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N-[Methoxy(4-nitrophenyl)methyl]-pyridin-2-amine

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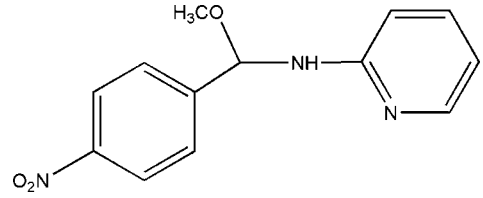
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.050; wR factor = 0.131; data-to-parameter ratio = 11.7.

In the title compound, $C_{13}H_{13}N_3O_3$, the nitro group is twisted away from the attached benzene ring by 19.5 (3)°. The dihedral angle between the benzene and pyridine rings is 78.2 (1)°. In the crystal structure, molecules are connected into chains along the [001] direction by N—H···N hydrogen bonds.

Related literature

For related literature, see: Boedeker & Courault (1980); Che & Wang (2007); Habibi *et al.* (2007); Hodnett & Dunn (1970); Panneerselvam *et al.* (2005).



Experimental

Crystal data

$C_{13}H_{13}N_3O_3$
M_r = 259.26
Monoclinic, $P2_1/c$
a = 10.661 (2) Å
b = 14.153 (3) Å
c = 9.0410 (18) Å
β = 110.70 (3)°
V = 1276.1 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.32 × 0.26 × 0.22 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1998)
T_{min} = 0.969, T_{max} = 0.979
2666 measured reflections
2500 independent reflections
1642 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.131
S = 1.01
2500 reflections
214 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2B···N3^{i}	0.87 (2)	2.13 (2)	2.976 (3)	167 (2)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); 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: CI2453).

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supplementary materials

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N-[Methoxy(4-nitrophenyl)methyl]pyridin-2-amine

R.-Q. Fang, H.-Q. Li, L. Shi, Z.-P. Xiao and H.-L. Zhu

Comment

There has been much research interest in the chemistry and biology of Schiff base compounds due to their facile synthesis and wide applications (Che *et al.*, 2007; Habibi *et al.*, 2007; Hodnett *et al.*, 1970; Panneerselvam *et al.*, 2005). To our knowledge, however, the addition products of Schiff bases are relatively less reported. In this paper, we report the synthesis and crystal structure of *N*-(methoxy(4-nitrophenyl)methyl)pyridin-2-amine (Boedeker *et al.*, 1980), which is a addition product of methyl 4-nitro-N-(pyridin-2-yl)benzimidate with methanol.

In the title molecule (Fig.1), the bond distances and angles are normal. The N3—C9—N2—C7, O3—C7—N2—C9 and N2—C7—O3—C8 torsion angles are −11.0 (3), −73.4 (3) and 165.83 (19)°, respectively. The dihedral angle between the nitryl plane and benzene ring is 19.5 (3)°, and that between the benzene and pyridine rings is 78.2 (1)°.

Intermolecular N2—H2B···N3 hydrogen bonds connect the molecules into chains along the *c* axis (Fig. 2).

Experimental

Equimolar quantities (0.5 mmol) of 4-nitrobenzaldehyde, 2-pyridinamine and $NiCl_2$ were dissolved in methanol (10 ml) and stirred at room temperature for several hours. The resulting precipitate was separated by filtration and recrystallized from methanol. Single crystals suitable for X-ray diffraction studies were obtained after 5 d by slow evaporation of an ethanol solution. Analysis found: C 60.21, H 5.08, N 16.19%; calculated for $C_{13}H_{13}N_3O_3$: C 60.20, H 5.05, N 16.21%.

Refinement

Methyl H atoms were placed in idealized positions (C—H = 0.96 Å), and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C). The remaining H atoms were located in a difference map and refined isotropically [N—H = 0.87 (2) Å and C—H = 0.93 (3)–0.99 (2) Å].

Figures

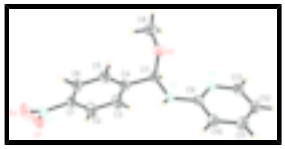


Fig. 1. The molecular structure of the title compound, showing 35% probability displacement ellipsoids (arbitrary spheres for the H atoms).

