

To the best of our knowledge, such a structure is unique in rhodium(I) chemistry. We have previously reported the structure of a mononuclear rhodium(I) phenoxide complex, *trans*-Rh(PPh<sub>3</sub>)<sub>2</sub>(CO)(OC<sub>6</sub>H<sub>5</sub>) (Bernard, Churchill, Janik & Atwood, 1990) in which Rh—OC<sub>6</sub>H<sub>5</sub> = 2.044 (2) Å. However, the overall core structure of (I) is perhaps most similar to that of Rh<sub>3</sub>(μ-Cl)<sub>3</sub>[μ-H<sub>3</sub>CN(PF<sub>2</sub>)<sub>2</sub>]<sub>3</sub> (Mague & Johnson, 1990; Mague, Johnson & Lloyd, 1989) in which three non-bonded rhodium(I) centers are held in a triangular arrangement at distances of 3.0755 (4)–3.1180 (4) Å (with molecular C<sub>3v</sub> symmetry) *via* sets of bridging chloro and bis(difluorophosphino)-methylamine ligands. It should be noted that the non-bonding Rh...Rh distances of 2.888 (2)–2.995 (2) Å in the present complex are not dramatically longer than the Rh—Rh bond lengths of

2.701 (8)–2.796 (8) Å found in Rh<sub>4</sub>(CO)<sub>9</sub>(μ-CO)<sub>3</sub> (Wei, 1969).

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## Structure of Triaqua[1,2-benzenedicarboxylato(2-)](2,2'-bipyridine)nickel(II) Monohydrate

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**Abstract.** [Ni(C<sub>8</sub>H<sub>4</sub>O<sub>4</sub>)(C<sub>10</sub>H<sub>8</sub>N<sub>2</sub>)(H<sub>2</sub>O)<sub>3</sub>].H<sub>2</sub>O, *M<sub>r</sub>* = 451.06, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 24.487 (13), *b* = 22.043 (8), *c* = 7.510 (2) Å, *V* = 4054 (3) Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.478 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 10.0 cm<sup>-1</sup>, *F*(000) = 1872, room temperature, *R* = 0.058 for 3987 independent reflections having *I* > 2σ(*I*). In the asymmetric part of the unit cell there are two discrete units nearly related by a center of symmetry. Both Ni atoms are in a distorted octahedral environment. The 1,2-benzenetetracarboxylate(2-) anions act as monodentate ligands.

**Experimental.** Blue, needle-like (0.18 × 0.09 × 0.08 mm) crystals suitable for X-ray determination were obtained from dilute aqueous solutions containing equimolar amounts of Ni(NO<sub>3</sub>)<sub>2</sub>, 2,2'-bipyridine and sodium phthalate. 18 reflections with 12.4 < θ <

19.6° were used for measuring lattice parameters. Data were collected on a Philips PW 1100 four-circle diffractometer with graphite-monochromated Mo *K*α radiation, ω/2θ scan, intensity measurement to 2θ<sub>max</sub> = 42° (0 ≤ *h* ≤ 34, 0 ≤ *k* ≤ 30, 0 ≤ *l* ≤ 10). Two standard reflections, no significant intensity variation, 4888 reflections were measured, 3987 observed [*I* > 2σ(*I*)] reflections were used for refinements. Data were corrected for Lorentz and polarization, not for absorption.

The positions of the Ni atoms were found by the Patterson method and the rest of the non-H atoms were located by alternate cycles of difference Fourier maps and isotropic least-squares refinement (on *F*). After anisotropic refinement was carried out in two blocks for non-H atoms, positions of H atoms were calculated and included in the model at fixed posi-

Table 1. Positional ( $\times 10^4$ , for Ni  $\times 10^5$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ ) with e.s.d.'s in parentheses

