organic compounds

2-Iodo-3-nitropyridine

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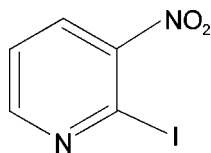Correspondence e-mail: lihuamao2009@yahoo.cn

In the crystal structure of the title compound, $C_5H_3IN_2O_2$, intermolecular C—H···N hydrogen-bonding interactions link the molecules into one-dimensional chains along the b axis.

Related literature

For the applications of 2-iodo-3-nitropyridine in organic synthesis, see: Baik *et al.* (2005); Choi-Sledeski *et al.* (2003). For the crystal structure of related compounds, see: Holmes *et al.* (2002); Saha *et al.* (2006).

Experimental

Crystal data

$C_5H_3IN_2O_2$	V = 694.8 (3) Å³
M_r = 249.99	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 8.0169 (15) Å	μ = 4.54 mm⁻¹
b = 12.313 (2) Å	T = 298 K
c = 8.0999 (15) Å	$0.60 \times 0.30 \times 0.21$ mm
β = 119.66 (2)°	

Data collection

Bruker SMART CCD area-detector diffractometer	3615 measured reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	1345 independent reflections
	1267 reflections with $I > 2\sigma(I)$
T_{min} = 0.147, T_{max} = 0.385	R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030	91 parameters
$wR(F^2)$ = 0.075	H-atom parameters constrained
S = 1.12	$\Delta\rho_{max}$ = 0.50 e Å⁻³
1345 reflections	$\Delta\rho_{min}$ = −1.09 e Å⁻³

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···N2ⁱ	0.93	2.61	3.529 (5)	172

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2322).

References

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Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Choi-Sledeski, Y. M., Pauls, H. W., Barton, J. N., Ewing, W. R., Green, D. M., Becker, M. R. & Gong, Y. (2003). US Patent No. 6 602 864.
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supporting information

2-Iodo-3-nitropyridine

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S1. Comment

In this paper, we report the crystal structure of the title compound, 2-iodo-3-nitropyridine, which is an important intermediate in organic synthesis (Baik *et al.*, 2005; Choi-Sledeski *et al.*, 2003).

   In the molecule of the title compound (Fig. 1), all bond lengths are normal and in a good agreement with those reported previously for 2,6-diiodopyridine (Holmes *et al.*, 2002) and 2-iodo-3hydroxypyridine (Saha *et al.*, 2006). Atoms I1 and N1 are slightly displaced on opposite sides of the pyridine ring by 0.0719 (3) and 0.015 (4) Å, respectively. The nitro group is tilted by 34.6 (3)° with respect to the pyridine ring. The crystal structure is stabilized by intermolecular C—H···N hydrogen bonds (Table 1) linking the molecules into one dimension chains along the *b* axis (Fig. 2).

S2. Experimental

The title compound was prepared by reaction of 2-amino-3-nitropyridine (1.1 g, 5 mmol), KNO_3 (1.01 g, 10 mmol), HI (6.6 g, 25 mmol, 50% aqueous solution), CuI (0.48 g, 2.5 mmol) and DMSO (60 ml) at 333K. After neutralizing with an alkaline solution, the reaction mixture was extracted several times with diethyl ether. The combined ethereal extracts were washed with water, dried over anhydrous sodium sulfate and concentrated to afford the crude product. Purification by flash chromatography gave 2-iodo-3-nitropyridine as a yellow solid in 70% isolated yield (0.875 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of a methanol solution at room temperature over a period of one week.

