conduit à la formation de chaînes moléculaires infinies en zig-zag, la cohésion entre celles-ci étant assurée par de nombreux contacts de van der Waals.

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The Structure of 2,2'-Diphenyl-1,1'-(1,3-phenylene)diethanedione, $C_{22}H_{14}O_4$

By Ashfaquzzaman Syed* and Edwin D. Stevens

Department of Chemistry, University of New Orleans, New Orleans, LA 70148, *USA*

and Cheryl L. Klein

Department of Chemistry, Xavier University of Louisiana, New Orleans, Louisiana 70125, *USA*

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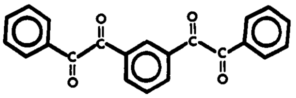
Abstract. $M_r = 342\cdot4$, orthorhombic, *Pbca*, $a = 7\cdot756\,(2)$, $b = 14\cdot864\,(1)$, $c = 29\cdot523\,(3)$ Å, $V = 3403\cdot6\,(14)$ Å^3, $Z = 8$, $D_m = 1\cdot34\,(1)$, $D_x = 1\cdot336$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 0\cdot86$ cm^{-1}, $F(000) = 1424$, $T = 300$ K, final $R = 0\cdot035$, $R_w = 0\cdot039$ for 1217 unique reflections. Adjacent carbonyl groups are arranged in an antiparallel configuration. Three of the carbonyl groups are approximately planar with the central phenyl ring. The other two phenyl rings are twisted by 40·4 (3) and 88·1 (3)° with respect to the central phenyl ring. The molecules are held together by van der Waals forces.

Introduction. The crystal structure analysis of the title compound was undertaken as a part of a program to determine the structures of benzophenones and related compounds to study the changes in ground- and excited-state geometry of the carbonyl group.



Experimental. Bright-yellow rectangular crystals obtained by slow evaporation of solution of compound in carbon tetrachloride–acetone mixture, D_m measured by flotation (xylene + bromobenzene). Data collected on CAD-4 diffractometer from crystal $0\cdot18 \times 0\cdot18 \times 0\cdot40$ mm; systematic absences $h0l$ for $l = 2n + 1$, $hk0$ for $h = 2n + 1$ and $0kl$ for $k = 2n + 1$ confirmed space group *Pbca*; cell constants from least-squares analysis of 25 reflections with $18 \le 2\theta \le 3\theta°$ measured on diffractometer; ω:2θ scan mode to measure 2982 reflections with $\theta \le 25°$; $0 \le h \le 9$, $0 \le k < 17$, $0 < l < 35$; 1217 unique reflections based on $I > 3\sigma(I)$ used in the refinement; Lorentz–polarization correction, no absorption correction; no significant variations in intensities of 3 standards; structure determined using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978; Frenz, 1982); E map revealed positions of all nonhydrogen atoms of the molecule; subsequent difference Fourier map gave all but three hydrogens which were located from the knowledge of the geometry of the benzene ring; isotropic full-matrix least-squares refinement with unit weights gave $R = 0\cdot090$; full-matrix least squares with anisotropic thermal parameters for all C's and O's and isotropic for all H's brought R to 0·039; extinction correction applied in final cycles refined to a coefficient $= 1\cdot7 \times 10^{-7}$ mm; refinement converged to $R = 0\cdot035$, $R_w = 0\cdot039$, minimizing $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F)$; $S = 1\cdot33$, max. $\Delta/\sigma = 0\cdot01$. Scattering factors from *International Tables for X-ray Crystallography* (1974). All calculations performed on PDP 11/34 computer using the *SDP* program system (Frenz, 1982).†

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* On leave from Tata Iron & Steel Co., Jamshedpur, India.

† Lists of structure factors, additional bond lengths and angles, and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39343 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters*

Anisotropically refined atoms are given in the form of the equivalent isotropic thermal parameter defind as $\frac{4}{3}[a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ab\cos\gamma\beta_{12} + ac\cos\beta\beta_{13} + bc\cos\alpha\beta_{23}]$.

