Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(1*R*,4*S*,8*R*,12*S*,13*S*,14*R*,16*S*,17*R*,19*R*)-14-Hydroxy-7,7,17-trimethyl-2,9,18-trioxo-3,10-dioxapentacyclo[14.2.1.0^{1,13}.0^{4,12}.0^{8,12}]nonadec-19-yl acetate

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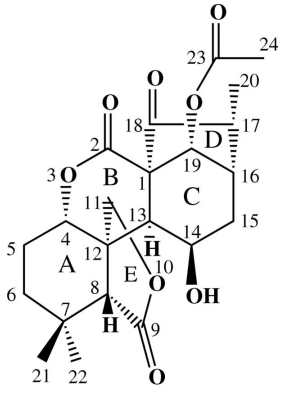
Received 18 October 2007; accepted 25 October 2007

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.038; wR factor = 0.095; data-to-parameter ratio = 7.5.

The title compound, $C_{22}H_{28}O_8$, was prepared from the natural diterpenoid macrocalyxin J by two steps. It is built up from five fused rings: three six-membered rings and two five-membered rings. Two molecules are present in the asymmetric unit; both independent molecules have the same absolute configuration, and the absolute configuration was deduced from the chirality of macrocalyxin A, which was isolated from the same plant (*i.e. Rabdosia macrocalyx*) as macrocalyxin J. The two molecules are linked by O—H···O hydrogen bonds, building a pseudo-dimer. Further O—H···O hydrogen bonds link these dimers to form a chain parallel to the *a* axis.

Related literature

For related literature, see: Cremer & Pople (1975); Shi *et al.* (2003, 2007); Spek (2003).



Experimental

Crystal data

$C_{22}H_{28}O_8$
M_r = 420.44
Orthorhombic, $P2_12_12_1$
a = 11.9500 (11) Å
b = 13.7531 (16) Å
c = 25.377 (3) Å
V = 4170.7 (8) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.50 × 0.46 × 0.27 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
T_{min} = 0.951, T_{max} = 0.973
20027 measured reflections
4122 independent reflections
2948 reflections with $I > 2\sigma(I)$
R_{int} = 0.051

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.095
S = 1.07
4122 reflections
550 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···O2A	0.82	2.27	2.980 (4)	145
O4A—H4A···O1^{i}	0.82	1.91	2.678 (4)	156

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

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: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

This project was supported by the Science Foundation of Zhejiang Province, China (grant No. Y205318).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2250).

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supplementary materials

Acta Cryst. (2007). E**63**, o4495 [**doi:10.1107/S1600536807053317**]

(1*R*,4*S*,8*R*,12*S*,13*S*,14*R*,16*S*,17*R*,19*R*)-14-Hydroxy-7,7,17-trimethyl-2,9,18-trioxo-3,10-dioxapentacyclo[14.2.1.$0^{1,13}$.$0^{4,12}$.$0^{8,12}$]nonadec-19-yl acetate

H. Shi

Comment

Since the natural diterpenoid Macrocalyxin J exhibits cytotoxicity *in vitro* against cultures of Hela cells (Shi *et al.*, 2007), we have derived the title compound from it.

Two unique molecules are present in the asymmetric unit, both molecule I (Fig.1) and molecule II (Fig.2) are built up from five fused rings, three six membered (A,B,*C*,A',B'and C') and two five membered rings (D,*E*,D' and E'). This two molecules are linked by a O—H···O hydrogen bond to form a pseudo dimer. The dimer are further interconnected through O—H···O hydrogen bonds to build up a chain parallel to the *a* axis (Table 1, Fig.3).

The conformations of the different rings extracted from the puckering parameters (Cremer & Pople, 1975) are given in Table 2. Ring A and A' adopt a chair conformation, ring B and B' exist in a screw-boat conformation and rings C and C' adopt a boat conformation. Rings D and D' present a twist conformation whereas rings E and E' adopt an envelope conformation.

Since the title compound was prepared from Macrocalyxin J, which was isolated from the same plant (*i.e. Rabdosia macrocalyx*) as Macrocalyxin A, the configuration can be deduced from the known chirality of the Macrocalyxin A (Shi *et al.*, 2003),and thus Fig. 1 and Fig. 2 represents the correct absolute configuration.

Experimental

Macrocalyxin J (200 mg; isolated from *Rabdosia macrocalyx*) was reacted with $NaBH_4$ in 2-propanol and compound (II) was gained, ^{1}H NMR (C_5D_5N, 400 MHz): 0.94,0.97(2× 3H,s), 1.24(3*H*,d, J=7 Hz),2.76(1*H*,s), 2.09(3*H*,s), 3.04(1*H*,d,J=4 Hz), 2.24(1*H*,m,13-H), 4.35,4.52(2×1H,d,AB, J=9 Hz), 5.99(1*H*,m), 3.09(H,q, J=7 Hz), 5.14(1*H*,m),5.78(1*H*,s), ^{13}C NMR (C_5D_5N, 100 MHz): 11.1 (CH_3), 21.3 (CH_3), 23.5(CH_3), 24.2(CH_2), 31.7(C), 31.7(CH_2), 33.3(CH_3), 37.4(CH_2), 38.3(CH), 44.9(CH), 50.1(C), 52.3(CH), 55.8(CH), 61.5(C), 65.2(CH), 74.4(CH_2), 75.3(CH), 78.5(CH), 102.0(CH), 168.2(C), 170.5(C), 213.7(C). The compound (II) was then reacted with Jones reagent in acetone, compound (I) was gained as a white residue. Recrystallization with methanol gave the title compound (I) as colorless crystals, ^{1}H NMR ($CDCl_3$, 400 MHz): 1.06,1.22(2×3H,s,2× Me), 1.15(3*H*,d, J=7 Hz), 2.69(1*H*,s,5-H), 2.06(3*H*,s, OAc), 3.05(H,q, J=7 Hz), 3.99,4.33(2×1H,d, J=10 Hz), 4.19(1*H*,brs), 6.71(1*H*,d, J=2 Hz, 14-H), 5.23(1*H*,dd,J=6,12 Hz).

Crystals suitable for X-ray structure analysis were obtained by slow evaporation from a solution of methanol at room temperature.

Refinement

H atoms were placed in calculated positions and treated as riding on their parent atoms, with C—H = 0.96 Å (CH_3), 0.97 Å (CH_2) and 0.98(CH) and with the temperature factors U_{iso}= 1.5 U_{eq}(CH_3) and 1.2 U_{eq}(CH_2, CH).

In the absence of significant anomalous scattering, the absolute configuration could not be reliably determined from the X-ray analyses and then the Friedel pairs were merged and any references to the Flack parameter were removed.

Figures

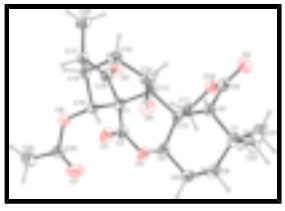


Fig. 1. Molecular view of molecule I with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

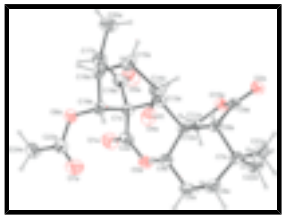


Fig. 2. Molecular view of molecule II with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

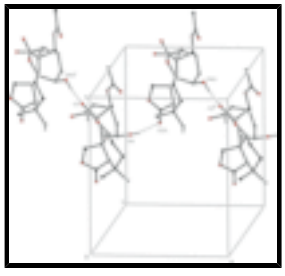


Fig. 3. Partial packing view of the title compound showing the formation of a chain parallel through O—H···O hydrogen bonding interactions. H atoms not involved in hydrogen bonds have been omitted for clarity·[Symmetry codes: (i) *x* − 1, *y*, *z*; (ii) 1.*x*, *y*, *z*].

