

amount to 1.907 (7) and 1.897 (7) Å, respectively. This can be ascribed to the large *trans* influence exerted by C(107).

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## Structure of Bis(2,3-butanedione dioximato)[(*S*)-1-cyanoethyl]- (dimethylphenylphosphine)cobalt(III) at 298 and 223 K

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**Abstract.** [Co(C<sub>4</sub>H<sub>7</sub>N<sub>2</sub>O<sub>2</sub>)<sub>2</sub>(C<sub>3</sub>H<sub>4</sub>N){P(CH<sub>3</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)}]<sub>2</sub>, *M<sub>r</sub>* = 481.37, monoclinic, *P*2<sub>1</sub>; *T* = 298 K, *a* = 9.5074 (9), *b* = 14.265 (1), *c* = 8.5947 (8) Å, β = 101.71 (1)°, *V* = 1141.37 (2) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.40 g cm<sup>-3</sup>, *F*(000) = 574, μ(Mo *K*α) = 9.02 cm<sup>-1</sup>, λ = 0.71069 Å, *R* = 0.053 for 2570 observed reflections; *T* = 223 K, *a* = 9.486 (4), *b* = 14.068 (3), *c* = 8.808 (3) Å, β = 101.27 (3)°, *V* = 1126.5 (6) Å<sup>3</sup>, *Z* = 2, *D<sub>x</sub>* = 1.42 g cm<sup>-3</sup>, μ(Mo *K*α) = 9.08 cm<sup>-1</sup>, *R* = 0.027 for 1987 observed reflections. The crystal shows crystalline-state racemization at 298 K. During the early stages of data collection, about 30% of the chiral cyanoethyl group was inverted. The initial structure was obtained at 223 K, since the rate of the racemization became sufficiently low.

**Introduction.** It has been reported that the chiral 1-cyanoethyl (cn) group in some crystals of the bis(dimethylglyoximato)cobalt(III) (dimethylglyoximato = 2,3-butanedione dioximato) (cobaloxime)

complex is racemized by X-ray exposure keeping the single-crystal form (Ohashi, 1988). The process of racemization has been found to be divided into three modes (Tomotake, Uchida, Ohashi, Sasada, Ohgo & Baba, 1985), of which the first mode is the simplest, since the crystal has only one molecule in its asymmetric unit so that the reactive cn group is separated from the other cn groups in the crystalline lattice. Three crystals have been observed to proceed *via* the first mode: [(*R*)-1-cyanoethyl][(*S*)-α-methylbenzylamine]cobaloxime, *R*-cn-*S*-mba (Ohashi, Yanagi, Kurihara, Sasada & Ohgo, 1981), [(*S*)-1-cyanoethyl][(*S*)-α-methylbenzylamine]cobaloxime, *S*-cn-*S*-mba (Ohashi, Sasada & Ohgo, 1978) and [(*R*)-1-cyanoethyl](pyrrolidine)cobaloxime, *R*-cn-pyrr (Tamura, 1986). The present crystal, *S*-cn-dmp, also belongs to the first mode. The racemization rate was so high that the chiral cn group was racemized during data collection. Therefore, the initial structure was obtained at 223 K. The present paper reports the

