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20-*O*-β-D-Xylopyranosyl(1→6)-β-D-glucopyranosyl-20(*S*)-protopanaxadiol methanol solvate

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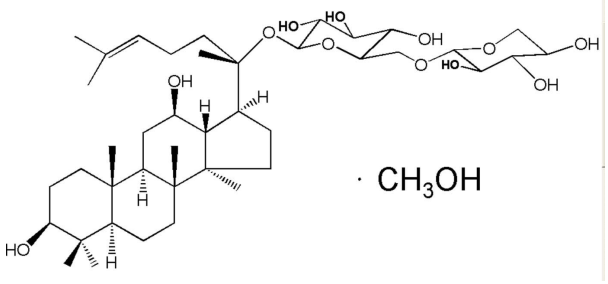
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.045; wR factor = 0.099; data-to-parameter ratio = 10.4.

The title compound, $C_{41}H_{70}O_{12}\cdot CH_4O$, was prepared by microbial transformation. Within the steroid skeleton of the molecule, three six-membered rings exhibit a chair conformation, while the five -membered ring adopts an envelope conformation. The two pyranosyl rings also adopt chair conformations. The molecules are held together by an extensive O—H···O hydrogen-bonding network.

Related literature

For general background, see: He *et al.* (2005); Hu *et al.* (2007). For related literature, see: Li *et al.* (2006).



Experimental

Crystal data

$C_{41}H_{70}O_{12}\cdot CH_4O$
M_r = 787.01
Orthorhombic, $P2_12_12_1$
a = 8.3044 (7) Å
b = 13.2927 (11) Å
c = 38.964 (3) Å
V = 4301.1 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.09 mm^{-1}
T = 293 (2) K
0.43 × 0.31 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: none
25549 measured reflections
5269 independent reflections
3467 reflections with $I > 2\sigma(I)$
R_{int} = 0.084

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.099
S = 0.89
5269 reflections
505 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O11^{i}	0.82	2.38	2.996 (3)	133
O2—H2···O3	0.83	1.96	2.752 (3)	160
O5—H5A···O1^{i}	0.84	2.14	2.839 (3)	140
O6—H6···O12^{ii}	0.83	2.04	2.781 (3)	147
O7—H7···O8^{iii}	0.83	2.31	3.009 (3)	142
O10—H10···O13^{iv}	0.82	2.05	2.824 (4)	156
O11—H11···O5^{v}	0.81	1.92	2.703 (3)	162
O12—H12A···O11^{vi}	0.84	2.02	2.827 (3)	162
O13—H13A···O2^{vii}	0.85	1.98	2.823 (3)	173

Symmetry codes: (i) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $x, y-1, z$; (iii) $x-\frac{1}{2}, -y+\frac{3}{2}, -z$; (iv) $x-1, y, z$; (v) $x, y+1, z$; (vi) $x-\frac{1}{2}, -y+\frac{5}{2}, -z$; (vii) $x+1, y, z$.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; 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*.

We gratefully acknowledge financial support from the SK Shanghai Foundation. We sincerely thank Dr J. Sun of Shanghai Institute of Organic Chemistry for assistance with the data collection.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU2351).

References

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supporting information

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20-*O*-*β*-D-Xylopyranosyl(1→6)-*β*-D-glucopyranosyl-20(*S*)-protopanaxadiol methanol solvate

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S1. Comment

20-O-*β*-*D*-xylopyranosyl(1→6)-*β*-*D*-glucopyranosyl-20(S)- protopanaxadiol is a kind of rare gensenoside, found existing in *notoginseng*. In recent studies, the compound has been related to an anticancer agent. It is believed to have activities including: cytotoxicity to and partial reversal of multidrug resistance of human tumor cells (He *et al.*, 2005). Besides that, the compound may be an important precurosor metabolite of Compound K, which is also a potential anticancer agent, during the process of microbial transformation of gisenoside Rb_3 (Hu *et al.*, 2007). In this article, the crystal structure is reported.

The structure mainly consists of a protopanaxadiol moiety with a disaccharide group. The bond distances and angels are normal. The C24?C25 of 1.313 (5) Å shows a typical double bound. Within the steroid skeleton of the molecule, three six membered rings all display the chair conformation, while a five membered ring displays an envelope conformation. Two pyranosyl rings are also exist in chair conformation. Extensive O—H···O hydrogen bonding occurs in the crystal structure (Table 1), which helps to stabilize the crystal structure.

S2. Experimental

The Fermentation broth of ginsenoside Rb_3 (300 mg) was centrifuged and the precipitation was extracted with EtOH for 24 h. Removal of the EtOH from the extract under reduced pressure gave crude extract. And the extract was subjected to silica gel column chromatography, eluting with $HCCl_3$:CH_3OH (10:1→7:3→5:1) to afford 12 fractions. Recrystallizing of fractions 8~10 yielded ginsenoside Rb_3 100 mg. Solvent loss technique was then employed for the growth of crystals at room temperature, using methanol as the solvent.

S3. Refinement

Hydroxyl H atoms were located in a Fourier map and refined as riding in as-found relative positions with U_{iso}(H) = 1.2U_{eq}(O). Other H atoms were placed in geometrically calculated positions with C—H = 0.93–0.98 Å and constrained to ride on their parental atoms with U_{iso}(H) = 1.2U_{eq}(C). Torsonal angles for methyl groups were refined to fit the electron density. In absence of significant anomalous scattering, Friedel pairs were meged.

