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3,7,7a-Tri-*epi*-casuarine pentaacetate

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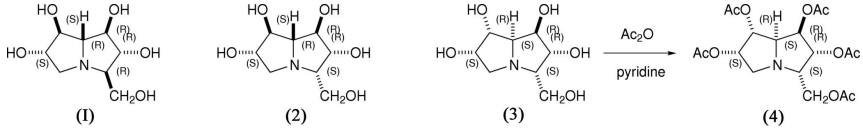
The relative stereochemistry at six contiguous centres in an analogue of the natural product casuarine, *viz*. 3,7,7a-tri-*epi*-casuarine pentaacetate, $C_{18}H_{25}NO_{10}$, has been established by an analysis of a crystalline pentaacetate.

Comment

The structure of casuarine, (1) (see scheme) (Nash *et al.*, 1994), also isolated as its 6-α-D-glucoside (Wormald *et al.*, 1996), has been determined by X-ray crystallography. The crystal structure of 3-*epi*-casuarine, (2), has also been reported (Newton *et al.*, 2004). Only two syntheses of casuarine have been published to date (Denmark & Hurd, 2000; Izquierdo *et al.*, 2005). Casuarine, with six contiguous stereogenic centres, is a potent α-glucosidase inhibitor and is the most heavily oxygenated of the polyhydroxylated alkaloids which can be viewed as sugar mimics (Asano *et al.*, 2000; Winchester & Fleet, 1992). Synthetic studies on the epimers of casuarine are scant, and none of the stereoisomers reported significantly inhibited any glycosidase (Bell et al., 1997). Nonetheless, some casuarine analogues have promise as vaccine adjuvants and as potential candidates for viral disease and non-cytotoxic cancer therapies (Nash *et al.*, 2004).



(I) (2) (3) (4)

As part of a structure–activity investigation of the stereoisomers of casuarine, the tri-*epi* casuarine (3) was prepared by a route which did not define the relative configuration at two centres. Although (3) has not been crystallized, peracetylation by acetic anhydride in pyridine gave the crystalline pentaacetate, (4), the crystal structure of which is reported in this paper (Fig. 1 and Table 1).

This study firmly establishes the relative configuration at all six stereogenic centres. The absolute configuration of (4) is determined by the use of D-glucose as the starting material in the synthesis. A combination of crystal structures and NMR studies have established solid-state and solution conformations of a number of stereoisomers of the less oxygenated alexines (Wormald *et al.*, 1998; Kato *et al.*, 2003) which may be used to rationalize their biological activity. Similar structural studies on the stereoisomers of casuarine may permit the development of rationales for their novel biological activities. The crystal packing, represented in Fig. 2, highlights long-range interactions between the acetate fragments that are both non-polar, *i.e.* between methyl groups, and polar, *i.e.* between O atoms.

Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.091
Data-to-parameter ratio = 11.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

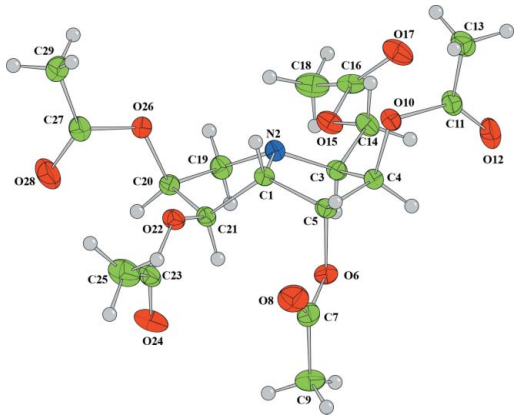


Figure 1
The molecular structure of (4), showing displacement ellipsoids drawn at the 50% probability level.

