

## 2-Ethoxymethyl-6-ethyl-2,3,4,5-tetrahydro-1,2,4-triazine-3,5-dione

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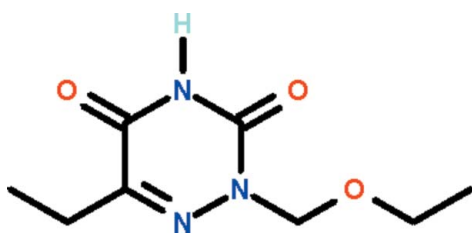
Received 19 December 2011; accepted 26 December 2011

Key indicators: single-crystal X-ray study;  $T = 100$  K; mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å;  $R$  factor = 0.042;  $wR$  factor = 0.107; data-to-parameter ratio = 16.6.

The 1,2,4-triazine ring of the title compound,  $\text{C}_8\text{H}_{13}\text{N}_3\text{O}_3$ , is nearly planar (r.m.s. deviation = 0.019 Å). The imino group is hydrogen-bond donor to the exocyclic O atom at the 5-position of an adjacent molecule, the  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bond generating a chain parallel to the  $b$  axis.

### Related literature

For the synthesis, see: El-Brollosy (2008).



### Experimental

#### Crystal data

$\text{C}_8\text{H}_{13}\text{N}_3\text{O}_3$   
 $M_r = 199.21$   
Monoclinic,  $C2/c$

$a = 20.4078$  (19) Å  
 $b = 4.4343$  (3) Å  
 $c = 22.6285$  (17) Å

$\beta = 111.813$  (10)°  
 $V = 1901.1$  (3) Å<sup>3</sup>  
 $Z = 8$   
Mo  $K\alpha$  radiation

$\mu = 0.11$  mm<sup>-1</sup>  
 $T = 100$  K  
 $0.35 \times 0.25 \times 0.15$  mm

#### Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector  
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)  
 $T_{\min} = 0.963$ ,  $T_{\max} = 0.984$

3623 measured reflections  
2174 independent reflections  
1739 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.107$   
 $S = 1.06$   
2174 reflections  
131 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.28$  e Å<sup>-3</sup>

**Table 1**

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1}\cdots\text{O1}^{\dagger}$	0.89 (2)	1.95 (2)	2.837 (2)	174.1 (17)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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: *publCIF* (Westrip, 2010).

We thank the Deanship of Scientific Research and the Research Center of the College of Pharmacy, King Saud University, and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5421).

### References

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Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.  
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Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.  
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

**supplementary materials**

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## 2-Ethoxymethyl-6-ethyl-2,3,4,5-tetrahydro-1,2,4-triazine-3,5-dione

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### Comment

The compound (Scheme 1) was synthesized for an evaluation of its anti-microbial properties (El-Brollosy, 2008). The 1,2,4-triazine ring is planar; the C atom at the 6-position deviates by 0.155 (2) Å from the mean plane whereas the C atom at the 2-position deviates from the mean plane by 0.081 (2) Å in the opposite direction (Fig. 1). The amino group is hydrogen-bond donor to the exocyclic O atom at the 5-position, the hydrogen bond generating a linear chain parallel to the *b*-axis of the monoclinic unit cell (Table 1, Fig. 2).

### Experimental

The compound was synthesized by using a reported method (El-Brollosy, 2008), and was recrystallized from ethanol.

### Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 0.98 Å,  $U_{\text{iso}}(\text{H})$  1.2 to 1.5 $U_{\text{eq}}(\text{C})$ ] and were included in the refinement in the riding model approximation.

The imino H-atom was located in a difference Fourier map, and was freely refined.

The -2 0 2 reflection that was affected by the beam-stop was omitted.

### Figures

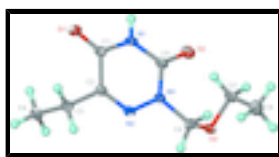


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of  $\text{C}_8\text{H}_{13}\text{N}_3\text{O}_3$  at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

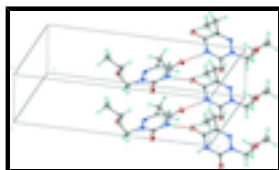


Fig. 2. Hydrogen-bonded chain structure.

