Sesquiterpenes from *Thapsia nitida* var. *meridionalis* and *Thapsia nitida* var. *nitida*

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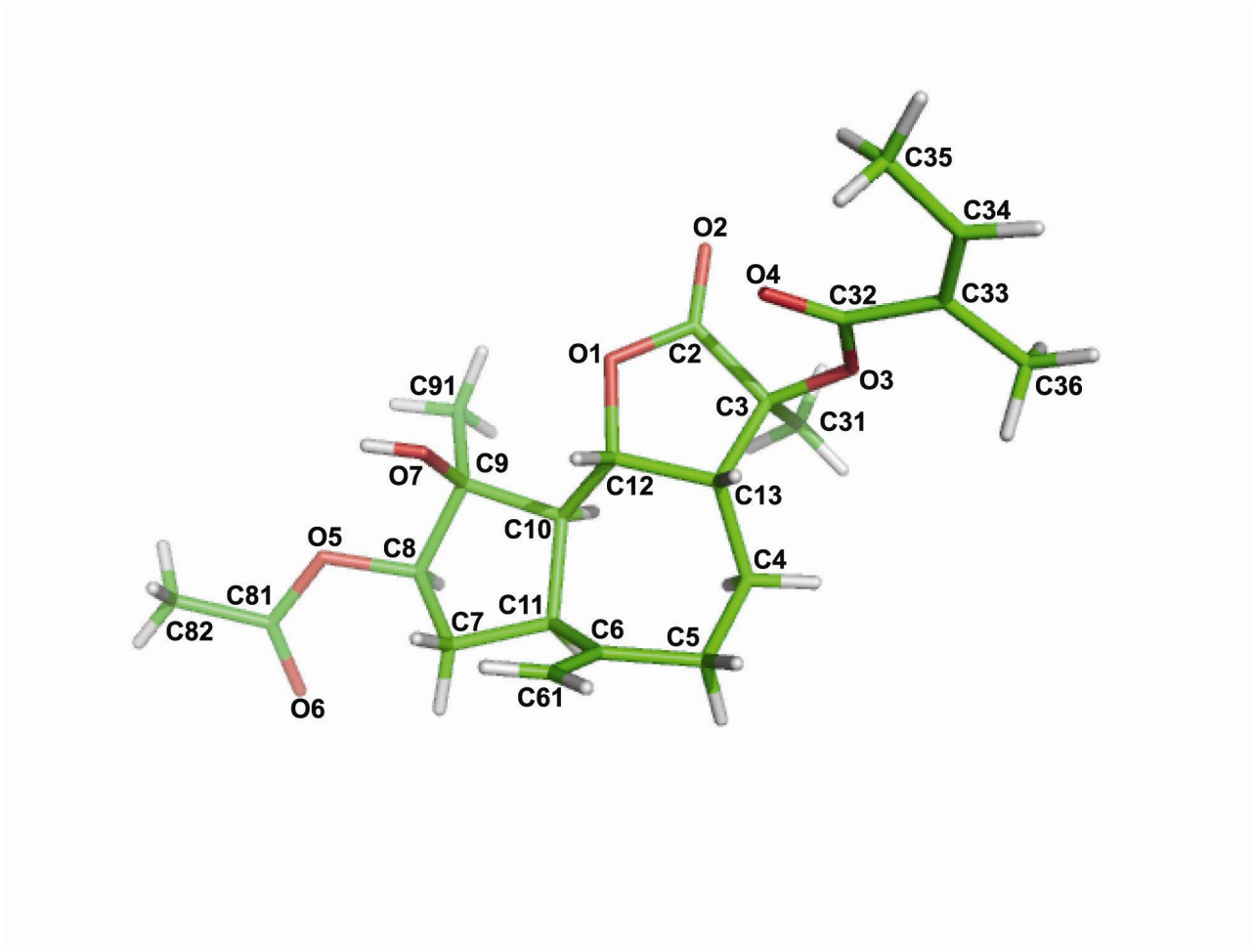
Supporting information for the X-ray structure determination of compound 13



