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2-[2-(4-Methoxyphenyl)vinyl]-3-[(2-maleimidyl)-acetyl]-2,3-dihydro-1,3-benzothiazole

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Key indicators

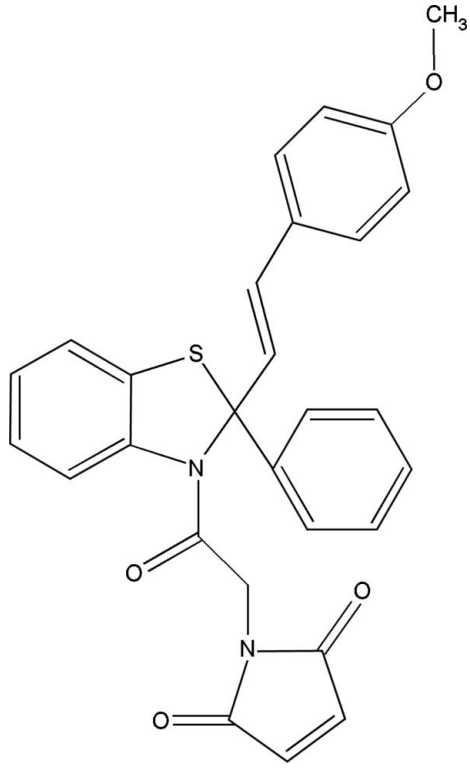
Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.102
Data-to-parameter ratio = 13.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The crystal packing of the title compound {systematic name: 3-[(2,3-dioxo-3-pyrrolin-1-yl)acetyl]-2-[2-(4-methoxyphenyl)-vinyl]-2,3-dihydro-1,3-benzothiazole}, $C_{28}H_{22}N_2O_4S$, is stabilized by intermolecular C—H···O and C—H···S hydrogen bonds and by π–π stacking interactions occurring between a benzene ring and the pyrrole ring of centrosymmetrically related molecules.

Comment

Nitrogen- and sulfur-containing heterocyclic compounds such as benzothiazoles and benzothiazepines have recently received considerable attention due to their wide range of physiological activities (Choi *et al.*, 2006; Mortimer *et al.*, 2006; Ansari *et al.*, 2005). Cycloaddition reactions of 1,5-benzothiazepines with ketenes may permit the construction of 2-azetidinone derivatives. When we reacted 4-(4-methoxyphenyl)-2-phenyl-1,5-benzothiazepine with 2-*tert*-butoxy-*N*-glycinyl succinimide, the title compound, (I), a derivative of benzothiazole, was obtained accidentally.



(I)

The molecular structure of the title compound, (I), is shown in Fig. 1. In (I), bond distances and angles are as expected for a

molecule of this kind (Allen *et al.*, 1987). The benzothiazole ring system is not planar, with the C9 atom displaced by 0.430 (2) Å from the plane of the remaining atoms [mean deviation 0.029 (2) Å]. The molecular conformation is stabilized by two weak intramolecular C—H···O and C—H···N hydrogen-bond interactions (Table 1).

The crystal packing of (I) is stabilized by intermolecular C—H···O and C—H···S hydrogen bonds (Table 1). In addition, there are π–π stacking interactions involving the C1–C6 benzene ring and the pyrrole ring of centrosymmetrically related molecules [$Cg1\cdots Cg2^{i} = 3.744$ (4) Å; $Cg1$ and $Cg2$ are the centroids of the C1–C6 and N1/C24–C25 rings, respectively; symmetry code: (i) $1-x, -y, -z$].

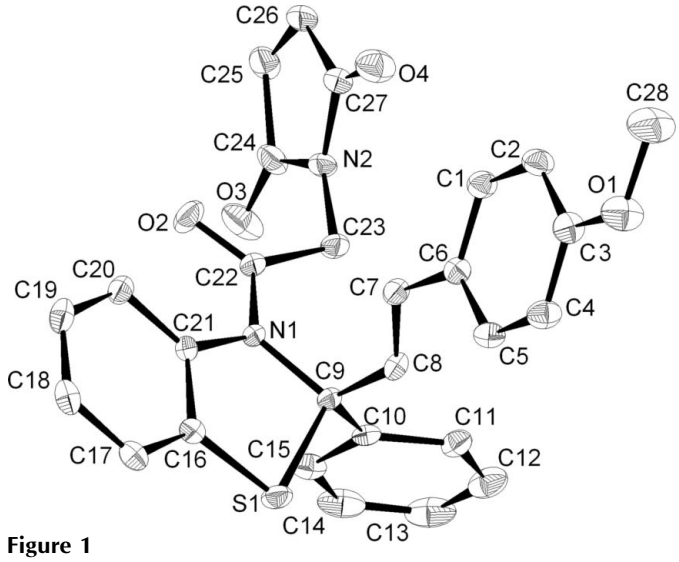


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

A mixture of 2-*tert*-butoxy-*N*-glycinyl succinimide (20 mmol) and $SOCl_2$ (30 ml) was refluxed for 4 h. After the reaction was complete, the $SOCl_2$ was removed. 4-(4-Methoxyphenyl)-2-phenyl-1,5-benzothiazepine (10 mmol), benzene (30 ml) and Et_3N (6 ml) were added and the mixture was stirred at room temperature for 1 h. After the reaction was complete, the mixture was filtered and the filtrate was washed with 10% HCl; the benzene layer was separated and dried over anhydrous Na_2SO_4. The benzene solution was evaporated and the residue was purified by column chromatography (ethyl acetate–petroleum ether = 1:3) to give the title product. Analysis calculated for $C_{28}H_{22}N_2O_4S$: C 69.63, H 4.56, N 5.80%; found: C 69.59, H 4.60, N 5.75%.

Crystal data

$C_{28}H_{22}N_2O_4S$
$M_r = 482.54$
Monoclinic, $P2_1/n$
$a = 9.970$ (5) Å
$b = 10.191$ (5) Å
$c = 23.941$ (11) Å
$\beta = 94.310$ (7)°
$V = 2426$ (2) Å^3
$Z = 4$
$D_x = 1.321$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.17$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.28 × 0.22 × 0.16 mm

Data collection

Bruker APEX II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.952$, $T_{max} = 0.973$
12808 measured reflections
4258 independent reflections
3012 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 25.0°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.102$
$S = 1.03$
4258 reflections
317 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0413P)^2 + 0.6152P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.34$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···N1	0.93	2.53	2.867 (3)	102
C20—H20···O2	0.93	2.26	2.789 (3)	116
C8—H8···S1^{i}	0.93	2.83	3.734 (2)	165
C18—H18···O2^{ii}	0.93	2.53	3.243 (3)	134

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

All H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93–0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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