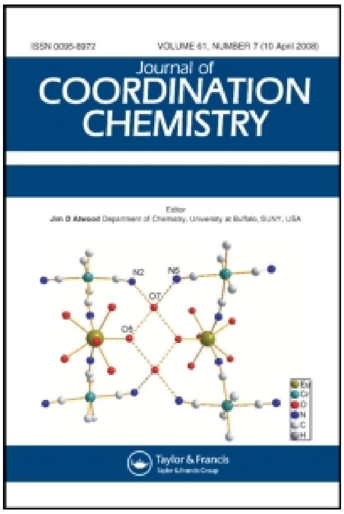

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Nickel(II) di(pentyl)dithiocarbamates with P ligands

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To cite this Article Pastorek, Richard , Kameníček, Jiří , Cvek, Boris , Pavlíček, Marek , Šindelář, Zdeněk and Žák, Zdirad(2003) 'Nickel(II) di(pentyl)dithiocarbamates with P ligands', Journal of Coordination Chemistry, 56: 13, 1123 — 1129

To link to this Article: DOI: 10.1080/0095897031000135397

URL: http://dx.doi.org/10.1080/0095897031000135397

J. Coord. Chem., Vol. 56, No. 13, 10 September 2003, pp. 1123–1129

NICKEL(II) DI(PENTYL)DITHIOCARBAMATES WITH P LIGANDS

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(Received 13 January 2003; In final form 3 April 2003)

Ni(II) di(pentyl)dithiocarbamates of composition [Ni(Pe$_2$dtc)$_2$], [NiX(Pe$_2$dtc)(PPh$_3$)] (X = Cl, Br, I, NCS), [Ni(NCS)(Pe$_2$dtc)(PBut$_3$)], [Ni(Pe$_2$dtc)(PPh$_3$)$_2$]ClO$_4$ and [Ni(Pe$_2$dtc)(PPh$_3$)$_2$]PF$_6$ (Pe$_2$dtc = di(pentyl)dithiocarbamate, PPh$_3$ = triphenylphosphine, PBut$_3$ = tributylphosphine) have been synthesized. The complexes have been characterized by the usual methods. X-ray structure analyses confirmed the nature of [NiI(Pe$_2$dtc)(PPh$_3$)] and [Ni(Pe$_2$dtc)(PPh$_3$)$_2$]ClO$_4$ complexes.

Keywords: Nickel(II) di(pentyl)dithiocarbamates; Synthesis; X-ray structure analysis

INTRODUCTION

Recently, some symmetrical Ni(II) dithiocarbamates with P ligands in the coordination sphere were reported including [NiX(R$_2$dtc)(PR$'_3$)] (X = Cl, Br, I, NCS; R = methyl, ethyl, butyl, hydroxyethyl; R$'$ = phenyl, ethyl, butyl; dtc = S$_2$CN$^-$) [1–7]. X-ray structure analyses of [NiCl(Et$_2$dtc)(PEt$_3$)] [7], [NiCl(Et$_2$dtc)(PPh$_3$)] [5,8], and [Ni(NCS)(But$_2$dtc)(PPh$_3$)] [6], confirmed distorted square coordination of NiS$_2$PX (X = Cl, N). Complexes containing the NiS$_2$P$_2$ chromophore, [Ni(R$_2$dtc)(PPh$_3$)$_2$]X (X = PF$_6$, ClO$_4$; R = methyl, ethyl, isopropyl, butyl, hydroxyethyl, 2-phthalimido-ethyl) were also studied [2,4,9,10]. X-ray structures of [Ni(Et$_2$dtc)(PPh$_3$)$_2$]ClO$_4$ (Et = ethyl) [4], [Ni(*i*-Pr$_2$dtc)(PPh$_3$)$_2$]ClO$_4$ $\cdot$ CHCl$_3$ (*i*-Pr = isopropyl) [9], and [Ni(Pie$_2$dtc)(PPh$_3$)$_2$]ClO$_4$ $\cdot$ CH$_3$OH $\cdot$ H$_2$O (Pie = 2-phthalimido-ethyl) [10], show that in these cases nickel has a distorted square arrangement. There are no literature data for nickel(II) di(pentyl)-dithiocarbamates with P ligands. The aim of this work was the synthesis of

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ISSN 0095-8972 print: ISSN 1029-0389 online © 2003 Taylor & Francis Ltd
DOI: 10.1080/0095897031000135397

[NiX(Pe_2dtc)(PR_3)] and [Ni(Pe_2dtc)($(PPh_3)_2$)]X complexes and comparison of the influence of donor atoms and substituents on structure and properties.

