Supplementary Material (ESI) for Chemical Communications

Mono-palladium (II) complexes of diamidopyridine-dipyrromethane hybrid macrocycles.

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Supporting Information.

General methods. All solvents were of reagent grade quality and purchased commercially. Starting materials were purchased from Aldrich Chemical Co. or Acros Organics and used without further purification. NMR spectra used in the characterization of products were recorded on Varian INOVA 500, Varian Mercury 400 or Varian UNITY+ 300 instruments. The NMR spectra were referenced to solvent and the spectroscopic solvents were purchased from Cambridge Isotope Laboratories. All high-resolution (HR) chemical ionization (CI) mass spectra were recorded on a VG ZAB-2E instrument. Elemental analyses were performed by Atlantic Microlabs, Inc., Atlanta, GA, and are reported as percentages. TLC analyses were carried out using Baker-flex Silica gel IB-F sheets. Column chromatography was performed using Whatman silica gel 60Å (230 – 400 mesh) as the solid support.

Complex 3. Ligand **1** (500 mg; 0.7 mmol) and $PdCl_2(CH_3CN_2)$ (181.2 mg; 0.7 mmol) were mixed in 25 ml of dry acetonitrile and stirred for 30 min. At this point, 10 equivalents of triethylamine were added and the reaction was stirred for 12 h at r.t. The resulting yellow precipitate was filtered off and washed with acetonitrile and dried in vacuo. This provided a crude product that was then recrystallized by slow diffusion of pentane into a solution of the complex in dichloromethane. This yielded complex **3** (494 mg; 86%) in the form of yellow crystals. M.p. >350 ºC decomp. MS (CI^+, MH^+): *m/e* calcd for $[C_{45}H_{43}N_7O_2Pd+H]$: 820.3, found 820.4. Anal. Calcd for $C_{45}H_{43}N_7O_2Pd$: C, 65.89; H, 5.28; N, 11.95. Found: C, 65.90; H, 5.30; N, 11.97. ^1H NMR (CD_2Cl_2, -50 ºC): δ (ppm): multiplets at 0.36 (3H), 0.85 (4H), 0.91 (1H), 1.25

(1H), 1.35 (1H), 1.41 (3H), 1.75 (1H), 2.03 (3H), 2.27 (3H); 5.31 (1H, s), 6.36 (0,28H, m), 6.46 (1H, m), 6.69 (1H, m), 6.60 (1H, m), 6.91 (1H, m), 7.04 (6H, m), 7.31 (6H, m), 7.99 (2H, m), 8.06 (1H, m), 8.23 (1H, m), 8.37 (1H, bs), 8.62 (1H, bs), 10.89 (1H, bs). ^{13}C NMR (CD_2Cl_2, -50 ºC): δ (ppm) 9.6, 14.0, 20.7, 22.4, 24.1, 24.4, 25.2, 26.8, 27.5, 46.6, 111.9, 117.1, 117.4, 123.57, 124.9. 125.0, 1251, 125.8, 126.2, 126.3, 127.0, 127.4, 127,7, 127.9, 128.8, 129.5, 130.7, 121.67, 132.4, 133.9, 134.2, 135.0, 135.4, 135.6, 136.5, 139.1, 143.6, 144.3, 145.4, 146.0, 147.1, 147.47, 158.3, 159.8, 161.9, 166.8. UV-vis (THF): λ_{max} [nm] (ε in M^{-1} cm^{-1}) 327 (23300), 358 (20500), 433 (17500), 455 (16200).

Complex 4. Ligand **2** (200 mg; 0.28 mmol) and $PdCl_2(CH_3CN_2)$ (72.6 mg; 0.28 mmol) were mixed in 10 ml of dry acetonitrile and stirred for 12 h under an argon atmosphere. The dark green crystals that formed were collected by filtration, washed with 5 ml of cold acetonitrile, and dried in vacuo. This yielded 144 mg (60%) of complex **4**. M.p. >200 ºC decomp. MS (CI^+, MH^+): *m/e* calcd for [$C_{45}H_{42}N_7O_2Pd^+$]: 818.2, found 818.3. Anal. Calcd for $C_{45}H_{42}ClN_7O_2Pd$: C, 63.23; H, 4.95; N, 11.47. Found: C, 63.25; H, 4.96; N, 11.50. ^{1}H NMR ($CDCl_3$, δ (ppm), *J* (Hz)): 0.86 (6H, m), 1.09 (4H, m), 1.38 (4H, m), 1.97 (6H, s), 2.16 and 2.30 (3H, m), 6.90 (1H, m), 7.02 (1H, m), 7.14 (1H, m), 7.32 (9H, m), 7.61 (1H, m), 8.14 (1H, t, *J*=7.2) 8.34 (1H, d, *J*=7.2), 8.40 (1H, d, *J*=7.2), 8.80 (1H, s), 9.68 (1H, bs), 10.48 (1H, bs). ^{13}C NMR ($CDCl_3$, δ (ppm)): 25.7, 35.3, 37.7, 39.2, 45.2, 46.8, 48.6, 50.4, 51.1, 89.7, 140.6, 144.2, 148.6, 149.1, 149.2, 149.9, 150.9, 151.1, 152.1, 152.2, 152.5, 154.1, 154.2, 155.4, 56.2, 157.8, 158.4, 159.6, 162.3, 163.3, 163.4, 164.0, 170.0, 170.4, 170.5, 171.2, 173.2, 173.3, 173.1, 183.2, 184.5, 186.3, 187.4, 187.5, 194,7. UV-vis (THF): λ_{max} [nm] (ε in M^{-1} cm^{-1}) 319 (17600), 445 (6800), 615 (6230), 655 (6220).

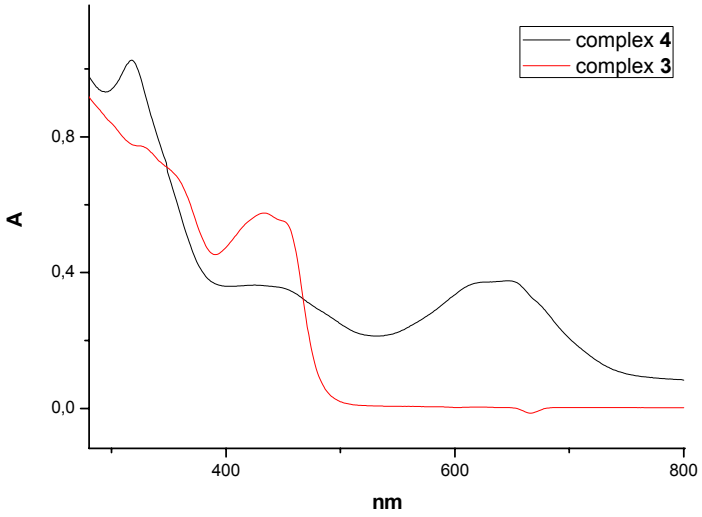

Fig. 1. UV-vis spectra for palladium(II) complexes **3** and **4** recorded in tetrahydrofuran..

X-ray experimental for complex 3.

X-ray experimental for complex **3**, $(C_{45}H_{43}N_7O_2)Pd - 1.6CH_2Cl_2$ (CCDC #615936): Crystals grew as thin, yellow lathes by slow evaporation from methylene chloride. The data crystal was a long lathe that had approximate dimensions; 0.35 x 0.10 x 0.03 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073 Å). A total of 403 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 141 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Data reduction was performed using DENZO-SMN.[1] The structure was solved by direct methods using SIR97,[2] and refined by full-matrix least-squares on F2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[3] The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for the methyl hydrogen atoms).

The complex crystallized along with some methylene chloride solvate molecules. The solvent appears in two locations in the asymmetric unit. In one location, a molecule of methylene chloride is disordered about two primary orientations given by atoms, C1a, Cl1a and Cl2a and C1aa, Cl3a and Cl4a. The disorder was modeled in the usual manner by setting the site occupancy factor for C1a, Cl1a and Cl2a to the variable x. The site occupancy factor to the other

part of the disordered solvent given by C1aa, Cl3a and Cl4a, was assigned a site occupancy factor of (1-x). The six atoms were allowed to refine with a common isotropic displacement parameter while restraining the geometry of the molecule to be approximately equal. In this way, the site occupancy of the major component, C1a, Cl1a and Cl2a, refined to 0.57(2).

The second region of disorder involved two atoms in one of the propyl side chains, C36 and C37. The two orientations were refined as described above and resulted in a site occupancy factor for the minor component, composed of atoms C36a and C37a, to refine to 38(2)%. Complicating the disorder in this region was the presence of some solvent. Due to the proximity of the solvent to C37a, it was impossible for both C37a and the solvent, composed of atoms C1b, Cl1b and Cl2b, to be present simultaneously. It was concluded that the maximum site occupancy for this solvent molecule was 62%. The molecule was subsequently refined with this as its site occupancy factor. The geometry of the methylene chloride molecules were restrained to be approximately equal throughout the refinement process.

The function, $\Sigma w(|Fo|^2 - |Fc|^2)2$, was minimized, where $w = 1/[(\sigma(Fo))^2 + (0.0419*P)^2 + (3.3176*P)]$ and $P = (|Fo|^2 + 2|Fc|^2)/3$. Rw(F2) refined to 0.157, with R(F) equal to 0.0722 and a goodness of fit, S, = 1.03. Definitions used for calculating R(F), Rw(F2) and the goodness of fit, S, are given below.[4] The data were corrected for secondary extinction effects. The correction takes the form: $Fcorr = kFc/[1 + (2.0(4)x10^{-6})* Fc2\ \gamma^3/(\sin 2\theta)]0.25$ where k is the overall scale factor. Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[5]

Table 1. Crystal data and structure refinement for **1**.

