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tert-Butyl *N*-[(*S*)-3-isobutyl-2-oxooxetan-3-yl]carbamate

Lesław Sieroń,[a]* Adam Kudaj,[b] Aleksandra Olma[b] and Janina Karolak-Wojciechowska[a]

[a]Institute of General & Ecological Chemistry, Technical University of Łódź, Żeromskiego 116, 90-924 Łódź, Poland, and [b]Institute of Organic Chemistry, Technical University of Łódź, Żeromskiego 116, 90-924 Łódź, Poland
Correspondence e-mail: lsieron@p.lodz.pl

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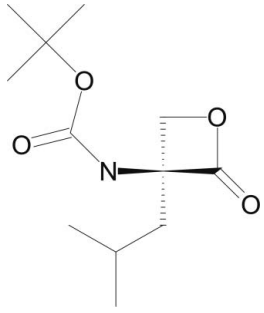
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.010 Å; R factor = 0.046; wR factor = 0.118; data-to-parameter ratio = 7.7.

The structure of the title compound, $C_{12}H_{21}NO_4$, contains two crystallographically independent molecules in the asymmetric unit. Molecules are linked into pseudosymmetric $R_2^2(8)$ dimers through two N—H···O hydrogen bonds. The dimers are connected by weak C—H···O interactions, resulting in a three-dimensional network.

Related literature

For related literature, see: Etter *et al.* (1990); Olma (2004); Olma & Kudaj (2005); Pansare *et al.* (1991); Smith & Goodman (2003); Yang & Romo (1999).



Experimental

Crystal data

$C_{12}H_{21}NO_4$
M_r = 243.30
Triclinic, $P\overline{1}$
a = 6.1642 (7) Å
b = 11.2018 (16) Å
c = 11.6915 (14) Å
α = 115.936 (14)°
β = 100.621 (10)°
γ = 95.362 (11)°
V = 699.58 (19) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 K
0.25 × 0.20 × 0.10 mm

Data collection

KUMA KM4CCD diffractometer
Absorption correction: none
7367 measured reflections
2451 independent reflections
1367 reflections with $I > 2\sigma(I)$
R_{int} = 0.032

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.118
S = 0.96
2451 reflections
317 parameters
3 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.12 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N8—H8···O29	0.86	2.12	2.937 (6)	158
N28—H28···O9	0.86	2.06	2.890 (6)	162
C12—H12*C*···O9	0.96	2.55	3.049 (9)	112
C13—H13*A*···O9	0.96	2.36	2.941 (11)	118
C27—H27*C*···O22	0.96	2.53	3.209 (9)	127
C32—H32*C*···O29	0.96	2.44	3.018 (9)	119

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL* and *POV-RAY* (Persistence of Vision, 2004); software used to prepare material for publication: *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SG2209).

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supporting information

Acta Cryst. (2008). E**64**, o207 [https://doi.org/10.1107/S1600536807063866]

tert-Butyl *N*-[(*S*)-3-isobutyl-2-oxooxetan-3-yl]carbamate

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S1. Comment

The discovery of a significant number of natural 2-oxetanones with very interesting biological activities has attracted much attention towards the preparation and its use as a synthetic intermediate (Yang & Romo, 1999). *N*-Protected-*α*-amino-*β*-lactones are useful intermediates for synthesis of *β*-substituted alanines *via* ring opening by various nucleophiles (Pansare *et al.*, 1991). Reactions of *N*-Boc-*α*-alkylserine *β*-lactones with soft sulfur nucleophiles (Olma, 2004) or with sodium azide (Olma & Kudaj, 2005) yield potentially interesting building blocks for medicinal chemistry.

As part of out studies in this area, we report here the crystal structure of the title compound, (I). In the crystal structure of (I) the asymmetric unit is composed of two molecules. Pairs of these molecules are connected into pesudocentrosymmetric dimers *via* N–H···N hydrogen bonds, forming eight-membered rings described by the $R_2^2(8)$ graph-set motif (Etter *et al.*, 1990) (Fig. 1). The same motif with comparable bond lengths is also observed in the structure of *α*-methyl analog (Smith & Goodman, 2003). The molecules in (I) differ mainly in the orientation of isobutyl substituents (Fig. 2), as indicated by torsion angles of -172.2 (6) and 85.9 (7)° for C2—C3—C5—C6 and C22—C23—C25—C26, respectively.

