Crystal Structure of Bis-(2,2′:6′,2″-terpyridyl)cobalt(II) Bromide Trihydrate

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The crystal structure of the title compound has been determined by Patterson and Fourier methods from single-crystal *X*-ray diffractometer data, and refined by least squares to R 0·096 (2704 reflections). Crystals are triclinic, probable space group $P\bar{1}$, $a = 19{\cdot}763(4)$, $b = 9{\cdot}563(2)$, $c = 8{\cdot}537(1)$ Å, $\alpha = 95{\cdot}96(1)$, $\beta = 93{\cdot}48(1)$, $\gamma = 94{\cdot}44(1)$°, $Z = 2$. Within the cation, each terpyridyl ligand co-ordinates *via* the three nitrogen atoms, the central Co–N distance being shorter than the outer [$\langle$Co–N$\rangle$, 1·89, 2·10(1) Å respectively] and the ligand being strained to accommodate the bonding; the overall cation symmetry is approximately D_{2d}. The bromide ions are disordered between lattice sites *ca.* 2·0 Å apart; the potential barrier between the two sites appears small and may be the determinant of the ' crossover ' situation which exists in this complex.

IN spite of the recent proliferation of examples of crystal-field ' crossover ' situations involving temperature-dependent equilibria between closely energetic states of different spin, there are still few cases in which the molecule has intrinsic symmetry rendering it amenable in some measure to meaningful theoretical treatment; the number of systems for which structural data is available is also limited. One of the more attractive systems is the series of salts of the bis-(2,2′:6′,2″-terpyridyl)cobalt(II) cation, $[Co(terpy)_2]^{2+}$, in which the crossover situation appears to be sufficiently delicately balanced to be determined by the lattice forces.[1] The bromide, $[Co(terpy)_2]Br_2,3H_2O$, has a room-temperature

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[1] J. S. Judge and W. A. Baker, *Inorg. Chim. Acta*, 1967, **1**, 68.

Table 1

Atomic fractional cell ($\times 10^4$) and thermal parameters ($\times 10^3$), with least-squares estimated standard deviations in parentheses

