Both $[Cu(bcen)(NO_3)(H_2O)]NO_3.H_2O$ (Lee, Lu, Liu, Chung & Lee, 1984) and the title compound contain 6,5,6-membered chelate rings. Comparing the crystal structures of these two complexes, we find the following results: (1) for each of these two complexes, the two asymmetric nitrogens are of the same *R* or *S* configuration, and the central five-membered chelate ring adopts a stable *gauche* conformation; (2) the two terminal six-membered chelate rings of each of these two complexes are in stable chair forms; (3) the coordination numbers for these two complexes are different; $[Cu(bcen)(NO_3)(H_2O)]NO_3.H_2O$ is of the 4+2 type but $[Cu(L\text{-}2,Cy,2)(H_2O)](ClO_4)_2$ is five-coordinate. This difference may be due to the steric effects of the cyclohexane substituent on the central chelate ring.

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References

Hong, C.-Y., Lee, T.-Y., Lee, T.-J., Chao, M.-S. & Chung, C.-S. (1987). *Acta Cryst.* C**43**, 34–37.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Lee, T.-J., Hong, C.-Y., Liu, S.-H., Chung, C.-S. & Lee, T.-Y. (1984). *Acta Cryst.* C**40**, 1673–1675.

Lee, T.-J., Lu, T.-H., Liu, S.-H., Chung, C.-S. & Lee, T.-Y. (1984). *Acta Cryst.* C**40**, 1131–1135.

Lu, T.-H., Shan, H.-C., Chao, M.-S. & Chung, C.-S. (1987). *Acta Cryst.* C**43**, 1524–1527.

Sheldrick, G. M. (1986). *SHELXTL PLUS User Manual.* Nicolet XRD Corporation, Madison, Wisconsin, USA.

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Structure of μ-Acetato-di-μ-carbonyl-bis[(2,2′-bipyridyl)carbonylruthenium(I)](*Ru–Ru*) Tetraphenylborate

By Margot M. de V. Steyn

Department of Chemistry, University of South Africa, PO Box 392, *Pretoria* 0001, *Republic of South Africa*

and Eric Singleton

National Chemical Research Laboratory, Council for Scientific and Industrial Research, PO Box 395,
Pretoria 0001, *Republic of South Africa*

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Abstract. $[Ru_2(\mu\text{-}O_2CCH_3)(CO)_2(\mu\text{-}CO)_2(bipy)_2]BPh_4$ (bipy = 2,2′-bipyridine), $[Ru_2(C_{10}H_8N_2)_2(C_2H_3O_2)(CO)_4][B(C_6H_5)_4]$, $M_r = 1004{\cdot}84$, monoclinic, $P2_1/c$, $a = 15{\cdot}900\,(3)$, $b = 17{\cdot}731\,(4)$, $c = 15{\cdot}739\,(7)$ Å, $\beta = 104{\cdot}16\,(3)°$, $V = 4302{\cdot}4$ Å^3, $Z = 4$, $D_x = 1{\cdot}55$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}7107$ Å, $\mu = 0{\cdot}668$ mm^{-1}, $F(000) = 2032$, $T = 295\,(1)$ K, $wR = 0{\cdot}033$ for 6115 unique reflections with $F_o \geq 4\sigma(F_o)$. The dimer consists of two Ru atoms bridged by two carbonyl ligands, an acetate ligand and a metal–metal bond, with the octahedral coordination around each metal atom completed by a terminal carbonyl and a chelate bipyridine ligand. The two octahedra thus formed share an edge *via* μ-CO ligands, resulting in the juxtaposition of two Ru atoms in an edge-sharing bioctahedron with an Ru—Ru distance of 2·709 (1) Å.