EXPERIMENTAL

Syntheses

[Ni(Pe_2dtc)$_2$]

The title complex was prepared by the reaction of CS_2 (50 mmol) with dipentylamine (50 mmol) in 96% ethanol (30 cm^3). A warm aqueous solution of $NiCl_2 \cdot 6H_2O$ (25 mmol in 50 cm^3) was added after 15 min. A deep-green substance was formed, and after standing for 1 h it was filtered off, washed with warm water to negative reaction for Cl^- ions and dried under an IR lamp at 40°C (yield 89%).

[NiX(Pe_2dtc)(PR_3)] (X = Cl, Br, I, NCS; R = phenyl, butyl)

The compounds were isolated by the reaction of a suspension of finely powdered complexes [Ni(Pe_2dtc)$_2$] (1 mmol) and [$NiX_2(PR_3)_2$] (1 mmol) [11], in chloroform (10 cm^3). The mixture was thoroughly stirred and after filtration, the violet solutions (in the case [Ni(NCS)(Pe_2dtc)($PBut_3$)] the red solution) were left to crystallize at room temperature. For [Ni(NCS)(Pe_2dtc)(PR_3)] complexes, the compounds were washed with *n*-hexane; for the remaining compounds the products were dried and powdered. Complexes with $PBut_3$ did not give solid products. All compounds were dried under an IR-lamp at 40°C except for [Ni(NCS)(Pe_2dtc)($PBut_3$)], which was placed in a refrigerator. Single crystals suitable for X-ray analysis were obtained by recrystallization from *n*-hexane. Yields were about 35–45%, owing to partial solubility in *n*-hexane.

[Ni(Pe_2dtc)(PPh_3)$_2$]X (X = ClO_4, PF_6)

A suspension of finely powdered [Ni(Pe_2dtc)$_2$] (1 mmol), PPh_3 (2 mmol) and $NiCl_2 \cdot 6H_2O$ (1 mmol) in 25 cm^3 of methanol was stirred under reflux. After 1 h, powdered $LiClO_4 \cdot 3H_2O$ (1 mmol) or K[PF_6] (1 mmol) was added and the mixture was refluxed for 1 h. For PF_6^- a red substance appeared; for ClO_4^- filtration was necessary and a red solid product was obtained in three days. Both compounds were washed with diethyl ether and dried under an IR lamp at 40°C. Yields: [Ni(Pe_2dtc)(PPh_3)$_2$]ClO_4 84%, [Ni(Pe_2dtc)(PPh_3)$_2$]PF_6 90%. Single crystal suitable for X-ray analysis were obtained from the solution after filtration. No products of this type with $PBut_3$ were formed.

Physical Measurements

Nickel was determined by chelatometric titration [12], and chlorine and bromine were by the Schöniger method [13]. C, H, N and S analyses were performed on an EA 1108 instrument (Fisons). Room temperature magnetic susceptibilities were measured by the Faraday method using Co[Hg$(NCS)_4$] as calibrant on a laboratory-designed instrument employing a Sartorius 4434 MP-8 microbalance. Conductivities were measured with a

LF 330 conductivity meter (WTW GmbH) at 25°C. Diffuse-reflectance electronic absorption spectra (45 000–11 000 cm^{-1}) were measured on a Specord M 40 and IR spectra (4000–400 cm^{-1}) on a Specord M 80 (Carl Zeiss, Jena) using nujol mulls. Thermal analysis was performed on an Exstar 6000 TG/DTA 6200 device (Seiko) with heating rate of 2.5°C/min; sample weight 9.43–16.89 mg, temperature range 20–1150°C.

