

the H atoms attached to N6 and O5' were obtained from a difference Fourier map and their positions fixed after constrained refinement. The other H atoms were included at calculated positions. All H atoms were given fixed isotropic temperature factors of approximately 1.5 times that of the parent atom. Anisotropic temperature factors were used for all non-hydrogen atoms, and the refinement converged at  $R = 0.044$ ,  $wR = 0.060$ ,  $w = 0.9675/[\sigma^2(F) + 0.001669F^2]$ . 189 refined parameters; max. shift/e.s.d.  $< 0.05$ ; max. difference peak, 0.24, min. difference peak  $-0.23 \text{ e } \text{Å}^{-3}$ .

Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The program packages *XANADU* (Roberts & Sheldrick, 1975) and *PLUTO* (Motherwell & Clegg, 1978) were also used. All calculations were carried out on the Dundee University DEC-10 computer.

**Discussion.** The atomic numbering is shown in the perspective drawing (Fig. 1), and tables of atomic parameters, bond lengths and angles are given (Tables 1 and 2).\* The *N*-glycosidic torsion angle  $\chi$  has value  $-165.7 (1)^\circ$ , in the *anti* range. The sugar pucker is  ${}_3E$  (C3'-*exo*), with  $P = 197 (1)^\circ$ , and  $\psi_m = 21 (1)^\circ$ . The C4'-C5' conformation, with  $\gamma = 48.3 (3)$  and  $\gamma' = -70.7 (3)^\circ$ , is *+sc* (*gauche-gauche*). The conformational parameters used follow the guidelines of the IUPAC-IUB Joint Commission on Biochemical Nomenclature (1983). There are three hydrogen bonds present in the structure (see Fig. 2); N6-(0.894 Å)-

H61... (2.134 Å)...N1(2-x, y- $\frac{1}{2}$ , -z) 2.974 (4) Å, H angle  $156.2 (2)^\circ$ ; N6-(0.896 Å)-H62... (2.106 Å)...N7(2-x,  $\frac{1}{2}+y$ , -z) 2.994 (4) Å, H angle  $170.9 (2)^\circ$  and O5'-(1.036 Å)-H5'... (1.847 Å)...N3(x, y-1, z) 2.811 (4) Å, H angle  $153.2 (2)^\circ$ . The bases are thus self base-paired in ribbons as in 2',3',5'-tri-*O*-acetyladenosine (Wilson, Tollin & Howie, 1986) and 8-bromo-2',3',5'-tri-*O*-acetyladenosine (Boyd, Low & Tollin, 1987). There is a propeller twist (Dickerson, 1983) of  $28^\circ$  between the planes of the two paired bases. This large propeller twist fits into the pattern observed for self-base-paired adenine bases (Wilson & Tollin, 1987).

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\* 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 44180 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of Hexamethyl Benzenehexacarboxylate

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**Abstract.**  $C_{18}H_{18}O_{12}$ ,  $M_r = 426.33$ , triclinic,  $P\bar{1}$ ,  $a = 11.233 (1)$ ,  $b = 18.940 (1)$ ,  $c = 10.341 (1) \text{ Å}$ ,  $\alpha = 92.29 (1)$ ,  $\beta = 105.59 (1)$ ,  $\gamma = 75.98 (1)^\circ$ ,  $U = 2055.3 (3) \text{ Å}^3$ ,  $Z = 4$ ,  $D_x = 1.38 \text{ Mg m}^{-3}$ ,  $\lambda(\text{Cu K}\alpha) = 1.5418 \text{ Å}$ ,  $\mu(\text{Cu K}\alpha) = 0.981 \text{ mm}^{-1}$ ,  $F(000) = 888$ . Final  $R = 0.067$  for 4662 reflexions. The two symmetry-independent molecules present the same geometry in terms of bond lengths and angles, but

