

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

4-[3-(4-Nitrophenoxy)propoxy]aniline

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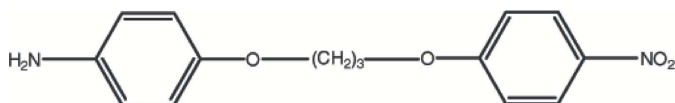
Received 27 September 2008; accepted 7 October 2008

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.046; wR factor = 0.126; data-to-parameter ratio = 13.2.

The molecules of the title compound, $\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_4$, are linked via $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming undulating one-dimensional chains. Adjacent chains are linked by weak $\text{C}-\text{H}\cdots\pi$ interactions, forming a three-dimensional network.

Related literature

For general background, see: Day & Arnold (2000); Day *et al.* (2002); Freeman *et al.* (1981); Kim *et al.* (2000).



Experimental

Crystal data

$\text{C}_{15}\text{H}_{16}\text{N}_2\text{O}_4$
 $M_r = 288.30$
 Orthorhombic, *Pccn*
 $a = 10.808$ (8) Å
 $b = 34.79$ (3) Å
 $c = 7.596$ (6) Å

$V = 2857$ (4) Å³
 $Z = 8$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 298$ (2) K
 $0.23 \times 0.19 \times 0.16$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
 $T_{\min} = 0.978$, $T_{\max} = 0.984$
 17736 measured reflections
 2509 independent reflections
 1554 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.126$
 $S = 1.05$
 2509 reflections
 190 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.16$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N2}-\text{H2B}\cdots\text{O1}^{\text{i}}$	0.86	2.29	3.123 (3)	164
$\text{C3}-\text{H3}\cdots\text{Cg1}^{\text{ii}}$	0.93	3.07	3.513 (4)	111
$\text{C7}-\text{H7B}\cdots\text{Cg2}^{\text{iii}}$	0.97	2.71	3.567 (4)	148
$\text{C13}-\text{H13}\cdots\text{Cg2}^{\text{iv}}$	0.93	3.01	3.757 (4)	139

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + \frac{1}{2}, y, z - \frac{3}{2}$; (iii) $x, -y - \frac{1}{2}, z - \frac{3}{2}$; (iv) $-x - \frac{1}{2}, y, z - \frac{1}{2}$. Cg1 and Cg2 are the centroids of the $\text{C1}-\text{C6}$ and $\text{C10}-\text{C15}$ phenyl rings, respectively.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINTE* (Bruker, 2005); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

We acknowledge the support of the National Natural Science Foundation of China (No. 20662003) and the Foundation of the Governor of Guizhou Province, China.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2638).

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supplementary materials

Acta Cryst. (2008). E64, o2110 [doi:10.1107/S1600536808032406]

4-[3-(4-Nitrophenoxy)propoxy]aniline

L.-M. Zheng, X. Wei, X.-R. Peng, J.-P. Zeng and Y.-Q. Zhang

Comment

As part of our ongoing investigation on bibenzene compound, we present the crystal structure of the title compound (I) containing multiple functional groups that can develop strong interactions with cucurbit[*n*]urils (CB[*n*]) (Freeman *et al.*, 1981; Day & Arnold, 2000; Day *et al.*, 2002; Kim *et al.*, 2000)

The crystal structure of (I) is shown in Fig.1. Two phenyl rings were linked by ethereal chain forming a non-coplanar structure and the dihedral angle between two phenyl ring is 26.13 (9) Å. Molecules are linked *via* N2—H2B···O1 hydrogen bonds forming a undulant one-dimensional chains (Fig. 2) and adjacent chains are linked by C—H··· π interaction forming a three-dimensional framework (Table 1, *Cg*1 and *Cg*2 are centroids of the phenyl ring (C1—C6) and (C10—C15), respectively).

