Å to the K^+ ion at a center of symmetry in a very distorted octahedral coordination (Table 3). The geometry of the hydrogen bonds formed by the two symmetry-independent water molecules is shown in Fig. 2. All four hydrogen bonds are of the type O—H···O with distances and angles in the usual range except O*w*(1)—H*w*(12)···O(1), which shows elongated distances to the acceptor O(1), but still may be considered as a hydrogen bond. The water molecules form infinite chains along *c* and link the complex anions into a three-dimensional array.

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$(-)_{579}$-Bis[(*S*)-(+)-*O*,*O*′-(1,1′-binaphthyl-2,2′-diyl)dithiophosphato]nickel(II)

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Abstract. $[Ni(C_{20}H_{12}O_2PS_2)_2]$, $C_{40}H_{24}NiO_4P_2S_4$, orthorhombic, $P2_12_12_1$, $a = 9{\cdot}630\ (3)$, $b = 10{\cdot}171\ (4)$, $c = 37{\cdot}489\ (13)$ Å, $Z = 4$, $D_m = 1{\cdot}44$, $D_x = 1{\cdot}48$ Mg m^{-3}, μ(Cu $K\alpha$) = 3·92 mm^{-1}, $M_r = 817{\cdot}5$. The structure was refined to $R = 0{\cdot}05$ and the absolute configuration determined. The Ni atom is coordinated by four S atoms in a square. The dihedral angles of the C—C bonds bridging the two naphthyl residues in each half of the molecule are +53·5 and +52·1° and define an (*S*,*S*) configuration.

Introduction. The technical application of several metal complexes of dithiophosphoric acid has become of interest during recent years (Wasson, Woltermann & Stoklosa, 1973). The crystal structures of two dithiophosphatonickel(II) complexes, $[Ni(S_2PR_2)_2]$, with all four substituents R identical, have been reported: $R = C_2H_5O$ (McConnell & Kastalsky, 1967) and $R = CH_3O$ (Kastalsky & McConnell, 1969). The title compound is the first dithiophosphato complex of Ni^{II} with a bidentate ligand of axial chirality, introduced as one enantiomer only. The preparation and preliminary structural results have been reported by Hoffmann, Kuchen, Poll & Wunderlich (1979) and Poll, Wunderlich & Wussow (1979).

The complex crystallizes from toluene on addition of hexane without a characteristic habit; its color is between brown and violet. Systematic absences are unique for the space group $P2_12_12_1$. The lattice parameters were determined from 15 optimized diffractometer angles by least-squares refinement. The intensities of all symmetry-independent reflections up to $2\theta = 135°$ were measured with Cu $K\alpha$ radiation (crystal monochromator) in an ω-scan mode on an automated diffractometer (Syntex $P2_1$). 2928 out of 3796 reflections were classified as observed ($F > 3\sigma_F$) and were used for the structure determination. The phase problem was solved by the Patterson function. All H atoms could be located from difference maps. The anomalous dispersion of Ni, S, and P was used to determine the absolute configuration. The refinement (556 parameters) converged at $R = 0{\cdot}055$ (0·074) and $R_w = 0{\cdot}053$ (0·056) for the observed (all) reflections. The corresponding refinement of the inverted molecule converged at a significantly larger R_w of 0·057 (0·060), precluding that configuration at the 99·5% probability level (Hamilton, 1974). Weights were derived from counting statistics by $1/w = \sigma_F^2 + 0{\cdot}0004F^2$. Scattering factors were taken from Cromer & Waber (1974). All calculations were performed with the system *EXTL*

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(Syntex) on an Eclipse computer (Data General). Tables 1 and 2* contain the final atomic parameters.†

Table 1. *Positional parameters* (×10⁴) *of the non-hydrogen atoms with e.s.d.'s from the least-squares refinement in parentheses*

The equivalent isotropic temperature factors $B_{\rm eq}$ (Å^2) are defined by $B_{\rm eq} = \frac{1}{3}(B_{11}a^{*2}a^2 + B_{12}a^*b^*ab\cos\gamma + \cdots)$.

