Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-Iodoanilinium perchlorate

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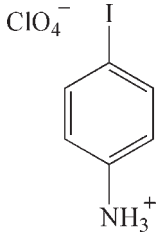
Received 27 July 2009; accepted 12 March 2010

Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.005 Å; R factor = 0.030; wR factor = 0.071; data-to-parameter ratio = 18.3.

In the crystal structure of the title compound, $C_6H_7IN^+\cdot ClO_4^-$, the ions are connected in a three-dimensional hydrogen-bonded network *via* N—H···O hydrogen bonds.

Related literature

For related structures, see: Paixao *et al.* (1999); Wiedenfeld *et al.* (2004); Bendjeddou *et al.* (2003); Kapoor *et al.* (2008). For the synthetic strategy, see: Cinčić & Kaitner (2007). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_6H_7IN^+\cdot ClO_4^-$
M_r = 319.48
Triclinic, $P\overline{1}$
a = 5.105 (1) Å
b = 7.2445 (14) Å
c = 13.359 (3) Å
α = 89.47 (3)°
β = 88.74 (3)°
γ = 74.61 (3)°
V = 476.22 (17) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 3.63 mm^{-1}
T = 298 K
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.484, T_{max} = 0.489
4945 measured reflections
2180 independent reflections
1956 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.071
S = 1.11
2180 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^{i}	0.89	2.12	3.002 (4)	174
N1—H1B···O3^{ii}	0.89	2.17	2.911 (4)	141
N1—H1C···O3^{iii}	0.89	2.21	3.069 (4)	162

Symmetry codes: (i) $x-1, y-1, z$; (ii) $-x+1, -y+1, -z$; (iii) $x-1, y, z$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *PRPKAPPA* (Ferguson, 1999).

This work was supported by a start-up grant from Southeast University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2083).

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supporting information

Acta Cryst. (2010). E**66**, o895 [doi:10.1107/S1600536810009347]

4-Iodoanilinium perchlorate

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S1. Comment

To the present day a lot of structures of phenylamine perchlorate have been reported (Paixao, *et al.*, (1999); Wiedenfeld, *et al.*, (2004); Bendjeddou, *et al.*, 2003; Kapoor, *et al.*, (2008))). As part of our on-going studies on new anilinium perchloratecompounds, the crystal structure of the title compound (I) is reported herein.

The molecular structure of the title compound is shown in Figure 1. The asymmetric unit consists of one protonated 4-iodobenzenamine cation and one perchlorate anion. All bond lengths and bond angles correspond to the geometry parameters expected for atom types and the type of hybirdization (Allen *et al.*, 1987).

The ions are connected in three-dimensional hydrogen-bonded network *via* N—H···O hydrogen bonds. All ammonium group H atoms are involved in the hydrogen bonding with three O-atoms of neighbouring perchlorate anion and O-atom of carbonyl group of neighbouring cation (Figure 2).

S2. Experimental

The preparation of 4-iodoanilinium perchlorateis analogous to that of the compound 4-acetylanilinium perchlorate (Cinčić & Kaitner, 2007). Perchloric acid (3ml,0.16mol/L) was added to a solution of 4-iodobenzenamine (100mg) in ethanol (10ml) and the mixture was stirred for 30 min at room temperature. Colourless crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the mixed solution at room temperature after 3 days.

S3. Refinement

H atoms were placed at calculated position and were allowed to ride on the respective carrier atom with C—H = 0.93 Å, N—H = 0.86 Å.

