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Synthesis, Solid-State and Solution Structure, and Reactivity of *cis*-$[Pt(C_6F_5)_2(NC_5H_4\text{-}2\text{-}CH_2C_6H_5)]$: A Compound Displaying an Arene–Platinum Interaction

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The reaction of *cis*-$[Pt(C_6F_5)_2(THF)_2]$ and 2-benzylpyridine (molar ratio 1:1) in CH_2Cl_2 yields *cis*-$[Pt(C_6F_5)_2(NC_5H_4\text{-}2\text{-}CH_2C_6H_5)]$ (1), which in the solid state (X-ray) shows a weak η^2-benzyl–Pt interaction. The structure in solution is discussed on the basis of its NMR data (^1H, ^{13}C, and ^{19}F). Complex 1 reacts with CO, yielding *cis*-$[Pt(C_6F_5)_2(NC_5H_4\text{-}2\text{-}CH_2C_6H_5)(CO)]$ (2), which does not contain any benzyl–Pt interaction. Crystal data for complex 1: $C_{24}H_{11}NF_{10}Pt$, M = 698.44; monoclinic, space group $P2_1/n$; $a = 9.283(1)$, $b = 13.836(1)$, $c = 16.615(1)$ Å; $\beta = 95.25(1)°$; $V = 2124.9(3)$ Å^3; $Z = 4$. The final R value is 0.0276 for 2513 unique observed ($F_o^2 \geq 2.5\sigma(F_o^2)$) absorption-corrected reflections collected at 223 K.

Introduction

η^2-Arene–metal complexes have been proposed as intermediates in homogeneous hydrogenation reactions of arenes catalyzed by transition-metal complexes[1] as well as in *ortho*-metalation reactions.[2] Most of these complexes have been obtained by generating *in situ* an electrophilic center which reacts with one π-bond of a benzene molecule[3] or of a phenyl fragment of the ligand present in the complex. In the latter case an intramolecular η^2-arene–metal compound is obtained.[4,5]

Recently we have prepared *cis*-$[Pt(C_6F_5)_2\{(\eta^2\text{-}C_6H_5)C(C_6H_5){=}NN{=}C(C_6H_5)_2\}]$, by reacting *cis*-$[Pt(C_6F_5)_2(THF)_2]$ (THF = tetrahydrofuran) with either Ph_2CN_2 or $(C_6H_5)_2C{=}NN{=}C(C_6H_5)_2$, and its thermolysis has rendered $[Pt(C_6F_5)\{(2\text{-}C_6H_4)C(C_6H_5){=}NN{=}(\eta^2\text{-}C_6H_5)C(C_6H_5)\}]$; both complexes display unusual intramolecular η^2-arene–Pt interactions,[5] the structure of the latter being established by X-ray diffraction. Moreover, the reaction between *cis*-$[Pd(C_6F_5)_2(THF)_2]$ and $C_6H_5CH_2NMe_2$ has rendered *cis*-$[Pd(C_6F_5)_2\{(\eta^1\text{-}C_6H_5)CH_2NMe_2\}]$, an unprecedented palladium complex displaying a η^1-arene–Pd interaction.[6]

In this paper we report the synthesis and structural characterization of *cis*-$[Pt(C_6F_5)_2(NC_5H_4\text{-}2\text{-}CH_2C_6H_5)]$ (1), a compound displaying a η^2-arene–Pt interaction in the solid state (X-ray). Its structure in solution is also discussed on the basis of its NMR data (^1H, ^{13}C, and ^{19}F). This arene–Pt interaction is weak and can be released by reaction of 1 with CO.

Results and Discussion

(a) Synthesis of *cis*-$[Pt(C_6F_5)_2(NC_5H_4\text{-}2\text{-}CH_2C_6H_5)]$ (1). Complex 1 can be obtained in good yield by reaction between *cis*-$[Pt(C_6F_5)_2(THF)_2]$ and 2-benzylpyridine (molar ratio 1:1) in CH_2Cl_2. Elemental analyses and relevant IR and NMR data are given in the Experimental Section.

