

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

2-Isopropyl-3-methylquinoxaline 1,4-dioxide

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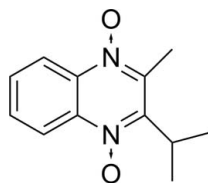
Received 26 May 2010; accepted 18 June 2010

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.057; wR factor = 0.203; data-to-parameter ratio = 15.1.

In the title compound, $\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2$, the quinoxaline ring system and the C atoms of the methylene and methyl substituents lie on a mirror plane. The crystal packing is stabilized by weak $\pi-\pi$ interactions [centroid-centroid distance = 3.680 (7) Å].

Related literature

For the preparation, see: Issidorides & Haddadin (1966). For the biological activity of quinoxaline di-*N*-oxide compounds, see: Amin *et al.* (2006); Edwards *et al.* (1975); Glazer & Chappel (1982).



Experimental

Crystal data

$\text{C}_{12}\text{H}_{14}\text{N}_2\text{O}_2$

$M_r = 218.25$

Orthorhombic, $Pnma$

$a = 13.3879$ (10) Å

$b = 6.8462$ (6) Å

$c = 11.8861$ (9) Å

$V = 1089.44$ (15) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 0.09$ mm⁻¹

$T = 296$ K

$0.29 \times 0.27 \times 0.26$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Absorption correction: multi-scan (SADABS; Bruker, 2005)

$T_{\min} = 0.651$, $T_{\max} = 0.745$

10376 measured reflections

1446 independent reflections

1062 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.203$

$S = 1.17$

1446 reflections

96 parameters

1 restraint

H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.38$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.35$ e Å⁻³

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); data reduction: SAINT; program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: WinGX (Farrugia, 1999).

This work was supported by the NSFC (grant No. 20625307), the National Basic Research Program of China (973 Program, 2009CB930103) and the Graduate Independent Innovation Foundation of Shandong University (GIIFSDU).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JH2164).

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supplementary materials

Acta Cryst. (2010). E66, o1906 [doi:10.1107/S1600536810023706]

2-Isopropyl-3-methylquinoxaline 1,4-dioxide

J.-Y. Li, T. Sun, A.-Y. Hao, H. Qiao and F. Xin

Comment

In recent years, the compounds of quinoxaline di-N-oxide have been used as important and widely-used drugs for sterilization and growth-promoting of animals. Quinoxaline di-N-oxide also has pharmacological properties usable as intermediates for producing plant protection agents. The research in the crystal of quinoxaline di-N-oxide has great meaning to pharmacology. The title compound 2-isopropyl-3-methylquinoxaline 1,4-dioxide was obtained by Beirut Reaction: benzofurazan-N-oxides reacted with cyclohexanone catalysed by triethylamine without any other solvent.

Experimental

Yellow crystals were obtained by slow evaporation of the solvents from solutions of the title compound in methanol. ^1H NMR (400 MHz, DMSO- d_6): δ 8.43 (2H, d, $J = 3.5$ Hz, Ar—H), 7.88 (2H, d, $J = 3.2$ Hz, Ar—H), 2.93 (4H, s, CH_2), 1.83 (4H, s, CH_2); IRvmax (KBr, cm^{-1}): 3455, 3125, 2943, 2866, 1987, 1953, 1737, 1605, 1516, 1441, 1422, 1400, 1357, 1315, 1277, 1246, 1125, 1089, 1016, 979, 933, 904, 842, 824, 776, 694, 668, 640, 613, 557, 528, 436; Calcd for $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_2$: C, 66.65; H, 5.59; N, 12.96. Found: C, 66.34; H, 5.32; N, 12.90; ESIMS calcd for $\text{C}_{12}\text{H}_{12}\text{N}_2\text{O}_2\text{H}^+$ m/z 217.24, found m/z 217.20.

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 $F^2 > 2\sigma(F^2)$ 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.

Figures

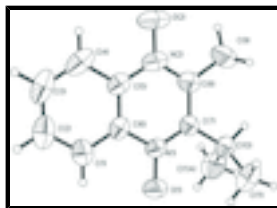


Fig. 1. Ellipsoid plot.

