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(25*R*)-3β,16β-Diacetoxy-23-acetyl-22,26-epoxy-cholesta-5,22-diene *n*-hexane 0.8-solvate

María-Guadalupe Hernández Linares,[a] Alan Carrasco-Carballo,[a] Gabriel Guerrero-Luna,[a] Sylvain Bernès[b]* and Gerardo Aguirre Hernández[c]

[a]Laboratorio de Investigación del Jardín Botánico, Instituto de Ciencias, Benemérita Universidad Autónoma de Puebla, Edif. 113 Complejo de Ciencias CU, San Manuel, 72570 Puebla, Pue., Mexico, [b]Instituto de Física, Benemérita Universidad Autónoma de Puebla, Av. San Claudio y 18 Sur, 72570 Puebla, Pue., Mexico, and [c]Centro de Graduados e Investigación del Instituto Tecnológico de Tijuana, Apdo. Postal 1166, 22500 Tijuana, B.C., Mexico. *Correspondence e-mail: sylvain_bernes@hotmail.com

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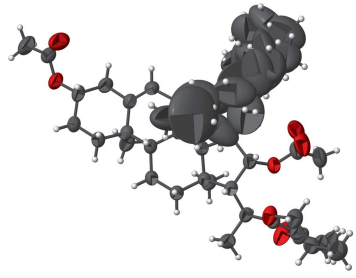
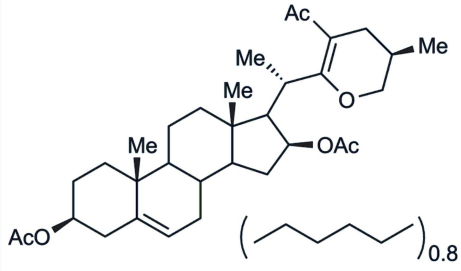
In the title solvate, $C_{33}H_{48}O_6\cdot0.8C_6H_{14}$, the steroid presents a conformation almost identical to that of its previously characterized benzene monosolvate [Sandoval-Ramírez *et al.* (1999). *Tetrahedron Lett.* **40**, 5143–5146]. The *n*-hexane solvent of crystallization is agglomerated in channels parallel to [100] in the crystal. The solvent molecule is disordered over two sites in the asymmetric unit, with occupancies of 0.46 and 0.34. A minor disorder for the carbonyl O atom of the acetyl substituent at position 16 in the steroid was also introduced, with two sites having occupancies of 0.7 and 0.3.

3D view



Chemical scheme



Structure description

The crystallization process for the title steroid afforded a hexane solvate (Fig. 1), which crystallizes in an orthorhombic cell close to that previously reported for the benzene monosolvate (Sandoval-Ramírez *et al.*, 1999; Refcode HOSKAB in the CSD). In both structures, the steroidal molecule adopts the same conformation. HOSKAB was reported and deposited with the wrong configuration; however, after inversion, a fit with the steroid reported here gives an r.m.s. deviation between the two molecules of 0.058 Å (Macrae *et al.*, 2008). The pyran ring C22–C26/O5 in the title structure also adopts the same twisted conformation found in related steroids bearing this ring (Sandoval-Ramírez *et al.*, 1999; Castro-Méndez *et al.*, 2002; Pérez-Díaz *et al.*, 2010).

Once the steroid is placed in the asymmetric unit, the refinement converges to R_1 = 0.073 (observed data), but *ca* 24% of the cell volume is unoccupied. Difference maps show that residual density is present in these voids, which may be modelled as two

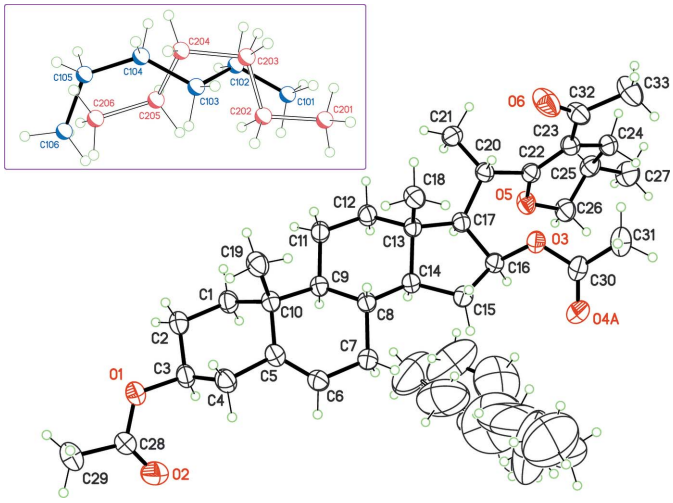


Figure 1
The structure of the title solvate, with displacement ellipsoids at the 30% probability level. Only one site for the disordered atom O4 has been retained. The inset represents the model used for the disordered solvent. The blue hexane molecule has a site occupancy factor of 0.46, and the red molecule has 0.34 occupancy.

disordered *n*-hexane molecules, C101–C106 and C201–C206 (Fig. 1, inset). After refining occupancies for each part, the hexane content may be roughly estimated as 0.8 hexane per asymmetric unit, and the refinement converges to $R_1 = 0.040$. Alternatively, starting from the unsolvated model, the *SQUEEZE* procedure (Spek, 2015) retrieves a density of 76 electrons in the unit cell, within two voids of 423 Å^3 (1.2 Å probe radius). Assuming that the solvent is hexane, these data correspond to a lower solvent content, *ca* 0.4 hexane per asymmetric unit. With the *SQUEEZE*-based data, the structure refinement converges to $R_1 = 0.042$.

The actual steroid:solvent stoichiometry for this crystal should thus be in the range 1:0.4 to 1:0.8, but the latter ratio was retained for the present report, which is based on diffraction data: after location of atoms C101–C206, the geometry in each part was restrained (see *Refinement details* section) and the structure refined with isotropic solvent. Occupancies for each part were then freely refined. In the last cycles, the solvent was refined with anisotropic displacement parameters, and site occupancies were fixed, in order to prevent correlations with thermal factors. In the final model, displacement for C atoms of hexane are rather large (Fig. 1), indicating that these disordered molecules interact poorly with the host molecules in the crystal. If, starting from this model, occupancies for solvent are decreased by 50%, residuals rise to $R_1 = 0.049$ for observed data and $wR_2 = 0.137$ for all data. On the other hand, the reported host:guest stoichiometry corresponds to the analysed single crystal, and solvent content may vary from crystal to crystal. The accurate stoichiometry and the reproducibility for a bulk preparation of this solvate remain unknown.