Experimental

Experimental:

X-ray crystallographic analysis of compound 13

Crystal data: single crystals suitable for X-ray diffraction studies were grown from MeOH/H_2O. $C_{22}H_{30}O_7$, M_r 406.46, orthorhombic, space group $P2_12_12_1$ (No 19), a = 10.1786(7), b = 14.1332(11), c = 14.6231(19) Å, V = 2103.6(3) Å^3, Z = 4, D_c = 1.283 Mg/m^3, F(000) = 872, μ(Mo$K\alpha$) = 0.095 mm^{-1}, crystal size: 0.4 x 0.23 x 0.22 mm.

Data collection and reduction: a single crystal was mounted and immersed in a stream of nitrogen gas [T = 122.0(5)K]. Data were collected, using graphite monochromated Mo$K\alpha$ radiation source (λ = 0.71073 Å) on a KappaCCD diffractometer. Data collection and cell refinement were performed using COLLECT (Nonius,1999) and DIRAX (Duisenberg, 1992). Data reduction was performed using EvalCCD (Duisenberg, 1998). Correction for absorption was performed using Gaussian integration (Coppens, 1970) as included in maXus (Mackay et al., 1999).

Structure solution and refinement: positions of all non-hydrogen atoms were found by direct methods (SHELXS97) (Sheldrick, 1990; Sheldrick, 1997a)). Full-matrix least squares refinements (SHELXL97) (Sheldrick, 1997b), were performed on F^2, minimizing $\Sigma w(F_o^2\text{-}kF_c^2)^2$, with anisotropic displacement parameters of the non-hydrogen atoms. The position of the hydrogen atoms were located in subsequent difference electron density maps and refined with fixed isotropic displacement parameters (U_{iso} = 1.2U_{eq} for CH and CH_2, U_{iso} = 1.5U_{eq} for OH and CH_3). Refinement (352 parameters, 4834 unique reflections) converged at R_F = 0.033, wR_F^2 = 0.082 [4638 reflections with $F_o > 4\sigma(F_o)$; $w^{-1} = (\sigma^2(F_o^2)+(0.0435P)^2 + 0.6636P)$, where $P = (F_o^2 + 2F_c^2)/3$; S = 1.063]. The residual electron density varied between -0.17 and 0.45 e Å^{-3}. (non-centrosymmetric space group but the absolute configuration cannot be determined (Flack = 0.0(6)). Complex scattering factors for neutral atoms were taken from International Tables for Crystallography as incorporated in SHELXL97 (Sheldrick, 1997b; International Tables for Crystallography, 1995).

Fractional atomic coordinates, list of anisotropic displacement parameters and a complete list of geometrical data have been deposited in Cambridge Crystallographic Data Centre (No. CCDC 611640).

References

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Table 1. Crystal data and structure refinement of compound 13

Empirical formula	$C_{22}\ H_{30}\ O_7$
Formula weight, M_w	406.46
Temperature	122.0(5) K
Radiation wavelength (Mo$K\alpha$)	0.71073 Å
Crystal form, space group	orthorhombic, $P2_12_12_1$
Unit cell dimensions	a=10.1786(7) Å
	b=14.1332(11) Å
	c=14.6231(19) Å
Volume	2103.6(3) $Å^3$
Z, calculated density	4, 1.238 Mg/m^3
Crystal size	0.4 x 0.23 x 0.22 mm
F(000)	872
Absorption coefficient	0.095 mm^{-1}
	T_{min}= 0.975
	T_{max}=0.988
Limiting indices	$-13\leq h\leq 13$, $-18\leq k\leq 18$, $-18\leq l\leq 18$
θ range for data collection	2.00 to 27.50°
Reflections collected/unique	53722 / 4834 [R_{int}=0.0362]
Reflections observed	4638 ($I>2\sigma(I)$)
Refinement method	Full-matrix least-squares on F^2
Reflections, parameters	4834, 352
Goodness-of-fit on F^2	1.063
Final R indices [$I>2\sigma(I)$]	$R1$ = 0.0328, $wR2$ = 0.0820
R indices (all data)	$R1$ = 0.0351, $wR2$ = 0.0842
Absolute structure parameter	0.0(6)
Largest diff. peak and hole	0.446 and -0.167 $e \cdot Å^{-3}$