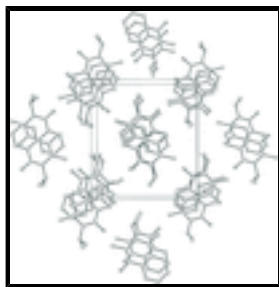


Fig. 2. Packing diagram.

2-Isopropyl-3-methylquinoxaline 1,4-dioxide

Crystal data

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$M_r = 218.25$

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$b = 6.8462$ (6) Å

$c = 11.8861$ (9) Å

$V = 1089.44$ (15) Å³

$Z = 4$

$F(000) = 464$

$D_x = 1.331$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4638 reflections

$\theta = 3.0$ – 27.9°

$\mu = 0.09$ mm⁻¹

$T = 296$ K

Block, colourless

$0.29 \times 0.27 \times 0.26$ mm

Data collection

Bruker APEXII CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

φ and ω scans

Absorption correction: multi-scan (*SADABS*; Bruker, 2005)

$T_{\min} = 0.651$, $T_{\max} = 0.745$

10376 measured reflections

1446 independent reflections

1062 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.019$

$\theta_{\max} = 28.6^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -18 \rightarrow 17$

$k = -9 \rightarrow 9$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.057$

$wR(F^2) = 0.203$

$S = 1.17$

1446 reflections

96 parameters

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0863P)^2 + 0.4481P]$

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

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.38$ e Å⁻³

$\Delta\rho_{\min} = -0.35$ e Å⁻³

1 restraint

Extinction correction: *SHELXL97* (Sheldrick, 2008),

$$F_c^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$$

Primary atom site location: structure-invariant direct methods

Extinction coefficient: 0.011 (4)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.9509 (2)	0.2500	1.1853 (3)	0.0603 (8)
H11	0.9026	0.2500	1.2416	0.072*
C2	1.0499 (3)	0.2500	1.2128 (4)	0.0731 (10)
H2	1.0689	0.2500	1.2880	0.088*
C3	1.1212 (2)	0.2500	1.1306 (4)	0.0723 (11)
H3	1.1883	0.2500	1.1510	0.087*
C4	1.0962 (2)	0.2500	1.0186 (4)	0.0641 (9)
H4	1.1454	0.2500	0.9633	0.077*
C5	0.99369 (19)	0.2500	0.9893 (2)	0.0449 (6)
C6	0.92245 (18)	0.2500	1.0730 (2)	0.0440 (6)
C7	0.79331 (18)	0.2500	0.9352 (2)	0.0427 (6)
C8	0.8664 (2)	0.2500	0.8500 (2)	0.0481 (6)
C9	0.8419 (3)	0.2500	0.7287 (3)	0.0705 (9)
H9A	0.9023	0.2500	0.6845	0.080*
H9B	0.8034	0.1355	0.7099	0.080*
C10	0.6836 (2)	0.2500	0.9063 (3)	0.0551 (7)
H10	0.6798	0.2500	0.8240	0.066*
C11	0.63084 (17)	0.0655 (4)	0.9452 (2)	0.0777 (8)
H11A	0.6657	-0.0468	0.9169	0.117*
H11B	0.5636	0.0652	0.9173	0.117*
H11C	0.6299	0.0615	1.0259	0.117*
N1	0.82094 (15)	0.2500	1.04424 (19)	0.0445 (6)
N2	0.96482 (17)	0.2500	0.8772 (2)	0.0513 (6)
O1	0.75580 (15)	0.2500	1.12490 (18)	0.0681 (7)
O2	1.03357 (18)	0.2500	0.8002 (2)	0.0809 (8)

Atomic displacement parameters (\AA^2)

