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2a-(4-Chlorophenyl)-2-(2,5-dioxo-3-pyrazolin-1-yl)-4-phenyl-2,2a,3,4-tetrahydro-1*H*-azeto[2,1-*d*][1,5]-benzothiazepin-1-one

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Key indicators

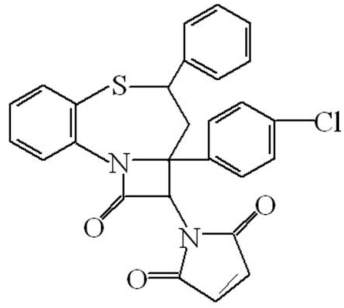
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.108
Data-to-parameter ratio = 13.5

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

The conformation of the seven-membered ring in the title molecule, $C_{27}H_{19}ClN_2O_3S$, is that of a chair. The 2,5-dioxo-3-pyrazolin-1-yl and 4-chlorophenyl groups attached to the β-lactam ring are in *cis* positions.

Comment

1,5-Benzothiazepines are useful and important compounds utilized in drug research owing to their well known bioactivities (Ansari *et al.*, 2005; Dandia *et al.*, 1998). Cycloaddition reactions of their C═N double bond with ketene may produce 2-azetidinone which is the essential component of some antibiotics (Gerard *et al.*, 2004). We are interested in the synthesis of 2-azetidinone derivatives of 1,5-benzothiazepines and we report here the synthesis and the structure of the title compound, (I).



(I)

Compound (I) was obtained by the cycloaddition of 2-*tert*-butoxy-*N*-glycinyl succinimide with 4-(4-chlorophenyl)-2-phenyl-1,5-benzothiazepine. The C—O bond of the *tert*-butoxy group is also cleaved and subsequent elimination of H_2O yielded the double bond, in agreement with previous work (Ping *et al.*, 2006).

A view of the molecule is shown in Fig. 1. The seven-membered ring in the molecule has a chair conformation, while in the 1,5-benzothiazepine it has a boat form (Lu *et al.*, 1987). Thus, the conformation of the seven-membered ring changes in the cycloaddition reaction. The four-membered ring in the molecule results from the cycloaddition reaction. The 2,5-dioxo-3-pyrazolin-1-yl and chlorophenyl groups attached to C22 and C15 are oriented away from the plane of the four-membered ring and are in *cis* positions.

Experimental

A mixture of 2-*tert*-butoxy-*N*-glycinyl succinimide (20 mmol) and $SOCl_2$ (30 ml) was refluxed for 4 h. When the reaction was complete,

$SOCl_2$ was removed. 4-(4-Chlorophenyl)-2-phenyl-1,5-benzothiazepine (10 mmol), benzene (30 ml) and Et_3N (6 ml) were added to the above mixture and 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 $C_{27}H_{19}ClN_2O_3S$: C 66.54, H 3.90, N 5.75%; found: C 66.49, H 3.97, N 5.70%.

Crystal data

$C_{27}H_{19}ClN_2O_3S$	$Z = 4$
$M_r = 486.95$	$D_x = 1.371$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 12.3034$ (12) Å	$\mu = 0.28$ mm^{-1}
$b = 16.6438$ (16) Å	$T = 293$ (2) K
$c = 12.2606$ (12) Å	Block, colorless
$\beta = 110.041$ (1)°	0.26 × 0.22 × 0.18 mm
$V = 2358.6$ (4) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	12674 measured reflections
	4157 independent reflections
φ and ω scans	2966 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)	$R_{int} = 0.025$
	$\theta_{max} = 25.0°$
$T_{min} = 0.834$, $T_{max} = 0.855$ (expected range = 0.927–0.950)	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 1.0352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.041$	
$wR(F^2) = 0.108$	
$S = 1.03$	$(\Delta/\sigma)_{max} < 0.001$
4157 reflections	$\Delta\rho_{max} = 0.26$ e Å^{-3}
307 parameters	$\Delta\rho_{min} = -0.32$ e Å^{-3}
H-atom parameters constrained	

H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H = 0.93 or 0.97 Å (aryl H) and $U_{iso}(H) = 1.2U_{eq}(C)$

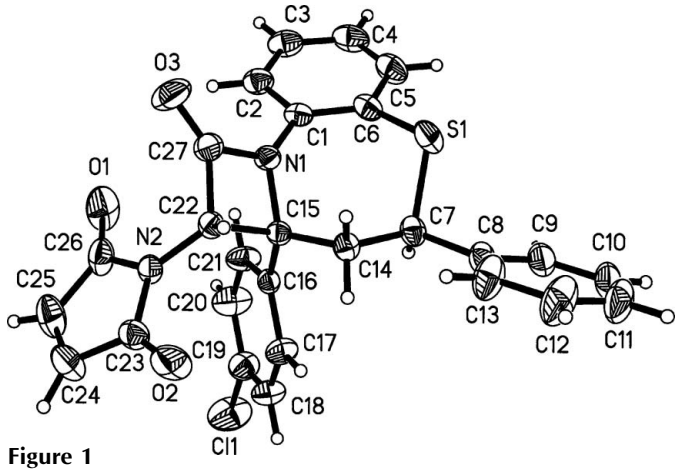


Figure 1
The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

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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References

Ansari, F. L., Umbreen, S., Hussain, L., Makhmoor, T. & Nawaz, S. (2005). *Chem. Biodiv.* **2**, 487–496.
Bruker (1997). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
Dandia, A., Upreti, M., Rani, B., Pant, U. C. & Gupta, I. J. (1998). *J. Fluorine Chem.* **91**, 171–174.
Gerard, S., Galleni, M., Dive, G. & Marchand, B. J. (2004). *Bioorg. Med. Chem.* **12**, 129–138.
Lu, Y. C., Liu, P. & Jin, S. (1987). *Sci. Sin. Ser. B*, **4**, 339–348.
Ping, Z., Na, L. & Yuan, L. (2006). *Acta Cryst.* E**62**, o3523–o3524.
Sheldrick, G. M. (1997). *SADABS*, *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.