Gold(I) catalyses the intermolecular hydroamination of alkynes with imines and produces α, α´,N-triarylbisenamines: studies on their use as intermediates in synthesis.

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SUPPORTING INFORMATION

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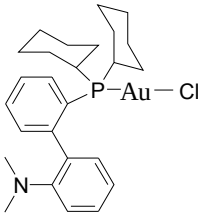
Experimental Section.

General.

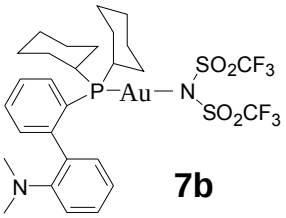
Glassware was dried in an oven at 175 ºC before use. Reagents and solvents were obtained from commercial sources and were used without further purification otherwise indicated. Gold (I) complexes **7a**,[S1] **7c**[S1] and **7d**[S2] and $AgNTf_2$[S3] were prepared as previously reported. All the products obtained were characterised by GC-MS, ^{1}H- and ^{13}C-NMR, and DEPT. Gas chromatographic analyses were performed in an instrument equipped with a 25 m capillary column of 5% phenylmethylsilicone. GC/MS analyses were performed on a spectrometer equipped with the same column as the GC and operated under the same conditions. HRMS were performed using the electrospray ionization technique. ^{1}H, ^{13}C, DEPT and ^{31}P-NMR were recorded in a 300 MHz instrument using $CDCl_3$ as solvent otherwise indicated, containing TMS as internal standard. IR spectra of the compounds were recorded on a spectrophotometer by impregnating the windows with a dichloromethane solution of the compound and leaving to evaporate before analysis. UV-Visible measurements were recorded on a spectrophotometer using CH_2Cl_2 as solvent. Elemental analyses of the solids were determined by chemical combustion using a CHNSO analyzer.

Syntheses of gold catalysts.



Synthesis of AuDavePhosCl. Tetrachloroauric acid trihydrate (394 mg, 1 mmol) was dissolved in distilled water (1 mL) under nitrogen and the solution cooled in ice. Then,

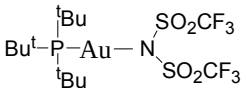
2,2´-thiodiethanol was slowly added over 45 min until disappearance of the color (ca. 300 μL). After that, 2'-dicyclohexylphosphanylbiphenyl-2-yl-dimethylamine (DavePhos, 394 mg, 1 mmol) was added and then 3 mL of ethanol. The mixture was stirred at rt for 3 h. Then, a $NaHCO_3$ aqueous solution (c.a. 20 mL) was added to the clear solution until pH~7, a white solid starting precipitating. The solid was filtered off and redissolved in CH_2Cl_2, extracted with brine, dried over $MgSO_4$ and filtered. The solution was concentrated to dryness to obtain AuDavePhosCl as a white powder (500 mg, 0.80 mmol, 80 %). IR (cm^{-1}): 2931, 2854, 1446, 1327, 1203, 1142, 1057, 741. ^1H NMR (δ, ppm; *J*, Hz): 7.60-7.40 (4H, mult), 7.36 (1H, ddd, *J*= 7.5, 4.1, 1.5), 7.06 (2H, mult), 6.97 (1H, dd, *J*= 7.4, 1.7), 2.46 (6H, s), 2.30 (1H, qt, *J*= 11.8, 2.8), 2.00 (3H, mult), 1.85 (1H, mult), 1.80-1.50 (8H, mult), 1.40-1.05 (9H, mult). ^{13}C NMR (δ, ppm; J_{C-P}, Hz): 151.1 (C), 148.5 (C, *J*= 12), 134.6 (C), 133.35 (CH, *J* =8), 132.2 (CH, *J* =4), 131.4 (CH), 130.8 (CH, *J* =2), 129.3 (CH), 126.9 (CH, *J* =7), 125.4 (C, *J* =53), 121.9 (CH), 119.7 (CH), 43.7 (CH_3, x2), 37.7 (CH, *J* =33), 35.6 (CH, *J* =34), 30.7 (CH_2, *J*=4), 30.2 (CH_2, *J*=3), 29.9 (CH_2), 28.8 (CH_2), 26.9 (CH_2), 26.6 (CH_2, *J*=3), 26.5 (CH_2, *J*=5), 26.4 (CH_2), 25.6 (CH_2, *J*=2), 25.5 (CH_2, *J*=2). ^{31}P NMR (δ, ppm): 39.4. E.A. (calculated for $C_{26}H_{36}AuClNP$: C, 49.89; H, 5.80; N, 2.24) found: C 49.19, H 5.74, N 2.20. HRMS (ESI) [M^+; calculated for $C_{26}H_{36}AuClNP$: 625.1939] found *m/z* 625.1936; [$(M-Cl)^+$, major peak ; calculated for $C_{26}H_{36}AuNP$: 590.2251] found *m/z* 590.2232.



7b

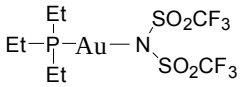
Synthesis of AuDavePhosNTf$_2$ (7b). AuDavePhosCl (350 mg, 0.56 mmol) and $AgNTf_2$ (217 mg, 0.56 mmol) were placed in a round-bottomed flask. Air evacuation-

nitrogen refilling cycles were carried out and a rubber septum was rapidly fitted after the last nitrogen refilling, a nitrogen balloon additionally coupled through a needle. Then, dry CH_2Cl_2 (15 mL) was added and the mixture was magnetically stirred at rt for 30 min. Then, the white solid formed (AgCl) was filtered off over celite and the clear filtrates were concentrated to dryness to obtain AuDavePhosNTf$_2$ as a yellow-bright crystalline powder (490 mg, quantitative). Crystals can be obtained by dissolving the solid in the minimum amount of CH_2Cl_2 and adding hexane until blurring is observed, then leaving the mixture into a fridge at – 14 ºC overnight and filtering the crystal thus obtained. IR (cm^{-1}): 2931, 2854, 1342, 1196, 1142, 1057. ^{1}H NMR (δ, ppm; *J*, Hz): 7.64-7.38 (5H, mult), 7.11 (2H, t, *J*= 7.7), 6.99 (1H, d, *J*= 7.0), 2.46 (6H, s), 2.28-2.00 (4H, mult), 1.86-1.62 (8H, mult), 1.48-1.18 (10H, mult). ^{13}C NMR (δ, ppm; $J_{\text{C-P, otherwise indicated}}$, Hz): 151.2 (C), 148.1 (C,), 133.4 (CH), 132.4 (CH,), 131.6 (CH, x2), 129.3 (CH), 127.3 (CH), 124.0 (C), 123.3 (C,), 122.2 (CH), 119.4 (C, $J_{\text{C-F}}$= 323), 119.1 (CH), 43.4 (CH_3, x2), 37.4 (CH, *J* =34), 36.3 (CH, *J* =34), 31.0 (CH_2), 29.6 (CH_2,), 26.6 (CH_2), 26.5 (CH_2, x2), 26.4 (CH_2), 26.3 (CH_2), 25.6 (CH_2), 25.5 (CH_2, x2). ^{31}P NMR (δ, ppm): 39.1. E.A. (calculated for $C_{28}H_{36}AuF_6N_2O_4PS_2$: C, 38,63; H, 4,17; N, 3,22; S, 7,37) found C, 39,53; H, 4,32; N, 3,24; S, 7,43. HRMS (ESI) [(M-Cl)$^+$, major peak ; calculated for $C_{26}H_{36}AuNP$: 590.2251] found *m/z* 590.2258.



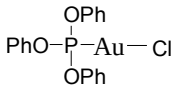
7c

Synthesis of Au(P^{t}Bu$_3$)NTf$_2$ 7c.[S1] Au(P^{t}Bu$_3$)Cl (435 mg, 1 mmol) and AgNTf$_2$ (388 mg, 1 mmol) were mixed in dry dichloromethane (25 mL). The mixture was stirred at rt under nitrogen for 30 min and filtered over celite. The solution was concentrated to dryness to obtain Au(P^{t}Bu$_3$)NTf$_2$ as a white solid (675 mg, 0.99 mmol, quantitative). Crystals can be obtained by dissolving the solid in the minimum amount of CH_2Cl_2 and leaving to evaporate at room temperature. IR (cm^{-1}): 3437, 2951, 1398, 1205, 1134, 958. ^{1}H NMR (δ, ppm; *J*, Hz): 1.52 (27H, d, *J*= 14.3). ^{13}C NMR (δ, ppm; *J*, Hz): 119.3 (2C, q, $J_{\text{C-F}}$= 323.0), 39.8 (3C, d, $J_{\text{C-P}}$= 11.9), 32.1 (9C, d, $J_{\text{C-P}}$= 3.8). ^{31}P NMR (δ, ppm): 90.9. FAB$^+$ [M$^+$; calculated for $C_{14}H_{27}AuF_6NO_4PS_2$: 679] found *m/z* 399 (M$^+$-N(SO$_2$CF$_3$)$_2$), 833 (P^{t}Bu$_3$AuClAuP^{t}Bu$_3$).

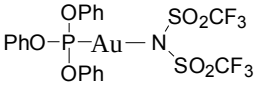


7d

Synthesis of Au(PEt$_3$)NTf$_2$ 7d. Following the same procedure than for **7c** but using Au(PEt$_3$)Cl (200 mg, 0.57 mmol) as gold precursor (grey solid, 340 mg, quantitative). IR (cm^{-1}): 3511, 2928, 1342, 1200, 1140, 1057. ^{1}H NMR (δ, ppm; *J*, Hz): 1.90 (4H, dq, J_{H-P}= 10.6, *J*= 7.8), 1.21 (6H, dt, J_{H-P}= 19.7, *J*= 7.7). ^{13}C NMR (δ, ppm): 119.3 (2C, q, J_{C-F}= 322.7), 38.4 (3C, d, J_{C-P}= 38.4), 9.2 (3C, d, J_{C-P}= 1.7). ^{31}P NMR (δ, ppm): 32.6.



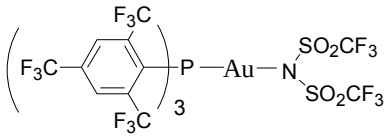
Synthesis of AuP(OPh)$_3$Cl. Tetrachloroauric acid trihydrate (394 mg, 1 mmol) was dissolved in distilled water (1 mL) under nitrogen and the solution cooled in ice. Then, 2,2´-thiodiethanol was slowly added over 45 min until disappearance of the color (ca. 300 μL). After that, triphenylphosphite (236 μL, 1 mmol) was added and then 3 mL of ethanol. The mixture was stirred at rt for 3 h. The solid was filtered off and washed with methanol, re-dissolved in dry dichloromethane and filtered again. The solution was concentrated to dryness to obtain AuP(OPh)$_3$Cl as a white solid. ^{1}H NMR (δ, ppm; *J*, Hz): 7.32 (6H, t, *J*= 8.0), 7.17 (9H, mult). ^{13}C NMR (δ, ppm; J_{C-P}, Hz): 149.3 (3C, d, *J*= 4.9), 130.5 (6C, d, *J*= 1.7), 126.7 (3C, d, *J*= 1.6), 121.1 (6C, d, *J*= 5.6). ^{31}P NMR (δ, ppm): 109.7.



7f

Synthesis of AuP(OPh)$_3$NTf$_2$ 7f. Following the same procedure than for **7c** but using AuP(OPh)$_3$Cl (130 mg, 0.24 mmol) as gold precursor (white solid, 100 mg, 54 % yield). IR (cm^{-1}): 3054, 2981, 1590, 1486, 1404, 1264, 1207, 1136, 952, 739. ^{1}H NMR

(δ, ppm; *J*, Hz): 7.44 (6H, t, *J*= 7.7), 7.34 (3H, tq, *J*= 7.6, 1.3), 7.20 (6H, mult). ^{13}C NMR (δ, ppm; $J_{C\text{-}P}$, Hz): 148.8 (3C, d, *J*= 5.0), 130.6 (6C, d, *J*= 2.2), 127.1 (3C, d, *J*= 2.2), 121.1 (6C, d, *J*= 6.0). ^{31}P NMR (δ, ppm): 103.7.



7g

Synthesis of AuP(*tris*CF_3-phenyl)$_3$$NTf_2$ 7g. Following the same procedure than for **7c** but using AuP(*tris*CF_3-phenyl)$_3$Cl complex (200 mg, 0.28 mmol) as gold precursor (slightly purple-white solid, 265 mg, 71 %). IR (cm^{-1}): 1396, 1327, 1203, 1134, 1057, 956, 833, 710, 601. ^{1}H NMR (δ, ppm; *J*, Hz): 7.87 (3H, ddt, *J*= 8.1, 2.3, 0.6), 7.84 (3H, ddt, *J*= 13.4, 8.1, 0.6). ^{13}C NMR (δ, ppm; $J_{C\text{-}P}$, Hz): 134.6 (3C, d, *J*= 14.9), 126.9 (3C, dq, *J*= 12.4, 3.2), 135.9-112.9 (23C, mult). ^{31}P NMR (δ, ppm): 30.7. HRMS (ESI) [M^++H^+; calculated for $C_{29}H_7AuF_{33}NO_4PS_2$: 1351.8691] found *m/z* 1352. 0453.

Reaction Procedures.

General double-hydroamination procedure (bisenamine 6). Complex **7b** (22 mg, 5 mol %) and *p*-toluidine **1** (54 mg, 0.5 mmol) were placed into a vial. Then, dry CH_3CN (0.25 mL) and phenylacetylene **2** (220 μL, 2 mmol) were sequentially added (alternatively, the reaction can be run without solvent). The vial was sealed and the mixture was magnetically stirred in a pre-heated oil bath at 80 ºC for 24 h. After cooling, an aliquot was taken for GC analysis. The CH_3CN was removed under vacuo and CH_2Cl_2 (1 mL) was added to re-dissolve the mixture. Then, *n*-hexane (10-20 mL) was added and the mixture was vigorously stirred for 15 min and filtered over celite. The resulting filtrates were concentrated under reduced pressure and analysed by NMR. The crude was purified by column chromatography on basic alumina (2-5 % AcOEt in *n*-hexane) to achieve bis-(1-phenylvinyl)-*p*-tolylamine **6** as a yellow oil (140 mg, 0.45 mmol, 90 %).

Hydrogenation procedure for bisenamine 6 and isolation of the corresponding tertiary amines 26 and 27. Pt/C (3 wt%, 12 mg, Pt: 15 mol %) and bisenamine **6** (39 mg, 0.125 mmol) were placed into a 2 ml thin double-walled vial having a pressure

controller and an H_2 inlet/outlet. Dry THF (1 ml) was added and the vial was sealed, purged three times with H_2 (8-10 atm) and finally loaded with H_2 (12 atm, 0.13 mmol, 5 eq.). The mixture was magnetically stirred in a pre-heated oil bath at 50 ºC for 6 h. Progressive loss of the yellow color of the solution was observed. After cooling, the remaining H_2 was removed and the mixture was filtered. The resulting solution was analysed by GC and GC-MS and the amines were quantitatively separated and purified by TLC on silica (2 % AcOEt in hexane). After extraction from the silica with neat AcOEt and removal of the solvents, 18 mg of each amine were obtained as white oils (>95 % yield).

Pd-catalyzed intramolecular oxidative coupling of 6. $Pd(OAc)_2$ (2.2 mg, 20 mol %), bisenamine **6** (15.6 mg, 0.05 mmol) and $NaHCO_3$ (12.6 mg, 3 eq.) were placed into a vial. Dry DMF (0.25 ml) and PhI (16.7 μl, 3 eq.) were added and the vial was sealed and magnetically stirred in a pre-heated oil bath at 140 ºC for 20 h. After cooling, the resulting solution was analysed by GC and GC-MS and the product was purified by TLC on silica (2 % AcOEt in hexane). After extraction from the silica with neat AcOEt and removal of the solvents, 15 mg of pyrrole **29** were obtained (>95 % yield). The spectroscopical data of **29** fit those previously reported.[S4]

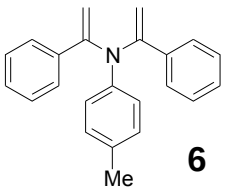
Typical reaction procedure (addition of bisenamine 6 to DMAD 31). Bisenamine **6** (15.6 mg, 0.05 mmol) was placed into a vial. Dry DMF (0.25 ml) and DMAD (24.6 μl, 4 eq.) were added and the vial was sealed and magnetically stirred in a pre-heated oil bath at 140 ºC for 20 h. After cooling, the resulting solution was analysed by GC and GC-MS and the product was purified by TLC on silica (10 % AcOEt in hexane). After extraction from the silica with neat AcOEt and removal of the solvents, 19 mg of **32** were obtained (85 % yield). Yellow crystals were obtained by re-dissolving in CH_2Cl_2 and slow evaporation.

Isotopic experiments (Schemes 5 and 6). *p*-Toluidine **1** (21.4 mg, 0.2 mmol) and $AuSPhosNTf_2$ **7a** or $AuP^tBu_3NTf_2$ **7c** (1 mol%) were placed into a vial equipped with a magnetic bar. Then, d^6-CH_3CN (0.4 mL, 0.5 M solution) and ^{13}C-marked phenylacetylene **2** (24 μL, 1.1 eq.) were sequentially added and the vial was sealed. The solution was magnetically stirred at room temperature for 24 h. Then, $AuP^tBu_3NTf_2$ **7c** (17 mg, 5 mol%) and phenylacetylene **2** or d^1-phenylacetylene **37** (63 μL, 3 eq.) were added and the mixture was magnetically stirred in a pre-heated oil bath at 80 ºC for 24 h. After this time, DMAD **31** (30 μL, 1.2 eq.) was added and the mixture was

magnetically stirred at 80 ºC for additional 20 h. The resulting solution was analysed by GC and GC-MS and the product was purified by TLC on silica (10-20 % AcOEt in hexane). After extraction from the silica with neat AcOEt and removal of the solvents, 21 mg of **36a,b** (23 %) or 30 mg of **42-45a,b** (35 %) were obtained. All the intermediated were analysed by *in-situ* NMR and GC-MS.

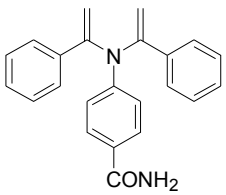
UV measurements. A 1 μM DCM solution of the compounds was prepared as follows: **32** (2.26 mg, 0.005 mmol) was diluted in 5 mL of DCM and then 25 μL of this solution were diluted in 25 mL of DCM. The same procedure was followed for **55** (2.32 mg, 0.005 mmol), **56** (2.40 mg, 0.005 mmol), and *p*-octylmethoxycinnamate (1.45 μL, 0.005 mmol).

Compound Characterization.



6

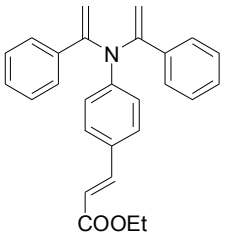
The yellow oil, after column chromatography, solidifies in a fridge at -14 ºC as yellow crystals, which were washed with *n*-hexane and dried. R_f (10 % AcOEt in hexane): 0.69. GC-MS (*m/z)*: 311 ($M^{+\cdot}$, 100 %), 283 (14 %), 194 (100 %). IR (cm^{-1}): 3034, 2924, 1611, 1505, 1321, 1258, 773, 702. ^{1}H NMR (δ, ppm; *J*, Hz): 7.49 (4H, mult), 7.11 (6H, mult), 6.89 (2H, d, *J*=8), 6.82 (2H, d, *J*=8), 4.91 (2H, s), 4.54 (2H, s), 2.08 (3H, s). ^{13}C NMR (δ, ppm): 151.7, 144.4 (2C), 138.9, 132.5 (2C), 129.3 (2C), 128.1 (2C), 127.9 (2C), 126.7 (4C), 125.2 (2C), 125.2 (2C), 106.1 (2C), 20.7. HRMS (ESI) [M^+; calculated for $C_{23}H_{21}N$: 311.1674] found *m/z* 311.1674.



9

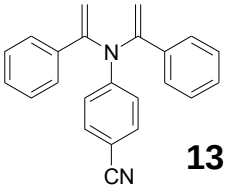
The reaction was run at 1 mmol scale. The crude was purified by column chromatography on basic alumina (0-3 % MeOH in AcOEt) to achieve 4-[bis-(1-

phenylvinyl)amino]-benzamide **9** as a yellowish orange solid (190 mg, 56 %). GC-MS (*m/z)*: 340 ($M^{+\cdot}$, 100 %), 312 (7 %), 223 (100 %). IR (cm^{-1}): 3353, 3207, 2921, 2855, 2304, 2200, 1806, 1708, 1494, 1371. ^{1}H NMR (δ, ppm; *J*, Hz): 7.48 (6H, mult), 7.20-7.17 (6H, mult), 6.97 (2H, dt, *J*=8.7, 1.9), 5.80 (2H, bs), 5.12 (2H, s), 4.81 (2H, s). ^{13}C NMR (δ, ppm): 169.0, 151.0 (2C), 150.3, 138.0 (2C), 128.3 (4C), 128.2 (2C), 128.1 (2C), 126.7 (4C), 126.5, 123.6 (2C), 108.2 (2C). HRMS (ESI) [$M+H^{+}$; calculated for $C_{23}H_{21}N_2O$: 341.1654] found *m/z* 341.1665.



11

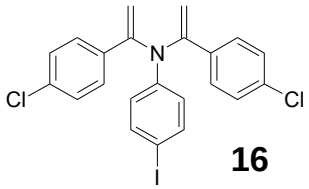
The reaction was run at 1 mmol scale. The crude was purified by column chromatography on basic alumina (1-5 % AcOEt in hexane) to achieve 3-{4-[bis-(1-phenylvinyl)amino]-phenyl}-acrylic acid ethyl ester **11** as a yellow oil (304 mg, 77 %). R_f (5 % AcOEt in hexane): 0.23. IR (cm^{-1}): 3057, 3032, 2977, 1706, 1624, 1600, 1505, 1315, 1265, 1171. ^{1}H NMR (δ, ppm; *J*, Hz): 7.61 (6H, mult), 7.29 (7H, mult), 7.06 (2H, dt, *J*=8.6, 2.1), 6.28 (1H, d, *J*=16.0), 5.20 (2H, s), 4.89 (2H, s), 4.26 (2H, q, *J*=7.1), 1.33 (3H, t, *J*=7.2). ^{13}C NMR (δ, ppm): 167.2, 151.0 (2C), 148.9, 144.2, 138.2 (2C), 128.6 (2C), 128.5, 128.3 (4C), 128.2 (2C), 126.7 (4C), 124.3 (2C), 116.1, 107.9 (2C), 60.2, 14.3. HRMS (ESI) [M^{+}; calculated for $C_{27}H_{26}NO_2$: 396.1964] found *m/z* 396.1955.



13

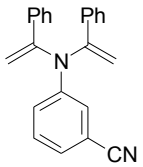
The reaction was run at 2 mmol scale. The crude was purified by column chromatography on basic alumina (0-10 % AcOEt in hexane) to achieve 4-[bis-(1-phenylvinyl)amino]-benzonitrile **13** as a yellow oil (152 mg, 47 %). The product was dissolved in the minimum amount of hexane and kept overnight in a fridge at -14 ºC to obtain yellow crystals, which were washed with cold *n*-hexane and dried. R_f (10 % AcOEt in hexane): 0.61 %. GC-MS (*m/z)*: 322 ($M^{+\cdot}$, 86 %), 294 (10 %), 245 (12 %),

205 (100 %). IR (cm^{-1}): 3062, 3024, 2918, 2852, 2221, 1600, 1500, 1317, 1206. ^{1}H NMR (CD_3CN, δ, ppm; *J*, Hz): 7.61 (4H, mult), 7.43 (2H, dt, *J*=9.0, 2.3), 7.31 (6H, mult), 7.09 (2H, dt, *J*=8.8, 2.3), 5.28 (2H, d, *J*=0.6), 4.95 (2H, d, *J*=0.6). ^{13}C NMR (CD_3CN, δ, ppm): 151.8 (2C), 151.7, 138.6 (2C), 133.6 (2C), 129.5 (2C), 129.3 (4C), 127.8 (4C), 124.7 (2C), 119.8, 109.7 (2C), 105.1. HRMS (ESI) [$M+H^+$; calculated for $C_{23}H_{19}N_2$: 323.15548] found *m/z* 323.1555.



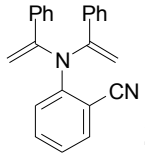
16

The reaction was run at 0.5 mmol scale (^{1}H-NMR yield, 58 %). The crude was purified by column chromatography on basic alumina (0-5 % AcOEt in hexane) to achieve bis-[1-(4-chlorophenyl)-vinyl]-(4-iodophenyl)-amine **16**. R_f (5 % AcOEt in hexane): 0.77. %). IR (cm^{-1}): ^{1}H NMR (ppm; *J*, Hz): 7.37 (4H, mult), 7.19 (4H, mult), 7.17 (2H, dt, *J*=8.8, 2.1), 6.71 (2H, dt, *J*=8.2, 2.1), 5.01 (2H, d, *J*=0.8), 4.64 (2H, d, *J*=0.8). ^{13}C NMR (δ, ppm): 150.0 (2C), 146.2, 137.8 (2C), 136.6 (2C), 134.0 (2C), 128.6 (4C), 127.9 (4C), 126.8 (2C), 107.6 (2C), 86.8.



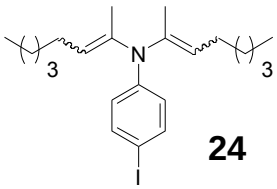
18

The reaction was run at 1 mmol scale. The crude was purified by column chromatography on basic alumina (0-15 % AcOEt in hexane) to achieve 3-[bis-(1-phenylvinyl)amino]-benzonitrile **18** as a yellow oil (306 mg, 95 %). R_f (10 % AcOEt in hexane): 0.52 %. IR (cm^{-1}): 3059, 3033, 2954, 2925, 2229, 1686, 1602, 1485, 1437, 1318, 1276. ^{1}H NMR (δ, ppm; *J*, Hz): 7.47 (4H, mult), 7.24-7.14 (8H, mult), 7.09 (1H, td, *J*=7.7, 0.5), 7.03 (1H, dt, *J*=7.6, 1.4), 5.10 (2H, s), 4.98 (2H, s). ^{13}C NMR (δ, ppm): 150.8 (2C), 147.6, 137.6 (2C), 129.4, 128.7, 128.4 (4C), 128.3, 127.4 (2C), 126.7 (4C), 125.8, 118.7, 112.6, 108.1 (2C). HRMS (ESI) [M^+; calculated for $C_{23}H_{19}N_2$: 323.1548] found *m/z* 323.1553.



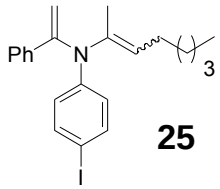
20

The reaction was run at 0.25 mmol scale for 48 h, using 20 mol% of **7c** (34 mg) as catalyst and 8 eq. of phenylacetylene **2** (220 μL). The crude was analysed by GC, GC-MS and ^{1}H, ^{13}C-NMR and DEPT. GC-MS (*m/z)*: 322 ($M^{+\cdot}$, 100 %), 294 (5 %), 245 (10 %), 205 (68 %).



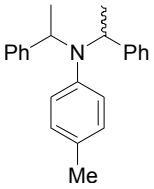
24

The reaction was run at 1 mmol scale. After 24 h, hexane (10-20 mL) was added and precipitated solid was filtered off. After removal of the volatiles under vacuum, **24** was obtained as an orangish red oil (417 mg, 95 %). GC-MS (*m/z*, three different peaks, major isomer shown here*)*: 439 ($M^{+\cdot}$, 12 %), 382 (40 %), 368 (100 %), 354 (33 %), 298 (29 %), 244 (79 %). ^{1}H- and ^{13}C NMR: see spectra.



25

The reaction was run at 1 mmol scale. The first imine with phenylacetylene **2** (121 μL, 1.1 eq.) was formed at 60 ºC for 24 h, using **7b** (9 mg, 1 mol%) as catalyst. Then, additional catalyst (36 mg, 4 mol%) was added together with 1-octyne (444 μL, 3 eq.) and the mixture was reacted at 60 ºC for 24 h, and analysed by GC-MS. After the reaction was completed, volatiles were removed under vacuum and hexane (10-20 mL) was added to precipitate the catalyst. The solid was filtered off and, after removal of the solvents, an orange solid, corresponding to the aromatic imine, was recovered (225 mg, 70 % yield). GC-MS of **25** (*m/z*, two peaks, major isomer shown here*)*: 431 ($M^{+\cdot}$, 46 %), 416 (48 %), 374 (100 %), 354 (13 %), 298 (6 %), 244 (31 %).

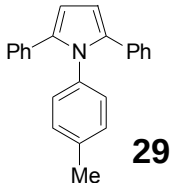


26 and **27**

As crystals have not been obtained yet, the characterisation is assigned by the different R_f's on silica TLC:

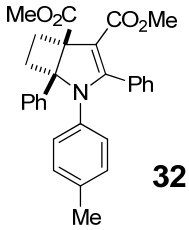
Upper: White oil. R_f (10 % AcOEt in hexane): 0.73. GC-MS (*m/z)*: 315 ($M^{+\cdot}$, 100 %), 300 (100 %), 211 (33 %), 196 (100 %), 134 (22 %), 105 (100 %). ^{1}H NMR (δ, ppm; *J*, Hz): 7.22-7.12 (10H, mult), 6.89 (2H, dd, *J*=8.6, 0.6), 6.60 (2H, d, *J*=8.3), 4.38 (2H, q, *J*=6.8), 2.21 (3H, s), 1.23 (6H, d, *J*=6.8). ^{13}C NMR (δ, ppm): 143.6 (2C), 143.0, 131.8, 128.3 (2C), 128.0 (4C), 128.8 (4C), 126.7 (2C), 126.3 (2C), 57.4 (2C), 20.8 (2C), 20.7.

Down: White oil. R_f (10 % AcOEt in hexane): 0.72. GC-MS (*m/z)*: same as above. ^{1}H NMR (δ, ppm; *J*, Hz): 7.30-7.12 (10H, mult), 6.83 (2H, dd, *J*=8.6, 0.5), 6.59 (2H, d, *J*=8.7), 4.64 (2H, q, *J*=6.8), 2.15 (3H, s), 1.31 (6H, d, *J*=6.8). ^{13}C NMR (δ, ppm): 144.7 (2C), 144.0, 129.6, 128.6 (2C), 128.1 (4C), 127.5 (4C), 126.6 (2C), 122.8 (2C), 56.8 (2C), 20.5, 18.3 (2C).



29

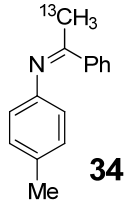
See ref. S4. GC-MS (*m/z)*: 309 ($M^{+\cdot}$, 100 %), 191 (26 %). ^{1}H NMR (δ, ppm; *J*, Hz): 7.60 (4H, dd, *J*=8.4, 1.3), 7.37 (4H, tt, *J*=8.1, 1.8), 7.22 (2H, d, *J*=5.0), 7.21 (2H, tt, *J*=8.7, 2.1), 7.12 (2H, d, *J*=4.7), 6.73 (2H, d, *J*=2.1), 2.37 (3H, s). ^{13}C NMR (δ, ppm): 137.8, 136.6, 135.2, 134.7, 132.8, 129.6 (2C), 128.7 (2C), 128.3 (2C), 128.1 (2C), 126.5, 125.8, 125.5 (2C), 125.3, 125.1 (2C), 120.9, 108.4, 21.0.



32

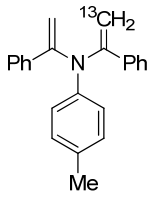
(1,5)-dimethyl-1,3-diphenyl-2-(*p*-tolyl)-2-azabicyclo[3.2.0]hept-3-ene-4,5-dicarboxylate (**32**). Rf (20 % AcOEt/Hexane): 0.24. GC-MS (*m/z)*: 453 ($M^{+\cdot}$, 3 %), 425

(100 %), 394 (39 %), 194 (17 %). ^{1}H NMR (δ, ppm; *J*, Hz): 7.46 (2H, dd, *J*=8.5, 1.7), 7.40 (2H, mult), 7.31 (4H, mult), 7.22 (2H, mult), 6.67 (2H, dd, *J*=8.6, 0.7), 6.37 (2H, dt, *J*=8.5, 2.1), 3.52 (3H, s), 3.24 (3H, s), 3.21-2.96 (3H, mult), 2.45 (1H, mult), 2.07 (3H, s). ^{13}C NMR (δ, ppm): 171.3, 161.8, 137.0, 136.4, 136.3, 133.8, 131.7, 129.6, 129.0, 128.8 (2C), 128.6, 128.0 (2C), 127.8, 127.7 (2C), 127.4, 127.0, 125.2 (2C), 105.7, 77.6, 62.1, 51.5, 50.5, 29.2, 27.6, 20.7. HRMS (ESI) [M+H^{+}; calculated for $C_{29}H_{28}NO_4$: 454.2018] found *m/z* 454.1956.



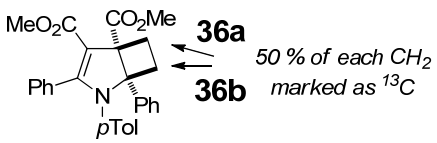
34

(*Z*)-4-methyl-*N*-(1-^{13}C-methyl-benzylidene)aniline (**34**). ^{1}H NMR (δ, ppm; *J*, Hz): 7.99-7.94 (2H, m), 7.53-7.43 (3H, m), 7.19 (2H, d, *J*=8.1), 6.73 (2H, d, *J*=8.1), 2.33 (3H, s), 2.21 (3H, d, $J^{1}_{C\text{-}H}$=128.3); enamine (~ 17 %): 5.03 (1H, d, $J^{1}_{C\text{-}H}$=130.0), 4.50 (1H, d, $J^{1}_{C\text{-}H}$=130.0), 3.55-3.25 (1H, bs). ^{13}C NMR (δ, ppm): 140.5, 133.3 (1C, d, $J^{1}_{C\text{-}C}$= 85), 131.4, 130.4 (2C), 129.5 (4C), 128.0 (2C), 120.4, 26.9, 18.1; enamine: 106.5.

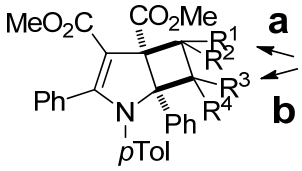


35

4-methyl-*N*,*N*-bis(1-phenylvinyl)aniline, ^{13}C-marked at one terminal vinyl carbon (**35**). ^{1}H NMR (δ, ppm; *J*, Hz): 8.04-7.93 (2H, m), 7.71-7.67 (2H, m), 7.57-7.51 (2H, m), 7.43-7.21 (4H, m), 7.06 (2H, d, *J*=9), 7.01 (2H, d, *J*= 9), 5.03 (1H,s), 5.02 (1H, d, $J^{1}_{C\text{-}H}$=159.7), 4.62 (1H,s), 4.61 (1H, d, $J^{1}_{C\text{-}H}$=158.8), 2.22 (3H, s).



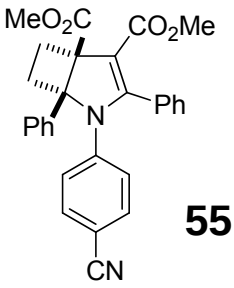
^{13}C-marked, (1,5)-dimethyl-1,3-diphenyl-2-(*p*-tolyl)-2-azabicyclo[3.2.0]hept-3-ene-4,5-dicarboxylates (**36a,b)**. GC-MS (*m/z)*: 454 ($M^{+\cdot}$, 4 %), 425 (100 %), 394 (57 %), 194 (18 %). ^{1}H NMR (δ, ppm; *J*, Hz): 7.439 (2H, dd, *J*=8.4, 1.5), 7.34-7.12 (8H, mult), 6.59 (2H, dd, *J*=8.4, 0.6), 6.30 (2H, dt, *J*=8.4, 1.9), 3.44 (3H, s), 3.18 (3H, s), 3.40-2.00 (4H, mult), 1.99 (3H, s). ^{13}C NMR (δ, ppm): 171.2, 161.8, 137.0, 136.4, 133.8, 133.5 131.7, 130.7, 129.6, 129.0, 128.8 (2C), 128.0 (2C), 127.8, 127.7 (2C), 127.4, 126.6, 125.2 (2C), 105.7, 77.6 (50 % d, $J^{1}_{C\text{-}H}$= 33.0, 50 % d, $J^{2}_{C\text{-}H}$= 7.1), 62.2 (50 % d, $J^{1}_{C\text{-}H}$= 34.0, 50 % d, $J^{2}_{C\text{-}H}$= 6.6), 51.5, 50.4, 29.2 (2 % $J^{1}_{C\text{-}H}$= 28.5), 27.6 (2 % $J^{1}_{C\text{-}H}$= 28.5), 20.6.



50 % of each CH_2 marked as ^{13}C

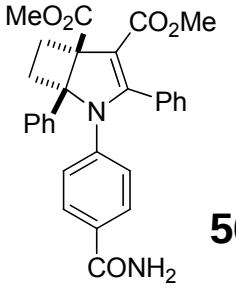
R^2=R^3=R^4= H; R^1= D **42a,b**
R^1=R^3=R^4= H; R^2= D **43a,b**
R^1=R^2=R^4= H; R^3= D **44a,b**
R^1=R^2=R^3= H; R^4= D **45a,b**

^{13}C-marked and ^{2}H-marked, (1,5)-dimethyl-1,3-diphenyl-2-(*p*-tolyl)-2-azabicyclo[3.2.0]hept-3-ene-4,5-dicarboxylates (**42-45a,b**). GC-MS (*m/z)*: 455 ($M^{+\cdot}$, 3 %), 425 (100 %), 394 (54 %), 194 (18 %).



55

(1,5)-dimethyl-1,3-diphenyl-2-(*p*-benzonitril)-2-azabicyclo[3.2.0]hept-3-ene-4,5-dicarboxylate (**55**). Rf (25 % AcOEt/Hexane): 0.21. ^{1}H NMR (δ, ppm; *J*, Hz): 7.44-7.35 (2H, m), 7.10 (2H, d, *J*=9), 6.39 (2H, d, *J*=9), 3.53 (3H, s), 3.24 (3H, s), 3.18 (2H, m), 2.98 (1H, m), 2.47 (1H, m). ^{13}C NMR (δ, ppm):170.5, 165.0, 159.0, 143.1, 135.8, 132.1 (2C), 130.9, 130.7, 129.7 (2C), 129.4, 128.5 (2C), 128.3 (2C), 128.2, 127.0 (2C), 123.5 (2C), 118.7, 109.5, 105.8, 62.2, 51.7, 50.8, 29.7, 27.3. HRMS (ESI) [M+H^{+}; calculated for $C_{29}H_{25}N_2O_4$: 465.1814] found *m/z* 465.1802.



56

(1,5)-dimethyl-1,3-diphenyl-2-(*p*-benzamidyl)-2-azabicyclo[3.2.0]hept-3-ene-4,5-dicarboxylate (**56**). Rf (75 % AcOEt/Hexane): 0.23. ^{1}H NMR (δ, ppm; *J*, Hz):7.60-7.10 (12H, m), 6.35 (2H, d, *J*=9), 5.85-5.15 (2H, bs), 3.90-3.40 (3H, m), 3.52 (3H, s), 3.24 (3H, s), 3.15 (1H, m). ^{13}C NMR (δ, ppm): 171.1, 168.4, 165.2, 159.9, 142.5, 136.2, 131.2, 129.5, 129.4, 128.3 (2C), 128.1 (4C), 128.0, 127.6, 127.5 (2C), 127.2 (2C), 123.6 (2C), 108.3, 62.2, 51.7, 50.7, 29.7, 27.5. HRMS (ESI) [M+H^{+}; calculated for $C_{29}H_{27}N_2O_5$: 483.1920] found *m/z* 483.1879.

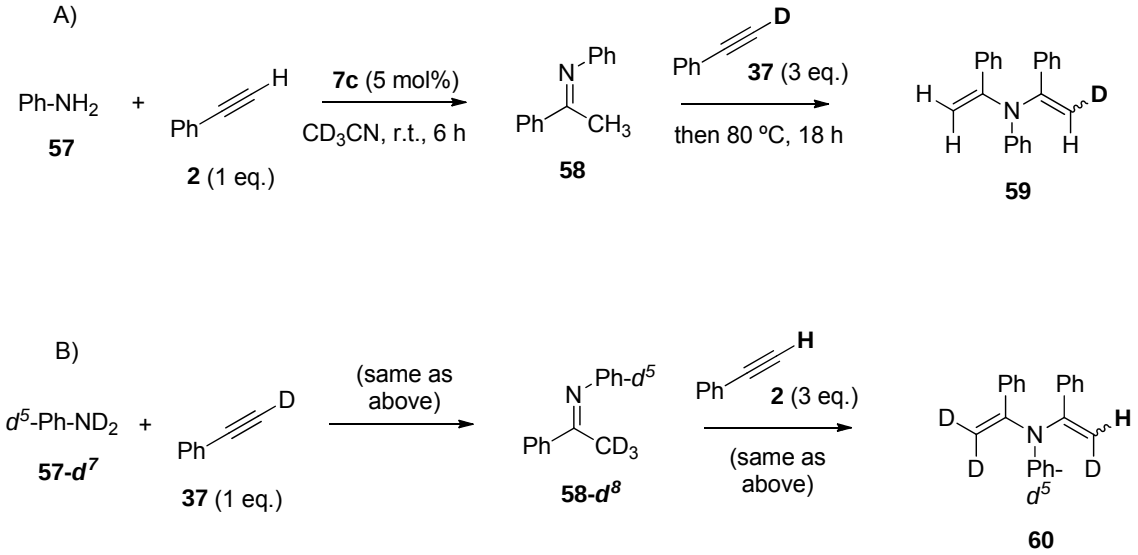
References.