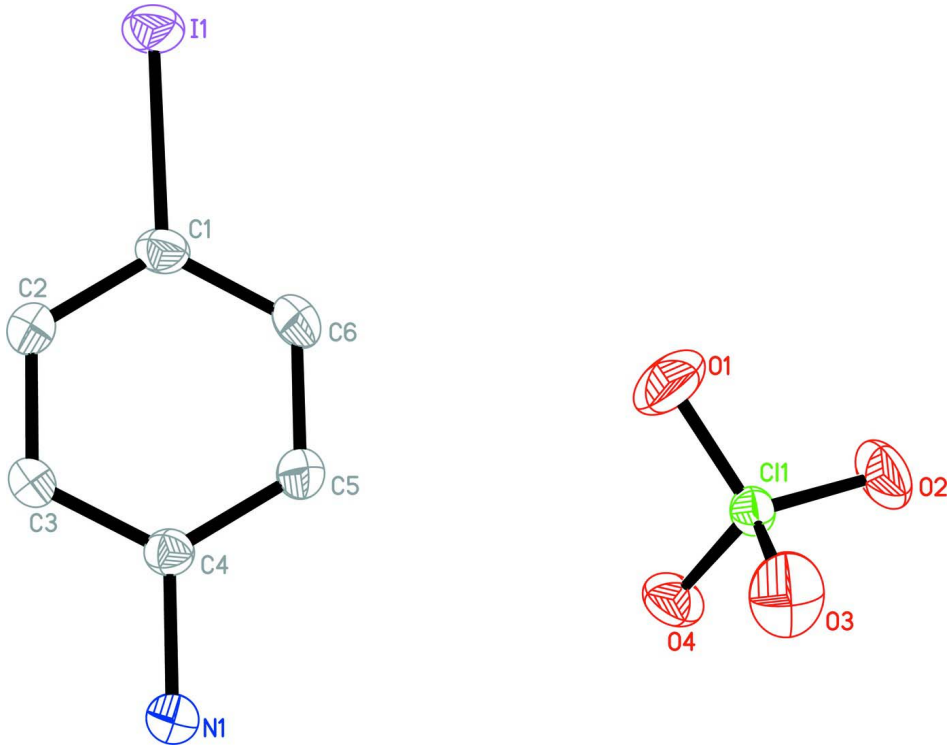

Figure 1

A partial packing diagram of the title compound, with the displacement ellipsoids were drawn at the 30% probability level.

