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Nickel(II)–triphenylphosphine complexes of ONS and ONN chelating 2-hydroxyacetophenone thiosemicarbazones

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ABSTRACT

The complexes $[Ni(L^1)(PPh_3)]$ (**1**) and $[Ni(L^2)(PPh_3)]\cdot HCl$ (**2**) were synthesized by the reaction of $[Ni(PPh_3)Cl_2]$ and dibasic 2-hydroxyacetophenone-S-R-4-R^1-thiosemicarbazones (R/R^1: H/CH_3, L^1H_2; CH_3/H, L^2H_2). The ligands and the complexes were characterized using elemental analysis, IR and ^1H NMR spectra. In both complexes, the thiosemicarbazone ligands coordinate to nickel(II) by giving two protons. Complex **1** is formed through the phenolate oxygen, azomethine nitrogen and sulfur atoms of L^1 and the P atom of a triphenylphosphine ligand. In complex **2**, L^2 is functional through an ONN donor set, containing a thioamide nitrogen instead of a sulfur atom. X-ray analysis indicated distorted square planar structures for the complexes, and the nickel atoms lie slightly above the planes structured by the donor atoms. In the crystal forms of **1** and **2**, some phenyl ring protons of the phosphine ligand give intramolecular hydrogen bonds with the donor atoms of the thiosemicarbazone moiety, namely the phenolate oxygen (in complexes **1** and **2**) and N^4 nitrogen (in complex **2**).

1. Introduction

Transition metal complexes of phosphine derivatives have been known since the 1950s. After the catalytic effective rhodium–triphenylphosphine compound was described [1], a large number of mixed-ligand complexes with various phosphines and classical ligands have been studied [2–5]. Metal–phosphine complexes of thiosemicarbazones have raised considerable interest because of their possible roles in stereoselective synthesis [5–8]. Thiosemicarbazones and their transition metal complexes have a wide range of biological activities, some of them are antiviral [9,10], antifungal [11], antibacterial [12,13], antitumor [14,15], anticancerogenic [16,17], antioxidant [18] and show insulin mimetic effects [19].

Thiosemicarbazones in metal–phosphine complexes behave depending on the parent carbonyl compounds and the metal ion, as in their common complexes. While N-heterocyclic thiosemicarbazones usually coordinate with a NNS donor set [20,21], some thiosemicarbazones of heterocyclic and aryl-carbonyl derivatives coordinate to the metals Cu(I) [22], Cu(II) [23], Ru(II) [24] and Pd(II) [25] through the azomethine nitrogen and sulfur atoms. 2-Salicylidene-thiosemicarbazones have O, N, and S atoms as donor sites, and they give five and six membered rings in the 1:1:1 complexes $[M(L)PPh_3]$ (where M = Ni(II) [26,27], Pt(II) [28], Pd(II) [29], Ru(II) [30] and Ru(III) [31]).

The ONS chelate structure is most common in metal–phosphine complexes of 2-hydroxyarylidene-thiosemicarbazones having sulfur as a terminal atom and one (or no) substituent on the N^4-nitrogen of the thioamide group [(CS)-N^4HR or (CS)-N^4H_2]. Differently, an ONN chelate structure for S-methyl-5-bromo-salicylaldehyde-thiosemicarbazone was reported in our previous paper [27].

Herein, we present two nickel(II)–triphenylphosphine complexes of 2-hydroxyacetophenone thiosemicarbazones with ONS and ONN coordination modes (Fig. 1). The ligands and complexes were characterized by elemental analysis, IR and ^1H NMR spectroscopies. The structures of the complexes were determined by the X-ray single-crystal diffraction method.

2. Experimental

2.1. Materials and physical measurements

All chemicals were of reagent grade and were used as commercially purchased without further purification. The elemental analyses were determined on a Thermo Finnigan Flash EA 1112 Series Elemental Analyser. Infrared spectra were recorded as KBr discs on a Mattson 1000 FT-IR spectrophotometer in the 4000–400 cm^{-1} range at room temperature. Electronic spectra of the compounds were recorded in a 10^{-5} M $CHCl_3$ solution with an ATI-Unicam spectrometer in the 800–200 nm range. The ^1H NMR spectra were recorded on a Bruker Avance-500 model spectrometer relative to $SiMe_4$, using $CDCl_3$ as the solvent.

