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Oxidation of the octa-(ethyl)diphosphaferrocene

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Abstract

The octa-(ethyl)-diphosphaferrocene (**1**) reacts with $NOBF_4$ in dichloromethane to yield a polymer (**2**) featuring diphosphaferrocene ligands (**1**) that bridge $[Fe(NO)_2]$ units through their two phosphorus atom lone pairs. The X-ray crystal structure of **2** has been recorded. The formation of the 1-P-fluoro-2,3,4,5-tetra-(ethyl)-phosphole oxide as a by-product demonstrates that phosphaferrocenium (**2**) is sensitive towards substitution reaction at iron and nucleophilic attack at phosphorus. Oxidation of **1** with iodine in dichloromethane yielded the corresponding phosphaferrocenium cation $[FeI_4]^-$ complex (**4**) which has been characterized structurally. The formation of **4** probably results from the partial decomposition of the diphosphaferrocenium cation having $[I_3]^-$ as the counter anion.

Keywords: Phosphorus; Heterocycles; Iron; Complexes; Cations

1. Introduction

Diphosphaferocenes [1] possess unusual binding properties that make them attractive ligands both for the synthesis of sophisticated polymetallic edifices [2] and for catalytic purposes [3]. On the other hand, only a little is known on their use as elementary bricks in the elaboration of charge transfer complexes [4]. Though the electrochemical oxidation processes of phospha- and diphosphaferrocenes have attracted attention from many groups, phospha and diphosphaferroceniums eluded structural characterization for a long time [5]. Suspecting that the electronic richness of the ring and the use of bulky susbtituents could prevent decomposition of these cationic species, we recently launched a program aimed at exploring the oxidation of polyalkylphospha and diphosphaferrocenes. We validated this approach and reported the X-ray crystal structure of the first phosphaferrocenium cation using the pentamethylcyclopentadienyl-tetra-(ethyl)phosphaferrocene [6]. This first encouraging result prompted us to investigate the oxidation of the corresponding octa-(ethyl)-diphosphaferrocene. Herein, we report on these results.

2. Results and discussion

Among many possible oxidizing agents, we first focused our work on the use of $NOBF_4$, which was successfully employed to oxidize decaphenyl ferrocene [7]. Oxidation of **1** was studied in several solvents (THF, MeCN, CH_2Cl_2,...). In all cases, ^{31}P-NMR spectra of the crude mixtures attested the presence of numerous decomposition compounds that could not be identified. However, a significant result was obtained using dichloromethane as solvent at room temperature. Addition of $NOBF_4$ in a dichloromethane solution of **1** provoked the precipitation of a dark green solid **2** and ^{31}P-NMR spectroscopy revealed the formation of compound **3** ($\delta = 64.50$ ppm) featuring a strong $^1J(P{-}F) = 1052$ Hz coupling constant. Compound **3** is the 1-fluorotetra-(ethyl)-phosphole oxide (Eq. (1)).

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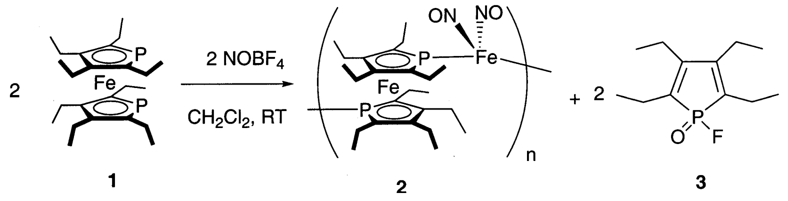


(1)

Compound **2** is a polymeric species, which could not be characterized by NMR spectroscopy due to its insolubility in common organic solvents. Fortunately, suitable crystals of **2** for X-ray crystallography were grown by diffusing n-hexane into a diluted dichloromethane solution of the polymer. Two views of complex **2** are presented in Figs. 1 and 2 and the most significant metric parameters are listed in Table 1. In Fig. 1, a view of some polymeric units is presented whereas a view of the monomeric unit is showed in Fig. 2. As can be seen, the diphosphaferrocene ligand behaves as a four-electron donor through the two phosphorus atom lone pairs, the overall geometry around iron being tetrahedral. From the X-ray data it is clearly apparent that the NO ligands are three electron donors thus yielding an 18 VE complex. Indeed it is well established that, whereas bent NO ligand ($\Theta = 120-140°$) is expected to behave as a one electron donor, linear NO ($\Theta = 160-180°$) must be considered as a three electron-donor ($\Theta = 174.6(2)°$in **2**). Thus, formally, **2** is a Fe(–II) complex [8].

