

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Julolidine-1,6-dione

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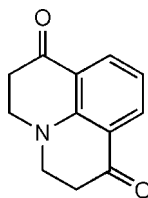
Received 10 October 2007; accepted 7 November 2007

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.061; wR factor = 0.163; data-to-parameter ratio = 16.0.

The title molecule, $\text{C}_{12}\text{H}_{11}\text{NO}_2$, has crystallographic mirror symmetry. Weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds help stabilize the crystal structure.

Related literature

For related literature, see: Johnson *et al.* (1949); Domenicano *et al.* (1975); Allen *et al.* (1987).



Experimental

Crystal data

 $\text{C}_{12}\text{H}_{11}\text{NO}_2$ $M_r = 201.22$ Orthorhombic, $Pnma$ $a = 10.069$ (2) Å $b = 14.081$ (3) Å $c = 6.9452$ (14) Å $V = 984.7$ (3) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 0.09$ mm⁻¹ $T = 293$ (2) K $0.15 \times 0.15 \times 0.15$ mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.720$, $T_{\max} = 1.00$
(expected range = 0.710–0.986)

9450 measured reflections
1170 independent reflections
767 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.086$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.061$ $wR(F^2) = 0.163$ $S = 1.10$

1170 reflections

73 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.15$ e Å⁻³ $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2A\cdots\text{O}1^i$	0.93	2.50	3.372 (3)	157
$\text{C}6-\text{H}6A\cdots\text{O}1^{ii}$	0.97	2.52	3.377 (3)	147

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

Data collection: *CrystalClear* (Rigaku, 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: *SHELXTL* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL*.

The authors are grateful for the financial support from the Southeast University Foundation (No. 9207041358).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2533).

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supplementary materials

Acta Cryst. (2007). E63, o4699 [doi:10.1107/S1600536807056802]

Julolidine-1,6-dione

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Comment

The title compound, belongs to a class of 4-dihydroquinolinone derivatives (William, *et al.*, 1949), which have hitherto received relatively little attention. We have recently found that the title compound undergoes two-photon absorption and two-photon excited fluorescence and is therefore of interest in the study of non-linear optics. An X-ray crystal structure determination of was undertaken in order to elucidate the conformation, and the results are presented here.

The bond lengths observed in the phenyl rings is comparable with average values reported in the literature (Domenicano *et al.*, 1975; Allen *et al.*, 1987). Atom C7 deviates by 0.493 (2) Å from the benzene ring. The mean plane through atoms O1/C5/C6 is twisted from the plane of the benzene ring by 7.5 (2) °, while in acetophenone the acetyl group is essentially co-planar with benzene ring. This twist is most likely due to the strain imposed by sp^3 -hybridized atoms C6 and C7 in the adjacent ring. The N atom lies approximately in the plane of the benzene ring with a deviation of 0.023 (2) Å, as might be expected for maximum conjugation normally found for N atom substituents on benzene rings. Weak intermolecular C—H···O hydrogen bonds help stabilize the crystal structure.

Experimental

Melting points were determined with a Yanagimoto MP-35 melting-point apparatus and were uncorrected. The ^1H NMR spectra were measured with a Bruker DRX (500 MHz) (relative to TMS) spectrometer. The solid state IR spectra were recorded from KBr discs on a Nicolet-170.

A mixture of N, *N*-bis-2-cyanoethylaniine (10 g, 0.055 mol), powdered aluminium chloride (55 g, 0.422 mol), 30 ml chlorobenzene and 1.5 ml concentrated hydrochloric acid was vigorously stirred at 433 K for 8 h. After cooling, the red oily mixture was hydrolysed with 200 ml ice water, and distilled in steam to remove chlorobenzene. The residue was recrystallized from ethanol to afford a yellow solid. Yield: 8.5 g (85%); m. p. 415 – 416 K. IR (KBr): $\nu = 1676\text{ cm}^{-1}$ (C=O). ^1H NMR (500 MHz, CDCl_3): δ 2.83–2.86 (t, 4H, 2CH_2 , $J = 7.0$ Hz), 3.49–3.52 (t, 4H, 2CH_2 , $J = 7.0$ Hz), 6.86–6.89 (t, 1H, ArH, $J = 7.5$ Hz), 8.11–8.12 (d, 2H, ArH, $J = 8.0$ Hz). Single crystals suitable for crystallographic analysis were obtained by slow evaporation of a methanol water (4:1 v/v) solution.

Refinement

H atoms were placed in calculated positions with C—H = 0.93–0.97 Å and included in the refinement in the riding-model approximation with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Figures

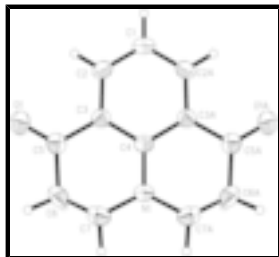


Fig. 1. The molecular structure with displacement ellipsoids were drawn at the 30% probability level [symmetry code: (A) $x, -y + 1/2, z$].

julolidine-1,6-dione

Crystal data

$C_{12}H_{11}NO_2$

$M_r = 201.22$

Orthorhombic, $Pnma$

Hall symbol: $-P\ 2ac\ 2n$

$a = 10.069\ (2)\ \text{\AA}$

$b = 14.081\ (3)\ \text{\AA}$

$c = 6.9452\ (14)\ \text{\AA}$

$V = 984.7\ (3)\ \text{\AA}^3$

$Z = 4$

$F_{000} = 424$

$D_x = 1.357\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073\ \text{\AA}$

Cell parameters from 7909 reflections

$\theta = 3.3\text{--}28.9^\circ$

$\mu = 0.09\ \text{mm}^{-1}$

$T = 293\ (2)\ \text{K}$

Block, colorless

$0.15 \times 0.15 \times 0.15\ \text{mm}$

Data collection

Rigaku Mercury2
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 293\ (2)\ \text{K}$

ω scans

Absorption correction: multi-scan
(CrystalClear; Rigaku, 2005)

$T_{\min} = 0.720, T_{\max} = 1.00$

9450 measured reflections

1170 independent reflections

767 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.086$

$\theta_{\max} = 27.5^\circ$

$\theta_{\min} = 3.3^\circ$

$h = -13 \rightarrow 13$

$k = -18 \rightarrow 18$

$l = -9 \rightarrow 9$

Standard reflections: ?