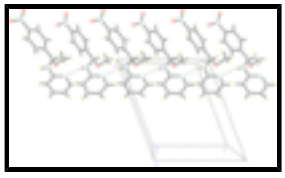
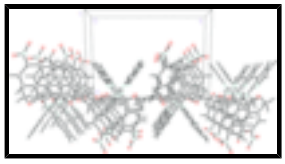


Fig. 2. A view of an N—H···N hydrogen-bonded (dashed lines) chain in the title compound.



_N_-[Methoxy(4-nitrophenyl)methyl]pyridin-2-amine

Crystal data

$C_{13}H_{13}N_3O_3$	F_{000} = 544
M_r = 259.26	D_x = 1.349 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 1247 reflections
a = 10.661 (2) Å	θ = 2.8–24.3º
b = 14.153 (3) Å	μ = 0.10 mm^{-1}
c = 9.0410 (18) Å	T = 293 (2) K
β = 110.70 (3)º	Block, yellow
V = 1276.1 (5) Å^3	0.32 × 0.26 × 0.22 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2500 independent reflections
Radiation source: fine-focus sealed tube	1642 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.026
T = 293(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 1998)	h = −13→12
T_{min} = 0.969, T_{max} = 0.979	k = −17→0
2666 measured reflections	l = 0→11

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.050	$w = 1/[\sigma^2(F_o^2) + (0.0583P)^2 + 0.2385P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.131	$(\Delta/\sigma)_{max}$ = 0.001
S = 1.01	$\Delta\rho_{max}$ = 0.21 e Å^{-3}
2500 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
214 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Extinction coefficient: 0.035 (4)

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.3678 (2)	0.36268 (17)	0.0210 (3)	0.0376 (6)
C2	0.3718 (3)	0.2834 (2)	0.1096 (3)	0.0531 (7)
C3	0.2638 (2)	0.2654 (2)	0.1565 (3)	0.0508 (7)
C4	0.1559 (2)	0.32650 (16)	0.1176 (2)	0.0342 (5)
C5	0.1559 (2)	0.40548 (17)	0.0287 (3)	0.0435 (6)
C6	0.2621 (3)	0.42391 (18)	−0.0214 (3)	0.0433 (6)
C7	0.0357 (2)	0.30320 (17)	0.1656 (3)	0.0362 (5)
C8	0.0389 (3)	0.4362 (2)	0.3262 (3)	0.0673 (9)
H8A	0.0500	0.3967	0.4163	0.101*
H8B	−0.0103	0.4919	0.3325	0.101*
H8C	0.1254	0.4540	0.3248	0.101*
C9	−0.1545 (2)	0.19259 (15)	0.0689 (2)	0.0324 (5)
C10	−0.2478 (3)	0.14749 (18)	−0.0610 (3)	0.0447 (6)
C11	−0.3474 (3)	0.0958 (2)	−0.0389 (3)	0.0564 (7)
C12	−0.3525 (3)	0.0876 (2)	0.1113 (3)	0.0541 (7)
C13	−0.2556 (3)	0.13170 (18)	0.2326 (3)	0.0451 (6)
N1	0.4801 (2)	0.38099 (16)	−0.0347 (2)	0.0476 (5)
N2	−0.05330 (18)	0.24491 (14)	0.0468 (2)	0.0369 (5)
N3	−0.15594 (18)	0.18427 (13)	0.2155 (2)	0.0356 (5)
O1	0.58639 (19)	0.34165 (17)	0.0330 (3)	0.0787 (7)
O2	0.46018 (19)	0.43389 (14)	−0.1466 (2)	0.0627 (6)
O3	−0.03278 (16)	0.38568 (12)	0.1852 (2)	0.0482 (5)
H7	0.068 (2)	0.2671 (15)	0.266 (3)	0.034 (6)*
H10	−0.243 (2)	0.1564 (16)	−0.163 (3)	0.044 (7)*
H2B	−0.070 (2)	0.2629 (16)	−0.050 (3)	0.039 (6)*
H3	0.264 (3)	0.2062 (19)	0.215 (3)	0.064 (8)*
H6	0.264 (2)	0.4794 (18)	−0.076 (3)	0.048 (7)*
H13	−0.252 (2)	0.1257 (17)	0.340 (3)	0.052 (7)*
H5	0.083 (3)	0.4520 (19)	0.002 (3)	0.059 (7)*
H2A	0.445 (3)	0.241 (2)	0.134 (3)	0.065 (8)*