(b) Structural Study. X-ray Crystal Structure. Figure 1 shows a structural drawing of the molecule. Selected bond distances and angles are given in Table I. As can be seen from Figure 1, the 2-benzylpyridine molecule, $NC_5H_4\text{-}2\text{-}CH_2C_6H_5$, is bonded to the "*cis*-Pt$(C_6F_5)_2$" fragment as a chelate ligand by using the N donor atom and a η^2 interaction (C(19) and C(20)) of the phenyl ring. The Pt–C distances for the η^2 interaction are equal within experimental error (Pt–C(19) = 2.375(7) Å and Pt–C(20) = 2.363(8) Å) and similar to the shorter distance found in the previous η^2-arene–Pt compound that was structurally characterized.[5] The coordinated phenyl ring is essentially planar, and the C–C distances within the ring show the sequence short–long–short, consistent with a perceptible loss of aromaticity as a consequence of the η^2 coordination. The coordinated C=C double bond (C(19)–C(20) = 1.400(10) Å) is longer than the other two (C(21)–C(22) = 1.352(13) Å and C(23)–C(24) = 1.345(13) Å), the former being similar to the corresponding distances found in other η^2-arene–metal complexes.[3-5] The C(19)–C(20) bond forms an angle of 10.0° with the perpendicular to the best least-squares coordination plane of platinum. Finally, the Pt–N distance has a normal value for neutral or anionic platinum complexes containing neutral nitrogen ligands[7,8] and the Pt–C distances for the pentafluorophenyl

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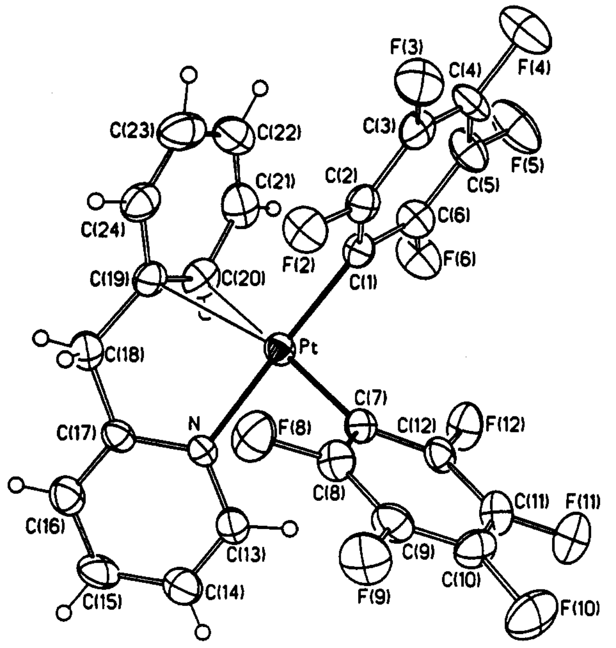

Figure 1. Molecular structure of *cis*-[Pt(C_6F_5)$_2${NC_5H_4-2-CH_2((1,2-η^2)-C_6H_5)}] (1).

Table I. Selected Bond Distances and Angles for *cis*-[Pt(C_6F_5)$_2${NC_5H_4-2-CH_2((1,2-η^2)-C_6H_5)}] (1)

	Bond Distances (Å)		
Pt–C(1)	2.004(7)	Pt–C(7)	2.000(7)
Pt–N	2.086(6)	Pt–C′[a]	2.263(8)
Pt–C(19)	2.375(7)	Pt–C(20)	2.363(8)
C(19)–C(20)	1.400(10)	C(20)–C(21)	1.421(12)
C(21)–C(22)	1.352(13)	C(22)–C(23)	1.408(13)
C(23)–C(24)	1.345(13)	C(19)–C(24)	1.411(10)
	Bond Angles (deg)		
C(1)–Pt–C(7)	87.1(3)	C(1)–Pt–N	178.3(2)
C(7)–Pt–N	92.6(3)	C(1)–Pt–C′	99.7(3)
C(7)–Pt–C′	170.0(3)	N–Pt–C′	80.8(3)
C(1)–Pt–C(19)	102.2(3)	C(7)–Pt–C(19)	153.9(3)
N–Pt–C(19)	78.8(2)	C(1)–Pt–C(20)	96.4(3)
C(7)–Pt–C(20)	169.7(3)	N–Pt–C(20)	83.6(3)
C(19)–Pt–C(20)	34.4(3)	Pt–N–C(13)	123.4(5)
Pt–N–C(17)	117.5(4)	C(19)–C(20)–C(21)	118.9(7)
C(20)–C(21)–C(22)	120.6(8)	C(21)–C(22)–C(23)	120.1(9)
C(22)–C(23)–C(24)	120.3(8)	C(19)–C(24)–C(23)	121.2(8)
C(20)–C(19)–C(24)	118.8(7)		

