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CRYSTAL STRUCTURE OF AN EPOXYCEMBRADIENOL, 3,15-EPOXY-4-HYDROXYCEMBRA-7(Z), 11(Z)-DIENE

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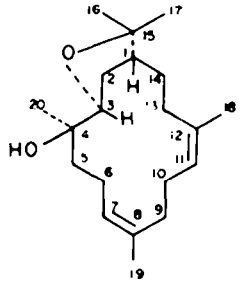
Abstract—The crystal structure of the title compound, $C_{20}H_{34}O_2$, has been determined by single crystal X-ray diffraction methods at 295 K from diffractometer data using direct methods and refined by least squares to a residual of 0.06 for 2152 "observed" reflections. Crystals are orthorhombic, $P2_12_12_1$, a = 32.770(8), b = 10.960(3), c = 10.850(3)Å, Z = 8. The asymmetric unit comprises two independent molecules, both of which confirm the structure proposed in the preceding paper.

The preceding paper[1] has described the isolation, characterization and chemistry of epoxycembradienol, the proposed structure being as shown. The present paper describes the structure determination of this compound by single crystal X-ray diffraction methods; atom numbering follows the systematic scheme given below the two independent molecules being designated I and II.



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A prismatic crystal fragment 0.2 × 0.3 × 0.3 mm was used for the crystallographic work. The unit cell was determined by a least squares fit of the angular parameters of 15 reflections with $2\theta \sim 30°$ centred in the counter aperture of a Syntex $P\bar{1}$ four-circle diffractometer. A unique data set was then gathered using a conventional $2\theta/\theta$ scan within the limit $2\theta < 100°$ yielding 2310 independent reflections; 2152 of these having $I > 2\sigma(I)$ were considered "observed" and used in the structure solution and refinement. No correction was applied for absorption. T was 295(1) K.

Crystal Data. $C_{20}H_{34}O_2$, M = 306.5, orthorhombic, space group $P2_12_12_1$ (D_2^4, No. 19), a = 32.770(8), b = 10.960(3), c = 10.850(3)Å, U = 3897(3)Å^3, D_m = 1.06(1), D_c (Z = 8) = 1.045 g cm^{-3}, $F(000)$ = 1360, Ni-filtered Cu(K_α) radiation (λ = 1.5418 Å), μ = 5.02 cm^{-1}, neutral atom scattering factors.[2-4]

The structure was solved using the MULTAN programme package[5] and refined using the X-RAY 72 program system[6] implemented on a CYBER 73 computer. Refinement was carried out using block diagonal least squares, the parameters of each C or O atom with those of any associated hydrogen atoms being refined within each block. Anisotropic thermal parameters were

Table 1. Atom fractional cell coordinates (x, y, z, × 10^3 H; × 10^4, others) and thermal parameters (U_{ij} × 10^3 Å^2) with least squares estimated standard deviations in the final digit in parentheses. Also given for hydrogen atoms is r Å, the C–H distance

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Molecule I									
C(1)	0902(2)	0924(6)	6726(5)	73(5)	70(4)	44(4)	10(4)	−7(4)	−14(4)
H(1)	052(2)	070(5)	694(5)	80(−)	(r = 1.28(6))				
C(2)	0870(2)	2175(6)	6121(6)	75(5)	78(5)	51(4)	10(4)	−2(4)	−8(4)
H(2a)	051(2)	225(5)	567(5)	80(−)	(r = 1.29(6))				
H(2b)	112(2)	232(6)	564(6)	80(−)	(r = 0.98(6))				
C(3)	0851(2)	3005(6)	6232(6)	69(4)	67(4)	51(4)	6(4)	−8(4)	−9(4)
H(3)	051(2)	299(5)	764(5)	80(−)	(r = 1.20(6))				
O(3)	1139(1)	2472(4)	8099(4)	85(3)	74(3)	55(3)	10(3)	−16(2)	−7(3)
C(4)	0953(2)	4359(6)	6979(6)	67(5)	73(5)	70(4)	−3(3)	1(4)	−3(4)
C(5)	0689(2)	4802(8)	5898(7)	89(6)	87(6)	62(5)	10(5)	−9(4)	12(5)
H(5a)	072(2)	592(7)	570(6)	100(−)	(r = 1.25(7))				
H(5b)	075(2)	443(7)	508(6)	100(−)	(r = 1.00(7))				
C(6)	0222(2)	4663(9)	6111(7)	94(6)	88(6)	68(5)	23(5)	0(5)	1(5)
H(6a)	014(2)	373(6)	662(6)	100(−)	(r = 1.19(7))				

