Supporting Information

for

Palladium-Catalyzed Cyanoesterification of Norbornenes with Cyanoformates via the NC–Pd–COOR (R = Me and Et) Intermediate

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General. All the reactions were carried out under an Ar atmosphere using standard Schlenk techniques. Glassware was dried in an oven (130 °C) and heated under reduced pressure before use. Dehydrated toluene, dichloromethane, hexane, and diethyl ether were purchased from Kanto Chemicals Co., Ltd. For thin layer chromatography (TLC) analyses throughout this work, Merck precoated TLC plates (silica gel 60 GF_{254}, 0.25 mm) were used. Silica gel column chromatography was carried out using Silica gel 60 N (spherical, neutral, 40-100 μm) from Kanto Chemicals Co., Ltd. NMR spectra (^{1}H, $^{13}C\{^{1}H\}$, and $^{31}P\{^{1}H\}$) were recorded on Varian INOVA-600 (600 MHz) or Mercury-300 (300 MHz) spectrometers. Peak positions of the $^{31}P\{^{1}H\}$ NMR spectra were referenced to an external 85% H_3PO_4. Infrared spectra were recorded on a JASCO FT-IR 5000 spectrophotometer. Gel permeation chromatography (GPC) analyses were carried out with JASCO HPLC system equipped with a UV detector using THF as an eluent at a flow rate of 1.0 mL/min, with a Shodex KF-806L column. Molecular weights and molecular weight distributions were estimated on the basis of the calibration curve obtained by polystyrene standards. GC analyses were performed on a SHIMADZU GC-14A equipped with a flame ionization detector using SHIMADZU CAPILLARY COLUMN (CBP1-M25-025) and SHIMADZU C-R6A-Chromatopac integrator. Melting Points were measured on a Yanagimoto micromelting point apparatus and are uncorrected. The GC yields were determined using suitable hydrocarbon internal standards. GC/MS analyses were carried out on a SHIMADZU GC-17A equipped with a SHIMADZU QP-5050 GC-MS system. Elemental analyses were carried out with a Perkin-Elmer 2400 CHN elemental analyzer at Osaka City University.

Materials. Ethyl cyanoformate, methyl cyanoformate, and ethyl (*Z*)-3-cyano-2-propenoate were purchased from Aldrich Chemical Co., Inc. 1,4-dihydro-1,4-methanonaphthalene (**2b**)[1] and was prepared according to the literature procedure.

(1) Coe, J. W.; Wirtz, M. C.; Bashore, C. G.; Candler, J. *Org. Lett.* **2004**, *6*, 1589-1592.

General Procedure for Cyanoesterification: Formation of (2*R,3*S**)-Ethyl 3-cyanobicyclo[2.2.1]heptane-2-carboxylate (3a).** To a solution tetrakis(triphenylphosphine)palladium (580 mg, 0.5 mmol, 10 mol %) in toluene (50 mL) were added ethyl cyanoformate (**1a**) (490 μL, 5.0 mmol) and norbornene (**2a**) (470 mg, 5.0 mmol) at room temperature. The reaction mixture was stirred for 24 h at 110 °C, quenched with 1 M hydrochloric acid (50 mL), and extracted with diethyl ether (25 mL x 2). The combined ethereal layer was washed with brine and dried over $MgSO_4$. Filtration and evaporation afforded a pale yellow oil. Bulb to bulb distillation (135 °C/ 2.0 Torr) gave **3a** (773 mg, 80% yield) as colorless oil. GC yield was 94%. FT-IR (neat, cm^{-1}): 2974 (s), 2882 (m), 2240 (m, *v*CN), 1738 (s, *v*CO), 1456 (m), 1375 (m), 1350 (m), 1290 (m), 1263 (m), 1222 (m), 1193 (s), 1154 (m), 1118 (m), 1040 (m), 926 (w), 853 (w), 748 (w). ^{1}H NMR ($CDCl_3$, 300 MHz, rt): δ 1.17-1.25 (m, 2H, ethylene C*H* (*endo*)), 1.28 (dt, J = 7 Hz, 1 Hz, 3H, CH_3), 1.42 (dt, J = 11 Hz, 2 Hz, 1H, one of methylene (*anti*)), 1.53-1.66 (m, 2H, ethylene C*H* (*exo*)), 1.95 (dt, J = 11 Hz, 2 Hz, 1H, one of methylene (*syn*)), 2.62-2.65 (m, 3H, 2 bridgehead methine protons + C*H*COOEt), 2.83 (dd, J = 10 Hz, 2 Hz, 1H, C*H*CN), 4.18 (qd, J = 7 Hz, 1 Hz, 2H, OCH_2); ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz, rt): δ 14.1 (*C*H_3), 27.8 (*C*H_2), 28.2 (CH_2), 36.6 (*C*H_2 + *C*HCN), 39.0 (bridgehead carbon (CN side)), 41.9 (bridgehead carbon (COOEt side)), 49.9 (*C*HCOOEt), 61.1 (O*C*H_2), 119.7 (*C*N), 171.1 (*C*O). MS (EI, m/z (relative intensity)): 193 (M^+, 6), 166 (18), 148 (29), 126 (64), 120 (74), 98 (100), 93 (46), 80 (27), 66 (95), 53 (20). Anal. Calcd for $C_{11}H_{15}NO_2$: C, 68.37; H, 7.82; N, 7.25%. Found: C, 68.24; H, 7.82; N, 6.94%.

Reaction of 1a with 2a in the Presence of a Catalytic Amount of $Pd(PPh_3)_4$ (Table 1). To a solution of $Pd(PPh_3)_4$ (58 mg, 0.05 mmol, 10 mol %) in toluene (3 mL), were added **1a** (49 μL, 0.5 mmol) and norbornene (**2a**, 47 mg, 0.5 mmol), and dodecane (115 μL, 0.5 mmol) as an internal standard. The reaction mixture was heated at 110 °C for 24 h to afford **3a** in 94% GC yield.

(2*R,3*S**)-Methyl 3-cyanobicyclo[2.2.1]heptane-2-carboxylate (3b).** Preparation of **3b** was carried out analogously to **3a** using **1a** and **2b.** Colorless liquid. Bp. 130 °C/1.6 Torr. Isolated yield was 28%. FT-IR (neat, cm^{-1}): 2962 (s), 2882 (m), 2240 (m, *v*CN), 1742 (s, *v*CO), 1458 (m), 1437 (m), 1363 (m), 1290 (m), 1265 (m), 1224 (m), 1197 (s), 1154 (m), 1120 (m), 1040 (m), 934 (w), 870 (w), 770 (w). ^{1}H NMR ($CDCl_3$, 300 MHz, rt): δ 1.24 (m, 2H, ethylene CH (*endo*)), 1.43 (dt, J = 11 Hz, 2 Hz, 1H, one of methylene (*anti*)), 1.62 (m, 2H, ethylene CH (*exo*)), 1.96 (dt, J = 11 Hz, 2 Hz, 1H, one of methylene (*syn*)), 2.60-2.69 (m, 3H, 2 bridgehead methine protons + C*H*COOMe), 2.83 (dd, J = 10 Hz, 2 Hz, 1H, C*H*CN), 3.74 (s, 3H, OCH_3); ^{13}C{^{1}H} NMR ($CDCl_3$, 75 MHz, rt): δ 27.7 (*C*H_2), 28.2 (*C*H_2), 36.6 (*C*H_2 + *C*HCN), 39.0 (bridgehead carbon (CN side)), 41.9 (bridgehead carbon (COOMe side)), 50.1 (*C*HCOOMe), 52.1 (O*C*H_3), 119.9 (*C*N), 171.8 (*C*O). MS (EI, m/z (relative intensity)): 179 (M^+, 1), 164 (2), 148 (14), 134 (2), 120 (42), 112 (100), 98 (40), 93 (22), 80 (29), 67 (60), 53 (15). Anal. Calcd for $C_{10}H_{13}NO_2$: C, 67.02; H, 7.31; N, 7.82%. Found: C, 67.23; H, 7.33; N, 7.86%.

(2*R,3*S**)-Ethyl 1,2,3,4-tetrahydro-3-cyano-1,4-methanonaphthalene-2-carboxylate (3c)**. White solid. Mp. 66-67 °C. Isolated yield was 83%. FT-IR (KBr, cm^{-1}): 2979 (s), 28860 (m), 2240 (s, *v*CN), 1739 (s, *v*CO), 1468 (m), 1374 (m), 1347 (m), 1312 (m), 1264 (m), 1240 (m), 1181 (s), 1156 (m), 1107 (m), 1036 (m), 951 (w), 857 (w), 754 (w). ^{1}H NMR ($CDCl_3$, 300 MHz, rt): δ 1.33 (t, J = 7 Hz, 3H, CH_3), 2.02 (d of quintet, J = 10 Hz, 2 Hz, 1H, one of methylene (*anti*)), 2.36 (dt, J =10 Hz, 2 Hz, 1H, one of methylene (*syn*)), 2.70 (dd, J

= 10 Hz, 2 Hz, 1H, C*H*COOEt), 2.85 (dd, *J* = 9 Hz, 2 Hz, 1H, C*H*CN), 3.72 (d, *J* = 7 Hz, 2H, 2 bridgehead methine protons), 4.28 (qd, *J* = 7 Hz, 4 Hz, 2H, OCH_2), 7.11-7.27 (m, 4H, aromatics); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75 MHz, rt): δ 14.1 (*C*H_3), 34.9 (*C*HCN), 46.5 (bridgehead carbon (CN side)), 47.1 (*C*H_2), 48.8 (bridgehead carbon (COOEt side) + *C*HCOOEt), 61.6 (O*C*H_2), 119.7 (*C*N), 121.2 , 121.7, 126.7, 127.3, 144.4, 146.0, 171.5 (*C*O). MS (EI, m/z (relative intensity)): 241 (M^+, 4), 196 (2), 167 (6), 141 (7), 126 (5), 116 (100), 98 (10), 89 (4), 80 (4), 63 (4), 51 (3). Anal. Calcd for $C_{15}H_{15}NO_2$: C, 74.67; H, 6.27; N, 5.80%. Found: C, 74.48; H, 6.24; N, 5.63%.

(2*R,3*S**)-Methyl 1,2,3,4-tetrahydro-3-cyano-1,4-methanonaphthalene-2-carboxylate (3d).** Preparation of **3d** was carried out analogously to **3c** using **1b** and **2b.** Colorless liquid. Bp. 175 °C/1.8 Torr. GC yield was 74%. Isolated yield was 65%. FT-IR (neat, cm^{-1}): 3024 (m), 2956 (m), 2242 (m, *v*CN), 1740 (s, *v*CO), 1462 (m), 1437 (m), 1352 (m), 1311 (m), 1267 (m), 1238 (m), 1201 (s), 1183 (m), 1156 (m), 1110 (m), 1038 (m), 959 (w), 849 (w), 756 (w). ^1H NMR ($CDCl_3$, 300 MHz, rt): δ 2.02 (d of quintet, *J* = 10.Hz, 2 Hz, 1H, one of methylene (*anti*)), 2.36 (dt, *J* = 11 Hz, 2Hz, 1H, one of methylene (*syn*)), 2.73 (dd, *J* = 10 Hz, 2 Hz, 1H, C*H*COOEt), 2.85 (dd, *J* = 10 Hz, 2 Hz, 1H, C*H*CN), 3.72 (d, *J* = 6 Hz, 2H, 2 bridgehead methine protons), 3.82 (s, 3H, OCH_3), 7.11-7.27 (m, 4H, aromatics); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75 MHz, rt): δ 34.9 (*C*HCN), 46.3 (bridgehead carbon (CN side)), 47.2 (*C*H_2), 48.9 (*C*HCOOMe), 49.1 (bridgehead carbon (COOMe side)), 52.5 (O*C*H_3), 119.8 (*C*N), 121.3, 121.8, 126.9, 127.5, 144.5, 146.0, 172.0 (*C*O). MS (EI, m/z (relative intensity)): 227 (M^+, 5), 196 (2), 167 (5), 141 (6), 128 (3), 116 (100), 102 (1), 89 (3), 80 (5), 63 (4), 51 (3). Anal. Calcd for $C_{14}H_{13}NO_2$: C, 73.99; H, 5.77; N, 6.16%. Found: C, 73.74; H, 5.71; N, 6.02%.

(2*R,3*S**)-Ethyl 3-cyanobicyclo[2.2.1]hept-5-ene-2-carboxylate (5)**. Colorless liquid. Bp. 135 °C/ 1.9 Torr. (5.0 mmol scale, 635 mg, 3.32 mmol). Isolated yield was 66%. GC yield was 83%. FT-IR (neat, cm^{-1}): 2986 (m), 2240 (m, *v*CN), 1736 (s, *v*CO), 1460 (m), 1373 (m), 1344 (m), 1265 (m), 1243 (m), 1189 (s), 1160 (m), 1112 (m), 1038 (m), 907 (w), 822 (w), 772 (w). ^1H NMR ($CDCl_3$, 300 MHz, rt): δ 1.31 (t, *J* = 7 Hz, 3H, CH_3), 1.66 (dt, *J* = 10 Hz, 2 Hz, 1H, one of methylene (*anti*)), 2.00 (d, *J* = 10 Hz, 1H, one of methylene (*syn*)), 2.57 (dd, *J* = 9 Hz, 2 Hz, 1H, C*H*COOEt), 2.68 (dd, *J* = 9 Hz, 2 Hz, 1H, C*H*CN), 3.20 (brs, 1H, bridgehead methine proton (CN side)), 3.26 (brs, 1H, methine), 4.24 (m, 2H, OCH_2), 6.15 (dd, *J* = 6 Hz, 3 Hz, 1H, =CH), 6.24 (dd, *J* = 6 Hz, 3 Hz, 1H, =CH); $^{13}C\{^1H\}$ NMR ($CDCl_3$, 75 MHz, rt): δ 14.3 (*C*H_3), 33.1 (*C*HCN), 45.0 (bridgehead carbon (CN side)), 46.5 (*C*H_2), 47.0 (*C*HCOOEt), 47.6 (bridgehead carbon (COOEt side)), 61.6 (O*C*H_2), 120.2 (*C*N), 135.9 (=*C*H), 138.4 (=*C*H), 171.8 (*C*O). MS (EI, m/z (relative intensity)): 191 (M^+, 1), 146 (5), 126 (2), 118 (5), 98 (4), 90 (2), 80 (8), 66 (100), 52 (3). Anal. Calcd for $C_{11}H_{13}NO_2$: C, 69.09; H, 6.85; N, 7.32%. Found: C, 69.07; H, 6.74; N, 7.13%.