S1 Leyva, A.; Corma, A. *J. Org. Chem.* **2009,** *74(5)*, 2067.

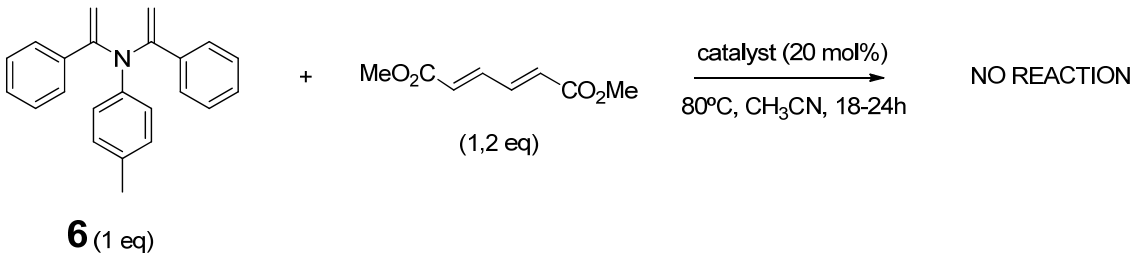
S2 Mezailles, N.; Ricard, L.; Gagosz, F. *Org. Lett.* **2005,** *7(19)*, 4133.

S3 Nandi, M.; Jin, J.; RajanBabu, T. V. *J. Am. Chem. Soc.* **1999,** *121*, 9899.

S4 Huerta, G.; Fomina, L.; Rumsh, L.; Zolotukhin, M. G. *Polymer Bull.* **2006**, *57*, 433.

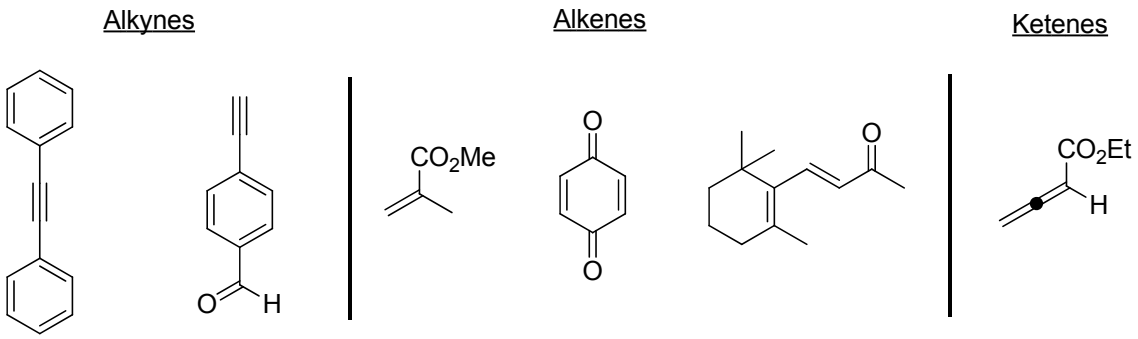


Scheme S1. Isotopic experiments: Scheme A) imine **58** as hydroaminating agent of deuterated phenylacetylene **37** and Scheme B) deuterated imine **58-*d*^{8}** as hydroaminating agent of phenylacetylene **2**, both under standard reaction conditions and using **7c** as catalyst. Deuterium incorporation was quantitative in all cases.



Catalysts tested: $FeCl_3$+3$AgNTf_2$, $AgNTf_2$, AgOTf, $La(OTf)_3$, $CuCl_2$, $(CuOTf)_2.C_6H_6$, $Cu(OTf)_2$, $CoCl_2$+2$AgNTf_2$, $Fe(Cp)_2$, $RhCl_3$, $PdCl_2$, $Pd(PPh_3)_4$.

Other electrophiles tested (CH_3CN, 80 ºC or DMF, 140 ºC):



Scheme S2. Failed attempts of addition of bisenamine **6** to different Michael-type acceptors.

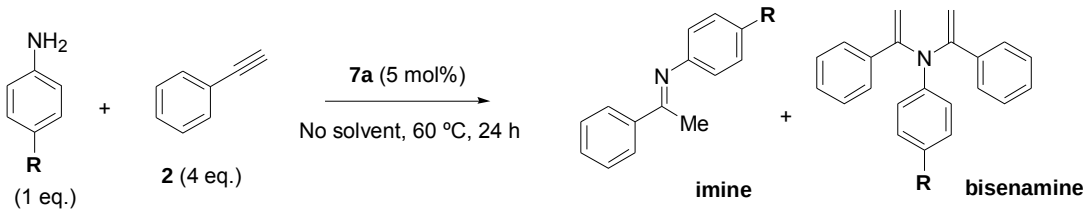
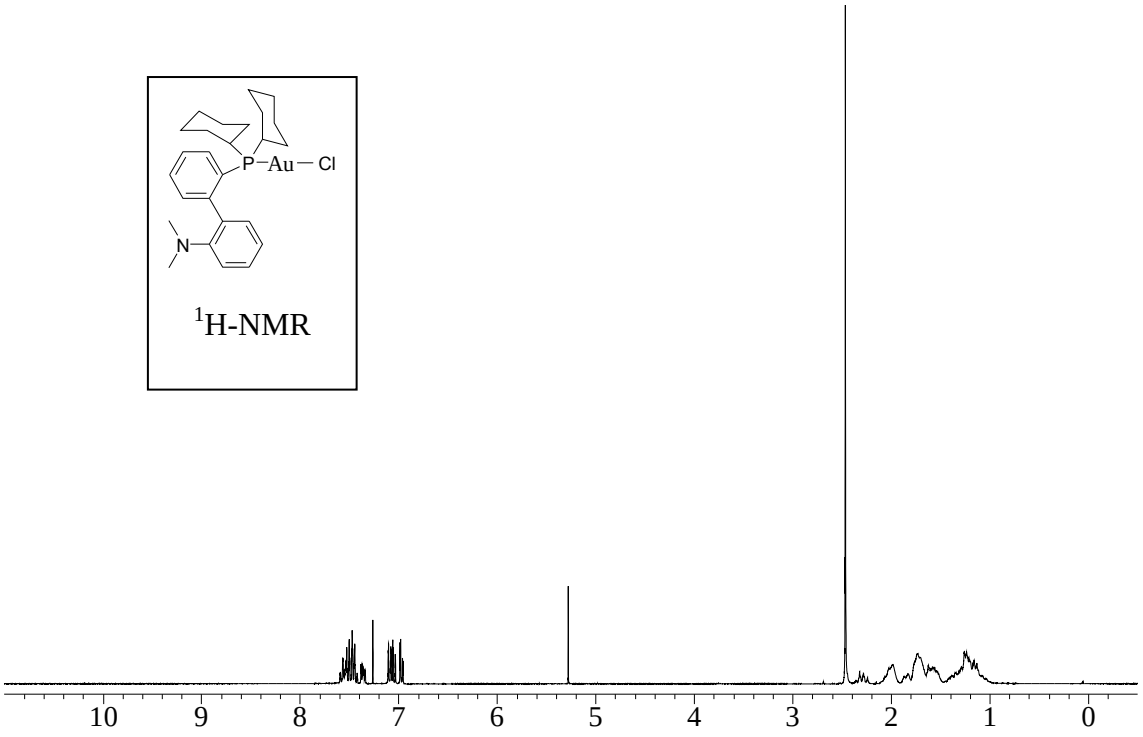
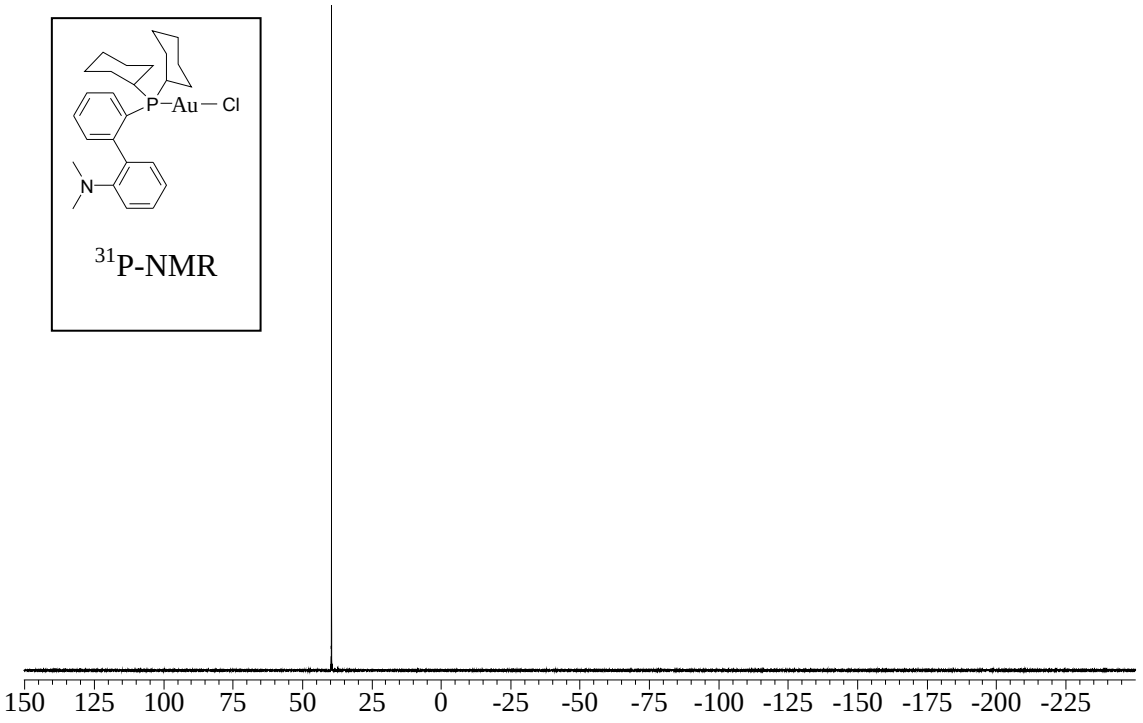


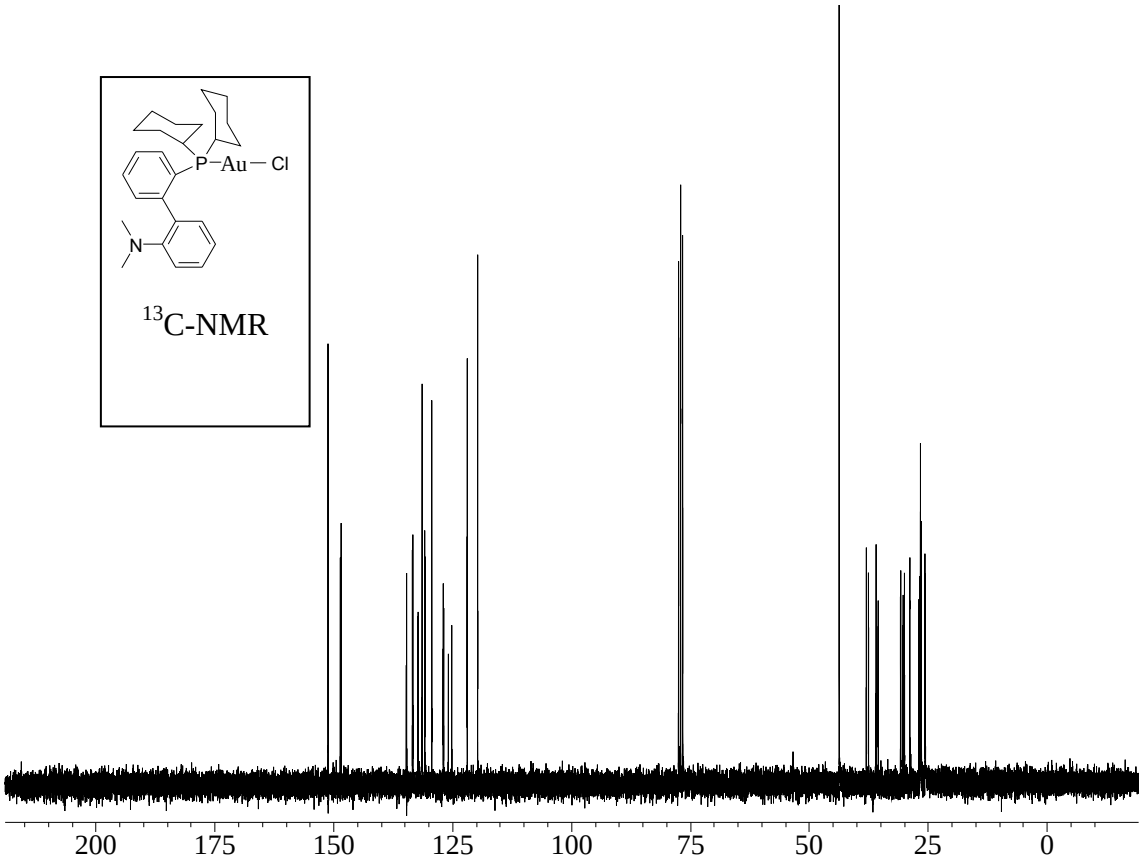
Table S1. Influence of the *p*-substitution of the aniline ring on the catalytic activity.

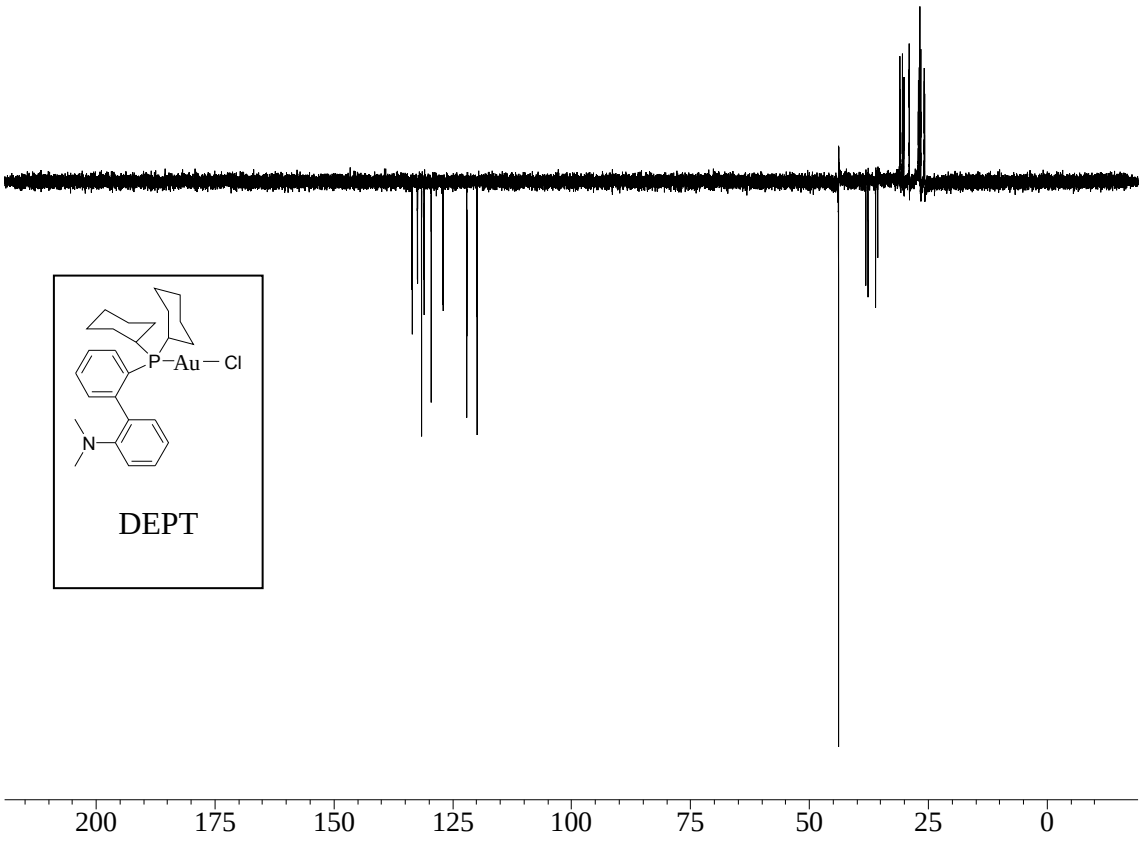
Entry	R	Conversion (%)[a]	Ratio Imine/Bisenamime
1	-CH=CH-COOEt **10**	> 95	1/20
2	$-CONH_2$ **8**	> 95	1/10
3	$-CH_3$ **1**	> 95	1/10
4	-OH **61**	> 95	3/1

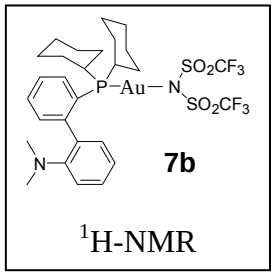
[a] GC conversion, minor by-products were found in some cases.


P–Au — Cl
N
^{1}H-NMR
10
9
8
7
6
5
4
3
2
1
0


P–Au — Cl
N
^{31}P-NMR
150
125
100
75
50
25
0
-25
-50
-75
-100
-125
-150
-175
-200
-225

