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A C-14 oxygenated taxoid from the heartwood of *Taxus wallichiana*

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Abstract

The title compound [Texa-4(20)11-diene-2α, 5α, 10β, 14β-tetraol-2α, 5α, 10β-triacetate-14β(2'-methyl butyrate)], $C_{31}H_{46}O_8$, a taxoid, contains one fused-ring system (A/B/C) and the overall conformation is similar to that of other taxane systems.

Experimental

Compound (1) was isolated from a methanol extract of the heartwood of Taxus wallichiana (Chattopadhyay *et al.*, 1996). Diffraction quality crystals were grown from Acetone/Hexane solution at room temperature.

Refinement

The structure was solved by direct methods and refined anisotropically on non-H atoms by using full matrix least-squares methods. All H-atoms were placed in geometrically idealized positions and allowed to ride on their parent atoms, to which each was bonded for the final cycles of refinement. Four reflections [most disagreeable, $\Delta(F^2)/\sigma>5.0$] were suppressed during the last cycles of refinement. The thermal parameters of the four atoms O4, C27, C28 and C29 in the side chains at C5 and C14 showed highest thermal motions (0.15 Å^2 < U_{eq}<0.30 Å^2). An attempt to resolve the disordered positions for these atoms and their refinement were unsuccessful.

Computing details

Data collection: CAD-4-MACH/PC (Enraf-Nonius, 1993); cell refinement: CAD-4-MACH/PC (Enraf-Nonius, 1993); data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993); molecular graphics: *NRCVAX* (Gabe *et al.*, 1989); software used to prepare material for publication: *SHELXL93* (Sheldrick, 1993).

Taxa-4(20),11-diene-2α,5α,10β,14β-tetrayl 2α,5α,10β-triacetate 14β-(2-methylbutyrate)

Crystal data

$C_{31}H_{46}O_8$	V = 3070 (3) Å^3
M_r = 546.68	Z = 4
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
a = 9.789 (7) Å	μ = 0.08 mm^{-1}
b = 12.754 (3) Å	T = 293 (2) K

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c = 24.586 (9) Å	0.4 × 0.3 × 0.2 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	R_{int} = 0.0000
Absorption correction: none	3 standard reflections
3055 measured reflections	every 60 min
3055 independent reflections	intensity decay: <0.5%
1460 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.067	Riding
$wR(F^2)$ = 0.270	$\Delta\rho_{max}$ = 0.24 e Å^{-3}
S = 0.73	$\Delta\rho_{min}$ = −0.24 e Å^{-3}
3051 reflections	Absolute structure: Flack (1983)
361 parameters	Flack parameter: −2 (3)

Table 1
Selected geometric parameters (°)

C15—C1—C2—C3	80.3 (7)	C15—C11—C12—C13	10.0 (9)
C14—C1—C2—C3	−47.2 (7)	C11—C12—C13—C14	30.2 (9)
C1—C2—C3—C4	120.7 (6)	C15—C1—C14—C13	−21.9 (8)
C1—C2—C3—C8	−108.5 (6)	C2—C1—C14—C13	106.0 (6)
C2—C3—C4—C5	−160.8 (6)	C12—C13—C14—C1	−22.4 (8)
C8—C3—C4—C5	66.5 (7)	C14—C1—C15—C11	58.3 (7)
C3—C4—C5—C6	−61.1 (8)	C2—C1—C15—C11	−68.5 (7)
C4—C5—C6—C7	50.3 (9)	C12—C11—C15—C1	−54.5 (7)
C5—C6—C7—C8	−48.3 (9)	O7—C25—C26—C27	154.0 (12)
C6—C7—C8—C9	177.9 (6)	O7—C25—C26—C28	−90.3 (10)
C6—C7—C8—C3	51.0 (7)	C25—C26—C27—C29	−67.5 (15)
C4—C3—C8—C9	179.7 (6)	C28—C26—C27—C29	176.1 (13)
C2—C3—C8—C9	43.8 (8)	O2—C16—O1—C2	2.2 (11)
C4—C3—C8—C7	−59.0 (7)	C17—C16—O1—C2	−174.2 (7)
C2—C3—C8—C7	165.1 (5)	O4—C19—O3—C5	8.0 (21)
C7—C8—C9—C10	−65.0 (8)	C20—C19—O3—C5	−179.8 (10)
C3—C8—C9—C10	55.9 (9)	O6—C22—O5—C10	5.3 (15)
C8—C9—C10—C11	−55.3 (10)	C23—C22—O5—C10	−176.3 (7)
C9—C10—C11—C12	105.7 (8)	O8—C25—O7—C14	−4.1 (12)
C9—C10—C11—C15	−59.6 (9)	C26—C25—O7—C14	170.3 (8)
C10—C11—C12—C13	−155.9 (6)		

