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1,4-Bis(4-methyl-2-nitrophenoxy)butane

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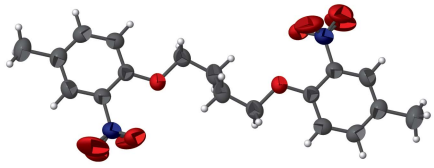
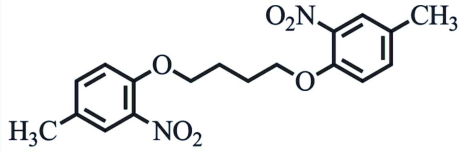
Structural data: full structural data are available from iucrdata.iucr.org

The asymmetric unit of the title compound, $C_{18}H_{20}N_2O_6$, contains one-half molecule, the mid-point of the central C—C bond being located on a crystallographic inversion centre. In the crystal, weak C—H···O interactions generate a layered structure. The O atoms of the nitro group are disordered over two sets of sites with a refined occupancy ratio of 0.700 (8):0.300 (8).

3D view



Chemical scheme



Structure description

The title compound (Fig. 1) crystallizes with the molecule being situated on a crystallographic inversion centre located at the midpoint of the C7—C7*A* bond. The two parallel phenyl rings are linked by an ethereal chain, forming a non-coplanar structure similar to that described by Elizondo *et al.* (2009).

In the crystal, the molecules are linked into chains by the C7—H7*A*···O3 interactions (Table 1, Fig. 2). The chains are connected into layers by C9—H9*A*···O2 interactions (Table 1, Fig. 3).

Synthesis and crystallization

To a solution of 4-methyl-2-nitrophenol (5.00 g, 32.7 mmol) in acetonitrile (100 ml) were added potassium carbonate (6.78 g, 50.0 mmol) and 1,4-dibromobutane (3.30 g, 15.3 mmol). After the reaction mixture had been refluxed for 6 h, all the volatile components were evaporated and the residue was partitioned between dichloromethane and water. The organic phase was washed with water, then dried in calcium chloride, and concentrated *in vacuo* to give an off-white solid. White single crystals were obtained in a yield of 62% using acetonitrile crude extraction.

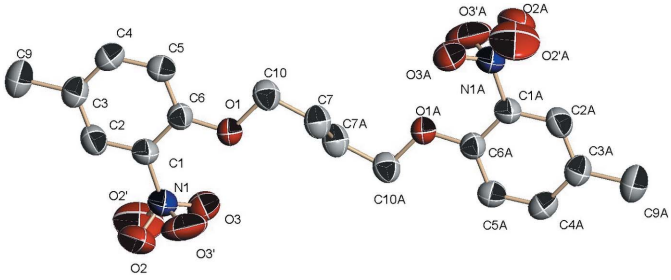


Figure 1
The molecular structure of the title molecule, with displacement ellipsoids drawn at the 50% probability level.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The O atoms of the nitro group are disordered over two sets of sites (O2/O2′ and O3/O3′) with a refined occupancy ratio of 0.700 (8):0.300 (8).

Funding information

This work was supported by the Graduate Students Innovative Program of Anhui University (J18515024, J18515019, 201310357155).

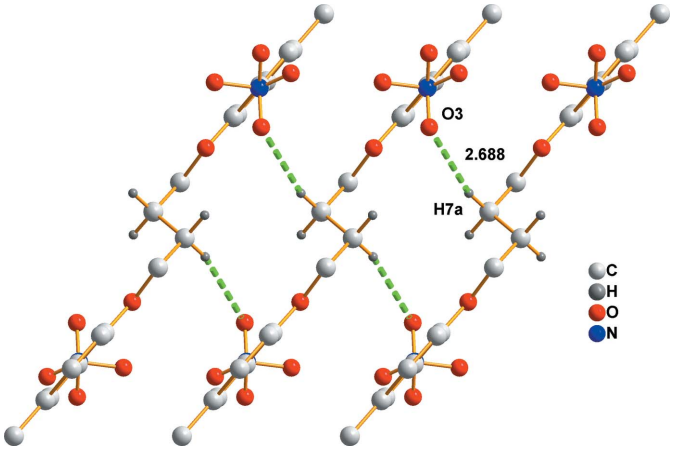


Figure 2
The chains generated by C7—H7*A*···O3 interactions (green dashed lines). H atoms not involved in these interactions have been omitted.

