checked with the difference map, except for the H atom attached to O(2), which was located from the difference map. All non-H atoms were refined anisotropically and all H atoms were fixed and not refined. The final $R(F) = 4\cdot81\%$ ($wR = 3\cdot33\%$). Weighting scheme from counting statistics, $w = 1/\sigma^2(F)$, $\Delta/\sigma < 0\cdot06$, peak in final difference map $< 0\cdot2$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). All the calculations were performed using the NRCC *SDP* PDP-11 package (Gabe & Lee, 1981), *MULTAN* and *ORTEPB* from the Enraf–Nonius *Structure Determination Package* (Enraf–Nonius, 1979) on a PDP–11/23 computer. Fig. 1 shows the molecular structure and labeling scheme.

Discussion. The positional and isotropic thermal parameters are given in Table 1.* Bond lengths and angles are given in Table 2. C(1)—O(1) is a double bond, while C(20)—O(2) is a single bond. All the C—C bonds are single, ranging from 1·495 to 1·582 Å. There is an intermolecular hydrogen bond between O(2) and O(1). The O(2)—H and O(1)···H distances are 1·00 (3) and 1·88 (3) Å respectively and the O(2)—H···O(1) angle is 165 (4)°.

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* Lists of anisotropic thermal parameters, structure factors and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43194 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 5-Isopropyl-4-methyl-1,3-diphenyl-3a,4,5,9b-tetrahydropyrazolo[3,4-*c*]isoquinoline

By René Mercier*

Laboratoire d'Electrochimie des Solides, UA CNRS 436, Université de Franche-Comté,
F-25030 Besançon CEDEX, France

Saïd Kitane, Kabula Tshiamala, Joël Vebrel and Bernard Laude

Laboratoire de Chimie Organique, Université de Franche-Comté, F-25030 Besançon CEDEX, France

and George M. Sheldrick

Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany

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Abstract. $C_{26}H_{27}N_3$, $M_r = 381\cdot5$, triclinic, $P\bar{1}$, $a = 9\cdot877$ (2), $b = 10\cdot493$ (2), $c = 11\cdot281$ (2) Å, $\alpha = 112\cdot18$ (3), $\beta = 98\cdot76$ (2), $\gamma = 93\cdot24$ (2)°, $V = 1062$ Å^3, $Z = 2$, $D_x = 1\cdot19$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 0\cdot47$ mm^{-1}, $F(000) = 408$, $T = 273$ K, $R = 0\cdot053$, $wR = 0\cdot064$ for 1759 independent reflections. The diastereoisomeric *trans* configuration is clearly evidenced. The pyrazolic ring is formed by regioselective 1,3-dipolar cycloaddition [C=N = 1·280 (4), N—N = 1·369 (3) Å]. The quasiplanar W-shaped H—C—N—C—H conformation explains the NMR coupling parameters.

Introduction. In a previous work (Kitane, Tshiamala, Laude, Vebrel & Cerutti, 1985), we discussed the stereochemistry of 1,3-dipolar cycloaddition of diphenylnitrilimine (1,3-dipole) to 1-isopropyl-2-methyl-1,2-dihydroisoquinoline (dipolarophile).

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* To whom correspondence should be addressed.

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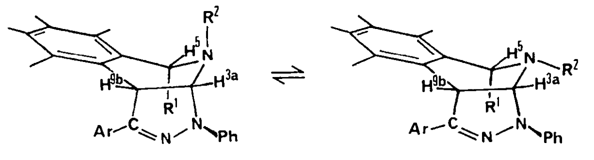
^{1}H NMR data suggest structures (1*a*) or (1*b*) for the cycloadduct: the existence of a coupling constant $^4J_{\mathrm{H(3a)-H(5)}} = 1{\cdot}5$ Hz is in good agreement with a quasi-coplanar geometry of the two protons H(3a) and H(5) arranged in the form of a letter W (Gaudemer, 1977). However, ^{1}H NMR is unable to distinguish between (1*a*) (R^1 and R^2 *trans*) or (1*b*) (R^1 and R^2 *cis*) invertomers.