Diels-Alder Reaction of Cyclopentadiene with ethyl-cis-(β-cyano)acrylate. Formation of (2*S,3*R**)-Ethyl 3-cyanobicyclo[2.2.1]hept-5-ene-2-carboxylate (5') as a major stereoisomer.** A solution of ethyl (*Z*)-3-cyano-2-propenoate (344 μL, 5.0 mmol) in CH_2Cl_2 (10 mL), was added freshly cracked cyclopentadiene (1.03 mL, 12.5 mmol) at 0 °C. After being stirred for additional 1 h at 0 °C, the reaction mixture was warmed to room temperature, stirred overnight. The volatiles were evaporated under reduced pressure to result in a brown oil. (5.0 mmol scale, 729 mg, 3.81 mmol). Isolated yield was 76%. Colorless liquid. FT-IR (neat, cm^{-1}): 2986 (m), 2242 (m, *v*CN), 1736 (s, *v*CO), 1460 (m), 1375 (m), 1336 (m), 1253 (m), 1253 (m), 1189 (s),

1098 (m), 1042 (m), 913 (w), 835 (w), 772 (w). 2-*endo*,3-*endo*-**5'** as a major stereoisomer; ^{1}H NMR ($CDCl_3$, 300 MHz, rt): δ 1.28 (t, J = 7 Hz, 3H, CH_3), 1.57 (dt, J = 9 Hz, 2 Hz, 1H, one of methylene), 3.22-3.32 (m, 3H, methine), 4.16 (m, 2H, OCH_2), 6.30 (dd, J = 6 Hz, 3 Hz, 1H, =CH), 6.46 (dd, J = 6 Hz, 3 Hz, 1H, =CH). Other proton signals were not unambiguously assigned due to the overlapping with those of **5**. $^{13}C\{^{1}H\}$ NMR ($CDCl_3$, 75 MHz, rt): δ 14.2 (CH_3), 33.2 (*C*HCN), 44.9 (bridgehead carbon (CN side)), 46.8 (CH_2), 47.9 (*C*HCOOEt), 48.6 (bridgehead carbon (COOEt side)), 61.1 (OCH_2), 119.8 (*C*N), 133.8 (=*C*H), 137.7 (=*C*H), 170.2 (*C*O). MS (EI, m/z (relative intensity)): 191 (M^+, 2), 146 (6), 126 (1), 118 (6), 98 (4), 91 (4), 80 (9), 66 (100), 52 (4). Anal. Calcd for $C_{11}H_{13}NO_2$: C, 69.09; H, 6.85; N, 7.32%. Found: C, 68.85; H, 6.67; N, 7.16%. Minor signals of ^{1}H and $^{13}C\{^{1}H\}$ NMR are completely identical to those of 2-*exo*,3-*exo*-**5**.

Preparation of *trans*-Pd(PPh$_3$)$_2$(CN)(CO$_2$Et) (6a). To a toluene (20 mL) suspension of $Pd(PPh_3)_4$ (693 mg, 0.60 mmol) was added ethyl cyanoformate (142 μL, 1.44 mmol). The reaction mixture was stirred at room temperature. The initially pale yellow suspension became white suspension after 48 h. The solvents were evaporated under vacuum. The resulting off-white solid was washed with hexane (20 mL) two times. The product was extracted with dichloromethane (10 mL). Removal of the solvent from the extracts gave **6a** (430 mg, 0.59 mmol, 98%). Recrystallization from dichloromethane/hexane afforded colorless needles (308 mg, 0.42 mmol, 70%). Mp. 131-132 °C (dec.). FT-IR (KBr, cm^{-1}): 2126 (w, νCN), 1638 (s, νCO). ^{1}H NMR (CD_2Cl_2, 300 MHz, rt): δ 0.52 (t, J = 7 Hz, 3H, CH_3), 2.73 (q, J = 7 Hz, 2H, CH_2), 7.38-7.51 (m, 18H, Ph), 7.66-7.78 (m, 12H, Ph). $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 75 MHz, rt): δ 13.2 (s, CH_3), 59.6 (s, OCH_2), 127.8 (t, J = 5 Hz, *ortho*-PPh_3), 130.1 (s, *para*- PPh_3), 131.0 (t, J = 24 Hz, *ipso*-PPh_3), 133.8 (t, J = 7 Hz, *meta*-PPh_3), 137.2 (t, J = 20 Hz, Pd-*C*N), 192.6 (t, J = 3 Hz, Pd-*C*O). $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 121 MHz, rt): δ 21.6 (s). Anal. Calcd for $C_{40}H_{35}NO_2P_2Pd$: C, 65.81; H, 4.83; N, 1.92%. Found: C, 65.78; H, 4.75; N, 1.88%.

Preparation of *trans*-Pd(PPh$_3$)$_2$(CN)(CO$_2$Me) (6b). To a toluene (20 mL) suspension of $Pd(PPh_3)_4$ (693 mg, 0.60 mmol) was added methyl cyanoformate (114 μL, 1.44 mmol). The reaction mixture was stirred at room temperature. The initially pale yellow suspension became white suspension after 24 h. The solvents were evaporated under vacuum. The resulting off-white solid was washed with hexane (20 mL) two times. The product was extracted with dichloromethane (10 mL). Removal of the solvent from the extracts gave the titled compound (416 mg, 0.58 mmol, 97%). Recrystallization from dichloromethane/hexane afforded colorless crystals of **6b** (364 mg, 0.51 mmol, 84%). Mp. 163-164 °C (dec). FT-IR (KBr, cm^{-1}): 2124 (w, νCN), 1663 (s, νCO). ^{1}H NMR (CD_2Cl_2, 300 MHz, rt): δ 2.44 (s, 3H, OCH_3), 7.39-7.55 (m, 18H, Ph), 7.67-7.79 (m, 12H, Ph). $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 75 MHz, rt): δ 50.0 (s, OCH_3), 127.8 (t, J = 5 Hz, *ortho*-PPh_3), 130.2 (s, *para*-PPh_3), 130.9 (t, J = 24 Hz, *ipso*-PPh_3), 133.8 (t, J = 7 Hz, *meta*-PPh_3), 136.8 (t, J = 20 Hz, Pd-*C*N), 192.9 (t, J = 2 Hz, Pd-*C*O). $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 121 MHz, rt): δ 21.8 (s). Anal. Calcd for $C_{39}H_{33}NO_2P_2Pd$: C, 65.42; H, 4.65; N, 1.96%. Found: C, 65.46; H, 4.61; N, 1.84%.

Reaction of 1a with 2a in the Presence of a Catalytic Amount of *trans*-Pd(PPh$_3$)$_2$(CN)(CO$_2$Et) (6a). To a solution of **6a** (15 mg, 0.02 mmol, 10 mol %) in toluene (2 mL), were added **1a** (20 μL, 0.2 mmol) and norbornene (**2a**, 19 mg, 0.2 mmol), and dodecane (46 μL, 0.2 mmol) as an internal standard. The reaction mixture was heated at 110 °C for 24 h to afford **3a** in 52% GC yield.

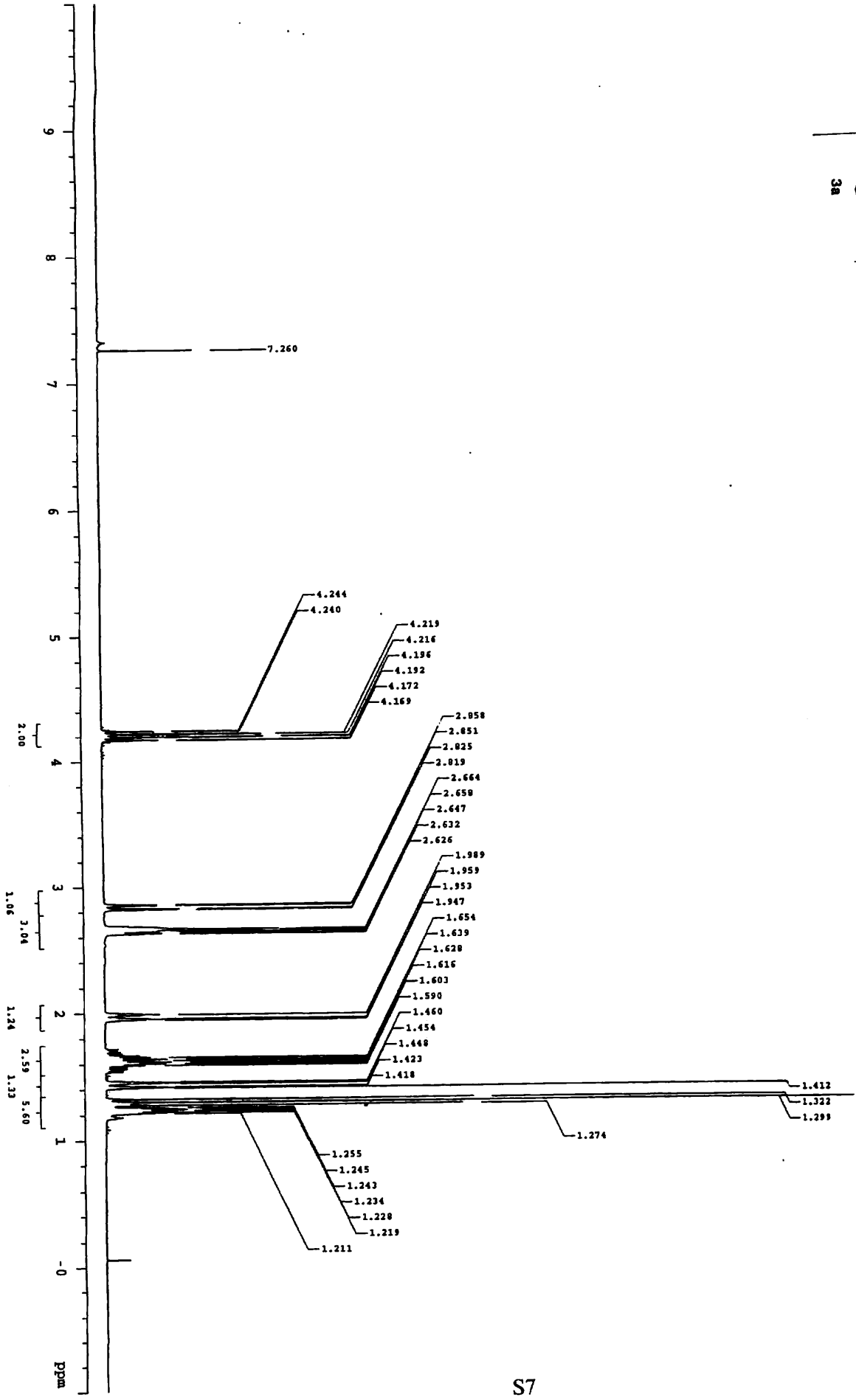
Stoichiometric Reaction of 2a with *trans*-Pd(PPh$_3$)$_2$(CN)(CO$_2$Et) (6a). To a solution of the isolated **6a** (36.5 mg, 0.05 mmol) in toluene-d_8 (0.6 mL), were added an excess (5 equiv) of norbornene (**2a**, 24 mg, 0.25 mmol) and mesitylene (7 μL, 0.05 mmol) as an internal standard. An NMR tube was heated at 110 °C for 24 h to afford **3a** in 70% NMR yield.

Stoichiometric Reaction of Ethylene with *trans*-Pd(PPh$_3$)$_2$(CN)(CO$_2$Et) (6a). To a solution of the isolated **6a** (146 mg, 0.20 mmol) in toluene-d_8 (2.4 mL), were added ethylene (5 atm) and mesitylene (28 μL, 0.20 mmol) as an internal standard. An autoclave was heated at 110 °C for 24 h. ^{1}H NMR showed the presence of neither acrylonitrile nor ethyl acrylate in the reaction mixture.

Decomposition of *trans*-Pd(PPh$_3$)$_2$(CN)(CO$_2$Et) (6a). A colorless solution of **6a** (51 mg, 0.07 mmol) in toluene (20 mL) was left at room temperature for 3 weeks in a Schlenk tube under Ar. Extensive deposit of brown solid was observed. The ^{1}H and ^{31}P{^{1}H} NMR spectroscopies showed *trans*-Pd(CN)$_2$(PPh$_3$)$_2$ [2] being generated in 43% yield, along with several unidentified compounds. ^{1}H NMR (300 MHz, CD_2Cl_2): δ 7.39-7.57 (m, 18H, Ph), 7.61-7.74 (m, 12H, Ph). ^{31}P{^{1}H} NMR (121 MHz, CD_2Cl_2): δ 23.4 (s).

