

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

1,4-Bis(4-chlorophenyl)butane-1,4-dione

Shuqin Li* and Huisheng Li

Department of Chemistry and Biology, Xiangfan University, Xiangfan 441053, People's Republic of China

Correspondence e-mail: shuqin2000@yahoo.com.cn

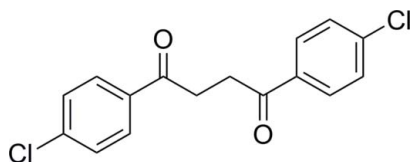
Received 12 November 2008; accepted 21 November 2008

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.042; wR factor = 0.117; data-to-parameter ratio = 15.4.

The molecule of title compound, $\text{C}_{16}\text{H}_{12}\text{Cl}_2\text{O}_2$, is centrosymmetric. Thus, the asymmetric unit comprises two half-molecules. The two benzene rings are coplanar in each independent molecule (dihedral angles = 0°). The crystal packing exhibits intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For applications of the title compound, see: Rao *et al.* (2004); Stauffer & Neier (2000); Shridhar *et al.* (1982). For the preparation of the title compound, see: Stetter (1976); Nimgirawath *et al.* (1976); Yamamoto *et al.* (2003); Yuguchi *et al.* (2004).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{12}\text{Cl}_2\text{O}_2$
 $M_r = 307.16$
 Monoclinic, $P2_1/c$
 $a = 10.3663$ (2) Å
 $b = 5.2532$ (1) Å
 $c = 26.1125$ (6) Å
 $\beta = 95.272$ (2)°

$V = 1415.97$ (5) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.46$ mm⁻¹
 $T = 298$ (2) K
 $0.20 \times 0.10 \times 0.10$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1997)
 $T_{\min} = 0.914$, $T_{\max} = 0.956$
 14170 measured reflections
 2789 independent reflections
 1836 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.117$
 $S = 0.97$
 2789 reflections
 181 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C8}-\text{H8B}\cdots\text{O1}^{\text{i}}$	0.97	2.59	3.553 (3)	173
$\text{C11}-\text{H11}\cdots\text{O1}^{\text{ii}}$	0.93	2.50	3.246 (2)	138
$\text{C8}-\text{H8A}\cdots\text{Cg1}^{\text{iii}}$	0.97	2.98	3.876 (2)	154

Symmetry codes: (i) $x, y + 1, z$; (ii) $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$. Cg1 is the centroid of C9–C14.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXTL.

The authors are grateful to Xiangfan University for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2197).

References

- Bruker (2001). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (1999). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
 Nimgirawath, S., Ritchie, E. & Taylor, W. C. (1976). *Aust. J. Chem.* **29**, 339–356.
 Rao, H. S. P., Jothilingam, S. & Scheeren, H. W. (2004). *Tetrahedron*, **60**, 1625–1630.
 Sheldrick, G. M. (1997). SADABS. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Shridhar, D. R., Jogibhukta, M., Rao, P. S. & Handa, V. K. (1982). *Synthesis*, pp. 1061–1062.
 Stauffer, F. & Neier, R. (2000). *Org. Lett.* **2**, 3535–3537.
 Stetter, H. (1976). *Angew. Chem. Int. Ed.* **15**, 639–648.
 Yamamoto, Y., Maekawa, H., Goda, S. & Nishiguchi, I. (2003). *Org. Lett.* **5**, 2755–2758.
 Yuguchi, M., Tokuda, M. & Orito, K. (2004). *J. Org. Chem.* **69**, 908–914.

supporting information

Acta Cryst. (2008). E64, o2470 [doi:10.1107/S1600536808039251]

1,4-Bis(4-chlorophenyl)butane-1,4-dione

Shuqin Li and Huisheng Li

S1. Comment

1,4-Diketones are very useful intermediates for the synthesis of substituted furans, pyrroles and thiophenes *via* Paal-Knorr cyclization reaction (Rao *et al.*, 2004; Stauffer *et al.*, 2000; Shridhar *et al.*, 1982). A variety of methods have been reported for the preparation of these 1,4-dicarbonyl compounds (Stetter *et al.*, 1976; Yamamoto *et al.*, 2003; Yuguchi *et al.*, 2004).

The molecule is centrosymmetric. There are two halves of the molecules in the asymmetric unit (Fig. 1). The two phenyl rings are co-planar in both molecules. Intermolecular C—H \cdots O hydrogen bonds and C—H \cdots π interactions stabilize the crystal packing (Table 1).

S2. Experimental

The title compound was synthesized as previously described by Nimgirawath *et al.* (1976). Colourless crystals suitable for X-ray data collection were obtained by slow evaporation of a 1:2 (v/v) ratio CH₂Cl₂:CH₃OH solution at 293 K.