Experimental

P-toluenesulfonyl chloride (7.62 g, 40 mmol) was added slowly, whilst stirring, to a pyridine solution (50 ml) containing 1,3-propanediol (1.52 g, 20 mmol). The mixture was stirred for about 4 h in the range of 268 K - 278 K. Water (40 ml) was added to the resulting solution, the precipitate was collected by filtration, the solid product was crystallized using ethanol. The solid product (6.85 g, 20 mmol) dissolved in DMF (100 ml) containing K₂CO₃ (2 g), *p*-nitrophenol (0.54 g, 4 mmol) was added slowly, to the DMF(100 ml) solution, and the mixture was heated at 353 K for 24 h, and then the solvent was removed into water and filtered, the residue was washed with water, and 1,3-bis(-nitrylphenoxy)-propane was obtained. Hydrazine (30 g, 80%) was added slowly to a stirred solution of ethanol (50 ml) containing 1,4-bis(-nitrylphenoxy)-propane (3.12 g, 10 mmol), FeCl₃·6H₂O (0.8 g) and active carbon (1.8 g) at 348 K for 5 h, and then the solvent was filtered, the solid product was crystallized using ethanol, Single crystals of (I) were obtained after a week.

Refinement

All H atoms were placed in calculated positions and refined as riding, with C—H = 0.97 Å (methylene) and 0.93 Å (aromatic), N—H = 0.861 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C},\text{N})$.

Figures

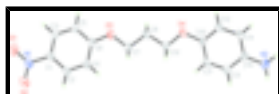


Fig. 1. The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level.

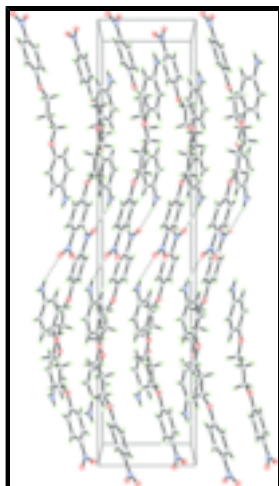


Fig. 2. Packing diagram of (I). Hydrogen bonds are shown as dashed lines.

4-[3-(4-Nitrophenoxy)propoxy]aniline

Crystal data

$C_{15}H_{16}N_2O_4$

$M_r = 288.30$

Orthorhombic, *Pccn*

Hall symbol: -P 2ab 2ac

$a = 10.808 (8) \text{ \AA}$

$b = 34.79 (3) \text{ \AA}$

$c = 7.596 (6) \text{ \AA}$

$V = 2857 (4) \text{ \AA}^3$

$Z = 8$

$F_{000} = 1216$

$D_x = 1.341 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2509 reflections

$\theta = 2.0\text{--}25.0^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 298 (2) \text{ K}$

Prism, brown

$0.23 \times 0.19 \times 0.16 \text{ mm}$

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2) \text{ K}$

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.978$, $T_{\max} = 0.984$

17736 measured reflections

2509 independent reflections

1554 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.065$

$\theta_{\max} = 25.0^\circ$

$\theta_{\min} = 2.0^\circ$

$h = -12 \rightarrow 12$

$k = -37 \rightarrow 41$

$l = -8 \rightarrow 9$

Refinement

Refinement on F^2

Least-squares matrix: full

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.046$	H-atom parameters constrained
$wR(F^2) = 0.126$	$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2509 reflections	$(\Delta/\sigma)_{\max} = 0.001$
190 parameters	$\Delta\rho_{\max} = 0.25 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$
	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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.3694 (2)	0.52612 (6)	0.2758 (3)	0.0531 (6)
C2	0.4692 (2)	0.54418 (6)	0.1976 (3)	0.0587 (6)
H2	0.5427	0.5308	0.1793	0.070*
C3	0.2592 (2)	0.54512 (6)	0.3021 (3)	0.0575 (6)
H3	0.1926	0.5326	0.3541	0.069*
C4	0.4590 (2)	0.58237 (6)	0.1467 (3)	0.0528 (6)
H4	0.5259	0.5949	0.0950	0.063*
C5	0.2486 (2)	0.58278 (6)	0.2507 (3)	0.0556 (6)
H5	0.1743	0.5957	0.2676	0.067*
C6	0.3483 (2)	0.60180 (6)	0.1735 (3)	0.0468 (5)
C7	0.4269 (2)	0.66196 (6)	0.0598 (3)	0.0540 (6)
H7A	0.4487	0.6531	-0.0571	0.065*
H7B	0.4994	0.6600	0.1346	0.065*
C8	0.3816 (2)	0.70308 (6)	0.0527 (3)	0.0577 (6)
H8A	0.3102	0.7046	-0.0244	0.069*
H8B	0.3558	0.7110	0.1695	0.069*
C9	0.4804 (2)	0.73000 (5)	-0.0131 (3)	0.0539 (6)
H9A	0.5580	0.7249	0.0468	0.065*
H9B	0.4929	0.7264	-0.1384	0.065*
C10	0.5238 (2)	0.79809 (6)	-0.0124 (3)	0.0459 (5)
C11	0.6378 (2)	0.79332 (6)	-0.0955 (3)	0.0507 (6)
H11	0.6630	0.7690	-0.1311	0.061*
C12	0.4878 (2)	0.83474 (6)	0.0389 (3)	0.0493 (6)
H12	0.4117	0.8383	0.0935	0.059*

