

Table 3. Distances (Å) and angles (°) in interactions of the type D—H...A

D	H	A	H...A	D—H...A
N(11)	H(112)	O(12)	1.95 (3)	134 (2)
N(111)	H(1B2)	O(121)	1.99 (3)	128 (2)
H <sub>2</sub> O(3)	HO(31)	O(11)	2.49 (2)	127 (2)
H <sub>2</sub> O(2)	HO(21)	H <sub>2</sub> O(4)	1.92 (2)	168 (2)
H <sub>2</sub> O(3)	HO(31)	H <sub>2</sub> O(1)	2.20 (2)	144 (2)
H <sub>2</sub> O(2)	HO(22)	O(10 <sup>b</sup> )	1.64 (2)	173 (1)
N(111)	H(1B1)	H <sub>2</sub> O(3 <sup>ii</sup> )	2.39 (3)	148 (3)
H <sub>2</sub> O(1)	HO(12)	N(51 <sup>ii</sup> )	1.67 (2)	160 (2)
H <sub>2</sub> O(3)	HO(32)	H <sub>2</sub> O(4 <sup>iii</sup> )	2.36 (2)	135 (2)
H <sub>2</sub> O(4)	HO(41)	O(12 <sup>iv</sup> )	2.38 (2)	154 (2)
H <sub>2</sub> O(4)	HO(41)	O(11 <sup>v</sup> )	2.56 (2)	124 (2)
H <sub>2</sub> O(4)	HO(42)	N(5 <sup>v</sup> )	2.08 (2)	162 (2)
H <sub>2</sub> O(4)	HO(42)	O(10 <sup>v</sup> )	2.64 (2)	143 (2)
N(11)	H(111)	H <sub>2</sub> O(3 <sup>vi</sup> )	2.09 (3)	176 (2)
N(111)	H(1B2)	H <sub>2</sub> O(2 <sup>vii</sup> )	2.41 (2)	126 (2)
H <sub>2</sub> O(1)	HO(11)	H <sub>2</sub> O(1 <sup>viii</sup> )	1.86 (2)	157 (2)

Equivalent positions: (i)  $x, y-1, z$ ; (ii)  $1-x, \bar{y}, 1-z$ ; (iii)  $x, y, 1+z$ ; (iv)  $x, y, z$ ; (v)  $x, 1-y, z$ ; (vi)  $x, 1-y, 1-z$ ; (vii)  $1-x, y, z$ ; (viii)  $x, 1+y, z$ .

The intramolecular hydrogen bonds N(11)—H(112)...O(12) and N(111)—H(1B2)...O(121) could explain the appearance of one signal for the resonance of each aminic proton in the <sup>1</sup>H NMR spectrum of this complex dissolved in Me<sub>2</sub>SO<sub>d</sub><sub>6</sub> (Moreno-Carretero, 1983).

On the other hand, it is clear that the difference found between the Zn—O(11) and Zn—O(1) distances is caused by the hydrogen-bond interactions between O(11) and H<sub>2</sub>O(3); furthermore, this could explain why the C(21)—O(11) bond length is slightly longer than the C(2)—O(1) distance.

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## Structure of Hexakis(pyridine *N*-oxide)cobalt(II) Perchlorate by Neutron Diffraction at 90 K

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**Abstract.** [Co(C<sub>5</sub>H<sub>5</sub>NO)<sub>6</sub>](ClO<sub>4</sub>)<sub>2</sub>, *M<sub>r</sub>* = 828.4, rhombohedral, *R* $\bar{3}$ , *a* = 12.317 (7), *c* = 18.827 (10) Å, *V* = 2473.5 Å<sup>3</sup>, *Z* = 3, *D<sub>x</sub>* = 1.668, *D<sub>m</sub>* = 1.62 g cm<sup>−3</sup> (room temperature),  $\lambda$  = 1.271 Å,  $\mu$  = 1.77 cm<sup>−1</sup>, *T* = 90 K, final *R*(*F*<sup>2</sup>) = 0.034 for 1309 independent observed reflections. The [Co(C<sub>5</sub>H<sub>5</sub>NO)<sub>6</sub>]<sup>2+</sup> ion possesses a nearly perfect octahedral coordination

geometry with Co—O = 2.083 (1) Å and O—Co—O = 89.77 (4)°. The analysis reveals that a small amount of the pyridine *N*-oxide ligand (~5%) is displaced by *N,N*-dimethylformamide (DMF) solvent molecules, so that the chemical formula for the crystal examined is more appropriately written as [Co(C<sub>5</sub>H<sub>5</sub>NO)<sub>5.7</sub>(DMF)<sub>0.3</sub>](ClO<sub>4</sub>)<sub>2</sub>.

