C2—C3	1.404 (10)	C2′—C3′	1.333 (12)
C3—C4	1.413 (11)	C3′—C4′	1.419 (12)
C4—C5	1.368 (12)	C4′—C12′	1.384 (10)
C4—C12	1.432 (11)	C4′—C5′	1.495 (10)
C5—C6	1.344 (12)	C5′—C6′	1.372 (12)
C6—C7	1.423 (11)	C6′—C7′	1.446 (10)
C7—C11	1.341 (11)	C7′—C8′	1.406 (11)
C7—C8	1.400 (12)	C7′—C11′	1.451 (9)
C8—C9	1.351 (13)	C8′—C9′	1.389 (10)
C9—C10	1.418 (10)	C9′—C10′	1.381 (11)
C11—C12	1.478 (10)	C11′—C12′	1.382 (11)
C13—C14	1.527 (8)	C13′—C14′	1.531 (8)
C14—C15	1.496 (10)	C14′—C15′	1.503 (10)
O1—Cu1—N3	84.7 (2)	O1′—Cu2—N3′	84.5 (2)
O1—Cu1—N1	90.9 (2)	O1′—Cu2—N1′	91.2 (2)
N3—Cu1—N1	171.7 (2)	N3′—Cu2—N1′	171.8 (2)
O1—Cu1—N2	163.9 (2)	O1′—Cu2—N2′	163.6 (2)
N3—Cu1—N2	100.3 (2)	N3′—Cu2—N2′	100.4 (2)
N1—Cu1—N2	82.1 (2)	N1′—Cu2—N2′	81.9 (2)
O1—Cu1—OW	97.8 (2)	O1′—Cu2—OW′	98.1 (2)
N3—Cu1—OW	96.0 (2)	N3′—Cu2—OW′	95.9 (2)
N1—Cu1—OW	91.5 (2)	N1′—Cu2—OW′	91.6 (2)
N2—Cu1—OW	96.8 (2)	N2′—Cu2—OW′	96.9 (2)
N1—C12—C11—N2	1.4 (9)	O1—C13—C14—N3	16.1 (7)
N1′—C12′—C11′—N2′	−1.4 (9)	O1′—C13′—C14′—N3′	23.8 (8)

H atoms bonded to C atoms were positioned geometrically and refined with a common *U* value. The remaining H atoms were located by difference Fourier synthesis and refined using distance restraints and common *U* values.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software* (Enraf–Nonius, 1989). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *PC-ORTEP* (Schmid & Bruggemann, 1990*a*) and *PC-PLUTO* (Schmid & Bruggemann, 1990*b*).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1141). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). C**51**, 1508–1510

Disilver *cis*-Butenedioate

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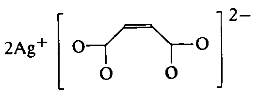
Abstract

Disilver *cis*-butenedioate, $[Ag_2(C_4H_2O_4)]$, has been synthesized and compared with other Ag carboxylates. One of the Ag atoms coordinates to the C═C double bond in the *cis*-butenedioic acid (trivial name: maleic acid) at a distance of 2.40 (1) Å.

Comment

Several Ag^I carboxylate complexes have hitherto been characterized by X-ray diffraction methods (Chen & Mak, 1991*a*,*b*). Some salient features of these compounds are (*a*) the aggregation of Ag atoms in dimers or, less commonly, polymers, and (*b*) the bridging carboxylate group connecting the metal atoms in the dimer.

The title compound, (I), can be characterized as containing isolated silver dimers. However, the Ag···Ag distance is significantly longer than the metal–metal contacts found in other dimeric silver(I) carboxylates, indicating a weak Ag···Ag interaction in the present compound. Moreover, no conclusion about bonding interactions can be drawn exlusively from the metal–metal distance (Jansen, 1987). The next shortest Ag···Ag contact is within the van der Waals radius of Ag, 3.44 Å (Bondi 1964). If this elongated Ag···Ag contact is included, the metal framework can be described as a linear tetramer. Other types of Ag-atom tetramers have been described (Sagatys, Smith, Bott, Lynch & Kennard, 1993; Michaelides, Kiritsis, Skoulika & Aubry, 1993).



