J Chem Crystallogr (2010) 40:831–836
DOI 10.1007/s10870-010-9749-z

ORIGINAL PAPER

Synthesis and Characterization of a Novel Unsymmetrical Tetradentate Schiff Base Complex of Zinc(II) Derived from *N*,*N*′-bis (5-Bromosalicylidene) 2,3-Diaminopyridine (H_2L): Crystal Structure of [Zn(II)L]Pyridine

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Received: 8 April 2008 / Accepted: 16 April 2010 / Published online: 5 May 2010

Abstract Yellow crystals of [Zn(*N*,*N*′-bis (5-bromosalicylidene) 2,3-diaminopyridine)] Pyridine, $[Zn(C_{19}H_{11}Br_2N_3O_2)]$ C_5H_5N, have been obtained and characterized by elemental analysis, FT-IR, UV–Vis, ^{1}H NMR spectroscopy and MALDI-TOF mass spectrometry. The structure has been determined by single crystal X-ray crystallography. The complex crystallizes in the triclinic space group P-1 with two molecules in the asymmetric unit and with unit cell dimensions $a = 8.0103(3)$ Å, $b = 12.8340(5)$ Å, $c = 22.3682(11)$ Å, $\alpha = 91.715(2)°$, $\beta = 93.918(2)°$, $\gamma = 103.708(2)°$. $V = 2226.30(16)$ Å^3, $Z = 4$, $R_1 = 0.0780$ and $wR_2 = 0.1192$. X-ray structure determination revealed that the complex consists of a $[Zn(C_{19}H_{11}Br_2N_3O_2)]$ moiety with a coordinated pyridine molecule on the metallic centre. The zinc atom adopts a distorted square pyramidal geometry as it is shown in the crystal structure.

Keywords Unsymmetrical Schiff base · Zinc(II) complex · Crystal structure · NMR-ROESY

Introduction

The synthesis of nonporphyrinic ligands as Schiff base complexes containing salen chelates with two oxygen and two nitrogen donor atoms (NNOO) have attracted considerable attention during the last decades owing to the ease of their synthesis and their structural versatility associated to their diverse applications. This class of complexes obtained from transition and non-transition metals with Schiff base ligands has emerged as of very promising materials for their use as efficient catalysts [1–3], materials [4, 5], biological sensors [6–20], antibacterial, antifungal [21] and also as anticancer drugs [22, 23]. For example, in catalysis and electrocatalysis, the Mn(III)- or Ni(II)-Schiff base complexes are currently used as catalysts in the epoxidation or oxidation reactions [24–30]. However, these compounds namely those of zinc seem to be efficient materials in the optoelectronic uses due to their outstanding photo- (PL) and electroluminescence (EL) properties particularly those of zinc [31–38] since, the first work reported by Vanslyke et al. [39] on the organic light-emitting devices (OLEDs) materials. Taking into account the existing extensive investigation of symmetrical tetradentate Schiff base complexes, our efforts have been focused on non-symmetrical species that have drawn relatively less attention [40–44]. More recently, we have described some tetradentate unsymmetrical Schiff base complexes [45, 46] where the bridging moiety positioning the two salicylaldehyde derivatives was 2,3-diaminophenol whereas in the present case, the chosen spacer is 2,3-diaminopyridine.

Pyridine was introduced as a general potential pH sensitive group, and also due to the nitrogen affinity for zinc(II). On the long term, this study is also motivated by further synthesis of new Schiff base structures containing electropolymerizable units as pyrrole, thiophene or aniline in order to use them as monomers to elaborate modified electrodes or electronic devices using quaternization of the pyridine moiety as a functionalization tool. In this work, the d^{10} electronic configuration of zinc(II) allows the

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formation of coordination complexes exhibiting a wide range of symmetries and various coordination numbers 4, 5 and 6 [47–49]. Thus, the ligand (H_2L) built on a pyridine spacer afforded a five coordinated complex, which was identified and characterized by spectroscopic methods such as UV–Vis, IR, ^1H NMR and X-rays studies. The crystal structure revealed that the four in-plane coordination sites are occupied by the salen moiety, whereas an apical fifth position is occupied by a pyridine ligand.

Experimental

Materials and Measurements

All preparations were carried out in reagent grade solvents and all commercially available reagents (Aldrich or Merck) were used without any further purification. The manipulations were carried out under inert atmosphere (N_2) following conventional procedures.

