C(1)	0.2678 (3)	0.0693 (3)	0.5685 (3)	0.0413 (9)
C(2)	0.2500 (4)	0.2288 (3)	0.3278 (3)	0.0492 (11)
C(3)	0.2371 (4)	0.1159 (3)	0.2253 (4)	0.0527 (12)
C(4)	0.2356 (3)	−0.0200 (3)	0.2967 (3)	0.0465 (11)
C(5)	0.2479 (7)	0.3801 (4)	0.2702 (6)	0.0734 (17)
C(6)	0.2117 (6)	−0.1486 (4)	0.1893 (4)	0.0660 (16)
C(7)	0.2666 (3)	−0.3844 (3)	0.7365 (3)	0.0427 (9)

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

S(1)—C(1)	1.685 (3)	S(2)—C(7)	1.705 (3)
N(1)—C(1)	1.369 (4)	N(1)—C(2)	1.353 (4)
N(2)—C(1)	1.344 (3)	N(2)—C(4)	1.337 (3)
N(3)—C(7)	1.320 (3)	N(4)—C(7)	1.327 (3)
C(2)—C(3)	1.354 (4)	C(2)—C(5)	1.493 (5)
C(3)—C(4)	1.397 (4)	C(4)—C(6)	1.485 (5)
C(1)—N(1)—C(2)	124.0 (2)	C(1)—N(2)—C(4)	119.4 (2)
N(1)—C(1)—N(2)	118.4 (2)	S(1)—C(1)—N(2)	122.1 (2)
S(1)—C(1)—N(1)	119.6 (2)	N(1)—C(2)—C(5)	117.2 (3)
N(1)—C(2)—C(3)	117.2 (2)	C(3)—C(2)—C(5)	125.6 (3)
C(2)—C(3)—C(4)	119.0 (3)	N(2)—C(4)—C(3)	122.0 (2)
C(3)—C(4)—C(6)	122.0 (3)	N(2)—C(4)—C(6)	115.9 (2)
N(3)—C(7)—N(4)	117.0 (2)	S(2)—C(7)—N(3)	122.1 (2)
S(2)—C(7)—N(3)	121.0 (2)		

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N(3)—H(31)$\cdots$S(1)	0.87 (3)	2.58 (3)	3.418 (3)	164 (3)
N(4)—H(42)$\cdots$N(2)	0.95 (3)	1.97 (3)	2.915 (4)	172 (3)
N(1)—H(1)$\cdots$S(2^i)	0.89 (3)	2.40 (3)	3.277 (3)	167 (2)
N(3)—H(32)$\cdots$S(2^{ii})	0.90 (3)	2.60 (2)	3.462 (3)	161 (2)
N(4)—H(41)$\cdots$S(2^{iii})	0.80 (3)	2.75 (2)	3.486 (3)	154 (3)

Symmetry codes: (i) $x, 1 + y, z$; (ii) $1 - x, -1 - y, 2 - z$; (iii) $-x, -1 - y, 1 - z$.

The thermal motion of the molecule is strongly anisotropic with U_{22} larger than the other components for most of the atoms.

Program systems used: *XRAYARC* (Vickery, Bright & Mallinson, 1971) for Patterson synthesis; *NORMAL, EXFFT* and *SEARCH* sequences of *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) for weighted Fourier syntheses, *SHELX76* (Sheldrick, 1976) for refinement, and *ORTEP*II (Johnson, 1976) for molecular graphics.

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1-Diphenylmethylene-4-(triphenylmethyl)-cyclohexa-2,5-diene Benzene Solvate

Leonid N. Bochkarev and Natalia E. Molosnova

Department of Chemistry, Nizhny Novgorod State Pedagogical University, Ulyanova 1, Nizhny Novgorod 603600, Russia

Lev N. Zakharov and Georgy K. Fukin

Institute of Organometallic Chemistry, Russian Academy of Sciences, Tropinina 49, Nizhny Novgorod 603600, Russia

Alexander I. Yanovsky and Yuri T. Struchkov

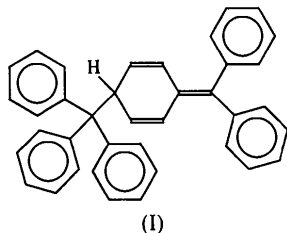*Institute of Organoelement Compounds, Russian Academy of Sciences, 28 Vavilov St., Moscow 117813, Russia*

Abstract

The crystal structure of the benzene solvate of 1-diphenylmethylene-4-(triphenylmethyl)cyclohexa-2,5-diene, $C_{38}H_{30}.C_6H_6$, has been determined by X-ray diffraction.

