Crystal structure of 5,12-dihydro-5-methylquino[2,3-*b*]acridine-7,14-dione, $C_{21}H_{14}N_2O_2$, *at 93 K*

J. Mizuguchi* and T. Senju

Yokohama National University, Graduate School of Engineering, Department of Applied Physics, 79-5 Tokiwadai, Hodogaya-ku, 240-8501 Yokohama, Japan

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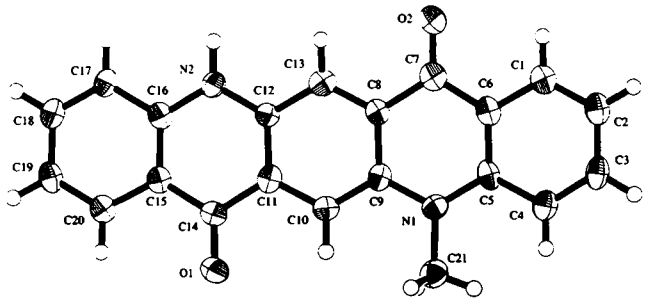

Abstract

$C_{21}H_{14}N_2O_2$, orthorhombic, *Pbca* (No. 61), $a = 13.517(2)$ Å, $b = 7.340(2)$ Å, $c = 29.033(2)$ Å, $V = 2880.5$ Å^3, $Z = 8$, $R_{gt}(F) = 0.072$, $wR_{ref}(F^2) = 0.162$, $T = 93$ K.

Source of material

The title compound was synthesized according to the method described in the literature [1]. The product was purified by sublimation, using a two zone furnace [2]. The single crystals were then grown from the vapor phase in a closed system at about 583 K for 20 h.

Discussion

Quinacridones are well known organic pigments of red color on the market [3]. The title compound is a mono-*N*-methyl quinacridone and is used as a colorant as well as a material for electroluminescence applications [1]. We have been conducting a series of investigations on the correlation between crystal and electronic structures in quinacridone compounds [4-6], using unsubstituted quinacridone with two NH groups, monomethyl derivative with one NH group and dimethyl derivative with no NH group. Special attention has been paid to the effect of intermolecular hydrogen bonds on the color as well as on the stability of the compounds. As part of the above investigation, the present structure analysis has been undertaken.

The molecule is entirely planar and has a molecular symmetry of C_1. A small dipole moment of about 0.44 D appears as a result of the C_1 symmetry. There are two kinds of stacking columns along the *b*-axis, crossing each other with an inclination angle of about 45°. In each column, the molecules of the one form shown in the figure and its inverted form are stacked pairwise alternately so as to cancel the dipole moment to stabilize themselves electrostatically. There are chains of intermolecular hydrogen bonds between the NH group of one molecule in the one column and the O atom of another molecule in the neighboring column. That is, the intermolecular H-bond bridges the two different stacking columns in such a way that one molecule is hydrogen-bonded to two neighboring molecules. The present molecular arrangement is basically quite similar to the one observed for the γ phase of unsubstituted quinacridone [5,7] and also indigos [8,9].

Table 1. Data collection and handling.

Crystal:	red platelet, size 0.03 × 0.08 × 0.30 mm
Wavelength:	Cu K_α radiation (1.5419 Å)
μ:	7.93 cm^{-1}
Diffractometer, scan mode:	Rigaku RAXIS-RAPID, 60 frames, $\Delta\omega = 15°$
$2\theta_{max}$:	136.42°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	26398, 2497
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1245
$N(param)_{refined}$:	226
Programs:	SHELXS-97 [10], teXsan [11], ABSCOR [12], ORTEPII [13]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(1)	8*c*	0.9507	0.3754	0.4887	0.042
H(2)	8*c*	0.8719	0.4850	0.5536	0.043
H(3)	8*c*	0.6971	0.4904	0.5558	0.043
H(4)	8*c*	0.6056	0.3873	0.4941	0.044
H(5)	8*c*	0.5933	0.1334	0.3382	0.036
H(6)	8*c*	0.5848	–0.1338	0.1803	0.038
H(7)	8*c*	0.9304	–0.1241	0.1799	0.037
H(8)	8*c*	0.8445	–0.2356	0.1170	0.040
H(9)	8*c*	0.6708	–0.2456	0.1174	0.042
H(10)	8*c*	0.9312	–0.0010	0.2561	0.037
H(11)	8*c*	0.9379	0.1239	0.3312	0.035
H(12)	8*c*	0.5378	0.3089	0.4436	0.049
H(13)	8*c*	0.5376	0.3347	0.3905	0.049
H(14)	8*c*	0.5369	0.1401	0.4114	0.049

* Correspondence author (e-mail: mizu-j@ynu.ac.jp)

