

Table 2. Selected bond lengths (Å) and angles (°)

Au—C(11)	2.067 (6)	Au—C(21)	2.055 (4)
Au—C(31)	2.069 (4)	Au—C(1)	2.109 (6)
P—C(41)	1.794 (4)	P—C(51)	1.800 (5)
P—C(1)	1.773 (6)	P—C(2)	1.794 (5)
C(11)—Au—C(21)	90.5 (2)	C(11)—Au—C(31)	89.8 (2)
C(21)—Au—C(31)	176.4 (2)	C(11)—Au—C(1)	176.5 (2)
C(21)—Au—C(1)	91.6 (2)	C(31)—Au—C(1)	87.9 (2)
C(41)—P—C(51)	106.9 (2)	C(41)—P—C(1)	112.0 (2)
C(51)—P—C(1)	111.0 (3)	C(41)—P—C(2)	108.8 (3)
C(51)—P—C(2)	107.9 (2)	C(1)—P—C(2)	110.1 (3)
Au—C(11)—C(12)	119.4 (5)	Au—C(11)—C(16)	125.0 (4)
C(12)—C(11)—C(16)	115.5 (5)	Au—C(21)—C(26)	123.5 (4)
Au—C(21)—C(22)	120.9 (5)	Au—C(31)—C(36)	124.7 (4)
C(22)—C(21)—C(26)	115.6 (4)	Au—C(1)—P	115.3 (3)
Au—C(31)—C(32)	120.5 (3)		
C(32)—C(31)—C(36)	114.7 (4)		

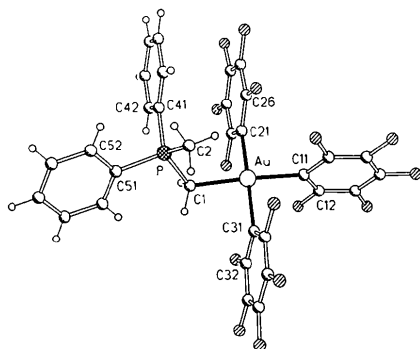


Fig. 1. The title compound in the crystal, showing the atom-numbering scheme. Radii are arbitrary.

(Usón, Laguna, Laguna, Gimeno, de Pablo, Jones, Meyer-Bäse & Freire Erdbrügger, 1987), 2.149 (13) Å in  $(\text{PPh}_3\text{CH}_2)\text{AuMe}_3$  (Stein, Fackler, Papparizos & Chen, 1981) and 2.079, 2.099 (13) Å in  $[(\text{Ph}_3\text{PCH}_2)_2\text{Au}.\text{AgClO}_4]_2$  (Usón, Laguna, Laguna, Usón, Jones & Freire Erdbrügger, 1987).

*Acta Cryst.* (1992). C48, 1211–1214

## Structure of *trans*-Tetraaquabis(trimethylammonioacetato)cobalt(II) Dichloride Tetrahydrate\*

BY XIAO-MING CHEN AND THOMAS C. W. MAK

*Department of Chemistry, The Chinese University of Hong Kong, Shatin, New Territories, Hong Kong*

(Received 14 October 1991; accepted 3 December 1991)

**Abstract.** A new cobalt(II) complex of trimethylammonioacetate (betaine, BET) has been prepared

\* Metal–Betaine Interactions. XVI. Part III: Chen & Mak (1991a).

The shortest nonbonded contact between non-H atoms is F(15)⋯F(34) ( $2-x, 1-y, 1-z$ ) 2.91 Å.

We thank the Fonds der Chemischen Industrie for financial support. The crystals were provided by Professors R. Usón and A. Laguna and co-workers at the University of Zaragoza. Intensity measurements were carried out at the Inorganic Chemistry Institute, University of Göttingen, Germany.

