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1-(4-Chlorobenzylideneamino)pyridinium iodide

Yong-Tao Cui, Jian-Qiang Wang, Chun-Xiang Ji, Hai-Bo Wang and Guo Cheng*

College of Science, Nanjing University of Technology, Xinfan Road No. 5 Nanjing, Nanjing 210009, People's Republic of China

Correspondence e-mail: guocheng@njut.edu.cn

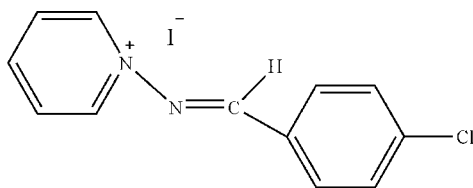
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.027; wR factor = 0.093; data-to-parameter ratio = 15.2.

In the title compound, $\text{C}_{12}\text{H}_{10}\text{ClN}_2^+\cdot\text{I}^-$, the aromatic rings are oriented at a dihedral angle of $54.55(3)^\circ$. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{I}$ and $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds link the molecules.

Related literature

For background, see: Okamoto *et al.* (1967). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{10}\text{ClN}_2^+\cdot\text{I}^-$ $M_r = 344.57$ Triclinic, $P\bar{1}$ $a = 6.5105(14)$ Å $b = 7.1748(15)$ Å $c = 14.223(3)$ Å $\alpha = 76.893(3)^\circ$ $\beta = 79.183(3)^\circ$ $\gamma = 80.753(3)^\circ$ $V = 630.7(2)$ Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 2.72$ mm⁻¹ $T = 291(2)$ K $0.10 \times 0.10 \times 0.08$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer

Absorption correction: ψ scan (North *et al.*, 1968) $T_{\min} = 0.772$, $T_{\max} = 0.812$

3220 measured reflections

2205 independent reflections

1892 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.063$

3 standard reflections

frequency: 120 min

intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.027$ $wR(F^2) = 0.093$ $S = 1.05$

2205 reflections

145 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.53$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.63$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C1}-\text{H1}\cdots\text{I1}$	0.93	3.04	3.857 (5)	147
$\text{C5}-\text{H5}\cdots\text{Cl1}^\dagger$	0.93	2.79	3.691 (6)	162

Symmetry code: (i) $-x, -y, -z + 1$.

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); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2600).

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supplementary materials

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1-(4-Chlorobenzylideneamino)pyridinium iodide

Y.-T. Cui, J.-Q. Wang, C.-X. Ji, H.-B. Wang and G. Cheng

Comment

Some derivatives of 1-aminopyridinium iodide 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/C1-C5) and B (C7-C12) are, of course, planar, and they are oriented at a dihedral angle of 54.55 (3)°.

In the crystal structure, intramolecular C-H...I and intermolecular C-H...Cl hydrogen bonds (Table 1) link the molecules (Fig. 2), in which they may be effective in the stabilization of the structure. The π - π contacts between the pyridine rings and the benzene rings, Cg1—Cg1ⁱ and Cg2—Cg2ⁱⁱ [symmetry codes: (i) 1 - x, -y, -z; (ii) 1 - x, -y, 1 - z, where Cg1 and Cg2 are centroids of the rings A (N1/C1-C5) and B (C7-C12), respectively] may further stabilize the structure, with centroid-centroid distances of 4.130 (3) Å and 4.056 (3) Å.

Experimental

For the preparation of the title compound, 1-aminopyridinium iodide (22.2 g, 0.10 mol) was dissolved in ethanol (20 ml), 4-methylbenzaldehyde (12.4 g, 0.1 mol) was added with stirring, and then the mixture was heated at reflux for 5 h. Upon cooling to room temperature, a precipitate formed, which was collected by filtration and washed with cold ethanol (2 X 10 ml) to obtain a yellow solid (yield; 24.0 g, 70%). Crystals suitable for X-ray analysis were obtained by slow evaporation of an ethanol solution.

Refinement

H atoms were positioned geometrically, with C-H = 0.93 Å for aromatic and methine H and constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

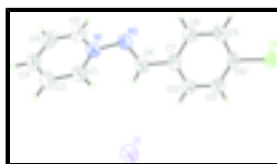


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

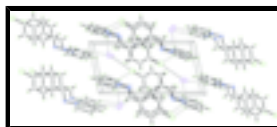


Fig. 2. A partial packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

1-(4-Chlorobenzylideneamino)pyridinium iodide

Crystal data

$C_{12}H_{10}ClN_2^+I^-$	$Z = 2$
$M_r = 344.57$	$F_{000} = 332$
Triclinic, $P\bar{1}$	$D_x = 1.814 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 6.5105 (14) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 7.1748 (15) \text{ \AA}$	Cell parameters from 25 reflections
$c = 14.223 (3) \text{ \AA}$	$\theta = 2.1\text{--}25.3^\circ$
$\alpha = 76.893 (3)^\circ$	$\mu = 2.72 \text{ mm}^{-1}$
$\beta = 79.183 (3)^\circ$	$T = 291 (2) \text{ K}$
$\gamma = 80.753 (3)^\circ$	Block, yellow
$V = 630.7 (2) \text{ \AA}^3$	$0.10 \times 0.10 \times 0.08 \text{ mm}$

