T-Muurolol Sesquiterpenes from the Marine *Streptomyces* sp. M491 and Revision of the Configuration of Previously Reported Amorphanes

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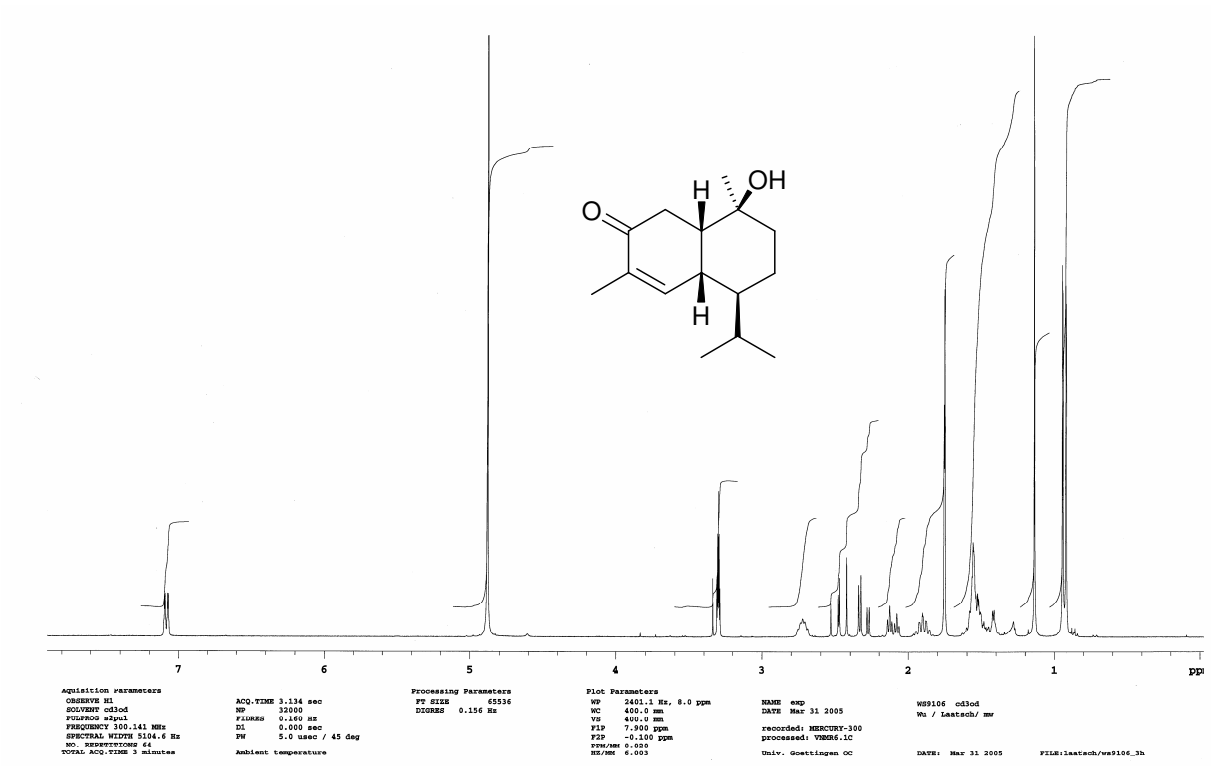
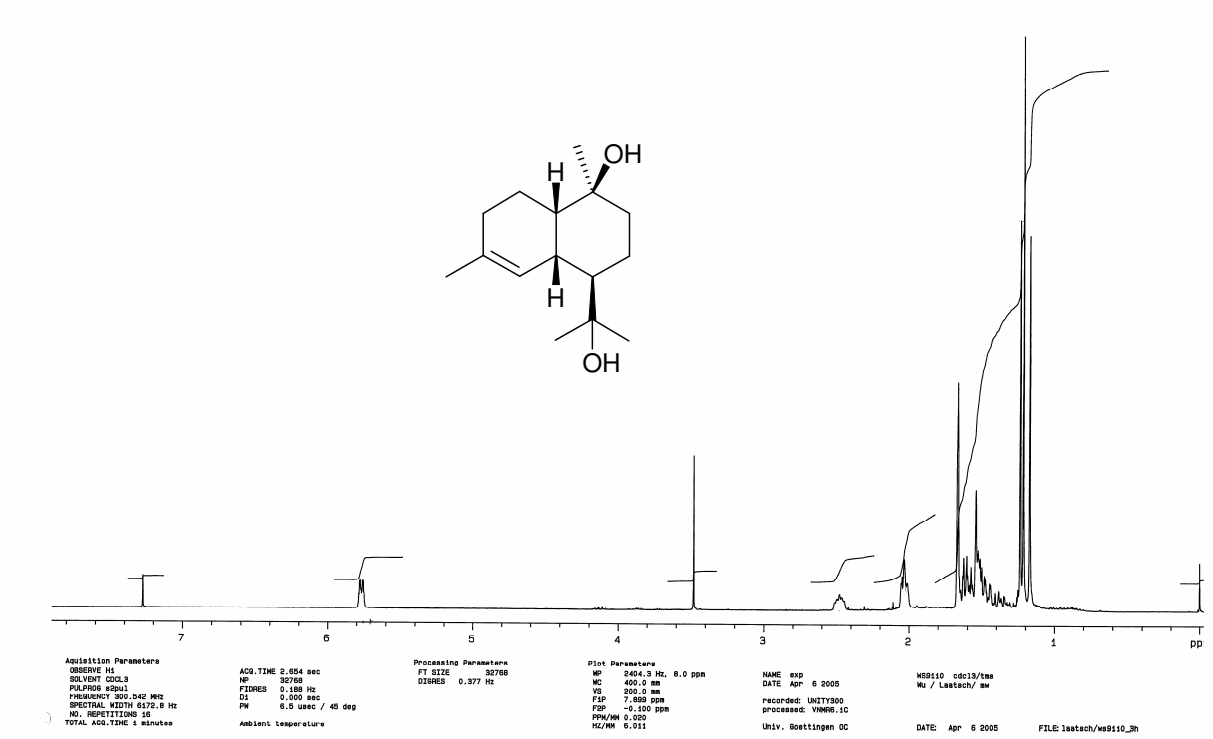
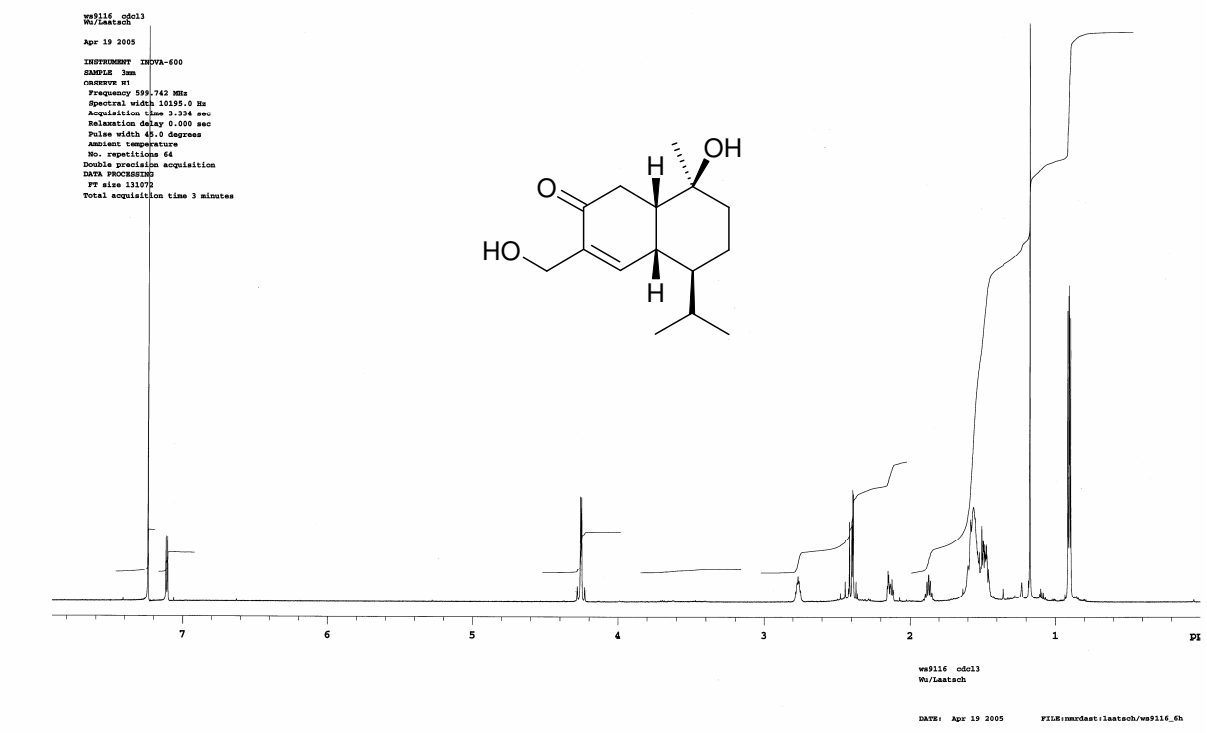
^{1}H NMR spectra of muurolanes isolated from *Streptomyces* sp. M491, and crystallographic data of 3-oxo-T-muurolol (3a)