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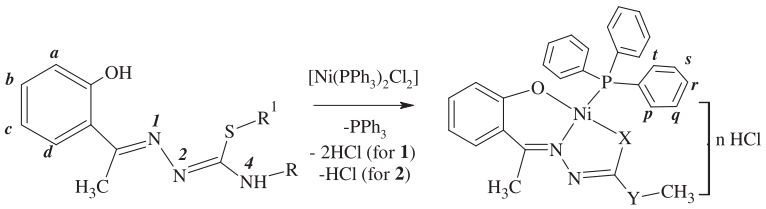

Fig. 1. The ligands, R/R^1: CH_3/H ($\mathbf{L}^1H_2$); CH_3/H ($\mathbf{L}^2H_2$). The complexes, X/Y/*n*: S/NH/0 (**1**); NH/S/1 (**2**).

Suitable crystals of **1** and **2** were mounted on an X-ray diffractomer, Rigaku RAXIS RAPID imaging plate area detector, with graphite monochromated Mo Kα radiation (λ = 0.71070 Å). The unit cell dimensions and intensity data were measured at 293 K (for **1**) and 294 K (for **2**). The data were corrected for Lorentz and polarization effects, and the structures of [Ni(L^1)(PPh_3)] (**1**) and [Ni(L^2)(PPh_3)]·HCl (**2**) were solved by direct methods using the program SIR92 [32]. Hydrogen atoms were refined using the riding model and the non-hydrogen atoms were refined anisotropically. All calculations were performed using the crystal structure crystallographic software package [33,34].

2.2. Synthesis of the ligands

L^1H_2 and L^2H_2 were prepared by the literature method [35]. The colors, m.p. (°C), yields (%), elemental analysis (%), UV–Vis [$\lambda_{max}(\varepsilon)$, nm ($dm^3\ cm^{-1}\ mol^{-1}$)], IR ($cm^{-1}$) and ^{1}H NMR (ppm, *J* in Hz) data of the ligands are as follows:

2.2.1. $\mathbf{L}^1H_2$

Cream, 143.4–144.6; 75. *Anal.* Calc. for $C_{10}H_{13}N_3OS$ (223.3 g): C, 53.79; H, 5.87; N, 18.82; S, 14.36. Found: C, 53.74; H, 5.82; N, 18.79; S, 14.30%. UV–Vis: 241 (26 500), 260 (shoulder), 304 (26 100), 316 (shoulder), 333 (25 300). IR: *v*(OH) 3514, *v*(N^{4}H) 3303, *v*(N^{2}H) 3272, *δ*(N^{4}H) 1643, *δ*(N^{2}H) 1620, *v*(C=N^1) 1601, *v*(—CS—NH) 1538, 1292, *v*(—C=S) 1231. ^{1}H NMR: 10.63 (s, 1H, O*H*), 8.58 (s, 1H, N^2*H*), 6.76 (br s, 1H, N^4*H*), 7.41 (dd, *J* = 1.46, *J* = 7.81, 1H, *d*), 7.25 (ddd, *J* = 1.47, *J* = 7.42, 1H, *b*), 6.92 (dd, *J* = 1.46, *J* = 8.30, 1H, *a*), 6.88 (ddd, *J* = 1.47, *J* = 7.32, 1H, *c*), 3.20 (s, 3H, N^4—C*H*$_3$), 2.31 (s, 3H, C—C*H*$_3$).

2.2.2. $\mathbf{L}^2H_2$

Yellow; 100.7–100.9; 72. *Anal.* Calc. for $C_{10}H_{13}N_3OS$ (223.3 g): C, 53.79; H, 5.87; N, 18.82; S, 14.36. Found: C, 53.76; H, 5.84; N, 18.78; S, 14.31%. UV–Vis: 242 (29 400), 256 (shoulder), 283 (29 100), 315 (shoulder), 324 (16 900). IR: *v*(OH) 3422, *v*(N^{4}H) 3414, 3306, *δ*(N^{4}H) 1647, *v*(C=N^1) 1601, *v*(—CS—NH) 1562, *v*(C—S) 846. ^{1}H NMR: 13.28, 13.16 (*cis*/*trans* ratio 2/1, 1H, O*H*), 7.46 (d, *J* = 1.46, *J* = 7.81, 1H, *d*), 7.20 (ddd, *J* = 1.46, *J* = 7.32, 1H, *b*), 6.89 (ddd, *J* = 0.98, *J* = 8.30, 1H, *a*), 6.81 (ddd, *J* = 0.97, *J* = 6.83, *J* = 7.32, 1H, *c*), 4.88, 4.66 (*cis*/*trans* ratio 2/1, 2H, N^{4}H), 2.46 (d, *J* = 5.37, 3H, S—C*H*$_3$), 2.41 (s, 3H, C—C*H*$_3$).

