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2-{[(2-Methylprop-2-en-1-yl)oxy]methyl}-6-phenyl-2,3,4,5-tetrahydro-1,2,4-triazine-3,5-dione

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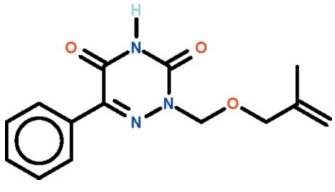
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.004 Å; R factor = 0.081; wR factor = 0.250; data-to-parameter ratio = 16.6.

The 1,2,4-triazine ring in the title compound, $C_{14}H_{15}N_3O_3$, is approximately planar (r.m.s. deviation = 0.019 Å); the C atom at the 6-position deviates by 0.026 (2) Å from the mean plane whereas the C atom at the 2-position deviates by 0.166 (4) Å in the opposite direction. The triazine ring is oriented at 8.60 (13)° with respect to the phenyl ring. The imino group is hydrogen-bond donor to the exocyclic O atom at the 3-position of an adjacent molecule, the hydrogen bond generating an inversion dimer.

Related literature

For the synthesis and antimicrobial activity of the title compound, see: El-Brollosy (2008).



Experimental

Crystal data

$C_{14}H_{15}N_3O_3$ M_r = 273.29
Triclinic, $P\overline{1}$
a = 4.6162 (5) Å
b = 11.7896 (14) Å
c = 12.5769 (10) Å
α = 81.588 (8)°
β = 85.836 (7)°
γ = 87.245 (9)°
V = 674.84 (12) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100 K
0.30 × 0.03 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.972, T_{max} = 0.997
9875 measured reflections
3088 independent reflections
1878 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.250
S = 1.01
3088 reflections
186 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.89 (1)	1.93 (1)	2.802 (3)	168 (4)

Symmetry code: (i) $-x+1, -y+1, -z+1$.

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).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5422).

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El-Brollosy, N. R. (2008). *Monatsh. Chem.* **139**, 1483–1490.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E**68**, o347 [doi:10.1107/S160053681105567X]

2-{[(2-Methylprop-2-en-1-yl)oxy]methyl}-6-phenyl-2,3,4,5-tetrahydro-1,2,4-triazine-3,5-dione

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S1. Comment

The compound (Scheme I) was synthesized for an evaluation of its antimicrobial activity (El-Brollosy, 2008). The 1,2,4-triazine ring of is planar (r.m.s. deviation 0.019 Å); the C atom at the 6-position deviates by 0.026 (2) Å from the mean plane whereas the C atom at the 2-position deviates from the mean plane by 0.166 (4) Å in the opposite direction (Fig. 1). The amino group is hydrogen-bond donor to the exocyclic O atom at the 3-position, the hydrogen bond generating a centrosymmetric dimer (Table 1, Fig. 2).

S2. Experimental

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

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 to 0.98 Å, U_{iso}(H) 1.2 to 1.5U_{eq}(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 refined with a distance restraint of N–H 0.88±0.01 Å; its temperature factor was refined.

