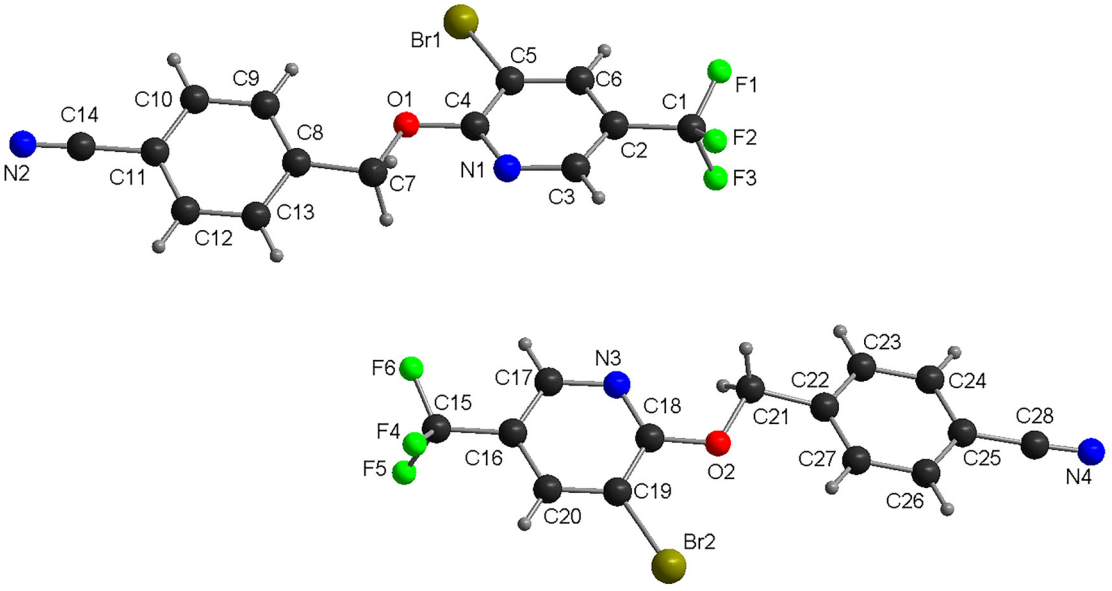
Zhi-Qiang Lu*

The crystal structure of 4-(((3-bromo-5-(trifluoromethyl)pyridin-2-yl)oxy)methyl) benzonitrile, $C_{28}H_{16}Br_2F_6N_4O_2$



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Abstract

$C_{28}H_{16}Br_2F_6N_4O_2$, triclinic, $P\bar{1}$ (no. 2), a = 8.0085(4) Å, b = 13.6578(7) Å, c = 14.1964(6) Å, α = 96.950(4)°, β = 105.376(4)°, γ = 106.772(5)°, V = 1400.32(12) Å^3, Z = 2, $R_{gt}(F)$ = 0.0570, $wR_{ref}(F^2)$ = 0.1268, T = 293(2) K.

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The asymmetric unit of the title crystal 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 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.25 × 0.24 × 0.21 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	2.97 mm^{-1}
Diffractometer, scan mode:	XtaLAB Synergy, ω
θ_{max}, completeness:	29.7°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	17,870, 6947, 0.056
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3806
$N(param)_{refined}$:	395
Programs:	CrysAlis^{PRO} [1], SHELX [2,3], Olex2 [4]

Source of material

The title compound was prepared according to the synthetic method reported in the literature [5]. The crude product was recrystallized from ethanol to afford crystals suitable for single crystal X-ray diffraction.

Experimental details

All of the non-hydrogen atoms were refined anisotropically. The hydrogen atoms were assigned with common isotropic displacement factors U_{iso}(H) = 1.2 times U_{eq}(C, aromatic ring). The final refinement by using geometrical restraints, with C—H = 0.93 Å (aromatic ring), C—H = 1.00 Å (CH).

***Corresponding author: Zhi-Qiang Lu**, College of Chemistry and Chemical Engineering, Luoyang Normal University, Luoyang, Henan 471022, P. R. China, E-mail: zqlu2000@163.com

