Crystal structure of 4-butyl-6-nitro-2*H*-1,4-benzothiazin-3-one, $C_{12}H_{14}N_2O_3S$

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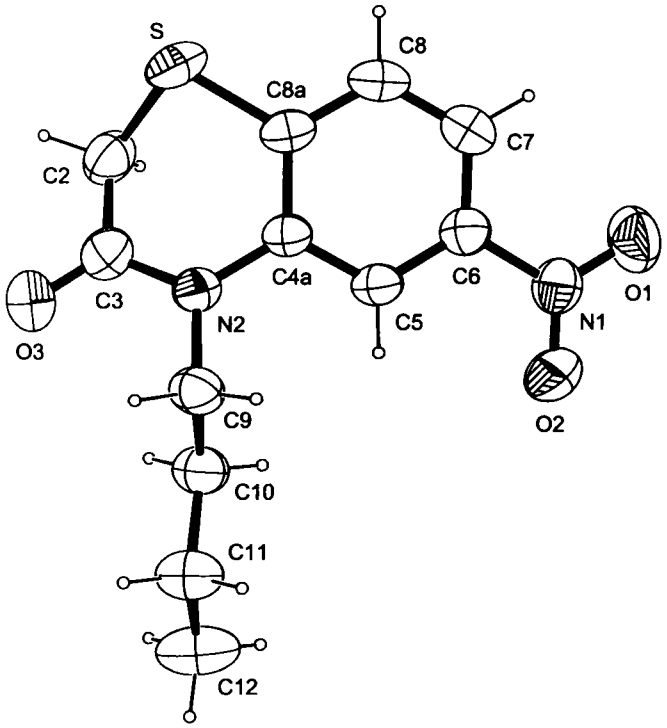

Abstract

$C_{12}H_{14}N_2O_3S$, monoclinic, $P12_1/n1$ (no. 14), a = 8.542(2) Å, b = 11.643(1) Å, c = 13.658(2) Å, β = 107.95(1)°, V = 1292.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.035, $wR_{ref}(F^2)$ = 0.094, T = 293 K.

Source of material

Crystals for X-ray diffraction studies were grown by slow evaporation from a EtOH/H_2O 95% solution.

Discussion

A large number of 2*H*-benzo(1,4)thiazine derivatives exhibit different pharmacological activities: antimicrobial, anticancerous, immunostimulating, aldose reductase inhibitor [1-5]. The alkylation of 4-N position of 2*H*-benzo(1,4)thiazines affords bactericidal and antifungal derivatives [6-7]. The title compound belongs to a class of these compounds and as knowledge of its stereochemistry may assist in the understanding of its pharmacological behavior, a crystal structure determination was undertaken.

The bond lengths and angles are in good agreement, to within experimental accuracy, with the values found in the literature [8]. The aromatic ring is slightly distorted with a twisted boat conformation (the largest deviation of 0.026(2) Å from the plane is exhibited by C4a atom) and a puckering amplitude of Q_T = 0.0411(2) Å. The conformation of the heterocyclic ring is of the pure chair and the puckering parameters [9] are: q_2 = 0.700(2) Å, q_3 = −1.824(2) Å, Q_T = 1.954(2) Å, τ = 159.0(6)° and φ = 187.9(2)°. In the packing, molecules interact through two intermolecular C–H···O hydrogen bonds: d(C2···O3^i) = 3.263(3) Å, d(H2B···O3^i) = 2.469 Å, ∠C2–H2B···O3^i = 139°, d(C9···O1^{ii}) = 3.463(2) Å, d(H9B···O1^{ii}) = 2.556(2) Å, ∠C9–H9B···O1^{ii} = 156°, forming layers parallel to the (103) plane (symmetry operations: (i) −x+1,−y+1,−z+1, (ii) x+½,−y+3/2,z−½).

Table 1. Data collection and handling.

