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2-(4-Methylphenoxy)-5-nitropyridine

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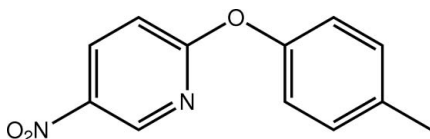
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.047; wR factor = 0.147; data-to-parameter ratio = 12.5.

The title molecule, $\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$, is twisted, the dihedral angle between the rings being $61.16(13)^\circ$. The nitro group is approximately coplanar with the pyridine ring to which it is attached [$\text{O}-\text{N}-\text{C}-\text{C}$ torsion angle = $-178.1(3)^\circ$]. Supramolecular chains along $[010]$ and mediated by $\text{C}-\text{H}\cdots\text{O}$ and $\pi-\pi$ [centroid(pyridyl)-(benzene) distance = $3.8259(18)$ Å] contacts feature in the crystal packing.

Related literature

For the structure of a related nitro-pyridine derivative, see: Nasir *et al.* (2010).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{10}\text{N}_2\text{O}_3$
 $M_r = 230.22$
 Orthorhombic, $Pbca$

$a = 7.2818(18)$ Å
 $b = 11.977(2)$ Å
 $c = 25.362(5)$ Å

$V = 2211.9(8)$ Å³
 $Z = 8$
 Mo $K\alpha$ radiation

$\mu = 0.10$ mm⁻¹
 $T = 293$ K
 $0.20 \times 0.18 \times 0.07$ mm

Data collection

Bruker SMART APEX diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.670$, $T_{\max} = 0.746$

15887 measured reflections
 1951 independent reflections
 1089 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.074$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.147$
 $S = 1.02$
 1951 reflections

156 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.15$ e Å⁻³
 $\Delta\rho_{\min} = -0.13$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C5}-\text{H5}\cdots\text{O}(3)^i$	0.93	2.43	3.135(3)	132

Symmetry code: (i) $-x + \frac{3}{2}, y - \frac{1}{2}, z$.

Data collection: APEX2 (Bruker, 2009); cell refinement: SAINT (Bruker, 2009); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 2006); software used to prepare material for publication: publCIF (Westrip, 2010).

We thank the University of Malaya (grant No. RG027/09AFR) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5116).

References

- Brandenburg, K. (2006). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
 Bruker (2009). *APEX2* and *SAINTE*. Bruker AXS Inc., Madison, Wisconsin, USA.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Nasir, S. B., Abdullah, Z., Fairuz, Z. A., Ng, S. W. & Tiekink, E. R. T. (2010). *Acta Cryst.* **E66**, o2428.
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
 Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

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supplementary materials

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2-(4-Methylphenoxy)-5-nitropyridine

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Comment

The synthesis and crystal structure determination of the title compound, (I), was determined in connection with studies of related species (Nasir *et al.*, 2010). In (I), the dihedral angle formed between the pyridyl and benzene rings is 61.16 (13)°, indicating significant twisting in the molecule. The nitro group is co-planar with the pyridyl ring to which it is connected as seen in the value of the O2—N2—C4—C3 torsion angle of -178.1 (3)°.

The most prominent features in the crystal packing are the formation of C—H···O, Table 1, and π – π interactions. The latter occur between the pyridyl and benzene rings with the separation between the ring centroids being 3.8259 (18) Å for symmetry operation $3/2 - x, 1/2 + y, z$. These interactions lead to supramolecular chains along the *b* axis, Fig. 2, which pack as shown in Fig. 3.

Experimental

p-Cresol (2.16 g, 20 mmol) and sodium hydroxide (0.80 g, 20 mmol) were dissolved in water (50 ml) and to the solution was added 2-chloro-5-nitropyridine (3.17 g, 20 mmol) dissolved in THF (50 ml). The mixture was heated for 5 h. Water was added and the organic phase extracted with chloroform. The chloroform solution was dried over sodium sulfate; slow evaporation led to the formation of colourless crystals.

Refinement

Hydrogen atoms were placed at calculated positions (C—H 0.93–0.96 Å) and were treated as riding on their parent carbon atoms, with $U(\text{H})$ set to 1.2–1.5 $U_{\text{eq}}(\text{C})$.

Figures

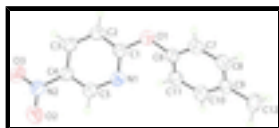


Fig. 1. The molecular structure of (I) showing the atom-labelling scheme and displacement ellipsoids at the 35% probability level.

