Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Amino-6-nitro-1*H*-benzoimidazol-3-ium chloride

You-Sheng Chen, Kun Zhang* and Su-Qing Zhao

Faculty of Chemical Engineering and Light Industry, Guangdong University of Technology, Waihuan Xi Road No. 100, Guangzhou Higher Education Mega Center, Panyu District, Guangzhou, Guangzhou 510006, People's Republic of China
Correspondence e-mail: gdut_chen@163.com

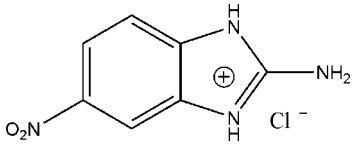
Received 27 June 2009; accepted 12 July 2009

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; R factor = 0.035; wR factor = 0.096; data-to-parameter ratio = 12.4.

In the cation of the title compound, $C_7H_7N_4O_2^+\cdot Cl^-$, the benzimidazole ring system is planar with a maximum deviation of −0.019 (3) Å. In the crystal structure, C—H···Cl, N—H···Cl, and N—H···Cl interactions link the molecules into a two-dimensional network. π–π contacts between benzimidazole rings [centroid–centroid distances = 3.928 (1) and 3.587 (1) Å] may further stabilize the structure.

Related literature

For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_7H_7N_4O_2^+\cdot Cl^-$
M_r = 214.62
Monoclinic, $C2/c$
a = 13.969 (3) Å
b = 7.8064 (19) Å
c = 16.490 (4) Å
β = 91.303 (3)°
V = 1797.7 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.40 mm^{-1}
T = 291 K
0.12 × 0.12 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.953, T_{max} = 0.961
4345 measured reflections
1580 independent reflections
1242 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
3 standard reflections
frequency: 120 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.096
S = 1.02
1580 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···Cl1	0.93	2.75	3.436 (2)	132
N1—H1A···Cl1	0.86	2.61	3.2830 (19)	135
N1—H1A···Cl1^{i}	0.86	2.76	3.4102 (19)	134
N3—H3A···Cl1^{ii}	0.86	2.55	3.269 (2)	142
N2—H2A···Cl1^{ii}	0.86	2.31	3.0601 (19)	145

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; 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: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2728).

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supporting information

Acta Cryst. (2009). E**65**, o1926 [doi:10.1107/S1600536809027342]

2-Amino-6-nitro-1*H*-benzoimidazol-3-ium chloride

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S1. Comment

Some derivatives of 2-aminobenzimidazolium are important chemical materials. We report herein the crystal structure of the title compound.

In the molecule of the title compound, (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (N1/N2/C1-C3) and B (C2-C7) are, of course, planar and they are oriented at a dihedral angle of 1.70 (3)°. The benzimidazole ring system is planar with a maximum deviation of -0.019 (3) Å for atom C5. Atoms O1, N3 and N4 are 0.064 (3), -0.044 (3) and -0.094 (3) Å away from the plane of the benzimidazole ring system, respectively.

In the crystal structure, intramolecular C-H···Cl and N-H···Cl and intermolecular N-H···Cl interactions (Table 1) link the molecules into a two dimensional network (Fig. 2), in which they may be effective in the stabilization of the structure. The *π*–*π* contacts between the benzimidazole rings, Cg1—Cg2^{i} and Cg2—Cg2^{ii} [symmetry codes: (i) 1/2 - x, 3/2 - y, -z, (ii) -x, 1 - y, -z, where Cg1 and Cg2 are centroids of the rings A (N1/N2/C1-C3) and B (C2-C7), respectively] may further stabilize the structure, with centroid-centroid distances of 3.928 (1) and 3.587 (1) Å, respectively.