Comment

After the discovery of the Ph$_3$C$^\bullet$ radical there was lengthy discussion about the structure of its dimer (McBride, 1974). The correct structure of this dimer, 1-diphenylmethylene-4-(triphenylmethyl)cyclohexa-2,5-diene, (I), was first established by an NMR study (Staab, Brettschneider & Brunner, 1970) and was later confirmed by X-ray analysis of the ethyl acetate solvate and unsolvated crystals (Allemand & Gerdil, 1978;

Blom, Roelofsen & Kanters, 1982). In the course of our systematic investigation of the reaction of metallic lanthanides with organoelement halides of Group IVB elements, we found that the reaction of Yb with Ph_3CCl gave a precipitate of $YbCl_2$ and a yellow solution of $Ph_3C^{\bullet}$ radicals in equilibrium with their dimer (Bochkarev, Molosnova, Zakharov, Fukin, Yanovsky & Struchkov, 1994). Compound (I) was isolated as crystals of its benzene solvate by crystallization from benzene/hexane solution. The crystals were stable in air for a few hours and were readily soluble in benzene and tetrahydrofuran, and sparingly soluble in hexane. They melted, with decomposition, at 413–417 K.

(I)

The structure of (I) found here is similar to that in both the ethyl acetate solvate (Allemand & Gerdil, 1978) and the unsolvated crystals (Blom, Roelofsen & Kanters, 1982). The C(20)—C(26) fragment of the molecule is planar to within 0.02 Å. The double bonds C(21)=C(22) [1.335 (4) Å] and C(24)=C(25) [1.337 (4) Å] are equal and are close to the average C=C bond length found for cyclohexa-1,3-dienes (1.332 Å; Allen, Kennard, Watson, Brammer, Orpen & Taylor, 1987). The C(26) atom has a planar trigonal environment; the C(23)=C(26), C(26)—C(27) and C(26)—C(33) bond lengths are 1.376 (4), 1.485 (4) and 1.483 (4) Å, respectively. The C(1)—C(20) bond length [1.597 (4) Å] is unusually long, just as it is in the ethyl acetate solvate structure (1.589 Å; Allemand & Gerdil, 1978) and in the two independent molecules of the unsolvated structure (1.60 and 1.61 Å; Blom, Roelofsen & Kanters, 1982). In these three structures, the differences between the bond lengths and angles are not greater than 3σ but there are, however, some conformational differences. In the benzene solvate structure, the dihedral angles between the phenyl rings C(27)—C(32) and C(33)—C(38), and the plane composed of atoms C(23), C(27) and C(33), are 51.68 (9) and 54.9 (1)°, respectively, and the dihedral angle between the average planes of the C(20)—C(25) and C(14)—C(19) fragments is 55.1 (1)°. The equivalent angles in the ethyl acetate solvate structure are 53.1, 55.2 and 52.2° (Allemand & Gerdil, 1978), and in the two independent molecules of the unsolvated structure the angles are 93.3, 62.6 and 41.6°, and 90.8, 45.9 and 39.4°, respectively (Blom, Roelofsen & Kanters, 1982). The packing in the ethyl acetate and benzene solvate structures is similar. The unit-cell lengths are similar but the differences in an-

gles are more significant (*cf.* a = 13.690, b = 12.198, c = 10.776 Å, α = 81.96, β = 79.06 and γ = 70.15° for the ethyl acetate solvate crystal).
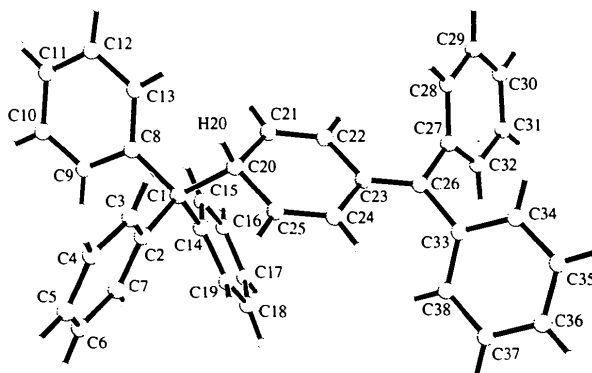

Fig. 1. A general view of 1-diphenylmethylene-4-(triphenylmethyl)-cyclohexa-2,5-diene with the labelling of non-H atoms shown.