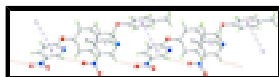


Fig. 2. Supramolecular chain along [010] in (I) sustained by C—H···O and π – π interactions, shown as orange and purple dashed lines, respectively.

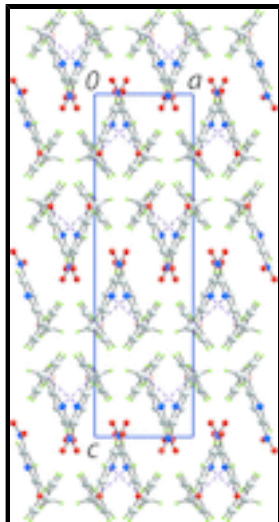


Fig. 3. Unit-cell contents for (I) shown in projection down the *b* axis highlighting the packing of supramolecular chains.

2-(4-Methylphenoxy)-5-nitropyridine

Crystal data

$C_{12}H_{10}N_2O_3$

$M_r = 230.22$

Orthorhombic, *Pbca*

Hall symbol: -P 2ac 2ab

$a = 7.2818 (18) \text{ \AA}$

$b = 11.977 (2) \text{ \AA}$

$c = 25.362 (5) \text{ \AA}$

$V = 2211.9 (8) \text{ \AA}^3$

$Z = 8$

$F(000) = 960$

$D_x = 1.383 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 1038 reflections

$\theta = 3.2\text{--}19.7^\circ$

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

$T = 293 \text{ K}$

Block, colourless

$0.20 \times 0.18 \times 0.07 \text{ mm}$

Data collection

Bruker SMART APEX
diffractometer

Radiation source: fine-focus sealed tube
graphite

ω scans

Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.670$, $T_{\max} = 0.746$

15887 measured reflections

1951 independent reflections

1089 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.074$

$\theta_{\max} = 25.0^\circ$, $\theta_{\min} = 1.6^\circ$

$h = -8 \rightarrow 8$

$k = -14 \rightarrow 14$

$l = -30 \rightarrow 30$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.047$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$wR(F^2) = 0.147$	$w = 1/[\sigma^2(F_o^2) + (0.0643P)^2 + 0.2807P]$
$S = 1.02$	where $P = (F_o^2 + 2F_c^2)/3$
1951 reflections	$(\Delta/\sigma)_{\max} = 0.001$
156 parameters	$\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$
0 restraints	$\Delta\rho_{\min} = -0.13 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: <i>SHELXL97</i> (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ Extinction coefficient: 0.0060 (13)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.5542 (3)	0.41426 (14)	0.32750 (7)	0.0725 (6)
O2	0.8217 (3)	0.59307 (19)	0.54024 (9)	0.1039 (8)
O3	0.7135 (4)	0.74378 (19)	0.50863 (8)	0.1135 (9)
N1	0.6779 (3)	0.41528 (17)	0.41124 (9)	0.0699 (7)
N2	0.7486 (4)	0.6456 (2)	0.50490 (10)	0.0758 (7)
C1	0.6005 (4)	0.4707 (2)	0.37191 (11)	0.0595 (7)
C2	0.5682 (3)	0.5841 (2)	0.37215 (10)	0.0613 (7)
H2	0.5145	0.6191	0.3433	0.074*
C3	0.6168 (4)	0.6439 (2)	0.41563 (10)	0.0648 (7)
H3	0.5978	0.7206	0.4173	0.078*
C4	0.6950 (4)	0.5868 (2)	0.45701 (10)	0.0576 (7)
C5	0.7247 (4)	0.4751 (2)	0.45359 (11)	0.0675 (8)
H5	0.7798	0.4389	0.4819	0.081*
C6	0.5381 (4)	0.2976 (2)	0.32825 (10)	0.0588 (7)
C7	0.6205 (4)	0.2394 (2)	0.28825 (9)	0.0625 (7)
H7	0.6943	0.2759	0.2638	0.075*
C8	0.5920 (4)	0.1252 (2)	0.28494 (10)	0.0639 (7)
H8	0.6459	0.0857	0.2574	0.077*
C9	0.4864 (4)	0.0685 (2)	0.32118 (10)	0.0601 (7)
C10	0.4088 (4)	0.1301 (2)	0.36136 (11)	0.0675 (8)
H10	0.3393	0.0936	0.3868	0.081*
C11	0.4313 (4)	0.2442 (2)	0.36485 (10)	0.0685 (8)
H11	0.3747	0.2844	0.3917	0.082*
C12	0.4559 (4)	-0.0556 (2)	0.31771 (12)	0.0854 (9)
H12A	0.4765	-0.0888	0.3517	0.128*
H12B	0.5397	-0.0871	0.2926	0.128*
H12C	0.3320	-0.0700	0.3067	0.128*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0976 (16)	0.0589 (12)	0.0612 (11)	0.0000 (10)	-0.0111 (11)	0.0016 (9)
O2	0.142 (2)	0.0885 (16)	0.0806 (14)	0.0159 (14)	-0.0259 (15)	-0.0119 (13)
O3	0.210 (3)	0.0523 (12)	0.0784 (14)	-0.0020 (15)	0.0069 (16)	-0.0057 (11)

