

Cheng Ma* and Yewei Yang

Department of Chemistry, Zhejiang University,
310027 Hangzhou, People's Republic of China

Correspondence e-mail: mcorg@zju.edu.cn

Key indicators

Single-crystal X-ray study
 $T = 295$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.039
 wR factor = 0.122
Data-to-parameter ratio = 14.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Dimethyl 3-(benzylamino)-2-(4-fluorophenyl)-
furan-4,5-dicarboxylate

In the crystal structure of the title compound, $\text{C}_{21}\text{H}_{18}\text{FNO}_5$, one benzene ring is almost perpendicular to the furan ring, forming a dihedral angle of $87.78(9)^\circ$. $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds link the molecules in the crystal structure into sheets parallel to (100).

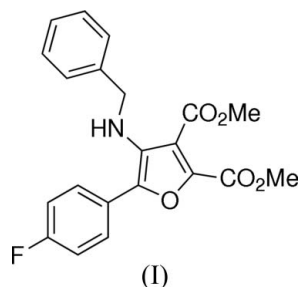
Received 15 September 2005

Accepted 29 September 2005

Online 5 October 2005

Comment

Substituted furans play an important role in organic chemistry, not only as key structural units in many natural products and important pharmaceuticals (Dean, 1963; Nakanishi *et al.*, 1974), but also as useful building blocks in synthetic chemistry (Lipshutz, 1986; Raczko & Jurcak, 1995). Recently, we reported a facile synthesis of polysubstituted 3-aminofurans *via* a thiazole carbene-mediated multicomponent reaction (Ma & Yang, 2005). In this paper, we report the crystal structure of dimethyl 3-(benzylamino)-2-(4-fluorophenyl)-furan-4,5-dicarboxylate, (I).



The molecular structure of (I) is shown in Fig. 1. Selected geometric parameters and hydrogen-bond geometry are listed in Tables 1 and 2, respectively. The dihedral angle between the 4-fluoro-substituted benzene ring and the furan ring is $15.96(8)^\circ$, while the other benzene ring is almost perpendicular to the furan ring, with a dihedral angle of $87.78(9)^\circ$.

In the crystal structure, $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonding is found which links the molecules into sheets that are parallel to (100) (Fig. 2).

Experimental

To a suspension of NaH (1.5 mmol) in anhydrous CH_2Cl_2 (3 ml), a solution 3-benzyl-5-(2-hydroxyethyl)-4-methyl-1,3-thiazolium chloride (1.0 mmol) in dry CH_2Cl_2 (2 ml) was added at 195 K under nitrogen. After 10–15 min, a mixture of *p*-fluorobenzaldehyde (0.5 mmol) and dimethyl acetylenedicarboxylate (0.75 mmol) in CH_2Cl_2 (2 ml) was added over a period of 10 min and afterwards stirred for 2 h. Afterwards, the reaction mixture was heated to 273 K and kept at this temperature for 2 h. The resulting mixture was carefully poured into an ice-cooled solution of NaHCO_3 and then

extracted with dichloromethane. The combined organic phases were washed with brine and dried over anhydrous Na_2SO_4 . The solvent was removed under reduced pressure and the resulting oil was purified by column chromatography to afford the product (I) (65 mg, yield 32%). Compound (I) was recrystallized from AcOEt as pale-yellow crystals (m.p. 363–364 K). ^1H NMR (500 MHz, CDCl_3): 7.89–7.86 (m, 2H), 7.28–7.25 (m, 3H), 7.15–7.14 (m, 4H), 4.50 (brs, 1H), 4.03 (s, 2H), 3.91 (s, 3H), 3.82 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3): δ 163.6 (d, $J_{\text{C-F}} = 13.3$ Hz), 161.7, 158.5, 142.9, 140.8, 139.1, 132.5, 128.8 (d, $J_{\text{C-F}} = 9.0$ Hz), 128.0, 127.6, 126.2 (d, $J_{\text{C-F}} = 3.6$ Hz), 120.2, 116.1, 115.9, 52.6, 52.5, 52.3; HRMS (ESI): m/z , calculated for $[\text{C}_{21}\text{H}_{18}\text{FNO}_5 + \text{Na}]^+$: 406.1061; found: 406.1060; IR (KBr): 3369, 2954, 1734, 1729, 1606, 1500, 1437, 1327, 1221, 1202.

Crystal data

$\text{C}_{21}\text{H}_{18}\text{FNO}_5$	$D_x = 1.373 \text{ Mg m}^{-3}$
$M_r = 383.38$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 11909 reflections
$a = 19.6280$ (4) Å	$\theta = 1.9\text{--}27.5^\circ$
$b = 13.1216$ (3) Å	$\mu = 0.11 \text{ mm}^{-1}$
$c = 15.2962$ (4) Å	$T = 295$ (1) K
$\beta = 109.6406$ (14)°	Block, colorless
$V = 3710.34$ (15) Å ³	$0.60 \times 0.55 \times 0.55 \text{ mm}$
$Z = 8$	

Data collection

Rigaku R-Axis RAPID diffractometer	4264 independent reflections
ω scans	2715 reflections with $F^2 > 2\sigma(F^2)$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$R_{\text{int}} = 0.025$
$T_{\text{min}} = 0.859$, $T_{\text{max}} = 0.944$	$\theta_{\text{max}} = 27.5^\circ$
17710 measured reflections	$h = -25 \rightarrow 23$
	$k = -17 \rightarrow 17$
	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	$w = 1/[0.0018F_o^2 + \sigma(F_o^2)]/(4F_o^2)$
$R[F^2 > 2\sigma(F^2)] = 0.039$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$wR(F^2) = 0.122$	$\Delta\rho_{\text{max}} = 0.26 \text{ e } \text{Å}^{-3}$
$S = 1.00$	$\Delta\rho_{\text{min}} = -0.31 \text{ e } \text{Å}^{-3}$
4264 reflections	Extinction correction: Larson (1970), equation 22
254 parameters	Extinction coefficient: $2.4(4) \times 10^2$
H-atom parameters constrained	

Table 1

Selected geometric parameters (Å, °).

