Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Hg(1)	0	0	0	0.0376 (3)
S(1)	0.2141 (2)	−0.0540 (2)	0.2176 (3)	0.042 (1)
S(2)	2/3	1/3	−0.3568 (5)	0.051 (1)
F(1)	0.6628 (7)	0.2316 (6)	−0.7020 (9)	0.098 (4)
O(1)	0.1832 (5)	0.0528 (5)	0.1848 (7)	0.050 (3)
O(2)	0.5516 (6)	0.2214 (6)	−0.3095 (9)	0.078 (4)
C(1)	0.2999 (9)	−0.0130 (8)	0.443 (1)	0.059 (5)
C(2)	0.3391 (9)	−0.0235 (10)	0.053 (1)	0.069 (5)
C(3)	2/3	1/3	−0.626 (2)	0.061 (6)

Table 2. *Selected geometric parameters* (Å, °)

Hg(1)—O(1)	2.347 (5)	Hg(1)···H(1*c*)	4.80
S(1)—O(1)	1.543 (5)	Hg(1)···H(2*a*)	5.05
S(1)—C(1)	1.766 (8)	Hg(1)···H(2*b*)	4.00
S(1)—C(2)	1.764 (9)	Hg(1)···H(2*c*)	4.73
Hg(1)···H(1*a*)	5.48	Hg(1)···H(1*c*^{i})	4.73
Hg(1)···H(1*b*)	5.11		
O(1)—Hg(1)—O(1^{ii})	94.6 (2)	C(1)—S(1)—C(2)	98.6 (5)
O(1)—S(1)—C(1)	104.1 (3)	Hg(1)—O(1)—S(1)	116.4 (3)
O(1)—S(1)—C(2)	106.5 (4)		

Symmetry codes: (i) $x, y, z-1$; (ii) $-y, x-y, z$.

The θ-scan width used was $(1.30 + 0.30\tan\theta)^\circ$ at a speed of 8.0° min^{-1} in ω. The weak reflections were rescanned a maximum of four times and the counts accumulated to ensure good counting statistics. Stationary background counts were made on each side of the reflection with 2:1 ratio of peak to background counting time.

The structure can be solved by inference: since the Laue class is $\bar{3}$ and $Z = 1$, the Hg atom must have site symmetry $\bar{3}$ and be centered at the origin [Wyckoff position (1*c*)]. Likewise, the anion has 3 symmetry and hence must be oriented about (*c*) or (*d*). The structure was expanded by Fourier techniques (Beurskens *et al.*, 1992). This is the three-dimensional analogue of the method used by Robertson & Woodward (1940) to solve the structure of Pt phthalocyanine. Methyl H atoms were fixed at ideal positions [$U_{iso} = 1.2U_{eq}$(C)].

Data collection and cell refinement were performed using *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1989). All other calculations, including data reduction and structure refinement, were performed using *TEXSAN* (Molecular Structure Corporation, 1993).

We would like to thank Dr Marcia Scudder and Mr Don Craig (UNSW) for their assistance with data analysis.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HR1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraaqua(3,4-toluenediamine-*N*,*N*,*N'*,*N'*-tetraacetato)dizinc(II)–Water (1/2)

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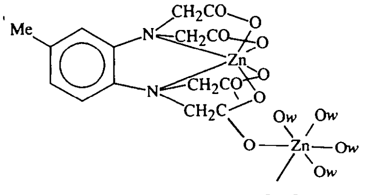
Abstract

The simplest unit of the title compound, $[Zn_2(C_{15}H_{14}N_2O_8)(H_2O)_4].2H_2O$, is a dinuclear Zn^{II} complex bridged by the O atoms of one carboxylate moiety of the 3,4-toluenediamine-*N*,*N*,*N'*,*N'*-tetraacetate ligand (3,4-tdta). The coordination geometry of the zinc-3,4-tdta moiety is close to trigonal prismatic, whereas the other zinc ion is in a nearly O_h environment comprising two O atoms from carboxylate groups of different ligands and four water molecules. The other two water molecules are waters of crystallization in the polynuclear complex $[Zn\text{-}\mu\text{-}(3,4\text{-tdta})\text{-}Zn(H_2O)_4]_n.2H_2O_n$.

