C45	0.5398 (3)	0.8649 (3)	0.5317 (2)	0.0601 (9)
C46	0.4603 (4)	0.8705 (3)	0.5823 (3)	0.0764 (12)
C47	0.4216 (4)	0.7908 (3)	0.6254 (3)	0.0730 (11)
C48	0.4615 (3)	0.7059 (3)	0.6178 (2)	0.0613 (9)
C49	0.5435 (3)	0.6968 (2)	0.5686 (2)	0.0487 (8)
C50	0.7141 (3)	0.8953 (2)	0.4052 (2)	0.0492 (8)
C51	0.7871 (3)	0.9648 (2)	0.4486 (3)	0.0568 (9)
C52	0.8204 (4)	1.0454 (3)	0.4002 (3)	0.0734 (12)
C53	0.7825 (4)	1.0583 (3)	0.3143 (4)	0.0797 (13)
C54	0.7060 (4)	0.9927 (3)	0.2717 (3)	0.0733 (11)
C55	0.6726 (3)	0.9117 (3)	0.3179 (2)	0.0590 (9)
C56	0.9394 (3)	0.7715 (3)	0.3957 (2)	0.0598 (9)
C57	0.5803 (4)	0.5997 (2)	0.5647 (2)	0.0643 (10)
C58	0.8314 (4)	0.9598 (3)	0.5425 (3)	0.0778 (12)

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

Rh—C01	1.783 (3)	P2—O2	1.521 (2)
Rh—O1	2.082 (2)	P2—C1	1.742 (3)
Rh—O2	2.094 (2)	P3—O3	1.489 (2)
Rh—P4	2.2313 (10)	P3—C1	1.761 (3)
P1—O1	1.526 (2)	C01—O01	1.151 (4)
P1—C1	1.755 (3)		
C01—Rh—O1	174.02 (12)	O3—P3—C1	116.56 (15)
C01—Rh—O2	92.70 (12)	P1—O1—Rh	124.27 (13)
O1—Rh—O2	84.92 (8)	P2—O2—Rh	125.43 (12)
C01—Rh—P4	93.69 (11)	O01—C01—Rh	174.8 (3)
O1—Rh—P4	89.38 (7)	P2—C1—P1	110.2 (2)
O2—Rh—P4	170.38 (6)	P2—C1—P3	127.1 (2)
O1—P1—C1	114.76 (14)	P1—C1—P3	120.7 (2)
O2—P2—C1	109.71 (14)		

Data collection and cell refinement: Syntex $P\bar{1}$ software. Structure solution: *SHELXS*86 (Sheldrick, 1990) to obtain the position of the heavy atom and *SHELXL*93 (Sheldrick, 1993) to solve the structure *via* successive Fourier maps. Structure refinement: *SHELXS*86. Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, bond distances and angles involving non-H atoms and least-squares-planes data have been deposited with the IUCr (Reference: MU1160). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Dinuclear Palladium Compound, $[Pd_2(PPh_3)_2(SC_2H_4S)_2]$

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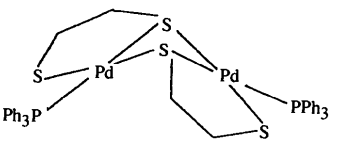
Abstract

The title compound, bis(μ-1,2-ethanedithiolato-*S*,*S*′:*S*)-bis[(triphenylphosphine)palladium(II)], is a dimer where the Pd atoms are linked by two S atoms, one from each $SC_2H_4S^{2-}$ ligand; each Pd atom is four-coordinate (one P and three S atoms) with a distorted square-planar geometry. The Pd···Pd distance is 3.038 (2) Å, and the average Pd—S and Pd—P distances are 2.332 (5) and 2.281 (5) Å, respectively.

Comment

The nickel group compounds with mixed sulfur and phosphine ligands have attracted attention because of their relevance and importance to a wide variety of chemical and industrial systems. Up to now, some dinuclear compounds with mixed thiolate and phosphine ligands, such as $[Pd_2(SC_6F_5)_2(PPh_3)_2]$ (Fenn & Segrott, 1972), $[Pt_2(SCH_2Ph)_4(PMePh_2)_2]$ (Bird, Siriwardane, Lai & Shaver, 1982) and $[\{Pt_2(SCH_2CH_2CMe{=}CH_2)_2\}_2(PPh_3)_2I_2]$ (Abel *et al.*, 1990), have been structurally characterized. We have recently reported dinickel compounds: $[Ni_2(PPh_3)_2(SC_2H_4S)_2]$ (Cao, Huang, Lei, Hong & Liu, 1992) and $[Ni_2(PPh_3)_2(SC_3H_6S)_2]$ (Cao, Huang, Lei, Kang, Hong & Liu, 1992). Here, we report the crystal structure of $[Pd_2(PPh_3)_2(SC_2H_4S)_2]$, (I), which is isomorphic with $[Ni_2(PPh_3)_2(SC_2H_4S)_2]$ and consists of two palladium quadrilaterals sharing a common edge.



