

# Crystal structure of 1-(piperidin-1-yl)-butane-1,3-dione

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Received 6 November 2014; accepted 25 November 2014

Edited by R. F. Baggio, Comisión Nacional de Energía Atómica, Argentina

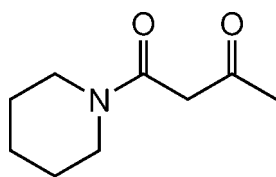
In the title compound, C<sub>9</sub>H<sub>15</sub>NO<sub>2</sub>, the piperidine ring exhibits a chair conformation. The butanedione subunit exhibits a conformation with the ketone C atom in an eclipsed position with respect to the amide carbonyl group. In the crystal, a two-dimensional layered arrangement is formed by hydrogen bonds of the C—H...O type between the methyl group and the exocyclic methylene unit as donor sites and the amide carbonyl O atom as the acceptor of a bifurcated hydrogen bond. These layers are oriented parallel to the *ab* plane.

**Keywords:** crystal structure; 1-(piperidin-1-yl)butane-1,3-dione; weak hydrogen bonding.

**CCDC reference:** 1035958

## 1. Related literature

For the synthetic procedure, see: Sridharan *et al.* (2010). For a survey concerning weak hydrogen bonds, see: Desiraju & Steiner (1999).



## 2. Experimental

### 2.1. Crystal data

C<sub>9</sub>H<sub>15</sub>NO<sub>2</sub>

*M<sub>r</sub>* = 169.22

Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 5.4455 (1) Å  
*b* = 9.1901 (2) Å  
*c* = 17.8837 (4) Å  
 $\beta$  = 94.506 (1)°  
*V* = 892.22 (3) Å<sup>3</sup>

*Z* = 4  
Mo *K*α radiation  
 $\mu$  = 0.09 mm<sup>−1</sup>  
*T* = 133 K  
0.12 × 0.09 × 0.07 mm

### 2.2. Data collection

Nonius KappaCCD diffractometer  
Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 2002)  
*T*<sub>min</sub> = 0.717, *T*<sub>max</sub> = 0.746

5612 measured reflections  
2035 independent reflections  
1777 reflections with *I* > 2σ(*I*)  
*R*<sub>int</sub> = 0.023

### 2.3. Refinement

*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.040  
*wR* (*F*<sup>2</sup>) = 0.107  
*S* = 1.14  
2035 reflections

169 parameters  
All H-atom parameters refined  
 $\Delta\rho_{\text{max}}$  = 0.31 e Å<sup>−3</sup>  
 $\Delta\rho_{\text{min}}$  = −0.20 e Å<sup>−3</sup>

**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>
C7—H7B...O1 <sup>i</sup>	0.96 (2)	2.459 (17)	3.378 (3)	161 (1)
C9—H9B...O1 <sup>ii</sup>	0.99 (2)	2.601 (18)	3.466 (3)	146 (1)

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

Data collection: *COLLECT* (Nonius, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; 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, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

## Acknowledgements

MS gratefully acknowledges a PhD grant from the Deutsche Bundesstiftung Umwelt.

Supporting information for this paper is available from the IUCr electronic archives (Reference: BG2540).

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## supporting information

*Acta Cryst.* (2014). E70, o1297 [doi:10.1107/S1600536814025768]

