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Table 1. Crystallographic Experimental Details

A. Crystal Data	
formula	$C_{19}H_{26}O_2$
formula weight	286.40
crystal dimensions (mm)	0.74 × 0.17 × 0.15
crystal system	monoclinic
space group	$P2_1/c$ (No. 14)
unit cell parameters[a]	
a (Å)	8.1883 (13)
b (Å)	9.7259 (15)
c (Å)	20.835 (3)
β (deg)	98.414 (3)
V (Å^3)	1641.4 (4)
Z	4
ρ_{calcd} (g cm^{-3})	1.159
μ (mm^{-1})	0.073
B. Data Collection and Refinement Conditions	
diffractometer	Bruker PLATFORM/SMART 1000 CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.2°) (25 s exposures)
data collection 2θ limit (deg)	52.86
total data collected	8961 ($-10 \le h \le 9, -11 \le k \le 12, -26 \le l \le 26$)
independent reflections	3348 (R_{int} = 0.0339)
number of observed reflections (*NO*)	2547 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	direct methods (*SIR97*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	multi-scan (*SADABS*)
range of transmission factors	0.9891–0.9479
data/restraints/parameters	3348 [$F_o^2 \ge -3\sigma(F_o^2)$] / 0 / 190
goodness-of-fit (*S*)[e]	1.045 [$F_o^2 \ge -3\sigma(F_o^2)$]
final R indices[f]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0523
wR_2 [$F_o^2 \ge -3\sigma(F_o^2)$]	0.1415
largest difference peak and hole	0.326 and –0.321 e Å^{-3}

[a]Obtained from least-squares refinement of 3487 reflections with $4.63° < 2\theta < 52.72°$.

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[c]Altomare, A.; Burla, M. C.; Camalli, M.; Cascarano, G. L.; Giacovazzo, C.; Guagliardi, A.; Moliterni, A. G. G.; Polidori, G.; Spagna, R. *J. Appl. Cryst.* **1999**, *32*, 115–119.

[d]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted *R*-factors wR_2 and all goodnesses of fit *S* are based on F_o^2; conventional *R*-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. *R*-factors based on F_o^2 are statistically about twice as large as those based on F_o, and *R*-factors based on ALL data will be even larger.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n-p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0677P)^2 + 0.5026P]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

Atom	*x*	*y*	*z*	U_{eq}, Å^2
O1	-0.03030(18)	0.27495(16)	0.03481(6)	0.0515(4)*
O2	0.33531(18)	0.31052(14)	0.39210(6)	0.0423(4)*
C1	-0.0699(2)	0.33698(16)	0.14530(8)	0.0260(4)*
C2	0.0070(2)	0.34497(19)	0.08249(8)	0.0323(4)*
C3	0.1387(2)	0.4541(2)	0.09001(9)	0.0367(4)*
C4	0.1868(2)	0.47069(17)	0.16372(8)	0.0295(4)*
C5	0.2984(2)	0.35151(18)	0.18998(8)	0.0314(4)*
C6	0.3287(2)	0.34562(19)	0.26395(8)	0.0342(4)*
C7	0.1768(2)	0.37607(16)	0.29467(8)	0.0275(4)*
C8	0.1856(2)	0.35817(17)	0.36211(8)	0.0319(4)*
C9	0.0539(2)	0.38973(18)	0.39414(8)	0.0362(5)*
C10	-0.0904(2)	0.44352(18)	0.35979(9)	0.0344(4)*
C11	-0.0992(2)	0.46425(17)	0.29352(8)	0.0300(4)*
C12	0.0324(2)	0.42964(16)	0.26031(8)	0.0255(4)*
C13	0.0172(2)	0.45363(16)	0.18781(8)	0.0253(4)*
C14	-0.2605(2)	0.34009(17)	0.13389(8)	0.0280(4)*
C15	-0.3324(2)	0.20186(19)	0.11016(9)	0.0374(4)*
C16	-0.3282(2)	0.4551(2)	0.08787(10)	0.0408(5)*
C17	0.2664(3)	0.60923(19)	0.18247(10)	0.0417(5)*
C18	0.3642(3)	0.3098(2)	0.46109(9)	0.0535(6)*
C19	-0.2349(3)	0.4769(2)	0.39491(11)	0.0499(6)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

Atom1	Atom2	Distance	Atom1	Atom2	Distance
O1	C2	1.206(2)	C6	C7	1.509(2)
O2	C8	1.373(2)	C7	C8	1.407(2)
O2	C18	1.422(2)	C7	C12	1.391(2)
C1	C2	1.535(2)	C8	C9	1.383(3)
C1	C13	1.548(2)	C9	C10	1.391(3)
C1	C14	1.544(2)	C10	C11	1.387(2)
C2	C3	1.505(3)	C10	C19	1.515(3)
C3	C4	1.537(2)	C11	C12	1.404(2)
C4	C5	1.527(2)	C12	C13	1.516(2)
C4	C13	1.553(2)	C14	C15	1.521(2)
C4	C17	1.523(2)	C14	C16	1.524(2)
C5	C6	1.526(2)			

Table 4. Selected Interatomic Angles (deg)

Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C8	O2	C18	117.85(16)	C8	C7	C12	118.38(16)
C2	C1	C13	103.80(13)	O2	C8	C7	114.07(15)
C2	C1	C14	113.51(14)	O2	C8	C9	124.24(16)
C13	C1	C14	116.41(13)	C7	C8	C9	121.68(17)
O1	C2	C1	125.99(17)	C8	C9	C10	119.92(16)
O1	C2	C3	125.10(16)	C9	C10	C11	118.91(16)
C1	C2	C3	108.90(14)	C9	C10	C19	119.81(17)
C2	C3	C4	104.65(14)	C11	C10	C19	121.27(19)
C3	C4	C5	109.32(14)	C10	C11	C12	121.58(18)
C3	C4	C13	101.61(14)	C7	C12	C11	119.50(15)
C3	C4	C17	112.75(15)	C7	C12	C13	120.67(14)
C5	C4	C13	108.64(13)	C11	C12	C13	119.82(15)
C5	C4	C17	111.61(16)	C1	C13	C4	104.62(12)
C13	C4	C17	112.41(14)	C1	C13	C12	114.91(13)
C4	C5	C6	113.00(14)	C4	C13	C12	113.07(14)
C5	C6	C7	113.50(15)	C1	C14	C15	111.57(14)
C6	C7	C8	118.33(16)	C1	C14	C16	112.22(14)
C6	C7	C12	123.14(15)	C15	C14	C16	110.85(15)

Table 5. Torsional Angles (deg)

Atom1	Atom2	Atom3	Atom4	Angle
C18	O2	C8	C7	170.36(16)
C18	O2	C8	C9	-8.2(3)
C13	C1	C2	O1	-177.16(18)
C13	C1	C2	C3	3.38(18)
C14	C1	C2	O1	-49.8(2)
C14	C1	C2	C3	130.70(16)
C2	C1	C13	C4	-26.78(16)
C2	C1	C13	C12	-151.37(14)
C14	C1	C13	C4	-152.27(14)
C14	C1	C13	C12	83.13(18)
C2	C1	C14	C15	77.04(18)
C2	C1	C14	C16	-48.06(19)
C13	C1	C14	C15	-162.53(14)
C13	C1	C14	C16	72.37(19)
O1	C2	C3	C4	-157.93(18)
C1	C2	C3	C4	21.53(19)
C2	C3	C4	C5	77.37(17)
C2	C3	C4	C13	-37.32(17)
C2	C3	C4	C17	-157.86(15)
C3	C4	C5	C6	-170.20(15)
C13	C4	C5	C6	-60.12(19)
C17	C4	C5	C6	64.37(19)
C3	C4	C13	C1	39.75(16)
C3	C4	C13	C12	165.51(13)
C5	C4	C13	C1	-75.45(16)
C5	C4	C13	C12	50.31(18)
C17	C4	C13	C1	160.53(15)
C17	C4	C13	C12	-73.71(18)
C4	C5	C6	C7	40.7(2)
C5	C6	C7	C8	172.85(15)
C5	C6	C7	C12	-11.6(2)
C6	C7	C8	O2	-1.5(2)
C6	C7	C8	C9	177.11(16)
C12	C7	C8	O2	-177.27(15)
C12	C7	C8	C9	1.3(2)
C6	C7	C12	C11	-175.45(15)
C6	C7	C12	C13	3.3(2)
C8	C7	C12	C11	0.1(2)
C8	C7	C12	C13	178.85(14)
O2	C8	C9	C10	177.01(16)
C7	C8	C9	C10	-1.5(3)
C8	C9	C10	C11	0.1(3)
C8	C9	C10	C19	179.49(16)
C9	C10	C11	C12	1.3(3)
C19	C10	C11	C12	-178.04(16)
C10	C11	C12	C7	-1.4(2)
C10	C11	C12	C13	179.80(15)
C7	C12	C13	C1	96.70(18)
C7	C12	C13	C4	-23.3(2)
C11	C12	C13	C1	-84.55(18)
C11	C12	C13	C4	155.42(14)

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	0.0499(9)	0.0759(10)	0.0298(7)	-0.0205(7)	0.0092(7)	-0.0022(7)
O2	0.0489(9)	0.0517(8)	0.0229(7)	0.0038(6)	-0.0056(6)	0.0024(6)
C1	0.0276(9)	0.0291(8)	0.0207(8)	0.0001(6)	0.0021(7)	0.0050(7)
C2	0.0292(10)	0.0454(10)	0.0216(9)	-0.0008(7)	0.0013(7)	0.0089(8)
C3	0.0361(11)	0.0506(11)	0.0248(9)	0.0050(8)	0.0089(8)	0.0015(8)
C4	0.0297(10)	0.0346(9)	0.0246(9)	0.0012(7)	0.0048(7)	-0.0010(7)
C5	0.0261(9)	0.0399(10)	0.0291(9)	-0.0028(7)	0.0071(7)	0.0034(7)
C6	0.0303(10)	0.0425(10)	0.0286(9)	-0.0009(8)	-0.0003(8)	0.0054(8)
C7	0.0322(10)	0.0274(8)	0.0224(8)	-0.0033(6)	0.0025(7)	-0.0024(7)
C8	0.0389(11)	0.0298(9)	0.0251(9)	-0.0013(7)	-0.0015(8)	-0.0058(7)
C9	0.0522(12)	0.0373(10)	0.0204(8)	-0.0028(7)	0.0095(9)	-0.0124(8)
C10	0.0431(11)	0.0332(9)	0.0293(10)	-0.0079(7)	0.0131(8)	-0.0096(8)
C11	0.0314(10)	0.0303(9)	0.0292(9)	-0.0055(7)	0.0073(8)	-0.0019(7)
C12	0.0300(9)	0.0238(8)	0.0227(8)	-0.0039(6)	0.0035(7)	-0.0020(6)
C13	0.0262(9)	0.0277(8)	0.0220(8)	0.0006(6)	0.0032(7)	0.0037(6)
C14	0.0267(9)	0.0335(9)	0.0237(9)	-0.0012(7)	0.0028(7)	0.0008(7)
C15	0.0367(11)	0.0388(10)	0.0351(10)	-0.0025(8)	0.0000(8)	-0.0034(8)
C16	0.0323(11)	0.0422(11)	0.0458(12)	0.0070(9)	-0.0014(9)	0.0053(8)
C17	0.0408(12)	0.0411(11)	0.0439(12)	0.0036(9)	0.0089(9)	-0.0074(8)
C18	0.0705(16)	0.0608(14)	0.0239(10)	0.0019(9)	-0.0107(10)	-0.0010(11)
C19	0.0579(14)	0.0551(12)	0.0424(12)	-0.0109(10)	0.0264(11)	-0.0061(10)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H1	-0.0351	0.2477	0.1669	0.031
H3A	0.0954	0.5416	0.0699	0.044
H3B	0.2349	0.4248	0.0696	0.044
H5A	0.2473	0.2640	0.1731	0.038
H5B	0.4057	0.3606	0.1738	0.038
H6A	0.3698	0.2529	0.2776	0.041
H6B	0.4159	0.4127	0.2802	0.041
H9	0.0621	0.3747	0.4396	0.043
H11	-0.1967	0.5028	0.2700	0.036
H13	-0.0472	0.5402	0.1774	0.030
H14	-0.2975	0.3588	0.1767	0.034
H15A	-0.4532	0.2071	0.1039	0.045
H15B	-0.2953	0.1309	0.1424	0.045
H15C	-0.2951	0.1786	0.0689	0.045
H16A	-0.4492	0.4538	0.0824	0.049
H16B	-0.2913	0.4414	0.0457	0.049
H16C	-0.2879	0.5438	0.1060	0.049
H17A	0.2933	0.6150	0.2298	0.050
H17B	0.1895	0.6832	0.1667	0.050
H17C	0.3677	0.6187	0.1630	0.050
H18A	0.4747	0.2732	0.4761	0.064
H18B	0.2815	0.2518	0.4775	0.064
H18C	0.3563	0.4038	0.4773	0.064
H19A	-0.3305	0.4213	0.3769	0.060
H19B	-0.2624	0.5746	0.3894	0.060
H19C	-0.2053	0.4563	0.4412	0.060

