Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

5,6-Dimethylpyrazine-2,3-dicarboxylic acid

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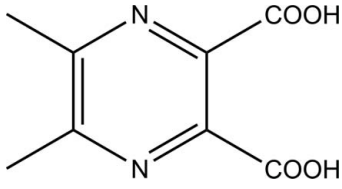
Received 13 November 2011; accepted 5 December 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.049; wR factor = 0.133; data-to-parameter ratio = 11.4.

The asymmetric unit of the title compound, $C_8H_8N_2O_4$, consists of one complete molecule and a second molecule generated by the application of twofold axis. The mean planes of the two carboxyl groups attached to the pyrazine ring at neighboring positions are twisted by 10.8 (1) and 87.9 (1)° in the complete molecule and 43.0 (1)° in the symmetry-generated molecule. The crystal packing features O—H···N hydrogen bonds, which link the molecules into layers along [101].

Related literature

For the synthesis of the title compound, see Tsuda & Fujishima (1981). For the structure of the hydrate of the title compound, see Vishweshwar *et al.* (2001, 2004). For a related compound containing pyrazine-2,3-dicarboxylic acid, see: Alborés & Rentschler (2009).



Experimental

Crystal data

$C_8H_8N_2O_4$ M_r = 196.16
Monoclinic, $C2/c$
a = 15.873 (3) Å
b = 14.057 (3) Å
c = 11.991 (2) Å
β = 109.21 (3)°
V = 2526.6 (9) Å^3
Z = 12
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 293 K
0.30 × 0.25 × 0.20 mm

Data collection

Rigaku SCX-mini diffractometer
10832 measured reflections
2230 independent reflections
1937 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.133
S = 1.07
2230 reflections
196 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.63 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N2^{i}	0.82 (1)	2.04	2.845 (2)	167
O3—H3···N3^{ii}	0.82 (1)	2.00	2.803 (2)	165
O5—H5···N1^{iii}	0.82 (1)	2.06	2.874 (2)	169

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-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: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2010).

The author thanks the Jilin Business and Technology College for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: JJ2111).

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supporting information

Acta Cryst. (2012). E**68**, o132 [doi:10.1107/S1600536811052366]

5,6-Dimethylpyrazine-2,3-dicarboxylic acid

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S1. Comment

2,3-dicarboxypyrazine-based ligands are well suited for the building of large clusters. With two carboxyl groups available for binding and a non-binding site opposite, selective interactions with metal ions are common. For example, a Co36 cluster using a pyrazine-2,3-dicarboxylic acid ligand similar to the title compound has been reported (Alborés & Rentschler, 2009) Similarly, the crystal structure of the title compound containing one water molecule in the unit cell has also been reported (Vishweshwar *et al.*, 2001; Vishweshwar *et al.*, 2004), In view of the importance of compounds containing this ligand we report herin the crystal structure of the title compound, (I).

The asymmetric unit of the title compound, $C_8H_8N_2O_4$, consists of one molecule and a second complete molecule generated by the application of a centre of inversion (Fig. 1). For each molecule, the mean planes of the two carboxyl groups attached to the pyrazine ring at neighboring positionsare twisted by 10.8 (1)° and 87.9 (3)°. Crystal packing is stabilized by O—H···N hydrogen bonds which link the molecules into layers along [101] (Fig. 2).

S2. Experimental

The compound was synthesized by a reported reaction (Tsuda & Fujishima, 1981) and crystallized by a solvent-thermal reaction as follows: 196.16 mg (1 mmol) $C_8H_8N_2O_4$ and 10 ml*N*, *N*-dimethylformamide (DMF) was added to a 20 ml Teflon vessel. The vessel was sealed and placed inside a stainless steel autoclave, which was kept at 130°C for 72 h. Crystals suitable for single-crystal analysis were formed upon standing.

