

Table 2. Bond lengths (Å) and angles (°) for $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$

Au(1)–P(1)	2.311 (3)	Au(1)–P(2)	2.310 (3)
Au(1)–Au(1')	2.931 (1)	P(1)–C(16)	1.801 (7)
P(1)–C(36)	1.814 (6)	P(1)–C(1')	1.819 (8)
P(2)–C(1)	1.839 (8)	P(2)–C(26)	1.810 (6)
P(2)–C(46)	1.797 (7)	F(1)–B(1)	1.336 (20)
F(2)–B(1)	1.391 (17)	F(3)–B(1)	1.313 (19)
F(4)–B(1)	1.393 (27)	F(2a)–B(1)	1.256 (39)
F(3a)–B(1)	1.446 (25)	F(4a)–B(1)	1.439 (23)
P(1)–Au(1)–P(2)	177.3 (1)	P(1)–Au(1)–Au(1')	90.4 (1)
P(2)–Au(1)–Au(1')	92.3 (1)	Au(1)–P(1)–C(16)	112.7 (2)
Au(1)–P(1)–C(36)	111.4 (2)	C(16)–P(1)–C(36)	105.1 (4)
Au(1)–P(1)–C(1')	112.5 (4)	C(16)–P(1)–C(1')	107.4 (3)
C(36)–P(1)–C(1')	107.3 (3)	Au(1)–P(2)–C(1)	110.6 (3)
Au(1)–P(2)–C(26)	113.2 (3)	C(1)–P(2)–C(26)	107.6 (3)
Au(1)–P(2)–C(46)	113.2 (2)	C(1)–P(2)–C(46)	108.3 (5)
C(26)–P(2)–C(46)	103.5 (3)	F(1)–B(1)–F(2)	108.8 (13)
F(1)–B(1)–F(3)	115.4 (16)	F(2)–B(1)–F(3)	118.5 (12)
F(1)–B(1)–F(4)	105.8 (12)	F(2)–B(1)–F(4)	101.0 (15)
F(3)–B(1)–F(4)	105.5 (15)	F(1)–B(1)–F(2a)	122.3 (17)
F(2)–B(1)–F(2a)	40.5 (15)	F(3)–B(1)–F(2a)	122.3 (20)
F(4)–B(1)–F(2a)	60.8 (17)	F(1)–B(1)–F(3a)	102.3 (17)
F(2)–B(1)–F(3a)	86.3 (11)	F(3)–B(1)–F(3a)	44.9 (10)
F(4)–B(1)–F(3a)	146.7 (16)	F(2a)–B(1)–F(3a)	116.7 (17)
F(1)–B(1)–F(4a)	114.4 (13)	F(2)–B(1)–F(4a)	136.6 (18)
F(3)–B(1)–F(4a)	42.8 (11)	F(4)–B(1)–F(4a)	64.5 (14)
F(2a)–B(1)–F(4a)	108.2 (23)	F(3a)–B(1)–F(4a)	87.8 (13)
P(2)–C(1)–P(1')	112.4 (4)	P(1)–C(16)–C(11)	120.5 (2)
P(1)–C(16)–C(15)	119.5 (2)	P(2)–C(26)–C(21)	122.4 (2)
P(2)–C(26)–C(25)	117.6 (2)	P(1)–C(36)–C(31)	122.9 (2)
P(1)–C(36)–C(35)	116.9 (2)	P(2)–C(46)–C(41)	120.5 (2)
P(2)–C(46)–C(45)	119.5 (2)		

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Absolute Structure of Di- μ -sulfoacetato-bis{[(1*R*,2*R*)-1,2-diaminocyclohexane]-platinum(II)} Trihydrate