S2. Experimental

The title compound was synthesized by treating complex of triphenylphosphine (525 mg, 2 mmol) and diethyldiazadicarboxylate in dry tetrahydrofurane with solution of Boc-(*S*)-*iso*-butylserine (*N*-Boc-(*S*)-*α*-hydroxymethylleucine) in dry THF (698 mg, 2 mmol) at 0°C. After stirring 1 hr at 0°C and then 16 hrs at room temperature, THF was removed *in vacuo* and the crude product was purified by flash chromatography on silica gel 60 (230–400 mesh), using ethyl acetate-*n*-hexane (1:1) as eluent. The *N*-Boc-(*S*)-*α*-benzylserine lactone was obtained in 95% yield. White crystals of (I) suitable for X-ray investigation were grown from chloroform.

S3. Refinement

In the absence of significant anomalous scattering effects, Friedel pairs were merged. The absolute configuration was assigned consistent with the starting material. All H atoms were included in calculated positions and treated as riding, C–H = 0.96–0.98 and N–H = 0.86 Å with U_{iso}(H) = 1.2 or 1.5U_{eq}(C) and 1.2U_{eq}(N).

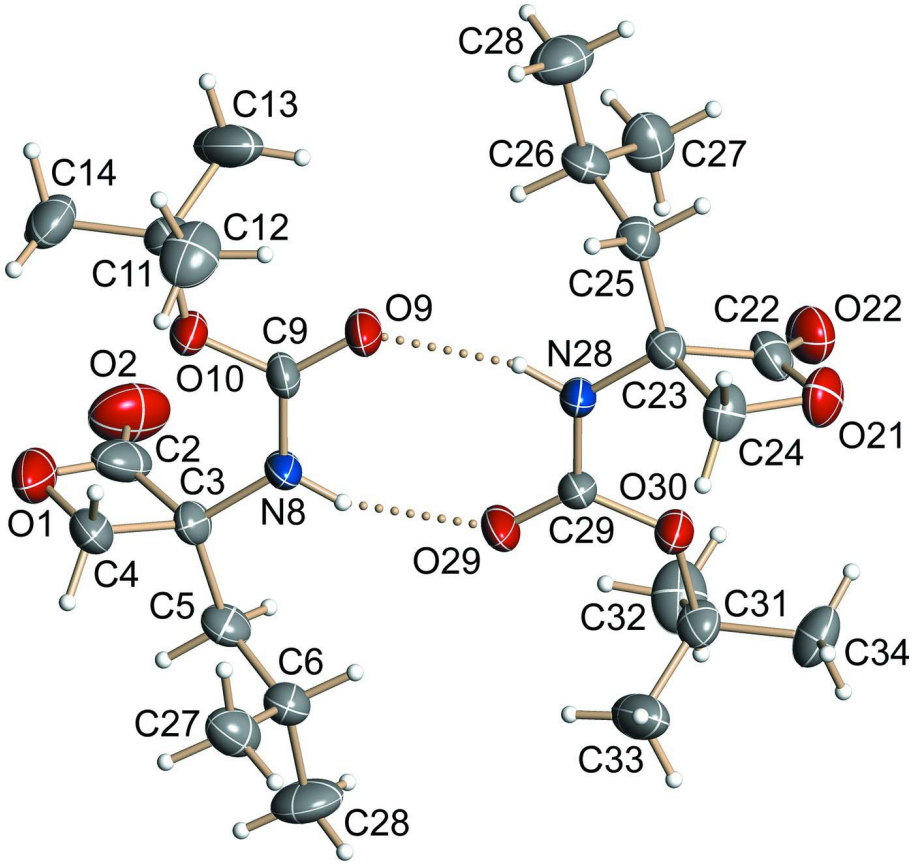

Figure 1

The structure of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dotted lines indicate hydrogen bonds.