S3. Refinement

All H atoms were found on difference maps, with C—H = 0.93 Å and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.
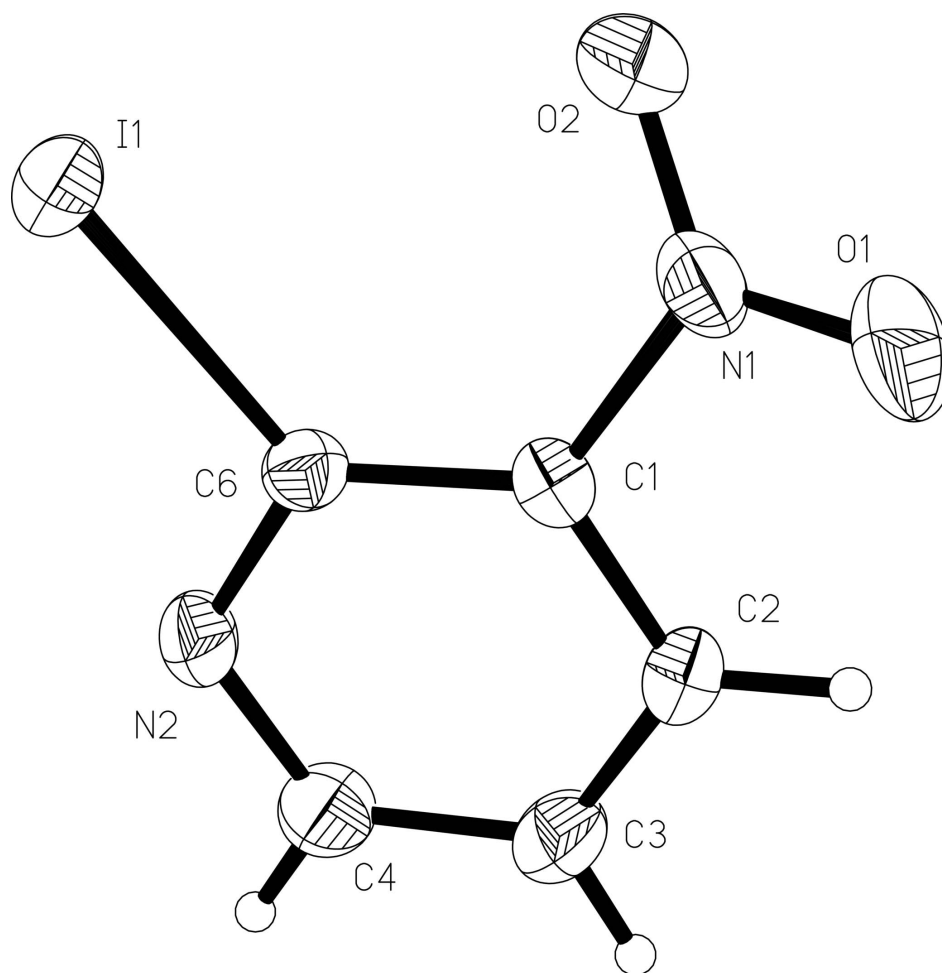
Figure 1

View of the title compound, with displacement ellipsoids drawn at the 40% probability level.
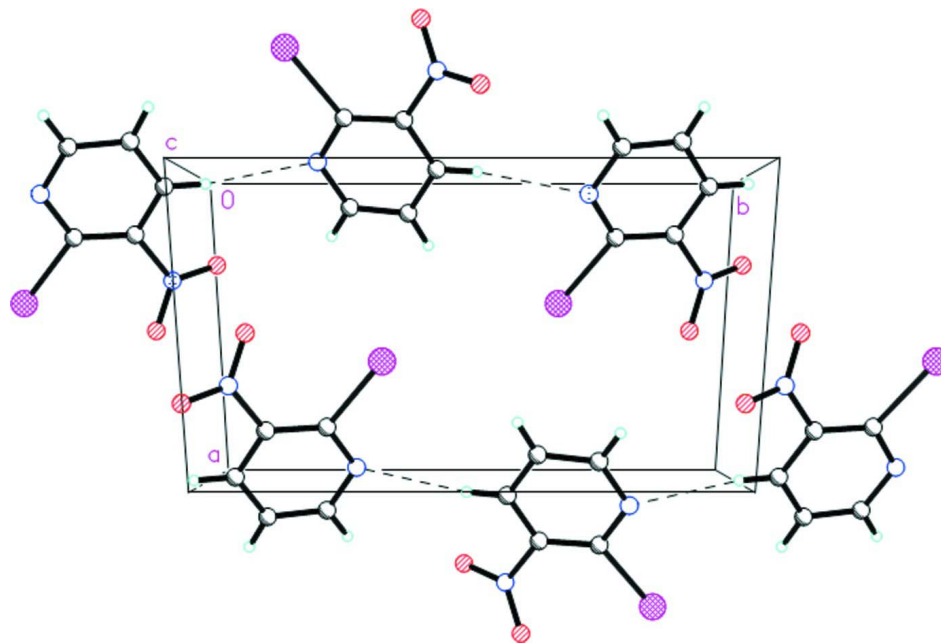
Figure 2
A partial packing diagram of the title compound viewed along the *c* axis. Intermolecular H bonds are shown as dashed lines.

