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4-Nitro-2-phenoxyaniline

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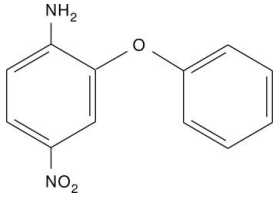
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.053; wR factor = 0.167; data-to-parameter ratio = 12.3.

In the title compound, $C_{12}H_{10}N_2O_3$, the oxygen atom bridging the two aromatic rings is in a synperiplanar (+*sp*) conformation. The dihedral angle between the aromatic rings is 71.40 (12)°. In the crystal, molecules are linked by intermolecular N—H···O hydrogen bonds.

Related literature

For the pharmacological properties of nitro-2-phenoxyaniline, see: Moore & Harrington (1974); Prasad *et al.* (2005). For the herbicidal applications of biphenyl ether derivatives, see: Yu *et al.*, (2008). For the applications of Schiff bases derived from aromatic amines, see: Singh *et al.* (1975); Cimerman *et al.* (2000). For their biological and pharmacological acitvity, see: Singh *et al.* (1975); Cimerman *et al.* (2000); Shah *et al.* (1992); Pandeya *et al.* (1999); More *et al.* (2001). For the preparation of 4-nitro-2-phenoxyaniline, see: Shreenivasa *et al.* (2009). For a related structure, see: Naveen *et al.* (2006).



Experimental

Crystal data

$C_{12}H_{10}N_2O_3$
M_r = 230.22
Monoclinic, $P2_1/c$
a = 10.4100 (12) Å
b = 15.6570 (18) Å
c = 6.9600 (17) Å
β = 103.406 (4)°
V = 1103.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.32 × 0.3 × 0.25 mm

Data collection

MacScience DIPLabo 32001 diffractometer
3336 measured reflections
1889 independent reflections
1498 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.167
S = 1.09
1889 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N10—H10A···O9^{i}	0.86	2.17	3.023 (3)	170

Symmetry code: (i) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *XPRESS* (MacScience, 2002); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FJ2288).

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supporting information

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4-Nitro-2-phenoxyaniline

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S1. Comment

The phenoxy anilines are versatile intermediates for synthesizing several pharmaceutical drugs i.e. Nimesulide, Ampxipine and Loxapine. The Nitro-2-phenoxyaniline is an intermediate for the synthesis of Nimesulide and it was probably the first COX-2 selective non-steroidal anti inflammatory drug (NSAID) identified with this key pharmacological properties (Moore & Harrington, 1974; Prasad et al. 2005). It is a unique molecule with twin aromatic ring structure. The nitro-2-phenoxyaniline is a derivative of biphenyl ether. More generally, biphenyl ether derivatives have many biological, herbicidal (Yu et al., 2008) and organic chemistry applications. Schiff bases derived from aromatic amines have a wide variety of applications in many fields, viz., biological, inorganic and anlytical chemistry (Singh et al., 1975; Cimerman et al., 2000). They are known to exhibit potent antibacterial, anticonvulsant, anti-inflammatory (Shah et al. 1992), anticancer (Pandeya et al., 1999), anti-hypertensive and hypnotic (More et al., 2001) activities. With this background, the title compound (I), was synthesized and we report its crystal structure here.

A perspective view of (I) is shown in Fig. 1. The two aromatic rings are not coplanar. This is confirmed by the dihedral angle value of 71.38 (12)° between two six-membered rings. The oxygen atom connecting the two aromatic rings is in syn-periplanar (sp) conformation as indicated by the torsion angle value of 13.0 (3)°. The nitro group lies in the plane of the aniline ring as indicated by the C2—C1—N7—O8 and C6—C1—N7—O9 torsion angles of -176.1 (2)° and -174.4 (2)°, respectively. These values are different from the values reported earlier (Naveen S. et al. 2006). The structure exhibits both inter and intramolecular N—H···O interaction. The intermolecular N10—H10A···O9 interaction has a length of 2.17Å and angle of 170° with symmetry codes 3/2-x,-1/2+y,1-z. The molecules exhibit layered stackings when viewd down the 'b′ axis as shown in Fig. 2.

S2. Experimental

The 4-nitro-2-phenoxyaniline was prepared by condensation of *o*-chloronitrobenzene with phenol followed by acetylation and nitration (Shreenivasa *et al.*, 2009). The final product obtained was recrystallized using ethanol as a solvent. Colorless crystals were appeared after 4 days by slow evaporation.

