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1-(4-Iodobutyl)pyrimidin-1-ium iodide

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.004 Å
R factor = 0.023
wR factor = 0.043
Data-to-parameter ratio = 24.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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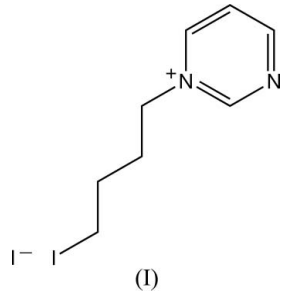
The title molecular salt, $C_8H_{12}IN_2^+\cdot I^-$, features weak C—H···N and C—H···I interactions in the crystal structure.

Comment

As part of our investigations of substituted pyrimidines (Brown, 1994), the title compound, (I), $C_8H_{12}IN_2^+\cdot I^-$, has been synthesized and structurally characterized (Fig. 1). Compound (I) possesses normal geometrical parameters (Allen *et al.*, 1987). The pyrimidine ring (C5–C8/N1/N2; centroid *Cg*) is almost planar (r.m.s. deviation from the mean plane = 0.007 Å). The bond-angle sum at N1 of 360.0° implies the expected sp^2-hybridization. The dihedral angle between the aromatic ring and the mean plane of the side-chain atoms (C1–C4/I1) is 53.63 (11)°.



(I)

A *PLATON* (Spek, 2003) analysis of (I) identified some short C—H···N and C—H···I interactions (Table 1) that might influence the crystal packing. Conversely, there are no π–π stacking interactions in (I), the shortest *Cg*···*Cg* separation being greater than 5.2 Å. The shortest I1···I2 contact of 3.7418 (3) Å in (I) is significantly less than the Bondi (1964) van der Waals I···I contact distance of 3.96 Å.

Experimental

Pyrimidine (2.50 mmol, 0.200 g) was carefully added to dry acetonitrile (60 ml) with stirring under nitrogen. The flask was degassed to remove any air and was stirred for 10 min. 1,4-Diiodobutane (10.1 mmol, 3.15 g) was then slowly added to the solution and the mixture was refluxed at 363 K for 8 h, monitoring the product using thin-layer chromatography (1:1 *v*/*v*, methanol, ethyl acetate, $R_f = 0.5$). The reaction vessel was covered with aluminium foil, as 1,4-diiodobutane is light-sensitive.

To ensure the complete consumption of pyrimidine, the reaction was stirred for a further 48 h. After this time, the reaction mixture was cooled to room temperature, revealing an orange crystalline product. The crystals were filtered off, washed with cold ethyl acetate (2 × 5 ml) and placed under reduced pressure to dry, yielding (I). The crystal quality was poor and not suitable for X-ray data collection.

The remaining filtrate was reduced *in vacuo* and washed with ethyl acetate (3 × 10 ml) to remove the excess 1,4-diiodobutane, producing an orange solid. This was dissolved in hot acetonitrile (20 ml), and recrystallization, initialized by a few drops of cold ethyl acetate, yielded orange rosettes in intergrown plates crystals of (I). The overall yield of both batches was 0.534 g (52%), m.p. 410–412 K. ν_{max} (KBr, cm^{-1}) 678 (alkyl-I), 817 (isolated aryl-H), 1431 (CH_2), 1619 (C═N, conjugated, cyclic), 2920 (CH_2), 3048 (CH-halogen).

Crystal data

$C_8H_{12}IN_2^+\cdot I^-$
M_r = 390.00
Monoclinic, $P2_1/c$
a = 16.9178 (6) Å
b = 9.1694 (3) Å
c = 7.6303 (2) Å
β = 97.329 (2)°
V = 1173.99 (6) Å^3
Z = 4
D_x = 2.207 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 5.32 mm^{-1}
T = 120 (2) K
Plate, orange
0.22 × 0.18 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
T_{min} = 0.388, T_{max} = 0.900
12760 measured reflections
2674 independent reflections
2302 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.043
S = 1.09
2674 reflections
110 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0082P)^2 + 0.8187P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.66 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00174 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6···N2^{i}	0.95	2.58	3.396 (4)	144
C7—H7···I2^{ii}	0.95	3.03	3.952 (3)	164
C8—H8···I2^{iii}	0.95	3.04	3.792 (3)	138

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

All H atoms were placed in calculated positions with C—H = 0.95–0.99 Å and refined as riding with $U_{iso}(H) = 1.2U_{eq}(C)$.

