Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2,2-Dimethyl-5-(1-naphthylamino-methylene)-1,3-dioxane-4,6-dione

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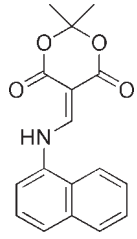
Received 29 July 2009; accepted 25 August 2009

Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.110; data-to-parameter ratio = 15.5.

The benzyl ring of the title compound, $C_{17}H_{15}NO_4$, is twisted away from the plane defined by five atoms of the dioxane ring by 34.83 (4)°. The dioxane ring exhibits a half-boat conformation, with the C atom between the dioxane O atoms 0.571 (8) Å out of the plane through the remainder of the ring. An intramolecular N—H···O hydrogen bond may contribute to the stabilization of the planar conformation of the molecule. In the crystal, inversion dimers linked by pairs of C—H···O bonds occur.

Related literature

For the synthesis of related compounds, see: Cassis *et al.* (1985). For the pharmacological activity of 4(1*H*)-quinolone structures, see: Ruchelman *et al.* (2003).



Experimental

Crystal data

$C_{17}H_{15}NO_4$
M_r = 297.30
Triclinic, $P\overline{1}$
a = 7.4696 (11) Å
b = 8.0805 (12) Å
c = 12.1240 (18) Å
α = 98.601 (2)°
β = 96.428 (2)°
γ = 92.198 (2)°
V = 717.87 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 153 K
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
4513 measured reflections
3194 independent reflections
2571 reflections with $I > 2\sigma(I)$
R_{int} = 0.014

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.110
S = 1.03
3194 reflections
206 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.920 (18)	1.982 (18)	2.7130 (16)	135.2 (15)
C1—H1C···O4^{i}	0.98	2.60	3.3709 (19)	136

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

This research was supported financially by the State Key Laboratory of Drug Research, Shanghai Institute of Materia Medica, Chinese Academy of Sciences.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2178).

References

Bruker (2000). *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2001). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Cassis, R., Tapia, R. & Valderrama, J. A. (1985). *Synth. Commun.* **15**, 125–133.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Ruchelman, A. L., Singh, S. K., Ray, A., Wu, X. H., Yang, J. M., Li, T. K., Liu, A., Liu, L. F. & LaVoie, E. J. (2003). *Bioorg. Med. Chem.* **11**, 2061–2073.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supporting information

Acta Cryst. (2009). E**65**, o2289 [doi:10.1107/S1600536809033984]

2,2-Dimethyl-5-(1-naphthylaminomethylene)-1,3-dioxane-4,6-dione

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S1. Comment

4(1*H*)-Quinolone structures have long attracted pharmacological interest as anticancer agents, anti-malarial agents and reversible (H+/K+) ATPase inhibitors (Ruchelman *et al.*, 2003). Thermolysis of 5-arylaminomethylene-2,2-dimethyl-1,3-dioxane-4,6-diones is an effective method to synthesize 4(1*H*)quinolone derivatives (Cassis *et al.*, 1985).

The benzyl ring is twisted away from the plane defined by the dioxane ring by 34.83 (4)°. In turn, the dioxane ring of the title compound exhibits an envelope conformation, in which the flap atom, the C atom between the dioxane oxygen atoms, is -0.571 (8) Å out of the plane. An intramolecular N—H···O hydrogen bond (Table 1) could lead to the dioxane ring and the aminomethylene group taking up their planar conformation.

S2. Experimental

An ethanol solution (50 ml) of 2,2-dimethyl-1,3-dioxane-4,6-dione (Meldrum's acid) (1.44 g, 0.01 mol) and methyl-orthoformate (1.27 g, 0.012 mol) was heated to reflux for 2 h, then the naphthalen-1-amine (1.43 g, 0.01 mol) was added into the above solution. The mixture was heated under reflux for another 8 h and then filtered. Single crystals were obtained from the filtrate after 2 days.

S3. Refinement

The imino H atom was located in a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and refined using a riding model with U_{iso}(H) = 1.5U_{eq}(C) for methyl and 1.2U_{eq}(C) for the others.

