Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-[3-(Bromomethyl)benzyloxy]-3-methoxybenzaldehyde

Jin-Jian Wei,[a] Lei Jin,[b] Cheng-He Zhou[a]* and Yi-Yi Zhang[a]

[a]Laboratory of Bioorganic & Medicinal Chemistry, School of Chemistry and Chemical Engineering, Southwest University, Chongqing 400715, People's Republic of China, and [b]School of Pharmaceutical Sciences, Southwest University, Chongqing 400715, People's Republic of China
Correspondence e-mail: zhouch@swu.edu.cn

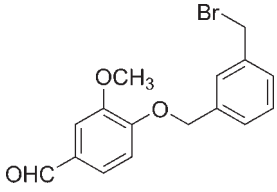
Received 21 March 2010; accepted 25 March 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.054; wR factor = 0.150; data-to-parameter ratio = 15.3.

In the title compound, $C_{16}H_{15}BrO_3$, the dihedral angle between the mean planes of the two benzene rings is 76.64 (2)°. In the crystal structure, there are weak π–π stacking interactions, with a centroid–centroid distance of 3.724 (3) Å, as well as an intermolecular C···Br distance [3.495 (2) Å] which is slightly less than the sum of the van der Waals radii for these atoms.

Related literature

For the applications of related compounds, see: Chen *et al.* (2001); Demestre *et al.* (2009); Liao *et al.* (2003); Xia & Hu (2004). For a related structure, see: Jin *et al.* (2009).



Experimental

Crystal data

$C_{16}H_{15}BrO_3$
M_r = 335.19
Monoclinic, $P2_1/c$
a = 14.275 (3) Å
b = 11.791 (2) Å
c = 8.7315 (17) Å
β = 95.671 (3)°
V = 1462.5 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.82 mm^{-1}
T = 298 K
0.08 × 0.08 × 0.06 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)
T_{min} = 0.798, T_{max} = 0.845
7566 measured reflections
2777 independent reflections
2014 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.150
S = 1.07
2777 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.85 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; 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*.

We thank Southwest University (SWUB2006018, XSGX0602 and SWUF2007023) and the Natural Science Foundation of Chongqing (2007BB5369) for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5019).

References

Bruker (2001). *SMART* and *SAINT*-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
Chen, Y.-J., Shiao, M.-S., Hsu, M.-L., Tsai, T.-H. & Wang, S.-Y. (2001). *J. Agric. Food Chem.* **49**, 5615–5619.
Demestre, M., Messerli, S. M., Celli, N., Shahhossini, M., Kluwe, L., Mautner, V. & Maruta, H. (2009). *Phytother. Res.* **23**, 226–230.
Jin, L., Wang, G.-Z. & Zhou, C.-H. (2009). *Acta Cryst.* E**65**, o2164.
Liao, H.-F., Chen, Y.-Y., Liu, J.-J., Hsu, M.-L., Shieh, H.-J., Liao, H.-J., Shieh, C.-J., Shiao, M.-S. & Chen, Y.-J. (2003). *J. Agric. Food Chem.* **51**, 7907–7912.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xia, C.-N. & Hu, W.-X. (2004). *Chin. J. Synth. Chem.* **12**, 545–550.

supporting information

Acta Cryst. (2010). E**66**, o987 [doi:10.1107/S1600536810011347]

4-[3-(Bromomethyl)benzyloxy]-3-methoxybenzaldehyde

Jin-Jian Wei, Lei Jin, Cheng-He Zhou and Yi-Yi Zhang

S1. Comment

Vanillin (4-hydroxy-3-methoxybenzaldehyde) and its derivatives are important medicinal intermediates which are extensively employed to prepare bioactive compounds such as anti-hypertension compounds, diureses and deodorisers (Chen, *et al.*, 2001; Demestre, *et al.*, 2009; Liao, *et al.*, 2003; Xia, *et al.*, 2004). Recently, our research has been focused on the development of vanillin-derived azole drugs, and a nitroimidazole derivative has been reported (Jin, *et al.*, 2009). Herein we report the crystal structure of the title compound a potential intermediate for the synthesis of azole antifungal agents.

