Electronic Supplementary Information

Title: Spontaneous resolution upon crystallization of allenyl-bis-phosphine oxides

Authors: G. Gangadhararao, R. N. Prasad Tulichala and K. C. Kumara Swamy*

School of Chemistry, University of Hyderabad, Hyderabad 500 046, Telangana, India. E-mail: kckssc@uohyd.ac.in, kckssc@yahoo.com; Fax: (+91)-40-23012460.

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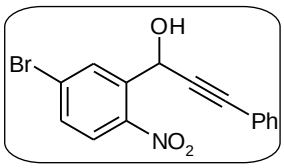
General Methods: All reactions were performed under nitrogen atmosphere unless stated otherwise. Chemicals/solvents were purified as required using standard procedures,[1] unless otherwise noted. Chlorodiphenylphosphine (Ph_2PCl), procured from Aldrich, was distilled prior to use. ^1H, ^{13}C and ^{31}P NMR spectra were recorded using a 400 MHz spectrometer in $CDCl_3$ (unless stated otherwise) with shifts referenced to $SiMe_4$ ($\delta = 0$) or 85 % H_3PO_4 ($\delta = 0$). All *J* values are in Hz. IR spectra were recorded neat or by using KBr pellets on a JASCOFT/IR 5300 spectrometer. Melting points were determined by using a local hot stage melting point apparatus and are uncorrected. Elemental analyses were carried out on a Perkin-Elmer 240C CHN or Thermo Finnigan EA1112 CHNS analyzer. For TLC, glass microslides were coated with silica-gel-GF_{254} (mesh size 75μ) and spots were identified using iodine or UV chamber as appropriate. For column chromatography, silica gel of100-200 mesh size was used. LC-MS data were obtained using electrospray ionization (positive mode) on a C-18 column. Mass spectra were recorded using HRMS (ESI-TOF analyzer) equipment.X-ray data were collected at 293 K on a Bruker AXS-SMART or on an OXFORD diffractometer using Mo-K_α radiation (λ = 0.71073 Å). Structures were solved and refined using standard methods.[2]

1 Synthesis of propargyl alcohols [2a-f, 3a-c, 4, 16 and 18]

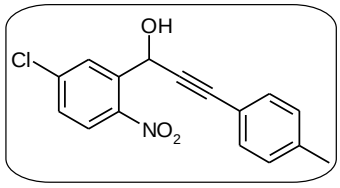
Propargyl alcohols **2-4**, **16**and **18** were prepared according to the known literature methods.[3] Among these **2a-b**[3], **2d**[3], **3a-c**[3], **16**[4] and **18**[3]areknown.

Compound 2c



In a round bottom flask (50 mL) equipped with phenylacetylene (0.42 mL, 3.8mmol), and THF (20 mL), *n*-BuLi (3.5 mL, 5.6 mmol) was added drop-wise *via* syringe at -30 ^{o}C. Then the mixture was stirred at -30 ^{o}C for 0.5 h. This was followed by the drop-wise addition of 5-bromo-2-nitro-benzaldehyde[5] (0.8 g, 3.48 mmol) dissolved in THF (10 mL) at -20 ^{o}C over a period of 15 min. After completion of addition, the mixture was warmed to 0 ^{o}C and stirring continued for 1-2 h. Then it was quenched with saturated NH_4Cl solution, the solvent removed and extracted with diethyl ether. The organic layer was washed with brine solution followed by drying the organic layer with anh. Na_2SO_4. The product **2c** was purified by column chromatography using silica gel with ethyl acetate-hexane (1:4) as the eluent.Yellow solid; Yield 1.04 g (90%); mp 68–70 ^{o}C; IR (KBr, cm^{-1}) 3222, 2953, 2926, 2849, 2219, 1605, 1562, 1518, 1348, 1293, 1173, 1074, 1036, 975, 899; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 1.6 Hz, 1H, Ar-*H*), 7.87 (d, *J* = 8.4 Hz, 1H, Ar-*H*), 7.62 (dd, *J* = 8.8 Hz, *J* = 2.0 Hz, 1H, Ar-*H*), 7.44-7.27 (m, 5H, Ar-*H*), 6.26 (s, 1H, C*H*OH), 3.46 (br, 1H, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 137.5, 132.4, 132.3, 131.9, 129.0, 128.9, 128.4, 126.6, 121.7 (Ar-*C*), 87.1 (*C*≡C), 86.1 (C≡*C*), 61.2 (*C*HOH); LC-MS *m/z* 332 $[M]^+$; Anal. Calcd. for $C_{15}H_{10}BrNO_3$:C, 54.24; H, 3.03; N, 4.22. Found: C, 54.32; H, 3.09; N, 4.28.

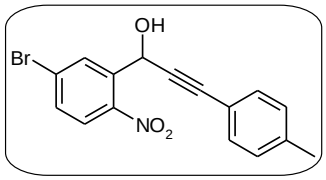
Compound 2e



Procedure was similar to that for compound **2c** using 4-ethynyl toluene (0.79 mL, 6.25 mmol) and 5-chloro-2-nitro-benzaldehyde[5] (1.05 g, 5.68 mmol). Brown solid; yield 1.49 g (87%); mp 70–72 ^{o}C; IR (KBr, cm^{-1}) 3260, 2964, 2921, 2866, 2219, 1605, 1573, 1523, 1353, 1288, 1178,

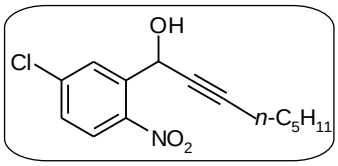
1112, 1079, 1041, 981, 877; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (dd→t, 1H, Ar-*H*), 7.94 (dd, *J* = 8.8 Hz, *J* = 2.4 Hz, 1H, Ar-*H*), 7.46-7.10 (m, 5H, Ar-*H*), 6.26 (d, *J* = 2.4 Hz, 1H, C*H*OH), 3.54 (br, 1H, O*H*), 2.34 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.0, 140.3, 139.2, 137.7, 131.8, 129.3, 129.2, 129.1, 126.6, 118.6 (Ar-*C*), 87.3 (*C*≡C), 85.4 (C≡*C*), 61.3 (*C*HOH), 21.5 (*C*H_3); LC-MS *m/z* 302 $[M]^+$; Anal. Calcd. for $C_{16}H_{12}ClNO_3$:C, 63.69; H, 4.01; N, 4.64. Found: C, 63.58; H, 4.07; N, 4.58.

Compound 2f



Procedure was similar to that for compound **2c** using 4-ethnyl toluene (0.49 mL, 3.83 mmol) and 5-bromo-2-nitrobenzaldehyde (0.80 g, 3.48 mmol). Brown solid; yield 1.03 g (86%); mp 76–78 ^{o}C; IR (KBr, cm^{-1}) 3266, 2964, 2915, 2860, 2219, 1600, 1562, 1523, 1348, 1293, 1173, 1079, 1041, 970, 860; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17 (d, *J* = 1.6 Hz, 1H, Ar-*H*), 7.86 (d, *J* = 8.4 Hz, 1H, Ar-*H*), 7.62 (dd, *J* = 8.8 Hz, *J* = 2.0 Hz, 1H, Ar-*H*), 7.34-7.10 (m, 4H, Ar-*H*), 6.25 (s, 1H, C*H*OH), 3.42 (br, 1H, O*H*), 2.35 (s, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.6, 139.3, 137.6, 132.3, 131.8, 129.1, 128.8, 126.6, 118.6 (Ar-*C*), 87.4 (*C*≡C), 85.4 (C≡*C*), 61.3 (*C*HOH), 21.6 (*C*H_3); LC-MS *m/z* 346 $[M]^+$; Anal. Calcd. for $C_{16}H_{12}BrNO_3$:C, 55.51; H, 3.49; N, 4.05. Found: C, 55.62; H, 3.45; N, 4.12.

Compound 4

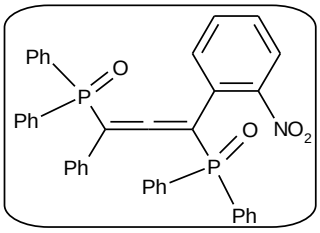


Procedure was similar to that for compound **2c** using1-heptyne (1.49 mL, 11.32 mmol) and 5-chloro-2-nitro-benzaldehyde (1.5 g, 8.08 mmol). Gummy liquid; yield 2.0 g (88%); IR (neat, cm^{-1}) 3414, 3101, 2959, 2926, 2855, 2225, 1611, 1573, 1523, 1468, 1342, 1293, 1178, 1140, 1107, 1074, 1014, 893; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.41 (m, 3H, Ar-*H*), 6.01 (s, 1H, C*H*OH), 3.27 (br, 1H, O*H*), 2.23 (t, 2H, CH_2), 1.51-1.27 (m, 6H, CH_2), 0.88 (t, 3H, CH_2CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 146.1, 140.2, 138.2, 129.3, 129.0, 126.5 (Ar-*C*), 88.7 (*C*≡C), 77.4 (C≡*C*), 60.9 (*C*HOH), 31.0, 28.0, 22.2 and 18.7 (CH_2), 14.0 (CH_3); LC-MS *m/z* 282 $[M]^+$; Anal. Calcd. for $C_{14}H_{16}ClNO_3$:C, 59.68; H, 5.72; N, 4.97. Found: C, 59.76; H, 5.69; N, 5.07.

2 Synthesis of allenyl-bis-phosphine oxides [6-15] and allenylphosphine oxides 17 and 19

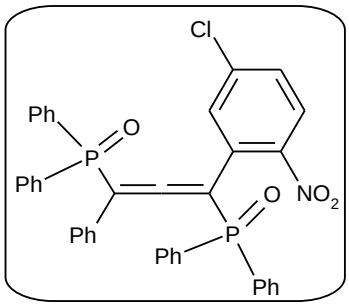
An oven dried 25 mL round-bottomed flask was charged with propargyl alcohol **2a** (0.34 g, 1.36 mmol), NEt_3 (0.4 mL, 2.85 mmol) and dry THF (5 mL). The mixture was stirred at 0 ^{o}C for 5 min. To this, **.1** (0.51 mL, 2.85 mmol) dissolved in dry THF (5 mL) was added drop-wise at 0 ^{o}C over a period of 15 min. Then the mixture was stirred at 0 ^{o}C for 1-2 h. Filtration followed by removal of the solvent and purification by column chromatography (hexane/ethyl acetate; 2:1) afforded **6** as a white solid. Similarly, compounds **7-15**, **17** and **19** were prepared.

Compound 6



Yield 0.797 g (92%); mp 176–178 ^{o}C; IR (KBr, cm^{-1}) 3057, 2924, 2853, 1935, 1738, 1593, 1526, 1435, 1346, 1198, 1117, 750; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 2H, Ar-*H*), 7.90 (dd, *J* = 7.6, 2.0 Hz, 1H, Ar-*H*), 7.58-7.19 (m, 25H, Ar-*H*), 6.77 (d, *J* = 7.2 Hz, 1H, Ar-*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.4 (C=*C*=C), 148.0, 132.9, 132.6, 132.5, 132.4, 132.2, 131.9, 131.7, 131.6, 130.9, 130.6, 130.3, 130.0, 129.3, 129.0, 128.8, 128.6, 128.5, 128.4, 128.2, 126.0, 124.8 (Ar-*C*), 104.9 (dd, ^{1}J(P-C) = 90.0 Hz, ^{3}J(P-C) = 12.9 Hz, P-*C*), 102.0 (dd, ^{1}J(P-C) = 90.7 Hz, ^{3}J(P-C) = 14.9 Hz, P-*C*); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.10 (d, *J* = 12.8 Hz), 26.83 (d, *J* = 12.8 Hz); LC-MS *m/z* 638 $[M+1]^{+}$; Anal. Calcd. for $C_{39}H_{29}NO_4P_2$: C, 73.47; H, 4.58; N, 2.20. Found: C, 73.65; H, 4.51; N, 2.28.

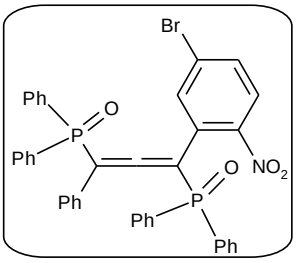
Compound 7



This compound was prepared by following a procedure similar to that for **6** using propargyl alcohol **2b** (0.31 g, 1.06 mmol). White solid; yield 0.653 g (91%); mp 164–166 ^{o}C; IR (KBr, cm^{-1}) 3052, 2926, 2855, 1940, 1600, 1529, 1441, 1337, 1200, 1112, 948; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96-7.91 (m, 2H, Ar-*H*), 7.80 (d, *J* = 8.8 Hz, 1H, Ar-*H*), 7.59-7.19 (m, 24H, Ar-*H*), 6.50 (1 s, 1H, Ar-*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.0 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.1, 139.2,

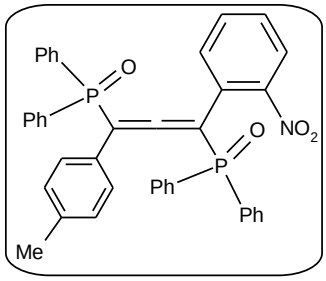
132.7, 132.5, 132.4, 132.3, 132.2, 132.1, 132.0, 131.6, 131.5, 129.3, 128.9, 128.8, 128.7, 128.6, 128.4, 128.3, 127.8, 126.0 (Ar-*C*), 105.5 (dd, ^{1}J(P-C) = 88.0 Hz, ^{3}J(P-C) = 13.0 Hz, P-*C*), 101.4 (dd, ^{1}J(P-C) = 91.0 Hz, ^{3}J(P-C) = 14.0 Hz, P-*C*); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.14 (d, *J* = 13.5 Hz), 26.80 (d, *J* = 13.5 Hz); HRMS (ESI):Calcd. for $C_{39}H_{28}ClNO_4P_2$ (M^+ + H and M^+ + H +2): *m/z* 672.1261 and 674.1261. Found: 672.1260 and 674.1245.

Compound 8



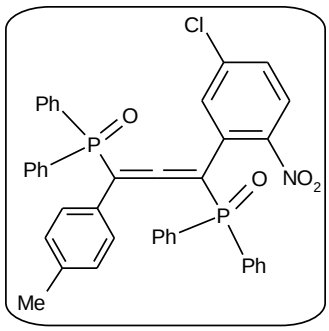
This compound was prepared by following a route similar to that for **6** using propargyl alcohol **2c** (0.32 g, 0.96 mmol).White solid; yield 0.595 g (86%); mp 158–160 ^{o}C; IR (KBr, cm^{-1}) 3052, 2926, 2849, 1934, 1595, 1562, 1529, 1436, 1347, 1205, 1118, 1074, 926; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95-7.90 (m, 2H, Ar-*H*), 7.74 (d, *J* = 8.8 Hz, 1H, Ar-*H*), 7.60-7.20 (m, 24H, Ar-*H*), 6.70 (1 s, 1H, Ar-*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.1 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.7, 135.1, 132.7, 132.6, 132.5, 132.4, 132.3, 132.2, 132.1, 131.9, 131.7_0, 131.6_6, 131.6, 131.4, 131.3, 130.9, 130.6, 130.3, 130.0_8, 130.0_5, 129.8_2, 129.7_7, 129.7, 129.0_1, 128.9_8, 128.9, 128.7_9, 128.7_5, 128.7, 128.5, 128.3, 127.9, 127.7, 126.1 (Ar-*C*), 105.6 (dd, ^{1}J(P-C) = 90.0 Hz, ^{3}J(P-C) = 12.5 Hz, P-*C*), 101.3 (dd, ^{1}J(P-C) = 91.5 Hz, ^{3}J(P-C) = 13.5 Hz, P-*C*); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.23 (d, *J* = 13.0 Hz), 26.83 (d, *J* = 13.0 Hz); HRMS (ESI): Calcd. for $C_{39}H_{28}BrNO_4P_2$ (M^+ + H and M^+ + H +2): *m/z* 716.0756 and 718.0756. Found: 716.0777 and 718.0766.