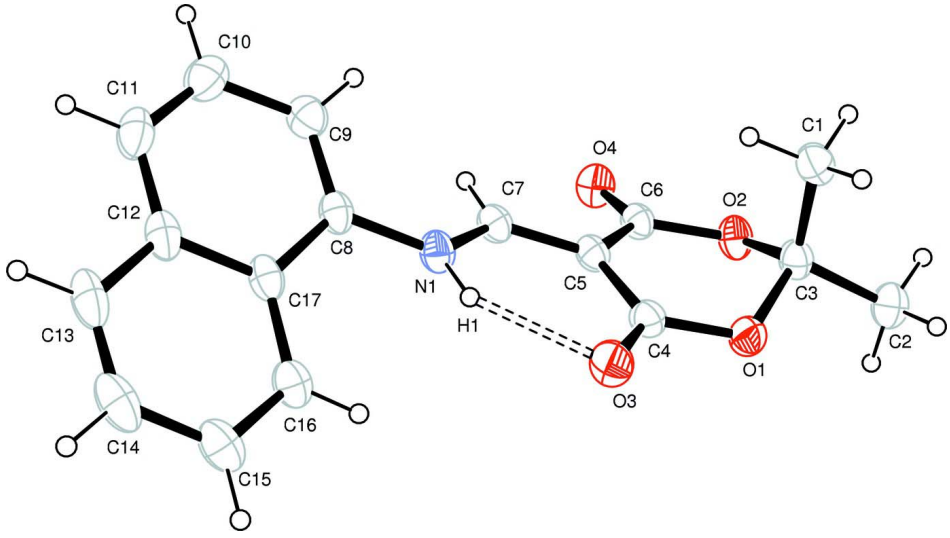

Figure 1

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level. The hydrogen bond is shown by a dashed line.

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Crystal data

$C_{17}H_{15}NO_4$
$M_r = 297.30$
Triclinic, $P\overline{1}$
$a = 7.4696\ (11)$ Å
$b = 8.0805\ (12)$ Å
$c = 12.1240\ (18)$ Å
$\alpha = 98.601\ (2)°$
$\beta = 96.428\ (2)°$
$\gamma = 92.198\ (2)°$
$V = 717.87\ (18)$ Å^3
$Z = 2$
$F(000) = 312$
$D_x = 1.375$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2197 reflections
$\theta = 2.6–27.5°$
$\mu = 0.10$ mm^{-1}
$T = 153$ K
Block, colourless
0.25 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
4513 measured reflections
3194 independent reflections
2571 reflections with $I > 2\sigma(I)$
$R_{int} = 0.014$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.6°$
$h = -9\rightarrow6$
$k = -10\rightarrow10$
$l = -15\rightarrow15$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 1.03$
3194 reflections
206 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.1278P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}

Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^{*}=kFc[1+0.001xFc^{2}\lambda^{3}/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.204 (9)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.75507 (13)	0.44446 (12)	0.48110 (8)	0.0482 (3)
O2	0.61634 (12)	0.29559 (12)	0.60505 (7)	0.0468 (3)
O3	0.66547 (15)	0.40648 (13)	0.29855 (8)	0.0567 (3)
O4	0.36615 (13)	0.13711 (12)	0.54766 (8)	0.0501 (3)
N1	0.41749 (15)	0.15238 (15)	0.21325 (9)	0.0431 (3)
H1	0.496 (3)	0.236 (2)	0.2012 (15)	0.074 (5)*
C1	0.9036 (2)	0.21096 (19)	0.54644 (13)	0.0529 (4)
H1A	0.9247	0.1511	0.6109	0.079*
H1B	1.0193	0.2541	0.5281	0.079*
H1C	0.8432	0.1340	0.4816	0.079*
C2	0.8669 (2)	0.4816 (2)	0.67397 (13)	0.0583 (4)
H2A	0.7830	0.5704	0.6887	0.087*
H2B	0.9809	0.5306	0.6571	0.087*
H2C	0.8895	0.4262	0.7404	0.087*
C3	0.78588 (18)	0.35520 (17)	0.57518 (11)	0.0433 (3)
C4	0.64805 (18)	0.36396 (16)	0.38885 (11)	0.0420 (3)
C5	0.51881 (17)	0.23873 (16)	0.40821 (10)	0.0384 (3)
C6	0.49111 (17)	0.21600 (16)	0.52166 (10)	0.0393 (3)
C7	0.41364 (17)	0.14044 (16)	0.32061 (10)	0.0401 (3)
H7	0.3322	0.0579	0.3385	0.048*
C8	0.32676 (17)	0.04147 (17)	0.12053 (10)	0.0412 (3)
C9	0.2809 (2)	−0.12053 (18)	0.12899 (12)	0.0502 (3)
H9	0.3068	−0.1605	0.1986	0.060*
C10	0.1953 (2)	−0.2285 (2)	0.03460 (14)	0.0598 (4)
H10	0.1584	−0.3399	0.0416	0.072*
C11	0.1648 (2)	−0.1750 (2)	−0.06667 (13)	0.0593 (4)
H11	0.1097	−0.2505	−0.1301	0.071*
C12	0.21389 (18)	−0.0086 (2)	−0.07868 (11)	0.0495 (4)
C13	0.1908 (2)	0.0502 (3)	−0.18422 (12)	0.0624 (5)
H13	0.1415	−0.0247	−0.2495	0.075*
C14	0.2376 (2)	0.2106 (3)	−0.19338 (13)	0.0663 (5)
H14	0.2247	0.2460	−0.2650	0.080*