The model including coordinates for the solvent is severely disordered, as reflected in the high ADP's for atoms C101–C206. It is worth mentioning that attempts to include AcOEt as the solvent in the model were unsuccessful. It thus seems that this steroid is prone to interact with non-polar solvents, such as hexane and benzene. In the crystal, hexane is located in channels parallel to [100], and is arranged in such a way that a continuous electronic density is observed along the channels (Fig. 2). The *n*-hexane conformation includes for both molecules one *cis* torsion angle, C103—C104—C105—C106 = 8(7)°, and C202—C203—C204—C205 = −23 (8)°. This conformation is not common, compared to the full-*trans* conformer, but has been observed in many non-disordered solvate structures (*e.g.* Weberski *et al.*, 2012; Soki *et al.*, 2008). This bent conformation for *n*-hexane is reflected in the zigzag shape of the channels containing solvent molecules.

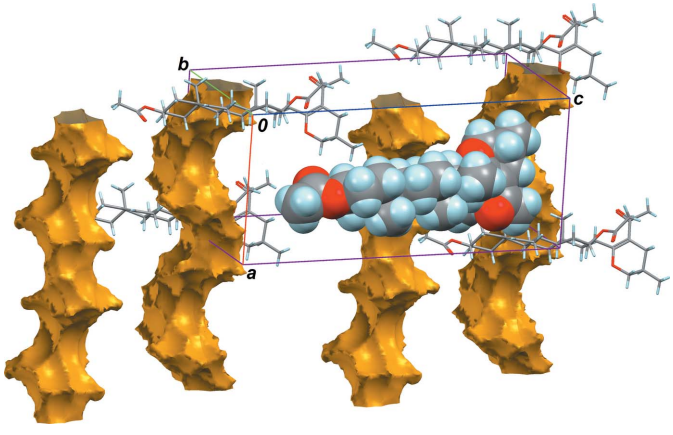


Figure 2
Part of the crystal structure for the title solvate. Five steroids are represented, one of which with a spacefill model. Channels parallel to [100] containing disordered *n*-hexane molecules are represented using the solvent accessible surface calculated using the non-solvated model (Macrae *et al.*, 2008). A probe radius of 1 Å was used, with a grid resolution of 0.25 Å.

Synthesis and crystallization

To a solution of diosgenin (2.0 g, 4.83 mmol) in acetic anhydride (10 ml) was added BF_3–OEt_2 (1.8 ml). The mixture was stirred for 10 min at room temperature. After 10 min, the mixture was poured into cold water (10 ml) and stirred for a further hour. The organic phase was neutralized with saturated $NaHCO_3$ solution (2 ×15 ml), DCM was added (15 ml) and the mixture washed with H_2O (2 ×10 ml). The organic phases were dried with Na_2SO_4 and evaporated under reduced pressure, affording a lacquer solid. This crude was purified by chromatography over silica gel (*n*-hexane:AcOEt, 85:15), giving the title epoxycholestene as a white solid (1.30 g, 75%). This compound was recrystallized from hexane solution, giving single crystals of the title solvate, m.p. 95–96 °C, $[\alpha_D]$ = −24 (c = 0.65, $CHCl_3$). Crystals are apparently air-stable for months, and were stored in a non-controlled atmosphere prior to crystallographic study. The IR spectrum of the crystallized compound shows better resolved absorption bands, compared to the crude lacquer, and vibrations in the range 681–832 cm^{-1} confirm the presence of hexane in the crystals.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{33}H_{48}O_6{\cdot}0.8C_6H_{14}$
M_r	609.65
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	294
a, b, c (Å)	11.64797 (17), 12.20869 (17), 25.7274 (4)
V (Å^3)	3658.60 (9)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	0.58
Crystal size (mm)	0.45 × 0.30 × 0.20
Data collection	
Diffractometer	Rigaku SuperNova
Absorption correction	Multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min}, T_{max}	0.663, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	19071, 6767, 5885
R_{int}	0.020
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.616
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.040, 0.106, 1.01
No. of reflections	6767
No. of parameters	477
No. of restraints	90
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.15, −0.11
Absolute structure	Flack x determined using 2066 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.09 (6)

Computer programs: *CrysAlis PRO* (Rigaku Oxford Diffraction, 2015), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015) and *Mercury* (Macrae *et al.*, 2008).

^{1}H-NMR (500 MHz, $CDCl_3$), δ: 5.35 (1*H*, *d*, J_{6-7e} = 4.5 Hz, H-6), 5.14 (1*H*, *ddd*, $J_{16-17} = J_{16-15e}$ = 7.5 and J_{16-15a} = 4.5 Hz, H-16), 4.59 (1*H*, *m*, H-3), 4.07 (1*H*, *m*, H-20), 3.48 (1*H*, *dd*, J_{26e-25} = 3.5 and $J_{26e-26a}$=10.5 Hz, H-26$_e$), 3.41 (1*H*, *dd*, $J_{26a-25} = J_{26a-26e}$ = 10.5 Hz, H-26$_a$), 2.20 (3*H*, *s*, C*H*$_3$-23^2), 2.02 (3*H*, *s*, C*H*$_3$CO$_2$-3), 1.84 (3*H*, *s*, C*H*$_3$CO$_2$-16), 1.18 (3*H*, *d*, J_{21-20} = 6.0 Hz, CH$_3$-21), 1.03 (3*H*, *s*, CH$_3$-19), 0.97 (3*H*, *d*, J_{27-25} = 6.0 Hz, CH$_3$-27), 0.92 (3*H*, *s*, CH$_3$-18). ^{13}C-NMR (125 MHz, $CDCl_3$), δ: 197.9 (C-23^1), 171.1 (C-22), 170.4 (CH$_3$*C*OO-3), 170.3 (CH$_3$*C*OO-16), 139.6 (C-5), 122.1 (C-6), 106.8 (C-23), 74.9 (C-16), 73.7 (C-3), 71.4 (C-26), 55.8 (C-17), 54.2 (C-14), 49.9 (C-9), 42.1 (C-13), 39.6 (C-12), 37.9 (C-4), 36.8 (C-1), 36.4 (C-10), 34.8 (C-15), 32.7 (C-20), 31.5 (C-24), 31.5 (C-7), 31.3 (C-8), 29.6 (C-23^2), 27.6 (C-2), 26.4 (C-25), 21.2 (*C*H$_3$COO-3), 21.0 (*C*H$_3$COO-16), 20.7 (C-11), 19.3 (C-21), 19.1 (C-19), 16.7 (C-27), 12.8 (C-18).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. For the disordered solvent, C—C bond lengths and 1,3-C···C distances were restrained to 1.53 (2) and 2.52 (3) Å, respectively. Moreover, C atoms were restrained with effective standard deviation of 0.1 Å^2 to have their U_{ij} components approximating an isotropic behaviour (*ISOR* command; Sheldrick, 2015). Site occupancies for molecules C101–C106 and C201–C206 were first refined, and then fixed in the final refinement cycles, to 0.46 and 0.34, respectively. A minor disorder in the steroidal molecule was also considered for the carbonyl O atom O4, disordered over two sites O4*A* and O4*B*, with occupancies 0.7 and 0.3, respectively. The absolute configuration expected for the steroid is in agreement with the refined Flack parameter, x = 0.09 (6).

