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[(1*R*,2*R*)-1,2-Diaminocyclohexane]bis(5-phenyldibenzo[*b*,*d*]phosphole)platinum(II) Dinitrate Dihydrate

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Abstract. $[Pt(C_6H_{14}N_2)(C_{18}H_{13}P)_2](NO_3)_2.2H_2O$, M_r = 989·87, triclinic, $P\bar{1}$, a = 11·965 (2), b = 17·064 (2), c = 11·304 (2) Å, α = 101·62 (1), β = 111·52 (1), γ = 98·01 (1)°, V = 2045 (1) Å^3, Z = 2, D_x = 1·61, D_m = 1·61 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 3·75 mm^{-1}, $F(000)$ = 992, T = 296 K, R = 0·041 and wR = 0·055 for 10 096 independent reflections with $F_o > 3{\cdot}0\sigma(F_o)$. The structure contains two independent $[Pt(C_6H_{14}N_2)(C_{18}H_{13}P)_2]$ cations. The Pt atoms are square-geometrically coordinated with Pt—P = 2·261 (5) and 2·279 (5) Å, Pt—N = 2·07 (1) and 2·109 (8) Å, P—Pt—P = 93·9 (2)° in cation 1 and Pt—P = 2·211 (6) and 2·225 (5) Å, Pt—N = 2·15 (1) and 2·115 (9) Å, P—Pt—P = 91·6 (2)° in cation 2. The conformations of the two independent cationic complexes differ from each other in the orientation of the almost planar dibenzophosphole (dbp) group.

Experimental. A solution of 5-phenyldibenzophosphole (0·0358 g) in acetone (30 ml) was added to an aqueous solution (8 ml) of 0·0297 g [(1*R*,2*R*)-1,2-diaminocyclohexane]dinitratoplatinum(II) at room temperature and the reaction mixture was evaporated to give a white solid product. Colorless prisms were obtained by recrystallization from chloroform. Crystal 0·5 × 0·3 × 0·2 mm; Rigaku AFC-5R diffractometer; Mo $K\alpha$ radiation (graphite monochromated); ω–2θ scan at 6° min^{-1} (ω); Lorentz–polarization corrections; empirical absorption correction applied; lattice constants by least squares from 25 reflections ($24{\cdot}6 < \theta < 24{\cdot}9°$); $(\sin\theta)/\lambda < 0{\cdot}76$ Å^{-1} ($0 \le h \le 18$, $-20 \le k \le 25$, $-17 \le l \le 17$); three standard reflections monitored every 150 reflections varied within ± 0·6%; of the 12 646 measured reflections, 10 096 unique with $F_o > 3{\cdot}0\sigma(F_o)$ were regarded as observed; structure was solved by the heavy-atom method; H atoms were not located; refinement (on F) was by full-matrix least squares, anisotropic thermal parameters for Pt and P atoms being applied; R = 0·041, wR = 0·055, S = 1·57, $(\Delta/\sigma)_{max}$ = 0·24, $(\Delta\rho)_{max}$ = 1·92 e Å^{-3}; $w^{-1} = \sigma^2(F_o) + (0{\cdot}01|F_o|)^2$; complex scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV); all calculations on MicroVAX II computers, performed with *TEXSAN* (Swepston, 1986). Final atomic parameters are presented in Table 1.* The dihedral angles between some least-squares planes are presented in Table 2. Fig. 1 shows a view of the structure.

Related literature. In the crystals of other dbp compounds, the dihedral angles between bridged benzene rings of dbp have values in the range 2·7–6·7° (Alyea, Ferguson, Malito & Ruhl, 1986; Watkin, 1976; Ashwell, Allen, Kennedy & Nowell, 1982; Chui & Powell, 1974). The Pt—P distances in the Pt^{II}dbp derivatives (Chui & Powell, 1974) were found in the range 2·25–2·34 Å. Pt^{II}(PPh$_3$)$_2$ derivatives have Pt—P distances and P—Pt—P angles in the range 2·22–2·36 Å and 96·9–99·8°, respectively (Gregg, Powell & Sawyer, 1988; Hallock, Galiano-Roth & Collum, 1988; Biefeld, Eick & Grubbs, 1973; Bhaduri, Johnson, Pickard, Raithby, Sheldrick & Zuccaro, 1977).