Table 1 (*Contd*)

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
H(6b)	015(2)	538(7)	653(6)	100(–)	(r = 0.94(7))				
C(7)	−0015(2)	4622(8)	4901(7)	86(5)	113(7)	82(6)	29(5)	7(5)	33(6)
H(7)	−003(2)	542(7)	437(6)	110(–)	(r = 1.04(7))				
C(8)	−0206(2)	3661(7)	4449(7)	81(5)	97(6)	98(6)	17(5)	0(5)	17(5)
C(9)	−0229(2)	2464(8)	5018(8)	73(5)	104(6)	80(5)	7(5)	12(5)	1(5)
H(9a)	−009(2)	236(6)	590(6)	100(–)	(r = 1.07(7))				
H(9b)	−052(2)	229(6)	522(6)	100(–)	(r = 1.00(7))				
C(10)	−0027(2)	1418(8)	4231(8)	64(5)	120(7)	76(5)	−13(5)	−6(5)	4(5)
H(10a)	−022(2)	147(6)	311(6)	100(–)	(r = 1.37(6))				
H(10b)	019(2)	169(7)	404(7)	100(–)	(r = 0.81(7))				
C(11)	−0007(2)	0218(8)	4887(7)	80(6)	103(6)	72(5)	−29(5)	7(4)	−5(5)
H(11)	−021(2)	−003(7)	535(6)	110(–)	(r = 0.88(7))				
C(12)	0309(2)	−0520(6)	5127(6)	69(5)	88(5)	71(5)	−6(4)	3(4)	−30(5)
C(13)	0735(2)	−0239(7)	4769(7)	76(5)	72(5)	68(5)	3(5)	1(4)	−14(5)
H(13a)	080(2)	−096(6)	434(6)	90(–)	(r = 0.95(7))				
H(13b)	072(2)	043(6)	444(7)	90(–)	(r = 0.82(7))				
C(14)	1028(2)	−0119(10)	5865(9)	87(7)	84(6)	65(5)	10(6)	−8(6)	−10(7)
H(14a)	099(2)	108(6)	642(6)	90(–)	(r = 1.22(6))				
H(14b)	120(2)	000(9)	570(9)	90(–)	(r = 0.61(8))				
C(15)	1201(2)	1180(6)	7815(6)	70(4)	75(5)	54(4)	6(4)	−6(4)	−4(4)
C(16)	1080(2)	0459(6)	8953(6)	129(7)	92(5)	60(5)	−9(5)	−16(5)	0(5)
H(16a)	109(–)	−043(–)	876(–)	110(–)	(r = 0.99(–))				
H(16b)	080(–)	070(–)	921(–)	110(–)	(r = 0.99(–)				
H(16c)	127(–)	063(–)	966(–)	110(–)	(r = 1.01(–))				
C(17)	1648(2)	1047(7)	7532(7)	80(5)	113(6)	80(5)	14(5)	−20(4)	−11(5)
H(17a)	172(–)	164(–)	686(–)	110(–)	(r = 1.01(–))				
H(17b)	170(–)	020(–)	724(–)	110(–)	(r = 1.00(–))				
H(17c)	184(–)	121(–)	823(–)	110(–)	(r = 1.00(–))				
C(18)	0244(3)	−1741(7)	5780(8)	140(7)	66(5)	120(7)	−16(5)	22(6)	23(6)
H(18a) } H(18b) } H(18c)	not located								
C(19)	−0431(3)	3799(9)	3194(7)	122(7)	177(9)	88(6)	20(7)	−53(6)	30(7)
H(19a) } H(19b) } H(19c)	not located								
C(20)	1398(2)	4553(7)	6668(7)	97(6)	93(5)	87(5)	−25(5)	−14(5)	−8(5)
H(20a)	146(–)	405(–)	589(–)	90(–)	(r = 1.03(–))				
H(20b)	157(–)	425(–)	735(–)	90(–)	(r = 0.99(–))				
H(20c)	147(–)	541(–)	648(–)	90(–)	(r = 0.99(–))				
O(20)	0845(1)	5077(4)	8035(4)	126(4)	68(4)	66(3)	14(3)	3(3)	−16(3)
H(20d)	not located								
Molecule II									
C(1)	2081(2)	2119(6)	2886(6)	68(5)	68(5)	46(4)	1(4)	−11(3)	7(4)
H(1)	230(2)	251(6)	241(6)	80(–)	(r = 0.99(6))				
C(2)	1735(2)	3027(7)	3043(6)	60(4)	71(5)	56(5)	12(4)	−8(4)	−4(4)
H(2a)	149(2)	267(6)	353(6)	90(–)	(r = 1.05(6))				
H(2b)	178(2)	376(6)	340(6)	90(–)	(r = 0.91(7))				
C(3)	1594(2)	3153(6)	1713(6)	66(5)	69(5)	48(8)	6(4)	−7(4)	2(4)
H(3)	177(2)	364(6)	136(6)	80(–)	(r = 0.87(6))				
O(3)	1636(1)	1951(4)	1193(4)	88(3)	76(3)	58(3)	14(3)	−14(3)	−14(3)
C(4)	1171(2)	3669(6)	1474(6)	75(5)	75(5)	53(4)	20(4)	−7(4)	−9(4)
C(5)	1115(2)	4866(7)	2175(7)	74(5)	72(5)	69(5)	10(4)	0(4)	−4(4)
H(5a)	081(2)	531(5)	199(5)	80(–)	(r = 1.14(6))				
H(5b)	111(2)	463(5)	316(5)	80(–)	(r = 1.10(6))				
C(6)	1421(3)	5860(7)	1922(8)	101(7)	78(6)	80(6)	32(5)	−1(5)	6(5)
H(6a)	165(2)	555(6)	175(6)	90(–)	(r = 0.85(6))				
H(6b)	124(2)	648(6)	133(6)	90(–)	(r = 1.11(6))				
C(7)	1498(2)	6659(6)	3048(7)	94(5)	66(5)	80(5)	14(4)	−14(5)	−1(5)
H(7)	115(2)	719(5)	334(5)	90(–)	(r = 1.30(6))				
C(8)	1821(2)	6793(6)	3726(7)	106(6)	48(4)	82(5)	9(4)	−2(5)	0(4)
C(9)	2229(3)	6172(7)	3501(8)	87(6)	66(5)	101(6)	1(5)	3(5)	9(5)
H(9a)	216(2)	553(6)	296(6)	100(–)	(r = 0.95(7))				
H(9b)	249(2)	691(6)	334(6)	100(–)	(r = 1.19(7))				
C(10)	2379(2)	5388(7)	4613(7)	85(6)	71(5)	81(6)	−8(4)	−19(5)	−2(5)
H(10a)	249(2)	602(6)	511(6)	90(–)	(r = 0.95(6))				
H(10b)	209(2)	477(6)	477(5)	90(–)	(r = 1.18(6))				
C(11)	2729(2)	4570(6)	4274(7)	73(5)	72(5)	85(5)	−14(4)	−4(4)	−2(5)
H(11)	304(2)	513(6)	375(6)	100(–)	(r = 1.32(6))				

