Electronic Supplementary Information for

The [1,2,3]Triazolo[1,5-*a*]pyridine Ring: A Sensitive Sensor for the Electronic Profile of Phosphorus Substituents

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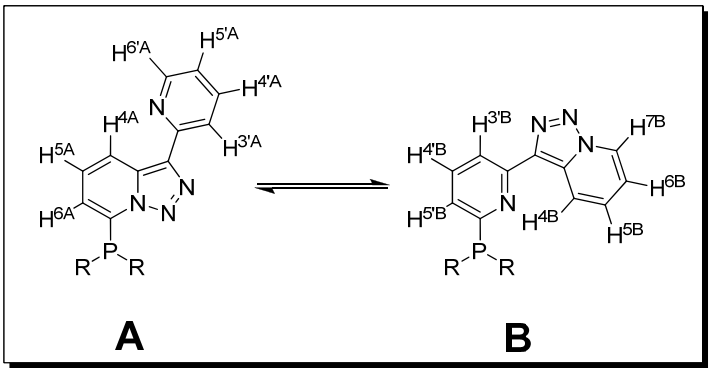
Experimental Section

General Methods

Starting materials, if commercial, were purchased and used as such, provided that adequate checks (melting ranges, refractive indices, and gas chromatography) had confirmed the claimed purity. When known compounds had to be prepared according to literature procedures, pertinent references are given. Air- and moisture-sensitive materials were stored in Schlenk tubes. They were protected by and handled under an atmosphere of argon, using appropriate glassware. Diethyl ether and tetrahydrofuran were dried by distillation from sodium after the characteristic blue color of sodium diphenyl ketyl (benzophenone-sodium "radical-anion") had been found to persist. Ethereal or other organic extracts were dried by washing with brine and then by storage over sodium sulfate. If no reduced pressure is specified, boiling ranges (b.p.) refer to ordinary atmosphere conditions (725 ± 25 Torr). Melting ranges (m.p.) given were found to be reproducible after recrystallization, unless stated otherwise ("decomp."), and are uncorrected. If melting points are missing, it means all attempts to crystallize the liquid at temperatures down to –75 °C failed. Thin-layer chromatography (TLC) were carried out on 0.25 mm Merck silica-gel (60-F254). The TLC plates were visualized with UV light and 7% phosphomolybdic acid. Column chromatography was carried out on a column packed with silica-gel 60N spherical neutral size 63-210 μm. The solid support was suspended in hexanes and, when all air bubbles had escaped, was washed into the column. When the level of the liquid was still 3 – 5 cm above the support layer, the dry powder, obtained by adsorption of the crude mixture to some 25 mL of silica and subsequent evaporation of the solvent, was poured on top of the column. ^{1}H and (^{1}H decoupled) ^{13}C nuclear magnetic resonance (NMR) spectra were recorded at 400 or 300 and 101 or 75 MHz, respectively. Chemical shifts are reported in δ units, parts per million (ppm) and were measured relative to the signals for residual chloroform (7.27 ppm). Coupling constants *J* are given in Hz. Coupling patterns are abbreviated as, for example, s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), td (triplet of doublets) and m (multiplet). H-H COSY experiment were performed for all compounds.

Assignment of hydrogen atoms:



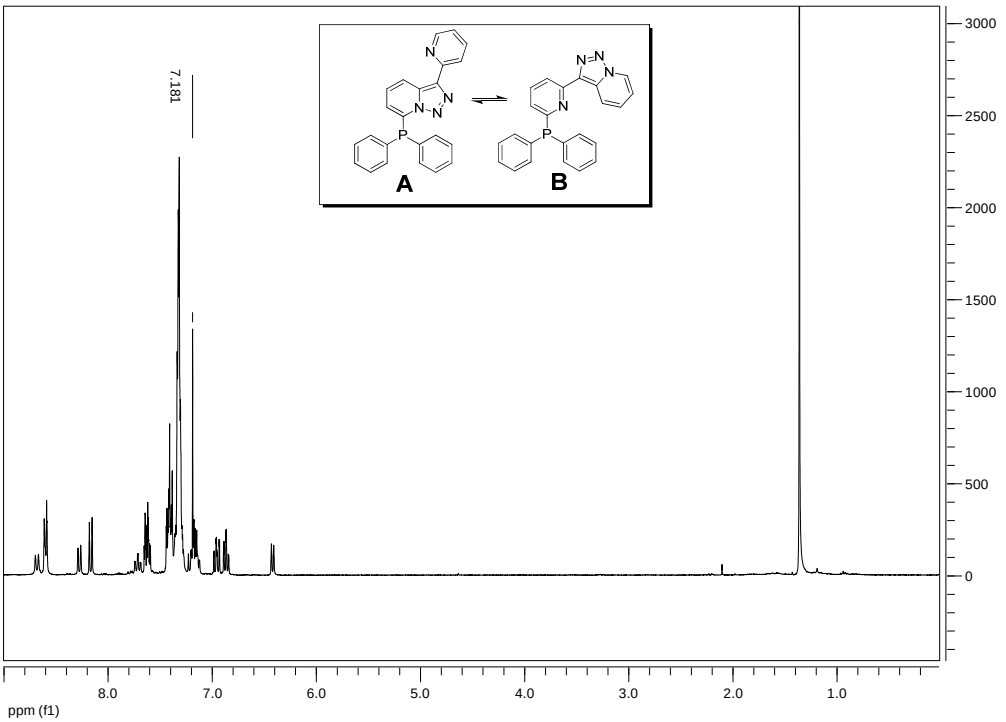
Typical Experimental procedure for the preparation of [1,2,3]triazolo[1,5-*a*]pyridine phosphines:

At -40 °C, butyllithium (3.6 mL, 5.6 mmol, 1.1 eq) in hexanes (1.5M) was added to a solution of 3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine **1** (1.0 g, 5.1 mmol, 1.0 eq) in toluene (60 mL). The mixture was kept for 30 min at -40 °C before a solution of the corresponding chlorophosphine (5.9 mmol, 1.15 eq) in toluene (6.0 mL) was added and allowed to reach 20 °C (2 h). The reaction mixture was quenched with water (20 mL). The resulting mixture was extracted with dichloromethane (3×50 mL) and the combined organic extracts were washed with brine (10 mL), dried over Na_2SO_4, filtered, and concentrated. Chromatography (silica gel, ethyl acetate/cyclohexane = 3:7) provided the corresponding isomeric A/B mixture of phosphines. The corresponding selenides were prepared according to standard procedure: the phosphines were allowed to react with selenium powder in refluxing chloroform during 5 hours.

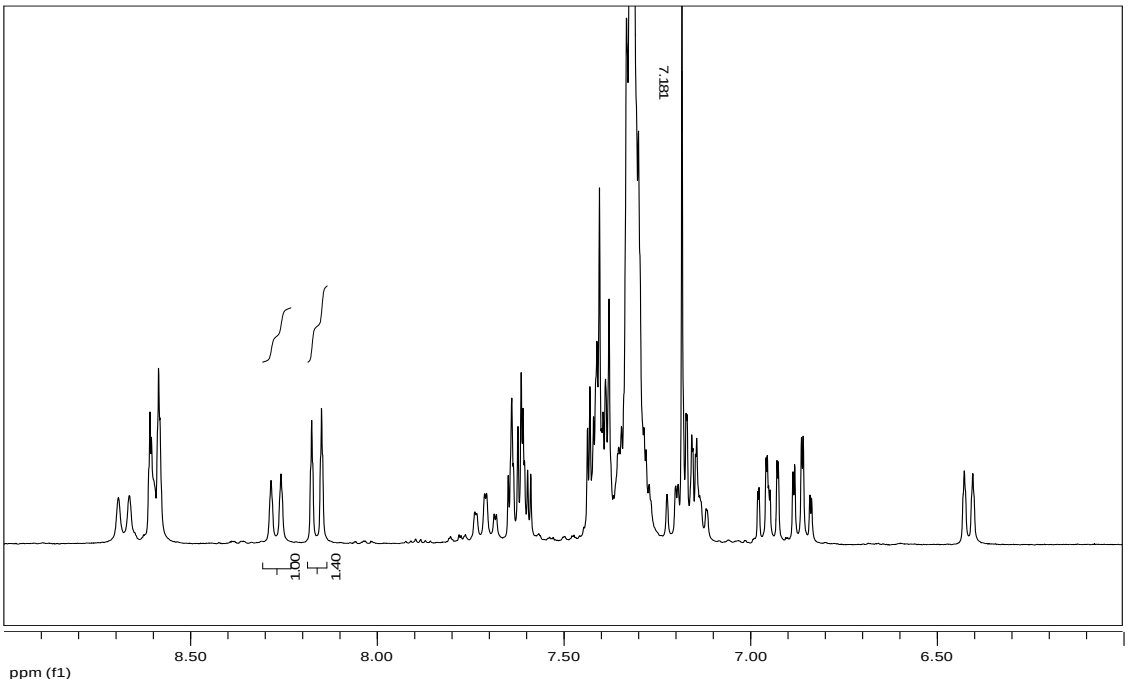
Example of NMR assignment

^{1}H NMR from systems **2d** are described in detail in order to show how the assignment has been done.

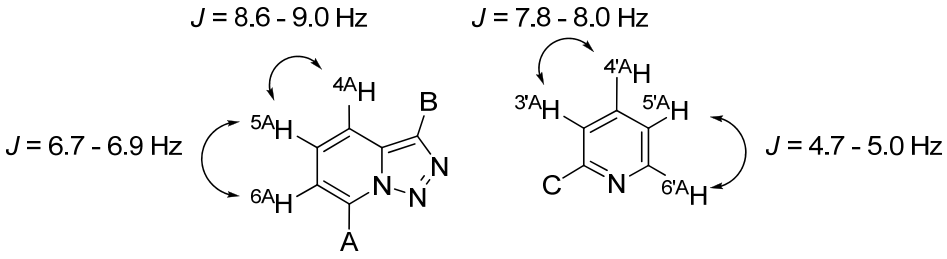
^{1}H NMR of 2d:



Zoom of the aromatic region of 2d:

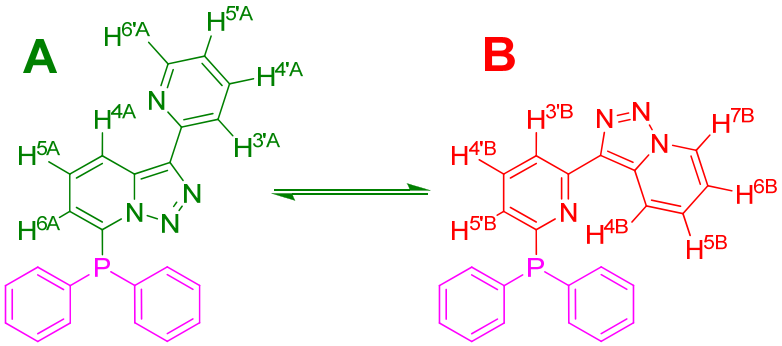


The δ and J values for isomers **A** compared to other **A** isomers described in the literature [ref 5] indicates that they have a pyridyl-triazolopyridinephosphine structure. They contain a 3,7-disubstituted triazolopyridine moiety and a 2-substituted pyridine. The presence of a proton near 8.8 ppm with a coupling constant of J = 8.8–9.0 Hz is significant, corresponding to the ^{4A}H triazolopyridine proton. Furthermore, another signal with the same integration can be found near 6.5 ppm with J = 6.7–6.9 Hz consistent with a ^{6A}H proton. In the same way two more signals (appearing as a doublet) with the same integration must be found ($^{3'A}$H and $^{6'A}$H). Although $^{6'A}$H has, normally, a similar chemical shift than ^{7B}H, $^{3'A}$H can be perfectly found near 8.4 ppm (J = 8.0 Hz). H-H COSY correlation allows assign all protons of these systems.



On the other hand, the isomers **B** have a 3-substituted triazolopyridine and a 2,6-disubstituted pyridine. The triazolopyridine presents its 4 hydrogen atoms; in this case ^{7B}H and ^{4B}H are mixed with other signals from the **A** and/or **B** structure. However, ^{6B}H and ^{5B}H appears as two apparent triplets. Once these signals are identified, H-H COSY allows the assignation of the whole 3-subtituted triazolopyridine. Near 8.3 ppm a doublet with J = 8.0 Hz can be found with the same integration than ^{6B}H and ^{5B}H. As it has been shown before ($^{3'A}$H : 8.5 ppm, d, J = 8.0 Hz) this signal can be assigned to $^{3'B}$H.

In the ^{1}H NMR Spectrum

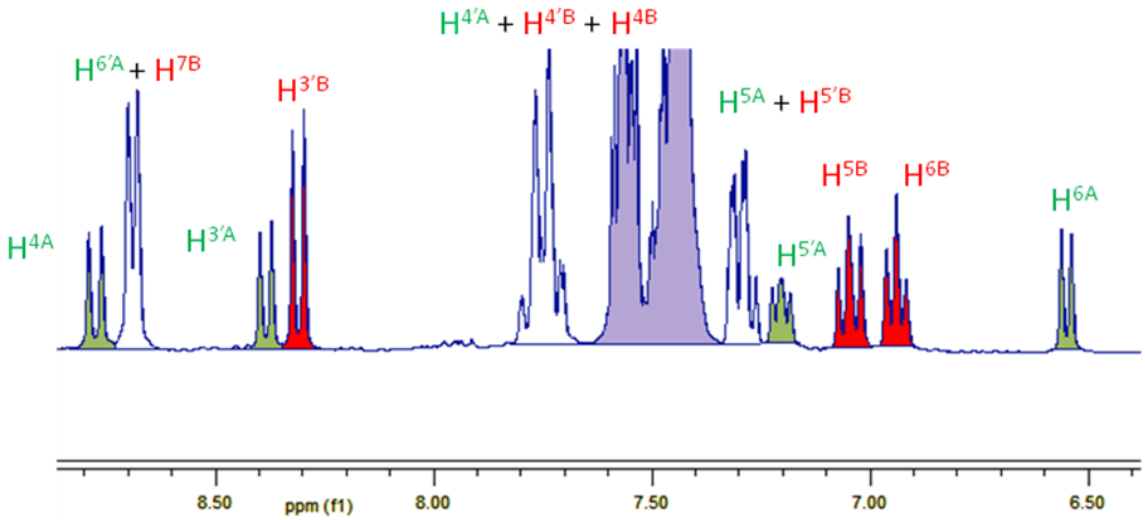


P(^{A}Ph$_2$) and P(^{B}Ph$_2$) are coloured in purple.

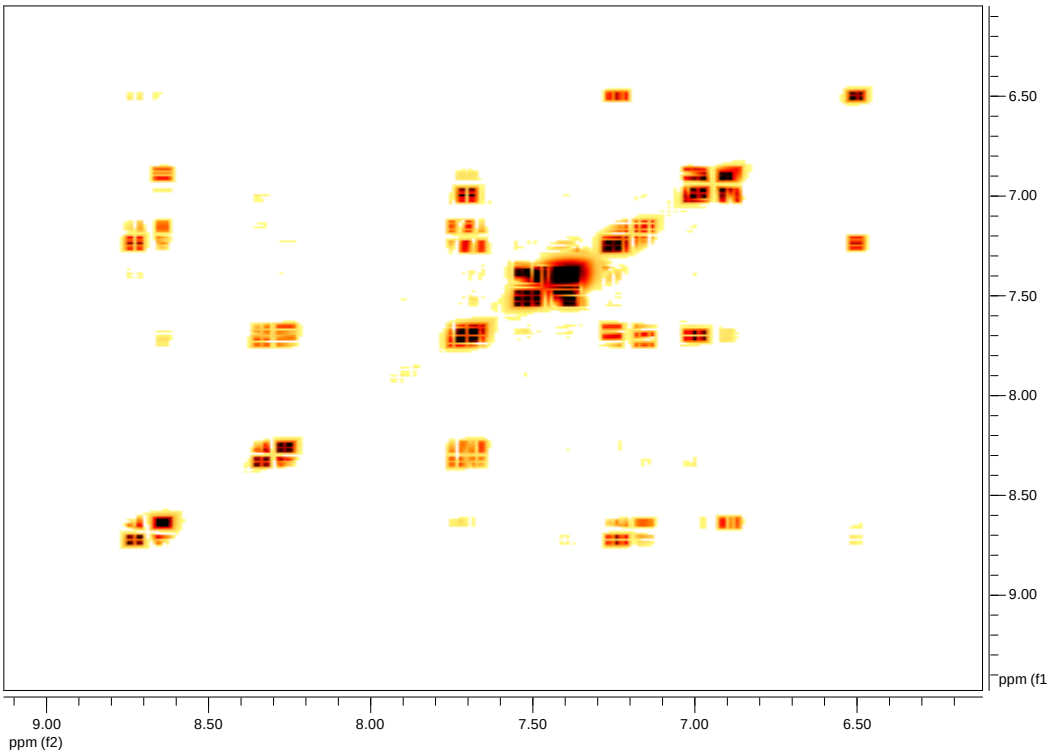
Red colour corresponds to the signals of the major isomer (**B** in this case): $^{3`B}$H, ^{5B}H and ^{6B}H.

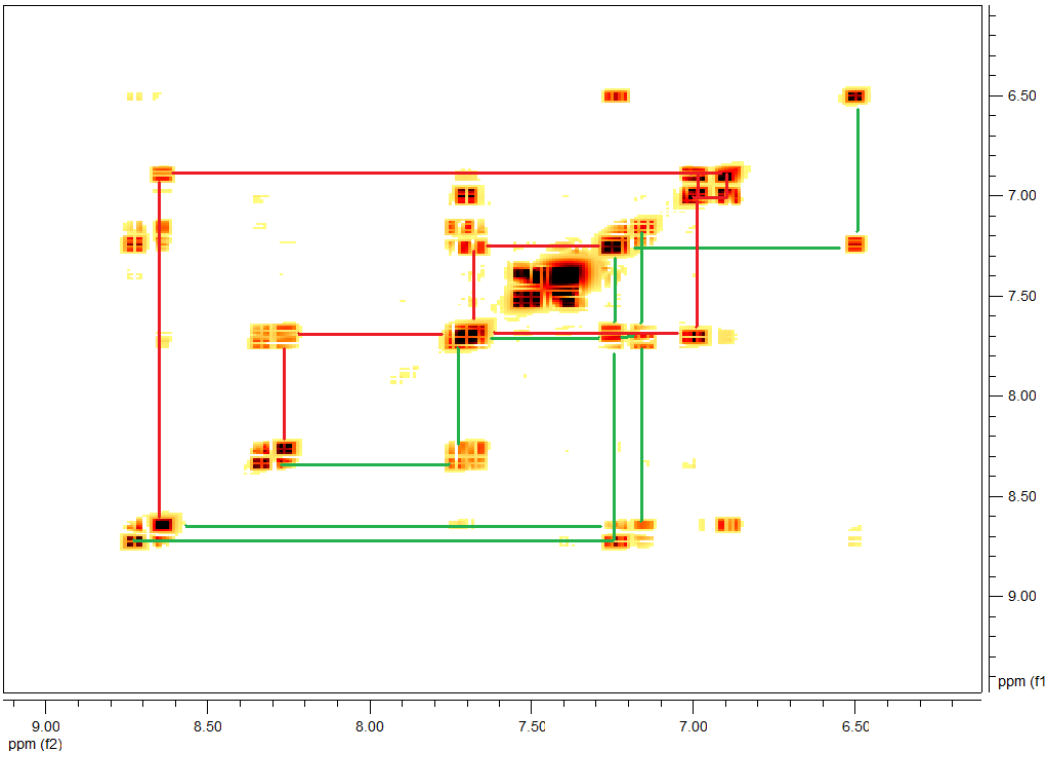
Green colour corresponds to the signals of minor isomer (**A** in this case): ^{4A}H, $^{3'A}$H $^{5'A}$H and ^{6A}H.

No colour is used when more than one hydrogen atom provides a signal at the same chemical shift.

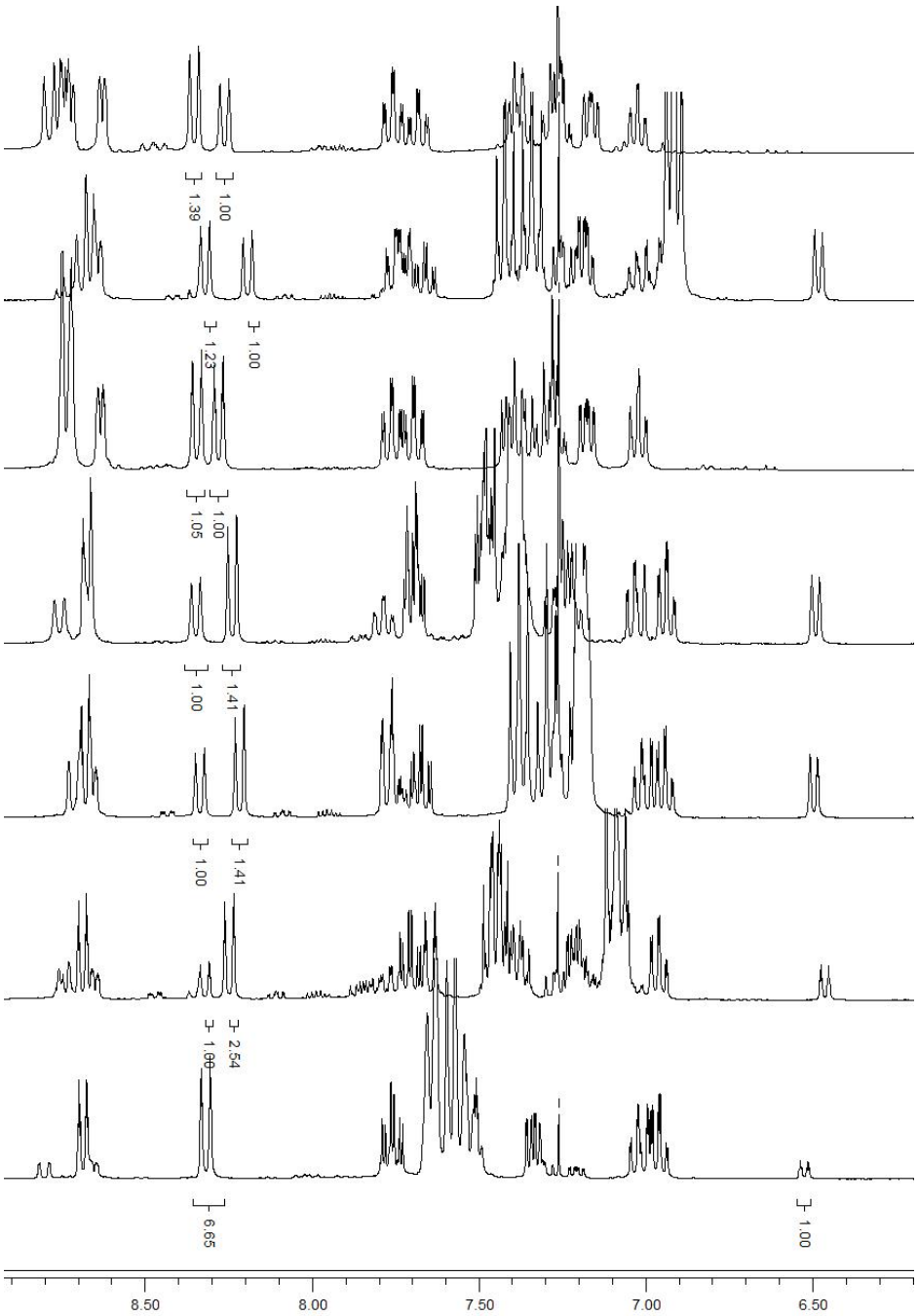


COSY NMR of 2d:



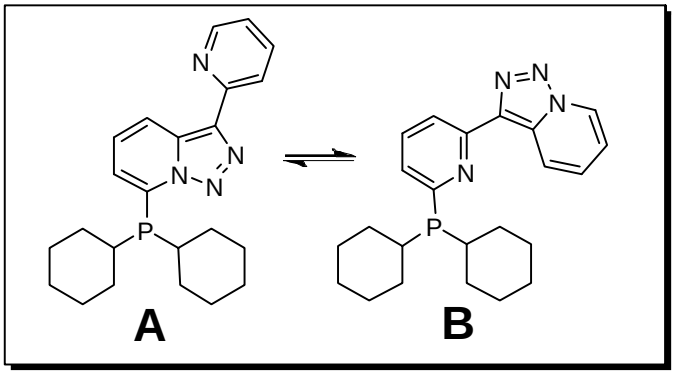


From Up to down: R = PCy_2 (**2a)**, P(*p*-OMePh)$_2$ (**2b)**, P(^{i}Pr)$_2$ (**2c)**, P(Ph)$_2$ (**2d)**, P(*p*-MePh)$_2$ (**2e)**, P(*p*-FPh)$_2$ (**2f)**, P(*p*-CF_3Ph)$_2$ (**2g)**



Experimental data

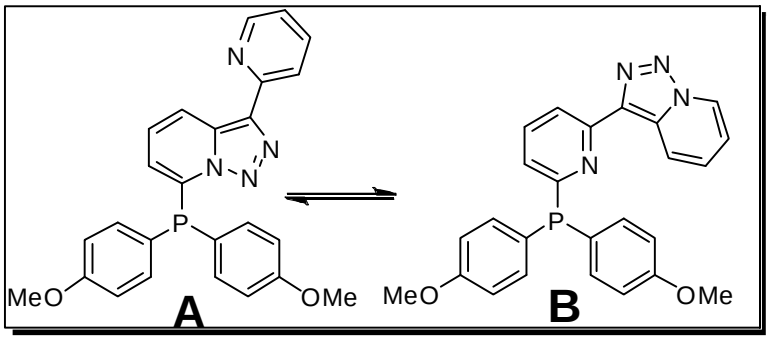
7-(Dicyclohexylphosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (2a-A) and 3-(6'-(dicyclohexylphosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (2a-B).



When dicyclohexylphosphine chloride was used, 0.43 g (22%) were obtained after chromatography; A/B ratio = 1.39. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.78 (d, J = 8.9 Hz, H^{4A}), 8.8-8.7 (m, H^{6A}+H^{7B}), 8.6 (br d, J = 4.9 Hz, $H^{6'A}$), 8.35 (d, J = 7.9 Hz, $H^{3'A}$), 8.26 (d, J = 8.0 Hz, $H^{3'B}$), 7.75 (ddd, J = 7.9, 7.6, 1.8 Hz, $H^{4'A}$), 7.68 (ddd, J = 8.0, 7.7, 1.9 Hz, $H^{4'B}$), 7.4-7.2 (m, H^{5A}+$H^{5'B}$+H^{5B}+H^{4B}), 7.16 (dd, J = 7.6, 4.9, Hz, $H^{5'A}$), 7.02 (ddd, J = 6.9, 6.8, 1.1 Hz, H^{6B}), 2.8-2.6 (m, 1H), 2.3-2.1 (m, 1H), 2.0-1.8 (m, 2H), 1.8-1.5 (m, 7H), 1.3-0.9 (m, 11H). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 160.82 (C), 160.66 (C), 152.11 (CH^{A}), 151.92 (d, $J_{C–P}$ = 5.62 Hz, C), 149.20 (CH^{A}), 137.79 (C), 137.35 (C), 137.14 (C), 136.77 (C), 136.49 (CH^{A}), 135.25 (d, $J_{C–P}$ = 8.92 Hz, CH^{B}), 132.13 (d, $J_{C–P}$ = 16.02 Hz, C), 129.25 (d, $J_{C–P}$ = 33.79 Hz, CH), 126.14 (CH), 125.71 (d, $J_{C–P}$ = 27.09 Hz, CH), 125.40 (d, $J_{C–P}$ = 8.78 Hz, CH), 125.18 (CH), 121.81 (CH), 121.57 (CH), 121.38 (CH), 120.36 (CH), 119.18 (CH), 115.75 (CH), 32.75 (d, $J_{C–P}$ = 11.11 Hz, CH), 32.60 (d, $J_{C–P}$ = 11.29 Hz, CH), 31.06 (d, $J_{C–P}$ = 19.49 Hz, CH_2), 30.06 (d, $J_{C–P}$ = 8.74 Hz, CH_2), 29.74 (d, $J_{C–P}$ = 14.97 Hz, CH_2), 29.16 (d, $J_{C–P}$ = 7.70 Hz, CH_2), 27.44 (CH_2), 27.28 (CH_2), 27.12 (CH_2), 27.01 (CH_2), 26.81 (CH_2), 26.75 (CH_2), 26.63 (CH_2), 26.36 (CH_2), 26.12 (CH_2). – ^{31}P NMR ($CDCl_3$, 161 MHz): δ= 8.92 (P^{B}), 8.01 (P^{A}); $J_{P\text{-}Se}$ = 705.5 Hz. – MS(EI): m/z (%) = 392.2 (24) [M^{+}],

364.2 (18) [M^+- N_2], 309.2 (74) [M^+- C_6H_{11}], 282.1 (74) [M^+- C_6H_{11} -N_2], 199.1 (100) [HM^+- N_2 - 2×C_6H_{11}], 168.1 (58) [HM^+- N_2 – $P(C_6H_{11})_2$]. – HRMS ESI-[TOF] for $C_{23}H_{29}N_4P$ [M+H^+]: calc 393.2203; found 393.2145, [M+O+Li]: calcd. 415.2245; found. 415.2161.

7-(Di(*p*-methoxyphenyl)phosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (**2b-A**) and **3-(6'-(di(*p*-methoxyphenyl)phosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine** (**2b-B**)

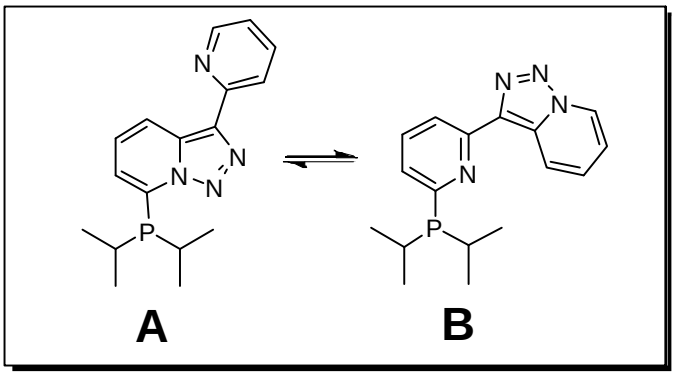


3-(Pyridine-2'-yl)-[1,2,3]triazolo[1,5-*a*]pyridine **1** (0.30 g, 1.5 mmol) and diphenylphosphine chloride were used affording 0.13 g (10%) of **2b** after chromatography; A/B ratio = 1.23. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.7-8.6 (m, $H^{6'A}$+H^{7B} +H^{4A}), 8.32 (d, *J* = 8.0 Hz, $H^{3'A}$), 8.19 (d, *J* = 7.9 Hz, $H^{3'B}$), 7.8-7.7 (m, H^{4B} + $H^{4'A}$), 7.66 (ddd, *J* = 7.9, 7.8, 2.5 Hz, $H^{4'B}$), 7.5-7.3 (m, (Ph)), 7.3-7.1 (m, $H^{5'B}$+H^{5A}+$H^{5'A}$), 7.1-6.9 (m, H^{5B}+H^{6B}+ Ph), 6.48 (d, *J* = 6.8 Hz, H^{6A}), 3.82 (s, $3H^B$), 3.80 (s, $3H^A$). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 164.06 (C), 160.96 (C-OMe), 160.46 (C-OMe), 152.14 (C), 152.03 (d, $J_{C–P}$ = 8.54 Hz, C), 149.11 (CH), 140.19 (C), 139.88 (C), 137.34 (C), 137.14 (d, $J_{C–P}$ = 1.95 Hz,1C), 136.59 (C), 136.55 (CH), 136.18-135.78 (m), 135.54 (C), 134.09 (d, *J* = 10.99 Hz, C), 132.14 (C), 131.83 (C), 127.67 (d, $J_{C–P}$ = 6.50 Hz, C), 126.25 (d, *J* = 27.01 Hz, CH), 125.85 (CH), 124.80 (CH), 123.47 (d, *J* = 5.02 Hz, C), 122.00-121.71 (m), 121.14 (CH), 120.56 (CH), 120.44 (CH), 118.32 (CH), 115.79 (CH), 114.70-113.86 (m), 55.38 (OMe), 55.25 (OMe). – ^{31}P NMR ($CDCl_3$, 161 MHz): δ= -0.35 (P^B), -17.62 (P^A); $J_{P\text{-}Se}$ = 725.4 Hz.