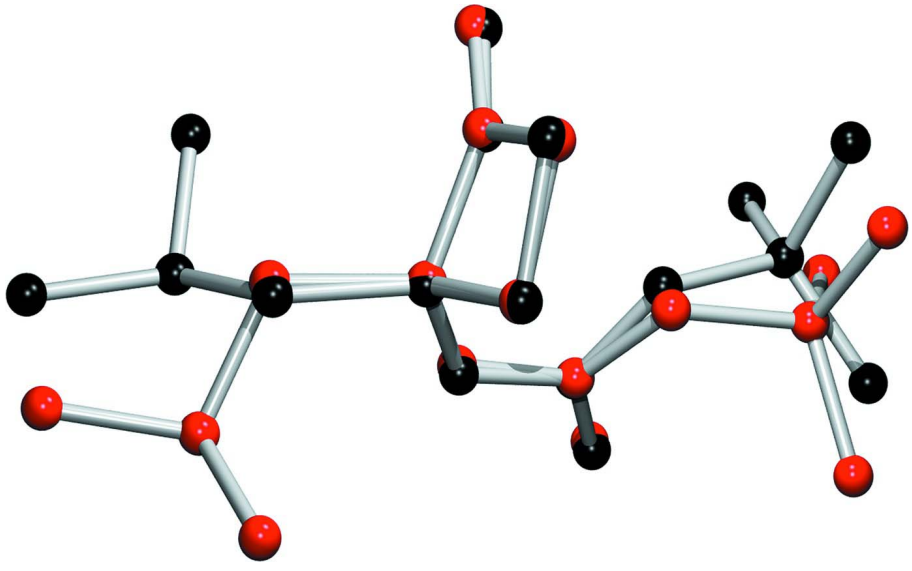

Figure 2

A least-squares overlay of the two independent molecules of (I), fitting on the central eight atoms. H atoms have been omitted.

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Crystal data

$C_{12}H_{21}NO_4$
$M_r = 243.30$
Triclinic, $P\overline{1}$
Hall symbol: P 1
$a = 6.1642\ (7)$ Å
$b = 11.2018\ (16)$ Å
$c = 11.6915\ (14)$ Å
$\alpha = 115.936\ (14)°$
$\beta = 100.621\ (10)°$
$\gamma = 95.362\ (11)°$
$V = 699.58\ (19)$ Å^3
$Z = 2$
$F(000) = 264$
$D_x = 1.155$ Mg m^{-3}
Melting point = 382–383 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4065 reflections
$\theta = 3.4–26.0°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Rectangular plate, colourless
0.25 × 0.20 × 0.10 mm

Data collection

KUMA KM4CCD diffractometer
Radiation source: CX-Mo12x0.4-S Seifert Mo tube
Graphite monochromator
Detector resolution: 8.2356 pixels mm^{-1}
ω scans
7367 measured reflections
2451 independent reflections
1367 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 25.0°$, $\theta_{min} = 3.4°$
$h = -7\rightarrow7$
$k = -13\rightarrow12$
$l = -13\rightarrow13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.118$
$S = 0.96$
2451 reflections
317 parameters
3 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: structure-invariant direct methods
Hydrogen site location: difference Fourier map
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.062P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.12$ e Å^{-3}
$\Delta\rho_{min} = -0.12$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement on F^2 for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	1.4399 (10)	0.1757 (5)	0.4830 (5)	0.109 (2)
O2	1.1035 (11)	0.2386 (5)	0.4645 (6)	0.142 (3)
O9	1.1871 (6)	0.1402 (4)	0.0465 (4)	0.0732 (16)