University of Alberta Department of Chemistry
X-Ray Crystallography Laboratory

STRUCTURE REPORT

XCL Code: DLC0302
Date: 6 March 2003

Compound 57: 1-Isopropyl-6-methoxy-3a,8-dimethyl-2,3,3a,4,5,9b-hexahydro-1*H*-cyclopenta[*a*]naphthalene-4,5-diol
Formula: $C_{19}H_{28}O_3$

Supervisor: D. L. J. Clive **Crystallographer:** R. McDonald

Figure Legends

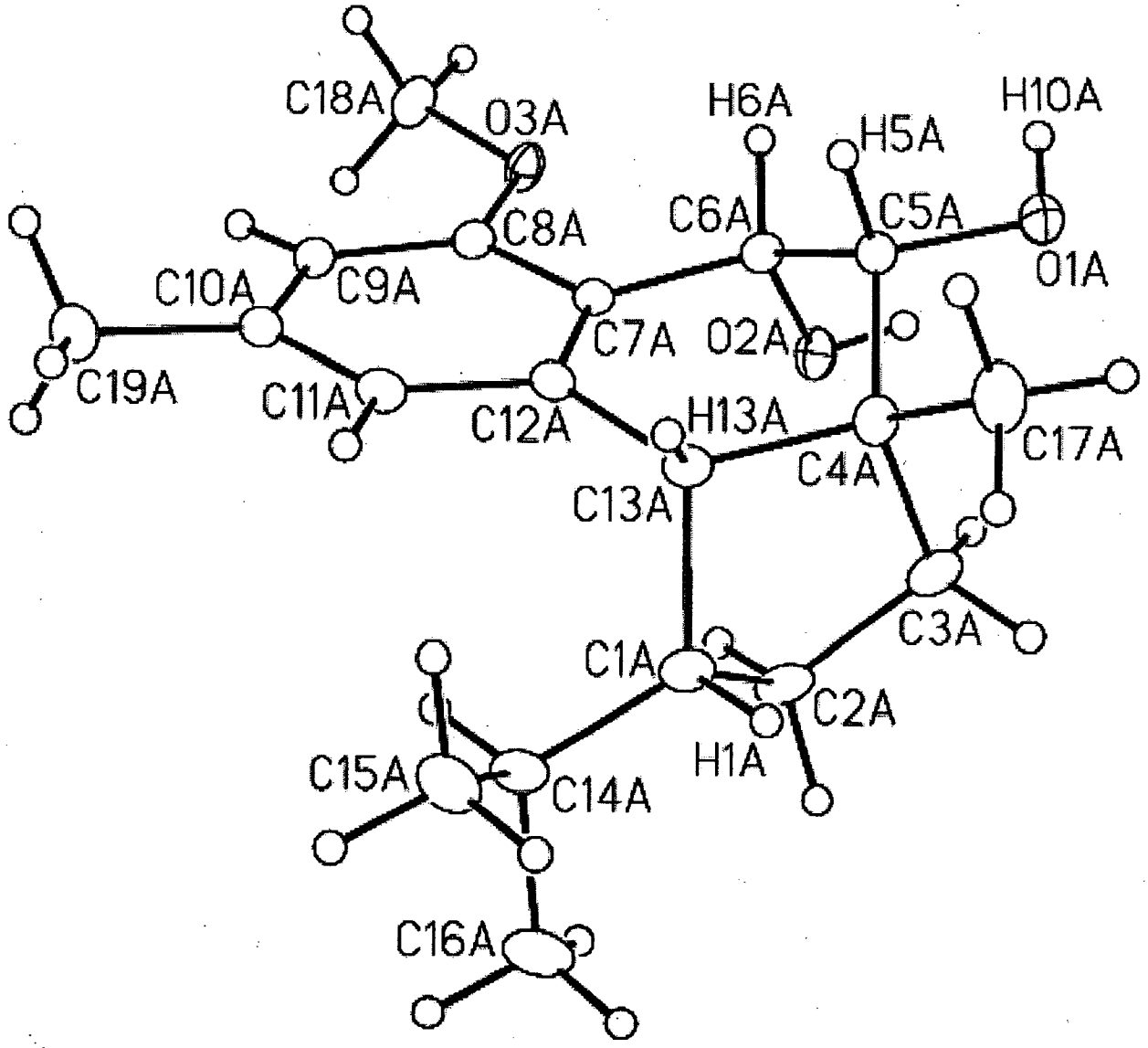
Figure 1. Perspective view of one of the two crystallographically-independent molecules of 1-isopropyl-6-methoxy-3a,8-dimethyl-2,3,3a,4,5,9b-hexahydro-1*H*-cyclopenta-[*a*]naphthalene-4,5-diol (molecule A) showing the atom labelling scheme. Non-hydrogen atoms are represented by Gaussian ellipsoids at the 20% probability level. Hydrogen atoms are shown with arbitrarily small thermal parameters.

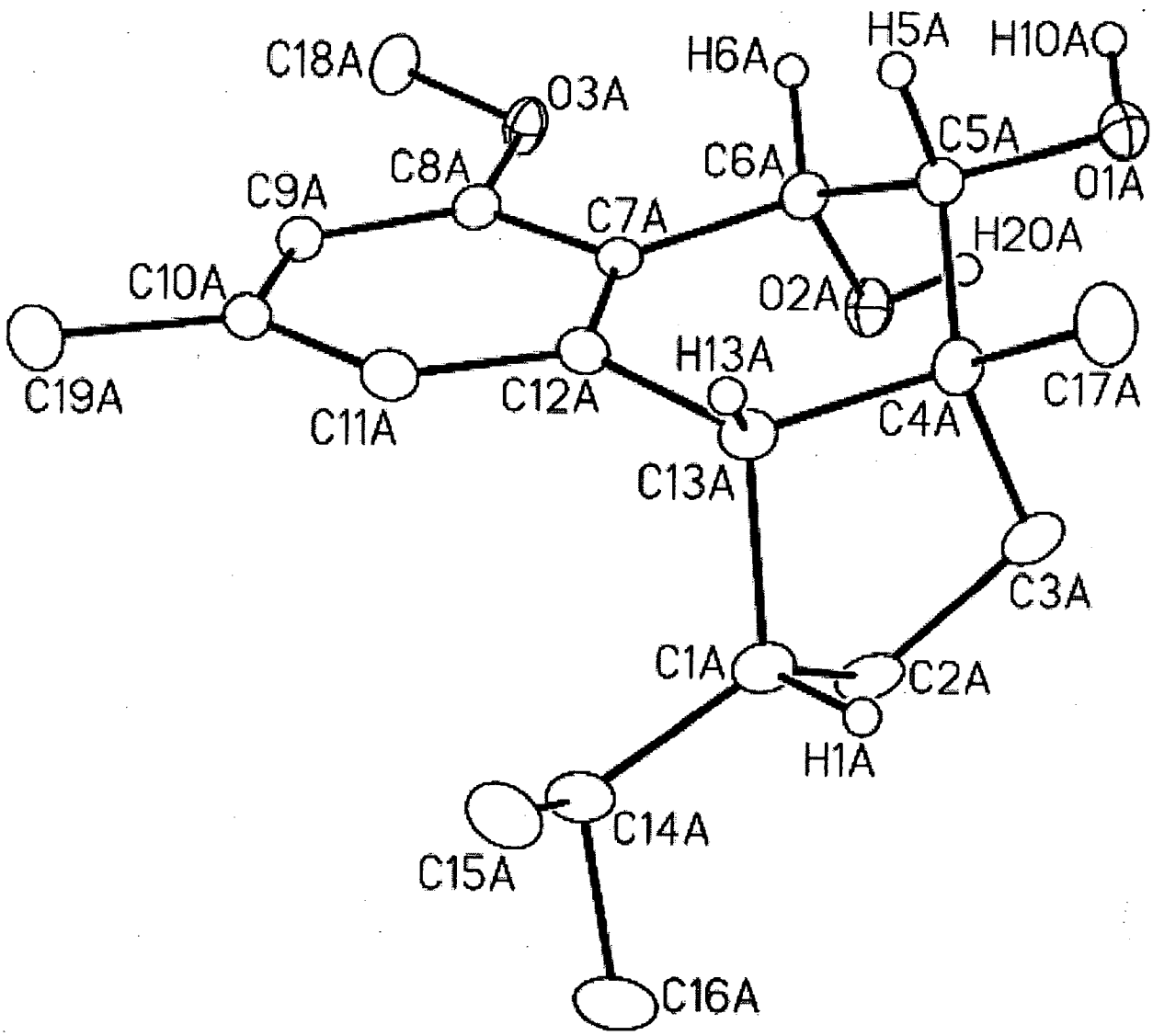
Figure 2. View of molecule A omitting all hydrogen atoms except for those of the hydroxyl groups and of the methine groups of the 2,3,3a,4,5,9b-hexahydro-1*H*-cyclopenta-[*a*]naphthalene ring.

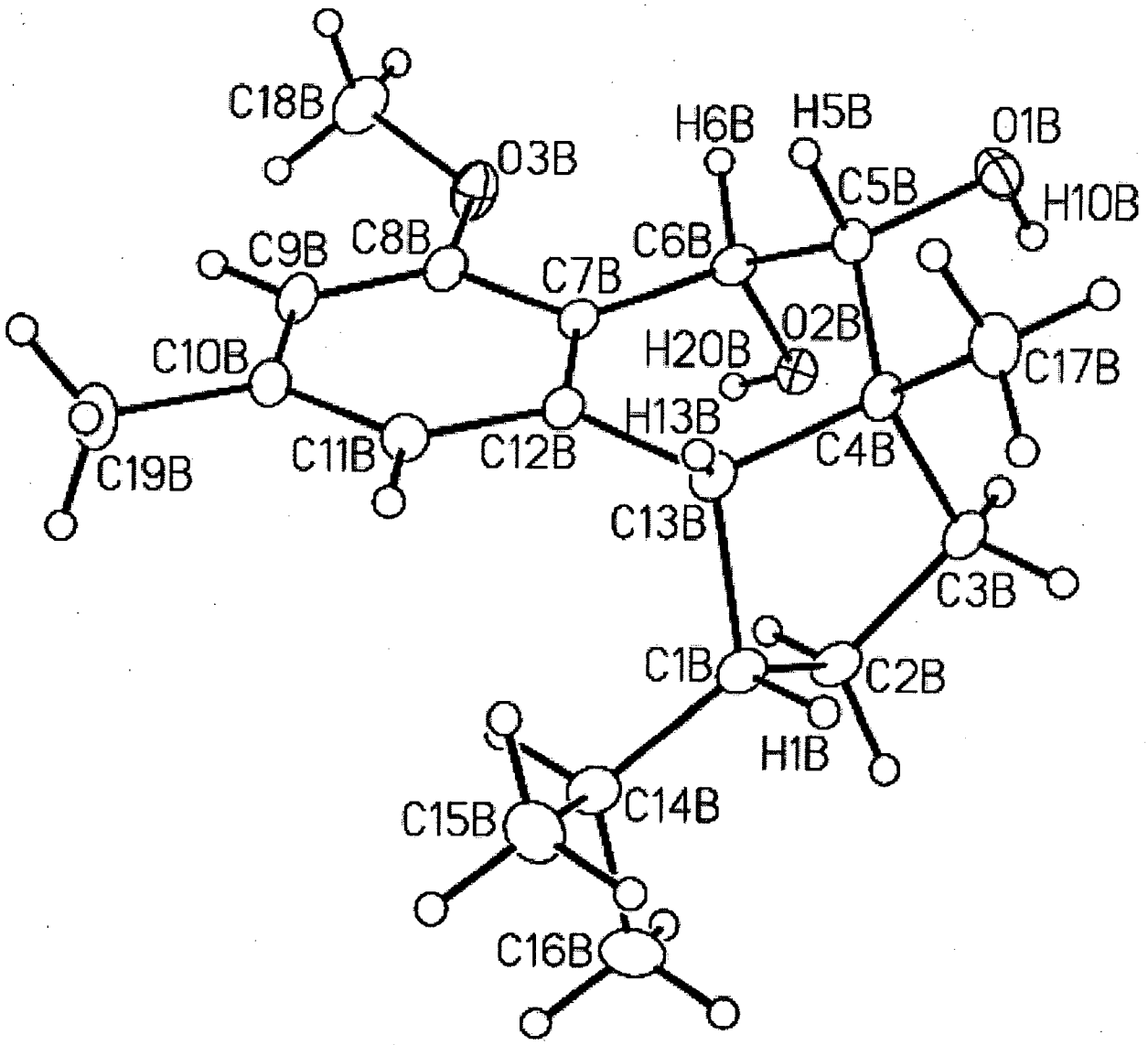
Figure 3. View of the second crystallographically-independent molecule of 1-isopropyl-6-methoxy-3a,8-dimethyl-2,3,3a,4,5,9b-hexahydro-1*H*-cyclopenta[*a*]naphthalene-4,5-diol (molecule B) showing all hydrogens.

