Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-2-(Cyclohexylmethylene)succinic acid

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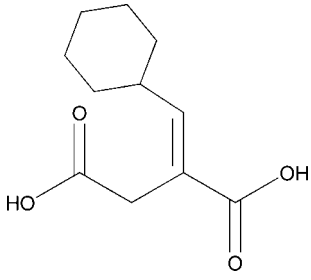
Received 6 November 2007; accepted 5 December 2007

Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.058; wR factor = 0.136; data-to-parameter ratio = 14.0.

The title compound, $C_{11}H_{16}O_4$, crystallizes with three molecules in the asymmetric unit. The cyclohexane ring adopts a chair conformation. Intermolecular O—H···O hydrogen bonds are observed and these help to establish the crystal packing.

Related literature

For related literature, see: Stobbe (1893).



Experimental

Crystal data

$C_{11}H_{16}O_4$
M_r = 212.24
Monoclinic, $P2_1/c$
a = 10.530 (2) Å
b = 18.762 (4) Å
c = 16.982 (3) Å
β = 91.42 (3)°
V = 3354.2 (12) Å^3
Z = 12
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 113 (2) K
0.10 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.981, T_{max} = 0.994
20420 measured reflections
5924 independent reflections
4780 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.136
S = 1.11
5924 reflections
424 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O12—H12···O11^{i}	0.87 (2)	1.78 (2)	2.656 (2)	175 (3)
O10—H10···O9^{ii}	0.95 (2)	1.63 (2)	2.586 (2)	175 (3)
O8—H8···O4^{iii}	0.95 (3)	1.70 (3)	2.642 (2)	172 (3)
O5—H5···O2	0.92 (2)	1.68 (2)	2.594 (2)	175 (3)
O3—H3···O7^{iv}	0.90 (2)	1.75 (2)	2.644 (2)	173 (3)
O1—H1···O6	0.93 (2)	1.66 (2)	2.591 (2)	175 (3)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: GW2033).

References

Bruker (1997). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.
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Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stobbe, H. (1893). *Ber. Dtsch. Chem. Ges.* **26**, 2312.

  doi:10.1107/S1600536807065695

supporting information

Acta Cryst. (2008). E**64**, o336 [https://doi.org/10.1107/S1600536807065695]

(*E*)-2-(Cyclohexylmethylene)succinic acid

Wei Wang, Yi Deng, Xiao-hui Cao, Yue Wang and Ling Qin

S1. Comment

The title compound is obtained by the condensation between dimethylsuccinate and cyclohexanecarbaldehyde. We report here the crystal structure of the title compound (Fig. 1).

In the title compound, $C_{11}H_{16}O_4$, the cyclohexane ring adopts chair conformation. Intermolecular O—H···O hydrogen bonds are observed. They help to establish the crystal packing.

S2. Experimental

The title compound was prepared according to the method of Stobbe condensation (Stobbe *et al.*,1893). To a room temperature stirred solution of 50% NaH (1.06 g, 0.022 mol) in anhydrous toluene (20 ml), a solution of dimethyl-succinate (3.2 g, 0.022 mol) and anhydrous methanol (1 ml) in anhydrous toluene (10 ml) was added dropwise. The mixture was stirred for 1 h, and then a solution of cyclohexanecarbaldehyde (2.24 g, 0.02 mol) in anhydrous toluene (20 ml) was added dropwise. The mixture was stirred at room temperature for 3 h and water (30 ml) was added. The water phase was washed with ethyl acetatethree times and then sodium hydroxide (3 g, 0.075 mol) was added. The mixture was refluxed for 4 and then acidified with hydrochloric acid to PH 2 and extracted with ethyl acetate. The organic layer was dried with anhydrous Mg_2SO_4, and then concentrated *in vacuo*. The title compound (3.65 g) was obtained in powder form in a yield of 86.1%. Crystals of (I) were obtained by slow evaporation of a solution of ethanol (m.p. 477–478 K).

S3. Refinement

All H atoms were positioned geometrically (C—H=0.95–0.99 Å), and refined as riding with U_{iso}(H)=1.2U_{eq}(carrier) or 1.5$_{eq}$(methyl groups).

