

direct methods using *SHELX* (Sheldrick, 1981). Full-matrix refinement was carried out, minimizing $\sum w(F_o - F_c)^2$.

H-atom positions were located by difference Fourier synthesis. In the final stages of refinement, C—H bond lengths and H—C—H angles were fixed at 0.96 Å and 109.5°. A fixed isotropic thermal parameter of 0.06 Å² was used for all H atoms. All non-H atoms were refined anisotropically. 298 parameters were refined. Final $wR = 0.0595$, $R = 0.0699$, with $w^{-1} = \sigma^2(F) + 0.0003F^2$. In the final difference Fourier map, the maximum and minimum peaks were 0.83 and $-0.92 e \text{ Å}^{-3}$, respectively. Max. $\Delta/\sigma = 0.006$. Mean $\Delta/\sigma = 0.001$. Scattering factors and f' , f'' were supplied by the computer programs.

Final atomic coordinates for all non-H atoms are presented in Table 1, with derived bond lengths and angles in Table 2.* A thermal ellipsoid (50%)

* 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 54063 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

drawing containing the numbering scheme employed is shown in Fig. 1.

Related literature. Diorganotin carboxylates are industrially important as PVC stabilizers (Davis & Smith, 1982), and as catalysts for transesterification reactions, polyurethane polymerizations (Evans & Karpel, 1985), and moisture-curable silicone sealants (Gross, 1989). The compound described here has more sterically demanding carboxylate ligands than the previously reported $\text{Me}_2\text{Sn}(\text{OAc})_2$ (Lockhart, Calabrese & Davidson, 1987).

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Acta Cryst. (1991). **C47**, 1971–1973

Structure of Bis(2,3-butanedione dioximato)[(*R,S*)-1-cyanoethyl](dimethylphenylphosphine)cobalt(III)

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(Received 29 October 1990; accepted 28 February 1991)

Abstract. $[\text{Co}(\text{C}_4\text{H}_7\text{N}_2\text{O}_2)_2(\text{C}_3\text{H}_4\text{N})\{\text{P}(\text{CH}_3)_2(\text{C}_6\text{H}_5)\}]$, $M_r = 499.39$, monoclinic, $P2_1/a$, $a = 12.972$ (4), $b = 16.019$ (7), $c = 11.438$ (5) Å, $\beta = 97.19$ (3)°, $V = 2358$ (1) Å³, $Z = 4$, $D_x = 1.407$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 8.61$ cm⁻¹, $F(000) = 1048$, $T = 298$ K, $R = 0.081$ for 1489 observed reflections. The *R* and *S* cyanoethyl groups have a disordered structure at the same position and a solvent water molecule links the neighboring molecules. This structure is different from the X-ray-induced racemic crystal in the crystalline state reaction.

Experimental. Recrystallized from an aqueous methanol solution. Red brown crystal, $0.35 \times 0.30 \times$

0.20 mm. Rigaku AFC5R diffractometer, graphite-monochromated Mo $K\alpha$ radiation, cell parameters using 25 reflections in the range $20 < 2\theta < 30^\circ$, intensity measurement $2\theta \leq 45^\circ$; range of hkl : -16 to 16 , -20 to 0 , 0 to 14 ; ω - 2θ scan, scan speed $16^\circ \text{ min}^{-1}$ (2θ), scan range $(1.2 + 0.35 \tan \theta)^\circ$; intensities of three standard reflections monitored every 150 reflections with only small random variations, 3385 reflections measured, 1489 observed with $|F_o| < 5\sigma(F_o)$. Intensity data corrected for Lorentz and polarization, empirical absorption correction based on ψ scans, normalized transmission factors 1.00 – 0.88 . Structure solved by direct methods with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson,