Experimental

Crystal data

$C_{38}H_{30}.C_6H_6$	Mo $K\alpha$ radiation
M_r = 564.73	λ = 0.71073 Å
Triclinic	Cell parameters from 24
$P\bar{1}$	reflections
a = 10.555 (3) Å	θ = 12–12.5°
b = 12.680 (3) Å	μ = 0.065 mm^{-1}
c = 13.675 (4) Å	T = 193 (2) K
α = 62.42 (2)°	Parallelepiped
β = 87.01 (2)°	0.6 × 0.5 × 0.4 mm
γ = 85.63 (2)°	Light yellow
V = 1617.2 (8) Å^3	
Z = 2	
D_x = 1.160 Mg m^{-3}	

Data collection

Syntex $P2_1$ diffractometer	θ_{max} = 24.05°
$\theta/2\theta$ scans	h = $-2 \rightarrow 11$
Absorption correction:	k = $-14 \rightarrow 14$
none	l = $-15 \rightarrow 15$
5197 measured reflections	2 standard reflections
4882 independent reflections	monitored every 98
3251 observed reflections	reflections
[$I > 2\sigma(I)$]	intensity decay: 1.1%
R_{int} = 0.0516	

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max}$ = -0.199
$R(F)$ = 0.0525	$\Delta\rho_{max}$ = 0.269 e Å^{-3}
$wR(F^2)$ = 0.1790	$\Delta\rho_{min}$ = -0.280 e Å^{-3}
S = 1.038	Atomic scattering factors
4871 reflections	from *International Tables*
541 parameters	*for Crystallography* (1992,
All H-atom parameters	Vol. C, Tables 4.2.6.8 and
refined	6.1.1.4)
$w = 1/[\sigma^2(F_o^2) + (0.0976P)^2$	
$+ 0.4246P]$	
where $P = (F_o^2 + 2F_c^2)/3$	

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}
C1	−0.3106 (3)	−0.2543 (2)	0.0643 (2)	0.0326 (6)
C2	−0.1935 (3)	−0.2699 (2)	0.1337 (2)	0.0337 (6)
C3	−0.1846 (3)	−0.2002 (3)	0.1874 (2)	0.0431 (7)
C4	−0.0852 (3)	−0.2190 (3)	0.2567 (3)	0.0521 (9)
C5	0.0079 (3)	−0.3072 (3)	0.2754 (3)	0.0577 (10)
C6	0.0014 (3)	−0.3776 (3)	0.2244 (3)	0.0533 (9)
C7	−0.0984 (3)	−0.3604 (3)	0.1549 (2)	0.0445 (8)
C8	−0.4154 (3)	−0.3173 (2)	0.1507 (2)	0.0319 (6)
C9	−0.3991 (3)	−0.4406 (2)	0.2185 (2)	0.0377 (7)
C10	−0.4870 (3)	−0.5027 (3)	0.2990 (2)	0.0426 (8)
C11	−0.5950 (3)	−0.4434 (3)	0.3150 (2)	0.0462 (8)
C12	−0.6134 (3)	−0.3220 (3)	0.2506 (2)	0.0449 (8)
C13	−0.5242 (3)	−0.2601 (3)	0.1699 (2)	0.0383 (7)
C14	−0.2884 (3)	−0.3081 (2)	−0.0158 (2)	0.0339 (7)
C15	−0.3860 (3)	−0.3611 (2)	−0.0386 (2)	0.0405 (7)
C16	−0.3696 (4)	−0.4037 (3)	−0.1157 (3)	0.0494 (8)
C17	−0.2562 (4)	−0.3959 (3)	−0.1702 (3)	0.0544 (9)
C18	−0.1588 (4)	−0.3416 (3)	−0.1519 (3)	0.0518 (8)
C19	−0.1752 (3)	−0.2964 (3)	−0.0762 (2)	0.0422 (7)
C20	−0.3479 (3)	−0.1162 (2)	−0.0110 (2)	0.0337 (7)
C21	−0.4568 (3)	−0.0985 (2)	−0.0847 (2)	0.0342 (7)
C22	−0.4565 (3)	−0.0273 (2)	−0.1934 (2)	0.0355 (7)
C23	−0.3471 (2)	0.0400 (2)	−0.2536 (2)	0.0331 (6)
C24	−0.2385 (3)	0.0222 (2)	−0.1855 (2)	0.0348 (7)
C25	−0.2381 (3)	−0.0475 (2)	−0.0764 (2)	0.0362 (7)
C26	−0.3477 (3)	0.1151 (2)	−0.3653 (2)	0.0348 (7)
C27	−0.4634 (3)	0.1464 (2)	−0.4329 (2)	0.0359 (7)
C28	−0.5763 (3)	0.1903 (3)	−0.4052 (3)	0.0478 (8)
C29	−0.6818 (3)	0.2200 (3)	−0.4704 (3)	0.0585 (10)
C30	−0.6758 (3)	0.2055 (3)	−0.5652 (3)	0.0545 (9)
C31	−0.5639 (3)	0.1658 (3)	−0.5959 (3)	0.0477 (8)
C32	−0.4585 (3)	0.1368 (3)	−0.5303 (2)	0.0415 (7)
C33	−0.2325 (3)	0.1731 (2)	−0.4282 (2)	0.0357 (7)
C34	−0.2375 (3)	0.2970 (3)	−0.4935 (2)	0.0410 (7)
C35	−0.1335 (3)	0.3534 (3)	−0.5561 (2)	0.0496 (8)
C36	−0.0240 (3)	0.2880 (3)	−0.5570 (3)	0.0534 (9)
C37	−0.0170 (3)	0.1647 (3)	−0.4944 (3)	0.0541 (9)
C38	−0.1209 (3)	0.1082 (3)	−0.4302 (2)	0.0445 (8)
C1S	−0.1602 (8)	0.1440 (8)	0.2444 (8)	0.149 (3)
C2S	−0.1369 (9)	0.1050 (6)	0.1644 (15)	0.180 (6)
C3S	−0.1713 (6)	0.1783 (6)	0.0572 (8)	0.112 (2)
C4S	−0.2240 (5)	0.2821 (5)	0.0336 (4)	0.0844 (13)
C5S	−0.2451 (6)	0.3185 (5)	0.1108 (4)	0.101 (2)
C6S	−0.2134 (6)	0.2488 (6)	0.2170 (5)	0.107 (2)