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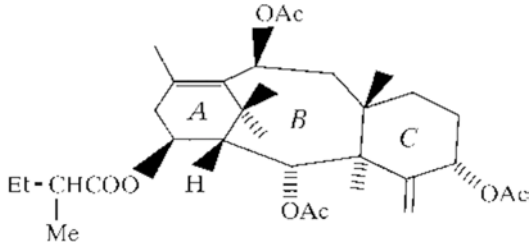
Scheme 1



supplementary materials

Taxa-4(20),11-diene-2α,5α,10β,14β-tetrayl 2α,5α,10β-triacetate 14β-(2-methylbutyrate)

Crystal data

$C_{31}H_{46}O_8$	$D_x = 1.183$ Mg m^{-3}
$M_r = 546.68$	Melting point: 107-108 K
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.789$ (7) Å	Cell parameters from 25 reflections
$b = 12.754$ (3) Å	$\theta = 9$–12.5º
$c = 24.586$ (9) Å	$\mu = 0.08$ mm^{-1}
$V = 3070$ (3) Å^3	$T = 293$ (2) K
$Z = 4$	Prism, colourless
$F_{000} = 1184$	0.4 × 0.3 × 0.2 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{int} = 0.0000$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0$º
Monochromator: graphite	$\theta_{min} = 1.7$º
$T = 293(2)$ K	$h = 0 \rightarrow 11$
ω–2θ scans	$k = 0 \rightarrow 15$
Absorption correction: none	$l = 0 \rightarrow 29$
3055 measured reflections	3 standard reflections
3055 independent reflections	every 60 min
1460 reflections with $I > 2\sigma(I)$	intensity decay: <0.5%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	Riding
$R[F^2 > 2\sigma(F^2)] = 0.067$	Calculated $w = 1/[\sigma^2(F_o^2) + (0.2499P)^2]$ where P $F_o^2 + 2F_c^2)/3$?
$wR(F^2) = 0.270$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
$S = 0.73$	$\Delta\rho_{min} = -0.24$ e Å^{-3}
3051 reflections	Extinction correction: none
361 parameters	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: −2 (3)
Secondary atom site location: difference Fourier map	