S3. Refinement

H atoms bonded to O atom were located in a Fourier difference map and refined with distance restraints of O—H = 0.820 Å, and with U_{iso}(H) = 1.5Ueq(O). The remaining H atoms were positioned geometrically and refined using the riding model, with C—H = 0.960 Å and with U_{iso}(H) = 1.5 times U_{eq}(C).

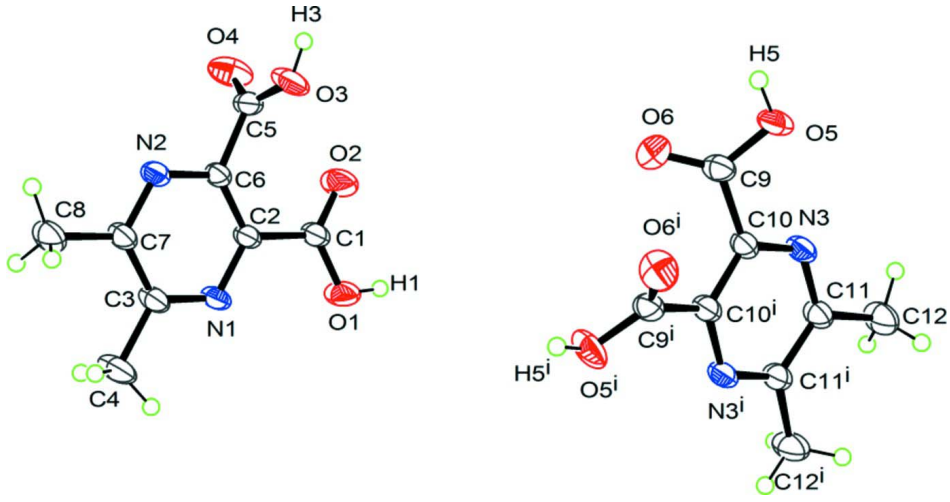

Figure 1

Molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

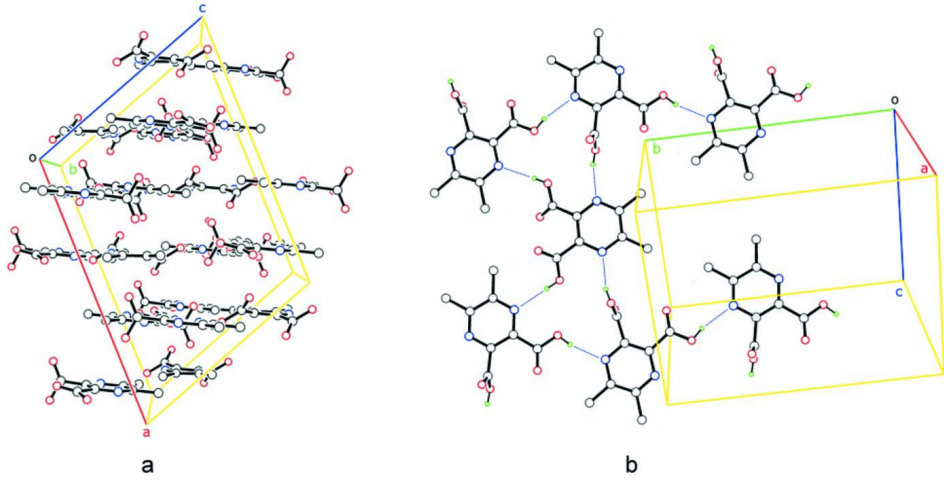

Figure 2

Packing view of (I). (*a*) View of layers in (I) along the *b* axis. (*b*) View of the title compound along the *a* axis. Dashed lines indicate O—H···N hydrogen bonds. H atoms not involved in hydrogen bonds have been deleted for clarity.

