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2,2-Dimethyl-5-[(pyridin-2-ylamino)-methylidene]-1,3-dioxane-4,6-dione

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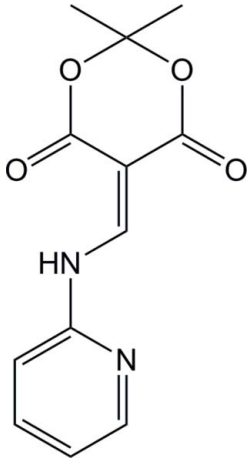
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.040; wR factor = 0.096; data-to-parameter ratio = 14.1.

In the title compound, $C_{12}H_{12}N_2O_4$, the dihedral angle between the pyridine and enamine planes is 3.5 (3)°, while the angle between the dioxanedione (seven atoms) and enamine planes is 4.6 (3)°. The dioxane ring approximates an envelope conformation.

Related literature

The title compound is an intermediate in the synthesis of 4(1*H*)-quinolone-based drugs. For the synthesis and structures of related antitumor precursors, see: Cassis *et al.* (1985); Ruchelman *et al.* (2003); Shi *et al.* (2009).



Experimental

Crystal data

$C_{12}H_{12}N_2O_4$
M_r = 248.24
Monoclinic, $P2_1/c$
a = 8.7344 (10) Å
b = 13.9712 (15) Å
c = 9.4744 (11) Å
β = 94.601 (11)°
V = 1152.4 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.22 × 0.18 × 0.16 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with an Eos CCD detector
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
T_{min} = 0.997, T_{max} = 1.0
4873 measured reflections
2334 independent reflections
1659 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.096
S = 1.03
2334 reflections
166 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2009); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009).

We thank Mr Zhihua Mao of the Analysis and Testing Center (Sichuan University) for the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2327).

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2,2-Dimethyl-5-[(pyridin-2-ylamino)methylidene]-1,3-dioxane-4,6-dione

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S1. Comment

The title compound is a key intermediates, which can be used to synthesize 4(1*H*)-quinolone derivatives by thermolysis. These compounds can be used as precursors for anti-malarial and anticancer agents (Cassis *et al.*, 1985; Ruchelman *et al.*, 2003; Shi *et al.*, 2009).

S2. Experimental

A mixture of 2,2-dimethyl-1,3-dioxane-4,6-dione (1.44 g, 0.01 mol) and methylorthoformate (1.27 g, 0.012 mol) was refluxed for 2.5 h. Then pyridin-2-amine (0.94 g, 0.01 mol) was added and the mixture was refluxed for 4 h, then poured into cold water and filtered, to afford the title compound as a powder. Single crystals were obtained by slow evaporation of a CH_2Cl_2-methanol solution over 3 days.

S3. Refinement

All H atoms were placed in calculated positions, with C—H bond lengths fixed to 0.93 (aromatic CH), 0.96 (methyl CH_3) or 0.86 Å (NH group). Isotropic displacement parameters for H atoms were calculated as 1.5 (methyl) or 1.2 (other H atoms) times that of the equivalent displacement parameter of the carrier C atom.

