C(1)—B(5)	1.552 (15)	C(1)—B(6)	1.711 (16)
C(1)—C(7)	1.516 (14)	C(2)—B(3)	1.571 (15)
C(2)—B(6)	1.715 (15)	B(3)—B(4)	1.673 (18)
B(3)—B(6)	1.763 (18)	B(4)—B(5)	1.683 (20)
B(4)—B(6)	1.731 (18)	B(5)—B(6)	1.782 (17)
N—C(21)	1.485 (10)	N—C(22)	1.468 (12)
N—C(23)	1.469 (12)		
Cnt(1)—Pb—N	122.1	Pb—N—C(21)	109.6 (5)
Pb—N—C(22)	113.1 (5)	Pb—N—C(23)	105.1 (5)
C(2)—C(1)—B(5)	113.1 (9)	C(2)—C(1)—B(6)	64.8 (6)
B(5)—C(1)—B(6)	66.0 (7)	Si—C(2)—C(1)	124.7 (7)
Si—C(2)—B(3)	123.2 (7)	C(1)—C(2)—B(3)	111.4 (8)
Si—C(2)—B(6)	132.0 (6)	C(1)—C(2)—B(6)	64.5 (6)
B(3)—C(2)—B(6)	64.7 (7)	C(2)—B(3)—B(4)	105.7 (9)
C(2)—B(3)—B(6)	61.6 (7)	B(4)—B(3)—B(6)	60.5 (7)
B(3)—B(4)—B(5)	104.3 (9)	B(3)—B(4)—B(6)	62.4 (7)
B(5)—B(4)—B(6)	62.9 (8)	C(1)—B(5)—B(4)	105.2 (9)
C(1)—B(5)—B(6)	61.3 (7)	B(4)—B(5)—B(6)	59.9 (7)
C(1)—B(6)—C(2)	50.7 (6)	C(1)—B(6)—B(3)	92.5 (8)
C(2)—B(6)—B(3)	53.7 (6)	C(1)—B(6)—B(4)	96.6 (8)
C(2)—B(6)—B(4)	97.2 (8)	B(3)—B(6)—B(4)	57.2 (7)
C(1)—B(6)—B(5)	52.7 (6)	C(2)—B(6)—B(5)	92.2 (8)
B(3)—B(6)—B(5)	96.8 (8)	B)4)—B(6)—B(5)	57.2 (7)
C(2)—C(1)—C(7)	121.0 (8)	B(5)—C(1)—C(7)	124.7 (9)
B(6)—C(1)—C(7)	128.8 (8)	C(2)—Si—C(8)	109.4 (5)
C(2)—Si—C(9)	112.6 (4)	C(8)—Si—C(9)	110.3 (5)
C(2)—Si—C(10)	109.7 (5)	C(8)—Si—C(10)	107.7 (5)
C(9)—Si—C(10)	107.0 (5)	N—C(21)—C(11)	110.9 (7)
C(21)—N—C(22)	109.9 (7)	C(21)—N—C(23)	110.3 (7)
C(22)—N—C(23)	108.8 (7)		
Fe—C(11)—C(21)—N	175.1 (6)	C(11)—C(21)—N—Pb	40.2 (8)
C(21)—N—Pb—C(2)	118.7 (6)	N—Pb—C(2)—Si	101.9 (7)

*Centroid of ring C(1), C(2), B(3), B(4), B(5).

†Centroid of ring C(11), C(12), C(13), C(14), C(15).

‡Centroid of ring C(16), C(17), C(18), C(19), C(20).

