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# 1-(2-Methoxyethoxy)-4-nitrobenzene

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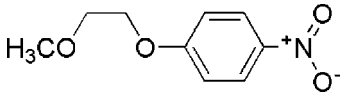
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.034; $wR$ factor = 0.088; data-to-parameter ratio = 13.5.

The title compound, $C_9H_{11}NO_4$, is an intermediate for dyes and drugs. The O—C—C—O chain adopts a synclinal conformation. The crystal structure is stabilized by C—H···O hydrogen bonds.

## Related literature

For related literature, see: Guo *et al.* (2006); Higson (1992).



## Experimental

*Crystal data*

$C_9H_{11}NO_4$
$M_r$ = 197.19
Orthorhombic, $Pna2_1$
$a$ = 11.280 (3) Å
$b$ = 20.430 (5) Å
$c$ = 4.1079 (10) Å
$V$ = 946.7 (4) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.11 mm$^{-1}$
$T$ = 100 (2) K
0.49 × 0.43 × 0.38 mm

*Data collection*

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min}$ = 0.948, $T_{max}$ = 0.959
4034 measured reflections
1023 independent reflections
982 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.030

*Refinement*

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.079
$S$ = 1.05
1023 reflections
130 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.12 e Å$^{-3}$
Absolute structure: Flack (1983)
Flack parameter: 2 (1)

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C5—H5···O1$^{i}$ | 0.93 | 2.65 | 3.419 (3) | 140 |
| C3—H3···O3$^{ii}$ | 0.93 | 2.50 | 3.317 (2) | 147 |

Symmetry codes: (i) $x+\frac{1}{2}, -y+\frac{3}{2}, z+1$; (ii) $-x, -y+1, z-\frac{1}{2}$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2633).

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# supplementary materials

*Acta Cryst.* (2008). E**64**, o12 [ **doi:10.1107/S1600536807061636** ]

# 1-(2-Methoxyethoxy)-4-nitrobenzene

**Y. Liu, W.-N. Xu, X.-L. Zhang, J.-P. Ma and D.-S. Guo**

## Comment

Nitroaromatic compounds are widely used as pesticides, explosives, and precursors for dyes and many pharmaceutical agents (Higson, 1992). Recently, we described the structure of a nitrobenzene derivative containing a polyether linkage which can be used as an asymmetric alkylating agent (Guo *et al.*, 2006). Herein, we report the structure of another nitrobenzene derivative, in which an asymmetric ethylene glycol ether strand is appended to the *para* position of nitro group.

The title compound consists of an ethylene glycol monomethyl ether unit and a nitro-substituted benzene ring (Fig. 1). In the crystal structure the nitro group is coplanar with the benzene ring. Interestingly, there are two intermolecular hydrogen bonds (Table 1).

## Experimental

To a mixture of 2-methoxyethanol (0.190 ml, 2.40 mmol) and potassium hydroxide (0.120 g, 3.00 mmol) in DMSO (5 ml), was added a solution of 1-chloro-4-nitrobenzene (0.315 g, 2.00 mmol) in DMSO (5 ml). The resulting mixture was stirred for 20 h at 333 K and cooled to room temperature. The reaction mixture was poured into HCl 5% solution. The precipitate was filtered off and washed with water. After drying in vacuum, the title compound was obtained as a yellow solid in 90% yield. Single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a solution of anhydrous ethanol at 273 K.

## Refinement

In the absence of anomalous scatterers Friedel pairs had been merged and the absolute configuration was arbitrarily assigned. All H atoms were included in calculated positions refined as riding model with $C_{methyl}$—H = 0.96 Å, $C_{methylene}$—H = 0.97Å and $C_{aromatic}$—H = 0.93Å and $U_{iso}(H) = 1.2U_{eq}(C)$ or $U_{iso}(H) = 1.5U_{eq}(C_{methyl})$.

## Figures

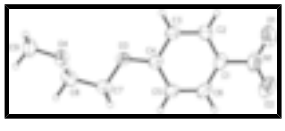

Fig. 1. The molecular structure of the title compound. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

## 1-(2-Methoxyethoxy)-4-nitrobenzene

*Crystal data*

| | |
|---|---|
| $C_9H_{11}NO_4$ | $D_x$ = 1.384 Mg m$^{-3}$ |
| $M_r$ = 197.19 | Mo $K\alpha$ radiation |