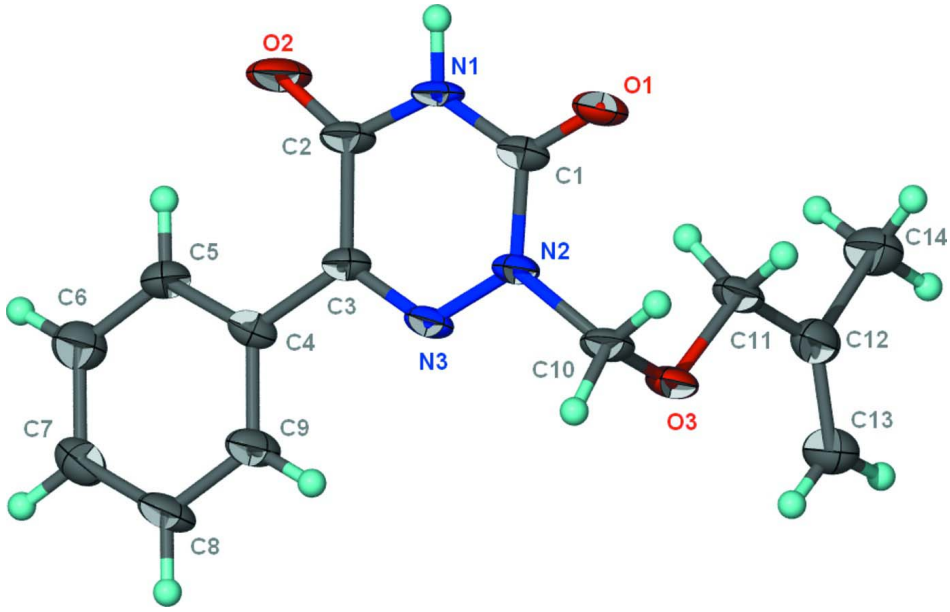

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{14}H_{15}N_3O_3$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

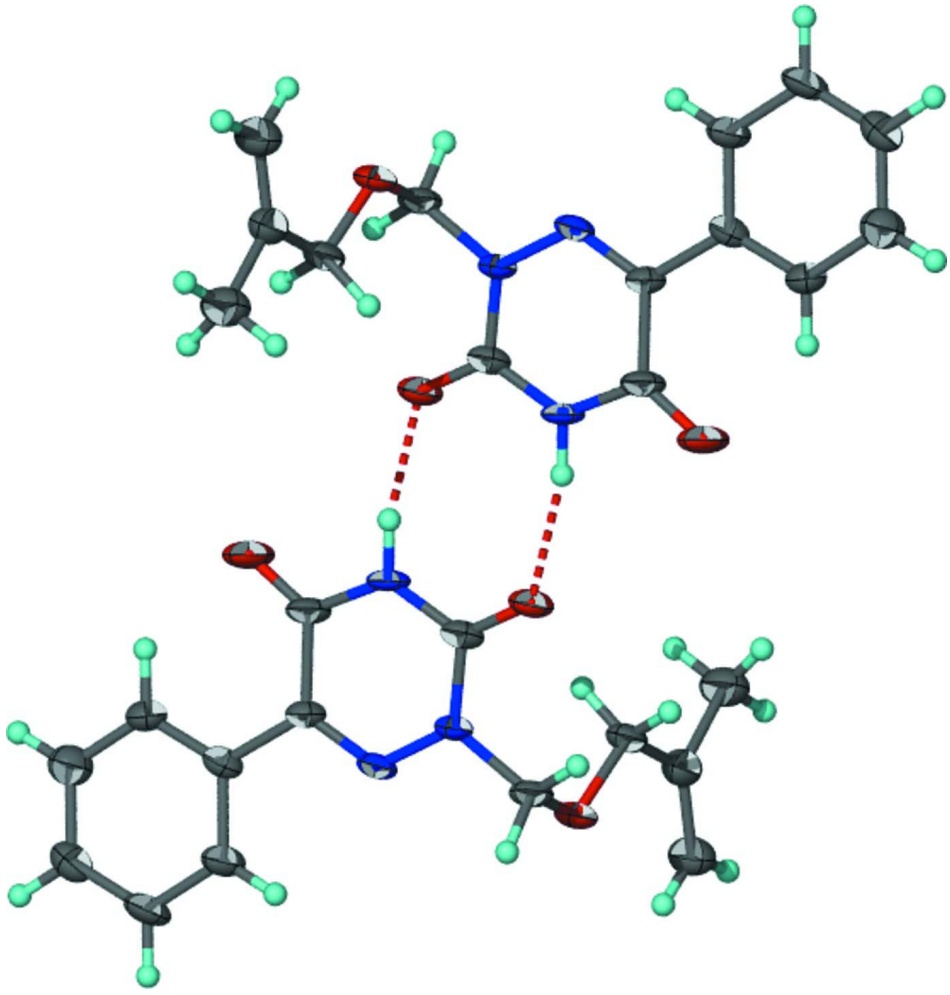

Figure 2

Dimeric hydrogen-bonded structure.

