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# The triclinic form of 2,9-dichloro-5,12-dihydroquino[2,3-*b*]acridine-7,14-dithione dimethylacetamide disolvate

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The title compound, $C_{20}H_{10}Cl_2N_2S_2\cdot 2C_4H_9NO$, is a dimethylacetamide (DMA) disolvate of DTQ-Cl, which is a thionated derivative of a 2,9-dichloroquinacridone pigment. The compound shows polymorphism and this paper reports the triclinic form (space group $P\overline{1}$, $Z = 1$). The DTQ-Cl molecule is centrosymmetric and planar, while the DMA molecule is orientationally disordered. Two symmetry-related DMA molecules are hydrogen bonded *via* their O atoms to the NH groups of DTQ-Cl. The molecular plane of DMA is twisted with respect to the DTQ-Cl skeleton by 73.6 (1)°.

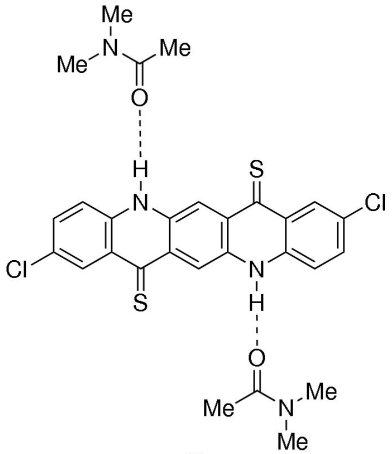
**Key indicators**

Single-crystal X-ray study
$T$ = 93 K
Mean $\sigma$(C–C) = 0.005 Å
Disorder in solvent or counterion
$R$ factor = 0.061
$wR$ factor = 0.216
Data-to-parameter ratio = 12.7

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

## Comment

The title compound (DTQ-Cl·2DMA), (I), is a dimethylacetamide (DMA) disolvate of DTQ-Cl, which is a thionated derivative of 2,9-dichloroquinacridone known as an industrially important red pigment (Herbst & Hunger, 1997). The background of the present study has been set out in our previous paper (Senju *et al.*, 2005). We obtained three kinds of solvated crystals of DTQ-Cl. One was isolated from a dimethylformamide solution (Senju *et al.*, 2005), and the other two polymorphic forms were obtained from one single solution in DMA. The structure of the monoclinic form, (I*a*), has been reported in the preceding paper (Hoki *et al.*, 2005). The present paper deals with the structure of the triclinic form, (I*b*).



(I)

Fig. 1 shows an *ORTEPIII* (Burnett & Johnson, 1996) plot of (I*b*). The DTQ-Cl molecule is centrosymmetric and planar, as characterized by the mean deviation of 0.019 Å from the least-squares plane (C1–C10/N1). The DMA molecule is orientationally disordered. The molecular plane of DMA is twisted with respect to the DTQ-Cl skeleton by 73.6 (1)°.

