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1-(3-Bromopropoxy)-4-iodobenzene

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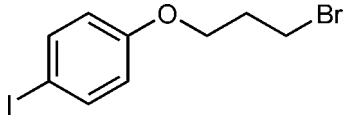
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.004 Å; R factor = 0.025; wR factor = 0.056; data-to-parameter ratio = 22.0.

In the title compound, $C_9H_{10}BrIO$, the dihedral angle between the benzene ring and its attached –O–CH_2–CH_2– group is 3.81 (19)°.

Related literature

For related literature, see: Lewis *et al.* (2004).



Experimental

Crystal data

$C_9H_{10}BrIO$
M_r = 340.98
Monoclinic, $P2_1/c$
a = 9.1604 (18) Å
b = 10.222 (2) Å
c = 10.903 (2) Å
β = 90.68 (3)°
V = 1020.9 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.00 mm^{-1}
T = 113 (2) K
0.10 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2003)
T_{min} = 0.541, T_{max} = 0.679
7486 measured reflections
2398 independent reflections
1934 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.025
$wR(F^2)$ = 0.056
S = 0.99
2398 reflections
109 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}

Data collection: *CrystalClear* (Rigaku, 2003); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2654).

References

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supplementary materials

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1-(3-Bromopropoxy)-4-iodobenzene

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Comment

The title compound, (I), is an intermediate in the synthesis of the dual-function H_1 antagonist/5-LO inhibitor compounds (Lewis *et al.*, 2004) which would provide a valuable alternative to the currently available therapies on asthma. A view of the molecular structure of (I) is shown in Fig. 1. A l l bonds lengths and angles are normal.

Experimental

To a suspension of (3-bromo-propoxy)-benzene (1.0 g, 4.6 mmol) and mercury(II) oxide (1.0 g, 4.6 mmol) in dichloromethane (30 ml) was added iodine (1.2 g, 4.6 mmol) and the resulting mixture was stirred at room temperature. After 40 h the reaction mixture was filtered to remove the insoluble material, and the filtrate was washed with $Na_2S_2O_3$ (20 wt % solution) and brine. The organic phase was then dried over anhydrous Na_2SO_4, filtered, and concentrated *in vacuo*. The pure product was obtained by column chromatography on silica gel with petroleum ether-ethyl acetate (20:1 *v*/*v*) as eluent to give the product (1.03 g, 65%). Colourless blocks of (I) were grown by slow evaporation of petroleum ether at room temperature over a period of 5 days.

Refinement

The H atoms were positioned geometrically (C—H = 0.93–0.97 Å) and refined as riding with $U_{iso} = 1.2U_{eq}$(C).

Figures

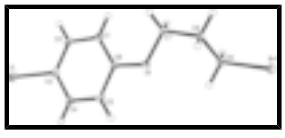

Fig. 1. A view of the molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as spheres of arbitrary radius.

1-(3-Bromopropoxy)-4-iodobenzene

Crystal data

$C_9H_{10}BrIO$	$F_{000} = 640$
$M_r = 340.98$	$D_x = 2.219$ Mg m^{-3}
Monoclinic, $P2_1/c$	Melting point: 340 K
Hall symbol: -P 2ybc	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.1604$ (18) Å	Cell parameters from 3015 reflections
$b = 10.222$ (2) Å	$\theta = 1.9$–27.9°
$c = 10.903$ (2) Å	$\mu = 7.00$ mm^{-1}

β = 90.68 (3)º	T = 113 (2) K
V = 1020.9 (4) Å^3	Block, colourless
Z = 4	0.10 × 0.08 × 0.06 mm

Data collection

Rigaku saturn diffractometer	2398 independent reflections
Radiation source: rotating anode	1934 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.040
T = 113(2) K	θ_{max} = 27.9º
ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (CrystalClear; Rigaku, 2003)	h = −12→11
T_{min} = 0.541, T_{max} = 0.679	k = −8→13
7486 measured reflections	l = −14→14

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.025	H-atom parameters constrained
$wR(F^2)$ = 0.056	$w = 1/[\sigma^2(F_o^2) + (0.0242P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.99	$(\Delta/\sigma)_{max}$ = 0.002
2398 reflections	$\Delta\rho_{max}$ = 1.46 e Å^{-3}
109 parameters	$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Primary atom site location: structure-invariant direct methods	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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
I1	0.40187 (2)	0.04322 (2)	0.787855 (18)	0.02246 (8)
Br1	1.17228 (3)	0.12604 (3)	0.04937 (3)	0.02077 (9)

