Crystal structure of (2*E*)-7-chloro-2-(7-chloro-3,4-dihydro-3-oxo-2*H*-1,4-benzothiazin-2-ylidene)-2*H*-1,4-benzothiazine-3(4*H*)-one, C₁₆H₈Cl₂N₂O₂S₂, at 93 K

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Abstract

C₁₆H₈Cl₂N₂O₂S₂, triclinic, *P* $\bar{1}$ (No. 2), *a* = 4.5934(6) Å, *b* = 7.8303(8) Å, *c* = 10.403(1) Å, α = 92.040(9)°, β = 99.38(1)°, γ = 94.19(1)°, *V* = 367.8 Å³, *Z* = 1, *R*_{gt}(*F*) = 0.031, *wR*_{ref}(*F*²) = 0.068, *T* = 93 K.

Source of material

The title compound was obtained from Clariant GmbH and purified by sublimation under argon at about 683 K, using a two-zone furnace [1]. The single crystals were then recrystallized from solution in dimethylsulfoxide.

Discussion

Thiazine-indigos are novel hydrogen-bonded pigments developed by Clariant [2]. These compounds cover a variety of shades from yellow to red, depending on the substituents. The molecular skeleton is similar to that of thioindigos and includes two pairs of NH and C=O groups as found in quinacridones and diketopyrrolopyrroles [3] for the formation of NH...O intermolecular hydrogen bonds. The title compound is a chlorinated derivative registered as Pigment Red 279.

The molecule is entirely planar. On the molecular plane, there are chains of intermolecular hydrogen bonds along the *b* axis between the N–H group of one molecule and the O atom of the neighboring one to form a sheet-like H-bond network. The pres-

ent two-dimensional network is typical of diketopyrrolopyrrole pigments [4] and also of 2,9-dimethylquinacridone [5]. The NH...O distance is 2.832(2) Å and the NH/O angle is 170°. Judging from these bond parameters, the present H-bond is apparently weaker than that of quinacridones and pyrrolopyrroles, as also shown by its considerably higher solubility in organic solvents. Another characteristic point of the H-bond is that there exist steps of about 0.57 Å between the H-bonded molecules as found in 2,9-dimethylquinacridone [5] as well as in modifications I and II of dithioketopyrrolopyrroles [6]. The molecules are stacked with small overlap along the *a* axis.

Table 1. Data collection and handling.

Crystal:	red platelet, size 0.02 × 0.04 × 0.10 mm
Wavelength:	Cu <i>K</i> _α radiation (1.5419 Å)
μ :	67.47 cm ⁻¹
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, 72 frames, $\Delta\omega$ = 10°
2 θ _{max} :	136.28°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3439, 121
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ (<i>I</i> _{obs}), 910
<i>N</i> (<i>param</i>) _{refined} :	109
Programs:	SHELXS-86 [7], teXsan [8], ORTEP-III [9], ABSCOR [10]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	2i	0.1725	1.4719	0.9285	0.018
H(2)	2i	0.4556	1.5746	0.7803	0.022
H(3)	2i	0.7305	1.5282	0.6132	0.023
H(4)	2i	0.6094	1.0129	0.6501	0.018

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
Cl(1)	2i	0.8818(2)	1.23045(9)	0.48851(7)	0.0234(5)	0.0201(4)	0.0199(4)	−0.0003(3)	0.0101(3)	0.0003(3)
S(1)	2i	0.2566(2)	0.97014(9)	0.83912(7)	0.0206(5)	0.0115(3)	0.0179(4)	0.0015(3)	0.0079(3)	0.0013(3)
O(1)	2i	−0.0865(5)	1.2971(2)	1.0604(2)	0.028(1)	0.0131(9)	0.022(1)	0.0025(8)	0.0117(9)	0.0003(8)
N(1)	2i	0.1931(6)	1.3532(3)	0.9090(2)	0.022(2)	0.012(1)	0.021(1)	0.0019(9)	0.008(1)	0.0014(9)
C(1)	2i	0.0554(7)	1.2408(3)	0.9789(3)	0.021(2)	0.014(1)	0.016(1)	0.002(1)	0.004(1)	0.000(1)
C(2)	2i	0.0704(7)	1.0534(4)	0.9618(3)	0.015(2)	0.016(1)	0.016(1)	0.003(1)	0.004(1)	0.000(1)
C(3)	2i	0.4059(7)	1.1554(3)	0.7734(3)	0.018(2)	0.014(1)	0.017(1)	0.001(1)	0.003(1)	0.003(1)
C(4)	2i	0.3605(7)	1.3213(4)	0.8119(3)	0.016(2)	0.018(1)	0.016(1)	0.001(1)	0.005(1)	0.002(1)
C(5)	2i	0.4837(7)	1.4601(3)	0.7523(3)	0.024(2)	0.012(1)	0.021(2)	0.000(1)	0.004(1)	0.002(1)
C(6)	2i	0.6465(7)	1.4333(4)	0.6540(3)	0.021(2)	0.018(1)	0.022(2)	−0.001(1)	0.005(1)	0.003(1)
C(7)	2i	0.6891(7)	1.2668(4)	0.6166(3)	0.017(2)	0.022(1)	0.017(1)	−0.001(1)	0.003(1)	0.002(1)
C(8)	2i	0.5735(7)	1.1278(4)	0.6760(3)	0.016(2)	0.015(1)	0.019(1)	0.002(1)	0.004(1)	0.000(1)

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