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 on F^2 for ALL reflections except for 4 with very negative F^2 or 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 $F^2 > 2\text{sigma}(F^2)$ 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}
C1	1.0435 (6)	0.4206 (5)	0.0764 (3)	0.053 (2)
H1	0.9937 (6)	0.3636 (5)	0.0585 (3)	0.063*
C2	0.9418 (6)	0.4726 (5)	0.1170 (2)	0.050 (2)
H2	0.9323 (6)	0.4264 (5)	0.1486 (2)	0.060*
C3	0.9830 (7)	0.5844 (5)	0.1377 (3)	0.054 (2)
H3	1.0661 (7)	0.6014 (5)	0.1173 (3)	0.064*
C4	0.8901 (7)	0.6753 (5)	0.1276 (3)	0.062 (2)
C5	0.9638 (8)	0.7772 (6)	0.1328 (4)	0.076 (2)
H5	0.9004 (8)	0.8348 (6)	0.1253 (4)	0.091*
C6	1.0197 (9)	0.7887 (6)	0.1899 (3)	0.081 (2)
H6A	1.0787 (9)	0.8498 (6)	0.1913 (3)	0.097*
H6B	0.9444 (9)	0.8004 (6)	0.2148 (3)	0.097*
C7	1.1014 (9)	0.6909 (6)	0.2087 (3)	0.072 (2)
H7A	1.1884 (9)	0.6901 (6)	0.1898 (3)	0.086*
H7B	1.1204 (9)	0.6975 (6)	0.2472 (3)	0.086*
C8	1.0291 (6)	0.5859 (6)	0.1988 (3)	0.056 (2)
C9	1.1252 (8)	0.4979 (6)	0.2175 (3)	0.067 (2)
H9A	1.0742 (8)	0.4328 (6)	0.2158 (3)	0.080*
H9B	1.1461 (8)	0.5104 (6)	0.2555 (3)	0.080*
C10	1.2619 (8)	0.4798 (6)	0.1878 (3)	0.066 (2)
H10	1.3191 (8)	0.5418 (6)	0.1938 (3)	0.079*
C11	1.2516 (6)	0.4626 (6)	0.1278 (3)	0.055 (2)
C12	1.2880 (6)	0.5372 (5)	0.0933 (3)	0.056 (2)
C13	1.2317 (6)	0.5420 (6)	0.0367 (3)	0.058 (2)
H13A	1.2309 (6)	0.6144 (6)	0.0246 (3)	0.070*
H13B	1.2915 (6)	0.5031 (6)	0.0125 (3)	0.070*
C14	1.0847 (7)	0.4967 (5)	0.0322 (2)	0.0502 (15)
H14	1.0196 (7)	0.5550 (5)	0.0319 (2)	0.060*
C15	1.1677 (7)	0.3701 (5)	0.1046 (3)	0.060 (2)
C16	0.7333 (8)	0.3900 (7)	0.0944 (4)	0.075 (2)
C17	0.6102 (9)	0.3948 (8)	0.0596 (5)	0.108 (3)
H17A	0.633 (2)	0.428 (5)	0.0258 (13)	0.162*
H17B	0.540 (3)	0.434 (5)	0.0778 (14)	0.162*
H17C	0.578 (5)	0.3251 (9)	0.053 (3)	0.162*
C18	0.7563 (8)	0.6782 (7)	0.1226 (3)	0.081 (2)
H18A	0.7056 (8)	0.6167 (7)	0.1254 (3)	0.098*