H12	−0.414 (3)	0.0485 (18)	0.136 (3)	0.058 (8)*
H11	−0.416 (3)	0.068 (2)	−0.128 (3)	0.065 (8)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0312 (12)	0.0492 (14)	0.0359 (12)	−0.0057 (11)	0.0162 (10)	−0.0033 (11)
C2	0.0367 (14)	0.0679 (18)	0.0582 (17)	0.0125 (13)	0.0209 (13)	0.0209 (14)
C3	0.0428 (14)	0.0571 (17)	0.0552 (16)	0.0074 (13)	0.0208 (12)	0.0252 (14)
C4	0.0319 (12)	0.0423 (13)	0.0304 (11)	−0.0046 (10)	0.0136 (9)	−0.0001 (10)
C5	0.0421 (14)	0.0415 (14)	0.0549 (15)	0.0058 (12)	0.0269 (12)	0.0051 (12)
C6	0.0481 (15)	0.0390 (14)	0.0527 (15)	−0.0023 (12)	0.0300 (12)	0.0075 (12)
C7	0.0344 (12)	0.0442 (13)	0.0325 (12)	−0.0034 (10)	0.0151 (10)	0.0004 (11)
C8	0.093 (2)	0.0586 (18)	0.0662 (18)	−0.0221 (17)	0.0483 (17)	−0.0214 (15)
C9	0.0317 (11)	0.0351 (12)	0.0354 (12)	0.0009 (10)	0.0179 (10)	−0.0010 (10)
C10	0.0496 (15)	0.0520 (16)	0.0355 (13)	−0.0111 (12)	0.0187 (11)	−0.0040 (12)
C11	0.0526 (16)	0.0654 (18)	0.0520 (16)	−0.0245 (15)	0.0195 (14)	−0.0117 (14)
C12	0.0523 (16)	0.0569 (17)	0.0637 (18)	−0.0216 (14)	0.0335 (14)	−0.0058 (14)
C13	0.0521 (15)	0.0475 (15)	0.0482 (15)	−0.0039 (12)	0.0331 (13)	0.0000 (12)
N1	0.0423 (12)	0.0576 (13)	0.0507 (13)	−0.0080 (11)	0.0261 (11)	−0.0066 (11)
N2	0.0385 (11)	0.0483 (12)	0.0281 (10)	−0.0092 (9)	0.0168 (8)	−0.0003 (9)
N3	0.0375 (10)	0.0406 (11)	0.0343 (10)	−0.0017 (9)	0.0196 (8)	−0.0007 (8)
O1	0.0417 (11)	0.1060 (17)	0.0993 (17)	0.0114 (12)	0.0382 (11)	0.0213 (14)
O2	0.0643 (13)	0.0778 (13)	0.0610 (12)	−0.0083 (10)	0.0406 (10)	0.0097 (11)
O3	0.0494 (10)	0.0494 (10)	0.0541 (10)	−0.0037 (8)	0.0285 (9)	−0.0097 (8)

Geometric parameters (Å, °)

C1—C6	1.365 (3)	C8—H8B	0.96
C1—C2	1.371 (3)	C8—H8C	0.96
C1—N1	1.476 (3)	C9—N3	1.337 (3)
C2—C3	1.384 (4)	C9—N2	1.380 (3)
C2—H2A	0.94 (3)	C9—C10	1.396 (3)
C3—C4	1.381 (3)	C10—C11	1.361 (3)
C3—H3	0.99 (3)	C10—H10	0.95 (2)
C4—C5	1.377 (3)	C11—C12	1.383 (4)
C4—C7	1.528 (3)	C11—H11	0.96 (3)
C5—C6	1.385 (3)	C12—C13	1.363 (4)
C5—H5	0.98 (3)	C12—H12	0.94 (3)
C6—H6	0.93 (3)	C13—N3	1.350 (3)
C7—N2	1.419 (3)	C13—H13	0.96 (3)
C7—O3	1.421 (3)	N1—O2	1.215 (3)
C7—H7	0.99 (2)	N1—O1	1.216 (3)
C8—O3	1.426 (3)	N2—H2B	0.87 (2)
C8—H8A	0.96		
C6—C1—C2	122.4 (2)	O3—C8—H8C	109.5
C6—C1—N1	118.8 (2)	H8A—C8—H8C	109.5
C2—C1—N1	118.8 (2)	H8B—C8—H8C	109.5