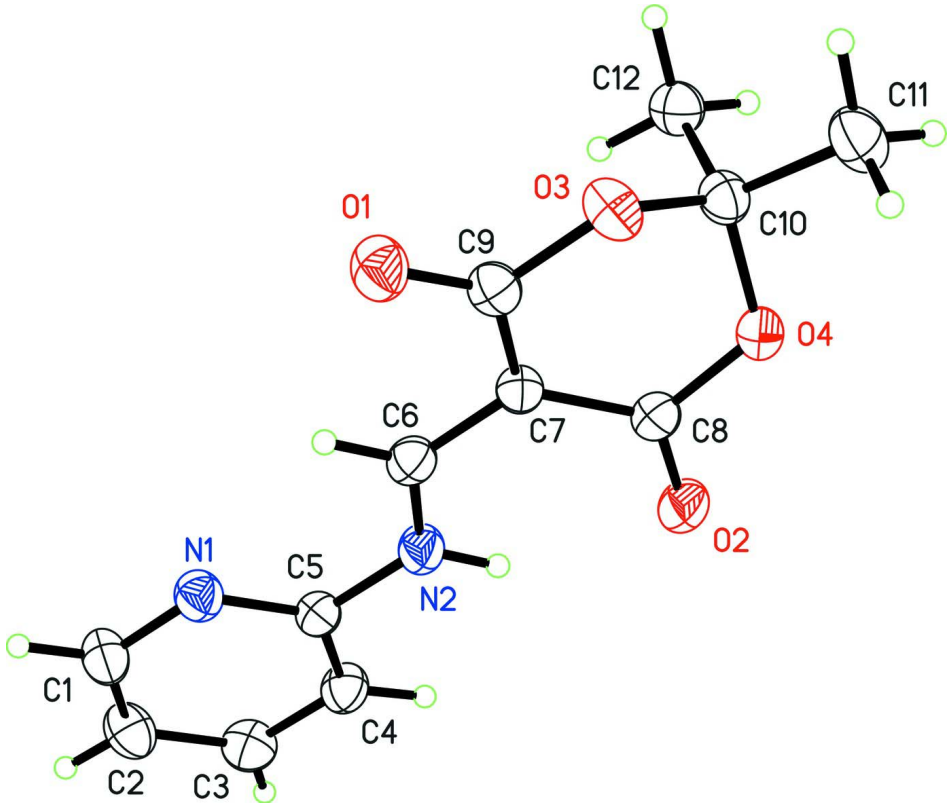

Figure 1

ORTEP-like view of the title compound.