The melting points for the ligand $\mathbf{H_2L}$ and its complex **Zn(II)L** were determined with an Electro-thermal 9100 digital apparatus and are uncorrected. Infrared spectra were recorded using KBr pellets on a Perkin Elmer FT-IR Paragon 1000 spectrophotometer. Electronic spectra were performed on a Unicam UV-300 spectrophotometer using DMF as solvent. Elemental analyses were carried out on an Elementar-Vario EL III CHNSO analyser (Institut de Chimie de Strasbourg—France). ^1H-NMR spectra were recorded on a Bruker Avance 500 MHz spectrometer using DMSO-d_6 as solvent and tetramethylsilane (TMS) as internal standard. Mass spectra of ligand $\mathbf{H_2L}$ and its complex **Zn(II)L** were obtained on a Bruker Daltonics Flex Analysis spectrometer with MALDI-TOF procedure using ditranol as matrix, at the "Institut de Chimie de Strasbourg—France".

Synthesis of the Schiff Base Ligand H_2L

The unsymmetrical [N_2O_2] tetradentate Schiff base, 2,3-bis(5-bromosalicylidene) diaminopyridine was prepared following literature methods [45]. To a MeOH solution (15 mL) of 5-bromosalicylaldehyde (0.244 g, 2 mmol) was slowly added a MeOH solution (5 mL) of 2,3-diaminopyridine (0.109 g, 1 mmol). The mixture was refluxed with constant stirring under nitrogen atmosphere for 2 h to yield an abundant orange precipitate that was collected by filtration. The product was washed with methanol (3 × 5 mL) then with diethyl ether (3 × 5 mL) and dried under vacuum for 4 h. Yield: 73%, mp: 255 °C. Analysis calculated for $C_{19}H_{13}O_2N_3Br_2$: C, 48.03; H, 2.76; N, 8.84%; found: C, 47.70; H, 2.92; N, 8.86%. Selected IR data (KBr pellets, υ cm^{-1}): 3445 (O–H), 1625 (C=N), 1607 (C=C), 1271 (C–O); UV–Vis: DMF, λ nm, [$\varepsilon \times 10^{-4}$ M^{-1} cm^{-1}]: 267 [3.114] 346 [1.978], 482 [0.973]; ^1H-NMR: (DMSO-d_6, δ ppm): 13.38 (s, OH), 12.79 (s, OH), 9.47 (s, CH=N), 8.57 (s, CH=N), 6.94–7.63 (m, ArH); MS (MALDI-TOF, ditranol): $M^+ = 475.94$.

Synthesis of the Complex Zn(II)L

9.17 mg (0.5 mmol) of $Zn(OAc)_2$ were dissolved in MeOH (10 mL). This solution was added dropwise to a stirred methanol solution (5 mL) containing 237 mg (0.5 mmol) of Schiff base ligand H_2L. The solution turned yellow and a solid precipitated almost immediately. The mixture was refluxed and stirred for 1 h under nitrogen atmosphere. The precipitate was collected by filtration, and washed successively with methanol (3 × 5 mL) and diethyl ether (3 × 5 mL), the product was then recrystallized from DMSO-heptane and dried under vacuum overnight. Yield: 71%; mp > 300 °C. Analysis calculated for $C_{19}H_{11}O_2N_3Br_2Zn$. DMSO: C, 40.90; H, 2.78; N, 6.81%; found: C, 40.19; H, 2.72; N, 7.57%; IR (KBr pellets, υ cm^{-1}): 3439 (O–H), 1610 (C=N), 1563 (C=C), 1301 (C–O); UV–Vis: DMF, λ nm, [$\varepsilon \times 10^{-4}$ M^{-1} cm^{-1}]: 268 [2.594], 309 [1.930], 421 [2.562], 470 [1.412]; ^1H-NMR: (DMSO-d_6, δ ppm): 9.43 (s, H11), 9.10 (s, H10), 6.71–8.50 (m, ArH1-9); MS (MALDI-TOF, ditranol): $M^+ = 537.83$.