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, intermolecular distances and equations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52716 (47 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

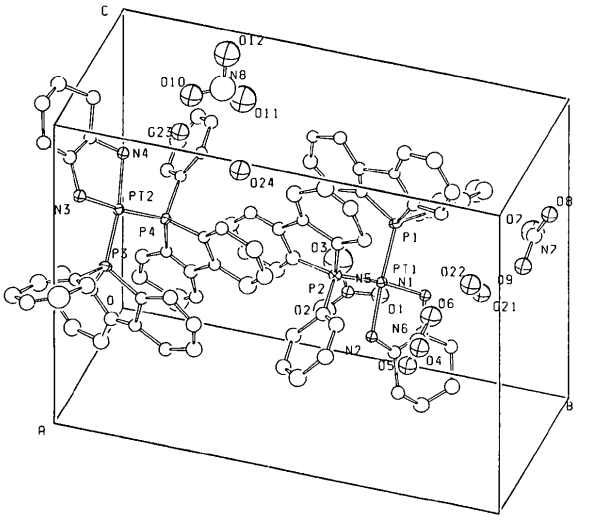

Fig. 1. View of the title compound. Thermal ellipsoids are scaled to enclose 30% probability for non-H atoms.

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Table 1. *Positional parameters* ($\times 10^3$; $\times 10^5$ *for* Pt; $\times 10^4$ *for* P) *and thermal parameters* ($Å^2$)