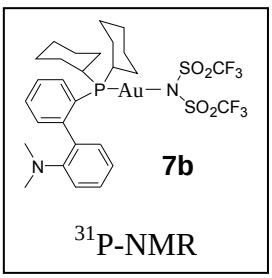

P–Au — Cl
N
13C-NMR
200
175
150
125
100
75
50
25
0


P–Au — Cl
N
DEPT
200
175
150
125
100
75
50
25
0



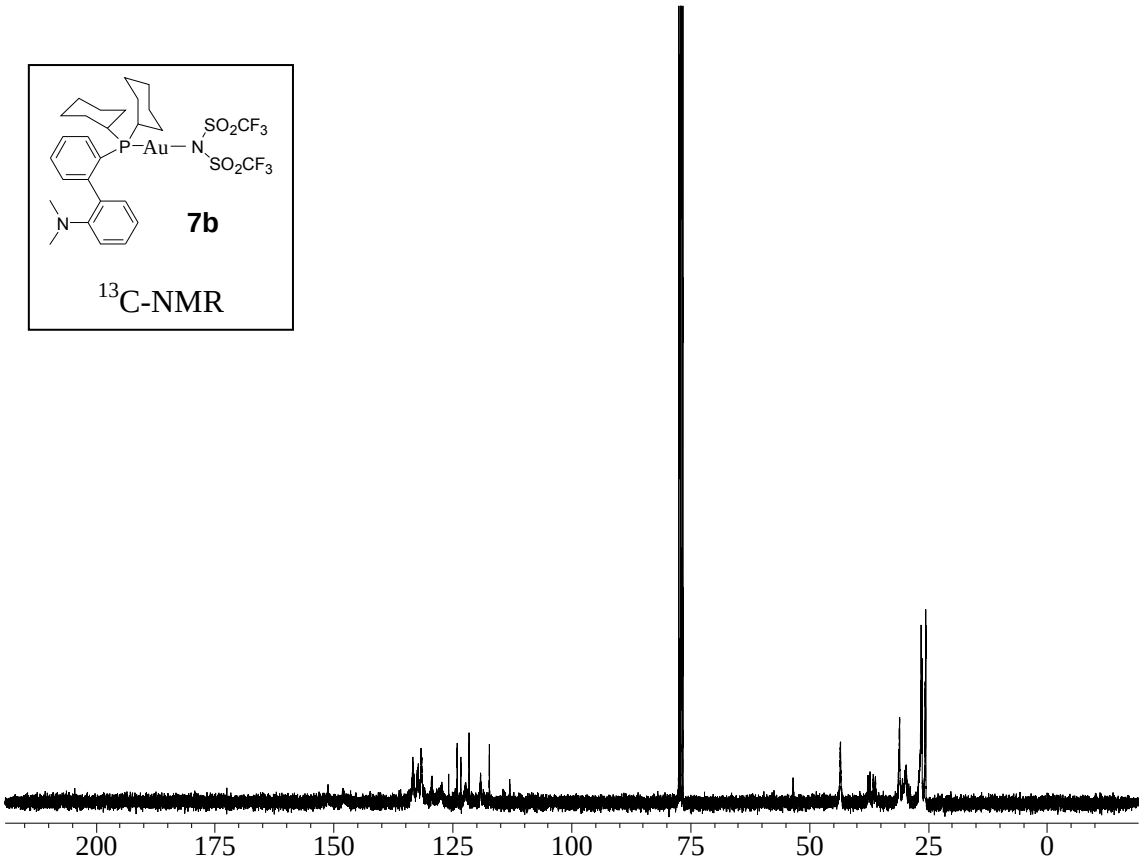
7b

^{1}H-NMR



7b

^{31}P-NMR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SO2CF3
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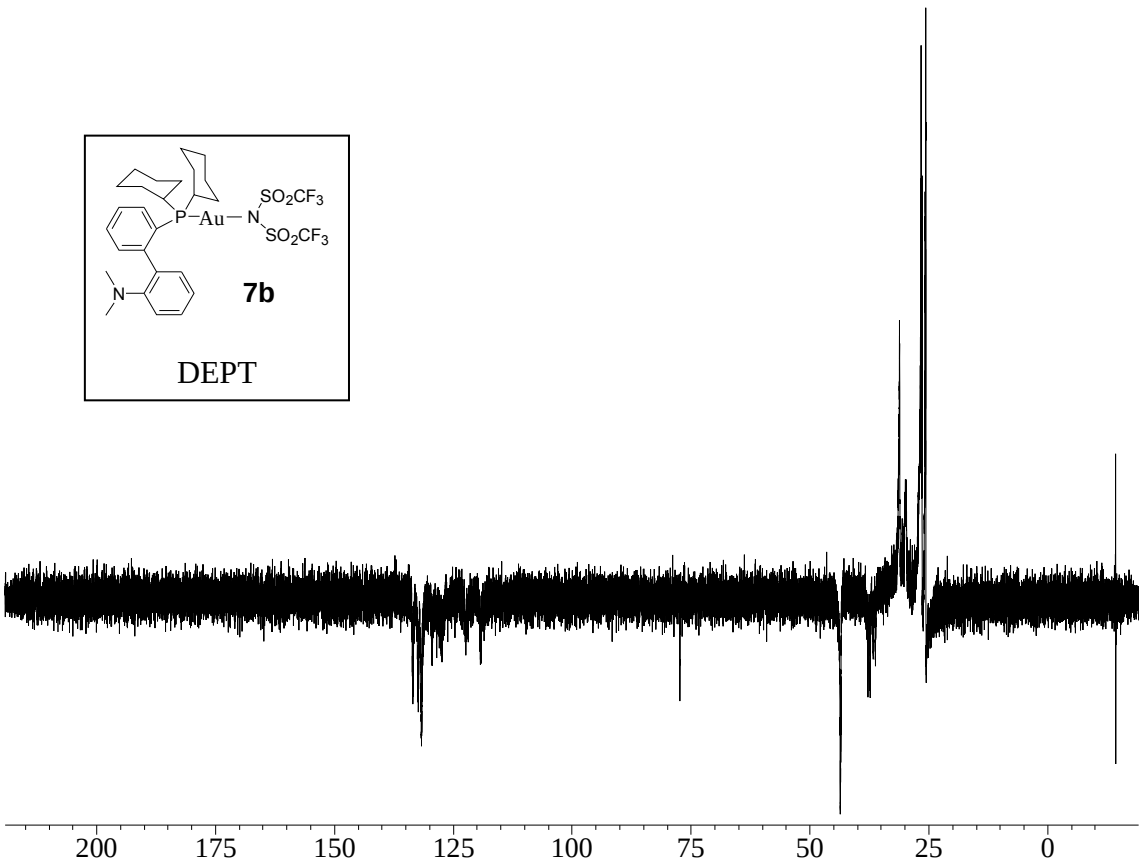
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P–Au–N
SO2CF3
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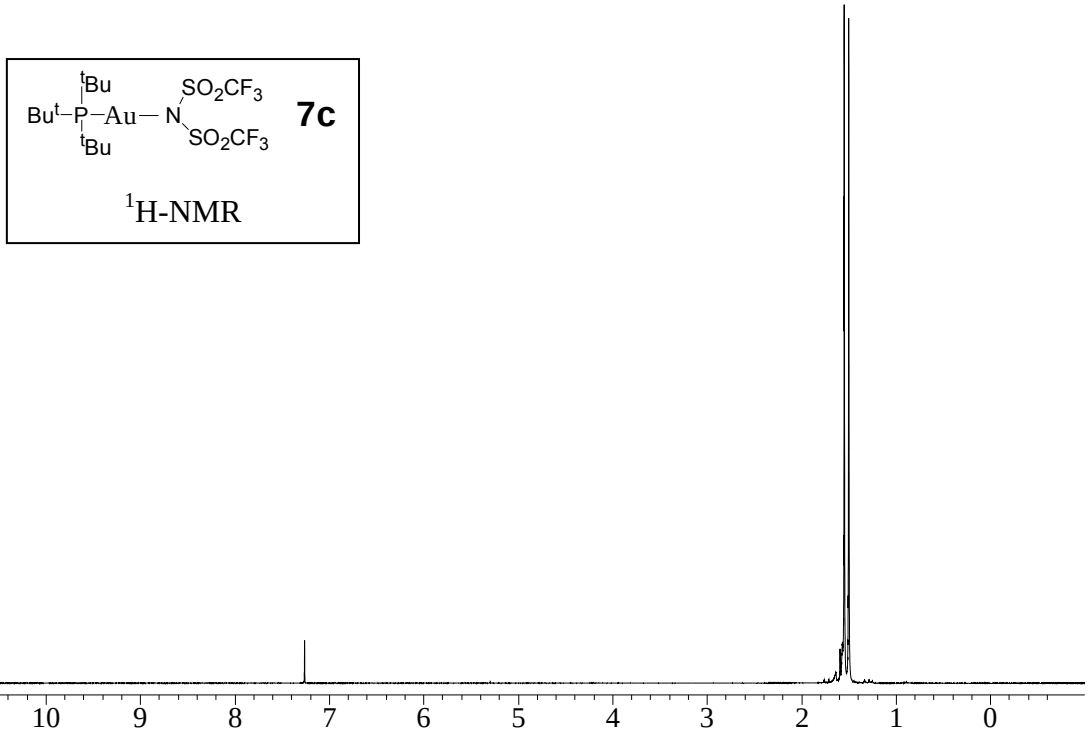
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SO2CF3
But–P–Au–N
SO2CF3
tBu
7c
1H-NMR
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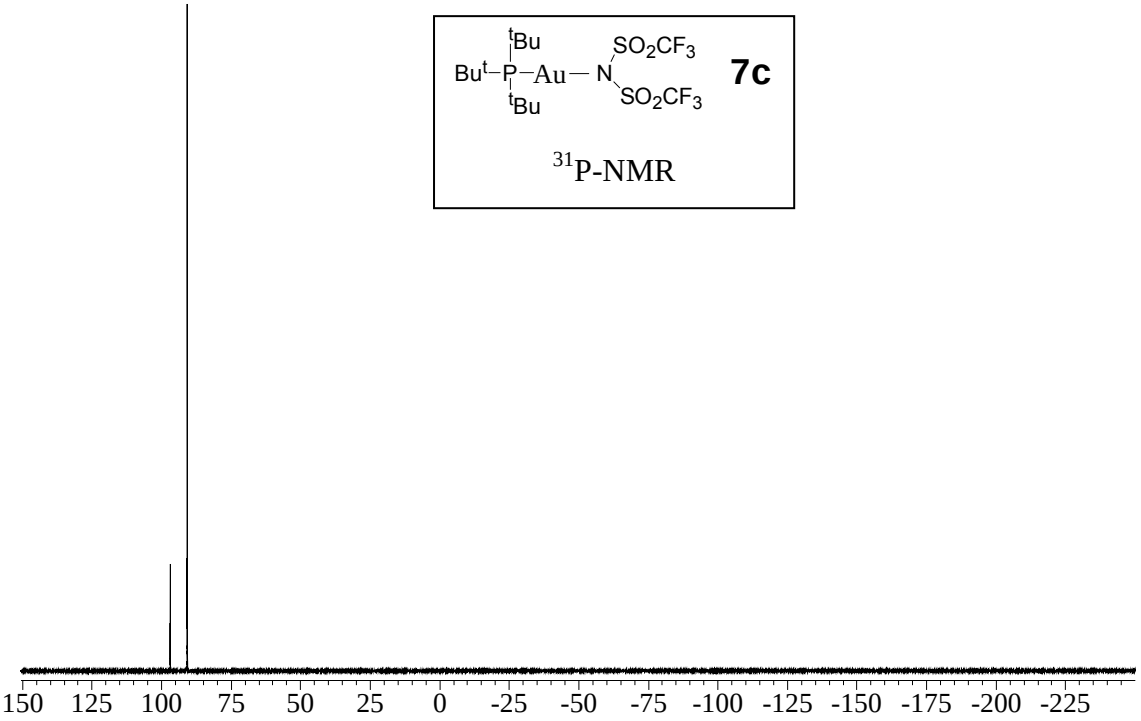
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But–P–Au–N
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tBu
7c
31P-NMR
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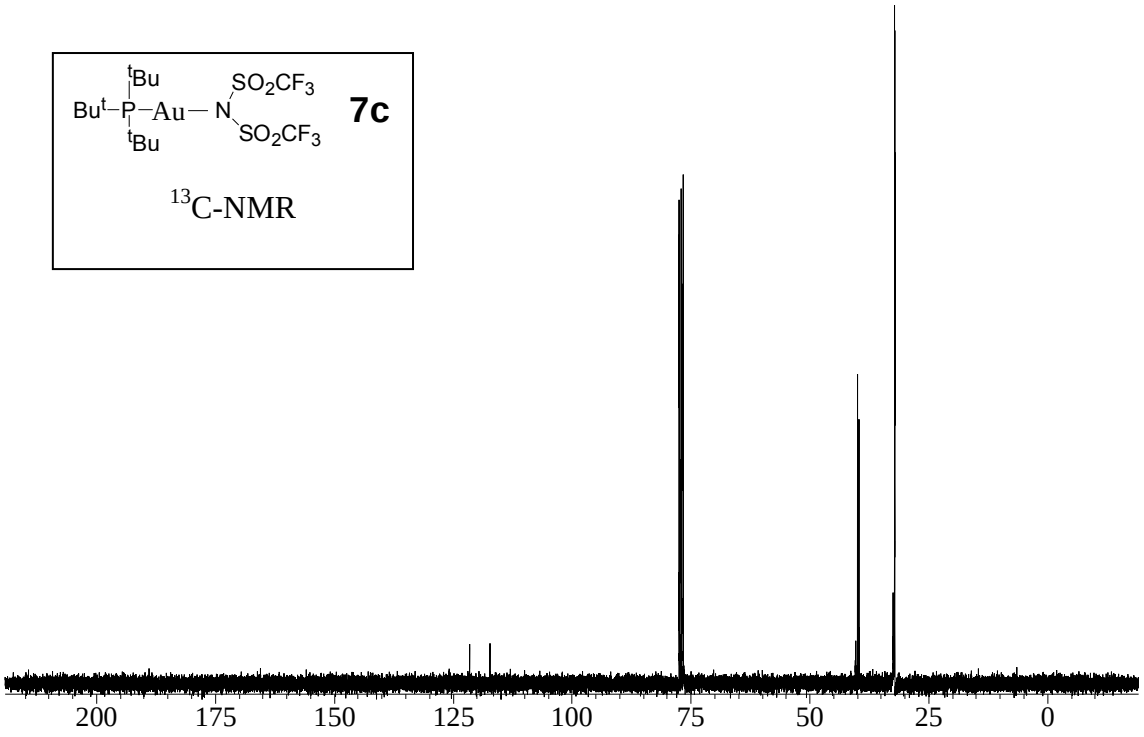
tBu
SO2CF3
Bu t–P–Au– N
tBu
SO2CF3
7c
13C-NMR
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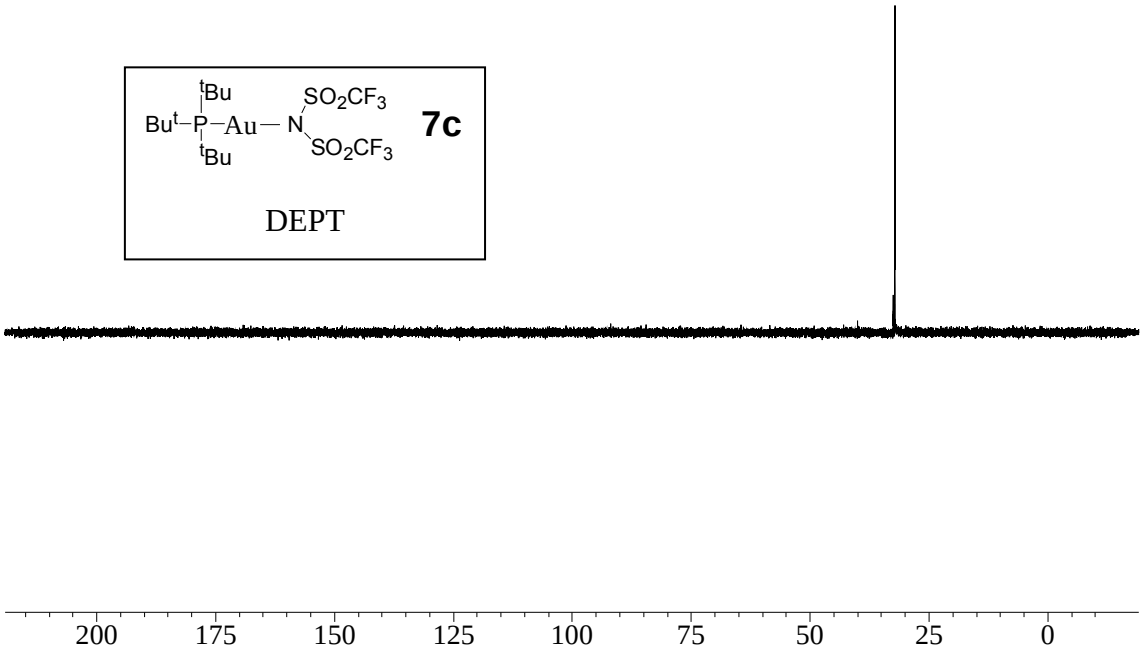
tBu
SO2CF3
Bu t–P–Au– N
tBu
SO2CF3
7c
DEPT
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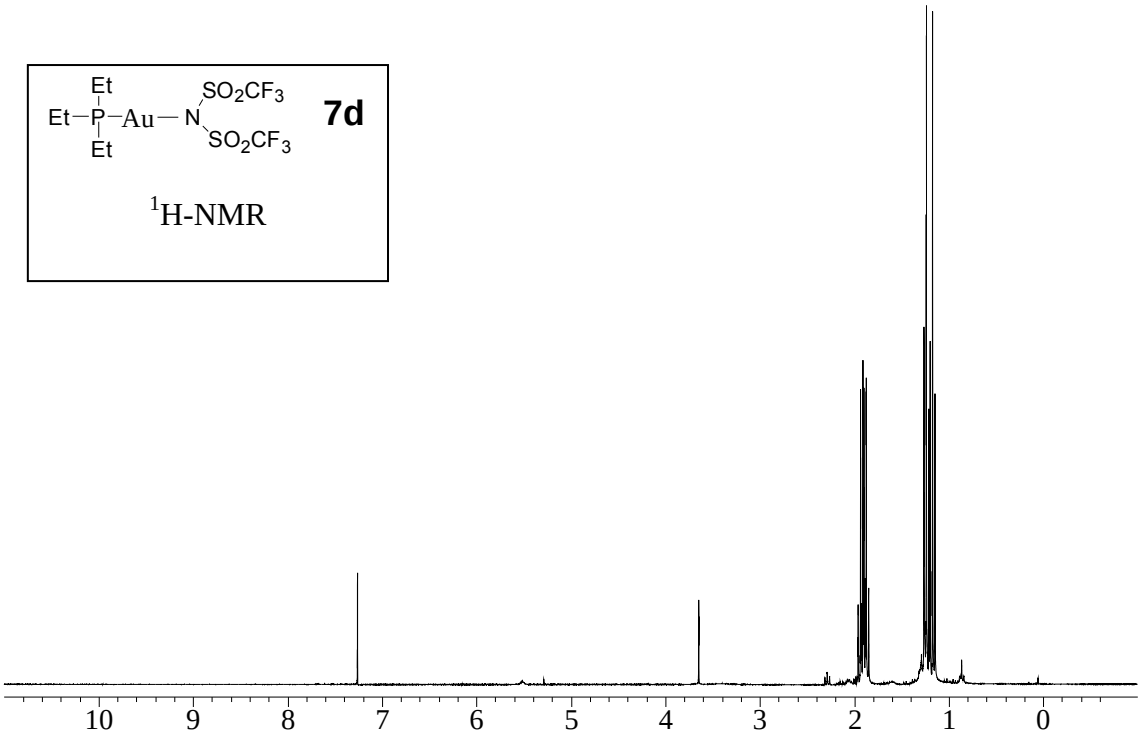
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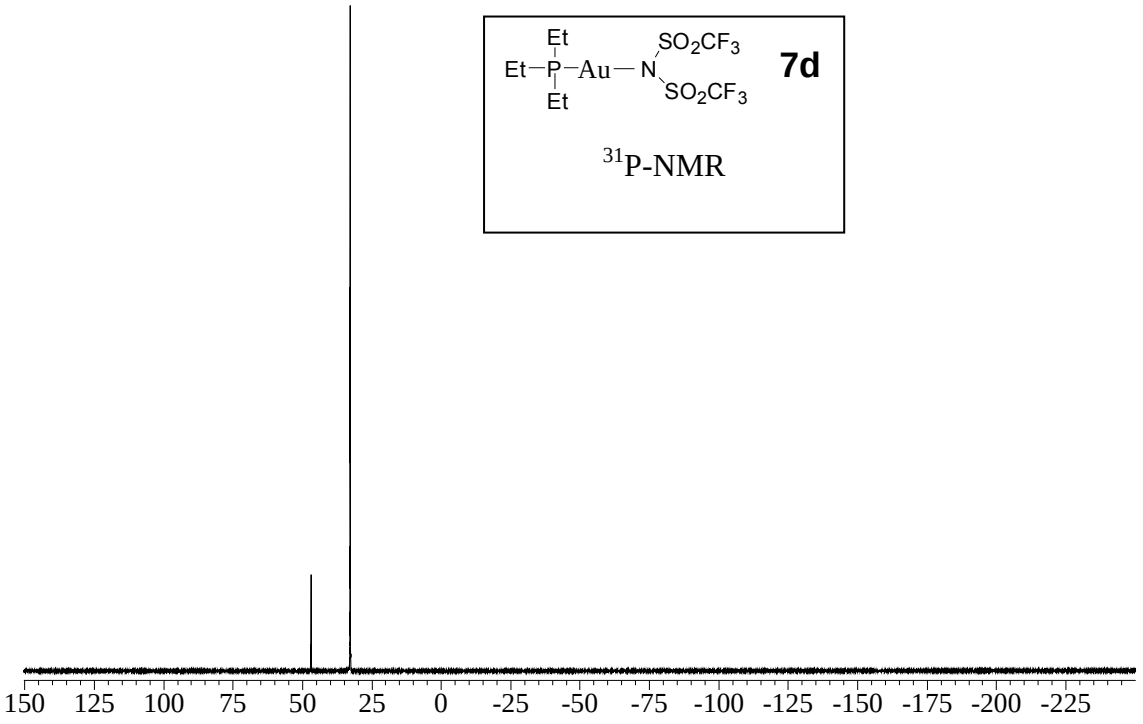
Et
SO2CF3
SO2CF3
7d
1H-NMR
10 9 8 7 6 5 4 3 2 1 0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31P-NMR
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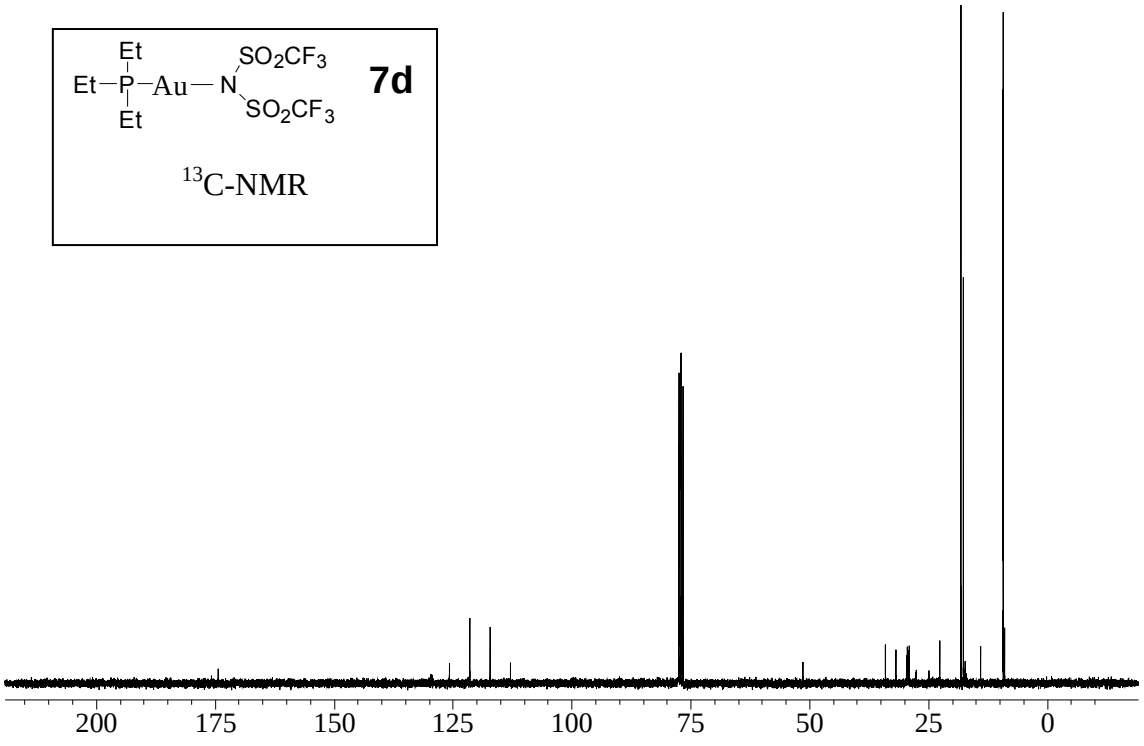
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7d
13C-NMR
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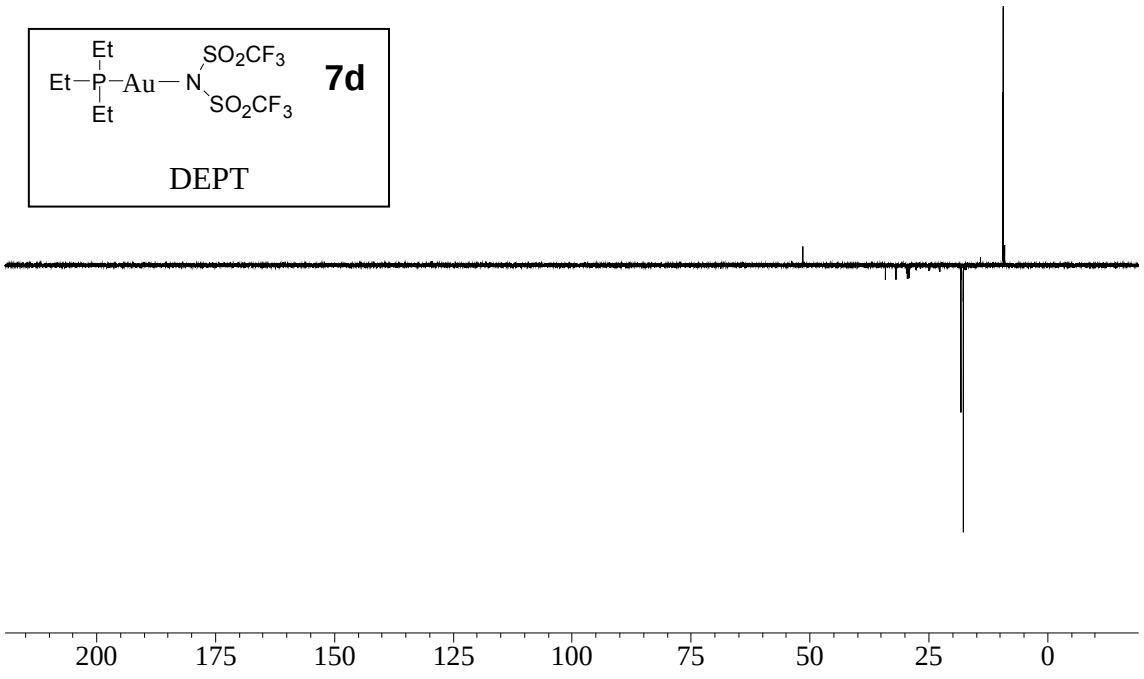
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Et—P—Au—N
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SO2CF3
SO2CF3
7d
DEPT
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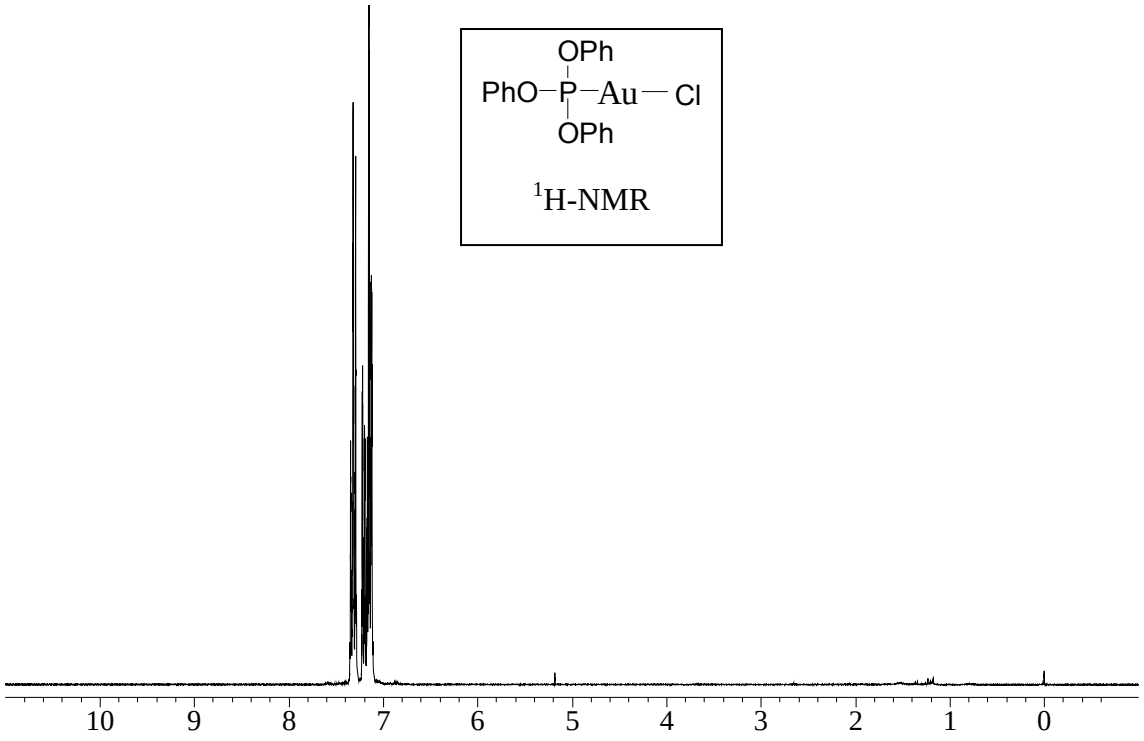
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1H-NMR
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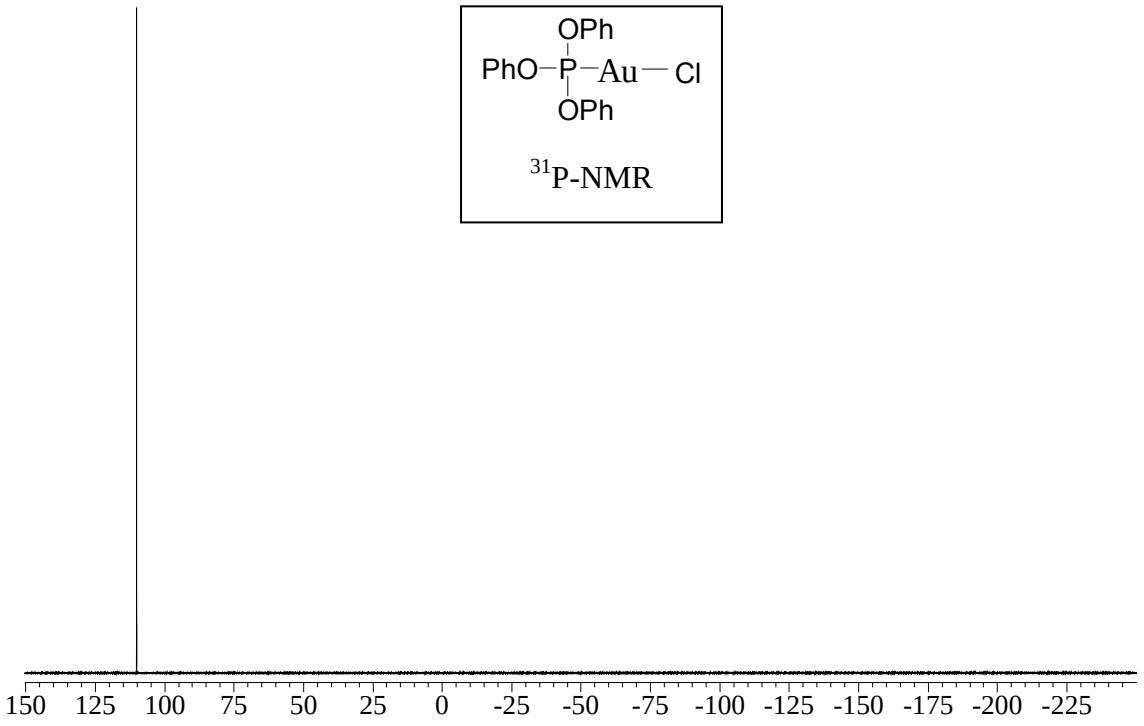
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PhO–P–Au–Cl
OPh
31P-NMR
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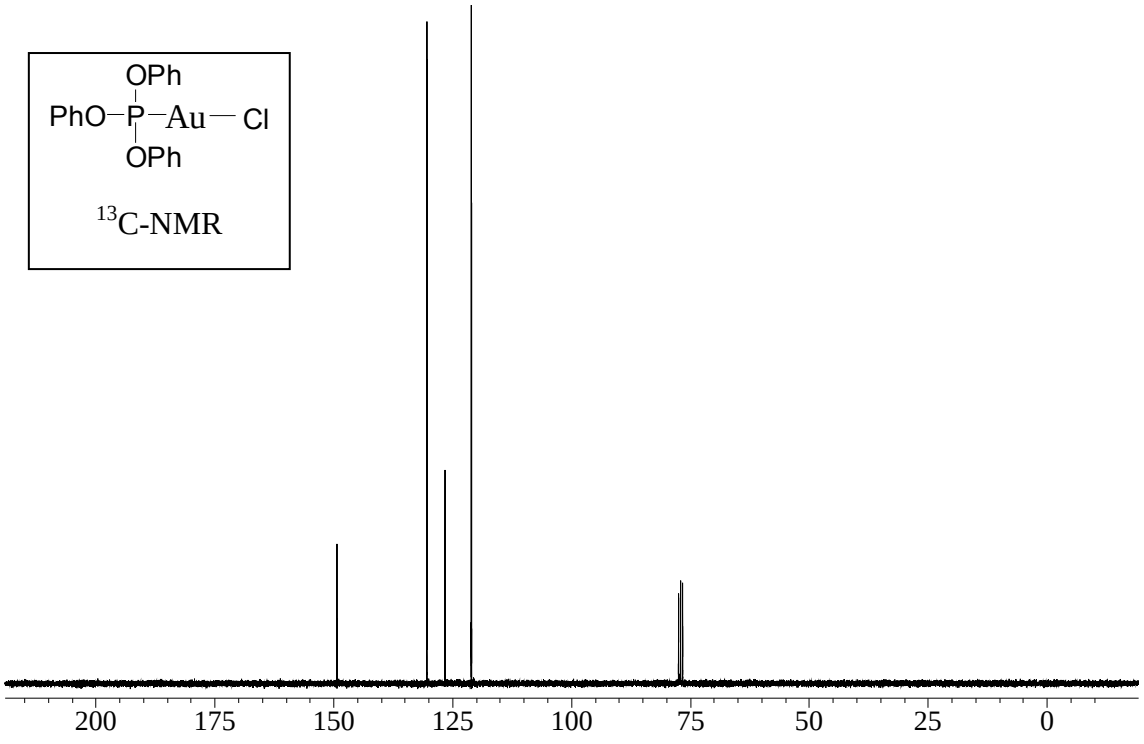
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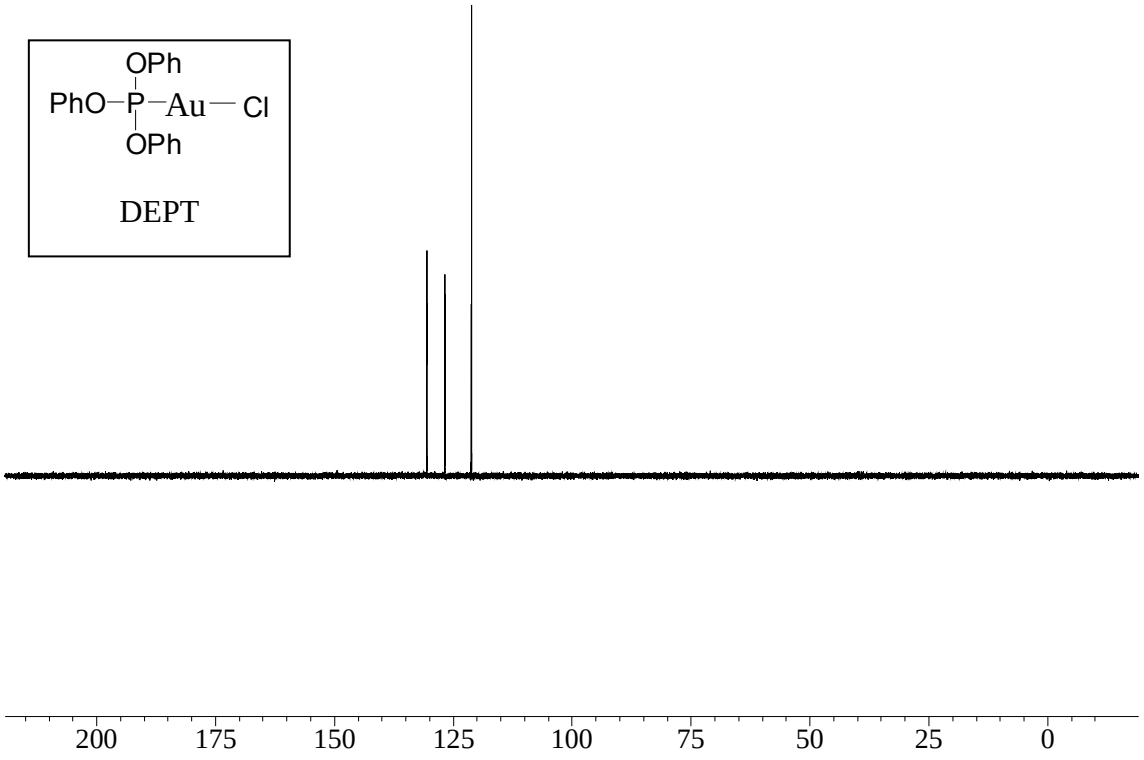
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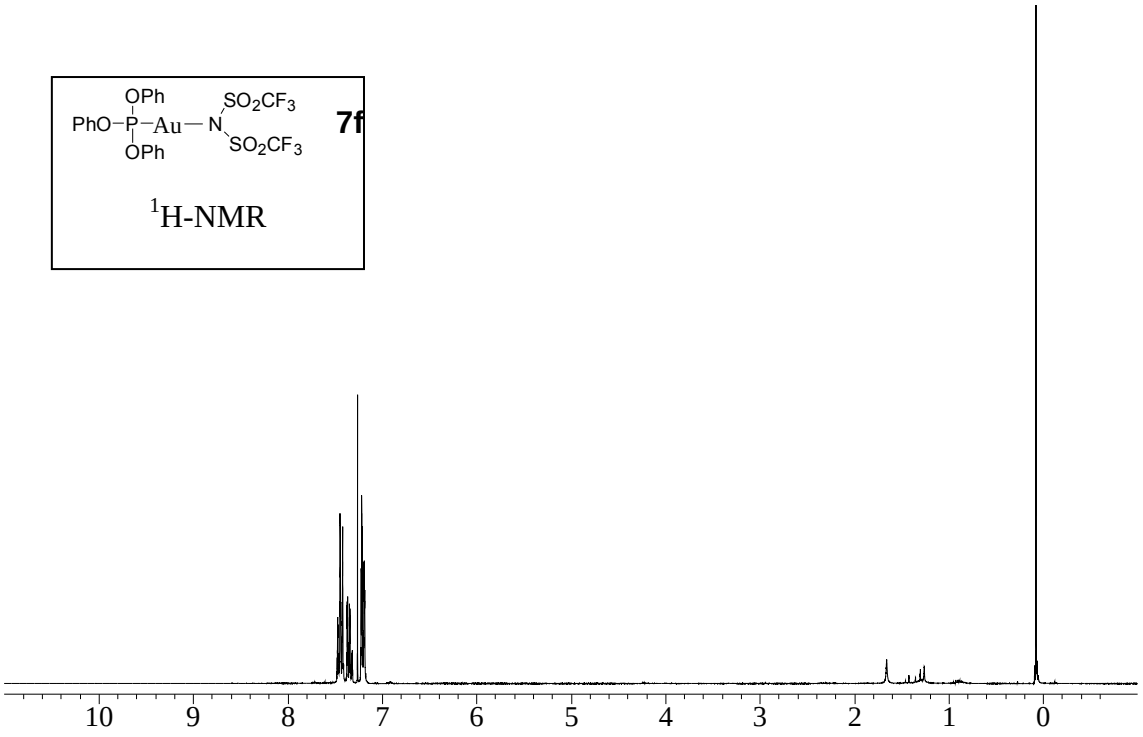
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PhO–P–Au–N
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SO2CF3
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1H-NMR
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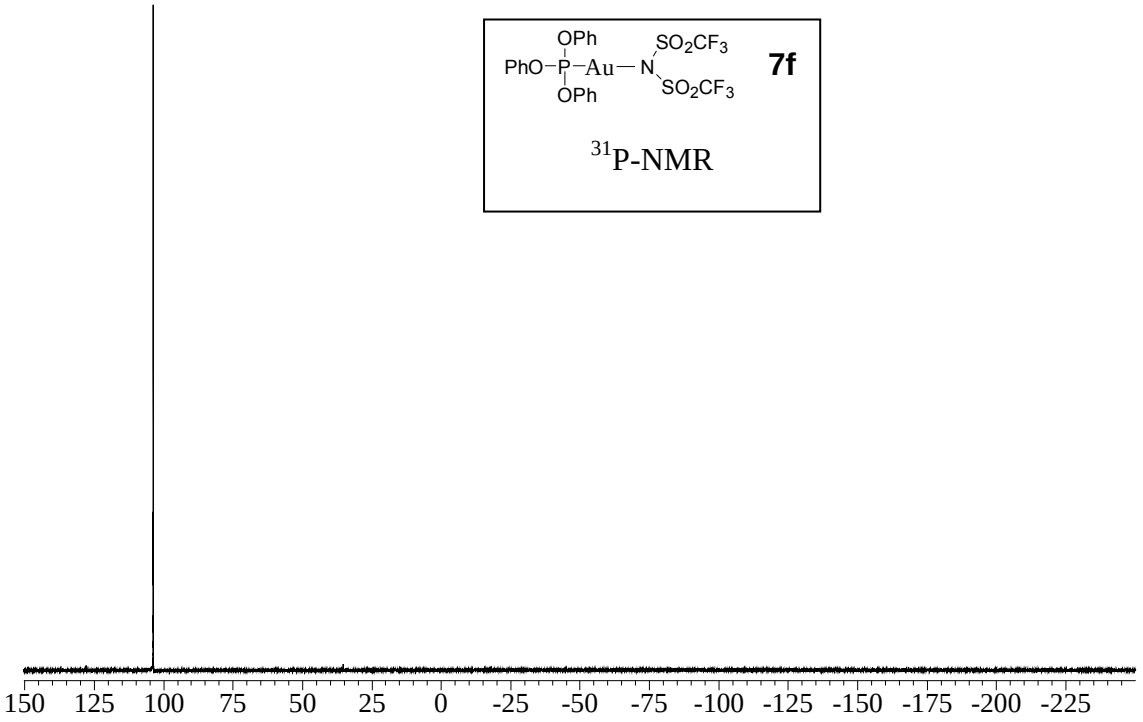
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SO2CF3
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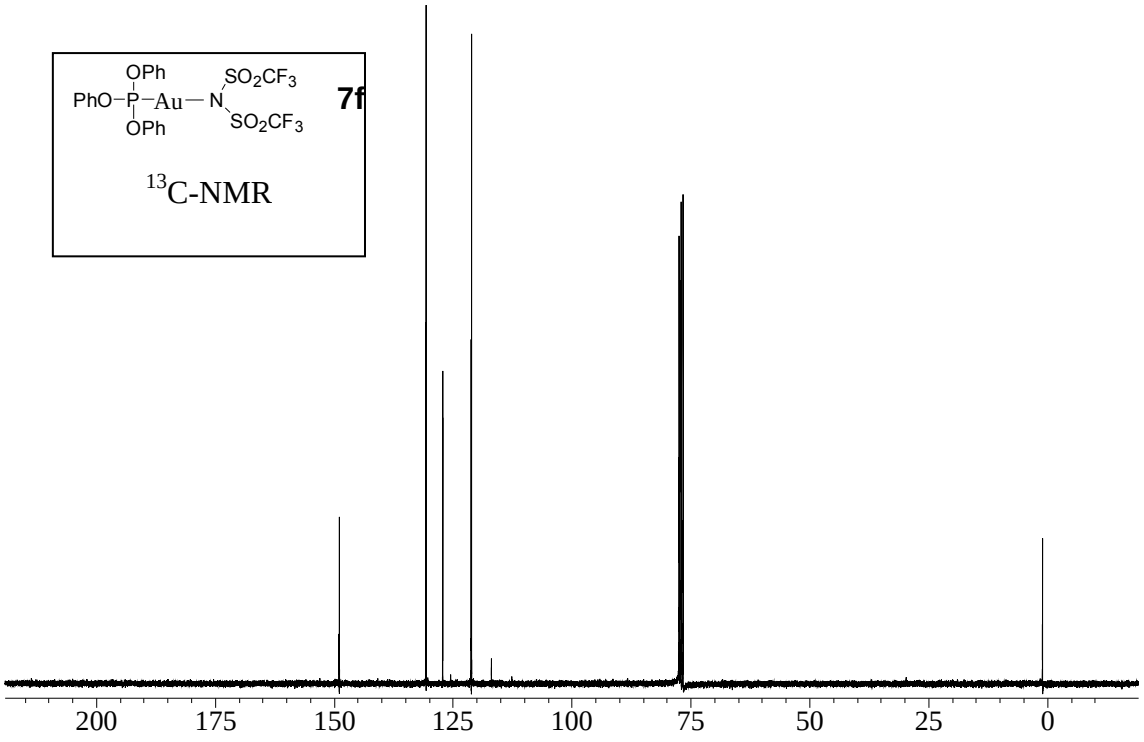
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PhO–P–Au–N
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SO2CF3
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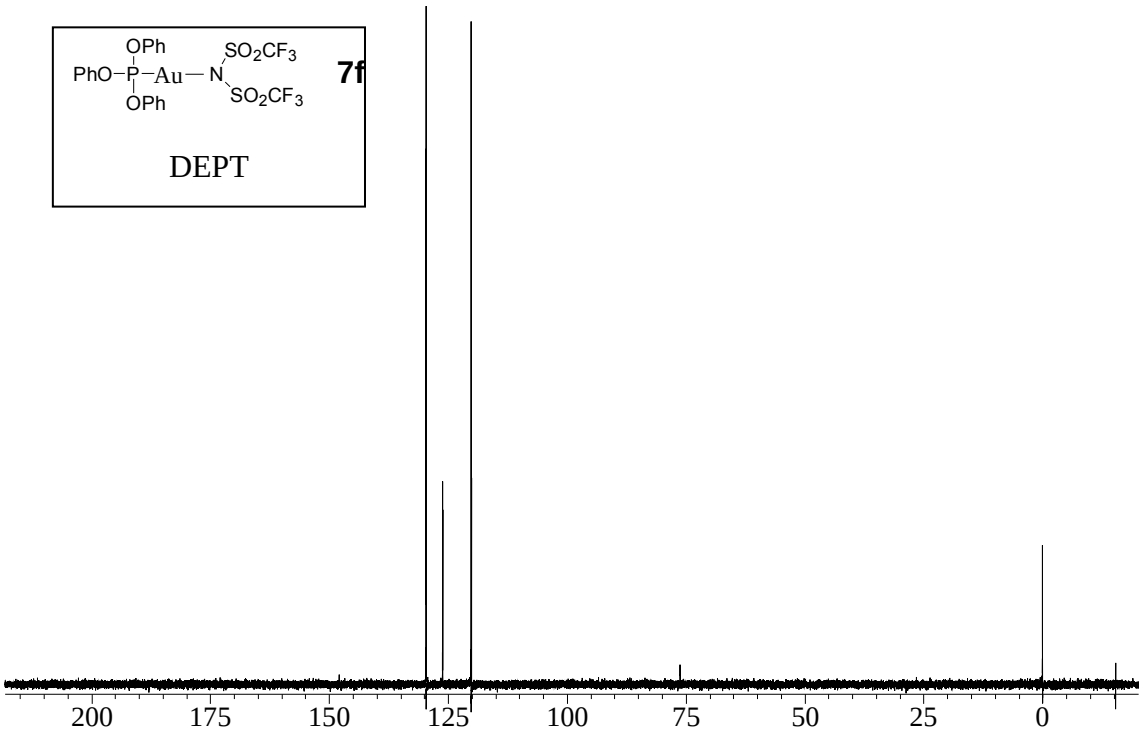
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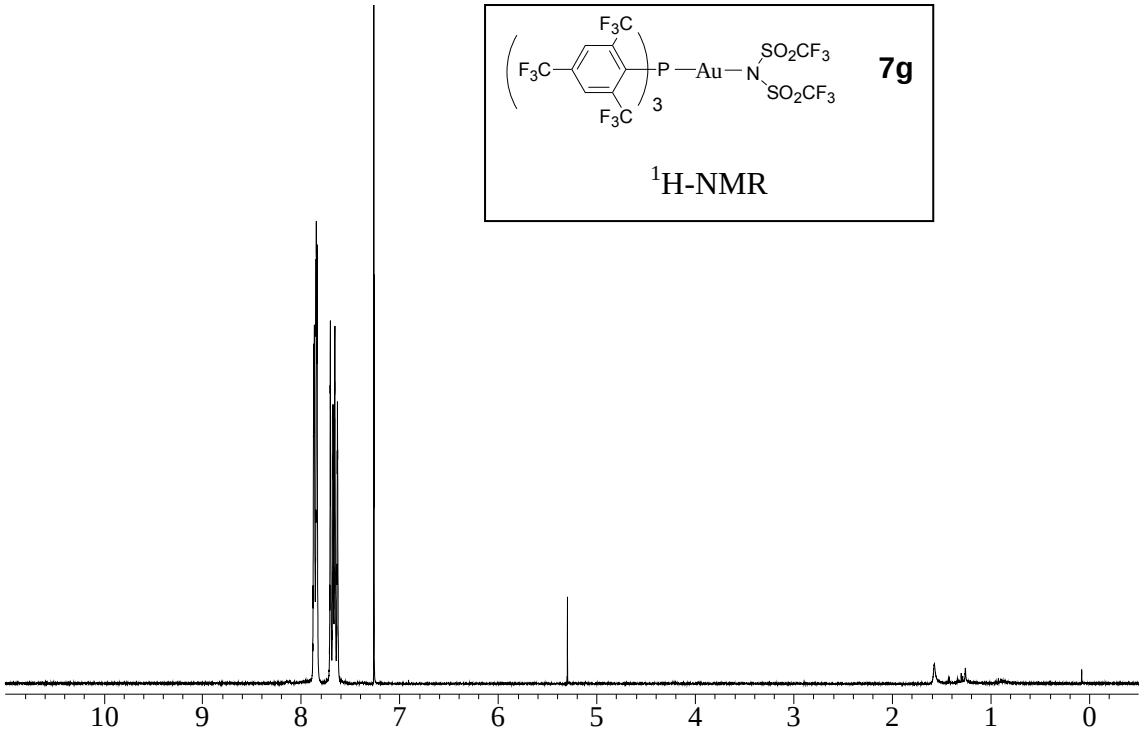
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F3C
F3C
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P
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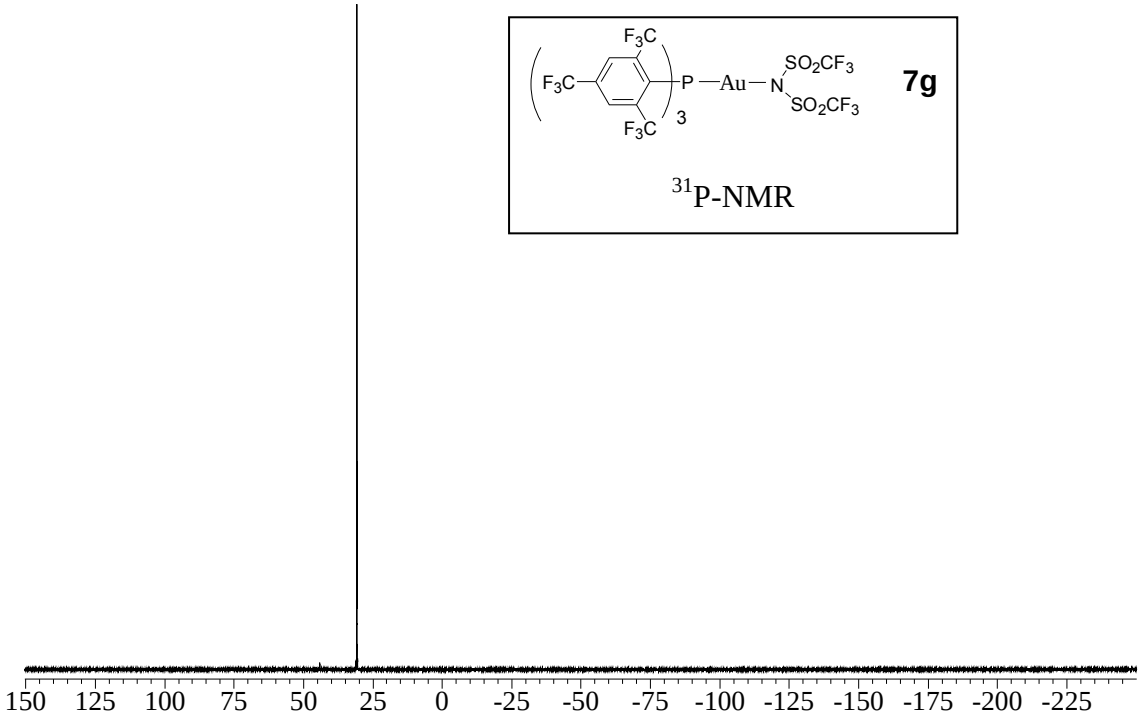
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F3C
F3C
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7g
31P-NMR
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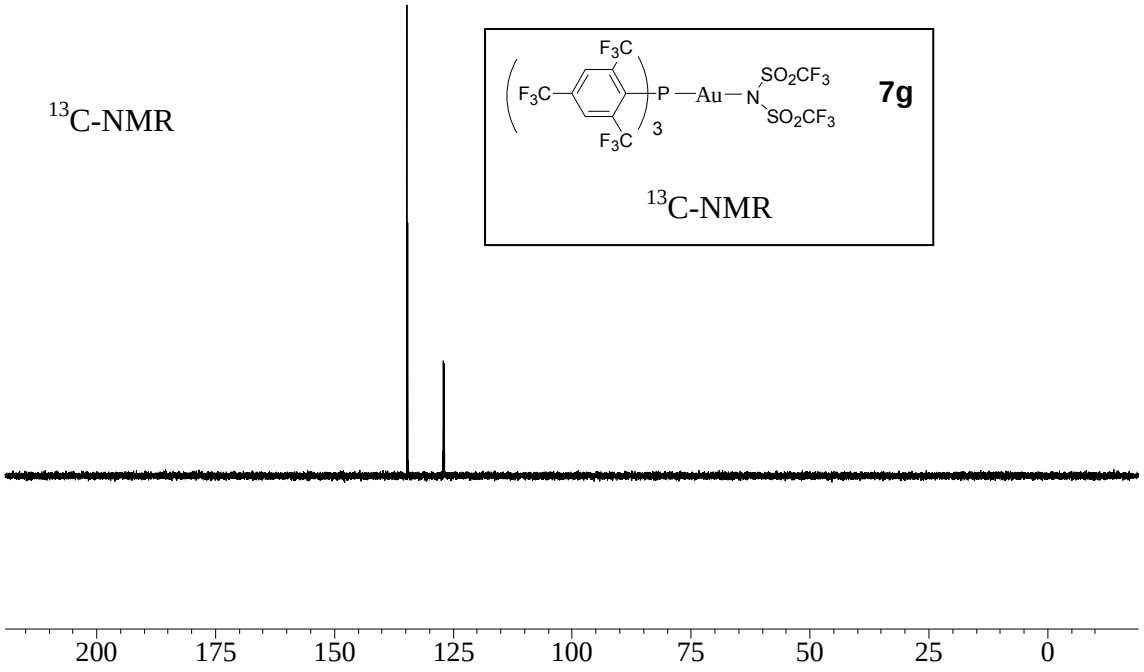
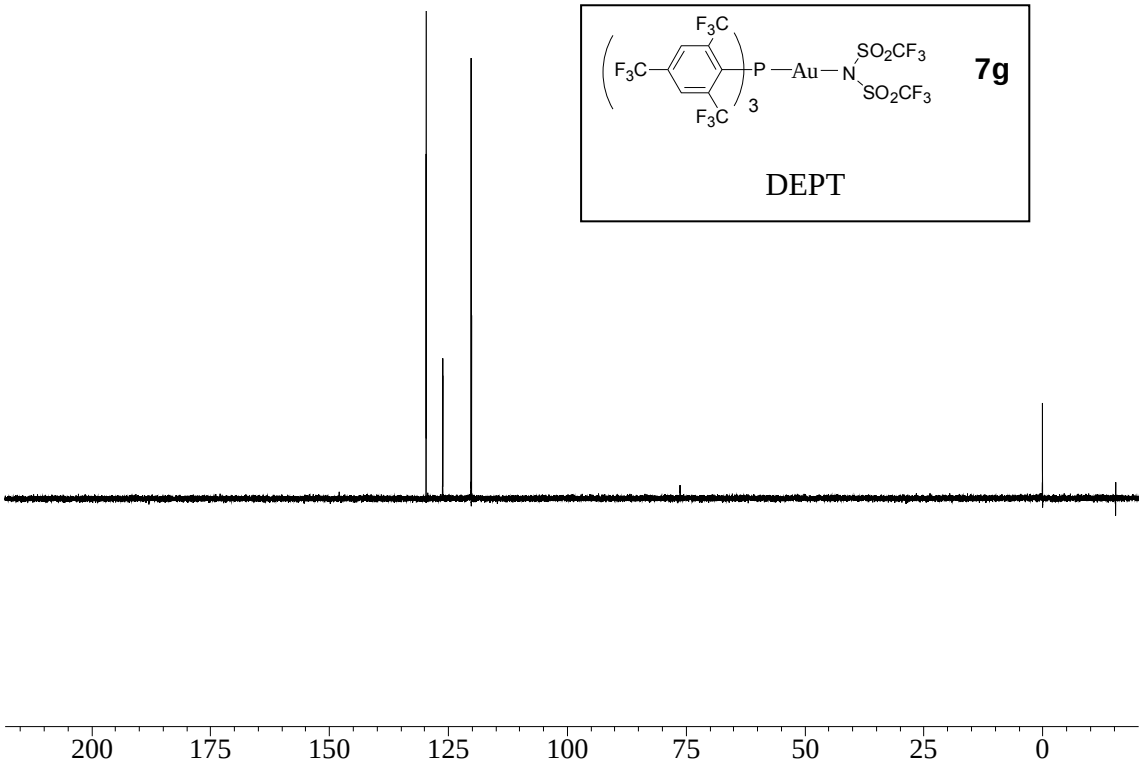
-225