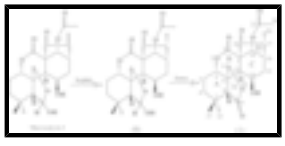


Fig. 4. The formation of the title compound.

(1*R*,4*S*,8*R*,12*S*,13*S*,14*R*,16*S*, 17*R*,19*R*)-14-Hydroxy-7,7,17-trimethyl-2,9,18-trioxo-3,10-dioxapentacyclo[14.2.1.$0^{1,13}$.$0^{4,12}$.$0^{8,12}$]nonadec-19-yl acetate

Crystal data

$C_{22}H_{28}O_8$	F_{000} = 1792
M_r = 420.44	D_x = 1.339 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 5045 reflections
a = 11.9500 (11) Å	θ = 2.2–21.9º

b = 13.7531 (16) Å	μ = 0.10 mm^{-1}
c = 25.377 (3) Å	T = 293 (2) K
V = 4170.7 (8) Å^3	Plate, colorless
Z = 8	0.50 × 0.46 × 0.27 mm

Data collection

Bruker SMART CCD area-detector diffractometer	4122 independent reflections
Radiation source: fine-focus sealed tube	2948 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.051
T = 293(2) K	θ_{max} = 25.0º
φ and ω scans	θ_{min} = 1.6º
Absorption correction: multi-scan (SADABS; Bruker, 1999)	h = −14→13
T_{min} = 0.951, T_{max} = 0.973	k = −16→6
20027 measured reflections	l = −29→30

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0342P)^2 + 1.1127P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.038	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.095	$\Delta\rho_{max}$ = 0.19 e Å^{-3}
S = 1.07	$\Delta\rho_{min}$ = −0.17 e Å^{-3}
4122 reflections	Extinction correction: none
550 parameters	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 $F^2 > 2$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	−0.0926 (2)	0.9061 (2)	0.15436 (10)	0.0632 (8)
O2	0.0368 (3)	0.4543 (2)	0.03746 (12)	0.0792 (10)
O3	0.0107 (2)	0.78491 (18)	0.18023 (9)	0.0483 (6)
O4	0.27213 (19)	0.74065 (19)	0.08031 (9)	0.0495 (6)
H4	0.3334	0.7412	0.0658	0.074*
O5	−0.1385 (2)	0.8632 (2)	0.04103 (12)	0.0623 (8)
O6	0.1031 (2)	1.00999 (17)	0.10644 (9)	0.0482 (6)
O7	0.1783 (3)	0.9770 (3)	0.18505 (11)	0.0803 (10)
O10	−0.0762 (2)	0.5583 (2)	0.07852 (10)	0.0585 (7)
O1A	0.4834 (3)	1.3128 (2)	0.15642 (12)	0.0773 (9)
O2A	0.4943 (2)	0.8266 (2)	0.05435 (10)	0.0567 (7)
O3A	0.5169 (2)	1.1686 (2)	0.18999 (10)	0.0598 (8)
O4A	0.7978 (2)	1.06592 (19)	0.12314 (11)	0.0609 (7)
H4A	0.8319	1.0142	0.1233	0.091*
O5A	0.4647 (3)	1.2672 (3)	0.04184 (13)	0.0871 (11)
O6A	0.7140 (2)	1.35784 (18)	0.12540 (10)	0.0546 (7)
O7A	0.7396 (3)	1.3172 (3)	0.20922 (13)	0.0962 (12)
O10A	0.4091 (2)	0.9613 (2)	0.08057 (9)	0.0508 (7)
C1	0.0354 (3)	0.8444 (2)	0.09003 (12)	0.0372 (8)
C2	−0.0205 (3)	0.8474 (3)	0.14339 (14)	0.0434 (9)
C4	0.0956 (3)	0.7135 (2)	0.16609 (13)	0.0399 (8)
H4B	0.1670	0.7468	0.1602	0.048*
C5	0.1073 (4)	0.6453 (3)	0.21185 (14)	0.0541 (10)
H5A	0.0350	0.6180	0.2209	0.065*
H5B	0.1360	0.6798	0.2423	0.065*
C6	0.1878 (4)	0.5645 (3)	0.19610 (15)	0.0587 (11)
H6A	0.2028	0.5241	0.2266	0.070*
H6B	0.2581	0.5933	0.1850	0.070*
C7	0.1427 (3)	0.5001 (3)	0.15139 (15)	0.0523 (10)
C8	0.1136 (3)	0.5667 (2)	0.10297 (13)	0.0416 (9)
H8	0.1816	0.5776	0.0822	0.050*
C9	0.0268 (4)	0.5191 (3)	0.06858 (15)	0.0547 (10)
C11	−0.0660 (3)	0.6361 (3)	0.11584 (15)	0.0470 (9)
H11A	−0.0876	0.6145	0.1508	0.056*
H11B	−0.1132	0.6904	0.1058	0.056*
C12	0.0580 (3)	0.6658 (2)	0.11490 (13)	0.0359 (8)
C13	0.0721 (3)	0.7405 (2)	0.06980 (12)	0.0339 (8)
H13	0.0169	0.7222	0.0430	0.041*
C14	0.1855 (3)	0.7421 (3)	0.04123 (13)	0.0416 (9)
H14	0.1917	0.6830	0.0199	0.050*
C15	0.1962 (3)	0.8300 (3)	0.00484 (13)	0.0468 (9)
H15A	0.1584	0.8154	−0.0280	0.056*
H15B	0.2748	0.8398	−0.0031	0.056*
C16	0.1483 (3)	0.9253 (3)	0.02698 (13)	0.0422 (9)
H16	0.1976	0.9800	0.0184	0.051*

C17	0.0278 (3)	0.9473 (3)	0.00972 (13)	0.0456 (9)
H17	0.0131	1.0147	0.0201	0.055*
C18	−0.0415 (3)	0.8848 (3)	0.04604 (14)	0.0443 (9)
C19	0.1352 (3)	0.9156 (2)	0.08652 (13)	0.0386 (8)
H19	0.2028	0.8902	0.1036	0.046*
C20	−0.0037 (4)	0.9399 (3)	−0.04832 (14)	0.0694 (13)
H20A	0.0026	0.8735	−0.0596	0.104*
H20B	0.0459	0.9798	−0.0688	0.104*
H20C	−0.0793	0.9617	−0.0531	0.104*
C21	0.2364 (4)	0.4304 (3)	0.13425 (17)	0.0732 (13)
H21A	0.3021	0.4671	0.1254	0.110*
H21B	0.2123	0.3939	0.1041	0.110*
H21C	0.2533	0.3865	0.1626	0.110*
C22	0.0444 (4)	0.4386 (3)	0.17108 (18)	0.0718 (13)
H22A	0.0685	0.3990	0.2001	0.108*
H22B	0.0180	0.3978	0.1430	0.108*
H22C	−0.0150	0.4807	0.1825	0.108*
C23	0.1316 (4)	1.0317 (3)	0.15682 (15)	0.0542 (11)
C24	0.0941 (5)	1.1328 (3)	0.17004 (17)	0.0873 (17)
H24A	0.1209	1.1501	0.2044	0.131*
H24B	0.0138	1.1356	0.1697	0.131*
H24C	0.1234	1.1775	0.1445	0.131*
C1A	0.6037 (3)	1.2117 (3)	0.10504 (13)	0.0409 (8)
C2A	0.5294 (3)	1.2360 (3)	0.15192 (15)	0.0523 (10)
C4A	0.5786 (3)	1.0783 (3)	0.18451 (13)	0.0487 (10)
H4C	0.6589	1.0925	0.1865	0.058*
C5A	0.5476 (4)	1.0122 (3)	0.22930 (13)	0.0652 (13)
H5C	0.4672	1.0032	0.2302	0.078*
H5D	0.5706	1.0411	0.2625	0.078*
C6A	0.6053 (4)	0.9150 (3)	0.22193 (14)	0.0670 (13)
H6C	0.5931	0.8754	0.2531	0.080*
H6D	0.6852	0.9255	0.2184	0.080*
C7A	0.5632 (3)	0.8600 (3)	0.17376 (15)	0.0521 (10)
C8A	0.5778 (3)	0.9269 (3)	0.12381 (12)	0.0397 (8)
H8A	0.6536	0.9189	0.1098	0.048*
C9A	0.4942 (3)	0.8973 (3)	0.08245 (14)	0.0437 (9)
C11A	0.4270 (3)	1.0396 (3)	0.11786 (14)	0.0493 (10)
H11C	0.3830	1.0295	0.1495	0.059*
H11D	0.4066	1.1017	0.1025	0.059*
C12A	0.5527 (3)	1.0366 (3)	0.13045 (12)	0.0386 (8)
C13A	0.6129 (3)	1.1009 (2)	0.08914 (12)	0.0359 (8)
H13A	0.5704	1.0937	0.0563	0.043*
C14A	0.7340 (3)	1.0734 (3)	0.07577 (14)	0.0461 (9)
H14A	0.7339	1.0099	0.0582	0.055*
C15A	0.7862 (4)	1.1474 (3)	0.03890 (16)	0.0569 (11)
H15C	0.7593	1.1352	0.0034	0.068*
H15D	0.8666	1.1380	0.0388	0.068*
C16A	0.7611 (3)	1.2542 (3)	0.05332 (15)	0.0524 (10)
H16A	0.8280	1.2945	0.0487	0.063*