S3. Refinement

All H atoms were refined independently; C—H (methyl H) = 0.96 Å, C—H (methine H) = 0.93 Å and 0.93 Å for thiophene H. The constraint $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$ was applied.

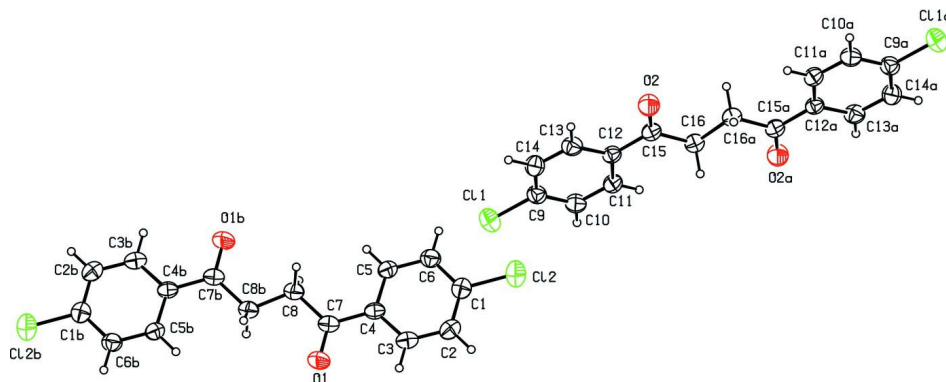


Figure 1

View of the title molecule showing the atom-labelling scheme. The displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by spheres of arbitrary radius.

1,4-Bis(4-chlorophenyl)butane-1,4-dione*Crystal data*C₁₆H₁₂Cl₂O₂ $M_r = 307.16$ Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

 $a = 10.3663$ (2) Å $b = 5.2532$ (1) Å $c = 26.1125$ (6) Å $\beta = 95.272$ (2)° $V = 1415.97$ (5) Å³ $Z = 4$ $F(000) = 632$ $D_x = 1.441$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 3417 reflections

 $\theta = 2.6$ – 23.1 ° $\mu = 0.46$ mm⁻¹ $T = 298$ K

Block, colourless

 $0.20 \times 0.10 \times 0.10$ mm*Data collection*

Bruker SMART CCD area-detector

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 1997)

 $T_{\min} = 0.914$, $T_{\max} = 0.956$

14170 measured reflections

2789 independent reflections

1836 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.058$ $\theta_{\text{max}} = 26.0$ °, $\theta_{\text{min}} = 1.6$ ° $h = -12$ → 12 $k = -6$ → 6 $l = -31$ → 32 *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.042$ $wR(F^2) = 0.117$ $S = 0.97$

2789 reflections

181 parameters

0 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0637P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\text{max}} = 0.001$ $\Delta\rho_{\text{max}} = 0.21$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.23$ e Å⁻³*Special details*

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3102 (2)	0.4151 (4)	0.32315 (8)	0.0609 (5)
C2	0.3437 (2)	0.2160 (4)	0.35628 (8)	0.0658 (6)
H2	0.4069	0.0995	0.3488	0.079*
C3	0.2824 (2)	0.1933 (4)	0.40024 (8)	0.0612 (6)