Table 1 (*Contd*)

Atom	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(12)	2750(2)	3375(6)	4392(6)	66(4)	78(5)	70(5)	16(4)	−12(4)	−5(4)
C(13)	2418(2)	2577(7)	4959(7)	77(5)	75(5)	58(4)	0(4)	−16(4)	9(4)
H(13a)	250(2)	211(6)	577(6)	90(−)	(r = 1.05(6))				
H(13b)	212(2)	307(6)	540(6)	90(−)	(r = 1.21(6))				
C(14)	2247(2)	1590(7)	4086(7)	83(6)	67(5)	68(5)	5(4)	−11(5)	6(5)
H(14a)	244(2)	110(6)	397(7)	80(−)	(r = 0.85(6))				
H(14b)	198(2)	120(5)	457(5)	80(−)	(r = 1.09(6))				
C(15)	1912(2)	1232(6)	1932(6)	68(4)	62(4)	61(5)	11(4)	4(4)	0(4)
C(16)	2226(2)	0724(8)	1092(7)	96(6)	113(6)	100(6)	21(5)	−8(5)	−36(6)
H(16a)	243(−)	028(−)	161(−)	120(−)	(r = 0.99(−))				
H(16b)	236(−)	141(−)	065(−)	120(−)	(r = 0.99(−))				
H(16c)	212(−)	014(−)	047(−)	120(−)	(r = 1.00(−))				
C(17)	1663(3)	0190(7)	2496(7)	136(7)	85(6)	104(6)	−32(6)	−19(6)	13(5)
H(17a)	143(−)	054(−)	297(−)	120(−)	(r = 0.99(−))				
H(17b)	185(−)	−028(−)	307(−)	120(−)	(r = 1.00(−))				
H(17c)	155(−)	−040(−)	187(−)	120(−)	(r = 1.00(−))				
C(18)	3135(2)	2697(7)	3990(8)	81(5)	93(6)	127(7)	4(5)	−14(5)	−15(6)
H(18a)	333(−)	333(−)	394(−)	120(−)	(r = 0.94(−))				
H(18b)	305(−)	196(−)	470(−)	120(−)	(r = 1.25(−)				
H(18c)	319(−)	193(−)	470(−)	120(−)	(r = 1.16(−))				
C(19)	1793(3)	7594(7)	4854(9)	153(8)	68(5)	129(7)	11(5)	−38(7)	−20(6)
H(19a)	204(−)	796(−)	467(−)	110(−)	(r = 0.93(−))				
H(19b)	189(−)	722(−)	561(−)	110(−)	(r = 0.96(−))				
H(19c)	163(−)	839(−)	485(−)	110(−)	(r = 1.02(−))				
C(20)	0835(2)	2790(7)	1827(7)	64(5)	107(6)	86(5)	−7(4)	−0(4)	−5(5)
H(20a)	086(−)	259(−)	272(−)	110(−)	(r = 1.00(−))				
H(20b)	087(−)	202(−)	133(−)	110(−)	(r = 1.00(−))				
H(20c)	055(−)	312(−)	166(−)	110(−)	(r = 1.00(−))				
O(20)	1145(2)	3943(4)	0172(4)	105(4)	94(4)	52(3)	20(3)	−31(3)	−10(3)
H(20d)	104(2)	319(6)	−024(6)	90(−)	(r = 1.00(6))				

Table 2. Interatomic distances and angles (Å, deg.) with least squares estimated standard deviations in the final digit in parentheses