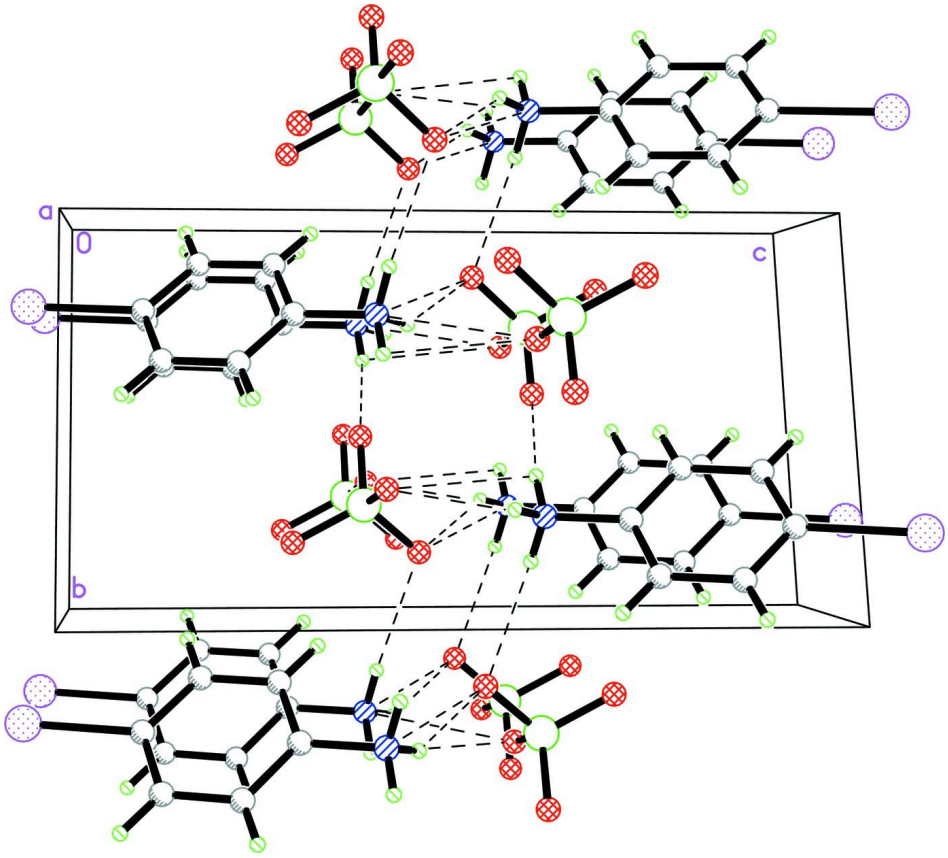

Figure 2

Packing diagram of the title compound viewed along the *a* axis. Intermolecular N—H···O hydrogen bonds are shown as dashed lines.

4-Iodoanilinium perchlorate

Crystal data

$C_6H_7IN^+·ClO_4^-$
M_r = 319.48
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 5.105 (1) Å
b = 7.2445 (14) Å
c = 13.359 (3) Å
α = 89.47 (3)°
β = 88.74 (3)°
γ = 74.61 (3)°
V = 476.22 (17) Å^3
Z = 2
$F(000)$ = 304
D_x = 2.228 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2239 reflections
θ = 2.6–27.5°
μ = 3.63 mm^{-1}
T = 298 K
Prism, colourless
0.20 × 0.20 × 0.20 mm

Data collection

Rigaku SCXmini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 13.6612 pixels mm^{-1}
CCD_Profile_fitting scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2005)
T_{min} = 0.484, T_{max} = 0.489
4945 measured reflections
2180 independent reflections
1956 reflections with $I > 2\sigma(I)$

R_{int} = 0.034
θ_{max} = 27.5°, θ_{min} = 3.1°
h = −6→6
k = −9→9
l = −17→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.071
S = 1.11
2180 reflections
119 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
$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 0.188P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.56 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.081 (3)

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}
I1	0.32432 (5)	0.26989 (3)	0.544100 (17)	0.05266 (14)
N1	−0.0662 (6)	0.2407 (4)	0.1042 (2)	0.0407 (6)
H1A	−0.1590	0.1529	0.1007	0.061*
H1B	0.0748	0.2111	0.0615	0.061*
H1C	−0.1738	0.3550	0.0885	0.061*
C4	0.0315 (6)	0.2457 (4)	0.2062 (2)	0.0323 (6)
C3	−0.0309 (7)	0.1257 (5)	0.2764 (3)	0.0436 (8)
H3A	−0.1318	0.0410	0.2600	0.052*
C1	0.2064 (7)	0.2578 (5)	0.3963 (2)	0.0371 (7)
C6	0.2694 (7)	0.3764 (5)	0.3249 (3)	0.0458 (8)
H6A	0.3722	0.4600	0.3410	0.055*
C5	0.1789 (7)	0.3708 (5)	0.2285 (3)	0.0418 (8)
H5A	0.2182	0.4518	0.1793	0.050*
C2	0.0591 (8)	0.1326 (5)	0.3724 (3)	0.0482 (9)
H2A	0.0193	0.0515	0.4214	0.058*
Cl1	0.53128 (14)	0.77646 (10)	0.10960 (6)	0.03387 (18)
O1	0.5785 (6)	0.6817 (5)	0.2029 (2)	0.0789 (10)
O2	0.6164 (6)	0.9461 (4)	0.1103 (2)	0.0662 (8)
O3	0.6814 (6)	0.6528 (4)	0.0338 (2)	0.0713 (9)
O4	0.2516 (5)	0.8204 (4)	0.0874 (2)	0.0545 (7)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0751 (2)	0.04546 (18)	0.03956 (17)	−0.01822 (13)	−0.01787 (12)	−0.00395 (11)
N1	0.0449 (15)	0.0431 (15)	0.0372 (15)	−0.0169 (13)	−0.0061 (12)	0.0021 (12)
C4	0.0333 (15)	0.0308 (15)	0.0315 (15)	−0.0063 (12)	−0.0031 (12)	−0.0006 (12)
C3	0.054 (2)	0.0471 (19)	0.0392 (18)	−0.0294 (17)	−0.0100 (15)	0.0040 (15)
C1	0.0432 (17)	0.0353 (16)	0.0326 (16)	−0.0092 (14)	−0.0074 (13)	−0.0042 (13)
C6	0.058 (2)	0.0442 (19)	0.0445 (19)	−0.0295 (17)	−0.0065 (16)	−0.0043 (15)
C5	0.056 (2)	0.0386 (17)	0.0366 (17)	−0.0218 (16)	−0.0043 (15)	0.0026 (14)
C2	0.066 (2)	0.050 (2)	0.0378 (18)	−0.0307 (19)	−0.0082 (17)	0.0097 (16)
Cl1	0.0339 (4)	0.0333 (4)	0.0364 (4)	−0.0124 (3)	−0.0025 (3)	0.0032 (3)
O1	0.078 (2)	0.098 (3)	0.0568 (18)	−0.0186 (19)	−0.0113 (16)	0.0420 (18)
O2	0.0738 (18)	0.0503 (16)	0.088 (2)	−0.0392 (15)	−0.0154 (16)	0.0023 (15)
O3	0.0726 (19)	0.0593 (18)	0.078 (2)	−0.0115 (15)	0.0276 (17)	−0.0246 (16)
O4	0.0369 (13)	0.0721 (18)	0.0562 (16)	−0.0164 (12)	−0.0093 (11)	−0.0044 (14)

