Acta Cryst. (1995). C**51**, 1712–1714

(±)-3,4-Bis(4-methylphenyl)-1,6-diphenyl-1,6-hexanedione

CHUAN-FENG CHEN† AND JIAN-HUA XU

Department of Chemistry, Nanjing University, Nanjing 210008, People's Republic of China

YING ZHU AND YOU-CHENG LIU

Department of Chemistry, Lanzhou University, Lanzhou 730000, People's Republic of China

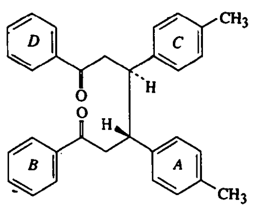
(*Received* 31 *January* 1994; *accepted* 17 *January* 1995)

Abstract

The title compound, $C_{32}H_{30}O_2$, was produced by photoinduced reduction of a chalcone in the presence of triethylamine. The structural studies indicate that in the molecule, the C12—C11—C16 angle [120.4 (3)°] about the *ipso* C atom of a terminal phenyl substituent, increases as the carbonyl angle O1—C10—C11 increases [119.1 (2)°], while C28—C27—C32 [119.1 (3)°] of the other terminal phenyl group decreases as its associated carbonyl angle O2—C26—C27 increases [120.3 (3)°]. Studies of the dihedral angles and the torsion angles of the title compound reveal that the molecule is a racemic compound and has an inverted umbrella-like structure.

Comment

The reduction reactions of chalcones have been studied extensively. *meso*-1,3,4,6-Tetraphenyl-1,6-hexanedione has been obtained by the thermal and photochemical reduction reactions of chalcone, but its racemic diketone compound has not been reported previously (Chiacchio, Corsaro, Compagnini & Purrello, 1983; Mirek, Gaweda & Kawalek, 1981; Honzl, Doskocilova, Pokorny & Uchytil, 1978). Recently, when studying the photoinduced reaction of chalcone in the presence of triethylamine in anhydrous acetonitrile, we obtained two types of 1,6-hexanedione derivative at the same time: one was *meso* and the other the racemic form, as indicated by the spectroscopic and physical data (Xu, Chen & Liu, 1994). In order to obtain further proof of the racemic product, the X-ray crystal structure of the title compound, (I), was undertaken.



(I)

The title compound possesses two chiral centres at C1 and C17. In the molecule, the benzene rings are planar within estimated limits of error. The six C—C bonds in the benzene rings vary in length from 1.369 (6) to 1.401 (5) Å. The mean value [1.389 (5) Å] is longer than the value found in 1,4-dibenzoylbutane (2DBC) (1.377 Å; Deguire & Brisse, 1988). It is observed that the C12—C11—C16 angle [120.4 (3)°] deviates slightly from the mean value. Deguire & Brisse (1988) stated that in order to minimize the interactions between the carbonyl group and the H atom on the C16 atom, the angle C12—C11—C16 should decrease with an increase in the O1—C10—C11 angle. In 2DBC, these angles are 116.9 and 119.5°, respectively. In the present structure, the angle O1—C10—C11 and the other carbonyl angle O2—C26—C27 are 119.1 (2) and 120.3 (3)°, respectively, similar to that in 2DBC. However, the angles C12—C11—C16 and C28—C27—C32 are 120.4 (3) and 119.1 (3)°, respectively, and are significantly greater than in 2DBC.

The dihedral angles between rings *A* and *B*, *A* and *C*, *A* and *D*, *B* and *C*, *B* and *D*, and *C* and *D* are 103.3, 26.7, 40.0, 76.8, 77.5 and 33.5°, respectively, which give rise to an inverted umbrella-like structure, with ring *B* as the umbrella handle. The torsion angles C2—C1—C17—C25, C9—C1—C17—C25, C2—C1—C17—C18 and C9—C1—C17—C18 are -56.0 (3), 175.6 (3), 71.1 (3) and -57.4 (3)°, respectively, which indicate that the title compound forms a racemic crystal structure.