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.063$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 25.0^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 2.9^\circ$
$T = 291(2) \text{ K}$	$h = -7 \rightarrow 7$
$\omega/2\theta$ scans	$k = -8 \rightarrow 8$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$l = -15 \rightarrow 16$
$T_{\text{min}} = 0.772$, $T_{\text{max}} = 0.812$	3 standard reflections
3220 measured reflections	every 120 min
2205 independent reflections	intensity decay: none
1892 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.027$	H-atom parameters constrained
$wR(F^2) = 0.093$	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2205 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
145 parameters	$\Delta\rho_{\text{max}} = 0.53 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{\text{min}} = -0.63 \text{ e \AA}^{-3}$
	Extinction correction: none

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
I1	0.91966 (5)	0.25912 (5)	0.16564 (2)	0.05410 (17)
Cl1	0.1793 (2)	0.4285 (2)	0.64323 (10)	0.0562 (3)
N1	0.4087 (6)	-0.1676 (5)	0.2056 (3)	0.0410 (9)
N2	0.3499 (6)	-0.1179 (6)	0.3000 (3)	0.0434 (9)
C1	0.6038 (8)	-0.1437 (7)	0.1537 (4)	0.0495 (12)
H1	0.7003	-0.0912	0.1775	0.059*
C2	0.6541 (10)	-0.1996 (7)	0.0648 (4)	0.0608 (15)
H2	0.7863	-0.1852	0.0274	0.073*
C3	0.5076 (10)	-0.2770 (7)	0.0313 (4)	0.0585 (14)
H3	0.5404	-0.3131	-0.0291	0.070*
C4	0.3107 (11)	-0.3012 (8)	0.0878 (4)	0.0654 (16)
H4	0.2111	-0.3533	0.0658	0.079*
C5	0.2669 (9)	-0.2469 (7)	0.1763 (4)	0.0562 (13)
H5	0.1380	-0.2654	0.2160	0.067*
C6	0.3740 (8)	0.0561 (7)	0.2976 (3)	0.0451 (11)
H6	0.4240	0.1326	0.2382	0.054*
C7	0.3254 (7)	0.1391 (7)	0.3855 (3)	0.0415 (11)
C8	0.2488 (7)	0.0346 (7)	0.4776 (3)	0.0442 (11)
H8	0.2289	-0.0937	0.4851	0.053*
C9	0.2026 (8)	0.1216 (7)	0.5574 (3)	0.0474 (12)
H9	0.1510	0.0536	0.6189	0.057*
C10	0.2355 (7)	0.3145 (7)	0.5436 (3)	0.0424 (11)
C11	0.3049 (8)	0.4181 (7)	0.4539 (3)	0.0479 (12)
H11	0.3202	0.5476	0.4461	0.058*
C12	0.3526 (8)	0.3297 (7)	0.3745 (3)	0.0460 (11)
H12	0.4033	0.3992	0.3132	0.055*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.0580 (3)	0.0587 (3)	0.0457 (2)	-0.01153 (17)	-0.00263 (16)	-0.01233 (16)
Cl1	0.0519 (8)	0.0723 (9)	0.0497 (7)	-0.0091 (6)	-0.0010 (6)	-0.0280 (6)

supplementary materials

N1	0.041 (2)	0.043 (2)	0.039 (2)	-0.0075 (18)	-0.0030 (18)	-0.0090 (17)
N2	0.045 (2)	0.048 (2)	0.038 (2)	-0.0064 (18)	-0.0026 (17)	-0.0132 (17)
C1	0.055 (3)	0.048 (3)	0.043 (3)	-0.004 (2)	-0.004 (2)	-0.009 (2)
C2	0.076 (4)	0.050 (3)	0.046 (3)	0.001 (3)	0.009 (3)	-0.010 (2)
C3	0.083 (4)	0.047 (3)	0.044 (3)	-0.009 (3)	0.000 (3)	-0.016 (2)
C4	0.094 (5)	0.050 (3)	0.061 (4)	-0.006 (3)	-0.023 (3)	-0.021 (3)
C5	0.060 (4)	0.055 (3)	0.055 (3)	-0.009 (3)	-0.008 (3)	-0.014 (3)
C6	0.050 (3)	0.049 (3)	0.036 (3)	0.001 (2)	-0.008 (2)	-0.010 (2)
C7	0.037 (3)	0.050 (3)	0.038 (3)	0.003 (2)	-0.008 (2)	-0.014 (2)
C8	0.041 (3)	0.046 (3)	0.043 (3)	-0.004 (2)	-0.003 (2)	-0.009 (2)
C9	0.047 (3)	0.056 (3)	0.036 (3)	-0.007 (2)	0.000 (2)	-0.008 (2)
C10	0.034 (3)	0.056 (3)	0.041 (3)	-0.006 (2)	-0.006 (2)	-0.017 (2)
C11	0.055 (3)	0.044 (3)	0.046 (3)	-0.008 (2)	-0.009 (2)	-0.009 (2)
C12	0.052 (3)	0.047 (3)	0.037 (3)	-0.010 (2)	-0.006 (2)	-0.003 (2)

