

Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J.-P. & Woolfson, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Universities of York, England, and Louvain, Belgium.

Nakai, H., Takasuka, M. & Shiro, M. (1984). *J. Chem. Soc. Perkin Trans. 2*, pp. 1459–1464.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.

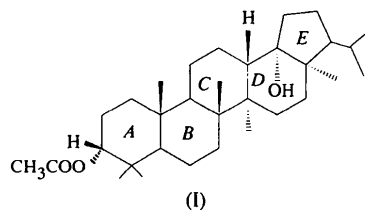
Patel, U., Haridas, M. & Singh, T. P. (1988). *Acta Cryst.* **C44**, 1264–1267.

Rambaud, J., Maury, L., Pauvert, B., Audran, M., Lasserre, Y., Berge, G. & Declercq, J. P. (1985). *Acta Cryst.* **C41**, 133–134.

Singh, C. (1965). *Acta Cryst.* **19**, 861–864.

Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.

1971). The structural elucidation *via* NMR spectra has already been carried out (Achari, Pal & Pakrashi, 1975). The present X-ray structure study was carried out in order to confirm these findings. Isoalangidiol is the 3 α -epimer of 18 α -B':A'-neogammacerane-3,18-diol. It represents the first example of a naturally occurring *D:E-cis* neohopane derivative and belongs to the rare group of pentacyclic triterpenes with a free hydroxyl group at the ring juncture.



Acta Cryst. (1997). **C53**, 1299–1301

Isoalangidiol Monoacetate, a Triterpene Alcohol

KINKINI BHATTACHARYYA,^a SIDDHARTHA CHAUDHURI,^b BASUDEB ACHARI,^c BISWANATH MAZUMDAR^d AND SUNIL KUMAR MAZUMDAR^a

^aDepartment of Materials Science, Indian Association for the Cultivation of Science, Jadavpur, Calcutta 700 032, India, ^bRSIC, Bose Institute, Calcutta 700 009, India, ^cIndian Institute of Chemical Biology, Jadavpur, Calcutta 700 032, India, and ^dSilchar Polytechnic, Silchar, Assam, India. E-mail: msskm@iacs.ernet.in

(Received 27 March 1996; accepted 11 November 1996)

Abstract

Isoalangidiol (3 α ,18 α -B':A'-neogammacerane-3,18-diol) has been extracted as a natural product from the leaves of *Alangium lamarckii* Thw. (Alangiaceae). The crystal structure study of its monoacetate, C₃₂H₅₄O₃, was undertaken in order to ascertain the conformation of the compound. The four six-membered rings are in chair conformations. The five-membered ring is distorted from a plane and is twisted, with a C19—C18—C17—C21 torsion angle of $-43.0(4)^\circ$. The molecular parameters of the compound are all within normal limits.

Comment

Isoalangidiol has been obtained from the petroleum ether extract of *Alangium lamarckii* Thw. (Alangiaceae) and purified as its monoacetate, (I), prepared by heating with acetic anhydride/pyridine at 373 K, by column chromatography over silica gel (Pakrashi & Achari,

The title compound consists of four *trans*-fused six-membered alicyclic rings (A, B, C and D) and a five-membered ring E *cis*-fused to ring D. Rings A, B, C and D are in chair conformations, as shown by the ring-puckering parameters given in Table 2 (Cremer & Pople, 1975). The acetoxy function, adjacent to a methylene group, is axial. There are altogether eight methyl groups in the structure. The isopropyl group is attached to ring E. The hydroxy group is attached to C18 in this neohopane derivative. The five-membered ring has a twisted conformation, with a C19—C18—C17—C21 torsion angle of $-43.0(4)^\circ$.

There is an intermolecular O—H...O hydrogen bond between the O18 and O32 atoms, joining the molecules into infinite chains along the *z* axis [O18...O32ⁱ 2.961(5) Å and O18—H18...O32ⁱ 124.1(4)°; symmetry code: (i) $-x - \frac{1}{2}, -y, z - \frac{1}{2}$].

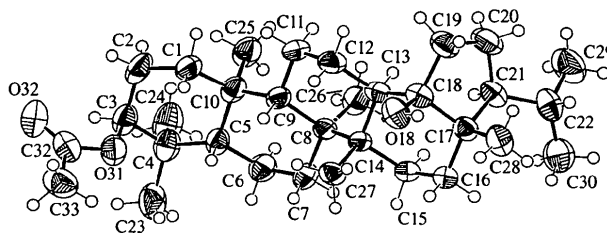


Fig. 1. The structure of (I) showing 30% probability displacement ellipsoids and the atom-numbering scheme.

