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Intramolecular oxidative addition of C–F and C–H bonds to $[Pt(dba)_2]$. Crystal structure of $[PtCl\{Me_2NCH_2CH_2NCH(2,4,5\text{-}C_6HF_3)\}]$

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Abstract

The reactions of compound $[Pt(dba)_2]$ with ligands $RCHNCH_2CH_2NMe_2$ (**1a**–**1f**) in which R is a fluorinated aryl ring produced activation of C–F bonds when two fluorine atoms are present in the *ortho* positions of the aryl ring or activation of C–H bonds for ligands containing only one fluoro substituent in *ortho*. Both C–F and C–H bond activation are favoured by an increase of the degree of fluorination of the ring. Further reaction with lithium halides produced cyclometallated platinum (II) compounds $[PtX(Me_2NCH_2CH_2NCHR)]$ (X = Br, Cl) (**2**) containing a terdentate [C,*N*,*N*′] ligand. The obtained compounds were fully characterized including a structure determination for $[PtCl\{Me_2NCH_2CH_2NCH(2,4,5\text{-}C_6HF_3)\}]$ (**2d**′).

Keywords: Platinum; Cyclometallation; N-donor ligands; C–F bond activation; C–H bond activation

1. Introduction

The bond dissociation energy of a C–F bond is larger than that of any other single bond, thus turning its activation into a subject of salient importance [1]. Several methods have been described for the activation of carbon–fluorine bonds of fluoroaromatic compounds by reaction at transition metal centres [2]. In particular, intramolecular oxidative addition at low valent metal centres such as Ni(0), W(0) and Pt(II) has been reported [3–5]. However, although intermolecular C–F bond activation at Pt(0) has been achieved [6], intramolecular processes involving platinum(0) have not been reported. Recent calculations carried out for intermolecular processes indicated a stronger preference for C–F activation at nickel(0) than at platinum(0) [7].

Following our previous work concerning intramolecular oxidative addition of C–Br and C–Cl bonds of nitrogen donor ligands to the platinum(0) compound $[Pt(dba)_2]$ (dba = dibenzylideneacetone) [8], we now report analogous reactions with fluorinated ligands in order to analyse the ability of this system to produce C–F bond activation. In order to analyse the effect of the degree of fluorination of the aryl group in the reactivity of the C–F bond as well as in the competition between C–F and C–H bond activation, several ligands $RCHNCH_2CH_2NMe_2$ in which R is a fluorinated aryl ring were tested.

2. Results and discussion

2.1. Reactions of the imines with $[Pt(dba)_2]$

Imines **1a**–**1f** were prepared as reported [5] and treated with $[Pt(dba)_2]$ in THF at 50 °C; activation of C–F or C–H bonds was followed by metathesis reaction with either

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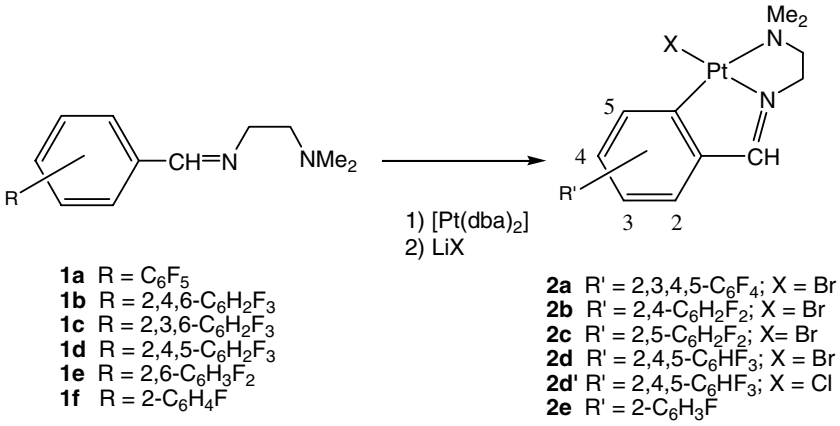

Scheme 1.