### References

- JONES, P. G. (1988). *J. Organomet. Chem.* **345**, 405–411.  
LAGUNA, A. & LAGUNA, M. (1990). *J. Organomet. Chem.* **394**, 743–756.  
MURRAY, H. H., FACKLER, J. P., PORTER, L. C., BRIGGS, D. A., GUERRA, M. A. & LAGOW, R. J. (1987). *Inorg. Chem.* **26**, 357–363.  
SHELDRICK, G. M. (1987). *SHELXTL-Plus*. Release 4.1 for Siemens R3m/V crystallographic system. Siemens Analytical X-ray Instruments, Inc., Madison, Wisconsin, USA.  
STEIN, J., FACKLER, J. P., PAPPARIZOS, C. & CHEN, H.-W. (1981). *J. Am. Chem. Soc.* **103**, 2192–2198.  
USÓN, R. (1986). *Coord. Chem. Rev.* **70**, 1–50.  
USÓN, R., LAGUNA, A., LAGUNA, M., CASTILLA, M. L., JONES, P. G. & FITTSCHEN, C. (1987). *J. Chem. Soc. Dalton Trans.* pp. 3017–3022.  
USÓN, R., LAGUNA, A., LAGUNA, M., FERNANDEZ, E., JONES, P. G. & SHELDRICK, G. M. (1982). *J. Chem. Soc. Dalton Trans.*, pp. 1971–1976.  
USÓN, R., LAGUNA, A., LAGUNA, M., FERNANDEZ, E., VILLACAMPA, M. D., JONES, P. G. & SHELDRICK, G. M. (1983). *J. Chem. Soc. Dalton Trans.* pp. 1679–1685.  
USÓN, R., LAGUNA, A., LAGUNA, M., GIMENO, M. C., DE PABLO, A., JONES, P. G., MEYER-BÄSE, K. & FREIRE ERDBRÜGGER, C. (1987). *J. Organomet. Chem.* **336**, 461–468.  
USÓN, R., LAGUNA, A., LAGUNA, M. & USÓN, A. (1983). *Inorg. Chim. Acta*, **73**, 63–66.  
USÓN, R., LAGUNA, A., LAGUNA, M., USÓN, A. & GIMENO, M. C. (1987). *Organometallics*, **6**, 682–683.  
USÓN, R., LAGUNA, A., LAGUNA, M., USÓN, A., JONES, P. G. & FREIRE ERDBRÜGGER, C. (1987). *Organometallics*, **6**, 1778–1880.

1.401 Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073 \text{ \AA}$ ,  $\mu = 0.99 \text{ mm}^{-1}$ ,  $F(000) = 538$ ,  $T = 298 (1) \text{ K}$ ,  $R = 0.043$  for 2160 unique Mo  $K\alpha$  data. The complex comprises distorted octahedral [Co(BET)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>]<sup>2+</sup> cations in which the metal atom is located at an inversion centre, being coordinated by two *trans*-related BET ligands through their unidentate carboxylato groups [2.097 (1) Å] and four aqua ligands [2.087 (2)–2.101 (2) Å]. The uncoordinated carboxy O atoms form intramolecular hydrogen bonds with the aqua ligands. Intermolecular hydrogen bonding involving the aqua ligands, lattice water molecules and chloride ions links the structure into a three-dimensional network.

**Introduction.** Among the structurally known cobalt(II) complexes of carboxylates and related ligands, some are carboxylate-bridged polymeric species such as diaquabis(glycine)cobalt(II) bromide (Ravikumar, Rajan, Natarajan, Ponnuswamy & Trotter, 1985), diaquadichloro(glycine)cobalt(II) and tetraaqua(glycine)cobalt(II) nitrate (Clegg, Lacy & Straughan, 1987), whereas monomeric species like tetraaquabis(acetato)cobalt(II) (van Niekerk & Schoening, 1953), tetraaquabis(*p*-nitrobenzoato)cobalt(II) dihydrate (Nadzhafov, Snulin & Mamedov, 1981), bis(*N*-tosylglycinato)tetraaqua-cobalt(II) (Battagaglia, Corradi, Menabue, Saladini, Sola & Gavioli, 1985), and tetraaquabis(4-carboxyphenoxyacetato)cobalt(II) (Kennard, Smith, O'Reilly & Manoharan, 1984) all have octahedral structures with the central atom coordinated by two *trans*-related carboxylato groups and four aqua ligands. An unusual example is tetraphenylarsonium tetrakis(trifluoroacetato)cobaltate(II) (Bergman & Cotton, 1966), in which the Co atom is tetrahedrally coordinated by four unidentate carboxylato groups.

