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2,3,4-Trichloro-6-(diethylamino)-9-oxo-9*H*-xanthene-1-carboxylic acid methanol solvate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.093
Data-to-parameter ratio = 17.7

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

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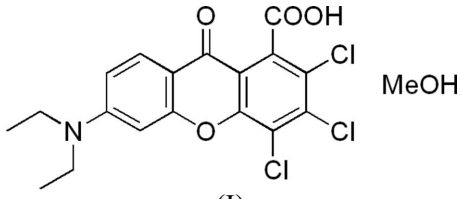
The crystal structure of the title compound, $C_{18}H_{14}Cl_3NO_4\cdot CH_3OH$, is characterized by O—H···O hydrogen bonds between the xanthene molecule and the methanol solvent molecule.

Comment

In the present paper, we report the crystal structure of the title compound, (I) (Fig. 1). An extensive hydrogen-bond network is built up by O—H···O hydrogen-bonding interactions between the xanthene molecule and the methanol solvent molecule (Table 1 and Fig. 2).



(I)

Experimental

Compound (I) was prepared according to the method described by Lee *et al.* (2002). A solution of 3-diethylaminophenol (0.36 g, 22 mmol) and tetrachlorophthalic anhydride (0.48 g, 22 mmol) was refluxed in toluene (15 ml) for 8 h. The solution was cooled to room temperature and the precipitate collected to yield 2,3,4-trichloro-6-(diethylamino)-9-oxo-9*H*-xanthene-1-carboxylic acid (0.59 g, 70%). The crude product, (I), was purified by silica-gel flash chromotography (methanol–dichloromethane, 1:10 *v*/*v*). Crystals of (I) (m.p. 550–551 K) suitable for X-ray diffraction were obtained by slow

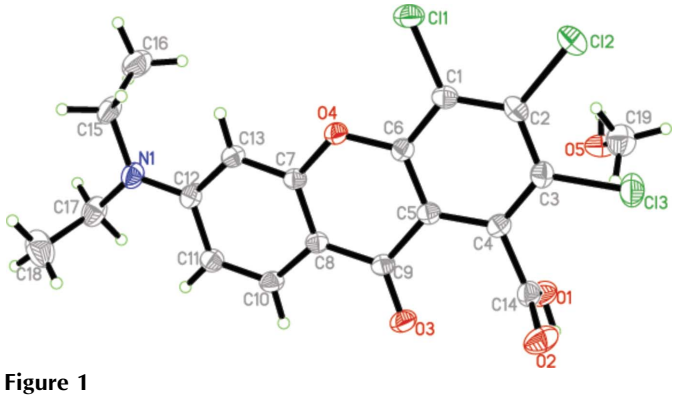


Figure 1
The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radius.

evaporation of a solution in ethyl acetate and acetone (1:3 *v*/*v*). ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 1.12 (*t*, 6H, *J* = 6.9 Hz), 3.35 (*dd*, 4H, *J* = 6.9 and 14.1 Hz), 6.40 (*d*, 1H, *J* = 2.1 Hz), 6.61 (*dd*, 1H, *J* = 2.4 and 9.3 Hz), 7.83 (*d*, 1H, *J* = 9.0 Hz).

Crystal data

$C_{18}H_{14}Cl_3NO_4\cdot CH_4O$
M_r = 446.69
Triclinic, $P\overline{1}$
a = 8.5125 (12) Å
b = 10.0649 (14) Å
c = 12.4028 (17) Å
α = 105.305 (5)°
β = 95.552 (9)°
γ = 101.563 (6)°
V = 991.5 (2) Å^3
Z = 2
D_x = 1.496 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 294 (2) K
Block, yellow
0.14 × 0.12 × 0.08 mm

Data collection

Rigaku Saturn diffractometer
ω scans
Absorption correction: multi-scan (Jacobson, 1998)
T_{min} = 0.934, T_{max} = 0.962
12536 measured reflections
4699 independent reflections
1969 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.9°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.093
S = 0.82
4699 reflections
265 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O5^{i}	0.84 (3)	1.78 (3)	2.589 (2)	163 (3)
O5—H2···O3^{ii}	0.80 (3)	2.03 (3)	2.807 (2)	163 (3)

Symmetry codes: (i) $-x+1, -y+1, -z+2$; (ii) $x+1, y, z$.

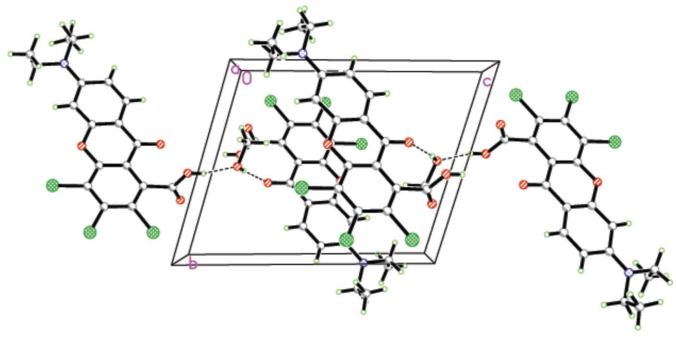


Figure 2
The crystal structure of (I), viewed along the *a* axis. Dashed lines indicate the hydrogen-bond interactions.

All O-bound H atoms were located in a difference map and then refined freely. The remaining H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å and U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXL97*; software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2005).

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