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1,4-Dihexyl-1,2,3,4-tetrahydroquinoxaline-2,3-dione

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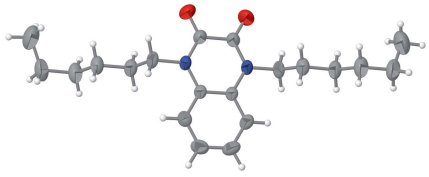
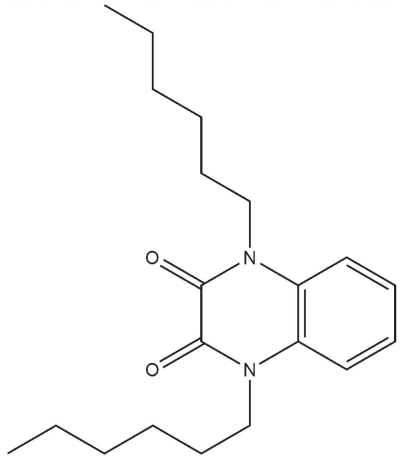
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{20}H_{30}N_2O_2$, has crystallographically imposed C_2 symmetry; the hexyl side chain adopts a *tttg* (*t* = *trans* and *g* = *gauche*) conformation. In the crystal, C—H···O hydrogen bonds link the molecules into chains extending along the *b*-axis direction. These chains pack to form zigzag sheets lying parallel to (101).

3D view



Chemical scheme



Structure description

This work was carried out in a continuation of our previous work on the synthesis and crystal structures of new quinoxaline-2,3-dione derivatives (Ferfra *et al.*, 2001; El Bourakadi *et al.*, 2017*a*,*b*).

The title molecule (Fig. 1) has crystallographically imposed C_2 rotation symmetry. In the bicyclic unit, the dihedral angle between the two rings is 3.64 (7)°. The *n*-hexyl side chain adopts a *tttg* (*t* = *trans* and *g* = *gauche*) conformation, as indicated by the following torsion angles: N1—C5—C6—C7 = 178.85 (13)°, C5—C6—C7—C8 = −179.63 (15)°, C6—C7—C8—C9 = −179.30 (16)°, and C7—C8—C9—C10 = 70.8 (3)°. In the crystal, molecules form chains extending along the *b*-axis direction through C1—H1···O1 hydrogen bonds (Table 1 and Fig. 2). These chains pack to form zigzag sheets lying parallel to (101), possibly aided by weak C5—H5a···π(*Cg*2) interactions [*Cg*2 is the centroid of the aromatic ring at $(-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{3}{2})$], with H···*Cg* = 3.84 Å and C—H···*Cg* = 138° (Fig. 3).

Synthesis and crystallization

A mixture of quinoxaline-2,3-dione (1.0 g, 6.17 mmol), potassium carbonate (1.7 g, 12.33 mmol), bromohexane (1.73 ml, 12.33 mmol) and tetra-*n*-butylammonium bromide

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O1^{ii}	0.93	2.54	3.396 (2)	153

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

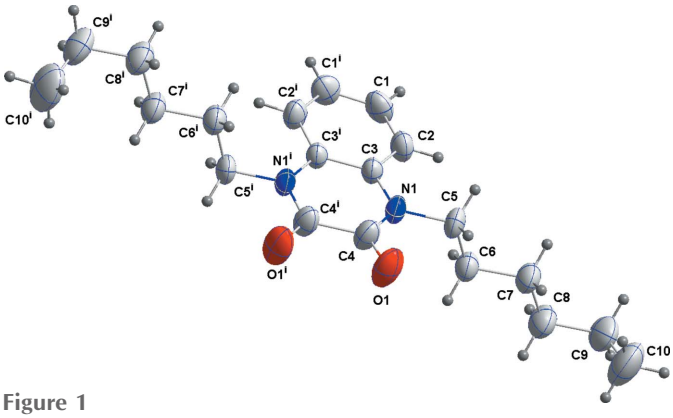


Figure 1
The title molecule with the atom-labeling scheme and 50% probability ellipsoids. [Symmetry code: (i) $-x+1, y, -z+\frac{3}{2}$.]