As part of a systematic investigation of the structural chemistry of betaines (Chen & Mak, 1991*a*), we have reported the crystal structure of the cobalt(II) complex [Co(pyBET)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>].2[Co(pyBET)Cl<sub>3</sub>] (pyBET = pyridine betaine, C<sub>5</sub>H<sub>5</sub>N<sup>+</sup>CH<sub>2</sub>COO<sup>-</sup>), which comprises octahedral [Co(pyBET)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>]<sup>2+</sup> cations and tetrahedral [Co(pyBET)Cl<sub>3</sub>]<sup>-</sup> anions (Chen & Mak, 1991*b*). We report herein the preparation and crystal structure of a new cobalt(II) complex of trimethylammonioacetate (commonly referred to as betaine, and designated as BET), namely [Co(BET)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>]Cl<sub>2</sub>·4H<sub>2</sub>O.

**Experimental.** The title complex was simply prepared by dissolving CoCl<sub>2</sub>·6H<sub>2</sub>O (0.357 g, 1.5 mmol) and betaine monohydrate (0.405 g, 3.0 mmol) in water (3 ml) under stirring. After the resulting solution was allowed to stand at room temperature in air for two weeks, brown plate-like crystals were obtained. IR data (cm<sup>-1</sup>, KBr): 3430<sub>vs</sub>, 3374<sub>vs</sub>, 1616<sub>vs</sub>, 1484<sub>s</sub>,

1434<sub>s</sub>, 1398<sub>vs</sub>, 1342<sub>s</sub>, 984<sub>w</sub>, 963<sub>w</sub>, 934<sub>m</sub>, 899<sub>m</sub>, 794<sub>m</sub>, 723<sub>s</sub>, 611<sub>m</sub>, 548<sub>m</sub>.  $D_m$  determined by flotation in CCl<sub>4</sub>/BrCH<sub>2</sub>CH<sub>2</sub>Br.

A crystal of size 0.20 × 0.38 × 0.40 mm was used to collect intensity data in the  $\omega$ -scan mode (rate 2.93–14.63° min<sup>-1</sup>; range 0.65° below  $K\alpha_1$  to 0.65° above  $K\alpha_2$ ) on a Nicolet R3m/V diffractometer using graphite-monochromated Mo  $K\alpha$  radiation,  $\lambda = 0.71073 \text{ \AA}$ ; lattice parameters were refined from 25 reflections,  $15 \leq 2\theta \leq 25^\circ$ ; 3123 reflections were collected,  $3 \leq 2\theta \leq 55^\circ$ ,  $0 \leq h \leq 11$ ,  $0 \leq k \leq 15$ ,  $-15 \leq l \leq 15$ ; two standards (154, 243) were checked after every 120 reflections, random deviation ( $\leq 2\%$ ) only;  $L_p$  correction, empirical absorption correction based on  $\psi$ -scan data (transmission factors 0.597–0.721); after merging ( $R_{\text{int}} = 0.023$ ) 2743 unique reflections, 2165 were considered observed with  $I \geq 3\sigma(I)$ . The structure was solved by Patterson superposition and subsequent difference Fourier synthesis. Non-H atoms were refined anisotropically. H atoms of the BET ligand were generated geometrically (C–H = 0.96 Å) and assigned the same isotropic thermal parameters,  $U = 0.08 \text{ \AA}^2$ , while H atoms of water molecules were located from the difference map and included in structure-factor calculations. The quantity minimized was  $\sum w(F_o - F_c)^2$ , where  $w = [\sigma^2(F_o) + 0.0001|F_o|^2]^{-1}$ . Final  $R = 0.043$ ,  $wR = 0.046$  and  $S = 2.325$  for 125 parameters and 2165 observed reflections,  $(\Delta/\sigma)_{\text{max}} = 0.0002$ ,  $(\Delta\rho)_{\text{max}} = 0.36$ ,  $(\Delta\rho)_{\text{min}} = -0.48 \text{ e \AA}^{-3}$ .

