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**Two Different Compounds in One Crystal:
di- μ -chlorobis[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]diplatinum(II) and
chloro(N,N-dimethylformamide- κ O)[2-(5-methyl-pyridin-
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Two different compounds in one crystal: di- μ -chlorobis[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]diplatinum(II) and chloro(N,N-dimethylformamide- κ O)[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]platinum(II)

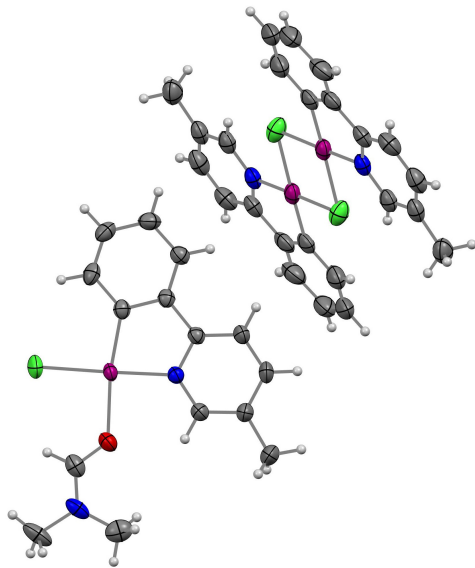
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Abstract: The crystal structures of di- μ -chlorobis[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)] diplatinum(II) (**1**) and chloro(N,N-dimethylformamide- κ O)[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]platinum(II) (**2**) have been determined by means of the X-ray diffraction. Both complexes cocrystallize in the monoclinic space group C2/c with $a = 39.5457(18)$ Å, $b = 7.2482(4)$ Å, $c = 21.2269(10)$ Å and $\beta = 121.460(2)^\circ$. The asymmetric unit consists of half a centrosymmetric compound (complex **1**) and one full independent compound (complex **2**).

Gaphical Abstract

The X-ray structures of co-crystallized di- μ -chlorobis[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]diplatinum(II) (**1**) as starting material and of the derivative chloro(N,N-dimethylformamide- κ O)[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]platinum(II) (**2**) are reported



Keywords: Platinum, cyclometalated, crystal structure, di-chloro bridged

Introduction

For decades, cyclometalated platinum complexes have been the subject of intensive research because of their outstanding luminescence properties [1,2]. Consequently, chemistry has been developed for diverse applications

[3] to include them as potential candidates in the elaboration of OLED [4], as memory devices [5], with NLO properties [6], as catalyst [7], as luminescent probes in biology [8] or as anticancer prodrugs [9,10]. To obtain orthoplatinated compounds, a number of syntheses start from K_2PtCl_4 and the bidentate C^N ligands leading to the dimer precursor $[Pt(\mu-Cl)(C^N)]_2$. The neutral monomeric platinum(II) complexes $Pt(II)(C^N)LCl$ (L and Cl as ligands) are produced by splitting the bridge upon dissolution to give complexes containing a solvent molecule as ancillary ligand or by using unidentate donor ligands [11,12,13]. Depending on the targeted complex, the ancillary ligand may be displaced [14] by a monodentate ligand of interest. In other cases, bidentate ligands L^L can convert the dimer to $Pt(II)(C^N)(L^L)$ [15,16].

In a regular way, when the C^N is a phenylpyridine-type ligand, the neutral monomeric species are formed by dissolution of the dimer in DMSO [11, 12, 17]. RX analyses have showed that the DMSO is coordinated to the platinum atom through the S atom and is *trans* to the N atom of the cyclometalated complex (Scheme 1) [17].

To date, despite attempts by Sakai et al. [18] and although it has been routinely mentioned, the structure of the dimer has never been described. Herein we report the syntheses and structures of chloro(N,N-dimethylformamide- κ O)[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]platinum(II) obtained from the binuclear di- μ -chlorobis[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]diplatinum(II).

Experimental

General

The ligand and complex **1** were synthesized as described in the literature [17]. K_2PtCl_4 (Strem), was purchased and used as received. DMF was of reagent grade. NMR spectra were recorded on a Bruker Avance 500 spectrometer.

