

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(3<sup>i</sup>) 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(3<sup>i</sup>) can be correlated with the weak interaction of O(3<sup>i</sup>) 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(1<sup>i</sup>) 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(C<sub>5</sub>H<sub>3</sub>O<sub>3</sub>)<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>], *M*<sub>r</sub> = 662.46, monoclinic, *P*2<sub>1</sub>/*n*, *a* = 8.915 (6), *b* = 17.241 (12), *c* = 15.774 (7) Å, β = 93.00 (5)°, *V* = 2421.2 Å<sup>3</sup>, *Z* = 4, *D*<sub>x</sub> = 1.817 Mg m<sup>-3</sup>, λ(Ag *K*α) = 0.5608 Å, μ = 2.2 mm<sup>-1</sup>, *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 Ph<sub>3</sub>BiCO<sub>3</sub> and 2-furan-carboxylic acid in CHCl<sub>3</sub>/methanol. Single crystals from ethanol. Crystal size 0.38 × 0.29 × 0.32 mm, ω/2θ scan, scan speed 2.5–6.7° min<sup>-1</sup> 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*<sub>int</sub> = 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*2<sub>1</sub>/*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-

Table 1. Atomic coordinates and equivalent thermal parameters ( $\text{\AA}^2 \times 10^3$ )
$$U_{eq} = (1/6\pi^2) \sum_i \sum_j \beta_{ij} a_i \cdot a_j$$

	x	y	z	$U_{eq}$
Bi(1)	0.40641 (2)	-0.34490 (1)	0.25301 (1)	34
O(1)	0.5663 (4)	-0.2597 (2)	0.3245 (2)	41
O(2)	0.6685 (4)	-0.2747 (3)	0.1999 (2)	50
O(3)	0.8015 (4)	-0.1749 (2)	0.3852 (2)	50
O(4)	0.2446 (4)	-0.4365 (2)	0.1962 (2)	49
O(5)	0.4139 (4)	-0.4273 (2)	0.0994 (2)	48
O(6)	0.0966 (4)	-0.5499 (2)	0.1104 (2)	45
C(1)	0.6733 (6)	-0.2497 (3)	0.2730 (3)	38
C(2)	0.8035 (6)	-0.2059 (3)	0.3057 (3)	38
C(3)	0.9359 (7)	-0.1389 (4)	0.3987 (4)	62
C(4)	1.0209 (7)	-0.1478 (4)	0.3331 (4)	68
C(5)	0.9371 (6)	-0.1905 (4)	0.2730 (4)	54
C(6)	0.2992 (6)	-0.4575 (3)	0.1260 (3)	39
C(7)	0.2196 (5)	-0.5166 (3)	0.0765 (3)	35
C(8)	0.0408 (7)	-0.6003 (4)	0.0502 (4)	57
C(9)	0.1237 (7)	-0.5988 (4)	-0.0176 (4)	59
C(10)	0.2400 (6)	-0.5457 (4)	-0.0010 (3)	47
C(11)	0.3206 (5)	-0.2508 (3)	0.1690 (3)	39
C(12)	0.3305 (7)	-0.1771 (4)	0.2035 (4)	50
C(13)	0.2722 (7)	-0.1157 (4)	0.1564 (4)	60
C(14)	0.2073 (7)	-0.1289 (4)	0.0765 (4)	62
C(15)	0.1982 (7)	-0.2013 (4)	0.0436 (4)	60
C(16)	0.2544 (6)	-0.2641 (4)	0.0896 (4)	51
C(21)	0.2623 (5)	-0.3414 (4)	0.3632 (3)	39
C(22)	0.3080 (6)	-0.2998 (4)	0.4339 (4)	50
C(23)	0.2142 (7)	-0.2941 (4)	0.5008 (4)	58
C(24)	0.0786 (7)	-0.3298 (4)	0.4963 (4)	60
C(25)	0.0331 (7)	-0.3700 (5)	0.4263 (4)	87
C(26)	0.1262 (7)	-0.3768 (5)	0.3584 (4)	71
C(31)	0.5783 (6)	-0.4336 (3)	0.2810 (3)	41
C(32)	0.5652 (7)	-0.4742 (4)	0.3552 (4)	57
C(33)	0.6723 (8)	-0.5276 (5)	0.3783 (4)	73
C(34)	0.7892 (7)	-0.5415 (4)	0.3281 (5)	70
C(35)	0.8013 (7)	-0.5013 (4)	0.2540 (4)	69
C(36)	0.6953 (7)	-0.4470 (4)	0.2288 (4)	58

ture factor for H atoms, which were placed in geometrically calculated positions (C-H 0.95 Å); refinement on  $F$  with 3256 reflections and 317 refined parameters;  $w = 4F_o^2 / [\sigma^2(F_o^2) + (0.025F_o^2)^2]$ ;  $S = 1.19$ ,  $R = 0.024$ ,  $wR = 0.025$ ,  $(\Delta/\sigma)_{\max} = 0.01$ ; no extinction correction; largest peak in final  $\Delta F$  map  $\pm 0.9 (3) e \text{\AA}^{-3}$ ; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: Enraf-Nonius *Structure Determination Package* (Frenz, 1981), *ORTEPII* (Johnson, 1976), *MULTAN80* (Main *et al.*, 1980), *POP1* (van de Waal, 1976).

