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2-Chloro-4-nitro-1*H*-imidazole

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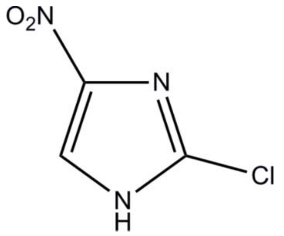
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.037; wR factor = 0.097; data-to-parameter ratio = 16.8.

The molecule of the title compound, $C_3H_2ClN_3O_2$, is almost planar; the dihedral angle between the imidazole ring and the nitro group is 1.7 (2)°. In the crystal structure, pairs of intermolecular C—H···O hydrogen bonds link inversion-related molecules into dimers, generating $R_2^2(10)$ ring motifs. The dimers are interconnected into two-dimensional networks parallel to (102) *via* intermolecular N—H···N hydrogen bonds. Further stabilization is provided by short intermolecular Cl···O interactions [3.142 (2) and 3.1475 (19) Å].

Related literature

For general background to and applications of imidazole derivatives, see: Anuradha *et al.* (2006); Clark & Macquarrie (1996); Jadhav *et al.* (2008); Kolavi *et al.* (2006); Susanta *et al.* (2000). For graph-set descriptions of hydrogen-bond ring motifs, see: Bernstein *et al.* (1995). For related 4-nitro-imidazole crystal structures, see: Ségalas *et al.* (1992); De Bondt *et al.* (1993). For the stability of the temperature controller used for the data collection, see: Cosier & Glazer (1986).



Experimental

Crystal data

$C_3H_2ClN_3O_2$
M_r = 147.53
Monoclinic, $P2_1/c$
a = 5.905 (2) Å
b = 10.033 (4) Å
c = 9.150 (3) Å
β = 105.180 (8)°
V = 523.2 (3) $Å^3$
Z = 4
Mo $K\alpha$ radiation
μ = 0.64 mm^{-1}
T = 100 K
0.29 × 0.19 × 0.04 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.837, T_{max} = 0.977
5484 measured reflections
1509 independent reflections
1195 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.097
S = 1.11
1509 reflections
90 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.42 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.44 e $Å^{-3}$

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*1···N2^{i}	0.86 (3)	2.07 (3)	2.900 (2)	163 (2)
C2—H2···O1^{ii}	0.92 (3)	2.48 (3)	3.317 (3)	151 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5106).

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supplementary materials

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2-Chloro-4-nitro-1*H*-imidazole

H.-K. Fun, **J. H. Goh**, **B. Chandrakantha**, **A. M. Isloor** and **P. Shetty**

Comment

The nitro aromatic compounds are used as key substrates for the preparation of useful materials such as dyes, pharmaceuticals, perfumes and plastics (Susanta *et al.*, 2000). Therefore, nitration of hydrocarbons particularly of aromatic compounds is probably one of the most widely studied organic reactions (Jadhav *et al.*, 2008). In addition, they have proven to be valuable reagents for the synthesis of complex target molecules (Kolavi *et al.*, 2006). Most of the substituted imidazoles are widely used in pharmaceutical ingredients (Clark & Macquarrie, 1996). The imidazole nucleus is one of the important heterocyclic groups due to its presence in a large number of bioactive pharmaceutical and agrochemicals (Anuradha *et al.*, 2006). It was also reported that a large number of compounds containing the imidazole ring possess some moderately useful activities. The environmentally friendly nitration reaction has been the focus of recent research.

In the title imidazole derivative, the 1*H*-imidazole ring with atom sequence C1/N1/C2/C3/N2 is essentially planar, with a maximum deviation of 0.003 (2) Å at atom N1. The nitro group is coplanar with the attached 1*H*-imidazole ring, as indicated by the dihedral angle of 1.7 (2)°. The geometric parameters agree well with those reported for related 4-nitroimidazole structures (Ségalas *et al.*, 1992; De Bondt *et al.*, 1993).

In the crystal structure, (Fig. 2), pairs of intermolecular C2—H2···O1 hydrogen bonds (Table 1) link inversion-related molecules into dimers, generating $R^2_2(10)$ hydrogen bond ring motifs (Bernstein *et al.*, 1995). These dimers are further interconnected into two-dimensional arrays parallel to the (102) plane *via* intermolecular N1—H1N1···N2 hydrogen bonds (Table 1). The interesting features of the crystal structure are the intermolecular short Cl···O interactions [Cl1···O1^{iii} = 3.143 (2) and Cl1···O2^{i} = 3.148 (2) Å; (i) 1-x, y-1/2, 1/2-z and (iii) 1+x, 3/2-y, z-1/2] which are shorter than the sum of the van der Waals radii of the relavant atoms and help to further stabilize the crystal structure.

Experimental

Nitronium tetrafluoroborate (1.42 g, 0.0107 mol) was dissolved in nitromethane (10 ml) and 2-chloroimidazole (1 g, 0.0097 mol) was then added in lot-wise. The reaction mixture was stirred at room temperature for 3 h. The reaction mixture was then neutrallized with an aqueous solution of sodium bicarbonate. The separated solid was then filtered. The crude product was purified by column chromatography using 60–120 silica gel. The fraction eluted at 10 % ethyl acetate in hexane was concentrated to afford the title compound as pale yellow single crystals (Yield 0.9 g, 62.93 %; *m.p.* 363–366 K).