supplementary materials

C13	0.7136 (2)	0.82491 (6)	-0.1252 (3)	0.0534 (6)
H13	0.7894	0.8214	-0.1808	0.064*
C14	0.5645 (2)	0.86604 (6)	0.0094 (3)	0.0512 (6)
H14	0.5393	0.8903	0.0455	0.061*
C15	0.6788 (2)	0.86171 (6)	-0.0736 (3)	0.0487 (5)
N1	0.3792 (2)	0.48636 (6)	0.3322 (3)	0.0763 (7)
N2	0.75855 (17)	0.89324 (5)	-0.0993 (3)	0.0694 (6)
H2A	0.8298	0.8898	-0.1474	0.083*
H2B	0.7361	0.9159	-0.0669	0.083*
O1	0.29183 (19)	0.47143 (5)	0.4107 (3)	0.1127 (8)
O2	0.4737 (2)	0.46834 (5)	0.3007 (4)	0.1200 (9)
O3	0.32733 (13)	0.63919 (4)	0.1305 (2)	0.0590 (4)
O4	0.44110 (13)	0.76862 (4)	0.0214 (2)	0.0570 (4)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0549 (15)	0.0357 (14)	0.0687 (15)	0.0006 (11)	-0.0019 (12)	0.0067 (11)
C2	0.0491 (15)	0.0477 (15)	0.0793 (17)	0.0004 (12)	0.0011 (12)	0.0025 (12)
C3	0.0561 (16)	0.0499 (15)	0.0664 (15)	-0.0045 (12)	0.0055 (12)	0.0072 (12)
C4	0.0490 (14)	0.0455 (14)	0.0640 (15)	-0.0046 (11)	-0.0003 (11)	0.0053 (11)
C5	0.0505 (15)	0.0487 (15)	0.0677 (15)	0.0011 (12)	0.0070 (12)	0.0050 (12)
C6	0.0545 (14)	0.0361 (13)	0.0496 (13)	-0.0013 (11)	-0.0073 (11)	0.0021 (10)
C7	0.0594 (15)	0.0428 (14)	0.0598 (14)	-0.0065 (11)	-0.0049 (12)	0.0052 (10)
C8	0.0602 (15)	0.0445 (14)	0.0684 (15)	-0.0038 (11)	-0.0088 (12)	0.0069 (11)
C9	0.0666 (16)	0.0383 (13)	0.0566 (14)	0.0025 (11)	-0.0009 (11)	0.0040 (10)
C10	0.0493 (14)	0.0380 (13)	0.0504 (12)	0.0008 (11)	-0.0028 (10)	0.0031 (10)
C11	0.0524 (14)	0.0404 (13)	0.0594 (14)	0.0082 (11)	0.0000 (12)	-0.0023 (10)
C12	0.0513 (14)	0.0453 (14)	0.0514 (13)	0.0054 (11)	0.0031 (10)	-0.0005 (10)
C13	0.0493 (14)	0.0524 (15)	0.0584 (14)	0.0026 (11)	0.0019 (11)	0.0001 (11)
C14	0.0615 (15)	0.0391 (13)	0.0530 (14)	0.0025 (11)	-0.0022 (12)	-0.0065 (10)
C15	0.0500 (14)	0.0465 (14)	0.0495 (12)	-0.0027 (11)	-0.0038 (11)	0.0005 (10)
N1	0.0723 (17)	0.0485 (15)	0.1081 (18)	0.0008 (12)	0.0043 (14)	0.0138 (12)
N2	0.0672 (13)	0.0544 (13)	0.0867 (15)	-0.0160 (11)	0.0080 (11)	-0.0116 (11)
O1	0.0956 (16)	0.0661 (13)	0.176 (2)	-0.0062 (11)	0.0244 (15)	0.0501 (13)
O2	0.0921 (15)	0.0634 (14)	0.205 (3)	0.0244 (12)	0.0344 (16)	0.0389 (14)
O3	0.0557 (9)	0.0410 (9)	0.0805 (11)	-0.0021 (7)	-0.0004 (8)	0.0110 (8)
O4	0.0583 (10)	0.0381 (9)	0.0745 (11)	0.0006 (8)	0.0061 (8)	0.0040 (7)

