

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 2,4-Dinitro-1-phenoxybenzene

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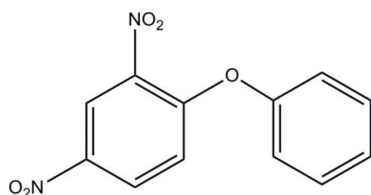
Received 19 December 2009; accepted 15 January 2010

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.006$  Å; disorder in main residue;  $R$  factor = 0.066;  $wR$  factor = 0.220; data-to-parameter ratio = 9.8.

The title compound,  $\text{C}_{12}\text{H}_8\text{N}_2\text{O}_5$ , was obtained by the reaction of 1-chloro-2,4-dinitrobenzene and phenol in the presence of potassium carbonate. The nitro-substituted benzene ring lies on a mirror plane, with one  $\text{NO}_2$  group in the same plane and the other disordered across this plane. The phenoxybenzene unit is placed perpendicular to this mirror, resulting in an exact orthogonal relationship between the phenyl and benzene rings in the molecule. The crystal packing exhibits no significantly short intermolecular contacts.

## Related literature

For the synthesis of the title ether, see: Williamson (1852); Paul & Gupta (2004). For a related structure, see: Gopal *et al.* (1980).



## Experimental

## Crystal data

$\text{C}_{12}\text{H}_8\text{N}_2\text{O}_5$   
 $M_r = 260.20$   
 Orthorhombic,  $Pnma$   
 $a = 21.012$  (13) Å  
 $b = 6.917$  (4) Å  
 $c = 8.211$  (5) Å

$V = 1193.4$  (12) Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.12$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.50 \times 0.47 \times 0.45$  mm

## Data collection

Bruker SMART APEX CCD area-detector diffractometer  
 Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)  
 $T_{\min} = 0.945$ ,  $T_{\max} = 0.950$

5246 measured reflections  
 1150 independent reflections  
 639 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.069$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$   
 $wR(F^2) = 0.220$   
 $S = 1.03$   
 1150 reflections  
 117 parameters

16 restraints  
 H-atom parameters constrained  
 $\Delta\rho_{\max} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.20$  e Å<sup>-3</sup>

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The authors acknowledge the support of the Foundation of Northwest A&F University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2266).

## References

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**supplementary materials**

*Acta Cryst.* (2010). E66, o415 [ doi:10.1107/S1600536810001911 ]

## 2,4-Dinitro-1-phenoxybenzene

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### Comment

One of the most common procedures for the synthesis of ethers was originally introduced by Williamson, and involves the reaction of alkoxides with alkyl halides (Williamson, 1852). This method has been known for nearly 170 years, and remains a very useful transformation in organic synthesis (Paul & Gupta, 2004).

In this paper, we present a new crystal structure, 2,4-dinitro-1-phenoxybenzene, (I), which was synthesized by the reaction of 1-chloro-2,4-dinitrobenzene and phenol, in the presence of potassium carbonate (see *Experimental*).

In (I) (Fig. 1), the bond lengths and angles are normal and comparable to those observed in related compounds (*e.g.* Gopal *et al.*, 1980). The angle between the benzene and the phenyl rings is 90° by symmetry. In the crystal structure, no significantly short intermolecular contacts are observed.

### Experimental

1-Chloro-2,4-dinitrobenzene (10 mmol), potassium carbonate (20 mmol), phenol (6 mmol), and 20 ml of acetone were mixed in a 50 ml flask. After stirring for 2 h. at 373 K, the crude product was obtained. Crystals were obtained by recrystallization from *n*-hexane/ethyl acetate. Elemental analysis: calculated for C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>O<sub>5</sub>: C 55.39, H 3.10, N 10.77%; found: C 55.21, H 3.18, N 10.59%.

### Refinement

All H atoms were positioned geometrically, with C—H = 0.93 Å, and refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier C})$ . The refinement was carried-out using a model which includes 16 restraints: in order to converge to a sensible geometry for the phenyl ring mirrored in the symmetry plane, bond lengths C7—C8, C8—C9 and C9—C10 were restrained to 1.39 (1) Å. For the disordered nitro group, bond lengths N1—O2 and N1—O3 were averaged, and atoms N1, O2 and O3 were restrained to have similar displacement parameters.

### Figures

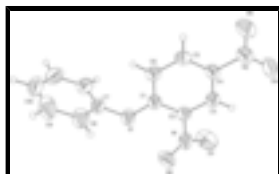


Fig. 1. ORTEP drawing of the title complex with atomic numbering scheme and thermal ellipsoids at 30% probability level. Disordered atoms O2 and O3 generated by symmetry  $x, 1/2-y, z$  ( $m$  plane) have been omitted. Unlabelled atoms in the phenyl ring are generated by symmetry  $x, 1/2-y, z$ .