The molecular structure of the title compound is shown in Fig. 1. The dihedral angle between the mean planes of the two benzene rings is 76.64 (2)°. In the crystal structure, there are weak π–π stacking interactions where Cg···Cg(-x,2-y,1-z) = 3.724 (3)Å [Cg is the centroid of the C2-C7 ring] as well as an intermolecular C13···Br1(-x+1, 0.5+y, 2.5-z) distance [3.495 (2)Å] which is slightly less than the sum of the van der Waals radii for these atoms.

S2. Experimental

A suspension of 4-hyhydroxy-3-methoxybenzaldehyde (200 mg, 1.31 mmol) and anhydrous potassium carbonate (200 mg, 1.45 mmol, 1.2 equiv) in CH_3CN (10 ml) was stirred for 30 min at 338 K, and then tetrabutyl ammonium iodide (TBAI, 5 mg) and 1,3-bis(bromomethyl)benzene (1 g, 3.78 mmol) were added. The resulting mixture was stirred for 5–7 h at 348–353 K (monitored by TLC, eluent, ethyl acetate/petroleum, V/V, 5/1). After the reaction solvent was evaporated under reduced pressure, water (10 mL) was added. The mixture was extracted with chloroform (3×10 ml). The organic layer was collected, dried over anhydrous Na_2SO_4 and evaporated under reduced pressure to give the crude product, which was purified by silica gel column chromatography (eluent, ethyl acetate/petroleum, V/V, 5/1) to afford the title compound (I). Single crystals were grown by slow evaporation of a solution of (I) in an ethyl acetate and petroleum mixture at room temperature.

S3. Refinement

Hydrogen atoms were placed in calculated positions with C—H = 0.93Å (aromatic), 0.97Å (methylene) and 0.96Å (methyl) with *Uiso*(H) = 1.2*Ueq*(C) (aromatic and methylene C) or 1.5*Ueq*(C) (methyl C).