	x	y	z	B or B_{eq}(Å^2)
O(1)	0·0469 (3)	−0·0746 (1)	0·25719 (7)	5·84 (6)
O(2)	0·2348 (1)	0·0855 (1)	0·31841 (6)	7·34 (7)
O(3)	0·2577 (4)	−0·0413 (2)	0·11269 (6)	7·01 (6)
O(4)	0·1822 (3)	0·1292 (2)	0·05650 (8)	7·73 (7)
C(1)	0·2590 (1)	0·0890 (2)	0·23901 (9)	4·11 (6)
C(2)	0·2545 (4)	0·0446 (2)	0·19791 (9)	4·13 (6)
C(3)	0·2921 (4)	0·0889 (2)	0·15779 (9)	4·08 (6)
C(4)	0·3385 (5)	0·1788 (2)	0·15911 (10)	5·36 (8)
C(5)	0·3468 (5)	0·2238 (2)	0·19984 (11)	6·08 (9)
C(6)	0·3062 (5)	0·1797 (2)	0·23919 (10)	5·39 (8)
C(7)	0·2215 (4)	0·0450 (2)	0·28300 (9)	4·59 (7)
C(8)	0·1513 (4)	−0·0506 (2)	0·28557 (9)	4·37 (7)
C(9)	0·1943 (4)	−0·1063 (2)	0·32539 (9)	4·17 (7)
C(10)	0·0814 (5)	−0·1732 (2)	0·33843 (10)	5·48 (8)
C(11)	0·1172 (5)	−0·2259 (2)	0·37586 (11)	6·69 (8)
C(12)	0·2678 (5)	−0·2127 (2)	0·39957 (10)	6·42 (9)
C(13)	0·3815 (5)	−0·1473 (2)	0·38671 (10)	5·78 (9)
C(14)	0·3443 (4)	−0·0942 (2)	0·34954 (9)	4·59 (7)
C(15)	0·2879 (4)	0·0387 (2)	0·11468 (9)	4·79 (8)
C(16)	0·3100 (4)	0·0907 (2)	0·07013 (10)	5·02 (7)
C(17)	0·4770 (4)	0·0919 (2)	0·04735 (9)	3·92 (7)
C(18)	0·4965 (5)	0·1390 (2)	0·00707 (9)	5·13 (8)
C(19)	0·6546 (5)	0·1446 (2)	−0·01369 (10)	5·87 (9)
C(20)	0·7924 (4)	0·1035 (2)	0·00535 (11)	5·97 (9)
C(21)	0·7781 (4)	0·0559 (2)	0·04524 (11)	5·64 (8)
C(22)	0·6191 (4)	0·0506 (2)	0·06580 (9)	4·71 (7)
H(2)	0·222 (3)	−0·018 (1)	0·1967 (6)	2·9 (5)*
H(4)	0·371 (3)	0·208 (2)	0·1299 (7)	4·3 (6)*
H(5)	0·379 (4)	0·284 (2)	0·1995 (8)	6·9 (7)*
H(6)	0·315 (3)	0·207 (2)	0·2682 (7)	5·4 (7)*
H(10)	−0·028 (4)	−0·182 (2)	0·3238 (9)	7·4 (8)*
H(11)	0·044 (4)	−0·272 (2)	0·3828 (9)	7·5 (8)*
H(12)	0·289 (4)	−0·249 (2)	0·4263 (8)	7·1 (8)*
H(13)	0·491 (4)	−0·137 (2)	0·4024 (9)	7·1 (8)*
H(14)	0·430 (3)	−0·048 (2)	0·3418 (7)	4·7 (6)*
H(18)	0·402 (3)	0·168 (2)	−0·0034 (8)	5·4 (7)*
H(19)	0·663 (4)	0·183 (2)	−0·0412 (9)	7·9 (8)*
H(20)	0·906 (3)	0·111 (2)	−0·0075 (8)	5·6 (7)*
H(21)	0·887 (5)	0·031 (2)	0·0588 (10)	9·4 (9)*
H(22)	0·614 (3)	0·019 (2)	0·0916 (8)	5·1 (6)*

* Refined isotropically.

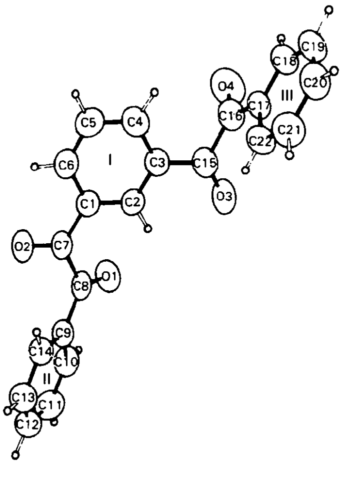

Fig. 1. *ORTEP* plot of the 2,2'-diphenyl-1,1'-(1,3-phenylene)-diethanedione molecule showing the atomic labeling scheme.

