Crystal structure of 6-butyl-2-chlorothiazolo[5,4-*a*]acridin-11(6*H*)-one, $C_{18}H_{15}ClN_2OS$

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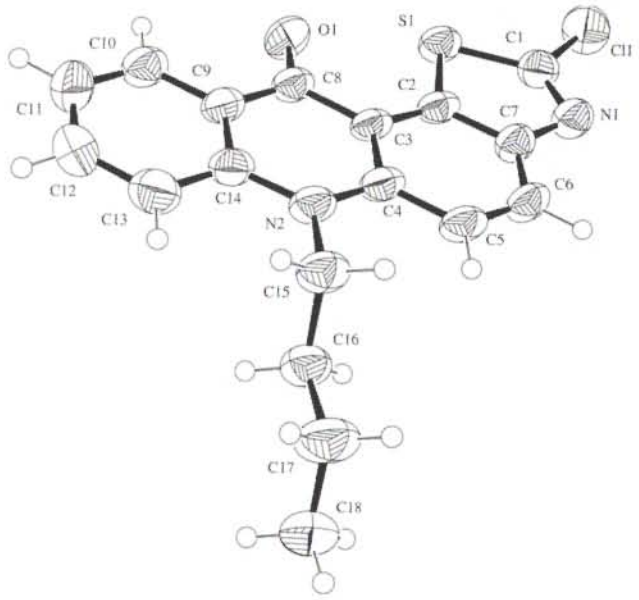

Abstract

$C_{18}H_{15}ClN_2OS$, orthorhombic, *Pbcn* (No. 60), $a = 8.360(1)$ Å, $b = 15.794(1)$ Å, $c = 23.949(1)$ Å, $V = 3162.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.044$, $wR_{ref}(F) = 0.047$, $T = 298$ K.

Source of material

This compound was prepared by a modification of the procedure used for the alkylation of 9-acridones [1]. The final 6-butyl-2-chlorothiazolo[5,4-*a*]acridin-11(6*H*)-one was obtained by alkylation of corresponding thiazolo[5,4-*a*]acridin-11(6*H*)-one [2] in dimethylformamide using sodium hydride and butyl bromide. The precipitate was purified by column chromatography and recrystallised in acetonitrile to yield the *N*-alkylated tetracycle in 55 % yield.

Discussion

9-Acridinones derivatives show a large spectrum of biological effects. As example, acronycine has a moderate anti-malarial activity against chloroquine-susceptible and chloroquine-resistant clones of *Plasmodium falciparum*. This molecule which act as tyrosine kinase inhibitor, suppress the proliferation of DHER cells stimulated by epidermal growth factor or calf serum [3]. Recently, the crystal structure of 9(10*H*)-acridone has been determinated [4], while three novel dimeric acridone alkaloids named citbismine-A, -B and -C have been isolated from Citrus plants and their structure have been elucidated by single crystal X-ray analysis and spectroscopic studies [5]. Moreover the first description of the structure of crown-ethers derivatives of 9-acridone has been obtained in our laboratory [6]. We now report the X-ray analysis of a new tetracyclic *N*-butyl derivative of acridone fused with a five-membered thiazolic ring. The examination of the bond distances and angles shows no surprising values despite of the presence of the sulfur atom. All distances are normal, due to the close presence of the atom O(1), the sulfur atom is repelled leading to a S1—C2—C3 angle of 127.7 (3)°. The whole molecule is remarkably flat. The carbon atoms C16, C17 and C18 are the only atoms out of the general medium plane. They are respectively at 1.38 Å, 1.366 Å, and 2.744 Å, out of this plane O1-N2-C3-...-C15 and form a plane perpendicular to this principal plane. From an intermolecular contact point of view, one single weak hydrogen bond between the atom C(6) and the atom O(1) from a neighboring molecule (symmetry $1.5-x, -0.5+y, z$) has been observed. No other contact has been detected with the exception of intramolecular contacts predictable for this type of compound.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.1 x 0.1 x 0.2 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	3.79 cm^{-1}
Diffractometer, scan mode:	Kappa CCD, 90 frames, $\Delta\varphi = 2°$
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3139, 2791
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2385
$N(param)_{refined}$:	209
Programs:	MAXUS [7], ORTEP [8]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(5)	8*d*	0.3332	0.2507	0.2975	0.0590
H(6)	8*d*	0.1809	0.2368	0.2171	0.0590
H(10)	8*d*	0.4464	0.6720	0.3399	0.0645
H(11)	8*d*	0.6022	0.6836	0.4199	0.0755
H(12)	8*d*	0.6724	0.5604	0.4693	0.0773
H(13)	8*d*	0.5989	0.4272	0.4371	0.0689
H(15A)	8*d*	0.5983	0.3242	0.3920	0.0610
H(15B)	8*d*	0.5132	0.2679	0.3470	0.0614
H(16A)	8*d*	0.2764	0.2742	0.4016	0.0661
H(16B)	8*d*	0.3729	0.3221	0.4480	0.0661
H(17A)	8*d*	0.4350	0.1562	0.4131	0.0888
H(17B)	8*d*	0.5345	0.2041	0.4587	0.0888
H(18A)	8*d*	0.3548	0.1125	0.5020	0.0883
H(18B)	8*d*	0.2136	0.1586	0.4715	0.0883
H(18C)	8*d*	0.3131	0.2065	0.5171	0.0883

