

Acta Crystallographica Section E

## Structure Reports

Online

ISSN 1600-5368

## 4-Iodoanilinium perchlorate

Weiwei SiMa

Ordered Matter Science Research Center, College of Chemistry and Chemical Engineering, Southeast University, Nanjing 210096, People's Republic of China  
Correspondence e-mail: nysima@126.com

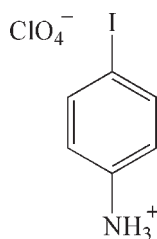
Received 27 July 2009; accepted 12 March 2010

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.030;  $wR$  factor = 0.071; data-to-parameter ratio = 18.3.

In the crystal structure of the title compound,  $\text{C}_6\text{H}_7\text{IN}^+\cdot\text{ClO}_4^-$ , the ions are connected in a three-dimensional hydrogen-bonded network via  $\text{N}-\text{H}\cdots\text{O}$  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

$\text{C}_6\text{H}_7\text{IN}^+\cdot\text{ClO}_4^-$   
 $M_r = 319.48$   
Triclinic,  $P\bar{1}$   
 $a = 5.105$  (1) Å  
 $b = 7.2445$  (14) Å  
 $c = 13.359$  (3) Å  
 $\alpha = 89.47$  (3)°  
 $\beta = 88.74$  (3)°

$\gamma = 74.61$  (3)°  
 $V = 476.22$  (17) Å<sup>3</sup>  
 $Z = 2$   
Mo  $K\alpha$  radiation  
 $\mu = 3.63$  mm<sup>-1</sup>  
 $T = 298$  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  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.034$

## 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  
 $\Delta\rho_{\text{max}} = 0.56$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.60$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{O2}^{\text{ii}}$	0.89	2.12	3.002 (4)	174
$\text{N1}-\text{H1B}\cdots\text{O3}^{\text{iii}}$	0.89	2.17	2.911 (4)	141
$\text{N1}-\text{H1C}\cdots\text{O3}^{\text{iii}}$	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).

## References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans 2*, pp. S1–19.
- Bendjeddou, L., Cherouana, A., Berrah, F. & Benali-Cherif, N. (2003). *Acta Cryst. E* **59**, o574–o576.
- Cinčić, D. & Kaitner, B. (2007). *Acta Cryst. E* **63**, o4672.
- Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
- Kapoor, I. P. S., Srivastava, P., Singh, G. & Frohlich, R. (2008). *J. Phys. Chem. A*, **112**, 652–659.
- Paixao, J. A., Matos Bejia, A., Ramos Silva, M., Alte da Veiga, L. & Martin-Gil, J. (1999). *Z. Kristallogr. New Cryst. Struct.* **214**, 85–86.
- Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Wiedenfeld, D., Minton, M., Nesterov, V. & Montoya, C. (2004). *J. Chem. Crystallogr.* **34**, 95–101.

**supplementary materials**

*Acta Cryst.* (2010). E66, o895 [ doi:10.1107/S1600536810009347 ]

## 4-Iodoanilinium perchlorate

W. SiMa

### 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 perchlorate compounds, 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 hybridization (Allen *et al.*, 1987).

The ions are connected in three-dimensional hydrogen-bonded network *via* N—H $\cdots$ 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).

### Experimental

The preparation of 4-iodoanilinium perchlorate is 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-iodobenzeneamine (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.

### 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.

### Figures

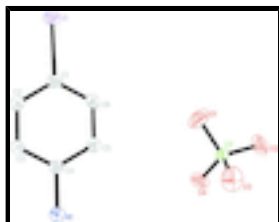


Fig. 1. A partial packing diagram of the title compound, with the displacement ellipsoids were drawn at the 30% probability level.

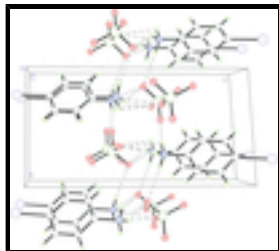


Fig. 2. Packing diagram of the title compound viewed along the *a* axis. Intermolecular N—H...O hydrogen bonds are shown as dashed lines.

## 4-Iodoanilinium perchlorate

### Crystal data

$C_6H_7IN^+ \cdot ClO_4^-$	$Z = 2$
$M_r = 319.48$	$F(000) = 304$
Triclinic, <i>PT</i>	$D_x = 2.228 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 5.105 (1) \text{ \AA}$	Cell parameters from 2239 reflections
$b = 7.2445 (14) \text{ \AA}$	$\theta = 2.6\text{--}27.5^\circ$
$c = 13.359 (3) \text{ \AA}$	$\mu = 3.63 \text{ mm}^{-1}$
$\alpha = 89.47 (3)^\circ$	$T = 298 \text{ K}$
$\beta = 88.74 (3)^\circ$	Prism, colourless
$\gamma = 74.61 (3)^\circ$	$0.20 \times 0.20 \times 0.20 \text{ mm}$
$V = 476.22 (17) \text{ \AA}^3$	