Comment

3,4-tdta acid (Mederos, Herrera, Felipe & Quesada, 1984) is similar to *o*-phdta acid (*o*-phenylenediamine-*N*,*N*,*N'*,*N'*-tetraacetic) (Nakasuka, Kunimatsu, Matsumura & Tanaka, 1985) derived from *o*-phenylenediamines. The octahedral structure is quite common in metal complexes of potentially hexadentate aminopolycarboxylates such as ethylenediaminetetraacetate (edta). This is, however, not the case for the complexes of *o*-phdta and 3,4-tdta acids, since the substitution of the ethylenic backbone (edta) for the phenylene (*o*-phdta) or toluene (3,4-tdta) groups imposes a planar configuration on the diamine chelate ring and the electron-withdrawing effect of the benzene ring reduces the basicity of the N donor atoms. *o*-phdta forms complexes with Co^{II} (McCandlish, Michael, Neal, Lingafelter & Rose, 1978), Mn^{II} (Nakasuka, Azuma, Katayama, Honda, Tanaka & Tanaka, 1985), Zn^{II} (Azuma, Nakasuka & Tanaka, 1986), Cu^{II} (Nakasuka, Azuma & Tanaka, 1986*a*), Cd^{II} (Nakasuka, Azuma & Tanaka, 1986*b*), Mg^{II} (Nakasuka & Shiro, 1989) and Fe^{III} (Mizuno, Funahashi, Nakasuka & Tanaka, 1991). The coordination geometries are antiprismatic, twisted toward the trigonal prismatic. The structure of 3,4-tdta with Fe^{III} (Hernández-Padilla, Sanchiz, Domìnguez, Mederos, Arrieta & Zúñiga, 1995) is similar. The presence of the CH_3 radical in 3,4-tdta increases the basicity of the N atom in the *para* position, shortening the length of the Fe—N bond [2.303 (1) Å in 3,4-tdta, 2.344 (3) Å in *o*-phdta].



[Zn-μ-(3,4-tdta)-Zn$(H_2O)_4]_n$

In the present work the crystalline solid complex [Zn-μ-(3,4-tdta)-Zn$(H_2O)_4].2H_2O$ has been prepared and characterized. The ligand is hexadentate. The compound is a polynuclear complex bridged by carboxylate groups. As is evident from Fig. 1 and Table 2, the coordination polyhedron for Zn1 is also twisted towards a trigonal prismatic configuration, analogous to the structure of $K_2[Zn(o\text{-phdta})].3H_2O$ (Azuma *et al.*, 1986). For Zn2, the polyhedron has almost O_h symmetry involving interactions with four water molecules and two O atoms from carboxylate groups of different ligands [O24($\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$) and O20, respectively; see Fig. 1]. A comparison of the Zn—N bond lengths with those in Zn-*o*-phdta (Azuma *et al.*, 1986) indicates that they are of the same order [2.191 (5) and 2.197 (4) Å for 3,4-tdta, 2.184 (3) and 2.191 (2) Å for *o*-phdta]. However, compared with the Fe^{III}-3,4-tdta complex (Hernández-Padilla *et al.*, 1995), the Zn—N bond [2.191 (5) Å] with the N atom in the *para* position with respect to the methyl radical, is shorter than the Zn—N bond [2.197 (4) Å] with the N atom in a *meta* position, in agreement with the *para* > *ortho* > *meta* electron-donor effect (Hehre, Random, Schleyer & Pople, 1986). Comparison of the Zn—N bond lengths with those in Zn-edta (Solans, Font-Altaba, Oliva & Herrera, 1983) shows that the mean values [2.194 (5) Å for Zn-3,4-tdta, 2.188 (3) Å for Zn-*o*-phdta] are considerably longer than the corresponding value [2.154 (7) Å] in the edta complex. This may result from the diminished electron density on the N atoms in *o*-phdta complexes (Nakasuka *et al.*, 1985; Matsumura, Nakasuka & Tanaka, 1987) and 3,4-tdta complexes (Mederos, Felipe, Hernández-Padilla, Brito, Chinea & Bazdikian, 1986; Mederos, Herrera & Felipe, 1987) as well as a stereochemical requirement imposed by the planar N—C—C—N linkage in the *o*-phenylenediaminetetraacetate and 3,4-toluenediaminetetraacetate anions.

