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6-Chloro-1,4-diethylquinoxaline-2,3(1*H*,4*H*)-dione

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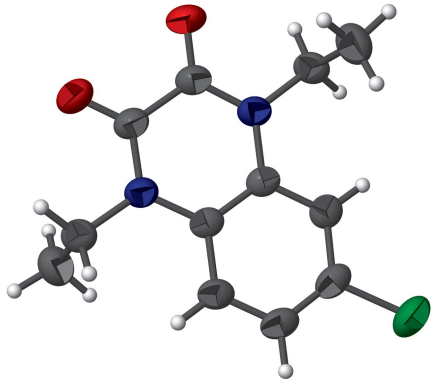
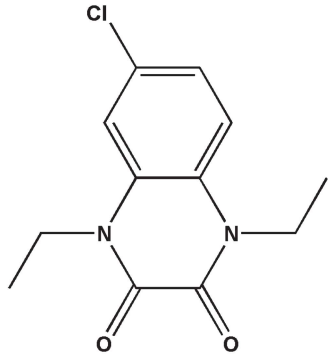
In the title compound, $C_{12}H_{13}ClN_2O_2$, the terminal C atoms of the ethyl groups deviate from the mean plane of the quinoxaline-2,3(1*H*,4*H*)-dione ring (r.m.s. deviation = 0.016 Å) in opposite directions by −1.451 (2) and 1.472 (2) Å. In the crystal, weak C—H···O interactions link the molecules into [100] chains and aromatic π–π stacking interactions [shortest centroid–centroid separation = 3.6631 (9) Å] are also observed.

3D view



Chemical scheme



Structure description

As a continuation of our studies of substituted quinoxaline derivatives (El Janati *et al.*, 2017), we now report the synthesis and structure of the title compound (Fig. 1) prepared by the condensation reaction of 6-chloroquinoxaline-2,3(1*H*,4*H*)-dione with iodoethane.

The terminal C atoms of the ethyl groups deviate from the mean plane of the quinoxaline-2,3(1*H*,4*H*)-dione ring (r.m.s. deviation = 0.016 Å) in opposite directions, by −1.451 (2) and 1.472 (2) Å, for C10 and C12, respectively. In the crystal, weak C—H···O interactions link the molecules into [100] chains with O2 acting as a double acceptor (Table 1, Fig. 2). Aromatic π–π stacking interactions occur between the chains: $Cg2\cdots Cg2(\frac{1}{2}-x,y,-z)$ = 3.6632 (2) Å and $Cg1\cdots Cg2(\frac{1}{2}-x,\frac{1}{2}-y,\frac{1}{2}-z)$ = 3.9508 (2) Å, where *Cg*1 and *Cg*2 are the centroids of the N1/C1/C2/N2/C3/C8 and C3–C8 rings, respectively.

Synthesis and crystallization

To a solution of 6-chloro-1,4-dihydroquinoxaline-2,3-dione 0.30 g (1.53 mmol) in DMF (20 ml), were added 0.52 g (3.84 mmol) of potassium carbonate and 0.1 mmol of tetra-*n*-

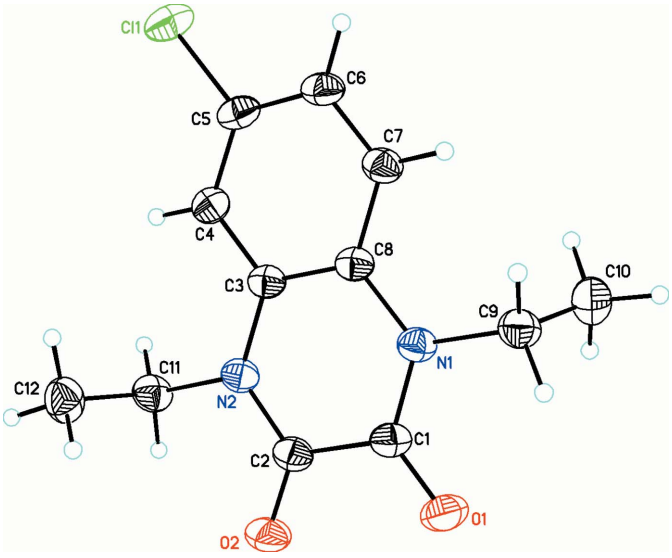


Figure 1
The molecular structure showing 30% probability displacement ellipsoids for the non-H atoms.

butyl ammonium. After 10 min of stirring, 3.85 mmol of iodoethane was added, then the mixture was allowed to stir at room temperature for 36 h. After filtration, the DMF was evaporated under reduced pressure and the residue obtained was dissolved in dichloromethane. The organic phase was dried over Na_2SO_4 and then concentrated. The mixture obtained was chromatographed on a silica gel column [eluent: hexane/ethyl acetate (3/1)]. Crystals were obtained when the solvent was allowed to evaporate.

