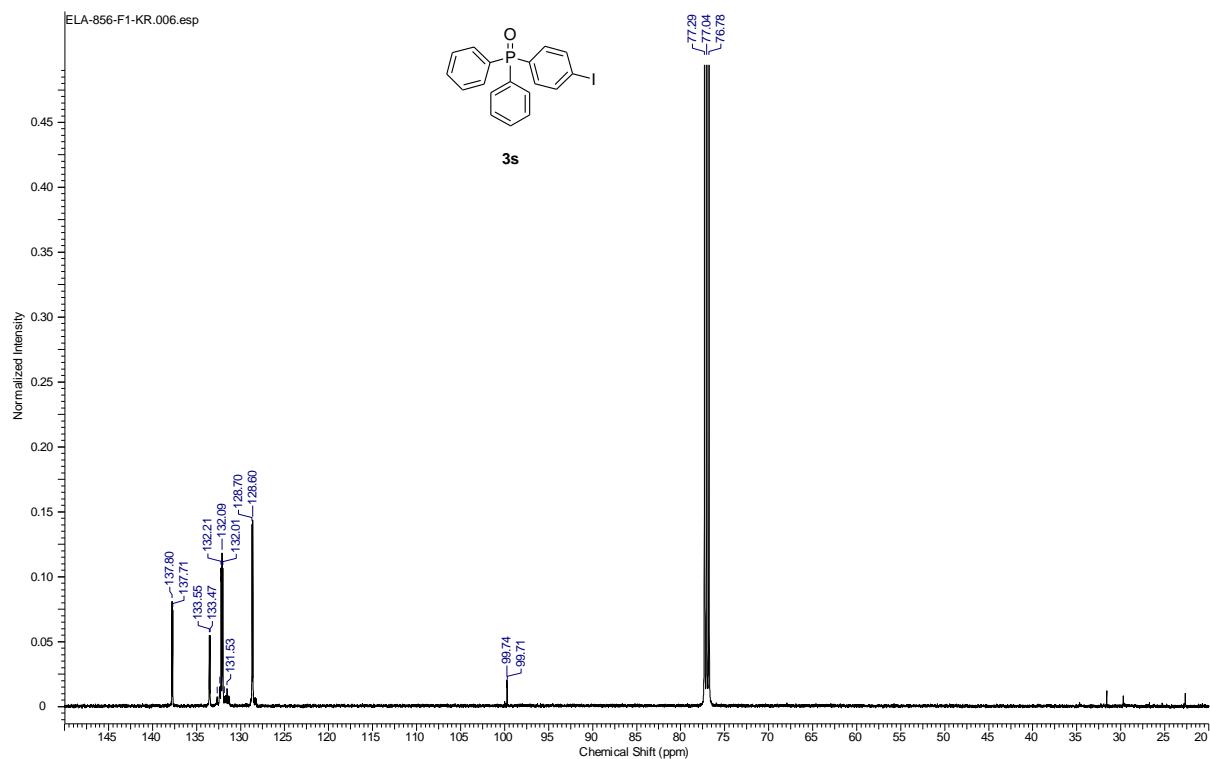
E:\FTP\NMR\ELA LASTAWIECKA\ELA-856-F1-KR\ELA-856-F1-KR.001.esp

¹H NMR spectra of (4-iodophenyl)diphenylphosphine oxide (**3s**) (CDCl₃, 500 MHz).



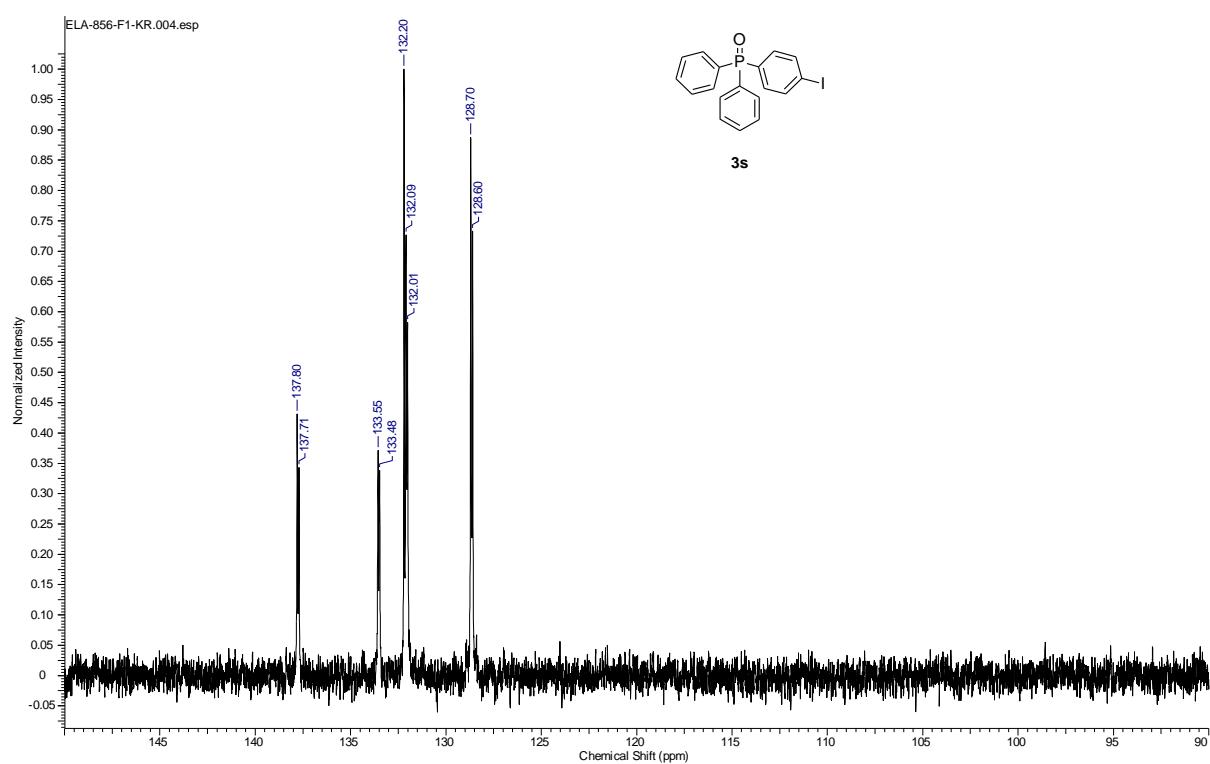
E:\FTP\NMR\ELA LASTAWIECKA\ELA-856-F1-KR\ELA-856-F1-KR.002.esp

³¹P NMR spectra of (4-iodophenyl)diphenylphosphine oxide (**3s**) (CDCl₃, 202 MHz).



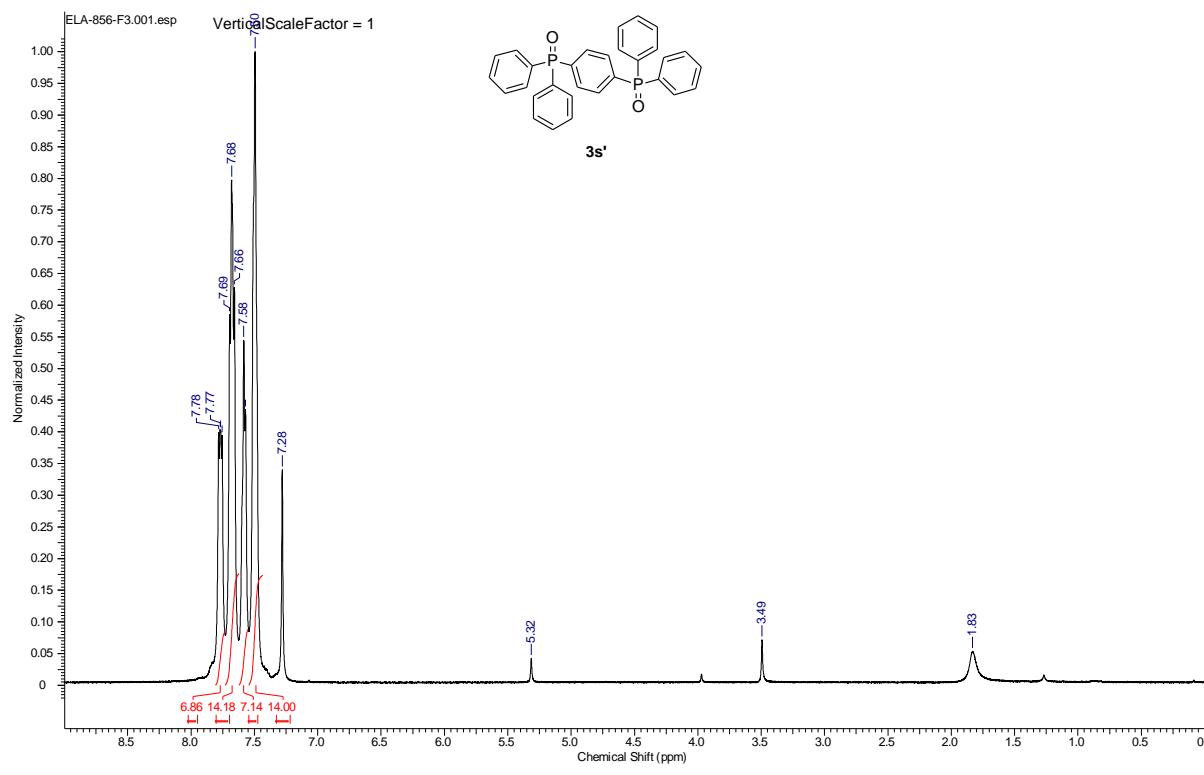
E:\FTP\NMR\ELA LASTAWIECKA\ELA-856-F1-KR\ELA-856-F1-KR.006.esp

³¹C NMR spectra of (4-iodophenyl)diphenylphosphine oxide (**3s**) (CDCl₃, 126 MHz).