O10	1.4040 (5)	0.2338 (4)	0.2538 (3)	0.0632 (12)
N8	1.1595 (7)	0.0423 (5)	0.1767 (4)	0.0608 (17)
C2	1.2281 (16)	0.1651 (7)	0.4225 (7)	0.094 (4)
C3	1.2177 (8)	0.0381 (6)	0.3000 (5)	0.056 (2)
C4	1.4709 (10)	0.0600 (7)	0.3684 (7)	0.080 (3)
C5	1.0644 (10)	−0.0818 (6)	0.2932 (6)	0.065 (2)
C6	1.0612 (10)	−0.2220 (6)	0.1830 (6)	0.071 (2)
C7	1.2908 (11)	−0.2562 (7)	0.1790 (6)	0.084 (3)
C8	0.9089 (13)	−0.3259 (7)	0.1990 (9)	0.116 (4)
C9	1.2485 (9)	0.1394 (6)	0.1522 (6)	0.057 (2)
C11	1.5447 (9)	0.3418 (6)	0.2464 (6)	0.0630 (19)
C12	1.6836 (12)	0.2758 (8)	0.1487 (7)	0.108 (3)
C13	1.4015 (13)	0.4276 (8)	0.2093 (10)	0.129 (4)
C14	1.6964 (11)	0.4200 (7)	0.3820 (7)	0.093 (3)
O21	0.5614 (6)	−0.1756 (4)	−0.4839 (4)	0.0800 (16)
O22	0.3389 (6)	−0.0513 (4)	−0.3675 (4)	0.0866 (16)
O29	0.7503 (6)	−0.1196 (4)	−0.0326 (4)	0.0771 (17)
O30	0.5507 (5)	−0.2205 (4)	−0.2453 (3)	0.0622 (16)
N28	0.8048 (6)	−0.0325 (4)	−0.1694 (4)	0.0567 (17)
C22	0.5175 (10)	−0.0792 (7)	−0.3759 (6)	0.066 (3)
C23	0.7590 (8)	−0.0328 (6)	−0.2933 (6)	0.052 (2)
C24	0.7917 (9)	−0.1606 (6)	−0.4128 (6)	0.071 (2)
C25	0.8812 (9)	0.0973 (5)	−0.2828 (6)	0.058 (2)
C26	0.8569 (10)	0.2326 (6)	−0.1764 (6)	0.067 (2)
C27	0.6166 (11)	0.2527 (7)	−0.1836 (7)	0.091 (3)
C28	1.0010 (13)	0.3476 (7)	−0.1818 (8)	0.104 (3)
C29	0.7063 (9)	−0.1247 (6)	−0.1397 (6)	0.055 (2)
C31	0.3953 (9)	−0.3211 (6)	−0.2350 (6)	0.063 (2)
C32	0.2568 (11)	−0.2483 (9)	−0.1438 (8)	0.107 (3)
C33	0.5216 (11)	−0.4104 (8)	−0.1981 (9)	0.108 (3)
C34	0.2499 (10)	−0.3999 (7)	−0.3762 (6)	0.091 (3)
H4A	1.50880	−0.01380	0.38540	0.0960*
H4B	1.57410	0.08380	0.32490	0.0960*
H5A	1.10960	−0.08350	0.37640	0.0780*
H5B	0.91190	−0.06560	0.28400	0.0780*
H6	0.99370	−0.22450	0.09910	0.0850*
H7A	1.36120	−0.25310	0.26080	0.1260*
H7B	1.38140	−0.19170	0.16490	0.1260*
H7C	1.27650	−0.34540	0.10860	0.1260*
H8	1.05900	−0.02320	0.11360	0.0730*
H8A	0.90490	−0.41500	0.13110	0.1740*
H8B	0.75960	−0.30710	0.19250	0.1740*
H8C	0.96630	−0.32100	0.28330	0.1740*
H12A	1.74390	0.20690	0.16460	0.1610*
H12B	1.80490	0.34300	0.15920	0.1610*
H12C	1.58960	0.23580	0.06080	0.1610*
H13A	1.30920	0.37430	0.12130	0.1930*
H13B	1.49690	0.50330	0.21410	0.1930*