lithium bromide or lithium chloride. Cyclometallated compounds containing a terdentate [C,*N*,*N*′] ligand were obtained as brown or dark red solids to which the formula $[PtX(C_6H_xF_{4-x}CHNCH_2CH_2NMe_2)]$ was assigned. According to the obtained results, shown in Scheme 1, activation of a C–F bond took place for ligands **1a**, **1b**, **1c** and **1e** containing two fluorine atoms in the *ortho* positions of the aryl group while ligands containing only one fluoro substituent in *ortho* (**1d** and **1f**) led to activation of a C–H bond. In the case of ligand **1c**, which presents two non-equivalent positions available for C–F bond activation, only that having a fluorine atom in the adjacent position is selectively activated giving one single isomer. As previously reported for analogous systems [4,5], this fact shows that the electron withdrawing effect of the adjacent fluorine atom might be decisive in enhancing the reactivity of C–F bonds. This result contrasts with the previously reported oxidative addition of chloro substituted imines to $[Pt(dba)_2]$. In this case, if the ligand presents two non-equivalent metallation sites, the oxidative addition occurs preferably in the less hindered C–Cl bond [8].

The reactions carried out for ligands **1e** and **1f** involving respectively cleavage of a C–F or of a C–H bond followed by reaction with lithium bromide both led to compound $[PtBr(C_6H_3FCHNCH_2CH_2NMe_2)]$ (**2e**). However, both processes took place with low yields, and, in addition, compound **2e** could not be isolated in a pure form due to its low stability. Previous results [5] indicated that C–F bond activation at platinum(II) could not be achieved for ligands containing only difluoro-substituted aromatic groups such as $2,6\text{-}C_6H_3F_2CHNCH_2CH_2NMe_2$ (**1e**). It has been suggested [6] that the C–F bond activation can be related in thermodynamic terms to the strength of the M–F and M–C(aryl) bonds formed. Taking into account that the strength of the Pt–C bond increases with the increasing electronegativity of the aryl group, C–F bond activation is less favoured for less fluorinated aryl rings. In the present study, using an electron rich platinum(0) substrate, C–F bond activation was achieved for ligand **1e** although with lower yields than those observed for ligands **1a**–**1c**.

On the other hand, the results obtained for ligands **1d** and **1f** indicate that when only one fluorine substituent is present in the *ortho* positions of the aryl group, C–H bond activation is more favoured than C–F bond activation. This result can be related to the higher energy of a C–F versus a C–H bond [1] or, as reported for analogous chloroderivatives [8], the second *ortho* C–F bond might facilitate the intramolecular oxidative addition via an N=CH···F interaction in the intermediate coordination complex, which reinforces the planarity of the ArC=N fragment and brings the C–F bond close to the metal. As observed for activation of C–F bonds, an increase of the degree of fluorination of the ring favours C–H bond activation leading to a better yield for **1d** than for **1f**.

In an attempt to obtain the compounds containing either Pt–F or Pt–H bonds arising, respectively, from intramolecular activation of C–F or C–H bonds the reactions of ligands **1a**–**1e** were carried out in the absence of lithium salts, however these reactions did not produce the desired compounds. The presence of free ligand, dibenzylideneacetone and metallic platinum indicated decomposition processes. For the reaction carried out for ligand **1d**, an hydride resonance was detected at $\delta = -12$ ppm, however the platinum hydrido complex could not be isolated from the reaction mixture. These results are not unexpected since platinum(II) hydrides with nitrogen ligands are unusual [9], and fluorocomplexes of palladium(II) and platinum(II) have been reported to be extremely labile, in particular in the absence of π-acceptor ligands *trans* to the fluoride, and only some *trans*-$[PtFAr(PR_3)_2]$ complexes have been described [10]. Recently a new oxidative fluorination–reductive elimination sequence using XeF_2 has been developed for the synthesis of *cis*-$[PtF_2(PR_3)_2]$ compounds [11].