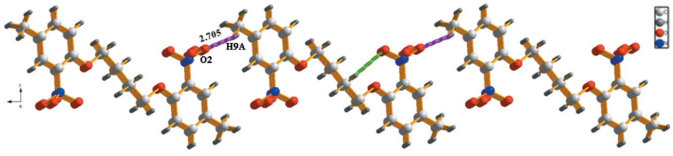


Figure 3
A view along the *c* axis of the crystal packing of the title compound. The C9—H9*A*···O interactions are represented by purple dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O3^{i}	0.97	2.69	3.637 (3)	166
C9—H9*A*···O2^{ii}	0.96	2.71	3.516 (4)	143

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{18}H_{20}N_2O_6$
M_r	360.36
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	296
a, *b*, *c* (Å)	4.7936 (7), 12.9828 (19), 14.632 (2)
β (°)	92.986 (2)
V (Å^3)	909.4 (2)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.2 × 0.2 × 0.2
Data collection	
Diffractometer	Bruker SMART CCD area detector
Absorption correction	Multi-scan (*SADABS*; Bruker, 2004)
T_{min}, T_{max}	0.950, 0.966
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	6305, 1603, 1348
R_{int}	0.022
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.594
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.043, 0.124, 1.08
No. of reflections	1603
No. of parameters	137
No. of restraints	16
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.21, −0.18

Computer programs: *SMART* and *SAINT* (Bruker, 2004), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *DIAMOND* (Brandenburg, 2007).

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full crystallographic data

IUCrData (2017). **2**, x171734 [https://doi.org/10.1107/S2414314617017345]

1,4-Bis(4-methyl-2-nitrophenoxy)butane

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Crystal data

$C_{18}H_{20}N_2O_6$
M_r = 360.36
Monoclinic, $P2_1/n$
a = 4.7936 (7) Å
b = 12.9828 (19) Å
c = 14.632 (2) Å
β = 92.986 (2)°
V = 909.4 (2) Å^3
Z = 2
$F(000)$ = 380
D_x = 1.316 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3390 reflections
θ = 2.9–25.0°
μ = 0.10 mm^{-1}
T = 296 K
Block, white
0.2 × 0.2 × 0.2 mm

