

Refinement

Refinement on *F**R* = 0.049*wR* = 0.052*S* = 1.35

1161 reflections

145 parameters

H atoms riding

w = 1/*σ*²(*F*) $(\Delta/\sigma)_{\max} = 0.00029$ $\Delta\rho_{\max} = 0.530 \text{ e } \text{Å}^{-3}$ $\Delta\rho_{\min} = -0.383 \text{ e } \text{Å}^{-3}$

Extinction correction: none

Atomic scattering factors

from *International Tables*for *X-ray Crystallography*

(1974, Vol. IV)

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1993).Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *MolEN*. 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.Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Cu1	0.47691 (7)	0.1828 (3)	0.94262 (4)	2.64 (2)
Cl1	1.0022 (2)	0.1002 (7)	0.8715 (1)	4.15 (5)
O1	0.5810 (3)	-0.1429 (15)	0.9789 (2)	2.8 (1)
O2	0.3691 (4)	0.4680 (15)	0.9065 (2)	3.3 (1)
N1	0.5757 (4)	0.1864 (16)	0.8795 (2)	2.0 (1)
C1	0.6807 (5)	-0.109 (2)	0.9543 (3)	2.2 (2)
C2	0.7773 (5)	-0.226 (2)	0.9813 (3)	2.6 (2)
C3	0.8753 (5)	-0.174 (2)	0.9550 (3)	2.9 (2)
C4	0.8771 (5)	0.003 (2)	0.9028 (3)	2.4 (2)
C5	0.7809 (5)	0.122 (2)	0.8737 (3)	2.4 (2)
C6	0.6805 (5)	0.0571 (18)	0.8995 (3)	1.8 (1)
C7	0.5428 (5)	0.2515 (18)	0.8245 (3)	1.9 (1)
C8	0.4409 (5)	0.4158 (19)	0.8118 (3)	2.2 (2)
C9	0.3632 (5)	0.512 (2)	0.8497 (3)	2.3 (2)
C10	0.6091 (5)	0.154 (2)	0.7731 (3)	3.1 (2)
C11	0.2577 (5)	0.684 (2)	0.8290 (3)	3.1 (2)

Table 2. Selected geometric parameters (Å, °)

Cu1...Cu1 ⁱ	2.994 (2)	N1—C6	1.426 (8)
Cu1...Cu1 ⁱⁱ	3.845 (5)	N1—C7	1.319 (8)
Cu1...Cu1 ⁱⁱⁱ	3.597 (2)	C1—C2	1.378 (9)
Cu1—O1 ⁱ	1.950 (4)	C1—C6	1.40 (1)
Cu1...O1 ⁱⁱ	2.986 (6)	C2—C3	1.371 (9)
Cu1...O2 ^{iv}	3.139 (6)	C3—C4	1.37 (1)
Cu1—O1	1.941 (5)	C4—C5	1.40 (1)
Cu1—O2	1.873 (5)	C5—C6	1.400 (9)
Cu1—N1	1.910 (5)	C7—C8	1.412 (9)
Cl1—C4	1.748 (7)	C7—C10	1.495 (9)
O1—C1	1.364 (8)	C8—C9	1.357 (9)
O2—C9	1.300 (8)	C9—C11	1.50 (1)
O1—Cu1—O1 ⁱ	79.4 (2)	C2—C3—C4	119.6 (7)
O2—Cu1—O1 ⁱ	100.0 (2)	Cl1—C4—C5	120.3 (6)
N1—Cu1—O1 ⁱ	161.8 (2)	Cl1—C4—C5	117.7 (6)
Cu1—O1—Cu1 ⁱ	100.6 (2)	C3—C4—C5	122.0 (7)
O1—Cu1—O2	175.6 (3)	C4—C5—C6	118.2 (7)
O1—Cu1—N1	84.4 (2)	N1—C6—C1	114.3 (6)
O2—Cu1—N1	96.8 (2)	N1—C6—C5	126.1 (7)
Cu1—O1—C1	109.9 (5)	C1—C6—C5	118.9 (7)
Cu1—O2—C9	121.4 (5)	N1—C7—C8	120.6 (6)
Cu1—N1—C6	109.9 (4)	N1—C7—C10	122.6 (6)
Cu1—N1—C7	122.4 (5)	C8—C7—C10	116.8 (6)
C6—N1—C7	126.9 (6)	C7—C8—C9	128.5 (7)
O1—C1—C2	123.0 (7)	O2—C9—C8	125.6 (7)
O1—C1—C6	116.0 (6)	O2—C9—C11	112.3 (6)
C2—C1—C6	121.0 (6)	C8—C9—C11	122.1 (7)
C1—C2—C3	120.1 (7)		

