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1-(Benzylideneamino)pyridinium iodide

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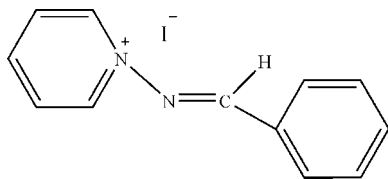
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; R factor = 0.030; wR factor = 0.075; data-to-parameter ratio = 15.7.

In the title compound, $\text{C}_{12}\text{H}_{11}\text{N}_2^+\cdot\text{I}^-$, the aromatic rings are oriented at a dihedral angle of $73.40(3)^\circ$. In the crystal structure, π - π contacts between the pyridine rings and the benzene and pyridine rings [centroid-centroid distances = $3.548(3)$ and $4.211(3)$ Å] may stabilize the structure.

Related literature

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



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{11}\text{N}_2^+\cdot\text{I}^-$ $M_r = 310.13$ Monoclinic, $P2_1/c$ $a = 10.5722(17)$ Å $b = 7.8219(13)$ Å $c = 15.386(3)$ Å $\beta = 108.354(2)^\circ$ $V = 1207.6(4)$ Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 2.62$ mm⁻¹ $T = 291(2)$ K $0.13 \times 0.12 \times 0.10$ mm

Data collection

Enraf-Nonius CAD-4 diffractometer

Absorption correction: ψ scan (North *et al.*, 1968) $T_{\min} = 0.727$, $T_{\max} = 0.780$

5768 measured reflections

2133 independent reflections

1713 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.067$

3 standard reflections

frequency: 120 min

intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.030$ $wR(F^2) = 0.075$ $S = 0.94$

2133 reflections

136 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 0.50$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.53$ e Å⁻³

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: HK2601).

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supplementary materials

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1-(Benzylideneamino)pyridinium iodide

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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 73.40 (3)°.

In the crystal structure, π - π contacts between the pyridine and the benzene rings and the pyridine rings, Cg1—Cg2ⁱ and Cg1—Cg1ⁱⁱ [symmetry codes: (i) $x, 3/2 - y, z - 1/2$; (ii) $1 - x, 2 - y, -z$, where Cg1 and Cg2 are centroids of the rings A (N1/C1-C5) and B (C7-C12), respectively] may stabilize the structure, with centroid-centroid distances of 3.548 (3) Å and 4.211 (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), benzaldehyde (10.6 g, 0.10 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; 21.7 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

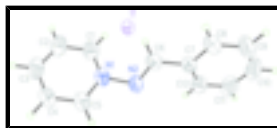


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

1-(Benzylideneamino)pyridinium iodide

Crystal data

$\text{C}_{12}\text{H}_{11}\text{N}_2^+\cdot\text{I}^-$

$F_{000} = 600$

supplementary materials

$M_r = 310.13$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.5722$ (17) Å

$b = 7.8219$ (13) Å

$c = 15.386$ (3) Å

$\beta = 108.354$ (2)°

$V = 1207.6$ (4) Å³

$Z = 4$

$D_x = 1.706$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 2.1$ – 25.3 °

$\mu = 2.62$ mm⁻¹

$T = 291$ (2) K

Block, yellow

$0.13 \times 0.12 \times 0.10$ mm

Data collection

Enraf–Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 291$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.727$, $T_{\max} = 0.780$