Data collection

Bruker SMART CCD area detector diffractometer
none scans
Absorption correction: multi-scan (SADABS; Bruker, 2004)
T_{min} = 0.950, T_{max} = 0.966
6305 measured reflections
1603 independent reflections
1348 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 25.0°, θ_{min} = 2.1°
h = −5→5
k = −14→15
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.124
S = 1.08
1603 reflections
137 parameters
16 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 0.1678P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. All hydrogen atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 Å and U_{ISO}(H) = 1.2 U_{eq}.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5424 (3)	0.69514 (12)	0.46590 (10)	0.0557 (4)	
C2	0.7120 (4)	0.62215 (12)	0.42827 (11)	0.0625 (4)	
H2	0.8114	0.5764	0.4665	0.075*	
C3	0.7361 (3)	0.61619 (12)	0.33476 (11)	0.0597 (4)	
C4	0.5797 (4)	0.68498 (13)	0.28134 (11)	0.0619 (4)	
H4	0.5918	0.6826	0.2181	0.074*	
C5	0.4057 (3)	0.75731 (12)	0.31813 (11)	0.0579 (4)	
H5	0.3029	0.8018	0.2796	0.069*	
C6	0.3835 (3)	0.76397 (11)	0.41205 (10)	0.0506 (4)	
C7	−0.0937 (3)	0.97110 (12)	0.46540 (12)	0.0615 (4)	
H7A	−0.2184	0.9270	0.4978	0.074*	
H7B	−0.2074	1.0203	0.4303	0.074*	
C9	0.9223 (4)	0.53703 (15)	0.29328 (15)	0.0833 (6)	
H9A	1.0814	0.5243	0.3343	0.125*	
H9B	0.9840	0.5623	0.2361	0.125*	
H9C	0.8200	0.4742	0.2831	0.125*	
C10	0.0657 (3)	0.90647 (13)	0.40078 (11)	0.0609 (4)	
H10A	−0.0619	0.8727	0.3567	0.073*	
H10B	0.1926	0.9491	0.3678	0.073*	
O1	0.2189 (2)	0.83138 (8)	0.45505 (7)	0.0604 (3)	
N1	0.5373 (4)	0.69963 (13)	0.56589 (11)	0.0819 (5)	
O2	0.5488 (9)	0.6235 (2)	0.6110 (2)	0.1177 (11)	0.7
O3	0.5494 (5)	0.78584 (16)	0.60470 (12)	0.0886 (6)	0.7
O2′	0.739 (2)	0.6707 (9)	0.6058 (5)	0.177 (4)	0.3
O3′	0.2837 (15)	0.6876 (6)	0.5918 (4)	0.141 (3)	0.3

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0667 (9)	0.0528 (8)	0.0476 (8)	0.0036 (7)	0.0022 (7)	0.0013 (6)
C2	0.0664 (10)	0.0537 (9)	0.0664 (10)	0.0092 (7)	−0.0050 (8)	0.0002 (7)
C3	0.0558 (9)	0.0574 (9)	0.0663 (10)	−0.0025 (7)	0.0067 (7)	−0.0111 (7)
C4	0.0663 (10)	0.0688 (10)	0.0511 (8)	−0.0019 (8)	0.0071 (7)	−0.0071 (7)
C5	0.0615 (9)	0.0610 (9)	0.0508 (8)	0.0028 (7)	−0.0002 (7)	0.0034 (7)
C6	0.0515 (8)	0.0478 (8)	0.0526 (8)	0.0000 (6)	0.0044 (6)	−0.0007 (6)
C7	0.0490 (8)	0.0572 (9)	0.0777 (11)	0.0043 (7)	−0.0025 (7)	−0.0042 (8)
C9	0.0799 (12)	0.0762 (12)	0.0949 (14)	0.0115 (10)	0.0164 (10)	−0.0192 (11)
C10	0.0570 (9)	0.0601 (9)	0.0646 (9)	0.0081 (7)	−0.0067 (7)	0.0000 (7)
O1	0.0699 (7)	0.0556 (6)	0.0562 (6)	0.0144 (5)	0.0083 (5)	0.0020 (5)
N1	0.1193 (14)	0.0729 (8)	0.0541 (9)	0.0261 (9)	0.0099 (9)	0.0099 (6)
O2	0.194 (3)	0.0827 (15)	0.0775 (16)	0.0153 (18)	0.0220 (19)	0.0265 (12)
O3	0.1283 (17)	0.0881 (11)	0.0489 (10)	0.0176 (12)	−0.0013 (10)	−0.0082 (8)
O2′	0.244 (10)	0.218 (10)	0.062 (4)	0.064 (8)	−0.063 (6)	0.014 (5)
O3′	0.176 (6)	0.186 (7)	0.064 (3)	0.028 (6)	0.041 (4)	0.030 (4)

Geometric parameters (Å, º)