Acknowledgements

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References

Castro-Méndez, A., Sandoval-Ramírez, J. & Bernès, S. (2002). *Acta Cryst.* E**58**, o606–o608.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Pérez-Díaz, J. O. H., Vega-Baez, J. L., Sandoval-Ramírez, J., Meza-Reyes, S., Montiel-Smith, S., Farfán, N. & Santillan, R. (2010). *Steroids*, **75**, 1127–1136.
Rigaku Oxford Diffraction (2015). *CrysAlis PRO*. Rigaku Americas Corporation, The Woodlands, TX, USA.
Sandoval-Ramírez, J., Castro-Méndez, A., Meza-Reyes, S., Reyes-Vázquez, F., Santillán, R. & Farfán, N. (1999). *Tetrahedron Lett.* **40**, 5143–5146.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Soki, F., Neudörfl, J.-M. & Goldfuss, B. (2008). *J. Organomet. Chem.* **693**, 2139–2146.
Spek, A. L. (2015). *Acta Cryst.* C**71**, 9–18.
Weberski, M. P. Jr, Chen, C., Delferro, M., Zuccaccia, C., Macchioni, A. & Marks, T. J. (2012). *Organometallics*, **31**, 3773–3789.

full crystallographic data

IUCrData (2016). **1**, x160622 [doi:10.1107/S2414314616006222]

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Crystal data

$C_{33}H_{48}O_6 \cdot 0.8C_6H_{14}$
M_r = 609.65
Orthorhombic, $P2_12_12_1$
a = 11.64797 (17) Å
b = 12.20869 (17) Å
c = 25.7274 (4) Å
V = 3658.60 (9) Å^3
Z = 4
$F(000)$ = 1336
D_x = 1.107 Mg m^{-3}
Melting point: 368 K
Cu $K\alpha$ radiation, λ = 1.54184 Å
Cell parameters from 10211 reflections
θ = 1.7–71.1°
μ = 0.58 mm^{-1}
T = 294 K
Irregular, colourless
0.45 × 0.30 × 0.20 mm

Data collection

Rigaku SuperNova diffractometer
Radiation source: micro-focus sealed X-ray tube, SuperNova (Cu) X-ray Source
Mirror monochromator
Detector resolution: 5.1980 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrysAlis PRO*; Rigaku Oxford Diffraction, 2015)
T_{min} = 0.663, T_{max} = 1.000
19071 measured reflections
6767 independent reflections
5885 reflections with $I > 2\sigma(I)$
R_{int} = 0.020
θ_{max} = 71.7°, θ_{min} = 3.4°
h = −14→14
k = −14→12
l = −31→28

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.106
S = 1.01
6767 reflections
477 parameters
90 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
$w = 1/[\sigma^2(F_o^2) + (0.0616P)^2 + 0.1048P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.11 e Å^{-3}
Extinction correction: *SHELXL2014* (Sheldrick, 2015), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.00090 (16)
Absolute structure: Flack x determined using 2066 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.09 (6)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.0960 (2)	0.3162 (2)	−0.14991 (9)	0.0593 (6)	
H1A	0.1179	0.3838	−0.1327	0.071*	
H1B	0.1618	0.2676	−0.1492	0.071*	
C2	0.0657 (2)	0.3415 (2)	−0.20665 (9)	0.0658 (6)	
H2A	0.0030	0.3938	−0.2079	0.079*	
H2B	0.1315	0.3739	−0.2239	0.079*	
C3	0.0314 (2)	0.2386 (2)	−0.23421 (8)	0.0603 (6)	
H3A	0.0972	0.1888	−0.2363	0.072*	
C4	−0.0677 (2)	0.1820 (2)	−0.20711 (9)	0.0634 (6)	
H4A	−0.0822	0.1121	−0.2238	0.076*	
H4B	−0.1363	0.2264	−0.2106	0.076*	
C5	−0.04301 (19)	0.1632 (2)	−0.14994 (9)	0.0541 (5)	
C6	−0.0558 (2)	0.0648 (2)	−0.12974 (9)	0.0628 (6)	
H6A	−0.0785	0.0086	−0.1519	0.075*	
C7	−0.0365 (3)	0.0368 (2)	−0.07381 (9)	0.0662 (7)	
H7A	0.0360	−0.0015	−0.0703	0.079*	
H7B	−0.0970	−0.0118	−0.0621	0.079*	
C8	−0.03482 (19)	0.13903 (18)	−0.03968 (8)	0.0503 (5)	
H8A	−0.1132	0.1677	−0.0372	0.060*	
C9	0.04179 (17)	0.22702 (18)	−0.06467 (8)	0.0469 (5)	
H9A	0.1166	0.1925	−0.0706	0.056*	
C10	−0.00335 (18)	0.26254 (18)	−0.11905 (8)	0.0488 (5)	
C11	0.0635 (2)	0.32403 (19)	−0.02824 (8)	0.0563 (6)	
H11A	−0.0056	0.3684	−0.0269	0.068*	
H11B	0.1239	0.3689	−0.0430	0.068*	
C12	0.0976 (2)	0.2932 (2)	0.02766 (9)	0.0551 (5)	
H12A	0.1735	0.2605	0.0274	0.066*	
H12B	0.1008	0.3589	0.0488	0.066*	
C13	0.01193 (17)	0.21232 (16)	0.05185 (8)	0.0448 (4)	
C14	0.0082 (2)	0.11360 (17)	0.01492 (8)	0.0500 (5)	
H14A	0.0879	0.0894	0.0109	0.060*	
C15	−0.0515 (3)	0.0249 (2)	0.04625 (9)	0.0656 (7)	
H15A	−0.0337	−0.0473	0.0327	0.079*	
H15B	−0.1341	0.0351	0.0461	0.079*	
C16	−0.0024 (2)	0.04006 (17)	0.10084 (8)	0.0537 (5)	
H16A	0.0571	−0.0151	0.1072	0.064*	
C17	0.05149 (18)	0.15607 (18)	0.10319 (8)	0.0480 (5)	
H17A	0.1347	0.1460	0.1003	0.058*	
C18	−0.1067 (2)	0.2655 (2)	0.05906 (9)	0.0577 (6)	
H18A	−0.1364	0.2872	0.0258	0.087*	
H18B	−0.0996	0.3287	0.0810	0.087*	
H18C	−0.1580	0.2138	0.0748	0.087*	
C19	−0.1025 (2)	0.3444 (2)	−0.11385 (9)	0.0671 (7)	
H19A	−0.1390	0.3537	−0.1470	0.101*	
H19B	−0.0732	0.4136	−0.1021	0.101*	