2,2-Dimethyl-5-[(pyridin-2-ylamino)methylidene]-1,3-dioxane-4,6-dione

Crystal data

$C_{12}H_{12}N_2O_4$
$M_r = 248.24$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.7344\ (10)$ Å
$b = 13.9712\ (15)$ Å
$c = 9.4744\ (11)$ Å
$\beta = 94.601\ (11)°$
$V = 1152.4\ (2)$ Å^3
$Z = 4$
$F(000) = 520$
$D_x = 1.431$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å
Cell parameters from 1780 reflections
$\theta = 3.1–29.2°$
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Block, colourless
0.22 × 0.18 × 0.16 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with an Eos CCD detector
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 16.0874 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2009)
$T_{min} = 0.997$, $T_{max} = 1.0$
4873 measured reflections
2334 independent reflections
1659 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 26.4°$, $\theta_{min} = 3.4°$
$h = -10\rightarrow9$
$k = -17\rightarrow16$
$l = -11\rightarrow11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.096$
$S = 1.03$
2334 reflections
166 parameters
1 restraint
0 constraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0383P)^2 + 0.0655P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.14$ e Å^{-3}
$\Delta\rho_{min} = -0.14$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0060 (9)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O3	0.21039 (13)	0.15916 (7)	0.50800 (11)	0.0497 (3)
O4	0.37760 (12)	0.11610 (7)	0.33485 (12)	0.0504 (3)
C6	0.22965 (17)	−0.10004 (11)	0.49185 (16)	0.0421 (4)
H6	0.1613	−0.1134	0.5596	0.050*
O1	0.10857 (14)	0.05240 (7)	0.64457 (11)	0.0556 (3)
O2	0.44968 (12)	−0.03377 (8)	0.30745 (12)	0.0559 (3)
N2	0.29233 (14)	−0.17386 (8)	0.43038 (13)	0.0455 (3)
H2	0.3571	−0.1614	0.3692	0.055*
C7	0.25719 (16)	−0.00541 (10)	0.46383 (15)	0.0388 (3)
N1	0.17056 (15)	−0.28950 (8)	0.55799 (13)	0.0474 (4)
C1	0.1423 (2)	−0.38227 (11)	0.58158 (18)	0.0532 (4)
H1	0.0776	−0.3976	0.6515	0.064*
C9	0.18429 (18)	0.06634 (10)	0.54492 (16)	0.0428 (4)
C8	0.36562 (17)	0.02142 (11)	0.36380 (16)	0.0434 (4)
C5	0.26224 (16)	−0.27096 (10)	0.45637 (16)	0.0400 (4)
C3	0.2993 (2)	−0.43431 (11)	0.40519 (18)	0.0547 (5)
H3	0.3432	−0.4829	0.3548	0.066*
C4	0.32929 (19)	−0.34013 (11)	0.37743 (17)	0.0487 (4)
H4	0.3930	−0.3233	0.3074	0.058*
C12	0.11654 (19)	0.16341 (11)	0.26210 (16)	0.0493 (4)
H12B	0.1452	0.1783	0.1689	0.074*
H12C	0.0343	0.2048	0.2851	0.074*
H12A	0.0833	0.0980	0.2649	0.074*
C11	0.3145 (2)	0.27839 (11)	0.36841 (19)	0.0606 (5)
H11A	0.3519	0.2923	0.2781	0.091*
H11C	0.3969	0.2843	0.4412	0.091*
H11B	0.2342	0.3226	0.3864	0.091*
C2	0.2036 (2)	−0.45560 (11)	0.50844 (18)	0.0582 (5)
H2A	0.1806	−0.5189	0.5286	0.070*
C10	0.25227 (18)	0.17780 (10)	0.36764 (16)	0.0439 (4)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O3	0.0704 (8)	0.0405 (6)	0.0386 (6)	−0.0001 (5)	0.0061 (5)	−0.0014 (5)
O4	0.0432 (7)	0.0437 (6)	0.0656 (8)	−0.0011 (5)	0.0137 (5)	0.0085 (5)
C6	0.0384 (9)	0.0485 (9)	0.0395 (9)	0.0012 (7)	0.0042 (7)	−0.0004 (7)
O1	0.0669 (8)	0.0573 (7)	0.0448 (7)	0.0015 (6)	0.0178 (6)	0.0005 (5)
O2	0.0470 (7)	0.0543 (7)	0.0692 (8)	0.0047 (6)	0.0208 (6)	0.0026 (6)
N2	0.0452 (8)	0.0426 (8)	0.0502 (8)	0.0012 (6)	0.0140 (6)	0.0015 (6)
C7	0.0374 (8)	0.0399 (8)	0.0390 (8)	−0.0006 (7)	0.0034 (6)	0.0022 (7)
N1	0.0516 (9)	0.0447 (8)	0.0471 (8)	0.0022 (6)	0.0102 (6)	0.0016 (6)
C1	0.0620 (11)	0.0477 (10)	0.0511 (10)	−0.0035 (9)	0.0121 (8)	0.0056 (8)
C9	0.0460 (9)	0.0445 (9)	0.0373 (9)	−0.0010 (7)	−0.0001 (7)	0.0006 (7)
C8	0.0372 (9)	0.0438 (9)	0.0491 (9)	−0.0005 (7)	0.0020 (7)	0.0017 (7)
C5	0.0376 (8)	0.0403 (9)	0.0418 (9)	−0.0009 (7)	0.0022 (6)	0.0015 (7)
C3	0.0624 (12)	0.0465 (10)	0.0557 (11)	0.0058 (8)	0.0072 (9)	−0.0100 (8)
C4	0.0478 (10)	0.0521 (10)	0.0477 (10)	0.0015 (8)	0.0123 (8)	−0.0025 (7)
C12	0.0534 (10)	0.0523 (9)	0.0422 (10)	0.0029 (8)	0.0047 (7)	0.0012 (7)
C11	0.0704 (13)	0.0451 (10)	0.0651 (12)	−0.0087 (9)	−0.0009 (9)	0.0058 (8)
C2	0.0728 (13)	0.0414 (9)	0.0606 (12)	−0.0031 (9)	0.0069 (9)	0.0017 (8)
C10	0.0495 (10)	0.0411 (9)	0.0419 (9)	−0.0002 (7)	0.0078 (7)	0.0016 (7)

Geometric parameters (Å, º)