Table 2. Final atomic coordinates and equivalent isotropic displacement parameters (Å^2) for compound 13

	x	y	z	U_{eqv} #
O1	0.71775(9)	0.93158(7)	0.85210(7)	0.0195(2)
C2	0.74565(13)	0.92967(9)	0.76297(9)	0.0184(3)
O2	0.81445(10)	0.98814(7)	0.72733(7)	0.0252(2)
C3	0.66830(13)	0.85127(10)	0.71523(9)	0.0178(3)
C31	0.56491(16)	0.89870(12)	0.65556(11)	0.0274(3)
C4	0.48052(14)	0.74674(10)	0.77785(10)	0.0217(3)
C5	0.44299(15)	0.67597(10)	0.85303(11)	0.0239(3)
C6	0.45654(13)	0.71625(9)	0.94834(10)	0.0197(3)
C61	0.50550(19)	0.66540(12)	1.01595(12)	0.0315(3)
C7	0.37596(14)	0.84940(10)	1.05596(9)	0.0194(3)
C8	0.39855(13)	0.95544(9)	1.05234(9)	0.0167(2)
C9	0.52690(12)	0.96601(9)	0.99777(9)	0.0168(3)
C10	0.50426(13)	0.89531(9)	0.91798(9)	0.0144(2)
C11	0.40755(13)	0.81730(9)	0.95800(9)	0.0154(2)
C12	0.63080(13)	0.85354(9)	0.88009(8)	0.0146(2)
C13	0.61708(13)	0.78931(9)	0.79396(9)	0.0162(3)
O3	0.74718(10)	0.79736(7)	0.65131(6)	0.0223(2)
C32	0.86447(15)	0.76520(10)	0.68087(10)	0.0217(3)
O4	0.91010(10)	0.78722(8)	0.75437(7)	0.0249(2)
C33	0.92295(17)	0.69932(11)	0.61145(11)	0.0283(3)
C34	1.04767(18)	0.67331(12)	0.61610(13)	0.0341(4)
C35	1.14885(19)	0.70447(14)	0.68238(15)	0.0388(4)
C36	0.83113(19)	0.66259(16)	0.53705(15)	0.0411(4)
O5	0.41972(10)	0.99866(7)	1.14075(7)	0.0215(2)
C81	0.31896(14)	1.00184(10)	1.19939(10)	0.0225(3)
O6	0.20950(11)	0.97677(11)	1.18064(10)	0.0440(4)
C82	0.36094(17)	1.04040(12)	1.29035(11)	0.0272(3)
O7	0.63336(10)	0.93044(8)	1.05061(7)	0.0229(2)
C91	0.55235(18)	1.06787(10)	0.96835(11)	0.0279(3)
H311	0.503(2)	0.9361(16)	0.6923(15)	0.041*
H312	0.608(2)	0.9400(16)	0.6112(15)	0.041*
H313	0.516(2)	0.8511(16)	0.6257(15)	0.041*
H41	0.4150(19)	0.8003(13)	0.7733(13)	0.026*
H42	0.4824(19)	0.7158(13)	0.7191(13)	0.026*
H51	0.496(2)	0.6188(14)	0.8470(13)	0.029*
H52	0.353(2)	0.6578(14)	0.8424(13)	0.029*
H61	0.542(2)	0.6041(15)	1.0050(15)	0.038*
H62	0.517(2)	0.6878(15)	1.0731(16)	0.038*
H71	0.4359(19)	0.8207(13)	1.0983(13)	0.023*
H72	0.2870(18)	0.8304(13)	1.0750(12)	0.023*
H8	0.3279(17)	0.9904(12)	1.0207(12)	0.020*