^{13}C-NMR



^{13}C-NMR



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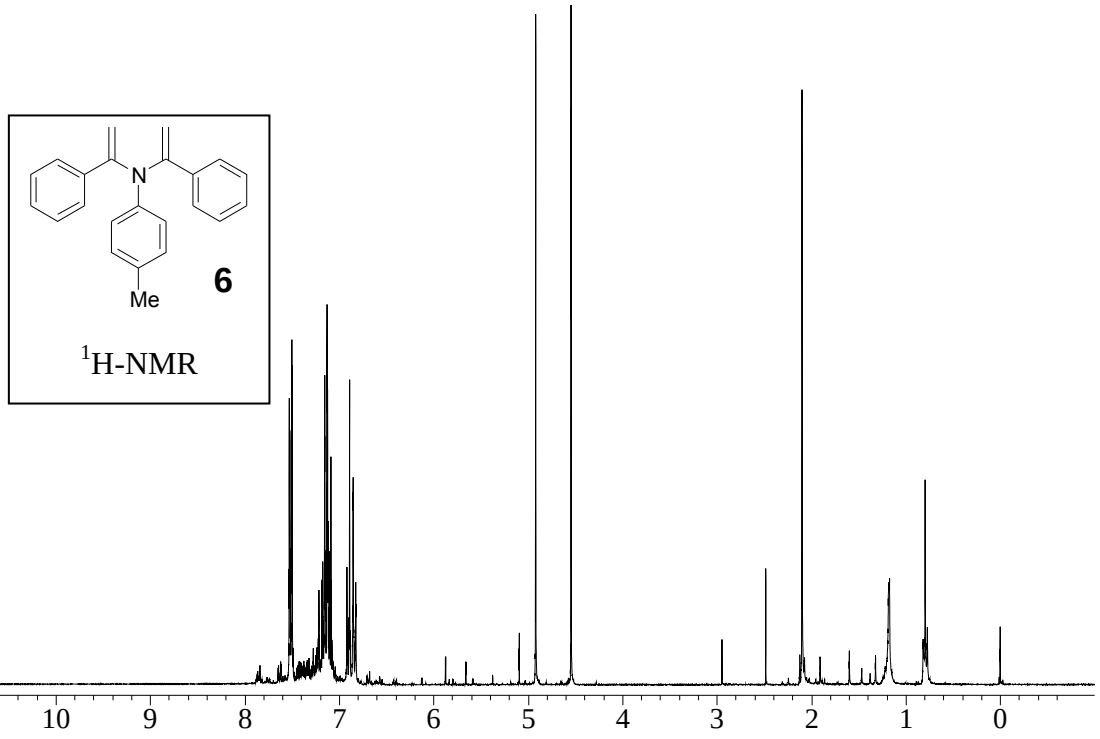
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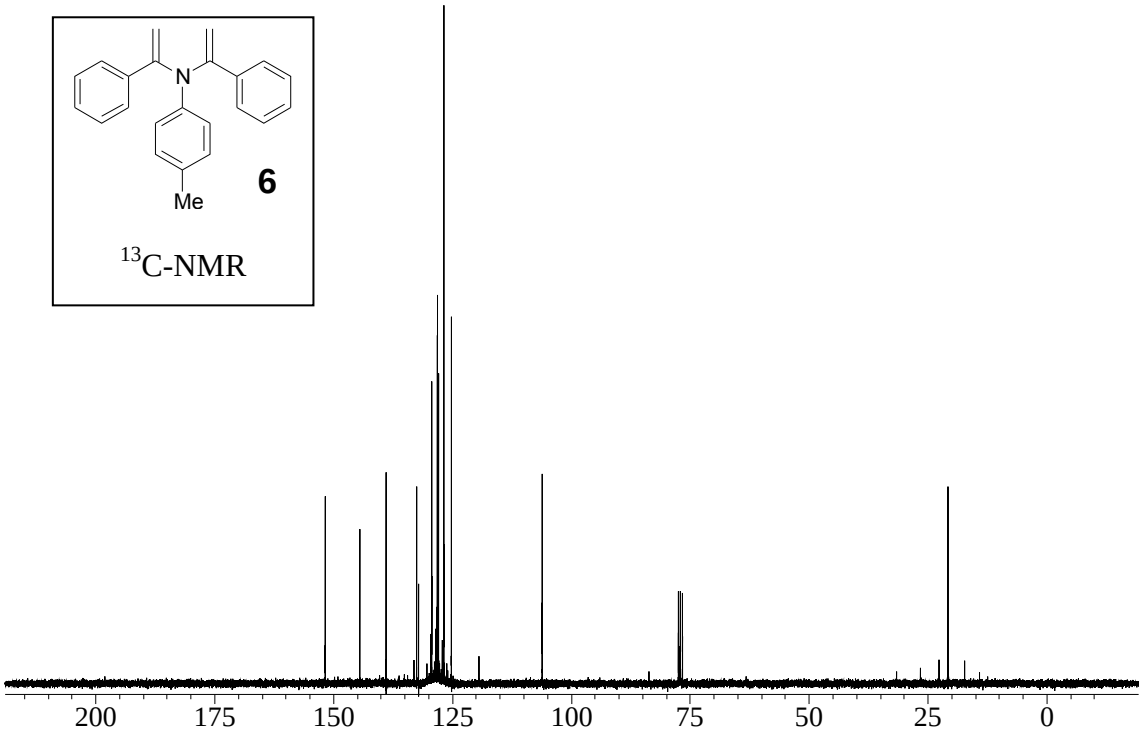
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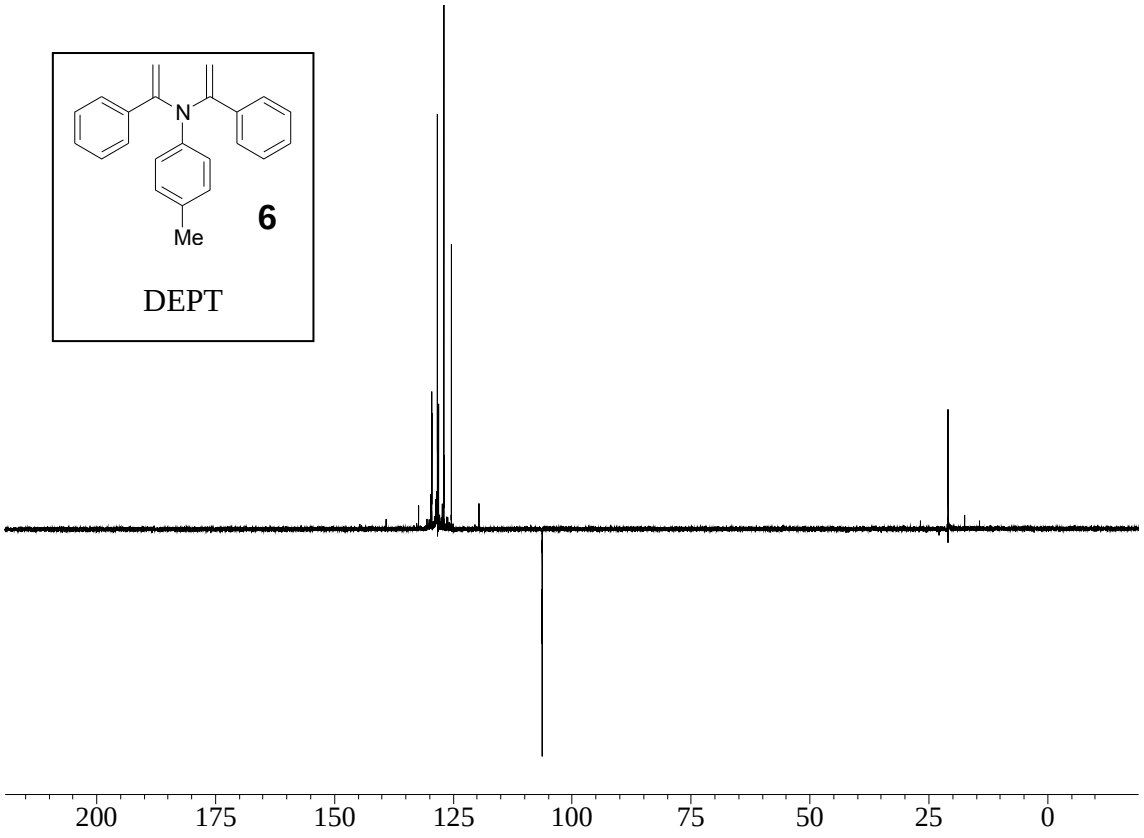
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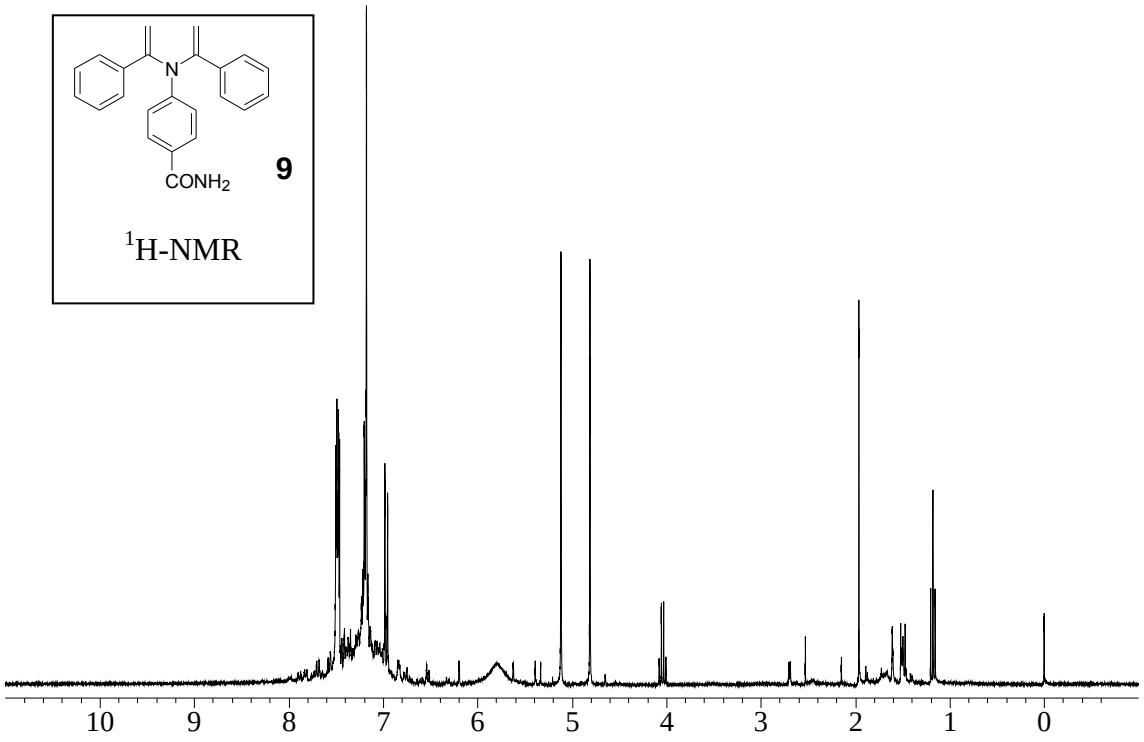
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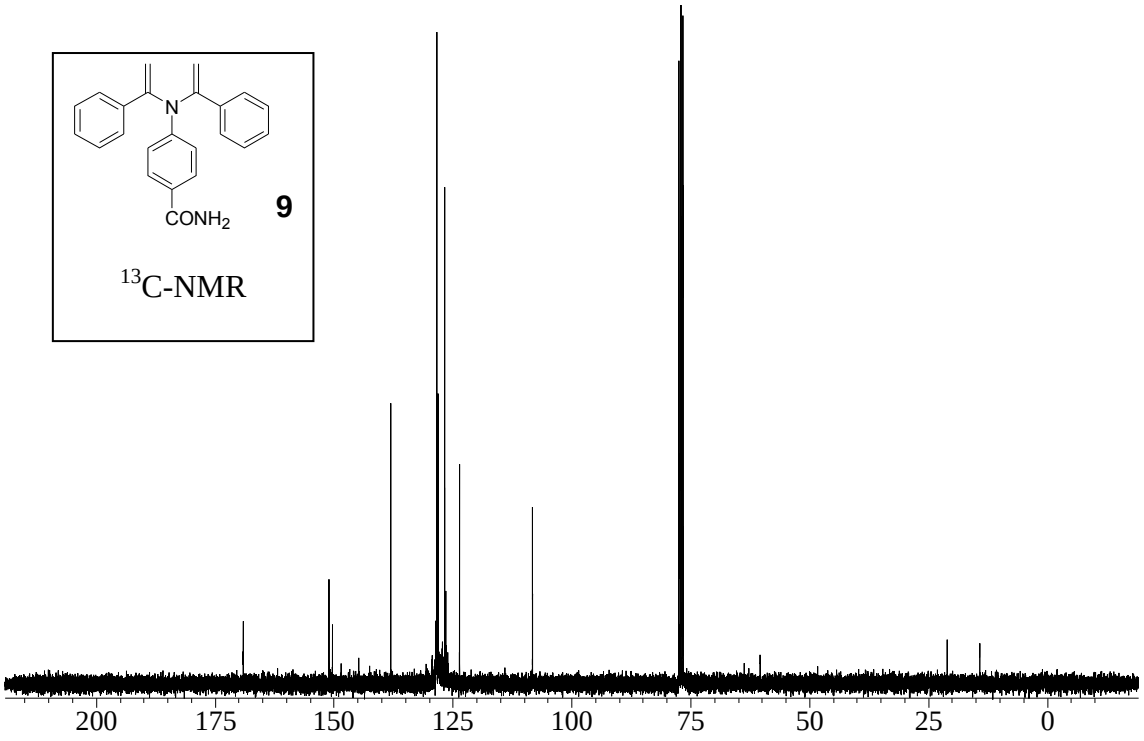
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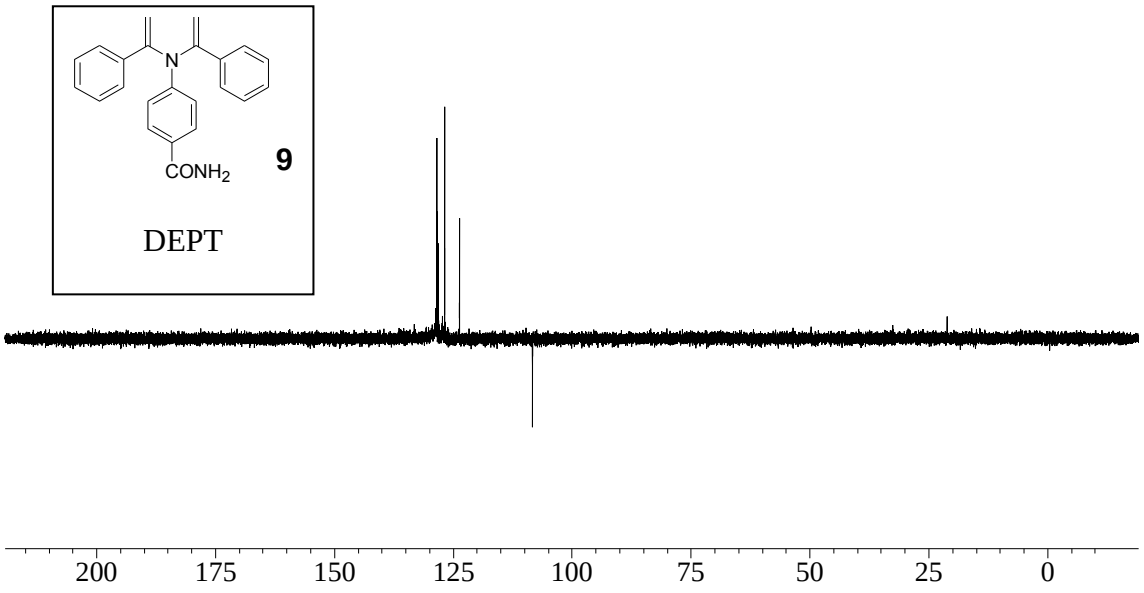
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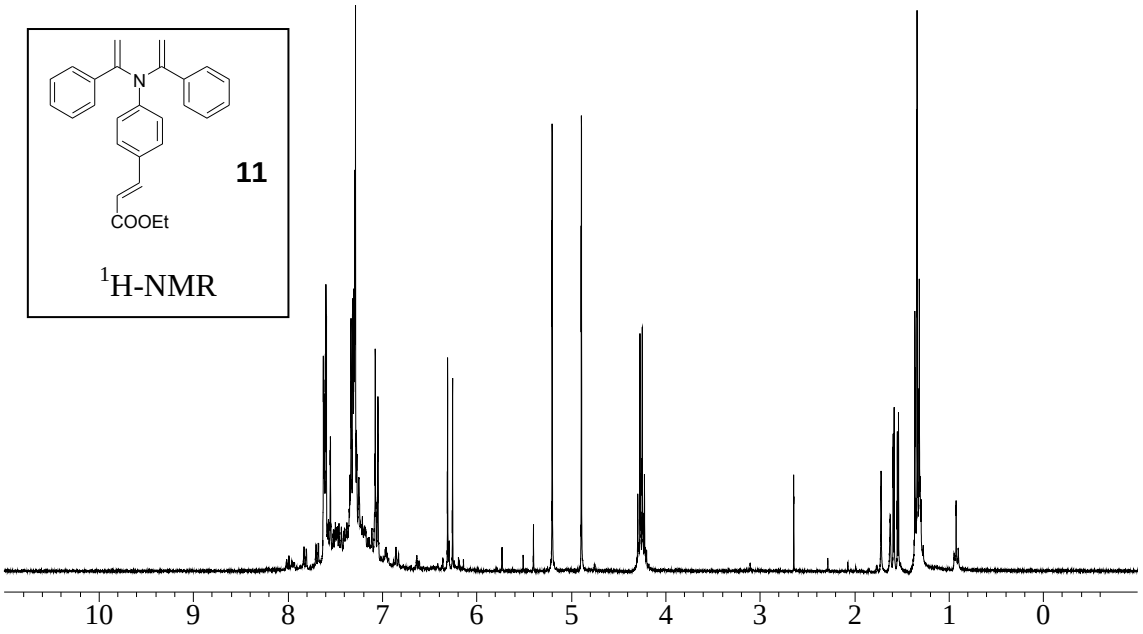
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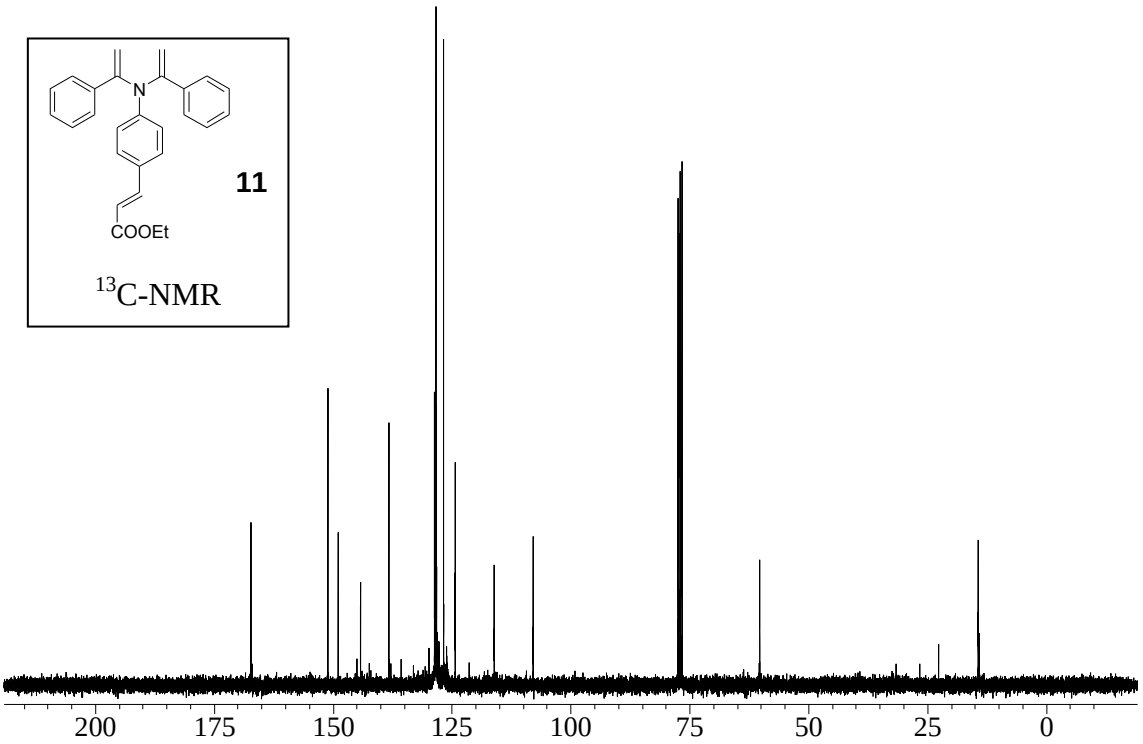
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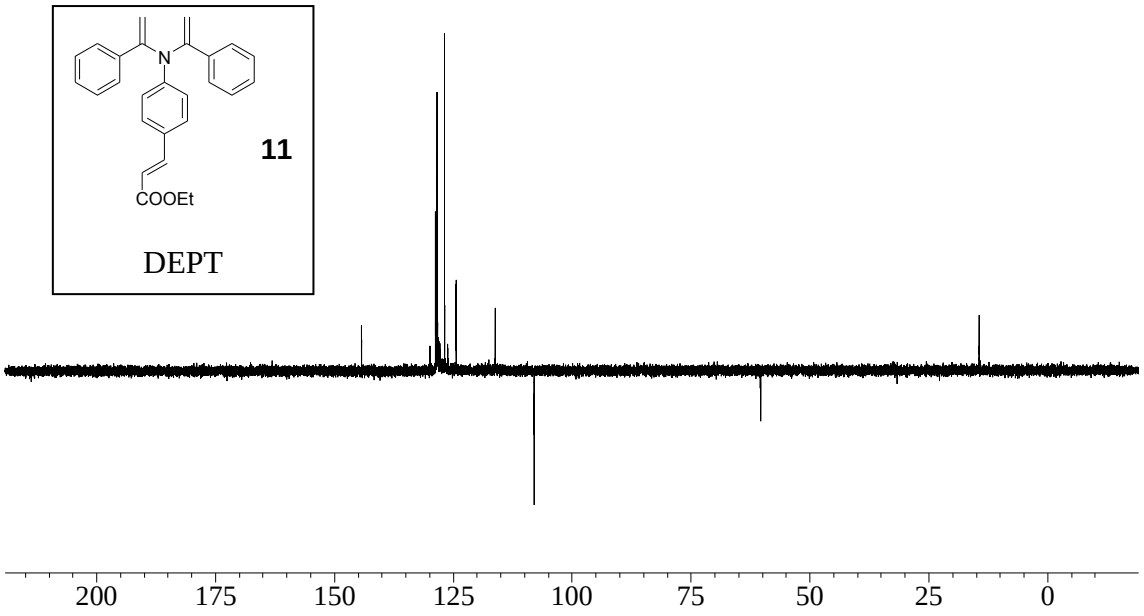
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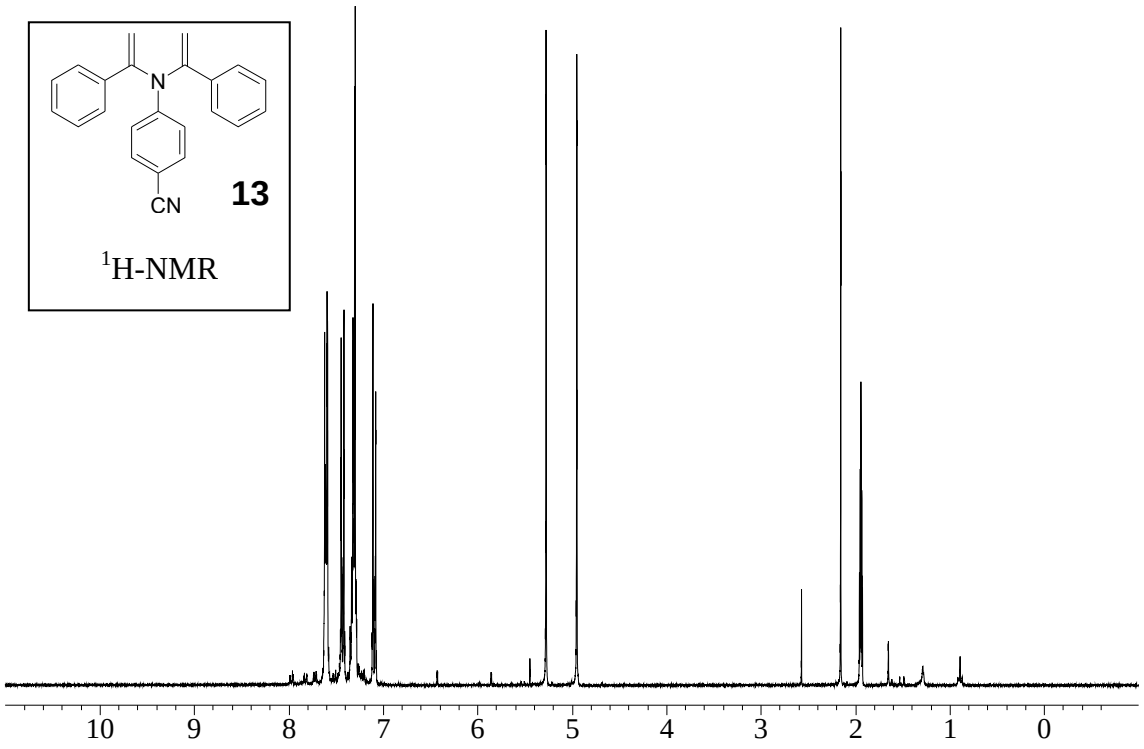
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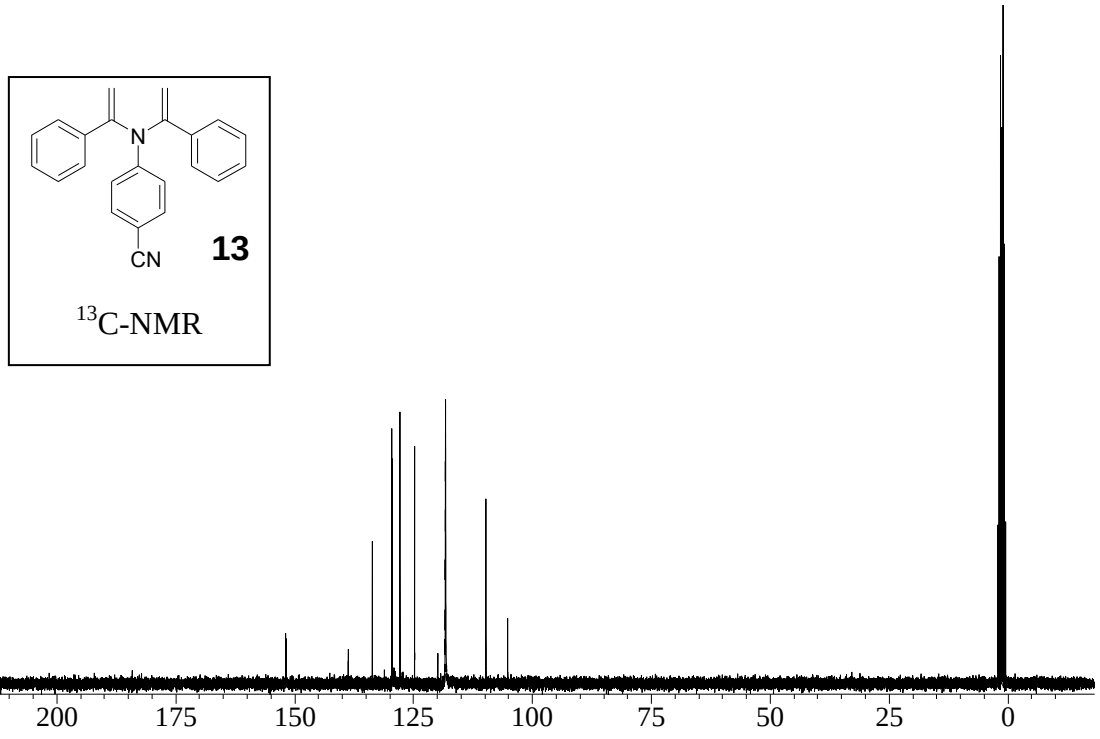
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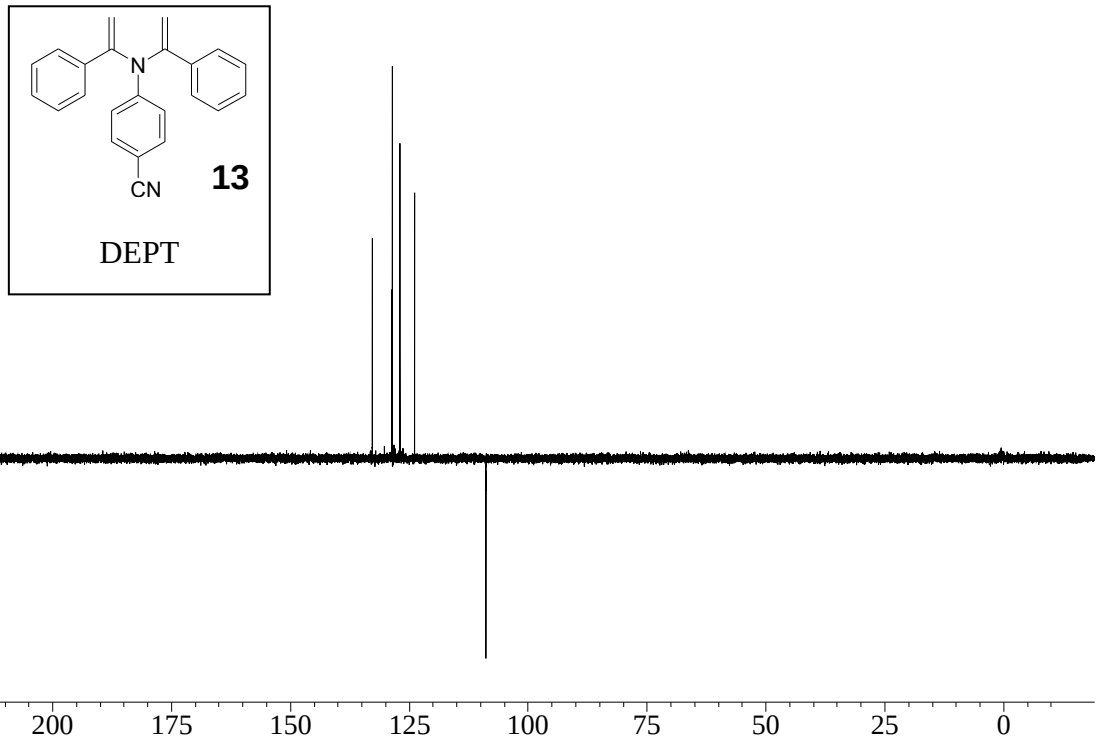