(I)

The two Pd atoms are bridged by two S atoms, one from each $SCH_2CH_2S^{2-}$ ligand. Each Pd atom is surrounded by one P and three S atoms in an approximately square-planar arrangement. Fig. 1 shows the structure with the atomic numbering scheme.

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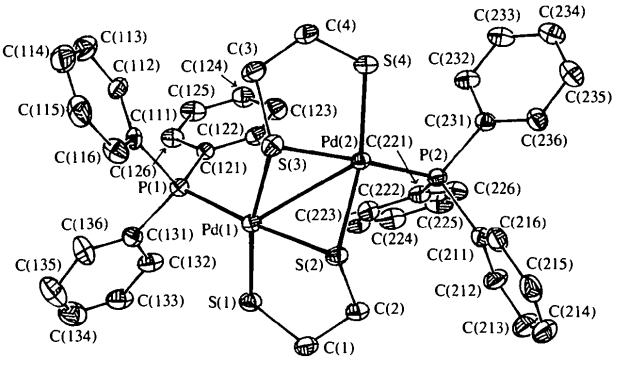

Fig. 1. Molecular structure of $[Pd_2(C_2H_4S_2)_2(C_{18}H_{15}P)_2]$. Displacement ellipsoids are plotted at the 30% probability level.

Experimental

The title compound was prepared by the reaction of $PdCl_2$, $Na_2SC_2H_4S$ and PPh_3 in the molar ratio 1:1:2 in MeOH and was recrystallized from CH_2Cl_2/MeOH solution.

Crystal data

$[Pd_2(C_2H_4S_2)_2(C_{18}H_{15}P)_2]$
$M_r = 921.75$
Triclinic
$P\bar{1}$
$a = 10.879\ (5)$ Å
$b = 17.591\ (7)$ Å
$c = 10.649\ (5)$ Å
$\alpha = 102.96\ (4)°$
$\beta = 97.46\ (3)°$
$\gamma = 81.64\ (3)°$
$V = 1955\ (2)$ Å^3
$Z = 2$
$D_x = 1.566$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 9$–$12°$
$\mu = 1.2213$ mm^{-1}
$T = 296$ K
Rectangular
0.65 × 0.55 × 0.20 mm
Violet

Data collection

MSC/Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans before and refined from ΔF (*DIFABS*; Walker & Stuart, 1983) during refinement
7280 measured reflections
7156 independent reflections
5949 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.023$
$\theta_{max} = 25°$
$h = 0 \rightarrow 12$
$k = -20 \rightarrow 20$
$l = -12 \rightarrow 12$
3 standard reflections monitored every 250 reflections
intensity decay: none

Refinement

Refinement on *F*
$R = 0.041$
$wR = 0.047$
$S = 3.327$
5949 reflections
433 parameters
H atoms not located
Unit weights applied
$(\Delta/\sigma)_{max} = 0.08$
$\Delta\rho_{max} = 0.78$ e Å^{-3}
$\Delta\rho_{min} = -0.53$ 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)

