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The crystal structure of [1-(4-(trifluoromethyl)phenyl)-3,4-dihydroquinolin-2(1H)-one], $C_{16}H_{12}F_3NO$

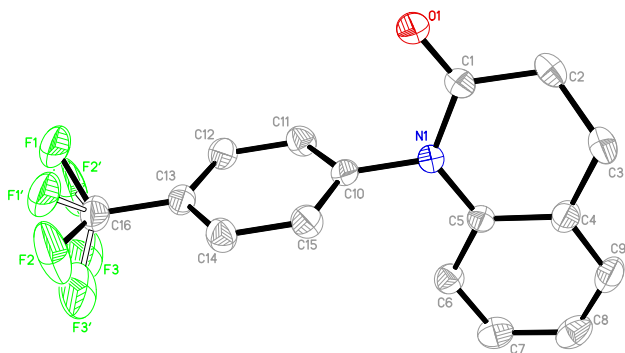


Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.40 × 0.33 × 0.31 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.12 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX II, φ and ω
$\theta_{\max}$, completeness:	25.0°, 97 %
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	5991, 2329, 0.085
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 1800
$N(\text{param})_{\text{refined}}$:	201
Programs:	Bruker [1], SHELX [2]

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Abstract

$C_{16}H_{12}F_3NO$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 6.9928(6)$ Å, $b = 8.9764(8)$ Å, $c = 21.216(2)$ Å, $V = 1331.7(2)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0583$, $wR_{\text{ref}}(F^2) = 0.1552$, $T = 298$ K.

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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.

1 Source of materials

1-(4-(trifluoromethyl)phenyl)-1,2,3,4-tetrahydroquinoline (5.0 mmol, 1.05 g) and cesium carbonate (Cs_2CO_3 , 7.5 mmol) were mixed in a 25 mL Schlenk tube containing a magnetic stirring bar, then we added dry DMF (50 mL) to the tube to

dissolve the substrate and finally added (diacetoxyiodo)benzene (1.0 mmol). Oxygen atmosphere was incorporated through an O_2 balloon. The resulting mixture was stirred at RT with the irradiation of a 20 W blue LED light for 36 h. After the reaction was completed, the reaction solution underwent an aqueous workup and was extracted three times with ethyl acetate. The combined organic layers were dried over sodium sulfate, and concentrated in vacuo. The title compound was purified by flash chromatography on silica gel using petroleum ether/ethyl acetate (10:1).

2 Experimental details

All hydrogen atomic positions were refined with variable isotropic displacement parameters. Hydrogen atoms were assigned with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times $U_{\text{iso}}(\text{C}, \text{phenyl ring and methylene carbon})$ and $U_{\text{iso}}(\text{H}) = 1.5$ times $U_{\text{eq}}(\text{C}, \text{methyl carbon})$. All the H atoms were refined as riding on their parent atom.

3 Comment

Nitrogen heterocyclic compounds are valuable and prevalent pharmacophores with diverse bioactivity [3], including anti-bacterial, anti-inflammatory, and anti-cancer activity, among others [4–7]. N-aryl tetrahydroquinolinone derivatives can be conveniently prepared according to the one-step

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

Atom	x	y	z	U _{iso} ^a /U _{eq}
F1 ^a	0.5240 (11)	0.4950 (7)	0.2014 (4)	0.100 (2)
F2 ^a	0.4364 (13)	0.5906 (14)	0.2861 (3)	0.120 (3)
F3 ^a	0.6505 (11)	0.6895 (9)	0.2334 (6)	0.117 (3)
F1 ^b	0.4046 (19)	0.5099 (11)	0.2596 (8)	0.100 (2)
F2 ^b	0.625 (2)	0.585 (2)	0.2007 (5)	0.120 (3)
F3 ^b	0.5472 (18)	0.6953 (15)	0.2792 (9)	0.117 (3)
N1	−0.0638 (4)	0.9572 (4)	0.09622 (16)	0.0388 (9)
O1	−0.1864 (4)	0.7488 (4)	0.05416 (18)	0.0568 (9)
C1	−0.1877 (6)	0.8832 (5)	0.0578 (2)	0.0434 (11)
C2	−0.3150 (7)	0.9794 (6)	0.0192 (2)	0.0570 (13)
H2A	−0.4252	0.9220	0.0058	0.068*
H2B	−0.2468	1.0113	−0.0183	0.068*
C3	−0.3814 (6)	1.1136 (6)	0.0548 (3)	0.0596 (14)
H3A	−0.4534	1.1781	0.0268	0.071*
H3B	−0.4655	1.0827	0.0887	0.071*
C4	−0.2166 (5)	1.1969 (6)	0.0813 (2)	0.0468 (11)
C5	−0.0591 (5)	1.1130 (5)	0.1010 (2)	0.0374 (10)
C6	0.0974 (5)	1.1859 (5)	0.1264 (2)	0.0459 (11)
H6	0.2034	1.1314	0.1394	0.055*
C7	0.0973 (7)	1.3366 (6)	0.1326 (2)	0.0570 (13)
H7	0.2028	1.3843	0.1500	0.068*
C8	−0.0563 (8)	1.4181 (6)	0.1134 (3)	0.0605 (14)
H8	−0.0567	1.5211	0.1180	0.073*
C9	−0.2088 (7)	1.3475 (6)	0.0874 (2)	0.0576 (14)
H9	−0.3116	1.4042	0.0734	0.069*
C10	0.0735 (5)	0.8710 (4)	0.1303 (2)	0.0384 (10)
C11	0.2299 (5)	0.8155 (6)	0.0997 (2)	0.0456 (11)
H11	0.2465	0.8334	0.0569	0.055*
C12	0.3621 (5)	0.7336 (5)	0.1323 (2)	0.0471 (12)
H12	0.4674	0.6939	0.1114	0.057*
C13	0.3396 (5)	0.7101 (4)	0.1952 (2)	0.0410 (10)
C14	0.1831 (6)	0.7657 (5)	0.2256 (2)	0.0481 (11)
H14	0.1670	0.7481	0.2685	0.058*
C15	0.0498 (6)	0.8470 (5)	0.1934 (2)	0.0459 (11)
H15	−0.0561	0.8857	0.2143	0.055*
C16	0.4824 (7)	0.6224 (6)	0.2297 (3)	0.0571 (13)

^aOccupancy: 0.611(5), ^bOccupancy: 0.389(5).

palladium-catalyzed N-arylation reaction. The construction of N-aryl tetrahydroisoquinolinones is regarded as a desirable and valuable synthetic goal [7–9], which would be of great significance to the synthetic and pharmaceutical fields [10, 11]. This electron-donor-acceptor complex-mediated oxidation process eliminates the need to use photo catalysts and allows for the effective preparation of a broad range of synthetically and biologically valuable quinolinones under very mild conditions. Mechanistic studies indicated a short radical chain reaction initiated by visible-light-induced electron transfer within the tertiary amine diacetoxiodo benzene electron-donor-acceptor complex [12]. Although its

molecular structure has been discovered, its crystal structure has not yet been reported.

The title compound contained one quinoline and one phenyl moiety. Owing to the carbons at positions C2 and C3 are saturated carbons containing two hydrogens, all the atoms of quinoline ring are not coplanar. And the torsion angles of C1–N1–C10–C15 and C5–N1–C10–C15 are −105.1° and 78.3°, respectively. The C1 position of the aromatic ring in the parent nucleus of 3,4-dihydroquinolin-2(1H)-one is oxidized. The trifluoromethyl group replaces the hydrogen in the benzene ring at position C13. All the bond lengths and angles are comparable with its analogues and in the expected ranges [13–16].

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

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