Crystal:	colorless, irregular, size 0.2 × 0.25 × 0.3 mm
Wavelength:	Cu K_α radiation (1.54184 Å)
μ:	22.65 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, non-profiled $\omega/2\theta$
$2\theta_{max}$:	119.88°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2006, 1864
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1673
$N(param)_{refined}$:	163
Programs:	SHELXS-97 [10], SHELXL-97 [11], ORTEP-3 [12], WinGX [13]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(8)	4*e*	−0.0246	0.3905	0.6857	0.062
H(7)	4*e*	−0.2071	0.5138	0.7248	0.061
H(5)	4*e*	0.0221	0.7812	0.6426	0.051
H(2A)	4*e*	0.4482	0.5312	0.7029	0.072
H(2B)	4*e*	0.5020	0.4725	0.6149	0.072
H(9A)	4*e*	0.0655	0.7902	0.4882	0.061
H(9B)	4*e*	0.2132	0.7701	0.4452	0.061
H(10A)	4*e*	0.2296	0.8987	0.6231	0.066
H(10B)	4*e*	0.3858	0.8709	0.5901	0.066
H(12A)	4*e*	0.2874	1.1611	0.5036	0.132
H(12B)	4*e*	0.4284	1.0884	0.5787	0.132
H(12C)	4*e*	0.2665	1.1133	0.6062	0.132
H(11A)	4*e*	0.2789	0.9766	0.4392	0.079
H(11B)	4*e*	0.1179	1.0008	0.4678	0.079

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
S	4*e*	0.24076(6)	0.41368(4)	0.60657(4)	0.0640(4)	0.0360(3)	0.0725(4)	0.0087(2)	0.0134(3)	–0.0072(2)
N(2)	4*e*	0.2245(2)	0.6717(1)	0.5676(1)	0.0550(9)	0.0366(8)	0.0500(9)	0.0010(7)	0.0223(7)	0.0000(7)
C(8)	4*e*	–0.0163(2)	0.4691(2)	0.6760(1)	0.060(1)	0.033(1)	0.054(1)	–0.0068(9)	0.0086(9)	0.0039(8)
N(1)	4*e*	–0.2121(2)	0.7385(2)	0.7196(1)	0.061(1)	0.058(1)	0.0503(9)	0.0022(8)	0.0238(8)	–0.0057(8)
C(6)	4*e*	–0.1049(2)	0.6578(2)	0.6887(1)	0.051(1)	0.043(1)	0.0387(9)	0.0001(8)	0.0136(8)	–0.0018(7)
C(4A)	4*e*	0.1169(2)	0.6283(1)	0.6196(1)	0.046(1)	0.0339(9)	0.0390(9)	–0.0015(7)	0.0108(7)	–0.0012(7)
C(7)	4*e*	–0.1238(2)	0.5418(2)	0.7007(1)	0.053(1)	0.049(1)	0.049(1)	–0.0088(9)	0.0151(9)	0.0033(9)
C(5)	4*e*	0.0128(2)	0.7022(2)	0.6492(1)	0.053(1)	0.0320(9)	0.0416(9)	0.0000(8)	0.0140(8)	–0.0001(7)
O(2)	4*e*	–0.1915(2)	0.8408(1)	0.7105(1)	0.103(1)	0.050(1)	0.108(1)	0.0117(8)	0.061(1)	–0.0047(8)
C(8A)	4*e*	0.1057(2)	0.5097(2)	0.6366(1)	0.050(1)	0.0330(9)	0.042(1)	–0.0002(8)	0.0052(8)	–0.0019(7)
O(3)	4*e*	0.4521(2)	0.6554(1)	0.5178(1)	0.083(1)	0.072(1)	0.103(1)	0.0029(8)	0.061(1)	0.0014(9)
O(1)	4*e*	–0.3165(2)	0.6997(2)	0.7550(1)	0.079(1)	0.085(1)	0.080(1)	–0.0074(9)	0.0485(9)	–0.0114(9)
C(3)	4*e*	0.3666(3)	0.6172(2)	0.5669(2)	0.063(1)	0.050(1)	0.066(1)	–0.000(1)	0.027(1)	–0.008(1)
C(2)	4*e*	0.4116(2)	0.5106(2)	0.6305(2)	0.052(1)	0.055(1)	0.072(1)	0.010(1)	0.019(1)	–0.003(1)
C(9)	4*e*	0.1836(3)	0.7787(2)	0.5079(1)	0.067(1)	0.045(1)	0.045(1)	–0.0017(9)	0.0217(9)	0.0025(8)
C(10)	4*e*	0.2682(3)	0.8846(2)	0.5644(2)	0.066(1)	0.045(1)	0.053(1)	–0.0025(9)	0.020(1)	0.0013(9)
C(12)	4*e*	0.3114(4)	1.0979(2)	0.5511(2)	0.116(2)	0.047(1)	0.085(2)	–0.004(1)	0.007(2)	0.007(1)
C(11)	4*e*	0.2358(3)	0.9899(2)	0.4962(2)	0.082(2)	0.047(1)	0.066(1)	–0.002(1)	0.017(1)	0.006(1)

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