SUPPORTING INFORMATION

for

"A Delicate Balance of Energetics. Subtleties Associated with α-Ketol-Based Bridge Migration to Afford 9-Keto-10β-*p*-Methoxybenzyloxy Taxanes"

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(18 pages)

X-Ray Crystallographic Analysis of 10.

The data collection crystal was a clear, colorless, very thin plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated a monoclinic crystal system. All work was done at 200 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure a quadrant of reciprocal space with a redundancy factor of 4, which means that 90% of the reflections were measured at least 4 times. A combination of phi and omega scans with a frame width of 1.0° were used. Because of the extreme thinness of the crystal (less than 0.04 mm), the data was measured with long exposures of 10 minutes per frame. Data integration was done with Denzo[1], and scaling and merging of the data was done with Scalepack[1]. Merging the data and averaging the symmetry equivalent reflections resulted in an Rint value of 0.048. The teXsan[2] package indicated the space group to be $P2_1$.

The structure was solved by the direct methods procedure in SHELXS-86[3]. The correct enantiomer was chosen based on the known absolute configuration of some chiral centers. There is also a water molecule present in the asymmetric unit. Full-matrix least-squares refinements based on F^2 were performed in SHELXL-93[4].

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The two hydroxyl hydrogen atoms bonded to O(1) and O(4) and the two hydrogen atoms of the water molecule were located on difference electron density maps and refined. These four hydrogen atoms are involved in intermolecular hydrogen bonds. The final refinement cycle was based on all 2444 intensities and 356 variables and resulted in agreement factors of R1(F) = 0.069 and wR2(F^2) = 0.136. For the subset of data with $I > 2\sigma(I)$, the R1(F) value is 0.050 for 1959 reflections. An extinction parameter was also refined in the final cycles. The final difference electron density map contains maximum and minimum peak heights of 0.58 and -0.20 e/$\AA^3$. This maximum peak is 1.10 $\AA$ from O(8), and based on its geometry it cannot be an alternate position for C(28). The second largest peak in the map is 0.16 e/$\AA^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion[5].

References

(1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.

(2) teXsan: Crystal Structure Analysis Package, version 1.7-2, Molecular Structure Corporation, The Woodlands, TX (1995).

(3) SHELXS-86: Sheldrick, G. M., Acta Cryst., (1990), A46, 467-473.

(4) SHELXL-93: Sheldrick, G. M., Universitat Gottingen, Germany, 1993.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

