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1,4-Dimethylpiperazine-2,3-dione

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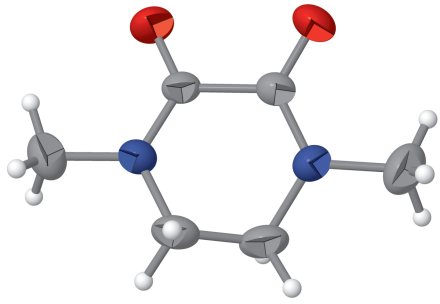
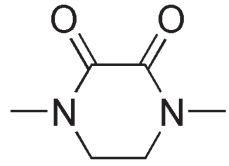
In the title compound, $C_6H_{10}N_2O_2$, the piperazine-2,3-dione ring adopts a half-chair conformation. In the crystal, the molecules are linked by weak C—H···O hydrogen bonds, forming (010) sheets.

3D view



Chemical scheme



Structure description

Piperazine and its derivatives are found within biologically active molecules across a diverse range of therapeutic areas, including antifungal, antibacterial, antimalarial, antipsychotic, antidepressant, and antitumor applications targeting colon, prostate, breast, lung, and leukemia cancers (Brockunier *et al.*, 2004; Bogatcheva *et al.*, 2005). As part of our studies in this area, we now describe the structure of the title compound, $C_6H_{10}N_2O_2$.

The asymmetric unit is shown in Fig. 1. The piperazine-2,3-dione ring adopts a half chair conformation, with C1 and C2 displaced from the other ring atoms by 0.279 (3) and −0.342 (3) Å, respectively. The molecule possesses local C_2 symmetry about an axis passing through the midpoints of the C1—C2 and C3—C4 bonds. In the crystal (Fig. 2), the molecules are connected by weak C2—H2*A*···O1 and C5—H5*C*···O2 hydrogen bonds (Table 1) to generate (010) layers.

A search of the Cambridge Structural Database (CSD; Version 5.43, update November 2022; Groom *et al.*, 2016) revealed some similar structures to the title compound, including 3,6-dibenzylidene-1,4-dimethylpiperazine-2,5-dione (CSD refcode IQOCEZ; Ge *et al.*, 2019), 2,5-bis(1-methyl-2-oxoindol-3-ylidene)-1,4-dimethylpiperazine-3,6-dione acetone solvate (PALVUT; Gompper *et al.*, 1992) and 6-(bromobenzyl)-3-benzylidene-6-*erythro*-hydroxy-1,4-dimethylpiperazine-2,5-dione (SAWSEO; Sterns *et al.*, 1989).

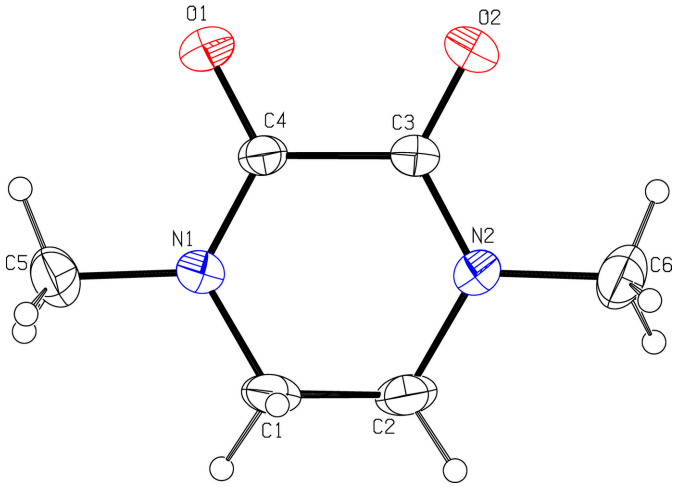


Figure 1
The asymmetric unit with displacement ellipsoids drawn at the 50% probability level.

Synthesis and crystallization

The title compound was prepared according to the literature method (Haraguchi *et al.*, 2015). Recrystallization of the solid from dichloromethane solution gave colorless plates, which were suitable for X-ray diffraction.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

References

Agilent (2012). *CrysAlis PRO* and *CrysAlis RED*. Agilent Technologies Ltd, Yarnton, England.

Bogatcheva, E., Hanrahan, C., Nikonenko, B., Samala, R., Chen, P., Gearhart, J., Barbosa, F., Einck, L., Nacy, C. A. & Protopopova, M. (2005). *J. Med. Chem.* **49**, 3045–3048.

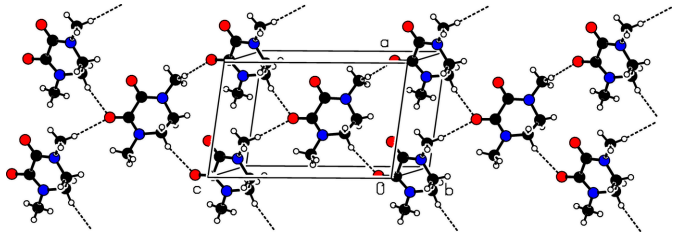


Figure 2
The crystal packing of the title compound.

