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Structure of a Diterpene from *Euphorbia neriifolia*

BY L. L. KOH, A. S. NG AND G. K. TAN

Department of Chemistry, Faculty of Science, National University of Singapore, Singapore 0511

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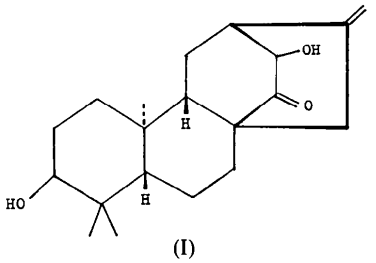
Abstract. Neriifolene (*ent*-3β,13-dihydroxyatis-16-en-14-one), $C_{20}H_{30}O_3$, $M_r = 318.4$, orthorhombic, $P2_12_12_1$, $a = 7.2720$ (10), $b = 10.962$ (3), $c = 21.582$ (8) Å, $V = 1720.3$ (8) Å^3, $Z = 4$, $D_x = 1.229$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.75$ cm^{-1}, $F(000) = 696$, $R = 0.045$ for 1035 observed reflections [$I > 3\sigma(I)$] at room temperature. The result confirmed the structure based on spectroscopic analysis [Ng (1990). *Phytochemistry*, **29**, 662–664]. The only difference is the orientation of the C(5)—H(5) bond, which was found to be *trans* with respect to C(20).

Experimental. Neriifolene (I) was isolated from the roots of *Euphorbia neriifolia*, as reported by Ng (1990). Crystals were obtained by recrystallization from hexane–$CHCl_3$. A single crystal of demensions 0.45 × 0.35 × 0.20 mm was used for X-ray structure analysis with a Siemens *R3m/V*2000 diffractometer. The lattice parameters were obtained from 15 reflections in the 2θ range of 6 to 20°. The intensity data were collected with Mo $K\alpha$ radiation (graphite crystal monochromator) using ω-scan technique. 1571 reflections in the range $3 < 2\theta < 48°$ were measured for $h = 0$ to 8, $k = 0$ to 11, $l = 0$ to 22. 1552 unique reflections, 1035 with $I > 3\sigma(I)$ used for structure determination.



(I)

The structure was solved by direct methods and refined by full-matrix least squares (on *F*) with anisotropic thermal parameters for all non-H atoms. H-atom positions were obtained from difference Fourier maps. Their positions were not refined but were included in the least-squares calculations with fixed isotropic thermal parameters. Final refinement converged to $R = 0.045$ and $wR = 0.055$ for the observed reflections. A weighting scheme of $w^{-1} =$

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$)