## Crystal structure of 1-(piperidin-1-yl)butane-1,3-dione

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### S1. Comment

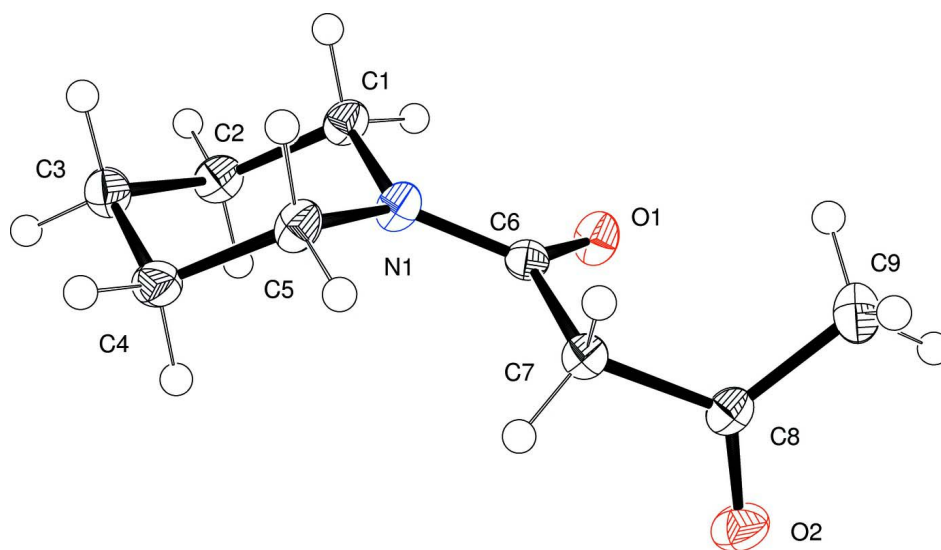
The title compound is an intermediate in the synthesis of 2,2-dimethoxy-1-(pyridin-2-yl)ethanone and has been synthesized from piperidine and 2,2,6-trimethyl-4*H*-1,3-dioxin-4-one following a modified procedure (Sridharan *et al.*, 2010). As it is expected the piperidine ring shows a chair conformation and the amide substructure is planar. The butane-dione subunit exhibits a conformation with the ketone carbon atom in an eclipsed position with respect to the amide carbonyl group (Figure 1). The dihedral angle between the two carbonyl groups therefore measures to 81.4°. In the crystal structure, a two-dimensional layered arrangement is formed by hydrogen bonds of the C–H···O type between the methyl group and the exocyclic methylene unit as donor sites and the amide carbonyl oxygen atom as the acceptor of a bifurcated hydrogen bond. These layers are oriented along to the *ab* plane (Figure 2).

### S2. Experimental

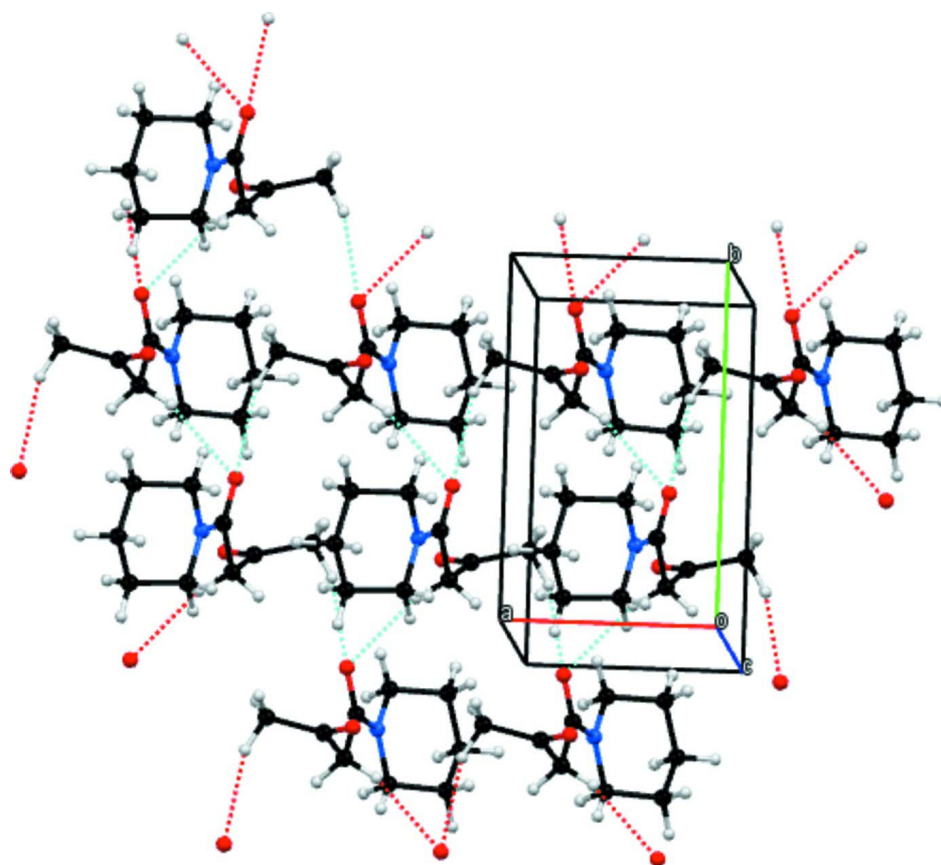
0.2 Mol of piperidine (17.0 g, 19.8 ml), 0.2 mol of sodium acetate (17.0 g) and a slight excess (0.26 mol, 37.0 g, 34.6 ml) 2,2,6-trimethyl-4*H*-1,3-dioxin-4-one together with 40 ml THF are refluxed for 24 h. After cooling down to room temperature the solution is filtered and the remaining sodium acetate is washed with diethylether (3 × 20 ml). The combined THF and diethylether solutions are treated with brine (3 × 25 ml) and dried with Na<sub>2</sub>SO<sub>4</sub>. After filtration the organic solution is evaporated to dryness. The raw product may either be purified by chromatography (silica, light petroleum: ethyl acetate = 9: 1, yield 67%) or by distillation *in vacuo* (0.2 mbar, yield: 83%). If only the product fraction that is condensed into a Schlenk tube which is cooled with liquid nitrogen using a bath temperature above 100°C is collected, crystalline material of the title compound suitable for X-ray crystallography is obtained.

