S21	0.35732 (12)	0.08546 (11)	0.05263 (14)	0.0393 (3)
Cl	−0.02363 (12)	0.25050 (12)	−0.49818 (13)	0.0453 (4)
N11	0.2767 (3)	0.3370 (4)	−0.0614 (4)	0.0313 (8)
N21	0.1732 (4)	0.1489 (4)	−0.1955 (5)	0.0346 (9)
C11	0.3639 (4)	0.4014 (4)	0.0646 (5)	0.032 (1)
C12	0.2349 (6)	0.5664 (5)	−0.1491 (6)	0.044 (1)
C13	0.1796 (5)	0.4269 (5)	−0.1679 (6)	0.040 (1)
C21	0.2617 (4)	0.2013 (4)	−0.0774 (5)	0.0313 (9)
C22	0.2784 (6)	−0.0500 (5)	−0.0804 (7)	0.047 (1)
C23	0.1540 (5)	0.0063 (5)	−0.1871 (7)	0.046 (1)

Table 2. *Selected geometric parameters (Å, °)*

S11—C11	1.643 (4)	N11—C13	1.485 (6)
S12—C11	1.723 (4)	N11—C21	1.368 (5)
S12—C12	1.798 (5)	N21—C21	1.300 (5)
S21—C21	1.741 (4)	N21—C23	1.448 (6)
S21—C22	1.836 (5)	C12—C13	1.511 (7)
N11—C11	1.389 (5)	C22—C23	1.503 (7)
C11—S12—C12	94.8 (2)	S11—C11—S12	121.9 (2)
C21—S21—C22	89.5 (2)	C13—C12—S12	105.1 (3)
C21—N11—C11	125.0 (4)	N11—C13—C12	108.0 (4)
C21—N11—C13	119.7 (3)	N21—C21—N11	121.2 (4)
C11—N11—C13	114.2 (4)	N21—C21—S21	114.6 (3)
C21—N21—C23	115.7 (4)	N11—C21—S21	124.2 (3)
N11—C11—S11	127.3 (3)	C23—C22—S21	106.2 (3)
N11—C11—S12	110.7 (3)	N21—C23—C22	106.4 (4)
C21—N11—C11—S11	0.1 (7)	C23—N21—C21—S21	10.3 (6)
C13—N11—C11—S11	168.1 (4)	C11—N11—C21—N21	176.3 (4)
C21—N11—C11—S12	−177.4 (3)	C13—N11—C21—N21	8.9 (7)
C13—N11—C11—S12	−9.5 (5)	C11—N11—C21—S21	−4.5 (6)
C12—S12—C11—N11	−6.8 (4)	C13—N11—C21—S21	−171.9 (4)
C12—S12—C11—S11	175.5 (3)	C22—S21—C21—N21	6.6 (4)
C11—S12—C12—C13	20.0 (4)	C22—S21—C21—N11	−172.6 (4)
C21—N11—C13—C12	−166.6 (4)	C21—S21—C22—C23	−20.1 (4)
C11—N11—C13—C12	24.8 (6)	C21—N21—C23—C22	−25.7 (6)
S12—C12—C13—N11	−27.3 (6)	S21—C22—C23—N21	28.0 (6)
C23—N21—C21—N11	−170.4 (4)		

The unit-cell parameters and space group were found initially from oscillation and Weissenberg photographs. The structure was solved by direct methods. The H atoms were found from difference synthesis maps and their parameters refined without restriction.

Data collection: *XTL/XTLE* (Syntex, 1976). Cell refinement: *XTL/XTLE*. Data reduction: *XTL/XTLE*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1250). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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2-(7,9-Diphenylcyclopenta[*a*]acenaphthadien-6b-yl)-2-phenylethanol

Timo Repo,[a] Martti Klinga,[a] Markku Leskelä,[a]
Mika Polamo[a] and Bernhard Rieger[b]

[a]*Laboratory of Inorganic Chemistry, Box 55, 00014 University of Helsinki, Finland, and* [b]*Universität Ulm, Organische Chemie III, Makromolekulare Chemie, Albert-Einstein-Allee 11, D-89091 Ulm, Germany. E-mail: timo.repo@helsinki.fi*