The distances (Table 2) C23—O24, C23—O25, C15—O17, C11—O13, C19—O21 and C19—O20, between a carboxyl C atom and an O atom bonded to Zn, are longer than C15—O16 and C11—O12 involving

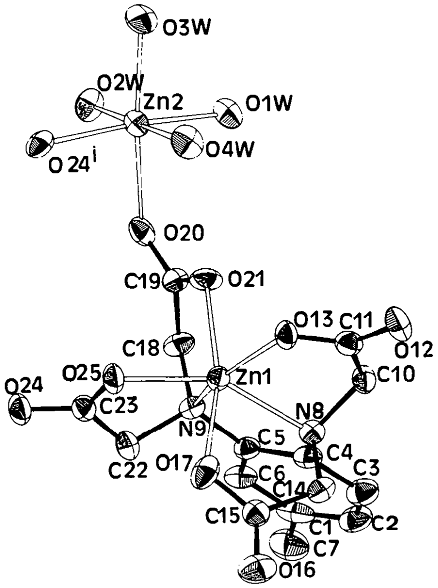


Fig. 1. *ORTEP*II (Johnson, 1976) view of the simplest unit of the [Zn-μ-(3,4-tdta)-Zn$(H_2O)_4]$ dinuclear complex (50% probability ellipsoids; H atoms excluded).

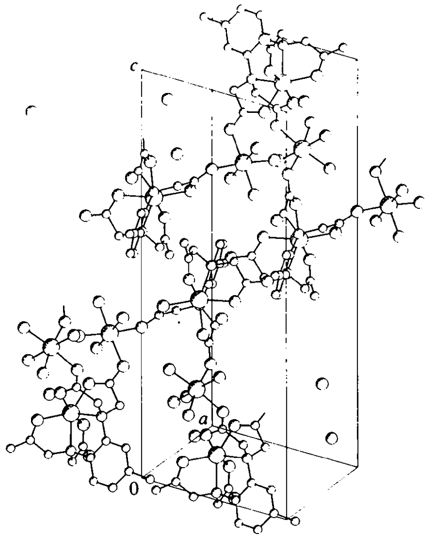


Fig. 2. Molecular packing in the crystal of [Zn-μ-(3,4-tdta)-Zn$(H_2O)_4]_n.2H_2O_n$

non-bonded O atoms. This is also found for the complexes $K_2[Zn(o\text{-phdta})].3H_2O$ (Azuma *et al.*, 1986) and $K[Fe(OH_2)(3,4\text{-tdta})].1.5H_2O$ (Hernández-Padilla *et al.*, 1995).