– MS(EI): m/z (%) = 440.1 (87) [M^+], 412.1 (64) [M^+- N_2], 411.1 (100) [M^+ -H - N_2], 305.1 (48) [M^+- MeOPh - N_2], 245.1 (98) [P(*p*-MeOPh)$_2$]. – HRMS ESI-[TOF] for $C_{25}H_{21}N_4O_2P$ [M+H]: calcd. 441.1480; found. 441.1421. $C_{25}H_{21}N_4O_2P$ [M+Li+O]: calcd. 463.1511; found. 463.1446.

7-(Diisopropylphosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (2c-A) and

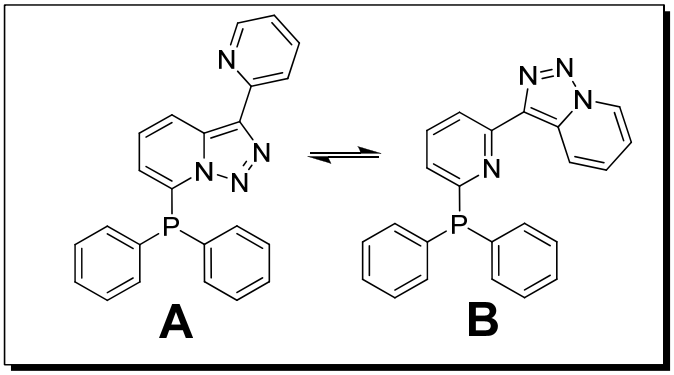
3-(6'-(diisopropylphosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (2c-B).



When diisopropylphosphine chloride was used 0.21 g (13%) were obtained after chromatography; A/B ratio = 1.04. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.8-8.7 (m, H^{4A} +H^{6A}+H^{7B}), 8.66 (br d, *J* = 4.9 Hz, $H^{6'A}$), 8.37 (br d, *J* = 8.0 Hz, $H^{3'A}$), 8.30 (br d, *J* = 8.0 Hz, $H^{3'B}$), 7.78 (ddd, *J* = 7.9, 7.6, 1.8 $H^{4'A}$), 7.72 (ddd, *J* = 7.9, 7.7, 1.9 Hz, $H^{4'B}$), 7.5-7.2 (m, H^{5A}+$H^{5'B}$+H^{5B}+H^{4B}), 7.20 (ddd, *J* = 7.6, 4.9, 1.2 Hz, $H^{5'A}$), 7.1-7.0 (m, H^{6B}), 2.92 (qd, *J* = 13.9, 6.9 Hz, 1H), 2.5-2.3 (m, 1H), 1.3-1.1 (m, 6H), 0.96 (ddd, *J* = 20.3, 12.5, 6.9 Hz, 6 H). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 160.88 (C), 160.72 (C), 152.04 (d, J_{C-P} = 6.13 Hz, C),152.0 (CH) 149.15 (CH), 137.96 (C), 137.66 (C), 137.38 (C), 137.19 (C), 136.51 (CH), 135.27 (d, J_{C-P} = 8.54 Hz,CH), 132.07 (d, J_{C-P} = 15.42 Hz, C), 129.01 (d, J_{C-P} = 32.52 Hz,CH), 126.31 (CH), 125.45 (d, J_{C-P} = 8.17 Hz, CH), 125.27 (d, J_{C-P} = 26.08 Hz,CH), 125.15 (CH), 121.83 (CH), 121.62 (CH), 121.14 (CH), 120.34 (CH), 119.32 (CH), 115.73 (CH), 23.06 (d, J_{C-P} = 11.26 Hz, CH), 22.85 (d, J_{C-P} = 10.75 Hz,CH), 20.65 (d, J_{C-P} = 15.43 Hz, CH_3), 20.43 (d, J_{C-P} = 5.22 Hz, CH_3), 19.68 (d, J_{C-P} = 16.83 Hz, CH_3), 19.20 (d, J_{C-P} = 9.14 Hz, CH_3). – ^{31}P NMR ($CDCl_3$, 161 MHz): δ= 17.07 (P^B), 16.92 (P^A); $J_{P\text{-}Se}$ =

713.5 Hz. – MS(EI): m/z (%) = 312.2 (23) [M^+], 284.2 (8) [M^+- N_2], 269.2 (95) [M^+- C_3H_7], 241.1 (49) [M^+- C_3H_7 -N_2], 199.1 (100) [HM^+- N_2 - 2×C_6H_{11}], 168.1 (29) [HM^+- N_2 – $P(C_6H_{11})_2$]. – HRMS ESI-[TOF] for $C_{17}H_{21}N_4P$ [M+Li-N_2]: calc 305.1552; found 307.1542, $C_{17}H_{21}N_4P$ [M+O+Li]: calcd. 335.1613; found.335.1581.

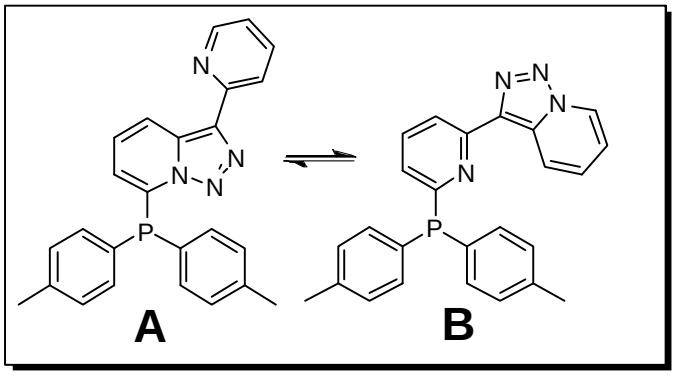
7-(Diphenylphosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-*a*]pyridine (**2d-A**) and **3-(6'-(diphenylphosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-*a*]pyridine** (**2d-B**)



When diphenylphosphine chloride was used 0.79 g (40%) were obtained after chromatography. A/B ratio = 0.72. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.77 (d, *J* = 8.8 Hz, H^{4A}), 8.69 (app d, *J* = 6.3 Hz, $H^{6'A}$+H^{7B}), 8.38 (d, *J* = 8.0 Hz, $H^{3'A}$), 8.31 (d, *J* = 8.0 Hz, $H^{3'B}$), 7.8-7.6 (m, $H^{4'A}$ + $H^{4'B}$ + H^{4B}), 7.6-7.3 (m, $(PPh_2)^A$ + $(PPh_2)^B$), 7.3-7.2 (m, H^{5A} + $H^{5'B}$), 7.2-7.1 (m, $H^{5'A}$), 7.1-7.0 (app t, *J* = 8.6, Hz, H^{5B}), 6.94 (app t, *J* = 6.8, Hz, H^{6B}), 6.55 (d, *J* = 6.8 Hz, H^{6A}). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 162.74 (s,2 C), 152.12 (d, *J* = 8.61 Hz, C), 151.91 (C), 149.02 (CH^A), 138.66 (d, J_{C-P} = 23.20 Hz, C), 137.20 (d, J_{C-P} = 1.84 Hz, C), 136.98 (C), 136.31 (CH^A), 136.30 (d, J_{C-P} = 9.69 Hz, C(PPh_2), 135.96 (d, J_{C-P} = 5.81 Hz, CH^A), 134.29 (d, *J* = 19.71 Hz, CH (PPh_2)), 134.03 (d, *J* = 20.48 Hz, CH^B), 132.31 (d, J_{C-P} = 8.31 Hz, C), 131.96 (C), 131.66 (C), 129.66 (CH^B), 128.79 (CH(PPh_2)), 128.74 (d, J_{C-P} = 7.78 Hz, CH^B), 128.39 (d, J_{C-P} = 7.34 Hz, CH(PPh_2)), 126.54 (d, J_{C-P} = *2*7.79 Hz, CH$(PPh_2)^A$), 125.96 (CH^B),125.72 (CH^A),124.63 (CH^A), 121.55 (d, J_{C-P} = 22.59 Hz, CH^A), 121.37 (CH^B), 120.68 (CH^A),120.30 (CH^A), 118.52 (CH^B), 115.63 (CH^B). – ^{31}P NMR ($CDCl_3$, 161 MHz): δ= -0.30 (P^B), -14.77 (P^A); $J_{P\text{-}Se}$ = 736.4 Hz. – MS(EI): m/z (%) =

380 (66) [M^+], 352 (67) [M^+- N_2], 275.1(8) [M^+- Ph - N_2], 183.1 (100). – HRMS ESI-[TOF] for $C_{23}H_{17}N_4P$ [M^++O+Li]: calcd. 403.1300; found. 403.1230.

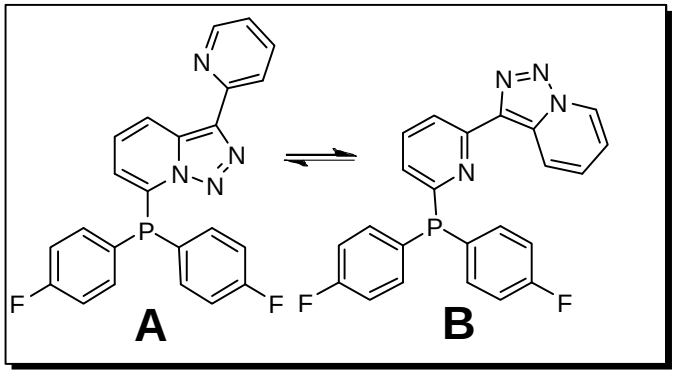
7-(Di(*p*-methylphenyl)phosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (**2e-A**) and **3-(6'-(di(*p*-methylphenyl)phosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine** (**2e-B**)



3-(Pyridine-2'-yl)-[1,2,3]triazolo[1,5-*a*]pyridine **1** (0.30 g, 1.5 mmol) and diphenylphosphine chloride were used affording 0.14 g (22%) of **2e** after chromatography; A/B ratio = 0.71. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.7-8.6 (m, H^{4A}+$H^{6'A}$+ H^{7B}), 8.33 (br d, *J* = 8.1 Hz, $H^{3'A}$), 8.22 (br d, *J* = 7.9, Hz, $H^{3'B}$), 7.8-7.7 (m, H^{4B}+$H^{4'A}$), 7.67 (ddd, *J* = 7.9, 7.8, 2.5 Hz, $H^{4'B}$), 7.4-7.3 (m, $H^{5'A}$+ H^{5A} +(*p*-MePh)), 7.2-7.1 (m, $H^{5'B}$+(*p*-MePh)), 7.1-6.9 (m, H^{6B}+H^{5B}), 6.42 (d, *J* = 6.8 Hz, H^{6A}), 2.38 (s, $3H^B$), 2.36 (s, $3H^A$). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 163.56 (C), 152.15 (C), 152.03 (C), 149.09 (CH), 139.87 (C), 139.74 (C), 139.43 (C), 137.35 (C), 136.54 (CH), 136.01 (d, J_{C-P} = 5.36 Hz, CH), 134.45 (d, J_{C-P} = 20.16 Hz, CH), 134.17 (d, J_{C-P} = 21.50 Hz, CH), 133.12 (d, J_{C-P} = 8.19 Hz, C), 132.15 (s, C), 131.83 (C), 129.69 (d, J_{C-P} = 8.08 Hz, CH), 129.31 (d, J_{C-P} = 7.60 Hz, CH), 129.06 (d, J_{C-P} = 6.81 Hz, C), 126.46 (d, J_{C-P} = 26.41 Hz, CH), 125.86 (CH), 125.79 (CH), 124.81 (CH), 121.89-121.75 (m), 121.30 (CH), 120.61-120.48 (m), 118.46 (CH), 115.74 (CH), 21.34 (CH_3), 21.01 (CH_3). – ^{31}P NMR ($CDCl_3$, 161 MHz): δ= -2.06 (P^B), -16.34 (P^A); J_{P-Se} = 729.6 Hz. – MS(EI): m/z (%) = 408.2 (72) [M^+], 396 (100) [OM^+- N_2], 396.2 (100) [MO^+

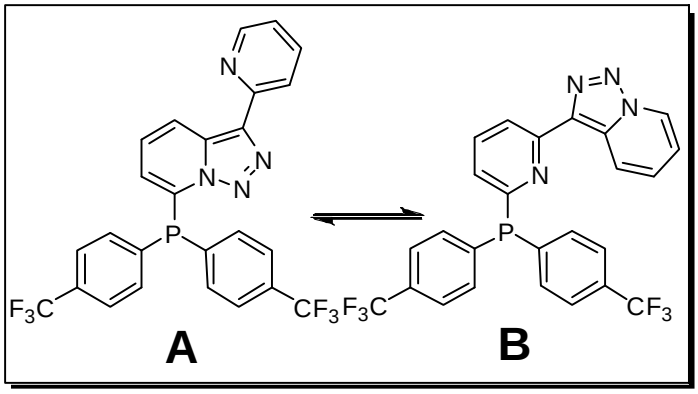
- N_2], 380.2 (68) [M^+- N_2], 289.1(53) [M^+- *p*-MePh - N_2]. – HRMS ESI-[TOF] for $C_{25}H_{21}N_4P$ [M+Li+O]: calcd. 431.1613; found. 431.1537.

7-(Di(*p*-fluorophenyl)phosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (**2f-A**) and **3-(6'-(di(*p*-fluorophenyl)phosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine** (**2f-B**)



3-(Pyridine-2'-yl)-[1,2,3]triazolo[1,5-*a*]pyridine **1** (0.30 g, 1.5 mmol) and di(*p*-fluorophenyl)phosphine chloride afforded 0.43 g (66%) of **2f** after chromatography; A/B ratio = 0.40. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.74 (d, *J* = 8.9 Hz, H^{4A}), 8.68 (d, *J* = 7.0 Hz, H^{7B}), 8.65 (d, *J* = 4.9 Hz, $H^{6'A}$), 8.32 (d, *J* = 8.0 Hz, $H^{3'A}$), 8.24 (d, *J* = 8.0 Hz, $H^{3'B}$), 7.8-7.7 (m, $H^{4'A}$), 7.7 (ddd, *J* = 7.9, 7.8, 2.5 Hz, $H^{4'B}$), 7.64 (br d, *J* = 8.9 Hz, H^{4B}), 7.42 (m, (Ph-F)), 7.3-7.2 (m, H^{5A} + $H^{5'A}$ + $H^{5'B}$), 7.1-7.0 (m, H^{5B} + (Ph-F)), 6.96 (ddd, *J* = 6.9, 6.7, 1.4 Hz, H^{6B}), 6.46 (d, *J* = 6.8 Hz, H^{6A}). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 163.99 (d, J_{C-F} = 251.04 Hz, C), 163.58 (d, J_{C-F} = 249.60 Hz, C), 162.41 (C), 152.40 (d, J_{C-P} = 8.23 Hz, C), 151.89 (C), 149.16 (CH), 137.05 (C), 136.61-135.97 (m), 132.14-131.75 (m), 126.60 (d, J_{C-P} = 29.40 Hz, CH), 126.10 (CH), 125.81 (CH), 124.96 (CH), 122.01 (CH), 121.42 (d, *J* = 1.38 Hz, CH), 121.29 (CH), 120.58 (CH), 118.92 (CH), 116.55-115.57 (m). – ^{31}P NMR ($CDCl_3$, 161 MHz): δ= -3.22 (t, J_{P-F} = 4.03 Hz, P^B), -17.16 (t, J_{P-F} = 4.05 Hz, P^A); J_{P-Se} = 744.4 Hz. – MS(EI): m/z (%) = 416.1 (39) [M^+], 404.1 (80) [M^+- N_2 + O] 388.1 (100) [M^+- N_2], 293.1(90) [M^+- *p*-F-Ph - N_2], 219.1 (65) [M^+- N_2 - *p*-F-Ph - Py]. – HRMS ESI-[TOF] for $C_{23}H_{15}F_2N_4P$ [M+O+Li]: calcd. 439.1112; found. 439.1081.

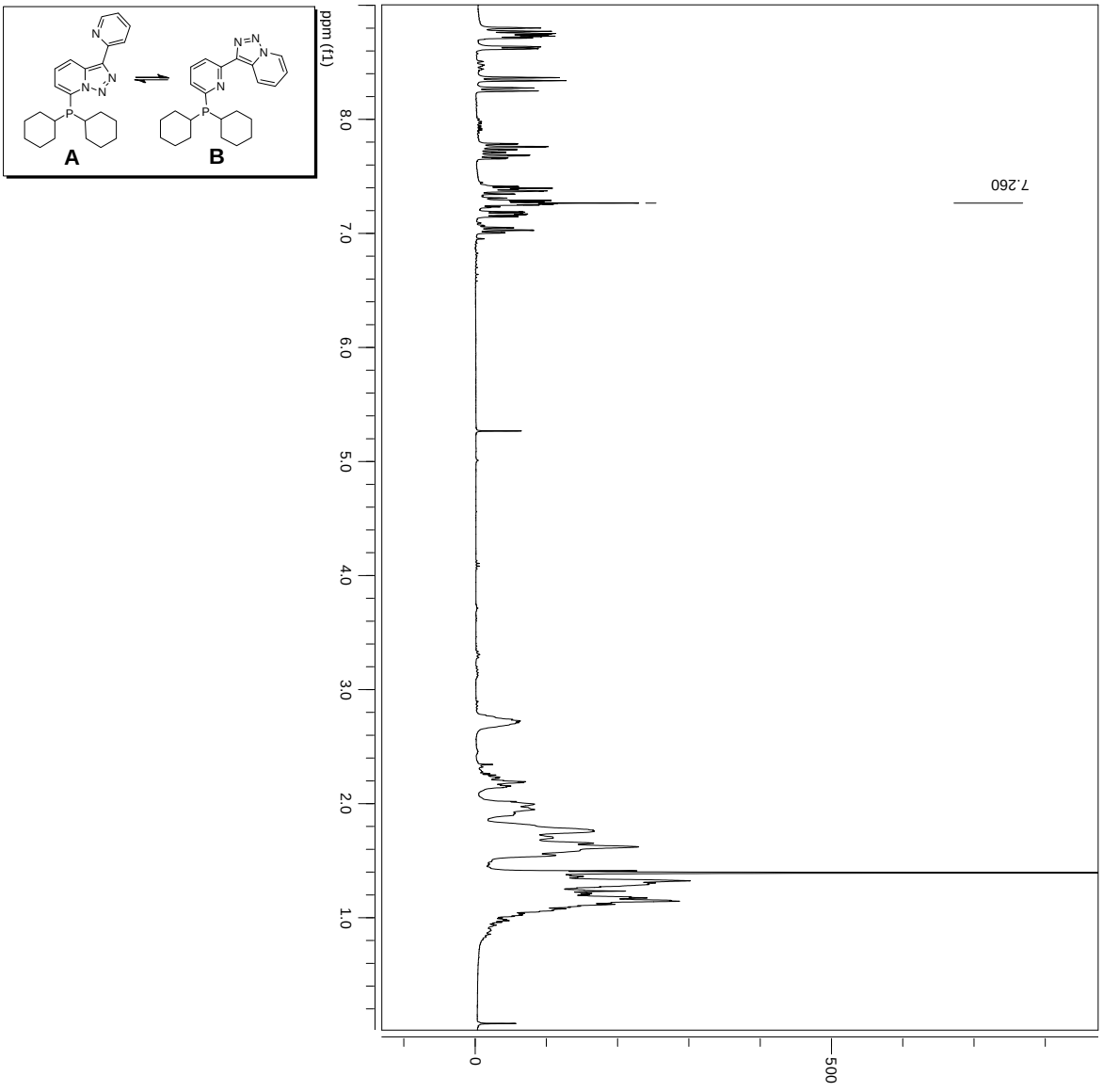
7-(Di(*p*-trifluoromethylphenyl)phosphino)-3-(pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine (**2g-A**) and **3-(6'-(di(*p*-trifluoromethylphenyl)phosphino)pyridin-2'-yl)-[1,2,3]triazolo[1,5-a]pyridine** (**2g-B**)

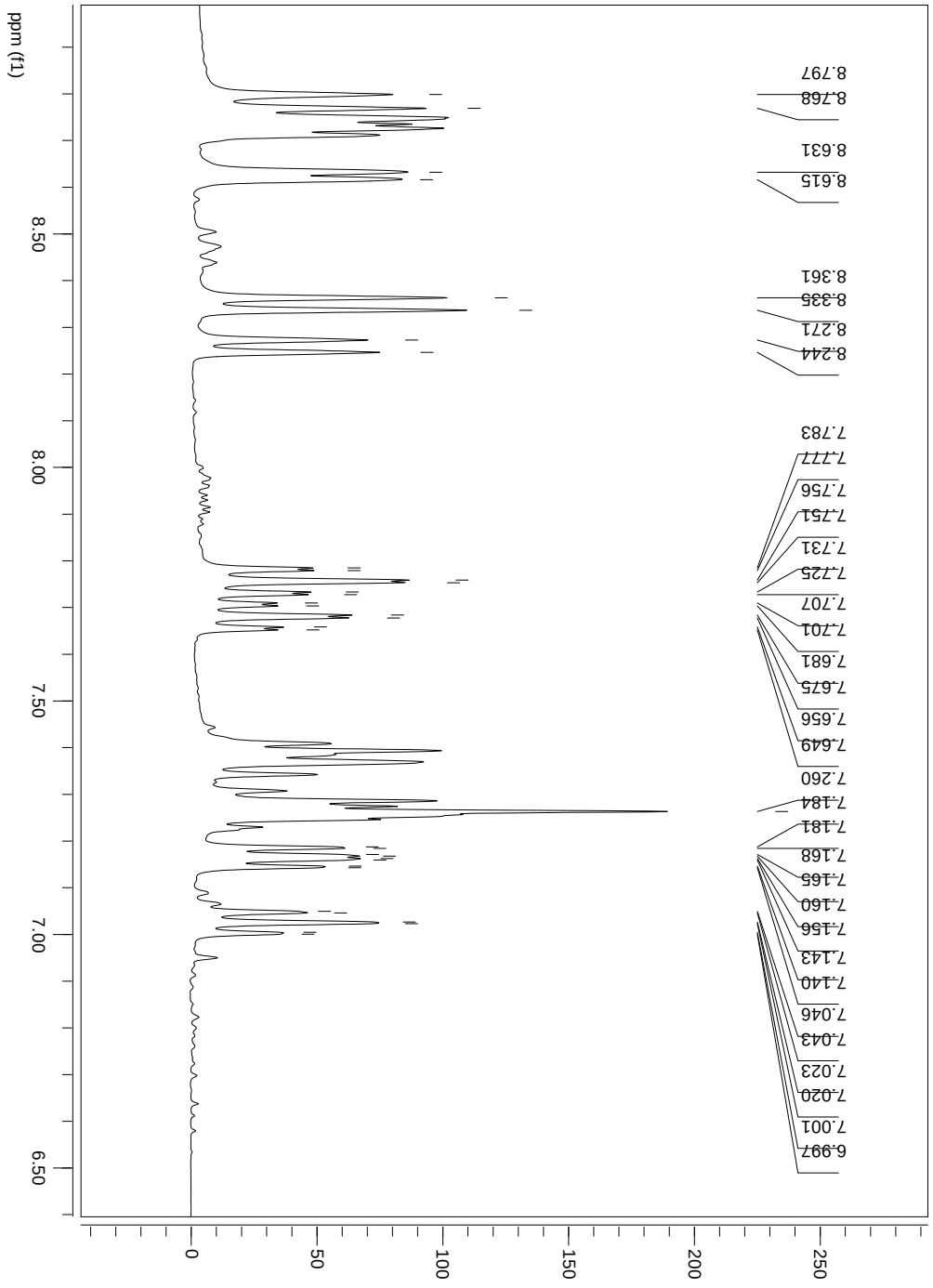


3-(Pyridine-2'-yl)-[1,2,3]triazolo[1,5-*a*]pyridine **1** (0.30 g, 1.5 mmol) and di(*p*-trifluorophenyl)phosphine chloride were used affording 0.36 g (46%) of **2g** after chromatography; A/B ratio = 0.18. – ^{1}H NMR (300 MHz, $CDCl_3$): δ = 8.80 (d, *J* = 8.7 Hz, H^{4A}), 8.7-8.6 (m, H^{7B}+$H^{6'A}$), 8.32 (app d, *J* = 8.0 Hz, $H^{3'A}$+$H^{3'B}$), 7.77 (dd, *J* = 7.9, 7.7 Hz, $H^{4'B}$), 7.76 (ddd, *J* = 7.9, 7.7, 2.8 Hz, $H^{4'A}$), 7.7-7.5 (m, (*p*-CF_3Ph)$_2$+ H^{4B}), 7.4-7.3 (m, $H^{5'A}$+ $H^{5'B}$), 7.21 (dd, *J* = 8.7, 6.8 Hz, H^{5A}), 7.1-6.9 (m, H^{5B}+ H^{6B}), 6.53 (d, *J* = 6.8 Hz, H^{6A}). – ^{13}C NMR (75.5 MHz, $CDCl_3$): δ = 160.34 (C), 152.92 (d, $J_{C\text{-}P}$= 7.75 Hz, C), 151.68 (C), 149.19 (CH), 140.82 (d, $J_{C\text{-}P}$ = 13.06 Hz, C), 137.81 (C), 136.78 (C), 136.71-136.47 (C+CH), 134.54 (d, $J_{C\text{-}P}$ = 19.91 Hz, CH), 134.46 (d, *J* = 21.50 Hz, CH), 132.13 (q, $^{2}J_{C\text{-}F}$ = 33.7 Hz,1 C^{A}),132.03 (C), 131,8 (C), 131.44 (C), 131.23 (q, $^{2}J_{C\text{-}F}$ = 32.6 Hz,1C^{B}), 131.01 (C), 130.58 (C), 127.50 (d, $J_{C\text{-}P}$ = 33.58 Hz, CH), 126.27 (CH), 125.94-125.63 (m), 125.50-125.16 (m), 125.06 (CH), 123.91 (q, $J_{C\text{-}F}$= 272,3 Hz, C), 122.19-121.83 (m), 120.78 (CH), 120.60 (CH), 119.71 (CH), 118.50 (CH), 115.90 (CH); ^{31}P NMR ($CDCl_3$, 161 MHz): δ= -1.15 (P^{B}), -15.31 (P^{A}); $J_{P\text{-}Se}$ = 760.6 Hz; MS(EI): m/z(%) = 516.1 (12) [M^+], 488.1 (80) [M^+- N_2], 293.1(100) [M^+- *p*-CF_3-Ph - N_2]. – HRMS ESI-[TOF] for $C_{25}H_{15}F_6N_4P$ [M+K]: calcd. 555.0576 ; found. 555.0513.

