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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.008$ Å
 R factor = 0.070
 wR factor = 0.208
Data-to-parameter ratio = 13.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Succinimidyl 7-(diethylamino)-2-oxo-2H-
chromene-3-carboxylate chloroform solvate

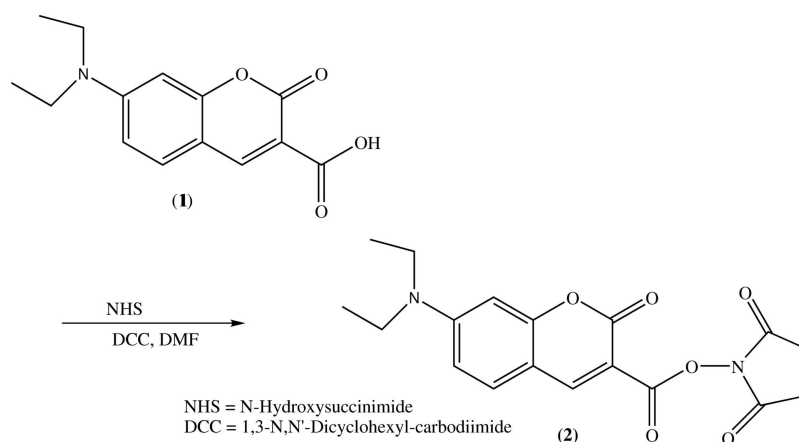
In the title molecular structure, $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}_6 \cdot \text{CHCl}_3$, the dihedral angle between the two fused, essentially planar, six-membered rings is $5.4(2)^\circ$. In the crystal structure, weak intermolecular $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds and $\pi-\pi$ stacking interactions connect molecules into two-dimensional layers.

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Comment

For background information and relevant references see the previous paper (Bardajee *et al.*, 2006). A view of the molecular structure of the title compound, (2), is shown in Fig. 1, and selected bond lengths and angles are given in Table 1. Unlike in the molecular structure of 7-diethylaminocoumarin-3-carboxylic acid, (1) (Bardajee *et al.*, 2006), the two fused six-membered rings in (2) are not coplanar but instead have an angle of $5.4(2)^\circ$ between their least-squares planes. The dihedral angles between the six-membered rings $\text{C}4-\text{C}9$ and $\text{C}1-\text{C}4/\text{C}9/\text{O}1$ and the five-membered ring $\text{N}2/\text{C}15-\text{C}18$ are $73.9(2)$ and $77.1(2)^\circ$, respectively. In the crystal structure, weak $\text{C}-\text{H} \cdots \text{O}$ hydrogen bonds (Table 2) and significant $\pi-\pi$ stacking interactions [where $\text{Cg}1 \cdots \text{Cg}2(\frac{3}{2} - x, \frac{3}{2} - y, 1 - z) = 3.695(3)$ Å ($\text{Cg}1$ and $\text{Cg}2$ are the centroids defined by ring atoms $\text{O}1/\text{C}1-\text{C}4/\text{C}9$ and $\text{C}4-\text{C}9$ respectively) and the perpendicular distance is 3.43 Å] connect molecules into layers, and chloroform solvent molecules are located between these layers (Fig. 2).



Experimental

The title compound was obtained from the reaction of 7-diethylaminocoumarin-3-carboxylic acid, (1), with *N*-hydroxysuccinimide, whose preparation was described previously (Berthelot *et al.*, 2005). In a typical procedure, to a solution of (1) (289.31 mg, 1 mmol) in dimethylformamide (5 ml), *N*-hydroxysuccinimide (115 mg, 1 mmol) was added. After dissolution, the reaction was cooled to 263 K. After

stirring for 1 h at this temperature, 1,3-*N,N'*-dicyclohexylcarbodiimide (226.96 mg, 1.1 mmol) was added. The solution was stirred at 263 K for 12 h. The solution was filtered to remove dicyclohexylurea. A 2-propanol–hexane mixture (1:20) was added to the filtrate to give product (2) as a yellow solid (315 mg, 88%). X-ray quality crystals were obtained by slow recrystallization of (2) from chloroform at 277 K.

Crystal data

$C_{18}H_{18}N_2O_6 \cdot CHCl_3$
 $M_r = 477.71$
 Monoclinic, $C2/c$
 $a = 19.601 (3) \text{ \AA}$
 $b = 9.3170 (14) \text{ \AA}$
 $c = 22.807 (3) \text{ \AA}$
 $\beta = 93.429 (9)^\circ$
 $V = 4157.6 (10) \text{ \AA}^3$

$Z = 8$
 $D_x = 1.526 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 0.48 \text{ mm}^{-1}$
 $T = 150 (1) \text{ K}$
 Needle, yellow
 $0.30 \times 0.16 \times 0.08 \text{ mm}$

Data collection

Bruker–Nonius KappaCCD diffractometer
 φ scans and ω scans with κ offsets
 Absorption correction: multi-scan (SORTAV; Blessing, 1995)
 $T_{\min} = 0.746, T_{\max} = 0.964$

11417 measured reflections
 3598 independent reflections
 2151 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.126$
 $\theta_{\text{max}} = 25.2^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.070$
 $wR(F^2) = 0.209$
 $S = 1.05$
 3598 reflections
 272 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.094P)^2 + 7.3572P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.30 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.39 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters ($\text{\AA}, ^\circ$).