H10	0.4625(16)	0.9282(12)	0.8712(12)	0.017*
H11	0.3262(17)	0.8217(12)	0.9217(12)	0.019*
H12	0.6766(17)	0.8217(12)	0.9303(12)	0.018*
H13	0.6750(17)	0.7386(13)	0.8008(12)	0.019*
H34	1.078(2)	0.6232(15)	0.5684(15)	0.041*
H351	1.132(3)	0.676(2)	0.7391(19)	0.058*
H352	1.142(3)	0.769(2)	0.6916(18)	0.058*
H352	1.242(3)	0.6875(18)	0.6622(18)	0.058*
H361	0.801(3)	0.707(2)	0.496(2)	0.062*
H362	0.747(3)	0.6389(19)	0.5655(18)	0.062*
H363	0.872(3)	0.6135(19)	0.4987(18)	0.062*
H821	0.296(2)	1.0429(16)	1.3292(15)	0.041*
H822	0.400(2)	1.0978(17)	1.2833(15)	0.041*
H823	0.430(2)	1.0037(16)	1.3153(15)	0.041*
H7	0.637(2)	0.9596(16)	1.0989(15)	0.034*
H911	0.480(2)	1.0882(16)	0.9252(16)	0.042*
H912	0.639(2)	1.0755(16)	0.9378(15)	0.042*
H913	0.550(2)	1.1069(16)	1.0239(15)	0.042*

* isotropic displacement parameters

Ueqv = $(1/3)\ \Sigma_i\ \Sigma_j\ U_{ij}\ a_i^*\ a_j^*\ \mathbf{a_i} \cdot \mathbf{a_j}$

Table 3. Anisotropic displacement parameters for non-hydrogen atoms ($Å^2$) of compound 13

	U11	U22	U33	U23	U13	U12
O1	0.0204(5)	0.0217(4)	0.0164(4)	-0.0019(4)	0.0047(4)	-0.0063(4)
C2	0.0181(6)	0.0195(6)	0.0176(6)	0.0004(5)	0.0019(5)	0.0048(5)
O2	0.0287(5)	0.0231(5)	0.0237(5)	0.0044(4)	0.0081(4)	-0.0018(4)
C3	0.0204(6)	0.0208(6)	0.0121(6)	-0.0003(5)	0.0008(5)	0.0053(5)
C31	0.0284(8)	0.0321(8)	0.0216(7)	0.0067(6)	-0.0071(6)	0.0048(6)
C4	0.0208(7)	0.0247(7)	0.0196(6)	-0.0070(5)	-0.0031(5)	-0.0023(5)
C5	0.0234(7)	0.0180(6)	0.0302(8)	-0.0054(6)	0.0026(6)	-0.0050(5)
C6	0.0180(6)	0.0161(6)	0.0250(7)	0.0003(5)	0.0042(5)	-0.0034(5)
C61	0.0412(9)	0.0216(7)	0.0315(8)	0.0036(6)	0.0034(7)	0.0054(7)
C7	0.0224(7)	0.0188(6)	0.0172(6)	0.0011(5)	0.0036(5)	-0.0027(5)
C8	0.0171(6)	0.0184(6)	0.0147(6)	-0.0019(5)	0.0006(5)	0.0005(5)
C9	0.0160(6)	0.0176(6)	0.0168(6)	-0.0013(5)	0.0017(5)	-0.0016(5)
C10	0.0149(6)	0.0148(5)	0.0135(5)	0.0012(5)	-0.0004(5)	-0.0012(5)
C11	0.0141(5)	0.0158(6)	0.0164(6)	0.0011(5)	-0.0001(5)	-0.0013(5)
C12	0.0140(5)	0.0165(6)	0.0134(5)	0.0004(5)	-0.0003(4)	-0.0019(5)
C13	0.0188(6)	0.0170(6)	0.0130(5)	-0.0005(5)	-0.0002(5)	0.0013(5)
O3	0.0277(5)	0.0258(5)	0.0134(4)	-0.0022(4)	0.0033(4)	0.0059(4)
C32	0.0248(7)	0.0199(6)	0.0205(6)	0.0049(5)	0.0047(6)	0.0020(5)
O4	0.0221(5)	0.0271(5)	0.0255(5)	0.0034(4)	0.0005(4)	0.0026(4)
C33	0.0323(8)	0.0258(7)	0.0267(7)	0.0004(6)	0.0076(6)	-0.0005(6)
C34	0.0338(8)	0.0288(8)	0.0396(9)	-0.0056(7)	0.0077(7)	0.0024(7)
C35	0.0334(9)	0.0373(9)	0.0457(10)	-0.0043(8)	-0.0005(8)	0.0079(8)
C36	0.0329(9)	0.0485(11)	0.0420(10)	-0.0255(9)	0.0082(8)	-0.0054(8)
O5	0.0192(5)	0.0273(5)	0.0181(5)	-0.0073(4)	0.0038(4)	-0.0021(4)
C81	0.0198(6)	0.0212(6)	0.0266(7)	-0.0043(6)	0.0069(5)	0.0026(5)
O6	0.0196(5)	0.0654(9)	0.0470(8)	-0.0276(7)	0.0098(5)	-0.0049(5)
C82	0.0292(7)	0.0296(8)	0.0227(7)	-0.0052(6)	0.0075(6)	-0.0004(7)
O7	0.0171(5)	0.0346(6)	0.0169(4)	-0.0090(4)	-0.0029(4)	0.0015(4)
C91	0.0362(8)	0.0163(6)	0.0313(8)	-0.0036(6)	0.0117(7)	-0.0065(6)