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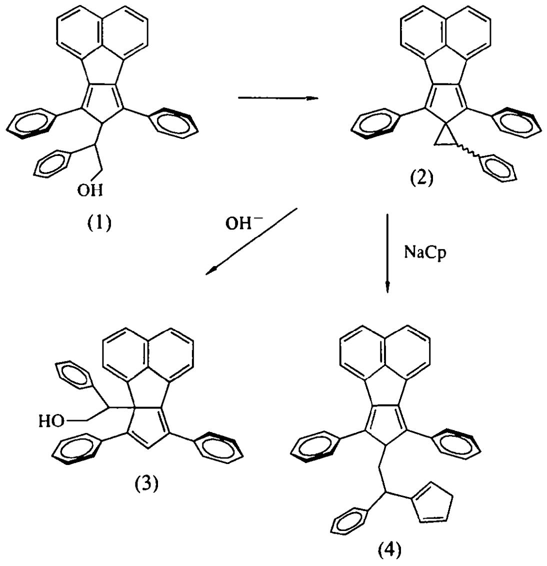
Abstract

The synthetic route to obtain single crystals of 2-(7,9-diphenyl-6b*H*-cyclopenta[*a*]acenaphth-6b-yl)-2-phenylethanol, $C_{35}H_{26}O$, is described. There are only weak van der Waals-type forces between neighbouring monomeric molecules.

Comment

We earlier described the use of the cyclopentadienyl derivative 7,9-diphenylcyclopenta[*a*]acenaphthadiene in the preparation of unsymmetric ethylene-bridged zirconocene dichlorides and their use in the polymerization of propene (Rieger, Repo & Jany, 1995*a*,*b*). In this work the ring opening of spiro[(7,9-diphenylcyclopenta[*a*]acenaphthadiene)-8,1′-(2′-phenylcyclopropane)], (2), by reaction with nucleophilic NaCp results in the formation of 1-cyclopentadienyl-2-(7,9-diphenylcyclopenta[*a*]acenaphthadien-8-yl)-1-phenylethane, (4). When KOH was used instead of NaCp to open the cyclopropyl ring, rearrangement of the five-membered ring to give the title compound, (3), took place. The mechanism for this rearrangement is unknown.



In compound (3) (Fig. 1) the bond lengths in the aromatic rings are unexceptional; the C—C distances in the naphthalene nucleus (C8–C17) vary from 1.359 (4) to 1.424 (3) Å and those in the three phenyl rings from 1.365 (5) to 1.403 (3) Å. The lengths of the formally single bonds [C4—C23 1.471 (3), C5—C6 1.455 (3), C6—C29 1.472 (3) and C7—C8 1.459 (3) Å] may be compared to the usual conjugated C—C bond length of 1.46 Å (Norman, 1978). Bond lengths and angles around the quaternary C3 atom are somewhat distorted from ideal tetrahedral values. For example, the C2—C3 bond length of 1.607 (3) Å is significantly longer and weaker than a typical C—C bond length of 1.54 Å. The electron-withdrawing substituents on the C2 atom, *viz* the phenyl ring and the ethanol group, are probably one reason for this weakening. The angles around C3 range from 101.6 (2) to 125.0 (2)°. There is no evidence of intermolecular interactions in the solid state other than van der Waals forces.

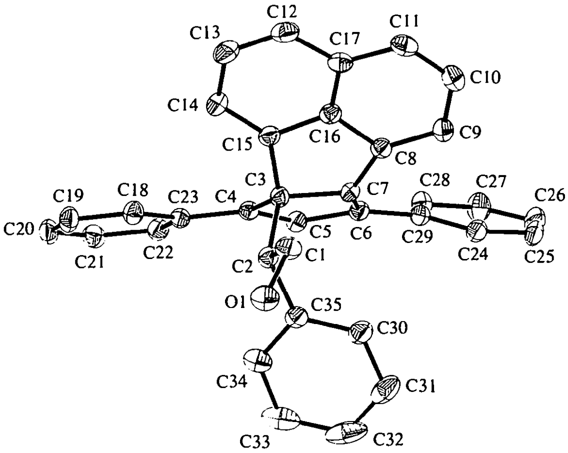


Fig. 1. View of the title compound, $C_{35}H_{26}O$, with the atom labels. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted for clarity.

