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2-(4-Bromophenyl)-2-oxoethyl 2-methoxybenzoate

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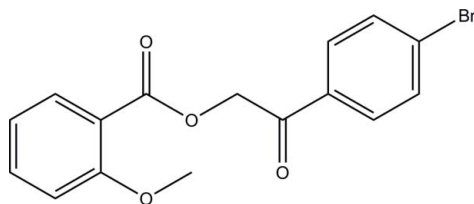
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.037; wR factor = 0.095; data-to-parameter ratio = 24.9.

In the title molecule, $\text{C}_{16}\text{H}_{13}\text{BrO}_4$, the dihedral angle between the benzene rings is $85.92(10)^\circ$. In the crystal, molecules are linked into chains along $[100]$ via weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For general background to and applications of phenacyl benzoate derivatives, see: Rather & Reid (1919); Sheehan & Umezaw (1973); Ruzicka *et al.* (2002); Litera *et al.* (2006); Huang *et al.* (1996); Gandhi *et al.* (1995). For standard bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$\text{C}_{16}\text{H}_{13}\text{BrO}_4$	$V = 2966.5(3)$ Å ³
$M_r = 349.17$	$Z = 8$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation
$a = 7.8424(5)$ Å	$\mu = 2.78$ mm ⁻¹
$b = 14.6799(9)$ Å	$T = 296$ K
$c = 25.7677(14)$ Å	$0.56 \times 0.25 \times 0.12$ mm

Data collection

Bruker SMART APEXII DUO	19358 measured reflections
CCD area-detector	4731 independent reflections
diffractometer	2916 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan	$R_{\text{int}} = 0.035$
(<i>SADABS</i> ; Bruker, 2009)	
$T_{\text{min}} = 0.306$, $T_{\text{max}} = 0.733$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	190 parameters
$wR(F^2) = 0.095$	H-atom parameters constrained
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.51$ e Å ⁻³
4731 reflections	$\Delta\rho_{\text{min}} = -0.54$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C2}-\text{H2A}\cdots\text{O1}^i$	0.93	2.45	3.360 (3)	165

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5263).

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supplementary materials

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2-(4-Bromophenyl)-2-oxoethyl 2-methoxybenzoate

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Comment

Phenacyl benzoates derivatives are very important in the identification of organic acids (Rather & Reid, 1919). They undergo photolysis in neutral and mild conditions (Sheehan & Umezaw, 1973; Ruzicka *et al.*, 2002; Litera *et al.*, 2006). They find applications in the field of synthetic chemistry for the synthesis of oxazoles, imidazoles (Huang *et al.*, 1996), benzoxazepine (Gandhi *et al.*, 1995). We report herein the crystal structure of 2-(4-bromophenyl)-2-oxoethyl 2-methoxybenzoate which is potentially of commercial importance.

The molecular structure of the title compound is shown in Fig. 1. The benzene rings (C1-C6 and C10-C15) form a dihedral angle of $85.92(10)^\circ$. Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. In the crystal (Fig. 2), the molecules are linked into one-dimensional chains along [100] *via* weak intermolecular C2–H2A \cdots O1ⁱ hydrogen bonds (Table 1).

Experimental

A mixture of 2-methoxybenzoic acid (1.00 g, 0.0065 mol) potassium carbonate (0.98 g, 0.0071 mol) and 2-bromo-1-(4-bromophenyl)ethanone (1.80 g, 0.0065 mol) in dimethylformamide (10 ml) was stirred at room temperature for 2 h. On cooling, colorless needle shaped crystals of 2-(4-bromophenyl)-2-oxoethyl 2-methoxybenzoate began to separate. These were collected by filtration and recrystallized from ethanol. Yield: 2.15 g, 93.8 %, *m.p.* : 388–389 K.

