Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(*N*-Phenylacetamido)-6*H*-1,3-thiazin-6-one

Michel Evain,[a]* Cyrille Landreau,[b] David Deniaud,[b] Alain Reliquet[b] and Jean Claude Meslin[b]

[a]Institut des Matériaux Jean Rouxel, 2 rue de la Houssinière, BP 32229, 44322 Nantes CEDEX 3, France, and [b]Laboratoire de Synthèse Organique, UMR CNRS 6513, Faculté des Sciences et des Techniques, 2 rue de la Houssinière, BP 92208, 44322 Nantes CEDEX 3, France

Correspondence e-mail: evain@cnrs-imn.fr

Key indicators

Single-crystal X-ray study
T = 300 K
Mean σ(C–C) = 0.002 Å
R factor = 0.045
wR factor = 0.119
Data-to-parameter ratio = 24.9

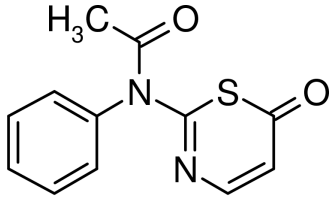
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 31 January 2002
Accepted 7 February 2002
Online 22 February 2002

The title compound, $C_{12}H_{10}O_2N_2S$, is a potential antitumor agent. The compound, obtained by the reaction of 2,4-diamino-1-thia-3-azabutadiene with ketene, crystallizes with monoclinic symmetry in space group $P2_1$.

Comment

Heterocyclic compounds containing S and N atoms have attracted much attention because of their broad spectrum of biological activities (Wishka *et al.*, 1998). In particular, 1,3-thiazinones and pyrimidinethiones have been explored as antitumor (Mayer *et al.*, 1999) and antibacterial agents (Vig *et al.*, 1998), as well as antiviral agents for human immunodeficiency virus (HIV) (Danel *et al.*, 1997). We report here the structure of one such thiazinone, 2-(*N*-phenylacetamido)-6*H*-1,3-thiazin-6-one, (I), which is a useful intermediate for the synthesis of *N*-deacylated 2-amino-1,3-thiazine-6-thione. The thiazine ring and amide group are nearly planar. The phenyl substituent is approximately perpendicular to the acetamido-thiazin-6-one ring, with a dihedral angle of 79.04 (4)°.



(I)

Experimental

The synthesis was carried out starting from 2,4-diamino-1-thia-3-azabutadiene and ketene (Landreau *et al.*, 2000). Ketene, formed by cracking of acetone, was passed through a solution of azabutadiene (4 mmol) in anhydrous dichloromethane (150 ml) until the starting product had disappeared. The mixture was cautiously evaporated and the residue was purified twice by flash chromatography (silica gel, dichloromethane/ethyl acetate, 9/1). Single crystals suitable for X-ray analysis were obtained by slow evaporation at room temperature from diethyl ether.

Crystal data

$C_{12}H_{10}N_2O_2S$
M_r = 246.3
Monoclinic, $P2_1/c$
a = 6.0696 (5) Å
b = 18.0716 (9) Å
c = 10.8780 (8) Å
β = 99.579 (10)°
V = 1176.54 (15) Å^3
Z = 4
D_x = 1.39 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11369 reflections
θ = 4.1–32.1°
μ = 0.27 mm^{-1}
T = 300 K
Block, colorless
0.31 × 0.23 × 0.15 mm

Data collection

Nonius KappaCCD diffractometer	$R_{int} = 0.039$
φ and ω scans	$\theta_{max} = 32.1^\circ$
Absorption correction: none	$h = -9 \rightarrow 9$
27839 measured reflections	$k = -26 \rightarrow 26$
4086 independent reflections	$l = -16 \rightarrow 16$
2758 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(I) + 0.001024I^2]$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$(\Delta/\sigma)_{max} = 0.001$
$wR(F^2) = 0.119$	$\Delta\rho_{max} = 0.33$ e Å^{-3}
$S = 1.65$	$\Delta\rho_{min} = -0.27$ e Å^{-3}
4086 reflections	Extinction correction: B–C type 1 Lorentzian isotropic (Becker & Coppens, 1974)
164 parameters	Extinction coefficient: 0.41 (19)
H atoms treated by a mixture of independent and constrained refinement	

The positions of the CH_3 group H atoms were refined. Other H atoms were fixed at calculated positions. A riding isotropic displacement parameter was used for all H atoms.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1995); program(s) used to refine structure: *JANA*2000 (Petricek & Dusek, 2000); molecular graphics: *DIAMOND* (Brandenburg & Berndt, 1999); software used to prepare material for publication: *JANA*2000.

The authors gratefully acknowledge financial support by the French Ministry of Education and the CNRS.

References

Becker, P. J. & Coppens, P. (1974). *Acta Cryst.* A**30**, 129–153.
Brandenburg, K. & Berndt, M. (1999). *DIAMOND*. Crystal Impact, Bonn, Germany.
Danel, K., Nielsen, C. & Pedersen, E. B. (1997). *Acta Chem. Scand.* **51**, 426–430.

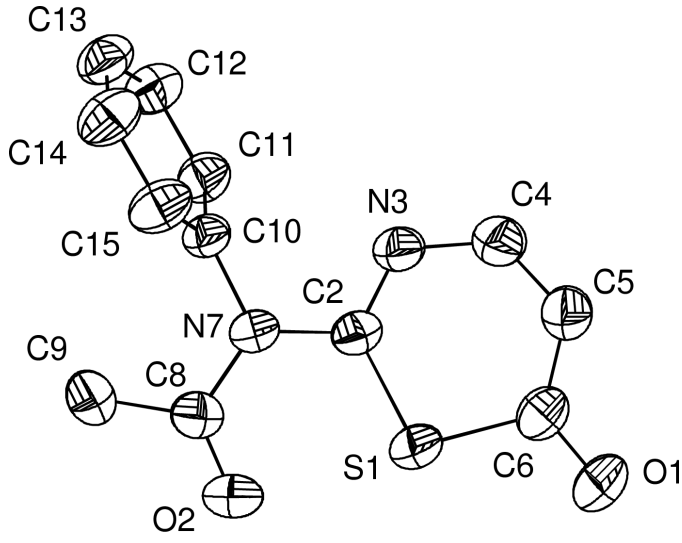


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids. H have been omitted for clarity.

Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Landreau, C., Deniaud, D., Reliquet, F., Reliquet, A. & Meslin, J. C. (2000). *Heterocycles*, **53**, 2667–2677.
Mayer, T. U., Kapoor, T. M., Haggarty, S. J., King, R. W., Schreiber, S. L. & Mitchison, T. J. (1999). *Science*, **286**, 971–1466.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter and R. M. Sweet, pp. 307–326. London: Academic Press.
Petricek, V. & Dusek, M. (2000). *JANA*2000. Institute of Physics, Praha, Czech Republic.
Sheldrick, G. M. (1995). *SHELXTL*. Version 5.0. Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Vig, R., Mao, C., Verkatachalam, T. K., Tuel-Ahlgren, L., Sudbeck, E. A. & Vokun F. M. (1998). *Bioorg. Med. Chem. Lett.* **8**, 1461–1466.
Wishka, D. G., Graber, D. R. & Morris, J. (1998). *J. Med. Chem.* **41**, 1357–1360.