drawn with the *DEAM*-4 program written by Takenaka (1972).

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Structure and Absolute Configuration of the Sesquiterpene Lemnacarnol

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Abstract. Lemnacarnol, $C_{15}H_{24}O_3$, tetragonal, $a=b=9.208$ (1), $c=16.918$ (3) Å, $M=252.3$, $Z=4$, $D_m=1.169$ g cm^{-3}, μ(Cu $K\alpha$)$=6.4$ cm^{-1}. The compound was extracted from the scft coral *Lemnalia Carnosa* and its structure and absolute configuration have been determined by single-crystal X-ray diffraction.

Introduction. Chemical and spectroscopic investigations of lemnacarnol have been summarized by Tursch, Colin, Daloze, Losman & Karlsson (1975). Crystals were grown from ether. Weissenberg photographs indicated a tetragonal cell with systematic extinctions characteristic for space group $P4_1$ and its enantiomer $P4_3$. A crystal of $0.25 \times 0.30 \times 0.30$ mm, cut from a larger crystal, was fixed to a thin glass fibre (0.025 mm). Data for half the reciprocal sphere and $\theta < 60°$ were collected by ω–2θ scan on a Philips PW 1100 diffractom-eter with graphite monochromatized Cu $K\alpha$ radiation. Lp-corrected data were reduced to 1103 unique reflexions. The structure was solved by direct methods (*MULTAN*). The 24 H atoms were located by Fourier difference synthesis. The positional and thermal parameters for all C and O atoms and the thermal parameters for all H atoms were refined by full-matrix least squares to a conventional R value of 0.05, calculated from all data with unit weights.* Final parameters ($P4_3$) are given for C and O in Table 1 and for H in Table 2.

The correct space group, $P4_3$, was determined from

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* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31675 (3 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and vibrational components* ($\times 10^3$)

$$T=\exp\{-2\pi^2(U_{11}h^2a^{*2}+\ldots+2U_{12}hka^*b^*\ldots)\}.$$

	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(1)	3585 (6)	301 (7)	3816 (0)	34 (3)	48 (4)	22 (3)	−2 (3)	0 (3)	1 (3)
C(2)	2224 (6)	688 (7)	4259 (5)	29 (3)	46 (4)	38 (4)	10 (3)	−5 (3)	0 (3)
C(3)	2550 (6)	1217 (7)	5088 (5)	33 (3)	47 (4)	43 (4)	10 (3)	2 (3)	−11 (3)
C(4)	3720 (6)	296 (7)	5493 (5)	27 (3)	44 (4)	27 (3)	2 (3)	3 (3)	−6 (3)
C(5)	5179 (6)	320 (6)	5027 (5)	24 (3)	36 (3)	26 (3)	−1 (2)	3 (2)	−2 (3)
C(6)	6214 (6)	−901 (7)	5328 (5)	24 (3)	46 (3)	24 (3)	3 (2)	4 (2)	2 (3)
C(7)	7583 (6)	−1099 (6)	4804 (5)	22 (3)	36 (3)	32 (4)	−2 (2)	4 (2)	−3 (3)
C(8)	7596 (6)	−335 (8)	4015 (5)	27 (3)	70 (5)	28 (3)	3 (3)	8 (3)	0 (3)
C(9)	6132 (7)	−406 (9)	3623 (5)	29 (3)	112 (7)	23 (3)	2 (4)	4 (3)	−10 (4)
C(10)	4872 (6)	104 (7)	4138 (5)	23 (3)	42 (3)	23 (3)	−4 (2)	0 (2)	0 (3)
C(11)	5561 (7)	−2447 (7)	5381 (6)	35 (3)	49 (4)	63 (5)	−5 (3)	6 (3)	22 (4)
C(12)	6857 (8)	−3413 (8)	5184 (7)	50 (4)	48 (4)	90 (7)	−1 (3)	11 (5)	10 (4)
C(13)	4916 (9)	−2909 (10)	6179 (7)	67 (5)	97 (7)	95 (8)	15 (5)	38 (5)	59 (6)
C(14)	3865 (7)	758 (10)	6362 (5)	35 (4)	101 (3)	24 (4)	2 (3)	3 (3)	−15 (3)
C(15)	5922 (7)	1827 (7)	5145 (5)	44 (4)	41 (6)	46 (4)	−13 (4)	−5 (3)	−6 (4)
O(1)	7697 (5)	−2621 (4)	4637 (5)	52 (3)	40 (2)	67 (3)	−5 (2)	18 (3)	−11 (3)
O(2)	1288 (4)	−573 (5)	4332 (4)	24 (2)	70 (3)	36 (3)	−11 (2)	2 (2)	−10 (2)
O(3)	8812 (4)	−683 (5)	5266 (4)	24 (2)	58 (3)	30 (2)	0 (2)	0 (2)	0 (2)