Empirical formula	C46.57 H46.24 Cl3.24 N7 O2 Pd	
Formula weight	957.24	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.3506(4) Å	α= 60.823(2)°.
	b = 15.7321(5) Å	β= 87.392(2)°.
	c = 15.8708(6) Å	β = 86.802(2)°.
Volume	2252.43(14) Å^3	
Z	2	
Density (calculated)	1.411 Mg/m^3	

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Absorption coefficient	0.650 mm^{-1}
F(000)	983
Crystal size	0.35 x 0.10 x 0.03 mm
Theta range for data collection	2.94 to 25.00°.
Index ranges	-12<=h<=11, -18<=k<=18, -18<=l<=16
Reflections collected	13785
Independent reflections	7817 [R(int) = 0.1000]
Completeness to theta = 25.00°	98.6 %
Absorption correction	None
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	7817 / 99 / 601
Goodness-of-fit on F^2	1.018
Final R indices [I>2sigma(I)]	R1 = 0.0722, wR2 = 0.1279
R indices (all data)	R1 = 0.1798, wR2 = 0.1569
Extinction coefficient	$2.0(4)x10^{-6}$
Largest diff. peak and hole	0.820 and -0.731 $e.Å^{-3}$

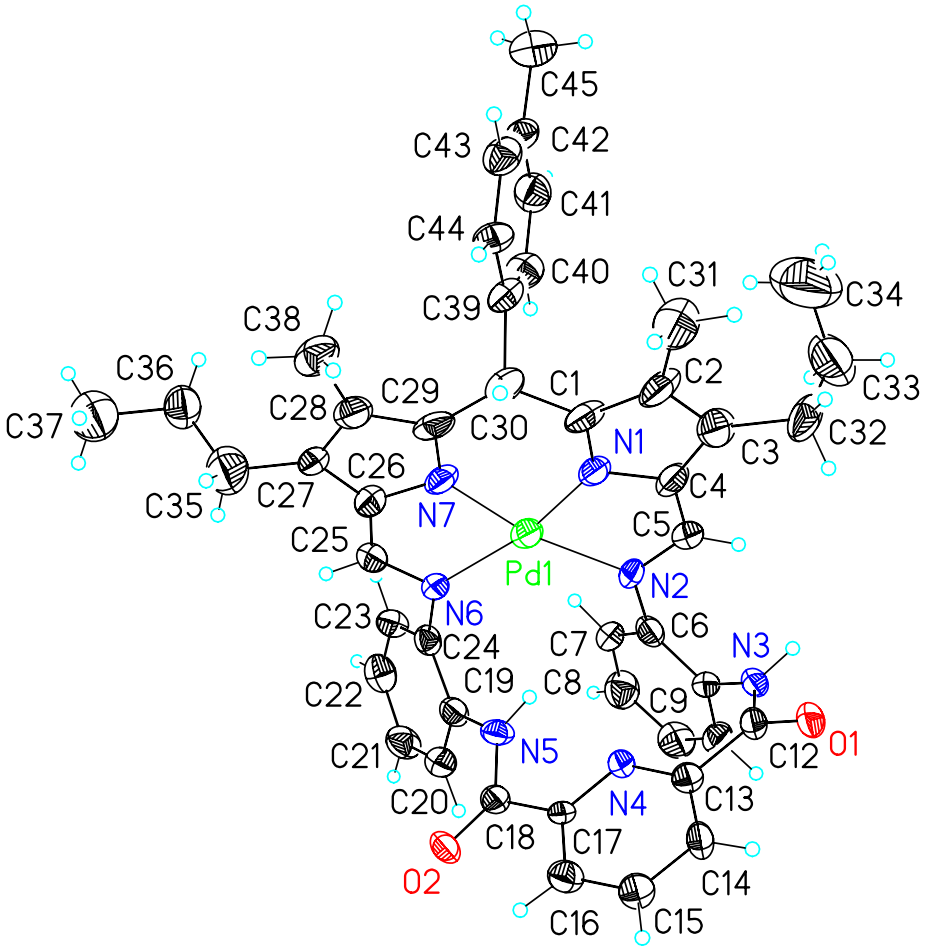

Fig. 2. View of the macrocycle complex in **3** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. The atoms of the minor component of the disorder in the propyl group, C35, C36 and C37 have been omitted for clarity.

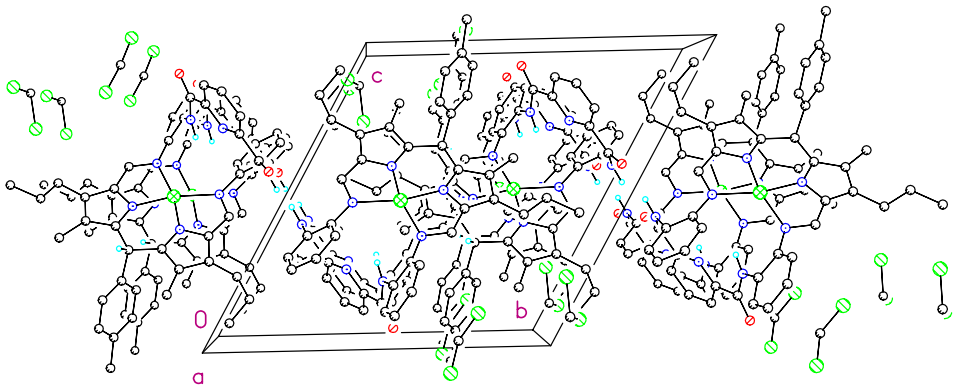

Fig. 3. Unit cell packing diagram for **complex 3**. The view is approximately down the **a** axis.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **3**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd1	2479(1)	3379(1)	4867(1)	35(1)
O1	-1006(5)	828(3)	4125(3)	39(1)
O2	1950(4)	5097(3)	788(3)	37(1)
N1	1356(6)	2625(4)	5963(4)	37(2)
N2	2547(6)	2055(4)	4832(4)	33(2)
N3	1145(5)	932(4)	4151(4)	29(1)
N4	790(5)	2895(4)	2660(4)	27(1)
N5	2640(5)	4094(4)	2313(4)	31(2)
N6	3585(5)	4468(4)	3745(4)	31(2)
N7	2168(6)	4434(4)	5166(4)	36(2)
C1	627(7)	2814(6)	6576(6)	43(2)
C2	-13(8)	1957(7)	7241(6)	54(2)
C3	354(8)	1220(6)	7022(6)	54(2)
C4	1214(8)	1662(6)	6202(6)	46(2)
C5	1885(7)	1393(6)	5579(5)	40(2)
C6	3091(7)	1785(5)	4166(5)	34(2)
C7	4352(7)	2063(5)	3817(5)	36(2)
C8	4922(8)	1864(6)	3133(6)	49(2)

C9	4263(8)	1348(6)	2788(6)	48(2)
C10	3025(7)	1066(5)	3133(5)	39(2)
C11	2422(6)	1276(5)	3805(5)	25(2)
C12	-23(8)	1303(5)	3765(5)	32(2)
C13	-151(7)	2276(5)	2869(5)	32(2)
C14	-1238(7)	2468(5)	2322(5)	33(2)
C15	-1365(7)	3368(5)	1511(5)	33(2)
C16	-412(7)	4038(5)	1271(5)	38(2)
C17	655(6)	3770(5)	1857(5)	24(2)
C18	1797(7)	4392(5)	1596(5)	30(2)
C19	3930(7)	4403(5)	2228(5)	31(2)
C20	4708(7)	4550(5)	1440(5)	38(2)
C21	5958(7)	4833(5)	1365(6)	44(2)
C22	6461(7)	4933(5)	2105(6)	42(2)
C23	5685(7)	4788(5)	2892(5)	37(2)
C24	4403(7)	4537(5)	2963(5)	30(2)
C25	3501(7)	5262(6)	3830(5)	41(2)
C26	2736(8)	5298(6)	4564(6)	41(2)
C27	2308(8)	6001(5)	4830(5)	45(2)
C28	1460(8)	5547(6)	5612(6)	44(2)
C29	1388(7)	4571(6)	5806(6)	40(2)
C30	603(7)	3771(5)	6588(5)	38(2)
C31	-961(9)	1876(7)	8033(7)	78(3)
C32	-6(10)	169(7)	7526(7)	79(4)
C33	922(14)	-515(8)	8317(9)	119(5)
C34	938(16)	-294(9)	9084(9)	163(7)
C35	2622(11)	7050(6)	4340(7)	81(3)
C36	3353(13)	7410(9)	4845(10)	53(4)
C37	3317(16)	8538(9)	4363(11)	85(5)
C36A	3983(18)	7203(15)	4010(20)	110(11)
C37A	4450(20)	8238(16)	3626(18)	84(8)
C38	709(8)	5999(6)	6138(6)	54(2)
C39	1022(7)	3520(5)	7613(5)	37(2)
C40	2180(7)	3009(5)	7972(6)	42(2)
C41	2506(8)	2701(5)	8917(6)	46(2)
C42	1698(8)	2910(6)	9517(6)	43(2)

C43	557(8)	3421(6)	9157(6)	43(2)
C44	226(7)	3735(5)	8208(5)	38(2)
C45	2074(8)	2549(6)	10566(6)	58(2)
Cl1A	4179(7)	7289(6)	-550(4)	61(2)
Cl2A	3900(7)	7183(5)	1288(4)	77(2)
C1A	3076(14)	7089(12)	401(8)	59(5)
Cl4A	3862(9)	7736(6)	1066(6)	75(3)
Cl3A	3786(13)	7640(9)	-676(7)	113(5)
C1AA	3770(30)	6945(11)	590(10)	87(10)
Cl1B	3963(7)	10069(4)	1012(5)	167(3)
Cl2B	5825(10)	8922(8)	2434(7)	267(5)
C1B	5571(13)	9660(16)	1181(10)	158(9)

Table 3. Bond lengths [Å] and angles [°] for **3**.