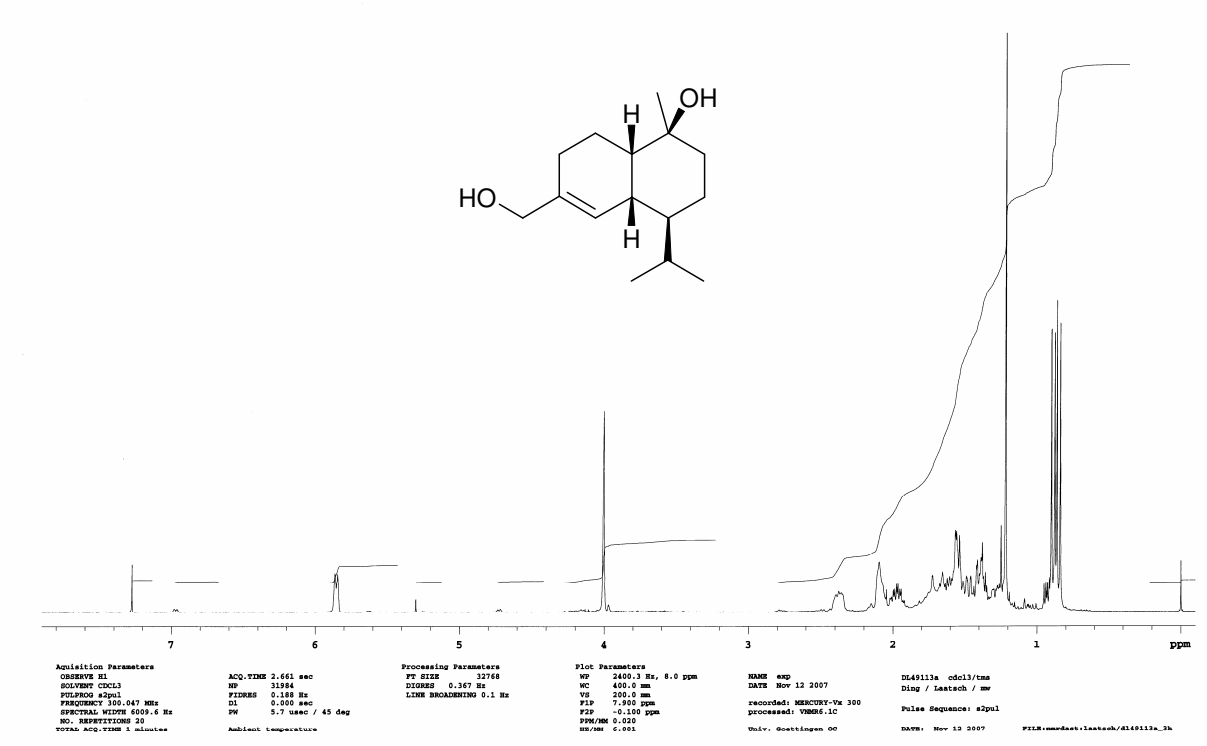
^{1}H NMR spectrum (300 MHz) of 3-oxo-T-muurolol (**3a**) in MeOH-d_4



