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1-Benzyloxy-4-nitrobenzene

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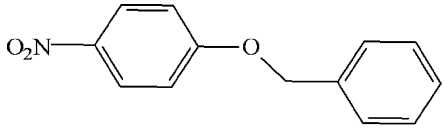
Key indicators: single-crystal X-ray study; T = 120 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.114; data-to-parameter ratio = 14.4.

The title compound, $C_{13}H_{11}NO_3$, was synthesized from 4-nitrophenol and benzyl bromide. The crystal structure is stabilized by C—H$\cdots\pi$ interactions involving two H atoms of the benzyloxy group [3.432 (2) Å and 148°, and 3.555 (2) Å and 148°].

Related literature

For related literature, see: Baker *et al.* (2000); Bridges (2000, 2003); Evans *et al.* (2002); Sharma *et al.* (2004).



Experimental

Crystal data

$C_{13}H_{11}NO_3$
M_r = 229.23
Monoclinic, $P2_1/c$
a = 14.0913 (18) Å
b = 10.7640 (14) Å
c = 7.6056 (10) Å
β = 100.349 (2)°
V = 1134.8 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 120 K
0.27 × 0.10 × 0.07 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
6209 measured reflections
2221 independent reflections
1747 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.114
S = 1.02
2221 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

Cg is the centroid of the benzyl ring.

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C8—H8$A\cdots Cg^{i}$	0.95	2.57	3.4323 (17)	148
C11—H11$\cdots Cg^{ii}$	0.95	2.76	3.5547 (17)	143

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LW2030).

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supplementary materials

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1-Benzyloxy-4-nitrobenzene

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Comment

Benzyl ethers and their derivatives are used as protecting group (Sharma *et al.*, 2004) for alcohols and phenols in the synthesis of natural products (Evans *et al.*, 2002). The title compound, a benzylether derivative, is an important intermediate in the synthesis of biologically active compounds such as irreversible inhibitors of tyrosine kinases (James, 2003). Similarly several nitro compounds are known to show anti-bacterial and other biological activity (Baker *et al.*, 2000). During the course of synthesis of some biologically active compounds, we were interested in making use of 1-benzyloxy-4-nitrobenzene as an important intermediate.

There are at least two literature report on the preparation of the title compound (Bridges, 2003). However these methods involve use of expensive starting materials such as 1-fluoro-4-nitrobenzene and benzyl alcohol. We have developed a new and simple method for the synthesis of the title compound using readily available starting material 4-nitrophenol and benzylbromide. The title compound was obtained in very high yield compared to previously reported methods. The compound was recrystallized from ethyl alcohol. The title compound is shown in Fig 1.

The weak C—H···π interactions involving the benzyloxy group of hydrogen atoms of H8A & H11 [C8—H8A···Bz = 3.4327 (17) Å; C11—H11···Bz = 3.5547 (17)Å and 148° & 143°] stabilize the crystal structure.

Experimental

4-nitrophenol (2.00 g, 0.01438 mol) was treated with a base K_2CO_3 (2.38 g, 0.01725 mol) in the presence of solvent DMF (15 ml) for 1 h at 70°C. The reaction mixture was cooled to room temperature and benzyl bromide (2.70 g, 0.015 mol) was added slowly and stirred at room temperature for 45 min. The reaction mixture was poured in to water (50 ml) and extracted with ethyl acetate (3 *x* 20 ml). Combined organic layer was washed with water (3 *x* 10 ml) dried with anhydrous Na_2SO_4 and concentrated in a rotavap to get solid (3.10 g) in 95% yield. The compound was recrystallized from ethyl alcohol.

Refinement

All H atoms were positioned geometrically and were refined using a riding model. C—H distance is 0.93–0.97%/A with U_{iso}(H)=1.5Ueq.