2-{[(2-Methylprop-2-en-1-yl)oxy]methyl}-6-phenyl-2,3,4,5-tetrahydro-1,2,4- triazine-3,5-dione

Crystal data

$C_{14}H_{15}N_3O_3$
$M_r = 273.29$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.6162\ (5)$ Å
$b = 11.7896\ (14)$ Å
$c = 12.5769\ (10)$ Å
$\alpha = 81.588\ (8)°$
$\beta = 85.836\ (7)°$
$\gamma = 87.245\ (9)°$
$V = 674.84\ (12)$ Å^3
$Z = 2$
$F(000) = 288$
$D_x = 1.345$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1917 reflections
$\theta = 2.6–27.5°$
$\mu = 0.10$ mm^{-1}
$T = 100$ K
Prism, colorless
0.30 × 0.03 × 0.03 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.972, T_{max} = 0.997
9875 measured reflections
3088 independent reflections
1878 reflections with $I > 2\sigma(I)$
R_{int} = 0.086
θ_{max} = 27.6°, θ_{min} = 2.6°
h = −6→6
k = −15→15
l = −16→16

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.081
$wR(F^2)$ = 0.250
S = 1.01
3088 reflections
186 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.1384P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.45 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{eq}
O1	0.5220 (4)	0.40726 (17)	0.61847 (14)	0.0268 (5)
O2	−0.1301 (4)	0.7060 (2)	0.55343 (15)	0.0370 (6)
O3	0.0363 (4)	0.24966 (17)	0.82369 (14)	0.0261 (5)
N1	0.1946 (5)	0.5578 (2)	0.58770 (16)	0.0224 (6)
H1	0.277 (7)	0.580 (3)	0.5224 (14)	0.062 (12)*
N2	0.1685 (5)	0.4378 (2)	0.74966 (16)	0.0219 (5)
N3	−0.0574 (4)	0.5017 (2)	0.78892 (16)	0.0216 (5)
C1	0.3099 (6)	0.4641 (2)	0.6493 (2)	0.0227 (6)
C2	−0.0396 (6)	0.6266 (3)	0.6174 (2)	0.0248 (6)
C3	−0.1586 (5)	0.5929 (2)	0.73057 (19)	0.0207 (6)
C4	−0.3984 (5)	0.6607 (2)	0.7819 (2)	0.0217 (6)
C5	−0.5471 (6)	0.7527 (2)	0.7267 (2)	0.0257 (6)
H5	−0.5003	0.7749	0.6520	0.031*
C6	−0.7664 (6)	0.8135 (3)	0.7801 (2)	0.0305 (7)
H6	−0.8691	0.8762	0.7415	0.037*
C7	−0.8330 (6)	0.7822 (3)	0.8889 (2)	0.0292 (7)
H7	−0.9804	0.8239	0.9252	0.035*
C8	−0.6862 (6)	0.6905 (3)	0.9448 (2)	0.0299 (7)
H8	−0.7345	0.6689	1.0195	0.036*
C9	−0.4675 (6)	0.6294 (3)	0.8926 (2)	0.0263 (7)
H9	−0.3655	0.5668	0.9318	0.032*
C10	0.2431 (6)	0.3324 (3)	0.8212 (2)	0.0248 (6)
H10A	0.4350	0.3013	0.7963	0.030*
H10B	0.2581	0.3506	0.8950	0.030*