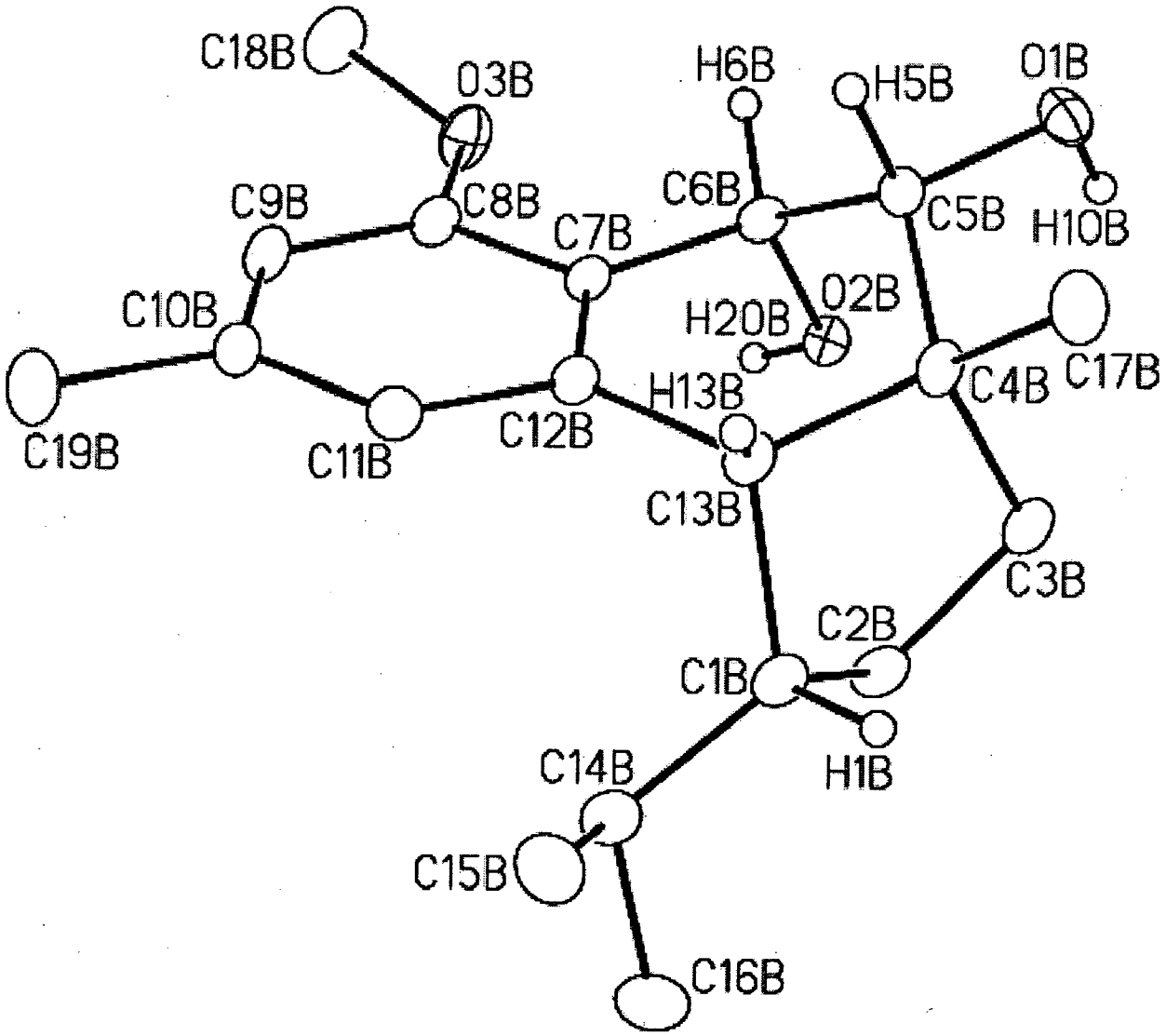
Figure 4. View of molecule B omitting all hydrogen atoms except for those of the hydroxyl groups and of the methine groups of the 2,3,3a,4,5,9b-hexahydro-1*H*-cyclopenta-[*a*]naphthalene ring.

Figure 5. Illustration of hydrogen-bonded interactions between adjacent molecules in the crystal lattice. The cyclic tetrameric unit consists of one molecule each of molecules A and B along with an inversion related molecule of each (indicated by primed atom labels) centered upon the crystallographic inversion center (0, 0, $^1/_2$).









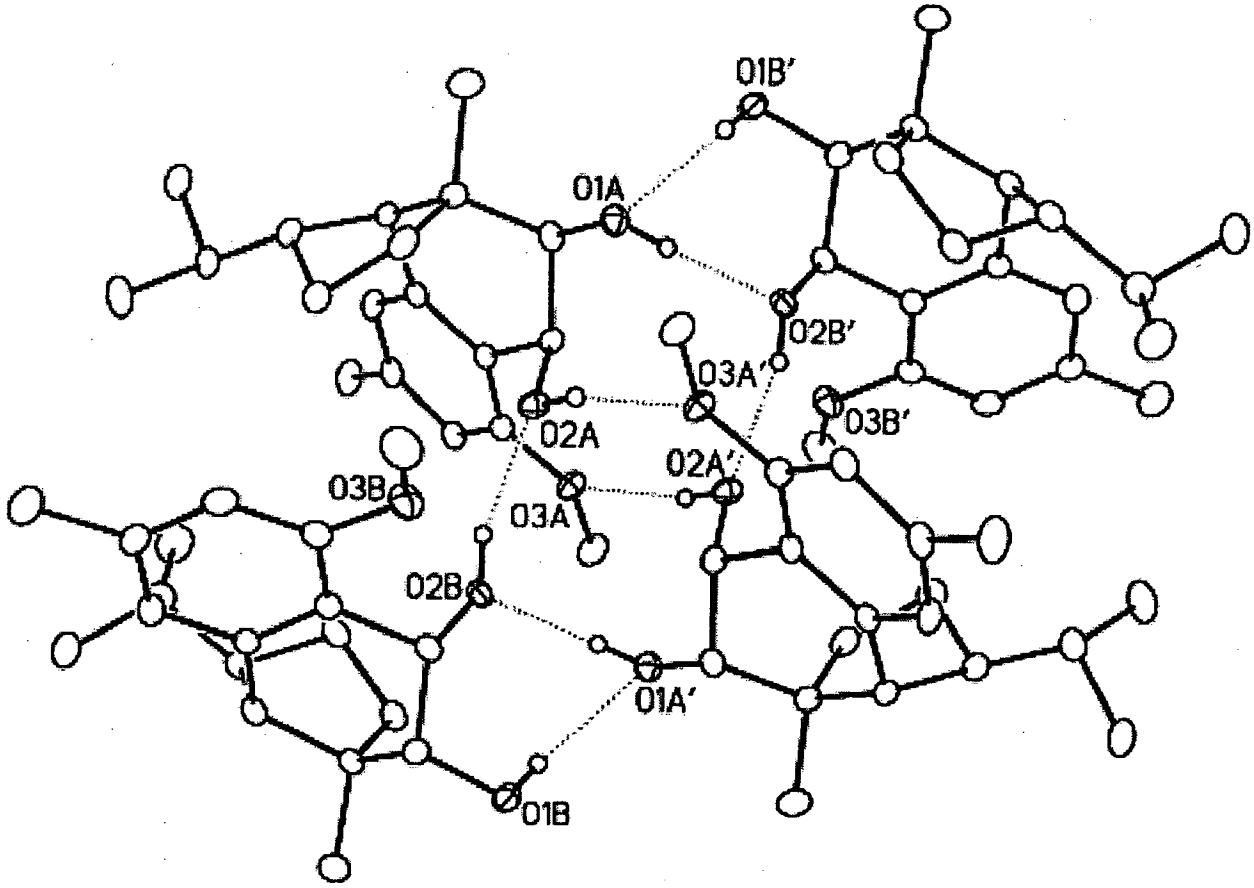


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Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

Table 1. Crystallographic Experimental Details

A. Crystal Data	
formula	$C_{19}H_{28}O_3$
formula weight	304.41
crystal dimensions (mm)	0.73 × 0.25 × 0.07
crystal system	triclinic
space group	$P\bar{1}$ (No. 2)
unit cell parameters[a]	
a (Å)	10.4105 (15)
b (Å)	11.5629 (17)
c (Å)	14.918 (2)
α (deg)	80.133 (3)
β (deg)	75.273 (3)
γ (deg)	81.329 (3)
V (Å^3)	1700.2 (4)
Z	4
ρ_{calcd} (g cm^{-3})	1.189
μ (mm^{-1})	0.078
B. Data Collection and Refinement Conditions	
diffractometer	Bruker PLATFORM/SMART 1000 CCD[b]
radiation (λ [Å])	graphite-monochromated Mo Kα (0.71073)
temperature (°C)	–80
scan type	ω scans (0.2°) (25 s exposures)
data collection 2θ limit (deg)	52.82
total data collected	9329 ($-10 \le h \le 13$, $-14 \le k \le 13$, $-18 \le l \le 18$)
independent reflections	6815 (R_{int} = 0.0527)
number of observed reflections (*NO*)	3458 [$F_o^2 \ge 2\sigma(F_o^2)$]
structure solution method	direct methods (*SHELXS–86*[c])
refinement method	full-matrix least-squares on F^2 (*SHELXL–93*[d])
absorption correction method	Gaussian integration (face-indexed)
range of transmission factors	0.9945–0.9449
data/restraints/parameters	6815 [$F_o^2 \ge -3\sigma(F_o^2)$] / 0 / 403
goodness-of-fit (S)[e]	0.942 [$F_o^2 \ge -3\sigma(F_o^2)$]
final R indices[f]	
R_1 [$F_o^2 \ge 2\sigma(F_o^2)$]	0.0598
wR_2 [$F_o^2 \ge -3\sigma(F_o^2)$]	0.1423
largest difference peak and hole	0.219 and –0.172 e Å^{-3}

[a]Obtained from least-squares refinement of 3158 reflections with $4.89° < 2\theta < 52.41°$.

(continued)

Table 1. Crystallographic Experimental Details (continued)

[b]Programs for diffractometer operation, data collection, data reduction and absorption correction were those supplied by Bruker.

[c]Sheldrick, G. M. *Acta Crystallogr.* **1990**, *A46*, 467–473.

[d]Sheldrick, G. M. *SHELXL-93*. Program for crystal structure determination. University of Göttingen, Germany, 1993. Refinement on F_o^2 for all reflections (all of these having $F_o^2 \geq -3\sigma(F_o^2)$). Weighted R-factors wR_2 and all goodnesses of fit S are based on F_o^2; conventional R-factors R_1 are based on F_o, with F_o set to zero for negative F_o^2. The observed criterion of $F_o^2 > 2\sigma(F_o^2)$ is used only for calculating R_1, and is not relevant to the choice of reflections for refinement. R-factors based on F_o^2 are statistically about twice as large as those based on F_o, and R-factors based on ALL data will be even larger.

