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SHORT-FORMAT PAPERS

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Acta Cryst. (1987). C**43**, 1209–1211

Structure of *trans*-Dicyanobis(ethylenediamine)cobalt(III) Perchlorate

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Abstract. *trans*-$[\mathrm{Co(CN)_2(C_2H_8N_2)_2}]^+.\mathrm{ClO_4^-}$, $M_r = 330{\cdot}6$, monoclinic, $C2/c$, $a = 17{\cdot}556\,(3)$, $b = 6{\cdot}930\,(1)$, $c = 10{\cdot}604\,(2)$ Å, $\beta = 93{\cdot}07\,(1)°$, $V = 1288{\cdot}3\,(4)$ Å^3, $Z = 4$, $D_m = 1{\cdot}706$, $D_x = 1{\cdot}705$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}61$ mm^{-1}, $F(000) = 680$, room temperature, $R = 0{\cdot}037$ for 2433 observed reflections and 116 refined parameters. The cation reveals the point symmetry $\bar{1}$ (C_i) with the Co atom in the special position. The coordination of the Co atom is slightly distorted from an ideal octahedron; the axis formed by the two cyano groups is almost parallel to the c axis. The anion is located on a twofold axis (C_2) and one O atom shows a tendency to disorder while the other one forms a weak N—H···O hydrogen bridge.

Experimental. Spectroscopic investigations and the preparation of the compound under study have been reported by Hakamata, Urushiyama, Degen, Kupka & Schmidtke (1983). Well shaped yellow crystals have been crystallized by the same authors. Photographs around directions [011] (Weissenberg) and [100] (Buerger–precession) showed monoclinic symmetry and systematic absences of the space group Cc or $C2/c$ [fully reduced mesh, cell choice 1, *International Tables for Crystallography* (1983)]; the latter has been confirmed by the structure determination. Density, determined by flotation, yielded $Z = 4$ formula units per cell. A crystal shaped as a parallelogram of approx. $0{\cdot}5 \times 0{\cdot}4 \times 0{\cdot}2$ mm has been used for the determination of the lattice constants (15 reflections, $56 < 2\theta < 60°$, Mo $K\alpha$, Syntex $P2_1$, crystal monochromator) as well as for the data collection. The intensities of all 2840 symmetry-independent reflections up to $(\sin\theta)/\lambda = 0{\cdot}704$ Å^{-1} have been measured with an ω scan of 0·7° and variable speeds between 0·7 and 29·3° min^{-1}. The indices ranged from 0 to $h = 28$, $k = 11$ and from $l = -17$ to 16. Three standard reflections ($3\bar{3}7$, $5\bar{5}3$, 337) measured every 100 reflections varied only within experimental errors. Six reflections with too high intensities and at a later stage five weak reflections with $F_o \gg F_c$ showing asymmetric

Table 1. *Atomic positions and equivalent isotropic thermal parameters* ($\times 100$ Å^2) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}(U_{11}a^{*2}a^2 + U_{12}a^*b^*ab\cos\gamma + \ldots).$$

	x	y	z	U_{eq}
Co	0·25	0·25	0·5	2·32 (1)
Cl	0·0	0·0700 (1)	0·25	4·29 (1)
O(1)	−0·0604 (1)	−0·0479 (3)	0·1962 (2)	7·00 (8)
O(2)	0·0298 (2)	0·1762 (8)	0·1530 (4)	12·98 (15)
N(1)	0·3522 (1)	0·3494 (2)	0·4672 (1)	3·42 (4)
N(2)	0·2982 (1)	−0·0050 (2)	0·5078 (1)	3·22 (4)
N(3)	0·2222 (1)	0·2159 (2)	0·2127 (1)	3·84 (4)
C(1)	0·4016 (1)	0·1843 (4)	0·4349 (2)	4·46 (5)
C(2)	0·3824 (1)	0·0195 (4)	0·5221 (2)	4·58 (6)
C(3)	0·2308 (1)	0·2262 (2)	0·3210 (1)	2·72 (4)

Table 2. *Bond angles* (°)

Angles within the cation (point symmetry $\bar{1}$); the e.s.d.'s are 0·1 at Co and N, 0·2° at C atoms

N(1)—Co—N(2)	85·8	N(1)—C(1)—C(2)	106·7
N(1)—Co—C(3)	88·2	C(1)—C(2)—N(2)	106·0
N(2)—Co—C(3)	91·0	C(2)—N(2)—Co	109·1
Co—N(1)—C(1)	108·7	Co—C(3)—N(3)	177·1