The diamine-containing chelate ring Zn1—N9—C5—C4—N8 is practically planar judging from the sum of the interior angles of 540 (2)° {539.9 (9) for $K_2[Zn(o\text{-phdta})].3H_2O$, 540 (1) for $K[Fe(OH_2)(3,4\text{-tdta})].1.5H_2O$, 540° for an ideal pentagon}. This is not the case, however, for any of the glycinate rings: Zn1—O25—C23—C22—N9 523 (2), Zn1—O21—C19—C18—N9 534 (2), Zn1—O13—C11—C10—N8 522 (2), Zn1—O17—C15—C14—N8 535 (2)° {520.9 (8)–536.7 (8) for $K_2[Zn(o\text{-phdta})].3H_2O$, 521 (1)–537 (1)° for $K[Fe(OH_2)(3,4\text{-tdta})].1.5H_2O$, while one of four rings is almost planar, 539.5°, in the Mg[Zn(edta)] complex (Solans *et al.*, 1983)}. A packing diagram is presented in Fig. 2.

Experimental

The monopotassium salt of 3,4-tdta was obtained by the method of Mederos, Herrera, Felipe & Quesada (1984). The complex $Zn_2(3,4\text{-tdta}).6H_2O$ were prepared by mixing an aqueous solution of the monopotassium salt of 3,4-tdta with a concentrated solution of $Zn(NO_3)_2.6H_2O$ in a 1:2 ligand:metal ratio. The pH was adjusted to 4 with KOH. Crystals suitable for X-ray crystallography were grown by liquid–vapour diffusion, using water as solvent and 2-propanol as precipitant. Elemental analysis: calculated for $C_{15}H_{26}N_2O_{14}Zn_2$ C 30.58, N 4.75, H 4.45%; found C 30.27, N 4.68 and H 4.13%. The crystal density D_m was measured by picnometry.

Crystal data

$[Zn_2(C_{15}H_{14}N_2O_8)(H_2O)_4].2H_2O$
$M_r = 589.14$
Monoclinic
$P2_1/n$
$a = 8.140 (2)$ Å
$b = 9.672 (3)$ Å
$c = 27.079 (4)$ Å
$\beta = 94.26 (2)°$
$V = 2126 (1)$ Å^3
$Z = 4$
$D_x = 1.84$ Mg m^{-3}
$D_m = 1.82$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 10–30°$
$\mu = 2.385$ mm^{-1}
$T = 293$ K
Prismatic
0.25 × 0.08 × 0.08 mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans [$\Delta 2\theta = (0.8 + 0.35 \tan\theta)°$]
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.761$, $T_{\max} = 0.855$
7130 measured reflections

6589 independent reflections
3576 observed reflections [$I \geq 3\sigma(I)$]
$R_{\rm int} = 0.0868$
$\theta_{\max} = 30°$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 13$
$l = -38 \rightarrow 38$
3 standard reflections frequency: 120 min intensity decay: 5%

Refinement

Refinement on F
$R = 0.0526$
$wR = 0.0571$
$S = 1.31$
3576 reflections
323 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o) + 0.0218(F_o)^2]$