H19C	−0.1574	0.3172	−0.0892	0.101*	
C20	0.03072 (19)	0.21759 (17)	0.15443 (8)	0.0515 (5)	
H20A	−0.0518	0.2322	0.1573	0.062*	
C21	0.0939 (3)	0.32813 (19)	0.15666 (10)	0.0680 (7)	
H21A	0.0831	0.3607	0.1903	0.102*	
H21B	0.0638	0.3760	0.1304	0.102*	
H21C	0.1744	0.3167	0.1507	0.102*	
C22	0.06676 (18)	0.15316 (18)	0.20189 (8)	0.0508 (5)	
C23	0.0169 (2)	0.15302 (19)	0.24931 (8)	0.0533 (5)	
C24	0.0759 (2)	0.0987 (2)	0.29503 (9)	0.0597 (6)	
H24A	0.0695	0.1456	0.3253	0.072*	
H24B	0.0375	0.0301	0.3029	0.072*	
C25	0.2011 (2)	0.0766 (2)	0.28404 (10)	0.0630 (6)	
H25A	0.2418	0.1468	0.2836	0.076*	
C26	0.2101 (2)	0.0263 (2)	0.23068 (10)	0.0700 (7)	
H26A	0.2900	0.0102	0.2231	0.084*	
H26B	0.1678	−0.0421	0.2300	0.084*	
C27	0.2561 (3)	0.0042 (3)	0.32510 (12)	0.0888 (9)	
H27A	0.3369	−0.0020	0.3183	0.133*	
H27B	0.2217	−0.0672	0.3241	0.133*	
H27C	0.2446	0.0361	0.3588	0.133*	
C28	0.0022 (3)	0.1973 (2)	−0.32408 (10)	0.0747 (7)	
C29	−0.0428 (4)	0.2421 (3)	−0.37442 (12)	0.1170 (15)	
H29A	−0.0289	0.1902	−0.4018	0.175*	
H29B	−0.0044	0.3096	−0.3823	0.175*	
H29C	−0.1238	0.2551	−0.3714	0.175*	
C30	−0.1137 (3)	−0.0708 (2)	0.15718 (11)	0.0731 (7)	
C31	−0.2033 (3)	−0.0710 (3)	0.19839 (12)	0.0910 (9)	
H31A	−0.2010	−0.1394	0.2168	0.137*	
H31B	−0.2775	−0.0619	0.1828	0.137*	
H31C	−0.1893	−0.0119	0.2222	0.137*	
C32	−0.0957 (2)	0.2042 (2)	0.25880 (10)	0.0671 (7)	
C33	−0.1545 (3)	0.1791 (3)	0.30966 (11)	0.0949 (11)	
H33A	−0.2336	0.2008	0.3077	0.142*	
H33B	−0.1174	0.2185	0.3372	0.142*	
H33C	−0.1501	0.1019	0.3165	0.142*	
O1	−0.00373 (18)	0.27141 (14)	−0.28652 (7)	0.0729 (5)	
O2	0.0358 (3)	0.10678 (19)	−0.31779 (9)	0.1093 (9)	
O3	−0.09271 (14)	0.02925 (12)	0.13953 (6)	0.0584 (4)	
O4A	−0.0784 (7)	−0.1523 (5)	0.1346 (2)	0.106 (2)	0.7
O4B	−0.0395 (14)	−0.1374 (12)	0.1589 (5)	0.106 (4)	0.3
O5	0.16551 (14)	0.09843 (14)	0.19159 (6)	0.0634 (4)	
O6	−0.1443 (2)	0.2660 (2)	0.22891 (8)	0.1006 (8)	
C101	0.286 (3)	−0.111 (2)	0.0273 (17)	0.293 (18)	0.46
H10C	0.3148	−0.0639	0.0542	0.440*	0.46
H10D	0.2331	−0.0710	0.0059	0.440*	0.46
H10E	0.3490	−0.1363	0.0063	0.440*	0.46
C102	0.227 (3)	−0.205 (4)	0.0508 (12)	0.307 (16)	0.46

H10I	0.1780	−0.1793	0.0786	0.369*	0.46
H10J	0.2842	−0.2534	0.0662	0.369*	0.46
C103	0.156 (5)	−0.271 (4)	0.0135 (17)	0.51 (5)	0.46
H10K	0.0984	−0.2226	−0.0015	0.616*	0.46
H10L	0.2050	−0.2953	−0.0146	0.616*	0.46
C104	0.097 (4)	−0.368 (3)	0.0355 (15)	0.42 (4)	0.46
H10M	0.1251	−0.4287	0.0152	0.500*	0.46
H10N	0.1301	−0.3763	0.0698	0.500*	0.46
C105	−0.026 (4)	−0.3922 (18)	0.0431 (12)	0.29 (2)	0.46
H10O	−0.0353	−0.3978	0.0805	0.354*	0.46
H10P	−0.0357	−0.4659	0.0298	0.354*	0.46
C106	−0.132 (4)	−0.330 (4)	0.025 (2)	0.44 (4)	0.46
H10F	−0.1576	−0.2812	0.0514	0.665*	0.46
H10G	−0.1925	−0.3819	0.0170	0.665*	0.46
H10H	−0.1141	−0.2896	−0.0062	0.665*	0.46
C201	0.333 (4)	−0.081 (3)	−0.014 (2)	0.28 (2)	0.34
H20B	0.3018	−0.0096	−0.0199	0.423*	0.34
H20C	0.3818	−0.1015	−0.0423	0.423*	0.34
H20D	0.3761	−0.0813	0.0178	0.423*	0.34
C202	0.236 (4)	−0.162 (3)	−0.0096 (18)	0.31 (3)	0.34
H20H	0.1738	−0.1298	0.0098	0.367*	0.34
H20I	0.2083	−0.1798	−0.0441	0.367*	0.34
C203	0.274 (3)	−0.264 (3)	0.016 (2)	0.267 (19)	0.34
H20J	0.3068	−0.2456	0.0500	0.321*	0.34
H20K	0.3327	−0.2988	−0.0043	0.321*	0.34
C204	0.174 (3)	−0.345 (3)	0.025 (2)	0.30 (3)	0.34
H20L	0.1731	−0.3992	−0.0029	0.365*	0.34
H20M	0.1829	−0.3828	0.0577	0.365*	0.34
C205	0.064 (4)	−0.278 (4)	0.024 (3)	0.52 (6)	0.34
H20N	0.0573	−0.2432	0.0580	0.621*	0.34
H20O	0.0742	−0.2198	−0.0011	0.621*	0.34
C206	−0.049 (4)	−0.334 (6)	0.013 (4)	0.60 (8)	0.34
H20E	−0.0720	−0.3175	−0.0223	0.904*	0.34
H20F	−0.1062	−0.3089	0.0365	0.904*	0.34
H20G	−0.0396	−0.4120	0.0162	0.904*	0.34