### Data collection

Rigaku SCXmini diffractometer	2180 independent reflections
Radiation source: fine-focus sealed tube graphite	1956 reflections with $I > 2\sigma(I)$
Detector resolution: $13.6612 \text{ pixels mm}^{-1}$	$R_{\text{int}} = 0.034$
CCD_Profile_fitting scans	$\theta_{\text{max}} = 27.5^\circ$ , $\theta_{\text{min}} = 3.1^\circ$
Absorption correction: multi-scan (CrystalClear; Rigaku, 2005)	$h = -6 \rightarrow 6$
$T_{\text{min}} = 0.484$ , $T_{\text{max}} = 0.489$	$k = -9 \rightarrow 9$
4945 measured reflections	$l = -17 \rightarrow 17$

### Refinement

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.030$	H-atom parameters constrained
$wR(F^2) = 0.071$	$w = 1/[\sigma^2(F_o^2) + (0.0248P)^2 + 0.188P]$
$S = 1.11$	where $P = (F_o^2 + 2F_c^2)/3$
	$(\Delta/\sigma)_{\text{max}} < 0.001$

2180 reflections	$\Delta\rho_{\max} = 0.56 \text{ e } \text{\AA}^{-3}$
119 parameters	$\Delta\rho_{\min} = -0.60 \text{ e } \text{\AA}^{-3}$
0 restraints	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	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 ( $\text{\AA}^2$ )*

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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*
C11	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 ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
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)

## supplementary materials

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)
C11	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 ( $\text{\AA}$ , $^\circ$ )

11—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)	C11—O2	1.408 (2)
C4—C3	1.362 (4)	C11—O1	1.412 (3)
C3—C2	1.377 (5)	C11—O4	1.416 (2)
C3—H3A	0.9300	C11—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—C11—O1	110.7 (2)
C4—C3—H3A	120.8	O2—C11—O4	109.57 (18)
C2—C3—H3A	120.8	O1—C11—O4	110.10 (18)
C2—C1—C6	120.4 (3)	O2—C11—O3	108.95 (19)
C2—C1—11	118.9 (2)	O1—C11—O3	108.8 (2)
C6—C1—11	120.6 (2)	O4—C11—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)
11—C1—C6—C5	-177.3 (3)	11—C1—C2—C3	177.6 (3)
C3—C4—C5—C6	0.3 (5)	C4—C3—C2—C1	0.3 (6)

### Hydrogen-bond geometry ( $\text{\AA}$ , $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1A $\cdots$ O2 <sup>i</sup>	0.89	2.12	3.002 (4)	174.

N1—H1B···O3 <sup>ii</sup>	0.89	2.17	2.911 (4)	141.
N1—H1C···O3 <sup>iii</sup>	0.89	2.21	3.069 (4)	162.

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

Fig. 1

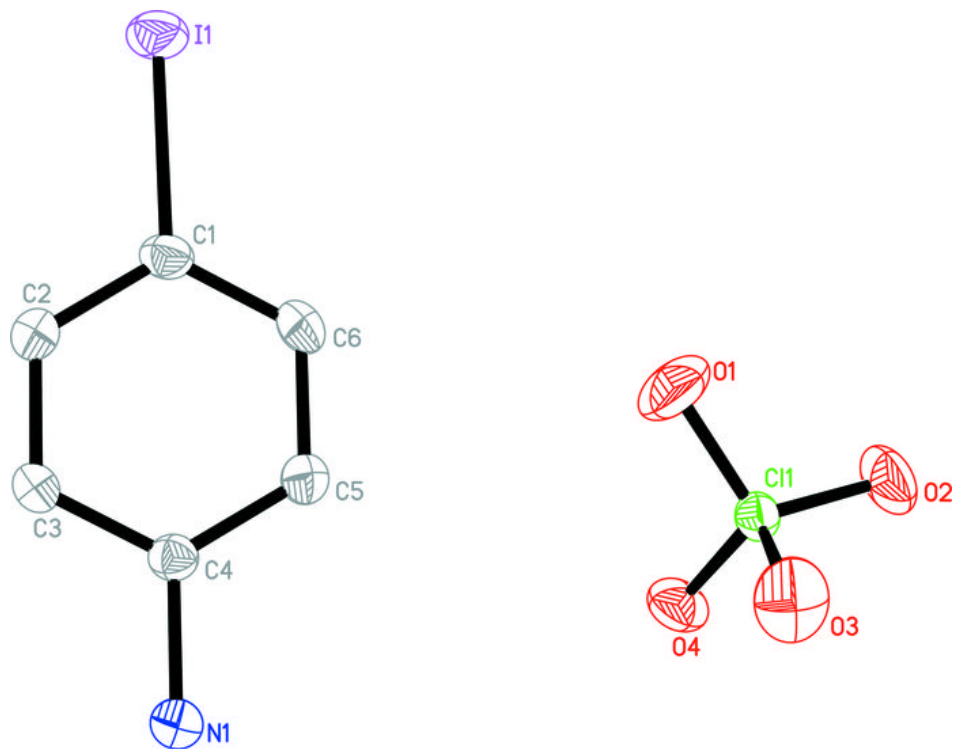




Fig. 2