[a] C′ denotes the midpoint of the coordinated C=C (C(19)–C(20)).

groups are similar to those found in other (pentafluorophenyl)platinum complexes.[9]

NMR Spectra. The ^{1}H NMR signals for 1 were first tentatively assigned on the basis of their characteristic parameters (chemical shifts and coupling constants) and then confirmed by a ^{1}H,^{1}H-COSY experiment (Figure 2). Only one signal is observed for the two methylene protons (δ 4.46 ppm). In addition, the two *ortho* H atoms (H(20) and H(24)) are also isochronous (δ 6.95 ppm). These spectroscopic features are inconsistent with the static η^2-arene–Pt interaction observed in the solid state and suggest, rather, that a mirror plane is developed on the NMR time scale. This mirror plane would be coincident with the central core of the molecule and should contain the pyridine ring as well as the C(18), C(19), and C(22) atoms, so that the phenyl ring is divided in equivalent halves. Since the signal due to the *ortho* H (H(20) and H(24)) shows platinum satellites ($J(^{195}Pt,H)$ = 17 Hz), a mechanism involving dissociative processes of the arene–Pt interaction can be ruled out. The signal due to the *meta* H (H(21) and H(23)) overlaps with that corresponding to H(14), which belongs to the pyridine ring.

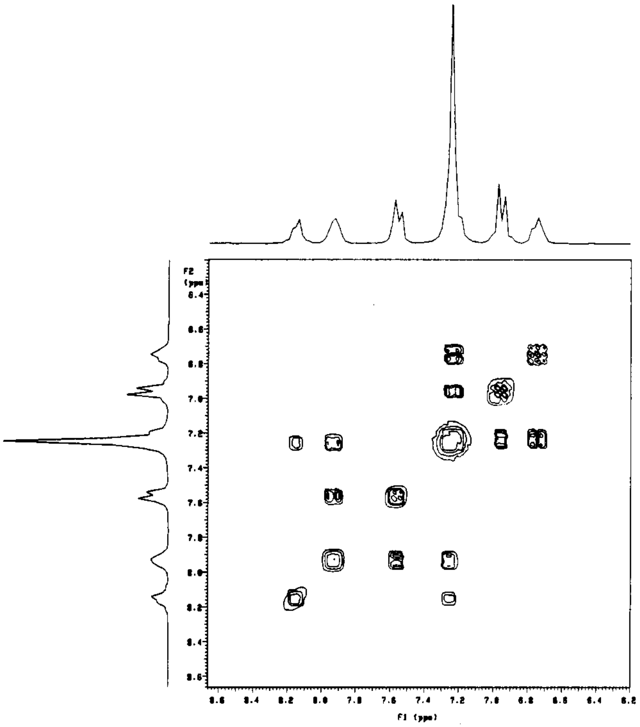

Figure 2. ^{1}H,^{1}H-COSY spectrum of complex 1 (aromatic region only).

The ^{19}F NMR spectrum of 1 at both room and low temperature (−55 °C) shows two *para* F signals, implying the presence of two nonequivalent C_6F_5 groups. The existence of a mirror plane containing the coordination plane of Pt, as suggested above, is also in agreement with the presence of only two *ortho* F signals together with two *meta* F signals.

The signals of the ^{13}C NMR spectrum were assigned in agreement with literature references[10] and confirmed by a ^{13}C,^{1}H-HETCOR experiment (Figure 3). As expected from the previously presented NMR data, the two *ortho* C atoms (C(20) and C(24)) are equivalent (δ 116.26 ppm) and so are the two *meta* C atoms (C(21), C(23): δ 128.87 ppm). The resonance assigned to C(19) appears very close to the broad signal due to C(20) and C(24). Both of these signals suffer a noticeable upfield shift when compared with the corresponding resonances in the free ligand (*ipso* C, δ 139.51; *ortho* C, δ 128.57 ppm) or in [NBu_4][Pt(C_6F_5)$_3$(NC_5H_4-2-$CH_2C_6H_5$)][11] (*ipso* C, δ 139.25; *ortho* C δ 129.81 ppm), a compound without any arene–Pt interaction. Remarkably, for none of these signals do we observe the expected platinum satellites.