Table 1. Final atomic coordinates ( $\times 10^4$  for 298 K) and equivalent isotropic thermal parameters
$$B_{eq} = (1/3) \sum_i \sum_j B_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	$B_{eq}(\text{\AA}^2)$
298 K				
Co	2430 (1)	2731	9402 (1)	2.9
P	1547 (2)	1647 (1)	7471 (2)	3.2
N(1)	2098 (7)	3679 (4)	7849 (7)	4.1
N(2)	537 (5)	3027 (4)	9521 (6)	3.5
N(3)	2791 (5)	1806 (4)	10992 (6)	3.1
N(4)	4326 (5)	2422 (4)	9259 (6)	3.6
O(1)	3084 (6)	3940 (4)	7026 (6)	5.3
O(2)	-193 (4)	2558 (4)	10496 (5)	4.1
O(3)	1800 (4)	1534 (4)	11814 (5)	4.0
O(4)	5007 (4)	2823 (5)	8189 (5)	4.9
C(1)	845 (8)	4095 (5)	7623 (8)	4.3
C(2)	-87 (6)	3693 (4)	8606 (7)	3.7
C(3)	4061 (6)	1431 (5)	11283 (7)	3.5
C(4)	4976 (6)	1799 (5)	10246 (8)	3.8
C(5)	414 (10)	4866 (6)	6471 (11)	6.3
C(6)	-1619 (7)	3978 (6)	8496 (10)	5.5
C(7)	4542 (8)	703 (6)	12528 (9)	4.9
C(8)	6473 (6)	1481 (7)	10253 (10)	5.5
C(9)	3082 (9)	3792 (7)	11057 (11)	5.1
C(10A)	2754 (14)	3634 (9)	12703 (12)	5.2
C(11)	4524 (7)	4061 (5)	11106 (8)	4.3
N(5)	5660 (8)	4311 (7)	11154 (10)	7.5
C(12)	2744 (8)	1381 (7)	6132 (10)	5.6
C(13)	1156 (8)	499 (5)	8233 (9)	5.0
C(14)	-127 (6)	1989 (4)	6191 (7)	3.4
C(15)	-133 (8)	2498 (5)	4792 (8)	4.6
C(16)	-1432 (9)	2797 (8)	3853 (8)	5.7
C(17)	-2697 (8)	2597 (9)	4314 (9)	6.0
C(18)	-2704 (7)	2096 (8)	5683 (10)	5.9
C(19)	-1422 (7)	1793 (6)	6611 (8)	4.5
C(10B)	229 (5)	410 (3)	1217 (5)	7.1 (9)*
223 K				
Co	0.24103 (4)	0.2724	-0.06245 (5)	1.89
P	0.1524 (1)	0.16089 (7)	-0.2514 (1)	2.18
O(1)	0.3080 (3)	0.3916 (2)	-0.3040 (3)	3.6
O(2)	-0.0217 (3)	0.2563 (2)	0.0481 (3)	2.7
O(3)	0.1770 (3)	0.1533 (2)	0.1825 (3)	2.6
O(4)	0.4996 (2)	0.2798 (3)	-0.1849 (3)	3.3
N(1)	0.2082 (4)	0.3666 (2)	-0.2203 (4)	2.6
N(2)	0.0515 (3)	0.3037 (2)	-0.0490 (3)	2.2
N(3)	0.2768 (3)	0.1798 (2)	0.0979 (4)	2.1
N(4)	0.4309 (3)	0.2409 (2)	-0.0768 (3)	2.3
N(5)	0.5702 (4)	0.4263 (3)	0.1173 (5)	5.3
C(1)	0.0838 (4)	0.4090 (3)	-0.2428 (4)	2.7
C(2)	-0.0111 (4)	0.3695 (3)	-0.1438 (4)	2.6
C(3)	0.4047 (4)	0.1420 (3)	0.1285 (4)	2.2
C(4)	0.4959 (4)	0.1782 (3)	0.0226 (4)	2.4
C(5)	0.0424 (7)	0.4871 (4)	-0.3588 (7)	4.3
C(6)	-0.1638 (5)	0.3989 (3)	-0.1579 (6)	3.6
C(7)	0.4519 (5)	0.0695 (3)	0.2544 (6)	3.3
C(8)	0.6465 (4)	0.1454 (4)	0.0240 (6)	3.6
C(9)	0.3049 (5)	0.3802 (3)	0.1012 (6)	2.7
C(10)	0.2711 (7)	0.3675 (4)	0.2640 (6)	4.5
C(11)	0.4526 (4)	0.4054 (3)	0.1087 (5)	3.0
C(12)	0.2742 (5)	0.1317 (4)	-0.3828 (6)	3.8
C(13)	0.1110 (5)	0.0469 (3)	-0.1727 (5)	3.2
C(14)	-0.0150 (4)	0.1958 (3)	-0.3815 (4)	2.2
C(15)	-0.0144 (5)	0.2462 (3)	-0.5196 (4)	3.1
C(16)	-0.1416 (5)	0.2763 (4)	-0.6136 (4)	3.9
C(17)	-0.2710 (4)	0.2574 (4)	-0.5697 (5)	4.1
C(18)	-0.2721 (4)	0.2070 (4)	-0.4318 (5)	4.0
C(19)	-0.1459 (4)	0.1761 (3)	-0.3387 (4)	3.1

\* Isotropic thermal factor.

process of racemization of *S*-cn-dmp, and the reason why such a high reaction rate is observed for this crystal.