The structure of **2** can be compared to that of [Fe(NO)$_2$(CO)$_2$] [9], [Fe(NO)$_2$(PPh$_3$)$_2$] [10] and that of the [(3,4-dimethylmonophosphaferrocene) Fe(CO)$_4$] complex [11]. A comparison of metric parameters of **2** with those of [Fe(NO)$_2$(CO)$_2$] indicates that Fe–N bonds are shorter in **2** (average 1.655(2) Å in **2** vs. 1.688(3) Å in the carbonyl complex) but the N–O bonds are longer (1.191(3) in **2** vs. 1.171(4) Å in the carbonyl complex). These differences are in good agreement with the respective π-accepting capacity of the two ligands, the diphosphaferrocene being a less efficient π-acceptor than CO. Accordingly, metric parameters of the [Fe(NO)$_2$(PPh$_3$)$_2$] complex are comparable with those of **2**. Thus, the Fe–N bonds in the phosphine complex lies at 1.650(7) Å and the N–O bond at 1.189(4) Å. Accordingly, ν(NO) in **2** (1733 and 1693 cm^{-1}) compare with those of the phosphine complex (1714 and 1674 cm^{-1}) but are markedly different from those of the carbonyl complex (1847 and 1808 cm^{-1}).

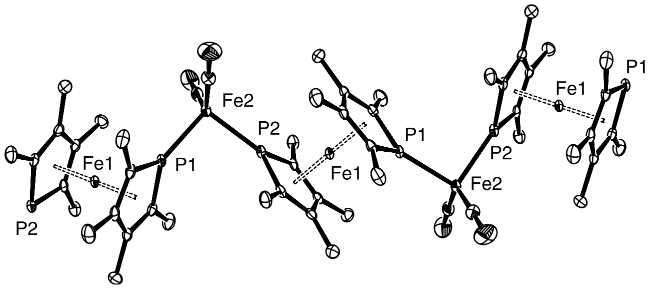


Fig. 1. ORTEP view showing the spatial arrangement of polymer **2**. Methyl groups have been omitted for clarity. Ellipsoids are scaled to enclose 50% of the electron density.

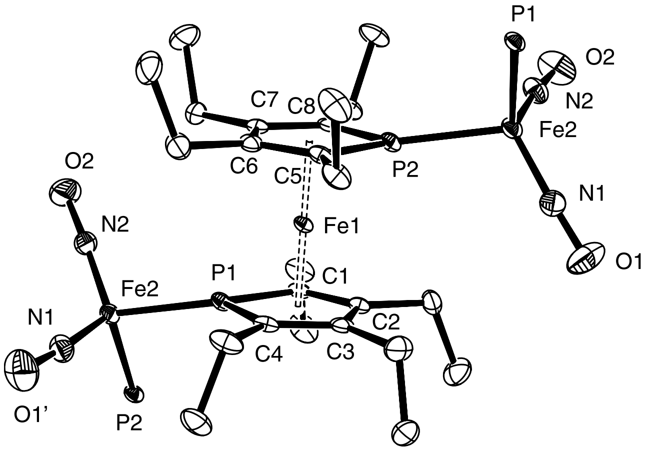


Fig. 2. ORTEP view of one molecule of complex **2**. Ellipsoids are scaled to enclose 50% of the electron density.

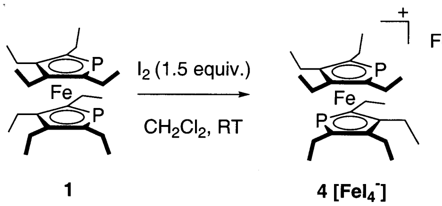
Table 1
Selected bond lengths (Å) and bond angles (°) for complex **2**