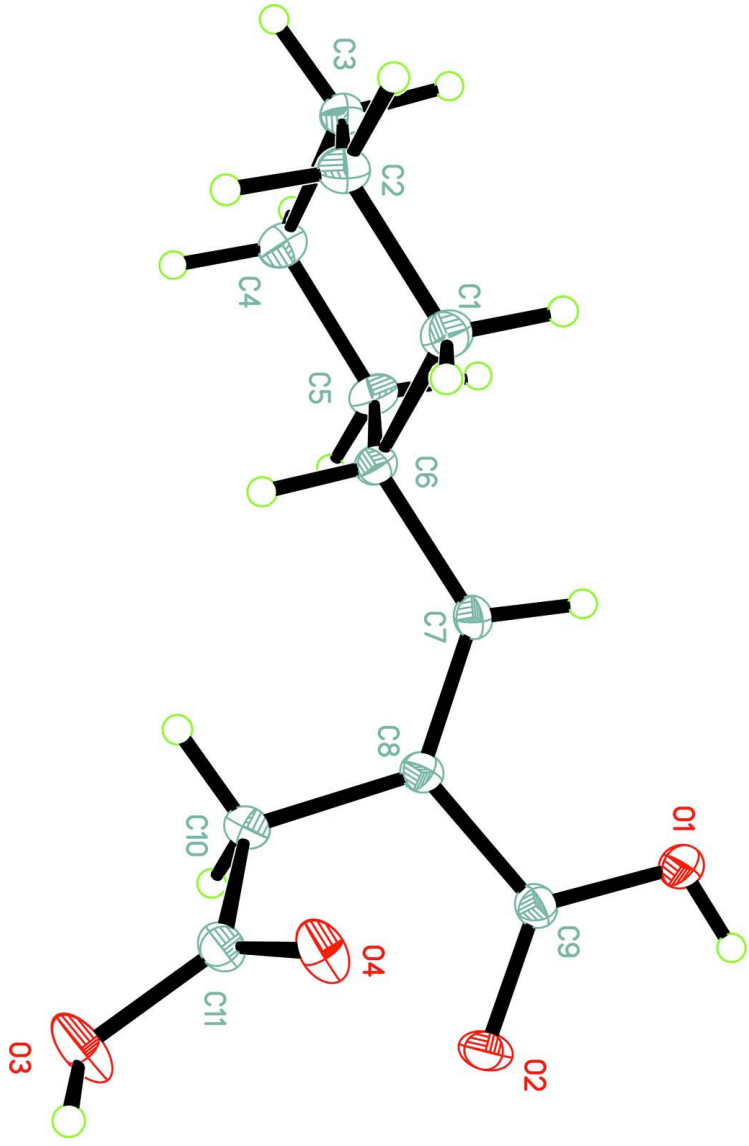

Figure 1

A view of the molecular of (I). Displacement ellopsoids are drawn at the 30% probability level and H atoms are shown as small spheres of arbitrary radii.

(*E*)-2-(Cyclohexylmethylene)succinic acid

Crystal data

$C_{11}H_{16}O_4$
$M_r = 212.24$
Monoclinic, $P2_1/c$
$a = 10.530\ (2)$ Å
$b = 18.762\ (4)$ Å
$c = 16.982\ (3)$ Å
$\beta = 91.42\ (3)°$
$V = 3354.2\ (12)$ Å^3
$Z = 12$
$F(000) = 1368$
$D_x = 1.261$ Mg m^{-3}
Melting point = 477–478 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 266 reflections
$\theta = 1.6–27.9°$
$\mu = 0.10$ mm^{-1}
$T = 113$ K
Block, colorless
0.10 × 0.08 × 0.06 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.981, T_{max} = 0.994
20420 measured reflections
5924 independent reflections
4780 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 25.0°, θ_{min} = 1.6°
h = −12→12
k = −22→17
l = −20→20

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.136
S = 1.11
5924 reflections
424 parameters
6 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
$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.7047P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 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	0.52956 (15)	0.70841 (9)	0.41522 (9)	0.0304 (4)
H1	0.484 (2)	0.6721 (13)	0.4393 (15)	0.046*
O2	0.66462 (15)	0.69150 (9)	0.51719 (9)	0.0332 (4)
O3	0.99241 (18)	0.73367 (10)	0.54599 (10)	0.0411 (5)
H3	1.041 (3)	0.6941 (14)	0.5465 (18)	0.062*
O4	0.92339 (15)	0.68425 (9)	0.43354 (9)	0.0316 (4)
O5	0.52767 (15)	0.59208 (9)	0.57875 (9)	0.0316 (4)
H5	0.572 (2)	0.6279 (13)	0.5551 (15)	0.047*
O6	0.39596 (15)	0.60645 (9)	0.47430 (9)	0.0302 (4)
O7	0.13511 (15)	0.61815 (9)	0.55937 (9)	0.0329 (4)
O8	0.06732 (15)	0.56926 (10)	0.44624 (9)	0.0298 (4)
H8	0.022 (3)	0.6127 (15)	0.4416 (15)	0.045*
O9	0.01165 (15)	0.54289 (9)	0.91554 (9)	0.0291 (4)
O10	0.13307 (16)	0.55344 (10)	1.02487 (9)	0.0319 (4)
H10	0.078 (2)	0.5177 (13)	1.0442 (15)	0.048*