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

C1—C14	1.537 (4)	C22—C23	1.463 (4)
C1—C2	1.543 (4)	C23—C26	1.376 (4)
C1—C8	1.544 (4)	C23—C24	1.453 (4)
C1—C20	1.597 (4)	C24—C25	1.337 (4)
C20—C25	1.493 (4)	C26—C33	1.483 (4)
C20—C21	1.503 (4)	C26—C27	1.485 (4)
C21—C22	1.335 (4)		
C14—C1—C2	113.4 (2)	C21—C22—C23	122.8 (3)
C14—C1—C8	111.1 (2)	C26—C23—C24	122.8 (2)
C2—C1—C8	104.2 (2)	C26—C23—C22	122.8 (2)
C14—C1—C20	105.6 (2)	C24—C23—C22	114.4 (2)
C2—C1—C20	110.5 (2)	C25—C24—C23	123.5 (3)
C8—C1—C20	112.1 (2)	C24—C25—C20	124.1 (3)
C25—C20—C21	110.9 (2)	C23—C26—C33	122.6 (2)
C25—C20—C1	113.1 (2)	C23—C26—C27	123.2 (2)
C21—C20—C1	111.6 (2)	C33—C26—C27	114.3 (2)
C22—C21—C20	124.3 (3)		

Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

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Microbial Transformation of a Dihydroxybiphenyl

MOHAMMAD SONDOSSI* AND BARRY A. LLOYD

Departments of Microbiology and Chemistry, Weber State University, Ogden, Utah 84408, USA

DIANE BARIAULT AND MICHEL SYLVESTRE

Université du Québec, INRS-Santé, 245 Boulevard Hymus, Pointe-Claire, Québec, Canada H9R 1G6

MICHEL SIMARD

Laboratoire de Diffraction des Rayons X, Départment de Chimie, Université de Montréal, CP 6128, Succ. A, Montréal, Québec, Canada H3C 3J7

Abstract

The X-ray crystal structure of (3S,4R)-3,4-dihydroxy-5-(3-hydroxyphenyl)-5-cyclohexen-1-one, $C_{12}H_{12}O_4$, was determined and showed that the molecule consists of