## 2,4-Dinitro-1-phenoxybenzene

### Crystal data

$C_{12}H_8N_2O_5$	$F(000) = 536$
$M_r = 260.20$	$D_x = 1.448 \text{ Mg m}^{-3}$
Orthorhombic, $Pnma$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
Hall symbol: -P 2ac 2n	Cell parameters from 1105 reflections
$a = 21.012 (13) \text{ \AA}$	$\theta = 2.7\text{--}21.4^\circ$
$b = 6.917 (4) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$c = 8.211 (5) \text{ \AA}$	$T = 298 \text{ K}$
$V = 1193.4 (12) \text{ \AA}^3$	Block, red
$Z = 4$	$0.50 \times 0.47 \times 0.45 \text{ mm}$

### Data collection

Bruker SMART APEX CCD area-detector diffractometer	1150 independent reflections
Radiation source: fine-focus sealed tube graphite	639 reflections with $I > 2\sigma(I)$
$\varphi$ and $\omega$ scans	$R_{\text{int}} = 0.069$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 25.0^\circ$ , $\theta_{\text{min}} = 1.9^\circ$
$T_{\text{min}} = 0.945$ , $T_{\text{max}} = 0.950$	$h = -23 \rightarrow 25$
5246 measured reflections	$k = -8 \rightarrow 8$
	$l = -5 \rightarrow 9$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.066$	H-atom parameters constrained
$wR(F^2) = 0.220$	$w = 1/[\sigma^2(F_o^2) + (0.1048P)^2 + 0.4983P]$
$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
1150 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
117 parameters	$\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
16 restraints	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
0 constraints	Extinction correction: SHELXL97 (Sheldrick, 2008),
Primary atom site location: structure-invariant direct methods	$F_c^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.023 (6)

### Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
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N1	0.4600 (2)	0.2500	0.9620 (5)	0.0970 (18)	
N2	0.5869 (2)	0.2500	0.4695 (6)	0.0782 (13)	
O1	0.35033 (13)	0.2500	0.7731 (4)	0.0844 (13)	
O2	0.4181 (2)	0.3065 (14)	1.0352 (6)	0.120 (3)	0.50
O3	0.5041 (3)	0.1576 (11)	1.0291 (7)	0.146 (3)	0.50
O4	0.63351 (19)	0.2500	0.5549 (6)	0.1135 (17)	
O5	0.5895 (2)	0.2500	0.3233 (6)	0.1127 (16)	
C1	0.4064 (2)	0.2500	0.6926 (6)	0.0600 (13)	
C2	0.4620 (2)	0.2500	0.7843 (5)	0.0597 (13)	
C3	0.5208 (2)	0.2500	0.7124 (6)	0.0641 (13)	
H3	0.5577	0.2500	0.7752	0.077*	
C4	0.5240 (2)	0.2500	0.5464 (6)	0.0590 (12)	
C5	0.4704 (2)	0.2500	0.4509 (6)	0.0633 (13)	
H5	0.4738	0.2500	0.3380	0.076*	
C6	0.4115 (2)	0.2500	0.5237 (6)	0.0662 (14)	
H6	0.3750	0.2500	0.4598	0.079*	
C7	0.2935 (2)	0.2500	0.6851 (6)	0.0691 (15)	
C8	0.26597 (18)	0.4227 (7)	0.6495 (5)	0.0960 (14)	
H8	0.2854	0.5386	0.6781	0.115*	
C9	0.2084 (2)	0.4202 (10)	0.5698 (6)	0.130 (2)	
H9	0.1891	0.5362	0.5410	0.155*	
C10	0.1795 (3)	0.2500	0.5328 (9)	0.139 (4)	
H10	0.1400	0.2500	0.4820	0.167*	

Atomic displacement parameters ( $\text{\AA}^2$ )