O11	0.38062 (15)	0.52336 (9)	0.94221 (9)	0.0314 (4)
O12	0.47862 (16)	0.59219 (10)	1.03214 (10)	0.0334 (4)
H12	0.529 (3)	0.5553 (13)	1.0388 (17)	0.050*
C1	0.7758 (2)	0.86243 (13)	0.23578 (13)	0.0286 (6)
H1A	0.8336	0.8210	0.2322	0.034*
H1B	0.6963	0.8508	0.2063	0.034*
C2	0.8377 (2)	0.92728 (13)	0.19892 (13)	0.0303 (6)
H2A	0.9206	0.9365	0.2258	0.036*
H2B	0.8536	0.9174	0.1428	0.036*
C3	0.7543 (2)	0.99311 (13)	0.20515 (13)	0.0283 (6)
H3A	0.6752	0.9860	0.1734	0.034*
H3B	0.7994	1.0348	0.1836	0.034*
C4	0.7209 (2)	1.00790 (13)	0.29086 (13)	0.0300 (6)
H4A	0.7989	1.0211	0.3212	0.036*
H4B	0.6611	1.0485	0.2928	0.036*
C5	0.6608 (2)	0.94265 (12)	0.32821 (13)	0.0290 (6)
H5A	0.5780	0.9328	0.3015	0.035*
H5B	0.6448	0.9527	0.3843	0.035*
C6	0.7462 (2)	0.87649 (12)	0.32232 (12)	0.0232 (5)
H6	0.8277	0.8859	0.3520	0.028*
C7	0.6815 (2)	0.81489 (12)	0.35909 (12)	0.0224 (5)
H7	0.6067	0.7984	0.3325	0.027*
C8	0.7153 (2)	0.77971 (12)	0.42489 (12)	0.0217 (5)
C9	0.6326 (2)	0.72300 (12)	0.45483 (12)	0.0233 (5)
C10	0.8316 (2)	0.79437 (12)	0.47521 (13)	0.0256 (5)
H10A	0.8785	0.8348	0.4523	0.031*
H10B	0.8050	0.8088	0.5284	0.031*
C11	0.9193 (2)	0.73159 (13)	0.48279 (13)	0.0258 (5)
C12	0.1708 (2)	0.47583 (12)	0.72032 (13)	0.0254 (5)
H12A	0.2003	0.5118	0.7593	0.030*
H12B	0.1197	0.5006	0.6790	0.030*
C13	0.0883 (2)	0.42064 (14)	0.76085 (14)	0.0316 (6)
H13A	0.0163	0.4450	0.7859	0.038*
H13B	0.0531	0.3871	0.7210	0.038*
C14	0.1639 (2)	0.37941 (14)	0.82281 (13)	0.0319 (6)
H14A	0.1094	0.3418	0.8451	0.038*
H14B	0.1899	0.4120	0.8661	0.038*
C15	0.2816 (2)	0.34522 (13)	0.78859 (14)	0.0342 (6)
H15A	0.2554	0.3075	0.7508	0.041*
H15B	0.3327	0.3227	0.8315	0.041*
C16	0.3624 (2)	0.40022 (13)	0.74674 (14)	0.0300 (6)
H16A	0.4351	0.3759	0.7223	0.036*
H16B	0.3969	0.4348	0.7858	0.036*
C17	0.2857 (2)	0.44026 (12)	0.68315 (12)	0.0221 (5)
H17	0.2536	0.4047	0.6436	0.026*
C18	0.3650 (2)	0.49360 (12)	0.64108 (12)	0.0210 (5)
H18	0.4416	0.5081	0.6670	0.025*
C19	0.3392 (2)	0.52292 (12)	0.57084 (12)	0.0220 (5)