$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.98$ e Å^{-3}
$\Delta\rho_{\min} = -0.79$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
Zn1	0.6457 (1)	0.0849 (1)	0.3647 (1)	1.8 (1)
C1	0.3784 (8)	0.3520 (6)	0.5143 (3)	3.0 (2)
C2	0.5194 (8)	0.3088 (6)	0.5404 (2)	3.0 (1)
C3	0.6350 (7)	0.2322 (6)	0.5177 (2)	2.8 (1)
C4	0.6090 (6)	0.1968 (5)	0.4677 (2)	2.1 (1)
C5	0.4620 (6)	0.2376 (5)	0.4409 (2)	1.9 (1)
C6	0.3481 (7)	0.3170 (6)	0.4647 (2)	2.5 (1)
C7	0.2565 (9)	0.4405 (7)	0.5402 (3)	4.0 (2)
N8	0.7339 (5)	0.1253 (5)	0.4417 (2)	1.9 (1)
N9	0.4293 (5)	0.1922 (4)	0.3900 (2)	1.8 (1)
C10	0.7706 (7)	−0.0138 (5)	0.4603 (2)	2.2 (1)
C11	0.8606 (7)	−0.0937 (5)	0.4215 (2)	2.3 (1)
O12	0.9675 (5)	−0.1767 (5)	0.4362 (2)	3.2 (1)
O13	0.8147 (5)	−0.0682 (4)	0.3762 (1)	2.3 (1)
C14	0.8864 (6)	0.2082 (6)	0.4409 (2)	2.2 (1)
C15	0.8996 (7)	0.2881 (6)	0.3935 (2)	2.2 (1)
O16	1.0045 (5)	0.3817 (5)	0.3939 (2)	3.5 (1)
O17	0.8112 (5)	0.2503 (5)	0.3553 (2)	3.0 (1)
C18	0.2882 (7)	0.0966 (6)	0.3845 (2)	2.5 (1)
C19	0.3201 (6)	−0.0338 (5)	0.3554 (2)	2.0 (1)
O20	0.1930 (5)	−0.0914 (4)	0.3352 (2)	2.5 (1)
O21	0.4655 (5)	−0.0760 (4)	0.3550 (2)	2.7 (1)
C22	0.4092 (7)	0.3061 (6)	0.3535 (2)	2.4 (1)
C23	0.4363 (6)	0.2517 (5)	0.3023 (2)	2.0 (1)
O24	0.3527 (5)	0.3029 (4)	0.2657 (1)	2.5 (1)
O25	0.5432 (5)	0.1596 (4)	0.2985 (1)	2.4 (1)
Zn2	0.1513 (1)	−0.2911 (1)	0.3051 (1)	2.1 (1)
O1*W*	0.1330 (5)	−0.3579 (4)	0.3785 (2)	2.7 (1)
O2*W*	−0.1064 (5)	−0.2567 (5)	0.3059 (2)	2.8 (1)
O3*W*	0.1247 (5)	−0.4927 (4)	0.2813 (2)	3.0 (1)
O4*W*	0.4076 (5)	−0.3239 (5)	0.3074 (2)	3.0 (1)
O5*W*	0.6984 (6)	0.9035 (5)	0.2337 (2)	3.7 (1)
O6*W*	0.9596 (6)	0.0871 (5)	0.2785 (2)	4.0 (1)

Table 2. *Selected geometric parameters* (Å, °)

Zn1—N8	2.191 (5)	C11—O12	1.228 (7)
Zn1—O13	2.029 (4)	C14—C15	1.508 (8)
Zn1—O21	2.141 (4)	C15—O17	1.269 (7)
Zn1—N9	2.197 (4)	C19—O21	1.253 (6)
Zn1—O17	2.119 (5)	C23—O24	1.262 (7)
Zn1—O25	2.053 (4)	C19—O20	1.264 (6)
Zn2—O20	2.114 (5)	C1—C6	1.389 (9)
Zn2—O1*W*	2.105 (4)	C2—C3	1.380 (9)
Zn2—O3*W*	2.060 (5)	C4—C5	1.408 (7)
Zn2—O24	2.119 (4)	C5—C6	1.397 (8)
Zn2—O2*W*	2.125 (4)	N8—C10	1.460 (7)
Zn2—O4*W*	2.107 (4)	N9—C18	1.473 (7)
C1—C2	1.367 (9)	C10—C11	1.532 (8)
C1—C7	1.521 (10)	C11—O13	1.279 (7)
C3—C4	1.399 (9)	C15—O16	1.244 (7)
C4—N8	1.453 (7)	C18—C19	1.519 (8)
C5—N9	1.452 (7)	C22—C23	1.511 (8)
N8—C14	1.479 (7)	C23—O25	1.255 (7)
N9—C22	1.484 (7)		