U^{11} U^{22} U^{33} U^{12} U^{13} U^{23}

supplementary materials

C1	0.0617 (17)	0.0652 (18)	0.0539 (18)	0.000	-0.0125 (14)	0.000
C2	0.067 (2)	0.066 (2)	0.085 (3)	0.000	-0.0309 (19)	0.000
C3	0.0494 (16)	0.0531 (16)	0.114 (3)	0.000	-0.0284 (19)	0.000
C4	0.0403 (13)	0.0433 (14)	0.109 (3)	0.000	0.0089 (16)	0.000
C5	0.0405 (12)	0.0326 (11)	0.0616 (17)	0.000	0.0047 (11)	0.000
C6	0.0389 (12)	0.0377 (12)	0.0553 (15)	0.000	-0.0038 (11)	0.000
C7	0.0408 (12)	0.0438 (12)	0.0434 (14)	0.000	-0.0006 (10)	0.000
C8	0.0557 (14)	0.0409 (12)	0.0477 (15)	0.000	0.0076 (12)	0.000
C9	0.084 (2)	0.081 (2)	0.0466 (17)	0.000	0.0075 (16)	0.000
C10	0.0409 (13)	0.0706 (18)	0.0537 (17)	0.000	-0.0040 (12)	0.000
C11	0.0525 (11)	0.0818 (17)	0.099 (2)	-0.0170 (11)	-0.0097 (12)	0.0027 (15)
N1	0.0368 (10)	0.0509 (12)	0.0457 (12)	0.000	0.0050 (9)	0.000
N2	0.0485 (12)	0.0406 (11)	0.0648 (15)	0.000	0.0196 (11)	0.000
O1	0.0461 (10)	0.1067 (18)	0.0516 (12)	0.000	0.0126 (9)	0.000
O2	0.0716 (15)	0.0876 (17)	0.0836 (18)	0.000	0.0422 (13)	0.000

Geometric parameters (Å, °)

C1—C2	1.365 (4)	C7—C10	1.508 (4)
C1—C6	1.389 (4)	C8—N2	1.357 (4)
C1—H11	0.9300	C8—C9	1.479 (4)
C2—C3	1.366 (6)	C9—H9A	0.964 (4)
C2—H2	0.9300	C9—H9B	0.964 (2)
C3—C4	1.373 (5)	C10—C11 ⁱ	1.519 (3)
C3—H3	0.9300	C10—C11	1.519 (3)
C4—C5	1.416 (4)	C10—H10	0.9800
C4—H4	0.9300	C11—H11A	0.9600
C5—C6	1.378 (4)	C11—H11B	0.9600
C5—N2	1.387 (4)	C11—H11C	0.9600
C6—N1	1.401 (3)	N1—O1	1.296 (3)
C7—N1	1.348 (3)	N2—O2	1.298 (3)
C7—C8	1.409 (4)		
C2—C1—C6	119.7 (3)	C7—C8—C9	123.1 (3)
C2—C1—H11	120.1	C8—C9—H9A	110.2 (3)
C6—C1—H11	120.1	C8—C9—H9B	110.1 (2)
C1—C2—C3	120.5 (3)	H9A—C9—H9B	108.8 (2)
C1—C2—H2	119.7	C7—C10—C11 ⁱ	112.56 (16)
C3—C2—H2	119.7	C7—C10—C11	112.56 (16)
C2—C3—C4	121.5 (3)	C11 ⁱ —C10—C11	112.5 (3)
C2—C3—H3	119.2	C7—C10—H10	106.2
C4—C3—H3	119.2	C11 ⁱ —C10—H10	106.2
C3—C4—C5	118.4 (3)	C11—C10—H10	106.2
C3—C4—H4	120.8	C10—C11—H11A	109.5
C5—C4—H4	120.8	C10—C11—H11B	109.5
C6—C5—N2	120.0 (2)	H11A—C11—H11B	109.5
C6—C5—C4	119.6 (3)	C10—C11—H11C	109.5
N2—C5—C4	120.4 (3)	H11A—C11—H11C	109.5
C5—C6—C1	120.3 (3)	H11B—C11—H11C	109.5