Figures

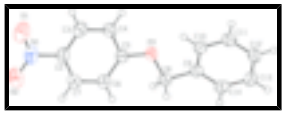

Fig. 1. *ORTEP* diagram of the asymmetric unit of (I) showing 50% probability ellipsoids

1-benzyloxy-4-nitrobenzene

Crystal data

$C_{13}H_{11}NO_3$	$F_{000} = 480$
$M_r = 229.23$	$D_x = 1.342$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 25 reflections
$a = 14.0913$ (18) Å	$\theta = 1.5$–26.0º
$b = 10.7640$ (14) Å	$\mu = 0.10$ mm^{-1}
$c = 7.6056$ (10) Å	$T = 120$ K
$\beta = 100.349$ (2)º	Sheet, colorless
$V = 1134.8$ (3) Å^3	0.27 × 0.10 × 0.07 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	1747 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.021$
Monochromator: graphite	$\theta_{max} = 26.0$º
$T = 120$ K	$\theta_{min} = 1.5$º
φ & ω scans	$h = -17\rightarrow17$
Absorption correction: none	$k = -13\rightarrow12$
6209 measured reflections	$l = -7\rightarrow9$
2221 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0486P)^2 + 0.2657P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.02$	$(\Delta/\sigma)_{max} = 0.001$
2221 reflections	$\Delta\rho_{max} = 0.15$ e Å^{-3}
154 parameters	$\Delta\rho_{min} = -0.17$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All e.s.d.'s are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating –*R*-factor-obs *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}
O1	−0.04574 (11)	0.20677 (18)	0.4021 (3)	0.1064 (8)
O2	−0.05990 (11)	0.02631 (18)	0.2800 (3)	0.1123 (8)
O3	0.37607 (8)	0.13805 (10)	0.28478 (15)	0.0479 (4)
N1	−0.01246 (12)	0.11769 (19)	0.3344 (3)	0.0773 (8)
C2	0.08975 (12)	0.12064 (18)	0.3181 (3)	0.0544 (6)
C3	0.14322 (13)	0.22473 (17)	0.3745 (3)	0.0556 (6)
C4	0.23923 (12)	0.22642 (16)	0.3611 (2)	0.0502 (6)
C5	0.28081 (11)	0.12556 (14)	0.2906 (2)	0.0403 (5)
C6	0.22564 (12)	0.02160 (16)	0.2337 (2)	0.0507 (6)
C7	0.12917 (13)	0.01972 (17)	0.2477 (3)	0.0580 (7)
C8	0.42374 (11)	0.03767 (15)	0.2114 (2)	0.0464 (5)
C9	0.52496 (11)	0.07715 (14)	0.2045 (2)	0.0396 (5)
C10	0.54179 (11)	0.18143 (14)	0.1078 (2)	0.0439 (5)
C11	0.63458 (12)	0.21673 (16)	0.0975 (2)	0.0491 (6)
C12	0.71203 (12)	0.14829 (17)	0.1832 (2)	0.0522 (6)
C13	0.69640 (12)	0.04353 (17)	0.2779 (2)	0.0516 (6)
C14	0.60320 (12)	0.00838 (15)	0.2882 (2)	0.0469 (6)
H3	0.11500	0.29260	0.42080	0.0670*
H4	0.27660	0.29580	0.39960	0.0600*
H6	0.25330	−0.04630	0.18640	0.0610*
H7	0.09130	−0.04930	0.20970	0.0700*
H8A	0.38980	0.01780	0.09210	0.0560*
H8B	0.42430	−0.03570	0.28550	0.0560*
H10	0.49000	0.22800	0.04950	0.0530*
H11	0.64510	0.28700	0.03250	0.0590*
H12	0.77460	0.17280	0.17690	0.0630*
H13	0.74840	−0.00340	0.33470	0.0620*
H14	0.59290	−0.06240	0.35230	0.0560*

Atomic displacement parameters (Å^2)

