

151 (2)°. Chaque molécule se trouve ainsi unie à quatre molécules voisines. Il semble que l'on puisse admettre aussi l'existence de la liaison hydrogène C(8)—H'(8)...Cl(2ⁱⁱⁱ) [3,496 (2) Å, 136 (2)°] (iii: $-\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$).

La Fig. 2 est une vue stéréoscopique de la structure.

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Diphenylbismuth *N*-Benzoylglycinate

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Abstract. C₂₁H₁₈BiNO₃, *M_r* = 541.36, monoclinic, *P*2₁/*c*, *a* = 11.078 (7), *b* = 9.869 (7), *c* = 17.945 (8) Å, β = 107.27 (5)°, *V* = 1873.5 Å³, *Z* = 4, *D_x* = 1.919 Mg m⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 9.4 mm⁻¹, *F*(000) = 1032, *T* = 291 (1) K, final *R* = 0.023 for 2142 unique observed [*F* ≥ 4.0σ(*F*)] diffractometer data. Bi is in the center of a distorted ψ-trigonal bipyramid, two O atoms, one from each bridging bidentate carboxylate group [Bi—O distance, mean: 2.440 (6) Å; angle O—Bi—O: 160.63 (2)°], being in apical positions; two C(phenyl) atoms [Bi—C distance, mean: 2.248 (8) Å; angle C—Bi—C: 95.10 (3)°] are arranged equatorially leaving ample space for the inert electron pair. In addition, weak interactions exist between Bi and O(benzoyl) from an adjacent molecule [3.267 (6) Å] and an O from one of the carboxylate groups [3.297 (6) Å].

Introduction. The structural chemistry of organobismuth(III) carboxylates has not yet been studied extensively. Actually, only the structure of the complex anion [Ph₂Bi(O₂CCF₃)₂]⁻ (Barton, Charpiot, Dau, Motherwell, Pascard & Pichon, 1984) has been reported; no common diorganobismuth(III) carboxylates have been described. To gain a better understanding (i) of the coordination behavior of bismuth in

such compounds, and (ii) of the rules governing the choice of a certain bonding mode (unidentate, bridging, chelation) of the carboxylate group of an amino acid or of an amino-acid derivative to bismuth and also the possible formation of weak inter- or intramolecular bonding interactions by such oligofunctional donor ligands, we extended our investigations on organometal compounds with biologically relevant ligands to study appropriate bismuth compounds. As a first result we report here the structure of Ph₂BiO₂CCH₂NHCOC₆H₅.

Experimental. From Ph₂BiOEt and *N*-benzoylglycine in diethyl ether at room temperature. Single crystals from methanol. Crystal size ~ 0.4 × 0.3 × 0.2 mm. ω/2θ scan, scan speed 4.0° min⁻¹ in θ, Nonius CAD-4 diffractometer, graphite-monochromated Mo *K*α; lattice parameters from least-squares fit with 25 reflections up to 2θ = 27.0°; three standard reflections recorded every 2.0 h, only random deviations; 2936 reflections measured, 1.0 ≤ θ ≤ 23.0°, -12 ≤ *h* ≤ 12, 0 ≤ *k* ≤ 10, 0 ≤ *l* ≤ 19; after averaging (*R*_{int} = 0.014): 2786 unique reflections, 2142 with *F* ≥ 4.0σ(*F*); Lorentz-polarization correction and absorption correction *via* ψ scans; max./min. transmission 1.00/0.73; systematic absences (*h*0*l*) *l* = 2*n* + 1, (0*k*0) *k* = 2*n* + 1 conform to space group *P*2₁/*c*; structure solution *via*

Patterson function, ΔF syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic temperature factor for H atoms, which were placed in geometrically calculated positions (C-H 0.95 Å); refinement on F with 2142 reflections and 236 refined parameters; $w = 1/\sigma(F^2)$, $S = 3.4$, $R = 0.023$, $wR = 0.035$, $(\Delta/\sigma)_{\max} = 0.05$, no extinction correction; largest peak in final ΔF map $\pm 0.7(3) e \text{ \AA}^{-3}$, atomic scattering factors for neutral atoms and real and imaginary dispersion terms from *International Tables for X-ray Crystallography* (1974); programs: Enraf-Nonius *SDP-Plus* (Frenz, 1985), *SHELXTL-Plus* (Sheldrick, 1987), *POP1* (van de Waal, 1976).

