

Relatively high e.s.d.'s for the parameters as well as unusually large or small values for a few thermal parameters were obtained. These unsatisfactory results are due to some inevitable difficulties: insufficient accuracies of the $|F_o|$ values owing to the overlap of the reflections from the other individuals of the twinned crystal; the neglect of the geometrical difference between the octahedral Pt^{IV} and planar Pt^{II} complexes; the exclusion of many low-angle reflections owing to the restrictive use of the reflection data (the light atoms contribute mainly to the low-angle reflections).

The positional symmetry of the anions and the water O atoms, as a whole, is closely related to that of 8(c) sites of the space group *I4mm*. The formation of the twinned crystal consisting of four individuals is probably caused by misfits in the arrangement of the anions and the water molecules accompanied by a slight shift between the chains of the complex ions: *i.e.* exchanging the positions of the anions and the water molecules and a $\pi/2$ rotation of the mirror plane around *c*. Twinning occurs in the present complex, but not in Reihlen's green. This appears to depend on whether the deviation of the cell parameter β from 90° is small or large: the deviation is 0.98° in Wolfram's red, whereas it is 2.7° in Reihlen's green.

A rough estimation of the cell parameters at 100 K based on the θ values of six reflections ($15 < \theta < 18^\circ$) is: $a = 13.22$ (7), $b = 13.24$ (3), $c = 5.37$ (1) Å, $\beta = 91.4$ (3)°. No anomalous expansion of the cell dimensions as reported for tetraamineplatinum(II) tetraamminedibromoplatinum(IV) hydrogensulfate (Tanaka, Tsujikawa, Toriumi & Ito, 1982) was observed.

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Aqua[1,9-di(2-pyridyl)-2,5,8-triazanonane]cobalt(III) Nitrate Diperchlorate

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Abstract. [Co(C₁₆H₂₃N₅)(H₂O)](ClO₄)₂(NO₃), $M_r = 623.2$, monoclinic, $P2_1/a$, $a = 16.587$ (3), $b = 10.791$ (2), $c = 13.538$ (2) Å, $\beta = 94.1$ (1)°, $U = 2416.9$ (7) Å³, $Z = 4$, $D_x = 1.71$ Mg m⁻³, $\lambda(\text{Mo K}\alpha) = 0.71069$ Å, $\mu = 93.3$ mm⁻¹, $F(000) = 1280$, $T = 295$ K, $R = 0.064$ ($wR = 0.066$) for 2324 unique observed reflections. The coordination geometry of the complex cation [Co(picdien)(H₂O)]³⁺ is nearly regular

octahedral with the pentadentate picdien ligand in α, β configuration.

Introduction. The great sensitivity of complexes of the type *syn*- and *anti*- α, β [Co(picdien)X]²⁺ ($X = \text{Cl, Br, NO}_2, \text{NCS}$) to base-catalysed hydrolysis which is observed as an inverse-first-order acid-dependent rate law even in moderately acid solution has led us to examine the reverse process which also has an inverse acid dependence (Humanes, 1984). This allows a

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kinetic study of base-catalysed ligand substitution which has not been attempted before. In spite of much effort, only one isomer of the $[\text{Co}(\text{picdien})(\text{H}_2\text{O})]^{3+}$ cation has been isolated and, because of the substitutional lability of the coordinated water in all suitable solvents and the rapid exchange of some of the amine protons in D_2O , it was not possible to assign the configuration through ^1H NMR spectroscopy. Furthermore, although the infrared spectrum of the salt, which was analysed as $\text{Co}(\text{picdien})\text{NO}_3(\text{ClO}_4)_2 \cdot \text{H}_2\text{O}$, indicated that the NO_3 was not bound to the metal, it was not obvious why NaClO_4 should precipitate the nitrate diperchlorate salt from a solution containing no great excess of NO_3^- in preference to the triperchlorate and we wished additional proof that NO_3^- was not coordinated. It was therefore necessary to determine the geometry and isomeric form of the complex cation by a single-crystal X-ray diffraction study.