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Abstract. $[\text{Pt}_2(\text{C}_2\text{H}_2\text{O}_5\text{S})_2(\text{C}_6\text{H}_{14}\text{N}_2)_2] \cdot 3\text{H}_2\text{O}$, $M_r = 948.80$, orthorhombic, $P2_12_12_1$, $a = 19.186$ (4), $b = 14.577$ (2), $c = 9.876$ (1) Å, $V = 2762$ (1) Å³, $Z = 4$, $D_x = 2.28$, $D_m = 2.25$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 10.859$ mm⁻¹, $F(000) = 1816$, $T = 298$ K, $R = 0.033$, $wR = 0.031$ for 2455 independent reflections. Formal local charges on Pt atoms and SO₃ groups are not compensated and the molecule has a divalent zwitterionic character. The geometric requirement imposed by the bridging carboxyl group causes the relatively short Pt–Pt distance [3.0435 (8) Å] but it

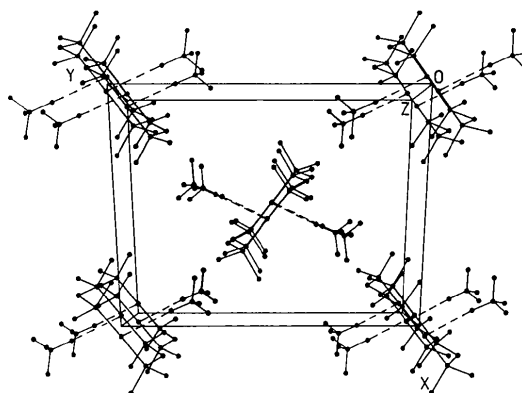


Fig. 2. Packing diagram of $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$. The shortest $\text{BF}_4^- \cdots \text{Au}$ separation, 3.28 Å, $\text{Au}(1) \cdots \text{F}(3)$, is indicated by the dashed line. Only one orientation of the disordered BF_4^- is shown.

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is slightly too long to be regarded as a metal–metal bond. Coordination planes around Pt are not parallel because of the steric repulsion between the cyclohexane rings (dihedral angle between the coordination planes is 42.9°).

Experimental. The title compound was synthesized from a reaction of dihydroxyl[(1*R*,2*R*)-1,2-diaminocyclohexane]platinum(II) and sulfoacetic acid and was recrystallized from H₂O. Yellow prism crystals; 0.23 × 0.15 × 0.10 mm; D_m by flotation in C₂H₄Br₂–CH₂I₂.

Table 1. Final positional parameters ($\times 10^4$; $\times 10^5$ for Pt) and thermal parameters ($B_{eq}/\text{\AA}^2$) of non-H atoms
$$B_{eq} = \frac{1}{3} \pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	B_{eq}
Pt1	98939 (5)	34598 (3)	73043 (8)	2.56 (3)
Pt2	98915 (5)	13719 (3)	72916 (8)	2.27 (3)
S1	11814 (3)	2423 (4)	4149 (5)	4.5 (2)
S2	9206 (2)	2368 (3)	12110 (4)	3.2 (2)
O1	10910 (7)	3172 (8)	6877 (14)	3.5 (7)
O2	10893 (8)	1634 (8)	6749 (13)	3.5 (7)
O3	11438 (12)	1633 (11)	3809 (17)	7 (1)
O4	12518 (7)	2398 (17)	3663 (14)	8 (1)
O5	11454 (13)	3274 (11)	3828 (19)	8 (1)
O11	10017 (9)	3198 (6)	9325 (10)	3.3 (6)
O12	10011 (8)	1686 (6)	9299 (11)	3.0 (6)
O13	8905 (9)	1537 (9)	11631 (15)	4.9 (8)
O14	9345 (6)	2360 (9)	13579 (9)	4.6 (6)
O15	8818 (9)	3165 (10)	11640 (16)	5.6 (9)
O21	8143 (6)	2485 (11)	8935 (12)	4.9 (6)
O22	8981 (11)	4887 (11)	301 (18)	4.5 (9)
O23	10958 (11)	4910 (8)	4801 (15)	3.3 (8)
N1	9723 (9)	3862 (7)	5403 (13)	2.5 (6)
N2	8958 (9)	3982 (8)	7630 (17)	3.4 (8)
N11	9728 (9)	860 (8)	5419 (14)	2.9 (7)
N12	8894 (8)	968 (8)	7659 (17)	2.5 (6)
C1	9014 (9)	4247 (10)	5210 (18)	2.9 (8)
C2	8808 (10)	4660 (11)	6560 (18)	2.8 (8)
C3	8089 (14)	5036 (13)	6530 (33)	5 (1)
C4	8006 (14)	5704 (19)	5391 (38)	8 (2)
C5	8239 (16)	5280 (18)	4002 (28)	7 (2)
C6	8951 (20)	4910 (14)	4031 (22)	4 (1)
C11	11180 (8)	2406 (14)	6614 (14)	3.1 (7)
C12	11869 (8)	2387 (16)	5914 (16)	4.1 (9)
C21	10029 (8)	2437 (10)	9867 (11)	2.6 (6)
C22	10055 (8)	2426 (11)	11420 (12)	3.2 (6)
C31	9150 (10)	184 (11)	5547 (18)	2.6 (8)
C32	8588 (10)	642 (10)	6361 (17)	2.8 (8)
C33	7975 (13)	-32 (12)	6582 (30)	4 (1)
C34	7692 (12)	-414 (16)	5278 (23)	5 (1)
C35	8283 (13)	-845 (13)	4480 (24)	5 (1)
C36	8900 (16)	-186 (17)	4261 (21)	3 (1)