	x	y	z	$B_{\rm eq}$
Ni	2933 (1)	2372 (1)	1794 (0)	4·31
S(1)	2583 (3)	253 (2)	1921 (1)	5·49
S(2)	2242 (3)	2910 (2)	2346 (0)	5·27
S(3)	3577 (3)	1857 (2)	1234 (1)	5·50
S(4)	3235 (3)	4504 (2)	1666 (1)	5·21
P(1)	2139 (2)	983 (2)	2396 (1)	4·97
P(2)	3294 (2)	3761 (2)	1182 (1)	4·28
O(1)	687 (5)	419 (6)	2536 (1)	5·5
O(2)	3207 (5)	516 (5)	2698 (1)	5·0
O(3)	1893 (5)	4166 (5)	981 (1)	4·2
O(4)	4469 (5)	4349 (5)	923 (1)	4·1
C(10)	395 (8)	758 (8)	2897 (2)	4·9
C(11)	1047 (8)	18 (7)	3160 (2)	4·4
C(12)	852 (8)	455 (8)	3522 (2)	4·7
C(13)	1604 (8)	−115 (8)	3812 (2)	5·0
C(14)	1450 (9)	350 (9)	4149 (2)	5·8
C(15)	568 (10)	1365 (10)	4223 (2)	6·9
C(16)	−200 (10)	1949 (9)	3963 (3)	6·8
C(17)	−56 (9)	1513 (8)	3599 (2)	5·5
C(18)	−778 (9)	2131 (9)	3315 (3)	6·6
C(19)	−537 (8)	1770 (9)	2976 (3)	6·3
C(20)	3045 (8)	−788 (8)	2821 (2)	4·8
C(21)	1982 (7)	−1066 (7)	3055 (2)	3·8
C(22)	1846 (8)	−2395 (8)	3175 (2)	4·5
C(23)	716 (8)	−2793 (8)	3404 (2)	4·4
C(24)	613 (9)	−4073 (9)	3507 (2)	5·4
C(25)	1578 (11)	−5032 (8)	3389 (2)	6·0
C(26)	2623 (11)	−4654 (9)	3170 (2)	6·6
C(27)	2773 (8)	−3389 (8)	3047 (2)	5·0
C(28)	3812 (9)	−2994 (10)	2809 (2)	6·3
C(29)	3966 (8)	−1750 (10)	2684 (2)	5·8
C(30)	1797 (8)	3926 (7)	615 (2)	3·8
C(31)	2522 (7)	4712 (6)	378 (2)	3·1
C(32)	2408 (7)	4364 (6)	4 (2)	3·4
C(33)	3210 (8)	5020 (7)	−263 (2)	3·9
C(34)	3096 (9)	4647 (8)	−610 (2)	4·7
C(35)	2239 (9)	3645 (8)	−719 (2)	5·2
C(36)	1492 (9)	2980 (7)	−470 (2)	5·2
C(37)	1543 (8)	3325 (6)	−103 (2)	4·1
C(38)	773 (9)	2633 (8)	159 (2)	5·4
C(39)	903 (9)	2901 (8)	515 (2)	5·0
C(40)	4259 (8)	5628 (7)	786 (2)	3·6
C(41)	3320 (7)	5816 (6)	516 (1)	2·8
C(42)	3138 (7)	7137 (6)	386 (1)	3·1
C(43)	2123 (8)	7513 (6)	137 (2)	3·8
C(44)	2000 (10)	8761 (7)	22 (2)	5·0
C(45)	2855 (10)	9744 (7)	153 (2)	5·4
C(46)	3805 (10)	9446 (8)	405 (2)	5·4
C(47)	3998 (8)	8155 (7)	536 (2)	3·9
C(48)	4944 (9)	7865 (9)	805 (2)	5·1
C(49)	5072 (9)	6635 (9)	936 (2)	5·0

Discussion. In the crystal structures of the dithiophosphato complexes of Ni^{II} cited above, the molecules reveal a point symmetry of $\bar{1}$ with Ni at a crystallographic center of symmetry. This is true also for several analogous dithiophosphinato complexes, *e.g.* with $R = CH_3$ (Jones, Ansell & Katz, 1969), $R = C_2H_5$ (Shetty & Fernando, 1969), and $R = C_6H_5$ (Porta, Sgamellotti & Vinciguerra, 1968). Because of the optical activity of the dithiophosphoric acid used for the synthesis, this point symmetry is impossible in the compound under study. Point symmetry 2 would be possible but is not provided by the space group observed. However, as can be seen from the coordinates, there does exist in the projection along **a** a noncrystallographic twofold axis parallel to x through the Ni atom, superposing the coordinates y and z of corresponding atoms.