Table 4. Bond lengths (Å) for compound 13

O1 C2	1.3342(16)	C31 H311	0.99(2)
O1 C12	1.4723(15)	C31 H312	0.98(2)
C2 O2	1.2021(17)	C31 H313	0.94(2)
C2 C3	1.5283(19)	C4 H41	1.011(19)
C3 O3	1.4486(15)	C4 H42	0.964(19)
C3 C31	1.5226(19)	C5 H51	0.98(2)
C3 C13	1.5375(18)	C5 H52	0.96(2)
C4 C13	1.5329(19)	C61 H61	0.95(2)
C4 C5	1.535(2)	C61 H62	0.90(2)
C5 C6	1.512(2)	C7 H71	0.96(2)
C6 C61	1.320(2)	C7 H72	0.985(19)
C6 C11	1.5192(18)	C8 H8	0.987(18)
C7 C8	1.5172(18)	C10 H10	0.931(17)
C7 C11	1.5366(18)	C11 H11	0.986(18)
C8 O5	1.4460(15)	C12 H12	0.980(18)
C8 C9	1.5381(18)	C13 H13	0.933(18)
C9 O7	1.4227(16)	C34 H34	1.04(2)
C9 C91	1.5247(19)	C35 H351	0.94(3)
C9 C10	1.5533(18)	C35 H352	0.93(3)
C10 C12	1.5212(18)	C35 H352	1.02(3)
C10 C11	1.5897(18)	C36 H361	0.93(3)
C12 C13	1.5589(17)	C36 H362	1.01(3)
O3 C32	1.3486(18)	C36 H363	0.98(3)
C32 O4	1.2117(19)	C82 H821	0.87(2)
C32 C33	1.500(2)	C82 H822	0.91(2)
C33 C34	1.323(3)	C82 H823	0.95(2)
C33 C36	1.525(3)	O7 H7	0.82(2)
C34 C35	1.481(3)	C91 H911	1.01(2)
O5 C81	1.3376(17)	C91 H912	0.99(2)
C81 O6	1.2009(19)	C91 H913	0.98(2)
C81 C82	1.500(2)		