Angles within the anion (point symmetry 2); the e.s.d.'s are 0·2°; symmetry code: (i) $-x, y, 0{\cdot}5-z$

O(1)—Cl—O(1^i)	110·5	O(1)—Cl—O(2^i)	107·3
O(1)—Cl—O(2)	107·9	O(2)—Cl—O(2^i)	116·1

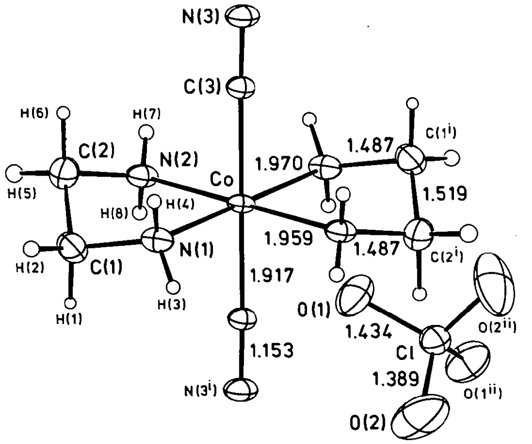

Fig. 1. Cation (point symmetry $\bar{1}$) and anion (point symmetry 2) of the title compound in arbitrary crystallographic orientation with bond lengths in Å. The e.s.d.'s are: Co—N,C 0·002, C—N 0·003 and Cl—O 0·004 Å. Thermal ellipsoids of 30% probability (*ORTEP*II, Johnson, 1976) are shown; the isotropic U values of the H atoms are reduced to 1/5. Codes of symmetry-related atoms (i): $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$; (ii): $-x, y, \frac{1}{2}-z$.

peaks were eliminated; the final set of data contained 2433 observed ($I > 1{\cdot}96\sigma_I$) out of 2829 reflections. No absorption correction was applied.

Initial coordinates of the Co and Cl atoms were obtained from the Patterson function; the structure was completed and refined in the usual way. All H atoms were located in a difference electron density map and were included with isotropic B values in the refinement on F (116 parameters) converging at $S = 1{\cdot}93$ with all $\Delta/\sigma < 0{\cdot}01$. Weights were derived from $1/w = \sigma_F^2 + (0{\cdot}02F_o)^2$ and yielded $R = 0{\cdot}037$ (0·044) and $wR = 0{\cdot}054$ (0·056) for the observed (all) data, scattering factors from Cromer & Waber (1974), corrections for anomalous dispersion of Co and Cl atoms from Cromer & Liberman (1974). Residual electron density ranged from −0·53 to +0·54 e Å^{-3}. The final parameters of the non-H atoms are listed in Table 1.* The anisotropic displacement parameters of the atom O(2) range up to $U_{ii} = 0{\cdot}18$ Å^2 and indicate a tendency to disorder (*cf.* Fig. 1) within the perchlorate ion. All calculations were performed with a local modified version of the program system *EXTL* (Syntex).

Both ions are displayed in Fig. 1 with main bond lengths; relevant bond angles are given in Table 2. The geometry of the H atoms is in the expected range for X-ray data with averaged bond lengths N—H 0·85 (3), C—H 0·97 (3) Å and angles deviating up to 10° from 109°. The axis defined by the N atoms of the two cyano groups of Table 1 forms angles of 80·8, 85·6, and 10·2° with the crystallographic *a*, *b*, and *c* axes. The five-membered ring is puckered with torsion angles between −41 and 53°. Both ions are linked by a weak hydrogen bridge: N(1^i)—H(4^i)···O(1) with distances N···O 3·122 (3), H···O 2·31 (3) Å, and an angle N—H···O 169 (3)° [symmetry code: (i) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$]. This reduces the possible disorder of the atom O(1) of the anion whilst the disorder of O(2) is compensated by enlarged displacement parameters ($U_{22} = 0{\cdot}18$ Å^2).

Related literature. Crystal structures of identical or comparable cations have been reported: *trans*-dicyanobis(ethylenediamine)cobalt(III) chloride (Okamoto, Matsumoto & Kuroya, 1970) and *trans*-dichlorobis(ethylenediamine)cobalt(III) perchlorate (Niederhoffer, Peascoe, Rudolf, Clearfield & Martell, 1986).