H13C	1.30720	0.45980	0.26880	0.1930*
H14A	1.60960	0.43350	0.44550	0.1400*
H14B	1.76670	0.50620	0.39410	0.1400*
H14C	1.80990	0.37030	0.39360	0.1400*
H24A	0.90530	−0.14110	−0.45310	0.0860*
H24B	0.81610	−0.23510	−0.39400	0.0860*
H25A	0.83120	0.09650	−0.36690	0.0700*
H25B	1.04040	0.09470	−0.27000	0.0700*
H26	0.91720	0.23590	−0.09120	0.0800*
H27A	0.55490	0.25370	−0.26470	0.1370*
H27B	0.61350	0.33720	−0.11110	0.1370*
H27C	0.52870	0.17990	−0.17940	0.1370*
H28	0.90560	0.03320	−0.10670	0.0680*
H28A	0.94980	0.34450	−0.26590	0.1560*
H28B	1.15540	0.33770	−0.16910	0.1560*
H28C	0.98850	0.43280	−0.11380	0.1560*
H32A	0.14490	−0.31310	−0.14260	0.1600*
H32B	0.18430	−0.19110	−0.17350	0.1600*
H32C	0.35230	−0.19400	−0.05690	0.1600*
H33A	0.63820	−0.43120	−0.24390	0.1630*
H33B	0.42080	−0.49270	−0.22130	0.1630*
H33C	0.58780	−0.36520	−0.10510	0.1630*
H34A	0.34100	−0.44600	−0.43350	0.1370*
H34B	0.18640	−0.33820	−0.40280	0.1370*
H34C	0.13090	−0.46490	−0.38080	0.1370*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.150 (5)	0.095 (4)	0.059 (3)	−0.023 (3)	−0.016 (3)	0.040 (3)
O2	0.215 (6)	0.089 (3)	0.128 (4)	0.045 (4)	0.085 (4)	0.038 (3)
O9	0.078 (2)	0.077 (3)	0.055 (3)	−0.016 (2)	−0.014 (2)	0.040 (2)
O10	0.064 (2)	0.063 (2)	0.052 (2)	−0.014 (2)	−0.005 (2)	0.030 (2)
N8	0.066 (3)	0.060 (3)	0.055 (3)	−0.011 (2)	−0.008 (2)	0.039 (3)
C2	0.149 (8)	0.068 (5)	0.081 (6)	0.021 (5)	0.033 (5)	0.048 (4)
C3	0.070 (4)	0.058 (4)	0.037 (3)	0.007 (3)	0.006 (3)	0.024 (3)
C4	0.091 (5)	0.077 (4)	0.071 (4)	0.000 (4)	−0.007 (3)	0.048 (4)
C5	0.077 (4)	0.070 (4)	0.066 (4)	0.014 (3)	0.023 (3)	0.046 (4)
C6	0.080 (4)	0.064 (4)	0.071 (4)	0.006 (3)	0.005 (3)	0.040 (4)
C7	0.112 (5)	0.079 (4)	0.073 (4)	0.032 (4)	0.028 (4)	0.042 (4)
C8	0.125 (6)	0.072 (4)	0.158 (8)	−0.008 (4)	0.036 (5)	0.064 (5)
C9	0.050 (3)	0.068 (4)	0.048 (4)	0.001 (3)	−0.006 (3)	0.033 (4)
C11	0.062 (3)	0.049 (3)	0.071 (4)	−0.006 (3)	0.004 (3)	0.030 (3)
C12	0.097 (5)	0.123 (6)	0.097 (6)	−0.009 (5)	0.037 (4)	0.046 (5)
C13	0.108 (6)	0.082 (5)	0.203 (10)	0.003 (5)	−0.002 (6)	0.091 (6)
C14	0.095 (5)	0.075 (4)	0.080 (5)	−0.023 (4)	0.001 (4)	0.027 (4)
O21	0.087 (3)	0.089 (3)	0.041 (2)	0.005 (2)	0.007 (2)	0.016 (2)
O22	0.063 (2)	0.100 (3)	0.093 (3)	0.014 (2)	0.004 (2)	0.048 (2)