S3. Refinement

H atoms were placed at idealized positions and allowed to ride on their parent atoms with C–H distances in the range 0.93–0.98 Å; U_{iso}(H) = 1.2U_{eq}(carrier atom) for all H atoms.

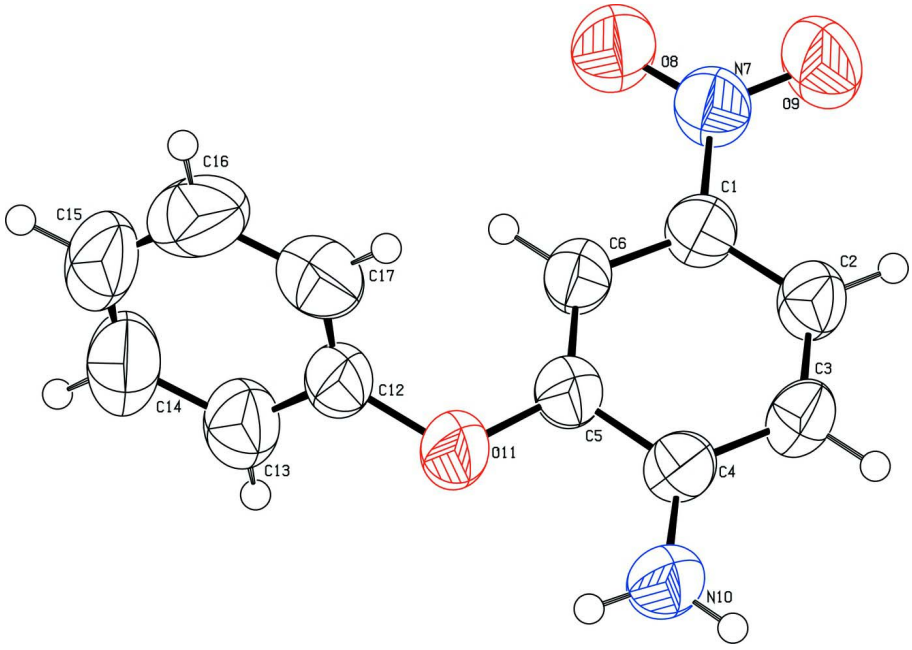

Figure 1

A view of (I), with 50% probability displacement ellipsoids.

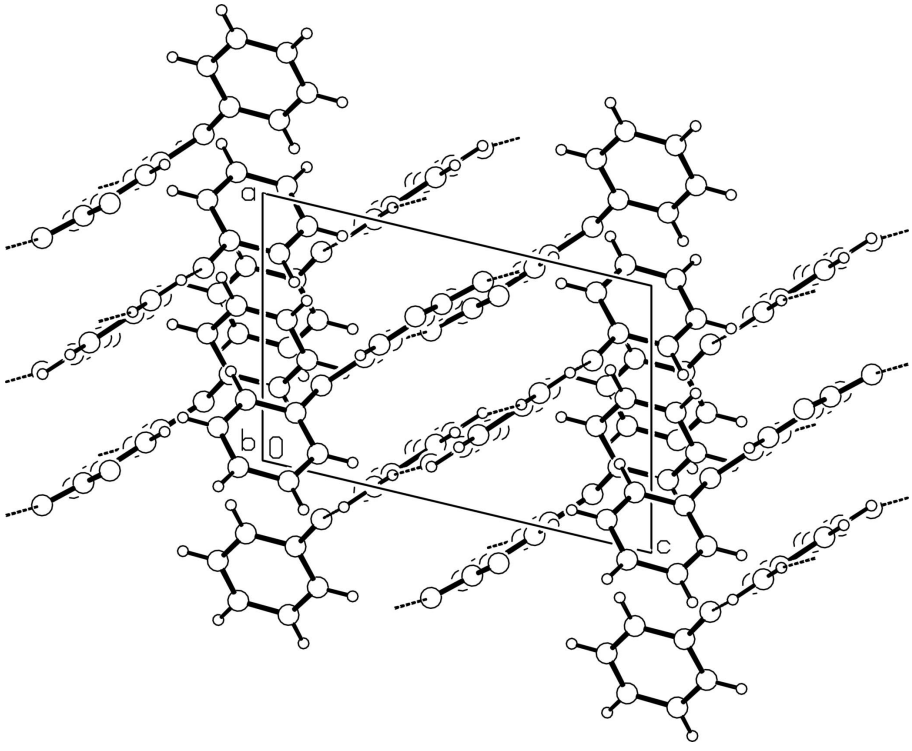

Figure 2

Packing diagram of the molecule viewed down the 'b′ axis. The dotted lines represents the hydrogen bonds.