X-ray Crystallography

Single crystals of **Zn(II)L** complex were grown by slow layer diffusion of pyridine into a MeOH solution at room temperature. A yellow plate single crystal of dimensions 0.20 mm × 0.15 mm × 0.10 mm suitable for X-ray analysis was used for data collection at 173(2) K on a Nonius Kappa-CCD diffractometer equipped with an Oxford Cryosystem liquid N_2 device, using graphite-monochromated Mo K_α ($\lambda = 0.71073$ Å) radiation. A semi-empirical absorption correction was applied using the MULscanABS routine in PLATON [50]. Transmission factors: $T_{\min}/T_{\max} = 0.45355/0.55121$. Atomic scattering factors and anomalous dispersion constants were taken from the International Tables for Crystallography, Volume C [51].The structure was solved by direct methods using the SHELXS-97 and SHELXL-97 programs and refined on F^2 by the full-matrix least-squares methods. All of the non-hydrogen atoms were refined anisotropically and the hydrogen atoms were included in geometric positions but not refined.

Results and Discussion

Crystal Structure Description of the Complex

The main crystal parameters are reported in Table 1, selected bond distances and angles are given in Table 2 and geometrical hydrogen bonds are given in Table 3. An ORTEP view of the molecule with the atom- numbering scheme is shown in Fig. 1.

The asymmetric unit comprises two independent mononuclear zinc(II) complexes that exhibit very small differences. The complex crystallizes in the triclinic space group P-1. Zn atom is coordinated by the pairs of N and O atoms of the Schiff base and by N atom of a pyridine molecule. The molecule structure features the zinc atom in a five-coordinate pyramidal geometry forming two–six-membered chelate rings. The bond angles subtended at the Zn atom, ranged from 98.2(2)° to 105.2(2)° for Zn1 atom, indicate a distorted square pyramidal geometry. All angles that do not involve the Zn atom are equal to 120° (with minor variations) showing sp^2 hybridization of the corresponding carbon atoms.

Table 1 Crystal data and refinement parameters for the complex

Molecular formula moiety	$C_{24}H_{16}Br_2N_4O_2Zn$
Molecular weight	617.60
Temperature (K)	173(2)
Radiation λ	Mo K_α (0.71073 Å)
Crystal system	Triclinic
Space group	P-1
a/Å	8.0103(3)
b/Å	12.8340(5)
c/Å	22.3682(11)
α°	91.715(2)
β°	93.918(2)
γ°	103.708(2)
V/Å^3	2226.30(16)
Z	4
D_{calc} (g cm^{-3})	1.841
Crystal size (mm)	0.20 × 0.15 × 0.10
Crystal description	Plate
Crystal colour	Yellow
Absorption coefficient (mm^{-1})	4.723
Absorption correction T_{min} and T_{max}	0.45355 and 0.55121
F(000)	1216
Reflections collected/unique	23533/10101 [$R_{int} = 0.0816$]
Range/indices (h, k, l)	−10, 10; −16, 16; −28, 24
θ limit	1.64–27.43
No. of observed data, $I > 2\sigma(I)$	6725
No. of variables	595
No. of restraints	0
Goodness of fit on F^2	1.121
Largest diff. peak and hole (e Å^{-3})	1.006 and −1.365
R_1, wR_2 [$I \geq 2\sigma(I)$][a]	0.0780, 0.1192
R_1, wR_2 (all data)[a]	0.1309, 0.1331

[a] $R_1 = \sum||F_0| - |F_c||/\sum|F_0|$, $wR_2 = [\sum w(F_0^2 - F_c^2)^2/\sum w(F_0^2)^2]^{1/2}$, $w = 1/[\sigma^2(F_0^2) + (0.0254P)^2 + 6.7173P]$, $P = (F_0^2 + 2F_c^2)/3$

Table 2 Selected bond lengths (Å) and angles (°) for the complex

Bond distances			
N2–Zn1	2.125(5)	N6–Zn2	2.127(5)
N3–Zn1	2.091(5)	N7–Zn2	2.090(5)
N4–Zn1	2.094(5)	N8–Zn2	2.085(5)
O1–Zn1	1.967(4)	O3–Zn2	1.971(4)
O2–Zn1	1.986(4)	O4–Zn2	1.983(4)
Bond angles			
O1–Zn1–O2	96.10(17)	O3–Zn2–O4	97.21(17)
O1–Zn1–N3	151.39(18)	O3–Zn2–N7	153.42(19)
O2–Zn1–N3	89.59(18)	O4–Zn2–N7	89.11(18)
O1–Zn1–N4	101.53(19)	O3–Zn2–N8	97.31(19)
O2–Zn1–N4	98.94(19)	O4–Zn2–N8	100.9(2)
N3–Zn1–N4	105.2(2)	N7–Zn2–N8	106.85(19)
O1–Zn1–N2	87.97(18)	O3–Zn2–N6	88.50(18)
O2–Zn1–N2	161.17(19)	O4–Zn2–N6	159.65(18)
N4–Zn1–N2	98.2(2)	N8–Zn2–N6	97.7(2)
N3–Zn1–N2	78.46(19)	N7–Zn2–N6	77.59(19)