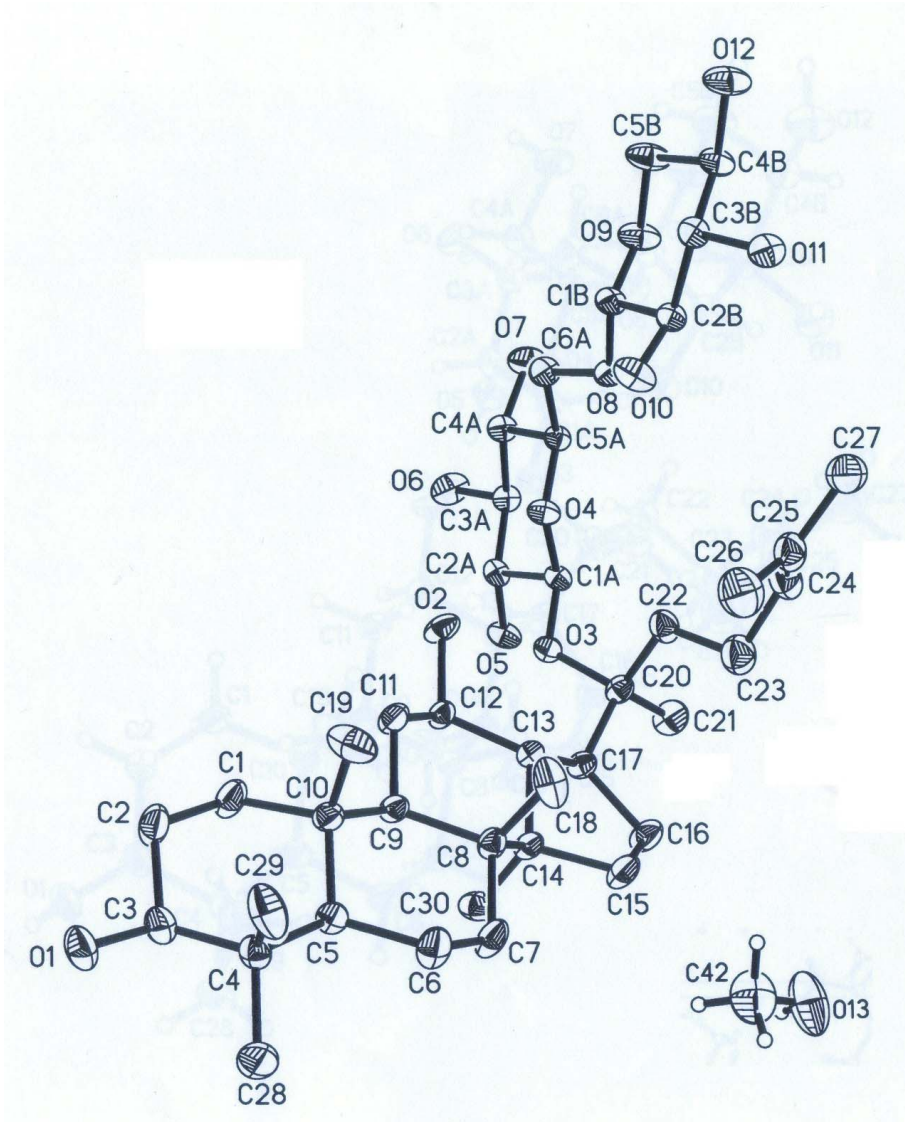

Figure 1

The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

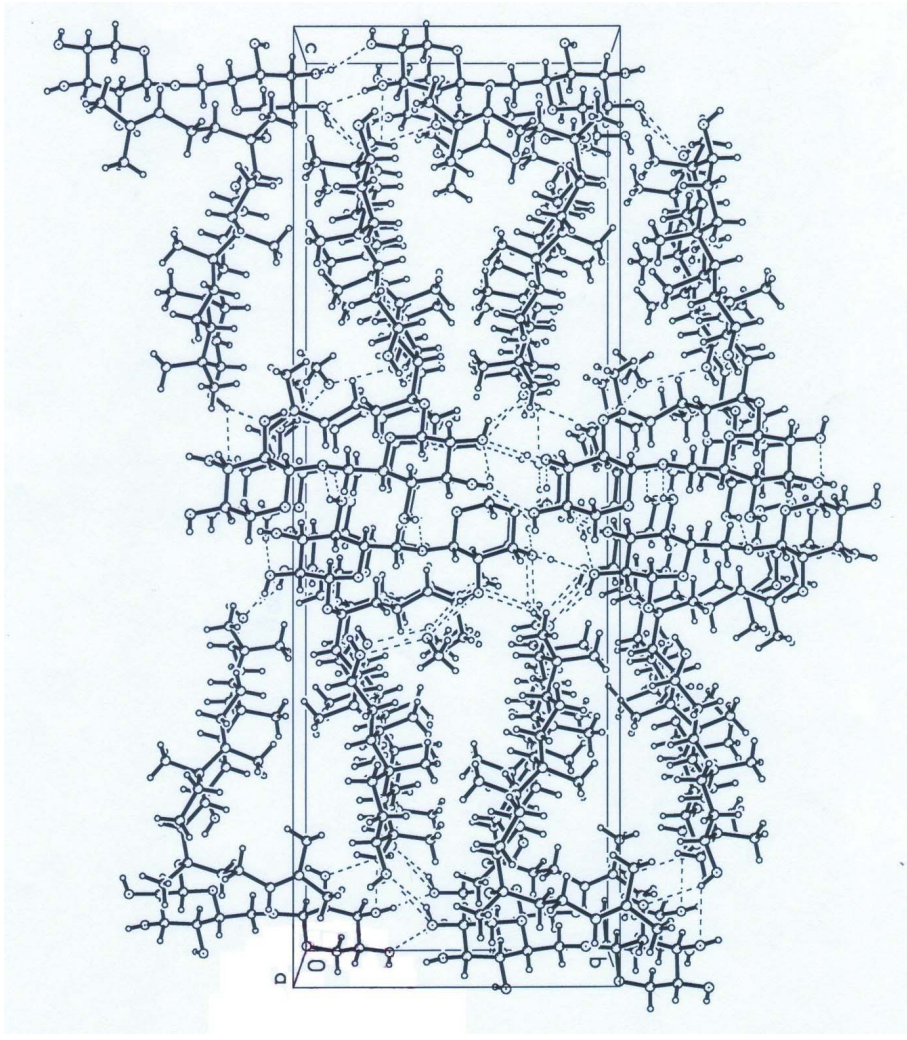

Figure 2

The packing of (I), viewed down the *c* axis. H atoms not involved in hydrogen bonding have been omitted.