4-Nitro-2-phenoxyaniline

Crystal data

$C_{12}H_{10}N_2O_3$
$M_r = 230.22$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.4100$ (12) Å
$b = 15.6570$ (18) Å
$c = 6.9600$ (17) Å
$\beta = 103.406$ (4)°
$V = 1103.5$ (3) Å^3
$Z = 4$
$F(000) = 480$
$D_x = 1.386$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 14613 reflections
$\theta = 2.4$–$32.5°$
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, colorless
0.32 × 0.3 × 0.25 mm

Data collection

MacScience DIPLabo 32001 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
ω scan
3336 measured reflections
1889 independent reflections
1498 reflections with $I > 2\sigma(I)$
$R_{int} = 0.033$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.4°$
$h = -12 \rightarrow 12$
$k = -18 \rightarrow 18$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.167$
$S = 1.09$
1889 reflections
154 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0811P)^2 + 0.2121P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.13$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ 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. 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
C1	0.4153 (2)	0.11783 (12)	0.1849 (3)	0.0575 (5)
C2	0.4872 (2)	0.04830 (14)	0.2715 (3)	0.0624 (5)
H2	0.5641	0.0561	0.3692	0.075*
C3	0.4446 (2)	−0.03248 (13)	0.2129 (3)	0.0618 (5)
H3	0.4920	−0.0794	0.2737	0.074*

C4	0.3320 (2)	−0.04544 (12)	0.0647 (3)	0.0578 (5)
C5	0.2615 (2)	0.02705 (13)	−0.0233 (3)	0.0637 (6)
C6	0.3010 (2)	0.10777 (13)	0.0368 (3)	0.0643 (6)
H6	0.2527	0.1550	−0.0200	0.077*
N7	0.46086 (19)	0.20258 (12)	0.2446 (3)	0.0696 (5)
O8	0.39280 (19)	0.26405 (10)	0.1748 (3)	0.0929 (6)
O9	0.56703 (18)	0.21113 (11)	0.3643 (3)	0.0956 (6)
N10	0.28775 (19)	−0.12433 (11)	0.0016 (3)	0.0742 (6)
H10A	0.3300	−0.1689	0.0542	0.089*
H10B	0.2174	−0.1298	−0.0909	0.089*
O11	0.15146 (19)	0.00647 (10)	−0.1675 (3)	0.0991 (7)
C12	0.0845 (2)	0.06917 (13)	−0.2929 (3)	0.0710 (6)
C13	−0.0425 (2)	0.08508 (17)	−0.2880 (4)	0.0802 (7)
H13	−0.0804	0.0568	−0.1974	0.096*
C14	−0.1149 (3)	0.1424 (2)	−0.4153 (5)	0.0973 (9)
H14	−0.2022	0.1528	−0.4110	0.117*
C15	−0.0624 (4)	0.18388 (18)	−0.5465 (5)	0.1026 (10)
H15	−0.1127	0.2234	−0.6318	0.123*
C16	0.0659 (4)	0.16812 (19)	−0.5551 (4)	0.1079 (11)
H16	0.1024	0.1967	−0.6469	0.129*
C17	0.1418 (3)	0.10918 (18)	−0.4262 (5)	0.0934 (8)
H17	0.2287	0.0976	−0.4309	0.112*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0635 (11)	0.0525 (10)	0.0538 (11)	0.0002 (9)	0.0079 (9)	−0.0054 (8)
C2	0.0669 (12)	0.0677 (13)	0.0474 (10)	0.0017 (10)	0.0026 (9)	0.0018 (9)
C3	0.0713 (13)	0.0586 (11)	0.0523 (11)	0.0093 (9)	0.0079 (9)	0.0073 (9)
C4	0.0647 (12)	0.0528 (11)	0.0553 (11)	0.0008 (9)	0.0125 (9)	0.0022 (8)
C5	0.0613 (12)	0.0559 (11)	0.0657 (12)	−0.0024 (9)	−0.0018 (10)	0.0025 (9)
C6	0.0619 (12)	0.0545 (11)	0.0690 (13)	0.0044 (9)	−0.0003 (10)	0.0015 (9)
N7	0.0731 (11)	0.0609 (11)	0.0687 (11)	0.0001 (9)	0.0042 (9)	−0.0105 (9)
O8	0.0977 (12)	0.0569 (9)	0.1100 (14)	0.0061 (9)	−0.0044 (10)	−0.0108 (9)
O9	0.0884 (12)	0.0789 (11)	0.0993 (13)	−0.0071 (9)	−0.0195 (10)	−0.0197 (10)
N10	0.0811 (12)	0.0523 (10)	0.0814 (13)	−0.0015 (8)	0.0031 (10)	0.0031 (9)
O11	0.0898 (12)	0.0568 (9)	0.1187 (15)	−0.0097 (8)	−0.0408 (11)	0.0116 (9)
C12	0.0720 (14)	0.0539 (11)	0.0716 (14)	−0.0052 (10)	−0.0151 (11)	−0.0015 (10)
C13	0.0787 (15)	0.0843 (16)	0.0693 (14)	0.0030 (13)	0.0001 (11)	−0.0023 (12)
C14	0.0888 (18)	0.0959 (19)	0.0906 (19)	0.0195 (15)	−0.0131 (15)	−0.0042 (16)
C15	0.119 (2)	0.0790 (17)	0.0801 (18)	0.0069 (17)	−0.0377 (17)	−0.0022 (15)
C16	0.148 (3)	0.088 (2)	0.0770 (18)	−0.031 (2)	0.0041 (19)	0.0114 (15)
C17	0.0778 (16)	0.0823 (17)	0.113 (2)	−0.0124 (13)	0.0075 (15)	−0.0026 (16)

