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3-Methylquinoxaline-2-carboxylic acid 4-oxide monohydrate

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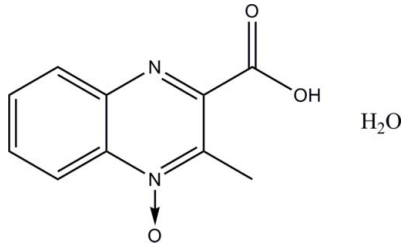
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.008 Å; R factor = 0.098; wR factor = 0.256; data-to-parameter ratio = 10.2.

In the crystal structure of the title compound, $C_{10}H_8N_2O_3\cdot H_2O$, molecules are linked *via* intermolecular O—H···O and O—H···N hydrogen bonds into a two-dimensional network.

Related literature

For the synthesis of the starting material, see: Robertson & Kasublck (1973). For the synthesis of the title compound, see: Dirlam & McFarland (1977).



Experimental

Crystal data

$C_{10}H_8N_2O_3\cdot H_2O$
M_r = 222.20
Monoclinic, $P2_1/c$
a = 6.0526 (13) Å
b = 18.068 (4) Å
c = 8.9195 (19) Å
β = 98.520 (15)°
V = 964.7 (4) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 1.02 mm^{-1}
T = 173 K
0.20 × 0.20 × 0.04 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: numerical (*ABSCOR*; Higashi, 1995)
T_{min} = 0.822, T_{max} = 0.960
6140 measured reflections
1568 independent reflections
900 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.098
$wR(F^2)$ = 0.256
S = 1.10
1568 reflections
154 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1WB···O1^{i}	0.85 (4)	1.97 (2)	2.794 (5)	164 (5)
O1W—H1WA···N2^{ii}	0.86 (4)	2.14 (2)	2.968 (5)	163 (5)
O3—H3···O1W	0.84	1.76	2.574 (5)	162

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

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5056).

References

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supporting information

Acta Cryst. (2010). E**66**, o1801 [doi:10.1107/S160053681002266X]

3-Methylquinoxaline-2-carboxylic acid 4-oxide monohydrate

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S1. Comment

The molecular structure of the title compound is shown in Fig. 1. In the crystal structure, molecules are linked via intermolecular O-H···O hydrogen bonds into a two-dimensional network.

S2. Experimental

Following the procedure of Dirlam & McFarland (1977) ethyl-3-methyl-2-quinoxalinecarboxylate-1,4-dioxide (2.0 g, 8 mmol) (Robertson & Kasublck, 1973) was dissolved in 1-propanol (20 ml), trimethyl phosphate (2.0 g, 16 mmol) was added dropwise to the solution. The reaction mixture was heated under reflux for 2.5 h, and evaporated to dryness. The residue was recrystallized from ether-hexane (1:1) to yeild 1.6 g (80%) ethyl 2-methyl-3-quinoxalinecarboxylate-1-oxide. Ethyl 2-methyl-3-quinoxalinecarboxylate-1-oxide (5 g, 22 mmol) was suspended in aqueous 0.5*M* sodium hydroxide solution (50 mL), and stirred for 2 h. Then used concentrated hydrochloric acid to adjust the PH=2. The white solid was collected and recrystallized from water to give 4.0 g (80%) of the the title compound.

S3. Refinement

All H atoms (except for those bonded to the solvent water) were placed in calculated positions C-H = 0.95-0.98Å; O-H = 0.86Å and refined in a riding-model approximation with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl},O). The H atoms of the solvent water molecule were located in a difference Fourier and there positions were refined with restraints and with U_{iso}(H) = 1.5U_{eq}(O).

The crystals of the title compound were of low quality and the data used has resulted in a crystal structure which has lower than normal precision. The precision of the data however is adequate to describe the nature of the hydrogen bonding.