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}
O(1)	8*c*	0.5588(2)	0.0095(4)	0.26014(9)	0.033(1)	0.046(2)	0.043(2)	–0.002(2)	–0.005(1)	–0.005(2)
O(2)	8*c*	0.9736(2)	0.2458(4)	0.4106(1)	0.041(2)	0.056(2)	0.044(2)	–0.006(2)	–0.004(2)	–0.011(2)
N(1)	8*c*	0.6694(2)	0.2586(4)	0.4149(1)	0.031(2)	0.029(2)	0.034(2)	–0.001(2)	0.004(2)	0.002(2)
N(2)	8*c*	0.8609(2)	–0.0007(4)	0.2568(1)	0.034(2)	0.027(2)	0.030(2)	0.002(2)	0.003(2)	–0.002(2)
C(1)	8*c*	0.8805(3)	0.3786(5)	0.4899(1)	0.043(2)	0.024(2)	0.037(3)	–0.004(2)	–0.003(2)	0.002(2)
C(2)	8*c*	0.8345(3)	0.4431(5)	0.5284(1)	0.053(3)	0.022(3)	0.036(3)	0.000(2)	–0.004(2)	–0.003(2)
C(3)	8*c*	0.7303(3)	0.4453(5)	0.5295(1)	0.058(3)	0.025(3)	0.029(2)	0.004(2)	0.002(2)	–0.002(2)
C(4)	8*c*	0.6759(3)	0.3848(5)	0.4929(1)	0.047(2)	0.025(3)	0.037(3)	0.003(2)	0.002(2)	–0.004(2)
C(5)	8*c*	0.7234(3)	0.3192(5)	0.4526(1)	0.046(3)	0.013(2)	0.029(2)	–0.006(2)	–0.006(2)	–0.001(2)
C(6)	8*c*	0.8274(3)	0.3162(5)	0.4513(1)	0.043(2)	0.018(2)	0.031(3)	0.001(2)	–0.005(2)	0.003(2)
C(7)	8*c*	0.8815(3)	0.2506(6)	0.4118(2)	0.041(2)	0.025(2)	0.036(3)	0.001(2)	–0.004(2)	0.005(2)
C(8)	8*c*	0.8222(3)	0.1880(5)	0.3728(1)	0.033(2)	0.018(2)	0.030(2)	–0.004(2)	–0.002(2)	0.003(2)
C(9)	8*c*	0.7162(3)	0.1934(5)	0.3753(1)	0.039(2)	0.017(2)	0.030(2)	–0.003(2)	0.004(2)	0.001(2)
C(10)	8*c*	0.6634(3)	0.1320(5)	0.3375(1)	0.030(2)	0.025(2)	0.036(2)	0.000(2)	0.001(2)	0.001(2)
C(11)	8*c*	0.7097(3)	0.0673(5)	0.2978(1)	0.041(2)	0.016(2)	0.028(2)	0.002(2)	0.003(2)	0.004(2)
C(12)	8*c*	0.8138(3)	0.0631(5)	0.2952(1)	0.028(2)	0.018(2)	0.032(2)	0.000(2)	–0.001(2)	–0.003(2)
C(13)	8*c*	0.8677(3)	0.1252(5)	0.3333(1)	0.035(2)	0.019(2)	0.039(3)	–0.007(2)	0.004(2)	0.004(2)
C(14)	8*c*	0.6511(3)	0.0038(5)	0.2589(1)	0.030(2)	0.025(2)	0.033(2)	0.000(2)	–0.002(2)	0.005(2)
C(15)	8*c*	0.7045(3)	–0.0653(5)	0.2196(1)	0.037(2)	0.018(2)	0.031(2)	0.003(2)	–0.002(2)	0.001(2)
C(16)	8*c*	0.8091(3)	–0.0634(5)	0.2194(1)	0.037(2)	0.017(2)	0.032(2)	0.001(2)	–0.004(2)	–0.001(2)
C(17)	8*c*	0.8605(3)	–0.1270(5)	0.1805(1)	0.040(2)	0.020(2)	0.034(2)	0.002(2)	0.004(2)	–0.003(2)
C(18)	8*c*	0.8094(3)	–0.1930(5)	0.1434(1)	0.045(2)	0.022(2)	0.032(3)	0.001(2)	0.005(2)	0.001(2)
C(19)	8*c*	0.7058(3)	–0.1983(5)	0.1432(1)	0.049(3)	0.018(2)	0.035(3)	–0.004(2)	–0.005(2)	–0.003(2)
C(20)	8*c*	0.6552(3)	–0.1331(5)	0.1808(1)	0.039(2)	0.017(2)	0.038(3)	0.002(2)	–0.003(2)	0.000(2)
C(21)	8*c*	0.5608(3)	0.2593(6)	0.4151(1)	0.041(2)	0.042(3)	0.041(3)	–0.005(2)	0.006(2)	0.000(2)

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