C20	0.4251 (2)	0.57730 (12)	0.53837 (12)	0.0218 (5)
C21	0.2249 (2)	0.50708 (12)	0.51964 (13)	0.0241 (5)
H21A	0.1769	0.4672	0.5429	0.029*
H21B	0.2529	0.4918	0.4670	0.029*
C22	0.1394 (2)	0.57048 (13)	0.51064 (12)	0.0245 (5)
C23	0.1341 (2)	0.74770 (13)	0.74597 (15)	0.0337 (6)
H23A	0.0935	0.7147	0.7074	0.040*
H23B	0.0662	0.7703	0.7766	0.040*
C24	0.2075 (3)	0.80494 (14)	0.70232 (16)	0.0406 (7)
H24A	0.1494	0.8293	0.6643	0.049*
H24B	0.2396	0.8409	0.7405	0.049*
C25	0.3181 (2)	0.77368 (15)	0.65866 (14)	0.0392 (7)
H25A	0.2850	0.7438	0.6147	0.047*
H25B	0.3683	0.8129	0.6359	0.047*
C26	0.4034 (2)	0.72914 (14)	0.71173 (14)	0.0330 (6)
H26A	0.4466	0.7604	0.7510	0.040*
H26B	0.4695	0.7062	0.6799	0.040*
C27	0.3288 (2)	0.67178 (13)	0.75447 (14)	0.0279 (6)
H27A	0.3868	0.6449	0.7904	0.033*
H27B	0.2917	0.6379	0.7156	0.033*
C28	0.2228 (2)	0.70562 (12)	0.80172 (12)	0.0232 (5)
H28	0.2624	0.7398	0.8403	0.028*
C29	0.1525 (2)	0.65043 (12)	0.84662 (12)	0.0227 (5)
H29	0.0754	0.6334	0.8233	0.027*
C30	0.1871 (2)	0.62253 (12)	0.91624 (12)	0.0222 (5)
C31	0.1035 (2)	0.56922 (12)	0.95195 (12)	0.0214 (5)
C32	0.3049 (2)	0.64220 (13)	0.96253 (13)	0.0258 (5)
H32A	0.2806	0.6630	1.0136	0.031*
H32B	0.3520	0.6790	0.9333	0.031*
C33	0.3907 (2)	0.57935 (13)	0.97767 (12)	0.0247 (5)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0250 (9)	0.0345 (11)	0.0316 (9)	−0.0096 (8)	−0.0043 (8)	0.0093 (8)
O2	0.0306 (10)	0.0391 (11)	0.0297 (8)	−0.0028 (8)	−0.0050 (8)	0.0159 (8)
O3	0.0440 (12)	0.0446 (12)	0.0337 (9)	0.0166 (9)	−0.0211 (9)	−0.0119 (9)
O4	0.0300 (10)	0.0369 (11)	0.0275 (8)	0.0057 (8)	−0.0083 (8)	−0.0069 (8)
O5	0.0248 (10)	0.0374 (11)	0.0323 (9)	−0.0098 (8)	−0.0041 (8)	0.0096 (8)
O6	0.0281 (9)	0.0362 (10)	0.0262 (8)	−0.0030 (8)	−0.0017 (7)	0.0101 (7)
O7	0.0321 (10)	0.0372 (11)	0.0288 (8)	0.0094 (8)	−0.0097 (8)	−0.0087 (8)
O8	0.0272 (9)	0.0369 (11)	0.0246 (8)	0.0041 (8)	−0.0087 (7)	−0.0017 (7)
O9	0.0263 (9)	0.0322 (10)	0.0286 (8)	−0.0066 (8)	−0.0060 (8)	0.0064 (7)
O10	0.0312 (10)	0.0412 (11)	0.0231 (8)	−0.0089 (8)	−0.0023 (7)	0.0107 (7)
O11	0.0306 (10)	0.0321 (10)	0.0312 (8)	0.0027 (8)	−0.0079 (8)	−0.0075 (8)
O12	0.0295 (10)	0.0379 (11)	0.0321 (9)	0.0044 (8)	−0.0141 (8)	−0.0088 (8)
C1	0.0292 (13)	0.0280 (14)	0.0286 (12)	−0.0017 (11)	0.0026 (11)	0.0027 (10)
C2	0.0317 (14)	0.0348 (15)	0.0245 (11)	−0.0047 (11)	0.0027 (11)	0.0037 (10)