Pd1-N1	1.934(6)	C8-H8	0.96
Pd1-N7	1.943(6)	C9-C10	1.379(10)
Pd1-N2	2.108(6)	C9-H9	0.96
Pd1-N6	2.115(5)	C10-C11	1.377(9)
O1-C12	1.239(8)	C10-H10	0.96
O2-C18	1.231(7)	C12-C13	1.503(9)
N1-C1	1.340(9)	C13-C14	1.381(9)
N1-C4	1.385(9)	C14-C15	1.377(9)
N2-C5	1.327(8)	C14-H14	0.96
N2-C6	1.405(9)	C15-C16	1.386(9)
N3-C12	1.348(8)	C15-H15	0.96
N3-C11	1.437(8)	C16-C17	1.386(9)
N3-H3N	0.90	C16-H16	0.96
N4-C13	1.330(8)	C17-C18	1.488(9)
N4-C17	1.349(8)	C19-C20	1.383(9)
N5-C18	1.343(8)	C19-C24	1.396(9)
N5-C19	1.424(8)	C20-C21	1.373(9)
N5-H5N	0.90	C20-H20	0.96
N6-C25	1.319(9)	C21-C22	1.386(10)
N6-C24	1.432(8)	C21-H21	0.96
N7-C29	1.360(9)	C22-C23	1.381(10)
N7-C26	1.370(9)	C22-H22	0.96
C1-C2	1.421(10)	C23-C24	1.389(9)
C1-C30	1.514(10)	C23-H23	0.96
C2-C3	1.393(11)	C25-C26	1.401(10)
C2-C31	1.519(10)	C25-H25	0.96
C3-C4	1.432(10)	C26-C27	1.407(10)
C3-C32	1.505(11)	C27-C28	1.388(11)
C4-C5	1.396(10)	C27-C35	1.490(11)
C5-H5	0.96	C28-C29	1.417(10)
C6-C7	1.401(9)	C28-C38	1.506(10)
C6-C11	1.413(9)	C29-C30	1.512(10)
C7-C8	1.369(10)	C30-C39	1.552(10)
C7-H7	0.96	C30-H30	0.96
C8-C9	1.399(10)	C31-H31A	0.96

C31-H31B	0.96	C38-H38C	0.96
C31-H31C	0.96	C39-C44	1.376(9)
C32-C33	1.528(14)	C39-C40	1.385(10)
C32-H32A	0.96	C40-C41	1.386(10)
C32-H32B	0.96	C40-H40	0.96
C33-C34	1.421(14)	C41-C42	1.383(10)
C33-H33A	0.96	C41-H41	0.96
C33-H33B	0.96	C42-C43	1.369(10)
C34-H34A	0.96	C42-C45	1.537(10)
C34-H34B	0.96	C43-C44	1.391(10)
C34-H34C	0.96	C43-H43	0.96
C35-C36	1.443(13)	C44-H44	0.96
C35-C36A	1.470(15)	C45-H45A	0.96
C35-H35C	0.96	C45-H45B	0.96
C35-H35D	0.96	C45-H45C	0.96
C35-H35A	0.96	Cl1A-C1A	1.762(9)
C35-H35B	0.96	Cl2A-C1A	1.747(9)
C36-C37	1.550(15)	Cl2A-H1AC	1.7711
C36-H36A	0.96	Cl2A-H1AD	1.7136
C36-H36B	0.96	C1A-H1AA	0.96
C37-H37A	0.96	C1A-H1AB	0.96
C37-H37B	0.96	Cl4A-C1AA	1.752(10)
C37-H37C	0.96	Cl3A-C1AA	1.758(10)
C36A-C37A	1.532(17)	C1AA-H1AA	1.3170
C36A-H36C	0.96	C1AA-H1AC	0.96
C36A-H36D	0.96	C1AA-H1AD	0.96
C37A-H37D	0.96	Cl1B-C1B	1.735(10)
C37A-H37E	0.96	Cl2B-C1B	1.770(10)
C37A-H37F	0.96	C1B-H1BA	0.96
C38-H38A	0.96	C1B-H1BB	0.96
C38-H38B	0.96		

N1-Pd1-N7	86.6(3)	N2-Pd1-N6	113.3(2)
N1-Pd1-N2	80.1(2)	C1-N1-C4	108.6(6)
N7-Pd1-N2	166.7(2)	C1-N1-Pd1	134.8(5)
N1-Pd1-N6	166.6(2)	C4-N1-Pd1	116.6(5)
N7-Pd1-N6	80.0(2)	C5-N2-C6	117.4(6)

C5-N2-Pd1	110.1(5)	C7-C8-C9	120.2(8)
C6-N2-Pd1	132.4(5)	C7-C8-H8	119.5
C12-N3-C11	130.7(6)	C9-C8-H8	120.3
C12-N3-H3N	114.8	C10-C9-C8	118.7(7)
C11-N3-H3N	114.5	C10-C9-H9	121.2
C13-N4-C17	116.9(6)	C8-C9-H9	120.1
C18-N5-C19	126.5(6)	C11-C10-C9	122.1(7)
C18-N5-H5N	117.0	C11-C10-H10	118.5
C19-N5-H5N	116.5	C9-C10-H10	119.4
C25-N6-C24	114.6(6)	C10-C11-C6	119.3(6)
C25-N6-Pd1	109.1(5)	C10-C11-N3	118.9(6)
C24-N6-Pd1	136.3(5)	C6-C11-N3	121.7(6)
C29-N7-C26	107.8(6)	O1-C12-N3	120.4(6)
C29-N7-Pd1	134.7(6)	O1-C12-C13	118.7(7)
C26-N7-Pd1	117.1(5)	N3-C12-C13	120.8(6)
N1-C1-C2	109.0(7)	N4-C13-C14	124.6(6)
N1-C1-C30	124.1(7)	N4-C13-C12	117.6(6)
C2-C1-C30	126.8(7)	C14-C13-C12	117.8(6)
C3-C2-C1	108.3(7)	C15-C14-C13	117.7(6)
C3-C2-C31	126.4(8)	C15-C14-H14	121.8
C1-C2-C31	125.3(8)	C13-C14-H14	120.5
C2-C3-C4	105.2(7)	C14-C15-C16	119.7(7)
C2-C3-C32	129.5(8)	C14-C15-H15	120.3
C4-C3-C32	125.3(8)	C16-C15-H15	120.0
N1-C4-C5	113.7(7)	C17-C16-C15	118.3(7)
N1-C4-C3	109.0(7)	C17-C16-H16	120.3
C5-C4-C3	137.2(8)	C15-C16-H16	121.4
N2-C5-C4	119.2(7)	N4-C17-C16	122.9(6)
N2-C5-H5	120.1	N4-C17-C18	114.8(6)
C4-C5-H5	120.6	C16-C17-C18	122.1(6)
C7-C6-N2	118.2(7)	O2-C18-N5	124.1(6)
C7-C6-C11	118.2(7)	O2-C18-C17	122.3(6)
N2-C6-C11	123.6(6)	N5-C18-C17	113.6(6)
C8-C7-C6	121.4(7)	C20-C19-C24	120.6(6)
C8-C7-H7	120.0	C20-C19-N5	120.6(6)
C6-C7-H7	118.6	C24-C19-N5	118.8(6)