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
(*a*) The cation									
Co	7543(1)	6517(2)	4204(3)	60(1)	38(1)	65(2)	3(1)	8(1)	3(1)
(i) Ligand (1) unique atoms									
N(11)	8500(5)	655(1)	425(1)	70(7)	35(6)	43(6)	3(5)	0(5)	8(5)
C(13)	9909(7)	671(2)	406(2)	60(10)	67(10)	94(12)	−4(8)	7(8)	15(9)
(ii) Ligand (1) part (a)									
N(1a2)	7669(5)	446(1)	316(1)	64(7)	40(6)	60(7)	−6(5)	4(6)	1(5)
C(1a1)	8812(7)	542(1)	355(2)	63(9)	40(7)	59(9)	5(6)	11(7)	11(6)
C(1a2)	9506(7)	551(1)	343(2)	70(10)	70(10)	66(10)	26(8)	16(8)	18(8)
C(1a4)	8317(7)	423(1)	305(2)	62(9)	47(8)	64(9)	8(7)	13(7)	2(7)
C(1a5)	7174(8)	343(1)	281(2)	79(11)	64(9)	78(11)	−1(8)	−1(9)	12(8)
C(1a6)	8504(8)	288(2)	250(2)	96(12)	57(9)	76(11)	21(8)	11(9)	−6(8)
C(1a7)	7333(9)	203(2)	224(2)	101(14)	71(11)	116(15)	−17(10)	−4(11)	−1(10)
C(1a8)	7987(9)	180(2)	213(2)	112(14)	44(9)	124(15)	−1(9)	12(12)	−18(9)
(iii) Ligand (1) part (b)									
N(1b2)	7831(5)	859(1)	526(1)	64(7)	35(6)	59(7)	7(5)	10(6)	6(5)
C(1b1)	8907(6)	770(1)	484(2)	45(8)	39(7)	72(9)	1(6)	12(7)	5(7)
C(1b2)	9592(7)	781(1)	482(2)	63(9)	56(9)	77(10)	−4(7)	12(8)	7(8)
C(1b4)	8508(7)	886(1)	554(2)	52(8)	55(8)	67(9)	−11(7)	2(7)	21(7)
C(1b5)	7415(7)	957(1)	572(2)	75(10)	47(8)	82(11)	8(7)	15(8)	1(8)
C(1b6)	8796(8)	012(1) †	635(2)	75(10)	38(8)	101(12)	−10(7)	6(9)	−2(8)
C(1b7)	7677(8)	084(1) †	651(2)	87(12)	49(9)	101(13)	2(8)	19(10)	−7(8)
C(1b8)	8355(8)	111(1) †	685(2)	81(11)	43(9)	125(14)	−13(8)	7(10)	−5(9)
(iv) Ligand (2) unique atoms									
N(21)	6597(5)	661(1)	418(1)	71(8)	45(6)	51(7)	−6(6)	9(6)	11(5)
C(23)	5227(8)	702(2)	428(2)	63(11)	119(15)	111(14)	16(10)	13(10)	36(12)
(v) Ligand (2) part (a)									
N(2a2)	7359(5)	730(1)	204(1)	54(7)	41(6)	75(8)	5(5)	3(6)	−3(6)
C(2a1)	6259(7)	719(1)	301(2)	57(9)	59(9)	83(11)	1(8)	5(8)	6(8)
C(2a2)	5565(8)	738(2)	306(2)	71(11)	98(13)	103(13)	17(9)	6(10)	34(11)
C(2a4)	6702(7)	758(1)	178(2)	67(9)	51(8)	64(9)	1(7)	0(7)	−1(7)
C(2a5)	7809(7)	764(1)	100(2)	74(10)	43(8)	69(10)	1(7)	25(8)	4(7)
C(2a6)	6503(8)	822(1)	044(2)	74(11)	72(10)	80(11)	11(8)	−8(8)	16(9)
C(2a7)	7624(8)	829(1)	−034(2)	95(12)	57(9)	64(10)	0(8)	13(9)	6(8)
C(2a8)	6953(8)	858(2)	−057(2)	96(12)	62(10)	80(11)	13(9)	11(9)	12(8)
(vi) Ligand (2) part (b)									
N(2b2)	7338(5)	569(1)	631(1)	53(7)	40(6)	69(8)	7(5)	1(6)	1(5)
C(2b1)	6259(7)	623(1)	537(2)	53(9)	48(8)	79(10)	18(7)	18(7)	35(7)
C(2b2)	5562(8)	643(2)	550(2)	78(11)	102(13)	80(12)	4(10)	12(9)	22(10)
C(2b4)	6672(7)	561(1)	657(2)	53(8)	56(8)	60(9)	0(7)	7(7)	−4(7)
C(2b5)	7767(7)	516(1)	727(2)	66(10)	52(9)	88(11)	8(7)	0(8)	−3(8)
C(2b6)	6435(8)	499(2)	787(2)	75(11)	75(11)	84(12)	4(9)	12(9)	11(9)
C(2b7)	7561(9)	452(2)	859(2)	95(12)	74(11)	79(11)	19(9)	6(9)	5(9)
C(2b8)	6889(9)	444(2)	883(2)	106(13)	62(10)	81(11)	−4(9)	15(10)	18(9)
(*b*) Other species									
Br(1)	9494(1)	7347(2)	9099(3)	92(1)	60(1)	121(2)	12(1)	14(1)	10(1)
Br(21) *	5419(2)	3500(7)	0967(6)	67(3)	231(7)	106(4)	−7(3)	20(2)	20(4)
Br(22) *	5361(4)	142(1)	115(1)	138(7)	228(11)	265(12)	93(7)	−53(7)	−53(8)
O(1)	9150(6)	390(1)	912(1)	83(8)	76(8)	138(11)	−10(6)	0(7)	2(7)
O(2)	9486(6)	001(1)	206(1) †	123(10)	78(8)	120(10)	−1(7)	32(8)	6(7)
O(31) *	576(1)	044(2)	762(3)	113(7) ‡					
O(32) *	600(1)	085(4)	504(4)	148(13) ‡					

* Population parameters: Br(21) 0·472(7), Br(22) 0·389(9), O(31) 0·57, O(32) 0·43 (restricted). † Add 1. ‡ Isotropic.

magnetic moment typical of a mixture of appreciable quantities of both low- and high-spin d^7 species (μ_{eff} 2·94 B.M. at 300 K). We have determined its crystal structure at room temperature and report it here.