Refinement

Atoms H1N1 and H2 were located in a difference Fourier map and allowed to refine freely [N1—H1N1 = 0.86 (3) and C2—H2A = 0.93 (3) Å].

Figures

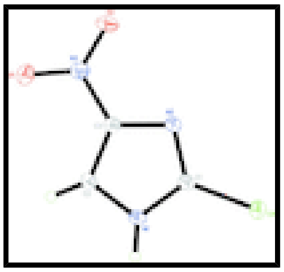

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids for non-H atoms and the atom-numbering scheme.

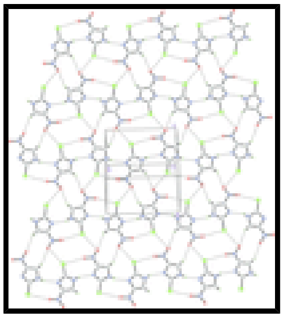

Fig. 2. The crystal structure of the title compound, showing a two-dimensional network. Intermolecular interactions are shown as dashed lines.

2-Chloro-4-nitro-1*H*-imidazole

Crystal data

$C_3H_2ClN_3O_2$

$M_r = 147.53$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 5.905$ (2) Å

$b = 10.033$ (4) Å

$c = 9.150$ (3) Å

$\beta = 105.180$ (8)°

$V = 523.2$ (3) Å^3

$Z = 4$

$F(000) = 296$

$D_x = 1.873$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2073 reflections

$\theta = 3.6–30.0°$

$\mu = 0.64$ mm^{-1}

$T = 100$ K

Plate, yellow

0.29 × 0.19 × 0.04 mm

Data collection

Bruker APEXII DUO CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2009)

$T_{min} = 0.837$, $T_{max} = 0.977$

5484 measured reflections

1509 independent reflections

1195 reflections with $I > 2\sigma(I)$

$R_{int} = 0.037$

$\theta_{max} = 30.0°$, $\theta_{min} = 3.1°$

$h = -6 \rightarrow 8$

$k = -13 \rightarrow 14$

$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.037$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.097$	All H-atom parameters refined
$S = 1.11$	$w = 1/[\sigma^2(F_o^2) + (0.0453P)^2 + 0.1822P]$ where $P = (F_o^2 + 2F_c^2)/3$
1509 reflections	$(\Delta/\sigma)_{max} = 0.001$
90 parameters	$\Delta\rho_{max} = 0.42$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.44$ e Å^{-3}

Special details

Experimental. The crystal was placed in the cold stream of an Oxford Cryosystems Cobra open-flow nitrogen cryostat (Cosier & Glazer, 1986) operating at 100.0 (1)K.

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 > 2sigma(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}
Cl1	0.72178 (8)	0.63986 (4)	0.10842 (5)	0.01869 (15)
O1	0.0169 (3)	0.65199 (14)	0.47907 (19)	0.0268 (4)
O2	0.1231 (3)	0.84048 (13)	0.40048 (18)	0.0250 (3)
N1	0.4713 (3)	0.49414 (15)	0.25596 (19)	0.0157 (3)
N2	0.4212 (3)	0.71387 (14)	0.26450 (18)	0.0151 (3)
N3	0.1318 (3)	0.71851 (16)	0.41138 (19)	0.0190 (3)
C1	0.5304 (3)	0.61677 (16)	0.2149 (2)	0.0149 (4)
C2	0.3104 (3)	0.51281 (17)	0.3371 (2)	0.0164 (4)
C3	0.2845 (3)	0.64762 (17)	0.3405 (2)	0.0150 (4)
H1N1	0.525 (4)	0.417 (3)	0.240 (3)	0.025 (6)*
H2	0.246 (4)	0.441 (3)	0.375 (3)	0.025 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0218 (2)	0.0162 (2)	0.0214 (3)	−0.00093 (16)	0.01140 (19)	0.00052 (17)

O1	0.0311 (8)	0.0228 (7)	0.0340 (9)	−0.0028 (6)	0.0221 (7)	−0.0015 (6)
O2	0.0308 (8)	0.0120 (6)	0.0351 (9)	0.0043 (5)	0.0140 (7)	−0.0022 (6)
N1	0.0193 (8)	0.0096 (7)	0.0196 (8)	0.0010 (6)	0.0078 (7)	−0.0003 (6)
N2	0.0179 (8)	0.0107 (6)	0.0184 (8)	0.0001 (5)	0.0075 (6)	0.0000 (6)
N3	0.0206 (8)	0.0151 (7)	0.0231 (9)	0.0008 (6)	0.0090 (7)	−0.0017 (6)
C1	0.0174 (9)	0.0113 (8)	0.0163 (9)	−0.0013 (6)	0.0053 (7)	−0.0004 (6)
C2	0.0186 (9)	0.0114 (8)	0.0208 (10)	−0.0010 (6)	0.0082 (8)	0.0001 (7)
C3	0.0167 (9)	0.0122 (8)	0.0170 (9)	−0.0007 (6)	0.0060 (7)	−0.0019 (7)