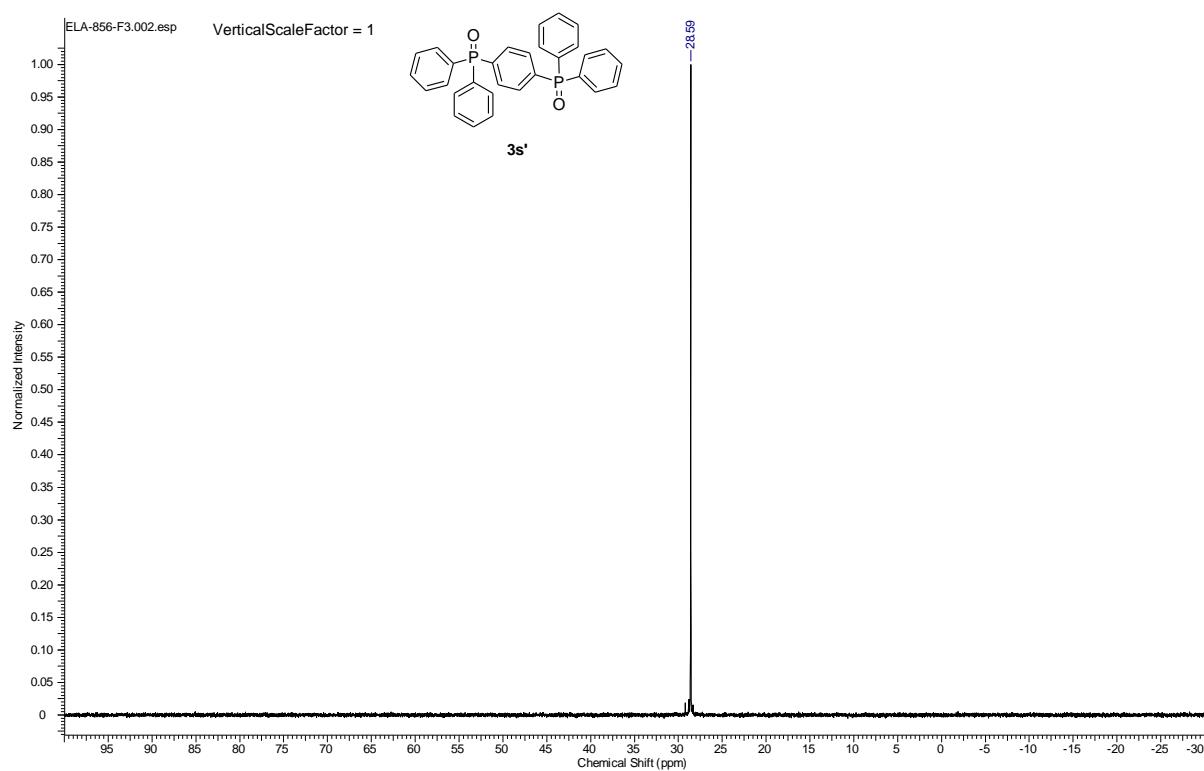


E:\FTP\NMR\ELA LASTAWIECKA\ELA-856-F1-KR\ELA-856-F1-KR.004.esp

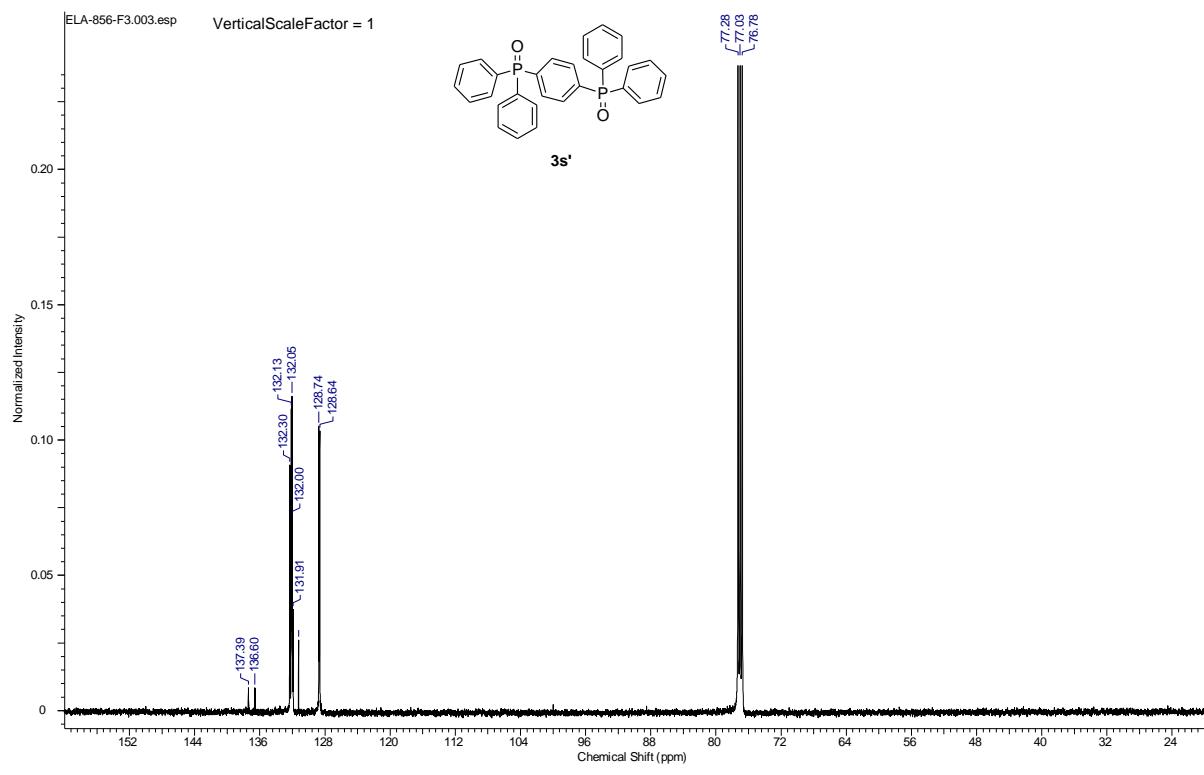
DEPT 135 spectra of (4-iodophenyl)diphenylphosphine oxide (**3s**) (CDCl₃, 126 MHz).



¹H NMR spectra of 1,4-phenylenebis(diphenylphosphine) dioxide (**3s'**) (CDCl₃, 500 MHz).

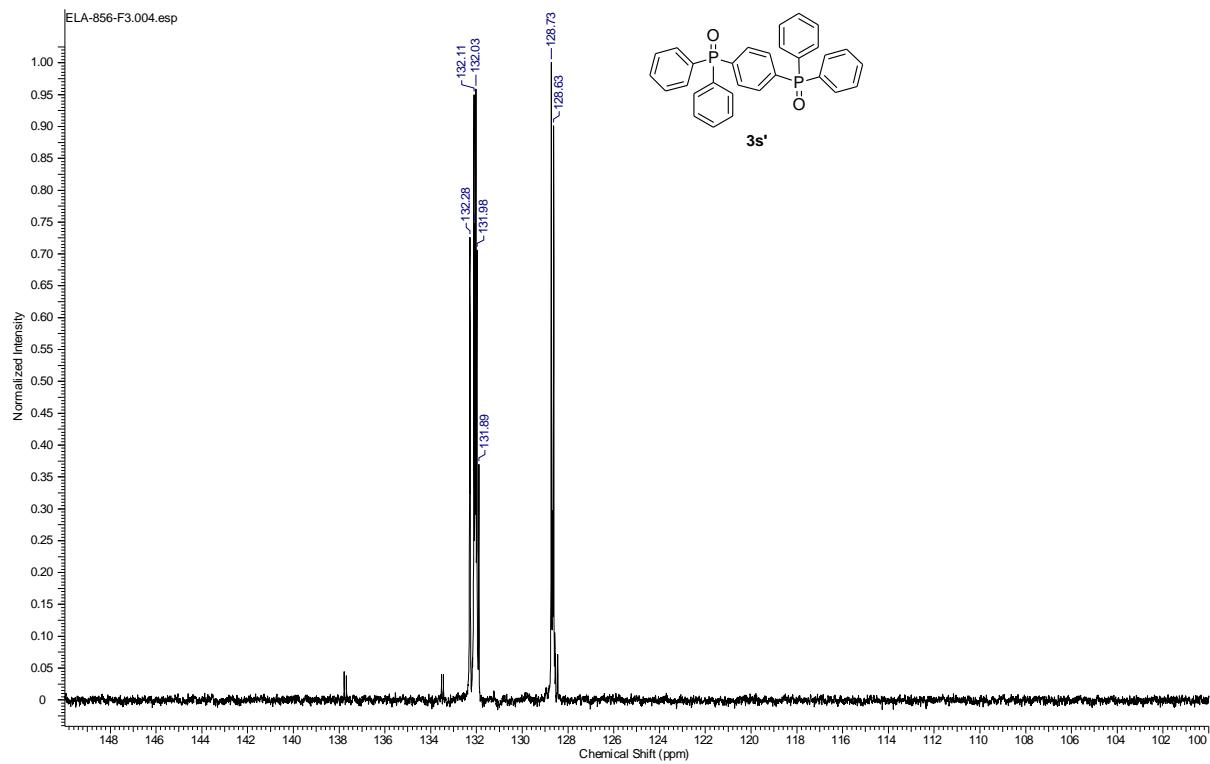


³¹P NMR spectra of 1,4-phenylenebis(diphenylphosphine) dioxide (**3s'**) (CDCl₃, 202 MHz).



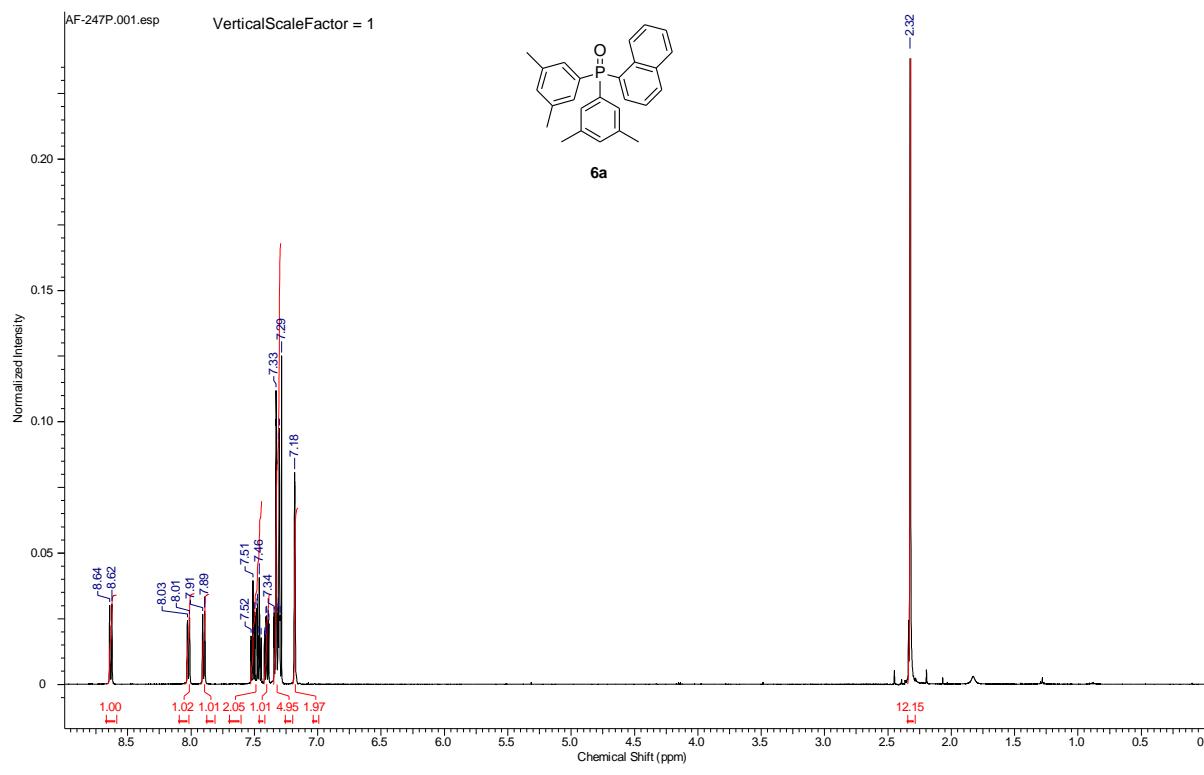
E:\FTP\NMR\KRYSTYNA ZYGO\ELA-856-F3\ELA-856-F3.003.esp

³¹C NMR spectra of 1,4-phenylenebis(diphenylphosphine) dioxide (**3s'**) (CDCl₃, 126 MHz).