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pt(1)	17257	60713	33682	2·42 (2)
Pt(2)	68795 (3)	9710 (2)	62719 (3)	2·43 (2)
P(1)	1708 (5)	6385 (4)	5418 (5)	2·8 (1)
P(2)	3047 (5)	5241 (3)	3865 (5)	2·7 (1)
P(3)	6918 (6)	681 (4)	4292 (6)	2·9 (1)
P(4)	5673 (5)	1839 (3)	5744 (5)	2·9 (1)
O(1)	−196 (1)	547 (1)	133 (1)	5·6 (2)
O(2)	−215 (2)	420 (1)	36 (2)	9·6 (4)
O(3)	−130 (3)	463 (2)	247 (3)	16·1 (8)
O(4)	573 (1)	755 (1)	317 (2)	6·9 (3)
O(5)	381 (1)	698 (1)	165 (1)	6·8 (3)
O(6)	403 (2)	754 (1)	355 (2)	8·6 (4)
O(7)	273 (2)	962 (1)	651 (2)	10·2 (6)
O(8)	447 (1)	1027 (1)	798 (1)	5·1 (2)
O(9)	432 (1)	965 (1)	601 (1)	5·6 (2)
O(10)	41 (2)	159 (1)	774 (2)	9·5 (4)
O(11)	−16 (2)	266 (2)	771 (3)	13·6 (7)
O(12)	73 (2)	243 (1)	951 (2)	12·9 (6)
O(21)	227 (1)	839 (1)	393 (1)	6·5 (3)
O(22)	614 (2)	875 (1)	582 (2)	8·0 (4)
O(23)	856 (1)	262 (1)	1000 (1)	6·0 (3)
O(24)	1017 (1)	421 (1)	982 (2)	7·9 (4)
N(1)	63 (1)	687 (1)	274 (1)	2·6 (2)
N(2)	157 (1)	583 (1)	140 (1)	2·5 (1)
N(3)	826 (1)	31 (1)	705 (1)	3·0 (2)
N(4)	681 (1)	106 (1)	814 (1)	2·8 (2)
N(5)	−176 (1)	478 (1)	131 (1)	2·9 (1)
N(6)	452 (1)	733 (1)	284 (1)	5·4 (3)
N(7)	382 (1)	982 (1)	687 (1)	4·5 (2)
N(8)	36 (3)	229 (2)	816 (4)	13 (1)
C(1)	318 (1)	702 (1)	681 (1)	2·8 (2)
C(2)	384 (2)	773 (1)	699 (2)	3·5 (3)
C(3)	475 (2)	812 (1)	816 (2)	4·8 (3)
C(4)	505 (2)	777 (1)	923 (2)	5·2 (4)
C(5)	444 (1)	701 (1)	900 (2)	4·3 (3)
C(6)	344 (1)	663 (1)	783 (1)	2·3 (2)
C(7)	170 (1)	562 (1)	624 (1)	2·7 (2)
C(8)	75 (1)	487 (1)	570 (1)	2·9 (2)
C(9)	81 (2)	433 (1)	651 (2)	4·2 (3)
C(10)	180 (1)	454 (1)	779 (1)	2·9 (2)
C(11)	273 (1)	530 (1)	833 (1)	3·3 (2)
C(12)	242 (1)	568 (1)	731 (1)	3·3 (3)
C(13)	67 (1)	698 (1)	557 (1)	2·8 (2)
C(14)	91 (2)	776 (1)	629 (2)	4·6 (3)
C(15)	−26 (2)	805 (1)	625 (2)	5·8 (4)
C(16)	−118 (2)	774 (2)	579 (3)	9·6 (6)
C(17)	−160 (2)	686 (1)	501 (2)	5·4 (5)
C(18)	−66 (1)	653 (1)	487 (2)	4·4 (3)
C(19)	432 (1)	550 (1)	549 (1)	3·2 (3)
C(20)	523 (1)	624 (1)	619 (1)	3·8 (2)
C(21)	607 (1)	605 (1)	752 (2)	4·6 (3)
C(22)	613 (2)	550 (1)	803 (2)	5·6 (5)
C(23)	498 (1)	462 (1)	713 (2)	4·9 (3)
C(24)	411 (1)	468 (1)	594 (1)	2·7 (2)
C(25)	228 (2)	418 (1)	383 (2)	3·9 (4)
C(26)	128 (2)	365 (1)	288 (2)	4·9 (4)
C(27)	92 (1)	289 (1)	311 (2)	4·2 (3)
C(28)	163 (1)	270 (1)	425 (1)	3·9 (2)
C(29)	273 (1)	329 (1)	523 (1)	3·2 (2)
C(30)	301 (1)	398 (1)	501 (1)	3·3 (2)
C(31)	386 (2)	510 (1)	282 (2)	3·9 (4)
C(32)	307 (1)	431 (1)	156 (1)	4·3 (3)
C(33)	368 (2)	418 (1)	69 (2)	4·4 (3)
C(34)	481 (2)	467 (1)	92 (2)	5·8 (4)
C(35)	540 (2)	539 (1)	207 (2)	5·0 (3)
C(36)	484 (2)	554 (1)	298 (2)	4·6 (3)