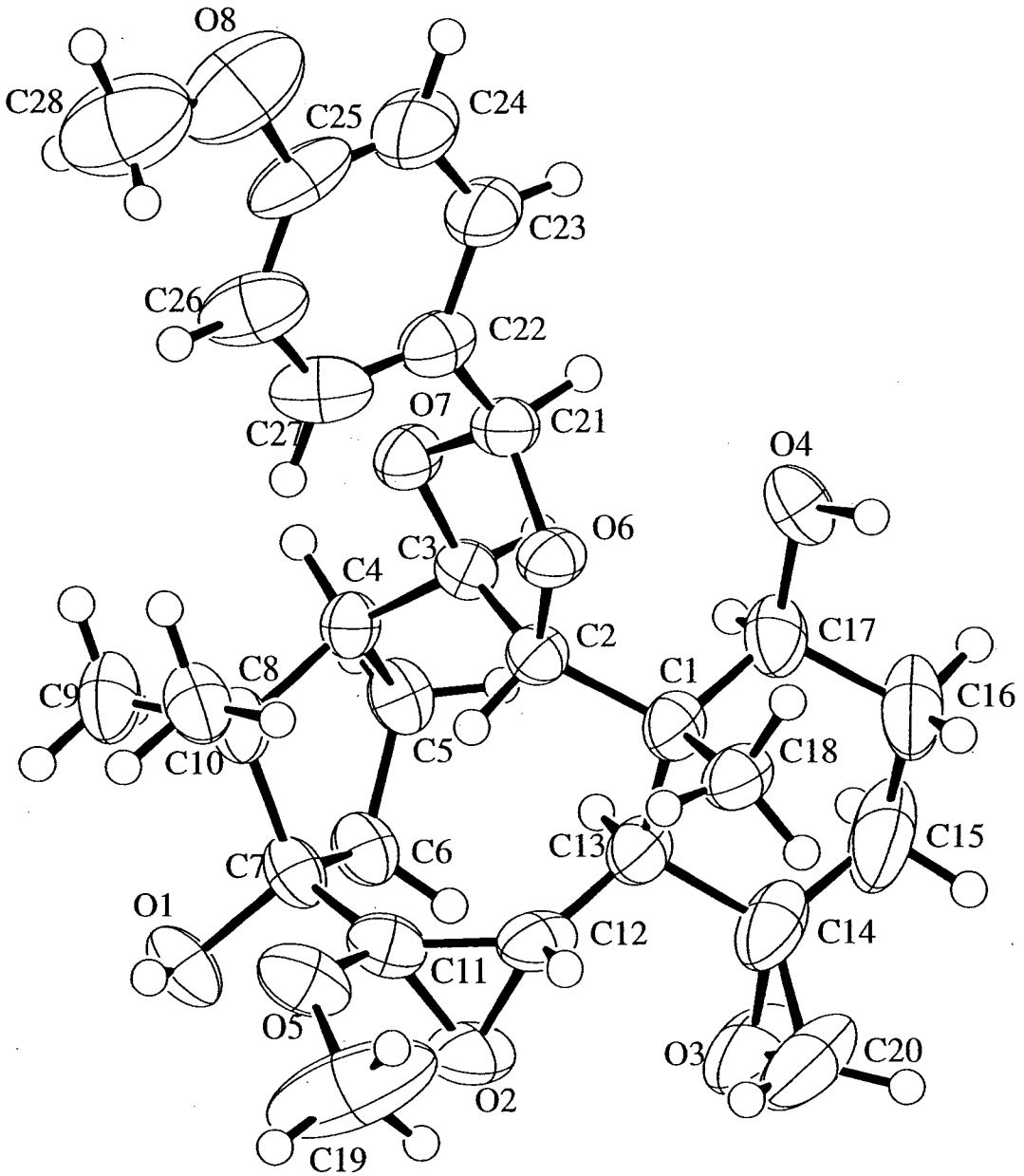

Figure 1. ORTEP drawing for **10**. The molecular structure is drawn with 50% probability displacement ellipsoids for the non-H atoms. The H atoms are drawn with circles of arbitrary radii. The water molecule has been omitted from this drawing.

Empirical formula	C28 H40 O9
Formula weight	520.60
Temperature	200 K
Wavelength	0.71073 A
Crystal system	monoclinic
Space group	P2(1)
Unit cell dimensions	a = 9.5049(2) A b = 13.5002(3) A c = 10.3767(2) A beta = 95.548(1) deg.
Volume	1325.28(5) A^3
Z	2
Density (calculated)	1.305 Mg/m^3
Absorption coefficient	0.097 mm^-1
F(000)	560
Crystal size	0.04 x 0.19 x 0.25 mm
Theta range for data collection	2.15 to 24.99 deg.
Index ranges	0<=h<=11, 0<=k<=16, -12<=l<=12
Reflections collected	20928
Independent reflections	2444 [R(int) = 0.048]
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2444 / 1 / 356
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0503, wR2 = 0.1246
R indices (all data)	R1 = 0.0686, wR2 = 0.1356
Extinction coefficient	0.024(4)
Largest diff. peak and hole	0.580 and -0.197 e/A^3

Bond lengths [A] and angles [deg] for **10**.

O(1)-C(7)	1.432(5)
O(1)-H(1O1)	0.90(7)
O(2)-C(11)	1.426(6)
O(2)-C(12)	1.463(5)
O(3)-C(20)	1.445(8)
O(3)-C(14)	1.446(6)
O(4)-C(17)	1.443(6)
O(4)-H(104)	1.18(11)
O(5)-C(11)	1.404(6)
O(5)-C(19)	1.420(8)
O(6)-C(21)	1.433(5)
O(6)-C(2)	1.448(5)
O(7)-C(21)	1.410(5)
O(7)-C(3)	1.433(5)
O(8)-C(28)	1.301(11)
O(8)-C(25)	1.343(7)
O(9)-H(1O9)	0.97(8)
O(9)-H(2O9)	1.12(9)
C(1)-C(17)	1.536(7)
C(1)-C(2)	1.549(6)
C(1)-C(18)	1.549(6)
C(1)-C(13)	1.576(5)
C(2)-C(3)	1.543(6)
C(2)-H(2)	1.00
C(3)-C(4)	1.541(5)
C(3)-H(3)	1.00
C(4)-C(5)	1.548(6)
C(4)-C(8)	1.556(6)
C(4)-H(4)	1.00
C(5)-C(6)	1.540(6)
C(5)-H(5A)	0.99
C(5)-H(5B)	0.99
C(6)-C(7)	1.534(6)
C(6)-H(6A)	0.99
C(6)-H(6B)	0.99
C(7)-C(11)	1.529(6)
C(7)-C(8)	1.581(6)
C(8)-C(10)	1.533(6)
C(8)-C(9)	1.541(6)
C(9)-H(9A)	0.98
C(9)-H(9B)	0.98
C(9)-H(9C)	0.98
C(10)-H(10A)	0.98
C(10)-H(10B)	0.98
C(10)-H(10C)	0.98
C(11)-C(12)	1.471(7)
C(12)-C(13)	1.494(7)
C(12)-H(12)	1.00
C(13)-C(14)	1.535(6)
C(13)-H(13)	1.00
C(14)-C(20)	1.478(10)
C(14)-C(15)	1.503(10)
C(15)-C(16)	1.541(8)
C(15)-H(15A)	0.99
C(15)-H(15B)	0.99
C(16)-C(17)	1.515(7)
C(16)-H(16A)	0.99
C(16)-H(16B)	0.99
C(17)-H(17)	1.00
C(18)-H(18A)	0.98
C(18)-H(18B)	0.98
C(18)-H(18C)	0.98

C(19)-H(19B)	0.98
C(19)-H(19C)	0.98
C(20)-H(20A)	0.99
C(20)-H(20B)	0.99
C(21)-C(22)	1.486(6)
C(21)-H(21)	1.00
C(22)-C(27)	1.373(8)
C(22)-C(23)	1.381(6)
C(23)-C(24)	1.382(8)
C(23)-H(23)	0.95
C(24)-C(25)	1.381(10)
C(24)-H(24)	0.95
C(25)-C(26)	1.375(9)
C(26)-C(27)	1.380(7)
C(26)-H(26)	0.95
C(27)-H(27)	0.95
C(28)-H(28A)	0.98
C(28)-H(28B)	0.98
C(28)-H(28C)	0.98

