Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-Methylpyrazine 1,4-dioxide

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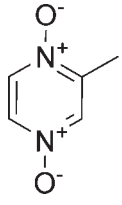
Received 2 November 2009; accepted 4 November 2009

Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.122; data-to-parameter ratio = 20.6.

The title compound, $C_5H_6N_2O_2$, was prepared from 2-methylpyrazine, acetic acid and hydrogen peroxide. In the crystal, π–π stacking interactions between neighboring molecules are observed, with a centroid–centroid distance of 3.7370 Å, an interplanar distance of 3.167 Å, and a slippage of 1.984 Å. Each molecule is linked to four neighbors through C—H···O hydrogen-bonding interactions, forming one-dimensional ribbons.

Related literature

For the synthesis of 2,2′-bipyridine *N,N*′-dioxide, see: Simpson *et al.* (1963). For the synthesis of lanthanide coordination networks with pyrazine *N,N*′-dioxide, see: Cardoso *et al.* (2001); Sun *et al.* (2004). For the use of 2-methylpyrazine 1,4-dioxide in the synthesis of a cadmium (II) coordination network, see: Shi *et al.* (2006). For the use of 2-methylpyrazine 1,4-dioxide in the synthesis of several molecular complexes, see: Sun *et al.* (2005); Xu *et al.* (2005*a*,*b*).



Experimental

Crystal data

$C_5H_6N_2O_2$
M_r = 126.12
Orthorhombic, *Pbca*
a = 6.3953 (9) Å
b = 12.2472 (18) Å
c = 13.6613 (19) Å
V = 1070.0 (3) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 173 K
0.53 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.763, T_{max} = 1.000
5556 measured reflections
1708 independent reflections
1407 reflections with $I > 2\sigma(I)$
R_{int} = 0.025

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.122
S = 1.07
1708 reflections
83 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 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
C1—H1···O2^{i}	0.93	2.31	3.2224 (16)	165
C2—H2···O2^{ii}	0.93	2.23	3.1405 (17)	167
C3—H3···O1^{iii}	0.93	2.29	3.2090 (15)	168

Symmetry codes: (i) $x-1, y, z$; (ii) $x-\frac{1}{2}, y, -z+\frac{3}{2}$; (iii) $x+1, y, z$.

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT-Plus* (Bruker, 2007); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *X-SEED*.

The authors are thankful to Allegheny College for providing funding in support of this research. The diffractometer was funded by the NSF (grant No. 0087210), the Ohio Board of Regents (grant No. CAP-491) and by Youngstown State University. The authors would also like to acknowledge the STaRBURSTT CyberInstrumentation Consortium for assistance with the crystallography.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2250).

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supporting information

Acta Cryst. (2009). E**65**, o3040 [doi:10.1107/S1600536809046534]

2-Methylpyrazine 1,4-dioxide

Jessica L. Gratton and Jacqueline M. Knaust

S1. Comment

The use of pyrazine *N,N*′-dioxide in the synthesis of lanthanide coordination networks has been of recent interest (Cardoso *et al.* (2001), Sun *et al.* (2004)). Shi *et al.* (2006) recently reported the use 2-methylpyrazine 1,4-dioxide in the synthesis of a cadmium (II) coordination network, and Sun *et al.* (2005), Xu *et al.* (2005*a*), and Xu *et al.* (2005*b*) report its use in the synthesis of several molecular complexes. The title compound was prepared using the reaction the conditions described by Simpson *et al.* (1963) to prepare 2,2′-bipyridine *N,N*′-dioxide.

The asymmetric unit of the title compound contains one 2-methylpyrazine 1,4-dioxide molecule (Figure 1). π-π stacking interactions with a centroid to centroid distance of 3.7370 Å, an interplanar distance of 3.167 Å, and a slippage of 1.984 Å. are observed between neighboring N-oxide molecules [symmetry code: -*x* + 1, -*y* + 1, -*z* + 1] (Figure 2). The title compound forms six C—H···O hydrogen bonds with four neighboring N-oxide molecules, and these hydrogen bonding interactions result in the formation of one-dimensional ribbons that propagate parallel to the *a* axis (Figure 4). As seen in the packing diagram, each one-dimensional ribbon is surrounded by six similar ribbons. (Figure 5)

S2. Experimental

2-Methylpyrazine (5.871 ml, 64.0 mmol), acetic acid (75 ml), and 30% hydrogen peroxide (13 ml) were heated at 343–353 K for 3 h. Additional hydrogen peroxide (9 ml) was added, and heating was continued. After an additional 19 h of heating the solution was cooled to room temperature. Crystals formed upon the addition of acetone (1*L*) and cooling to 273 K, and were recrystallized from hot water by addition of excess acetone and cooling to 273 K.