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.055 (3)	0.182 (5)	0.054 (3)	0.000	-0.006 (2)	0.000
N2	0.071 (3)	0.089 (3)	0.074 (3)	0.000	0.015 (3)	0.000
O1	0.053 (2)	0.149 (4)	0.0508 (19)	0.000	-0.0026 (16)	0.000
O2	0.089 (3)	0.209 (10)	0.062 (3)	0.054 (4)	-0.001 (2)	-0.017 (4)
O3	0.146 (4)	0.223 (8)	0.070 (3)	0.075 (5)	-0.013 (3)	0.022 (4)
O4	0.061 (2)	0.181 (5)	0.099 (3)	0.000	0.015 (2)	0.000
O5	0.100 (3)	0.161 (4)	0.077 (3)	0.000	0.030 (2)	0.000
C1	0.052 (3)	0.074 (3)	0.054 (3)	0.000	0.000 (2)	0.000
C2	0.053 (3)	0.076 (3)	0.050 (2)	0.000	-0.003 (2)	0.000
C3	0.055 (3)	0.076 (3)	0.061 (3)	0.000	-0.006 (2)	0.000
C4	0.055 (3)	0.057 (3)	0.065 (3)	0.000	0.008 (2)	0.000
C5	0.074 (3)	0.069 (3)	0.047 (3)	0.000	0.004 (2)	0.000
C6	0.062 (3)	0.082 (3)	0.054 (3)	0.000	-0.007 (2)	0.000
C7	0.049 (3)	0.110 (4)	0.048 (3)	0.000	0.000 (2)	0.000
C8	0.085 (3)	0.120 (4)	0.083 (3)	0.007 (3)	-0.004 (2)	0.016 (3)
C9	0.089 (4)	0.209 (7)	0.091 (3)	0.044 (4)	-0.003 (3)	0.042 (4)
C10	0.055 (4)	0.299 (14)	0.063 (4)	0.000	-0.006 (3)	0.000

Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

N1—O2 <sup>i</sup>	1.137 (6)	C3—C4	1.365 (6)
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## supplementary materials

N1—O2	1.137 (6)	C3—H3	0.9300
N1—O3	1.253 (6)	C4—C5	1.373 (6)
N1—O3 <sup>i</sup>	1.253 (6)	C5—C6	1.373 (6)
N1—C2	1.459 (6)	C5—H5	0.9300
N2—O5	1.202 (6)	C6—H6	0.9300
N2—O4	1.204 (6)	C7—C8 <sup>i</sup>	1.358 (5)
N2—C4	1.465 (6)	C7—C8	1.358 (5)
O1—C1	1.351 (5)	C8—C9	1.375 (5)
O1—C7	1.397 (5)	C8—H8	0.9300
O2—O2 <sup>i</sup>	0.78 (2)	C9—C10	1.360 (6)
O3—O3 <sup>i</sup>	1.278 (15)	C9—H9	0.9300
C1—C2	1.389 (6)	C10—C9 <sup>i</sup>	1.360 (6)
C1—C6	1.391 (6)	C10—H10	0.9300
C2—C3	1.371 (6)		
O2 <sup>i</sup> —N1—O3	99.5 (6)	C3—C4—C5	122.0 (4)
O2—N1—O3	121.1 (6)	C3—C4—N2	118.3 (4)
O2 <sup>i</sup> —N1—O3 <sup>i</sup>	121.1 (6)	C5—C4—N2	119.6 (4)
O2—N1—O3 <sup>i</sup>	99.5 (6)	C6—C5—C4	119.4 (4)
O2 <sup>i</sup> —N1—C2	123.4 (4)	C6—C5—H5	120.3
O2—N1—C2	123.4 (4)	C4—C5—H5	120.3
O3—N1—C2	114.8 (4)	C5—C6—C1	120.2 (4)
O3 <sup>i</sup> —N1—C2	114.8 (4)	C5—C6—H6	119.9
O5—N2—O4	123.1 (5)	C1—C6—H6	119.9
O5—N2—C4	118.1 (5)	C8 <sup>i</sup> —C7—C8	123.1 (5)
O4—N2—C4	118.8 (5)	C8 <sup>i</sup> —C7—O1	118.4 (3)
C1—O1—C7	119.5 (4)	C8—C7—O1	118.4 (3)
O1—C1—C2	117.9 (4)	C7—C8—C9	117.7 (5)
O1—C1—C6	123.7 (4)	C7—C8—H8	121.1
C2—C1—C6	118.4 (4)	C9—C8—H8	121.1
C3—C2—C1	121.6 (4)	C10—C9—C8	120.7 (6)
C3—C2—N1	117.1 (4)	C10—C9—H9	119.7
C1—C2—N1	121.3 (4)	C8—C9—H9	119.7
C4—C3—C2	118.3 (4)	C9—C10—C9 <sup>i</sup>	120.0 (7)
C4—C3—H3	120.9	C9—C10—H10	120.0
C2—C3—H3	120.9	C9 <sup>i</sup> —C10—H10	120.0

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

### Hydrogen-bond geometry ( $\text{\AA}, ^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C5—H5 $\cdots$ O3 <sup>ii</sup>	0.93	2.69	3.593 (8)	163
C9—H9 $\cdots$ O2 <sup>iii</sup>	0.93	2.50	3.274 (8)	141

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

Fig. 1