The systematic absences (*h*0*l*, *h* + *l* odd) were consistent with the space group $P2_1/n$. Data were collected with a scan range of 1.20° plus $K\alpha$ separation and a variable scan speed of 5.00–25.00° $\min^{-1}$. Data were corrected for Lorentz, polarization and absorption effects, the latter correction being applied on the basis of the azimuthal scan data of 4 high-κ reflections with an increment of 10°. The structure was solved by heavy-atom methods with *SHELXTL-Plus* (Sheldrick, 1990). A full-matrix least-squares refinement was performed. All non-H atoms were refined anisotropically. The cage H atoms were located on difference Fourier maps while other H atoms were calculated. No H-atom parameters were refined. Diagrams were produced and most calculations performed using *SHELXTL-Plus*.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55709 (25 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1014]

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A Diphenylformamidinate-Bridged Terpyridineplatinum(II) Dimer

James A. Bailey, Vincent M. Miskowski
and Harry B. Gray

Division of Chemistry and Chemical Engineering†
and Beckman Institute, Mail Code 139-74,
California Institute of Technology, *Pasadena*,
California 91125, *USA*

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Abstract

The compound μ-*N*,*N*′-diphenylformamidinato-bis[2,2′,2″-terpyridineplatinum(II)] perchlorate monohydrate (1) is formed by reaction of diphenylformamidine and [Pt(tpy)Cl]Cl (tpy = terpyridine) in the presence of excess base. The two platinum centers are bridged by a single diphenylformamidinato ligand with the remaining coordination geometry completed by the terpyridine group. The platinum–platinum separation is 3.049 (1) Å.

Comment

We are presently investigating the effect of metal–metal separation on the spectroscopic properties of binuclear platinum–terpyridine complexes. The complex $[\{Pt(tpy)\}_2(\mu\text{-dpf})](ClO_4)_3$ (1) (dpf = *N*,*N*′-diphenylformamidinate, tpy = terpyridine) is deep red in color as compared to the orange pyrazolyl-bridged dimer (Bailey & Gray, 1992). The luminescence properties of these complexes are also dependent on the metal–metal interaction. The structural analysis of (1) is described herein.

Fig. 1 shows the cation with atom labels; Fig. 2 shows the contents of the unit cell viewed down the *a*

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† Contribution No. 8646.

axis. Each platinum atom has nearly square-planar geometry, distorted by the restricted bite angle of the tridentate terpyridine ligand, which is commonly observed in terpyridine complexes (Dewan, Lippard & Bauer, 1980; Ratilla, Scott, Moxness & Kostic, 1990; Wong & Lippard, 1977; Jennette, Gill, Sadownick & Lippard, 1976; Bailey & Gray, 1992). This causes the *cis* N—Pt—N angles within the four metallacyclic rings formed by tpy ligation to be less than 90° [between 80.9 (4) and 81.6 (4)°]. The Pt1—N4 and the Pt2—N7 lengths are relatively short [1.934 (9) and 1.941 (9) Å] also as a result of the geometrical constraints imposed by the tpy ligand. The bond lengths and angles in the pyridine and phenyl rings are characteristic of delocalized aromatic systems with lengths ranging from 1.313 (14) to 1.428 (18) Å and angles ranging from 115.8 (13) to 123.5 (10)°.

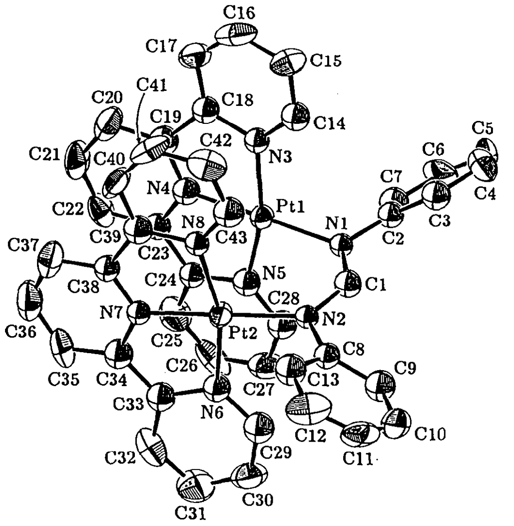

Fig. 1. An *ORTEP* drawing of the cation with 50% probability ellipsoids showing the numbering system. H atoms are not shown.