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Crystal data

$C_{41}H_{70}O_{12}\cdot CH_4O$
$M_r = 787.01$
Orthorhombic, $P2_12_12_1$
Hall symbol: P 2ac 2ab
$a = 8.3044\ (7)$ Å
$b = 13.2927\ (11)$ Å
$c = 38.964\ (3)$ Å
$V = 4301.1\ (6)$ Å^3
$Z = 4$
$F(000) = 1720$
$D_x = 1.215$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3975 reflections
$\theta = 2.2–20.3°$
$\mu = 0.09$ mm^{-1}
$T = 293$ K
Prismatic, colorless
0.43 × 0.31 × 0.21 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
25549 measured reflections
5269 independent reflections
3467 reflections with $I > 2\sigma(I)$
$R_{int} = 0.084$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.6°$
$h = -8 \rightarrow 10$
$k = -16 \rightarrow 16$
$l = -49 \rightarrow 36$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.099$
$S = 0.89$
5269 reflections
505 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0447P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.21$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	−0.1448 (3)	0.78606 (18)	0.37658 (5)	0.0555 (6)
H1	−0.1513	0.7395	0.3903	0.067*
O2	0.0242 (2)	0.70266 (16)	0.15197 (5)	0.0478 (6)
H2	0.0585	0.6714	0.1351	0.057*
O3	0.2082 (2)	0.59757 (13)	0.10544 (5)	0.0332 (5)
O4	0.0549 (2)	0.69683 (12)	0.07077 (5)	0.0355 (5)
O5	0.1065 (3)	0.42488 (12)	0.06866 (5)	0.0410 (5)
H5A	0.1328	0.4117	0.0891	0.049*
O6	−0.1631 (3)	0.45653 (15)	0.02402 (6)	0.0564 (7)
H6	−0.1048	0.4060	0.0217	0.068*
O7	−0.1595 (3)	0.65240 (15)	−0.00867 (6)	0.0554 (6)
H7	−0.2445	0.6200	−0.0101	0.066*
O8	0.0245 (2)	0.89395 (12)	0.04299 (5)	0.0383 (5)
O9	−0.0675 (3)	0.99980 (13)	0.00267 (5)	0.0494 (6)
O10	0.0865 (3)	1.06672 (15)	0.08776 (5)	0.0536 (6)
H10	0.0749	1.0105	0.0963	0.064*
O11	0.1701 (2)	1.23411 (14)	0.04880 (5)	0.0436 (5)

H11	0.1346	1.2905	0.0517	0.052*
O12	−0.0620 (3)	1.27492 (13)	−0.00414 (6)	0.0588 (7)
H12A	−0.1258	1.2720	−0.0208	0.071*
O13	1.0284 (5)	0.9088 (2)	0.13462 (9)	0.1152 (12)
H13A	1.0334	0.8464	0.1387	0.138*
C1	−0.0982 (4)	0.7430 (3)	0.28168 (8)	0.0577 (10)
H1A	−0.1708	0.7610	0.2632	0.069*
H1B	−0.0866	0.6704	0.2816	0.069*
C2	−0.1738 (4)	0.7753 (3)	0.31584 (8)	0.0598 (10)
H2A	−0.1978	0.8467	0.3150	0.072*
H2B	−0.2744	0.7395	0.3191	0.072*
C3	−0.0647 (4)	0.7546 (2)	0.34554 (8)	0.0437 (8)
H3	−0.0490	0.6816	0.3469	0.052*
C4	0.1004 (4)	0.8032 (2)	0.34246 (7)	0.0388 (7)
C5	0.1721 (3)	0.7717 (2)	0.30679 (7)	0.0353 (7)
H5	0.1791	0.6982	0.3081	0.042*
C6	0.3460 (4)	0.8051 (3)	0.30089 (8)	0.0550 (9)
H6A	0.3482	0.8764	0.2956	0.066*
H6B	0.4080	0.7945	0.3217	0.066*
C7	0.4216 (4)	0.7463 (3)	0.27159 (8)	0.0556 (10)
H7A	0.4276	0.6760	0.2781	0.067*
H7B	0.5309	0.7702	0.2682	0.067*
C8	0.3295 (3)	0.7549 (2)	0.23745 (7)	0.0361 (7)
C9	0.1476 (3)	0.7339 (2)	0.24388 (7)	0.0326 (7)
H9	0.1429	0.6627	0.2503	0.039*
C10	0.0674 (4)	0.7909 (2)	0.27473 (7)	0.0368 (7)
C11	0.0535 (4)	0.7403 (2)	0.21037 (7)	0.0433 (8)
H11A	−0.0575	0.7222	0.2150	0.052*
H11B	0.0542	0.8098	0.2027	0.052*
C12	0.1155 (3)	0.6744 (2)	0.18123 (7)	0.0343 (7)
H12	0.0924	0.6039	0.1867	0.041*
C13	0.2970 (3)	0.6870 (2)	0.17658 (7)	0.0299 (6)
H13	0.3148	0.7565	0.1691	0.036*
C14	0.3875 (3)	0.6743 (2)	0.21106 (7)	0.0348 (7)
C15	0.5622 (4)	0.6825 (3)	0.19860 (8)	0.0508 (9)
H15A	0.6353	0.6525	0.2151	0.061*
H15B	0.5921	0.7523	0.1952	0.061*
C16	0.5670 (4)	0.6248 (3)	0.16445 (8)	0.0510 (9)
H16A	0.6102	0.5577	0.1679	0.061*
H16B	0.6344	0.6598	0.1480	0.061*
C17	0.3895 (3)	0.6188 (2)	0.15113 (7)	0.0349 (7)
H17	0.3529	0.5499	0.1555	0.042*
C18	0.3600 (5)	0.8615 (2)	0.22343 (9)	0.0596 (10)
H18A	0.2832	0.8762	0.2057	0.089*
H18B	0.4669	0.8652	0.2141	0.089*
H18C	0.3490	0.9095	0.2417	0.089*
C19	0.0399 (5)	0.9034 (2)	0.26662 (9)	0.0659 (11)
H19A	0.0007	0.9103	0.2436	0.099*