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	$U_{eq}$
Ni(1)	21370 (3)	21880 (4)	-1484 (10)	26.4 (2)
O(W11)	2096 (2)	2037 (2)	2570 (6)	39 (1)
O(W12)	2187 (2)	2298 (3)	-2909 (6)	36 (1)
O(W13)	1476 (2)	2794 (2)	-6 (7)	36 (1)
O(W14)	1734 (3)	889 (3)	3310 (8)	53 (2)
O(11)	1554 (2)	1531 (2)	-456 (7)	34 (1)
O(12)	1681 (3)	1237 (3)	-3254 (8)	51 (2)
O(13)	672 (3)	1366 (3)	2591 (8)	49 (2)
O(14)	518 (2)	2085 (2)	531 (7)	37 (1)
N(11)	2756 (2)	2808 (3)	201 (8)	32 (1)
N(12)	2821 (2)	1626 (3)	-322 (8)	31 (1)
C(11)	2687 (4)	3412 (4)	497 (13)	48 (2)
C(12)	3119 (4)	3799 (4)	835 (16)	62 (3)
C(13)	3643 (4)	3548 (5)	907 (14)	61 (3)
C(14)	3710 (4)	2948 (4)	580 (13)	53 (2)
C(15)	3259 (3)	2577 (3)	233 (11)	34 (2)
C(16)	3304 (3)	1934 (4)	-169 (11)	35 (2)
C(17)	3795 (3)	1626 (5)	-405 (14)	56 (3)
C(18)	3780 (4)	998 (5)	-771 (15)	61 (3)
C(19)	3296 (4)	719 (4)	-820 (14)	58 (3)
C(110)	2810 (4)	1030 (4)	-607 (12)	45 (2)
C(111)	1400 (4)	1264 (4)	-1868 (13)	31 (2)
C(112)	868 (4)	949 (4)	-1780 (14)	34 (2)
C(113)	717 (5)	532 (4)	-3103 (14)	47 (2)
C(114)	234 (5)	201 (5)	-3009 (15)	53 (3)
C(115)	-115 (5)	302 (4)	-1588 (15)	57 (3)
C(116)	25 (4)	717 (5)	-262 (15)	56 (2)
C(117)	500 (4)	1058 (4)	-374 (14)	33 (2)
C(118)	592 (4)	1540 (4)	1033 (13)	38 (2)
Ni(2)	-349 (4)	27682 (4)	53402 (10)	27.6 (2)
O(W21)	-13 (3)	2905 (3)	2597 (7)	41 (1)
O(W22)	-95 (2)	2670 (3)	8102 (7)	39 (1)
O(W23)	637 (2)	2201 (2)	5200 (8)	37 (1)
O(W24)	282 (4)	4074 (4)	1861 (10)	81 (3)
O(21)	531 (2)	3458 (3)	5619 (7)	32 (1)
O(22)	386 (2)	3762 (3)	8392 (7)	42 (1)
O(23)	1591 (2)	2895 (2)	4630 (7)	35 (1)
O(24)	1448 (3)	3645 (3)	2690 (7)	41 (1)
N(21)	-746 (2)	3276 (3)	5364 (9)	38 (1)
N(22)	-620 (2)	2101 (3)	4971 (10)	38 (1)
C(21)	-783 (3)	3878 (4)	5673 (13)	46 (2)
C(22)	-1266 (4)	4184 (5)	5753 (17)	62 (3)
C(23)	-1740 (4)	3855 (5)	5549 (16)	63 (3)
C(24)	-1714 (3)	3232 (4)	5275 (14)	49 (2)
C(25)	-1203 (3)	2971 (4)	5135 (10)	36 (1)
C(26)	-1124 (3)	2296 (4)	4806 (10)	37 (2)
C(27)	-1549 (4)	1919 (5)	4283 (14)	53 (2)
C(28)	-1431 (4)	1318 (5)	4004 (16)	57 (3)
C(29)	-930 (4)	1108 (4)	4274 (16)	59 (3)
C(210)	-516 (4)	1512 (4)	4754 (14)	50 (2)
C(211)	667 (4)	3732 (4)	7033 (13)	30 (2)
C(212)	1221 (4)	4032 (4)	7034 (14)	31 (1)
C(213)	1338 (4)	4437 (5)	8408 (14)	37 (2)
C(214)	1844 (4)	4719 (5)	8459 (15)	44 (2)
C(215)	2232 (4)	4602 (4)	7151 (16)	46 (2)
C(216)	2112 (4)	4192 (4)	5802 (15)	41 (2)
C(217)	1604 (4)	3910 (4)	5718 (14)	31 (1)
C(218)	1539 (3)	3455 (4)	4217 (13)	29 (2)

