

almost linear Ru—carbonyl groups are similar to those found in the above-mentioned clusters. The bridged carbonyls lie within 0.08 Å of the equatorial Ru(2),-Ru(3),Ru(4),Ru(5) plane and are adjacent to the long Ru(3)—Ru(4) bond. Both are semi-bridged with a shorter Ru—C value of 1.801 (29) and 1.964 (54) Å (ave. 1.88 Å) and a longer one of 2.193 (26) and 2.519 (6) Å (ave. 2.35 Å). Bridging Ru—C—O angles range from 121.0 (2.0) to 150.5 (60)°. Bond distances and angles within the [As(Ph)<sub>4</sub>]<sup>+</sup> counterion are normal.

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## Structure of Bis[ $\mu$ -( $\beta$ -alanine)-O,O']-disilver(I) Dinitrate, [Ag<sub>2</sub>(C<sub>3</sub>H<sub>7</sub>NO<sub>2</sub>)<sub>2</sub>](NO<sub>3</sub>)<sub>2</sub>

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**Abstract.**  $M_r = 517.932$ , monoclinic,  $P2_1/n$ ,  $a = 6.656$  (5),  $b = 8.280$  (5),  $c = 12.975$  (4) Å,  $\beta = 94.90$  (3)°,  $V = 712.58$  (20) Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 2.382$ ,  $D_x = 2.414$  Mg m<sup>-3</sup>, Cu  $K\alpha$ ,  $\lambda = 1.54051$  Å,  $\mu = 23.3$  mm<sup>-1</sup>,  $F(000) = 504$ ,  $T = 297$  K,  $R = 0.0888$  for 545 observed reflexions. The centrosymmetric dimers have an Ag—Ag separation 2.855 (4) Å. The Ag—O bond distances are 2.210 (19) and 2.198 (19) Å. The nitrate ion is bonded to the alanine moiety by two hydrogen bonds of the N—H...O type: N...O 3.09 (3) and 2.96 (3) Å. A pair of oxygen atoms from two adjacent nitrate ions are involved in weak covalent bonds with Ag, at distances of 2.58 (3) and 2.57 (3) Å.

**Introduction.** Alanine residues have been the subject of intensive study because of the variety of conformations in which alanine is found to be bonded in certain peptides, appearing, for instance, repetitively in the

form of an Ala-Pro sequence over a segment of the light chain of rabbit skeletal muscle myosin (Kamwaya, Oster, Bradaczek, Ponnuswamy, Parthasarathy, Naraj & Balaram, 1982) and because of some interesting physical properties in complexes of metallic salts with amino acids (Tomita, 1961; Jose, Pant & Biswas, 1964; Jose & Pant, 1965; Herak, Prelesnik, Manojlović-Muir & Muir, 1974). Further, it has been observed that certain derivatives of  $\beta$ -alanine with Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O form a bacteriocidal composition against *Bacillus subtilis* (Aeloney, 1982).

**Experimental.** Silver nitrate and  $\beta$ -alanine taken in stoichiometric proportion and dissolved in distilled water, filtered and then allowed to evaporate slowly; crystals slightly photosensitive; density determined by flotation in bromoform-xylene; preliminary examination by oscillation and Weissenberg photographs;

Table 1. Atomic coordinates and equivalent isotropic temperature factors

$B_{eq}$  calculated as given by Willis & Pryor (1975).

	x	y	z	$B_{eq}(\text{\AA}^2)$
Ag	0.2990 (3)	0.5614 (2)	0.4879 (2)	6.3 (1)
O(1)	0.277 (3)	0.330 (2)	0.4042 (16)	5.9 (1)
O(2)	0.579 (3)	0.230 (2)	0.4205 (19)	7.5 (2)
C(1)	0.396 (4)	0.221 (3)	0.398 (2)	4.5 (9)
C(2)	0.315 (4)	0.065 (4)	0.348 (2)	7.3 (9)
C(3)	0.125 (5)	0.089 (3)	0.282 (2)	5.0 (1)
N(1)	-0.049 (3)	0.133 (3)	0.3385 (20)	6.4 (9)
O(3)	0.114 (4)	0.428 (3)	0.632 (3)	10.7 (9)
N(2)	0.105 (3)	0.281 (3)	0.623 (2)	6.8 (9)
O(4)	0.231 (3)	0.193 (3)	0.668 (3)	10.8 (9)
O(5)	-0.033 (3)	0.224 (3)	0.570 (2)	8.3 (9)