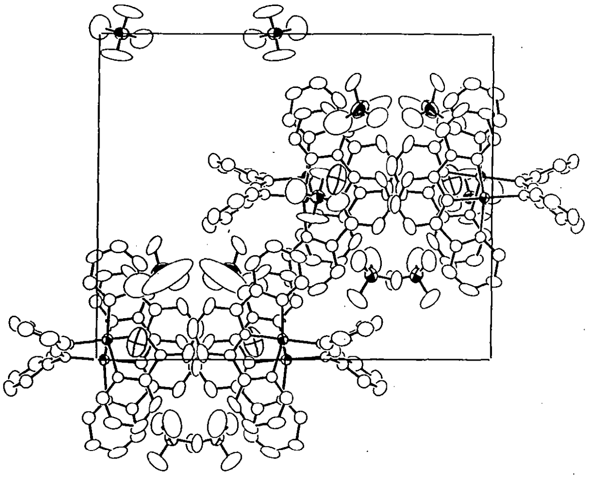

Fig. 2. An *ORTEP* drawing of the contents of one unit cell with a unit cell outlined. Atoms are shown as 50% probability ellipsoids; H atoms are not shown. The view is down the *a* axis, with *b* horizontal. Pt and Cl atoms are shown with shaded octants; the O atom in the solvent water has outlined octants.

The two platinum centers are bridged by a bidentate diphenylformamidinato ligand which imparts a metal–metal separation of 3.049 (1) Å. This distance is near the short extreme observed for binuclear platinum(II) complexes. Diarylformamidinato-type ligands have been observed occupying a bridging site in a number of complexes. Commonly, the metal atoms have a formally metal–metal bonded configuration (*e.g.* d_2^7) in which the *M*—*M* distance can be as short as 2.4336 Å (Piraino, Bruno, Lo Schiavo, Laschi & Zanello, 1987). Binuclear platinum(II) terpyridine complexes exhibit a range of Pt—Pt distances that vary from as short as 2.998 (2) Å in the μ-canaverine structure (Ratilla *et al.*, 1990) to as long as 3.432 (3) Å observed in the μ-pyrazolyl structure (Bailey & Gray, 1992).

We have found* that the colors of these complexes are highly dependent on the Pt—Pt separation. Those complexes with short *M*—*M* separations [*e.g.* μ-canaverine, Pt—Pt = 2.998 (2) Å; μ-diphenylformamidinato, Pt—Pt = 3.049 (1) Å] are deep red while complexes with longer Pt—Pt distances [*e.g.* μ-az, az = anion of 7-azaindole, Pt—Pt = 3.13 (2) Å]† are orange–red; complexes with still longer distances are orange [μ-pyrazolyl, Pt—Pt = 3.432 (3) Å].

Experimental

Crystal data

$[Pt_2(C_{13}H_{11}N_2)(C_{15}H_{11}N_3)_2]$-
$(ClO_4)_3.H_2O$
M_r = 1368.33
Orthorhombic
$Pna2_1$
a = 13.409 (3) Å
b = 20.196 (5) Å
c = 16.517 (4) Å
Z = 4
D_x = 2.03 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 13–14°
μ = 65.61 mm^{-1}
T = 295 K
Irregular parallelepiped
0.27 × 0.23 × 0.21 mm
Deep red

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* Spectroscopic characterization of several binuclear terpyridine–platinum complexes will be reported elsewhere.

† The compound $[\{Pt(tpy)\}_2(\mu\text{-}az)](PF_6)_3$ was crystallized from acetonitrile by slow evaporation as orange–red prisms and X-ray data were collected at room temperature. The space group is $P2_1/n$, a = 10.407 (4), b = 13.601 (10), c = 21.614 (3) Å, β = 103.96 (4)°, Z = 2. Least-squares refinement was eventually terminated because extensive disorder was apparent for many of the light-atom positions. In view of the poor quality of this structure, we do not intend to publish any details; however, the Pt—Pt distance is considered to be reliable within the stated e.s.d. Additional information regarding this structure refinement may be obtained from one of the authors (JAB).