C(1)—C(2)	1.525(9),	1.518(10)	C(2)—C(1)—C(14)	115.2(6),	114.8(6)
C(1)—C(14)	1.533(12),	1.526(10)	C(2)—C(1)—C(15)	102.0(5),	102.9(5)
C(1)—C(15)	1.560(9),	1.525(9)	C(14)—C(1)—C(15)	115.4(6),	117.8(6)
C(2)—C(3)	1.512(9),	1.523(9)	C(1)—C(2)—C(3)	101.5(5),	100.4(5)
C(3)—O(3)	1.456(8),	1.440(8)	C(2)—C(3)—C(4)	115.2(5),	118.2(5)
O(3)—C(15)	1.465(8),	1.442(8)	C(2)—C(3)—O(3)	104.4(5),	105.1(5)
C(3)—C(4)	1.546(9),	1.518(9)	C(4)—C(3)—O(3)	111.0(5),	111.2(5)
C(4)—C(5)	1.537(10),	1.528(10)	C(3)—O(3)—C(15)	109.9(4),	110.0(4)
C(4)—C(20)	1.510(10),	1.512(10)	C(3)—C(4)—C(20)	112.6(6),	112.6(5)
C(4)—O(20)	1.436(8),	1.449(8)	C(3)—C(4)—O(20)	109.3(5),	107.3(5)
C(5)—C(6)	1.557(11),	1.503(11)	C(3)—C(4)—C(5)	108.5(6),	110.1(5)
C(6)—C(7)	1.527(11),	1.520(11)	C(20)—C(4)—O(20)	109.8(5),	109.7(5)
C(7)—C(8)	1.319(11),	1.323(10)	C(20)—C(4)—C(5)	109.2(6),	109.5(5)
C(8)—C(9)	1.453(11),	1.520(10)	O(20)—C(4)—C(5)	107.4(5),	107.5(5)
C(8)—C(19)	1.557(11),	1.509(11)	C(4)—C(5)—C(6)	114.2(6),	116.9(6)
C(9)—C(10)	1.576(12),	1.561(11)	C(5)—C(6)—C(7)	112.0(6),	111.6(7)
C(10)—C(11)	1.497(12),	1.502(11)	C(6)—C(7)—C(8)	125.8(8),	129.2(7)
C(11)—C(12)	1.338(10),	1.317(10)	C(7)—C(8)—C(9)	126.0(7),	125.8(7)
C(12)—C(13)	1.482(10),	1.526(10)	C(7)—C(8)—C(19)	118.2(7),	117.8(7)
C(12)—C(18)	1.530(10),	1.530(10)	C(9)—C(8)—C(19)	115.8(7),	116.4(7)
C(13)—C(14)	1.535(12),	1.544(11)	C(8)—C(9)—C(10)	113.8(7),	113.5(6)
C(15)—C(16)	1.520(9),	1.485(10)	C(9)—C(10)—C(11)	113.6(7),	112.3(6)
C(15)—C(17)	1.506(10),	1.532(10)	C(10)—C(11)—C(12)	131.2(7),	127.5(6)
			C(11)—C(12)—C(13)	123.5(7),	124.9(6)
			C(11)—C(12)—C(18)	120.7(6),	119.9(6)
			C(13)—C(12)—C(18)	115.8(6),	115.2(7)
			C(12)—C(13)—C(14)	113.9(6),	114.4(6)
			C(13)—C(14)—C(1)	111.6(6),	112.8(6)
			C(1)—C(15)—C(16)	111.1(5),	113.9(6)
			C(1)—C(15)—C(17)	116.0(5),	113.4(5)
			C(1)—C(15)—O(3)	104.4(5),	104.9(5)
			C(16)—C(15)—C(17)	111.6(6),	109.7(6)
			C(16)—C(15)—O(3)	107.3(5),	107.3(5)
			C(17)—C(15)—O(3)	105.7(5),	107.2(5)