S2. Experimental

For the preparation of the title compound, a suspension of 4-nitro-*o*-phenylenediamine (1.4 g, 9.1 mmol) in a solution of BrCN (0.97 g, 9.2 mmol) in water (30 ml) was refluxed for 7 h, and then cooled and neutralized with NH_4OH (25%) to pH = 11. The formed precipitate was filtered, washed with water and dried to give the title compound, as a yellow solid (yield; 1.5 g, 92%). Crystals suitable for X-ray analysis were obtained after 3 d by slow evaporation of the mother liquid at room temperature.

S3. Refinement

H atoms were positioned geometrically, with N-H = 0.86 Å (for NH and NH_2) and C-H = 0.93 Å for aromatic H, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

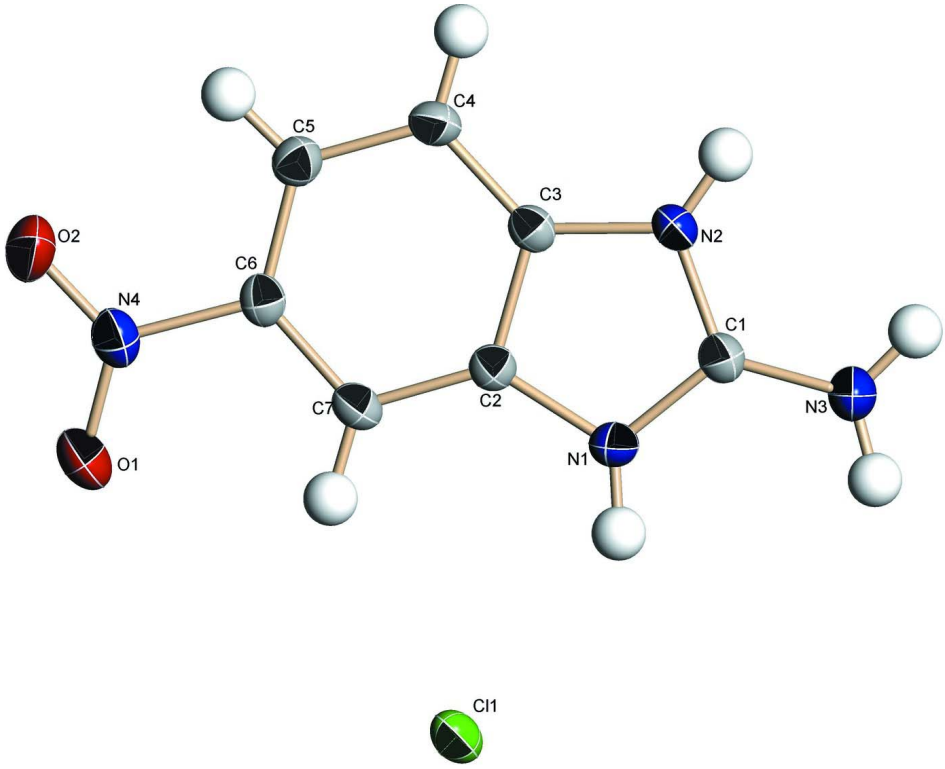

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

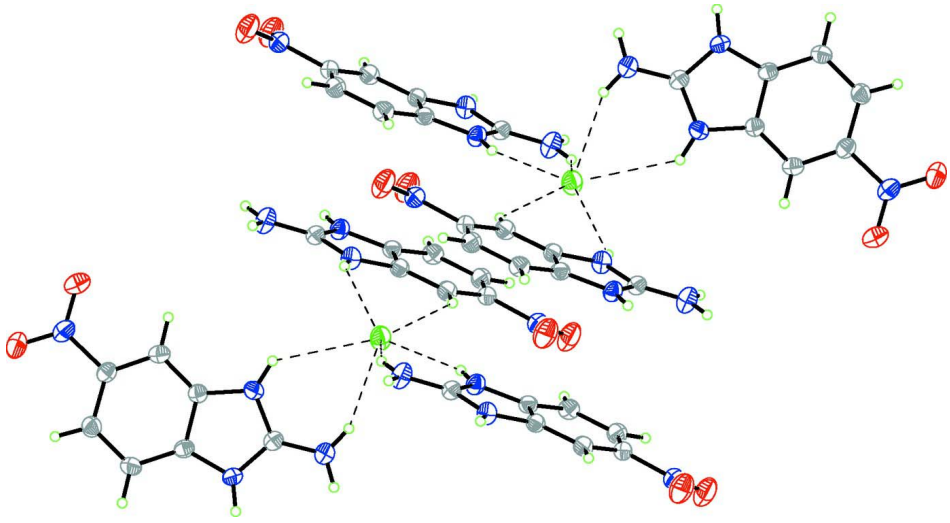

Figure 2

A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_7H_7N_4O_2^+ \cdot Cl^-$
$M_r = 214.62$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 13.969$ (3) Å
$b = 7.8064$ (19) Å
$c = 16.490$ (4) Å
$\beta = 91.303$ (3)°
$V = 1797.7$ (7) Å^3
$Z = 8$
$F(000) = 880$
$D_x = 1.586$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 2.1$–25.3°
$\mu = 0.40$ mm^{-1}
$T = 291$ K
Block, yellow
0.12 × 0.12 × 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)
$T_{min} = 0.953$, $T_{max} = 0.961$
4345 measured reflections
1580 independent reflections
1242 reflections with $I > 2\sigma(I)$
$R_{int} = 0.041$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
$h = -16 \rightarrow 16$
$k = -9 \rightarrow 9$
$l = -19 \rightarrow 13$
3 standard reflections every 120 min
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.096$
$S = 1.02$
1580 reflections
127 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
$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.969P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ 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}
Cl1	0.40318 (4)	0.59647 (8)	0.67245 (4)	0.0535 (2)