2-Iodo-3-nitropyridine

Crystal data

$C_5H_3IN_2O_2$	$F(000) = 464$
$M_r = 249.99$	$D_x = 2.390$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 1077 reflections
$a = 8.0169$ (15) Å	$\theta = 2.5–25.9°$
$b = 12.313$ (2) Å	$\mu = 4.54$ mm^{-1}
$c = 8.0999$ (15) Å	$T = 298$ K
$\beta = 119.66$ (2)°	Block, yellow
$V = 694.8$ (3) Å^3	$0.60 \times 0.30 \times 0.21$ mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	3615 measured reflections
	1345 independent reflections
Radiation source: fine-focus sealed tube	1267 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.037$
φ and ω scans	$\theta_{max} = 26.0°$, $\theta_{min} = 2.9°$
Absorption correction: multi-scan	$h = -9{\rightarrow}8$
(*SADABS*; Sheldrick, 1996)	$k = -15{\rightarrow}14$
$T_{min} = 0.147$, $T_{max} = 0.385$	$l = -9{\rightarrow}9$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier
Least-squares matrix: full	map
$R[F^2 > 2\sigma(F^2)] = 0.030$	Hydrogen site location: inferred from
$wR(F^2) = 0.075$	neighbouring sites
$S = 1.12$	H-atom parameters constrained
1345 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0401P)^2 + 0.1824P]$
91 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant	$\Delta\rho_{max} = 0.50$ e Å^{-3}
direct methods	$\Delta\rho_{min} = -1.09$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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}
I1	0.59372 (3)	0.162020 (19)	0.16542 (4)	0.03968 (14)
O1	0.7237 (5)	0.5149 (2)	0.3364 (5)	0.0601 (9)
O2	0.5036 (5)	0.4081 (3)	0.1331 (6)	0.0635 (9)
N1	0.6704 (5)	0.4373 (3)	0.2292 (5)	0.0395 (7)
C1	0.8177 (5)	0.3758 (3)	0.2090 (5)	0.0304 (7)
C2	0.9711 (5)	0.4353 (3)	0.2237 (6)	0.0378 (8)
H2A	0.9807	0.5095	0.2477	0.045*
C3	1.1084 (6)	0.3811 (4)	0.2019 (6)	0.0443 (10)
H3A	1.2127	0.4180	0.2084	0.053*
C4	1.0884 (5)	0.2712 (4)	0.1703 (6)	0.0465 (11)
H4A	1.1812	0.2349	0.1544	0.056*
N2	0.9430 (5)	0.2136 (2)	0.1610 (5)	0.0389 (7)
C5	0.8074 (5)	0.2654 (3)	0.1774 (5)	0.0300 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0391 (2)	0.0313 (2)	0.0496 (2)	−0.00698 (8)	0.02262 (15)	−0.00258 (9)
O1	0.073 (2)	0.0392 (16)	0.074 (2)	0.0116 (15)	0.0408 (19)	−0.0131 (16)
O2	0.0423 (18)	0.0458 (17)	0.107 (3)	0.0055 (15)	0.0404 (19)	0.0006 (19)
N1	0.043 (2)	0.0296 (16)	0.053 (2)	0.0098 (14)	0.0293 (17)	0.0093 (16)
C1	0.0318 (18)	0.0274 (17)	0.0322 (17)	0.0040 (14)	0.0161 (14)	0.0012 (15)
C2	0.040 (2)	0.0275 (17)	0.045 (2)	−0.0054 (15)	0.0204 (18)	−0.0024 (17)
C3	0.038 (2)	0.040 (2)	0.057 (2)	−0.0087 (17)	0.0248 (19)	−0.003 (2)
C4	0.038 (2)	0.043 (2)	0.067 (3)	0.0012 (16)	0.033 (2)	−0.0095 (19)

N2	0.0402 (17)	0.0257 (15)	0.0535 (19)	0.0024 (13)	0.0253 (15)	−0.0042 (15)
C5	0.0270 (16)	0.0306 (18)	0.0291 (16)	−0.0016 (14)	0.0114 (14)	0.0006 (14)

Geometric parameters (Å, º)

I1—C5	2.097 (3)	C2—H2A	0.9300
O1—N1	1.217 (4)	C3—C4	1.371 (8)
O2—N1	1.222 (5)	C3—H3A	0.9300
N1—C1	1.478 (4)	C4—N2	1.335 (5)
C1—C5	1.378 (5)	C4—H4A	0.9300
C1—C2	1.385 (5)	N2—C5	1.322 (4)
C2—C3	1.372 (6)		
O1—N1—O2	124.9 (3)	C4—C3—H3A	120.8
O1—N1—C1	117.6 (3)	C2—C3—H3A	120.8
O2—N1—C1	117.5 (3)	N2—C4—C3	123.6 (3)
C5—C1—C2	120.5 (3)	N2—C4—H4A	118.2
C5—C1—N1	123.2 (3)	C3—C4—H4A	118.2
C2—C1—N1	116.3 (3)	C5—N2—C4	118.5 (3)
C3—C2—C1	117.8 (4)	N2—C5—C1	121.2 (3)
C3—C2—H2A	121.1	N2—C5—I1	113.3 (2)
C1—C2—H2A	121.1	C1—C5—I1	125.4 (2)
C4—C3—C2	118.5 (3)		
O1—N1—C1—C5	146.8 (4)	C3—C4—N2—C5	2.2 (6)
O2—N1—C1—C5	−35.1 (5)	C4—N2—C5—C1	−1.9 (5)
O1—N1—C1—C2	−33.1 (5)	C4—N2—C5—I1	−178.8 (3)
O2—N1—C1—C2	145.0 (4)	C2—C1—C5—N2	0.2 (5)
C5—C1—C2—C3	1.3 (6)	N1—C1—C5—N2	−179.7 (3)
N1—C1—C2—C3	−178.7 (4)	C2—C1—C5—I1	176.6 (3)
C1—C2—C3—C4	−1.2 (6)	N1—C1—C5—I1	−3.3 (5)
C2—C3—C4—N2	−0.6 (7)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···N2^i	0.93	2.61	3.529 (5)	172

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