EXPERIMENTAL

The title compound was prepared as previously described,[1] recrystallization by slow cooling of a hot aqueous solution yielding flat plates from which a section 0·17 × 0·10 × 0·30 mm suitable for *X*-ray work was obtained.

Cell dimensions were obtained by a least-squares fit of the angular parameters of 15 reflections centred in the counter aperture of a Syntex P$\bar{1}$ diffractometer. A unique set of 3283 independent reflections in the range $2\theta < 100°$ were collected by a conventional 2θ—θ scan, of which 2704 having $I > 2\sigma(I)$ were considered observed and used in the subsequent structure solution and refinement with unit weights.

Crystal Data.—$C_{30}H_{28}Br_2CoN_6O_3$, $M = 738·3$, Triclinic, $a = 19·763(4)$, $b = 9·563(2)$, $c = 8·537(1)$ Å, $\alpha = 95·96(1)$, $\beta = 93·48(1)$, $\gamma = 94·44(1)°$, $U = 1596·0(5)$ Å^3, $D_m = 1·53$, $Z = 2$, $D_c = 1·54$ g cm^{-3}, $F(000) = 740$. Ni-filtered Cu-$K_{\bar{\alpha}}$ radiation, $\lambda = 1·5418$ Å; μ(Cu-$K_{\bar{\alpha}}$) = 77·45 cm^{-1}. Probable space group (see Discussion) $P\bar{1}$ (C_i^1, No. 2).

Intensity statistics showed the space group to be centric; the structure was solved by Patterson and Fourier methods and refined by 9 × 9 block-diagonal least-squares to a final R 0·09, and R' 0·10 $\{R' = [\Sigma(|F_o| - |F_c|)^2/\Sigma|F_o|^2]^{\frac{1}{2}}\}$; anisotropic thermal parameters employed were of the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$. Scattering factors used were for the neutral atoms,[2] those for Co, Br, being corrected for anomalous dispersion ($\Delta f'$, $\Delta f''$).[3] One of the bromine atoms was found to be disordered and was refined as two separate entities with variable population parameters [Br(21,22)]; a nearby water molecule also appeared to be disordered and was refined with populations fixed from a difference map to give a total of 1. Hydrogen atoms were also located from a difference map and their positional parameters refined, isotropic thermal parameters being fixed at 0·10 Å^2; it was not possible to locate the hydrogen atoms of the water molecule in this way and they were not included. In the final stages of refinement, the parameters of the CoN_6 cationic core were refined as a single matrix so as to approximate to a full-matrix process; shifts in the parameters during the final least-squares cycle were $< 0·2\sigma$. The rather high final R is probably a consequence of the disorder in the structure.

All data processing, including absorption correction and computation, was carried out on the CDC 6200 machine at this University using a local adaptation of the '*X*-Ray '72' program system.[4] Final atomic co-ordinates and thermal parameters are listed in Table 1, and distances and angles in Table 2. Hydrogen atom parameters, and structure factors are deposited as Supplementary Publication No. SUP 21032 (15 pp., 1 microfiche).*

TABLE 2

Interatomic distances (Å) and angles (°), with least-squares estimated standard deviations in parentheses