H19B	0.1397	0.9392	0.2689	0.099*
H19C	−0.0378	0.9306	0.2823	0.099*
C20	0.3724 (3)	0.6361 (2)	0.11227 (7)	0.0355 (7)
C21	0.4888 (4)	0.5676 (2)	0.09287 (8)	0.0553 (9)
H21A	0.4776	0.4998	0.1011	0.083*
H21B	0.5973	0.5901	0.0966	0.083*
H21C	0.4647	0.5698	0.0688	0.083*
C22	0.3795 (4)	0.7460 (2)	0.10016 (8)	0.0459 (8)
H22A	0.3657	0.7460	0.0754	0.055*
H22B	0.2871	0.7805	0.1098	0.055*
C23	0.5271 (5)	0.8082 (3)	0.10821 (10)	0.0629 (10)
H23A	0.5320	0.8208	0.1327	0.075*
H23B	0.6229	0.7711	0.1017	0.075*
C24	0.5236 (4)	0.9067 (3)	0.08935 (10)	0.0587 (10)
H24	0.5410	0.9030	0.0658	0.070*
C25	0.4993 (4)	0.9973 (3)	0.10175 (9)	0.0549 (9)
C26	0.4625 (6)	1.0201 (3)	0.13858 (11)	0.0854 (13)
H26A	0.3543	1.0450	0.1405	0.128*
H26B	0.5362	1.0702	0.1469	0.128*
H26C	0.4734	0.9600	0.1520	0.128*
C27	0.5084 (5)	1.0876 (3)	0.07860 (10)	0.0718 (12)
H27A	0.6062	1.1238	0.0831	0.108*
H27B	0.4178	1.1307	0.0828	0.108*
H27C	0.5071	1.0660	0.0551	0.108*
C28	0.2058 (4)	0.7593 (3)	0.37114 (8)	0.0678 (11)
H28A	0.3092	0.7916	0.3709	0.102*
H28B	0.2194	0.6884	0.3675	0.102*
H28C	0.1549	0.7705	0.3929	0.102*
C29	0.0899 (5)	0.9178 (2)	0.34767 (9)	0.0641 (11)
H29A	−0.0138	0.9413	0.3403	0.096*
H29B	0.1725	0.9502	0.3344	0.096*
H29C	0.1045	0.9333	0.3715	0.096*
C30	0.3678 (4)	0.5653 (2)	0.22474 (8)	0.0505 (9)
H30A	0.3915	0.5184	0.2067	0.076*
H30B	0.2591	0.5555	0.2324	0.076*
H30C	0.4406	0.5547	0.2435	0.076*
C42	0.8920 (6)	0.9444 (3)	0.15107 (13)	0.1016 (17)
H42A	0.8403	0.8901	0.1630	0.152*
H42B	0.8192	0.9721	0.1344	0.152*
H42C	0.9220	0.9957	0.1672	0.152*
C1A	0.1450 (3)	0.60535 (18)	0.07231 (7)	0.0301 (7)
H1A1	0.2311	0.6051	0.0551	0.036*
C2A	0.0281 (4)	0.52007 (18)	0.06611 (7)	0.0316 (7)
H2A1	−0.0585	0.5235	0.0832	0.038*
C3A	−0.0432 (4)	0.53004 (18)	0.03063 (7)	0.0331 (7)
H3A	0.0430	0.5229	0.0136	0.040*
C4A	−0.1214 (4)	0.6325 (2)	0.02629 (7)	0.0366 (7)
H4A	−0.2199	0.6355	0.0401	0.044*