O21—Zn1—O25	85.3 (2)	C5—N9—C18	111.9 (4)
O17—Zn1—O21	165.9 (2)	N8—C10—C11	109.0 (5)
O13—Zn1—O21	86.5 (2)	C10—C11—O12	118.1 (5)
N9—Zn1—O25	79.9 (2)	C10—C11—O13	116.0 (5)
N9—Zn1—O17	102.3 (2)	O12—C11—O13	125.9 (6)
N8—Zn1—O25	148.5 (2)	C5—C4—N8	118.9 (5)
N8—Zn1—O17	79.2 (2)	C6—C5—N9	121.5 (5)
N8—Zn1—N9	80.3 (2)	C4—N8—C10	113.9 (5)
O17—Zn1—O25	81.4 (2)	C5—N9—C22	114.4 (5)
O13—Zn1—O25	128.2 (2)	C18—N9—C22	110.5 (4)
O13—Zn1—O17	98.0 (2)	C10—C11—O13	116.0 (5)
N9—Zn1—O21	79.9 (2)	O12—C11—O13	125.9 (5)
N9—Zn1—O13	147.8 (2)	C10—C11—O12	118.1 (5)
N8—Zn1—O21	114.8 (2)	N8—C14—C15	113.8 (4)
N8—Zn1—O13	79.2 (2)	C14—C15—O17	118.3 (5)
O20—Zn2—O24	87.0 (2)	O16—C15—O17	124.4 (6)
O24—Zn2—O3*W*	97.4 (2)	C18—C19—O21	118.3 (5)
O24—Zn2—O1*W*	171.1 (2)	C22—C23—O25	118.2 (5)
O20—Zn2—O3*W*	174.6 (2)	O24—C23—O25	123.4 (5)
O20—Zn2—O1*W*	86.4 (2)	Zn1—N9—C5	110.2 (3)
O2*W*—Zn2—O4*W*	177.7 (2)	Zn1—O25—C23	114.6 (4)
O1*W*—Zn2—O4*W*	93.7 (2)	Zn1—O21—C19	113.6 (4)
O1*W*—Zn2—O2*W*	84.2 (2)	C18—N9—Zn1	108.0 (4)
O24—Zn2—O4*W*	92.3 (2)	Zn1—N8—C10	101.9 (4)
O24—Zn2—O2*W*	89.6 (2)	C14—C15—O16	117.2 (5)
O20—Zn2—O4*W*	89.7 (2)	N9—C18—C19	114.3 (5)
O20—Zm2—O2*W*	89.1 (2)	N9—C22—C23	109.8 (5)
O3*W*—Zn2—O4*W*	87.0 (2)	C22—C23—O24	118.4 (5)
O2*W*—Zn2—O3*W*	94.0 (2)	O20—C19—O21	126.6 (6)
O1*W*—Zn2—O3*W*	89.5 (2)	C4—N8—Zn1	110.7 (3)
C3—C4—N8	121.4 (5)	Zn1—N9—C22	101.1 (3)
C4—C5—N9	119.4 (5)	Zn1—N8—C14	107.3 (3)
C4—N8—C14	111.6 (5)	Zn1—O13—C11	115.8 (4)
C10—N8—C14	110.8 (5)	Zn1—O17—C15	116.6 (4)

Calculated positions were used for the H atoms of the organic skeleton, all were refined will a common overall isotropic displacement parameter.

Data collection and cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *XRAY System* (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Structure solution: *SHELXS*86 (Sheldrick, 1985). Structure refinement: *SHELX*76 (Sheldrick, 1976). Software used to obtain parameters of interest: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1187). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(phenylthiolato-*S*)bis(tri-*n*-butylphosphine-*P*)nickel(II), $[Ni(SPh)_2(P^nBu_3)_2]$

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Abstract

The title compound, $[Ni(C_6H_5S)_2(C_{12}H_{27}P)_2]$, was isolated from the reaction of $NiCl_2.6H_2O$, NaSPh and P^nBu_3 in MeOH. The molecule possesses a crystallographic inversion center at the Ni atom which is fourfold coordinated by two P and two S atoms with almost square-planar geometry. The Ni—S and Ni—P distances are 2.217 (2) and 2.245 (2) Å, respectively.

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