C(37)	24 (1)	673 (1)	124 (1)	3·1 (2)
C(38)	57 (1)	618 (1)	60 (1)	3·1 (1)
C(39)	68 (1)	628 (1)	−69 (1)	3·3 (2)
C(40)	−37 (1)	664 (1)	−141 (1)	4·7 (2)
C(41)	−59 (2)	729 (1)	−68 (2)	4·5 (3)
C(42)	−55 (1)	727 (1)	67 (1)	3·5 (2)
C(43)	562 (1)	15 (1)	288 (2)	3·8 (3)
C(44)	478 (2)	−64 (2)	278 (3)	6·3 (6)
C(45)	371 (2)	−98 (1)	145 (2)	5·9 (5)
C(46)	348 (2)	−61 (1)	50 (2)	5·8 (4)
C(47)	437 (2)	18 (1)	63 (2)	4·9 (4)
C(48)	543 (1)	58 (1)	191 (2)	3·9 (3)
C(49)	721 (1)	155 (1)	360 (1)	2·9 (2)
C(50)	806 (2)	233 (1)	418 (2)	4·3 (3)
C(51)	797 (2)	283 (1)	339 (2)	4·4 (3)
C(52)	716 (1)	271 (1)	221 (2)	4·8 (3)
C(53)	625 (1)	194 (1)	162 (1)	3·5 (2)
C(54)	606 (1)	122 (1)	213 (1)	3·0 (2)
C(55)	825 (1)	21 (1)	423 (1)	3·1 (2)
C(56)	937 (2)	65 (1)	460 (2)	5·2 (4)
C(57)	1015 (3)	13 (2)	435 (3)	9·6 (8)
C(58)	1004 (2)	−61 (1)	391 (2)	4·8 (4)
C(59)	864 (2)	−115 (1)	344 (2)	5·2 (3)
C(60)	790 (2)	−67 (1)	372 (2)	5·7 (4)
C(61)	446 (1)	168 (1)	413 (1)	2·7 (2)
C(62)	352 (1)	100 (1)	340 (1)	4·1 (3)
C(63)	255 (1)	78 (1)	227 (1)	4·5 (3)
C(64)	273 (3)	166 (2)	183 (3)	9·6 (7)
C(65)	350 (1)	219 (1)	240 (1)	3·5 (2)
C(66)	437 (1)	221 (1)	359 (1)	3·6 (3)
C(67)	625 (1)	280 (1)	564 (1)	2·5 (2)
C(68)	736 (1)	337 (1)	653 (1)	3·2 (2)
C(69)	761 (2)	414 (1)	617 (2)	5·6 (4)
C(70)	676 (2)	424 (1)	497 (2)	5·6 (4)
C(71)	578 (2)	370 (1)	412 (2)	5·9 (5)
C(72)	546 (1)	292 (1)	446 (1)	2·8 (2)
C(73)	492 (1)	207 (1)	693 (1)	2·4 (2)
C(74)	362 (1)	156 (1)	647 (1)	3·7 (3)
C(75)	296 (2)	170 (1)	724 (2)	5·2 (3)
C(76)	357 (2)	224 (1)	845 (2)	4·6 (3)
C(77)	472 (1)	271 (1)	886 (1)	3·6 (2)
C(78)	535 (1)	259 (1)	803 (1)	2·7 (2)
C(79)	810 (1)	6 (1)	820 (1)	3·4 (2)
C(80)	731 (1)	39 (1)	863 (1)	2·9 (1)
C(81)	752 (1)	50 (1)	1010 (1)	3·7 (2)
C(82)	808 (1)	−20 (1)	1057 (1)	4·6 (2)
C(83)	905 (2)	−41 (1)	1023 (2)	4·5 (3)
C(84)	891 (1)	−41 (1)	886 (1)	3·7 (2)

Table 2. *Dihedral angles* (°) *between mean least-squares planes*

Plane (I) is formed by P(1), P(2), N(1) and N(2); (II) by P(1) and C from C(1) to C(12); (III) P(2) and C(19) to C(30); (IV) P(3), P(4), N(3) and N(4); (V) P(3) and C(43) to C(54); (VI) P(4) and C(61) to C(72); (VII) C(1) to C(6); (VIII) C(7) to C(12); (IX) C(19) to C(24); (X) C(25) to C(30); (XI) C(43) to C(48); (XII) C(49) to C(54); (XIII) C(61) to C(66); (XIV) C(67) to C(72).

	(I)	(II)	(III)	(IV)	(V)
(II)	79·58				
(III)	77·44	4·94			
(IV)	2·85	81·26	79·30		
(V)	79·87	0·87	5·81	81·53	
(VI)	78·65	5·15	1·35	80·53	6·01

(VII)–(VIII) 4·68; (IX)–(X) 1·84; (XI)–(XII) 8·28; (XIII)–(XIV) 3·99

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