Compound 9



This compound was prepared by following a procedure similar to that for **6** using propargyl alcohol **2d** (0.31 g, 1.16 mmol).White solid; Yield: 0.711 g (94%); mp 190–192 ^{o}C; IR (KBr, cm^{-1}) 3052, 2997, 2953, 2915, 1934, 1600, 1573, 1523, 1436, 1348, 1195, 1123, 997, 904; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 2H, Ar-*H*), 7.87 (dd, *J* = 6.8 Hz, *J* = 2.4 Hz, 1H, Ar-*H*), 7.60-7.01 (m, 24H, Ar-*H*), 6.83 (d, *J* = 6.8 Hz, 1H, Ar-*H*), 2.29 (s, 3H, C*H*$_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.5 (t, *J* = 6.0 Hz, C=*C*=C), 148.1, 138.4, 132.8, 132.6, 132.5, 132.4, 132.2, 132.1, 131.8, 131.7, 131.6, 131.0_3, 130.9_7, 130.8, 130.4, 129.4, 129.2, 128.8, 128.7_1, 128.6_6, 128.5_9, 128.5_5, 128.3, 128.2, 126.8, 126.1, 124.7 (Ar-*C*), 104.9 (dd, ^{1}J(P-C) = 95.0 Hz, ^{3}J(P-C) = 8.0 Hz, P-*C*), 101.8 (dd, ^{1}J(P-C) = 97.5 Hz, ^{3}J(P-C) = 9.5 Hz, P-*C*), 21.3 (*C*H$_3$); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.01 and 26.84 (AB quartet, *J* ~ 13.0 Hz); HRMS (ESI): Calcd. for $C_{40}H_{31}NO_4P_2$ (M^+ + H): *m/z* 652.1807. Found: 652.1804.

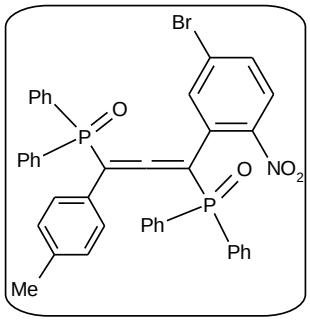
Compound 10



Procedure was similar to that for compound **6** using propargyl alcohol **2e** (0.35 g, 1.16 mmol).White solid; yield 0.713 g (89%); mp 180–182 ^{o}C; IR (KBr, cm^{-1}) 3058, 2926, 2849,

1918, 1600, 1556, 1523, 1436, 1342, 1195, 1112, 937; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.94-7.89 (m, 2H, Ar-*H*), 7.75 (d, *J* = 8.4 Hz, 1H, Ar-*H*), 7.56-7.00 (m, 23H, Ar-*H*), 6.55 (1 s, 1H, Ar-*H*), 2.24 (1 s, 3H, C*H*$_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.0 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.1, 139.0, 138.5, 132.5, 132.3, 132.2_4, 132.1_5, 132.0, 131.9, 131.7, 131.5, 131.4, 131.2, 130.7, 130.5, 130.2, 129.3, 129.2, 128.9, 128.7, 128.5, 128.3, 128.2, 127.7, 126.4, 125.9 (Ar-*C*), 105.3 (dd, ^{1}J(P-C) = 90.0 Hz, ^{3}J(P-C) = 12.0 Hz, P-*C*), 101.0 (dd, ^{1}J(P-C) = 93.0 Hz, ^{3}J(P-C) = 13.0 Hz, P-*C*), 21.1 (Ph*C*H$_3$); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.25 (d, *J* ~ 13.0 Hz), 26.76 (d, *J* ~ 13.0 Hz); HRMS (ESI): Calcd. for $C_{40}H_{30}ClNO_4P_2$ (M^+ + Na and M^+ + Na +2): *m/z* 708.1237 and 710.1237. Found: 708.1236 and 710.1219.

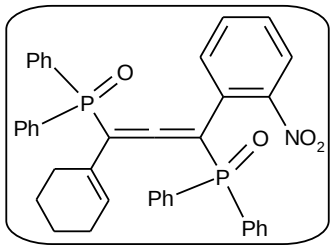
Compound 11



This compound was prepared by following a procedure similar to that for **6** using propargyl alcohol **2f** (0.30 g, 0.86 mmol).White solid; yield 0.577 g (91%); mp 184–186 ^{o}C; IR (KBr, cm^{-1}) 3052, 2921, 2855 1912, 1595, 1556, 1523, 1436, 1348, 1200, 1118, 932; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91-7.02 (m, 26H, Ar-*H*), 6.74 (1 s, 1H, Ar-*H*), 2.24 (1 s, 3H, C*H*$_3$); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.0 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.6, 138.5, 134.9, 132.5, 132.3_4, 132.2_5, 132.2, 132.0, 131.9, 131.7, 131.5, 131.4, 131.3, 130.7, 130.5, 130.2, 130.0, 129.3, 128.9, 128.7, 128.6, 128.3, 128.2, 127.8, 127.5, 126.4, 125.9 (Ar-*C*), 105.3 (dd, ^{1}J(P-C) = 90.0 Hz, ^{3}J(P-C) = 12.0 Hz, P-*C*), 100.9 (dd, ^{1}J(P-C) = 93.5 Hz, ^{3}J(P-C) = 12.5 Hz, P-*C*), 21.1 (Ph*C*H$_3$); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.41 (d, *J* = 13.1 Hz), 26.79 (d, *J* = 13.1 Hz); HRMS (ESI): Calcd. for

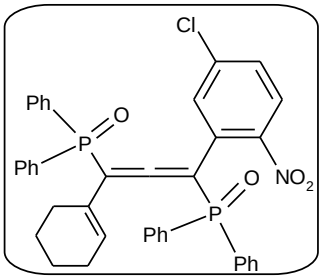
$C_{40}H_{30}BrNO_4P_2$ (M^+ + Na and M^+ + Na +2); *m/z* 752.0731 and 754.0731. Found: 752.0733 and 754.0723.

Compound 12



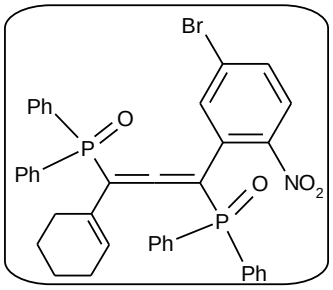
This compound was prepared by following a route similar to that for **6** using propargyl alcohol **3a** (0.45 g, 1.75 mmol).White solid; yield 1.061 g (94%); mp 180–182 ^oC; IR (KBr, cm^{-1}) 3052, 2921, 2855, 1918, 1611, 1573, 1529, 1479, 1436, 1342, 1195, 1118, 997; ^1H NMR (400 MHz, $CDCl_3$) δ 8.02-7.97 (m, 2H, Ar-*H*), 7.85 (d, *J* = 7.6 Hz, 1H, Ar-*H*), 7.67-7.29 (m, 20H, Ar-*H*), 6.77 (d, *J* = 7.2 Hz, 1H, Ar-*H*), 5.76 (br s, 1H, =C*H*), 1.94-1.89 (m, 4H, CH_2), 1.55-1.45 (m, 4H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 210.1 (t, *J* ~ 6.0 Hz, C=*C*=C), 148.0, 132.6, 132.5_1, 132.4_7, 132.4, 132.3, 132.2, 131.9, 131.8, 131.7, 131.6, 131.5, 131.4, 131.3, 130.8, 129.7, 128.9, 128.5, 128.4, 128.2, 128.1, 127.8, 126.4, 124.5 (Ar-*C*), 106.3 (dd, ^1J(P-C) = 90.5 Hz, ^3J(P-C) = 11.5 Hz, P-*C*), 101.8 (dd, ^1J(P-C) = 95.0 Hz, ^3J(P-C) = 13.0 Hz, P-*C*), 28.4, 25.9, 22.6 and 21.4 (*C*H_2); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.12 (d, *J* = 13.8 Hz), 25.93 (d, *J* = 13.8 Hz); HRMS (ESI): Calcd. for $C_{39}H_{33}NO_4P_2$ (M^+ + H): *m/z* 642.1964. Found 642.1964. After obtaining the crystals [ethyl acetate + chloroform in the v/v ratio ~1:1)], the two enantiomers (*R* and *S*) were separated by hand picking, based on slightly different morphology. For solid state CD spectra, two/ three single crystals having similar morphology were combined, and made into KBr pellet. Combination of single crystals was done several times to get the best CD spectra. For the other enantiomer also, a similar procedure was adapted.

Compound 13



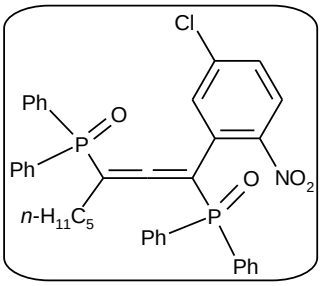
This compound was prepared by following a procedure similar to that for **6** using propargyl alcohol **3b** (0.36 g, 1.22 mmol).White solid; yield 0.752 g (91%); mp 148–150 ^{o}C; IR (KBr, cm^{-1}) 3058, 2926, 2860, 1907, 1595, 1562, 1534, 1441, 1342, 1195, 1112, 932; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 2H, Ar-*H*), 7.76 (d, *J* = 8.8 Hz, 1H, Ar-*H*), 7.64-7.30 (m, 19H, Ar-*H*), 6.49 (1 s, 1H, Ar-*H*), 5.84 (br s, 1H, =C*H*), 1.93 (br, 4H, CH_2), 1.55-1.45 (m, 4H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.7 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.1, 139.0, 132.6, 132.4, 132.3, 132.2_4, 132.1_5, 132.1, 132.0, 131.9, 131.7, 131.6, 131.4, 131.3_2, 131.2_8, 131.0, 130.5, 129.5, 129.1, 128.7, 128.6_7, 128.6_5, 128.5_9, 128.5_5, 128.5, 128.4, 128.3, 128.2, 127.6_3, 127.5_8, 127.5, 125.9 (Ar-*C*), 107.1 (dd, ^{1}J(P-C) = 90.0 Hz, ^{3}J(P-C) = 11.2 Hz, P-*C*), 101.2 (dd, ^{1}J(P-C) = 95.5 Hz, ^{3}J(P-C) = 12.5 Hz, P-*C*), 28.5, 26.1, 22.7 and 21.5 (*C*H_2); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.55 (d, *J* = 13.4 Hz), 25.59 (d, *J* = 13.4 Hz); HRMS (ESI): Calcd. for $C_{39}H_{32}ClNO_4P_2$ (M^+ + Na and M^+ + Na +2): *m/z* 698.1393 and 700.1393. Found: 698.1391 and 700.1373.

Compound 14



Procedure was similar to that for compound **6** using propargyl alcohol **3c** (0.22 g, 0.65 mmol).White solid; yield 0.433 g (93%); mp 188–190 ^{o}C; IR (KBr, cm^{-1}) 3052, 2926, 2855, 1912, 1595, 1562, 1534, 1436, 1353, 1205, 1123, 932; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99-7.94 (m, 2H, Ar-*H*), 7.70-7.31 (m, 20H, Ar-*H*), 6.69 (1 s, 1H, Ar-*H*), 5.85 (br s, 1H, =C*H*), 1.93 (br, 4H, CH_2), 1.55-1.46 (m, 4H, CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.7 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.7, 134.9, 132.5, 132.3, 132.2, 132.1$_2$, 132.0$_6$, 132.0, 131.9, 131.8, 131.6, 131.5$_4$, 131.4$_8$, 131.4, 131.3, 131.2, 130.9, 130.4, 129.4, 128.7, 128.6, 128.5$_3$, 128.4$_9$, 128.3, 128.2, 127.5, 127.4, 125.9 (Ar-*C*), 107.0 (dd, ^{1}J(P-C) = 90.0 Hz, ^{3}J(P-C) = 11.0 Hz, P-*C*), 101.0 (dd, ^{1}J(P-C) = 95.5 Hz, ^{3}J(P-C) = 12.5 Hz, P-*C*), 28.5, 26.0, 22.6 and 21.4 (*C*H$_2$); ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.58 (d, *J* = 13.4 Hz), 25.64 (d, *J* = 13.4 Hz); HRMS (ESI): Calcd. for $C_{39}H_{32}BrNO_4P_2$ (M^+ + Na and M^+ + Na +2): *m/z* 742.0888 and 744.0888. Found: 742.0889 and 744.0880.

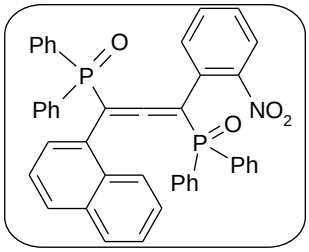
Compound 15



This compound was prepared by following a procedure similar to that for **6** using propargyl alcohol **4** (0.45 g, 1.61 mmol).White solid; yield 0.946 g (88%); mp 124–126 ^{o}C; IR (KBr, cm^{-1}) 3063, 2959, 2926, 2849, 1934, 1595, 1567, 1523, 1468, 1436, 1353, 1195, 1129, 1003, 937; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.04-7.99 (m, 2H, Ar-*H*), 7.80 (d, *J* = 8.8 Hz, 1H, Ar-*H*), 7.67-7.30 (m, 19H, Ar-*H*), 6.86 (1 s, 1H, Ar-*H*), 2.08-2.01 (m, 2H, CH_2), 1.27-1.07 (m, 6H, CH_2), 0.78 (t, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.0 (t, *J* ~ 6.0 Hz, C=*C*=C), 146.6, 139.1, 132.7,

132.6, 132.5, 132.4, 132.2_2, 132.2_0, 132.1_4, 132.1_1, 131.9, 131.8, 131.7, 131.6, 131.5, 131.4, 131.2, 130.8, 130.5, 130.3, 130.1, 129.4, 129.1, 128.8, 128.7, 128.5, 128.4, 126.0 (Ar-*C*), 102.8 (dd, ^1J(P-C) = 90.5 Hz, ^3J(P-C) = 10.5 Hz, P-*C*), 100.1 (dd, ^1J(P-C) = 97.0 Hz, ^3J(P-C) = 13.0 Hz, P-*C*), 31.7, 28.7, 28.3 and 22.2 (CH_2), 13.9 (CH_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.27 (d, *J* = 13.8 Hz), 26.53 (d, *J* = 13.8 Hz); HRMS (ESI): Calcd. for $C_{38}H_{34}ClNO_4P_2$ (M^+ + Na and M^+ + Na +2): *m/z* 688.1550 and 690.1550. Found: 688.1551 and 690.1533.

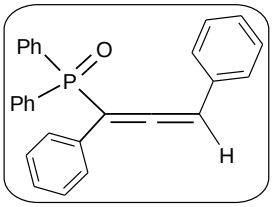
Compound A



3-Naphthalen-1-yl-1-(2-nitro-phenyl)-prop-2-yn-1-ol [IR (KBr, cm^{-1}) 3267, 2924, 2854, 2220, 1609, 1522, 1396, 1345, 1289, 1182, 1099, 1011, 947, 859, 777; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 8.0 Hz, 1H, Ar-*H*), 8.12 (d, *J* = 8.0 Hz, 1H, Ar-*H*), 8.04 (d, *J* = 8.0 Hz, 1H, Ar-*H*), 7.87 (d, *J* = 8.0 Hz , 2H, Ar-*H*), 7.74-7.71 (m, 4H, Ar-*H*), 7.62-7.42 (m, 4H, Ar-*H*), 6.41 (s, 1H, C*H*OH), 3.47 (s, 1H, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 147.7, 135.5, 133.8, 133.1, 132.9, 130.9, 129.2, 129.1_2, 128.2, 126.9, 126.4, 125.8, 125.0, 124.9, 119.5, 91.6, 84.7,61.8] (2.0g, 6.5 mmol) was used. The reaction mixture soon after the column showed Compound **A** and its P(V)-P(III) precursor [ó(P): 25.6, -0.4] in the ratio ~1:4. Over a period of ca 12 h, in air complete conversion to **A** could be effected. Yield: 4.08 g (90%; light yellow solid); mp 148-150 ^{o}C; IR (KBr, cm^{-1}) 3057, 2924, 2852, 1926, 1639, 1573, 1522, 1436, 1344, 1192, 1118, 745, 698; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01-7.95 (m, 2H, Ar-*H*), 7.77-7.50 (m, 7H, Ar-H), 7.47-7.30 (m,13H, Ar-H), 7.25-7.00 (m, 7H, Ar-H), 6.89-6.72 (m, 2H, Ar-*H*); ^{13}C NMR (100 MHz,

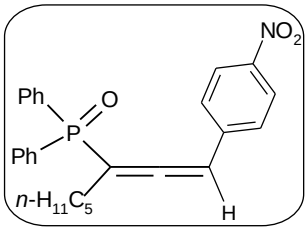
$CDCl_3$) δ 135.2, 135.0, 133.7, 133.5, 133.34, 133.27, 133.1, 132.9, 132.7, 132.6, 132.1, 132.0, 131.94, 131.90, 131.8, 129.9, 129.4, 129.0, 128.9, 128.8, 128.6, 128.50, 128.47,128.4, 128.33, 128.27, 128.20, 128.18, 128.12, 128.10, 128.06, 128.01, 127.81, 127.78, 126.2, 125.9, 125.8, 125.7, 125.5, 125.3, 125.1, 124.8, 124.7; ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.30 (d, *J* ~ 13.0 Hz), 24.86 (d, *J* ~ 13.0 Hz); HRMS (ESI) calcd. for $C_{43}H_{31}NO_4P_2Na$ [M^++Na] 710.1626, found: 710.1625.