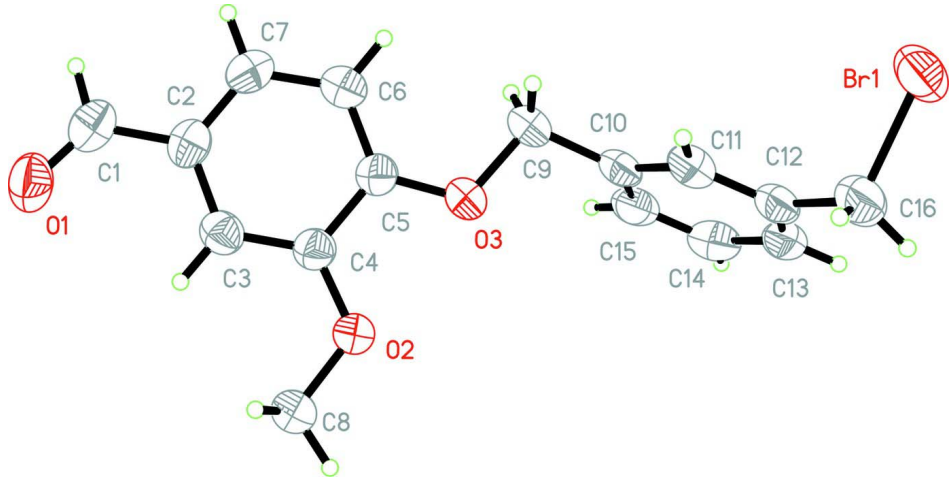


Figure 1

The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

4-[3-(Bromomethyl)benzyloxy]-3-methoxybenzaldehyde

Crystal data

$C_{16}H_{15}BrO_3$
$M_r = 335.19$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 14.275\ (3)$ Å
$b = 11.791\ (2)$ Å
$c = 8.7315\ (17)$ Å
$\beta = 95.671\ (3)°$
$V = 1462.5\ (5)$ Å^3
$Z = 4$
$F(000) = 680$
$D_x = 1.522$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2434 reflections
$\theta = 2.3–24.0°$
$\mu = 2.82$ mm^{-1}
$T = 298$ K
Block, colourless
0.08 × 0.08 × 0.06 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick,1996)
$T_{min} = 0.798$, $T_{max} = 0.845$
7566 measured reflections
2777 independent reflections
2014 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 25.7°$, $\theta_{min} = 2.2°$
$h = -17\rightarrow17$
$k = -14\rightarrow14$
$l = -10\rightarrow5$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.150$
$S = 1.07$
2777 reflections
182 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2 + 1.2302P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.85$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.58913 (3)	0.68699 (4)	1.13810 (7)	0.0709 (3)
C1	−0.0081 (3)	1.0150 (5)	0.1918 (5)	0.0559 (11)
H1	−0.0372	0.9491	0.1526	0.067*
C2	0.0624 (3)	1.0028 (4)	0.3238 (5)	0.0450 (9)
C3	0.1048 (3)	1.0973 (3)	0.3971 (5)	0.0437 (9)
H3	0.0915	1.1693	0.3572	0.052*
C4	0.1657 (3)	1.0851 (3)	0.5270 (4)	0.0409 (9)
C5	0.1854 (3)	0.9748 (3)	0.5878 (5)	0.0411 (9)
C6	0.1453 (3)	0.8818 (4)	0.5123 (5)	0.0483 (10)
H6	0.1598	0.8093	0.5493	0.058*
C7	0.0832 (3)	0.8959 (4)	0.3805 (5)	0.0492 (10)
H7	0.0556	0.8328	0.3305	0.059*
C8	0.1941 (3)	1.2830 (4)	0.5499 (6)	0.0532 (11)
H8A	0.2168	1.2873	0.4502	0.080*
H8B	0.2278	1.3363	0.6180	0.080*
H8C	0.1282	1.3007	0.5412	0.080*
C9	0.2625 (3)	0.8629 (3)	0.7868 (5)	0.0561 (12)
H9A	0.2991	0.8176	0.7216	0.067*
H9B	0.2040	0.8234	0.7983	0.067*
C10	0.3167 (3)	0.8807 (3)	0.9420 (5)	0.0446 (10)
C11	0.4120 (3)	0.8598 (3)	0.9632 (5)	0.0482 (10)
H11	0.4430	0.8359	0.8802	0.058*
C12	0.4628 (3)	0.8737 (3)	1.1059 (5)	0.0486 (10)
C13	0.4153 (4)	0.9079 (3)	1.2281 (5)	0.0576 (12)
H13	0.4481	0.9167	1.3247	0.069*
C14	0.3197 (3)	0.9293 (4)	1.2093 (6)	0.0581 (12)
H14	0.2886	0.9527	1.2925	0.070*
C15	0.2709 (3)	0.9158 (3)	1.0667 (6)	0.0530 (11)
H15	0.2065	0.9303	1.0537	0.064*
C16	0.5658 (3)	0.8494 (4)	1.1234 (7)	0.0683 (14)
H16A	0.5948	0.8865	1.2153	0.082*
H16B	0.5942	0.8798	1.0356	0.082*
O1	−0.0319 (2)	1.1038 (3)	0.1288 (4)	0.0668 (9)
O2	0.2083 (2)	1.1717 (2)	0.6096 (3)	0.0501 (7)
O3	0.2435 (2)	0.9726 (2)	0.7192 (3)	0.0516 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0595 (3)	0.0524 (3)	0.0992 (5)	0.0119 (2)	0.0002 (3)	0.0131 (3)
C1	0.056 (3)	0.067 (3)	0.045 (3)	−0.005 (2)	0.003 (2)	−0.007 (2)
C2	0.040 (2)	0.056 (2)	0.040 (2)	−0.0003 (18)	0.0050 (18)	−0.003 (2)
C3	0.047 (2)	0.041 (2)	0.044 (2)	0.0044 (17)	0.0074 (19)	0.0016 (19)
C4	0.041 (2)	0.039 (2)	0.043 (2)	0.0013 (16)	0.0025 (18)	−0.0020 (18)
C5	0.041 (2)	0.040 (2)	0.042 (2)	0.0006 (16)	0.0021 (18)	−0.0009 (18)
C6	0.053 (2)	0.038 (2)	0.053 (3)	0.0029 (18)	0.004 (2)	0.0008 (19)
C7	0.049 (2)	0.048 (2)	0.050 (2)	−0.0055 (19)	0.002 (2)	−0.010 (2)
C8	0.063 (3)	0.039 (2)	0.055 (3)	0.0037 (19)	−0.007 (2)	0.000 (2)
C9	0.071 (3)	0.033 (2)	0.061 (3)	0.005 (2)	−0.011 (2)	0.007 (2)
C10	0.052 (2)	0.0263 (18)	0.054 (3)	0.0026 (16)	0.001 (2)	0.0082 (18)
C11	0.051 (2)	0.036 (2)	0.058 (3)	0.0052 (18)	0.008 (2)	0.006 (2)
C12	0.051 (2)	0.0295 (19)	0.064 (3)	0.0006 (17)	−0.002 (2)	0.009 (2)
C13	0.083 (3)	0.034 (2)	0.052 (3)	−0.001 (2)	−0.011 (2)	0.003 (2)
C14	0.072 (3)	0.039 (2)	0.064 (3)	0.001 (2)	0.016 (3)	−0.003 (2)
C15	0.050 (2)	0.033 (2)	0.075 (3)	0.0017 (18)	0.005 (2)	0.001 (2)
C16	0.058 (3)	0.046 (3)	0.098 (4)	−0.002 (2)	−0.009 (3)	0.013 (3)
O1	0.063 (2)	0.078 (2)	0.055 (2)	0.0041 (18)	−0.0122 (16)	0.0038 (19)
O2	0.0631 (18)	0.0365 (15)	0.0474 (17)	−0.0004 (13)	−0.0112 (14)	0.0001 (12)
O3	0.0621 (18)	0.0359 (14)	0.0536 (18)	0.0014 (13)	−0.0108 (15)	0.0060 (13)