Experimental

Spiro[(7,9-diphenylcyclopenta[*a*]acenaphthadiene)-8,1′-(2′-phenylcyclopropane)], (2), was prepared according to the literature procedure of Rieger, Repo & Jany (1995*a*) starting from 2-(7,9-diphenylcyclopenta[*a*]acenaphthadienyl)-2-phenylethanol, (1). Compound (2) (2.00 g, 4.50 mmol) was dissolved in 60 ml of a saturated acetone solution of KOH. The reaction mixture was refluxed for 1 h. Crystallization of the yellow product was performed by slow evaporation of acetone. Yellow crystals of (3) were washed with water and isolated by filtration (1.62 g, 3.51 mmol, 78%). ^{1}H NMR ($CDCl_3$): 3.04–3.22 (*m*, 1H, ethanol CH), 3.68 (*t*, *J* = 10.62 Hz, 1H, ethanol CH_2), 3.87 (*dd*, *J* = 10.16, 4.50 Hz, 1H, ethanol CH_2), 6.68 (*s*, 1H, CH_{Cp}), 6.8–8.1 p.p.m. (*m*, 21H, aromatic H). EIHRMS: calculated for $C_{35}H_{26}O$ 462.1983, observed 462.1999.

Crystal data

$C_{35}H_{26}O$
$M_r = 462.56$
Triclinic
$P\overline{1}$
$a = 11.677 (2)$ Å
$b = 12.431 (2)$ Å
$c = 9.443 (2)$ Å
$\alpha = 108.60 (3)^\circ$
$\beta = 111.44 (3)^\circ$
$\gamma = 89.96 (3)^\circ$
$V = 1198.7 (4)$ Å^3
$Z = 2$
$D_x = 1.282$ Mg m^{-3}
$D_m = 1.28$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 4.85$–9.23°
$\mu = 0.075$ mm^{-1}
$T = 193 (2)$ K
Prismatic
0.40 × 0.35 × 0.30 mm
Yellow

Data collection

Rigaku AFC-7*S* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3972 measured reflections
3779 independent reflections
3032 observed reflections [$F^2 > 2\sigma(F^2)$]

$R_{\rm int} = 0.0257$
$\theta_{\rm max} = 25^\circ$
$h = 0 \rightarrow 13$
$k = -14 \rightarrow 14$
$l = -11 \rightarrow 10$
3 standard reflections monitored every 200 reflections
intensity decay: <2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0563$
$wR(F^2) = 0.1392$
$S = 1.056$
3762 reflections
353 parameters
H atoms refined as riding model
$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 0.34P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$