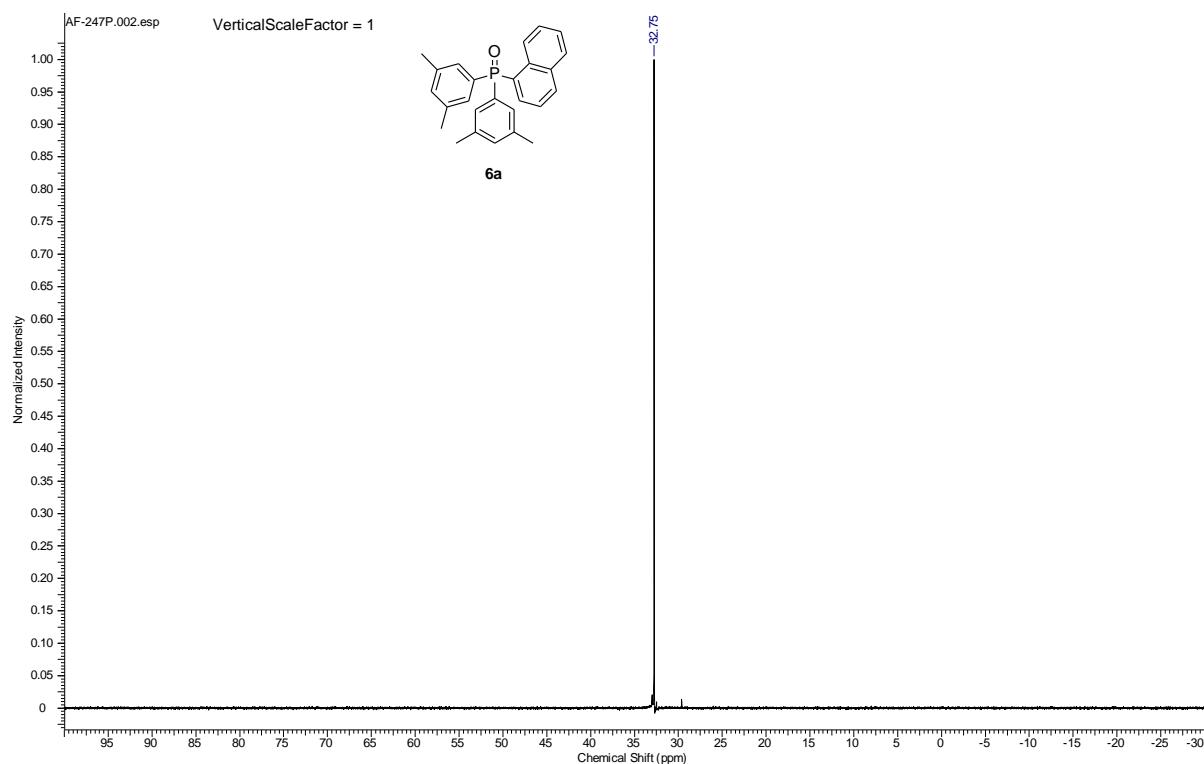


E:\FTP\NMR\IELA LASTAWIECKA\ELA-856-F3\ELA-856-F3.004.esp

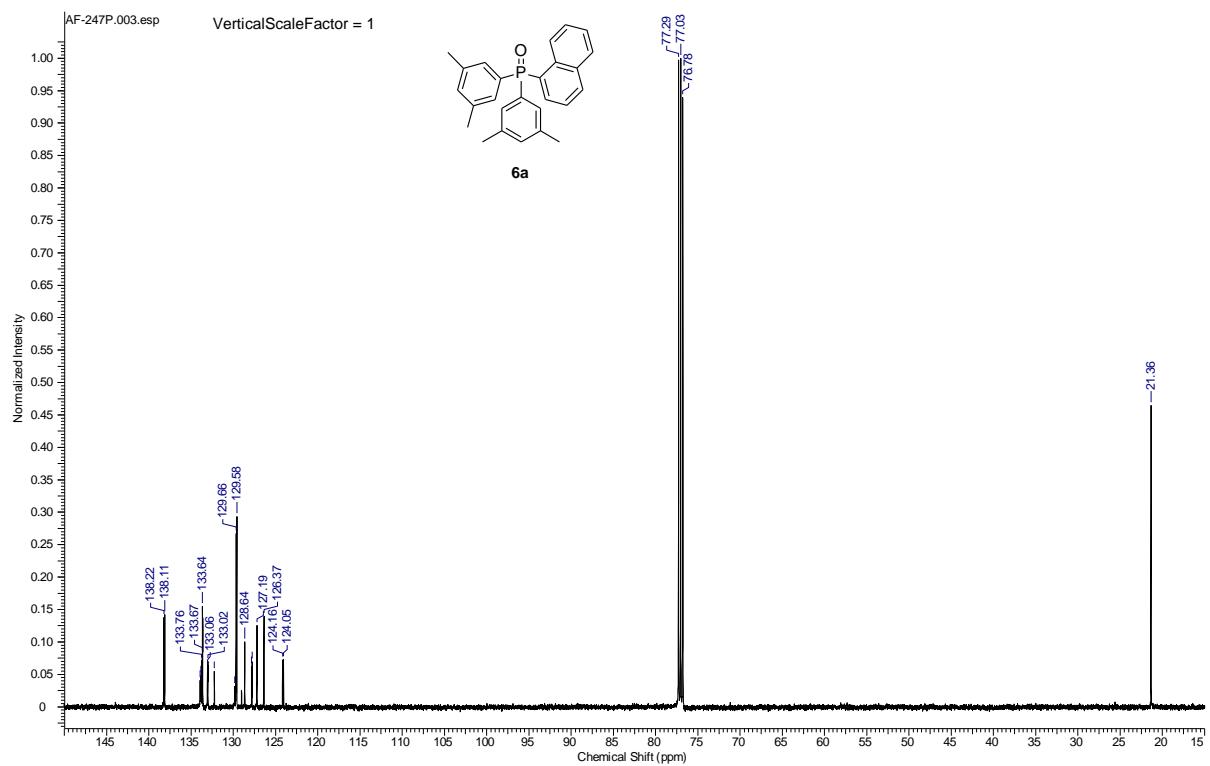
DEPT 135 spectra of 1,4-phenylenebis(diphenylphosphine) dioxide (**3s'**) (CDCl₃, 126 MHz).



¹H NMR spectra of bis(3,5-xylyl)(1-naphthyl)phosphine oxide (**6a**) (CDCl₃, 500 MHz).

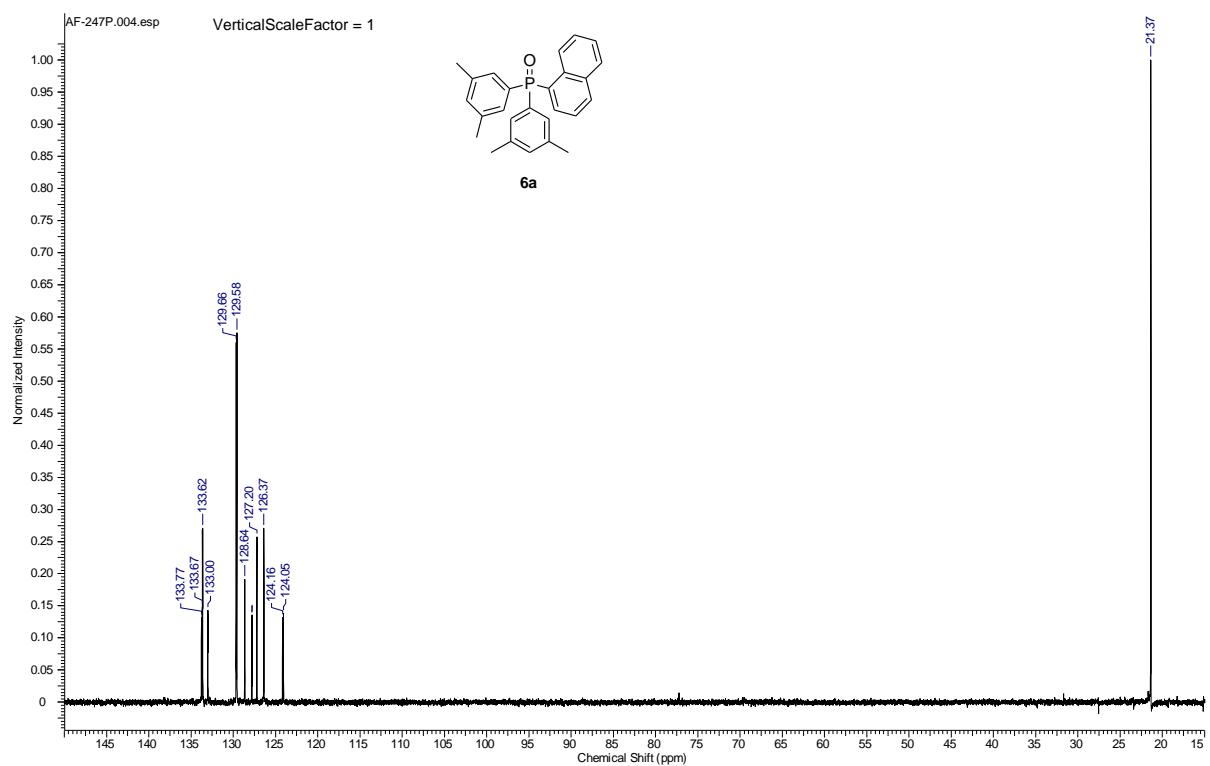


³¹P NMR spectra of bis(3,5-xylyl)(1-naphthyl)phosphine oxide (**6a**) (CDCl₃, 202 MHz).



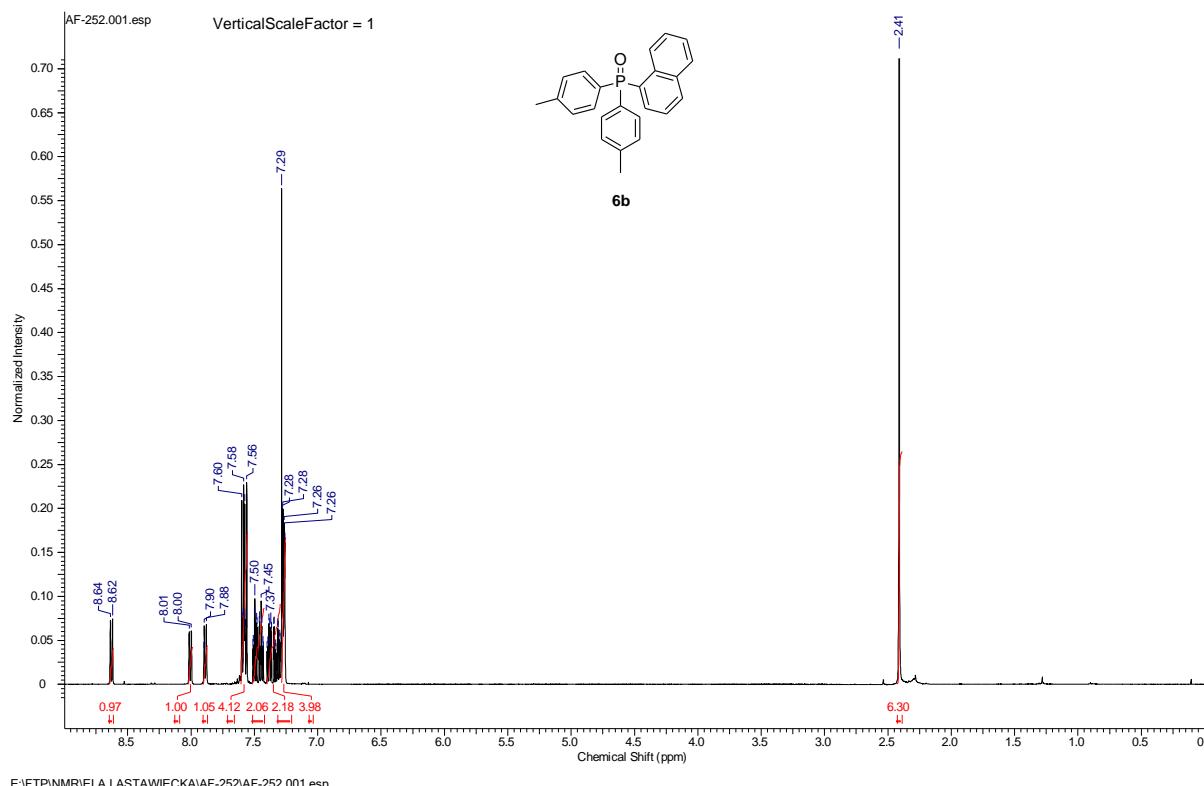
E:\FTP\NMR\ELA LASTAWIECKA\AF-247P\AF-247P.003.esp

^{31}C NMR spectra of bis(3,5-xylyl)(1-naphthyl)phosphine oxide (**6a**) (CDCl_3 , 126 MHz).

