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# Benzylisopropyldithiocarbamate complexes of nickel with triphenylphosphine in the coordination sphere

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### Abstract

Nickel(II) benzylisopropyldithiocarbamate complexes of the composition  $[Ni(bz^{i}Prdtc)_{2}]$ ,  $[NiCl(bz^{i}Prdtc)(PPh_{3})] \cdot CHCl_{3}$  and  $[NiX(bz^{i}Prdtc)(PPh_{3})] \cdot (X=Cl, Br, I, NCS; bz=C_{7}H_{7}, {}^{i}Pr=C_{3}H_{7}, dtc=S_{2}CN^{-}, PPh_{3}=triphenylphosphine)$  have been synthesized. The complexes have been characterized by elemental analyses, IR and electronic spectroscopies, thermal analysis, magnetochemical and conductivity measurements. A single crystal X-ray analysis of the  $[NiCl(bz^{i}Prdtc)(PPh_{3})] \cdot CHCl_{3}$ ,  $[NiBr(bz^{i}Prdtc)(PPh_{3})]$  and  $[NiI(bz^{i}Prdtc)(PPh_{3})]$  confirms four-coordinated nickel in a distorted square-planar arrangement with the NiS<sub>2</sub>PX chromophore. © 1999 Elsevier Science Ltd. All rights reserved.

Keywords: Nickel(II) benzylisopropyldithiocarbamate complexes; Synthesis; Study; X-ray structure

## 1. Introduction

Recently, the unsymmetrical Ni(II) dithiocarbamate complexes of the type  $[Ni(NO)(HRdtc)(PPh_3)]$  (R=Me, Et. Pr. Bu. Ph, p-ClPh, p-MeOPh [1], [Ni(NCS)(HRdtc)(PPh<sub>3</sub>)] (R = Et,[2], Bu) [NiX(HRdtc)(PPh<sub>3</sub>)] (X=Cl, Br, I, R=Me, Et, Pr, Bu, Ph, p-ClPh, p-MeOPh [3] and  $[Ni(NO_2)(HRdtc)(PBu_3)]$  (R= Me, Et, <sup>1</sup>Pr, <sup>s</sup>Bu) were studied [4]. However, no information about the complexes of the  $[NiX(R_1R_2dtc)(PPh_3)]$  type were found in the literature. Therefore, the aim of this work was detailed study of the coordination compounds of composition [NiX(bz<sup>1</sup>Prdtc)(PPh<sub>3</sub>)], including the influence of X-donor atom on the structure and properties of the complexes.

# 2. Experimental

### 2.1. Materials

N-benzylisopropylamine (97%) was purchased from

LANCASTER Co., nitromethane and triphenylphosphine from FLUKA Co., the remaining reagents from LA-CHEMA Co. All reagents were of p.a. purity.

### 2.2. Syntheses

## 2.2.1. Preparation of [Ni(bz<sup>i</sup>Prdtc)<sub>2</sub>]

The compound was prepared by the reaction of  $CS_2$  (50 mmol) with *N*-benzylisopropylamine (50 mmol) in EtOH (25 cm<sup>3</sup>). A warm water solution of NiCl<sub>2</sub>·6H<sub>2</sub>O (25 mmol in 50 cm<sup>3</sup>) was added after 15 min. The deep-green substance was formed and after standing for 1 h it was filtered off, washed by warm water until negative reaction on Cl<sup>-</sup> ions and dried under an IR lamp at 40°C (nearly quantitative yield).

# 2.2.2. Preparation of $[NiX(bz^iPrdtc)(PPh_3)]$ (X=Cl, Br, I, NCS)

These compounds were synthesized by the reaction of suspension of fine pulverized complexes  $[Ni(bz^{i}Prdtc)_{2}]$  (1 mmol) and  $[NiX_{2}(PPh_{3})_{2}]$  (1 mmol) [5] in CHCl<sub>3</sub> (25 cm<sup>3</sup>). The mixture was stirred till all components were dissolved. After filtration, an Et<sub>2</sub>O was added to creation a slight turbidity. Then, the solution was left to crystallize at

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room temperature. The formed crystals were filtered, washed by  $Et_2O$  and dried under an IR lamp at 40°C (yield 76–80%). Single crystals suitable for X-ray analysis were obtained by recrystallization from a  $CHCl_3/Et_2O$  mixture (5:1).