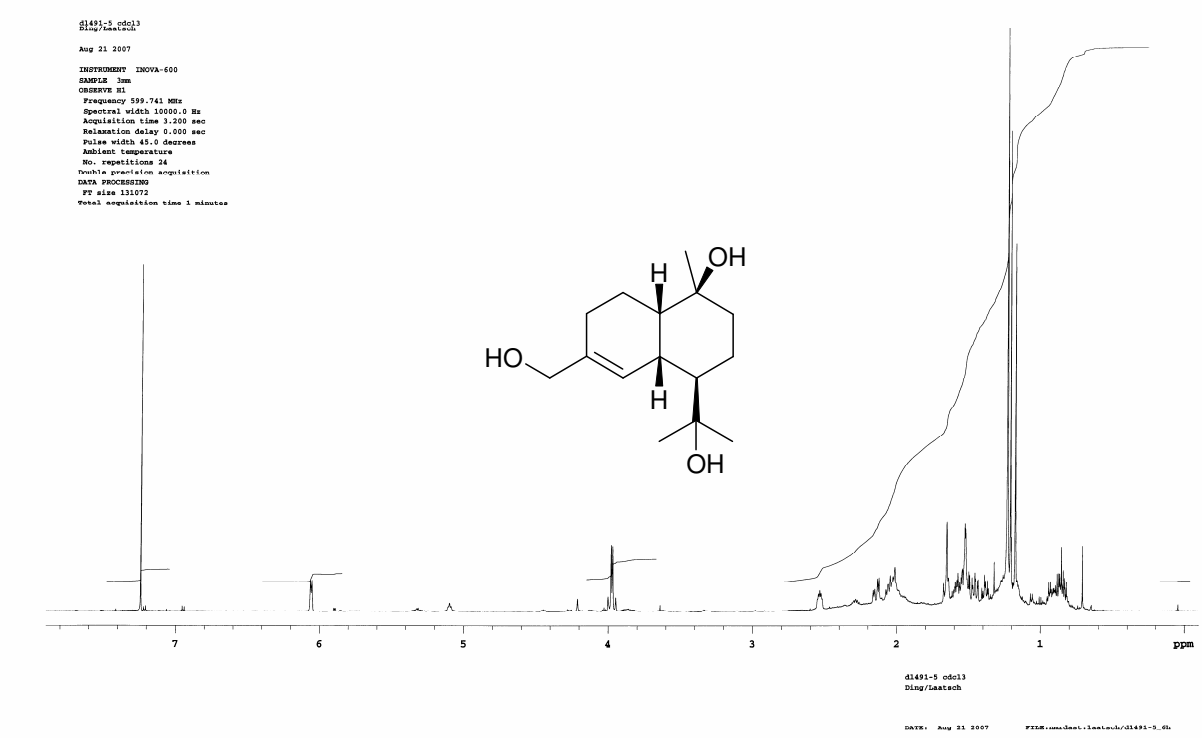
^{1}H NMR spectrum (300 MHz) of 11-hydroxy-T-muurolol (**3b**) in $CDCl_3$



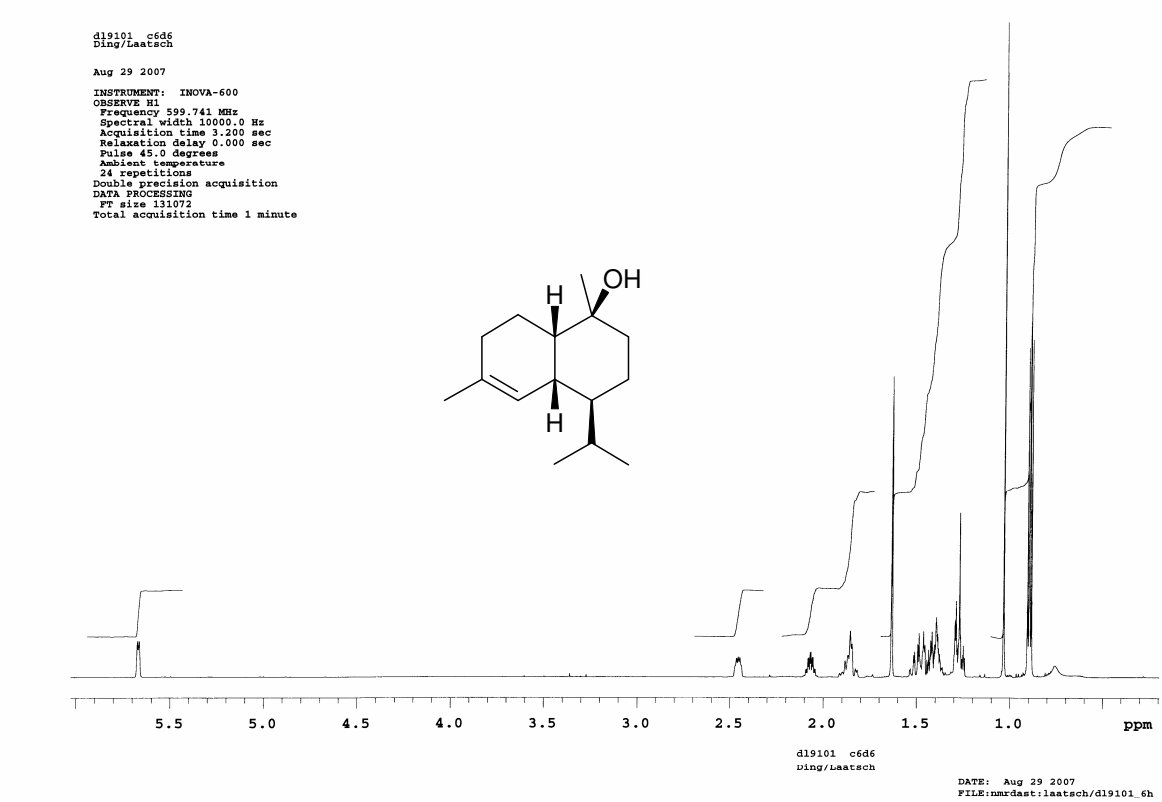
^{1}H NMR spectrum (600 MHz) of 3-oxo-15-hydroxy-T-muurolol (**3c**) in $CDCl_3$



