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Crystal structure of 1-heptylpyridazin-1-ium iodide, $C_{11}H_{19}N_2I$

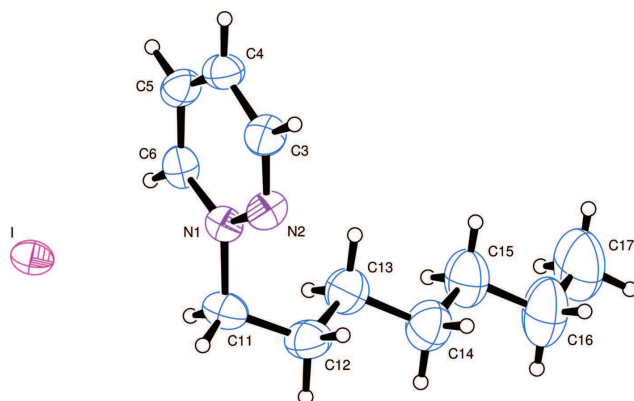


Table 1: Data collection and handling.

Crystal:	Light yellow prism
Size:	0.35 × 0.11 × 0.10 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.21 mm ⁻¹
Diffractometer, scan mode:	Xcalibur 3/Sapphire3, Thin slice φ and ω -scans
θ_{\max} , completeness:	27.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11282, 3285, 0.041
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2794
$N(\text{param})_{\text{refined}}$:	127
Programs:	CrysAlis [1], SHELX [2, 3], WinGX [4]

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Abstract

$C_{11}H_{19}N_2I$, triclinic, $P\bar{1}$ (no. 2), $a = 5.7074(3)$ Å, $b = 8.9811(5)$ Å, $c = 14.4312(8)$ Å, $\alpha = 100.989(4)^\circ$, $\beta = 94.768(4)^\circ$, $\gamma = 97.327(4)^\circ$, $V = 715.83(7)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0386$, $wR_{\text{ref}}(F^2) = 0.0913$, $T = 295(2)$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of materials

The title ionic liquid was prepared according to a method reported earlier [5, 6]. To a solution of pyridazine (1 g, 12.5 mmol in 10 mL of toluene) was added dropwise

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1-iodoheptane (2.825 g, 12.5 mmol) and the mixture was placed in a closed container and exposed to irradiation for 5 hours at room temperature using a sonication bath. Completion of the reaction was marked by the precipitation of a solid from the initially obtained clear and homogenous mixture in toluene. The pyridazinium-based ionic liquid is isolated by filtration and washed three times with ethyl acetate to remove any unreacted starting materials and solvent. Finally the 1-heptylpyridazin-1-ium iodide was dried at a reduced pressure to remove all volatile organic compounds to produce a yellow powder. (Yield 77%, m.p. 89–92 °C). Crystals were obtained from a mixture of dichloromethane and *n*-hexane (1:2). Elemental analysis: Anal. Calc. for $C_{11}H_{19}IN_2$: C, 43.15%; H, 6.25%; N, 9.15%; Found: C, 43.10%; H, 6.19%; N, 9.11%. ¹H NMR (DMSO, 400 MHz): $\delta = 0.81$ (t, 3H), 1.23–1.30 (m, 8H), 1.99 (quintet, 2H), 4.83 (t, 2H), 7.65 (t, 1H), 8.77 (t, 1H), 9.66 (d, 1H), 10.02 (d, 1H); ¹³C NMR (DMSO, 100 MHz): $\delta = 14.4$ (CH₃), 22.6 (CH₂), 25.8 (CH₂), 28.8 (CH₂), 29.9 (CH₂), 31.7 (CH₂), 65.0 (CH₂), 136.5 (CH), 137.0 (CH), 150.3 (CH), 155.0 (CH).

Experimental details

The diffraction data were processed using the CrysAlisPro-CCD and -RED [1] programs. The structure was determined by the intrinsic phasing routines in the SHELXT program [2] and refined by full-matrix least-squares methods, on F^2 's, in SHELXL [3]. Hydrogen atoms were included in idealized positions and their U_{iso} values were set to ride on the U_{eq} values of the parent carbon atoms.