Table 1. Final atomic coordinates ($\times 10^4$, C10B $\times 10^3$) and equivalent isotropic thermal parameters (\AA^2)

$$B_{eq} = (8\pi^2/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	B_{eq}/B
Co	2200 (2)	700 (2)	2117 (2)	3.3
P	3528 (3)	1615 (3)	2658 (3)	3.1
O1	804 (10)	1909 (8)	2829 (10)	5.3
O2	3322 (10)	-556 (8)	3511 (11)	5.3
O3	3623 (9)	-486 (8)	1421 (10)	5.2
O4	1102 (9)	1963 (9)	749 (9)	5.2
O5	5024 (11)	1715 (9)	-560 (11)	7.5
N1	1394 (10)	1240 (10)	3127 (11)	3.8
N2	2613 (11)	52 (9)	3470 (12)	4.1
N3	2989 (10)	167 (9)	1060 (12)	3.8
N4	1748 (10)	1342 (10)	761 (11)	3.9
N5	-672 (15)	249 (15)	2226 (22)	10.4
C1	1436 (12)	935 (11)	4175 (14)	3.7
C2	2135 (12)	251 (12)	4396 (14)	3.7
C3	2866 (13)	448 (12)	-40 (13)	3.9
C4	2150 (14)	1148 (12)	-186 (13)	3.7
C5	809 (17)	1255 (15)	5121 (16)	5.9
C6	2438 (18)	-160 (13)	5603 (16)	6.5
C7	3385 (17)	45 (17)	-951 (16)	6.2
C8	1920 (17)	1607 (14)	-1327 (15)	5.9
C9	1036 (16)	-158 (16)	1528 (20)	8.0
C10A†	1181 (24)	-961 (18)	1069 (21)	5.5
C11	120 (18)	56 (15)	1923 (15)	6.2
C12	4774 (15)	1225 (14)	2366 (18)	5.0
C13	3394 (17)	2628 (11)	1937 (16)	5.1
C14	3684 (11)	1877 (11)	4216 (12)	3.2
C15	3063 (14)	2463 (11)	4644 (16)	4.6
C16	3206 (18)	2647 (14)	5849 (17)	6.3
C17	3875 (16)	2259 (14)	6624 (15)	5.3
C18	4489 (16)	1661 (13)	6184 (16)	7.0
C19	4419 (14)	1482 (12)	5006 (15)	5.0
C10B‡	122 (8)	-105 (8)	176 (14)	6.1 (8)

† Occupancy factor 0.68.

‡ Occupancy factor 0.32.

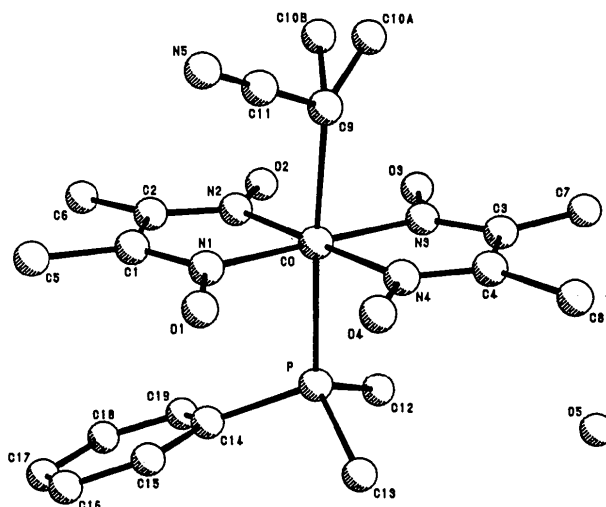
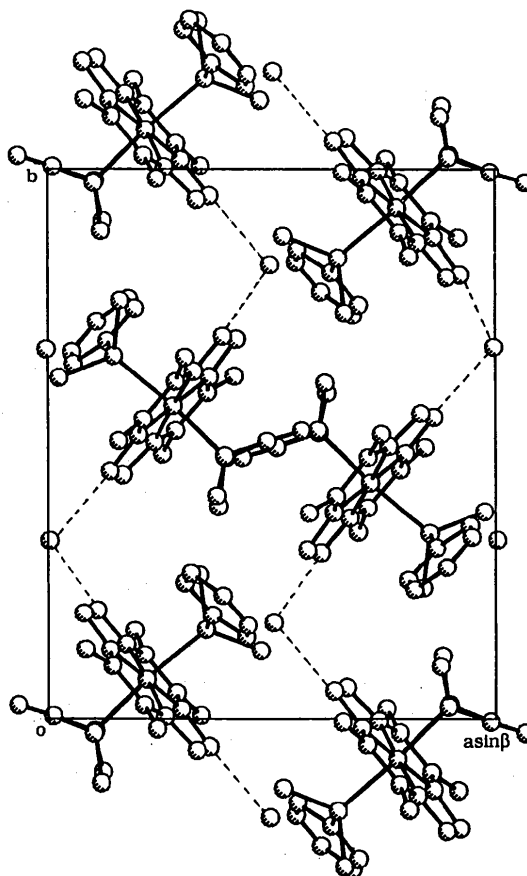