2.3. Synthesis of the complexes

A solution of 2-hydroxy-acetophenone-4-methyl-thiosemicarbazone (L^1H_2) (2.23 g, 1 mmol) in dichloromethane (10 ml) was added dropwise to a solution of [Ni(PPh_3)$_2$$Cl_2$] (6.54 g, 1 mmol) in 10 ml of absolute ethanol. The mixture was stirred for 4 h at room temperature and left to stand for 6 days. The crystals of the complex [Ni(L^1)(PPh_3)] (**1**) were filtered off and washed with *n*-hexane (10 cm^3) (yield: 85%).

The complex [Ni(L^2)(PPh_3)]·HCl (**2**) was prepared in a similar manner. The colors, m.p. (°C), yields (%), elemental analysis (%), UV–Vis [λ_{max} (ε), nm ($dm^3\ cm^{-1}\ mol^{-1}$)], IR (cm^{-1}) and ^{1}H NMR (ppm, *J* in Hz, *p*–*t* are the symbols for the PPh_3 protons) data of the nickel complexes are given below:

2.3.1. Complex **1**

Dark red; 199.2–199.6; 85. *Anal.* Calc. for $C_{28}H_{26}N_3OPSNi$ (542.26 g): C, 62.02; H, 4.83; N, 7.75; S, 5.91. Found: C, 61.92; H, 4.75; N, 7.82; S, 5.98%. UV–Vis: 241 (85 600), 302 (35 300), 362 (20 900), 408 (12 200). IR: *v*(N^{4}H) 3437, *δ*(N^{4}H) 1639, *v*(C=N^1) 1605, *v*(—CS—NH) 1547, *v*(PPh_3) 1439, 1100, 1031, 1000, 754, 700, *v*(—C—S) 877. ^{1}H NMR: 7.73 (ddd, *J* = 1.47, *J* = 8.79, 6H, *p, t*), 7.41 (m, 3H, *r*), 7.33 (t, *J* = 1.95, *J* = 7.81, 6H, *q, s*), 7.55 (dd, *J* = 1.47, *J* = 8.30, 1H, *d*), 6.90 (ddd, *J* = 1.95, *J* = 6.83, 1H, *b*), 6.53 (ddd, *J* = 1.46, *J* = 8.30, 1H, c), 6.23 (d, *J* = 0.97, *J* = 8.29, 1H, *a*), 4.41 (s, 1H, N^{4}H), 2.83 (s, 3H, N^4—C*H*$_3$), 2.69 (s, 3H, C—C*H*$_3$).

2.3.2. Complex **2**

Dark red; 156.2–157.2; 65. *Anal.* Calc. for $C_{28}H_{27}N_3OPSNiCl$ (578.72 g): C, 58.11; H, 4.70; N, 7.26; S, 5.54. Found: C, 58.01; H, 4.65; N, 7.10; S, 5.50. UV–Vis: 241 (54 200), 297 (24 300), 377 (16 600), 397 (14 000), 408 (7500). IR: *v*(N^{4}H) 3434, *δ*(N^{4}H) 1635, *v*(C=N^1) 1601, *v*(—CS—NH) 1543, *v*(PPh_3) 1435, 1100, 1027, 1000, 750, 696, *v*(—C—S) 869. ^{1}H NMR: 7.75 (ddd, *J* = 1.46, *J* = 8.78, 6H, *p, t*), 7.45 (t, *J* = 6.34, *J* = 7.32, 3H, *r*), 7.38 (t, *J* = 6.34, *J* = 6.83, 6H, *q, s*), 7.65 (dd, *J* = 1.46, *J* = 8.84, 1H, *d*), 6.93 (t, *J* = 7.32, 1H, *b*), 6.61 (t, *J* = 7.32, 1H, *c*), 6.55 (dd, *J* = 1.46, *J* = 8.78, 1H, *a*), 5.23 (s, 1H, N^4*H*), 2.85 (s, 3H, S—C*H*$_3$), 2.15 (s, 3H, C—C*H*$_3$).