C11	0.0328 (6)	0.2037 (3)	0.7240 (2)	0.0266 (7)
H11A	0.2318	0.1769	0.7022	0.032*
H11B	−0.0311	0.2649	0.6670	0.032*
C12	−0.1666 (6)	0.1064 (3)	0.7350 (2)	0.0297 (7)
C13	−0.2911 (7)	0.0595 (3)	0.8283 (2)	0.0357 (8)
H13A	−0.2548	0.0879	0.8928	0.043*
H13B	−0.4166	−0.0025	0.8307	0.043*
C14	−0.2130 (7)	0.0672 (3)	0.6292 (2)	0.0375 (8)
H14A	−0.3299	−0.0013	0.6420	0.056*
H14B	−0.0245	0.0489	0.5934	0.056*
H14C	−0.3148	0.1284	0.5833	0.056*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0280 (11)	0.0367 (12)	0.0136 (9)	0.0045 (9)	0.0047 (8)	−0.0017 (8)
O2	0.0420 (12)	0.0492 (15)	0.0141 (10)	0.0119 (11)	0.0064 (8)	0.0055 (9)
O3	0.0328 (11)	0.0321 (12)	0.0121 (9)	0.0010 (9)	0.0050 (7)	−0.0025 (8)
N1	0.0264 (12)	0.0306 (14)	0.0083 (10)	−0.0017 (10)	0.0040 (9)	0.0015 (10)
N2	0.0275 (12)	0.0259 (13)	0.0108 (10)	0.0025 (10)	0.0039 (9)	−0.0007 (9)
N3	0.0249 (12)	0.0287 (13)	0.0108 (10)	0.0010 (10)	0.0027 (8)	−0.0042 (9)
C1	0.0241 (14)	0.0313 (16)	0.0126 (12)	−0.0017 (12)	0.0007 (10)	−0.0040 (11)
C2	0.0284 (14)	0.0340 (17)	0.0106 (12)	0.0002 (12)	0.0005 (10)	0.0000 (11)
C3	0.0235 (14)	0.0255 (15)	0.0122 (12)	−0.0018 (11)	0.0011 (10)	−0.0003 (10)
C4	0.0203 (13)	0.0283 (16)	0.0165 (12)	−0.0005 (11)	0.0025 (10)	−0.0058 (11)
C5	0.0323 (15)	0.0263 (15)	0.0168 (13)	−0.0006 (12)	0.0022 (11)	0.0006 (11)
C6	0.0307 (16)	0.0345 (18)	0.0260 (15)	0.0010 (14)	−0.0010 (12)	−0.0046 (13)
C7	0.0299 (15)	0.0323 (17)	0.0266 (14)	−0.0006 (13)	0.0038 (12)	−0.0107 (13)
C8	0.0314 (16)	0.0422 (19)	0.0161 (13)	0.0044 (14)	0.0045 (11)	−0.0086 (13)
C9	0.0257 (14)	0.0360 (17)	0.0155 (13)	0.0053 (13)	0.0019 (10)	−0.0024 (12)
C10	0.0289 (14)	0.0339 (17)	0.0104 (11)	0.0030 (12)	−0.0002 (10)	−0.0009 (11)
C11	0.0333 (15)	0.0326 (17)	0.0128 (12)	0.0060 (13)	0.0015 (11)	−0.0037 (12)
C12	0.0353 (16)	0.0298 (17)	0.0223 (14)	0.0072 (13)	0.0012 (12)	−0.0024 (12)
C13	0.0461 (18)	0.0342 (18)	0.0258 (15)	−0.0044 (15)	0.0058 (13)	−0.0038 (13)
C14	0.0500 (19)	0.0383 (19)	0.0240 (15)	−0.0034 (16)	0.0003 (13)	−0.0045 (14)

Geometric parameters (Å, º)

O1—C1	1.230 (3)	C6—H6	0.9500
O2—C2	1.220 (3)	C7—C8	1.378 (4)
O3—C10	1.393 (3)	C7—H7	0.9500
O3—C11	1.438 (3)	C8—C9	1.393 (4)
N1—C1	1.362 (4)	C8—H8	0.9500
N1—C2	1.383 (4)	C9—H9	0.9500
N1—H1	0.888 (10)	C10—H10A	0.9900
N2—N3	1.362 (3)	C10—H10B	0.9900
N2—C1	1.379 (3)	C11—C12	1.490 (4)
N2—C10	1.466 (3)	C11—H11A	0.9900