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	*y*	*z*	B_{eq}
Pd(1)	0.8837 (1)	0.14807 (7)	0.3391 (1)	2.47 (3)
Pd(2)	0.8811 (1)	0.32547 (7)	0.4034 (1)	2.60 (3)
S(1)	0.8862 (4)	0.0788 (3)	0.1295 (4)	3.5 (1)
S(2)	1.0238 (4)	0.2251 (3)	0.2968 (4)	3.1 (1)
S(3)	0.9103 (4)	0.2366 (3)	0.5418 (4)	3.3 (1)
S(4)	0.7475 (5)	0.4062 (3)	0.5408 (5)	4.4 (1)
P(1)	0.7251 (4)	0.0853 (2)	0.3722 (4)	2.66 (9)
P(2)	0.8419 (4)	0.4044 (3)	0.2561 (4)	2.9 (1)
C(1)	1.017 (2)	0.1142 (10)	0.072 (2)	4.0 (5)
C(2)	1.013 (2)	0.2030 (10)	0.117 (2)	3.7 (4)
C(3)	0.776 (2)	0.2708 (10)	0.639 (2)	4.4 (5)
C(4)	0.771 (2)	0.3592 (10)	0.682 (2)	4.8 (5)
C(111)	0.724 (2)	0.0770 (9)	0.5412 (10)	2.8 (4)
C(112)	0.616 (2)	0.0943 (10)	0.605 (2)	3.7 (4)
C(113)	0.622 (2)	0.0906 (10)	0.733 (2)	4.9 (6)
C(114)	0.734 (2)	0.0659 (10)	0.797 (2)	5.5 (6)
C(115)	0.843 (2)	0.0494 (10)	0.736 (2)	5.4 (6)
C(116)	0.838 (2)	0.0548 (10)	0.607 (2)	4.4 (5)
C(121)	0.574 (1)	0.1401 (9)	0.333 (1)	2.4 (3)
C(122)	0.570 (1)	0.2174 (10)	0.324 (2)	3.4 (4)
C(123)	0.456 (2)	0.2621 (10)	0.295 (2)	4.5 (5)
C(124)	0.347 (2)	0.2291 (10)	0.280 (2)	4.1 (5)
C(125)	0.350 (2)	0.1509 (10)	0.292 (2)	4.2 (5)
C(126)	0.462 (2)	0.1054 (10)	0.318 (2)	3.6 (4)
C(131)	0.714 (2)	−0.0160 (10)	0.275 (2)	3.4 (4)
C(132)	0.661 (2)	−0.0243 (10)	0.145 (2)	4.2 (5)
C(133)	0.660 (2)	−0.0991 (10)	0.067 (2)	5.3 (6)
C(134)	0.710 (2)	−0.1639 (10)	0.115 (2)	6.2 (7)
C(135)	0.760 (3)	−0.1566 (10)	0.238 (3)	7.2 (8)
C(136)	0.763 (2)	−0.0805 (10)	0.323 (2)	5.2 (6)
C(211)	0.982 (2)	0.4071 (9)	0.175 (2)	3.6 (4)
C(212)	0.979 (2)	0.3935 (10)	0.041 (2)	5.0 (6)
C(213)	1.093 (3)	0.3916 (10)	−0.011 (2)	6.5 (7)
C(214)	1.201 (2)	0.4053 (10)	0.066 (3)	6.5 (7)
C(215)	1.204 (2)	0.4194 (10)	0.199 (3)	5.8 (7)
C(216)	1.093 (2)	0.4202 (10)	0.255 (2)	4.3 (5)
C(221)	0.722 (2)	0.3723 (10)	0.122 (2)	3.3 (4)
C(222)	0.693 (2)	0.2943 (10)	0.101 (2)	3.7 (4)
C(223)	0.601 (2)	0.2685 (10)	−0.000 (2)	5.1 (6)
C(224)	0.542 (2)	0.3186 (10)	−0.077 (2)	5.1 (6)
C(225)	0.570 (2)	0.3939 (10)	−0.055 (2)	5.0 (5)
C(226)	0.661 (2)	0.4226 (10)	0.044 (2)	4.2 (5)
C(231)	0.780 (2)	0.5082 (9)	0.3134 (10)	3.1 (4)
C(232)	0.655 (2)	0.5242 (10)	0.342 (2)	4.1 (5)
C(233)	0.606 (2)	0.6019 (10)	0.387 (2)	4.7 (5)
C(234)	0.679 (2)	0.6612 (10)	0.404 (2)	4.7 (5)
C(235)	0.803 (2)	0.6458 (10)	0.377 (2)	4.7 (5)
C(236)	0.856 (2)	0.5678 (10)	0.333 (2)	3.7 (4)

