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[1,9-Bis(diphenylphosphino)-3,7-dithianonane]monobromonickel(II) Tetraphenylborate, C₃₁H₃₄BrNiP₂S₂⁺·C₂₄H₂₀B⁻, at 295 K, and [1,9-Bis(diphenylphosphino)-3,7-dithianonane]monoiodonickel(II) Tetraphenylborate, C₃₁H₃₄INiP₂S₂⁺·C₂₄H₂₀B⁻, at 120 K*

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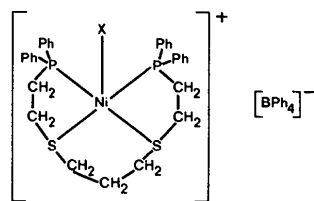
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Abstract. [Ni(C₃₁H₃₄P₂S₂)Br] [B(C₆H₅)₄]: $M_r = 990.53$, Pn , $a = 14.472$ (2), $b = 10.0092$ (9), $c = 16.573$ (2) Å, $\beta = 94.61$ (1)°, $V = 2393$ Å³, $Z = 2$, $D_m = 1.37$, $D_x = 1.375$ Mg m⁻³, $Cu K\alpha$, $\lambda = 1.54056$ Å, $\mu = 3.34$ mm⁻¹, $T = 295$ K, $R = 0.085$, $wR = 0.108$, 3085 unique reflexions. [Ni(C₃₁H₃₄P₂S₂)I] [B(C₆H₅)₄]: $M_r = 1037.53$, Pn , $a = 14.381$ (3), $b = 9.886$ (1), $c = 16.510$ (3) Å, $\beta = 94.31$ (1)°, $V = 2341$ Å³, $Z = 2$, $Mo K\alpha$, $\lambda = 0.71069$ Å, $\mu = 1.23$ mm⁻¹, $T = 120$ K, $R = 0.078$, $wR = 0.100$, 2336 unique reflexions. The isostructural title compounds are built up of discrete [Ni(C₃₁H₃₄P₂S₂)X]⁺ and [B(C₆H₅)₄]⁻ ions, X = Br and I. In the square-pyramidal cations a single ligand molecule, (C₆H₅)₂P(CH₂)₂S(CH₂)₃S(CH₂)₂P(C₆H₅)₂, denoted pssp is coordinated to one Ni *via* the P and S atoms thus forming two five-membered (NiPC₂S) and one six-membered (NiSC₃S) chelate rings. The P and S atoms constitute the basal plane in the square pyramid and the halide atom is situated at the apex. All interatomic distances and angles are normal. [Ni(pssp)Br] [BPh₄] (Ph = C₆H₅) is diamagnetic. [Ni(pssp)I] [BPh₄] is diamagnetic at 120 K but shows temperature-independent paramagnetism at 295 K.

Introduction. Five-coordinated Ni^{II} complexes with linear open-chain ligands, like pssp, are often of square-pyramidal type (Morassi, Bertini & Sacconi, 1973). According to Orioli (1971) the spin state of the Ni²⁺ ion in the complex depends on the electronegativity of the coordinated donor atoms. Complexes with low electronegative donors, such as P and S, are most often diamagnetic. The stereochemical activity of

the filled d_{z^2} orbital of low-spin Ni^{II} causes the Ni–apical-ligand distance in the square pyramid to be longer than analogous Ni–basal-ligand distances. The Ni–basal-ligand distances are comparable with similar four-coordinated square-planar distances. The Ni-atom displacement from the basal plane towards the apical-ligand atom is smaller than in high-spin complexes where it is of the order 0.3–0.4 Å.

The crystal structure of [Ni(pssp)I] [BPh₄] has been determined at 295 K (Aurivillius & Bertinsson, 1980). This compound shows magnetic phase transitions in the temperature interval 80–290 K and there is a colour change from dark red to light brown ~150 K on cooling. The structure determinations of [Ni(pssp)Br] [BPh₄] at 295 K and [Ni(pssp)I] [BPh₄] at 120 K



were undertaken to study possible differences in the coordination polyhedra and in the Ni–ligand distances and angles. This investigation is part of a more general study of complexes between transition-metal halides and substituted phosphines or related ligands.