### 2.2.3. Physical measurements

The content of nickel was estimated by the chelatometric titration on murexid as an indicator, while chlorine and bromine were determined according Schöniger's method [6]. The elemental analyses (C, H, N, S) were performed on an EA 1108 instrument (FISONS). All physico-chemical measurements were carried out using the same devices as in the previous work [7].

# 2.3. X-ray crystallography

X-ray measurements were performed on a KUMA KM-4 diffractometer with graphite monochromated Mo K<sub> $\alpha$ </sub> radiation ( $\lambda$ =0.71073 Å) using an  $\omega/2\theta$  scan technique. The structures were solved by the direct method using the SHELX-97 program [8]. The structures were refined anisotropically by the full-matrix least-square procedure on  $F^2$  using SHELXL-97 program [9]. Most of H-atoms were found from differential Fourier maps and they were refined isotropically. Additional calculations were made using

Table 1

Crystal data and structure refinement for [NiX(bz<sup>i</sup>Prdtc)(PPh<sub>3</sub>)], X=Cl, Br, I

PARST95 program [10]. All of important crystallographic parameters are summarised in Table 1.

### 3. Results and discussion

Composition and analytical data for all complexes are given in Table 2, and the important results of physico– chemical studies are shown in Table 3.

All prepared compounds are diamagnetic and behave as complex non-electrolytes [11]. Slightly increased values of molar conductivities for [NiX(bz<sup>i</sup>Prdtc)(PPh<sub>3</sub>)] could be explain as a consequence of partial dissociation in the solvent:

 $[NiX(bz^{i}Prdtc)(PPh_{3})] \rightleftharpoons [Ni(bz^{i}Prdtc)(PPh_{3})]^{+} + X^{-}$ 

From Table 3, it follows that this dissociation increases in the order NCS <Cl<Br<I. The results of magnetochemical and conductivity measurements show that nickel atom is coordinated in the square-planar arrangement with NiS<sub>4</sub> chromophore in the case of the complex 1 and NiS<sub>2</sub>PX in remaining complexes **2–6**. This assumption is supported by results of the electron spectroscopy; the strong bands in the 15 700–20 500 cm<sup>-1</sup> region can be attributed to the d–d electron transitions for Ni(II) squareplanar complexes [4,12]. The maxima between 28 700– 40 000 cm<sup>-1</sup> are connected with intraligand transitions in

