Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Nitro-*N*^2^-(pyridin-4-ylmethylidene)-benzene-1,2-diamine

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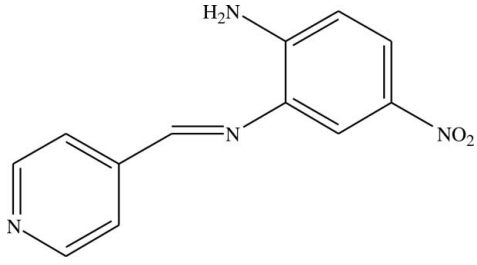
Received 20 May 2013; accepted 4 June 2013

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.058; wR factor = 0.175; data-to-parameter ratio = 12.6.

In the title compound, $C_{12}H_{10}N_4O_2$, the dihedral angle between the aromatic rings is 43.18 (16)°. The nitro group is rotated from its attached ring by 7.8 (2)° and a short intramolecular N—H···N contact occurs. In the crystal, the molecules are linked by N—H···N and C—H···O hydrogen bonds, generating a three-dimensional network.

Related literature

For the synthesis, see: Luo *et al.* (2009).



Experimental

Crystal data

$C_{12}H_{10}N_4O_2$
M_r = 242.24
Monoclinic, $C2/c$
a = 21.324 (4) Å
b = 9.1480 (18) Å
c = 12.950 (3) Å
β = 116.36 (3)°
V = 2263.5 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.970, T_{max} = 0.990
2128 measured reflections
2070 independent reflections
1247 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.175
S = 1.01
2070 reflections
164 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···N4^{i}	0.86	2.42	3.091 (3)	135
N2—H2B···N3	0.86	2.42	2.751 (3)	103
C10—H10A···O1^{ii}	0.93	2.49	3.156 (5)	128

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *PLATON* (Spek, 2009).

The authors thank the Center of Test and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB7085).

References

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supporting information

Acta Cryst. (2013). E**69**, o1073 [https://doi.org/10.1107/S1600536813015481]

4-Nitro-*N*^2^-(pyridin-4-ylmethylidene)benzene-1,2-diamine

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S1. Experimental

The title compound, (I), which may have applications as a metal fluoresence probe, was prepared by the literature method (Luo *et al.*, 2009). Yellow blocks were obtained by dissolving (I) (0.18 g, 1.0mmol) in ethanol (25 ml) and evaporating the solvent slowly at room temperature.

S2. Refinement

H atoms were positioned geometrically and refined as riding groups, with N—H = 0.86 and C—H = 0.93 Å for aromatic H, and constrained to ride on their parent atoms, with U_{iso}(H) = xU_{eq}(C), where x = 1.2 for aromatic H, and x = 1.5 for other H.