| | |
|---|---|
| | λ = 0.71073 Å |
| Orthorhombic, $Pna2_1$ | Cell parameters from 2528 reflections |
| $a$ = 11.280 (3) Å | θ = 2.7–27.8º |
| $b$ = 20.430 (5) Å | μ = 0.11 $mm^{-1}$ |
| $c$ = 4.1079 (10) Å | $T$ = 100 (2) K |
| $V$ = 946.7 (4) $Å^3$ | Block, colourless |
| $Z$ = 4 | 0.49 × 0.43 × 0.38 mm |
| $F_{000}$ = 416 | |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 1023 independent reflections |
| Radiation source: fine-focus sealed tube | 982 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.030 |
| $T$ = 100(2) K | $\theta_{max}$ = 25.7º |
| phi and ω scans | $\theta_{min}$ = 2.0º |
| Absorption correction: multi-scan (SADABS; Bruker, 1999) | $h = -12 \rightarrow 13$ |
| $T_{min}$ = 0.948, $T_{max}$ = 0.959 | $k = -15 \rightarrow 24$ |
| 4034 measured reflections | $l = -4 \rightarrow 4$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)]$ = 0.031 | $w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.1764P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2)$ = 0.079 | $(\Delta/\sigma)_{max}$ = 0.001 |
| $S$ = 1.05 | $\Delta\rho_{max}$ = 0.13 e $Å^{-3}$ |
| 1023 reflections | $\Delta\rho_{min}$ = −0.12 e $Å^{-3}$ |
| 130 parameters | Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| 1 restraint | Extinction coefficient: 0.032 (4) |
| Primary atom site location: structure-invariant direct methods | Absolute structure: Flack (1983) |
| Secondary atom site location: difference Fourier map | Flack parameter: 2 (1) |

*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 calculat-

ing *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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| C1 | −0.05088 (16) | 0.68859 (9) | −0.1216 (7) | 0.0407 (5) |
| C2 | −0.08278 (16) | 0.62314 (9) | −0.1160 (6) | 0.0373 (5) |
| H2 | −0.1522 | 0.6090 | −0.2160 | 0.045* |
| C3 | −0.00976 (16) | 0.57971 (9) | 0.0401 (6) | 0.0341 (5) |
| H3 | −0.0303 | 0.5357 | 0.0490 | 0.041* |
| C4 | 0.09501 (15) | 0.60085 (8) | 0.1858 (6) | 0.0310 (5) |
| C5 | 0.12605 (16) | 0.66669 (9) | 0.1780 (6) | 0.0382 (5) |
| H5 | 0.1956 | 0.6811 | 0.2762 | 0.046* |
| C6 | 0.05189 (18) | 0.71044 (9) | 0.0221 (7) | 0.0446 (6) |
| H6 | 0.0715 | 0.7546 | 0.0143 | 0.054* |
| C7 | 0.27095 (16) | 0.57084 (10) | 0.4767 (6) | 0.0363 (5) |
| H7A | 0.2588 | 0.6054 | 0.6354 | 0.044* |
| H7B | 0.3259 | 0.5866 | 0.3127 | 0.044* |
| C8 | 0.31983 (17) | 0.51107 (10) | 0.6395 (6) | 0.0411 (5) |
| H8A | 0.3888 | 0.5228 | 0.7677 | 0.049* |
| H8B | 0.2608 | 0.4928 | 0.7851 | 0.049* |
| C9 | 0.3945 (2) | 0.40593 (11) | 0.5514 (9) | 0.0536 (7) |
| H9A | 0.4592 | 0.4164 | 0.6939 | 0.080* |
| H9B | 0.4212 | 0.3762 | 0.3858 | 0.080* |
| H9C | 0.3319 | 0.3858 | 0.6744 | 0.080* |
| N1 | −0.12710 (18) | 0.73499 (9) | −0.2931 (7) | 0.0580 (6) |
| O1 | −0.21750 (15) | 0.71456 (9) | −0.4228 (6) | 0.0687 (6) |
| O2 | −0.09783 (16) | 0.79250 (8) | −0.3004 (9) | 0.0948 (10) |
| O3 | 0.16033 (10) | 0.55315 (6) | 0.3289 (4) | 0.0354 (4) |
| O4 | 0.35171 (11) | 0.46405 (7) | 0.4039 (4) | 0.0414 (4) |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| C1 | 0.0379 (10) | 0.0363 (10) | 0.0479 (14) | 0.0084 (8) | 0.0100 (11) | 0.0041 (11) |
| C2 | 0.0314 (8) | 0.0389 (10) | 0.0418 (12) | 0.0007 (7) | 0.0028 (10) | −0.0049 (11) |
| C3 | 0.0333 (9) | 0.0295 (8) | 0.0396 (11) | −0.0048 (7) | 0.0042 (9) | −0.0033 (10) |
| C4 | 0.0296 (9) | 0.0298 (9) | 0.0336 (11) | −0.0002 (7) | 0.0077 (8) | −0.0041 (9) |
| C5 | 0.0335 (9) | 0.0320 (9) | 0.0490 (14) | −0.0071 (7) | 0.0042 (10) | −0.0042 (11) |
| C6 | 0.0444 (11) | 0.0268 (9) | 0.0626 (16) | −0.0005 (8) | 0.0092 (12) | 0.0013 (11) |
| C7 | 0.0317 (9) | 0.0415 (10) | 0.0357 (13) | −0.0061 (8) | 0.0023 (9) | −0.0074 (9) |
| C8 | 0.0382 (10) | 0.0512 (12) | 0.0338 (12) | −0.0022 (9) | −0.0032 (10) | −0.0010 (11) |
| C9 | 0.0465 (11) | 0.0506 (12) | 0.0637 (17) | 0.0076 (9) | −0.0042 (13) | 0.0163 (14) |
| N1 | 0.0531 (11) | 0.0456 (11) | 0.0752 (18) | 0.0149 (9) | 0.0053 (12) | 0.0099 (13) |
| O1 | 0.0553 (10) | 0.0677 (11) | 0.0830 (15) | 0.0171 (8) | −0.0121 (12) | 0.0117 (13) |
| O2 | 0.0856 (13) | 0.0423 (9) | 0.157 (3) | 0.0116 (8) | −0.0187 (18) | 0.0259 (15) |
| O3 | 0.0298 (6) | 0.0289 (6) | 0.0474 (10) | −0.0041 (5) | −0.0013 (7) | 0.0000 (7) |