**Experimental.** *R*-cn-dmp was prepared in a similar way to that reported previously (Ohgo, Takeuchi, Natori, Yoshimura, Ohashi & Sasada, 1981). Orange plate-like crystals obtained from an aqueous metha-

nol solution. Systematic absences  $0k0$  for  $k = 2n + 1$ . At 298 K: crystal dimensions  $0.4 \times 0.2 \times 0.3$  mm, Rigaku AFC-4 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation, cell parameters refined by least-squares methods on the basis of 18 reflections in the range  $20 < 2\theta < 30^\circ$ , some change in cell dimensions observed during data collection, the mean values before and after data collection were adopted, intensity measurement performed up to  $2\theta = 55^\circ$ , range of  $hkl$ :  $-12$  to  $12$ ,  $0$  to  $18$ ,  $0$  to  $11$ ,  $\omega$ - $2\theta$  scan, scan speed  $8^\circ \text{ min}^{-1}$  ( $2\theta$ ), scan range  $(1.0 + 0.35 \tan \theta)^\circ$ . Intensities of three standard reflections ( $\bar{1}0\bar{5}$ ,  $190$ ,  $6\bar{2}\bar{2}$ ) monitored every 50 reflections showed only statistical fluctuation. 2723 reflections measured, 2570 observed with  $|F_o| > 3\sigma(F_o)$ . Final agreement parameters:  $R = 0.053$ ,  $wR = 0.057$ ,  $w = [\sigma^2(F_o) + 0.002F_o^2]^{-1}$ ,  $(\Delta/\sigma)_{\max} = 0.48$ ,  $\Delta\rho_{\max} = 0.8 \text{ e \AA}^{-3}$ . At 223 K: crystal dimensions  $0.5 \times 0.5 \times 0.3$  mm obtained from the same batch as that at 298 K, Rigaku AFC5R diffractometer, graphite-

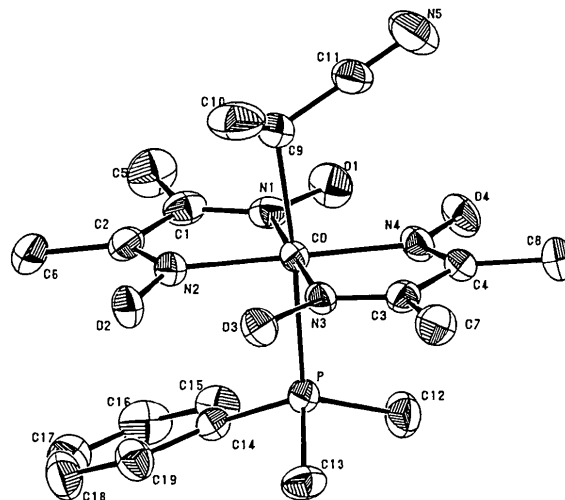


Fig. 1. ORTEP drawing of *S*-cn-dmp at 223 K with the atomic numbering scheme. Thermal ellipsoids are shown at the 50% probability level.

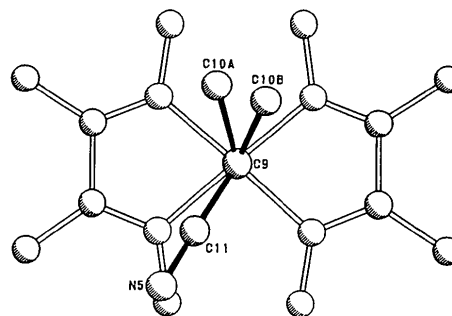


Fig. 2. Projection of the disordered cyanoethyl group onto the mean plane of cobaloxime moiety.

Table 2. Selected bond distances (Å), bond angles (°) and torsion angles (°)