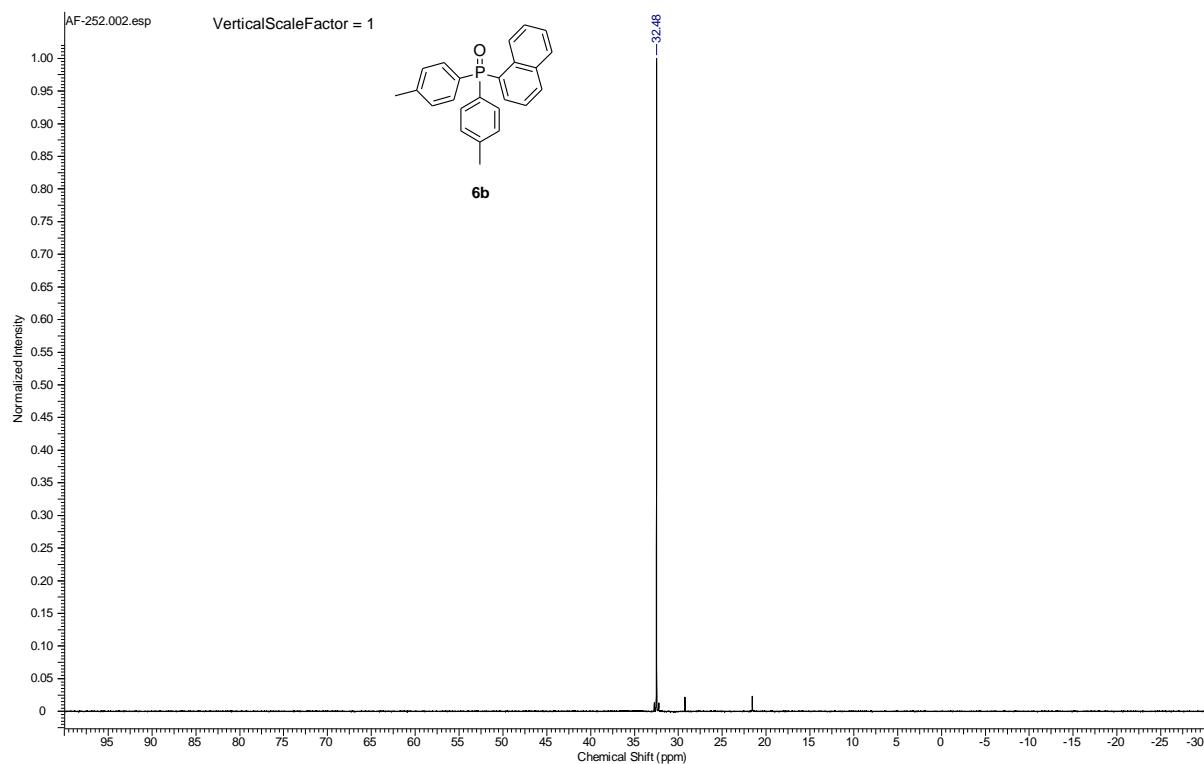


E:\FTP\NMR\ELA LASTAWIECKA\AF-247P\AF-247P.004.esp

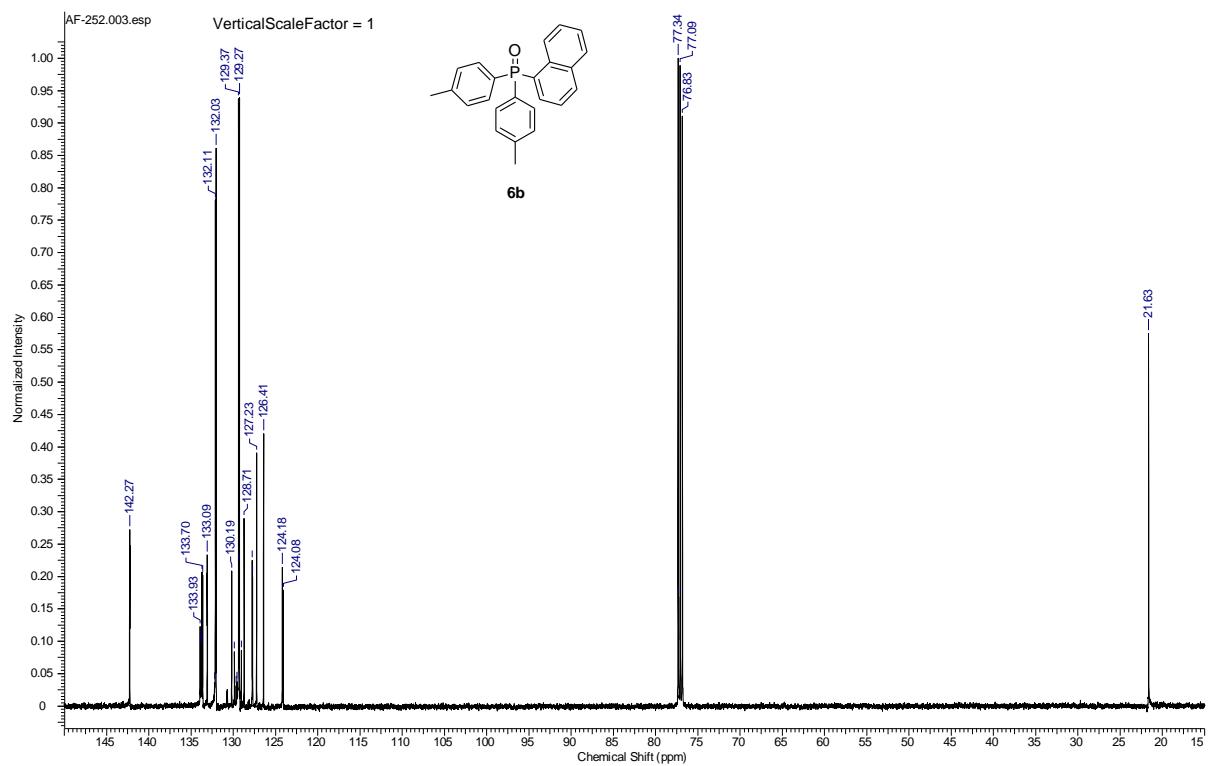
DEPT 135 spectra of bis(3,5-xylyl)(1-naphthyl)phosphine oxide (**6a**) (CDCl_3 , 126 MHz).



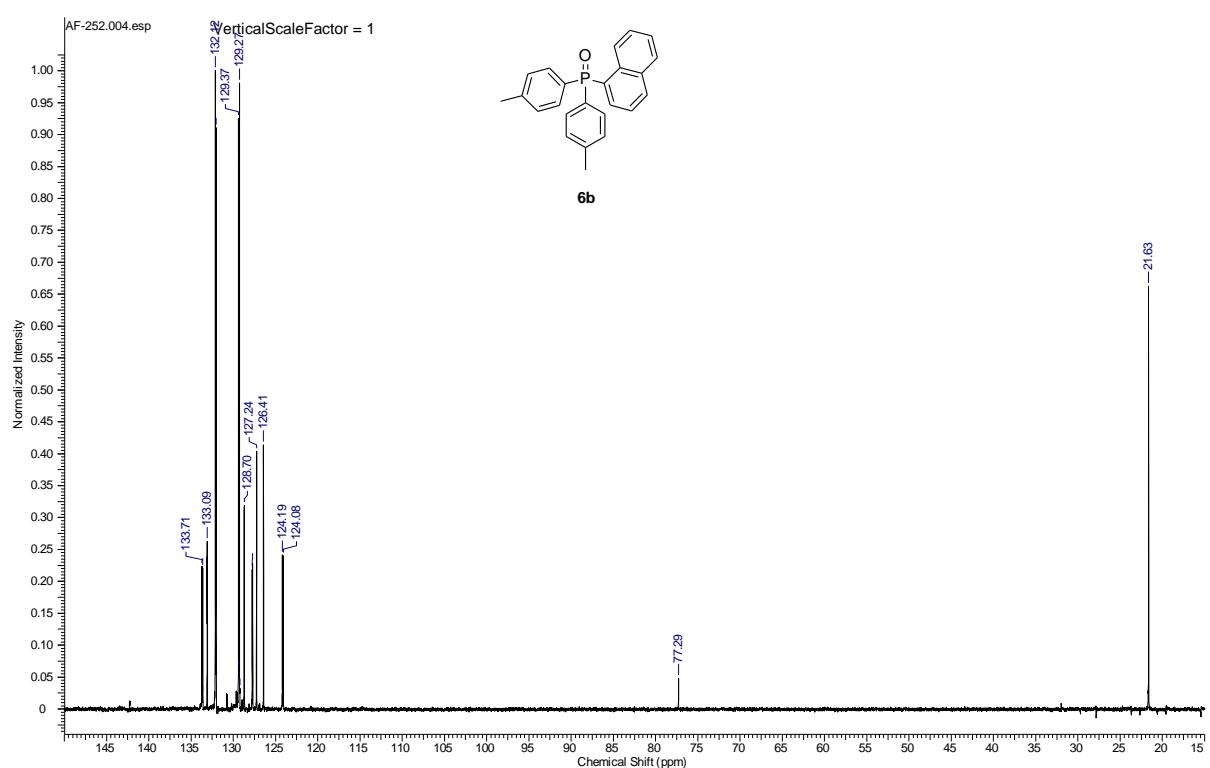
^1H NMR spectra of (1-naphthyl)bis(*p*-tolyl)phosphine oxide (**6b**) (CDCl_3 , 500 MHz).



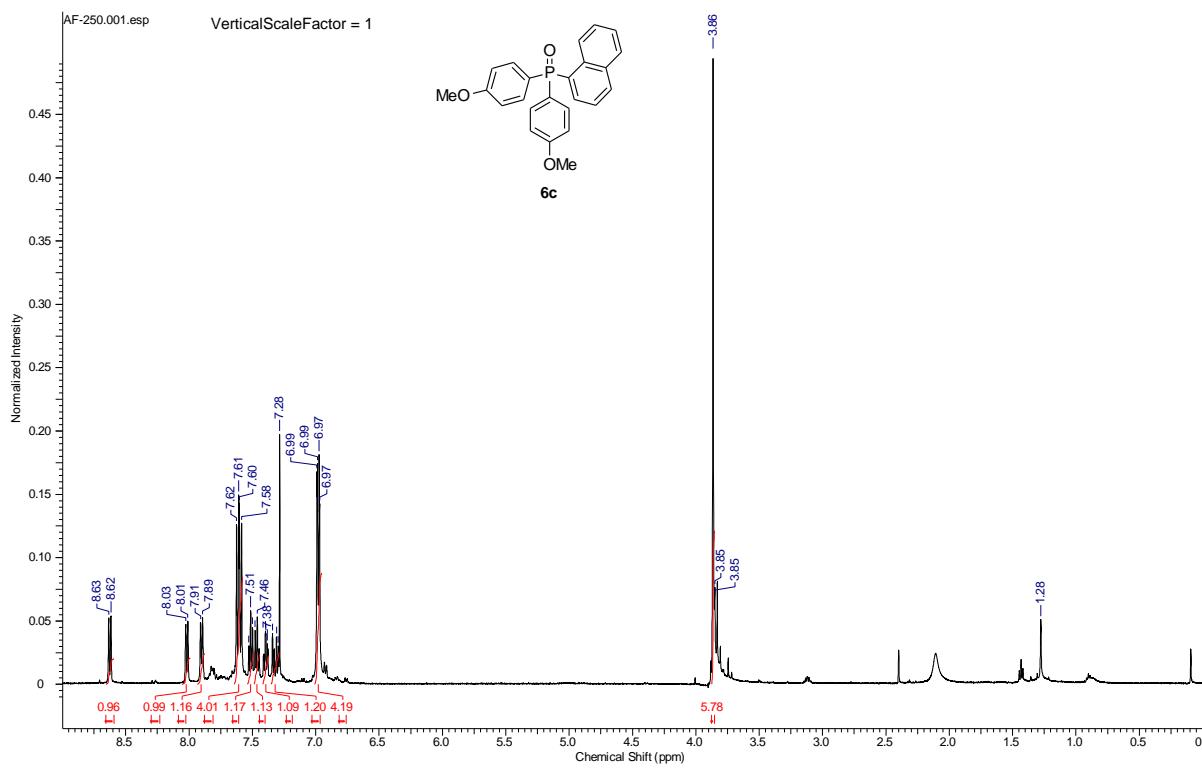
^{31}P NMR spectra of (1-naphthyl)bis(*p*-tolyl)phosphine oxide (**6b**) (CDCl_3 , 202 MHz).