C21-C20-C19	120.7(7)	C2-C31-H31A	110.1
C21-C20-H20	119.6	C2-C31-H31B	109.1
C19-C20-H20	119.7	H31A-C31-H31B	109.5
C20-C21-C22	119.5(7)	C2-C31-H31C	109.3
C20-C21-H21	120.2	H31A-C31-H31C	109.5
C22-C21-H21	120.2	H31B-C31-H31C	109.5
C23-C22-C21	119.8(7)	C3-C32-C33	114.2(8)
C23-C22-H22	121.0	C3-C32-H32A	108.7
C21-C22-H22	119.2	C33-C32-H32A	110.6
C22-C23-C24	121.4(7)	C3-C32-H32B	108.3
C22-C23-H23	119.7	C33-C32-H32B	107.0
C24-C23-H23	119.0	H32A-C32-H32B	107.9
C23-C24-C19	117.9(7)	C34-C33-C32	112.1(11)
C23-C24-N6	120.5(6)	C34-C33-H33A	110.4
C19-C24-N6	121.5(6)	C32-C33-H33A	107.0
N6-C25-C26	120.8(7)	C34-C33-H33B	109.5
N6-C25-H25	120.4	C32-C33-H33B	109.9
C26-C25-H25	118.8	H33A-C33-H33B	107.9
N7-C26-C25	112.9(7)	C33-C34-H34A	108.1
N7-C26-C27	109.3(7)	C33-C34-H34B	109.7
C25-C26-C27	137.6(8)	H34A-C34-H34B	109.5
C28-C27-C26	106.8(7)	C33-C34-H34C	110.6
C28-C27-C35	125.6(8)	H34A-C34-H34C	109.5
C26-C27-C35	127.4(8)	H34B-C34-H34C	109.5
C27-C28-C29	106.8(7)	C36-C35-C27	119.4(9)
C27-C28-C38	127.4(7)	C36A-C35-C27	112.2(11)
C29-C28-C38	125.7(8)	C36A-C35-H35C	105.2
N7-C29-C28	109.1(7)	C27-C35-H35C	108.5
N7-C29-C30	122.9(7)	C36A-C35-H35D	112.2
C28-C29-C30	127.9(7)	C27-C35-H35D	110.2
C29-C30-C1	116.5(6)	H35C-C35-H35D	108.3
C29-C30-C39	112.0(6)	C36-C35-H35A	104.7
C1-C30-C39	106.1(6)	C27-C35-H35A	107.4
C29-C30-H30	107.1	C36-C35-H35B	108.9
C1-C30-H30	107.1	C27-C35-H35B	108.3
C39-C30-H30	107.6	H35A-C35-H35B	107.5

C35-C36-C37	113.5(10)	C40-C39-C30	119.9(7)
C35-C36-H36A	106.2	C39-C40-C41	120.3(7)
C37-C36-H36A	110.8	C39-C40-H40	119.0
C35-C36-H36B	109.3	C41-C40-H40	120.7
C37-C36-H36B	108.8	C42-C41-C40	121.1(7)
H36A-C36-H36B	108.1	C42-C41-H41	118.1
C36-C37-H37A	109.1	C40-C41-H41	120.8
C36-C37-H37B	108.6	C43-C42-C41	118.5(8)
H37A-C37-H37B	109.5	C43-C42-C45	121.5(7)
C36-C37-H37C	110.6	C41-C42-C45	120.0(8)
H37A-C37-H37C	109.5	C42-C43-C44	120.7(7)
H37B-C37-H37C	109.5	C42-C43-H43	119.5
C35-C36A-C37A	116.0(14)	C44-C43-H43	119.8
C37A-C36A-H35B	111.5	C39-C44-C43	120.9(7)
C35-C36A-H36C	104.2	C39-C44-H44	119.1
C37A-C36A-H36C	105.4	C43-C44-H44	120.0
C35-C36A-H36D	111.4	C42-C45-H45A	110.5
C37A-C36A-H36D	111.4	C42-C45-H45B	109.0
H36C-C36A-H36D	107.7	H45A-C45-H45B	109.5
C36A-C37A-Cl2B	123.9(18)	C42-C45-H45C	108.9
C36A-C37A-H37B	114.4	H45A-C45-H45C	109.4
Cl2B-C37A-H37B	115.1	H45B-C45-H45C	109.5
C36A-C37A-H37D	110.2	Cl2A-C1A-Cl1A	108.2(7)
C36A-C37A-H37E	106.0	Cl2A-C1A-H1AA	109.3
H37D-C37A-H37E	109.5	Cl1A-C1A-H1AA	109.5
C36A-C37A-H37F	112.1	Cl2A-C1A-H1AB	110.8
H37D-C37A-H37F	109.5	Cl1A-C1A-H1AB	110.3
H37E-C37A-H37F	109.5	H1AA-C1A-H1AB	108.7
C28-C38-H38A	109.6	Cl1A-C1A-H1AC	124.3
C28-C38-H38B	110.0	H1AB-C1A-H1AC	117.3
H38A-C38-H38B	109.5	Cl4A-C1AA-Cl3A	108.4(8)
C28-C38-H38C	108.7	Cl4A-C1AA-H1AA	124.3
H38A-C38-H38C	109.5	Cl3A-C1AA-H1AA	96.5
H38B-C38-H38C	109.5	Cl4A-C1AA-H1AC	107.8
C44-C39-C40	118.5(7)	Cl3A-C1AA-H1AC	107.8
C44-C39-C30	121.5(7)	Cl4A-C1AA-H1AD	111.8

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Cl3A-C1AA-H1AD	112.2
H1AA-C1AA-H1AD	102.7
H1AC-C1AA-H1AD	108.7
C1B-Cl2B-C37A	130.5(9)
Cl1B-C1B-Cl2B	108.6(9)
Cl1B-C1B-H1BA	110.3
Cl2B-C1B-H1BA	111.7
Cl1B-C1B-H1BB	108.9
Cl2B-C1B-H1BB	109.0
H1BA-C1B-H1BB	108.2

Supplementary Material (ESI) for Chemical Communications

X-ray experimental for complex 4.

X-ray experimental for complex **4**, $(C_{45}H_{42}N_7O_2)PdCl - CH_3CN - C_3H_6O - C_5H_{12}$ (CCDC #615936): Crystals grew as long black needles by slow evaporation. The data crystal was cut from a long needle and had approximate dimensions; 0.42 x 0.08 x 0.07 mm. The data were collected on a Nonius Kappa CCD diffractometer using a graphite monochromator with MoKα radiation (λ = 0.71073 Å). A total of 363 frames of data were collected using ω-scans with a scan range of 1° and a counting time of 191 seconds per frame. The data were collected at 153 K using an Oxford Cryostream low temperature device. Details of crystal data, data collection and structure refinement are listed in Table 2. Data reduction were performed using DENZO-SMN.[1] The structure was solved by direct methods using SIR97,[2] and refined by full-matrix least-squares on F^2 with anisotropic displacement parameters for the non-H atoms using SHELXL-97.[3] The hydrogen atoms were calculated in ideal positions with isotropic displacement parameters set to 1.2xUeq of the attached atom (1.5xUeq for the methyl hydrogen atoms).

One of the methyl groups on an n-propyl group was disordered about two positions defined by C37 and C37a. The disorder was modeled by assigning the variable x to the site occupancy for C37 and the variable (1-x) to the site occupancy for C37a. The C-C bond length for the two atoms were restrained to be equal throughout the refinement. An isotropic displacement parameter was constrained to be equal for both C37 and C37a. In this way, the site occupancy for C37 refined to 60(2)%.

Three regions were observed to contain solvent molecules. From the geometry of the peaks in these regions, it appeared that the solvent molecules consisted of acetonitrile, acetone and n-pentane. The solvent, particularly, the n-pentane was difficult to model. As a result, the utility SQUEEZE in PLATON98[4] was used to remove the contribution of the solvent to the structure factors. PLATON98 was used as incorporated into WinGX.[5]

The function, $\Sigma w(|F_o|^2 - |F_c|^2)^2$, was minimized, where $w = 1/[(\sigma(F_o))^2 + (0.055*P)^2]$ and $P = (|F_o|^2 + 2|F_c|^2)/3$. $R_w(F^2)$ refined to 0.158, with R(F) equal to 0.0688 and a goodness of fit, S, = 1.05. Definitions used for calculating R(F), $R_w(F^2)$ and the goodness of fit, S, are given below.[6] The data were checked for secondary extinction effects but no correction was necessary.

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Neutral atom scattering factors and values used to calculate the linear absorption coefficient are from the International Tables for X-ray Crystallography (1992).[7]

Table 4. Crystal data and structure refinement for **4**.

Empirical formula	C55 H63 Cl N8 O3 Pd	
Formula weight	1025.98	
Temperature	153(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 35.3431(8) Å	α= 90°.
	b = 16.0453(6) Å	β= 127.270(2)°.
	c = 21.7368(7) Å	γ = 90°.
Volume	9809.5(5) $Å^3$	
Z	8	
Density (calculated)	1.389 Mg/m^3	
Absorption coefficient	0.486 mm^{-1}	
F(000)	4288	
Crystal size	0.42 x 0.08 x 0.07 mm	
Theta range for data collection	2.92 to 25.00°.	
Index ranges	-41<=h<=42, -16<=k<=19, -25<=l<=25	
Reflections collected	15320	
Independent reflections	8607 [R(int) = 0.0932]	
Completeness to theta = 25.00°	99.7 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8607 / 13 / 514	
Goodness-of-fit on F^2	1.047	
Final R indices [I>2sigma(I)]	R1 = 0.0688, wR2 = 0.1421	
R indices (all data)	R1 = 0.1564, wR2 = 0.1581	
Largest diff. peak and hole	0.651 and -0.362 $e.Å^{-3}$	