U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}

O1	0.0582 (10)	0.1091 (14)	0.1601 (18)	0.0135 (9)	0.0413 (10)	−0.0171 (12)
O2	0.0469 (9)	0.1010 (13)	0.190 (2)	−0.0160 (9)	0.0236 (11)	−0.0177 (13)
O3	0.0399 (6)	0.0478 (7)	0.0588 (7)	−0.0044 (5)	0.0161 (5)	−0.0123 (5)
N1	0.0436 (10)	0.0806 (13)	0.1084 (16)	0.0035 (9)	0.0152 (10)	0.0036 (11)
C2	0.0371 (9)	0.0631 (11)	0.0637 (12)	0.0014 (8)	0.0106 (8)	0.0066 (9)
C3	0.0493 (10)	0.0578 (11)	0.0622 (12)	0.0061 (9)	0.0167 (8)	−0.0050 (9)
C4	0.0482 (10)	0.0492 (10)	0.0547 (10)	−0.0043 (8)	0.0131 (8)	−0.0076 (8)
C5	0.0380 (8)	0.0446 (9)	0.0385 (9)	−0.0012 (7)	0.0075 (7)	0.0013 (7)
C6	0.0457 (10)	0.0447 (9)	0.0622 (11)	−0.0006 (7)	0.0111 (8)	−0.0044 (8)
C7	0.0441 (10)	0.0522 (11)	0.0765 (13)	−0.0085 (8)	0.0075 (9)	−0.0009 (9)
C8	0.0436 (9)	0.0414 (9)	0.0558 (10)	−0.0004 (7)	0.0131 (8)	−0.0070 (7)
C9	0.0407 (8)	0.0396 (8)	0.0390 (8)	0.0005 (7)	0.0088 (7)	−0.0056 (7)
C10	0.0441 (9)	0.0400 (8)	0.0475 (9)	0.0062 (7)	0.0077 (7)	0.0000 (7)
C11	0.0549 (10)	0.0416 (9)	0.0539 (10)	−0.0047 (8)	0.0180 (8)	−0.0002 (8)
C12	0.0400 (9)	0.0588 (11)	0.0595 (11)	−0.0055 (8)	0.0139 (8)	−0.0128 (9)
C13	0.0423 (9)	0.0562 (10)	0.0537 (11)	0.0100 (8)	0.0013 (8)	−0.0053 (8)
C14	0.0523 (10)	0.0424 (9)	0.0460 (10)	0.0039 (7)	0.0086 (8)	0.0033 (7)

Geometric parameters (Å, °)

O1—N1	1.221 (3)	C11—C12	1.380 (2)
O2—N1	1.219 (3)	C12—C13	1.377 (2)
O3—C5	1.358 (2)	C13—C14	1.382 (2)
O3—C8	1.4370 (19)	C3—H3	0.9307
N1—C2	1.468 (2)	C4—H4	0.9302
C2—C3	1.375 (3)	C6—H6	0.9305
C2—C7	1.372 (3)	C7—H7	0.9300
C3—C4	1.375 (3)	C8—H8A	0.9705
C4—C5	1.386 (2)	C8—H8B	0.9694
C5—C6	1.387 (2)	C10—H10	0.9296
C6—C7	1.383 (3)	C11—H11	0.9300
C8—C9	1.498 (2)	C12—H12	0.9298
C9—C10	1.386 (2)	C13—H13	0.9300
C9—C14	1.384 (2)	C14—H14	0.9298
C10—C11	1.377 (2)		
O1···H3	2.4266	H3···O1	2.4266
O1···H12^{i}	2.8140	H4···C13^{vii}	3.0434
O1···H7^{ii}	2.7988	H4···C5^{iv}	3.0822
O2···H7	2.4284	H6···C8	2.5411
O2···H12^{i}	2.8077	H6···H8A	2.2772
O2···H13^{i}	2.8242	H6···H8B	2.3947
O3···H10	2.7837	H7···O2	2.4284
O3···H14^{iii}	2.8354	H7···O1^{ix}	2.7988
O3···H10^{iv}	2.7475	H8A···C6	2.7217
C4···C5^{iv}	3.587 (2)	H8A···H6	2.2772
C5···C4^{v}	3.587 (2)	H8A···C9^{vi}	2.9240
C8···C10^{vi}	3.481 (2)	H8A···C10^{vi}	2.8924