C(7)-O(1)-H(101)	103(4)
C(11)-O(2)-C(12)	61.2(3)
C(20)-O(3)-C(14)	61.5(4)
C(17)-O(4)-H(104)	111(5)
C(11)-O(5)-C(19)	117.5(5)
C(21)-O(6)-C(2)	108.0(3)
C(21)-O(7)-C(3)	105.4(3)
C(28)-O(8)-C(25)	116.9(8)
H(109)-O(9)-H(209)	109(6)
C(17)-C(1)-C(2)	114.4(4)
C(17)-C(1)-C(18)	111.8(3)
C(2)-C(1)-C(18)	107.1(4)
C(17)-C(1)-C(13)	106.3(4)
C(2)-C(1)-C(13)	106.9(3)
C(18)-C(1)-C(13)	110.4(4)
O(6)-C(2)-C(3)	103.7(3)
O(6)-C(2)-C(1)	112.1(3)
C(3)-C(2)-C(1)	117.8(4)
O(7)-C(3)-C(4)	106.4(3)
O(7)-C(3)-C(2)	102.5(3)
C(4)-C(3)-C(2)	123.6(3)
C(3)-C(4)-C(5)	114.4(3)
C(3)-C(4)-C(8)	121.3(4)
C(5)-C(4)-C(8)	103.9(3)
C(6)-C(5)-C(4)	107.5(4)
C(7)-C(6)-C(5)	108.0(4)
O(1)-C(7)-C(11)	104.2(4)
O(1)-C(7)-C(6)	107.0(3)
C(11)-C(7)-C(6)	113.1(4)
O(1)-C(7)-C(8)	111.8(3)
C(11)-C(7)-C(8)	117.0(3)
C(6)-C(7)-C(8)	103.5(4)
C(10)-C(8)-C(9)	105.8(4)
C(10)-C(8)-C(4)	116.2(3)
C(9)-C(8)-C(4)	105.4(4)
C(10)-C(8)-C(7)	116.8(4)
C(9)-C(8)-C(7)	107.3(3)
C(4)-C(8)-C(7)	104.5(3)
O(5)-C(11)-O(2)	116.2(4)
O(5)-C(11)-C(12)	118.6(4)
O(2)-C(11)-C(12)	60.6(3)
O(5)-C(11)-C(7)	107.3(4)
O(2)-C(11)-C(7)	114.8(4)
C(12)-C(11)-C(7)	130.5(4)
O(2)-C(12)-C(11)	58.1(3)

C(11)-C(12)-C(13)	129.2(4)
C(12)-C(13)-C(14)	114.0(4)
C(12)-C(13)-C(1)	110.2(4)
C(14)-C(13)-C(1)	110.1(3)
O(3)-C(14)-C(20)	59.2(4)
O(3)-C(14)-C(15)	112.5(5)
C(20)-C(14)-C(15)	120.8(5)
O(3)-C(14)-C(13)	114.7(4)
C(20)-C(14)-C(13)	120.7(6)
C(15)-C(14)-C(13)	115.1(5)
C(14)-C(15)-C(16)	113.1(5)
C(17)-C(16)-C(15)	108.5(4)
O(4)-C(17)-C(16)	110.7(4)
O(4)-C(17)-C(1)	112.8(4)
C(16)-C(17)-C(1)	113.2(5)
O(3)-C(20)-C(14)	59.3(4)
O(7)-C(21)-O(6)	104.2(3)
O(7)-C(21)-C(22)	109.1(3)
O(6)-C(21)-C(22)	113.1(4)
C(27)-C(22)-C(23)	118.6(5)
C(27)-C(22)-C(21)	121.4(4)
C(23)-C(22)-C(21)	119.8(5)
C(22)-C(23)-C(24)	121.1(6)
C(25)-C(24)-C(23)	119.1(5)
O(8)-C(25)-C(26)	125.4(7)
O(8)-C(25)-C(24)	114.0(7)
C(26)-C(25)-C(24)	120.5(5)
C(25)-C(26)-C(27)	119.3(6)
C(22)-C(27)-C(26)	121.4(5)

Hydrogen bonds in **10**.

Donor --- H....Acceptor	[ARU]	D - H	H...A	D...A	D - H...A
O(1) -- H(11O).. O(5)	[ ]	0.91(7)	2.12(6)	2.613(5)	113(4)
O(1) -- H(11O).. O(9)	[2545.02]	0.91(7)	2.08(6)	2.818(6)	138(5)
O(9) -- H(19O).. O(4)	[ ]	0.96(8)	1.91(8)	2.851(6)	165(7)
O(9) -- H(29O).. O(6)	[2655.01]	1.12(9)	2.00(9)	3.016(5)	149(7)
O(4) -- H(104).. O(1)	[1655.01]	1.18(11)	1.64(11)	2.785(5)	162(8)

Translation of ARU-code to Equivalent Position

[2545.00]	= -x,-1/2+y,-z
[1655.00]	= 1+x,y,z
[1554.00]	= x,y,-1+z
[2655.00]	= 1-x,1/2+y,-z