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	*y*	*z*	U_{eq}
C(1)	5581 (7)	4492 (5)	2267 (2)	37 (2)
C(2)	5023 (8)	4251 (6)	1595 (3)	45 (2)
C(3)	3536 (8)	5137 (5)	1394 (2)	36 (2)
C(4)	1781 (8)	5024 (5)	1799 (2)	34 (2)
C(5)	2387 (7)	5242 (4)	2485 (2)	28 (2)
C(6)	803 (8)	5239 (5)	2958 (2)	39 (2)
C(7)	1418 (8)	5767 (5)	3569 (2)	37 (2)
C(8)	3094 (7)	5153 (5)	3850 (2)	30 (2)
C(9)	4647 (7)	4965 (5)	3356 (2)	33 (2)
C(10)	3982 (7)	4409 (5)	2727 (2)	31 (2)
C(11)	6273 (7)	4286 (6)	3654 (3)	45 (2)
C(12)	6042 (7)	4158 (6)	4357 (3)	44 (2)
C(13)	4373 (8)	3358 (5)	4464 (2)	38 (2)
C(14)	2675 (8)	3962 (5)	4182 (2)	32 (2)
C(15)	3927 (9)	5950 (5)	4379 (2)	41 (2)
C(16)	5715 (9)	5416 (6)	4616 (2)	48 (2)
C(17)	6852 (12)	5954 (8)	5004 (3)	87 (3)
C(18)	756 (8)	3833 (5)	1661 (3)	49 (2)
C(19)	472 (9)	6058 (6)	1614 (2)	51 (2)
C(20)	3414 (8)	3083 (5)	2809 (2)	39 (2)
O(3)	3190 (6)	4934 (5)	752 (2)	58 (2)
O(13)	3972 (6)	3145 (3)	5107 (2)	50 (1)
O(14)	1137 (5)	3585 (4)	4272 (2)	48 (1)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(1)—C(2)	1.528 (8)	C(1)—C(10)	1.533 (7)
C(2)—C(3)	1.517 (8)	C(3)—C(4)	1.552 (7)
C(3)—O(3)	1.425 (6)	C(4)—C(5)	1.563 (7)
C(4)—C(18)	1.533 (8)	C(4)—C(19)	1.534 (8)
C(5)—C(6)	1.539 (7)	C(5)—C(10)	1.566 (7)
C(6)—C(7)	1.508 (7)	C(7)—C(8)	1.518 (7)
C(8)—C(9)	1.567 (7)	C(8)—C(14)	1.521 (7)
C(8)—C(15)	1.560 (7)	C(9)—C(10)	1.564 (7)
C(9)—C(11)	1.538 (7)	C(10)—C(20)	1.521 (7)
C(11)—C(12)	1.532 (8)	C(12)—C(13)	1.515 (8)
C(12)—C(16)	1.507 (9)	C(13)—C(14)	1.527 (8)
C(13)—O(13)	1.437 (6)	C(14)—O(14)	1.208 (7)
C(15) C(16)	1.514 (9)	C(16)—C(17)	1.316 (10)
C(2)—C(1)—C(10)	113.8 (4)	C(1)—C(2)—C(3)	110.5 (5)
C(2)—C(3)—C(4)	112.0 (4)	C(2)—C(3)—O(3)	107.7 (4)
C(4)—C(3)—O(3)	112.9 (4)	C(3)—C(4)—C(5)	106.8 (4)
C(3)—C(4)—C(18)	111.0 (4)	C(5)—C(4)—C(18)	116.9 (4)
C(3)—C(4)—C(19)	107.7 (4)	C(5)—C(4)—C(19)	108.0 (4)
C(18)—C(4)—C(19)	106.1 (5)	C(4)—C(5)—C(6)	114.7 (4)
C(4)—C(5)—C(10)	115.9 (4)	C(6)—C(5)—C(10)	109.3 (4)
C(5)—C(6)—C(7)	111.0 (4)	C(6)—C(7)—C(8)	114.6 (4)
C(7)—C(8)—C(9)	111.5 (4)	C(7)—C(8)—C(14)	114.1 (4)
C(9)—C(8)—C(14)	110.6 (4)	C(7)—C(8)—C(15)	110.8 (4)
C(9)—C(8)—C(15)	107.0 (4)	C(14)—C(8)—C(15)	102.3 (4)
C(8)—C(9)—C(10)	114.7 (4)	C(8)—C(9)—C(11)	109.5 (4)
C(10)—C(9)—C(11)	114.3 (4)	C(1)—C(10)—C(5)	108.1 (4)
C(1)—C(10)—C(9)	107.7 (4)	C(5)—C(10)—C(9)	107.0 (4)
C(1)—C(10)—C(20)	109.7 (4)	C(5)—C(10)—C(20)	113.3 (4)
C(9)—C(10)—C(20)	110.9 (4)	C(9)—C(11)—C(12)	112.0 (4)
C(11)—C(12)—C(13)	107.0 (4)	C(11)—C(12)—C(16)	107.5 (5)
C(13)—C(12)—C(16)	110.3 (5)	C(12)—C(13)—C(14)	109.6 (4)
C(12)—C(13)—O(13)	113.8 (4)	C(14)—C(13)—O(13)	106.9 (4)
C(8)—C(14)—C(13)	113.4 (4)	C(8)—C(14)—O(14)	123.7 (5)
C(13)—C(14)—O(14)	122.5 (5)	C(8)—C(15)—C(16)	111.4 (4)
C(12)—C(16)—C(15)	111.3 (5)	C(12)—C(16)—C(17)	123.2 (6)
C(15)—C(16)—C(17)	125.5 (6)		