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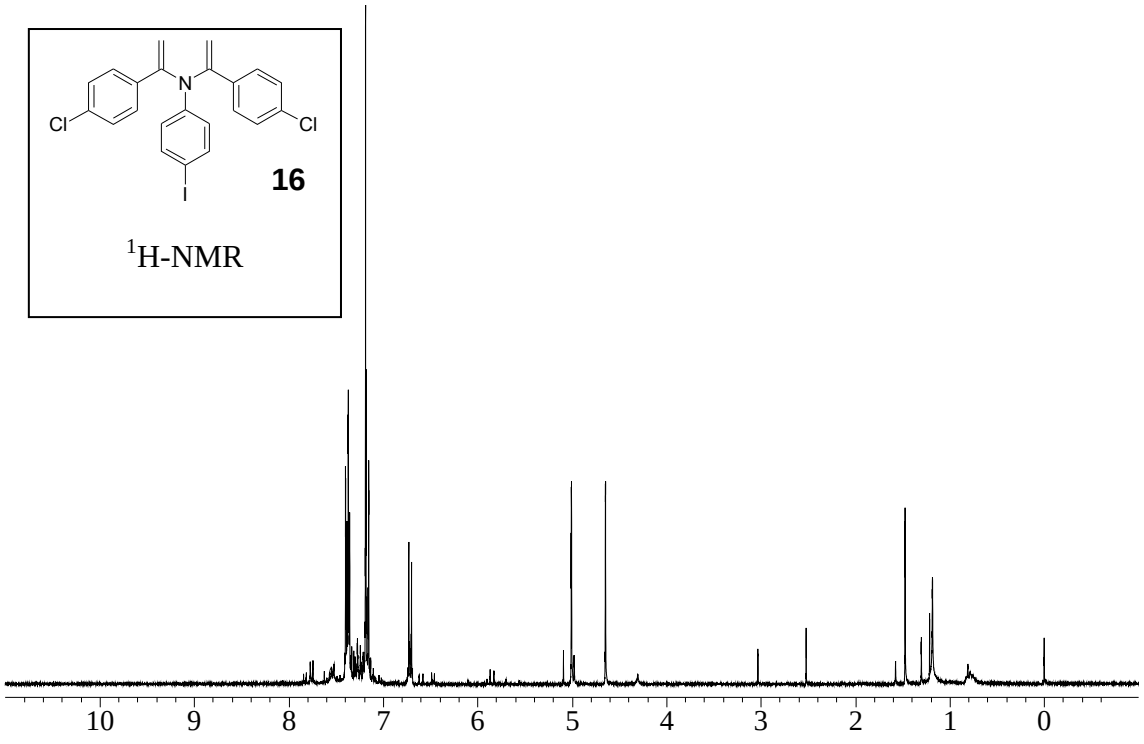
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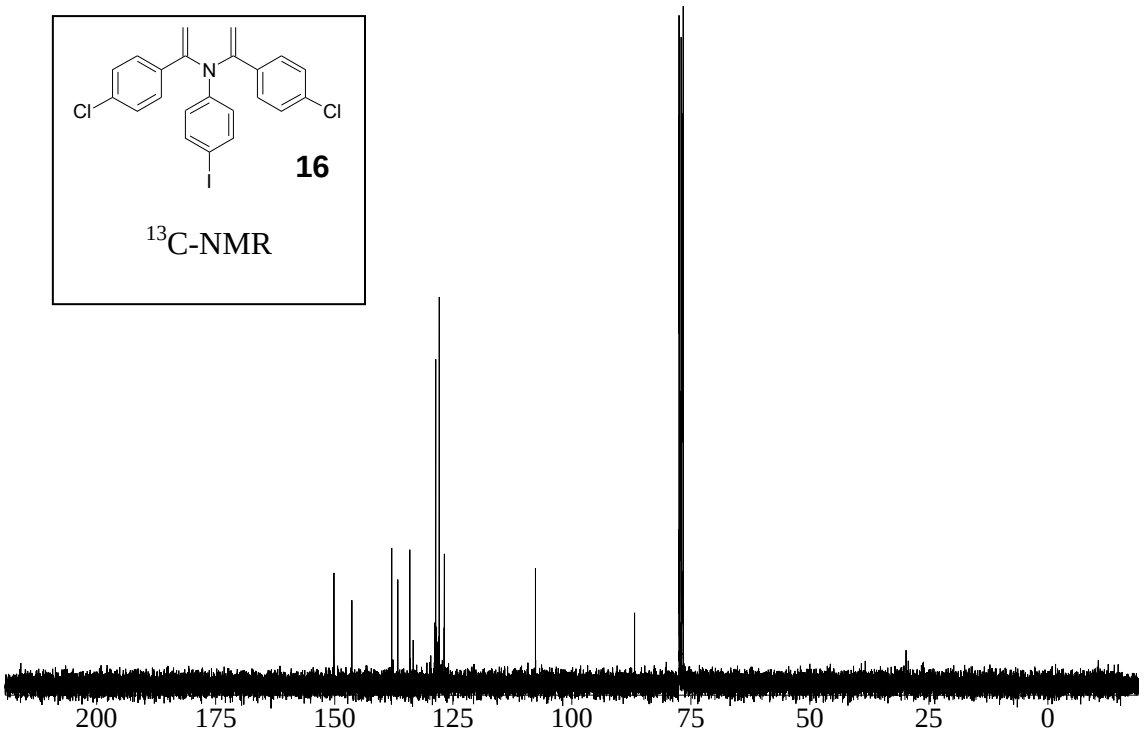
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13C-NMR
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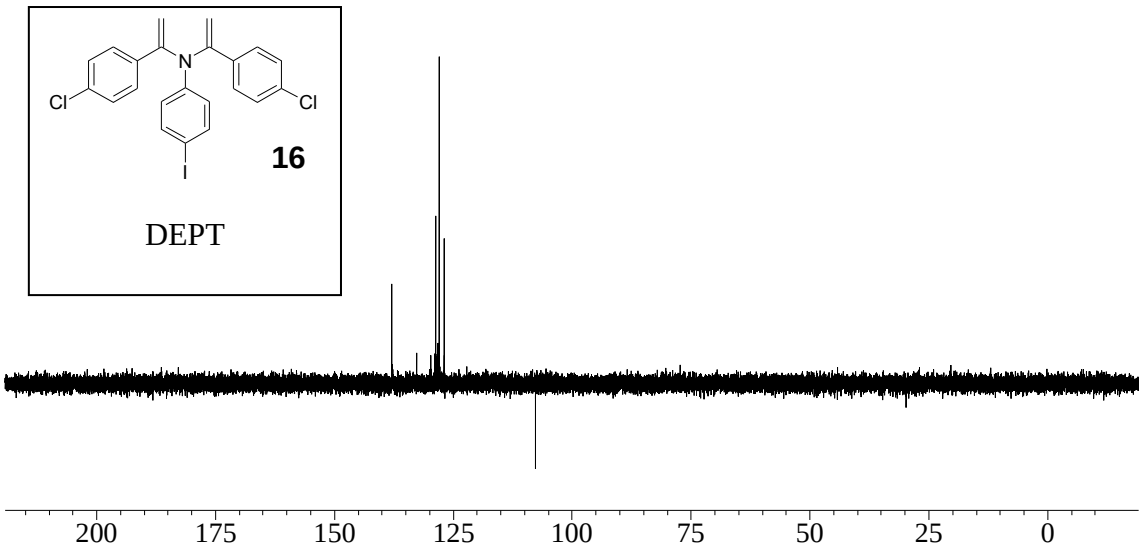
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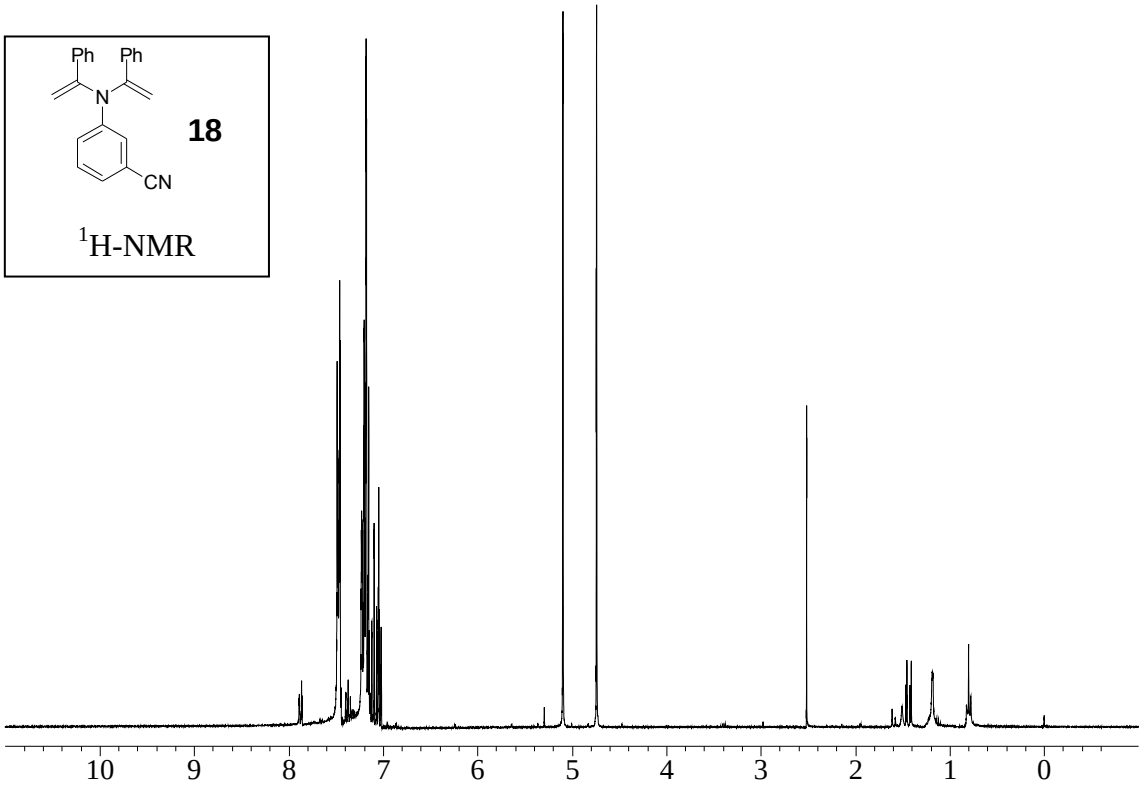
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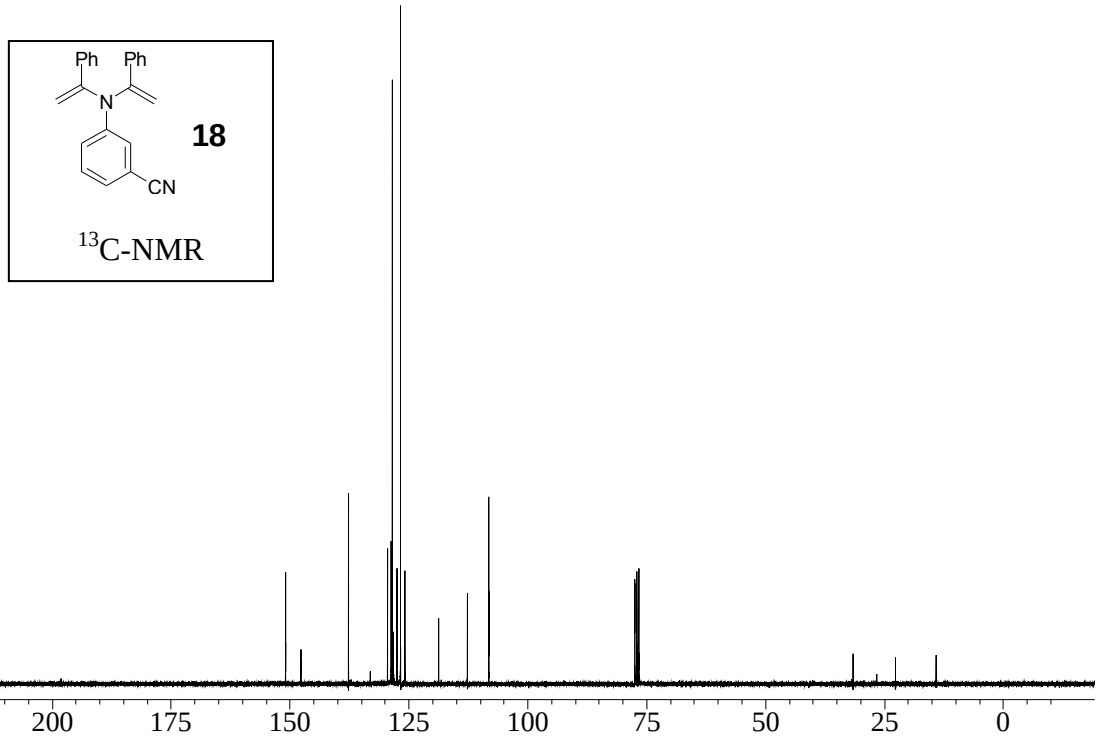
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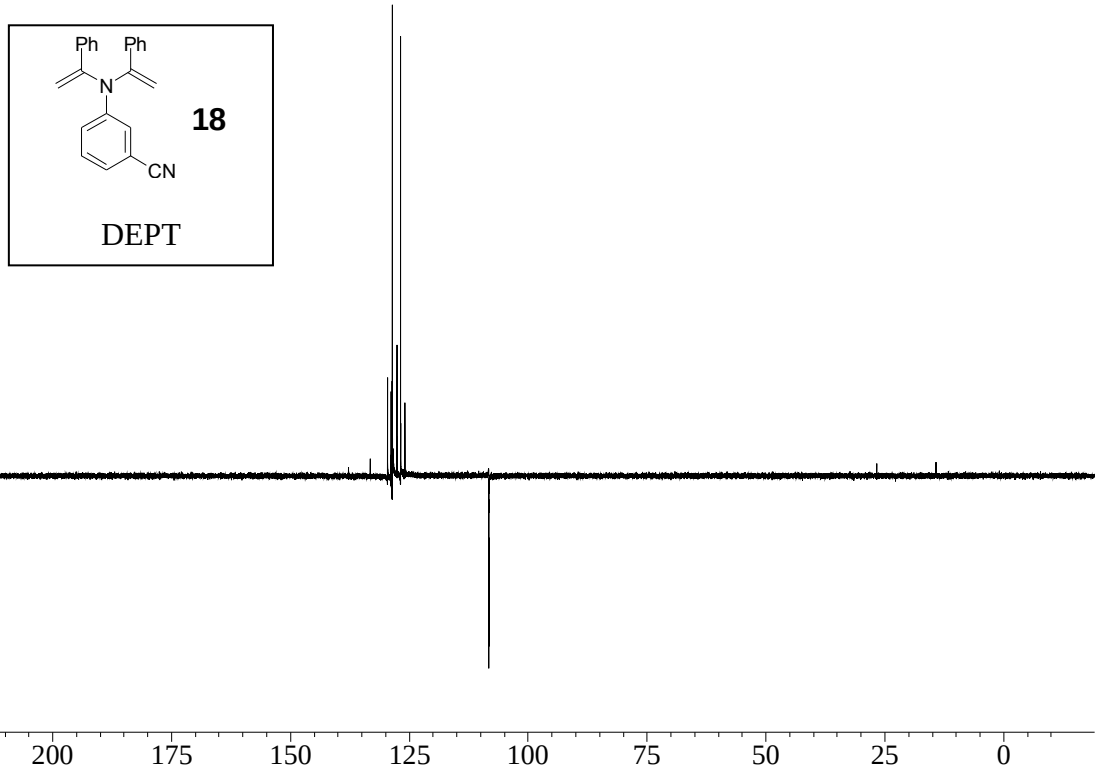
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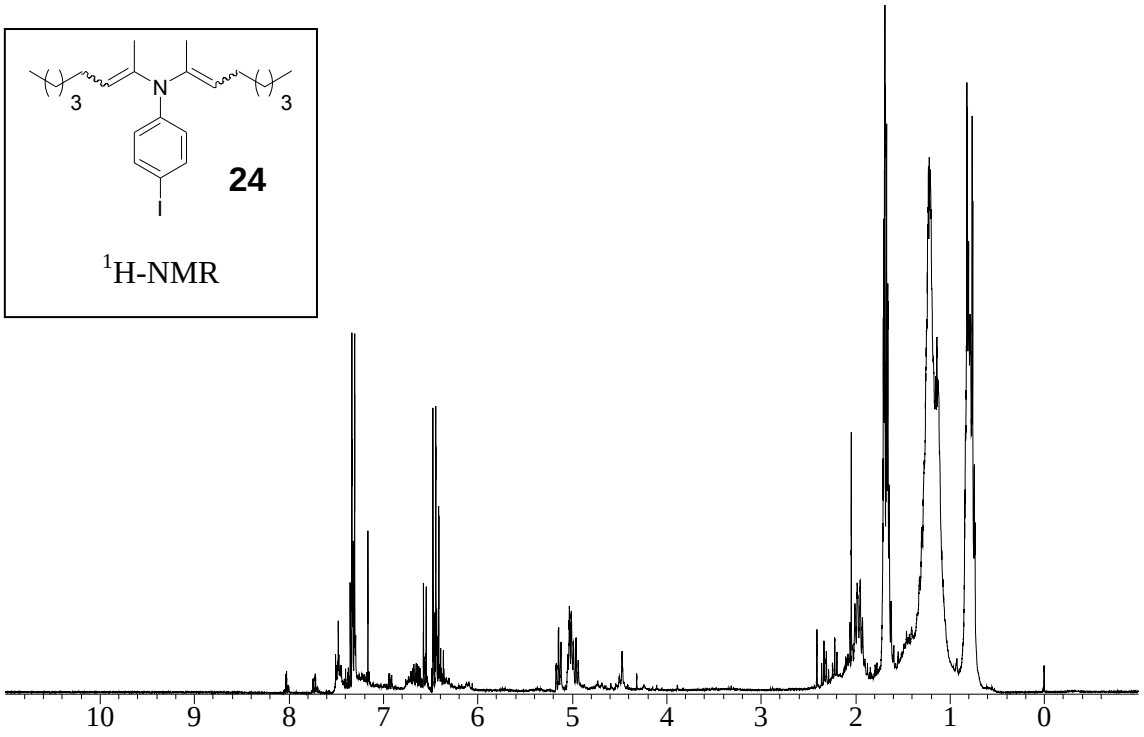
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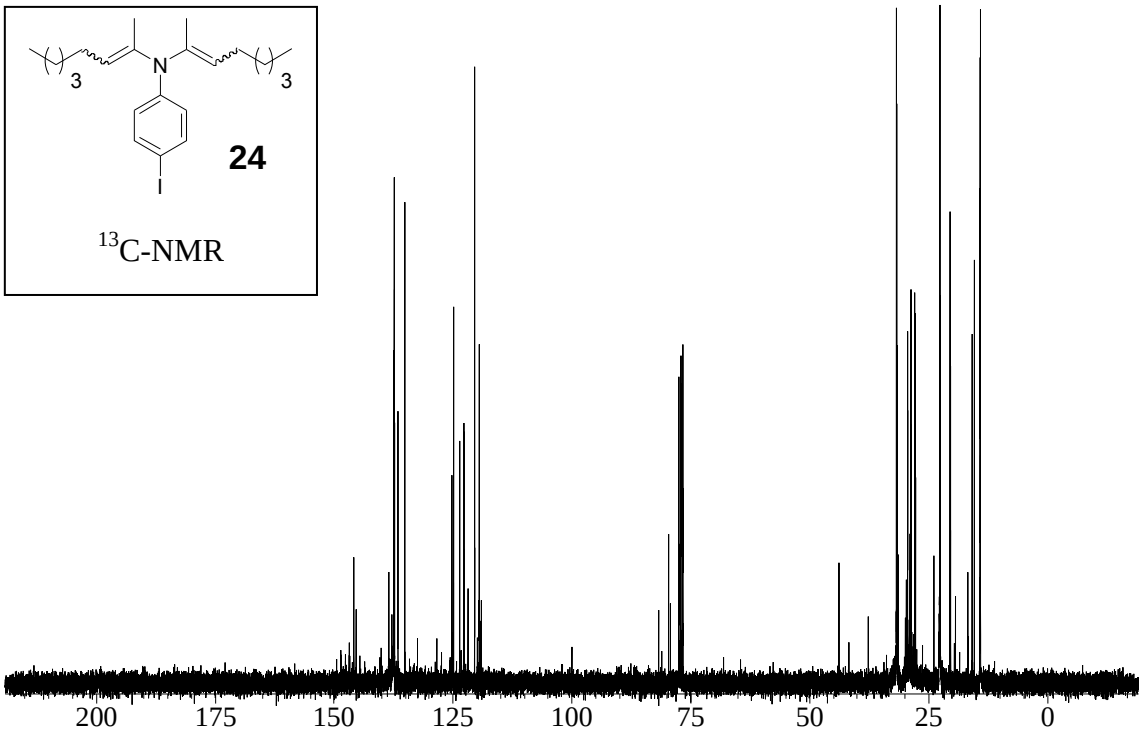
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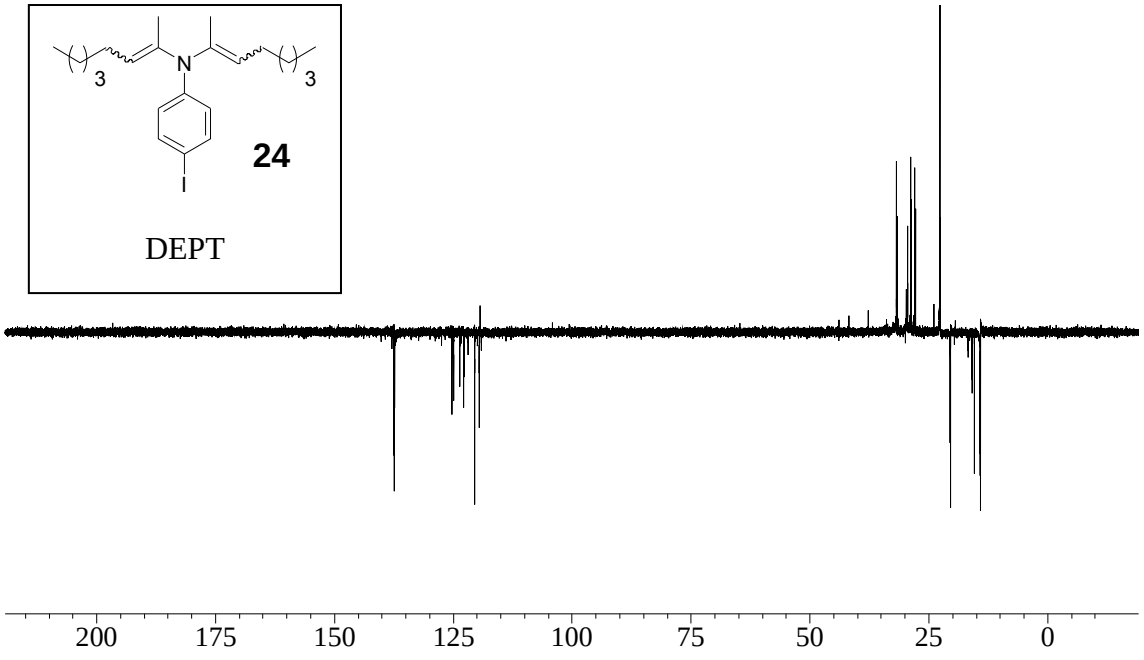
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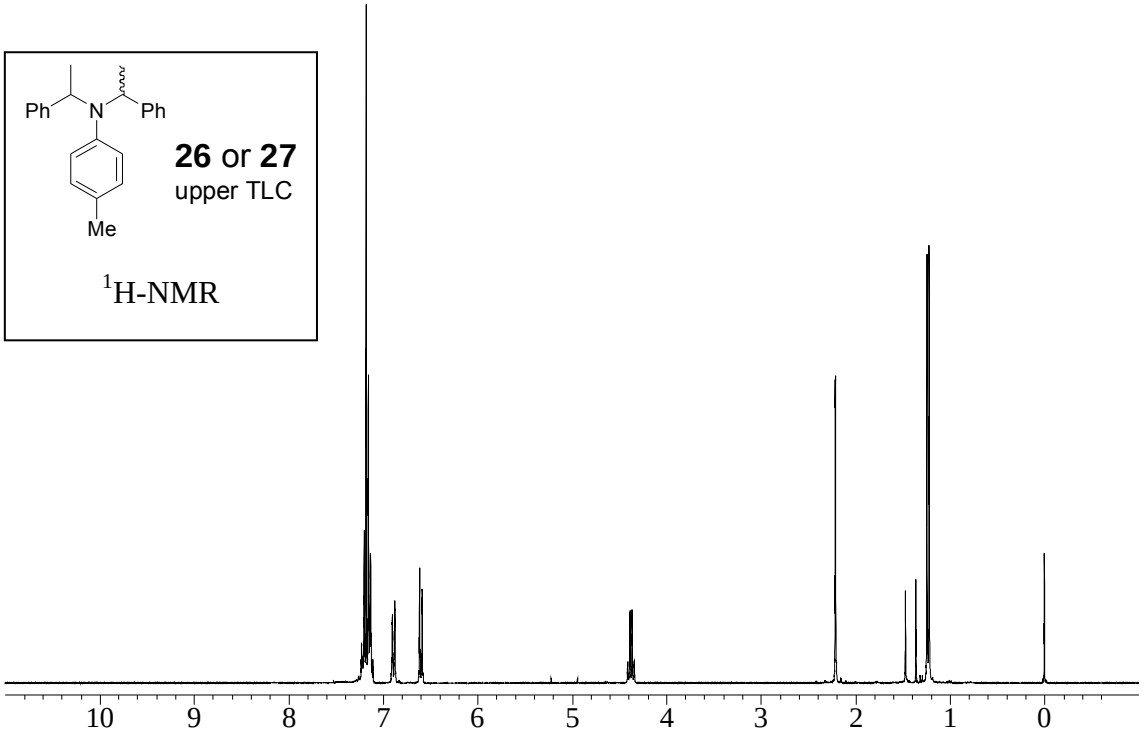
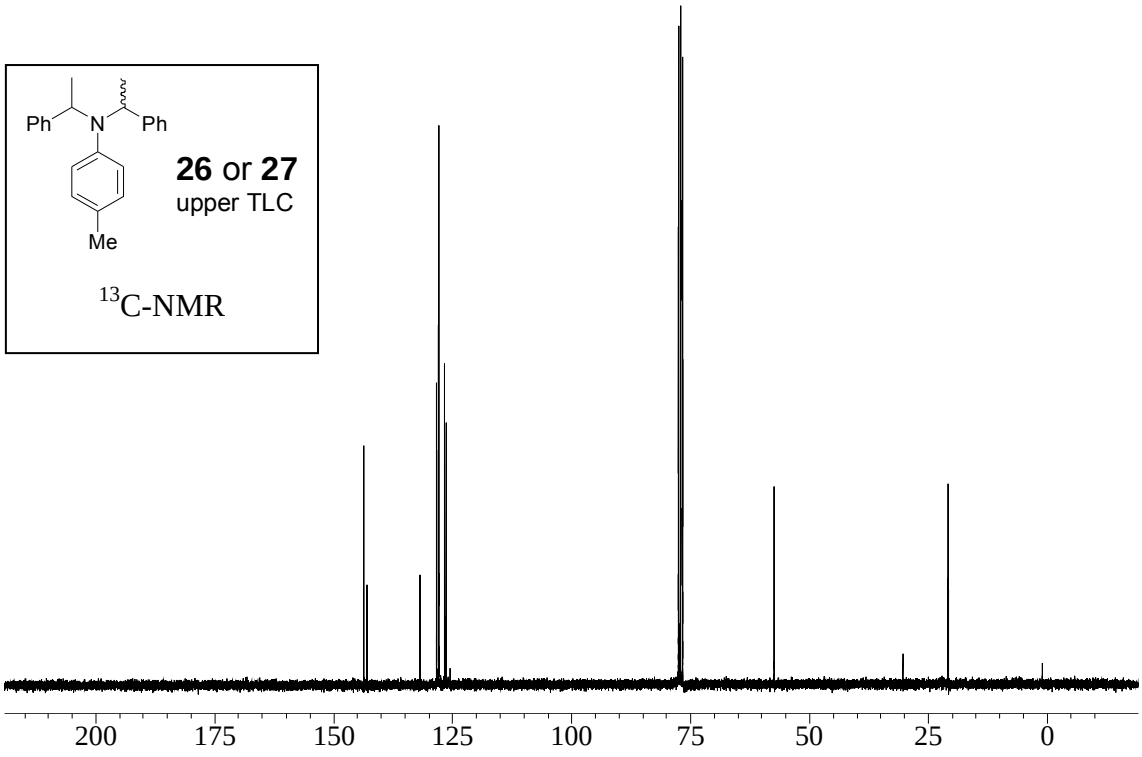
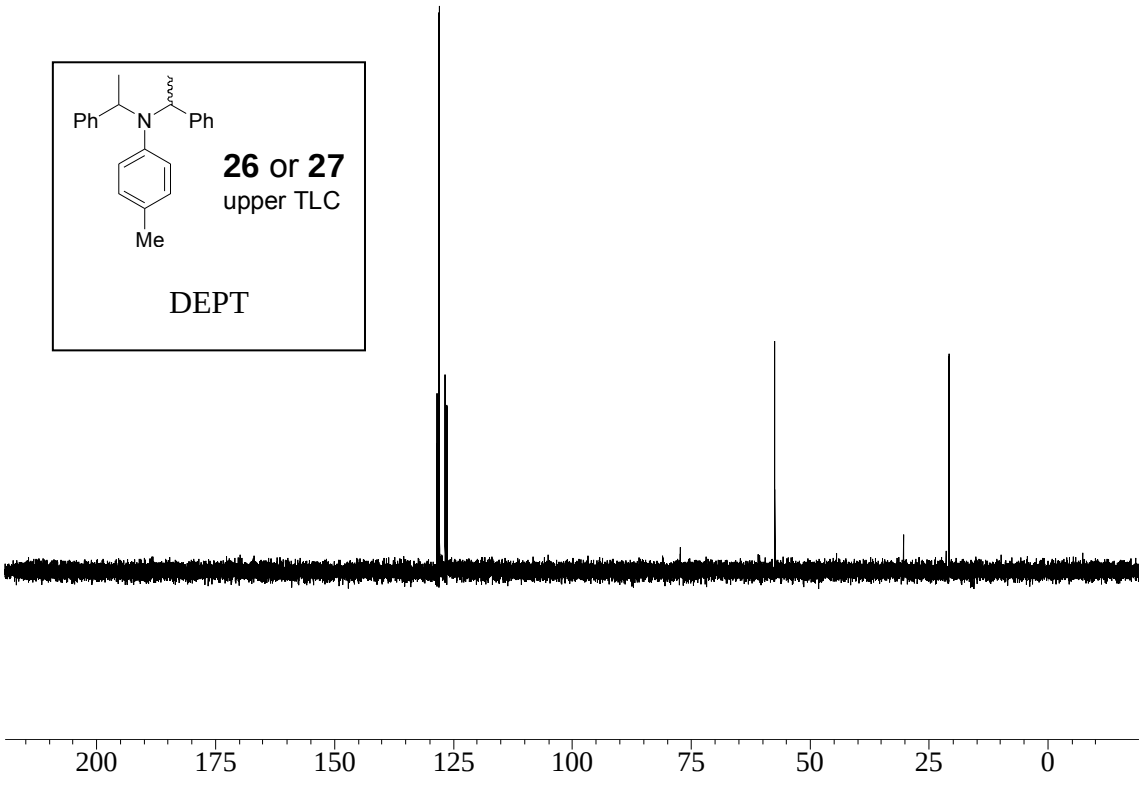
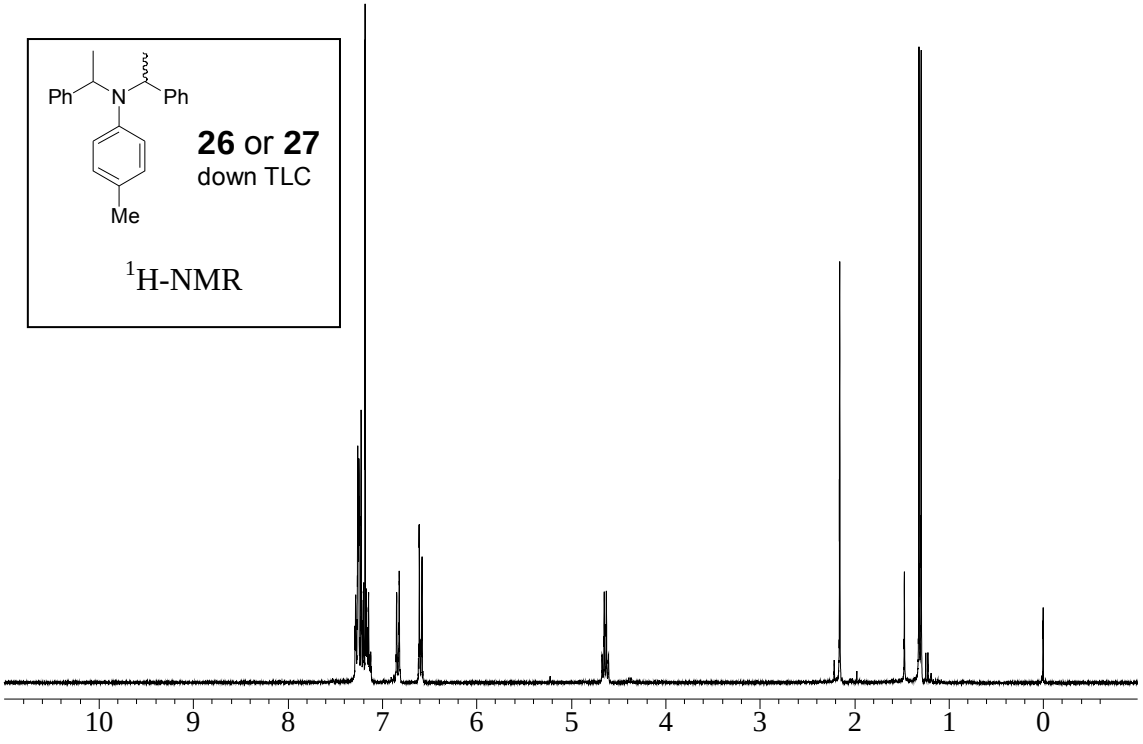
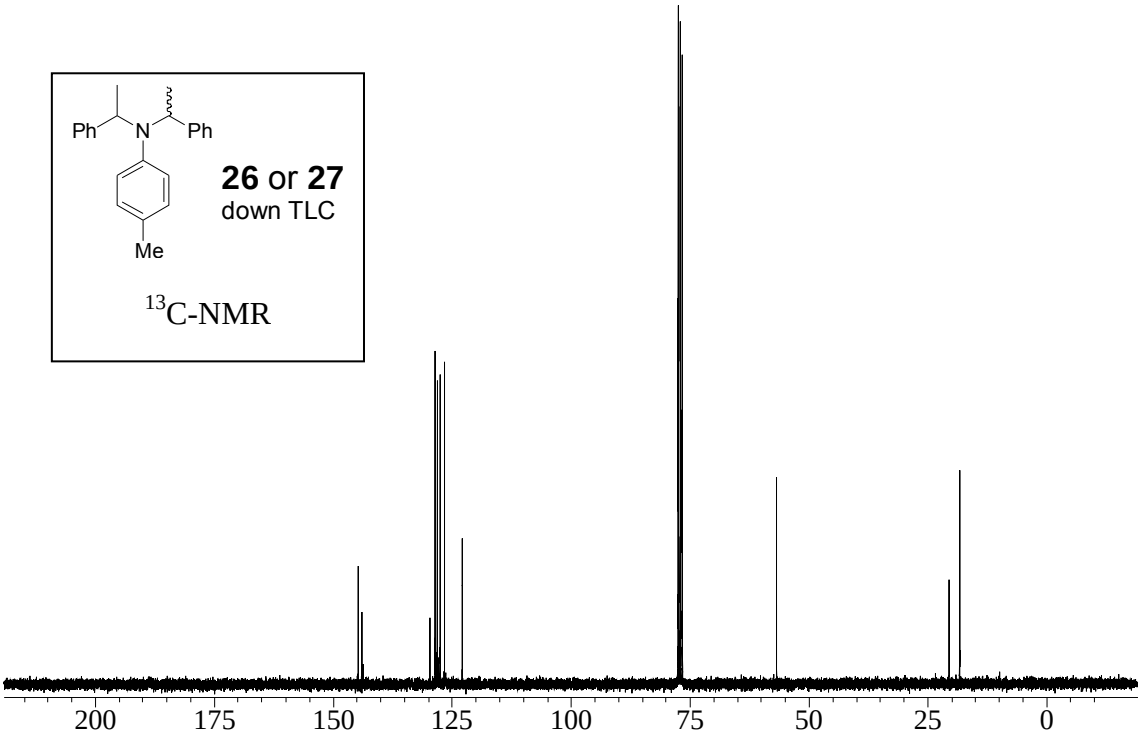
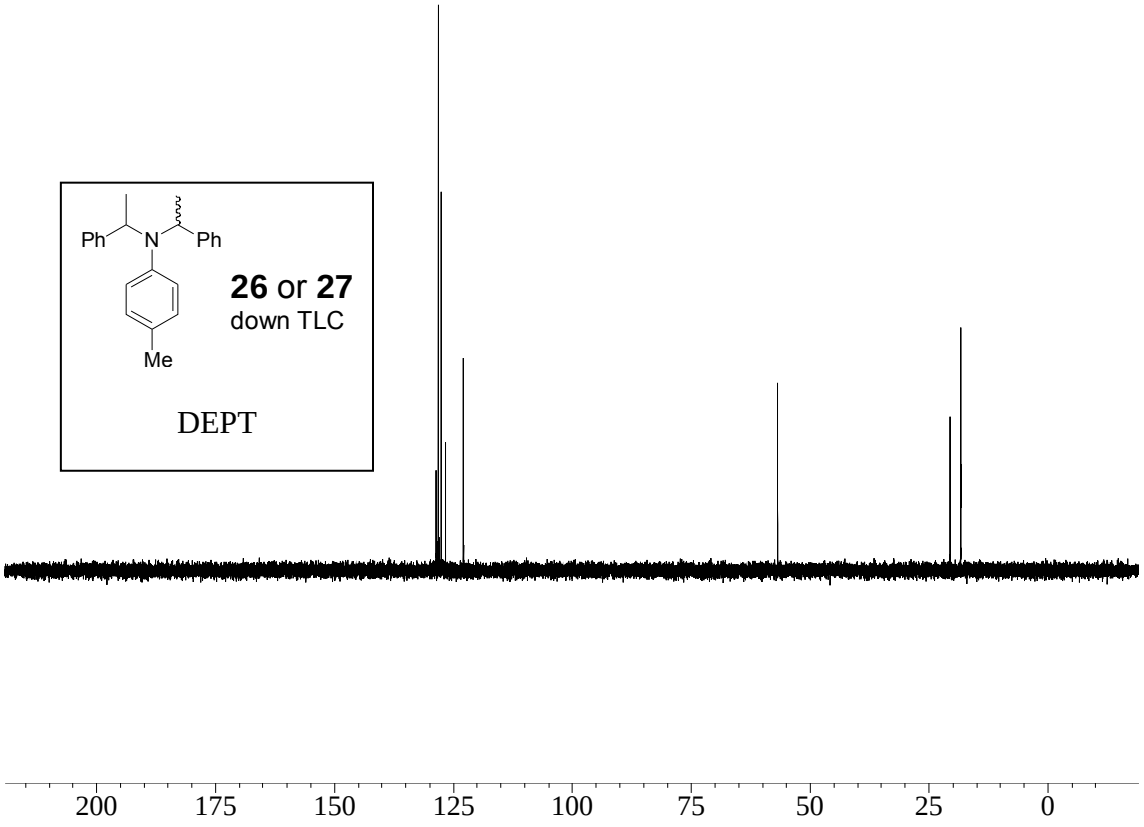