Synthesis

Complex **2** was directly obtained by heating complex **1** in DMF at 50°C for 48h. Isolation of the complex was carried out after evaporation of the DMF, precipitation with diethyloxide, and filtration to give a pale green powder. 1H NMR (300, DMF- d_7): δ 8.81 (dd, 1H, $J = 0.8$ Hz, $J_{H-Pt} \approx 42$ Hz, Py6); 8.08-8.14 (m, 2H); 8.02 (dd, 1H, $J = 7$ and 1.8 Hz); 7.74 (dd, 1H, $J = 7.2$ and 2.1Hz), 7.14-7.23 (m, 2H); 3.14 and 2.98 (s, Me of DMF); 2.46 (s, Me). $^{13}C\{^1H\}$ NMR (75.47MHz, DMF- d_7): δ 206.17; 162.48; 146.57; 145.08; 139.64; 134.89; 133.15; 132.03; 128.39; 123.21; 123.19; 118.80; 17.61. Due to the importance of the residual peaks of the non-deuterated DMF, it was not possible to identify the ^{13}C signals from the methyl groups of the DMF ligand.

In order to get crystals, the synthesis was followed as described above, before isolation. After leaving the DMF solution at room temperature for few days, yellow crystals of suitable size and quality for single X-ray diffraction analysis were obtained.

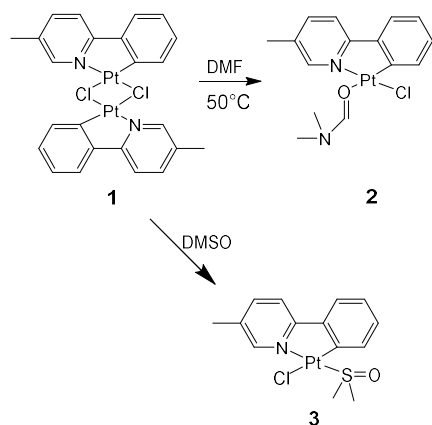
X-ray Structure Determination

Diffraction data were collected at low temperature on a Bruker Kappa APEX II diffractometer using a graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å) and equipped with an Oxford Cryosystems Cryostream cooler device. Phi- and Omega- scans were used. The structure was solved by direct methods with SHELXS 97 [19] and refined by full-matrix least-squares on F^2 [20]. All non-hydrogen atoms were refined with anisotropic displacement parameters. The hydrogen atoms were refined isotropically at calculated positions using a riding model with their isotropic displacement parameters constrained to be equal to 1.5 times the equivalent isotropic displacement parameters of their pivot atoms for terminal carbon sp^3 atoms and 1.2 times for all other carbon atoms. Crystal and experimental data are listed in Table 1.

Results and Discussion

Synthesis

The syntheses of complexes **2** (with DMF) and **3** (with DMSO) are depicted on Scheme 1. For discussion purposes, the synthesis of complex **3** is added.



Scheme 1 Preparation of complexes **2** and **3**.

Depending on the nature of the solvent used, the bridge-splitting does not produce similar complexes. As usually described when using DMSO, the solvent molecule is coordinated to the platinum atom through the sulfur atom and is *trans* to the Pt-N bond [11,17] as depicted on Scheme 1.

Cyclometalated complexes with DMF as ancillary ligand are scarce [18, 21]. The synthesis of complex **2** was carried out by gentle heating of complex **1** in DMF for two days. It was isolated after elimination of the excess of solvent and precipitation with ether. The X-ray analysis revealed a κ O-DMF is coordination to the platinum atom and is *vide infra*.

X-ray Structure

The starting complex di- μ -chlorobis[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]diplatinum(II) **1** and the final complex chloro(N,N-dimethylformamide- κ O)[2-(5-methyl-pyridin-2-yl- κ N)phenyl- κ C(1)]platinum(II) **2** cocrystallize in DMF.