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Table 1. Final fractional coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2 \times 10^3$ )
$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}}$
C(11)	0.7520 (6)	0.4376 (3)	0.9979 (6)	42 (2)
C(111)	0.7567 (6)	0.3588 (3)	0.9762 (7)	49 (3)
O(111)	0.8147 (5)	0.3119 (2)	1.0604 (5)	70 (2)
O(112)	0.6925 (5)	0.3470 (2)	0.8538 (5)	65 (2)
C(112)	0.6938 (10)	0.2709 (4)	0.8193 (9)	93 (4)
C(12)	0.7774 (5)	0.4793 (3)	0.9068 (6)	41 (2)
C(121)	0.8009 (6)	0.4491 (4)	0.7762 (7)	50 (3)
O(121)	0.7335 (5)	0.4748 (3)	0.6694 (5)	69 (2)
O(122)	0.9006 (5)	0.3939 (3)	0.7967 (5)	71 (2)
C(112)	0.9295 (10)	0.3590 (6)	0.6734 (9)	104 (5)
C(13)	0.7754 (5)	0.5532 (3)	0.9298 (6)	42 (2)
C(131)	0.7997 (6)	0.5977 (3)	0.8284 (6)	45 (2)
O(131)	0.7276 (5)	0.6527 (3)	0.7764 (5)	67 (2)
O(132)	0.9090 (4)	0.5676 (3)	0.8010 (5)	59 (2)
C(132)	0.9353 (8)	0.6004 (5)	0.6893 (9)	87 (4)
C(14)	0.7442 (5)	0.5852 (3)	1.0442 (6)	42 (2)
C(141)	0.7426 (7)	0.6646 (3)	1.0667 (6)	49 (3)
O(141)	0.8279 (5)	0.6898 (3)	1.0626 (6)	72 (2)
O(142)	0.6327 (5)	0.7008 (2)	1.0859 (5)	64 (2)
C(142)	0.6245 (9)	0.7792 (4)	1.1124 (10)	85 (4)
C(15)	0.7183 (6)	0.5432 (3)	1.1360 (6)	43 (2)
C(151)	0.6899 (7)	0.5758 (4)	1.2617 (7)	52 (3)
O(151)	0.7538 (6)	0.6105 (3)	1.3366 (5)	84 (3)
O(152)	0.5875 (4)	0.5597 (2)	1.2811 (4)	55 (2)
C(152)	0.5582 (8)	0.5792 (4)	1.4091 (7)	68 (3)
C(16)	0.7219 (5)	0.4690 (3)	1.1135 (6)	41 (2)
C(161)	0.7003 (6)	0.4237 (3)	1.2162 (7)	46 (3)
O(161)	0.7670 (4)	0.4130 (3)	1.3276 (5)	58 (2)
O(162)	0.5983 (4)	0.3969 (3)	1.1638 (5)	62 (2)
C(162)	0.5717 (8)	0.3485 (5)	1.2541 (9)	86 (4)
C(21)	0.2000 (5)	1.0232 (3)	0.6190 (6)	37 (2)
C(211)	0.1564 (6)	1.0528 (3)	0.7391 (6)	44 (2)
O(211)	0.2271 (4)	1.0586 (3)	0.8458 (4)	63 (2)
O(212)	0.0303 (4)	1.0726 (3)	0.7100 (4)	54 (2)
C(212)	-0.0218 (7)	1.1065 (5)	0.8193 (8)	81 (4)
C(22)	0.2733 (6)	0.9519 (3)	0.6244 (6)	39 (2)
C(221)	0.3015 (7)	0.9038 (3)	0.7463 (6)	47 (3)
O(221)	0.4040 (5)	0.8687 (3)	0.8009 (5)	80 (3)
O(222)	0.1967 (4)	0.9078 (3)	0.7841 (4)	61 (2)
C(222)	0.2088 (9)	0.8685 (5)	0.9059 (8)	84 (4)
C(23)	0.3244 (5)	0.9253 (3)	0.5172 (6)	38 (2)
C(231)	0.4014 (6)	0.8491 (3)	0.5212 (6)	46 (3)
O(232)	0.5182 (4)	0.8469 (2)	0.5155 (5)	53 (2)
O(231)	0.3589 (5)	0.7968 (3)	0.5232 (6)	72 (2)
C(232)	0.5998 (7)	0.7748 (4)	0.5059 (9)	72 (3)
C(24)	0.2980 (5)	0.9701 (3)	0.4045 (5)	36 (2)
C(241)	0.3553 (5)	0.9432 (3)	0.2918 (6)	38 (2)
O(241)	0.3515 (4)	0.8854 (2)	0.2398 (4)	50 (2)
O(242)	0.4092 (4)	0.9918 (2)	0.2582 (4)	49 (2)
C(242)	0.4556 (7)	0.9779 (4)	0.1369 (7)	66 (3)
C(25)	0.2187 (5)	1.0401 (3)	0.3960 (5)	34 (2)
C(251)	0.1799 (6)	1.0819 (3)	0.2646 (6)	40 (2)
O(251)	0.1282 (5)	1.0581 (2)	0.1617 (4)	56 (2)
O(252)	0.2101 (5)	1.1455 (2)	0.2780 (4)	58 (2)
C(252)	0.1772 (10)	1.1888 (5)	0.1517 (8)	91 (5)
C(26)	0.1714 (5)	1.0670 (3)	0.5046 (5)	36 (2)
C(261)	0.0962 (6)	1.1441 (3)	0.5048 (6)	41 (2)
O(261)	0.1237 (5)	1.1845 (3)	0.5949 (5)	67 (2)
O(262)	-0.0003 (4)	1.1613 (2)	0.3982 (4)	56 (2)
C(262)	-0.0791 (8)	1.2358 (4)	0.3895 (8)	75 (3)