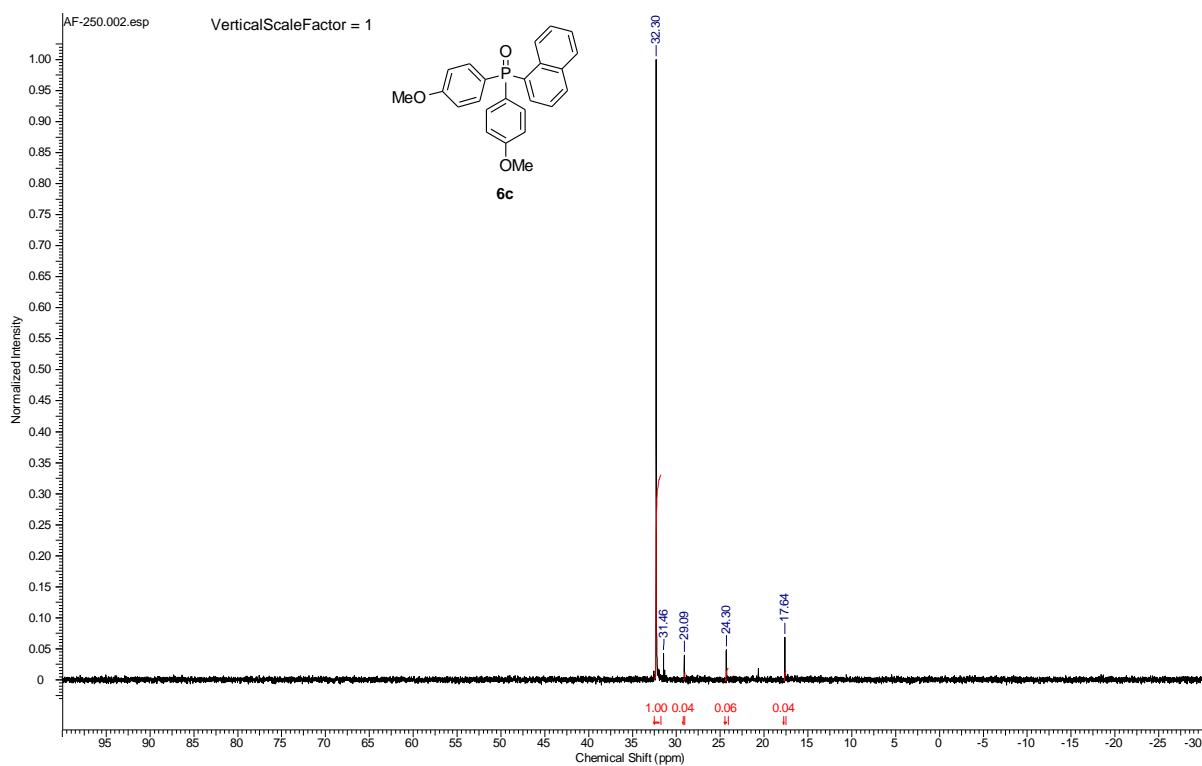
³¹C NMR spectra of (1-naphthyl)bis(*p*-tolyl)phosphine oxide (**6b**) (CDCl₃, 126 MHz).



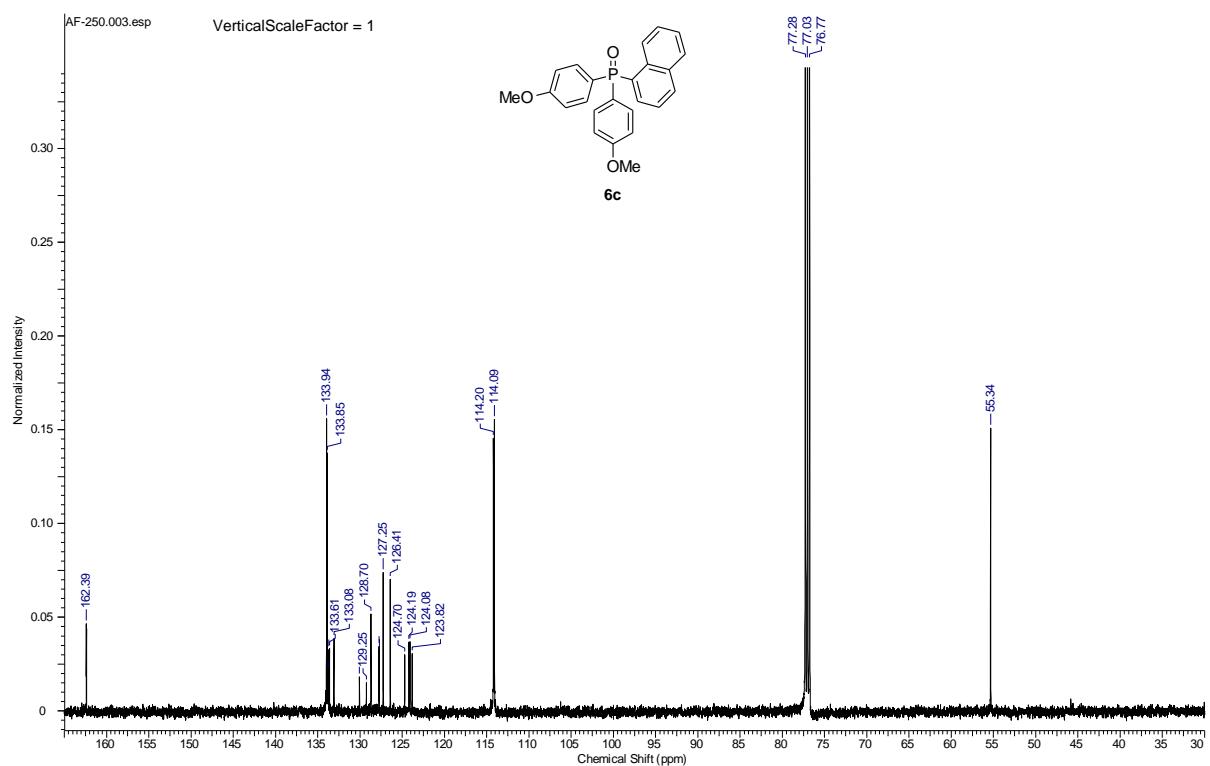
DEPT 135 spectra of (1-naphthyl)bis(*p*-tolyl)phosphine oxide (**6b**) (CDCl₃, 126 MHz).



¹H NMR spectra of bis(4-methoxyphenyl)(1-naphthyl)phosphine oxide (**6c**) (CDCl₃, 500 MHz).

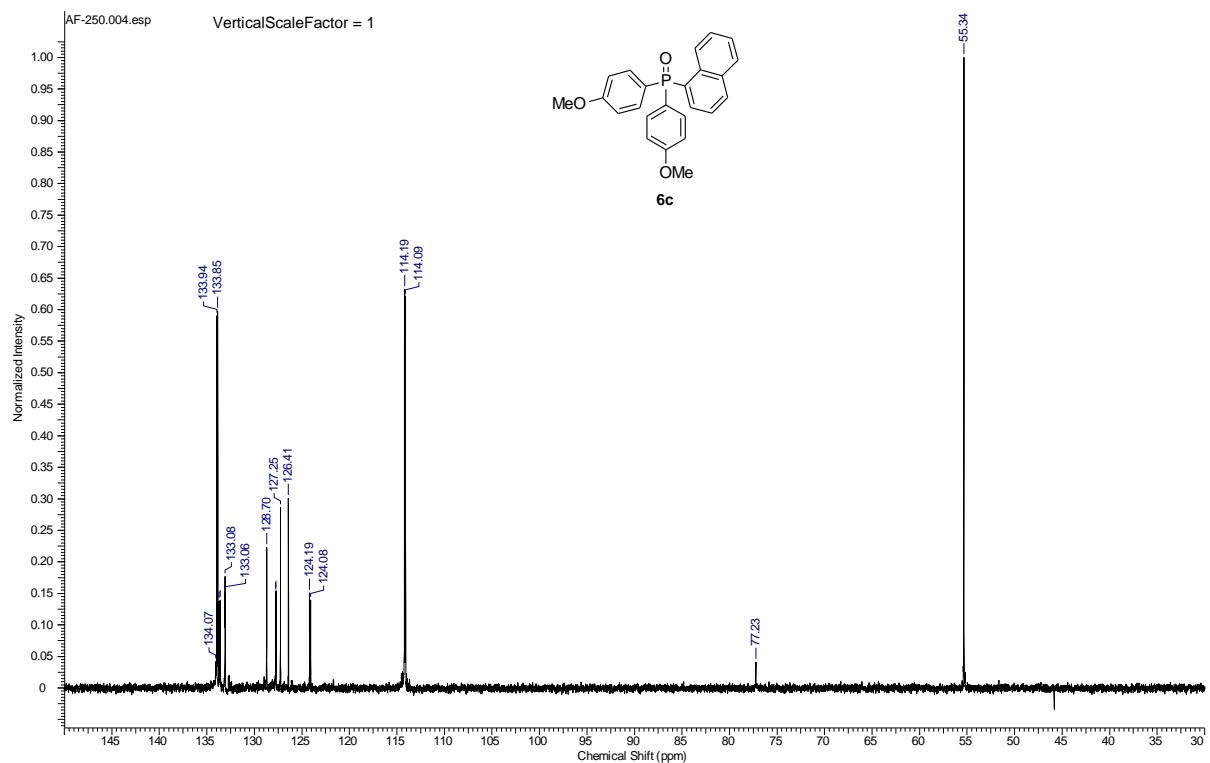


³¹P NMR spectra of bis(4-methoxyphenyl)(1-naphthyl)phosphine oxide (**6c**) (CDCl₃, 202 MHz).