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.163$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.076P)^2 + 0.0053P]$$

where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.10$	$(\Delta/\sigma)_{\max} < 0.001$
1170 reflections	$\Delta\rho_{\max} = 0.15 \text{ e } \text{\AA}^{-3}$
73 parameters	$\Delta\rho_{\min} = -0.19 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.2702 (3)	0.2500	1.2858 (4)	0.0675 (9)
H1A	0.2326	0.2500	1.4080	0.081*
C2	0.2983 (2)	0.16584 (14)	1.1947 (3)	0.0578 (6)
H2A	0.2760	0.1089	1.2545	0.069*
C3	0.35918 (18)	0.16362 (13)	1.0158 (2)	0.0469 (5)
C4	0.3938 (2)	0.2500	0.9238 (3)	0.0423 (6)
C5	0.37694 (18)	0.07268 (15)	0.9155 (3)	0.0529 (5)
C6	0.4347 (2)	0.07766 (16)	0.7178 (3)	0.0643 (6)
H6A	0.3633	0.0809	0.6242	0.077*
H6B	0.4852	0.0203	0.6928	0.077*
C7	0.5239 (2)	0.16269 (14)	0.6933 (3)	0.0613 (6)
H7A	0.6025	0.1548	0.7725	0.074*
H7B	0.5521	0.1670	0.5601	0.074*
O1	0.34091 (15)	-0.00237 (11)	0.9865 (2)	0.0714 (5)
N1	0.4561 (2)	0.2500	0.7476 (3)	0.0506 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.090 (2)	0.071 (2)	0.0412 (15)	0.000	0.0151 (14)	0.000
C2	0.0705 (13)	0.0577 (13)	0.0450 (11)	-0.0091 (10)	0.0034 (9)	0.0066 (9)
C3	0.0479 (10)	0.0494 (12)	0.0435 (11)	-0.0025 (7)	-0.0032 (7)	-0.0006 (8)
C4	0.0389 (12)	0.0524 (16)	0.0356 (12)	0.000	-0.0048 (10)	0.000
C5	0.0513 (11)	0.0526 (12)	0.0548 (12)	-0.0049 (9)	-0.0075 (9)	-0.0041 (9)
C6	0.0735 (14)	0.0618 (14)	0.0576 (12)	0.0065 (11)	-0.0012 (10)	-0.0175 (9)
C7	0.0589 (12)	0.0700 (16)	0.0549 (12)	0.0059 (10)	0.0125 (9)	-0.0053 (9)
O1	0.0794 (11)	0.0532 (10)	0.0815 (11)	-0.0125 (7)	0.0014 (7)	-0.0043 (7)

supplementary materials

N1 0.0511 (12) 0.0577 (15) 0.0429 (12) 0.000 0.0085 (9) 0.000

Geometric parameters (\AA , $^\circ$)

C1—C2 ⁱ	1.373 (2)	C5—O1	1.221 (2)
C1—C2	1.373 (2)	C5—C6	1.493 (3)
C1—H1A	0.9300	C6—C7	1.506 (3)
C2—C3	1.386 (3)	C6—H6A	0.9700
C2—H2A	0.9300	C6—H6B	0.9700
C3—C4	1.417 (2)	C7—N1	1.456 (2)
C3—C5	1.469 (3)	C7—H7A	0.9700
C4—N1	1.376 (3)	C7—H7B	0.9700
C4—C3 ⁱ	1.417 (2)	N1—C7 ⁱ	1.456 (2)
C2 ⁱ —C1—C2	119.4 (2)	C3—C5—C6	116.30 (18)
C2 ⁱ —C1—H1A	120.3	C5—C6—C7	111.92 (16)
C2—C1—H1A	120.3	C5—C6—H6A	109.2
C1—C2—C3	121.6 (2)	C7—C6—H6A	109.2
C1—C2—H2A	119.2	C5—C6—H6B	109.2
C3—C2—H2A	119.2	C7—C6—H6B	109.2
C2—C3—C4	119.56 (18)	H6A—C6—H6B	107.9
C2—C3—C5	119.94 (18)	N1—C7—C6	111.27 (17)
C4—C3—C5	120.29 (16)	N1—C7—H7A	109.4
N1—C4—C3 ⁱ	120.87 (11)	C6—C7—H7A	109.4
N1—C4—C3	120.87 (11)	N1—C7—H7B	109.4
C3 ⁱ —C4—C3	118.2 (2)	C6—C7—H7B	109.4
O1—C5—C3	121.77 (18)	H7A—C7—H7B	108.0
O1—C5—C6	121.85 (19)		

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2A \cdots O1 ⁱⁱ	0.93	2.50	3.372 (3)	157
C6—H6A \cdots O1 ⁱⁱⁱ	0.97	2.52	3.377 (3)	147

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

Fig. 1