Once the dissociative mechanism could be excluded, there remained at least two plausible ways of rationalizing the NMR data: (1) a static, symmetric arene–Pt interaction centered at *ipso* C (η^1 or η^3) or (2) an alternating η^2 coordination (Scheme I, A and A′) involving the previous bonding mode as a transition state (Scheme I, B).

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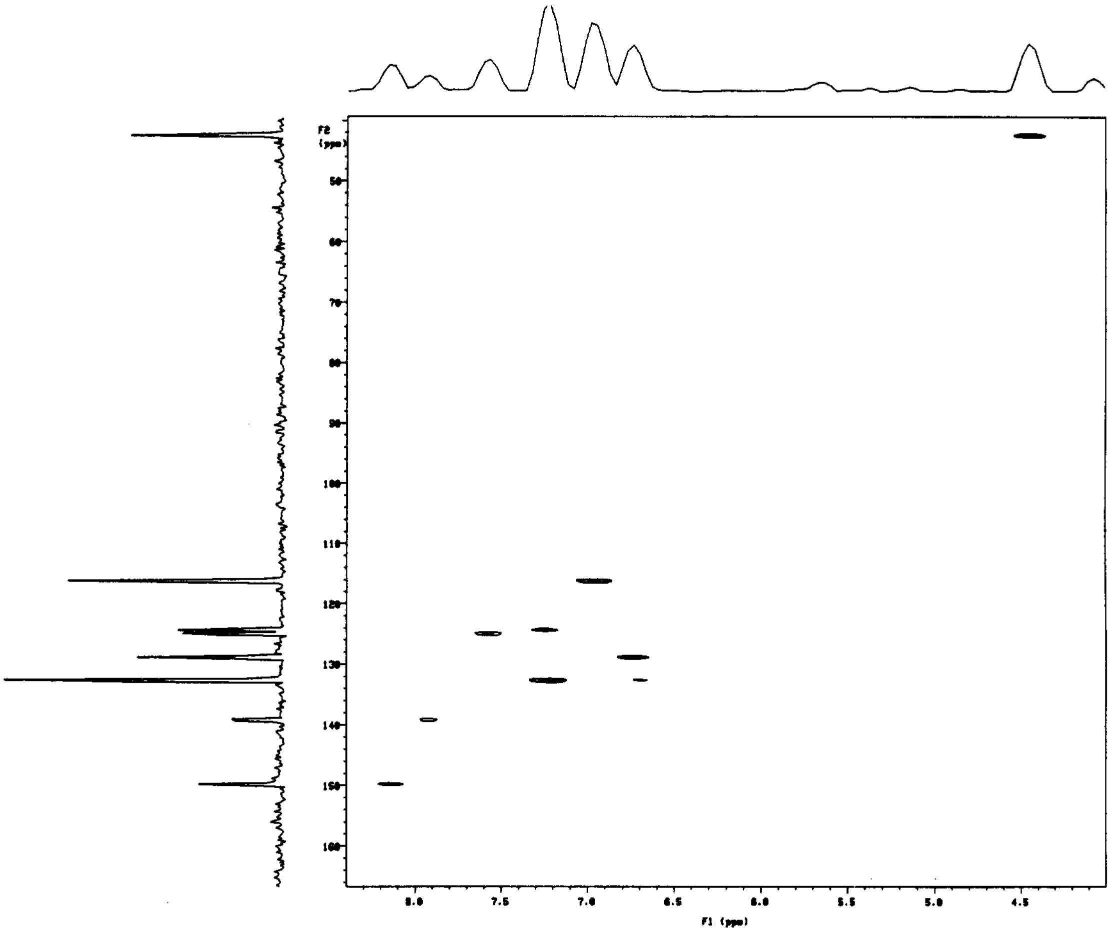
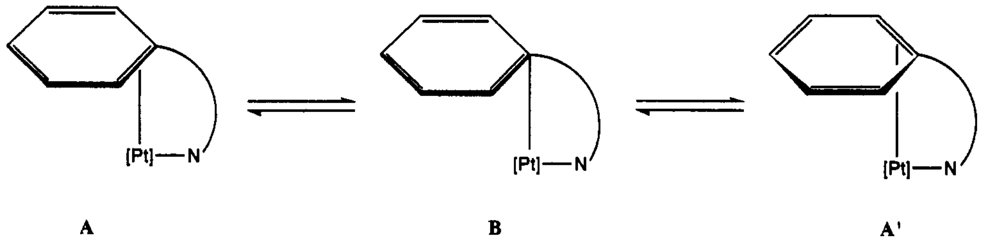


Figure 3. $^{13}C,^{1}H$-HETCOR spectrum of complex 1.