Data collection

CAD-4 diffractometer
ω scans
Absorption correction: empirical [0.885 to 1.169 (relative)]
14036 measured reflections
4176 independent reflections
4175 observed reflections
$R_{\rm int} = 0.008$
$\theta_{\rm max} = 20°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 19$
$l = -15 \rightarrow 15$
4 standard reflections intensity variation: none

Refinement

Refinement on F^2
Final $R = 0.030$
$wR = 0.003$
$S = 2.07$
4175 reflections
487 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F_o^2)$
$(\Delta/\sigma)_{\rm max} = 0.23$ for O12; 0.13 for all others
$\Delta\rho_{\rm max} = 0.74$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.72$ e Å^{-3}
Extinction correction: Larson (1967)
Extinction coefficient: $0.258\ (6) \times 10^{-6}$

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

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

	x	y	z	$U_{\rm eq}$
Pt1	0.14600 (3)	0.02020 (2)	0.0000	0.0329 (1)
N1	0.1814 (6)	−0.0788 (4)	0.0085 (6)	0.00023 (2)*
C1	0.2676 (8)	−0.1024 (4)	0.0263 (7)	0.00031 (2)*
C2	0.1011 (8)	−0.1226 (5)	−0.0141 (7)	0.00030 (2)*
C3	0.1195 (9)	−0.1773 (6)	−0.0653 (7)	0.00036 (3)*
C4	0.0418 (11)	−0.2179 (6)	−0.0871 (7)	0.0605 (39)
C5	−0.0519 (13)	−0.2065 (7)	−0.0592 (10)	0.0726 (49)
C6	−0.0702 (8)	−0.1516 (7)	−0.0120 (9)	0.0578 (39)
C7	0.0070 (9)	−0.1107 (5)	0.0097 (8)	0.00038 (2)*
N2	0.3495 (6)	−0.0694 (4)	0.0493 (5)	0.00023 (2)*
C8	0.4374 (8)	−0.1088 (4)	0.0643 (7)	0.00028 (2)*
C9	0.4322 (9)	−0.1720 (6)	0.0992 (7)	0.00039 (3)*
C10	0.5235 (13)	−0.2067 (6)	0.1113 (9)	0.0636 (44)
C11	0.6128 (11)	−0.1795 (8)	0.0948 (8)	0.0620 (43)
C12	0.6183 (9)	−0.1188 (7)	0.0653 (10)	0.0718 (41)
C13	0.5288 (8)	−0.0822 (5)	0.0516 (7)	0.00041 (3)*
N3	0.1546 (7)	0.0290 (4)	−0.1219 (8)	0.00027 (2)*
C14	0.1714 (10)	−0.0179 (5)	−0.1767 (9)	0.00034 (3)*
C15	0.1724 (10)	−0.0066 (8)	−0.2599 (9)	0.0498 (42)
C16	0.1567 (11)	0.0581 (8)	−0.2839 (9)	0.0645 (52)
C17	0.1368 (10)	0.1079 (7)	−0.2303 (9)	0.0643 (38)
C18	0.1389 (9)	0.0920 (6)	−0.1493 (8)	0.00031 (3)*
N4	0.1179 (6)	0.1138 (4)	−0.0083 (7)	0.00031 (2)*
C19	0.1206 (9)	0.1424 (6)	−0.0820 (8)	0.00037 (3)*
C20	0.1115 (10)	0.2100 (6)	−0.0899 (9)	0.0585 (42)