as a catalyst in *N*,*N*-dimethylformamide (60 ml) was stirred at room temperature for 48 h. After completion of the reaction (monitored by thin-layer chromatography), the solvent was removed under vacuum and the residue was chromatographed on a silica-gel column using hexane and ethyl acetate (80:20 *v*/*v*) as eluent. The compound obtained was recrystallzed from ethanol solution to afford the title compound as colourless blocks.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{20}H_{30}N_2O_2$
M_r	330.46
Crystal system, space group	Monoclinic, *C*2/*c*
Temperature (K)	295
a, b, c (Å)	13.357 (3), 9.209 (2), 16.743 (4)
β (°)	113.277 (3)
V (Å^3)	1891.9 (7)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.08
Crystal size (mm)	0.28 × 0.25 × 0.18
Data collection	
Diffractometer	Bruker SMART APEX CCD
Absorption correction	Multi-scan (*SADABS*; Bruker, 2016)
T_{min}, T_{max}	0.72, 0.99
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	8628, 2323, 1475
R_{int}	0.040
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.666
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.062, 0.196, 1.04
No. of reflections	2323
No. of parameters	110
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.26, −0.22

Computer programs: *APEX3* (Bruker, 2016), *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2014* (Sheldrick, 2015*b*), *DIAMOND* (Brandenburg & Putz, 2012) and *SHELXTL* (Sheldrick, 2008).

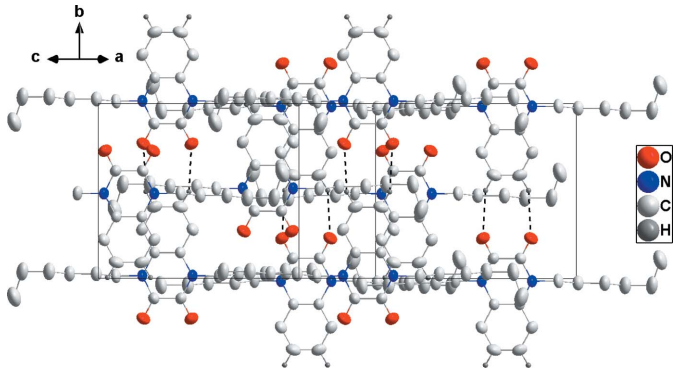


Figure 2
Packing viewed towards (101). C—H···O hydrogen bonds are depicted by dashed lines.

Refinement

Crystal and refinement details are given in Table 2.

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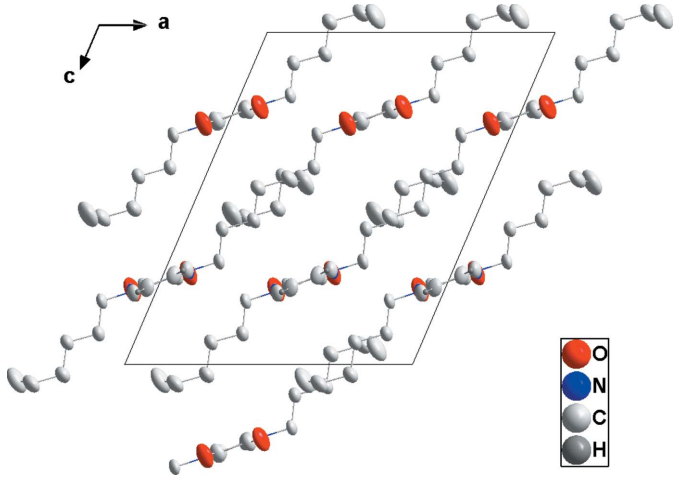

Figure 3
Packing viewed along the *b*-axis direction.

full crystallographic data

IUCrData (2017). **2**, x171019 [https://doi.org/10.1107/S2414314617010197]

1,4-Dihexyl-1,2,3,4-tetrahydroquinoxaline-2,3-dione

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1,4-Dihexyl-1,2,3,4-tetrahydroquinoxaline-2,3-dione

Crystal data

$C_{20}H_{30}N_2O_2$
M_r = 330.46
Monoclinic, $C2/c$
a = 13.357 (3) Å
b = 9.209 (2) Å
c = 16.743 (4) Å
β = 113.277 (3)°
V = 1891.9 (7) Å^3
Z = 4
$F(000)$ = 720
D_x = 1.160 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2136 reflections
θ = 2.7–27.4°
μ = 0.08 mm^{-1}
T = 295 K
Block, colourless
0.28 × 0.25 × 0.18 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3333 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2016)
T_{min} = 0.72, T_{max} = 0.99
8628 measured reflections
2323 independent reflections
1475 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 28.3°, θ_{min} = 2.7°
h = −17→17
k = −12→12
l = −21→22

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.196
S = 1.04
2323 reflections
110 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0925P)^2 + 0.3797P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Special details

Experimental. The diffraction data were collected in three sets of 363 frames (0.5° width in ω) at φ = 0, 120 and 240°. A scan time of 60 sec/frame was used.