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### Crystal data

$\text{C}_8\text{H}_{13}\text{N}_3\text{O}_3$

$M_r = 199.21$

$F(000) = 848$

$D_x = 1.392 \text{ Mg m}^{-3}$

# supplementary materials

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Monoclinic,  $C2/c$   
Hall symbol:  $-C\ 2yc$   
 $a = 20.4078$  (19) Å  
 $b = 4.4343$  (3) Å  
 $c = 22.6285$  (17) Å  
 $\beta = 111.813$  (10)°  
 $V = 1901.1$  (3) Å<sup>3</sup>  
 $Z = 8$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073$  Å  
Cell parameters from 1592 reflections  
 $\theta = 2.3$ – $27.5$ °  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 100$  K  
Prism, colorless  
 $0.35 \times 0.25 \times 0.15$  mm

## Data collection

Agilent SuperNova Dual  
diffractometer with an Atlas detector  
Radiation source: SuperNova (Mo) X-ray Source  
Mirror  
Detector resolution: 10.4041 pixels mm<sup>-1</sup>  
 $\omega$  scan  
Absorption correction: multi-scan  
(*CrysAlis PRO*; Agilent, 2010)  
 $T_{\min} = 0.963$ ,  $T_{\max} = 0.984$   
3623 measured reflections

2174 independent reflections  
1739 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.023$   
 $\theta_{\max} = 27.6$ °,  $\theta_{\min} = 3.4$ °  
 $h = -26 \rightarrow 26$   
 $k = -5 \rightarrow 5$   
 $l = -15 \rightarrow 29$

## Refinement

Refinement on  $F^2$   
Least-squares matrix: full  
 $R[F^2 > 2\sigma(F^2)] = 0.042$   
 $wR(F^2) = 0.107$   
 $S = 1.06$   
2174 reflections  
131 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 atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o^2) + (0.0432P)^2 + 1.2698P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} = 0.001$   
 $\Delta\rho_{\max} = 0.31$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.28$  e Å<sup>-3</sup>

## Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å<sup>2</sup>)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.73492 (5)	0.9252 (3)	0.68461 (5)	0.0201 (3)
O2	0.58065 (6)	0.2354 (3)	0.70746 (5)	0.0214 (3)
O3	0.42473 (5)	0.6126 (2)	0.59416 (5)	0.0172 (3)
N1	0.65902 (7)	0.5753 (3)	0.69639 (6)	0.0152 (3)
N2	0.56241 (6)	0.7358 (3)	0.57988 (6)	0.0138 (3)
N3	0.54722 (6)	0.5285 (3)	0.61778 (5)	0.0144 (3)
C1	0.67786 (7)	0.7953 (3)	0.66355 (7)	0.0141 (3)
C2	0.62375 (7)	0.8617 (3)	0.59987 (6)	0.0130 (3)

