organic papers

3,3,6,6-Tetramethyl-9-(3,4-methylene-dioxylphenyl)-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione

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The crystal structure of the title compound, $C_{24}H_{27}NO_4$, reveals that there are two roughly identical molecules in the asymmetric unit. In both acridine moieties, the central pyridine rings are slightly distorted and adopt a boat conformation, whereas the two outer six-membered rings display half-chair conformations. Each molecule is linked through N—H$\cdots$O hydrogen bonds to symmetry-related molecules, forming infinite chains.

Comment

The use of water as a solvent with the catalyst TEBA (triethylbenzylammonium chloride) in organic chemistry was rediscovered in the 1980's (Rideout & Breslow, 1980). Compared with an organic solvent, it is cheap, not inflammable and not toxic. 1,4-Dihydropyridines are well known compounds, as a consequence of their pharmacological profile as calcium channel modulators (Janis *et al.*, 1987), which have become almost indispensable for the treatment of cardiovascular diseases such as hypertension, cardiac arrhythmia, or angina.

(I)
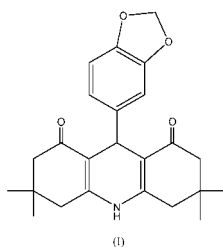
We report here the crystal structure of 3,3,6,6-tetramethyl-9-(3,4-methylenedioxylphenyl)-1,2,3,4,5,6,7,8,9,10-decahydro-acridine-1,8-dione, (I), The X-ray crystal structure determination indicates that there are two roughly identical molecules in the asymmetric unit. Only one of these molecules is shown in Fig. 1. In both molecules, the central pyridine ring of the acridine moity is slightly distorted and adopts a boat conformation. Atoms C1, C6, C8 and C13 are coplanar, with C7 and N1 deviating from the plane by 0.313 (7) and 0.107 (7) Å, respectively [0.267 (7) and 0.108 (7) Å for molecule 2]. A similar distortion was observed in the structures of 3,3,6,6-tetramethyl-9-(4-chlorophenyl)-10-(4-methylphenyl)-1,2,3,4,5,6,7,8,9,10-decahydroacridine-1,8-dione [0.192 (3) for C13 and 0.091 (3) Å for N; Wang *et al.*, 2003) and 7,7-di-methyl-2-(4-bromophenyl)-4-phenyl-5-oxo-1,4,5,6,7,8-hexa-hydroquinolin [0.294 (2) for C4 and 0.137 (2) Å for N; Shi *et al.*, 2002). The two outer six-membered rings of the acridine moiety adopt half-chair conformations; atoms C3 and C11
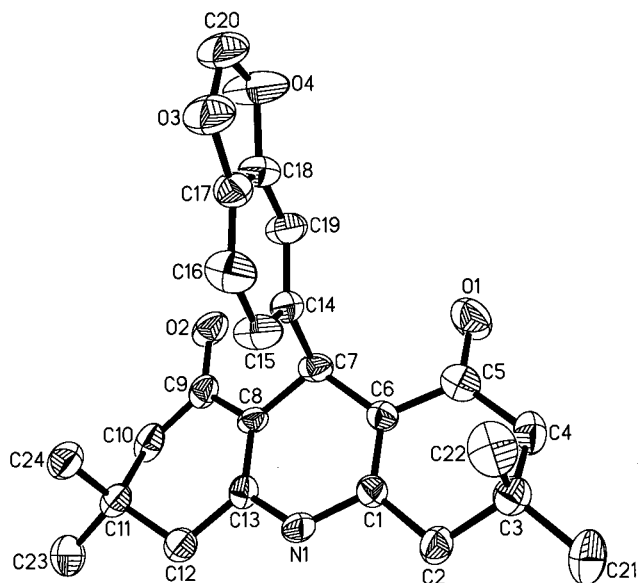
similar conformation has been found in the structure of 7,7-dimethyl-2-amino-3-cyano-4-(3,4-methylenedioxylphenyl)-5-oxo-5,6,7,8-tetrahydro-4H-benzo-[b]-pyran (Wang *et al.*, 2002). The fused dioxyphenyl rings are nearly perpendicular to the central pyridine rings, forming dihedral angles of 85.9 (1) and 85.7 (1)°, respectively.

The crystal packing, shown in Fig. 2, reveals that each molecule is linked through N—H···O hydrogen bonds to symmetry-related molecules, forming infinite chains.

Experimental

The title compound, (I), was prepared by the reaction of 3,4-methylenedioxylbenzaldehyde, 5,5-dimethyl-1,3-cyclohexanedione and ammonium acetate in water catalysed by TEBA; m.p. 599–601 K. Single crystals suitable for X-ray diffraction were obtained by slow evaporation ethanol solution.

Figure 1
View of one of the molecules of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. H atoms are omitted.