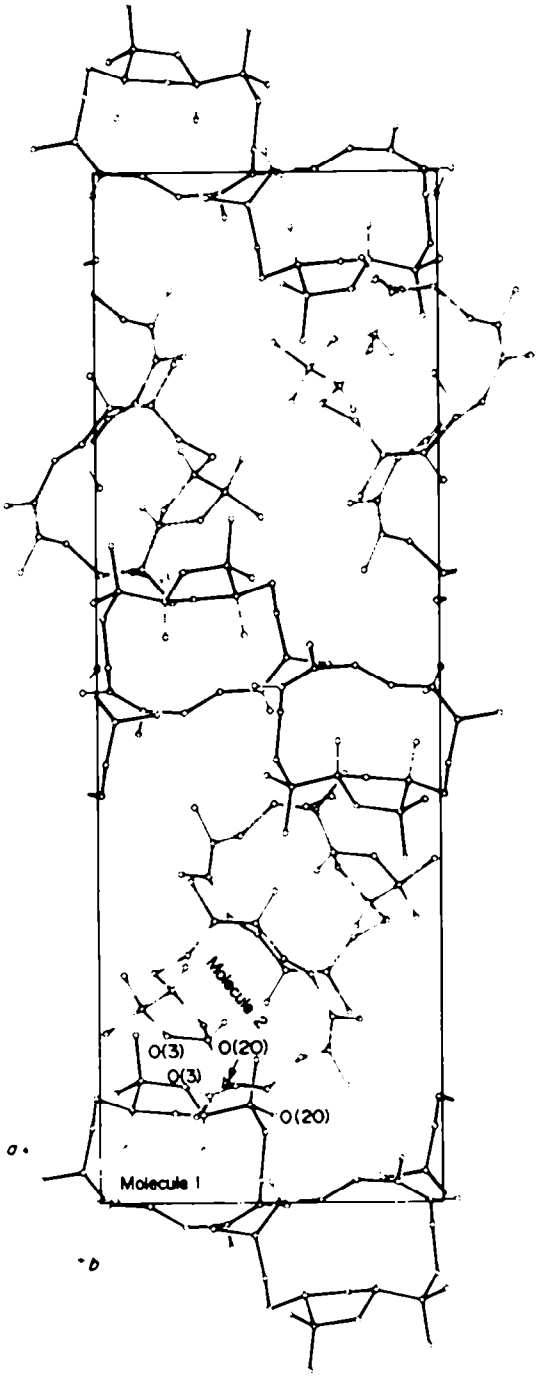

Fig. 1. Unit cell contents projected down c; tertiary and hydroxyl hydrogen atoms only are shown, attached to the molecular skeleton by line bonds. Numbering of the oxygen atoms is given. Hydrogen bonds are shown as dotted lines. Molecule I has solid bonds.

