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Polyhedron 25 (2006) 945–952

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Synthesis, spectroscopic characterization and structural studies of nickel complexes of *O*,*O*′-diaryl(dibenzyl) dithiophosphates: Crystal structures of $Ni[S_2P(OC_6H_4CH_3\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4CH_3\text{-}m)_2]_2$ and $Ni[S_2P(OCH_2Ph)_2]_2$

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Received 14 August 2005; accepted 22 October 2005
Available online 6 December 2005

Dedicated with affection and gratitude to Professor M.B. Hursthouse on the occasion of his 'retirement' and J.E.D. wishes to thank Mike Hursthouse for being such a good friend in deed at a time of need.

Abstract

Purple-coloured, tetra-coordinated Ni(II) complexes, $Ni[S_2P(OR)_2]_2$ (R = *o*-, *m*-, *p*-OC_6H_4Me, Ph and CH_2Ph) have been obtained by reaction of $NiCl_2$ with salts of corresponding *O*,*O*′-diaryl(dibenzyl) dithiophosphates and have been characterized by elemental analysis, UV, IR, ^1H, and ^{31}P NMR spectroscopy. Trends in the data are discussed. The crystal structures of $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$ and $Ni[S_2P(OCH_2Ph)_2]_2$ are also described. $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$ crystallizes as monoclinic in the space group $P2_1/n$ with cell parameters $a = 9.069(2)$, $b = 13.503(2)$, $c = 23.735(4)$ Å, $\alpha = 90.00°$, $\beta = 90.11(1)°$, $\gamma = 90.00°$, $V = 2906.6(8)$ Å^{-3}, $Z = 4$, $R = 0.0351$, $R_w = 0.0787$, $Ni[S_2P(OC_6H_4CH_3\text{-}m)_2]_2$ as triclinic in the space group $P\bar{1}$ with cell parameters $a = 9.4675(4)$, $b = 12.8568(5)$, $c = 13.1174(6)$ Å, $\alpha = 88.810(4)°$, $\beta = 83.416(5)°$, $\gamma = 73.711(4)°$, $V = 1522.4(1)$ Å^{-3}, $Z = 2$, $R = 0.0354$, $R_w = 0.0830$, and $Ni[S_2P(OCH_2Ph)_2]_2$ as triclinic in the space group $P\bar{1}$ with cell parameters $a = 10.887(2)$, $b = 14.081(3)$, $c = 16.238(3)$ Å, $\alpha = 72.70(3)°$, $\beta = 76.57(3)°$, $\gamma = 72.76(3)°$, $V = 2241.4(8)$ Å^{-3}, $Z = 2$, $R = 0.0365$, $R_w = 0.0785$. Each of these has a distorted square-planar geometry around nickel with MS_4 coordination environment.

Keywords: Nickel; *o*-, *m*-, *p*-tolyl; Dithiophosphates; Benzyl; Structure

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1. Introduction

Among the variety of dithio ligating systems, nickel complexes with *O*,*O*′-dialkyl dithiophosphates [1–11] and *O*,*O*′-alkylene dithiophosphates [12–17] have consistently retained interest for the last few decades. These ligands exhibit versatile modes of coordination and are known to exist in bidentate and monodentate forms. However, complexes with analogous aromatic ligands have been less studied. A survey of the literature confirms the importance of nickel complexes with thio ligands where the main thrust of research has been on the preparation of complexes having varied applications in agriculture as pesticides [18], in lubrication engineering as antiwear and extreme pressure additives [19,20], and in industry as antioxidants of polyolefines [21] and as catalyst stabilizers [22].

Our interest in exploring the coordination and bonding properties of thio ligands led us to work on *O*,*O*′-diaryl dithiophosphate ligand moieties. Some of these nickel(II) complexes have been reported [23,24] in a fragmented

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doi:10.1016/j.poly.2005.10.022

manner. Therefore, a systematic, consolidated and comparative discussion on complexes of this series may prove useful. In continuation of our search for new nickel(II) complexes has led to this paper in which we report synthesis of nickel(II) complexes of the types, $Ni[S_2P(OR)_2]_2$, (R = *o*-, *m*-, *p*-C_6H_4Me, Ph, CH_2Ph) and their characterization by various spectroscopic techniques. We also report on the X-ray single crystal structures of $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$, and $Ni[S_2P(OCH_2Ph)_2]_2$.

2. Results and discussions

Reactions of nickel(II) chloride with ammonium salts of *O*,*O*′-ditolyl/diphenyl/dibenzyl dithiophosphates in 1:2 molar ratio in aqueous solution yield nickel(II) complexes of the types, $Ni[S_2P(OR)_2]_2$

$$NiCl_2 + 2NH_4S_2P(OR)_2 \longrightarrow Ni[S_2P(OR)_2]_2 + 2NH_4Cl$$

(R = *o*-, *m*-, *p*-OC_6H_4Me, Ph and CH_2Ph).

The precipitated purple solid was separated by filtration and repeatedly washed with benzene. All of these nickel(II) complexes are purple, powdery solids, soluble in all common organic solvents and were characterized by elemental analysis, IR, UV–Vis, ^{1}H and ^{31}P NMR spectroscopy.

2.1. Electronic absorption spectra

The electronic spectra of the nickel(II) complexes, recorded in C_6H_6, show three bands in the region 681–690, 517–530 and 390–395 nm in the visible region with a single band at 320–322 nm in the near ultraviolet region. The first three bands can be assigned to $^1A_{1g} \rightarrow {}^1A_{2g}$, $^1A_{1g} \rightarrow {}^1B_{2g}$, and $^1A_{1g} \rightarrow {}^1E_g$ transitions, respectively. Almost identical electronic transitions for open chain and cyclic dithiophosphate complexes were observed [25,26] indicating consistent square-planar geometry.

2.2. IR spectra

The relevant assignments of the IR bands (Table 1) have been made by comparisons with the spectra of the ammonium salts of *O*,*O*′-ditolyl/diphenyl/dibenzyl dithiophosphates [27,28] and analogous nickel(II) *O*,*O*′-dialkyl dithiophosphate complexes [25,29]. The two strong intensity bands present in the 1043–985 and 835–770 cm^{-1} regions are assigned to ν[(P)–O–C] and ν[P–O–(C)] stretching vibrations, respectively. Bands due to ν(P=S) or ν(P–S)$_{asym}$ and ν(P–S) or ν(P–S)$_{sym}$ observed at 690–670 and 595–540 cm^{-1} in the ditolyl dithiophosphoric acids and their ammonium salts, are shifted to lower frequency by ca. 10–30 cm^{-1} in the corresponding nickel(II) complexes, indicating bidentate attachment of the ligand to the metal atom. In addition, the bands characteristic of *ortho* substitution can be seen in the region 1944–1733w cm^{-1}, 755s cm^{-1}, those of meta substitution in 1952–1809w cm^{-1}, 897, 778s cm^{-1} and those of para substitution in 1884–1745w cm^{-1}, 816s cm^{-1}. Similarly, mono substitution in complexes **4** and **5** are observed in the region 1952–1809w cm^{-1} as well as two peaks at 695, 744s cm^{-1} [30].