C8···C9^{vi}	3.588 (2)	H8A···C11^{vi}	2.8970
C9···C9^{vi}	3.485 (2)	H8A···C12^{vi}	2.9257
C9···C8^{vi}	3.588 (2)	H8A···C13^{vi}	2.9329
C10···C8^{vi}	3.481 (2)	H8A···C14^{vi}	2.9254
C5···H4^{v}	3.0822	H8B···C6	2.8242
C5···H14^{iii}	3.0445	H8B···H6	2.3947
C6···H8B	2.8242	H8B···H14	2.3561
C6···H8A	2.7217	H8B···C11^{viii}	2.9744
C8···H6	2.5411	H10···O3	2.7837
C9···H8A^{vi}	2.9240	H10···O3^{v}	2.7475
C10···H8A^{vi}	2.8924	H11···C12^{v}	3.0549
C11···H8B^{vii}	2.9744	H11···C13^{v}	2.8465
C11···H8A^{vi}	2.8970	H11···C14^{v}	2.8737
C12···H8A^{vi}	2.9257	H12···O1^{x}	2.8140
C12···H11^{iv}	3.0549	H12···O2^{x}	2.8077
C13···H4^{viii}	3.0434	H13···O2^{x}	2.8242
C13···H8A^{vi}	2.9329	H14···H8B	2.3561
C13···H11^{iv}	2.8465	H14···O3^{iii}	2.8354
C14···H8A^{vi}	2.9254	H14···C5^{iii}	3.0445
C14···H11^{iv}	2.8737		
C5—O3—C8	118.17 (12)	C2—C3—H3	120.68
O1—N1—O2	123.23 (18)	C4—C3—H3	120.71
O1—N1—C2	118.40 (18)	C3—C4—H4	119.75
O2—N1—C2	118.38 (19)	C5—C4—H4	119.72
N1—C2—C3	118.83 (18)	C5—C6—H6	120.28
N1—C2—C7	119.21 (17)	C7—C6—H6	120.28
C3—C2—C7	121.96 (17)	C2—C7—H7	120.26
C2—C3—C4	118.62 (17)	C6—C7—H7	120.33
C3—C4—C5	120.54 (16)	O3—C8—H8A	110.04
O3—C5—C4	115.09 (14)	O3—C8—H8B	110.02
O3—C5—C6	124.88 (14)	C9—C8—H8A	110.01
C4—C5—C6	120.03 (15)	C9—C8—H8B	110.06
C5—C6—C7	119.45 (16)	H8A—C8—H8B	108.37
C2—C7—C6	119.41 (17)	C9—C10—H10	119.70
O3—C8—C9	108.34 (12)	C11—C10—H10	119.74
C8—C9—C10	120.21 (14)	C10—C11—H11	119.88
C8—C9—C14	121.12 (14)	C12—C11—H11	119.83
C10—C9—C14	118.63 (15)	C11—C12—H12	120.08
C9—C10—C11	120.56 (15)	C13—C12—H12	120.11
C10—C11—C12	120.29 (15)	C12—C13—H13	120.07
C11—C12—C13	119.81 (16)	C14—C13—H13	120.15
C12—C13—C14	119.78 (16)	C9—C14—H14	119.52
C9—C14—C13	120.92 (15)	C13—C14—H14	119.56
C8—O3—C5—C4	−179.30 (13)	C4—C5—C6—C7	0.1 (2)
C8—O3—C5—C6	1.2 (2)	O3—C5—C6—C7	179.55 (16)

C5—O3—C8—C9	175.12 (12)	C5—C6—C7—C2	−0.2 (3)
O2—N1—C2—C3	178.2 (2)	O3—C8—C9—C10	−58.81 (18)
O1—N1—C2—C3	−1.9 (3)	O3—C8—C9—C14	123.63 (15)
O2—N1—C2—C7	−2.0 (3)	C8—C9—C10—C11	−178.53 (14)
O1—N1—C2—C7	177.9 (2)	C10—C9—C14—C13	0.8 (2)
C7—C2—C3—C4	−0.7 (3)	C14—C9—C10—C11	−0.9 (2)
N1—C2—C3—C4	179.17 (19)	C8—C9—C14—C13	178.44 (14)
N1—C2—C7—C6	−179.37 (19)	C9—C10—C11—C12	0.2 (2)
C3—C2—C7—C6	0.5 (3)	C10—C11—C12—C13	0.7 (2)
C2—C3—C4—C5	0.6 (3)	C11—C12—C13—C14	−0.7 (2)
C3—C4—C5—C6	−0.3 (2)	C12—C13—C14—C9	0.0 (2)
C3—C4—C5—O3	−179.81 (16)		

Symmetry codes: (i) *x*−1, *y*, *z*; (ii) −*x*, *y*+1/2, −*z*+1/2; (iii) −*x*+1, −*y*, −*z*+1; (iv) *x*, −*y*+1/2, *z*+1/2; (v) *x*, −*y*+1/2, *z*−1/2; (vi) −*x*+1, −*y*, −*z*; (vii) −*x*+1, *y*+1/2, −*z*+1/2; (viii) −*x*+1, *y*−1/2, −*z*+1/2; (ix) −*x*, *y*−1/2, −*z*+1/2; (x) *x*+1, *y*, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C8—H8A···Cg2^{vi}	0.95	2.57	3.4323 (17)	148
C11—H11···Cg2^{v}	0.95	2.76	3.5547 (17)	143

Symmetry codes: (vi) −*x*+1, −*y*, −*z*; (v) *x*, −*y*+1/2, *z*−1/2.

Fig. 1