Zheng-Bao Qi, Man-Jing Zhu*, Xu-Liang Nie, Guo-Ping Huang and Jian-Ping Huang*

Crystal structure of 3-[(furan-2-ylmethyl)-amino]-2-(2,3,4,5-tetrafluoro-benzoyl)-acrylic acid ethyl ester, $C_{17}H_{13}F_4NO_4$

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Abstract

$C_{17}H_{13}F_4NO_4$, monoclinic, $P2_1/n$ (no. 14), a = 5.470(4) Å, b = 26.141(17) Å, c = 11.689(8) Å, β = 90.01°, V = 1671.3(19) Å^3, Z = 4, R_{gt} (F) = 0.0612, wR_{ref} (F^2) = 0.1650, T = 296(2) K.

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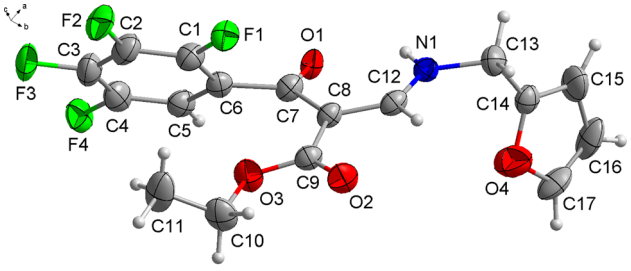
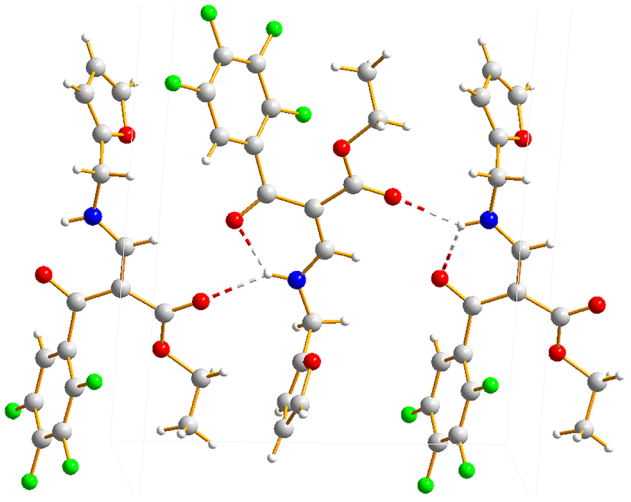
The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.





1 Source of materials

To a stirred solution of 3-dimethylamino-2-(2,3,4,5-tetrafluoro-benzoyl)-acrylic acid ethyl ester (31.9 g, 0.1 mol) in CH_2Cl_2 (150 mL) was added α-furan-2-yl-methylamine (1.0 g, 0.1 mol), and then the reaction mixture was refluxed for about 0.5 h. After the reaction completed (monitored by TLC), the CH_2Cl_2 was distillated under reduced pressure to give crude product. The crude product was poured into water (40 mL) and extracted with EtOAc (50 mL × 3). The EtOAc solvent was evaporated to provide 3-[(furan-2-ylmethyl)-amino]-2-(2,3,4,5-tetrafluoro-benzoyl)-acrylic acid ethyl ester being suitable for X-ray analysis in 85.0 % yield. ^{1}H (400 MHz) and ^{13}C (100 MHz) NMR spectra of 3-[(furan-2-ylmethyl)-amino]-2-

Table 1: Data collection and handling.

Crystal:	Colorless block
Size:	0.23 × 0.15 × 0.12 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	0.13 mm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ_{max}, completeness:	25.5°, >99 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	12318, 3110, 0.038
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 $\sigma(I_{obs})$, 2003
$N(param)_{refined}$:	236
Programs:	Bruker [1], SHELX [2, 3], Diamond [4]

***Corresponding authors: Man-Jing Zhu**, Academic Affairs Office, Jiangxi Open University, Nanchang 330046, People's Republic of China, E-mail: zhumanjing@jxrtvu.com. https://orcid.org/0000-0002-4788-5796 (M.-J. Zhu); and **Jian-Ping Huang**, Key Laboratory of Chemical Utilization of Plant Resources of Nanchang, College of Chemistry and Materials, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China; and Nanchang Jiangteng Pharmaceutical Ltd, Nanchang 330045, People's Republic of China, E-mail: huangchenchuan@126.com. https://orcid.org/0000-0001-9708-6856 (J.-P. Huang)
Zheng-Bao Qi, United Testing Services (Jiangxi) Co., Ltd., Nanchang 330001, People's Republic of China
Xu-Liang Nie, Key Laboratory of Chemical Utilization of Plant Resources of Nanchang, College of Chemistry and Materials, Jiangxi Agricultural University, Nanchang 330045, People's Republic of China. https://orcid.org/0000-0002-9449-8932
Guo-Ping Huang, Institute of Life Sciences, Jiangsu University, Zhenjiang 212013, People's Republic of China