C21	0.1027 (10)	0.2462 (6)	−0.0215 (10)	0.0600 (43)
C22	0.0946 (9)	0.2164 (5)	0.0542 (10)	0.0496 (35)
C23	0.1018 (7)	0.1480 (5)	0.0614 (8)	0.00031 (2)*
N5	0.1176 (7)	0.0391 (5)	0.1190 (6)	0.00032 (2)*
C24	0.0986 (8)	0.1055 (5)	0.1300 (7)	0.00027 (2)*
C25	0.0742 (11)	0.1275 (7)	0.2068 (10)	0.0592 (40)
C26	0.0688 (10)	0.0824 (8)	0.2684 (10)	0.0621 (44)
C27	0.0886 (11)	0.0168 (7)	0.2574 (10)	0.0586 (45)
C28	0.1113 (9)	−0.0034 (6)	0.1797 (8)	0.00035 (3)
Pt2	0.3592 (3)	0.02920 (2)	0.06320 (3)	0.0333 (1)
N6	0.3441 (8)	0.0326 (5)	0.1844 (8)	0.00032 (3)*
C29	0.3402 (10)	−0.0177 (6)	0.2370 (11)	0.00040 (3)*
C30	0.3319 (11)	−0.0071 (8)	0.3180 (10)	0.0624 (46)
C31	0.3288 (12)	0.0547 (9)	0.3486 (9)	0.0680 (51)
C32	0.3336 (10)	0.1075 (7)	0.2971 (10)	0.0619 (41)
C33	0.3435 (9)	0.0973 (6)	0.2124 (8)	0.00035 (3)*
N7	0.3634 (6)	0.1244 (4)	0.0781 (6)	0.00025 (2)*
C34	0.3504 (9)	0.1483 (6)	0.1542 (7)	0.00037 (3)*
C35	0.3409 (10)	0.2159 (6)	0.1640 (9)	0.0577 (41)
C36	0.3443 (11)	0.2564 (6)	0.0968 (11)	0.0678 (49)
C37	0.3599 (9)	0.2317 (6)	0.0202 (10)	0.0563 (42)
C38	0.3699 (8)	0.1629 (5)	0.0143 (7)	0.00030 (2)*
N8	0.3841 (6)	0.0563 (4)	−0.0534 (6)	0.00025 (2)*
C39	0.3856 (9)	0.1253 (6)	−0.0611 (8)	0.00036 (3)*
C40	0.4032 (10)	0.1509 (6)	−0.1366 (10)	0.0594 (40)
C41	0.4168 (11)	0.1107 (8)	−0.2033 (9)	0.0647 (44)
C42	0.4161 (11)	0.0436 (8)	−0.1930 (10)	0.0585 (44)
C43	0.3993 (9)	0.0185 (6)	−0.1171 (8)	0.00039 (3)*
Cl1	0.2894 (4)	0.3078 (2)	−0.2402 (2)	0.0768 (13)
O1	0.3036 (9)	0.3426 (5)	0.3123 (7)	0.1114 (36)
O2	0.2622 (8)	0.2428 (4)	−0.2561 (6)	0.0957 (39)
O3	0.2149 (11)	0.3393 (5)	−0.1964 (8)	0.1412 (48)
O4	0.3752 (11)	0.3066 (6)	−0.1961 (11)	0.1540 (54)
Cl2	0.1555 (2)	0.9441 (2)	0.5013 (2)	0.0541 (8)
O5	0.0921 (9)	0.9562 (8)	0.4353 (6)	0.1463 (61)
O6	0.2228 (9)	0.8929 (6)	0.4853 (6)	0.1314 (39)
O7	0.1026 (7)	0.9277 (7)	0.5690 (6)	0.1202 (45)
O8	0.2093 (11)	0.9996 (6)	0.5189 (10)	0.1645 (50)
Cl3	0.1297 (4)	0.3395 (3)	0.2707 (3)	0.0940 (14)
O9	0.1204 (10)	0.3569 (5)	0.3520 (7)	0.1289 (51)
O10	0.0658 (9)	0.2949 (6)	0.2440 (7)	0.1244 (41)
O11	0.2157 (13)	0.3222 (16)	0.2552 (16)	0.3830 (139)
O12	0.1177 (25)	0.3945 (11)	0.2294 (12)	0.3791 (149)
O13	0.0837 (8)	0.3966 (5)	0.0549 (10)	0.1412 (45)