C17A	0.6619 (4)	1.2986 (3)	0.02463 (15)	0.0620 (12)
H17A	0.6661	1.3689	0.0304	0.074*
C18A	0.5612 (4)	1.2629 (3)	0.05495 (15)	0.0545 (11)
C19A	0.7221 (3)	1.2568 (3)	0.11028 (14)	0.0445 (9)
H19A	0.7708	1.2193	0.1338	0.053*
C20A	0.6506 (5)	1.2834 (4)	−0.03485 (17)	0.0914 (18)
H20D	0.6298	1.2172	−0.0418	0.137*
H20E	0.7208	1.2973	−0.0516	0.137*
H20F	0.5941	1.3262	−0.0485	0.137*
C21A	0.6354 (4)	0.7688 (3)	0.16527 (19)	0.0782 (14)
H21D	0.7128	0.7872	0.1631	0.117*
H21E	0.6135	0.7374	0.1331	0.117*
H21F	0.6251	0.7249	0.1943	0.117*
C22A	0.4420 (4)	0.8259 (4)	0.18302 (16)	0.0721 (14)
H22D	0.4378	0.7910	0.2157	0.108*
H22E	0.4194	0.7840	0.1547	0.108*
H22F	0.3933	0.8813	0.1844	0.108*
C23A	0.7245 (4)	1.3779 (3)	0.17677 (17)	0.0605 (11)
C24A	0.7089 (4)	1.4837 (3)	0.18666 (17)	0.0733 (14)
H24D	0.6305	1.4983	0.1881	0.110*
H24E	0.7430	1.5202	0.1587	0.110*
H24F	0.7433	1.5008	0.2196	0.110*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0655 (18)	0.0564 (17)	0.0676 (17)	0.0286 (16)	0.0207 (15)	0.0124 (15)
O2	0.112 (3)	0.0530 (18)	0.0722 (19)	0.0032 (19)	−0.0168 (19)	−0.0168 (17)
O3	0.0573 (16)	0.0443 (14)	0.0434 (13)	0.0148 (13)	0.0081 (12)	0.0071 (13)
O4	0.0304 (12)	0.0618 (17)	0.0562 (14)	0.0032 (13)	−0.0017 (11)	0.0044 (14)
O5	0.0391 (16)	0.0651 (19)	0.083 (2)	0.0028 (14)	−0.0143 (14)	0.0105 (16)
O6	0.0636 (17)	0.0387 (15)	0.0424 (14)	0.0013 (13)	−0.0037 (13)	−0.0006 (12)
O7	0.095 (2)	0.096 (2)	0.0492 (17)	0.013 (2)	−0.0199 (17)	−0.0014 (18)
O10	0.0553 (18)	0.0510 (17)	0.0692 (17)	−0.0096 (15)	−0.0172 (15)	0.0004 (15)
O1A	0.076 (2)	0.073 (2)	0.083 (2)	0.0269 (19)	0.0140 (17)	−0.0199 (18)
O2A	0.0623 (18)	0.0582 (17)	0.0494 (15)	−0.0120 (15)	0.0006 (14)	−0.0097 (15)
O3A	0.0631 (18)	0.0687 (19)	0.0477 (15)	−0.0078 (16)	0.0164 (14)	−0.0128 (15)
O4A	0.0511 (16)	0.0486 (16)	0.0830 (19)	0.0128 (14)	−0.0104 (15)	0.0132 (16)
O5A	0.079 (2)	0.097 (3)	0.086 (2)	0.037 (2)	−0.0359 (19)	0.005 (2)
O6A	0.0729 (19)	0.0415 (15)	0.0494 (16)	0.0006 (14)	−0.0010 (14)	−0.0018 (13)
O7A	0.140 (3)	0.087 (3)	0.0612 (19)	−0.003 (2)	−0.020 (2)	0.006 (2)
O10A	0.0396 (15)	0.0643 (17)	0.0486 (14)	−0.0070 (14)	−0.0077 (12)	−0.0090 (14)
C1	0.0352 (19)	0.0392 (19)	0.0372 (18)	0.0036 (17)	−0.0021 (15)	0.0062 (16)
C2	0.044 (2)	0.038 (2)	0.049 (2)	0.0052 (19)	0.0031 (18)	0.0057 (18)
C4	0.041 (2)	0.038 (2)	0.0414 (19)	0.0093 (17)	0.0003 (16)	0.0024 (17)
C5	0.066 (3)	0.054 (2)	0.042 (2)	0.011 (2)	−0.007 (2)	0.0043 (19)
C6	0.067 (3)	0.054 (3)	0.055 (2)	0.017 (2)	−0.013 (2)	0.013 (2)
C7	0.060 (3)	0.041 (2)	0.056 (2)	0.013 (2)	−0.005 (2)	0.007 (2)

