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3,8-Dimethylquinazoline-2,4(1*H*,3*H*)-dione

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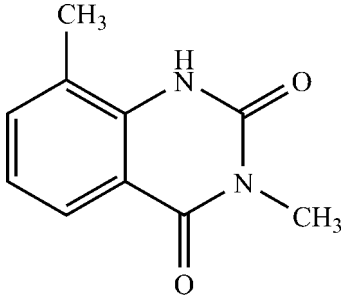
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.002 Å; R factor = 0.043; wR factor = 0.143; data-to-parameter ratio = 15.7.

In the title compound, $C_{10}H_{10}N_2O_2$, all non-H atoms are approximately co-planar with an r.m.s. deviation of 0.016 Å. In the crystal, molecules are linked into inversion dimers by pairs of N—H···O hydrogen bonds. Chains along [010] are buiilt up by π–π interactions [centroid–centroid distance = 3.602 (1) Å] between the benzene and piperazine rings of adjacent molecules.

Related literature

For the synthesis and background to the title compound, see Feng *et al.* (2010).



Experimental

Crystal data

$C_{10}H_{10}N_2O_2$ M_r = 190.20
Monoclinic, $P2_1/c$
a = 8.3604 (17) Å
b = 4.8599 (10) Å
c = 22.288 (5) Å
β = 92.09 (3)°
V = 905.0 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 295 K
0.29 × 0.23 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.972, T_{max} = 0.981
8373 measured reflections
2085 independent reflections
1497 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.143
S = 1.10
2085 reflections
133 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1···O2^{i}	0.888 (19)	2.011 (19)	2.8931 (17)	171.8 (17)

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

Data collection: *RAPID-AUTO* (Rigaku, 1998); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG5192).

References

Feng, Q., Liu, Z.-L., Xiong, L.-X., Wang, M.-Z., Li, Y.-Q. & Li, Z.-M. (2010). *J. Agric. Food Chem.* **58**, 12327–12336.
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Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

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S1. Comment

The title compound is the intermediate of a kind of highly potent and selective insecticide (Feng *et al.*, 2010). Herein, we report the synthesis and crystal structure of the title compound.

In the title compound, $C_{10}H_{10}N_2O_2$, all non-hydrogen atoms lie on the same plane with the Rms about 0.016 Å, the largest deviation being 0.037 (1) Å for atom O2 (Figure 1).

The isolated title compound molecules are linked by N—H···O hydrogen bonds into dimers (Figure 2, Table 1). Furthermore, the chain structures alone [010] direction are bulit up by /pi-/pi interatcions (center to center distances of 3.602 (1) Å) between the phenyl groups and piperazinyl groups of the adjacent molecules (Figure 3).

S2. Experimental

The title compound was synthesized as the reference method (Feng *et al.*, 2010): To a solution of 2-amino-*N*,3-dimethyl-benzamide (1.64 g, 1.0 mmol) in THF (20 ml), bis(trichloromethyl)-carbonate (1.0 g, 0.33 mmol) was added, and then keep stirring for 2h. After that THF was removed and water (20 ml) was added slowly. The resulting suspension was filtered, and the solids were washed with water (15 ml) and dried (yield 65%). The crystals suitable for X-ray diffraction were obtained by slow evaporation from methanol solution at room temperature for several days.

S3. Refinement

H atoms bound to C atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.93 Å (aromatic); C—H = 0.96 Å (methyl), and with U_{iso}(H) = 1.2U_{eq}(C), while N-bound H atom was found from Fourier-map and was freely refined.