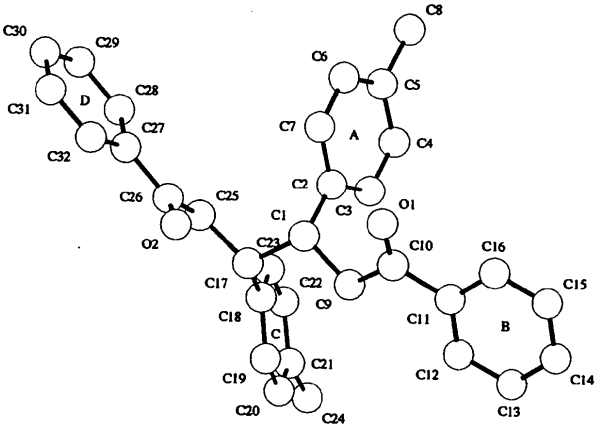


Fig. 1. The molecule geometry of the title compound.

---

† Permanent address for correspondence: Institute of Chemistry, Academia Sinica, Beijing 100080, People's Republic of China.

Acta Crystallographica Section C
ISSN 0108-2701 ©1995

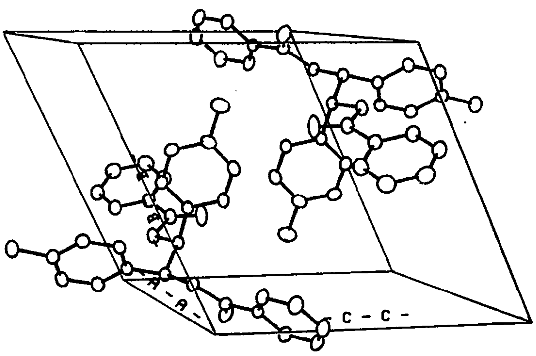


Fig. 2. The unit cell of the title compound.

Experimental

The title compound was obtained by photoinduced reaction of chalcone and triethylamine in acetonitrile, and crystallized from a solution of ethyl acetate and ethanol.

Crystal data

$C_{32}H_{30}O_2$
$M_r = 446.6$
Triclinic
$P\bar{1}$
$a = 11.011$ (2) Å
$b = 11.395$ (2) Å
$c = 11.491$ (2) Å
$\alpha = 114.5$ (2)°
$\beta = 101.8$ (1)°
$\gamma = 93.3$ (2)°
$V = 1267.7$ Å^3
$Z = 2$
$D_x = 1.17$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.5418$ Å
Cell parameters from 25 reflections
$\theta = 18$–32°
$\mu = 0.52$ mm^{-1}
$T = 296$ K
Prismatic
0.25 × 0.20 × 0.20 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans [width (0.50 + 0.140tanθ)° in ω, speed 1–10° min^{-1}]
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{\min} = 0.92$, $T_{\max} = 1.00$
3826 measured reflections
3215 independent reflections

3238 observed reflections [$I \geq 3\sigma(I)$]
$R_{\rm int} = 0.019$
$\theta_{\max} = 58°$
$h = 0 \rightarrow 12$
$k = -12 \rightarrow 12$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 90 min
intensity decay: 1.1%

Refinement

Refinement on F
$R = 0.069$
$wR = 0.068$
$S = 0.854$
3238 reflections
307 parameters
Unit weights applied