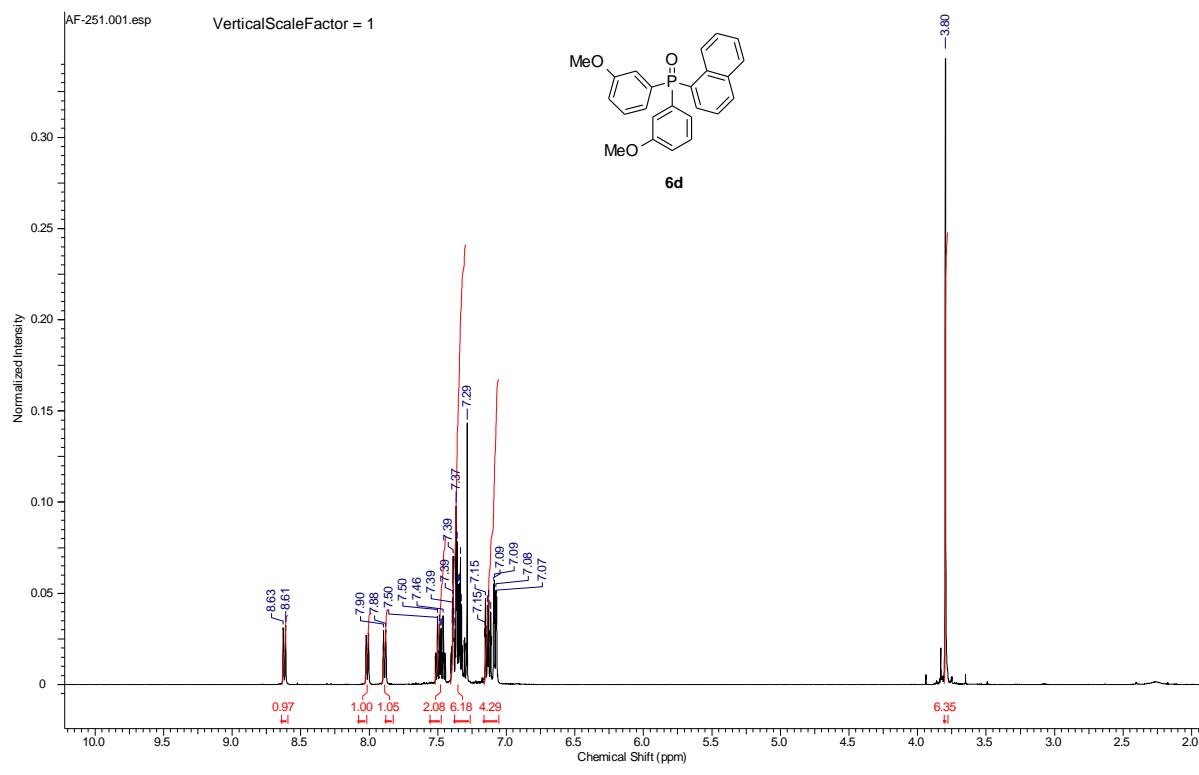
E:\FTP\NMR\ELA LASTAWIECKA\AF-250AF-250.003.esp

³¹C NMR spectra of bis(4-methoxyphenyl)(1-naphthyl)phosphine oxide (**6c**) (CDCl₃, 126 MHz).



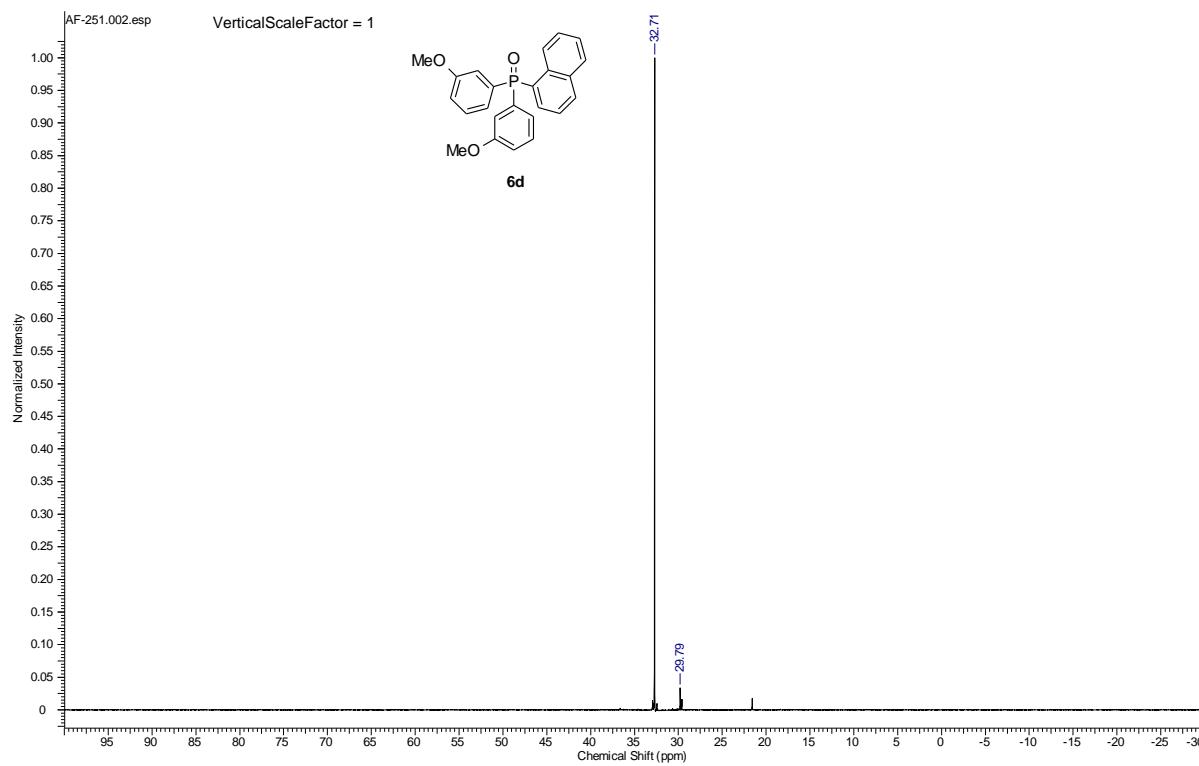
E:\FTP\NMR\ELA LASTAWIECKA\AF-250AF-250.004.esp

DEPT 135 spectra of bis(4-methoxyphenyl)(1-naphthyl)phosphine oxide (**6c**) (CDCl₃, 126 MHz).



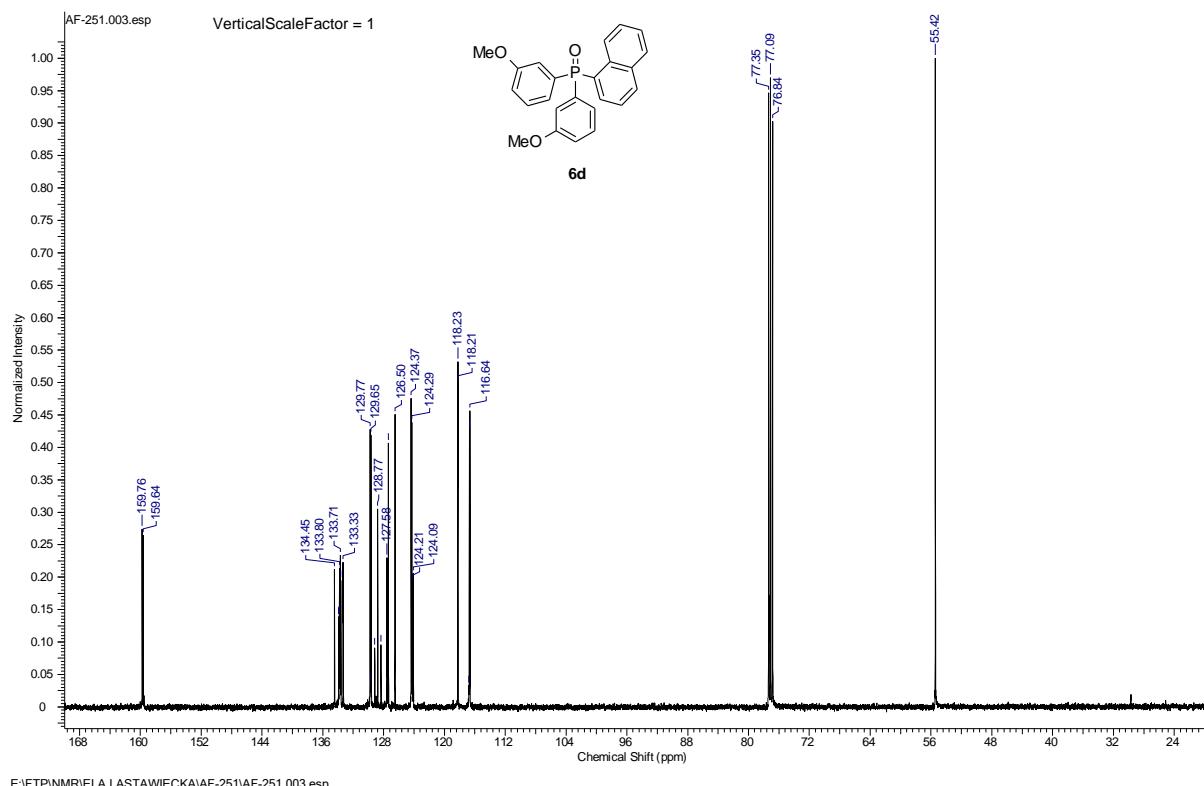
E:\FTP\NMR\ELA LASTAWIECKA\AF-251AF-251.001.esp

¹H NMR spectra of bis(3-methoxyphenyl)(1-naphthyl)phosphine oxide (**6d**) (CDCl₃, 500 MHz).