Introduction. Although a number of dimers containing the metal–metal bonded ruthenium(I)–ruthenium(I) unit are known, of which $[(\eta^5\text{-}C_5H_5)Ru(CO)_2]_2$ (Fischer & Vogler, 1962) represents the most widely studied, reliable synthetic routes to ruthenium(I) complexes are not available. To address this problem we have investigated the use of the readily available carboxylato polymer *catena*-$[Ru(O_2CMe)(CO)_2]$ [(1), Me = CH_3] (Crooks, Johnson, Lewis, Williams & Gamlen, 1969) as a precursor for ruthenium(I) dimers. Our investigations have illustrated the versatility of (1) and the acetonitrile derivative, $[Ru(O_2CMe)(CO)_2(NCMe)]_2$, in the syntheses of a range of neutral and cationic dimeric complexes containing isocyanide (Albers, Liles, Singleton, Stead & Steyn, 1986) and mono- and bidentate tertiary phosphorus ligands (Sherlock, Cowie, Singleton & Steyn, 1988; Singleton, Steyn & van Rooyen, 1988). The acetate complex provides the only readily accessible precursor for high-yield syntheses of the known carbonyl dimers, $[(\eta^2\text{-}C_5R_5)Ru(CO)_2]_2$ (R = H, Me), and the new polypyrazolylborato analogue, $[\{HB(pz)_3\}Ru(CO)_2]_2$ (pz = pyrazolyl) (Liles, Robinson, Singleton & Steyn, 1987). The extension of this study of reactions of (1) with a variety of diamine ligands led to the formation of a series of cationic bioctahedral structures with bridging carbonyl ligands. Confirmatory structural evidence has been

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Table 1. *Fractional coordinates* (× 10^4, × 10^5 *for* Ru) *and equivalent isotropic temperature factors* (Å^2 × 10^3, × 10^4 *for* Ru) *for* $[Ru_2(\mu\text{-}O_2CCH_3)(\mu\text{-}CO)_2(CO)_2(bipy)_2](BPh_4)$

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*(\mathbf{a}_i.\mathbf{a}_j).$$