differences are observed in the relative twists of the six methyl carboxylate groups.

**Introduction.** The title compound was synthesized by catalytic cyclotrimerization of dimethyl acetylenedicarboxylate with a ruthenium(II) complex as catalyst (Romero, Santos & Vegas, 1987). The same reaction is also catalysed by other metal complexes, for example  $[\text{Pd}(\text{PhCN})_2\text{Cl}_2]$  (Maitlis, 1973),  $[\text{Ru}_2(\text{CO})_6\{\text{glyoxal-bis}(\text{tert-butylimine})\}]$  (Staal, Van Koten, Vrieze, Van Santen & Stan, 1981),  $[\{\text{Rh}(\text{CO})_2(\text{PPh}_3)_2\}]$  (Booth, Haszeldini & Perkins, 1981),  $[\text{Pd}_2(\text{dpm})_2\text{X}_2]$  [ $\text{X} = \text{Cl}$ ,

$\text{Br}$ ,  $\text{I}$ ;  $\text{dpm} = \text{bis}(\text{diphenylphosphine})\text{methane}$ ] (Lee, Hunt & Balch, 1981) and  $[\text{Co}(\eta^5\text{-C}_5\text{Me}_5)(\text{C}_2\text{H}_4)_2]$  (Kölle & Fuss, 1986). Although the polymeric nature of the product could be inferred from physical properties and spectral data, its degree of polymerization (2, 3 or 4) was not clear, so that a crystal structure determination was necessary.