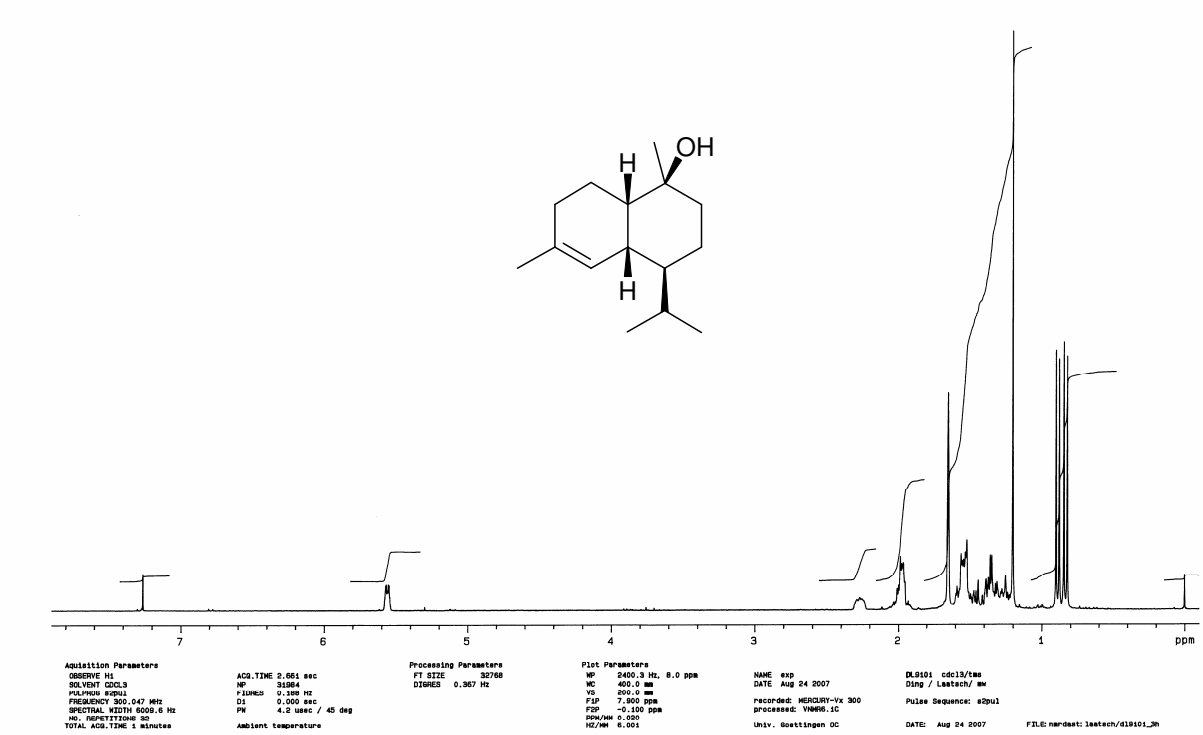
^{1}H NMR spectrum (300 MHz) of 15-hydroxy-T-muurolol (**3d**) in $CDCl_3$



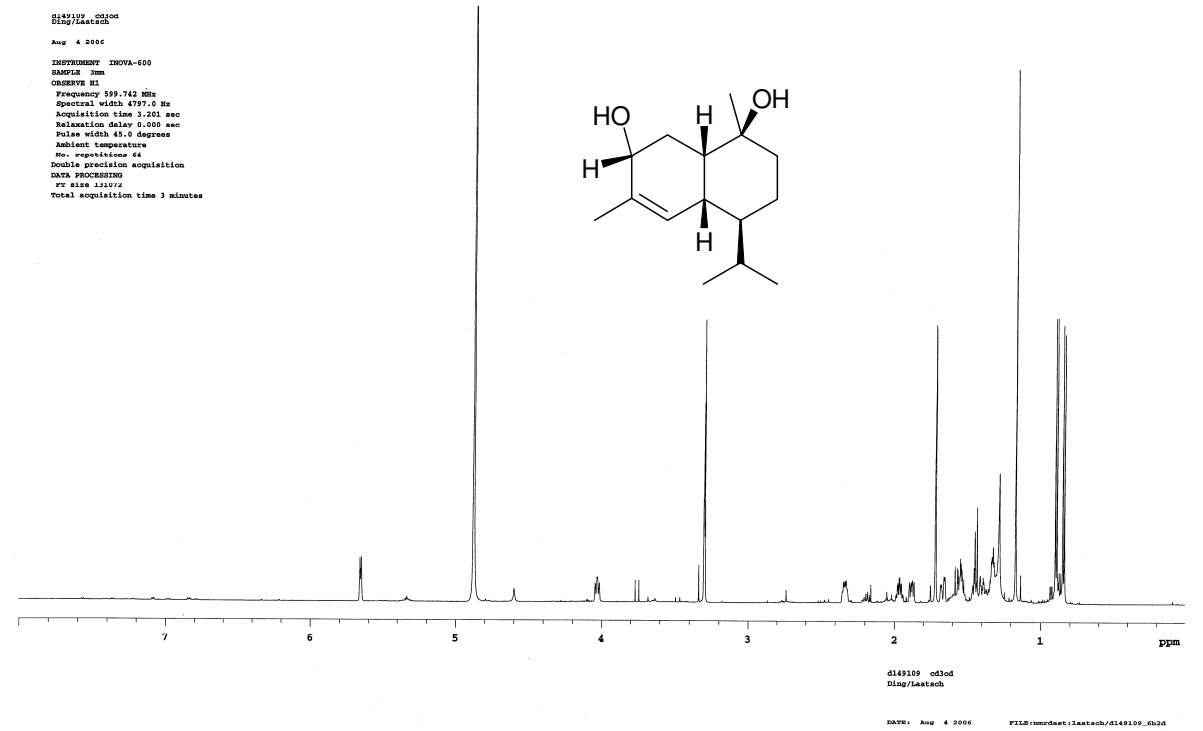
^{1}H NMR spectrum (600 MHz) of 11,15-dihydroxy-T-muurolol (**3e**) in $CDCl_3$



