Luo Jiewei, Zhang Shilu, Liu Yiming and Zhao Bo*

The crystal structure of 3-benzyl-1-((8-(benzyloxy)quinolin-2-yl)methyl)-1*H*-imidazol-3-ium hexafluorophosphate, C₂₇H₂₄N₃OF₆P

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Abstract

C₂₇H₂₄N₃OF₆P, monoclinic, *Cc* (no. 9), *a* = 18.0589(18) Å, *b* = 12.5100(9) Å, *c* = 13.3659(12) Å, β = 124.522(7)°, *V* = 2487.9(4) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0437, *wR*_{ref}(*F*²) = 0.1141, *T* = 113(2) K.

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The crystal structure is shown in the figure (H atoms 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 material

The title compound was prepared by the reaction of 8-(benzyloxy)-2-(bromomethyl)quinoline (3.28 g, 10 mmol), 1-benzyl-1*H*-imidazole (1.58 g, 10 mmol) and a grain of KI in

Table 1: Data collection and handling.

Crystal:	Colorless prism
Size:	0.24 × 0.20 × 0.18 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.18 mm ^{−1}
Diffractometer, scan mode:	Rigaku Saturn 724, ω
$\theta_{\max}$, completeness:	27.9°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	12927, 5042, 0.052
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 2955
<i>N</i> (<i>param</i>) _{refined} :	343
Programs:	Bruker [1], SHELX [2, 3]

THF [4]. The mixture refluxed for 2 days. The volatiles were removed under reduced pressure, and then the crude product dissolved in methanol. Ammonium hexafluorophosphate (1 equiv) was added to the mixture, which was stirred at room temperature for 0.5 h. The residual mixture was filtrated, and washed with THF. The product was obtained as a white solid (4.54 g, 82.4%). ¹H NMR (400 MHz, CDCl₃, 298.15 K): δ [ppm] 5.27 (s, 2H, Ar–CH₂–), 5.36 (s, 2H, Ar–CH₂–), 5.81 (s, 2H, Ar–CH₂–O–), 7.30–7.61 (m, 14H), 7.88–7.91 (m, 2H), 8.41–8.43 (d, *J* = 8 Hz, 1H), 9.43 (s, 1H, NCHN). ¹³C NMR (100 MHz, DMSO-d₆, 298.15 K): δ [ppm] 52.37, 54.01, 70.36, 111.75, 120.28, 120.53, 122.63, 124.49, 127.66, 127.68, 128.14, 128.57, 128.82, 128.84, 129.13, 129.35, 134.95, 137.50, 137.72, 137.81, 139.41, 153.12, 154.06.

Experimental details

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

Comment

Imidazolium salts, especially those that are room temperature ionic liquids are best known for their application as alternative solvents and catalysts [5, 6]. Quinolines possessing an aromatic ring system that can block imine formation and stabilizes the target intermediates are usually used in separation and enrichment of metal ions [7, 8].

Generally all geometric parameters are in the expected ranges, but some parameters should be mentioned: the angle of C7–O1–C17 is 117.9(3)°. The O–C bond lengths

*Corresponding author: Zhao Bo, North Sichuan Medical college, School of Basic Medical Sciences, Nanchong 637000, P.R. China, e-mail: zhaobo19871001@126.com. <https://orcid.org/0000-0003-0987-7602>

Luo Jiewei, Zhang Shilu and Liu Yiming: North Sichuan Medical college, School of Basic Medical Sciences, Nanchong 637000, P.R. China