Crystal Structure Determinations

X-ray data collections were made on a KUMA KM-4 diffractometer with graphite-monochromatized Mo K_α radiation and an area-sensitive CCD-4 detector. Structures were solved by direct methods using SHELXS-86 program [14]. No absorption corrections were applied. The structures were refined anisotropically by full-matrix least-squares procedures using the program SHELXL-97 [15]. Additional calculations were done using the PARST program [16].

RESULTS AND DISCUSSION

Analytical data for all complexes are given in Table I; selected measured properties are summarized in Table II.

All compounds are diamagnetic [Ni(Pe$_2$dtc)$_2$] and [NiX(Pe$_2$dtc)(PR$_3$)] are non-electrolytes, and [Ni(Pe$_2$dtc)(PPh$_3$)$_2$]X behaves as a 1:1 electrolyte [17], in accord with assumption of a square planar arrangement of NiS$_4$, NiS$_2$PX and NiS$_2$P$_2$ chromophores in the coordination sphere. Ionic character of the anion in [Ni(Pe$_2$dtc)-(PPh$_3$)$_2$]X is also confirmed by IR spectroscopy; for ClO_4^- a singlet ν_3 at 1090 cm^{-1} and ν_4 at 630 cm^{-1} [18]; for PF_6^- a single vibration at 830 cm^{-1} was found [19]. [Ni(NCS)(Pe$_2$dtc)(PR$_3$)] complexes exhibit coordination NCS via nitrogen to the nickel atom: ν(C≡N) is in the range 2088–2085 cm^{-1} and ν(C–S) in the range 842–840 cm^{-1} [20]. IR spectra of all compounds contain $\nu(\mathrm{C}\overset{\cdots}{-}\mathrm{N})$ and $\nu(\mathrm{C}\overset{\cdots}{-}\mathrm{S})$ vibrations at 1512–1490 cm^{-1} and 1005–990 cm^{-1} typical of dithiocarbamate complexes [21,22]. Square planar coordination is supported by electronic spectroscopy; bands in the 15 600–25 200 cm^{-1} region are typical of square planar nickel(II) complexes [23, 24]. Bands above 30 000 cm^{-1} are probably connected with intraligand transitions in the S$_2$CN$^-$group [25].

TABLE I Analytical data for the complexes

	Compound	*Found (Calcd)* (%)						
		M_1	*Ni*	*C*	*H*	*N*	*S*	X^a
I	[Ni(Pe$_2$dtc)$_2$]	523.6	11.1 (11.2)	50.8 (50.5)	9.0 (8.5)	5.4 (5.4)	23.9 (24.5)	
II	[NiCl(Pe$_2$dtc)(PPh$_3$)]	588.9	9.8 (10.0)	59.0 (59.2)	6.5 (6.3)	2.4 (2.4)	10.6 (10.9)	5.8 (6.0)
III	[NiBr(Pe$_2$dtc)(PPh$_3$)]	633.3	9.1 (9.3)	54.9 (55.0)	6.1 (5.9)	2.3 (2.2)	9.5 (10.1)	12.2 (12.6)
IV	[NiI(Pe$_2$dtc)(PPh$_3$)]	680.3	8.6 (8.6)	51.1 (51.2)	5.6 (5.5)	2.1 (2.1)	8.9 (9.4)	18.9 (18.6)
V	[Ni(NCS)(Pe$_2$dtc)(PPh$_3$)]	611.5	9.6 (9.6)	58.8 (58.9)	6.3 (6.1)	4.6 (4.6)	15.2 (15.7)	
VI	[Ni(NCS)(Pe$_2$dtc)(PBut$_3$)]	551.5	10.5 (10.6)	52.3 (52.3)	9.5 (9.0)	5.0 (5.1)	17.0 (17.4)	
VII	[Ni(Pe$_2$dtc)(PPh$_3$)$_2$]ClO$_4$	915.1	6.7 (6.4)	61.2 (61.7)	5.6 (5.7)	1.5 (1.5)	6.8 (7.0)	3.6 (3.9)
VIII	[Ni(Pe$_2$dtc)(PPh$_3$)$_2$]PF$_6$	960.7	6.0 (6.1)	58.6 (58.8)	5.6 (5.5)	1.5 (1.5)	6.1 (6.7)	

[a] X = Cl, Br, I.