Experimental. Values of [Ni(pssp)Br] [BPh₄], and for [Ni(pssp)I] [BPh₄] at 120 K in parentheses if they differ, are given. Dark-red crystals in the form of plates, D_m by flotation, 0.12 × 0.22 × 0.12 mm (0.19 × 0.27 × 0.08 mm), Enraf–Nonius CAD-4 diffractometer. $Cu K\alpha$, Ni filter, (Mo $K\alpha$, Zr filter), cell parameters from 49 (50) accurately measured θ values,

* Structures of Complexes between Metal Halides and Phosphinothioethers or Related Ligands. XV.

Lp and absorption corrections, transmission factors 0.59–0.78 (0.81–0.92), 4462 (3440) unique reflexions in the θ interval 4–65° (3–30°), 3085 (2336) with $I > 3 \sigma(I)$, the three standard reflexions show 10% (4%) decrease, starting positional parameters from [Ni(pssp)I] [BPh₄] at 295 K (Aurivillius & Bertinsson, 1980), H geometrically generated (SHELX, Sheldrick, 1976), least-squares refinement with SHELX, $w^{-1} = \sigma^2(F)$, scattering factors for non-H from Doyle & Turner (1968) and for H from Stewart, Davidson & Simpson (1965), 185 (183) parameters, $R = 0.085$ (0.078), $wR = 0.108$ (0.100), highest residuals 1.1 (1.2) e Å⁻³, deepest pits 1.4 (0.9) e Å⁻³, $F(000) = 1028$ (1064), correction for anomalous dispersion (Cromer & Liberman, 1970); the intensities show no indication of extinction; magnetic-susceptibility measurements with a Faraday balance (Blom & Hörlin, 1977) have shown the compounds to be diamagnetic, [Ni(pssp)Br] [BPh₄] in the temperature interval 80–290 K and [Ni(pssp)I] [BPh₄] in the interval 80–150 K (Aurivillius & Bertinsson, 1980).

Table 1. Fractional coordinates and isotropic temperature factors for the non-hydrogen atoms in [Ni(C₃₁H₃₄P₂S₂)Br] [B(C₆H₅)₄]

The values for H are deposited. E.s.d.'s are in parentheses. For Br, Ni, S(1), S(2), P(1) and P(2) the isotropic mean values of U_{ij} are given, calculated according to Willis & Pryor (1975).