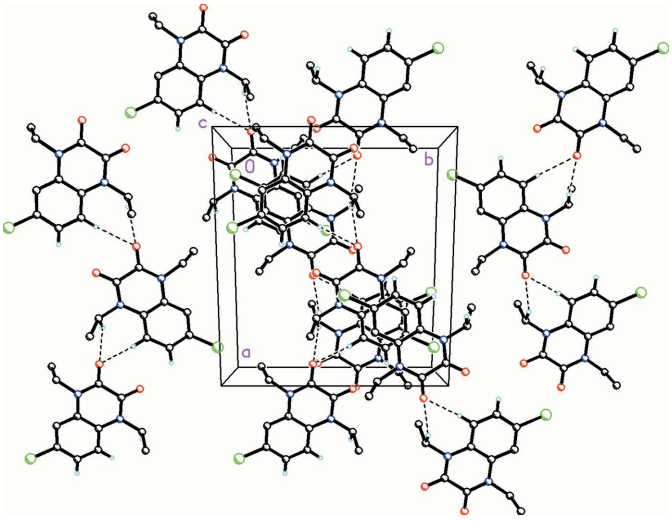


Figure 2
The packing viewed along the *c* axis. Dashed lines indicate weak C—H···O interactions with atom O2 serving as a double acceptor, linking the molecules into [100] chains. H atoms not involved in the packing are omitted for clarity.

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O2^{i}	0.93	2.53	3.440 (2)	167
C9—H9B···O2^{i}	0.97	2.35	3.180 (2)	144

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{13}ClN_2O_2$
M_r	252.69
Crystal system, space group	Monoclinic, *I*2/*a*
Temperature (K)	293
a, *b*, *c* (Å)	14.6454 (8), 12.0415 (5), 15.1149 (9)
β (°)	115.621 (7)
V (Å^3)	2403.5 (3)
Z	8
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.76
Crystal size (mm)	0.22 × 0.18 × 0.12
Data collection	
Diffractometer	Rigaku, Oxford diffraction
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.749, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	4349, 2291, 1944
R_{int}	0.015
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.614
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.045, 0.128, 1.03
No. of reflections	2291
No. of parameters	156
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.23, −0.31

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXT2014* (Sheldrick, 2015*b*), *SHELXL* (Sheldrick, 2015*a*) and *OLEX2* (Dolomanov *et al.*, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements

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full crystallographic data

IUCrData (2017). **2**, x171052 [https://doi.org/10.1107/S2414314617010525]

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Crystal data

$C_{12}H_{13}ClN_2O_2$
M_r = 252.69
Monoclinic, *I*2/*a*
a = 14.6454 (8) Å
b = 12.0415 (5) Å
c = 15.1149 (9) Å
β = 115.621 (7)°
V = 2403.5 (3) Å^3
Z = 8
F(000) = 1056
D_x = 1.397 Mg m^{-3}
Cu *Kα* radiation, *λ* = 1.54184 Å
Cell parameters from 1748 reflections
θ = 3.5–71.4°
μ = 2.76 mm^{-1}
T = 293 K
Irregular, orange
0.22 × 0.18 × 0.12 mm

Data collection

Rigaku, Oxford diffraction diffractometer
Radiation source: fine-focus sealed X-ray tube, Enhance (Cu) X-ray Source
Graphite monochromator
Detector resolution: 16.0416 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.749, T_{max} = 1.000
4349 measured reflections
2291 independent reflections
1944 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 71.3°, θ_{min} = 4.9°
h = −15→17
k = −14→14
l = −16→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.03
2291 reflections
156 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0765P)^2 + 0.5895P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Special details