Table 2. Selected bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

(a) In the coordination polyhedra			
Ni(1)—O(W11)	2.071 (5)	Ni(2)—O(W21)	2.083 (5)
Ni(1)—O(W12)	2.091 (5)	Ni(2)—O(W22)	2.091 (5)
Ni(1)—O(W13)	2.101 (5)	Ni(2)—O(W23)	2.069 (5)
Ni(1)—O(11)	2.047 (5)	Ni(2)—O(21)	2.068 (6)
Ni(1)—N(11)	2.058 (6)	Ni(2)—N(21)	2.070 (6)
Ni(1)—N(12)	2.087 (6)	Ni(2)—N(22)	2.072 (6)
O(W11)—Ni(1)—O(W12)	177.4 (2)	O(W21)—Ni(2)—O(W22)	176.5 (2)
O(W11)—Ni(1)—O(W13)	90.8 (2)	O(W21)—Ni(2)—O(W23)	91.0 (2)
O(W11)—Ni(1)—O(11)	87.9 (2)	O(W21)—Ni(2)—O(21)	88.7 (2)
O(W11)—Ni(1)—N(11)	91.0 (2)	O(W21)—Ni(2)—N(21)	87.2 (2)
O(W11)—Ni(1)—N(12)	90.3 (2)	O(W21)—Ni(2)—N(22)	89.3 (3)
O(W12)—Ni(1)—O(W13)	91.3 (2)	O(W22)—Ni(2)—O(W23)	92.5 (2)
O(W12)—Ni(1)—O(11)	90.6 (2)	O(W22)—Ni(2)—O(21)	91.3 (2)
O(W12)—Ni(1)—N(11)	90.4 (2)	O(W22)—Ni(2)—N(21)	89.3 (2)
O(W12)—Ni(1)—N(12)	87.7 (2)	O(W22)—Ni(2)—N(22)	90.6 (2)
O(W13)—Ni(1)—O(11)	85.3 (2)	O(W23)—Ni(2)—O(21)	85.2 (2)
O(W13)—Ni(1)—N(11)	98.0 (2)	O(W23)—Ni(2)—N(21)	174.9 (2)
O(W13)—Ni(1)—N(12)	176.9 (2)	O(W23)—Ni(2)—N(22)	96.6 (2)
O(11)—Ni(1)—N(11)	176.6 (2)	O(21)—Ni(2)—N(21)	99.5 (2)
O(11)—Ni(1)—N(12)	97.6 (2)	O(21)—Ni(2)—N(22)	177.3 (2)
N(11)—Ni(1)—N(12)	79.1 (2)	N(21)—Ni(2)—N(22)	78.7 (2)
(b) In the carboxylate groups			
O(11)—C(111)	1.270 (11)	O(21)—C(211)	1.266 (11)
O(12)—C(111)	1.249 (12)	O(22)—C(211)	1.233 (11)
O(13)—C(118)	1.247 (11)	O(23)—C(218)	1.279 (10)
O(14)—C(118)	1.272 (10)	O(24)—C(218)	1.241 (11)

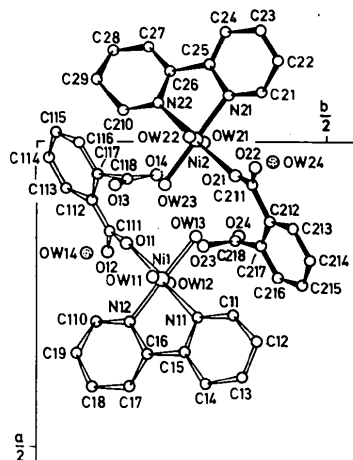


Fig. 1. Asymmetric part of the unit cell viewed along the *c* axis. H atoms are omitted for clarity.

The final positional parameters and equivalent isotropic temperature factors are given in Table 1. Selected interatomic bond distances and angles are given in Table 2.\* A view of the structure along the *c* axis is shown in Fig. 1.

**Related literature.** The structures of two similar complexes, [Ni(C<sub>8</sub>H<sub>4</sub>O<sub>4</sub>)(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)(H<sub>2</sub>O)<sub>3</sub>].H<sub>2</sub>O and [Ni(C<sub>8</sub>H<sub>4</sub>O<sub>4</sub>)(C<sub>10</sub>H<sub>9</sub>N<sub>3</sub>)(H<sub>2</sub>O)<sub>3</sub>].2H<sub>2</sub>O, have recently

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, possible hydrogen bonding and least-squares-planes data, and a full list of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53169 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

tions with fixed isotropic thermal parameters equal to those of the C atoms to which they are attached.