	x	y	z	$U(\text{Å}^2)$
Br	-0.0623 (2)	-0.0695 (2)	-0.1466 (2)	0.054 (1)
Ni	0.0000	0.0101 (3)	0.0000	0.038 (1)
S(1)	-0.0569 (4)	0.2169 (5)	0.0042 (4)	0.052 (1)
S(2)	-0.1206 (4)	-0.0489 (5)	0.0671 (4)	0.052 (1)
P(1)	0.1238 (3)	0.1022 (4)	-0.0460 (4)	0.041 (1)
P(2)	0.0541 (3)	-0.1878 (4)	0.0339 (4)	0.043 (4)
C(1)	0.075 (1)	0.240 (2)	-0.103 (1)	0.052 (5)
C(2)	0.011 (1)	0.322 (2)	-0.056 (1)	0.062 (6)
C(3)	-0.168 (1)	0.211 (2)	-0.052 (1)	0.063 (6)
C(4)	-0.240 (2)	0.133 (2)	-0.033 (1)	0.060 (6)
C(5)	-0.223 (1)	-0.012 (2)	0.003 (1)	0.062 (6)
C(6)	-0.118 (2)	-0.232 (2)	0.071 (2)	0.039 (4)
C(7)	-0.047 (1)	-0.293 (2)	0.024 (1)	0.077 (7)
C(8)	0.2047 (9)	0.025 (1)	-0.1062 (7)	0.083 (8)
C(9)	0.1757 (9)	-0.009 (1)	-0.1859 (7)	0.068 (6)
C(10)	0.2392 (9)	-0.060 (1)	-0.2370 (7)	0.062 (6)
C(11)	0.3317 (9)	-0.077 (1)	-0.2083 (7)	0.055 (5)
C(12)	0.3607 (9)	-0.044 (1)	-0.1286 (7)	0.036 (4)
C(13)	0.2972 (9)	0.007 (1)	-0.0775 (7)	0.042 (5)
C(14)	0.1986 (9)	0.177 (1)	0.0384 (7)	0.055 (5)
C(15)	0.2220 (9)	0.096 (1)	0.1055 (7)	0.065 (6)
C(16)	0.2818 (9)	0.144 (1)	0.1692 (7)	0.056 (5)
C(17)	0.3181 (9)	0.273 (1)	0.1657 (7)	0.065 (6)
C(18)	0.2946 (9)	0.354 (1)	0.0986 (7)	0.056 (5)
C(19)	0.2349 (9)	0.306 (1)	0.0349 (7)	0.035 (4)
C(20)	0.0869 (9)	-0.218 (1)	0.1424 (8)	0.056 (5)
C(21)	0.1140 (9)	-0.347 (1)	0.1664 (8)	0.063 (6)
C(22)	0.1333 (9)	-0.376 (1)	0.2484 (8)	0.069 (7)
C(23)	0.1256 (9)	-0.276 (1)	0.3063 (8)	0.075 (7)
C(24)	0.0984 (9)	-0.147 (1)	0.2822 (8)	0.064 (6)
C(25)	0.0791 (9)	-0.118 (1)	0.2003 (8)	0.051 (5)
C(26)	0.146 (1)	-0.270 (2)	-0.0133 (8)	0.064 (6)
C(27)	0.124 (1)	-0.328 (2)	-0.0890 (8)	0.088 (8)
C(28)	0.193 (1)	-0.392 (2)	-0.1291 (8)	0.085 (8)
C(29)	0.284 (1)	-0.397 (2)	-0.0935 (8)	0.077 (7)
C(30)	0.305 (1)	-0.339 (2)	-0.0179 (8)	0.068 (7)
C(31)	0.237 (1)	-0.276 (2)	0.0222 (8)	0.043 (5)
C(32)	0.5295 (8)	0.639 (1)	0.1385 (8)	0.046 (5)
C(33)	0.4703 (8)	0.565 (1)	0.1836 (8)	0.052 (5)
C(34)	0.4001 (8)	0.629 (1)	0.2218 (8)	0.062 (6)
C(35)	0.3892 (8)	0.767 (1)	0.2149 (8)	0.065 (6)
C(36)	0.4484 (8)	0.841 (1)	0.1698 (8)	0.056 (6)
C(37)	0.5185 (8)	0.777 (1)	0.1316 (8)	

Table 1 (cont.)

	x	y	z	$U(\text{Å}^2)$
C(38)	0.7117 (8)	0.562 (1)	0.1559 (8)	0.040 (4)
C(39)	0.7808 (8)	0.466 (1)	0.1493 (8)	0.059 (6)
C(40)	0.8634 (8)	0.474 (1)	0.1986 (8)	0.056 (6)
C(41)	0.8769 (8)	0.578 (1)	0.2545 (8)	0.066 (6)
C(42)	0.8078 (8)	0.673 (1)	0.2611 (8)	0.058 (6)
C(43)	0.7252 (8)	0.665 (1)	0.2118 (8)	0.054 (5)
C(44)	0.5784 (8)	0.398 (1)	0.0720 (7)	0.042 (4)
C(45)	0.5922 (8)	0.298 (1)	0.1305 (7)	0.038 (4)
C(46)	0.5570 (8)	0.170 (1)	0.1151 (7)	0.054 (5)
C(47)	0.5080 (8)	0.142 (1)	0.0412 (7)	0.051 (5)
C(48)	0.4941 (8)	0.241 (1)	-0.0174 (7)	0.049 (5)
C(49)	0.5293 (8)	0.369 (1)	-0.0020 (7)	0.046 (5)
C(50)	0.6346 (8)	0.624 (1)	-0.0011 (8)	0.045 (5)
C(51)	0.5692 (8)	0.705 (1)	-0.0439 (8)	0.072 (7)
C(52)	0.5862 (8)	0.753 (1)	-0.1204 (8)	0.083 (8)
C(53)	0.6686 (8)	0.720 (1)	-0.1540 (8)	0.070 (7)
C(54)	0.7339 (8)	0.639 (1)	-0.1111 (8)	0.066 (6)
C(55)	0.7169 (8)	0.591 (1)	-0.0346 (8)	0.061 (6)
B	0.612 (1)	0.559 (2)	0.094 (1)	0.041 (5)