Geometric parameters (\AA , $^\circ$)

C1—C3	1.377 (3)	C9—O4	1.433 (2)
C1—C2	1.383 (3)	C9—H9A	0.9700
C1—N1	1.453 (3)	C9—H9B	0.9700
C2—C4	1.388 (3)	C10—O4	1.385 (2)
C2—H2	0.9300	C10—C12	1.389 (3)
C3—C5	1.372 (3)	C10—C11	1.393 (3)
C3—H3	0.9300	C11—C13	1.390 (3)
C4—C6	1.390 (3)	C11—H11	0.9300

C4—H4	0.9300	C12—C14	1.387 (3)
C5—C6	1.394 (3)	C12—H12	0.9300
C5—H5	0.9300	C13—C15	1.391 (3)
C6—O3	1.361 (3)	C13—H13	0.9300
C7—O3	1.440 (2)	C14—C15	1.394 (3)
C7—C8	1.513 (3)	C14—H14	0.9300
C7—H7A	0.9700	C15—N2	1.409 (3)
C7—H7B	0.9700	N1—O2	1.221 (2)
C8—C9	1.506 (3)	N1—O1	1.232 (3)
C8—H8A	0.9700	N2—H2A	0.8600
C8—H8B	0.9700	N2—H2B	0.8600
C3—C1—C2	121.3 (2)	O4—C9—H9A	110.1
C3—C1—N1	118.6 (2)	C8—C9—H9A	110.1
C2—C1—N1	120.1 (2)	O4—C9—H9B	110.1
C1—C2—C4	119.5 (2)	C8—C9—H9B	110.1
C1—C2—H2	120.2	H9A—C9—H9B	108.4
C4—C2—H2	120.2	O4—C10—C12	116.53 (19)
C5—C3—C1	119.3 (2)	O4—C10—C11	124.51 (19)
C5—C3—H3	120.4	C12—C10—C11	119.0 (2)
C1—C3—H3	120.4	C13—C11—C10	120.0 (2)
C2—C4—C6	119.6 (2)	C13—C11—H11	120.0
C2—C4—H4	120.2	C10—C11—H11	120.0
C6—C4—H4	120.2	C14—C12—C10	120.5 (2)
C3—C5—C6	120.6 (2)	C14—C12—H12	119.7
C3—C5—H5	119.7	C10—C12—H12	119.7
C6—C5—H5	119.7	C11—C13—C15	121.5 (2)
O3—C6—C4	124.99 (19)	C11—C13—H13	119.3
O3—C6—C5	115.26 (19)	C15—C13—H13	119.3
C4—C6—C5	119.8 (2)	C12—C14—C15	121.17 (19)
O3—C7—C8	106.95 (18)	C12—C14—H14	119.4
O3—C7—H7A	110.3	C15—C14—H14	119.4
C8—C7—H7A	110.3	C13—C15—C14	117.81 (19)
O3—C7—H7B	110.3	C13—C15—N2	120.8 (2)
C8—C7—H7B	110.3	C14—C15—N2	121.4 (2)
H7A—C7—H7B	108.6	O2—N1—O1	121.3 (2)
C9—C8—C7	111.73 (19)	O2—N1—C1	119.5 (2)
C9—C8—H8A	109.3	O1—N1—C1	119.2 (2)
C7—C8—H8A	109.3	C15—N2—H2A	120.0
C9—C8—H8B	109.3	C15—N2—H2B	120.0
C7—C8—H8B	109.3	H2A—N2—H2B	120.0
H8A—C8—H8B	107.9	C6—O3—C7	119.42 (17)
O4—C9—C8	108.19 (18)	C10—O4—C9	117.95 (17)
C3—C1—C2—C4	0.9 (3)	C10—C11—C13—C15	0.0 (3)
N1—C1—C2—C4	-179.1 (2)	C10—C12—C14—C15	0.6 (3)
C2—C1—C3—C5	-0.4 (4)	C11—C13—C15—C14	0.0 (3)
N1—C1—C3—C5	179.5 (2)	C11—C13—C15—N2	177.85 (19)
C1—C2—C4—C6	-0.6 (3)	C12—C14—C15—C13	-0.3 (3)
C1—C3—C5—C6	-0.3 (3)	C12—C14—C15—N2	-178.14 (19)