H3	0.3041	0.0584	0.4225	0.073*
C4	0.18851 (19)	0.3661 (3)	0.41264 (8)	0.0524 (5)
C5	0.1579 (2)	0.5660 (4)	0.37840 (8)	0.0590 (5)
H5	0.0960	0.6851	0.3859	0.071*
C6	0.2175 (2)	0.5899 (4)	0.33398 (8)	0.0627 (6)
H6	0.1955	0.7228	0.3113	0.075*
C7	0.1272 (2)	0.3362 (4)	0.46133 (8)	0.0559 (5)
C8	0.0298 (2)	0.5308 (4)	0.47540 (7)	0.0576 (5)
H8A	-0.0386	0.5432	0.4476	0.069*
H8B	0.0719	0.6955	0.4790	0.069*
C9	0.1559 (2)	0.7318 (4)	0.15908 (8)	0.0593 (5)
C10	0.1743 (2)	0.6200 (4)	0.11264 (8)	0.0623 (6)
H10	0.1275	0.4755	0.1019	0.075*
C11	0.2621 (2)	0.7227 (4)	0.08217 (8)	0.0575 (5)
H11	0.2745	0.6459	0.0509	0.069*
C12	0.33228 (18)	0.9383 (3)	0.09714 (7)	0.0508 (5)
C13	0.3118 (2)	1.0476 (4)	0.14417 (8)	0.0628 (6)
H13	0.3581	1.1926	0.1549	0.075*
C14	0.2249 (2)	0.9467 (4)	0.17514 (8)	0.0674 (6)
H14	0.2127	1.0220	0.2066	0.081*
C15	0.42674 (19)	1.0574 (4)	0.06459 (7)	0.0537 (5)
C16	0.45259 (19)	0.9285 (4)	0.01517 (8)	0.0558 (5)
H16A	0.3713	0.9095	-0.0061	0.067*
H16B	0.4865	0.7594	0.0229	0.067*
Cl1	0.04740 (6)	0.59996 (12)	0.19828 (2)	0.0828 (2)
Cl2	0.38806 (6)	0.44975 (15)	0.26764 (2)	0.0908 (3)
O1	0.15594 (16)	0.1582 (3)	0.48988 (6)	0.0830 (5)
O2	0.48192 (15)	1.2536 (3)	0.07810 (6)	0.0746 (5)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0561 (13)	0.0641 (12)	0.0619 (13)	-0.0073 (11)	0.0012 (10)	-0.0094 (10)
C2	0.0571 (13)	0.0598 (13)	0.0800 (15)	0.0045 (11)	0.0034 (12)	-0.0096 (12)
C3	0.0596 (14)	0.0472 (11)	0.0746 (14)	0.0029 (10)	-0.0055 (11)	0.0030 (10)
C4	0.0524 (12)	0.0432 (10)	0.0596 (12)	-0.0020 (9)	-0.0061 (10)	0.0006 (9)
C5	0.0639 (14)	0.0499 (11)	0.0632 (13)	0.0086 (10)	0.0063 (10)	0.0010 (10)
C6	0.0705 (15)	0.0559 (12)	0.0609 (13)	0.0026 (11)	0.0023 (11)	0.0044 (10)
C7	0.0566 (13)	0.0454 (11)	0.0637 (13)	-0.0034 (9)	-0.0049 (10)	0.0052 (9)
C8	0.0648 (14)	0.0473 (11)	0.0600 (12)	-0.0027 (10)	0.0017 (10)	0.0039 (9)
C9	0.0569 (13)	0.0607 (12)	0.0600 (13)	0.0043 (10)	0.0036 (10)	0.0077 (10)
C10	0.0634 (14)	0.0543 (12)	0.0682 (14)	-0.0080 (10)	0.0007 (11)	-0.0027 (10)
C11	0.0614 (13)	0.0531 (11)	0.0575 (12)	-0.0005 (10)	0.0036 (10)	-0.0074 (9)
C12	0.0530 (12)	0.0478 (10)	0.0504 (11)	0.0042 (9)	-0.0017 (9)	-0.0011 (9)
C13	0.0733 (15)	0.0528 (12)	0.0609 (13)	-0.0079 (11)	-0.0011 (11)	-0.0067 (10)
C14	0.0807 (16)	0.0673 (14)	0.0543 (12)	-0.0008 (12)	0.0064 (11)	-0.0065 (11)
C15	0.0502 (12)	0.0520 (11)	0.0577 (12)	0.0030 (10)	-0.0020 (9)	-0.0021 (9)
C16	0.0504 (12)	0.0559 (11)	0.0599 (12)	0.0011 (9)	-0.0012 (9)	-0.0014 (9)

C11	0.0788 (5)	0.0941 (5)	0.0772 (4)	-0.0099 (3)	0.0169 (3)	0.0112 (3)
C12	0.0818 (5)	0.1167 (6)	0.0764 (4)	0.0026 (4)	0.0215 (3)	-0.0058 (4)
O1	0.0957 (13)	0.0697 (10)	0.0855 (11)	0.0224 (9)	0.0175 (9)	0.0298 (9)
O2	0.0803 (11)	0.0631 (9)	0.0819 (11)	-0.0212 (8)	0.0150 (8)	-0.0150 (8)

Geometric parameters (Å, °)