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Pt1—Pt2	3.0435 (8)	Pt1—O1	2.04 (1)	Pt1—O11	2.05 (1)
Pt1—N1	1.99 (1)	Pt1—N2	1.98 (2)	Pt2—O2	2.03 (2)
Pt2—O12	2.05 (1)	Pt2—N11	2.02 (1)	Pt2—N12	2.04 (1)
S1—O3	1.40 (2)	S1—O4	1.43 (1)	S1—O5	1.46 (2)
S1—C12	1.75 (2)	S2—O13	1.42 (1)	S2—O14	1.48 (1)
S2—O15	1.45 (1)	S2—C22	1.77 (2)	O1—C11	1.26 (2)
O2—C11	1.26 (2)	O11—C21	1.23 (1)	O12—C21	1.23 (1)
N1—C1	1.48 (2)	N2—C2	1.48 (2)	N11—C31	1.49 (2)
N12—C32	1.49 (2)	C1—C2	1.52 (2)	C1—C6	1.52 (3)
C2—C3	1.48 (3)	C3—C4	1.50 (4)	C4—C5	1.57 (4)
C5—C6	1.47 (4)	C11—C12	1.49 (2)	C21—C22	1.53 (2)
C31—C32	1.50 (2)	C31—C36	1.46 (3)	C32—C33	1.55 (3)
C33—C34	1.50 (3)	C34—C35	1.52 (3)	C35—C36	1.54 (3)
Pt2—Pt1—O1	78.2 (3)	Pt2—Pt1—O11	79.5 (3)		
Pt2—Pt1—N2	106.8 (3)	Pt2—Pt1—N2	112.6 (4)		
O1—Pt1—O11	93.0 (6)	O1—Pt1—N1	91.3 (6)		
O1—Pt1—N2	169.0 (5)	O11—Pt1—N1	173.0 (5)		
O11—Pt1—N2	91.1 (7)	N1—Pt1—N2	83.7 (7)		
Pt1—Pt2—O2	79.1 (3)	Pt1—Pt2—O12	76.8 (2)		
Pt1—Pt2—N11	112.0 (3)	Pt1—Pt2—N12	106.8 (3)		
O2—Pt2—O12	96.2 (6)	O2—Pt2—N11	88.6 (6)		
O2—Pt2—N12	172.4 (5)	O12—Pt2—N11	170.8 (5)		
O12—Pt2—N12	89.9 (7)	N11—Pt2—N12	84.8 (7)		
O3—S1—O4	113 (1)	O3—S1—O5	114 (1)		
O3—S1—C12	104 (1)	O4—S1—O5	113 (1)		
O4—S1—C12	106.1 (9)	O5—S1—C12	106 (1)		
O13—S2—O15	113.2 (9)	O13—S2—O15	111.5 (8)		
O13—S2—C22	106.6 (9)	O14—S2—O15	114 (1)		
O14—S2—C22	102.3 (7)	O15—S2—C22	108.1 (9)		
Pt1—O1—C11	128 (1)	Pt2—O2—C11	127 (1)		
Pt1—O11—C21	126.5 (8)	Pt2—O12—C21	130.1 (9)		
Pt1—N2—C1	112 (1)	Pt1—N2—C2	109 (1)		
Pt2—N11—C31	106 (1)	Pt2—N12—C32	108 (1)		
N1—C1—C2	106 (1)	N1—C1—C6	114 (2)		
C2—C1—C6	114 (2)	N2—C2—C1	108 (1)		
N2—C2—C3	116 (2)	C1—C2—C3	112 (2)		
C2—C3—C4	111 (2)	C3—C4—C5	112 (2)		
C4—C5—C6	113 (2)	C1—C6—C5	109 (2)		
O1—C11—O2	126 (1)	O1—C11—C12	118 (2)		
O2—C11—C12	115 (2)	S1—C12—C11	114 (1)		
O11—C21—O12	127 (1)	O11—C21—C22	116 (1)		
O12—C21—C22	117 (1)	S2—C22—C21	111 (1)		
N11—C31—C32	107 (1)	N11—C31—C36	114 (2)		
C32—C31—C36	113 (2)	N12—C32—C31	109 (2)		
N12—C32—C33	112 (2)	C31—C32—C33	110 (1)		
C32—C33—C34	113 (2)	C33—C34—C35	109 (2)		
C34—C35—C36	113 (2)	C31—C36—C35	111 (2)		

