Br—Cd—O3	90.10 (9)	Cd—N^{ii}—C5^{ii}	120.8 (3)
Br—Cd—N^{ii}	103.66 (8)	C1—N—C5	118.2 (3)
Br^{i}—Cd—O1	95.0 (1)	N—C1—C2	122.8 (4)
Br^{i}—Cd—O2	95.32 (8)	C1—C2—C3	118.2 (3)
Br^{i}—Cd—O3	177.8 (1)	C1—C2—C6	120.7 (4)
Br^{i}—Cd—N^{ii}	92.47 (9)	C3—C2—C6	121.1 (3)
O1—Cd—O2	56.0 (1)	C2—C3—C4	119.2 (4)
O1—Cd—O3	85.9 (1)	C3—C4—C5	119.2 (4)
O1—Cd—N^{ii}	91.5 (1)	N—C5—C4	122.4 (4)
O2—Cd—O3	86.9 (1)	O1—C6—O2	121.7 (4)
O2—Cd—N^{ii}	147.1 (1)	O1—C6—C2	118.7 (4)
O3—Cd—N^{ii}	85.5 (1)	O2—C6—C2	119.6 (3)
Cd—Br—Cd^{i}	90.48 (3)		

Symmetry codes: (i) $2-x, 2-y, -z$; (ii) $x, 1+y, z$; (iii) $1-x, 1-y, -z$.

The structure was solved by the heavy-atom method and refined anisotropically for all non-H atoms. The H-atom sites were obtained from a difference Fourier synthesis and not refined. All computations were performed on a MicroVAX 3100 computer.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1989). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *ORTEP*II (Johnson, 1976).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the IUCr (Reference: AS1203). 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. (1996). **C52**, 593–595

Poly[bis(*p*-nitrosalicylato-*O*:*O*′)disilver(I)-*O*^{3}:*Ag*′;*Ag*:*O*$^{3\prime}$]

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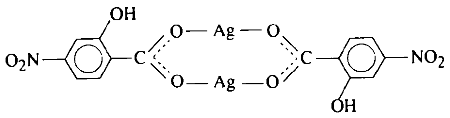
Abstract

The structure of the title compound, $[Ag_2(C_7H_4NO_5)_2]_n$, consists of polymeric networks made up of centrosymmetric bis(carboxylato-*O*,*O*′)-bridged Ag dimers, which are cross-linked *via* Ag—OH bonds [2.47 (1) Å]. The distances in the chelate ring are Ag—O 2.20 (1) and 2.16 (1), and Ag···Ag 2.796 (2) Å.

Comment

Several silver(I) carboxylate complexes have been reported previously. The majority of these structures consist of discrete centrosymmetric carboxylate-bridged Ag dimers, such as silver(I) benzoate and silver(I) 4-hydroxybenzoate (Usubaliev *et al.*, 1981), and silver(I) 2,6-dihydroxybenzoate (Smith, Kennard & Mak, 1988). Carboxylate-bridged Ag dimers are also known to cross-link *via* Ag—OH bonds forming polymeric sheets, as observed in silver(I) 2,6-dihydroxybenzoate (Mak, Smith & Kennard, 1993). The structure of poly[bis(*p*-nitrosalicylato-*O*,*O*′)disilver(I)-*O*^{3}:*Ag*′;*Ag*:*O*$^{3\prime}$], (I), reported here, also has polymeric bands of OH-cross-linked centrosymmetric Ag dimers.



(I)

The *p*-nitrosalicylato ligand is approximately planar, as can be seen from the torsion angles C2—C1—C7—O2 and O5—N1—C4—C5 of −8 (3) and 8 (3)°, respectively. There is also an intraligand hydrogen bond present [O3—H···O2 2.49 (1) Å; Fig. 1]. The ring planes are parallel to the (100) face, while the Ag—OH bonds lie approximately along the diagonal of the same face of the unit cell (Fig. 2). Bond lengths and angles are comparable with those found for analogous complexes.

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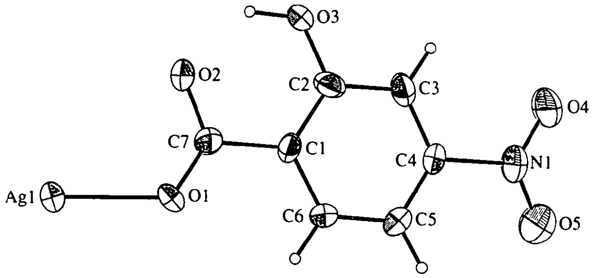

Fig. 1. An *ORTEP*II drawing (Johnson, 1976) of the asymmetric unit of the title compound showing the numbering scheme employed. Displacement ellipsoids are shown at the 50% probability level and H atoms are drawn as small circles of arbitrary radii.