All computations were performed on a DEC MicroVAXII computer with the *SHELXTL-Plus* program package (Sheldrick, 1982, 1985). Analytic expressions of neutral-atom scattering factors were employed, and anomalous-dispersion corrections were incorporated (*International Tables for X-ray Crystallography*, 1974, Vol. IV, pp. 55, 99, 149).

**Discussion.** Atomic coordinates and thermal parameters are listed in Table 1,\* and bond distances and angles in Table 2.

The crystal structure consists of discrete [Co(BET)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>]<sup>2+</sup> cations, chloride anions and lattice water molecules. As illustrated in Fig. 1, each metal atom, located at an inversion centre, is coordinated by four aqua ligands [Co–O(aqua) = 2.087 (2)–2.101 (2) Å] and two *trans*-related BET ligands through their unidentate carboxylato groups [Co–O(carboxy) = 2.097 (2) Å] in a slightly distorted octahedral environment, the most distorted O–Co–O bond angle being 87.5 (1)°. Each

\* 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 54927 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0565]

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^4$  for Co, Cl;  $\times 10^3$  for others)

Equivalent isotropic  $U$  is defined as one third of the trace of the orthogonalized  $U_{ij}$  tensor.

	$x$	$y$	$z$	$U_{eq}$
Co(1)	0	0	0	277 (2)
Cl(1)	1164 (1)	-1376 (1)	3676 (1)	562 (3)
O(1w)	682 (2)	1465 (2)	929 (2)	40 (1)
O(2w)	1587 (3)	-983 (2)	1209 (2)	38 (1)
O(3w)	1906 (3)	1185 (2)	3190 (2)	61 (1)
O(4w)	4164 (3)	-2481 (2)	5285 (2)	76 (1)
O(1)	1815 (2)	185 (2)	-890 (2)	38 (1)
O(2)	2349 (3)	2007 (2)	-651 (2)	57 (1)
C(1)	2431 (3)	1084 (2)	-1114 (2)	36 (1)
C(2)	3351 (4)	1106 (2)	-2043 (3)	44 (1)
N(1)	3714 (3)	13 (2)	-2557 (2)	38 (1)
C(3)	4785 (4)	-699 (3)	-1674 (3)	58 (1)
C(4)	4598 (5)	289 (3)	-3471 (3)	58 (1)
C(5)	2217 (4)	-621 (3)	-3092 (3)	59 (1)