supplementary materials

N1	0.0881 (17)	0.0516 (13)	0.0699 (14)	0.0073 (11)	-0.0110 (14)	-0.0033 (12)
N2	0.104 (2)	0.0597 (16)	0.0639 (15)	-0.0023 (14)	0.0124 (14)	-0.0004 (13)
C1	0.0622 (17)	0.0557 (16)	0.0607 (16)	0.0015 (13)	0.0020 (14)	0.0036 (14)
C2	0.0640 (18)	0.0586 (17)	0.0615 (16)	0.0076 (13)	0.0040 (14)	0.0104 (14)
C3	0.0754 (19)	0.0496 (15)	0.0695 (17)	0.0059 (14)	0.0120 (16)	0.0042 (14)
C4	0.0645 (17)	0.0508 (16)	0.0574 (15)	-0.0002 (13)	0.0085 (14)	-0.0002 (13)
C5	0.081 (2)	0.0566 (17)	0.0650 (17)	0.0056 (15)	-0.0076 (15)	0.0019 (14)
C6	0.0642 (18)	0.0568 (17)	0.0555 (16)	0.0006 (13)	-0.0087 (14)	-0.0002 (13)
C7	0.0632 (17)	0.0729 (18)	0.0513 (15)	-0.0010 (14)	-0.0007 (13)	0.0029 (14)
C8	0.0646 (17)	0.0730 (19)	0.0541 (15)	0.0055 (15)	-0.0050 (14)	-0.0100 (14)
C9	0.0536 (16)	0.0654 (18)	0.0614 (16)	-0.0024 (14)	-0.0105 (14)	-0.0089 (14)
C10	0.0596 (17)	0.075 (2)	0.0683 (18)	-0.0082 (14)	0.0032 (15)	0.0007 (15)
C11	0.0697 (19)	0.0705 (19)	0.0651 (17)	0.0041 (15)	0.0042 (15)	-0.0086 (15)
C12	0.088 (2)	0.071 (2)	0.098 (2)	-0.0109 (16)	-0.0140 (19)	-0.0137 (17)

Geometric parameters (Å, °)

O1—C1	1.356 (3)	C6—C11	1.369 (3)
O1—C6	1.403 (3)	C6—C7	1.369 (3)
O2—N2	1.217 (3)	C7—C8	1.386 (4)
O3—N2	1.208 (3)	C7—H7	0.9300
N1—C1	1.324 (3)	C8—C9	1.377 (3)
N1—C5	1.336 (3)	C8—H8	0.9300
N2—C4	1.457 (3)	C9—C10	1.379 (3)
C1—C2	1.379 (3)	C9—C12	1.505 (3)
C2—C3	1.361 (3)	C10—C11	1.380 (3)
C2—H2	0.9300	C10—H10	0.9300
C3—C4	1.376 (3)	C11—H11	0.9300
C3—H3	0.9300	C12—H12A	0.9600
C4—C5	1.358 (3)	C12—H12B	0.9600
C5—H5	0.9300	C12—H12C	0.9600
C1—O1—C6	120.4 (2)	C7—C6—O1	117.4 (2)
C1—N1—C5	116.4 (2)	C6—C7—C8	118.8 (3)
O3—N2—O2	122.5 (3)	C6—C7—H7	120.6
O3—N2—C4	118.6 (3)	C8—C7—H7	120.6
O2—N2—C4	118.8 (2)	C9—C8—C7	122.0 (2)
N1—C1—O1	118.8 (2)	C9—C8—H8	119.0
N1—C1—C2	124.3 (3)	C7—C8—H8	119.0
O1—C1—C2	116.9 (2)	C8—C9—C10	117.3 (2)
C3—C2—C1	118.5 (2)	C8—C9—C12	122.0 (2)
C3—C2—H2	120.7	C10—C9—C12	120.7 (3)
C1—C2—H2	120.7	C9—C10—C11	121.9 (3)
C2—C3—C4	117.7 (2)	C9—C10—H10	119.0
C2—C3—H3	121.2	C11—C10—H10	119.0
C4—C3—H3	121.2	C6—C11—C10	119.1 (3)
C5—C4—C3	120.4 (3)	C6—C11—H11	120.4
C5—C4—N2	119.1 (3)	C10—C11—H11	120.4
C3—C4—N2	120.5 (2)	C9—C12—H12A	109.5
N1—C5—C4	122.7 (3)	C9—C12—H12B	109.5