(I)

A structural classification scheme for silver(I) carboxylates has been suggested by Mak, Yip, Kennard, Smith & O'Reilly (1986). This scheme divides the different coordination modes between carboxylate groups and Ag^+ ions into four principal types, *A–D*. The crystal structure of silver butenedioate is similar to the 'modified *D*-type' structure described in the Mak scheme. The main difference between the modified *D*-type structure and that adopted by the present compound is the absence of metal–ligand (*e.g.* water) interactions at Ag2.

The coordination modes of the two types of silver ions to O atoms are quite dissimilar. One of the atoms, Ag1, coordinates to three O atoms with bonding distances of 2.35 (4) Å, while the other atom, Ag2, has two shorter [2.20 (3) Å] and two longer [2.60 (2) Å] contacts to O atoms, thereby forming an approximately linear geometry if the two short contacts are considered the most important. The only possible silver–ligand interaction not involving the O atoms is represented by the unsymmetrical coordination [2.40 (1) Å] of Ag1 to the C2=C3 double bond. The normal C2=C3 distance could be interpreted as σ-donation dominating the metal–olefin interaction. This is in agreement with the reduced ability of Ag^I-to-olefin π-backbonding (Munakata, Kitagawa, Kosome & Asahara, 1986)

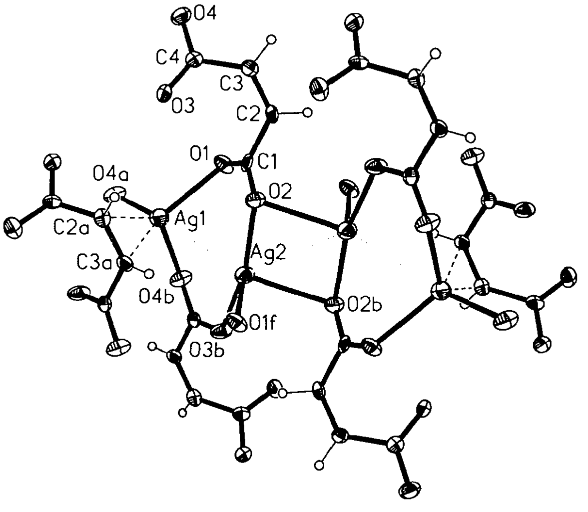


Fig. 1. Prespective view showing 50% probability ellipsoids. H atoms are drawn as small circles of arbitrary radii.

Experimental

Crystals were obtained by mixing aqueous solutions of silver nitrate and *cis*-butenedioic acid. After addition of a sodium hydroxide solution, enough to remove both protons of the acid, well formed crystals of the title compound precipitated.

Crystal data

$[Ag_2(C_4H_2O_4)]$
$M_r = 329.79$
Monoclinic
$P2_1/n$
$a = 5.2983\,(6)$ Å
$b = 9.2529\,(8)$ Å
$c = 11.261\,(2)$ Å
$\beta = 101.850\,(14)°$
$V = 540.29\,(11)$ Å^3
$Z = 4$
$D_x = 4.054$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 28 reflections
$\theta = 10.1–12.4°$
$\mu = 7.179$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.07 × 0.05 × 0.03 mm
Colourless

Data collection

Siemens Stoe AED-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: integration (*SHELXTL-Plus*; Sheldrick, 1991)
$T_{\min} = 0.7168$, $T_{\max} = 0.8596$
1537 measured reflections
799 independent reflections

624 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0486$
$\theta_{\max} = 23.49°$
$h = -5 \rightarrow 5$
$k = 0 \rightarrow 10$
$l = 0 \rightarrow 12$
3 standard reflections
frequency: 90 min
intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0234$
$wR(F^2) = 0.0493$
$S = 1.104$
799 reflections
92 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0189P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 1.107$ e Å^{-3}
$\Delta\rho_{\min} = -0.810$ e Å^{-3}
Extinction correction: *SHELXL93* (Sheldrick, 1993)
Extinction coefficient: 0.0110 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Ag1	0.33598 (12)	−0.37289 (6)	0.39030 (5)	0.0284 (2)
Ag2	0.00858 (12)	−0.17593 (6)	0.50769 (5)	0.0276 (2)
O1	0.5232 (9)	−0.1460 (5)	0.4014 (5)	0.0262 (12)
O2	0.1421 (9)	−0.0343 (5)	0.3726 (4)	0.0242 (12)
O3	0.4847 (11)	−0.1472 (5)	0.1336 (5)	0.0286 (12)
O4	0.8340 (10)	−0.0459 (6)	0.0914 (5)	0.0309 (12)
C1	0.3727 (13)	−0.0440 (7)	0.3630 (6)	0.018 (2)
C2	0.4821 (14)	0.0843 (7)	0.3081 (6)	0.0179 (15)
C3	0.6092 (13)	0.0828 (7)	0.2163 (6)	0.020 (2)
C4	0.6456 (13)	−0.0481 (7)	0.1434 (6)	0.018 (2)