Bond lengths			
P(2)–C(5)	1.763(3)	C(3)–C(4)	1.425(4)
C(5)–C(6)	1.423(4)	C(4)–P(1)	1.763(3)
C(6)–C(7)	1.449(4)	P(1)–Fe(1)	2.2798(7)
C(7)–C(8)	1.416(4)	P(2)–Fe(2)	2.2580(8)
C(8)–P(2)	1.766(3)	Fe(2)–N(1)	1.654(2)
P(2)–Fe(1)	2.2465(8)	Fe(2)–N(2)	1.656(2)
P(1)–C(1)	1.764(3)	N(1)–O(1)	1.191(3)
C(1)–C(2)	1.427(4)	N(2)–O(2)	1.191(3)
C(2)–C(3)	1.446(4)	Fe(1)–Ct(1)	1.662(3)
Bond angles			
P(1)–C(1)–C(2)	111.3(2)	C(5)–C(6)–C(7)	112.3(2)
C(1)–C(2)–C(3)	112.4(2)	C(6)–C(7)–C(8)	112.4(2)
C(2)–C(3)–C(4)	112.8(2)	C(7)–C(8)–P(2)	111.9(2)
C(3)–C(4)–P(1)	111.2(2)	N(1)–Fe(2)–P(1)	108.7(1)
C(4)–P(1)–C(1)	92.3(1)	N(2)–Fe(2)–P(1)	109.07(8)
P(2)–C(5)–C(6)	111.8(2)	N(1)–Fe(2)–P(2)	110.2(1)
C(5)–P(2)–C(8)	91.6(1)	N(2)–Fe(2)–P(2)	109.8(1)
P(2)–Fe(2)–N(2)	109.8(1)	O(1)–N(1)–Fe(2)	174.7(2)
P(2)–Fe(1)–P(1)	173.52(3)	O(2)–N(2)–Fe(2)	174.6(2)
N(1)–Fe(2)–N(2)	121.1(1)		

On the other hand, it appears that bond lengths in the phosphaferrocene unit are not significantly modified by the coordination to iron. Thus, the two PC bonds (for example P(2)–C(5) = 1.763(3) Å) are nearly equivalent to those recorded for the free ligand in the C_{2h} conformation (1.773(2) and 1.778(2) Å) as well as the Cα–Cβ (1.423(4) Å in **2** vs. 1.418(2) Å in **1**) and the Cβ–Cβ′ connection (1.449(4) Å in **2** vs. 1.445(4) Å in **1**) [3a]. A similar comparison can be drawn on examining the internal bond angles and the Fe1–centroid bond distances, which are nearly equivalent (Fe(1)–Ct = 1.662(3) in **2** vs. 1.656(2) in **1**). Apart from this, bond distances in the ligand also compare with those of the phospholide unit in the 3,4-dimethylmonophosphaferrocene $Fe(CO)_4$ complex [11].

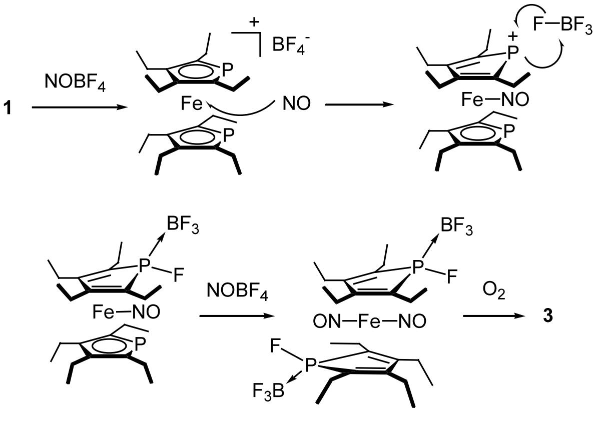
Compound **3** was characterized successfully by NMR and mass spectroscopy. The formation of **2** and **3** raises the problem of the stability of the diphosphaferrocenium cation. Though no mechanistic studies have been undertaken, one may propose that the decomposition of the expected cation results from a nucleophilic attack at the iron atom. A plausible mechanism is depicted in Scheme 1. If we admit that the cation is formed in a first step, one may propose that coordination of NO may occur at the iron atom in a second step leading to a new cationic intermediate. This intermediate would then react with BF_4^- to form a η^4-coordinated 1-P-fluorophosphole BF_3 complex through reaction of F^- at the phosphorus atom. Then, reaction of a second equivalent of $NOBF_4$, following a similar mechanism, would yield two equivalents of the 1-P-fluorophosphole and the $[Fe(NO)_2]$ fragment. Thus, formation of **2** is rationalized easily by coordination of two equivalents of **1** to this metal fragment. Unfortunately, despite many efforts, the 1-P-fluorophosphole BF_3 complex could not be isolated and all our attempts yielded the oxide **3**. The high reactivity of phospha and diphosphaferrocenium towards anions is not unprecedented and has already been discussed on the basis of electrochemical experiments carried out in the presence of chloride and bromide anions [12].