O1	0.8837 (2)	0.1443 (2)	0.40015 (18)	0.0175 (4)
C1	0.6576 (3)	0.2147 (3)	0.4965 (3)	0.0180 (6)
H1	0.6523	0.2904	0.4493	0.022*
C2	0.5516 (3)	0.1885 (3)	0.5830 (3)	0.0182 (6)
H2	0.4749	0.2469	0.5937	0.022*
C3	0.5592 (3)	0.0770 (3)	0.6528 (3)	0.0169 (6)
C4	0.6714 (3)	−0.0122 (3)	0.6366 (3)	0.0181 (6)
H4	0.6750	−0.0883	0.6832	0.022*
C5	0.7780 (3)	0.0127 (3)	0.5506 (3)	0.0177 (6)
H5	0.8537	−0.0465	0.5393	0.021*
C6	0.7710 (3)	0.1273 (3)	0.4809 (3)	0.0141 (6)
C7	0.8748 (3)	0.2560 (3)	0.3197 (3)	0.0182 (6)
H7A	0.8752	0.3364	0.3670	0.022*
H7B	0.7858	0.2527	0.2708	0.022*
C8	1.0080 (3)	0.2501 (3)	0.2374 (3)	0.0165 (6)
H8A	1.0104	0.3275	0.1860	0.020*
H8B	1.0960	0.2494	0.2879	0.020*
C9	1.0049 (3)	0.1299 (3)	0.1576 (3)	0.0188 (6)
H9A	0.9156	0.1291	0.1089	0.023*
H9B	1.0056	0.0524	0.2089	0.023*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.02027 (12)	0.02375 (13)	0.02352 (13)	−0.00457 (9)	0.00680 (8)	−0.00334 (8)
Br1	0.02236 (16)	0.02035 (17)	0.01971 (17)	−0.00170 (13)	0.00538 (12)	−0.00201 (12)
O1	0.0161 (10)	0.0142 (11)	0.0224 (11)	0.0028 (9)	0.0045 (9)	0.0058 (9)
C1	0.0183 (15)	0.0124 (14)	0.0232 (15)	0.0015 (13)	0.0018 (12)	0.0035 (13)
C2	0.0141 (14)	0.0138 (15)	0.0267 (16)	0.0030 (12)	0.0016 (12)	−0.0033 (13)
C3	0.0126 (14)	0.0194 (16)	0.0187 (16)	−0.0030 (12)	0.0012 (12)	−0.0031 (12)
C4	0.0201 (15)	0.0132 (15)	0.0209 (16)	−0.0016 (13)	0.0016 (12)	0.0009 (12)
C5	0.0173 (14)	0.0141 (15)	0.0218 (16)	0.0039 (13)	−0.0001 (12)	−0.0017 (12)
C6	0.0132 (13)	0.0134 (14)	0.0157 (14)	−0.0019 (12)	0.0002 (11)	−0.0029 (12)
C7	0.0216 (15)	0.0117 (15)	0.0214 (15)	0.0041 (13)	0.0024 (12)	0.0018 (12)
C8	0.0208 (15)	0.0116 (15)	0.0171 (15)	−0.0005 (12)	0.0023 (12)	0.0015 (11)
C9	0.0175 (14)	0.0183 (16)	0.0206 (16)	−0.0040 (13)	0.0041 (12)	−0.0019 (13)

Geometric parameters (Å, °)

I1—C3	2.101 (3)	C4—H4	0.9300
Br1—C9	1.946 (3)	C5—C6	1.397 (4)
O1—C6	1.376 (3)	C5—H5	0.9300
O1—C7	1.441 (3)	C7—C8	1.524 (4)
C1—C6	1.382 (4)	C7—H7A	0.9700
C1—C2	1.388 (4)	C7—H7B	0.9700
C1—H1	0.9300	C8—C9	1.506 (4)
C2—C3	1.372 (4)	C8—H8A	0.9700
C2—H2	0.9300	C8—H8B	0.9700
C3—C4	1.387 (4)	C9—H9A	0.9700

C4—C5	1.386 (4)	C9—H9B	0.9700
C6—O1—C7	116.9 (2)	O1—C7—C8	106.7 (2)
C6—C1—C2	119.5 (3)	O1—C7—H7A	110.4
C6—C1—H1	120.3	C8—C7—H7A	110.4
C2—C1—H1	120.3	O1—C7—H7B	110.4
C3—C2—C1	120.4 (3)	C8—C7—H7B	110.4
C3—C2—H2	119.8	H7A—C7—H7B	108.6
C1—C2—H2	119.8	C9—C8—C7	111.2 (2)
C2—C3—C4	120.6 (3)	C9—C8—H8A	109.4
C2—C3—I1	119.6 (2)	C7—C8—H8A	109.4
C4—C3—I1	119.8 (2)	C9—C8—H8B	109.4
C5—C4—C3	119.6 (3)	C7—C8—H8B	109.4
C5—C4—H4	120.2	H8A—C8—H8B	108.0
C3—C4—H4	120.2	C8—C9—Br1	110.9 (2)
C4—C5—C6	119.6 (3)	C8—C9—H9A	109.5
C4—C5—H5	120.2	Br1—C9—H9A	109.5
C6—C5—H5	120.2	C8—C9—H9B	109.5
O1—C6—C1	124.6 (3)	Br1—C9—H9B	109.5
O1—C6—C5	115.1 (2)	H9A—C9—H9B	108.0
C1—C6—C5	120.3 (3)		
C6—C1—C2—C3	0.1 (4)	C2—C1—C6—O1	−178.5 (3)
C1—C2—C3—C4	−1.1 (4)	C2—C1—C6—C5	0.8 (4)
C1—C2—C3—I1	177.6 (2)	C4—C5—C6—O1	178.6 (3)
C2—C3—C4—C5	1.1 (4)	C4—C5—C6—C1	−0.8 (4)
I1—C3—C4—C5	−177.6 (2)	C6—O1—C7—C8	−178.0 (2)
C3—C4—C5—C6	−0.2 (4)	O1—C7—C8—C9	63.5 (3)
C7—O1—C6—C1	−5.4 (4)	C7—C8—C9—Br1	178.25 (18)
C7—O1—C6—C5	175.3 (2)		

Fig. 1