Table 2. *Bond distances* (Å) *and bond angles* (°)

Numbers in parentheses are e.s.d.'s in the least significant digits.

O(1)C(8)	1·219 (3)	C(4)C(5)	1·377 (4)
O(2)C(7)	1·210 (3)	C(5)C(6)	1·370 (4)
O(3)C(15)	1·213 (3)	C(7)C(8)	1·524 (4)
O(4)C(16)	1·213 (3)	C(8)C(9)	1·476 (4)
C(1)C(2)	1·382 (4)	C(3)C(15)	1·475 (4)
C(1)C(6)	1·397 (4)	C(15)C(16)	1·535 (4)
C(1)C(7)	1·483 (4)	C(16)C(17)	1·460 (4)
C(2)C(3)	1·386 (4)		
C(3)C(4)	1·385 (4)		
O(3)C(15)C(3)	122·8 (3)	C(2)C(1)C(6)	118·1 (3)
O(3)C(15)C(16)	118·2 (3)	C(2)C(1)C(7)	123·6 (3)
C(3)C(15)C(16)	118·9 (3)	C(6)C(1)C(7)	118·3 (3)
O(4)C(16)C(15)	115·5 (3)	C(1)C(2)C(3)	121·2 (3)
O(4)C(16)C(17)	124·5 (3)	C(2)C(3)C(4)	119·3 (3)
C(15)C(16)C(17)	120·0 (3)	C(2)C(3)C(15)	119·5 (3)
C(16)C(17)C(18)	119·9 (3)	C(4)C(3)C(15)	121·2 (3)
C(16)C(17)C(22)	121·6 (3)	C(3)C(4)C(5)	120·3 (3)
O(1)C(8)C(9)	122·2 (3)	C(4)C(5)C(6)	119·8 (3)
C(7)C(8)C(9)	118·8 (3)	C(1)C(6)C(5)	121·2 (3)
C(8)C(9)C(10)	118·9 (3)	O(2)C(7)C(1)	121·4 (3)
C(8)C(9)C(14)	122·0 (3)	O(2)C(7)C(8)	116·8 (3)
		C(1)C(7)C(8)	121·7 (3)
		O(1)C(8)C(7)	118·4 (3)

Table 3. *Selected torsion angles* (°)

The e.s.d.'s for the torsion angles are in the range 0·4–0·5°.

O(1)C(8)C(7)O(2)	137·7	O(3)C(15)C(16)O(4)	96·1
O(1)C(8)C(9)C(14)	159·9	O(4)C(16)C(17)C(22)	176·6
O(1)C(8)C(9)C(10)	−19·4	O(4)C(16)C(17)C(18)	−0·5
C(1)C(7)C(8)C(9)	150·3	C(3)C(15)C(16)C(17)	99·8
C(7)C(8)C(9)C(10)	152·4	C(15)C(16)C(17)C(18)	−180·0
C(7)C(8)C(9)C(14)	−28·4	C(15)C(16)C(17)C(22)	−2·8
C(8)C(7)C(1)C(6)	173·5	C(16)C(15)C(3)C(2)	173·3
C(8)C(7)C(1)C(2)	−8·3	C(16)C(15)C(3)C(4)	−8·8
O(1)C(8)C(7)C(1)	−37·6	O(3)C(15)C(16)C(17)	−84·4
O(2)C(7)C(8)C(9)	−34·3	O(4)C(16)C(15)C(3)	−79·7
O(2)C(7)C(1)C(2)	176·5	O(3)C(15)C(3)C(4)	175·6
O(2)C(7)C(1)C(6)	−1·6	O(3)C(15)C(3)C(2)	−2·3

Discussion. Final atomic positional and equivalent isotropic thermal parameters (e.s.d.'s shown in parentheses) are given in Table 1. Bond distances and angles are given in Table 2 and selected torsion angles in Table 3. An *ORTEP* (Johnson. 1976) plot of the molecule and numbering scheme is shown in Fig. 1.