Table 3 Geometrical parameters for hydrogen bonds

D–H···A	d(H···A) (Å)	d(D···A) (Å)	<(D–H···A) (°)
C(27)–H(27)···O(1)	2.401	3.336	168.01
C(26)–H(26)···O(2)	2.674	3.450	139.46

The Zn1 center lies 0.385 Å (Zn2: 0.373 Å) above the N_2O_2 coordination plane, the atoms N2, N3, O1 and O2 define the basal plane and the pyridine molecule occupies the apical position. The bond lengths related to the Zn atom are comparable to the values observed in other similar Schiff base zinc(II) complexes [52, 53]. The Zn1–N4(pyridine) distance (2.094(5) Å) is also comparable to those of Zn1–N2(ligand) and Zn1–N3(ligand) (2.125(5) and 2.091(5) Å) of the analogous coordination patterns [54, 55]. Neighboring molecules in the asymmetric unit are linked to each other with short C–H···O interactions to form a dimer complex (Table 3).

The unit cell holds two pairs of molecules (two dimers) as it is reported for similar iron complex [56] with dibrominated ligand [57]. The packing along the a axis (Fig. 2) shows that the molecules adopt a zigzag form and the neighboring molecules lie in opposite directions regarding to the apical pyridine molecules in order to minimize the steric effects.

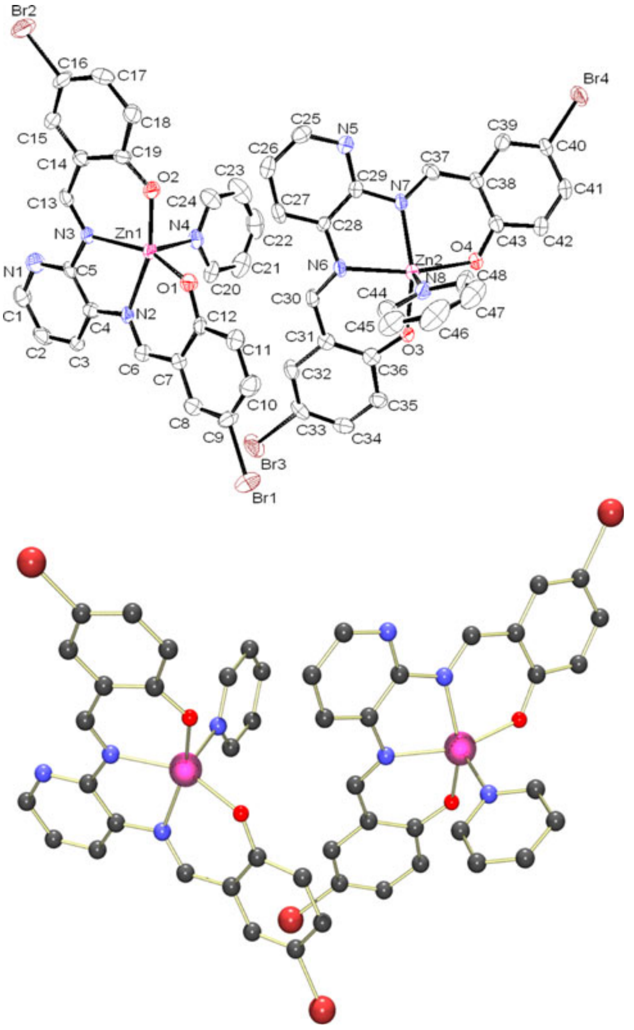

Fig. 1 The molecular structure of the complex (two molecules in the asymmetric unit), showing the atomic numbering scheme. Thermal ellipsoids are drawn at 50% probability. Hydrogen bonds are omitted for clarity

Molecular Formula

Both the complex and the ligand are stable in air. They are much more soluble in polar organic solvents, such as DMSO, DMF and pyridine, than in apolar or water. The elemental analysis of the synthesized compounds were in good agreement with the proposed chemical formulae for H_2L and Zn(II)L. However, the complex shows a ligand–metal stoichiometric ratio of 1:1 affording mononuclear compound.