Molecular formula	C <sub>30</sub> H <sub>30</sub> Cl <sub>4</sub> NNiPS <sub>2</sub>	C <sub>29</sub> H <sub>29</sub> BrNNiPS <sub>2</sub>	C <sub>29</sub> H <sub>29</sub> INNiPS <sub>2</sub>
Formula weight	700.15	625.24	672.23
Temperature (K)	295(2)	291(2)	293(2)
Wavelength (Å)	0.71073	0.71073	0.71073
Crystal system	Monoclinic	Triclinic	Monoclinic
Space group	$P2_1/a$	PĪ	$P2_1/c$
Unit cell dimensions	a = 11.975(2)	a = 10.1391(13)	a = 8.969(2)
(Å, °)	b = 10.001(2)	b = 10.851(2)	b = 19.356(4)
	c = 26.698(5)	c = 14.105(2)	c = 16.960(3)
	a = 90	a = 109.08(2)	a = 90
	$\beta = 96.080(3)$	$\beta = 91.512(12)$	$\beta = 96.46(3)$
	$\gamma = 90$	$\gamma = 105.52(2)$	$\gamma = 90$
Volume ( $Å^3$ ); Z	3179(2); 4	1402.0(4); 2	2925.6(10); 4
Crystal size (mm)	$0.25 \times 0.20 \times 0.07$	$0.70 \times 0.25 \times 0.20$	$0.4 \times 0.4 \times 0.3$
$\theta$ range for data collection (°)	3.6 to 25.5	1.5 to 27.6	1.6 to 25.1
Index ranges	11 = h = 14	-13 = h = 1	-1 = h = 10
-	-12 = k = 12	-13 = k = 14	-15 = k = 0
	-32 = l = 20	-18 = l = 18	-16 = l = 16
Reflections collected	14714	7429	4625
Independent reflections	5385 [ $R(int) = 0.1124$ ]	6496 [R(int)=0.0238]	3926[R(int)=0.0376]
Data/restraints/parameters	5385/0/374	6496/0/432	3926/0/345
Goodness-of-fit on $F^2$	1.286	1.019	1.027
Final R indices $[I > 2s(I)]$	R1 = 0.0933	R1 = 0.0318	R1 = 0.0302
	wR2 = 0.1933	wR2 = 0.0855	wR2 = 0.0837
R indices (all data)	R1 = 0.1756	R1 = 0.0915	R1 = 0.0449
	wR2 = 0.2281	wR2 = 0.1037	wR2 = 0.0900
Largest peak and hole $(e.\text{\AA}^{-3})$	0.753;-0.722	0.462; -0.591	0.630; -0.358

I ab	le 2			
The	results	of	elemental	analyses

Compound	Ni	С	Н	Ν	S	$\mathbf{X}^{\mathrm{a}}$
Found (calcd.)%						
<b>1</b> , $[Ni(bz^{i}Prdtc)_{2}]$	11.3 (11.6)	51.7 (52.1)	5.5 (5.6)	5.2 (5.5)	24.7 (25.3)	
<b>2</b> , [NiCl(bz <sup>i</sup> Prdtc)(PPh <sub>3</sub> )]	9.8 (10.1)	59.9 (60.0)	5.0 (5.0)	2.1 (2.4)	11.5 (11.0)	5.8 (6.1)
3, [NiCl(bz <sup>i</sup> Prdtc)(PPh <sub>3</sub> )]·CHCl <sub>3</sub>	8.5 (8.4)	51.9 (51.4)	4.3 (4.3)	1.8 (2.0)	9.8 (9.2)	19.8 (20.2)
4, [NiBr(bz <sup>i</sup> Prdtc)(PPh <sub>3</sub> )]	9.7 (9.4)	55.4 (55.7)	5.1 (4.7)	2.2 (2.2)	9.7 (10.3)	13.1 (12.8)
5, [NiI(bz <sup>i</sup> Prdtc)(PPh <sub>3</sub> )]	9.0 (8.7)	52.3 (51.8)	4.7 (4.4)	2.4 (2.1)	9.1 (9.5)	
6, [Ni(NCS)(bz <sup>i</sup> Prdtc)(PPh <sub>3</sub> )]	9.3 (9.7)	59.7 (59.7)	4.6 (4.8)	4.8 (4.6)	15.5 (15.9)	

<sup>a</sup> X = Cl, Br.

**T** 11 0

Table 3 The results of physico-chemical studies<sup>a</sup>

Compound	$\lambda_{_{ m M}}$	$IR^{b} (cm^{-1})$		UV/VIS	T <sup>°</sup> (°C)	Color	
	$(\mathrm{S} \mathrm{cm}^2 \mathrm{mol}^{-1})$	$\nu(C - S)$	<i>ν</i> (C:N)	$(\times 10^{3} \text{ cm}^{-1})$			
1	2.6 <sup>d</sup>	999 w	1540 w	15.7; 24.7; 28.7	80	deep green	
2	6.4 <sup>e</sup>	992 m	1526 m	19.5; 29.1	140	violet	
4	12.5 <sup>e</sup>	988 m	1516 w	19.2; 28.7; 36.8; 37.6	180	violet	
5	13.2 <sup>e</sup>	988 m	1511 m	19.4; 24.4; 29.2; 30.5; 36.8; 37.6	190	violet	
6	4.5 <sup>e</sup>	990 m	1515 w	20.5; 28.6; 36.8	130	red	

<sup>a</sup> Compound **3** is not included (small amount of crystal available).