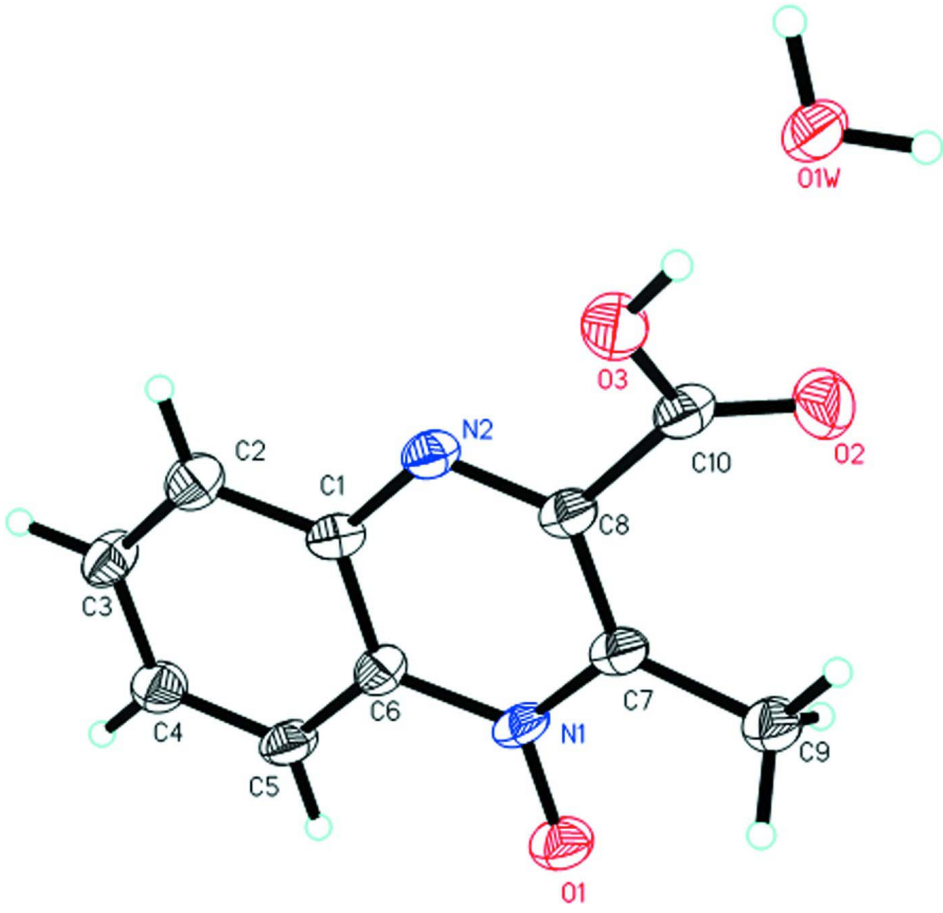

Figure 1

The molecular structure of the title compound, showing the labelling scheme. Displacement ellipsoids are drawn at the 30% probability level for all non-H atoms.

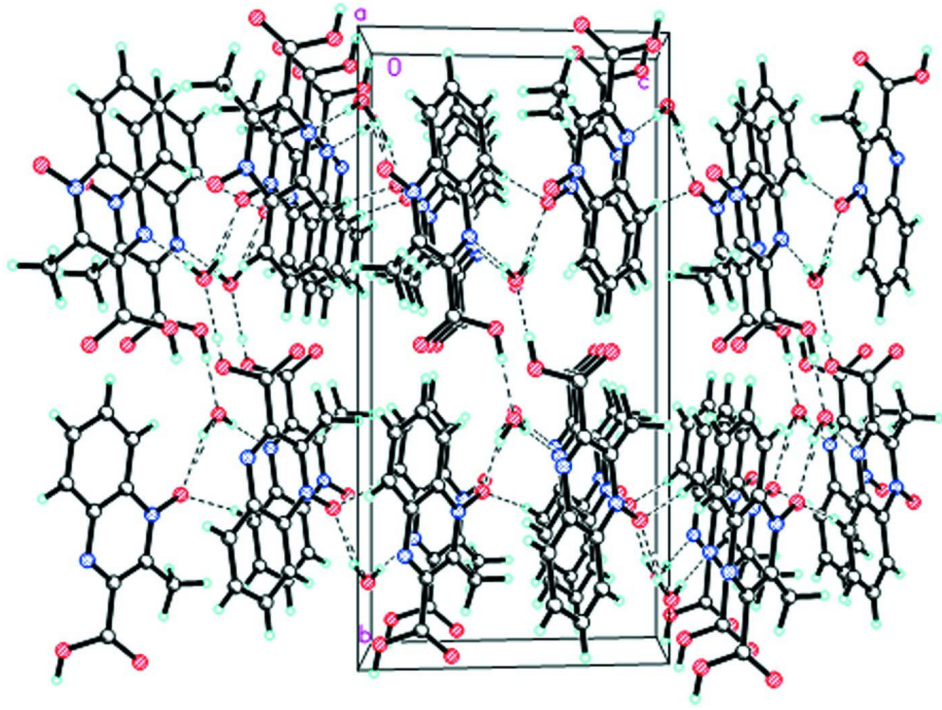

Figure 2

Part of the crystal structure of the title compound viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