anomalous dispersion measurements (Bijvoet, Peerdeman & van Bommel, 1951) with Cu $K\alpha$ radiation. The practical procedure used here for adequately measuring Bijvoet differences will be described elsewhere by the author. To confirm the Bijvoet differences by independent measurements, two crystals were used, that used for the data collection and another of about a quarter of the volume. The latter was enclosed in a capillary of diameter 0·30 mm, just large enough to accommodate the crystal. The space between the capillary and the crystal was completely filled with epoxy resin in order to prevent the crystal from moving and to give it a uniform surrounding. The results are given in Table 3 with standard deviations based on counting statistics. The anomalous dispersion parameters of Cromer & Liberman (1970) were used.

Discussion. The absolute configuration is shown in Fig. 1, bond lengths and angles are given in Fig. 2.
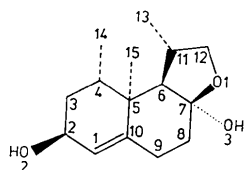

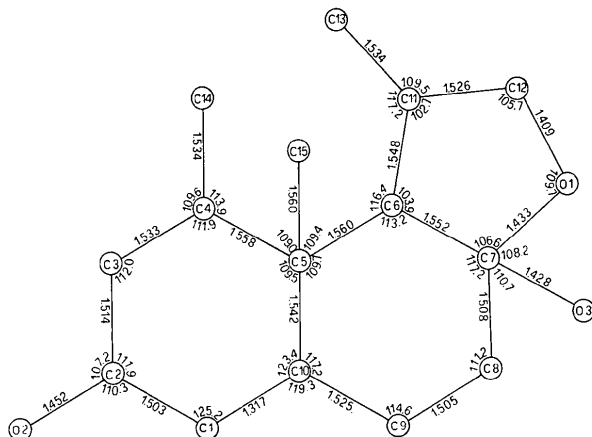Fig. 1. Lemnacarnol, absolute configuration.

Fig. 2. Lemnacarnol, bond distances and angles; the e.s.d.'s are 0·009 Å and 0·5°.

Fig. 3 is a stereoscopic drawing of lemnacarnol. Table 4 gives the dihedral angles of the three rings. The cyclohexene ring has the half-chair conformation and

Table 2. *Fractional coordinates* ($\times 10^3$) *and temperature factors* ($\times 100$) *for hydrogen*

$$T = \exp\left(-8\pi^2 U \sin^2 \theta / \lambda^2\right).$$

	x	y	z	U
HC(1)	354	10	330	2
HC(2)	174	159	400	5
H(1)C(3)	149	126	540	6
H(2)C(3)	285	220	508	4
HC(4)	336	−68	550	2
HC(6)	657	−61	590	2
H(1)C(8)	827	−7	370	2
H(2)C(8)	791	69	410	3
H(1)C(9)	590	−157	340	7
H(2)C(9)	611	−10	310	6
HC(11)	499	−259	500	3
H(1)C(12)	735	−364	565	7
H(2)C(12)	652	−451	505	8
H(1)C(13)	564	−270	655	6
H(2)C(13)	471	−418	615	18
H(3)C(13)	448	−221	660	17
H(1)C(14)	467	39	665	6
H(2)C(14)	311	52	665	3
H(3)C(14)	404	176	650	6
H(1)C(15)	658	216	480	9
H(2)C(15)	622	199	560	6
H(3)C(15)	534	281	510	8
HO(2)	98	−88	400	8
HO(3)	945	−70	500	8

Table 3. *Calculated* (X_c) *and observed* (X_o) *Bijvoet ratios* ($\times 100$) *with e.s.d.'s in parentheses*

Data measured from the crystal in the capillary are indicated by an asterisk.

h	k	l	F_o	X_c	X_o
0	3	1	12·9	−1·1	−1·5 (3)
*1	4	1	12·6	1·6	1·2 (4)
3	1	2	12·7	−1·0	−0·8 (3)
2	2	2	22·3	−0·8	−1·4 (2)
3	3	2	19·5	1·5	1·7 (3)
*3	3	2	19·5	1·5	1·2 (2)
3	3	3	11·5	2·3	0·4 (5)
0	2	4	42·5	0·4	1·2 (6)
4	2	4	16·2	−2·4	−1·2 (3)
*4	2	4	16·2	−2·4	−2·2 (4)
1	4	4	27·6	1·0	0·8 (2)
*2	4	4	11·1	3·9	1·1 (4)
*0	2	5	16·1	1·1	1·6 (3)
*0	4	12	18·8	1·4	0·0 (3)
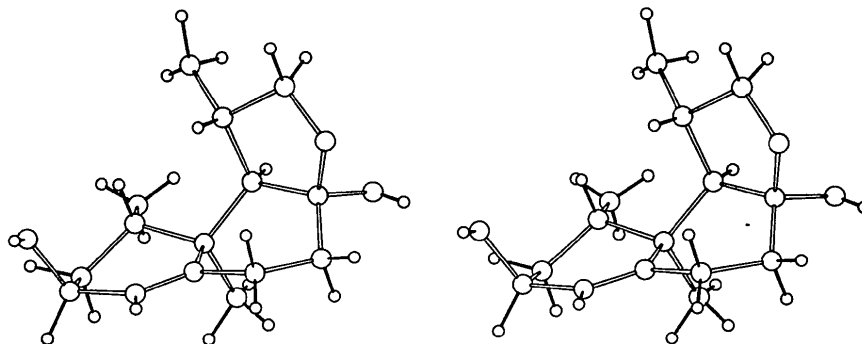
Fig. 3. Lemnacarnol, a stereoscopic view.