Experimental. Red-orange crystals of $[\text{Co}(\text{picdien})\text{H}_2\text{O}](\text{ClO}_4)_2(\text{NO}_3)$ were prepared as reported (Humanes, 1984).

Prismatic crystals $0.14 \times 0.08 \times 0.24$ mm; Philips PW 1100 automatic four-circle diffractometer, Mo $K\alpha$ radiation, graphite monochromated; lattice parameters refined by least squares from 25 reflections with $8 < \theta < 12^\circ$. Two standard reflections 322 and 521 measured at 180 min intervals. No significant fluctuations in intensities other than those expected from Poisson statistics; intensities measured up to $2\theta = 50^\circ$; $\theta/2\theta$ -scan techniques, with a scan width 1.20° in θ at a scan speed of $1.8^\circ \text{min}^{-1}$. Intensities corrected for Lorentz and polarization effects; empirical absorption correction using ψ scans (North, Phillips & Mathews, 1968) applied, min., max. transmission coefficients 0.90, 0.95; 4706 reflections measured, including 2324 reflections with $[I \geq 3\sigma(I)]$; index range $-19 < h < 19, 0 < k < 12, 0 < l < 16$.

Co-atom parameters found by direct methods with *SHELX76* (Sheldrick, 1976) and all non-H atoms located in subsequent difference Fourier synthesis; function in full-matrix least-squares refinement $\sum w(\Delta F)^2$; $w = 2.8035[\sigma^2(F_o) + 0.000495(F_o)^2]^{-1}$; all non-H atoms except the two perchlorate groups refined anisotropically, H atoms bound to N atoms included at the positions indicated by difference Fourier synthesis and those on the C atoms included at calculated positions ($\text{C}-\text{H} = 0.95 \text{ \AA}$); H atoms riding on their bonded atoms with a fixed isotropic temperature factor, $U = 0.07 \text{ \AA}^2$ (164 variable parameters). The two independent ClO_4^- anions are disordered, with two main orientations about the Cl atoms, refined with 50% occupancy for each orientation with isotropic thermal parameters. Final $R = 0.064$ and $wR = 0.066$. ρ_{max} in final difference map 0.8 e \AA^{-3} in the proximity of a ClO_4^- anion; $S = 2.15$; final $(\Delta/\sigma)_{\text{max}}$ in final least-squares cycle 0.7 (for ClO_4^- anions) and 0.05 (for the

Table 1. Atomic coordinates ($\times 10^3$) with e.s.d.'s in parentheses, isotropic temperature factors U_{eq} ($\text{\AA}^2 \times 10^3$) or U_{iso} ($\text{\AA}^2 \times 10^3$)