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Crystal data

$C_{10}H_8N_2O_3·H_2O$
$M_r = 222.20$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 6.0526$ (13) Å
$b = 18.068$ (4) Å
$c = 8.9195$ (19) Å
$\beta = 98.520$ (15)°
$V = 964.7$ (4) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.530$ Mg m^{-3}
Cu $K\alpha$ radiation, $\lambda = 1.54186$ Å
Cell parameters from 426 reflections
$\theta = 3.1$–$68.1°$
$\mu = 1.02$ mm^{-1}
$T = 173$ K
Plate, yellow
0.20 × 0.20 × 0.04 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans at fixed $\chi = 45°$
Absorption correction: numerical (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.822$, $T_{max} = 0.960$
6140 measured reflections
1568 independent reflections
900 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 63.7°$, $\theta_{min} = 4.9°$
$h = -7 \rightarrow 7$
$k = -20 \rightarrow 20$
$l = -10 \rightarrow 9$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.098$
$wR(F^2) = 0.256$
$S = 1.10$
1568 reflections
154 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1186P)^2 + 0.0706P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.38$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.009 (2)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1W	0.2035 (6)	0.6094 (2)	0.4980 (5)	0.0458 (12)
O1	0.6172 (6)	0.2378 (2)	0.1107 (4)	0.0546 (12)
O2	0.3088 (6)	0.4949 (2)	0.2089 (5)	0.0614 (13)
O3	0.2260 (7)	0.4704 (2)	0.4389 (4)	0.0532 (12)
H3	0.2018	0.5162	0.4401	0.080*
N1	0.4745 (7)	0.2663 (3)	0.1891 (5)	0.0393 (12)
N2	0.1661 (7)	0.3286 (2)	0.3563 (5)	0.0389 (12)
C1	0.1772 (8)	0.2529 (3)	0.3378 (6)	0.0403 (14)
C2	0.0313 (9)	0.2070 (3)	0.4034 (6)	0.0461 (15)
H2	−0.0732	0.2280	0.4609	0.055*
C3	0.0398 (9)	0.1315 (3)	0.3845 (6)	0.0474 (16)
H3A	−0.0604	0.1004	0.4277	0.057*
C4	0.1961 (9)	0.1006 (3)	0.3014 (6)	0.0490 (16)
H4	0.2012	0.0483	0.2900	0.059*
C5	0.3405 (9)	0.1436 (3)	0.2369 (6)	0.0451 (16)
H5	0.4457	0.1219	0.1810	0.054*
C6	0.3311 (8)	0.2207 (3)	0.2547 (6)	0.0387 (14)
C7	0.4655 (9)	0.3408 (3)	0.2072 (6)	0.0402 (15)
C8	0.3058 (9)	0.3687 (3)	0.2920 (6)	0.0383 (14)
C9	0.6295 (9)	0.3835 (3)	0.1352 (6)	0.0482 (16)
H9B	0.7788	0.3625	0.1647	0.072*
H9C	0.6287	0.4352	0.1684	0.072*
H9A	0.5896	0.3812	0.0247	0.072*

C10	0.2818 (9)	0.4516 (3)	0.3070 (7)	0.0434 (15)
H1WB	0.261 (8)	0.643 (2)	0.450 (6)	0.065*
H1WA	0.080 (5)	0.626 (3)	0.522 (6)	0.065*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1W	0.041 (2)	0.044 (3)	0.056 (3)	−0.003 (2)	0.0210 (19)	−0.003 (2)
O1	0.049 (2)	0.054 (3)	0.065 (3)	0.007 (2)	0.021 (2)	−0.005 (2)
O2	0.081 (3)	0.050 (3)	0.059 (3)	0.009 (2)	0.030 (2)	0.008 (2)
O3	0.065 (3)	0.043 (3)	0.056 (3)	0.004 (2)	0.021 (2)	−0.002 (2)
N1	0.032 (2)	0.052 (3)	0.037 (3)	0.005 (2)	0.016 (2)	−0.004 (2)
N2	0.034 (2)	0.040 (3)	0.045 (3)	0.001 (2)	0.013 (2)	−0.006 (2)
C1	0.031 (3)	0.043 (4)	0.048 (3)	0.004 (3)	0.010 (3)	−0.004 (3)
C2	0.044 (3)	0.051 (4)	0.045 (3)	−0.005 (3)	0.010 (3)	−0.007 (3)
C3	0.045 (4)	0.046 (4)	0.056 (4)	−0.011 (3)	0.021 (3)	−0.008 (3)
C4	0.050 (4)	0.040 (4)	0.060 (4)	0.004 (3)	0.018 (3)	−0.003 (3)
C5	0.037 (3)	0.048 (4)	0.053 (4)	0.006 (3)	0.018 (3)	−0.004 (3)
C6	0.037 (3)	0.044 (4)	0.036 (3)	0.000 (3)	0.012 (3)	−0.002 (3)
C7	0.033 (3)	0.049 (4)	0.040 (3)	0.004 (3)	0.008 (2)	−0.003 (3)
C8	0.034 (3)	0.046 (4)	0.036 (3)	0.004 (3)	0.008 (2)	−0.002 (3)
C9	0.045 (4)	0.047 (4)	0.056 (4)	0.003 (3)	0.016 (3)	−0.002 (3)
C10	0.038 (3)	0.047 (4)	0.048 (4)	0.000 (3)	0.015 (3)	−0.006 (3)