The basic configuration of the structure comprises twisted molecules, and the main features are comparable to those reported for similar systems by van der Heijdin, Griffith, Chandler & Robertson (1975). The twisted molecule geometry is best described by the torsion angles given in Table 3. Carbonyl groups adopt an antiparallel configuration yielding a sterically least-hindered structure. Bond lengths and angles of the phenyl rings (called I, II and III in Fig. 1), which are planar within 0·003 (3) Å, are in the range of values found for similar systems (Breton, Précigoux, Courseille & Hospital, 1975; Ottersen, Vance, Doorenbos, Chang & El-Feraly, 1977; Liebich, 1976). Atoms O(2) and O(3) are 0·073 (3) and −0·106 (3) Å away from plane I whereas O(1) shows the maximum deviation, 0·344 (2) Å, with respect to II [torsion angle O(1)-C(8)C(9)C(14) = 159·9°], and O(4) is almost planar, −0·008 (2) Å, with respect to plane III.

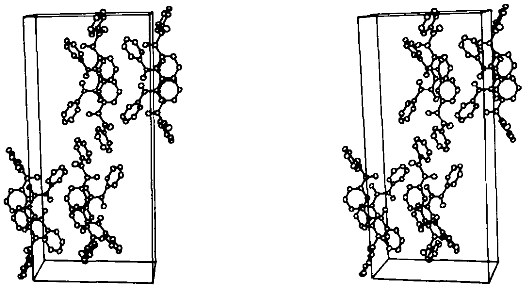

Fig. 2. Stereoview of the molecular packing in the unit cell.

Three of the four carbonyl groups show small but significant deviations from planarity. The largest deviations are observed in the C(7)C(8)O(1)C(9) group, where C(8) is 0·043 (2) Å from the least-squares plane through the four atoms. In contrast, the C(15)-C(16)O(4)C(17) is highly planar, with C(16) only 0·003 (3) Å from the least-squares plane. The C(16)—C(17) distance [1·460 (4) Å] is shorter than the C(8)—C(9) distance [1·476 (4) Å], possibly indicating greater conjugation of the O(4)—C(16) bond with the phenyl ring, in agreement with the much smaller torsion angle O(4)C(16)C(17)C(18) [−0·5°] compared with O(1)C(8)C(9)C(10) [−19·4°].

The average C—O distance, 1·24 Å, agrees well with various carbonyl bond lengths observed in other similar compounds (Brown & Colclough, 1983; Hirshfeld & Rabinovich, 1967; Fonseca, Martínez-Carrera & García-Blanco, 1982). The C(7)—C(8) and C(15)—C(16) distances, 1·524 (4) and 1·535 (4) Å, are very close to a C(sp^3)—C(sp^3) bond (1·54 Å) (Greenhough & Trotter, 1980; Acton, Roth, Katz, Frank, Maier & Paul, 1972), while C(1)—C(7) [1·483 (4) Å], C(8)—C(9) [1·476 (4) Å], C(3)—C(15) [1·475 (4) Å] and C(16)—C(17) [1·460 (4) Å] are similar and correspond to the C(sp^2)—C(sp^2) distances (1·48 Å) (Somayajulu, 1959; Kuchitsu, Fukuyama & Morino, 1968). The stereoscopic view of the molecular packing is shown in Fig. 2. The molecules are held together by van der Wäals forces. No unusually short intermolecular distances were noticed.

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The Structure of 2,2′-Diphenyl-1,1′-(1,4-phenylene)diethanedione, $C_{22}H_{14}O_4$

By Ashfaquzzaman Syed* and Edwin D. Stevens

Department of Chemistry, University of New Orleans, New Orleans, LA 70148, *USA*

(*Received* 5 *July* 1983; *accepted* 14 *March* 1984)

Abstract. $M_r = 342\cdot4$, triclinic, $P\bar{1}$, $a = 4\cdot466\,(2)$, $b = 12\cdot785\,(2)$, $c = 16\cdot104\,(2)$ Å, $\alpha = 68\cdot46\,(1)$, $\beta = 88\cdot16\,(2)$, $\gamma = 88\cdot08\,(2)°$, $V = 854\cdot67\,(14)$ Å^3, $Z = 2$, $D_x = 1\cdot330$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0\cdot71073$ Å, $\mu = 0\cdot9$ cm^{-1}, $F(000) = 356$, $T = 300$ K, final $R = 0\cdot035$, $R_w = 0\cdot047$ for 1555 independent reflections. The molecular structure is closely related to that of 2,2′-diphenyl-1,1′-(1,3-phenylene)diethanedione with twisted

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* On leave from Tata Iron & Steel Co., Jamshedpur, India.

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