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **10**.

	x	y	z	U(eq)
O(1)	-3782(3)	4806(3)	-1183(4)	55(1)
O(2)	-1869(3)	4737(3)	-3299(3)	63(1)
O(3)	-293(4)	6087(4)	-4888(4)	95(2)
O(4)	4087(3)	6148(3)	-756(3)	57(1)
O(5)	-1927(3)	3466(2)	-1685(4)	64(1)
O(6)	2690(3)	4435(2)	327(3)	45(1)
O(7)	1555(3)	5344(2)	1713(3)	45(1)
O(8)	4598(8)	2695(5)	5928(5)	123(2)
O(9)	4904(4)	8073(3)	166(5)	80(1)
C(1)	1977(4)	5264(4)	-1775(4)	46(1)
C(2)	1535(4)	4909(3)	-453(4)	39(1)
C(3)	1001(4)	5695(3)	463(4)	40(1)
C(4)	-575(4)	5918(3)	557(4)	41(1)
C(5)	-1203(4)	6722(3)	-397(4)	46(1)
C(6)	-2298(4)	6213(3)	-1370(5)	47(1)
C(7)	-2332(4)	5109(3)	-1027(4)	44(1)
C(8)	-1703(4)	5080(3)	440(4)	46(1)
C(9)	-2878(5)	5429(4)	1259(5)	62(1)
C(10)	-1224(5)	4068(4)	989(5)	55(1)
C(11)	-1615(5)	4453(3)	-1973(5)	50(1)
C(12)	-407(4)	4654(4)	-2720(4)	52(1)
C(13)	573(4)	5522(4)	-2647(4)	50(1)
C(14)	923(5)	5905(6)	-3971(5)	77(2)
C(15)	1977(7)	6737(6)	-3925(6)	98(2)
C(16)	3319(5)	6528(5)	-3008(6)	79(2)
C(17)	2877(4)	6211(4)	-1706(4)	53(1)
C(18)	2748(4)	4388(4)	-2366(4)	52(1)
C(19)	-1805(15)	2748(6)	-2668(7)	158(5)
C(20)	649(7)	5286(7)	-5143(5)	102(3)
C(21)	2899(4)	4951(4)	1538(4)	47(1)
C(22)	3358(5)	4291(4)	2646(4)	51(1)
C(23)	4578(5)	4505(5)	3430(5)	70(2)
C(24)	4964(6)	3957(6)	4532(5)	82(2)
C(25)	4123(7)	3174(5)	4838(5)	76(2)
C(26)	2918(7)	2940(4)	4055(5)	76(2)
C(27)	2536(6)	3513(4)	2977(5)	67(1)
C(28)	3794(12)	2000(7)	6321(9)	128(3)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Anisotropic displacement parameters (A^2 x 10^3) for **10**.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	29(1)	50(2)	84(2)	7(2)	-1(1)	-1(1)
O(2)	45(2)	86(2)	55(2)	-10(2)	-15(1)	11(2)
O(3)	76(3)	164(5)	43(2)	20(2)	0(2)	41(3)
O(4)	34(1)	61(2)	75(2)	0(2)	9(1)	-2(2)
O(5)	52(2)	50(2)	85(2)	-16(2)	-10(2)	2(2)
O(6)	37(2)	57(2)	38(2)	-4(1)	-1(1)	7(1)
O(7)	42(2)	54(2)	39(2)	-3(1)	4(1)	8(1)
O(8)	175(6)	121(4)	70(3)	30(3)	-3(3)	50(4)
O(9)	57(2)	71(3)	117(4)	-30(2)	28(2)	-12(2)
C(1)	32(2)	63(3)	43(2)	1(2)	7(2)	12(2)
C(2)	30(2)	50(2)	37(2)	0(2)	1(2)	6(2)
C(3)	35(2)	47(2)	39(2)	1(2)	0(2)	0(2)
C(4)	36(2)	46(2)	43(2)	-3(2)	10(2)	4(2)
C(5)	39(2)	40(2)	60(3)	2(2)	9(2)	4(2)
C(6)	32(2)	49(2)	58(3)	3(2)	4(2)	7(2)
C(7)	28(2)	44(2)	58(3)	5(2)	4(2)	1(2)
C(8)	34(2)	50(3)	54(3)	4(2)	13(2)	5(2)
C(9)	46(3)	76(4)	67(3)	11(3)	23(2)	7(2)
C(10)	45(2)	57(3)	64(3)	13(2)	7(2)	-2(2)
C(11)	42(2)	49(3)	56(3)	-4(2)	-8(2)	4(2)
C(12)	40(2)	72(3)	41(2)	-10(2)	-7(2)	18(2)
C(13)	36(2)	74(3)	40(2)	3(2)	4(2)	12(2)
C(14)	50(3)	137(6)	44(3)	27(3)	10(2)	30(3)
C(15)	80(4)	147(6)	73(4)	54(4)	33(3)	17(4)
C(16)	54(3)	108(5)	78(4)	35(4)	27(3)	6(3)
C(17)	38(2)	66(3)	57(3)	13(2)	13(2)	8(2)
C(18)	40(2)	79(3)	39(2)	-3(2)	4(2)	14(2)
C(19)	332(16)	57(4)	75(5)	-15(4)	-39(7)	25(6)
C(20)	97(4)	170(7)	40(3)	8(4)	13(3)	47(5)
C(21)	39(2)	61(3)	39(2)	-7(2)	-1(2)	0(2)
C(22)	49(2)	59(3)	42(2)	-5(2)	1(2)	14(2)
C(23)	42(2)	123(5)	44(3)	3(3)	-4(2)	8(3)
C(24)	60(3)	133(6)	51(3)	15(4)	-7(3)	12(4)
C(25)	104(5)	88(4)	34(3)	2(3)	-4(3)	50(4)
C(26)	119(5)	50(3)	54(3)	-4(2)	-15(3)	11(3)
C(27)	84(4)	51(3)	59(3)	-7(3)	-21(3)	11(3)
C(28)	191(10)	100(6)	86(5)	10(5)	-14(6)	9(7)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **10**.