C5—C6—N1	119.7 (3)	O1—N1—C7	121.8 (2)
C1—C6—N1	120.0 (3)	O1—N1—C6	118.2 (2)
N1—C7—C8	120.0 (2)	C7—N1—C6	120.0 (2)
N1—C7—C10	119.1 (2)	O2—N2—C8	121.4 (3)
C8—C7—C10	120.9 (3)	O2—N2—C5	118.7 (2)
N2—C8—C7	120.2 (3)	C8—N2—C5	120.0 (2)
N2—C8—C9	116.6 (3)		
C6—C1—C2—C3	0.000 (2)	C8—C7—C10—C11	115.8 (2)
C1—C2—C3—C4	0.000 (2)	C8—C7—N1—O1	180.0
C2—C3—C4—C5	0.000 (1)	C10—C7—N1—O1	0.0
C3—C4—C5—C6	0.000 (1)	C8—C7—N1—C6	0.0
C3—C4—C5—N2	180.000 (1)	C10—C7—N1—C6	180.0
N2—C5—C6—C1	180.0	C5—C6—N1—O1	180.0
C4—C5—C6—C1	0.000 (1)	C1—C6—N1—O1	0.0
N2—C5—C6—N1	0.0	C5—C6—N1—C7	0.0
C4—C5—C6—N1	180.0	C1—C6—N1—C7	180.0
C2—C1—C6—C5	0.000 (1)	C7—C8—N2—O2	180.0
C2—C1—C6—N1	180.0	C9—C8—N2—O2	0.0
N1—C7—C8—N2	0.0	C7—C8—N2—C5	0.0
C10—C7—C8—N2	180.0	C9—C8—N2—C5	180.0
N1—C7—C8—C9	180.0	C6—C5—N2—O2	180.0
C10—C7—C8—C9	0.0	C4—C5—N2—O2	0.0
N1—C7—C10—C11 ⁱ	64.2 (2)	C6—C5—N2—C8	0.0
C8—C7—C10—C11 ⁱ	-115.8 (2)	C4—C5—N2—C8	180.0
N1—C7—C10—C11	-64.2 (2)		

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

Fig. 1

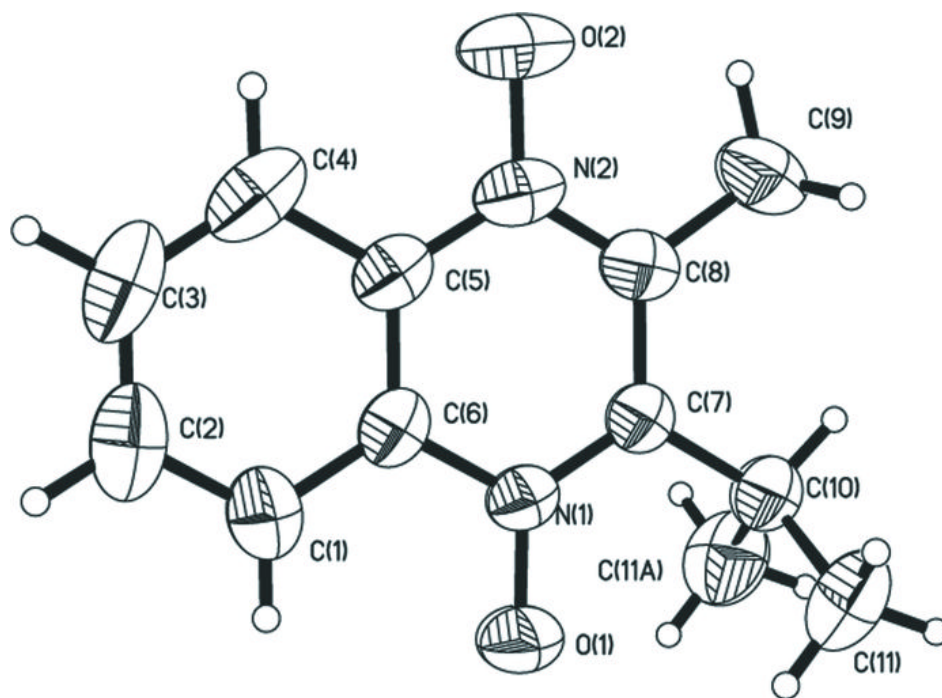


Fig. 2