(*a*) Intra-cation geometry

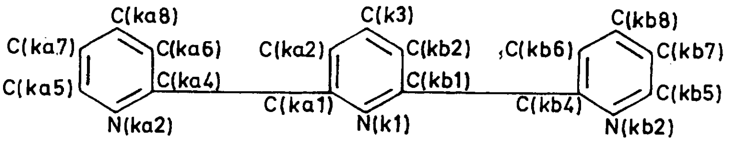
Co–N(11)	1·89(1)	Co–N(21)	1·88(1)
C(1a1)–N(11)–C(1b1)	117(1)	C(2a1)–N(21)–C(2b1)	119(1)
C(1a2)–C(13)–C(1b2)	118(1)	C(2a2)–C(23)–C(2b2)	120(2)
N(11)–Co–N(2a2)	99·2(4)	N(1a2)–Co–N(2a2)	93·5(4)
N(11)–Co–N(2b2)	101·9(4)	N(1a2)–Co–N(2b2)	89·4(4)
N(21)–Co–N(1a2)	104·5(4)	N(1b2)–Co–N(2a2)	89·8(4)
N(21)–Co–N(1b2)	97·7(4)	N(1b2)–Co–N(2b2)	95·4(4)

N(11)–Co–N(21) 176·3(4)

(*b*) Ligand fragment

kl	(1a)	(1b)	(2a)	(2b)
Co–N(kl2)	2·11(1)	2·11(1)	2·09(1)	2·08(1)
N(kl)–C(kl1)	1·39(2)	1·34(1)	1·35(2)	1·32(2)
N(kl2)–C(kl4)	1·32(2)	1·35(2)	1·36(2)	1·34(2)
N(kl2)–C(kl5)	1·33(2)	1·34(2)	1·34(2)	1·32(2)
Co–N(k1)–C(kl1)	120·6(8)	122·4(8)	120·8(8)	120·4(9)
Co–N(kl2)–C(kl4)	112·4(8)	113·2(8)	112·5(9)	113·2(9)
Co–N(kl2)–C(kl5)	125·4(9)	126·9(8)	128·3(9)	127·4(9)
N(k1)–Co–N(kl2)	79·0(4)	78·8(4)	79·6(4)	79·3(4)
N(ka2)–Co–N(kb2)	157·8(4)	—	158·8(4)	—
C(kl4)–N(kl2)–C(kl5)	122(1)	120(1)	119(1)	119(1)
C(kl1)–C(kl4)	1·45(2)	1·51(2)	1·47(2)	1·47(2)
C(kl1)–C(kl2)	1·38(2)	1·35(2)	1·40(2)	1·41(2)
C(kl2)–C(k3)	1·39(2)	1·39(2)	1·33(3)	1·39(3)
C(kl4)–C(kl1)–N(k1)	111(1)	112(1)	113(1)	114(1)
C(kl4)–C(kl1)–C(kl2)	128(1)	123(1)	126(1)	123(1)
N(k1)–C(kl1)–C(kl2)	120(1)	120(1)	122(1)	123(1)
C(kl1)–C(kl2)–C(k3)	118(1)	121(1)	118(1)	117(2)
C(kl4)–C(kl6)	1·41(2)	1·38(2)	1·41(2)	1·40(2)
C(kl5)–C(kl7)	1·44(2)	1·37(2)	1·40(2)	1·40(2)
C(kl6)–C(kl8)	1·39(2)	1·38(2)	1·33(2)	1·35(2)
C(kl7)–C(kl8)	1·34(3)	1·35(2)	1·38(2)	1·35(2)
N(kl2)–C(kl4)–C(kl1)	116(1)	113(1)	114(1)	113(1)
N(kl2)–C(k4)–C(kl6)	121(1)	122(1)	120(1)	121(1)
C(kl1)–C(kl4)–C(kl6)	123(1)	125(1)	126(1)	126(1)
N(kl2)–C(kl5)–C(kl7)	120(1)	120(1)	122(1)	123(1)
C(kl4)–C(kl6)–C(kl8)	118(1)	117(1)	121(1)	118(1)
C(kl5)–C(kl7)–C(kl8)	118(1)	121(1)	118(1)	117(1)
C(kl6)–C(kl8)–C(kl7)	122(1)	120(1)	120(2)	122(2)

The ligand numbering is shown in (I).