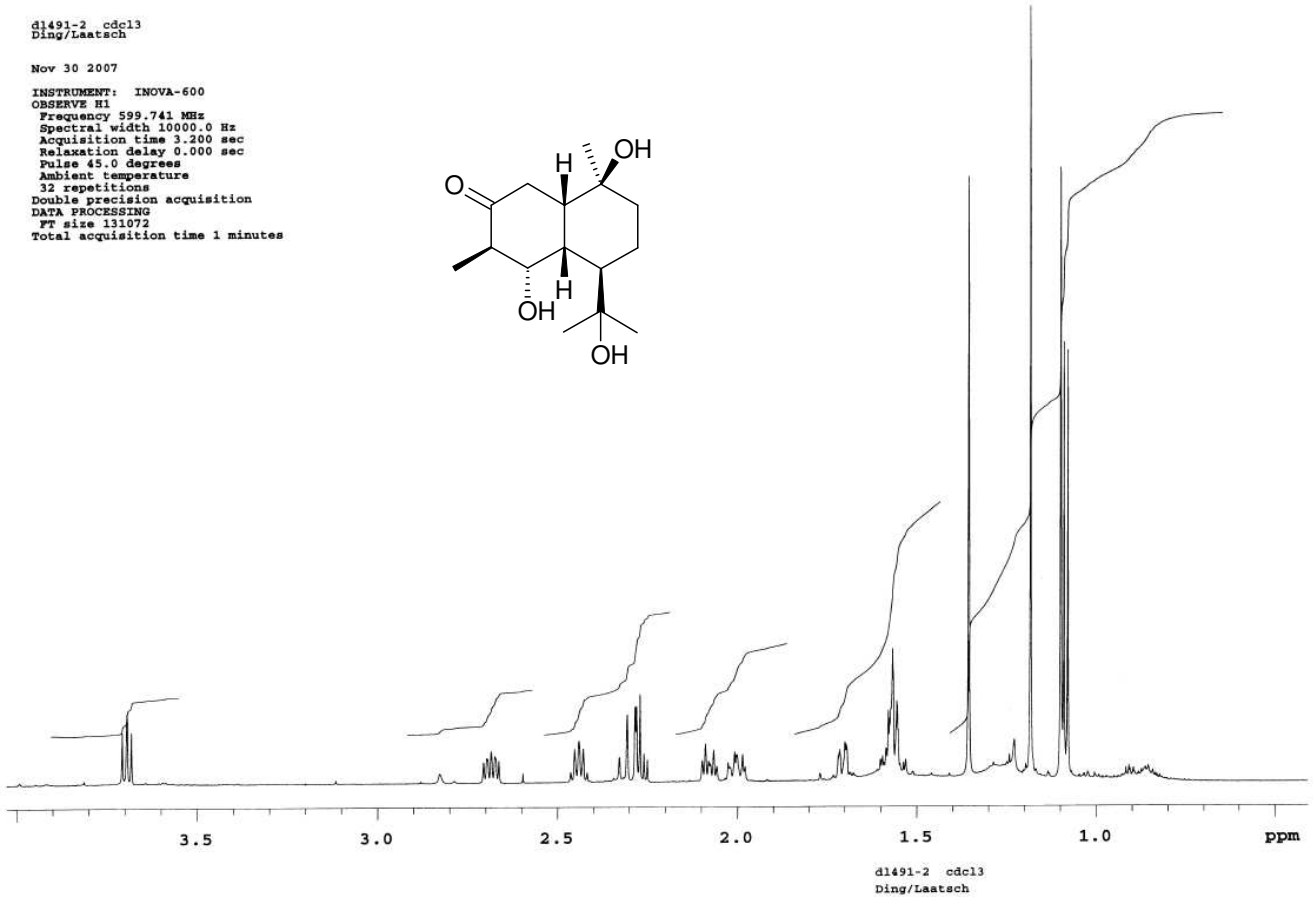
^{1}H NMR spectrum (600 MHz) of T-muurolol (**3f**) in C_6D_6



^{1}H NMR spectrum (300 MHz) of T-muurolol (**3f**) in $CDCl_3$



^{1}H NMR spectrum (600 MHz) of 3α-hydroxy-T-muurolol (**3g**) in MeOH-d_4



^{1}H NMR spectrum (300 MHz) of 3-oxo-5,15-dihydroxy-T-muurolol (**4**) in $CDCl_3$

Table 1. Crystal data and structure refinement for 3-oxo-T-muurolol (**3a**).

Identification code	terp1i	
Empirical formula	$C_{30}\ H_{50}\ O_5$	
Formula weight	490.70	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Tetragonal	
Space group	I 41	
Unit cell dimensions	a = 23.510(3) Å	a= 90°.
	b = 23.510(3) Å	b= 90°.
	c = 10.433(2) Å	g = 90°.
Volume	5766.5(16) $Å^3$	
Z	8	
Density (calculated)	1.130 Mg/m^3	
Absorption coefficient	0.590 mm^{-1}	
F(000)	2160	
Crystal size	0.07 x 0.05 x 0.05 mm^3	
Theta range for data collection	2.66 to 62.37°.	
Index ranges	-26<=h<=26, -26<=k<=26, -11<=l<=11	
Reflections collected	36187	
Independent reflections	4514 [R(int) = 0.0240]	
Completeness to theta = 62.37°	99.2 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9711 and 0.9599	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4514 / 1 / 338	
Goodness-of-fit on F^2	1.051	
Final R indices [I>2sigma(I)]	R1 = 0.0224, wR2 = 0.0576	
R indices (all data)	R1 = 0.0224, wR2 = 0.0577	
Absolute structure parameter	0.07(10)	
Largest diff. peak and hole	0.110 and -0.092 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 3-oxo-T-muurolol (**3a**). U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2670(1)	5502(1)	7356(1)	26(1)
C(2)	3099(1)	5666(1)	8396(1)	29(1)
O(3)	4114(1)	5648(1)	8544(1)	35(1)
C(3)	3677(1)	5406(1)	8180(1)	28(1)
C(4)	3713(1)	4849(1)	7524(1)	30(1)
C(5)	3233(1)	4608(1)	7081(1)	28(1)
C(6)	2643(1)	4848(1)	7209(1)	26(1)
C(7)	2307(1)	4559(1)	8317(1)	27(1)
C(8)	1709(1)	4821(1)	8389(1)	31(1)
C(9)	1733(1)	5464(1)	8572(1)	30(1)
O(10)	1805(1)	5678(1)	6301(1)	30(1)
C(10)	2077(1)	5770(1)	7531(1)	28(1)
C(11)	2305(1)	3902(1)	8225(1)	35(1)
C(12)	2105(1)	3629(1)	9462(2)	47(1)
C(13)	1976(1)	3676(1)	7076(1)	44(1)
C(14)	2104(1)	6408(1)	7794(1)	34(1)
C(15)	4292(1)	4589(1)	7375(1)	42(1)
C(1A)	4842(1)	7135(1)	5814(1)	26(1)
C(2A)	5177(1)	6804(1)	4803(1)	30(1)
O(3A)	6091(1)	6360(1)	4802(1)	39(1)
C(3A)	5802(1)	6768(1)	5134(1)	30(1)
C(4A)	6055(1)	7246(1)	5856(1)	31(1)
C(5A)	5731(1)	7687(1)	6228(1)	29(1)
C(6A)	5101(1)	7735(1)	6001(1)	26(1)
C(7A)	4960(1)	8146(1)	4878(1)	27(1)
C(8A)	4314(1)	8165(1)	4689(1)	29(1)
C(9A)	4063(1)	7574(1)	4466(1)	29(1)
O(10A)	3930(1)	7405(1)	6686(1)	30(1)
C(10A)	4195(1)	7164(1)	5560(1)	28(1)
C(11A)	5234(1)	8742(1)	5040(1)	33(1)
C(12A)	5209(1)	9082(1)	3797(1)	43(1)
C(13A)	4993(1)	9085(1)	6151(1)	40(1)
C(14A)	3938(1)	6577(1)	5310(1)	35(1)
C(15A)	6682(10)	7203(8)	6070(20)	40(1)
C(15B)	6687(10)	7219(8)	6170(20)	40(1)
O(1)	2766(1)	7629(1)	6378(1)	39(1)