Refinement

All H atoms were positioned geometrically and refined using a riding model with C–H = 0.93 – 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2$ or $1.5 U_{\text{eq}}(\text{C})$. The highest residual electron density peak is located at 0.79 Å and the deepest hole is located at 0.78 Å from Br1, respectively.

Figures

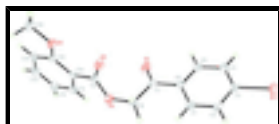


Fig. 1. The molecular structure of the title compound showing 20% probability displacement ellipsoids for non-H atoms.

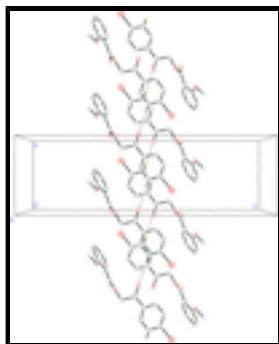


Fig. 2. Part of the crystal structure of the title compound, viewed along the *b* axis. H atoms not involved in hydrogen bonds (dashed lines) have been omitted for clarity.

2-(4-Bromophenyl)-2-oxoethyl 2-methoxybenzoate

Crystal data

$C_{16}H_{13}BrO_4$	$F(000) = 1408$
$M_r = 349.17$	$D_x = 1.564 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbca</i>	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ac 2ab	Cell parameters from 3938 reflections
$a = 7.8424 (5) \text{ \AA}$	$\theta = 2.8\text{--}26.5^\circ$
$b = 14.6799 (9) \text{ \AA}$	$\mu = 2.78 \text{ mm}^{-1}$
$c = 25.7677 (14) \text{ \AA}$	$T = 296 \text{ K}$
$V = 2966.5 (3) \text{ \AA}^3$	Needle, colourless
$Z = 8$	$0.56 \times 0.25 \times 0.12 \text{ mm}$

Data collection

Bruker SMART APEXII DUO CCD area-detector diffractometer	4731 independent reflections
Radiation source: fine-focus sealed tube graphite	2916 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.035$
Absorption correction: multi-scan (<i>SADABS</i> ; Bruker, 2009)	$\theta_{\text{max}} = 31.0^\circ$, $\theta_{\text{min}} = 2.8^\circ$
$T_{\text{min}} = 0.306$, $T_{\text{max}} = 0.733$	$h = -11 \rightarrow 11$
19358 measured reflections	$k = -21 \rightarrow 14$
	$l = -37 \rightarrow 37$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.037$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.095$	H-atom parameters constrained
$S = 1.00$	$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2 + 1.2401P]$
4731 reflections	where $P = (F_o^2 + 2F_c^2)/3$
	$(\Delta/\sigma)_{\text{max}} = 0.001$