Scheme I



Metallotropic shifts, which are frequently observed in (η^1-cyclopentadienyl)metal complexes,[12] have also been related with the fluxionality of some nonassisted η^2-arene–metal compounds.[13] In our case, however, the arene–Pt interaction is assisted by the N end of the 2-benzylpyridine ligand and, because of this, such a metallotropic shift would be restricted to the *ipso* and *ortho* C atoms. This type of fluxional process was originally suggested to occur through intermediates containing arenium moieties. However, the recent isolation of *cis*-$[Pd(C_6F_5)_2\{(\eta^1\text{-}C_6H_5)CH_2NMe_2\}]$ indicates that this intermediate is strictly not needed. The crystal structure of this complex reveals that the η^1-arene–metal interaction involves neither loss of aromaticity nor change in the hybridization of the *ipso* C. This compound displays obvious relationships with complex 1, and both are prepared by similar methods. It also provides a good model for the above-mentioned static structure of complex 1 in solution.

Aiming to distinguish between these two possibilities (static and dynamic), we recorded the ^1H and ^{19}F NMR spectra of 1 at low temperature (−55 °C).[14] Since no changes could be detected, we still have no experimental evidence to prefer one situation over the other.

(c) Reaction of 1 with CO. As expected, the weak arene–Pt interaction in 1 is released when CO is bubbled at room temperature through a dichloromethane solution of the complex, and from this solution *cis*-$[Pt(C_6F_5)_2(NC_5H_4\text{-}2\text{-}CH_2C_6H_5)(CO)]$ (2) can be obtained. The IR spectrum of 2 shows a sharp absorption, assignable to ν(CO), at 2107 cm^{-1}. The ^1H and ^{13}C NMR spectra of 2 show perceptible changes in the signals assigned to the phenyl ring atoms when compared to the corresponding NMR spectra of 1. The ^1H NMR spectrum shows a complex multiplet centered at 7.35 ppm with the typical

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[14] This lower limit is imposed by the solubility of complex 1 in dichloromethane.

aspect of a phenyl group at frequencies similar to those for the free ligand.

In the ^{13}C NMR spectrum of 2 ([^2H]acetone, room temperature) the *ipso* C and *ortho* C atoms of the benzyl ring appear at 138.08 and 130.01 ppm, respectively. This is a noticeable shift toward higher frequencies compared with the corresponding values observed for these atoms in 1 (see above) and can be taken as evidence that the arene–Pt interaction has in fact been released. It seems sensible, therefore, to conclude that 2-benzylpyridine is acting in 2 as a conventional monodentate N-donor ligand.

The ^1H and ^{19}F NMR spectra of 2 are temperature-dependent and will be discussed together as a function of the temperature. At −80 °C, the ^{19}F NMR spectrum shows two *para* F signals, in accord with the existence of two nonequivalent C_6F_5 groups; furthermore, there appear two sets of three signals with relative integration values 1:1:2, each one corresponding to the *ortho* F and *meta* F substituents. From this pattern it follows that, in contrast with the situation observed in complex 1, the coordination plane in 2 cannot be a mirror plane and the C_6F_5 groups are not free to rotate about the Pt–C bond either. It seems sensible to assume that the lowering of the symmetry is caused by the 2-benzylpyridine ligand, whose important steric requirements could well hinder its rotation around the Pt–N bond. Apparently, the geometry in solution of the central core of the molecule seems to be static on the NMR time scale at this temperature. In accord with this static picture, the ^1H NMR spectrum shows two doublets assignable to the methylene protons of the 2-benzylpyridine ligand. At room temperature, however, these methylene protons are no longer diastereotropic and give rise, in turn, to a single resonance. The ^{19}F NMR spectrum at this temperature shows two *para* F signals, indicating that the C_6F_5 groups keep their chemical nonequivalency. However, only two signals due to the *meta* F substituents are now observed together with two other signals assignable to the *ortho* F atoms, one of which has coalesced. These spectroscopic features can be easily explained by allowing the 2-benzylpyridine ligand to rotate around the Pt–N bond.