*Isotropic displacement parameter, *B*.

Table 2. *Geometric parameters* (Å, °)

Pt1—Pt2	3.049 (1)	N4—C23	1.360 (14)
Pt1—N1	2.058 (8)	C19—C20	1.378 (18)
Pt1—N3	2.024 (10)	C20—C21	1.351 (19)
Pt1—N4	1.934 (9)	C21—C22	1.391 (18)
Pt1—N5	2.039 (10)	C22—C23	1.390 (17)
Pt2—N2	2.009 (8)	C23—C24	1.421 (16)
Pt2—N6	2.014 (11)	N5—C24	1.377 (15)
Pt2—N7	1.941 (9)	N5—C28	1.323 (16)
Pt2—N8	2.030 (9)	C24—C25	1.383 (18)
N1—C1	1.284 (13)	C25—C26	1.37 (2)
N1—C2	1.443 (13)	C26—C27	1.36 (2)
C1—N2	1.339 (13)	C27—C28	1.380 (19)
C2—C3	1.412 (16)	N6—C29	1.336 (18)
C2—C7	1.343 (15)	N6—C33	1.387 (17)
C3—C4	1.375 (17)	C29—C30	1.36 (2)
C4—C5	1.36 (2)	C30—C31	1.35 (2)
C5—C6	1.38 (2)	C31—C32	1.37 (2)
C6—C7	1.371 (17)	C32—C33	1.421 (19)
N2—C8	1.443 (13)	C33—C34	1.411 (17)
C8—C9	1.403 (15)	N7—C34	1.358 (14)
C8—C13	1.355 (15)	N7—C38	1.313 (13)
C9—C10	1.424 (18)	C34—C35	1.379 (18)
C10—C11	1.35 (2)	C35—C36	1.38 (2)
C11—C12	1.32 (2)	C36—C37	1.38 (2)
C12—C13	1.428 (18)	C37—C38	1.401 (17)
N3—C14	1.329 (17)	C38—C39	1.474 (16)
N3—C18	1.365 (16)	N8—C39	1.398 (15)
C14—C15	1.393 (19)	N8—C43	1.316 (15)
C15—C16	1.38 (2)	C39—C40	1.370 (19)
C16—C17	1.37 (2)	C40—C41	1.38 (2)
C17—C18	1.375 (18)	C41—C42	1.36 (2)
C18—C19	1.528 (17)	C42—C43	1.37 (2)
N4—C19	1.346 (15)		
N1—Pt1—N3	98.1 (4)	C43—N8—C39	120.2 (10)
N1—Pt1—N5	99.2 (4)	C40—C39—C38	126.7 (11)
N3—Pt1—N5	162.3 (4)	C41—C40—C39	121.7 (13)
N1—Pt1—N4	177.9 (4)	N2—Pt2—N7	177.8 (3)
N3—Pt1—N4	81.6 (4)	N6—Pt2—N7	80.9 (4)
N4—Pt1—N5	81.3 (4)	N7—Pt2—N8	81.3 (4)
N2—Pt2—N6	98.1 (4)	N2—C1—N1	128.2 (9)
N2—Pt2—N8	99.8 (3)	C7—C2—N1	121.1 (10)
N6—Pt2—N8	161.9 (4)	C4—C3—C2	119.4 (11)
C2—N1—C1	120.2 (9)	C6—C5—C4	119.6 (14)
C3—C2—N1	120.3 (9)	C6—C7—C2	121.7 (11)
C7—C2—C3	118.6 (10)	C9—C8—N2	122.1 (9)
C5—C4—C3	120.7 (12)	C13—C8—C9	118.0 (10)
C7—C6—C5	119.8 (12)	C11—C10—C9	122.3 (13)
C8—N2—C1	116.5 (8)	C13—C12—C11	119.4 (13)

