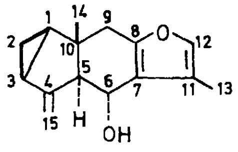
Crystal and Molecular Structure of Lindenenol

By **Yoshiko Mizuno-Tsukuda** and **Hirozo Koyama,*** Shionogi Research Laboratory, Shionogi and Co. Ltd., Fukushima-ku, Osaka, 553 Japan

The crystal structure of the title compound has been determined from a three-dimensional *X*-ray analysis. Crystals are orthorhombic, space group $P2_12_12_1$, $a = 21{\cdot}008(6)$, $b = 9{\cdot}051(3)$, $c = 13{\cdot}521(4)$ Å; $Z = 8$, and hence there are two molecules per asymmetric unit. The structure was solved by the symbolic addition procedure, and refined by block-diagonal least-squares calculations to a final R of 0·063 for 1816 independent observed reflexions. The two molecules in the asymmetric unit have nearly identical geometries. Each molecule contains four rings: furan, cyclohexene *trans*-fused to cyclopentane, and cyclopropane. The cyclopentane ring has an envelope conformation and the cyclohexene ring is in the half-chair form. Both molecules are stacked along the *b* axis parallel to each other with an interplanar spacing of *ca.* 3·8 Å.

LINDENENOL, $C_{15}H_{18}O_2$, a furosesquiterpenoid isolated[1] from the root of *Lindera strychnifolia*, was assigned[2,3] the structure (I) on the basis of chemical and spectroscopic investigations. This *X*-ray analysis was undertaken in order to confirm the structural assignment and to obtain more detailed information of the molecular conformation.



(I)

EXPERIMENTAL

Crystals were grown from acetic anhydride–hexane as prisms (m.p. 143—145 °C) elongated along the *b* axis. Precession and Weissenberg photographs were taken with Cu-K_α ($\lambda = 1{\cdot}5418$ Å) radiation; space group and unit-cell dimensions were determined from precession photographs and refined from the positions of a number of reflexions determined on the automatic diffractometer.

Crystal Data.—$C_{15}H_{18}O_2$, $M = 230{\cdot}3$. Orthorhombic, $a = 21{\cdot}008(6)$, $b = 9{\cdot}051(3)$, $c = 13{\cdot}521(4)$ Å, $U = 2571$ Å^3, $D_m = 1{\cdot}184$ (by flotation), $Z = 8$, $D_c = 1{\cdot}189$, $F(000) = 992$. Space group $P2_12_12_1$ (D_2^4) from systematic absences: $h00$ when h is odd, $0k0$ when k is odd, $00l$ when l is odd. Mo-K_α radiation, $\lambda = 0{\cdot}7107$ Å; μ(Mo-K_α) = 0·836 cm^{-1}.

Three-dimensional intensity data were collected on a Hilger and Watts automatic four-circle Y 290 diffractometer controlled by a PDP 8 computer. Integrated intensities were measured for $\theta \leqslant 27{\cdot}5°$ by the θ—2θ scan technique by use of Mo-K_α radiation and a scintillation counter (with a zirconium filter and pulse-height analyser). Each reflexion was integrated in 80 steps of intervals of 0·01°. A standard reflexion was monitored every 10 reflexions. The scan counts (P) and background counts (B_1, B_2) were combined to yield the integrated intensities $I = P - 2(B_1 + B_2)$. Thus 3334 independent reflexions were recorded, of which 1816 having intensities $I \geqslant 2\sigma(I)$ were considered observed. All intensities were corrected for Lorentz and polarization factors, and the normalized structure factor $|E|$ as well as structure amplitudes $|F|$ were derived. No absorption corrections were applied since the specimen was considered to be sufficiently small (0·30 × 0·30 × 0·33 mm).

Structure Analysis.—The structure was solved by the symbolic addition procedure[4] for the determination of phases in non-centrosymmetric space groups.

