Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(4-Bromobenzyl)-1,3-diphenylpropane-1,3-dione

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Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.031
wR factor = 0.085
Data-to-parameter ratio = 8.3

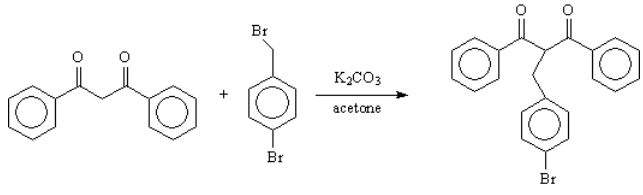
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 September 2004
Accepted 15 October 2004
Online 22 October 2004

The title compound, $C_{22}H_{17}BrO_2$, was obtained from the monoalkylation reaction of dibenzoylmethane with 4-bromo-1-(bromomethyl)benzene catalyzed by anhydrous potassium carbonate in acetone. A 1:1 ratio of dibenzoylmethane to *p*-bromobenzyl bromide was utilized to produce this compound.

Comment

Stable ozonides are known to be synthesized from the reaction of ozone with inden-1-ones. The title compound, (I), was synthesized for use as a precursor to a substituted inden-1-one. Compound (I) is part of a larger study on the stability of ozonides from inden-1-one derivatives. Various substituted ozonides were synthesized and were found to be stable for several days.



(I)

The reaction followed a modified procedure of Johnson *et al.* (1973), in which the monoalkylation of 4-bromo-1-(bromomethyl)benzene with propane-1,3-dione formed (I) in the presence of K_2CO_3 in acetone, as shown in the reaction scheme.

The molecular structure of (I) is shown in Fig. 1 and selected geometric parameters are listed in Table 1. All bond lengths and angles are within normally observed ranges. The C1—C2—C3 angle of 107.1 (2)° is contracted slightly from ideal tetrahedral. The two benzoyl groups are twisted such that the angle formed between their mean planes is 84.97 (7)°. Long-range interactions (Table 2) between atom O1 of one molecule and atom H13 of an adjoining molecule were noticed and appear to provide further stablility to the compound in its crystalline form.

Experimental

The title compound was prepared by the reaction of 4-bromo-1-(bromomethyl)benzene (5.85 g, 23.4 mmol) with propane-1,3-dione (5.00 g, 22.3 mmol) in dry acetone over 18 h under reflux. The product was isolated in 89% (7.8 g, 19.8 mmol) yield (Kalyanam *et al.*, 1979) by recrystallization from ethanol.

Crystal data

$C_{22}H_{17}BrO_2$
M_r = 393.27
Monoclinic, $P2_1$
a = 8.7227 (13) Å
b = 5.8099 (9) Å
c = 18.074 (3) Å
β = 103.203 (6)°
V = 891.8 (2) Å^3
Z = 2
D_x = 1.465 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3450 reflections
θ = 2.3–23.8°
μ = 2.32 mm^{-1}
T = 298 (2) K
Needle, colorless
0.37 × 0.09 × 0.06 mm

Data collection

Bruker APEX2 CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.725, T_{max} = 0.869
7930 measured reflections
1874 independent reflections
1600 reflections with $I > 2\sigma(I)$
R_{int} = 0.034
θ_{max} = 25.8°
$h = -10 \rightarrow 10$
$k = -7 \rightarrow 6$
$l = -21 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.09
1874 reflections
226 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.029
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.030 (11)

Table 1
Selected geometric parameters (Å, °).

Br1—C20	1.895 (3)	O2—C3	1.214 (4)
O1—C1	1.214 (4)		
C1—C2—C3	107.1 (3)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C13—H13···O1^{i}	0.93	2.54	3.157 (5)	124

Symmetry code: (i) $-x, \frac{1}{2}+y, 1-z$.

H atoms were included in calculated positions (C—H = 0.93 Å) and refined as riding, with U_{iso}(H) = 1.2U_{iso}(C).

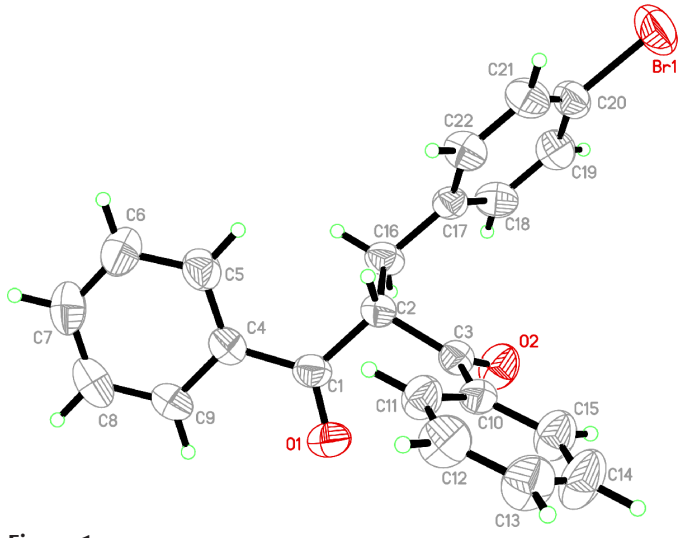


Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level

Data collection: *APEX*2 (Bruker, 2003); cell refinement: *APEX*2; data reduction: *SAINT-Plus* (Bruker, 2003); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2003); software used to prepare material for publication: *SHELXTL*.

The Bruker X8 APEX diffractometer was purchased with funds received from the National Science Foundation Major Research Instrumentation Program Grant CHE-0321214. KK thanks the Robert A. Welch Foundation for support (AA-1508).

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