3. Results and discussion

3.1. Some physical properties of the compounds

The thiosemicarbazones L^1H_2 and L^2H_2 separated with precipitation from the reaction mixture in the form of powder crystals. The ligands are soluble in common solvents. The reactions of the thiosemicarbazones with [Ni(PPh_3)$_2$$Cl_2$] in a 1:1 molar ratio in a mixture of dichloromethane and ethanol (1:1) yielded solid complexes corresponding to the formulas [Ni(L^1)(PPh_3)] (**1**) and [Ni(L^2)(PPh_3)]·HCl (**2**) (Fig. 1). The compositions of the complexes are stable for at least 4 weeks in air, but the brightness of the crystals decreases within 1–2 weeks.

3.2. Spectral data

UV–Vis spectra of the ligands showed $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ bands at 241–260 and 283–333 nm, respectively [36]. The $n \rightarrow \pi^*$ transitions of the complexes were observed at lower wavelengths, 362 (for **1**) and 377 nm (for **2**). The spectra of **1** and **2** show broad CT bands in the range 362–408 nm, which can be attributed primarily to a S(N) → nickel(II) charge-transfer transition. The d–d transitions related to the square planar structure of the nickel structure could not be clearly observed because of quite low band intensities.

The infrared spectra of the ligands distinctly showed the stretching vibrations of the OH, NH, C=N^1 and N^2=C groups. The structuring of the ONS and ONN chelate rings can be easily

monitored by means of IR spectra due to the fact that the ν(OH) and N^2H bands are absent in the spectra of the complexes. Furthermore, a series of medium and weak bands belonging to PPh_3 are recorded between 696 and 1439 cm^{-1} in the complex spectra.

Coupling results of the aromatic and aliphatic protons on the ligand molecules were recorded at the expected chemical shift values. Because the complex spectra do not contain signals of hydroxy and one of the thioamide moiety protons, complex formation can also be confirmed from the ^1H NMR spectra. Separation of the thioamide protons occurs through the sulfur atom (in **1**) and the N^4 atom (in **2**).

There are some considerable differences in the chemical shift values of equivalent complex protons. The N^4 proton of **1** was recorded at 4.41 ppm, but the N^4H signal of **2** was observed in the higher field (at 5.23 ppm) due to the δ+ effect of the nickel atom bonded to N^4. Depending on the coordinated atom, S or N^4, the C—CH_3 protons of complex **1** were recorded with a shift of approximately 0.38 ppm to higher field compared to the free ligand, while those of complex **2** shifted to lower field by ca. 0.26 ppm.

The spectral results indicate the ONS and ONN coordination modes of L^1H and L^2H, respectively. In these chelation modes, the thiosemicarbazone ligands act as dianionic ligand structures which are formed by the separation of two protons from the OH and SH (in L^1) or N^3H_2 (in L^2) groups.

3.3. Crystallography

Single crystals of complexes **1** and **2**, suitable for X-ray diffraction studies, were grown by crystallization from ethanol–dichloromethane (1:2). Data collection conditions and the parameters of the refinement process are given in Table 1. Ellipsoidal plots of the complexes are shown in Figs. 2 and 3.

Complex **1** is formed by the chelation of the doubly deprotonated thiosemicarbazone ligand to a nickel atom with one PPh_3 group attached (Fig. 1). The chelate structure of complex **1** consists

Table 1
Crystal data and structure refinement parameters for complexes **1** and **2**.