2.3. ^{1}H NMR spectra

The spectra were recorded in $CDCl_3$ solutions at room temperature. The ^{1}H NMR data of nickel(II) complexes are remarkably similar to those of the salts of dithiophosphoric acids [31] probably due to the large separation between the nickel and protons. Negligible shift for methyl protons is observed in complexes **1**–**3** as compared to the corresponding salts, showing a singlet in the range 2.24–2.37 ppm. The phenyl protons appear in the region 6.85–7.41 ppm in complexes **1**–**5**, undergoing slight down field shifts of ca. 0.1–0.2 ppm compared to their position in the free ligand, presumably as a consequence of the coordination of the ligand.

2.4. ^{31}P NMR spectra

The ^{31}P NMR spectra of complexes **1**–**5**, show a relatively sharp, singlet in the region 79–94 ppm, indicating that the phosphorous nuclei are equivalent and only one type of phosphorous is present in each case in solution. The ^{31}P NMR chemical shifts for these complexes are shifted approximately 20–25 ppm upfield from those of the corresponding salts of dithiophosphoric acids.

2.5. Molecular structures of $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$ and $Ni[S_2P(OCH_2Ph)_2]_2$

$Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$ (**1**) crystallizes in the space group $P2_1/n$ and $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$ (**2**) and $Ni[S_2P(OCH_2Ph)_2]_2$ (**5**) crystallize in the space group $P\bar{1}$. The ORTEP diagram in Fig. 1 shows the single molecule in

Table 1
IR spectral data (cm^{-1}) for the bis[*O*,*O*′-diaryl(dibenzyl)dithiophosphate]nickel(II) complexes[a]

No.	Compound	ν [(P)–O–C]	ν [P–O–(C)]	ν (P=S)	ν (P–S)
1	$Ni[S_2P(OC_6H_4CH_3\text{-}o)_2]_2$	1043m	725m	659, 603s	560m
2	$Ni[S_2P(OC_6H_4CH_3\text{-}m)_2]_2$	1010s	718m	622, 603s	565m
3	$Ni[S_2P(OC_6H_4CH_3\text{-}p)_2]_2$	1017m	713m	665, 556s	535m
4	$Ni[S_2P(OC_6H_5)_2]_2$	1005m	723m	667, 603s	562m
5	$Ni[S_2P(OCH_2C_6H_5)_2]_2$	985s	821m	654, 603s	565m

[a] Key: s = strong, m = medium, w = weak.

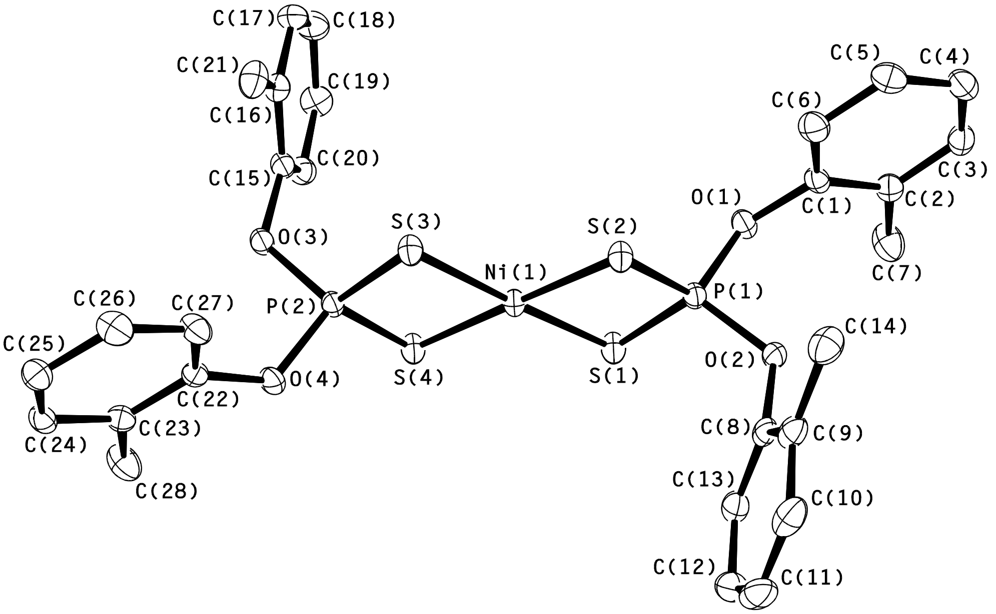

Fig. 1. ORTEP plot of Ni$\{S_2[P(OC_6H_4CH_3\text{-}o)_2]_2\}_2$. The non-hydrogen atoms are drawn with 50% probability ellipsoids. Hydrogen atoms omitted for clarity.

the asymmetric unit of **1**. In displaying the expanded two molecules of the asymmetric unit of **2** (Fig. 2) and the one and one-half molecules in the asymmetric unit of **5** (Fig. 3), the hydrogen atoms were omitted for clarity. Figs. 1–3 illustrate that the immediate environment about the Ni atom is essentially distorted square-planar and that the two dithiophosphate groups are bidentate in both complexes. The average Ni–S distance is 2.2332(6) for **1**, 2.236(6) for