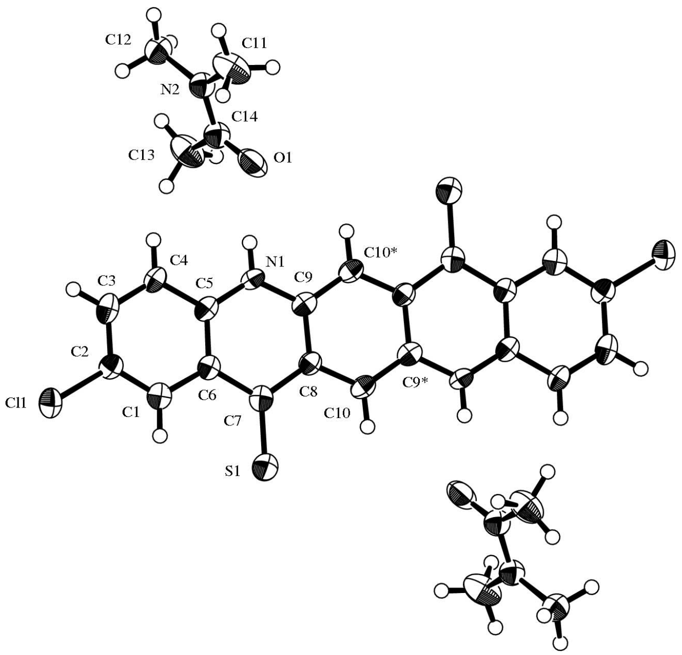


**Figure 1**
A view of the molecular structure of (I*b*), showing 50% probability displacement ellipsoids for non-H atoms. Atoms labelled with an asterisk (*) are at the symmetry position $(1 - x, 2 - y, -z)$. Unlabelled atoms are related to labelled atoms by this same symmetry operator. Only the major disorder component is shown.

There are N—H···O intermolecular hydrogen bonds (Table 2) between the NH group of DTQ-Cl and the O atom of DMA. DTQ-Cl molecules are stacked parallel to each other along the *b* axis, forming one column (Fig. 2). Similarly, DMA molecules form another column sandwiched by two columns of DTQ-Cl.

## Experimental

DTQ-Cl was synthesized by thionation of commercially available 2,9-dichloroquinacridone using Lawesson's reagent (Rochat *et al.*, 1988). Crystals of (I) were grown by gradual cooling of a DMA solution prepared at about 420 K. Single crystals of both monoclinic and triclinic forms were obtained at the same time from one single solution. Both crystal forms appeared dark green. However, the crystal shapes were different, being needle for (I*a*) and platelet for (I*b*).

*Crystal data*

$C_{20}H_{10}Cl_2N_2S_2\cdot 2C_4H_9NO$
$M_r = 587.58$
Triclinic, $P\overline{1}$
$a = 7.156$ (2) Å
$b = 8.941$ (3) Å
$c = 11.487$ (4) Å
$\alpha = 104.32$ (2)°
$\beta = 96.91$ (2)°
$\gamma = 107.369$ (18)°
$V = 664.5$ (4) Å$^3$
$Z = 1$
$D_x = 1.468$ Mg m$^{-3}$
Cu $K\alpha$ radiation
Cell parameters from 5253 reflections
$\theta = 4.1$–68.0°
$\mu = 3.95$ mm$^{-1}$
$T = 93.1$ K
Platelet, dark green
0.40 × 0.40 × 0.08 mm