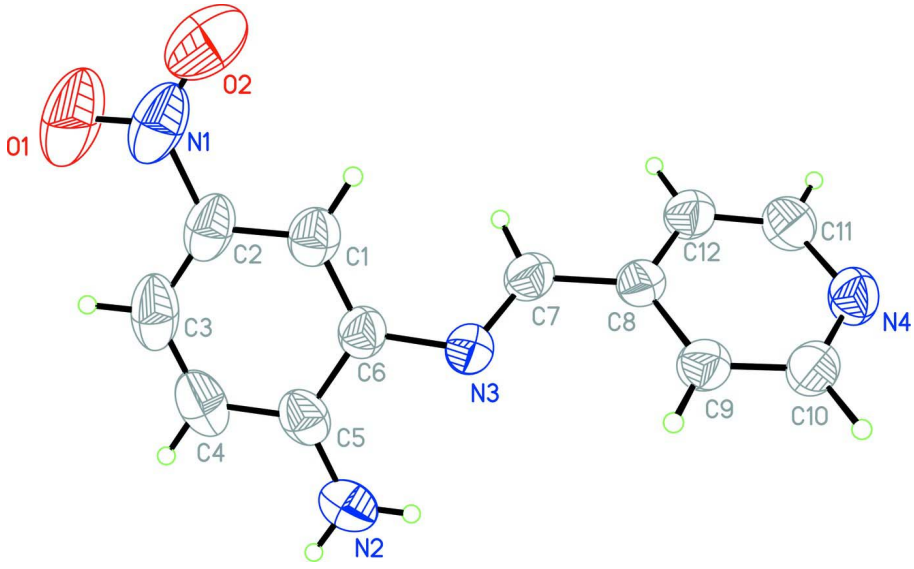

Figure 1

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

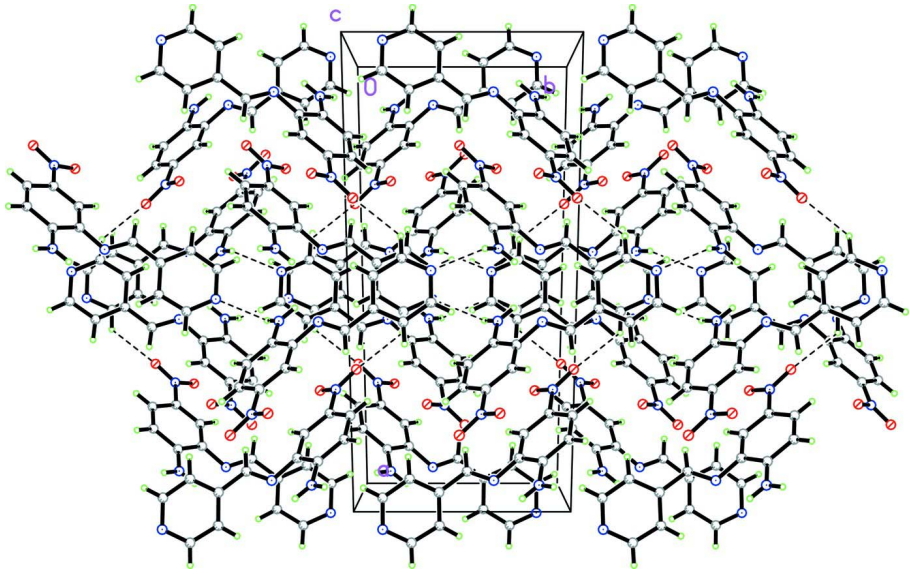

Figure 2

A packing diagram of the title molecule.

4-Nitro-*N*^{2}-(pyridin-4-ylmethylidene)benzene-1,2-diamine

Crystal data

$C_{12}H_{10}N_4O_2$
$M_r = 242.24$
Monoclinic, $C2/c$
$a = 21.324$ (4) Å
$b = 9.1480$ (18) Å
$c = 12.950$ (3) Å
$\beta = 116.36$ (3)°
$V = 2263.5$ (8) Å^3
$Z = 8$
$F(000) = 1008$
$D_x = 1.422$ Mg m^{-3}
Melting point: 449.65 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10$–13°
$\mu = 0.10$ mm^{-1}
$T = 293$ K
Block, yellow
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.970$, $T_{max} = 0.990$
2128 measured reflections
2070 independent reflections
1247 reflections with $I > 2\sigma(I)$
$R_{int} = 0.038$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.1°$
$h = 0 \rightarrow 25$
$k = 0 \rightarrow 11$
$l = -15 \rightarrow 14$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.175$
$S = 1.01$
2070 reflections
164 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
$w = 1/[\sigma^2(F_o^2) + (0.090P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.19$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}
Extinction correction: *SHELXL*,
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0052 (12)