Compound 17



This compound was prepared by following a route similar to that for **6** using propargyl alcohol **16** (0.35 g, 1.70 mmol).White solid; yield 0.613 g (92%); mp 146–148 ^{o}C; IR (KBr, cm^{-1}) 3085, 3052, 3030, 1923, 1655, 1595, 1490, 1436, 1381, 1118, 1074, 926, 827; ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83-7.70 (m, 6H, Ar-*H*), 7.46-7.12 (m, 14H, Ar-*H*), 6.30 (d, *J* = 10.8 Hz, 1H, C*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 212.9 (d, ^2J(P-C) = 5.0 Hz, C=*C*=C), 132.9, 132.5, 131.9, 131.8, 131.7, 131.6, 131.5, 128.7_0, 128.6_8, 128.3_2, 128.2_7, 128.2, 128.1, 127.9, 127.8, 126.9 (Ar-*C*), 105.4 (d, ^1J(P-C) = 98.0 Hz, P*C*), 98.4 (d, ^3J(P-C) = 13.0 Hz, *C*H); ^{31}P NMR (162 MHz, $CDCl_3$)δ 29.64; HRMS (ESI): Calcd. for $C_{27}H_{21}OP$ (M^+ + H): *m/z* 393.1409. Found 393.1410.

Compound 19



Procedure was similar to that for compound **6** using propargyl alcohol **18** (0.25 g, 0.99 mmol).Yellow liquid; yield 0.346 g (81%); IR (neat, cm^{-1}) 3058, 2959, 2932, 2860, 1929, 1595, 1512, 1436, 1342, 1195, 1118, 866; ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.10 (d, *J* = 8.4 Hz, 2H, Ar-*H*), 7.79-7.65 (m, 4H, Ar-*H*), 7.55-7.31 (m, 6H, Ar-*H*), 7.18 (d, *J* = 8.8 Hz, 2H, Ar-*H*), 6.17 (d, *J* = 10.4 Hz, 1H, C*H*), 2.50-2.36 (m, 2H, CH_2), 1.59-1.51 (m, 2H, CH_2), 1.33-1.21 (m, 4H, CH_2), 0.80 (t, 3H, CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 209.8 (^{2}J(P-C) = 6.0 Hz, C=*C*=C), 146.8, 140.1, 132.3, 132.1, 132.0, 131.6, 131.5, 131.4, 131.3, 130.9, 130.5, 128.6, 128.5, 128.4, 128.3, 127.0, 124.0 (Ar-*C*), 104.6 (d, ^{1}J(P-C) = 95.0 Hz, P*C*), 96.1 (d, ^{3}J(P-C) = 13.0 Hz, *C*H), 31.3, 28.0_9, 28.0_6 and 22.3 (*C*H_2), 13.9 (*C*H_3); ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.27; HRMS (ESI): Calcd. for $C_{26}H_{26}NO_3P$ (M^+ + Na): *m/z* 454.1548. Found: 454.1549.

References

(1) D. D. Perrin, W. L. F. Armarego, D. R. Perrin, *Purification of Laboratory Chemicals*, Pergamon, Oxford, 1986.

(2) G. M. Sheldrick, *SADABS, Siemens Area Detector AbsorptionCorrection*, University of Göttingen, Germany, **1996**; G. M. Sheldrick, SHELX-97- *A program for crystal structure solution and refinement*, University of Göttingen, **1997**; G. M. Sheldrick, *SHELXTL NT Crystal Structure Analysis Package*, Bruker AXS, Analytical X-ray System, WI, USA, **1999**, version 5.10.

(3) V. Srinivas, K. V. Sajna and K. C. Kumara Swamy, *Chem. Commun.*, 2011, **47**, 5629; G. Gangadhararao and K. C. Kumara Swamy, *Tetrahedron*, 2014, **70**, 2643.

(4) W. Yan, Q. Wang, Y. Chen, J. L. Petersen and X. Shi, *Org. Lett*., 2012, **14**, 5880.

(5) (a) Benington, F.; Morin, R. D.; Clark, L. C. *J. Org. Chem.* **1960**, *25*, 1542. (b) Fan, J.; Wan, C.; Sun, G.; Wang, Z. *J. Org. Chem.* **2008**, *73*, 8608.

X-ray Data:

Compound 5: $C_{39}H_{29}NO_3P_2$, M = 621.57, orthorhombic, space group $P2_12_12_1$, a = 11.2790(6), b = 16.1962(8), c = 17.5843(9) Å, V = 3212.1(3) Å^3, Z = 4, μ = 0.175 mm^{-1}, Flack parameter -0.03(9), data/restraints/parameters: 5347/0/406, R indices (I> 2σ(I)): R1 = 0.0421, *w*R2 (all data) = 0.1120. CCDC No. 1041550.

Compound 6: $C_{39}H_{29}NO_4P_2$, M = 637.57, orthorhombic, space group $P2_12_12_1$, a = 11.2810(5), b = 16.1001(8), c = 17.7437(9) Å, V = 3222.7(2) Å^3, Z = 4, μ = 0.178 mm^{-1}, Flack parameter -0.09(12), data/restraints/parameters: 5547/0/415, R indices (I> 2σ(I)): R1 = 0.0586, *w*R2 (all data) = 0.1120. CCDC No. 1041551.

Compound 9: $C_{49}H_{31}NO_4P_2$, M = 651.60, orthorhombic, space group $P2_12_12_1$, a = 11.1755(11), b = 15.950(2), c = 18.117(2) Å, V = 3229.3(7) Å^3, Z = 4, μ = 0.179 mm^{-1}, Flack parameter -0.03(16), data/restraints/parameters: 5200/0/425, R indices (I> 2σ(I)): R1 = 0.0765, *w*R2 (all data) = 0.1471. CCDC No. 1041552.

Compound 10: $C_{49}H_{30}ClNO_4P_2$, M = 686.04, triclinic, space group $P\bar{1}$, a = 9.9475(3), b = 13.0705(5), c = 14.9853(5) Å, α = 106.240(3), β = 94.143(3), γ = 110.585(3), V = 1719.36(11) Å^3, Z = 2, μ = 2.211 mm^{-1}, data/restraints/parameters: 6544/0/434, R indices (I> 2σ(I)): R1 = 0.0435, *w*R2 (all data) = 0.1252. CCDC No. 1041553.

Compound 11: $C_{42}H_{32}BrCl_6NO_4P_2$, M = 969.24, triclinic, space group $P\bar{1}$, a = 11.5145(7), b = 13.3968(8), c = 14.6374(9) Å, α = 86.833(5), β = 75.632(5), γ = 87.590(5), V =

2183.0(2) Å^3, Z = 2, μ = 1.424 mm^{-1}, data/restraints/parameters: 7681/0/507, R indices (I> 2σ(I)): R1 = 0.0729, *w*R2 (all data) = 0.2226. CCDC No. 1041554.

Compound (*R*)-12: $C_{39}H_{33}NO_4P_2$, M = 641.60, orthorhombic, space group $P2_12_12_1$, a = 11.3350(6), b = 17.7472(12), c = 16.1457(8) Å, V = 3247.9(3) Å^3, Z = 4, μ = 0.177 mm^{-1}, Flack parameter -0.13(12), data/restraints/parameters: 5525/0/415, R indices (I> 2σ(I)): R1 = 0.0564, *w*R2 (all data) = 0.1610. CCDC No. 1041555.

Compound (*S*)-12: $C_{39}H_{33}NO_4P_2$, M = 641.60, orthorhombic, space group $P2_12_12_1$, a = 11.3101(10), b = 17.7378(17), c = 16.0426(14) Å, V = 3218.4(5) Å^3, Z = 4, μ = 0.179 mm^{-1}, Flack parameter -0.27(11), data/restraints/parameters: 5523/0/415, R indices (I> 2σ(I)): R1 = 0.0487, *w*R2 (all data) = 0.1354. CCDC No. 1041556.

Compound (*S*)-12 [Low temperature (100K) data]: $C_{39}H_{33}NO_4P_2$, M = 641.60, orthorhombic, space group $P2_12_12_1$, a = 11.326(2), b = 15.940(3), c = 17.593(3) Å, V = 3176(1) Å^3, Z = 4, μ = 0.181 mm^{-1}, Flack parameter -0.03(9), data/restraints/parameters: 5582/0/416, R indices (I> 2σ(I)): R1 = 0.0402, *w*R2 (all data) = 0.1079. CCDC No. 1051469.

Compound 13: $C_{39}H_{33}ClNO_4P_2$, M = 677.05, triclinic, space group $P\bar{1}$, a = 9.7782(6), b = 12.6735(7), c = 15.2180(9) Å, α = 72.422(5), β = 88.076(5), γ = 69.077(5), V = 1673.42(17) Å^3, Z = 2, μ = 0.253 mm^{-1}, data/restraints/parameters: 5885/0/424, R indices (I> 2σ(I)): R1 = 0.0534, *w*R2 (all data) = 0.1545. CCDC No. 1041557.

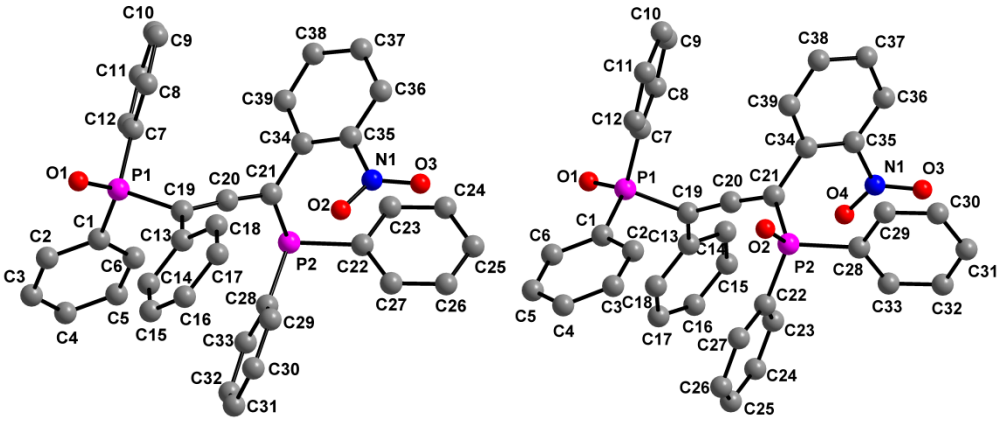


Fig. S1 Left: Molecular structure of compound **5**. Selected bond parameters: P1-O1 1.472(2), P1-C19 1.825(3), C19-C13 1.495(4), C19-C20 1.322(4), C20-C21 1.293(4), C21-C34 1.488(4), C21-P2 1.861(3) (Å). **Right:** Molecular structure of compound **6**. Selected bond parameters: P1-O1 1.474(3), P1-C19 1.821(4), C19-C13 1.492(5), C19-C20 1.319(6), C20-C21 1.300(6), C21-C34 1.495(5), C21-P2 1.834(4), P2-O2 1.483(3) (Å). Hydrogen atoms are omitted for clarity in both the drawings.

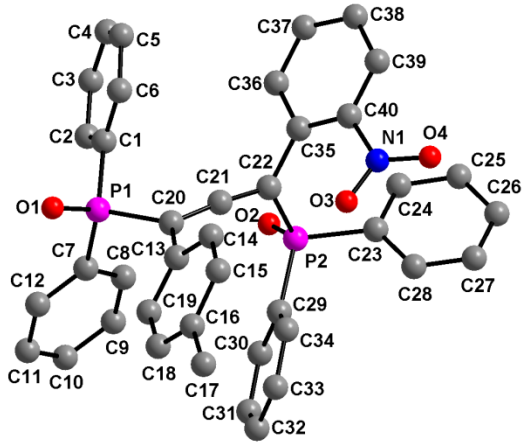


Fig. S2 Molecular structure of compound **9**. Hydrogen atoms are omitted for clarity. Selected bond parameters: P1-O1 1.468(4), P1-C20 1.806(6), C20-C13 1.463(8), C20-C21 1.322(8), C21-C22 1.278(8), C22-C35 1.478(8), C22-P2 1.829(6), P2-O2 1.467(4) (Å).

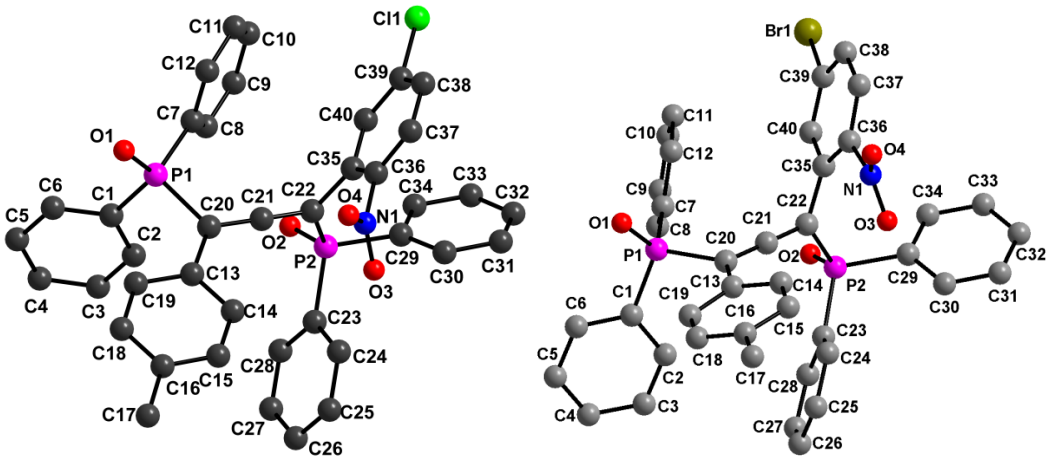


Fig. S3 Left: Molecular structure of compound **10**. Selected bond parameters: P1-O1 1.4828(13), P1-C20 1.8300(17), C20-C13 1.487(2), C20-C21 1.311(2), C21-C22 1.304(2), C22-C35 1.494(2), C22-P2 1.8395(17), P2-O2 1.4616(16) (Å). **Right:** Molecular structure of compound **11**. Selected bond parameters: P1-O1 1.488(4), P1-C20 1.828(5), C20-C13 1.481(7), C20-C21 1.307(7), C21-C22 1.305(6), C22-C35 1.483(6), C22-P2 1.828(5), P2-O2 1.482(3) (Å).

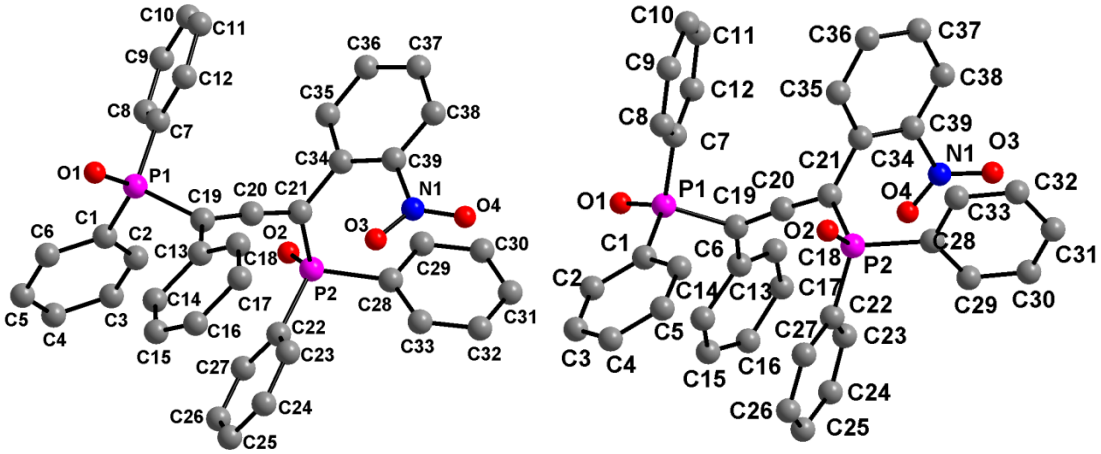


Fig. S4 Molecular structures for compounds (a) (*R*)-**12** (*b*) (*S*)-**12**. Hydrogen atoms are omitted for clarity. Selected bond parameters with esd's in parentheses follow. **Compound (*R*)-12:** P1-O1 1.475(3), P1-C19 1.823(4), C19-C13 1.487(5), C19-C20 1.307(5), C20-C21 1.301(5), C21-

C34 1.480(5), C21-P2 1.824(4), P2-O2 1.476(3) (Å). **Compound (*S*)-12**: P1-O1 1.472(2), P1-C19 1.818(3), C19-C13 1.493(4), C19-C20 1.300(4), C20-C21 1.295(5), C21-C34 1.484(4), C21-P2 1.821(3), P2-O2 1.473(2) (Å).