Table 2. Angles (°) in the coordination polyhedra of Ni, P(1), P(2), S(1) and S(2)

E.s.d.'s are given in parentheses. For labelling, see Figs. 1(a) and 1(b) and Table 2. X = Br or I.

	[Ni(pssp)Br] [BPh ₄]	[Ni(pssp)I] [BPh ₄]
X–Ni–P(1)	91.5 (2)	91.8 (2)
X–Ni–P(2)	93.2 (2)	93.8 (2)
X–Ni–S(1)	101.9 (2)	103.6 (2)
X–Ni–S(2)	99.5 (2)	100.5 (2)
P(1)–Ni–S(1)	86.3 (2)	86.6 (3)
S(1)–Ni–S(2)	85.7 (2)	85.1 (2)
S(2)–Ni–P(2)	84.8 (2)	84.7 (3)
P(2)–Ni–P(1)	100.6 (2)	100.2 (3)
P(1)–Ni–S(2)	167.5 (2)	166.5 (3)
P(2)–Ni–S(1)	163.3 (2)	161.2 (3)
Ni–P(1)–C(1)	102.0 (6)	102.1 (8)
Ni–P(1)–C(8)	127.0 (5)	126.9 (6)
Ni–P(1)–C(14)	110.4 (5)	111.9 (6)
C(1)–P(1)–C(8)	107.0 (8)	104.4 (9)
C(1)–P(1)–C(14)	105.6 (7)	106.8 (9)
C(8)–P(1)–C(14)	103.2 (6)	103.2 (7)
Ni–P(2)–C(7)	103.6 (7)	101.4 (9)
Ni–P(2)–C(20)	117.2 (4)	118.1 (5)
Ni–P(2)–C(26)	124.4 (5)	123.1 (6)
C(7)–P(2)–C(20)	97.7 (9)	98 (1)
C(7)–P(2)–C(26)	108.6 (9)	108 (1)
C(20)–P(2)–C(26)	102.0 (6)	104.6 (7)
Ni–S(1)–C(2)	107.7 (7)	107.0 (9)
Ni–S(1)–C(3)	105.6 (8)	104.0 (9)
C(2)–S(1)–C(3)	104 (1)	103 (1)
Ni–S(2)–C(5)	107.4 (8)	104.4 (8)
Ni–S(2)–C(6)	105.7 (8)	105.2 (8)
C(5)–S(2)–C(6)	103 (1)	104 (1)

Discussion. Final positional and isotropic temperature parameters for [Ni(pssp)Br] [BPh₄] are given in Table 1.* selected interatomic distances and angles are given in Fig. 1(a) and Table 2. A view of the atomic arrangement in [Ni(pssp)Br]⁺ and a stereoview of the packing of [Ni(pssp)I] [BPh₄] in the unit cell are given in Fig. 2 (Johnson, 1965) and by Aurivillius &

* Lists of structure factors, anisotropic thermal parameters, fractional coordinates and isotropic temperature factors for the H atoms and final positional parameters for [Ni(pssp)I] [BPh₄] have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38366 (41 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Bertinsson (1980) respectively. From the positional parameters it can be seen that the title compounds are isostructural with $[Ni(pssp)I][BPh_4]$ at 295 K. In these monomeric complexes, pssp is quadridentate *via* the P and S atoms. Thus two five- and one six-membered chelate rings are formed. The donor atoms of pssp constitute the basal plane in the square-pyramidal polyhedron of Ni and the halogen atoms are situated at the apex, see Fig. 2. The Ni atoms are displaced 0.239 (2) (Br compound) and 0.247 (2) Å (I compound) above the basal planes of the square pyramids towards the halogen atoms. The value for the I compound at 295 K is 0.26 (1) Å.