Geometric parameters (Å, º)

Br1—C16	1.946 (5)	C8—H8C	0.9600
C1—O1	1.216 (6)	C9—O3	1.437 (5)
C1—C2	1.460 (6)	C9—C10	1.508 (6)
C1—H1	0.9300	C9—H9A	0.9700
C2—C7	1.376 (6)	C9—H9B	0.9700
C2—C3	1.393 (6)	C10—C11	1.376 (6)
C3—C4	1.367 (6)	C10—C15	1.388 (6)
C3—H3	0.9300	C11—C12	1.388 (6)
C4—O2	1.358 (5)	C11—H11	0.9300
C4—C5	1.422 (5)	C12—C13	1.380 (6)
C5—O3	1.348 (5)	C12—C16	1.492 (6)
C5—C6	1.375 (6)	C13—C14	1.381 (7)
C6—C7	1.392 (6)	C13—H13	0.9300
C6—H6	0.9300	C14—C15	1.375 (7)
C7—H7	0.9300	C14—H14	0.9300
C8—O2	1.420 (5)	C15—H15	0.9300
C8—H8A	0.9600	C16—H16A	0.9700
C8—H8B	0.9600	C16—H16B	0.9700
O1—C1—C2	125.6 (4)	O3—C9—H9B	110.2
O1—C1—H1	117.2	C10—C9—H9B	110.2
C2—C1—H1	117.2	H9A—C9—H9B	108.5