**Experimental.** A colourless single crystal of dimensions  $0.5 \times 0.6 \times 0.4$  mm was used to collect the data on a PW1100 four-circle diffractometer with graphite-monochromated  $\text{Cu K}\alpha$  radiation,  $\omega$ - $2\theta$  scan mode, scan amplitude  $1.50^\circ$ , speed  $0.06^\circ \text{ s}^{-1}$ . Unit-cell parameters obtained from least-squares refinement of  $2\theta$  values of 95 reflexions. No systematic absences led to the space group  $P1$  or  $P\bar{1}$ , the latter being confirmed in the course of refinement. 6997 unique reflexions ( $\pm h \pm k \pm l$ ) measured in the range  $2 < \theta < 65^\circ$ , of which 4662 with  $I > 4\sigma(I)$  were considered observed and used in the crystal structure solution.  $-12 \leq h \leq 12$ ,  $-22 \leq k \leq 22$ ,  $0 \leq l \leq 12$ . Crystal decay was not observed, as shown by two standard reflexions (113 and  $\bar{1}\bar{1}\bar{3}$ ) monitored every 90 min (maximal variation of 1.8%). Scattering factors for neutral C and O were taken from *International Tables for X-ray Crystallography* (1974). Structure solved by *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) from  $E$  map calculated with 458 largest  $E$  values ( $E > 1.87$ ) leading to location of 57 of the 60 non-H atoms. The rest were located in a Fourier map. After isotropic refinement, an empirical absorption correction was applied (Walker & Stuart, 1983): the max. and min. absorption corrections were 1.784 and 0.755 respectively. A further anisotropic full-matrix least-squares refinement on  $F$  of the 60 non-hydrogen atoms using unit weights gave  $R = 0.067$ ,  $S = 1.85$ , max. and mean  $\Delta/\sigma < 0.38$  and 0.063 respectively.  $-0.28 \leq \Delta\rho \leq 0.45 \text{ e \AA}^{-3}$ . H atoms, all belonging to  $-\text{CH}_3$  groups, could not be located in the difference map and were not taken into account in the final least-squares calculations which were performed with the *XRAY80* system (Stewart, Kundell & Baldwin, 1980). The final atomic coordinates are collected in Table 1.\*

**Discussion.** Fig. 1 shows a drawing of the two independent molecules forming the asymmetric unit. The structure reveals the threefold polymerization of the starting material. The molecules are packed in columns parallel to  $\mathbf{a}$ . Within the columns, the molecules are related by centres of symmetry at  $0, \frac{1}{2}, 0$  (molecule 1) and  $0, 0, \frac{1}{2}$  (molecule 2) (see Fig. 2).

\* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44138 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The geometry of the benzene rings is normal, the mean C—C bond lengths being 1.397 (9) Å and the C—C—C angles 120.0 (5)°. The remaining molecular dimensions, listed in Table 2, agree within a  $3\sigma$  limit for the two independent molecules. However, as can be observed in Fig. 1 and also from the twist angles about the C(ring)—C(methoxycarbonyl) bonds, listed in Table 3, the conformation of the six methylcarboxylate groups is not the same in both molecules. In molecule 1, four adjacent methyls (1,6,5,4) are up and the other two (2,3) down relative to the ring plane, whereas in molecule 2, three adjacent methyls (6,1,2) are up and three (3,4,5) down. The mean values of the torsion angles for both groups of substituents are 125 and  $-55^\circ$  respectively for molecule 1 and  $-121$  and  $52^\circ$  respectively for molecule 2.

As in other overcrowded benzene derivatives some C atoms directly bonded to the rings deviate from the benzene planes, for example in hexaquaacobalt(II) dihydrogen 1,2,4,5-benzenetetracarboxylate (Ward & Luehrs, 1983) and in trimethyl 4,5,6-trichloro-1,2,3-

benzenetricarboxylate (Galešič, Matijašič & Bruvo, 1984). In our case, the most significant deviations, in terms of  $\Delta/\sigma$ , are for substituents 2 and 6 of molecule 1 and substituents 1,4,5 and 6 of molecule 2. These deviations correlate with the relative orientations of the methyl carboxylate groups within each molecule. Thus,