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

Ag-O(1)-C(1)	131.8 (17)	C(1)-C(2)-C(3)	113 (2)
O(1)-Ag-O(2)	161.6 (8)	C(2)-C(3)-N(1)	114 (3)
O(1)-C(1)-O(2)	125 (2)	O(3)-N(2)-O(4)	122 (3)
O(1)-C(1)-C(2)	117 (2)	O(4)-N(2)-O(5)	120 (3)
O(2)-C(1)-C(2)	118 (2)	O(5)-N(2)-O(3)	118 (3)
Ag-Ag'	2.855 (4)	C(1)-C(2)	1.52 (4)
Ag-O(1)	2.198 (19)	C(2)-C(3)	1.48 (4)
Ag-O(2)	2.210 (19)	C(3)-N(1)	1.47 (3)
Ag-O(3)	2.58 (3)	N(2)-O(3)	1.22 (3)
Ag-O(5 <sup>h</sup> )	2.572 (19)	N(2)-O(4)	1.22 (3)
O(1)-C(1)	1.22 (3)	N(2)-O(5)	1.20 (3)
C(1)-O(2)	1.23 (3)		
Ag-O(1)-C(1)-C(2)	169.02 (2)		
Ag-O(2)-C(1)-C(2)	-177.34 (8)		
O(1)-C(1)-C(2)-C(3)	21.44 (3)		
O(2)-C(1)-C(2)-C(3)	-152.86 (2)		
C(1)-C(2)-C(3)-N(1)	-68.24 (8)		
O(3)-O(5)-N(2)-O(4)	179.07 (7)		

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

reflexions  $0k0$  absent when  $k$  odd,  $h0l$  when  $h+l$  odd; intensity data collected from crystal of size  $0.35 \times 0.3 \times 0.3$  mm, Enraf-Nonius CAD-4 diffractometer at the Indian Institute of Technology, Madras; Cu  $K\alpha$  radiation, graphite monochromator; cell dimensions determined from 24 high-angle reflexions and refined by least-squares analysis; intensity data for 1527 independent reflexions collected,  $2\theta < 120^\circ$ ; only 545 reflexions used in analysis. The criterion used to eliminate weak reflexions was that if the background intensity (left background or right background) was equal to 50% of the peak intensity, then these reflexions were treated as weak; two standard reflections monitored after every 100 reflexions, some deterioration observed, probably due to photosensitive nature of crystal, index range  $h \pm 7, k 0-9, l 0-8$ ; absorption correction applied, assuming spherical crystal,  $\mu_r = 3.5$ , transmission factor range 13.5-41.9; structure solved by Patterson and Fourier methods; refinement (on  $F$ ) performed at the University of Science, Penang, Malaysia, using the IBM 370/148 computer by least squares with *SHELX76* (Sheldrick, 1976); anisotropic non-H atoms; H atoms fixed in ideal positions with bond lengths of

1.08  $\text{\AA}$ ; max. and min. electron densities in final difference synthesis 0.89 and  $-0.87 e \text{\AA}^{-3}$ , max.  $\Delta/\sigma = 0.005$ , unit weights; atomic scattering factors from *SHELX*. Positional and thermal parameters are given in Table 1,\* bond angles, selected torsion angles and interatomic lengths in Table 2. Fig. 1 is a view of the dimeric molecule and Fig. 2 shows the packing in the unit cell along  $c$ .

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39388 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

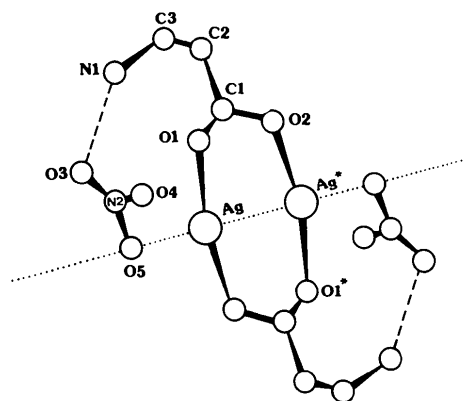


Fig. 1. The dimer of bis[ $\mu$ -( $\beta$ -alanine)-O,O']-disilver dinitrate showing the atomic numbering.