	[Ni(L^1)(PPh_3)] (**1**)	[Ni(L^2)(PPh_3)]·HCl (**2**)
Empirical formula	$C_{28}H_{26}N_3NiOPS$	$C_{28}H_{27}N_3NiOPSCl$
Formula weight	542.27	578.73
T (K)	293	294
Wavelength (Å)	0.71070	0.71070
Crystal dimensions (mm)	0.40 × 0.20 × 0.10	0.70 × 0.30 × 0.10
Crystal system	monoclinic	triclinic
Space group	$P2_1/c$	$P\bar{1}$
Unit cell		
a (Å)	16.2610(7)	8.76050
b (Å)	8.8505(4)	12.49440
c (Å)	19.4604(9)	13.80760
α (°)	90	78.597(6)
β (°)	113.277(2)	72.531(5)
γ (°)	90	72.858(5)
V (Å^3)	2572.7(2)	1367.67(5)
Z	4	2
D_{calc} (g/cm^3)	1.400	1.405
λ (Mo Kα) (mm^{-1})	0.924	0.968
F(0 0 0)	1128.0	600.0
h, k, l Range	$-23 \le h \le 23$	$-12 \le h \le 12$
	$-12 \le k \le 12$	$-17 \le k \le 17$
	$-27 \le l \le 27$	$-19 \le l \le 19$
Reflections collected	1 48 978	1 07 570
Unique reflections	8046	8056
R_{int}	0.039	0.028
R	0.038[$I > 2\sigma(I)$]	0.053[$I > 3\sigma(I)$]
R_w	0.045[$I > 2\sigma(I)$]	0.044[$I > 3\sigma(I)$]
Goodness-of-fit (GOF) on indicator	1.074	1.053

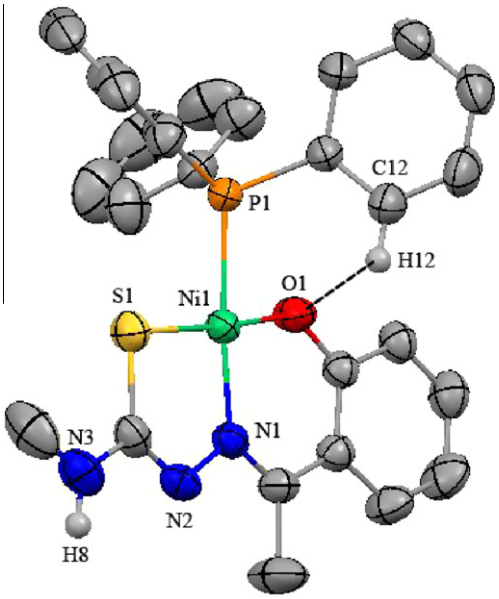

Fig. 2. A view of complex **1** with displacement ellipsoids at the 50% level. The intramolecular hydrogen bond is indicated by broken lines. For clarity, hydrogen atoms (except H8 and H12) are excluded, and labeled atoms are as mentioned in the text.

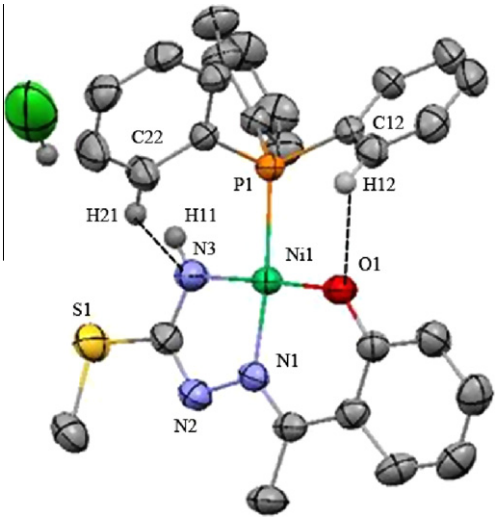

Fig. 3. A view of complex **2** with displacement ellipsoids at the 50% level. The intramolecular hydrogen bond is indicated by broken lines. For clarity, hydrogen atoms (except H11, H12, and H21) are excluded, and labeled atoms are as mentioned in the text.

of two rings, NiOC3N1 and NiN1N2CS, through the O, N1 and S atoms of L^1. The PPh_3 ligand coordinates to nickel(II), and the fourth coordination site of the central atom is completed by the phosphorus atom (Fig. 2). The donor atoms of the complex **1** are placed in the corners of a slightly deformed square plane, like in the nickel–triphenylphosphine complex obtained from 5-bromosalicylaldehyde-thiosemicarbazone [27]. The coordination bonds of both complexes are of approximately the same lengths, although the Ni1—O1 bond of complex **1** is slightly shorter than the equivalent bond of the other complex by approximately 0.03 Å. Additionally, the intramolecular bond between the phenolate oxygen and one of phenyl rings of the phosphine ligand in complex **1** is longer. The relevant geometrical parameters are: C12—H12···O1,

Table 2
The nickel centered bond distances (Å) and angles (°) of **1** and **2**.