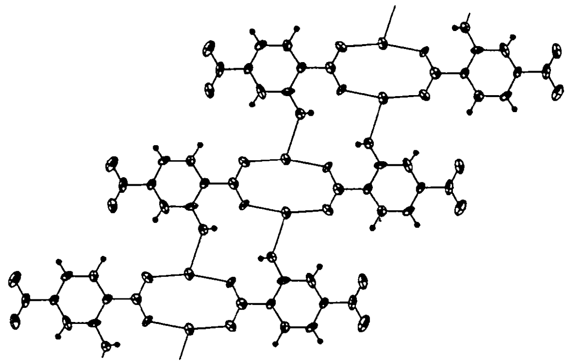

Fig. 2. Silver dimers cross-linked *via* Ag—OH bonds resulting in a polymeric band structure.

Experimental

The title compound was prepared from an aqueous solution of $AgNO_3$ and sodium *p*-nitrosalicylate. The reaction mixture was stored in the dark until crystals appeared.

Crystal data

[$Ag_2(C_7H_4NO_5)_2$]
$M_r = 579.968$
Monoclinic
$P2_1/n$
$a = 3.614\,(1)$ Å
$b = 31.543\,(2)$ Å
$c = 6.750\,(1)$ Å
$\beta = 93.05\,(1)°$
$V = 768.4\,(2)$ Å^3
$Z = 2$
$D_x = 2.51$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15.19–18.83°$
$\mu = 2.62$ mm^{-1}
$T = 295$ K
Needle
0.4 × 0.12 × 0.08 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{min} = 0.705$, $T_{max} = 0.999$
1613 measured reflections
1383 independent reflections

833 observed reflections [$I > 3\sigma(I)$]
$R_{int} = 0.137$
$\theta_{max} = 26°$
$h = -4 \rightarrow 4$
$k = 0 \rightarrow 35$
$l = 0 \rightarrow 8$
3 standard reflections frequency: 120 min intensity decay: 4.4%

Refinement

Refinement on F
$R = 0.054$
$wR = 0.065$
$S = 0.70$
631 reflections
127 parameters
Modified unit-weights: if $F \geq$ threshold then $w = (\text{threshold}/F)^2$, if $F^2 <$ cutoff × $\sigma^2(F)$ then $w = 0$ (where threshold = 24, cutoff = 6.2)

$(\Delta/\sigma)_{max} < 0.0001$
$\Delta\rho_{max} = 1.02$ e Å^{-3}
$\Delta\rho_{min} = -0.77$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Ag1	−0.1814 (5)	−0.02807 (5)	0.1225 (2)	3.61 (3)
O1	−0.016 (4)	−0.0764 (4)	−0.082 (2)	3.5 (3)
O2	0.218 (3)	−0.0309 (4)	−0.295 (2)	3.0 (3)
O3	0.405 (4)	−0.0546 (4)	−0.626 (2)	3.4 (3)
O4	0.580 (5)	−0.1936 (5)	−0.928 (2)	5.7 (4)
O5	0.343 (5)	−0.2382 (5)	−0.731 (3)	7.1 (5)
N1	0.434 (5)	−0.2032 (5)	−0.779 (3)	4.1 (4)
C1	0.210 (5)	−0.1040 (5)	−0.379 (3)	2.5 (4)
C2	0.340 (5)	−0.0946 (6)	−0.565 (3)	2.6 (4)
C3	0.412 (5)	−0.1270 (6)	−0.698 (3)	2.6 (4)
C4	0.349 (5)	−0.1682 (6)	−0.637 (3)	2.7 (4)
C5	0.223 (6)	−0.1784 (6)	−0.455 (3)	3.1 (4)
C6	0.147 (5)	−0.1457 (5)	−0.327 (3)	2.5 (4)
C7	0.124 (5)	−0.0682 (6)	−0.240 (3)	2.6 (4)