Crystal data

$C_{24}H_{27}NO_4$	Mo $K\alpha$ radiation
$M_r = 393.47$	Cell parameters from 27
Orthorhombic, $Pca2_1$	reflections
$a = 14.080$ (2) Å	$\theta = 3.1–12.0°$
$b = 15.158$ (3) Å	$\mu = 0.08$ mm^{-1}
$c = 20.233$ (4) Å	$T = 296$ (2) K
$V = 4318.1$ (14) Å^3	Block, yellow
$Z = 8$	$0.58 \times 0.36 \times 0.12$ mm
$D_x = 1.210$ Mg m^{-3}	

Data collection

Siemens $P4$ diffractometer	$\theta_{max} = 27.0°$
ω scans	$h = 0 \to 17$
Absorption correction: none	$k = 0 \to 19$
8775 measured reflections	$l = -21 \to 25$
4854 independent reflections	3 standard reflections
1942 reflections with $I > 2\sigma(I)$	every 97 reflections
$R_{int} = 0.031$	intensity decay: 7.4%

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0355P)^2]$
$wR(F^2) = 0.090$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.75$	$(\Delta/\sigma)_{max} < 0.001$
4854 reflections	$\Delta\rho_{max} = 0.18$ e Å^{-3}
531 parameters	$\Delta\rho_{min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

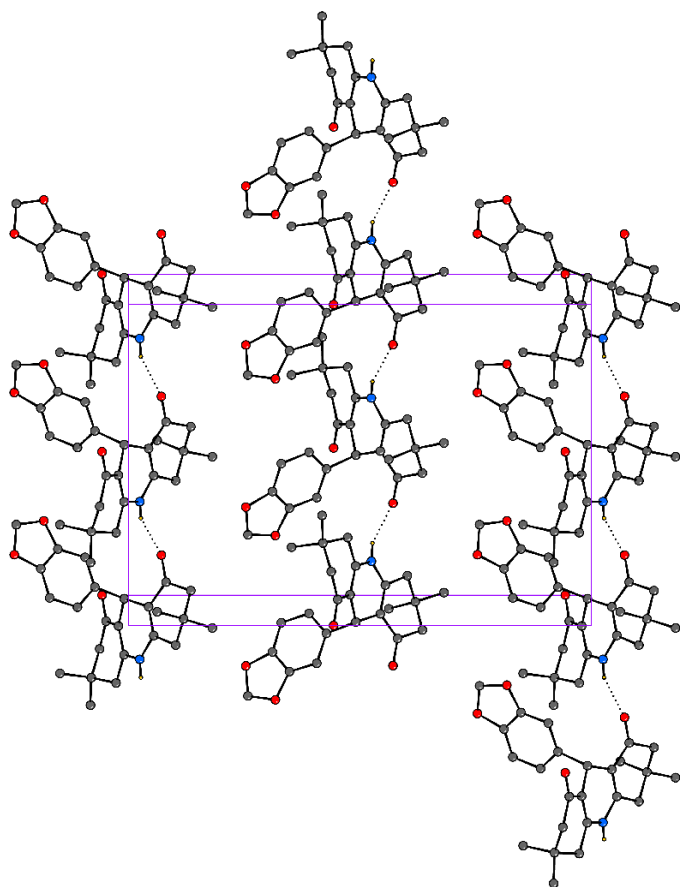|			
O1—C5	1.234 (5)	O5—C29	1.239 (5)
O2—C9	1.243 (5)	O6—C33	1.222 (5)
O3—C17	1.382 (7)	O7—C41	1.373 (7)
O3—C20	1.394 (7)	O7—C44	1.428 (6)
O4—C18	1.378 (5)	O8—C42	1.373 (5)
O4—C20	1.429 (7)	O8—C44	1.426 (7)
N1—C13	1.376 (5)	N2—C25	1.369 (5)
N1—C1	1.396 (5)	N2—C37	1.377 (5)
C1—C6	1.346 (5)	C25—C30	1.336 (5)
C6—C7	1.514 (5)	C30—C31	1.514 (6)
C7—C8	1.521 (6)	C31—C32	1.518 (5)
C8—C13	1.331 (5)	C32—C37	1.343 (5)
C13—N1—C1	121.7 (3)	C25—N2—C37	121.5 (3)
C6—C1—N1	119.5 (4)	C30—C25—N2	120.9 (4)
C1—C6—C7	121.4 (4)	C25—C30—C31	121.7 (4)
C6—C7—C8	109.5 (3)	C30—C31—C32	109.3 (3)
C13—C8—C7	121.4 (4)	C37—C32—C31	121.9 (4)
C8—C13—N1	120.6 (4)	C32—C37—N2	120.1 (4)

Figure 2
The molecular packing, showing the hydrogen-bonding network. H atoms not involved in hydrogen bonding are omitted.

deviate from the mean planes defined by C1, C2, C4, C5, C6 and C8, C9, C10, C12, C13 by 0.627 (7) and 0.628 (6) Å, respectively [0.636 (6) and 0.649 (7) Å for molecule 2]. A

Table 2
Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1$\cdots$O2^i	0.86	2.06	2.868 (4)	157
N2—H2$\cdots$O5^{ii}	0.86	2.08	2.847 (4)	148

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

The positions of all H atoms were fixed geometrically (C—H 0.86–0.97 Å), with U_{iso}(H) set to 1.2 or 1.5 times U_{eq} of the parent atom. As there are no atoms heavier than O, Friedel pairs were merged and the Flack (1983) parameter was not refined.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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