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3-[(*R*)-1-Hydroxybutan-2-yl]-1,2,3-benzotriazin-4(3*H*)-one

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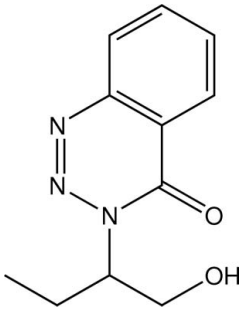
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.034; wR factor = 0.076; data-to-parameter ratio = 13.4.

The crystal structure of the title compound, $C_{11}H_{13}N_3O_2$, is stabilized by O—H···O hydrogen bonds, which link the molecules into chains along [100].

Related literature

For biological and synthetic applications of benzo-1,2,3-triazinones, see: Caliendo *et al.* (1999); Zheng *et al.* (2005); Vaisburg *et al.* (2004); Chollet *et al.* (2002); Le Diguarher *et al.* (2003); Clark *et al.* (1995); Carpino *et al.* (2004); Janout *et al.* (2003); Gierasch *et al.* (2000). For structures of benzo-1,2,3-triazinones, see: Hjortås *et al.* (1973); Hunt *et al.* (1983); Reingruber *et al.* (2009). For bond-length data, see: Allen *et al.* (1987). For the synthesis, see: Gómez *et al.* (2005).



Experimental

Crystal data

$C_{11}H_{13}N_3O_2$
M_r = 219.24
Orthorhombic, $P2_12_12_1$
a = 8.9668 (13) Å
b = 10.1506 (15) Å
c = 12.0238 (17) Å
V = 1094.4 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 298 K
0.32 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
9057 measured reflections
2000 independent reflections
1700 reflections with $I > 2\sigma(I)$
R_{int} = 0.044

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.076
S = 0.93
2000 reflections
149 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.11 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2···O1^{i}	0.85 (1)	2.03 (1)	2.8712 (19)	171 (2)

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZJ2097).

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3-[(*R*)-1-Hydroxybutan-2-yl]-1,2,3-benzotriazin-4(3*H*)-one

Fernando Rocha-Alonzo, David Morales-Morales, Simón Hernández-Ortega, Reyna Reyes-Martínez and Miguel Parra-Hake

S1. Comment

Benzo-1,2,3-triazinones are compounds widely investigated for their potential biological and chemical properties. These heterocyclic compounds have been studied as anesthetic (Caliendo *et al.*, 1999), anti-inflammatory (Zheng *et al.*, 2005), anticancer (Vaisburg *et al.*., 2004; Chollet *et al.*, 2002), and antitumoural (Le Diguarher *et al.*, 2003; Clark *et al.*, 1995) agents. In organic synthesis, 1,2,3-triazinones are used as an activating moiety in coupling agents for the preparation of peptides and amino acids (Carpino *et al.*, 2004; Janout *et al.*, 2003; Gierasch *et al.*, 2000). As result of its biological and synthetic importance, we have developed an alternative method for obtaining compounds with 1,2,3-triazinone moiety and in this paper we are describing the crystal structure of the title compound (**I**, Figure 1).

In the molecular structure of **I**, the N1=N2 bond [1.2636 (17) Å] is longer than the typical values for N=N double bonds (1.236 Å), whereas the N2–N3 bond [1.3735 (18) Å] is shorter than typical values for a N–N single bonds (1.404 Å) (Allen *et al.*., 1987). The structure of **I** shows co-planarity between two rings (1.30°). These measurements are in agreement with other benzo-1,2,3-triazinone crystal structure reports (Hjortås *et al.*, 1973; Hunt *et al.*, 1983; Reingruber *et al.*, 2009). Of interest to pharmaceutical applications, it has been suggested that co-planar structure in benzo-1,2,3-triazinones could give DNA-intercalating abilities such as those displayed by some anticancer agents (Reingruber *et al.*, 2009).

In the crystal structure, adjacent units are arranged into one-dimensional chain along [100] direction *via* O–H···O intermolecular hydrogen bonds (Figure 2 and Table 1).

