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3,7-Diphenyl-5,6;10,11-dibenzotricyclo-[7.2.1.0^{3,8}]dodeca-5,7,10-trien-4-one

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Abstract

3,7-Diphenyl-5,6;10,11-dibenzotricyclo[7.2.1.0^{3,8}]dodeca-5,7,10-trien-4-one consists of a fused ring system containing four six-membered rings and a five-membered ring with two pendant phenyl groups. The four phenyl rings are planar while the cyclohexadienone ring exhibits a flattened 1,3-diplanar conformation.

Comment

The reaction of (1) with potassium *tert*-butoxide in the presence of diphenylbenzoisofuran (2) gives allene-like trapping products; however, an alternative alkyne intermediate was shown to provide the best rationalization for the mechanistic pathway (Taskesenligil, Kashyap, Watson & Balcı, 1993). The title compound (3) (Fig. 1) arises from the opening of an epoxide and subsequent rearrangement during chromatography over either SiO_2 or Al_2O_3. The phenyl rings are all planar (0.002–0.008 Å r.m.s. deviation) and there is no pyramidalization at C13 and C18 resulting from the C20 bridge (Watson, 1983). The cyclohexadienone ring exhibits a flattened 1,3-diplanar conformation (Bucourt, 1974). All bond lengths and angles are normal and are reproduced by molecular-mechanics modeling (Allinger, Yuh & Lii, 1989; Technical Utilization Corporation, 1992).

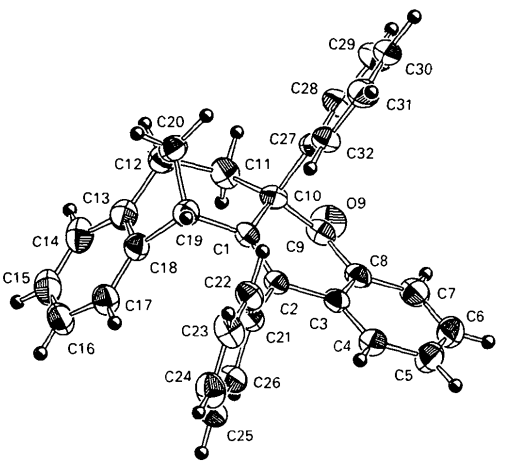


Fig. 1. Thermal ellipsoid drawing of compound (3). Thermal ellipsoids are drawn at the 35% prbability level while H atoms are represented by spheres of arbitrary size.

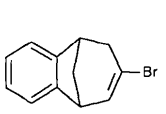
Experimental

Crystal data

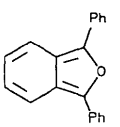
$C_{32}H_{24}O$
M_r = 424.54
Monoclinic
$P2_1/c$
a = 16.356 (4) Å
b = 8.070 (2) Å
c = 18.379 (3) Å
β = 114.55 (1)°
V = 2207 (2) Å^3
Z = 4
D_x = 1.278 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 12.5–27.5°
μ = 5.44 cm^{-1}
T = 295 K
Stable
0.50 × 0.38 × 0.33 mm
Colorless

Data collection

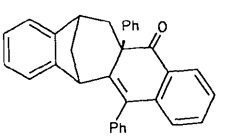
Rigaku AFC-6*S* diffractometer
ω–2θ scans
Absorption correction: empirical
T_{min} = 0.79, T_{max} = 1.00
4170 measured reflections
4022 independent reflections
2597 observed reflections [$I \geq 3\sigma(I)$]
R_{int} = 0.027
θ_{max} = 65.10°
h = 0 → 17
k = 0 → 9
l = −21 → 21
3 standard reflections monitored every 150 reflections
intensity variation: none



(1)



(2)



(3)

Refinement

Refinement on F	$\Delta\rho_{max}$ = 0.17 e Å^{-3}
Final R = 0.0488	$\Delta\rho_{min}$ = −0.21 e Å^{-3}
wR = 0.0513	Extinction correction: Zachariasen type 2 Gaussian isotropic
S = 4.187	Extinction coefficient: 26.14×10^{-7}
2597 reflections	Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2A, 2.3.1)
395 parameters	
All H-atom parameters refined	
Weighting scheme based on measured e.s.d.'s	
$(\Delta/\sigma)_{max}$ = 0.012	