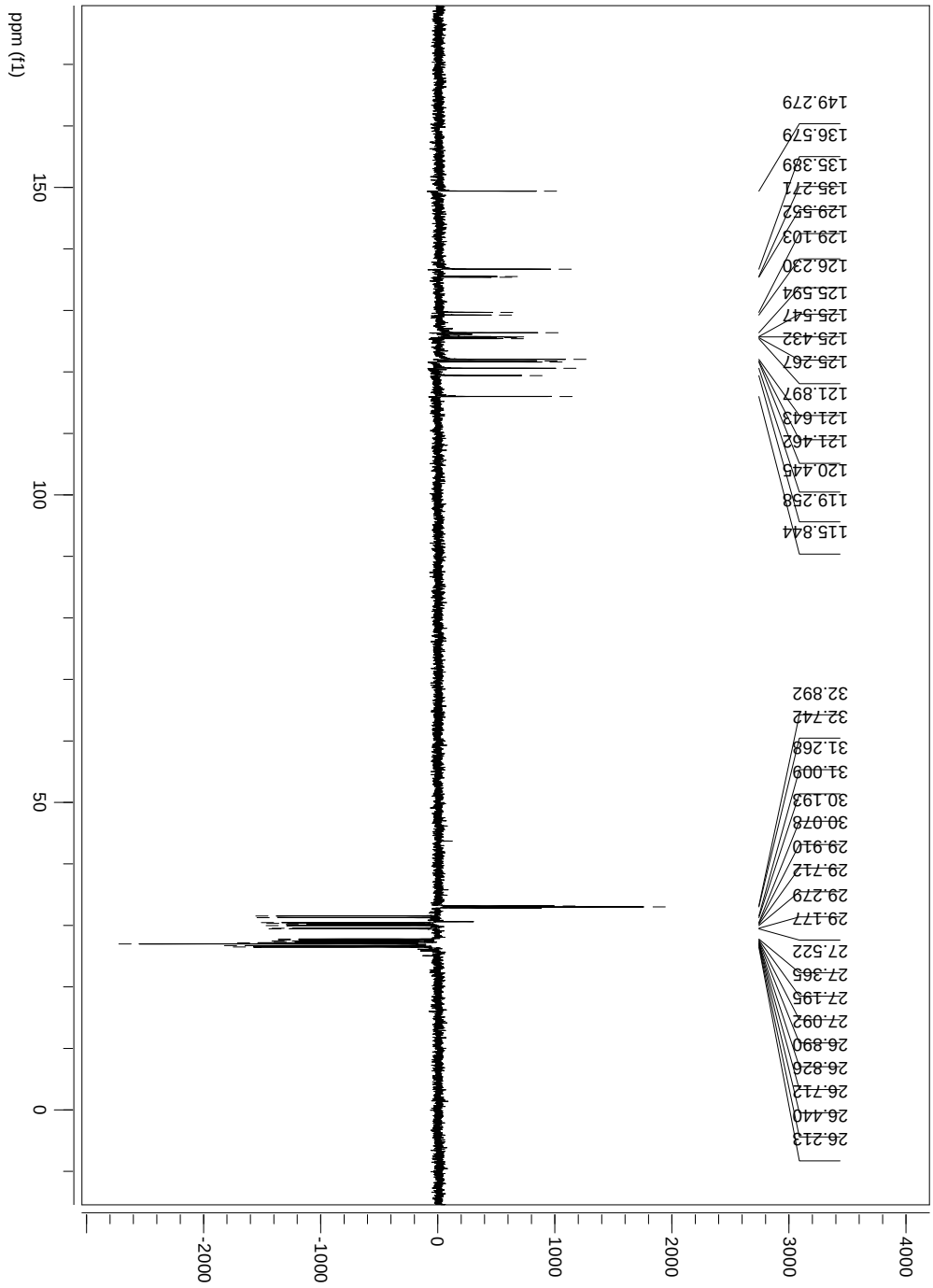
Copies of ^{1}H, ^{13}C, ^{31}P NMR and COSY NMR spectra

^{1}H NMR of 2a:

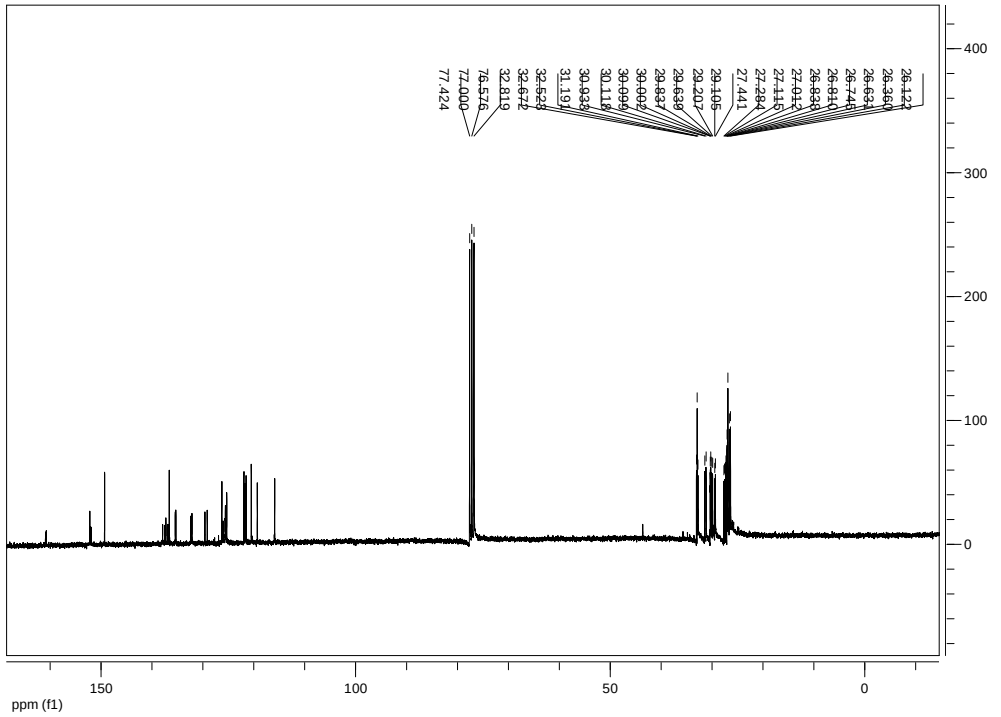


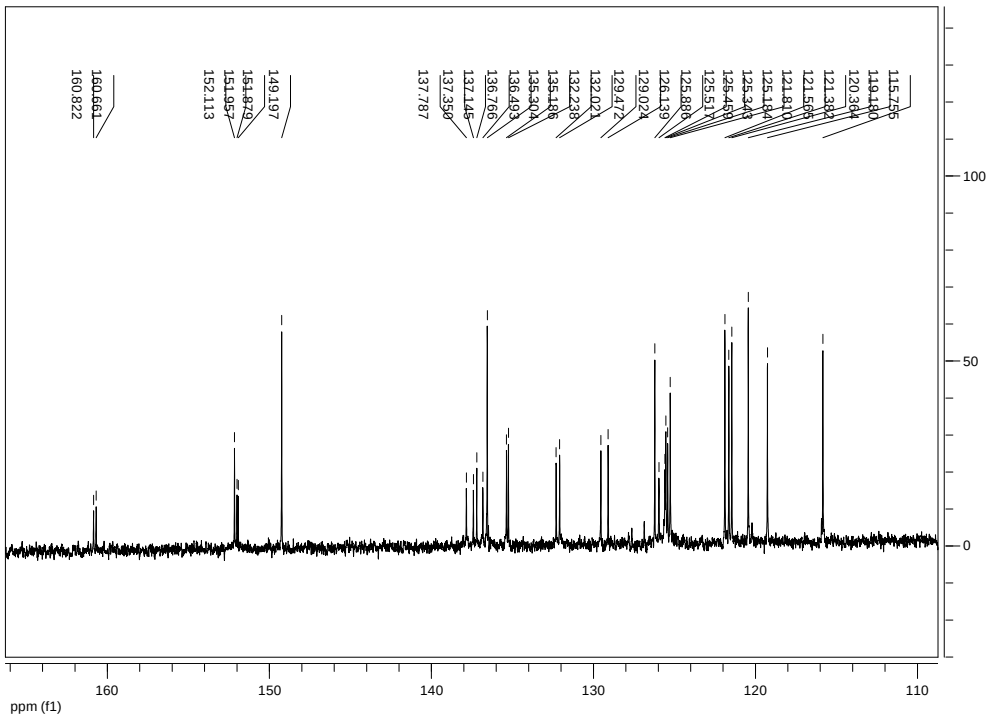

ppm (f1)
8.50
8.00
7.50
7.00
6.50
0
50
100
150
200
250
8.797
8.768
8.631
8.615
8.361
8.335
8.271
8.244
7.783
7.777
7.756
7.751
7.731
7.725
7.707
7.701
7.681
7.675
7.656
7.649
7.260
7.184
7.181
7.168
7.165
7.160
7.156
7.143
7.140
7.046
7.043
7.023
7.020
7.001
6.997

^{13}C NMR DEPT of 2a:

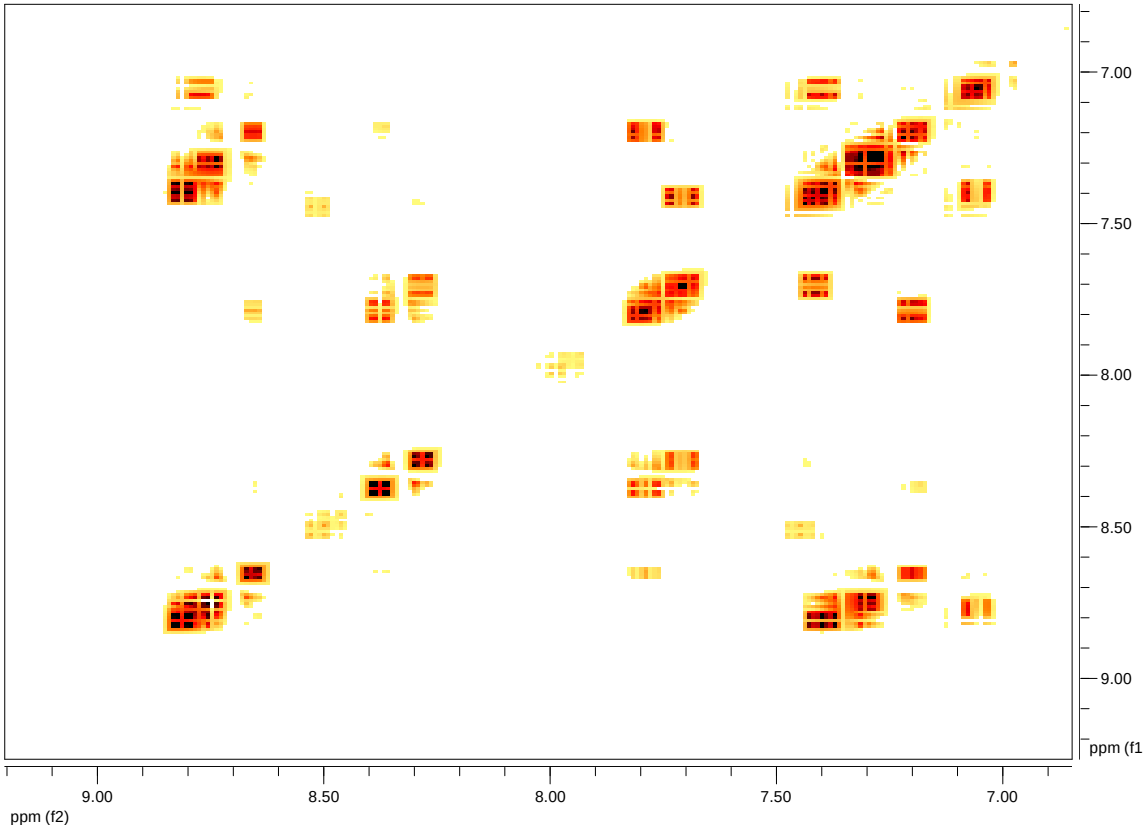


^{13}C NMR of 2a:

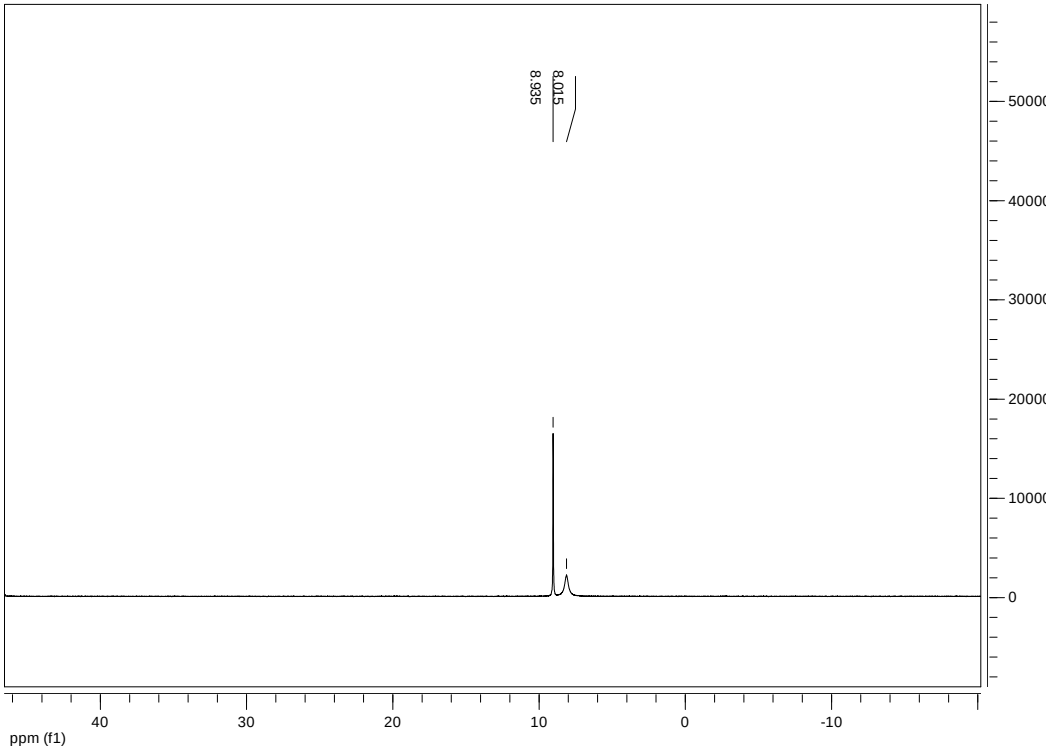

77.424
77.000
76.576
32.819
32.672
32.525
31.197
30.938
30.118
30.000
29.837
29.639
29.207
29.105
27.441
27.284
27.115
27.012
26.938
26.810
26.748
26.633
26.360
26.127
400
300
200
100
0
150
100
50
0
ppm (f1)


160.822
160.661
152.113
151.957
151.879
149.197
137.787
137.350
137.145
136.766
136.493
135.304
135.108
132.238
132.021
129.472
129.024
126.139
125.888
125.517
125.460
125.343
125.184
121.810
121.595
121.382
120.364
119.130
115.755
100
50
0
160
150
140
130
120
110
ppm (f1)

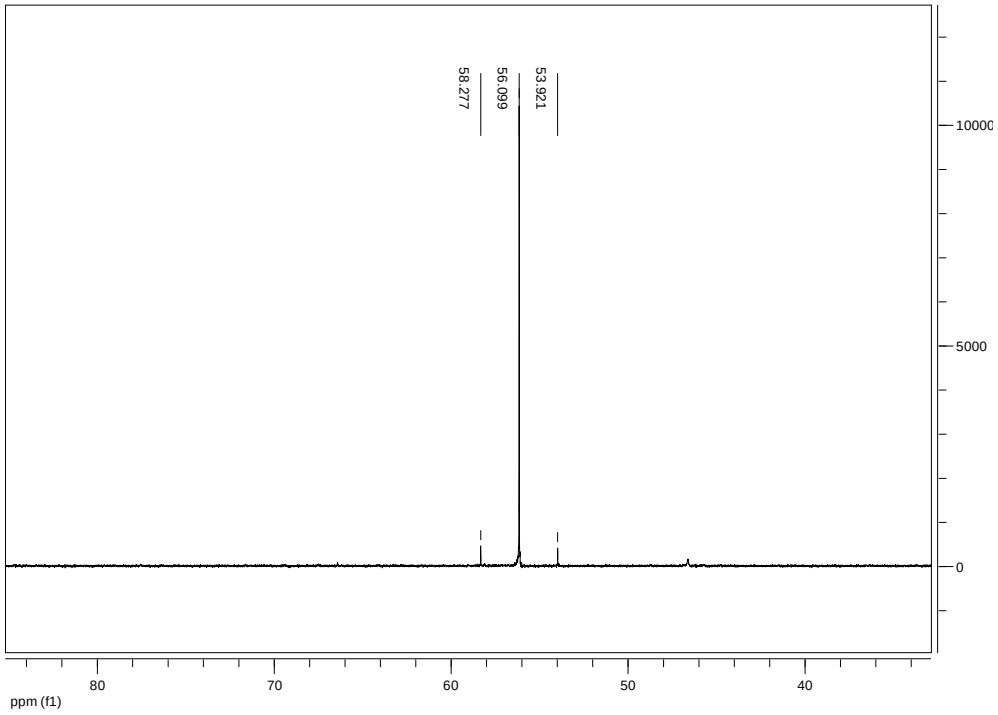
COSY H-H of 2a:



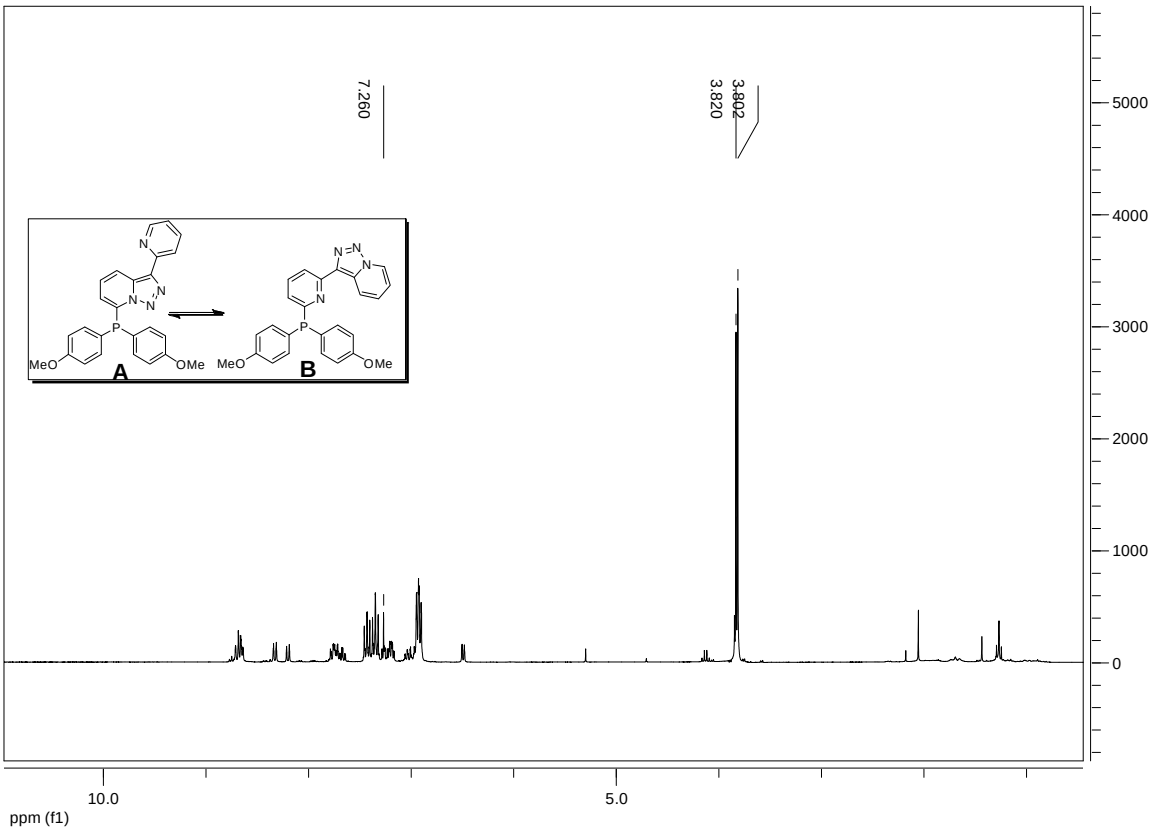
^{31}P NMR of 2a:

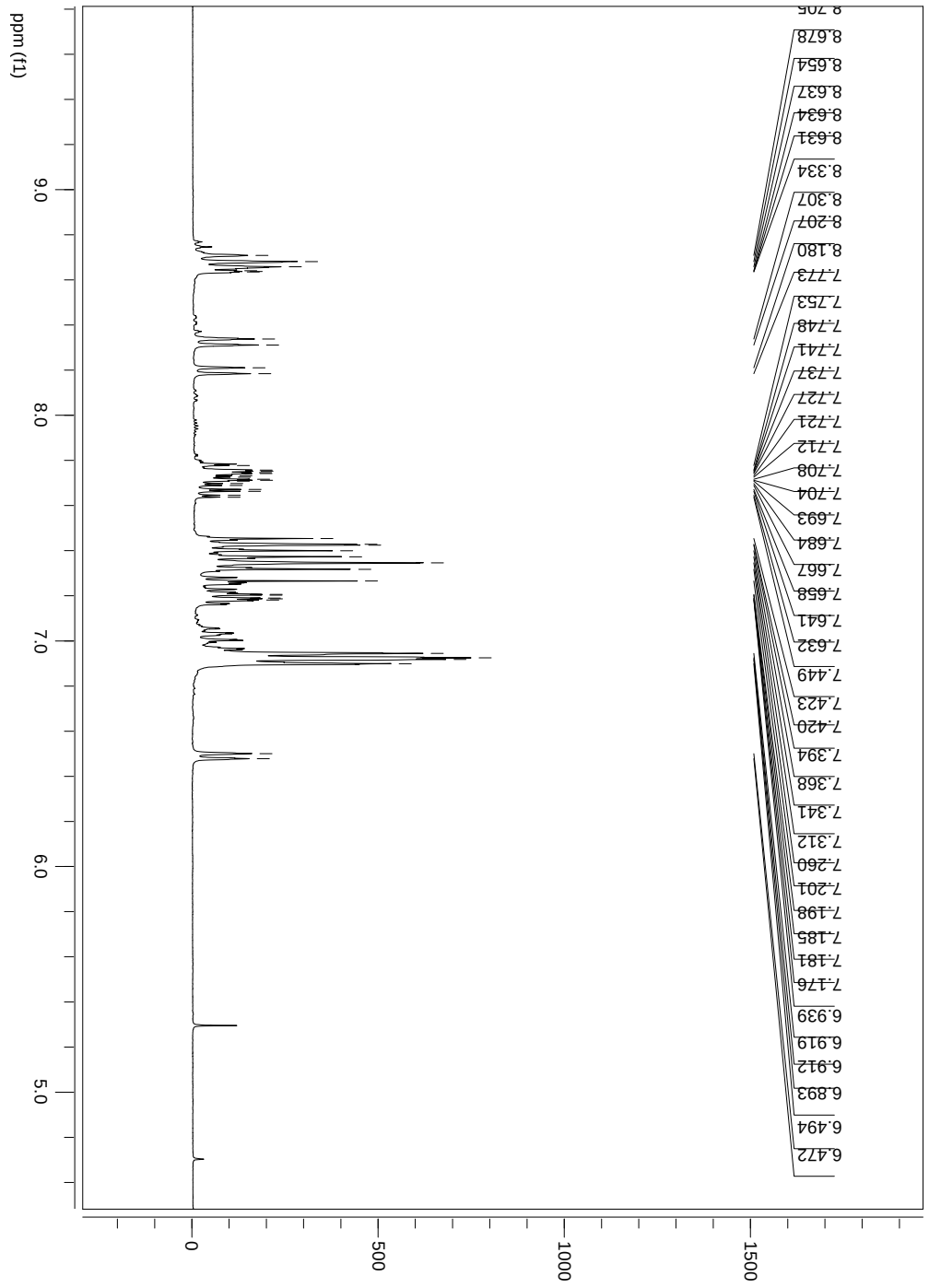


^{31}P NMR of 3a:

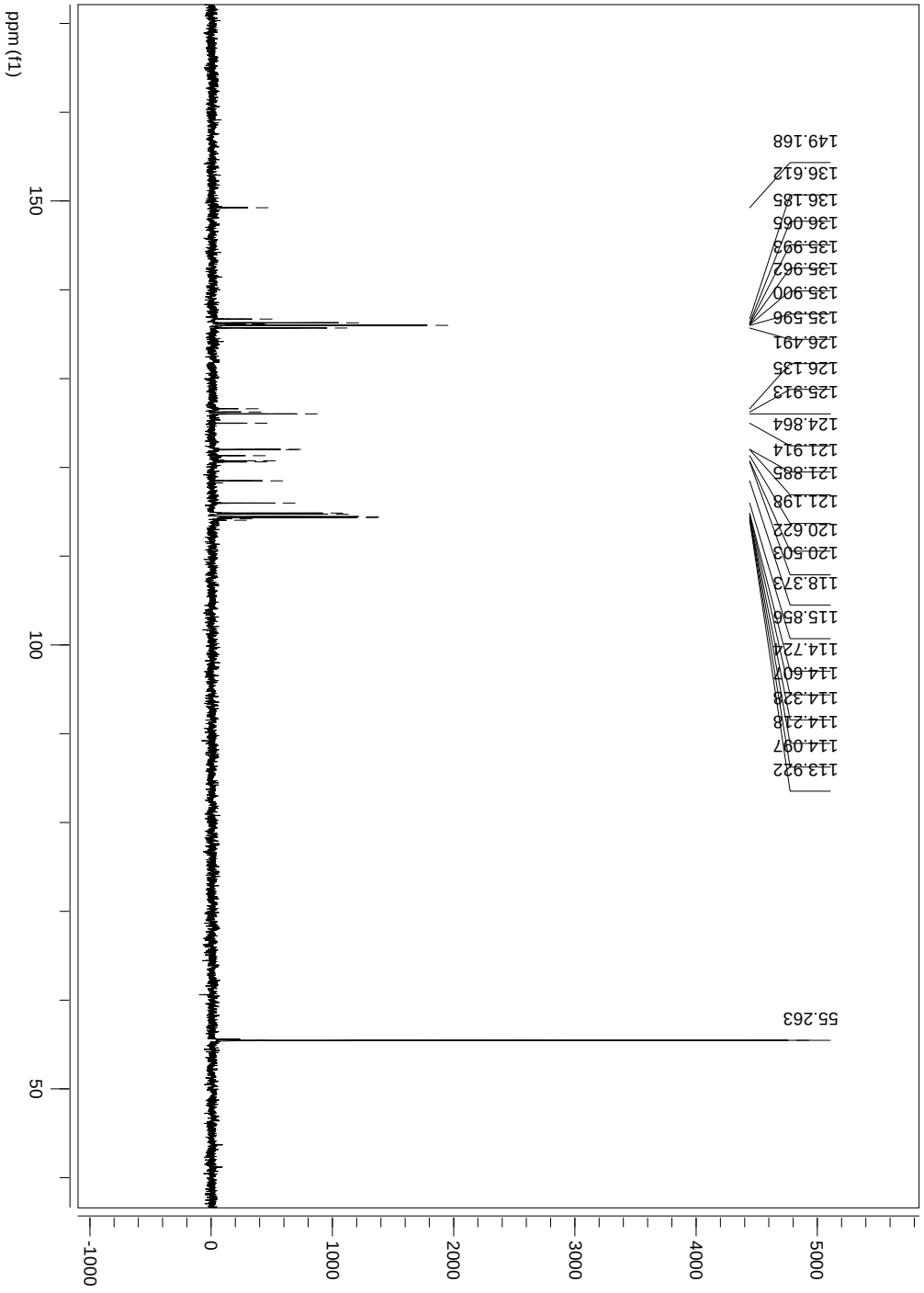


^{1}H NMR of 2b:

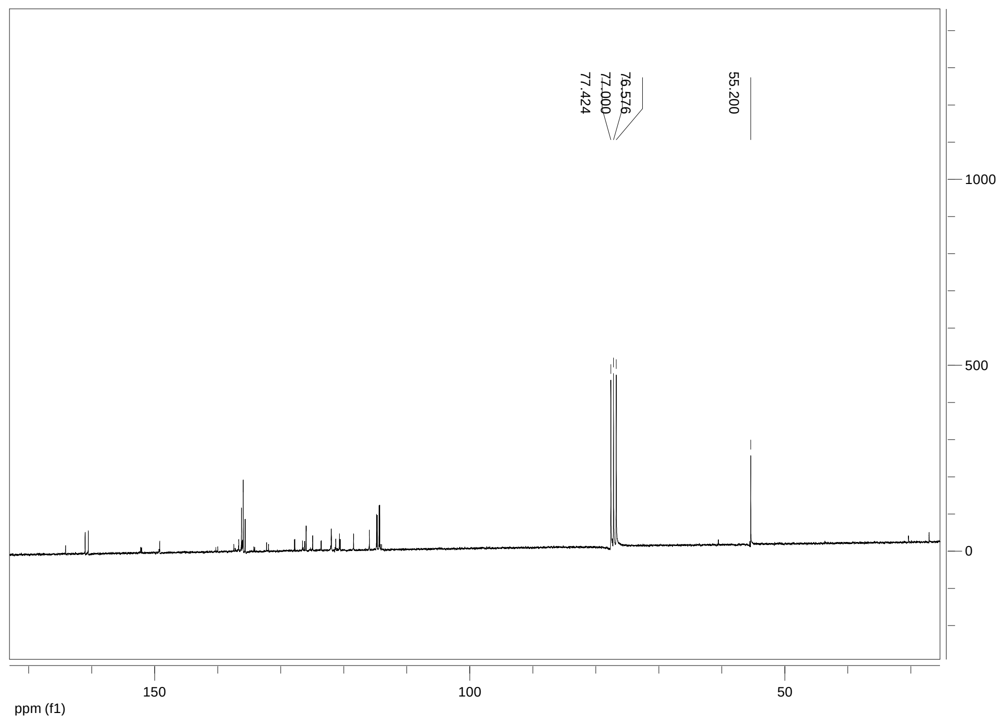

7.260
3.820
3.802
5000
4000
3000
2000
1000
0
MeO
OMe
A
MeO
OMe
B
10.0
5.0
ppm (f1)

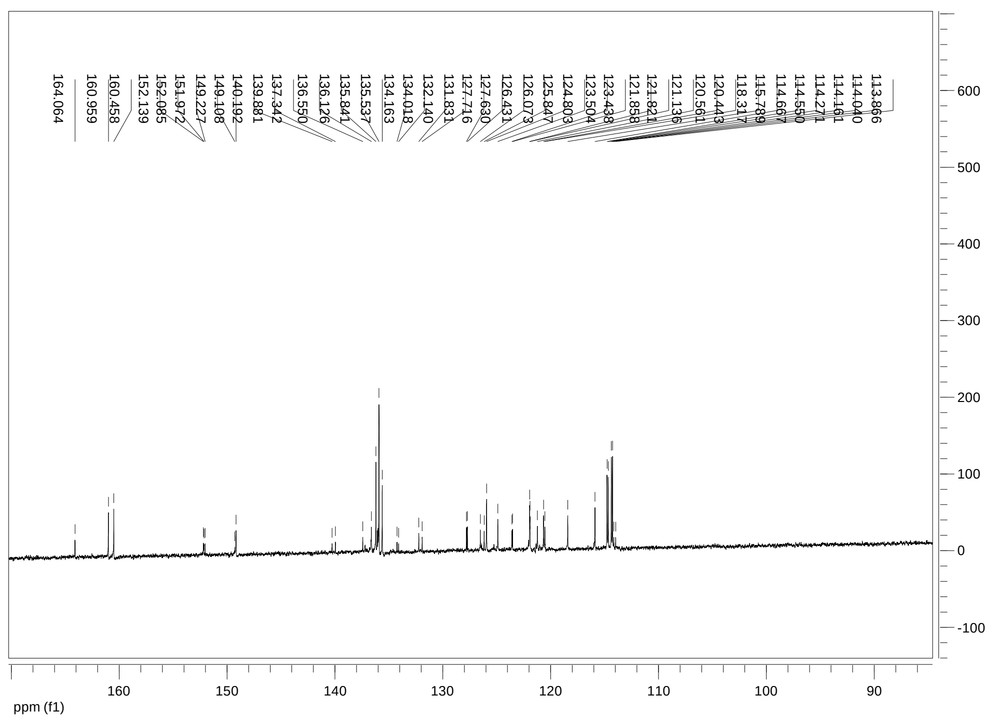


^{13}C NMR DEPT of 2b:

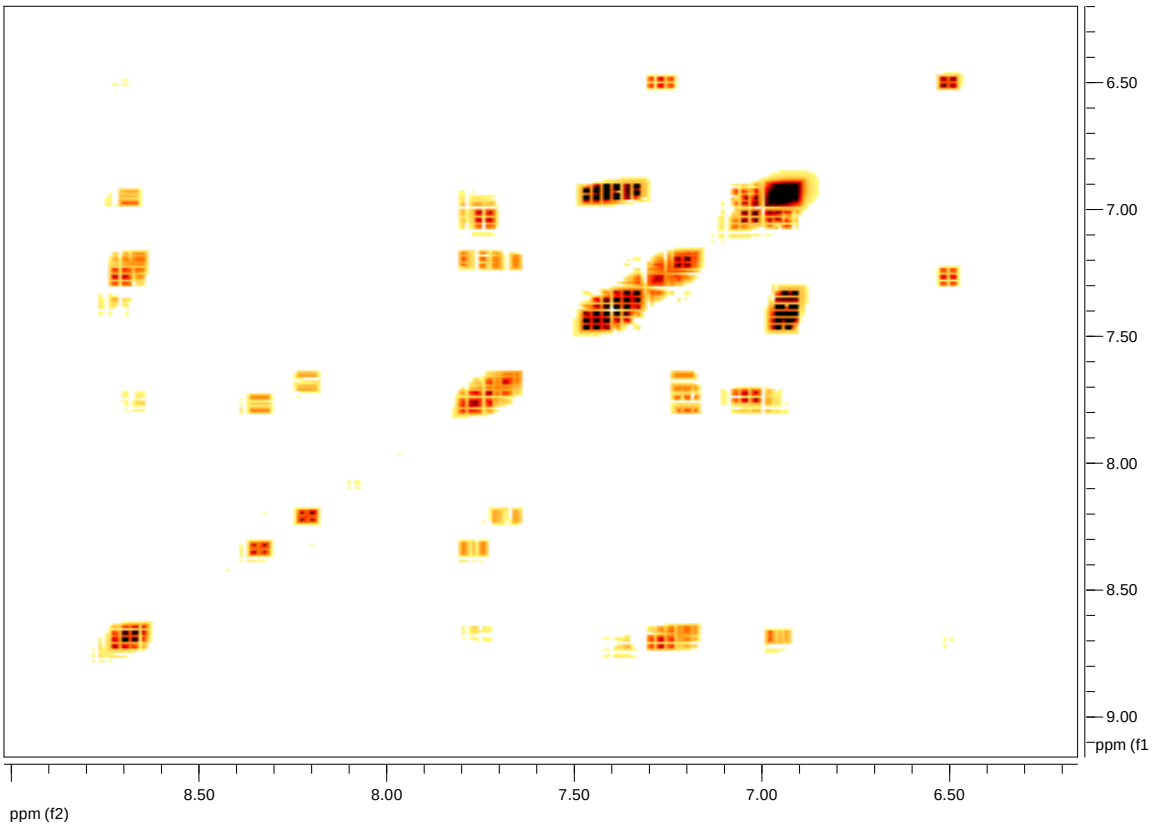


^{13}C NMR of 2b:

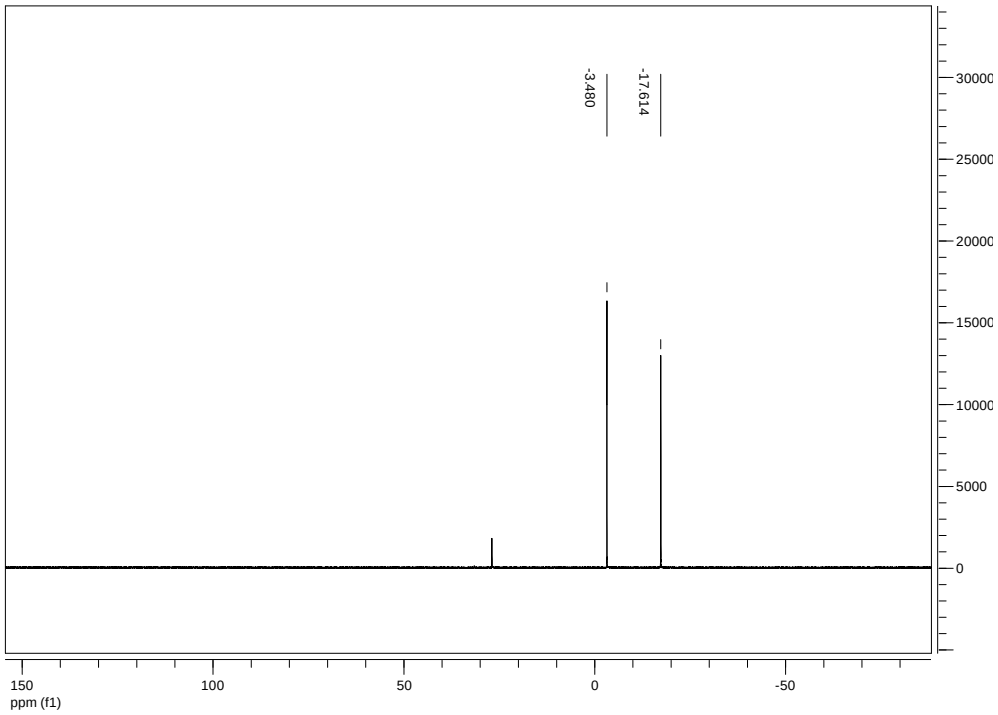

77.424
77.000
76.576
55.200
1000
500
0
150
100
50
ppm (f1)


164.064
160.959
160.458
152.139
152.085
151.972
149.227
149.108
140.192
139.881
137.342
136.550
136.126
135.841
135.537
134.163
134.018
132.140
131.831
127.716
127.630
126.431
126.073
125.847
124.803
123.504
123.438
121.858
121.821
121.136
120.561
120.443
118.317
115.789
114.667
114.550
114.271
114.161
114.040
113.866
600
500
400
300
200
100
0
-100
160
150
140
130
120
110
100
90
ppm (f1)

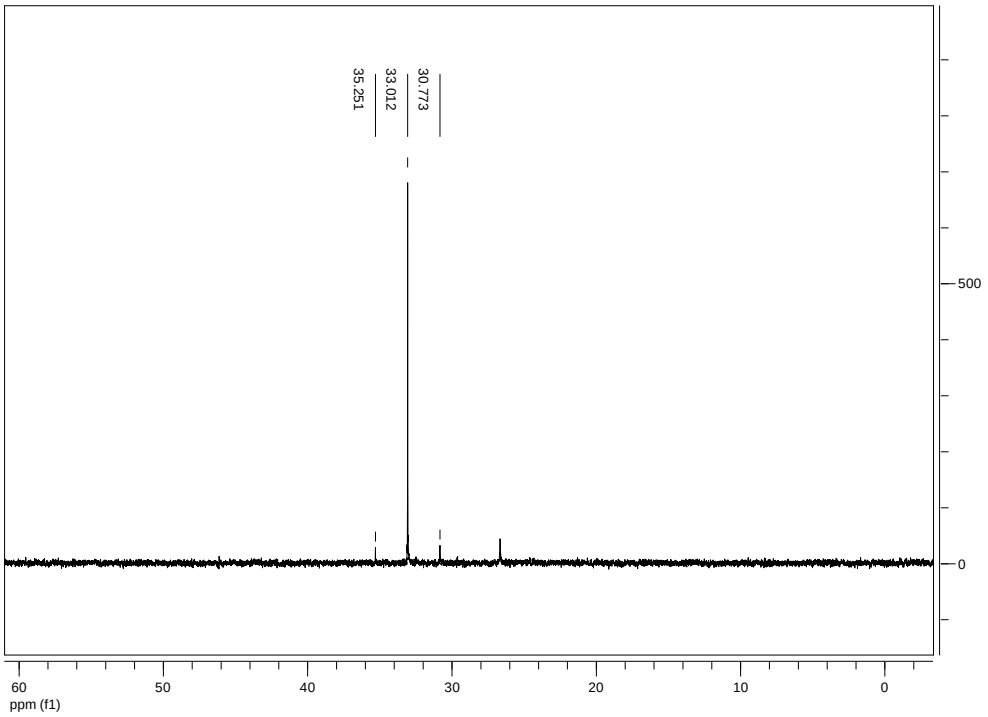
COSY H-H of 2b:



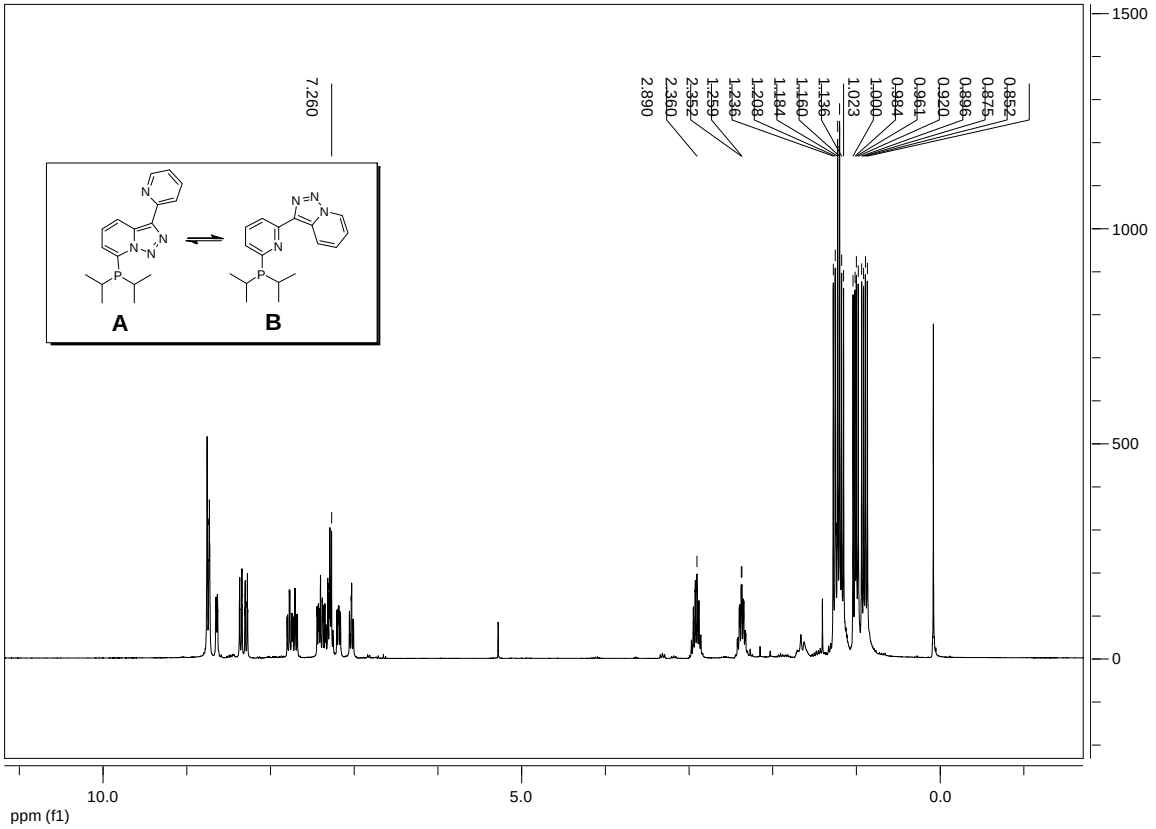
^{31}P NMR of 2b:



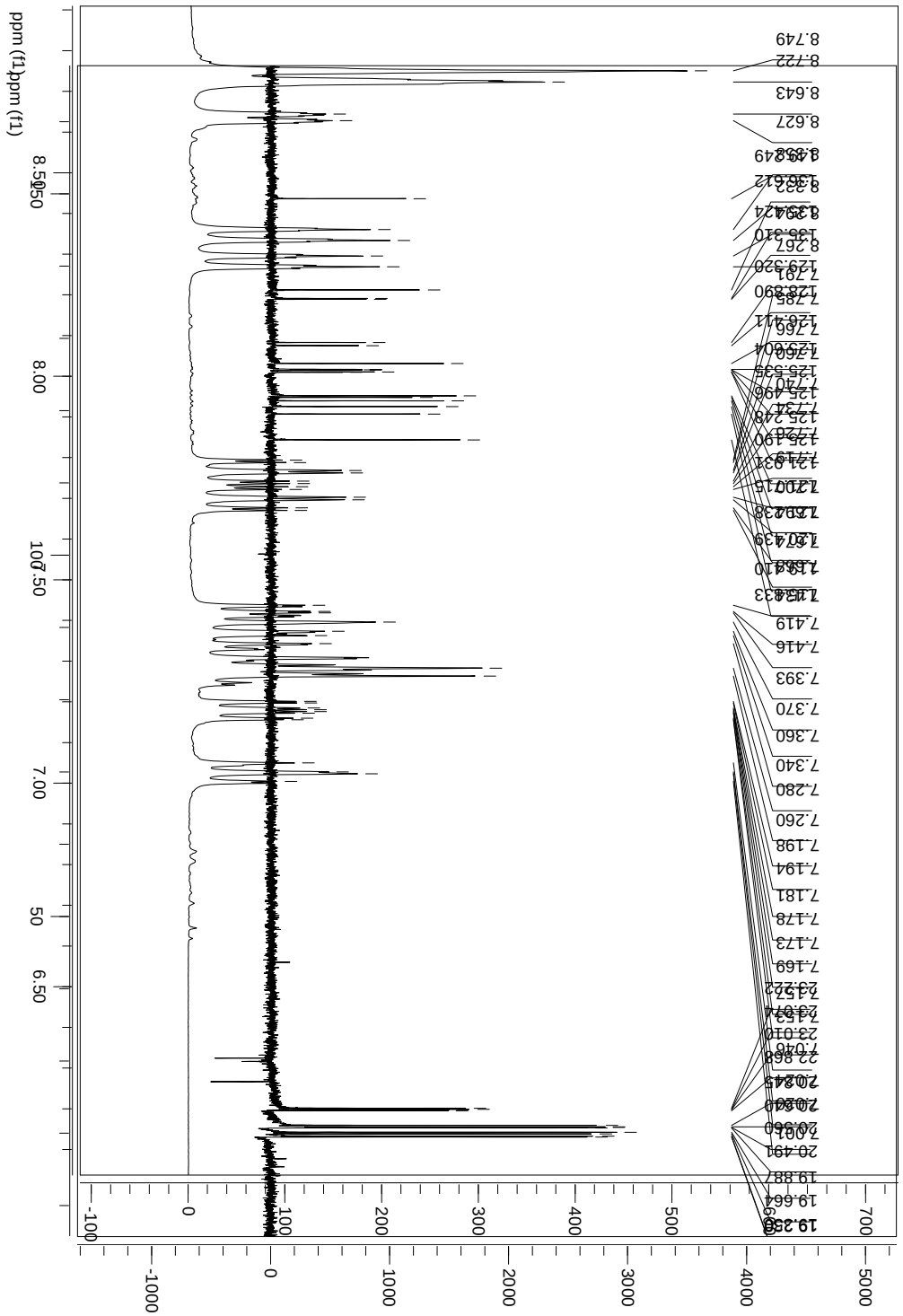
^{31}P NMR of 3b:



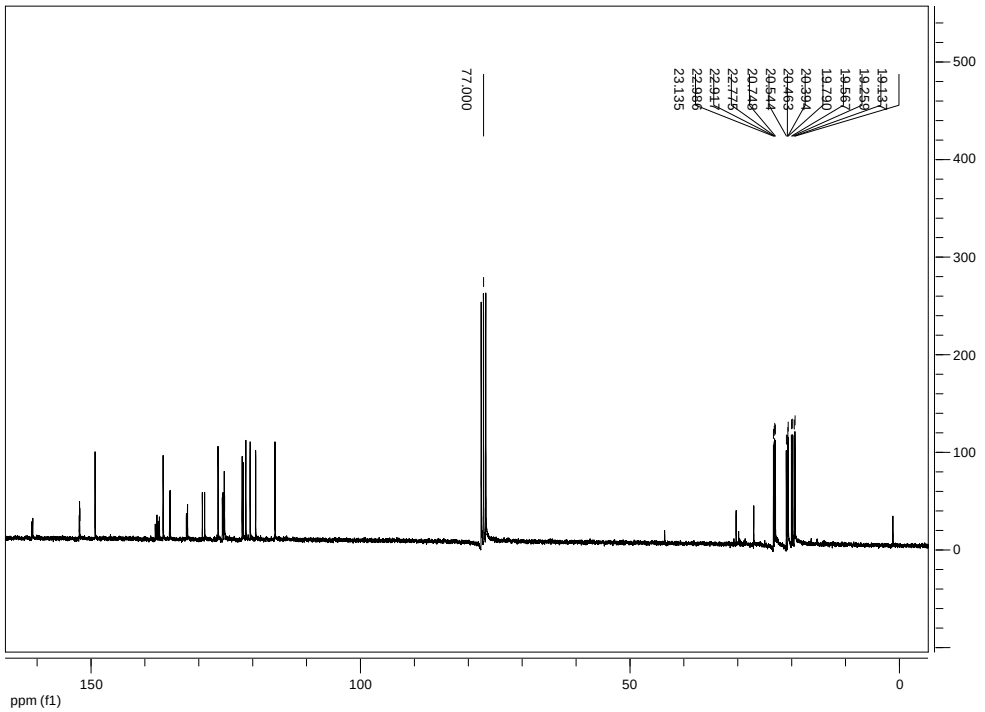
^{1}H NMR of 2c:

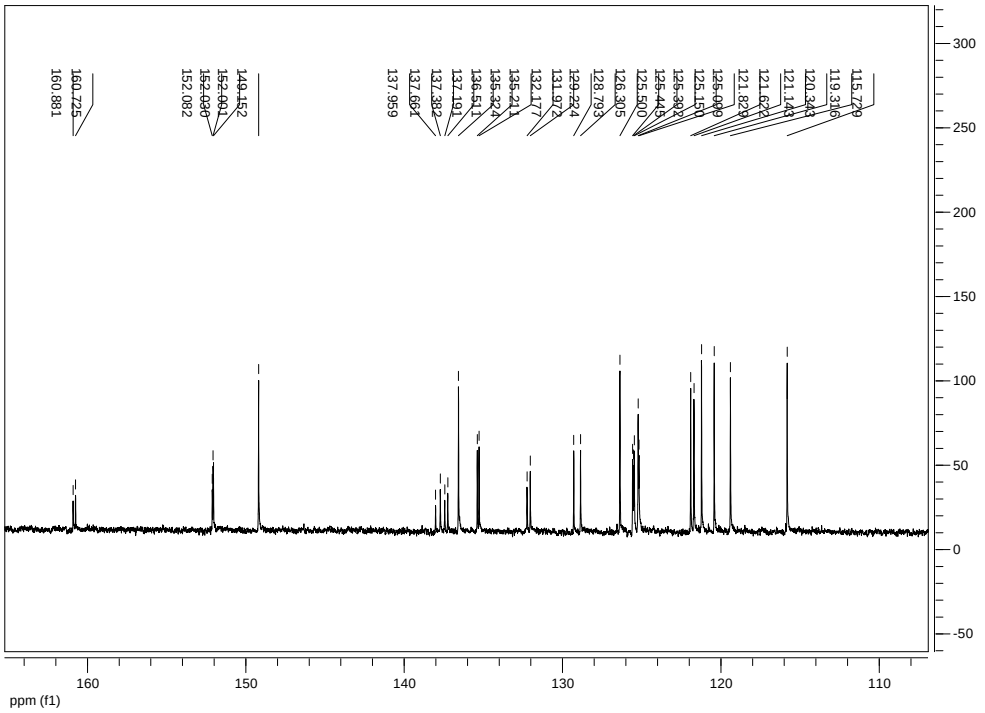

7.260
2.890
2.360
2.352
2.259
1.236
1.208
1.184
1.160
1.136
1.023
1.000
0.984
0.961
0.920
0.896
0.875
0.852
A
B
1500
1000
500
0
10.0
5.0
0.0
ppm (f1)

^{13}C NMR DEPT of 2c:

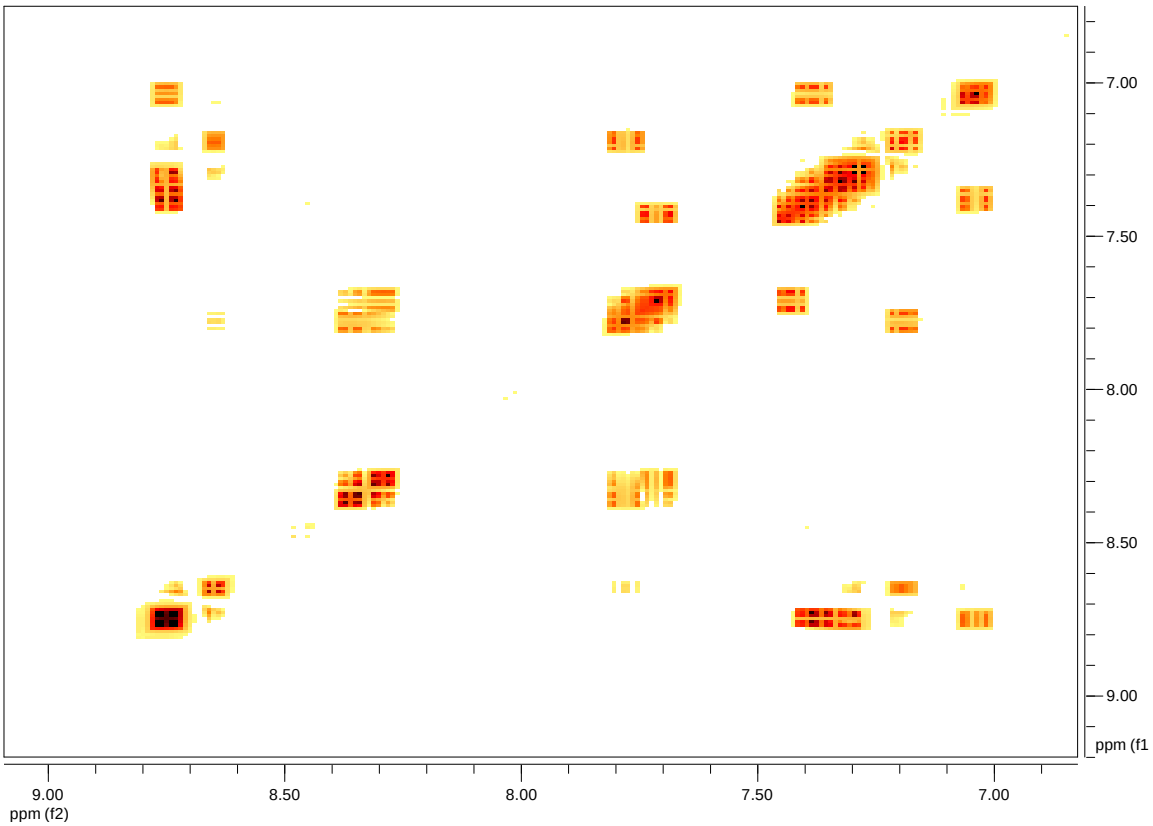


^{13}C NMR of 2c:

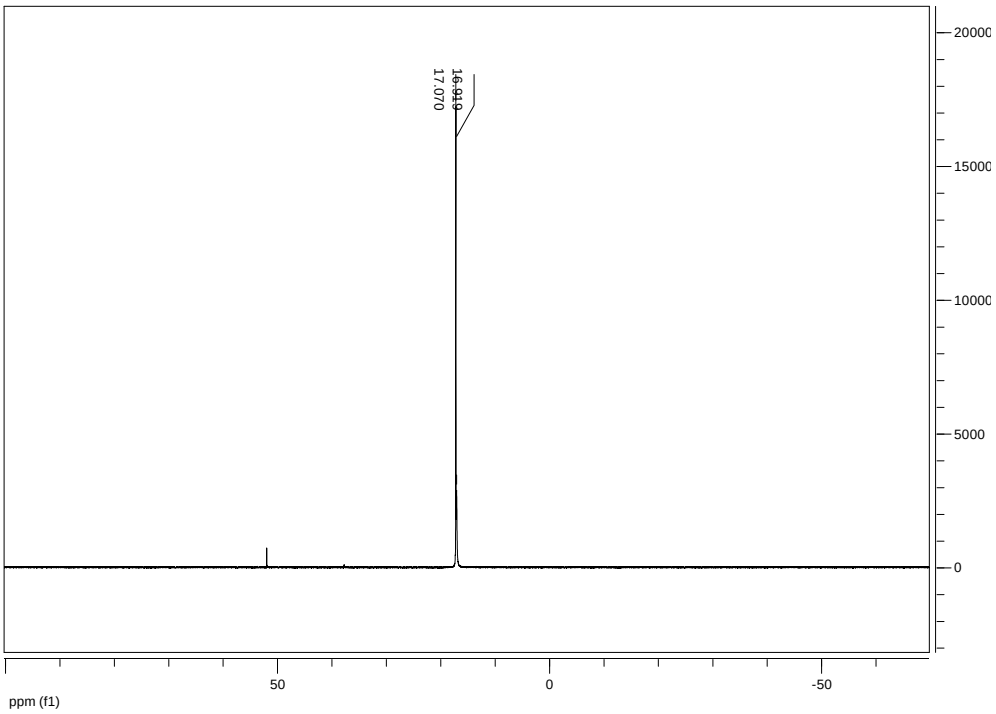

77.000
23.135
22.986
22.911
22.775
20.748
20.644
20.463
20.394
19.790
19.567
19.259
19.137
500
400
300
200
100
0
150
100
50
0
ppm (f1)


160.881
160.725
152.082
152.036
152.001
149.152
137.959
137.664
137.382
137.191
136.517
135.324
135.211
132.177
131.972
129.224
128.793
126.305
125.500
125.445
125.382
125.150
125.039
121.829
121.620
121.143
120.343
119.316
115.729
300
250
200
150
100
50
0
-50
160
150
140
130
120
110
ppm (f1)

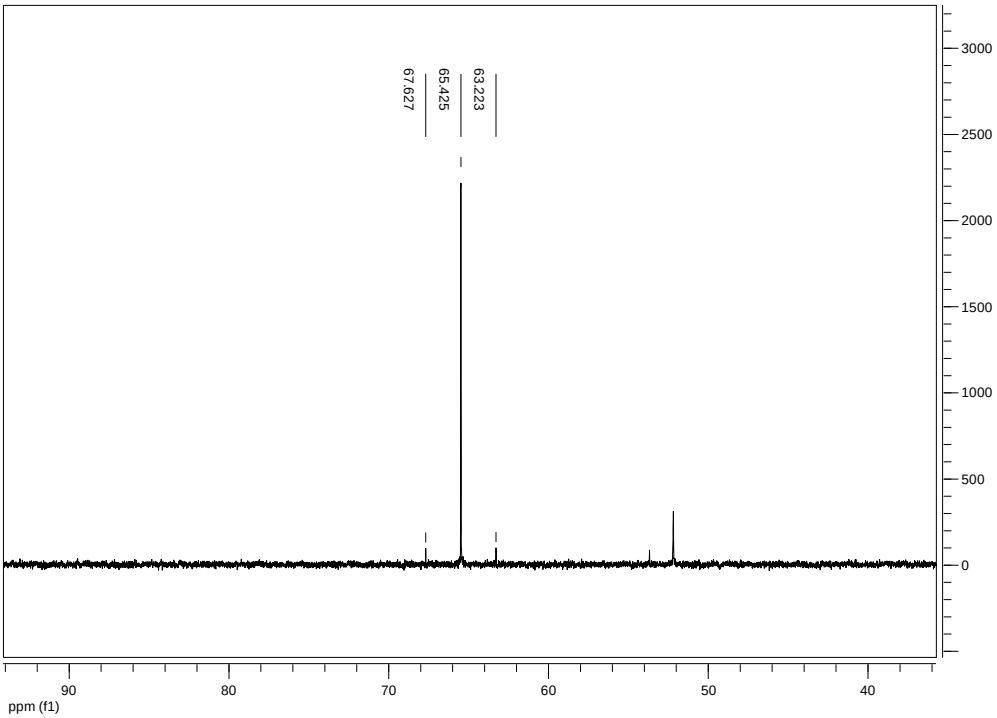
COSY H-H of 2c:



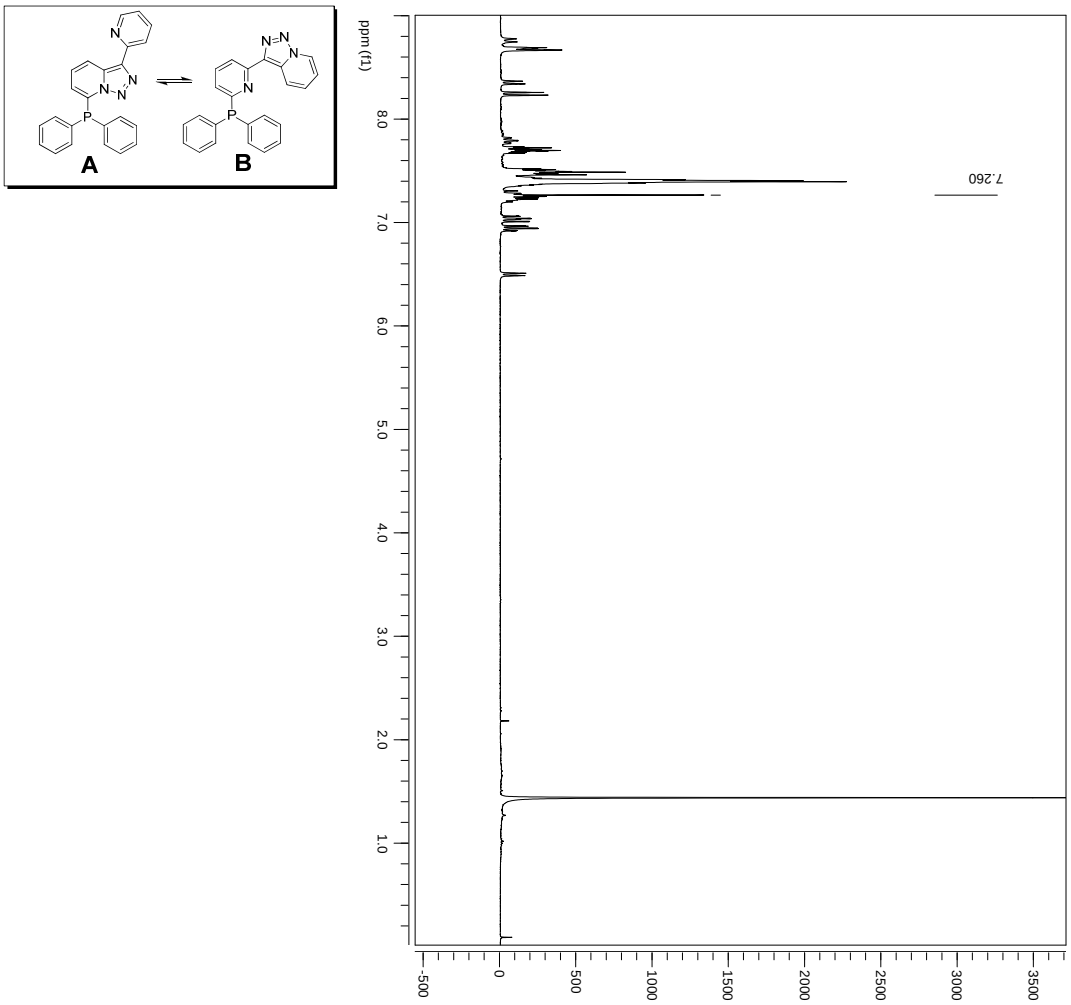
^{31}P NMR of 2c:

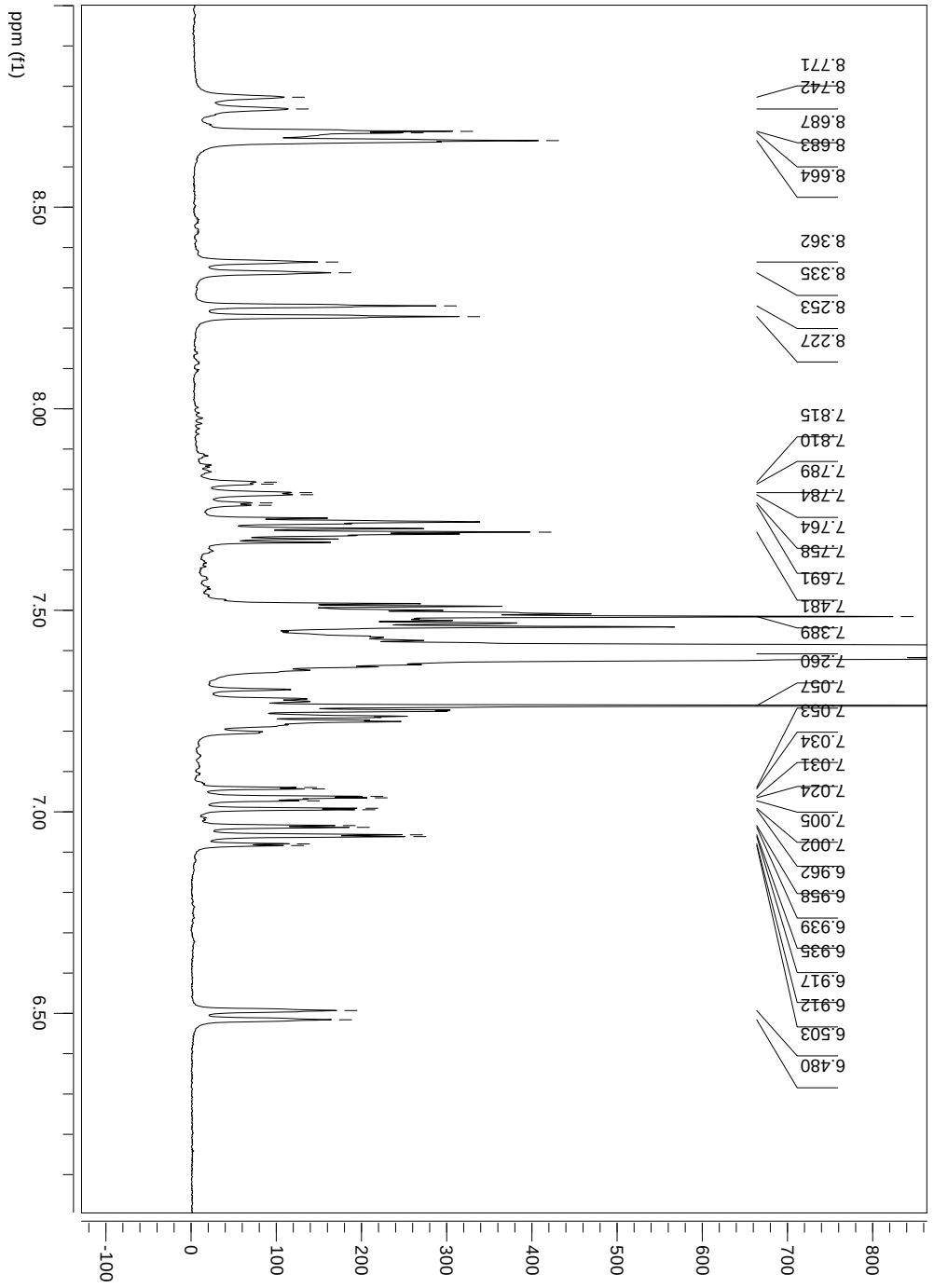


^{31}P NMR 3c:

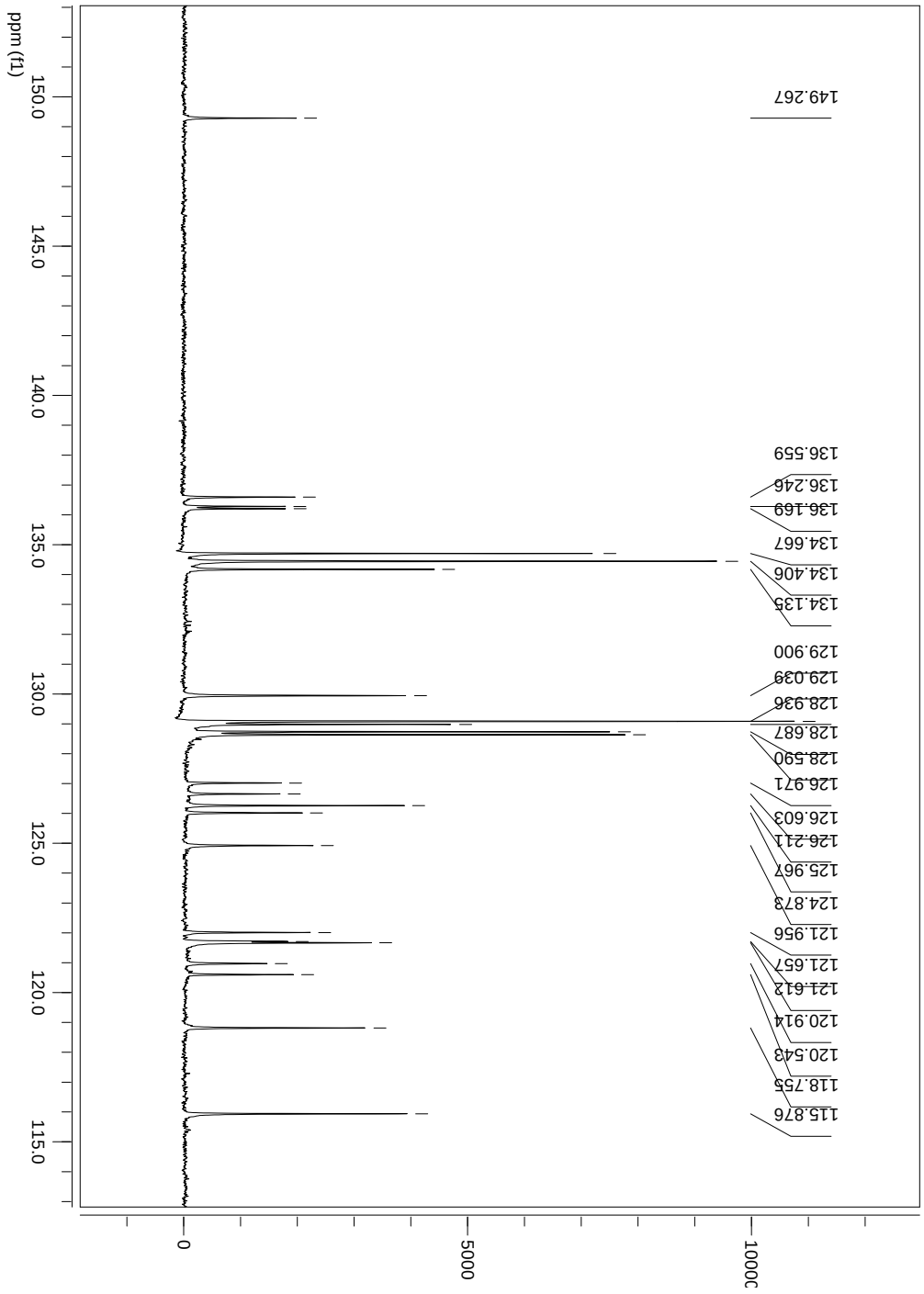


^{1}H NMR of 2d:

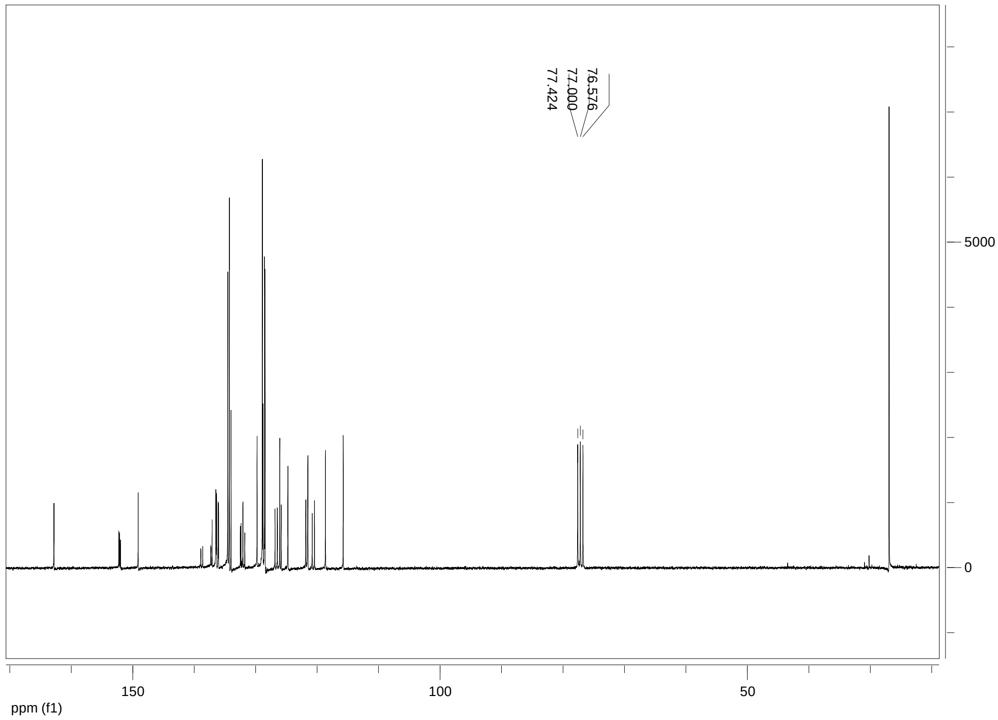

A
B
ppm (f1)
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
7.260
-500
0
500
1000
1500
2000
2500
3000
3500

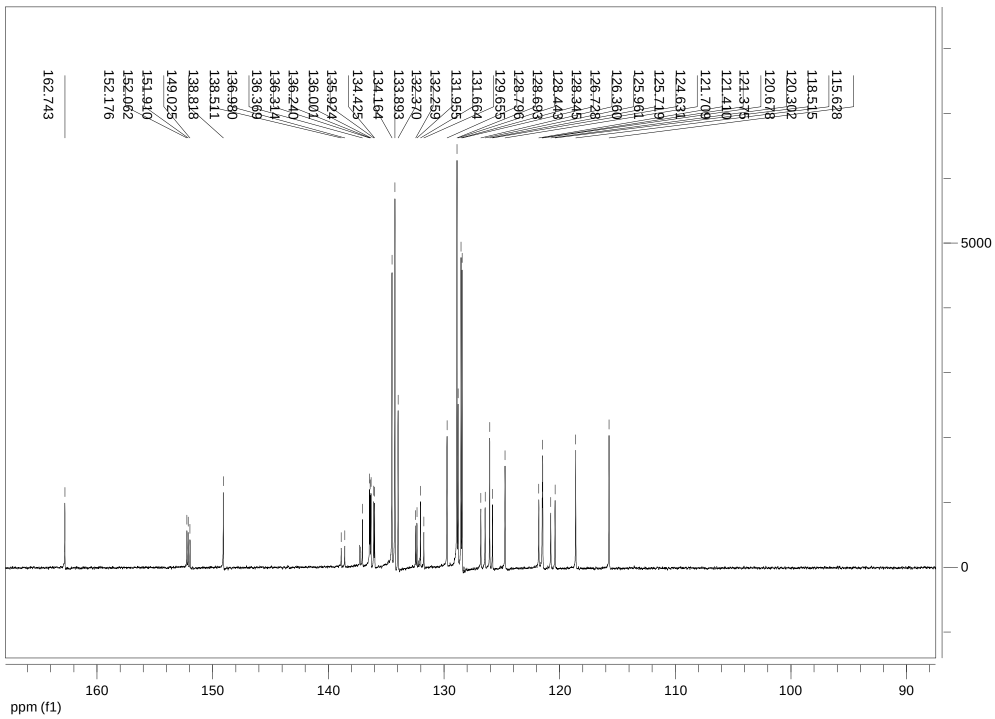


^{13}C NMR DEPT of 2c:

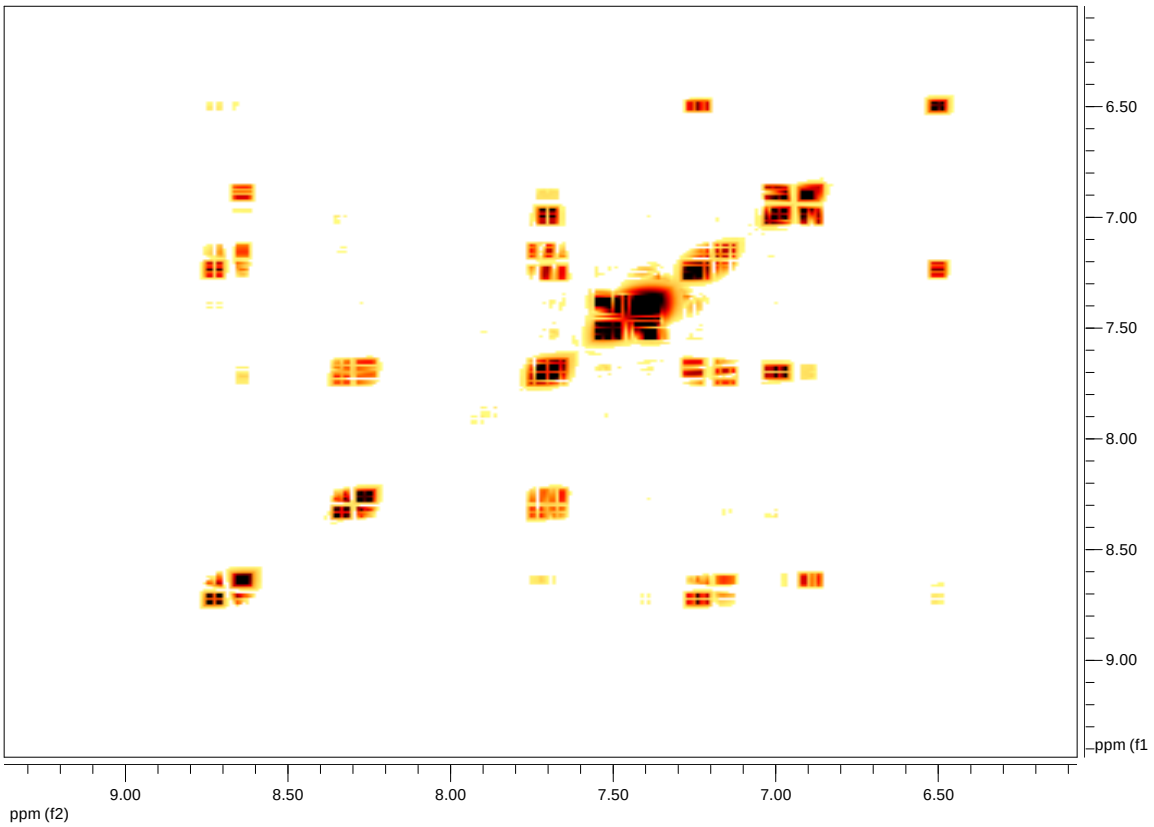


^{13}C NMR of 2c:

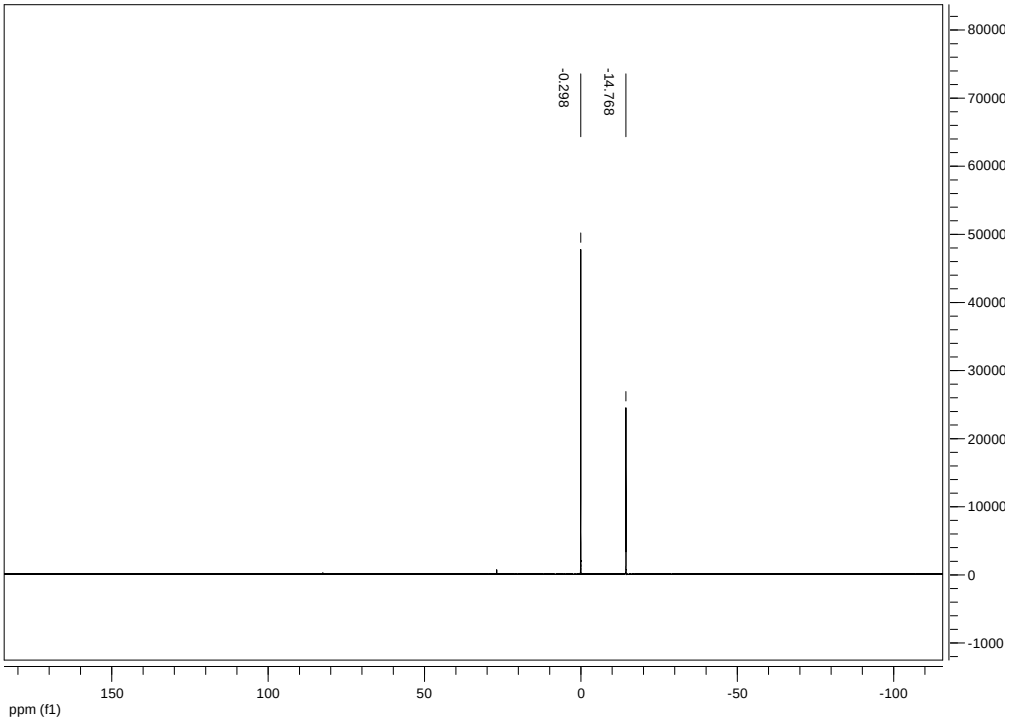

76.576
77.000
77.424
5000
0
150
100
50
ppm (f1)


162.743
152.176
152.062
151.910
149.025
138.816
138.511
136.980
136.369
136.314
136.240
136.001
135.924
134.425
134.164
133.893
132.370
132.259
131.955
131.664
129.655
128.796
128.693
128.443
128.345
126.728
126.360
125.961
125.719
124.631
121.709
121.410
121.375
120.678
120.302
118.515
115.628
5000
0
160
150
140
130
120
110
100
90
ppm (f1)

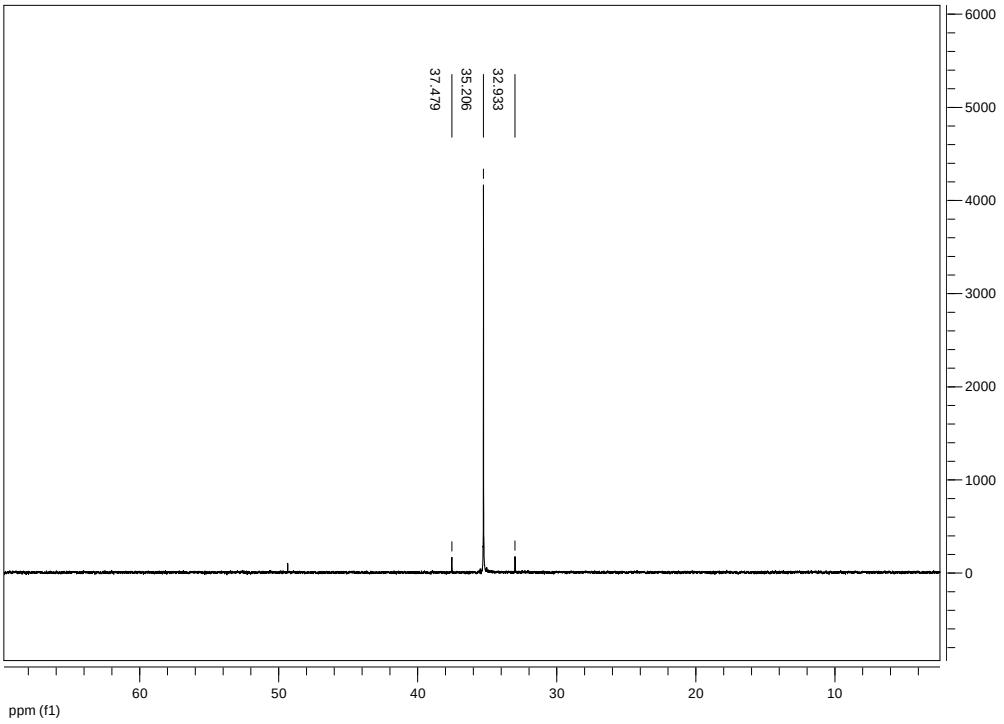
COSY H-H of 2c:



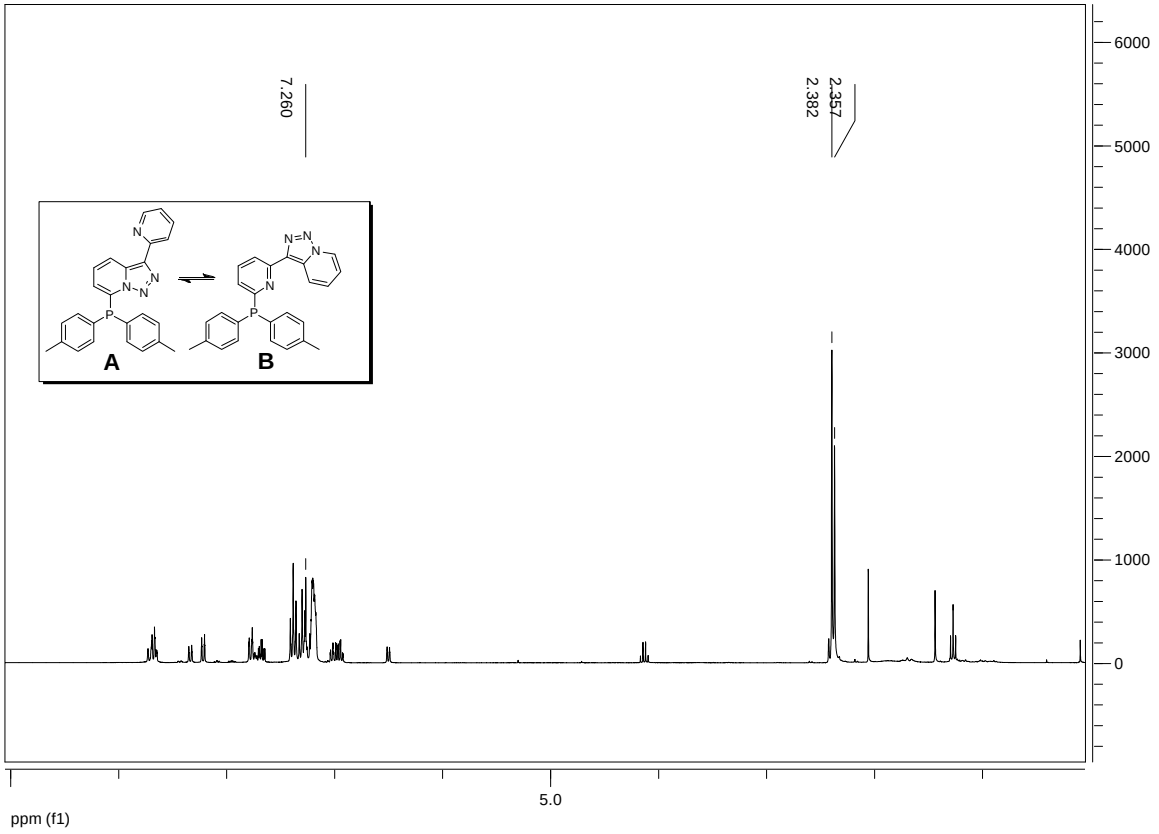
^{31}P NMR of 2c:

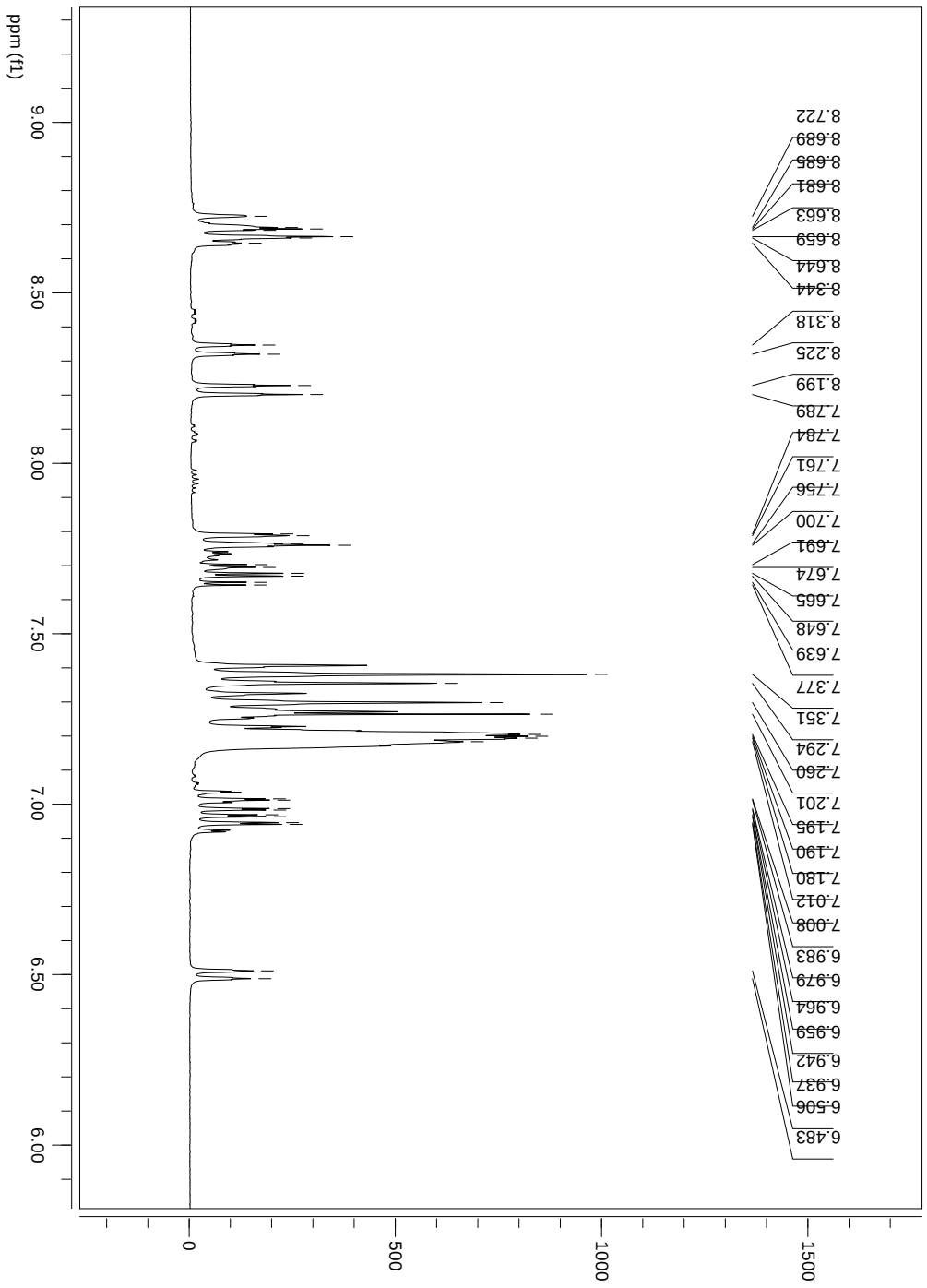


^{31}P NMR of 3c:

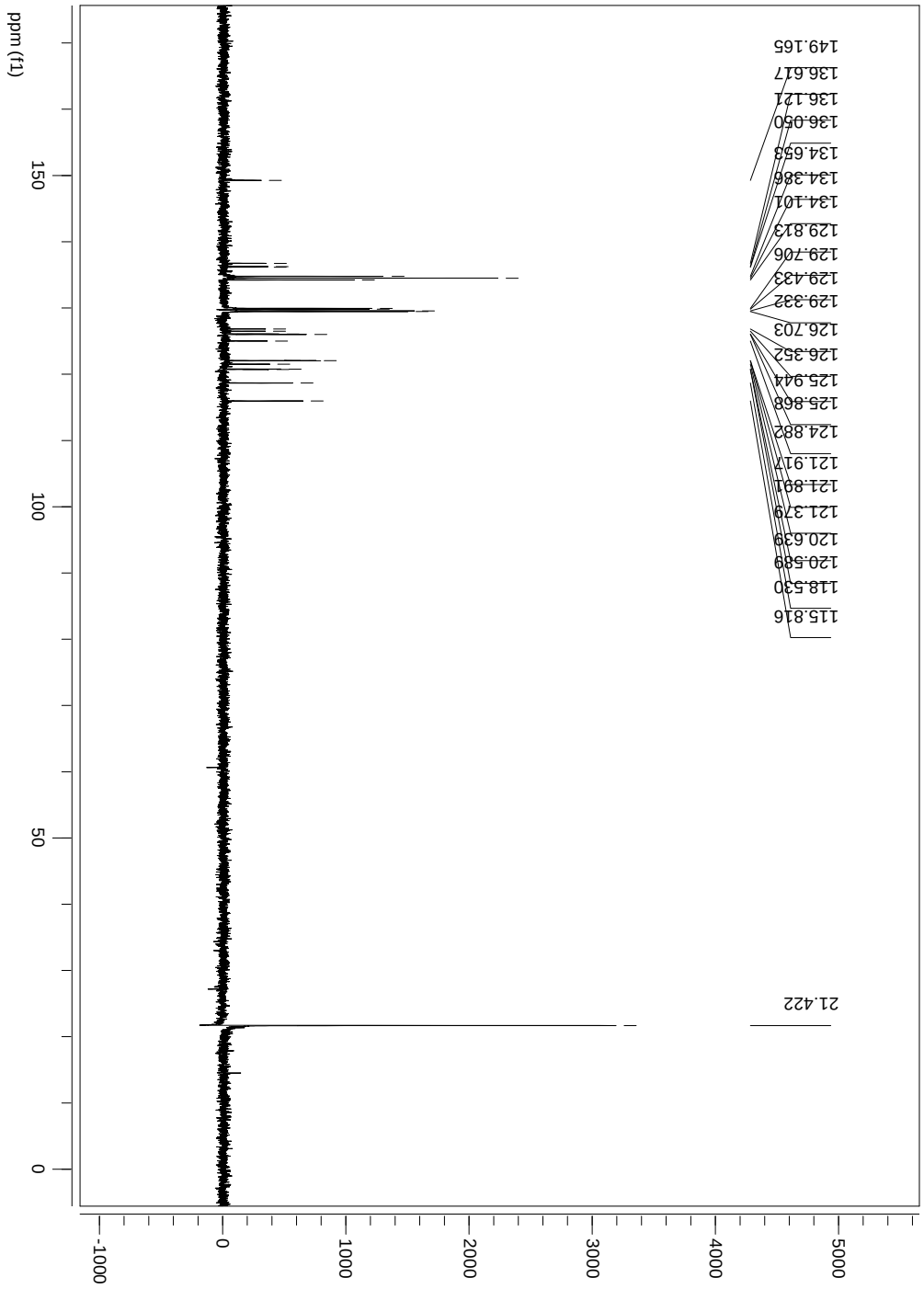


^{1}H NMR of 2e:

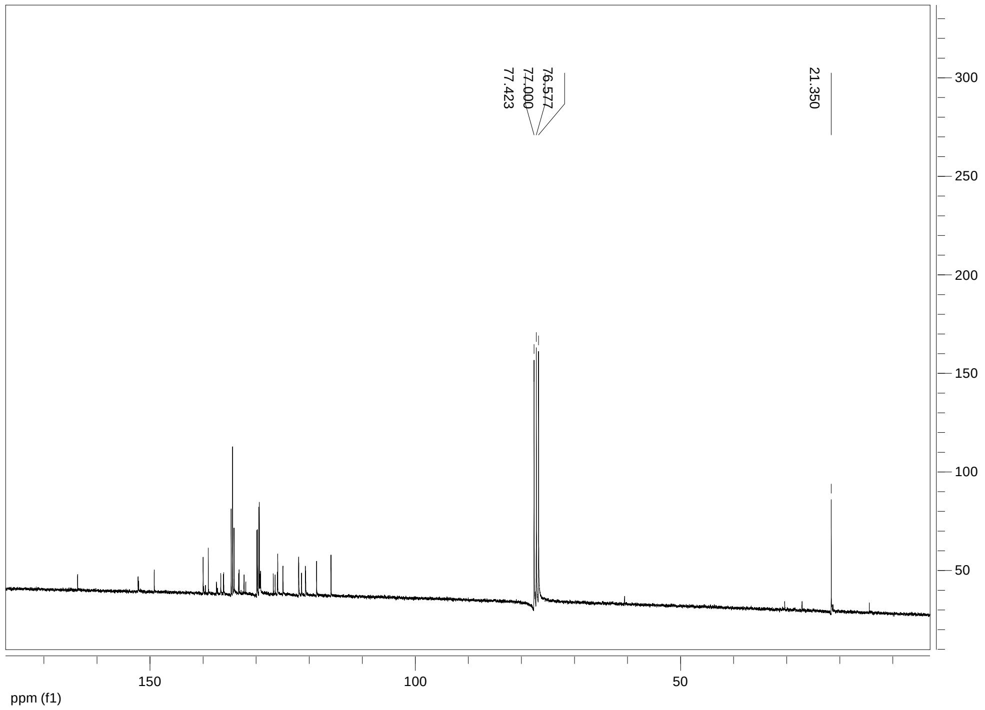


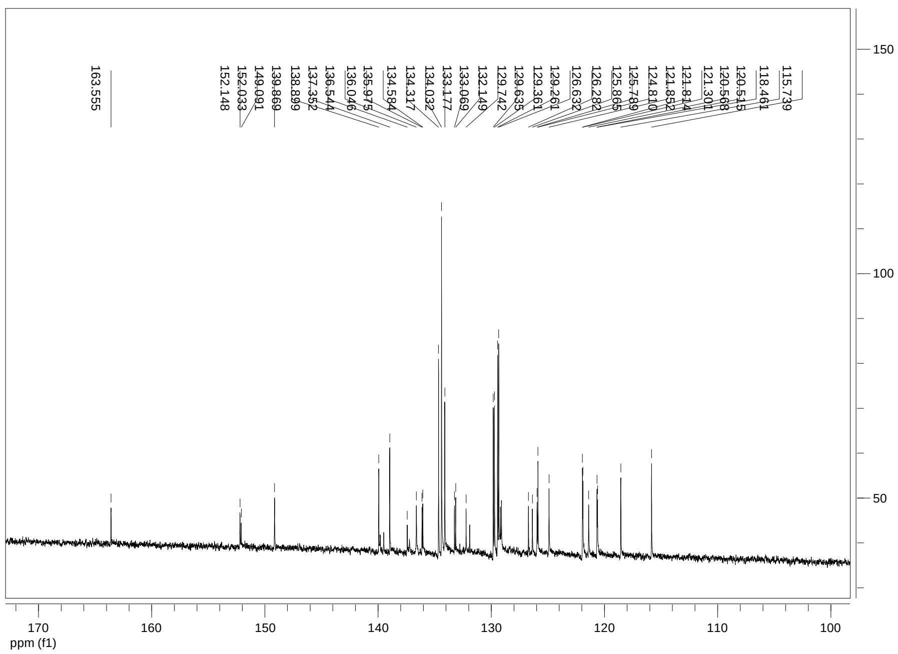


^{13}C NMR DEPT of 2e:

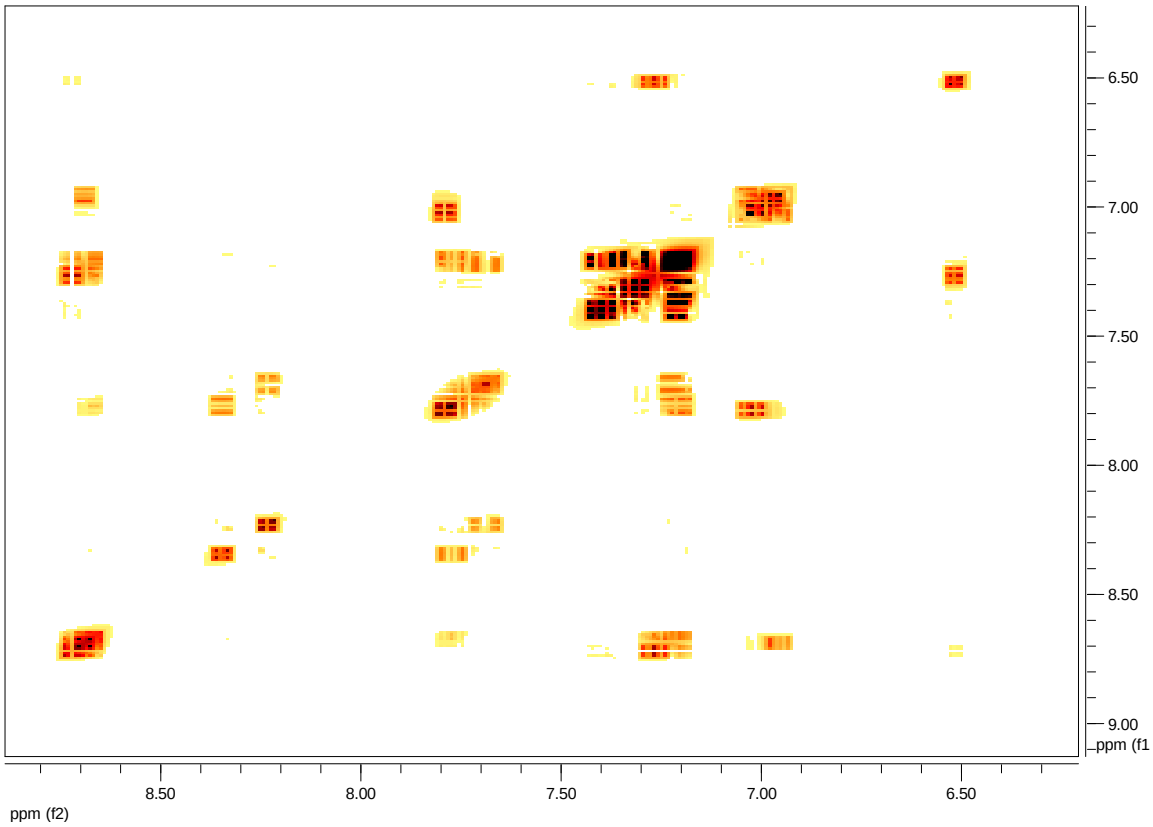


^{13}C NMR of 2e:

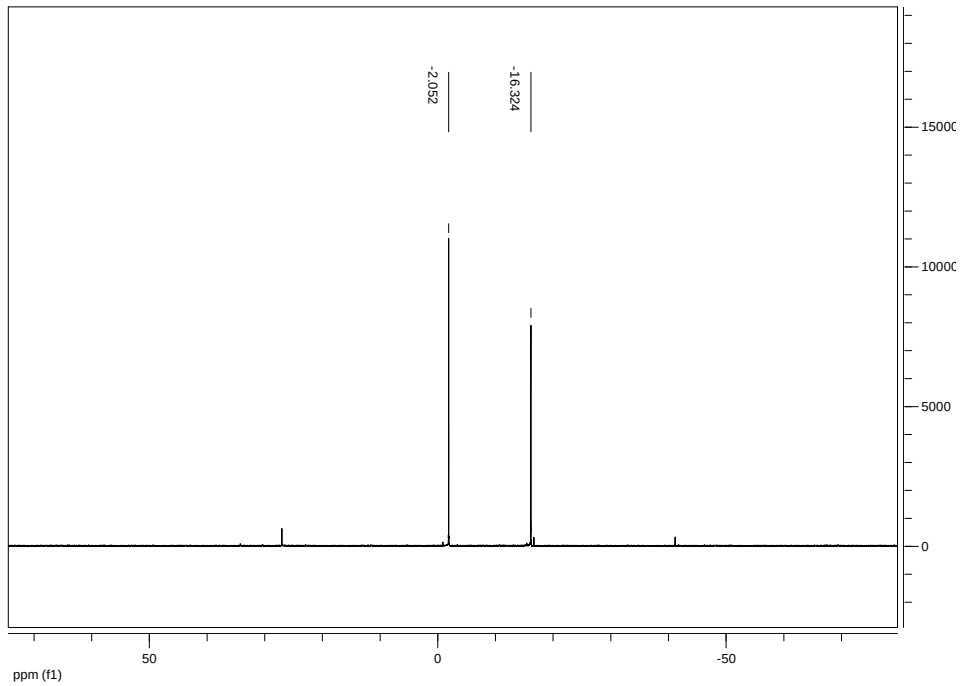

76.577
77.000
77.423
21.350
300
250
200
150
100
50
150
100
50
ppm (f1)


163.555
152.148
152.033
149.091
139.869
138.899
137.352
136.544
136.046
135.975
134.584
134.317
134.032
133.177
133.069
132.149
129.742
129.635
129.361
129.261
126.632
126.282
125.865
125.789
124.810
121.852
121.814
121.301
120.568
120.515
118.461
115.739
150
100
50
170
160
150
140
130
120
110
100
ppm (f1)

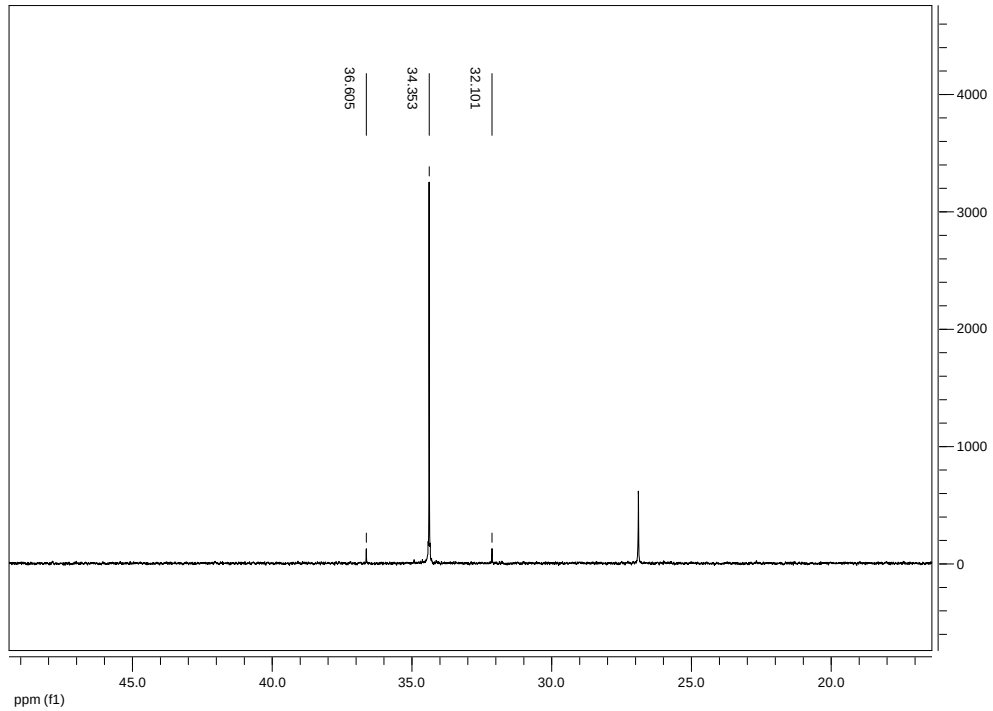
COSY H-H of 2e:



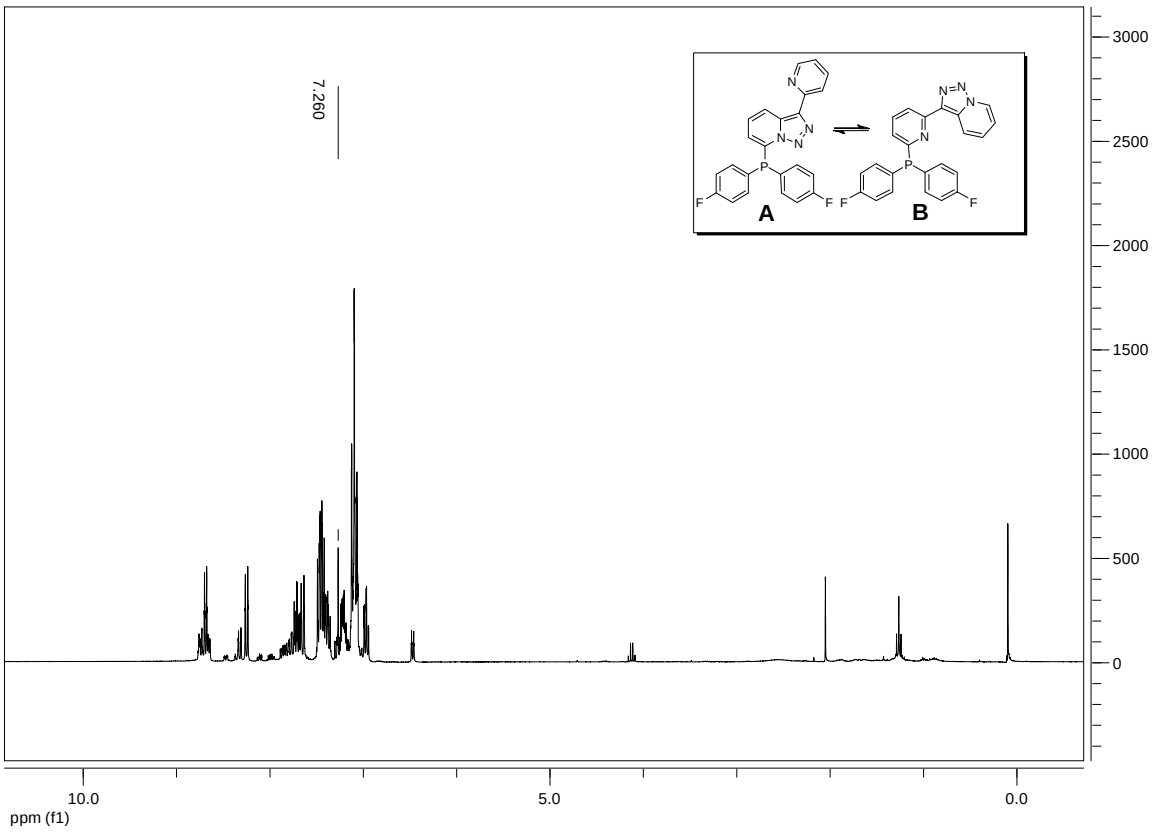
^{31}P NMR of 2e:

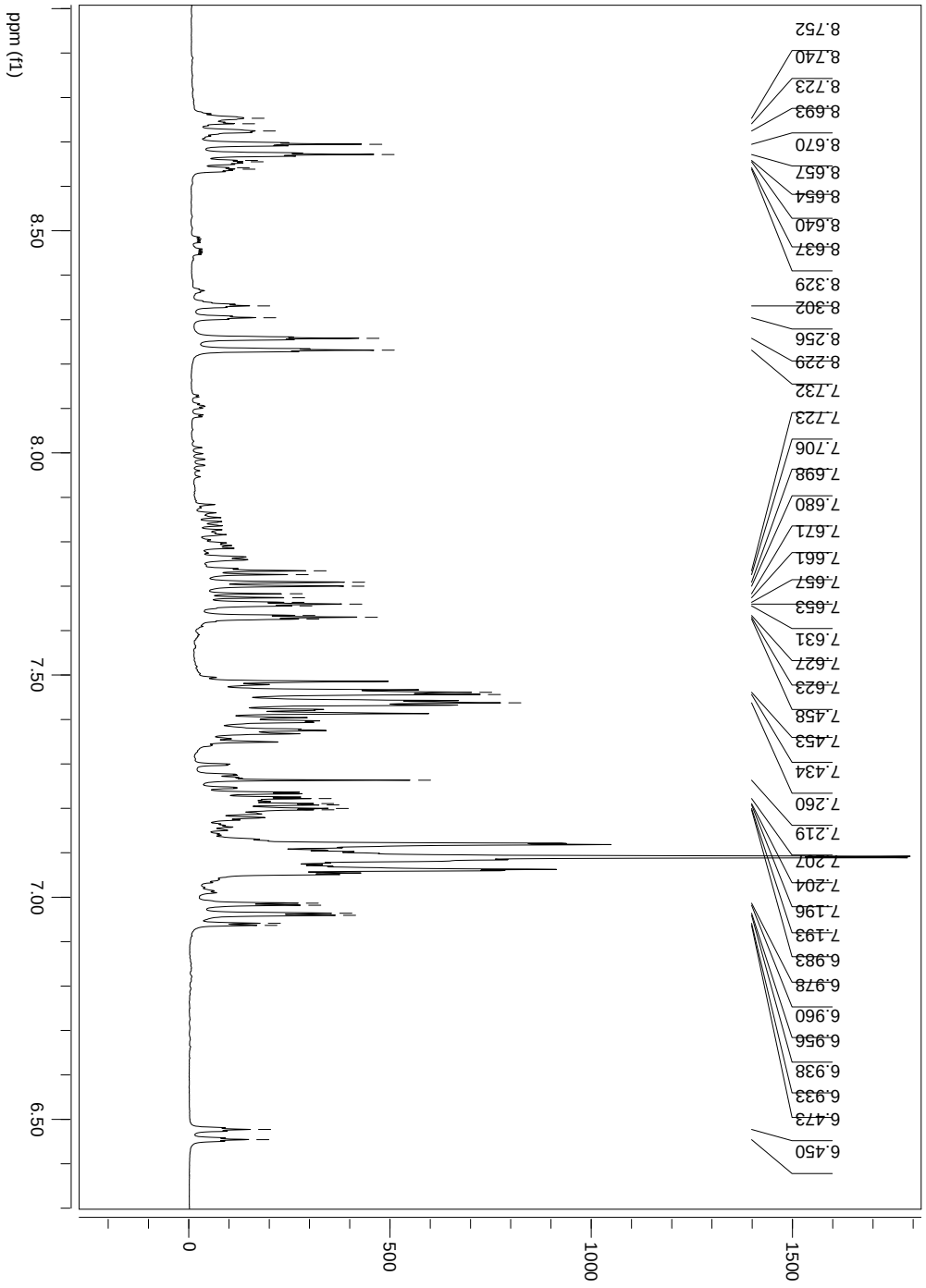


^{31}P NMR of 3e:

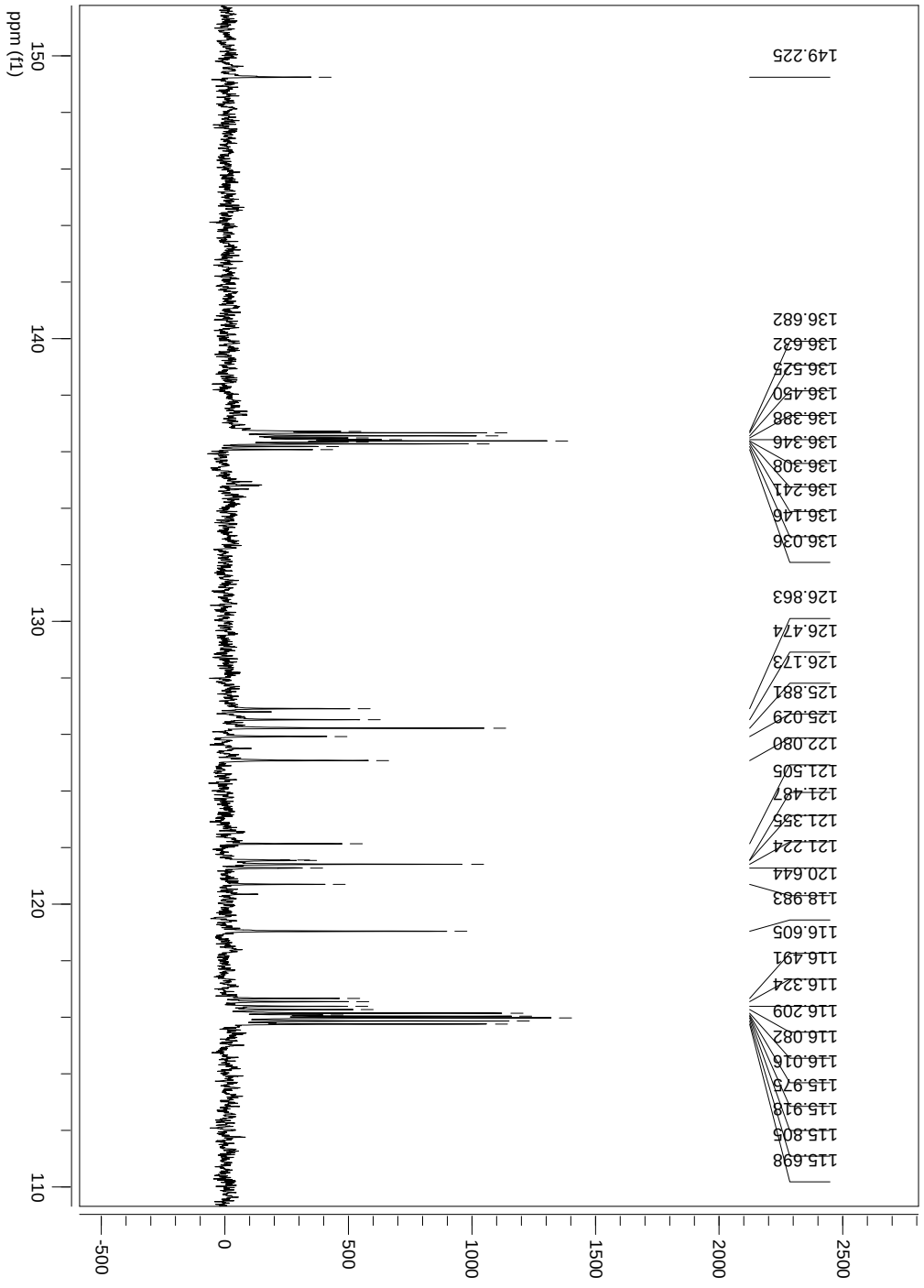


^{1}H NMR of 2f:

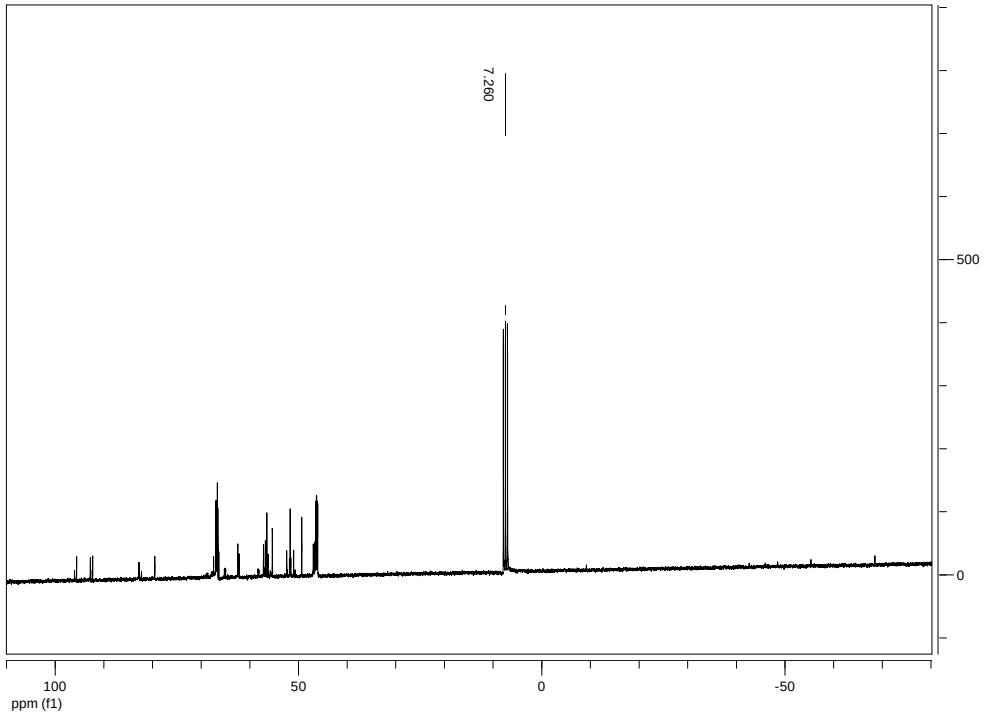


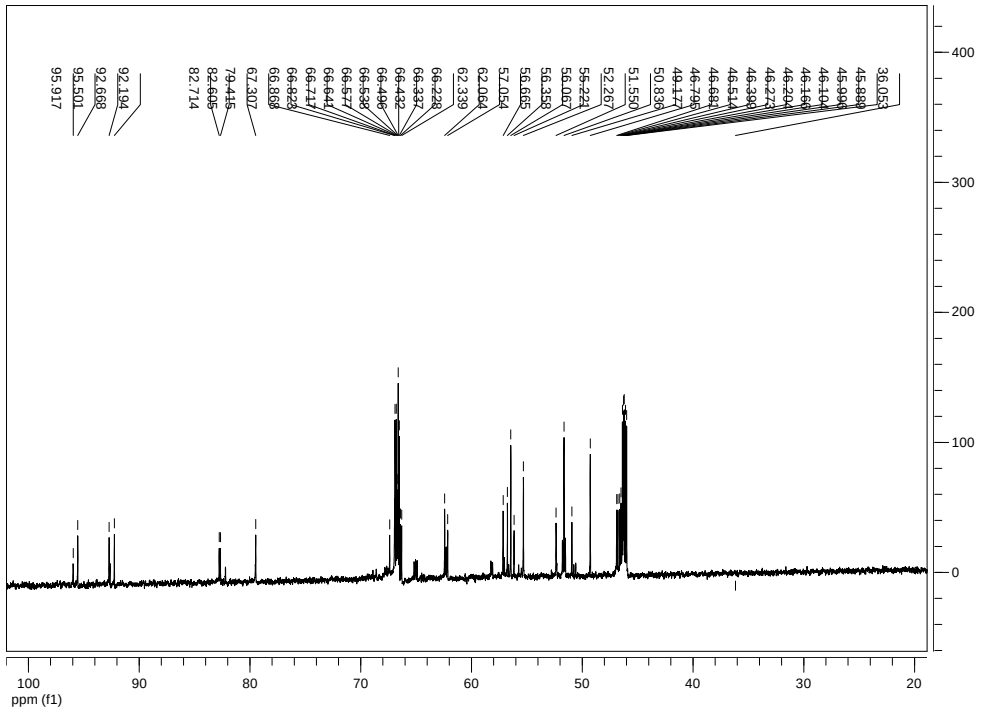

ppm (f1)
8.50
8.00
7.50
7.00
6.50
0
500
1000
1500
8.752
8.740
8.723
8.693
8.670
8.657
8.654
8.640
8.637
8.329
8.302
8.256
8.229
7.732
7.723
7.706
7.698
7.680
7.671
7.661
7.657
7.653
7.631
7.627
7.623
7.458
7.453
7.434
7.260
7.219
7.207
7.204
7.196
7.193
6.983
6.978
6.960
6.956
6.938
6.933
6.473
6.450

^{13}C NMR DEPT of 2f:

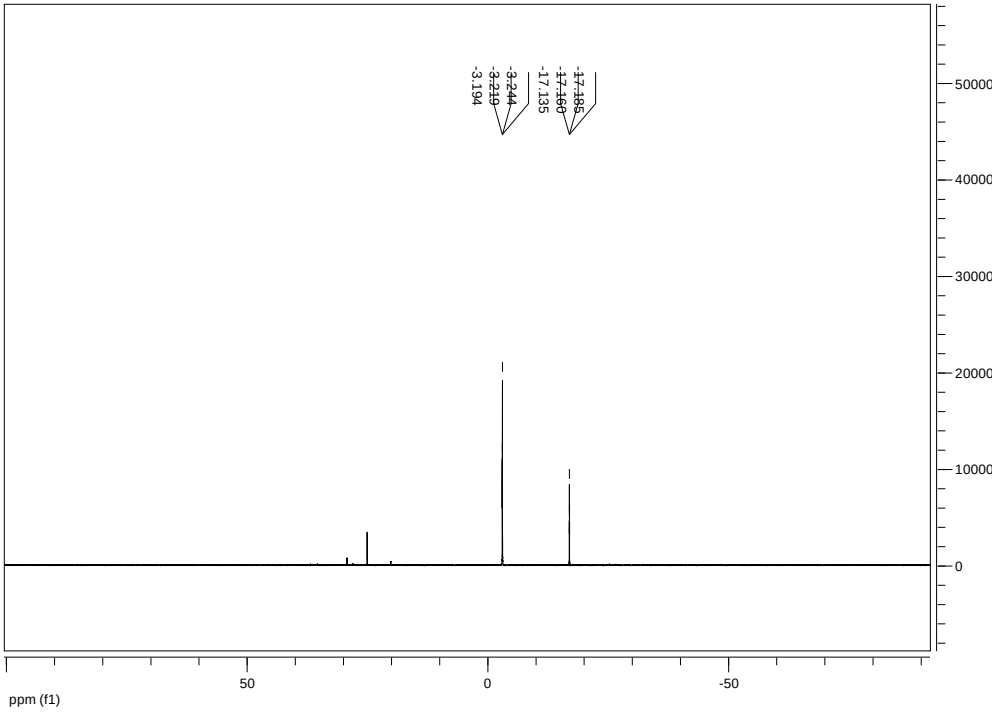


^{13}C NMR of 2f:

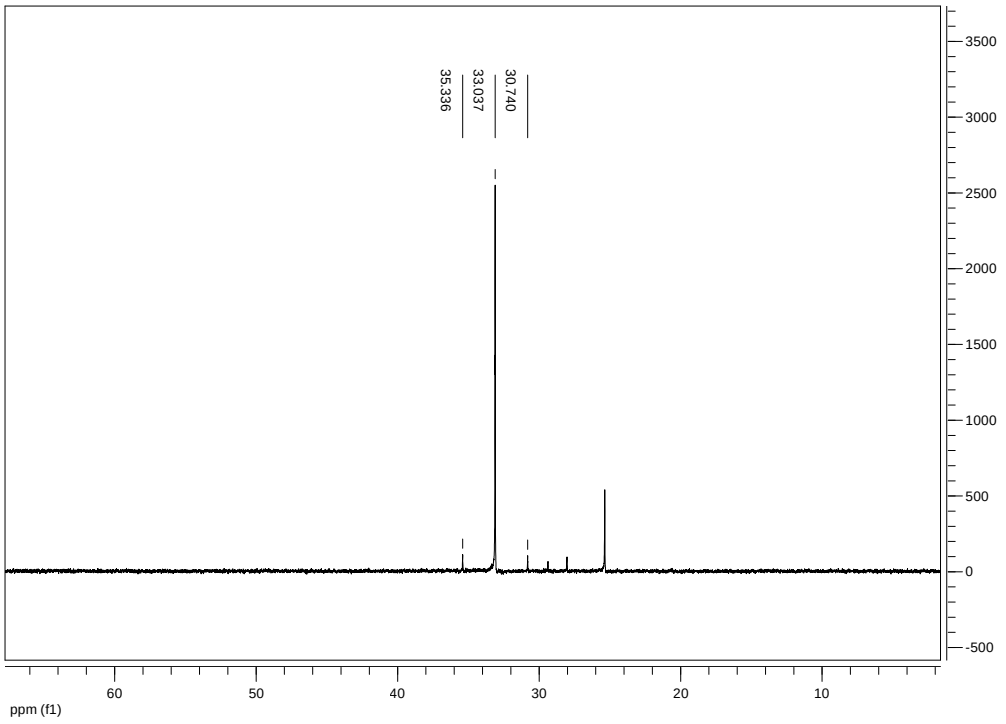

7.260
500
0
100
50
0
-50
ppm (f1)


95.917
95.501
92.668
92.194
82.714
82.605
79.415
67.307
66.868
66.822
66.717
66.641
66.577
66.538
66.496
66.432
66.332
66.228
62.339
62.054
57.054
56.665
56.358
56.067
55.221
52.267
51.550
50.838
49.177
46.796
46.681
46.514
46.390
46.272
46.204
46.160
46.104
45.998
45.880
36.053
400
300
200
100
0
100
90
80
70
60
50
40
30
20
ppm (f1)

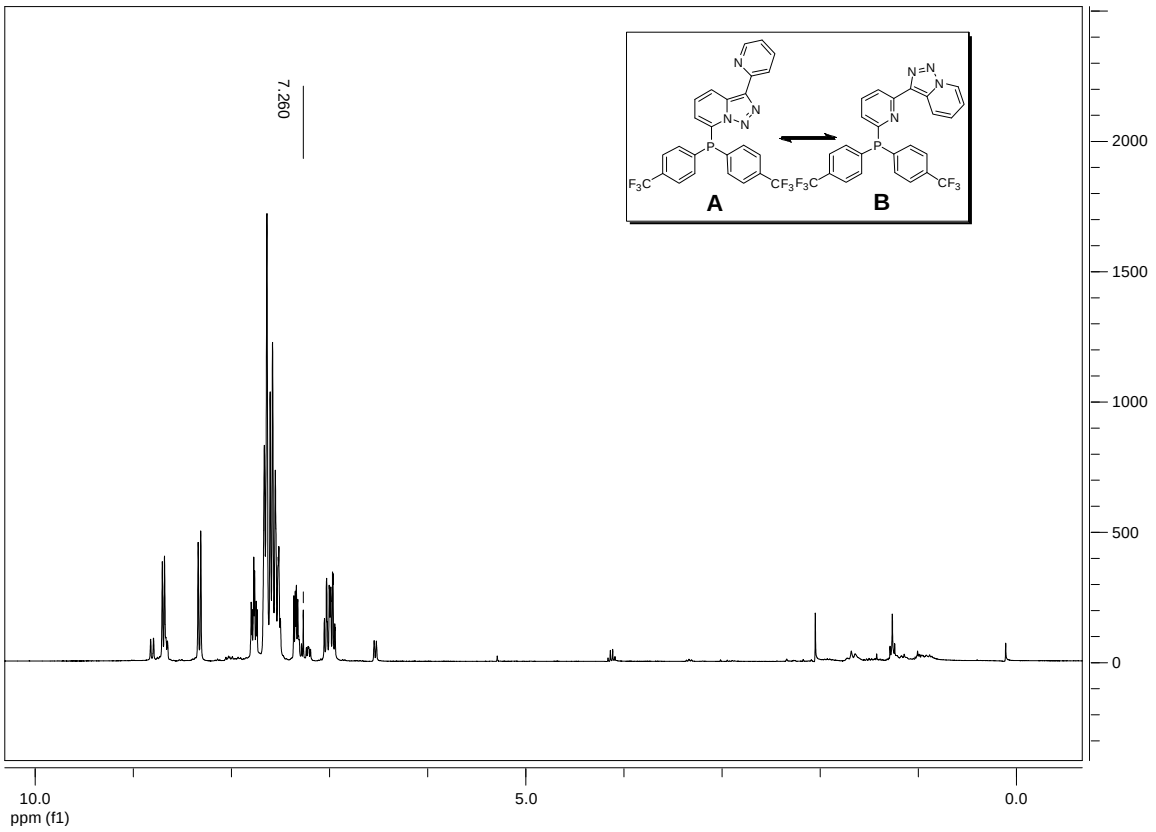
^{31}P NMR of 2f:

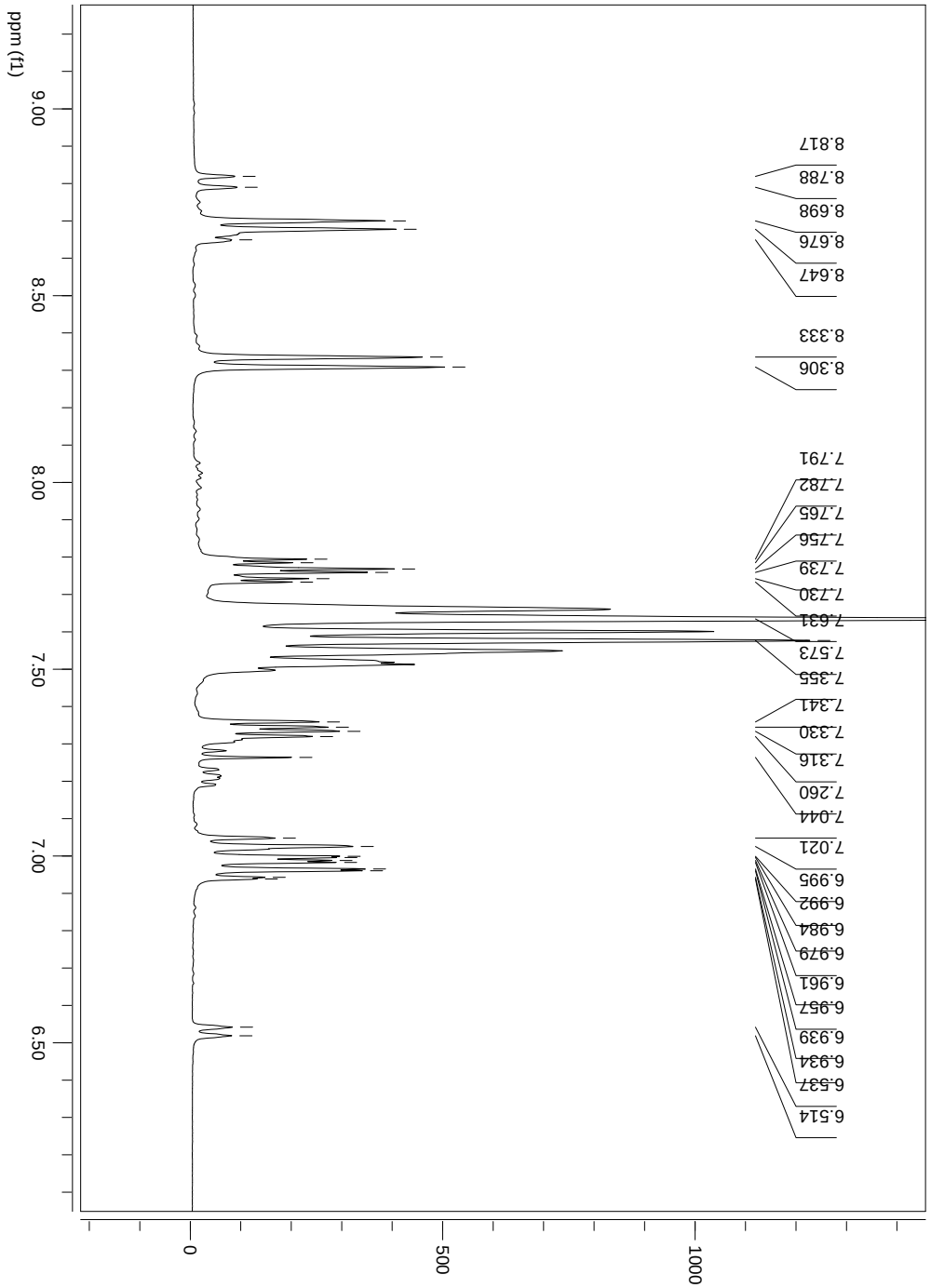


^{31}P NMR of 3f:

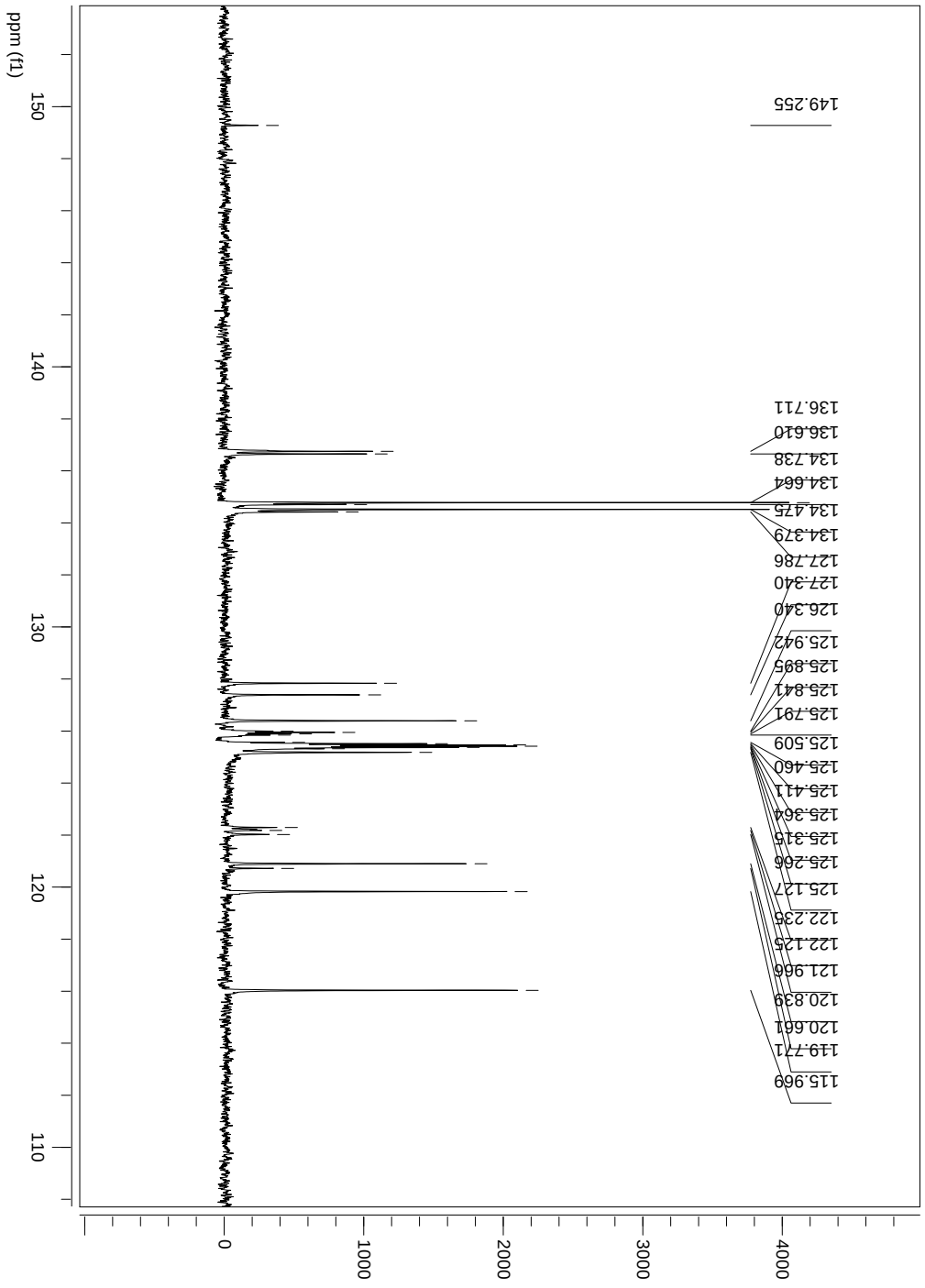


^{1}H NMR of 2g:

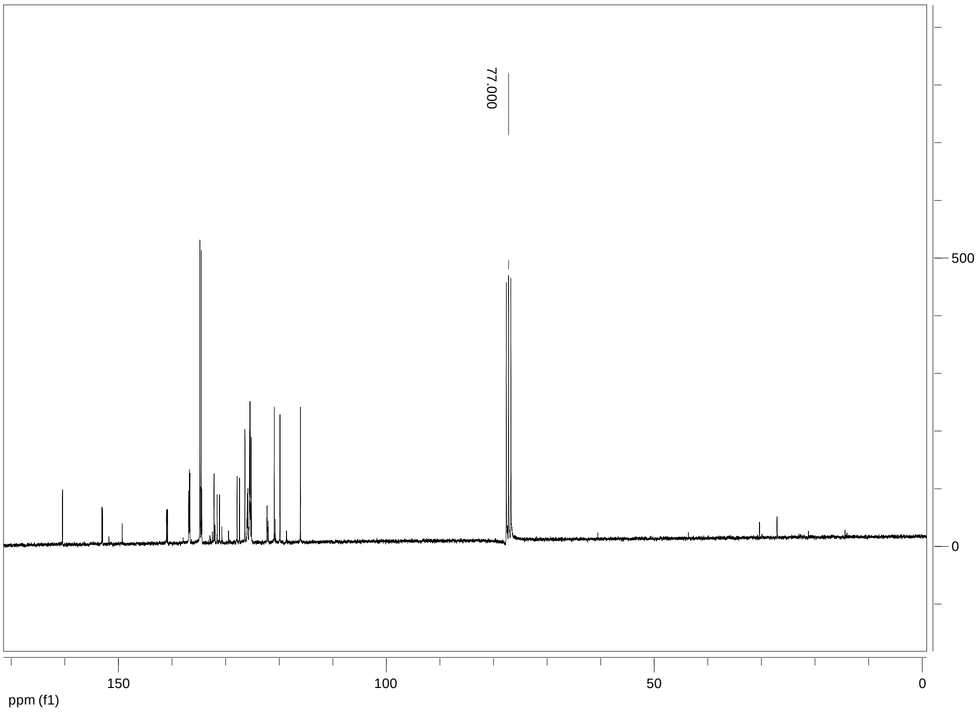


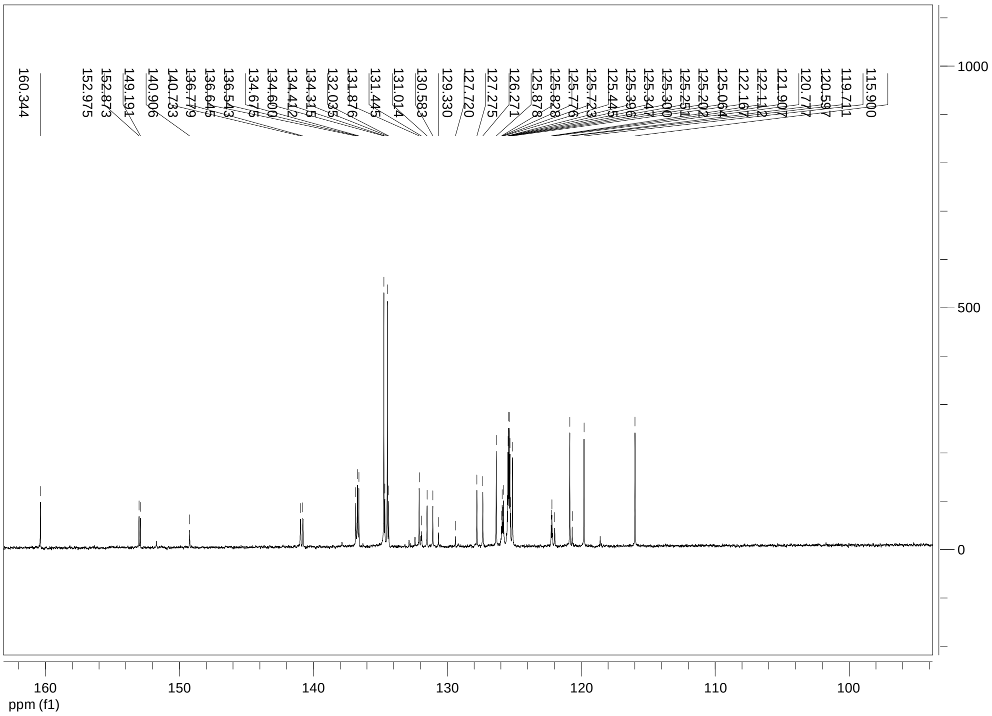

ppm (f1)
9.00
8.50
8.00
7.50
7.00
6.50
0
500
1000
8.817
8.788
8.698
8.676
8.647
8.333
8.306
7.791
7.782
7.765
7.756
7.739
7.730
7.631
7.573
7.355
7.341
7.330
7.316
7.260
7.044
7.021
6.995
6.992
6.984
6.979
6.961
6.957
6.939
6.934
6.537
6.514

^{13}C NMR DEPT of 2g:

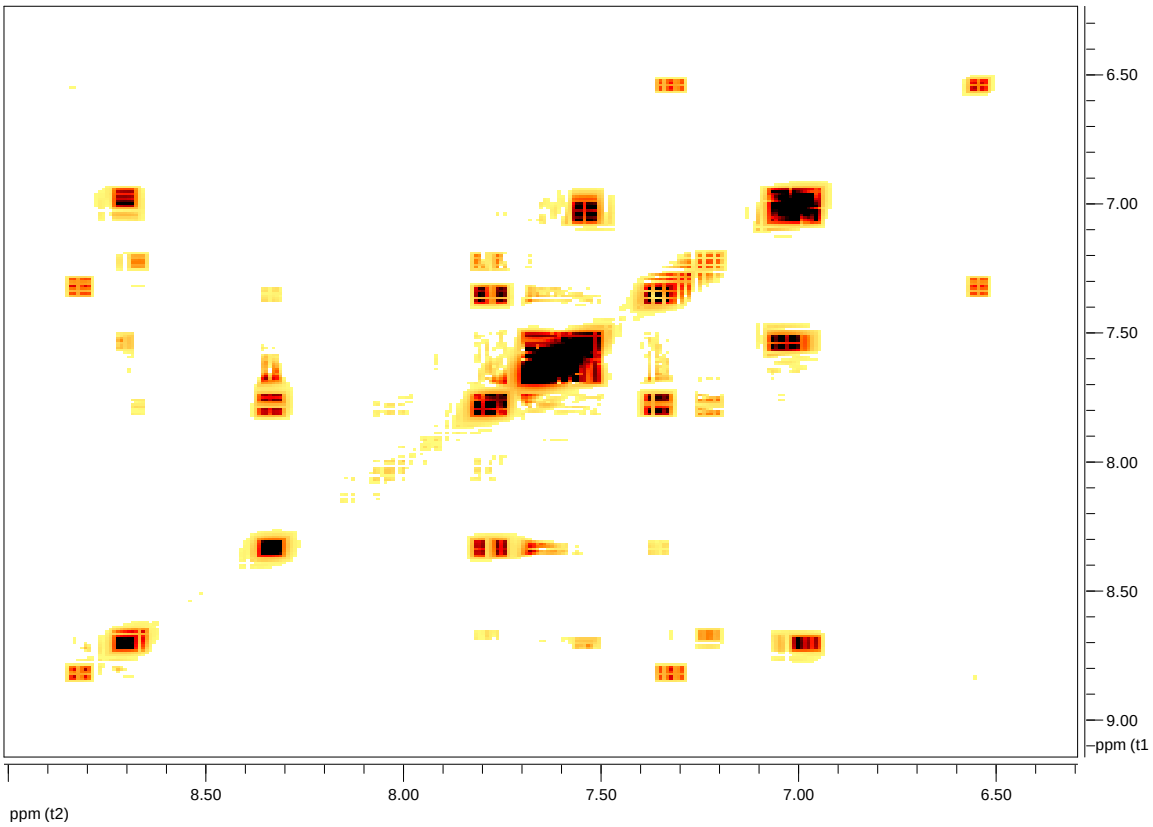


^{13}C NMR of 2g:

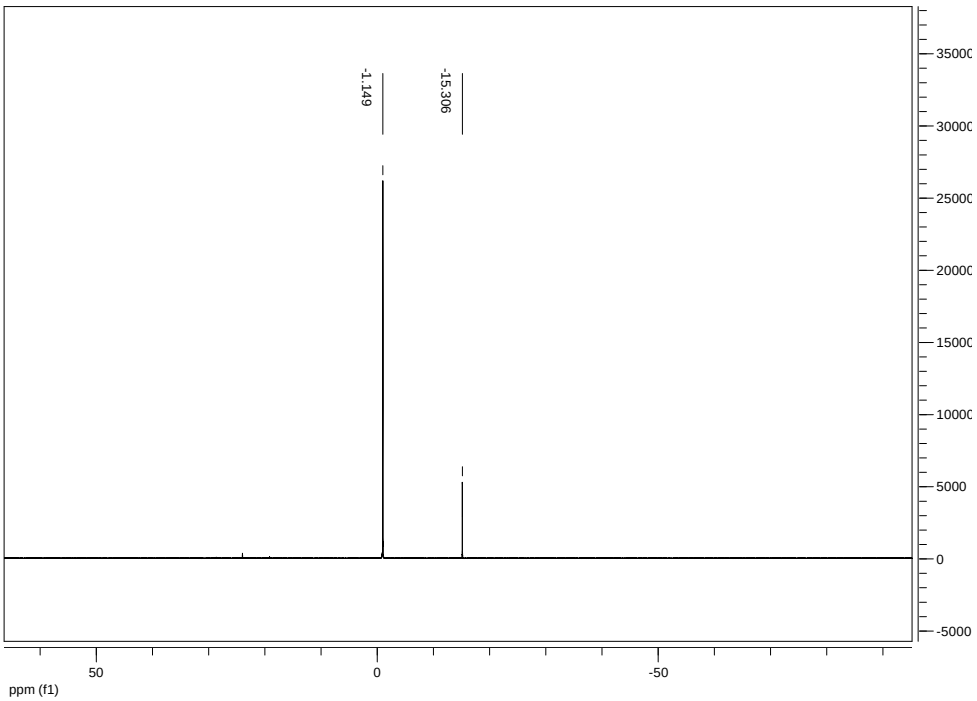

77.000
500
0
150
100
50
0
ppm (f1)


160.344
152.975
152.873
149.191
140.906
140.733
136.779
136.645
136.543
134.675
134.600
134.412
134.315
132.035
131.876
131.445
131.014
130.583
129.330
127.720
127.275
126.271
125.878
125.828
125.776
125.723
125.445
125.396
125.347
125.300
125.251
125.202
125.064
122.167
122.112
121.907
120.777
120.597
119.711
115.900
1000
500
0
160
150
140
130
120
110
100
ppm (f1)

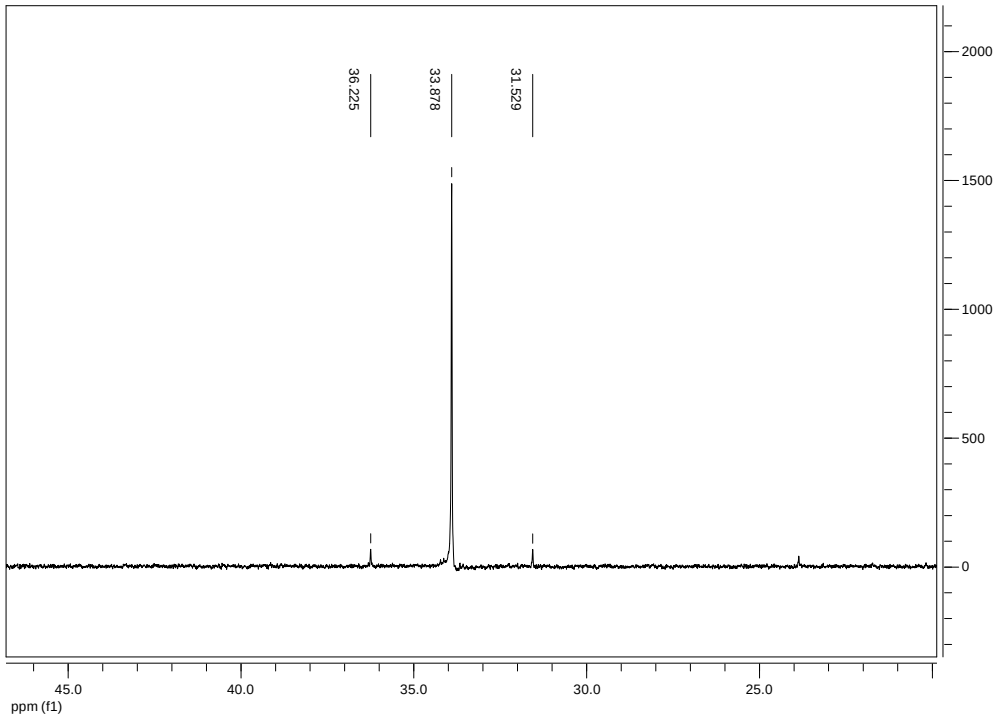
COSY H-H of 2g:



^{31}P NMR of 2g:



^{31}P NMR of 3g:



Crystal Structure Analysis

Compound **2d (B)**:

Crystal data

$C_{23}H_{17}N_4P$	D_x = 1.335 Mg m^{-3}
M_r = 380.38	
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 8814 reflections
a = 13.3540 (8) Å	θ = 1.0–27.5°
b = 14.4253 (4) Å	μ = 0.16 mm^{-1}
c = 19.6452 (10) Å	*T* = 173 (2) K
V = 3784.4 (3) Å^3	Cell measurement pressure: ? kPa
Z = 8	Block, colorless
F_{000} = 1584	0.30 × 0.25 × 0.15 mm

Data collection

KappaCCD diffractometer	4321 independent reflections
Radiation source: fine-focus sealed tube	2533 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.072
Detector resolution: ? pixels mm^{-1}	θ_{max} = 27.5°
T = 173(2) K	θ_{min} = 2.1°
P = ? kPa	*h* = −17 10
phi and ω scans	*k* = −18 12
Absorption correction: none	*l* = −18 25
16637 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.178$	$w = 1/[\sigma^2(F_o^2) + (0.0964P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
4321 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
254 parameters	$\Delta\rho_{min} = -0.53$ e Å^{-3}
? constraints	Extinction correction: SHELXL, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.0064 (10)

Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2917 (2)	0.3962 (2)	0.63462 (15)	0.0445 (7)
H1	0.2829	0.4109	0.6814	0.053*
C2	0.2707 (2)	0.31117 (19)	0.61084 (15)	0.0459 (8)
H2	0.2462	0.2652	0.6412	0.055*
C3	0.2843 (2)	0.28902 (18)	0.54184 (15)	0.0422 (7)
H3	0.2695	0.2282	0.5262	0.051*
C4	0.3184 (2)	0.35348 (16)	0.49725 (14)	0.0350 (7)

H4	0.3280	0.3383	0.4507	0.042*
C5	0.33937 (19)	0.44364 (16)	0.52139 (13)	0.0300 (6)
C6	0.3724 (2)	0.52895 (16)	0.49557 (13)	0.0306 (6)
C7	0.3990 (2)	0.55390 (16)	0.42598 (13)	0.0288 (6)
C8	0.4414 (2)	0.63897 (16)	0.40926 (14)	0.0354 (7)
H8	0.4536	0.6842	0.4434	0.042*
C9	0.4653 (2)	0.65633 (17)	0.34235 (15)	0.0402 (7)
H9	0.4940	0.7141	0.3298	0.048*
C10	0.4473 (2)	0.58907 (16)	0.29312 (15)	0.0375 (7)
H10	0.4648	0.5995	0.2469	0.045*
C11	0.4029 (2)	0.50588 (16)	0.31341 (13)	0.0312 (6)
C12	0.3848 (2)	0.31085 (16)	0.30077 (13)	0.0306 (6)
C13	0.3125 (2)	0.24143 (17)	0.30123 (13)	0.0380 (7)
H13	0.2513	0.2502	0.2773	0.046*
C14	0.3294 (3)	0.15884 (18)	0.33672 (14)	0.0446 (8)
H14	0.2805	0.1110	0.3359	0.054*
C15	0.4170 (3)	0.14667 (17)	0.37289 (14)	0.0419 (8)
H15	0.4282	0.0906	0.3970	0.050*
C16	0.4882 (2)	0.21564 (17)	0.37407 (14)	0.0400 (7)
H16	0.5479	0.2075	0.3997	0.048*
C17	0.4731 (2)	0.29699 (16)	0.33797 (14)	0.0347 (7)
H17	0.5232	0.3438	0.3384	0.042*
C18	0.4487 (2)	0.41595 (15)	0.18499 (13)	0.0307 (6)
C19	0.4092 (2)	0.40861 (17)	0.11937 (13)	0.0356 (7)
H19	0.3387	0.4095	0.1131	0.043*
C20	0.4709 (2)	0.40008 (19)	0.06368 (15)	0.0428 (7)
H20	0.4428	0.3948	0.0194	0.051*