[e]$S = [\Sigma w(F_o^2 - F_c^2)^2/(n - p)]^{1/2}$ (n = number of data; p = number of parameters varied; $w = [\sigma^2(F_o^2) + (0.0570P)^2]^{-1}$ where $P = [\mathrm{Max}(F_o^2, 0) + 2F_c^2]/3$).

[f]$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $wR_2 = [\Sigma w(F_o^2 - F_c^2)^2/\Sigma w(F_o^4)]^{1/2}$.

Table 2. Atomic Coordinates and Equivalent Isotropic Displacement Parameters

(a) Molecule A

Atom	*x*	*y*	*z*	U_{eq}, Å2
O1A	-0.12276(18)	-0.12296(15)	0.36620(12)	0.0375(5)*
O2A	0.05457(17)	0.02103(16)	0.39291(13)	0.0356(5)*
O3A	-0.07374(17)	0.23232(15)	0.49659(12)	0.0357(5)*
C1A	0.0471(3)	0.1597(3)	0.10813(18)	0.0368(7)*
C2A	0.1466(3)	0.0805(3)	0.15754(19)	0.0412(7)*
C3A	0.0755(3)	-0.0284(3)	0.19959(19)	0.0390(7)*
C4A	-0.0760(3)	0.0176(2)	0.22591(18)	0.0314(6)*
C5A	-0.1363(3)	0.0006(2)	0.33114(17)	0.0307(6)*
C6A	-0.0788(3)	0.0687(2)	0.38703(18)	0.0297(6)*
C7A	-0.0838(2)	0.1969(2)	0.34804(17)	0.0263(6)*
C8A	-0.0897(2)	0.2794(2)	0.40816(18)	0.0295(6)*
C9A	-0.1130(2)	0.3990(2)	0.37938(18)	0.0328(6)*
C10A	-0.1391(3)	0.4395(2)	0.29182(19)	0.0352(7)*
C11A	-0.1319(3)	0.3577(2)	0.23253(18)	0.0361(7)*
C12A	-0.1005(2)	0.2364(2)	0.25764(18)	0.0294(6)*
C13A	-0.0858(2)	0.1523(2)	0.18640(17)	0.0302(6)*
C14A	0.0842(3)	0.2795(3)	0.05636(19)	0.0444(8)*
C15A	-0.0096(3)	0.3318(3)	-0.0076(2)	0.0541(9)*
C16A	0.2279(3)	0.2720(3)	-0.0018(2)	0.0657(10)*
C17A	-0.1546(3)	-0.0479(3)	0.1802(2)	0.0486(8)*
C18A	-0.0910(3)	0.3137(2)	0.56197(19)	0.0425(8)*
C19A	-0.1774(3)	0.5692(3)	0.2643(2)	0.0534(9)*

(b) Molecule B

Atom	*x*	*y*	*z*	U_{eq}, Å2
O1B	0.41186(18)	0.14616(18)	0.55736(12)	0.0413(5)*
O2B	0.23244(16)	0.13749(16)	0.44190(12)	0.0320(4)*
O3B	0.37267(19)	-0.07112(17)	0.30881(13)	0.0479(5)*
C1B	0.3803(3)	0.4337(2)	0.28819(19)	0.0352(7)*
C2B	0.2532(2)	0.4035(2)	0.36012(19)	0.0356(7)*
C3B	0.3019(2)	0.3690(2)	0.44998(18)	0.0340(7)*
C4B	0.4435(2)	0.3013(2)	0.42158(18)	0.0299(6)*
C5B	0.4488(3)	0.1691(2)	0.45722(17)	0.0317(6)*
C6B	0.3733(2)	0.1006(2)	0.41327(18)	0.0308(6)*
C7B	0.4275(2)	0.1182(2)	0.30838(17)	0.0281(6)*
C8B	0.4330(3)	0.0254(2)	0.25797(19)	0.0338(7)*
C9B	0.4956(3)	0.0325(3)	0.1641(2)	0.0388(7)*
C10B	0.5556(3)	0.1319(3)	0.11821(18)	0.0364(7)*
C11B	0.5499(3)	0.2238(2)	0.16810(18)	0.0361(7)*

Table 2. Atomic Coordinates and Displacement Parameters (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
C12B	0.4844(2)	0.2197(2)	0.26266(17)	0.0289(6)*
C13B	0.4823(2)	0.3252(2)	0.31186(17)	0.0304(6)*
C14B	0.3725(3)	0.4715(3)	0.1865(2)	0.0425(7)*
C15B	0.4936(3)	0.5350(3)	0.1314(2)	0.0553(9)*
C16B	0.2443(3)	0.5528(3)	0.1788(2)	0.0639(10)*
C17B	0.5433(3)	0.3517(3)	0.4602(2)	0.0444(8)*
C18B	0.4063(3)	-0.1791(3)	0.2711(2)	0.0592(9)*
C19B	0.6280(3)	0.1400(3)	0.0160(2)	0.0584(9)*

Anisotropically-refined atoms are marked with an asterisk (*). The form of the anisotropic displacement parameter is: $\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$.

Table 3. Selected Interatomic Distances (Å)

(a) Molecule A

Atom1	Atom2	Distance
O1A	C5A	1.433(3)
O2A	C6A	1.433(3)
O3A	C8A	1.379(3)
O3A	C18A	1.429(3)
C1A	C2A	1.525(4)
C1A	C13A	1.569(4)
C1A	C14A	1.520(4)
C2A	C3A	1.516(4)
C3A	C4A	1.558(3)
C4A	C5A	1.526(3)
C4A	C13A	1.566(4)
C4A	C17A	1.535(3)
C5A	C6A	1.515(3)
C6A	C7A	1.494(3)
C7A	C8A	1.402(3)
C7A	C12A	1.393(3)
C8A	C9A	1.380(3)
C9A	C10A	1.390(3)
C10A	C11A	1.383(4)
C10A	C19A	1.507(4)
C11A	C12A	1.400(3)
C12A	C13A	1.525(3)
C14A	C15A	1.525(4)
C14A	C16A	1.525(4)

(b) Molecule B

Atom1	Atom2	Distance
O1B	C5B	1.433(3)
O2B	C6B	1.439(3)
O3B	C8B	1.376(3)
O3B	C18B	1.417(3)
C1B	C2B	1.521(4)
C1B	C13B	1.570(3)
C1B	C14B	1.524(4)
C2B	C3B	1.522(4)
C3B	C4B	1.554(3)
C4B	C5B	1.528(4)
C4B	C13B	1.569(3)
C4B	C17B	1.533(3)
C5B	C6B	1.517(3)
C6B	C7B	1.512(3)
C7B	C8B	1.399(3)
C7B	C12B	1.386(3)
C8B	C9B	1.381(4)
C9B	C10B	1.381(4)
C10B	C11B	1.384(4)
C10B	C19B	1.513(4)
C11B	C12B	1.398(3)
C12B	C13B	1.523(3)
C14B	C15B	1.532(4)
C14B	C16B	1.530(4)

(c) hydrogen-bonded interactions

Atom1	Atom2	Distance
O1A	H1OB'	2.19
O2A	H2OB	1.94
O3A	H2OA'	2.28
O2B	H1OA'	1.95

Primed atoms are related to unprimed ones via the crystallographic inversion center (0, 0, 1/2).

Table 4. Selected Interatomic Angles (deg)

(a) Molecule A				*(b) Molecule B*			
Atom1	Atom2	Atom3	Angle	Atom1	Atom2	Atom3	Angle
C8A	O3A	C18A	116.7(2)	C8B	O3B	C18B	117.8(2)
C2A	C1A	C13A	101.1(2)	C2B	C1B	C13B	101.5(2)
C2A	C1A	C14A	118.6(2)	C2B	C1B	C14B	118.2(2)
C13A	C1A	C14A	119.5(2)	C13B	C1B	C14B	118.7(2)
C1A	C2A	C3A	103.1(2)	C1B	C2B	C3B	102.3(2)
C2A	C3A	C4A	105.5(2)	C2B	C3B	C4B	105.7(2)
C3A	C4A	C5A	113.2(2)	C3B	C4B	C5B	113.3(2)
C3A	C4A	C13A	105.6(2)	C3B	C4B	C13B	105.0(2)
C3A	C4A	C17A	109.9(2)	C3B	C4B	C17B	110.2(2)
C5A	C4A	C13A	110.1(2)	C5B	C4B	C13B	111.4(2)
C5A	C4A	C17A	107.7(2)	C5B	C4B	C17B	107.7(2)
C13A	C4A	C17A	110.4(2)	C13B	C4B	C17B	109.3(2)
O1A	C5A	C4A	109.1(2)	O1B	C5B	C4B	111.9(2)
O1A	C5A	C6A	110.0(2)	O1B	C5B	C6B	111.5(2)
C4A	C5A	C6A	114.9(2)	C4B	C5B	C6B	114.9(2)
O2A	C6A	C5A	112.3(2)	O2B	C6B	C5B	109.1(2)
O2A	C6A	C7A	110.7(2)	O2B	C6B	C7B	113.6(2)
C5A	C6A	C7A	111.0(2)	C5B	C6B	C7B	108.8(2)
C6A	C7A	C8A	118.6(2)	C6B	C7B	C8B	119.2(2)
C6A	C7A	C12A	121.4(2)	C6B	C7B	C12B	121.2(2)
C8A	C7A	C12A	119.4(2)	C8B	C7B	C12B	119.2(2)
O3A	C8A	C7A	115.5(2)	O3B	C8B	C7B	115.2(2)
O3A	C8A	C9A	123.6(2)	O3B	C8B	C9B	123.7(2)
C7A	C8A	C9A	121.0(2)	C7B	C8B	C9B	121.2(3)
C8A	C9A	C10A	120.3(2)	C8B	C9B	C10B	120.2(2)
C9A	C10A	C11A	118.4(3)	C9B	C10B	C11B	118.6(3)
C9A	C10A	C19A	120.1(3)	C9B	C10B	C19B	121.0(3)
C11A	C10A	C19A	121.5(3)	C11B	C10B	C19B	120.4(3)
C10A	C11A	C12A	122.6(2)	C10B	C11B	C12B	122.2(3)
C7A	C12A	C11A	118.1(2)	C7B	C12B	C11B	118.6(2)
C7A	C12A	C13A	122.2(2)	C7B	C12B	C13B	122.8(2)
C11A	C12A	C13A	119.6(2)	C11B	C12B	C13B	118.6(2)
C1A	C13A	C4A	101.9(2)	C1B	C13B	C4B	103.25(19)
C1A	C13A	C12A	112.8(2)	C1B	C13B	C12B	114.3(2)
C4A	C13A	C12A	115.7(2)	C4B	C13B	C12B	115.4(2)
C1A	C14A	C15A	110.7(2)	C1B	C14B	C15B	110.5(2)
C1A	C14A	C16A	111.9(3)	C1B	C14B	C16B	111.6(2)
C15A	C14A	C16A	109.1(3)	C15B	C14B	C16B	109.2(3)

Table 5. Torsional Angles (deg)