F1—C8	1.3618 (18)	O3—C18	1.3170 (17)
O1—C1	1.3734 (15)	N1—C2	1.4002 (17)
O2—C18	1.2053 (19)	N1—C11	1.470 (2)
C1—O1—C4	107.86 (11)	C2—N1—C11	116.19 (11)
C18—O3—C19	116.18 (13)	N1—C11—C12	117.40 (12)

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H101 \cdots O2	0.97	2.28	3.009 (2)	132
C13—H13 \cdots O4 ⁱ	0.97	2.49	3.392 (2)	155
C15—H15 \cdots O2 ⁱⁱ	0.97	2.47	3.430 (2)	172

Symmetry codes: (i) $-x + \frac{1}{2}, -y + \frac{3}{2}, -z + 1$; (ii) $+x, -y + 1, +z + \frac{1}{2}$.

The NH H atom was found in a difference Fourier map and refined as riding, with $N\text{—}H = 0.97$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$. Other H atoms were placed in calculated positions and refined using a riding model with $C\text{—}H = 0.97$ Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

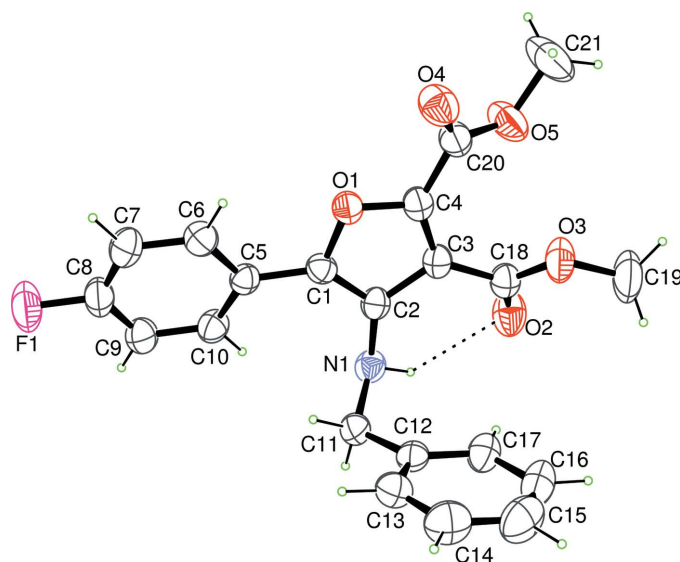


Figure 1

The crystal structure of (I), with displacement ellipsoids drawn at the 50% probability level. The dashed line indicates an intramolecular hydrogen bond.

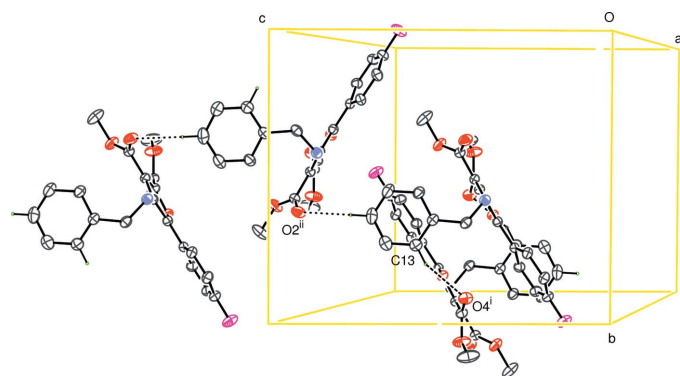


Figure 2

The molecular packing of (I). Dashed lines indicate hydrogen-bonding interactions. H atoms not involved in hydrogen bonding have been omitted. [Symmetry codes: (i) $\frac{1}{2} - x, 1.5 - y, 1 - z$; (ii) $x, 1 - y, \frac{1}{2} + z$.]

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *CrystalStructure* and *PLATON* (Spek, 2003).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K. & Watkin, D. J. (2003). *J. Appl. Cryst.* **36**, 1487.
- Dean, F. A. (1963). *Naturally Occurring Oxygen Ring Compounds*. London: Butterworth.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
- Larson, A. C. (1970). *Crystallographic Computing*, edited by F. R. Ahmed, S. R. Hall & C. P. Huber, pp. 291–294. Copenhagen: Munksgaard.

- Lipshutz, B. H. (1986). *Chem. Rev.* **86**, 795–819.
- Ma, C. & Yang, Y. (2005). *Org. Lett.* **7**, 1343–45.
- Nakanishi, K., Goto, T., Ito, S., Natori, S. & Nozoe, S. (1974). Editors. *Natural Products Chemistry*, Vols. 1–3. Tokyo: Kodansha.
- Raczko, J. & Jurcak, J. (1995). *Stud. Nat. Prod. Chem.* **16**, 639–726.
- Rigaku (1998). *PROCESS-AUTO*. Version 1.06. Rigaku Corporation, Tokyo, Japan.
- Rigaku/MSK (2004). *CrystalStructure*. Version 3.7.0. Rigaku/MSK, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
- Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.