O1–C9	1.380 (6)	O4–N2	1.390 (6)
O1–C1	1.393 (7)	O4–C14	1.393 (7)
O2–C1	1.201 (6)	N2–C18	1.383 (8)
O3–C14	1.212 (7)	N2–C15	1.400 (8)
C7–N1–C12	121.4 (5)	C18–N2–C15	115.2 (5)
C7–N1–C10	122.3 (5)	O4–N2–C15	121.4 (5)
C12–N1–C10	116.2 (5)	O2–C1–C2	128.3 (6)
C18–N2–O4	122.2 (5)		

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C15–H15A \cdots O6 ⁱ	1.00	2.28	3.240 (7)	160
C3–H3A \cdots O3 ⁱⁱ	0.95	2.59	3.219 (7)	124
C5–H5A \cdots O6 ⁱⁱⁱ	0.95	2.50	3.395 (7)	157
C11–H11C \cdots O6 ⁱⁱⁱ	0.98	2.53	3.473 (9)	162
C12–H12B \cdots O3 ^{iv}	0.99	2.54	3.358 (8)	140
C16–H16B \cdots O4 ^v	0.99	2.42	3.195 (8)	135

Symmetry codes: (i) $x - \frac{1}{2}, y + \frac{1}{2}, z$; (ii) $-x + \frac{3}{2}, -y + \frac{1}{2}, -z + 1$; (iii) $-x + \frac{3}{2}, -y + \frac{3}{2}, -z + 1$; (iv) $x, y + 1, z$; (v) $-x + \frac{3}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

Analysis of the data using PLATON (Spek, 2003) revealed that the crystal was a non-merohedral twin and a twin rotation matrix $(-100, 010, 0.139 \ 0 \ 1)$ was applied. In subsequent refinements the

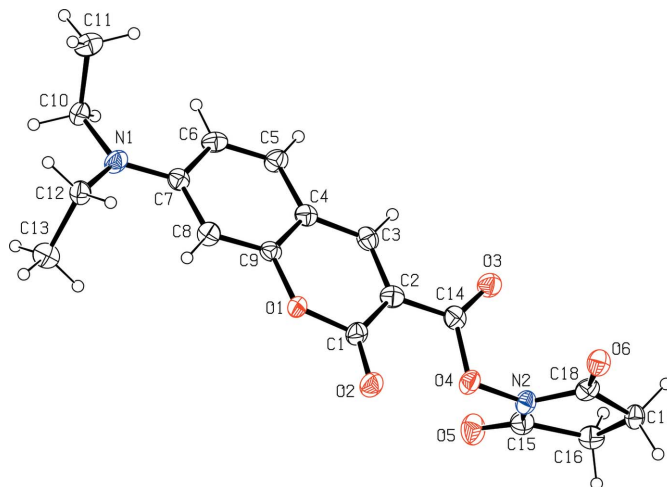


Figure 1

Molecular structure of (2), showing 30% probability displacement ellipsoids (arbitrary spheres for H atoms). The solvent molecule has been omitted.

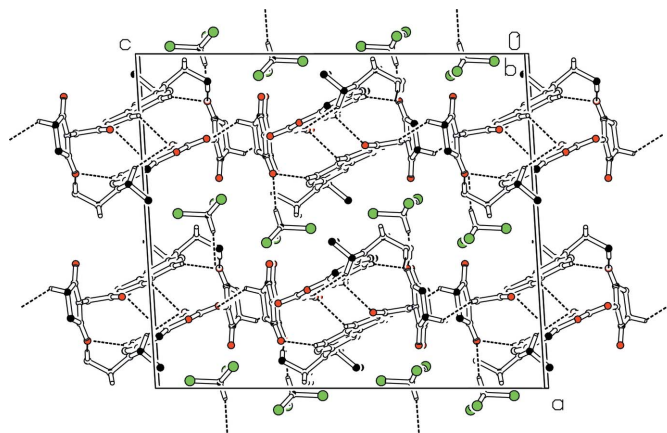


Figure 2

A partial packing plot (Spek, 2003) of (2), showing hydrogen bonds as dashed lines.

value of the weighted R -factor (for all data) improved from 0.303 to 0.209. The twin fraction refined to 0.884:0.116 (2). The relatively high R_{int} value is related to the twinning. H atoms bonded to C atoms were placed in calculated positions with $C-H = 0.95 \text{ \AA}$ (aromatic), 0.99 \AA (methylene), 0.98 \AA (methyl) or 1.00 \AA (chloroform) and were included in the refinement in a riding-model approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms.

Data collection: COLLECT (Nonius, 2002); cell refinement: DENZO-SMN (Otwinowski & Minor, 1997); data reduction: DENZO-SMN; program(s) used to solve structure: SIR92 (Altomare *et al.*, 1994); program(s) used to refine structure: SHELXTL (Sheldrick, 2001); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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