Views of complexes **1** and **2** are shown on Fig. 1 and 2 respectively and selected bond lengths and angles are given in Table 2. In both complexes the platinum atom is located in a square-planar environment. The ligand coordinated to the platinum shows a certain degree of planarity: the dihedral angles between the two aromatics rings of the ligand in complexes **1** and **2** are 5.95° and 3.33°, respectively.

The Pt-C (1.981(9) - 1.954(7)Å), Pt-N (1.986(7) - 2.002(6) Å) and Pt-Cl (2.447(2)/ 2.317(2) - 2.298(2) Å) distances are in accordance with bond lengths reported in other analogous platinum complexes. Analysis of the molecular packing shows the presence of rows of **1** which are interleaved by rows of **2** (Fig. 3). Viewed along the crystallographic *c*-axis, the structure is composed of stacks of **1** and **2** complexes (Fig. 4).

In complex **1** we can notice that both Pt-Cl bonds are not similar: indeed, the bond length of Pt2-Cl2i is slightly shorter (2.317(2) Å) than the Pt2-Cl2 bond (2.447(2) Å), revealing the *trans* influence of the σ -carbon. This observation was previously reported on chloro-bridge dimers[22-25].

Thanks to the methyl substituent on the pyridine ring, the positions of the C and the N atoms are determined unambiguously. Examination of the structure reveals a κ O-DMF coordination *trans* to the Pt-C. Only one structure of DMF coordinated to a platinum atom with this type of ligand has been published [18]. The authors described the cyclometalated platinum complex with phenylpyridine (ppy) as ligand. The locations of N(ppy) and C(ppy) donors were determined by examining their thermal parameters.

Starting from the same binuclear species, depending of which solvent is used for the following step, a striking difference between the complexes obtained is observed, as depicted on scheme 1. When using DMSO, the solvent is coordinated through the S atom, *trans* to the Pt-N bond, as with DMF the coordination of the solvent occurs by the oxygen atom on the Pt atom *trans* to the Pt-C bond.

Table 1 Crystal data, data collection and structure refinement of **1** and **2**

| | |
|---|---|
| Chemical formula | C ₂₄ H ₂₀ Cl ₂ N ₂ Pt ₂ ·2(C ₁₅ H ₁₇ ClN ₂ OPt) |
| Formula weight | 1741.15 |
| Crystal system | Monoclinic |
| Space group | C 2/c |
| T (K) | 180(2) |
| <i>a</i> (Å) | 39.5457(18) |
| <i>b</i> (Å) | 7.2482(4) |
| <i>c</i> (Å) | 21.2269(10) |
| β (°) | 121.460(2) |
| <i>V</i> (Å ³) | 5190.0(5) |
| <i>Z</i> | 4 |
| ρ_{calc} (gcm ⁻³) | 2.228 |
| <i>F</i> (000) | 3264 |
| μ (mm ⁻¹) | 11.002 |
| Crystal size (mm) | 0,12 x 0,1 x 0,06 |
| θ range (°) | 1.92-25.35 |
| <i>hkl</i> range | -47 ≤ <i>h</i> ≤ 47 -8 ≤ <i>k</i> ≤ 8 -25 ≤ <i>l</i> ≤ 25 |
| Reflections collected/unique | 42024 / 4757 |
| <i>R</i> _{int} | 0.0233 |
| Number of parameters | 320 |
| <i>R</i> ₁ [<i>I</i> > 2 σ (<i>I</i>)] ^a | 0.0338 |
| <i>wR</i> ² [<i>I</i> > 2 σ (<i>I</i>)] ^b | 0.0813 |
| <i>R</i> ₁ [all data] | 0.0382 |
| <i>wR</i> ² [all data] | 0.0841 |
| Goodness of fit | 1.191 |
| $\Delta\rho_{\text{min}}/\Delta\rho_{\text{max}}$ (eÅ ⁻³) | 2.72 / -2.01 |

$$^a R_1 = \frac{\sum ||F_o| - |F_c||}{\sum |F_o|} \quad ^b wR_2 = \frac{[\sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2]]^{1/2}}$$