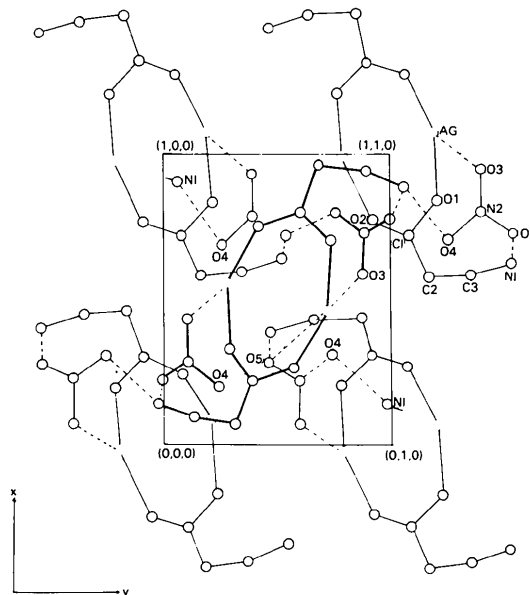


Fig. 2. Packing in the unit cell along  $c$ . The nitrate ion is bonded to the alanine moiety by two hydrogen bonds of type N-H...O: O(4)...N(1) = 2.96 (3)  $\text{\AA}$  (symmetry position  $-x, -y, 1-z$ ) and O(5)...N(1) = 3.09 (3)  $\text{\AA}$ .

**Discussion.** Two alanine groups and two  $\text{Ag}^+$  ions form a centrosymmetric dimer. Within this dimer the Ag–Ag separation of 2.855 (4) Å is comparable to the smallest distance of 2.88 Å found in metallic silver (Griffith, 1943). The nitrate groups are in a *trans* configuration. The Ag–O bond distances are 2.210 (19) and 2.198 (19) Å, which are comparable to those reported, respectively, for silver perfluorobutyrate and silver oxalate (Blakeslee & Hoard, 1956; Griffith, 1943). The O–Ag–O angle is 161.6 (8)°, which is comparable to the usual value of 160–163° (Blakeslee & Hoard, 1956; Rao & Viswamitra, 1972). Two independent Ag–O distances to the nitro groups are 2.58 (3) and 2.57 (3) Å. There are hydrogen bonds of type N–H...O between the alanine and the nitro groups which stabilize the crystal packing.

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### Acetato(triphenylphosphine)gold(I), [Au(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)(C<sub>18</sub>H<sub>15</sub>P)]\*

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**Abstract.**  $M_r = 518.3$ , orthorhombic,  $P2_12_12_1$ ,  $a = 11.088$  (3),  $b = 12.050$  (4),  $c = 13.839$  (5) Å,  $V = 1849$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.862$  (1) Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu(\text{Mo } K\alpha) = 8.0$  mm<sup>-1</sup>,  $F(000) = 992$ ,  $R = 0.028$ , 3055 reflections. Isostructural with  $\text{Ph}_3\text{PAuCl}$  and  $\text{Ph}_3\text{AsAuBr}$ . The Au–O bond length is 2.063 (6) Å, with a short Au–P bond of 2.207 (3) Å. The second acetate O is not involved in bonding at the metal [Au...O 2.93 (1) Å], which thus shows a linear coordination geometry [P–Au–O 177.3 (2)°].

**Introduction.** We have begun a systematic study of gold carboxylate complexes and here report the structure of  $\text{Ph}_3\text{PAuOCOCH}_3$ , (1). This is one of the few gold carboxylate complexes to have been chemically characterized (Nichols & Charleston, 1969). Colourless

prisms were obtained by diffusion of petroleum ether (40°–60° C) into a dichloromethane solution of (1).

**Experimental.**  $D_m$  not determined. Crystal 0.6 × 0.25 × 0.1 mm, elongated along **b**. 3247 profile-fitted intensities (Clegg, 1981) recorded on a Stoe–Siemens four-circle diffractometer. Monochromated Mo  $K\alpha$  radiation.  $2\theta_{\text{max}} 50^\circ$ , octants  $hkl$  and  $\bar{h}\bar{k}\bar{l}$  (no equivalents). Three standard reflections, no intensity change.  $L_p$  and empirical absorption corrections ( $\psi$  scans; transmissions 0.61–0.96). 3055 reflections with  $F > 4\sigma(F)$  used for all calculations (program system *SHELXTL*, Sheldrick, 1978). Cell constants refined from  $2\theta$  values of 48 reflections in the range 20–24°. Structure solution by heavy-atom method. Refinement on  $F$  to  $R$  0.028,  $R_w$  0.028 [all non-H atoms anisotropic; phenyl H atoms using riding model with C–H 0.96 Å, H on external bisector of appropriate C–C–C angle; methyl H not included: 217

\* Carboxylate and Related Complexes of Gold. 2. Part 1: Jones (1984).