(a) Molecule A

Atom1	Atom2	Atom3	Atom4	Angle
C18A	O3A	C8A	C7A	-174.7(2)
C18A	O3A	C8A	C9A	3.9(3)
C13A	C1A	C2A	C3A	47.5(2)
C14A	C1A	C2A	C3A	-179.8(2)
C2A	C1A	C13A	C4A	-42.6(2)
C2A	C1A	C13A	C12A	82.1(3)
C14A	C1A	C13A	C4A	-174.7(2)
C14A	C1A	C13A	C12A	-50.1(3)
C2A	C1A	C14A	C15A	165.9(2)
C2A	C1A	C14A	C16A	44.1(3)
C13A	C1A	C14A	C15A	-70.0(3)
C13A	C1A	C14A	C16A	168.2(2)
C1A	C2A	C3A	C4A	-33.5(3)
C2A	C3A	C4A	C5A	-114.2(2)
C2A	C3A	C4A	C13A	6.3(3)
C2A	C3A	C4A	C17A	125.4(2)
C3A	C4A	C5A	O1A	-60.4(3)
C3A	C4A	C5A	C6A	63.6(3)
C13A	C4A	C5A	O1A	-178.33(19)
C13A	C4A	C5A	C6A	-54.3(3)
C17A	C4A	C5A	O1A	61.2(3)
C17A	C4A	C5A	C6A	-174.7(2)
C3A	C4A	C13A	C1A	22.3(2)
C3A	C4A	C13A	C12A	-100.5(2)
C5A	C4A	C13A	C1A	144.7(2)
C5A	C4A	C13A	C12A	22.0(3)
C17A	C4A	C13A	C1A	-96.5(2)
C17A	C4A	C13A	C12A	140.8(2)
O1A	C5A	C6A	O2A	51.9(3)
O1A	C5A	C6A	C7A	176.4(2)
C4A	C5A	C6A	O2A	-71.7(3)
C4A	C5A	C6A	C7A	52.8(3)
O2A	C6A	C7A	C8A	-81.1(3)
O2A	C6A	C7A	C12A	107.5(2)
C5A	C6A	C7A	C8A	153.5(2)
C5A	C6A	C7A	C12A	-18.0(3)
C6A	C7A	C8A	O3A	7.6(3)
C6A	C7A	C8A	C9A	-171.0(2)
C12A	C7A	C8A	O3A	179.3(2)

(b) Molecule B

Atom1	Atom2	Atom3	Atom4	Angle
C18B	O3B	C8B	C7B	-162.4(2)
C18B	O3B	C8B	C9B	17.4(4)
C13B	C1B	C2B	C3B	47.4(2)
C14B	C1B	C2B	C3B	179.1(2)
C2B	C1B	C13B	C4B	-39.3(2)
C2B	C1B	C13B	C12B	86.8(2)
C14B	C1B	C13B	C4B	-170.7(2)
C14B	C1B	C13B	C12B	-44.5(3)
C2B	C1B	C14B	C15B	161.9(2)
C2B	C1B	C14B	C16B	40.2(4)
C13B	C1B	C14B	C15B	-74.6(3)
C13B	C1B	C14B	C16B	163.6(2)
C1B	C2B	C3B	C4B	-37.4(3)
C2B	C3B	C4B	C5B	-109.3(2)
C2B	C3B	C4B	C13B	12.4(3)
C2B	C3B	C4B	C17B	129.9(2)
C3B	C4B	C5B	O1B	-61.2(3)
C3B	C4B	C5B	C6B	67.4(3)
C13B	C4B	C5B	O1B	-179.23(18)
C13B	C4B	C5B	C6B	-50.7(3)
C17B	C4B	C5B	O1B	61.0(3)
C17B	C4B	C5B	C6B	-170.4(2)
C3B	C4B	C13B	C1B	16.4(2)
C3B	C4B	C13B	C12B	-109.0(2)
C5B	C4B	C13B	C1B	139.4(2)
C5B	C4B	C13B	C12B	14.0(3)
C17B	C4B	C13B	C1B	-101.7(2)
C17B	C4B	C13B	C12B	132.8(2)
O1B	C5B	C6B	O2B	61.6(3)
O1B	C5B	C6B	C7B	-173.9(2)
C4B	C5B	C6B	O2B	-67.1(3)
C4B	C5B	C6B	C7B	57.3(3)
O2B	C6B	C7B	C8B	-92.7(3)
O2B	C6B	C7B	C12B	94.1(3)
C5B	C6B	C7B	C8B	145.6(2)
C5B	C6B	C7B	C12B	-27.6(3)
C6B	C7B	C8B	O3B	7.3(3)
C6B	C7B	C8B	C9B	-172.5(2)
C12B	C7B	C8B	O3B	-179.3(2)

Table 5. Torsional Angles (continued)

(a) Molecule A

Atom1	Atom2	Atom3	Atom4	Angle
C12A	C7A	C8A	C9A	0.6(4)
C6A	C7A	C12A	C11A	167.0(2)
C6A	C7A	C12A	C13A	-12.9(4)
C8A	C7A	C12A	C11A	-4.4(3)
C8A	C7A	C12A	C13A	175.7(2)
O3A	C8A	C9A	C10A	-174.6(2)
C7A	C8A	C9A	C10A	3.9(4)
C8A	C9A	C10A	C11A	-4.4(4)
C8A	C9A	C10A	C19A	174.2(3)
C9A	C10A	C11A	C12A	0.5(4)
C19A	C10A	C11A	C12A	-178.1(3)
C10A	C11A	C12A	C7A	3.9(4)
C10A	C11A	C12A	C13A	-176.2(2)
C7A	C12A	C13A	C1A	-106.4(3)
C7A	C12A	C13A	C4A	10.4(3)
C11A	C12A	C13A	C1A	73.7(3)
C11A	C12A	C13A	C4A	-169.5(2)

(b) Molecule B

Atom1	Atom2	Atom3	Atom4	Angle
C12B	C7B	C8B	C9B	0.9(4)
C6B	C7B	C12B	C11B	170.9(2)
C6B	C7B	C12B	C13B	-7.1(4)
C8B	C7B	C12B	C11B	-2.3(3)
C8B	C7B	C12B	C13B	179.6(2)
O3B	C8B	C9B	C10B	-179.1(2)
C7B	C8B	C9B	C10B	0.7(4)
C8B	C9B	C10B	C11B	-0.8(4)
C8B	C9B	C10B	C19B	178.3(3)
C9B	C10B	C11B	C12B	-0.7(4)
C19B	C10B	C11B	C12B	-179.8(3)
C10B	C11B	C12B	C7B	2.2(4)
C10B	C11B	C12B	C13B	-179.6(2)
C7B	C12B	C13B	C1B	-105.2(3)
C7B	C12B	C13B	C4B	14.3(3)
C11B	C12B	C13B	C1B	76.8(3)
C11B	C12B	C13B	C4B	-163.7(2)

Table 6. Anisotropic Displacement Parameters (U_{ij}, Å^2)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1A	0.0425(12)	0.0293(12)	0.0387(11)	-0.0038(9)	-0.0067(10)	-0.0043(9)
O2A	0.0394(12)	0.0305(12)	0.0400(12)	-0.0043(9)	-0.0185(9)	0.0019(8)
O3A	0.0482(12)	0.0302(11)	0.0295(11)	-0.0098(9)	-0.0116(9)	0.0040(9)
C1A	0.0384(17)	0.048(2)	0.0240(15)	-0.0093(13)	-0.0032(13)	-0.0067(14)
C2A	0.0283(16)	0.059(2)	0.0330(17)	-0.0130(15)	-0.0001(13)	0.0010(14)
C3A	0.0379(17)	0.047(2)	0.0280(16)	-0.0115(14)	-0.0046(13)	0.0076(14)
C4A	0.0334(16)	0.0305(17)	0.0325(16)	-0.0069(12)	-0.0110(12)	-0.0023(12)
C5A	0.0280(15)	0.0252(16)	0.0354(16)	-0.0024(12)	-0.0032(12)	-0.0014(11)
C6A	0.0299(15)	0.0310(17)	0.0265(15)	-0.0042(12)	-0.0055(12)	-0.0003(12)
C7A	0.0218(14)	0.0269(16)	0.0273(15)	-0.0018(12)	-0.0023(11)	-0.0021(11)
C8A	0.0237(15)	0.0323(17)	0.0301(16)	-0.0037(13)	-0.0040(12)	-0.0004(11)
C9A	0.0309(16)	0.0293(17)	0.0352(17)	-0.0071(13)	-0.0002(12)	-0.0046(12)
C10A	0.0315(16)	0.0289(17)	0.0371(17)	0.0005(13)	0.0021(13)	-0.0017(12)
C11A	0.0371(17)	0.0375(18)	0.0275(16)	0.0042(13)	-0.0035(13)	-0.0022(13)
C12A	0.0237(15)	0.0311(17)	0.0317(16)	-0.0021(12)	-0.0028(12)	-0.0064(11)
C13A	0.0280(15)	0.0369(17)	0.0254(14)	-0.0037(12)	-0.0065(12)	-0.0035(12)
C14A	0.048(2)	0.052(2)	0.0302(16)	-0.0080(15)	0.0046(14)	-0.0165(15)
C15A	0.071(2)	0.053(2)	0.0341(18)	0.0060(15)	-0.0061(16)	-0.0166(17)
C16A	0.060(2)	0.079(3)	0.049(2)	-0.0045(19)	0.0129(17)	-0.0274(19)
C17A	0.062(2)	0.042(2)	0.052(2)	-0.0096(15)	-0.0276(16)	-0.0075(15)
C18A	0.055(2)	0.0392(19)	0.0344(17)	-0.0141(14)	-0.0123(14)	0.0046(14)
C19A	0.070(2)	0.036(2)	0.0437(19)	0.0032(15)	-0.0026(16)	-0.0014(16)
O1B	0.0373(12)	0.0562(14)	0.0300(11)	-0.0043(10)	-0.0087(9)	-0.0046(10)
O2B	0.0272(10)	0.0342(12)	0.0345(11)	-0.0071(8)	-0.0028(8)	-0.0084(8)
O3B	0.0576(14)	0.0334(13)	0.0537(13)	-0.0153(10)	-0.0029(10)	-0.0152(10)
C1B	0.0376(17)	0.0242(16)	0.0441(18)	-0.0062(13)	-0.0087(13)	-0.0040(12)
C2B	0.0259(15)	0.0298(17)	0.0486(18)	-0.0075(13)	-0.0050(13)	0.0003(12)
C3B	0.0311(16)	0.0295(17)	0.0387(17)	-0.0112(13)	0.0012(13)	-0.0039(12)
C4B	0.0269(15)	0.0303(17)	0.0340(16)	-0.0111(12)	-0.0038(12)	-0.0057(11)
C5B	0.0263(15)	0.0398(18)	0.0283(15)	-0.0095(13)	-0.0047(12)	0.0011(12)
C6B	0.0265(15)	0.0261(16)	0.0378(16)	-0.0037(12)	-0.0046(12)	-0.0024(11)
C7B	0.0242(14)	0.0276(16)	0.0318(16)	-0.0072(12)	-0.0056(12)	0.0009(11)
C8B	0.0306(16)	0.0312(17)	0.0411(18)	-0.0113(14)	-0.0084(13)	-0.0016(12)
C9B	0.0393(17)	0.0400(19)	0.0419(18)	-0.0213(15)	-0.0126(14)	0.0031(14)
C10B	0.0357(17)	0.0421(19)	0.0316(16)	-0.0121(14)	-0.0076(13)	0.0029(13)
C11B	0.0348(16)	0.0360(18)	0.0341(17)	-0.0024(13)	-0.0051(13)	-0.0016(12)
C12B	0.0225(14)	0.0324(17)	0.0306(16)	-0.0075(12)	-0.0044(12)	0.0010(11)
C13B	0.0233(14)	0.0303(16)	0.0359(16)	-0.0071(12)	-0.0015(12)	-0.0051(11)
C14B	0.0475(19)	0.0333(19)	0.0472(19)	-0.0050(14)	-0.0141(15)	-0.0015(14)
C15B	0.077(2)	0.043(2)	0.0420(19)	0.0043(15)	-0.0079(17)	-0.0172(17)

Table 6. Anisotropic Displacement Parameters (continued)

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C16B	0.071(3)	0.055(2)	0.064(2)	0.0025(18)	-0.0278(19)	0.0074(18)
C17B	0.0389(18)	0.055(2)	0.0443(18)	-0.0167(16)	-0.0100(14)	-0.0106(15)
C18B	0.070(2)	0.034(2)	0.076(2)	-0.0192(18)	-0.0106(18)	-0.0103(16)
C19B	0.064(2)	0.070(3)	0.0384(19)	-0.0192(17)	-0.0037(16)	-0.0002(18)

The form of the anisotropic displacement parameter is:

$\exp[-2\pi^2(h^2a^{*2}U_{11} + k^2b^{*2}U_{22} + l^2c^{*2}U_{33} + 2klb^*c^*U_{23} + 2hla^*c^*U_{13} + 2hka^*b^*U_{12})]$

Table 7. Derived Atomic Coordinates and Displacement Parameters for Hydrogen Atoms