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.32477 (14)	0.2795 (3)	−0.0542 (3)	0.0521 (8)
H1A	0.3229	0.2145	−0.1108	0.062*
N1	0.23039 (15)	0.4169 (4)	−0.2041 (3)	0.0792 (10)
O1	0.18599 (16)	0.5126 (4)	−0.2272 (3)	0.1194 (12)
N2	0.42131 (15)	0.3378 (3)	0.2557 (2)	0.0666 (8)
H2A	0.4219	0.3980	0.3072	0.080*
H2B	0.4499	0.2652	0.2754	0.080*
C2	0.27963 (16)	0.3970 (4)	−0.0840 (3)	0.0596 (9)
O2	0.23537 (15)	0.3379 (4)	−0.2766 (3)	0.0991 (10)
N3	0.42154 (12)	0.1420 (2)	0.09465 (19)	0.0461 (6)
C3	0.28139 (17)	0.4952 (4)	−0.0015 (4)	0.0714 (11)
H3B	0.2506	0.5740	−0.0223	0.086*
C4	0.32854 (17)	0.4753 (3)	0.1102 (4)	0.0664 (10)
H4A	0.3297	0.5414	0.1656	0.080*
N4	0.53086 (14)	−0.3605 (2)	0.1282 (2)	0.0546 (7)
C5	0.37562 (15)	0.3574 (3)	0.1443 (3)	0.0526 (8)
C6	0.37257 (14)	0.2579 (3)	0.0586 (2)	0.0455 (7)
C7	0.40423 (15)	0.0216 (3)	0.0405 (2)	0.0472 (7)
H7A	0.3597	0.0141	−0.0205	0.057*
C8	0.45009 (14)	−0.1054 (3)	0.0682 (2)	0.0440 (7)
C9	0.52046 (15)	−0.1025 (3)	0.1456 (2)	0.0475 (8)
H9A	0.5420	−0.0148	0.1789	0.057*
C10	0.55786 (15)	−0.2301 (3)	0.1725 (2)	0.0501 (7)
H10A	0.6050	−0.2258	0.2247	0.060*
C11	0.46362 (17)	−0.3624 (3)	0.0523 (3)	0.0578 (9)
H11A	0.4435	−0.4513	0.0194	0.069*
C12	0.42246 (16)	−0.2395 (3)	0.0201 (3)	0.0541 (8)
H12A	0.3759	−0.2464	−0.0341	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0439 (16)	0.0497 (17)	0.0602 (19)	0.0008 (14)	0.0209 (15)	0.0038 (15)
N1	0.0479 (17)	0.081 (2)	0.093 (3)	0.0097 (17)	0.0174 (18)	0.035 (2)
O1	0.0750 (18)	0.108 (2)	0.143 (3)	0.0420 (18)	0.0195 (18)	0.053 (2)
N2	0.0734 (19)	0.0603 (17)	0.0600 (18)	−0.0034 (15)	0.0241 (15)	−0.0172 (14)
C2	0.0397 (16)	0.056 (2)	0.075 (2)	0.0068 (15)	0.0181 (16)	0.0182 (17)
O2	0.0711 (19)	0.129 (3)	0.072 (2)	0.0121 (17)	0.0095 (15)	0.0210 (18)
N3	0.0456 (14)	0.0417 (14)	0.0456 (14)	0.0029 (11)	0.0154 (11)	0.0011 (11)
C3	0.0437 (18)	0.047 (2)	0.118 (3)	0.0074 (15)	0.031 (2)	0.014 (2)
C4	0.057 (2)	0.0462 (19)	0.102 (3)	−0.0031 (16)	0.041 (2)	−0.0124 (19)
N4	0.0548 (16)	0.0442 (15)	0.0627 (17)	0.0053 (12)	0.0242 (14)	0.0004 (12)
C5	0.0489 (17)	0.0417 (17)	0.070 (2)	−0.0083 (14)	0.0285 (17)	−0.0072 (15)
C6	0.0416 (15)	0.0404 (15)	0.0510 (17)	−0.0003 (13)	0.0174 (14)	0.0020 (13)
C7	0.0415 (15)	0.0455 (17)	0.0451 (16)	−0.0009 (13)	0.0106 (13)	0.0002 (13)
C8	0.0461 (16)	0.0438 (17)	0.0389 (15)	0.0014 (13)	0.0160 (13)	0.0006 (12)
C9	0.0471 (17)	0.0437 (17)	0.0465 (17)	−0.0015 (13)	0.0159 (14)	−0.0017 (13)
C10	0.0442 (16)	0.0512 (18)	0.0503 (17)	0.0023 (14)	0.0170 (13)	0.0015 (14)
C11	0.064 (2)	0.0414 (18)	0.063 (2)	−0.0051 (16)	0.0235 (17)	−0.0064 (15)
C12	0.0486 (17)	0.0504 (19)	0.0527 (17)	−0.0013 (15)	0.0129 (14)	−0.0052 (15)

Geometric parameters (Å, º)