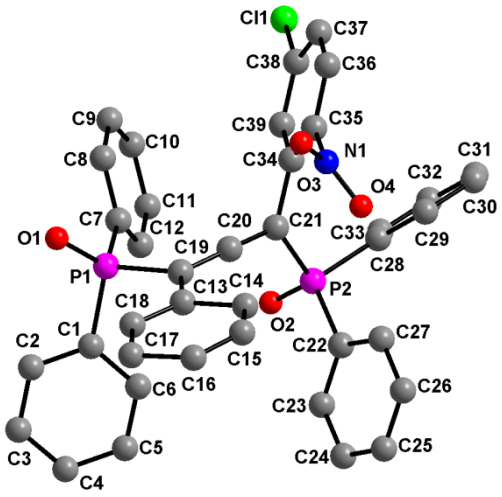


Fig. S5 Molecular structure of compound **13**. Hydrogen atoms are omitted for clarity. Selected bond parameters with esd's in parentheses: P1-O1 1.4779(19), P1-C19 1.828(3), C19-C13 1.493(4), C19-C20 1.309(3), C20-C21 1.306(3), C21-C34 1.492(3), C21-P2 1.831(3), P2-O2 1.4713(19) (Å).

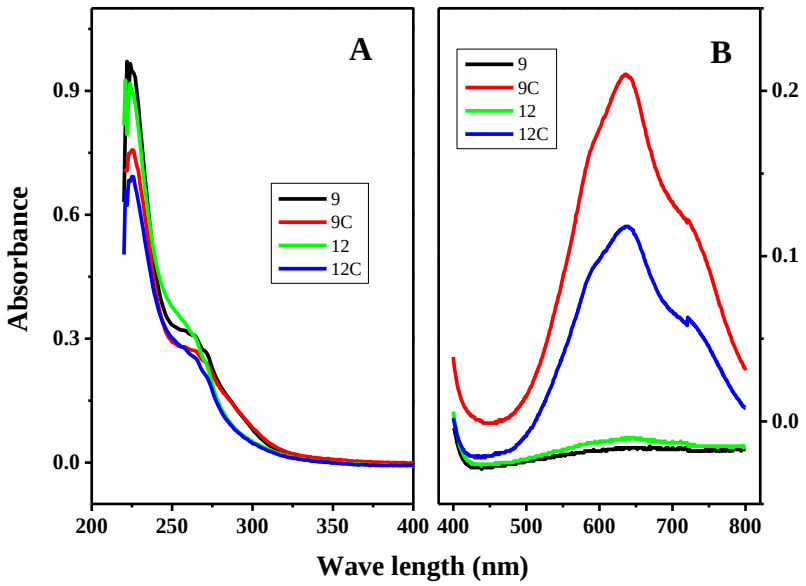


Fig. S6 The UV-Visible spectra of compounds **9** and **12** before and after colour change with (**A**) c = 1.7 x 10^{-5}mol/L and (**B**) c = 1.0 x 10^{-3}mol/L. Whereas curves for **9** or **12** represent fresh solutions, those represented by **9C** or **12C** are for bluish-green coloured solutions formed after 2-days.