S2. Experimental

The synthesis of the tittle compound included reagents and solvents of reagent grade, which were used without further purification. To a solution of 2-[(4*R*)-4-ethyl-4,5-dihydro-1,3-oxazol-2-yl]aniline (Gómez *et al.*, 2005) (0.89 g, 4.7 mmol, dissolved in 85 ml of methanol) was slowly added isoamyl nitrite (4.40 g, 37.6 mmol, 8 equiv) and the reaction mixture was stirred at room temperature until the disappearance of the aniline (followed by TLC, hexane/ethyl acetate, 3:1). The solvent was evaporated under reduced pressure to give a crude product that was purified by washing with petroleum ether and recrystallization from hexane/ethyl acetate. Crystalline colorless prisms of **I** were grown by slow diffusion of hexane over saturated ethyl acetate solutions of **I**. Yield > 99%, based on 2-[(4*R*)-4-Ethyl-4,5-dihydro-1,3-oxazol-2-yl]aniline; m.p., 89–90 °C. = -5.45° (*c* 0.22, MeOH). FTIR (KBr pellet, cm^{-1}): 3439, 1686, 1663, 1296. ^{1}H NMR [$(CD_3)_2CO$, 200 MHz] d 8.29 (ddd, *J* = 0.6, 1.5, 7.9 Hz, 2H), 8.16 (ddd, *J* = 0.6, 1.5, 8.1 Hz, 2H), 8.07 (ddd, *J* = 1.5, 7.0, 8.2 Hz, 2H), 7.91 (ddd,, *J* = 1.5, 7.0, 7.9 Hz, 2H), 5.22 (ddd, *J* = 5.1, 7.6, 15.5 Hz, 2H), 4.10 (dd, *J* = 8.4, 11.3 Hz, 2H), 3.96 (dd, J = 5.1, 11.3 Hz, 2H), 1.99 (dd, *J* = 7.5, 15.0 Hz, 4H), 0.90 (t, *J* = 7.4 Hz, 6H). ^{13}C NMR [$(CD_3)_2CO$, 50 MHz] d 156.6, 144.5, 135.8, 133.2, 128.8, 125.7, 120.3, 64.2, 62.3, 24.2, 10.8. ESI-HRMS:220.1091 (100), calculated for $[M+H]^+$, $C_{11}H_{14}N_3O_2^+$, 220.1081; 192.1025 (8), calculated for $[M+H—N_2]^+$, $C_{11}H_{14}NO_2^+$, 192.1019. Anal for $C_{11}H_{13}N_3O_2$ (%

Calcd./found) C, 60.26/60.73; H, 5.98/6.45; N, 19.17/19.47.

S3. Refinement

H atoms were included in calculated positions (C—H = 0.93 Å for aromatic H, C—H = 0.98 for methyn, C—H =0.97 Å for methylene H, and C—H= 0.96 Å for methyl H), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq} of the carrier atoms. The hydroxyl H atoms were located in a difference map and refined with O–H = 0.85±0.01 Å, and with U_{iso}(H) = 1.2U_{eq}(O).