Table 2. Bond lengths (Å) and angles ( $^\circ$ ) for the two independent molecules of  $C_{18}H_{18}O_{12}$

	x = 1	x = 2		x = 1	x = 2
C(x1)—C(x2)	1.380 (10)	1.396 (8)	C(x51)—O(x52)	1.324 (10)	1.322 (8)
C(x2)—C(x3)	1.406 (9)	1.404 (9)	C(x6)—C(x61)	1.495 (10)	1.498 (8)
C(x3)—C(x4)	1.398 (9)	1.392 (8)	C(x61)—O(x62)	1.338 (8)	1.308 (6)
C(x4)—C(x5)	1.391 (10)	1.398 (7)	C(x11)—O(x11)	1.204 (7)	1.192 (7)
C(x5)—C(x6)	1.409 (9)	1.396 (8)	O(x12)—C(x12)	1.468 (9)	1.465 (10)
C(x6)—C(x1)	1.399 (9)	1.391 (8)	C(x21)—O(x21)	1.203 (7)	1.182 (8)
C(x1)—C(x11)	1.491 (9)	1.496 (9)	O(x22)—C(x22)	1.489 (11)	1.446 (10)
C(x11)—O(x12)	1.317 (8)	1.326 (7)	C(x31)—O(x31)	1.197 (7)	1.202 (9)
C(x2)—C(x21)	1.509 (9)	1.503 (9)	O(x32)—C(x32)	1.462 (12)	1.465 (8)
C(x21)—O(x22)	1.309 (8)	1.321 (10)	C(x41)—O(x41)	1.180 (10)	1.204 (7)
C(x3)—C(x31)	1.490 (10)	1.488 (8)	O(x42)—C(x42)	1.484 (9)	1.474 (10)
C(x31)—O(x32)	1.322 (8)	1.320 (9)	C(x51)—O(x51)	1.198 (9)	1.195 (7)
C(x4)—C(x41)	1.508 (9)	1.494 (9)	O(x52)—C(x52)	1.460 (9)	1.481 (10)
C(x41)—O(x42)	1.322 (9)	1.321 (9)	C(x61)—O(x61)	1.187 (7)	1.205 (8)
C(x5)—C(x51)	1.495 (9)	1.504 (8)	O(x62)—C(x62)	1.467 (12)	1.464 (8)

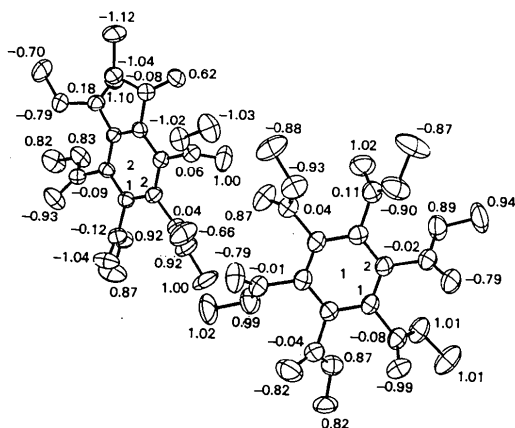


Fig. 1. ORTEP (Johnson, 1965) drawing of the two crystallographically independent molecules projected on the *bc* plane, with deviations (Å) of the atoms from the respective benzene plane. Atoms can be identified from the numbering of the two atoms in the rings.

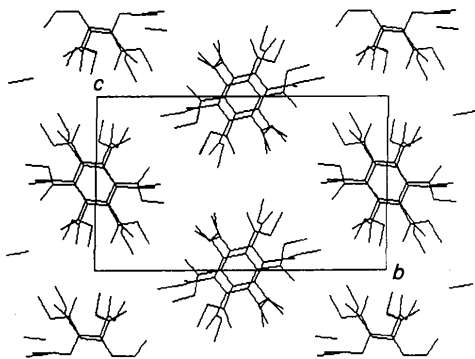


Fig. 2. Unit-cell projection viewed along the *a* axis to show the molecular packing (Vila & Vegas, 1987).