Table 2. Bond lengths ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

Co(1)—O(1w)	2.087 (2)	Co(1)—O(2w)	2.101 (2)
Co(1)—O(1)	2.097 (2)		
O(1)—C(1)	1.251 (3)	O(2)—C(1)	1.246 (4)
C(1)—C(2)	1.518 (5)	C(2)—N(1)	1.509 (4)
N(1)—C(3)	1.496 (4)	N(1)—C(4)	1.516 (5)
N(1)—C(5)	1.494 (4)		
O(1w)—Co(1)—O(2w)	92.2 (1)	O(1w)—Co(1)—O(1)	92.5 (1)
O(2w)—Co(1)—O(1)	89.2 (1)	O(1w)—Co(1)—O(1w <sup>a</sup> )	180.0 (1)
O(2w)—Co(1)—O(1w <sup>a</sup> )	87.8 (1)	O(1)—Co(1)—O(1w <sup>a</sup> )	87.5 (1)
O(2w)—Co(1)—O(2w <sup>a</sup> )	180.0 (1)	O(1)—Co(1)—O(2w <sup>a</sup> )	90.8 (1)
O(1)—Co(1)—O(1 <sup>a</sup> )	180.0 (1)	Co(1)—O(1)—C(1)	126.9 (2)
O(1)—C(1)—O(2)	126.1 (3)	O(1)—C(1)—C(2)	119.5 (3)
O(2)—C(1)—C(2)	114.4 (3)	C(1)—C(2)—N(1)	119.0 (2)
C(2)—N(1)—C(3)	110.2 (2)	C(2)—N(1)—C(4)	107.5 (2)
C(3)—N(1)—C(4)	108.7 (2)	C(2)—N(1)—C(5)	112.3 (2)
C(3)—N(1)—C(5)	109.8 (2)	C(4)—N(1)—C(5)	108.3 (2)
O(2) $\cdots$ O(1w)	2.718 (5)	O(1w) $\cdots$ O(3w)	2.712 (4)
O(2w) $\cdots$ Cl(1)	3.129 (4)	O(2w) $\cdots$ O(2 <sup>b</sup> )	2.706 (5)
O(3w) $\cdots$ Cl(1)	3.201 (4)	O(3w) $\cdots$ O(4w <sup>a</sup> )	2.753 (5)
O(4w) $\cdots$ Cl(1)	3.119 (4)	O(4w) $\cdots$ Cl(1 <sup>d</sup> )	3.185 (4)

Symmetry code: (a)  $-x, -y, -z$ ; (b)  $\frac{1}{2} - x, -\frac{1}{2} + y, -z$ ; (c)  $\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$ ; (d)  $\frac{1}{2} + x, -\frac{1}{2} - y, z$ .

uncoordinated carboxy O atom forms an intramolecular hydrogen bond with an aqua ligand [O(2) $\cdots$ O(1w) = 2.718 (4)  $\text{\AA}$ ] and an intermolecular hydrogen bond with a lattice water molecule [O(2) $\cdots$ O(2wg) = 2.706 (5)  $\text{\AA}$ ]. The intramolecular hydrogen bonding may account for the fact that the two carboxylato C—O bonds are essentially equivalent [1.251 (3), 1.246 (4)  $\text{\AA}$ ], in contrast to the markedly different bond lengths found in protonated carboxy groups of BET [C—O(H) = 1.316 (2), C=O = 1.193 (2)  $\text{\AA}$ ] (Fischer, Templeton & Zalkin, 1970; Yip, Wang & Mak, 1990) and in unidentate carboxylato ligands (Mehrotra & Brohra, 1983). The carboxylato group in the present complex may be described as acting in a 'pseudo-chelating' mode; the

same structure motif has been found in several simple cobalt(II) dicarboxylates such as tetraaqua-bis(*p*-nitrobenzoato)cobalt(II) dihydrate (Nadzhafov *et al.*, 1981), bis(*N*-tosylglycinato)tetraaquacobalt(II) (Battaglia *et al.*, 1985), and tetraaqua-bis(4-carboxyphenoxyacetato)cobalt(II) (Kennard *et al.*, 1984). On the other hand, this structure is different from the other known cobalt(II) complex of a betaine analogue, namely [Co(pyBET)<sub>4</sub>(H<sub>2</sub>O)<sub>2</sub>].2[Co(pyBET)Cl<sub>3</sub>] (Chen & Mak, 1991b), in which the metal atom is octahedrally coordinated by four unidentate pyBET ligands and two aqua ligands in the cation, or tetrahedrally coordinated by one unidentate pyBET ligand and three chloro ligands in the anion.

Hydrogen bonding plays an important role in consolidating the crystal structure. Each aqua ligand forms two donor hydrogen bonds, each lattice water molecule two donor and one acceptor hydrogen bonds, while the chloride ion is involved in four acceptor hydrogen bonds in a distorted tetrahedral geometry as illustrated in Fig. 1. These hydrogen bonds build up a three-dimensional network (Fig. 2). Details of the hydrogen-bond lengths are listed in Table 2.