(a) Molecule A

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H1OA	-0.1505	-0.1324	0.4247	0.056
H2OA	0.0551	-0.0502	0.4171	0.053
H1A	0.0366	0.1149	0.0592	0.044
H2AA	0.2319	0.0605	0.1126	0.049
H2BA	0.1648	0.1188	0.2068	0.049
H3AA	0.1061	-0.0686	0.2559	0.047
H3BA	0.0928	-0.0846	0.1536	0.047
H5A	-0.2340	0.0280	0.3410	0.037
H6A	-0.1362	0.0621	0.4522	0.036
H9A	-0.1112	0.4539	0.4196	0.039
H11A	-0.1490	0.3850	0.1724	0.043
H13A	-0.1632	0.1722	0.1562	0.036
H14A	0.0750	0.3339	0.1036	0.053
H15A	-0.1021	0.3377	0.0295	0.065
H15B	-0.0001	0.2805	-0.0554	0.065
H15C	0.0131	0.4106	-0.0379	0.065
H16A	0.2888	0.2390	0.0389	0.079
H16B	0.2493	0.3513	-0.0319	0.079
H16C	0.2380	0.2210	-0.0499	0.079
H17A	-0.2498	-0.0186	0.1971	0.058
H17B	-0.1423	-0.1327	0.2022	0.058
H17C	-0.1219	-0.0340	0.1120	0.058
H18A	-0.0771	0.2706	0.6219	0.051
H18B	-0.1817	0.3551	0.5711	0.051
H18C	-0.0259	0.3712	0.5377	0.051
H19A	-0.2136	0.5803	0.2085	0.064
H19B	-0.0982	0.6114	0.2507	0.064
H19C	-0.2452	0.6003	0.3158	0.064

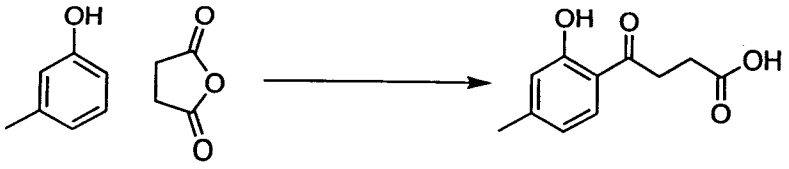
(b) Molecule B

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H1OB	0.3291	0.1637	0.5760	0.062
H2OB	0.1906	0.1037	0.4145	0.048
H1B	0.4082	0.5027	0.3079	0.042
H2AB	0.2166	0.3370	0.3449	0.043
H2BB	0.1841	0.4726	0.3646	0.043
H3AB	0.2412	0.3178	0.4965	0.041
H3BB	0.3060	0.4401	0.4774	0.041
H5B	0.5449	0.1366	0.4387	0.038

Table 7. Derived Parameters for Hydrogen Atoms (continued)

Atom	*x*	*y*	*z*	U_{eq}, Å^2
H6B	0.3899	0.0147	0.4370	0.037
H9B	0.4974	-0.0311	0.1309	0.047
H11B	0.5918	0.2920	0.1370	0.043
H13B	0.5737	0.3515	0.2922	0.036
H14B	0.3732	0.3992	0.1579	0.051
H15D	0.5759	0.4827	0.1353	0.066
H15E	0.4935	0.6069	0.1579	0.066
H15F	0.4889	0.5558	0.0657	0.066
H16D	0.1666	0.5122	0.2133	0.077
H16E	0.2412	0.5734	0.1128	0.077
H16F	0.2430	0.6250	0.2054	0.077
H17D	0.5192	0.3372	0.5287	0.053
H17E	0.5409	0.4369	0.4392	0.053
H17F	0.6335	0.3132	0.4372	0.053
H18D	0.3567	-0.2401	0.3140	0.071
H18E	0.5025	-0.2032	0.2630	0.071
H18F	0.3830	-0.1687	0.2104	0.071
H19D	0.7039	0.1856	0.0047	0.070
H19E	0.5665	0.1791	-0.0228	0.070
H19F	0.6604	0.0603	0.0000	0.070

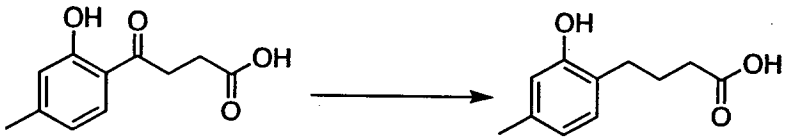
4-(2-Hydroxy-4-methylphenyl)-4-oxobutyric Acid (13).



12 13

$AlCl_3$ (116.0 g, 0.87 mol) was added portionwise over ca 25 min to a stirred and cooled (0 °C) solution of *m*-cresol (52.5 g, 0.46 mol) and succinic anhydride (40.0 g, 0.40 mol) in $Cl_2CHCHCl_2$ (400 mL). The mixture was stirred at room temperature for 3 h, then heated at 130 °C for 2 h, and cooled to room temperature. Concentrated hydrochloric acid and ice were added alternately in small portions to the resulting solid mass, which was then extracted with CH_2Cl_2 (3 x 400 mL). The solvent was evaporated and the residual green oil was dissolved in aqueous NaOH (2 N, 200 mL). The solution was boiled with charcoal (30.0 g) (oil bath at 110 °C) for 3 h and filtered through a Celite pad (5 x 2.5 cm) while hot. The filtrate was acidified with concentrated hydrochloric acid and the resulting precipitate was collected and recrystallized from ethanol to give crude **13**[6] as slightly green crystals. This material was used in the next step without further purification.

4-(2-Hydroxy-4-methylphenyl)butyric Acid (14).



13 14

A mixture of Zn pieces (120.0 g, 1.84 mol), $HgCl_2$ (12.0 g, 44 mmol), concentrated hydrochloric acid (6 mL) and water (180 mL) was stirred for 5 min. The solution was decanted and concentrated hydrochloric acid (200 mL), water (90 mL), PhMe

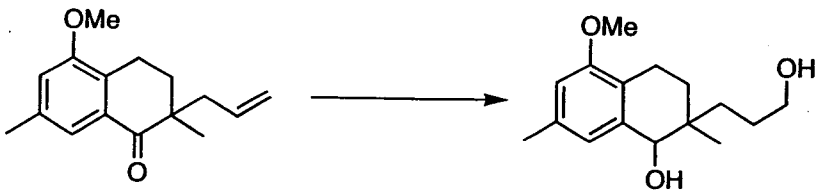
(120 mL) and crude acid **13** were added. The resulting mixture was refluxed (oil bath at 110 °C) for 2 days, concentrated hydrochloric acid (50 mL) being added every 12 h. The mixture was cooled and extracted with Et_2O (3 x 400 mL), and the combined organic extracts were washed with brine, dried (Na_2SO_4) and evaporated. The crude **14**[6] was used for the next step without further purification.

4-(2-Methoxy-4-methylphenyl)butyric Acid (15).

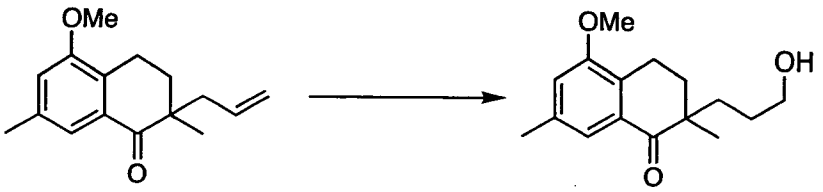
14 **15**

Me_2SO_4 (130 mL, 1.34 mol) was added dropwise over ca 20 min to a stirred and heated (100 °C) solution of **14** (crude from previous reaction), NaOH (100 g, 2.50 mol) and $Na_2S_2O_4$ (7 g, 40 mmol) in water (400 mL). Stirring was continued for 10 h at 100 °C. The mixture was cooled, acidified with concentrated hydrochloric acid and then extracted with CH_2Cl_2 (3 x 400 mL). The combined organic extracts were washed with brine, dried (Na_2SO_4) and evaporated. Flash chromatography of the residue over silica gel (7.5 x 40 cm), using 1:4 EtOAc-hexane, containing 1% AcOH), gave acid **15**[6] (55.2 g, 67% over 3 steps) as a white solid.

2-(3-Hydroxypropyl)-5-methoxy-2,7-dimethyl-1,2,3,4-tetrahydronaphthalen-1-ol (33) and 2-(3-Hydroxypropyl)-5-methoxy-2,7-dimethyl-3,4-dihydro-2*H*-naphthalen-1-one (32).



31 33



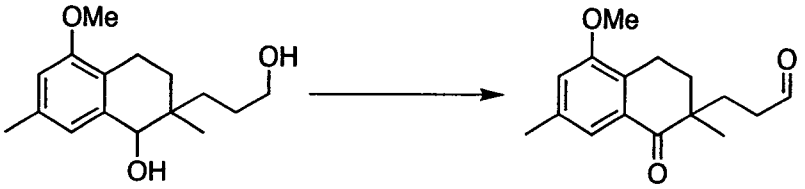
31 32

9-BBN (0.5 M in THF, 5.0 mL, 2.50 mmol) was added dropwise over ca 10 min to a stirred and cooled (0 °C) solution of olefin **31** (0.20 g, 0.82 mmol) in THF (10 mL). The ice bath was removed and stirring was continued for 10 h. The mixture was cooled to 0 °C and quenched by successive slow addition of MeOH (1 mL), NaOH (2 N, 2 mL) and H_2O_2 (30%, 1 mL). The ice bath was left in place but not recharged, and stirring was continued for 2 h. Water (10 mL) was added and the mixture was extracted with Et_2O (3 x 20 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$) and evaporated. Flash chromatography of the residue over silica gel (2 x 25 cm), using 1:3 EtOAc-hexane, gave diol **33** (126 mg, 70%) and **32** (43 mg, 20%), both as colorless oils. Diol **33** had: FTIR (CH_2Cl_2, cast) 3346, 2934, 1613, 1585 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 0.80 (s, 3 H), 1.29-1.36 (m, 1 H), 1.42-1.48 (m, 1 H), 1.50-1.56 (m, 1 H), 1.58-1.73 (m, 4 H), 1.77-1.84 (m, 1 H), 2.31 (s, 3 H), 2.44-2.52 (m, 1 H), 2.66 (td, J = 5.4, 12.7 Hz, 1 H), 3.60-3.69 (m, 2 H), 3.79 (s, 3 H), 4.23 (s, 1 H), 6.56 (s, 1 H), 6.80 (s, 1 H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ 19.8 (t), 20.8 (q), 21.5 (q), 26.5 (t), 29.0 (t), 32.5 (t), 35.6 (t), 55.2 (q), 63.7 (s), 109.9 (d), 121.93 (s), 121.95 (d), 136.5 (s), 138.7 (s), 156.9 (s); exact mass (electrospray) m/z calcd for $C_{16}H_{24}NaO_3$ 287.162315, found 287.162362.

Keto alcohol **32** had: FTIR (CH_2Cl_2, cast) 3430, 2935, 1680, 1608 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.15 (s, 3 H), 1.48-1.60 (m, 4 H), 1.68-1.75 (m, 1 H), 1.84-1.90 (m, 1 H), 1.99-2.05 (m, 1 H), 2.34 (s, 3 H), 2.75-2.88 (m, 2 H), 3.57 (t, J = 6.0 Hz, 2

H), 3.83 (s, 3 H), 6.80 (d, J = 1.0 Hz, 1 H), 7.44 (s, 1 H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ 18.9 (t), 21.5 (q), 22.1 (q), 27.4 (t), 32.5 (t), 33.1 (t), 44.0 (t), 55.6 (q), 63.1 (s), 115.1 (d), 119.6 (d), 129.3 (s), 132.1 (s), 136.8 (s), 156.6 (s), 203.0 (s); exact mass m/z calcd for $C_{16}H_{22}O_3$ 262.15689, found 262.15695.

3-(5-Methoxy-2,7-dimethyl-1-oxo-1,2,3,4-tetrahydronaphthalen-2-yl)propionaldehyde (34).