(I)

k (= 1 or 2) is the ligand number

DISCUSSION

The crystal structure is comprised of discrete [Co(terpy)$_2$]$^{2+}$ cations. Each cation is accompanied by two bromide ions, one of which is disordered between two sites. Refinement of the population parameters of the two sites in the least-squares procedure gives values of

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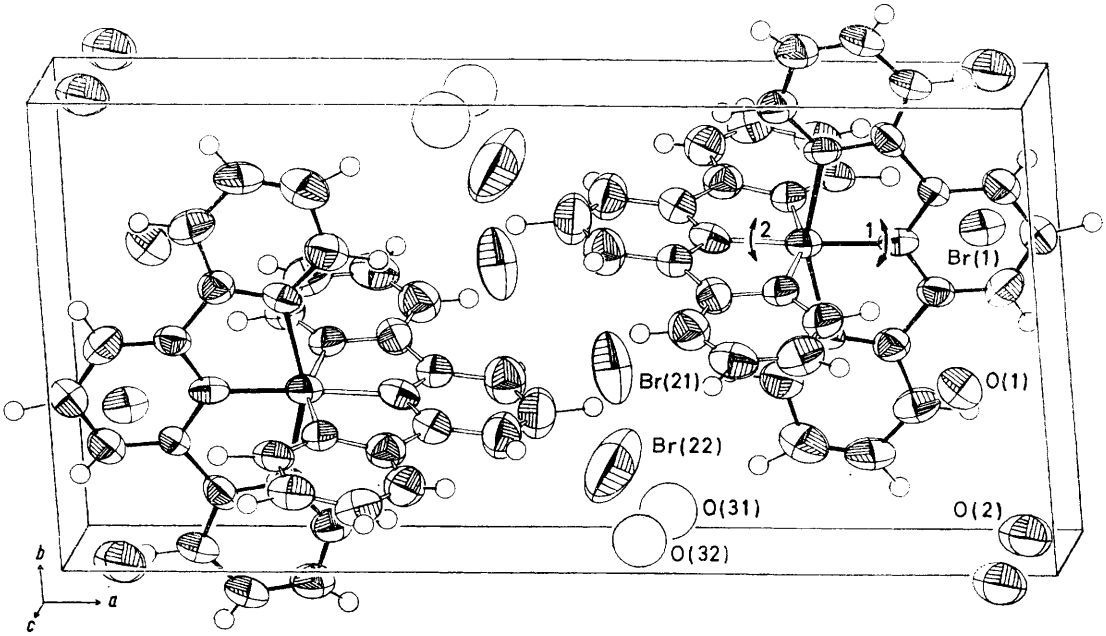
* For details see Notice to Authors No. 7 in *J.C.S. Dalton*, 1973, Index issue.

[2] D. T. Cromer and J. B. Mann, *Acta Cryst.*, **1968**, **A24**, 321.

[3] D. T. Cromer, *Acta Cryst.*, 1965, **18**, 17.

[4] *X*-Ray System of programs, Computer Science Centre, Technical Report TR 192, University of Maryland, U.S.A., 1972.