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0628 (13)	0.0644 (14)	0.0507 (12)	−0.0102 (12)	0.0027 (11)	0.0054 (11)
C2	0.0828 (16)	0.0650 (15)	0.0497 (12)	−0.0087 (14)	0.0082 (12)	0.0072 (12)
C3	0.0723 (15)	0.0647 (14)	0.0438 (11)	0.0051 (12)	0.0018 (10)	0.0098 (11)
C4	0.0687 (14)	0.0737 (16)	0.0478 (12)	−0.0062 (13)	−0.0075 (11)	0.0009 (12)
C5	0.0523 (11)	0.0646 (14)	0.0455 (11)	−0.0043 (11)	0.0004 (9)	0.0021 (10)
C6	0.0794 (16)	0.0605 (14)	0.0485 (12)	−0.0132 (13)	0.0006 (11)	−0.0048 (11)
C7	0.0938 (18)	0.0540 (13)	0.0507 (13)	−0.0121 (13)	0.0000 (12)	0.0008 (11)
C8	0.0556 (11)	0.0518 (12)	0.0435 (11)	−0.0056 (10)	0.0012 (9)	0.0017 (10)
C9	0.0445 (9)	0.0512 (11)	0.0450 (10)	0.0020 (9)	0.0018 (8)	0.0043 (9)

C10	0.0485 (10)	0.0537 (12)	0.0442 (10)	0.0022 (10)	0.0022 (8)	0.0041 (9)
C11	0.0664 (13)	0.0550 (13)	0.0475 (11)	−0.0102 (11)	−0.0017 (10)	0.0069 (10)
C12	0.0598 (12)	0.0577 (13)	0.0479 (11)	−0.0088 (11)	−0.0033 (10)	0.0035 (10)
C13	0.0451 (10)	0.0464 (11)	0.0427 (10)	0.0017 (9)	0.0003 (8)	0.0035 (9)
C14	0.0559 (11)	0.0492 (11)	0.0450 (11)	0.0008 (10)	0.0038 (9)	0.0034 (9)
C15	0.0924 (18)	0.0519 (13)	0.0524 (13)	−0.0123 (13)	0.0015 (13)	0.0033 (11)
C16	0.0640 (12)	0.0478 (11)	0.0494 (12)	0.0065 (11)	0.0076 (10)	0.0044 (10)
C17	0.0473 (10)	0.0513 (11)	0.0453 (11)	0.0042 (9)	0.0020 (9)	0.0039 (9)
C18	0.0545 (12)	0.0680 (15)	0.0508 (12)	0.0119 (11)	−0.0013 (10)	0.0030 (11)
C19	0.0675 (14)	0.0796 (17)	0.0541 (13)	0.0201 (14)	−0.0013 (11)	0.0066 (13)
C20	0.0586 (12)	0.0519 (12)	0.0439 (11)	0.0088 (10)	−0.0002 (9)	0.0033 (9)
C21	0.0969 (19)	0.0523 (13)	0.0546 (13)	0.0006 (14)	−0.0073 (13)	−0.0002 (11)
C22	0.0537 (11)	0.0494 (11)	0.0493 (11)	0.0044 (10)	0.0000 (9)	0.0036 (10)
C23	0.0592 (12)	0.0545 (12)	0.0462 (11)	0.0026 (11)	−0.0024 (9)	0.0010 (10)
C24	0.0725 (14)	0.0600 (13)	0.0467 (11)	0.0003 (12)	−0.0038 (11)	0.0050 (11)
C25	0.0677 (14)	0.0560 (13)	0.0653 (14)	0.0014 (12)	−0.0151 (12)	0.0077 (12)
C26	0.0734 (16)	0.0723 (16)	0.0643 (15)	0.0201 (14)	−0.0021 (13)	0.0139 (13)
C27	0.103 (2)	0.087 (2)	0.0760 (18)	0.0138 (18)	−0.0292 (17)	0.0129 (16)
C28	0.109 (2)	0.0649 (16)	0.0504 (13)	−0.0007 (17)	0.0046 (14)	−0.0018 (12)
C29	0.195 (4)	0.105 (3)	0.0506 (16)	0.000 (3)	−0.008 (2)	0.0054 (17)
C30	0.090 (2)	0.0578 (15)	0.0709 (17)	−0.0037 (15)	0.0106 (15)	0.0163 (14)
C31	0.103 (2)	0.085 (2)	0.084 (2)	−0.0164 (19)	0.0232 (18)	0.0194 (17)
C32	0.0706 (15)	0.0810 (17)	0.0498 (12)	0.0131 (14)	0.0009 (12)	−0.0039 (13)
C33	0.0777 (18)	0.139 (3)	0.0684 (18)	0.020 (2)	0.0171 (15)	0.0072 (19)
O1	0.1068 (13)	0.0673 (10)	0.0445 (8)	0.0106 (11)	−0.0007 (9)	0.0058 (8)
O2	0.173 (3)	0.0748 (14)	0.0803 (14)	0.0225 (16)	−0.0116 (15)	−0.0131 (11)
O3	0.0677 (9)	0.0522 (9)	0.0552 (9)	0.0003 (8)	0.0112 (8)	0.0089 (7)
O4A	0.169 (6)	0.0501 (19)	0.099 (4)	−0.003 (3)	0.043 (3)	0.002 (3)
O4B	0.128 (10)	0.065 (7)	0.125 (11)	0.030 (7)	0.042 (8)	0.040 (7)
O5	0.0620 (9)	0.0723 (10)	0.0560 (9)	0.0210 (8)	0.0059 (7)	0.0161 (8)
O6	0.1014 (15)	0.1323 (19)	0.0680 (12)	0.0602 (15)	0.0140 (11)	0.0117 (13)
C101	0.21 (3)	0.154 (19)	0.52 (6)	0.004 (18)	−0.05 (3)	0.02 (3)
C102	0.24 (3)	0.35 (5)	0.33 (4)	0.08 (4)	0.06 (3)	0.02 (4)
C103	0.62 (9)	0.47 (9)	0.45 (6)	0.11 (7)	−0.36 (7)	0.06 (6)
C104	0.71 (10)	0.28 (4)	0.26 (3)	−0.14 (6)	0.22 (5)	−0.05 (3)
C105	0.51 (7)	0.139 (14)	0.23 (3)	0.01 (3)	0.11 (4)	−0.031 (15)
C106	0.50 (8)	0.37 (7)	0.46 (7)	−0.25 (6)	−0.01 (7)	−0.04 (6)
C201	0.27 (4)	0.16 (2)	0.41 (6)	−0.05 (3)	0.07 (4)	0.04 (3)
C202	0.34 (5)	0.25 (4)	0.32 (5)	−0.01 (4)	−0.12 (5)	−0.08 (4)
C203	0.26 (4)	0.19 (3)	0.35 (5)	0.07 (3)	−0.04 (4)	−0.07 (3)
C204	0.18 (3)	0.37 (6)	0.37 (6)	0.01 (4)	−0.04 (3)	0.16 (5)
C205	0.80 (13)	0.32 (6)	0.44 (9)	0.25 (8)	−0.04 (9)	−0.21 (6)
C206	0.81 (15)	0.43 (9)	0.56 (11)	−0.17 (10)	0.23 (10)	0.22 (8)