Table 5. Bond angles (°) for compound 13

C2 O1 C12	112.60(10)	C3 C31 H311	111.6(13)
O2 C2 O1	122.25(13)	C3 C31 H312	109.5(13)
O2 C2 C3	126.92(12)	H311 C31 H312	109.3(18)
O1 C2 C3	110.57(11)	C3 C31 H313	108.4(13)
O3 C3 C31	104.17(11)	H311 C31 H313	107.2(19)
O3 C3 C2	112.99(11)	H312 C31 H313	110.8(18)
C31 C3 C2	107.38(12)	C13 C4 H41	108.4(11)
O3 C3 C13	111.81(11)	C5 C4 H41	111.8(11)
C31 C3 C13	116.46(12)	C13 C4 H42	107.3(12)
C2 C3 C13	104.22(10)	C5 C4 H42	110.3(11)
C13 C4 C5	111.80(12)	H41 C4 H42	107.1(15)
C6 C5 C4	113.09(11)	C6 C5 H51	110.1(12)
C61 C6 C5	121.33(14)	C4 C5 H51	109.6(11)
C61 C6 C11	124.48(14)	C6 C5 H52	109.6(12)
C5 C6 C11	114.18(12)	C4 C5 H52	107.1(12)
C8 C7 C11	103.15(10)	H51 C5 H52	107.1(16)
O5 C8 C7	114.13(11)	C6 C61 H61	121.0(13)
O5 C8 C9	107.23(10)	C6 C61 H62	123.4(14)
C7 C8 C9	104.06(10)	H61 C61 H62	115(2)
O7 C9 C91	110.94(12)	C8 C7 H71	110.1(11)
O7 C9 C8	109.31(11)	C11 C7 H71	110.1(11)
C91 C9 C8	112.48(11)	C8 C7 H72	114.7(11)
O7 C9 C10	107.09(10)	C11 C7 H72	112.1(11)
C91 C9 C10	114.88(12)	H71 C7 H72	106.7(15)
C8 C9 C10	101.60(10)	O5 C8 H8	108.4(10)
C12 C10 C9	113.42(10)	C7 C8 H8	113.6(10)
C12 C10 C11	112.90(10)	C9 C8 H8	109.1(10)
C9 C10 C11	105.17(10)	C12 C10 H10	108.3(10)
C6 C11 C7	115.64(11)	C9 C10 H10	107.4(10)
C6 C11 C10	114.50(11)	C11 C10 H10	109.5(10)
C7 C11 C10	105.53(10)	C6 C11 H11	106.5(10)
O1 C12 C10	108.63(10)	C7 C11 H11	107.9(10)
O1 C12 C13	105.41(9)	C10 C11 H11	106.2(10)
C10 C12 C13	116.38(10)	O1 C12 H12	105.4(10)
C4 C13 C3	114.58(11)	C10 C12 H12	108.0(10)
C4 C13 C12	115.73(11)	C13 C12 H12	112.4(10)
C3 C13 C12	104.06(10)	C4 C13 H13	106.7(11)
C32 O3 C3	117.45(11)	C3 C13 H13	107.7(11)
O4 C32 O3	122.50(13)	C12 C13 H13	107.7(11)
O4 C32 C33	127.41(14)	C33 C34 H34	115.8(13)
O3 C32 C33	110.08(13)	C35 C34 H34	116.0(12)
C34 C33 C32	121.20(16)	C34 C35 H351	108.8(17)
C34 C33 C36	122.00(16)	C34 C35 H352	109.8(17)
C32 C33 C36	116.79(14)	H351 C35 H352	106(2)

C33 C34 C35	128.17(17)	C34 C35 H352	112.7(15)
C81 O5 C8	118.23(11)	H351 C35 H352	109(2)
O6 C81 O5	123.71(14)	H352 C35 H352	110(2)
O6 C81 C82	125.05(14)	C33 C36 H361	115.6(17)
O5 C81 C82	111.24(12)	C33 C36 H362	109.8(15)
		H361 C36 H362	103(2)
		C33 C36 H363	113.0(16)
		H362 C36 H363	111(2)
		C81 C82 H821	112.2(15)
		C81 C82 H822	110.3(14)
		H821 C82 H822	112(2)
		C81 C82 H823	110.8(13)
		H821 C82 H823	109.4(19)
		H822 C82 H823	102.2(19)
		C9 O7 H7	109.0(15)
		C9 C91 H911	108.6(13)
		C9 C91 H912	112.4(13)
		H911 C91 H912	109.6(17)
		C9 C91 H913	107.0(13)
		H911 C91 H913	109.6(18)
		H912 C91 H913	109.7(19)