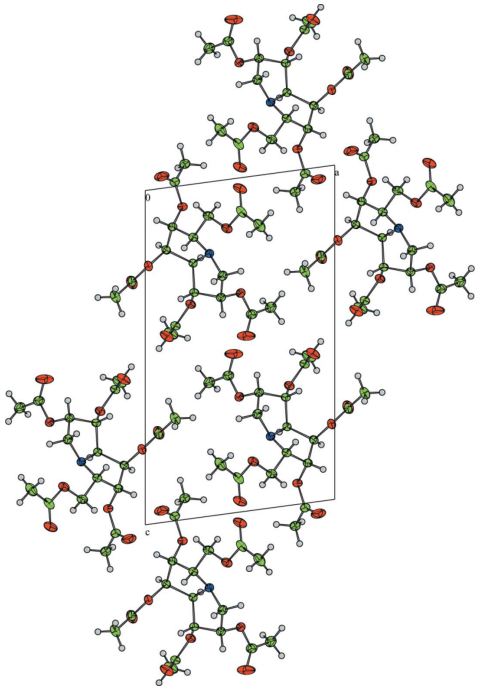


Figure 2
Packing diagram of (4), viewed down the *b* axis.

Experimental

Compound (4) was crystallized by dissolving it in cyclohexane, adding ethanol (in an approximate ratio of 9:1), and allowing slow competitive evaporation of the two solvents until clear colourless crystals formed.

Crystal data

$C_{18}H_{25}NO_{10}$
M_r = 415.40
Monoclinic, $P2_1$
a = 9.8357 (3) Å
b = 5.9443 (2) Å
c = 17.2146 (6) Å
β = 97.6513 (12)°
V = 997.51 (6) Å^3
Z = 2
D_x = 1.383 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2338 reflections
θ = 5–30°
μ = 0.11 mm^{-1}
T = 120 K
Needle, colourless
0.30 × 0.10 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω scans
Absorption correction: multi-scan (*DENZO*/*SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.99, T_{max} = 0.99
5106 measured reflections
3067 independent reflections
2513 reflections with $I > 2\sigma(I)$
R_{int} = 0.017
θ_{max} = 30.0°
$h = -13 \rightarrow 13$
$k = -8 \rightarrow 7$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.091
S = 0.94
3067 reflections
263 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.2P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max}$ <0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 1.8 (4) × 10^2

Table 1
Selected geometric parameters (Å, °).

C1—N2	1.486 (2)	C11—C13	1.491 (3)
C1—C5	1.540 (3)	C14—O15	1.448 (2)
C1—C21	1.517 (3)	O15—C16	1.351 (2)
N2—C3	1.466 (2)	C16—O17	1.208 (3)
N2—C19	1.477 (2)	C16—C18	1.483 (3)
C3—C4	1.528 (3)	C19—C20	1.513 (3)
C3—C14	1.508 (3)	C20—C21	1.523 (3)
C4—C5	1.525 (3)	C20—O26	1.456 (2)
C4—O10	1.448 (2)	C21—O22	1.432 (2)
C5—O6	1.453 (2)	O22—C23	1.363 (2)
O6—C7	1.357 (3)	C23—O24	1.197 (3)
C7—O8	1.197 (3)	C23—C25	1.490 (3)
C7—C9	1.493 (4)	O26—C27	1.355 (2)
O10—C11	1.355 (2)	C27—O28	1.193 (3)
C11—O12	1.200 (3)	C27—C29	1.482 (3)
N2—C1—C5	106.11 (15)	O12—C11—C13	126.35 (19)
N2—C1—C21	104.58 (15)	C3—C14—O15	107.24 (15)
C5—C1—C21	118.30 (16)	C14—O15—C16	114.62 (16)
C1—N2—C3	108.97 (14)	O15—C16—O17	122.2 (2)
C1—N2—C19	108.76 (15)	O15—C16—C18	112.17 (18)
C3—N2—C19	116.67 (16)	O17—C16—C18	125.6 (2)
N2—C3—C4	103.27 (16)	N2—C19—C20	105.06 (17)
N2—C3—C14	113.92 (16)	C19—C20—C21	101.52 (15)
C4—C3—C14	112.75 (15)	C19—C20—O26	108.48 (16)
C3—C4—C5	103.33 (15)	C21—C20—O26	109.03 (16)
C3—C4—O10	111.18 (15)	C20—C21—C1	103.69 (16)
C5—C4—O10	106.33 (16)	C20—C21—O22	114.18 (16)
C1—C5—C4	103.08 (15)	C1—C21—O22	110.11 (16)
C1—C5—O6	111.65 (15)	C21—O22—C23	117.04 (16)
C4—C5—O6	104.60 (16)	O22—C23—O24	122.9 (2)
C5—O6—C7	116.67 (17)	O22—C23—C25	110.27 (19)
O6—C7—O8	123.5 (2)	O24—C23—C25	126.8 (2)
O6—C7—C9	110.9 (2)	C20—O26—C27	117.07 (16)
O8—C7—C9	125.6 (2)	O26—C27—O28	122.8 (2)
C4—O10—C11	116.98 (16)	O26—C27—C29	112.16 (18)
O10—C11—O12	122.9 (2)	O28—C27—C29	125.1 (2)
O10—C11—C13	110.71 (19)		