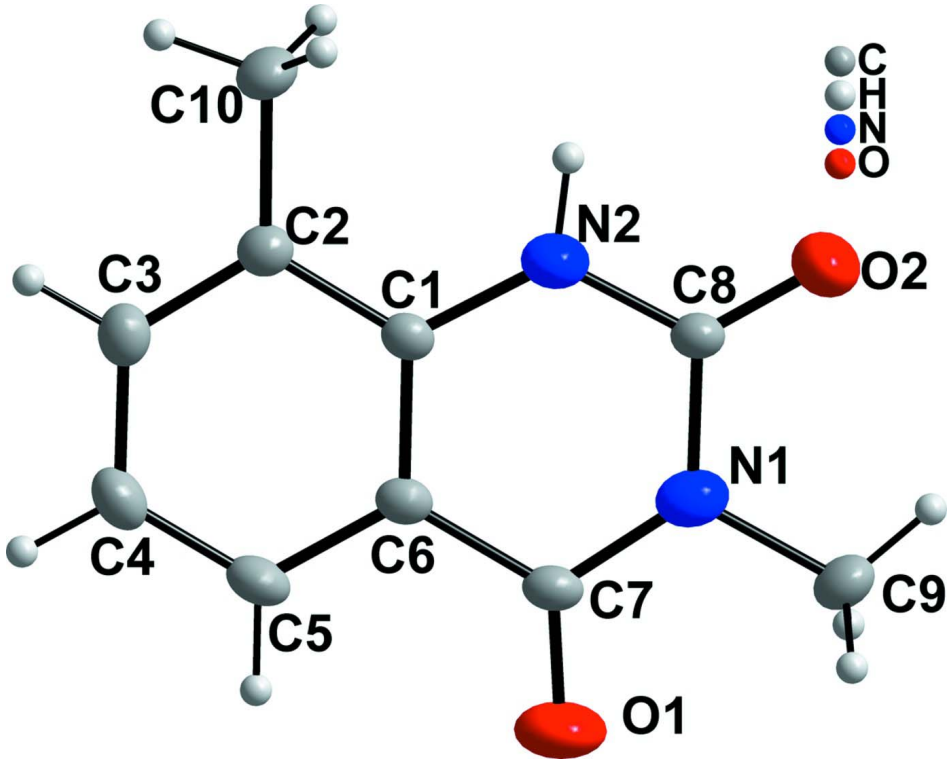

Figure 1

The molecular structure of the title compound, showing displacement ellipsoids at the 50% probability level for non-H atoms.

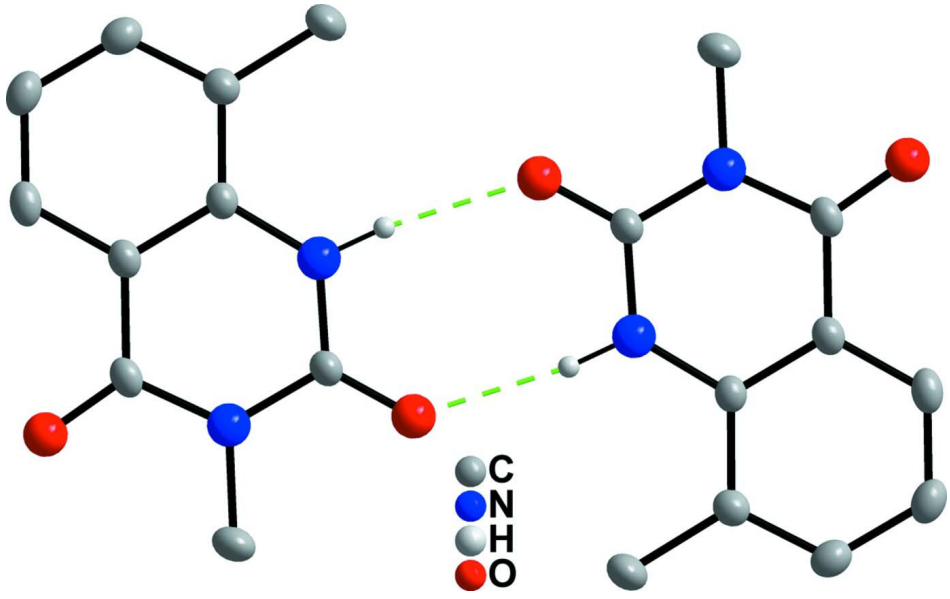

Figure 2

A dimer structure showing the N—H···O hydrogen bonds, no involving H atoms have been omitted for clarity.