Table 6. Torsion angles (°) for compound 13

C12 O1 C2 O2	179.43(12)	C9 C10 C11 C7	2.25(13)
C12 O1 C2 C3	5.01(14)	C2 O1 C12 C10	-118.77(11)
O2 C2 C3 O3	49.74(18)	C2 O1 C12 C13	6.63(14)
O1 C2 C3 O3	-136.16(11)	C9 C10 C12 O1	-55.75(13)
O2 C2 C3 C31	-64.55(17)	C11 C10 C12 O1	-175.26(10)
O1 C2 C3 C31	109.55(13)	C9 C10 C12 C13	-174.44(11)
O2 C2 C3 C13	171.33(13)	C11 C10 C12 C13	66.05(14)
O1 C2 C3 C13	-14.57(14)	C5 C4 C13 C3	172.72(11)
C13 C4 C5 C6	52.66(16)	C5 C4 C13 C12	-66.16(15)
C4 C5 C6 C61	-139.06(16)	O3 C3 C13 C4	-92.88(13)
C4 C5 C6 C11	41.84(16)	C31 C3 C13 C4	26.69(17)
C11 C7 C8 O5	-159.80(11)	C2 C3 C13 C4	144.75(11)
C11 C7 C8 C9	-43.27(13)	O3 C3 C13 C12	139.78(11)
O5 C8 C9 O7	52.56(13)	C31 C3 C13 C12	-100.65(14)
C7 C8 C9 O7	-68.69(13)	C2 C3 C13 C12	17.40(12)
O5 C8 C9 C91	-71.14(14)	O1 C12 C13 C4	-141.65(11)
C7 C8 C9 C91	167.61(12)	C10 C12 C13 C4	-21.22(16)
O5 C8 C9 C10	165.51(10)	O1 C12 C13 C3	-15.03(13)
C7 C8 C9 C10	44.26(13)	C10 C12 C13 C3	105.41(12)
O7 C9 C10 C12	-36.96(14)	C31 C3 O3 C32	165.01(12)
C91 C9 C10 C12	86.73(14)	C2 C3 O3 C32	48.79(15)
C8 C9 C10 C12	-151.56(10)	C13 C3 O3 C32	-68.41(15)
O7 C9 C10 C11	86.88(12)	C3 O3 C32 O4	-7.15(19)
C91 C9 C10 C11	-149.43(12)	C3 O3 C32 C33	171.52(12)
C8 C9 C10 C11	-27.72(12)	O4 C32 C33 C34	-14.8(2)
C61 C6 C11 C7	-18.8(2)	O3 C32 C33 C34	166.58(15)
C5 C6 C11 C7	160.29(12)	O4 C32 C33 C36	163.92(16)
C61 C6 C11 C10	104.26(17)	O3 C32 C33 C36	-14.7(2)
C5 C6 C11 C10	-76.67(14)	C32 C33 C34 C35	-2.9(3)
C8 C7 C11 C6	152.28(11)	C36 C33 C34 C35	178.36(19)
C8 C7 C11 C10	24.64(13)	C7 C8 O5 C81	-68.18(16)
C12 C10 C11 C6	-1.91(15)	C9 C8 O5 C81	177.15(12)
C9 C10 C11 C6	-126.08(12)	C8 O5 C81 O6	-5.3(2)
C12 C10 C11 C7	126.42(11)	C8 O5 C81 C82	174.65(12)

Table 7. Hydrogen bond geometry (Å,°)

D-H…A	D-H	H…A	D…A	D-H…A	symmetry A
O7 H7 O2	0.82(2)	2.08(2)	2.8784(15)	166(2)	1.5-x, 2-y, 0.5+z