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	0.4256 (7)	0.10776 (13)	0.1482 (3)	0.0640 (9)
C2	0.2953 (8)	0.06564 (14)	0.1825 (4)	0.0774 (11)
C3	0.0946 (8)	0.07244 (17)	0.2500 (4)	0.0809 (11)
C4	0.0326 (7)	0.12043 (17)	0.2814 (3)	0.0708 (10)
C5	0.1619 (6)	0.16284 (14)	0.2500 (3)	0.0605 (9)
H5	0.1158	0.1951	0.2757	0.073*
C6	0.3622 (6)	0.15700 (12)	0.1794 (2)	0.0525 (8)
C7	0.5149 (6)	0.20285 (11)	0.1511 (2)	0.0526 (8)
C8	0.5884 (6)	0.21432 (11)	0.0353 (2)	0.0488 (7)
C9	0.4732 (6)	0.19397 (12)	−0.0691 (3)	0.0539 (8)
C10	0.1507 (8)	0.14148 (17)	−0.1463 (3)	0.0848 (12)
H10A	0.0057	0.1607	−0.1673	0.102*
H10B	0.2596	0.1408	−0.2117	0.102*
C11	0.0838 (13)	0.0905 (2)	−0.1161 (4)	0.143 (2)
H11A	0.2276	0.0695	−0.1128	0.215*
H11B	−0.0264	0.0771	−0.1725	0.215*
H11C	0.0050	0.0907	−0.0427	0.215*
C12	0.7787 (6)	0.24847 (11)	0.0165 (3)	0.0527 (8)
H12	0.8230	0.2534	−0.0595	0.063*
C13	1.0980 (6)	0.31036 (13)	0.0625 (3)	0.0636 (9)
H13A	1.1491	0.3042	−0.0157	0.076*
H13B	1.2378	0.3045	0.1119	0.076*
C14	1.0188 (6)	0.36375 (13)	0.0739 (3)	0.0577 (8)
C15	1.1403 (9)	0.40227 (17)	0.1157 (4)	0.1003 (15)
H15	1.2973	0.4017	0.1462	0.120*
C16	0.9833 (15)	0.44570 (19)	0.1051 (5)	0.132 (2)
H16	1.0164	0.4784	0.1322	0.158*
C17	0.7904 (13)	0.43205 (19)	0.0524 (5)	0.1170 (19)
H17	0.6623	0.4536	0.0318	0.140*
F1	0.6232 (4)	0.09953 (8)	0.0822 (2)	0.0898 (7)
F2	0.3637 (6)	0.01847 (9)	0.1490 (3)	0.1212 (10)
F3	−0.0329 (6)	0.03107 (11)	0.2835 (3)	0.1268 (11)
F4	−0.1667 (5)	0.12543 (11)	0.3494 (2)	0.1085 (9)
N1	0.9043 (5)	0.27469 (9)	0.0925 (2)	0.0562 (7)
H1	0.8700	0.2705	0.1637	0.067*
O1	0.5822 (5)	0.22975 (9)	0.23312 (18)	0.0718 (7)
O2	0.5445 (5)	0.20273 (10)	−0.16518 (19)	0.0744 (7)
O3	0.2722 (5)	0.16653 (10)	−0.05031 (19)	0.0758 (7)
O4	0.8016 (6)	0.37999 (12)	0.0304 (3)	0.1080 (11)

(2,3,4,5-tetrafluoro-benzoyl)-acrylic acid ethyl ester were recorded on a Bruker Avance 400 spectrometer in $CDCl_3$ using tetramethylsilane (TMS) and $CDCl_3$ (13C, δ 77.0 ppm) as internal standards. *J*-values were given in Hertz. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.09–1.13 (t, *J* = 8.0, 3H, OCH_2CH_3), 4.05–4.10 (q, *J* = 8.0, 12.0 Hz, 2H, OCH_2CH_3), 4.57–4.59 (d, *J* = 8.0 Hz, 2H, NHCH2), 6.34–6.39 (m, 2H, Furanyl-H), 6.95–7.01 (m, 1H, Ph–H), 7.45–7.47 (d, *J* = 8.0 Hz, 1H, OCH=C), 8.18–8.21 (d, *J* = 12.0 Hz, 1H, C=CHNH), 10.98 (br, 1H, NH) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 187.14, 166.25, 160.56, 160.11, 148.29, 143.69, 143.58, 143.50, 110.72, 109.98, 109.79, 109.29, 109.09, 101.62, 60.06, 46.50, 14.00 ppm.

2 Experimental details

All H atoms were included in calculated positions and refined as riding atoms, with C–H = 0.90–0.97 Å with U_{iso}(H) = 1.5 U_{eq}(C) for methyl H atoms and 1.2 U_{eq}(C) for all other H atoms.

3 Comment

Quinolones have been popularized rapidly in clinic and become significant drug in the development, production and application all over the world due to wider antibacterial spectrum and stronger antibacterial activity than common antibiotics [5, 6]. After being used or even abused for a long time, quinolones have been seriously resisted by bacteria because of gene mutations and also have the inevitable side effects [7–9]. Nowadays, a new effective fluoroquinolones against bacteria drug-resistant and side effects have been researched and developed [10–13]. Recently, an impactful and high-yielding method for the synthesis of fluoroquinolones is developed in our group, and single crystals of several key intermediates are achieved.

In the molecule of the title compound bond lengths and angles within 3-[(furan-2-ylmethyl)-amino]-2-(2,3,4,5-tetra fluoro-benzoyl)-acrylic acid ethyl ester are very similar to those given in the literature [11, 12]. The dihedral angles between the C1–C6 benzene ring, O2–C9–O3 carboxyl group and the furan ring are 61.0 (1)°, 66.2 (3)° and 86.3 (3)°. The torsion angles of C5–C6–C7–C8, C6–C7–C8–C9, C6–C7–C8–C12, C7–C8–C12–N1, C8–C12–N1–C13, C12–N1–C13–C14 and N1–C13–C14–C15 are −132.6 (3)°, 18.9 (5)°, −163.1 (3)°, −2.5 (5)°, −178.7 (3)°, 104.7 (4)°, and 138.6 (4)°, respectively. Intermolecular N–H···O hydrogen bonds between nitrogen atom (N1) of amino group and oxygen atoms (O2) of carboxyl group connect the adjacent title compound to form a one dimensional chain.

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

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