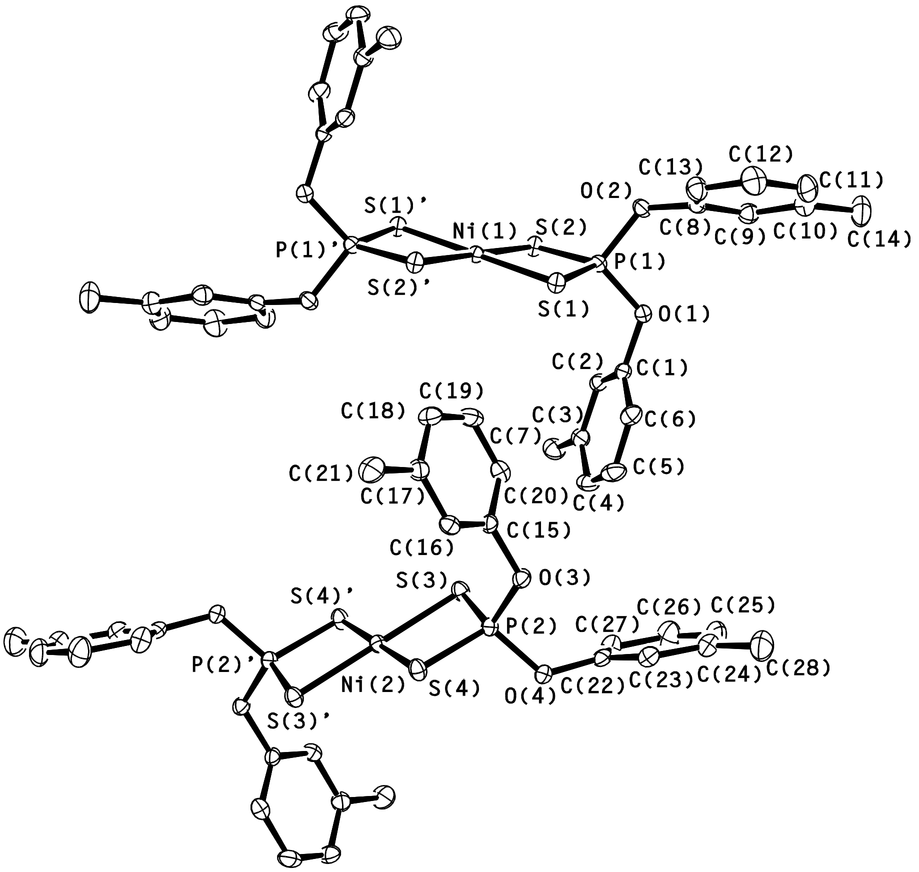

Fig. 2. ORTEP plot of the expanded two independent molecule of Ni$[S_2P(OC_6H_4CH_3\text{-}m)_2]_2$. The non-hydrogen atoms are drawn with 50% probability ellipsoids. Hydrogen atoms omitted for clarity.

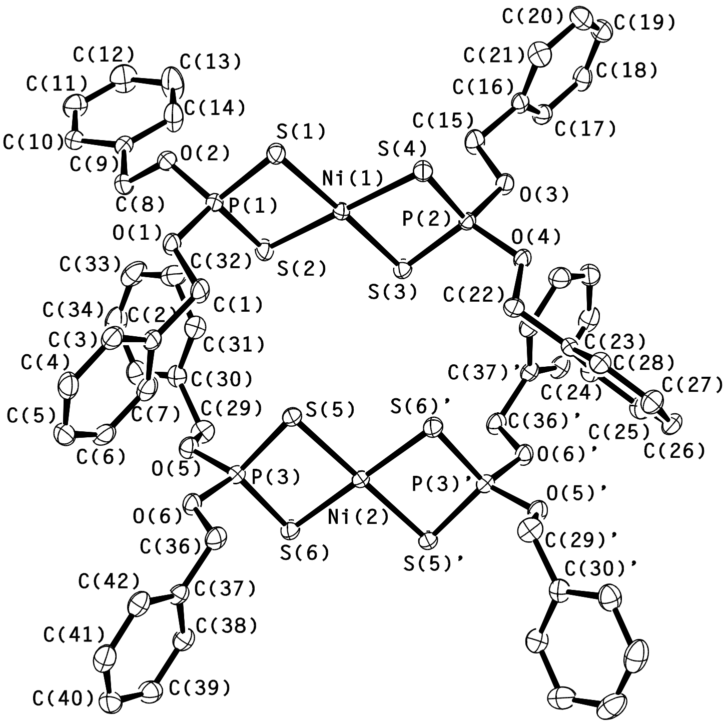

Fig. 3. ORTEP plot of the two independent molecules of $Ni[S_2P(OCH_2Ph)_2]_2$. The non-hydrogen atoms are drawn with 50% probability ellipsoids. The hydrogen atoms are omitted for clarity.

both molecules in **2** and 2.235(7) Å for those in **5**, are similar to the average value of 2.232(4) Å for $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2]$ [23]. This bond length is marginally longer than found in dialkyl dithiophosphate analogue, $Ni[S_2P(OEt)_2]_2$ (2.21 Å) [32,33] and $Ni[S_2P(OPr^i)_2]_2$ (2.222(5) Å) [34]. The average S–P distances are 1.9841(7) in **1**, 1.982(7) in **2** and 1.996(5) Å in **5**, which are comparable to the values observed in $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2]$ (1.976(2) Å), $Ni[S_2P(OEt)_2]_2$(1.97) and $Ni[S_2P(OPr^i)_2]_2$ (1.992(1) Å). The average P–O distances of 1.586(1) in **1**, 1.585(5) in **2** and 1.578(4) Å in **5**, lie between the values reported for the analogues, $Ni[S_2P(OEt)_2]_2$ (1.63) and $Ni[S_2P(OPr^i)_2]_2$ (1.565(1) Å) while that of **2** is identical to that of $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2]$ (1.585(2) Å).

The S–Ni–S ligand bite angles are 88.39(2)° and 88.59(2)° in **1**, are 88.79(2)° and 89.18(2)° in the two molecules of **2**, and range from 87.95(3)° to 88.52(3)° in **5**; similar to the values in $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2]$ (89.12(2)°) and the two analogues, $Ni[S_2P(OEt)_2]_2$ (88°) and $Ni[S_2P(OPr^i)_2]_2$ (88.10(4)°). The S–P–S bond angles vary from 103.32(3)° to 103.69(3)° in **1** and range from 104.18(4)° to 104.80(4)° in **2**, thus, having similar values as in the *para* substituted analogue, $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2]$ (104.85(3)°). The values are marginally smaller in the OCH_2Ph derivative **5** (101.73(4)–102.81(4)°). The angles reported in $Ni[S_2P(OEt)_2]_2$ and $Ni[S_2P(OPr^i)_2]_2$ are 103° and 101.7(1)°, respectively. The O–P–O bond angles are 99.37(7)–99.24(7)° in **1**, range from 99.83(9)° to 100.20(9)° in **2** and are 99.39(7)° in $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2$. These values are bracketed by those in **5** ranging from 95.22(8)° to 101.04(9)°. Thus, there appear to be no significant changes or obvious trends in the distances and bond angles as the nature of R is changed in the $Ni[S_2P(OR)_2]_2$ complexes although in general the tolyl derivatives, $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}p)_2]_2$, probably show the most similarities.