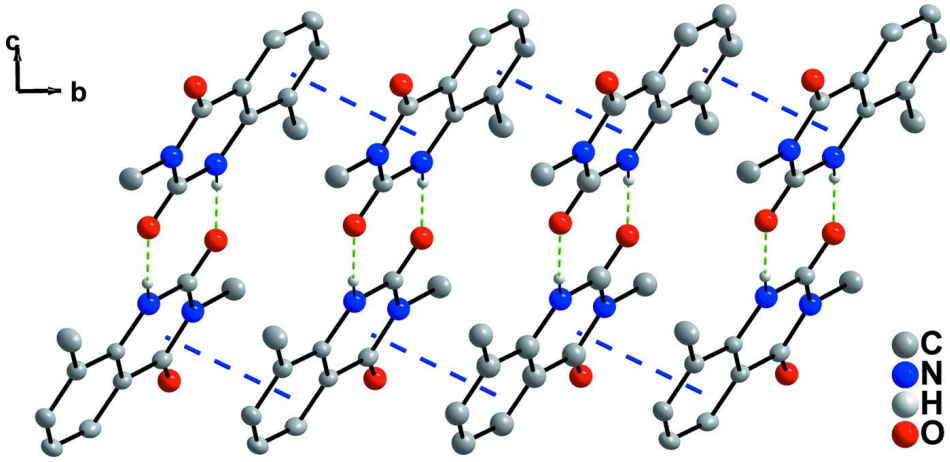

Figure 3

A partial packing view, showing chain structure forming by π-π interactions along [010] direction, no involving H atoms have been omitted for clarity.

3,8-Dimethylquinazoline-2,4(1*H*,3*H*)-dione

Crystal data

$C_{10}H_{10}N_2O_2$
$M_r = 190.20$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 8.3604$ (17) Å
$b = 4.8599$ (10) Å
$c = 22.288$ (5) Å
$\beta = 92.09$ (3)°
$V = 905.0$ (3) Å^3
$Z = 4$
$F(000) = 400$
$D_x = 1.396$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6086 reflections
$\theta = 3.0$–$27.5°$
$\mu = 0.10$ mm^{-1}
$T = 295$ K
Block, colorless
0.29 × 0.23 × 0.19 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min} = 0.972$, $T_{max} = 0.981$
8373 measured reflections
2085 independent reflections
1497 reflections with $I > 2\sigma(I)$
$R_{int} = 0.030$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.0°$
$h = -10 \rightarrow 10$
$k = -6 \rightarrow 6$
$l = -28 \rightarrow 28$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.143$
$S = 1.10$
2085 reflections
133 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2 + 0.0221P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ 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. 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.36317 (16)	0.6713 (2)	0.37609 (6)	0.0319 (3)
C2	0.47592 (17)	0.4802 (3)	0.35641 (6)	0.0378 (3)
C3	0.43348 (19)	0.3256 (3)	0.30632 (7)	0.0436 (4)
H3	0.5058	0.1965	0.2927	0.052*
C4	0.2866 (2)	0.3552 (3)	0.27525 (7)	0.0459 (4)
H4	0.2625	0.2481	0.2415	0.055*
C5	0.17879 (18)	0.5421 (3)	0.29463 (6)	0.0420 (4)
H5	0.0807	0.5633	0.2741	0.050*
C6	0.21521 (16)	0.7020 (3)	0.34530 (6)	0.0343 (3)
C7	0.09745 (16)	0.8962 (3)	0.36722 (6)	0.0371 (3)
C8	0.29243 (17)	1.0222 (3)	0.44833 (6)	0.0359 (3)
C9	0.03023 (19)	1.2458 (4)	0.44098 (7)	0.0499 (4)
H9A	0.0786	1.3429	0.4745	0.075*
H9B	−0.0632	1.1502	0.4536	0.075*
H9C	0.0002	1.3743	0.4099	0.075*
C10	0.63640 (19)	0.4454 (4)	0.38882 (8)	0.0536 (4)
H10A	0.6956	0.3027	0.3698	0.080*
H10B	0.6206	0.3963	0.4299	0.080*
H10C	0.6950	0.6150	0.3874	0.080*
N1	0.14501 (13)	1.0469 (2)	0.41799 (5)	0.0374 (3)
H1	0.486 (2)	0.829 (3)	0.4482 (8)	0.056 (5)*
N2	0.39514 (14)	0.8334 (2)	0.42650 (5)	0.0368 (3)
O1	−0.03642 (13)	0.9286 (3)	0.34450 (5)	0.0551 (4)
O2	0.32575 (13)	1.1631 (2)	0.49285 (5)	0.0510 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0322 (7)	0.0340 (6)	0.0292 (6)	−0.0020 (5)	−0.0025 (5)	0.0018 (5)
C2	0.0353 (7)	0.0401 (7)	0.0378 (7)	0.0022 (6)	0.0001 (6)	0.0014 (6)
C3	0.0483 (9)	0.0425 (8)	0.0404 (8)	0.0021 (7)	0.0063 (6)	−0.0053 (6)
C4	0.0548 (9)	0.0472 (8)	0.0354 (7)	−0.0065 (7)	−0.0019 (7)	−0.0077 (6)
C5	0.0393 (8)	0.0504 (8)	0.0355 (7)	−0.0073 (7)	−0.0088 (6)	0.0019 (6)
C6	0.0329 (7)	0.0372 (7)	0.0324 (7)	−0.0013 (6)	−0.0040 (5)	0.0042 (5)
C7	0.0321 (7)	0.0425 (7)	0.0361 (7)	−0.0007 (6)	−0.0069 (5)	0.0052 (6)
C8	0.0321 (7)	0.0410 (7)	0.0343 (7)	0.0042 (6)	−0.0046 (5)	−0.0005 (6)