X-Ray Crystallogrtaphy. Single crystals of **6b** suitable for X-ray diffraction study were obtained by recrystallization from toluene-hexane at room temperature. Data were collected at 113 K on Rigaku Saturn CCD diffractometer equipped with monochromated MoKα radiation (λ = 0.7107 Å). Calculations were carried out by using a program package CrystalStructure for Windows. A full-matrix least-squares refinement was used for the non-hydrogen atoms with anisotropic thermal parameters. Crystallographic data for the structural analyses have been deposited with the Cambridge Crystallographic Data Centre. Copies of this information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336-033; e-mail: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

(2) Uson, R.; Fornies, J.; Uson, M. A.; Lalinde, E. *J. Organomet. Chem.* **1980**, *185*, 359-366.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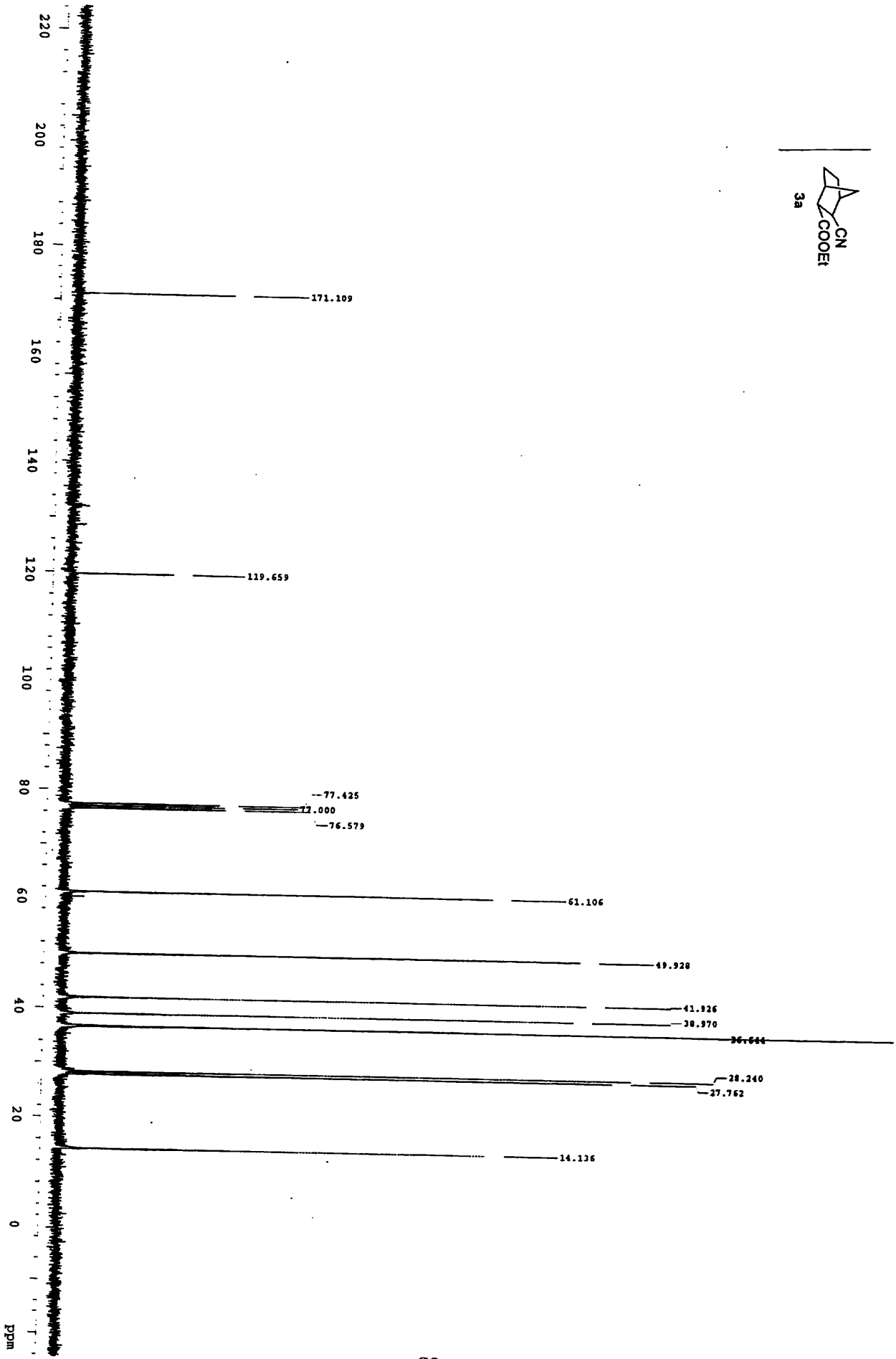
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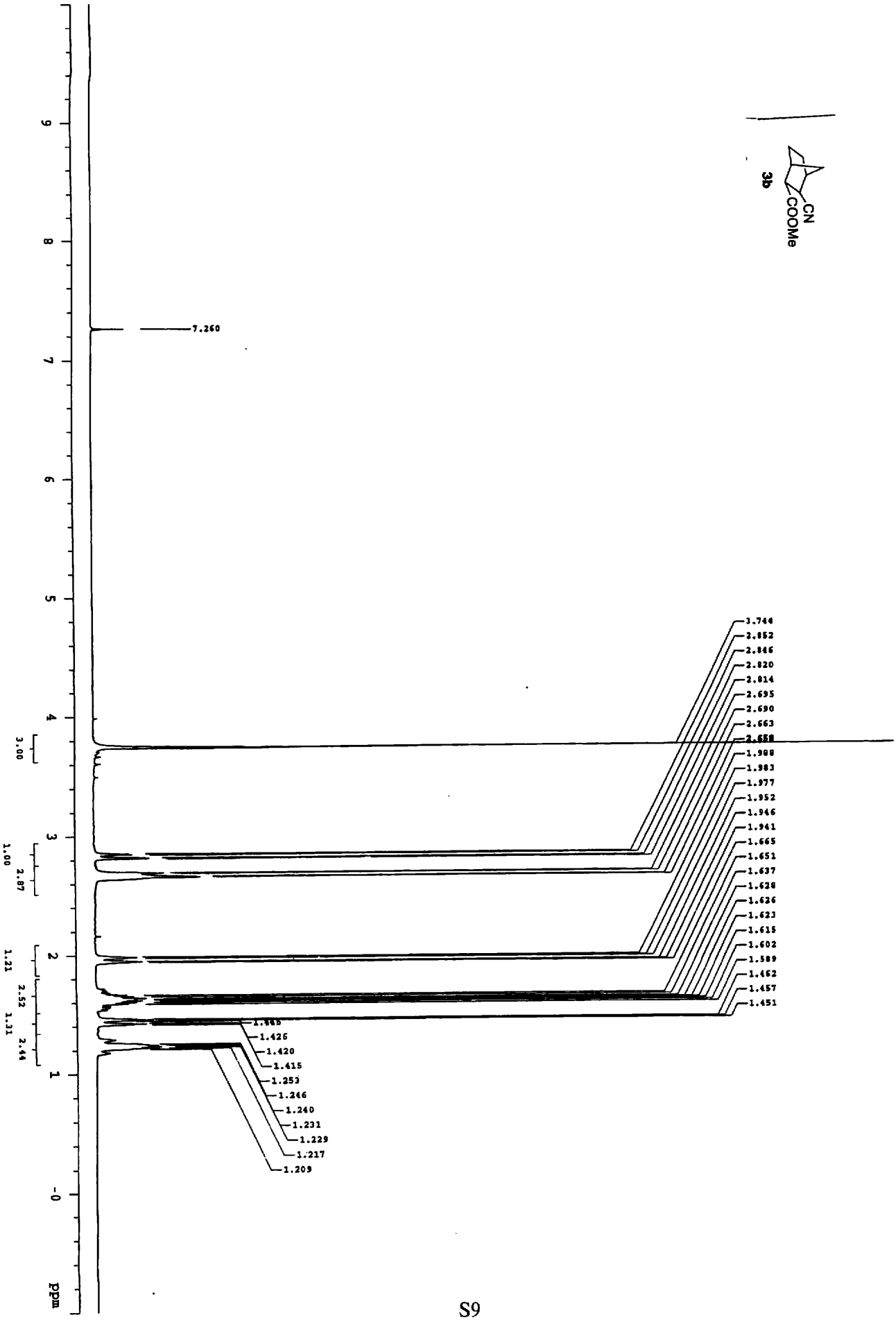
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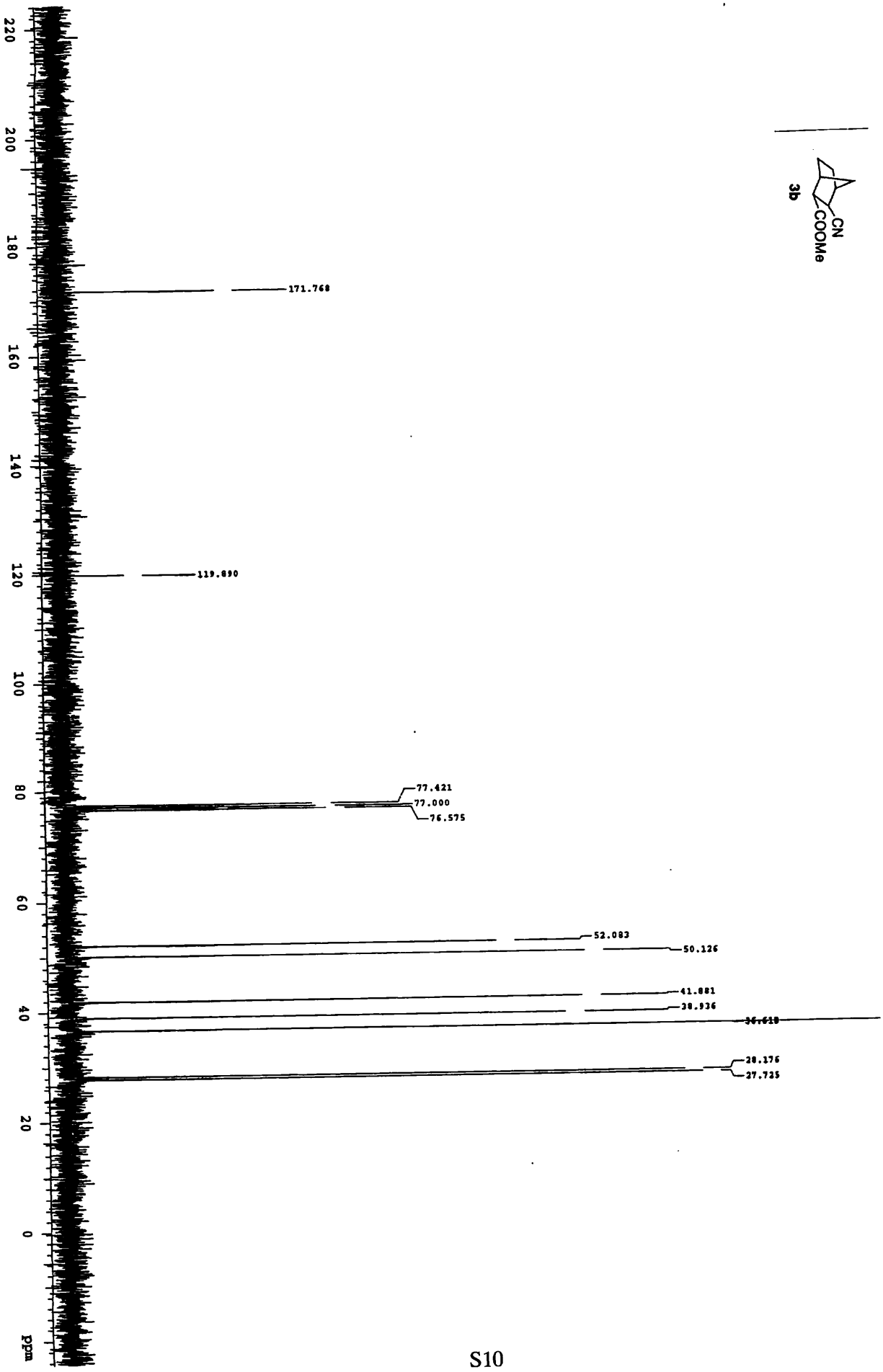
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28.176
27.725
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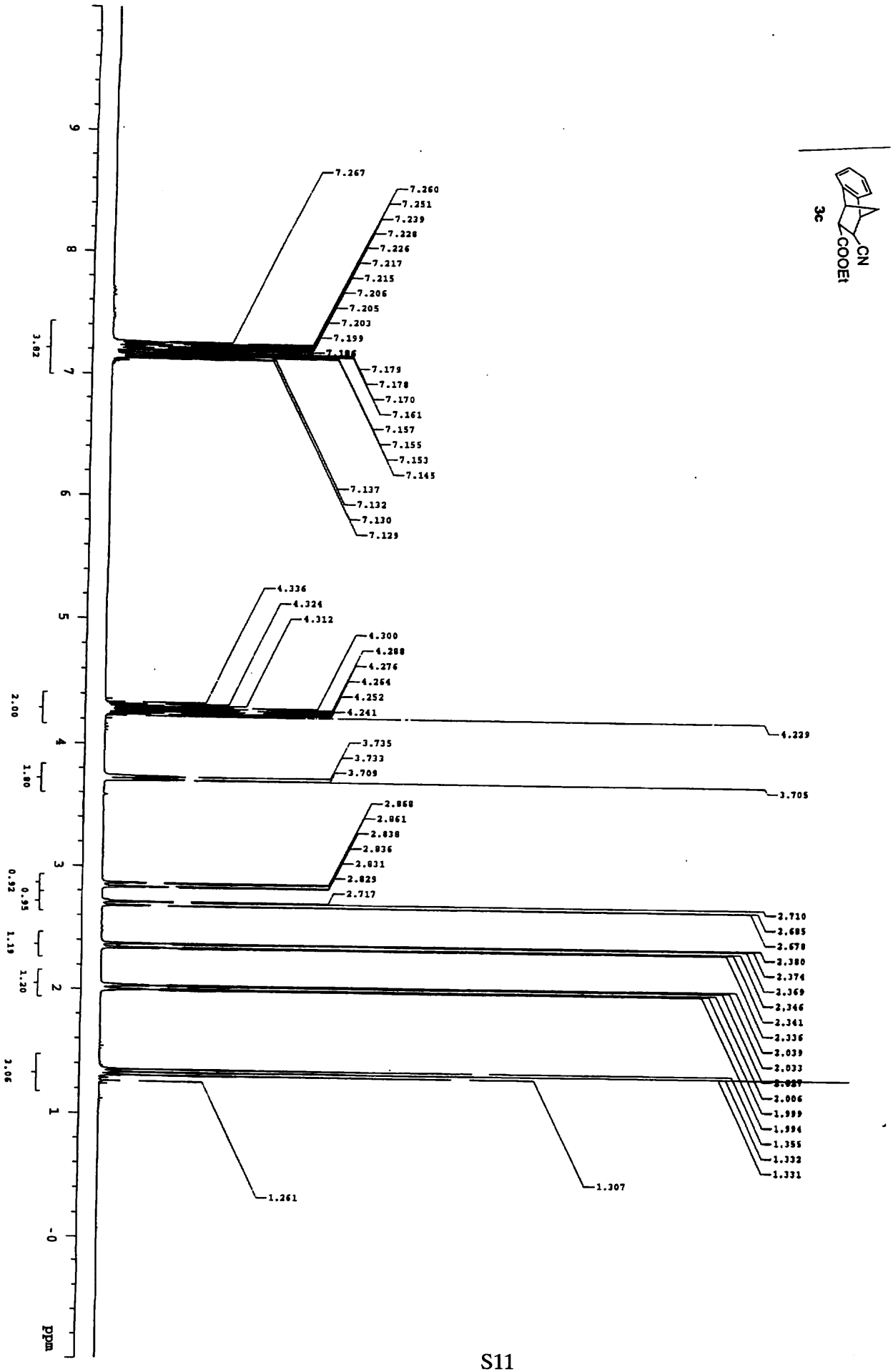
200
180
160
140
120
100
80
60
40
20
0
ppm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COOEt
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7.215
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7.203
7.199
7.186
7.179
7.178
7.170
7.161
7.157
7.155
7.153
7.145
7.137
7.132
7.130
7.129
4.336
4.324
4.312
4.300
4.288
4.276
4.264
4.252
4.241
4.229
3.735
3.733
3.709
3.705
2.868
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2.838
2.836
2.831
2.829
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2.710
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2.374
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2.346
2.341
2.336
2.039
2.033
2.006
1.999
1.994
1.355
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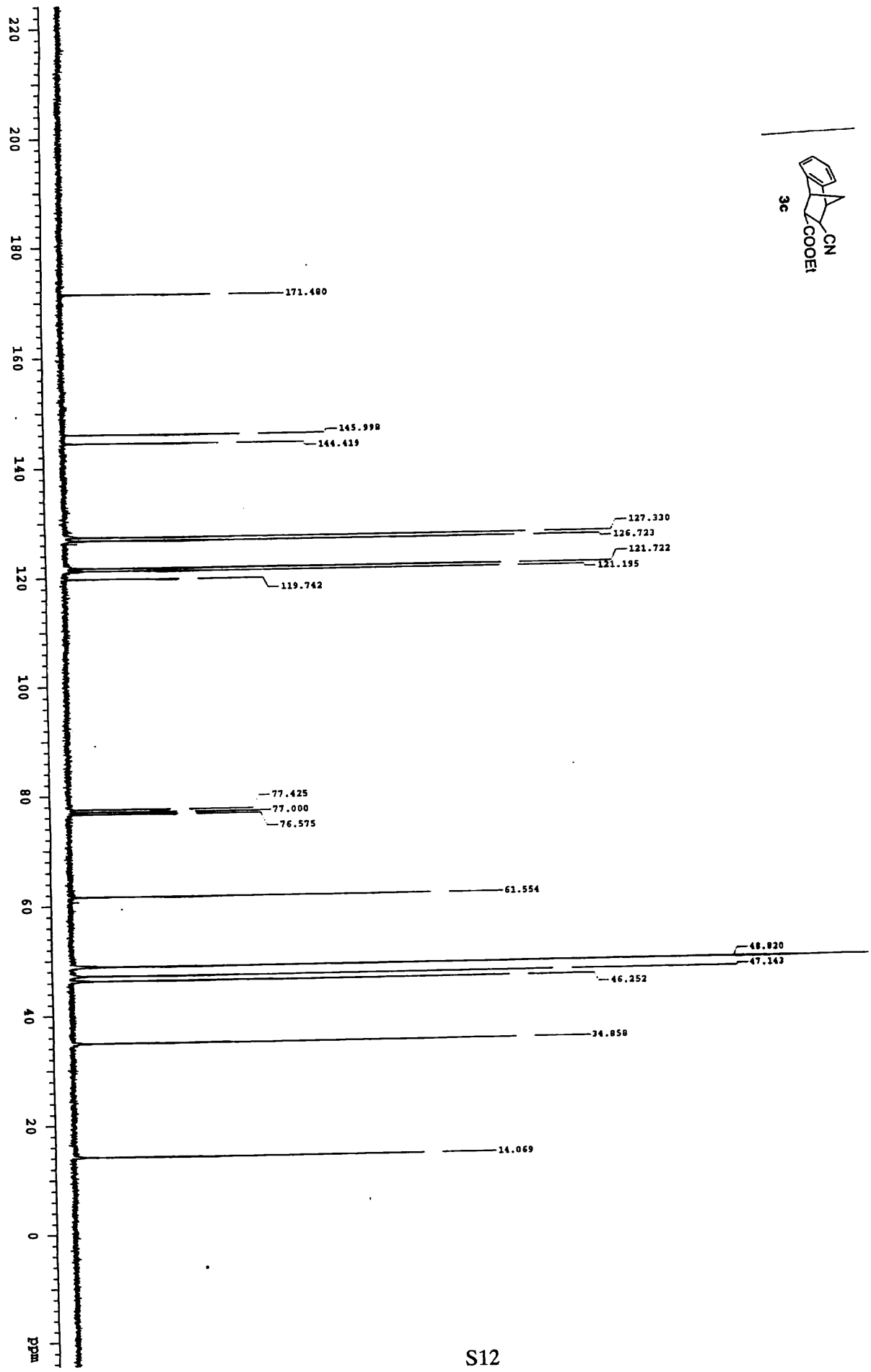
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76.575
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34.858
14.069
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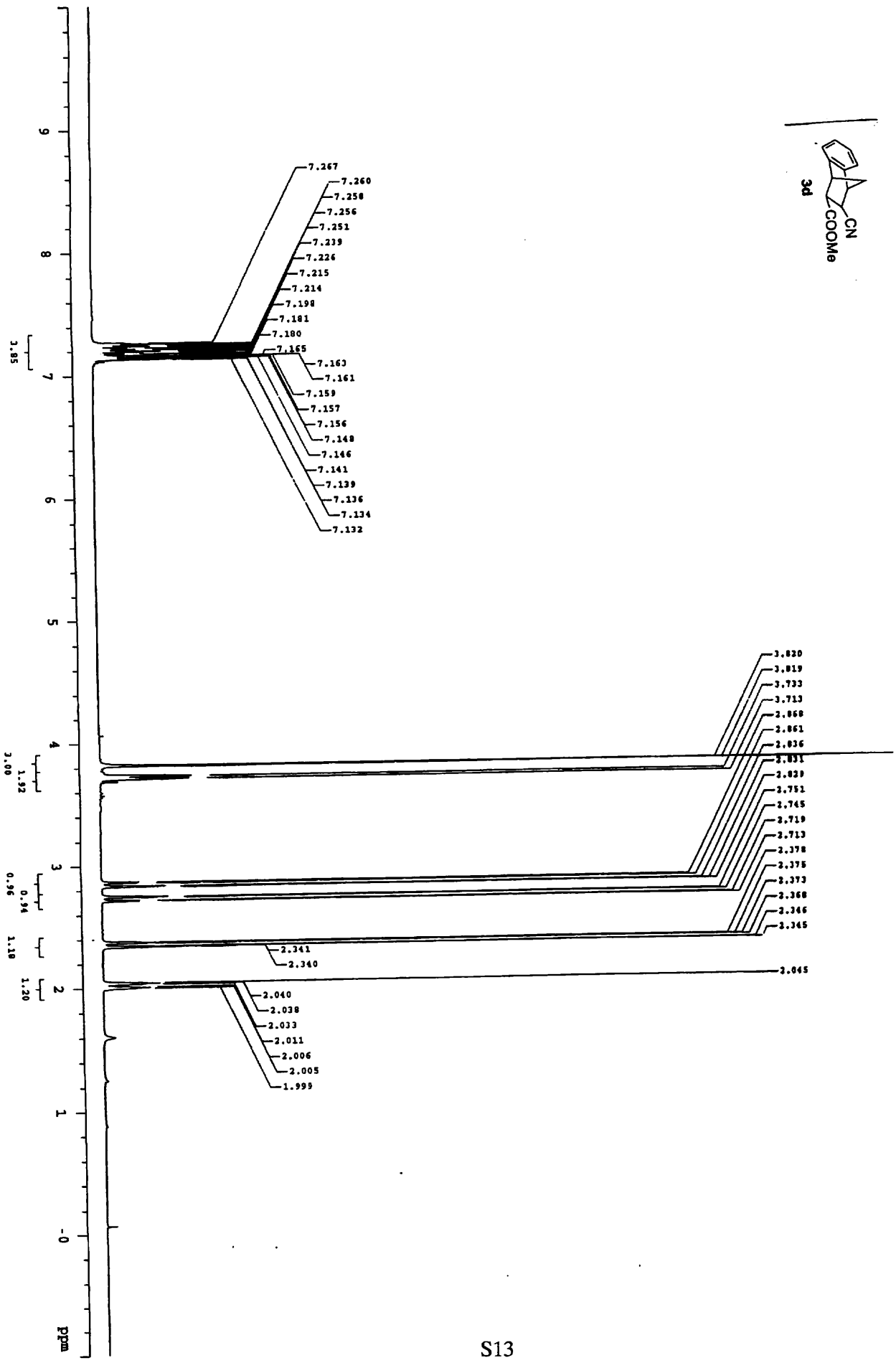
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7.180
7.165
7.163
7.161
7.159
7.157
7.156
7.148
7.146
7.141