Since a convenient method to prepare late-transition metal fluoro complexes is the metathesis reaction with silver fluoride [12], the reaction of compound **2d** with AgF in toluene was also tested, however compound **2d** was recovered unaltered.

The new compounds **2a**–**2d** were characterised by elemental analyses, FAB-mass spectra, ^{1}H, ^{19}F and ^{195}Pt NMR, and **2d′** was also characterised crystallographically. In the ^{1}H NMR spectra, the NMe_2 and CH=N resonances were coupled to platinum, showing the coordination of both nitrogen atoms to platinum. The aromatic region of the ^{1}H NMR spectra shows one (**2d** and **2d′**) or two (**2b** and **2c**) resonances, while four (**2a**), three (**2d** and **2d′**) or two (**2b** and **2c**) distinct fluorine signals appear in the ^{19}F NMR spectra. The observed values of the couplings between F–F, F–H and H–H are in the range expected for analogous compounds [5]. In addition, J(F–Pt) couplings are observed and the largest values correspond to the fluoro substituent adjacent to the metallated carbon (F^5) [13]. For **2c**, two fluorine resonances with J(F–Pt) couplings of 116 and 60 Hz were assigned, respectively, to F^5 and F^2, indicating that C–F bond activation of **1c** took place at the position having an adjacent fluorine substituent. The ^{195}Pt NMR spectra show only one signal, the position of which is consistent with the nature of the ligands bound to platinum(II) [14]. The FAB mass spectra of the new complexes show intense signals corresponding to the molecular peak and to the loss of the halide.

2.2. Crystal structure

Suitable crystals of compound **2d′** were grown in acetone solution. The crystal structure is composed of discrete molecules separated by van der Waals distances. The structure is shown in Fig. 1 and selected molecular dimensions are listed in Table 1. A fused [5,5,6] tricyclic system containing a five membered metallacycle, a chelate ring with two nitrogen atoms and the phenyl group results from terdentate [C,*N*,*N′*] coordination of the ligand. A chloro ligand completes the square-planar coordination around the platinum center. The metallacycle is nearly coplanar with the coordination plane, the dihedral angle between the mean planes being 7.7°.

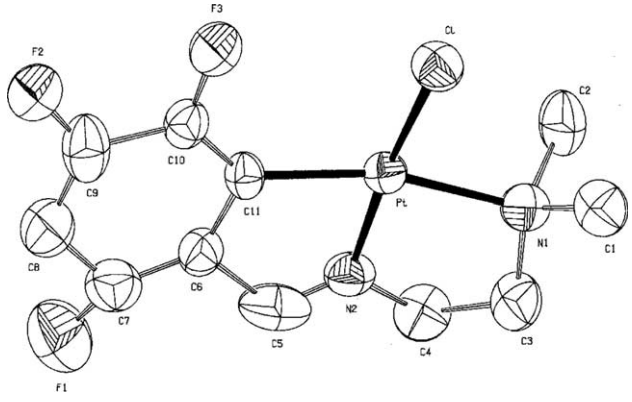

Fig. 1. Molecular structure of compound **2d′**.

Table 1
Selected bond lengths (Å) and angles (°) for compound **2d′** with estimated standard deviations

Pt–N(2)	1.970(14)	Pt–C(11)	1.997(10)
Pt–N(1)	2.135(14)	Pt–Cl	2.296(4)
N(1)–C(3)	1.44(3)	N(2)–C(5)	1.14(3)
N(2)–C(4)	1.37(2)	C(3)–C(4)	1.51(3)
C(5)–C(6)	1.56(3)	C(6)–C(11)	1.42(2)
N(2)–Pt–C(11)	81.9(6)	C(11)–Pt–Cl	101.3(4)
N(2)–Pt–N(1)	81.7(6)	N(1)–Pt–Cl	94.9(4)

Bond lengths and angles are well within the range of values obtained for analogous cyclometallated compounds. In particular, the imine C=N bond lengths lie in the usual range, and Pt-amine distances are larger than Pt-imine distances consistent with the weaker ligating ability of amines for platinum. Most bond angles at platinum are close to the ideal value of 90°, and the smallest angles correspond to the terdentate ligand (N(2)–Pt–C(11) = 81.9(6)° and N(1)–Pt–N(2) = 81.7(6)°).