Rigaku AFC-5R goniometer at Research Centre for Spectrochemistry, Faculty of Science, The University of Tokyo; Mo K α radiation (graphite-monochromatized), 40 kV, 120 mA; cell parameters from 24 reflections ($12.5 < \theta < 22.5^\circ$); ω - 2θ scan at $8^\circ \text{ min}^{-1}(\omega)$; $5 \leq 2\theta \leq 55^\circ$ ($0 \leq h \leq 29$, $0 \leq k \leq 22$, $0 \leq l \leq 14$); three standard reflections (200, 020, 002) monitored every 150 reflections varied within $\pm 5.0\%$; an empirical absorption correction by ψ -scan based on 020, 040, 060, 080, 0,10,0, 0,12,0, 0,14,0; of the 3621 measured reflections, 2455 reflections with $F_o \geq 3.0\sigma(F_o)$ were used for calculation. Structure solved by the heavy-atom method; absolute configuration determined by anomalous scattering factors; enantiomeric structure rejected (final $R = 0.040$, $wR = 0.037$ for the enantiomeric structure); H atoms except crystal water located from calculation and not refined; full-matrix least-squares refinement on F ; anisotropic thermal parameters for non-H atoms and isotropic ones for H atoms; final $R = 0.033$, $wR = 0.031$, $w = 1/\sigma^2(F_o)$, $S = 1.24$, $(\Delta/\sigma)_{\text{max}}$ for non-H atoms 0.63, $(\Delta\rho)_{\text{max}}$ 0.88 e \AA^{-3} . All calculations used TEXSAN (Sweepston, 1986) with complex scattering factors from *International Tables for X-ray Crystallography* (1974) and carried out on MicroVAX II at Research Centre of Spectrochemistry. Final atomic

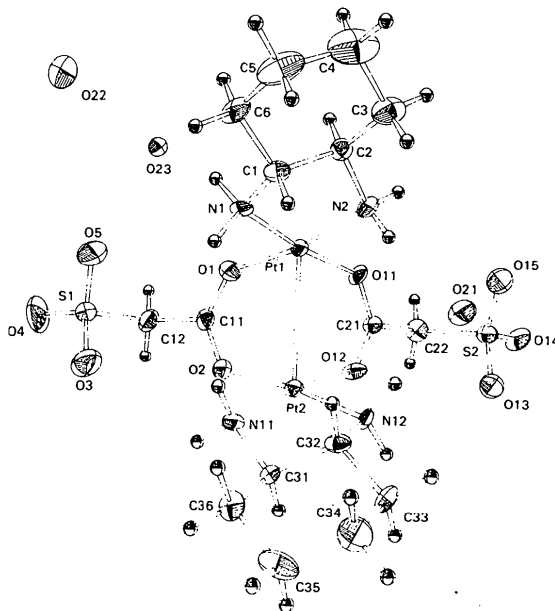


Fig. 1. An ORTEP (Johnson, 1976) view of the title compound. Thermal ellipsoids are scaled to enclose 30% probability for non-H atoms.

parameters are presented in Table 1* and bond lengths and angles in Table 2. A view of the molecule is shown in Fig. 1.