<sup>b</sup> In nujol.

<sup>c</sup> The beginning of thermal decomposition.

<sup>d</sup> Measured in dimethylformamide solution.

<sup>e</sup> Measured in nitromethane solution.

the S<sub>2</sub>CN<sup>-</sup> group [13]. In the IR spectra of all the complexes maxima at 1540–1511 cm<sup>-1</sup> and at 999–990 cm<sup>-1</sup> were observed. They are characteristic for dithiocarbamates and can be assigned to [ $\nu$ (C $\dots$ N)] and [ $\nu$ C $\dots$ S)], respectively [3,14]. Middle absorption peaks for complex **VI** at 2080 cm<sup>-1</sup> [ $\nu$ (C $\equiv$ N)] and 835 cm<sup>-1</sup> [ $\nu$ (C-S)] may imply the assumption that the NCS group is coordinated to the nickel *via* nitrogen atom [15]. It was recently confirmed by X-ray analysis of similar complexes [Ni(NCS)(Bu<sub>2</sub>dtc)(PPh<sub>3</sub>)] and [Ni(NCS) (plddtc)(PPh<sub>3</sub>)] (Bu=n-C<sub>4</sub>H<sub>9</sub>, pld=C<sub>4</sub>H<sub>8</sub>) [2,16].

The thermal decomposition of  $[Ni(bz^1prdtc)_2]$  starts at 80°C. The TG-curve exhibits a plateau in the interval 410–460°C, which can be probably due to formation of NiS<sub>2</sub> (loss of mass found/calcd. = 73.7/73.8%). The decomposition was not complete till 1000°C. The thermal degradations of remaining complexes **2–6** start at 130–190°C and its thermal stability increases depending on X: NCS < Cl < Br < I. The thermolysis is continual, without transient intermediates.

The results of X-ray analysis of complexes **3**, **4**, and **5** (Tables 4 and 5; Figs. 1–3) confirm previous assumptions of distorted square–planar arrangement of nickel in these compounds. This fact is apparent from the different values of the bond lengths and angles between nickel and coordinated atoms of ligands and from deviations of atoms from LSQ-plane Ni(1)S(1)S(2)P(1)X(1) (Table 6). The relatively high value of R1 in the case **3** can be explained by the poor quality of crystal (it was a great problem to

Table 4											
Selected	bond	lengths	[Å]	for	[NiX(	bz <sup>i</sup> Prdt	c)(PPh <sub>2</sub>	)1 (	X = Cl	Br,	I)

	-	
Cl	Br	I
2.176(3)	2.1851(10)	2.1882(12)
2.195(3)	2.2023(9)	2.2094(12)
2.215(3)	2.2205(8)	2.2087(12)
2.189(3)	2.3153(7)	2.5257(8)
1.722(10)	1.720(3)	1.724(4)
1.714(11)	1.711(3)	1.709(5)
1.822(11)	1.817(3)	1.828(4)
1.819(11)	1.825(3)	1.851(4)
1.817(12)	1.826(3)	1.828(4)
1.326(14)	1.316(4)	1.325(5)
1.498(13)	1.475(4)	1.475(5)
1.472(13)	1.499(4)	1.479(6)
	Cl 2.176(3) 2.195(3) 2.215(3) 2.189(3) 1.722(10) 1.714(11) 1.822(11) 1.819(11) 1.817(12) 1.326(14) 1.498(13) 1.472(13)	Cl         Br           2.176(3)         2.1851(10)           2.195(3)         2.2023(9)           2.215(3)         2.2205(8)           2.189(3)         2.3153(7)           1.722(10)         1.720(3)           1.714(11)         1.711(3)           1.822(11)         1.817(3)           1.819(11)         1.825(3)           1.326(14)         1.316(4)           1.498(13)         1.475(4)           1.472(13)         1.499(4)

obtain crystals suitable for X-ray measurements and many attempts to obtain a crystal of better quality were unsuccessful). In contrast to 4 and 5, the structure of 3 contains chloroform as solvent situated out of the coordination sphere—the chlorine derivative without solvent 2 gives a very poor crystals without a chance of solving the X-ray structure.