3. Experimental

3.1. General

NMR spectra were recorded at the Unitat de RMN d′Alt Camp de la Universitat de Barcelona using Varian Gemini 200 (^{1}H, 200 MHz), Bruker 250 (^{195}Pt, 54 MHz) and Varian XL300FT (^{19}F, 282.2 MHz) spectrometers, and referenced to $SiMe_4$ (^{1}H), H_2PtCl_6 in D_2O (^{195}Pt) and $CFCl_3$ (^{19}F). δ values are given in ppm and J values in Hz. Microanalyses were performed at the Servei de Recursos Científics i Tècnics de la Universitat Rovira i Virgili. Mass spectra were performed at the Servei d′Espectrometria de Masses de la Universitat de Barcelona using a VG-Quattro (FAB, NBA) spectrometer.

3.2. Preparation of compounds

Compounds **1a**–**1f** [5] and $[Pt(dba)_2]$ [15] were prepared as reported.

3.2.1. *[PtBr(Me$_2$NCH$_2$CH$_2$NCHC$_6$F$_4$)] (2a)*

Compound (**2a**) was obtained from 0.30 g (0.452×10^{-3} mol) of $[Pt(dba)_2]$ and the equimolar amount (0.12 g) of ligand $C_6F_5CHNCH_2CH_2NMe_2$ (**1a**) in 25 mL of THF. The mixture was stirred at 50 °C under N_2 for 6 h and insoluble materials were filtered off. LiBr (45 mg; 0.518×10^{-3} mol) was added to the

filtrate and the solution was stirred at room temperature for 2 h. The solvent was evaporated in vacuo and the remaining residue was treated with diethylether to yield a brown solid. Yield 106 mg (45%). Anal. Found: C, 25.2; H, 2.2; N, 5.4. Calc. for $C_{11}H_{11}BrF_4N_2Pt$: C, 25.30; H, 2.12; N, 5.36%. FAB-MS, *m/z*: 522 [M], 441 [M–Br]. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.94 [s, J(Pt–H) = 18, 6H, H^a]; 3.17 [t, J(H–H) = 6, 2H, H^b]; 4.12 [t, J(H–H) = 6, J(H–Pt) = 33, 2H, H^c]; 8.72 [s, J(H-Pt) = 141, 1H, H^d]. ^{19}F NMR (282.2 MHz, $CDCl_3$): δ = −119.16 [dd, J(F–Pt) = 143, J(F^5–F^4) = 20, J(F^5–F^2) = 15, F^5]; −138.12 [m, J(F–Pt) ca. 50, J(F^2–F^3) = 20, J(F^2–F^5) = 15, J(F^2–F^4) = 5, F^2]; −145.13 [m, J(F–Pt) = 69, J(F^4–F^3) = J(F^4–F^5) = 20, J(F^4–F^2) = 5, F^4]; −162.99 [t, J(F^3–F^2) = J(F^3–F^4) = 20, F^3]. ^{195}Pt NMR (54 MHz, $CDCl_3$): δ = −3297 [s, br].

Compounds **2b**–**2e** were obtained using the same procedure from 0.30 g (0.452×10^{-3} mol) of [Pt(dba)$_2$] and the equimolar amount of the corresponding ligand (100 mg of **1b**–**1d** and 96 mg of **1e**) and the lithium halide (45 mg of LiBr for **2b**–**2d** and **2e** and 22 mg of LiCl for **2d′**).