O1	0.38248 (13)	0.6546 (3)	0.39469 (12)	0.0718 (6)
O2	0.42766 (13)	0.8483 (3)	0.31128 (11)	0.0743 (6)

N1	0.61668 (12)	0.7489 (2)	0.63485 (10)	0.0424 (5)
H1A	0.5835	0.6840	0.6656	0.051*
N2	0.72946 (12)	0.9189 (2)	0.59344 (10)	0.0403 (4)
H2A	0.7805	0.9804	0.5934	0.048*
N3	0.73958 (14)	0.8248 (3)	0.72889 (12)	0.0552 (6)
H3A	0.7916	0.8812	0.7380	0.066*
H3B	0.7150	0.7649	0.7669	0.066*
N4	0.43847 (13)	0.7660 (3)	0.37360 (12)	0.0491 (5)
C1	0.69763 (15)	0.8301 (3)	0.65691 (13)	0.0392 (5)
C2	0.59515 (14)	0.7865 (3)	0.55416 (12)	0.0363 (5)
C3	0.66716 (14)	0.8960 (3)	0.52782 (13)	0.0361 (5)
C4	0.66616 (16)	0.9615 (3)	0.44987 (13)	0.0420 (5)
H4	0.7147	1.0332	0.4323	0.050*
C5	0.59021 (16)	0.9163 (3)	0.39925 (13)	0.0432 (5)
H5	0.5859	0.9590	0.3466	0.052*
C6	0.52047 (15)	0.8067 (3)	0.42752 (13)	0.0392 (5)
C7	0.52051 (15)	0.7385 (3)	0.50434 (13)	0.0401 (5)
H7	0.4728	0.6644	0.5213	0.048*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0422 (4)	0.0708 (4)	0.0475 (4)	−0.0144 (3)	0.0034 (3)	−0.0083 (3)
O1	0.0580 (12)	0.0805 (13)	0.0762 (13)	−0.0252 (10)	−0.0151 (10)	0.0019 (11)
O2	0.0612 (12)	0.1070 (16)	0.0539 (12)	−0.0053 (11)	−0.0168 (9)	0.0130 (11)
N1	0.0447 (11)	0.0431 (11)	0.0393 (11)	−0.0091 (9)	0.0011 (8)	0.0035 (8)
N2	0.0328 (10)	0.0461 (11)	0.0419 (10)	−0.0076 (8)	−0.0012 (8)	0.0000 (8)
N3	0.0523 (13)	0.0663 (14)	0.0466 (12)	−0.0117 (10)	−0.0096 (10)	0.0075 (10)
N4	0.0387 (11)	0.0593 (13)	0.0491 (12)	0.0016 (10)	−0.0039 (9)	−0.0090 (10)
C1	0.0351 (11)	0.0413 (12)	0.0411 (13)	0.0005 (9)	−0.0013 (9)	−0.0016 (10)
C2	0.0357 (11)	0.0366 (11)	0.0367 (12)	0.0003 (9)	0.0019 (9)	−0.0012 (9)
C3	0.0311 (11)	0.0368 (11)	0.0405 (12)	0.0005 (9)	0.0022 (9)	−0.0035 (9)
C4	0.0372 (12)	0.0445 (13)	0.0446 (13)	−0.0050 (10)	0.0064 (10)	0.0019 (10)
C5	0.0430 (13)	0.0479 (13)	0.0387 (12)	0.0007 (10)	0.0026 (10)	0.0006 (10)
C6	0.0364 (12)	0.0420 (12)	0.0391 (12)	0.0023 (9)	−0.0021 (9)	−0.0063 (10)
C7	0.0362 (12)	0.0393 (12)	0.0448 (13)	−0.0059 (9)	0.0039 (10)	−0.0051 (10)