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

	x	y	z	$U_{\text{iso}}^\dagger/U_{\text{eq}}$	K^\ddagger
Co	3205 (1)	6523 (1)	7524 (1)	35.7 (3)	
N(11)	2464 (4)	5112 (6)	7505 (5)	42 (2)	
C(1)	1657 (5)	5180 (9)	7371 (6)	57 (3)	
C(2)	1189 (7)	4143 (12)	7455 (8)	91 (5)	
C(3)	1544 (9)	3044 (12)	7691 (9)	97 (6)	
C(4)	2370 (8)	2952 (10)	7821 (7)	77 (5)	
C(5)	2823 (6)	4031 (8)	7725 (6)	50 (3)	
C(6)	3715 (6)	4096 (8)	7868 (7)	56 (4)	
N(2)	3965 (4)	5364 (6)	8174 (5)	46 (3)	
C(7)	3985 (5)	5555 (9)	9273 (6)	60 (4)	
C(8)	3172 (5)	6015 (9)	9535 (6)	57 (3)	
N(3)	2937 (4)	7041 (6)	8844 (5)	44 (2)	
C(9)	3348 (5)	8233 (8)	9129 (7)	59 (4)	
C(10)	3698 (5)	8808 (8)	8262 (7)	60 (4)	
N(4)	3965 (4)	7866 (6)	7625 (5)	53 (3)	
C(11)	4203 (6)	8208 (8)	6640 (7)	59 (3)	
C(12)	4095 (5)	7163 (8)	5938 (6)	44 (3)	
C(13)	4429 (5)	7132 (10)	5024 (7)	64 (4)	
C(14)	4287 (6)	6125 (11)	4427 (7)	67 (4)	
C(15)	3807 (6)	5187 (10)	4717 (7)	66 (4)	
C(16)	3474 (5)	5282 (9)	5623 (6)	50 (3)	
N(5)	3617 (4)	6238 (5)	6228 (4)	38 (2)	
O(5)	2396 (3)	7608 (5)	6861 (4)	54 (2)	
N(6)	6008 (4)	5550 (7)	7837 (5)	49 (3)	
O(61)	5594 (4)	6276 (7)	7330 (5)	76 (3)	
O(62)	5673 (4)	4731 (6)	8306 (5)	68 (3)	
O(63)	6760 (3)	5619 (6)	7882 (5)	70 (3)	
Cl(1)	6531 (2)	8546 (3)	4396 (2)	80 (2)†	
O(1)	5953 (9)	7833 (15)	3780 (11)	64 (5)†	0.5
O(2)	6950 (11)	9583 (18)	4156 (14)	106 (6)†	0.5
O(3)	6924 (10)	7602 (15)	4943 (12)	71 (5)†	0.5
O(4)	6164 (11)	9369 (18)	5038 (14)	93 (6)†	0.5
O(11)	5905 (12)	8303 (19)	3673 (15)	97 (7)†	0.5
O(12)	7004 (9)	9202 (14)	3686 (12)	73 (4)†	0.5
O(13)	7211 (11)	7602 (17)	4700 (13)	87 (6)†	0.5
O(14)	6169 (18)	8821 (27)	5296 (22)	175 (12)†	0.5
Cl(2)	9124 (1)	3227 (2)	152 (2)	58 (1)†	
O(21)	8632 (12)	4146 (20)	-165 (16)	131 (7)†	0.5
O(22)	9547 (11)	3394 (19)	1138 (13)	105 (6)†	0.5
O(23)	9751 (8)	2800 (14)	-442 (11)	70 (4)†	0.5
O(24)	8563 (7)	2160 (12)	297 (10)	60 (3)†	0.5
O(221)	8934 (12)	4469 (19)	412 (15)	116 (6)†	0.5
O(222)	9570 (17)	2671 (30)	853 (21)	188 (11)†	0.5
O(223)	9551 (12)	3334 (20)	-713 (15)	120 (7)†	0.5
O(224)	8398 (12)	2605 (19)	-152 (15)	121 (7)†	0.5

† Site occupancy factors 1.0 if not shown.

rest of the atoms). Scattering factors and anomalous-dispersion terms for Co from *International Tables for X-ray Crystallography* (1974) and the other atom factors from *SHELX76*.*

Other programs used: *PARST* (Nardelli, 1983) and *PLUTO* for figure drawing (Motherwell & Clegg, 1978).

Discussion. Atomic coordinates are given in Table 1, bond lengths and bond angles in Table 2. The stereochemical arrangement of the complex cation $[\text{Co}(\text{picdien})\text{H}_2\text{O}]^{3+}$ and of the three anions 2ClO_4^- and NO_3^- with the atom numbering is shown in Fig. 1 (only

* 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 44047 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

one of the two orientations for the ClO₄⁻ anions is given).