**Introduction.** In conjunction with a program of study of charge-density distributions of coordination compounds with interesting magnetic properties, we have undertaken a neutron-structure analysis of the title compound,  $[\text{Co}(\text{pyO})_6](\text{ClO}_4)_2$ , to provide data for  $X-N$  deformation-density mapping. This particular compound was chosen for such a study principally because, although the coordination geometry of the Co atom is virtually perfectly octahedral, the trigonal distortions of the electronic properties from those expected for a  $d^7$  ion in  $O_h$  symmetry are unusually large and give rise, among other properties, to weak exchange coupling between nearest-neighbor cations in the pseudo-cubic structure (Algra, de Jongh, Huiskamp & Carlin, 1976). In addition, we were also interested in comparing the deformation density for a coordinated pyridine  $N$ -oxide with that found for 4-nitropyridine  $N$ -oxide (Coppens & Lehmann, 1976) and in particular in examining if the distribution around the O atom would give information relating to the form of the exchange pathway between neighboring complex cations. The present study showed that the crystals used for the neutron analysis contained a small amount of DMF which displaces some of the pyridine  $N$ -oxide ligand. The crystal used for the X-ray work was grown from ethanol and the analysis was not subject to this complicating feature (Wood, 1984, and unpublished results).

**Experimental.** The complex was prepared as previously described (Bergendahl & Wood, 1975), and crystals suitable for neutron-structure analysis were grown from DMF-ethanol solution. Diffractometer used: four-circle instrument D-8 at the ILL High Flux Beam Reactor, Grenoble. Monochromator: Cu crystal, 200 reflection. Cryostat: Displex closed-loop refrigerator (Allibon, Filhol, Lehmann, Mason & Simms, 1981). Cell dimensions: from 15 reflections in range  $25 \leq \theta \leq 30^\circ$ . Crystal dimensions:  $2.03 \times 1.58 \times 0.96$  mm, rhombohedral crystal bounded by faces of the form  $\{11\bar{1}\}$  and  $\{001\}$  (hexagonal indices), volume  $3.03 \text{ mm}^3$ , weight 4.8 mg. Density measured by flotation at 295 K. Absorption corrections: Gaussian integration; maximum and minimum transmission factors, 0.848 and 0.767. Intensity measurements:  $\omega/2\theta$  step scan; measurements in octants,  $hkl$ ,  $h\bar{k}l$  and  $\bar{h}kl$ ;  $\sin \theta/\lambda \leq 0.66 \text{ \AA}^{-1}$ . Reflections recorded: 2039 total, 1320 independent ( $h-14 \rightarrow 13$ ,  $k-15 \rightarrow 15$ ,  $l 0 \rightarrow 21$ ); internal agreement factors on averaging the 619 reflections measured in duplicate,  $R=0.017$ ,  $wR=0.024$ . Three standards measured every 30 reflections, no significant variation in intensity with time. Step scans analyzed using *PROFILE* (Blessing, Coppens & Becker, 1974). Standard deviations assigned as  $\sigma(F^2) = [\sigma^2(F^2)_{\text{count}} + (0.03 F^2)^2]^{1/2}$  where  $\sigma(F^2)_{\text{count}}$  is based on counting statistics. Initial coordinates for the  $[\text{Co}(\text{C}_5\text{H}_5\text{NO})_6](\text{ClO}_4)_2$  molecule (16 independent atoms) taken from room-temperature

