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4-(9-Acridin-9-ylamino)-2,2,6,6-tetramethylpiperidine *N*-oxide

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Key indicators

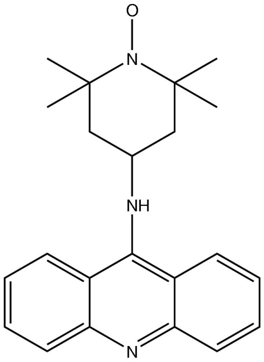
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.120
Data-to-parameter ratio = 9.3

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

The title compound, $C_{22}H_{26}N_3O$, crystallizes in space group $P2_1$ with two molecules in the asymmetric unit. Intermolecular N—H···O hydrogen bonds play an important role in the formation of polymeric chains running along the crystallographic *a* axis.

Comment

The title compound is a potential fluorescnet probe to detect the antioxidant capability (Aliga *et al.*, 2003). The asymmetric unit of the title compound, (I), is shown in Fig. 1. The compound crystallizes in space group $P2_1$ with two molecules in the asymmetric unit. There are no significant differences between the bonds lengths and angles of the two molecules; however, there are significant differences in the magnitudes of some of the equivalent torsion angles involving the atoms N1 and N4, N2 and N5 (Tables 1).



(I)

The intermolecular N2—H···O hydrogen bonds play an important role in the formation of polymeric chains running along the crystallographic *a* axis (Fig. 2). Hydrogen-bond parameters are listed in Table 2.

Experimental

To a vigorously stirred solution of 9-chloroacridine (214 mg, 1 mmol) in methanol (5 ml) was added dropwise a solution of 4-amino-TEMPOL (TEMPOL is 2,2,6,6-tetramethylpiperidine-1-oxyl) (171 mg, 1 mmol) in methanol (2 ml). This solution was then stirred under reflux for 2 h. The resulting mixture was diluted with CH_2Cl_2 (10 ml), washed with saturated Na_2CO_3 and water, then dried over anhydrous Na_2SO_4, and concentrated. The residue was chromatographed on silica gel (hexane/ethyl acetate = 2:1) to give the title compound as an orange solid. Orange crystals (320 mg, yield 92%) were obtained from a CH_2Cl_2/hexane solution after it was left to stand for 4 d. HRMS (ESI) *m*/*z* found for $[M + H]^+$ = 349.2115, calculated for $C_{22}H_{27}N_3O^+$ = 349.2149.

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Crystal data

$C_{22}H_{26}N_3O$
$M_r = 348.46$
Monoclinic, $P2_1$
$a = 11.1168$ (3) Å
$b = 14.2464$ (3) Å
$c = 11.8709$ (3) Å
$\beta = 92.113$ (1)°
$V = 1878.77$ (8) Å^3
$Z = 4$

$D_x = 1.232$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 12 213 reflections
$\theta = 2.2$–27.4°
$\mu = 0.08$ mm^{-1}
$T = 293$ (2) K
Block, orange
0.40 × 0.30 × 0.18 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.954$, $T_{\max} = 0.986$
17 974 measured reflections

4441 independent reflections
3581 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.039$
$\theta_{\max} = 27.4°$
$h = -14 \rightarrow 14$
$k = -18 \rightarrow 18$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.120$
$S = 1.01$
4441 reflections
480 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0776P)^2 + 0.0762P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0046 (13)

Table 1
Selected geometric parameters (Å, °).