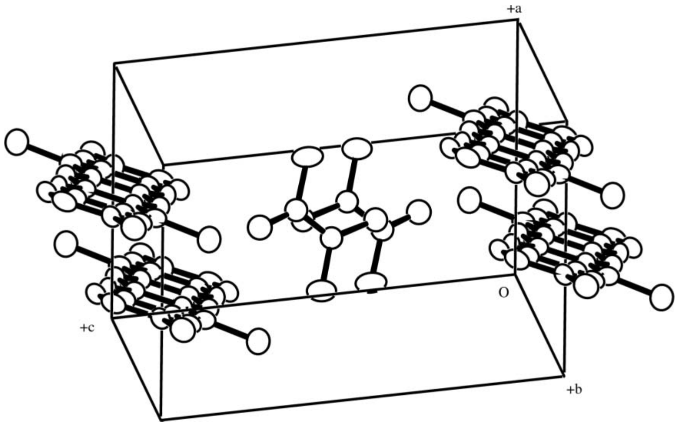


**Figure 2**
The molecular arrangement of DMA-solvated DTQ-Cl in (I*b*). H atoms have been omitted.

*Data collection*

Rigaku R-AXIS RAPID-F imaging-plate diffractometer
$\omega$ scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.281$, $T_{\max} = 0.729$
5955 measured reflections
2164 independent reflections
1551 reflections with $F^2 > 2\sigma(F^2)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 68.1°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -11 \rightarrow 10$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.061$
$wR(F^2) = 0.217$
$S = 1.18$
2164 reflections
171 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1319P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.46$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.53$ e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Cl1—C2 | 1.752 (4) | C3—C4 | 1.376 (5) |
| S1—C7 | 1.630 (4) | C4—C5 | 1.417 (5) |
| O1—C15 | 1.191 (11) | C5—C6 | 1.402 (6) |
| O1—C14 | 1.236 (8) | C6—C7 | 1.459 (5) |
| N1—C5 | 1.352 (5) | C7—C8 | 1.475 (5) |
| N1—C9 | 1.370 (4) | C8—C10 | 1.389 (5) |
| C1—C2 | 1.358 (5) | C8—C9 | 1.417 (6) |
| C1—C6 | 1.418 (5) | C9—C10$^{\rm i}$ | 1.392 (5) |
| C2—C3 | 1.390 (6) | | |
| C5—N1—C9 | 122.2 (3) | C1—C6—C7 | 120.3 (4) |
| C2—C1—C6 | 119.8 (4) | C6—C7—C8 | 114.8 (4) |
| C1—C2—C3 | 122.5 (4) | C6—C7—S1 | 123.1 (3) |
| C1—C2—Cl1 | 118.9 (3) | C8—C7—S1 | 122.1 (3) |
| C3—C2—Cl1 | 118.5 (3) | C10—C8—C9 | 118.4 (4) |
| C4—C3—C2 | 119.0 (4) | C10—C8—C7 | 121.1 (4) |
| C3—C4—C5 | 120.2 (4) | C9—C8—C7 | 120.5 (3) |
| N1—C5—C6 | 121.1 (3) | N1—C9—C10$^{\rm i}$ | 119.6 (4) |
| N1—C5—C4 | 118.9 (4) | N1—C9—C8 | 120.2 (4) |
| C6—C5—C4 | 120.0 (4) | C10$^{\rm i}$—C9—C8 | 120.2 (3) |
| C5—C6—C1 | 118.5 (4) | C8—C10—C9$^{\rm i}$ | 121.4 (4) |
| C5—C6—C7 | 121.2 (3) | | |

Symmetry code: (i) $-x + 1, -y + 2, -z$.

*Acta Cryst.* (2005). E**61**, o1930–o1932 Senju *et al.* • $C_{20}H_{10}Cl_2N_2S_2\cdot 2C_4H_9NO$ **o1931**

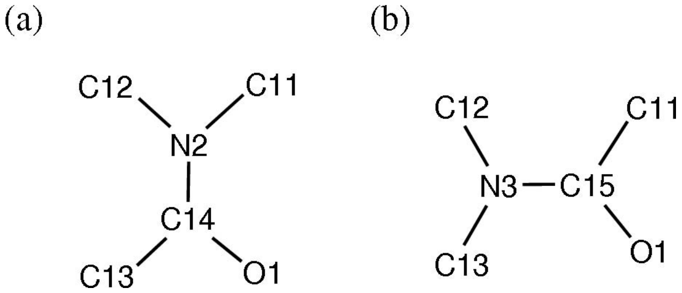


**Figure 3**
A schematic representation of the orientationally disordered DMA in (I*b*). (*a*) The major component, N2/C14 (62%). (*b*) The minor component, N3/C15 (38%).

**Table 2**
Hydrogen-bond geometry (Å, °).

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N1—H1N···O1 | 0.88 | 1.90 | 2.774 (4) | 173 |

The planar DMA molecule has two possible orientations (Fig. 3), and the positions of not only atom O1 but also of the three methyl atoms, C11, C12 and C13, are strongly overlapped. The clearly split atoms, N2/C14 and N3/C15, were refined isotropically. The site-occupancy factor was determined by equalizing, as much as possible, the $U_{\rm iso}$ parameters for the two sites, the occupancy of the major site (N2/C14) being 62%. Methyl H atoms were placed in geometrically idealized positions considering the orientational disorder of the DMA molecule, and constrained to ride on their parent C atoms, with C—H = 0.98 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C). All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H = 0.95 Å and N—H = 0.88 Å, and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom).

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996); software used to prepare material for publication: *CrystalStructure*.

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