X-ray analysis (Bergendahl & Wood, 1975). Least-squares refinement: function minimized  $\sum w(F_o^2 - F_c^2)^2$  with weights  $w = [\sigma(F^2)]^{-2}$ . Observations used: 1309 reflections with  $F^2 \geq 0$ . Computer program: *LINEX74*, a modified version of *ORFLS* (Busing, Martin & Levy, 1962). Examination of difference maps at convergence (based on  $F_o - F_c$ ) showed significant density remaining in the center of the pyridine ring and at the midpoints of alternate bonds, that at the ring center being *ca* 6% of the peak height for N in the normal Fourier map. The disposition of these four residual peaks suggested that they belonged to a DMF molecule, coordinated to the Co atom, which had been incorporated into the complex during crystallization. Such a situation had been encountered previously in the analogous Cu complex (Keijzers, McMullan, Wood, van Kalker, Srinivasan & de Boer, 1982). The four extra atoms (three C and one N) of the partial DMF molecule were included in the refinement and their site occupancies (all constrained to the same value) varied in increments of 0.01, from 0.01 to 0.07, a general position having occupancy 1.0. Based on the behavior of the goodness-of-fit index, an occupancy of 0.05 seemed to be most appropriate, so that the formula for the complex in the crystal may be written as  $[\text{Co}(\text{C}_5\text{H}_5\text{NO})_{5.7}(\text{DMF})_{0.3}](\text{ClO}_4)_2$ . No attempt was made to include the O atom of the DMF molecule as an additional peak, the O of the pyridine  $N$ -oxide ligand being given an occupancy of 1.0. Refinement of the positional and isotropic thermal parameters for the partial DMF molecule with its occupancy fixed at 0.05 gave convergence with  $R(F^2)=0.034$ ,  $wR=0.041$  and  $S=1.18$ , a substantial improvement in the latter from the value of 2.94 obtained without the DMF molecule. Refinement was over a total of 144 variables and included an isotropic extinction parameter for a type I crystal with a Lorentzian distribution of mosaicity (Becker & Coppens, 1974). In final cycles, scattering length for Co refined. At convergence, all  $\Delta p_i \leq 0.05 \sigma(p_i)$  and the largest extinction correction was  $1.03 F_o^2$ . Neutron scattering lengths: Koester (1977); the value for Co refined to 2.47 (2) fm compared to the literature values of 2.78 (4) and 2.50 (5) fm. All calculations carried out on a CDC-Cyber 175 computer.

**Discussion.** The final atomic coordinates are given in Table 1,\* and *ORTEP* views (Johnson, 1965) illustrate the molecular packing of the  $[\text{Co}(\text{C}_5\text{H}_5\text{NO})_6]^{2+}$  complex ions and perchlorate ions (Fig. 1) and the relative orientations of the pyridine  $N$ -oxide ligand and

\* Lists of anisotropic thermal parameters and observed and calculated neutron structure amplitudes have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42661 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^5$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$ ) for  $[\text{Co}(\text{C}_5\text{H}_5\text{NO})_{5.7}\{(\text{CH}_3)_2\text{NCHO}\}_{0.3}](\text{ClO}_4)_2$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> <sub>eq</sub> <sup>*</sup>
Co	0	0	0	132 (18)
O(1)	15512 (9)	10836 (8)	6416 (5)	143 (5)
N(1)	20576 (5)	5231 (5)	10005 (5)	129 (3)
C(1)	27242 (8)	894 (8)	6508 (5)	164 (5)
C(2)	32546 (8)	−4992 (8)	10223 (5)	196 (5)
C(3)	31080 (8)	−6408 (8)	17557 (5)	204 (5)
C(4)	24296 (8)	−1722 (8)	20986 (5)	192 (5)
C(5)	19144 (7)	4108 (8)	17122 (4)	153 (5)
H(1)	27697 (20)	2237 (22)	860 (9)	361 (13)
H(2)	37699 (21)	−8509 (23)	7286 (11)	411 (13)
H(3)	34953 (22)	−11215 (22)	20499 (11)	429 (14)
H(4)	22738 (21)	−2545 (23)	26621 (11)	401 (14)
H(5)	13976 (18)	8134 (19)	19388 (11)	323 (12)
N(2)†	2514 (9)	−8 (9)	1304 (5)	13 (2)
C(6)†	1772 (12)	570 (13)	1279 (7)	23 (3)
C(7)†	2914 (13)	−223 (13)	1960 (8)	23 (3)
C(8)†	3050 (13)	−237 (13)	643 (8)	24 (4)
Cl	0	0	34968 (5)	162 (4)
O(2)	0	0	42580 (10)	279 (8)
O(3)	715 (11)	−10644 (11)	32414 (6)	296 (6)

<sup>\*</sup>  $U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i \cdot a_j a_j^*$ .  
† Atoms refined isotropically; coordinates  $\times 10^4$  and  $U_{iso} \text{\AA}^2 \times 10^3$ .