This failure prompted us to investigate other oxidizing reagents. Following a similar route than that employed for the synthesis of the pentamethylcyclopentadienyl-tetra-(ethyl)-phosphaferrocenium cation, we then focused our study on the reaction of iodine with **1**. Oxidation of **1** was carried out in dichloromethane as solvent using 1.5 equivalents of iodine. Contrary to what previously observed in the above-mentioned experiments, a violet coloration rapidly developed upon contact with I_2 and the absence of ^{31}P-NMR spectrum indicated the presence of paramagnetic species. After purification, complex **4** was isolated as a purple solid (Eq. (2)).



(2)

The formulation of **4** was established by X-ray crystallography. As expected, **4** is a diphosphaferrocenium complex but the counteranion is FeI_4^-, not I_3^-. An ORTEP view of one molecule of **4** is presented in Fig. 2 and relevant metric parameters are listed in Table 2. As can be seen, complex **4** adopts a C_{2h} conformation in which the two phosphorus atoms point in opposite directions ($\Theta = 180°$). No contact occurs between the FeI_4^- counter anion and the diphosphaferrocenium. Comparison of bond lengths and bond angles with those of **1** indicates that removal of one electron does not significantly modify the overall structure. Thus, P–C bond lengths at 1.773(4) and 1.778(4) Å are equivalent (1.773(2) and 1.778(2) Å) in **1**) as well as the Cα–Cβ (1.415(5) Å and 1.418(5) in **4** vs. 1.418(2) Å in **1**) and the Cβ–Cβ′ bond (1.434(5) Å in **4** vs. 1.445(4) Å in **1**). The only significant difference between the two structures concerns the Fe–centroid bond, which is elongated in **4**



Scheme 1.

Table 2
Selected bond lengths (Å) and bond angles (°) for complex **4**

Bond lengths			
P(6)–C(4)	1.773(4)	P(6)–Fe(1)	2.328(1)
C(4)–C(3)	1.418(5)	C(4)–Fe(1)	2.121(3)
C(3)–C(2)	1.434(5)	C(3)–Fe(1)	2.130(3)
C(2)–C(1)	1.415(5)	C(2)–Fe(1)	2.137(3)
C(1)–P(6)	1.778(4)	C(1)–Fe(1)	2.151(3)
Fe(1)–Ct(01)	1.722(4)		
Bond angles			
P(6)–C(4)–C(3)	113.4(2)	C(2)–C(1)–P(6)	112.7(2)
C(4)–C(3)–C(2)	111.5(3)	C(1)–P(6)–C(4)	89.6(2)
C(3)–C(2)–C(1)	112.7(3)		

(1.722(4) Å in **4** vs. in 1.656(2) in **1**). This data confirms the idea that oxidation has probably taken place at 3d orbital at the metal. According to Fenske, the HOMO of diphosphaferrocene is the non-bonding $3d_{z^2}$ orbital [12]. However, recent theoretical calculations (DFT, B3LYP using DZVP2 as basis set) reveal that the first three HOMOs are mainly centered on the iron atom ($3d_{xy}$, $3d_{x^2-y^2}$ and $3d_{z^2}$) and that their energetic order will depend on the conformation of the diphosphaferrocene. Interestingly, these three orbitals, which are quite close in energy, also feature a non-negligible contribution of the two phospholide ligands (π^*_C and π^*_P and n_P at phosphorus). This explain why the removing of one-electron will tend to increase the Fe–Ct distance by removing electron density in Fe–C and Fe–P bonds [13] (Fig. 3).

The presence of the FeI_4^- as counteranion confirms the high sensitivity of diphosphaferrocenium cations towards nucleophilic attack. Indeed, a mechanism similar to that depicted for the formation of **2** but involving I^- as the nucleophilic ligand, can be proposed. As previously mentioned, on the basis of electrochemical data, a mechanism involving the formation of a $[FeCl_2(solv)_4]^+[FeCl_4]^-$ complex resulting from a decomposition of an electrochemically generated diphosphaferrocenium cation by Cl^- anion was proposed by Lemoine [14]. Thus, the formation of **4** probably results from the partial decomposition of the corresponding [$\mathbf{1}^+$][I_3^-] complex as depicted in the following scheme (Scheme 2). In his paper, Lemoine proposed that nucleophilic attack of the cation could lead to the formation of the corresponding 1,1′-biphosphole. Unfortunately, we could not confirm this hypothesis and the ^{31}P-NMR spectrum of the mother liquors (after precipitation of complex **4**) only showed the formation of many unidentified phosphorus derivatives. Additional experiments aimed at evaluating the stability of **4** towards iodine showed that addition of solution of iodine in benzene to complex **4**, even at low temperature, provokes a rapid destruction.