C21	0.5739 (2)	0.39922 (17)	0.07201 (15)	0.0408 (7)
H21	0.6164	0.3932	0.0335	0.049*
C22	0.6144 (2)	0.40708 (16)	0.13581 (15)	0.0373 (7)
H22	0.6851	0.4067	0.1414	0.045*
C23	0.5524 (2)	0.41566 (16)	0.19283 (14)	0.0345 (7)
H23	0.5810	0.4213	0.2369	0.041*
N1	0.32636 (18)	0.46137 (14)	0.58918 (11)	0.0346 (5)
N2	0.35066 (19)	0.55064 (16)	0.60527 (12)	0.0412 (6)
N3	0.37766 (17)	0.59021 (14)	0.54793 (12)	0.0367 (6)
N4	0.37844 (17)	0.48871 (13)	0.37881 (10)	0.0293 (5)
P1	0.35641 (6)	0.41764 (4)	0.25394 (3)	0.0322 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0382 (18)	0.0616 (19)	0.0336 (16)	0.0025 (15)	0.0079 (15)	0.0098 (14)
C2	0.044 (2)	0.0508 (18)	0.0430 (18)	−0.0017 (15)	0.0055 (16)	0.0161 (14)
C3	0.0424 (19)	0.0372 (14)	0.0472 (18)	−0.0016 (13)	−0.0046 (16)	0.0092 (13)
C4	0.0356 (16)	0.0348 (14)	0.0346 (15)	0.0000 (12)	−0.0046 (13)	0.0013 (11)
C5	0.0254 (15)	0.0362 (14)	0.0283 (14)	0.0048 (11)	−0.0018 (12)	0.0023 (11)
C6	0.0284 (15)	0.0313 (13)	0.0321 (15)	0.0032 (11)	−0.0023 (12)	−0.0040 (11)
C7	0.0251 (14)	0.0273 (13)	0.0341 (15)	0.0037 (11)	−0.0038 (12)	−0.0001 (11)
C8	0.0364 (17)	0.0287 (14)	0.0410 (16)	0.0013 (12)	0.0006 (14)	−0.0010 (11)

C9	0.0376 (18)	0.0249 (13)	0.058 (2)	−0.0013 (12)	0.0038 (16)	0.0068 (13)
C10	0.0421 (18)	0.0312 (14)	0.0391 (16)	0.0041 (12)	0.0085 (14)	0.0076 (11)
C11	0.0315 (16)	0.0270 (13)	0.0351 (15)	0.0066 (11)	0.0038 (13)	0.0031 (11)
C12	0.0366 (16)	0.0284 (13)	0.0269 (14)	−0.0018 (11)	0.0054 (13)	−0.0036 (10)
C13	0.0401 (18)	0.0430 (15)	0.0310 (15)	−0.0077 (13)	0.0023 (14)	−0.0045 (12)
C14	0.058 (2)	0.0343 (15)	0.0416 (17)	−0.0156 (14)	0.0087 (17)	0.0002 (12)
C15	0.064 (2)	0.0272 (13)	0.0347 (16)	−0.0018 (14)	0.0032 (16)	0.0004 (11)
C16	0.0484 (19)	0.0366 (15)	0.0350 (16)	0.0033 (13)	−0.0024 (15)	−0.0028 (12)
C17	0.0400 (18)	0.0285 (13)	0.0356 (16)	−0.0042 (12)	−0.0016 (14)	0.0015 (11)
C18	0.0334 (16)	0.0262 (12)	0.0324 (15)	0.0000 (11)	0.0012 (13)	0.0016 (10)
C19	0.0328 (16)	0.0441 (15)	0.0299 (15)	0.0004 (12)	−0.0009 (13)	−0.0006 (11)
C20	0.046 (2)	0.0522 (17)	0.0303 (15)	0.0006 (14)	−0.0001 (15)	−0.0036 (12)
C21	0.049 (2)	0.0363 (15)	0.0369 (17)	0.0032 (13)	0.0128 (16)	−0.0032 (12)
C22	0.0320 (16)	0.0314 (14)	0.0485 (18)	0.0013 (12)	0.0031 (14)	0.0023 (12)
C23	0.0383 (17)	0.0322 (13)	0.0328 (15)	0.0012 (12)	−0.0021 (14)	0.0038 (11)
N1	0.0326 (14)	0.0419 (12)	0.0292 (13)	0.0010 (10)	0.0021 (11)	−0.0003 (10)
N2	0.0408 (15)	0.0473 (14)	0.0356 (14)	0.0001 (12)	0.0047 (12)	−0.0082 (11)

N3	0.0345 (14)	0.0396 (12)	0.0360 (14)	0.0015 (10)	0.0005 (11)	−0.0054 (10)
N4	0.0329 (14)	0.0256 (10)	0.0295 (12)	0.0023 (9)	0.0012 (11)	0.0009 (9)
P1	0.0329 (4)	0.0351 (4)	0.0287 (4)	0.0011 (3)	0.0018 (3)	0.0015 (3)

Geometric parameters (Å)

C1—C2	1.342 (4)	C12—P1	1.834 (3)
C1—N1	1.377 (3)	C13—C14	1.399 (4)
C1—H1	0.9500	C13—H13	0.9500
C2—C3	1.405 (4)	C14—C15	1.380 (4)
C2—H2	0.9500	C14—H14	0.9500
C3—C4	1.356 (4)	C15—C16	1.377 (4)
C3—H3	0.9500	C15—H15	0.9500
C4—C5	1.412 (3)	C16—C17	1.386 (4)
C4—H4	0.9500	C16—H16	0.9500
C5—N1	1.367 (3)	C17—H17	0.9500
C5—C6	1.402 (3)	C18—C23	1.394 (4)
C6—N3	1.358 (3)	C18—C19	1.397 (4)
C6—C7	1.458 (4)	C18—P1	1.831 (3)
C7—N4	1.348 (3)	C19—C20	1.374 (4)
C7—C8	1.391 (3)	C19—H19	0.9500
C8—C9	1.376 (4)	C20—C21	1.385 (4)
C8—H8	0.9500	C20—H20	0.9500
C9—C10	1.391 (4)	C21—C22	1.370 (4)
C9—H9	0.9500	C21—H21	0.9500
C10—C11	1.396 (3)	C22—C23	1.398 (4)
C10—H10	0.9500	C22—H22	0.9500

C11—N4	1.349 (3)	C23—H23	0.9500
C11—P1	1.836 (3)	N1—N2	1.365 (3)
C12—C13	1.392 (4)	N2—N3	1.313 (3)
C12—C17	1.401 (4)		
C2—C1—N1	117.9 (3)	C15—C14—C13	120.2 (3)
C2—C1—H1	121.0	C15—C14—H14	119.9
N1—C1—H1	121.0	C13—C14—H14	119.9
C1—C2—C3	121.1 (3)	C16—C15—C14	120.2 (3)
C1—C2—H2	119.4	C16—C15—H15	119.9
C3—C2—H2	119.4	C14—C15—H15	119.9
C4—C3—C2	120.7 (3)	C15—C16—C17	120.2 (3)
C4—C3—H3	119.6	C15—C16—H16	119.9
C2—C3—H3	119.6	C17—C16—H16	119.9
C3—C4—C5	118.8 (3)	C16—C17—C12	120.6 (2)
C3—C4—H4	120.6	C16—C17—H17	119.7
C5—C4—H4	120.6	C12—C17—H17	119.7
N1—C5—C6	103.2 (2)	C23—C18—C19	118.5 (3)
N1—C5—C4	118.3 (2)	C23—C18—P1	125.9 (2)
C6—C5—C4	138.5 (2)	C19—C18—P1	115.5 (2)
N3—C6—C5	108.3 (2)	C20—C19—C18	121.1 (3)
N3—C6—C7	122.5 (2)	C20—C19—H19	119.5
C5—C6—C7	129.2 (2)	C18—C19—H19	119.5
N4—C7—C8	122.4 (2)	C19—C20—C21	120.1 (3)
N4—C7—C6	115.0 (2)	C19—C20—H20	120.0
C8—C7—C6	122.6 (2)	C21—C20—H20	120.0
C9—C8—C7	118.7 (2)	C22—C21—C20	120.0 (3)
C9—C8—H8	120.6	C22—C21—H21	120.0

C7—C8—H8	120.6	C20—C21—H21	120.0
C8—C9—C10	119.8 (2)	C21—C22—C23	120.4 (3)
C8—C9—H9	120.1	C21—C22—H22	119.8
C10—C9—H9	120.1	C23—C22—H22	119.8
C9—C10—C11	118.3 (3)	C18—C23—C22	120.0 (3)
C9—C10—H10	120.8	C18—C23—H23	120.0
C11—C10—H10	120.8	C22—C23—H23	120.0
N4—C11—C10	122.2 (2)	N2—N1—C5	111.8 (2)
N4—C11—P1	113.37 (18)	N2—N1—C1	125.0 (2)
C10—C11—P1	123.9 (2)	C5—N1—C1	123.1 (2)
C13—C12—C17	118.6 (2)	N3—N2—N1	106.1 (2)
C13—C12—P1	117.6 (2)	N2—N3—C6	110.6 (2)
C17—C12—P1	123.75 (19)	C7—N4—C11	118.5 (2)
C12—C13—C14	120.2 (3)	C18—P1—C12	102.74 (11)
C12—C13—H13	119.9	C18—P1—C11	104.60 (12)
C14—C13—H13	119.9	C12—P1—C11	101.13 (11)
N1—C1—C2—C3	0.4 (5)	C20—C21—C22—C23	0.2 (4)
C1—C2—C3—C4	−0.5 (5)	C19—C18—C23—C22	−0.7 (3)
C2—C3—C4—C5	−0.4 (4)	P1—C18—C23—C22	175.05 (18)
C3—C4—C5—N1	1.4 (4)	C21—C22—C23—C18	0.2 (4)
C3—C4—C5—C6	−178.6 (3)	C6—C5—N1—N2	−0.8 (3)
N1—C5—C6—N3	0.5 (3)	C4—C5—N1—N2	179.2 (2)
C4—C5—C6—N3	−179.6 (3)	C6—C5—N1—C1	178.4 (2)
N1—C5—C6—C7	179.9 (3)	C4—C5—N1—C1	−1.5 (4)
C4—C5—C6—C7	−0.1 (5)	C2—C1—N1—N2	179.8 (3)
N3—C6—C7—N4	−172.5 (2)	C2—C1—N1—C5	0.6 (4)
C5—C6—C7—N4	8.1 (4)	C5—N1—N2—N3	0.9 (3)

N3—C6—C7—C8	6.8 (4)	C1—N1—N2—N3	−178.4 (3)
C5—C6—C7—C8	−172.6 (3)	N1—N2—N3—C6	−0.6 (3)
N4—C7—C8—C9	−1.5 (4)	C5—C6—N3—N2	0.1 (3)
C6—C7—C8—C9	179.2 (3)	C7—C6—N3—N2	−179.4 (2)
C7—C8—C9—C10	−0.3 (4)	C8—C7—N4—C11	2.1 (4)
C8—C9—C10—C11	1.4 (4)	C6—C7—N4—C11	−178.6 (2)
C9—C10—C11—N4	−0.8 (4)	C10—C11—N4—C7	−0.9 (4)
C9—C10—C11—P1	170.2 (2)	P1—C11—N4—C7	−172.77 (18)
C17—C12—C13—C14	−1.7 (4)	C23—C18—P1—C12	−60.1 (2)
P1—C12—C13—C14	−179.3 (2)	C19—C18—P1—C12	115.75 (19)
C12—C13—C14—C15	1.6 (4)	C23—C18—P1—C11	45.1 (2)
C13—C14—C15—C16	−0.2 (4)	C19—C18—P1—C11	−138.98 (18)
C14—C15—C16—C17	−1.2 (4)	C13—C12—P1—C18	−114.6 (2)
C15—C16—C17—C12	1.0 (4)	C17—C12—P1—C18	67.9 (2)
C13—C12—C17—C16	0.4 (4)	C13—C12—P1—C11	137.4 (2)
P1—C12—C17—C16	177.8 (2)	C17—C12—P1—C11	−40.0 (3)
C23—C18—C19—C20	0.8 (4)	N4—C11—P1—C18	−149.50 (19)
P1—C18—C19—C20	−175.4 (2)	C10—C11—P1—C18	38.8 (3)
C18—C19—C20—C21	−0.4 (4)	N4—C11—P1—C12	−43.0 (2)
C19—C20—C21—C22	−0.1 (4)	C10—C11—P1—C12	145.3 (2)

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Data collection: Collect (Nonius B.V., 1998); cell refinement: *DENZO* (Nonius B.V., 1998); data reduction: *DENZO* (Nonius B.V., 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* 98 (Spek, 1998); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).

Compound **3e (B)**:

Crystal data

$C_{25}H_{21}N_4PSe$	F_{000} = 496
M_r = 487.39	D_x = 1.456 Mg m^{-3}
Triclinic, *P*	
Hall symbol: -P 1	Mo *K*α radiation λ = 0.71073 Å
a = 8.6696 (8) Å	Cell parameters from 13838 reflections
b = 11.0202 (11) Å	θ = 1.0–27.5°
c = 12.2054 (15) Å	μ = 1.78 mm^{-1}
α = 88.075 (6)°	*T* = 173 (2) K
β = 87.392 (6)°	Cell measurement pressure: ? kPa
γ = 72.639 (6)°	Prism, colorless
V = 1111.6 (2) Å^3	0.16 × 0.12 × 0.10 mm
Z = 2	

Data collection

KappaCCD diffractometer	10682 measured reflections
Radiation source: fine-focus sealed tube	5065 independent reflections
Monochromator: graphite	2903 reflections with $I > 2\sigma(I)$
Detector resolution: ? pixels mm^{-1}	R_{int} = 0.079
T = 173(2) K	θ_{max} = 27.6°
P = ? kPa	θ_{min} = 1.7°
phi and ω scans	*h* = −11 9
Absorption correction: multi-scan MULscanABS in PLATON (Spek, 2003)	*k* = −14 14
T_{min} = 0.724, T_{max} = 0.846	*l* = −13 15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.092$	H-atom parameters constrained
$wR(F^2) = 0.263$	$w = 1/[\sigma^2(F_o^2) + (0.113P)^2 + 2.7079P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} \leq 0.001$
5065 reflections	$\Delta\rho_{max} = 0.63$ e Å^{-3}
282 parameters	$\Delta\rho_{min} = -0.88$ e Å^{-3}
? constraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2$sigma(F^2) is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.6687 (10)	−0.2313 (7)	−0.0289 (8)	0.041 (2)
H1	0.6735	−0.2967	−0.0788	0.049*
C2	0.6005 (10)	−0.2332 (7)	0.0724 (8)	0.043 (2)
H2	0.5573	−0.3003	0.0945	0.052*
C3	0.5939 (10)	−0.1366 (7)	0.1442 (7)	0.0383 (19)
H3	0.5455	−0.1388	0.2154	0.046*
C4	0.6540 (9)	−0.0399 (7)	0.1162 (6)	0.0303 (17)

H4	0.6494	0.0247	0.1669	0.036*
C5	0.7245 (8)	−0.0369 (6)	0.0090 (6)	0.0278 (17)
C6	0.7978 (8)	0.0405 (6)	−0.0533 (6)	0.0253 (16)
C7	0.8244 (8)	0.1601 (7)	−0.0289 (6)	0.0279 (17)
C8	0.9074 (9)	0.2210 (7)	−0.1025 (6)	0.0308 (17)
H8	0.9495	0.1847	−0.1711	0.037*
C9	0.9263 (9)	0.3351 (7)	−0.0724 (6)	0.0320 (17)
H9	0.9797	0.3796	−0.1214	0.038*
C10	0.8677 (9)	0.3845 (7)	0.0288 (6)	0.0311 (17)
H10	0.8835	0.4613	0.0518	0.037*
C11	0.7848 (8)	0.3186 (6)	0.0963 (6)	0.0249 (16)
C12	0.8150 (8)	0.4679 (6)	0.2812 (6)	0.0247 (15)
C13	0.9782 (9)	0.4065 (7)	0.2901 (6)	0.0300 (17)
H13	1.0227	0.3221	0.2649	0.036*
C14	1.0746 (10)	0.4666 (7)	0.3347 (6)	0.0338 (18)
H14	1.1862	0.4224	0.3414	0.041*
C15	1.0160 (10)	0.5918 (7)	0.3716 (6)	0.0329 (18)
C18	1.1258 (11)	0.6564 (8)	0.4200 (7)	0.046 (2)
H18A	1.0610	0.7310	0.4604	0.069*
H18B	1.1898	0.6834	0.3611	0.069*
H18C	1.1985	0.5972	0.4702	0.069*
C16	0.8540 (10)	0.6507 (7)	0.3611 (7)	0.038 (2)
H16	0.8097	0.7354	0.3857	0.046*
C17	0.7535 (10)	0.5920 (7)	0.3166 (6)	0.0331 (18)
H17	0.6419	0.6362	0.3100	0.040*
C19	0.6952 (8)	0.2530 (6)	0.3213 (6)	0.0250 (15)
C20	0.8219 (9)	0.1411 (7)	0.3190 (6)	0.0292 (17)

H20	0.9026	0.1278	0.2617	0.035*
C21	0.8311 (9)	0.0487 (7)	0.3998 (6)	0.0298 (17)
H21	0.9183	−0.0278	0.3967	0.036*
C22	0.7153 (9)	0.0649 (7)	0.4863 (6)	0.0322 (18)
C25	0.7301 (11)	−0.0331 (8)	0.5759 (7)	0.049 (2)
H25A	0.7921	−0.1167	0.5482	0.073*
H25B	0.6221	−0.0353	0.6013	0.073*
H25C	0.7859	−0.0117	0.6371	0.073*
C23	0.5894 (9)	0.1778 (8)	0.4867 (7)	0.038 (2)
H23	0.5091	0.1908	0.5443	0.046*
C24	0.5757 (9)	0.2723 (7)	0.4067 (6)	0.0317 (18)
H24	0.4878	0.3483	0.4092	0.038*
N1	0.7305 (7)	−0.1347 (5)	−0.0588 (5)	0.0318 (15)
N2	0.8016 (8)	−0.1189 (6)	−0.1577 (5)	0.0406 (17)
N3	0.8405 (8)	−0.0134 (6)	−0.1534 (5)	0.0365 (16)
N4	0.7625 (7)	0.2083 (5)	0.0696 (5)	0.0248 (13)
P1	0.6831 (2)	0.38533 (17)	0.22541 (15)	0.0248 (4)
Se1	0.44749 (10)	0.50076 (8)	0.19694 (8)	0.0445 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.040 (5)	0.024 (4)	0.060 (6)	−0.006 (4)	−0.027 (4)	−0.008 (4)
C2	0.044 (5)	0.029 (4)	0.058 (6)	−0.012 (4)	−0.008 (4)	0.007 (4)
C3	0.041 (5)	0.037 (4)	0.039 (5)	−0.014 (4)	−0.007 (4)	0.005 (4)
C4	0.031 (4)	0.027 (4)	0.030 (4)	−0.005 (3)	−0.006 (3)	−0.002 (3)
C5	0.022 (4)	0.023 (4)	0.033 (4)	0.002 (3)	−0.011 (3)	−0.005 (3)
C6	0.021 (4)	0.025 (4)	0.027 (4)	−0.003 (3)	−0.003 (3)	−0.003 (3)

C7	0.024 (4)	0.027 (4)	0.030 (4)	−0.003 (3)	−0.004 (3)	−0.005 (3)
C8	0.024 (4)	0.041 (4)	0.026 (4)	−0.007 (3)	−0.003 (3)	0.000 (3)
C9	0.032 (4)	0.038 (4)	0.033 (5)	−0.023 (3)	−0.003 (3)	0.009 (3)
C10	0.034 (4)	0.030 (4)	0.034 (5)	−0.016 (3)	−0.006 (3)	0.002 (3)
C11	0.021 (4)	0.025 (4)	0.027 (4)	−0.004 (3)	−0.004 (3)	−0.001 (3)
C12	0.028 (4)	0.025 (4)	0.025 (4)	−0.013 (3)	−0.003 (3)	−0.001 (3)
C13	0.035 (4)	0.023 (4)	0.033 (4)	−0.010 (3)	−0.001 (3)	−0.001 (3)
C14	0.034 (4)	0.033 (4)	0.037 (5)	−0.014 (3)	−0.005 (3)	−0.001 (3)
C15	0.042 (5)	0.036 (4)	0.028 (4)	−0.022 (4)	−0.007 (3)	−0.002 (3)
C18	0.054 (5)	0.050 (5)	0.045 (5)	−0.031 (4)	−0.011 (4)	−0.010 (4)
C16	0.043 (5)	0.031 (4)	0.040 (5)	−0.008 (4)	−0.004 (4)	−0.017 (4)
C17	0.037 (4)	0.026 (4)	0.037 (5)	−0.009 (3)	0.001 (3)	−0.009 (3)
C19	0.026 (4)	0.029 (4)	0.025 (4)	−0.016 (3)	−0.003 (3)	−0.006 (3)
C20	0.026 (4)	0.034 (4)	0.030 (4)	−0.011 (3)	0.002 (3)	−0.009 (3)
C21	0.035 (4)	0.023 (4)	0.033 (4)	−0.012 (3)	−0.005 (3)	−0.001 (3)
C22	0.041 (5)	0.042 (4)	0.025 (4)	−0.028 (4)	−0.012 (3)	0.001 (3)
C25	0.057 (6)	0.052 (5)	0.046 (5)	−0.030 (5)	−0.009 (4)	0.008 (4)
C23	0.032 (4)	0.057 (5)	0.032 (5)	−0.024 (4)	0.005 (3)	−0.010 (4)
C24	0.028 (4)	0.034 (4)	0.032 (4)	−0.008 (3)	0.000 (3)	−0.002 (3)
N1	0.032 (4)	0.025 (3)	0.037 (4)	−0.006 (3)	−0.010 (3)	−0.008 (3)
N2	0.049 (4)	0.036 (4)	0.034 (4)	−0.006 (3)	−0.008 (3)	−0.013 (3)
N3	0.042 (4)	0.033 (4)	0.032 (4)	−0.006 (3)	0.002 (3)	−0.014 (3)
N4	0.024 (3)	0.024 (3)	0.028 (3)	−0.010 (3)	−0.002 (2)	−0.002 (2)
P1	0.0230 (9)	0.0232 (9)	0.0300 (11)	−0.0087 (8)	−0.0029 (8)	−0.0045 (7)
Se1	0.0340 (5)	0.0406 (5)	0.0593 (7)	−0.0112 (4)	−0.0020 (4)	−0.0036 (4)

Geometric parameters (Å)

C1—C2	1.348 (12)	C14—C15	1.402 (10)
C1—N1	1.361 (10)	C14—H14	0.9500
C1—H1	0.9500	C15—C16	1.371 (11)
C2—C3	1.387 (12)	C15—C18	1.499 (11)
C2—H2	0.9500	C18—H18A	0.9800
C3—C4	1.347 (11)	C18—H18B	0.9800
C3—H3	0.9500	C18—H18C	0.9800
C4—C5	1.422 (11)	C16—C17	1.369 (11)
C4—H4	0.9500	C16—H16	0.9500
C5—N1	1.368 (9)	C17—H17	0.9500
C5—C6	1.394 (10)	C19—C20	1.385 (10)
C6—N3	1.363 (9)	C19—C24	1.410 (10)
C6—C7	1.447 (10)	C19—P1	1.821 (7)
C7—N4	1.353 (9)	C20—C21	1.381 (10)
C7—C8	1.401 (11)	C20—H20	0.9500
C8—C9	1.379 (11)	C21—C22	1.401 (11)
C8—H8	0.9500	C21—H21	0.9500
C9—C10	1.380 (10)	C22—C23	1.389 (11)
C9—H9	0.9500	C22—C25	1.493 (11)
C10—C11	1.393 (10)	C25—H25A	0.9800
C10—H10	0.9500	C25—H25B	0.9800
C11—N4	1.340 (9)	C25—H25C	0.9800
C11—P1	1.834 (7)	C23—C24	1.386 (11)
C12—C13	1.382 (10)	C23—H23	0.9500
C12—C17	1.388 (10)	C24—H24	0.9500
C12—P1	1.826 (7)	N1—N2	1.359 (9)
C13—C14	1.353 (11)	N2—N3	1.307 (9)

C13—H13	0.9500	P1—Se1	2.0965 (19)
C2—C1—N1	119.3 (7)	H18A—C18—H18B	109.5
C2—C1—H1	120.4	C15—C18—H18C	109.5
N1—C1—H1	120.4	H18A—C18—H18C	109.5
C1—C2—C3	119.6 (8)	H18B—C18—H18C	109.5
C1—C2—H2	120.2	C17—C16—C15	122.4 (7)
C3—C2—H2	120.2	C17—C16—H16	118.8
C4—C3—C2	122.1 (8)	C15—C16—H16	118.8
C4—C3—H3	119.0	C16—C17—C12	119.9 (7)
C2—C3—H3	119.0	C16—C17—H17	120.1
C3—C4—C5	118.5 (7)	C12—C17—H17	120.1
C3—C4—H4	120.8	C20—C19—C24	119.7 (7)
C5—C4—H4	120.8	C20—C19—P1	123.1 (6)
N1—C5—C6	104.2 (6)	C24—C19—P1	117.0 (5)
N1—C5—C4	117.6 (7)	C21—C20—C19	120.2 (7)
C6—C5—C4	138.1 (7)	C21—C20—H20	119.9
N3—C6—C5	107.5 (6)	C19—C20—H20	119.9
N3—C6—C7	121.2 (7)	C20—C21—C22	121.7 (7)
C5—C6—C7	131.2 (6)	C20—C21—H21	119.2
N4—C7—C8	122.9 (7)	C22—C21—H21	119.2
N4—C7—C6	114.9 (6)	C23—C22—C21	117.0 (7)
C8—C7—C6	122.2 (7)	C23—C22—C25	121.6 (7)
C9—C8—C7	118.1 (7)	C21—C22—C25	121.3 (7)
C9—C8—H8	120.9	C22—C25—H25A	109.5
C7—C8—H8	120.9	C22—C25—H25B	109.5
C8—C9—C10	120.0 (7)	H25A—C25—H25B	109.5
C8—C9—H9	120.0	C22—C25—H25C	109.5

C10—C9—H9	120.0	H25A—C25—H25C	109.5
C9—C10—C11	118.1 (7)	H25B—C25—H25C	109.5
C9—C10—H10	120.9	C24—C23—C22	122.9 (7)
C11—C10—H10	120.9	C24—C23—H23	118.6
N4—C11—C10	123.6 (6)	C22—C23—H23	118.6
N4—C11—P1	114.9 (5)	C23—C24—C19	118.5 (7)
C10—C11—P1	121.3 (5)	C23—C24—H24	120.7
C13—C12—C17	118.9 (7)	C19—C24—H24	120.7
C13—C12—P1	120.1 (5)	N2—N1—C1	126.3 (7)
C17—C12—P1	120.9 (6)	N2—N1—C5	110.8 (6)
C14—C13—C12	119.9 (7)	C1—N1—C5	122.9 (7)
C14—C13—H13	120.0	N3—N2—N1	106.8 (6)
C12—C13—H13	120.0	N2—N3—C6	110.6 (6)
C13—C14—C15	122.5 (7)	C11—N4—C7	117.2 (6)
C13—C14—H14	118.8	C19—P1—C12	104.2 (3)
C15—C14—H14	118.8	C19—P1—C11	107.4 (3)
C16—C15—C14	116.4 (7)	C12—P1—C11	105.0 (3)
C16—C15—C18	122.3 (7)	C19—P1—Se1	114.6 (2)
C14—C15—C18	121.3 (7)	C12—P1—Se1	114.7 (2)
C15—C18—H18A	109.5	C11—P1—Se1	110.3 (2)
C15—C18—H18B	109.5		
N1—C1—C2—C3	0.2 (12)	C22—C23—C24—C19	−0.2 (12)
C1—C2—C3—C4	−0.1 (12)	C20—C19—C24—C23	0.3 (11)
C2—C3—C4—C5	0.8 (11)	P1—C19—C24—C23	−174.4 (6)
C3—C4—C5—N1	−1.5 (10)	C2—C1—N1—N2	−179.5 (7)
C3—C4—C5—C6	179.8 (8)	C2—C1—N1—C5	−1.1 (11)
N1—C5—C6—N3	0.9 (7)	C6—C5—N1—N2	−0.6 (7)

C4—C5—C6—N3	179.6 (8)	C4—C5—N1—N2	−179.6 (6)
N1—C5—C6—C7	178.0 (7)	C6—C5—N1—C1	−179.2 (6)
C4—C5—C6—C7	−3.2 (14)	C4—C5—N1—C1	1.7 (10)
N3—C6—C7—N4	174.0 (6)	C1—N1—N2—N3	178.6 (6)
C5—C6—C7—N4	−2.8 (11)	C5—N1—N2—N3	0.0 (8)
N3—C6—C7—C8	−5.7 (10)	N1—N2—N3—C6	0.5 (8)
C5—C6—C7—C8	177.5 (7)	C5—C6—N3—N2	−0.9 (8)
N4—C7—C8—C9	0.0 (10)	C7—C6—N3—N2	−178.4 (6)
C6—C7—C8—C9	179.7 (7)	C10—C11—N4—C7	−0.2 (10)
C7—C8—C9—C10	1.6 (11)	P1—C11—N4—C7	175.0 (5)
C8—C9—C10—C11	−2.4 (11)	C8—C7—N4—C11	−0.7 (10)
C9—C10—C11—N4	1.8 (11)	C6—C7—N4—C11	179.6 (6)
C9—C10—C11—P1	−173.1 (5)	C20—C19—P1—C12	−80.7 (6)
C17—C12—C13—C14	1.2 (11)	C24—C19—P1—C12	93.8 (6)
P1—C12—C13—C14	−177.9 (6)	C20—C19—P1—C11	30.3 (7)
C12—C13—C14—C15	−1.1 (12)	C24—C19—P1—C11	−155.2 (6)
C13—C14—C15—C16	0.6 (12)	C20—C19—P1—Se1	153.2 (5)
C13—C14—C15—C18	−179.5 (8)	C24—C19—P1—Se1	−32.4 (6)
C14—C15—C16—C17	−0.4 (12)	C13—C12—P1—C19	62.3 (6)
C18—C15—C16—C17	179.8 (8)	C17—C12—P1—C19	−116.7 (6)
C15—C16—C17—C12	0.6 (13)	C13—C12—P1—C11	−50.4 (7)
C13—C12—C17—C16	−1.0 (11)	C17—C12—P1—C11	130.5 (6)
P1—C12—C17—C16	178.1 (6)	C13—C12—P1—Se1	−171.6 (5)
C24—C19—C20—C21	0.1 (11)	C17—C12—P1—Se1	9.4 (7)
P1—C19—C20—C21	174.4 (6)	N4—C11—P1—C19	37.3 (6)
C19—C20—C21—C22	−0.5 (11)	C10—C11—P1—C19	−147.4 (6)
C20—C21—C22—C23	0.6 (11)	N4—C11—P1—C12	147.7 (5)

C20—C21—C22—C25	−177.3 (7)	C10—C11—P1—C12	−36.9 (6)
C21—C22—C23—C24	−0.2 (11)	N4—C11—P1—Se1	−88.3 (5)
C25—C22—C23—C24	177.7 (8)	C10—C11—P1—Se1	87.1 (6)

All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Data collection: Collect (Nonius B.V., 1998); cell refinement: *DENZO* (Nonius B.V., 1998); data reduction: *DENZO* (Nonius B.V., 1998); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* 98 (Spek, 1998); software used to prepare material for publication: *SHELXL97* (Sheldrick, 1997).