	x = 1	x = 2
C(x2)—C(x1)—C(x6)	120.2 (5)	120.4 (5)
C(x3)—C(x2)—C(x1)	120.6 (5)	120.0 (5)
C(x4)—C(x3)—C(x2)	119.7 (5)	119.2 (5)
C(x5)—C(x4)—C(x3)	119.7 (5)	120.7 (5)
C(x6)—C(x5)—C(x4)	120.5 (6)	119.7 (5)
C(x1)—C(x6)—C(x5)	119.4 (6)	119.8 (5)
C(x11)—C(x1)—C(x6)	118.7 (5)	120.4 (5)
C(x11)—C(x1)—C(x2)	121.1 (5)	119.2 (5)
C(x1)—C(x11)—O(x12)	111.9 (5)	111.1 (5)
C(x1)—C(x11)—O(x11)	123.4 (6)	123.6 (6)
O(x11)—C(x11)—O(x12)	124.6 (6)	125.2 (6)
C(x11)—O(x12)—C(x12)	116.8 (5)	115.3 (5)
C(x1)—C(x2)—C(x21)	121.8 (5)	120.5 (5)
C(x21)—C(x2)—C(x3)	117.5 (5)	119.5 (5)
C(x2)—C(x21)—O(x22)	111.5 (6)	110.4 (5)
C(x2)—C(x21)—O(x21)	121.7 (6)	123.9 (6)
O(x21)—C(x21)—O(x22)	126.8 (6)	125.6 (6)
C(x21)—O(x22)—C(x22)	115.5 (6)	117.5 (6)
C(x2)—C(x3)—C(x31)	119.8 (5)	120.5 (5)
C(x31)—C(x3)—C(x4)	120.5 (5)	120.3 (5)
C(x3)—C(x31)—O(x32)	111.3 (5)	111.5 (5)
C(x3)—C(x31)—O(x31)	124.0 (6)	123.3 (6)
O(x31)—C(x31)—O(x32)	124.6 (6)	125.1 (6)
C(x31)—O(x32)—C(x32)	116.6 (6)	116.7 (5)
C(x3)—C(x4)—C(x41)	118.7 (6)	119.6 (5)
C(x41)—C(x4)—C(x5)	121.6 (6)	119.7 (5)
C(x4)—C(x41)—O(x42)	110.8 (6)	110.1 (5)
C(x4)—C(x41)—O(x41)	123.3 (6)	124.2 (6)
O(x41)—C(x41)—O(x42)	125.9 (6)	125.7 (6)
C(x41)—O(x42)—C(x42)	114.2 (6)	116.9 (5)
C(x4)—C(x5)—C(x51)	120.5 (6)	118.0 (5)
C(x51)—C(x5)—C(x6)	119.0 (6)	122.1 (5)
C(x5)—C(x51)—O(x52)	111.4 (6)	112.4 (5)
C(x5)—C(x51)—O(x51)	124.0 (7)	122.0 (5)
O(x51)—C(x51)—O(x52)	124.6 (6)	125.7 (6)
C(x51)—O(x52)—C(x52)	117.6 (5)	115.2 (5)
C(x5)—C(x6)—C(x61)	119.8 (5)	121.5 (5)
C(x1)—C(x6)—C(x61)	120.7 (5)	118.6 (5)
C(x6)—C(x61)—O(x62)	110.5 (5)	112.6 (5)
C(x6)—C(x61)—O(x61)	123.6 (6)	122.3 (6)
O(x61)—C(x61)—O(x62)	125.9 (6)	125.0 (6)
C(x61)—O(x62)—C(x62)	115.2 (6)	116.5 (5)