The origin was specified by assignment of phases to three reflexions; 3,2,0, $|E| = 4{\cdot}18$, $\phi = \pi/2$; 7,0,11, $|E| = 2{\cdot}56$, $\phi = -\pi/2$; and 3,3,0, $|E| = 2{\cdot}97$, $\phi = -\pi/2$; one further reflexion (2,2,1, $|E| = 4{\cdot}13$) was assigned $\phi = a$, and one additional reflexion (6,4,0, $|E| = 2{\cdot}89$) was given the phase π from the Σ_1 relationship.

These 5 reflexions were expanded manually into a set of 32 reflexions with $|E| \leqslant 1{\cdot}5$. A measure of the reliability of the Σ_2 relationship[5] can be obtained by use of the variance, equations (3.33) of ref. 6.

The 32 initial phases were used as input into the tangent formula (1). The input phases were refined by one iteration

$$\tan \phi_{\vec{h}} = \frac{[\sum_{k} |E_{\vec{k}} E_{\vec{h}-\vec{k}}| \sin (\phi_{\vec{k}} + \phi_{\vec{h}-\vec{k}})]}{[\sum_{k} |E_{\vec{k}} E_{\vec{h}-\vec{k}}| \cos (\phi_{\vec{k}} + \phi_{\vec{h}-\vec{k}})]} \qquad (1)$$

and then phases for additional reflexions were obtained by the use of (1). A total of 763 reflexions with $|E| > 1{\cdot}2$ were used to compute E maps.

The map showed 10 relatively large peaks which were used as a basis for a partial structure determination. The remaining atoms were located by a recycling procedure[7] using phases from the known partial structure in the tangent formula to generate phases for other reflexions with $|E| > 1{\cdot}0$. The preliminary co-ordinates of the molecules, which were obtained after the fifth E map, were refined by use of the IBM 1620 program of King.[8] Several cycles of refinement with isotropic temperature factor reduced R to 0·101. At this stage a difference-Fourier synthesis was calculated from which the positions of the 36 hydrogen atoms were found and their positions (only) were allowed to refine subsequently (B values were fixed at 1·99 Å^2). The final block-diagonal refinement, which was performed on a CDC 6600 computer, decreased R to 0·063 for the 1816 observed reflexions. A weighting scheme was applied, *i.e.*, $w = (a + b|F_o| + c|F_o|^2)^{-1}$ with $a = 1{\cdot}25$, $b = -0{\cdot}033$, and $c = 0{\cdot}001$.

Observed and calculated structure amplitudes are listed in Supplementary Publication No. SUP 20949 (6 pp., 1 microfiche).† Atomic scattering factors were taken from

---

† See Notice to Authors No. 7 in *J.C.S. Perkin II*, 1972, Index issue.

[1] H. Kondo and T. Sanada, *Yakugaku Zasshi*, 1925, **45**, 1047.

[2] K. Takeda, *Pharm. Bull.* (*Japan*), 1953, **1**, 244.

[3] K. Takeda and M. Ikuta, *Tetrahedron Letters*, 1964, 277.

[4] J. Karle and I. L. Karle, *Acta Cryst.*, 1966, **21**, 849.

[5] J. Karle and H. Hauptman, *Acta Cryst.*, 1961, **14**, 217.

[6] J. Karle and H. Hauptman, *Acta Cryst.*, 1956, **9**, 635.

[7] J. Karle, *Acta Cryst.*, 1968, **B24**, 182.

[8] G. S. D. King, IBM 1620 Least-squares refinement program, ERA 302, 1963.

ref. 9 for non-hydrogen atoms and from ref. 10 for hydrogen atoms.

RESULTS AND DISCUSSION

Final positional and thermal parameters with their estimated standard deviations are given in Tables 1 and 2. The configuration of the two crystallographically independent molecules of lindenenol are illustrated[11] in Figure 1. The two molecules have very similar conformations and the skeleton of each molecule consists of four rings, as predicted from the chemical evidence: furan, cyclohexene, cyclopentane, and cyclopropane.

For purposes of comparison, values are given for the two molecules of the asymmetric unit, labelled A and B, those for the latter being in square brackets.