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

Atom	x	y	z	U_{iso}*/U_{eq}
Br1	0.65165 (5)	0.08642 (3)	0.37519 (3)	0.06294 (17)
F1	0.7290 (4)	−0.20471 (16)	0.59281 (17)	0.0827 (8)
F2	0.9822 (3)	−0.13818 (17)	0.71130 (17)	0.0727 (7)
F3	0.7419 (3)	−0.11413 (19)	0.72867 (17)	0.0773 (8)
O1	0.9990 (3)	0.24564 (17)	0.51948 (16)	0.0514 (7)
N1	1.0692 (4)	0.1522 (2)	0.64014 (19)	0.0476 (8)
N2	1.3663 (5)	0.7610 (3)	0.3753 (2)	0.0744 (12)
C1	0.8323 (5)	−0.1213 (3)	0.6631 (2)	0.0448 (10)
C2	0.8737 (4)	−0.0235 (3)	0.6208 (2)	0.0417 (9)
C3	1.0255 (5)	0.0628 (3)	0.6726 (2)	0.0470 (10)
H3	1.1017	0.0589	0.7331	0.056*
C4	0.9597 (5)	0.1577 (3)	0.5548 (2)	0.0449 (10)
C5	0.8017 (4)	0.0733 (3)	0.4967 (2)	0.0420 (9)
C6	0.7588 (4)	−0.0178 (3)	0.5301 (2)	0.0445 (10)
H6	0.6553	−0.0746	0.4931	0.053*
C7	1.1649 (6)	0.3298 (3)	0.5823 (3)	0.0529 (11)
H7A	1.268 (4)	0.301 (2)	0.592 (2)	0.042 (10)*
H7B	1.142 (5)	0.350 (3)	0.647 (3)	0.062 (11)*
C8	1.1935 (5)	0.4207 (3)	0.5304 (2)	0.0451 (9)
C9	1.1187 (5)	0.4135 (3)	0.4301 (3)	0.0581 (12)
H9	1.0397	0.3493	0.3895	0.070*
C10	1.1591 (5)	0.5003 (3)	0.3881 (3)	0.0586 (12)
H10	1.1078	0.4942	0.3198	0.070*
C11	1.2750 (5)	0.5957 (3)	0.4477 (3)	0.0512 (10)
C12	1.3470 (6)	0.6046 (3)	0.5485 (3)	0.0714 (14)
H12	1.4227	0.6694	0.5891	0.086*
C13	1.3075 (6)	0.5177 (3)	0.5903 (3)	0.0684 (14)
H13	1.3575	0.5242	0.6587	0.082*
C14	1.3228 (5)	0.6872 (3)	0.4055 (3)	0.0562 (11)
Br2	0.67363 (7)	0.25533 (4)	1.18819 (3)	0.0850 (2)
F4	0.8150 (4)	0.4869 (2)	0.9080 (2)	0.1080 (11)
F5	0.5773 (4)	0.5183 (2)	0.9082 (2)	0.1008 (10)
F6	0.5808 (5)	0.4261 (2)	0.78033 (18)	0.1218 (14)
O2	0.3809 (3)	0.08815 (18)	1.02212 (16)	0.0551 (7)
N3	0.3742 (4)	0.1712 (2)	0.8900 (2)	0.0508 (9)
N4	−0.0921 (5)	−0.3937 (3)	1.1830 (3)	0.0763 (12)
C15	0.6354 (6)	0.4429 (3)	0.8776 (3)	0.0582 (12)
C16	0.5726 (5)	0.3471 (3)	0.9178 (2)	0.0459 (9)
C17	0.4387 (5)	0.2576 (3)	0.8564 (2)	0.0503 (10)
H17	0.3910	0.2570	0.7890	0.060*
C18	0.4426 (5)	0.1726 (3)	0.9849 (2)	0.0441 (9)
C19	0.5811 (5)	0.2600 (3)	1.0530 (2)	0.0487 (10)
C20	0.6459 (5)	0.3487 (3)	1.0185 (2)	0.0515 (10)
H20	0.7366	0.4083	1.0615	0.062*
C21	0.2331 (6)	0.0002 (3)	0.9524 (3)	0.0532 (11)
H21A	0.129 (4)	0.031 (2)	0.920 (2)	0.046 (10)*
H21B	0.282 (4)	−0.025 (2)	0.905 (2)	0.040 (10)*
C22	0.1683 (5)	−0.0818 (3)	1.0092 (2)	0.0475 (10)
C23	0.0076 (5)	−0.1646 (3)	0.9586 (3)	0.0621 (12)
H23	−0.0567	−0.1667	0.8930	0.074*
C24	−0.0574 (5)	−0.2436 (3)	1.0049 (3)	0.0635 (12)
H24	−0.1652	−0.2988	0.9704	0.076*
C25	0.0372 (5)	−0.2409 (3)	1.1025 (3)	0.0504 (10)
C26	0.1952 (5)	−0.1586 (3)	1.1537 (3)	0.0550 (11)
H26	0.2586	−0.1561	1.2197	0.066*
C27	0.2603 (5)	−0.0786 (3)	1.1064 (2)	0.0519 (10)
H27	0.3669	−0.0227	1.1412	0.062*
C28	−0.0339 (6)	−0.3257 (3)	1.1496 (3)	0.0577 (11)

Comment

Both *O*-alkylation and *N*-alkylation of 2-pyridone are both an important molecular scaffold for their biological and pharmaceutical properties [6–8]. However, based on the Hard–Soft Acid–Base theory, the softer nitrogen atom will tends to bond the softer carbon atom than oxygen. And, the *N*-alkylation compound is more thermodynamic stable than *O*-alkylation. Thus, there are a large amount of methods for the *N*-alkylation for of 2-pyridone moiety, but the absolute selective synthesis of O-alkylation is even rarer. We have reported the syntheses and the structures of some related compounds [5, 9, 10].

Single crystal structure analysis reveals that the title compound crystallizes in the triclinic P-1 space group with two crystallographically independent molecules in the asymmetric unit (see the Figure). All bond lengths and angles are in the expected ranges. Every molecule contains of a substituted phenyl benzene ring and a pyridinyl ring, and the dihedral angles between the two planes are 21.1 and 12.2 °C in the two molecules, respectively.

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

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