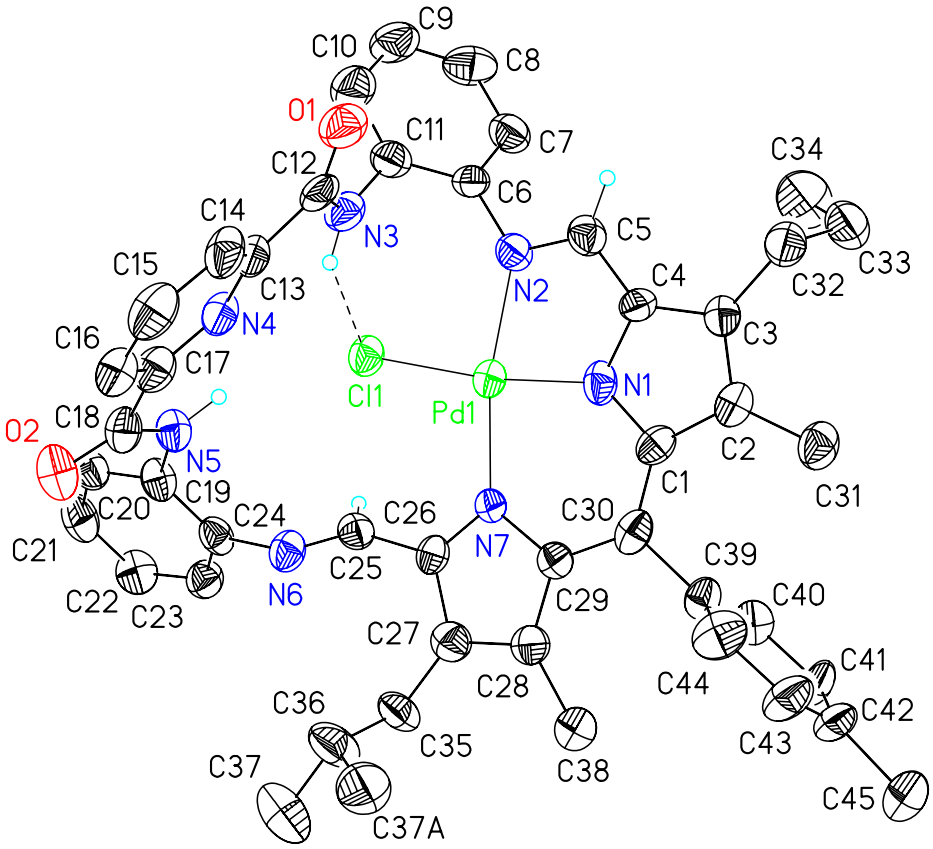


Fig. 4. View of the Pd complex in **4** showing the atom labeling scheme. Displacement ellipsoids are scaled to the 50% probability level. Most hydrogen atoms have been removed for clarity. A methyl group on one of the n-propyl groups is disordered about two positions given by C37 and C37A.

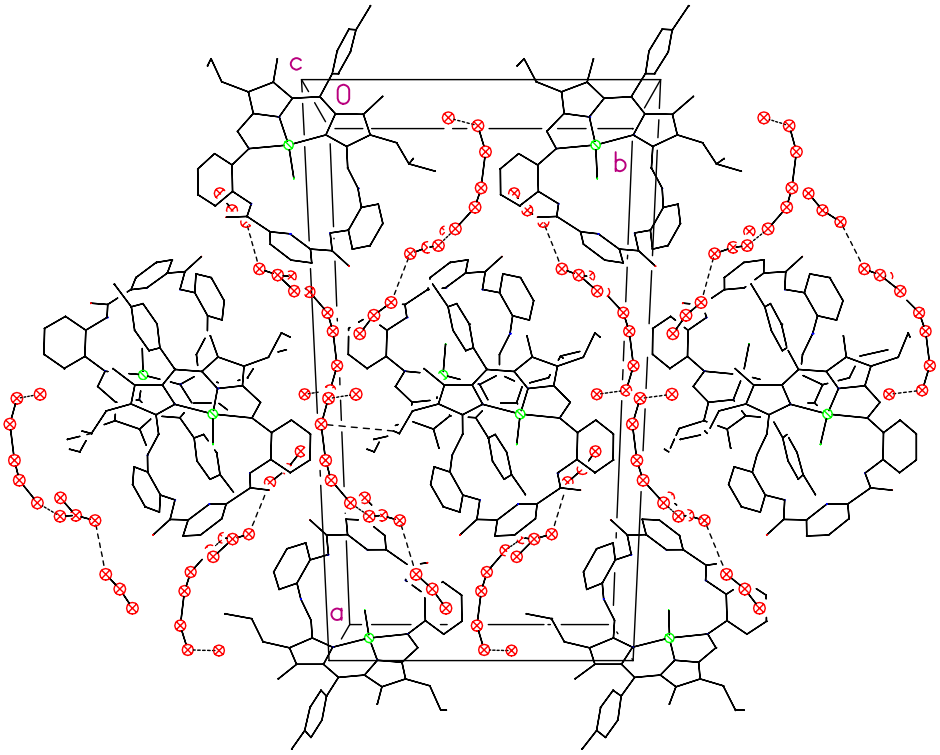


Fig. 5. Unit cell packing diagram for complex **4**. The view is approximately down the **c** axis. The red spheres represent the solvent molecule locations.

Table 5. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **4**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Pd1	4632(1)	3729(1)	3849(1)	36(1)
Cl1	4060(1)	3828(1)	4055(1)	42(1)
O1	3245(2)	1822(3)	1613(3)	54(1)
O2	2385(2)	5685(3)	1754(3)	55(1)
N1	5107(2)	3488(4)	3686(3)	40(2)
N2	4562(2)	2445(3)	3721(3)	38(1)
N3	3543(2)	2521(4)	2737(3)	42(2)
N4	2969(2)	3804(4)	1942(3)	40(1)

N5	3026(2)	5047(3)	2812(3)	41(1)
N6	3861(2)	5951(3)	3661(3)	42(2)
N7	4816(2)	4955(3)	3945(3)	31(1)
C1	5421(2)	3934(4)	3658(4)	38(2)
C2	5677(2)	3363(4)	3516(4)	37(2)
C3	5516(2)	2570(4)	3488(4)	35(2)
C4	5158(2)	2660(4)	3589(4)	38(2)
C5	4853(2)	2119(4)	3621(4)	41(2)
C6	4286(3)	1854(4)	3795(4)	40(2)
C7	4520(3)	1232(5)	4365(4)	49(2)
C8	4248(3)	639(5)	4403(5)	64(2)
C9	3758(3)	647(5)	3884(5)	63(2)
C10	3525(3)	1263(5)	3333(5)	61(2)
C11	3786(3)	1871(4)	3278(4)	43(2)
C12	3276(2)	2453(5)	1968(5)	43(2)
C13	2983(2)	3207(4)	1522(4)	36(2)
C14	2739(2)	3261(5)	742(4)	49(2)
C15	2444(3)	3941(5)	343(4)	54(2)
C16	2408(2)	4565(5)	760(4)	45(2)
C17	2684(2)	4472(5)	1561(4)	44(2)
C18	2680(3)	5132(5)	2044(4)	43(2)
C19	3098(2)	5602(4)	3379(4)	38(2)
C20	2762(2)	5714(4)	3488(4)	43(2)
C21	2847(3)	6279(5)	4039(4)	54(2)
C22	3276(3)	6709(5)	4499(4)	49(2)
C23	3617(2)	6555(4)	4398(4)	44(2)
C24	3526(2)	6025(4)	3832(4)	38(2)
C25	4256(2)	5599(4)	4159(4)	38(2)
C26	4623(2)	5597(4)	4018(4)	34(2)
C27	4816(2)	6385(4)	4011(4)	45(2)
C28	5167(2)	6180(4)	3934(4)	41(2)
C29	5164(2)	5298(4)	3879(4)	39(2)
C30	5454(2)	4812(4)	3769(3)	39(2)
C31	6060(2)	3537(4)	3430(4)	48(2)
C32	5677(2)	1767(4)	3380(4)	47(2)
C33	6108(3)	1381(5)	4098(4)	59(2)

C34	6051(3)	1165(5)	4710(5)	83(3)
C35	4684(3)	7234(4)	4122(4)	49(2)
C36	4277(3)	7639(5)	3401(5)	64(2)
C37	4171(5)	8489(8)	3619(9)	92(5)
C37A	4334(7)	7843(13)	2756(11)	85(7)
C38	5467(3)	6841(4)	3913(5)	60(2)
C39	5846(2)	5249(4)	3816(4)	37(2)
C40	6280(2)	5384(4)	4508(4)	44(2)
C41	6648(2)	5778(4)	4556(4)	46(2)
C42	6575(3)	6039(4)	3890(5)	50(2)
C43	6148(3)	5887(5)	3180(5)	56(2)
C44	5785(3)	5499(5)	3149(4)	56(2)
C45	6965(3)	6524(5)	3946(5)	77(3)

Table 6. Bond lengths [Å] and angles [°] for **4**.