(1*a*) (1*b*)

Ar = Ph R_1 = iPr R_2 = Me

The X-ray analysis shows that structure (1*a*) is correct and provides conformational information.

Experimental. Colourless prisms of the title compound were obtained by recrystallization from ethanol. Crystal size 0·3 × 0·3 × 0·1 mm. 3045 intensities recorded on a Nonius CAD-4 diffractometer at the Centre de Diffractometrie Automatique de Lyon. Monochromated Cu *K*α radiation, $2\theta_{\max}$ 120°. No significant intensity change of the check reflection. R_{int} 0·031 for 2756 unique reflections (index ranges after merging: h −7→6, k −12→12, l 0→13), 1759 data with $F > 4\sigma(F)$ used for all calculations (program system *SHELXTL*; Sheldrick, 1981). Cell constants refined from $30 < 2\theta < 40°$ reflections. No absorption correction.

Structure solution by routine direct methods in centrosymmetric space group $P\bar{1}$. Distinction between C(1) and N(3) atoms (each bearing a phenyl group) was unambiguously deduced from isotropic thermal parameters and bond lengths in the initial refinement in which both were assigned as C.

Refinement on F to R 0·053, wR 0·064 [all non-H atoms anisotropic; H atoms included using riding model with C—H 0·96 Å; U(H) = 0·075 Å^2 for aromatic and —C—H atoms; U(H) = 0·100 Å^2 for the H-methyl groups]; 272 parameters; weighting scheme $w^{-1} = \sigma^2(F) + 0{\cdot}008F^2$; $S = 1{\cdot}45$; $\Delta/\sigma_{\max} = 0{\cdot}2$. Max. and min. heights in final $\Delta\rho$ map 0·21 and −0·23 e Å^{-3}. Atomic scattering factors as incorporated in *SHELXTL*.

Discussion. Final atomic coordinates are given in Table 1 with derived parameters in Table 2.* An *ORTEP* (Johnson, 1965) view of the molecule (Fig. 1) displays the atom labelling and the main conformational features. Only three H atoms have been shown to illustrate the relative configurations. The R^1 and R^2 groups (isopropyl and methyl) lie on each side of the

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^4)

$U_{\mathrm{eq}} = (U^{\circ}_{11}U^{\circ}_{22}U^{\circ}_{33})^{1/3}$.

	x	y	z	U_{eq}
C(9a)	4937 (4)	8056 (2)	7078 (3)	614 (19)
C(5a)	4009 (4)	7534 (2)	7663 (3)	643 (18)
C(6)	4520 (4)	7350 (3)	8802 (3)	835 (23)
C(7)	5901 (6)	7654 (3)	9338 (3)	975 (26)
C(8)	6828 (4)	8075 (3)	8717 (4)	860 (24)
C(9)	6335 (4)	8264 (3)	7587 (3)	732 (21)
C(5)	2535 (4)	7025 (3)	6959 (3)	696 (18)
C(3a)	3152 (3)	7187 (3)	4969 (3)	665 (17)
C(9b)	4392 (3)	8276 (3)	5857 (3)	647 (17)
C(1)	3802 (3)	9631 (3)	6004 (2)	677 (18)
C(11)	4441 (4)	11058 (3)	6838 (3)	702 (20)
C(12)	5811 (4)	11350 (3)	7439 (3)	764 (22)
C(13)	6399 (4)	12719 (3)	8213 (3)	850 (22)
C(14)	5613 (5)	13776 (3)	8353 (3)	930 (27)
C(15)	4241 (5)	13496 (3)	7746 (3)	924 (26)
C(16)	3663 (4)	12152 (3)	6985 (3)	788 (20)
C(31)	1141 (4)	7597 (3)	3533 (3)	743 (19)
C(32)	495 (4)	8522 (3)	3101 (3)	866 (23)
C(33)	−483 (5)	8023 (4)	1965 (4)	1035 (29)
C(34)	−851 (4)	6627 (4)	1268 (4)	1002 (29)
C(35)	−253 (4)	5709 (4)	1723 (3)	870 (23)
C(36)	740 (4)	6174 (3)	2847 (3)	808 (21)
C(4)	3035 (4)	4999 (3)	5225 (3)	752 (20)
C(51)	1528 (4)	8106 (3)	7307 (3)	829 (23)
C(53)	69 (5)	7488 (4)	6520 (5)	1102 (33)
C(52)	1477 (5)	8635 (4)	8765 (4)	1056 (28)
N(4)	2500 (3)	6339 (2)	5545 (2)	648 (14)
N(2)	2639 (3)	9445 (2)	5245 (2)	741 (15)
N(3)	2194 (3)	8055 (2)	4617 (2)	735 (16)