of the form

$$\exp(-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{23}klb^*c^*))$$

for the non-hydrogen atoms; for the hydrogen atoms U was constrained at a value fixed by consideration of the thermal motion of the parent carbon. Not all methyl hydrogen atoms would refine meaningfully and in some cases could not even be located in difference maps. The

Table 3. Non-hydrogen skeletal torsion angles (deg.) for molecules I and II respectively

C(14)—C(1)—C(2)—C(3)	−166.2, −169.0
C(15)—C(1)—C(2)—C(3)	−40.5, −39.7
C(1)—C(2)—C(3)—O(3)	39.4, 36.6
C(1)—C(2)—C(3)—C(4)	161.4, 161.3
C(2)—C(3)—O(3)—C(15)	−22.7, −19.3
C(4)—C(3)—O(3)—C(15)	−147.5, 148.4
C(3)—O(3)—C(15)—C(1)	−3.2, −6.3
C(3)—O(3)—C(15)—C(16)	−121.1, −127.7
C(3)—O(3)—C(15)—C(17)	119.6, 114.5
C(2)—C(1)—C(15)—O(3)	27.4, 29.4
C(2)—C(1)—C(15)—C(16)	142.7, 146.4
C(2)—C(1)—C(15)—C(17)	−88.4, −87.2
C(14)—C(1)—C(15)—O(3)	153.1, 156.8
C(14)—C(1)—C(15)—C(16)	−91.6, −86.2
C(14)—C(1)—C(15)—C(17)	37.3, 40.2
C(2)—C(3)—C(4)—C(5)	51.8, 51.0
C(2)—C(3)—C(4)—C(20)	69.2, −71.6
C(2)—C(3)—C(4)—O(20)	168.5, 167.7
O(3)—C(3)—C(4)—C(5)	170.1, 172.6
O(3)—C(3)—C(4)—C(20)	49.2, 50.1
O(3)—C(3)—C(4)—O(20)	−73.1, −70.7
C(3)—C(4)—C(5)—C(6)	57.6, 56.6
C(20)—C(4)—C(5)—C(6)	−179.4, −179.1
O(20)—C(4)—C(5)—C(6)	60.4 − 60.0
C(4)—C(5)—C(6)—C(7)	−157.4, −149.0
C(5)—C(6)—C(7)—C(8)	110.9, 114.0
C(6)—C(7)—C(8)—C(9)	0.6, 1.6
C(6)—C(7)—C(8)—C(19)	−179.9, 176.0
C(7)—C(8)—C(9)—C(10)	−118.0, −122.1
C(19)—C(8)—C(9)—C(10)	62.4, 55.5
C(8)—C(9)—C(10)—C(11)	172.9, 167.2
C(9)—C(10)—C(11)—C(12)	−124.9, −123.7
C(10)—C(11)—C(12)—C(13)	1.2, −2.5
C(10)—C(11)—C(12)—C(18)	−177.5, 180.0
C(11)—C(12)—C(13)—C(14)	118.1, 120.7
C(18)—C(12)—C(13)—C(14)	−63.1, −61.6
C(12)—C(13)—C(14)—C(1)	−61.3, −56.2
C(13)—C(14)—C(1)—C(2)	57.1, 59.9
C(13)—C(14)—C(1)—C(15)	−175.6, 178.6

latter were presumed disordered and ignored, while the remainder were fixed as invariants in the refinement at the position observed in the difference map. Refinement terminated with all parameter shifts $<0.2\sigma$. The residuals were

$$R = \Sigma\|Fo| - |Fc\|/\Sigma|Fo| = 0.060,$$

$$R' = (\Sigma w(|Fo| - |Fc|)^2/\Sigma w|Fo|^2)^{1/2} = 0.072.$$

In a weighting scheme of the form

$$w = (\sigma^2(Fo) + n \times 10^4(Fo)^2)^{-1}$$

the optimum value of n was found to be 5.

The results are given in the Tables and Figures, tables of structure amplitudes are deposited with the Editor.