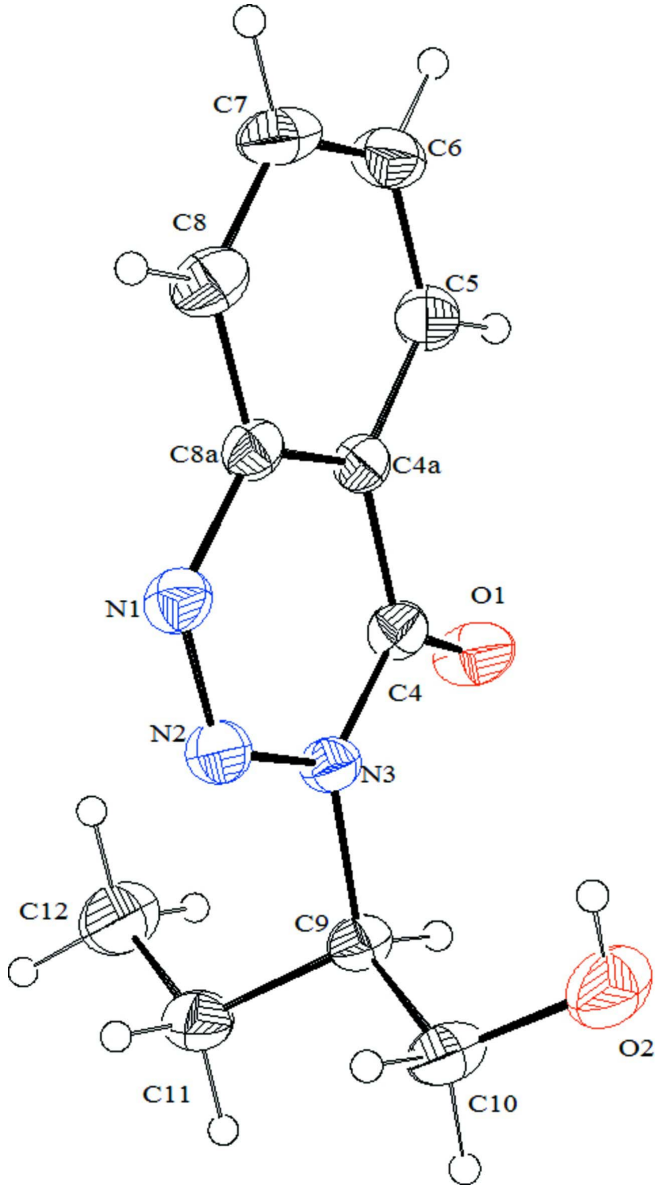

Figure 1

Molecular structure of (**I**) with displacement ellipsoids drawn at 50% probability.

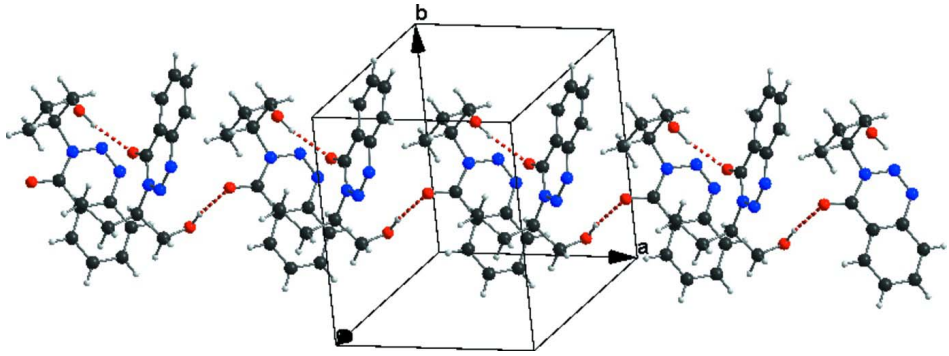

Figure 2

Packing of **I** showing the H-bonds. The molecules are forming a one-dimensional chain in the [100] direction. H-bonds are indicated by dashed lines.