	x	y	z	U(eq)
H(1O1)	-3724(58)	4154(51)	-995(53)	71(17)*
H(1O4)	4968(108)	5639(83)	-1126(90)	174(37)*
H(1O9)	4588(70)	7479(61)	-293(68)	102(22)*
H(2O9)	5875(89)	8363(65)	-236(75)	130(27)*
H(2)	768(4)	4406(3)	-629(4)	47
H(3)	1483(4)	6335(3)	299(4)	48
H(4)	-622(4)	6207(3)	1439(4)	49
H(5A)	-1660(4)	7248(3)	79(4)	55
H(5B)	-446(4)	7026(3)	-856(4)	55
H(6A)	-3243(4)	6511(3)	-1320(5)	56
H(6B)	-2032(4)	6299(3)	-2263(5)	56
H(9A)	-3210(28)	6085(13)	963(25)	93
H(9B)	-2504(12)	5465(27)	2171(7)	93
H(9C)	-3667(18)	4959(16)	1162(29)	93
H(10A)	-475(25)	3806(12)	499(21)	83
H(10B)	-2028(10)	3609(8)	917(30)	83
H(10C)	-864(33)	4140(6)	1901(10)	83
H(12)	60(4)	4033(4)	-2986(4)	62
H(13)	98(4)	6073(4)	-2212(4)	60
H(15A)	1525(7)	7348(6)	-3637(6)	118
H(15B)	2251(7)	6855(6)	-4808(6)	118
H(16A)	3881(5)	5997(5)	-3372(6)	94
H(16B)	3911(5)	7131(5)	-2907(6)	94
H(17)	2264(4)	6752(4)	-1413(4)	64
H(18A)	2697(31)	4465(14)	-3308(5)	79
H(18B)	2296(22)	3764(4)	-2155(28)	79
H(18C)	3741(9)	4380(15)	-2007(25)	79
H(19A)	-2595(56)	2281(41)	-2681(60)	238
H(19B)	-911(48)	2388(47)	-2492(50)	238
H(19C)	-1824(99)	3079(8)	-3510(14)	238
H(20A)	1270(7)	5377(7)	-5847(5)	122
H(20B)	314(7)	4600(7)	-5033(5)	122
H(21)	3597(4)	5500(4)	1480(4)	56
H(23)	5161(5)	5038(5)	3209(5)	85
H(24)	5798(6)	4116(6)	5072(5)	99
H(26)	2355(7)	2390(4)	4255(5)	91
H(27)	1686(6)	3366(4)	2453(5)	80
H(28A)	4191(42)	1760(37)	7171(32)	191
H(28B)	3741(60)	1451(23)	5700(36)	191
H(28C)	2844(23)	2264(15)	6389(65)	191

*Refined isotropically.

X-Ray Crystallographic Analysis of 13.

The data collection crystal was a clear, colorless, rectangular plate. Examination of the diffraction pattern on a Nonius Kappa CCD diffractometer indicated an orthorhombic crystal system. All work was done at 200 K using an Oxford Cryosystems Cryostream Cooler. The data collection strategy was set up to measure an octant of reciprocal space with a redundancy factor of 4, which means that 90% of the reflections were measured at least 4 times. A combination of phi and omega scans with a frame width of 1.0° were used. Data integration was done with Denzo[1], and scaling and merging of the data was done with Scalepack[1]. Merging the data and averaging the symmetry equivalent reflections (including the Friedel pairs) resulted in an Rint value of 0.042. The teXsan[2] package indicated the space group to be $P2_12_12_1$.

The structure was solved by the direct methods procedure in SHELXS-86[3]. The correct enantiomer was chosen based on the known absolute configuration of some chiral centers. Full-matrix least-squares refinements based on F^2 were performed in SHELXL-93[4].

The hydrogen atoms were included in the model at calculated positions using a riding model with U(H) = 1.2 * Ueq(attached atom). The four hydroxyl hydrogen atoms bonded to O(2), O(4), O(5) and O(7) were located on difference electron density maps and refined. These four hydrogen atoms are involved in inter and intramolecular hydrogen bonds. The final refinement cycle was based on all 1886 intensities and 264 variables and resulted in agreement factors of R1(F) = 0.045 and wR2(F^2) = 0.085. For the subset of data with $I > 2\sigma(I)$, the R1(F) value is 0.034 for 1647 reflections. The final difference electron density map contains maximum and minimum peak heights of 0.16 and -0.18 e/$Å^3$. Neutral atom scattering factors were used and include terms for anomalous dispersion[5].