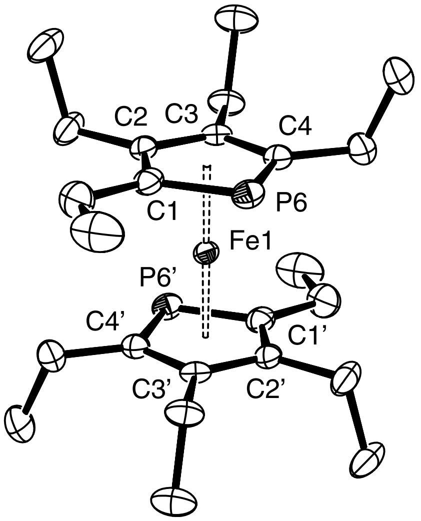


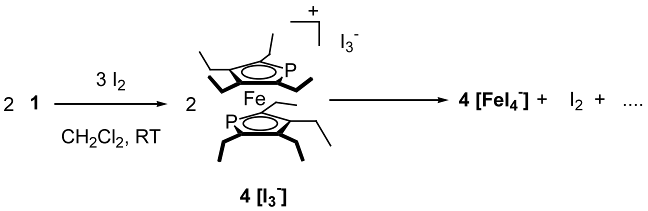
Fig. 3. ORTEP view of one molecule of complex **4**. Ellipsoids are scaled to enclose 50% of the electron density.

In conclusion, we showed that, diphosphaferroecenium cations can be structurally identified, provided that the ring is electronically enriched and sterically protected by alkyl ligands. As expected these cations show a high reactivity towards anions. However, the isolation and structural characterization of a stable diphosphaferrocenium complex paves the way for a systematic study of these complexes. It is expected that a much more stable species could be synthesized by increasing the steric bulk around the phospholide ligand. Syntheses of such species and their use in the elaboration of phosphorus-based charge transfer complexes are currently under active investigation in our laboratories.

3. Experimental

3.1. General

All reactions were routinely performed under an inert atmosphere of argon or nitrogen by using Schlenk and glove-box techniques and dry deoxygenated solvents. Dry hexanes were obtained by distillation from Na/benzophenone and dry CH_2Cl_2 and $CDCl_3$ from P_2O_5. Dry CD_2Cl_2 was distilled and stored, like $CDCl_3$, on 4 Å Linde molecular sieves. Nuclear magnetic resonance spectra were recorded on a Bruker Avance 300 spectrometer operating at 300 MHz for ^{1}H, 75.5 MHz for ^{13}C



Scheme 2.

and 121.5 MHz for ^{31}P. Solvent peaks are used as internal reference relative to Me_4Si for ^{1}H and ^{13}C chemical shifts (ppm); ^{31}P chemical shifts are relative to a 85% H_3PO_4 external reference. The following abbreviations are used: b; broad, singlet; d, doublet; t, triplet; m, multiplet; p, pentuplet; sext, sextuplet; sept, septuplet; v, virtual. Mass spectra were obtained at 70 eV with a HP 5989B spectrometer coupled to a HP 5980 chromatograph by the direct inlet method. Elemental analyses were performed by the 'Service d'analyse du CNRS', at Gif sur Yvette, France.