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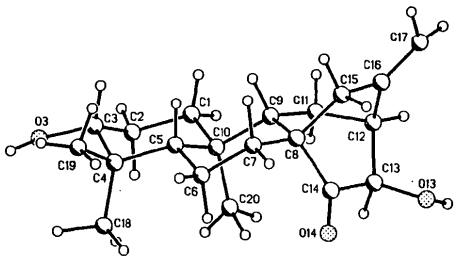

Fig. 1. Perspective view of the neriifolene molecule with atomic numbering.

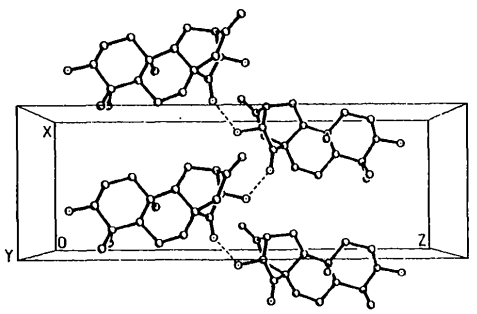

Fig. 2. Crystal packing of neriifolene viewed along the *b* axis.

$\sigma^2(F) + 0.0009F^2$ was used. The maximum $\Delta/\sigma = 0.167$ and goodness of fit = 1.33. The largest difference peak was 0.17 e Å^{-3} and deepest difference hole was -0.18 e Å^{-3}. All calculations were performed with *SHELXTL-Plus* (Siemens Analytical X-ray Instruments Inc., 1990) on a MicroVAX 2000 computer. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vo. IV). Figures were also prepared by *SHELXTL-Plus* graphics programs. The structure of neriifolene is shown in Fig. 1, which also shows the atomic numbering. Atomic coordinates of non-H atoms are given in Table 1.* Bond lengths and bond angles are given in Table 2. The packing of the crystal as viewed along the *b* axis is shown in Fig. 2. Neighbouring molecules are connected through hydrogen bonds between O(13) and O(14)′ with O(13)···O(14)′ distance of 2.806 Å.

Related literature. For isolation of neriifolene and investigation of *Euphorbia neriifolia* see Ng (1990). Isolation of compounds of the same molecular formula from other species of *Euphorbia* plants has also been reported (Cambie, Clark, Lal, Rickard, Rutledge & Woodgate, 1990; Lal, Cambie, Rutledge & Woodgate, 1990; Jia, Ding, Wang & Liu, 1990).

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54689 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of *N*-[(4-Cyanophenyl)(phenyl)methylene]aniline

BY HANS PREUT AND PAUL BLECKMANN

Fachbereich Chemie Universität Dortmund, Otto-Hahn-Straße 6, *D*-4600 *Dortmund* 50, *Germany*

AND TSONKO KOLEV AND IVAN JUCHNOVSKI

Institute of Organic Chemistry, Bulgarian Academy of Sciences, Sofia 1040, *Bulgaria*

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Abstract. 4-[α-(Phenylimino)benzyl]benzonitrile, $C_{20}H_{14}N_2$, $M_r = 282.34$, monoclinic, $C2$, $a = 15.796$ (4), $b = 7.332$ (2), $c = 14.498$ (4) Å, $\beta = 113.30$ (2)°, $V = 1542.2$ (8) Å^3, $Z = 4$, $D_x = 1.216$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.07$ mm^{-1}, $F(000) = 592$, $T = 291$ (1) K, final $R = 0.054$ for 1567 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. There are no short contacts between the molecules. All bond lengths, bond angles and dihedral angles are normal. Because of the con-

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