C8	0.046 (2)	0.037 (2)	0.0417 (19)	0.0068 (18)	0.0014 (17)	0.0027 (17)
C9	0.068 (3)	0.041 (2)	0.055 (2)	0.001 (2)	−0.010 (2)	0.006 (2)
C11	0.043 (2)	0.042 (2)	0.056 (2)	−0.0035 (18)	−0.0037 (18)	0.007 (2)
C12	0.0342 (19)	0.0336 (19)	0.0398 (18)	0.0037 (15)	0.0008 (15)	0.0043 (16)
C13	0.0319 (18)	0.0322 (18)	0.0377 (17)	0.0019 (15)	−0.0034 (15)	0.0031 (15)
C14	0.039 (2)	0.045 (2)	0.0410 (19)	0.0022 (18)	0.0048 (16)	0.0032 (17)
C15	0.046 (2)	0.054 (2)	0.0400 (19)	−0.0024 (19)	0.0048 (18)	0.0046 (19)
C16	0.047 (2)	0.038 (2)	0.042 (2)	−0.0067 (18)	−0.0008 (16)	0.0084 (18)
C17	0.056 (2)	0.037 (2)	0.0431 (19)	0.0015 (19)	−0.0081 (18)	0.0045 (17)
C18	0.041 (2)	0.041 (2)	0.051 (2)	0.0092 (18)	−0.0061 (19)	0.0014 (18)
C19	0.043 (2)	0.0327 (19)	0.0399 (19)	−0.0008 (17)	−0.0051 (16)	0.0004 (17)
C20	0.083 (3)	0.075 (3)	0.050 (2)	0.001 (3)	−0.020 (2)	0.015 (2)
C21	0.085 (3)	0.052 (3)	0.083 (3)	0.030 (3)	−0.007 (3)	0.001 (3)
C22	0.087 (3)	0.052 (3)	0.076 (3)	0.000 (3)	0.000 (3)	0.019 (2)
C23	0.064 (3)	0.060 (3)	0.038 (2)	−0.012 (2)	0.004 (2)	−0.002 (2)
C24	0.143 (5)	0.062 (3)	0.056 (3)	−0.004 (3)	0.020 (3)	−0.015 (2)
C1A	0.041 (2)	0.043 (2)	0.0389 (18)	0.0118 (18)	0.0010 (16)	−0.0014 (17)
C2A	0.048 (2)	0.059 (3)	0.050 (2)	0.008 (2)	0.0031 (19)	−0.013 (2)
C4A	0.048 (2)	0.062 (3)	0.0360 (19)	−0.009 (2)	0.0023 (17)	0.0004 (19)
C5A	0.074 (3)	0.090 (4)	0.0322 (19)	−0.027 (3)	−0.001 (2)	0.001 (2)
C6A	0.073 (3)	0.085 (3)	0.043 (2)	−0.025 (3)	−0.013 (2)	0.024 (2)
C7A	0.054 (2)	0.057 (3)	0.045 (2)	−0.020 (2)	−0.0078 (19)	0.016 (2)
C8A	0.0341 (19)	0.048 (2)	0.0374 (18)	−0.0072 (17)	−0.0020 (15)	0.0089 (17)
C9A	0.041 (2)	0.053 (2)	0.0372 (19)	−0.010 (2)	0.0041 (17)	0.0028 (19)
C11A	0.037 (2)	0.063 (3)	0.048 (2)	−0.0024 (19)	0.0049 (17)	−0.009 (2)
C12A	0.0348 (19)	0.049 (2)	0.0320 (17)	−0.0035 (17)	0.0030 (15)	−0.0006 (17)
C13A	0.0362 (19)	0.0389 (19)	0.0327 (17)	0.0032 (16)	0.0031 (15)	0.0034 (15)
C14A	0.042 (2)	0.040 (2)	0.056 (2)	0.0104 (18)	0.0109 (19)	0.011 (2)
C15A	0.060 (3)	0.041 (2)	0.070 (3)	0.009 (2)	0.027 (2)	0.009 (2)
C16A	0.058 (2)	0.037 (2)	0.061 (2)	−0.001 (2)	0.014 (2)	0.004 (2)
C17A	0.103 (4)	0.037 (2)	0.046 (2)	0.015 (2)	0.004 (2)	0.0053 (19)
C18A	0.070 (3)	0.045 (2)	0.049 (2)	0.025 (2)	−0.011 (2)	0.000 (2)
C19A	0.048 (2)	0.036 (2)	0.049 (2)	0.0019 (18)	0.0008 (17)	0.0035 (18)
C20A	0.162 (5)	0.063 (3)	0.049 (3)	0.024 (4)	0.003 (3)	0.007 (2)
C21A	0.081 (3)	0.069 (3)	0.084 (3)	−0.009 (3)	−0.018 (3)	0.035 (3)
C22A	0.076 (3)	0.089 (4)	0.051 (2)	−0.040 (3)	0.000 (2)	0.021 (2)
C23A	0.066 (3)	0.065 (3)	0.051 (3)	−0.012 (2)	0.001 (2)	−0.003 (2)
C24A	0.081 (3)	0.067 (3)	0.072 (3)	−0.021 (3)	0.011 (3)	−0.021 (3)

Geometric parameters (Å, °)

O1—C2	1.214 (4)	C20—H20B	0.9600
O2—C9	1.197 (5)	C20—H20C	0.9600
O3—C2	1.323 (4)	C21—H21A	0.9600
O3—C4	1.457 (4)	C21—H21B	0.9600
O4—C14	1.434 (4)	C21—H21C	0.9600
O4—H4	0.8200	C22—H22A	0.9600
O5—C18	1.204 (4)	C22—H22B	0.9600
O6—C23	1.356 (4)	C22—H22C	0.9600

O6—C19	1.446 (4)	C23—C24	1.500 (6)
O7—C23	1.179 (5)	C24—H24A	0.9600
O10—C9	1.368 (5)	C24—H24B	0.9600
O10—C11	1.434 (4)	C24—H24C	0.9600
O1A—C2A	1.196 (4)	C1A—C2A	1.522 (5)
O2A—C9A	1.205 (4)	C1A—C18A	1.540 (5)
O3A—C2A	1.347 (5)	C1A—C19A	1.550 (5)
O3A—C4A	1.451 (5)	C1A—C13A	1.581 (5)
O4A—C14A	1.427 (4)	C4A—C5A	1.502 (5)
O4A—H4A	0.8200	C4A—C12A	1.519 (5)
O5A—C18A	1.201 (5)	C4A—H4C	0.9800
O6A—C23A	1.338 (5)	C5A—C6A	1.516 (6)
O6A—C19A	1.445 (4)	C5A—H5C	0.9700
O7A—C23A	1.187 (5)	C5A—H5D	0.9700
O10A—C9A	1.346 (4)	C6A—C7A	1.522 (6)
O10A—C11A	1.450 (4)	C6A—H6C	0.9700
C1—C2	1.511 (5)	C6A—H6D	0.9700
C1—C19	1.545 (5)	C7A—C21A	1.538 (6)
C1—C18	1.549 (5)	C7A—C22A	1.541 (6)
C1—C13	1.581 (5)	C7A—C8A	1.576 (5)
C4—C5	1.500 (5)	C8A—C9A	1.505 (5)
C4—C12	1.523 (5)	C8A—C12A	1.547 (5)
C4—H4B	0.9800	C8A—H8A	0.9800
C5—C6	1.523 (5)	C11A—C12A	1.536 (5)
C5—H5A	0.9700	C11A—H11C	0.9700
C5—H5B	0.9700	C11A—H11D	0.9700
C6—C7	1.537 (5)	C12A—C13A	1.549 (5)
C6—H6A	0.9700	C13A—C14A	1.534 (5)
C6—H6B	0.9700	C13A—H13A	0.9800
C7—C22	1.532 (6)	C14A—C15A	1.516 (5)
C7—C21	1.537 (5)	C14A—H14A	0.9800
C7—C8	1.572 (5)	C15A—C16A	1.543 (5)
C8—C9	1.505 (5)	C15A—H15C	0.9700
C8—C12	1.546 (5)	C15A—H15D	0.9700
C8—H8	0.9800	C16A—C17A	1.519 (6)
C11—C12	1.537 (5)	C16A—C19A	1.519 (5)
C11—H11A	0.9700	C16A—H16A	0.9800
C11—H11B	0.9700	C17A—C18A	1.511 (6)
C12—C13	1.547 (4)	C17A—C20A	1.530 (5)
C13—C14	1.537 (4)	C17A—H17A	0.9800
C13—H13	0.9800	C19A—H19A	0.9800
C14—C15	1.526 (5)	C20A—H20D	0.9600
C14—H14	0.9800	C20A—H20E	0.9600
C15—C16	1.537 (5)	C20A—H20F	0.9600
C15—H15A	0.9700	C21A—H21D	0.9600
C15—H15B	0.9700	C21A—H21E	0.9600
C16—C19	1.525 (5)	C21A—H21F	0.9600
C16—C17	1.536 (5)	C22A—H22D	0.9600
C16—H16	0.9800	C22A—H22E	0.9600

