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1-(2-Methoxyethoxy)-4-nitrobenzene

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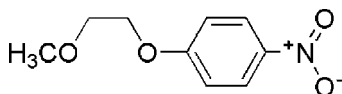
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.034; wR factor = 0.088; data-to-parameter ratio = 13.5.

The title compound, $\text{C}_9\text{H}_{11}\text{NO}_4$, is an intermediate for dyes and drugs. The $\text{O}-\text{C}-\text{C}-\text{O}$ chain adopts a synclinal conformation. The crystal structure is stabilized by $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

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

Experimental

Crystal data

 $\text{C}_9\text{H}_{11}\text{NO}_4$ $M_r = 197.19$ Orthorhombic, $Pna2_1$ $a = 11.280$ (3) Å $b = 20.430$ (5) Å $c = 4.1079$ (10) Å $V = 946.7$ (4) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.11$ mm⁻¹ $T = 100$ (2) K $0.49 \times 0.43 \times 0.38$ 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 $I > 2\sigma(I)$ $R_{\text{int}} = 0.030$

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

 $\Delta\rho_{\text{max}} = 0.13$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.12$ e Å⁻³

Absolute structure: Flack (1983)

Flack parameter: 2 (1)

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--|-------|-------------|-------------|---------------|
| $\text{C5}-\text{H5}\cdots\text{O1}^{\text{i}}$ | 0.93 | 2.65 | 3.419 (3) | 140 |
| $\text{C3}-\text{H3}\cdots\text{O3}^{\text{ii}}$ | 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: *SAINTE* (Bruker, 1999); data reduction: *SAINTE*; 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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supporting information

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1-(2-Methoxyethoxy)-4-nitrobenzene

Yang Liu, Wei-Na Xu, Xiao-Ling Zhang, Jian-Ping Ma and Dian-Shun Guo

S1. 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).

S2. 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.

S3. 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 $C_{\text{methyl}}\text{---H} = 0.96 \text{ \AA}$, $C_{\text{methylene}}\text{---H} = 0.97 \text{ \AA}$ and $C_{\text{aromatic}}\text{---H} = 0.93 \text{ \AA}$ and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$.

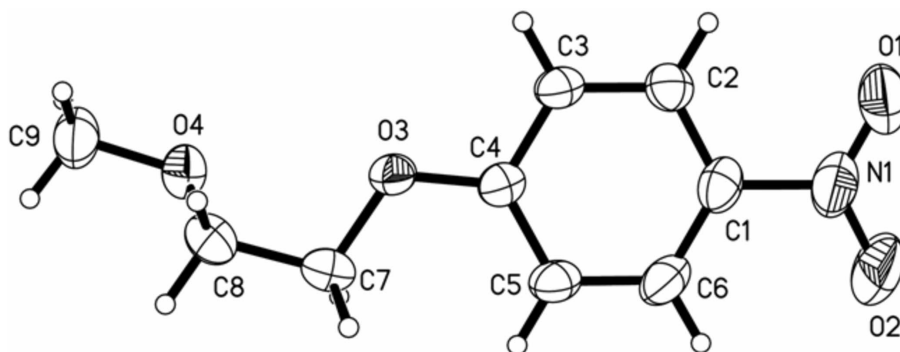


Figure 1

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

C₉H₁₁NO₄ $M_r = 197.19$ Orthorhombic, $Pna2_1$ $a = 11.280$ (3) Å $b = 20.430$ (5) Å $c = 4.1079$ (10) Å $V = 946.7$ (4) Å³ $Z = 4$ $F(000) = 416$ $D_x = 1.384$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2528 reflections

 $\theta = 2.7$ – 27.8° $\mu = 0.11$ mm⁻¹ $T = 100$ K

Block, colourless

 $0.49 \times 0.43 \times 0.38$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 ϕ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 1999)

 $T_{\min} = 0.948$, $T_{\max} = 0.959$

4034 measured reflections

1023 independent reflections

982 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.030$ $\theta_{\max} = 25.7^\circ$, $\theta_{\min} = 2.0^\circ$ $h = -12 \rightarrow 13$ $k = -15 \rightarrow 24$ $l = -4 \rightarrow 4$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.031$ $wR(F^2) = 0.079$ $S = 1.05$

1023 reflections

130 parameters

1 restraint

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0395P)^2 + 0.1764P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.001$ $\Delta\rho_{\max} = 0.13$ e Å⁻³ $\Delta\rho_{\min} = -0.12$ e Å⁻³

Extinction correction: SHELXL97,

 $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.032 (4)

Absolute structure: Flack (1983)

Absolute structure 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 $F^2 > \sigma(F^2)$ 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 | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|---------------|-------------|-------------|----------------------------------|
| 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 (Å²)

| | 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 (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------------------|-------|-------------|-------------|---------------|
| C5—H5 \cdots 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) $-x, -y+1, z-1/2$.