TABLE II Characteristic data for the complexes

Complex	*Colour*	$\lambda_M{}^{a}$ [$\mathrm{S\,cm^2\,mol^{-1}}$]	*IR* ($\mathrm{cm^{-1}}$)[b]				*UV–Vis* ($10^3\,\mathrm{cm^{-1}}$)	*B*	E_n	E_x	T_t
			$\nu(\mathrm{C}\overset{\cdots}{-}\mathrm{S})$	$\nu(\mathrm{C}\overset{\cdots}{-}\mathrm{N})$				(°C)[c]			
I	dark green	4.5	1005 w	1490 m			15.6; 20.6; 25.2; 31.0	129	93		91
II	violet	4.8	995 w	1510 w			19.0; 30.8	175	92	233	89
III	violet	10.4	995 m	1495 w			18.6; 25.0; 31.0	195	84	217	81
IV	dark violet	8.2	990 m	1500 w			18.4; 24.8; 31.5	188	91	220	88
V	red	4.2	995 w	1495 w	ν(C–S) : $\nu(\mathrm{C{\equiv}N})$:	840 s 2088 s	20.4; 30.0	191	145	224	142
VI	dark red	4.8	1000 m	1512 s	ν(C–S) : $\nu(\mathrm{C{\equiv}N})$:	842 m 2085 vs	20.4; 30.6	133	39	130	36
VII	red	95.3[d]	998 s	1495 w	$\nu_3(\mathrm{ClO_4^-})$:	1090 vs	20.0; 31.2	No study			
					$\nu_4(\mathrm{ClO_4^-})$:	630 vs					
VIII	red	144.2[d]	1005 w	1500 w	$\nu(\mathrm{PF_6^-})$:	830 vs	19.0; 31.4	182		199	210

[a] In nitromethane solution, $[\mathrm{Ni^{2+}}] = 10^{-3}\,\mathrm{mol\,dm^{-3}}$; [b] Maxima in nujol; [c] *B*: beginning of thermal decomposition, E_n: peak of endothermic effect, E_x: peak of exothermic effect, T_t: melting point; [d] In acetone solution.

TABLE III Crystal data and structure refinement details for IV and VII

	[NiI(Pe$_2$dtc)(PPh$_3$)]	*[Ni(Pe$_2$dtc)(PPh$_3$)$_2$]ClO$_4$*
Empirical formula	$C_{29}H_{37}INNiPS_2$	$C_{47}H_{52}ClNNiO_4P_2S_2$
Formula weight	680.30	915.12
Temperature	120(2) K	120(2) K
Wavelength	0.71073 Å	0.71073 Å
Crystal system, space group	Triclinic, $P\bar{1}$	Orthorhombic, *Pbca*
Unit cell dimensions	$a = 14.879(3)$ Å $\alpha = 89.88(3)^\circ$	$a = 16.293(3)$ Å
	$b = 17.317(3)$ Å $\beta = 89.86(3)^\circ$	$b = 15.376(3)$ Å
	$c = 23.586(5)$ Å $\gamma = 74.38(3)^\circ$	$c = 35.673(7)$ Å
Volume	5853(2) Å	8937(3) Å^3
Z, Calculated density	8, 1.544 Mg m^{-3}	8, 1.360 Mg m^{-3}
Absorption coefficient	1.934 mm^{-1}	0.703 mm^{-1}
F(000)	2768	3840
Crystal size	0.30 × 0.30 × 0.30 mm	0.40 × 0.30× 0.20 mm
θ range for data collection	3.14° to 28.60°	3.39° to 28.35°
Index ranges	$-19 \le h \le 17$, $-22 \le k \le 23$, $-30 \le l \le 30$	$-21 \le h \le 21$, $-20 \le k \le 12$, $-47 \le l \le 47$
Reflections collected/unique	47356/25610/[*R*(int) = 0.0614]	68611/10544 [*R*(int) = 0.0420]
Completeness to $2\theta = 28.50$	86.3%	87.1%
Max. and min. transmission	0.5946 and 0.5946	0.8772 and 0.7662
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data/restraints/parameters	25610/0/1401	1054/0/523
Goodness-of-fit on F^2	0.930	1.084
Final *R* indices [$I > 2\sigma(I)$]	$R1 = 0.0542$, $wR2 = 0.0802$	$R1 = 0.0445$, $wR2 = 0.0938$
R indices (all data)	$R1 = 0.1273$, $wR2 = 0.1003$	$R1 = 0.0712$, $wR2 = 0.1070$
Largest diff. peak and hole	0.825 and -0.573 e · Å^{-3}	0.634 and -0.445 e · Å^{-3}