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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 43728 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Dichlorobis(*N*-isopropylideneaniline)palladium(II)

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Abstract. $[PdCl_2(C_9H_{11}N)_2]$, $M_r = 443{\cdot}7$, monoclinic, $P2_1/n$, $a = 8{\cdot}5651\ (3)$, $b = 12{\cdot}0982\ (5)$, $c = 9{\cdot}2466\ (4)$ Å, $\beta = 93{\cdot}916\ (5)°$, $V = 955{\cdot}92$ Å^3, $Z = 2$, $D_x = 1{\cdot}541$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71073$ Å, $\mu = 1{\cdot}24$ mm^{-1}, $F(000) = 448$, $T = 293$ K, $R = 0{\cdot}023$ for 1421 unique observed reflections. Pd, on an inversion centre, is square-planar coordinated by two *trans*-Cl and two *trans*-imine ligands: Pd—Cl = 2·303 (1), Pd—N = 2·024 (2) Å, N—Pd—Cl = 89·9 (1)°, N=C = 1·273 (4) Å. The ligand plane [$CN{=}CC_2$, r.m.s. $\Delta = 0{\cdot}014\ (2)$ Å] is essentially perpendicular to the coordination plane [$PdCl_2N_2$, exactly planar; dihedral angle between planes =89·9 (1)°] and to the plane of the phenyl substituent [C_6, r.m.s. $\Delta = 0{\cdot}006\ (2)$ Å; dihedral angle = 91·4 (1)°].

Experimental. K_2PdCl_4 (1·25 mmol) reacts with diphenylformamidine (1·25 mmol) in a 2:5 methanol/water mixture (70 ml) at the reflux temperature to produce a yellow powder identified by elemental analysis as $[(C_6H_4NHCHNC_6H_5)PdCl]_n$. Attempts to grow crystals of this very sparingly soluble complex from acetone over three months under a nitrogen atmosphere at room temperature produced yellow crystals of the present complex in small yield; palladium metal, free *N*-(isopropylidene)aniline and N-phenylformamide were also formed. The reaction may be rationalized in terms of initial hydrolysis of the formamidino group by traces of water in the acetone, causing cleavage of the C=N bond to form coordinated formamide and aniline ligands. The latter would undergo a standard condensation reaction with acetone to form *N*-(isopropylidine)aniline.

Crystal size 0·2 × 0·25 × 0·4 mm, Siemens AED2 diffractometer, cell parameters from 2θ values of 32 reflections ($20 < 2\theta < 25°$). Intensity measurements in ω/θ scan mode, scan width 0·51° below α_1 to 0·51° above α_2, scan time = 14–56 s, $2\theta_{max} = 50°$, h −10→0, k 0→14, l −10→10, no significant variation in three standard reflections, semi-empirical absorption correction, transmission 0·577–0·615, extinction insignificant. 1680 reflections measured, all unique, 1421 with $F < 4\sigma(F)$. Patterson synthesis confirms Pd on centre of symmetry and locates Cl, other atoms from difference syntheses, blocked-cascade refinement on F, $w = 1/\sigma^2(F)$, anisotropic thermal parameters for all non-H atoms, H atoms constrained [C—H = 0·96 Å, H—C—H = 109·5° in rigid methyl groups, aromatic H on ring angle external bisectors, $U(H) = 1{\cdot}2U_{eq}(C)$]. 113 parameters, $R = 0{\cdot}023$, $wR = 0{\cdot}026$, max. $\Delta/\sigma = 0{\cdot}056$, mean = 0·016, slope of normal probability plot = 1·78, max $\Delta\rho = +0{\cdot}37$, min. = $-0{\cdot}23$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* programs (Sheldrick, 1985).

Fig. 1 gives a view of the molecule with atomic numbering scheme. Table 1 gives the atomic co-

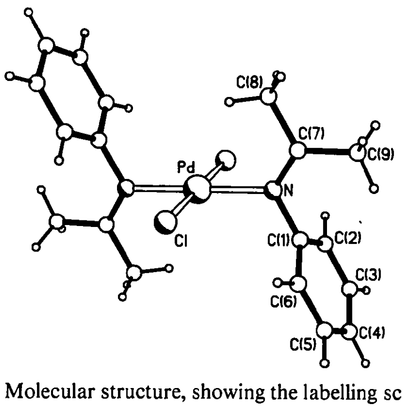

Fig. 1. Molecular structure, showing the labelling scheme.