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The crystal structure of 9-butoxy-2-(hydroxymethyl)-2*H*-imidazo[1,5-*a*]quinolin-10-ium bromide, C₁₇H₂₁O₂N₂Br

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Abstract

C₁₇H₂₁O₂N₂Br, orthorhombic, *Pbcn* (no. 36), *a* = 20.179(12) Å, *b* = 10.902(7) Å, *c* = 15.026(9) Å, *V* = 3306(3) Å³, *Z* = 8, *R*_{gt}(*F*) = 0.0465, *wR*_{ref}(*F*²) = 0.1276, *T* = 296(2) K.

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The crystal structure is shown in the figure. Hydrogen atoms are omitted for clarity. 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

8-Butoxyquinoline-2-carbaldehyde was prepared according to the literature [4]. The title compound was synthesized by the reaction of 8-butoxyquinoline-2-carbaldehyde

Table 1: Crystal collection and handling.

Crystal:	Rod, yellow
Size:	0.28 × 0.25 × 0.18 mm
Wavelength:	Mo <i>K</i> α radiation (λ = 0.71073 Å)
μ:	2.496 mm ^{−1}
Diffractometer, scan mode:	Bruker APEX II, φ and ω-scans
θ _{max} , completeness:	26.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	17024, 3243, 0.0864
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2σ(<i>I</i> _{obs}), 1923
<i>N</i> (<i>param</i>) _{refined} :	200
Programs:	Bruker programs [1], SHELX [2, 3]

(4.59 g, 20 mmol), formaldehyde (2.8 mL, 30 mmol) and 2-aminoethanol (1.20 mL, 20 mmol) in ethanol [5]. Hydrogen bromide (1 equiv) was added to the reaction mixture, which was stirred at room temperature for 12 h. The residual mixture was filtrated, and washed with ethanol and ether. The product was obtained as a yellow solid (6.04 g, 86%). ¹H NMR (400 MHz, CDCl₃, 25 °C): σ 10.29 (m, *J* = 1.2 Hz, 1H), 8.27 (d, *J* = 8 Hz, 1H), 8.06 (d, *J* = 8 Hz, 1H), 7.60 (t, *J* = 8 Hz, 1H), 7.44–7.46 (m, 1H), 7.16 (d, *J* = 8 Hz, 1H, Ar-H), 4.31 (t, *J* = 6.8 Hz, 2H), 2.02–2.09 (m, 2H), 1.57–1.65 (m, 2H), 1.05 (t, *J* = 7.4 Hz, 3H). ¹³C NMR (100 MHz, CDCl₃): σ 13.81, 19.19, 30.84, 69.03, 109.58, 117.66, 119.24, 129.67, 131.30, 137.09, 140.08, 151.33, 155.61, 193.80.

Experimental details

The structure was solved by Direct Methods, and the non-hydrogen atoms were subjected to anisotropic refinement using SHELXTL package [2].

Comment

The focus of many groups has been put on designing and assembling NHC precursor due to their potential applications [6–9]. The NHC precursor containing imidazo[1,5-*a*]pyridinium are quite rare [10–13].

The angle of N1–C11–N2 is 108.15(3)°. There is one molecule of the title compound in the asymmetric unit. Only some parameters should be mentioned: the angle of C1–O1–C14 is 118.34(3)°. The O–C bond lengths are between 1.35(4) Å and 1.43(5) Å. Hydrogen bonding is

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Br1	0.66944(2)	0.55079(4)	0.05020(3)	0.0648(2)
O1	0.57046(12)	0.94773(19)	0.17491(16)	0.0404(6)
O2	0.41762(15)	0.5944(3)	0.27339(19)	0.0609(8)
H2	0.3915	0.5782	0.3135	0.091
N1	0.44788(13)	0.8954(2)	0.10058(17)	0.0333(7)
N2	0.43354(15)	0.6996(2)	0.10192(19)	0.0386(7)
C1	0.53344(19)	1.0427(3)	0.1458(2)	0.0362(9)
C2	0.5533(2)	1.1630(3)	0.1508(3)	0.0475(10)
H2A	0.5938	1.1823	0.1768	0.057
C3	0.5139(2)	1.2549(3)	0.1180(3)	0.0535(11)
H3A	0.5283	1.3358	0.1215	0.064
C4	0.4547(2)	1.2298(3)	0.0807(3)	0.0501(11)
H4A	0.4291	1.2936	0.0582	0.060
C5	0.4312(2)	1.1098(3)	0.0751(2)	0.0394(9)
C6	0.47233(18)	1.0153(3)	0.1083(2)	0.0336(8)
C7	0.3685(2)	1.0820(4)	0.0371(3)	0.0488(11)
H7A	0.3421	1.1461	0.0168	0.059
C8	0.3463(2)	0.9677(4)	0.0295(3)	0.0514(11)
H8A	0.3051	0.9520	0.0041	0.062
C9	0.38627(19)	0.8709(3)	0.0607(2)	0.0402(9)
C10	0.3786(2)	0.7473(4)	0.0621(3)	0.0466(10)
H10A	0.3426	0.7034	0.0401	0.056
C11	0.47459(18)	0.7878(3)	0.1253(2)	0.0335(8)
H11A	0.5150	0.7774	0.1540	0.040
C12	0.4433(2)	0.5707(3)	0.1214(3)	0.0459(10)
H12A	0.4894	0.5566	0.1367	0.055
H12B	0.4331	0.5225	0.0689	0.055
C13	0.4001(2)	0.5302(3)	0.1970(3)	0.0541(11)
H13A	0.3540	0.5457	0.1827	0.065
H13B	0.4055	0.4428	0.2069	0.065
C14	0.63712(19)	0.9720(3)	0.2039(2)	0.0391(9)
H14A	0.6364	1.0182	0.2590	0.047
H14B	0.6602	1.0202	0.1593	0.047
C15	0.67182(18)	0.8534(3)	0.2176(3)	0.0423(9)
H15A	0.6748	0.8104	0.1612	0.051
H15B	0.6461	0.8030	0.2580	0.051
C16	0.74048(19)	0.8705(3)	0.2549(3)	0.0514(11)
H16A	0.7666	0.9183	0.2133	0.062
H16B	0.7376	0.9165	0.3100	0.062
C17	0.7748(3)	0.7521(4)	0.2723(5)	0.0946(18)
H17A	0.8181	0.7679	0.2960	0.142
H17B	0.7496	0.7051	0.3145	0.142
H17C	0.7787	0.7069	0.2178	0.142

present: (*d*(H···acceptor) and *d*(donor···acceptor), donor–H···acceptor): O2–H2···Br1ⁱ (2.41, 3.2145(3) Å), 168.5°; *i* = −*x* + 1, *y*, −*z* + 1/2.

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