C3	0.0277 (13)	0.0287 (14)	0.0284 (12)	−0.0051 (10)	−0.0045 (11)	0.0075 (10)
C4	0.0319 (14)	0.0266 (14)	0.0316 (12)	−0.0028 (11)	0.0041 (11)	0.0023 (10)
C5	0.0299 (13)	0.0279 (14)	0.0296 (12)	−0.0028 (11)	0.0057 (11)	0.0050 (10)
C6	0.0201 (12)	0.0248 (13)	0.0246 (11)	−0.0032 (10)	−0.0011 (10)	0.0063 (10)
C7	0.0181 (12)	0.0259 (13)	0.0231 (11)	−0.0026 (10)	−0.0025 (10)	0.0020 (10)
C8	0.0212 (12)	0.0224 (12)	0.0215 (10)	0.0004 (9)	−0.0011 (10)	0.0026 (9)
C9	0.0213 (13)	0.0263 (13)	0.0222 (11)	−0.0002 (10)	−0.0013 (10)	0.0009 (10)
C10	0.0283 (13)	0.0255 (14)	0.0228 (11)	−0.0017 (10)	−0.0040 (10)	0.0032 (10)
C11	0.0229 (13)	0.0318 (14)	0.0225 (11)	−0.0025 (10)	−0.0019 (10)	0.0027 (10)
C12	0.0252 (13)	0.0252 (13)	0.0258 (11)	0.0033 (10)	0.0013 (10)	0.0053 (10)
C13	0.0259 (13)	0.0401 (16)	0.0292 (12)	−0.0020 (11)	0.0049 (11)	0.0071 (11)
C14	0.0338 (14)	0.0357 (15)	0.0262 (12)	−0.0073 (11)	0.0027 (11)	0.0068 (11)
C15	0.0406 (15)	0.0293 (14)	0.0325 (13)	0.0038 (12)	−0.0004 (12)	0.0100 (11)
C16	0.0286 (14)	0.0307 (14)	0.0307 (12)	0.0042 (11)	0.0028 (11)	0.0063 (11)
C17	0.0244 (12)	0.0190 (12)	0.0228 (10)	−0.0013 (9)	0.0009 (10)	−0.0001 (9)
C18	0.0171 (11)	0.0229 (13)	0.0231 (11)	0.0008 (9)	−0.0010 (9)	−0.0024 (9)
C19	0.0204 (12)	0.0216 (12)	0.0242 (11)	0.0035 (9)	0.0021 (10)	0.0005 (9)
C20	0.0176 (12)	0.0255 (13)	0.0223 (11)	0.0025 (9)	−0.0010 (10)	−0.0001 (9)
C21	0.0237 (12)	0.0253 (13)	0.0232 (11)	−0.0025 (10)	0.0012 (10)	0.0009 (9)
C22	0.0203 (12)	0.0314 (14)	0.0218 (11)	−0.0025 (10)	0.0017 (10)	0.0029 (10)
C23	0.0331 (14)	0.0282 (14)	0.0398 (14)	0.0045 (11)	0.0011 (12)	0.0126 (11)
C24	0.0395 (16)	0.0344 (16)	0.0474 (15)	−0.0022 (13)	−0.0086 (13)	0.0212 (13)
C25	0.0419 (16)	0.0454 (17)	0.0299 (13)	−0.0208 (13)	−0.0069 (12)	0.0131 (12)
C26	0.0290 (14)	0.0380 (16)	0.0321 (13)	−0.0052 (11)	0.0036 (12)	0.0040 (11)
C27	0.0245 (13)	0.0267 (14)	0.0325 (12)	0.0002 (10)	0.0005 (11)	0.0031 (10)
C28	0.0277 (13)	0.0222 (13)	0.0197 (10)	−0.0035 (10)	−0.0023 (10)	0.0008 (9)
C29	0.0236 (12)	0.0221 (13)	0.0223 (11)	−0.0002 (10)	−0.0001 (10)	−0.0022 (9)
C30	0.0236 (12)	0.0213 (12)	0.0217 (10)	0.0019 (10)	0.0013 (10)	0.0003 (9)
C31	0.0199 (12)	0.0252 (13)	0.0190 (10)	0.0026 (10)	−0.0008 (10)	0.0019 (9)
C32	0.0286 (13)	0.0275 (14)	0.0213 (11)	−0.0037 (10)	−0.0022 (10)	0.0013 (10)
C33	0.0201 (12)	0.0345 (15)	0.0196 (10)	−0.0031 (10)	0.0017 (10)	0.0015 (10)

Geometric parameters (Å, º)