Corresponding Ni–P and Ni–S distances, Figs. 1(a) and 1(b), in the title compounds as well as in $[Ni(pssp)I][BPh_4]$ at 295 K are within 3σ (Lipson & Cochran, 1968). The Ni–P mean values are 2.196 (4), 2.196 (5) and 2.216 (6) Å and the Ni–S mean values are 2.227 (4), 2.248 (5) and 2.245 (5) Å in the Br and the I compounds at 120 and 295 K, respectively.

The Ni–I distance undergoes no variation between 120 K [2.638 (4) Å] and 295 K [2.649 (3) Å]. Despite a difference of about 0.2 Å (Pauling, 1960) in the ionic radii of Br^- and I^- there is no significant difference in the Ni–I [2.649 (3) Å] and the Ni–Br [2.645 (4) Å] distances. The softer character of I (Ahrlund, Chatt & Davies, 1958) will give the Ni–I bond a stronger covalent contribution which will shorten this bond relative to the Ni–Br bond.

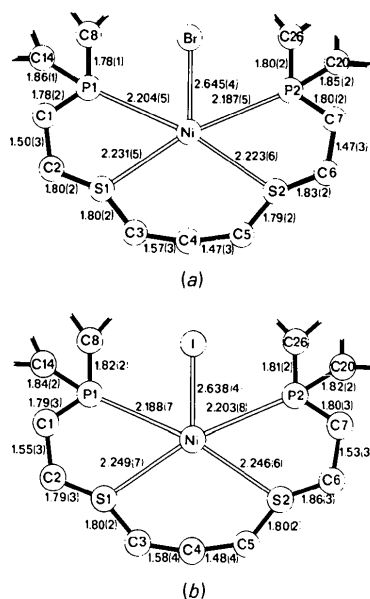


Fig. 1. Schematic drawings of the complex ions (a) $[Ni(pssp)Br]^+$ and (b) $[Ni(pssp)I]^+$, showing selected interatomic distances (Å). The C–C distances in the phenyl rings are assumed to be 1.395 Å. The bonds of the ligand chain are drawn with heavy lines, those in the coordination polyhedra of Ni with double lines.

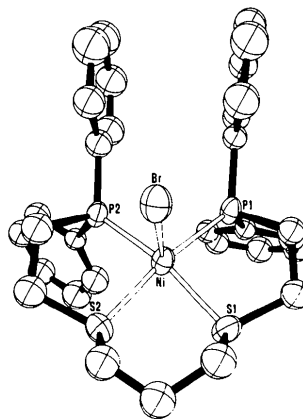


Fig. 2. A drawing of the complex ion $[Ni(pssp)Br]^+$. The bonds of the ligand molecule (pssp) are drawn with heavy lines, those of the coordination polyhedron of Ni with double lines. The H atoms are omitted. The ellipsoids are drawn to enclose 50% probability.

The atom H(25) from a phenyl group of the complex is situated 3.07 (1) Å (Br compound) and 3.11 (1) Å (I compound) from Ni, near the sixth coordination site.

The angles around Ni in the complexes (Table 2) are in good agreement with analogous angles in $[Ni(pssp)I][BPh_4]$ at 295 K which have previously been discussed by Aurivillius & Bertinsson (1980).

The P and S atoms are, as expected, sp^3 -hybridized, although in a distorted way. The angles (Table 2) and the P–C and S–C distances as well as the C–C distances (Fig. 1) in the ligand chain correspond well to values given in $[Ni(pssp)I][BPh_4]$ at 295 K and in $[Ni(pssp)][ClO_4]_2$ (Aurivillius & Bertinsson, 1981).

The five-membered rings (NiPC₂S) are puckered, one to λ and the other to σ configuration. Large deviations occur from the idealized configurations with twofold symmetry. The six-membered rings (NiSC₃S) are of chair type. These rings are folded up over the basal plane towards the halogen atom.