Geometric parameters (Å, º)

N1—H1A	0.8600	C2—C7	1.365 (3)
N2—H2A	0.8600	C3—N2	1.385 (3)
N3—H3A	0.8600	C3—C4	1.383 (3)
N3—H3B	0.8600	C4—C5	1.381 (3)
N4—O1	1.225 (3)	C4—H4	0.9300
N4—O2	1.219 (2)	C5—C6	1.385 (3)
C1—N1	1.340 (3)	C5—H5	0.9300
C1—N2	1.340 (3)	C6—C7	1.374 (3)
C1—N3	1.312 (3)	C6—N4	1.469 (3)

C2—N1	1.389 (3)	C7—H7	0.9300
C2—C3	1.397 (3)		
C1—N1—C2	108.84 (17)	C7—C2—C3	121.8 (2)
C1—N1—H1A	125.6	N2—C3—C2	106.28 (18)
C2—N1—H1A	125.6	C4—C3—N2	132.2 (2)
C1—N2—C3	109.26 (17)	C4—C3—C2	121.6 (2)
C1—N2—H2A	125.4	C3—C4—H4	121.4
C3—N2—H2A	125.4	C5—C4—C3	117.3 (2)
C1—N3—H3A	120.0	C5—C4—H4	121.4
C1—N3—H3B	120.0	C4—C5—C6	119.4 (2)
H3A—N3—H3B	120.0	C4—C5—H5	120.3
O1—N4—C6	118.4 (2)	C6—C5—H5	120.3
O2—N4—O1	123.1 (2)	C5—C6—N4	118.3 (2)
O2—N4—C6	118.5 (2)	C7—C6—N4	117.27 (19)
N2—C1—N1	109.02 (18)	C7—C6—C5	124.3 (2)
N3—C1—N1	126.0 (2)	C2—C7—C6	115.6 (2)
N3—C1—N2	125.0 (2)	C2—C7—H7	122.2
N1—C2—C3	106.60 (18)	C6—C7—H7	122.2
C7—C2—N1	131.6 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···Cl1	0.93	2.75	3.436 (2)	132
N1—H1*A*···Cl1	0.86	2.61	3.2830 (19)	135
N1—H1*A*···Cl1^{i}	0.86	2.76	3.4102 (19)	134
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N2—H2*A*···Cl1^{ii}	0.86	2.31	3.0601 (19)	145

Symmetry codes: (i) −*x*+1, *y*, −*z*+3/2; (ii) *x*+1/2, *y*+1/2, *z*.