TABLE 1

Final fractional atomic co-ordinates and anisotropic thermal parameters (all × 10^4),* with estimated standard deviations in parentheses

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
(*a*) Molecule (A)									
O(1)	6427(2)	2229(6)	4165(3)	23(1)	189(8)	65(3)	9(2)	5(1)	1(4)
O(2)	5042(2)	3656(4)	6599(2)	28(1)	107(5)	38(2)	−9(2)	0(1)	−4(3)
C(1)	4397(3)	4771(8)	3251(4)	32(2)	148(10)	45(3)	13(4)	2(2)	−2(5)
C(2)	3690(4)	4625(9)	3168(5)	36(2)	187(13)	63(4)	10(5)	−2(2)	5(7)
C(3)	3970(3)	5514(7)	3992(5)	28(1)	119(10)	68(4)	5(4)	−0(2)	13(5)
C(4)	4081(3)	4778(7)	4969(5)	23(1)	129(9)	56(4)	1(3)	−2(2)	−2(5)
C(5)	4774(2)	4123(6)	4899(4)	20(1)	104(7)	41(3)	−2(3)	1(1)	−2(4)
C(6)	4994(3)	3003(6)	5626(4)	26(1)	81(7)	36(3)	−12(3)	0(2)	3(4)
C(7)	5636(2)	2536(7)	5251(4)	19(1)	106(8)	51(3)	−5(3)	−1(2)	−4(5)
C(8)	5830(3)	2761(7)	4320(5)	25(1)	114(8)	57(3)	1(3)	5(2)	−1(5)
C(9)	5480(3)	3529(7)	3494(4)	26(1)	132(9)	44(3)	1(3)	7(2)	2(5)
C(10)	4777(2)	3577(7)	3798(4)	21(1)	119(8)	37(3)	1(3)	−1(1)	−3(4)
C(11)	6153(3)	1765(7)	5753(5)	30(1)	103(9)	62(4)	1(3)	−0(2)	0(5)
C(12)	6605(3)	1644(8)	5056(6)	26(1)	147(11)	89(5)	8(4)	−3(2)	15(7)
C(13)	6183(3)	1289(8)	6800(5)	35(2)	145(11)	68(4)	5(4)	−4(2)	22(6)
C(14)	4484(3)	2029(7)	3681(4)	28(1)	120(9)	43(3)	1(3)	−0(2)	−16(5)
C(15)	3688(3)	4700(10)	5699(5)	27(2)	252(16)	68(5)	20(5)	6(2)	9(8)
(*b*) Molecule (B)									
O(1′)	6038(2)	7301(6)	4004(3)	40(1)	205(9)	39(2)	12(3)	3(1)	12(4)
O(2′)	5436(1)	6433(4)	7211(2)	20(0)	110(5)	43(2)	0(2)	1(1)	−6(3)
C(1′)	7579(3)	4939(10)	6354(5)	21(1)	230(14)	73(5)	8(4)	−0(2)	−51(7)
C(2′)	7997(3)	5215(11)	7212(6)	24(2)	271(17)	105(7)	22(5)	−9(3)	−41(9)
C(3′)	7390(3)	4342(9)	7360(5)	24(1)	219(14)	58(4)	19(4)	−5(2)	−17(7)
C(4′)	6812(3)	5217(8)	7665(5)	28(1)	171(11)	57(4)	10(4)	−6(2)	0(6)
C(5′)	6517(3)	5734(7)	6719(4)	22(1)	110(8)	43(3)	−1(3)	1(2)	−10(5)
C(6′)	6002(2)	6897(6)	6698(4)	19(1)	91(7)	37(3)	−3(3)	−0(1)	−12(4)
C(7′)	5867(2)	7247(6)	5632(4)	19(1)	105(8)	40(3)	−9(3)	3(1)	1(4)
C(8′)	6265(3)	6847(7)	4901(4)	26(1)	145(10)	47(3)	−6(3)	3(2)	1(5)
C(9′)	6886(3)	6042(8)	4970(5)	26(1)	166(11)	60(4)	0(3)	14(2)	−8(6)
C(10′)	7098(2)	6119(8)	6056(5)	17(1)	145(11)	64(4)	−0(3)	1(2)	−26(6)
C(11′)	5339(3)	8029(7)	5172(4)	27(1)	115(9)	51(3)	0(3)	−2(2)	0(5)
C(12′)	5482(3)	8000(8)	4202(5)	36(2)	138(10)	54(4)	12(4)	−5(2)	9(6)
C(13′)	4765(3)	8630(8)	5648(5)	30(2)	137(10)	56(4)	8(4)	−0(2)	3(6)
C(14′)	7333(3)	7700(10)	6258(6)	20(1)	211(14)	96(6)	−14(4)	12(2)	6(8)
C(15′)	6625(3)	5479(11)	8574(5)	33(2)	283(17)	60(4)	38(5)	−8(2)	−6(8)