7.139
7.136
7.134
7.132
3.820
3.819
3.733
3.713
2.868
2.861
2.836
2.831
2.829
2.751
2.745
2.719
2.713
2.378
2.375
2.373
2.368
2.346
2.345
2.341
2.340
2.045
2.040
2.038
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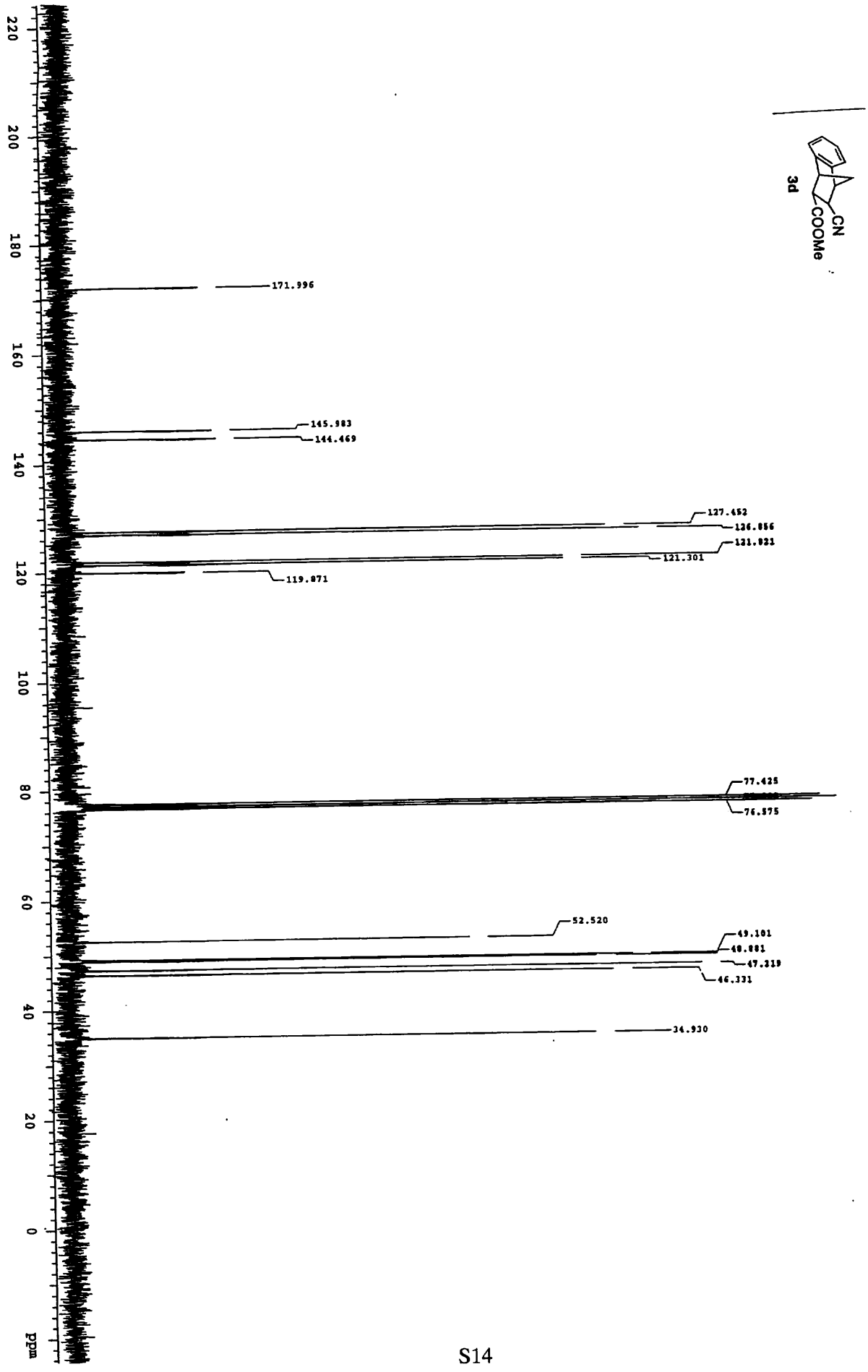
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126.856
121.921
121.301
119.871
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76.575
52.520
49.101
48.881
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46.331
34.930
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200
180
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140
120
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40
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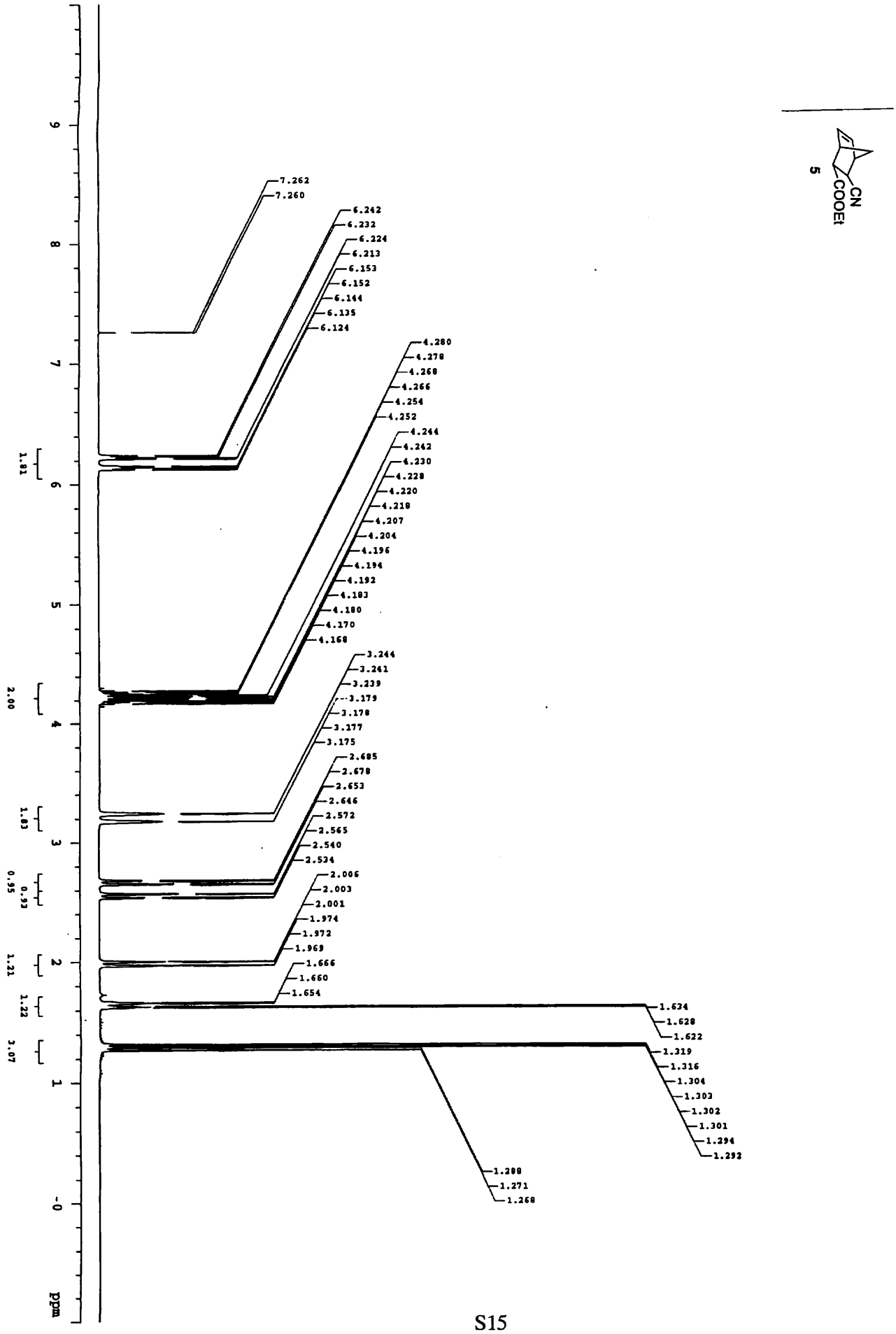
ppm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6.124
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4.278
4.268
4.266
4.254
4.252
4.244
4.242
4.230
4.228
4.220
4.218
4.207
4.204
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4.192
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4.170
4.168
3.244
3.241
3.239
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3.178
3.177
3.175
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2.565
2.540
2.534
2.006
2.003
2.001
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1.972
1.969
1.666
1.660
1.654
1.634
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1.304
1.303
1.302
1.301
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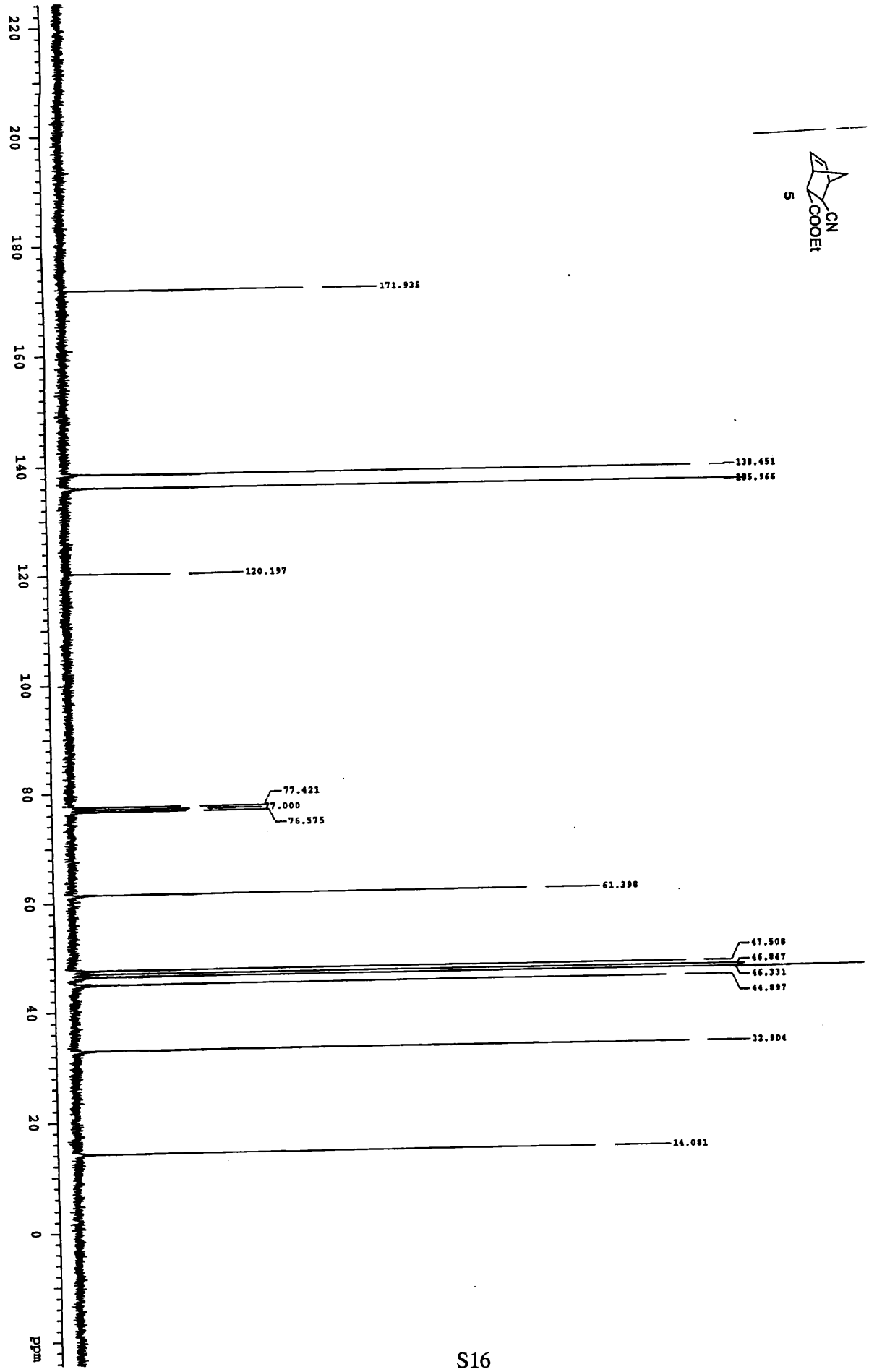
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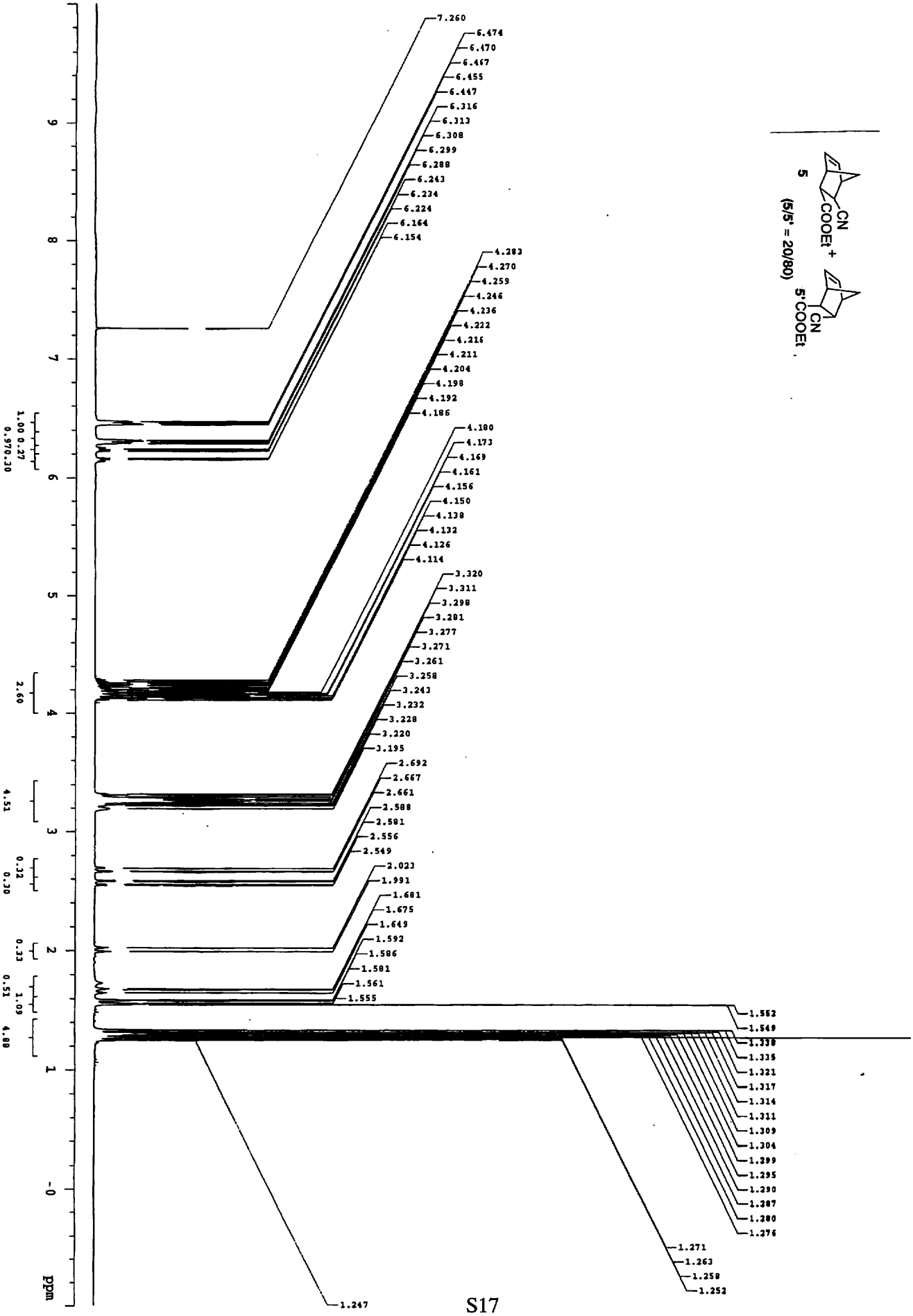
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6.467
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6.447
6.316
6.313
6.308
6.299
6.288
6.243
6.234
6.224
6.164
6.154
4.283
4.270
4.259
4.246
4.236
4.222
4.216
4.211
4.204
4.198
4.192
4.186
4.180
4.173
4.169
4.161
4.156
4.150
4.138
4.132
4.126
4.114
3.320
3.311
3.298
3.281
3.277
3.271
3.261
3.258
3.243
3.232
3.228
3.220
3.195
2.692
2.667
2.661
2.588
2.581
2.556
2.549
2.023
1.991
1.681
1.675
1.649
1.592
1.586
1.581
1.561
1.555
1.552
1.549
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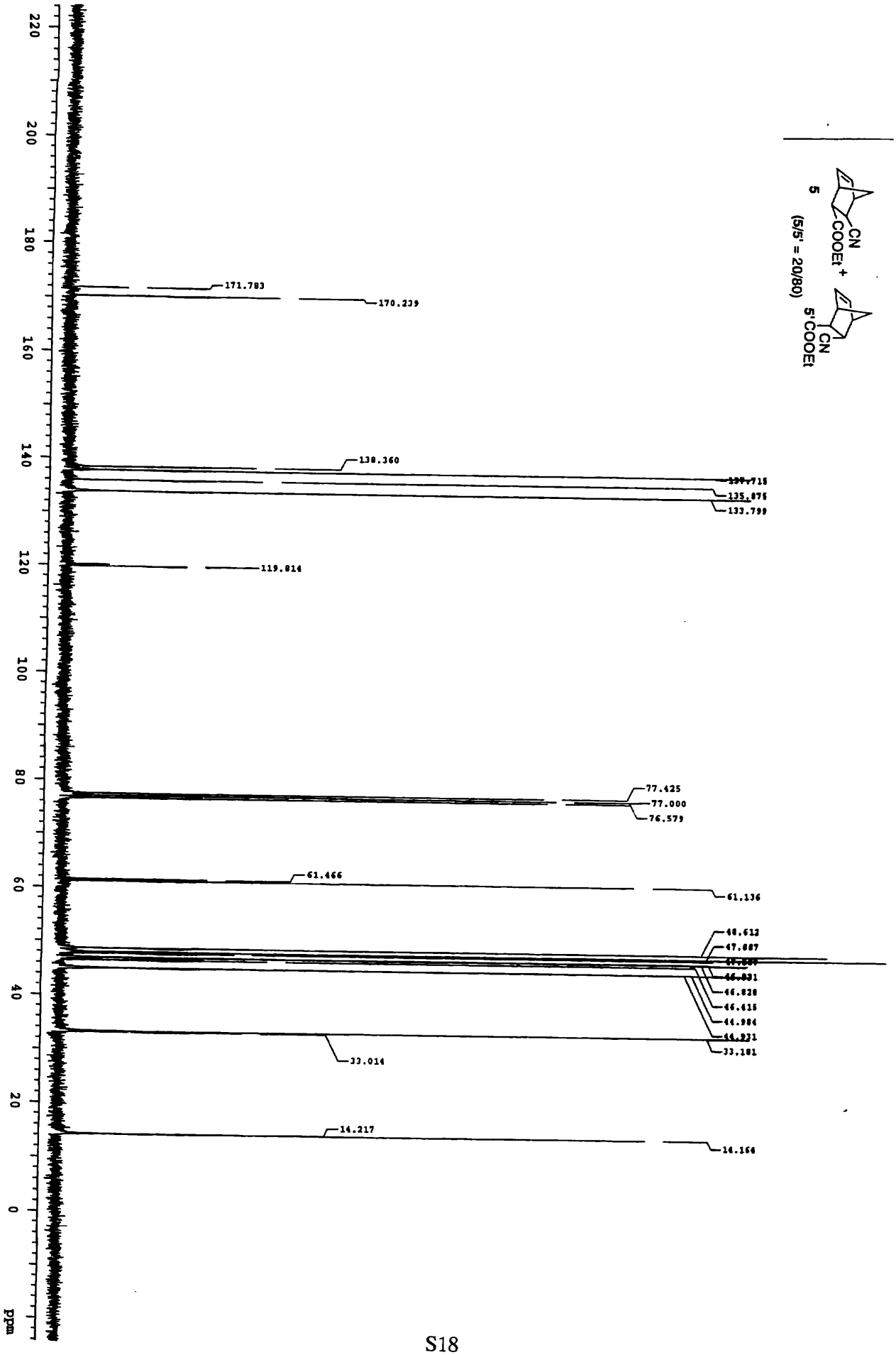
1.335
1.321
1.317
1.314
1.311
1.309
1.304
1.299
1.295
1.290
1.287
1.280
1.276
1.271
1.263
1.258
1.252
1.247
1.00 0.27
0.97 0.30
2.60
4.51
0.32
0.30
0.33
0.51
1.09
4.88
ppm
CN
COOEt
+
CN
5'COOEt
5
(5/5' = 20/80)