Data collection and cell refinement: *MSC/AFC Data Collection and Refinement Software* (Rigaku Corporation, 1988). Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *TEXSAN MITHRIL*. Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.7641 (2)	0.3893 (3)	0.6670 (2)	0.0448
C2	0.7542 (2)	0.3286 (3)	0.5958 (2)	0.0445
C3	0.7009 (2)	0.1767 (3)	0.5618 (2)	0.0474
C4	0.6753 (2)	0.1302 (4)	0.4828 (2)	0.0583
C5	0.6239 (2)	−0.0104 (4)	0.4521 (2)	0.0710
C6	0.5982 (2)	−0.1084 (5)	0.5001 (3)	0.0745
C7	0.6252 (2)	−0.0677 (4)	0.5791 (2)	0.0650
C8	0.6750 (2)	0.0747 (3)	0.6104 (2)	0.0499
C9	0.7016 (2)	0.1190 (3)	0.6945 (2)	0.0536
O9	0.7040 (2)	0.0178 (3)	0.7444 (1)	0.0774
C10	0.7216 (2)	0.3025 (3)	0.7169 (2)	0.0485
C11	0.7805 (2)	0.3163 (5)	0.8078 (2)	0.0626
C12	0.8355 (2)	0.4784 (4)	0.8339 (2)	0.0643
C13	0.9254 (2)	0.4616 (4)	0.8288 (2)	0.0606
C14	1.0099 (2)	0.4170 (4)	0.8868 (2)	0.0728
C15	1.0829 (3)	0.4178 (4)	0.8669 (3)	0.0791
C16	1.0716 (2)	0.4591 (4)	0.7912 (3)	0.0787
C17	0.9871 (2)	0.5016 (4)	0.7318 (2)	0.0677
C18	0.9143 (2)	0.5028 (3)	0.7524 (2)	0.0552
C19	0.8157 (2)	0.5464 (4)	0.7031 (2)	0.0522
C20	0.7914 (2)	0.6120 (4)	0.7702 (2)	0.0587
C21	0.7932 (2)	0.4143 (3)	0.5456 (2)	0.0466
C22	0.7592 (2)	0.5615 (4)	0.5069 (2)	0.0568
C23	0.7924 (2)	0.6358 (5)	0.4575 (2)	0.0688
C24	0.8627 (2)	0.5621 (5)	0.4465 (2)	0.0738
C25	0.8974 (2)	0.4162 (5)	0.4843 (2)	0.0682
C26	0.8645 (2)	0.3409 (4)	0.5340 (2)	0.0577
C27	0.6242 (2)	0.3717 (3)	0.6936 (2)	0.0501
C28	0.5758 (2)	0.3157 (4)	0.7357 (2)	0.0684
C29	0.4880 (3)	0.3693 (5)	0.7149 (2)	0.0805
C30	0.4483 (2)	0.4761 (5)	0.6531 (2)	0.0722
C31	0.4944 (2)	0.5313 (4)	0.6103 (2)	0.0699
C32	0.5823 (2)	0.4779 (4)	0.6304 (2)	0.0569

Table 2. *Geometric parameters* (Å, °)

C1—C2	1.343 (3)	C13—C18	1.380 (4)
C1—C10	1.531 (3)	C14—C15	1.385 (5)
C1—C19	1.514 (4)	C15—C16	1.366 (5)
C2—C3	1.483 (4)	C16—C17	1.402 (5)
C2—C21	1.493 (3)	C17—C18	1.392 (4)
C3—C4	1.385 (4)	C18—C19	1.527 (4)
C3—C8	1.402 (3)	C19—C20	1.538 (4)
C4—C5	1.385 (4)	C21—C22	1.377 (4)
C5—C6	1.374 (5)	C21—C26	1.400 (4)
C6—C7	1.372 (5)	C22—C23	1.376 (4)
C7—C8	1.388 (4)	C23—C24	1.381 (5)
C8—C9	1.466 (4)	C24—C25	1.365 (5)
C9—O9	1.217 (3)	C25—C26	1.379 (4)
C9—C10	1.536 (4)	C27—C28	1.392 (4)
C10—C11	1.547 (4)	C27—C32	1.376 (4)
C10—C27	1.570 (4)	C28—C29	1.394 (5)
C11—C12	1.547 (4)	C29—C30	1.358 (5)
C12—C13	1.517 (4)	C30—C31	1.369 (5)
C12—C20	1.533 (4)	C31—C32	1.395 (4)
C13—C14	1.397 (4)		
C2—C1—C10	121.0 (2)	C12—C13—C18	108.6 (3)
C2—C1—C19	123.5 (2)	C14—C13—C18	120.5 (3)
C10—C1—C19	115.4 (2)	C13—C14—C15	119.1 (4)
C1—C2—C3	121.6 (2)	C14—C15—C16	120.2 (4)
C1—C2—C21	121.6 (2)	C15—C16—C17	121.7 (4)
C3—C2—C21	116.8 (2)	C16—C17—C18	117.9 (3)
C2—C3—C4	122.3 (3)	C13—C18—C17	120.7 (3)
C2—C3—C8	119.8 (2)	C13—C18—C19	108.6 (3)
C4—C3—C8	117.9 (3)	C17—C18—C19	130.7 (3)
C3—C4—C5	121.1 (3)	C1—C19—C18	109.0 (2)
C4—C5—C6	120.4 (3)	C1—C19—C20	110.9 (2)
C5—C6—C7	119.4 (4)	C18—C19—C20	99.5 (2)
C6—C7—C8	120.8 (3)	C12—C20—C19	100.0 (2)
C3—C8—C7	120.2 (3)	C2—C21—C22	122.2 (2)
C3—C8—C9	119.5 (3)	C2—C21—C26	119.7 (3)
C7—C8—C9	120.3 (3)	C22—C21—C26	118.0 (3)
C8—C9—O9	122.3 (3)	C21—C22—C23	122.1 (3)
C8—C9—C10	117.0 (2)	C22—C23—C24	119.3 (3)
O9—C9—C10	120.6 (3)	C23—C24—C25	119.5 (3)
C1—C10—C9	112.3 (2)	C24—C25—C26	121.5 (4)
C1—C10—C11	112.3 (2)	C21—C26—C25	119.5 (3)
C1—C10—C27	109.7 (2)	C10—C27—C28	118.7 (3)
C9—C10—C11	109.0 (2)	C10—C27—C32	123.1 (2)
C9—C10—C27	101.0 (2)	C28—C27—C32	118.1 (3)
C11—C10—C27	112.0 (2)	C27—C28—C29	120.4 (3)
C10—C11—C12	114.6 (3)	C28—C29—C30	120.4 (4)
C11—C12—C13	111.0 (3)	C29—C30—C31	120.1 (3)
C11—C12—C20	109.2 (3)	C30—C31—C32	120.0 (3)
C13—C12—C20	100.2 (3)	C27—C32—C31	120.9 (3)
C12—C13—C14	130.8 (3)		

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Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71307 (29 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1058]

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