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4-(9*H*-Fluoren-9-yl)-4-methylmorpholin-4-ium bromide, C₁₈H₂₀BrNO

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Abstract

C₁₈H₂₀BrNO, orthorhombic, *Pbca* (No. 61), $a = 8.4285(4)$ Å, $b = 19.2055(9)$ Å, $c = 19.7809(12)$ Å, $Z = 8$, $V = 3202.0(3)$ Å³, $R_{\text{gt}}(F) = 0.0427$, $wR_{\text{ref}}(F^2) = 0.1025$, $T = 293(2)$ K.

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Table 1: Data collection and handling.

Crystal:	Colorless parallelepiped
Size:	0.40 × 0.30 × 0.20 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ :	2.57 mm ⁻¹
Diffractometer, scan mode:	Xcalibur, ω
θ_{max} , completeness:	26.3°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	9934, 3252, 0.035
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2038
$N(\text{param})_{\text{refined}}$:	191
Programs:	CrysAlis ^{PRO} [1], Olex2 [2], SHELX [3]

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The molecular structure is shown in the Figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

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

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.27202 (4)	0.124219 (18)	0.496124 (15)	0.04542 (14)
C1	0.7239 (4)	0.18858 (17)	0.47311 (14)	0.0396 (8)
H1A	0.6141	0.1940	0.4868	0.048*
H1B	0.7532	0.2292	0.4468	0.048*
C2	0.8267 (4)	0.18496 (17)	0.53505 (13)	0.0478 (8)
H2A	0.9371	0.1821	0.5215	0.057*
H2B	0.8131	0.2272	0.5614	0.057*
C3	0.8175 (4)	0.06494 (16)	0.53716 (14)	0.0465 (8)
H3A	0.7970	0.0242	0.5648	0.056*
H3B	0.9282	0.0639	0.5237	0.056*
C4	0.7136 (4)	0.06277 (17)	0.47508 (14)	0.0400 (8)
H4A	0.7360	0.0205	0.4499	0.048*
H4B	0.6033	0.0612	0.4889	0.048*
C5	0.8970 (3)	0.12153 (18)	0.39548 (13)	0.0481 (9)
H5A	0.9115	0.1622	0.3680	0.072*
H5B	0.9020	0.0807	0.3675	0.072*
H5C	0.9792	0.1195	0.4290	0.072*
C6	0.6053 (3)	0.12686 (15)	0.37476 (11)	0.0296 (6)
H6	0.5011	0.1311	0.3963	0.036*
C7	0.6303 (3)	0.18594 (15)	0.32504 (12)	0.0308 (7)
C8	0.6650 (3)	0.15827 (16)	0.26120 (13)	0.0327 (7)
C9	0.6511 (3)	0.08278 (17)	0.26382 (13)	0.0347 (7)
C10	0.6096 (3)	0.06295 (15)	0.32976 (12)	0.0318 (7)
C11	0.5731 (3)	−0.00549 (17)	0.34339 (15)	0.0455 (8)
H11	0.5410	−0.0186	0.3865	0.055*
C12	0.5846 (4)	−0.05482 (19)	0.29233 (16)	0.0587 (10)
H12	0.5605	−0.1012	0.3012	0.070*
C13	0.6321 (4)	−0.03478 (19)	0.22790 (16)	0.0593 (10)
H13	0.6412	−0.0682	0.1941	0.071*
C14	0.6654 (4)	0.03280 (19)	0.21334 (14)	0.0492 (9)
H14	0.6974	0.0455	0.1701	0.059*
C15	0.6950 (4)	0.20249 (18)	0.20700 (13)	0.0459 (8)
H15	0.7207	0.1845	0.1648	0.055*
C16	0.6861 (4)	0.27306 (18)	0.21682 (15)	0.0545 (9)
H16	0.7062	0.3030	0.1808	0.065*
C17	0.6479 (4)	0.30040 (18)	0.27922 (14)	0.0505 (9)
H17	0.6409	0.3484	0.2847	0.061*
C18	0.6198 (3)	0.25672 (16)	0.33375 (13)	0.0396 (7)
H18	0.5941	0.2752	0.3758	0.048*
N1	0.7387 (2)	0.12486 (13)	0.42956 (11)	0.0288 (5)
O1	0.7878 (3)	0.12632 (12)	0.57565 (10)	0.0504 (6)

Source of material

The title compound was prepared following procedure given in [4] for preparation of 9*H*-fluorenylquinuclidinium bromide. To a solution of 1.19 g (4.9 mmol) of 9-bromofluorene in 150 mL of dried toluene, 0.60 mL (5.4 mmol) of 4-methylmorpholine was added. The reaction mixture was stirred at room temperature under argon for 12 h. The precipitated product was isolated by vacuum filtration from the reaction solution and washed three times with 30 mL of cold toluene. The crystals were recrystallized from ethanol affording 1.20 g (93%), as colourless crystals.

Experimental details

Comment

The title compound is expected to be a photobase generator based on similarity to reported compounds [4]. It was synthesised in a one-step reaction in high yield. Photobased generators are important for the development of new advanced materials, such as, photoresists and 3D printing materials [4–6].

The central five-membered ring is slightly deviated from planarity, the mean deviation from the plane of the five membered ring (C6–C7–C8–C9–C10) is 0.0272 Å (see the Figure). The deviation of C6 is the highest, 0.0397 Å. The d(C6–C7) and d(C6–C10) bond distances are 1.516(3) and 1.517(3) Å, which are shorter than usual C–C single bond distance of 1.54 Å, but in the typical range for struture determinations at room temperature [7]. The C7–C6–C20 angle is 102.8(2)°, which is smaller than that of the ideal *sp*³

hybridized carbon atom geometry. The d(C6–N1) bond distance is 1.562(3) Å, which is significantly longer than the other d(C–N) bond distances. The d(N1–C1), d(N1–C4) and d(N1–C5) bond distances are 1.502(3), 1.496(3) and 1.509(3) Å.

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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