C7—C2—C3	120.0 (4)	C11—C10—C15	118.9 (4)
C7—C2—C1	118.7 (4)	C11—C10—C9	120.6 (4)
C3—C2—C1	121.2 (4)	C15—C10—C9	120.4 (4)
C4—C3—C2	120.6 (4)	C10—C11—C12	121.4 (4)
C4—C3—H3	119.7	C10—C11—H11	119.3
C2—C3—H3	119.7	C12—C11—H11	119.3
O2—C4—C3	125.2 (4)	C13—C12—C11	118.4 (4)
O2—C4—C5	115.3 (3)	C13—C12—C16	122.2 (5)
C3—C4—C5	119.5 (4)	C11—C12—C16	119.4 (4)
O3—C5—C6	125.8 (4)	C12—C13—C14	121.1 (4)
O3—C5—C4	114.7 (3)	C12—C13—H13	119.4
C6—C5—C4	119.5 (4)	C14—C13—H13	119.4
C5—C6—C7	120.1 (4)	C15—C14—C13	119.6 (4)
C5—C6—H6	119.9	C15—C14—H14	120.2
C7—C6—H6	119.9	C13—C14—H14	120.2
C2—C7—C6	120.3 (4)	C14—C15—C10	120.6 (4)
C2—C7—H7	119.9	C14—C15—H15	119.7
C6—C7—H7	119.9	C10—C15—H15	119.7
O2—C8—H8A	109.5	C12—C16—Br1	110.9 (3)
O2—C8—H8B	109.5	C12—C16—H16A	109.5
H8A—C8—H8B	109.5	Br1—C16—H16A	109.5
O2—C8—H8C	109.5	C12—C16—H16B	109.5
H8A—C8—H8C	109.5	Br1—C16—H16B	109.5
H8B—C8—H8C	109.5	H16A—C16—H16B	108.1
O3—C9—C10	107.6 (3)	C4—O2—C8	117.4 (3)
O3—C9—H9A	110.2	C5—O3—C9	116.2 (3)
C10—C9—H9A	110.2		

O1—C1—C2—C7	−178.7 (4)	C15—C10—C11—C12	−0.4 (6)
O1—C1—C2—C3	4.6 (7)	C9—C10—C11—C12	−178.8 (4)
C7—C2—C3—C4	−1.3 (6)	C10—C11—C12—C13	0.8 (6)
C1—C2—C3—C4	175.4 (4)	C10—C11—C12—C16	179.7 (4)
C2—C3—C4—O2	−177.8 (4)	C11—C12—C13—C14	−0.8 (6)
C2—C3—C4—C5	−0.3 (6)	C16—C12—C13—C14	−179.7 (4)
O2—C4—C5—O3	0.2 (5)	C12—C13—C14—C15	0.3 (6)
C3—C4—C5—O3	−177.6 (3)	C13—C14—C15—C10	0.1 (6)
O2—C4—C5—C6	179.9 (4)	C11—C10—C15—C14	−0.1 (6)
C3—C4—C5—C6	2.1 (6)	C9—C10—C15—C14	178.3 (4)
O3—C5—C6—C7	177.3 (4)	C13—C12—C16—Br1	100.3 (5)
C4—C5—C6—C7	−2.5 (6)	C11—C12—C16—Br1	−78.6 (5)
C3—C2—C7—C6	1.0 (6)	C3—C4—O2—C8	−5.1 (6)
C1—C2—C7—C6	−175.8 (4)	C5—C4—O2—C8	177.3 (4)
C5—C6—C7—C2	0.9 (6)	C6—C5—O3—C9	−1.7 (6)
O3—C9—C10—C11	−104.6 (4)	C4—C5—O3—C9	178.0 (3)
O3—C9—C10—C15	77.0 (5)	C10—C9—O3—C5	−172.1 (3)