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Crystal data

$C_8H_8N_2O_4$
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Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
a = 15.873 (3) Å
b = 14.057 (3) Å
c = 11.991 (2) Å
β = 109.21 (3)°
V = 2526.6 (9) Å^3
Z = 12
F(000) = 1224
D_x = 1.547 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 10631 reflections
θ = 3.4–27.8°
μ = 0.13 mm^{-1}
T = 293 K
Block, colourless
0.30 × 0.25 × 0.20 mm

Data collection

Rigaku SCX-mini diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
10832 measured reflections
2230 independent reflections
1937 reflections with $I > 2\sigma(I)$
$R_{int} = 0.043$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.4°$
$h = -18 \rightarrow 18$
$k = -16 \rightarrow 16$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.133$
$S = 1.07$
2230 reflections
196 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
$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 3.0039P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.63$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ 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 $F^2 > \sigma(F^2)$ 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}
O1	0.28005 (14)	0.38972 (11)	0.78999 (16)	0.0595 (6)
H1	0.2587	0.3468	0.7428	0.089*
O2	0.17345 (15)	0.47314 (13)	0.66739 (18)	0.0745 (7)
O3	0.20112 (11)	0.67092 (14)	0.57803 (14)	0.0482 (5)
H3	0.1591	0.6847	0.5190	0.072*
O4	0.09881 (12)	0.67291 (15)	0.66709 (16)	0.0567 (5)
O5	0.40546 (13)	0.35937 (12)	0.02848 (13)	0.0474 (5)
H5	0.3815	0.4101	0.0026	0.071*
O6	0.40159 (11)	0.41517 (11)	0.20060 (15)	0.0439 (4)
N1	0.34098 (12)	0.54156 (12)	0.92729 (14)	0.0318 (4)
N2	0.28289 (12)	0.72021 (12)	0.84036 (15)	0.0325 (4)
N3	0.43566 (12)	0.18493 (12)	0.14017 (14)	0.0311 (4)
C1	0.23855 (16)	0.46795 (15)	0.75119 (19)	0.0362 (5)
C2	0.27656 (14)	0.55308 (14)	0.82342 (17)	0.0291 (5)
C3	0.37616 (14)	0.61868 (15)	0.98780 (18)	0.0325 (5)
C4	0.44770 (18)	0.60708 (18)	1.1037 (2)	0.0491 (7)
H4A	0.4231	0.6160	1.1662	0.074*