Table 3. Torsion angles ( $^\circ$ ) at the C(ring)—C(methoxycarbonyl) bonds

	x = 1	x = 2
C(x2)—C(x1)—C(x11)—O(x11)	129.9 (7)	61.2 (9)
C(x3)—C(x2)—C(x21)—O(x21)	-57.4 (9)	46.0 (10)
C(x4)—C(x3)—C(x31)—O(x31)	-53.2 (9)	-113.5 (8)
C(x5)—C(x4)—C(x41)—O(x41)	127.5 (8)	-130.1 (7)
C(x6)—C(x5)—C(x51)—O(x51)	129.0 (8)	-119.8 (7)
C(x1)—C(x6)—C(x61)—O(x61)	115.1 (8)	49.4 (9)

molecule 1 shows more parallelism between substituents than molecule 2 (see Table 3) and the latter is more puckered. All atoms which deviate significantly from the ring plane are involved in the shortest intramolecular O—O and C—O contacts between atoms of different substituents, the minimum values corresponding to O(112)—O(122) (2.90), C(161)—O(152) (2.73), C(121)—O(112) (2.77), C(121)—O(132) (2.77), O(252)—O(262) (2.90), C(211)—O(222) (2.71) and (C251)—O(242) (2.74 Å).

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## Structure of Ethyl [4-Methyl-2(3*H*)-oxo-1,5-diphenylpyrrol-3-yl]acetate

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**Abstract.** C<sub>21</sub>H<sub>21</sub>NO<sub>3</sub>,  $M_r = 335.4$ , triclinic,  $P\bar{1}$ ,  $a = 14.743$  (5),  $b = 12.941$  (7),  $c = 10.548$  (3) Å,  $\alpha = 76.65$  (3),  $\beta = 90.92$  (4),  $\gamma = 68.46$  (4)°,  $V = 1810$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.222$ ,  $D_x = 1.23$  Mg m<sup>-3</sup>,  $\lambda(\text{Cu } K\alpha) = 1.54178$  Å,  $\mu = 0.62$  mm<sup>-1</sup>,  $F(000) = 712$ , room temperature,  $R = 0.065$  ( $wR = 0.074$ ) for 5314 observed reflections. The *N*-phenyl ring is almost coplanar with the pyrroline ring while the 5-phenyl is nearly perpendicular to it. The most significant difference between the two molecules lies in the different orientation of the ethoxycarbonyl chain.

**Introduction.** This compound has been synthesized in the Departamento de Química Orgánica de la Universidad de Oviedo. This work has been undertaken in order to ascertain whether the central group of the final product of this synthesis was a pyridine or a pyrroline.

**Experimental.** Colourless prismatic crystals (0.3 × 0.3 × 0.2 mm); Philips PW1100 diffractometer, Cu *K*α radiation with graphite monochromator,  $\omega/2\theta$  scan technique, room temperature.  $D_m$  by flotation. Refined unit-cell parameters obtained from setting angles of 32 reflections ( $2 < 2\theta < 25^\circ$ ). 6170 unique reflections

measured  $[(\sin\theta)/\lambda]_{\max} = 0.58$  Å<sup>-1</sup>, 5314 [ $I > 2\sigma(I)$ ] considered observed, index range:  $h - 17/17$ ,  $k - 14/14$ ,  $l 0/12$ ; two check reflections measured every 90 reflections, net count constant; Lorentz and polarization corrections, no absorption correction. Structure solved by direct methods, *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Positional and anisotropic thermal parameters for all non-hydrogen atoms refined (on  $F$ ) by block-diagonal least squares; positions of H atoms from a difference synthesis excepting H8, H9 and H10 that were calculated geometrically; H atoms assigned the same isotropic temperature factors as the atoms to which they are bonded and included in the refinement with positional and thermal parameters fixed (451 parameters); an empirical weighting scheme was used to give similar values of  $w\Delta F^2$  over ranges of  $(\sin\theta)/\lambda$  and  $F_o$ . Final  $R = 0.065$ ;  $wR = 0.074$ ,  $S = 6.3$ . ( $\Delta/\sigma$ )<sub>max</sub> = 0.38; residual electron density in difference map 0.57 e Å<sup>-3</sup>; atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Computations carried out on UNIVAC 1100 computer using the *XRAY* system (Stewart, Kundell & Baldwin, 1970), *PESOS* (Martinez-Ripoll & Cano, 1975) and *PARST* (Nardelli, 1983).