C1—C6	1.377 (3)	C9—C14	1.381 (3)
C1—C2	1.381 (3)	C9—C11	1.733 (2)
C1—C12	1.732 (2)	C10—C11	1.373 (3)
C2—C3	1.368 (3)	C10—H10	0.9300
C2—H2	0.9300	C11—C12	1.383 (3)
C3—C4	1.391 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—C13	1.390 (3)
C4—C5	1.397 (3)	C12—C15	1.492 (3)
C4—C7	1.481 (3)	C13—C14	1.371 (3)
C5—C6	1.369 (3)	C13—H13	0.9300
C5—H5	0.9300	C14—H14	0.9300
C6—H6	0.9300	C15—O2	1.215 (2)
C7—O1	1.216 (2)	C15—C16	1.503 (3)
C7—C8	1.506 (3)	C16—C16 ⁱⁱ	1.517 (4)
C8—C8 ⁱ	1.511 (4)	C16—H16A	0.9700
C8—H8A	0.9700	C16—H16B	0.9700
C8—H8B	0.9700	C11—C12	3.8985 (9)
C9—C10	1.376 (3)		
C6—C1—C2	121.1 (2)	C10—C9—C11	120.01 (17)
C6—C1—C12	119.31 (17)	C14—C9—C11	119.58 (16)
C2—C1—C12	119.60 (18)	C11—C10—C9	119.75 (19)
C3—C2—C1	118.8 (2)	C11—C10—H10	120.1
C3—C2—H2	120.6	C9—C10—H10	120.1
C1—C2—H2	120.6	C10—C11—C12	121.17 (19)
C2—C3—C4	121.84 (19)	C10—C11—H11	119.4
C2—C3—H3	119.1	C12—C11—H11	119.4
C4—C3—H3	119.1	C11—C12—C13	117.95 (19)
C3—C4—C5	117.68 (19)	C11—C12—C15	122.57 (17)
C3—C4—C7	119.70 (18)	C13—C12—C15	119.47 (17)
C5—C4—C7	122.62 (19)	C14—C13—C12	121.59 (19)
C6—C5—C4	121.17 (19)	C14—C13—H13	119.2
C6—C5—H5	119.4	C12—C13—H13	119.2
C4—C5—H5	119.4	C13—C14—C9	119.2 (2)
C5—C6—C1	119.39 (19)	C13—C14—H14	120.4
C5—C6—H6	120.3	C9—C14—H14	120.4
C1—C6—H6	120.3	O2—C15—C12	120.42 (18)
O1—C7—C4	120.50 (19)	O2—C15—C16	120.99 (19)
O1—C7—C8	120.30 (19)	C12—C15—C16	118.58 (17)
C4—C7—C8	119.20 (16)	C15—C16—C16 ⁱⁱ	113.6 (2)
C7—C8—C8 ⁱ	113.5 (2)	C15—C16—H16A	108.8

C7—C8—H8A	108.9	C16 ⁱⁱ —C16—H16A	108.8
C8 ⁱ —C8—H8A	108.9	C15—C16—H16B	108.8
C7—C8—H8B	108.9	C16 ⁱⁱ —C16—H16B	108.8
C8 ⁱ —C8—H8B	108.9	H16A—C16—H16B	107.7
H8A—C8—H8B	107.7	C9—C11—C12	75.29 (7)
C10—C9—C14	120.4 (2)	C1—C12—C11	86.46 (7)
C6—C1—C2—C3	0.5 (3)	C10—C11—C12—C13	0.3 (3)
Cl2—C1—C2—C3	179.23 (15)	C10—C11—C12—C15	-178.93 (18)
C1—C2—C3—C4	-0.6 (3)	C11—C12—C13—C14	0.0 (3)
C2—C3—C4—C5	0.2 (3)	C15—C12—C13—C14	179.26 (19)
C2—C3—C4—C7	-178.57 (18)	C12—C13—C14—C9	-0.2 (3)
C3—C4—C5—C6	0.5 (3)	C10—C9—C14—C13	0.2 (3)
C7—C4—C5—C6	179.23 (18)	C11—C9—C14—C13	179.13 (16)
C4—C5—C6—C1	-0.7 (3)	C11—C12—C15—O2	176.53 (19)
C2—C1—C6—C5	0.2 (3)	C13—C12—C15—O2	-2.7 (3)
Cl2—C1—C6—C5	-178.55 (15)	C11—C12—C15—C16	-3.9 (3)
C3—C4—C7—O1	-1.9 (3)	C13—C12—C15—C16	176.85 (17)
C5—C4—C7—O1	179.45 (18)	O2—C15—C16—C16 ⁱⁱ	-2.1 (3)
C3—C4—C7—C8	177.44 (18)	C12—C15—C16—C16 ⁱⁱ	178.39 (19)
C5—C4—C7—C8	-1.2 (3)	C10—C9—C11—C12	107.86 (17)
O1—C7—C8—C8 ⁱ	-3.6 (3)	C14—C9—C11—C12	-71.11 (16)
C4—C7—C8—C8 ⁱ	177.0 (2)	C6—C1—C12—C11	-43.73 (16)
C14—C9—C10—C11	0.1 (3)	C2—C1—C12—C11	137.48 (16)
Cl1—C9—C10—C11	-178.83 (15)	C9—C11—C12—C1	158.48 (10)
C9—C10—C11—C12	-0.4 (3)		

Symmetry codes: (i) $-x, -y+1, -z+1$; (ii) $-x+1, -y+2, -z$.

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C8—H8B \cdots O1 ⁱⁱⁱ	0.97	2.59	3.553 (3)	173
C11—H11 \cdots O1 ^{iv}	0.93	2.50	3.246 (2)	138
C8—H8A \cdots Cg1 ^v	0.97	2.98	3.876 (2)	154

Symmetry codes: (iii) $x, y+1, z$; (iv) $x, -y+1/2, z-1/2$; (v) $-x, y-1/2, -z+1/2$.