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

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically at a distance of 0.95 Å from their parent C atoms, with *B*_{iso}(H) = 1.3*B*_{eq}(C). After refining the coordinates of the methyl H atoms for a few cycles, a riding model was used for all H atoms.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1386). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

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Poly[bis(3,5-dinitrobenzoato-O¹:O²)-disilver(I)-O²:Ag;Ag':O²]

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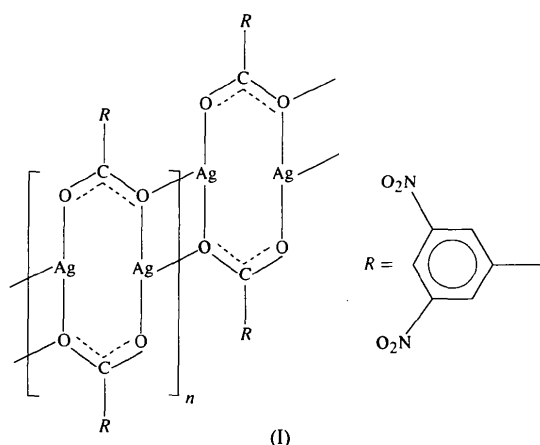
Abstract

The structure of the title compound, [Ag₂(C₇H₃N₂O₆)₂]_n, contains centrosymmetric bis(carboxylato-O,O')-bridged silver dimers, which are cross-linked via Ag—O(carboxyl) bonds [2.655 (7) Å] to form two stair-like polymeric bands. Within the chelate ring, the Ag—O distances are 2.176 (5) and 2.218 (6) Å, and the Ag...Ag distance is 2.835 (2) Å.

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Comment

The results of previously reported studies indicate that silver(I) carboxylate complexes mostly have discrete centrosymmetric dimeric-silver structures. Some of the known examples are silver(I) benzoate and silver(I) 4-hydroxybenzoate (Usubaliev *et al.*, 1981), and silver(I) 2,6-dihydroxybenzoate and silver(I) 2-hydroxybenzoate (Smith, Kennard & Mak, 1988). A second group of examples containing carboxylate-bridged silver dimers are polymeric. The centrosymmetric Ag dimers are cross-linked *via* Ag—OH bonds forming polymeric bands as observed in silver(I) 2,6-hydroxybenzoate (Mak, Smith & Kennard, 1993) and poly[bis(*p*-nitrosalicylato-*O*:*O'*)-disilver(I)-*O*³:Ag';Ag:*O*³] (Tahir, Ülkü & Movsumov, 1996). The structure of poly[bis(3,5-dinitrobenzoato-*O*:*O'*)-disilver(I)-*O*²:Ag;Ag':*O*²], (I), reported here also shows a polymeric nature.