S3. Refinement

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

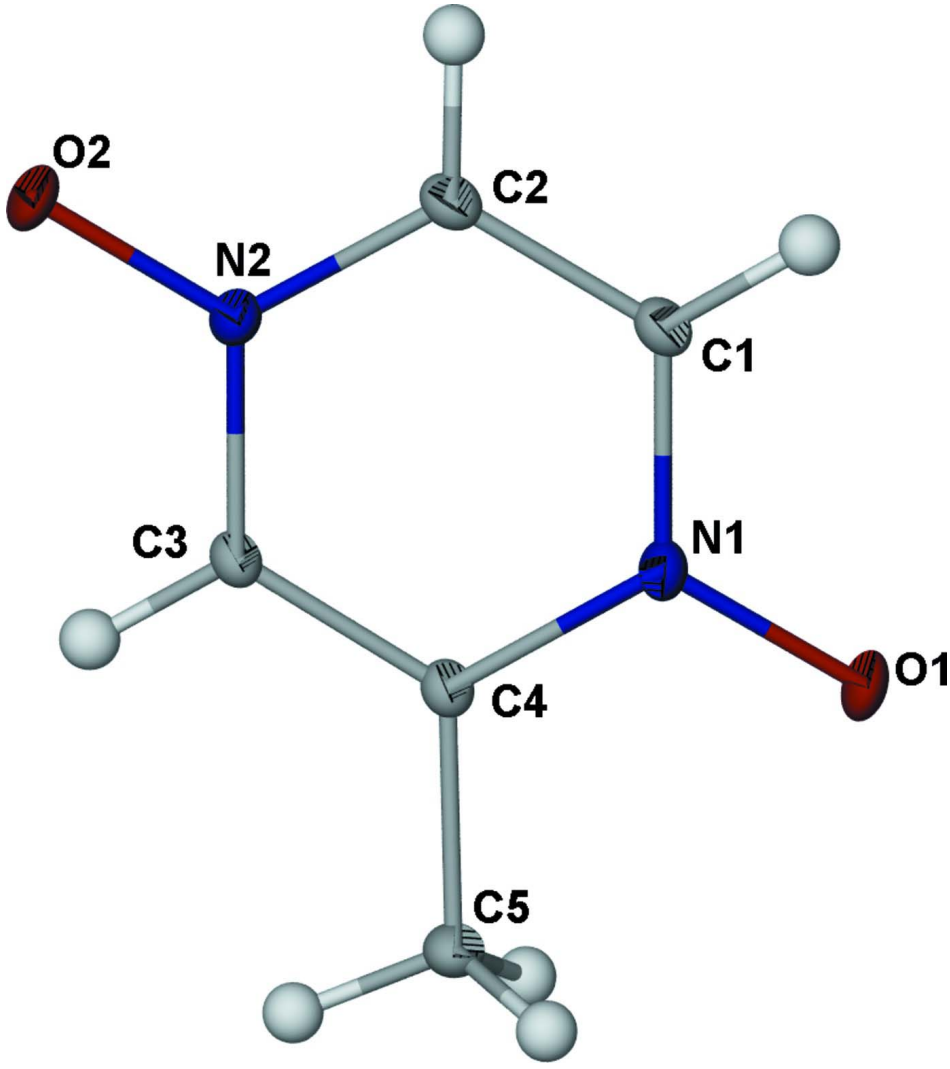

Figure 1

The molecular structure of the title compound with atom labels and 50% probability displacement ellipsoids for non-H atoms.