C17—C18	1.507 (5)	C22A—H22F	0.9600
C17—C20	1.523 (5)	C23A—C24A	1.488 (6)
C17—H17	0.9800	C24A—H24D	0.9600
C19—H19	0.9800	C24A—H24E	0.9600
C20—H20A	0.9600	C24A—H24F	0.9600
C2—O3—C4	117.3 (3)	H24A—C24—H24B	109.5
C14—O4—H4	109.5	C23—C24—H24C	109.5
C23—O6—C19	117.4 (3)	H24A—C24—H24C	109.5
C9—O10—C11	109.8 (3)	H24B—C24—H24C	109.5
C2A—O3A—C4A	117.6 (3)	C2A—C1A—C18A	110.6 (3)
C14A—O4A—H4A	109.5	C2A—C1A—C19A	112.2 (3)
C23A—O6A—C19A	116.8 (3)	C18A—C1A—C19A	100.9 (3)
C9A—O10A—C11A	110.5 (3)	C2A—C1A—C13A	116.9 (3)
C2—C1—C19	112.1 (3)	C18A—C1A—C13A	104.7 (3)
C2—C1—C18	112.0 (3)	C19A—C1A—C13A	110.1 (3)
C19—C1—C18	100.9 (3)	O1A—C2A—O3A	119.2 (4)
C2—C1—C13	115.9 (3)	O1A—C2A—C1A	122.5 (4)
C19—C1—C13	109.9 (3)	O3A—C2A—C1A	118.3 (3)
C18—C1—C13	104.7 (3)	O3A—C4A—C5A	108.7 (3)
O1—C2—O3	118.0 (3)	O3A—C4A—C12A	107.8 (3)
O1—C2—C1	122.5 (3)	C5A—C4A—C12A	113.8 (3)
O3—C2—C1	119.5 (3)	O3A—C4A—H4C	108.8
O3—C4—C5	107.2 (3)	C5A—C4A—H4C	108.8
O3—C4—C12	107.2 (3)	C12A—C4A—H4C	108.8
C5—C4—C12	114.7 (3)	C4A—C5A—C6A	109.2 (3)
O3—C4—H4B	109.2	C4A—C5A—H5C	109.8
C5—C4—H4B	109.2	C6A—C5A—H5C	109.8
C12—C4—H4B	109.2	C4A—C5A—H5D	109.8
C4—C5—C6	108.2 (3)	C6A—C5A—H5D	109.8
C4—C5—H5A	110.1	H5C—C5A—H5D	108.3
C6—C5—H5A	110.1	C5A—C6A—C7A	112.8 (4)
C4—C5—H5B	110.1	C5A—C6A—H6C	109.0
C6—C5—H5B	110.1	C7A—C6A—H6C	109.0
H5A—C5—H5B	108.4	C5A—C6A—H6D	109.0
C5—C6—C7	113.1 (3)	C7A—C6A—H6D	109.0
C5—C6—H6A	109.0	H6C—C6A—H6D	107.8
C7—C6—H6A	109.0	C6A—C7A—C21A	109.4 (4)
C5—C6—H6B	109.0	C6A—C7A—C22A	109.8 (4)
C7—C6—H6B	109.0	C21A—C7A—C22A	107.5 (4)
H6A—C6—H6B	107.8	C6A—C7A—C8A	108.6 (3)
C22—C7—C6	110.2 (3)	C21A—C7A—C8A	107.5 (3)
C22—C7—C21	107.9 (3)	C22A—C7A—C8A	113.9 (3)
C6—C7—C21	108.3 (3)	C9A—C8A—C12A	102.2 (3)
C22—C7—C8	114.0 (3)	C9A—C8A—C7A	109.2 (3)
C6—C7—C8	108.6 (3)	C12A—C8A—C7A	117.4 (3)
C21—C7—C8	107.7 (3)	C9A—C8A—H8A	109.2
C9—C8—C12	101.6 (3)	C12A—C8A—H8A	109.2
C9—C8—C7	110.6 (3)	C7A—C8A—H8A	109.2
C12—C8—C7	117.1 (3)	O2A—C9A—O10A	120.4 (3)

C9—C8—H8	109.0	O2A—C9A—C8A	129.1 (4)
C12—C8—H8	109.0	O10A—C9A—C8A	110.4 (3)
C7—C8—H8	109.0	O10A—C11A—C12A	105.1 (3)
O2—C9—O10	120.3 (4)	O10A—C11A—H11C	110.7
O2—C9—C8	129.7 (4)	C12A—C11A—H11C	110.7
O10—C9—C8	110.0 (3)	O10A—C11A—H11D	110.7
O10—C11—C12	105.7 (3)	C12A—C11A—H11D	110.7
O10—C11—H11A	110.6	H11C—C11A—H11D	108.8
C12—C11—H11A	110.6	C4A—C12A—C11A	112.2 (3)
O10—C11—H11B	110.6	C4A—C12A—C8A	115.3 (3)
C12—C11—H11B	110.6	C11A—C12A—C8A	101.2 (3)
H11A—C11—H11B	108.7	C4A—C12A—C13A	107.5 (3)
C4—C12—C11	112.7 (3)	C11A—C12A—C13A	107.3 (3)
C4—C12—C8	114.9 (3)	C8A—C12A—C13A	113.1 (3)
C11—C12—C8	100.6 (3)	C14A—C13A—C12A	116.6 (3)
C4—C12—C13	108.2 (3)	C14A—C13A—C1A	111.0 (3)
C11—C12—C13	107.0 (3)	C12A—C13A—C1A	110.2 (3)
C8—C12—C13	113.2 (3)	C14A—C13A—H13A	106.1
C14—C13—C12	117.1 (3)	C12A—C13A—H13A	106.1
C14—C13—C1	112.6 (3)	C1A—C13A—H13A	106.1
C12—C13—C1	109.3 (2)	O4A—C14A—C15A	110.4 (3)
C14—C13—H13	105.7	O4A—C14A—C13A	109.6 (3)
C12—C13—H13	105.7	C15A—C14A—C13A	111.1 (3)
C1—C13—H13	105.7	O4A—C14A—H14A	108.6
O4—C14—C15	111.7 (3)	C15A—C14A—H14A	108.6
O4—C14—C13	108.1 (2)	C13A—C14A—H14A	108.6
C15—C14—C13	111.8 (3)	C14A—C15A—C16A	114.4 (3)
O4—C14—H14	108.4	C14A—C15A—H15C	108.7
C15—C14—H14	108.4	C16A—C15A—H15C	108.7
C13—C14—H14	108.4	C14A—C15A—H15D	108.7
C14—C15—C16	115.0 (3)	C16A—C15A—H15D	108.7
C14—C15—H15A	108.5	H15C—C15A—H15D	107.6
C16—C15—H15A	108.5	C17A—C16A—C19A	101.9 (3)
C14—C15—H15B	108.5	C17A—C16A—C15A	114.8 (4)
C16—C15—H15B	108.5	C19A—C16A—C15A	107.9 (3)
H15A—C15—H15B	107.5	C17A—C16A—H16A	110.6
C19—C16—C17	101.8 (3)	C19A—C16A—H16A	110.6
C19—C16—C15	109.0 (3)	C15A—C16A—H16A	110.6
C17—C16—C15	114.4 (3)	C18A—C17A—C16A	104.3 (3)
C19—C16—H16	110.5	C18A—C17A—C20A	112.8 (4)
C17—C16—H16	110.5	C16A—C17A—C20A	119.1 (4)
C15—C16—H16	110.5	C18A—C17A—H17A	106.6
C18—C17—C20	114.7 (3)	C16A—C17A—H17A	106.6
C18—C17—C16	103.2 (3)	C20A—C17A—H17A	106.6
C20—C17—C16	119.6 (3)	O5A—C18A—C17A	127.4 (4)
C18—C17—H17	106.1	O5A—C18A—C1A	124.6 (4)
C20—C17—H17	106.1	C17A—C18A—C1A	107.8 (3)
C16—C17—H17	106.1	O6A—C19A—C16A	107.2 (3)
O5—C18—C17	127.2 (3)	O6A—C19A—C1A	110.3 (3)

