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3-(4-Bromophenyl)cyclobutanone

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The title compound, $C_{10}H_9BrO$, was produced *via* the [2 + 2]-cycloaddition reaction of 4-bromostyrene with trichloroacetyl chloride followed by dechlorination. The molecule is folded, as shown by the dihedral angle of 45.5 (7)° between the rings.

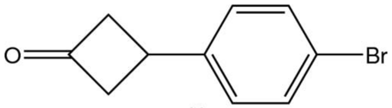
Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.012 Å
R factor = 0.060
wR factor = 0.146
Data-to-parameter ratio = 10.6

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

Comment

We recently developed 3-[4-(dihydroxyboryl)phenyl]cyclobutane as a potent BNCT agent for hepatoma treatment (Lo *et al.*, 2003, 2004) and the title compound, (I), is the precursor for its synthesis. The molecular structure of (I) (Fig. 1 and Table 1) is folded, as shown by the dihedral angle of 45.5 (7)° between the benzene and cyclobutane rings. The Br—C1 bond distance of 1.907 (6) Å is in good agreement with comparable bromophenyl compounds, in which values range from 1.900 (2)Å in 1-acetyl-3′-(4-bromophenyl)-3′-chlorospiro(3*H*-indole-3,2′-oxentan)-2(1*H*)-one (Usman *et al.*, 2002) to 1.904 (2) Å in 7-hydroxy-7-(*p*-bromophenyl)-1-azabicycloctan-2-one (Hashizume *et al.*, 1994).



(I)

A view of the unit-cell contents is shown in Fig. 2. The shortest intermolecular contact is a hydrogen bond between C7—H7 and O^i [$H7\cdots O^i$ = 2.71, $C7\cdots O^i$ = 3.520 (9) Å and $C7—H7\cdots O^i$ = 141°; symmetry code: (i) $-x, \frac{1}{2} + y, \frac{3}{2} - z$]. In addition, there is a $C2—H2\cdots\pi$ interaction of 2.96 Å involving the phenyl ring at ($\frac{1}{2} + x, \frac{1}{2} - y, 1 - z$).

Experimental

The title compound was synthesized by the following procedure (Fu, 2002; Srivastava *et al.*, 1999). All solvents were reagent grade and were distilled from suitable drying agents under a nitrogen atmosphere prior to use. All other chemicals were from Aldrich and used as received. Into a two-necked flask equipped with a reflux condenser and nitrogen-filled balloon were added 4-bromostyrene (54.6 mmol, 10.0 g) and diethyl ether (30 ml) along with Zn dust (305 mmol, 20.0 g) which was freshly cleaned by washing consecutively with NH_4Cl solution, ethanol and diethyl ether. A solution of trichloroacetyl chloride (247.1 mmol, 28.0 ml) and phosphorus oxychoride (241.5 mmol, 23.0 ml) in diethyl ether (70.0 ml) was prepared and added dropwise with a syringe. The reaction mixture was stirred for 1 h and refluxed at 353 K for 16 h. The mixture was cooled to room temperature and filtered through a pad of Celite. The organic extract was neutralized with 2 *M* ammonium chloride, washed with water and brine, and then dried over anhydrous magnesium sulfate. Finally, the

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organic solution was concentrated on a rotary evaporator to obtain the intermediate product as a light-yellow liquid. A two-necked round-bottomed flask equipped with a reflux condenser and nitrogen-filled balloon was charged with freshly cleaned zinc dust (218.4 mmol, 40.0 g) and glacial acetic acid (50 ml). The intermediate product was added dropwise. The reaction mixture was then refluxed at 393 K (oil bath) for 3 h. The mixture was cooled to room temperature, ethyl acetate (10 ml) added, and filtered through a pad of Celite. The solvent was removed under reduced pressure. The residue was dissolved in ethyl acetate and washed with water followed by brine. The organic solution was collected, dried over anhydrous magnesium sulfate, and concentrated on a rotary evaporator to obtain a light-yellow thick liquid. The crude product was purified by silica gel chromatography (2 and 5% ethyl acetate in hexane solutions) to obtain (I) as a colorless liquid (3.34 g, 33.4%). Slow evaporation of (I) dissolved in diethyl ether and frozen at 268–269 K gave crystals, R_F = 0.19 (5% ethyl acetate in hexane, thin-layer chromatography).