Related literature. In the molecules of the α -pyridonate-bridged *cis*-diammineplatinum isomers (Hollis & Lippard, 1983*a*), the Pt–Pt distance is 2.8981 (5) Å for the head-to-tail dimer, 2.8767 (7) and 3.1294 (4) Å for the head-to-head tetramer; the dihedral angle between the coordination plane is 28.8° for the head-to-tail dimer, 30.0° for the head-to-head tetramer.

* Lists of structure factors, anisotropic thermal parameters, and complete lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51650 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

In the head-to-head isomer of bis(μ -2-pyridonate-*N,O*-bis[(ethylenediamine)platinum(II)] (Hollis & Lippard, 1983*b*), the Pt–Pt distance is 2.9915 (4) and 3.2355 (5) Å and the dihedral angle between the coordination planes is 39.6°.

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Structure of Bis(*N,N*-diisobutyldiselenocarbamato)palladium(II), Pd[Se₂CN(*i*Bu)₂]₂

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Abstract. [Pd(C₉H₁₈NSe₂)₂], *M_r* = 702.7, monoclinic, *P*2₁/*c*, *a* = 6.684 (1), *b* = 12.393 (3), *c* = 15.648 (3) Å, β = 99.12 (1)°, *V* = 1279.8 (4) Å³, *Z* = 2, *D_x* = 1.83 g cm⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 63.5 cm⁻¹, *F*(000) = 680, *T* = 295 K. Final *R* = 0.0325 for 1274 unique observed reflections. The Pd atom occupies a center of symmetry in the molecule. The N–C bond distance [1.310 (8) Å] shows significant double-bond character.

Experimental. Crystals of bis(*N,N*-diisobutyldiselenocarbamato)palladium(II) were prepared according to Pan & Fackler (1978). An orange-red irregularly shaped crystal of dimensions 0.3 × 0.3 × 0.5 mm was mounted on a glass fiber. Cell constants were derived from least-squares refinement of 25 reflections having $10 < 2\theta < 26^\circ$. Intensity data were collected using ω -scan technique with $0 < 2\theta < 45^\circ$ (*h* = 0 to 14, *k* = 0 to 14, *l* = –17 to 17) on a Nicolet R3m/E diffractometer using graphite-monochromated Mo *K* α radiation. Three standard reflections (032, $\bar{1}22$, 12 $\bar{3}$), measured every 197 reflections, showed small (<4%) random variations. Data were corrected for Lorentz

and polarization effects, and for absorption effects using the empirical method: *T*_{min} = 0.472, *T*_{max} = 0.571. 1963 data were collected and averaged to 1274 unique observed reflections [*F*_o² > 3 σ (*F*_o²); *R*_{int}(*F*_o) = 0.039]. Scattering factors, including terms for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974). All computations were carried out using the *SHELXTL* crystallographic package (Sheldrick, 1986).

Pd was assigned to the special position (0, $\frac{1}{2}$, 0); all other non-H atoms were located by Fourier synthesis. Some (ten out of 18) of the H atoms were revealed by difference Fourier maps. These H atoms were not included in the least-squares refinements but they were included in the final structure-factor calculations. Non-H atoms were refined anisotropically. A partial correction for secondary extinction was applied by multiplying *F*_c by $|1 + 0.002xF_c^2(\sin 2\theta)|^{-1/4}$; refinement gave *x* = 0.00095. $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = [\sigma^2(F_o) + 0.00046F_o^2]^{-1}$; the final *R* value was 0.033, *wR* = 0.043 and *S* = 1.396 for 116 variables. The largest shift/e.s.d. in the final least-squares cycle was 0.005; the maximum and minimum residual electron densities in the difference Fourier map were less than 0.5 e Å⁻³. The molecule and the atomic

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