O5—C18—C1	124.0 (3)	C16A—C19A—C1A	100.9 (3)
C17—C18—C1	108.6 (3)	O6A—C19A—H19A	112.6
O6—C19—C16	107.1 (3)	C16A—C19A—H19A	112.6
O6—C19—C1	110.1 (3)	C1A—C19A—H19A	112.6
C16—C19—C1	101.1 (3)	C17A—C20A—H20D	109.5
O6—C19—H19	112.6	C17A—C20A—H20E	109.5
C16—C19—H19	112.6	H20D—C20A—H20E	109.5
C1—C19—H19	112.6	C17A—C20A—H20F	109.5
C17—C20—H20A	109.5	H20D—C20A—H20F	109.5
C17—C20—H20B	109.5	H20E—C20A—H20F	109.5
H20A—C20—H20B	109.5	C7A—C21A—H21D	109.5
C17—C20—H20C	109.5	C7A—C21A—H21E	109.5
H20A—C20—H20C	109.5	H21D—C21A—H21E	109.5
H20B—C20—H20C	109.5	C7A—C21A—H21F	109.5
C7—C21—H21A	109.5	H21D—C21A—H21F	109.5
C7—C21—H21B	109.5	H21E—C21A—H21F	109.5
H21A—C21—H21B	109.5	C7A—C22A—H22D	109.5
C7—C21—H21C	109.5	C7A—C22A—H22E	109.5
H21A—C21—H21C	109.5	H22D—C22A—H22E	109.5
H21B—C21—H21C	109.5	C7A—C22A—H22F	109.5
C7—C22—H22A	109.5	H22D—C22A—H22F	109.5
C7—C22—H22B	109.5	H22E—C22A—H22F	109.5
H22A—C22—H22B	109.5	O7A—C23A—O6A	123.0 (4)
C7—C22—H22C	109.5	O7A—C23A—C24A	126.2 (4)
H22A—C22—H22C	109.5	O6A—C23A—C24A	110.7 (4)
H22B—C22—H22C	109.5	C23A—C24A—H24D	109.5
O7—C23—O6	123.4 (4)	C23A—C24A—H24E	109.5
O7—C23—C24	126.7 (4)	H24D—C24A—H24E	109.5
O6—C23—C24	109.9 (4)	C23A—C24A—H24F	109.5
C23—C24—H24A	109.5	H24D—C24A—H24F	109.5
C23—C24—H24B	109.5	H24E—C24A—H24F	109.5
C4—O3—C2—O1	178.7 (3)	C4A—O3A—C2A—O1A	−176.8 (4)
C4—O3—C2—C1	−2.3 (5)	C4A—O3A—C2A—C1A	2.1 (5)
C19—C1—C2—O1	85.4 (4)	C18A—C1A—C2A—O1A	−32.5 (5)
C18—C1—C2—O1	−27.2 (5)	C19A—C1A—C2A—O1A	79.3 (5)
C13—C1—C2—O1	−147.3 (3)	C13A—C1A—C2A—O1A	−152.1 (4)
C19—C1—C2—O3	−93.5 (4)	C18A—C1A—C2A—O3A	148.6 (3)
C18—C1—C2—O3	153.9 (3)	C19A—C1A—C2A—O3A	−99.6 (4)
C13—C1—C2—O3	33.8 (5)	C13A—C1A—C2A—O3A	29.0 (5)
C2—O3—C4—C5	−174.0 (3)	C2A—O3A—C4A—C5A	−176.9 (3)
C2—O3—C4—C12	−50.4 (4)	C2A—O3A—C4A—C12A	−53.1 (4)
O3—C4—C5—C6	174.9 (3)	O3A—C4A—C5A—C6A	176.0 (3)
C12—C4—C5—C6	56.0 (4)	C12A—C4A—C5A—C6A	55.8 (4)
C4—C5—C6—C7	−65.2 (4)	C4A—C5A—C6A—C7A	−66.1 (4)
C5—C6—C7—C22	−69.4 (4)	C5A—C6A—C7A—C21A	173.1 (3)
C5—C6—C7—C21	172.8 (3)	C5A—C6A—C7A—C22A	−69.2 (4)
C5—C6—C7—C8	56.2 (4)	C5A—C6A—C7A—C8A	55.9 (4)
C22—C7—C8—C9	−32.3 (4)	C6A—C7A—C8A—C9A	−154.6 (3)
C6—C7—C8—C9	−155.6 (3)	C21A—C7A—C8A—C9A	87.1 (4)

C21—C7—C8—C9	87.4 (4)	C22A—C7A—C8A—C9A	−31.9 (5)
C22—C7—C8—C12	83.5 (4)	C6A—C7A—C8A—C12A	−39.0 (4)
C6—C7—C8—C12	−39.9 (4)	C21A—C7A—C8A—C12A	−157.3 (3)
C21—C7—C8—C12	−156.9 (3)	C22A—C7A—C8A—C12A	83.8 (4)
C11—O10—C9—O2	−178.4 (3)	C11A—O10A—C9A—O2A	179.7 (3)
C11—O10—C9—C8	3.8 (4)	C11A—O10A—C9A—C8A	1.7 (4)
C12—C8—C9—O2	158.2 (4)	C12A—C8A—C9A—O2A	161.0 (4)
C7—C8—C9—O2	−76.8 (5)	C7A—C8A—C9A—O2A	−74.0 (5)
C12—C8—C9—O10	−24.3 (4)	C12A—C8A—C9A—O10A	−21.2 (3)
C7—C8—C9—O10	100.8 (3)	C7A—C8A—C9A—O10A	103.8 (3)
C9—O10—C11—C12	18.8 (4)	C9A—O10A—C11A—C12A	18.9 (4)
O3—C4—C12—C11	−46.2 (4)	O3A—C4A—C12A—C11A	−45.7 (4)
C5—C4—C12—C11	72.7 (4)	C5A—C4A—C12A—C11A	75.0 (4)
O3—C4—C12—C8	−160.5 (3)	O3A—C4A—C12A—C8A	−160.8 (3)
C5—C4—C12—C8	−41.7 (4)	C5A—C4A—C12A—C8A	−40.1 (4)
O3—C4—C12—C13	71.9 (3)	O3A—C4A—C12A—C13A	72.1 (3)
C5—C4—C12—C13	−169.2 (3)	C5A—C4A—C12A—C13A	−167.3 (3)
O10—C11—C12—C4	−155.1 (3)	O10A—C11A—C12A—C4A	−153.8 (3)
O10—C11—C12—C8	−32.3 (3)	O10A—C11A—C12A—C8A	−30.4 (3)
O10—C11—C12—C13	86.1 (3)	O10A—C11A—C12A—C13A	88.4 (3)
C9—C8—C12—C4	154.3 (3)	C9A—C8A—C12A—C4A	151.6 (3)
C7—C8—C12—C4	33.6 (4)	C7A—C8A—C12A—C4A	32.2 (4)
C9—C8—C12—C11	33.0 (3)	C9A—C8A—C12A—C11A	30.4 (3)
C7—C8—C12—C11	−87.6 (4)	C7A—C8A—C12A—C11A	−89.1 (3)
C9—C8—C12—C13	−80.8 (3)	C9A—C8A—C12A—C13A	−84.1 (3)
C7—C8—C12—C13	158.6 (3)	C7A—C8A—C12A—C13A	156.4 (3)
C4—C12—C13—C14	89.6 (3)	C4A—C12A—C13A—C14A	87.7 (4)
C11—C12—C13—C14	−148.8 (3)	C11A—C12A—C13A—C14A	−151.5 (3)
C8—C12—C13—C14	−38.9 (4)	C8A—C12A—C13A—C14A	−40.8 (4)
C4—C12—C13—C1	−39.9 (3)	C4A—C12A—C13A—C1A	−40.1 (4)
C11—C12—C13—C1	81.8 (3)	C11A—C12A—C13A—C1A	80.8 (3)
C8—C12—C13—C1	−168.4 (3)	C8A—C12A—C13A—C1A	−168.5 (3)
C2—C1—C13—C14	−140.9 (3)	C2A—C1A—C13A—C14A	−137.7 (3)
C19—C1—C13—C14	−12.5 (4)	C18A—C1A—C13A—C14A	99.6 (3)
C18—C1—C13—C14	95.1 (3)	C19A—C1A—C13A—C14A	−8.1 (4)
C2—C1—C13—C12	−9.1 (4)	C2A—C1A—C13A—C12A	−6.9 (4)
C19—C1—C13—C12	119.3 (3)	C18A—C1A—C13A—C12A	−129.6 (3)
C18—C1—C13—C12	−133.0 (3)	C19A—C1A—C13A—C12A	122.7 (3)
C12—C13—C14—O4	−46.4 (4)	C12A—C13A—C14A—O4A	−52.4 (4)
C1—C13—C14—O4	81.4 (3)	C1A—C13A—C14A—O4A	74.9 (4)
C12—C13—C14—C15	−169.7 (3)	C12A—C13A—C14A—C15A	−174.7 (3)
C1—C13—C14—C15	−41.8 (4)	C1A—C13A—C14A—C15A	−47.4 (4)
O4—C14—C15—C16	−81.2 (4)	O4A—C14A—C15A—C16A	−78.4 (4)
C13—C14—C15—C16	40.0 (4)	C13A—C14A—C15A—C16A	43.4 (5)
C14—C15—C16—C19	17.9 (4)	C14A—C15A—C16A—C17A	−94.9 (4)
C14—C15—C16—C17	−95.2 (4)	C14A—C15A—C16A—C19A	18.0 (5)
C19—C16—C17—C18	−37.2 (3)	C19A—C16A—C17A—C18A	−35.4 (4)
C15—C16—C17—C18	80.1 (3)	C15A—C16A—C17A—C18A	81.0 (4)
C19—C16—C17—C20	−166.1 (3)	C19A—C16A—C17A—C20A	−162.3 (4)