H18B	0.7124 (8)	0.7417 (7)	0.1163 (3)	0.098*
C19	1.0863 (13)	0.8561 (10)	0.0582 (5)	0.117 (4)
C20	1.2058 (13)	0.8496 (11)	0.0232 (5)	0.146 (5)
H20A	1.195 (5)	0.793 (5)	−0.002 (3)	0.218*
H20B	1.286 (2)	0.838 (8)	0.0450 (6)	0.218*
H20C	1.216 (6)	0.914 (3)	0.004 (3)	0.218*
C21	0.9046 (9)	0.5748 (7)	0.2365 (3)	0.079 (2)
H21A	0.851 (3)	0.516 (3)	0.2253 (15)	0.119*
H21B	0.850 (3)	0.637 (2)	0.2345 (19)	0.119*
H21C	0.9352 (9)	0.565 (5)	0.2732 (5)	0.119*
C22	1.4628 (13)	0.3895 (9)	0.2165 (4)	0.098 (3)
C23	1.5161 (15)	0.2925 (10)	0.2429 (5)	0.148 (5)
H23A	1.444 (3)	0.259 (5)	0.263 (4)	0.222*
H23B	1.588 (8)	0.3106 (14)	0.268 (4)	0.222*
H23C	1.551 (11)	0.246 (4)	0.2156 (7)	0.222*
C24	1.3809 (7)	0.6283 (6)	0.1060 (3)	0.071 (2)
H24A	1.3271 (8)	0.6872 (15)	0.118 (2)	0.107*
H24B	1.432 (4)	0.647 (3)	0.0740 (7)	0.107*
H24C	1.443 (4)	0.6089 (16)	0.1345 (16)	0.107*
C25	1.0704 (10)	0.5011 (7)	−0.0644 (3)	0.077 (2)
C26	1.0856 (14)	0.4352 (8)	−0.1151 (3)	0.108 (4)
H26	1.0496 (14)	0.3645 (8)	−0.1088 (3)	0.130*
C27	1.024 (3)	0.4805 (13)	−0.1618 (5)	0.214 (11)
H27A	1.056 (3)	0.5518 (13)	−0.1669 (5)	0.257*
H27B	1.047 (3)	0.4402 (13)	−0.1940 (5)	0.257*
C28	1.2443 (18)	0.4303 (11)	−0.1281 (6)	0.180 (8)
H28A	1.261 (3)	0.376 (8)	−0.154 (4)	0.269*
H28B	1.294 (2)	0.415 (11)	−0.0953 (13)	0.269*
H28C	1.274 (3)	0.497 (4)	−0.142 (5)	0.269*
C29	0.8675 (18)	0.4791 (16)	−0.1520 (8)	0.206 (9)
H29A	0.8208 (18)	0.478 (13)	−0.1863 (8)	0.309*
H29B	0.841 (3)	0.541 (7)	−0.132 (6)	0.309*
H29C	0.843 (3)	0.418 (7)	−0.131 (6)	0.309*
C30	1.2533 (7)	0.3080 (6)	0.0627 (3)	0.073 (2)
H30A	1.334 (3)	0.281 (4)	0.0801 (6)	0.109*
H30B	1.280 (5)	0.3536 (12)	0.0335 (12)	0.109*
H30C	1.200 (2)	0.251 (3)	0.0486 (17)	0.109*
C31	1.1215 (8)	0.2839 (6)	0.1446 (3)	0.074 (2)
H31A	1.1985 (14)	0.261 (3)	0.1655 (16)	0.110*
H31B	1.084 (5)	0.226 (2)	0.1245 (3)	0.110*
H31C	1.053 (4)	0.3116 (14)	0.1686 (15)	0.110*
O1	0.8128 (4)	0.4749 (3)	0.0886 (2)	0.0569 (11)
O2	0.7638 (7)	0.3179 (5)	0.1223 (3)	0.116 (2)
O3	1.0779 (6)	0.7821 (4)	0.0946 (2)	0.081 (2)
O4	1.0082 (15)	0.9254 (13)	0.0602 (6)	0.278 (11)
O5	1.3262 (6)	0.3920 (4)	0.2155 (2)	0.086 (2)
O6	1.5303 (8)	0.4593 (9)	0.1991 (4)	0.130 (3)
O7	1.0755 (5)	0.4412 (4)	−0.0200 (2)	0.0630 (13)
O8	1.0658 (7)	0.5933 (5)	−0.0632 (2)	0.094 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.041 (3)	0.063 (4)	0.054 (4)	0.000 (3)	−0.007 (3)	−0.003 (3)
C2	0.039 (3)	0.065 (4)	0.045 (3)	0.001 (3)	0.004 (3)	0.008 (3)