Experimental

Isoalangidiol monoacetate, (I), was crystallized from a solution of benzene and ethanol.

Crystal data

C₃₂H₅₄O₃
M_r = 486.75

Mo K α radiation
 λ = 0.71073 Å

Acta Crystallographica Section C
ISSN 0108-2701 © 1997

Orthorhombic

P2₁2₁2₁

a = 12.201 (2) Å

b = 12.8625 (14) Å

c = 18.571 (2) Å

V = 2914.4 (7) Å³

Z = 4

D_x = 1.109 Mg m⁻³D_m not measured

Data collection

Enraf-Nonius CAD-4
diffractometer

ω-2θ scans

Absorption correction: none

2315 measured reflections

2315 independent reflections

1853 reflections with

I > 2σ(I)

Refinement

Refinement on F²

R(F) = 0.0525

wR(F²) = 0.1575

S = 0.981

2309 reflections

315 parameters

All H atoms refined

w = 1/[σ²(F_o²) + (0.1018P)²

+ 0.5002P]

where P = (F_o² + 2F_c²)/3Cell parameters from 25
reflections

θ = 10–15°

μ = 0.069 mm⁻¹

T = 293 (2) K

Tablet

0.28 × 0.24 × 0.10 mm

Colourless

θ_{max} = 22.96°

h = 0 → 13

k = 0 → 14

l = 0 → 20

3 standard reflections

every 100 reflections

intensity decay: 1%

(Δ/σ)_{max} = 0.063Δρ_{max} = 0.219 e Å⁻³Δρ_{min} = -0.206 e Å⁻³

Extinction correction: none

Scattering factors from

International Tables for
Crystallography (Vol. C)

Absolute configuration:

Flack (1983)

Flack parameter = 0 (3)

C27—C14—C8	111.5 (3)	O32—C32—O31	125.2 (5)
C13—C14—C8	108.2 (3)	O32—C32—C33	124.2 (5)
C28—C17—C16	108.4 (3)	O31—C32—C33	110.5 (5)
C28—C17—C21	111.0 (4)		
C10—C1—C2—C3	56.3 (5)	C9—C11—C12—C13	56.3 (5)
C1—C2—C3—O31	66.5 (5)	C11—C12—C13—C18	169.1 (3)
C1—C2—C3—C4	-53.2 (5)	C12—C13—C14—C15	-178.8 (3)
C2—C3—C4—C24	-77.3 (5)	C18—C13—C14—C8	-170.2 (3)
O31—C3—C4—C5	-71.5 (5)	C9—C8—C14—C15	-179.1 (3)
C2—C3—C4—C23	166.7 (4)	C7—C8—C14—C13	-178.7 (3)
C3—C4—C5—C6	177.5 (4)	C9—C8—C14—C13	-59.5 (4)
C24—C4—C5—C10	70.9 (5)	C8—C14—C15—C16	171.6 (3)
C10—C5—C6—C7	62.8 (5)	C14—C15—C16—C17	-58.9 (5)
C4—C5—C6—C7	-162.7 (4)	C15—C16—C17—C21	-59.5 (5)
C5—C6—C7—C8	-57.3 (5)	C14—C13—C18—C19	161.6 (3)
C6—C7—C8—C14	167.2 (3)	C14—C13—C18—C17	48.2 (4)
C7—C8—C9—C11	177.6 (3)	C21—C17—C18—C13	75.3 (4)
C14—C8—C9—C11	57.2 (4)	C16—C17—C18—C19	-166.1 (3)
C2—C1—C10—C9	-167.6 (3)	C21—C17—C18—C19	-43.0 (4)
C6—C5—C10—C1	-173.6 (4)	C17—C18—C19—C20	33.8 (4)
C4—C5—C10—C9	168.0 (3)	C19—C20—C21—C22	-145.3 (4)
C8—C9—C10—C1	168.4 (3)	C19—C20—C21—C17	-15.9 (5)
C11—C9—C10—C5	-173.8 (3)	C16—C17—C21—C20	154.6 (4)
C10—C9—C11—C12	169.6 (3)	C2—C3—O31—C32	82.9 (5)
C8—C9—C11—C12	-55.1 (5)	C4—C3—O31—C32	-153.5 (4)

Table 2. Ring puckering parameters for four rings of the title compound

	q ₂ (Å)	q ₃ (Å)	Q _T (Å)	θ (°)
Ring A	0.038 (5)	-0.534 (5)	0.535 (5)	175.9 (6)
Ring B	0.094 (5)	-0.564 (4)	0.572 (4)	170.6 (4)
Ring C	0.056 (4)	-0.606 (4)	0.609 (4)	174.7 (4)
Ring D	0.057 (4)	0.530 (4)	0.533 (4)	6.1 (4)

Ring-puckering parameters were calculated using PARST (Nardelli, 1983). All 54 H atoms were introduced at calculated positions and refined isotropically, riding on their parent atoms, using the AFIX function in SHELXL93 (Sheldrick, 1993).