As expected, the Ni(1)–X(1) bond lengths increase in order to larger atomic radius in the series Cl < Br < I and the length of Ni(1)–P(1) bond, too, whereas the S(2)Ni(1)X(1) angles are slightly decreasing. Very interesting is the fact that the Ni(1)–S(1) and Ni(1)–S(2) bond lengths are not equivalent. For all three solved structures are the Ni(1)–S(1) distances significantly shorter in com-

Table 5 Selected angles [°] for [NiX(bz<sup>i</sup>Prdtc)(PPh<sub>3</sub>)], (X=Cl, Br, I)

Angle	Cl	Br	Ι
S(1)-Ni(1)-P(1)	95.29(12)	92.14(3)	95.86(5)
S(1)-Ni(1)-S(2)	78.83(12)	78.05(3)	78.13(5)
P(1)-Ni(1)-S(2)	170.18(13)	169.49(3)	173.95(5)
S(1)-Ni(1)-X(1)	171.80(13)	170.87(3)	169.13(3)
P(1)-Ni(1)-X(1)	92.66(12)	96.84(3)	94.96(4)
S(2)-Ni(1)-X(1)	93.02(12)	92.87(3)	91.06(4)
C(1)-S(1)-Ni(1)	86.8(4)	87.47(10)	87.2(2)
C(1)-S(2)-Ni(1)	85.8(4)	86.53(10)	86.92(13)
C(21)-P(1)-C(31)	101.0(5)	105.45(14)	103.1(2)
C(21)-P(1)-C(11)	106.6(5)	103.12(14)	105.9(2)
C(31)-P(1)-C(11)	106.6(5)	103.98(13)	103.0(2)
C(21)-P(1)-Ni(1)	121.3(4)	109.25(10)	115.05(14)
C(31) - P(1) - Ni(1)	115.1(4)	111.84(9)	115.16(12)
C(11) - P(1) - Ni(1)	105.2(4)	121.79(9)	113.22(13)
C(1)-N(1)-C(40)	119.4(9)	120.7(2)	119.7(4)
C(1)-N(1)-C(2)	121.5(9)	119.0(2)	120.7(3)
C(40)-N(1)-C(2)	119.1(9)	119.8(2)	119.6(3)
N(1)-C(1)-S(2)	126.2(8)	126.9(2)	126.9(3)
N(1)-C(1)-S(1)	125.4(8)	125.1(2)	125.3(3)
S(2)-C(1)-S(1)	108.4(6)	107.9(2)	107.7(2)



Fig. 1. Molecular structure of  $[NiCl(bz^iPrdtc)(PPh_3)] \cdot CHCl_3$  with the atom numbering scheme (thermal ellipsoids are drawn at 30% probability level).



Fig. 2. Molecular structure of  $[NiBr(bz^iPrdtc)(PPh_3)]$  with the atom numbering scheme (thermal ellipsoids are drawn at 30% probability level).



Fig. 3. Molecular structure of  $[NiI(bz^iPrdtc)(PPh_3)]$  with the atom numbering scheme (thermal ellipsoids are drawn at 30% probability level).

Table 6

Deviations of atoms from the LSQ Ni(1)S(1)S(2)P(1)X-plane in  $[NiX(bz^{i}Prdtc)(PPh_{3})]$ , (X=Cl, Br, I) [Å]

Atom	Cl	Br	Ι
Ni(1)	0.045(4)	-0.025(5)	0.005(6)
S(1)	0.023(8)	-0.001(14)	-0.029(13)
S(2)	-0.108(8)	0.051(11)	0.021(14)
P(1)	-0.107(9)	0.049(11)	0.012(12)
X(1)	0.012(9)	0.002(6)	-0.003(4)

parison to Ni(1)–S(2) ones (see Table 4). This fact was at first described for dithiocarbamates of the type  $[NiX(R_1R_2dtc)(PPh_3)]$  [2].