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) in  $[\text{Co}(\text{C}_5\text{H}_5\text{NO})_{0.7}\{(\text{CH}_3)_2\text{NCHO}\}_{0.3}](\text{ClO}_4)_2$

(i) $\text{Co}(\text{C}_5\text{H}_5\text{NO})$ moiety			
Co—O	2.083 (1)	O(1)—N(1)	1.324 (1)
N(1)—C(1)	1.353 (1)	N(1)—C(5)	1.350 (1)
C(1)—C(2)	1.384 (1)	C(4)—C(5)	1.380 (1)
C(2)—C(3)	1.392 (1)	C(3)—C(4)	1.389 (1)
C(1)—H(1)	1.073 (2)	C(5)—H(5)	1.072 (2)
C(2)—H(2)	1.082 (2)	C(4)—H(4)	1.074 (2)
C(3)—H(3)	1.080 (2)		
O(1)—Co—O(1) <sub>3</sub>	89.77 (4)*	O(1)—Co—O(1) <sub>3</sub>	90.23 (4)
Co—O(1)—N(1)	118.83 (6)		
O(1)—N(1)—C(1)	119.63 (7)	O(1)—N(1)—C(5)	119.27 (7)
N(1)—C(1)—C(2)	119.96 (8)	N(1)—C(5)—C(4)	120.42 (8)
C(1)—C(2)—C(3)	120.08 (8)	C(5)—C(4)—C(3)	120.00 (8)
C(2)—C(3)—C(4)	118.42 (8)	C(1)—N(1)—C(5)	121.09 (7)
N(1)—C(1)—H(1)	114.61 (12)	N(1)—C(5)—H(5)	115.16 (13)
C(2)—C(1)—H(1)	125.40 (13)	C(4)—C(5)—H(5)	124.41 (13)
C(1)—C(2)—H(2)	118.45 (15)	C(5)—C(4)—H(4)	117.59 (15)
C(3)—C(2)—H(2)	121.46 (15)	C(3)—C(4)—H(4)	122.40 (15)
C(2)—C(3)—H(3)	120.80 (16)	C(4)—C(3)—H(3)	120.77 (16)
(ii) Perchlorate ion			
O(2)—Cl	1.433 (2)	O(3)—Cl	1.440 (1)
O(2)—Cl—O(3)	109.51 (6)	O(3)—Cl—O(3) <sub>3</sub>	109.43 (9)
(iii) DMF molecule			
C(6)—O(1)	1.51 (2)	N(2)—C(6)	1.41 (2)
N(2)—C(7)	1.40 (2)	N(2)—C(8)	1.50 (2)
O(1)—C(6)—N(2)	124 (1)	C(6)—N(2)—C(7)	120 (1)
C(6)—N(2)—C(8)	122 (1)	C(7)—N(2)—C(8)	118 (1)
(iv) Least-squares plane for pyridine ring (atoms C <sub>5</sub> N)			
Plane: $0.8364X + 0.5342Y + 0.1227Z - 1.739 = 0$			
Distances of atoms from plane ( $\text{\AA} \times 10^3$ )			
N(1) −5 (3), C(1) 4 (3), C(2) 1 (3), C(3) −5 (3), C(4) 1 (3), C(5) 4 (3), O(1) −4, Co 1.739, H(1) −12, H(2) −7, H(3) −35, H(4) −11, H(5) 31			

\* Subscripts denote symmetry-related atoms.

the partial DMF molecule (Fig. 2). Bond distances and angles are given in Table 2. As noted in the *Experimental* section, partial replacement of pyridine *N*-oxide ligands by DMF solvent molecules has also been found for the analogous Cu complex (Keijzers *et al.*, 1982). For the latter complex, however, the O atom of the DMF molecule was also resolved, the substitution giving a tetragonally elongated geometry for the Cu atom. In the present instance, the  $\text{CoO}_6$  coordination unit does not appear to be distorted by the substitution, and the average Co—O distance of 2.083 (1)  $\text{\AA}$  compares with the room-temperature