5
CN
COOEt +
CN
5'COOEt
(5/5' = 20/80)
171.783
170.239
138.360
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119.814
77.425
77.000
76.579
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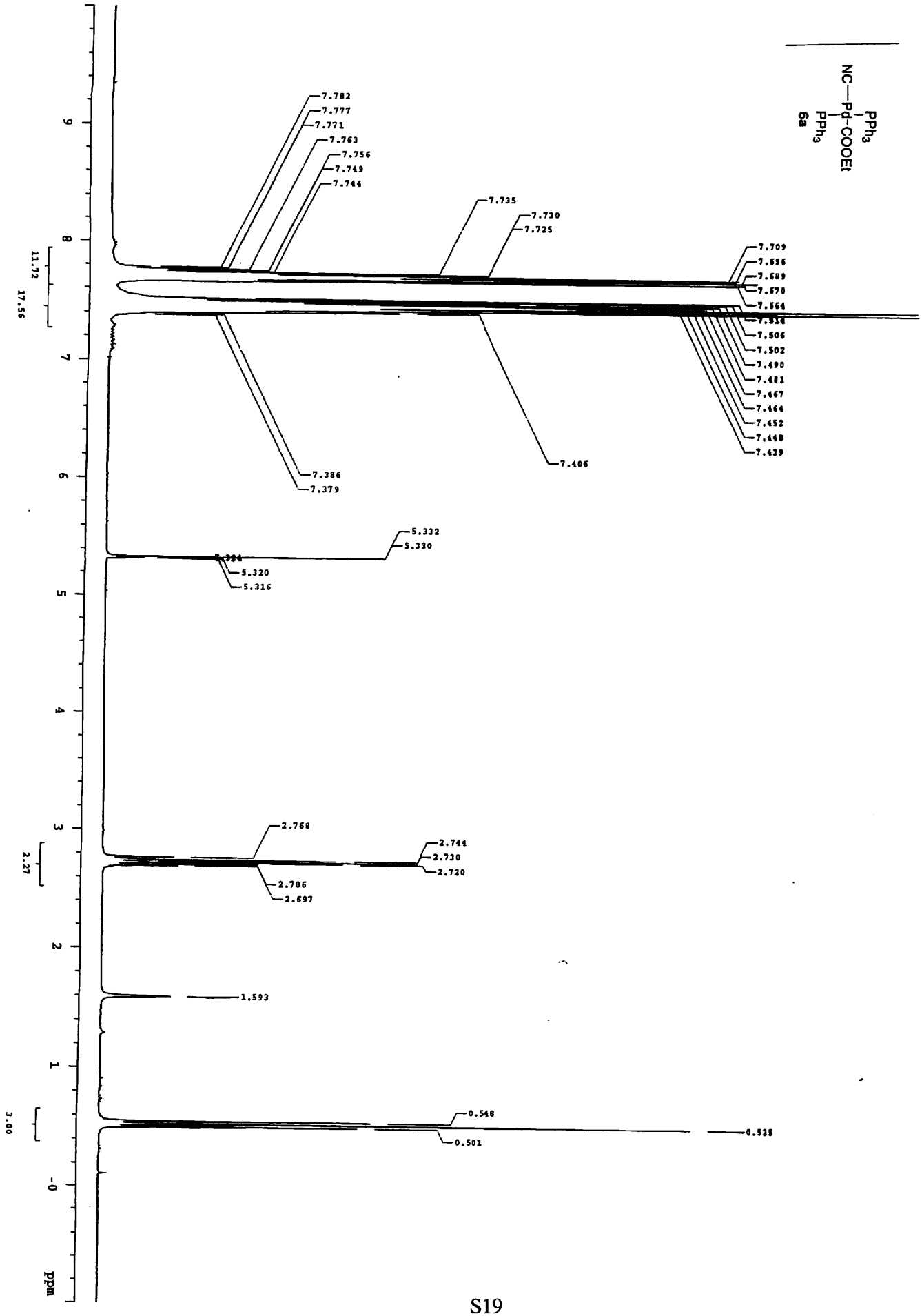
61.136
33.014
14.217
14.166
ppm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NC—Pd—COOEt
PPh3
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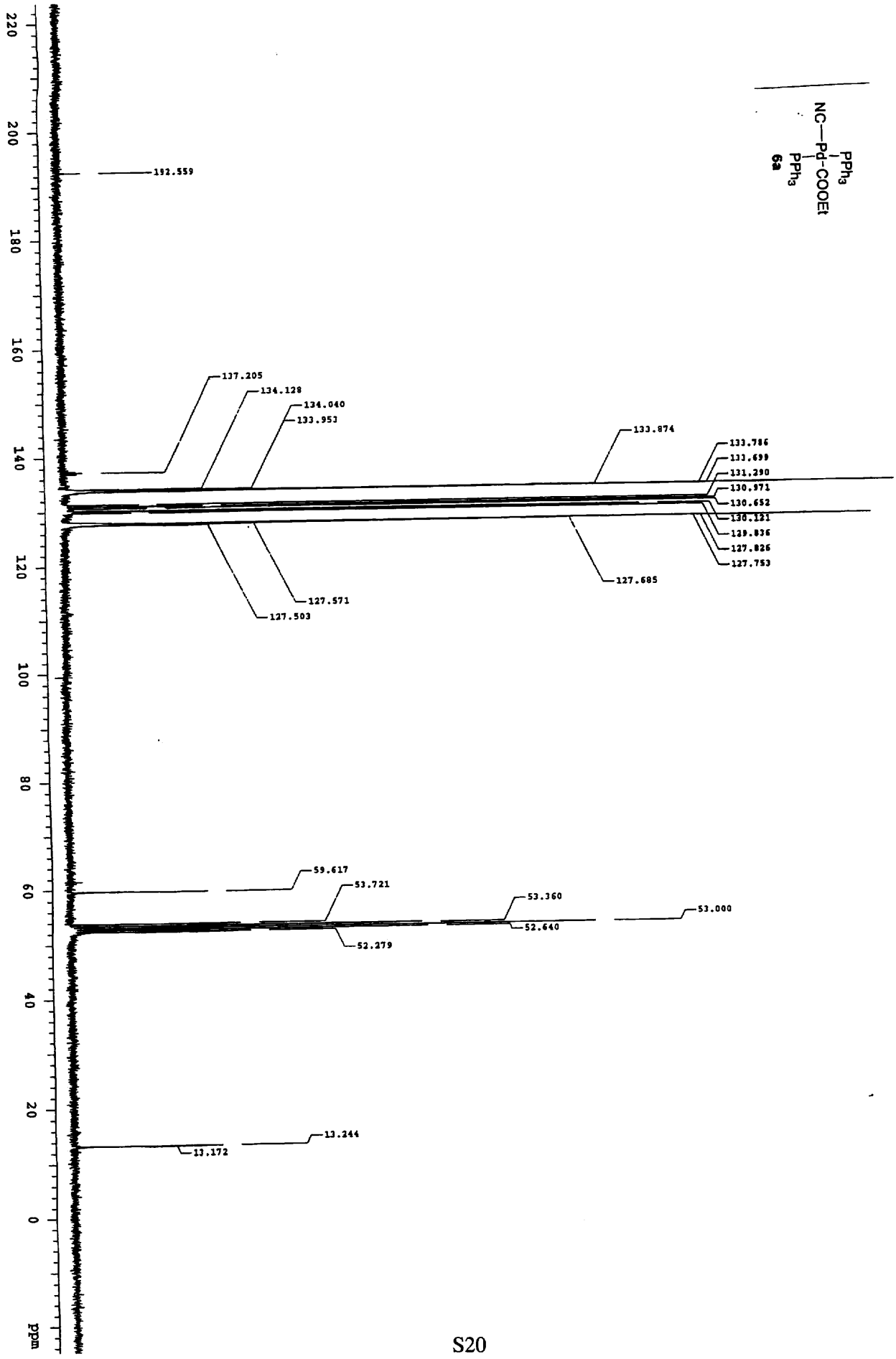
7.763
7.756
7.749
7.744
7.735
7.730
7.725
7.709
7.696
7.689
7.670
7.664
7.506
7.502
7.490
7.481
7.467
7.464
7.452
7.448
7.429
7.406
7.386
7.379
5.332
5.330
5.320
5.316
2.768
2.744
2.730
2.720
2.706
2.697
1.593
0.548
0.535
0.501
11.72
17.56
2.27
3.00
ppm