Table 2. *Selected geometric parameters* (Å, °)

Ag1—O1	2.314 (5)	Ag2—O1^{v}	2.619 (5)
Ag1—O4^{i}	2.349 (5)	Ag2···Ag2^{iv}	3.2606 (11)
Ag1—O4^{ii}	2.388 (5)	O1—C1	1.254 (8)
Ag1—C3^{iii}	2.452 (7)	O2—C1	1.252 (8)
Ag1—C2^{iii}	2.539 (6)	O3—C4	1.242 (8)
Ag1···Ag2	3.0032 (8)	O4—C4	1.256 (8)
Ag2—O3^{ii}	2.185 (5)	C1—C2	1.508 (10)
Ag2—O2	2.230 (4)	C2—C3	1.345 (10)
Ag2—O2^{iv}	2.585 (5)	C3—C4	1.498 (10)
O1—Ag1—O4^{i}	108.1 (2)	O2^{iv}—Ag2···Ag1	162.63 (11)
O1—Ag1—O4^{ii}	108.6 (2)	O3^{ii}—Ag2···Ag2^{iv}	141.02 (14)
O4^{i}—Ag1—O4^{ii}	81.4 (2)	O2—Ag2···Ag2^{iv}	52.16 (12)
O1—Ag1···Ag2	72.88 (13)	O2^{iv}—Ag2···Ag2^{iv}	42.94 (10)
O4^{i}—Ag1···Ag2	149.55 (13)	Ag1···Ag2···Ag2^{iv}	126.50 (3)
O4^{ii}—Ag1···Ag2	70.00 (12)	Ag2—O2—Ag2^{iv}	84.90 (15)
O3^{ii}—Ag2—O2	162.0 (2)	Ag1^{vi}—O4—Ag1^{vii}	98.6 (2)
O3^{ii}—Ag2—O2^{iv}	99.2 (2)	C3—C2—C1	127.0 (6)
O2—Ag2—O2^{iv}	95.10 (15)	C3—C2—Ag1^{viii}	70.8 (4)
O3^{ii}—Ag2···Ag1	86.94 (13)	C2—C3—C4	124.8 (6)
O2—Ag2···Ag1	76.31 (12)	C2—C3—Ag1^{viii}	78.0 (4)

Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $x-\frac{1}{2}, -\frac{1}{2}-y, \frac{1}{2}+z$; (iii) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (iv) $-x, -y, 1-z$; (v) $x-1, y, z$; (vi) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; (vii) $\frac{1}{2}+x, -\frac{1}{2}-y, z-\frac{1}{2}$; (viii) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

Data collection: *DIF*4 (Stoe & Cie, 1991). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1991). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *EMACS* (Free Software Foundation Inc., 1993).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1232). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(Ammine)(aqua)[*N*-(2,6-dimethylphenyl)-*N*-(carboxymethyl)glycinato-*N*,*O*,*O*″]copper(II)

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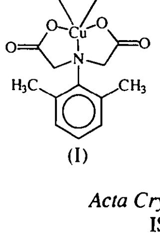
Abstract

The structure of the title compound [(ammine)(aqua)(2,6-dimethylphenyliminodiacetato-*N*,*O*,*O*″)copper(II)] consists of discrete neutral [$Cu(C_{12}H_{13}NO_4)(H_2O)(NH_3)$] molecules. The Cu atom is five-coordinate, with four short bonds formed with the N atom and two O atoms of the doubly deprotonated ligand and the N atom of the ammonia molecule. The water molecule is coordinated in an axial position at a longer distance. The coordination polyhedron can be described as a square-based pyramid with a small trigonal-bipyramidal distortion.

Comment

The crystal structure of the title compound, (I), consists of discrete neutral [$CuL(H_2O)(NH_3)$] molecules, where H_2L is *N*-(2,6-dimethylphenyl)-*N*-(carboxymethyl)glycine.



(I)