Final difference map showed  $\Delta\rho_{\min} = -0.070$  and  $\Delta\rho_{\max} = 1.830 \text{ e \AA}^{-3}$ . Final *R* factor was 0.058,  $wR = 0.063$ ,  $w = 0.37/[\sigma^2(F) + 0.0095F^2]$  for 523 parameters,  $(\Delta/\sigma)_{\max} = 0.03$ . All calculations were performed on an IBM PC AT compatible computer using *CRYSTAN* (Burzlaff, Böhme & Gomm, 1977), *SHELX76* (Sheldrick, 1976) and *CSU* for geometry calculation (Vicković, 1986). Scattering factors were from Cromer & Mann (1968),  $f'$  and  $f''$  values from Cromer & Liberman (1970).

been reported (Poleti, Stojaković, Prelesnik & Manojlović-Muir, 1990).

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## Structure of 2-(4-Dimethylaminobenzylidene)cyclohexanone

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**Abstract.**  $C_{15}H_{19}NO$ ,  $M_r = 229.32$ , monoclinic,  $P2_1/c$ ,  $a = 7.179$  (4),  $b = 11.002$  (2),  $c = 16.110$  (3) Å,  $\beta = 95.15$  (4)°,  $V = 1267.25$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.201$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.5418$  Å,  $\mu = 0.59$  mm<sup>-1</sup>,  $F(000) = 496$ ,  $T = 287$  K,  $R = 0.044$  ( $wR = 0.045$ ) for 1921 observed reflections. The cyclohexanone ring exhibits a chair conformation, flattened at the ketone end. The nonbonded steric strain between H atoms on the cyclohexanone and phenyl rings causes increases in the bond angles at the C atoms joining the rings, and rotation of the phenyl group about the C—Ph bond, at the expense of the conjugation energy of the system. The dimethylamino substituent, being an electron-donating group, makes the internal aromatic angle smaller than 120°.

**Experimental.** The synthesis of the title compound has been reported previously (Smith, Dimmock & Turner, 1973), m.p. 400–401 K. Orange-plate crystal, recrystallized from ethanol, dimensions 0.05 × 0.25 × 0.33 mm. The cell parameters were obtained by least squares using 25 reflections with  $14.93 < \theta < 22.03^\circ$ . Enraf–Nonius CAD-4 diffractometer used for data collection, 2601 unique reflections,  $-8 \leq h \leq 8$ ,  $0 \leq k \leq 13$ ,  $0 \leq l \leq 20$ ,  $[(\sin \theta)/\lambda]_{\max} = 0.62653$  Å<sup>-1</sup>. Three monitoring reflections, intensity constant to within 1.4%. No absorption correction. Merging  $R$  based on intensities of 0.0050 for 223 replicate reflections. Structure solved by direct methods using *XTAL2.6* (Hall & Stewart, 1989), all

non-H atoms found on  $E$  map and refined anisotropically; H atoms found on  $\Delta F$  map and refined isotropically. 1921 reflections with  $I > 2\sigma(I)$  used in refinement.  $R = 0.044$ ,  $wR = 0.045$  [ $w = 1/\sigma^2(F)$ ],  $S = 2.34$  for 1921 observed reflections. 231 parameters refined and  $F$  magnitudes used in the refinement. Extinction correction included and secondary-extinction coefficient  $g = 2.8$  (6) × 10<sup>-5</sup>. Final  $(\Delta/\sigma)_{\text{av}} = 0.026$ ,  $(\Delta/\sigma)_{\text{max}} = 0.36$ . Maximum and minimum  $\Delta\rho$  in final difference map of +0.18 and -0.15 e Å<sup>-3</sup>, respectively. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV). All calculations performed on a VAX6330 computer at the University of Saskatchewan.

The atomic parameters are summarized in Table 1.† Bond distances and angles are listed in Table 2. Fig. 1 is an *ORTEP* drawing (Johnson, 1976) of the title compound and Fig. 2 shows the packing in the unit cell.

**Related literature.** Other structures in this series of cyclic conjugated benzylidene ketones, which have various cytotoxicities towards P388 leukemia cells (Dimmock, Arora, Wonko, Hamon, Quail, Jia, Warrington, Fang & Lee, 1989; Dimmock, Arora, Jia,

† Lists of structure amplitudes, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53057 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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