### S3. Refinement

The positions of all hydrogen atoms have been determined from a Fourier map and all hydrogen atoms were refined without any constraints.

**Figure 1**

Molecular structure of the title compound with thermal ellipsoids drawn at the 50% probability level.

**Figure 2**

Crystal structure of the title compound showing layers of molecules along the *ab* plane which are built up by bifurcated C–H...O hydrogen bonds.

**1-(Piperidin-1-yl)butane-1,3-dione***Crystal data*

$C_9H_{15}NO_2$	$Z = 4$
$M_r = 169.22$	$F(000) = 368$
Monoclinic, $P2_1/c$	$D_x = 1.260 \text{ Mg m}^{-3}$
Hall symbol: $-P 2_1bc$	Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$
$a = 5.4455 (1) \text{ \AA}$	$\mu = 0.09 \text{ mm}^{-1}$
$b = 9.1901 (2) \text{ \AA}$	$T = 133 \text{ K}$
$c = 17.8837 (4) \text{ \AA}$	Prism, colourless
$\beta = 94.506 (1)^\circ$	$0.12 \times 0.09 \times 0.07 \text{ mm}$
$V = 892.22 (3) \text{ \AA}^3$	

*Data collection*

Nonius KappaCCD	5612 measured reflections
diffractometer	2035 independent reflections
Radiation source: fine-focus sealed tube	1777 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{\text{int}} = 0.023$
$\phi$ - $\omega$ -scan	$\theta_{\text{max}} = 27.5^\circ$ , $\theta_{\text{min}} = 2.3^\circ$
Absorption correction: multi-scan	$h = -7 \rightarrow 6$
( <i>SADABS</i> ; Sheldrick, 2002)	$k = -7 \rightarrow 11$
$T_{\text{min}} = 0.717$ , $T_{\text{max}} = 0.746$	$l = -23 \rightarrow 21$

*Refinement*

Refinement on $F^2$	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.040$	All H-atom parameters refined
$wR(F^2) = 0.107$	$w = 1/[\sigma^2(F_o^2) + (0.041P)^2 + 0.4171P]$
$S = 1.14$	where $P = (F_o^2 + 2F_c^2)/3$
2035 reflections	$(\Delta/\sigma)_{\text{max}} < 0.001$
169 parameters	$\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$
0 restraints	$\Delta\rho_{\text{min}} = -0.19 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	