Geometric parameters (Å, º)

C1—C2	1.533 (3)	C25—C26	1.507 (4)
C1—C10	1.549 (3)	C25—C27	1.519 (4)

C1—H1A	0.9700	C25—H25A	0.9800
C1—H1B	0.9700	C26—O5	1.434 (3)
C2—C3	1.498 (4)	C26—H26A	0.9700
C2—H2A	0.9700	C26—H26B	0.9700
C2—H2B	0.9700	C27—H27A	0.9600
C3—O1	1.462 (3)	C27—H27B	0.9600
C3—C4	1.515 (4)	C27—H27C	0.9600
C3—H3A	0.9800	C28—O2	1.183 (3)
C4—C5	1.516 (3)	C28—O1	1.326 (3)
C4—H4A	0.9700	C28—C29	1.500 (4)
C4—H4B	0.9700	C29—H29A	0.9600
C5—C6	1.317 (3)	C29—H29B	0.9600
C5—C10	1.522 (3)	C29—H29C	0.9600
C6—C7	1.496 (3)	C30—O4B	1.188 (15)
C6—H6A	0.9300	C30—O4A	1.224 (7)
C7—C8	1.526 (3)	C30—O3	1.326 (3)
C7—H7A	0.9700	C30—C31	1.488 (4)
C7—H7B	0.9700	C31—H31A	0.9600
C8—C14	1.523 (3)	C31—H31B	0.9600
C8—C9	1.537 (3)	C31—H31C	0.9600
C8—H8A	0.9800	C32—O6	1.217 (3)
C9—C11	1.531 (3)	C32—C33	1.509 (4)
C9—C10	1.556 (3)	C33—H33A	0.9600
C9—H9A	0.9800	C33—H33B	0.9600
C10—C19	1.533 (3)	C33—H33C	0.9600
C11—C12	1.539 (3)	C101—C102	1.47 (2)
C11—H11A	0.9700	C101—H10C	0.9600
C11—H11B	0.9700	C101—H10D	0.9600
C12—C13	1.535 (3)	C101—H10E	0.9600
C12—H12A	0.9700	C102—C103	1.50 (2)
C12—H12B	0.9700	C102—H10I	0.9700
C13—C14	1.535 (3)	C102—H10J	0.9700
C13—C18	1.537 (3)	C103—C104	1.48 (2)
C13—C17	1.558 (3)	C103—H10K	0.9700
C14—C15	1.518 (3)	C103—H10L	0.9700
C14—H14A	0.9800	C104—C105	1.47 (2)
C15—C16	1.527 (3)	C104—H10M	0.9700
C15—H15A	0.9700	C104—H10N	0.9700
C15—H15B	0.9700	C105—C106	1.53 (3)
C16—O3	1.454 (3)	C105—H10O	0.9700
C16—C17	1.551 (3)	C105—H10P	0.9700
C16—H16A	0.9800	C106—H10F	0.9600
C17—C20	1.536 (3)	C106—H10G	0.9600
C17—H17A	0.9800	C106—H10H	0.9600
C18—H18A	0.9600	C201—C202	1.50 (2)
C18—H18B	0.9600	C201—H20B	0.9600
C18—H18C	0.9600	C201—H20C	0.9600
C19—H19A	0.9600	C201—H20D	0.9600

C19—H19B	0.9600	C202—C203	1.48 (2)
C19—H19C	0.9600	C202—H20H	0.9700
C20—C22	1.512 (3)	C202—H20I	0.9700
C20—C21	1.538 (3)	C203—C204	1.54 (2)
C20—H20A	0.9800	C203—H20J	0.9700
C21—H21A	0.9600	C203—H20K	0.9700
C21—H21B	0.9600	C204—C205	1.52 (3)
C21—H21C	0.9600	C204—H20L	0.9700
C22—C23	1.351 (3)	C204—H20M	0.9700
C22—O5	1.356 (3)	C205—C206	1.52 (3)
C23—C32	1.472 (4)	C205—H20N	0.9700
C23—C24	1.515 (3)	C205—H20O	0.9700
C24—C25	1.510 (4)	C206—H20E	0.9600
C24—H24A	0.9700	C206—H20F	0.9600
C24—H24B	0.9700	C206—H20G	0.9600

C2—C1—C10	113.7 (2)	C25—C24—H24B	109.3
C2—C1—H1A	108.8	C23—C24—H24B	109.3
C10—C1—H1A	108.8	H24A—C24—H24B	107.9
C2—C1—H1B	108.8	C26—C25—C24	108.1 (2)
C10—C1—H1B	108.8	C26—C25—C27	111.6 (2)
H1A—C1—H1B	107.7	C24—C25—C27	112.4 (2)
C3—C2—C1	110.0 (2)	C26—C25—H25A	108.2
C3—C2—H2A	109.7	C24—C25—H25A	108.2
C1—C2—H2A	109.7	C27—C25—H25A	108.2
C3—C2—H2B	109.7	O5—C26—C25	111.3 (2)
C1—C2—H2B	109.7	O5—C26—H26A	109.4
H2A—C2—H2B	108.2	C25—C26—H26A	109.4
O1—C3—C2	106.27 (19)	O5—C26—H26B	109.4
O1—C3—C4	109.6 (2)	C25—C26—H26B	109.4
C2—C3—C4	111.6 (2)	H26A—C26—H26B	108.0
O1—C3—H3A	109.8	C25—C27—H27A	109.5
C2—C3—H3A	109.8	C25—C27—H27B	109.5
C4—C3—H3A	109.8	H27A—C27—H27B	109.5
C3—C4—C5	111.77 (19)	C25—C27—H27C	109.5
C3—C4—H4A	109.3	H27A—C27—H27C	109.5
C5—C4—H4A	109.3	H27B—C27—H27C	109.5
C3—C4—H4B	109.3	O2—C28—O1	123.7 (3)
C5—C4—H4B	109.3	O2—C28—C29	125.0 (3)
H4A—C4—H4B	107.9	O1—C28—C29	111.2 (3)
C6—C5—C4	119.9 (2)	C28—C29—H29A	109.5
C6—C5—C10	123.7 (2)	C28—C29—H29B	109.5
C4—C5—C10	116.4 (2)	H29A—C29—H29B	109.5
C5—C6—C7	124.8 (2)	C28—C29—H29C	109.5
C5—C6—H6A	117.6	H29A—C29—H29C	109.5
C7—C6—H6A	117.6	H29B—C29—H29C	109.5
C6—C7—C8	111.6 (2)	O4B—C30—O3	120.6 (8)
C6—C7—H7A	109.3	O4A—C30—O3	121.6 (4)