The coordination geometry of the cation is almost regular octahedral with the pentadentate picdien ligand present in the α,β configuration (Snow, 1972). A water molecule completes the coordination sphere around the metal ion. Two secondary N atoms and the two

Table 2. Distances (Å) and principal angles (°) with *e.s.d.*'s in parentheses

Cobalt environment			
Co—N(1)	1.956 (7)	Co—N(4)	1.919 (7)
Co—N(2)	1.941 (7)	Co—N(5)	1.952 (6)
Co—N(3)	1.954 (7)	Co—N(_{mean})	1.944 (15)
Co—O(5)	1.951 (6)		
N(1)—Co—N(2)	83.8 (4)	N(2)—Co—O(5)	176.6 (5)
N(1)—Co—N(3)	92.9 (4)	N(3)—Co—N(4)	84.8 (4)
N(1)—Co—N(4)	176.2 (3)	N(3)—Co—N(5)	169.8 (5)
N(1)—Co—N(5)	97.2 (4)	N(3)—Co—O(5)	93.2 (3)
N(1)—Co—O(5)	92.9 (4)	N(4)—Co—N(5)	85.0 (4)
N(2)—Co—N(3)	87.3 (3)	N(4)—Co—O(5)	90.4 (4)
N(2)—Co—N(4)	93.0 (4)	N(5)—Co—O(5)	87.4 (3)
N(2)—Co—N(5)	92.8 (3)		
Picdien			
N(1)—C(1)	1.34 (1)	N(3)—C(9)	1.49 (1)
N(1)—C(5)	1.33 (1)	C(9)—C(10)	1.48 (1)
C(1)—C(2)	1.37 (2)	C(10)—N(4)	1.42 (1)
C(2)—C(3)	1.35 (2)	N(4)—C(11)	1.46 (1)
C(3)—C(4)	1.37 (2)	C(11)—C(12)	1.48 (1)
C(4)—C(5)	1.40 (2)	C(12)—C(13)	1.39 (1)
C(5)—C(6)	1.48 (1)	C(12)—N(5)	1.35 (1)
C(6)—N(2)	1.48 (1)	C(13)—C(14)	1.36 (2)
N(2)—C(7)	1.50 (1)	C(14)—C(15)	1.36 (2)
C(7)—C(8)	1.50 (1)	C(15)—C(16)	1.38 (1)
C(8)—N(3)	1.48 (1)	C(16)—N(5)	1.33 (1)
C(1)—N(1)—C(5)	120 (1)	N(3)—C(9)—C(10)	110.7 (8)
N(1)—C(1)—C(2)	121 (1)	C(9)—C(10)—N(4)	109.7 (8)
C(1)—C(2)—C(3)	120 (1)	C(10)—N(4)—C(11)	119.2 (8)
C(2)—C(3)—C(4)	121 (1)	N(4)—C(11)—C(12)	111.4 (8)
C(3)—C(4)—C(5)	118 (1)	C(11)—C(12)—N(5)	115.1 (8)
N(1)—C(5)—C(6)	121 (1)	C(11)—C(12)—C(13)	124 (1)
C(4)—C(5)—C(6)	125 (1)	C(13)—C(12)—N(5)	121.3 (8)
N(1)—C(5)—C(6)	114.4 (9)	C(12)—C(13)—C(14)	119 (1)
C(5)—C(6)—N(2)	109.7 (9)	C(13)—C(14)—C(15)	120 (1)
C(6)—N(2)—C(7)	113.0 (8)	C(14)—C(15)—C(16)	119 (1)
N(2)—C(7)—C(8)	108.8 (7)	C(15)—C(16)—N(5)	122.6 (9)
C(7)—C(8)—N(3)	107.3 (8)	C(12)—N(5)—C(16)	118.5 (7)
C(8)—N(3)—C(9)	112.7 (7)		
Nitrate			
N(6)—O(61)	1.22 (1)	O(61)—N(6)—O(62)	119.3 (9)
N(6)—O(62)	1.24 (1)	O(61)—N(6)—O(63)	120.7 (9)
N(6)—O(63)	1.247 (8)	O(62)—N(6)—O(63)	120.0 (8)