3.2.2. [PtBr{Me$_2$NCH$_2$CH$_2$NCH(2,4-C$_6$H$_2$F$_2$)}] (2b)

Yield 101 mg (46%). Anal. Found: C, 26.0; H, 2.7; N, 5.1. Calc. for $C_{11}H_{13}BrF_2N_2Pt \cdot H_2O$: C, 26.20; H, 3.00; N, 5.56%. FAB-MS, *m/z*: 486[M], 406 [M–Br]. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.94 [s, J(Pt–H) = 14, 6H, H^a]; 3.13 [t, J(H–H) = 6, 2H, H^b]; 4.09 [t, J(H–H) = 6, J(H–Pt) = 35, 2H, H^c]; 6.42 [m, J(H^3–F^4) = 11, J(H^3–F^2) = 9, J(H^3–H^5) = 2, 1H, H^3]; 7.42 [dd, J(H^5–F^4) = 9, J(H^5–H^3) = 2, J(H–Pt) = 52, 1H, H^5]; 8.53 [s, J(H–Pt) = 142, 1H, H^d]. ^{19}F NMR (282.2 MHz, $CDCl_3$): δ = −109.69 [dd, J(F–Pt) = 76, J(F^4–H^3) = 12, J(F^4–H^5) = 9, F^4]; −98.86 [m, J(F–Pt) = 43, J(F^2–H^3) = 9, F^2]. ^{195}Pt NMR (54 MHz, $CDCl_3$): δ = −3300 [s, br].

3.2.3. [PtBr{Me$_2$NCH$_2$CH$_2$NCH(2,5-C$_6$H$_2$F$_2$)}] (2c)

Yield 97 mg (44%). Anal. Found: C, 28.0; H, 3.1; N, 5.5. Calc. for $C_{11}H_{13}BrF_2N_2Pt$: C, 27.17; H, 2.69; N, 5.76%. FAB-MS, *m/z*: 486[M], 406 [M–Br]. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.94 [s, J(Pt–H) = 17, 6H, H^a]; 3.16 [t, J(H–H) = 6, 2H, H^b]; 4.08 [t, J(H–H) = 6, J(H–Pt) = 33, 2H, H^c]; 6.64 [td, J(H^3–F^2) = J(H^3–H^4) = 9, J(H^3–F^5) = 4, 1H, H^3]; 6.94 [m, H^4]; 8.62 [s, J(H-Pt) = 141, 1H, H^d]. ^{19}F NMR (282.2 MHz, $CDCl_3$): δ = −101.06 [dd, J(F–Pt) = 116, J(F^5–F^2) = 21, J(F^5–H^4) = 6, F^5]; −121.10 [ddd, J(F–Pt) = 60, J(F^2–F^5) = 21, J(F^2–H^3) = 9, J(F^2–H^4) = 5, F^2]. ^{195}Pt NMR (54 MHz, $CDCl_3$): δ = −3348 [s, br].

3.2.4. [PtBr{Me$_2$NCH$_2$CH$_2$NCH(2,4,5-C$_6$HF$_3$)}] (2d)

Yield 100 mg (44%). Anal. Found: C, 23.4; H, 2.8; N, 5.1. Calc. for $C_{11}H_{12}BrF_3N_2Pt \cdot 3H_2O$: C, 23.67; H, 3.25; N, 5.01%. FAB-MS, *m/z*: 504[M], 424 [M–Br]. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.94 [s, J(Pt–H) = 18, 6H, H^a]; 3.15 [t, J(H–H) = 6, 2H, H^b]; 4.07 [t, J(H–H) = 6, J(H–Pt) = 33, 2H, H^c]; 6.57 [td, J(H^3–F^2) = J(H^3–F^4) = 9, J(H^3–F^5) = 5, 1H, H^3]; 8.63 [s, J(H–Pt) = 141, 1H, H^d]. ^{19}F NMR (282.2 MHz, $CDCl_3$): δ = −118.43 [dt, J(F–Pt) = 74, J(F^2–F^5) = 18, J(F^2–F^4) = J(F^2–H^3) = 9, F^2]; −125.22 [dt, J(F–Pt) = 73, J(F^{4}F^5) = 23, J(F^4–H^3) = J(F^4–F^2) = 9, F^4]; −127.24 [ddd, J(F–Pt) = 144, J(F^{5}F^4) = 23, J(F^5–F^2) = 18, J(F^5–H^3) = 5, F^5]. ^{195}Pt NMR (54 MHz, $CDCl_3$): δ = −3323 [s, br].