Thermal decomposition of the complexes starts between 129 and 195°C (complex VII was not studied for safety reasons) and melting is accompanied by endothermic effects in the range 39–145°C. No thermally stable intermediates were observed. Exothermic effects at 130–233°C were recorded for complexes with P ligands, due to oxygen insertion in the Ni–P bond [26]. In these cases (III, IV, VI), a mass increase on the TG curve appeared. Similar results were recently described for non-symmetrically substituted [Ni(R$_1$R$_2$dtc)(PPh$_3$)$_2$]X complexes [27].

The X-ray structures of two compounds [NiI(Pe$_2$dtc)(PPh$_3$)] and [Ni(Pe$_2$dtc)(PPh$_3$)$_2$]ClO$_4$ (see Table III, Fig. 1 and 2) confirmed previous conclusions concerning slightly distored square planar coordination of nickel. This is apparent in different values of Ni–S, Ni–P and Ni–I bond lengths and associated angles (see Table IV). The Ni(1)–S(2) distance for IV is significantly shorter than Ni(1)–S(1). This was described recently for non-symmetrical dithiocarbamate complexes [6]. Deviation of the chromophore atoms from least-squares planes are as follows:

NiS$_2$PI–Ni(1) 0.42(1); S(1) 0.21(3); S(2) 0.21(3); P(1) 0.06(3), I(1) 0.01(1) Å;
NiS$_2$P$_2$–Ni(1) 0.00(1); S(1) 0.02(1); S(2) 0.01(1); P(1) 0.01(1); P(2) 0.02(1) Å.

As expected, the NiS$_2$P$_2$ chromophore is more planar than NiS$_2$PI. A significant part of π bonds in C(1)–N(1), C(1)–S(1) and C(1)–S(2) bonds was found (see Table IV). For both structures, the bond lengths are shorter than published values for simple σ(C–N) and σ(C–S) bonds (1.47 and 1.81 Å) [28]. The ClO_4^- group in VII is not in the coordination sphere; Ni(1)–Cl(1) = 10.229(2) Å. X-ray structure analyses confirmed that the type of PR$_3$ or anionic ligand affects some of the properties of these complexes but no significant influence on the coordination polyhedron was found.

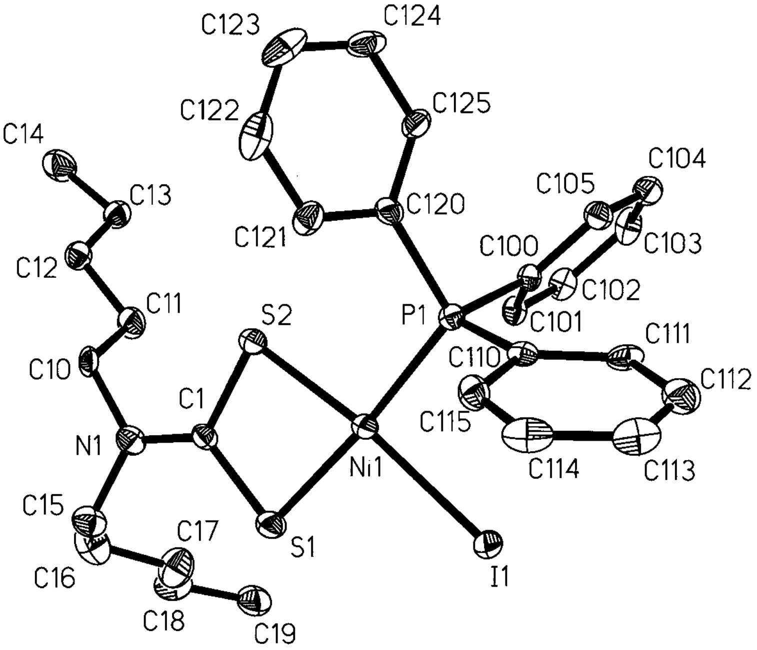

FIGURE 1 ORTEP drawing of [NiI(Pe_2dtc)(PPh_3)] with the atom labelling scheme. Atoms are drawn with 40% probability ellipsoids.