Geometric parameters (Å, º)

C1—C2	1.378 (3)	N10—H10A	0.8600
C1—C6	1.390 (3)	N10—H10B	0.8600

C1—N7	1.438 (3)	O11—C12	1.389 (3)
C2—C3	1.371 (3)	C12—C13	1.353 (4)
C2—H2	0.9300	C12—C17	1.367 (4)
C3—C4	1.385 (3)	C13—C14	1.359 (4)
C3—H3	0.9300	C13—H13	0.9300
C4—N10	1.355 (3)	C14—C15	1.337 (5)
C4—C5	1.412 (3)	C14—H14	0.9300
C5—C6	1.365 (3)	C15—C16	1.374 (5)
C5—O11	1.375 (3)	C15—H15	0.9300
C6—H6	0.9300	C16—C17	1.397 (4)
N7—O8	1.227 (2)	C16—H16	0.9300
N7—O9	1.227 (2)	C17—H17	0.9300

C2—C1—C6	121.29 (18)	C4—N10—H10B	120.0
C2—C1—N7	119.55 (18)	H10A—N10—H10B	120.0
C6—C1—N7	119.14 (18)	C5—O11—C12	120.32 (16)
C3—C2—C1	119.54 (19)	C13—C12—C17	121.1 (2)
C3—C2—H2	120.2	C13—C12—O11	117.8 (2)
C1—C2—H2	120.2	C17—C12—O11	121.0 (2)
C2—C3—C4	121.10 (18)	C12—C13—C14	120.2 (3)
C2—C3—H3	119.4	C12—C13—H13	119.9
C4—C3—H3	119.4	C14—C13—H13	119.9
N10—C4—C3	122.70 (19)	C15—C14—C13	120.9 (3)
N10—C4—C5	119.24 (19)	C15—C14—H14	119.6
C3—C4—C5	118.06 (18)	C13—C14—H14	119.6
C6—C5—O11	125.60 (19)	C14—C15—C16	119.9 (3)
C6—C5—C4	121.45 (19)	C14—C15—H15	120.1
O11—C5—C4	112.93 (18)	C16—C15—H15	120.1
C5—C6—C1	118.53 (19)	C15—C16—C17	120.1 (3)
C5—C6—H6	120.7	C15—C16—H16	119.9
C1—C6—H6	120.7	C17—C16—H16	119.9
O8—N7—O9	121.99 (19)	C12—C17—C16	117.9 (3)
O8—N7—C1	119.21 (17)	C12—C17—H17	121.1
O9—N7—C1	118.80 (18)	C16—C17—H17	121.1
C4—N10—H10A	120.0		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H10*A*···O9^{i}	0.86	2.17	3.023 (3)	170

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