Of the eight independent phenyl groups in the structures, two groups of two rings are mutually parallel. In the complex the rings with the atoms C(8)–C(13) and C(26)–C(31) are parallel at an average distance of 3.56 Å (Br compound) and 3.48 Å (I compound) and one ring from the complex, C(14)–C(19), and one from the BPh_4^- ion, C(44)–C(49), are parallel at an average distance of 3.61 Å and 3.54 Å for the Br and I compounds, respectively.

The mean values of the B–C distances in the BPh_4^- ions are 1.71 (2) Å (Br) and 1.71 (1) Å (I) and the angles around the B atoms are between 103 (1) and 114 (1)°, see Table 3. These values are quite normal.

The packing of the ions in the unit cell is described by Aurivillius & Bertinsson (1980). Short intermolecular contacts exist between atoms of different cations, Br–H(16) 2.84 (1) and I–H(16) 2.85 (2) Å. The shortest contacts between anions and cations are

Table 3. Distances (Å) and angles (°) in the $B(C_6H_5)_4^-$ ions

E.s.d.'s are given in parentheses.

	[Ni(pssp)Br][BPh ₄]	[Ni(pssp)I][BPh ₄]
B—C(32)	1.66 (3)	1.71 (3)
B—C(38)	1.70 (2)	1.70 (3)
B—C(44)	1.72 (2)	1.71 (3)
B—C(50)	1.76 (3)	1.71 (3)
C(32)—B—C(38)	109 (1)	107 (2)
C(32)—B—C(44)	110 (1)	107 (2)
C(32)—B—C(50)	114 (1)	113 (2)
C(38)—B—C(44)	111 (1)	111 (2)
C(38)—B—C(50)	109 (1)	111 (2)
C(44)—B—C(50)	103 (1)	107 (2)

C(55)—H(6A) 2.35 (3) and 2.29 (3) Å in the Br and I compounds, respectively. These distances are shorter than the sum of van der Waals radii (Br—H 2.95; I—H 3.15; C—H 2.7 Å) (Pauling, 1960; Baur, 1972). The H atoms have, however, been geometrically generated.

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The *catena* Structure and EPR Properties of Bis(oxamide oximato)copper(II)—Oxamide Oxime, $[Cu(C_2H_5N_4O_2)_2] \cdot C_2H_6N_4O_2$

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Abstract. $M_r = 415.83$, monoclinic, $C2/c$, $a = 23.504$ (28), $b = 5.047$ (3), $c = 20.103$ (16) Å, $\beta = 144.03$ (7)°, $V = 1400.7$ Å³, $Z = 4$, $D_x = 1.97$ g cm⁻³, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 16.2$ cm⁻¹, room temperature, $F(000) = 852$, final $R = 0.046$ for 821 observed reflections. The planar centrosymmetric complex molecules form stacks along **b**, the normals of the planes being inclined at $\sim 50^\circ$ to the stacking axis. Close axial interactions between Cu and the amino groups of adjacent complex molecules within a stack, 2.885 (10) Å, result in an elongated square-bipyramidal coordination around Cu. Free oxamide oxime, present in the *s-trans* form, links the stacks into sheets by forming H bridges to the complex molecules. EPR signal intensities follow a Curie–Weiss law in the temperature range 110–400K.

Introduction. Oxamide oxime, $HON=C(NH_2)-C(NH_2)=NOH$ (diaminoglyoxime, oaoH₂) (Ephraim, 1889) forms a variety of complexes with the ions of the Ni triad, depending on crystallization conditions (Endres, 1982). As the structural chemistry of Cu^{II} complexes of α,β -dione dioximes is usually different from that of Ni^{II}, Pd^{II}, Pt^{II}, we started a survey of the structures of Cu complexes of oaoH₂. We were especially interested as to whether the amino groups would coordinate to Cu as well, giving rise to polymeric complexes. Here we report the *catena* structure found in the title compound.

Experimental. The compound was prepared by a diffusion method: A frit was placed inversely in a beaker with ethanol, copper acetate was dissolved outside the frit, and solid oaoH₂ was placed inside the frit. Within a few days brown crystals several mm long

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