C3	0.047 (3)	0.060 (4)	0.054 (4)	0.002 (3)	0.008 (3)	−0.002 (3)
C4	0.053 (4)	0.066 (4)	0.066 (4)	0.015 (4)	0.004 (3)	0.001 (4)
C5	0.065 (5)	0.054 (4)	0.109 (6)	0.009 (4)	0.005 (4)	−0.004 (4)
C6	0.083 (6)	0.071 (5)	0.088 (6)	−0.001 (5)	0.006 (5)	−0.025 (4)
C7	0.076 (5)	0.086 (5)	0.053 (4)	−0.007 (5)	0.003 (4)	−0.017 (4)
C8	0.045 (3)	0.070 (4)	0.053 (4)	0.004 (3)	−0.001 (3)	0.000 (3)
C9	0.069 (4)	0.086 (5)	0.045 (4)	−0.001 (4)	−0.003 (3)	0.001 (4)
C10	0.064 (4)	0.070 (4)	0.064 (4)	0.008 (4)	−0.012 (3)	−0.005 (4)
C11	0.036 (3)	0.073 (4)	0.057 (4)	0.009 (3)	−0.005 (3)	−0.004 (3)
C12	0.034 (3)	0.069 (4)	0.065 (4)	0.002 (3)	0.008 (3)	−0.010 (4)
C13	0.043 (3)	0.072 (4)	0.059 (4)	−0.010 (3)	0.008 (3)	−0.002 (3)
C14	0.051 (4)	0.054 (3)	0.045 (3)	−0.001 (3)	−0.003 (3)	−0.005 (3)
C15	0.055 (4)	0.064 (4)	0.062 (4)	0.008 (3)	−0.008 (3)	−0.004 (4)
C16	0.054 (4)	0.067 (5)	0.105 (6)	−0.004 (4)	0.009 (4)	−0.007 (5)
C17	0.048 (4)	0.110 (7)	0.165 (10)	−0.006 (5)	−0.008 (5)	−0.023 (7)
C18	0.059 (5)	0.077 (5)	0.108 (6)	0.011 (4)	−0.003 (5)	−0.008 (5)
C19	0.103 (8)	0.128 (8)	0.120 (8)	0.034 (7)	0.017 (7)	0.060 (7)
C20	0.117 (9)	0.169 (11)	0.151 (10)	0.020 (9)	0.056 (8)	0.076 (9)
C21	0.082 (5)	0.088 (5)	0.067 (4)	−0.006 (5)	0.018 (4)	0.001 (4)
C22	0.098 (9)	0.112 (8)	0.085 (7)	0.041 (7)	−0.026 (6)	−0.021 (6)
C23	0.170 (12)	0.141 (10)	0.134 (9)	0.070 (10)	−0.073 (9)	−0.017 (8)
C24	0.044 (4)	0.084 (5)	0.085 (5)	−0.011 (4)	0.002 (4)	−0.013 (4)
C25	0.098 (6)	0.076 (5)	0.057 (4)	−0.034 (5)	−0.011 (4)	0.010 (4)
C26	0.188 (11)	0.089 (6)	0.048 (4)	−0.052 (7)	0.012 (6)	−0.005 (4)
C27	0.40 (3)	0.166 (13)	0.077 (8)	−0.118 (18)	−0.077 (13)	0.034 (8)
C28	0.233 (18)	0.139 (10)	0.167 (13)	−0.059 (12)	0.125 (14)	−0.053 (10)
C29	0.165 (14)	0.219 (18)	0.23 (2)	−0.062 (15)	−0.106 (15)	0.010 (15)
C30	0.049 (4)	0.081 (5)	0.088 (5)	0.015 (4)	0.004 (4)	−0.011 (4)
C31	0.074 (5)	0.071 (4)	0.076 (5)	0.003 (4)	−0.012 (4)	0.011 (4)
O1	0.035 (2)	0.065 (3)	0.071 (3)	−0.007 (2)	0.000 (2)	0.001 (2)
O2	0.079 (4)	0.094 (4)	0.174 (7)	−0.025 (4)	0.009 (5)	0.031 (5)
O3	0.077 (3)	0.072 (3)	0.095 (4)	0.002 (3)	0.028 (3)	0.018 (3)
O4	0.205 (12)	0.318 (17)	0.312 (17)	0.161 (13)	0.142 (12)	0.224 (15)
O5	0.084 (4)	0.100 (4)	0.073 (3)	0.024 (3)	−0.031 (3)	0.005 (3)
O6	0.074 (5)	0.187 (9)	0.128 (6)	0.035 (6)	−0.022 (4)	0.008 (6)
O7	0.075 (3)	0.066 (3)	0.048 (2)	−0.013 (2)	0.001 (2)	−0.006 (2)
O8	0.119 (5)	0.095 (5)	0.067 (3)	−0.013 (4)	−0.020 (3)	0.020 (3)