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}
C4	0.2131(3)	0.6948(3)	0.6143(4)	0.0209(10)
C6	0.0867(3)	0.8203(4)	0.6072(4)	0.0252(10)
C7	0.1338(3)	0.9631(4)	0.5311(4)	0.0223(10)
C8	0.1426(3)	0.8562(3)	0.5726(4)	0.0201(9)
C9	0.4999(3)	0.8815(4)	0.6412(4)	0.0260(10)
H9A	0.4563	0.9032	0.5557	0.031 [*]
H9B	0.5562	0.8593	0.6506	0.031 [*]
C10	0.6197(4)	1.0725(4)	0.9058(4)	0.0334(12)
H10	0.6781	1.0822	0.9778	0.040 [*]
C11	0.0719(3)	1.0298(4)	0.5275(4)	0.0251(10)
H11	0.0657	1.1012	0.4997	0.030 [*]
C12	0.1596(3)	0.6508(4)	0.6500(4)	0.0302(11)
H12	0.1674	0.5786	0.6761	0.036 [*]
C13	0.0967(3)	0.7132(4)	0.6466(4)	0.0275(11)
H13	0.0599	0.6853	0.6703	0.033 [*]
C14	0.0243(3)	0.8917(4)	0.6036(4)	0.0289(11)
H14	−0.0128	0.8681	0.6285	0.035 [*]
C15	0.3773(3)	0.7665(3)	0.6094(4)	0.0228(10)
H15	0.3301	0.8025	0.5392	0.027 [*]
C16	0.5187(3)	0.9750(3)	0.7240(4)	0.0201(9)
C17	0.1774(3)	1.0916(3)	0.4432(4)	0.0266(11)
H17A	0.1882	1.1492	0.5009	0.032 [*]
H17B	0.1154	1.0989	0.3697	0.032 [*]
C18	0.4507(3)	0.6543(4)	0.7609(4)	0.0257(10)
H18	0.4634	0.5971	0.8153	0.031 [*]
C19	0.2837(3)	0.6265(3)	0.6194(4)	0.0280(11)
H19A	0.2968	0.5649	0.6735	0.034 [*]
H19B	0.2592	0.5981	0.5372	0.034 [*]
C20	0.0175(3)	0.9930(4)	0.5649(4)	0.0305(11)
H20	−0.0246	1.0401	0.5628	0.037 [*]
C21	0.5113(3)	0.7222(4)	0.7670(4)	0.0269(11)
H21	0.5744	0.7223	0.8260	0.032 [*]
C22	0.3712(3)	1.1063(4)	0.3567(4)	0.0307(12)
H22	0.4159	1.1088	0.3402	0.037 [*]
C23	0.2582(3)	1.0106(4)	0.3586(4)	0.0245(10)
H23	0.2238	0.9473	0.3410	0.029 [*]
C24	0.2934(3)	1.1907(4)	0.4320(4)	0.0301(11)
H24	0.2839	1.2514	0.4661	0.036 [*]
C25	0.4503(3)	1.0425(4)	0.6976(4)	0.0282(11)
H25	0.3918	1.0317	0.6261	0.034 [*]
C26	0.3230(3)	1.0166(4)	0.3332(4)	0.0303(11)
H26	0.3332	0.9567	0.2988	0.036 [*]
C27	0.2445(3)	1.0988(4)	0.4102(4)	0.0236(10)
C28	0.3563(3)	1.1945(4)	0.4044(4)	0.0321(12)
H28	0.3893	1.2583	0.4183	0.039 [*]
C29	0.6038(3)	0.9916(4)	0.8280(4)	0.0259(10)
H29	0.6517	0.9458	0.8454	0.031 [*]
C30	0.4659(4)	1.1260(4)	0.7742(5)	0.0397(14)
H30	0.4185	1.1736	0.7546	0.048 [*]
C31	0.5504(4)	1.1408(4)	0.8797(5)	0.0356(12)
H31	0.5609	1.1973	0.9337	0.043 [*]
F1	0.7026(2)	0.8912(3)	0.6874(3)	0.0577(10)
F2	0.7685(2)	0.7378(3)	0.6991(3)	0.0646(10)
F3	0.8484(3)	0.8872(3)	0.7513(4)	0.0803(13)

Table 2 (continued)

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
F4	0.87749(18)	0.7712(2)	0.8925(2)	0.0437(8)
F5	0.8100(2)	0.9254(3)	0.8787(4)	0.0745(12)
F6	0.73181(18)	0.7742(2)	0.8308(3)	0.0414(7)
N1	0.4633(2)	0.7910(3)	0.6706(3)	0.0222(8)
N2	0.3679(2)	0.6827(3)	0.6627(3)	0.0214(8)
N3	0.2053(2)	0.7921(3)	0.5759(3)	0.0196(8)
O2	0.18934(19)	0.9902(2)	0.4973(3)	0.0256(7)
P1	0.79050(9)	0.82961(11)	0.79089(12)	0.0353(3)

are between 1.357(5) Å and 1.415(5) Å. Each imidazolium cation is surrounded by hexafluorophosphate anions. Weak interionic contacts are observed (d(H···acceptor) and d(donor···acceptor), ∠donor-H···acceptor): F1···H—C9 (2.43 Å, 3.51 Å, 154.1°), F1···H—C12ⁱ (2.41 Å, 3.31 Å, 158.0°), F2···H—C17ⁱⁱ (2.45 Å, 3.37 Å, 155.0°), F2···H—C30ⁱⁱⁱ (2.50 Å, 3.41 Å, 159.9°), F3···H—C19ⁱ (2.41 Å, 3.33 Å, 155.4°), F6···H—C15ⁱⁱⁱ (2.49 Å, 3.15 Å, 126.3°). Symmetry codes: (i) $-1+x$, $-1-y$, z ; (ii) $-1+x$, $-y$, z ; (iii) $-1/2+x$, $3/2-y$, $-1/2+z$.

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