C13—C8—N2	119.6 (9)	C18—N3—C14	117.6 (11)
C10—C9—C8	117.6 (11)	C16—C15—C14	115.8 (13)
C12—C11—C10	120.3 (14)	C18—C17—C16	116.9 (13)
C12—C13—C8	122.1 (11)	C19—C18—N3	113.8 (10)
C15—C14—N3	123.8 (12)	C23—N4—C19	123.5 (10)
C17—C16—C15	122.8 (14)	C42—C41—C40	118.9 (14)
C17—C18—N3	122.9 (11)	C43—C42—C41	118.8 (14)
C19—C18—C17	123.3 (11)	C42—C43—N8	122.9 (12)
N4—C19—C18	112.1 (10)	C22—C21—C20	121.6 (12)
C20—C19—C18	127.3 (11)	C22—C23—N4	116.3 (10)
C20—C19—N4	120.5 (11)	C24—C23—C22	131.8 (11)
C21—C20—C19	117.8 (12)	N5—C24—C23	118.5 (10)
C23—C22—C21	120.1 (12)	C25—C24—N5	118.5 (11)
C24—C23—N4	111.9 (10)	C27—C26—C25	122.5 (14)
C28—N5—C24	121.4 (10)	C27—C28—N5	121.8 (12)
C25—C24—C23	123.0 (11)	C30—C29—N6	121.6 (13)
C26—C25—C24	118.8 (13)	C32—C31—C30	119.2 (15)
C28—C27—C26	117.0 (13)	C32—C33—N6	117.7 (11)
C33—N6—C29	119.9 (11)	C34—C33—C32	124.8 (12)
C31—C30—C29	121.2 (15)	N7—C34—C33	112.3 (10)
C33—C32—C31	120.3 (13)	C35—C34—N7	118.2 (11)
C34—C33—N6	117.5 (11)	C37—C36—C35	122.0 (13)
C38—N7—C34	122.8 (9)	C37—C38—N7	121.7 (10)
C35—C34—C33	129.5 (11)	C39—C38—C37	125.8 (10)
C36—C35—C34	119.3 (12)	N8—C39—C38	115.7 (10)
C38—C37—C36	116.0 (12)	C40—C39—N8	117.5 (11)
C39—C38—N7	112.6 (9)		

An empirical absorption correction based on ψ scans of six reflections was used. The goodness of fit for merging in point group *mm*2 was 1.35 (two data sets were collected within the stated octants; $R_{\rm merge}$ for 165 reflections with exactly two observations, 0.008). Attempted merging of the data in point group *mmm* gave a goodness of fit of only 2.0, and the molecular symmetry could not be satisfied by the centrosymmetric equivalent of the space group (*Pnma*). All reflections were used in solution and refinement of the structure. Coordinates of the Pt atoms were found from a Patterson map, the remaining heavy atoms (including a water molecule of crystallization and three perchlorate ions) were found by successive structure factor–Fourier calculations. F_o^2 magnitudes were used in full-matrix least-squares refinement, which minimized $\Sigma w(F_o^2-F_c^2)$; R based on F; wR based on F^2; H atoms were positioned by calculation (C—H, 0.95 Å) and included as constant contributions to the structure factors (except H atoms on the water molecule, which were ignored). Hydrogen parameters were not refined but repositioned twice near the end of the refinement. The weights were taken as $1/\sigma^2(F_o^2)$; variances $[\sigma^2(F_o^2)]$ derived from counting statistics plus an additional term, $(0.014I)^2$; variances of the merged data by propagation of error plus an additional term $(0.014\bar{I})^2$. Atomic scattering factors and values for $\Delta f'$ taken from Cromer & Waber (1974) and Cromer (1974); programs used were those from the *CRYM* crystallographic computing system (Duchamp, 1964) and *ORTEP* (Johnson, 1976). Final atomic coordinates and $U_{\rm eq}$ values are listed in Table 1 with selected distances and angles given in Table 2. The data were averaged in point group *mm*2 for final refinement. Refinement with f'' positive gave significantly poorer R indices and goodness of fit than with f'' negative. A background correction derived from the backgrounds of weak reflections as a function of 2θ was applied to all reflections.

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55628 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SP1005]

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Structure of [1,9-Bis(2-pyridyl)-2,5,8-triazanonane]zinc(II) Tetrachlorozincate(II)

MARTHA E. SOSA-TORRES* AND
VÍCTOR M. UGALDE-SALDÍVAR

División de Estudios de Posgrado, Facultad de Química, Universidad Nacional Autónoma de México, Ciudad Universitaria, Coyoacán, DF 04510, Mexico

MARÍA J. ROSALES-HOZ* AND R. A. TOSCANO

Instituto de Química, Universidad Nacional Autónoma de México, Circuito Exterior, Ciudad Universitaria, Coyoacán, DF 04510, Mexico

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Abstract

The cationic complex is five coordinated and shows a distorted geometry intermediate between a square-based pyramid and a trigonal bipyramid. Not all the Zn—N distances are equivalent. The $ZnCl_4^{2-}$ anion shows a tetrahedral geometry.

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