supplementary materials

C2—C4—C6—O3	179.49 (19)	C3—C1—N1—O2	175.7 (2)
C2—C4—C6—C5	-0.1 (3)	C2—C1—N1—O2	-4.4 (4)
C3—C5—C6—O3	-179.1 (2)	C3—C1—N1—O1	-4.1 (4)
C3—C5—C6—C4	0.6 (3)	C2—C1—N1—O1	175.8 (2)
O3—C7—C8—C9	177.80 (17)	C4—C6—O3—C7	-3.4 (3)
C7—C8—C9—O4	-167.07 (17)	C5—C6—O3—C7	176.24 (18)
O4—C10—C11—C13	179.26 (19)	C8—C7—O3—C6	-171.25 (17)
C12—C10—C11—C13	0.3 (3)	C12—C10—O4—C9	-173.68 (17)
O4—C10—C12—C14	-179.64 (18)	C11—C10—O4—C9	7.3 (3)
C11—C10—C12—C14	-0.6 (3)	C8—C9—O4—C10	173.62 (17)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N2—H2B \cdots O1 ⁱ	0.86	2.29	3.123 (3)	164
C3—H3 \cdots Cg1 ⁱⁱ	0.93	3.07	3.513 (4)	111
C7—H7B \cdots Cg2 ⁱⁱⁱ	0.97	2.71	3.567 (4)	148
C13—H13 \cdots Cg2 ^{iv}	0.93	3.01	3.757 (4)	139

Symmetry codes: (i) $-x+1, y+1/2, -z+1/2$; (ii) $-x+1/2, y, z-3/2$; (iii) $x, -y-1/2, z-3/2$; (iv) $-x-1/2, y, z-1/2$.

Fig. 1

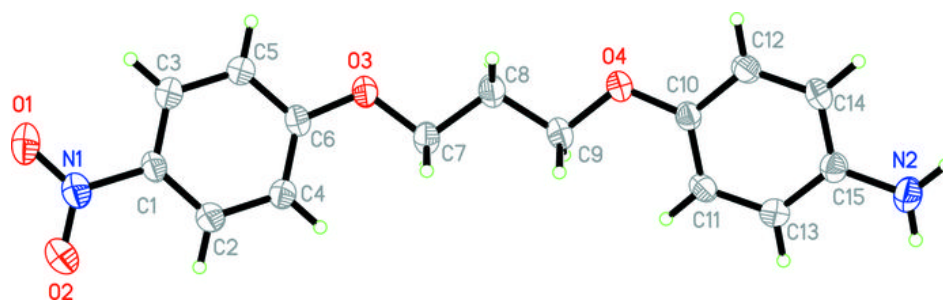


Fig. 2