Geometric parameters (Å, º)

O1W—H1WB	0.85 (4)	C2—H2	0.9500
O1W—H1WA	0.86 (4)	C3—C4	1.401 (7)
O1—N1	1.296 (5)	C3—H3A	0.9500
O2—C10	1.202 (6)	C4—C5	1.359 (7)
O3—C10	1.316 (6)	C4—H4	0.9500
O3—H3	0.8400	C5—C6	1.404 (7)
N1—C7	1.357 (7)	C5—H5	0.9500
N1—C6	1.388 (6)	C7—C8	1.406 (7)
N2—C8	1.309 (6)	C7—C9	1.477 (7)
N2—C1	1.380 (6)	C8—C10	1.514 (8)
C1—C6	1.401 (6)	C9—H9B	0.9800
C1—C2	1.401 (7)	C9—H9C	0.9800
C2—C3	1.377 (6)	C9—H9A	0.9800
H1WB—O1W—H1WA	108 (4)	C6—C5—H5	120.6
C10—O3—H3	109.5	N1—C6—C1	118.8 (5)
O1—N1—C7	120.0 (5)	N1—C6—C5	120.3 (5)
O1—N1—C6	120.0 (5)	C1—C6—C5	120.9 (5)
C7—N1—C6	120.1 (4)	N1—C7—C8	117.5 (5)
C8—N2—C1	116.8 (4)	N1—C7—C9	115.2 (5)
N2—C1—C6	121.6 (5)	C8—C7—C9	127.3 (5)
N2—C1—C2	119.4 (5)	N2—C8—C7	125.2 (5)

C6—C1—C2	119.0 (5)	N2—C8—C10	115.6 (5)
C3—C2—C1	119.9 (5)	C7—C8—C10	119.0 (5)
C3—C2—H2	120.1	C7—C9—H9B	109.5
C1—C2—H2	120.1	C7—C9—H9C	109.5
C2—C3—C4	120.0 (5)	H9B—C9—H9C	109.5
C2—C3—H3A	120.0	C7—C9—H9A	109.5
C4—C3—H3A	120.0	H9B—C9—H9A	109.5
C5—C4—C3	121.5 (5)	H9C—C9—H9A	109.5
C5—C4—H4	119.3	O2—C10—O3	124.3 (6)
C3—C4—H4	119.3	O2—C10—C8	123.7 (5)
C4—C5—C6	118.7 (5)	O3—C10—C8	112.0 (5)
C4—C5—H5	120.6		

C8—N2—C1—C6	0.2 (7)	C4—C5—C6—C1	0.4 (8)
C8—N2—C1—C2	−179.8 (5)	O1—N1—C7—C8	179.4 (4)
N2—C1—C2—C3	179.4 (4)	C6—N1—C7—C8	−0.7 (7)
C6—C1—C2—C3	−0.6 (8)	O1—N1—C7—C9	−1.3 (7)
C1—C2—C3—C4	0.9 (8)	C6—N1—C7—C9	178.7 (4)
C2—C3—C4—C5	−0.6 (8)	C1—N2—C8—C7	−0.4 (8)
C3—C4—C5—C6	0.0 (8)	C1—N2—C8—C10	176.9 (4)
O1—N1—C6—C1	−179.5 (4)	N1—C7—C8—N2	0.6 (8)
C7—N1—C6—C1	0.6 (7)	C9—C7—C8—N2	−178.7 (5)
O1—N1—C6—C5	0.2 (7)	N1—C7—C8—C10	−176.5 (4)
C7—N1—C6—C5	−179.7 (4)	C9—C7—C8—C10	4.2 (8)
N2—C1—C6—N1	−0.3 (7)	N2—C8—C10—O2	−143.8 (5)
C2—C1—C6—N1	179.6 (5)	C7—C8—C10—O2	33.6 (8)
N2—C1—C6—C5	180.0 (5)	N2—C8—C10—O3	35.6 (6)
C2—C1—C6—C5	−0.1 (8)	C7—C8—C10—O3	−147.0 (5)
C4—C5—C6—N1	−179.4 (5)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*WB*···O1^{i}	0.85 (4)	1.97 (2)	2.794 (5)	164 (5)
O1*W*—H1*WA*···N2^{ii}	0.86 (4)	2.14 (2)	2.968 (5)	163 (5)
O3—H3···O1*W*	0.84	1.76	2.574 (5)	162

Symmetry codes: (i) −*x*+1, *y*+1/2, −*z*+1/2; (ii) −*x*, −*y*+1, −*z*+1.