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Reinvestigation of 5,12-dihydro-5,12-dimethylquino[2,3-*b*]acridine-7,4-dione

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Key indicators

Single-crystal X-ray study
T = 223 K
Mean σ(C–C) = 0.002 Å
R factor = 0.044
wR factor = 0.126
Data-to-parameter ratio = 14.8

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

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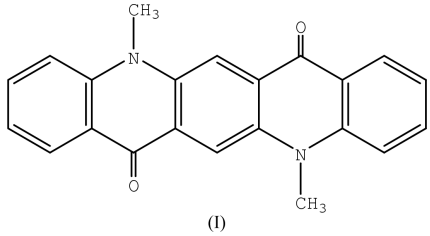
The structure of the title compound, $C_{22}H_{16}N_2O_2$ commonly called *trans*-*N*,*N*′-dimethylquinacridone, has been reinvestigated at 223 K [the original determination was by Zavodnik, Chetkina & Val'kova (1981). *Zh. Struckt. Khim*. **22**, 188–190; and also by Ohmasa & Susse (1976). *Naturwissenschaften*, **63**, 387–388]. The structure, in space group $P2_1/c$, is basically in good agreement with those reported earlier, but is determined with higher precision. The entirely planar centrosymmetric molecules are arranged with major overlap of the acridine skeleton along the stacking axis.

Comment

The crystal structure of the title compound, (I), was reported not only by Zavodnik *et al.* (1981) but also by Ohmasa & Susse (1976). The present paper provides some additional results at 223 K with higher precision. We have been conducting a series of investigations on the correlation between the color in the solid state and crystal structures of quinacridone pigments. Special attention has been focused on the effect of N—H···O intermolecular hydrogen bonds on the color as well as on the stability of the compounds. For this reason, we have examined three representative quinacridone compounds with various hydrogen bonds, namely unsubstituted quinacridone with two NH groups (four hydrogen bonds per molecule; Mizuguchi *et al.*, 2002), the monomethyl derivative with one NH group (two hydrogen bonds per molecule; Zambounis & Mizuguchi, 1996; Mizuguchi & Senju, 2002) and the dimethyl derivative with no NH group (*i.e.* the title compound; no hydrogen bonds per molecule). Compound (I) has also recently attracted attention as a doping material for electroluminescence applications (Shi & Tang, 1997). In this context, the present reinvestigation has been carried out.



(I)

Our results are basically in good agreement with those of Zavodnik *et al.* (1981). The molecule is entirely planar, as shown in Fig. 1, and belongs to the point group C_i. The molecules are stacked with major overlap of the acridine skeleton along the *a* axis. Our study has $R = 0.044$ for 1349 reflections with $I > 2\sigma$, compared with $R = 0.047$ for only 532 reflections

with $I > 3\sigma$ in Zavodnik's study, and also with $R = 0.15$ for 726 non-zero reflections in Ohmasa's study. Since the measurement temperatures were different (at 223 K, in our case, but room temperature in Zavodnik's study), direct comparison of the structural parameters seems rather difficult. However, the s.u. values of the bond parameters have been improved significantly in the present investigation: the values in (I) are 0.002 Å for the bond lengths C—C, C—N and C—O, whereas the values were in the range 0.005–0.007 Å in Zavodnik's study. As for the bond angles C—C—C, C—C—N, C—N—C and C—C—O, our s.u. values are 0.1°, while a range of 0.4–0.6° was reported for Zavodnik's study.

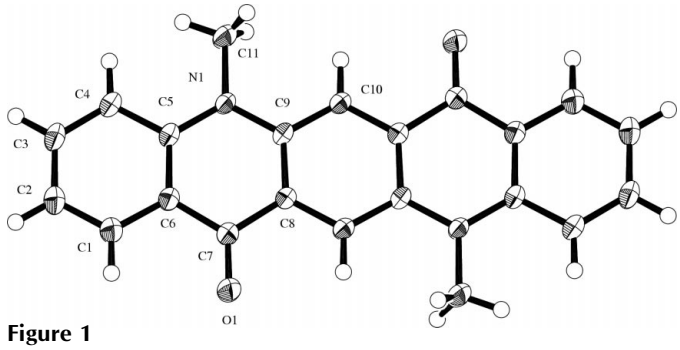


Figure 1
A view of the molecular structure of (I), showing 50% displacement ellipsoids for the non-H atoms.

Experimental

The title compound, (I), was prepared according to a method described in the literature (Zambounis & Mizuguchi, 1996). The sample was purified by sublimation at about 623 K, using a two-zone furnace (Mizuguchi, 1981). Single crystals of (I) were then grown from a solution in dimethylformamide. The present material is quite fragile, so that cutting of the crystal using a safety razor impairs the quality of the crystal. For this reason, we used a single crystal as grown.

Crystal data

$C_{22}H_{16}N_2O_2$
$M_r = 340.38$
Monoclinic, $P2_1/c$
$a = 4.928$ (3) Å
$b = 11.103$ (3) Å
$c = 14.462$ (2) Å
$\beta = 98.39$ (2)°
$V = 782.8$ (4) Å^3
$Z = 2$
$D_x = 1.444$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 14.7$–$14.9°$
$\mu = 0.09$ mm^{-1}
$T = 223.2$ K
Platelet, red
1.18 × 0.17 × 0.07 mm

Data collection

Rigaku AFC-7*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.947$, $T_{max} = 0.998$
2107 measured reflections
1797 independent reflections
1349 reflections with $F^2 > 2\sigma(F^2)$
$R_{int} = 0.013$
$\theta_{max} = 27.5°$
$h = -6 \rightarrow 0$
$k = 0 \rightarrow 14$
$l = -18 \rightarrow 18$
3 standard reflections
every 150 reflections
intensity decay: 1.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.126$
$S = 1.59$
1747 reflections
118 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + \{0.05[\max(F_o^2,0) + 2F_c^2]/3\}^2]$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å).

O1—C7	1.234 (2)	C4—C5	1.414 (2)
N1—C5	1.380 (2)	C5—C6	1.413 (2)
N1—C9	1.389 (1)	C6—C7	1.459 (2)
N1—C11	1.461 (2)	C7—C8	1.468 (2)
C1—C2	1.376 (2)	C8—C9	1.416 (2)
C1—C6	1.405 (2)	C8—C10^{i}	1.393 (2)
C2—C3	1.398 (2)	C9—C10	1.392 (2)
C3—C4	1.376 (2)		

Symmetry code: (i) $-x, 1-y, -z$.

All H atoms were positioned by geometrically but were not refined. Methyl H atoms were positioned on the basis of difference Fourier maps.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *TEXSAN* (Molecular Structure Corporation, 2001); program(s) used to solve structure: *SIR*88 (Burla *et al.*, 1989); program(s) used to refine structure: *TEXSAN*; molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *TEXSAN*.

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