Pd1-N1	1.950(5)	C8-C9	1.382(10)
Pd1-N7	2.042(5)	C8-H8A	0.96
Pd1-N2	2.073(5)	C9-C10	1.376(10)
Pd1-Cl1	2.3278(15)	C9-H9A	0.96
O1-C12	1.236(7)	C10-C11	1.396(9)
O2-C18	1.213(8)	C10-H10A	0.96
N1-C1	1.354(7)	C12-C13	1.503(9)
N1-C4	1.375(7)	C13-C14	1.364(9)
N2-C5	1.284(7)	C14-C15	1.390(9)
N2-C6	1.438(8)	C14-H14A	0.96
N3-C12	1.335(8)	C15-C16	1.407(9)
N3-C11	1.409(8)	C15-H15A	0.96
N3-H3N	0.90	C16-C17	1.396(9)
N4-C13	1.345(8)	C16-H16A	0.96
N4-C17	1.355(8)	C17-C18	1.498(9)
N5-C18	1.352(8)	C19-C20	1.358(8)
N5-C19	1.412(7)	C19-C24	1.385(9)
N5-H5N	0.90	C20-C21	1.382(9)
N6-C25	1.268(7)	C20-H20A	0.96
N6-C24	1.445(8)	C21-C22	1.393(10)
N7-C26	1.297(7)	C21-H21A	0.96
N7-C29	1.432(8)	C22-C23	1.374(9)
C1-C30	1.422(9)	C22-H22A	0.96
C1-C2	1.444(9)	C23-C24	1.363(9)
C2-C3	1.379(8)	C23-H23A	0.96
C2-C31	1.501(8)	C25-C26	1.503(8)
C3-C4	1.418(8)	C25-H25A	0.96
C3-C32	1.482(9)	C26-C27	1.442(9)
C4-C5	1.419(8)	C27-C28	1.389(9)
C5-H5A	0.96	C27-C35	1.505(9)
C6-C11	1.407(9)	C28-C29	1.419(9)
C6-C7	1.405(9)	C28-C38	1.518(9)
C7-C8	1.390(9)	C29-C30	1.419(8)
C7-H7A	0.96	C30-C39	1.501(8)

C31-H31A	0.96	C37-H37C	0.96
C31-H31B	0.96	C37A-H37D	0.96
C31-H31C	0.96	C37A-H37E	0.96
C32-C33	1.504(9)	C37A-H37F	0.96
C32-H32A	0.96	C38-H38A	0.96
C32-H32B	0.96	C38-H38B	0.96
C33-C34	1.503(9)	C38-H38C	0.96
C33-H33A	0.96	C39-C40	1.366(8)
C33-H33B	0.96	C39-C44	1.389(9)
C34-H34A	0.96	C40-C41	1.393(8)
C34-H34B	0.96	C40-H40A	0.96
C34-H34C	0.96	C41-C42	1.371(9)
C35-C36	1.489(9)	C41-H41A	0.96
C35-H35A	0.96	C42-C43	1.378(10)
C35-H35B	0.96	C42-C45	1.524(9)
C36-C37	1.562(12)	C43-C44	1.391(9)
C36-C37A	1.567(13)	C43-H43A	0.96
C36-H36A	0.96	C44-H44A	0.96
C36-H36B	0.96	C45-H45A	0.96
C36-H36C	0.96	C45-H45B	0.96
C37-H37A	0.96	C45-H45C	0.96
C37-H37B	0.96		
N1-Pd1-N7	87.4(2)	C11-N3-H3N	116.5
N1-Pd1-N2	80.0(2)	C13-N4-C17	118.0(6)
N7-Pd1-N2	167.4(2)	C18-N5-C19	124.4(6)
N1-Pd1-Cl1	172.47(18)	C18-N5-H5N	117.5
N7-Pd1-Cl1	100.03(14)	C19-N5-H5N	118.2
N2-Pd1-Cl1	92.60(15)	C25-N6-C24	117.8(6)
C1-N1-C4	108.4(5)	C26-N7-C29	104.6(5)
C1-N1-Pd1	136.3(5)	C26-N7-Pd1	128.3(4)
C4-N1-Pd1	115.2(4)	C29-N7-Pd1	126.8(4)
C5-N2-C6	114.6(6)	N1-C1-C30	120.0(6)
C5-N2-Pd1	112.6(5)	N1-C1-C2	108.1(6)
C6-N2-Pd1	132.5(4)	C30-C1-C2	131.9(6)
C12-N3-C11	126.5(6)	C3-C2-C1	107.6(5)
C12-N3-H3N	117.0	C3-C2-C31	122.9(6)

C1-C2-C31	129.5(6)	C13-C14-H14A	120.4
C2-C3-C4	106.3(6)	C14-C15-C16	118.9(7)
C2-C3-C32	128.5(6)	C14-C15-H15A	120.9
C4-C3-C32	125.2(6)	C16-C15-H15A	120.2
N1-C4-C5	114.2(6)	C17-C16-C15	117.4(7)
N1-C4-C3	109.6(6)	C17-C16-H16A	120.7
C5-C4-C3	136.3(7)	C15-C16-H16A	121.9
N2-C5-C4	118.0(6)	N4-C17-C16	123.0(6)
N2-C5-H5A	122.0	N4-C17-C18	116.8(6)
C4-C5-H5A	120.1	C16-C17-C18	120.2(7)
C11-C6-C7	120.2(6)	O2-C18-N5	124.4(7)
C11-C6-N2	120.4(6)	O2-C18-C17	121.5(7)
C7-C6-N2	119.3(6)	N5-C18-C17	114.1(7)
C8-C7-C6	118.6(7)	C20-C19-C24	120.4(6)
C8-C7-H7A	121.7	C20-C19-N5	121.4(7)
C6-C7-H7A	119.7	C24-C19-N5	118.2(6)
C7-C8-C9	121.0(8)	C19-C20-C21	119.0(7)
C7-C8-H8A	119.1	C19-C20-H20A	119.6
C9-C8-H8A	119.9	C21-C20-H20A	121.4
C10-C9-C8	120.7(7)	C20-C21-C22	121.2(6)
C10-C9-H9A	119.9	C20-C21-H21A	119.5
C8-C9-H9A	119.4	C22-C21-H21A	119.3
C9-C10-C11	119.9(7)	C23-C22-C21	118.4(7)
C9-C10-H10A	119.8	C23-C22-H22A	121.6
C11-C10-H10A	120.2	C21-C22-H22A	120.0
C6-C11-C10	119.5(7)	C24-C23-C22	120.5(7)
C6-C11-N3	121.2(6)	C24-C23-H23A	119.5
C10-C11-N3	119.2(7)	C22-C23-H23A	119.9
O1-C12-N3	125.2(7)	C23-C24-C19	120.4(6)
O1-C12-C13	119.3(7)	C23-C24-N6	120.1(6)
N3-C12-C13	115.4(6)	C19-C24-N6	119.3(6)
N4-C13-C14	122.9(7)	N6-C25-C26	117.8(6)
N4-C13-C12	115.5(6)	N6-C25-H25A	120.9
C14-C13-C12	121.6(7)	C26-C25-H25A	121.3
C15-C14-C13	119.7(7)	N7-C26-C27	114.2(6)
C15-C14-H14A	119.8	N7-C26-C25	127.5(6)

C27-C26-C25	118.2(6)	H34B-C34-H34C	109.5
C28-C27-C26	104.9(6)	C36-C35-C27	114.9(6)
C28-C27-C35	128.3(7)	C36-C35-H35A	106.8
C26-C27-C35	126.7(6)	C27-C35-H35A	107.9
C27-C28-C29	106.4(6)	C36-C35-H35B	109.4
C27-C28-C38	121.9(6)	C27-C35-H35B	109.7
C29-C28-C38	131.7(6)	H35A-C35-H35B	107.8
C30-C29-N7	123.8(6)	C35-C36-C37	108.5(8)
C30-C29-C28	126.3(6)	C35-C36-C37A	117.9(10)
N7-C29-C28	109.9(5)	C37-C36-C37A	105.9(11)
C29-C30-C1	125.5(6)	C35-C36-H36A	108.9
C29-C30-C39	117.6(6)	C37-C36-H36A	109.5
C1-C30-C39	116.8(6)	C37A-C36-H36A	105.9
C2-C31-H31A	109.9	C35-C36-H36B	111.0
C2-C31-H31B	109.0	C37-C36-H36B	110.2
H31A-C31-H31B	109.5	H36A-C36-H36B	108.7
C2-C31-H31C	109.6	C35-C36-H36C	109.8
H31A-C31-H31C	109.5	C37A-C36-H36C	107.5
H31B-C31-H31C	109.5	H36A-C36-H36C	106.3
C3-C32-C33	116.0(6)	H36B-C36-H36C	112.0
C3-C32-H32A	108.6	C36-C37-H37A	108.7
C33-C32-H32A	109.8	H36C-C37-H37A	106.0
C3-C32-H32B	107.7	C36-C37-H37B	109.9
C33-C32-H32B	106.5	H36C-C37-H37B	115.1
H32A-C32-H32B	107.8	H37A-C37-H37B	109.5
C34-C33-C32	114.7(6)	C36-C37-H37C	109.8
C34-C33-H33A	108.6	H36C-C37-H37C	107.2
C32-C33-H33A	106.5	H37A-C37-H37C	109.5
C34-C33-H33B	108.3	H37B-C37-H37C	109.5
C32-C33-H33B	110.7	C36-C37A-H37D	108.1
H33A-C33-H33B	107.8	H36B-C37A-H37D	101.2
C33-C34-H34A	110.7	C36-C37A-H37E	110.8
C33-C34-H34B	108.7	H36B-C37A-H37E	121.3
H34A-C34-H34B	109.5	H37D-C37A-H37E	109.5
C33-C34-H34C	109.0	C36-C37A-H37F	109.6
H34A-C34-H34C	109.5	H36B-C37A-H37F	105.3

H37D-C37A-H37F	109.5
H37E-C37A-H37F	109.5
C28-C38-H38A	110.6
C28-C38-H38B	108.9
H38A-C38-H38B	109.5
C28-C38-H38C	108.9
H38A-C38-H38C	109.5
H38B-C38-H38C	109.5
C40-C39-C44	117.7(6)
C40-C39-C30	121.6(6)
C44-C39-C30	120.6(6)
C39-C40-C41	121.9(6)
C39-C40-H40A	118.8
C41-C40-H40A	119.3
C42-C41-C40	119.2(7)
C42-C41-H41A	119.6
C40-C41-H41A	121.2
C41-C42-C43	120.5(6)
C41-C42-C45	119.2(7)
C43-C42-C45	120.2(7)
C42-C43-C44	119.0(7)
C42-C43-H43A	120.2
C44-C43-H43A	120.8
C43-C44-C39	121.5(7)
C43-C44-H44A	120.1
C39-C44-H44A	118.4
C42-C45-H45A	108.9
C42-C45-H45B	109.6
H45A-C45-H45B	109.5
C42-C45-H45C	109.9
H45A-C45-H45C	109.5
H45B-C45-H45C	109

Fig. 6. ^{1}H NMR spectrum of complex **3** recorded in CD_2Cl_2 at -50°C (top trace) and 27°C (bottom trace).