H4B	0.4936	0.6534	1.1110	0.074*
H4C	0.4726	0.5444	1.1090	0.074*
C5	0.17291 (15)	0.66150 (14)	0.66717 (19)	0.0324 (5)
C6	0.24752 (13)	0.64193 (14)	0.78045 (17)	0.0276 (5)
C7	0.34688 (14)	0.70945 (15)	0.94401 (18)	0.0315 (5)
C8	0.38542 (18)	0.79617 (17)	1.0119 (2)	0.0489 (7)
H8A	0.4458	0.8040	1.0132	0.073*
H8B	0.3846	0.7900	1.0913	0.073*
H8C	0.3508	0.8506	0.9752	0.073*
C9	0.42126 (14)	0.35590 (15)	0.14246 (18)	0.0316 (5)
C10	0.46645 (14)	0.26613 (14)	0.19620 (17)	0.0286 (5)
C11	0.46841 (15)	0.10400 (15)	0.19300 (18)	0.0315 (5)
C12	0.43794 (18)	0.01403 (16)	0.1277 (2)	0.0462 (6)
H12A	0.3870	0.0264	0.0593	0.069*
H12B	0.4220	−0.0306	0.1780	0.069*
H12C	0.4851	−0.0120	0.1036	0.069*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0717 (13)	0.0232 (8)	0.0508 (11)	0.0069 (8)	−0.0244 (9)	−0.0016 (7)
O2	0.0871 (15)	0.0368 (10)	0.0546 (12)	0.0090 (10)	−0.0375 (11)	−0.0110 (9)
O3	0.0448 (10)	0.0646 (12)	0.0236 (8)	0.0094 (9)	−0.0042 (7)	0.0075 (8)
O4	0.0354 (10)	0.0753 (14)	0.0467 (11)	0.0054 (9)	−0.0037 (8)	0.0138 (9)
O5	0.0688 (12)	0.0361 (9)	0.0240 (8)	0.0145 (9)	−0.0028 (8)	0.0065 (7)
O6	0.0525 (11)	0.0343 (9)	0.0447 (10)	0.0100 (8)	0.0157 (8)	0.0008 (7)
N1	0.0353 (10)	0.0277 (9)	0.0221 (9)	−0.0014 (8)	−0.0044 (8)	0.0045 (7)
N2	0.0346 (10)	0.0269 (9)	0.0257 (9)	−0.0005 (8)	−0.0040 (8)	0.0010 (7)
N3	0.0375 (10)	0.0278 (9)	0.0201 (9)	−0.0013 (8)	−0.0014 (7)	−0.0005 (7)
C1	0.0447 (13)	0.0259 (11)	0.0258 (11)	−0.0001 (10)	−0.0049 (10)	0.0017 (9)
C2	0.0313 (11)	0.0263 (11)	0.0223 (10)	−0.0008 (9)	−0.0011 (8)	0.0021 (8)
C3	0.0344 (12)	0.0310 (11)	0.0232 (11)	−0.0061 (9)	−0.0027 (9)	0.0032 (8)
C4	0.0528 (16)	0.0414 (13)	0.0319 (13)	−0.0110 (12)	−0.0148 (11)	0.0073 (10)
C5	0.0341 (13)	0.0226 (11)	0.0295 (12)	0.0000 (9)	−0.0042 (9)	0.0024 (8)
C6	0.0291 (11)	0.0241 (10)	0.0226 (10)	−0.0021 (9)	−0.0011 (8)	−0.0004 (8)
C7	0.0340 (12)	0.0298 (11)	0.0229 (11)	−0.0052 (9)	−0.0012 (9)	0.0002 (8)
C8	0.0577 (16)	0.0324 (13)	0.0369 (14)	−0.0090 (11)	−0.0111 (12)	−0.0027 (10)
C9	0.0312 (11)	0.0287 (11)	0.0275 (11)	−0.0012 (9)	−0.0006 (9)	0.0019 (9)
C10	0.0339 (11)	0.0263 (11)	0.0210 (10)	−0.0003 (9)	0.0026 (8)	−0.0011 (8)
C11	0.0392 (12)	0.0260 (11)	0.0239 (11)	−0.0021 (9)	0.0027 (9)	−0.0010 (8)
C12	0.0618 (16)	0.0295 (12)	0.0338 (13)	−0.0040 (11)	−0.0025 (11)	−0.0054 (10)

Geometric parameters (Å, º)

O1—C1	1.288 (3)	C3—C7	1.400 (3)
O1—H1	0.8200	C3—C4	1.487 (3)
O2—C1	1.183 (3)	C4—H4A	0.9600
O3—C5	1.294 (3)	C4—H4B	0.9600

O3—H3	0.8200	C4—H4C	0.9600
O4—C5	1.187 (3)	C5—C6	1.505 (3)
O5—C9	1.307 (3)	C7—C8	1.482 (3)
O5—H5	0.8200	C8—H8A	0.9600
O6—C9	1.192 (3)	C8—H8B	0.9600
N1—C3	1.322 (3)	C8—H8C	0.9600
N1—C2	1.336 (3)	C9—C10	1.489 (3)
N2—C7	1.330 (3)	C10—C10^{i}	1.377 (4)
N2—C6	1.333 (3)	C11—C11^{i}	1.405 (4)
N3—C11	1.323 (3)	C11—C12	1.482 (3)
N3—C10	1.333 (3)	C12—H12A	0.9600
C1—C2	1.484 (3)	C12—H12B	0.9600
C2—C6	1.372 (3)	C12—H12C	0.9600