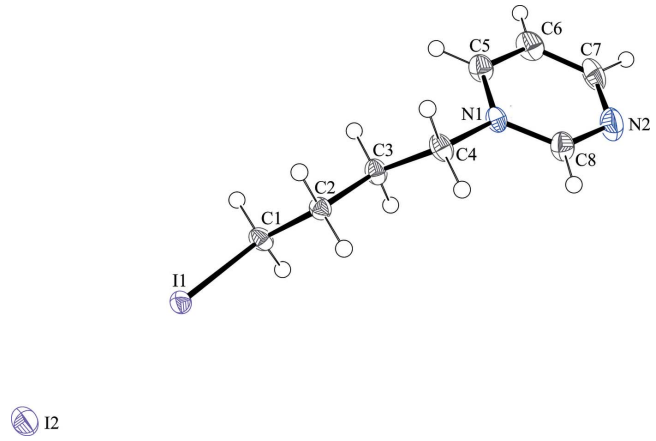


Figure 1
View of the molecular structure of (I) showing the atom labelling and 50% probability displacement ellipsoids.

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* and *DENZO* (Otwinowski & Minor, 1997), and *SORTAV* (Blessing, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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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.
Blessing, R. H. (1995). *Acta Cryst.* A**51**, 33–38.
Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.
Brown, D. J. (1994). *The Pyrimidines*. New York: Wiley-Interscience.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Nonius (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2001). *SADABS*. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

supporting information

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Hall symbol: -P 2ybc
$a = 16.9178\ (6)$ Å
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$c = 7.6303\ (2)$ Å
$\beta = 97.329\ (2)°$
$V = 1173.99\ (6)$ Å^3
$Z = 4$
$F(000) = 720$
$D_x = 2.207$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2769 reflections
$\theta = 2.9–27.5°$
$\mu = 5.32$ mm^{-1}
$T = 120$ K
Slab, orange
0.22 × 0.18 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)
$T_{min} = 0.388$, $T_{max} = 0.900$
12760 measured reflections
2674 independent reflections
2302 reflections with $I > 2\sigma(I)$
$R_{int} = 0.037$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$
$h = -21\rightarrow21$
$k = -11\rightarrow11$
$l = -8\rightarrow9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.023$
$wR(F^2) = 0.043$
$S = 1.09$
2674 reflections
110 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: none
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0082P)^2 + 0.8187P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.78$ e Å^{-3}
$\Delta\rho_{min} = -0.66$ e Å^{-3}
Extinction correction: SHELXL97, $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00174 (11)

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.54275 (18)	0.3242 (3)	0.3053 (4)	0.0204 (7)
H1A	0.5677	0.3096	0.4286	0.024*
H1B	0.5456	0.2306	0.2417	0.024*
C2	0.58877 (17)	0.4387 (3)	0.2187 (4)	0.0178 (7)
H2A	0.5825	0.5343	0.2757	0.021*
H2B	0.5671	0.4473	0.0924	0.021*
C3	0.67736 (18)	0.3991 (3)	0.2346 (4)	0.0210 (7)
H3A	0.6976	0.3793	0.3599	0.025*
H3B	0.6842	0.3095	0.1658	0.025*
C4	0.72465 (19)	0.5222 (3)	0.1668 (4)	0.0225 (7)
H4A	0.7074	0.5356	0.0389	0.027*
H4B	0.7133	0.6137	0.2277	0.027*
C5	0.8421 (2)	0.3727 (3)	0.1321 (4)	0.0255 (7)
H5	0.8081	0.3034	0.0680	0.031*
C6	0.9232 (2)	0.3494 (4)	0.1607 (4)	0.0298 (8)
H6	0.9461	0.2632	0.1200	0.036*
C7	0.9699 (2)	0.4549 (4)	0.2501 (4)	0.0314 (8)
H7	1.0260	0.4405	0.2699	0.038*
C8	0.8615 (2)	0.5916 (3)	0.2825 (4)	0.0262 (7)
H8	0.8388	0.6766	0.3266	0.031*
N1	0.81170 (15)	0.4937 (3)	0.1953 (3)	0.0188 (6)
N2	0.93938 (17)	0.5776 (3)	0.3106 (4)	0.0344 (7)
I1	0.419543 (11)	0.38361 (2)	0.30573 (2)	0.01817 (7)
I2	0.204826 (12)	0.48311 (2)	0.30184 (3)	0.02537 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0175 (17)	0.0191 (16)	0.0256 (16)	0.0038 (13)	0.0066 (13)	0.0007 (13)
C2	0.0153 (17)	0.0203 (16)	0.0177 (15)	0.0008 (12)	0.0016 (12)	0.0009 (12)
C3	0.0161 (18)	0.0191 (16)	0.0278 (17)	0.0011 (13)	0.0031 (13)	0.0014 (13)
C4	0.0132 (18)	0.0243 (17)	0.0302 (18)	0.0038 (13)	0.0035 (13)	0.0038 (14)
C5	0.0209 (19)	0.0222 (18)	0.0338 (19)	0.0023 (14)	0.0047 (14)	0.0002 (14)
C6	0.021 (2)	0.0267 (18)	0.042 (2)	0.0095 (15)	0.0071 (16)	0.0021 (16)
C7	0.0135 (19)	0.041 (2)	0.040 (2)	−0.0024 (16)	0.0027 (15)	0.0084 (17)
C8	0.023 (2)	0.0217 (17)	0.0341 (19)	−0.0017 (14)	0.0035 (15)	0.0007 (14)
N1	0.0136 (15)	0.0193 (14)	0.0233 (14)	0.0009 (11)	0.0013 (11)	0.0041 (11)
N2	0.0206 (17)	0.0363 (18)	0.0458 (19)	−0.0064 (14)	0.0032 (14)	−0.0017 (14)
I1	0.01703 (13)	0.01706 (12)	0.02101 (12)	−0.00227 (8)	0.00464 (8)	−0.00121 (8)
I2	0.01859 (14)	0.02792 (13)	0.03015 (13)	−0.00015 (9)	0.00529 (9)	−0.00106 (9)