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

Ring (1): benzo ring			
C(5a)—C(9a)	1·405 (5)	C(8)—C(7)	1·381 (7)
C(9a)—C(9)	1·384 (5)	C(7)—C(6)	1·372 (6)
C(9)—C(8)	1·384 (6)	C(6)—C(5a)	1·394 (5)
Ring (2): phenyl ring			
C(11)—C(12)	1·379 (5)	C(14)—C(15)	1·384 (6)
C(12)—C(13)	1·403 (4)	C(15)—C(16)	1·376 (4)
C(13)—C(14)	1·363 (6)	C(16)—C(11)	1·390 (5)
Ring (3): phenyl ring			
C(31)—C(32)	1·386 (5)	C(34)—C(35)	1·373 (7)
C(32)—C(33)	1·381 (5)	C(35)—C(36)	1·382 (4)
C(33)—C(34)	1·371 (5)	C(36)—C(31)	1·397 (4)
Ring (4): pentaatomic heterocycle			
C(3a)—C(9b)	1·553 (4)	N(2)—N(3)	1·369 (3)
C(9b)—C(1)	1·527 (4)	N(3)—C(3a)	1·463 (4)
C(1)—N(2)	1·280 (4)		
Ring (5): hexaatomic heterocycle			
C(5a)—C(5)	1·509 (4)	C(3a)—C(9b)	1·553 (4)
C(5)—N(4)	1·476 (3)	C(9b)—C(9a)	1·507 (4)
N(4)—C(3a)	1·459 (4)	C(9a)—C(5a)	1·405 (5)
C(1)—C(11)	1·475 (3)	C(51)—C(52)	1·534 (6)
C(5)—C(51)	1·529 (5)	N(4)—C(4)	1·464 (4)
N(3)—C(31)	1·386 (4)	C(51)—C(53)	1·535 (5)
C(9a)—C(5a)—C(5)	119·2 (3)	C(3a)—C(9b)—C(1)	101·3 (2)
C(5a)—C(5)—N(4)	109·5 (3)	C(9b)—C(1)—N(2)	112·9 (2)
C(5)—N(4)—C(3a)	115·1 (2)	C(1)—N(2)—N(3)	109·7 (2)
N(4)—C(3a)—C(9b)	116·7 (2)	N(2)—N(3)—C(3a)	113·2 (2)
C(3a)—C(9b)—C(9a)	112·9 (3)	N(3)—C(3a)—C(9b)	102·0 (2)
C(9b)—C(9a)—C(5a)	119·0 (3)		
C(3a)—N(3)—C(31)	125·2 (2)	C(3a)—N(4)—C(4)	111·6 (3)
N(2)—N(3)—C(31)	119·4 (3)	C(5)—N(4)—C(4)	110·9 (2)
N(2)—C(1)—C(11)	119·1 (3)	C(5a)—C(5)—C(51)	115·1 (2)
C(9b)—C(1)—C(11)	127·9 (3)	N(4)—C(5)—C(51)	113·4 (3)

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* Lists of structure factors, anisotropic thermal parameters, torsion angles and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43233 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