In Table 3 are gathered selected bonds of complexes **2**, **3** and of the complex described by Sakai [18]. At a first glance, structures of complex **2** and of Sakai's one are similar, with slightly longer bonds in the latter. The Pt-O bond length in complex **2** is 2.141(5) Å, longer than those reported for the Pt-O distances *trans* to a pyridyl donor (e.g. 2.051(6) Å)[26] as previously observed [18]. When comparing the bond lengths of Pt-Cl in complex **2** (2.298(2) Å) and in complex **3** (2.4054(10) Å) we can notice that it is longer (0.107 Å) when located *trans* to the carbon atom. All these observations highlight the strong *trans* effect of the σ -carbon, as previously mentioned. However, the bond lengths of the Pt-N in both complexes are similar regardless the nature of the atom *trans* to this bond: Pt-Cl for complex **2** and Pt-S for complex **3**.

Deacon and Edwards have studied the mechanism of addition of unidentate ligands to cycloplatinated complexes [11] by a systematic investigation of the bridge cleavage. In fact in the dimeric complex, due to the *trans* effect of the σ -carbon, the Pt-Cl bond *trans* to the aryl substituent should be cleaved first leading to the isomer with the halogen *cis* to the carbon ligand, as observed with DMF in complex **2**. In complex **3** the coordination of the DMSO *trans* to the Pt-N bond, results from a rearrangement to the more stable configuration (through an associative mechanism via a five-coordinate transition state or intermediate).

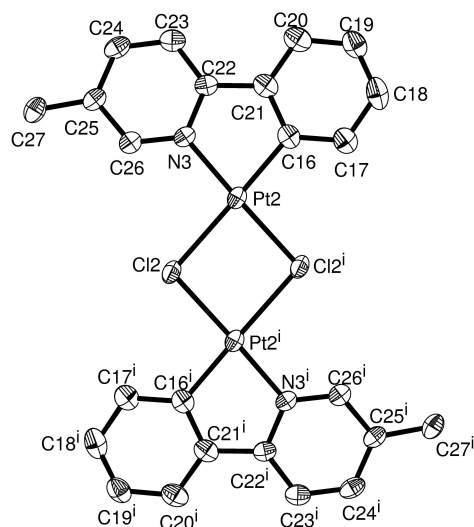


Fig. 1 Molecular structure of complex **1** with atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level. All hydrogen atoms are omitted. [Symmetry code : (i) -x,-y,1-z]

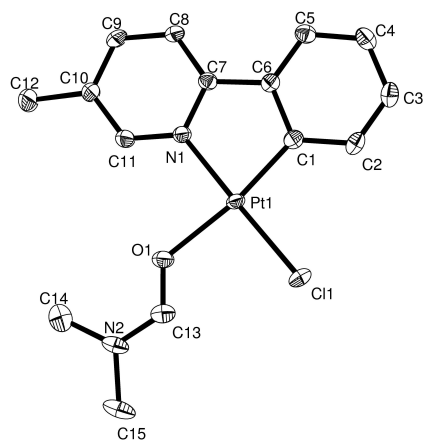


Fig. 2 Molecular structure of complex **2** with atom labeling scheme. Thermal ellipsoids are drawn at the 30% probability level. All hydrogen atoms are omitted.

Therefore it should be pointed out that further substitution of the DMF ancillary ligand by molecules of biological interest may lead to cyclometalated complexes not yet explored, as the usual starting complex is complex **3** to date [27, 28].

Table 2 Selected bond lengths (Å) and angles (°) for complexes **1** and **2**

| 1 | |
|--------------------------|------------|
| Pt2-C16 | 1.981(9) |
| Pt2-N3 | 1.986(7) |
| Pt2-Cl2 | 2.447(2) |
| Pt2-Cl2 ⁱ | 2.317(2) |
| C16-Pt2-N3 | 80.9(3) |
| C16-Pt2-Cl2 ⁱ | 97.3(3) |
| C16-Pt2-Cl2 | 177.4(2) |
| N3-Pt2-Cl2 ⁱ | 178.2(2) |
| 2 | |
| Pt1-C1 | 1.954(7) |
| Pt1-N1 | 2.002(6) |
| Pt1-Cl1 | 2.298(2) |
| Pt1-O1 | 2.141(5) |
| C1-Pt1-N1 | 81.9(3) |
| N1-Pt1-O1 | 89.8(2) |
| C1-Pt1-O1 | 171.7(2) |
| N1-Pt1-Cl1 | 178.46(17) |