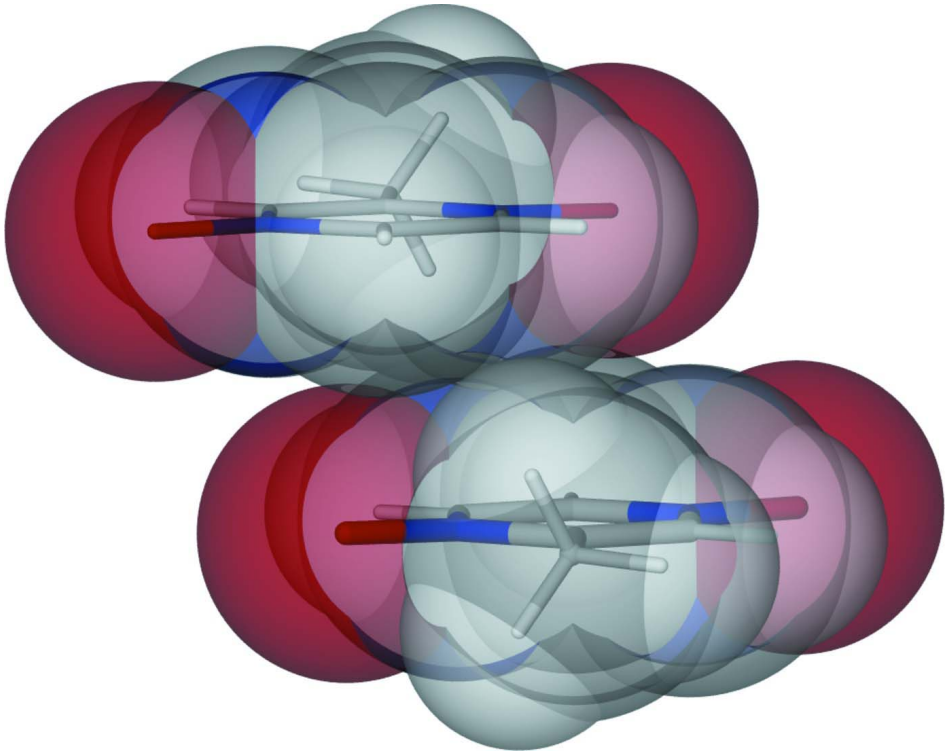

Figure 2

π-π interactions between neighboring 2-methylpyrazine 1,4-dioxide molecules.

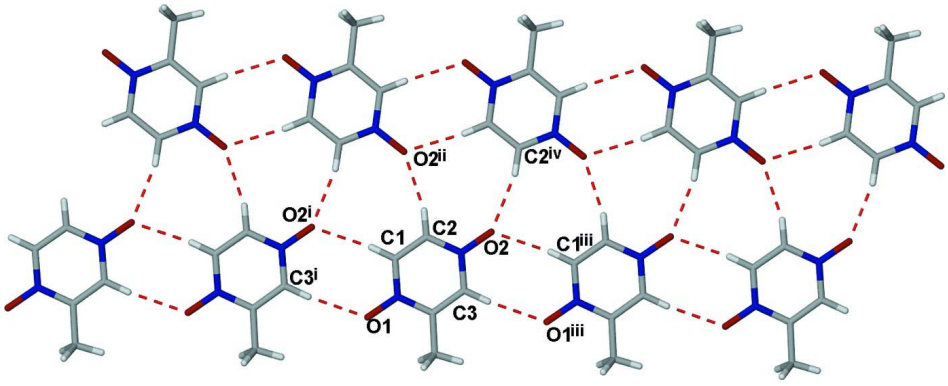

Figure 3

C—H···O hydrogen bonding interactions between neighboring 2-methylpyrazine 1,4-dioxide molecules. Hydrogen bonds are shown as dashed lines. Symmetry codes: (i) x - 1, y, z; (ii) x - 1/2, y, -z + 3/2; (iii) x + 1, y, z; (iv) x + 1/2, y, -z + 3/2.

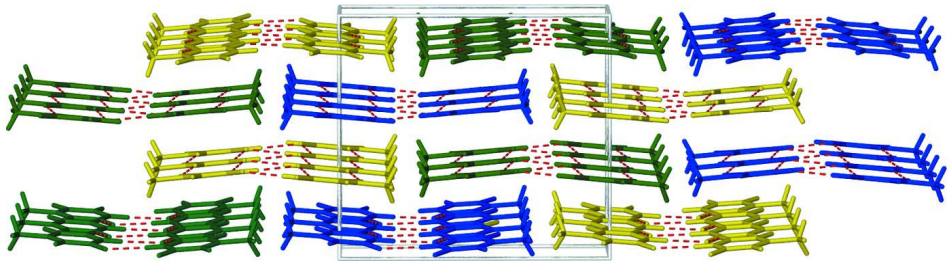

Figure 4

Packing of the title compound viewed down the *a* axis. Color scheme indicates individual C—H···O hydrogen bonded ribbons. Hydrogen bonds are shown as dashed lines

2-Methylpyrazine 1,4-dioxide

Crystal data

$C_5H_6N_2O_2$
$M_r = 126.12$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 6.3953\ (9)$ Å
$b = 12.2472\ (18)$ Å
$c = 13.6613\ (19)$ Å
$V = 1070.0\ (3)$ Å^3
$Z = 8$
$F(000) = 528$
$D_x = 1.566$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1890 reflections
$\theta = 3.0$–$31.5°$
$\mu = 0.12$ mm^{-1}
$T = 173$ K
Rod, colorless
0.53 × 0.20 × 0.15 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.763$, $T_{max} = 1.000$
5556 measured reflections
1708 independent reflections
1407 reflections with $I > 2\sigma(I)$
$R_{int} = 0.025$
$\theta_{max} = 31.8°$, $\theta_{min} = 3.0°$
$h = -9\rightarrow8$
$k = -17\rightarrow8$
$l = -11\rightarrow19$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.122$
$S = 1.07$
1708 reflections
83 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
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.6359P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.31$ e Å^{-3}

