Musa A. Said*, Mohamed R. Aouad, Saud M. Almutairi, David L. Hughes* and Mouslim Messali*

Crystal structure of 1-heptylpyridazin-1-ium iodide, C_{11}H_{19}N_{2}I

Abstract

C_{11}H_{19}N_{2}I, triclinic, P1 (no. 2), \( a = 5.707(3) \) 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, \( Z = 2 \), \( R_{int}(F) = 0.0386 \), \( wR_{int}(F^2) = 0.0913 \), \( T = 295(2) \) K.

CCDC no.: 1836644

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

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}I_{2}N_{2}: C, 43.15%; H, 6.25%; N, 9.15%; Found: C, 43.10%; H, 6.19%; N, 9.11%.

Table 1: Data collection and handling.

| Crystal: | Light yellow prism |
| Size: | 0.35 \times 0.11 \times 0.10 \text{ mm} |
| Wavelength: | Mo Kα radiation (0.71073 Å) |
| \( \mu \): | 2.21 \text{ mm}^{-1} |
| Diffractometer, scan mode: | Xcalibur 3/Sapphire3, Thin slice \( \phi \) and \( \omega \)-scans |
| \( \theta_{\text{max}} \), completeness: | 27.5°, >99% |
| \( N(hkl)_{\text{measured}}, N(hkl)_{\text{unique}}, R_{\text{int}} \): | 11282, 3285, 0.041 |
| Criterion for \( I_{\text{obs}} \), \( N(hkl)_{\text{refined}} \): | \( I_{\text{obs}} > 2 \sigma(I_{\text{obs}}), 2794 \) |
| Programs: | CrysAlis [1], SHELX [2, 3], WinGX [4] |

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 \( F^2 \), in SHELXL [3]. Hydrogen atoms were included in idealized positions and their \( U_{eq} \) values were set to ride on the \( U_{eq} \) values of the parent carbon atoms.

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H17C − H17B − H17A C17 − − −

isotropic displacement parameters (Å² reported including anticancer, antituberculosis, antihyper-
ol of biological activities of pyridazine derivatives have been

Furthermore, a number of developments of ILs has been reported in a variety of different fields,
[7]. Based on these characteristics, a broad range of applica-

negligible vapor pressure, non-volatility, non-flammability,

Ionic liquids (ILs) have received increased attention in recent

Comment

Ionic liquids (ILs) have received increased attention in recent years due to their outstanding and unique properties, such as

based on these characteristics, a broad range of applica-

including the electrodeposition of metals and the develop-

vantilical properties and attractive antimicrobi-

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 pyridazinium 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 a̅b 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 pyridi-

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].

References

2. Sheldrick, G. M.: SHELXT – Integrated space-group and
3–8.
3. Sheldrick, G. M.: Crystal structure refinement with SHELXL.
5. Messali, M.: An efficient and green sonochemical synthesis of
some new eco-friendly functionalized ionic liquids. Arab.
J. Chem. 7 (2014) 63–70.
pyridinium-based ionic liquids with attractive antimicrobi-
effective additives during zinc electrodeposition from aqueous
Hamed, O.; Messali, M.; Samhan, S.; Zougagh, M.; Oudda, H.: Pyridinium derivatives as corrosion inhibitors for mild steel in
1M HCl: electrochemical, surface and quantum chemical