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

Pd(1)—Pd(2)	3.038 (2)	Pd(2)—P(2)	2.281 (5)
Pd(1)—S(1)	2.292 (4)	S(1)—C(1)	1.86 (2)
Pd(1)—S(2)	2.327 (5)	S(2)—C(2)	1.86 (2)
Pd(1)—S(3)	2.372 (4)	S(3)—C(3)	1.86 (3)
Pd(1)—P(1)	2.281 (5)	S(4)—C(4)	1.85 (2)
Pd(2)—S(2)	2.362 (4)	C(1)—C(2)	1.52 (3)
Pd(2)—S(3)	2.342 (6)	C(3)—C(4)	1.52 (3)
Pd(2)—S(4)	2.300 (6)		
S(1)—Pd(1)—S(2)	88.6 (2)	S(3)—Pd(2)—S(4)	88.7 (2)
S(1)—Pd(1)—S(3)	167.4 (2)	S(3)—Pd(2)—P(2)	174.8 (2)
S(1)—Pd(1)—P(1)	91.6 (2)	S(4)—Pd(2)—P(2)	92.8 (2)
S(2)—Pd(1)—S(3)	78.9 (2)	Pd(1)—S(1)—C(1)	103.0 (5)
S(2)—Pd(1)—P(1)	172.0 (2)	Pd(1)—S(2)—Pd(2)	80.8 (1)
S(3)—Pd(1)—P(1)	100.8 (2)	Pd(1)—S(2)—C(2)	103.0 (6)
S(2)—Pd(2)—S(3)	78.8 (2)	Pd(2)—S(2)—C(2)	119.1 (6)
S(2)—Pd(2)—S(4)	167.4 (2)	Pd(1)—S(3)—Pd(2)	80.2 (1)
S(2)—Pd(2)—P(2)	99.9 (2)	Pd(1)—S(3)—C(3)	119.5 (7)
Pd(2)—S(3)—C(3)	101.9 (7)	Pd(2)—S(4)—C(4)	102.4 (6)

Program used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program used to refine structure: *LSFM* (B. A. Frenz & Associates Inc., 1985). Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: AB1192). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sodium 4-Hydroxy-3-nitrobenzenesulfonate Trihydrate

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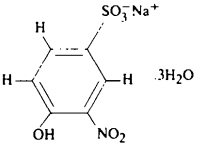
Abstract

The title compound, $Na^+.C_6H_4NO_6S^-.3H_2O$, crystallizes in double layers with the nearly planar organic anions oriented parallel to one another and the sulfonate groups directed towards the center of the sandwich, where they are bonded to Na^+ cations. Each Na^+ cation bonds to six O atoms in a distorted octahedral geometry. The coordination sphere contains two sulfonate O atoms, one nitro O atom from a different anion and three water molecules. There are hydrogen bonds involving the water molecules, the sulfonate O atoms and the hydroxyl groups.

Comment

In an earlier study of layered metal–arylsulfonate compounds (Shubnell, Kosnic & Squattrito, 1994), sodium 4-hydroxy-3-nitrobenzenesulfonate hemihydrate, $C_6H_4NO_6NaS.\frac{1}{2}H_2O$, was identified as a minor reaction product. We subsequently confirmed that the reported trihydrate (King, 1921) is the usual product of the nitration of sodium 4-hydroxybenzenesulfonate in aqueous nitric acid. Having determined the structure of the hemihydrate, it was of interest to see how the structure of the trihydrate, (I), would differ.



(I)

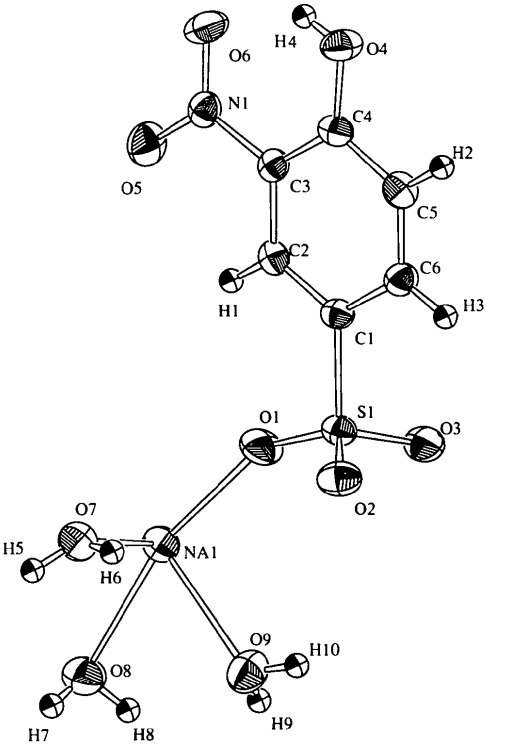


Fig. 1. *ORTEP*II (Johnson, 1976) diagram of the title compound showing the atom-labeling scheme. In this and Fig. 2, the displacement ellipsoids of the non-H atoms are shown at the 50% probability level.