C5A	−0.0067 (3)	0.71584 (18)	0.03752 (7)	0.0317 (7)
H5A1	0.0830	0.7198	0.0212	0.038*
C6A	−0.0913 (4)	0.8157 (2)	0.03872 (9)	0.0416 (8)
H6A1	−0.1508	0.8262	0.0176	0.050*
H6A2	−0.1670	0.8167	0.0577	0.050*
C1B	−0.0405 (4)	0.98966 (18)	0.03842 (8)	0.0361 (7)
H1B1	−0.1422	0.9959	0.0510	0.043*
C2B	0.0804 (4)	1.06560 (19)	0.05159 (7)	0.0337 (7)
H2B1	0.1873	1.0487	0.0426	0.040*
C3B	0.0367 (4)	1.17108 (18)	0.04037 (7)	0.0347 (7)
H3B	−0.0572	1.1934	0.0536	0.042*
C4B	−0.0042 (4)	1.17607 (19)	0.00276 (8)	0.0400 (8)
H4B	0.0935	1.1640	−0.0107	0.048*
C5B	−0.1275 (5)	1.0974 (2)	−0.00591 (9)	0.0551 (10)
H5B1	−0.2259	1.1104	0.0067	0.066*
H5B2	−0.1521	1.1002	−0.0302	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0617 (15)	0.0692 (16)	0.0356 (13)	−0.0109 (13)	0.0146 (11)	−0.0030 (11)
O2	0.0348 (12)	0.0773 (15)	0.0312 (11)	0.0163 (11)	−0.0092 (10)	−0.0133 (11)
O3	0.0385 (11)	0.0318 (10)	0.0293 (11)	0.0001 (9)	−0.0021 (9)	−0.0018 (9)
O4	0.0495 (12)	0.0212 (9)	0.0356 (11)	0.0047 (9)	−0.0054 (10)	−0.0023 (9)
O5	0.0643 (15)	0.0236 (10)	0.0350 (11)	0.0072 (10)	−0.0016 (11)	−0.0019 (9)
O6	0.0563 (15)	0.0287 (11)	0.0840 (18)	−0.0062 (11)	−0.0208 (14)	−0.0112 (12)
O7	0.0634 (16)	0.0416 (12)	0.0613 (16)	−0.0077 (12)	−0.0271 (13)	0.0009 (11)
O8	0.0368 (11)	0.0198 (9)	0.0585 (14)	−0.0011 (9)	−0.0072 (10)	0.0026 (9)
O9	0.0730 (16)	0.0220 (10)	0.0533 (14)	−0.0001 (11)	−0.0240 (13)	−0.0018 (10)
O10	0.0816 (17)	0.0382 (12)	0.0410 (13)	−0.0050 (12)	−0.0082 (13)	0.0031 (10)
O11	0.0510 (13)	0.0217 (10)	0.0580 (14)	−0.0023 (10)	−0.0099 (11)	−0.0066 (10)
O12	0.0804 (17)	0.0240 (11)	0.0721 (16)	0.0042 (11)	−0.0285 (14)	0.0032 (11)
O13	0.132 (3)	0.078 (2)	0.136 (3)	0.024 (2)	0.057 (2)	0.036 (2)
C1	0.0291 (17)	0.105 (3)	0.0393 (19)	−0.0002 (19)	−0.0025 (16)	−0.012 (2)
C2	0.0330 (18)	0.106 (3)	0.041 (2)	−0.003 (2)	0.0072 (16)	−0.007 (2)
C3	0.0475 (19)	0.0480 (18)	0.0355 (18)	−0.0041 (16)	0.0082 (16)	−0.0025 (15)
C4	0.0415 (18)	0.0449 (18)	0.0299 (16)	−0.0049 (15)	0.0018 (14)	−0.0048 (14)
C5	0.0366 (17)	0.0378 (16)	0.0315 (16)	−0.0015 (14)	−0.0014 (13)	−0.0074 (14)
C6	0.041 (2)	0.087 (3)	0.0374 (19)	−0.0151 (19)	−0.0019 (16)	−0.0193 (19)
C7	0.0280 (17)	0.099 (3)	0.0398 (19)	−0.0020 (19)	−0.0025 (15)	−0.019 (2)
C8	0.0297 (16)	0.0453 (18)	0.0332 (16)	−0.0040 (14)	−0.0014 (13)	−0.0067 (14)
C9	0.0279 (15)	0.0382 (16)	0.0316 (16)	0.0010 (13)	−0.0045 (13)	−0.0024 (13)
C10	0.0335 (17)	0.0454 (18)	0.0315 (16)	0.0072 (14)	−0.0002 (13)	−0.0004 (14)
C11	0.0276 (16)	0.066 (2)	0.0360 (18)	0.0071 (16)	−0.0014 (14)	−0.0041 (16)
C12	0.0250 (15)	0.0467 (18)	0.0311 (16)	−0.0025 (13)	−0.0038 (13)	−0.0037 (14)
C13	0.0284 (15)	0.0306 (15)	0.0306 (16)	0.0018 (13)	−0.0016 (13)	−0.0036 (13)
C14	0.0273 (16)	0.0434 (17)	0.0339 (17)	0.0031 (14)	−0.0044 (13)	−0.0042 (14)
C15	0.0286 (18)	0.080 (2)	0.043 (2)	0.0047 (18)	−0.0049 (15)	−0.0126 (19)

C16	0.0325 (18)	0.070 (2)	0.051 (2)	0.0158 (18)	0.0010 (16)	−0.0043 (18)
C17	0.0328 (16)	0.0348 (16)	0.0372 (17)	0.0065 (13)	−0.0019 (14)	−0.0043 (14)
C18	0.075 (3)	0.049 (2)	0.054 (2)	−0.023 (2)	0.022 (2)	−0.0168 (18)
C19	0.101 (3)	0.055 (2)	0.041 (2)	0.033 (2)	0.015 (2)	0.0047 (17)
C20	0.0318 (16)	0.0403 (16)	0.0345 (17)	0.0049 (14)	−0.0002 (14)	−0.0065 (14)
C21	0.054 (2)	0.071 (2)	0.042 (2)	0.0104 (19)	0.0031 (18)	−0.0154 (18)
C22	0.048 (2)	0.054 (2)	0.0360 (18)	−0.0120 (17)	−0.0008 (15)	0.0009 (16)
C23	0.060 (2)	0.066 (2)	0.063 (2)	−0.015 (2)	0.001 (2)	0.002 (2)
C24	0.064 (2)	0.056 (2)	0.056 (2)	−0.018 (2)	0.001 (2)	0.0047 (19)
C25	0.048 (2)	0.061 (2)	0.056 (2)	−0.0144 (18)	−0.0009 (18)	0.001 (2)
C26	0.091 (3)	0.078 (3)	0.087 (3)	−0.011 (3)	0.004 (3)	−0.005 (3)
C27	0.066 (3)	0.061 (2)	0.089 (3)	−0.011 (2)	−0.016 (2)	0.012 (2)
C28	0.055 (2)	0.114 (3)	0.0339 (19)	0.005 (2)	−0.0049 (17)	−0.003 (2)
C29	0.089 (3)	0.054 (2)	0.049 (2)	−0.020 (2)	0.025 (2)	−0.0180 (18)
C30	0.058 (2)	0.053 (2)	0.0408 (19)	0.0216 (18)	−0.0010 (17)	0.0010 (16)
C42	0.098 (4)	0.079 (3)	0.128 (4)	0.024 (3)	0.011 (4)	0.027 (3)
C1A	0.0401 (17)	0.0214 (14)	0.0289 (16)	0.0023 (13)	0.0021 (14)	−0.0015 (12)
C2A	0.0420 (17)	0.0181 (13)	0.0348 (16)	0.0017 (13)	0.0071 (14)	−0.0037 (12)
C3A	0.0397 (17)	0.0236 (14)	0.0359 (17)	−0.0028 (13)	−0.0034 (14)	−0.0079 (13)
C4A	0.0425 (18)	0.0263 (14)	0.0410 (18)	−0.0012 (14)	−0.0051 (15)	−0.0031 (13)
C5A	0.0357 (16)	0.0266 (14)	0.0329 (16)	−0.0017 (13)	−0.0021 (13)	−0.0001 (12)
C6A	0.0417 (18)	0.0252 (15)	0.058 (2)	−0.0049 (14)	−0.0047 (16)	0.0017 (15)
C1B	0.0426 (18)	0.0196 (14)	0.0460 (19)	−0.0010 (13)	−0.0028 (15)	0.0020 (13)
C2B	0.0388 (17)	0.0283 (15)	0.0340 (17)	0.0006 (13)	−0.0004 (14)	−0.0013 (13)
C3B	0.0406 (18)	0.0214 (14)	0.0420 (18)	−0.0009 (13)	−0.0003 (15)	−0.0062 (13)
C4B	0.055 (2)	0.0183 (14)	0.0469 (19)	0.0024 (14)	−0.0043 (16)	0.0011 (13)
C5B	0.079 (3)	0.0264 (16)	0.059 (2)	0.0001 (17)	−0.028 (2)	0.0042 (16)

