Crystal structure of 2,4,5-tribromo-1-methyl-1*H*-imidazole, $C_4H_3Br_3N_2$

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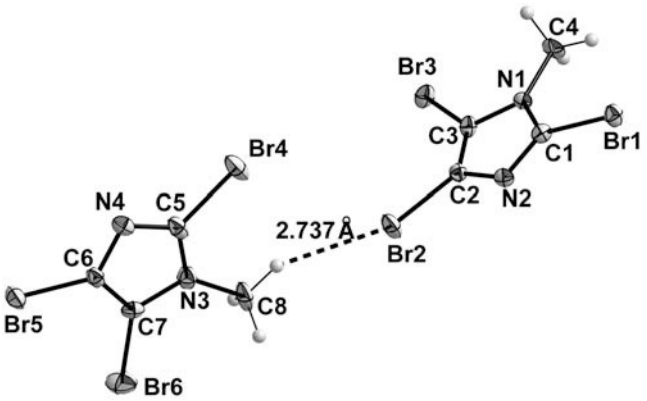

Abstract

$C_4H_3Br_3N_2$, triclinic, $P\bar{1}$ (no. 2), a = 6.9066(3) Å, b = 7.0812(3) Å, c = 16.1046(6) Å, α = 78.093(2)°, β = 82.467(2)°, γ = 89.781(2)°, V = 763.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.026, $wR_{ref}(F^2)$ = 0.064, T = 173 K.

Source of material

1-Methyl-1*H*-imidazole (24.6 g, 0.3 mol) and sodium acetate trihydrate (100 g, 0.7 mol) were dissolved in 350 ml glacial acetic acid and a solution of bromine (143.6 g, 0.9 mol) in 50 ml of glacial acetic acid was added slowly under cooling. The resulting solution was stirred overnight and then poured onto 500 ml of ice water. The precipitate of 2,4,5-tribromo-1-methyl-1*H*-imidazole was filtered off and recrystallized from aqueous ethanol. Yield: 50.0 g (52 %), mp. 93 °C.

Discussion

Already in the late 19th century Wyss and Wallach reported the correct molecular formula of 2,4,5-tribromo-1-methyl-1*H*-imidazole and they discussed possible chemical structures [1,2]. Until now there has been no X-ray determination of the crystal structure of this compound.

The imidazole core is a key for a huge variety of important biologically active substances. It has found many applications in metal-carbene catalysts and is most important in ionic liquids (ILs) and magnetic ILs [3-8]. Polybrominated imidazole building blocks are useful as starting materials in the synthesis of very dense halogenated ILs [9].

The title compound crystallizes with two symmetry independent molecules in the asymmetric unit. The orientation of the molecules gives short hydrogen-bonding contacts between Br atoms and methyl groups of neighbouring units. The figure shows such an intermolecular hydrogen-bonding contact as thermal ellipsoid plot at the 50 % probability level.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.20 × 0.30 × 0.45 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	157.56 cm^{-1}
Diffractometer, scan mode:	Bruker-Nonius X8 APEX CCD, φ/ω
$2\theta_{max}$:	52.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	10213, 3016
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2684
$N(param)_{refined}$:	164
Program:	SHELXTL [10]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	2*i*	0.1751	0.5805	0.664	0.035
H(4B)	2*i*	0.0842	0.7679	0.6105	0.035
H(4C)	2*i*	0.3129	0.7635	0.6182	0.035
H(8A)	2*i*	0.2722	–0.3101	1.1183	0.046
H(8B)	2*i*	0.4013	–0.1602	1.0439	0.046
H(8C)	2*i*	0.4029	–0.3857	1.0427	0.046

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

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(1)	2*i*	0.2357(4)	0.6060(4)	0.5359(2)	0.018(2)	0.014(2)	0.016(2)	–0.000(1)	–0.003(1)	–0.006(1)
C(1)	2*i*	0.2379(5)	0.4171(6)	0.5309(3)	0.016(2)	0.014(2)	0.021(2)	–0.002(2)	–0.002(2)	–0.004(2)
Br(1)	2*i*	0.19232(6)	0.22092(6)	0.62813(3)	0.0271(2)	0.0170(2)	0.0196(2)	–0.0018(2)	–0.0029(2)	–0.0013(2)
N(2)	2*i*	0.2748(4)	0.3897(5)	0.4520(2)	0.021(2)	0.015(2)	0.019(2)	–0.001(1)	–0.004(1)	–0.006(1)
C(2)	2*i*	0.2972(5)	0.5742(6)	0.4032(2)	0.019(2)	0.018(2)	0.016(2)	0.003(2)	–0.005(2)	–0.004(2)
Br(2)	2*i*	0.34948(7)	0.61574(7)	0.28420(3)	0.0413(3)	0.0331(3)	0.0146(2)	–0.0005(2)	–0.0037(2)	–0.0071(2)
C(3)	2*i*	0.2749(5)	0.7081(6)	0.4523(2)	0.020(2)	0.013(2)	0.018(2)	–0.002(2)	–0.005(2)	–0.001(2)
Br(3)	2*i*	0.28569(6)	0.97697(6)	0.42686(3)	0.0269(2)	0.0131(2)	0.0259(2)	–0.0001(2)	–0.0074(2)	–0.0036(2)
C(4)	2*i*	0.1989(6)	0.6862(6)	0.6136(3)	0.036(2)	0.019(2)	0.018(2)	0.002(2)	–0.004(2)	–0.013(2)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(3)	2*i*	0.1475(5)	–0.2538(5)	1.0039(2)	0.029(2)	0.023(2)	0.021(2)	0.001(2)	–0.007(1)	–0.006(2)
C(5)	2*i*	–0.0463(6)	–0.2657(6)	1.0316(3)	0.037(2)	0.015(2)	0.017(2)	–0.002(2)	0.001(2)	–0.003(2)
Br(4)	2*i*	–0.14694(8)	–0.31837(7)	1.14670(3)	0.0613(3)	0.0287(3)	0.0160(2)	–0.0014(2)	0.0040(2)	–0.0049(2)
N(4)	2*i*	–0.1556(5)	–0.2348(5)	0.9690(2)	0.024(2)	0.023(2)	0.019(2)	–0.002(2)	0.001(1)	–0.005(2)
C(6)	2*i*	–0.0218(6)	–0.1994(6)	0.8964(2)	0.026(2)	0.017(2)	0.016(2)	0.000(2)	–0.002(2)	–0.005(2)
Br(5)	2*i*	–0.09572(6)	–0.14231(7)	0.78685(3)	0.0279(2)	0.0412(3)	0.0163(2)	–0.0010(2)	–0.0033(2)	–0.0052(2)
C(7)	2*i*	0.1630(5)	–0.2096(6)	0.9153(3)	0.018(2)	0.017(2)	0.024(2)	–0.001(2)	0.002(2)	–0.006(2)
Br(6)	2*i*	0.40074(6)	–0.17698(8)	0.84577(3)	0.0236(2)	0.0491(3)	0.0434(3)	–0.0013(2)	0.0046(2)	–0.0116(2)
C(8)	2*i*	0.3222(7)	–0.2799(7)	1.0571(3)	0.052(3)	0.026(2)	0.016(2)	–0.001(2)	–0.013(2)	–0.005(2)

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