*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 ( $\text{\AA}^2$ )*

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.73273 (18)	0.88779 (10)	0.27173 (5)	0.0232 (2)
O2	0.6753 (2)	0.70560 (11)	0.11035 (5)	0.0279 (3)
N1	0.5948 (2)	0.73198 (12)	0.35732 (6)	0.0202 (3)
C1	0.5498 (3)	0.84895 (15)	0.41067 (7)	0.0210 (3)
H1B	0.661 (3)	0.8308 (18)	0.4570 (9)	0.022 (4)*

H1A	0.594 (3)	0.9416 (19)	0.3881 (9)	0.023 (4)*
C2	0.2840 (3)	0.84538 (15)	0.43124 (8)	0.0236 (3)
H2B	0.257 (3)	0.9213 (19)	0.4687 (10)	0.027 (4)*
H2A	0.175 (3)	0.868 (2)	0.3865 (11)	0.032 (5)*
C3	0.2232 (3)	0.69623 (16)	0.46223 (8)	0.0236 (3)
H3A	0.331 (3)	0.6804 (19)	0.5103 (10)	0.027 (4)*
H3B	0.049 (4)	0.6953 (19)	0.4749 (10)	0.032 (5)*
C4	0.2753 (3)	0.57716 (16)	0.40620 (8)	0.0230 (3)
H4B	0.166 (3)	0.588 (2)	0.3604 (10)	0.033 (5)*
H4A	0.245 (3)	0.4812 (19)	0.4270 (9)	0.027 (4)*
C5	0.5391 (3)	0.58557 (14)	0.38390 (8)	0.0212 (3)
H5B	0.657 (3)	0.5641 (18)	0.4283 (9)	0.021 (4)*
H5A	0.565 (3)	0.5139 (19)	0.3448 (9)	0.023 (4)*
C6	0.6852 (2)	0.76255 (14)	0.29111 (7)	0.0170 (3)
C7	0.7267 (2)	0.63574 (14)	0.23861 (7)	0.0180 (3)
H7B	0.575 (3)	0.5836 (18)	0.2286 (9)	0.022 (4)*
H7A	0.854 (3)	0.5704 (19)	0.2607 (9)	0.024 (4)*
C8	0.8154 (2)	0.69439 (14)	0.16614 (7)	0.0190 (3)
C9	1.0830 (3)	0.73141 (18)	0.16694 (9)	0.0270 (3)
H9C	1.137 (4)	0.780 (2)	0.2127 (12)	0.046 (6)*
H9B	1.177 (4)	0.639 (2)	0.1650 (12)	0.049 (6)*
H9A	1.116 (4)	0.784 (2)	0.1229 (13)	0.053 (6)*

*Atomic displacement parameters ( $\text{\AA}^2$ )*

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0316 (5)	0.0149 (5)	0.0240 (5)	0.0001 (4)	0.0072 (4)	0.0018 (4)
O2	0.0346 (6)	0.0292 (6)	0.0191 (5)	−0.0014 (4)	−0.0030 (4)	0.0028 (4)
N1	0.0295 (6)	0.0133 (5)	0.0183 (5)	−0.0003 (4)	0.0048 (4)	−0.0007 (4)
C1	0.0284 (7)	0.0164 (6)	0.0183 (6)	−0.0016 (5)	0.0035 (5)	−0.0030 (5)
C2	0.0285 (7)	0.0198 (7)	0.0226 (7)	0.0029 (5)	0.0024 (5)	−0.0021 (5)
C3	0.0231 (7)	0.0237 (7)	0.0244 (7)	−0.0008 (5)	0.0040 (5)	−0.0001 (5)
C4	0.0269 (7)	0.0208 (7)	0.0209 (7)	−0.0043 (5)	−0.0007 (5)	0.0003 (5)
C5	0.0309 (7)	0.0144 (6)	0.0188 (6)	−0.0006 (5)	0.0042 (5)	0.0020 (5)
C6	0.0167 (6)	0.0164 (6)	0.0176 (6)	0.0011 (5)	−0.0006 (5)	0.0005 (5)
C7	0.0200 (6)	0.0156 (6)	0.0183 (6)	0.0004 (5)	0.0015 (5)	0.0000 (5)
C8	0.0249 (7)	0.0142 (6)	0.0183 (6)	0.0023 (5)	0.0032 (5)	−0.0013 (5)
C9	0.0238 (7)	0.0298 (8)	0.0281 (8)	0.0013 (6)	0.0076 (6)	0.0033 (6)

*Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

O1—C6	1.2353 (16)	C4—C5	1.523 (2)
O2—C8	1.2117 (17)	C4—H4B	0.980 (18)
N1—C6	1.3472 (17)	C4—H4A	0.976 (18)
N1—C5	1.4667 (16)	C5—H5B	1.001 (16)
N1—C1	1.4706 (16)	C5—H5A	0.978 (17)
C1—C2	1.521 (2)	C6—C7	1.5244 (18)
C1—H1B	1.001 (16)	C7—C8	1.5171 (18)

C1—H1A	0.981 (17)	C7—H7B	0.959 (17)
C2—C3	1.525 (2)	C7—H7A	0.976 (17)
C2—H2B	0.987 (18)	C8—C9	1.496 (2)
C2—H2A	0.981 (19)	C9—H9C	0.96 (2)
C3—C4	1.525 (2)	C9—H9B	0.99 (2)
C3—H3A	1.012 (17)	C9—H9A	0.95 (2)
C3—H3B	0.993 (19)		
C6—N1—C5	125.13 (11)	H4B—C4—H4A	107.2 (14)
C6—N1—C1	120.59 (11)	N1—C5—C4	110.79 (11)
C5—N1—C1	114.27 (10)	N1—C5—H5B	107.6 (9)
N1—C1—C2	110.57 (11)	C4—C5—H5B	110.1 (9)
N1—C1—H1B	107.1 (9)	N1—C5—H5A	110.0 (10)
C2—C1—H1B	108.7 (9)	C4—C5—H5A	110.2 (9)
N1—C1—H1A	108.0 (10)	H5B—C5—H5A	108.2 (13)
C2—C1—H1A	112.9 (9)	O1—C6—N1	122.69 (12)
H1B—C1—H1A	109.4 (13)	O1—C6—C7	119.67 (12)
C1—C2—C3	110.19 (11)	N1—C6—C7	117.64 (11)
C1—C2—H2B	110.2 (10)	C8—C7—C6	109.09 (10)
C3—C2—H2B	109.9 (10)	C8—C7—H7B	110.2 (10)
C1—C2—H2A	108.8 (11)	C6—C7—H7B	109.2 (10)
C3—C2—H2A	110.9 (11)	C8—C7—H7A	107.6 (10)
H2B—C2—H2A	106.8 (14)	C6—C7—H7A	110.8 (10)
C2—C3—C4	110.44 (12)	H7B—C7—H7A	109.9 (14)
C2—C3—H3A	108.1 (10)	O2—C8—C9	122.56 (13)
C4—C3—H3A	109.2 (10)	O2—C8—C7	120.78 (12)
C2—C3—H3B	109.2 (10)	C9—C8—C7	116.62 (12)
C4—C3—H3B	111.9 (10)	C8—C9—H9C	110.5 (13)
H3A—C3—H3B	107.8 (14)	C8—C9—H9B	108.1 (12)
C5—C4—C3	111.32 (11)	H9C—C9—H9B	107.5 (18)
C5—C4—H4B	107.5 (11)	C8—C9—H9A	110.6 (13)
C3—C4—H4B	110.3 (11)	H9C—C9—H9A	113.9 (18)
C5—C4—H4A	109.8 (10)	H9B—C9—H9A	105.9 (18)
C3—C4—H4A	110.6 (10)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C7—H7B $\cdots$ O1 <sup>i</sup>	0.96 (2)	2.459 (17)	3.378 (3)	161 (1)
C9—H9B $\cdots$ O1 <sup>ii</sup>	0.99 (2)	2.601 (18)	3.466 (3)	146 (1)

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