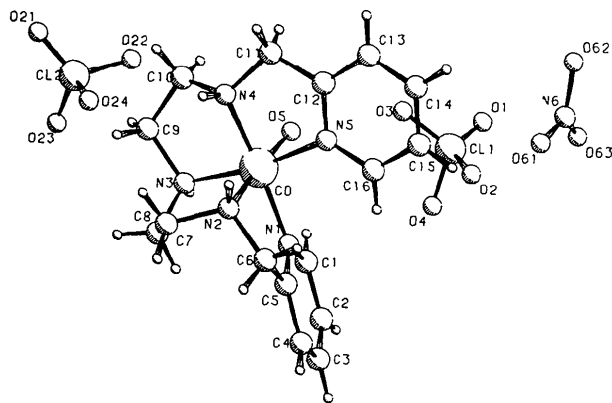


Fig. 1. Perspective view of the complex.

pyridine N atoms of the picdien ligand are nearly coplanar [the deviations of the atoms from their best mean plane are N(1) -0.018 (9), N(3) 0.021 (9), N(4) -0.022 (9) and N(5) 0.018 (8) Å] with the Co atom 0.033 (6) Å out of the plane in the direction of the water molecule. The configuration of the 'flat' (the central member of a meridional set of three) N(4) atom presents both chiralities *R* and *S* as does the 'angular' (the central atom of a facial set of three) N(2), owing to the requirements of the centrosymmetric space group. Therefore, the relative configuration can be designated as $\Delta\text{-}\alpha,\beta\text{-}R$ (or $\Delta\text{-}\alpha,\beta\text{-}S$) (IUPAC, 1970). This is the same as that reported for [Co(picdien)Br](ClO₄)₂ (Bombieri, Forsellini, Del Pra, Tobe, Chatterjee & Cooksey, 1983) where the water position is occupied by a Br atom and for [Co(picdien)Cl](ZnCl₂) (Bombieri, Forsellini, Del Pra & Tobe, 1981) with a Cl atom instead of a water molecule. As in the quoted examples, the N(4)—H bond and the Co—O(H₂O) bond lie on the opposite side with respect to the square plane, so we can label this the *anti* isomer as in the bromine and chlorine derivatives. The two pyridine rings are planar within experimental error and are inclined at 76.3 (3)° (dihedral angle). An examination of the Co—N bond distances reveals the same trend as that of the bromine derivative with the Co—N bond to the 'flat' N atom [Co—N(4) 1.919 (7) Å] shorter than the other Co—N bonds which average 1.951 (7) Å. This has been also found in several Co^{III} complexes with trien (triethylenetetraamine) in the β configuration or picdien in

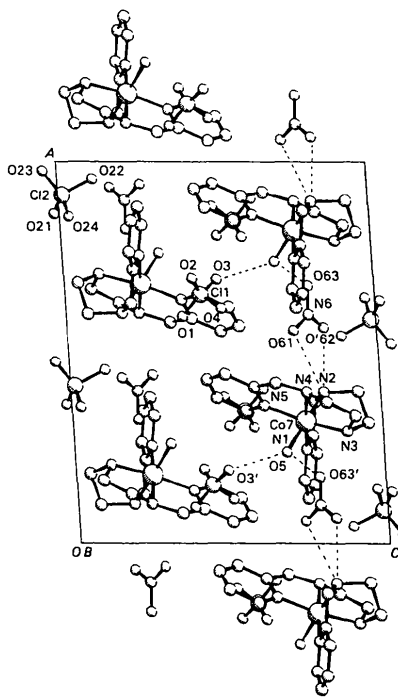


Fig. 2. Unit-cell content as viewed down *b*.