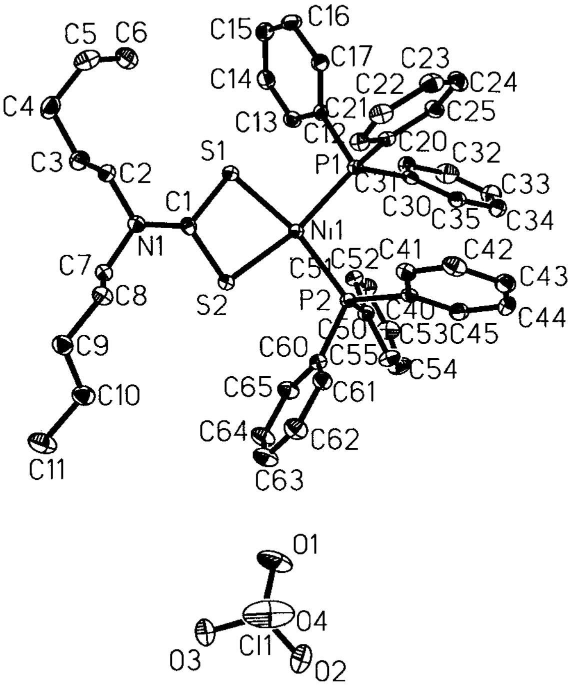

FIGURE 2 ORTEP drawing of [Ni(Pe_2dtc)(PPh_3)$_2$]ClO_4 with the atom labelling scheme. Atoms are drawn with 40% probability ellipsoids.

TABLE IV Selected bond lengths [Å] and angles [°] for IV and VII

$[NiI(Pe_2dtc)(PPh_3)]$				$[Ni(Pe_2dtc)(PPh_3)_2]ClO_4$			
Ni(1)–S(2)	2.1770(19)	S(2)–Ni(1)–P(1)	96.75(7)	Ni(1)–S(2)	2.2105(7)	S(2)–Ni(1)–P(2)	89.80(3)
Ni(1)–P(1)	2.1919(18)	S(2)–Ni(1)–S(1)	78.87(7)	Ni(1)–P(2)	2.2134(7)	S(2)–Ni(1)–S(1)	78.35(3)
Ni(1)–S(1)	2.1987(18)	P(1)–Ni(1)–S(1)	174.79(7)	Ni(1)–S(1)	2.2226(8)	P(2)–Ni(1)–S(1)	168.07(3)
Ni(1)–I(1)	2.4927(12)	S(2)–Ni(1)–I(1)	166.96(5)	Ni(1)–P(1)	2.2464(7)	S(2)–Ni(1)–P(1)	169.10(3)
S(1)–C(1)	1.703(6)	P(1)–Ni(1)–I(1)	93.67(6)	S(1)–C(1)	1.724(2)	P(2)–Ni(1)–P(1)	101.10(3)
S(2)–C(1)	1.716(6)	S(1)–Ni(1)–I(1)	91.06(6)	S(2)–C(1)	1.726(3)	S(1)–Ni(1)–P(1)	90.77(3)
C(1)–N(1)	1.313(7)	C(1)–S(1)–Ni(1)	85.9(2)	N(1)–C(1)	1.302(3)	C(1)–S(1)–Ni(1)	86.32(3)
N(1)–C(10)	1.456(7)	C(1)–S(2)–Ni(1)	86.3(2)	N(1)–C(2)	1.483(3)	C(1)–S(2)–Ni(1)	86.66(8)
N(1)–C(15)	1.461(7)	N(1)–C(1)–S(1)	125.9(5)				
C(10)–C(11)	1.513(8)	N(1)–C(1)–S(2)	125.3(5)				

Acknowledgments

The authors would like to thank the Granting Agency of Czech Republic for support of this work (grant no. 203/02/0436).

Supplementary Data

Crystallographic data are deposited in the Cambridge Crystallographic Data Centre, Nos CCDC 197222 and 197223.

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