190 parameters

$$\Delta\rho_{\max} = 0.51 \text{ e } \text{\AA}^{-3}$$

0 restraints

$$\Delta\rho_{\min} = -0.54 \text{ e } \text{\AA}^{-3}$$

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\sigma(F^2)$ 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	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	1.19995 (4)	0.063553 (17)	0.603716 (11)	0.07024 (11)
O1	0.4583 (2)	0.13020 (14)	0.46925 (7)	0.0717 (5)
O2	0.4899 (2)	0.19606 (9)	0.37415 (6)	0.0579 (4)
O3	0.4628 (2)	0.04790 (10)	0.35626 (6)	0.0604 (4)
O4	0.19571 (19)	0.00663 (10)	0.29651 (6)	0.0544 (4)
C1	0.9183 (3)	0.14467 (14)	0.47702 (8)	0.0488 (5)
H1A	0.9406	0.1695	0.4445	0.059*
C2	1.0517 (3)	0.12695 (15)	0.51036 (8)	0.0517 (5)
H2A	1.1635	0.1396	0.5007	0.062*
C3	1.0158 (3)	0.09006 (13)	0.55825 (8)	0.0487 (5)
C4	0.8516 (3)	0.07055 (14)	0.57344 (8)	0.0538 (5)
H4A	0.8302	0.0457	0.6060	0.065*
C5	0.7199 (3)	0.08825 (15)	0.53991 (8)	0.0504 (5)
H5A	0.6087	0.0749	0.5498	0.060*
C6	0.7504 (3)	0.12584 (13)	0.49133 (7)	0.0428 (4)
C7	0.6034 (3)	0.14284 (14)	0.45588 (8)	0.0470 (4)
C8	0.6423 (3)	0.17663 (14)	0.40202 (8)	0.0496 (5)
H8A	0.7113	0.2313	0.4042	0.059*
H8B	0.7073	0.1308	0.3835	0.059*
C9	0.4043 (3)	0.12319 (13)	0.35557 (7)	0.0444 (4)
C10	0.2356 (3)	0.15108 (13)	0.33476 (7)	0.0420 (4)
C11	0.1753 (3)	0.23898 (15)	0.34381 (8)	0.0540 (5)
H11A	0.2411	0.2790	0.3633	0.065*
C12	0.0207 (3)	0.26793 (17)	0.32457 (9)	0.0654 (6)
H12A	-0.0176	0.3268	0.3311	0.078*
C13	-0.0762 (3)	0.20873 (18)	0.29561 (9)	0.0643 (6)
H13A	-0.1803	0.2280	0.2823	0.077*
C14	-0.0216 (3)	0.12187 (16)	0.28619 (8)	0.0551 (5)
H14A	-0.0894	0.0827	0.2668	0.066*
C15	0.1337 (3)	0.09149 (13)	0.30522 (7)	0.0442 (4)

supplementary materials

C16	0.0967 (4)	-0.05475 (17)	0.26574 (11)	0.0735 (7)
H16A	0.1556	-0.1118	0.2627	0.110*
H16B	0.0799	-0.0292	0.2318	0.110*
H16C	-0.0120	-0.0644	0.2820	0.110*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.07526 (19)	0.05554 (15)	0.07991 (18)	0.00639 (12)	-0.03345 (13)	-0.00558 (11)
O1	0.0385 (8)	0.1055 (14)	0.0713 (10)	-0.0059 (9)	0.0015 (8)	-0.0039 (10)
O2	0.0574 (9)	0.0434 (8)	0.0729 (9)	-0.0015 (7)	-0.0214 (8)	0.0012 (7)
O3	0.0584 (9)	0.0481 (8)	0.0746 (10)	0.0109 (7)	-0.0218 (8)	-0.0110 (7)
O4	0.0577 (9)	0.0459 (7)	0.0595 (8)	-0.0012 (7)	-0.0141 (7)	-0.0071 (6)
C1	0.0433 (11)	0.0560 (11)	0.0472 (10)	-0.0059 (10)	0.0009 (9)	-0.0014 (9)
C2	0.0372 (10)	0.0562 (12)	0.0617 (12)	-0.0049 (9)	-0.0018 (9)	-0.0084 (10)
C3	0.0535 (12)	0.0381 (9)	0.0544 (11)	0.0025 (9)	-0.0136 (10)	-0.0098 (8)
C4	0.0634 (14)	0.0506 (11)	0.0474 (10)	-0.0032 (11)	-0.0006 (10)	-0.0014 (9)
C5	0.0430 (11)	0.0542 (11)	0.0540 (11)	-0.0053 (10)	0.0062 (9)	-0.0046 (9)
C6	0.0381 (9)	0.0421 (10)	0.0481 (10)	-0.0023 (8)	0.0013 (8)	-0.0093 (8)
C7	0.0398 (11)	0.0466 (10)	0.0547 (11)	-0.0035 (9)	-0.0017 (9)	-0.0102 (8)
C8	0.0444 (11)	0.0434 (10)	0.0608 (12)	-0.0064 (9)	-0.0111 (9)	0.0010 (9)
C9	0.0505 (12)	0.0430 (10)	0.0398 (9)	0.0012 (9)	-0.0048 (8)	-0.0011 (8)
C10	0.0453 (10)	0.0437 (9)	0.0371 (8)	0.0042 (8)	-0.0024 (8)	0.0010 (7)
C11	0.0603 (14)	0.0510 (11)	0.0508 (11)	0.0078 (10)	-0.0030 (10)	-0.0057 (9)
C12	0.0679 (15)	0.0613 (14)	0.0671 (14)	0.0228 (13)	-0.0041 (12)	-0.0009 (11)
C13	0.0505 (13)	0.0780 (16)	0.0646 (13)	0.0163 (12)	-0.0071 (11)	0.0099 (12)
C14	0.0486 (12)	0.0654 (14)	0.0513 (11)	-0.0005 (11)	-0.0093 (10)	0.0052 (10)
C15	0.0475 (11)	0.0490 (10)	0.0362 (8)	0.0017 (9)	0.0003 (8)	0.0040 (7)
C16	0.0819 (18)	0.0597 (14)	0.0788 (16)	-0.0071 (13)	-0.0264 (15)	-0.0160 (11)