3.2.5. 3[PtCl{Me$_2$NCH$_2$CH$_2$NCH(2,4,5-C$_6$HF$_3$)}] (2d′)

Yield 91 mg (44%). Anal. Found: C, 27.2; H, 3.1; N, 5.1. Calc. for $C_{11}H_{12}ClF_3N_2Pt \cdot H_2O$: C, 27.65; H, 2.95; N, 5.86%. FAB-MS, *m/z*: 459[M], 424 [M–Cl]. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.90 [s, J(Pt–H) = 17, 6H, H^a]; 3.18 [t, J(H–H) = 6, 2H, H^b]; 4.06 [t, J(H–H) = 6, J(H–Pt) = 31, 2H, H^c]; 6.56 [td, J(H^3–F^2) = J(H^3–F^4) = 9, J(H^3–F^5) = 6, 1H, H^3]; 8.57 [s, J(H–Pt) = 139, 1H, H^d]. ^{19}F NMR (282.2 MHz, $CDCl_3$): δ = −115.64 [m, F^2]; −123.21 [dt, J(F–Pt) = 90, J(F^{4}F^5) = 24, J(F^4–H^3) = J(F^4–F^2) = 8, F^4]; −129.45 [m, J(F–Pt) = 140, F^5]. ^{195}Pt NMR (54 MHz, $CDCl_3$): δ = −3183 [s, br].

3.2.6. [PtBr{Me$_2$NCH$_2$CH$_2$NCH(2-C$_6$H$_3$F)}] (2e)

Yield 27 mg (13%). FAB-MS, *m/z*: 468[M], 450 [M–Br]. ^{1}H NMR (200 MHz, $CDCl_3$): δ = 2.94 [s, J(Pt–H) = 15, 6H, H^a]; 3.13 [t, J(H–H) = 6, 2H, H^b]; 4.12 [t, J(H–H) = 6, J(H–Pt) = 33, 2H, H^c]; 8.63 [s, J(H–Pt) = 145, 1H, H^d]. ^{19}F NMR (282.2 MHz, $CDCl_3$): δ = −115.20 [dd, J(F–Pt) = 65, J(F^{2}H^3) = 9, J(F^2–H^4) = 6, F^2]. ^{195}Pt NMR (54 MHz, $CDCl_3$): δ = −3529 [s, br].

Table 2
Crystallographic and refinement data for compound **2d′**

Formula	$C_{11}H_{12}ClF_3N_2Pt$
Formula weight	459.77
Temperature (K)	293(2)
Wavelength (Å)	0.71073
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a (Å)	17.4680(10)
b (Å)	6.3400(10)
c (Å)	11.6930(10)
V (Å)^3, Z	1295.0, 4
$d_{calc.}$ (Mg/m^3)	2.358
Absorption coefficient (mm^{-1})	11.060
F(000)	856
Reflections collected/unique	70332/2739
Data/restraint/parameter	2739/0/164
Goodness-of-fit on F^2	1.075
R_1 ($I > 2\sigma(I)$)	0.0635
wR_2 (all data)	0.1639
Peak and hole, e Å^{-3}	0.918 and −0.814

3.3. X-ray structure analysis

Prismatic crystals were selected and mounted on a MAR345 with an image plate detector diffractometer. Intensities were collected with graphite monochromatized Mo Kα radiation. Lorentz polarisation but not absorption corrections were made. The structure was solved by direct methods using SHELXS computer program and refined by the full-matrix least-squares method, with the SHELXL-97 computer program [16] using 2739 reflections (very negative intensities were not assumed). Further details are given in Table 2.

4. Supplementary material

The crystallographic data of compound **2d′** have been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 236373. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033; e-mail:deposit@ccdc.cam.ac.uk or www:http://www.ccdc.cam.ac.uk).

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