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.99 Å). All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.58538 (12)	0.26991 (13)	0.72515 (11)	0.0825 (5)
N1	0.58320 (10)	0.51512 (13)	0.71964 (8)	0.0427 (4)
C1	0.53793 (13)	0.90866 (18)	0.73135 (12)	0.0572 (5)
H1	0.5624	0.9960	0.7177	0.069*
C2	0.57667 (12)	0.77985 (17)	0.71467 (10)	0.0500 (4)
H2	0.6282	0.7807	0.6902	0.060*
C3	0.54071 (10)	0.64763 (15)	0.73341 (9)	0.0387 (4)
C4	0.54659 (13)	0.38519 (17)	0.73476 (11)	0.0523 (4)
C5	0.67419 (13)	0.51352 (18)	0.69052 (11)	0.0485 (4)
H5A	0.7150	0.4241	0.7099	0.058*
H5B	0.7229	0.5937	0.7176	0.058*
C6	0.63673 (13)	0.52534 (19)	0.59270 (11)	0.0515 (4)
H6A	0.5950	0.6139	0.5727	0.062*
H6B	0.5896	0.4439	0.5652	0.062*
C7	0.73340 (14)	0.5263 (2)	0.56635 (11)	0.0561 (5)
H7A	0.7807	0.6071	0.5948	0.067*
H7B	0.7747	0.4375	0.5866	0.067*
C8	0.70034 (17)	0.5390 (2)	0.46884 (13)	0.0676 (6)
H8A	0.6597	0.6283	0.4488	0.081*
H8B	0.6523	0.4589	0.4405	0.081*
C9	0.79629 (19)	0.5382 (3)	0.44171 (14)	0.0778 (6)
H9A	0.7709	0.5675	0.3813	0.093*
H9B	0.8492	0.6095	0.4761	0.093*
C10	0.8516 (3)	0.3937 (3)	0.4521 (2)	0.1177 (10)
H10A	0.7992	0.3214	0.4204	0.177*
H10B	0.8832	0.3679	0.5126	0.177*
H10C	0.9078	0.3991	0.4301	0.177*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1072 (12)	0.0452 (8)	0.1324 (13)	0.0121 (7)	0.0871 (11)	−0.0001 (7)

N1	0.0406 (7)	0.0436 (7)	0.0549 (8)	0.0030 (5)	0.0307 (6)	0.0019 (5)
C1	0.0525 (10)	0.0403 (9)	0.0791 (12)	−0.0077 (7)	0.0264 (8)	0.0037 (8)
C2	0.0438 (9)	0.0476 (9)	0.0668 (10)	−0.0059 (7)	0.0306 (8)	0.0017 (7)
C3	0.0343 (7)	0.0391 (8)	0.0479 (8)	0.0002 (5)	0.0218 (6)	0.0002 (6)
C4	0.0642 (11)	0.0406 (8)	0.0686 (11)	0.0034 (7)	0.0438 (9)	−0.0002 (7)
C5	0.0396 (8)	0.0610 (10)	0.0558 (10)	0.0072 (7)	0.0306 (7)	0.0027 (7)
C6	0.0446 (9)	0.0628 (10)	0.0552 (10)	0.0012 (7)	0.0282 (7)	−0.0001 (7)
C7	0.0522 (10)	0.0712 (11)	0.0555 (10)	−0.0018 (8)	0.0328 (8)	−0.0013 (8)
C8	0.0656 (12)	0.0890 (14)	0.0603 (11)	−0.0026 (10)	0.0377 (10)	0.0003 (9)
C9	0.0850 (15)	0.0988 (16)	0.0699 (12)	−0.0136 (12)	0.0524 (11)	−0.0046 (10)
C10	0.140 (2)	0.116 (2)	0.145 (2)	0.0097 (19)	0.108 (2)	−0.0140 (18)