**Discussion.** The structure of the title compound is shown in Fig. 1 and in a stereoview in Fig. 2. Positional parameters and equivalent values of the anisotropic temperature factors are given in Table 1,\* bond lengths and angles in Table 2.

The unit cell contains four molecules. The atoms O(1), O(4), C(11), C(21) and C(31) can be considered

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44683 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

Bi(1)-C(11)	2.206 (5)	C(2)-O(3)	1.364 (6)
Bi(1)-C(21)	2.215 (5)	O(3)-C(3)	1.356 (7)
Bi(1)-C(31)	2.193 (5)	C(3)-C(4)	1.323 (9)
Bi(1)-O(1)	2.301 (3)	C(4)-C(5)	1.387 (9)
Bi(1)-O(2)	2.799 (4)	C(5)-C(2)	1.348 (7)
Bi(1)-O(4)	2.290 (4)	C(6)-C(7)	1.447 (7)
Bi(1)-O(5)	2.813 (4)	C(7)-O(6)	1.370 (6)
O(1)-C(1)	1.297 (6)	O(6)-C(8)	1.362 (7)
O(2)-C(1)	1.230 (6)	C(8)-C(9)	1.332 (9)
O(4)-C(6)	1.285 (6)	C(9)-C(10)	1.398 (9)
O(5)-C(6)	1.241 (6)	C(10)-C(7)	1.343 (7)
C(1)-C(2)	1.457 (7)		
C(11)-Bi(1)-C(21)	104.73 (2)	O(2)-C(1)-C(2)	120.08 (4)
C(11)-Bi(1)-C(31)	148.23 (2)	O(4)-C(6)-C(7)	117.83 (4)
C(21)-Bi(1)-C(31)	106.79 (2)	O(5)-C(6)-C(7)	120.19 (5)
O(1)-Bi(1)-O(4)	173.55 (1)	C(1)-C(2)-C(5)	132.11 (5)
C(11)-Bi(1)-O(1)	90.66 (2)	C(1)-C(2)-O(3)	118.65 (4)
C(11)-Bi(1)-O(4)	94.63 (2)	C(2)-C(5)-C(4)	107.37 (5)
C(21)-Bi(1)-O(1)	88.18 (2)	C(5)-C(4)-C(3)	106.60 (5)
C(21)-Bi(1)-O(4)	86.91 (2)	C(4)-C(3)-O(3)	111.15 (5)
C(31)-Bi(1)-O(1)	86.45 (2)	O(3)-C(2)-C(5)	109.19 (5)
C(31)-Bi(1)-O(4)	90.97 (2)	C(2)-O(3)-C(3)	105.66 (4)
O(1)-Bi(1)-O(2)	50.58 (1)	C(6)-C(7)-C(10)	131.98 (5)
O(2)-Bi(1)-O(5)	84.40 (1)	C(6)-C(7)-O(6)	117.63 (4)
O(4)-Bi(1)-O(5)	50.03 (1)	C(7)-C(10)-C(9)	106.31 (5)
Bi(1)-O(1)-C(1)	103.58 (3)	C(10)-C(9)-C(8)	107.42 (5)
O(1)-C(1)-O(2)	123.44 (5)	C(9)-C(8)-O(6)	110.47 (5)
Bi(1)-O(4)-C(6)	105.81 (3)	O(6)-C(7)-C(10)	110.36 (5)
O(4)-C(6)-O(5)	121.96 (5)	C(7)-O(6)-C(8)	105.43 (4)
O(1)-C(1)-C(2)	116.48 (4)		

The C-C bond lengths and C-C-C angles in the phenyl groups range from 1.349 (9) to 1.393 (9) Å [mean 1.371 (9) Å] and from 118.1 (6) to 121.8 (5)° [mean 120.0 (6)°] respectively.