Experimental Section

General Methods. C, H, and N analyses were made with a Perkin-Elmer 240B microanalyzer. The IR spectra were recorded over the 4000–200-cm^{-1} range on a Perkin-Elmer 883 spectrophotometer using Nujol mulls between polyethylene sheets. The ^1H, ^{13}C, and ^{19}F NMR spectra were recorded on a Varian XL-200 or a Unity 300 instrument in $CDCl_3$ or HDA solutions. *cis*-[$Pt(C_6F_5)_2(THF)_2$] was prepared as described previously.[15] 2-Benzylpyridine was used as received (Aldrich).

***cis*-[$Pt(C_6F_5)_2(NC_5H_4$-2-$CH_2C_6H_5)$] (1).** To a solution of *cis*-[$Pt(C_6F_5)_2(THF)_2$] (0.300 g, 0.445 mmol) in CH_2Cl_2 (20 mL) at room temperature was added NC_5H_4-2-$CH_2C_6H_5$ (73 μL, 0.445 mmol), and the mixture was stirred at room temperature for 20 min. After evaporation to dryness and addition of *n*-hexane (20 mL) a white solid was isolated and identified as *cis*-[$Pt(C_6F_5)_2(NC_5H_4$-2-$CH_2C_6H_5)$], 90% yield. Anal. Found (calcd): C, 41.11 (41.27); H, 1.48 (1.59); N, 1.95 (2.01). IR (Nujol; ν, cm^{-1}): C_6F_5, 1638 m, 1505 vs, 1066 vs, 961 vs; X-sensitive mode,[16] 811 s, 801 s; NC_5H_4-2-$CH_2C_6H_5$, 1617 s, 1606 m, 1558 m, 1488 s, 1418 s, 1278 s, 1200 m, 1178 m, 1124 m, 1113 m, 1038 m, 1021 m, 933 m, 848 m, 840 m, 761 s, 725 s, 704 s, 637 w, 613 w, 473 w, 439 w.

Table II. Crystallographic Data for *cis*-[$Pt(C_6F_5)_2\{NC_5H_4$-2-$CH_2((1,2$-$\eta^2)$-$C_6H_5)\}$] (1)

formula	$PtF_{10}NC_{24}H_{11}$
mol wt	698.436
cryst syst	monoclinic
space group	$P2_1/n$
a, Å	9.283(1)
b, Å	13.836(1)
c, Å	16.615(1)
β, deg	95.25(1)
V, Å^3	2124.9(3)
Z	4
d_{calc}, g cm^{-3}	2.18
cryst dimens, mm	0.46 × 0.23 × 0.15
μ(Mo Kα), cm^{-1}	67.7
diffractometer	Siemens/STOE AED-2
radiation	Mo Kα (λ_α = 0.710 73 Å)
temp, °C	−50 ± 1
scan method	$2\theta/\omega$
scan range, deg	$4 < 2\theta < 48$
no. of data with $F_o^2 > 2.5\sigma(F_o^2)$	2513
abs cor	ψ scan
no. of refined param	359
R^a	0.028
R_w^b	0.027
GOF	1.0137
largest shift/error	0.001
final diff Fourier max peak, e Å^{-3}	0.77

[a] $R = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$; $w^{-1} = [\sigma^2(|F_o|) + 0.00030|F_o|]$.

Table III. Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Coefficients (Å^2 × 10^3) for *cis*-[$Pt(C_6F_5)_2\{NC_5H_4$-2-$CH_2((1,2$-$\eta^2)$-$C_6H_5)\}$] (1)