In the absence of significant anomalous scattering effects, Friedel pairs were merged, and the absolute configuration was assigned from the known configuration of the starting material. H atoms were seen in a difference density synthesis. Those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bond lengths and angles to regularize their geometry, after which they were included with riding constraints, with C—H = 0.93–0.98 Å and with U_{iso}(H) values in the range 1.2–1.5U_{eq} of the carrier atom.

Data collection: *COLLECT* (Nonius, 2001); cell refinement and data reduction: *DENZO*/*SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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$h = -13 \rightarrow 13$
$k = -8 \rightarrow 7$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.091$
$S = 0.94$
3067 reflections
263 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F^2) + (0.04P)^2 + 0.2P]$, where $P = [\max(F_o^2,0) + 2F_c^2]/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}
Extinction correction: Larson (1970), equation 22
Extinction coefficient: 180 (40)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.25143 (19)	0.1388 (3)	0.23642 (10)	0.0187

N2	0.33807 (16)	0.3266 (3)	0.21412 (9)	0.0193
C3	0.2539 (2)	0.4752 (3)	0.15921 (11)	0.0201
C4	0.1427 (2)	0.3181 (4)	0.11951 (11)	0.0219
C5	0.1107 (2)	0.1667 (4)	0.18624 (11)	0.0212
O6	0.01706 (14)	0.2978 (3)	0.22668 (8)	0.0255
C7	−0.0695 (2)	0.1806 (5)	0.26674 (13)	0.0297
O8	−0.07114 (18)	−0.0205 (3)	0.27014 (10)	0.0399
C9	−0.1587 (2)	0.3359 (5)	0.30573 (15)	0.0419
O10	0.19617 (14)	0.1727 (3)	0.06328 (7)	0.0237
C11	0.1557 (2)	0.2161 (4)	−0.01361 (12)	0.0286
O12	0.0866 (2)	0.3756 (4)	−0.03595 (9)	0.0480
C13	0.2093 (2)	0.0416 (4)	−0.06385 (12)	0.0333
C14	0.3328 (2)	0.5918 (4)	0.10152 (12)	0.0252
O15	0.42996 (15)	0.7417 (3)	0.14594 (8)	0.0262
C16	0.5080 (2)	0.8625 (4)	0.10221 (13)	0.0279
O17	0.49969 (18)	0.8407 (3)	0.03195 (9)	0.0368
C18	0.6021 (3)	1.0182 (4)	0.15052 (15)	0.0370
C19	0.4086 (2)	0.4316 (4)	0.28622 (11)	0.0240
C20	0.3997 (2)	0.2597 (3)	0.35015 (11)	0.0217
C21	0.2588 (2)	0.1574 (3)	0.32480 (11)	0.0197
O22	0.23933 (14)	−0.0592 (2)	0.35803 (8)	0.0226
C23	0.1593 (2)	−0.0686 (4)	0.41678 (12)	0.0253
O24	0.11413 (18)	0.0956 (3)	0.44445 (10)	0.0387
C25	0.1360 (3)	−0.3071 (4)	0.43825 (15)	0.0405
O26	0.50414 (14)	0.0887 (3)	0.34492 (8)	0.0244
C27	0.5616 (2)	−0.0068 (4)	0.41296 (12)	0.0293
O28	0.5325 (2)	0.0480 (4)	0.47537 (9)	0.0593
C29	0.6623 (2)	−0.1838 (4)	0.39999 (13)	0.0326
H11	0.2925	−0.0032	0.2234	0.0223*
H31	0.2060	0.5904	0.1875	0.0248*
H41	0.0609	0.4045	0.0941	0.0275*
H51	0.0706	0.0200	0.1689	0.0255*
H91	−0.2507	0.2740	0.3010	0.0636*
H92	−0.1571	0.4835	0.2819	0.0633*
H93	−0.1231	0.3461	0.3599	0.0636*
H131	0.1851	0.0841	−0.1188	0.0471*
H132	0.1720	−0.1038	−0.0543	0.0467*
H133	0.3078	0.0384	−0.0515	0.0471*
H141	0.2686	0.6793	0.0640	0.0318*
H142	0.3809	0.4833	0.0725	0.0319*
H181	0.6624	1.0880	0.1172	0.0567*
H182	0.5493	1.1306	0.1729	0.0568*
H183	0.6548	0.9355	0.1916	0.0567*
H191	0.3629	0.5702	0.2997	0.0295*
H192	0.5052	0.4634	0.2795	0.0288*
H201	0.4091	0.3228	0.4030	0.0262*
H211	0.1877	0.2619	0.3382	0.0231*
H251	0.0736	−0.3105	0.4783	0.0641*