Geometric parameters (Å, °)

C1—C15	1.541 (9)	C17—H17B	0.96
C1—C14	1.512 (9)	C17—H17C	0.96
C1—C2	1.557 (9)	C18—H18A	0.93

C1—H1	0.98	C18—H18B	0.93
C2—O1	1.443 (7)	C19—O4	1.170 (14)
C2—C3	1.567 (10)	C19—O3	1.303 (11)
C2—H2	0.98	C19—C20	1.45 (2)
C3—C4	1.495 (9)	C20—H20A	0.96
C3—C8	1.567 (9)	C20—H20B	0.96
C3—H3	0.98	C20—H20C	0.96
C4—C18	1.316 (10)	C21—H21A	0.96
C4—C5	1.492 (11)	C21—H21B	0.96
C5—O3	1.462 (9)	C21—H21C	0.96
C5—C6	1.513 (12)	C22—O6	1.189 (13)
C5—H5	0.98	C22—O5	1.338 (13)
C6—C7	1.552 (11)	C22—C23	1.491 (15)
C6—H6A	0.97	C23—H23A	0.96
C6—H6B	0.97	C23—H23B	0.96
C7—C8	1.535 (10)	C23—H23C	0.96
C7—H7A	0.97	C24—H24A	0.96
C7—H7B	0.97	C24—H24B	0.96
C8—C9	1.535 (10)	C24—H24C	0.96
C8—C21	1.538 (10)	C25—O8	1.177 (9)
C9—C10	1.541 (10)	C25—O7	1.332 (9)
C9—H9A	0.97	C25—C26	1.511 (12)
C9—H9B	0.97	C26—C27	1.42 (2)
C10—O5	1.454 (8)	C26—C28	1.59 (2)
C10—C11	1.497 (10)	C26—H26	0.98
C10—H10	0.98	C27—C29	1.55 (3)
C11—C12	1.323 (9)	C27—H27A	0.97
C11—C15	1.546 (10)	C27—H27B	0.97
C12—C24	1.508 (9)	C28—H28A	0.96
C12—C13	1.499 (10)	C28—H28B	0.96
C13—C14	1.554 (9)	C28—H28C	0.96
C13—H13A	0.97	C29—H29A	0.96
C13—H13B	0.97	C29—H29B	0.96
C14—O7	1.469 (7)	C29—H29C	0.96
C14—H14	0.98	C30—H30A	0.96
C15—C31	1.542 (10)	C30—H30B	0.96
C15—C30	1.547 (10)	C30—H30C	0.96
C16—O2	1.186 (10)	C31—H31A	0.96
C16—O1	1.341 (9)	C31—H31B	0.96
C16—C17	1.479 (14)	C31—H31C	0.96
C17—H17A	0.96		
C15—C1—C14	112.5 (5)	O1—C16—C17	112.1 (8)
C15—C1—C2	113.2 (5)	C16—C17—H17A	109.5 (6)
C14—C1—C2	111.0 (5)	C16—C17—H17B	109.5 (5)
C15—C1—H1	106.6 (4)	H17A—C17—H17B	109.5
C14—C1—H1	106.6 (3)	C16—C17—H17C	109.5 (5)
C2—C1—H1	106.6 (3)	H17A—C17—H17C	109.5
O1—C2—C1	105.0 (5)	H17B—C17—H17C	109.5
O1—C2—C3	111.3 (5)	C4—C18—H18A	120.0 (5)

C1—C2—C3	115.5 (5)	C4—C18—H18B	120.0 (5)
O1—C2—H2	108.2 (3)	H18A—C18—H18B	120.0
C1—C2—H2	108.2 (3)	O4—C19—O3	118.5 (10)
C3—C2—H2	108.2 (3)	O4—C19—C20	126.4 (10)
C4—C3—C2	119.7 (5)	O3—C19—C20	114.6 (10)
C4—C3—C8	108.9 (5)	C19—C20—H20A	109.5 (8)
C2—C3—C8	113.4 (5)	C19—C20—H20B	109.5 (7)
C4—C3—H3	104.4 (4)	H20A—C20—H20B	109.5
C2—C3—H3	104.4 (3)	C19—C20—H20C	109.5 (6)
C8—C3—H3	104.4 (3)	H20A—C20—H20C	109.5
C18—C4—C3	130.0 (7)	H20B—C20—H20C	109.5
C18—C4—C5	117.7 (7)	C8—C21—H21A	109.5 (4)
C3—C4—C5	111.5 (6)	C8—C21—H21B	109.5 (4)
O3—C5—C4	110.6 (6)	H21A—C21—H21B	109.5
O3—C5—C6	108.4 (6)	C8—C21—H21C	109.5 (4)
C4—C5—C6	109.8 (7)	H21A—C21—H21C	109.5
O3—C5—H5	109.3 (4)	H21B—C21—H21C	109.5
C4—C5—H5	109.3 (4)	O6—C22—O5	122.1 (10)
C6—C5—H5	109.3 (4)	O6—C22—C23	125.7 (12)
C7—C6—C5	112.6 (6)	O5—C22—C23	112.2 (12)
C7—C6—H6A	109.1 (4)	C22—C23—H23A	109.5 (8)
C5—C6—H6A	109.1 (4)	C22—C23—H23B	109.5 (7)
C7—C6—H6B	109.1 (4)	H23A—C23—H23B	109.5
C5—C6—H6B	109.1 (4)	C22—C23—H23C	109.5 (6)
H6A—C6—H6B	107.8	H23A—C23—H23C	109.5
C8—C7—C6	114.6 (6)	H23B—C23—H23C	109.47 (6)
C8—C7—H7A	108.6 (4)	C12—C24—H24A	109.5 (3)
C6—C7—H7A	108.6 (4)	C12—C24—H24B	109.5 (4)
C8—C7—H7B	108.6 (4)	H24A—C24—H24B	109.5
C6—C7—H7B	108.6 (4)	C12—C24—H24C	109.5 (4)
H7A—C7—H7B	107.6	H24A—C24—H24C	109.5
C9—C8—C7	107.9 (6)	H24B—C24—H24C	109.5
C9—C8—C21	103.7 (6)	O8—C25—O7	123.7 (7)
C7—C8—C21	110.5 (6)	O8—C25—C26	125.4 (7)
C9—C8—C3	117.0 (6)	O7—C25—C26	110.6 (7)
C7—C8—C3	107.2 (6)	C25—C26—C27	113.6 (12)
C21—C8—C3	110.4 (5)	C25—C26—C28	106.5 (9)
C8—C9—C10	120.0 (6)	C27—C26—C28	105.5 (13)
C8—C9—H9A	107.3 (4)	C25—C26—H26	110.3 (4)
C10—C9—H9A	107.3 (4)	C27—C26—H26	110.3 (8)
C8—C9—H9B	107.3 (4)	C28—C26—H26	110.3 (7)
C10—C9—H9B	107.3 (3)	C29—C27—C26	106.7 (15)
H9A—C9—H9B	106.9	C29—C27—H27A	110.4 (11)
O5—C10—C11	112.3 (6)	C26—C27—H27A	110.4 (8)
O5—C10—C9	105.6 (6)	C29—C27—H27B	110.4 (10)
C11—C10—C9	115.5 (6)	C26—C27—H27B	110.4 (10)
O5—C10—H10	107.7 (4)	H27A—C27—H27B	108.6
C11—C10—H10	107.7 (4)	C26—C28—H28A	109.5 (6)
C9—C10—H10	107.7 (4)	C26—C28—H28B	109.5 (7)