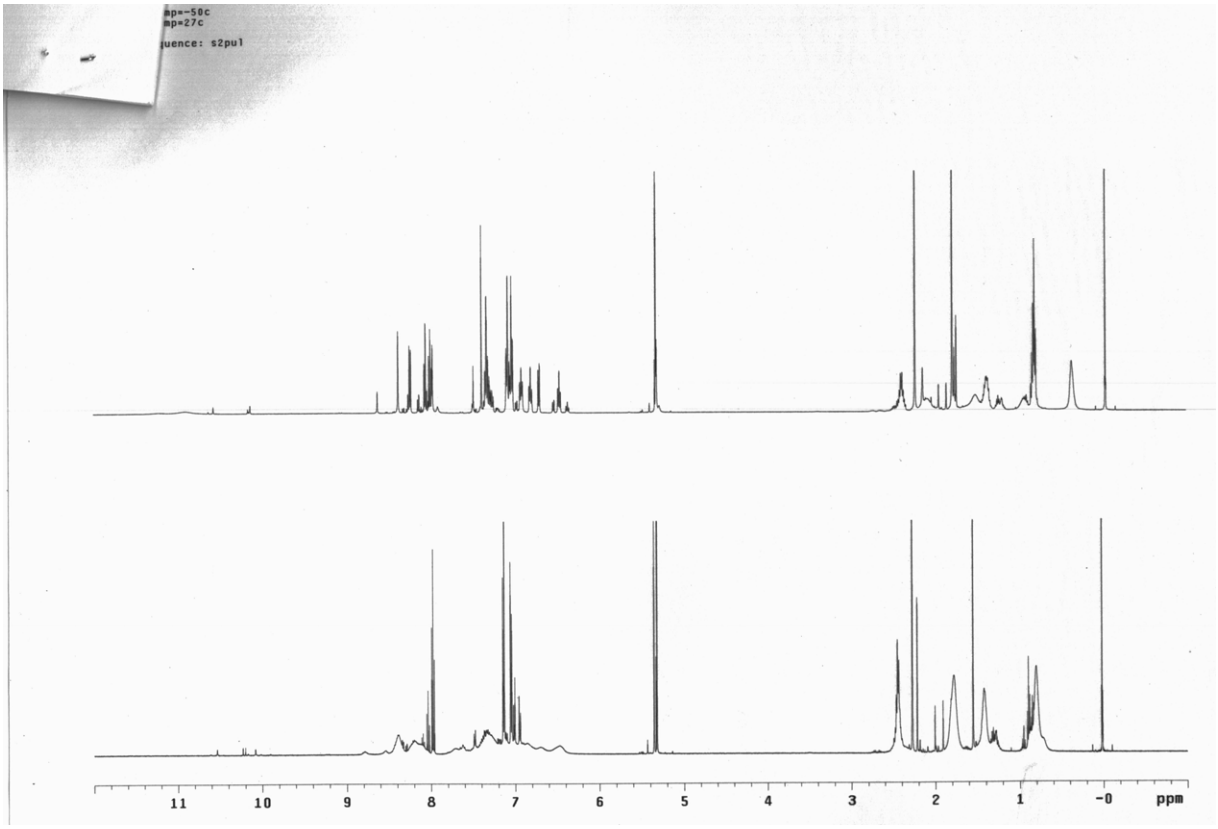


Fig. 7. ^{1}H NMR spectrum of complex **3** recorded in CD_2Cl_2 in the presence of (1) added D_2O (top trace), after further (2) dilution with CD_2Cl_2, and (3) in the absence of any additives (bottom trace).

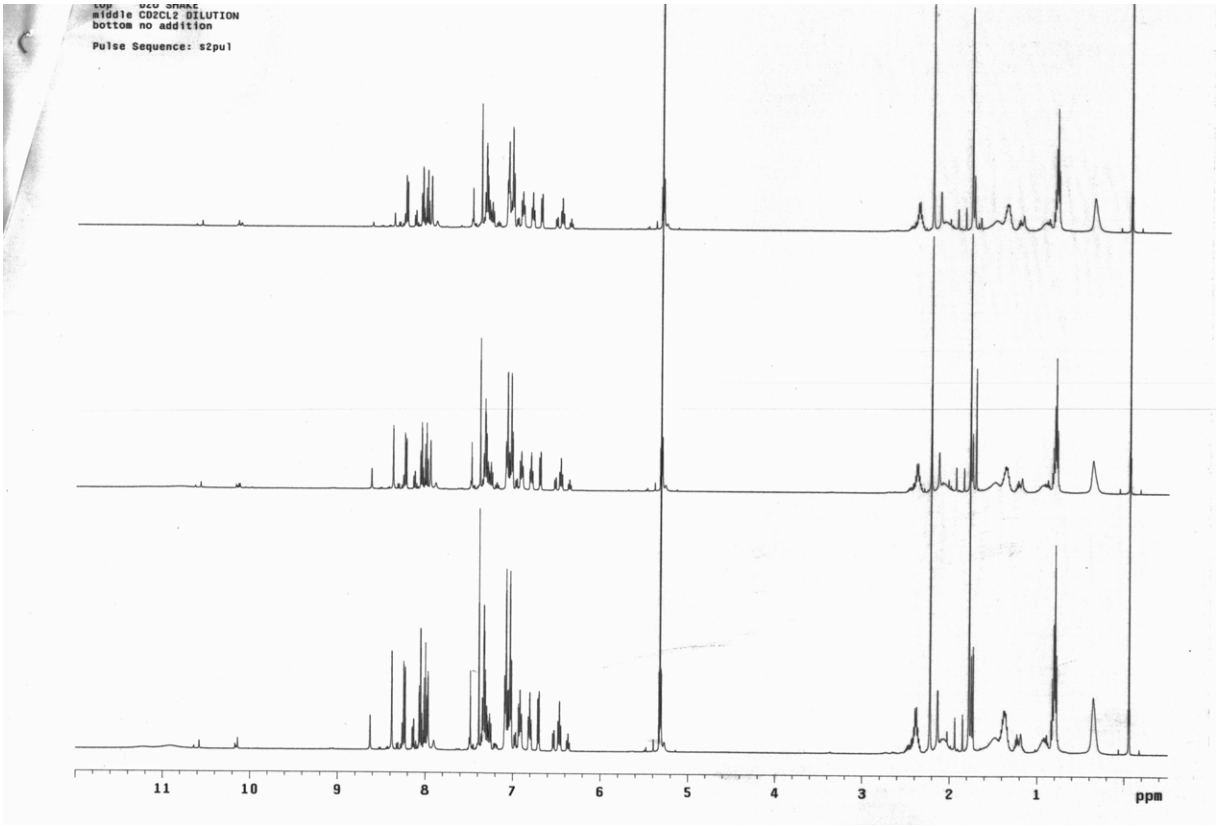


Fig. 8. ^{1}H NMR COSY spectrum for complex **3**.