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

Br1—C3	1.900 (2)	C7—C8	1.505 (3)
O1—C7	1.203 (3)	C8—H8A	0.9700
O2—C9	1.351 (2)	C8—H8B	0.9700
O2—C8	1.423 (2)	C9—C10	1.485 (3)
O3—C9	1.197 (2)	C10—C11	1.394 (3)
O4—C15	1.356 (2)	C10—C15	1.408 (3)
O4—C16	1.430 (3)	C11—C12	1.377 (3)
C1—C2	1.378 (3)	C11—H11A	0.9300
C1—C6	1.395 (3)	C12—C13	1.375 (3)
C1—H1A	0.9300	C12—H12A	0.9300
C2—C3	1.377 (3)	C13—C14	1.367 (3)
C2—H2A	0.9300	C13—H13A	0.9300
C3—C4	1.376 (3)	C14—C15	1.386 (3)
C4—C5	1.371 (3)	C14—H14A	0.9300
C4—H4A	0.9300	C16—H16A	0.9600
C5—C6	1.389 (3)	C16—H16B	0.9600
C5—H5A	0.9300	C16—H16C	0.9600

C6—C7	1.492 (3)		
C9—O2—C8	115.94 (15)	H8A—C8—H8B	108.0
C15—O4—C16	118.42 (17)	O3—C9—O2	122.39 (19)
C2—C1—C6	120.95 (19)	O3—C9—C10	126.97 (18)
C2—C1—H1A	119.5	O2—C9—C10	110.63 (16)
C6—C1—H1A	119.5	C11—C10—C15	118.24 (19)
C3—C2—C1	118.55 (19)	C11—C10—C9	119.80 (18)
C3—C2—H2A	120.7	C15—C10—C9	121.96 (17)
C1—C2—H2A	120.7	C12—C11—C10	121.6 (2)
C4—C3—C2	121.9 (2)	C12—C11—H11A	119.2
C4—C3—Br1	119.56 (16)	C10—C11—H11A	119.2
C2—C3—Br1	118.56 (17)	C13—C12—C11	119.2 (2)
C5—C4—C3	119.1 (2)	C13—C12—H12A	120.4
C5—C4—H4A	120.5	C11—C12—H12A	120.4
C3—C4—H4A	120.5	C14—C13—C12	120.8 (2)
C4—C5—C6	120.9 (2)	C14—C13—H13A	119.6
C4—C5—H5A	119.5	C12—C13—H13A	119.6
C6—C5—H5A	119.5	C13—C14—C15	120.8 (2)
C5—C6—C1	118.64 (19)	C13—C14—H14A	119.6
C5—C6—C7	119.04 (18)	C15—C14—H14A	119.6
C1—C6—C7	122.30 (18)	O4—C15—C14	123.50 (19)