	x	y	z	U_{eq}
Ru(1)	18788 (2)	52125 (2)	18523 (2)	312 (1)
Ru(2)	16238 (2)	67238 (2)	18200 (2)	326 (1)
C(1)	2150 (3)	5096 (3)	787 (3)	47 (1)
O(1)	2279 (3)	5021 (2)	116 (2)	79 (1)
C(2)	1952 (3)	6929 (3)	792 (3)	43 (1)
O(2)	2161 (2)	7042 (2)	166 (2)	66 (1)
C(3)	858 (3)	5854 (2)	1273 (3)	37 (1)
O(3)	145 (2)	5770 (2)	842 (2)	50 (1)
C(4)	2707 (3)	6088 (2)	2256 (3)	35 (1)
O(4)	3415 (2)	6182 (2)	2634 (2)	46 (1)
O(51)	1568 (2)	5287 (2)	3083 (2)	39 (1)
O(52)	1303 (2)	6528 (2)	3030 (2)	40 (1)
C(51)	1394 (3)	5902 (3)	3412 (3)	37 (1)
C(52)	1317 (3)	5876 (3)	4348 (3)	49 (1)
N(11)	2826 (2)	4367 (2)	2476 (2)	37 (1)
N(12)	1167 (2)	4149 (2)	1704 (2)	35 (1)
C(111)	3667 (3)	4506 (3)	2812 (3)	48 (1)
C(112)	4218 (3)	3969 (3)	3289 (3)	56 (1)
C(113)	3897 (3)	3287 (3)	3423 (3)	59 (1)
C(114)	3045 (4)	3122 (3)	3065 (3)	56 (1)
C(115)	2520 (3)	3672 (3)	2577 (3)	38 (1)
C(121)	345 (3)	4062 (3)	1249 (3)	46 (1)
C(122)	−86 (3)	3381 (3)	1189 (3)	52 (1)
C(123)	347 (4)	2766 (3)	1603 (4)	58 (2)
C(124)	1186 (3)	2844 (3)	2056 (3)	52 (1)
C(125)	1598 (3)	3542 (2)	2111 (3)	41 (1)
N(21)	2199 (2)	7764 (2)	2485 (2)	37 (1)
N(22)	582 (2)	7572 (2)	1527 (2)	40 (1)
C(211)	3008 (3)	7819 (3)	2964 (3)	47 (1)
C(212)	3316 (3)	8449 (3)	3455 (3)	49 (1)
C(213)	2756 (3)	9040 (3)	3438 (3)	50 (1)
C(214)	1923 (3)	8993 (3)	2937 (3)	47 (1)
C(215)	1655 (3)	8354 (2)	2460 (3)	39 (1)
C(221)	−212 (3)	7450 (3)	997 (3)	50 (1)
C(222)	−835 (3)	8011 (3)	809 (3)	58 (1)
C(223)	−636 (3)	8711 (3)	1178 (3)	59 (1)
C(224)	170 (3)	8841 (3)	1698 (3)	52 (1)
C(225)	783 (3)	8264 (3)	1884 (3)	42 (1)
B	6446 (3)	5889 (3)	3277 (3)	37 (1)
C(311)	7062 (2)	7204 (2)	4033 (2)	52 (1)
C(312)	6967 (2)	7874 (2)	4464 (2)	68 (2)
C(313)	6186 (2)	8032 (2)	4683 (2)	74 (2)
C(314)	5501 (2)	7520 (2)	4471 (2)	69 (2)
C(315)	5597 (2)	6849 (2)	4040 (2)	55 (1)
C(316)	6377 (2)	6691 (2)	3821 (2)	42 (1)
C(321)	5354 (2)	5178 (2)	1948 (2)	61 (1)
C(322)	4710 (2)	5172 (2)	1166 (2)	82 (2)
C(323)	4332 (2)	5848 (2)	809 (2)	81 (2)
C(324)	4598 (2)	6529 (2)	1234 (2)	73 (2)
C(325)	5242 (2)	6534 (2)	2016 (2)	56 (1)
C(326)	5620 (2)	5859 (2)	2373 (2)	42 (1)
C(331)	6156 (2)	5147 (1)	4686 (2)	47 (1)
C(332)	6120 (2)	4491 (1)	5166 (2)	56 (1)
C(333)	6366 (2)	3802 (1)	4874 (2)	54 (1)
C(334)	6647 (2)	3768 (1)	4102 (2)	50 (1)
C(335)	6683 (2)	4424 (1)	3622 (2)	46 (1)
C(336)	6438 (2)	5113 (1)	3914 (2)	40 (1)
C(341)	7419 (2)	6015 (2)	2103 (2)	42 (1)
C(342)	8219 (2)	6021 (2)	1886 (2)	54 (1)
C(343)	8979 (2)	5886 (2)	2529 (2)	63 (2)
C(344)	8940 (2)	5744 (2)	3391 (2)	58 (2)
C(345)	8141 (2)	5738 (2)	3608 (2)	49 (1)
C(346)	7381 (2)	5873 (2)	2965 (2)	39 (1)

obtained for the 2,2′-bipyridine complex by performing an X-ray structural determination.

Table 2. *Selected bond lengths* (Å) *and angles* (°) *for* $[Ru_2(\mu\text{-}O_2CCH_3)(\mu\text{-}CO)_2(CO)_2(bipy)_2](BPh_4)$