Discussion. The structure of the title compound and the numbering scheme are shown in Fig. 1 and a stereoscopic view in Fig. 2. Positional parameters and

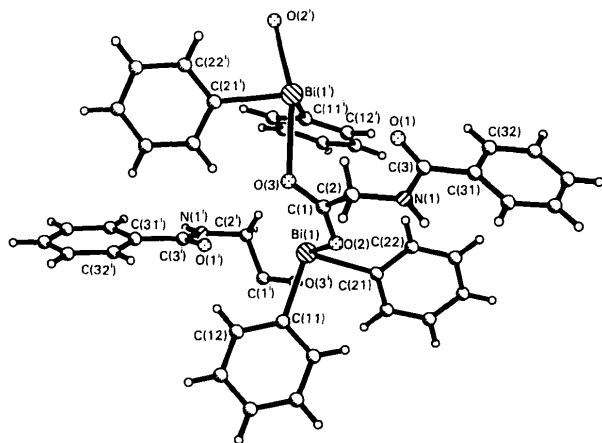


Fig. 1. General view of the molecule, showing the atom-numbering scheme. [i] corresponds to the symmetry operation: $2-x, -0.5+y, 1.5-z$.]

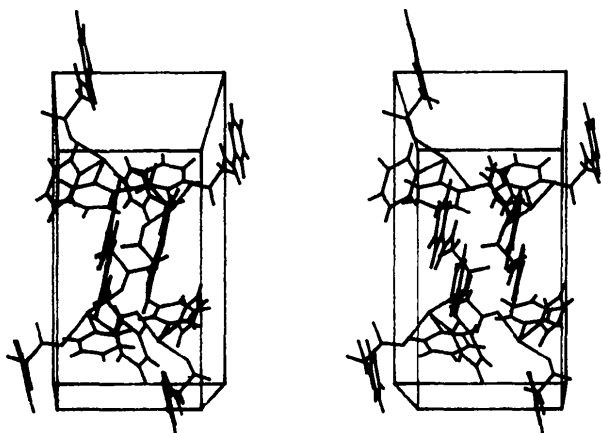


Fig. 2. Stereoscopic view of the unit cell, viewed down b .

Table 1. Atomic coordinates and equivalent isotropic thermal parameters ($\text{\AA}^2 \times 10^3$)

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

	x	y	z	U_{eq}
Bi(1)	0.97101 (3)	0.20346 (3)	0.76681 (1)	25
O(1)	0.8408 (7)	-0.2356 (8)	0.8391 (4)	53
O(2)	1.0162 (6)	0.0570 (6)	0.8782 (3)	29
O(3)	1.1011 (6)	-0.0959 (6)	0.8183 (3)	35
N(1)	0.9423 (7)	-0.1573 (8)	0.9580 (4)	29
C(1)	1.0572 (8)	-0.0604 (9)	0.8722 (5)	27
C(2)	1.0597 (9)	-0.1589 (9)	0.9382 (5)	30
C(3)	0.8367 (9)	-0.197 (1)	0.9036 (5)	34
C(11)	1.1010 (8)	0.3525 (9)	0.8466 (5)	28
C(12)	1.2112 (8)	0.392 (1)	0.8312 (5)	34
C(13)	1.2946 (9)	0.480 (1)	0.8802 (7)	47
C(14)	1.270 (1)	0.525 (1)	0.9466 (7)	49
C(15)	1.163 (1)	0.487 (1)	0.9637 (6)	43
C(16)	1.0771 (9)	0.402 (1)	0.9140 (5)	37
C(21)	0.8026 (8)	0.253 (1)	0.8071 (5)	29
C(22)	0.734 (1)	0.146 (1)	0.8243 (6)	39
C(23)	0.628 (1)	0.170 (1)	0.8478 (7)	53
C(24)	0.5883 (9)	0.301 (1)	0.8515 (6)	54
C(25)	0.653 (1)	0.408 (1)	0.8337 (6)	47
C(26)	0.7602 (9)	0.386 (1)	0.8112 (6)	38
C(31)	0.7143 (9)	-0.189 (1)	0.9215 (5)	34
C(32)	0.605 (1)	-0.210 (1)	0.8607 (6)	53
C(33)	0.487 (1)	-0.203 (1)	0.8725 (8)	68
C(34)	0.479 (1)	-0.175 (1)	0.9461 (8)	63
C(35)	0.587 (1)	-0.154 (1)	1.0064 (6)	47
C(36)	0.7033 (9)	-0.161 (1)	0.9946 (6)	38

Table 2. Bond distances (\AA) and selected bond angles ($^\circ$)

Bi(1)-C(21)	2.246 (7)	C(1)-O(3)	1.26 (1)
Bi(1)-C(11)	2.250 (8)	C(1)-C(2)	1.53 (1)
Bi(1)-O(2)	2.396 (6)	C(2)-N(1)	1.445 (9)
Bi(1)-O(3')	2.484 (6)	N(1)-C(3)	1.34 (1)
Bi(1)-O(3)	3.297 (6)	C(3)-O(1)	1.23 (1)
Bi(1)-O(1')	3.267 (6)	C(3)-C(31)	1.49 (1)
C(1)-O(2)	1.26 (1)		
C(21)-Bi(1)-C(11)	95.10 (3)	O(2)-Bi(1)-O(3')	160.63 (2)
C(21)-Bi(1)-O(2)	81.38 (3)	O(2)-C(1)-O(3)	123.61 (8)
C(11)-Bi(1)-O(3')	83.02 (3)	O(2)-C(1)-C(2)	116.31 (7)
C(11)-Bi(1)-O(2)	85.85 (3)	O(3)-C(1)-C(2)	119.96 (8)
C(21)-Bi(1)-O(3)	84.16 (3)		

the equivalent values of the anisotropic temperature factors for the non-H atoms are given in Table 1.* Bond lengths and angles are given in Table 2.