C9	0.0401 (8)	0.0572 (9)	0.0522 (9)	0.0160 (7)	0.0007 (7)	−0.0022 (8)
C10	0.0378 (8)	0.0624 (10)	0.0599 (10)	0.0144 (8)	−0.0063 (7)	−0.0102 (8)
N1	0.0291 (6)	0.0434 (6)	0.0393 (6)	0.0069 (5)	−0.0045 (5)	0.0007 (5)
N2	0.0306 (6)	0.0436 (6)	0.0356 (6)	0.0068 (5)	−0.0081 (5)	−0.0050 (5)
O1	0.0362 (6)	0.0710 (8)	0.0566 (7)	0.0086 (5)	−0.0182 (5)	−0.0021 (6)
O2	0.0438 (6)	0.0614 (7)	0.0467 (6)	0.0140 (5)	−0.0139 (5)	−0.0198 (5)

Geometric parameters (Å, º)

C1—N2	1.3901 (17)	C7—N1	1.3938 (17)
C1—C6	1.4005 (18)	C8—O2	1.2291 (17)
C1—C2	1.4049 (19)	C8—N2	1.3588 (18)
C2—C3	1.381 (2)	C8—N1	1.3891 (17)
C2—C10	1.5099 (19)	C9—N1	1.4675 (19)
C3—C4	1.395 (2)	C9—H9A	0.9600
C3—H3	0.9300	C9—H9B	0.9600
C4—C5	1.361 (2)	C9—H9C	0.9600
C4—H4	0.9300	C10—H10A	0.9600
C5—C6	1.395 (2)	C10—H10B	0.9600
C5—H5	0.9300	C10—H10C	0.9600
C6—C7	1.461 (2)	N2—H1	0.888 (19)
C7—O1	1.2217 (16)		
N2—C1—C6	118.44 (12)	O2—C8—N2	122.52 (12)
N2—C1—C2	121.04 (11)	O2—C8—N1	121.05 (13)
C6—C1—C2	120.52 (12)	N2—C8—N1	116.43 (11)
C3—C2—C1	117.16 (13)	N1—C9—H9A	109.5
C3—C2—C10	121.54 (13)	N1—C9—H9B	109.5
C1—C2—C10	121.29 (12)	H9A—C9—H9B	109.5
C2—C3—C4	122.73 (14)	N1—C9—H9C	109.5
C2—C3—H3	118.6	H9A—C9—H9C	109.5
C4—C3—H3	118.6	H9B—C9—H9C	109.5
C5—C4—C3	119.49 (13)	C2—C10—H10A	109.5
C5—C4—H4	120.3	C2—C10—H10B	109.5
C3—C4—H4	120.3	H10A—C10—H10B	109.5
C4—C5—C6	120.07 (13)	C2—C10—H10C	109.5
C4—C5—H5	120.0	H10A—C10—H10C	109.5
C6—C5—H5	120.0	H10B—C10—H10C	109.5
C5—C6—C1	120.02 (13)	C8—N1—C7	124.86 (11)
C5—C6—C7	120.04 (12)	C8—N1—C9	117.83 (12)
C1—C6—C7	119.92 (12)	C7—N1—C9	117.31 (11)
O1—C7—N1	119.92 (13)	C8—N2—C1	124.52 (11)
O1—C7—C6	124.26 (13)	C8—N2—H1	111.4 (11)
N1—C7—C6	115.81 (11)	C1—N2—H1	124.1 (11)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1···O2^{i}	0.888 (19)	2.011 (19)	2.8931 (17)	171.8 (17)

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