Geometric parameters (Å, º)

I1—C1	2.086 (3)	C1—C6	1.368 (4)
N1—C4	1.465 (4)	C6—C5	1.382 (5)
N1—H1A	0.8900	C6—H6A	0.9300
N1—H1B	0.8900	C5—H5A	0.9300
N1—H1C	0.8900	C2—H2A	0.9300
C4—C5	1.361 (4)	Cl1—O2	1.408 (2)
C4—C3	1.362 (4)	Cl1—O1	1.412 (3)
C3—C2	1.377 (5)	Cl1—O4	1.416 (2)
C3—H3A	0.9300	Cl1—O3	1.426 (3)
C1—C2	1.366 (5)		
C4—N1—H1A	109.5	C1—C6—C5	119.4 (3)
C4—N1—H1B	109.5	C1—C6—H6A	120.3
H1A—N1—H1B	109.5	C5—C6—H6A	120.3
C4—N1—H1C	109.5	C4—C5—C6	119.3 (3)
H1A—N1—H1C	109.5	C4—C5—H5A	120.3
H1B—N1—H1C	109.5	C6—C5—H5A	120.3
C5—C4—C3	121.9 (3)	C1—C2—C3	120.6 (3)
C5—C4—N1	119.4 (3)	C1—C2—H2A	119.7
C3—C4—N1	118.7 (3)	C3—C2—H2A	119.7
C4—C3—C2	118.4 (3)	O2—Cl1—O1	110.7 (2)
C4—C3—H3A	120.8	O2—Cl1—O4	109.57 (18)
C2—C3—H3A	120.8	O1—Cl1—O4	110.10 (18)
C2—C1—C6	120.4 (3)	O2—Cl1—O3	108.95 (19)
C2—C1—I1	118.9 (2)	O1—Cl1—O3	108.8 (2)
C6—C1—I1	120.6 (2)	O4—Cl1—O3	108.73 (19)
C5—C4—C3—C2	0.0 (5)	N1—C4—C5—C6	179.6 (3)
N1—C4—C3—C2	−179.3 (3)	C1—C6—C5—C4	−0.8 (5)

C2—C1—C6—C5	1.1 (5)	C6—C1—C2—C3	−0.8 (6)
I1—C1—C6—C5	−177.3 (3)	I1—C1—C2—C3	177.6 (3)
C3—C4—C5—C6	0.3 (5)	C4—C3—C2—C1	0.3 (6)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O2^{i}	0.89	2.12	3.002 (4)	174
N1—H1*B*···O3^{ii}	0.89	2.17	2.911 (4)	141
N1—H1*C*···O3^{iii}	0.89	2.21	3.069 (4)	162

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