C8—C7—H7A	109.3	O4B—C30—C31	118.9 (7)
C6—C7—H7B	109.3	O4A—C30—C31	124.9 (4)
C8—C7—H7B	109.3	O3—C30—C31	112.0 (3)
H7A—C7—H7B	108.0	C30—C31—H31A	109.5
C14—C8—C7	111.60 (18)	C30—C31—H31B	109.5
C14—C8—C9	109.69 (17)	H31A—C31—H31B	109.5
C7—C8—C9	109.76 (17)	C30—C31—H31C	109.5
C14—C8—H8A	108.6	H31A—C31—H31C	109.5
C7—C8—H8A	108.6	H31B—C31—H31C	109.5
C9—C8—H8A	108.6	O6—C32—C23	124.9 (2)
C11—C9—C8	112.35 (17)	O6—C32—C33	117.5 (3)
C11—C9—C10	112.98 (18)	C23—C32—C33	117.5 (2)
C8—C9—C10	111.99 (17)	C32—C33—H33A	109.5
C11—C9—H9A	106.3	C32—C33—H33B	109.5
C8—C9—H9A	106.3	H33A—C33—H33B	109.5
C10—C9—H9A	106.3	C32—C33—H33C	109.5
C5—C10—C19	109.64 (19)	H33A—C33—H33C	109.5
C5—C10—C1	107.22 (18)	H33B—C33—H33C	109.5
C19—C10—C1	109.38 (19)	C28—O1—C3	118.0 (2)
C5—C10—C9	110.49 (18)	C30—O3—C16	116.84 (19)
C19—C10—C9	110.96 (17)	C22—O5—C26	118.21 (17)
C1—C10—C9	109.05 (17)	C102—C101—C106^{i}	104 (3)
C9—C11—C12	115.15 (18)	C102—C101—H10C	109.5
C9—C11—H11A	108.5	C106^{i}—C101—H10C	126.5
C12—C11—H11A	108.5	C102—C101—H10D	109.5
C9—C11—H11B	108.5	C106^{i}—C101—H10D	96.7
C12—C11—H11B	108.5	H10C—C101—H10D	109.5
H11A—C11—H11B	107.5	C102—C101—H10E	109.5
C13—C12—C11	111.63 (18)	H10C—C101—H10E	109.5
C13—C12—H12A	109.3	H10D—C101—H10E	109.5
C11—C12—H12A	109.3	C101—C102—C103	114 (3)
C13—C12—H12B	109.3	C101—C102—H10I	108.7
C11—C12—H12B	109.3	C103—C102—H10I	108.7
H12A—C12—H12B	108.0	C101—C102—H10J	108.7
C12—C13—C14	105.79 (17)	C103—C102—H10J	108.7
C12—C13—C18	111.18 (18)	H10I—C102—H10J	107.6
C14—C13—C18	112.38 (17)	C104—C103—C102	116 (3)
C12—C13—C17	115.77 (17)	C104—C103—H10K	108.3
C14—C13—C17	100.78 (15)	C102—C103—H10K	108.3
C18—C13—C17	110.45 (17)	C104—C103—H10L	108.3
C15—C14—C8	118.96 (19)	C102—C103—H10L	108.3
C15—C14—C13	104.12 (17)	H10K—C103—H10L	107.4
C8—C14—C13	114.83 (17)	C105—C104—C103	132 (3)
C15—C14—H14A	106.0	C105—C104—H10M	104.3
C8—C14—H14A	106.0	C103—C104—H10M	104.3
C13—C14—H14A	106.0	C105—C104—H10N	104.3
C14—C15—C16	103.34 (19)	C103—C104—H10N	104.3
C14—C15—H15A	111.1	H10M—C104—H10N	105.6

C16—C15—H15A	111.1	C104—C105—C106	130 (3)
C14—C15—H15B	111.1	C104—C105—H10O	104.7
C16—C15—H15B	111.1	C106—C105—H10O	104.7
H15A—C15—H15B	109.1	C104—C105—H10P	104.7
O3—C16—C15	110.4 (2)	C106—C105—H10P	104.7
O3—C16—C17	110.43 (17)	H10O—C105—H10P	105.7
C15—C16—C17	107.33 (18)	C105—C106—H10F	109.5
O3—C16—H16A	109.6	C105—C106—H10G	109.5
C15—C16—H16A	109.6	H10F—C106—H10G	109.5
C17—C16—H16A	109.6	C105—C106—H10H	109.5
C20—C17—C16	114.60 (17)	H10F—C106—H10H	109.5
C20—C17—C13	117.73 (17)	H10G—C106—H10H	109.5
C16—C17—C13	104.46 (17)	C202—C201—H20B	109.5
C20—C17—H17A	106.4	C202—C201—H20C	109.5
C16—C17—H17A	106.4	H20B—C201—H20C	109.5
C13—C17—H17A	106.4	C202—C201—H20D	109.5
C13—C18—H18A	109.5	H20B—C201—H20D	109.5
C13—C18—H18B	109.5	H20C—C201—H20D	109.5
H18A—C18—H18B	109.5	C203—C202—C201	112 (3)
C13—C18—H18C	109.5	C203—C202—H20H	109.3
H18A—C18—H18C	109.5	C201—C202—H20H	109.3
H18B—C18—H18C	109.5	C203—C202—H20I	109.3
C10—C19—H19A	109.5	C201—C202—H20I	109.3
C10—C19—H19B	109.5	H20H—C202—H20I	108.0
H19A—C19—H19B	109.5	C202—C203—C204	113 (2)
C10—C19—H19C	109.5	C202—C203—H20J	109.1
H19A—C19—H19C	109.5	C204—C203—H20J	109.1
H19B—C19—H19C	109.5	C202—C203—H20K	109.1
C22—C20—C17	113.26 (17)	C204—C203—H20K	109.1
C22—C20—C21	107.06 (18)	H20J—C203—H20K	107.8
C17—C20—C21	112.67 (18)	C205—C204—C203	107 (3)
C22—C20—H20A	107.9	C205—C204—H20L	110.4
C17—C20—H20A	107.9	C203—C204—H20L	110.4
C21—C20—H20A	107.9	C205—C204—H20M	110.4
C20—C21—H21A	109.5	C203—C204—H20M	110.4
C20—C21—H21B	109.5	H20L—C204—H20M	108.6
H21A—C21—H21B	109.5	C206—C205—C204	119 (3)
C20—C21—H21C	109.5	C206—C205—H20N	107.5
H21A—C21—H21C	109.5	C204—C205—H20N	107.5
H21B—C21—H21C	109.5	C206—C205—H20O	107.5
C23—C22—O5	122.72 (19)	C204—C205—H20O	107.5
C23—C22—C20	127.61 (19)	H20N—C205—H20O	107.0
O5—C22—C20	109.51 (17)	C205—C206—H20E	109.5
C22—C23—C32	122.1 (2)	C205—C206—H20F	109.5
C22—C23—C24	120.4 (2)	H20E—C206—H20F	109.5
C32—C23—C24	117.4 (2)	C205—C206—H20G	109.5
C25—C24—C23	111.8 (2)	H20E—C206—H20G	109.5
C25—C24—H24A	109.3	H20F—C206—H20G	109.5