Symmetry code : (i) -x,-y,1-z

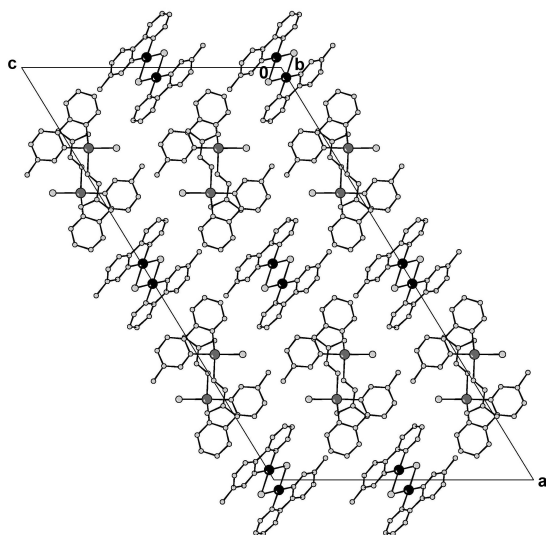


Fig. 3 View of the crystal packing along the *b*-axis. All hydrogen atoms are omitted. For clarity, the platinum atom of **1** is represented as black spheres and the platinum atom of **2** as grey spheres.

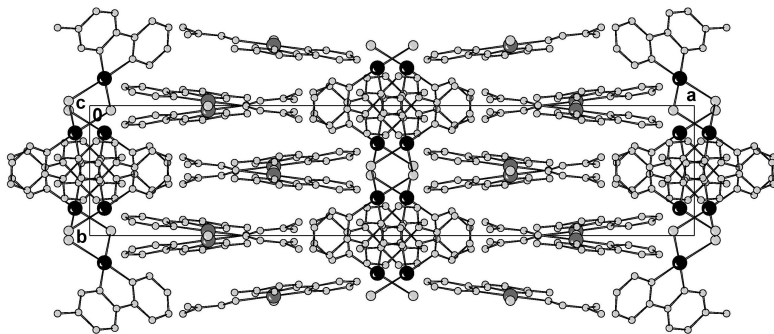


Fig. 4 View of the crystal packing along the *c*-axis. All hydrogen atoms are omitted. For clarity, the platinum atom of **1** is represented by black spheres and the platinum atom of **2** by grey spheres.

Table 3 Selected bond lengths (Å) for complexes **2**, **3** and from ref [18].

| | 2 | [18] | 3 |
|-------|----------|--------------------|------------|
| Pt-C | 1.954(7) | 1.958(7); 1.974(8) | 2.013(4) |
| Pt-N | 2.002(6) | 2.005(6); 2.010(6) | 2.068(3) |
| Pt-Cl | 2.298(2) | 2.305(2); 2.313(2) | 2.4054(10) |
| Pt-O | 2.141(5) | 2.158(5); 2.149(5) | - |
| Pt-S | - | - | 2.2219(10) |

Conclusions

We were able to characterize the routinely mentioned dichloro bridge dinuclear cyclometalated platinum complex with a phenylpyridine-type ligand by X-ray analysis and the resulting mononuclear species after bridge-splitting with DMF. Since the number of cyclometalated platinum complexes with DMF as ancillary ligand are limited, the structural insight achieved from this study provides impulse for further exploration of this complex as starting material to produce new compounds in medicinal chemistry.

Supplementary Material

CCDC-2206545 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Center via www.ccdc.cam.ac.uk/data_request/cif.

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Compliance with Ethical Standards

Conflict of Interest There is no conflict of interest/competing interests to declare.

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