H252	0.0947	−0.3875	0.3925	0.0643*
H253	0.2224	−0.3763	0.4583	0.0642*
H291	0.7195	−0.2230	0.4466	0.0486*
H292	0.6200	−0.3170	0.3772	0.0491*
H293	0.7217	−0.1292	0.3640	0.0489*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0188 (9)	0.0192 (9)	0.0185 (9)	−0.0001 (7)	0.0039 (7)	−0.0001 (7)
N2	0.0206 (8)	0.0185 (8)	0.0186 (7)	−0.0027 (7)	0.0019 (6)	−0.0008 (7)
C3	0.0225 (10)	0.0193 (9)	0.0190 (9)	0.0011 (8)	0.0046 (8)	0.0002 (8)
C4	0.0223 (10)	0.0254 (10)	0.0179 (9)	0.0034 (9)	0.0027 (7)	0.0003 (8)
C5	0.0206 (9)	0.0238 (9)	0.0200 (9)	−0.0014 (8)	0.0050 (7)	−0.0014 (8)
O6	0.0186 (7)	0.0320 (8)	0.0268 (7)	0.0007 (6)	0.0061 (6)	0.0013 (6)
C7	0.0168 (10)	0.0447 (14)	0.0277 (11)	−0.0037 (10)	0.0028 (8)	0.0057 (11)
O8	0.0318 (9)	0.0436 (10)	0.0464 (10)	−0.0063 (8)	0.0132 (8)	0.0078 (9)
C9	0.0269 (12)	0.0608 (18)	0.0406 (13)	0.0054 (13)	0.0141 (10)	0.0000 (14)
O10	0.0255 (7)	0.0282 (7)	0.0175 (6)	0.0002 (6)	0.0028 (5)	−0.0013 (6)
C11	0.0257 (11)	0.0404 (13)	0.0190 (10)	−0.0031 (10)	0.0007 (8)	0.0007 (9)
O12	0.0587 (12)	0.0609 (13)	0.0228 (8)	0.0217 (11)	−0.0005 (8)	0.0080 (9)
C13	0.0285 (11)	0.0487 (15)	0.0228 (10)	−0.0068 (11)	0.0028 (9)	−0.0075 (11)
C14	0.0314 (11)	0.0233 (10)	0.0219 (10)	−0.0036 (9)	0.0072 (8)	0.0001 (9)
O15	0.0310 (8)	0.0242 (7)	0.0251 (7)	−0.0069 (6)	0.0099 (6)	−0.0022 (6)
C16	0.0286 (11)	0.0220 (10)	0.0365 (12)	0.0012 (9)	0.0169 (9)	0.0012 (9)
O17	0.0489 (10)	0.0343 (9)	0.0309 (8)	−0.0048 (8)	0.0194 (7)	0.0041 (8)
C18	0.0356 (13)	0.0320 (12)	0.0469 (14)	−0.0081 (11)	0.0185 (11)	−0.0050 (11)
C19	0.0242 (10)	0.0239 (10)	0.0234 (10)	−0.0037 (8)	0.0015 (8)	−0.0019 (8)
C20	0.0230 (10)	0.0223 (9)	0.0196 (9)	0.0028 (8)	0.0022 (8)	−0.0021 (8)
C21	0.0230 (9)	0.0193 (9)	0.0170 (9)	0.0023 (8)	0.0038 (7)	0.0011 (8)
O22	0.0265 (8)	0.0212 (7)	0.0214 (7)	0.0015 (6)	0.0079 (6)	0.0033 (6)
C23	0.0253 (10)	0.0322 (12)	0.0193 (9)	−0.0023 (9)	0.0066 (8)	0.0013 (9)
O24	0.0484 (10)	0.0351 (9)	0.0377 (9)	−0.0026 (9)	0.0239 (8)	−0.0063 (8)
C25	0.0505 (16)	0.0342 (13)	0.0408 (14)	−0.0034 (12)	0.0212 (12)	0.0090 (11)
O26	0.0220 (7)	0.0304 (8)	0.0203 (7)	0.0037 (7)	0.0011 (6)	−0.0005 (6)
C27	0.0324 (12)	0.0323 (11)	0.0229 (10)	0.0036 (10)	0.0030 (9)	0.0032 (9)
O28	0.0895 (16)	0.0656 (15)	0.0238 (9)	0.0433 (13)	0.0116 (9)	0.0103 (9)
C29	0.0326 (12)	0.0351 (12)	0.0292 (11)	0.0086 (11)	0.0001 (9)	0.0005 (10)