Ru(1)—Ru(2)	2·709 (1)	Ru(1)—C(1)	1·843 (6)
Ru(1)—C(3)	2·009 (4)	Ru(1)—C(4)	2·035 (4)
Ru(1)—O(51)	2·117 (3)	Ru(1)—N(11)	2·181 (3)
Ru(1)—N(12)	2·183 (3)	Ru(2)—C(2)	1·854 (5)
Ru(2)—C(3)	2·022 (4)	Ru(2)—C(4)	2·032 (4)
Ru(2)—O(52)	2·118 (3)	Ru(2)—N(21)	2·205 (3)
Ru(2)—N(22)	2·201 (4)	C(1)—O(1)	1·132 (7)
C(2)—O(2)	1·131 (7)	C(3)—O(3)	1·180 (5)
C(4)—O(4)	1·149 (5)	O(51)—C(51)	1·266 (6)
O(52)—C(51)	1·253 (6)	B—C(316)	1·679 (6)
B—C(326)	1·685 (6)	B—C(336)	1·705 (6)
B—C(346)	1·675 (6)		
Ru(2)—Ru(1)—O(51)	83·6 (1)	Ru(2)—Ru(1)—C(1)	99·3 (1)
C(3)—Ru(1)—N(11)	170·3 (2)	C(3)—Ru(1)—C(4)	95·7 (2)
C(4)—Ru(1)—N(12)	166·4 (1)	C(1)—Ru(1)—O(51)	177·1 (2)
N(11)—Ru(1)—N(12)	74·8 (1)	C(4)—Ru(1)—N(11)	93·5 (1)
C(3)—Ru(2)—C(4)	95·4 (2)	C(3)—Ru(1)—N(12)	95·8 (2)
C(2)—Ru(2)—O(52)	176·9 (2)	Ru(1)—Ru(2)—C(2)	97·9 (1)
C(3)—Ru(2)—N(21)	167·5 (2)	Ru(1)—Ru(2)—O(52)	83·6 (1)
C(3)—Ru(2)—N(22)	95·0 (2)	C(4)—Ru(2)—N(21)	94·8 (1)
O(52)—Ru(2)—N(22)	88·1 (1)	C(4)—Ru(2)—N(22)	169·1 (1)
Ru(1)—C(1)—O(1)	177·0 (4)	N(21)—Ru(2)—N(22)	74·4 (1)
Ru(1)—C(3)—Ru(2)	84·4 (2)	Ru(2)—C(2)—O(2)	178·7 (4)
Ru(1)—O(51)—C(51)	123·5 (3)	Ru(1)—C(4)—Ru(2)	83·5 (2)
O(51)—C(51)—O(52)	125·3 (4)	Ru(2)—O(52)—C(51)	123·5 (3)
C(316)—B—C(336)	111·9 (3)	C(316)—B—C(326)	108·9 (3)
C(316)—B—C(346)	109·7 (3)	C(326)—B—C(336)	110·8 (3)
C(336)—B—C(346)	107·1 (3)	C(326)—B—C(346)	108·4 (3)

Experimental. A suspension of (1) in boiling ethanol dissolves within 30 min of treatment with two equivalents of 2,2′-bipyridine to give a clear orange solution. Addition of $NaBPh_4$ afforded the title complex as orange crystals in 65% yield, m.p. 408 K (dec.). Analysis: found: C, 59·95; H, 3·74; N, 5·73%; calculated for $C_{50}H_{39}BN_4O_6Ru_2$: C, 59·77; H, 3·91; N, 5·58%; ^{1}H NMR (300 MHz, acetone-d_6, 303 K) $\delta = 10{\cdot}29$ (*d*, $J = 5{\cdot}4$ Hz, 4H, bipy), 8·87 (*d*, $J = 8{\cdot}0$ Hz, 4H, bipy), 8·51 (*td*, $J = 7{\cdot}9$, 1·6 Hz, 4H, bipy), 8·20 (*ddd*, $J = 7{\cdot}6$, 5·6, 1·5 Hz, 4H, bipy), 7·33 (*m*, 8H, BPh_4), 6·91 (*t*, $J = 7{\cdot}4$, 8H, BPh_4), 4·12 (*tt*, $J = 7{\cdot}2$, one *t* unresolved, 4H, BPh_4), 0·98 (*s*, 3H, O_2CMe); IR(CH_2Cl_2, cm^{-1}) ν(CO) 2025, 1996(*sh*), 1747, ν(acetate) 1539, 1479.