Ph
N
Ph
Me
26 or 27
upper TLC
1H-NMR
10
9
8
7
6
5
4
3
2
1
0

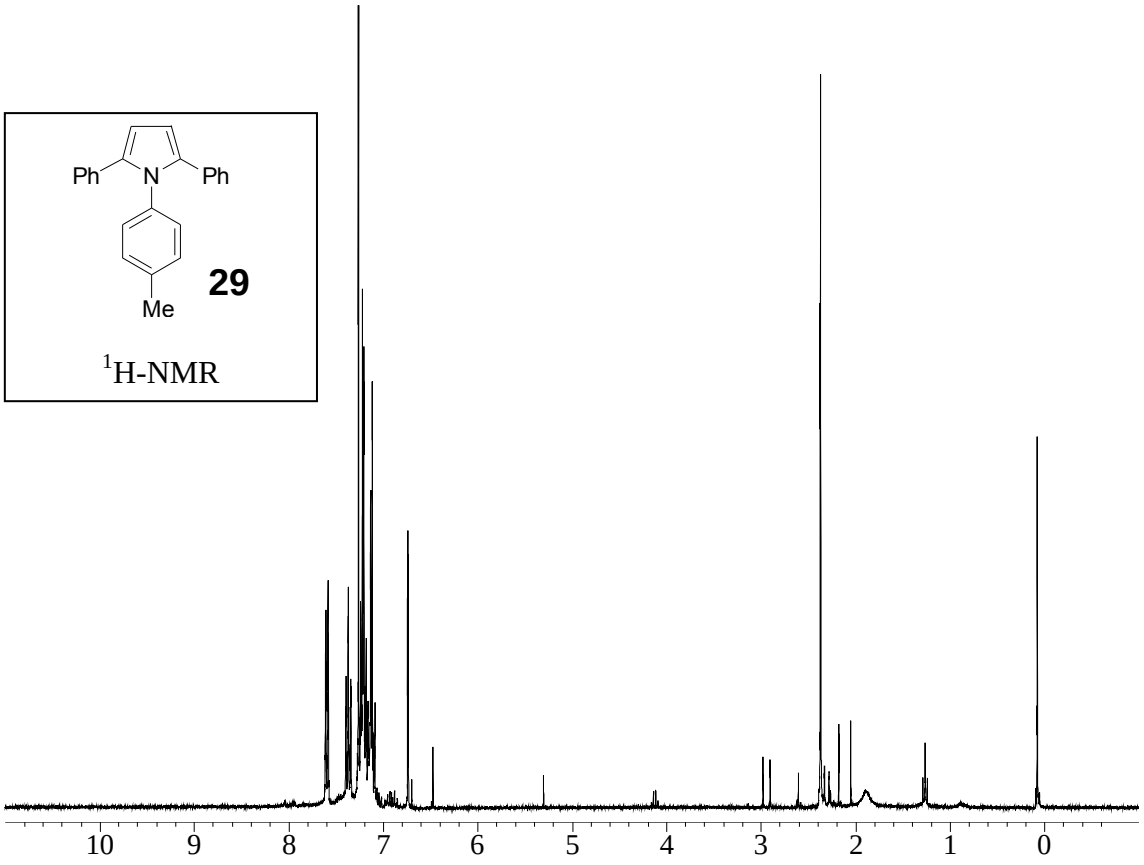

Ph
N
Ph
Me
26 or 27
upper TLC
13C-NMR
200
175
150
125
100
75
50
25
0

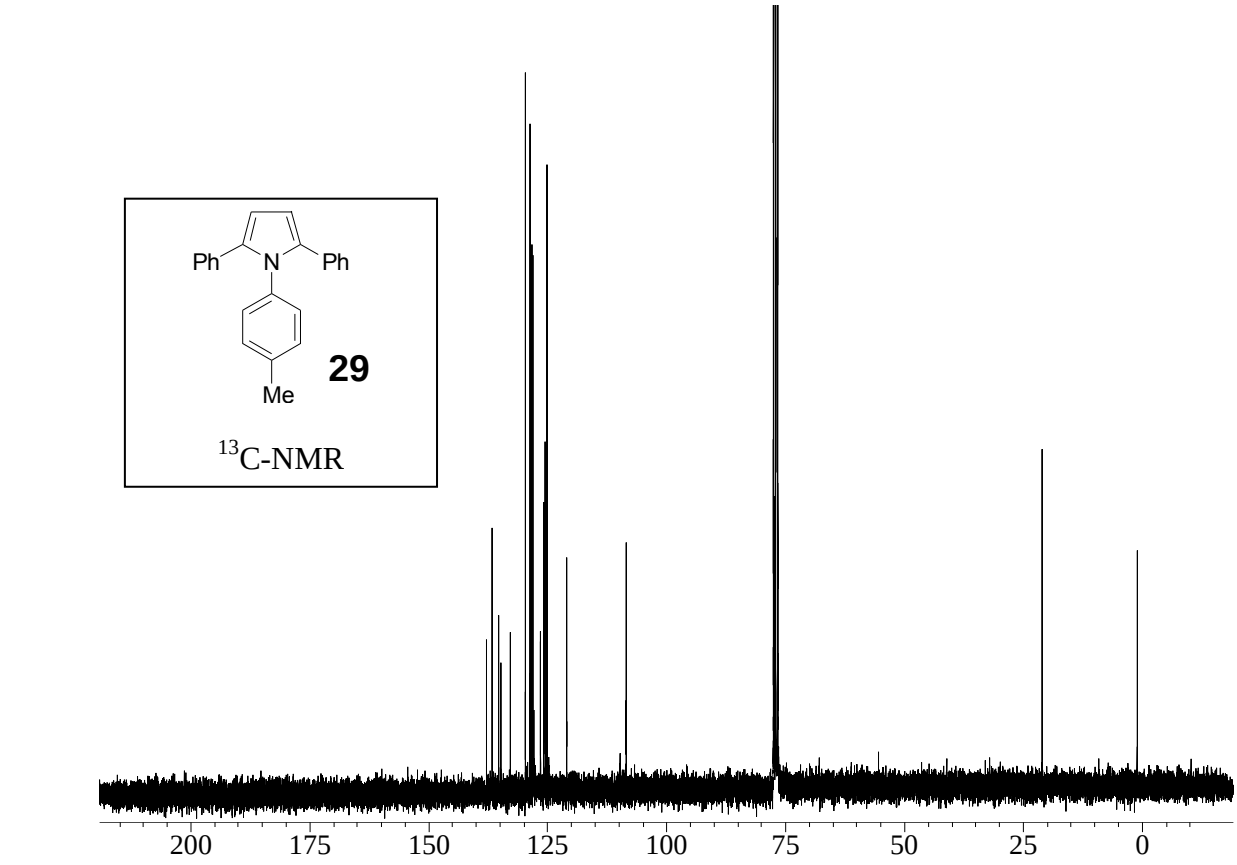

Ph
N
Ph
Me
26 or 27
upper TLC
DEPT
200
175
150
125
100
75
50
25
0

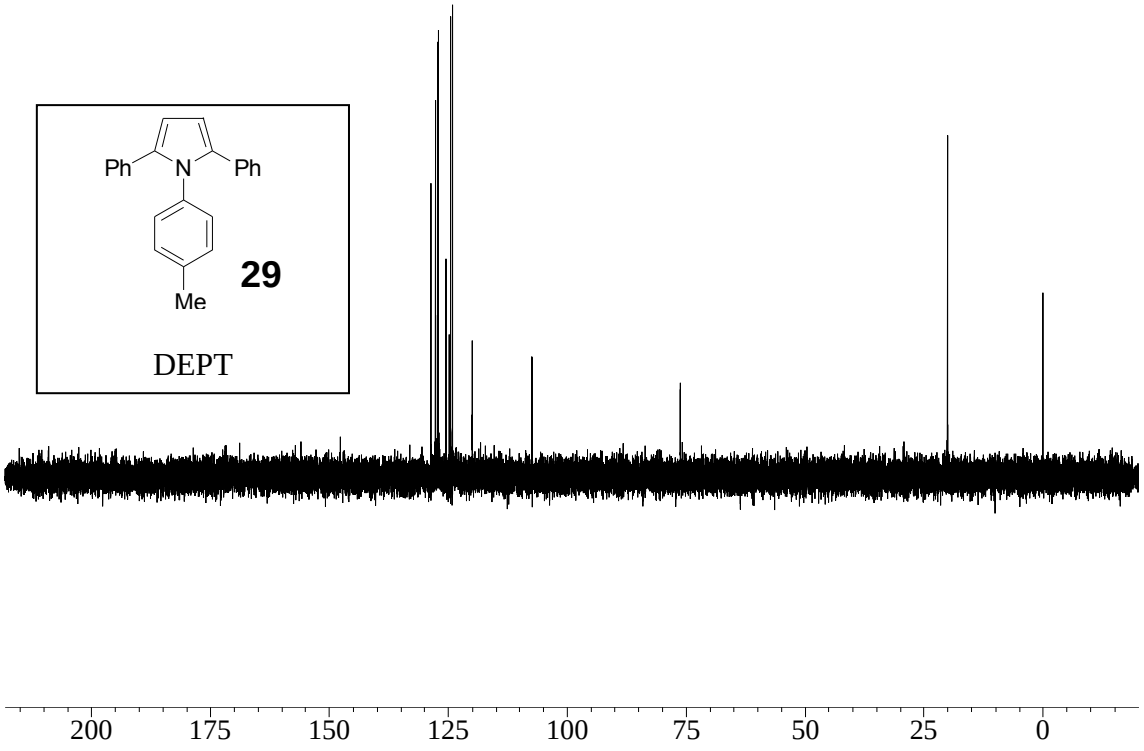

Ph
N
Ph
Me
26 or 27
down TLC
1H-NMR
10
9
8
7
6
5
4
3
2
1
0

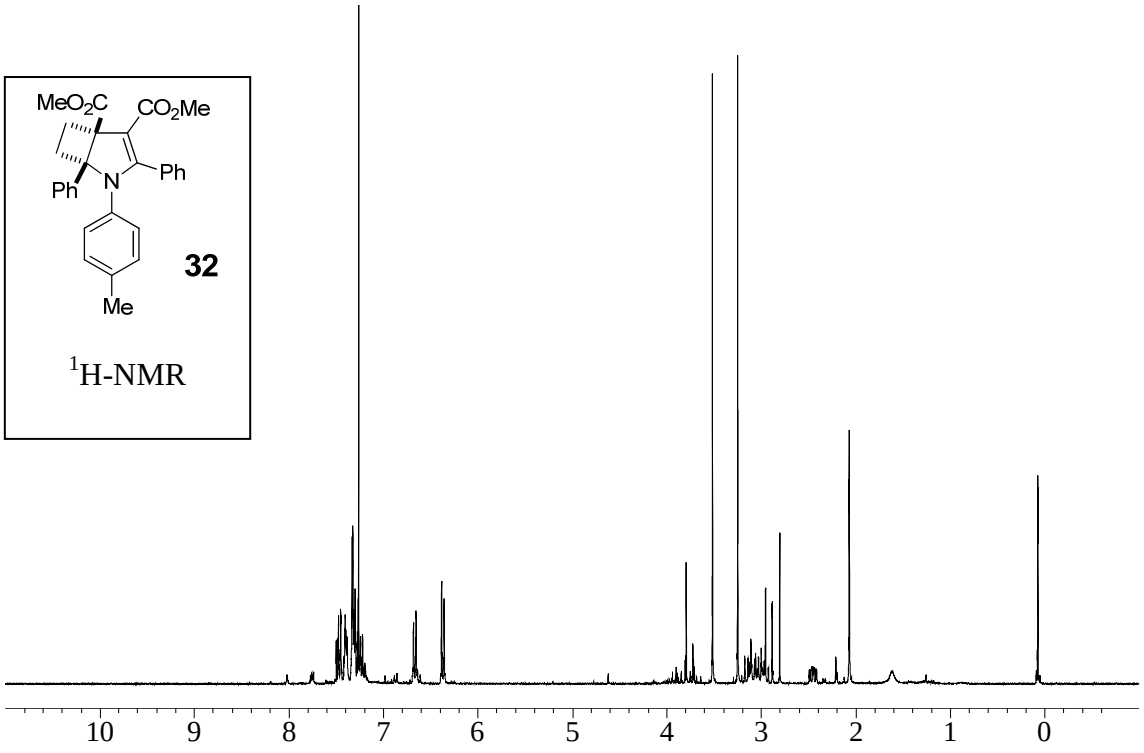

Ph
N
Ph
Me
26 or 27
down TLC
13C-NMR
200
175
150
125
100
75
50
25
0

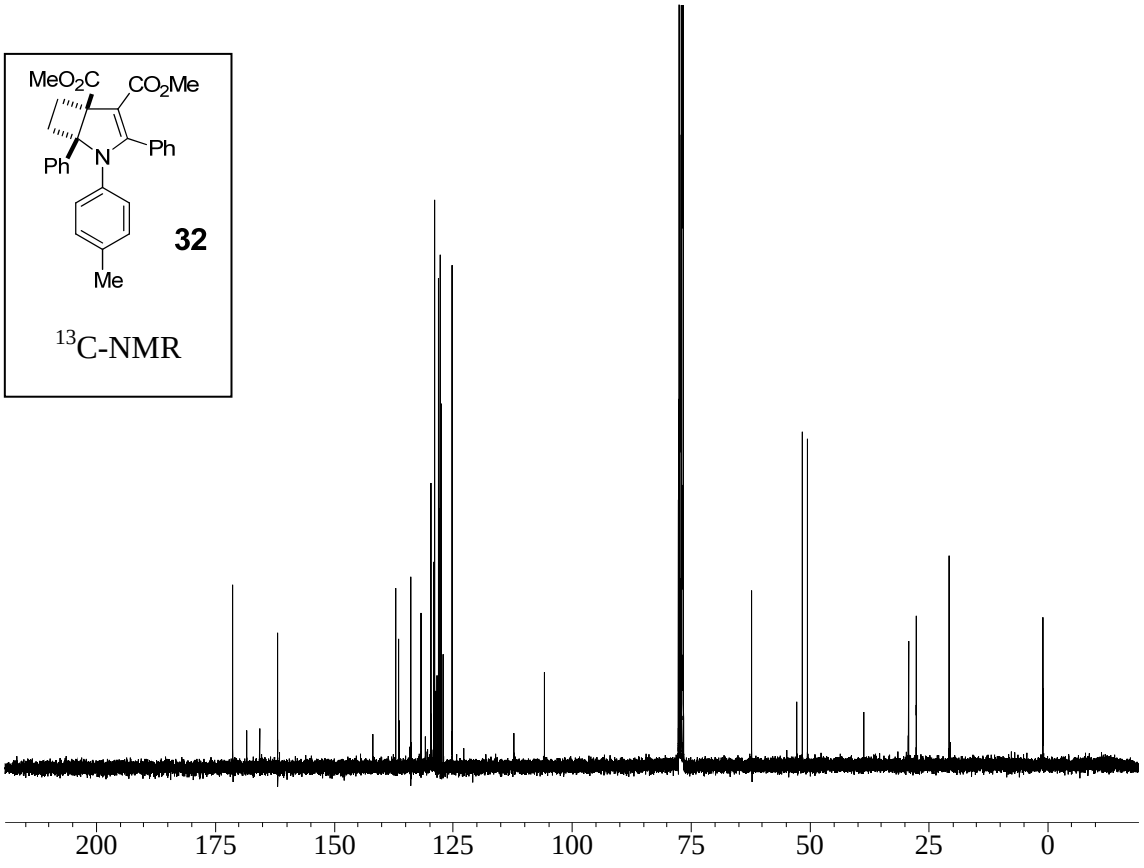

Ph
N
Ph
Me
26 or 27
down TLC
DEPT
200
175
150
125
100
75
50
25
0

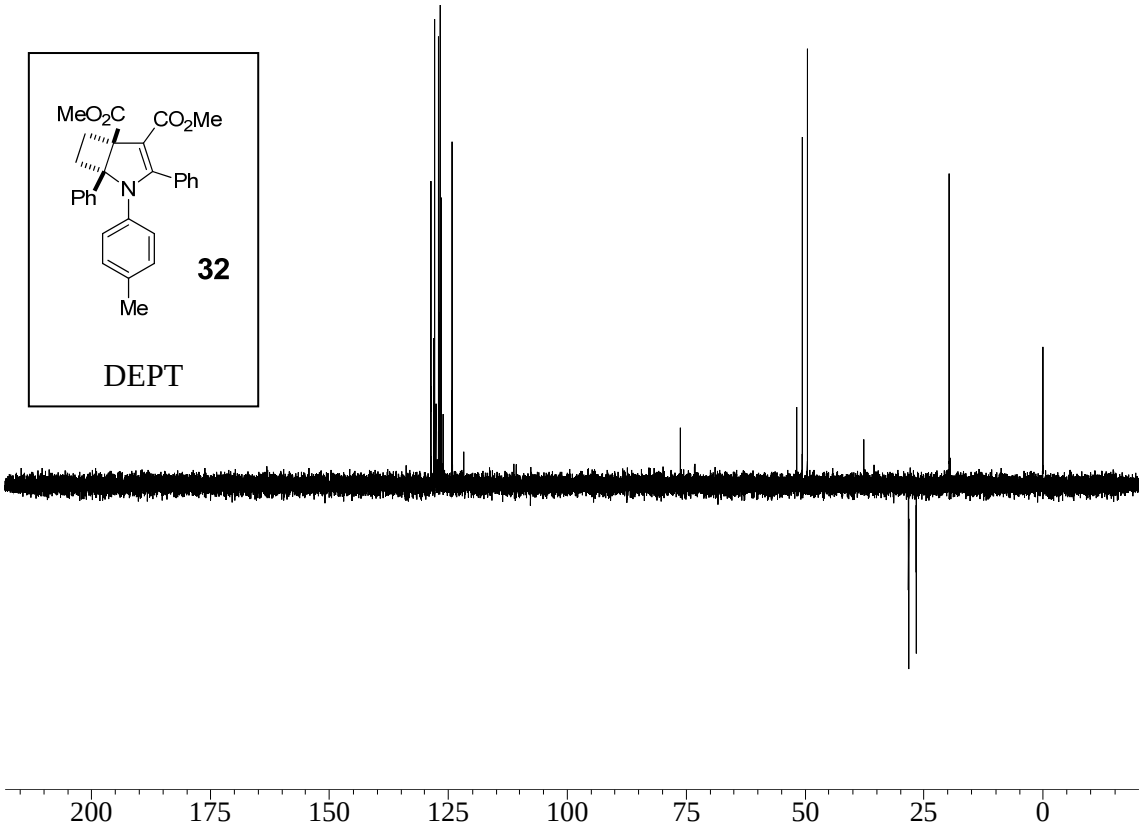

Ph
N
Ph
Me
29
1H-NMR
10
9
8
7
6
5
4
3
2
1
0

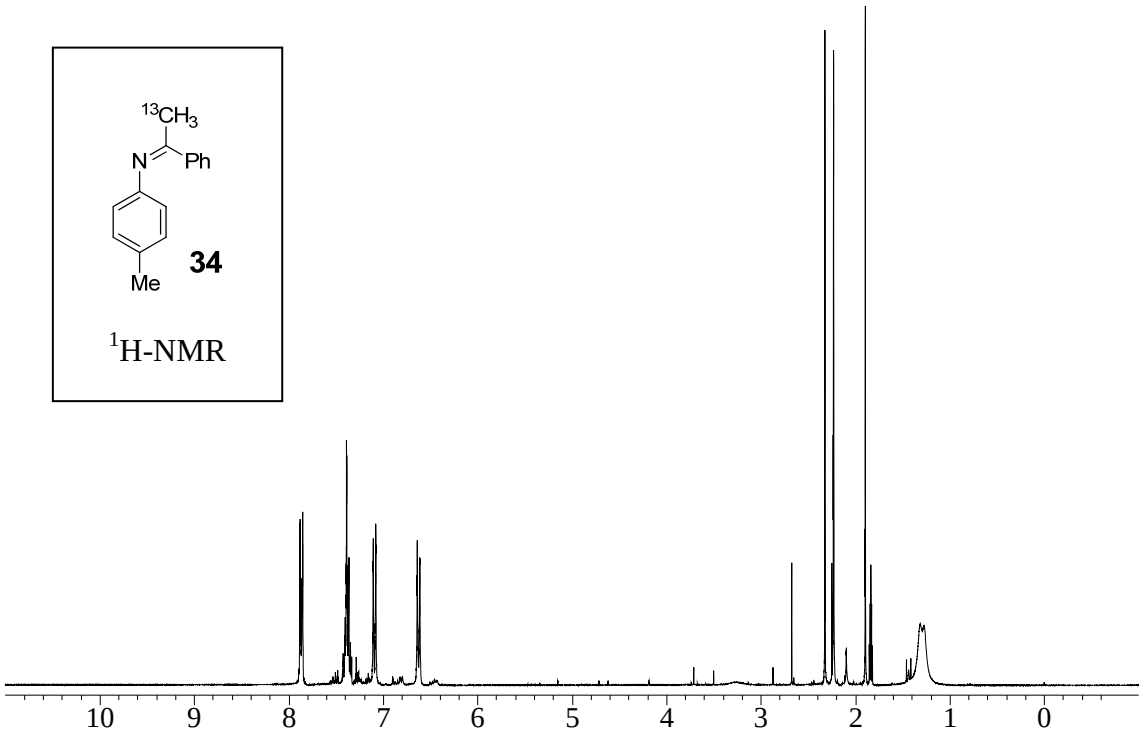

Ph
N
Ph
Me
29
^{13}C-NMR
200
175
150
125
100
75
50
25
0

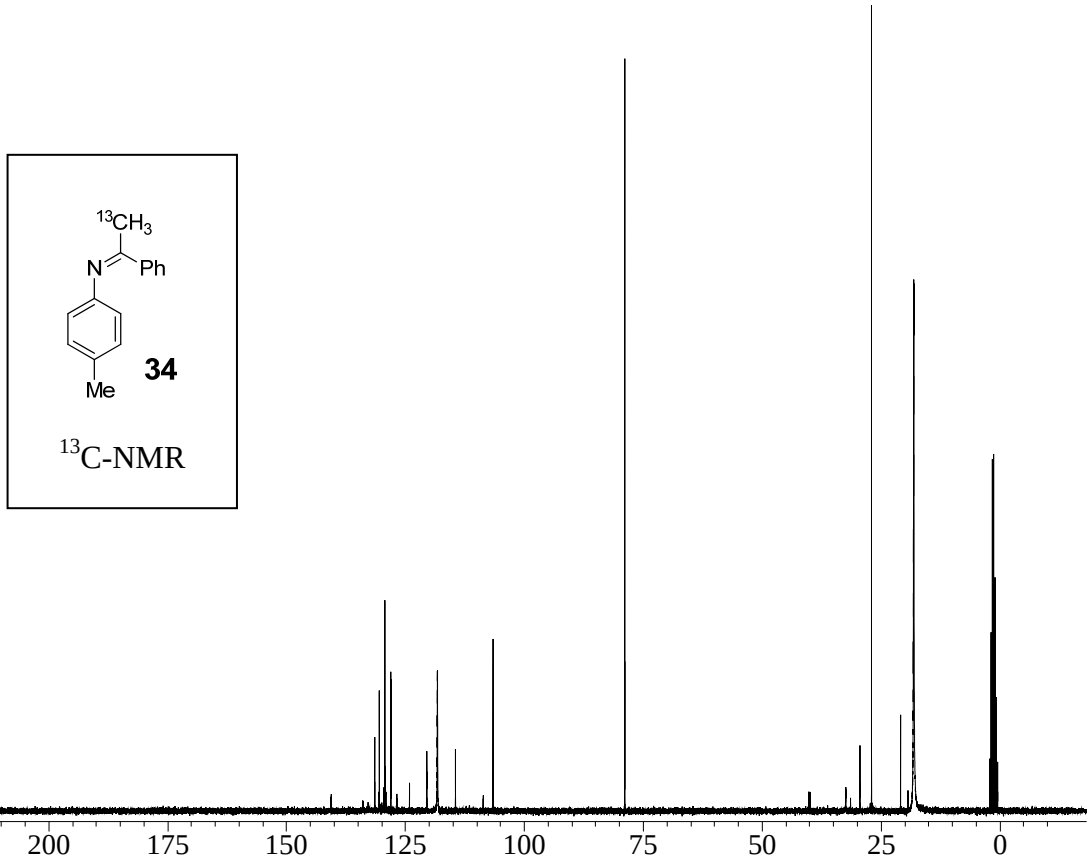

Ph
N
Ph
Me
29
DEPT
200
175
150
125
100
75
50
25
0

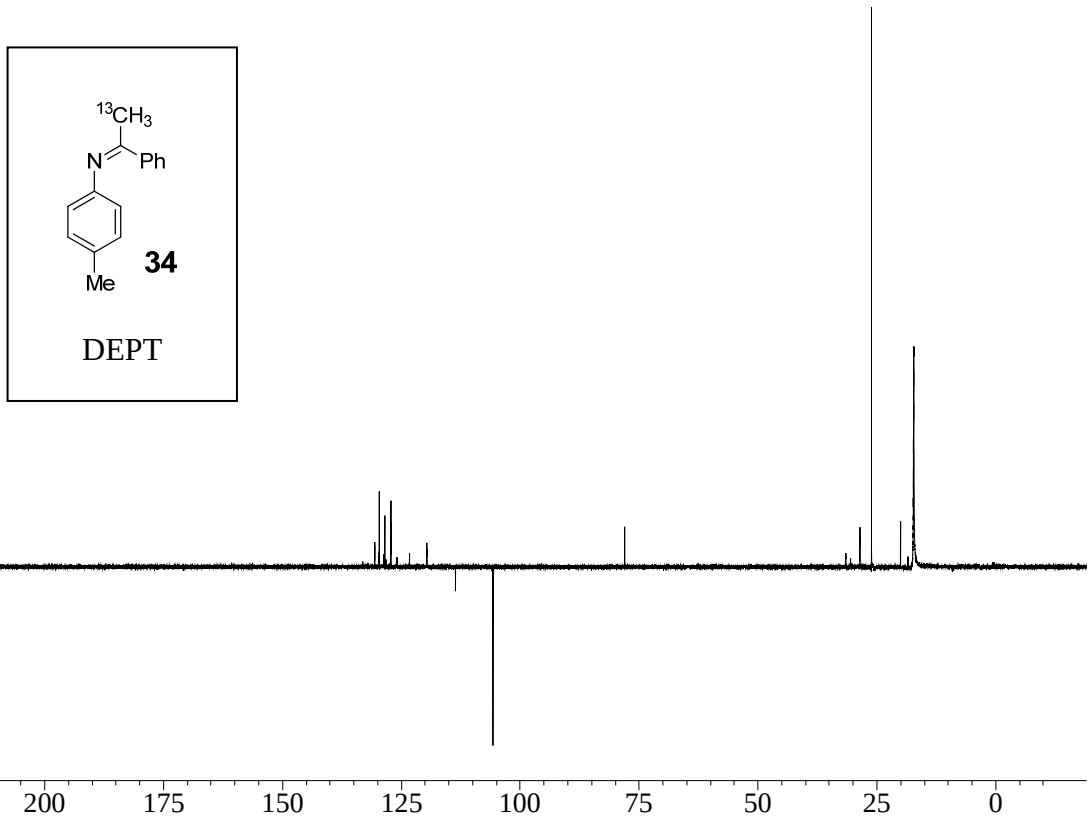

MeO2C
CO2Me
Ph
Ph
N
Me
32
1H-NMR
10
9
8
7
6
5
4
3
2
1
0

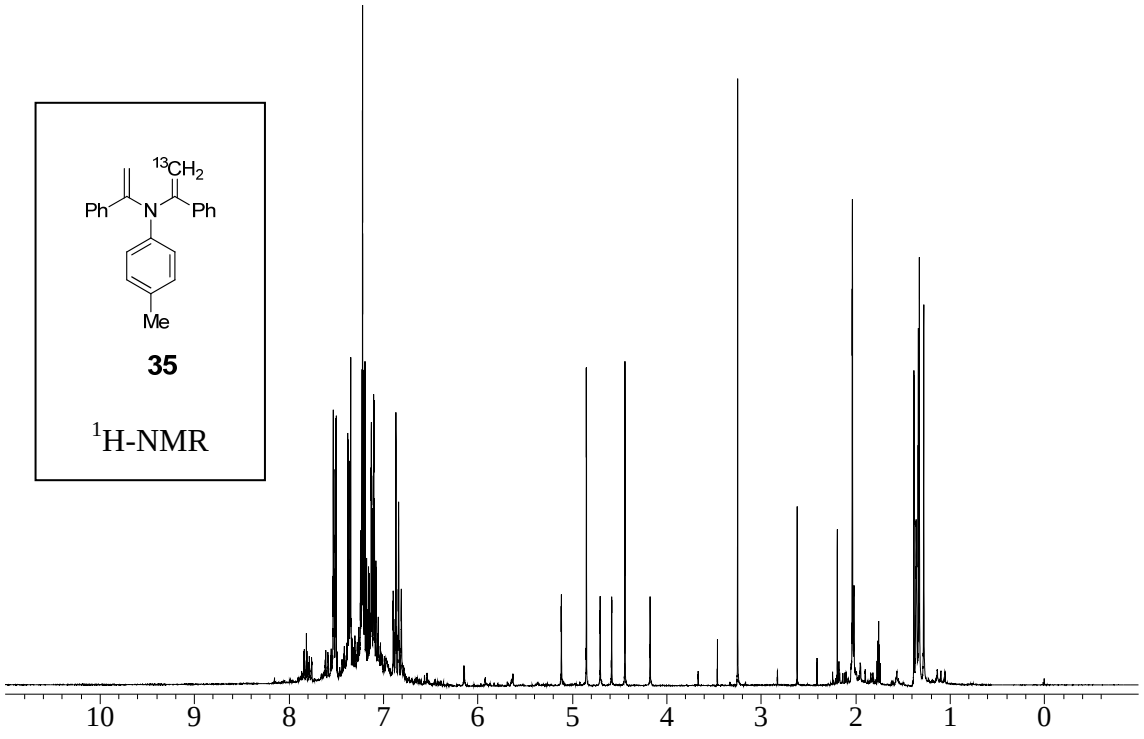

MeO2C
CO2Me
Ph
Ph
N
Me
32
13C-NMR
200
175
150
125
100
75
50
25
0

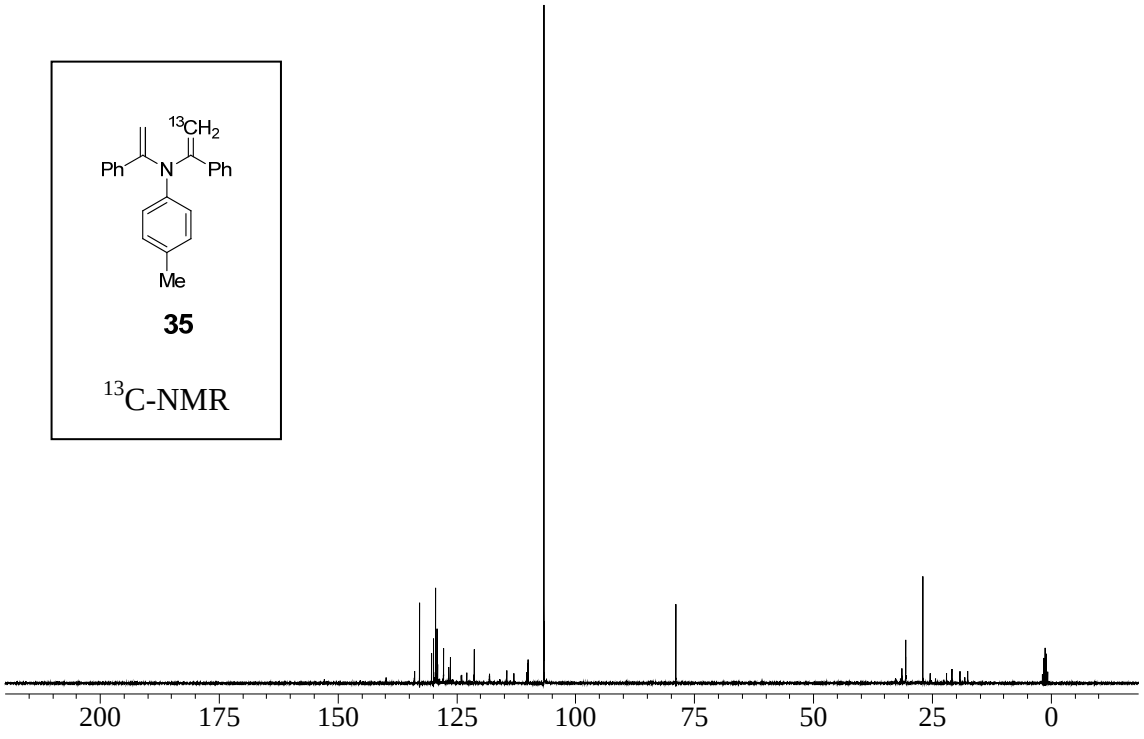

MeO2C
CO2Me
Ph
Ph
N
Me
32
DEPT
200
175
150
125
100
75
50
25
0

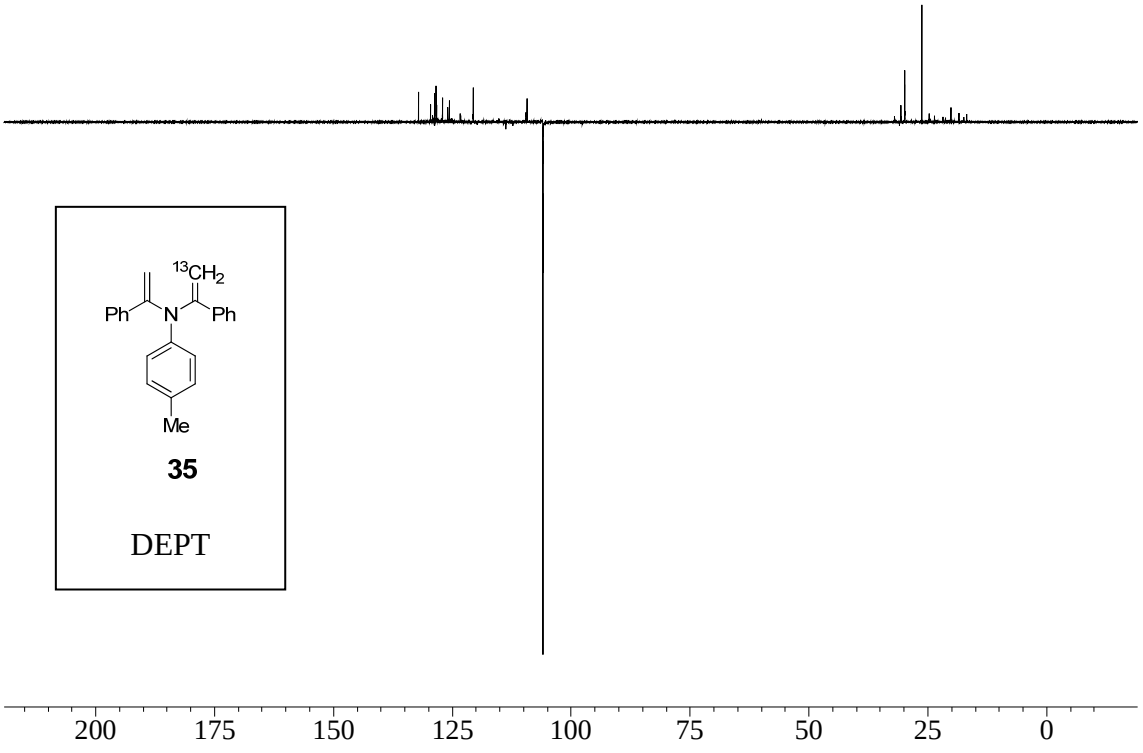

13CH3
N
Ph
Me
34
1H-NMR
10
9
8
7
6
5
4
3
2
1
0

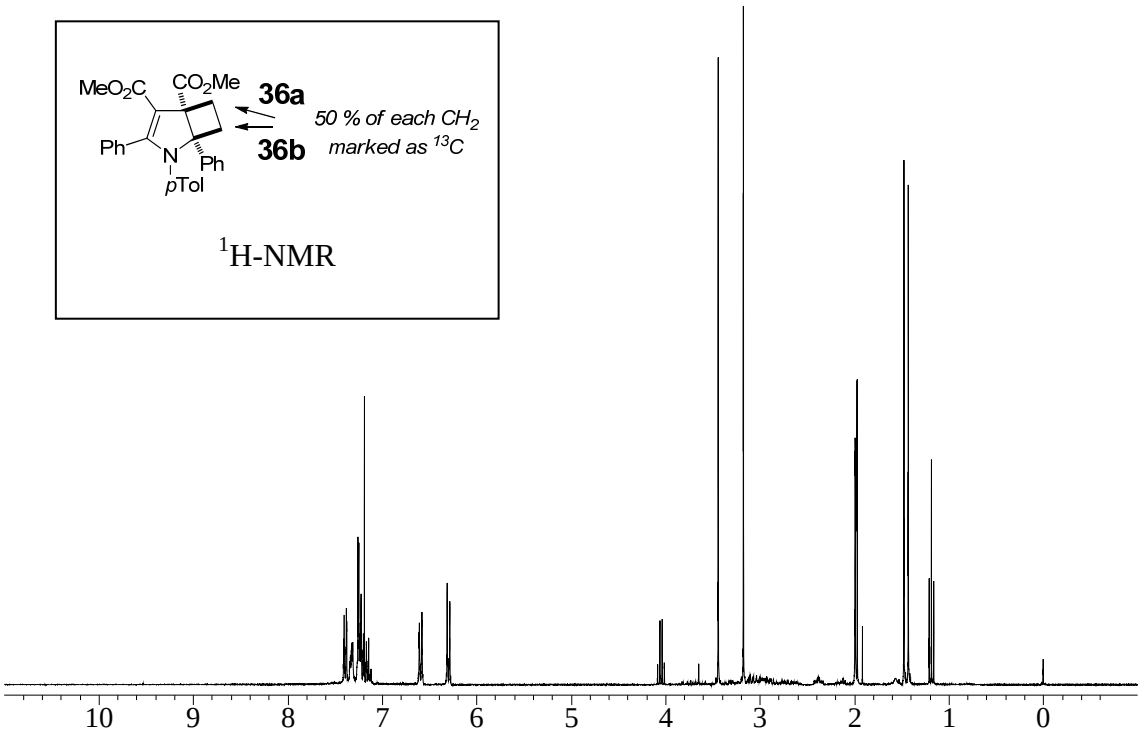

$^{13}CH_3$
N
Ph
Me
34
^{13}C-NMR
200
175
150
125
100
75
50
25
0

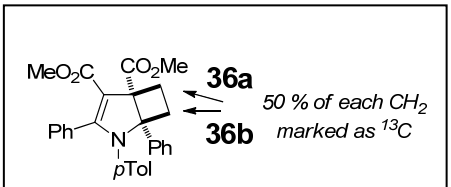

$^{13}CH_3$
N
Ph
Me
34
DEPT
200
175
150
125
100
75
50
25
0


13CH2
Ph
N
Ph
Me
35
1H-NMR
10
9
8
7
6
5
4
3
2
1
0


13CH2
Ph
N
Ph
Me
35
13C-NMR
200
175
150
125
100
75
50
25
0


13CH2
Ph
N
Ph
Me
35
DEPT
200
175
150
125
100
75
50
25
0


MeO2C
CO2Me
Ph
N
Ph
pTol
36a
36b
50 % of each CH2
marked as 13C
1H-NMR
10
9
8
7
6
5
4
3
2
1
0

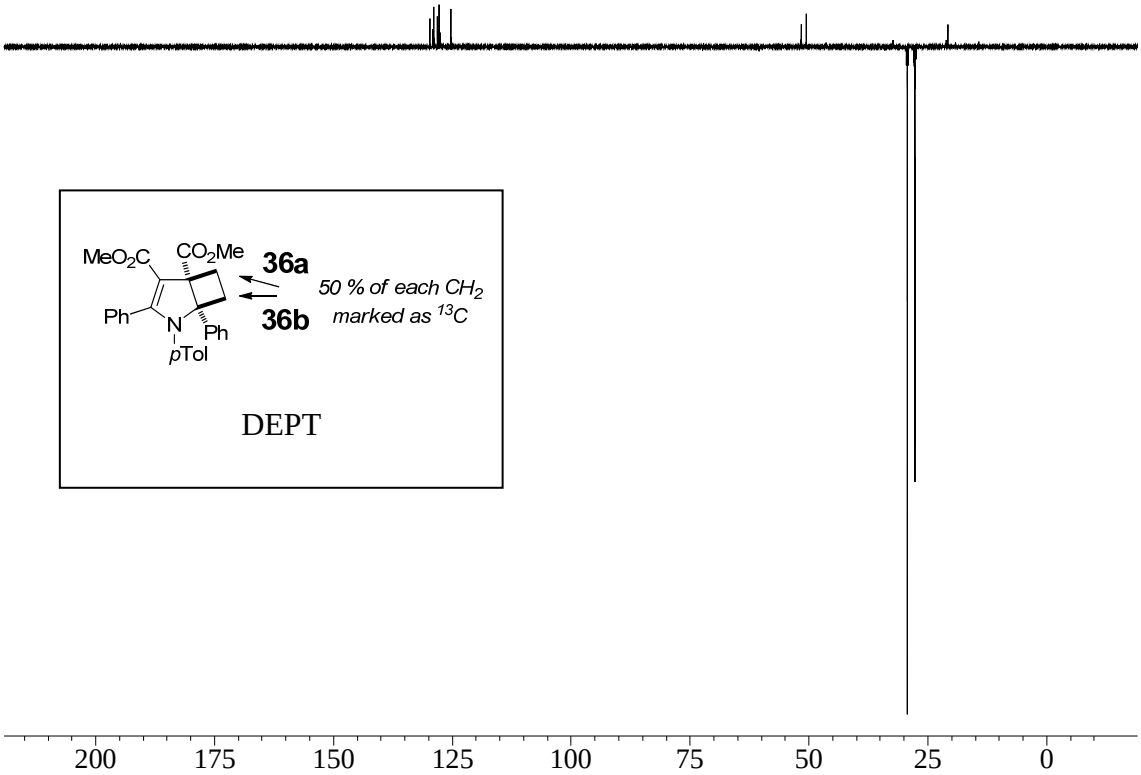


36a

36b

50 % of each CH_2 marked as ^{13}C

^{13}C-NMR

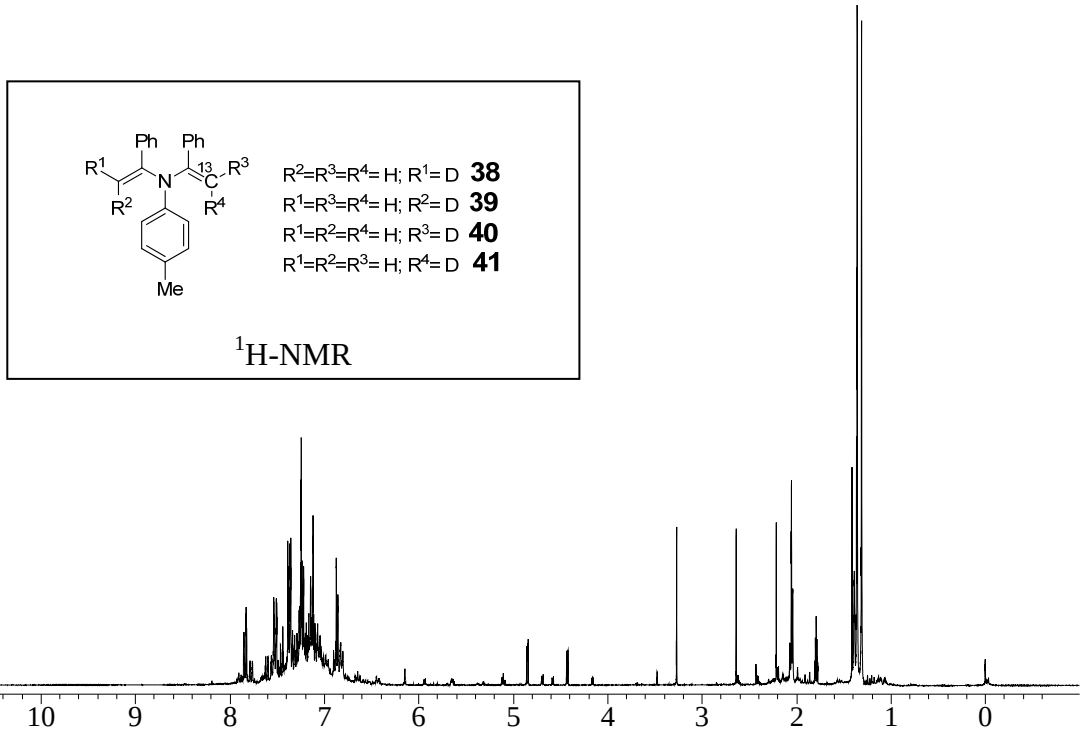


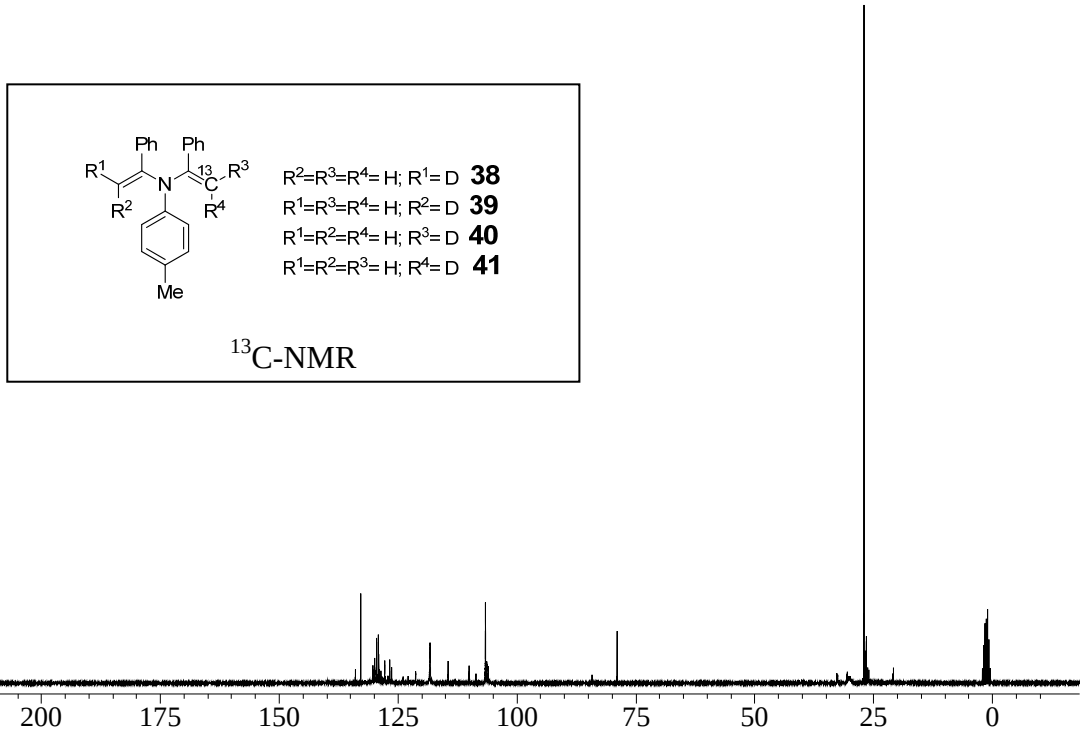
36a

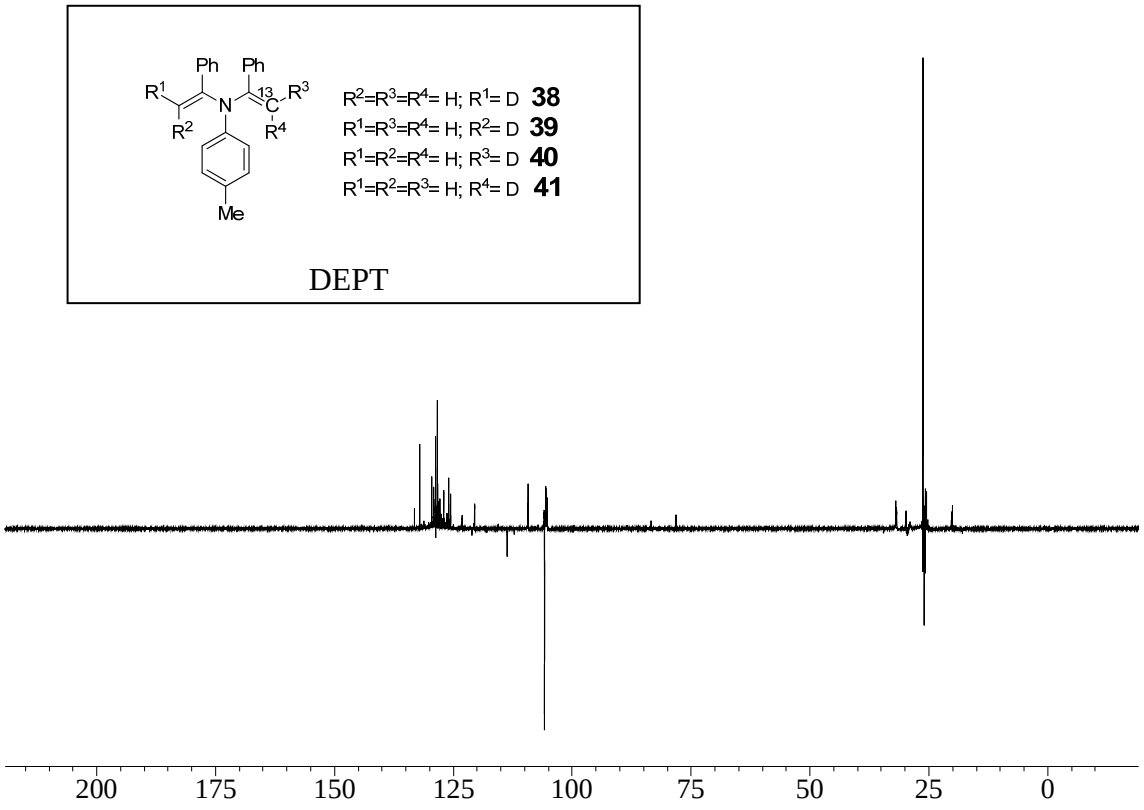
36b

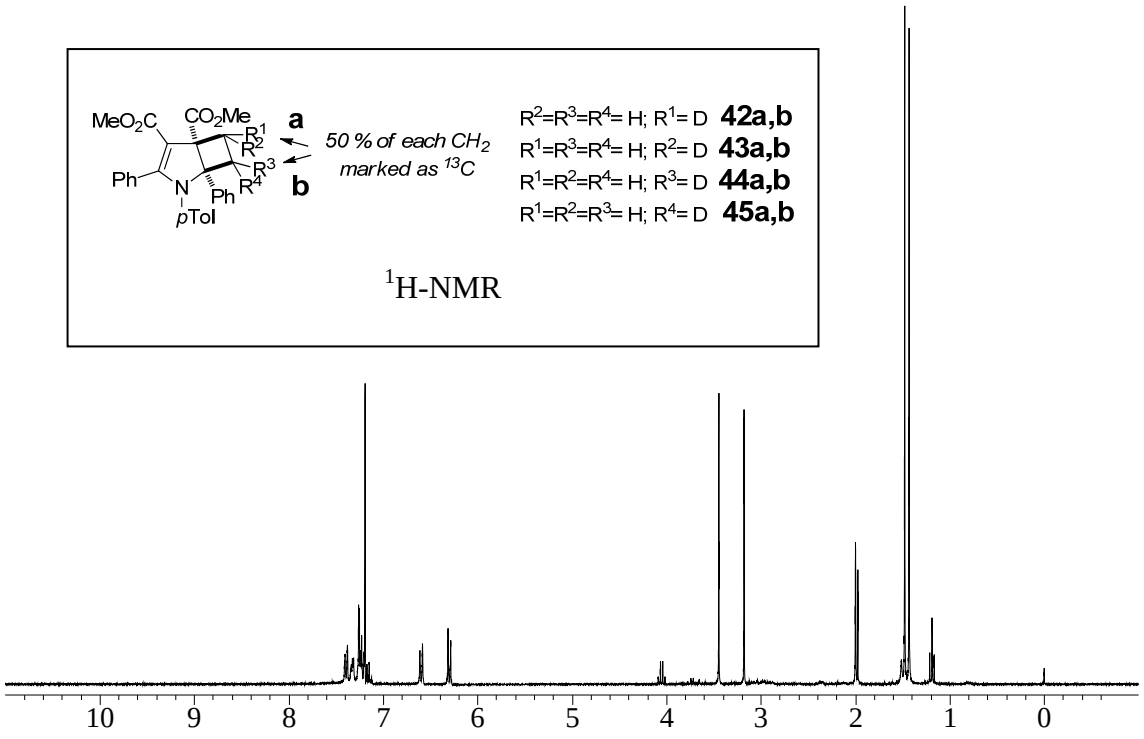
50 % of each CH_2 marked as ^{13}C

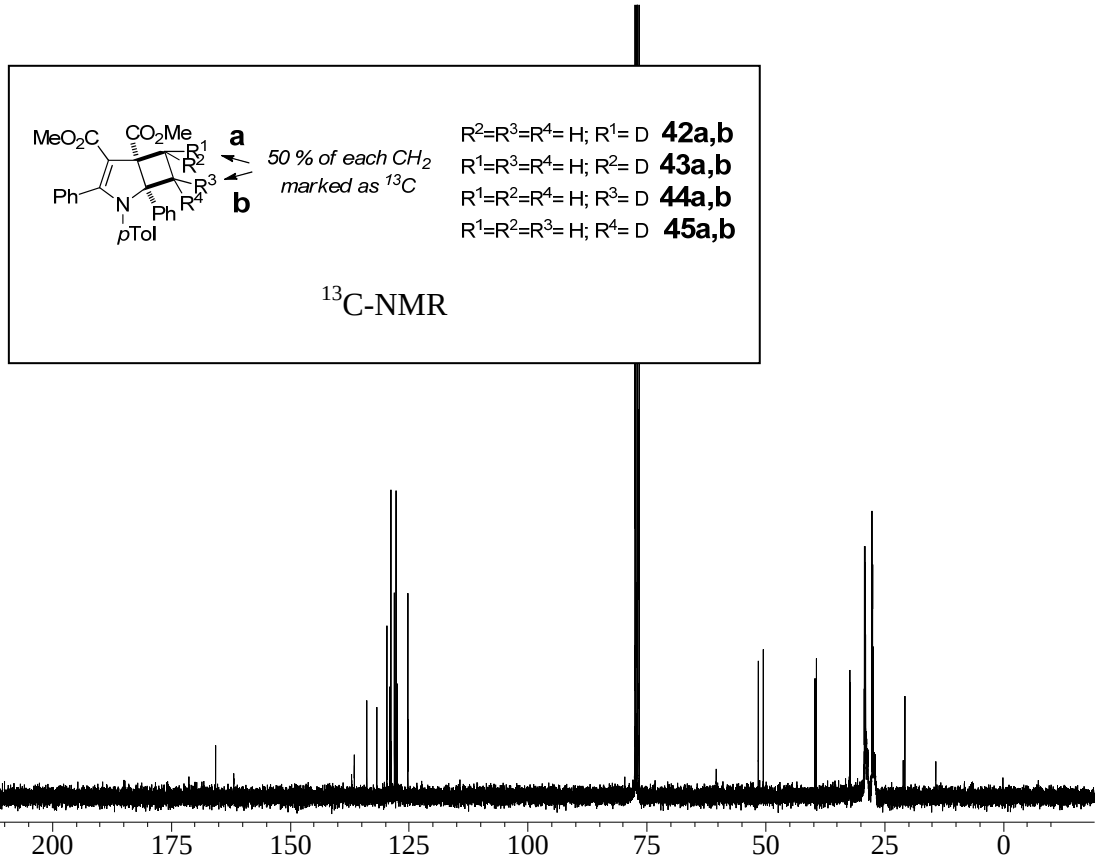
DEPT

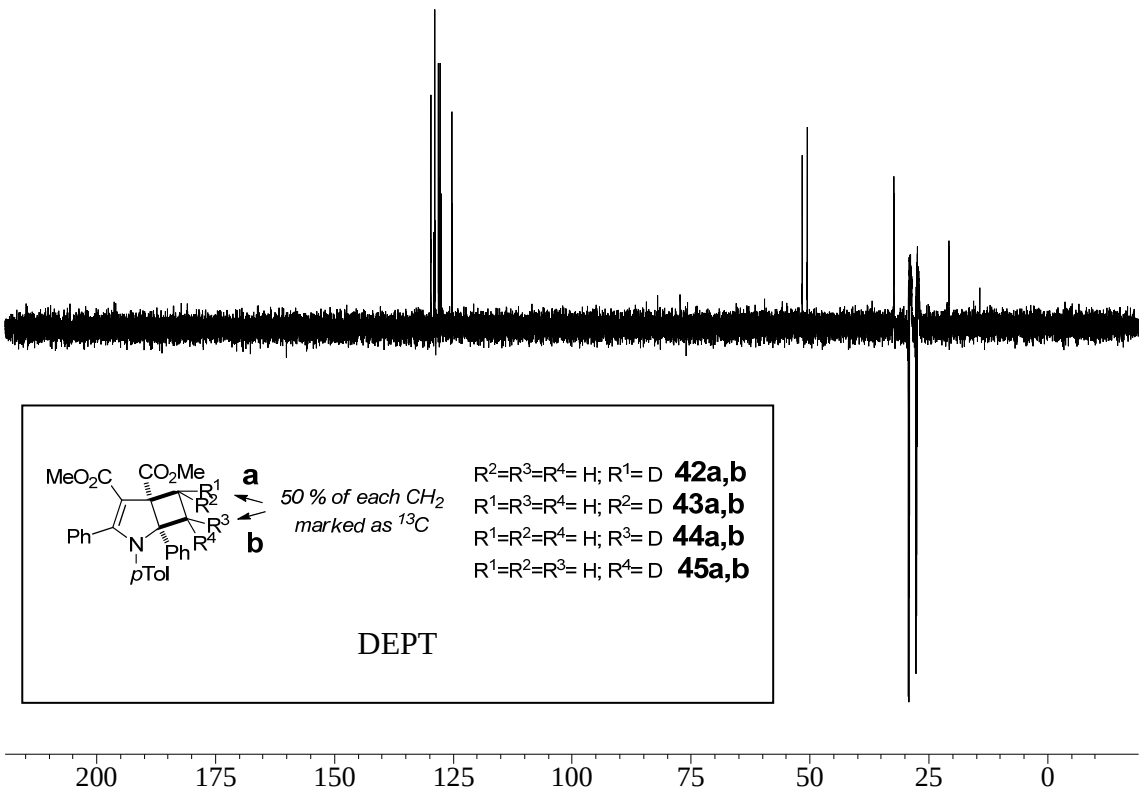

Ph
Ph
R1
N
13C
R3
R2
R4
Me
R2=R3=R4= H; R1= D 38
R1=R3=R4= H; R2= D 39
R1=R2=R4= H; R3= D 40
R1=R2=R3= H; R4= D 41
1H-NMR
10
9
8
7
6
5
4
3
2
1
0


Ph
Ph
R1
N
13C
R3
R2
R4
Me
R2=R3=R4= H; R1= D 38
R1=R3=R4= H; R2= D 39
R1=R2=R4= H; R3= D 40
R1=R2=R3= H; R4= D 41
13C-NMR
200
175
150
125
100
75
50
25
0


Ph
Ph
R1
N
13C
R3
R2
R4
Me
R2=R3=R4= H; R1= D 38
R1=R3=R4= H; R2= D 39
R1=R2=R4= H; R3= D 40
R1=R2=R3= H; R4= D 41
DEPT
200
175
150
125
100
75
50
25
0

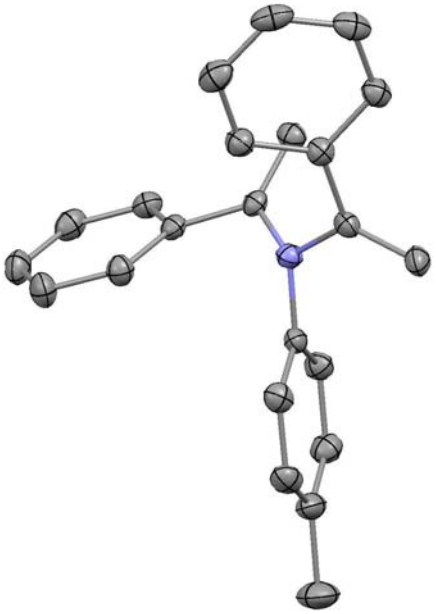

MeO2C
CO2Me
R1
R2
R3
R4
Ph
N
Ph
pTol
a
b
50 % of each CH2 marked as 13C
R2=R3=R4= H; R1= D 42a,b
R1=R3=R4= H; R2= D 43a,b
R1=R2=R4= H; R3= D 44a,b
R1=R2=R3= H; R4= D 45a,b
1H-NMR
10
9
8
7
6
5
4
3
2
1
0


MeO2C
CO2Me
R1
R2
R3
R4
Ph
N
Ph
pTol
a
b
50 % of each CH2 marked as 13C
R2=R3=R4= H; R1= D 42a,b
R1=R3=R4= H; R2= D 43a,b
R1=R2=R4= H; R3= D 44a,b
R1=R2=R3= H; R4= D 45a,b
13C-NMR
200
175
150
125
100
75
50
25
0


MeO2C
CO2Me
R1
R2
R3
R4
Ph
N
Ph
pTol
a
b
50 % of each CH2 marked as 13C
R2=R3=R4= H; R1= D 42a,b
R1=R3=R4= H; R2= D 43a,b
R1=R2=R4= H; R3= D 44a,b
R1=R2=R3= H; R4= D 45a,b
DEPT
200
175
150
125
100
75
50
25
0

DRX data

Compound 6.