N1—C5—H5	118.7	H12A—C12—H12B	109.5
C4—C5—H5	118.7	C9—C12—H12C	109.5
C11—C6—C7	120.9 (2)	H12A—C12—H12C	109.5
C11—C6—O1	121.5 (2)	H12B—C12—H12C	109.5
C5—N1—C1—O1	178.5 (2)	C3—C4—C5—N1	-1.2 (4)
C5—N1—C1—C2	0.7 (4)	N2—C4—C5—N1	179.4 (2)
C6—O1—C1—N1	18.3 (4)	C1—O1—C6—C11	52.0 (3)
C6—O1—C1—C2	-163.8 (2)	C1—O1—C6—C7	-133.5 (2)
N1—C1—C2—C3	-0.7 (4)	C11—C6—C7—C8	0.9 (4)
O1—C1—C2—C3	-178.6 (2)	O1—C6—C7—C8	-173.7 (2)
C1—C2—C3—C4	-0.2 (4)	C6—C7—C8—C9	-1.2 (4)
C2—C3—C4—C5	1.1 (4)	C7—C8—C9—C10	0.0 (4)
C2—C3—C4—N2	-179.4 (2)	C7—C8—C9—C12	-179.8 (2)
O3—N2—C4—C5	-176.4 (3)	C8—C9—C10—C11	1.6 (4)
O2—N2—C4—C5	1.3 (4)	C12—C9—C10—C11	-178.6 (3)
O3—N2—C4—C3	4.1 (4)	C7—C6—C11—C10	0.7 (4)
O2—N2—C4—C3	-178.1 (3)	O1—C6—C11—C10	175.0 (2)
C1—N1—C5—C4	0.2 (4)	C9—C10—C11—C6	-1.9 (4)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C5-H5\cdots O(3)^i$	0.93	2.43	3.135 (3)	132

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

Fig. 1

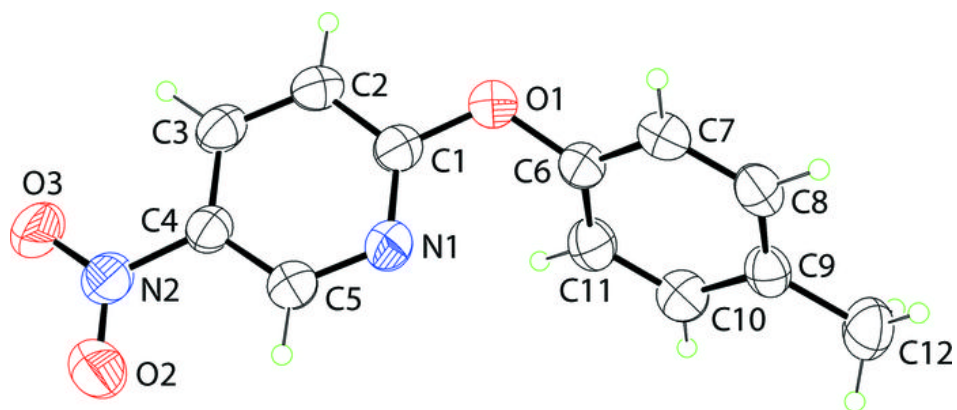


Fig. 2

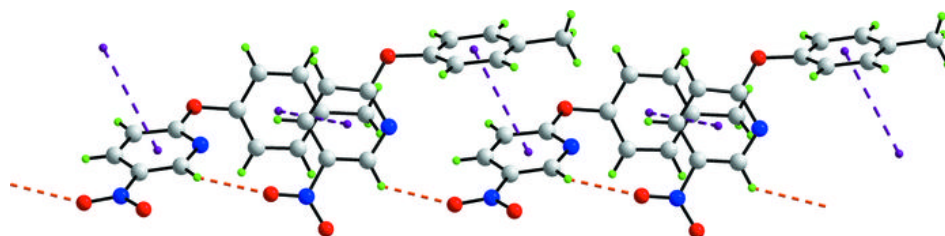


Fig. 3