C1—C2—C3	118.2 (2)	N3—C9—N2	118.30 (19)
C1—C2—H2A	120.3 (17)	N3—C9—C10	122.6 (2)
C3—C2—H2A	121.5 (17)	N2—C9—C10	119.01 (19)
C4—C3—C2	121.0 (2)	C11—C10—C9	118.9 (2)
C4—C3—H3	120.7 (15)	C11—C10—H10	121.9 (15)
C2—C3—H3	118.3 (15)	C9—C10—H10	119.1 (14)
C5—C4—C3	119.0 (2)	C10—C11—C12	119.5 (3)
C5—C4—C7	120.9 (2)	C10—C11—H11	120.0 (16)
C3—C4—C7	120.0 (2)	C12—C11—H11	120.4 (16)
C4—C5—C6	120.8 (2)	C13—C12—C11	118.0 (2)
C4—C5—H5	121.6 (15)	C13—C12—H12	118.2 (16)
C6—C5—H5	117.6 (15)	C11—C12—H12	123.5 (16)
C1—C6—C5	118.5 (2)	N3—C13—C12	124.3 (2)
C1—C6—H6	120.8 (15)	N3—C13—H13	114.4 (15)
C5—C6—H6	120.5 (15)	C12—C13—H13	121.3 (15)
N2—C7—O3	109.65 (18)	O2—N1—O1	124.0 (2)
N2—C7—C4	108.29 (17)	O2—N1—C1	117.9 (2)
O3—C7—C4	112.19 (18)	O1—N1—C1	118.2 (2)
N2—C7—H7	108.5 (12)	C9—N2—C7	123.40 (18)
O3—C7—H7	109.9 (12)	C9—N2—H2B	114.1 (15)
C4—C7—H7	108.2 (12)	C7—N2—H2B	116.0 (15)
O3—C8—H8A	109.5	C9—N3—C13	116.6 (2)
O3—C8—H8B	109.5	C7—O3—C8	112.76 (19)
H8A—C8—H8B	109.5		
C6—C1—C2—C3	0.3 (4)	C9—C10—C11—C12	−1.2 (4)
N1—C1—C2—C3	−177.8 (2)	C10—C11—C12—C13	−0.6 (4)
C1—C2—C3—C4	−1.1 (4)	C11—C12—C13—N3	1.2 (4)
C2—C3—C4—C5	1.0 (4)	C6—C1—N1—O2	−18.6 (3)
C2—C3—C4—C7	177.8 (2)	C2—C1—N1—O2	159.5 (2)
C3—C4—C5—C6	0.0 (4)	C6—C1—N1—O1	162.1 (2)
C7—C4—C5—C6	−176.7 (2)	C2—C1—N1—O1	−19.8 (3)
C2—C1—C6—C5	0.7 (4)	N3—C9—N2—C7	−11.0 (3)
N1—C1—C6—C5	178.8 (2)	C10—C9—N2—C7	171.5 (2)
C4—C5—C6—C1	−0.9 (4)	O3—C7—N2—C9	−73.4 (3)
C5—C4—C7—N2	90.5 (3)	C4—C7—N2—C9	163.9 (2)
C3—C4—C7—N2	−86.2 (3)	N2—C9—N3—C13	−179.29 (19)
C5—C4—C7—O3	−30.7 (3)	C10—C9—N3—C13	−1.9 (3)
C3—C4—C7—O3	152.6 (2)	C12—C13—N3—C9	0.0 (4)
N3—C9—C10—C11	2.5 (4)	N2—C7—O3—C8	165.83 (19)
N2—C9—C10—C11	179.9 (2)	C4—C7—O3—C8	−73.8 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2B···N3^{i}	0.87 (2)	2.13 (2)	2.976 (3)	167 (2)