	x	*y*	*z*	*U*(eq)[a]
Pt	1529(1)	1714(1)	654(1)	25(1)
C(1)	1124(7)	1345(5)	1777(4)	26(2)
C(2)	1767(7)	1784(5)	2465(4)	29(2)
C(3)	1545(8)	1526(5)	3241(4)	30(3)
C(4)	613(9)	789(6)	3374(4)	35(3)
C(5)	−99(8)	338(6)	2720(5)	39(3)
C(6)	165(8)	625(5)	1953(4)	33(2)
F(2)	2735(5)	2525(3)	2376(3)	38(1)
F(3)	2216(5)	2014(3)	3874(2)	43(2)
F(4)	381(6)	540(4)	4131(3)	57(2)
F(5)	−1017(6)	−388(4)	2824(3)	61(2)
F(6)	−574(5)	130(3)	1339(3)	48(2)
C(7)	251(8)	2860(5)	770(4)	26(2)
C(8)	857(8)	3784(6)	874(4)	32(2)
C(9)	34(8)	4615(5)	937(5)	33(3)
C(10)	−1444(8)	4533(5)	884(5)	34(3)
C(11)	−2087(8)	3649(6)	780(5)	35(3)
C(12)	−1233(8)	2848(5)	727(4)	28(2)
F(8)	2313(5)	3898(3)	928(3)	47(2)
F(9)	684(5)	5474(3)	1044(3)	46(2)
F(10)	−2232(5)	5350(3)	915(3)	55(2)
F(11)	−3534(4)	3570(3)	722(3)	47(2)
F(12)	−1928(4)	1986(3)	630(3)	41(2)
N	1888(6)	2082(4)	−529(3)	25(2)
C(13)	841(9)	2403(5)	−1079(5)	31(3)
C(14)	1081(9)	2571(6)	−1866(5)	39(3)
C(15)	2430(9)	2393(6)	−2117(5)	41(3)
C(16)	3495(9)	2055(6)	−1556(5)	38(3)
C(17)	3220(7)	1908(5)	−768(4)	27(2)
C(18)	4348(9)	1584(7)	−122(5)	37(3)
C(19)	3787(8)	949(5)	527(4)	30(2)
C(20)	2698(8)	261(6)	348(5)	36(3)
C(21)	2462(10)	−456(6)	933(6)	46(3)
C(22)	3243(11)	−457(7)	1661(6)	47(3)
C(23)	4353(10)	226(7)	1831(5)	51(3)
C(24)	4603(9)	910(6)	1285(5)	37(3)

[a] Equivalent isotropic *U*, defined as one-third of the trace of the orthogonalized U_{ij} tensor.

^1H NMR: δ 4.46 (s, 2H, 18-H), 6.74 (t, 1H, 22-H), 6.95 [d, ^3J(H,H) = 7.5 Hz, ^3J(^{195}Pt,H) = 17.0 Hz, 2H, 20-H and 24-H], 7.23 (t, 2H, 21-H and 23-H), 8.13 [d, ^3J(H,H) = 5.6 Hz, ^3J(^{195}Pt,H) = 28.0 Hz,

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1H, 13-H], 7.92 [td, ^3J(H,H) = 7.6 Hz, ^4J(H,H) = 1.6 Hz, 1H, 15-H], 7.56 [d, ^3J(H,H) = 7.9 Hz, 1H, 16-H], 7.27 (t, 1H, 14-H). ^{13}C{^{1}H} NMR: δ 42.62 [C(18)], 116.26 [C(20), C(24)], 116.33 [C(19)], 132.65 [C(21), C(23)], 128.87 [C(22)], 149.77 [^2J(^{195}Pt,C) = 46.0 Hz, C(13)], 124.34 [^3J(^{195}Pt,C) = 29.0 Hz, C(14)], 139.19 [C(15)], 124.98 [C(16)], 161.83 [C(17)]. ^{19}F NMR: δ −117.82 [m_c, ^3J(^{195}Pt,F) = 403.0 Hz, 2F, *o*-F], −119.19 [m_c, ^3J(^{195}Pt,F) = 483.6 Hz, 2F, *o*-F], −163.00 (m_c, 2F, *m*-F), −165.43 (m_c, 2F, *m*-F), −160.09 (m_c, 1F, *p*-F), −162.01 (m_c, 1F, *p*-F).