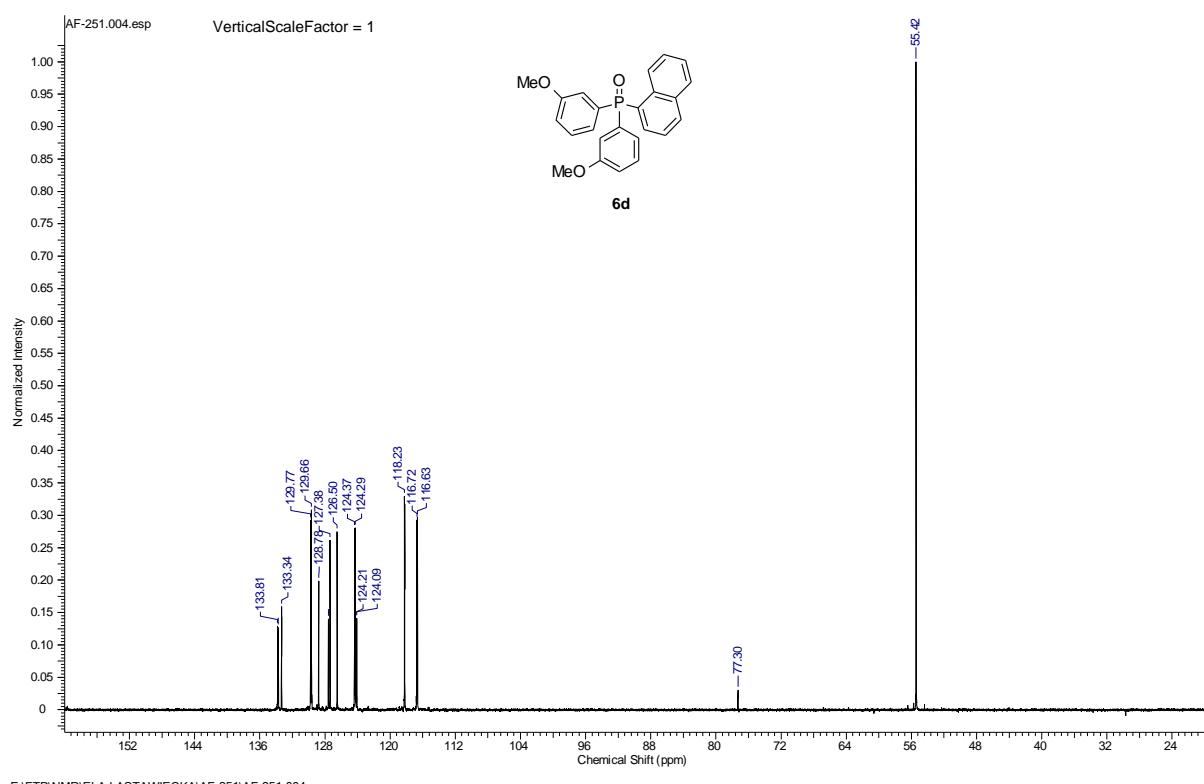


E:\FTP\NMR\ELA LASTAWIECKA\AF-251AF-251.002.esp

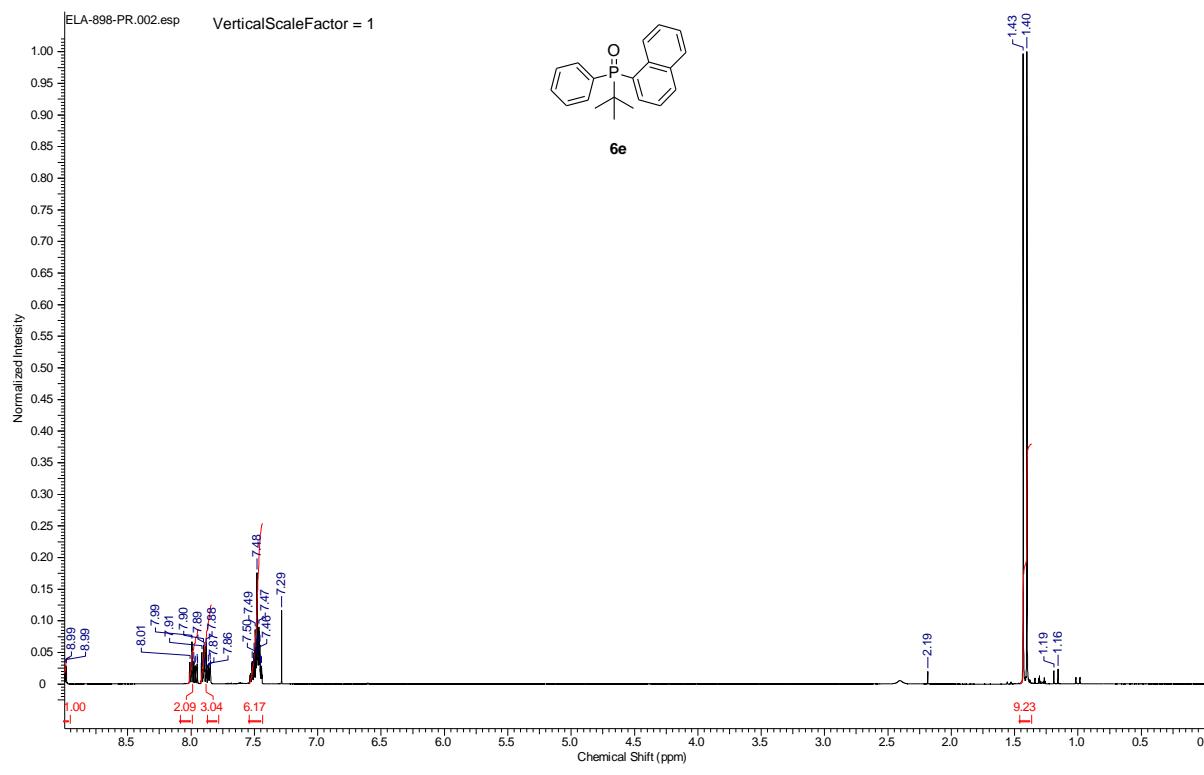
³¹P NMR spectra of bis(3-methoxyphenyl)(1-naphthyl)phosphine oxide (**6d**) (CDCl₃, 202 MHz).



³¹C NMR spectra of bis(3-methoxyphenyl)(1-naphthyl)phosphine oxide (**6d**) (CDCl₃, 126 MHz).

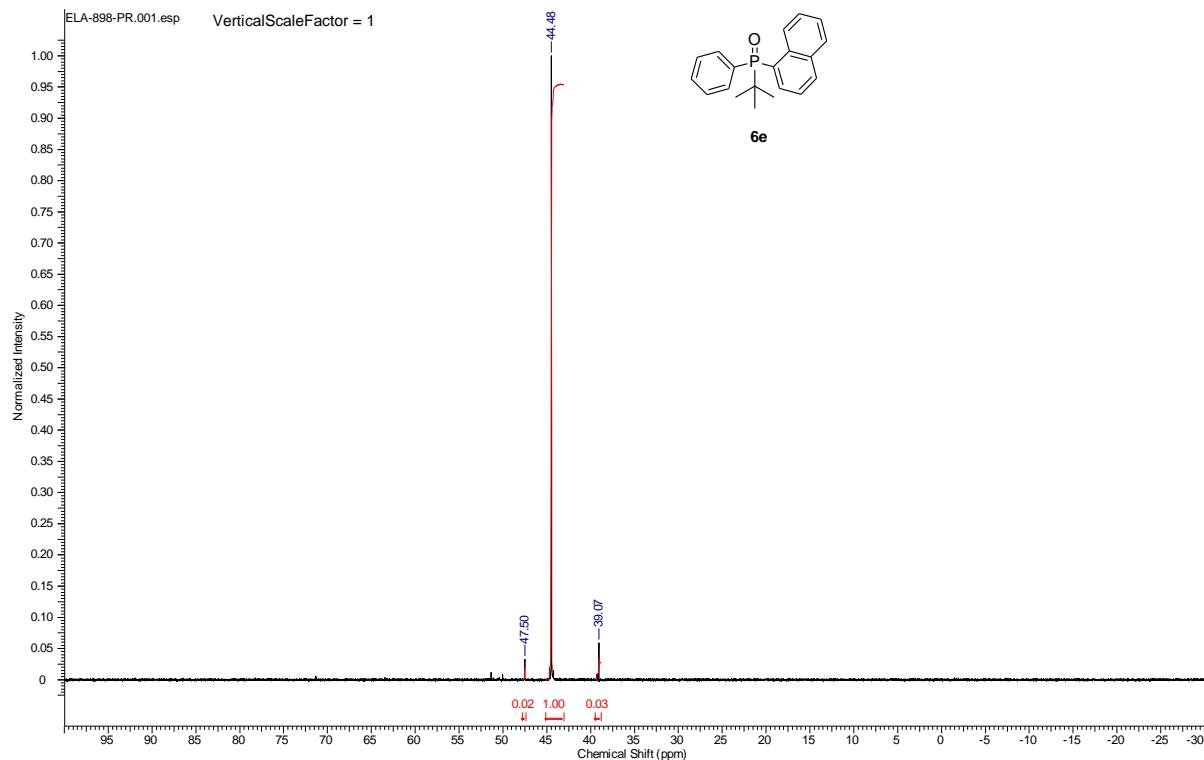


DEPT 135 spectra of bis(3-methoxyphenyl)(1-naphthyl)phosphine oxide (**6d**) (CDCl₃, 126 MHz).



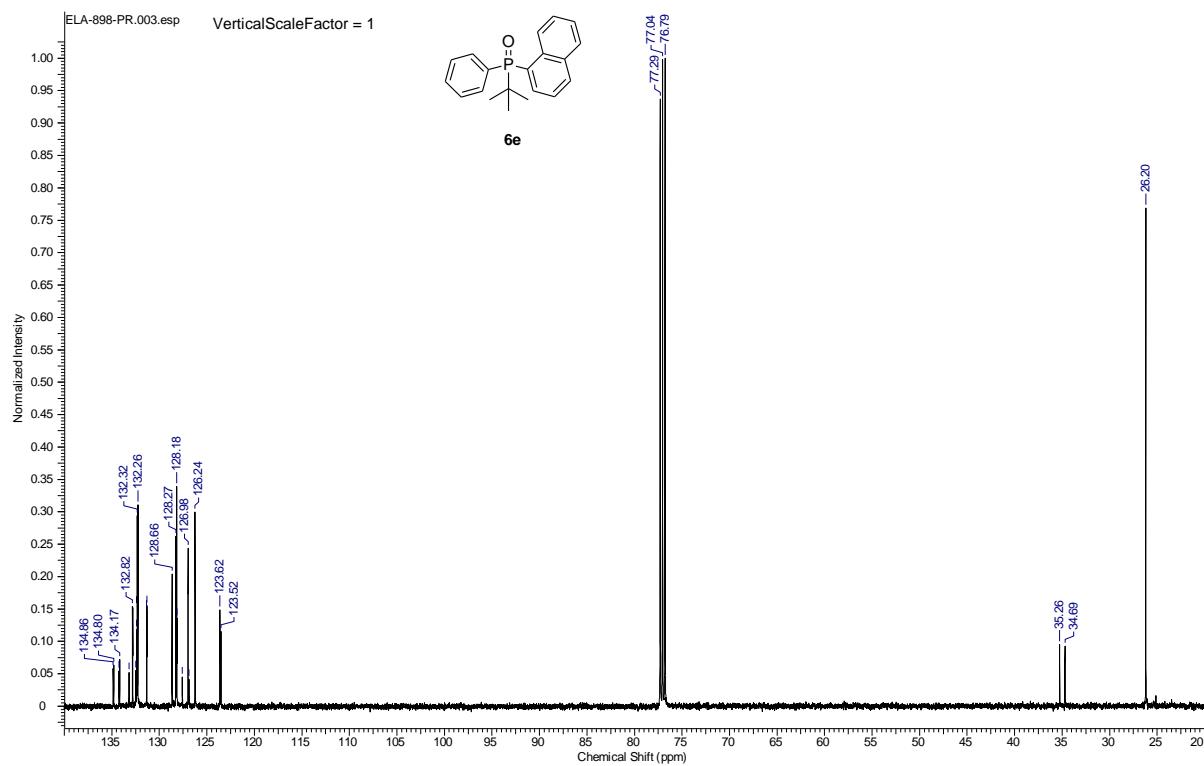
E:\FTP\NMR\ELA LASTAWIECKA\ELA-898-PR\ELA-898-PR.002.esp

¹H NMR spectra of *tert*-butyl(1-naphthyl)phenylphosphine oxide (**6e**) (CDCl₃, 500 MHz).