3. Experimental

Nickel chloride, benzyl alcohol, *o*-, *m*-, *p*-cresols were purchased from E. Merck. Solvents (benzene, *n*-hexane, diethyl ether) were dried by standard methods before use. Stringent precautions were taken to exclude moisture during experimental manipulations. Literature methods were used for the preparation of *O*,*O*′-ditolyl [27] and *O*,*O*′-diphenyl [28] dithiophosphoric acids. *O*,*O*′-dibenzyl dithiophosphoric acid was prepared by the reaction of P_2S_5 and benzyl alcohol in 1:4 molar ratio. Ammonium salts of the dithiophosphoric acids were prepared by the reaction of parent acids with an equimolar amount of ammonia in benzene.

3.1. Preparations

3.1.1. $Ni[S_2P(OC_6H_4Me\text{-}o)_2]$ (1)

Typically, to an aqueous solution of $NiCl_2 \cdot 6H_2O$ (0.287 g, 1.2073 mmol), was added an aqueous suspension of $NH_4S_2P(OC_6H_4Me\text{-}o)_2$ (0.801 g, 2.449 mmol) in 1:2 molar ratio with constant stirring. A purple solid was immediately obtained. After stirring for 3 h, the precipitated $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$ was filtered off. The compound was purified with repeated washing with dried benzene. Purple powdery solid. Yield: 0.768 g, 94%; m.p. 90 °C. Calc. for $C_{28}H_{28}O_4S_4P_2Ni$: Ni, 8.71; S, 18.90. Found: Ni, 8.75; S, 18.72%. ^{1}H NMR ($CDCl_3$): δ 2.25 (s, Me), 7.04–7.41 (m, OC_6H_4), ^{31}P NMR ($CDCl_3$): δ 84.10 ppm. UV/Vis electronic absorption spectral data [λ_{max}, nm(A)] in benzene 685, 520, 392, 322.

3.1.2. *Ni[$S_2P(OC_6H_4CH_3$-m$)_2]_2$ (**2**)*

Method as above gave a purple powdery solid. Yield: 0.807 g, 93%; m.p. 106 °C. Calc. for $C_{28}H_{28}O_4S_4P_2Ni$: Ni, 8.71; S, 18.90. Found: Ni, 8.60; S, 18.98%. ^{1}H NMR ($CDCl_3$): δ 2.38 (s, Me), 7.07–7.29 (dm, OC_6H_4), ^{31}P NMR ($CDCl_3$): δ 84.95 ppm. UV/Vis electronic absorption spectral data [λ_{max}, nm(A)] in benzene 690, 517, 394, 320.

3.1.3. *Ni[$S_2P(OC_6H_4CH_3 - p)_2]_2$ (**3**)*

Method as above gave a pale purple powdery solid. Yield: 0.797 g, 89%; m.p. 171 °C. Calc. for $C_{28}H_{28}O_4S_4$-P_2Ni: Ni, 8.71; S, 18.90. Found: Ni, 8.47; S, 18.88%. ^{1}H NMR ($CDCl_3$): δ 2.31 (s, Me), 6.85–7.37 (m, OC_6H_4), ^{31}P NMR ($CDCl_3$): δ 79.10 ppm. UV/Vis electronic absorption spectral data [λ_{max}, nm(A)] in benzene 681, 530, 395, 322.

3.1.4. *Ni[$S_2P(OPh)_2]_2$ (**4**)*

Method as above gave a purple powdery solid. Yield: 0.759 g, 91%; m.p. 122 °C. Calc. for $C_{24}H_{24}O_4S_4P_2Ni$: Ni, 8.28; S, 18.53. Found: Ni, 8.12; S, 18.60%. ^{1}H NMR ($CDCl_3$): δ 7.23–7.32 (dm, OC_6H_5), ^{31}P NMR ($CDCl_3$): δ 84.59 ppm. UV/Vis electronic absorption spectral data [λ_{max}, nm(A)] in benzene 688, 525, 394, 321.

3.1.5. *Ni[$S_2P(OCH_2Ph)_2]_2$ (**5**)*

Method as above gave a purple powdery solid. Yield: 0.668 g, 86%; m.p. 125 °C. Calc. for $C_{28}H_{28}O_4S_4P_2Ni$: Ni, 8.71; S, 18.90. Found: Ni, 8.64; S, 18.78%. ^{1}H NMR ($CDCl_3$): δ 5.22 (s, OCH_2), 6.99–7.33 (bs, C_6H_5), ^{31}P NMR ($CDCl_3$): δ 93.66 ppm. UV/Vis electronic absorption spectral data [λ_{max}, nm(A)] in benzene 683, 527, 390, 320.

3.2. *Physical measurements*

Sulfur was estimated gravimetrically as barium sulfate (Messenger's method) and Nickel as dimethylglyoximate. IR spectra were recorded on a Perkin–Elmer 577 spectrophotometer in KBr pellets in the range 4000–200 cm^{-1}. Electronic spectra in the range 200–800 nm were recorded on UV visible spectrophotometer-2201 in C_6H_6. ^{1}H NMR spectra were recorded on a Bruker DRX300 (300MHzFT NMR) spectrometer in $CDCl_3$ using TMS as an internal standard.

Table 2
Crystal data and structure refinement for $NiS_2[P(OC_6H_4CH_3$-o$)_2]_2$ (**1**), $NiS_2[P(OC_6H_4CH_3$-m$)_2]_2$ (**2**), and Ni[$S_2P(OCH_2Ph)_2]_2$ (**5**)