References

(1) DENZO: Otwinowski, Z. & Minor, W., Methods in Enzymology, Vol 276: Macromolecular Crystallography, part A, 307-326, (1997), Carter, Jr., C. W. & Sweet, R. M., Eds., Academic Press.

(2) teXsan: Crystal Structure Analysis Package, version 1.7-2, Molecular Structure Corporation, The Woodlands, TX (1995).

(3) SHELXS-86: Sheldrick, G. M., Acta Cryst., (1990), A46, 467-473.

(4) SHELXL-93: Sheldrick, G. M., Universitat Gottingen, Germany, 1993.

(5) International Tables for Crystallography (1992). Volume C. Dordrecht: Kluwer Academic Publishers.

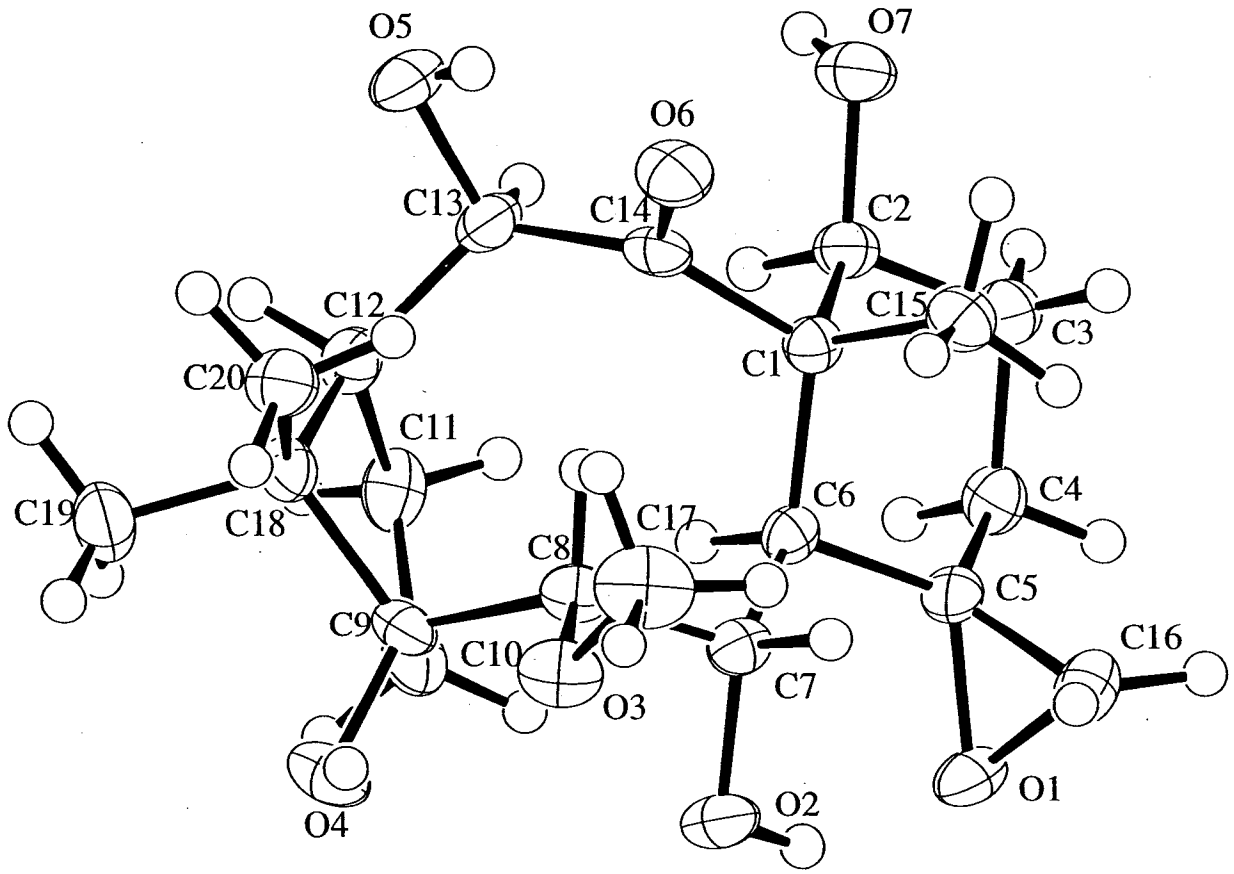

Figure 2. ORTEP drawing for **13**. The molecular structure is drawn with 50% probability displacement ellipsoids for the non-H atoms. The H atoms are drawn with circles of arbitrary radii.

Empirical formula	C20 H32 O7
Formula weight	384.46
Temperature	200 K
Wavelength	0.71073 A
Crystal system	orthorhombic
Space group	$P2_12_12_1$
Unit cell dimensions	a = 7.0758(1) A b = 14.7063(2) A c = 17.6913(3) A
Volume	1840.94(5) A^3
Z	4
Density (calculated)	1.387 Mg/m^3
Absorption coefficient	0.104 mm^-1
F(000)	832
Crystal size	0.04 x 0.19 x 0.27 mm
Theta range for data collection	2.69 to 25.02 deg.
Index ranges	0<=h<=8, 0<=k<=17, 0<=l<=21
Reflections collected	19361
Independent reflections	1886 [R(int) = 0.042]
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1886 / 0 / 264
Goodness-of-fit on F^2	1.062
Final R indices [I>2sigma(I)]	R1 = 0.0341, wR2 = 0.0799
R indices (all data)	R1 = 0.0448, wR2 = 0.0851
Largest diff. peak and hole	0.164 and -0.177 e/A^3

Bond lengths [A] and angles [deg] for **13**.