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. All the H atoms were placed in calculated positions and refined using the riding model with C—H bond lengths of 0.93 Å (CH) or 0.97 Å (CH_2) or 0.96 Å (CH_3). Isotropic displacement parameters for these atoms were set to 1.2 (CH) or 1.5 (CH_3) times U_{eq} of the parent atom. Idealized Me groups were refined as rotating groups.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.13648 (4)	0.04185 (5)	0.36551 (4)	0.0703 (2)
O1	0.45160 (11)	0.58336 (12)	0.38236 (13)	0.0645 (4)
O2	0.56068 (10)	0.39761 (13)	0.40065 (12)	0.0646 (4)
N1	0.31793 (10)	0.48193 (12)	0.37493 (10)	0.0429 (3)
N2	0.42883 (10)	0.28937 (12)	0.38566 (10)	0.0429 (3)
C1	0.41348 (13)	0.49391 (16)	0.38203 (13)	0.0464 (4)
C2	0.47412 (13)	0.38917 (16)	0.39038 (13)	0.0465 (4)
C3	0.33032 (12)	0.28080 (14)	0.37896 (10)	0.0392 (4)
C4	0.28666 (13)	0.17761 (15)	0.37675 (11)	0.0446 (4)
H4	0.3225	0.1128	0.3799	0.054*
C5	0.18971 (14)	0.17216 (16)	0.36987 (12)	0.0484 (4)
C6	0.13456 (13)	0.26574 (18)	0.36620 (12)	0.0496 (4)
H6	0.0696	0.2601	0.3623	0.059*
C7	0.17739 (13)	0.36817 (16)	0.36843 (12)	0.0454 (4)
H7	0.1408	0.4321	0.3661	0.054*
C8	0.27497 (12)	0.37745 (15)	0.37412 (10)	0.0392 (4)
C9	0.25998 (14)	0.58452 (16)	0.36690 (15)	0.0525 (4)
H9A	0.3067	0.6458	0.3947	0.063*
H9B	0.2212	0.5762	0.4047	0.063*
C10	0.18908 (18)	0.6114 (2)	0.26220 (18)	0.0694 (6)
H10A	0.2268	0.6166	0.2239	0.104*
H10B	0.1561	0.6810	0.2599	0.104*
H10C	0.1392	0.5538	0.2361	0.104*
C11	0.49092 (14)	0.18977 (17)	0.39550 (14)	0.0514 (4)
H11A	0.5418	0.2065	0.3725	0.062*
H11B	0.4482	0.1309	0.3547	0.062*
C12	0.54246 (17)	0.15049 (17)	0.50063 (16)	0.0628 (5)
H12A	0.5870	0.0899	0.5055	0.094*
H12B	0.4923	0.1262	0.5213	0.094*
H12C	0.5809	0.2104	0.5418	0.094*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0777 (4)	0.0587 (3)	0.0866 (4)	−0.0233 (2)	0.0469 (3)	−0.0045 (2)
O1	0.0571 (8)	0.0507 (8)	0.0965 (11)	−0.0130 (6)	0.0433 (8)	−0.0084 (7)
O2	0.0429 (7)	0.0682 (9)	0.0922 (11)	−0.0037 (6)	0.0380 (7)	0.0008 (8)
N1	0.0392 (7)	0.0452 (8)	0.0479 (7)	−0.0025 (6)	0.0221 (6)	−0.0040 (6)
N2	0.0403 (7)	0.0482 (8)	0.0447 (7)	−0.0005 (6)	0.0225 (6)	−0.0036 (6)
C1	0.0422 (8)	0.0493 (10)	0.0517 (9)	−0.0068 (7)	0.0242 (7)	−0.0054 (7)
C2	0.0402 (8)	0.0550 (10)	0.0494 (9)	−0.0019 (7)	0.0241 (7)	−0.0016 (7)

C3	0.0392 (7)	0.0481 (9)	0.0334 (7)	−0.0039 (7)	0.0186 (6)	−0.0033 (6)
C4	0.0506 (9)	0.0462 (9)	0.0419 (8)	−0.0024 (7)	0.0245 (7)	−0.0019 (7)
C5	0.0524 (9)	0.0551 (10)	0.0430 (8)	−0.0154 (8)	0.0256 (7)	−0.0030 (7)
C6	0.0434 (8)	0.0630 (11)	0.0488 (9)	−0.0078 (8)	0.0261 (7)	0.0008 (8)
C7	0.0405 (8)	0.0559 (10)	0.0447 (8)	0.0003 (7)	0.0231 (7)	0.0011 (7)
C8	0.0392 (8)	0.0485 (9)	0.0327 (7)	−0.0043 (7)	0.0182 (6)	−0.0026 (6)
C9	0.0465 (9)	0.0466 (9)	0.0669 (11)	−0.0023 (8)	0.0269 (8)	−0.0100 (8)
C10	0.0692 (13)	0.0590 (12)	0.0808 (14)	0.0115 (10)	0.0331 (11)	0.0122 (11)
C11	0.0481 (9)	0.0526 (10)	0.0605 (10)	0.0039 (8)	0.0302 (8)	−0.0069 (8)
C12	0.0639 (12)	0.0498 (10)	0.0673 (12)	0.0076 (9)	0.0214 (10)	−0.0022 (9)

Geometric parameters (Å, º)