Empirical formula	$C_{28}H_{28}O_4P_2S_4Ni$	$C_{28}H_{28}O_4P_2S_4Ni$	$C_{42}H_{42}O_{1.5}P_3S_6Ni_{1.5}$
Formula weight	677.39	677.39	1016.90
Temperature (K)	120(2)	120(2)	120(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	monoclinic	triclinic	triclinic
Space group	$P2_1/n$	$P\bar{1}$	$P\bar{1}$
a (Å)	9.093(2)	9.4675(4)	10.887(2)
b (Å)	13.529(2)	12.8568(5)	14.081(3)
c (Å)	23.779(4)	13.1174(6)	16.238(3)
α (°)	90.00	88.810(4)	72.70(3)
β (°)	90.17(1)	83.416(5)	76.57(3)
γ (°)	90.00	73.711(4)	72.76(3)
Volume (Å^3)	2925.3(8)	1522.4(1)	2241.4(8)
Z	4	2	2
D_{calc} (g/cm^3)	1.548	1.478	1.506
Absorption coefficient (mm^{-1})	1.099	1.049	1.069
$F(000)$	1400	700	1050
Crystal size (mm^3)	0.10 × 0.10 × 0.05	0.50 × 0.30 × 0.14	0.10 × 0.08 × 0.08
θ Range for data collection (°)	2.98–27.48	2.99–27.54	2.91–27.47
Index ranges	$-11 \leqslant h \leqslant 10$, $-14 \leqslant k \leqslant 17$, $-24 \leqslant l \leqslant 30$	$-12 \leqslant h \leqslant 12$, $-16 \leqslant k \leqslant 16$, $-17 \leqslant l \leqslant 17$	$-14 \leqslant h \leqslant 14$, $-18 \leqslant k \leqslant 18$, $-21 \leqslant l \leqslant 21$
Reflections collected	23640	28956	47686
Independent reflections (R_{int})	6621 (0.0418)	6973 (0.0383)	10182 (0.0536)
Maximum and minimum transmission	0.9471 and 0.8980	0.8670 and 0.6220	0.9193 and 0.9006
Refinement method	full-matrix least-squares on F^2	full-matrix least-squares on F^2	full-matrix least-squares on F^2
Data/restraints/parameters	6621/0/356	6973/0/355	10182/0/571
Goodness-of-fit on F^2	1.036	1.031	1.035
Final R indices [$F^2 > 4\sigma(F^2)$]	$R_1 = 0.0351$, $wR_2 = 0.0787$	$R_1 = 0.0354$, $wR_2 = 0.0830$	$R_1 = 0.0365$, $wR_2 = 0.0785$
R indices (all data)	$R_1 = 0.0456$, $wR_2 = 0.0843$	$R_1 = 0.0503$, $wR_2 = 0.0899$	$R_1 = 0.0545$, $wR_2 = 0.0857$
Largest difference peak and hole (e Å^{-3})	0.356 and −0428	0.563 and −0.573	0.322 and −0.467

Table 3
Bond lengths (Å) and angles (°) for Ni{$S_2[P(OC_6H_4CH_3\text{ -}o)_2]_2\}_2$ (**1**)

Ni(1)–S(1)	2.2415(6)	Ni(1)–S(3)	2.2353(6)
Ni(1)–S(2)	2.2241(6)	Ni(1)–S(4)	2.2318(6)
S(1)–P(1)	1.9839(7)	S(3)–P(2)	1.9854(7)
S(2)–P(1)	1.9850(7)	S(4)–P(2)	1.9820(7)
P(1)–O(1)	1.578(1)	P(2)–O(3)	1.592(1)
P(1)–O(2)	1.595(1)	P(2)–O(4)	1.579(1)
O(1)–C(1)	1.420(2)	O(3)–C(15)	1.411(2)
O(2)–C(8)	1.418(2)	O(4)–C(22)	1.419(2)
C(1)–C(6)	1.380(3)	C(15)–C(16)	1.389(3)
C(1)–C(2)	1.388(3)	C(15)–C(20)	1.390(3)
C(2)–C(3)	1.388(3)	C(16)–C(17)	1.394(3)
C(3)–C(4)	1.387(3)	C(17)–C(18)	1.375(3)
C(4)–C(5)	1.383(3)	C(18)–C(19)	1.384(3)
C(5)–C(6)	1.391(3)	C(19)–C(20)	1.383(3)
C(2)–C(7)	1.494(3)	C(16)–C(21)	1.495(3)
C(8)–C(9)	1.384(3)	C(22)–C(27)	1.372(3)
C(8)–C(13)	1.393(3)	C(22)–C(23)	1.391(3)
C(9)–C(10)	1.395(3)	C(23)–C(24)	1.394(3)
C(10)–C(11)	1.379(3)	C(24)–C(25)	1.380(3)
C(11)–C(12)	1.382(3)	C(25)–C(26)	1.380(3)
C(12)–C(13)	1.379(3)	C(26)–C(27)	1.391(3)
C(9)–C(14)	1.493(3)	C(23)–C(28)	1.498(3)
S(1)–Ni(1)–S(2)	88.39(2)	S(3)–Ni(1)–S(4)	88.59(2)
S(1)–Ni(1)–S(3)	178.51(2)	S(2)–Ni(1)–S(4)	178.00(2)
S(1)–Ni(1)–S(4)	92.04(2)	S(2)–Ni(1)–S(3)	91.03(2)
Ni(1)–S(1)–P(1)	83.86(3)	Ni(1)–S(3)–P(2)	83.57(3)
Ni(1)–S(2)–P(1)	84.30(3)	Ni(1)–S(4)–P(2)	83.74(3)
S(1)–P(1)–S(2)	103.32(3)	S(3)–P(2)–S(4)	103.69(3)
O(1)–P(1)–O(2)	99.37(7)	O(3)–P(2)–O(4)	99.24(7)
O(1)–P(1)–S(1)	108.82(6)	O(3)–P(2)–S(3)	113.64(6)
O(1)–P(1)–S(2)	116.03(6)	O(3)–P(2)–S(4)	115.39(6)
O(2)–P(1)–S(2)	114.41(6)	O(4)–P(2)–S(4)	109.03(6)
O(2)–P(1)–S(1)	115.31(6)	O(4)–P(2)–S(3)	116.29(6)
C(1)–O(1)–P(1)	126.4(1)	C(15)–O(3)–P(2)	122.1(1)
C(8)–O(2)–P(1)	123.1(1)	C(22)–O(4)–P(2)	124.3(1)
C(2)–C(1)–C(6)	123.0(2)	C(16)–C(15)–C(20)	123.4(2)
C(6)–C(1)–O(1)	120.9(2)	C(20)–C(15)–O(3)	118.3(2)
C(2)–C(1)–O(1)	116.1(2)	C(16)–C(15)–O(3)	118.1(2)
C(1)–C(2)–C(3)	116.8(2)	C(15)–C(16)–C(17)	115.7(2)
C(2)–C(3)–C(4)	121.9(2)	C(16)–C(17)–C(18)	122.6(2)
C(3)–C(4)–C(5)	119.6(2)	C(17)–C(18)–C(19)	119.8(2)
C(4)–C(5)–C(6)	120.1(3)	C(18)–C(19)–C(20)	120.2(2)
C(5)–C(6)–C(1)	118.7(2)	C(19)–C(20)–C(15)	118.4(2)
C(1)–C(2)–C(7)	121.7(2)	C(15)–C(16)–C(21)	122.1(2)
C(3)–C(2)–C(7)	121.6(2)	C(17)–C(16)–C(21)	122.2(2)
C(9)–C(8)–C(13)	122.8(2)	C(23)–C(22)–C(27)	122.9(2)
C(13)–C(8)–O(2)	117.5(2)	C(27)–C(22)–O(4)	120.6(2)
C(9)–C(8)–O(2)	119.5(2)	C(23)–C(22)–O(4)	116.4(2)
C(8)–C(9)–C(10)	116.3(2)	C(22)–C(23)–C(24)	116.9(2)
C(9)–C(10)–C(11)	121.9(2)	C(23)–C(24)–C(25)	121.4(2)
C(10)–C(11)–C(12)	120.3(2)	C(24)–C(25)–C(26)	120.1(2)
C(11)–C(12)–C(13)	119.7(2)	C(25)–C(26)–C(27)	120.1(2)
C(12)–C(13)–C(8)	119.0(2)	C(26)–C(27)–C(22)	118.8(2)
C(8)–C(9)–C(14)	122.6(2)	C(22)–C(23)–C(28)	121.4(2)
C(10)–C(9)–C(14)	121.1(2)	C(24)–C(23)–C(28)	121.8(2)