* In the form: $T = \exp\{-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + 2\beta_{12}hk + 2\beta_{13}hl + 2\beta_{23}kl)\}$.

TABLE 2

Final fractional hydrogen atomic co-ordinates (× 10^3)

	Molecule (A)			Molecule (B) *		
	x	y	z	x	y	z
H[O(2)]	487	312	695	530	570	700
H(1)	464	530	282	768	429	588
H(2a)	359	537	262	820	457	734
H(2b)	352	358	324	805	632	736
H(3)	389	650	401	737	320	755
H(5)	504	497	501	635	485	642
H(6)	472	214	568	615	770	706
H(9a)	569	460	345	682	489	482
H(9b)	551	301	288	719	655	453
H(12)	700	115	513	524	865	371
H(13a)	608	206	719	451	787	604
H(13b)	656	83	697	448	914	514
H(13c)	581	54	703	486	940	623
H(14a)	475	130	396	703	854	601
H(14b)	439	184	296	771	790	589
H(14c)	411	203	403	745	773	686
H(15a)	326	496	558	683	504	913
H(15b)	372	418	633	629	614	873

* Atom labels for molecule (B) are primed: H[O(2′)], H(1′) *etc.*

Bond distances and valency angles are given in Table 3, together with their estimated standard deviations computed from the least-squares residuals. For non-hydrogen atoms the mean estimated standard deviations are 0·009 Å for bond distances and 0·5° for angles.

Although the C–C single bonds vary over a wide range (1·455—1·570 Å), for the most part they do not differ significantly from their expected values.[12] Except for the cyclopropane and furan rings, the mean C(sp^3)–C(sp^3) is 1·536 [1·532] and C(sp^2)–C(sp^3) is 1·500 Å. The C–C bond distances (mean 1·500 Å) in the cyclopropane ring are similar to those found in a recent structure determination.

[9] P. A. Doyle and P. S. Turner, *Acta Cryst.*, 1968, **A24**, 390.

[10] R. F. Stewart, E. R. Davidson, and W. I. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

[11] C. K. Johnson, ORTEP, A Fortran Thermal-Ellipsoid Plot Program, Oak Ridge National Laboratory, Tennessee, Report ORNL 3794, 1965.

[12] L. E. Sutton, *Chem. Soc. Special Publ.*, No. 18, 1965.

The mean terminal Me bond distance at C(11) is 1·476 Å which is noticeably shorter than that of 1·538 Å at C(10).

Bond distances involving hydrogen atoms are also given in Table 3(*c*). The mean values of 0·98 Å for C–H and 0·77 Å for O–H are less than the internuclear separations measured spectroscopically. The bond angles in the cyclopropane ring are close to their expected value (*ca.* 60°). The error in other C–C–C bond angles is probably closer to 3° than to the estimated 1·5—2·0° obtained from the errors given in Table 3.