O1—C9	1.292 (3)	C13—C14	1.516 (3)
O1—H1	0.93 (2)	C13—H13A	0.9900
O2—C9	1.252 (3)	C13—H13B	0.9900
O3—C11	1.306 (3)	C14—C15	1.524 (3)
O3—H3	0.90 (2)	C14—H14A	0.9900
O4—C11	1.222 (3)	C14—H14B	0.9900
O5—C20	1.295 (3)	C15—C16	1.525 (3)
O5—H5	0.92 (2)	C15—H15A	0.9900
O6—C20	1.249 (3)	C15—H15B	0.9900
O7—C22	1.220 (3)	C16—C17	1.530 (3)
O8—C22	1.316 (3)	C16—H16A	0.9900
O8—H8	0.95 (3)	C16—H16B	0.9900
O9—C31	1.238 (3)	C17—C18	1.496 (3)
O10—C31	1.303 (3)	C17—H17	1.0000

O10—H10	0.95 (2)	C18—C19	1.335 (3)
O11—C33	1.214 (3)	C18—H18	0.9500
O12—C33	1.315 (3)	C19—C20	1.479 (3)
O12—H12	0.87 (2)	C19—C21	1.497 (3)
C1—C2	1.522 (3)	C21—C22	1.498 (3)
C1—C6	1.533 (3)	C21—H21A	0.9900
C1—H1A	0.9900	C21—H21B	0.9900
C1—H1B	0.9900	C23—C24	1.526 (3)
C2—C3	1.520 (3)	C23—C28	1.533 (3)
C2—H2A	0.9900	C23—H23A	0.9900
C2—H2B	0.9900	C23—H23B	0.9900
C3—C4	1.531 (3)	C24—C25	1.514 (4)
C3—H3A	0.9900	C24—H24A	0.9900
C3—H3B	0.9900	C24—H24B	0.9900
C4—C5	1.524 (3)	C25—C26	1.509 (4)
C4—H4A	0.9900	C25—H25A	0.9900
C4—H4B	0.9900	C25—H25B	0.9900
C5—C6	1.538 (3)	C26—C27	1.526 (3)
C5—H5A	0.9900	C26—H26A	0.9900
C5—H5B	0.9900	C26—H26B	0.9900
C6—C7	1.487 (3)	C27—C28	1.529 (3)
C6—H6	1.0000	C27—H27A	0.9900
C7—C8	1.338 (3)	C27—H27B	0.9900
C7—H7	0.9500	C28—C29	1.493 (3)
C8—C9	1.474 (3)	C28—H28	1.0000
C8—C10	1.502 (3)	C29—C30	1.335 (3)
C10—C11	1.500 (3)	C29—H29	0.9500
C10—H10A	0.9900	C30—C31	1.472 (3)
C10—H10B	0.9900	C30—C32	1.499 (3)
C12—C13	1.526 (3)	C32—C33	1.504 (3)
C12—C17	1.532 (3)	C32—H32A	0.9900
C12—H12A	0.9900	C32—H32B	0.9900
C12—H12B	0.9900		

C9—O1—H1	111.1 (17)	C15—C16—C17	111.7 (2)
C11—O3—H3	108 (2)	C15—C16—H16A	109.3
C20—O5—H5	110.8 (18)	C17—C16—H16A	109.3
C22—O8—H8	109.4 (17)	C15—C16—H16B	109.3
C31—O10—H10	110.7 (17)	C17—C16—H16B	109.3
C33—O12—H12	111 (2)	H16A—C16—H16B	107.9
C2—C1—C6	110.85 (19)	C18—C17—C16	112.02 (19)
C2—C1—H1A	109.5	C18—C17—C12	111.17 (18)
C6—C1—H1A	109.5	C16—C17—C12	109.38 (17)
C2—C1—H1B	109.5	C18—C17—H17	108.0
C6—C1—H1B	109.5	C16—C17—H17	108.0
H1A—C1—H1B	108.1	C12—C17—H17	108.0
C3—C2—C1	111.55 (19)	C19—C18—C17	126.8 (2)
C3—C2—H2A	109.3	C19—C18—H18	116.6