Crystal data

$C_{10}H_9BrO$
$M_r = 225.08$
Orthorhombic, $P2_12_12_1$
$a = 5.4751$ (9) Å
$b = 9.4755$ (14) Å
$c = 17.885$ (4) Å
$V = 927.9$ (3) Å^3

$Z = 4$
$D_x = 1.611$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 4.38$ mm^{-1}
$T = 294$ (2) K
Irregular, colorless
0.08 × 0.05 × 0.01 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{\min} = 0.694$, $T_{\max} = 0.951$

5593 measured reflections
1166 independent reflections
977 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.126$
$\theta_{\max} = 26.7°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.146$
$S = 0.89$
1166 reflections
110 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.009$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.64$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.104 (7)
Absolute structure: Flack (1983), 751 Friedel pairs
Flack parameter: 0.01 (1)

Table 1
Selected geometric parameters (Å, °).

Br—C1	1.907 (6)	C9—O	1.200 (9)
C6—C1—Br	120.8 (6)	C2—C1—Br	119.3 (6)
Br—C1—C2—C3	−179.5 (6)	Br—C1—C6—C5	−179.8 (6)

H atoms were positioned geometrically and included in the riding-model approximation, with C—H = 0.93–0.98Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

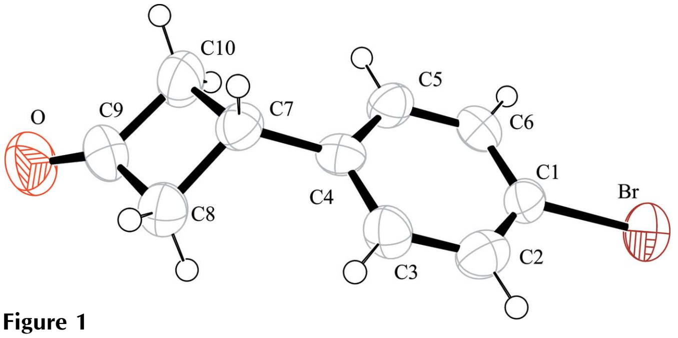

Figure 1
The molecular structure of (I), showing the atom-labeling scheme and 30% probability displacement ellipoids.

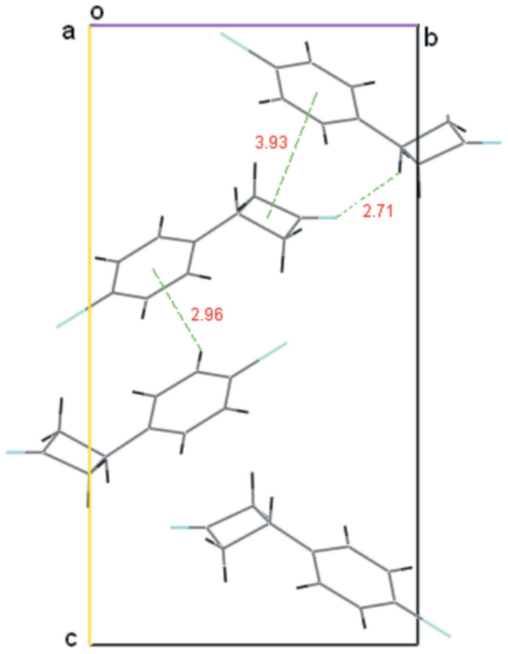

Figure 2
A view of supramolecular aggregation in (I), with dashed lines indicating C—H···O and C—H···π interactions.

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