Cl1—C5	1.7407 (19)	C6—C7	1.378 (3)
O1—C1	1.212 (2)	C7—H7	0.9300
O2—C2	1.213 (2)	C7—C8	1.399 (2)
N1—C1	1.364 (2)	C9—H9A	0.9700
N1—C8	1.404 (2)	C9—H9B	0.9700
N1—C9	1.474 (2)	C9—C10	1.505 (3)
N2—C2	1.359 (2)	C10—H10A	0.9600
N2—C3	1.406 (2)	C10—H10B	0.9600
N2—C11	1.473 (2)	C10—H10C	0.9600
C1—C2	1.516 (3)	C11—H11A	0.9700
C3—C4	1.391 (2)	C11—H11B	0.9700
C3—C8	1.402 (2)	C11—C12	1.510 (3)
C4—H4	0.9300	C12—H12A	0.9600
C4—C5	1.380 (2)	C12—H12B	0.9600
C5—C6	1.373 (3)	C12—H12C	0.9600
C6—H6	0.9300		
C1—N1—C8	122.40 (15)	C3—C8—N1	119.77 (14)
C1—N1—C9	116.90 (15)	C7—C8—N1	120.93 (16)
C8—N1—C9	120.69 (14)	C7—C8—C3	119.30 (16)
C2—N2—C3	122.07 (15)	N1—C9—H9A	109.2
C2—N2—C11	116.65 (14)	N1—C9—H9B	109.2
C3—N2—C11	121.12 (14)	N1—C9—C10	112.20 (16)
O1—C1—N1	123.32 (18)	H9A—C9—H9B	107.9
O1—C1—C2	119.13 (16)	C10—C9—H9A	109.2
N1—C1—C2	117.55 (16)	C10—C9—H9B	109.2
O2—C2—N2	122.68 (18)	C9—C10—H10A	109.5
O2—C2—C1	118.88 (17)	C9—C10—H10B	109.5
N2—C2—C1	118.43 (15)	C9—C10—H10C	109.5
C4—C3—N2	120.94 (15)	H10A—C10—H10B	109.5
C4—C3—C8	119.39 (15)	H10A—C10—H10C	109.5
C8—C3—N2	119.67 (15)	H10B—C10—H10C	109.5
C3—C4—H4	120.3	N2—C11—H11A	109.3
C5—C4—C3	119.45 (17)	N2—C11—H11B	109.3
C5—C4—H4	120.3	N2—C11—C12	111.54 (15)

C4—C5—Cl1	118.37 (15)	H11A—C11—H11B	108.0
C6—C5—Cl1	119.48 (14)	C12—C11—H11A	109.3
C6—C5—C4	122.15 (17)	C12—C11—H11B	109.3
C5—C6—H6	120.7	C11—C12—H12A	109.5
C5—C6—C7	118.69 (16)	C11—C12—H12B	109.5
C7—C6—H6	120.7	C11—C12—H12C	109.5
C6—C7—H7	119.5	H12A—C12—H12B	109.5
C6—C7—C8	121.02 (17)	H12A—C12—H12C	109.5
C8—C7—H7	119.5	H12B—C12—H12C	109.5
Cl1—C5—C6—C7	−179.16 (13)	C4—C3—C8—N1	−179.12 (14)
O1—C1—C2—O2	2.5 (3)	C4—C3—C8—C7	0.8 (2)
O1—C1—C2—N2	−177.21 (17)	C4—C5—C6—C7	0.7 (3)
N1—C1—C2—O2	−177.15 (16)	C5—C6—C7—C8	0.1 (2)
N1—C1—C2—N2	3.1 (2)	C6—C7—C8—N1	179.05 (15)
N2—C3—C4—C5	−179.83 (13)	C6—C7—C8—C3	−0.9 (2)
N2—C3—C8—N1	0.7 (2)	C8—N1—C1—O1	179.63 (17)
N2—C3—C8—C7	−179.38 (13)	C8—N1—C1—C2	−0.7 (2)
C1—N1—C8—C3	−1.2 (2)	C8—N1—C9—C10	−81.9 (2)
C1—N1—C8—C7	178.90 (14)	C8—C3—C4—C5	0.0 (2)
C1—N1—C9—C10	97.43 (19)	C9—N1—C1—O1	0.3 (3)
C2—N2—C3—C4	−178.34 (15)	C9—N1—C1—C2	179.98 (15)
C2—N2—C3—C8	1.8 (2)	C9—N1—C8—C3	178.14 (14)
C2—N2—C11—C12	93.20 (19)	C9—N1—C8—C7	−1.8 (2)
C3—N2—C2—O2	176.57 (16)	C11—N2—C2—O2	1.1 (3)
C3—N2—C2—C1	−3.7 (2)	C11—N2—C2—C1	−179.15 (15)
C3—N2—C11—C12	−82.3 (2)	C11—N2—C3—C4	−3.1 (2)
C3—C4—C5—Cl1	179.10 (12)	C11—N2—C3—C8	177.12 (14)
C3—C4—C5—C6	−0.8 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C7—H7···O2^{i}	0.93	2.53	3.440 (2)	167
C9—H9*B*···O2^{i}	0.97	2.35	3.180 (2)	144

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