C1—N2	1.479 (3)	C25—N4	1.485 (4)
C3—N1	1.493 (3)	C28—N4	1.490 (4)
C6—N1	1.479 (4)	C32—N5	1.387 (3)
C10—N2	1.395 (3)	N1—O1	1.281 (3)
C23—N5	1.472 (3)	N4—O2	1.284 (3)
O1—N1—C6	116.1 (2)	O2—N4—C25	116.3 (2)
O1—N1—C3	115.8 (2)	O2—N4—C28	116.0 (2)
C6—N1—C3	124.97 (18)	C25—N4—C28	124.20 (19)
C10—N2—C1	121.7 (2)	C32—N5—C23	126.5 (2)
C5—C3—N1—O1	50.5 (3)	C30—C28—N4—O2	69.0 (3)
C4—C3—N1—O1	−67.7 (3)	C29—C28—N4—O2	−49.1 (3)
C11—C10—N2—C1	121.6 (3)	C44—C32—N5—C23	−123.9 (3)
C22—C10—N2—C1	−62.1 (4)	C33—C32—N5—C23	61.8 (4)
C2—C1—N2—C10	−98.5 (3)	C24—C23—N5—C32	−133.0 (3)
C9—C1—N2—C10	143.0 (3)	C31—C23—N5—C32	109.1 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H555···O1	0.87	2.19	3.031 (3)	162
N2—H222···O2$^{\rm i}$	0.89	2.27	3.109 (3)	160

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

Atoms H222 and H555 were found in a difference Fourier map and fixed in position. The methyl H atoms were constrained to an ideal geometry [C—H = 0.96 Å and $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$] and were allowed to rotate freely about the C—C bonds. The other H atoms were placed in calculated positions, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$ of the carrier atoms and included in the final cycles of refinement in the riding model approximation.

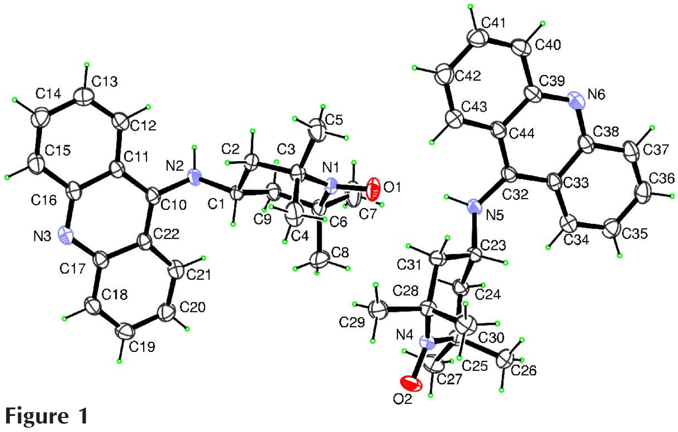

Figure 1
View of the asymmetric unit of (I). Displacement ellipsoids are drawn at the 30% probability level.

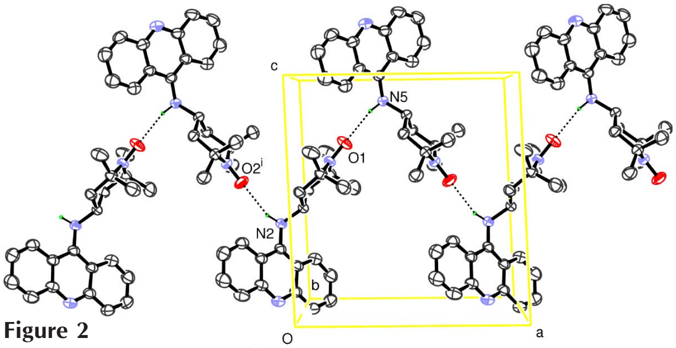

Figure 2
A view of the structure of (I), viewed along the b axis, showing hydrogen-bonded chains (dashed lines) running along the a axis. H atoms not involved in the hydrogen bonding have been omitted.

Data collection: *PROCESS-AUTO* (Version 1.06; Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Version 3.60; Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* publication routines (Farrugia, 1999).

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References

Aliaga, C., Aspee, A. & Scaiano, J. C. (2003). *Org. Lett.* **5**, 4145.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS*-AUTO. Version 1.06. Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2004). *CrystalStructure*. Version 3.6.0. Rigaku/MSC, 9009 New Trails Drive, The Woodlands, TX 77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.