3-[(*R*)-1-Hydroxybutan-2-yl]-1,2,3-benzotriazin-4(3*H*)-one

Crystal data

$C_{11}H_{13}N_3O_2$
$M_r = 219.24$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.9668$ (13) Å
$b = 10.1506$ (15) Å
$c = 12.0238$ (17) Å
$V = 1094.4$ (3) Å^3
$Z = 4$
$F(000) = 464$
$D_x = 1.331$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4276 reflections
$\theta = 2.6$–25.2°
$\mu = 0.09$ mm^{-1}
$T = 298$ K
Prism, colourless
0.32 × 0.10 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 0.83 pixels mm^{-1}
ω scans
9057 measured reflections
2000 independent reflections
1700 reflections with $I > 2\sigma(I)$
$R_{int} = 0.044$
$\theta_{max} = 25.4°$, $\theta_{min} = 2.6°$
$h = -10 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.076$
$S = 0.93$
2000 reflections
149 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
$w = 1/[\sigma^2(F_o^2) + (0.0412P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.11$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.56004 (14)	0.68985 (11)	1.02583 (10)	0.0584 (4)
N1	0.87770 (16)	0.64809 (13)	0.79106 (11)	0.0438 (4)
O2	0.82337 (18)	0.99854 (13)	0.99816 (15)	0.0797 (5)
H2	0.885 (2)	0.9362 (16)	0.992 (2)	0.096*
N2	0.80536 (16)	0.75426 (13)	0.80106 (11)	0.0430 (4)
N3	0.70312 (14)	0.76906 (12)	0.88520 (11)	0.0372 (3)
C4	0.66320 (18)	0.67321 (15)	0.96026 (14)	0.0386 (4)
C4A	0.75154 (18)	0.55429 (16)	0.95179 (13)	0.0362 (4)
C5	0.7338 (2)	0.44941 (16)	1.02537 (14)	0.0469 (4)
H5	0.6642	0.4546	1.0826	0.056*
C6	0.8194 (2)	0.33848 (17)	1.01301 (16)	0.0545 (5)
H6	0.8081	0.2686	1.0623	0.065*
C7	0.9226 (2)	0.32979 (18)	0.92741 (17)	0.0568 (5)
H7	0.9790	0.2535	0.9191	0.068*
C8	0.94229 (19)	0.43244 (17)	0.85511 (16)	0.0517 (5)
H8	1.0127	0.4266	0.7985	0.062*
C8A	0.85625 (17)	0.54564 (15)	0.86684 (13)	0.0382 (4)
C9	0.62783 (18)	0.89954 (14)	0.88408 (15)	0.0420 (4)
H9	0.5594	0.9021	0.9476	0.050*
C10	0.7406 (2)	1.00892 (17)	0.89991 (17)	0.0553 (5)
H10A	0.8088	1.0088	0.8372	0.066*
H10B	0.6886	1.0927	0.8998	0.066*
C11	0.5352 (2)	0.91729 (17)	0.77929 (16)	0.0559 (5)
H11A	0.4838	1.0012	0.7833	0.067*
H11B	0.6015	0.9202	0.7156	0.067*
C12	0.4222 (2)	0.80997 (19)	0.76146 (19)	0.0765 (7)
H12A	0.3540	0.8081	0.8230	0.092*
H12B	0.4722	0.7266	0.7560	0.092*
H12C	0.3681	0.8266	0.6940	0.092*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0643 (8)	0.0449 (7)	0.0660 (9)	0.0042 (7)	0.0294 (8)	0.0035 (6)
N1	0.0471 (8)	0.0409 (8)	0.0434 (8)	0.0020 (7)	0.0101 (7)	−0.0012 (7)
O2	0.0754 (11)	0.0612 (10)	0.1026 (12)	0.0097 (8)	−0.0365 (10)	−0.0201 (9)

N2	0.0473 (8)	0.0401 (8)	0.0415 (8)	0.0014 (7)	0.0075 (7)	0.0013 (7)
N3	0.0410 (8)	0.0319 (7)	0.0387 (8)	0.0029 (6)	0.0047 (7)	0.0002 (6)
C4	0.0396 (9)	0.0364 (9)	0.0398 (9)	−0.0021 (8)	0.0043 (8)	−0.0018 (8)
C4A	0.0380 (9)	0.0339 (8)	0.0366 (9)	−0.0030 (7)	−0.0024 (8)	−0.0013 (7)
C5	0.0532 (11)	0.0426 (10)	0.0448 (10)	−0.0046 (9)	−0.0004 (9)	0.0019 (8)
C6	0.0638 (12)	0.0392 (10)	0.0604 (12)	−0.0009 (9)	−0.0109 (10)	0.0083 (9)
C7	0.0538 (12)	0.0365 (10)	0.0800 (14)	0.0100 (9)	−0.0071 (11)	−0.0013 (10)
C8	0.0435 (10)	0.0465 (11)	0.0651 (12)	0.0061 (9)	0.0060 (9)	−0.0089 (10)
C8A	0.0379 (9)	0.0352 (9)	0.0416 (10)	−0.0030 (7)	−0.0017 (8)	−0.0044 (8)
C9	0.0457 (9)	0.0337 (9)	0.0467 (10)	0.0058 (7)	0.0009 (9)	−0.0019 (8)
C10	0.0587 (11)	0.0362 (10)	0.0711 (12)	0.0034 (8)	−0.0022 (12)	−0.0055 (9)
C11	0.0648 (12)	0.0429 (10)	0.0600 (12)	0.0132 (9)	−0.0118 (10)	−0.0007 (9)
C12	0.0765 (14)	0.0602 (13)	0.0927 (17)	0.0111 (12)	−0.0357 (14)	−0.0133 (12)