MALDI-TOF spectra of the Schiff base and its corresponding complex show the molecular ion peaks at 475.94 and 537.83 respectively and the expected bromine isotope patterns supported by typical distribution of dibrominated compounds [58].

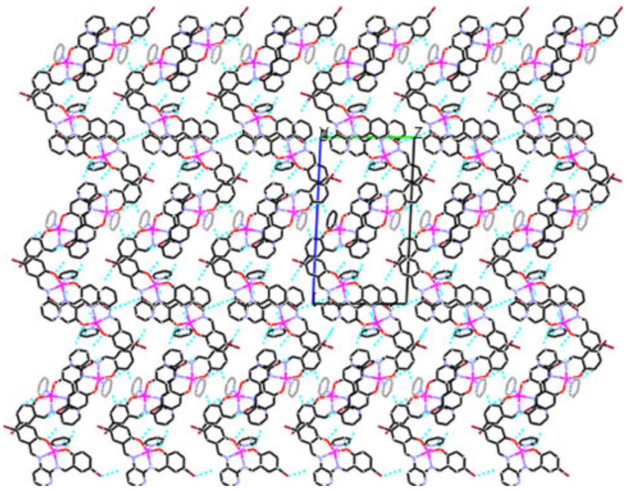

Fig. 2 Molecular packing of the complex along the *a* axis with the unit cell and the short contacts. Hydrogen bonds are not shown

Spectral Characterisation of the Ligand and the Complex

Infrared Spectra

The IR spectra in KBr discs of **H_2L** and **Zn(II)L**, recorded in the region of 4,000–600 cm^{-1}, contain all the characteristic bands of the coordinated tetradentate ligand. The IR spectrum of the complex, compared to its ligand, shows that the υ(C=N) band appearing at 1,625 cm^{-1} is shifted to lower energy by 15 cm^{-1}, indicating that the ligand is coordinated to the zinc metal through the nitrogen atoms of the azomethine groups. In addition, the stretching vibration of C–O, υ(C–O/phenolate), appears at 1,301 cm^{-1} in the ligand form whereas in the complex of **Zn(II)L** it shifted to higher energy by 30 cm^{-1} suggesting that, in this case, the metallic centre causes a drastic increase of the electronic density in the vicinity of the oxygen atoms. This behavior can be interpreted as a result of coordination of both deprotonated oxygens of phenolic groups to the zinc metal center giving an N_2O_2 tetracoordinated complex [59, 60].

Electronic Spectra

The electronic spectra of the ligand and the complex carried out in DMF and recorded in the region of 800–200 nm, show only intraligand or charge transfer bands. The electronic absorption spectrum of the free Schiff base ligand exhibits intense bands centred at 267, 346 and 482 nm. The intense band in high-energy region of the spectrum (267 nm) is related to $\pi \rightarrow \pi^*$ transitions of phenyl rings [61]. The two other bands can be assigned respectively to $\pi \rightarrow \pi^*$ transitions associated with azomethine (N=C) chromophore [62] and to the azopyridine moiety (N-Pyr.).

The corresponding **Zn(II)L** complex exhibits mainly the absorption bands of the ligand which are slightly red shifted. The absorption band appearing at 268 nm coincides with that observed in the free ligand whereas the other bands are rather shifted to higher energy when compared to those of **H_2L**. A shoulder appearing at 470 nm is attributable to metal–ligand d → π^* charge transfer transitions (MLCT).

^{1}H-NMR

^{1}H-NMR and 2D ^{1}H–^{1}H ROESY spectra were recorded and analyzed to support the structure of the zinc complex shown in Fig. 1. The data collected are given in the "Experimental" section. ^{1}H-NMR spectra in DMSO-d_6 showed the aromatic protons as multiplet in the range 6.94–7.63 ppm. The intrinsically unsymmetrical tetradentate Schiff base possess two phenolic and two azomethine groups, both azomethine protons (CH=N) and OH protons of the phenolic groups appearing as sets of sharp singlets at respectively 9.47 and 8.57 ppm, and 13.38 and 12.79 ppm [45, 60]. The two phenolic proton signals disappear in the case of the corresponding unsymmetrical complex **Zn(II)L**, indicating that the OH groups have been deprotonated and bonded to metal ions as oxygen anions. The azomethine proton resonances of the complex form, compared to those of its ligand, are slightly shifted to higher fields for H11, 9.43 ppm and to lower fields for H10, 9.12 ppm (Fig. 1). Thus, NMR data favour a $\{ZnN_2O_2\}$ coordination for the complex in solution (Fig. 3).