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···O2^{i}	0.97	2.49	3.419 (3)	161
C5—H5C···O2^{ii}	0.96	2.54	3.481 (3)	168

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_6H_{10}N_2O_2$
M_r	142.16
Crystal system, space group	Monoclinic, $P2_1/n$
Temperature (K)	293
a, b, c (Å)	7.3781 (6), 8.0050 (6), 12.1306 (8)
β (°)	99.767 (7)
V (Å^3)	706.07 (9)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.10
Crystal size (mm)	0.37 × 0.32 × 0.29
Data collection	
Diffractometer	Agilent Xcalibur, Atlas, Gemini
Absorption correction	Analytical (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.507, 0.578
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	2746, 1624, 1194
R_{int}	0.016
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.681
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.063, 0.181, 1.07
No. of reflections	1624
No. of parameters	93
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.45, −0.21

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXT2018/2* (Sheldrick, 2015*a*), *SHELXL2018/3* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2020).

Brockunier, L. L., He, J., Colwell, L. F. Jr, Habulihaz, B., He, H., Leiting, B., Lyons, K. A., Marsilio, F., Patel, R. A., Teffera, Y., Wu, J. K., Thornberry, N. A., Weber, A. E. & Parmee, E. R. (2004). *Bioorg. Med. Chem. Lett.* **14**, 4763–4766.

Ge, Y., Han, Z., Wang, Z. & Ding, K. (2019). *J. Am. Chem. Soc.* **141**, 8981–8988.

Gompper, R., Kellner, R. & Polborn, K. (1992). *Angew. Chem. Int. Ed. Engl.* **31**, 1202–1205.

Groom, C. R., Bruno, I. J., Lightfoot, M. P. & Ward, S. C. (2016). *Acta Cryst.* B**72**, 171–179.

Haraguchi, R., Takada, Y. & Matsubara, S. (2015). *Org. Biomol. Chem.* **13**, 241–247.

Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.

Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.

Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

Spek, A. L. (2020). *Acta Cryst.* E**76**, 1–11.

Sterns, M., Patrick, J. M., Patrick, V. A. & White, A. H. (1989). *Aust. J. Chem.* **42**, 349.

full crystallographic data

IUCrData (2024). **9**, x240936 [https://doi.org/10.1107/S2414314624009362]

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Crystal data

$C_6H_{10}N_2O_2$
M_r = 142.16
Monoclinic, $P2_1/n$
a = 7.3781 (6) Å
b = 8.0050 (6) Å
c = 12.1306 (8) Å
β = 99.767 (7)°
V = 706.07 (9) Å^3
Z = 4
$F(000)$ = 304
D_x = 1.337 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 9307 reflections
θ = 3.5–26.4°
μ = 0.10 mm^{-1}
T = 293 K
Plate, colourless
0.37 × 0.32 × 0.29 mm

Data collection

Agilent Xcalibur, Atlas, Gemini diffractometer
Radiation source: fine-focus sealed tube
ω scans
Absorption correction: analytical (SADABS; Krause *et al.*, 2015)
T_{min} = 0.507, T_{max} = 0.578
2746 measured reflections
1624 independent reflections
1194 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 29.0°, θ_{min} = 3.1°
h = −10→8
k = −10→5
l = −6→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.181
S = 1.07
1624 reflections
93 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0839P)^2 + 0.2479P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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. All the H atoms were positioned geometrically (C—H = 0.96–0.97 Å) and were refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.4932 (2)	0.2262 (2)	0.62587 (12)	0.0591 (5)
O1	0.8009 (2)	0.3382 (3)	0.55770 (14)	0.0642 (6)
N1	0.6498 (2)	0.3592 (2)	0.38031 (14)	0.0427 (5)
N2	0.3516 (2)	0.1982 (2)	0.44717 (14)	0.0421 (5)
C4	0.6624 (3)	0.3190 (3)	0.48783 (16)	0.0380 (5)
C3	0.4923 (3)	0.2422 (2)	0.52590 (15)	0.0364 (5)
C5	0.7995 (4)	0.4489 (4)	0.3415 (2)	0.0616 (7)
H5A	0.899882	0.462273	0.402473	0.092*
H5B	0.756751	0.556833	0.313902	0.092*
H5C	0.840112	0.386644	0.282610	0.092*
C1	0.4771 (4)	0.3452 (3)	0.30300 (18)	0.0547 (7)
H1A	0.502520	0.333921	0.227477	0.066*
H1B	0.406408	0.446718	0.306177	0.066*
C2	0.3670 (4)	0.2011 (3)	0.32859 (19)	0.0555 (6)
H2A	0.245130	0.207406	0.283752	0.067*
H2B	0.424361	0.098582	0.309359	0.067*
C6	0.1915 (3)	0.1170 (4)	0.4791 (3)	0.0649 (8)
H6A	0.223240	0.073267	0.553547	0.097*
H6B	0.151882	0.027484	0.428037	0.097*
H6C	0.093814	0.196898	0.476737	0.097*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0637 (11)	0.0816 (12)	0.0319 (8)	−0.0121 (9)	0.0082 (7)	0.0062 (8)
O1	0.0445 (9)	0.0987 (14)	0.0438 (9)	−0.0153 (9)	−0.0083 (7)	0.0067 (9)
N1	0.0455 (10)	0.0500 (10)	0.0318 (9)	−0.0055 (8)	0.0045 (7)	0.0017 (7)
N2	0.0376 (9)	0.0480 (10)	0.0393 (10)	−0.0067 (8)	0.0023 (7)	−0.0029 (8)
C4	0.0358 (10)	0.0455 (11)	0.0307 (10)	0.0003 (9)	−0.0004 (8)	−0.0013 (8)
C3	0.0388 (10)	0.0384 (10)	0.0308 (10)	0.0031 (8)	0.0023 (8)	0.0000 (8)
C5	0.0650 (15)	0.0706 (17)	0.0544 (15)	−0.0116 (13)	0.0250 (12)	0.0023 (12)
C1	0.0653 (15)	0.0642 (15)	0.0299 (10)	−0.0058 (12)	−0.0055 (10)	0.0061 (10)
C2	0.0572 (14)	0.0649 (15)	0.0377 (12)	−0.0054 (12)	−0.0113 (10)	−0.0033 (10)
C6	0.0440 (13)	0.0738 (17)	0.0774 (19)	−0.0156 (12)	0.0118 (12)	−0.0092 (14)