Table 4
Selected bond lengths (Å) and angles (°) for $NiS_2[P(OC_6H_4CH_3\text{-}m)_2]_2$ (**2**)[a]

Molecule 1		Molecule 2	
Ni(1)–S(1)	2.2421(6)	Ni(2)–S(3)	2.2430(6)
Ni(1)–S(1)′	2.2421(6)	Ni(2)–S(3)″	2.2430(6)
Ni(1)–S(2)	2.2310(6)	Ni(2)–S(4)	2.2286(6)
Ni(1)–S(2)′	2.2310(6)	Ni(2)–S(4)″	2.2286(6)
S(1)–P(1)	1.9884(8)	S(3)–P(2)	1.9849(8)
S(2)–P(1)	1.9778(8)	S(4)–P(2)	1.9774(8)
P(1)–O(1)	1.589(2)	P(2)–O(3)	1.591(2)
P(1)–O(2)	1.579(2)	P(2)–O(4)	1.582(2)
O(1)–C(1)	1.418(3)	O(3)–C(15)	1.420(3)
O(2)–C(8)	1.417(3)	O(4)–C(22)	1.420(3)
C(1)–C(6)	1.375(3)	C(15)–C(20)	1.371(3)
C(1)–C(2)	1.381(3)	C(15)–C(16)	1.384(3)
C(2)–C(3)	1.394(3)	C(16)–C(17)	1.395(3)
C(3)–C(4)	1.395(4)	C(17)–C(18)	1.391(4)
C(4)–C(5)	1.388(4)	C(18)–C(19)	1.386(4)
C(5)–C(6)	1.393(4)	C(19)–C(20)	1.391(4)
C(3)–C(7)	1.504(3)	C(17)–C(21)	1.507(3)
C(8)–C(13)	1.376(3)	C(22)–C(27)	1.381(4)
C(8)–C(9)	1.379(3)	C(22)–C(23)	1.371(4)
C(9)–C(10)	1.394(3)	C(23)–C(24)	1.394(4)
C(10)–C(11)	1.385(4)	C(24)–C(25)	1.398(4)
C(11)–C(12)	1.388(4)	C(25)–C(26)	1.376(4)
C(12)–C(13)	1.387(4)	C(26)–C(27)	1.390(4)
C(10)–C(14)	1.516(4)	C(24)–C(28)	1.498(4)
S(1)–Ni(1)–S(2)	88.79(2)	S(3)–Ni(2)–S(4)	89.18(2)
S(1)′–Ni(1)–S(2)′	88.79(2)	S(3)″–Ni(2)–S(4)″	89.18(2)
S(1)–Ni(1)–S(1)′	180.00	S(3)–Ni(2)–S(3)″	180.00
S(2)–Ni(1)–S(2)′	180.00	S(4)–Ni(2)–S(4)″	180.00
S(1)′–Ni(1)–S(2)	91.21(2)	S(3)″–Ni(2)–S(4)	90.82(2)
S(1)–Ni(1)–S(2)′	91.21(2)	S(3)–Ni(2)–S(4)″	90.82(2)
Ni(1)–S(1)–P(1)	82.95(3)	Ni(2)–S(3)–P(2)	82.41(3)
Ni(1)–S(2)–P(1)	83.47(3)	Ni(2)–S(4)–P(2)	82.95(3)
S(1)–P(1)–S(2)	104.18(4)	S(3)–P(2)–S(4)	104.80(4)
O(1)–P(1)–O(2)	100.20(9)	O(3)–P(2)–O(4)	99.83(9)
O(1)–P(1)–S(1)	113.01(7)	O(3)–P(2)–S(3)	113.63(7)
O(1)–P(1)–S(2)	115.74(7)	O(3)–P(2)–S(4)	115.05(7)
O(2)–P(1)–S(2)	108.70(7)	O(4)–P(2)–S(4)	108.64(7)
O(2)–P(1)–S(1)	115.39(7)	O(4)–P(2)–S(3)	115.17(7)
C(1)–O(1)–P(1)	120.9(1)	C(15)–O(3)–P(2)	119.9(1)
C(8)–O(2)–P(1)	124.9(1)	C(22)–O(4)–P(2)	123.1(2)
C(2)–C(1)–C(6)	122.9(2)	C(16)–C(15)–C(20)	122.7(2)
C(6)–C(1)–O(1)	118.5(2)	C(20)–C(15)–O(3)	118.5(2)
C(2)–C(1)–O(1)	118.6(2)	C(16)–C(15)–O(3)	118.7(2)
C(1)–C(2)–C(3)	119.4(2)	C(15)–C(16)–C(17)	119.3(2)
C(2)–C(3)–C(4)	118.3(2)	C(16)–C(17)–C(18)	118.4(2)
C(3)–C(4)–C(5)	121.3(2)	C(17)–C(18)–C(19)	121.2(2)
C(4)–C(5)–C(6)	120.1(2)	C(18)–C(19)–C(20)	120.3(2)
C(5)–C(6)–C(1)	117.9(2)	C(19)–C(20)–C(15)	118.0(2)
C(2)–C(3)–C(7)	120.1(2)	C(16)–C(17)–C(21)	120.2(2)
C(4)–C(3)–C(7)	121.5(2)	C(18)–C(17)–C(21)	121.4(2)
C(9)–C(8)–C(13)	122.7(2)	C(23)–C(22)–C(27)	122.9(2)
C(13)–C(8)–O(2)	119.6(2)	C(27)–C(22)–O(4)	120.0(2)
C(9)–C(8)–O(2)	117.5(2)	C(23)–C(22)–O(4)	117.0(2)
C(8)–C(9)–C(10)	119.6(2)	C(22)–C(23)–C(24)	120.0(2)
C(9)–C(10)–C(11)	118.3(2)	C(23)–C(24)–C(25)	117.5(3)
C(10)–C(11)–C(12)	121.1(2)	C(24)–C(25)–C(26)	121.6(3)
C(11)–C(12)–C(13)	120.7(2)	C(25)–C(26)–C(27)	120.8(3)
C(12)–C(13)–C(8)	117.6(2)	C(26)–C(27)–C(22)	117.2(2)
C(9)–C(10)–C(14)	120.0(2)	C(23)–C(24)–C(28)	120.6(3)
C(11)–C(10)–C(14)	121.6(2)	C(25)–C(24)–C(28)	121.9(3)