Distance (Å)		Angles (°)	
*Complex **1***			
Ni1–O1	1.821(2)	O1–Ni1–S1	173.20(6)
Ni1–N1	1.896(2)	P1–Ni1–N1	175.38(6)
Ni1–P1	2.1948(4)	O1–Ni1–N1	95.26(8)
Ni1–S1	2.1198(7)	O1–Ni1–P1	81.21(5)
		P1–Ni1–S1	94.66(2)
		N1– Ni1–S1	89.14(6)
*Complex **2***			
Ni1–O1	1.811(1)	O1–Ni1–N3	177.67(6)
Ni1–N1	1.883(1)	P1–Ni1–N1	173.74(5)
Ni1–P1	2.12082(4)	O1–Ni1–N1	94.97(6)
Ni1–N3	1.842(2)	O1–Ni1–P1	90.18(4)
		P1–Ni1–N3	92.01(5)
		N1–Ni1–N3	82.79(7)

C—H= 0.95 Å, H···O= 2.656 Å, C···O= 3.127 Å and C—H···O= 111° (for complex **1**), and C—H= 0.93 Å, H···O= 2.52 Å, C···O= 2.914 Å and C—H···O= 106° (for the complex in Ref. [27]).

Complex **2** is formed by chelation of L^2, consisting of two rings, namely NiOC3N1 and NiN1N2CN3, and the square planar environment of the nickel is completed by the phosphorus atom of the second ligand, PPh_3. L^2 displays the ONN coordination mode due to the interaction of its N3 atom with the metal center (Fig. 3). The bond lengths and angles show that complex **2** has a slightly deformed square planar geometry, similar to complex **1** (Table 2). Geometrical parameters are in line with similar nickel–phosphine complexes of thiosemicarbazones [26,27,37]. As distinct from similar molecules, the Ni—P bond length of complex **2** is shorter, being 2.12 Å as compared to 2.19–2.21 Å in the related molecules. This difference may be important for probable catalyst features of complex **2**.

Recrystallizations of complex **2** gave the composition $[Ni(L^2)(PPh_3)]\cdot HCl$. The presence of the HCl molecule in the crystal structure of **2** can be considered to be the result of structural reasons. Although it is very difficult to estimate, it can be said that the backbone of L^2 formed according to the ONN coordination mode might be the cause of the presence of the HCl molecule in the unit cell.

The molecular structure of complex **2** has two intramolecular hydrogen bonds involved protons of PPh_3. Geometrical parameters of these interactions, C12—H12···O1 and C22—H21···N3, leading to the formation of six-membered rings, are C—H= 0.95 Å, H···O= 2.565 Å, C···O= 2.940 Å, C—H···O= 103.77° and C—H= 0.95 Å, H···N= 2.687 Å, C···N= 3.451 Å, C—H···O= 138.02°, respectively. There are no hydrogen-bonds or significant intermolecular interactions in the crystal structures of complexes **1** and **2**.

4. Conclusion

There is a limited number of nickel–phosphine complexes with thiosemicarbazones [26,27,37]. In these complexes, except a nickel complex in our previous paper [27], the thiosemicarbazone ligands having a sulfur as a terminal atom are functional with the ONS coordination mode. In this study, new nickel(II)–triphenylphosphine complexes with 2-hydroxyacetophenone thiosemicarbazones with ONS and ONN coordination modes were characterized. By describing the ONN chelate complex of L^2, we have shown this time using acetophenone thiosemicarbazones that the nitrogen atom (N^3) of the thioamide moiety can react with nickel(II).

5. Supplementary data

CCDC 776732 and 728255 contain the supplementary crystallographic data for complexes **1** ($C_{28}H_{26}N_3NiOPS$) and **2** ($C_{28}H_{27}Cl_1N_3NiOPS$). These data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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