C1—C2	1.382 (2)	C7—C7^{i}	1.516 (3)
C1—C6	1.392 (2)	C7—H7A	0.9700
C1—N1	1.466 (2)	C7—H7B	0.9700
C2—C3	1.381 (2)	C9—H9A	0.9600
C2—H2	0.9300	C9—H9B	0.9600
C3—C4	1.382 (2)	C9—H9C	0.9600
C3—C9	1.509 (2)	C10—O1	1.4353 (18)
C4—C5	1.383 (2)	C10—H10A	0.9700
C4—H4	0.9300	C10—H10B	0.9700
C5—C6	1.387 (2)	N1—O2′	1.164 (7)
C5—H5	0.9300	N1—O2	1.188 (3)
C6—O1	1.3550 (18)	N1—O3	1.255 (2)
C7—C10	1.502 (2)	N1—O3′	1.301 (6)
C2—C1—C6	122.06 (14)	C7^{i}—C7—H7B	108.9
C2—C1—N1	117.77 (14)	H7A—C7—H7B	107.8
C6—C1—N1	120.16 (14)	C3—C9—H9A	109.5
C3—C2—C1	120.97 (15)	C3—C9—H9B	109.5
C3—C2—H2	119.5	H9A—C9—H9B	109.5
C1—C2—H2	119.5	C3—C9—H9C	109.5
C2—C3—C4	116.94 (14)	H9A—C9—H9C	109.5
C2—C3—C9	121.21 (17)	H9B—C9—H9C	109.5
C4—C3—C9	121.84 (16)	O1—C10—C7	107.07 (13)
C3—C4—C5	122.61 (15)	O1—C10—H10A	110.3
C3—C4—H4	118.7	C7—C10—H10A	110.3
C5—C4—H4	118.7	O1—C10—H10B	110.3
C4—C5—C6	120.46 (15)	C7—C10—H10B	110.3
C4—C5—H5	119.8	H10A—C10—H10B	108.6
C6—C5—H5	119.8	C6—O1—C10	118.39 (12)
O1—C6—C5	125.24 (14)	O2—N1—O3	119.4 (2)
O1—C6—C1	117.81 (13)	O2′—N1—O3′	125.3 (6)
C5—C6—C1	116.94 (14)	O2′—N1—C1	115.6 (5)
C10—C7—C7^{i}	113.19 (16)	O2—N1—C1	121.2 (2)
C10—C7—H7A	108.9	O3—N1—C1	118.97 (16)
C7^{i}—C7—H7A	108.9	O3′—N1—C1	110.5 (3)
C10—C7—H7B	108.9		
C6—C1—C2—C3	1.6 (3)	C7^{i}—C7—C10—O1	61.7 (2)
N1—C1—C2—C3	−177.34 (16)	C5—C6—O1—C10	−3.8 (2)
C1—C2—C3—C4	−1.1 (2)	C1—C6—O1—C10	176.66 (13)
C1—C2—C3—C9	179.65 (16)	C7—C10—O1—C6	−179.22 (12)
C2—C3—C4—C5	0.1 (2)	C2—C1—N1—O2′	26.8 (7)
C9—C3—C4—C5	179.30 (16)	C6—C1—N1—O2′	−152.2 (7)
C3—C4—C5—C6	0.5 (3)	C2—C1—N1—O2	−37.3 (4)
C4—C5—C6—O1	−179.66 (14)	C6—C1—N1—O2	143.8 (3)
C4—C5—C6—C1	−0.1 (2)	C2—C1—N1—O3	135.5 (2)

C2—C1—C6—O1	178.64 (14)	C6—C1—N1—O3	−43.5 (3)
N1—C1—C6—O1	−2.4 (2)	C2—C1—N1—O3′	−122.8 (4)
C2—C1—C6—C5	−0.9 (2)	C6—C1—N1—O3′	58.2 (4)
N1—C1—C6—C5	177.98 (16)		

Symmetry code: (i) −*x*, −*y*+2, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7*A*···O3^{ii}	0.97	2.69	3.637 (3)	166
C9—H9*A*···O2^{iii}	0.96	2.71	3.516 (4)	143

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