C15	0.3050 (2)	0.3242 (2)	−0.09805 (13)	0.0608 (4)
H15	0.3325	0.4378	−0.1047	0.073*
C16	0.3315 (2)	0.2734 (2)	0.00486 (12)	0.0513 (4)
H16	0.3775	0.3521	0.0690	0.062*
C17	0.29151 (17)	0.10513 (18)	0.01706 (10)	0.0428 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0558 (6)	0.0465 (5)	0.0405 (5)	−0.0043 (4)	−0.0028 (4)	0.0094 (4)
O2	0.0450 (5)	0.0633 (6)	0.0309 (5)	−0.0001 (4)	0.0023 (4)	0.0057 (4)
O3	0.0686 (7)	0.0633 (6)	0.0400 (6)	−0.0081 (5)	0.0034 (5)	0.0195 (5)
O4	0.0499 (6)	0.0620 (6)	0.0394 (5)	−0.0027 (5)	0.0111 (4)	0.0081 (4)
N1	0.0441 (6)	0.0532 (7)	0.0315 (6)	0.0003 (5)	0.0015 (4)	0.0076 (5)
C1	0.0452 (8)	0.0568 (8)	0.0550 (9)	0.0065 (6)	−0.0014 (6)	0.0080 (7)
C2	0.0610 (9)	0.0622 (9)	0.0457 (8)	−0.0021 (7)	−0.0061 (7)	−0.0001 (7)
C3	0.0440 (7)	0.0503 (7)	0.0346 (7)	0.0002 (6)	0.0001 (5)	0.0074 (5)
C4	0.0478 (7)	0.0433 (7)	0.0349 (7)	0.0044 (5)	0.0014 (5)	0.0081 (5)
C5	0.0398 (7)	0.0448 (7)	0.0311 (6)	0.0056 (5)	0.0023 (5)	0.0079 (5)
C6	0.0398 (7)	0.0447 (7)	0.0335 (6)	0.0069 (5)	0.0037 (5)	0.0055 (5)
C7	0.0391 (7)	0.0483 (7)	0.0341 (6)	0.0060 (5)	0.0047 (5)	0.0087 (5)
C8	0.0357 (6)	0.0547 (8)	0.0322 (6)	0.0057 (5)	0.0033 (5)	0.0030 (5)
C9	0.0539 (8)	0.0557 (8)	0.0414 (7)	0.0080 (6)	0.0071 (6)	0.0062 (6)
C10	0.0617 (10)	0.0548 (9)	0.0594 (10)	0.0020 (7)	0.0080 (7)	−0.0026 (7)
C11	0.0517 (9)	0.0699 (10)	0.0475 (9)	0.0058 (7)	−0.0028 (7)	−0.0136 (7)
C12	0.0386 (7)	0.0714 (9)	0.0354 (7)	0.0127 (6)	0.0010 (5)	−0.0017 (6)
C13	0.0523 (9)	0.0978 (13)	0.0327 (7)	0.0195 (8)	−0.0036 (6)	−0.0017 (8)
C14	0.0590 (10)	0.1060 (14)	0.0386 (8)	0.0215 (9)	0.0055 (7)	0.0229 (9)
C15	0.0567 (9)	0.0829 (11)	0.0477 (9)	0.0095 (8)	0.0052 (7)	0.0246 (8)
C16	0.0489 (8)	0.0663 (9)	0.0391 (7)	0.0034 (7)	0.0023 (6)	0.0119 (6)
C17	0.0343 (6)	0.0622 (8)	0.0315 (6)	0.0089 (6)	0.0040 (5)	0.0044 (6)