Table 4. *Dihedral angles* (°) *for the three rings*

1–2–3–4	−41	6–11–12–O	−33
2–3–4–5	63	11–12–O–7	33
3–4–5–10	−48	12–O–7–6	−18
4–5–10–1	18	O–7–6–11	3
5–10–1–2	−3	7–6–11–12	21
5–6–7–8	−12		
6–7–8–9	−40		
7–8–9–10	52		
8–9–10–5	−12		
9–10–5–6	−39		

the cyclohexane ring has a conformation between the boat and twisted-boat forms (Hendrickson, 1967). Both rings have only C_2 symmetry. The five-membered ring has the envelope conformation with atom C(12) lying 'below' the plane through atoms C(11), C(6), C(7) and O(1) depicted in Fig. 3. A conformation with C(12) 'above' the plane can be approximately generated (authors' program, unpublished) by a rotation of C(12) around the C(6)–C(11) axis until at 38·5° the C(12)–O(1) distance becomes the same. The HC(11)–HC(4) distance however is then decreased from 2·00 to 1·77 Å, based on calculated H positions. There are two intermolecular hydrogen bonds, O(2)–H···O(3)′ (1−y,

$x-1, z-\frac{1}{4})$ and O(3)–H···O(2)′ (1+x,y,z), 2·764 (9) and 2·766 (9) Å respectively. The torsion angles H–O–C–O′ are each 2°; this indicates that the H atoms are practically as close as possible to the O′ atoms, in keeping with the partially electrostatic nature of the hydrogen bond. The attraction H···O′ is partly balanced by the deviations, indicated by the torsion angles H–O(2)–C(4)–C(5) (65°) and H–O(3)–C(9)–C(8) (45°), of the H atoms from the staggered positions.

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p-Phenylenedi[α-cyanoacrylic Acid] Di-n-propyl Ester, a Photopolymerizable Crystal

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Abstract. $C_{20}H_{20}N_2O_4$, M.W. 352·4. Monoclinic, $P2_1/n$ (No. 14), $a = 5·341$ (2), $b = 26·112$ (7), $c = 6·882$ (2) Å, $\beta = 103·81$ (2)°. $Z = 2$, $D_x = 1·265$, $D_m = 1·262$ g cm^{-3}. The molecules are nearly planar and are piled up, with displacement in the direction of the long molecular axis, to form a parallel plane-to-plane stack along the c axis. In the stack double bonds related by the centre of symmetry are situated at the shortest distance of 3·931 Å.

Introduction. A group of diolefinic compounds are photopolymerized in the crystalline state to give a three-dimensionally oriented polymer containing a cyclobutane ring in the main chain (for a review see Hasegawa, Suzuki, Nakanishi & Nakanishi, 1973). In order to clarify the polymerization mechanism, we have been studying the crystal structures of the diolefins and have discussed the mechanism on the basis of crystal structures of monomers and polymers (Naka-

nishi, Hasegawa & Sasada, 1972). The present compound, p-phenylenedi[α-cyanoacrylic acid] di-n-propyl ester (p-CPAnPr), is a tri-substituted olefin and shows the highest rate of polymerization among the group of compounds at room temperature.

p-CPAnPr was synthesized by the method of Nakanishi & Hasegawa (1969). Colourless plate-like crystals were grown from a tetrahydrofuran solution by slow evaporation. The space group was determined from Weissenberg photographs. The precise lattice constants and intensity data were derived from measurements on a Rigaku computer-controlled four-circle diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. The specimen used was a fragment of a plate, $0·20 \times 0·25 \times 0·10$ mm. All reflexions within the range of $2\theta < 56°$ were collected by use of the 2θ–ω scan mode with a scanning rate of 2° min^{-1}. Stationary background counts were accumulated for 10 s before and after each scan. Out of 2295 reflexions, 1669