

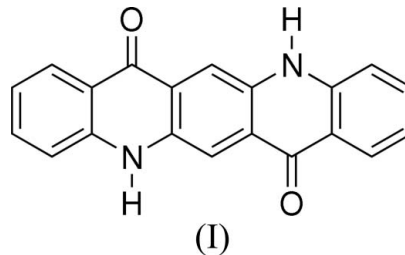
5,7,12,14-Tetrahydro[2,3-*b*]quinolinoacridine (β form)Naoko Nishimura, Takatoshi
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Key indicators

Single-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.009$ Å
 R factor = 0.160
 wR factor = 0.431
Data-to-parameter ratio = 10.8For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The title compound, $\text{C}_{20}\text{H}_{12}\text{N}_2\text{O}_2$, is a centrosymmetric pigment molecule. The molecules are doubly linked *via* $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds, forming ribbons along $[110]$ and $[\bar{1}\bar{1}0]$.Received 5 September 2006
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Comment

The title compound, commonly known as quinacridone (QA), (I), is an industrially important red pigment (Herbst & Hunger, 1997). As early as 1958, three crystal modifications of QA (α , β and γ forms) were known to exist as powders, of which only the β and γ forms are of commercial value. The structure analyses of QA derivatives based on single crystals were reported recently for dithio-QA (Mizuguchi & Rihs, 1992) and for γ -QA (Potts *et al.*, 1994; Mizuguchi *et al.*, 2002). The γ form is typically characterized by a three-dimensional $\text{N}-\text{H}\cdots\text{O}$ hydrogen-bond network, where one molecule is hydrogen-bonded to four neighbouring molecules. As for the β form, Paulus *et al.* (1989) published the lattice parameters, but details of the molecular arrangement remained unknown.

We report here the structure of the β form of QA based on single crystals grown from the vapour phase. The key factor for the successful growth of the β form was found in the course of our investigation of the QA latent pigment (a precursor of QA), from which QA can be thermally regenerated around 473 K (Imura *et al.*, 2005). We noticed that the regenerated crystals were always of the β form, whereas the γ form was obtained at a higher temperature of around 623 K (Mizuguchi, Sasaki *et al.*, 2002). Therefore, we firmly believed that the β form could be grown at a condensation temperature of about 473–523 K. In fact, this condition led us to the successful growth of the β form from a purified sample of QA. The lattice parameters of the present investigation are in fairly good agreement with those of Paulus *et al.* (1989).

An *ORTEP*III (Burnett & Johnson, 1996) plot of (I) is shown in Fig. 1. The molecule has C_i symmetry, but it is not entirely planar. The dihedral angles between the planes *A* (C1–C5/C9) and *B* (C5/N1/C6–C9), *A* and *C* [C6/C7/C10ⁱ/C6ⁱ/C7ⁱ/C10; symmetry code (i) $-x, 2 - y, -z$], and *B* and *C* are 2.1 (2), 1.8 (2), and 0.5 (2)°, respectively.

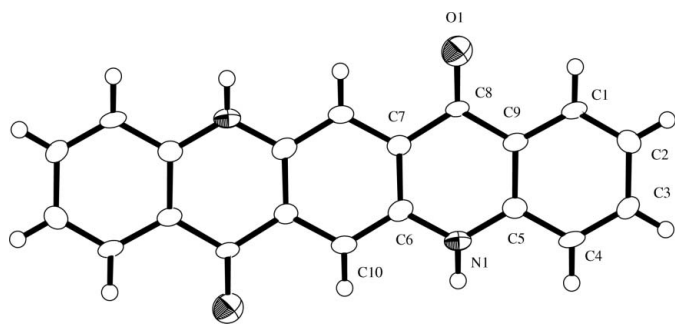


Figure 1
A view of the molecular structure of (I), showing 50% probability displacement ellipsoids. Unlabelled atoms are related to labelled atoms by the symmetry operation $(-x, 2 - y, -z)$.