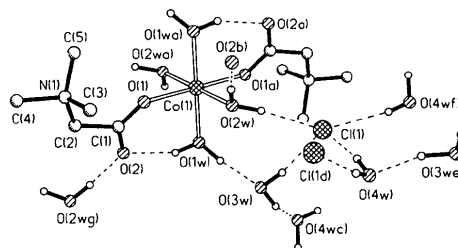


Fig. 1. Perspective view showing the coordination geometry in [Co(BET)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>]Cl<sub>2</sub>·4H<sub>2</sub>O, the hydrogen-bonding scheme and atom numbering. The H atoms of BET ligands are omitted for clarity and hydrogen bonds are represented by broken lines. Symmetry codes are given in Table 2.

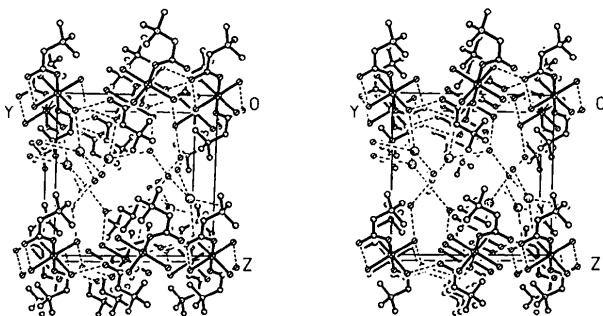


Fig. 2. Stereoview of the crystal structure of [Co(BET)<sub>2</sub>(H<sub>2</sub>O)<sub>4</sub>]Cl<sub>2</sub>·4H<sub>2</sub>O. The origin of the unit cell lies at the upper right corner, with a pointing towards the reader, b from right to left, and c downwards. Hydrogen bonds are represented by broken lines.

This work was supported by a Hong Kong RGC Earmarked Grant for Research (Acc. No. 221600010).

#### References

- BATTAGAGLIA, L. P., CORRADI, A. B., MENABUE, L., SALADINI, M., SOLA, M. & GAVIOLI, G. B. (1985). *Inorg. Chim. Acta*, **107**, 73–79.
- BERGMAN, J. G. & COTTON, F. A. (1966). *Inorg. Chem.* **5**, 1420–1428.
- CHEN, X.-M. & MAK, T. C. W. (1991a). *J. Chem. Soc. Dalton Trans.* pp. 1219–1222.
- CHEN, X.-M. & MAK, T. C. W. (1991b). *J. Crystallogr. Spectrosc. Res.* **21**, 471–478.
- CLEGG, W., LACY, O. M. & STRAUGHAN, B. P. (1987). *Acta Cryst.* **C43**, 794–797.
- FISCHER, M. S., TEMPLETON, D. H. & ZALKIN, A. (1970). *Acta Cryst.* **B26**, 1392–1397.
- KENNARD, C. H. L., SMITH, G., O'REILLY, E. J. & MANOHARAN, P. T. (1984). *Inorg. Chim. Acta*, **82**, 35–40.
- MEHROTRA, R. C. & BROHRA, R. (1983). *Metal Carboxylates*. New York: Academic Press.
- NADZHAFOV, G. N., SNULIN, A. N. & MAMEDOV, KH. S. (1981). *J. Struct. Chem.* **22**, 898–902.
- NIEKERK, J. N. VAN & SCHOENING, F. R. L. (1953). *Acta Cryst.* **6**, 609–612.
- RAVIKUMAR, K., RAJAN, S. S., NATARAJAN, S., PONNUSWAMY, M. N. & TROTTER, J. (1985). *Z. Kristallogr.* **171**, 201–207.
- SHELDRIK, G. M. (1982). *Computational Crystallography*, edited by D. SAYRE, pp. 506–514. Oxford Univ. Press.
- SHELDRIK, G. M. (1985). *Crystallographic Computing 3: Data Collection, Structure Determination, Proteins and Databases*, edited by G. M. SHELDRIK, C. KRÜGER & R. GODDARD, pp. 175–189. Oxford Univ. Press.
- YIP, W. H., WANG, R.-J. & MAK, T. C. W. (1990). *Acta Cryst.* **C46**, 717–719.