C3	0.64247 (8)	1.0709 (3)	0.55670 (7)	0.0159 (3)
H3A	0.6664	1.2510	0.5810	0.019*
H3B	0.5987	1.1387	0.5221	0.019*
C4	0.69101 (8)	0.9188 (4)	0.52750 (7)	0.0197 (3)
H4A	0.7019	1.0617	0.4994	0.030*
H4B	0.6672	0.7418	0.5029	0.030*
H4C	0.7349	0.8559	0.5616	0.030*
C5	0.59417 (8)	0.4319 (3)	0.67624 (7)	0.0152 (3)
C6	0.47519 (8)	0.4069 (3)	0.59096 (7)	0.0167 (3)
H6A	0.4642	0.3508	0.5459	0.020*
H6B	0.4726	0.2216	0.6144	0.020*
C7	0.42810 (8)	0.6726 (4)	0.65804 (7)	0.0200 (3)
H7A	0.4709	0.7914	0.6818	0.024*
H7B	0.4300	0.4809	0.6810	0.024*
C8	0.36268 (9)	0.8478 (4)	0.65258 (8)	0.0252 (4)
H8A	0.3635	0.8925	0.6953	0.038*
H8B	0.3207	0.7277	0.6292	0.038*
H8C	0.3614	1.0369	0.6298	0.038*
H1	0.6913 (10)	0.515 (4)	0.7331 (9)	0.024 (5)*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0179 (6)	0.0261 (6)	0.0138 (5)	-0.0056 (5)	0.0028 (4)	-0.0018 (4)
O2	0.0237 (6)	0.0226 (6)	0.0166 (5)	-0.0020 (5)	0.0059 (4)	0.0063 (5)
O3	0.0153 (5)	0.0246 (6)	0.0112 (5)	0.0001 (5)	0.0043 (4)	-0.0008 (4)
N1	0.0148 (6)	0.0179 (7)	0.0095 (6)	0.0019 (5)	0.0007 (5)	0.0013 (5)
N2	0.0168 (6)	0.0133 (6)	0.0118 (6)	0.0010 (5)	0.0059 (5)	0.0008 (5)
N3	0.0141 (6)	0.0171 (6)	0.0102 (6)	-0.0017 (5)	0.0026 (5)	0.0019 (5)
C1	0.0146 (7)	0.0161 (7)	0.0115 (7)	0.0006 (6)	0.0047 (5)	-0.0019 (6)
C2	0.0153 (7)	0.0126 (7)	0.0110 (7)	0.0019 (6)	0.0048 (5)	-0.0006 (6)
C3	0.0183 (7)	0.0149 (7)	0.0138 (7)	-0.0010 (6)	0.0049 (6)	0.0006 (6)
C4	0.0205 (8)	0.0245 (8)	0.0153 (7)	-0.0030 (7)	0.0081 (6)	-0.0011 (6)
C5	0.0171 (7)	0.0163 (7)	0.0121 (7)	0.0027 (6)	0.0054 (6)	0.0001 (6)
C6	0.0159 (7)	0.0188 (8)	0.0141 (7)	-0.0035 (6)	0.0039 (6)	-0.0015 (6)
C7	0.0246 (8)	0.0243 (8)	0.0118 (7)	-0.0063 (7)	0.0075 (6)	-0.0017 (6)
C8	0.0254 (9)	0.0310 (9)	0.0226 (8)	-0.0068 (8)	0.0127 (7)	-0.0089 (7)

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

O1—C1	1.2256 (18)	C3—H3A	0.9900
O2—C5	1.2164 (18)	C3—H3B	0.9900
O3—C6	1.3974 (18)	C4—H4A	0.9800
O3—C7	1.4461 (17)	C4—H4B	0.9800
N1—C1	1.3654 (19)	C4—H4C	0.9800
N1—C5	1.3839 (19)	C6—H6A	0.9900
N1—H1	0.889 (19)	C6—H6B	0.9900
N2—C2	1.2894 (19)	C7—C8	1.509 (2)
N2—N3	1.3688 (16)	C7—H7A	0.9900