Table 3. Bond lengths [Å] and angles [°] for 3-oxo-T-muurolol (**3a**).

C(1)-C(2)	1.5311(16)
C(1)-C(10)	1.5415(15)
C(1)-C(6)	1.5466(15)
C(2)-C(3)	1.5070(16)
O(3)-C(3)	1.2326(14)
C(3)-C(4)	1.4799(16)
C(4)-C(5)	1.3441(16)
C(4)-C(15)	1.5018(16)
C(5)-C(6)	1.5063(15)
C(6)-C(7)	1.5558(16)
C(7)-C(8)	1.5355(16)
C(7)-C(11)	1.5483(16)
C(8)-C(9)	1.5263(17)
C(9)-C(10)	1.5329(16)
O(10)-C(10)	1.4500(14)
C(10)-C(14)	1.5254(16)
C(11)-C(12)	1.5164(19)
C(11)-C(13)	1.523(2)
C(1A)-C(2A)	1.5302(15)
C(1A)-C(6A)	1.5462(15)
C(1A)-C(10A)	1.5468(16)
C(2A)-C(3A)	1.5118(17)
O(3A)-C(3A)	1.2256(14)
C(3A)-C(4A)	1.4764(17)
C(4A)-C(5A)	1.3435(17)
C(4A)-C(15A)	1.49(2)
C(4A)-C(15B)	1.52(2)
C(5A)-C(6A)	1.5061(16)
C(6A)-C(7A)	1.5552(16)
C(7A)-C(8A)	1.5315(15)
C(7A)-C(11A)	1.5507(16)
C(8A)-C(9A)	1.5284(16)
C(9A)-C(10A)	1.5264(16)
O(10A)-C(10A)	1.4457(14)
C(10A)-C(14A)	1.5276(17)
C(11A)-C(13A)	1.5223(18)
C(11A)-C(12A)	1.5234(19)
O(1)-H(1W)	0.876(17)
O(1)-H(2W)	0.895(20)
C(2)-C(1)-C(10)	114.20(9)
C(2)-C(1)-C(6)	110.34(9)

C(10)-C(1)-C(6)	112.33(9)
C(3)-C(2)-C(1)	112.75(9)
O(3)-C(3)-C(4)	120.29(10)
O(3)-C(3)-C(2)	121.15(10)
C(4)-C(3)-C(2)	118.57(9)
C(5)-C(4)-C(3)	119.10(10)
C(5)-C(4)-C(15)	123.53(11)
C(3)-C(4)-C(15)	117.36(10)
C(4)-C(5)-C(6)	125.71(10)
C(5)-C(6)-C(1)	110.12(9)
C(5)-C(6)-C(7)	111.71(9)
C(1)-C(6)-C(7)	112.43(9)
C(8)-C(7)-C(11)	113.58(9)
C(8)-C(7)-C(6)	109.06(9)
C(11)-C(7)-C(6)	113.01(9)
C(9)-C(8)-C(7)	111.65(9)
C(8)-C(9)-C(10)	113.33(9)
O(10)-C(10)-C(14)	108.95(9)
O(10)-C(10)-C(9)	108.91(9)
C(14)-C(10)-C(9)	110.86(9)
O(10)-C(10)-C(1)	103.49(8)
C(14)-C(10)-C(1)	112.60(9)
C(9)-C(10)-C(1)	111.69(9)
C(12)-C(11)-C(13)	111.38(11)
C(12)-C(11)-C(7)	111.74(11)
C(13)-C(11)-C(7)	113.52(10)
C(2A)-C(1A)-C(6A)	110.42(9)
C(2A)-C(1A)-C(10A)	114.26(9)
C(6A)-C(1A)-C(10A)	111.66(9)
C(3A)-C(2A)-C(1A)	111.79(9)
O(3A)-C(3A)-C(4A)	121.16(11)
O(3A)-C(3A)-C(2A)	121.15(11)
C(4A)-C(3A)-C(2A)	117.68(10)
C(5A)-C(4A)-C(3A)	120.50(10)
C(5A)-C(4A)-C(15A)	124.5(8)
C(3A)-C(4A)-C(15A)	115.0(8)
C(5A)-C(4A)-C(15B)	121.4(7)
C(3A)-C(4A)-C(15B)	118.1(7)
C(4A)-C(5A)-C(6A)	124.69(11)
C(5A)-C(6A)-C(1A)	109.80(9)
C(5A)-C(6A)-C(7A)	111.96(9)
C(1A)-C(6A)-C(7A)	112.85(9)
C(8A)-C(7A)-C(11A)	113.55(9)
C(8A)-C(7A)-C(6A)	109.00(9)