Geometric parameters (Å, °)

Cl1—C1	1.690 (2)	N2—C1	1.313 (2)
O1—N3	1.228 (2)	N2—C3	1.368 (2)
O2—N3	1.228 (2)	N3—C3	1.430 (2)
N1—C1	1.359 (2)	C2—C3	1.362 (2)
N1—C2	1.363 (3)	C2—H2	0.93 (3)
N1—H1N1	0.86 (3)		

C1—N1—C2	107.01 (15)	N2—C1—Cl1	124.11 (14)
C1—N1—H1N1	129.2 (17)	N1—C1—Cl1	122.87 (14)
C2—N1—H1N1	123.7 (17)	C3—C2—N1	104.32 (16)
C1—N2—C3	102.95 (15)	C3—C2—H2	135.0 (16)
O2—N3—O1	124.46 (17)	N1—C2—H2	120.7 (16)
O2—N3—C3	118.46 (16)	C2—C3—N2	112.71 (17)
O1—N3—C3	117.08 (16)	C2—C3—N3	126.29 (18)
N2—C1—N1	113.01 (17)	N2—C3—N3	120.99 (16)

C3—N2—C1—N1	−0.4 (2)	C1—N2—C3—C2	0.0 (2)
C3—N2—C1—Cl1	178.70 (15)	C1—N2—C3—N3	−179.05 (17)
C2—N1—C1—N2	0.6 (2)	O2—N3—C3—C2	−177.8 (2)
C2—N1—C1—Cl1	−178.52 (14)	O1—N3—C3—C2	1.9 (3)
C1—N1—C2—C3	−0.5 (2)	O2—N3—C3—N2	1.1 (3)
N1—C2—C3—N2	0.3 (2)	O1—N3—C3—N2	−179.10 (18)
N1—C2—C3—N3	179.32 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N1···N2^{i}	0.86 (3)	2.07 (3)	2.900 (2)	163 (2)
C2—H2···O1^{ii}	0.92 (3)	2.48 (3)	3.317 (3)	151 (2)

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

Fig. 1

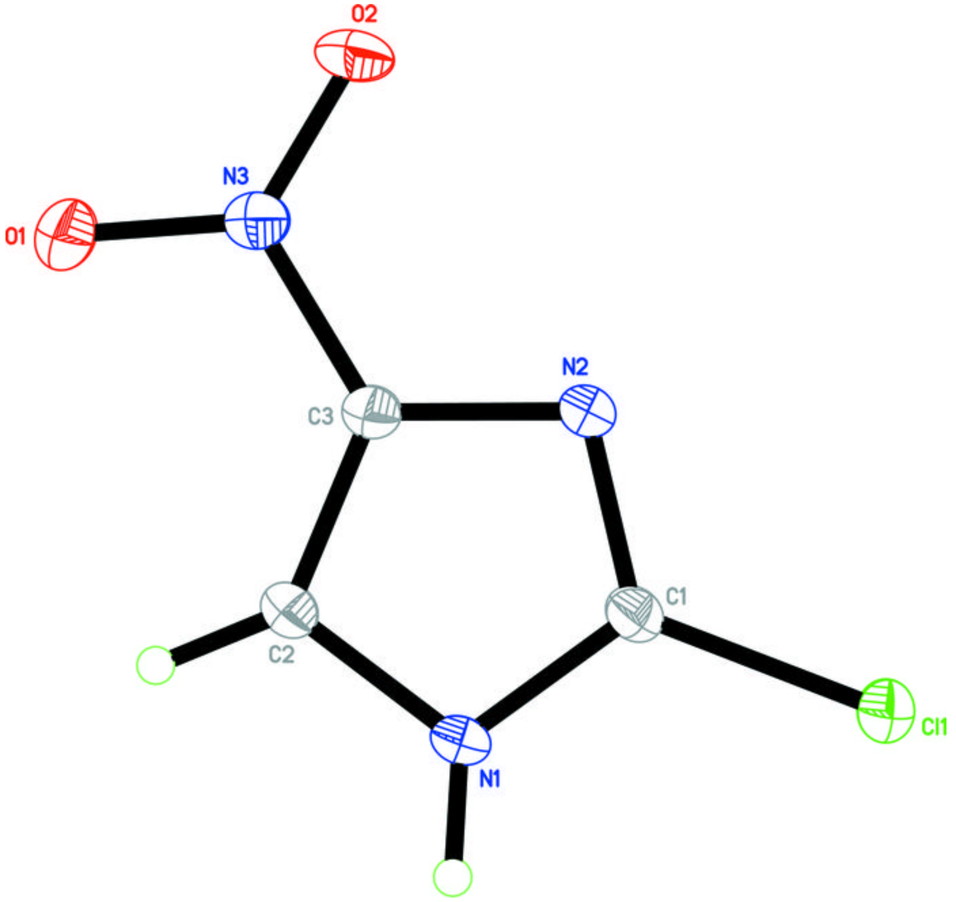

Fig. 2