TABLE 3

Interatomic distances (Å) and angles (°), with standard deviations in parentheses. Values are given for molecule (A), with those for molecule (B) in square brackets

(*a*) Bond distances

O(1)–C(8)	1·359(8) [1·366(8)]
O(1)–C(12)	1·368(9) [1·355(9)]
O(2)–C(6)	1·445(7) [1·440(7)]
C(1)–C(2)	1·494(11) [1·475(11)]
C(1)–C(3)	1·503(10) [1·516(10)]
C(1)–C(10)	1·533(9) [1·525(10)]
C(2)–C(3)	1·495(10) [1·513(11)]
C(3)–C(4)	1·498(9) [1·507(10)]
C(4)–C(5)	1·517(8) [1·495(9)]
C(4)–C(15)	1·288(10) [1·312(10)]
C(5)–C(6)	1·506(8) [1·509(8)]
C(5)–C(10)	1·570(8) [1·553(8)]
C(6)–C(7)	1·502(8) [1·501(8)]
C(7)–C(8)	1·338(9) [1·344(8)]
C(7)–C(11)	1·458(9) [1·455(8)]
C(8)–C(9)	1·506(9) [1·497(9)]
C(9)–C(10)	1·533(8) [1·535(9)]
C(10)–C(14)	1·538(9) [1·537(11)]
C(11)–C(12)	1·341(10) [1·345(9)]
C(11)–C(13)	1·481(10) [1·471(9)]

(*b*) Bond angles

C(8)–O(1)–C(12)	104·7(5) [105·4(5)]
C(2)–C(1)–C(3)	59·8(5) [60·7(5)]
C(2)–C(1)–C(10)	119·4(6) [118·9(7)]
C(3)–C(1)–C(10)	107·7(5) [108·2(6)]
C(1)–C(2)–C(3)	60·3(5) [60·9(5)]
C(1)–C(3)–C(2)	59·8(5) [58·2(5)]
C(1)–C(3)–C(4)	107·2(5) [105·5(6)]
C(2)–C(3)–C(4)	118·5(6) [116·1(7)]
C(3)–C(4)–C(5)	105·1(5) [105·3(5)]
C(3)–C(4)–C(15)	126·8(6) [126·3(7)]
C(5)–C(4)–C(15)	127·9(6) [128·3(7)]
C(4)–C(5)–C(6)	122·7(5) [122·1(5)]
C(4)–C(5)–C(10)	102·8(4) [103·8(5)]
C(6)–C(5)–C(10)	113·0(5) [113·2(5)]
O(2)–C(6)–C(5)	110·0(4) [112·3(4)]
O(2)–C(6)–C(7)	111·0(4) [111·5(4)]
C(5)–C(6)–C(7)	106·3(4) [107·5(4)]
C(6)–C(7)–C(8)	123·2(5) [121·9(5)]
C(6)–C(7)–C(11)	130·2(5) [131·1(5)]
C(8)–C(7)–C(11)	106·4(5) [106·8(5)]
O(1)–C(8)–C(7)	111·7(5) [110·7(5)]
O(1)–C(8)–C(9)	119·9(5) [120·3(5)]
C(7)–C(8)–C(9)	128·2(6) [128·8(6)]
C(8)–C(9)–C(10)	106·4(5) [106·8(5)]
C(1)–C(10)–C(5)	102·2(4) [102·1(5)]
C(1)–C(10)–C(9)	113·0(5) [114·3(6)]
C(1)–C(10)–C(14)	112·5(5) [113·0(5)]
C(5)–C(10)–C(9)	107·8(4) [108·3(5)]
C(5)–C(10)–C(14)	111·5(4) [111·0(5)]
C(9)–C(10)–C(14)	109·3(5) [107·7(6)]
C(7)–C(11)–C(12)	103·8(6) [103·7(5)]
C(7)–C(11)–C(13)	127·9(6) [128·0(5)]
C(12)–C(11)–C(13)	128·0(6) [128·0(6)]
O(1)–C(12)–C(11)	113·0(6) [113·1(6)]

TABLE 3 (*Continued*)

(*c*) Bond distance associated with hydrogen positions

O(2)–H(O2)	0·77 [0·76]
C(1)–H(1)	0·91 [0·89]
C(2)–H(2a)	1·02 [0·74]
C(2)–H(2b)	1·01 [1·03]
C(3)–H(3)	0·90 [1·06]
C(5)–H(5)	1·00 [0·94]
C(6)–H(6)	0·96 [0·94]
C(9)–H(9a)	1·07 [1·06]
C(9)–H(9b)	0·94 [0·99]
C(12)–H(12)	0·95 [1·01]
C(13)–H(13a)	0·90 [1·02]
C(13)–H(13b)	0·92 [1·01]
C(13)–H(13c)	1·06 [1·07]
C(14)–H(14a)	0·94 [1·03]
C(14)–H(14b)	1·00 [0·96]
C(14)–H(14c)	0·90 [0·86]
C(15)–H(15a)	0·92 [0·95]
C(15)–H(15b)	0·98 [0·95]

Within these limits of error the molecule shows no unusual geometric features.