In order to assign the ^{1}H-NMR spectrum of **Zn(II)L** complex, cross-peaks in 2D ROESY spectrum due to a through-space magnetic exchange mechanism were obtained for neighboring protons and, cross-peaks from through bond couplings were obtained from ^{1}H–^{1}H COSY. The most important cross-peaks in the 2D ROESY spectra were found for pairs of resonances at δ 9.43/7.70, 9.10/8.33 and 9.10/7.60 corresponding to space proximity of protons H11/H4, H10/H3 and H10/H7 respectively. Thus, H10 proton is in space interaction with two protons, H3 and H7, while H11 proton is only with one proton, H4, as is shown in Fig. 1. The 2D ROESY spectrum is shown in Fig. 2. To our knowledge, these azomethine proton assignments for similar unsymmetrical complexes were not reported in the literature [59, 63]. Consequently, these attributions allowed us to deduce the chemical shifts of all the signals of the aromatic protons not yet assigned. The Table 4 lists the chemical shift attributions. Moreover, the relative areas of the resonances are in perfect agreement with the corresponding protons (Fig. 4).

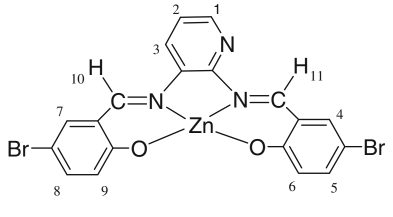

Fig. 3 Structural representation of Zn(II)L showing the labels employed in ^{1}H-NMR and 2D ROESY assignments

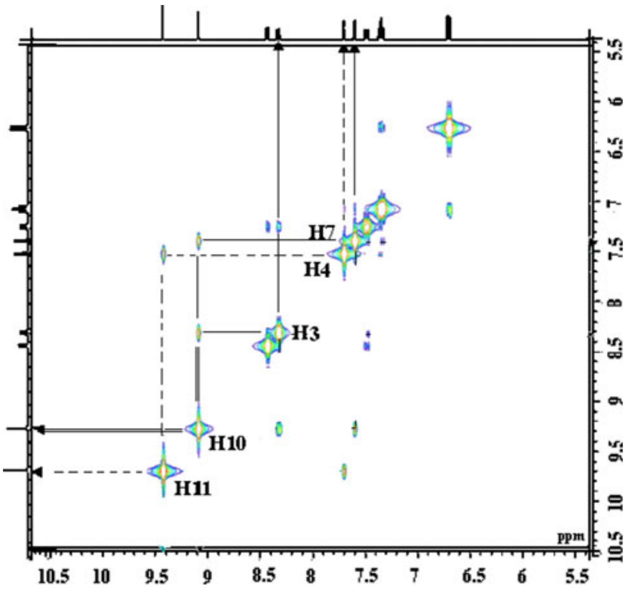

Fig. 4 2D ROESY spectrum of Zn(II)L complex in DMSO-d_6 at 500 MHz

Conclusion

The solid state structure of unsymmetrical zinc-Schiff base crystal was obtained and showed a five coordinate complex involving a pyridine molecule as an apical base. In solution the zinc complex appears as a typical four coordinate zinc(II)salen complex. Amplification of the unsymmetrical character using quaternization or weak interactions involving the pyridine nitrogen atom is under investigation.

Table 4 Proton chemical shifts (ppm) of Zn(II)L in DMSO-d_6 at 500 MHz

Hi	H11	H10	H1	H3	H4	H7	H2	H5,8	H6,9
δ(ppm)	9.43(s)	9.10(s)	8.44(dd)	8.33(dd)	7.70(d)	7.60(d)	7.49(dd)	7.35(dd)	6.70(dd)

s Singlet, *d* doublet

Supplementary Material

CCDC-684010 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/data_request/cif by e-mailing: data_request@ccdc.cam.ac.uk; or by contacting: The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44(0)1223-336033.

Acknowledgments The authors acknowledge gratefully Dr Lydia Brelot for the radiocrystallography facility—Institut de Chimie de Strasbourg, for single crystal X-ray.

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