^{1}H and ^{13}C NMR spectra

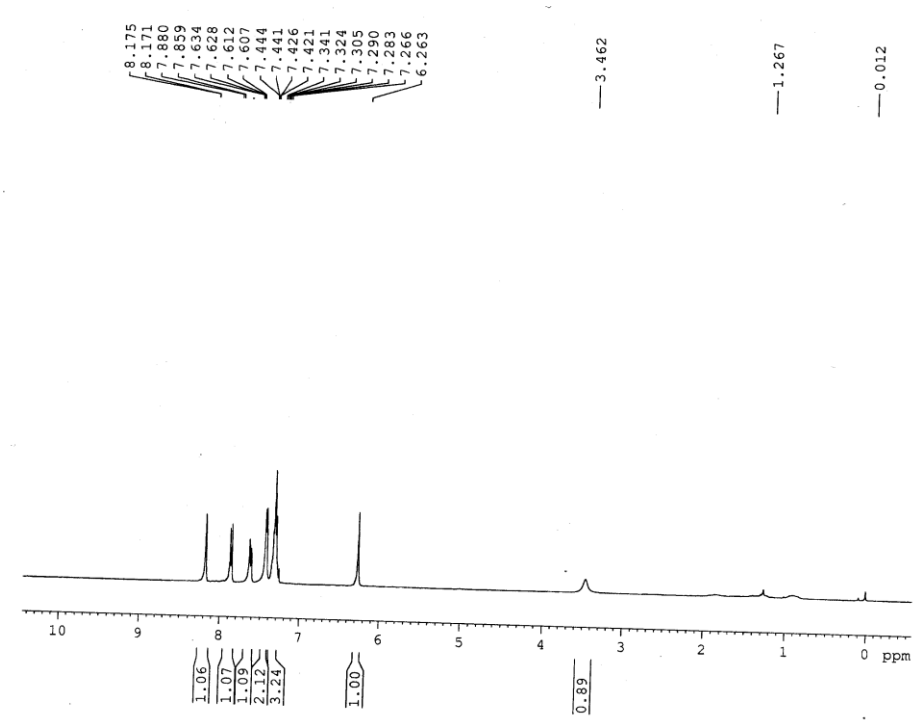


Figure S7. ^{1}H NMR spectrum of compound **2c**

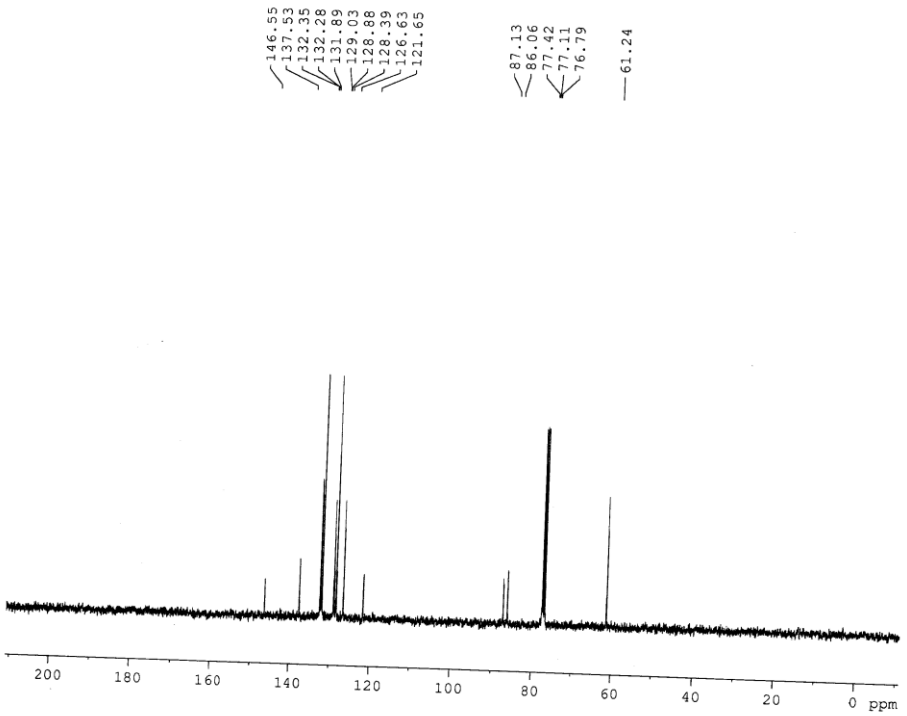


Figure S8. ^{13}C NMR spectrum of compound **2c**

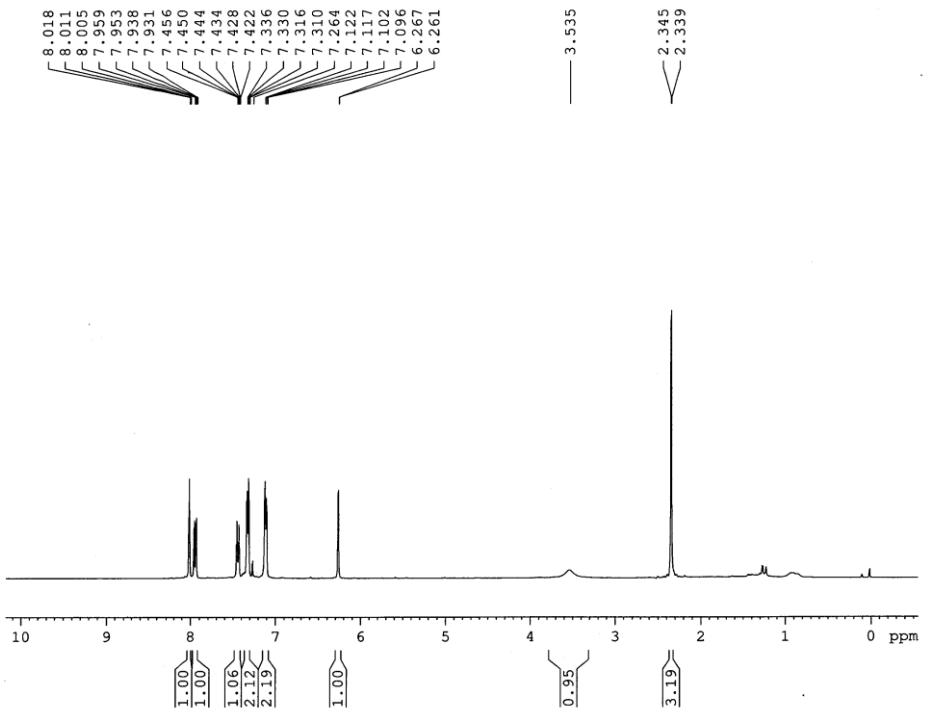


Figure S9.[1]H NMR spectrum of compound **2e**

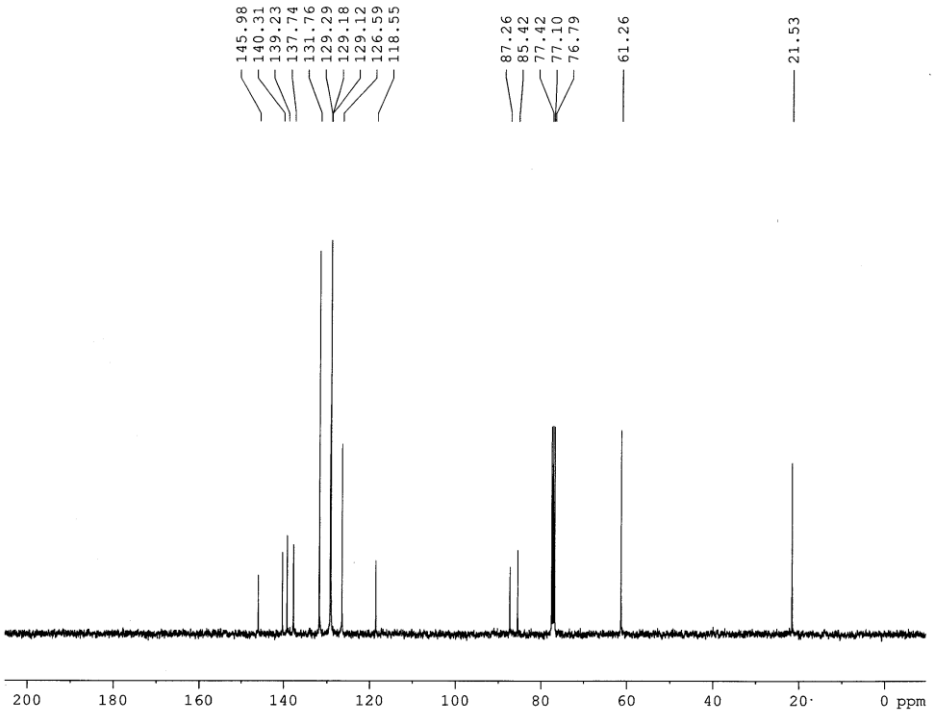


Figure S10.[13]C NMR spectrum of compound **2e**

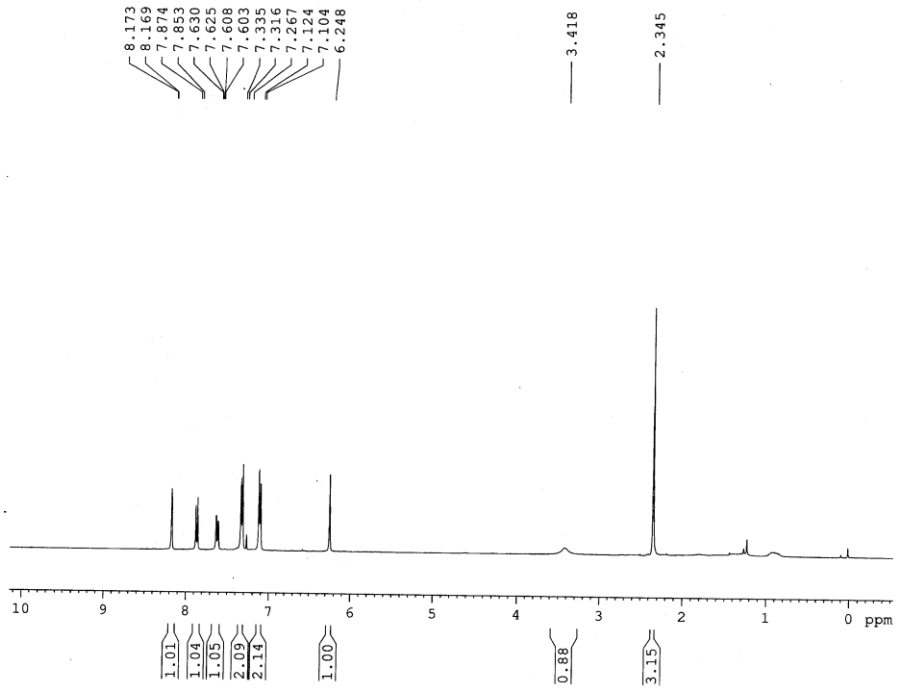


Figure S11. ^{1}H NMR spectrum of compound **2f**

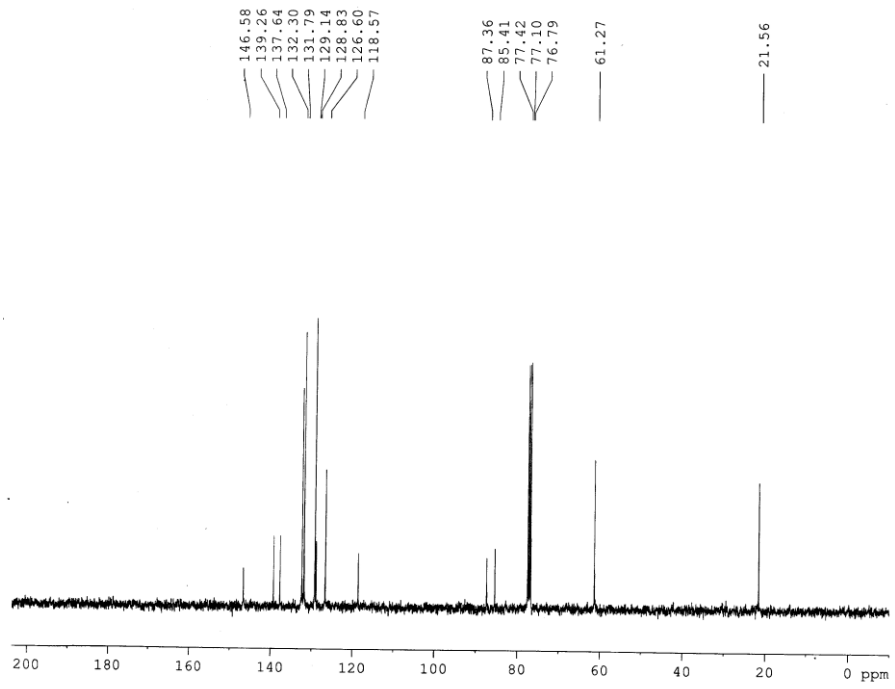


Figure S12. ^{13}C NMR spectrum of compound **2f**

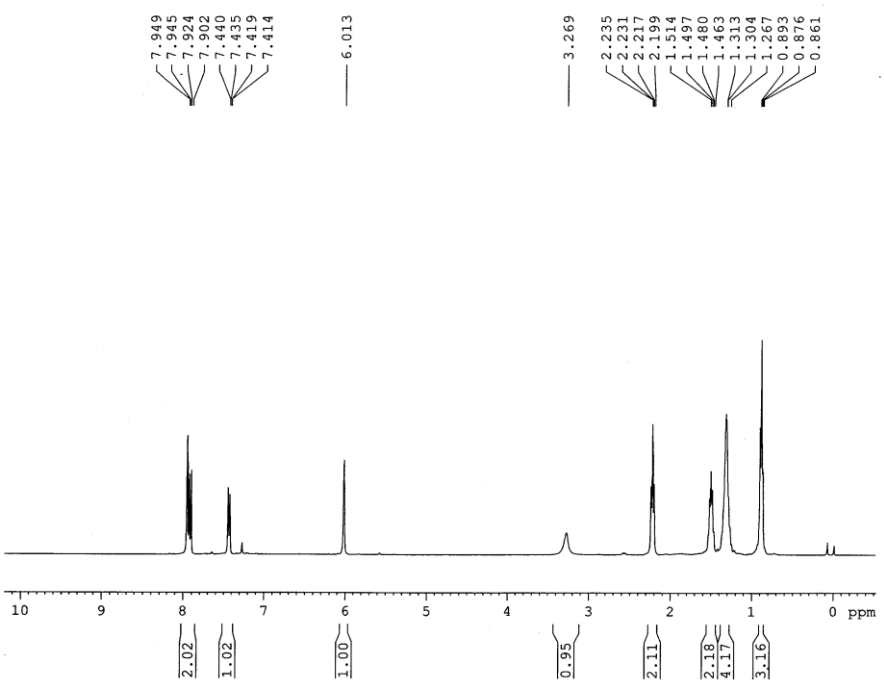


Figure S13. ^{1}H NMR spectrum of compound **4**

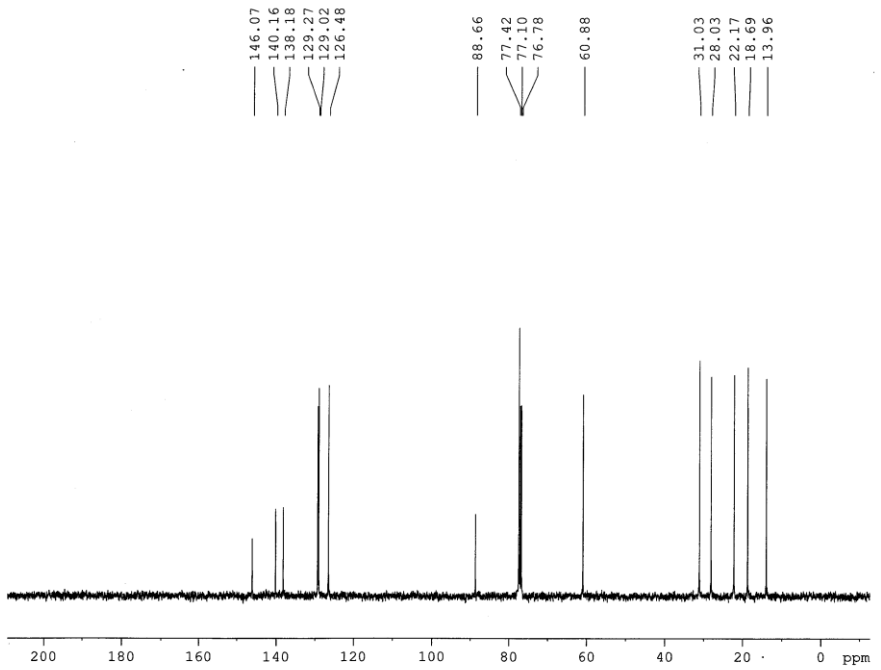


Figure S14. ^{13}C NMR spectrum of compound **4**

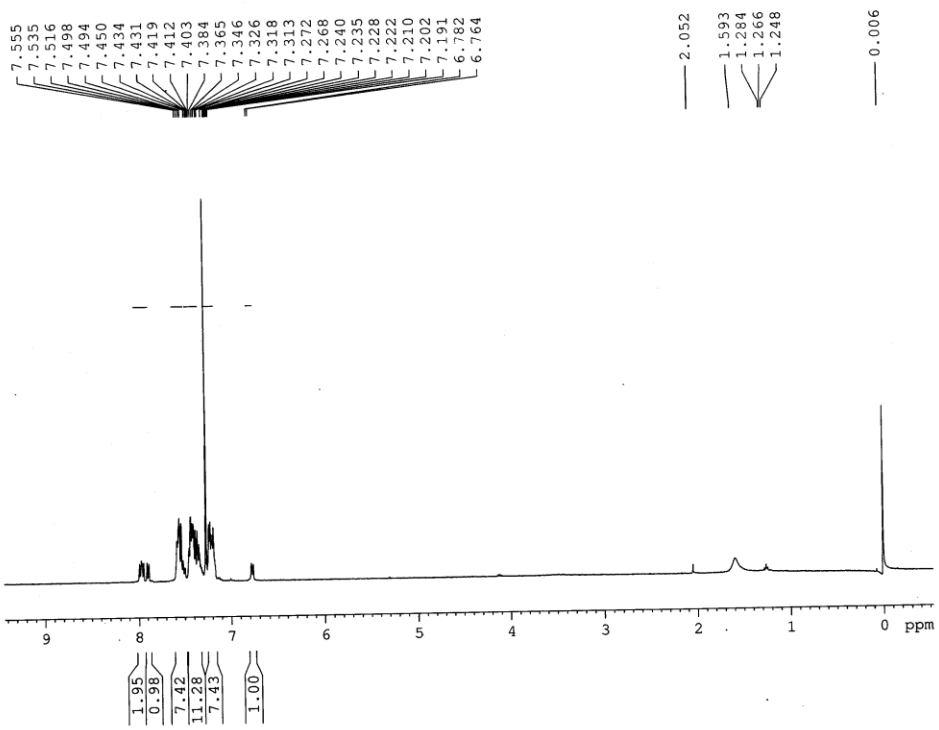


Figure S15. ^{1}H NMR spectrum of compound **6**

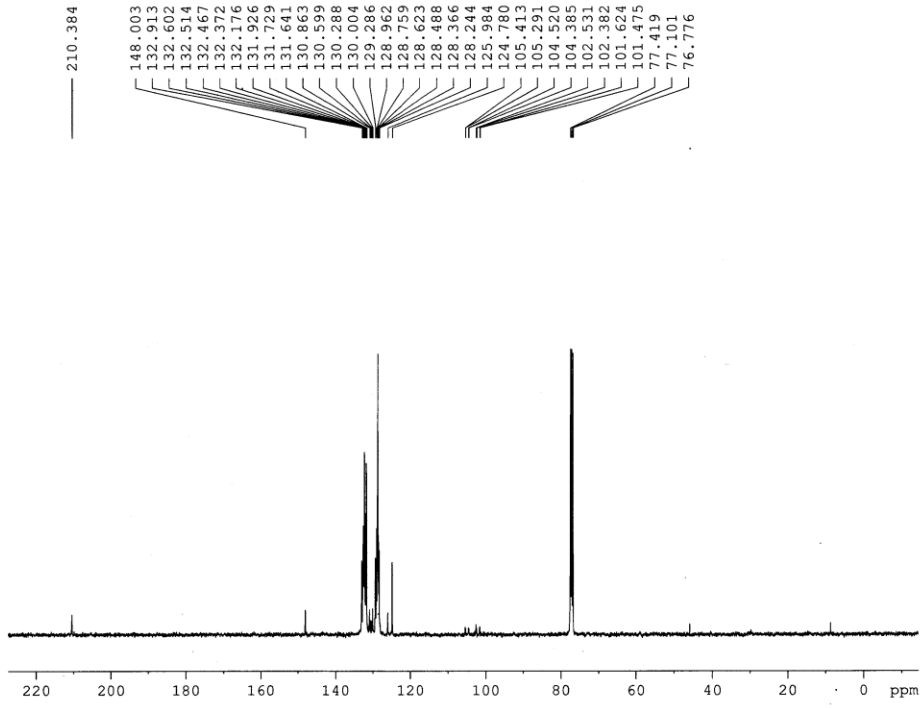


Figure S16. ^{13}C NMR spectrum of compound **6**

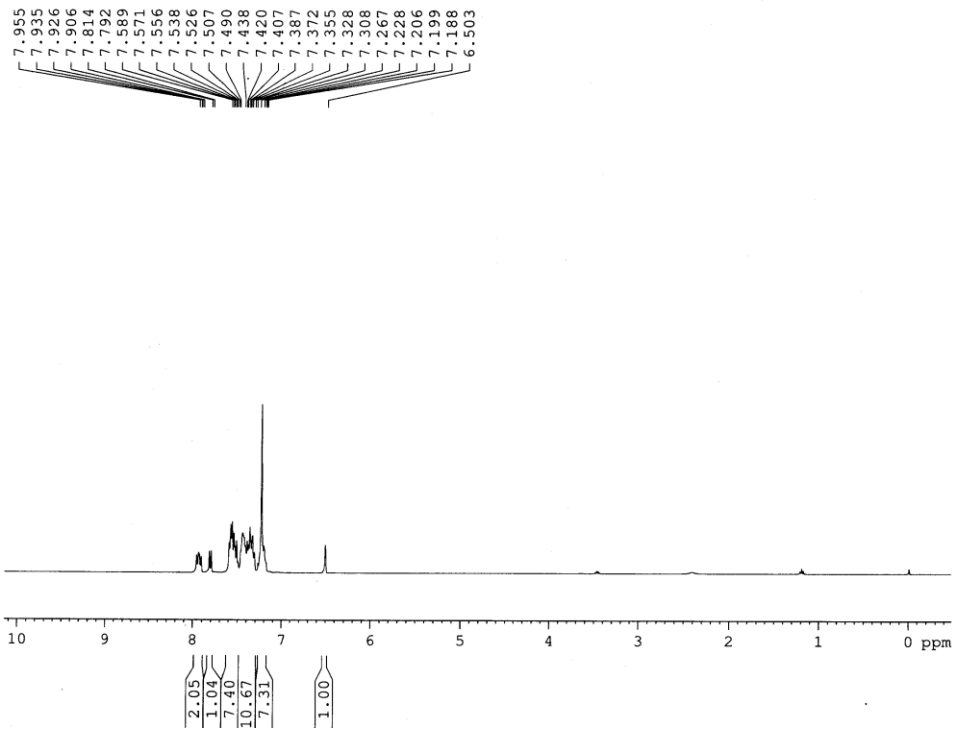


Figure S17. ^{1}H NMR spectrum of compound **7**

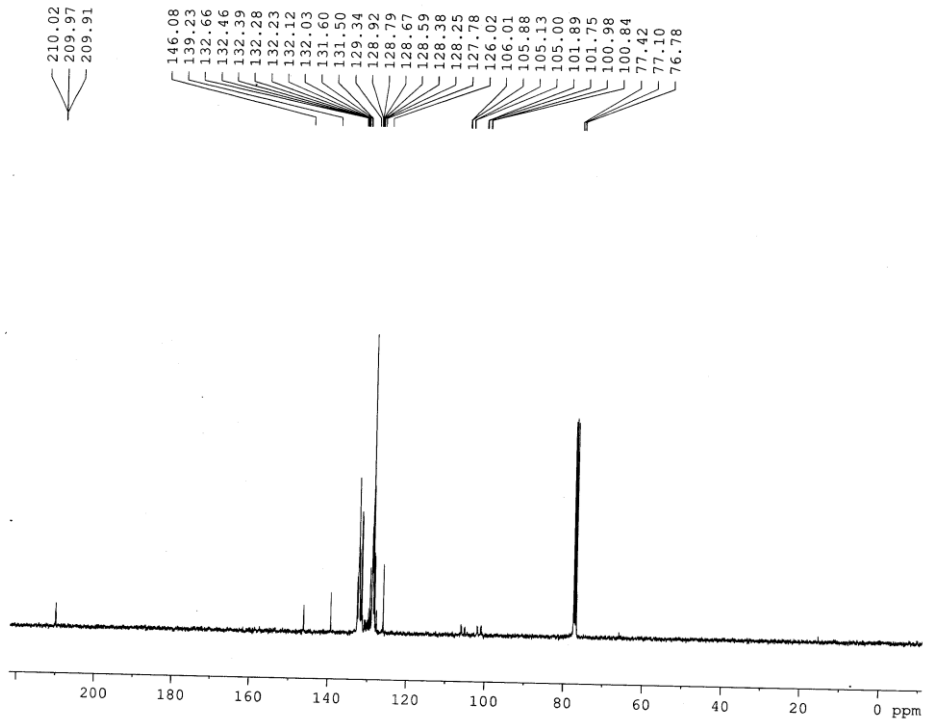