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^ > σ(*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}
O1	0.33101 (13)	0.64269 (8)	0.41676 (7)	0.0164 (2)
O2	0.99921 (14)	0.59296 (8)	0.64921 (7)	0.0183 (2)
N1	0.49403 (15)	0.63273 (8)	0.47271 (8)	0.0122 (2)
N2	0.83721 (15)	0.60672 (9)	0.59294 (8)	0.0128 (2)
C1	0.47150 (19)	0.62449 (10)	0.57203 (9)	0.0136 (2)
H1	0.3386	0.6279	0.5995	0.016*
C2	0.64141 (19)	0.61132 (10)	0.63137 (9)	0.0142 (2)
H2	0.6230	0.6055	0.6987	0.017*
C3	0.86033 (18)	0.61629 (10)	0.49464 (9)	0.0126 (2)
H3	0.9940	0.6141	0.4679	0.015*
C4	0.69092 (18)	0.62919 (10)	0.43327 (9)	0.0125 (2)
C5	0.7106 (2)	0.63969 (11)	0.32565 (9)	0.0165 (2)
H5A	0.6439	0.7059	0.3046	0.025*
H5B	0.6448	0.5783	0.2946	0.025*
H5C	0.8559	0.6417	0.3080	0.025*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0092 (4)	0.0223 (5)	0.0177 (4)	0.0005 (3)	−0.0044 (3)	0.0008 (3)
O2	0.0112 (4)	0.0298 (5)	0.0140 (4)	0.0004 (3)	−0.0053 (3)	0.0002 (4)
N1	0.0090 (4)	0.0139 (4)	0.0138 (5)	0.0001 (3)	−0.0009 (4)	−0.0004 (3)
N2	0.0103 (4)	0.0169 (5)	0.0113 (5)	−0.0005 (3)	−0.0013 (4)	−0.0003 (4)
C1	0.0118 (5)	0.0155 (5)	0.0136 (5)	0.0000 (4)	0.0027 (4)	0.0001 (4)
C2	0.0137 (5)	0.0167 (5)	0.0122 (5)	−0.0006 (4)	0.0026 (4)	−0.0006 (4)
C3	0.0097 (4)	0.0160 (5)	0.0120 (5)	−0.0007 (4)	0.0013 (4)	−0.0006 (4)
C4	0.0104 (5)	0.0143 (5)	0.0126 (5)	0.0000 (4)	0.0008 (4)	−0.0004 (4)
C5	0.0142 (5)	0.0242 (6)	0.0112 (5)	0.0008 (4)	0.0007 (4)	0.0015 (5)

Geometric parameters (Å, º)

O1—N1	1.2985 (13)	C2—H2	0.9300
O2—N2	1.3011 (13)	C3—C4	1.3789 (16)
N1—C1	1.3681 (16)	C3—H3	0.9300
N1—C4	1.3703 (15)	C4—C5	1.4813 (18)
N2—C3	1.3561 (16)	C5—H5A	0.9600
N2—C2	1.3590 (15)	C5—H5B	0.9600
C1—C2	1.3653 (17)	C5—H5C	0.9600
C1—H1	0.9300		

O1—N1—C1	120.41 (10)	N2—C3—C4	121.75 (11)
O1—N1—C4	120.62 (10)	N2—C3—H3	119.1
C1—N1—C4	118.97 (10)	C4—C3—H3	119.1
O2—N2—C3	120.63 (10)	N1—C4—C3	119.11 (11)
O2—N2—C2	120.72 (10)	N1—C4—C5	117.75 (11)
C3—N2—C2	118.65 (10)	C3—C4—C5	123.14 (11)
C2—C1—N1	120.92 (11)	C4—C5—H5A	109.5
C2—C1—H1	119.5	C4—C5—H5B	109.5
N1—C1—H1	119.5	H5A—C5—H5B	109.5
N2—C2—C1	120.59 (11)	C4—C5—H5C	109.5
N2—C2—H2	119.7	H5A—C5—H5C	109.5
C1—C2—H2	119.7	H5B—C5—H5C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{i}	0.93	2.31	3.2224 (16)	165
C2—H2···O2^{ii}	0.93	2.23	3.1405 (17)	167
C3—H3···O1^{iii}	0.93	2.29	3.2090 (15)	168

Symmetry codes: (i) $x-1, y, z$; (ii) $x-1/2, y, -z+3/2$; (iii) $x+1, y, z$.