Computing details
Data collection: *APEX2* v2009-3.0 (BRUKER AXS, Madison, 2009); cell refinement: *APEX2* v2009-3.0 (BRUKER AXS,
Madison, 2009); data reduction: *APEX2* v2009-3.0 (BRUKER AXS, Madison, 2009); program(s) used to solve structure:
SIR97 (Altomare *et al.*, 1998); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics:
ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines
(Farrugia, 1999).

(cc04zg1n)
Crystal data
$C_{23}H_{21}N_1$ F_{000} = 664
M_r = 311.41 D_x = 1.152 Mg m−3
Monoclinic, *Ia* Mo *K*α radiation, λ = 0.71073 Å
Hall symbol: I -2ya Cell parameters from 5078 reflections
a = 10.5194 (3) Å θ = 2.2–25.8º
b = 12.4510 (3) Å μ = 0.07 mm−1
c = 13.8791 (5) Å *T* = 100 K
β = 99.0690 (10)º Prism, colourless
V = 1795.12 (9) Å3 0.27 × 0.16 × 0.12 mm
Z = 4
Data collection
Bruker X8 kappa APEXII CCD
diffractometer 1827 reflections with $I > 2\sigma(I)$
Monochromator: graphite R_{int} = 0.032
T = 100 K θ_{max} = 27.1º
ω and phi scans θ_{min} = 2.2º
Absorption correction: multi-scan
BRUKER AXS - SADABS *h* = −13→13
T_{min} = 0.907, T_{max} = 1.000 *k* = 0→15

13971 measured reflections l = 0→17
1976 independent reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.032 H-atom parameters constrained
$wR(F^2)$ = 0.075
$w = 1/[\sigma^2(F_o^2) + (0.0416P)^2 + 0.4118P]$
where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07 $(\Delta/\sigma)_{max} < 0.001$
1976 reflections $\Delta\rho_{max}$ = 0.13 e Å^{-3}
218 parameters $\Delta\rho_{min}$ = −0.17 e Å^{-3}
2 restraints Extinction correction: none
Primary atom site location: structure-invariant direct methods

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The
cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between
s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is
used for estimating s.u.'s involving l.s. planes.
Refinement. 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 Rfactors(
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 (Å2)

x y z U_{iso}*/U_{eq}

N1 0.22069 (13) 0.23677 (12) 0.34649 (11) 0.0197 (3)
C11 0.33773 (16) 0.23039 (14) 0.30543 (12) 0.0187 (4)
C12 0.35221 (17) 0.15690 (16) 0.23281 (13) 0.0236 (4)
H12 0.2849 0.1079 0.2103 0.028*
C13 0.46543 (19) 0.15526 (17) 0.19320 (14) 0.0274 (4)
H13 0.4746 0.1046 0.1435 0.033*
C14 0.56566 (19) 0.22605 (17) 0.22460 (16) 0.0288 (4)
C15 0.55005 (18) 0.29782 (15) 0.29842 (15) 0.0271 (4)
H15 0.6179 0.3461 0.3217 0.033*
C16 0.43794 (18) 0.30061 (15) 0.33891 (14) 0.0244 (4)
H16 0.4294 0.3503 0.3894 0.029*
C17 0.6887 (2) 0.2221 (2) 0.1820 (2) 0.0453 (6)
H17A 0.7256 0.2944 0.1822 0.068*
H17B 0.6705 0.1953 0.1148 0.068*
H17C 0.7501 0.1741 0.2212 0.068*
C21 0.14433 (16) 0.33041 (15) 0.32517 (13) 0.0199 (4)
C22 0.15445 (18) 0.39431 (16) 0.24943 (13) 0.0254 (4)
H22A 0.2146 0.3778 0.2072 0.03*
H22B 0.1015 0.4562 0.2378 0.03*
C23 0.05975 (17) 0.35826 (14) 0.39793 (13) 0.0210 (4)
C24 0.09966 (18) 0.33839 (16) 0.49729 (14) 0.0254 (4)
H24 0.18 0.3042 0.5183 0.03*
C25 0.0230 (2) 0.36811 (18) 0.56529 (14) 0.0306 (5)
H25 0.0511 0.3545 0.6326 0.037*
C26 −0.09462 (19) 0.41772 (16) 0.53535 (16) 0.0306 (5)
H26 −0.1471 0.438 0.5821 0.037*
C27 −0.13532 (19) 0.43756 (16) 0.43781 (16) 0.0290 (4)
H27 −0.216 0.4714 0.4174 0.035*

C28 −0.05900 (18) 0.40827 (15) 0.36912 (14) 0.0246 (4)
H28 −0.0878 0.4224 0.302 0.029*
C31 0.15947 (17) 0.13858 (14) 0.36555 (12) 0.0185 (4)
C32 0.03549 (17) 0.12061 (16) 0.33390 (14) 0.0224 (4)
H32A −0.0154 0.1741 0.2973 0.027*
H32B −0.0023 0.0543 0.3479 0.027*
C33 0.24294 (16) 0.06138 (15) 0.42843 (12) 0.0192 (4)
C34 0.34444 (17) 0.09678 (16) 0.49793 (14) 0.0241 (4)
H34 0.363 0.1714 0.5041 0.029*
C35 0.41879 (19) 0.02409 (17) 0.55832 (14) 0.0291 (4)
H35 0.4875 0.0492 0.6057 0.035*
C36 0.3932 (2) −0.08500 (17) 0.54968 (14) 0.0293 (4)
H36 0.4439 −0.1346 0.5912 0.035*
C37 0.29347 (19) −0.12123 (16) 0.48026 (15) 0.0274 (4)
H37 0.2763 −0.196 0.4739 0.033*
C38 0.21858 (17) −0.04908 (15) 0.42003 (14) 0.0226 (4)
H38 0.1502 −0.0747 0.3727 0.027*

Atomic displacement parameters (Å2)

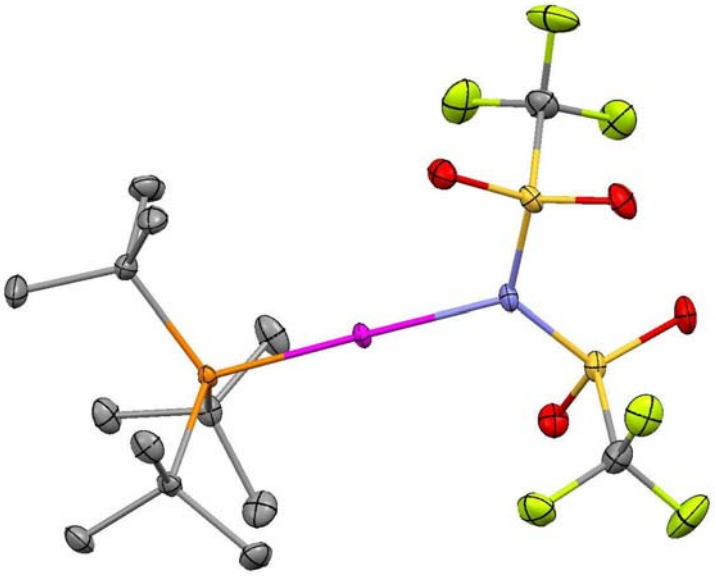
*U*11 *U*22 *U*33 *U*12 *U*13 *U*23
N1 0.0176 (7) 0.0197 (8) 0.0223 (8) 0.0017 (6) 0.0046 (6) 0.0008 (6)
C11 0.0164 (8) 0.0191 (9) 0.0205 (8) 0.0020 (6) 0.0024 (7) 0.0033 (7)
C12 0.0227 (9) 0.0245 (10) 0.0234 (9) −0.0021 (7) 0.0033 (7) −0.0012 (8)
C13 0.0297 (10) 0.0270 (11) 0.0270 (10) 0.0021 (8) 0.0088 (8) −0.0010 (8)
C14 0.0237 (9) 0.0304 (11) 0.0341 (11) 0.0030 (8) 0.0105 (8) 0.0090 (9)
C15 0.0222 (9) 0.0209 (9) 0.0374 (11) −0.0044 (8) 0.0024 (8) 0.0036 (8)
C16 0.0247 (9) 0.0185 (9) 0.0300 (10) 0.0007 (7) 0.0039 (8) 0.0002 (8)
C17 0.0295 (12) 0.0553 (16) 0.0558 (16) 0.0004 (11) 0.0212 (11) 0.0061 (12)
C21 0.0176 (8) 0.0216 (9) 0.0196 (8) 0.0015 (7) −0.0001 (7) −0.0011 (7)
C22 0.0252 (9) 0.0287 (10) 0.0217 (9) 0.0073 (8) 0.0022 (7) 0.0046 (8)
C23 0.0221 (9) 0.0176 (9) 0.0231 (9) 0.0000 (7) 0.0030 (7) −0.0009 (7)
C24 0.0242 (10) 0.0275 (10) 0.0243 (9) 0.0005 (8) 0.0037 (7) −0.0013 (8)
C25 0.0363 (11) 0.0318 (11) 0.0250 (10) −0.0066 (9) 0.0089 (8) −0.0057 (8)
C26 0.0317 (11) 0.0235 (10) 0.0412 (12) −0.0081 (8) 0.0204 (9) −0.0097 (9)
C27 0.0230 (9) 0.0184 (9) 0.0469 (12) 0.0016 (7) 0.0088 (9) −0.0032 (8)
C28 0.0232 (9) 0.0200 (9) 0.0302 (10) 0.0020 (8) 0.0032 (8) 0.0014 (8)
C31 0.0215 (8) 0.0184 (9) 0.0164 (8) 0.0004 (7) 0.0055 (7) −0.0006 (7)
C32 0.0202 (8) 0.0238 (9) 0.0235 (9) 0.0010 (7) 0.0040 (7) −0.0024 (7)
C33 0.0207 (9) 0.0202 (9) 0.0177 (8) 0.0003 (7) 0.0064 (7) 0.0002 (7)
C34 0.0259 (10) 0.0214 (10) 0.0244 (9) 0.0001 (7) 0.0021 (8) 0.0008 (8)
C35 0.0298 (11) 0.0299 (10) 0.0250 (10) 0.0016 (8) −0.0040 (8) 0.0028 (8)
C36 0.0345 (11) 0.0270 (10) 0.0261 (10) 0.0072 (8) 0.0041 (9) 0.0074 (8)
C37 0.0338 (11) 0.0195 (10) 0.0305 (10) 0.0005 (8) 0.0106 (8) 0.0019 (8)
C38 0.0222 (9) 0.0226 (10) 0.0237 (9) −0.0024 (7) 0.0061 (7) −0.0006 (7)

Geometric parameters (Å, °)

N1—C21 1.420 (2) C24—H24 0.95
N1—C31 1.426 (2) C25—C26 1.387 (3)
N1—C11 1.438 (2) C25—H25 0.95
C11—C12 1.387 (3) C26—C27 1.376 (3)
C11—C16 1.392 (3) C26—H26 0.95
C12—C13 1.388 (3) C27—C28 1.388 (3)
C12—H12 0.95 C27—H27 0.95
C13—C14 1.391 (3) C28—H28 0.95
C13—H13 0.95 C31—C32 1.328 (3)
C14—C15 1.389 (3) C31—C33 1.488 (2)
C14—C17 1.507 (3) C32—H32A 0.95
C15—C16 1.384 (3) C32—H32B 0.95
C15—H15 0.95 C33—C34 1.393 (2)
C16—H16 0.95 C33—C38 1.400 (3)
C17—H17A 0.98 C34—C35 1.388 (3)
C17—H17B 0.98 C34—H34 0.95
C17—H17C 0.98 C35—C36 1.386 (3)
C21—C22 1.336 (3) C35—H35 0.95
C21—C23 1.488 (2) C36—C37 1.383 (3)
C22—H22A 0.95 C36—H36 0.95
C22—H22B 0.95 C37—C38 1.385 (3)
C23—C28 1.397 (3) C37—H37 0.95
C23—C24 1.399 (3) C38—H38 0.95

C24—C25 1.385 (3)
C21—N1—C31 119.04 (14) C23—C24—H24 119.7
C21—N1—C11 117.01 (14) C24—C25—C26 120.18 (19)
C31—N1—C11 117.78 (14) C24—C25—H25 119.9
C12—C11—C16 119.52 (16) C26—C25—H25 119.9
C12—C11—N1 121.54 (16) C27—C26—C25 119.94 (18)
C16—C11—N1 118.93 (16) C27—C26—H26 120
C11—C12—C13 119.78 (17) C25—C26—H26 120
C11—C12—H12 120.1 C26—C27—C28 120.29 (18)
C13—C12—H12 120.1 C26—C27—H27 119.9
C12—C13—C14 121.46 (19) C28—C27—H27 119.9
C12—C13—H13 119.3 C27—C28—C23 120.56 (18)
C14—C13—H13 119.3 C27—C28—H28 119.7
C15—C14—C13 117.85 (18) C23—C28—H28 119.7
C15—C14—C17 121.30 (19) C32—C31—N1 122.13 (16)
C13—C14—C17 120.8 (2) C32—C31—C33 122.89 (17)
C16—C15—C14 121.54 (18) N1—C31—C33 114.87 (15)
C16—C15—H15 119.2 C31—C32—H32A 120
C14—C15—H15 119.2 C31—C32—H32B 120
C15—C16—C11 119.83 (18) H32A—C32—H32B 120
C15—C16—H16 120.1 C34—C33—C38 118.59 (17)
C11—C16—H16 120.1 C34—C33—C31 121.25 (16)
C14—C17—H17A 109.5 C38—C33—C31 120.14 (16)
C14—C17—H17B 109.5 C35—C34—C33 120.62 (18)
H17A—C17—H17B 109.5 C35—C34—H34 119.7
C14—C17—H17C 109.5 C33—C34—H34 119.7
H17A—C17—H17C 109.5 C36—C35—C34 120.22 (18)
H17B—C17—H17C 109.5 C36—C35—H35 119.9
C22—C21—N1 122.70 (17) C34—C35—H35 119.9
C22—C21—C23 121.70 (17) C37—C36—C35 119.71 (18)
N1—C21—C23 115.39 (15) C37—C36—H36 120.1
C21—C22—H22A 120 C35—C36—H36 120.1
C21—C22—H22B 120 C36—C37—C38 120.36 (19)
H22A—C22—H22B 120 C36—C37—H37 119.8
C28—C23—C24 118.49 (16) C38—C37—H37 119.8
C28—C23—C21 120.86 (16) C37—C38—C33 120.49 (18)
C24—C23—C21 120.60 (16) C37—C38—H38 119.8
C25—C24—C23 120.53 (18) C33—C38—H38 119.8
C25—C24—H24 119.7
C21—N1—C11—C12 −111.29 (19) C21—C23—C24—C25 177.51 (18)
C31—N1—C11—C12 40.9 (2) C23—C24—C25—C26 0.2 (3)
C21—N1—C11—C16 68.0 (2) C24—C25—C26—C27 0.0 (3)
C31—N1—C11—C16 −139.83 (17) C25—C26—C27—C28 −0.1 (3)
C16—C11—C12—C13 −1.0 (3) C26—C27—C28—C23 0.1 (3)
N1—C11—C12—C13 178.25 (17) C24—C23—C28—C27 0.1 (3)
C11—C12—C13—C14 −0.1 (3) C21—C23—C28—C27 −177.64 (17)
C12—C13—C14—C15 1.0 (3) C21—N1—C31—C32 22.8 (3)
C12—C13—C14—C17 179.0 (2) C11—N1—C31—C32 −128.79 (18)
C13—C14—C15—C16 −1.0 (3) C21—N1—C31—C33 −153.55 (15)
C17—C14—C15—C16 −179.0 (2) C11—N1—C31—C33 54.8 (2)
C14—C15—C16—C11 −0.1 (3) C32—C31—C33—C34 −147.64 (19)
C12—C11—C16—C15 1.1 (3) N1—C31—C33—C34 28.7 (2)
N1—C11—C16—C15 −178.19 (16) C32—C31—C33—C38 30.9 (3)
C31—N1—C21—C22 −131.63 (19) N1—C31—C33—C38 −152.73 (15)
C11—N1—C21—C22 20.2 (2) C38—C33—C34—C35 −0.8 (3)
C31—N1—C21—C23 53.5 (2) C31—C33—C34—C35 177.82 (17)
C11—N1—C21—C23 −154.71 (15) C33—C34—C35—C36 0.4 (3)
C22—C21—C23—C28 37.2 (3) C34—C35—C36—C37 0.3 (3)
N1—C21—C23—C28 −147.85 (16) C35—C36—C37—C38 −0.5 (3)
C22—C21—C23—C24 −140.47 (18) C36—C37—C38—C33 0.1 (3)
N1—C21—C23—C24 34.5 (2) C34—C33—C38—C37 0.5 (3)
C28—C23—C24—C25 −0.2 (3) C31—C33—C38—C37 −178.09 (16)

Compound 7c.



Computing details
Data collection: APPEX2 (BRUKER AXS, 2005); cell refinement: APPEX2 (BRUKER AXS, 2005); data reduction:
APPEX2 (BRUKER AXS, 2005); program(s) used to solve structure: *SIR97* (Giacovazzo *et al.*l, 1999; program(s) used to
refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used
to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

(cc03zg1n)
Crystal data
$C_{14}H_{27}AuF_6NO_4PS_2$ F_{000} = 2640
M_r = 679.45 D_x = 2.027 Mg m^{-3}
Monoclinic, *C*2/*c* Mo *K*α radiation, λ = 0.7107 Å
Hall symbol: -C 2yc Cell parameters from 9730 reflections
a = 26.6340 (5) Å θ = 4.5–72.6º
b = 11.4521 (2) Å μ = 6.94 mm^{-1}
c = 15.0970 (3) Å *T* = 100 K
β = 104.7820 (10)º Prism, colourless
V = 4452.41 (14) Å^3 0.39 × 0.25 × 0.17 mm
Z = 8
Data collection
BRUKER APPEX-II CCD
diffractometer 9034 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube R_{int} = 0.035
T = 100 K θ_{max} = 35.6º
ω and phi scans θ_{min} = 1.6º
Absorption correction: multi-scan
BRUKER SADABS *h* = −43→42
T_{min} = 0.589, T_{max} = 1 *k* = 0→18
121396 measured reflections *l* = 0→24
10271 independent reflections
Refinement
Refinement on F^2 Secondary atom site location: difference Fourier map
Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)]$ = 0.018 All H-atom parameters refined
$wR(F^2)$ = 0.037
$w = 1/[\sigma^2(F_o^2) + (0.0134P)^2 + 0.1582P]$
where $P = (F_o^2 + 2F_c^2)/3$
S = 1.08 $(\Delta/\sigma)_{max}$ = 0.001
10271 reflections $\Delta\rho_{max}$ = 0.68 e Å^{-3}
370 parameters $\Delta\rho_{min}$ = −1.43 e Å^{-3}
Primary atom site location: structure-invariant direct
methods Extinction correction: none

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The
cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between
s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is
used for estimating s.u.'s involving l.s. planes.
Refinement. 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 Rfactors(
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 (Å2)

x y z U_{iso}*/U_{eq}

Au1	0.120789 (2)	0.093982 (4)	0.070895 (4)	0.009380 (10)
N2	0.11487 (5)	0.27788 (11)	0.06303 (8)	0.0116 (2)
S3	0.104487 (14)	0.34073 (3)	−0.03670 (3)	0.01199 (6)
O4	0.08113 (5)	0.45257 (10)	−0.03972 (9)	0.0202 (2)
O5	0.08375 (5)	0.25348 (10)	−0.10381 (8)	0.0175 (2)
C6	0.17038 (7)	0.36560 (16)	−0.05046 (12)	0.0205 (3)
F7	0.19616 (4)	0.26570 (11)	−0.04288 (9)	0.0315 (3)
F8	0.16628 (5)	0.40930 (12)	−0.13322 (9)	0.0361 (3)
F9	0.19704 (4)	0.43931 (11)	0.01130 (9)	0.0297 (3)
S10	0.124776 (14)	0.35127 (3)	0.15817 (3)	0.01222 (6)
O11	0.15164 (5)	0.27583 (10)	0.22998 (8)	0.0181 (2)
O12	0.14258 (5)	0.46733 (10)	0.15076 (8)	0.0183 (2)
C13	0.05927 (6)	0.36564 (15)	0.17620 (12)	0.0185 (3)
F14	0.03983 (4)	0.26065 (10)	0.18564 (8)	0.0258 (2)
F15	0.06287 (5)	0.42567 (12)	0.25277 (9)	0.0339 (3)
F16	0.02745 (4)	0.42035 (10)	0.10744 (8)	0.0256 (2)
P17	0.129523 (14)	−0.10100 (3)	0.08401 (3)	0.00870 (6)
C18	0.13866 (6)	−0.16073 (13)	−0.02763 (10)	0.0144 (3)
C19	0.13277 (8)	−0.29398 (15)	−0.03653 (13)	0.0228 (3)
C20	0.19303 (7)	−0.12638 (17)	−0.03639 (14)	0.0227 (3)
C21	0.10014 (7)	−0.10194 (15)	−0.10902 (11)	0.0188 (3)
C22	0.06882 (6)	−0.16338 (13)	0.10921 (10)	0.0120 (2)
C23	0.02531 (6)	−0.16275 (16)	0.02038 (12)	0.0186 (3)
C24	0.07481 (7)	−0.28842 (15)	0.14734 (13)	0.0189 (3)
C25	0.05047 (7)	−0.08313 (16)	0.17668 (12)	0.0188 (3)
C26	0.18859 (6)	−0.12988 (13)	0.18181 (11)	0.0141 (3)
C27	0.20892 (7)	−0.25572 (15)	0.18609 (13)	0.0193 (3)
C28	0.17484 (8)	−0.10037 (17)	0.27191 (12)	0.0222 (3)
C29	0.23259 (7)	−0.04461 (16)	0.17551 (14)	0.0235 (4)
H27A	0.2219 (8)	−0.2770 (18)	0.1357 (14)	0.016 (5)*
H25B	0.0468 (8)	−0.0036 (19)	0.1567 (15)	0.021 (6)*
H23A	0.0294 (9)	−0.216 (2)	−0.0229 (15)	0.025 (6)*
H29C	0.2604 (9)	−0.050 (2)	0.2269 (17)	0.031 (6)*
H24C	0.0861 (9)	−0.340 (2)	0.1100 (15)	0.024 (6)*
H23B	0.0224 (9)	−0.0896 (19)	−0.0081 (16)	0.023 (6)*

H29B 0.2470 (9) −0.056 (2) 0.1250 (17) 0.027 (6)*
H20B 0.1953 (8) −0.1518 (19) −0.0968 (15) 0.023 (6)*
H23C −0.0064 (9) −0.183 (2) 0.0370 (15) 0.025 (6)*
H20C 0.2190 (8) −0.1630 (19) 0.0056 (14) 0.019 (5)*
H21A 0.1105 (9) −0.1255 (19) −0.1648 (15) 0.021 (5)*
H24A 0.0985 (9) −0.289 (2) 0.2084 (16) 0.026 (6)*
H25C 0.0181 (10) −0.113 (2) 0.1827 (17) 0.032 (7)*
H20A 0.2006 (9) −0.035 (2) −0.0282 (16) 0.033 (6)*
H28A 0.1517 (9) −0.157 (2) 0.2888 (15) 0.022 (6)*
H29A 0.2218 (9) 0.032 (2) 0.1722 (16) 0.029 (6)*
H28C 0.2066 (10) −0.110 (2) 0.3208 (18) 0.034 (7)*
H21B 0.1026 (9) −0.013 (2) −0.1045 (16) 0.027 (6)*
H24B 0.0391 (9) −0.313 (2) 0.1526 (15) 0.026 (6)*
H27C 0.2381 (10) −0.266 (2) 0.2405 (17) 0.036 (7)*
H25A 0.0731 (9) −0.0830 (18) 0.2377 (16) 0.024 (6)*
H21C 0.0639 (10) −0.129 (2) −0.1151 (17) 0.035 (7)*
H28B 0.1608 (9) −0.020 (2) 0.2725 (15) 0.023 (6)*
H27B 0.1830 (9) −0.316 (2) 0.1883 (16) 0.029 (6)*
H19A 0.1421 (8) −0.3190 (19) −0.0952 (15) 0.022 (5)*
H19B 0.1554 (10) −0.337 (2) 0.0131 (18) 0.039 (7)*
H19C 0.0956 (10) −0.320 (2) −0.0432 (16) 0.033 (6)*

Atomic displacement parameters (Å2)

*U*11 *U*22 *U*33 *U*12 *U*13 *U*23
Au1 0.00861 (2) 0.00577 (2) 0.01353 (2) 0.00023 (2) 0.00243 (2) 0.00002 (2)
N2 0.0139 (6) 0.0063 (5) 0.0141 (5) −0.0006 (4) 0.0026 (4) −0.0004 (4)
S3 0.01020 (14) 0.01008 (14) 0.01498 (15) −0.00139 (12) 0.00190 (12) 0.00206 (12)
O4 0.0205 (6) 0.0123 (5) 0.0263 (6) 0.0032 (4) 0.0031 (5) 0.0067 (4)
O5 0.0182 (5) 0.0181 (5) 0.0150 (5) −0.0055 (4) 0.0017 (4) −0.0011 (4)
C6 0.0158 (7) 0.0230 (8) 0.0244 (8) −0.0059 (6) 0.0079 (6) −0.0018 (7)
F7 0.0167 (5) 0.0334 (6) 0.0467 (7) 0.0031 (4) 0.0120 (5) −0.0081 (5)
F8 0.0318 (6) 0.0519 (8) 0.0284 (6) −0.0161 (6) 0.0147 (5) 0.0067 (6)
F9 0.0188 (5) 0.0347 (6) 0.0368 (6) −0.0147 (5) 0.0091 (5) −0.0127 (5)
S10 0.01084 (15) 0.00927 (14) 0.01515 (15) 0.00002 (11) 0.00074 (12) −0.00201 (12)
O11 0.0183 (5) 0.0172 (5) 0.0156 (5) 0.0018 (4) −0.0014 (4) 0.0012 (4)
O12 0.0191 (5) 0.0097 (5) 0.0241 (6) −0.0027 (4) 0.0016 (4) −0.0031 (4)
C13 0.0154 (7) 0.0206 (7) 0.0194 (7) 0.0002 (6) 0.0040 (6) −0.0056 (6)
F14 0.0212 (5) 0.0283 (6) 0.0296 (6) −0.0065 (4) 0.0098 (4) 0.0006 (4)
F15 0.0261 (6) 0.0468 (7) 0.0299 (6) 0.0009 (5) 0.0092 (5) −0.0217 (5)
F16 0.0145 (5) 0.0287 (6) 0.0315 (6) 0.0083 (4) 0.0022 (4) 0.0002 (4)
P17 0.00768 (14) 0.00673 (14) 0.01192 (15) 0.00027 (11) 0.00292 (11) 0.00009 (12)
C18 0.0194 (7) 0.0108 (6) 0.0152 (6) 0.0020 (5) 0.0082 (5) −0.0001 (5)
C19 0.0382 (10) 0.0108 (7) 0.0216 (8) 0.0027 (7) 0.0113 (7) −0.0021 (6)
C20 0.0222 (8) 0.0229 (8) 0.0286 (9) 0.0064 (7) 0.0166 (7) 0.0060 (7)
C21 0.0262 (8) 0.0173 (7) 0.0134 (6) 0.0016 (6) 0.0060 (6) 0.0006 (6)
C22 0.0100 (6) 0.0116 (6) 0.0154 (6) −0.0012 (5) 0.0048 (5) 0.0009 (5)
C23 0.0108 (6) 0.0204 (7) 0.0230 (8) −0.0036 (6) 0.0014 (6) 0.0009 (6)
C24 0.0195 (8) 0.0144 (7) 0.0245 (8) −0.0016 (6) 0.0088 (6) 0.0056 (6)
C25 0.0171 (7) 0.0210 (8) 0.0215 (7) 0.0033 (6) 0.0106 (6) 0.0002 (6)
C26 0.0097 (6) 0.0122 (6) 0.0183 (7) 0.0007 (5) −0.0002 (5) 0.0008 (5)
C27 0.0147 (7) 0.0148 (7) 0.0273 (8) 0.0046 (5) 0.0035 (6) 0.0051 (6)
C28 0.0231 (8) 0.0240 (8) 0.0156 (7) 0.0022 (7) −0.0020 (6) −0.0011 (6)
C29 0.0125 (7) 0.0193 (8) 0.0341 (10) −0.0043 (6) −0.0027 (7) 0.0048 (7)

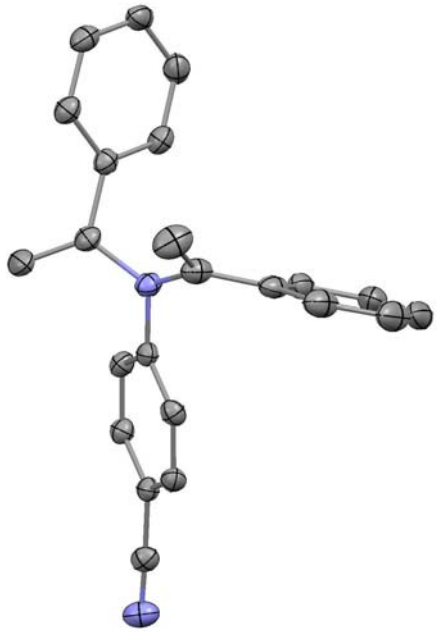
Geometric parameters (Å, °)

Au1—N2 2.1130 (12) C21—H21A 0.99 (2)
Au1—P17 2.2486 (4) C21—H21B 1.02 (2)
N2—S10 1.6265 (13) C21—H21C 0.99 (3)
N2—S3 1.6277 (13) C22—C23 1.532 (2)
S3—O4 1.4195 (12) C22—C24 1.536 (2)
S3—O5 1.4299 (12) C22—C25 1.541 (2)
S3—C6 1.8412 (17) C23—H23A 0.92 (2)
C6—F9 1.322 (2) C23—H23B 0.94 (2)
C6—F7 1.324 (2) C23—H23C 0.97 (2)
C6—F8 1.324 (2) C24—H24C 0.92 (2)
S10—O12 1.4252 (12) C24—H24A 0.98 (2)
S10—O11 1.4275 (12) C24—H24B 1.01 (2)
S10—C13 1.8404 (17) C25—H25B 0.96 (2)
C13—F16 1.319 (2) C25—H25C 0.95 (3)

C13—F15 1.327 (2) C25—H25A 0.96 (2)
C13—F14 1.331 (2) C26—C28 1.534 (2)
P17—C18 1.8913 (15) C26—C27 1.535 (2)
P17—C26 1.8918 (15) C26—C29 1.547 (2)
P17—C22 1.8927 (15) C27—H27A 0.95 (2)
C18—C19 1.536 (2) C27—H27C 0.98 (3)
C18—C20 1.539 (2) C27—H27B 0.98 (2)
C18—C21 1.540 (2) C28—H28A 0.97 (2)
C19—H19A 1.02 (2) C28—H28C 0.98 (3)
C19—H19B 0.97 (3) C28—H28B 0.99 (2)
C19—H19C 1.01 (2) C29—H29C 0.93 (2)
C20—H20B 0.97 (2) C29—H29B 0.95 (2)
C20—H20C 0.91 (2) C29—H29A 0.92 (3)
C20—H20A 1.07 (3)
N2—Au1—P17 177.86 (3) C18—C21—H21B 111.4 (13)
S10—N2—S3 122.64 (8) H21A—C21—H21B 107.5 (17)
S10—N2—Au1 118.19 (7) C18—C21—H21C 111.9 (15)
S3—N2—Au1 119.04 (7) H21A—C21—H21C 108.2 (19)
O4—S3—O5 121.40 (7) H21B—C21—H21C 110.9 (19)
O4—S3—N2 113.47 (7) C23—C22—C24 108.38 (13)
O5—S3—N2 106.86 (7) C23—C22—C25 106.20 (13)
O4—S3—C6 106.22 (8) C24—C22—C25 109.16 (13)
O5—S3—C6 103.61 (8) C23—C22—P17 108.44 (10)
N2—S3—C6 103.35 (7) C24—C22—P17 114.50 (11)
F9—C6—F7 108.50 (15) C25—C22—P17 109.82 (11)
F9—C6—F8 108.92 (15) C22—C23—H23A 114.7 (14)
F7—C6—F8 108.91 (15) C22—C23—H23B 111.2 (14)
F9—C6—S3 112.04 (12) H23A—C23—H23B 106.3 (19)
F7—C6—S3 110.17 (12) C22—C23—H23C 106.7 (13)
F8—C6—S3 108.26 (12) H23A—C23—H23C 107.0 (19)
O12—S10—O11 120.77 (7) H23B—C23—H23C 110.9 (19)
O12—S10—N2 113.26 (7) C22—C24—H24C 113.1 (14)
O11—S10—N2 107.15 (7) C22—C24—H24A 109.6 (13)
O12—S10—C13 105.97 (8) H24C—C24—H24A 110.1 (19)
O11—S10—C13 104.61 (8) C22—C24—H24B 106.2 (13)
N2—S10—C13 103.32 (7) H24C—C24—H24B 109.1 (19)
F16—C13—F15 109.18 (14) H24A—C24—H24B 108.6 (18)
F16—C13—F14 108.90 (14) C22—C25—H25B 112.6 (13)
F15—C13—F14 108.56 (15) C22—C25—H25C 107.3 (15)
F16—C13—S10 111.67 (12) H25B—C25—H25C 110.3 (19)
F15—C13—S10 108.34 (11) C22—C25—H25A 113.9 (13)
F14—C13—S10 110.14 (12) H25B—C25—H25A 106.8 (18)
C18—P17—C26 111.21 (7) H25C—C25—H25A 106 (2)
C18—P17—C22 110.69 (7) C28—C26—C27 109.33 (14)
C26—P17—C22 111.10 (7) C28—C26—C29 105.76 (14)
C18—P17—Au1 108.34 (5) C27—C26—C29 109.30 (14)
C26—P17—Au1 106.80 (5) C28—C26—P17 108.33 (11)
C22—P17—Au1 108.54 (5) C27—C26—P17 114.28 (11)
C19—C18—C20 108.89 (14) C29—C26—P17 109.50 (11)
C19—C18—C21 109.39 (14) C26—C27—H27A 113.9 (13)
C20—C18—C21 105.63 (14) C26—C27—H27C 110.3 (15)
C19—C18—P17 113.81 (11) H27A—C27—H27C 105.5 (18)
C20—C18—P17 108.79 (12) C26—C27—H27B 114.8 (14)
C21—C18—P17 109.99 (10) H27A—C27—H27B 104.5 (18)
C18—C19—H19A 108.2 (12) H27C—C27—H27B 107.1 (19)
C18—C19—H19B 114.2 (15) C26—C28—H28A 113.5 (13)
H19A—C19—H19B 106.8 (19) C26—C28—H28C 106.9 (15)
C18—C19—H19C 112.1 (14) H28A—C28—H28C 102.4 (19)
H19A—C19—H19C 106.2 (18) C26—C28—H28B 112.8 (13)
H19B—C19—H19C 109 (2) H28A—C28—H28B 110.2 (19)
C18—C20—H20B 106.8 (13) H28C—C28—H28B 110.5 (18)
C18—C20—H20C 112.7 (14) C26—C29—H29C 111.5 (15)
H20B—C20—H20C 107.1 (18) C26—C29—H29B 115.8 (14)
C18—C20—H20A 113.3 (13) H29C—C29—H29B 105 (2)
H20B—C20—H20A 110.3 (18) C26—C29—H29A 112.0 (15)
H20C—C20—H20A 106.4 (19) H29C—C29—H29A 106 (2)
C18—C21—H21A 106.7 (13) H29B—C29—H29A 106 (2)

S10—N2—S3—O4 −28.05 (11) N2—S10—C13—F14 61.82 (13)
Au1—N2—S3—O4 156.17 (7) C26—P17—C18—C19 76.06 (14)
S10—N2—S3—O5 −164.52 (9) C22—P17—C18—C19 −47.95 (14)
Au1—N2—S3—O5 19.70 (9) Au1—P17—C18—C19 −166.85 (11)
S10—N2—S3—C6 86.53 (10) C26—P17—C18—C20 −45.53 (13)
Au1—N2—S3—C6 −89.24 (9) C22—P17—C18—C20 −169.55 (11)
O4—S3—C6—F9 55.35 (15) Au1—P17—C18—C20 71.55 (11)
O5—S3—C6—F9 −175.70 (13) C26—P17—C18—C21 −160.79 (11)
N2—S3—C6—F9 −64.34 (14) C22—P17—C18—C21 75.20 (12)
O4—S3—C6—F7 176.23 (12) Au1—P17—C18—C21 −43.70 (12)
O5—S3—C6—F7 −54.82 (14) C18—P17—C22—C23 −42.97 (13)
N2—S3—C6—F7 56.54 (13) C26—P17—C22—C23 −167.05 (11)
O4—S3—C6—F8 −64.77 (14) Au1—P17—C22—C23 75.81 (11)
O5—S3—C6—F8 64.18 (14) C18—P17—C22—C24 78.18 (13)
N2—S3—C6—F8 175.53 (12) C26—P17—C22—C24 −45.90 (13)
S3—N2—S10—O12 −22.13 (11) Au1—P17—C22—C24 −163.05 (10)
Au1—N2—S10—O12 153.68 (7) C18—P17—C22—C25 −158.62 (11)
S3—N2—S10—O11 −157.81 (9) C26—P17—C22—C25 77.31 (12)
Au1—N2—S10—O11 18.00 (9) Au1—P17—C22—C25 −39.84 (11)
S3—N2—S10—C13 92.05 (10) C18—P17—C26—C28 −169.55 (11)
Au1—N2—S10—C13 −92.14 (9) C22—P17—C26—C28 −45.77 (13)
O12—S10—C13—F16 60.02 (13) Au1—P17—C26—C28 72.43 (11)
O11—S10—C13—F16 −171.33 (11) C18—P17—C26—C27 −47.42 (14)
N2—S10—C13—F16 −59.31 (13) C22—P17—C26—C27 76.36 (13)
O12—S10—C13—F15 −60.25 (14) Au1—P17—C26—C27 −165.44 (11)
O11—S10—C13—F15 68.40 (14) C18—P17—C26—C29 75.57 (13)
N2—S10—C13—F15 −179.58 (12) C22—P17—C26—C29 −160.65 (12)
O12—S10—C13—F14 −178.85 (11) Au1—P17—C26—C29 −42.45 (13)
O11—S10—C13—F14 −50.20 (13)

Compound 13.