$\Delta\rho_{\rm max} = 0.28$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.012 (2)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
O1	0.8696 (2)	0.8299 (2)	0.5653 (2)	0.0499 (5)
C1	0.8565 (3)	0.7482 (2)	0.4122 (3)	0.0445 (7)
C2	0.8927 (2)	0.8044 (2)	0.3114 (3)	0.0333 (6)
C3	0.9079 (2)	0.7166 (2)	0.1543 (3)	0.0305 (5)
C4	0.9607 (2)	0.7862 (2)	0.0807 (3)	0.0307 (5)
C5	0.8680 (2)	0.7864 (2)	−0.0560 (3)	0.0317 (5)
C6	0.7561 (2)	0.7132 (2)	−0.0938 (3)	0.0309 (5)
C7	0.7802 (2)	0.6653 (2)	0.0230 (3)	0.0306 (5)
C8	0.7446 (2)	0.5550 (2)	0.0294 (3)	0.0319 (5)
C9	0.6392 (2)	0.4767 (2)	−0.0439 (3)	0.0376 (6)
C10	0.6462 (3)	0.3703 (2)	−0.0190 (3)	0.0450 (7)
C11	0.7537 (3)	0.3418 (2)	0.0756 (3)	0.0450 (7)
C12	0.9825 (3)	0.4015 (2)	0.2480 (3)	0.0486 (7)
C13	1.0819 (3)	0.4828 (2)	0.3074 (4)	0.0522 (7)
C14	1.0719 (2)	0.5884 (2)	0.2806 (3)	0.0446 (7)
C15	0.9587 (2)	0.6106 (2)	0.1915 (3)	0.0339 (6)
C16	0.8564 (2)	0.5250 (2)	0.1265 (3)	0.0330 (5)
C17	0.8642 (2)	0.4198 (2)	0.1523 (3)	0.0392 (6)
C18	1.1720 (2)	0.8587 (2)	0.3050 (3)	0.0362 (6)
C19	1.2881 (2)	0.9210 (2)	0.3702 (3)	0.0433 (6)
C20	1.3221 (2)	0.9795 (2)	0.2871 (3)	0.0441 (7)
C21	1.2384 (2)	0.9737 (2)	0.1352 (3)	0.0414 (6)
C22	1.1222 (2)	0.9115 (2)	0.0701 (3)	0.0373 (6)
C23	1.0849 (2)	0.8524 (2)	0.1528 (3)	0.0311 (5)
C24	0.5271 (2)	0.6638 (2)	−0.2419 (3)	0.0389 (6)
C25	0.4209 (2)	0.6398 (2)	−0.3822 (3)	0.0472 (7)
C26	0.4293 (3)	0.6451 (3)	−0.5210 (3)	0.0521 (7)
C27	0.5429 (3)	0.6751 (2)	−0.5206 (3)	0.0503 (7)
C28	0.6492 (2)	0.6977 (2)	−0.3819 (3)	0.0402 (6)
C29	0.6428 (2)	0.6907 (2)	−0.2409 (3)	0.0332 (5)
C30	0.6797 (3)	0.8678 (3)	0.2113 (3)	0.0489 (7)
C31	0.6040 (3)	0.9501 (3)	0.1789 (3)	0.0656 (10)
C32	0.6548 (4)	1.0583 (3)	0.2105 (4)	0.0778 (12)
C33	0.7839 (4)	1.0845 (3)	0.2714 (4)	0.0639 (10)
C34	0.8573 (3)	1.0009 (2)	0.2994 (3)	0.0450 (7)
C35	0.8073 (2)	0.8923 (2)	0.2715 (3)	0.0356 (6)

Table 2. *Selected geometric parameters (Å, °)*

O1—C1	1.430 (3)	C4—C23	1.471 (3)
C1—C2	1.513 (3)	C5—C6	1.455 (3)
C2—C35	1.523 (3)	C6—C7	1.357 (3)
C2—C3	1.607 (3)	C6—C29	1.472 (3)
C3—C4	1.520 (3)	C7—C8	1.459 (3)
C3—C7	1.521 (3)	C8—C16	1.424 (3)
C3—C15	1.534 (3)	C15—C16	1.409 (3)
C4—C5	1.349 (3)		
O1—C1—C2	111.2 (2)	C4—C3—C15	125.0 (2)
C1—C2—C35	111.1 (2)	C7—C3—C15	101.6 (2)
C1—C2—C3	114.6 (2)	C4—C3—C2	107.7 (2)
C35—C2—C3	112.6 (2)	C7—C3—C2	109.3 (2)
C4—C3—C7	102.7 (2)	C15—C3—C2	109.4 (2)

The intensity data were corrected for Lorentz and polarization effects, and also for extinction. All non-H atoms were anisotropically refined. H atoms were refined on calculated positions with isotropic displacement parameters. The ethanolic H atom was allowed to rotate freely on its calculated position and although its final displacement parameter was large compared with the other H atoms, the refinement converged.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993*a*). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1993*b*). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXL93*.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1261). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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cis-Aconitic Acid at 150 K

Norbert Nagel, Uwe Endruschat and Hans Bock*

Institut für Anorganische Chemie der Universität Frankfurt,
Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany.
E-mail: nagel@bock.anorg.chemie.uni-frankfurt.de

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Abstract

cis-Aconitic acid (*cis*-1-propene-1,2,3-tricarboxylic acid, $C_6H_6O_6$) crystallizes with an intramolecular hydrogen bond between the two carboxylic acid groups linked by the C═C double bond and exhibits a complex intermolecular hydrogen-bond pattern. The two carboxylic acid groups involved in the intramolecular hydrogen bond are coplanar with the propene skeleton, while the third carboxylic group is twisted out of this plane with a dihedral angle of 66.21 (9)°.

Comment

Within the Krebs cycle, *cis*-aconitate originates from citrate catalyzed by the enzyme aconitase. The crystal structure of dipotassium *cis*-aconitate (Glusker,