Crystal structure of 1,1,3-trimethyl-2-oxo-imidazolidinium iodide — water (1:0.13), $[C_6H_{13}N_2O]I \cdot 0.13H_2O$

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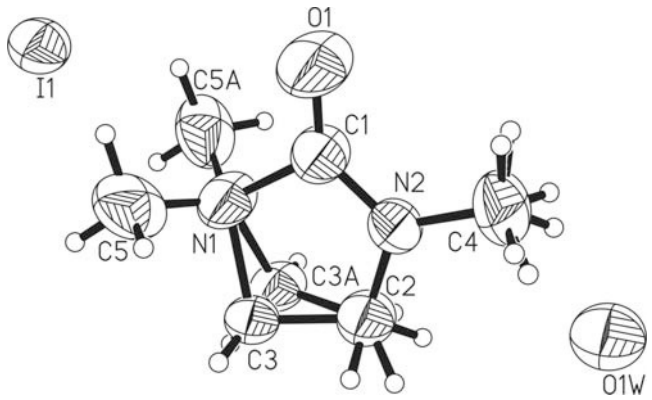

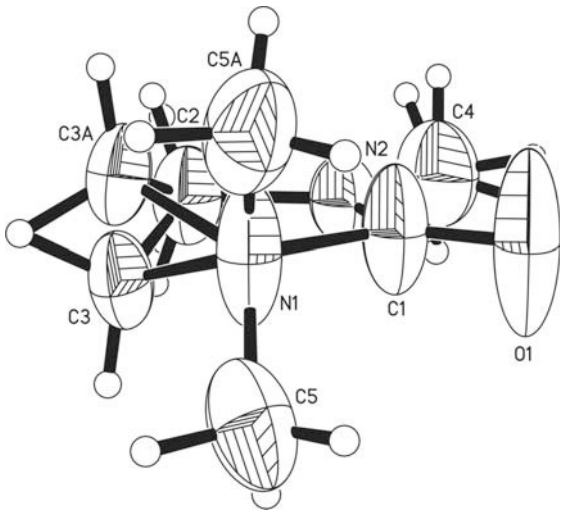

Abstract

$C_6H_{13.26}IN_2O_{1.13}$, monoclinic, *C*12/*m*1 (no. 12), $a = 11.910(2)$ Å, $b = 8.011(2)$ Å, $c = 10.071(2)$ Å, $\beta = 91.24(2)°$, $V = 960.6$ Å^3, $Z = 4$, $R_{gt}(F) = 0.041$, $wR_{ref}(F^2) = 0.088$, $T = 293$ K.

Source of material

The title compound was isolated as a hydrolysed side-product, which was formed in an abnormal way by addition of methyl iodide to the (*S*)-*N*-(1,3-dimethyl-imidazolidin-2-ylidene)-*N*-(1-phenylethyl)amine [1,2]. The usual reactions of guanidines with methylating agents take place at the imine nitrogen [2]. The compound was crystallized from acetonitrile and diethyl ether to give colourless crystals (m.p. 418 K), which turn amorphous at ca. 393 K [1].

Experimental details

Displacement ellipsoids of O1, C1, and N1 show large elongations perpendicular to the mirror plane (figure, bottom). This is an indication of a disorder, but the resolution of the data is too weak for refinement of discrete disorder positions, with respect to the flat geometry forced by the carbonyl function. Twin refinement strategies also did not lead to better results. The disordered hydrogen positions of C2, C3, C3A and C4 are generated by the mirror plane operator. The hydrogen atoms of the methylene functions at C2 and C3 were calculated and then fixed with respect to the disorder. The hydrogen atoms of the methyl groups were refined with fixed individual displacement parameters using a riding model with a *d*(C—H) distance of 0.96 Å and were allowed to rotate but not to tip.

Discussion

In the title crystal structure, the atoms O1, C1, N1, C2, C4, and I1 lie on special positions of the mirror plane *x*,0,*z*. The mirror plane operator *x*,−*y*,*z* completes the ammonium ion with the methyl group C5A and generates the second domain of the disordered methylene functions of C3, C3A. The five-membered ring system shows an envelope conformation, where the disordered positions of the methylene groups at C3 and C3A are situated 0.470(9) Å below and above the mirror plane. We localized an electron density peak, which was identified as a fraction of an oxygen atom of a solvent water molecule. Free refinement of coordinates, anisotropic displacement parameters and of the population factor converged by a fraction of 1/8 of the occupancy factor. Hydrogen atoms of the water molecule could not be localized. O1W lies on the special position ½,*y*,0. The O1W···I1 contact is 3.34(3) Å, which is remarkably short.

Table 1. Data collection and handling.

Crystal:	colorless plates, size 0.15 × 0.25 × 0.7 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	32.82 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, Wyckoff
$2\theta_{max}$:	59.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2920, 1494
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1326
$N(param)_{refined}$:	68
Programs:	SHELXS-97 [3], SHELXL-97 [4], SHELXTL-Plus [5]

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Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{iso}
H(2A)	8*j*	0.50	0.2943	–0.1112	0.919o	0.076
H(2B)	8*j*	0.50	0.3281	0.0753	0.9502	0.076
H(3A)	4*i*		0.1283	0	0.8725	0.066
H(3B)	8*j*	0.50	0.1811	0.1762	0.8358	0.066
H(4A)	8*j*	0.50	0.5249	–0.0278	0.6974	0.121
H(4B)	8*j*	0.50	0.5174	–0.0809	0.8467	0.121
H(4C)	8*j*	0.50	0.5193	0.1088	0.8085	0.121
H(5A)	8*j*		0.1510	0.1414	0.5223	0.128
H(5B)	8*j*		0.1851	0.2480	0.6474	0.128
H(5C)	8*j*		0.0685	0.1572	0.6409	0.128

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	4*i*		0.14794(3)	0	0.20522(4)	0.0511(2)	0.0844(3)	0.0528(2)	0	0.0097(2)	0
O(1)	4*i*		0.3566(4)	0	0.5430(5)	0.055(3)	0.28(1)	0.050(3)	0	0.020(2)	0
N(1)	4*i*		0.1974(4)	0	0.6801(5)	0.040(2)	0.160(7)	0.040(2)	0	0.010(2)	0
C(1)	4*i*		0.3235(5)	0	0.6546(6)	0.040(3)	0.139(7)	0.047(3)	0	0.010(2)	0
N(2)	4*i*		0.3730(4)	0	0.7701(5)	0.041(2)	0.068(3)	0.050(2)	0	0.007(2)	0
C(2)	4*i*		0.2995(5)	0	0.8810(6)	0.054(3)	0.089(5)	0.048(3)	0	0.008(2)	0
C(3)	8*j*	0.50	0.1905(6)	0.059(1)	0.8283(7)	0.048(3)	0.082(6)	0.037(3)	0.008(3)	0.010(3)	–0.002(3)
C(4)	4*i*		0.4937(5)	0	0.7816(8)	0.041(3)	0.113(6)	0.088(5)	0	0.004(3)	0
C(5)	8*j*		0.1460(4)	0.1499(8)	0.6171(6)	0.052(2)	0.112(5)	0.093(4)	–0.004(3)	0.016(2)	–0.025(4)
O(1W)	4*h*	0.13	½	–0.253(7)	0	0.10(2)	0.12(3)	0.08(2)	0	0.02(2)	0

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