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

Ag1···Ag1^i	2.796 (2)	N1—C4	1.51 (3)
Ag1—O1	2.16 (1)	C1—C2	1.40 (3)
Ag1—O2^i	2.20 (1)	C1—C6	1.38 (2)
Ag1—O3^{ii}	2.47 (1)	C1—C7	1.51 (3)
O1—C7	1.23 (2)	C2—C3	1.39 (3)
O2—C7	1.28 (2)	C3—C4	1.38 (3)
O3—C2	1.35 (2)	C4—C5	1.37 (3)
O4—N1	1.20 (2)	C5—C6	1.38 (3)
O5—N1	1.20 (2)		
O1—Ag1—O2^i	163.6 (5)	O3—C2—C1	123 (2)
O1—Ag1—O3^{ii}	113.4 (5)	O3—C2—C3	117 (2)
O2^i—Ag1—O3^{ii}	82.4 (4)	C1—C2—C3	120 (2)
Ag1—O1—C7	123 (2)	C2—C3—C4	117 (2)
Ag1—O2^i—C7^i	127 (2)	N1—C4—C3	117 (2)
Ag1—O3^{ii}—C2^{ii}	130 (2)	N1—C4—C5	119 (2)
O4—N1—O5	127 (2)	C3—C4—C5	124 (2)
O4—N1—C4	118 (2)	C4—C5—C6	118 (2)
O5—N1—C4	116 (2)	C1—C6—C5	121 (2)
C2—C1—C6	120 (2)	O1—C7—O2	125 (2)
C2—C1—C7	119 (2)	O1—C7—C1	119 (2)
C6—C1—C7	121 (2)	O2—C7—C1	116 (2)
Ag1—O1—C7—O2	6 (3)	C2—C1—C7—O2	−8 (3)
O5—N1—C4—C5	8 (3)		

Symmetry codes: (i) $-x, -y, -z$; (ii) $x - 1, y, 1 + z$.

All H atoms were located geometrically at a distance of 0.95 Å from their attached atom and a riding model was used.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Structure solution: Perschar & Schenk (1987). Program(s) used to refine structure: *MolEN LSFM*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *MolEN*.

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under grant DPT/TBAG1 of The Scientific and Technical Research Council of Turkey.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: BM1026). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tricarbonyl(η^6-3-methyl-2-vinylbenzoic acid)chromium

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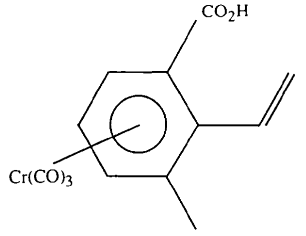
Abstract

The benzylic deprotonation of tricarbonyl(η^6-1-*tert*-butoxycarbonyl-2,3-dimethylbenzene)chromium occurs preferentially at the *ortho* position, whereas the *meta* position is still reactive but to a lesser extent. X-ray study of the title compound, $[Cr(CO)_3(C_{10}H_{10}O_2)]$, obtained in a very good yield, confirms this result and shows a strong hydrogen bond between neighbouring carboxy groups. The acid group and the phenyl ring are coplanar and the torsion angle between the phenyl ring and the vinyl group is 61.2°(4).

Comment

The chemistry and applications of tricarbonyl(η^6-arene)chromium are of great interest (Uemura, 1991). The behaviour of stabilized benzylic anions generated by potassium *tert*-butoxide in the presence of aldehyde and by ^{n}BuLi in the presence of alkyl halide has been analysed (Jaouen, Top, Laconi, Couturier & Brocard, 1984; Blagg, Davies & Mobbs, 1985; Blagg & Davies, 1985). Electronic effects in directing the regioselectivity of benzylic attack were found (Brocard & Lebibi, 1987). In order to study the selectivity of proton abstraction from *ortho* and *meta* benzylic sites relative to an electron-withdrawing group, tricarbonyl(η^6-1-*tert*-butoxycarbonyl-2,3-dimethylbenzene)chromium was reacted at room temperature with potassium *tert*-butoxide in DMSO with aldehyde (H_2—C=O) and affored the *ortho* ethylenic acid tricarbonyl(η^6-3-methyl-2-vinylbenzoic acid)chromium, (I), in 97% yield. The difference in reactivity opens up the possibility of controlled introduction of two different electrophiles (Brocard, Pelinski & Goetgheluck, 1994).



(I)

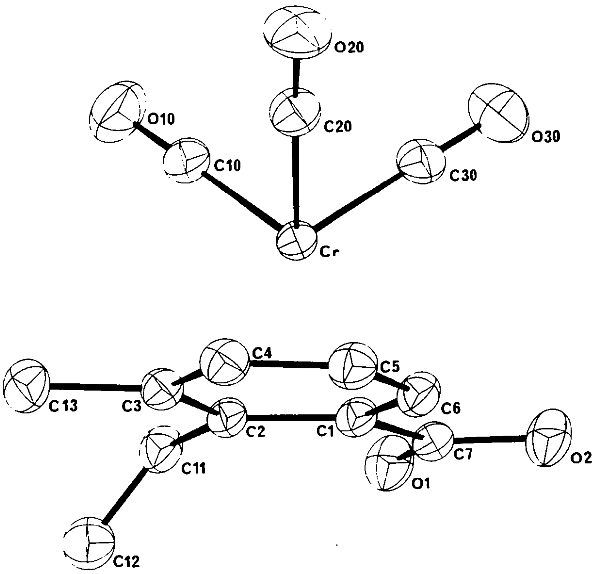


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of the title compound showing the atom-labelling scheme with ellipsoids at the 50% probability level.

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