Geometric parameters (Å, º)

C1—C2	1.509 (4)	C4—H4B	0.9900
C1—I1	2.155 (3)	C5—N1	1.339 (4)
C1—H1A	0.9900	C5—C6	1.379 (4)
C1—H1B	0.9900	C5—H5	0.9500
C2—C3	1.532 (4)	C6—C7	1.374 (5)
C2—H2A	0.9900	C6—H6	0.9500
C2—H2B	0.9900	C7—N2	1.344 (4)
C3—C4	1.512 (4)	C7—H7	0.9500
C3—H3A	0.9900	C8—N2	1.313 (4)
C3—H3B	0.9900	C8—N1	1.348 (4)
C4—N1	1.484 (4)	C8—H8	0.9500
C4—H4A	0.9900		
C2—C1—I1	112.19 (19)	C3—C4—H4A	109.2
C2—C1—H1A	109.2	N1—C4—H4B	109.2
I1—C1—H1A	109.2	C3—C4—H4B	109.2
C2—C1—H1B	109.2	H4A—C4—H4B	107.9
I1—C1—H1B	109.2	N1—C5—C6	119.5 (3)
H1A—C1—H1B	107.9	N1—C5—H5	120.3
C1—C2—C3	110.8 (2)	C6—C5—H5	120.3
C1—C2—H2A	109.5	C7—C6—C5	117.9 (3)
C3—C2—H2A	109.5	C7—C6—H6	121.1
C1—C2—H2B	109.5	C5—C6—H6	121.1
C3—C2—H2B	109.5	N2—C7—C6	122.5 (3)
H2A—C2—H2B	108.1	N2—C7—H7	118.7
C4—C3—C2	110.5 (2)	C6—C7—H7	118.7
C4—C3—H3A	109.5	N2—C8—N1	124.5 (3)
C2—C3—H3A	109.5	N2—C8—H8	117.7
C4—C3—H3B	109.5	N1—C8—H8	117.7
C2—C3—H3B	109.5	C5—N1—C8	119.0 (3)
H3A—C3—H3B	108.1	C5—N1—C4	120.8 (3)
N1—C4—C3	112.2 (2)	C8—N1—C4	120.1 (3)
N1—C4—H4A	109.2	C8—N2—C7	116.6 (3)
I1—C1—C2—C3	175.21 (19)	N2—C8—N1—C5	0.2 (5)
C1—C2—C3—C4	−173.5 (3)	N2—C8—N1—C4	−178.4 (3)
C2—C3—C4—N1	174.7 (2)	C3—C4—N1—C5	56.1 (4)
N1—C5—C6—C7	−1.7 (5)	C3—C4—N1—C8	−125.3 (3)
C5—C6—C7—N2	0.5 (5)	N1—C8—N2—C7	−1.4 (5)
C6—C5—N1—C8	1.4 (4)	C6—C7—N2—C8	1.0 (5)
C6—C5—N1—C4	180.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C6—H6···N2^{i}	0.95	2.58	3.396 (4)	144

C7—H7···I2^{ii}	0.95	3.03	3.952 (3)	164
C8—H8···I2^{iii}	0.95	3.04	3.792 (3)	138

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