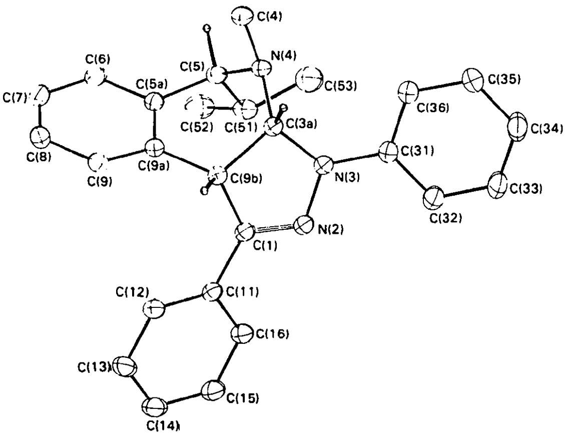

Fig. 1. *ORTEP* (Johnson, 1965) plot of 5-isopropyl-4-methyl-1,3-diphenyl-3a,4,5,9b-tetrahydropyrazolo[3,4-*c*]isoquinoline.

mean plane of the benzo ring (Table 2) in a pseudo-axial *trans* configuration corresponding to (1*a*) [torsion angle C(4)—N(4)—C(5)—C(51) 157·8°]. The pyrazolinic ring lies on the same side of the six-membered ring as the R^1 group.

It is interesting to note the coplanarity of the C(5)—H(5) and C(3a)—H(3a) bonds, this mean plane being orthogonal to that of the benzo ring. The 'W' geometry of H(5)—C(5)—N(4)—C(3a)—H(3a) is also evident, the N atom being slightly displaced from the mean plane formed by the two C—H bonds.

The C(3a)—H(3a) bond is pseudoaxial (62·6°) whereas the C(5)—H(5) and C(9b)—H(9b) bonds are effectively equatorial (they form angles of 24·4 and 20·5° respectively with the benzo ring).

The regiospecificity of the cycloaddition is clearly confirmed. The bond lengths in the pyrazolinic ring are quite normal (C=N 1·280 Å) (Mercier, Vebrel, Schmidt & Sheldrick, 1984). The C—C bond common to the five- and six-membered rings is comparable to analogous bonds observed in fused pentagonal rings (Mercier, Ramah, Laude, Faure & Loiseleur, 1984). The C=N double bond is conjugated with the α-phenyl group (dihedral angle 12·7°).

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Structure Determination of Aplyviolene from *Chelonaplysilla violacea*

BY JOHN S. BUCKLETON, PATRICIA R. BERGQUIST, RICHARD C. CAMBIE, GEORGE R. CLARK,* PETER KARUSO AND CLIFTON E. F. RICKARD

Departments of Chemistry, and Zoology,† *The University of Auckland, Auckland, New Zealand*

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Abstract. 6-Acetoxy-8-(decahydro-1,4,4-trimethyl-8-methylene-1-azulenyl)-2,7-dioxabicyclo[3.2.1]octan-3-one, $C_{22}H_{32}O_5$, $M_r = 376{\cdot}50$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}103\ (2)$, $b = 11{\cdot}632\ (2)$, $c = 21{\cdot}774\ (3)$ Å, $V = 2052{\cdot}22$ Å^3, $T = 293 \pm 1$ K, $D_m = 1{\cdot}214\ (5)$, $D_x = 1{\cdot}218$ g cm^{-3}, $Z = 4$, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, Zr filter, $\mu = 0{\cdot}091$ cm^{-1}, $F(000) = 816$, $R = 0{\cdot}042$ for 1260 $[I > 2{\cdot}5\sigma(I)]$ reflections. The X-ray analysis indicates that the previously proposed structure of dendrillolide A is incorrect. There is a long C—C bond [1·577 (6) Å] within the five-membered ring of the azulenyl moiety, apparently caused by steric crowding around the 1-methyl substituent. The ring oxygens are involved in short [1·412 (6)] and long [1·447 (6) Å] C—O bonds at the bridgehead of the bicyclooctanone fragment. Other bond lengths are normal.

Introduction. Recently, Sullivan & Faulkner (1984) reported the isolation and structural determination of some metabolites from an unidentified species of *Dendrilla* sponge from Palau. They included dendrillolide A (1) and dendrillolide B (2), with a novel

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* To whom correspondence should be addressed.

† Department of Chemistry: JSB, RCC, GRC, PK, CEFR. Department of Zoology: PRB, PK.

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