C15—C16—C17—C20	−48.7 (5)	C15A—C16A—C17A—C20A	−45.9 (5)
C20—C17—C18—O5	−32.7 (6)	C16A—C17A—C18A—O5A	−167.4 (4)
C16—C17—C18—O5	−164.5 (4)	C20A—C17A—C18A—O5A	−36.6 (6)
C20—C17—C18—C1	142.8 (3)	C16A—C17A—C18A—C1A	8.3 (4)
C16—C17—C18—C1	11.0 (4)	C20A—C17A—C18A—C1A	139.0 (4)
C2—C1—C18—O5	−46.0 (5)	C2A—C1A—C18A—O5A	−44.0 (5)
C19—C1—C18—O5	−165.4 (4)	C19A—C1A—C18A—O5A	−162.9 (4)
C13—C1—C18—O5	80.5 (4)	C13A—C1A—C18A—O5A	82.7 (5)
C2—C1—C18—C17	138.3 (3)	C2A—C1A—C18A—C17A	140.2 (3)
C19—C1—C18—C17	18.9 (3)	C19A—C1A—C18A—C17A	21.3 (4)
C13—C1—C18—C17	−95.2 (3)	C13A—C1A—C18A—C17A	−93.1 (3)
C23—O6—C19—C16	−152.6 (3)	C23A—O6A—C19A—C16A	−154.4 (3)
C23—O6—C19—C1	98.3 (4)	C23A—O6A—C19A—C1A	96.6 (4)
C17—C16—C19—O6	−65.6 (3)	C17A—C16A—C19A—O6A	−66.5 (4)
C15—C16—C19—O6	173.2 (3)	C15A—C16A—C19A—O6A	172.2 (3)
C17—C16—C19—C1	49.6 (3)	C17A—C16A—C19A—C1A	48.9 (4)
C15—C16—C19—C1	−71.5 (3)	C15A—C16A—C19A—C1A	−72.4 (4)
C2—C1—C19—O6	−47.8 (4)	C2A—C1A—C19A—O6A	−47.3 (4)
C18—C1—C19—O6	71.5 (3)	C18A—C1A—C19A—O6A	70.4 (3)
C13—C1—C19—O6	−178.3 (2)	C13A—C1A—C19A—O6A	−179.4 (3)
C2—C1—C19—C16	−160.9 (3)	C2A—C1A—C19A—C16A	−160.4 (3)
C18—C1—C19—C16	−41.5 (3)	C18A—C1A—C19A—C16A	−42.7 (3)
C13—C1—C19—C16	68.7 (3)	C13A—C1A—C19A—C16A	67.6 (3)
C19—O6—C23—O7	−1.9 (6)	C19A—O6A—C23A—O7A	−0.2 (6)
C19—O6—C23—C24	178.6 (3)	C19A—O6A—C23A—C24A	−177.3 (3)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···O2A	0.82	2.27	2.980 (4)	145
O4A—H4A···O1^{i}	0.82	1.91	2.678 (4)	156

Symmetry codes: (i) *x*+1, *y*, *z*.

Comparison of the puckering parameters (Å, °) for the six- and five-membered rings between molecules I and II

Molecules		I			II		
Puckering parameters	Q	θ	φ	Q	θ	φ	Conformation
Ring A	0.527 (4)	159.0 (5)	271.9 (12)	0.524 (4)	157.5 (5)	272.1 (12)	chair
Ring B	0.661 (4)	107.6 (3)	94.6 (3)	0.646 (4)	109.8 (3)	95.3 (3)	screw-boat
Ring C	0.823 (4)	79.0 (3)	297.7 (3)	0.849 (4)	80.8 (3)	295.5 (3)	boat
Puckering parameters		Q2	φ2		Q2	φ2	Conformation
Ring D		0.485 (4)	131.2 (5)		0.481 (4)	134.5 (5)	twist
Ring E		0.349 (4)	282.0 (6)		0.324 (4)	284.9 (6)	envelope

