organic compounds

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

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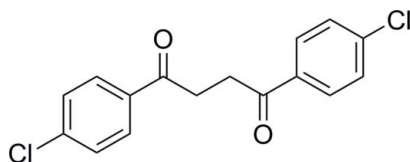Correspondence e-mail: shuqin2000@yahoo.com.cn

The molecule of title compound, $C_{16}H_{12}Cl_2O_2$, 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 C—H···O hydrogen bonds and C—H···π 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

$C_{16}H_{12}Cl_2O_2$	V = 1415.97 (5) Å^3
M_r = 307.16	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 10.3663 (2) Å	μ = 0.46 mm^{-1}
b = 5.2532 (1) Å	T = 298 (2) K
c = 26.1125 (6) Å	$0.20 \times 0.10 \times 0.10$ mm
β = 95.272 (2)°	

Data collection

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

Refinement

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

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8B···O1^{i}	0.97	2.59	3.553 (3)	173
C11—H11···O1^{ii}	0.93	2.50	3.246 (2)	138
C8—H8A···Cg1^{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*.

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.* A**64**, 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.

supplementary materials

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

S. Li and H. Li

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⋯O hydrogen bonds and C—H⋯π interactions stabilize the crystal packing (Table 1).

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 CH_2Cl_2:CH_3OH solution at 293 K.

Refinement

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

Figures
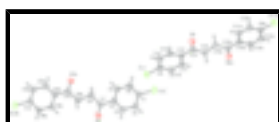
Fig. 1. View of the title molecule showing the atom-labelling scheme. The displacement ellipsoids are drawn at the 30% probability level. H atoms are represented by spheres of arbitrary radius.

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

Crystal data

$C_{16}H_{12}Cl_2O_2$	$F_{000} = 632$
$M_r = 307.16$	$D_x = 1.441$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 3417 reflections
$a = 10.3663$ (2) Å	$\theta = 2.6–23.1°$
$b = 5.25320$ (10) Å	$\mu = 0.46$ mm^{-1}
$c = 26.1125$ (6) Å	$T = 298$ (2) K

$\beta = 95.272\ (2)^\circ$	Block, colourless
$V = 1415.97\ (5)\ \text{Å}^3$	$0.20 \times 0.10 \times 0.10$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2789 independent reflections
Radiation source: fine-focus sealed tube	1836 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.058$
$T = 298(2)$ K	$\theta_{max} = 26.0^\circ$
φ and ω scans	$\theta_{min} = 1.6^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	$h = -12 \rightarrow 12$
$T_{min} = 0.914$, $T_{max} = 0.956$	$k = -6 \rightarrow 6$
14170 measured reflections	$l = -31 \rightarrow 32$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.042$	H-atom parameters constrained
$wR(F^2) = 0.117$	$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{max} = 0.001$
2789 reflections	$\Delta\rho_{max} = 0.21$ e Å^{-3}
181 parameters	$\Delta\rho_{min} = -0.23$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 ($Å^2$)

	x	y	z	$U_{iso}*/U_{eq}$
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 (Å²)

	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)
Cl1	0.0788 (5)	0.0941 (5)	0.0772 (4)	−0.0099 (3)	0.0169 (3)	0.0112 (3)
Cl2	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—Cl1	1.733 (2)
C1—Cl2	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	Cl1—Cl2	3.8985 (9)
C9—C10	1.376 (3)		
C6—C1—C2	121.1 (2)	C10—C9—Cl1	120.01 (17)
C6—C1—Cl2	119.31 (17)	C14—C9—Cl1	119.58 (16)
C2—C1—Cl2	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—Cl1—Cl2	75.29 (7)
C10—C9—C14	120.4 (2)	C1—Cl2—Cl1	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)	Cl1—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—Cl1—Cl2	107.86 (17)
O1—C7—C8—C8[i]	−3.6 (3)	C14—C9—Cl1—Cl2	−71.11 (16)
C4—C7—C8—C8[i]	177.0 (2)	C6—C1—Cl2—Cl1	−43.73 (16)
C14—C9—C10—C11	0.1 (3)	C2—C1—Cl2—Cl1	137.48 (16)
Cl1—C9—C10—C11	−178.83 (15)	C9—Cl1—Cl2—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, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8B···O1[iii]	0.97	2.59	3.553 (3)	173
C11—H11···O1[iv]	0.93	2.50	3.246 (2)	138
C8—H8A···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.

Fig. 1