Geometric parameters (Å, º)

C1—N2	1.486 (2)	C14—H142	0.975
C1—C5	1.540 (3)	O15—C16	1.351 (2)
C1—C21	1.517 (3)	C16—O17	1.208 (3)
C1—H11	0.974	C16—C18	1.483 (3)
N2—C3	1.466 (2)	C18—H181	0.972
N2—C19	1.477 (2)	C18—H182	0.958
C3—C4	1.528 (3)	C18—H183	0.956

C3—C14	1.508 (3)	C19—C20	1.513 (3)
C3—H31	0.994	C19—H191	0.981
C4—C5	1.525 (3)	C19—H192	0.991
C4—O10	1.448 (2)	C20—C21	1.523 (3)
C4—H41	1.004	C20—O26	1.456 (2)
C5—O6	1.453 (2)	C20—H201	0.976
C5—H51	0.987	C21—O22	1.432 (2)
O6—C7	1.357 (3)	C21—H211	0.986
C7—O8	1.197 (3)	O22—C23	1.363 (2)
C7—C9	1.493 (4)	C23—O24	1.197 (3)
C9—H91	0.970	C23—C25	1.490 (3)
C9—H92	0.969	C25—H251	0.982
C9—H93	0.954	C25—H252	0.964
O10—C11	1.355 (2)	C25—H253	0.966
C11—O12	1.200 (3)	O26—C27	1.355 (2)
C11—C13	1.491 (3)	C27—O28	1.193 (3)
C13—H131	0.977	C27—C29	1.482 (3)
C13—H132	0.962	C29—H291	0.946
C13—H133	0.964	C29—H292	0.954
C14—O15	1.448 (2)	C29—H293	0.963
C14—H141	0.989		