N3—C3	1.297 (4)	C11—H11B	0.9900
C2—C3	1.493 (3)	C12—C13	1.325 (4)
C3—C4	1.498 (3)	C12—C14	1.503 (4)
C4—C5	1.381 (4)	C13—H13A	0.9500
C4—C9	1.405 (4)	C13—H13B	0.9500
C5—C6	1.401 (4)	C14—H14A	0.9800
C5—H5	0.9500	C14—H14B	0.9800
C6—C7	1.380 (4)	C14—H14C	0.9800
C10—O3—C11	113.35 (19)	C7—C8—H8	119.7
C1—N1—C2	126.5 (2)	C9—C8—H8	119.7
C1—N1—H1	119 (3)	C8—C9—C4	119.7 (3)
C2—N1—H1	115 (3)	C8—C9—H9	120.1
N3—N2—C1	124.4 (2)	C4—C9—H9	120.1
N3—N2—C10	114.3 (2)	O3—C10—N2	111.8 (2)
C1—N2—C10	121.2 (2)	O3—C10—H10A	109.3
C3—N3—N2	120.6 (2)	N2—C10—H10A	109.3
O1—C1—N1	123.2 (2)	O3—C10—H10B	109.3
O1—C1—N2	122.5 (3)	N2—C10—H10B	109.3
N1—C1—N2	114.2 (2)	H10A—C10—H10B	107.9
O2—C2—N1	120.4 (2)	O3—C11—C12	111.1 (2)
O2—C2—C3	126.1 (3)	O3—C11—H11A	109.4
N1—C2—C3	113.5 (2)	C12—C11—H11A	109.4
N3—C3—C2	120.7 (2)	O3—C11—H11B	109.4
N3—C3—C4	117.0 (2)	C12—C11—H11B	109.4
C2—C3—C4	122.4 (3)	H11A—C11—H11B	108.0
C5—C4—C9	119.2 (2)	C13—C12—C11	123.4 (3)
C5—C4—C3	123.4 (2)	C13—C12—C14	123.5 (3)
C9—C4—C3	117.3 (2)	C11—C12—C14	113.1 (2)
C4—C5—C6	120.5 (2)	C12—C13—H13A	120.0
C4—C5—H5	119.8	C12—C13—H13B	120.0
C6—C5—H5	119.8	H13A—C13—H13B	120.0
C7—C6—C5	119.9 (3)	C12—C14—H14A	109.5
C7—C6—H6	120.0	C12—C14—H14B	109.5
C5—C6—H6	120.0	H14A—C14—H14B	109.5
C8—C7—C6	120.1 (3)	C12—C14—H14C	109.5
C8—C7—H7	119.9	H14A—C14—H14C	109.5
C6—C7—H7	119.9	H14B—C14—H14C	109.5
C7—C8—C9	120.5 (3)		
C1—N2—N3—C3	1.5 (4)	C2—C3—C4—C5	6.4 (4)
C10—N2—N3—C3	−175.2 (2)	N3—C3—C4—C9	8.3 (4)
C2—N1—C1—O1	−178.9 (2)	C2—C3—C4—C9	−172.1 (2)
C2—N1—C1—N2	1.1 (4)	C9—C4—C5—C6	−0.6 (4)
N3—N2—C1—O1	176.5 (2)	C3—C4—C5—C6	−179.1 (2)
C10—N2—C1—O1	−7.1 (4)	C4—C5—C6—C7	0.6 (4)
N3—N2—C1—N1	−3.5 (4)	C5—C6—C7—C8	−0.6 (4)
C10—N2—C1—N1	172.9 (2)	C6—C7—C8—C9	0.7 (4)

C1—N1—C2—O2	−177.2 (2)	C7—C8—C9—C4	−0.7 (4)
C1—N1—C2—C3	2.8 (4)	C5—C4—C9—C8	0.7 (4)
N2—N3—C3—C2	3.0 (4)	C3—C4—C9—C8	179.2 (2)
N2—N3—C3—C4	−177.40 (19)	C11—O3—C10—N2	68.7 (3)
O2—C2—C3—N3	175.1 (3)	N3—N2—C10—O3	72.8 (2)
N1—C2—C3—N3	−4.9 (4)	C1—N2—C10—O3	−104.0 (3)
O2—C2—C3—C4	−4.5 (4)	C10—O3—C11—C12	173.3 (2)
N1—C2—C3—C4	175.5 (2)	O3—C11—C12—C13	−8.5 (4)
N3—C3—C4—C5	−173.3 (2)	O3—C11—C12—C14	171.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.89 (1)	1.93 (1)	2.802 (3)	168 (4)

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