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
I	0.90826(4)	0.75400(3)	0.58391(2)	0.06733(12)
N1	0.5799(5)	0.6420(3)	0.34437(19)	0.0579(7)
N2	0.7257(5)	0.7389(4)	0.3100(2)	0.0685(8)
C3	0.6800(7)	0.8800(4)	0.3215(3)	0.0741(10)
H3	0.7812	0.9504	0.2984	0.089*
C4	0.4921(8)	0.9308(5)	0.3657(3)	0.0754(11)
H4	0.4659	1.0322	0.3718	0.090*
C5	0.3474(7)	0.8281(6)	0.3997(3)	0.0766(11)
H5	0.2169	0.8566	0.4294	0.092*
C6	0.3969(6)	0.6810(5)	0.3895(2)	0.0676(9)
H6	0.3026	0.6089	0.4141	0.081*
C11	0.6434(8)	0.4853(5)	0.3325(3)	0.0798(11)
H11A	0.8012	0.4903	0.3648	0.096*
H11B	0.5328	0.4234	0.3624	0.096*
C12	0.6393(10)	0.4109(6)	0.2323(4)	0.0920(14)
H12A	0.6896	0.3110	0.2289	0.110*
H12B	0.7520	0.4720	0.2027	0.110*
C13	0.3966(9)	0.3914(6)	0.1775(3)	0.1012(15)
H13A	0.3547	0.4920	0.1750	0.121*
H13B	0.2812	0.3412	0.2116	0.121*
C14	0.3793(10)	0.2984(8)	0.0763(4)	0.1124(17)
H14A	0.4929	0.3495	0.0419	0.135*
H14B	0.4239	0.1984	0.0787	0.135*
C15	0.1357(11)	0.2765(9)	0.0219(4)	0.126(2)
H15A	0.0218	0.2274	0.0570	0.151*
H15B	0.0927	0.3765	0.0185	0.151*
C16	0.1157(14)	0.1844(13)	-0.0753(5)	0.160(3)
H16A	0.1625	0.0851	-0.0725	0.192*
H16B	0.2248	0.2350	-0.1116	0.192*
C17	-0.1308(14)	0.1613(12)	-0.1255(5)	0.184(4)
H17A	-0.1341	0.1008	-0.1883	0.275*
H17B	-0.2392	0.1092	-0.0907	0.275*
H17C	-0.1769	0.2591	-0.1298	0.275*

Comment

Ionic liquids (ILs) have received increased attention in recent years due to their outstanding and unique properties, such as negligible vapor pressure, non-volatility, non-flammability, excellent thermal stability, and high electrical conductivity [7]. Based on these characteristics, a broad range of applications of ILs has been reported in a variety of different fields, including the electrodeposition of metals and the development of corrosion inhibitors [8–10]. Furthermore, a number of biological activities of pyridazine derivatives have been reported including anticancer, antituberculosis, antihypertensive and antimicrobial activities [11–13].

The asymmetric unit of the title crystal structure contains one cation and the iodide anion. Bond lengths and angles in the organic cation are in the expected ranges. The heptyl group has an all-*trans* arrangement and is aligned about C(11)

Table 3: ‘Weak’ hydrogen bonds, in Ångstroms and degrees.

D–H···A	d(D–H)	d(H···A)	d(D···A)	<(DHA)
C(3)–H(3)···I#1	0.93	3.11	3.739(4)	126.8
C(4)–H(4)···I#2	0.93	3.08	3.861(4)	143.2
C(5)–H(5)···I#3	0.93	3.15	3.892(4)	137.8
C(11)–H(11A)···I#4	0.97	3.08	3.808(4)	133.4
C(11)–H(11B)···I#5	0.97	3.04	3.985(5)	164.8

Symmetry transformations used to generate equivalent atoms:

#1 : 2–x, 2–y, 1–z #2 : 1–x, 2–y, 1–z #3 : x–1, y, z
 #4 : 2–x, 1–y, 1–z #5 : 1–x, 1–y, 1–z.

with a *cis* N(2)–N(1)–C(11)–C(12) torsion angle of –61.6(5)°; the N(2)–N(1)–C(11)–H(11b) angle is *trans* at 177.2°.

The iodide ion lies over the pyridinium ring at 3.686 Å from N(1). There are also five short H···I contacts in the range 3.04–3.15 Å, to neighboring cations, forming ‘weak’ C–H···I hydrogen bonds, Table 3, which link ions in planes parallel to the *ab* plane, at *z* ~ 1/2. All the short inter-ion distances involve the iodide ion; the heptyl chains lie parallel but do not show any close contacts between chains. In a recent work from our group, we found a bromide anion linked to a pyridinium cation by a C–H···Br hydrogen bond with the H···Br distance 2.89 Å; there were four further short C–H···Br contacts, to three separate cations, at distances ranging from *ca* 3.07 to 3.11 Å [14].

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