Figure S18. ^{13}C NMR spectrum of compound **7**

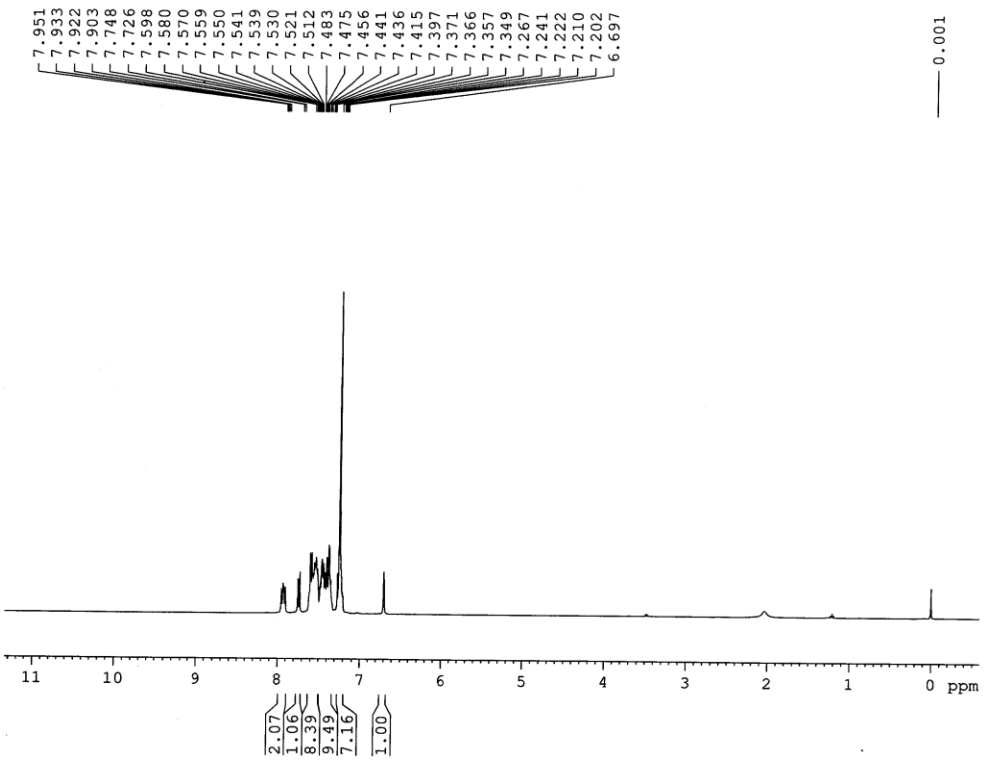


Figure S19. ^{1}H NMR spectrum of compound **8**

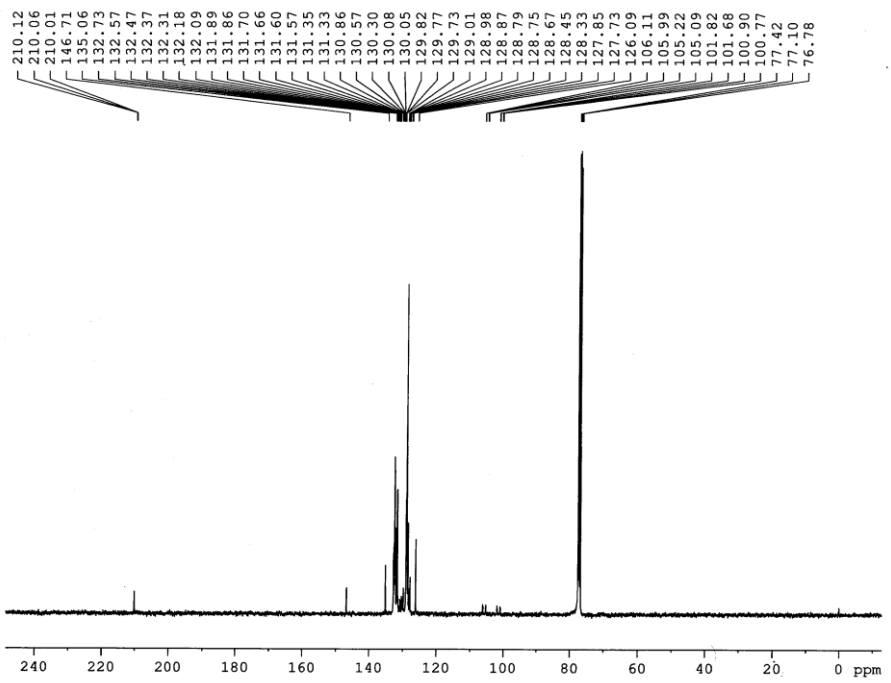


Figure S20. ^{13}C NMR spectrum of compound **8**

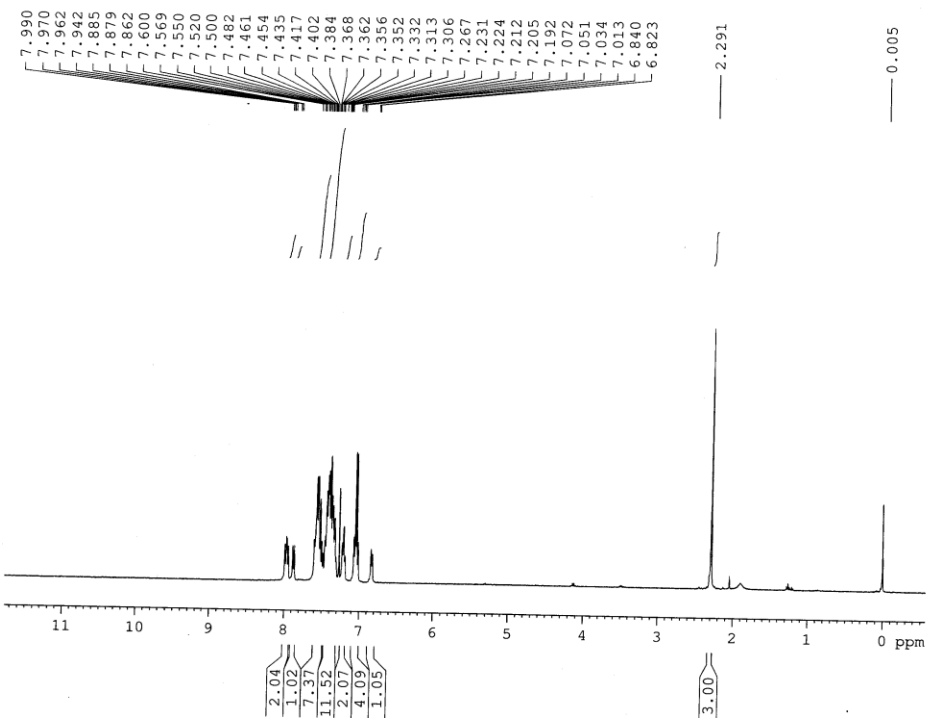


Figure S21. ^{1}H NMR spectrum of compound **9**

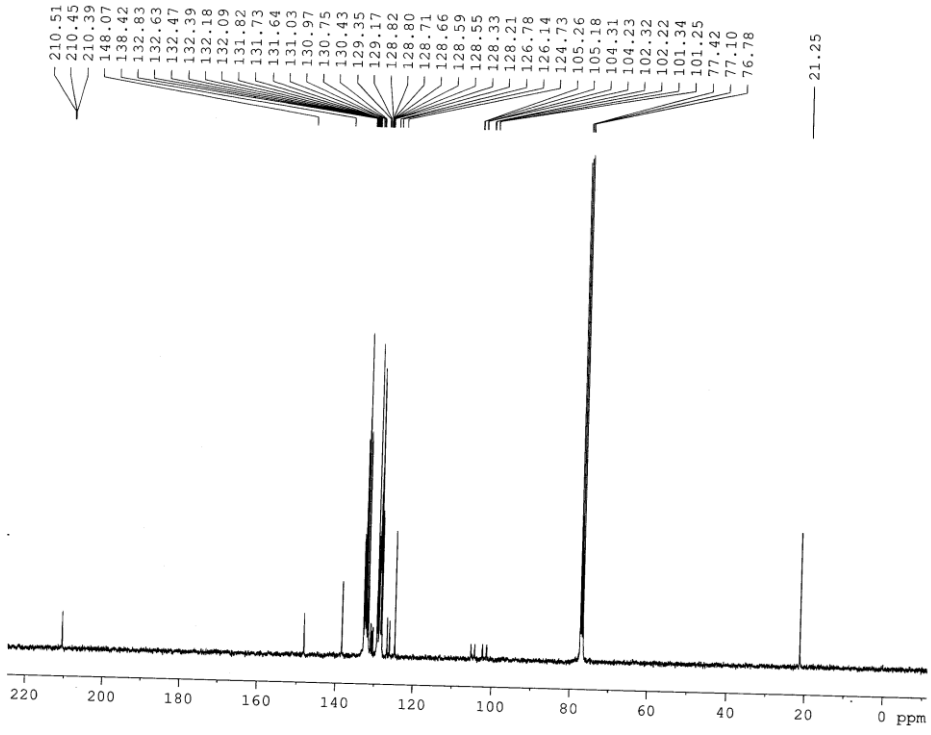


Figure S22. ^{13}C NMR spectrum of compound **9**

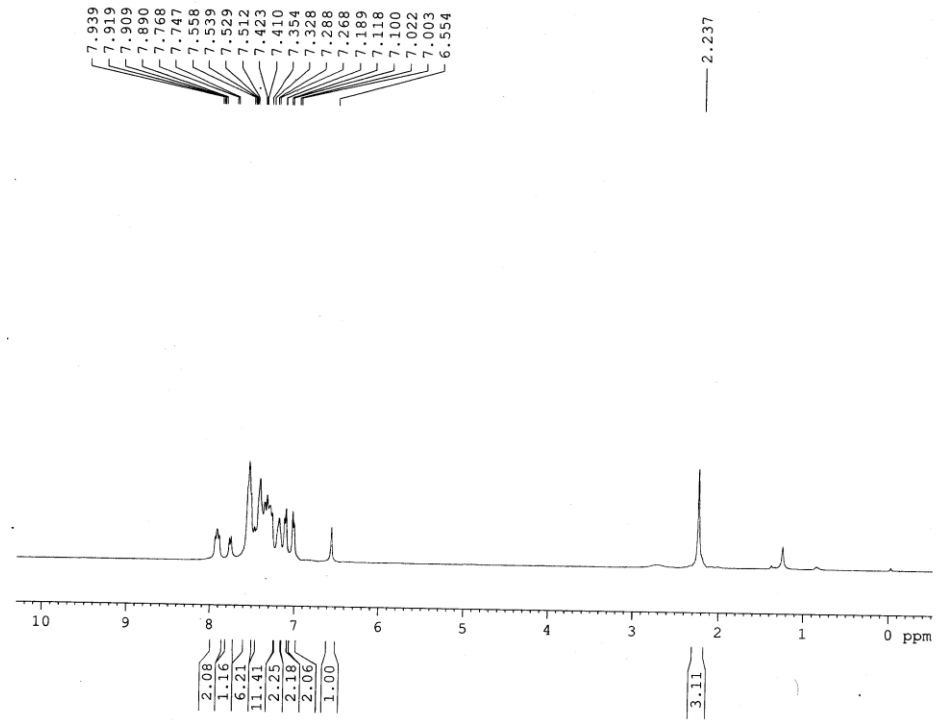


Figure S23. ^{1}H NMR spectrum of compound **10**

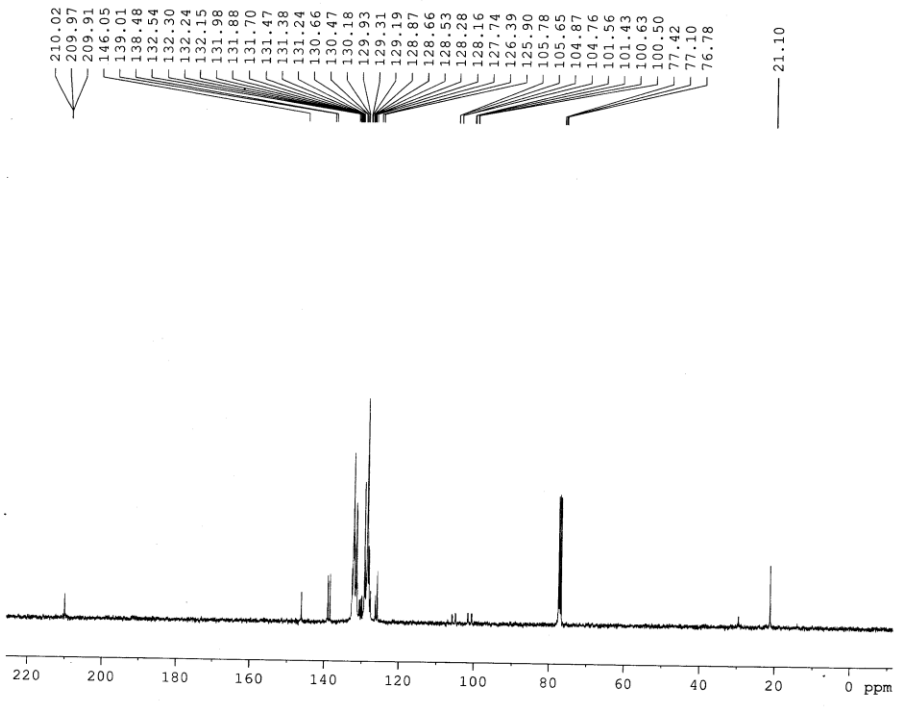


Figure S24. ^{13}C NMR spectrum of compound **10**

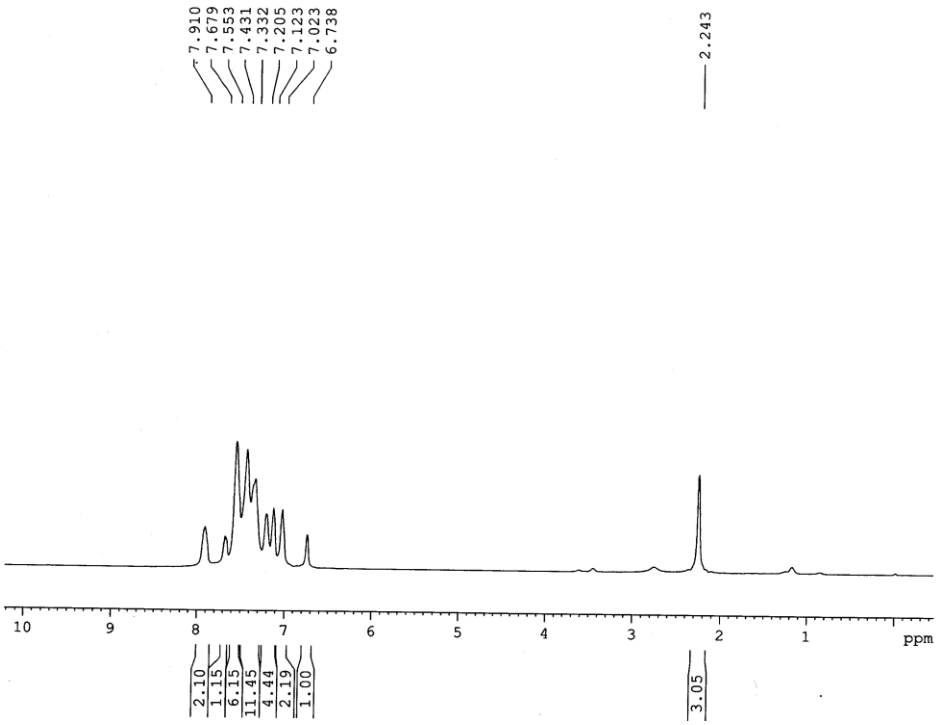


Figure S25. ^{1}H NMR spectrum of compound **11**

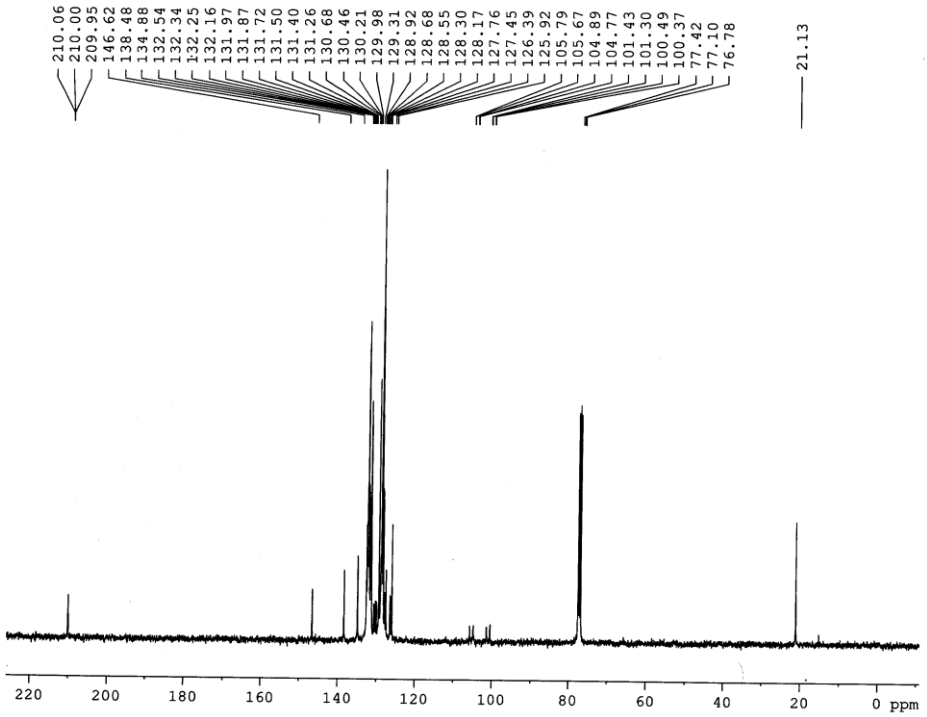


Figure S26. ^{13}C NMR spectrum of compound **11**

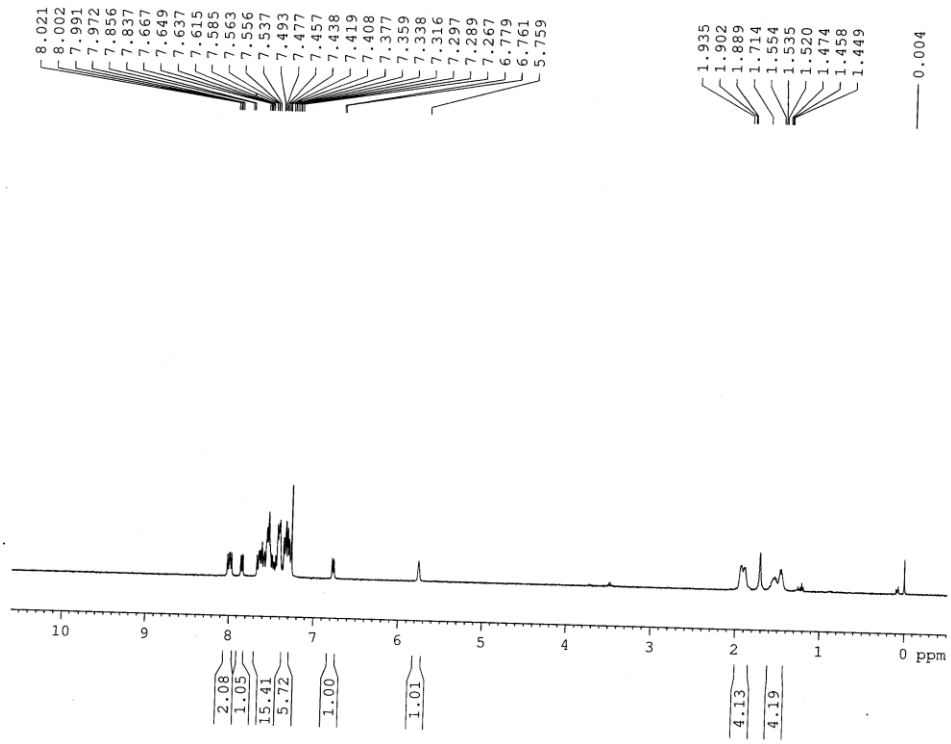


Figure S27. ^{1}H NMR spectrum of compound **12**

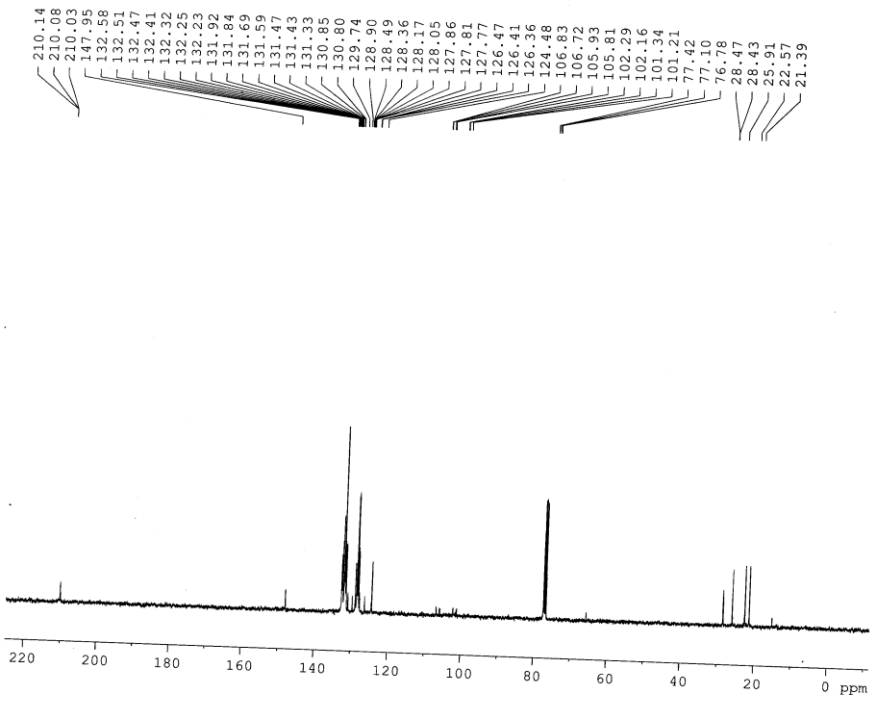


Figure S28. ^{13}C NMR spectrum of compound **12**

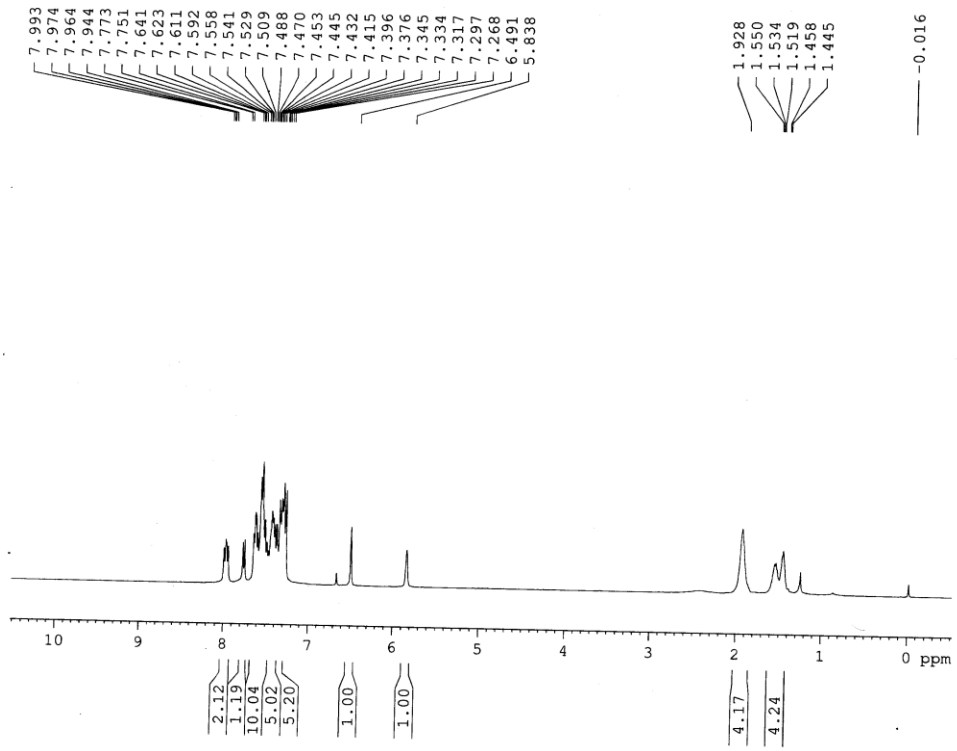


Figure S29. ^{1}H NMR spectrum of compound **13**

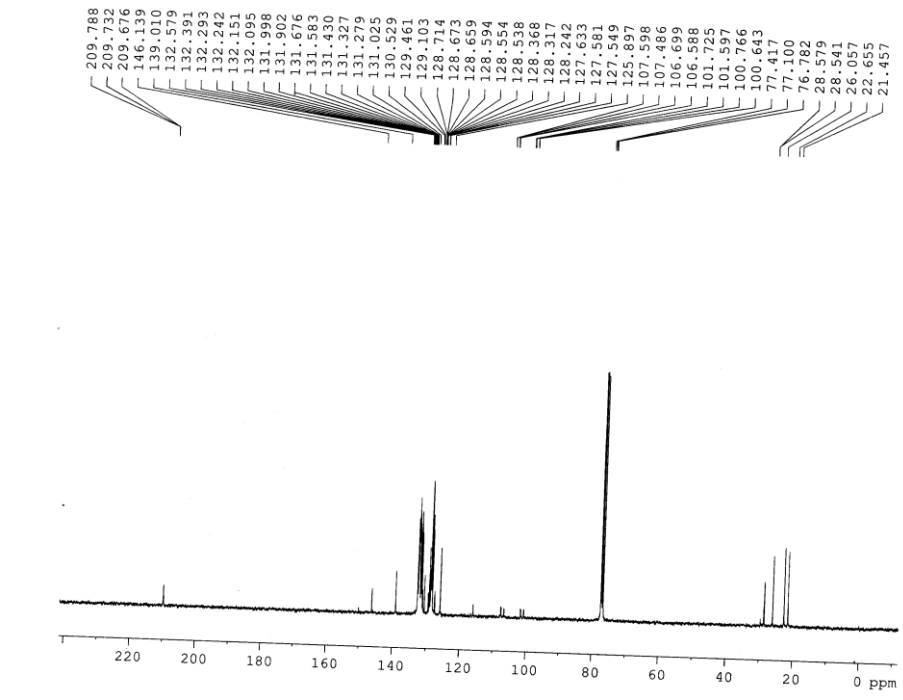