***cis*-[Pt(C_6F_5)$_2$(NC_5H_4-2-$CH_2C_6H_5$)(CO)] (2).** Through a solution of 1 in CH_2Cl_2 (20 mL) at room temperature was bubbled CO for 10 min. After evaporation to dryness and addition of *n*-hexane (20 mL) a white solid was isolated and identified as *cis*-[Pt(C_6F_5)$_2$(NC_5H_4-2-$CH_2C_6H_5$)(CO)] (2), 91% yield. Anal. Found (calcd): C, 41.33 (41.44); H, 1.53 (1.55); N, 1.93 (1.97). IR (Nujol; ν, cm^{-1}): CO, 2107 vs; C_6F_5, 1637 m, 1507 vs, 1066 vs, 965 vs; X-sensitive mode,[16] 807 s, 799 s; NC_5H_4-2-$CH_2C_6H_5$, 1605 m, 1571 m, 1489 s, 1429 s, 1162 m, 957 vs, 770 s, 695 m, 516 m, 460 m. ^{1}H NMR ([^{2}H]acetone, room temperature): δ 4.78 (s, 2H), 7.31 (m_c, 3H), 7.36 (d, 2H), 766 (d, 2H), 812 (d, 1H), 9.15 [d, ^3J(H,H) = 5.1 Hz, ^3J(^{195}Pt,H) = 29.9 Hz, 1H]. ^{13}C{^{1}H} NMR: δ 44.97 [^2J(^{195}Pt,C) = 34.8 Hz, CH_2], 125.14 [^3J(^{195}Pt,C) = 28.7 Hz, *m*-py], 128.09 (*p*-C_6H_5), 129.16 [^3J(^{195}Pt,C) = 16.5 Hz, *m*-py], 129.78 (*m*-C_6H_5), 130.01 (*o*-C_6H_5), 138.08 (*ipso*-C_6H_5), 141.15 (*p*-py), 153.61 [^2J(^{195}Pt,C) = 12.2 Hz, *o*-py], 163.49 (*o*-py), 171.7 [^1J(^{195}Pt,C) = 1220 Hz, CO]. ^{19}F NMR ($CDCl_3$, room temperature): δ = −117.76 [m_c, ^3J(^{195}Pt,F) = 402.3 Hz, 2F, *o*-F], −119.4 (broad, 2F, *o*-F), −163.73 (m_c, 2F, *m*-F), −164.38 (m_c, 1F, *m*-F), −160.03 (t, 1F, *p*-F), −160.93 (tt, 1F, *p*-F). ^{19}F NMR ([^{2}H]acetone, −80 °C): δ −117.04 [m_c, ^3J(^{195}Pt,F) = 374.8 Hz, 1F, *o*-F], −117.96 [m_c, ^3J(^{195}Pt,F) = 399.6 Hz, 2F, *o*-F], −120.83 [m_c, ^3J(^{195}Pt,F) = 299.0 Hz, 1F, *o*-F], −162.55 (m_c, 1F, *m*-F), −163.08 (m_c, 2F, *m*-F), −163.43 (m_c, 1F, *m*-F), −158.89 (m_c, 1F, *p*-F), −159.82 (m_c, 1F, *p*-F).

Crystal Structure Determination. Crystallographic parameters are given in Table II. Atomic coordinates are presented in Table III. Suitable crystals for X-ray studies were grown by slow evaporation of a $CHCl_3$ solution of 1 at room temperature. Data were collected on a Siemens STOE/AED-2 four-circle diffractometer with an Oxford Cryosystem low-temperature device at −50 °C. The scan range was $4 \le 2\theta \le 48°$. Accurate lattice parameters were determined from accurate positions of 60 reflections ($18 \le 2\theta \le 28°$), including Friedel pairs. The intensities of three standard reflections were checked every 60 min, and no decay was observed. Intensity data were corrected for Lorentz and polarization effects. Absorption correction (10 ψ scans) was applied (rescaled maximum and minimum transmission factors 0.521 and 0.330). The structure was solved by the use of Patterson and Fourier methods. All calculations were carried out with SHELXTL-PLUS.[17] All the H atoms were located from the Fourier difference map and refined with a common thermal parameter (0.034 16 Å^2), leaving the positional coordinates free to move.

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Supplementary Material Available: Tables of atomic positional and equivalent isotropic displacement parameters and bond distances and bond angles for 1 and figures giving NMR spectra for 1 and 2 (10 pages). Ordering information is given on any current masthead page.

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[17] SHELXTL-PLUS: Software Package for the Determination of Crystal Structure, Release 4.0; Siemens Analytical X-Ray Instruments, Inc., Madison, WI, 1990.