5768 measured reflections

2133 independent reflections

1713 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.067$

$\theta_{\text{max}} = 25.0$ °

$\theta_{\text{min}} = 2.8$ °

$h = -12 \rightarrow 12$

$k = -9 \rightarrow 7$

$l = -18 \rightarrow 18$

3 standard reflections

every 120 min

intensity decay: none

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.075$

$S = 0.94$

2133 reflections

136 parameters

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

$$w = 1/[\sigma^2(F_o^2) + (0.0365P)^2]$$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.50$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.52$ e Å⁻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	0.36542 (2)	0.37261 (3)	0.175113 (16)	0.05509 (13)
N1	0.2501 (3)	0.8944 (3)	0.03899 (19)	0.0462 (7)
N2	0.1744 (3)	0.8956 (3)	0.10105 (19)	0.0482 (7)
C1	0.3602 (4)	0.7989 (5)	0.0544 (3)	0.0543 (9)
H1	0.3942	0.7393	0.1091	0.065*
C2	0.4222 (4)	0.7904 (5)	-0.0117 (3)	0.0597 (10)
H2	0.4975	0.7225	-0.0024	0.072*
C3	0.3734 (4)	0.8812 (5)	-0.0909 (3)	0.0586 (10)
H3	0.4162	0.8775	-0.1353	0.070*
C4	0.2612 (4)	0.9776 (6)	-0.1045 (2)	0.0633 (10)
H4	0.2274	1.0402	-0.1582	0.076*
C5	0.1982 (4)	0.9821 (5)	-0.0388 (2)	0.0617 (10)
H5	0.1205	1.0454	-0.0484	0.074*
C6	0.2447 (3)	0.9324 (4)	0.1829 (2)	0.0439 (8)
H6	0.3347	0.9579	0.1961	0.053*
C7	0.1831 (3)	0.9341 (5)	0.2553 (2)	0.0447 (8)
C8	0.2456 (4)	1.0226 (5)	0.3354 (2)	0.0584 (9)
H8	0.3250	1.0802	0.3419	0.070*
C9	0.0654 (4)	0.8452 (5)	0.2465 (3)	0.0566 (10)
H9	0.0240	0.7838	0.1934	0.068*
C10	0.0110 (4)	0.8488 (5)	0.3166 (3)	0.0687 (12)
H10	-0.0678	0.7902	0.3109	0.082*
C11	0.0729 (5)	0.9391 (6)	0.3955 (3)	0.0748 (13)
H11	0.0352	0.9419	0.4425	0.090*
C12	0.1899 (5)	1.0252 (6)	0.4051 (3)	0.0716 (12)
H12	0.2315	1.0851	0.4588	0.086*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.05786 (18)	0.0622 (2)	0.04976 (17)	-0.00118 (11)	0.02344 (12)	0.00535 (11)
N1	0.0537 (16)	0.0507 (17)	0.0363 (15)	-0.0039 (13)	0.0172 (13)	-0.0040 (13)
N2	0.0509 (16)	0.0610 (18)	0.0355 (15)	-0.0047 (13)	0.0176 (13)	-0.0016 (14)
C1	0.064 (2)	0.050 (2)	0.052 (2)	0.0022 (18)	0.0222 (18)	0.0048 (18)
C2	0.066 (2)	0.055 (2)	0.067 (3)	0.0024 (19)	0.034 (2)	-0.005 (2)
C3	0.069 (2)	0.066 (2)	0.050 (2)	-0.012 (2)	0.0314 (19)	-0.013 (2)
C4	0.067 (2)	0.082 (3)	0.0402 (19)	0.001 (2)	0.0172 (18)	0.010 (2)
C5	0.061 (2)	0.083 (3)	0.043 (2)	0.010 (2)	0.0183 (17)	0.004 (2)
C6	0.0466 (18)	0.0441 (19)	0.0398 (19)	0.0008 (14)	0.0118 (15)	0.0004 (15)

supplementary materials

C7	0.0471 (19)	0.0505 (19)	0.0351 (18)	0.0047 (15)	0.0107 (15)	0.0027 (16)
C8	0.061 (2)	0.070 (3)	0.0405 (19)	0.0007 (19)	0.0111 (17)	-0.0024 (19)
C9	0.054 (2)	0.068 (3)	0.050 (2)	-0.0020 (17)	0.0190 (18)	0.0039 (18)
C10	0.067 (3)	0.078 (3)	0.074 (3)	0.003 (2)	0.040 (2)	0.012 (2)
C11	0.097 (3)	0.082 (3)	0.062 (3)	0.033 (3)	0.049 (3)	0.019 (3)
C12	0.098 (3)	0.078 (3)	0.039 (2)	0.015 (3)	0.022 (2)	-0.004 (2)

Geometric parameters (Å, °)

N1—N2	1.426 (4)	C6—C7	1.457 (4)
C1—N1	1.340 (4)	C6—H6	0.9300
C1—C2	1.375 (5)	C7—C8	1.385 (5)
C1—H1	0.9300	C7—C9	1.394 (5)
C2—C3	1.364 (5)	C8—C12	1.377 (5)
C2—H2	0.9300	C8—H8	0.9300
C3—C4	1.365 (5)	C9—C10	1.374 (6)
C3—H3	0.9300	C9—H9	0.9300
C4—C5	1.374 (5)	C10—C11	1.378 (7)
C4—H4	0.9300	C10—H10	0.9300
C5—N1	1.339 (4)	C11—C12	1.375 (6)
C5—H5	0.9300	C11—H11	0.9300
C6—N2	1.277 (4)	C12—H12	0.9300
C5—N1—C1	122.2 (3)	N2—C6—H6	120.2
C5—N1—N2	116.0 (3)	C7—C6—H6	120.2
C1—N1—N2	121.6 (3)	C8—C7—C9	119.8 (3)
C6—N2—N1	112.8 (3)	C8—C7—C6	118.8 (3)
N1—C1—C2	119.1 (4)	C9—C7—C6	121.4 (3)
N1—C1—H1	120.4	C12—C8—C7	120.0 (4)
C2—C1—H1	120.4	C12—C8—H8	120.0
C3—C2—C1	120.1 (4)	C7—C8—H8	120.0
C3—C2—H2	119.9	C10—C9—C7	119.7 (4)
C1—C2—H2	119.9	C10—C9—H9	120.1
C2—C3—C4	119.3 (4)	C7—C9—H9	120.1
C2—C3—H3	120.3	C9—C10—C11	120.1 (4)
C4—C3—H3	120.3	C9—C10—H10	120.0
C3—C4—C5	120.1 (4)	C11—C10—H10	120.0
C3—C4—H4	119.9	C12—C11—C10	120.5 (4)
C5—C4—H4	119.9	C12—C11—H11	119.7
N1—C5—C4	119.2 (4)	C10—C11—H11	119.7
N1—C5—H5	120.4	C11—C12—C8	119.9 (4)
C4—C5—H5	120.4	C11—C12—H12	120.0
N2—C6—C7	119.7 (3)	C8—C12—H12	120.0

Fig. 1