DISCUSSION

The unit cell contents comprise discrete molecules of epoxycembradienol, the asymmetric unit containing two independent molecules; although these molecules have the same configuration as expected, their conformations differ surprisingly little (Table 3) in spite of the large, supposedly flexible ring system. The only abnormally close intermolecular contact is the hydrogen bond H(20d,

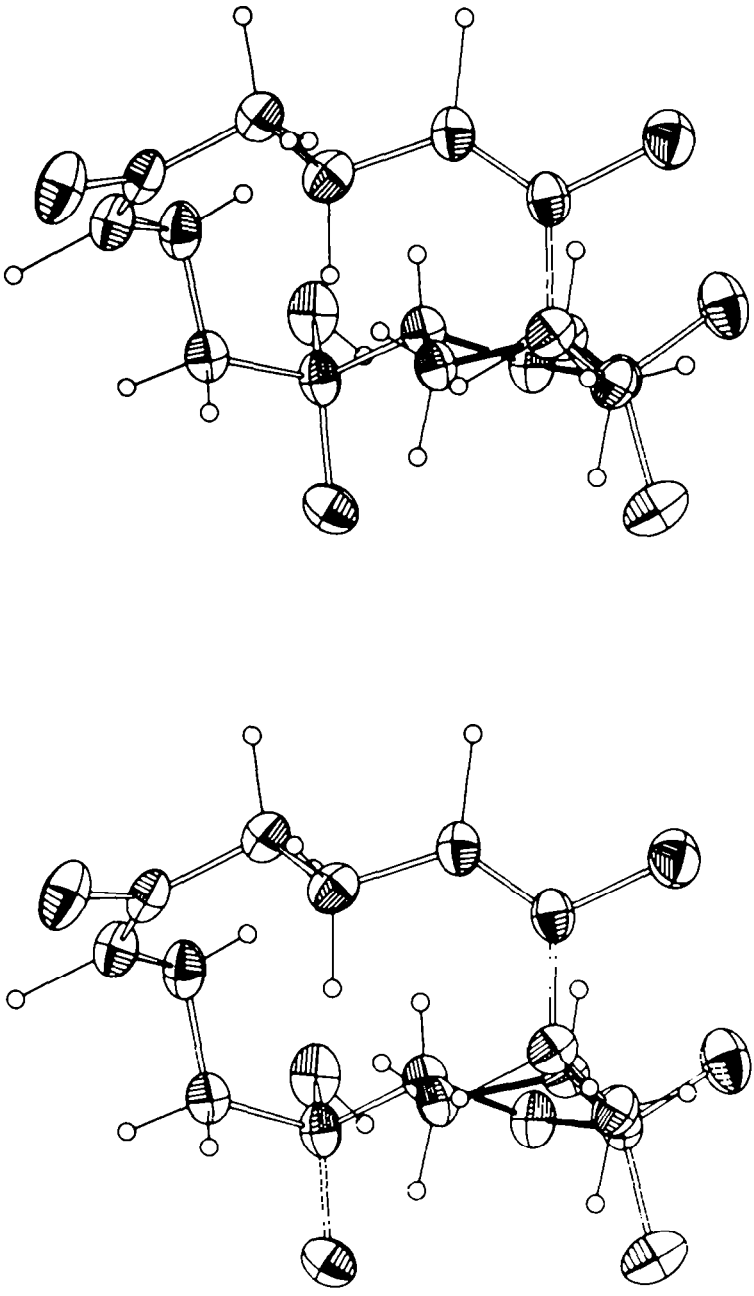

Fig. 2(a). *Stereoscopic projection of molecule I showing 50% thermal ellipsoids; the bonds in the epoxide ring are shown as solids for clarity. Methyl hydrogen atoms are omitted for the same reason.*