PPh3
NC—Pd-COOEt
PPh3
6a
192.559
137.205
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134.040
133.953
133.874
133.786
133.699
131.290
130.971
130.652
130.121
129.836
127.826
127.753
127.685
127.571
127.503
59.617
53.721
53.360
53.000
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13.172
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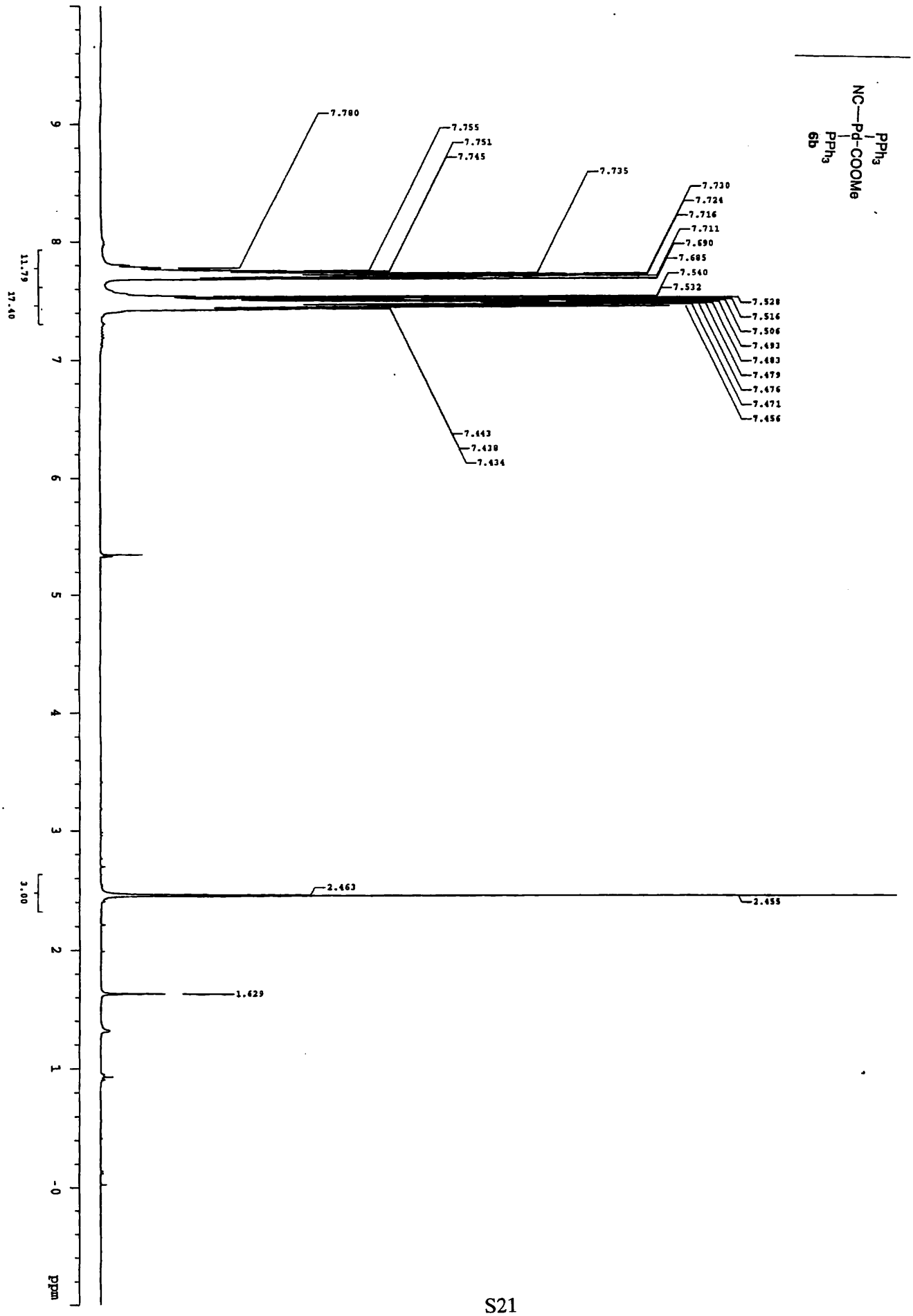
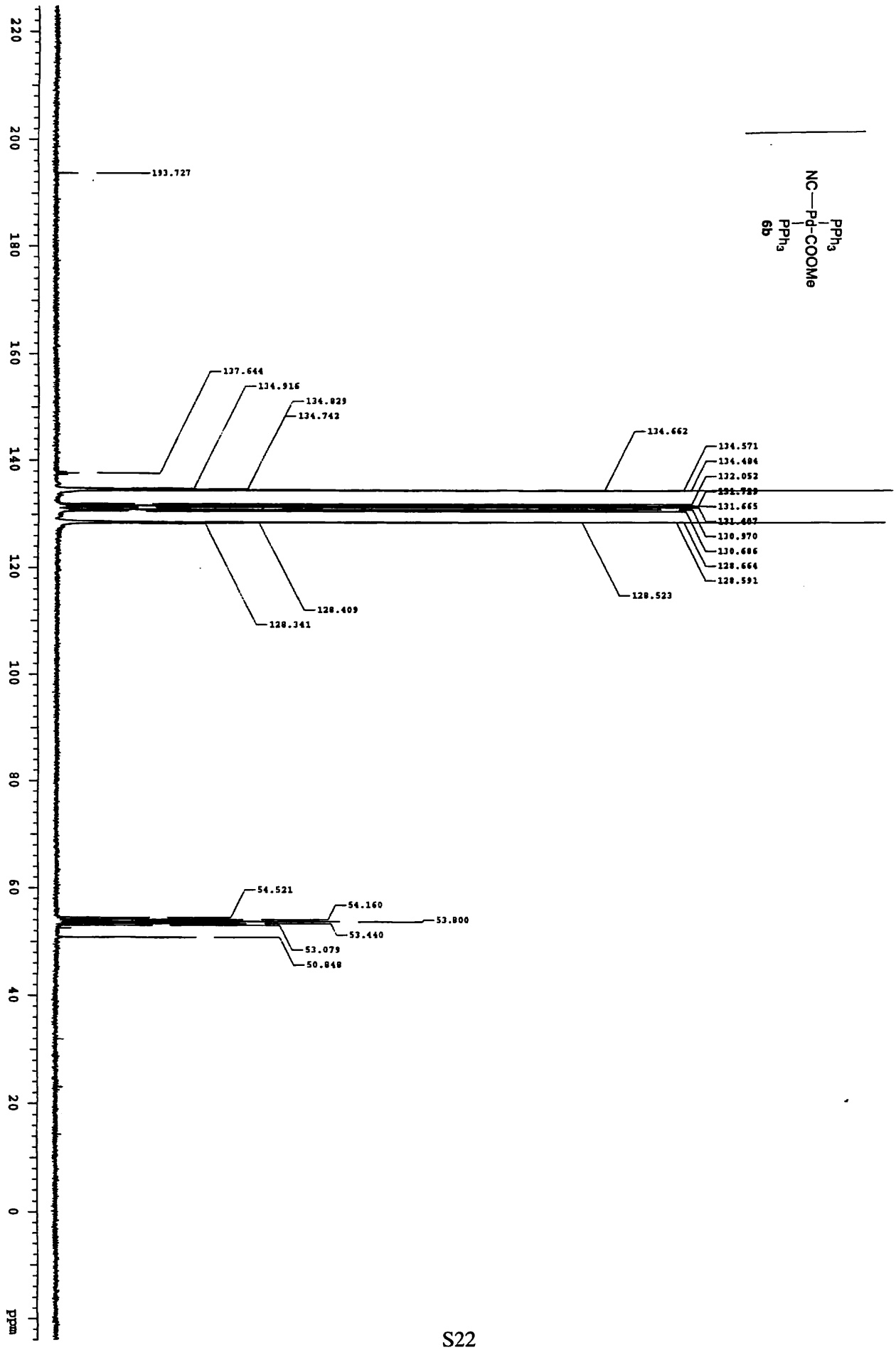
200
180
160
140
120
100
80
60
40
20
0
ppm