C1—C6	1.375 (4)	C4—H4A	0.9300
C1—C2	1.379 (4)	N4—C11	1.331 (4)
C1—H1A	0.9300	N4—C10	1.338 (3)
N1—O1	1.225 (4)	C5—C6	1.414 (4)
N1—O2	1.226 (4)	C7—C8	1.457 (4)
N1—C2	1.450 (5)	C7—H7A	0.9300
N2—C5	1.347 (4)	C8—C12	1.383 (4)
N2—H2A	0.8600	C8—C9	1.386 (4)
N2—H2B	0.8600	C9—C10	1.368 (4)
C2—C3	1.384 (5)	C9—H9A	0.9300
N3—C7	1.269 (3)	C10—H10A	0.9300
N3—C6	1.414 (3)	C11—C12	1.372 (4)
C3—C4	1.359 (5)	C11—H11A	0.9300
C3—H3B	0.9300	C12—H12A	0.9300
C4—C5	1.405 (4)		
C6—C1—C2	120.4 (3)	C4—C5—C6	118.1 (3)
C6—C1—H1A	119.8	C1—C6—C5	119.7 (3)
C2—C1—H1A	119.8	C1—C6—N3	123.3 (3)
O1—N1—O2	123.7 (4)	C5—C6—N3	117.0 (2)
O1—N1—C2	117.7 (4)	N3—C7—C8	123.8 (3)
O2—N1—C2	118.6 (3)	N3—C7—H7A	118.1
C5—N2—H2A	120.0	C8—C7—H7A	118.1
C5—N2—H2B	120.0	C12—C8—C9	116.8 (3)

H2A—N2—H2B	120.0	C12—C8—C7	119.4 (2)
C1—C2—C3	120.9 (3)	C9—C8—C7	123.7 (3)
C1—C2—N1	118.7 (4)	C10—C9—C8	119.3 (3)
C3—C2—N1	120.3 (3)	C10—C9—H9A	120.4
C7—N3—C6	118.5 (2)	C8—C9—H9A	120.4
C4—C3—C2	119.3 (3)	N4—C10—C9	124.1 (3)
C4—C3—H3B	120.4	N4—C10—H10A	118.0
C2—C3—H3B	120.4	C9—C10—H10A	118.0
C3—C4—C5	121.7 (3)	N4—C11—C12	123.2 (3)
C3—C4—H4A	119.2	N4—C11—H11A	118.4
C5—C4—H4A	119.2	C12—C11—H11A	118.4
C11—N4—C10	116.5 (2)	C11—C12—C8	120.2 (3)
N2—C5—C4	120.9 (3)	C11—C12—H12A	119.9
N2—C5—C6	121.0 (3)	C8—C12—H12A	119.9

C6—C1—C2—C3	0.0 (5)	N2—C5—C6—N3	−2.8 (4)
C6—C1—C2—N1	179.5 (3)	C4—C5—C6—N3	178.6 (2)
O1—N1—C2—C1	172.5 (3)	C7—N3—C6—C1	−32.3 (4)
O2—N1—C2—C1	−7.6 (5)	C7—N3—C6—C5	149.7 (3)
O1—N1—C2—C3	−8.0 (5)	C6—N3—C7—C8	179.3 (2)
O2—N1—C2—C3	171.9 (3)	N3—C7—C8—C12	167.2 (3)
C1—C2—C3—C4	0.3 (5)	N3—C7—C8—C9	−9.4 (4)
N1—C2—C3—C4	−179.2 (3)	C12—C8—C9—C10	−1.7 (4)
C2—C3—C4—C5	0.0 (5)	C7—C8—C9—C10	175.0 (3)
C3—C4—C5—N2	−178.9 (3)	C11—N4—C10—C9	1.1 (4)
C3—C4—C5—C6	−0.4 (4)	C8—C9—C10—N4	0.2 (4)
C2—C1—C6—C5	−0.4 (4)	C10—N4—C11—C12	−0.7 (5)
C2—C1—C6—N3	−178.3 (3)	N4—C11—C12—C8	−0.9 (5)
N2—C5—C6—C1	179.1 (3)	C9—C8—C12—C11	2.0 (4)
C4—C5—C6—C1	0.6 (4)	C7—C8—C12—C11	−174.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···N4^{i}	0.86	2.42	3.091 (3)	135
N2—H2*B*···N3	0.86	2.42	2.751 (3)	103
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