O29	0.086 (3)	0.082 (3)	0.061 (3)	−0.016 (2)	−0.014 (2)	0.050 (3)
O30	0.063 (2)	0.063 (3)	0.053 (3)	−0.010 (2)	−0.002 (2)	0.031 (2)
N28	0.060 (3)	0.053 (3)	0.050 (3)	−0.008 (2)	−0.006 (2)	0.029 (2)
C22	0.056 (4)	0.089 (5)	0.065 (4)	0.012 (3)	0.008 (3)	0.050 (4)
C23	0.042 (3)	0.061 (4)	0.058 (4)	0.002 (3)	0.006 (3)	0.035 (3)
C24	0.060 (3)	0.082 (4)	0.054 (4)	0.007 (3)	0.011 (3)	0.019 (3)
C25	0.059 (3)	0.064 (4)	0.055 (4)	0.008 (3)	0.013 (3)	0.032 (3)
C26	0.077 (4)	0.064 (4)	0.070 (4)	0.011 (3)	0.014 (3)	0.043 (3)
C27	0.094 (5)	0.094 (5)	0.077 (5)	0.032 (4)	0.018 (3)	0.031 (4)
C28	0.123 (6)	0.074 (5)	0.112 (6)	−0.001 (4)	0.029 (4)	0.044 (4)
C29	0.056 (4)	0.047 (4)	0.055 (4)	−0.001 (3)	−0.002 (3)	0.027 (4)
C31	0.047 (3)	0.072 (4)	0.067 (4)	−0.003 (3)	0.008 (3)	0.035 (4)
C32	0.078 (5)	0.151 (7)	0.086 (5)	0.010 (5)	0.029 (4)	0.050 (5)
C33	0.084 (4)	0.090 (5)	0.157 (7)	0.000 (4)	−0.012 (5)	0.082 (5)
C34	0.066 (4)	0.085 (5)	0.083 (5)	−0.013 (4)	−0.002 (4)	0.017 (4)

Geometric parameters (Å, º)

O1—C2	1.333 (11)	C12—H12A	0.96
O1—C4	1.461 (9)	C12—H12B	0.96
O2—C2	1.189 (12)	C12—H12C	0.96
O9—C9	1.228 (8)	C13—H13B	0.96
O10—C9	1.319 (7)	C13—H13A	0.96
O10—C11	1.465 (8)	C13—H13C	0.96
O21—C22	1.350 (8)	C14—H14B	0.96
O21—C24	1.460 (7)	C14—H14C	0.96
O22—C22	1.182 (8)	C14—H14A	0.96
O29—C29	1.206 (8)	C22—C23	1.518 (8)
O30—C31	1.470 (8)	C23—C24	1.565 (9)
O30—C29	1.345 (7)	C23—C25	1.520 (10)
N8—C3	1.443 (7)	C25—C26	1.526 (9)
N8—C9	1.336 (9)	C26—C27	1.512 (9)
N8—H8	0.8600	C26—C28	1.527 (11)
N28—C23	1.422 (8)	C31—C33	1.481 (12)
N28—C29	1.353 (9)	C31—C34	1.530 (9)
N28—H28	0.8600	C31—C32	1.495 (10)
C2—C3	1.499 (10)	C24—H24A	0.97
C3—C5	1.531 (10)	C24—H24B	0.97
C3—C4	1.560 (8)	C25—H25A	0.97
C5—C6	1.530 (9)	C25—H25B	0.97
C6—C8	1.524 (11)	C26—H26	0.98
C6—C7	1.504 (10)	C27—H27A	0.96
C11—C14	1.497 (9)	C27—H27B	0.96
C11—C13	1.508 (12)	C27—H27C	0.96
C11—C12	1.525 (10)	C28—H28A	0.96
C4—H4B	0.97	C28—H28B	0.96
C4—H4A	0.97	C28—H28C	0.96
C5—H5B	0.97	C32—H32A	0.96

C5—H5A	0.97	C32—H32B	0.96
C6—H6	0.98	C32—H32C	0.96
C7—H7A	0.96	C33—H33A	0.96
C7—H7C	0.96	C33—H33B	0.96
C7—H7B	0.96	C33—H33C	0.96
C8—H8B	0.96	C34—H34A	0.96
C8—H8C	0.96	C34—H34B	0.96
C8—H8A	0.96	C34—H34C	0.96