O1—C7—C6	121.99 (19)	O4—C15—C10	117.17 (17)
O1—C7—C8	120.4 (2)	C14—C15—C10	119.32 (19)
C6—C7—C8	117.61 (18)	O4—C16—H16A	109.5
O2—C8—C7	111.19 (18)	O4—C16—H16B	109.5
O2—C8—H8A	109.4	H16A—C16—H16B	109.5
C7—C8—H8A	109.4	O4—C16—H16C	109.5
O2—C8—H8B	109.4	H16A—C16—H16C	109.5
C7—C8—H8B	109.4	H16B—C16—H16C	109.5
C6—C1—C2—C3	0.1 (3)	C8—O2—C9—C10	171.08 (17)
C1—C2—C3—C4	0.1 (3)	O3—C9—C10—C11	170.4 (2)
C1—C2—C3—Br1	178.86 (15)	O2—C9—C10—C11	-10.3 (3)
C2—C3—C4—C5	0.1 (3)	O3—C9—C10—C15	-10.5 (3)
Br1—C3—C4—C5	-178.69 (15)	O2—C9—C10—C15	168.90 (17)
C3—C4—C5—C6	-0.4 (3)	C15—C10—C11—C12	-0.2 (3)
C4—C5—C6—C1	0.5 (3)	C9—C10—C11—C12	179.0 (2)
C4—C5—C6—C7	179.23 (18)	C10—C11—C12—C13	-0.2 (3)
C2—C1—C6—C5	-0.4 (3)	C11—C12—C13—C14	0.5 (4)
C2—C1—C6—C7	-179.03 (19)	C12—C13—C14—C15	-0.6 (4)
C5—C6—C7—O1	4.6 (3)	C16—O4—C15—C14	0.5 (3)
C1—C6—C7—O1	-176.7 (2)	C16—O4—C15—C10	-178.7 (2)
C5—C6—C7—C8	-175.25 (18)	C13—C14—C15—O4	-179.0 (2)
C1—C6—C7—C8	3.4 (3)	C13—C14—C15—C10	0.2 (3)
C9—O2—C8—C7	-76.7 (2)	C11—C10—C15—O4	179.39 (17)
O1—C7—C8—O2	4.4 (3)	C9—C10—C15—O4	0.2 (3)
C6—C7—C8—O2	-175.70 (16)	C11—C10—C15—C14	0.1 (3)
C8—O2—C9—O3	-9.5 (3)	C9—C10—C15—C14	-179.03 (18)

supplementary materials

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C2-H2A\cdots O1^i$	0.93	2.45	3.360 (3)	165

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

Fig. 1

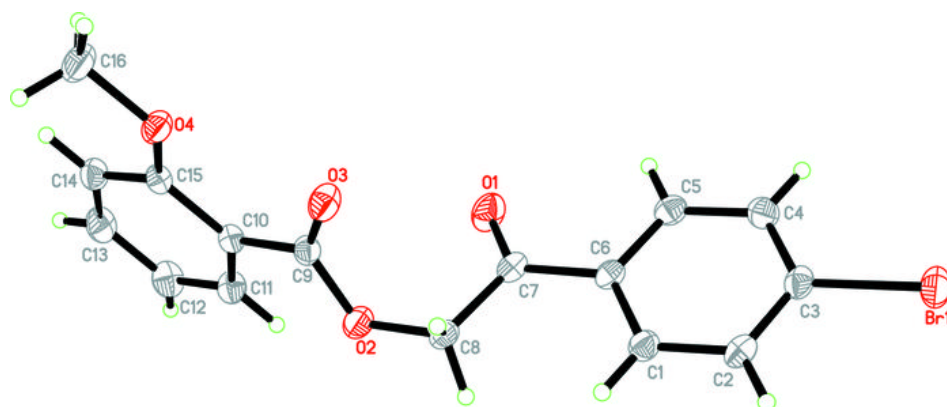


Fig. 2