Fig. 1. PLUTO (Motherwell & Clegg, 1978) drawing of the molecules along with crystallographic numbering.

Table 2. Bond distances (\AA) and angles ($^\circ$)

Co—P	2.288 (5)	N5—C11	1.17 (3)
Co—N1	1.87 (1)	C1—C2	1.42 (2)
Co—N2	1.88 (1)	C1—C5	1.52 (3)
Co—N3	1.88 (1)	C2—C6	1.54 (2)
Co—N4	1.89 (1)	C3—C4	1.45 (3)
Co—O9	2.09 (2)	C3—C7	1.46 (3)
P—C12	1.80 (2)	C4—C8	1.50 (2)
P—C13	1.82 (2)	C9—C10A	1.41 (4)
P—C14	1.82 (1)	C9—C10B	1.49 (10)
O1—N1	1.33 (2)	C9—C11	1.37 (3)
O2—N2	1.34 (2)	C14—C15	1.37 (2)
O3—N3	1.36 (2)	C14—C19	1.38 (2)
O4—N4	1.30 (2)	C15—C16	1.40 (3)
N1—C1	1.29 (2)	C16—C17	1.37 (3)
N2—C2	1.33 (2)	C17—C18	1.38 (3)
N3—C3	1.33 (2)	C18—C19	1.37 (3)
N4—C4	1.30 (2)		
P—Co—N1	89.8 (4)	Co—N4—O4	124 (1)
P—Co—N2	90.2 (4)	Co—N4—C4	116 (1)
P—Co—N3	90.7 (4)	O4—N4—C4	120 (1)
P—Co—N4	91.1 (4)	N1—C1—C2	114 (2)
P—Co—O9	176.2 (7)	N1—C1—C5	125 (2)
N1—Co—N2	82.6 (6)	C2—C1—C5	121 (2)
N1—Co—N3	178.4 (6)	N2—C2—C1	113 (1)
N1—Co—N4	96.6 (6)	N2—C2—C6	121 (2)
N1—Co—O9	93.8 (8)	C1—C2—C6	125 (2)
N2—Co—N3	99.0 (6)	N3—C3—C4	112 (1)
N2—Co—N4	178.5 (6)	N3—C3—C7	121 (2)
N2—Co—O9	91.3 (8)	C4—C3—C7	127 (2)
N3—Co—N4	81.8 (6)	N4—C4—C3	114 (1)
N3—Co—O9	85.7 (8)	N4—C4—C8	124 (2)
N4—Co—O9	87.5 (8)	C3—C4—C8	122 (2)
Co—P—C12	112.9 (7)	Co—O9—C10A	127 (2)
Co—P—C13	115.0 (7)	Co—O9—C10B	119 (4)
Co—P—C14	113.6 (5)	Co—O9—C11	111 (2)
C12—P—C13	105 (1)	C10A—O9—C11	121 (2)
C12—P—C14	105.6 (8)	C10B—O9—C11	108 (5)
C13—P—C14	103.4 (8)	N5—C11—O9	178 (2)
Co—N1—O1	124 (1)	P—C14—C15	121 (1)
Co—N1—C1	116 (1)	P—C14—C19	121 (1)
O1—N1—C1	120 (1)	C15—C14—C19	118 (1)
Co—N2—O2	124 (1)	C14—C15—C16	119 (2)
Co—N2—C2	114 (1)	C15—C16—C17	122 (2)
O2—N2—C2	122 (1)	C16—C17—C18	118 (2)
Co—N3—O3	121 (1)	C17—C18—C19	122 (2)
Co—N3—C3	116 (1)	C14—C19—C18	120 (2)
O3—N3—C3	123 (1)		

Fig. 2. The crystal structure viewed along the c axis. Hydrogen bonds are indicated by dotted lines.