$(\Delta/\sigma)_{\max} = 0.12$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.12$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$B_{\rm eq} = (4/3)\Sigma_i\Sigma_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{\rm eq}$
O1	0.6968 (2)	0.3109 (3)	0.3086 (2)	6.29 (7)
O2	0.3325 (2)	−0.0023 (2)	0.1454 (2)	5.98 (6)
C1	0.4620 (3)	0.2489 (3)	0.1311 (2)	3.30 (7)
C2	0.4302 (3)	0.3833 (3)	0.2052 (2)	3.16 (6)
C3	0.4336 (3)	0.4807 (3)	0.1614 (3)	4.01 (7)
C4	0.4033 (3)	0.6023 (3)	0.2330 (3)	4.57 (8)
C5	0.3709 (3)	0.6322 (3)	0.3527 (3)	4.52 (9)
C6	0.3680 (3)	0.5355 (3)	0.3965 (3)	4.12 (8)
C7	0.3969 (3)	0.4143 (3)	0.3244 (3)	3.59 (7)
C8	0.3410 (5)	0.7655 (4)	0.4331 (4)	7.2 (1)
C9	0.5842 (3)	0.2558 (3)	0.0871 (3)	3.93 (7)
C10	0.6990 (3)	0.3089 (3)	0.2024 (3)	4.04 (7)
C11	0.8206 (3)	0.3565 (3)	0.1839 (3)	3.87 (7)
C12	0.8394 (3)	0.3270 (3)	0.0599 (3)	4.45 (8)
C13	0.9556 (3)	0.3721 (3)	0.0496 (3)	5.29 (9)
C14	1.0496 (3)	0.4468 (4)	0.1613 (4)	5.9 (1)
C15	1.0309 (4)	0.4765 (4)	0.2847 (4)	6.0 (1)
C16	0.9163 (3)	0.4309 (3)	0.2971 (3)	5.01 (9)
C17	0.3539 (3)	0.1514 (3)	0.0137 (3)	3.45 (7)
C18	0.3260 (3)	0.1828 (3)	−0.1045 (3)	3.38 (7)
C19	0.3765 (3)	0.1163 (3)	−0.2115 (3)	4.13 (9)
C20	0.3545 (3)	0.1452 (3)	−0.3201 (3)	4.73 (8)
C21	0.2843 (3)	0.2410 (3)	−0.3247 (3)	4.55 (8)
C22	0.2318 (3)	0.3049 (3)	−0.2181 (3)	4.38 (8)
C23	0.2517 (3)	0.2756 (3)	−0.1108 (3)	4.07 (8)
C24	0.2661 (4)	0.2779 (4)	−0.4386 (3)	6.5 (1)
C25	0.2345 (5)	0.1365 (3)	0.0600 (3)	3.63 (7)
C26	0.2452 (3)	0.0554 (3)	0.1376 (3)	3.94 (7)
C27	0.1420 (3)	0.0462 (3)	0.2023 (3)	3.60 (7)
C28	0.0470 (3)	0.1206 (3)	0.2114 (3)	4.39 (8)
C29	−0.0440 (3)	0.1082 (4)	0.2758 (3)	5.32 (9)
C30	−0.0407 (3)	0.0195 (3)	0.3295 (3)	5.38 (9)
C31	0.0529 (4)	−0.0555 (3)	0.3201 (3)	5.72 (9)
C32	0.1450 (4)	−0.0432 (3)	0.2571 (3)	5.03 (9)

Table 2. *Selected geometric parameters* (Å, °)

O1—C10	1.216 (4)	C15—C16	1.394 (6)
O2—C26	1.206 (4)	C17—C18	1.521 (5)
C1—C2	1.515 (4)	C17—C25	1.541 (5)
C1—C9	1.540 (5)	C18—C19	1.397 (4)
C1—C17	1.555 (3)	C18—C23	1.391 (5)
C2—C3	1.397 (6)	C19—C20	1.396 (5)
C2—C7	1.398 (4)	C20—C21	1.386 (5)
C3—C4	1.389 (4)	C21—C22	1.401 (5)
C4—C5	1.401 (5)	C21—C24	1.512 (6)
C5—C6	1.389 (6)	C22—C23	1.385 (5)
C5—C8	1.514 (6)	C25—C26	1.521 (5)
C6—C7	1.380 (4)	C26—C27	1.501 (5)
C9—C10	1.510 (4)	C27—C28	1.377 (5)
C10—C11	1.503 (5)	C27—C32	1.401 (5)
C11—C12	1.385 (5)	C28—C29	1.395 (5)
C11—C16	1.394 (4)	C29—C30	1.384 (6)
C12—C13	1.400 (5)	C30—C31	1.369 (6)
C13—C14	1.377 (4)	C31—C32	1.393 (7)
C14—C15	1.378 (6)		
C2—C1—C9	112.1 (2)	C1—C17—C18	114.1 (3)
C2—C1—C17	114.9 (2)	C1—C17—C25	111.0 (2)
C9—C1—C17	111.0 (2)	C18—C17—C25	111.6 (2)
C1—C2—C3	123.3 (3)	C17—C18—C19	119.2 (3)
C1—C2—C7	119.5 (3)	C17—C18—C23	122.6 (3)
C3—C2—C7	117.2 (3)	C19—C18—C23	118.2 (4)
C2—C3—C4	121.0 (4)	C18—C19—C20	120.4 (3)
C3—C4—C5	121.1 (4)	C19—C20—C21	121.6 (4)
C4—C5—C6	117.9 (3)	C20—C21—C22	117.4 (3)
C4—C5—C8	121.4 (4)	C20—C21—C24	121.9 (3)
C6—C5—C8	120.7 (4)	C22—C21—C24	120.7 (3)
C5—C6—C7	120.9 (3)	C21—C22—C23	121.3 (3)
C2—C7—C6	122.0 (3)	C18—C23—C22	121.1 (3)