The displacements of atoms from some least-squares mean planes are listed in Table 4. The furan ring and *exo*-methylene group in both molecules are quite planar. The atoms C(9) and C(13) in both molecules are slightly displaced by 0·021 [0·023] and 0·024 [−0·059] Å from the mean plane of the furan ring. With respect to a plane through C(6)—(9), C(5) is displaced by 0·408 [−0·393] and C(10) by −0·416 [0·414] Å so that the cyclohexene ring in both molecules has a half-chair conformation. These deviations agree well with values of 0·399 and −0·361 Å observed recently.[13] The cyclopentane ring in both molecules has an envelope conformation, which appears to be the usual situation.[14,15] In order to obtain detailed information on the molecular shape and conformational features of (A) and (B), the displacements from the planes through C(7), C(8), C(11) in the furan ring and C(1), C(3), C(4) in the cyclopentane ring were calculated, and are shown in Figures 2 and 3. The cyclopentane ring in both molecules is

TABLE 4

Displacements (Å) of atoms from various planes for molecule (A); values for molecule (B) in parentheses

Plane (*a*): O(1), C(7), C(8), C(11), C(12)
O(1) −0·001 [0·001], C(6) −0·003 [0·006], C(7) 0·005 [−0·001], C(8) −0·002 [0], C(9) 0·021 [0·023], C(11) −0·006 [0·001], C(12) 0·005 [−0·001], C(13) 0·024 [−0·059]

Plane (*b*): C(6)—(9)
C(5) 0·408 [−0·393], C(6) −0·003 [−0·001], C(7) 0·008 [0·002], C(8) −0·008 [−0·002], C(9) 0·003 [0·001], C(10) −0·416 [0·414]

Plane (*c*): C(1), C(3), C(4), C(10)
C(1) −0·003 [0·012], C(2) −1·181 [1·190], C(3) 0·003 [−0·011], C(4) −0·002 [0·007], C(5) 0·585 [−0·573], C(10) 0·002 [−0·007], C(15) −0·441 [0·469]

Plane (*d*): C(3)—(5), C(15)
C(3) −0·002 [0·002], C(4) 0·006 [−0·007], C(5) −0·002 [0·002], C(15) −0·002 [0·003]

---

[13] D. M. Hawley and G. Ferguson, *J. Chem. Soc.* (*B*), 1971, 843.

[14] F. H. Herbstein and H. Reger, *J. Chem. Soc.* (*B*), 1971, 1696.

[15] D. M. Hawley, G. Ferguson, T. F. W. McKillop, and J. M. Robertson, *J. Chem. Soc.* (*B*), 1969, 599.