Figure S30. ^{13}C NMR spectrum of compound **13**

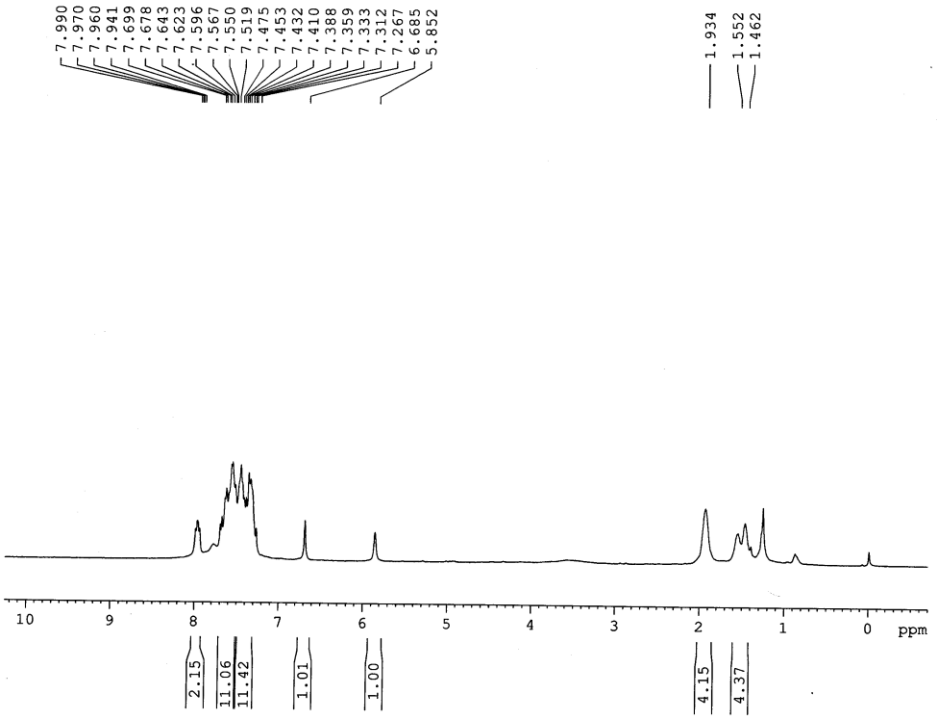


Figure S31. ^{1}H NMR spectrum of compound **14**

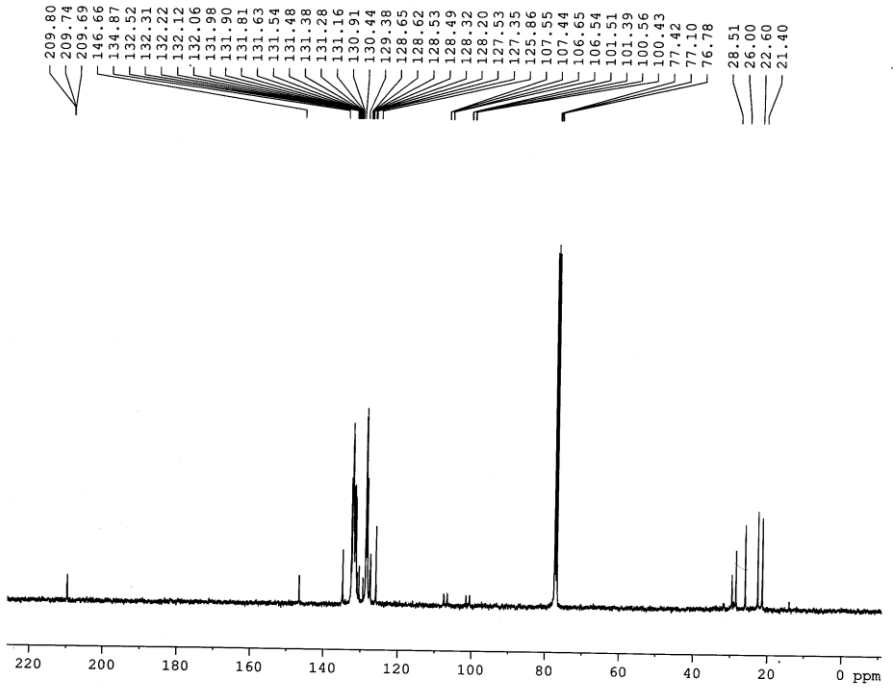


Figure S32. ^{13}C NMR spectrum of compound **14**

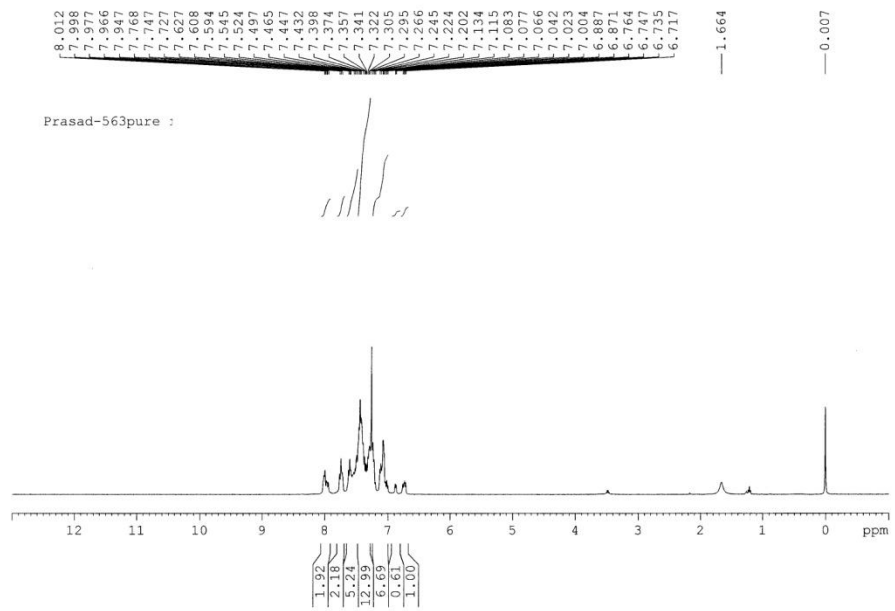


Figure S33.[1]H NMR spectrum of compound **A**

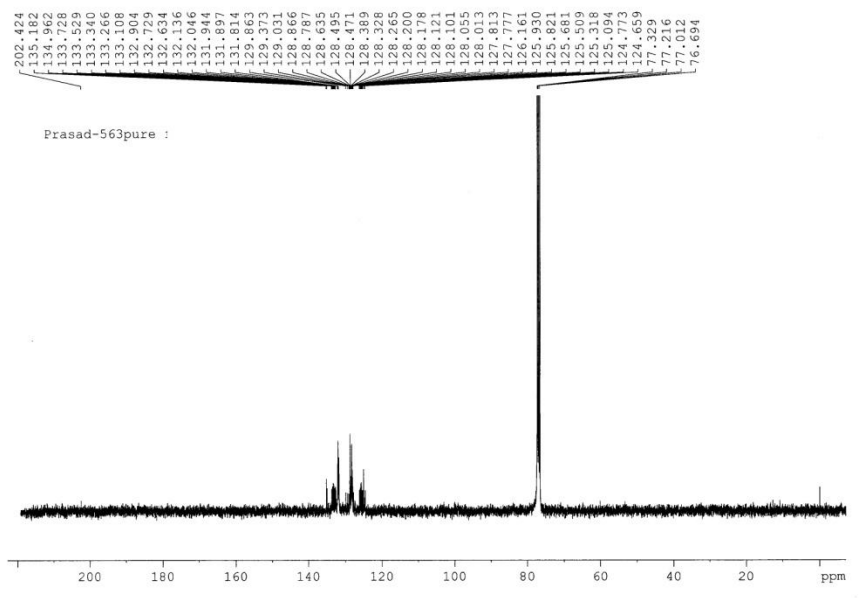


Figure S34.[13]C NMR spectrum of compound **A**

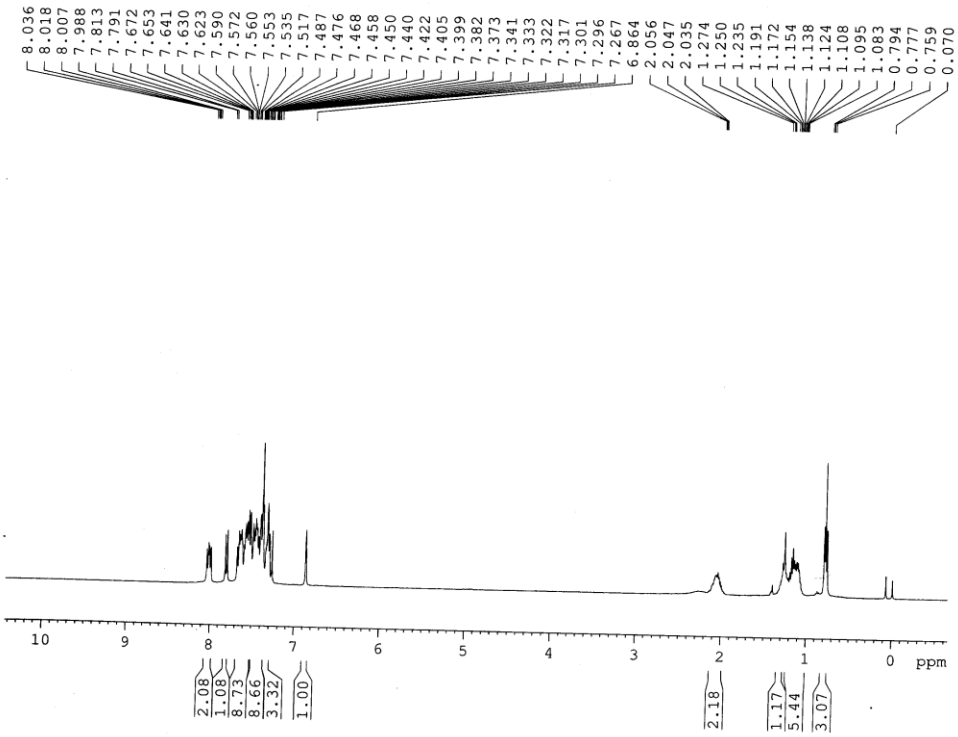


Figure S35. ^{1}H NMR spectrum of compound **15**

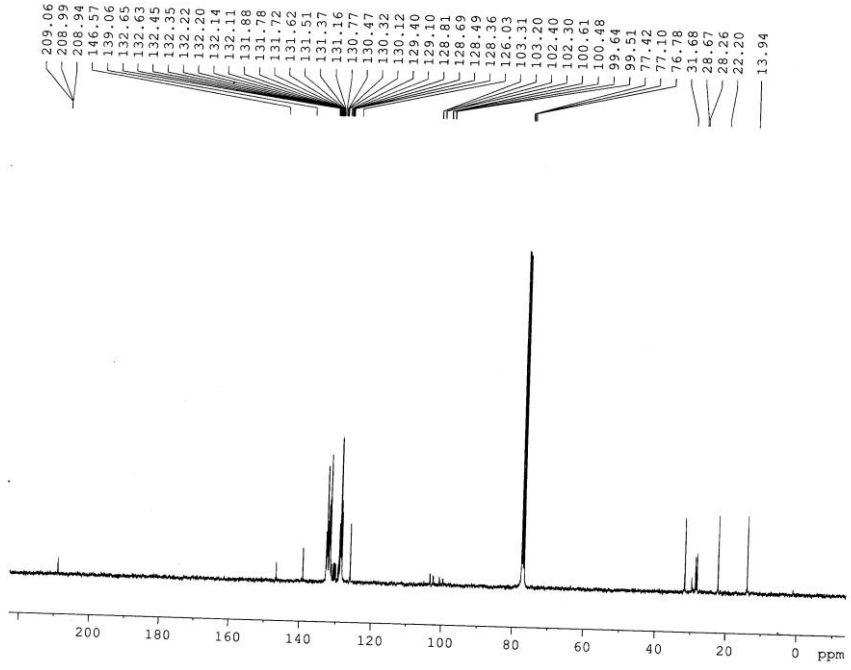


Figure S36. ^{13}C NMR spectrum of compound **15**

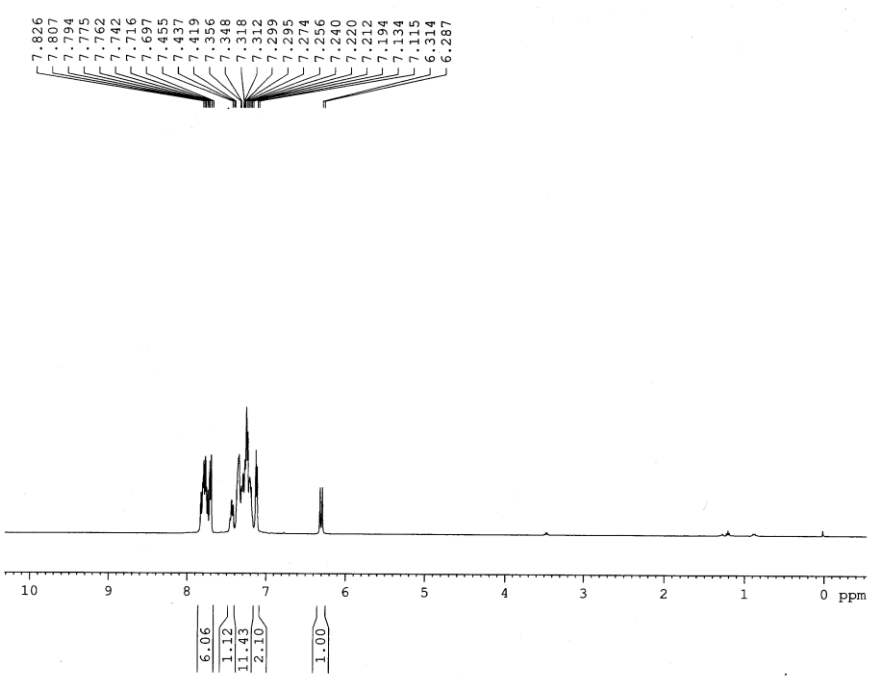


Figure S37. ^{1}H NMR spectrum of compound **17**

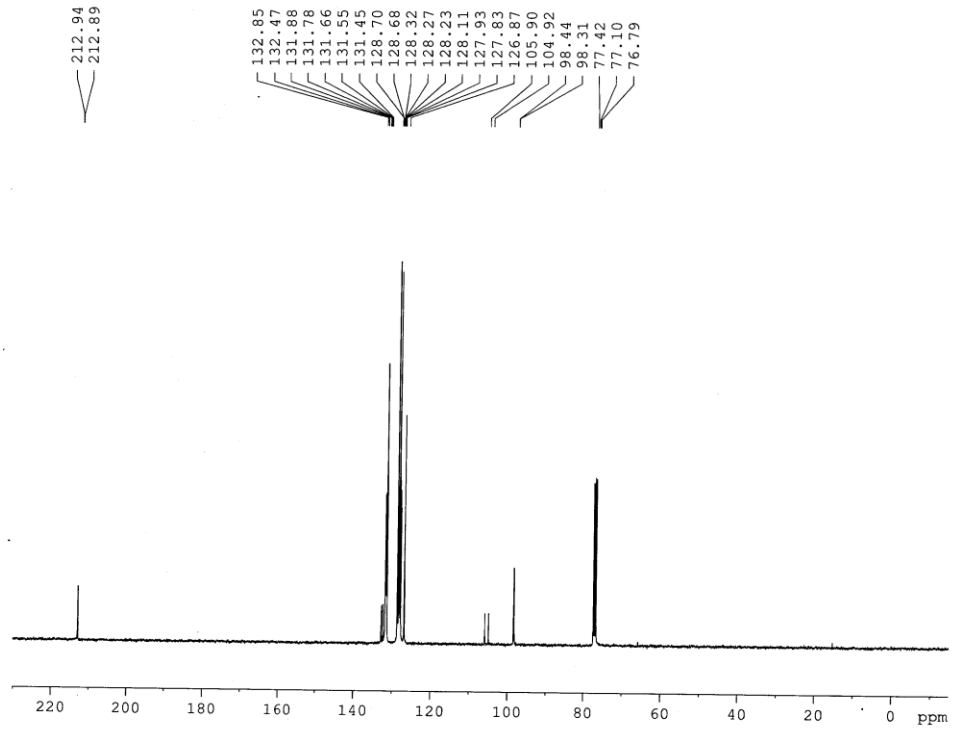


Figure S38. ^{13}C NMR spectrum of compound **17**

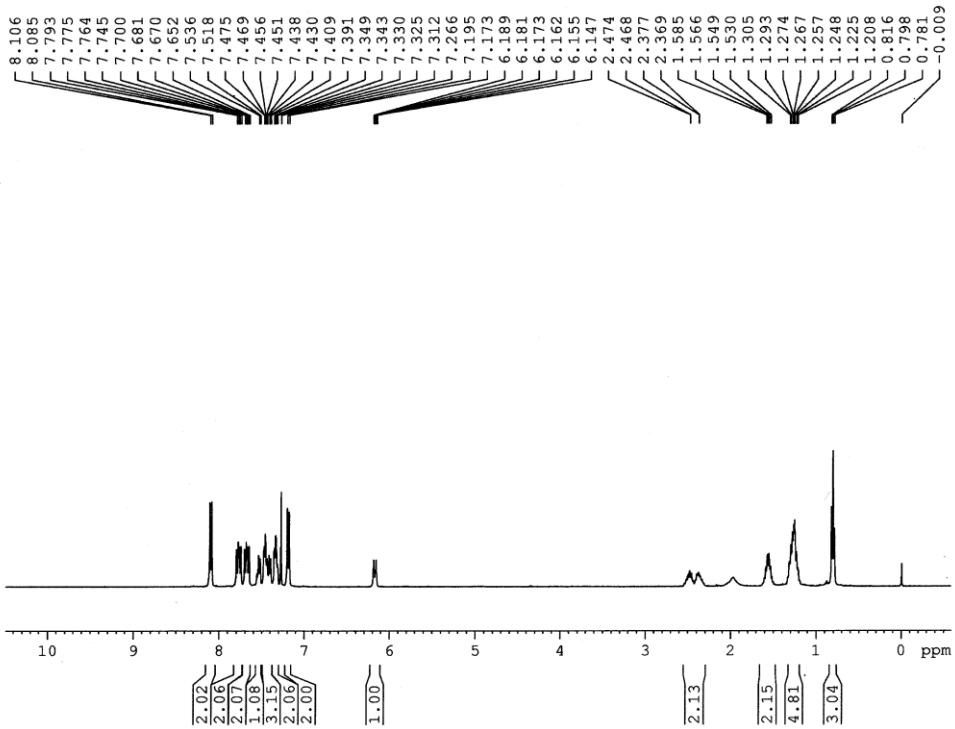


Figure S39. ^{1}H NMR spectrum of compound **19**

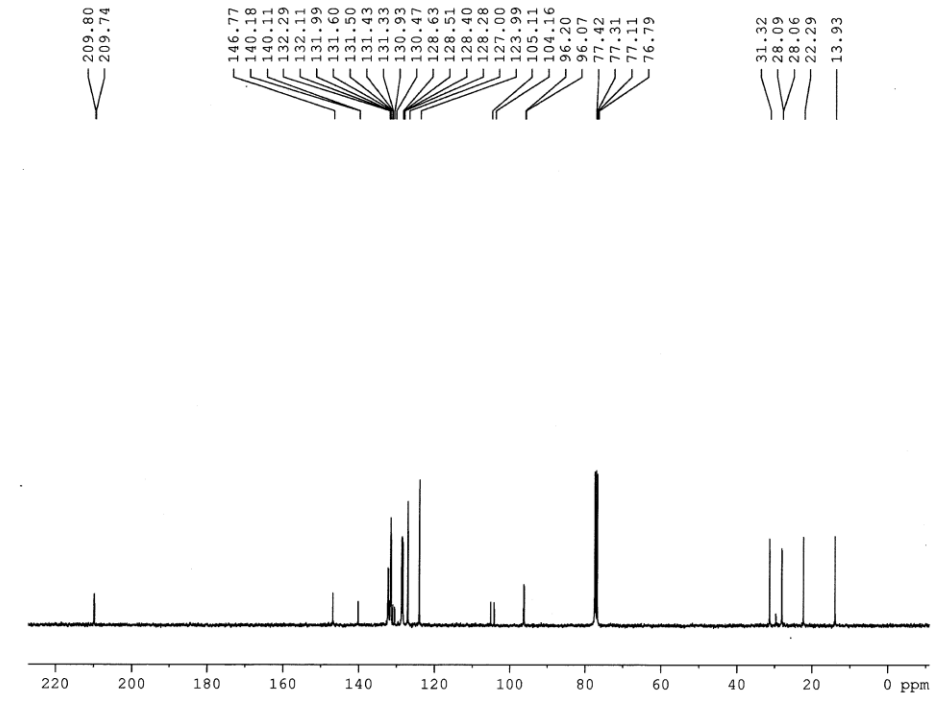
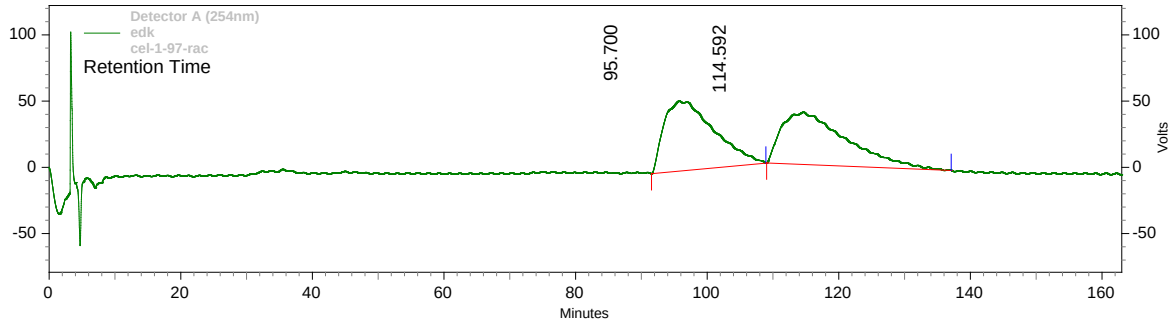


Figure S40. ^{13}C NMR spectrum of compound **19**

Compound 12-(racemic mixture after column chromatography)