C1—C2—H2A	109.3	C17—C18—H18	116.6
C3—C2—H2B	109.3	C18—C19—C20	120.3 (2)
C1—C2—H2B	109.3	C18—C19—C21	125.4 (2)
H2A—C2—H2B	108.0	C20—C19—C21	114.26 (19)
C2—C3—C4	111.1 (2)	O6—C20—O5	123.4 (2)
C2—C3—H3A	109.4	O6—C20—C19	119.2 (2)
C4—C3—H3A	109.4	O5—C20—C19	117.40 (19)
C2—C3—H3B	109.4	C19—C21—C22	111.99 (19)
C4—C3—H3B	109.4	C19—C21—H21A	109.2
H3A—C3—H3B	108.0	C22—C21—H21A	109.2
C5—C4—C3	111.0 (2)	C19—C21—H21B	109.2
C5—C4—H4A	109.4	C22—C21—H21B	109.2
C3—C4—H4A	109.4	H21A—C21—H21B	107.9
C5—C4—H4B	109.4	O7—C22—O8	123.1 (2)
C3—C4—H4B	109.4	O7—C22—C21	123.0 (2)
H4A—C4—H4B	108.0	O8—C22—C21	113.8 (2)
C4—C5—C6	111.80 (18)	C24—C23—C28	110.8 (2)
C4—C5—H5A	109.3	C24—C23—H23A	109.5
C6—C5—H5A	109.3	C28—C23—H23A	109.5
C4—C5—H5B	109.3	C24—C23—H23B	109.5
C6—C5—H5B	109.3	C28—C23—H23B	109.5
H5A—C5—H5B	107.9	H23A—C23—H23B	108.1
C7—C6—C1	112.03 (19)	C25—C24—C23	111.7 (2)
C7—C6—C5	108.97 (17)	C25—C24—H24A	109.3
C1—C6—C5	109.54 (19)	C23—C24—H24A	109.3
C7—C6—H6	108.7	C25—C24—H24B	109.3
C1—C6—H6	108.7	C23—C24—H24B	109.3
C5—C6—H6	108.7	H24A—C24—H24B	107.9
C8—C7—C6	128.2 (2)	C26—C25—C24	112.1 (2)
C8—C7—H7	115.9	C26—C25—H25A	109.2
C6—C7—H7	115.9	C24—C25—H25A	109.2
C7—C8—C9	119.8 (2)	C26—C25—H25B	109.2
C7—C8—C10	125.5 (2)	C24—C25—H25B	109.2
C9—C8—C10	114.64 (19)	H25A—C25—H25B	107.9
O2—C9—O1	123.1 (2)	C25—C26—C27	111.6 (2)
O2—C9—C8	118.9 (2)	C25—C26—H26A	109.3
O1—C9—C8	118.0 (2)	C27—C26—H26A	109.3
C11—C10—C8	113.3 (2)	C25—C26—H26B	109.3
C11—C10—H10A	108.9	C27—C26—H26B	109.3
C8—C10—H10A	108.9	H26A—C26—H26B	108.0
C11—C10—H10B	108.9	C26—C27—C28	110.34 (19)
C8—C10—H10B	108.9	C26—C27—H27A	109.6
H10A—C10—H10B	107.7	C28—C27—H27A	109.6
O4—C11—O3	123.6 (2)	C26—C27—H27B	109.6
O4—C11—C10	123.0 (2)	C28—C27—H27B	109.6
O3—C11—C10	113.3 (2)	H27A—C27—H27B	108.1
C13—C12—C17	110.75 (19)	C29—C28—C27	110.96 (18)
C13—C12—H12A	109.5	C29—C28—C23	111.73 (19)

C17—C12—H12A	109.5	C27—C28—C23	109.36 (18)
C13—C12—H12B	109.5	C29—C28—H28	108.2
C17—C12—H12B	109.5	C27—C28—H28	108.2
H12A—C12—H12B	108.1	C23—C28—H28	108.2
C14—C13—C12	111.4 (2)	C30—C29—C28	126.6 (2)
C14—C13—H13A	109.3	C30—C29—H29	116.7
C12—C13—H13A	109.3	C28—C29—H29	116.7
C14—C13—H13B	109.3	C29—C30—C31	118.6 (2)
C12—C13—H13B	109.3	C29—C30—C32	124.9 (2)
H13A—C13—H13B	108.0	C31—C30—C32	116.53 (19)
C13—C14—C15	111.59 (18)	O9—C31—O10	123.3 (2)
C13—C14—H14A	109.3	O9—C31—C30	122.18 (19)
C15—C14—H14A	109.3	O10—C31—C30	114.5 (2)
C13—C14—H14B	109.3	C30—C32—C33	112.5 (2)
C15—C14—H14B	109.3	C30—C32—H32A	109.1
H14A—C14—H14B	108.0	C33—C32—H32A	109.1
C14—C15—C16	111.2 (2)	C30—C32—H32B	109.1
C14—C15—H15A	109.4	C33—C32—H32B	109.1
C16—C15—H15A	109.4	H32A—C32—H32B	107.8
C14—C15—H15B	109.4	O11—C33—O12	124.0 (2)
C16—C15—H15B	109.4	O11—C33—C32	123.3 (2)
H15A—C15—H15B	108.0	O12—C33—C32	112.7 (2)