## supplementary materials

N3—C5	1.3821 (18)	C7—H7B	0.9900
N3—C6	1.4686 (19)	C8—H8A	0.9800
C1—C2	1.4832 (19)	C8—H8B	0.9800
C2—C3	1.496 (2)	C8—H8C	0.9800
C3—C4	1.536 (2)		
C6—O3—C7	114.18 (11)	H4A—C4—H4C	109.5
C1—N1—C5	125.26 (13)	H4B—C4—H4C	109.5
C1—N1—H1	117.7 (12)	O2—C5—N3	123.49 (14)
C5—N1—H1	117.0 (12)	O2—C5—N1	122.24 (13)
C2—N2—N3	119.17 (12)	N3—C5—N1	114.26 (13)
N2—N3—C5	124.75 (12)	O3—C6—N3	112.50 (12)
N2—N3—C6	114.37 (11)	O3—C6—H6A	109.1
C5—N3—C6	120.88 (12)	N3—C6—H6A	109.1
O1—C1—N1	122.90 (13)	O3—C6—H6B	109.1
O1—C1—C2	122.78 (13)	N3—C6—H6B	109.1
N1—C1—C2	114.33 (12)	H6A—C6—H6B	107.8
N2—C2—C1	122.02 (13)	O3—C7—C8	107.51 (12)
N2—C2—C3	119.31 (12)	O3—C7—H7A	110.2
C1—C2—C3	118.61 (13)	C8—C7—H7A	110.2
C2—C3—C4	111.76 (12)	O3—C7—H7B	110.2
C2—C3—H3A	109.3	C8—C7—H7B	110.2
C4—C3—H3A	109.3	H7A—C7—H7B	108.5
C2—C3—H3B	109.3	C7—C8—H8A	109.5
C4—C3—H3B	109.3	C7—C8—H8B	109.5
H3A—C3—H3B	107.9	H8A—C8—H8B	109.5
C3—C4—H4A	109.5	C7—C8—H8C	109.5
C3—C4—H4B	109.5	H8A—C8—H8C	109.5
H4A—C4—H4B	109.5	H8B—C8—H8C	109.5
C3—C4—H4C	109.5		
C2—N2—N3—C5	-2.5 (2)	C1—C2—C3—C4	74.86 (17)
C2—N2—N3—C6	178.28 (12)	N2—N3—C5—O2	-175.83 (13)
C5—N1—C1—O1	176.71 (14)	C6—N3—C5—O2	3.3 (2)
C5—N1—C1—C2	-4.0 (2)	N2—N3—C5—N1	3.3 (2)
N3—N2—C2—C1	-1.8 (2)	C6—N3—C5—N1	-177.56 (12)
N3—N2—C2—C3	175.47 (12)	C1—N1—C5—O2	179.42 (14)
O1—C1—C2—N2	-175.88 (14)	C1—N1—C5—N3	0.3 (2)
N1—C1—C2—N2	4.8 (2)	C7—O3—C6—N3	-68.72 (15)
O1—C1—C2—C3	6.9 (2)	N2—N3—C6—O3	-74.79 (15)
N1—C1—C2—C3	-172.48 (12)	C5—N3—C6—O3	106.00 (15)
N2—C2—C3—C4	-102.47 (16)	C6—O3—C7—C8	-169.27 (13)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 $\cdots$ O1 <sup>i</sup>	0.89 (2)	1.95 (2)	2.837 (2)	174.1 (17)

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

Fig. 1

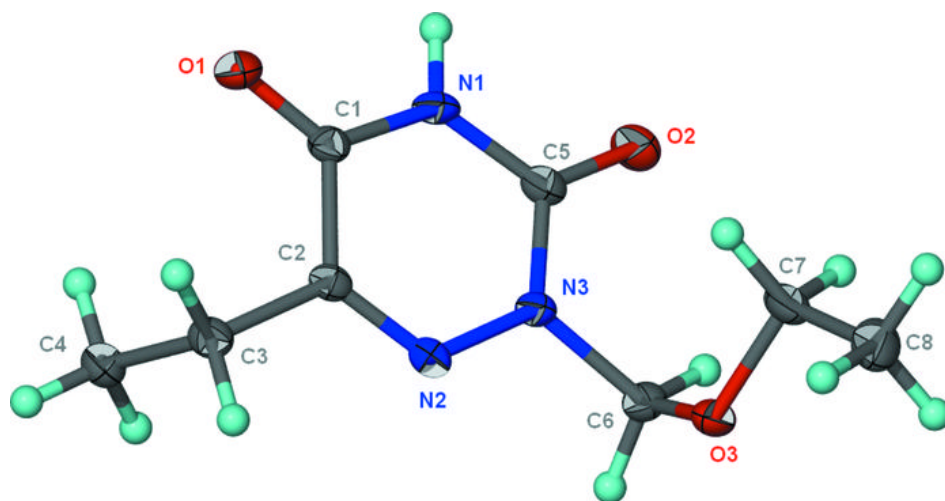


Fig. 2