O3—C9	1.3671 (16)	C1—C2	1.370 (2)
O3—C10	1.4311 (18)	C5—C4	1.381 (2)
O4—C8	1.3567 (17)	C3—H3	0.9300
O4—C10	1.4465 (18)	C3—C4	1.371 (2)
C6—H6	0.9300	C3—C2	1.370 (2)
C6—N2	1.3242 (18)	C4—H4	0.9300
C6—C7	1.374 (2)	C12—H12B	0.9600
O1—C9	1.2112 (17)	C12—H12C	0.9600
O2—C8	1.2172 (17)	C12—H12A	0.9600
N2—H2	0.8600	C12—C10	1.502 (2)
N2—C5	1.4072 (18)	C11—H11A	0.9600
C7—C9	1.442 (2)	C11—H11C	0.9600
C7—C8	1.442 (2)	C11—H11B	0.9600
N1—C1	1.3415 (19)	C11—C10	1.507 (2)
N1—C5	1.3263 (18)	C2—H2A	0.9300
C1—H1	0.9300		
O3—C9—C7	115.68 (13)	C5—N2—H2	117.1
O3—C10—O4	110.21 (11)	C5—N1—C1	116.06 (13)
O3—C10—C12	110.33 (13)	C5—C4—H4	120.9
O3—C10—C11	106.51 (12)	C3—C4—C5	118.16 (15)
O4—C8—C7	116.85 (13)	C3—C4—H4	120.9
O4—C10—C12	110.35 (12)	C3—C2—C1	118.99 (15)
O4—C10—C11	106.15 (13)	C3—C2—H2A	120.5

C6—N2—H2	117.1	C4—C5—N2	119.12 (13)
C6—N2—C5	125.74 (13)	C4—C3—H3	120.6
C6—C7—C9	118.33 (13)	C12—C10—C11	113.15 (13)
C6—C7—C8	120.77 (14)	H12B—C12—H12C	109.5
O1—C9—O3	117.68 (13)	H12B—C12—H12A	109.5
O1—C9—C7	126.57 (14)	H12C—C12—H12A	109.5
O2—C8—O4	118.03 (13)	H11A—C11—H11C	109.5
O2—C8—C7	125.08 (14)	H11A—C11—H11B	109.5
N2—C6—H6	117.3	H11C—C11—H11B	109.5
N2—C6—C7	125.42 (14)	C2—C1—H1	118.2
C7—C6—H6	117.3	C2—C3—H3	120.6
N1—C1—H1	118.2	C2—C3—C4	118.84 (15)
N1—C1—C2	123.64 (15)	C10—C12—H12B	109.5
N1—C5—N2	116.58 (13)	C10—C12—H12C	109.5
N1—C5—C4	124.30 (14)	C10—C12—H12A	109.5
C1—C2—H2A	120.5	C10—C11—H11A	109.5
C9—O3—C10	118.08 (11)	C10—C11—H11C	109.5
C9—C7—C8	120.72 (13)	C10—C11—H11B	109.5
C8—O4—C10	117.75 (11)		

C6—N2—C5—N1	−4.6 (2)	C9—O3—C10—C11	−165.07 (13)
C6—N2—C5—C4	175.96 (15)	C9—C7—C8—O4	−9.6 (2)
C6—C7—C9—O3	−177.16 (12)	C9—C7—C8—O2	168.03 (15)
C6—C7—C9—O1	6.0 (2)	C8—O4—C10—O3	47.99 (17)
C6—C7—C8—O4	175.36 (13)	C8—O4—C10—C12	−74.10 (16)
C6—C7—C8—O2	−7.0 (2)	C8—O4—C10—C11	162.95 (12)
N2—C6—C7—C9	−177.35 (14)	C8—C7—C9—O3	7.7 (2)
N2—C6—C7—C8	−2.2 (2)	C8—C7—C9—O1	−169.19 (15)
N2—C5—C4—C3	179.53 (14)	C5—N1—C1—C2	0.7 (2)
C7—C6—N2—C5	−178.57 (14)	C4—C3—C2—C1	−0.7 (3)
N1—C1—C2—C3	0.0 (3)	C2—C3—C4—C5	0.6 (2)
N1—C5—C4—C3	0.2 (2)	C10—O3—C9—O1	−159.37 (14)
C1—N1—C5—N2	179.83 (13)	C10—O3—C9—C7	23.48 (19)
C1—N1—C5—C4	−0.8 (2)	C10—O4—C8—O2	162.84 (14)
C9—O3—C10—O4	−50.35 (17)	C10—O4—C8—C7	−19.38 (19)
C9—O3—C10—C12	71.76 (16)		