223 K		298 K	
Co—P	2.297 (1)	C9—C10A	1.53 (1)
Co—N1	1.881 (3)	C9—C10B	1.40 (1)
Co—N2	1.877 (2)	C9—C11	1.42 (1)
Co—N3	1.880 (3)		
Co—N4	1.883 (3)		
Co—C9	2.078 (4)		
P1—C12	1.815 (4)		
P1—C13	1.814 (4)		
P1—C14	1.823 (4)		
C9—C10	1.508 (6)		
C9—C11	1.434 (6)		
P—Co—N1	89.1 (1)	Co—C9—C10A	115.9 (7)
P—Co—N2	88.95 (9)	Co—C9—C10B	124.6 (5)
P—Co—N3	92.0 (1)	Co—C9—C11	111.7 (6)
P—Co—N4	90.88 (9)	C10A—C9—C10B	34.5 (5)
P—Co—C9	175.0 (1)	C10A—C9—C11	113.0 (8)
N1—Co—N2	81.6 (1)	C10B—C9—C11	123.0 (7)
N1—Co—N3	178.8 (1)		
N1—Co—N4	98.3 (1)		
N1—Co—C9	87.7 (2)		
N2—Co—N3	98.9 (1)		
N2—Co—N4	179.8 (1)		
N2—Co—C9	86.7 (2)		
N3—Co—N4	81.2 (1)		
N3—Co—C9	91.2 (1)		
N4—Co—C9	93.4 (2)		
Co—P—C12	114.0 (2)		
Co—P—C13	114.6 (1)		
Co—P—C14	113.9 (1)		
Co—C9—C10	117.2 (3)		
Co—C9—C11	111.4 (3)		
C10—C9—C11	111.8 (4)		
N1—Co—C9—C10	146.1 (4)	N1—Co—C9—C10A	146.8 (7)
N1—Co—C9—C11	-83.5 (3)	N1—Co—C9—C10B	107.8 (6)
		N1—Co—C9—C11	-81.8 (6)

monochromated Mo *K*α radiation, cell parameters using 25 reflections in the range  $20 < 2\theta < 30^\circ$ , intensity measurement  $2\theta \leq 50^\circ$ , range of *hkl*: 0 to 11, 0 to 17, -10 to 10,  $\omega$ - $2\theta$  scan, scan speed  $32^\circ \text{ min}^{-1}$  ( $2\theta$ ), scan range  $(1.2 + 0.35 \tan \theta)^\circ$ . Intensities of three standard reflections (00 $\bar{1}$ ,  $\bar{1}10$ , 0 $\bar{1}\bar{1}$ ) monitored every 150 reflections with only small random variations. 2095 reflections measured, 1987 observed with  $|F_o| > 3\sigma(F_o)$ . Final agreement parameters  $R = 0.024$ ,  $wR = 0.030$ ,  $w = [\sigma^2(F_o) + 0.002F_o^2]^{-1}$ ,  $(\Delta/\sigma)_{\text{max}} = 0.04$ ,  $\Delta\rho_{\text{max}} = 0.4 \text{ e } \text{Å}^{-3}$ . Intensity data corrected for Lorentz and polarization, but not for absorption or extinction. Both structures solved by direct methods with *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Full-matrix least squares with *SHELX76* (Sheldrick, 1976) for the 298 K structure and with *TEXSAN* (Molecular Structure Corporation, 1985) for the 223 K structure. Refinements on *F*. Anisotropic thermal parameters for non-H atoms except for the methyl C atom, C10B, produced in the racemization at 298 K. Most H atoms from a difference map, remaining H atoms placed in calculated positions. The C—H distances of the methyl groups constrained to have a fixed value (1.0 Å) for 298 K structure. Atomic scattering factors including anomalous-dispersion terms from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The calculation carried out on the FACOM-HITAC system M-680H computer at the University of Tokyo for the 298 K structure, and on a MicroVAX II computer for the 223 K structure.

**Discussion.** Final atomic parameters for non-H atoms at 298 and 223 K are given in Table 1.\* The *ORTEP* (Johnson, 1965) drawing of the molecule at 223 K with numbering of the atoms is shown in Fig. 1. A new peak, C(10B), appeared and the peak of C(10A) decreased during the data collection at 298 K compared with the structure at 223 K. The occupancy ratio of C(10A) to C(10B) is 70:30. Fig. 2 shows the projection of the cyanoethyl group onto the mean plane of the cobaloxime moiety at 298 K. It was clear that the chiral cyanoethyl group is gradually inverted to have the opposite configuration on exposure to X-rays and that the crystalline-state racemization reached 60% during the data collection at 298 K. Selected bond distances, bond angles and torsion angles at 298 and 223 K are listed in Table 2. The Co—P distances 2.299 (2) (298 K) and 2.297 (1) Å (223 K) are significantly shorter than those observed in [(*R*)-1-cyanoethyl](triphenylphosphine)cobaloxime, 2.410 (3) Å (Kurihara, Uchida, Ohashi, Sasada, Ohgo & Baba, 1983) and [(*R*)-1-cyanoethyl](ethylidiphenylphosphine)cobal-

\* Lists of structure factors, anisotropic thermal parameters for non-H atoms, H-atom parameters, bond distances, bond angles and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53415 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