C(11A)-C(7A)-C(6A)	113.08(9)
C(9A)-C(8A)-C(7A)	112.12(9)
C(10A)-C(9A)-C(8A)	112.48(9)
O(10A)-C(10A)-C(9A)	105.80(9)
O(10A)-C(10A)-C(14A)	108.82(9)
C(9A)-C(10A)-C(14A)	111.27(9)
O(10A)-C(10A)-C(1A)	107.59(9)
C(9A)-C(10A)-C(1A)	110.78(9)
C(14A)-C(10A)-C(1A)	112.28(9)
C(13A)-C(11A)-C(12A)	110.83(10)
C(13A)-C(11A)-C(7A)	113.96(10)
C(12A)-C(11A)-C(7A)	111.41(10)
H(1W)-O(1)-H(2W)	101.3(16)

Symmetry transformations used to generate equivalent atoms

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$)for 3-oxo-T-muurolol (**3a**). The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	29(1)	28(1)	22(1)	-1(1)	1(1)	-1(1)
C(2)	31(1)	30(1)	26(1)	-3(1)	-2(1)	-2(1)
O(3)	31(1)	35(1)	38(1)	-2(1)	-6(1)	-6(1)
C(3)	31(1)	30(1)	23(1)	2(1)	-2(1)	-3(1)
C(4)	29(1)	33(1)	28(1)	-2(1)	0(1)	-1(1)
C(5)	31(1)	29(1)	25(1)	-3(1)	2(1)	-1(1)
C(6)	26(1)	28(1)	22(1)	-2(1)	-1(1)	-1(1)
C(7)	29(1)	31(1)	22(1)	0(1)	0(1)	-1(1)
C(8)	30(1)	36(1)	26(1)	2(1)	2(1)	-1(1)
C(9)	29(1)	38(1)	23(1)	-2(1)	1(1)	4(1)
O(10)	29(1)	38(1)	23(1)	-1(1)	-2(1)	4(1)
C(10)	31(1)	31(1)	22(1)	-1(1)	-3(1)	2(1)
C(11)	35(1)	32(1)	37(1)	3(1)	4(1)	-1(1)
C(12)	51(1)	39(1)	50(1)	13(1)	11(1)	4(1)
C(13)	49(1)	33(1)	49(1)	-5(1)	-1(1)	-7(1)
C(14)	37(1)	32(1)	34(1)	-1(1)	-2(1)	5(1)
C(15)	30(1)	46(1)	49(1)	-14(1)	-1(1)	2(1)
C(1A)	30(1)	27(1)	22(1)	-1(1)	1(1)	2(1)
C(2A)	37(1)	28(1)	26(1)	-3(1)	2(1)	2(1)
O(3A)	44(1)	34(1)	41(1)	-1(1)	6(1)	10(1)
C(3A)	36(1)	30(1)	24(1)	4(1)	6(1)	4(1)
C(4A)	32(1)	33(1)	28(1)	4(1)	1(1)	2(1)
C(5A)	33(1)	31(1)	24(1)	0(1)	-2(1)	-1(1)
C(6A)	30(1)	27(1)	22(1)	-2(1)	0(1)	3(1)
C(7A)	30(1)	28(1)	23(1)	-1(1)	1(1)	2(1)
C(8A)	30(1)	32(1)	26(1)	2(1)	0(1)	2(1)
C(9A)	31(1)	36(1)	22(1)	-1(1)	-2(1)	0(1)
O(10A)	31(1)	37(1)	21(1)	0(1)	1(1)	2(1)
C(10A)	31(1)	32(1)	22(1)	-2(1)	1(1)	-1(1)
C(11A)	30(1)	31(1)	36(1)	2(1)	-1(1)	0(1)
C(12A)	42(1)	41(1)	47(1)	10(1)	-2(1)	-8(1)
C(13A)	45(1)	29(1)	45(1)	-5(1)	-1(1)	-2(1)
C(14A)	36(1)	36(1)	32(1)	-2(1)	-1(1)	-4(1)
C(15A)	34(1)	45(1)	42(2)	-2(1)	-1(1)	5(1)
C(15B)	34(1)	45(1)	42(2)	-2(1)	-1(1)	5(1)
O(1)	35(1)	49(1)	32(1)	-8(1)	1(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 3-oxo-T-muurolol (**3a**).

	x	y	z	U(eq)
H(1)	2824	5654	6530	31
H(2A)	3137	6085	8419	35
H(2B)	2953	5541	9240	35
H(5)	3271	4254	6648	34
H(6)	2435	4765	6393	31
H(7)	2506	4661	9133	33
H(8A)	1499	4733	7590	37
H(8B)	1499	4648	9113	37
H(9A)	1904	5548	9418	36
H(9B)	1340	5616	8575	36
H(10)	1457	5759	6357	45
H(11)	2710	3783	8103	42
H(12A)	1707	3731	9617	70
H(12B)	2340	3765	10175	70
H(12C)	2139	3215	9394	70
H(13A)	2039	3265	6998	65
H(13B)	2108	3866	6295	65
H(13C)	1569	3750	7195	65
H(14A)	2347	6591	7152	51
H(14B)	2263	6473	8650	51
H(14C)	1721	6569	7748	51
H(15A)	4254	4203	7030	63
H(15B)	4481	4573	8212	63
H(15C)	4520	4820	6786	63
H(1A)	4893	6929	6643	31
H(21A)	5020	6415	4731	36
H(22A)	5133	6993	3960	36
H(5A)	5914	7991	6666	35
H(6A)	4928	7899	6795	32
H(7A)	5126	7975	4084	33
H(81A)	4136	8336	5457	35
H(82A)	4225	8411	3945	35
H(91A)	3645	7607	4369	35
H(92A)	4217	7417	3657	35
H(10A)	4015	7210	7332	44
H(11A)	5646	8678	5232	39
H(12D)	4812	9169	3591	65
H(12E)	5377	8858	3101	65

H(12F)	5422	9437	3901	65
H(13D)	5221	9430	6273	60
H(13E)	5003	8855	6934	60
H(13F)	4598	9191	5960	60
H(14D)	4056	6315	5990	52
H(14E)	4071	6435	4480	52
H(14F)	3522	6606	5301	52
H(15D)	6828	7571	6367	61
H(15E)	6870	7098	5267	61
H(15F)	6759	6912	6722	61
H(15G)	6775	7498	6839	61
H(15H)	6908	7306	5398	61
H(15I)	6783	6837	6473	61
H(1W)	3129(7)	7554(7)	6460(16)	50(4)
H(2W)	2705(7)	7529(8)	5560(20)	61(5)

Table 6. Torsion angles [°] for 3-oxo-T-muurolol (**3a**).