Geometric parameters (Å, º)

O1—C4	1.2195 (18)	C6—H6A	0.9700
N1—C4	1.3537 (19)	C6—H6B	0.9700
N1—C3	1.4027 (17)	C7—C8	1.518 (2)
N1—C5	1.4777 (17)	C7—H7A	0.9700
C1—C2	1.366 (2)	C7—H7B	0.9700
C1—C1^{i}	1.385 (3)	C8—C9	1.520 (3)
C1—H1	0.9300	C8—H8A	0.9700
C2—C3	1.3895 (19)	C8—H8B	0.9700
C2—H2	0.9300	C9—C10	1.498 (4)
C3—C3^{i}	1.403 (3)	C9—H9A	0.9700
C4—C4^{i}	1.520 (3)	C9—H9B	0.9700
C5—C6	1.515 (2)	C10—H10A	0.9600
C5—H5A	0.9700	C10—H10B	0.9600
C5—H5B	0.9700	C10—H10C	0.9600
C6—C7	1.521 (2)		
C4—N1—C3	122.59 (12)	H6A—C6—H6B	108.0
C4—N1—C5	117.25 (12)	C8—C7—C6	113.14 (15)
C3—N1—C5	120.12 (11)	C8—C7—H7A	109.0
C2—C1—C1^{i}	119.73 (9)	C6—C7—H7A	109.0
C2—C1—H1	120.1	C8—C7—H7B	109.0
C1^{i}—C1—H1	120.1	C6—C7—H7B	109.0
C1—C2—C3	121.46 (14)	H7A—C7—H7B	107.8
C1—C2—H2	119.3	C7—C8—C9	113.57 (17)
C3—C2—H2	119.3	C7—C8—H8A	108.9
C2—C3—N1	121.79 (12)	C9—C8—H8A	108.9
C2—C3—C3^{i}	118.72 (8)	C7—C8—H8B	108.9
N1—C3—C3^{i}	119.49 (7)	C9—C8—H8B	108.9
O1—C4—N1	122.75 (15)	H8A—C8—H8B	107.7
O1—C4—C4^{i}	119.42 (9)	C10—C9—C8	113.86 (19)
N1—C4—C4^{i}	117.83 (8)	C10—C9—H9A	108.8
N1—C5—C6	113.11 (13)	C8—C9—H9A	108.8
N1—C5—H5A	109.0	C10—C9—H9B	108.8
C6—C5—H5A	109.0	C8—C9—H9B	108.8

N1—C5—H5B	109.0	H9A—C9—H9B	107.7
C6—C5—H5B	109.0	C9—C10—H10A	109.5
H5A—C5—H5B	107.8	C9—C10—H10B	109.5
C5—C6—C7	111.00 (14)	H10A—C10—H10B	109.5
C5—C6—H6A	109.4	C9—C10—H10C	109.5
C7—C6—H6A	109.4	H10A—C10—H10C	109.5
C5—C6—H6B	109.4	H10B—C10—H10C	109.5
C7—C6—H6B	109.4		

C1^{i}—C1—C2—C3	0.7 (3)	C3—N1—C4—C4^{i}	1.9 (3)
C1—C2—C3—N1	−177.46 (15)	C5—N1—C4—C4^{i}	179.67 (16)
C1—C2—C3—C3^{i}	2.9 (3)	C4—N1—C5—C6	96.77 (17)
C4—N1—C3—C2	−177.72 (14)	C3—N1—C5—C6	−85.38 (17)
C5—N1—C3—C2	4.5 (2)	N1—C5—C6—C7	178.85 (13)
C4—N1—C3—C3^{i}	2.0 (3)	C5—C6—C7—C8	−179.63 (15)
C5—N1—C3—C3^{i}	−175.77 (15)	C6—C7—C8—C9	−179.30 (16)
C3—N1—C4—O1	−177.92 (16)	C7—C8—C9—C10	70.8 (3)
C5—N1—C4—O1	−0.1 (3)		

Symmetry code: (i) −*x*+1, *y*, −*z*+3/2.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O1^{ii}	0.93	2.54	3.396 (2)	153

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