Geometric parameters (Å, º)

O2—C3	1.218 (2)	C5—H5B	0.9600
O1—C4	1.222 (2)	C5—H5C	0.9600
N1—C4	1.331 (3)	C1—C2	1.474 (3)
N1—C1	1.452 (3)	C1—H1A	0.9700
N1—C5	1.461 (3)	C1—H1B	0.9700
N2—C3	1.333 (3)	C2—H2A	0.9700
N2—C6	1.457 (3)	C2—H2B	0.9700
N2—C2	1.462 (3)	C6—H6A	0.9600

C4—C3	1.537 (3)	C6—H6B	0.9600
C5—H5A	0.9600	C6—H6C	0.9600
C4—N1—C1	121.47 (18)	N1—C1—C2	112.27 (18)
C4—N1—C5	120.23 (19)	N1—C1—H1A	109.2
C1—N1—C5	117.27 (18)	C2—C1—H1A	109.2
C3—N2—C6	119.7 (2)	N1—C1—H1B	109.2
C3—N2—C2	121.30 (18)	C2—C1—H1B	109.2
C6—N2—C2	118.05 (19)	H1A—C1—H1B	107.9
O1—C4—N1	124.1 (2)	N2—C2—C1	110.93 (19)
O1—C4—C3	118.12 (18)	N2—C2—H2A	109.5
N1—C4—C3	117.77 (17)	C1—C2—H2A	109.5
O2—C3—N2	123.9 (2)	N2—C2—H2B	109.5
O2—C3—C4	118.32 (18)	C1—C2—H2B	109.5
N2—C3—C4	117.82 (17)	H2A—C2—H2B	108.0
N1—C5—H5A	109.5	N2—C6—H6A	109.5
N1—C5—H5B	109.5	N2—C6—H6B	109.5
H5A—C5—H5B	109.5	H6A—C6—H6B	109.5
N1—C5—H5C	109.5	N2—C6—H6C	109.5
H5A—C5—H5C	109.5	H6A—C6—H6C	109.5
H5B—C5—H5C	109.5	H6B—C6—H6C	109.5
C1—N1—C4—O1	−175.3 (2)	N1—C4—C3—O2	−170.0 (2)
C5—N1—C4—O1	−7.2 (3)	O1—C4—C3—N2	−170.1 (2)
C1—N1—C4—C3	5.3 (3)	N1—C4—C3—N2	9.4 (3)
C5—N1—C4—C3	173.39 (19)	C4—N1—C1—C2	−35.3 (3)
C6—N2—C3—O2	−3.8 (3)	C5—N1—C1—C2	156.3 (2)
C2—N2—C3—O2	−172.4 (2)	C3—N2—C2—C1	−37.7 (3)
C6—N2—C3—C4	176.84 (19)	C6—N2—C2—C1	153.5 (2)
C2—N2—C3—C4	8.3 (3)	N1—C1—C2—N2	49.3 (3)
O1—C4—C3—O2	10.5 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2*A*···O2^{i}	0.97	2.49	3.419 (3)	161
C5—H5*C*···O2^{ii}	0.96	2.54	3.481 (3)	168

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