O(1)-C(16)	1.438(3)
O(1)-C(5)	1.460(3)
O(2)-C(7)	1.437(3)
O(2)-H(1O2)	0.91(4)
O(3)-C(17)	1.428(3)
O(3)-C(8)	1.438(3)
O(4)-C(9)	1.430(3)
O(4)-H(1O4)	0.85(3)
O(5)-C(13)	1.423(3)
O(5)-H(1O5)	0.84(4)
O(6)-C(14)	1.217(3)
O(7)-C(2)	1.427(3)
O(7)-H(1O7)	0.78(5)
C(1)-C(15)	1.539(3)
C(1)-C(2)	1.555(3)
C(1)-C(14)	1.559(3)
C(1)-C(6)	1.574(3)
C(2)-C(3)	1.527(3)
C(2)-H(2)	1.00
C(3)-C(4)	1.528(3)
C(3)-H(3A)	0.99
C(3)-H(3B)	0.99
C(4)-C(5)	1.503(3)
C(4)-H(4A)	0.99
C(4)-H(4B)	0.99
C(5)-C(16)	1.463(3)
C(5)-C(6)	1.535(3)
C(6)-C(7)	1.547(3)
C(6)-H(6)	1.00
C(7)-C(8)	1.520(3)
C(7)-H(7)	1.00
C(8)-C(9)	1.576(3)
C(8)-H(8)	1.00
C(9)-C(10)	1.537(3)
C(9)-C(18)	1.568(3)
C(10)-C(11)	1.535(4)
C(10)-H(10A)	0.99
C(10)-H(10B)	0.99
C(11)-C(12)	1.542(4)
C(11)-H(11A)	0.99
C(11)-H(11B)	0.99
C(12)-C(18)	1.556(3)
C(12)-C(13)	1.561(3)
C(12)-H(12)	1.00
C(13)-C(14)	1.548(3)
C(13)-H(13)	1.00
C(15)-H(15A)	0.98
C(15)-H(15B)	0.98
C(15)-H(15C)	0.98
C(16)-H(16A)	0.99
C(16)-H(16B)	0.99
C(17)-H(17A)	0.98
C(17)-H(17B)	0.98
C(17)-H(17C)	0.98
C(18)-C(20)	1.529(3)
C(18)-C(19)	1.549(3)
C(19)-H(19A)	0.98
C(19)-H(19B)	0.98
C(19)-H(19C)	0.98
C(20)-H(20A)	0.98
C(20)-H(20B)	0.98
C(20)-H(20C)	0.98

Hydrogen bonding interactions for **13**.

Nr	Typ	Res	Donor	---	H	....Acceptor	[ARU]	D - H	H...A	D...A	D - H...A
1	Intra	1	O(2)	--	H(120)	.. O(1)	[ ]	0.91(3)	1.82(3)	2.642(2)	149(3)
2	Intra	1	O(4)	--	H(140)	.. O(3)	[ ]	0.86(3)	1.96(3)	2.487(3)	118(3)
3	Intra	1	O(5)	--	H(150)	.. O(6)	[ ]	0.84(4)	1.87(4)	2.538(3)	135(3)
4	Inter	1	O(7)	--	H(170)	.. O(2)	[4646.01]	0.77(5)	2.50(5)	3.171(3)	146(5)
5	Inter	1	O(7)	--	H(170)	.. O(4)	[4646.01]	0.77(5)	2.52(5)	3.095(3)	133(5)

Translation of ARU-code to Equivalent Position Code

[4646.] = 1-x,-1/2+y,3/2-z

Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for **13**.

	x	y	z	U(eq)
O(1)	7979(2)	9948(1)	6222(1)	32(1)
O(2)	6569(2)	10549(1)	7511(1)	32(1)
O(3)	3087(2)	10739(1)	8335(1)	33(1)
O(4)	5499(3)	10231(1)	9266(1)	38(1)
O(5)	1349(3)	7167(1)	8348(1)	44(1)
O(6)	620(2)	8378(1)	7369(1)	35(1)
O(7)	2709(3)	7053(1)	6238(1)	35(1)
C(1)	3575(3)	8533(2)	6720(1)	23(1)
C(2)	4300(4)	7626(2)	6373(1)	27(1)
C(3)	5435(3)	7778(2)	5650(1)	31(1)
C(4)	7116(3)	8399(2)	5814(1)	30(1)
C(5)	6484(3)	9265(2)	6186(1)	24(1)
C(6)	5358(3)	9126(2)	6919(1)	22(1)
C(7)	4912(3)	10023(2)	7341(1)	25(1)
C(8)	3873(3)	9889(2)	8085(1)	24(1)
C(9)	5011(3)	9494(2)	8777(1)	26(1)
C(10)	6866(3)	8991(2)	8604(1)	30(1)
C(11)	6370(4)	7981(2)	8504(1)	33(1)
C(12)	4263(3)	7876(2)	8707(1)	29(1)
C(13)	2985(4)	7580(2)	8031(1)	29(1)
C(14)	2309(3)	8225(2)	7394(1)	25(1)
C(15)	2277(3)	9030(2)	6158(1)	31(1)
C(16)	6378(4)	10099(2)	5737(1)	34(1)
C(17)	1286(4)	10948(2)	8015(2)	46(1)
C(18)	3811(3)	8741(2)	9181(1)	27(1)
C(19)	4622(4)	8585(2)	9983(1)	39(1)
C(20)	1718(4)	8969(2)	9282(2)	34(1)