1978). Full-matrix least squares with *SHELX76* (Sheldrick, 1976); refinement on *F*; anisotropic thermal parameters for non-H atoms except for the disordered C atom of minor component, C10B, which refined isotropically. Occupancy factors of the disordered atoms refined to 0.68 (C10A) and 0.32 (C10B). H atoms placed and fixed in calculated positions. Final agreement parameters: $R = 0.081$, $wR = 0.082$, $w = [\sigma^2(F_o) + 0.001F_o^2]^{-1}$, $(\Delta/\sigma)_{\max} = 0.17$, $\Delta\rho_{\max} = 0.71 \text{ e } \text{Å}^{-3}$ around the Co atom. Atomic scattering factors including anomalous-dispersion term from *International Tables for X-ray Crystallography* (1974, Vol. IV). The somewhat large *R* value would be caused by the large thermal vibration and the disordered structure. All calculations were performed on a MicroVAX II computer.

Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1,* bond distances and angles in Table 2. Fig. 1 shows the labelled

* Lists of structure factors, anisotropic thermal parameters for non-H atoms, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54050 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

molecular structure, and Fig. 2 illustrates the crystal packing.

Related literature. Crystalline state racemization of the cyanoethyl groups has been reported (Ohashi, 1988). The structure of the present complex with a chiral cyanoethyl group has been published (Kojima, Iwasaki, Ohashi, Baba & Ohgo, 1991). The racemic structure produced by X-ray irradiation was compared with the title compound (Uchida, Kojima, Sekine, Ohashi, Ohgo & Baba, 1990).

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Acta Cryst. (1991). **C47**, 1973–1975

Hexamethylethylenediammonium Hexachlorodicuprate(II)

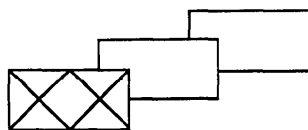
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(Received 2 July 1990; accepted 11 March 1991)

Abstract. $[\text{C}_8\text{H}_{22}\text{N}_2]^{2+} \cdot [\text{Cu}_2\text{Cl}_6]^{2-}$, $M_r = 486.1$, triclinic, $P\bar{1}$, $a = 6.254$ (1), $b = 8.269$ (2), $c = 9.753$ (2) Å, $\alpha = 103.96$ (2), $\beta = 103.67$ (2), $\gamma = 105.80$ (2)°, $V = 445.7$ (2) Å³, $Z = 1$, $D_x = 1.81 \text{ Mg m}^{-3}$, $\text{Cu } K\alpha$, $\lambda = 1.54182$ Å, $\mu = 11.32 \text{ mm}^{-1}$, $F(000) = 244$, $T = 295 \text{ K}$, $R = 0.049$ for 889 unique observed [$F \geq 3\sigma(F)$] reflections and 85 parameters. The structure consists of separate organic dication and stacks, (I), of quasi-planar

$\text{Cu}_2\text{Cl}_6^{2-}$ anions. The stacking arrangement is such that each Cu^{II} ion assumes a 4 + 1 coordination geometry. Within the dimer, terminal Cu—Cl distances average 2.264 Å and bridging distances average 2.318 Å while the interdimer semicoordinate Cu...Cl distance is 2.665 (1) Å. The bifold angle between the central Cu_2Cl_2 core and the folded CuCl_3 flap of the dimer is 19.1°.



(I)

Experimental. In an attempt to extend the study of the dynamic Jahn–Teller effect in ACuCl_3 salts containing tribridged chains we have sought to prepare analogs of $(\text{CH}_3)_4\text{NCuCl}_3$ (Weenk & Spek, 1976; Willett, Bond, Haije, Soonieus & Maaskant, 1988). One cation selected was the $\text{Me}_6\text{en}^{2+}$ dication