C2—O1—C4	92.1 (5)	H14A—C14—H14B	109
C9—O10—C11	122.9 (5)	C11—C14—H14A	109
C22—O21—C24	91.9 (4)	H14B—C14—H14C	110
C29—O30—C31	121.5 (4)	C11—C14—H14B	110
C3—N8—C9	125.5 (5)	C11—C14—H14C	109
C3—N8—H8	117	O21—C22—O22	125.3 (6)
C9—N8—H8	117	O22—C22—C23	139.1 (6)
C23—N28—C29	126.8 (5)	O21—C22—C23	95.6 (5)
C23—N28—H28	117	N28—C23—C24	116.9 (6)
C29—N28—H28	117	N28—C23—C22	118.4 (5)
O1—C2—C3	96.4 (7)	C22—C23—C25	114.2 (6)
O1—C2—O2	126.8 (7)	N28—C23—C25	111.0 (5)
O2—C2—C3	136.8 (8)	C22—C23—C24	81.9 (5)
N8—C3—C4	119.1 (5)	C24—C23—C25	111.6 (5)
C2—C3—C5	110.7 (5)	O21—C24—C23	89.3 (4)
N8—C3—C5	109.9 (5)	C23—C25—C26	118.8 (5)
N8—C3—C2	117.4 (6)	C25—C26—C28	109.3 (5)
C4—C3—C5	115.0 (6)	C25—C26—C27	114.1 (5)
C2—C3—C4	82.3 (5)	C27—C26—C28	110.6 (6)
O1—C4—C3	88.8 (5)	O29—C29—N28	124.3 (6)
C3—C5—C6	116.7 (5)	O29—C29—O30	125.2 (6)
C5—C6—C8	107.9 (6)	O30—C29—N28	110.5 (5)
C7—C6—C8	111.0 (6)	O30—C31—C33	110.2 (5)
C5—C6—C7	113.8 (5)	O30—C31—C34	101.3 (5)
O9—C9—O10	124.5 (6)	C32—C31—C34	110.5 (5)
O9—C9—N8	122.8 (6)	C33—C31—C34	110.5 (6)
O10—C9—N8	112.8 (5)	C32—C31—C33	114.9 (7)
C12—C11—C14	109.6 (5)	O30—C31—C32	108.6 (6)
O10—C11—C14	103.5 (5)	O21—C24—H24A	114
C12—C11—C13	112.7 (6)	O21—C24—H24B	114
C13—C11—C14	112.1 (7)	C23—C24—H24A	114
O10—C11—C13	110.6 (5)	C23—C24—H24B	114
O10—C11—C12	107.8 (6)	H24A—C24—H24B	111
C3—C4—H4A	114	C23—C25—H25A	108
C3—C4—H4B	114	C23—C25—H25B	108
O1—C4—H4B	114	C26—C25—H25A	108
O1—C4—H4A	114	C26—C25—H25B	108
H4A—C4—H4B	111	H25A—C25—H25B	107
H5A—C5—H5B	107	C25—C26—H26	107

C6—C5—H5B	108	C27—C26—H26	108
C3—C5—H5A	108	C28—C26—H26	108
C6—C5—H5A	108	C26—C27—H27A	110
C3—C5—H5B	108	C26—C27—H27B	109
C7—C6—H6	108	C26—C27—H27C	109
C8—C6—H6	108	H27A—C27—H27B	110
C5—C6—H6	108	H27A—C27—H27C	109
C6—C7—H7C	109	H27B—C27—H27C	109
C6—C7—H7A	110	C26—C28—H28A	109
H7A—C7—H7B	109	C26—C28—H28B	109
H7B—C7—H7C	109	C26—C28—H28C	109
H7A—C7—H7C	110	H28A—C28—H28B	109
C6—C7—H7B	109	H28A—C28—H28C	110
C6—C8—H8A	109	H28B—C28—H28C	109
C6—C8—H8B	109	C31—C32—H32A	109
H8B—C8—H8C	110	C31—C32—H32B	109
H8A—C8—H8B	109	C31—C32—H32C	109
C6—C8—H8C	109	H32A—C32—H32B	110
H8A—C8—H8C	109	H32A—C32—H32C	110
C11—C12—H12B	110	H32B—C32—H32C	109
C11—C12—H12C	110	C31—C33—H33A	110
C11—C12—H12A	109	C31—C33—H33B	109
H12A—C12—H12B	109	C31—C33—H33C	109
H12A—C12—H12C	109	H33A—C33—H33B	109
H12B—C12—H12C	109	H33A—C33—H33C	109
C11—C13—H13C	109	H33B—C33—H33C	109
H13A—C13—H13B	109	C31—C34—H34A	110
H13A—C13—H13C	109	C31—C34—H34B	109
C11—C13—H13B	109	C31—C34—H34C	109
H13B—C13—H13C	109	H34A—C34—H34B	109
C11—C13—H13A	109	H34A—C34—H34C	109
H14A—C14—H14C	109	H34B—C34—H34C	109