Geometric parameters (Å, º)

O1—C4	1.3622 (15)	C7—H7	0.9500
O1—C3	1.4403 (15)	C8—C9	1.362 (2)
O2—C6	1.3632 (15)	C8—C17	1.4271 (18)
O2—C3	1.4405 (16)	C9—C10	1.405 (2)
O3—C4	1.2142 (15)	C9—H9	0.9500
O4—C6	1.2072 (15)	C10—C11	1.360 (2)
N1—C7	1.3229 (16)	C10—H10	0.9500
N1—C8	1.4170 (16)	C11—C12	1.413 (2)
N1—H1	0.920 (18)	C11—H11	0.9500
C1—C3	1.510 (2)	C12—C17	1.4204 (19)
C1—H1A	0.9800	C12—C13	1.425 (2)
C1—H1B	0.9800	C13—C14	1.353 (3)
C1—H1C	0.9800	C13—H13	0.9500
C2—C3	1.5063 (19)	C14—C15	1.396 (2)

C2—H2A	0.9800	C14—H14	0.9500
C2—H2B	0.9800	C15—C16	1.367 (2)
C2—H2C	0.9800	C15—H15	0.9500
C4—C5	1.4345 (18)	C16—C17	1.414 (2)
C5—C7	1.3743 (17)	C16—H16	0.9500
C5—C6	1.4508 (17)		

C4—O1—C3	116.84 (10)	N1—C7—H7	117.6
C6—O2—C3	118.48 (10)	C5—C7—H7	117.6
C7—N1—C8	126.21 (12)	C9—C8—N1	121.29 (12)
C7—N1—H1	113.8 (11)	C9—C8—C17	121.54 (12)
C8—N1—H1	119.7 (11)	N1—C8—C17	117.13 (12)
C3—C1—H1A	109.5	C8—C9—C10	119.85 (14)
C3—C1—H1B	109.5	C8—C9—H9	120.1
H1A—C1—H1B	109.5	C10—C9—H9	120.1
C3—C1—H1C	109.5	C11—C10—C9	120.64 (15)
H1A—C1—H1C	109.5	C11—C10—H10	119.7
H1B—C1—H1C	109.5	C9—C10—H10	119.7
C3—C2—H2A	109.5	C10—C11—C12	120.82 (14)
C3—C2—H2B	109.5	C10—C11—H11	119.6
H2A—C2—H2B	109.5	C12—C11—H11	119.6
C3—C2—H2C	109.5	C11—C12—C17	119.39 (13)
H2A—C2—H2C	109.5	C11—C12—C13	122.49 (14)
H2B—C2—H2C	109.5	C17—C12—C13	118.11 (15)
O1—C3—O2	110.06 (10)	C14—C13—C12	121.36 (15)
O1—C3—C2	106.69 (11)	C14—C13—H13	119.3
O2—C3—C2	105.71 (11)	C12—C13—H13	119.3
O1—C3—C1	109.85 (11)	C13—C14—C15	120.28 (15)
O2—C3—C1	110.62 (11)	C13—C14—H14	119.9
C2—C3—C1	113.75 (12)	C15—C14—H14	119.9
O3—C4—O1	118.14 (12)	C16—C15—C14	120.52 (16)
O3—C4—C5	125.52 (12)	C16—C15—H15	119.7
O1—C4—C5	116.31 (11)	C14—C15—H15	119.7
C7—C5—C4	121.36 (11)	C15—C16—C17	120.84 (15)
C7—C5—C6	117.92 (11)	C15—C16—H16	119.6
C4—C5—C6	120.69 (11)	C17—C16—H16	119.6
O4—C6—O2	118.28 (11)	C16—C17—C12	118.73 (13)
O4—C6—C5	125.74 (12)	C16—C17—C8	123.65 (12)
O2—C6—C5	115.95 (11)	C12—C17—C8	117.61 (13)
N1—C7—C5	124.76 (12)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.920 (18)	1.982 (18)	2.7130 (16)	135.2 (15)
C1—H1*C*···O4^{i}	0.98	2.60	3.3709 (19)	136

Symmetry code: (i) −*x*+1, −*y*, −*z*+1.