Mean-plane calculations for (I) indicate that both the chelating moiety and the benzene ring of the 3,5-dinitrobenzoate ligand are planar within experimental error; the dihedral angle between them is 13.7(6)°. The nitro groups are not coplanar with their respective ligand ring, but the maximum torsion angle is 8(1)° for O3—N1—C3—C2. The observed bond lengths and angles within the chelating ring are comparable to values observed in related polymeric structures. In the present structure, each Ag atom of the dimer is cross-linked with one of the carboxyl O atoms belonging to a chelate moiety above and below, so that each centrosymmetric dimer has two Ag—O bridges resulting in a polymeric stair-like band structure (Fig. 1). The individual dimers of the polymeric band are shifted relative to one another, so that it has the appearance of stairs, with a step height of approximately 2.527(7) Å and a lateral offset of 2.218(6) Å. The dihedral angle between the bridging plane (Ag1—O2'—Ag1ⁱⁱ—O2ⁱⁱⁱ) and the chelating moiety is 114.16(5)° (symmetry codes are as in Table 2). There are two sets of orientationally different but symmetry-related polymeric bands in the

structure. These bands run parallel to the *a* axis and at an angle of 71.9° to the long axis (C7...C7') of the dimers.

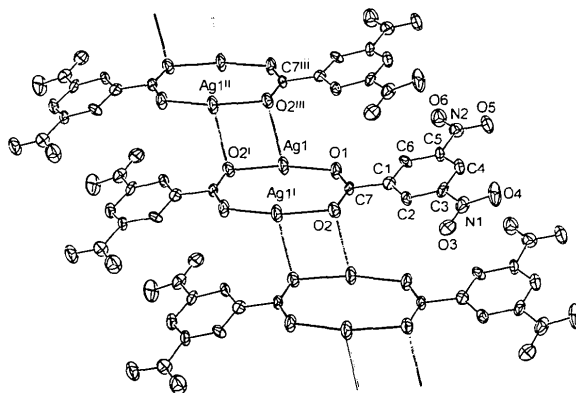


Fig. 1. ORTEP (Johnson, 1976) plot of (I) showing the centrosymmetric carboxylato-*O*,*O'*-bridged dimers linked by further Ag...O contacts to form an infinite stair-like band. The step height from one dimer to the next is 2.527(7) Å and the lateral offset is 2.218(6) Å. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. (See Table 2 for symmetry operations.)

Experimental

The title compound was prepared from an aqueous solution of AgNO₃ and sodium 3,5-dinitrobenzoate, the mixture being kept in the dark until crystals appeared.

Crystal data

[Ag₂(C₇H₃N₂O₆)₂]
M_r = 637.96
 Monoclinic
*P*2₁/*c*
a = 5.428(2) Å
b = 9.747(3) Å
c = 16.115(2) Å
 β = 94.336(4)°
V = 850.2(6) Å³
Z = 2
D_x = 2.492 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 20 reflections
 θ = 6.95–18.24°
 μ = 2.36 mm⁻¹
T = 295 K
 Lath
 0.40 × 0.15 × 0.08 mm
 Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: empirical *via* ψ scans (*MOLLEN*; Fair, 1990)
 T_{\min} = 0.76, T_{\max} = 0.83
 2020 measured reflections
 1632 independent reflections

888 observed reflections
 $[I > 2\sigma(I)]$
 R_{int} = 0.033
 θ_{max} = 26.32°
 h = -6 → 0
 k = 0 → 12
 l = -20 → 20
 3 standard reflections
 frequency: 120 min
 intensity decay: 0.28%

Refinement

Refinement on <i>F</i>	$(\Delta/\sigma)_{\max} = 0.00007$
<i>R</i> = 0.049	$\Delta\rho_{\max} = 0.70 \text{ e } \text{Å}^{-3}$
<i>wR</i> = 0.040	$\Delta\rho_{\min} = -0.67 \text{ e } \text{Å}^{-3}$
<i>S</i> = 1.0	Extinction correction: none
888 reflections	Atomic scattering factors
145 parameters	from <i>International Tables</i>
H atoms: see below	for <i>X-ray Crystallography</i>
<i>w</i> = $[1/\sigma^2(F)]$	(1974, Vol. IV)