Fig. 1

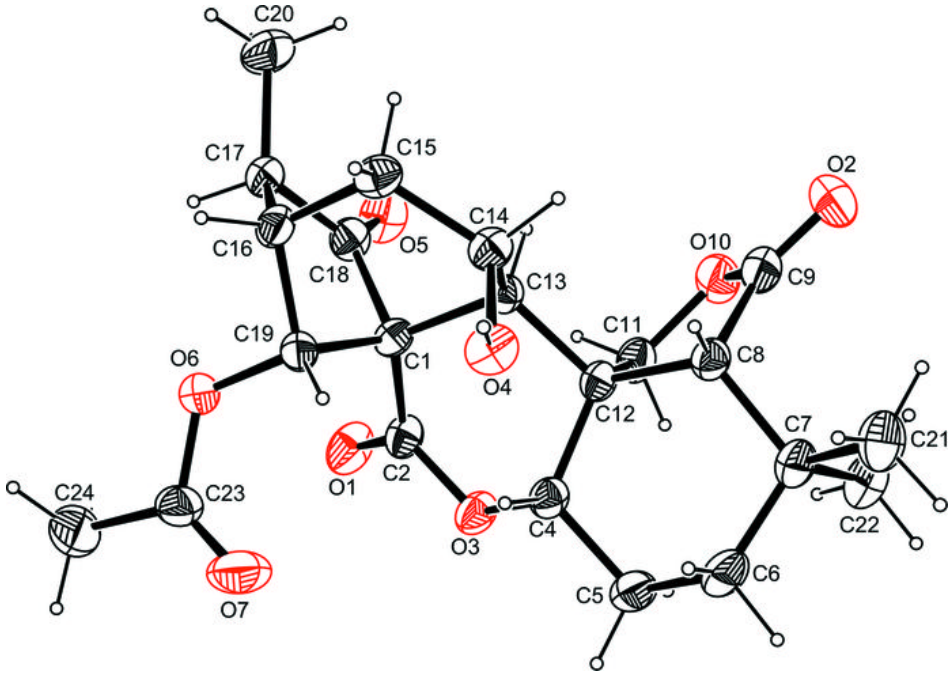

C20
C17
C15
O2
C14
O10
C16
O5
C18
C9
C13
C19
C11
O6
C1
C8
O4
C12
C7
C21
O1
C2
C24
C23
C4
C22
O3
C6
C5
O7

Fig. 2

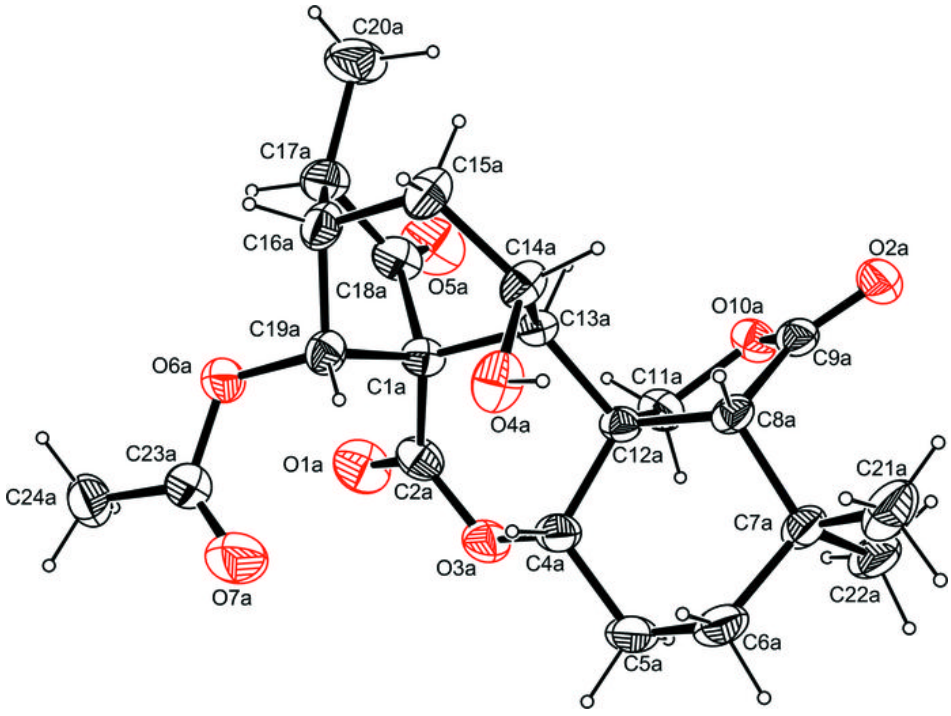

C20a
C17a
C15a
C16a
C18a
O5a
C14a
O2a
O10a
C19a
C13a
C9a
O6a
C1a
C11a
O4a
C8a
C23a
O1a
C12a
C21a
C24a
C2a
C7a
O3a
C4a
C22a
O7a
C6a
C5a

Fig. 3

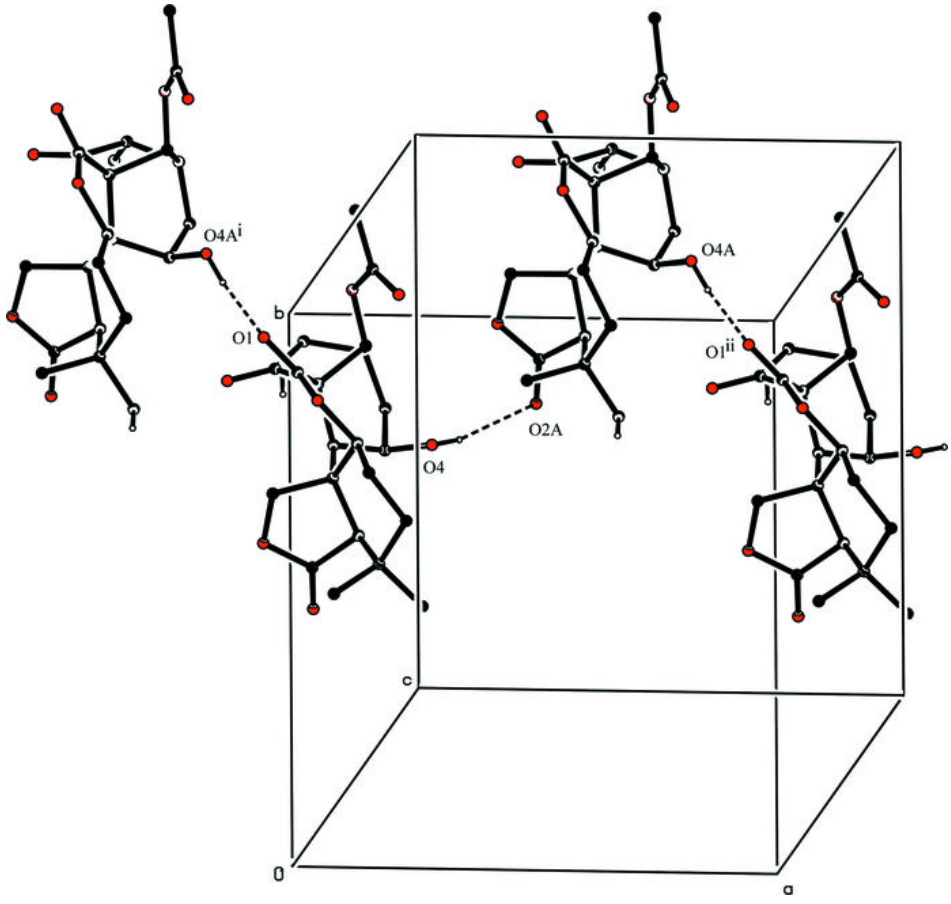
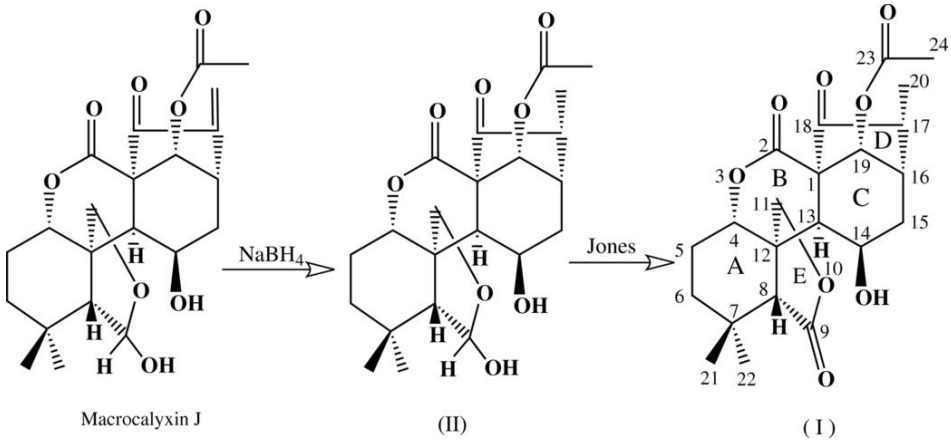


Fig. 4



Macrocalyxin J (II) (I)