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

Fig. 1

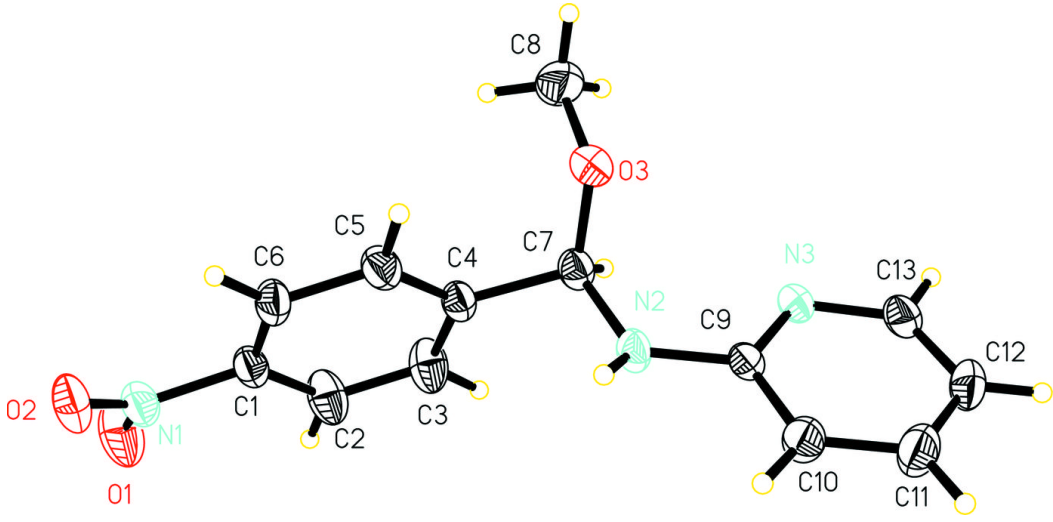


Fig. 2

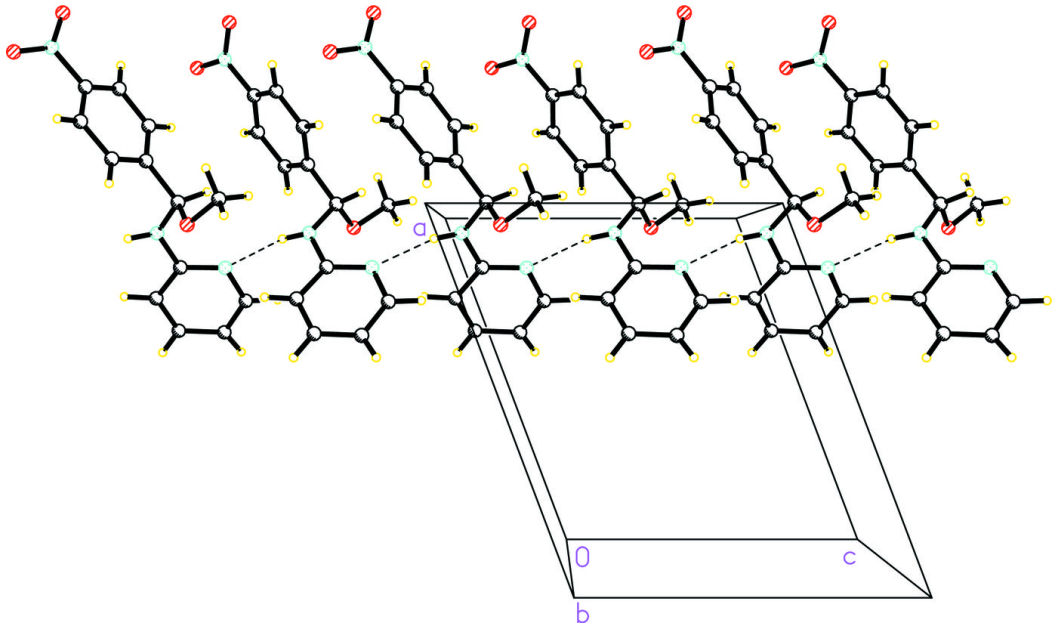


Fig. 3