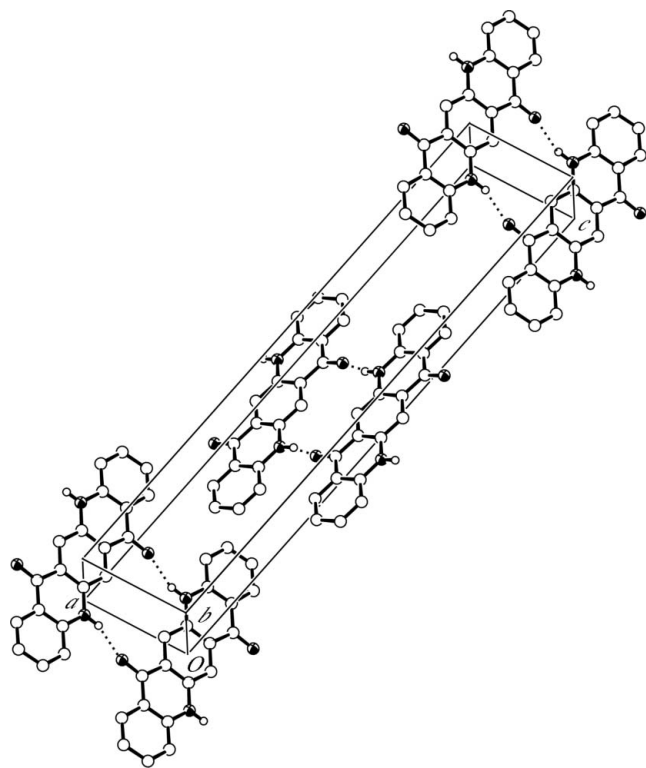


Figure 2
The packing arrangement in (I). H atoms have been omitted unless these are involved in hydrogen bonds (dashed lines).

Fig. 2 shows the packing arrangement of (I) in the crystal structure. There are intermolecular N—H...O hydrogen bonds (Table 1) between the QA molecules, forming ribbons along $[110]$ and $[1\bar{1}0]$. One QA molecule is bonded to two neighbouring molecules through four N—H...O hydrogen bonds with a small step of *ca* 0.53 Å between the hydrogen-bonded molecular planes. The QA molecules are stacked along the *b* axis.

Experimental

QA was obtained from CIBA Specialty Chemicals and purified three times by sublimation under vacuum using a two-zone furnace (Mizuguchi, 1981). Single crystals were then grown from the vapour

phase in a closed system at sublimation and condensation temperatures of 623 and 473 K, respectively. A number of tiny platelet crystals were obtained over a period of 72 h.

Crystal data

$C_{20}H_{12}N_2O_2$
 $M_r = 312.32$
Monoclinic, $P2_1/c$
 $a = 5.7366$ (6) Å
 $b = 3.8851$ (4) Å
 $c = 29.880$ (4) Å
 $\beta = 95.861$ (8)°
 $V = 662.46$ (13) Å³

$Z = 2$
 $D_x = 1.566$ Mg m⁻³
Cu $K\alpha$ radiation
 $\mu = 0.83$ mm⁻¹
 $T = 296.1$ K
Platelet, red
0.42 × 0.20 × 0.04 mm

Data collection

Rigaku R-Axis RAPID
diffractometer
 ω scans
Absorption correction: multi-scan
(*ABSCOR*; Higashi, 1995)
 $T_{\min} = 0.705$, $T_{\max} = 0.965$

5102 measured reflections
1187 independent reflections
977 reflections with $F^2 > 2\sigma(F^2)$
 $R_{\text{int}} = 0.120$
 $\theta_{\max} = 68.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.160$
 $wR(F^2) = 0.431$
 $S = 1.06$
1187 reflections
110 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.1985P)^2 + 9.1P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 2.40$ e Å⁻³
 $\Delta\rho_{\min} = -0.48$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
N1—H1N...O1 ⁱⁱ	0.86	2.02	2.848 (8)	161

Symmetry code: (ii) $x + 1, y + 1, z$.

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.93 and N—H = 0.86 Å, and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$. The highest residual density peak is located 1.51 Å from atom O1. The U_{eq} of O1 is larger than those of the other non-H atoms. This might be due to the extremely small size of the crystal and also its poor crystallinity.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSK, 2005); program(s) used to solve structure: *SIR2002* (Burla *et al.*, 2003); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP III* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

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