[a] Symmetry equivalent positions ($x+1$, $-y+1$, $-z$) given by a prime and ($-x$, $-y$, $-z$) by a double prime.

3.3. X-ray diffraction analysis

A translucent purple, block crystal of $Ni[S_2P(OC_6H_4\text{-}Me\text{-}o)_2]_2$, a brown/purple, slab crystal of $NiS_2[P(OC_6H_4\text{-}Me\text{-}m)_2]_2$ and a pink/purple block crystal of $Ni[S_2P(OCH_2\text{-}Ph)_2]_2$ were mounted on glass fibres. Data were collected on a Bruker-Nonius Kappa CCD area detector diffractometer, with ϕ and ω scans chosen to give a complete asymmetric unit. Cell refinement [35] gave cell constants corresponding to a monoclinic for **1** and triclinic cells for **2** and **5** whose dimensions are given in Table 2 along with other experimental parameters. An absorption correction was applied [36].

The structure was solved by direct methods [37] and was refined using the WINGX version [38] of SHELXL97 [39]. All of the non-hydrogen atoms were treated anisotropically. Hydrogen atoms were included in idealized positions with isotropic thermal parameters set at 1.2 times that of the carbon atom to which they were attached. The final cycle

Table 5
Bond lengths (Å) and angles (°) for $Ni[S_2P(OCH_2Ph)_2]_2$[a]

Molecule 1				Molecule 2	
Ni(1)–S(1)	2.233(1)	Ni(1)–S(3)	2.227(1)	Ni(2)–S(5)	2.236(1)
Ni(1)–S(2)	2.2343(8)	Ni(1)–S(4)	2.2469(8)	Ni(2)–S(6)	2.2313(8)
S(1)–P(1)	1.9900(9)	S(3)–P(2)	1.9934(9)	S(5)–P(3)	2.0026(9)
S(2)–P(1)	1.999(1)	S(4)–P(2)	1.993(1)	S(6)–P(3)	1.996(1)
P(1)–O(1)	1.583(2)	P(2)–O(3)	1.577(2)	P(3)–O(5)	1.575(2)
P(1)–O(2)	1.571(2)	P(2)–O(4)	1.583(2)	P(3)–O(6)	1.579(2)
O(1)–C(1)	1.471(3)	O(3)–C(15)	1.475(3)	O(5)–C(29)	1.472(3)
O(2)–C(8)	1.466(3)	O(4)–C(22)	1.464(2)	O(6)–C(36)	1.475(3)
C(1)–C(2)	1.505(3)	C(15)–C(16)	1.502(3)	C(29)–C(30)	1.498(3)
C(2)–C(7)	1.385(3)	C(16)–C(21)	1.382(3)	C(30)–C(31)	1.383(3)
C(2)–C(3)	1.387(3)	C(16)–C(17)	1.390(3)	C(30)–C(35)	1.385(3)
C(3)–C(4)	1.390(3)	C(17)–C(18)	1.390(3)	C(31)–C(32)	1.386(3)
C(4)–C(5)	1.383(4)	C(18)–C(19)	1.381(3)	C(32)–C(33)	1.388(4)
C(5)–C(6)	1.384(4)	C(19)–C(20)	1.381(4)	C(33)–C(34)	1.374(4)
C(6)–C(7)	1.385(3)	C(20)–C(21)	1.391(3)	C(34)–C(35)	1.391(4)
C(8)–C(9)	1.507(3)	C(22)–C(23)	1.498(3)	C(36)–C(37)	1.494(3)
C(9)–C(10)	1.388(3)	C(23)–C(28)	1.388(3)	C(37)–C(38)	1.392(3)
C(9)–C(14)	1.388(3)	C(23)–C(24)	1.393(3)	C(37)–C(42)	1.394(3)
C(10)–C(11)	1.389(3)	C(24)–C(25)	1.391(3)	C(38)–C(39)	1.384(3)
C(11)–C(12)	1.380(4)	C(25)–C(26)	1.382(3)	C(39)–C(40)	1.389(4)
C(12)–C(13)	1.388(4)	C(26)–C(27)	1.382(3)	C(40)–C(41)	1.379(4)
C(13)–C(14)	1.385(3)	C(27)–C(28)	1.391(3)	C(41)–C(42)	1.388(3)
S(1)–Ni(1)–S(2)	88.52(3)	S(3)–Ni(1)–S(4)	88.00(3)	S(5)–Ni(2)–S(6)	87.95(3)
S(1)–Ni(1)–S(3)	178.39(2)	S(2)–Ni(1)–S(4)	178.24(2)	S(5)–Ni(2)–S(5)″	180.00
S(1)–Ni(1)–S(4)	92.86(3)	S(2)–Ni(1)–S(3)	90.66(3)	S(5)–Ni(2)–S(6)″	92.05(3)
Ni(1)–S(1)–P(1)	84.10(3)	Ni(1)–S(3)–P(2)	84.61(3)	Ni(2)–S(5)–P(3)	84.73(3)
Ni(1)–S(2)–P(1)	83.86(3)	Ni(2)–S(4)–P(2)	84.09(3)	Ni(2)–S(6)–P(3)	85.00(3)
S(1)–P(1)–S(2)	102.81(4)	S(3)–P(2)–S(4)	102.44(4)	S(5)–P(3)–S(6)	101.73(4)
O(1)–P(1)–O(2)	101.04(9)	O(3)–P(2)–O(4)	95.22(8)	O(5)–P(3)–O(6)	96.56(9)
O(1)–P(1)–S(1)	115.07(7)	O(3)–P(2)–S(3)	114.76(7)	O(5)–P(3)–S(5)	115.38(7)
O(1)–P(1)–S(2)	112.30(7)	O(3)–P(2)–S(4)	114.12(7)	O(5)–P(3)–S(6)	115.89(7)
O(2)–P(1)–S(2)	115.14(7)	O(4)–P(2)–S(4)	115.83(7)	O(6)–P(3)–S(6)	114.23(7)
O(2)–P(1)–S(1)	111.00(7)	O(4)–P(2)–S(3)	115.19(7)	O(6)–P(3)–S(5)	113.82(7)
C(1)–O(1)–P(1)	118.9(1)	C(15)–O(3)–P(2)	119.2(1)	C(29)–O(5)–P(3)	121.2(1)
C(8)–O(2)–P(1)	121.9(1)	C(22)–O(4)–P(2)	120.7(1)	C(36)–O(6)–P(3)	119.4(1)
O(1)–C(1)–C(2)	108.1(2)	O(3)–C(15)–C(16)	107.4(2)	O(5)–C(29)–C(30)	109.9(2)
C(7)–C(2)–C(3)	119.3(2)	C(21)–C(16)–C(17)	119.4(2)	C(31)–C(30)–C(35)	119.4(2)
C(7)–C(2)–C(1)	119.8(2)	C(21)–C(16)–C(15)	119.6(2)	C(31)–C(30)–C(29)	120.1(2)
C(3)–C(2)–C(1)	120.8(2)	C(17)–C(16)–C(15)	121.0(2)	C(35)–C(30)–C(29)	120.4(2)
C(2)–C(3)–C(4)	120.5(2)	C(18)–C(17)–C(16)	120.5(2)	C(30)–C(31)–C(32)	120.5(2)
C(5)–C(4)–C(3)	119.7(2)	C(19)–C(18)–C(17)	119.6(2)	C(31)–C(32)–C(33)	119.9(3)
C(4)–C(5)–C(6)	120.1(2)	C(18)–C(19)–C(20)	120.2(2)	C(34)–C(33)–C(32)	119.9(2)
C(5)–C(6)–C(7)	120.1(2)	C(19)–C(20)–C(21)	120.0(2)	C(33)–C(34)–C(35)	120.2(3)
C(2)–C(7)–C(6)	120.3(2)	C(16)–C(21)–C(20)	120.2(2)	C(30)–C(35)–C(34)	120.1(2)
O(2)–C(8)–C(9)	108.7(2)	O(4)–C(22)–C(23)	108.6(2)	O(6)–C(36)–C(37)	108.3(2)
C(10)–C(9)–C(14)	119.1(2)	C(28)–C(23)–C(24)	118.7(2)	C(38)–C(37)–C(42)	118.7(2)
C(10)–C(9)–C(8)	120.3(2)	C(28)–C(23)–C(22)	120.0(2)	C(38)–C(37)–C(36)	119.8(2)
C(14)–C(9)–C(8)	120.6(2)	C(24)–C(23)–C(22)	121.3(2)	C(42)–C(37)–C(36)	121.5(2)
C(9)–C(10)–C(11)	120.4(2)	C(25)–C(24)–C(23)	120.4(2)	C(39)–C(38)–C(37)	120.8(2)
C(12)–C(11)–C(10)	120.4(2)	C(26)–C(25)–C(24)	120.1(2)	C(38)–C(39)–C(40)	119.8(2)
C(11)–C(12)–C(13)	119.4(2)	C(27)–C(26)–C(25)	120.1(2)	C(41)–C(40)–C(39)	120.1(2)
C(14)–C(13)–C(12)	120.4(3)	C(26)–C(27)–C(28)	119.6(2)	C(40)–C(41)–C(42)	120.1(2)
C(13)–C(14)–C(9)	120.4(2)	C(23)–C(28)–C(27)	121.1(2)	C(41)–C(42)–C(37)	120.5(2)