PPh3
NC—Pd-COOMe
PPh3
6b
7.780
7.755
7.751
7.745
7.735
7.730
7.724
7.716
7.711
7.690
7.685
7.540
7.532
7.528
7.516
7.506
7.493
7.483
7.479
7.476
7.471
7.456
7.443
7.438
7.434
2.463
2.455
1.629
11.79
17.40
3.00
ppm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134.742
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132.052
131.665
130.970
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128.664
128.591
128.523
128.409
128.341
54.521
54.160
53.800
53.440
53.079
50.848
NC—Pd—COOMe
PPh3
PPh3
6b
220
200
180
160
140
120
100
80
60
40
20
0
ppm

X-ray Structure Report for **7b**

Experimental

Data Collection

A colorless prismatic crystal of $C_{39}H_{33}NO_2P_2Pd$ having approximate dimensions of 0.30 x 0.28 x 0.25 mm was mounted on a glass fiber. All measurements were made on a Rigaku Saturn CCD area detector with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 12.082(2) Å
b = 23.223(3) Å β = 113.374(2)°
c = 12.587(2) Å
V = 3241.8(7) Å^3

For Z = 4 and F.W. = 716.04, the calculated density is 1.47 g/cm^3. The systematic absences of:

h0l: h+l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of -160 ± 1°C to a maximum 2θ value of 55.0°. A total of 720 oscillation images were collected. A sweep of data was done using ω scans from -110.0 to 70.0° in 0.5° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 30.0 [sec./°]. The detector swing angle was -20.28°. A second sweep was performed using ω scans from -110.0 to 70.0° in 0.5° step, at χ=45.0° and ϕ = 90.0°. The exposure rate was 30.0 [sec./°]. The detector swing angle was -20.28°. The crystal-to-detector distance was 45.00 mm. Readout was performed in the 0.547 mm pixel mode.

Data Reduction

Of the 24355 reflections that were collected, 23549 were unique (R_{int} = 0.020). Data were collected and processed using CrystalClear (Rigaku). Net intensities and sigmas were derived as follows:

$$F^2 = [\Sigma(P_i - mB_{ave})] \cdot Lp^{-1}$$

where P_i is the value in counts of the i^{th} pixel
m is the number of pixels in the integration area
B_{ave} is the background average
Lp is the Lorentz and polarization factor

$$B_{ave} = \Sigma(B_j)/n$$

where n is the number of pixels in the background area

B_j is the value of the j^{th} pixel in counts

$$\sigma^2(F^2_{hkl}) = [(\Sigma P_i) + m((\Sigma(B_{ave} - B_j)^2)/(n-1))] \cdot Lp \cdot errmul + (erradd \cdot F^2)^2$$

where erradd = 0.00

errmul = 1.00

The linear absorption coefficient, μ, for Mo-Kα radiation is 7.1 cm^{-1}. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement on F was based on 19419 observed reflections ($I > 3.00\sigma(I)$) and 439 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.039$$

$$R_W = [\, \Sigma \, w \, (|Fo| - |Fc|)^2 \, / \, \Sigma \, w \, Fo^2]^{1/2} = 0.052$$

The standard deviation of an observation of unit weight was 0.95. A Sheldrick weighting scheme was used. Plots of $\Sigma \, w \, (|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 5.10 and -6.16 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber. Anomalous dispersion effects were included in Fcalc; the values for Δf' and Δf'' were those of Creagh and McAuley. The values for the mass attenuation coefficients are those of Creagh and Hubbell. All calculations were performed using the CrystalStructure crystallographic software package.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{39}H_{33}NO_2P_2Pd$
Formula Weight	716.04
Crystal Color, Habit	colorless, prismatic
Crystal Dimensions	0.30 x 0.28 x 0.25 mm
Crystal System	monoclinic
Lattice Type	Primitive
Detector Position	45.00 mm
Pixel Size	0.137 mm
Lattice Parameters	a = 12.082(2) Å b = 23.223(3) Å c = 12.587(2) Å β = 113.374(2) ° V = 3241.8(7) Å^3
Space Group	$P2_1/n$ (#14)
Z value	4
D_{calc}	1.467 g/cm^3
F_{000}	1464.00
μ(MoKα)	7.08 cm^{-1}

B. Intensity Measurements

Detector	Rigaku Saturn
Goniometer	Rigaku AFC10
Radiation	MoKα (λ = 0.71070 Å) graphite monochromated
Detector Aperture	70 mm x 70 mm
Data Images	720 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	-110.0 - 70.0°
Exposure Rate	30.0 sec./°
Detector Swing Angle	-20.28°
ω oscillation Range (χ=45.0, ϕ=90.0)	-110.0 - 70.0°
Exposure Rate	30.0 sec./°
Detector Swing Angle	-20.28°
Detector Position	45.00 mm
Pixel Size	0.137 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 24355 Unique: 23549 (R_{int} = 0.020)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR97)
Refinement	Full-matrix least-squares on F
Function Minimized	Σ w (|Fo| - |Fc|)^2
Least Squares Weights	1/[0.0010Fo^2+3.0000σ(Fo^2)+0.2000]

Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	19419
No. Variables	439
Reflection/Parameter Ratio	44.23
Residuals: R (I>3.00σ(I))	0.039
Residuals: Rw (I>3.00σ(I))	0.052
Goodness of Fit Indicator	0.949
Max Shift/Error in Final Cycle	0.004
Maximum peak in Final Diff. Map	5.10 $e^-/Å^3$
Minimum peak in Final Diff. Map	-6.16 $e^-/Å^3$

Table S1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Pd(1)	0.976220(10)	0.140880(10)	0.807720(10)	1.224(3)
P(1)	0.78601(4)	0.16216(2)	0.80269(4)	1.261(9)
P(2)	1.16970(4)	0.12388(2)	0.81814(4)	1.251(9)
O(1)	1.0400(1)	0.25639(6)	0.8968(2)	4.07(4)
O(2)	0.9714(1)	0.24950(7)	0.7070(2)	4.56(4)
N(1)	0.9288(2)	0.00665(8)	0.7945(2)	2.91(4)
C(1)	0.9443(1)	0.05151(8)	0.8006(1)	1.29(4)
C(2)	1.0032(2)	0.22660(10)	0.8101(2)	3.74(6)
C(3)	0.9819(3)	0.31581(12)	0.7299(4)	9.74(13)
C(4)	0.7349(2)	0.23687(7)	0.7772(2)	1.54(4)
C(5)	0.7734(2)	0.27708(8)	0.8676(2)	2.17(4)
C(6)	0.7351(2)	0.33364(9)	0.8481(2)	2.64(5)
C(7)	0.6607(2)	0.35192(8)	0.7387(2)	2.94(5)
C(8)	0.6251(2)	0.31304(9)	0.6480(2)	2.58(5)
C(9)	0.6610(2)	0.25572(8)	0.6667(2)	1.91(4)
C(10)	0.7718(2)	0.14427(7)	0.9377(2)	1.44(4)
C(11)	0.6864(2)	0.17154(8)	0.9691(2)	1.88(4)
C(12)	0.6788(2)	0.15916(9)	1.0738(2)	2.23(4)
C(13)	0.7557(2)	0.11912(9)	1.1474(2)	2.14(4)
C(14)	0.8394(2)	0.09073(8)	1.1170(2)	1.89(4)
C(15)	0.8478(2)	0.10368(8)	1.0124(2)	1.65(4)
C(16)	0.6658(2)	0.12353(8)	0.6888(2)	1.73(4)
C(17)	0.5569(2)	0.11165(9)	0.6954(2)	2.31(4)
C(18)	0.4642(2)	0.08569(9)	0.6051(2)	2.81(5)
C(19)	0.4794(2)	0.07210(11)	0.5053(2)	4.99(6)
C(20)	0.5873(2)	0.0847(2)	0.4967(3)	7.56(9)
C(21)	0.6805(2)	0.10930(13)	0.5892(2)	4.80(7)
C(22)	1.2655(2)	0.18501(7)	0.8189(2)	1.50(4)
C(23)	1.2984(2)	0.22281(8)	0.9122(2)	2.16(4)
C(24)	1.3763(2)	0.26785(9)	0.9218(2)	3.07(5)
C(25)	1.4215(2)	0.27641(9)	0.8382(2)	3.15(5)
C(26)	1.3886(2)	0.24004(10)	0.7439(2)	3.12(5)
C(27)	1.3102(2)	0.19402(9)	0.7336(2)	2.25(4)
C(28)	1.1620(2)	0.07996(7)	0.6952(1)	1.36(3)
C(29)	1.0970(2)	0.10187(9)	0.5831(2)	1.96(4)