0·472(7) and 0·389(9); although the sum of these should be unity, it seems unlikely that the complex is non-stoicheiometric or that the bromide occupies additional sites to a lesser extent. The situation appears to be a consequence of poor resolution of the bromide ions because of their high thermal motion. In fact (see Figure), the pair of partial bromide ions have thermal ellipsoids which are grossly elongated along one of the axes; moreover, the separation between their baricentres is only 2·00(1) Å and the long axes of the thermal tensors are mutually directed so as almost to overlap. In view of this, it may well be that the potential-energy difference between the pair of sites is small and that there exists a dynamic migration provoked by thermal motion between the two sites. Such a situation would be compatible with the observed magnetic behaviour. If the [Co(terpy)$_2$]$^{2+}$ system is poised so that ^2E_g and $^4T_{1g}$ states (O_h approximation) are almost equi-energetic then it may be that distortions imposed by the bromide ion *via* lattice forces are determined according to its site: when the bromide is in one site, the cation is ^2E_g, and when the bromide is in the other site, the cation becomes $^4T_{1g}$; the crucial potential-energy barrier in such a situation would not simply be the $^2E_g - {}^4T_{1g}$ separation but could well be dominated by the difference in site energies of the bromide pair. In this case, the final crossover determinant may thus be a lattice disorder potential-energy barrier of a few hundred cm^{-1} rather than the almost purely electronic one observed in the iron(III) dialkyldithiocarbamate system.[5] This situation should be reflected in a number of ways: the magnetic moment, for example, might be expected to correspond at room temperature to a 472 : 389 mixture of ^2E_g and $^4T_{1g}$ components. A calculation assuming $\mu_{\rm eff}(^2E_g)_{295}$ 1·8, and $\mu_{\rm eff}(^4T_{1g})_{295}$ 4·5 B.M. in this ratio yields a value for μ of 3·3 BM; although this value is in the vicinity of that observed (2·9 B.M.) agreement would not be expected to be exact because of the impossibility of refining the total bromide population as 1. In such a situation, the distortion produced by the site exchange of the bromide should be reflected in the ligand; thermal motion generally is high, although not excessively so for a structure of this type, and it is observed that the thermal terms of the carbon atoms in the vicinity of the disordered bromine [C(23), C(1a 7,8)] are the highest. This latter observation is not necessarily remarkable or indicative of ligand displacement in any abnormal degree however, since the carbon atoms involved are peripheral and, as such would be expected to exhibit high thermal motion under normal circumstances. The additional bromine density might also be accounted for by the regions of electron density assigned and refined as the disordered water molecule, the disorder of this species presumably correlating that of the bromine and refined with fixed fractional positional parameters corresponding to about the same ratio estimated from the difference map in which they were located; however, it seems likely that since analysis required three water molecules, this assignment is genuine and that the bromine discrepancy arises from the inability of the scattering factors and thermal tensor to accommodate its disorder properly between the sites. The possibility that the structure was acentric rather than centric was considered, tested, and abandoned; in an acentric space group ($P1$) the



Unit cell contents depicted down c^* (50% ellipsoids). Bonds for ligand (1) are closed, those for ligand (2) open

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[5] A. H. Ewald, R. L. Martin, E. Sinn, and A. H. White, *Inorg. Chem.*, 1969, **8**, 1837.

population parameters of the pair of bromines should be equal although, in the light of the foregoing, the fact that they do not appear to be so may not be conclusive. Intensity statistics and our inability to refine the structure sensibly in $P1$ also led us to conclude that, while the true space group might be $P1$, the perturbation on $P\bar{1}$ was slight and it was not possible to give a meaningful description of it.

TABLE 3

(*a*) Equations of least-squares planes in the form $10^{-4}pX + 10^{-4}qY + 10^{-4}rZ = s$; atomic deviations (Å) are given in square brackets for planes (i) and (ii). *X*, *Y*, and *Z* are orthogonal co-ordinates in Å, so that *X* is parallel to *a*, and *Z* lies in the *ac* plane

	p	q	r	s	σ
Plane (i): Ligand (1)	0504	−3254	9442	1·5851	0·12
Plane (ii): Ligand (2)	1060	9064	4089	8·1830	0·06
Plane (iii): Ligand (1), outer pyridine (a)	0253	−2293	9730	1·5755	0·01
Plane (iv): Ligand (1), central pyridine	0806	−3820	9206	1·6413	0·02
Plane (v): Ligand (1), outer pyridine (b)	−0874	−3900	9167	−1·1713	0·01
Plane (vi): Ligand (2), outer pyridine (a)	1046	8979	4275	8·0707	0·00
Plane (vii): Ligand (2), central pyridine	1357	9121	3869	8·4949	0·01
Plane (viii): Ligand (2), outer pyridine (b)	0356	8824	4691	7·4918	0·01

[N(1a2) −0·12, N(11) −0·02, N(1b2) −0·11, C(1a1) −0·08, C(1a2) −0·15, C(13) −0·10, C(1a4) −0·05, C(1a5) 0·03, C(1a6) 0·08, C(a17) 0·11, C(1a8) 0·18, C(1b1) 0·03, C(1b2) 0·03, C(1b4) 0·07, C(1b5) −0·19, C(1b6) 0·23, C(1b7) −0·05, C(1b8) 0·18]

Non-defining atoms: Co −0·14, N(21) −0·29, N(2a2) −2·21, N(2b2) 1·87.