*Acta Cryst.* (1992). **C48**, 1214–1216

## Structure of Dithiocyanato[(-)-spartein]cobalt(II)

BY KATHLEEN M. JUPP AND PAUL R. RAITHY

*University Chemical Laboratory, Lensfield Road, Cambridge CB2 1EW, England*

(Received 8 October 1991; accepted 4 December 1991)

**Abstract.** (Dodecahydro-7,14-methano-2*H*,6*H*-diprido[1,2-*a*:1',2'-*e*][1,5]diazocine-*N,N*)dithiocyanatocobalt(II), [Co(C<sub>15</sub>H<sub>26</sub>N<sub>2</sub>)(NCS)<sub>2</sub>], *M<sub>r</sub>* = 409.5, orthorhombic, *P*2<sub>1</sub>2<sub>1</sub>2<sub>1</sub>, *a* = 8.014 (4), *b* = 13.660 (7), *c* = 18.023 (7) Å, *V* = 1973.0 (16) Å<sup>3</sup>, *Z* = 4, *D<sub>x</sub>* = 1.378 Mg m<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 1.077 mm<sup>-1</sup>, *F*(000) = 860, *T* = 290 K, *R* = 0.067 for 1208 observed reflections [*F* > 4.0σ(*F<sub>o</sub>*)] and 169 parameters. A clear indication of absolute configuration [*η* = 1.13 (16)] is also obtained. The structure is found to be very similar to that of the dichloro[(-)-spartein]cobalt(II) complex characterized by Kuroda & Mason [*J. Chem. Soc. Dalton Trans.* (1977), pp. 371–373].

**Introduction.** As part of a study of *d*-electron optical activity, Mason & Peacock (1973) prepared a series of compounds of the type [*M*(spartein)*X*<sub>2</sub>], where *M* = Co<sup>II</sup>, Ni<sup>II</sup>, Cu<sup>II</sup> and Zn<sup>II</sup>; and *X* = Cl<sup>-</sup>, Br<sup>-</sup> or I<sup>-</sup>. Both magnetic moments and electronic spectra for the compounds were reported, as well as the differential absorption [or circular dichroism (CD)] of [Ni(sp)Cl<sub>2</sub>]. Later, detailed studies of the *d*-electron optical activity of both dichloro[(-)-spartein]cobalt(II) (Drake, Kuroda, Mason, Peacock & Stewart, 1981) and dichloro[(-)-α-isosparteine]cobalt(II)

(Drake, Hirst, Kuroda & Mason, 1982) were reported.

A recent ligand-field study (Fenton & Gerloch, 1990) has successfully reproduced the *d-d* transition energies, intensities and differential absorption of these two Co compounds, as well as those of a third compound, dichloro[(+)-*N,N,N',N'*-tetramethyl-1,2-propylenediamine]cobalt(II). The authors found that they could reproduce both transition and differential absorption intensities only by inclusion of misdirected valency, or 'bent bonding', in the first coordination shell. The parameter set of the bent bonding was shown to correlate strongly with the detailed coordination geometry of the central metal ion. The two spartein complexes analysed possess pseudo-*C*<sub>2</sub> symmetry at the chromophore (Kuroda & Mason, 1977, 1979): the coordination planes CoNN' and CoClCl' are rotated from orthogonality by *ca* 6 and 19° in the two complexes following intramolecular steric crowding. The angle of rotation is important in determining part of the bent bonding in these complexes.

As part of a program to extend the ligand-field analysis to magneto circular dichroism (MCD) as well as natural CD, and to enlarge the range of object systems, we report here an X-ray structural