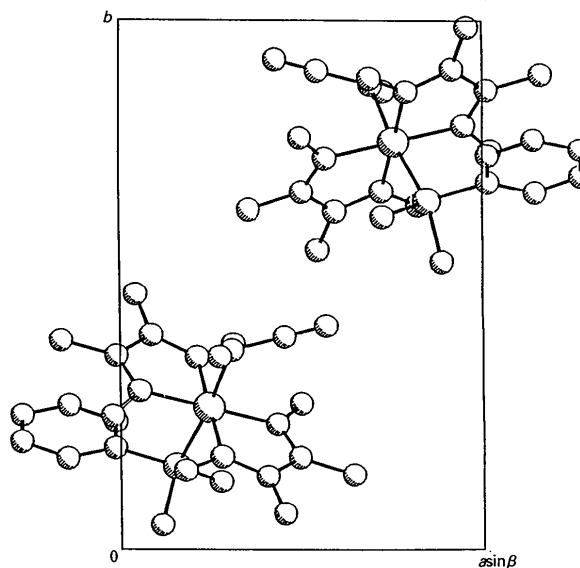


Fig. 3. Crystal structure of *S*-cn-dmp at 223 K viewed along the *c* axis.

Table 3. *Cavity volume ( $\text{\AA}^3$ ) of crystals of mode I*

Crystal	Volume
S-cn-dmp (298K)	17.97
S-cn-dmp (223K)	15.54
R-cn-S-mba (298K)	14.53
S-cn-S-mba (298K)	12.23
R-cn-pyrr (298K)	11.55

oxime, 2.370 (1)  $\text{\AA}$  (Tomotake, Uchida, Ohashi, Sasada, Ohgo & Baba, 1984), and slightly shorter than those observed in [(R)-1-cyanoethyl](tributylphosphine)cobaloxime, 2.316  $\text{\AA}$  (Kurihara, Uchida, Ohashi, Sasada, Ohgo & Baba, 1983) and [(R)-1-cyanoethyl](diethylphenylphosphine)cobaloxime, 2.313 (1)  $\text{\AA}$  (Tomotake, Uchida, Ohashi, Sasada, Ohgo & Baba, 1984).

The crystal structure at 223 K is shown in Fig. 3. There is one molecule in the asymmetric unit and the reactive cyanoethyl group is isolated from the other cyanoethyl group. The racemization occurring in such a crystal packing is classified as mode I. There is no unusually short contact between the molecules.

In order to examine why the present crystal is so rapidly racemized by X-ray exposure, the cavity for the cyanoethyl group was calculated (Ohashi, Uchida, Sasada & Ohgo, 1983) and the volume compared with the corresponding volumes of other crystals of mode I in Table 3. Although for the present crystal, the cavity at 298 K is not exact since the structure is not the initial one but that racemized at the 60% level, its volume, 17.97  $\text{\AA}^3$ , is significantly greater than those of the other crystals. Since the volume at 223 K, 15.54  $\text{\AA}^3$ , is still greater than those of the other cavities, the present crystal may be

racemized by X-ray exposure at 223 K. Experiments to obtain the rate constants at various temperatures are in progress.

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*Acta Cryst.* (1991). **C47**, 303–305

## Structure of $(\eta^5\text{-C}_5\text{H}_4\text{CH}_2\text{CH}_2\text{OH})_2\text{Mo}_2(\text{CO})_6$ , Hexacarbonylbis- $[\eta^5\text{-1-(2-hydroxyethyl)cyclopentadienyl}]$ dimolybdenum

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**Abstract.**  $\text{C}_{20}\text{H}_{18}\text{Mo}_2\text{O}_8$ ,  $M_r = 578.2$ , orthorhombic,  $P2_12_12_1$ ,  $a = 13.874$  (2),  $b = 16.870$  (4),  $c = 9.131$  (2)  $\text{\AA}$ ,  $V = 2137$  (1)  $\text{\AA}^3$ ,  $Z = 4$ ,  $D_x = 1.80$   $\text{Mg m}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 0.71069$   $\text{\AA}$ ,  $\mu =$

$1.19$   $\text{mm}^{-1}$ ,  $F(000) = 1144$ ,  $T = 294$  K,  $R = 0.036$  for 1915 independent data [ $I \geq 3\sigma(I)$ ]. Each Mo atom exhibits 'four-legged piano stool' bond geometry, with the other Mo atom at a basal position (Mo—Mo 3.211  $\text{\AA}$ ). The molecule is close to being centrosymmetric if the orientations of the terminal

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