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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.048
 wR factor = 0.144
Data-to-parameter ratio = 16.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(E)-Dimethyl 4-(ethylamino)-5-styrylfuran-2,3-dicarboxylate**

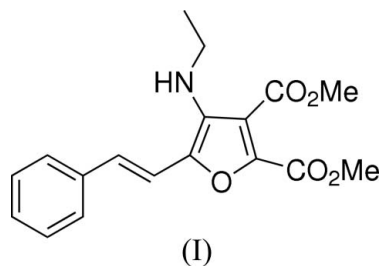
The title compound, $\text{C}_{18}\text{H}_{19}\text{NO}_5$, synthesized *via* a thiazole carbene-mediated multicomponent reaction, has a crystal structure stabilized by π - π stacking interactions involving the phenyl and furan rings.

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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 *et al.*, 2005). In this paper, we report the crystal structure of a compound belonging to this class of heterocycles, (I).



The molecular structure of (I) is depicted in Fig. 1. There is a π - π stacking interaction in the crystal structure, involving the phenyl and furan rings (Fig. 2). This can be described in terms of a $\text{Cg}1 \cdots \text{Cg}2(1+x, y, z)$ centroid-to-centroid distance of 3.628 (3) Å, where $\text{Cg}1$ is the centroid of the furan ring and $\text{Cg}2$ is the centroid of the phenyl ring.

Experimental

To a suspension of NaH (1.2 mmol) in anhydrous CH_2Cl_2 (3 ml), a solution of 3-ethyl-4-methylthiazolium bromide (1.0 mmol) in dry CH_2Cl_2 (2 ml) was added at 195 K under nitrogen. After 10–15 min, a mixture of cinnamaldehyde (0.5 mmol) and dimethyl acetylenedicarboxylate (0.75 mmol) in CH_2Cl_2 (2 ml) was added over a period of 10 min, then stirred at 195 K for 2 h. Afterwards, the reaction temperature was raised slowly to 273 K over a period of 1 h, and kept at 273 K for an additional 5 h. The resulting mixture was carefully poured into ice-cooled NaHCO_3 and then extracted with CH_2Cl_2 (10 ml). 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 (I) (yield 77 mg, 47%). Compound (I) was recrystallized from EtOAc as orange crystals (m.p. 391–392 K).

Crystal data

$C_{18}H_{19}NO_5$
 $M_r = 329.34$
 Triclinic, $P\bar{1}$
 $a = 7.765$ (4) Å
 $b = 9.043$ (4) Å
 $c = 13.044$ (11) Å
 $\alpha = 70.17$ (3)°
 $\beta = 82.36$ (3)°
 $\gamma = 75.52$ (3)°

$V = 833.1$ (9) Å³
 $Z = 2$
 $D_x = 1.313$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 298$ (2) K
 Needle, orange
 $0.57 \times 0.18 \times 0.16$ mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 ω scans
 Absorption correction: none
 8262 measured reflections

3772 independent reflections
 2119 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.027$
 $\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.144$
 $S = 0.99$
 3772 reflections
 225 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0782P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.26$ e Å⁻³
 $\Delta\rho_{min} = -0.27$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.032 (6)

Amine atom H1 was found in a difference map and refined with free coordinates [$N-H = 0.84$ (2)] and an isotropic displacement parameter. The methyl H atoms were constrained to an ideal geometry [$C-H = 0.96$ Å and $U_{iso}(H) = 1.5U_{eq}(C)$] and were allowed to rotate freely about the C–C bonds. Aromatic and H atoms of the methylene groups were placed in calculated positions, with C–H distances of 0.93 and 0.97 Å, respectively, and were included in the final cycles of refinement as riding on their carrier atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MS, 2004); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
 Dean, F. A. (1963). *Naturally Occurring Oxygen Ring Compounds*. London: Butterworth.

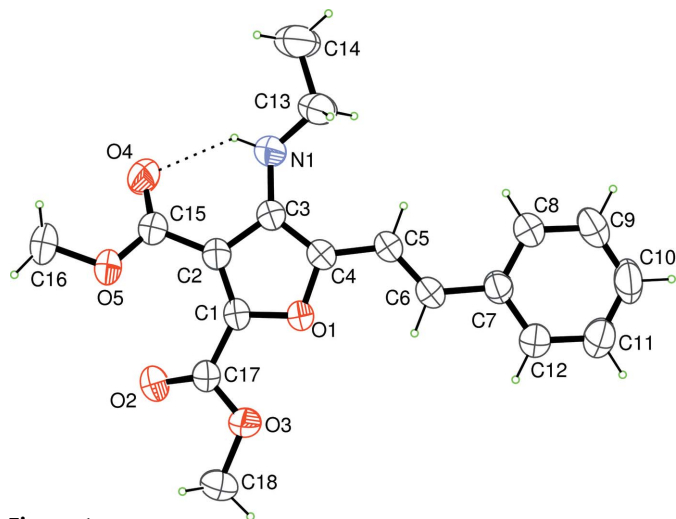


Figure 1
 The molecular structure of (I), with displacement ellipsoids drawn at the 50% probability level for non-H atoms. The dotted line represents a hydrogen bond.

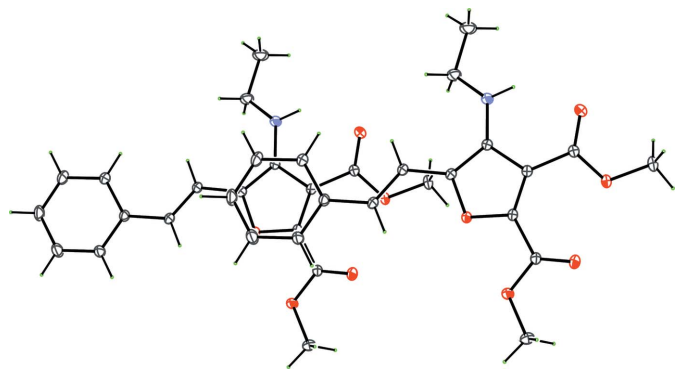


Figure 2
 The π - π stacking in the crystal structure of (I), viewed perpendicular to the phenyl ring.

- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Lipshutz, B. H. (1986). *Chem. Rev.* **86**, 795–819.
 Ma, C., Ding, H., Wu, G. & Yang, Y. (2005). *J. Org. Chem.* **70**, 8919–8923.
 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/MS (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MS, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.