[N(2a2) −0·04, N(21) −0·02, N(2b2) 0·12, (C2a1) −0·03, C(2a2) 0·00, C(23) 0·06, C(2a4) −0·04, C(2a5) −0·02, C(2a6) −0·02, C(2a7) 0·00, C(2a8) 0·02, C(2b1) 0·01, C(2b2) 0·07, C(2b4) 0·00, C(2b5) 0·11, C(2b6) −0·10, C(2b7) 0·00, C(2b8) −0·13]

Non-defining atoms: Co 0·11, N(11) 0·36, N(1a2) −1·89, N(1b2) 2·21.

(*b*) Angles (°) between planes

(i)–(ii) 84·5, (i)–(iii) 5·9, (i)–(iv) 3·9, (i)–(v) 8·9, (i)–(vi) 83·3, (i)–(vii) 85·7, (i)–(viii) 80·9, (ii)–(iii) 78·9, (ii–(iv) 87·8, (ii)–(v) 89·3, (ii)–(vi) 1·2, (ii)–(vii) 2·1, (ii)–(viii) 5·5, (iii)–(iv) 9·8, (iii)–(v) 11·7, (iii)–(vi) 77·7, (iii)–(vii) 80·2, (iii)–(viii) 75·2, (iv)–(v) 9·6, (iv)–(vi) 86·6, (iv)–(vii) 88·9, (iv)–(viii) 84·4, (v)–(vi) 88·1, (v)–(vii) 89·3, (v)–(viii) 85·3, (vi)–(vii) 3·0, (vi)–(viii) 4·7, (vii)–(viii) 7·6

The present structure determination is the first bis-(terpyridyl)–metal derivative to be so studied. The symmetry of the $[Co(terpy)_2]^{2+}$ species is a very good approximation to D_{2d}, both ligands being tolerably planar and approximately normal (Table 3), although slight but significant deviations from both ideals are observed. The cobalt–nitrogen distances in the present complex are presumably rather longer than would be observed in a purely ' low-spin ' derivative of the type; the Jahn–Teller distortion expected of the low-spin state is imposed automatically by the ligands. Valid comparisons are difficult to find; the complex is obviously far from octahedral and the strain evident in the terpy systems make it equally evident that pure diagonal co-ordination likewise provides an inappropriate description; certainly the cobalt–nitrogen (central) bonds [1·88, 1·89(1) Å] are very short and the others [2·08—2·11(1 Å)] quite long. Table 4 compares the ligand geometry with that found in μ-(2,2′:6′2″-terpyridylcadmium)-bis(pentacarbonylmanganese)(2*Cd*–*Mn*),[6] which has the longest metal–nitrogen distances hitherto reported in a symmetrically co-ordinated terpy ligand. The increase in ligand strain is significant and apparent in the cobalt(II) complex.

TABLE 4

Comparison of terpy ligand geometries

	$[Co(terpy)_2]Br_2,3H_2O$	$(terpy)Cd[(Mn(CO)_5]_2$
⟨M–N(1)⟩	1·89 Å	2·40 Å
⟨M–N(2)⟩	2·10	2·48
⟨N(1)–M–N(2)⟩	79·2	66·5
⟨N(2)–M–N(2′)⟩	158·3	133
⟨N(1)–C(1)–C(4)⟩	112·5	116
⟨N(2)–C(4)–C(1)⟩	114	117·5

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[6] W. Clegg and P. J. Wheatley, *J.C.S. Dalton*, 1973, 90.