C23—C24—H24A	109.3		
C10—C1—C2—C3	−58.5 (3)	O3—C16—C17—C20	19.7 (2)
C1—C2—C3—O1	176.0 (2)	C15—C16—C17—C20	140.0 (2)
C1—C2—C3—C4	56.6 (3)	O3—C16—C17—C13	−110.59 (18)
O1—C3—C4—C5	−170.4 (2)	C15—C16—C17—C13	9.7 (2)
C2—C3—C4—C5	−53.0 (3)	C12—C13—C17—C20	85.2 (2)
C3—C4—C5—C6	−128.3 (3)	C14—C13—C17—C20	−161.23 (18)
C3—C4—C5—C10	51.4 (3)	C18—C13—C17—C20	−42.2 (2)
C4—C5—C6—C7	−178.7 (2)	C12—C13—C17—C16	−146.38 (18)
C10—C5—C6—C7	1.7 (4)	C14—C13—C17—C16	−32.8 (2)
C5—C6—C7—C8	16.8 (4)	C18—C13—C17—C16	86.2 (2)
C6—C7—C8—C14	−168.6 (2)	C16—C17—C20—C22	53.6 (2)
C6—C7—C8—C9	−46.8 (3)	C13—C17—C20—C22	177.03 (18)
C14—C8—C9—C11	−47.4 (2)	C16—C17—C20—C21	175.31 (19)
C7—C8—C9—C11	−170.39 (19)	C13—C17—C20—C21	−61.3 (3)
C14—C8—C9—C10	−175.84 (17)	C17—C20—C22—C23	−145.6 (2)
C7—C8—C9—C10	61.2 (2)	C21—C20—C22—C23	89.6 (3)
C6—C5—C10—C19	−111.6 (3)	C17—C20—C22—O5	38.9 (2)
C4—C5—C10—C19	68.7 (3)	C21—C20—C22—O5	−85.9 (2)
C6—C5—C10—C1	129.8 (2)	O5—C22—C23—C32	−174.2 (2)
C4—C5—C10—C1	−49.9 (3)	C20—C22—C23—C32	10.9 (4)
C6—C5—C10—C9	11.0 (3)	O5—C22—C23—C24	5.4 (4)
C4—C5—C10—C9	−168.65 (18)	C20—C22—C23—C24	−169.5 (2)
C2—C1—C10—C5	53.1 (3)	C22—C23—C24—C25	15.4 (3)
C2—C1—C10—C19	−65.7 (3)	C32—C23—C24—C25	−164.9 (2)
C2—C1—C10—C9	172.8 (2)	C23—C24—C25—C26	−46.1 (3)
C11—C9—C10—C5	−170.04 (18)	C23—C24—C25—C27	−169.6 (2)
C8—C9—C10—C5	−42.0 (2)	C24—C25—C26—O5	60.1 (3)
C11—C9—C10—C19	−48.2 (3)	C27—C25—C26—O5	−175.8 (2)
C8—C9—C10—C19	79.9 (2)	C22—C23—C32—O6	−13.3 (4)
C11—C9—C10—C1	72.3 (2)	C24—C23—C32—O6	167.0 (3)
C8—C9—C10—C1	−159.58 (18)	C22—C23—C32—C33	167.4 (3)
C8—C9—C11—C12	46.7 (3)	C24—C23—C32—C33	−12.3 (3)
C10—C9—C11—C12	174.63 (19)	O2—C28—O1—C3	−1.2 (5)
C9—C11—C12—C13	−52.4 (3)	C29—C28—O1—C3	176.9 (3)
C11—C12—C13—C14	56.5 (2)	C2—C3—O1—C28	155.7 (3)
C11—C12—C13—C18	−65.8 (2)	C4—C3—O1—C28	−83.5 (3)
C11—C12—C13—C17	167.13 (19)	O4B—C30—O3—C16	29.4 (9)
C7—C8—C14—C15	−55.4 (3)	O4A—C30—O3—C16	−15.8 (6)
C9—C8—C14—C15	−177.3 (2)	C31—C30—O3—C16	177.1 (2)
C7—C8—C14—C13	−179.72 (19)	C15—C16—O3—C30	88.5 (3)
C9—C8—C14—C13	58.4 (2)	C17—C16—O3—C30	−153.0 (2)
C12—C13—C14—C15	165.85 (18)	C23—C22—O5—C26	8.4 (3)
C18—C13—C14—C15	−72.6 (2)	C20—C22—O5—C26	−175.9 (2)
C17—C13—C14—C15	44.9 (2)	C25—C26—O5—C22	−42.1 (3)
C12—C13—C14—C8	−62.4 (2)	C101—C102—C103—C104	179 (4)
C18—C13—C14—C8	59.2 (2)	C102—C103—C104—C105	114 (5)

C17—C13—C14—C8	176.73 (17)	C103—C104—C105—C106	8 (7)
C8—C14—C15—C16	−168.6 (2)	C201—C202—C203—C204	176 (5)
C13—C14—C15—C16	−39.2 (2)	C202—C203—C204—C205	−23 (8)
C14—C15—C16—O3	138.11 (19)	C203—C204—C205—C206	159 (7)
C14—C15—C16—C17	17.7 (2)		

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20*A*···O6	0.98	2.17	2.859 (3)	126
C26—H26*B*···O6^{ii}	0.97	2.59	3.431 (4)	146
C33—H33*B*···O4*B*^{iii}	0.96	2.54	3.283 (15)	135

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