Orange needle-shaped crystals deposited from dichloromethane/ethanol; crystal of dimensions 0·3 × 0·5 × 0·3 mm used for data collection; Enraf–Nonius CAD-4F diffractometer with graphite-crystal-monochromatized Mo $K\alpha$ radiation; cell dimensions and their e.s.d.'s obtained by least-squares refinement of 25 reflections ($8 < \theta < 18°$); 7808 measured, 6956 unique ($R_{int} = 0{\cdot}0636$) reflections [$\omega/2\theta$ scans, scan angle $(0{\cdot}60 + 0{\cdot}17\tan\theta)°$, $(\sin\theta/\lambda)_{max} = 0{\cdot}59$ Å^{-1}, *hkl* range: +18, +21, ±18]; orientation (every 200 reflections) and intensity control (every 3600 s) by means of three standard reflections ($15\bar{4}$, $60\bar{4}$, $6,13,\bar{2}$), no significant changes during data collection; corrections for background, Lorentz, polarization effects and absorption (transmission factors min.–max: 0·8268–0·8689) (Coppens, Leiserowitz & Rabinovich, 1965); space group indicated by systematic absences; solution by Patterson methods and location of remaining atoms by difference Fourier synthesis; H atoms calculated at idealized geometric positions; phenyl rings of the counter ions refined as regular hexagons; refinement with anisotropic thermal parameters, H atoms with common isotropic thermal parameter [0·075 (3) Å^2] (546 refined parameters, blocked matrix); function minimized: $\sum w(|F_o| - |F_c|)^2$, where $w = 1·1839\sigma^{-2}(F_o)$; $R = 0·040$, $wR = 0·033$; max. shift/e.s.d. for the positional parameters in final cycles = 0·1; final difference map qualitatively featureless, max. residual electron density: 0·5 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (Cromer & Waber, 1974); computations performed with *SHELX*76 (Sheldrick, 1976) and figure drawn with *ORTEP* (Johnson, 1965).

The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1. Table 2 lists principal bond lengths and angles.*

Discussion. A perspective view of $[Ru_2(\mu\text{-}O_2CMe)(CO)_2(\mu\text{-}CO)_2(bipy)_2]^+$ with numbering scheme is shown in Fig. 1.

The most significant feature of the structure of the complex cation is the edge-sharing bioctahedral configuration of the core atoms. The two Ru atoms are at a distance of 2·709 (1) Å which is within the range for a single metal–metal bond. The C atoms from the bridging carbonyls, the N atoms and the dimetal unit form an approximately planar array, though the individual planes around each metal have a dihedral angle of 18·9 (2)° between them. Perpendicular to this plane are the *M*—O and *M*—C (terminal carbonyl) vectors. Bond angles around the Ru atoms range from 74·4 (1) to 95·8 (2)°; the largest angular distortions from ideal octahedral angles are those of N(11)—Ru(1)—N(12) and N(21)—Ru(2)—N(22). The angles at the C atoms in the central $Ru(\mu\text{-}CO)_2Ru$ quadrilateral are 84·4 (2) and 83·5 (2)° and 95·7 (2) and 95·4 (2)° at the Ru atoms.

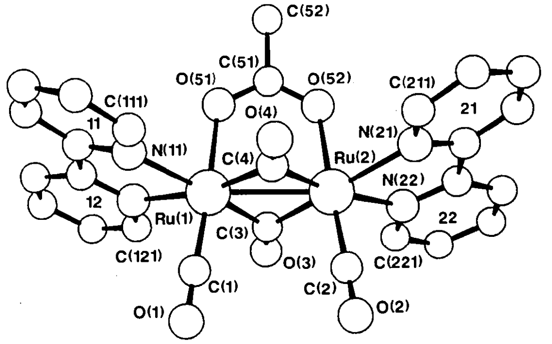

Fig. 1. A perspective drawing of the complex cation showing the atom-numbering scheme (H atoms not shown).