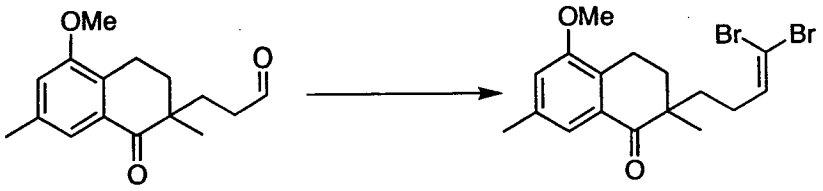


33 **34**

Dry DMSO (0.85 mL, 12.0 mmol) in CH_2Cl_2 (5 mL) was added dropwise over ca 10 min to a stirred and cooled (-78 °C) solution of $(COCl)_2$ (0.89 mL, 10.0 mmol) in CH_2Cl_2 (30 mL). Stirring was continued for 30 min, and diol **33** (0.70 g, 2.66 mmol) in CH_2Cl_2 (5 mL) was added dropwise over ca 5 min, and a further portion of CH_2Cl_2 (1 mL) was used as a rinse. Stirring at -78 °C was continued for 1 h, and Et_3N (2.8 mL, 20.0 mmol) was added dropwise over ca 2 min. Stirring was continued for 1 h, the dry-ice bath was removed and stirring was continued for 10 h. Saturated aqueous NH_4Cl (40 mL) was added and the mixture was extracted with Et_2O (3 x 50 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$) and evaporated. Flash chromatography of the residue over silica gel (2 x 25 cm), using 1:9 EtOAc-hexane, gave keto aldehyde **34** (0.63 g, 91%) as a yellow oil: FTIR (CH_2Cl_2, cast) 2933, 2854, 2723, 1723, 1679, 1608 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.18 (s, 3 H), 1.83-2.03 (m, 4 H), 2.34 (s, 3 H), 2.36-2.54 (m, 2 H), 2.76-2.92 (m, 2 H),

3.81 (s, 3 H), 6.81 (d, J = 1.1 Hz, 1 H), 7.42 (d, J = 0.7 Hz, 1 H), 9.74 (t, J = 1.5 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 18.8 (t), 21.5 (q), 22.0 (q), 28.6 (t), 33.4 (t), 39.1 (t), 43.6 (s), 55.6 (q), 115.2 (d), 119.5 (d), 129.1 (s), 131.9 (s), 136.9 (s), 156.6 (s), 202.0 (d), 202.2 (s); exact mass m/z calcd for $C_{16}H_{20}O_3$ 260.14124, found 260.14148.

2-(4,4-Dibromobut-3-enyl)-5-methoxy-2,7-dimethyl-3,4-dihydro-2*H*-naphthalen-1-one (35).

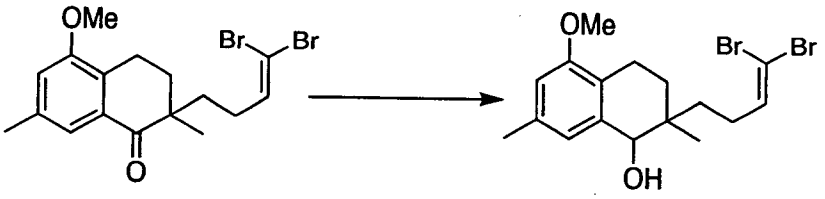


34 **35**

Ph_3P (1.10 g, 4.82 mmol) was added to a stirred and cooled (-15 °C) solution of CBr_4 (1.40 g, 4.82 mmol) in CH_2Cl_2 (40 mL). Stirring at -15 °C was continued for 30 min and the mixture was then cooled to -78 °C. A solution of keto aldehyde **34** (0.83 g, 3.21 mmol) in CH_2Cl_2 (10 mL) was added dropwise over ca 5 min, followed by Et_3N (0.71 mL, 5.10 mmol). The cooling bath was removed and stirring was continued for 30 h. Water (30 mL) was added and the mixture was extracted with Et_2O (3 x 50 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$) and evaporated. Flash chromatography of the residue over silica gel (2.5 x 25 cm), using 1:9 EtOAc-hexane, gave ketone **35** [1.07 g, 98%, corrected for recovered **34** (0.15 g)] as a yellow oil: FTIR (CH_2Cl_2, cast) 2932, 2854, 1681, 1609 cm^{-1}; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.18 (s, 3 H), 1.62 (ddd, J = 5.3, 11.8, 13.7 Hz, 1 H), 1.74 (ddd, J = 5.3, 11.4, 13.7 Hz, 1 H), 1.86-1.92 (m, 1 H), 1.98-2.14 (m, 3 H), 2.35 (s, 3 H), 2.76-2.88 (m, 2 H), 3.80 (s, 3 H), 6.34 (t, J = 7.3 Hz, 1 H), 6.81 (d, J = 0.9 Hz, 1 H), 7.43

(s, 1 H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ 18.8 (t), 21.5 (q), 21.8 (q), 28.1 (t), 33.0 (t), 34.2 (t), 44.0 (s), 55.6 (q), 89.0 (s), 115.1 (d), 119.6 (d), 129.1 (s), 132.1 (s), 136.9 (s), 138.3 (d), 156.6 (s), 202.2 (d); exact mass *m/z* calcd for $C_{17}H_{20}{}^{81}Br_2O_2$ 417.97891, found 417.97874.

2-(4,4-Dibromobut-3-enyl)-5-methoxy-2,7-dimethyl-1,2,3,4-tetrahydronaphthalen-1-ol (36).

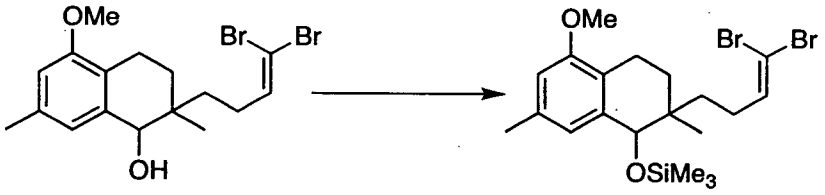


35 **36**

DIBAL-H (1 M in cyclohexane, 3.50 mL, 3.50 mmol) was added dropwise to a stirred and cooled (-78 °C) solution of ketone **35** (1.04 g, 2.50 mmol) in CH_2Cl_2 (30 mL). Stirring was continued for 2 h at -78 °C and $Na_2SO_4.10H_2O$ (1.0 g) was added. The cooling bath was removed, stirring was continued for 30 min, and the mixture was filtered through a Celite pad (1 x 1.5 cm), using CH_2Cl_2 as a rinse. Evaporation of the solvent and flash chromatography of the residue over silica gel (2 x 25 cm), using 1:19 EtOAc-hexane, gave alcohol **36** (0.70 g, 67%) as a yellow oil: FTIR (CH_2Cl_2, cast) 3396, 2931, 2857, 1613 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 0.85 (s, 3 H), 1.39 (ddd, *J* = 5.3, 11.7, 13.7 Hz, 1 H), 1.43-1.50 (m, 2 H), 1.59 (ddd, *J* = 5.6, 11.7, 13.7 Hz, 1 H), 1.80 (ddd, *J* = 6.5, 9.2, 13.7 Hz, 1 H), 2.11-2.24 (m, 2 H), 2.32 (s, 3 H), 2.46-2.54 (m, 1 H), 2.66 (td, *J* = 5.6, 18.1 Hz, 1 H), 3.80 (s, 3 H), 4.21 (s, 1 H), 6.41 (t, *J* = 7.2 Hz, 1 H), 6.57 (s, 1 H), 6.80 (s, 1 H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ 19.8 (t), 20.8 (q), 21.5 (q), 27.6 (t), 28.6 (t), 34.3 (t), 35.8 (s), 55.2 (q), 75.4 (d), 88.4 (s), 110.0 (d), 121.66 (d), 121.69

(s), 136.7 (s), 138.7 (s), 139.2 (d), 156.9 (s); exact mass *m/z* calcd for $C_{17}H_{22}{}^{81}Br_2O_2$ 419.99457, found 419.99441.

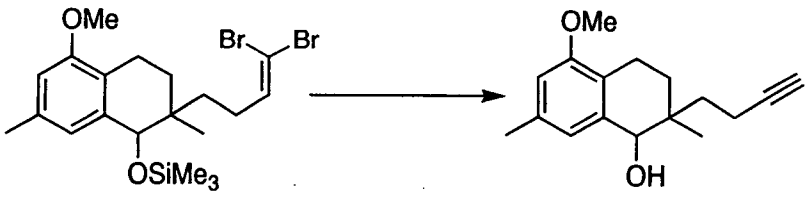
[2-(4,4-Dibromobut-3-enyl)-5-methoxy-2,7-dimethyl-1,2,3,4-tetrahydronaphthalen-1-yloxy]trimethylsilane (37).



36 **37**

Me_3SiCl (0.15 mL, 1.16 mmol) was added dropwise to a stirred and cooled (0 °C) solution of alcohol **36** (0.42 g, 1.02 mmol) and imidazole (84 mg, 1.22 mmol) in CH_2Cl_2 (20 mL). The ice bath was removed and stirring was continued for 30 min. Saturated aqueous NH_4Cl (15 mL) was added and the mixture was extracted with Et_2O (3 x 30 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$) and evaporated. Flash chromatography of the residue over silica gel (2 x 25 cm), using 1:19 EtOAc-hexane, gave dibromide **37** (0.47 g, 94%) as a yellow oil: FTIR (CH_2Cl_2, cast) 2954, 2858, 1614 cm^{-1}; ^1H NMR (C_6D_6, 400 MHz) δ 0.13 (s, 9 H), 0.75 (s, 3 H), 1.22-1.34 (m, 2 H), 1.50 (ddd, *J* = 5.5, 11.9, 13.4 Hz, 1 H), 1.84-2.06 (m, 3 H), 2.25 (s, 3 H), 2.71 (td, *J* = 7.5, 18.4 Hz, 1 H), 2.87 (td, *J* = 6.3, 18.4 Hz, 1 H), 3.37 (s, 3 H), 4.29 (s, 1 H), 6.05 (t, *J* = 7.2 Hz, 1 H), 6.41 (d, *J* = 0.8 Hz, 1 H), 6.86 (s, 1 H); ^{13}C NMR (C_6D_6, 100.6 MHz) δ 1.0 (q), 20.5 (t), 21.6 (q), 21.7 (q), 28.1 (t), 29.4 (t), 34.3 (t), 36.6 (s), 54.8 (q), 77.7 (d), 88.6 (s), 109.8 (d), 122.2 (s), 122.3 (d), 135.7 (s), 139.5 (s), 139.9 (d), 157.4 (s); exact mass *m/z* calcd for $C_{20}H_{30}{}^{81}Br_2O_2Si$ 492.03409, found 492.03434.

2-But-3-ynyl-5-methoxy-2,7-dimethyl-1,2,3,4-tetrahydronaphthalen-1-ol (38).

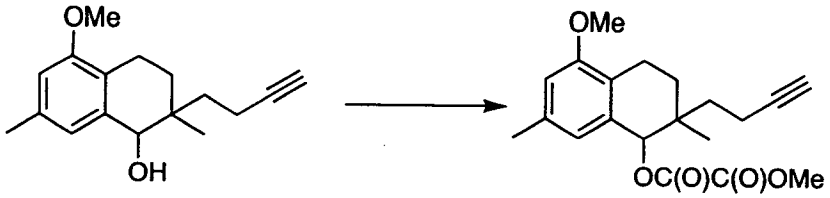


37 **38**

n-BuLi (2.5 M in hexane, 0.85 mL, 2.13 mmol) was added dropwise over ca 10 min to a stirred and cooled (-78 °C) solution of dibromide **37** (0.45 g, 0.92 mmol) in THF (20 mL). The cooling bath was left in place but not recharged, and stirring was continued for 10 h. Saturated aqueous NH_4Cl (15 mL) was added and the mixture was extracted with Et_2O (3 x 25 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$) and evaporated. Bu_4NF (1 M in THF, 1.0 mL, 1.0 mmol) was added to a stirred solution of the residue in THF (10 mL). Stirring was continued for 3 h and the solvent was evaporated. Flash chromatography of the residue over silica gel (2 x 20 cm), using 1:19 EtOAc-hexane, gave acetylene **38** (0.12 g, 53%) as a colorless oil: FTIR (CH_2Cl_2, cast) 3575, 3258, 2919, 2106, 1611 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.85 (s, 3 H), 1.42-1.49 (m, 1 H), 1.54-1.63 (m, 2 H), 1.76-1.85 (m, 2 H), 1.94 (t, J = 2.7 Hz, 1 H), 2.20-2.38 (m, 2 H), 2.31 (s, 3 H), 2.43-2.52 (m, 1 H), 2.64-2.73 (m, 1 H), 3.79 (s, 3 H), 4.22 (s, 1 H), 6.57 (s, 1 H), 6.79 (s, 1 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 13.0 (t), 19.8 (t), 20.5 (q), 21.5 (q), 28.6 (t), 35.8 (t), 35.9 (s), 55.2 (q), 68.0 (d), 75.0 (d), 85.4 (s), 110.0 (d), 121.7 (s), 121.9 (d), 136.6 (s), 138.6 (s), 156.9 (s); exact mass m/z calcd for $C_{17}H_{22}O_2$ 258.16199, found 258.16200.