3.2. Synthesis of polymer (2) and 1-P-fluro-2,3,4,5-tetra-(ethyl)phosphole oxide (3)

$NOBF_4$ (130 mg, 1.10 mmol) was added to a solution of diphosphaferrocene **1** (500 mg, 1.10 mmol) in dichloromethane (15 ml) at room temperature (r.t.). The solution turned green instantaneously and the mixture was stirred for 3 h. This part of the reaction was carried out in the glove-box under argon. The second part of the reaction was carried out under nitrogen using vacuum-line techniques. After evaporation of the solvent, hexane (25 ml) was added and the solution was filtered yielding complex **2** as a green powder. Yield: 186 mg (60%). Compound **3** was isolated after evaporation with hexane as a colorless oil. Purification of **3** was achieved by column chromatography on silica gel using hexane–MeOH (1:1) as eluent. Yield: 76 mg (30%). **2**; νNO (KBr, cm^{-1}) = 1733, 1693. Anal. Calc. for $C_{24}H_{40}Fe_2N_2O_2P_2$: C, 51.27; H, 7.17. Found: C, 50.90; H, 7.08%. **3**; ^{31}P-NMR ($CDCl_3$): δ 64.5 (d, ^1J(P–F) = 1052.3). ^{1}H NMR ($CDCl_3$): δ 1.04 (t, 6H, ^3J(H–H) = 7.50 Hz, 2 × Me), 1.17 (t, 6H, ^3J(H–H) = 7.50 Hz, 2 × Me), 2.30 (m, 8H, 4 × CH_2). ^{13}C-NMR ($CDCl_3$): δ 13.90 (s, Me), 14.40 (s, Me), 19.10 (d, ^4J(C–F) = 10.90 Hz, CH_2), 20.45 (d, ^3J(C–F) = 19.90 Hz, CH_2), 125.60 (dd, ^1J(C–P) = 15.70 Hz, ^2J(C–F) = 121.80 Hz, C2 and C5 of phosphole), 152.30 (dd, ^2J(C–P) = 7.20 Hz, ^3J(C–F) = 37.40 Hz, C3 and C4 of phosphole). MS, *m/z* (relative intensity): 230 [100, M$^+$].

3.3. Synthesis of the octa-(ethyl)phosphaferrocenium tetra-iodoferrate complex (4)

Iodine (190 mg, 0.75 mmol) was added to a solution of **1** (225 mg, 0.5 mmol) in dichloromethane (15 ml) at r.t. The resulting solutions turned violet instantaneously. Stirring was pursued for 3 h and the solvent was evaporated yielding a purple solid. After washings with hexanes (3 × 10 ml), complex **4** was crystallized from a mixture of dichloromethane and hexanes (1:1) at r.t. Yield: 227 mg (90%). Complex **4** being paramagnetic, no NMR spectra could be recorded. Anal. Calc. for $C_{24}H_{40}Fe_2I_4P_2$: C, 28.55; H, 3.99. Found: C, 28.34; H, 3.65%.

Table 3
Crystal Data and Structural Refinement Details for structure of compounds **2** and **4**

	2	**4**
Empirical formula	$C_{24}H_{40}Fe2N_2O_2P_2$	$C_{24}H_{40}Fe_2I_4P_2$
f_w	562.22	1009.80
Crystal system	monoclinic	monoclinic
Space group	$P2_1/c$	$C2/c$
Unit cell dimensions		
a (Å)	9.5976(3)	20.996(5)
b (Å)	17.8833(9)	10.284(5)
c (Å)	15.4121(7)	16.244(5)
β (°)	91.864(3)	108.900(5)
V (Å^3)	2643.9(2)	3318(2)
Z	4	4
D_{calc} (g cm^{-3})	1.412	2.021
μ (cm^{-1})	1.241	4.704
h, k, l ranges	−12 to 11; −21 to 23; −20 to 13	−28 to 28; −12 to 13; −21 to 21
Crystal size (mm^3)	0.14 × 0.08 × 0.06	0.20 × 0.12 × 0.10
Crystal color and habit	yellow, plate	purple, plate
$2\Theta_{max}$ (°)	27.48	28.70
No. of reflections measured	14281	7535
No. of individual reflections	6041	4268
No. of reflections used	4644	3504
R_1 [a] $[I > 2\sigma(I)]$	0.0366	0.0322
wR_2 [b] $[I > 2\sigma(I)]$	0.1157	0.1004
GOF on F^2	1.109	1.068
Largest difference peak and hole (e Å^{-3})	0.720(0.281)/−0.891(0.281)	0.837(0.147)/−1.166(0.147)

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

4. X-ray structural determination of 2 and 4

Suitable crystals of compound **2** and **4** were obtained by diffusing hexane into a diluted dichloromethane solution of the compound. Data were collected on a Nonius κCCD diffractometer, at 150.0 K using a Mo–K_α ($\lambda = 0.71070$ Å) X-ray source and a graphite monochromator. Experimental details are described in Table 3. The crystal structures were solved using SIR-97 [15] and SHELXL-97 [16]. ORTEP drawings were made using ORTEP III for Windows [17].

5. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC nos. 199522 and 199521 for compounds **2** and **4**, respectively. 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).

Acknowledgements

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