Computing details
Data collection: *APEX2* v2009-3.0 (BRUKER AXS, Madison, 2009); cell refinement: *APEX2* v2009-3.0 (BRUKER AXS,
Madison, 2009); data reduction: *APEX2* v2009-3.0 (BRUKER AXS, Madison, 2009); program(s) used to solve structure:
SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics:
ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines
(Farrugia, 1999).

(cc02zg1n)
Crystal data
$C_{23}H_{18}N_2$ F_{000} = 680
M_r = 322.39 D_x = 1.191 Mg m^{-3}
Monoclinic, $P2_1/c$ Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2ybc Cell parameters from 1998 reflections
a = 9.4170 (3) Å θ = 2.6–25.9º
b = 22.4201 (8) Å μ = 0.07 mm^{-1}
c = 9.2632 (3) Å *T* = 100 K
β = 113.168 (2)º Prism, yellow
V = 1798.02 (10) Å^3 0.5 × 0.2 × 0.18 mm
Z = 4
Data collection
Bruker X8 kappa APEXII CCD
diffractometer 2942 reflections with $I > 2\sigma(I)$
Monochromator: graphite R_{int} = 0.030
T = 100 K θ_{max} = 26.4º
ω and phi scans θ_{min} = 1.8º
Absorption correction: multi-scan
BRUKER AXS - SADABS *h* = −11→10
T_{min} = 0.919, T_{max} = 1.000 *k* = 0→28
17137 measured reflections *l* = 0→11
3671 independent reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
Least-squares matrix: full Hydrogen site location: inferred from neighbouring
sites
$R[F^2 > 2\sigma(F^2)]$ = 0.045 H-atom parameters constrained
$wR(F^2)$ = 0.123
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 0.6922P]$
where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03 $(\Delta/\sigma)_{max} < 0.001$
3671 reflections $\Delta\rho_{max}$ = 0.37 e Å^{-3}
226 parameters $\Delta\rho_{min}$ = −0.18 e Å^{-3}
Primary atom site location: structure-invariant direct
methods Extinction correction: none

Special details

Geometry. All s.u.'s (except the s.u. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The
cell s.u.'s are taken into account individually in the estimation of s.u.'s in distances, angles and torsion angles; correlations between
s.u.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell s.u.'s is
used for estimating s.u.'s involving l.s. planes.
Refinement. 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 Rfactors(
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 (Å2)

x y z U_{iso}*/U_{eq}

N1 0.09831 (14) 0.10653 (5) 0.29157 (14) 0.0241 (3)
C11 0.19737 (16) 0.07369 (6) 0.42211 (16) 0.0216 (3)
C12 0.25739 (17) 0.09900 (7) 0.57212 (17) 0.0239 (3)
H12 0.2291 0.1385 0.5869 0.029*
C13 0.35746 (16) 0.06713 (7) 0.69918 (17) 0.0239 (3)
H13 0.3979 0.0847 0.8007 0.029*
C14 0.39878 (16) 0.00932 (6) 0.67804 (16) 0.0221 (3)
C15 0.34009 (16) −0.01634 (7) 0.52835 (17) 0.0231 (3)
H15 0.3689 −0.0558 0.5139 0.028*
C16 0.24065 (16) 0.01548 (6) 0.40199 (17) 0.0236 (3)
H16 0.2011 −0.0021 0.3005 0.028*
C17 0.49794 (17) −0.02556 (7) 0.80954 (17) 0.0254 (3)
N18 0.57439 (16) −0.05462 (6) 0.91279 (15) 0.0336 (3)
C21 −0.03654 (16) 0.07903 (7) 0.17786 (17) 0.0253 (3)
C22 −0.11464 (18) 0.03697 (8) 0.2152 (2) 0.0347 (4)
H22A −0.0818 0.0239 0.321 0.042*
H22B −0.2036 0.0198 0.1363 0.042*
C23 −0.07725 (17) 0.09933 (6) 0.01334 (17) 0.0238 (3)
C24 0.03605 (17) 0.10453 (7) −0.04695 (18) 0.0272 (3)
H24 0.1405 0.0955 0.0176 0.033*
C25 −0.00252 (19) 0.12274 (7) −0.20051 (19) 0.0323 (4)
H25 0.0753 0.1253 −0.2415 0.039*
C26 −0.15365 (19) 0.13726 (7) −0.29487 (18) 0.0313 (4)
H26 −0.179 0.1507 −0.3993 0.038*
C27 −0.26739 (18) 0.13210 (7) −0.23665 (18) 0.0304 (4)
H27 −0.3713 0.1419 −0.3011 0.037*
C28 −0.22967 (17) 0.11256 (7) −0.08400 (18) 0.0272 (3)
H28 −0.3087 0.1081 −0.0453 0.033*
C31 0.10060 (18) 0.17010 (7) 0.30212 (17) 0.0289 (4)
C32 −0.0303 (2) 0.20039 (8) 0.2676 (2) 0.0430 (5)
H32A −0.1259 0.1797 0.2358 0.052*

H32B −0.0282 0.2427 0.2748 0.052*
C33 0.25394 (19) 0.19853 (7) 0.34249 (17) 0.0295 (4)
C34 0.36937 (19) 0.17053 (7) 0.30848 (18) 0.0304 (4)
H34 0.3499 0.1324 0.2598 0.036*
C35 0.5118 (2) 0.19720 (8) 0.34423 (19) 0.0391 (4)
H35 0.5889 0.1773 0.3202 0.047*
C36 0.5422 (2) 0.25284 (9) 0.4150 (2) 0.0458 (5)
H36 0.6399 0.2712 0.4399 0.055*
C37 0.4288 (3) 0.28130 (8) 0.4488 (2) 0.0486 (5)
H37 0.4485 0.3197 0.4957 0.058*
C38 0.2866 (2) 0.25453 (7) 0.41536 (19) 0.0394 (4)
H38 0.211 0.2743 0.442 0.047*

Atomic displacement parameters (Å2)

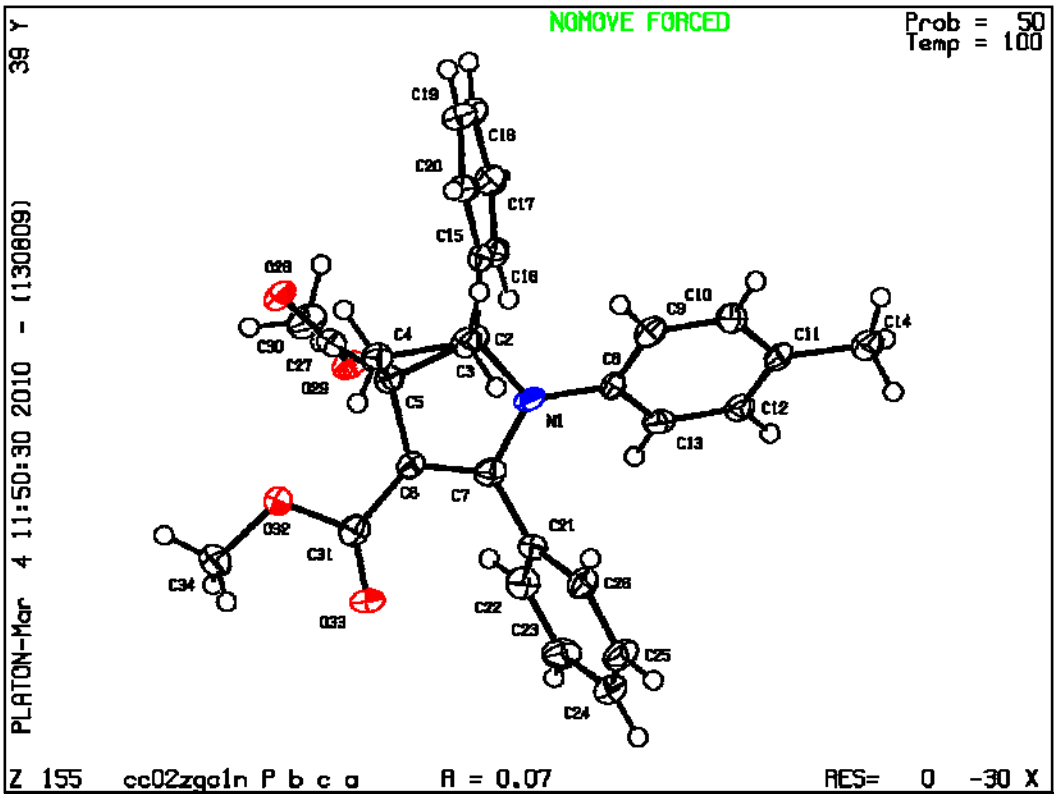
U_{11} U_{22} U_{33} U_{12} U_{13} U_{23}
N1 0.0240 (6) 0.0222 (6) 0.0227 (6) 0.0018 (5) 0.0056 (5) 0.0009 (5)
C11 0.0178 (7) 0.0248 (7) 0.0222 (7) −0.0015 (6) 0.0078 (6) 0.0023 (6)
C12 0.0269 (8) 0.0228 (7) 0.0251 (8) 0.0002 (6) 0.0135 (6) −0.0005 (6)
C13 0.0242 (7) 0.0282 (8) 0.0200 (7) −0.0028 (6) 0.0094 (6) −0.0019 (6)
C14 0.0180 (7) 0.0264 (8) 0.0222 (7) −0.0004 (6) 0.0082 (6) 0.0025 (6)
C15 0.0212 (7) 0.0234 (7) 0.0245 (7) 0.0004 (6) 0.0089 (6) −0.0008 (6)
C16 0.0225 (7) 0.0246 (8) 0.0220 (7) −0.0014 (6) 0.0071 (6) −0.0030 (6)
C17 0.0245 (8) 0.0299 (8) 0.0229 (8) 0.0010 (6) 0.0107 (6) −0.0035 (6)
N18 0.0349 (8) 0.0391 (8) 0.0241 (7) 0.0097 (6) 0.0087 (6) 0.0007 (6)
C21 0.0197 (7) 0.0278 (8) 0.0262 (8) 0.0028 (6) 0.0066 (6) 0.0007 (6)
C22 0.0223 (8) 0.0464 (10) 0.0307 (9) −0.0036 (7) 0.0054 (7) 0.0086 (7)
C23 0.0237 (8) 0.0194 (7) 0.0256 (8) −0.0004 (6) 0.0068 (6) −0.0011 (6)
C24 0.0213 (7) 0.0293 (8) 0.0287 (8) −0.0014 (6) 0.0074 (6) −0.0030 (6)
C25 0.0344 (9) 0.0348 (9) 0.0314 (9) −0.0092 (7) 0.0167 (7) −0.0059 (7)
C26 0.0406 (9) 0.0268 (8) 0.0217 (8) −0.0078 (7) 0.0070 (7) −0.0019 (6)
C27 0.0283 (8) 0.0277 (8) 0.0272 (8) 0.0012 (6) 0.0023 (7) −0.0025 (6)
C28 0.0227 (8) 0.0288 (8) 0.0288 (8) 0.0008 (6) 0.0086 (6) −0.0027 (6)
C31 0.0366 (9) 0.0239 (8) 0.0232 (8) 0.0043 (6) 0.0084 (7) −0.0005 (6)
C32 0.0442 (11) 0.0337 (9) 0.0426 (10) 0.0115 (8) 0.0079 (8) −0.0064 (8)
C33 0.0426 (9) 0.0210 (7) 0.0196 (7) −0.0006 (7) 0.0066 (7) 0.0032 (6)
C34 0.0382 (9) 0.0261 (8) 0.0238 (8) −0.0073 (7) 0.0089 (7) 0.0005 (6)
C35 0.0431 (10) 0.0441 (10) 0.0266 (8) −0.0128 (8) 0.0099 (8) 0.0034 (7)
C36 0.0590 (12) 0.0419 (10) 0.0276 (9) −0.0247 (9) 0.0074 (9) 0.0033 (7)
C37 0.0743 (14) 0.0271 (9) 0.0314 (9) −0.0181 (9) 0.0069 (9) −0.0016 (7)
C38 0.0593 (12) 0.0240 (8) 0.0274 (9) −0.0015 (8) 0.0091 (8) −0.0004 (6)

Geometric parameters (Å, °)

N1—C11 1.4096 (18) C25—C26 1.384 (2)
N1—C31 1.4282 (19) C25—H25 0.95
N1—C21 1.4317 (19) C26—C27 1.381 (2)
C11—C12 1.398 (2) C26—H26 0.95
C11—C16 1.401 (2) C27—C28 1.387 (2)
C12—C13 1.382 (2) C27—H27 0.95
C12—H12 0.95 C28—H28 0.95
C13—C14 1.389 (2) C31—C32 1.332 (2)
C13—H13 0.95 C31—C33 1.485 (2)
C14—C15 1.399 (2) C32—H32A 0.95
C14—C17 1.439 (2) C32—H32B 0.95
C15—C16 1.376 (2) C33—C34 1.394 (2)
C15—H15 0.95 C33—C38 1.401 (2)
C16—H16 0.95 C34—C35 1.384 (2)
C17—N18 1.1471 (19) C34—H34 0.95
C21—C22 1.323 (2) C35—C36 1.385 (3)
C21—C23 1.489 (2) C35—H35 0.95
C22—H22A 0.95 C36—C37 1.382 (3)
C22—H22B 0.95 C36—H36 0.95
C23—C24 1.390 (2) C37—C38 1.386 (3)
C23—C28 1.394 (2) C37—H37 0.95
C24—C25 1.384 (2) C38—H38 0.95
C24—H24 0.95
C11—N1—C31 118.39 (12) C24—C25—H25 119.8
C11—N1—C21 120.15 (12) C26—C25—H25 119.8
C31—N1—C21 117.73 (12) C27—C26—C25 119.77 (15)
C12—C11—C16 119.03 (13) C27—C26—H26 120.1

C12—C11—N1 120.86 (13) C25—C26—H26 120.1
C16—C11—N1 120.09 (13) C26—C27—C28 119.87 (15)
C13—C12—C11 120.67 (14) C26—C27—H27 120.1
C13—C12—H12 119.7 C28—C27—H27 120.1
C11—C12—H12 119.7 C27—C28—C23 120.82 (14)
C12—C13—C14 119.82 (13) C27—C28—H28 119.6
C12—C13—H13 120.1 C23—C28—H28 119.6
C14—C13—H13 120.1 C32—C31—N1 120.31 (15)
C13—C14—C15 120.01 (13) C32—C31—C33 123.94 (15)
C13—C14—C17 120.80 (13) N1—C31—C33 115.63 (13)
C15—C14—C17 119.16 (13) C31—C32—H32A 120
C16—C15—C14 120.08 (14) C31—C32—H32B 120
C16—C15—H15 120 H32A—C32—H32B 120
C14—C15—H15 120 C34—C33—C38 117.99 (16)
C15—C16—C11 120.40 (13) C34—C33—C31 121.13 (14)
C15—C16—H16 119.8 C38—C33—C31 120.88 (16)
C11—C16—H16 119.8 C35—C34—C33 121.29 (16)
N18—C17—C14 178.18 (17) C35—C34—H34 119.4
C22—C21—N1 122.55 (14) C33—C34—H34 119.4
C22—C21—C23 122.68 (14) C34—C35—C36 120.17 (19)
N1—C21—C23 114.71 (12) C34—C35—H35 119.9
C21—C22—H22A 120 C36—C35—H35 119.9
C21—C22—H22B 120 C37—C36—C35 119.24 (18)
H22A—C22—H22B 120 C37—C36—H36 120.4
C24—C23—C28 118.69 (14) C35—C36—H36 120.4
C24—C23—C21 120.50 (13) C36—C37—C38 120.94 (17)
C28—C23—C21 120.80 (13) C36—C37—H37 119.5
C25—C24—C23 120.33 (14) C38—C37—H37 119.5
C25—C24—H24 119.8 C37—C38—C33 120.35 (18)
C23—C24—H24 119.8 C37—C38—H38 119.8
C24—C25—C26 120.48 (15) C33—C38—H38 119.8
C31—N1—C11—C12 20.7 (2) C21—C23—C24—C25 179.24 (14)
C21—N1—C11—C12 −137.04 (14) C23—C24—C25—C26 1.4 (2)
C31—N1—C11—C16 −157.40 (14) C24—C25—C26—C27 −1.7 (2)
C21—N1—C11—C16 44.84 (19) C25—C26—C27—C28 0.2 (2)
C16—C11—C12—C13 −0.3 (2) C26—C27—C28—C23 1.5 (2)
N1—C11—C12—C13 −178.43 (13) C24—C23—C28—C27 −1.7 (2)
C11—C12—C13—C14 −0.1 (2) C21—C23—C28—C27 179.28 (14)
C12—C13—C14—C15 0.5 (2) C11—N1—C31—C32 −128.23 (16)
C12—C13—C14—C17 −177.53 (13) C21—N1—C31—C32 30.1 (2)
C13—C14—C15—C16 −0.4 (2) C11—N1—C31—C33 55.68 (18)
C17—C14—C15—C16 177.65 (13) C21—N1—C31—C33 −146.02 (13)
C14—C15—C16—C11 0.0 (2) C32—C31—C33—C34 −150.35 (17)
C12—C11—C16—C15 0.4 (2) N1—C31—C33—C34 25.6 (2)
N1—C11—C16—C15 178.52 (13) C32—C31—C33—C38 29.3 (2)
C11—N1—C21—C22 31.9 (2) N1—C31—C33—C38 −154.73 (14)
C31—N1—C21—C22 −125.96 (17) C38—C33—C34—C35 −0.5 (2)
C11—N1—C21—C23 −145.16 (13) C31—C33—C34—C35 179.22 (14)
C31—N1—C21—C23 56.94 (18) C33—C34—C35—C36 −0.1 (2)
C22—C21—C23—C24 −131.90 (17) C34—C35—C36—C37 −0.2 (3)
N1—C21—C23—C24 45.20 (19) C35—C36—C37—C38 1.1 (3)
C22—C21—C23—C28 47.1 (2) C36—C37—C38—C33 −1.7 (3)
N1—C21—C23—C28 −135.84 (14) C34—C33—C38—C37 1.4 (2)
C28—C23—C24—C25 0.3 (2) C31—C33—C38—C37 −178.34 (15)

Compound 32.



Crystal data

$C_{29}H_{27}NO_4$ D_x = 1.301 Mg m^{-3}
M_r = 453.52 Mo $K\alpha$ radiation, λ = 0.7107 Å
Orthorhombic, *Pbca* Cell parameters from 1984 reflections
a = 7.5332 (12) Å θ = 2.2–18.4°
b = 18.566 (3) Å μ = 0.09 mm^{-1}
c = 33.103 (5) Å *T* = 100 K
V = 4629.8 (13) Å^3 Prism, colourless
Z = 8 0.35 × 0.19 × 0.02 mm
F(000) = 1920

Data collection

BRUKER APPEX-II CCD
diffractometer 1936 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube R_{int} = 0.210
ω and phi scans θ_{max} = 23.3°, θ_{min} = 2.2°
Absorption correction: multi-scan
BRUKER SADABS *h* = 0→8
T_{min} = 0.707, T_{max} = 1 *k* = 0→20
64702 measured reflections *l* = 0→36
3335 independent reflections

Refinement

Refinement on F^2 Secondary atom site location: difference Fourier map
Least-squares matrix: full Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.065 H-atom parameters constrained
$wR(F^2)$ = 0.170
$w = 1/[\sigma^2(F_o$

$_2$) + (0.0826P)$_2$ + 0.4631P]
where P = (F_o
$_2$ + 2F_c
$_2$)/3
S = 1.02 $(\Delta/\sigma)_{max}$ < 0.001
3335 reflections $\Delta\rho_{max}$ = 0.30 e Å^{-3}
311 parameters $\Delta\rho_{min}$ = −0.28 e Å^{-3}
0 restraints
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: 0.0048 (8)

Special details

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

N1 0.4260 (4) 0.00033 (17) 0.13757 (9) 0.0236 (8)
C2 0.5143 (5) −0.0553 (2) 0.16253 (11) 0.0221 (10)
C3 0.6598 (5) −0.0260 (2) 0.19166 (12) 0.0268 (11)
H3A 0.6816 0.0263 0.1888 0.032*
H3B 0.6397 −0.0391 0.2203 0.032*
C4 0.7997 (5) −0.0729 (2) 0.17070 (12) 0.0269 (11)
H4A 0.8275 −0.1179 0.1854 0.032*
H4B 0.9095 −0.0465 0.1636 0.032*
C5 0.6723 (6) −0.0836 (2) 0.13480 (12) 0.0239 (10)
C6 0.6766 (5) −0.0269 (2) 0.10196 (12) 0.0236 (10)
C7 0.5353 (5) 0.0183 (2) 0.10532 (11) 0.0236 (10)
C8 0.2761 (5) 0.0394 (2) 0.15199 (12) 0.0235 (10)
C9 0.2506 (6) 0.0477 (2) 0.19352 (12) 0.0258 (10)
H9 0.3331 0.0273 0.212 0.031*
C10 0.1055 (6) 0.0855 (2) 0.20789 (12) 0.0263 (11)
H10 0.0896 0.0902 0.2362 0.032*
C11 −0.0169 (5) 0.1166 (2) 0.18221 (12) 0.0223 (10)
C12 0.0059 (6) 0.1051 (2) 0.14070 (12) 0.0241 (10)
H12 −0.0793 0.1239 0.1224 0.029*
C13 0.1486 (6) 0.0672 (2) 0.12585 (12) 0.0234 (10)
H13 0.1602 0.0599 0.0976 0.028*
C14 −0.1659 (6) 0.1619 (2) 0.19859 (12) 0.0302 (11)
H14A −0.18 0.1524 0.2275 0.045*
H14B −0.2763 0.1498 0.1845 0.045*
H14C −0.1385 0.2129 0.1944 0.045*
C15 0.3912 (5) −0.1132 (2) 0.17790 (12) 0.0225 (10)
C16 0.2494 (6) −0.1363 (2) 0.15393 (12) 0.0255 (10)
H16 0.222 −0.1113 0.1297 0.031*
C17 0.1485 (6) −0.1952 (2) 0.16514 (12) 0.0289 (11)
H17 0.0534 −0.2108 0.1485 0.035*
C18 0.1862 (6) −0.2312 (2) 0.20062 (13) 0.0294 (11)
H18 0.1176 −0.2719 0.2083 0.035*
C19 0.3220 (6) −0.2083 (2) 0.22453 (13) 0.0324 (12)

H19 0.3458 −0.2329 0.2491 0.039*
C20 0.4257 (6) −0.1501 (2) 0.21382 (11) 0.0272 (11)
H20 0.5204 −0.1352 0.2308 0.033*
C21 0.4960 (5) 0.0809 (2) 0.07860 (12) 0.0234 (10)
C22 0.4348 (6) 0.0700 (2) 0.03955 (12) 0.0289 (11)
H22 0.4154 0.0224 0.0299 0.035*
C23 0.4019 (6) 0.1282 (2) 0.01456 (12) 0.0327 (12)
H23 0.36 0.1206 −0.0122 0.039*
C24 0.4303 (6) 0.1980 (2) 0.02855 (12) 0.0325 (11)
H24 0.4061 0.2379 0.0115 0.039*
C25 0.4936 (6) 0.2094 (2) 0.06717 (12) 0.0307 (11)
H25 0.516 0.2571 0.0764 0.037*
C26 0.5245 (5) 0.1510 (2) 0.09266 (12) 0.0260 (10)
H26 0.5647 0.1587 0.1195 0.031*
C27 0.6518 (6) −0.1600 (2) 0.11884 (12) 0.0265 (11)
O28 0.7213 (4) −0.21262 (14) 0.13349 (8) 0.0301 (8)
O29 0.5354 (4) −0.16282 (13) 0.08794 (8) 0.0273 (7)
C30 0.4843 (6) −0.2349 (2) 0.07624 (13) 0.0348 (12)
H30A 0.5904 −0.2625 0.0689 0.052*
H30B 0.4039 −0.2325 0.053 0.052*
H30C 0.424 −0.2585 0.0989 0.052*
C31 0.8133 (5) −0.0222 (2) 0.07123 (12) 0.0254 (11)
O32 0.9218 (4) −0.08147 (14) 0.07344 (8) 0.0282 (8)
O33 0.8352 (4) 0.02467 (15) 0.04631 (8) 0.0351 (8)
C34 1.0614 (6) −0.0842 (2) 0.04322 (13) 0.0348 (12)
H34A 1.0084 −0.0898 0.0164 0.052*
H34B 1.1397 −0.1252 0.0488 0.052*
H34C 1.1303 −0.0395 0.0441 0.052*

Atomic displacement parameters (Å²)

U_{11} U_{22} U_{33} U_{12} U_{13} U_{23}

N1 0.031 (2) 0.0177 (19) 0.0217 (19) 0.0018 (17) 0.0007 (17) 0.0069 (15)
C2 0.025 (3) 0.017 (2) 0.025 (2) 0.001 (2) 0.002 (2) −0.0015 (18)
C3 0.031 (3) 0.021 (2) 0.028 (2) 0.000 (2) 0.002 (2) −0.0005 (19)
C4 0.026 (3) 0.024 (2) 0.031 (2) −0.002 (2) 0.001 (2) 0.001 (2)
C5 0.029 (3) 0.018 (2) 0.025 (2) −0.001 (2) 0.002 (2) 0.0022 (18)
C6 0.025 (3) 0.017 (2) 0.029 (2) 0.001 (2) 0.003 (2) 0.0026 (18)
C7 0.029 (3) 0.016 (2) 0.026 (2) −0.007 (2) −0.001 (2) −0.0025 (18)
C8 0.030 (3) 0.011 (2) 0.029 (2) 0.002 (2) 0.007 (2) −0.0013 (19)
C9 0.029 (3) 0.018 (2) 0.030 (2) −0.001 (2) −0.005 (2) 0.0001 (19)
C10 0.038 (3) 0.018 (2) 0.022 (2) −0.002 (2) 0.005 (2) −0.0062 (19)
C11 0.030 (3) 0.010 (2) 0.027 (2) −0.002 (2) −0.002 (2) −0.0028 (18)
C12 0.027 (3) 0.017 (2) 0.028 (3) −0.002 (2) 0.000 (2) 0.0024 (19)
C13 0.033 (3) 0.019 (2) 0.018 (2) −0.003 (2) 0.001 (2) 0.0028 (19)
C14 0.037 (3) 0.020 (2) 0.033 (3) 0.000 (2) 0.004 (2) 0.000 (2)
C15 0.025 (3) 0.017 (2) 0.026 (2) 0.003 (2) 0.006 (2) −0.0011 (19)
C16 0.032 (3) 0.020 (2) 0.024 (2) 0.004 (2) 0.002 (2) −0.0009 (19)
C17 0.032 (3) 0.022 (2) 0.033 (3) −0.002 (2) 0.004 (2) −0.002 (2)
C18 0.033 (3) 0.017 (2) 0.038 (3) 0.000 (2) 0.008 (2) 0.003 (2)
C19 0.040 (3) 0.024 (3) 0.033 (3) 0.000 (2) 0.004 (2) 0.010 (2)
C20 0.032 (3) 0.025 (3) 0.025 (2) −0.002 (2) 0.003 (2) 0.004 (2)
C21 0.024 (2) 0.021 (2) 0.025 (2) −0.001 (2) 0.006 (2) 0.0029 (19)
C22 0.033 (3) 0.027 (3) 0.027 (2) −0.004 (2) −0.001 (2) −0.003 (2)
C23 0.043 (3) 0.035 (3) 0.020 (2) −0.003 (2) −0.003 (2) 0.003 (2)
C24 0.039 (3) 0.027 (3) 0.031 (3) 0.006 (2) 0.003 (2) 0.008 (2)
C25 0.043 (3) 0.017 (2) 0.032 (3) −0.002 (2) −0.002 (2) 0.000 (2)
C26 0.030 (3) 0.020 (2) 0.027 (2) 0.001 (2) −0.002 (2) −0.002 (2)
C27 0.027 (3) 0.026 (3) 0.026 (3) 0.000 (2) 0.008 (2) 0.002 (2)
O28 0.0381 (19) 0.0175 (16) 0.0348 (17) 0.0040 (15) 0.0016 (14) 0.0041 (14)
O29 0.0363 (19) 0.0190 (16) 0.0266 (16) −0.0026 (14) −0.0028 (14) 0.0018 (13)
C30 0.047 (3) 0.021 (3) 0.037 (3) −0.008 (2) −0.005 (2) −0.004 (2)
C31 0.030 (3) 0.017 (2) 0.029 (3) −0.004 (2) −0.001 (2) −0.005 (2)
O32 0.0318 (18) 0.0214 (17) 0.0313 (17) 0.0037 (14) 0.0093 (14) 0.0025 (13)
O33 0.043 (2) 0.0265 (17) 0.0360 (18) 0.0029 (15) 0.0135 (15) 0.0142 (15)
C34 0.039 (3) 0.031 (3) 0.035 (3) 0.002 (2) 0.014 (2) 0.000 (2)

Geometric parameters (Å, °)

N1—C7 1.389 (5) C16—C17 1.382 (5)
N1—C8 1.425 (5) C16—H16 0.95
N1—C2 1.480 (5) C17—C18 1.382 (5)
C2—C15 1.508 (5) C17—H17 0.95
C2—C3 1.558 (5) C18—C19 1.362 (6)
C2—C5 1.592 (6) C18—H18 0.95
C3—C4 1.532 (5) C19—C20 1.379 (5)
C3—H3A 0.99 C19—H19 0.95
C3—H3B 0.99 C20—H20 0.95
C4—C5 1.541 (5) C21—C22 1.387 (6)
C4—H4A 0.99 C21—C26 1.398 (5)
C4—H4B 0.99 C22—C23 1.383 (6)
C5—C6 1.514 (5) C22—H22 0.95
C5—C27 1.521 (5) C23—C24 1.393 (6)
C6—C7 1.360 (5) C23—H23 0.95
C6—C31 1.450 (6) C24—C25 1.381 (6)
C7—C21 1.491 (5) C24—H24 0.95
C8—C13 1.392 (6) C25—C26 1.394 (5)
C8—C9 1.397 (5) C25—H25 0.95
C9—C10 1.383 (6) C26—H26 0.95
C9—H9 0.95 C27—O28 1.210 (5)
C10—C11 1.381 (6) C27—O29 1.348 (5)
C10—H10 0.95 O29—C30 1.445 (4)
C11—C12 1.401 (5) C30—H30A 0.98
C11—C14 1.504 (5) C30—H30B 0.98
C12—C13 1.376 (5) C30—H30C 0.98
C12—H12 0.95 C31—O33 1.211 (5)
C13—H13 0.95 C31—O32 1.372 (4)
C14—H14A 0.98 O32—C34 1.453 (5)
C14—H14B 0.98 C34—H34A 0.98
C14—H14C 0.98 C34—H34B 0.98
C15—C20 1.397 (5) C34—H34C 0.98
C15—C16 1.399 (6)
C7—N1—C8 127.3 (3) C20—C15—C16 118.3 (4)
C7—N1—C2 109.3 (3) C20—C15—C2 121.5 (4)
C8—N1—C2 121.6 (3) C16—C15—C2 119.8 (4)
N1—C2—C15 114.2 (3) C17—C16—C15 120.7 (4)
N1—C2—C3 114.7 (3) C17—C16—H16 119.7
C15—C2—C3 118.2 (3) C15—C16—H16 119.7
N1—C2—C5 104.2 (3) C18—C17—C16 119.9 (4)
C15—C2—C5 114.7 (3) C18—C17—H17 120
C3—C2—C5 86.9 (3) C16—C17—H17 120
C4—C3—C2 90.3 (3) C19—C18—C17 119.8 (4)
C4—C3—H3A 113.6 C19—C18—H18 120.1
C2—C3—H3A 113.6 C17—C18—H18 120.1
C4—C3—H3B 113.6 C18—C19—C20 121.4 (4)
C2—C3—H3B 113.6 C18—C19—H19 119.3
H3A—C3—H3B 110.9 C20—C19—H19 119.3
C3—C4—C5 89.7 (3) C19—C20—C15 119.9 (4)
C3—C4—H4A 113.7 C19—C20—H20 120.1
C5—C4—H4A 113.7 C15—C20—H20 120.1
C3—C4—H4B 113.7 C22—C21—C26 119.8 (4)
C5—C4—H4B 113.7 C22—C21—C7 120.4 (4)
H4A—C4—H4B 110.9 C26—C21—C7 119.8 (4)
C6—C5—C27 113.6 (3) C23—C22—C21 120.2 (4)
C6—C5—C4 116.8 (3) C23—C22—H22 119.9
C27—C5—C4 116.9 (3) C21—C22—H22 119.9
C6—C5—C2 101.6 (3) C22—C23—C24 120.0 (4)
C27—C5—C2 115.6 (3) C22—C23—H23 120
C4—C5—C2 88.7 (3) C24—C23—H23 120
C7—C6—C31 125.2 (4) C25—C24—C23 120.3 (4)
C7—C6—C5 110.7 (3) C25—C24—H24 119.9
C31—C6—C5 124.1 (4) C23—C24—H24 119.9
C6—C7—N1 112.3 (3) C24—C25—C26 119.9 (4)

C6—C7—C21 126.0 (4) C24—C25—H25 120.1
N1—C7—C21 121.7 (4) C26—C25—H25 120.1
C13—C8—C9 118.5 (4) C25—C26—C21 119.8 (4)
C13—C8—N1 121.8 (4) C25—C26—H26 120.1
C9—C8—N1 119.7 (4) C21—C26—H26 120.1
C10—C9—C8 120.2 (4) O28—C27—O29 123.7 (4)
C10—C9—H9 119.9 O28—C27—C5 124.7 (4)
C8—C9—H9 119.9 O29—C27—C5 111.5 (4)
C11—C10—C9 121.9 (4) C27—O29—C30 114.4 (3)
C11—C10—H10 119.1 O29—C30—H30A 109.5
C9—C10—H10 119.1 O29—C30—H30B 109.5
C10—C11—C12 117.3 (4) H30A—C30—H30B 109.5
C10—C11—C14 120.7 (4) O29—C30—H30C 109.5
C12—C11—C14 122.0 (4) H30A—C30—H30C 109.5
C13—C12—C11 121.6 (4) H30B—C30—H30C 109.5
C13—C12—H12 119.2 O33—C31—O32 122.1 (4)
C11—C12—H12 119.2 O33—C31—C6 128.2 (4)
C12—C13—C8 120.4 (4) O32—C31—C6 109.7 (3)
C12—C13—H13 119.8 C31—O32—C34 115.0 (3)
C8—C13—H13 119.8 O32—C34—H34A 109.5
C11—C14—H14A 109.5 O32—C34—H34B 109.5
C11—C14—H14B 109.5 H34A—C34—H34B 109.5
H14A—C14—H14B 109.5 O32—C34—H34C 109.5
C11—C14—H14C 109.5 H34A—C34—H34C 109.5
H14A—C14—H14C 109.5 H34B—C34—H34C 109.5
H14B—C14—H14C 109.5
C7—N1—C2—C15 −140.0 (3) C10—C11—C12—C13 −2.9 (6)
C8—N1—C2—C15 54.4 (5) C14—C11—C12—C13 176.0 (4)
C7—N1—C2—C3 79.0 (4) C11—C12—C13—C8 −0.5 (6)
C8—N1—C2—C3 −86.5 (4) C9—C8—C13—C12 3.3 (6)
C7—N1—C2—C5 −14.1 (4) N1—C8—C13—C12 −179.6 (3)
C8—N1—C2—C5 −179.7 (3) N1—C2—C15—C20 −152.1 (3)
N1—C2—C3—C4 −119.7 (3) C3—C2—C15—C20 −12.5 (5)
C15—C2—C3—C4 101.0 (4) C5—C2—C15—C20 87.8 (4)
C5—C2—C3—C4 −15.5 (3) N1—C2—C15—C16 34.9 (5)
C2—C3—C4—C5 16.0 (3) C3—C2—C15—C16 174.4 (3)
C3—C4—C5—C6 86.6 (4) C5—C2—C15—C16 −85.2 (4)
C3—C4—C5—C27 −133.9 (4) C20—C15—C16—C17 −1.4 (6)
C3—C4—C5—C2 −15.7 (3) C2—C15—C16—C17 171.9 (4)
N1—C2—C5—C6 13.1 (4) C15—C16—C17—C18 0.8 (6)
C15—C2—C5—C6 138.6 (3) C16—C17—C18—C19 0.4 (6)
C3—C2—C5—C6 −101.7 (3) C17—C18—C19—C20 −1.1 (6)
N1—C2—C5—C27 −110.4 (4) C18—C19—C20—C15 0.5 (6)
C15—C2—C5—C27 15.1 (5) C16—C15—C20—C19 0.7 (6)
C3—C2—C5—C27 134.8 (4) C2—C15—C20—C19 −172.4 (4)
N1—C2—C5—C4 130.2 (3) C6—C7—C21—C22 73.1 (6)
C15—C2—C5—C4 −104.3 (3) N1—C7—C21—C22 −107.3 (4)
C3—C2—C5—C4 15.4 (3) C6—C7—C21—C26 −105.6 (5)
C27—C5—C6—C7 116.5 (4) N1—C7—C21—C26 74.0 (5)
C4—C5—C6—C7 −102.8 (4) C26—C21—C22—C23 0.0 (6)
C2—C5—C6—C7 −8.3 (4) C7—C21—C22—C23 −178.7 (4)
C27—C5—C6—C31 −63.3 (5) C21—C22—C23—C24 0.0 (6)
C4—C5—C6—C31 77.4 (5) C22—C23—C24—C25 0.9 (7)
C2—C5—C6—C31 171.9 (3) C23—C24—C25—C26 −1.8 (7)
C31—C6—C7—N1 179.7 (3) C24—C25—C26—C21 1.8 (6)
C5—C6—C7—N1 −0.1 (5) C22—C21—C26—C25 −0.9 (6)
C31—C6—C7—C21 −0.7 (6) C7—C21—C26—C25 177.7 (4)
C5—C6—C7—C21 179.5 (4) C6—C5—C27—O28 144.3 (4)
C8—N1—C7—C6 174.1 (4) C4—C5—C27—O28 3.7 (6)
C2—N1—C7—C6 9.6 (4) C2—C5—C27—O28 −98.8 (5)
C8—N1—C7—C21 −5.6 (6) C6—C5—C27—O29 −40.3 (5)
C2—N1—C7—C21 −170.1 (3) C4—C5—C27—O29 179.1 (3)
C7—N1—C8—C13 45.2 (6) C2—C5—C27—O29 76.6 (4)
C2—N1—C8—C13 −152.0 (4) O28—C27—O29—C30 6.4 (5)
C7—N1—C8—C9 −137.8 (4) C5—C27—O29—C30 −169.0 (3)
C2—N1—C8—C9 25.0 (5) C7—C6—C31—O33 7.4 (7)
C13—C8—C9—C10 −2.8 (6) C5—C6—C31—O33 −172.9 (4)

N1—C8—C9—C10 −180.0 (3) C7—C6—C31—O32 −172.0 (4)
C8—C9—C10—C11 −0.6 (6) C5—C6—C31—O32 7.8 (5)
C9—C10—C11—C12 3.4 (6) O33—C31—O32—C34 −1.3 (5)
C9—C10—C11—C14 −175.5 (4) C6—C31—O32—C34 178.2 (3