Bidentate bridging carboxylate groups link Ph_2Bi units to form a one-dimensional polymer. Bi is in the center of a heavily distorted ψ -trigonal bipyramid, O(2) and O(3') being in apical positions, while C(11) and C(21) are arranged equatorially leaving ample space for a stereochemically active inert pair. The distances

* Lists of H-atom coordinates, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44705 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(1)—O(2,3) are not significantly different, indicating equivalent bonding quality in the carboxylate bridge unit. However, Bi(1)—O(2) and Bi(1)—O(3ⁱ) show different values, though both correspond to bond lengths in bismuth(III) formate with values 2.34 to 2.56 Å which shows similar carboxylate bridging (Stålhandske, 1969). The greater distance Bi(1)—O(3ⁱ) can be correlated with the weak interaction of O(3ⁱ) and the Bi atom of the adjacent molecule [corresponding atoms in Fig. 1: O(3) and Bi(1)] [3.297 (6) Å]. A similar weak interaction Bi(1)—O(1ⁱ) can be inferred from the distance 3.267 (6) Å. Both values are distinctly smaller than the sum of the van der Waals radii which is presumably greater than 3.60 Å. These interactions would formally extend the coordination number of Bi to 7.

The coordination behavior of the *N*-benzoylglycinate ligand of the title compound seems to be similar to that proposed for triorganotin(IV) and triorgano-

lead(IV) *N*-benzoylglycinates (Roge, Huber, Silvestri & Barbieri, 1982).

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Bis[2-furoato(1–)]triphenylbismuth(V)

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Abstract. [Bi(C₅H₃O₃)₂(C₆H₅)₃], *M*_r = 662.46, monoclinic, *P*2₁/*n*, *a* = 8.915 (6), *b* = 17.241 (12), *c* = 15.774 (7) Å, β = 93.00 (5)°, *V* = 2421.2 Å³, *Z* = 4, *D*_x = 1.817 Mg m⁻³, λ(Ag *K*α) = 0.5608 Å, μ = 2.2 mm⁻¹, *F*(000) = 1280, *T* = 291 (1) K, final *R* = 0.024 for 3256 unique observed diffractometer data and 317 variables. In the molecule each of the two carboxylic ligands is bound to the central Bi atom by one short [mean 2.296 (4) Å] and one weak Bi—O bond [mean 2.806 (4) Å]. These four O atoms (the two weakly bonded being *cis*) and one C(phenyl) atom can be considered to form the equatorial plane of a distorted pentagonal bipyramid around Bi, two of the C(phenyl) atoms being arranged in apical positions. The O(furan) atom is not involved in coordination.

Introduction. The possibility that the carboxylate group responds specifically to the electronic and steric conditions at a metal center with different modes of bonding is an interesting field of research. We are studying in this context organometal group V derivatives of mainly oligofunctional carboxylic acids, and

we report here as a first example of a triorganobismuth dicarboxylate the structure of the title compound.

Experimental. Prepared from Ph₃BiCO₃ and 2-furan-carboxylic acid in CHCl₃/methanol. Single crystals from ethanol. Crystal size 0.38 × 0.29 × 0.32 mm, ω/2θ scan, scan speed 2.5–6.7° min⁻¹ in θ, Nonius CAD-4 diffractometer, graphite-monochromated Ag *K*α; lattice parameters from least-squares fit with 25 reflections up to 2θ = 24.7°; five standard reflections recorded every 2.5 h showed decay of 8.6% during data collection; 9719 reflections measured; 1 ≤ θ ≤ 20°, -10 ≤ *h* ≤ 10, 0 ≤ *k* ≤ 21, -19 ≤ *l* ≤ 19; after averaging (*R*_{int} = 0.017) 4767 unique reflections obtained, 3256 with *I* > 1.96σ(*I*); Lorentz–polarization correction, absorption correction *via* ψ scans and decay correction, min., max. transmission coefficient 0.83, 1.00; systematic absences *h*0*l*, *h*+*l* = 2*n* + 1; 0*k*0, *k* = 2*n* + 1, space group *P*2₁/*n*; structure solution *via* direct methods, Δ*F* syntheses and full-matrix least-squares refinement with anisotropic temperature factors for all non-H atoms and a common isotropic tempera-