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Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq}
Ag1	1.1920 (1)	−0.0784 (1)	0.46845 (4)	3.29 (1)
O1	0.9120 (9)	−0.0906 (8)	0.3648 (3)	2.8 (1)
O2	0.599 (1)	0.0267 (7)	0.4121 (3)	2.8 (1)
O3	−0.082 (1)	0.1523 (7)	0.2097 (4)	3.1 (1)
O4	−0.066 (1)	0.0990 (9)	0.0788 (4)	4.7 (2)
O5	0.557 (1)	−0.2139 (7)	−0.0021 (4)	3.3 (2)
O6	0.864 (1)	−0.2670 (7)	0.0866 (4)	3.6 (2)
N1	0.014 (1)	0.0975 (9)	0.1526 (4)	2.6 (2)
N2	0.665 (1)	−0.2095 (9)	0.0680 (4)	2.8 (2)
C1	0.562 (1)	−0.0565 (9)	0.2728 (5)	2.1 (2)
C2	0.345 (2)	0.0171 (9)	0.2548 (5)	1.9 (2)
C3	0.235 (2)	0.014 (1)	0.1730 (5)	2.1 (2)
C4	0.338 (2)	−0.0597 (9)	0.1111 (5)	2.3 (2)
C5	0.549 (2)	−0.134 (1)	0.1330 (5)	2.1 (2)
C6	0.663 (2)	−0.1302 (9)	0.2118 (5)	2.0 (2)
C7	0.703 (1)	−0.0395 (9)	0.3582 (5)	1.8 (2)

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Bis[*N*-(2,6-dimethylphenyl)amino-glyoximato-*N,N'*]nickel(II)

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Abstract

The title compound consists of discrete centrosymmetric [Ni(C₁₀H₁₂N₃O₂)₂] molecules. The coordination around the Ni atom is square planar, involving four oxime N atoms of two chelating C₂H₂N₂O₂ groups. The oxime O atoms are linked by an intramolecular hydrogen bond [O ··· O 2.546 (2) Å]. The Ni—N distances are 1.891 (2) and 1.846 (2) Å, and the N—Ni—N angle within the NiC₂N₂ chelate ring is 82.75 (8)°. In the central portion of the molecule, the C=N distances are equal [1.297 (3) and 1.298 (3) Å], while the two N—O distances are different [1.303 (3) and 1.386 (3) Å].

Comment

Aminoglyoximes and related ligands are of interest because of their free amino substituents, which gives them the ability to form new ligand systems or metal complexes. The structures of bis(oxamideoximato)-cobalt(II) oxamide oxime (Bekaroğlu, Sarisaban, Koray

Table 2. Selected geometric parameters (Å, °)

Ag1···Ag1 ⁱ	2.835 (2)	Ag1—O2 ⁱ	2.218 (6)
Ag1···Ag1 ⁱⁱⁱ	3.746 (2)	Ag1—O2 ⁱⁱⁱ	2.655 (7)
Ag1—O1	2.176 (5)		
Ag1—O1—C7	128.9 (5)	O1—Ag1—O2 ⁱⁱⁱ	108.0 (2)
Ag1 ⁱ —O2—C7	119.4 (5)	O2 ⁱ —Ag1—O2 ⁱⁱⁱ	79.9 (2)
Ag1—O2 ⁱⁱⁱ —C7 ⁱⁱⁱ	118.07 (5)	Ag1—O2 ⁱ —Ag1 ⁱ	100.1 (3)
O1—Ag1—O2 ⁱ	164.0 (2)		

Symmetry codes: (i) 2−*x*, −*y*, 1−*z*; (ii) 3−*x*, −*y*, 1−*z*; (iii) 1+*x*, *y*, *z*.

All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their corresponding C atoms. A riding model was used for all H atoms, with *U*_{iso}(H) = 1.3*U*_{eq}(C).

Data collection: *CAD-4 Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIMPEL* in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEPII* (Johnson, 1976) in *MolEN*. 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 and complete geometry have been deposited with the IUCr (Reference: BM1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.