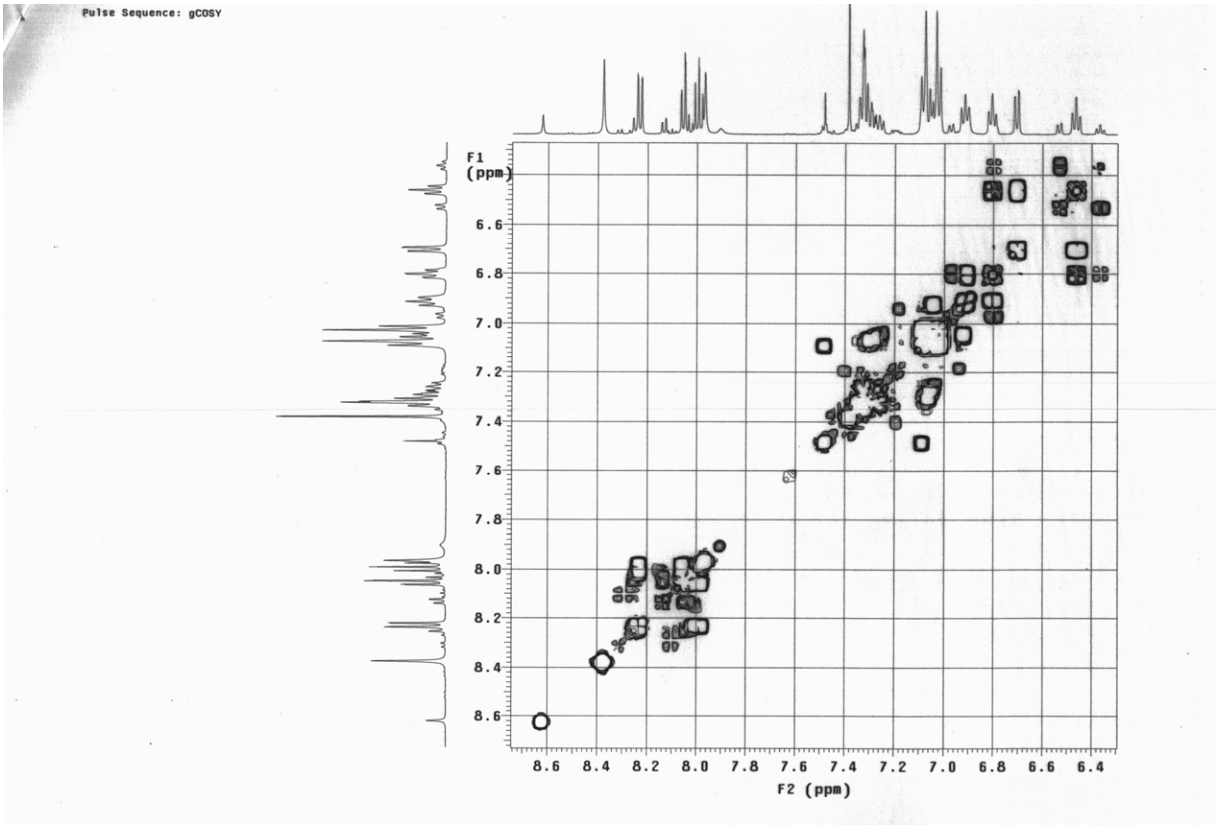


Fig. 9. NMR HSQC spectrum for complex **3**.

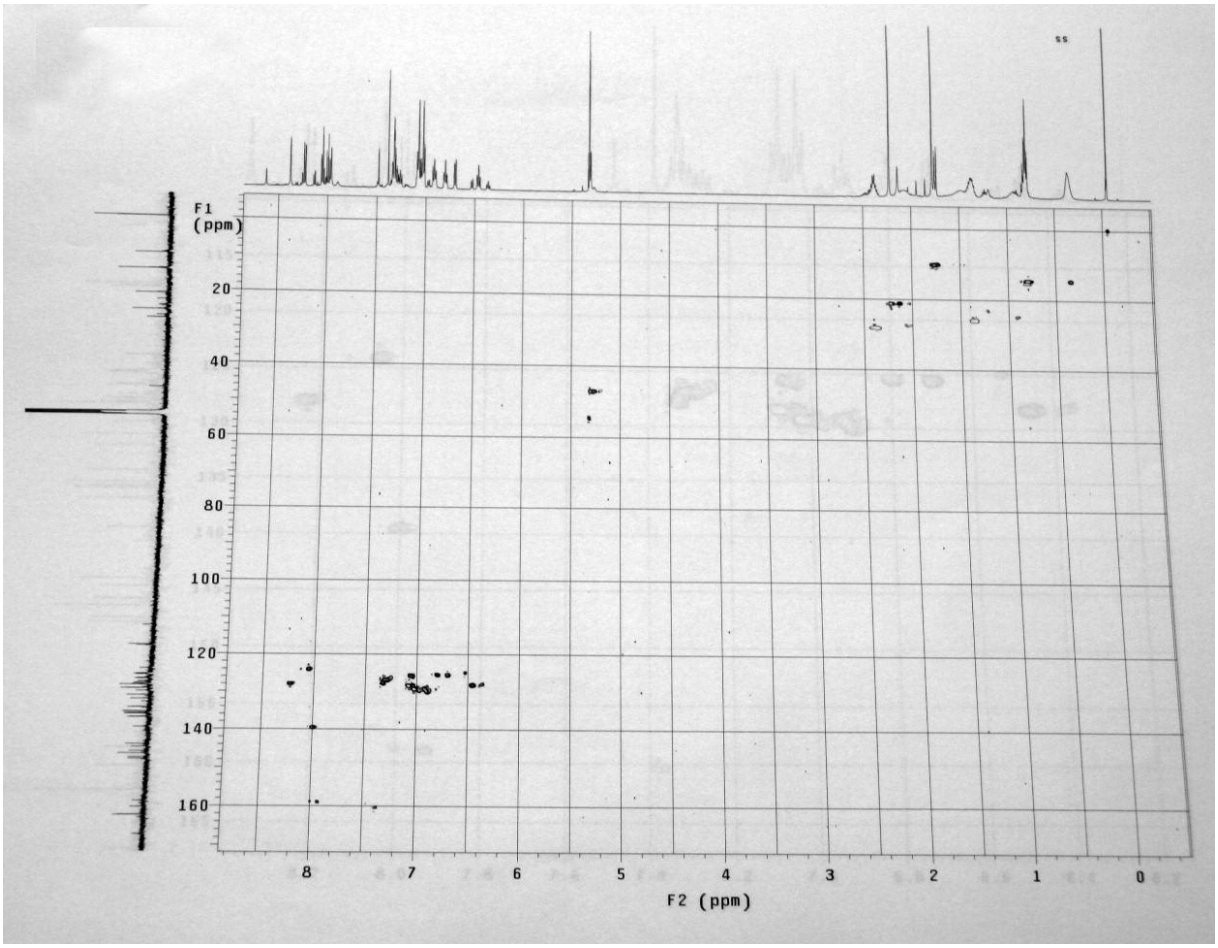
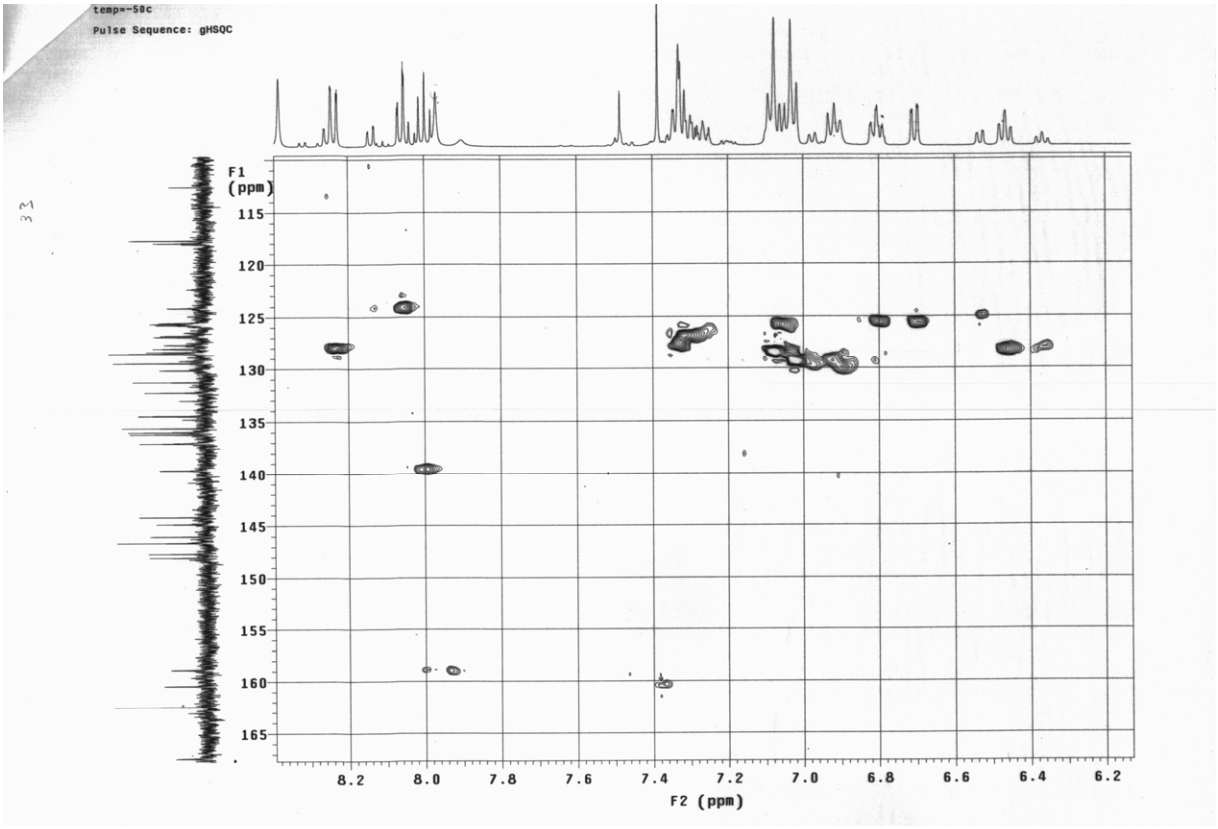


Fig. 10. NMR HSQC spectrum for complex **3**.



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6. $R_w(F^2) = \{\Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w(|F_o|)^4\}^{1/2}$ where w is the weight given each reflection.

   $R(F) = \Sigma(|F_o| - |F_c|)/\Sigma|F_o|\}$ for reflections with $F_o > 4(\sigma(F_o))$.

   $S = [\Sigma w(|F_o|^2 - |F_c|^2)^2/(n - p)]^{1/2}$, where n is the number of reflections and p is the number of refined parameters.

7. International Tables for X-ray Crystallography (1992). Vol. C, Tables 4.2.6.8 and 6.1.1.4, A. J. C. Wilson, editor, Boston: Kluwer Academic Press.