Data collection: CAD-4 Software (Enraf-Nonius, 1989). Cell refinement: CAD-4 Software. Data reduction: CAD-4 Software. Program(s) used to solve structure: SHELXS86 (Sheldrick, 1990). Program(s) used to refine structure: SHELXL93. Molecular graphics: ORTEP (Johnson, 1965). Software used to prepare material for publication: SHELXL93.

The authors thank Professor S. P. SenGupta, Department of Materials Science, Indian Association for the Cultivation of Science, for extending all the facilities and for his keen interest in our work. We also thank the Council of Scientific and Industrial Research (CSIR), Government of India, for financial help.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1038). Services for accessing these data are described at the back of the journal.

References

- Achary, B., Pal, A. & Pakrashi, S. C. (1975). *Tetrahedron Lett.* **48**, 4275–4278.
- Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.
- Enraf-Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf-Nonius, Delft, The Netherlands.
- Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
- Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Table 1. Selected geometric parameters (Å, °)

C1—C10	1.540 (6)	C14—C15	1.546 (5)
C3—O31	1.465 (5)	C16—C17	1.537 (6)
C3—C4	1.535 (7)	C17—C28	1.527 (6)
C4—C24	1.524 (7)	C17—C21	1.562 (6)
C4—C5	1.562 (6)	C17—C18	1.571 (6)
C4—C23	1.564 (8)	C18—O18	1.428 (5)
C5—C10	1.551 (6)	C18—C19	1.536 (6)
C7—C8	1.535 (6)	C19—C20	1.521 (7)
C8—C26	1.550 (5)	C20—C21	1.542 (6)
C8—C9	1.569 (5)	C21—C22	1.543 (6)
C8—C14	1.594 (5)	C22—C30	1.510 (6)
C9—C10	1.572 (6)	C22—C29	1.525 (6)
C10—C25	1.552 (6)	O31—C32	1.328 (6)
C13—C18	1.539 (6)	C32—O32	1.202 (6)
C13—C14	1.562 (5)	C32—C33	1.507 (7)
C14—C27	1.544 (5)		
O31—C3—C2	108.5 (4)	C16—C17—C21	116.0 (3)
O31—C3—C4	107.5 (4)	C28—C17—C18	110.2 (4)
C2—C3—C4	113.9 (4)	C16—C17—C18	109.5 (3)
C24—C4—C5	115.3 (4)	C21—C17—C18	101.6 (3)
C3—C4—C5	109.2 (4)	O18—C18—C13	107.3 (3)
C24—C4—C23	107.5 (4)	O18—C18—C19	112.0 (3)
C3—C4—C23	108.1 (4)	C13—C18—C19	110.2 (3)
C7—C8—C26	107.3 (3)	O18—C18—C17	112.5 (3)
C7—C8—C9	108.6 (3)	C13—C18—C17	112.6 (3)
C26—C8—C9	111.6 (3)	C19—C18—C17	102.2 (3)
C26—C8—C14	109.9 (3)	C20—C19—C18	105.9 (4)
C1—C10—C25	107.8 (3)	C19—C20—C21	117.7 (4)
C1—C10—C5	107.2 (3)	C22—C21—C20	111.9 (4)
C25—C10—C5	115.0 (4)	C22—C21—C17	118.8 (4)
C25—C10—C9	112.7 (4)	C20—C21—C17	104.0 (3)
C5—C10—C9	106.1 (3)	C30—C22—C29	108.5 (3)
C27—C14—C15	106.2 (3)	C30—C22—C21	115.0 (4)
C27—C14—C13	110.8 (3)	C29—C22—C21	109.8 (4)
C15—C14—C13	108.8 (3)	C32—O31—C3	117.2 (4)

- Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.
 Pakrashi, S. C. & Achari, B. (1971). *Tetrahedron Lett.* **4**, 365–368.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Acta Cryst. (1997). **C53**, 1301–1302