Fig. 1 shows the molecule with the main bond lengths; the corresponding bond angles are listed in Table 3. The geometry of the H atoms is in the usual range and is not reported. The Ni atom is coordinated by four S atoms in an almost perfect square with mean Ni S = 2·241 Å. The NiS_4 group is planar within 0·02 Å, but the adjacent P atoms deviate 0·09 [P(1)] and 0·46 Å [P(2)] from this plane. The mean P—S of 1·970 Å is short and the mean angle S—P—S of 106·5° is large in comparison with the corresponding values of the related compounds cited above.

Table 2. *Positional* (×10³) *and thermal parameters of the* H *atoms with e.s.d.'s in parentheses*

The H atoms are connected to the C atoms with identical labelling.

	x	y	z	B (Å^2)
H(13)	210 (8)	−75 (7)	374 (2)	6 (2)
H(14)	196 (8)	−13 (7)	435 (2)	8 (2)
H(15)	49 (9)	180 (8)	447 (2)	8 (2)
H(16)	−96 (8)	282 (7)	394 (2)	7 (2)
H(18)	−138 (7)	288 (7)	340 (2)	6 (2)
H(19)	−82 (8)	233 (8)	279 (2)	7 (2)
H(23)	10 (8)	−205 (8)	350 (2)	7 (2)
H(24)	−9 (8)	−427 (8)	366 (2)	6 (2)
H(25)	140 (7)	−586 (7)	351 (2)	6 (2)
H(26)	316 (8)	−531 (7)	314 (2)	7 (2)
H(28)	438 (7)	−344 (6)	273 (2)	4 (2)
H(29)	454 (6)	−130 (6)	250 (2)	4 (1)
H(33)	364 (5)	563 (5)	−19 (1)	2 (1)
H(34)	357 (7)	520 (6)	−79 (2)	5 (2)
H(35)	230 (7)	332 (6)	−94 (2)	5 (2)
H(36)	110 (6)	244 (6)	−53 (1)	3 (1)
H(38)	24 (7)	199 (6)	9 (2)	5 (2)
H(39)	49 (7)	241 (7)	70 (2)	6 (2)
H(43)	152 (6)	689 (6)	5 (1)	4 (1)
H(44)	139 (7)	901 (6)	−14 (2)	5 (2)
H(45)	273 (7)	1069 (6)	5 (1)	5 (2)
H(46)	437 (7)	995 (7)	52 (2)	6 (2)
H(48)	544 (8)	842 (8)	88 (2)	8 (2)
H(49)	542 (7)	649 (6)	108 (2)	5 (2)

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* For rational labelling the atom numbering is different from that of the chemical nomenclature; see also Figs. 1 and 2.

† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35047 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

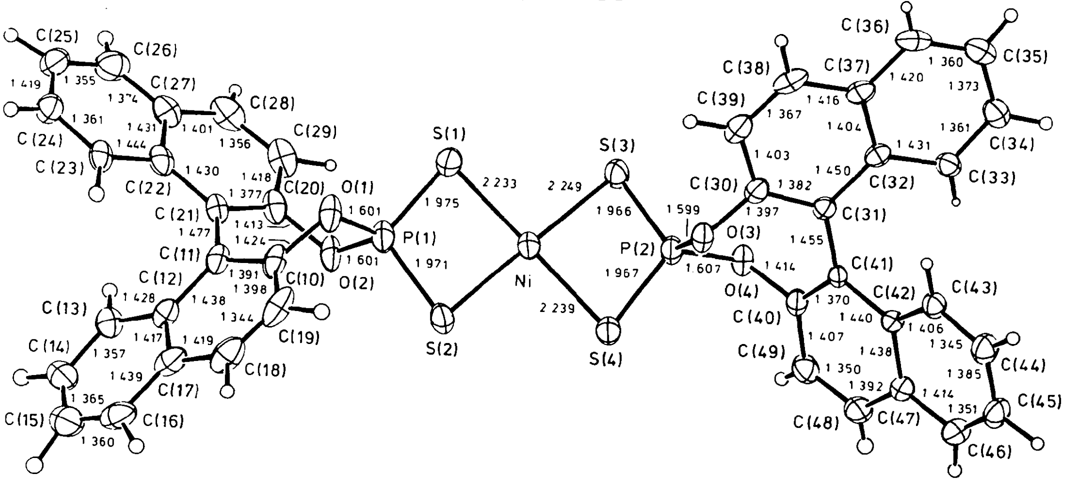

Fig. 1. A molecule of $[Ni(C_{20}H_{12}O_2PS_2)_2]$ in arbitrary crystallographic orientation with thermal ellipsoids at 25% probability (*ORTEP* II; Johnson, 1976) and bond lengths in Å. The e.s.d.'s are: Ni—S, S—P 0·002–0·003; P—O 0·005–0·006; C—O, C—C 0·008–0·014 Å. The H atoms are shown with radii corresponding to 0·2 *B* (Å²).