C1—O1—H1	109.5	C2—C6—C5	124.95 (18)
C5—O3—H3	109.5	N2—C7—C3	120.76 (18)
C9—O5—H5	109.5	N2—C7—C8	118.06 (19)
C3—N1—C2	117.91 (17)	C3—C7—C8	121.17 (19)
C7—N2—C6	117.82 (18)	C7—C8—H8A	109.5
C11—N3—C10	118.29 (17)	C7—C8—H8B	109.5
O2—C1—O1	124.0 (2)	H8A—C8—H8B	109.5
O2—C1—C2	121.3 (2)	C7—C8—H8C	109.5
O1—C1—C2	114.60 (18)	H8A—C8—H8C	109.5
N1—C2—C6	121.33 (18)	H8B—C8—H8C	109.5
N1—C2—C1	119.07 (18)	O6—C9—O5	126.0 (2)
C6—C2—C1	119.54 (18)	O6—C9—C10	121.38 (19)
N1—C3—C7	120.89 (18)	O5—C9—C10	112.56 (19)
N1—C3—C4	118.57 (19)	N3—C10—C10^{i}	120.98 (11)
C7—C3—C4	120.54 (19)	N3—C10—C9	117.57 (18)
C3—C4—H4A	109.5	C10^{i}—C10—C9	121.30 (11)
C3—C4—H4B	109.5	N3—C11—C11^{i}	120.57 (11)
H4A—C4—H4B	109.5	N3—C11—C12	118.19 (19)
C3—C4—H4C	109.5	C11^{i}—C11—C12	121.23 (13)
H4A—C4—H4C	109.5	C11—C12—H12A	109.5
H4B—C4—H4C	109.5	C11—C12—H12B	109.5
O4—C5—O3	126.7 (2)	H12A—C12—H12B	109.5
O4—C5—C6	120.7 (2)	C11—C12—H12C	109.5
O3—C5—C6	112.4 (2)	H12A—C12—H12C	109.5
N2—C6—C2	121.28 (18)	H12B—C12—H12C	109.5
N2—C6—C5	113.73 (17)		

C3—N1—C2—C6	0.0 (3)	O4—C5—C6—C2	93.3 (3)
C3—N1—C2—C1	177.4 (2)	O3—C5—C6—C2	−91.5 (3)
O2—C1—C2—N1	169.2 (3)	C6—N2—C7—C3	0.4 (3)
O1—C1—C2—N1	−8.3 (3)	C6—N2—C7—C8	−179.0 (2)
O2—C1—C2—C6	−13.3 (4)	N1—C3—C7—N2	0.0 (3)
O1—C1—C2—C6	169.1 (2)	C4—C3—C7—N2	−180.0 (2)
C2—N1—C3—C7	−0.2 (3)	N1—C3—C7—C8	179.4 (2)

C2—N1—C3—C4	179.8 (2)	C4—C3—C7—C8	−0.6 (4)
C7—N2—C6—C2	−0.6 (3)	C11—N3—C10—C10^{i}	3.0 (4)
C7—N2—C6—C5	177.3 (2)	C11—N3—C10—C9	−172.7 (2)
N1—C2—C6—N2	0.5 (3)	O6—C9—C10—N3	134.4 (2)
C1—C2—C6—N2	−177.0 (2)	O5—C9—C10—N3	−44.4 (3)
N1—C2—C6—C5	−177.3 (2)	O6—C9—C10—C10^{i}	−41.3 (4)
C1—C2—C6—C5	5.3 (3)	O5—C9—C10—C10^{i}	140.0 (3)
O4—C5—C6—N2	−84.5 (3)	C10—N3—C11—C11^{i}	2.3 (4)
O3—C5—C6—N2	90.6 (2)	C10—N3—C11—C12	−176.5 (2)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N2^{ii}	0.82 (1)	2.04	2.845 (2)	167
O3—H3···N3^{iii}	0.82 (1)	2.00	2.803 (2)	165
O5—H5···N1^{iv}	0.82 (1)	2.06	2.874 (2)	169

Symmetry codes: (ii) −*x*+1/2, *y*−1/2, −*z*+3/2; (iii) −*x*+1/2, *y*+1/2, −*z*+1/2; (iv) *x*, *y*, *z*−1.