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

Anisotropic displacement parameters (A^2 x 10^3) for **13**.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(1)	25(1)	31(1)	39(1)	6(1)	4(1)	-7(1)
O(2)	31(1)	25(1)	40(1)	-5(1)	7(1)	-9(1)
O(3)	30(1)	25(1)	45(1)	-8(1)	2(1)	5(1)
O(4)	33(1)	42(1)	38(1)	-15(1)	-3(1)	-6(1)
O(5)	51(1)	43(1)	37(1)	2(1)	10(1)	-24(1)
O(6)	23(1)	45(1)	37(1)	-5(1)	5(1)	-5(1)
O(7)	34(1)	33(1)	39(1)	-8(1)	3(1)	-9(1)
C(1)	22(1)	22(1)	24(1)	1(1)	0(1)	0(1)
C(2)	28(1)	25(1)	28(1)	-2(1)	1(1)	-4(1)
C(3)	33(1)	31(1)	28(1)	-5(1)	5(1)	-3(1)
C(4)	28(1)	34(1)	26(1)	-1(1)	6(1)	0(1)
C(5)	17(1)	30(1)	26(1)	3(1)	0(1)	-4(1)
C(6)	22(1)	21(1)	24(1)	4(1)	-1(1)	1(1)
C(7)	22(1)	23(1)	30(1)	3(1)	-1(1)	-1(1)
C(8)	23(1)	19(1)	32(1)	-4(1)	0(1)	3(1)
C(9)	22(1)	31(1)	25(1)	-6(1)	-4(1)	-4(1)
C(10)	20(1)	41(1)	28(1)	1(1)	-4(1)	2(1)
C(11)	31(1)	37(1)	31(1)	8(1)	0(1)	12(1)
C(12)	33(1)	27(1)	27(1)	4(1)	1(1)	0(1)
C(13)	35(1)	22(1)	28(1)	1(1)	7(1)	-7(1)
C(14)	25(2)	21(1)	29(1)	-9(1)	1(1)	-5(1)
C(15)	24(1)	35(1)	33(1)	2(1)	-4(1)	0(1)
C(16)	28(1)	40(2)	33(1)	9(1)	5(1)	0(1)
C(17)	31(2)	39(2)	67(2)	-5(1)	0(1)	12(1)
C(18)	23(1)	35(1)	22(1)	-1(1)	2(1)	0(1)
C(19)	42(2)	52(2)	24(1)	1(1)	-2(1)	-3(1)
C(20)	31(1)	36(1)	33(1)	-5(1)	7(1)	-3(1)

Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for **13**.

	x	y	z	U(eq)
H(1O2)	7369(54)	10497(22)	7110(19)	67(11) *
H(1O4)	4543(44)	10585(20)	9272(17)	49(9) *
H(1O5)	529(54)	7481(27)	8123(21)	79(14) *
H(1O7)	3073(75)	6586(33)	6380(26)	119(20) *
H(2)	5137(4)	7322(2)	6752(1)	32
H(3A)	4618(3)	8059(2)	5260(1)	37
H(3B)	5891(3)	7187(2)	5454(1)	37
H(4A)	8021(3)	8079(2)	6148(1)	36
H(4B)	7774(3)	8544(2)	5335(1)	36
H(6)	6167(3)	8753(2)	7264(1)	27
H(7)	4086(3)	10399(2)	7005(1)	30
H(8)	2792(3)	9468(2)	7986(1)	29
H(10A)	7447(3)	9233(2)	8137(1)	36
H(10B)	7771(3)	9069(2)	9026(1)	36
H(11A)	7159(4)	7601(2)	8841(1)	39
H(11B)	6596(4)	7790(2)	7975(1)	39
H(12)	4210(3)	7360(2)	9074(1)	35
H(13)	3687(4)	7079(2)	7771(1)	34
H(15A)	3042(4)	9296(9)	5752(5)	46
H(15B)	1590(16)	9514(7)	6421(2)	46
H(15C)	1373(14)	8597(3)	5941(6)	46
H(16A)	6577(4)	10048(2)	5185(1)	40
H(16B)	5449(4)	10566(2)	5892(1)	40
H(17A)	814(13)	11518(7)	8231(8)	69
H(17B)	396(8)	10456(6)	8128(9)	69
H(17C)	1411(6)	11013(13)	7466(2)	69
H(19A)	5963(8)	8424(12)	9946(1)	59
H(19B)	3931(17)	8089(8)	10230(4)	59
H(19C)	4486(23)	9142(4)	10282(3)	59
H(20A)	1111(6)	9009(11)	8785(2)	50
H(20B)	1596(4)	9553(6)	9544(8)	50
H(20C)	1107(6)	8491(6)	9580(8)	50

*Refined isotropically