[a] Symmetry equivalent positions $(-x+1, -y, -z+1)$ given by a double prime and equivalent distances and angles not listed are Ni(2)–S(5)″ 2.236(1), Ni(2)–S(6)″ 2.2313(8) Å; S(6)″–Ni(2)–S(5)″ 87.95(3)°, S(6)–Ni(2)–S(6)″ 180.00°, S(6)–Ni(2)–S(5)″ 92.05(3)°.

of full-matrix least-squares refinement was based on 6621 (for **1**), 6973 (for **2**) and 10 182 (for **5**), observed reflections (5515 (for **1**), 5651 (for **2**) and 7969 (for **5**) for $F^2 > 4\sigma(F^2)$) and 356 (for **1**), 355 (for **2**) and 571 (for **5**) variable parameters and converged (largest parameter shift was 0.001 times its esd).

Selected distances and bond angles are given in Tables 3–5 for molecules **1**, **2** and **5**, respectively, and ORTEP diagram are displayed in Figs. 1–3.

4. Concluding comments

We have successfully prepared and characterized the following complexes, $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$, $Ni[S_2P(OC_6H_4CH_3\text{-}p)_2]_2$, $Ni[S_2P(OC_6H_5)_2]_2$ and $Ni[S_2P(OCH_2Ph)_2]_2$. The IR, UV–Vis and ^1H NMR spectra of all of these nickel complexes are consistent with the proposed structure. The molecular structures of $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$ and $Ni[S_2P(OCH_2Ph)_2]_2$ were all determined to have a distorted square-planar environment around nickel, as has been found for the structures reported for the analogues, complexes $Ni[S_2P(OEt)_2]_2$ and $Ni[S_2P(OPr^i)_2]_2$.

5. Supplementary material

Additional material available from the Cambridge Crystallographic Data Centre (CCDC Nos. 280426, 280427, and 280706 for $Ni[S_2P(OC_6H_4Me\text{-}o)_2]_2$, $Ni[S_2P(OC_6H_4Me\text{-}m)_2]_2$, and $Ni[S_2P(OCH_2Ph)_2]_2$, respectively, comprises the final atomic coordinates for all atoms, thermal parameters, and a complete listing of bond distances and angles). Copies of this information may be obtained free of charge on application to The Director, 12 Union Road, Cambridge CB2 2EZ, UK (fax: +44 1223 336033; email: deposit@ccdc.cam.ac.uk or http://www.ccdc. cam.ac.uk).

Acknowledgements

One of us (M.N.) is grateful to CSIR, New Delhi for financial support. M.E.L. thanks the UK Engineering and Physical Sciences Council for support of the X-ray facilities at Southampton University. J.E.D. wishes to thank the University of Windsor for financial support. We are thankful to RSIC, CDRI, Lucknow for the spectral analyses.

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