C6—C1—C2—C3	−57.4 (3)	C17—C18—C19—C20	177.59 (19)
C1—C2—C3—C4	55.7 (3)	C17—C18—C19—C21	−0.7 (4)
C2—C3—C4—C5	−54.5 (3)	C18—C19—C20—O6	−176.6 (2)
C3—C4—C5—C6	55.6 (3)	C21—C19—C20—O6	1.9 (3)
C2—C1—C6—C7	177.96 (19)	C18—C19—C20—O5	3.4 (3)
C2—C1—C6—C5	56.9 (3)	C21—C19—C20—O5	−178.14 (18)
C4—C5—C6—C7	−179.48 (19)	C18—C19—C21—C22	114.2 (2)
C4—C5—C6—C1	−56.6 (3)	C20—C19—C21—C22	−64.2 (2)
C1—C6—C7—C8	125.9 (2)	C19—C21—C22—O7	−25.6 (3)
C5—C6—C7—C8	−112.7 (3)	C19—C21—C22—O8	155.81 (18)
C6—C7—C8—C9	175.5 (2)	C28—C23—C24—C25	55.4 (3)
C6—C7—C8—C10	−2.1 (4)	C23—C24—C25—C26	−53.0 (3)
C7—C8—C9—O2	−178.8 (2)	C24—C25—C26—C27	53.8 (3)
C10—C8—C9—O2	−0.9 (3)	C25—C26—C27—C28	−56.9 (3)
C7—C8—C9—O1	0.9 (3)	C26—C27—C28—C29	−177.58 (19)
C10—C8—C9—O1	178.79 (19)	C26—C27—C28—C23	58.7 (3)
C7—C8—C10—C11	−120.5 (2)	C24—C23—C28—C29	178.6 (2)
C9—C8—C10—C11	61.8 (2)	C24—C23—C28—C27	−58.2 (3)
C8—C10—C11—O4	25.3 (3)	C27—C28—C29—C30	82.5 (3)
C8—C10—C11—O3	−156.38 (19)	C23—C28—C29—C30	−155.2 (2)
C17—C12—C13—C14	57.2 (3)	C28—C29—C30—C31	179.0 (2)
C12—C13—C14—C15	−54.9 (3)	C28—C29—C30—C32	0.1 (4)
C13—C14—C15—C16	53.7 (3)	C29—C30—C31—O9	9.4 (3)
C14—C15—C16—C17	−55.4 (3)	C32—C30—C31—O9	−171.6 (2)
C15—C16—C17—C18	−179.20 (19)	C29—C30—C31—O10	−169.4 (2)

C15—C16—C17—C12	57.1 (3)	C32—C30—C31—O10	9.7 (3)
C13—C12—C17—C18	178.21 (19)	C29—C30—C32—C33	−120.9 (2)
C13—C12—C17—C16	−57.6 (3)	C31—C30—C32—C33	60.1 (2)
C16—C17—C18—C19	160.7 (2)	C30—C32—C33—O11	15.5 (3)
C12—C17—C18—C19	−76.6 (3)	C30—C32—C33—O12	−165.74 (17)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O11^{i}	0.87 (2)	1.78 (2)	2.656 (2)	175 (3)
O10—H10···O9^{ii}	0.95 (2)	1.63 (2)	2.586 (2)	175 (3)
O8—H8···O4^{iii}	0.95 (3)	1.70 (3)	2.642 (2)	172 (3)
O5—H5···O2	0.92 (2)	1.68 (2)	2.594 (2)	175 (3)
O3—H3···O7^{iv}	0.90 (2)	1.75 (2)	2.644 (2)	173 (3)
O1—H1···O6	0.93 (2)	1.66 (2)	2.591 (2)	175 (3)

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+2; (ii) −*x*, −*y*+1, −*z*+2; (iii) *x*−1, *y*, *z*; (iv) *x*+1, *y*, *z*.