Oxalic Acid 2-But-3-ynyl-5-methoxy-2,7-dimethyl-1,2,3,4-

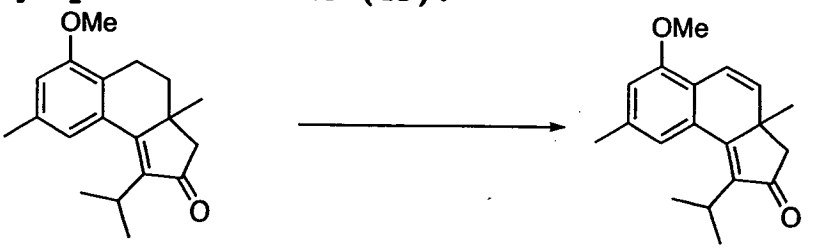
tetrahydronaphthalen-1-yl Methyl Ester (30).



38 **30**

Pyridine (81 μL, 1.0 mmol), followed by MeO_2CCOCl (86 μL, 0.9 mmol) were added to a stirred solution of alcohol **38** (112 mg, 0.45 mmol) in CH_2Cl_2 (10 mL). Stirring was continued for 1.5 h and the solvent was then evaporated. Flash chromatography of the residue over silica gel (1 x 20 cm), using 1:19 EtOAc-hexane, gave ester **30** (123 mg, 80%) as a colorless oil: FTIR (CH_2Cl_2, cast) 3290, 2953, 2118, 1769, 1742, 1614 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 0.85 (s, 3 H), 1.58-1.78 (m, 3 H), 1.92 (t, *J* = 2.7 Hz, 1 H), 1.89-1.98 (m, 1 H), 2.18-2.26 (m, 2 H), 2.28 (s, 3 H), 2.46-2.57 (m, 1 H), 2.78 (ddd, *J* = 3.5, 6.6, 18.5 Hz, 1 H), 3.78 (s, 3 H), 3.85 (s, 3 H), 5.78 (d, *J* = 0.7 Hz, 1 H), 6.61 (d, *J* = 0.7 Hz, 1 H), 6.74 (s, 1 H); ^{13}C NMR ($CDCl_3$, 100.6 MHz) δ 12.9 (t), 19.6 (t), 19.8 (q), 21.4 (q), 28.3 (t), 35.6 (t), 36.2 (s), 53.4 (q), 55.2 (q), 68.2 (d), 79.3 (d), 84.6 (s), 111.0 (d), 122.4 (s), 122.5 (d), 133.1 (s), 136.7 (s), 156.8 (s), 157.4 (s), 158.3 (s); exact mass *m/z* calcd for $C_{20}H_{24}O_5$ 344.16238, found 344.16239.

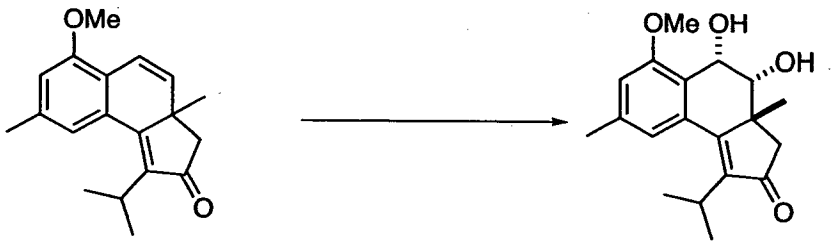
1-Isopropyl-6-methoxy-3a,8-dimethyl-3,3a-dihydro-cyclopenta[*a*]naphthalen-2-one (45).



42 45

DDQ (1.20 g, 5.3 mmol) was added in one lot to a stirred solution of ketone **42** (1.01 g, 3.6 mmol) in 1,4-dioxane (60 mL) and the mixture was refluxed for 8.5 h (Ar atmosphere), cooled to room temperature, and filtered through a short pad of silica gel (5 x 3.5 cm), using Et_2O (100 mL) as a rinse. Evaporation of the solvent and flash chromatography of the residue over silica gel (2 x 30 cm), using 1:9 EtOAc-hexane, gave enone **45** (0.78 g, 78%) as a yellow oil: FTIR ($CHCl_3$, cast) 2958, 1696, 1603 cm^{-1}; ^1H NMR ($CDCl_3$, 400 MHz) δ 1.17 (d, J = 6.8 Hz, 3 H), 1.18 (s, 3 H), 1.36 (d, J = 7.1 Hz, 3 H), 2.40 (s, 3 H), 2.44 (AB q, J = 17.6 Hz, $\Delta\nu_{AB}$ = 109.1 Hz, 2 H), 3.02 (septet, J = 7.0 Hz, 1 H), 3.80 (s, 3 H), 6.05 (d, J = 9.6 Hz, 1 H), 6.72 (d, J = 9.5 Hz, 1 H), 6.73 (s, 1 H), 6.85 (s, 1 H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ 19.8 (q), 20.7 (q), 22.0 (q), 25.5 (q), 27.3 (d), 42.3 (t), 48.4 (s), 55.6 (q), 113.1 (d), 119.2 (d), 119.8 (s), 120.6 (d), 129.8 (s), 136.7 (d), 138.0 (s), 141.8 (s), 154.9 (s), 170.0 (s), 207.3 (s); exact mass *m/z* calcd for $C_{19}H_{22}O_2$ 282.16199, found 282.16155.

(3a*R,4*R**,5*S**)-4,5-Dihydroxy-1-isopropyl-6-methoxy-3a,8-dimethyl-3,3a,4,5-tetrahydrocyclopenta[*a*]naphthalene-2-one (46).**

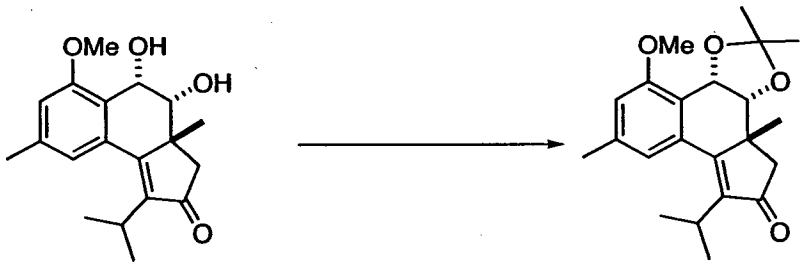


45 **46**

OsO_4 (50 mg, 0.2 mmol) and NMO (1.93 g, 16.0 mmol) were added to a stirred solution of olefin **45** (1.50 g, 5.3 mmol) in a

mixture of CCl_4 (30 mL), water (6 mL), *t*-BuOH (25 mL), and acetone (40 mL) and stirring was continued for 12 h. The mixture was diluted with water (40 mL) and extracted with CH_2Cl_2 (3 x 50 mL). The combined organic extracts were washed with brine, dried ($MgSO_4$) and evaporated. Flash chromatography of the residue over silica gel (2.5 x 30 cm), using 1:4 *t*-BuOMe-CH_2Cl_2, gave diol **46** (1.66 g, 98%) as a colorless oil: FTIR ($CHCl_3$, cast) 3525, 2961, 1691, 1608 cm^{-1}; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.04 (s, 3 H), 1.23 (d, J = 6.8 Hz, 3 H), 1.37 (d, J = 7.1 Hz, 3 H), 2.40 (s, 3 H), 2.47 (AB q, J = 18.1 Hz, $\Delta\nu_{AB}$ = 401.6 Hz, 2 H), 3.05 (s, 1 H), 3.16 (septet, J = 7.0 Hz, 1 H), 3.93 (s, 3 H), 4.04 (d, J = 4.4 Hz, 1 H), 4.36 (s, 1 H), 5.03 (d, J = 4.4 Hz, 1 H), 6.79 (s, 1 H), 6.94 (s, 1 H); ^{13}C NMR ($CDCl_3$, 125.7 MHz) δ 20.0 (q), 20.5 (q), 21.9 (q), 22.1 (q), 26.1 (d), 46.1 (t), 46.4 (s), 55.6 (q), 66.5 (d), 73.1 (d), 112.7 (d), 121.4 (d), 121.6 (s), 131.5 (s), 138.8 (s), 144.6 (s), 158.6 (s), 163.9 (s), 208.1 (s); exact mass *m/z* calcd for $C_{19}H_{24}O_4$ 316.16745, found 316.16764.

(3a*R,3b*R**,10b*S**)-6-Isopropyl-10-methoxy-2,2,3b-trimethyl-3a,3b,4,10b-tetrahydrocyclopenta[3,4]naphtho[1,2-d][1,3]dioxol-5-one (47).**

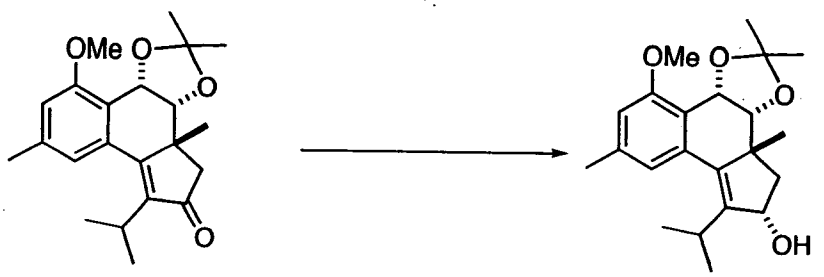


46 **47**

Pyridinium *p*-toluenesulfonate (0.1 g, 0.39 mmol) was added to a stirred solution of diol **46** (1.30 g, 4.11 mmol) in acetone

(30 mL) and $MeC(OMe)_2Me$ (30 mL), and the mixture was stirred at room temperature for 4 h. Evaporation of the solvent and flash chromatography of the residue over silica gel (2 X 25 mL), using 1:4 EtOAc-hexane, gave ketone **47** (1.29 g, 88%) as a white solid: mp 185-187 °C; FTIR (CH_2Cl_2, cast) 2961, 1697, 1607 cm^{-1}; ^1H NMR (C_6D_6, 400 MHz) δ 0.66 (s, 3 H), 1.14 (s, 3 H), 1.28 (s, 3 H), 1.45 (d, J = 6.9 Hz, 3 H), 1.58 (d, J = 7.0 Hz, 3 H), 2.10 (s, 3 H), 2.46 (AB q, J = 18.2 Hz, $\Delta\nu_{AB}$ = 398.6 Hz, 2 H), 3.30 (septet, J = 7.0 Hz, 1 H), 3.33 (s, 3 H), 4.04 (d, J = 6.5 Hz, 1 H), 5.56 (d, J = 6.5 Hz, 1 H), 6.42 (s, 1 H), 6.90 (s, 1 H); ^{13}C NMR (C_6D_6, 100.6 MHz) δ 20.8 (q), 20.9 (q), 21.8 (q), 23.2 (q), 25.8 (q), 26.8 (q), 27.5 (d), 45.2 (t), 46.0 (s), 55.3 (q), 70.2 (d), 79.7 (d), 108.5 (s), 112.9 (d), 120.4 (d), 121.8 (s), 133.0 (s), 139.3 (s), 144.8 (s), 159.0 (s), 163.7 (s), 205.9 (s); exact mass m/z calcd for $C_{22}H_{28}O_4$ 356.19876, found 356.19828.

(3a*R,3b*R**,5*S**,10b*S**)-6-Isopropyl-10-methoxy-2,2,3b-trimethyl-3a,4,5,10b-tetrahydro-3b*H*-cyclopenta[3,4]naphtho[1,2-d][1,3]dioxol-5-ol (48). (3a*R**,3b*R**,10b*S**)-6-Isopropyl-10-methoxy-2,2,3b-trimethyl-3b,10b-dihydro-3a*H*-cyclopenta[3,4]naphtho[1,2-d][1,3]dioxole (50).**



47 **48**