C1—C9—C10	112.1 (2)	C17—C25—C26	112.6 (3)
O1—C10—C9	121.9 (4)	O2—C26—C25	121.5 (3)
O1—C10—C11	119.1 (2)	O2—C26—C27	120.3 (3)
C9—C10—C11	119.0 (3)	C25—C26—C27	118.2 (3)
C10—C11—C12	122.1 (2)	C26—C27—C28	124.0 (3)
C10—C11—C16	117.5 (3)	C26—C27—C32	116.9 (4)
C12—C11—C16	120.4 (3)	C28—C27—C32	119.1 (3)
C11—C12—C13	119.3 (2)	C27—C28—C29	120.4 (4)
C12—C13—C14	120.2 (3)	C28—C29—C30	120.2 (3)
C13—C14—C15	120.5 (4)	C29—C30—C31	119.8 (4)
C14—C15—C16	120.1 (3)	C30—C31—C32	120.6 (4)
C11—C16—C15	119.5 (3)	C27—C32—C31	119.9 (3)

The structure was solved by direct methods and difference Fourier syntheses. Refinement was by full-matrix least-squares methods with anisotropic displacement parameters for all non-H atoms and isotropic displacement parameters for H atoms.

All calculations were performed on a PDP 11/44 computer with the *SDP* (Frenz, 1978) programs.

This work is supported by National Nature Science Foundation of China and State Key Laboratory of Applied Organic Chemistry in Lanzhou University.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CR1131). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Chiacchio, U., Corsaro, A., Compagnini, A. & Purrello, G. (1983). *J. Chem. Soc. Perkin Trans.* 1, pp. 671–674.
Deguire, S. & Brisse, F. (1988). *Can. J. Chem.* **66**, 341–347.
Frenz, B. A. (1978). *The Enraf–Nonius CAD-4 SDP – a Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Solution. Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft Univ. Press.
Honzl, J., Doskocilova, D., Pokorny, S. & Uchytil, B. (1978). *Collect. Czech. Chem. Commun.* **43**, 1895–1897.
Mirek, J., Gaweda, M. & Kawalek, B. (1981). *Pol. J. Chem.* **55**, 987–993.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.
Xu, J.-H., Chen, C.-F. & Liu, Y.-C. (1994). In preparation.

Acta Cryst. (1995). C**51**, 1714–1716

2,6-Dichlorophenylguanidinium Chloride

JAMES BARKER

The Associated Octel Company Ltd, PO Box 17,
R & D Laboratories, Oil Sites Road, Ellesmere Port,
South Wirral L65 4HF, England

HAROLD R. POWELL

Cambridge Crystallographic Data Centre,
12 Union Road, Cambridge CB2 1EZ, England

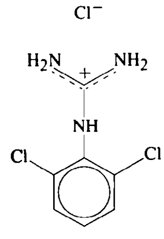
(*Received* 11 *November* 1994; *accepted* 13 *February* 1995)

Abstract

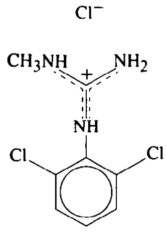
The C—N bonds in $C_7H_8Cl_2N_3^+.Cl^-$ were found to differ in length [1.304 (4), 1.308 (4) and 1.493 (4) Å]. There is not, therefore, the symmetrical delocalization around the central N_3C expected for a guanidinium system, and the molecule is closer in nature to a substituted amidinium salt.

Comment

Structural studies of guanidines and their salts continue to attract attention (Carpy, Leger, Wermuth & Leclerc, 1981; Hafelinger & Kuske, 1991) because of their biological and pharmaceutical, *e.g.* neuroleptic and antihypersensitive, activity (Rouot, Leclerc, Wermuth, Miesch & Schwarz, 1978; Bream, Lauener, Picard, Scholtysik & White, 1975). In the title compound, (I), the asymmetric unit contains one molecule, resolved as the *E* isomer (Fig. 1).



(I)



(II)

Two of the three C—N bond lengths of the N_3C fragment are similar [N(14)—C(12) 1.304 (4) and N(13)—C(12) 1.308 (4) Å] and are only slightly longer than the mean of 277 $C_{sp^2}{=}N$ double bonds [1.295 Å] located in the Cambridge Structural Database (Allen *et al.*, 1987), indicating delocalization around the N—C—N fragment. The third C—N bond length [C(12)—N(11) 1.493 (4) Å] and the C(aryl)—N distance [C(1)—N(11) 1.519 (4) Å] are clearly single bond in character, and thus the delocalization is not across the whole of the