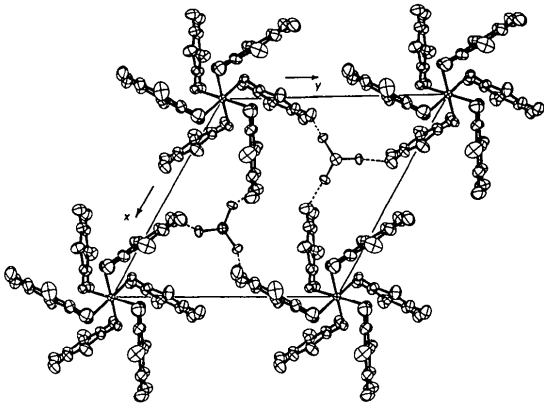


Fig. 1. Projection of one layer of the structure of  $[\text{Co}(\text{C}_5\text{H}_5\text{NO})_6](\text{ClO}_4)_2$  onto the (001) plane showing the molecular packing. Hydrogen bonds are indicated by broken lines. Ellipsoids are at the 67% probability level.

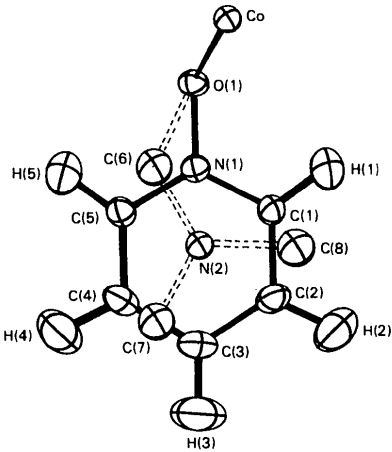


Fig. 2. A view normal to the pyridine *N*-oxide plane, showing the relative orientations of the partial DMF molecule and the ligand. Ellipsoids at the 67% probability level.

X-ray value of 2.088 (2) Å (Bergendahl & Wood, 1975). The nearly perfect octahedral geometry of the Co atom is maintained at low temperature, as is the orientation of the ligand, the Co atom being 1.727 Å from the ligand plane, compared to 1.723 Å at room temperature. In contrast to the majority of the structures where  $\text{ClO}_4^-$  features as a counterion, the geometry here is well defined and the present neutron results indicate that the absence of disorder, which is in fact found for the analogous complexes with  $\text{BF}_4^-$  as counterion (van Ingen Schenau, Verschoor & Romers, 1974), is probably attributable to hydrogen bonding between atom C(2) of the pyridine ring and O(3). The distance C(2)···O(3) of 3.011 (1) Å is appreciably shorter than the average of 3.23 Å expected for this type of hydrogen bond (Vinogradov & Linnell, 1971), while the H(2)···O(3) distance is 2.176 (2) Å. The angle at the H is 132.0 (2)°, indicating a markedly non-linear bond.

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## Hexachlorotellurate(IV) de Bis(triméthyl-2,4,6 pyridinium)

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**Abstract.**  $[\text{C}_8\text{H}_{12}\text{N}]_2[\text{TeCl}_6]$ ,  $M_r = 584.7$ , triclinic,  $P\bar{1}$ ,  $a = 7.962$  (4),  $b = 9.378$  (4),  $c = 9.569$  (5) Å,  $\alpha = 99.78$  (5),  $\beta = 109.25$  (6),  $\gamma = 110.83$  (6)°,  $V = 596$  Å<sup>3</sup>,  $Z = 1$ ,  $D_x = 1.628$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.7107$  Å,  $\mu = 1.93$  mm<sup>-1</sup>,  $F(000) = 288$ ,  $T = 296$  (1) K,  $R = 0.044$  for 2161 independent reflections. Te is surrounded by six Cl atoms in a regular octahedron. The pyridinium bond lengths and angles are normal. The structure can be regarded as constituted of layers of  $[\text{TeCl}_6]^{2-}$  anions separated by double layers of  $[\text{C}_8\text{H}_{12}\text{N}]^+$  cations parallel to the (100) face. A hydrogen bond N—H···Cl takes part in the cohesion of the structure.

**Introduction.** Le présent travail fait partie d'une étude des composés formés par les acides hexachlorotellurique(IV) et hexachloroplatinique(IV) avec les amines aromatiques. Le produit utilisé a été préparé à la température ambiante en ajoutant, en quantité stoechiométrique, la collidine base à une solution d'acide hexachlorotellurique obtenue en dissolvant le dioxyde de tellure dans l'acide chlorhydrique concentré en excès. L'évaporation lente de la solution donne des cristaux parallélépipédiques, de couleur jaune, qui s'hydrolysent en quelques jours lorsqu'on les abandonne à l'air ambiant. Leur composition a été établie par l'étude structurale.