Table 3. *Bond angles* (°) *involving non-hydrogen atoms*

(i) Angles involving atoms of the NiS_4 group (e.s.d.'s are 0·1 °)

S(1)–Ni–S(2)	89·6	Ni–S(1)–P(1)	82·1
S(1)–Ni–S(3)	90·9	Ni–S(2)–P(1)	81·9
S(1)–Ni–S(4)	178·8	Ni–S(3)–P(2)	79·9
S(2)–Ni–S(3)	178·6	Ni–S(4)–P(2)	80·2
S(2)–Ni–S(4)	90·0		
S(3)–Ni–S(4)	89·5		

(ii) Angles at P and O atoms (e.s.d.'s at P atoms are 0·1–0·3 ° and at O atoms 0·4–0·5 °)

S(1)–P(1)–S(2)	106·1	S(3)–P(2)–S(4)	106·9
S(1)–P(1)–O(1)	110·5	S(3)–P(2)–O(3)	114·7
S(1)–P(1)–O(2)	112·7	S(3)–P(2)–O(4)	109·2
S(2)–P(1)–O(1)	115·6	S(4)–P(2)–O(3)	108·2
S(2)–P(1)–O(2)	109·3	S(4)–P(2)–O(4)	115·8
O(1)–P(1)–O(2)	102·8	O(3)–P(2)–O(4)	102·3
P(1)–O(1)–C(10)	113·5	P(2)–O(3)–C(30)	118·2
P(1)–O(2)–C(20)	116·1	P(2)–O(4)–C(40)	117·5

(iii) Angles involving the C—C bond bridging two naphthyl residues (e.s.d.'s are 0·5–0·7 °)

C(10)–C(11)–C(21)	119·3	C(30)–C(31)–C(41)	119·0
C(12)–C(11)–C(21)	124·1	C(32)–C(31)–C(41)	124·8
C(20)–C(21)–C(11)	118·0	C(40)–C(41)–C(31)	120·2
C(22)–C(21)–C(11)	124·5	C(42)–C(41)–C(31)	122·5

(iv) Angles of the four naphthyl residues (e.s.d.'s are 0·5–0·9 °)

	$n = 1$	$n = 2$	$n = 3$	$n = 4$
O(*n*)–C(*n*0)–C(*n*1)	117·0	118·9	119·7	119·4
O(*n*)–C(*n*0)–C(*n*9)	120·5	117·4	115·6	116·4
C(*n*1)–C(*n*0)–C(*n*9)	122·6	123·7	124·6	124·2
C(*n*0)–C(*n*1)–C(*n*2)	116·3	117·5	116·1	117·3
C(*n*1)–C(*n*2)–C(*n*3)	121·8	121·4	121·5	124·2
C(*n*1)–C(*n*2)–C(*n*7)	120·5	120·4	120·3	118·1
C(*n*3)–C(*n*2)–C(*n*7)	117·7	118·1	118·1	117·6
C(*n*2)–C(*n*3)–C(*n*4)	120·8	119·5	119·8	122·0
C(*n*3)–C(*n*4)–C(*n*5)	121·4	121·4	122·6	121·0
C(*n*4)–C(*n*5)–C(*n*6)	121·5	118·7	118·9	119·2
C(*n*5)–C(*n*6)–C(*n*7)	119·5	123·1	121·6	122·7
C(*n*6)–C(*n*7)–C(*n*8)	121·8	123·8	121·9	122·3
C(*n*6)–C(*n*7)–C(*n*2)	119·2	119·0	118·9	117·3
C(*n*8)–C(*n*7)–C(*n*2)	119·0	117·2	119·1	120·4
C(*n*7)–C(*n*8)–C(*n*9)	120·3	124·4	121·9	121·4
C(*n*8)–C(*n*9)–C(*n*0)	120·9	116·8	117·7	118·5

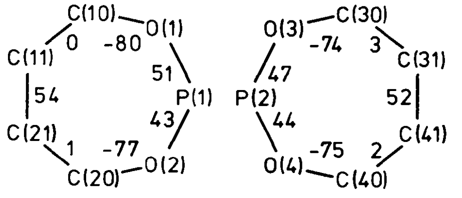

Fig. 2. Torsion angles (°) within the seven-membered rings, indicating half-chair conformations. The e.s.d.'s are *ca* 1 °.

