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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 = 0.056
 wR factor = 0.187
Data-to-parameter ratio = 17.9For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(E)-Dimethyl 4-butylamino-5-(4-methylstyryl)-furan-2,3-dicarboxylate**

The title compound, $\text{C}_{21}\text{H}_{25}\text{NO}_5$, was synthesized *via* a thiazole carbene-mediated multicomponent reaction. Without π - π stacking or other weak intermolecular interactions, the crystal packing is controlled by van der Waals forces.

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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).

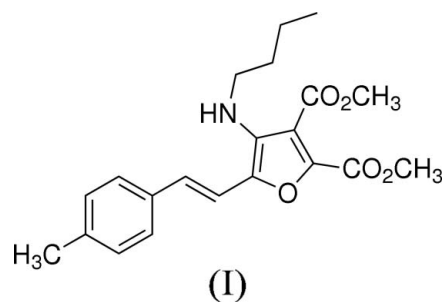


Fig. 1 shows the structure of (I). There are no obvious π - π stacking or other weak intermolecular interactions in (I), and the crystal packing is controlled by van der Waals forces.

Experimental

To a suspension of NaH (1.2 mmol) in anhydrous CH_2Cl_2 (3 ml), a solution of 3-butyl-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 4-methylcinnamaldehyde (0.5 mmol) and dimethyl acetylenedicarboxylate (0.75 mmol) in CH_2Cl_2 (2 ml) was added over a period of 10 min, and the mixture was stirred at this temperature for 2 h. Afterwards, the reaction temperature was raised slowly to 273 K within 1 h, and kept at 273 K for an additional 5 h. The resulting mixture was carefully poured in to an ice-cooled aqueous NaHCO_3 solution 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 [on Merck silica gel Kieselgel 60 (300–400 mesh) with a hexane–ethyl acetate mixture (2:1)] to afford the product, (I) (65 mg, yield 35%). Compound (I) was recrystallized from EtOAc as orange crystals (m.p. 391–392 K).

Crystal data

$C_{21}H_{25}NO_5$
 $M_r = 371.42$
 Triclinic, $P\bar{1}$
 $a = 5.446$ (3) Å
 $b = 10.454$ (7) Å
 $c = 18.386$ (12) Å
 $\alpha = 77.94$ (3)°
 $\beta = 88.97$ (2)°
 $\gamma = 78.68$ (2)°

$V = 1003.5$ (11) Å³
 $Z = 2$
 $D_x = 1.229$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 298$ (2) K
 Prism, orange
 $0.4 \times 0.35 \times 0.12$ mm

Data collection

Rigaku R-AXIS RAPID
 diffractometer
 ω scans
 Absorption correction: none
 9965 measured reflections

4536 independent reflections
 2368 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.033$
 $\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.056$
 $wR(F^2) = 0.187$
 $S = 1.07$
 4536 reflections
 253 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$w = 1/[\sigma^2(F_o^2) + (0.0923P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.22$ e Å⁻³
 $\Delta\rho_{min} = -0.25$ e Å⁻³
 Extinction correction: *SHELXL97*
 (Sheldrick, 1997)
 Extinction coefficient: 0.014 (4)

Atom H1, attached to N1, was found in a difference Fourier map and refined freely. 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. The H atoms of the methylene groups and of the aromatic ring were placed in calculated positions, with C—H distances of 0.97 and 0.93 Å, respectively, and were included in the final cycles of the least-squares 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).

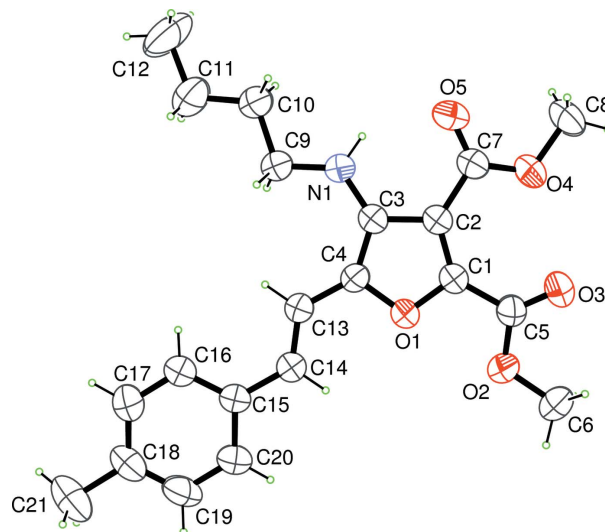


Figure 1
 The molecular structure of compound (I). Displacement ellipsoids are drawn at the 50% probability level.

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