



IUCrData

ISSN 2414-3146

1,4-Dimethylpiperazine-2,3-dione

Themmila Khamrang,^a C. Ponraj,^b Madhukar Hemamalini,^c G. Jerald Maria Antony^{b*} and Dhandayutham Saravanan^b

^aDepartment of Chemistry, Dhanamanjuri University, Manipur 795 001, India, ^bDepartment of Chemistry, National College, Tiruchirappalli, Tamil Nadu, India, and ^cDepartment of Chemistry, Mother Teresa Women's University, Kodaikanal, Tamil Nadu, India. *Correspondence e-mail: jerelewin.mine@gmail.com

Received 29 August 2024

Accepted 23 September 2024

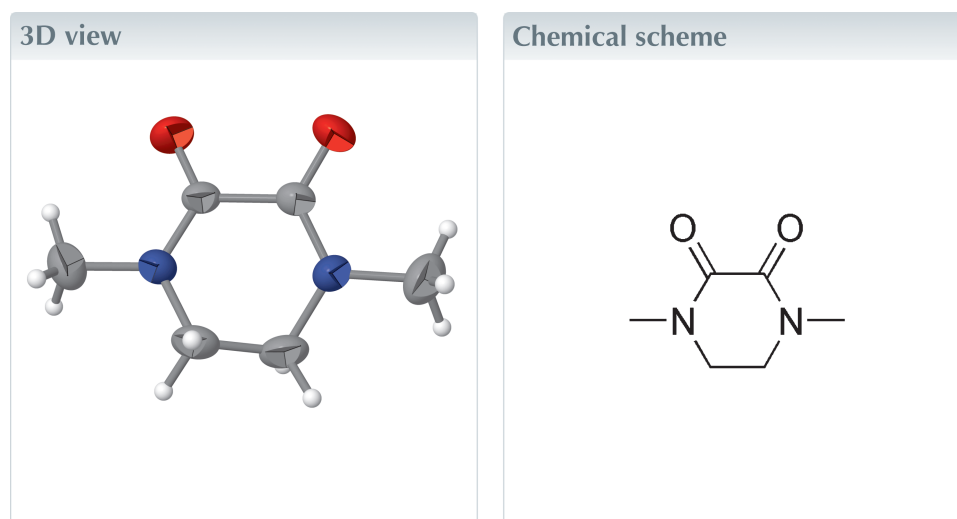
Edited by W. T. A. Harrison, University of Aberdeen, United Kingdom

Keywords: crystal structure; half chair; hydrogen bonding.

CCDC reference: 2386002

Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, C₆H₁₀N₂O₂, 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.



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, C₆H₁₀N₂O₂.

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 C₂ 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—H2A···O1 and C5—H5C···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-oxindol-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).

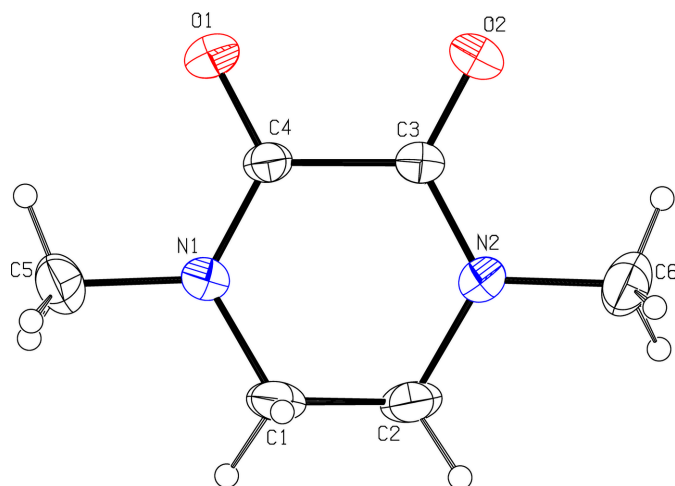


Figure 1
The asymmetric unit with displacement ellipsoids drawn at the 50% probability level.

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.

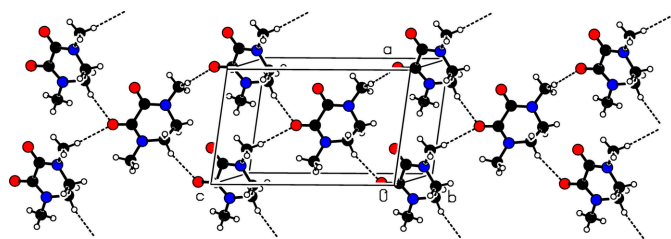


Figure 2
The crystal packing of the title compound.

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

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
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 ₆ H ₁₀ N ₂ O ₂
<i>M_r</i>	142.16
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	293
<i>a</i> , <i>b</i> , <i>c</i> (Å)	7.3781 (6), 8.0050 (6), 12.1306 (8)
β (°)	99.767 (7)
<i>V</i> (Å ³)	706.07 (9)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	0.10
Crystal size (mm)	0.37 × 0.32 × 0.29
Data collection	
Diffractometer	Agilent Xcalibur, Atlas, Gemini
Absorption correction	Analytical (<i>SADABS</i> ; Krause <i>et al.</i> , 2015)
<i>T_{min}</i> , <i>T_{max}</i>	0.507, 0.578
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	2746, 1624, 1194
<i>R_{int}</i>	0.016
(sin θ/λ) _{max} (Å ⁻¹)	0.681
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.063, 0.181, 1.07
No. of reflections	1624
No. of parameters	93
H-atom treatment	H-atom parameters constrained
Δρ _{max} , Δρ _{min} (e Å ⁻³)	0.45, −0.21

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXT2018/2* (Sheldrick, 2015a), *SHELXL2018/3* (Sheldrick, 2015b) 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.* **B72**, 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. (2015a). *Acta Cryst.* **A71**, 3–8.
Sheldrick, G. M. (2015b). *Acta Cryst.* **C71**, 3–8.
Spek, A. L. (2020). *Acta Cryst.* **E76**, 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]

1,4-Dimethylpiperazine-2,3-dione

Themmila Khamrang, C. Ponraj, Madhukar Hemamalini, G. Jerald Maria Antony and
Dhandayutham Saravanan

1,4-Dimethylpiperazine-2,3-dione

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

$\beta = 99.767$ (7)°

$V = 706.07$ (9) Å³

$Z = 4$

$F(000) = 304$

$D_x = 1.337$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9307 reflections

$\theta = 3.5$ – 26.4 °

$\mu = 0.10$ mm⁻¹

$T = 293$ K

Plate, colourless

$0.37 \times 0.32 \times 0.29$ 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.016$

$\theta_{\max} = 29.0$ °, $\theta_{\min} = 3.1$ °

$h = -10 \rightarrow 8$

$k = -10 \rightarrow 5$

$l = -6 \rightarrow 16$

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 $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^2)

	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 (\AA , $^\circ$)

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 (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C2—H2A \cdots O2 ⁱ	0.97	2.49	3.419 (3)	161
C5—H5C \cdots O2 ⁱⁱ	0.96	2.54	3.481 (3)	168

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