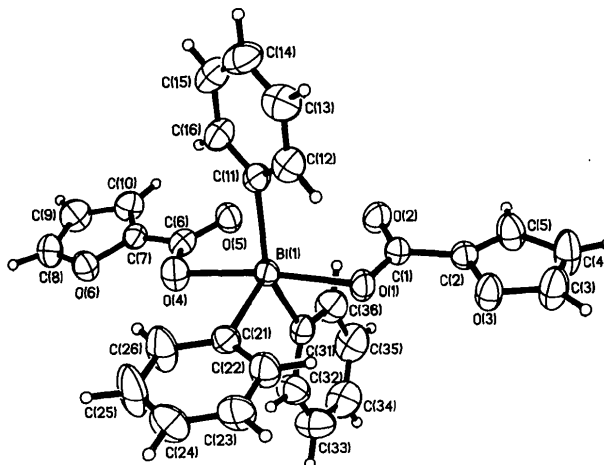


Fig. 1. General view of the molecule.

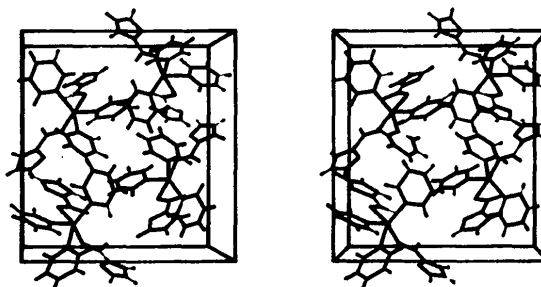


Fig. 2. Stereoscopic view of the unit cell (b vertical, c horizontal).

to form a distorted trigonal bipyramid around the central Bi atom. However, from the two additional short intramolecular Bi—O(2) and Bi—O(5) distances [mean 2.806 (4) Å; sum of the van der Waals radii presumably > 3.60 Å], the polyhedron around Bi appears to be a distorted pentagonal bipyramid, in which the four O(carboxylate) and C(21) atoms are in the equatorial and C(11) and C(31) are in the apical positions. This interpretation gains justification by considering the opening of the angle C(11)—Bi(1)—C(31) to 148.23 (2)° to allow the positioning of O(2) and O(5). A value of 120° would be expected in a regular trigonal bipyramid. The structure is very similar to that found in trimethylbis[2-thenoato(1-)]antimony (Preut, Domagala & Huber, 1987) and triphenylbis[2-thenoato(1-)]antimony (Domagala, Huber & Preut, unpublished).

The distances Bi(1)—O(1) and Bi(1)—O(4) are appreciably greater than the value of 2.075 Å, which was estimated for a Bi—O single bond (March & Ferguson, 1975). In Ph<sub>3</sub>BiClOx (Ox = 8-quinolinolate), a Bi—O bond length of 2.175 Å was determined and the bond was assumed to be covalent (Barton, Charpiot, Dau, Motherwell, Pascard & Pichon, 1984). It therefore may be speculated that the bond between Bi and the carboxylate ligand has an appreciable ionic character. The bond distances and angles of the furan ring are essentially the same as found in solid 2-furancarboxylic acid (Hudson, 1962).

Coordination of O(furan) to Bi can be excluded. Intermolecular distances smaller than the sum of the van der Waals radii do not exist.

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### Structure of *catena*-Poly[tri- $\mu$ -cyano-(ammine)(2-amino-3-methylpyridine)cadmium- $\mu$ -cyano-nickel]

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**Abstract.** [CdNi(CN)<sub>4</sub>(C<sub>5</sub>H<sub>8</sub>N<sub>2</sub>)(NH<sub>3</sub>)],  $M_r = 400.1$ , orthorhombic, *Pna*2<sub>1</sub>,  $a = 13.535$  (1),  $b = 13.607$  (1),  $c = 7.645$  (1) Å,  $V = 1407.99$  Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.875$ ,  $D_x = 1.888$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu(\text{Mo}) = 26.76$  cm<sup>-1</sup>,  $F(000) = 784$ ,  $T = 293$  K,  $R = 0.033$  for 1632 observed reflections. The structure consists of corrugated polymeric networks made up of tetracyanonickelate ions coordinated to Cd. The 2-amino-3-methylpyridine and ammonia molecules bound to Cd in *trans* positions are located on both sides of the network.

**Introduction.** This structure determination is part of a series of studies of Hofmann-type pyridine complexes,  $M(\text{C}_5\text{H}_5\text{N})_2\text{Ni}(\text{CN})_4$ , where  $M$  is a transition metal. Our previous X-ray investigation of a single crystal of Cd(C<sub>5</sub>H<sub>5</sub>N)<sub>2</sub>Ni(CN)<sub>4</sub> (Ülkü, 1975) and studies of powder samples of the analogous compounds with  $M = \text{Mn}, \text{Zn}, \text{Ni}, \text{Co}, \text{Fe}$  and  $\text{Cu}$  (Morehouse, Aytac & Ülkü, 1977) showed its complex nature. In the search for new Hofmann pyridine complexes, compounds with substituted pyridine derivatives were prepared. The first example of a Hofmann-type complex with a pyridine