Shimadzu CLASS-VP V6.14 SP1 *Area % Report* Page 1 of 1
Method Name: E:\Class VP\Data\suresh\2,4\washing.met
Data Name: E:\Class VP\Data\edk\Prasad\al-p-rac-567\cel-1-97-rac
Acquired: 2/27/2015 12:27:41
Printed: 2/27/2015 15:12:22
Column: Cellulose-1(chiral); Flow rate (97: 3 of hexane and 2-propanol) : 1.0 ml/ min

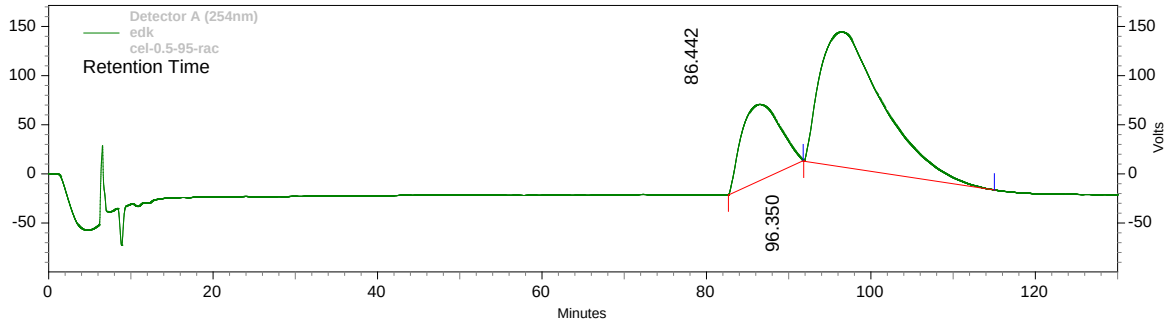


Detector A (254nm)

Pk #	Retention Time	Area	Area %	Height	Height %
1	95.700	27658130	50.616	52865	57.287
2	114.592	26985170	49.384	39416	42.713
Totals		54643300	100.000	92281	100.000

Compound 12- [ten handpicked crystals after crystallization enantiomer ratio ca 3:1]

Shimadzu CLASS-VP V6.14 SP1 *Area % Report* Page 1 of 1
Method Name: C:\CLASS-VP\untitled.met
Data Name: E:\Class VP\Data\edk\Prasad\al-p-rac-567\cel-0.5-95-rac
Acquired: 2/26/2015 17:41:14
Printed: 2/26/2015 19:58:37
Column: Cellulose-1(chiral); Flow rate (97: 3 of hexane and 2-propanol): 0.5 ml/ min.



Detector A (254nm)

Pk #	Retention Time	Area	Area %	Height	Height %
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2	96.350	73222287	74.343	137161	63.839
Totals		98492886	100.000	214854	100.000