

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

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## Key indicators

Single-crystal X-ray study  
 $T = 120$  K  
 Mean  $\sigma(C-C) = 0.004$  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>.

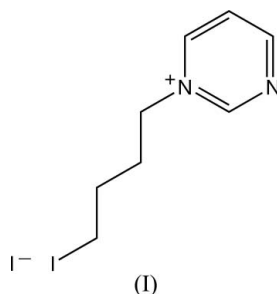
The title molecular salt,  $C_8H_{12}IN_2^+ \cdot I^-$ , features weak  $C-H \cdots N$  and  $C-H \cdots I$  interactions in the crystal structure.

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## 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)°.



A PLATON (Spek, 2003) analysis of (I) identified some short  $C-H \cdots N$  and  $C-H \cdots I$  interactions (Table 1) that might influence the crystal packing. Conversely, there are no  $\pi-\pi$  stacking interactions in (I), the shortest  $Cg \cdots Cg$  separation being greater than 5.2 Å. The shortest  $I1 \cdots I2$  contact of 3.7418 (3) Å in (I) is significantly less than the Bondi (1964) van der Waals  $I \cdots I$  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.  $\nu_{\max}$  (KBr,  $\text{cm}^{-1}$ ) 678 (alkyl-I), 817 (isolated aryl-H), 1431 ( $\text{CH}_2$ ), 1619 ( $\text{C}=\text{N}$ , conjugated, cyclic), 2920 ( $\text{CH}_2$ ), 3048 (CH-halogen).

#### Crystal data

$\text{C}_8\text{H}_{12}\text{IN}_2^+\cdot\text{I}^-$	$Z = 4$
$M_r = 390.00$	$D_x = 2.207 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 16.9178$ (6) Å	$\mu = 5.32 \text{ mm}^{-1}$
$b = 9.1694$ (3) Å	$T = 120$ (2) K
$c = 7.6303$ (2) Å	Plate, orange
$\beta = 97.329$ (2)°	$0.22 \times 0.18 \times 0.02 \text{ mm}$
$V = 1173.99$ (6) Å <sup>3</sup>	

#### Data collection

Nonius KappaCCD diffractometer	12760 measured reflections
$\omega$ and $\varphi$ scans	2674 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2001)	2302 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.388$ , $T_{\max} = 0.900$	$R_{\text{int}} = 0.037$
	$\theta_{\text{max}} = 27.5^\circ$

#### Refinement

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

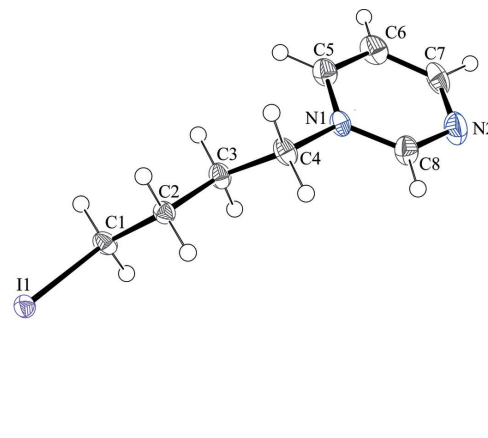
**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C6}-\text{H6}\cdots\text{N2}^{\text{i}}$	0.95	2.58	3.396 (4)	144
$\text{C7}-\text{H7}\cdots\text{I2}^{\text{ii}}$	0.95	3.03	3.952 (3)	164
$\text{C8}-\text{H8}\cdots\text{I2}^{\text{iii}}$	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  $\text{C}-\text{H} = 0.95\text{--}0.99$  Å and refined as riding with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .



**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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