Metal–metal interactions across the shared edge of the bioctahedral structure and the role of bridging acceptor ligands have been discussed from a theoretical point of view in an overview of M_2L_{10} complexes (Shaik, Hoffmann, Fisel & Summerville, 1980). To date bridging donor–acceptor bonds have mainly been observed for Ru^{I} complexes buttressed by terminal cyclopentadienyl ligands (Fischer & Vogler, 1962; Davies, Gracey, Guerchais, Knox & Orpen, 1984). The structures of the related homodinuclear compounds (Field, Haines, Sampson, Sundermeyer & Moodley, 1987) bridged by diphosphine ligands and having bridging carbonyl ligands in the plane perpendicular to that defined by two Ru and four P atoms were found to be dependent on both the donor/acceptor and stereochemical properties of the bridging diphosphorus ligands. The title complex together with $[Ru_2(\mu\text{-}O_2CMe)(\mu\text{-}CO)_2(CO)_2(dppe)_2]PF_6$ [dppe = *P,P,P′,P′*-tetraphenylethylenebis(phosphine)] (Sherlock, Cowie, Singleton & Steyn, 1988) represent rare examples of this geometry for metal–metal bonded complexes of Ru^{I} not containing cyclopentadienyl ligands. The presence of the bridging carboxylate group spanning adjacent axial positions of the dimer has the effect of minimizing steric repulsions between axial ligands, thus facilitating the closer approach of the metal atoms. This results in unsymmetrical ligand–ligand repulsive forces around the dinuclear core which causes slight tilting of the equatorial planes. The combination of favourable steric properties of the ligands involved, as well as the electronic properties of the μ-CO ligands in particular lead to the bioctahedral structure observed. This is accompanied by an Ru—Ru separation which is relatively short in comparison with distances observed in related dimers. Replacement of the carboxylate group by other typically bridging anionic groups such as pyrazolyl, hydroxypyridyl or methylaminopyridyl groups results in structurally and stoichiometrically homologous compounds (Singleton & Steyn, 1987) and provides the opportunity to study Ru^{I}—Ru^{I} bonding under a variety of conditions.

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References

ALBERS, M. O., LILES, D. C., SINGLETON, E., STEAD, J. E. & STEYN, M. M. DE V. (1986). *Organometallics*, **5**, 1262–1264.
COPPENS, P., LEISEROWITZ, L. & RABINOVICH, D. (1965). *Acta Cryst.* **18**, 1035–1038.
CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, pp. 71–147. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

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* A full list of bond lengths and angles, lists of structure factors, atomic coordinates and isotropic temperature factors for H atoms, anisotropic temperature factors and least-squares planes for equatorial planes around the metal centres have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51076 (47 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

CROOKS, G. R., JOHNSON, B. F. G., LEWIS, J., WILLIAMS, I. G. & GAMLEN, G. (1969). *J. Chem. Soc. A*, pp. 2761–2766.
DAVIES, L. D., GRACEY, B. P., GUERCHAIS, V., KNOX, S. A. R. & ORPEN, A. G. (1984). *J. Chem. Soc. Chem. Commun.* pp. 841–843, and references therein.
FIELD, J. S., HAINES, R. J., SAMPSON, C. N., SUNDERMEYER, J. & MOODLEY, K. G. (1987). *J. Organomet. Chem.* **322**, C7–C12.
FISCHER, E. O. & VOGLER, A. (1962). *Z. Naturforsch. Teil B*, **17**, 421–422.
JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
LILES, D. C., ROBINSON, D. J., SINGLETON, E. & STEYN, M. M. DE V. (1987). Unpublished results.
SHAIK, S., HOFFMANN, R., FISEL, C. R. & SUMMERVILLE, R. H. (1980). *J. Am. Chem. Soc.* **102**, 4555–4572.
SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
SHERLOCK, S. J., COWIE, M., SINGLETON, E. & STEYN, M. M. DE V. (1988). *Organometallics*, **7**, 1663–1666.
SINGLETON, E. & STEYN, M. M. DE V. (1987). Unpublished results.
SINGLETON, E., STEYN, M. M. DE V. & VAN ROOYEN, P. H. (1988). *S. Afr. J. Chem.* Submitted.