Geometric parameters (Å, º)

O1—C3	1.442 (3)	C16—C17	1.565 (4)
O1—H1	0.8206	C16—H16A	0.9700
O2—C12	1.419 (3)	C16—H16B	0.9700
O2—H2	0.8267	C17—C20	1.538 (4)
O3—C1A	1.398 (3)	C17—H17	0.9800
O3—C20	1.481 (3)	C18—H18A	0.9600
O4—C5A	1.416 (3)	C18—H18B	0.9600
O4—C1A	1.429 (3)	C18—H18C	0.9600
O5—C2A	1.426 (3)	C19—H19A	0.9600
O5—H5A	0.8444	C19—H19B	0.9600
O6—C3A	1.418 (3)	C19—H19C	0.9600
O6—H6	0.8326	C20—C21	1.528 (4)
O7—C4A	1.423 (3)	C20—C22	1.536 (4)
O7—H7	0.8293	C21—H21A	0.9600
O8—C1B	1.393 (3)	C21—H21B	0.9600
O8—C6A	1.426 (3)	C21—H21C	0.9600
O9—C1B	1.418 (3)	C22—C23	1.512 (4)
O9—C5B	1.429 (3)	C22—H22A	0.9700

O10—C2B	1.410 (3)	C22—H22B	0.9700
O10—H10	0.8225	C23—C24	1.501 (5)
O11—C3B	1.427 (3)	C23—H23A	0.9700
O11—H11	0.8128	C23—H23B	0.9700
O12—C4B	1.424 (3)	C24—C25	1.313 (5)
O12—H12A	0.8392	C24—H24	0.9300
O13—C42	1.385 (5)	C25—C26	1.498 (5)
O13—H13A	0.8463	C25—C27	1.503 (5)
C1—C2	1.533 (4)	C26—H26A	0.9600
C1—C10	1.539 (4)	C26—H26B	0.9600
C1—H1A	0.9700	C26—H26C	0.9600
C1—H1B	0.9700	C27—H27A	0.9600
C2—C3	1.495 (4)	C27—H27B	0.9600
C2—H2A	0.9700	C27—H27C	0.9600
C2—H2B	0.9700	C28—H28A	0.9600
C3—C4	1.520 (4)	C28—H28B	0.9600
C3—H3	0.9800	C28—H28C	0.9600
C4—C28	1.535 (4)	C29—H29A	0.9600
C4—C29	1.539 (4)	C29—H29B	0.9600
C4—C5	1.569 (4)	C29—H29C	0.9600
C5—C6	1.529 (4)	C30—H30A	0.9600
C5—C10	1.543 (4)	C30—H30B	0.9600
C5—H5	0.9800	C30—H30C	0.9600
C6—C7	1.519 (4)	C42—H42A	0.9600
C6—H6A	0.9700	C42—H42B	0.9600
C6—H6B	0.9700	C42—H42C	0.9600
C7—C8	1.539 (4)	C1A—C2A	1.511 (4)
C7—H7A	0.9700	C1A—H1A1	0.9800
C7—H7B	0.9700	C2A—C3A	1.510 (4)
C8—C18	1.539 (4)	C2A—H2A1	0.9800
C8—C9	1.557 (4)	C3A—C4A	1.518 (4)
C8—C14	1.562 (4)	C3A—H3A	0.9800
C9—C11	1.524 (4)	C4A—C5A	1.526 (4)
C9—C10	1.569 (4)	C4A—H4A	0.9800
C9—H9	0.9800	C5A—C6A	1.503 (4)
C10—C19	1.545 (4)	C5A—H5A1	0.9800
C11—C12	1.524 (4)	C6A—H6A1	0.9700
C11—H11A	0.9700	C6A—H6A2	0.9700
C11—H11B	0.9700	C1B—C2B	1.513 (4)
C12—C13	1.528 (4)	C1B—H1B1	0.9800
C12—H12	0.9800	C2B—C3B	1.513 (4)
C13—C17	1.547 (4)	C2B—H2B1	0.9800
C13—C14	1.549 (4)	C3B—C4B	1.506 (4)
C13—H13	0.9800	C3B—H3B	0.9800
C14—C15	1.534 (4)	C4B—C5B	1.502 (4)
C14—C30	1.552 (4)	C4B—H4B	0.9800
C15—C16	1.536 (4)	C5B—H5B1	0.9700
C15—H15A	0.9700	C5B—H5B2	0.9700