C(10)-C(1)-C(2)-C(3)	-178.84(9)
C(6)-C(1)-C(2)-C(3)	53.51(12)
C(1)-C(2)-C(3)-O(3)	149.87(11)
C(1)-C(2)-C(3)-C(4)	-30.50(14)
O(3)-C(3)-C(4)-C(5)	-176.73(11)
C(2)-C(3)-C(4)-C(5)	3.64(16)
O(3)-C(3)-C(4)-C(15)	2.32(17)
C(2)-C(3)-C(4)-C(15)	-177.31(11)
C(3)-C(4)-C(5)-C(6)	-1.32(17)
C(15)-C(4)-C(5)-C(6)	179.70(12)
C(4)-C(5)-C(6)-C(1)	25.15(15)
C(4)-C(5)-C(6)-C(7)	-100.54(13)
C(2)-C(1)-C(6)-C(5)	-49.64(12)
C(10)-C(1)-C(6)-C(5)	-178.31(9)
C(2)-C(1)-C(6)-C(7)	75.64(11)
C(10)-C(1)-C(6)-C(7)	-53.04(12)
C(5)-C(6)-C(7)-C(8)	-179.62(9)
C(1)-C(6)-C(7)-C(8)	55.98(12)
C(5)-C(6)-C(7)-C(11)	-52.27(12)
C(1)-C(6)-C(7)-C(11)	-176.67(9)
C(11)-C(7)-C(8)-C(9)	176.04(10)
C(6)-C(7)-C(8)-C(9)	-56.95(12)
C(7)-C(8)-C(9)-C(10)	56.10(13)
C(8)-C(9)-C(10)-O(10)	62.32(11)
C(8)-C(9)-C(10)-C(14)	-177.81(9)
C(8)-C(9)-C(10)-C(1)	-51.36(12)
C(2)-C(1)-C(10)-O(10)	165.83(9)
C(6)-C(1)-C(10)-O(10)	-67.55(11)
C(2)-C(1)-C(10)-C(14)	48.34(13)
C(6)-C(1)-C(10)-C(14)	174.96(9)
C(2)-C(1)-C(10)-C(9)	-77.16(12)
C(6)-C(1)-C(10)-C(9)	49.46(12)
C(8)-C(7)-C(11)-C(12)	-69.02(13)
C(6)-C(7)-C(11)-C(12)	166.06(10)
C(8)-C(7)-C(11)-C(13)	57.95(14)
C(6)-C(7)-C(11)-C(13)	-66.97(13)
C(6A)-C(1A)-C(2A)-C(3A)	55.81(12)
C(10A)-C(1A)-C(2A)-C(3A)	-177.33(9)
C(1A)-C(2A)-C(3A)-O(3A)	149.82(11)
C(1A)-C(2A)-C(3A)-C(4A)	-31.05(14)
O(3A)-C(3A)-C(4A)-C(5A)	-179.11(11)
C(2A)-C(3A)-C(4A)-C(5A)	1.76(16)

O(3A)-C(3A)-C(4A)-C(15A)	3.3(9)
C(2A)-C(3A)-C(4A)-C(15A)	-175.8(9)
O(3A)-C(3A)-C(4A)-C(15B)	0.4(9)
C(2A)-C(3A)-C(4A)-C(15B)	-178.7(9)
C(3A)-C(4A)-C(5A)-C(6A)	1.99(18)
C(15A)-C(4A)-C(5A)-C(6A)	179.3(9)
C(15B)-C(4A)-C(5A)-C(6A)	-177.5(9)
C(4A)-C(5A)-C(6A)-C(1A)	23.28(15)
C(4A)-C(5A)-C(6A)-C(7A)	-102.90(13)
C(2A)-C(1A)-C(6A)-C(5A)	-51.00(12)
C(10A)-C(1A)-C(6A)-C(5A)	-179.28(9)
C(2A)-C(1A)-C(6A)-C(7A)	74.66(11)
C(10A)-C(1A)-C(6A)-C(7A)	-53.62(12)
C(5A)-C(6A)-C(7A)-C(8A)	179.03(9)
C(1A)-C(6A)-C(7A)-C(8A)	54.54(12)
C(5A)-C(6A)-C(7A)-C(11A)	-53.66(12)
C(1A)-C(6A)-C(7A)-C(11A)	-178.16(9)
C(11A)-C(7A)-C(8A)-C(9A)	177.11(9)
C(6A)-C(7A)-C(8A)-C(9A)	-55.85(12)
C(7A)-C(8A)-C(9A)-C(10A)	57.52(13)
C(8A)-C(9A)-C(10A)-O(10A)	61.95(12)
C(8A)-C(9A)-C(10A)-C(14A)	179.98(9)
C(8A)-C(9A)-C(10A)-C(1A)	-54.36(12)
C(2A)-C(1A)-C(10A)-O(10A)	170.81(9)
C(6A)-C(1A)-C(10A)-O(10A)	-62.99(11)
C(2A)-C(1A)-C(10A)-C(9A)	-73.99(12)
C(6A)-C(1A)-C(10A)-C(9A)	52.21(12)
C(2A)-C(1A)-C(10A)-C(14A)	51.09(13)
C(6A)-C(1A)-C(10A)-C(14A)	177.30(9)
C(8A)-C(7A)-C(11A)-C(13A)	57.54(14)
C(6A)-C(7A)-C(11A)-C(13A)	-67.34(13)
C(8A)-C(7A)-C(11A)-C(12A)	-68.77(13)
C(6A)-C(7A)-C(11A)-C(12A)	166.35(10)

Symmetry transformations used to generate equivalent atoms

Table 7. Hydrogen bonds for 3-oxo-T-muurolol (**3a**) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(10)-H(10)...O(3)#1	0.84	1.95	2.7754(12)	165.8
O(10A)-H(10A)...O(10)#2	0.84	1.94	2.7777(11)	171.8
O(1)-H(1W)...O(10A)	0.876(17)	1.929(17)	2.8048(13)	178.2(17)
O(1)-H(2W)...O(1)#1	0.90(2)	1.90(2)	2.7870(7)	172.9(18)

Symmetry transformations used to generate equivalent atoms:

#1 y-1/2,-x+1,z-1/4 #2 -y+1,x+1/2,z+1/4