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Structure of a Ternary Cobalt(II) Complex: Triaqua(2,2′-bipyridyl)(monophenyl phosphato)cobalt(II) Monohydrate

BY T. GŁOWIAK AND A. W. SZEMIK

Institute of Chemistry, University of Wrocław, F. Joliot-Curie Street 14, 50-383 *Wrocław, Poland*

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Abstract. $[Co(C_{10}H_8N_2)(C_6H_5OPO_3H)(H_2O)_3].H_2O$, monoclinic, $P2_1/c$, $a = 7{\cdot}145\ (3)$, $b = 24{\cdot}141\ (6)$, $c = 11{\cdot}495\ (4)$ Å, $\beta = 94{\cdot}90\ (4)°$, $V = 1975{\cdot}5\ (8)$ Å^3, $Z = 4$, $D_m = 1{\cdot}54\ (1)$ (flotation in chloroform and carbon tetrachloride), $D_x = 1{\cdot}544\ (1)$ Mg m^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu(\text{Mo}\ K\alpha) = 1{\cdot}03$ mm^{-1}, $F(000) = 948$, $T = 293$ K. Final $R = 0{\cdot}058$ for 2238 observed reflections. The Co^{II} coordination is a distorted octahedron, best described as a shortened tetragonal bipyramid, formed by the chelating bipyridyl ligand, three water molecules (two axial), and one oxygen atom of the phosphate group. Co—O(ax) are 2·060 (4), Co—O(eq) are 2·123 (4) for O—P and 2·139 (4) for O(water), Co—N(eq) average to 2·129 (5) Å.

Introduction. The structure of the title compound has been determined as a further step in the synthesis and structural study of metal(II) complexes with aryl phosphates and other coordinating *N*, *N*-, *N*—O, and O—O donor ligands. This study complements the recently published structures of the mixed-ligand complexes $[Cu(bpy)_2(MPhP)].MPhP.H_2O$ (MPhP = monophenyl phosphate) (Głowiak, Szemik & Wnęk, 1986) and $[Cu(DPhP)_2(H_2O)_2]$ (DPhP = diphenyl phosphate) (Głowiak & Szemik, 1987).

Experimental. The compound was obtained by reaction of cobalt(II) monophenyl phosphate and 2,2′-bipyridyl in aqueous solution. Orange crystals, 0·35 × 0·40 × 0·48 mm, unit-cell dimensions from a least-squares fit for 15 reflections ($22 < 2\theta < 30°$), Syntex $P2_1$ diffractometer, graphite-monochromatized Mo $K\alpha$ radiation, variable θ–2θ scans, $2\theta_{max} = 55°$, scan rate 2·0–29·3° min^{-1}. Two standard reflections every 50 measurements (intensity variation ±5%), 2878 independent reflections ($0 \le h \le 9$, $0 \le k \le 30$, $-16 \le l \le 15$), 2238 with $I > 3\sigma(I)$, Lp corrections, absorption ignored. Patterson and Fourier method for structure determination, full-matrix least-squares anisotropic refinement, H atoms of water molecules could not be located from difference Fourier syntheses and were not included, other H atoms were included in calculated positions and fixed isotropic thermal parameter $B = 5{\cdot}5$ Å^2, 253 parameters refined, final $R = 0{\cdot}058$, $wR = 0{\cdot}065$ for 2238 reflections, $S = 3{\cdot}58$, minimizing $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{max} = 0{\cdot}25$, $(\Delta\rho)_{max} = +0{\cdot}53$ and $(\Delta\rho)_{min} = -0{\cdot}45$ e Å^{-3}, scattering factors from *International Tables for X-ray Crystallography* (1974). Computer programs: Syntex (1976) *XTL* system.

Discussion. The final parameters for non-hydrogen atoms are listed in Table 1.* Bond lengths and valence angles are given in Table 2. Fig. 1 shows a perspective view of $[Co(bpy)(MPhP)(H_2O)_3].H_2O$ including the atomic nomenclature used here.

The geometry about the six-coordinate cobalt atom involves a slightly distorted octahedron with a characteristic foreshortening (0·07 Å) along the O(6)—Co—

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51039 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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