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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}
Cl(1)	8*d*	–0.0763(3)	0.4679(1)	0.0825(1)	0.082(1)	0.0682(9)	0.0683(8)	0.0057(8)	–0.0104(7)	0.0050(7)
S(1)	8*d*	0.1136(3)	0.5217(2)	0.1812(1)	0.0602(7)	0.0349(6)	0.0557(7)	0.0029(6)	0.0080(6)	0.0078(6)
O(1)	8*d*	0.2784(8)	0.6012(4)	0.2650(3)	0.083(3)	0.029(1)	0.070(2)	0.001(2)	–0.000(2)	0.007(1)
N(1)	8*d*	0.0644(8)	0.3636(5)	0.1533(3)	0.062(3)	0.039(2)	0.059(2)	–0.001(2)	0.007(2)	0.000(2)
N(2)	8*d*	0.4405(8)	0.3889(5)	0.3454(4)	0.062(3)	0.034(2)	0.051(2)	–0.001(2)	0.006(2)	0.010(2)
C(1)	8*d*	0.0369(1)	0.4412(5)	0.1399(4)	0.054(3)	0.049(3)	0.052(3)	0.000(2)	0.007(2)	0.002(2)
C(2)	8*d*	0.1964(1)	0.4435(6)	0.2225(4)	0.044(3)	0.030(2)	0.052(3)	0.002(2)	0.012(2)	0.008(2)
C(3)	8*d*	0.2910(2)	0.4538(8)	0.2704(5)	0.048(3)	0.027(2)	0.049(3)	–0.000(2)	0.013(2)	0.006(2)
C(4)	8*d*	0.3455(1)	0.3808(6)	0.2982(4)	0.051(3)	0.032(2)	0.053(3)	–0.001(2)	0.015(2)	0.006(2)
C(5)	8*d*	0.2992(1)	0.3002(5)	0.2774(4)	0.062(3)	0.030(2)	0.057(3)	0.003(2)	0.007(3)	0.013(2)
C(6)	8*d*	0.2086(1)	0.2922(5)	0.2305(4)	0.062(3)	0.027(2)	0.066(3)	–0.001(2)	0.009(3)	0.004(2)
C(7)	8*d*	0.1560(1)	0.3639(6)	0.2017(4)	0.052(3)	0.035(3)	0.054(3)	–0.000(2)	0.009(2)	–0.001(2)
C(8)	8*d*	0.3306(2)	0.5379(8)	0.2898(5)	0.056(3)	0.032(2)	0.053(3)	–0.001(2)	0.015(2)	0.003(2)
C(9)	8*d*	0.4305(1)	0.5427(6)	0.3390(4)	0.051(3)	0.036(2)	0.052(3)	–0.004(2)	0.014(2)	0.002(2)
C(10)	8*d*	0.4780(1)	0.6219(6)	0.3597(4)	0.065(3)	0.041(3)	0.062(3)	–0.004(2)	0.017(3)	–0.003(2)
C(11)	8*d*	0.5687(1)	0.6290(6)	0.4067(4)	0.084(4)	0.057(3)	0.064(3)	–0.011(3)	0.008(3)	–0.018(3)
C(12)	8*d*	0.6116(1)	0.5559(6)	0.4355(4)	0.085(4)	0.074(4)	0.048(3)	–0.008(3)	0.003(3)	–0.009(3)
C(13)	8*d*	0.5688(1)	0.4768(5)	0.4164(4)	0.072(3)	0.060(3)	0.053(3)	–0.001(3)	0.006(3)	0.008(3)
C(14)	8*d*	0.4803(1)	0.4682(6)	0.3667(4)	0.058(3)	0.042(3)	0.050(3)	–0.003(2)	0.017(2)	0.002(2)
C(15)	8*d*	0.4981(1)	0.3119(7)	0.3742(5)	0.065(3)	0.040(2)	0.060(3)	0.005(2)	0.005(3)	0.013(2)
C(16)	8*d*	0.3798(1)	0.2812(6)	0.4184(4)	0.076(3)	0.044(3)	0.057(3)	0.001(3)	0.007(3)	0.010(2)
C(17)	8*d*	0.4302(1)	0.1974(8)	0.4426(5)	0.107(5)	0.054(3)	0.082(4)	0.005(3)	0.016(4)	0.025(3)
C(18)	8*d*	0.3181(1)	0.1657(7)	0.4875(5)	0.108(5)	0.066(3)	0.064(3)	–0.013(3)	–0.001(3)	0.020(3)

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