Geometric parameters (Å, º)

O1—C4	1.2271 (18)	C7—C8	1.368 (2)
N1—N2	1.2636 (17)	C7—H7	0.9300
N1—C8A	1.396 (2)	C8—C8A	1.391 (2)
O2—C10	1.399 (2)	C8—H8	0.9300
O2—H2	0.846 (9)	C9—C10	1.514 (2)
N2—N3	1.3735 (18)	C9—C11	1.520 (2)
N3—C4	1.3745 (19)	C9—H9	0.9800
N3—C9	1.4866 (19)	C10—H10A	0.9700
C4—C4A	1.447 (2)	C10—H10B	0.9700
C4A—C8A	1.390 (2)	C11—C12	1.503 (2)
C4A—C5	1.393 (2)	C11—H11A	0.9700
C5—C6	1.371 (2)	C11—H11B	0.9700
C5—H5	0.9300	C12—H12A	0.9600
C6—C7	1.387 (3)	C12—H12B	0.9600
C6—H6	0.9300	C12—H12C	0.9600
N2—N1—C8A	120.17 (13)	C8—C8A—N1	118.22 (15)
C10—O2—H2	109.5 (17)	N3—C9—C10	110.42 (13)
N1—N2—N3	120.40 (12)	N3—C9—C11	111.17 (14)
N2—N3—C4	125.45 (12)	C10—C9—C11	112.49 (14)
N2—N3—C9	113.20 (12)	N3—C9—H9	107.5
C4—N3—C9	121.22 (13)	C10—C9—H9	107.5
O1—C4—N3	121.38 (14)	C11—C9—H9	107.5
O1—C4—C4A	124.93 (15)	O2—C10—C9	113.91 (15)
N3—C4—C4A	113.68 (14)	O2—C10—H10A	108.8
C8A—C4A—C5	119.70 (15)	C9—C10—H10A	108.8
C8A—C4A—C4	118.29 (14)	O2—C10—H10B	108.8
C5—C4A—C4	122.01 (15)	C9—C10—H10B	108.8
C6—C5—C4A	119.66 (17)	H10A—C10—H10B	107.7
C6—C5—H5	120.2	C12—C11—C9	113.63 (15)
C4A—C5—H5	120.2	C12—C11—H11A	108.8
C5—C6—C7	120.42 (17)	C9—C11—H11A	108.8

C5—C6—H6	119.8	C12—C11—H11B	108.8
C7—C6—H6	119.8	C9—C11—H11B	108.8
C8—C7—C6	120.59 (17)	H11A—C11—H11B	107.7
C8—C7—H7	119.7	C11—C12—H12A	109.5
C6—C7—H7	119.7	C11—C12—H12B	109.5
C7—C8—C8A	119.55 (17)	H12A—C12—H12B	109.5
C7—C8—H8	120.2	C11—C12—H12C	109.5
C8A—C8—H8	120.2	H12A—C12—H12C	109.5
C4A—C8A—C8	120.07 (15)	H12B—C12—H12C	109.5
C4A—C8A—N1	121.71 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2···O1^{i}	0.85 (1)	2.03 (1)	2.8712 (19)	171 (2)

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