N2—C1—C5	106.11 (15)	O15—C14—H142	110.3
N2—C1—C21	104.58 (15)	H141—C14—H142	109.0
C5—C1—C21	118.30 (16)	C14—O15—C16	114.62 (16)
N2—C1—H11	108.8	O15—C16—O17	122.2 (2)
C5—C1—H11	109.5	O15—C16—C18	112.17 (18)
C21—C1—H11	109.1	O17—C16—C18	125.6 (2)
C1—N2—C3	108.97 (14)	C16—C18—H181	108.6
C1—N2—C19	108.76 (15)	C16—C18—H182	109.2
C3—N2—C19	116.67 (16)	H181—C18—H182	110.2
N2—C3—C4	103.27 (16)	C16—C18—H183	109.3
N2—C3—C14	113.92 (16)	H181—C18—H183	110.1
C4—C3—C14	112.75 (15)	H182—C18—H183	109.3
N2—C3—H31	111.3	N2—C19—C20	105.06 (17)
C4—C3—H31	106.4	N2—C19—H191	112.1
C14—C3—H31	108.9	C20—C19—H191	108.8
C3—C4—C5	103.33 (15)	N2—C19—H192	109.5
C3—C4—O10	111.18 (15)	C20—C19—H192	111.2
C5—C4—O10	106.33 (16)	H191—C19—H192	110.1
C3—C4—H41	111.5	C19—C20—C21	101.52 (15)
C5—C4—H41	113.3	C19—C20—O26	108.48 (16)
O10—C4—H41	110.8	C21—C20—O26	109.03 (16)
C1—C5—C4	103.08 (15)	C19—C20—H201	114.2
C1—C5—O6	111.65 (15)	C21—C20—H201	113.0
C4—C5—O6	104.60 (16)	O26—C20—H201	110.2
C1—C5—H51	111.6	C20—C21—C1	103.69 (16)
C4—C5—H51	114.1	C20—C21—O22	114.18 (16)

O6—C5—H51	111.4	C1—C21—O22	110.11 (16)
C5—O6—C7	116.67 (17)	C20—C21—H211	109.2
O6—C7—O8	123.5 (2)	C1—C21—H211	109.9
O6—C7—C9	110.9 (2)	O22—C21—H211	109.6
O8—C7—C9	125.6 (2)	C21—O22—C23	117.04 (16)
C7—C9—H91	109.0	O22—C23—O24	122.9 (2)
C7—C9—H92	109.0	O22—C23—C25	110.27 (19)
H91—C9—H92	112.0	O24—C23—C25	126.8 (2)
C7—C9—H93	108.5	C23—C25—H251	109.0
H91—C9—H93	108.8	C23—C25—H252	109.3
H92—C9—H93	109.4	H251—C25—H252	109.2
C4—O10—C11	116.98 (16)	C23—C25—H253	109.7
O10—C11—O12	122.9 (2)	H251—C25—H253	110.1
O10—C11—C13	110.71 (19)	H252—C25—H253	109.6
O12—C11—C13	126.35 (19)	C20—O26—C27	117.07 (16)
C11—C13—H131	108.8	O26—C27—O28	122.8 (2)
C11—C13—H132	110.7	O26—C27—C29	112.16 (18)
H131—C13—H132	110.5	O28—C27—C29	125.1 (2)
C11—C13—H133	107.9	C27—C29—H291	112.4
H131—C13—H133	109.0	C27—C29—H292	112.8
H132—C13—H133	109.8	H291—C29—H292	108.8
C3—C14—O15	107.24 (15)	C27—C29—H293	109.4
C3—C14—H141	109.4	H291—C29—H293	106.6
O15—C14—H141	109.9	H292—C29—H293	106.6
C3—C14—H142	111.1		