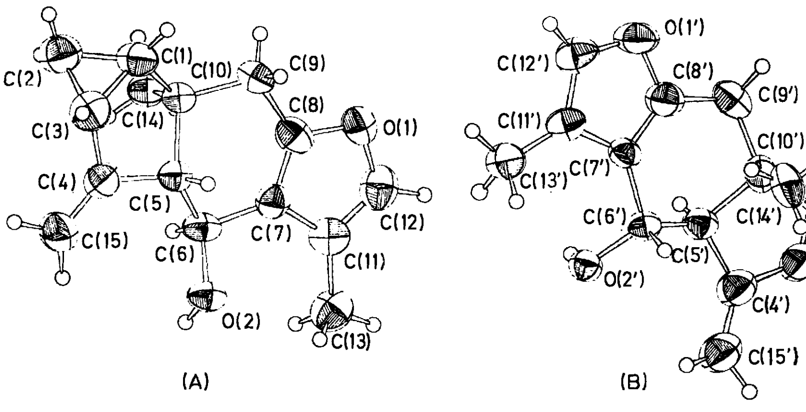


FIGURE 1 Perspective drawings of the two molecules of lindenenol

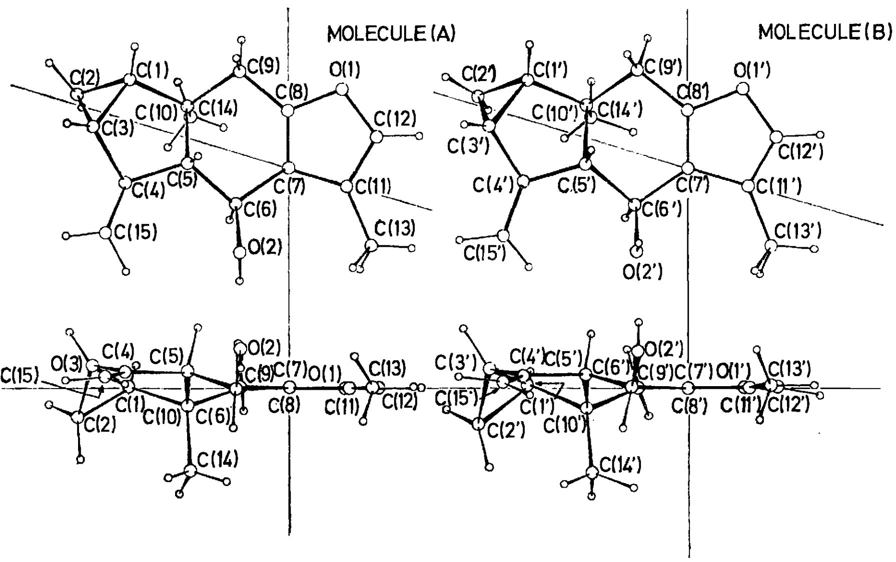


FIGURE 2 Projections of the molecules on the C(8), C(7), and C(11) plane

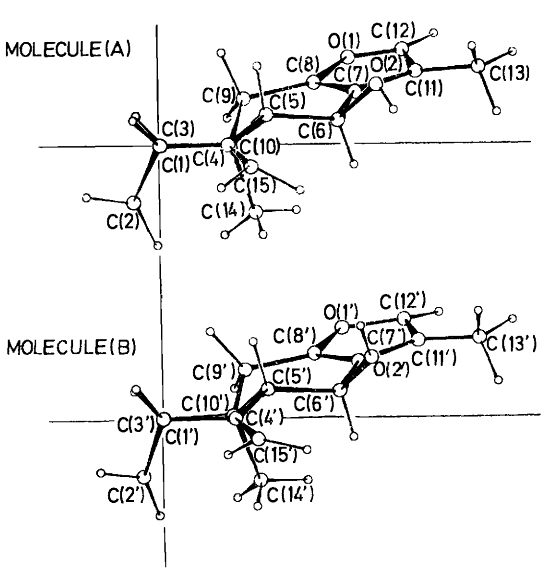


FIGURE 3 Projections of the molecules perpendicular to the C(1), C(3), and C(4) plane

cis-fused to the cyclopropane ring and joins the cyclohexene ring with a *trans*-juncture. The atoms C(2), C(14) and O(2), H(5) are on the opposite side of the ring system of the furosesquiterpenoid skeleton. The bicyclohexene system along the best plane through atoms C(1), C(3), C(4), C(10) has an overall chair-like conformation. The dihedral angles between the planes defined by C(1), C(3), C(4), C(10) and the cyclopropane ring and the plane C(4), C(5), C(10) are **113·8** [112·5] and 142·5 [142·3]° respectively. The value of **113·8** [112·5]° is in good agreement with those for other compounds [16,17] involving the cyclopropane ring.

The major conformational differences in the two molecules are hydrogen positions at O(2). The orientation of the hydrogen atom attached to O(2) is described by the torsion angle H[O(2)]–O(2)–C(6)–H(6), which was 3 [170]°. This means that the position of the hydrogen

[16] L. J. Guggenberger and R. A. Jacobson, *Acta Cryst.*, 1969, **B25**, 888.