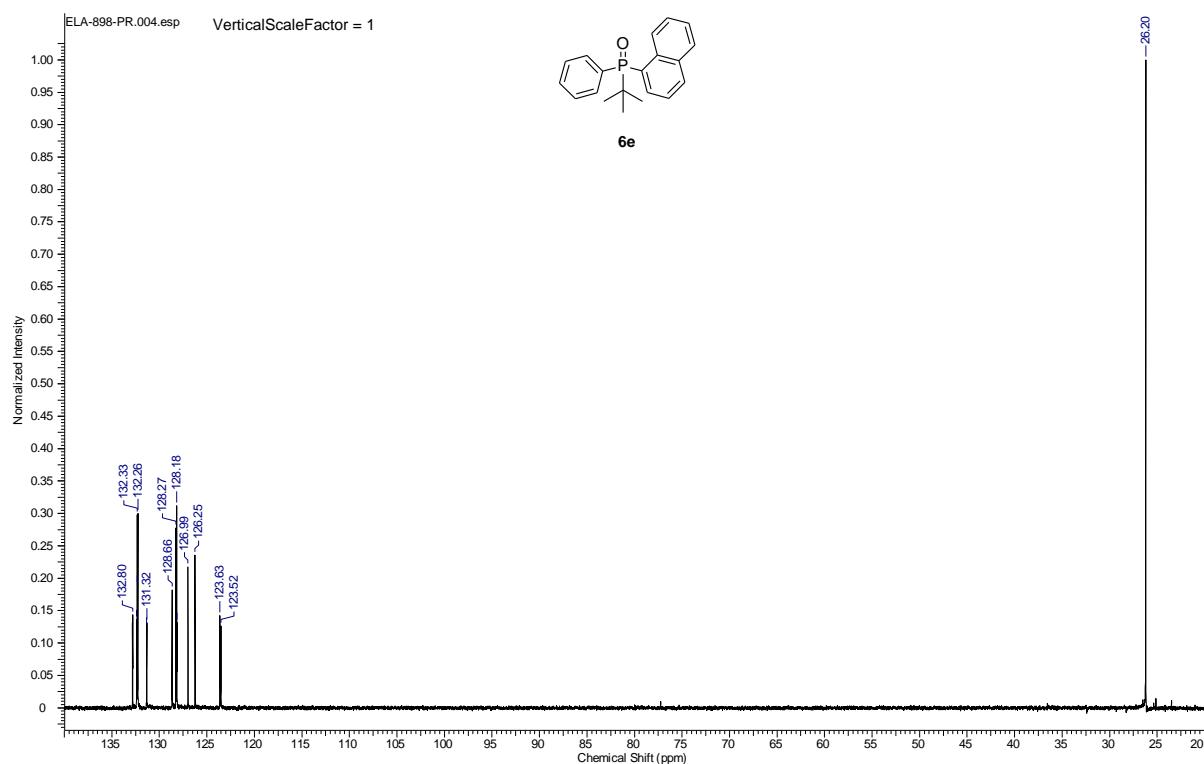
E:\FTP\NMR\ELA LASTAWIECKA\ELA-898-PR\ELA-898-PR.001.esp

³¹P NMR spectra of *tert*-butyl(1-naphthyl)phenylphosphine oxide (**6e**) (CDCl₃, 202 MHz).



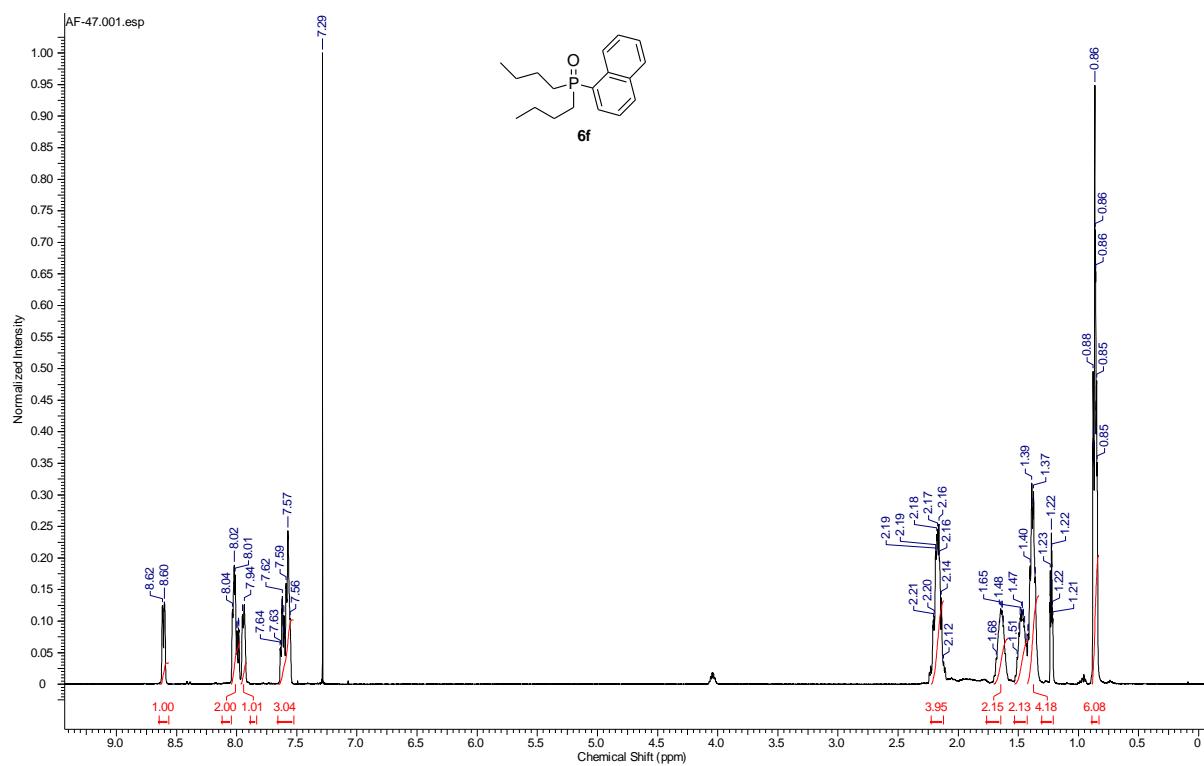
E:\FTP\NMR\ELA LASTAWIECKA\ELA-898-PR\ELA-898-PR.003.esp

³¹C NMR spectra of *tert*-butyl(1-naphthyl)phenylphosphine oxide (**6e**) (CDCl₃, 126 MHz).

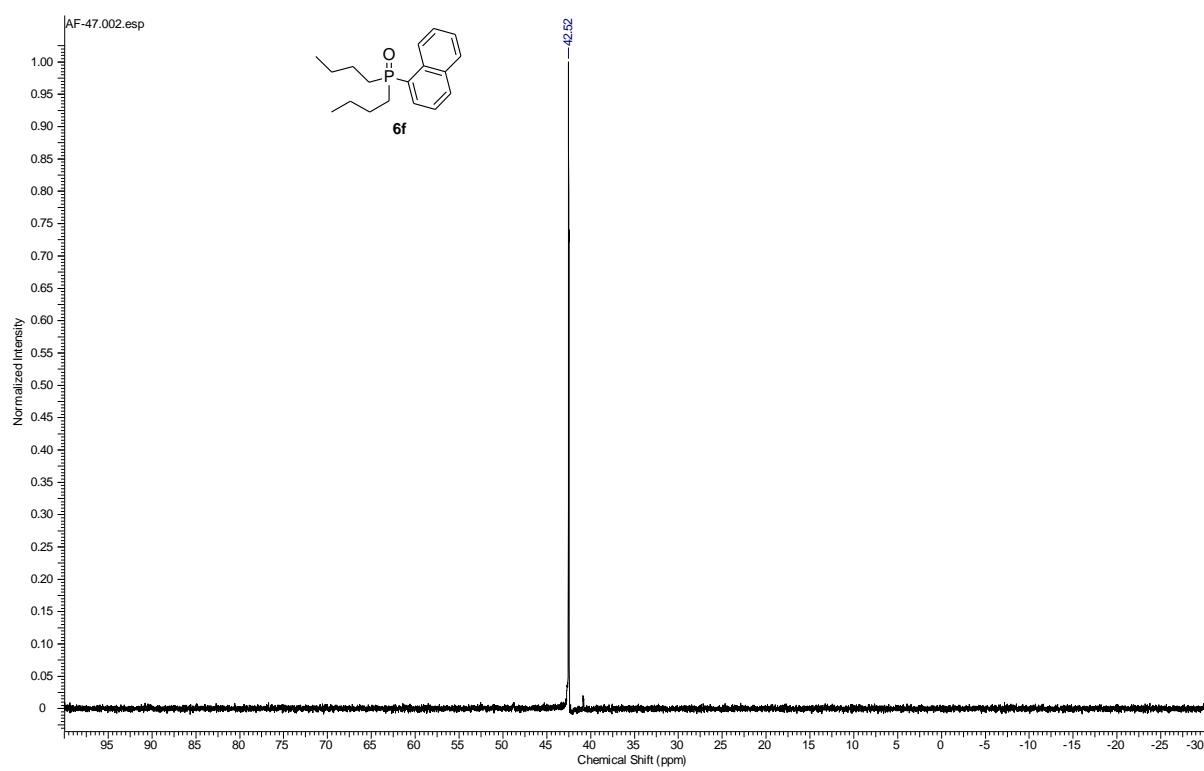


E:\FTP\NMR\ELA LASTAWIECKA\ELA-898-PR\ELA-898-PR.004.esp

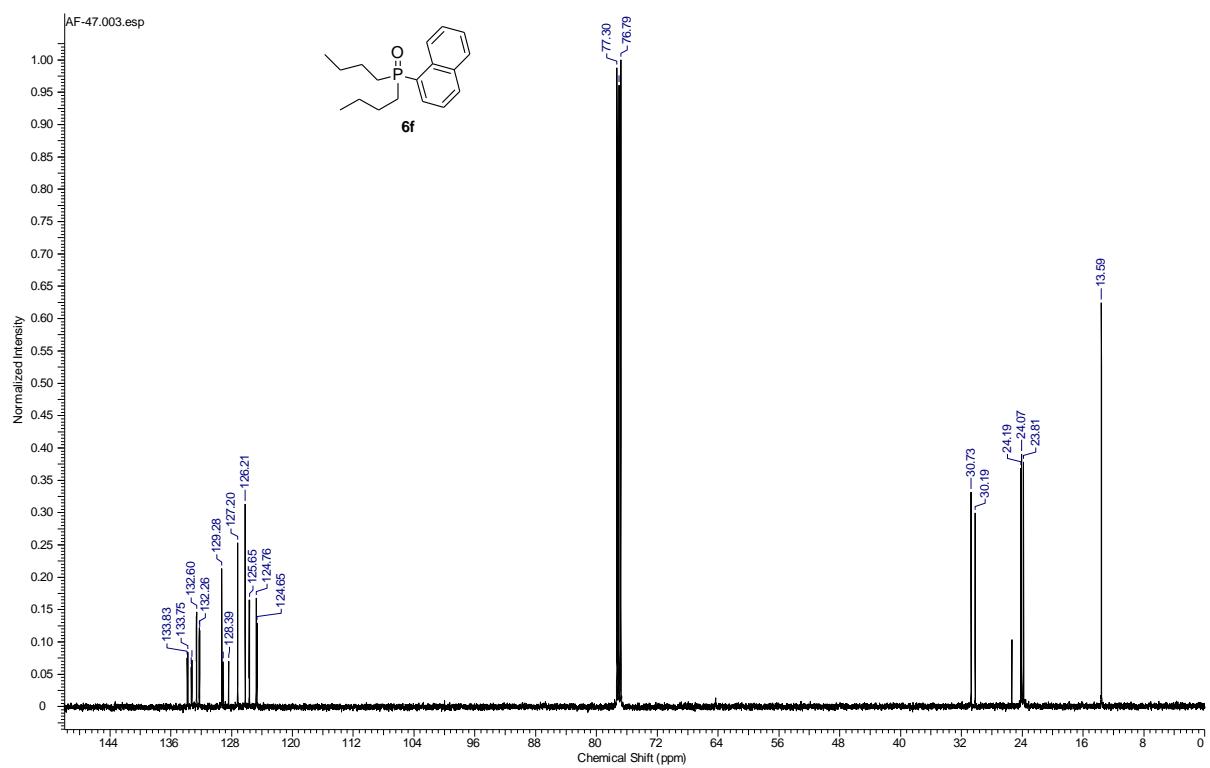
DEPT 135 spectra of *tert*-butyl(1-naphthyl)phenylphosphine oxide (**6e**) (CDCl₃, 126 MHz).



¹H NMR spectra of di(*n*-butyl(1-naphthyl))phosphine oxide (**6f**) (CDCl₃, 500 MHz)



³¹P NMR spectra of di(*n*-butyl(1-naphthyl))phosphine oxide (**6f**) (CDCl_3 , 202 MHz)



³¹C NMR spectra of di(*n*-butyl(1-naphthyl))phosphine oxide (**6f**) (CDCl₃, 126 MHz).