A significant deal of  $\pi$ -bonds in C(1)–N(1), C(1)–S(1) and C(1)–S(2) was found. The lengths of the simple  $\sigma$ (N–C) and (C–S) bonds are 1.47 Å and 1.81 Å, respectively [17]. For comparison, the appropriate values are in pyridine 1.339 Å, thiophene 1.718 Å [18]. We have found, that complexes **3**, **4**, **5** form hydrogen bonds between the both S(1), S(2) atoms and C(2), C(40) atoms respectively. Moreover, compound **3** exhibits intramolecular hydrogen bonds between Cl(1) and C(26); Cl(1) and C(50) from solvent molecule. Some intermolecular contacts between Cl(3) and C(22); Cl(4) and C(3) were observed, too (Table 7, interpretation according [19]). This fact can be explained by increased electronegativity of chlorine in comparison to bromine and iodine.

### Supplementary data

Crystallographic data are available from the Cambridge Crystallographic Data Centre (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK on request, quoting the deposition numbers 115882 (for **5**), 115883 (for **4**) and 115884 (for **3**).

Table 7 Hydrogen bonds (distances in Å and angles in °) in  $[NiX(bz^{i}Prdtc)(PPh_{3})]$ ,  $(X=Cl, Br, I)^{a}$ 

D-H	D-A	H A	∠DHA
Cl			
C(2)-H(2)	$C(2) \dots S(1)$	$H(2) \dots S(1)$	$C(2)-H(2)S(1)^{b}$
0.96(22)	3.10(9)	2.59(2)	112.5(1.9)
C(40)-H(40A)	$C(40) \dots S(2)$	$H(40A) \dots S(2)$	$C(40)-H(40A)\dots S(2)^{b}$
1.03(21)	3.08(7)	2.55(8)	111.5(1.7)
C(26)-H(26)	C(26)Cl(1)	H(26)Cl(1)	$C(26)-H(26)Cl(1)^{b}$
0.96(24)	3.44(18)	2.72(3)	131.8(1.2)
C(50)-H(50)	$C(50) \dots Cl(1)$	$H(50) \dots Cl(1)$	$C(50)-H(50)Cl(1)^{b}$
1.10(20)	3.46(22)	2.94(4)	109.4(1.0)
C(3)-H(3B)	$C(3) \dots Cl(4)$	$H(3B) \dots Cl(4)$	$C(3)-H(3B)\ldots Cl(4)^{c}$
0.96(28)	3.71(17)	2.90(38)	142.7(1.4)
C(22)-H(22)	C(22)Cl(3)	H(22)Cl(3)	$C(22)-H(22)Cl(3)^{c}$
1.11(26)	3.76(29)	2.77(11)	147.9(4.4)
Br			
C(2)-H(2)	$C(2) \dots S(1)$	H2S1	$C(2)-H(2)S(1)^{b}$
0.96(28)	3.04(2)	2.50(13)	115.5(3.0)
C(40)-H(40A)	$C(40) \dots S(2)$	H40AS2	$C(40)-H(40A)\dots S(2)^{b}$
0.96(6)	3.10(2)	2.61(26)	111.8(2.8)
Ι			
C(2)-H(2)	$C(2) \dots S1$	H2S1	$C(2)-H(2)S(1)^{b}$
0.90(48)	3.07(1)	2.56(18)	116.9(8.8)
C(40)-H(40B)	$C(40) \dots S2$	H40B S2	$C(40) - H(40B) \dots S(2)^{b}$
0.87(28)	3.08(5)	2.49(34)	125.2(7.6)

<sup>a</sup> D = donor; A = acceptor.

<sup>b</sup> Intramolecular contacts.

<sup>c</sup> Intermolecular contacts.

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