C15—H15B	0.9700

C3—O1—H1	111.1	O3—C20—C17	102.1 (2)
C12—O2—H2	108.6	C21—C20—C17	109.8 (2)
C1A—O3—C20	119.1 (2)	C22—C20—C17	116.2 (2)
C5A—O4—C1A	112.29 (19)	C20—C21—H21A	109.5
C2A—O5—H5A	111.6	C20—C21—H21B	109.5
C3A—O6—H6	99.6	H21A—C21—H21B	109.5
C4A—O7—H7	99.1	C20—C21—H21C	109.5
C1B—O8—C6A	113.0 (2)	H21A—C21—H21C	109.5
C1B—O9—C5B	111.8 (2)	H21B—C21—H21C	109.5
C2B—O10—H10	112.8	C23—C22—C20	119.2 (3)
C3B—O11—H11	106.9	C23—C22—H22A	107.5
C4B—O12—H12A	108.5	C20—C22—H22A	107.5
C42—O13—H13A	106.7	C23—C22—H22B	107.5
C2—C1—C10	113.8 (3)	C20—C22—H22B	107.5
C2—C1—H1A	108.8	H22A—C22—H22B	107.0
C10—C1—H1A	108.8	C24—C23—C22	111.1 (3)
C2—C1—H1B	108.8	C24—C23—H23A	109.4
C10—C1—H1B	108.8	C22—C23—H23A	109.4
H1A—C1—H1B	107.7	C24—C23—H23B	109.4
C3—C2—C1	111.8 (3)	C22—C23—H23B	109.4
C3—C2—H2A	109.2	H23A—C23—H23B	108.0
C1—C2—H2A	109.2	C25—C24—C23	128.5 (4)
C3—C2—H2B	109.2	C25—C24—H24	115.7
C1—C2—H2B	109.2	C23—C24—H24	115.7
H2A—C2—H2B	107.9	C24—C25—C26	124.7 (4)
O1—C3—C2	108.4 (3)	C24—C25—C27	120.3 (3)
O1—C3—C4	111.0 (2)	C26—C25—C27	115.0 (3)
C2—C3—C4	114.0 (3)	C25—C26—H26A	109.5
O1—C3—H3	107.7	C25—C26—H26B	109.5
C2—C3—H3	107.7	H26A—C26—H26B	109.5
C4—C3—H3	107.7	C25—C26—H26C	109.5
C3—C4—C28	107.2 (3)	H26A—C26—H26C	109.5
C3—C4—C29	111.0 (3)	H26B—C26—H26C	109.5
C28—C4—C29	108.2 (3)	C25—C27—H27A	109.5
C3—C4—C5	107.4 (2)	C25—C27—H27B	109.5
C28—C4—C5	109.1 (2)	H27A—C27—H27B	109.5
C29—C4—C5	113.7 (3)	C25—C27—H27C	109.5
C6—C5—C10	111.3 (3)	H27A—C27—H27C	109.5
C6—C5—C4	114.5 (2)	H27B—C27—H27C	109.5
C10—C5—C4	117.3 (2)	C4—C28—H28A	109.5
C6—C5—H5	104.0	C4—C28—H28B	109.5
C10—C5—H5	104.0	H28A—C28—H28B	109.5
C4—C5—H5	104.0	C4—C28—H28C	109.5
C7—C6—C5	110.7 (3)	H28A—C28—H28C	109.5
C7—C6—H6A	109.5	H28B—C28—H28C	109.5
C5—C6—H6A	109.5	C4—C29—H29A	109.5

C7—C6—H6B	109.5	C4—C29—H29B	109.5
C5—C6—H6B	109.5	H29A—C29—H29B	109.5
H6A—C6—H6B	108.1	C4—C29—H29C	109.5
C6—C7—C8	114.0 (3)	H29A—C29—H29C	109.5
C6—C7—H7A	108.8	H29B—C29—H29C	109.5
C8—C7—H7A	108.8	C14—C30—H30A	109.5
C6—C7—H7B	108.8	C14—C30—H30B	109.5
C8—C7—H7B	108.8	H30A—C30—H30B	109.5
H7A—C7—H7B	107.7	C14—C30—H30C	109.5
C7—C8—C18	107.1 (3)	H30A—C30—H30C	109.5
C7—C8—C9	109.3 (2)	H30B—C30—H30C	109.5
C18—C8—C9	112.5 (3)	O13—C42—H42A	109.5
C7—C8—C14	111.4 (3)	O13—C42—H42B	109.5
C18—C8—C14	110.3 (2)	H42A—C42—H42B	109.5
C9—C8—C14	106.4 (2)	O13—C42—H42C	109.5
C11—C9—C8	110.4 (2)	H42A—C42—H42C	109.5
C11—C9—C10	114.3 (2)	H42B—C42—H42C	109.5
C8—C9—C10	116.6 (2)	O3—C1A—O4	107.4 (2)
C11—C9—H9	104.7	O3—C1A—C2A	109.5 (2)
C8—C9—H9	104.7	O4—C1A—C2A	107.2 (2)
C10—C9—H9	104.7	O3—C1A—H1A1	110.9
C1—C10—C5	107.0 (2)	O4—C1A—H1A1	110.9
C1—C10—C19	107.7 (3)	C2A—C1A—H1A1	110.9
C5—C10—C19	114.1 (3)	O5—C2A—C3A	108.7 (2)
C1—C10—C9	108.3 (2)	O5—C2A—C1A	111.2 (2)
C5—C10—C9	107.5 (2)	C3A—C2A—C1A	109.4 (2)
C19—C10—C9	111.9 (2)	O5—C2A—H2A1	109.2
C12—C11—C9	115.7 (2)	C3A—C2A—H2A1	109.2
C12—C11—H11A	108.4	C1A—C2A—H2A1	109.2
C9—C11—H11A	108.4	O6—C3A—C2A	112.4 (2)
C12—C11—H11B	108.4	O6—C3A—C4A	107.3 (2)
C9—C11—H11B	108.4	C2A—C3A—C4A	110.4 (2)
H11A—C11—H11B	107.4	O6—C3A—H3A	108.9
O2—C12—C11	105.4 (2)	C2A—C3A—H3A	108.9
O2—C12—C13	113.7 (2)	C4A—C3A—H3A	108.9
C11—C12—C13	111.0 (2)	O7—C4A—C3A	111.6 (2)
O2—C12—H12	108.8	O7—C4A—C5A	106.1 (2)
C11—C12—H12	108.8	C3A—C4A—C5A	110.6 (2)
C13—C12—H12	108.8	O7—C4A—H4A	109.5
C12—C13—C17	120.1 (2)	C3A—C4A—H4A	109.5
C12—C13—C14	111.3 (2)	C5A—C4A—H4A	109.5
C17—C13—C14	104.6 (2)	O4—C5A—C6A	107.4 (2)
C12—C13—H13	106.7	O4—C5A—C4A	111.0 (2)
C17—C13—H13	106.7	C6A—C5A—C4A	111.0 (2)
C14—C13—H13	106.7	O4—C5A—H5A1	109.1
C15—C14—C13	100.2 (2)	C6A—C5A—H5A1	109.1
C15—C14—C30	105.9 (3)	C4A—C5A—H5A1	109.1
C13—C14—C30	110.4 (2)	O8—C6A—C5A	109.5 (2)