C4—O1—C2—O2	−176.6 (9)	O1—C2—C3—C5	109.5 (6)
C4—O1—C2—C3	4.7 (6)	O2—C2—C3—C5	−69.0 (12)
C2—O1—C4—C3	−4.5 (6)	O1—C2—C3—N8	−123.3 (6)
C11—O10—C9—O9	−8.8 (9)	O2—C2—C3—C4	177.1 (11)
C11—O10—C9—N8	172.5 (5)	O2—C2—C3—N8	58.2 (13)
C9—O10—C11—C12	−62.4 (7)	N8—C3—C5—C6	56.6 (7)
C9—O10—C11—C13	61.2 (8)	C4—C3—C5—C6	−81.1 (7)
C9—O10—C11—C14	−178.5 (5)	C2—C3—C5—C6	−172.2 (6)
C22—O21—C24—C23	−8.8 (5)	N8—C3—C4—O1	121.1 (6)
C24—O21—C22—O22	−172.7 (8)	C2—C3—C4—O1	4.0 (5)
C24—O21—C22—C23	9.1 (6)	C5—C3—C4—O1	−105.3 (6)
C31—O30—C29—O29	8.2 (9)	C3—C5—C6—C8	177.1 (6)
C31—O30—C29—N28	−170.7 (5)	C3—C5—C6—C7	53.4 (8)
C29—O30—C31—C32	61.1 (7)	O22—C22—C23—C24	173.7 (11)
C29—O30—C31—C33	−65.5 (7)	O22—C22—C23—C25	−76.2 (12)

C29—O30—C31—C34	177.5 (5)	O21—C22—C23—N28	−124.8 (6)
C9—N8—C3—C2	47.2 (8)	O21—C22—C23—C24	−8.6 (5)
C3—N8—C9—O9	−177.5 (5)	O21—C22—C23—C25	101.5 (6)
C3—N8—C9—O10	1.3 (8)	O22—C22—C23—N28	57.4 (14)
C9—N8—C3—C4	−49.5 (9)	N28—C23—C24—O21	125.8 (5)
C9—N8—C3—C5	174.8 (5)	N28—C23—C25—C26	−51.2 (7)
C29—N28—C23—C25	172.4 (5)	C22—C23—C25—C26	85.9 (7)
C23—N28—C29—O30	−0.8 (8)	C24—C23—C25—C26	176.6 (5)
C29—N28—C23—C24	−58.1 (7)	C25—C23—C24—O21	−105.0 (5)
C23—N28—C29—O29	−179.6 (6)	C22—C23—C24—O21	7.9 (5)
C29—N28—C23—C22	37.4 (9)	C23—C25—C26—C28	177.8 (6)
O1—C2—C3—C4	−4.4 (6)	C23—C25—C26—C27	−57.9 (8)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N8—H8···O29	0.86	2.12	2.937 (6)	158
N28—H28···O9	0.86	2.06	2.890 (6)	162
C12—H12*C*···O9	0.96	2.55	3.049 (9)	112
C13—H13*A*···O9	0.96	2.36	2.941 (11)	118
C27—H27*C*···O22	0.96	2.53	3.209 (9)	127
C32—H32*C*···O29	0.96	2.44	3.018 (9)	119