the α and β configurations (Bombieri *et al.*, 1981, 1983; Bombieri, Forsellini, Del Pra, Tobe & Henderson, 1979). Another common characteristic of such compounds is the expansion of the external angle at the 'flat' N(4), C(10)—N(4)—C(11) 119.2 (8)°. This, and the shorter Co—N(4) bond distance, could be a consequence of the reduction of angular strain at this centre. No other significant angular distortions are present in the polyamine ligand and the angles to the metal ions agree with the literature data. The Co—N(2) distance [1.941 (7) Å] *trans* to the Co—O(H₂O) is of the same order as Co—N [1.951 (7) Å] *trans* to the Co—N(NO₂) group in the nitro derivative [Co(picdien)(NO₂)](ClO₄)₂ (Bombieri *et al.*, 1983), showing a comparable *trans* influence by the NO₂ and H₂O groups. While the nitrate group shows regularity in bond distances and angles [angles around 120° and N—O 1.22 (1), 1.24 (1) Å], the perchlorate anions are rather disordered. The disorder has been described with two main orientations of the two ClO₄⁻ anions.

The crystal packing. Fig. 2 shows the packing diagram of the cationic and anionic units together with some details of the hydrogen-bonding scheme present in the structure.

There are some relatively short contacts between O(5)(water) and O(63ⁱ)(nitrate) [(i) at $x - \frac{1}{2}, \frac{3}{2} - y, z$] [O(5)···O(63ⁱ) 2.63 (1) Å], O(5)_{water} and O(3ⁱ)-(perchlorate) [O(5)···O(3ⁱ) 2.67 (2) Å], as well as between the nitrate O atoms O(61) and O(62) with N(2) of the adjacent cationic unit [N(2)···O(61) 3.16 (1), H(21)···O(61) 2.25 (3) Å, N(2)—H(21)···O(61) 162 (2)°; N(2)···O(62) 2.91 (1), H(21)···O(62) 2.12 (3) Å, N(2)—H(21)···O(62) 140 (2)°]. This suggests that the role of the nitrate group in the structure is to establish a hydrogen-bonding system between adjacent cationic units with the formation of chains running in the *a*-axis direction [the O atoms of the nitrate anion

are in contact with the coordinated water molecule of one cationic unit and N(2) of the adjacent cationic unit]. The other interactions with perchlorate anions are dealing with the two orientations of a disordered ClO₄ group {N(3)···O(24ⁱⁱ) [(ii) 1-x, 1-y, 1-z] 2.95 (1), H(31)···O(24ⁱⁱ) 2.10 (4) Å, N(3)—H(31)···O(24ⁱⁱ) 147 (3)°; N(3)···O(224ⁱⁱ) 2.96 (2), H(31)···O(224ⁱⁱ) 2.25 (4) Å, N(3)—H(31)···O(24ⁱⁱ) 131 (3)°, N(4)···O(22ⁱⁱⁱ) [(iii) $\frac{3}{2} - x, \frac{1}{2} + y, 1 - z$] 2.94 (2), H(4)···O(22) 2.12 (3) Å, N(4)—H(4)···O(22) 143 (3)°}.

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cis-Dichlorobis(di-*n*-propyl sulfoxide)platinum(II) and *cis*-Dichlorobis(ethyl methyl sulfoxide)platinum(II)

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Abstract. [PtCl₂(C₆H₁₄OS)₂], $M_r = 534.48$, triclinic, $P\bar{1}$, $a = 8.274$ (5), $b = 11.256$ (6), $c = 11.949$ (7) Å, $\alpha = 115.92$ (4), $\beta = 98.30$ (5), $\gamma = 99.86$ (5)°, $V = 955.5$ (10) Å³, $Z = 2$, $D_x = 1.857$, $D_m = 1.83$ (3) Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu =$

7.91 mm⁻¹, $F(000) = 520$, $T = 295$ K, $R = 0.061$ for 2942 unique observed reflections. Pt has *cis* square-planar coordination to two Cl and to two S atoms of the dipropyl sulfoxide ligands. The bond distances are Pt—Cl = 2.289 (3), 2.302 (3) and Pt—S = 2.263 (3)

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