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1,4-Diphenylbutane-1,4-dione

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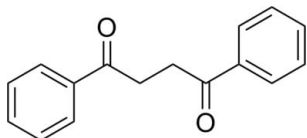
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
R factor = 0.054; wR factor = 0.129; data-to-parameter ratio = 9.2.

The asymmetric unit of the title compound, $\text{C}_{16}\text{H}_{14}\text{O}_2$, contains one half-molecule, located on a twofold rotation axis. In the molecule, the two benzene rings form a dihedral angle of $72.28(2)^\circ$.

Related literature

For useful applications of 1,4-dicarbonyl compounds, see: Chiu & Sammes (1990); Greatrex *et al.* (2003); Nagarajan & Shechter (1984). For details of the synthesis, see Nevar *et al.* (2000).



Experimental

Crystal data

 $\text{C}_{16}\text{H}_{14}\text{O}_2$ $M_r = 238.27$ Orthorhombic, $P2_12_12$
 $a = 8.3781(13)$ Å
 $b = 14.161(2)$ Å
 $c = 5.3186(8)$ Å
 $V = 631.00(17)$ Å³ $Z = 2$
Mo $K\alpha$ radiation
 $\mu = 0.08$ 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)
 $T_{\min} = 0.984$, $T_{\max} = 0.992$ 4063 measured reflections
762 independent reflections
640 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.163$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.129$
 $S = 1.05$
762 reflections83 parameters
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.22$ e Å⁻³

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; 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 author is grateful to Ling Fan for a valuable discussion.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2479).

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supplementary materials

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1,4-Diphenylbutane-1,4-dione

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Comment

1,4-Dicarbonyl compounds constitute key intermediates in various natural product syntheses, and they are important synthetic precursors of cyclopentenones, cyclopenta-1,3-diones, butenolides, and derivatives of furan and pyrrole (Chiu & Sammes, 1990; Greatrex *et al.*, 2003; Nagarajan & Shechter, 1984). Herewith we present the title compound (I) (Fig. 1). The asymmetric unit of (I) contains a half of the molecule located on a twofold rotational axis. Two benzene rings form a dihedral angle of 72.28 (2)°.

Experimental

The title compound was synthesized as previously described by Nevar *et al.* (2000). Colourless crystals suitable for X-ray data collection were obtained by slow evaporation of a 1:3 ratio EtOAc:cyclohexane solution at room temperature.

Refinement

All H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding, allowing for free rotation of the methyl groups. The constraint $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ or $1.5 U_{\text{eq}}(\text{C})$ (methyl C) was applied. In the absence of anomalous scatterers, no attempt was made to establish the absolute configuration of the title compound, and 488 Friedel pairs were merged before the final refinement.

Figures

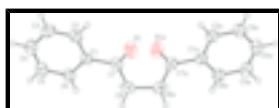


Fig. 1. View of the title molecule showing the atom-labelling scheme. The displacement ellipsoids are drawn at the 30% probability level [symmetry code: (a) $-x, -y + 1, z$].

1,4-Diphenylbutane-1,4-dione

Crystal data

$\text{C}_{16}\text{H}_{14}\text{O}_2$

$M_r = 238.27$

Orthorhombic, $P2_12_12$

Hall symbol: P 2 2ab

$a = 8.3781$ (13) Å

$b = 14.161$ (2) Å

$c = 5.3186$ (8) Å

$F_{000} = 252$

$D_x = 1.254$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 1423 reflections

$\theta = 2.8$ – 22.3°

$\mu = 0.08$ mm⁻¹

$T = 298$ (2) K

supplementary materials

$V = 631.00 (17) \text{ \AA}^3$
 $Z = 2$

Block, colourless
 $0.20 \times 0.10 \times 0.10 \text{ mm}$

Data collection

| | |
|---|---------------------------------------|
| Bruker SMART CCD area-detector diffractometer | 762 independent reflections |
| Radiation source: fine-focus sealed tube | 640 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.163$ |
| $T = 298(2) \text{ K}$ | $\theta_{\text{max}} = 26.0^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 2.8^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1997) | $h = -10 \rightarrow 9$ |
| $T_{\text{min}} = 0.984$, $T_{\text{max}} = 0.992$ | $k = -16 \rightarrow 17$ |
| 4063 measured reflections | $l = -6 \rightarrow 6$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.054$ | H-atom parameters constrained |
| $wR(F^2) = 0.129$ | $w = 1/[\sigma^2(F_o^2) + (0.0734P)^2]$ |
| $S = 1.05$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 762 reflections | $(\Delta/\sigma)_{\text{max}} = 0.012$ |
| 83 parameters | $\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$ |
| | 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 (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|------------|--------------|------------|----------------------------------|
| C1 | 0.1095 (3) | 0.30567 (15) | 0.2151 (4) | 0.0508 (6) |
| C7 | 0.1070 (3) | 0.40988 (16) | 0.1653 (5) | 0.0574 (7) |
| C4 | 0.1208 (3) | 0.11385 (19) | 0.3179 (6) | 0.0699 (7) |