The 4:2 Diels–Alder Adduct of 1,3-Cyclopentadiene with 1,4-Naphthoquinone

BILAL GÜNEŞ,^a HÜSEYİN SOYLU,^a SÜHEYLÂ ÖZBEY,^b
 ENGIN KENDİ^b AND ALI AYDIN^c

^aPhysics Department, Gazi Education Faculty, Gazi University, 06500 Beşevler, Ankara, Turkey, ^bPhysics Engineering Department, Engineering Faculty, Hacettepe University, 06532 Beytepe, Ankara, Turkey, and ^cChemistry Department, Gazi Education Faculty, Gazi University, 06500 Beşevler, Ankara, Turkey. E-mail: bgunes@cc.gazi.edu.tr

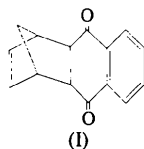
(Received 4 July 1996; accepted 17 April 1997)

Abstract

The title compound, 1,4-methano-1,4,4a,9a-tetrahydroanthracene-9,10-dione, C₁₅H₁₂O₂, is a Diels–Alder adduct resulting from a [4+2] cycloaddition of 1,3-cyclopentadiene with *p*-naphthoquinone.

Comment

The structure analysis of the title compound is part of an ongoing program of obtaining cage compounds by photochemical [2+2] cycloaddition (Maruyama, Terada & Yamamoto, 1981; Kaftory & Weisz, 1984). One aim is to obtain the norbornadiene derivatives from the title compound (Diels & Alder, 1929). The present structural analysis has been carried out in order to establish more details of the molecular geometry of the title compound, (I), thus providing a basis of comparison for our further studies.



The unit cell contains two independent molecules, related by a pseudo-*c* glide perpendicular to the *a* axis, in the asymmetric unit, the average deviation from *c* glide symmetry being 0.02 (2) Å between the two molecules. The C and O atoms of each dihydronaphthoquinone ring are coplanar (Pizzotti, Cenini, Ugo &

Demartin, 1991). The r.m.s. deviations of fitted atoms for these planes are 0.025 and 0.057 Å, the maximum deviations from these planes are 0.04 (3) Å for C6 and 0.164 (3) Å for C1A. The two five-membered rings adopt envelope conformations with C15 and C15A at the flaps. The dihedral angles between the envelope planes through C11–C14 and C11A–C14A, respectively, and the dihydronaphthoquinone moieties are 55.5 (1) and 49.7 (2)° for each independent molecule. The dihedral angle is 29.8 (2)° between the two envelope planes and 57.7 (1)° between the two dihydronaphthoquinone moieties in the asymmetric unit.

The bond lengths and angles are quite normal and comparable with corresponding values observed in related molecules (Kerr, 1987; Beddoes, Gorman & McNeeney, 1993).

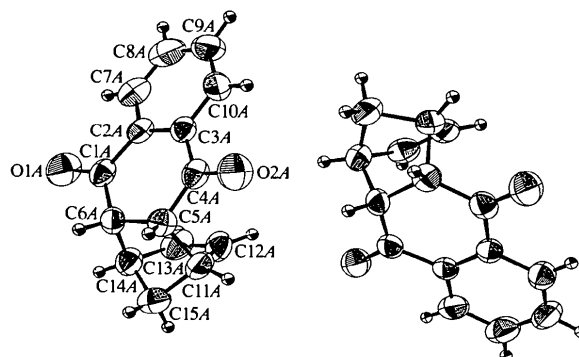


Fig. 1. A perspective view of the molecular structure of the title compound with the atom-numbering scheme. The displacement ellipsoids are drawn at the 50% probability level.

Experimental

p-Naphthoquinone (31.63 g, 0.20 mol) was suspended in benzene (100 ml). A cyclopentadiene (13.88 g, 0.21 mol) solution in cooled benzene (20 ml) was added to the above suspension. After keeping the mixture overnight, the resulting colourless solid was separated and recrystallized from ethanol.

Crystal data

C₁₅H₁₂O₂
M_r = 224.25
 Triclinic
*P*1̄
a = 9.4077 (12) Å
b = 10.1752 (10) Å
c = 12.5542 (13) Å
 α = 71.698 (9)°
 β = 86.980 (9)°
 γ = 77.019 (10)°
V = 1111.6 (2) Å³
Z = 4
D_x = 1.340 Mg m⁻³
D_m not measured

Mo *K*α radiation
 λ = 0.71073 Å
 Cell parameters from 25 reflections
 θ = 9–18°
 μ = 0.088 mm⁻¹
T = 293 (2) K
 Prismatic
 0.64 × 0.44 × 0.24 mm
 Colourless