| | | | | | | |
|---|---|---|---|---|---|---|
| O4 | 0.0450 (7) | 0.0415 (7) | 0.0376 (8) | 0.0067 (6) | −0.0007 (8) | 0.0043 (7) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.375 (3) | C7—C8 | 1.497 (3) |
| C1—C2 | 1.385 (3) | C7—H7A | 0.9700 |
| C1—N1 | 1.461 (3) | C7—H7B | 0.9700 |
| C2—C3 | 1.370 (3) | C8—O4 | 1.410 (3) |
| C2—H2 | 0.9300 | C8—H8A | 0.9700 |
| C3—C4 | 1.393 (3) | C8—H8B | 0.9700 |
| C3—H3 | 0.9300 | C9—O4 | 1.418 (3) |
| C4—O3 | 1.356 (2) | C9—H9A | 0.9600 |
| C4—C5 | 1.390 (2) | C9—H9B | 0.9600 |
| C5—C6 | 1.382 (3) | C9—H9C | 0.9600 |
| C5—H5 | 0.9300 | N1—O2 | 1.221 (2) |
| C6—H6 | 0.9300 | N1—O1 | 1.224 (3) |
| C7—O3 | 1.434 (2) | | |
| C6—C1—C2 | 121.69 (19) | O3—C7—H7B | 110.2 |
| C6—C1—N1 | 119.50 (18) | C8—C7—H7B | 110.2 |
| C2—C1—N1 | 118.8 (2) | H7A—C7—H7B | 108.5 |
| C3—C2—C1 | 118.48 (19) | O4—C8—C7 | 110.1 (2) |
| C3—C2—H2 | 120.8 | O4—C8—H8A | 109.6 |
| C1—C2—H2 | 120.8 | C7—C8—H8A | 109.6 |
| C2—C3—C4 | 120.68 (17) | O4—C8—H8B | 109.6 |
| C2—C3—H3 | 119.7 | C7—C8—H8B | 109.6 |
| C4—C3—H3 | 119.7 | H8A—C8—H8B | 108.2 |
| O3—C4—C5 | 124.65 (18) | O4—C9—H9A | 109.5 |
| O3—C4—C3 | 115.11 (15) | O4—C9—H9B | 109.5 |
| C5—C4—C3 | 120.25 (19) | H9A—C9—H9B | 109.5 |
| C6—C5—C4 | 118.95 (19) | O4—C9—H9C | 109.5 |
| C6—C5—H5 | 120.5 | H9A—C9—H9C | 109.5 |
| C4—C5—H5 | 120.5 | H9B—C9—H9C | 109.5 |
| C1—C6—C5 | 119.96 (18) | O2—N1—O1 | 122.9 (2) |
| C1—C6—H6 | 120.0 | O2—N1—C1 | 118.5 (2) |
| C5—C6—H6 | 120.0 | O1—N1—C1 | 118.61 (18) |
| O3—C7—C8 | 107.69 (15) | C4—O3—C7 | 118.38 (14) |
| O3—C7—H7A | 110.2 | C8—O4—C9 | 111.3 (2) |
| C8—C7—H7A | 110.2 | | |
| C6—C1—C2—C3 | 0.6 (4) | O3—C7—C8—O4 | 67.3 (2) |
| N1—C1—C2—C3 | 178.8 (2) | C6—C1—N1—O2 | −0.9 (4) |
| C1—C2—C3—C4 | −0.9 (3) | C2—C1—N1—O2 | −179.2 (3) |
| C2—C3—C4—O3 | −179.2 (2) | C6—C1—N1—O1 | 179.2 (3) |
| C2—C3—C4—C5 | 0.8 (3) | C2—C1—N1—O1 | 0.9 (4) |
| O3—C4—C5—C6 | 179.6 (2) | C5—C4—O3—C7 | −1.6 (3) |
| C3—C4—C5—C6 | −0.4 (3) | C3—C4—O3—C7 | 178.42 (19) |
| C2—C1—C6—C5 | −0.2 (4) | C8—C7—O3—C4 | 175.33 (18) |
| N1—C1—C6—C5 | −178.4 (2) | C7—C8—O4—C9 | −177.94 (16) |
| C4—C5—C6—C1 | 0.1 (4) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C5—H5···O1$^{i}$ | 0.93 | 2.65 | 3.419 (3) | 140 |
| C3—H3···O3$^{ii}$ | 0.93 | 2.50 | 3.317 (2) | 147 |

Symmetry codes: (i) *x*+1/2, −*y*+3/2, *z*+1; (ii) −*x*, −*y*+1, *z*−1/2.

**Fig. 1**