C12—C11—C10	120.6 (6)	H28A—C28—H28B	109.5
C12—C11—C15	117.2 (6)	C26—C28—H28C	109.5 (7)
C10—C11—C15	120.7 (6)	H28A—C28—H28C	109.5
C11—C12—C24	125.8 (6)	H28B—C28—H28C	109.5
C11—C12—C13	121.7 (6)	C27—C29—H29A	109.5 (9)
C24—C12—C13	112.5 (6)	C27—C29—H29B	109.5 (11)
C14—C13—C12	113.0 (5)	H29A—C29—H29B	109.5
C14—C13—H13A	109.0 (4)	C27—C29—H29C	109.5 (10)
C12—C13—H13A	109.0 (4)	H29A—C29—H29C	109.5
C14—C13—H13B	109.0 (3)	H29B—C29—H29C	109.5
C12—C13—H13B	109.0 (4)	C15—C30—H30A	109.5 (4)
H13A—C13—H13B	107.8	C15—C30—H30B	109.5 (4)
O7—C14—C1	107.7 (5)	H30A—C30—H30B	109.5
O7—C14—C13	107.4 (5)	C15—C30—H30C	109.5 (4)
C1—C14—C13	115.7 (5)	H30A—C30—H30C	109.5
O7—C14—H14	108.6 (3)	H30B—C30—H30C	109.5
C1—C14—H14	108.6 (3)	C15—C31—H31A	109.5 (4)
C13—C14—H14	108.6 (4)	C15—C31—H31B	109.5 (4)
C1—C15—C31	110.7 (6)	H31A—C31—H31B	109.5
C1—C15—C30	110.0 (5)	C15—C31—H31C	109.5 (4)
C31—C15—C30	102.6 (6)	H31A—C31—H31C	109.5
C1—C15—C11	105.4 (5)	H31B—C31—H31C	109.5
C31—C15—C11	117.8 (6)	C16—O1—C2	116.1 (6)
C30—C15—C11	110.4 (6)	C19—O3—C5	121.4 (7)
O2—C16—O1	122.8 (8)	C22—O5—C10	117.3 (8)
O2—C16—C17	124.9 (8)	C25—O7—C14	116.1 (5)
C15—C1—C2—O1	−156.6 (5)	C24—C12—C13—C14	−146.6 (5)
C14—C1—C2—O1	75.9 (6)	C15—C1—C14—O7	98.2 (6)
C15—C1—C2—C3	80.3 (7)	C2—C1—C14—O7	−133.9 (5)
C14—C1—C2—C3	−47.2 (7)	C15—C1—C14—C13	−21.9 (8)
O1—C2—C3—C4	1.0 (8)	C2—C1—C14—C13	106.0 (6)
C1—C2—C3—C4	120.7 (6)	C12—C13—C14—O7	−142.6 (6)
O1—C2—C3—C8	131.8 (5)	C12—C13—C14—C1	−22.4 (8)
C1—C2—C3—C8	−108.5 (6)	C14—C1—C15—C31	−173.3 (5)
C2—C3—C4—C18	29.4 (11)	C2—C1—C15—C31	59.9 (8)
C8—C3—C4—C18	−103.3 (9)	C14—C1—C15—C30	−60.7 (7)
C2—C3—C4—C5	−160.8 (6)	C2—C1—C15—C30	172.5 (5)
C8—C3—C4—C5	66.5 (7)	C14—C1—C15—C11	58.3 (7)
C18—C4—C5—O3	−130.3 (8)	C2—C1—C15—C11	−68.5 (7)
C3—C4—C5—O3	58.5 (8)	C12—C11—C15—C1	−54.5 (7)
C18—C4—C5—C6	110.1 (8)	C10—C11—C15—C1	111.3 (7)
C3—C4—C5—C6	−61.1 (8)	C12—C11—C15—C31	−178.6 (6)
O3—C5—C6—C7	−70.7 (8)	C10—C11—C15—C31	−12.8 (9)
C4—C5—C6—C7	50.3 (9)	C12—C11—C15—C30	64.2 (7)
C5—C6—C7—C8	−48.3 (9)	C10—C11—C15—C30	−130.0 (7)
C6—C7—C8—C9	177.9 (6)	O8—C25—C26—C27	−31.8 (18)
C6—C7—C8—C21	−69.3 (8)	O7—C25—C26—C27	154.0 (12)
C6—C7—C8—C3	51.0 (7)	O8—C25—C26—C28	83.9 (14)
C4—C3—C8—C9	179.7 (6)	O7—C25—C26—C28	−90.3 (10)