Fig. 2 shows the torsion angles within the nonplanar seven-membered rings. They conform to a pseudo twofold axis in each ring through the P atom and the centre of the opposite C—C bond, thus indicating half-chair conformations.

The naphthyl skeletons are planar within 0·08 (first digit of atomic labelling $n = 1$), 0·04 ($n = 2$), 0·05 ($n = 3$), and 0·05 Å ($n = 4$). The axial chirality (Cahn, Ingold & Prelog, 1966) of the compound is given by the sign of the torsion angles of the bonds C(11)—C(21) and C(31)—C(41) which are +53·5 and +52·1° and define an (*S*,*S*) configuration, from which the absolute configuration of the dithiophosphoric acid used for the preparation appears as *S*.

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The Structure of Bis(benzamido)mercury(II)

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Abstract. $[Hg(C_7H_6NO)_2]$, $C_{14}H_{12}HgN_2O_2$, monoclinic, $C2/c$, $a = 21.33$ (2), $b = 15.69$ (2), $c = 8.42$ (2) Å, $\beta = 110.5$ (1)°, $D_m = 2.19$, $D_x = 2.216$ Mg m^{-3} with $Z = 8$. The structure has been determined from 931 diffractometer-measured intensities ($R = 0.064$). Hg is almost linearly bonded to two N atoms [Hg—N = 2·06 (3), 2·04 (2) Å, N—Hg—N = 172 (1)°] and also equatorially to two O atoms of adjacent molecules [Hg—O = 2·67 (2) and 2·83 (3) Å]. The O—Hg—O angle is 76 (1)°. There is also a weak intermolecular hydrogen bond [N···O = 3·03 (5) Å].

Introduction. Interest in the structures of the Hg derivatives of acid amides lies in the nature of the Hg—O interaction, whether the amide group is to any extent bidentate and, in the crystal, the extent of intermolecular coordination. The title compound, kindly supplied by Dr F. G. Thorpe, was recrystallized by slow evaporation at 277 K from an ethanol solution to give needles elongated about c showing the forms {110} but without well-defined end faces. The crystals decomposed slowly upon irradiation. Cell dimensions and systematic absences (hkl when $h + k = 2n + 1$, $h0l$ when $l = 2n + 1$) were obtained from Weissenberg photographs with Cu $K\alpha$ radiation ($\lambda = 1.542$ Å). The crystal density was measured by flotation. With a crystal of dimensions 0·06 × 0·05 × 0·29 mm set about c, intensity data for 1387 reflexions in the range $4° < 2\theta < 40°$ were measured on a Stoe STADI-2 automatic two-circle diffractometer; graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) was used. Absorption corrections ($\mu = 11.234$ mm^{-1}) were made using the *SHELX* 76 suite of programs (Sheldrick, 1976). Only 931 reflexions, for which $I > 3\sigma(I)$, were used in subsequent calculations employing the XRAY 72 system (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Patterson maps which gave strong peaks at $y = 0$ indicated the space group $C2/c$ rather than Cc. Hg coordinates obtained from these maps were used to phase $(F_o - F_c)$ maps to give the remainder of the atoms other than H. Full-matrix least-squares refinement (using unit weights) of all non-H atom positions, anisotropic U_{ij} values for Hg and isotropic U's for the remaining non-H atoms was alternated with refinement of the scale factors for each layer ($l = 0$ to 7) of the diffractometer data, U_{33} being fixed to avoid excessive correlation. At an intermediate stage an $(F_o - F_c)$ map showed positive regions in the neighbourhoods of most expected H positions. Inclusion of H atoms for the phenyl groups at calculated positions (fixed 1·08 Å from the parent atom with U fixed at 0·05 Å^2) was found to improve the agreement. When refinement was complete (shift/error < 0·1 for all parameters), R was 0·064 and a final $(F_o - F_c)$ map showed no significant features. Atomic scattering factors for Hg, including anomalous scattering, were taken from Cromer & Mann (1968) and Cromer & Liberman (1970); those for all other atoms were from *International Tables for X-ray Crystallography* (1962). Final coordinates and temperature factors for atoms other than H are given in Table 1.† A diagram of the molecule is shown in Fig. 1.

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† Lists of structure factors and bond lengths and angles around non-Hg atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35053 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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