[17] A. F. Cameron, G. Ferguson, and J. M. Robertson, *J. Chem. Soc.* (*B*), 1969, 692.

atom at O(2) in molecule (A) is *cis* to the hydrogen atom at C(6), while in molecule (B) it is *trans*.

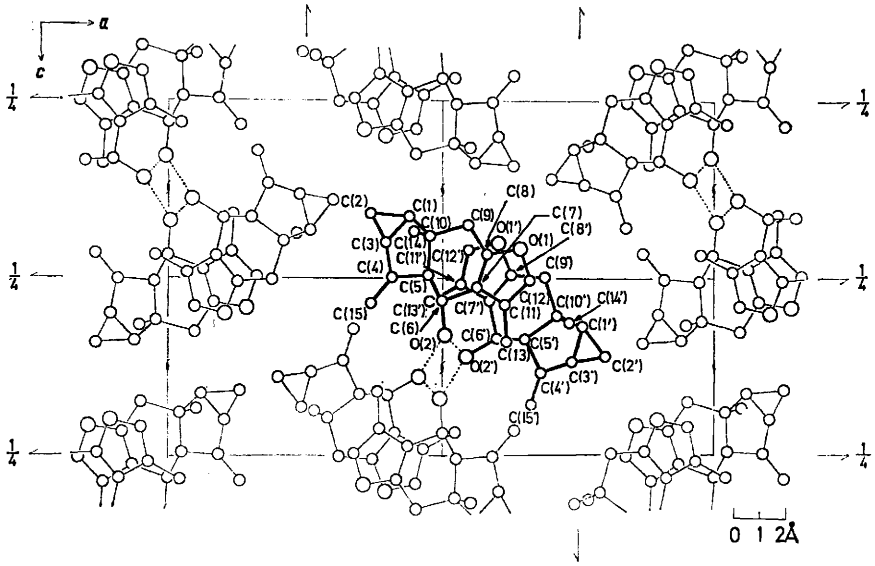


FIGURE 4 The molecular packing as viewed along *b* axis

The molecular packing arrangement along the *b* axis is illustrated in Figure 4. Two molecules are stacked along the *b* axis parallel to each other, with interplanar spacing *ca.* 3·8 Å. The two molecules are linked by two intermolecular hydrogen bonds, O(2) · · · H–O(2') (2·773 Å) and O(2)–H · · · O(2'^{I}) (2·765 Å), which run in a helical fashion around a screw axis.

TABLE 5

Intermolecular distances (<3·6 Å)

O(2) · · · C(6')	3·562	O(2) · · · H–O(2')	2·773
O(2) · · · C(6'^{I})	3·558	O(2)–H · · · O(2'^{I})	2·765
C(6) · · · O(2'^{I})	3·373		
C(13') · · · C(15'^{II})	3·526		

Roman numerals as superscripts refer to the following equivalent positions relative to the reference molecule at x, y, z:

I $1 - x, y - \frac{1}{2}, \frac{3}{2} - 2$ II $1 - z, \frac{1}{2} + y, \frac{3}{2} - z$

All intermolecular distances were calculated, and the most significant contacts (⩽3·60 Å) are given in Table 5. The molecular packing is efficient with several contacts approaching the sum of the van der Waals radii; the shortest non-hydrogen contact is C(6) · · · O(2'^{I}) 3·373 Å. In both molecules, the hydrogen atom H(C2)$_b$ of the cyclopropane ring has short contacts with the hydrogen atom H(C14)$_c$ at C(14): 2·157 in (A) and 1·927 Å in (B).

We thank Dr. K. Takeda, Shionogi Research Laboratory, for suggesting this investigation, Dr. H. Tada and Miss Miyawaki for the preparation of suitable crystals, Drs. I. L. Kare and G. S. D. King for computational assistance and programs, and Miss K. Oda and Mr. H. Nakai for technical assistance.

[3/2216 *Received*, 29*th October*, 1973]