C2—C3—C8—C9	43.8 (8)	C25—C26—C27—C29	−67.5 (15)
C4—C3—C8—C7	−59.0 (7)	C28—C26—C27—C29	176.1 (13)
C2—C3—C8—C7	165.1 (5)	O2—C16—O1—C2	2.2 (11)
C4—C3—C8—C21	61.5 (8)	C17—C16—O1—C2	−174.2 (7)
C2—C3—C8—C21	−74.5 (7)	C1—C2—O1—C16	88.8 (6)
C7—C8—C9—C10	−65.0 (8)	C3—C2—O1—C16	−145.5 (6)
C21—C8—C9—C10	177.8 (6)	O4—C19—O3—C5	8.0 (21)
C3—C8—C9—C10	55.9 (9)	C20—C19—O3—C5	−179.8 (10)
C8—C9—C10—O5	−180.0 (6)	C4—C5—O3—C19	121.6 (9)
C8—C9—C10—C11	−55.3 (10)	C6—C5—O3—C19	−117.9 (9)
O5—C10—C11—C12	−133.1 (7)	O6—C22—O5—C10	5.3 (15)
C9—C10—C11—C12	105.7 (8)	C23—C22—O5—C10	−176.3 (7)
O5—C10—C11—C15	61.6 (8)	C11—C10—O5—C22	83.5 (9)
C9—C10—C11—C15	−59.6 (9)	C9—C10—O5—C22	−149.8 (7)
C10—C11—C12—C24	20.5 (10)	O8—C25—O7—C14	−4.1 (12)
C15—C11—C12—C24	−173.7 (6)	C26—C25—O7—C14	170.3 (8)
C10—C11—C12—C13	−155.9 (6)	C1—C14—O7—C25	161.8 (6)
C15—C11—C12—C13	10.0 (9)	C13—C14—O7—C25	−72.9 (7)
C11—C12—C13—C14	30.2 (9)		