C15—C14—C8	116.8 (2)	O8—C6A—H6A1	109.8
C13—C14—C8	110.3 (2)	C5A—C6A—H6A1	109.8
C30—C14—C8	112.5 (2)	O8—C6A—H6A2	109.8
C14—C15—C16	105.3 (2)	C5A—C6A—H6A2	109.8
C14—C15—H15A	110.7	H6A1—C6A—H6A2	108.2
C16—C15—H15A	110.7	O8—C1B—O9	105.9 (2)
C14—C15—H15B	110.7	O8—C1B—C2B	108.0 (2)
C16—C15—H15B	110.7	O9—C1B—C2B	112.0 (2)
H15A—C15—H15B	108.8	O8—C1B—H1B1	110.3
C15—C16—C17	106.7 (2)	O9—C1B—H1B1	110.3
C15—C16—H16A	110.4	C2B—C1B—H1B1	110.3
C17—C16—H16A	110.4	O10—C2B—C3B	106.7 (2)
C15—C16—H16B	110.4	O10—C2B—C1B	111.7 (2)
C17—C16—H16B	110.4	C3B—C2B—C1B	111.2 (2)
H16A—C16—H16B	108.6	O10—C2B—H2B1	109.1
C20—C17—C13	119.8 (2)	C3B—C2B—H2B1	109.1
C20—C17—C16	114.0 (2)	C1B—C2B—H2B1	109.1
C13—C17—C16	103.0 (2)	O11—C3B—C4B	111.9 (2)
C20—C17—H17	106.4	O11—C3B—C2B	106.9 (2)
C13—C17—H17	106.4	C4B—C3B—C2B	112.1 (2)
C16—C17—H17	106.4	O11—C3B—H3B	108.6
C8—C18—H18A	109.5	C4B—C3B—H3B	108.6
C8—C18—H18B	109.5	C2B—C3B—H3B	108.6
H18A—C18—H18B	109.5	O12—C4B—C5B	111.7 (3)
C8—C18—H18C	109.5	O12—C4B—C3B	107.5 (2)
H18A—C18—H18C	109.5	C5B—C4B—C3B	110.0 (3)
H18B—C18—H18C	109.5	O12—C4B—H4B	109.2
C10—C19—H19A	109.5	C5B—C4B—H4B	109.2
C10—C19—H19B	109.5	C3B—C4B—H4B	109.2
H19A—C19—H19B	109.5	O9—C5B—C4B	110.0 (3)
C10—C19—H19C	109.5	O9—C5B—H5B1	109.7
H19A—C19—H19C	109.5	C4B—C5B—H5B1	109.7
H19B—C19—H19C	109.5	O9—C5B—H5B2	109.7
O3—C20—C21	106.7 (2)	C4B—C5B—H5B2	109.7
O3—C20—C22	108.0 (2)	H5B1—C5B—H5B2	108.2
C21—C20—C22	113.0 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···O11^{i}	0.82	2.38	2.996 (3)	133
O2—H2···O3	0.83	1.96	2.752 (3)	160
O5—H5*A*···O1^{i}	0.84	2.14	2.839 (3)	140
O6—H6···O12^{ii}	0.83	2.04	2.781 (3)	147
O7—H7···O8^{iii}	0.83	2.31	3.009 (3)	142
O10—H10···O13^{iv}	0.82	2.05	2.824 (4)	156
O11—H11···O5^{v}	0.81	1.92	2.703 (3)	162

O12—H12*A*···O11^{vi}	0.84	2.02	2.827 (3)	162
O13—H13*A*···O2^{vii}	0.85	1.98	2.823 (3)	173

Symmetry codes: (i) −*x*, *y*−1/2, −*z*+1/2; (ii) *x*, *y*−1, *z*; (iii) *x*−1/2, −*y*+3/2, −*z*; (iv) *x*−1, *y*, *z*; (v) *x*, *y*+1, *z*; (vi) *x*−1/2, −*y*+5/2, −*z*; (vii) *x*+1, *y*, *z*.