| | | | | |
|-----|------------|--------------|-------------|------------|
| H4 | 0.1241 | 0.0496 | 0.3532 | 0.084* |
| C8 | 0.0072 (4) | 0.44647 (16) | -0.0468 (5) | 0.0670 (7) |
| H8A | -0.0986 | 0.4191 | -0.0354 | 0.080* |
| H8B | 0.0540 | 0.4263 | -0.2047 | 0.080* |
| C3 | 0.2031 (3) | 0.17678 (17) | 0.4670 (5) | 0.0704 (8) |
| H3 | 0.2625 | 0.1548 | 0.6026 | 0.085* |
| C6 | 0.0283 (3) | 0.24197 (17) | 0.0654 (5) | 0.0608 (7) |
| H6 | -0.0307 | 0.2634 | -0.0713 | 0.073* |
| C2 | 0.1980 (3) | 0.27214 (16) | 0.4163 (5) | 0.0608 (7) |
| H2 | 0.2541 | 0.3142 | 0.5176 | 0.073* |
| O1 | 0.1843 (3) | 0.46221 (13) | 0.2966 (5) | 0.0984 (8) |
| C5 | 0.0345 (3) | 0.14619 (18) | 0.1188 (6) | 0.0712 (8) |
| H5 | -0.0206 | 0.1037 | 0.0177 | 0.085* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|--------------|--------------|--------------|
| C1 | 0.0464 (11) | 0.0571 (13) | 0.0490 (11) | 0.0054 (10) | 0.0025 (10) | -0.0053 (10) |
| C7 | 0.0558 (13) | 0.0561 (13) | 0.0603 (14) | 0.0011 (11) | -0.0034 (12) | -0.0105 (11) |
| C4 | 0.0636 (15) | 0.0595 (13) | 0.0867 (18) | 0.0061 (13) | 0.0053 (15) | 0.0043 (15) |
| C8 | 0.0839 (17) | 0.0614 (14) | 0.0555 (13) | 0.0048 (13) | -0.0040 (14) | -0.0074 (11) |
| C3 | 0.0658 (16) | 0.0769 (17) | 0.0686 (16) | 0.0155 (14) | -0.0023 (15) | 0.0091 (15) |
| C6 | 0.0613 (14) | 0.0604 (13) | 0.0607 (13) | -0.0011 (11) | -0.0092 (13) | -0.0065 (12) |
| C2 | 0.0560 (14) | 0.0676 (14) | 0.0589 (14) | 0.0046 (12) | -0.0060 (12) | -0.0094 (12) |
| O1 | 0.1149 (17) | 0.0632 (11) | 0.1172 (17) | -0.0067 (11) | -0.0544 (16) | -0.0120 (12) |
| C5 | 0.0696 (17) | 0.0597 (14) | 0.0843 (17) | -0.0059 (13) | -0.0060 (16) | -0.0117 (14) |

Geometric parameters (\AA , $^\circ$)

| | | | |
|-----------------------|-------------|------------|-----------|
| C1—C6 | 1.382 (3) | C8—H8A | 0.9700 |
| C1—C2 | 1.386 (3) | C8—H8B | 0.9700 |
| C1—C7 | 1.499 (3) | C3—C2 | 1.378 (3) |
| C7—O1 | 1.207 (3) | C3—H3 | 0.9300 |
| C7—C8 | 1.496 (3) | C6—C5 | 1.387 (4) |
| C4—C5 | 1.361 (4) | C6—H6 | 0.9300 |
| C4—C3 | 1.378 (3) | C2—H2 | 0.9300 |
| C4—H4 | 0.9300 | C5—H5 | 0.9300 |
| C8—C8 ⁱ | 1.521 (4) | | |
| C6—C1—C2 | 119.0 (2) | H8A—C8—H8B | 107.8 |
| C6—C1—C7 | 122.3 (2) | C4—C3—C2 | 120.4 (2) |
| C2—C1—C7 | 118.74 (19) | C4—C3—H3 | 119.8 |
| O1—C7—C8 | 121.5 (2) | C2—C3—H3 | 119.8 |
| O1—C7—C1 | 119.7 (2) | C1—C6—C5 | 120.1 (2) |
| C8—C7—C1 | 118.8 (2) | C1—C6—H6 | 119.9 |
| C5—C4—C3 | 119.7 (2) | C5—C6—H6 | 119.9 |
| C5—C4—H4 | 120.1 | C3—C2—C1 | 120.2 (2) |
| C3—C4—H4 | 120.1 | C3—C2—H2 | 119.9 |
| C7—C8—C8 ⁱ | 112.9 (2) | C1—C2—H2 | 119.9 |

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| | | | |
|--------------------------|------------|-------------|------------|
| C7—C8—H8A | 109.0 | C4—C5—C6 | 120.6 (2) |
| C8 ⁱ —C8—H8A | 109.0 | C4—C5—H5 | 119.7 |
| C7—C8—H8B | 109.0 | C6—C5—H5 | 119.7 |
| C8 ⁱ —C8—H8B | 109.0 | | |
| C6—C1—C7—O1 | -177.5 (3) | C2—C1—C6—C5 | 0.7 (3) |
| C2—C1—C7—O1 | 1.7 (4) | C7—C1—C6—C5 | 179.8 (3) |
| C6—C1—C7—C8 | 2.8 (3) | C4—C3—C2—C1 | 0.2 (4) |
| C2—C1—C7—C8 | -178.0 (2) | C6—C1—C2—C3 | -0.6 (4) |
| O1—C7—C8—C8 ⁱ | -9.8 (4) | C7—C1—C2—C3 | -179.9 (2) |
| C1—C7—C8—C8 ⁱ | 169.9 (3) | C3—C4—C5—C6 | -0.3 (4) |
| C5—C4—C3—C2 | 0.3 (4) | C1—C6—C5—C4 | -0.2 (4) |

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

Fig. 1