C(30)	1.0889(2)	0.07019(9)	0.4872(2)	2.30(4)
C(31)	1.1427(2)	0.01677(9)	0.5002(2)	2.34(4)
C(32)	1.2047(2)	-0.00535(8)	0.6108(2)	2.18(4)
C(33)	1.2149(2)	0.02653(8)	0.7077(2)	1.74(4)
C(34)	1.2663(2)	0.08492(7)	0.9477(1)	1.44(3)
C(35)	1.3898(2)	0.08078(8)	0.9742(2)	1.85(4)
C(36)	1.4654(2)	0.05238(8)	1.0728(2)	2.19(4)
C(37)	1.4195(2)	0.02785(8)	1.1470(2)	2.25(4)
C(38)	1.2967(2)	0.03140(8)	1.1208(2)	2.20(4)
C(39)	1.2209(2)	0.05983(8)	1.0222(2)	1.77(4)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table S2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Pd(1)	0.01394(7)	0.01467(7)	0.01674(7)	0.00046(5)	0.00486(5)	0.00027(5)
P(1)	0.0153(2)	0.0151(2)	0.0169(2)	0.0018(2)	0.0056(2)	0.0013(2)
P(2)	0.0143(2)	0.0160(2)	0.0175(2)	-0.0009(2)	0.0065(2)	-0.0005(2)
O(1)	0.0346(9)	0.0257(9)	0.0830(13)	-0.0038(7)	0.0114(9)	-0.0227(9)
O(2)	0.0394(9)	0.0438(10)	0.0654(12)	-0.0135(8)	-0.0054(8)	0.0345(9)
N(1)	0.0275(10)	0.0449(13)	0.0402(11)	0.0024(9)	0.0156(8)	-0.0042(9)
C(1)	0.0087(8)	0.0259(10)	0.0194(9)	0.0023(7)	0.0109(7)	-0.0003(7)
C(2)	0.0120(10)	0.0296(13)	0.090(2)	0.0065(9)	0.0094(12)	0.0192(13)
C(3)	0.135(3)	0.028(2)	0.286(5)	0.017(2)	0.168(4)	0.033(2)
C(4)	0.0202(9)	0.0153(9)	0.0267(10)	0.0033(7)	0.0132(8)	0.0026(7)
C(5)	0.0297(11)	0.0204(10)	0.0342(11)	-0.0018(8)	0.0147(9)	-0.0022(8)
C(6)	0.0386(12)	0.0188(10)	0.053(1)	-0.0027(9)	0.0282(11)	-0.0063(10)
C(7)	0.0372(13)	0.0173(11)	0.068(2)	0.0078(9)	0.0316(13)	0.0108(10)
C(8)	0.0295(11)	0.0300(12)	0.0427(13)	0.0101(9)	0.0186(10)	0.0181(10)
C(9)	0.0233(10)	0.0229(10)	0.0306(10)	0.0060(8)	0.0150(8)	0.0087(8)
C(10)	0.0173(9)	0.0186(9)	0.0183(9)	-0.0057(7)	0.0064(7)	0.0016(7)
C(11)	0.0195(9)	0.0244(10)	0.0265(10)	0.0024(8)	0.0080(8)	0.0040(8)
C(12)	0.0285(11)	0.0299(11)	0.0333(11)	-0.0008(9)	0.0198(9)	0.0009(9)
C(13)	0.0313(11)	0.0302(11)	0.0222(10)	-0.0080(9)	0.0133(9)	0.0029(8)
C(14)	0.0252(10)	0.0215(10)	0.0229(9)	-0.0049(8)	0.0070(8)	0.0057(7)
C(15)	0.0174(9)	0.0208(9)	0.0230(9)	-0.0028(7)	0.0062(7)	0.0016(7)
C(16)	0.0190(9)	0.0179(9)	0.0228(9)	0.0051(7)	0.0020(8)	0.0002(7)
C(17)	0.0286(10)	0.0353(12)	0.0225(10)	-0.0064(9)	0.0088(8)	0.0003(8)
C(18)	0.0261(11)	0.0307(12)	0.0387(12)	-0.0039(9)	0.0010(9)	0.0020(9)
C(19)	0.0258(12)	0.078(2)	0.059(2)	0.0162(12)	-0.0123(11)	-0.047(1)
C(20)	0.030(1)	0.183(4)	0.063(2)	0.011(2)	0.0060(13)	-0.087(2)
C(21)	0.0223(12)	0.112(2)	0.041(1)	0.008(1)	0.0052(11)	-0.042(2)
C(22)	0.0152(9)	0.0190(9)	0.0228(9)	0.0018(7)	0.0074(7)	0.0045(7)
C(23)	0.0242(10)	0.0251(11)	0.0305(11)	-0.0048(8)	0.0087(8)	-0.0020(8)
C(24)	0.0301(11)	0.0244(11)	0.049(1)	-0.0051(9)	0.0023(10)	-0.0046(10)
C(25)	0.0211(11)	0.0235(11)	0.067(2)	-0.0061(9)	0.0090(11)	0.0133(11)
C(26)	0.0279(11)	0.0392(13)	0.056(2)	-0.0006(10)	0.0209(11)	0.0173(11)
C(27)	0.0258(10)	0.0301(11)	0.0316(11)	-0.0008(9)	0.0135(9)	0.0044(9)

C(28)	0.0152(8)	0.0199(9)	0.0172(9)	-0.0031(7)	0.0072(7)	-0.0007(7)
C(29)	0.0222(10)	0.0274(11)	0.0220(10)	0.0019(8)	0.0057(8)	0.0038(8)
C(30)	0.0291(11)	0.0393(12)	0.0178(9)	-0.0082(9)	0.0081(8)	-0.0005(8)
C(31)	0.0310(11)	0.0352(12)	0.0301(11)	-0.0124(9)	0.0200(9)	-0.0113(9)
C(32)	0.0327(11)	0.0232(10)	0.0341(11)	-0.0024(9)	0.0209(9)	-0.0064(8)
C(33)	0.0219(9)	0.0231(10)	0.0228(9)	-0.0014(8)	0.0105(8)	0.0005(7)
C(34)	0.0180(9)	0.0162(9)	0.0182(9)	0.0014(7)	0.0047(7)	-0.0018(7)
C(35)	0.0208(9)	0.0229(10)	0.0270(10)	-0.0014(8)	0.0098(8)	0.0028(8)
C(36)	0.0188(9)	0.0251(11)	0.0346(11)	-0.0008(8)	0.0054(8)	0.0025(9)
C(37)	0.0318(11)	0.0236(10)	0.0219(10)	0.0021(8)	0.0019(9)	0.0058(8)
C(38)	0.0323(11)	0.0273(11)	0.0245(10)	-0.0045(9)	0.0118(9)	0.0038(8)
C(39)	0.0204(9)	0.0245(10)	0.0233(9)	-0.0035(8)	0.0096(8)	-0.0021(8)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

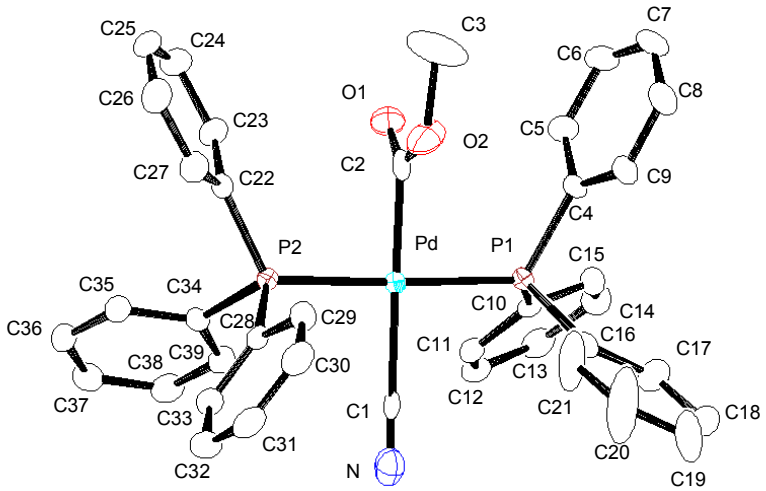
Table S3. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Pd(1)	P(1)	2.3268(5)	Pd(1)	P(2)	2.3225(5)
Pd(1)	C(1)	2.106(2)	Pd(1)	C(2)	2.015(2)
P(1)	C(4)	1.827(2)	P(1)	C(10)	1.823(2)
P(1)	C(16)	1.821(2)	P(2)	C(22)	1.829(2)
P(2)	C(28)	1.825(2)	P(2)	C(34)	1.825(2)
O(1)	C(2)	1.217(3)	O(2)	C(2)	1.311(3)
O(2)	C(3)	1.563(3)	N(1)	C(1)	1.056(3)
C(4)	C(5)	1.401(3)	C(4)	C(9)	1.393(2)
C(5)	C(6)	1.382(3)	C(6)	C(7)	1.380(3)
C(7)	C(8)	1.383(3)	C(8)	C(9)	1.391(3)
C(10)	C(11)	1.395(3)	C(10)	C(15)	1.389(2)
C(11)	C(12)	1.387(3)	C(12)	C(13)	1.380(3)
C(13)	C(14)	1.382(3)	C(14)	C(15)	1.393(3)
C(16)	C(17)	1.380(3)	C(16)	C(21)	1.374(4)
C(17)	C(18)	1.378(2)	C(18)	C(19)	1.376(4)
C(19)	C(20)	1.381(4)	C(20)	C(21)	1.383(3)
C(22)	C(23)	1.392(3)	C(22)	C(27)	1.396(3)
C(23)	C(24)	1.379(3)	C(24)	C(25)	1.380(4)
C(25)	C(26)	1.381(3)	C(26)	C(27)	1.400(3)
C(28)	C(29)	1.409(2)	C(28)	C(33)	1.376(2)
C(29)	C(30)	1.383(3)	C(30)	C(31)	1.380(3)
C(31)	C(32)	1.391(3)	C(32)	C(33)	1.390(3)
C(34)	C(35)	1.396(3)	C(34)	C(39)	1.389(3)
C(35)	C(36)	1.382(2)	C(36)	C(37)	1.384(3)
C(37)	C(38)	1.389(3)	C(38)	C(39)	1.383(2)

Table S4. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
P(2)	Pd(1)	P(1)	177.09(2)	C(1)	Pd(1)	P(1)	92.62(5)
C(2)	Pd(1)	P(1)	86.72(7)	Pd(1)	P(1)	C(4)	117.88(7)

Pd(1)	P(1)	C(10)	112.55(6)	Pd(1)	P(1)	C(16)	112.77(7)
C(1)	Pd(1)	P(2)	89.90(5)	C(2)	Pd(1)	P(2)	90.80(7)
Pd(1)	P(2)	C(22)	119.25(6)	Pd(1)	P(2)	C(28)	109.60(6)
Pd(1)	P(2)	C(34)	114.05(7)	Pd(1)	C(1)	N(1)	178.0(2)
C(2)	Pd(1)	C(1)	178.42(7)	Pd(1)	C(2)	O(1)	125.0(2)
Pd(1)	C(2)	O(2)	113.8(2)	C(10)	P(1)	C(4)	103.29(9)
C(16)	P(1)	C(4)	102.80(8)	P(1)	C(4)	C(5)	120.7(1)
P(1)	C(4)	C(9)	120.8(1)	P(1)	C(10)	C(15)	120.3(2)
C(16)	P(1)	C(10)	106.35(9)	P(1)	C(10)	C(11)	120.9(1)
P(1)	C(16)	C(17)	122.2(2)	P(1)	C(16)	C(21)	119.0(2)
C(28)	P(2)	C(22)	104.95(9)	C(34)	P(2)	C(22)	101.60(7)
P(2)	C(22)	C(23)	117.6(2)	P(2)	C(22)	C(27)	123.3(1)
C(34)	P(2)	C(28)	106.26(8)	P(2)	C(28)	C(29)	117.9(1)
P(2)	C(28)	C(33)	122.8(1)	P(2)	C(34)	C(35)	119.5(2)
P(2)	C(34)	C(39)	121.8(1)	O(1)	C(2)	O(2)	121.1(2)
C(3)	O(2)	C(2)	104.3(2)	C(9)	C(4)	C(5)	118.4(2)
C(4)	C(5)	C(6)	120.7(2)	C(4)	C(9)	C(8)	120.2(2)
C(5)	C(6)	C(7)	120.6(2)	C(6)	C(7)	C(8)	119.3(2)
C(7)	C(8)	C(9)	120.7(2)	C(15)	C(10)	C(11)	118.8(2)
C(10)	C(11)	C(12)	120.6(2)	C(10)	C(15)	C(14)	120.6(2)
C(11)	C(12)	C(13)	119.8(2)	C(12)	C(13)	C(14)	120.5(2)
C(13)	C(14)	C(15)	119.7(2)	C(21)	C(16)	C(17)	118.6(2)
C(16)	C(17)	C(18)	121.3(2)	C(16)	C(21)	C(20)	120.7(3)
C(17)	C(18)	C(19)	119.7(2)	C(18)	C(19)	C(20)	119.6(2)
C(19)	C(20)	C(21)	120.1(3)	C(27)	C(22)	C(23)	119.1(2)
C(22)	C(23)	C(24)	120.6(2)	C(22)	C(27)	C(26)	119.9(2)
C(23)	C(24)	C(25)	120.3(2)	C(24)	C(25)	C(26)	120.1(2)
C(25)	C(26)	C(27)	120.0(3)	C(33)	C(28)	C(29)	119.3(2)
C(28)	C(29)	C(30)	120.0(2)	C(28)	C(33)	C(32)	120.3(2)
C(29)	C(30)	C(31)	120.5(2)	C(30)	C(31)	C(32)	119.4(2)
C(31)	C(32)	C(33)	120.4(2)	C(39)	C(34)	C(35)	118.7(1)
C(34)	C(35)	C(36)	120.7(2)	C(34)	C(39)	C(38)	120.6(2)
C(35)	C(36)	C(37)	120.2(2)	C(36)	C(37)	C(38)	119.4(2)
C(37)	C(38)	C(39)	120.4(2)				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P2
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C11
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C17
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C1
C31
C20
C32
N
C19