Geometric parameters (Å, °)

C11—C10	1.746 (5)	C5—H5	0.9300
N1—C5	1.331 (7)	C6—C7	1.465 (6)
N1—C1	1.359 (6)	C6—H6	0.9300
N1—N2	1.434 (5)	C7—C12	1.377 (6)
N2—C6	1.275 (6)	C7—C8	1.399 (6)
C1—C2	1.379 (7)	C8—C9	1.378 (6)
C1—H1	0.9300	C8—H8	0.9300
C2—C3	1.380 (8)	C9—C10	1.399 (7)
C2—H2	0.9300	C9—H9	0.9300
C3—C4	1.393 (9)	C10—C11	1.358 (7)
C3—H3	0.9300	C11—C12	1.380 (6)
C4—C5	1.367 (7)	C11—H11	0.9300
C4—H4	0.9300	C12—H12	0.9300
C5—N1—C1	123.4 (4)	N2—C6—H6	119.0
C5—N1—N2	116.0 (4)	C7—C6—H6	119.0
C1—N1—N2	120.4 (4)	C12—C7—C8	119.8 (4)
C6—N2—N1	112.2 (4)	C12—C7—C6	117.4 (4)
N1—C1—C2	118.0 (5)	C8—C7—C6	122.8 (4)
N1—C1—H1	121.0	C9—C8—C7	120.1 (4)
C2—C1—H1	121.0	C9—C8—H8	119.9
C1—C2—C3	119.7 (5)	C7—C8—H8	119.9
C1—C2—H2	120.1	C8—C9—C10	118.4 (4)
C3—C2—H2	120.1	C8—C9—H9	120.8
C2—C3—C4	120.2 (5)	C10—C9—H9	120.8
C2—C3—H3	119.9	C11—C10—C9	121.8 (4)
C4—C3—H3	119.9	C11—C10—C11	118.7 (4)
C5—C4—C3	118.6 (5)	C9—C10—C11	119.5 (4)
C5—C4—H4	120.7	C10—C11—C12	119.5 (4)
C3—C4—H4	120.7	C10—C11—H11	120.2
N1—C5—C4	120.0 (5)	C12—C11—H11	120.2
N1—C5—H5	120.0	C7—C12—C11	120.4 (4)
C4—C5—H5	120.0	C7—C12—H12	119.8

N2—C6—C7

122.0 (4)

C11—C12—H12

119.8

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C1—H1 \cdots I1	0.93	3.04	3.857 (5)	147
C5—H5 \cdots C11 ⁱ	0.93	2.79	3.691 (6)	162

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

Fig. 1

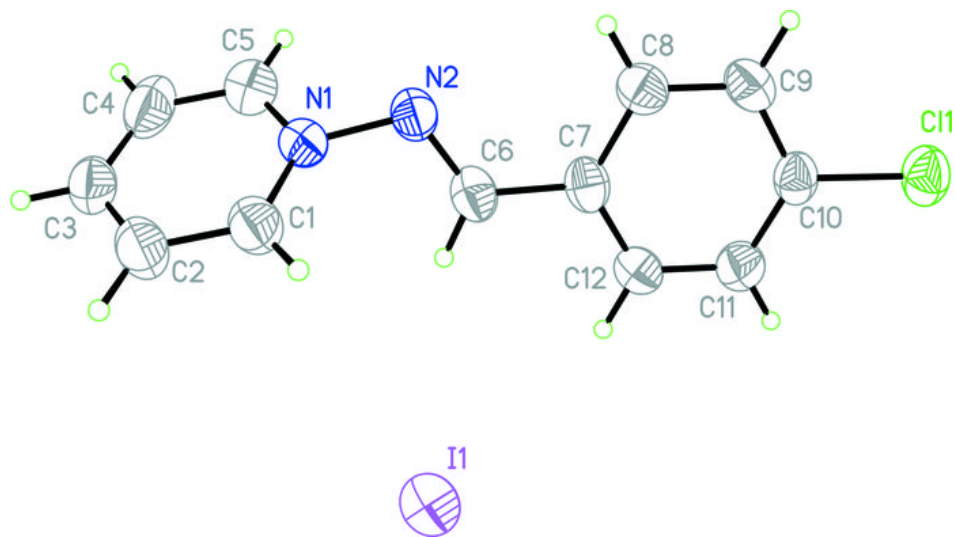


Fig. 2