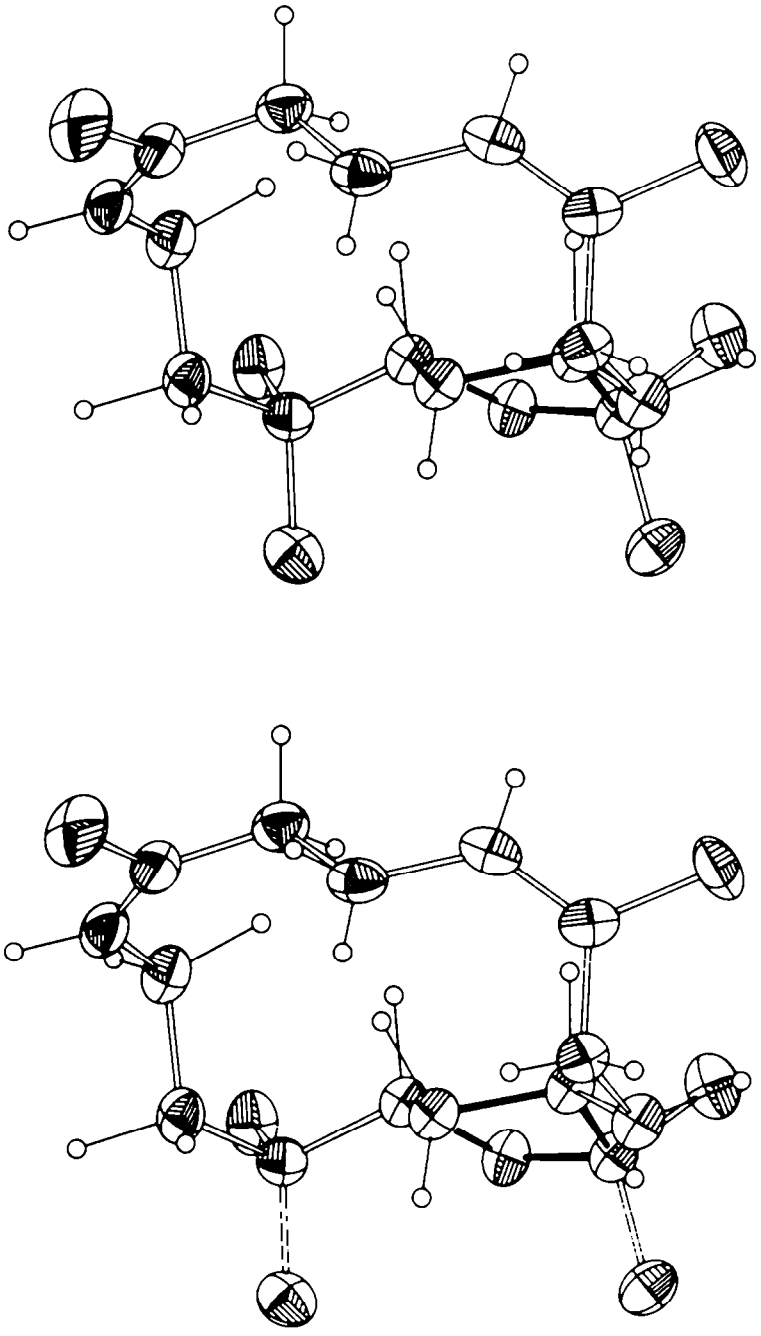

Fig. 2(b). Stereoscopic projection of molecule II shown similarly.

II)...O(3, I) (x, y, $z-1$), 1.99(6)Å, shown in Fig. 1. The following points are of interest:

(i) the double bonds are *cis*; this is not usual in a ring this size derived from a natural product.

(ii) the absolute configuration at C(1) has been established by chemical degradation to (−) homoterpenylketone,[1] known to have the *R* configuration; the absolute configuration of the molecule is thus established and shown in Fig. 1.

(iii) the two hydrogen atoms on C(1) and C(3) are shown to be *cis*. Bond lengths and angles are otherwise as expected.

(iv) the stereochemistry at C(4) was not established unambiguously from the chemical work and has been shown to be *R*.

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REFERENCES

[1]E. L. Ghisalberti, P. R. Jefferies, J. R. Knox and P. N. Sheppard, *Tetrahedron* **33**, 3301 (1977).
[2]D. T. Cromer and J. B. Mann; *Acta Cryst.* **A24**, 321 (1968).
[3]D. T. Cromer and D. Liberman, *J. Chem. Phys.* **53**, 1891 (1970).
[4]R. F. Stewart, E. R. Davidson and W. T. Simpson, *J. Chem. Phys.* **42**, 3175 (1965).
[5]G. Germain, P. Main and M. A. Woolfson, *Acta Cryst.* **B26**, 274 (1970).
[6]"The X-Ray System-Version of June, 1972", Technical Report TR-192 of the Computer Science Centre, University of Maryland, U.S.A.