Table 3. *Bond distances* (Å) *and angles* (°)

	(2)	(3)		(2)	(3)
O10—C10	1.205 (6)	1.220 (7)	C6—C7	1.452 (7)	1.460 (8)
O16—C3	1.409 (6)	1.422 (6)	C7—C8	1.321 (7)	1.315 (9)
O16—C16	1.366 (7)	1.447 (8)	C8—C9	1.481 (6)	1.495 (9)
O17—C3	1.431 (5)	1.411 (7)	C9—C10	1.532 (6)	1.513 (7)
O17—C17	1.401 (7)	1.453 (6)	C9—C13	1.569 (6)	1.562 (9)
C1—C2	1.542 (7)	1.539 (7)	C10—C11	1.502 (6)	1.525 (10)
C1—C14	1.555 (7)	1.549 (9)	C11—C12	1.539 (7)	1.556 (8)
C1—C15	1.520 (7)	1.533 (9)	C12—C13	1.565 (6)	1.556 (9)
C2—C3	1.526 (7)	1.521 (10)	C12—C15	1.521 (7)	1.535 (9)
C2—C11	1.572 (6)	1.577 (8)	C13—C14	1.543 (6)	1.546 (8)
C3—C4	1.526 (6)	1.544 (7)	C16—C17	1.421 (9)	
C4—C5	1.483 (6)	1.485 (9)	C16—C18		1.528 (7)
C4—C9	1.576 (6)	1.579 (8)	C16—C19		1.506 (9)
C4—C14	1.547 (7)	1.571 (9)	C17—C20		1.502 (10)
C5—C6	1.320 (7)	1.336 (9)	C17—C21		1.519 (9)
C3—O16—C16	108.4 (4)	118.2 (4)	C8—C9—C10	115.5 (4)	116.5 (5)
C3—O17—C17	107.8 (4)	117.1 (4)	C8—C9—C13	120.1 (4)	118.2 (4)
C2—C1—C14	100.1 (4)	100.2 (5)	C10—C9—C13	101.1 (3)	101.0 (5)
C2—C1—C15	104.7 (4)	104.0 (4)	O10—C10—C9	126.6 (4)	127.2 (6)
C14—C1—C15	103.2 (4)	103.1 (4)	O10—C10—C11	127.7 (4)	126.2 (5)
C1—C2—C3	103.3 (4)	103.8 (4)	C9—C10—C11	104.8 (4)	105.4 (5)
C1—C2—C11	103.0 (4)	103.4 (4)	C2—C11—C10	111.2 (4)	110.7 (5)
C3—C2—C11	111.8 (4)	112.5 (5)	C2—C11—C12	102.3 (4)	102.6 (4)
O16—C3—O17	106.9 (3)	111.8 (4)	C10—C11—C12	101.4 (4)	100.4 (4)
O16—C3—C2	113.7 (4)	107.7 (4)	C11—C12—C13	101.1 (4)	100.4 (5)
O16—C3—C4	113.5 (4)	116.4 (5)	C11—C12—C15	104.9 (4)	103.3 (4)
O17—C3—C2	110.1 (4)	113.7 (5)	C13—C12—C15	102.6 (4)	103.3 (4)
O17—C3—C4	110.3 (4)	105.7 (4)	C9—C13—C12	107.9 (4)	109.1 (4)
C2—C3—C4	102.3 (4)	101.3 (5)	C9—C13—C14	90.5 (3)	91.1 (5)
C3—C4—C5	116.3 (4)	118.7 (5)	C12—C13—C14	103.1 (4)	103.3 (5)
C3—C4—C9	110.6 (3)	112.2 (4)	C1—C14—C4	108.4 (4)	108.0 (4)
C3—C4—C14	103.7 (4)	103.1 (5)	C1—C14—C13	103.0 (4)	103.7 (5)
C5—C4—C9	114.9 (4)	113.5 (5)	C4—C14—C13	90.8 (3)	90.1 (4)
C5—C4—C14	118.1 (4)	115.6 (4)	C1—C15—C12	95.3 (4)	95.6 (5)
C9—C4—C14	90.0 (3)	89.6 (4)	O16—C16—C17	109.6 (5)	
C4—C5—C6	123.0 (4)	124.4 (5)	O16—C16—C18		107.7 (5)
C5—C6—C7	121.7 (4)	120.3 (6)	O16—C16—C19		110.2 (5)
C6—C7—C8	122.2 (4)	123.0 (6)	C18—C16—C19		110.5 (5)
C7—C8—C9	122.1 (4)	121.4 (5)	O17—C17—C16	106.4 (5)	
C4—C9—C8	116.1 (4)	117.1 (5)	O17—C17—C20		111.8 (4)
C4—C9—C10	111.8 (3)	110.8 (4)	O17—C17—C21		105.6 (5)
C4—C9—C13	88.7 (3)	89.2 (4)	C20—C17—C21		110.2 (5)

For (2): structure solution by direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); refinement by full-matrix least squares (Enraf-Nonius, 1990). For (3): structure solution by direct methods and full-matrix least-squares refinement with *SHELXTL-Plus* (Sheldrick, 1991). H atoms were located from difference maps but included in calculated positions. No attempt was made to model the disorder of C16 in (2) suggested by its bonding and thermal parameters.

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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 71265 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1054]

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8-Pentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undec-3$'$-yl-*exo*-pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane

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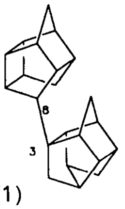
Abstract

The title compound is composed of two $C_{11}H_{14}$ geometric isomers, pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane (PCU) and pentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane (trishomocubane), connected by a single bond. The PCU isomer contains four five-membered rings, a four-membered ring and a six-membered ring fused into a cage-like structure, while the trishomocubane isomer is a fusion of six five-membered rings. Elongated bond lengths in these molecules are compared with literature structures and with the results of molecular-mechanics calculations.

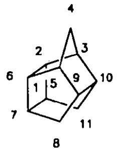
Comment

As part of a research program concerned with the synthesis and chemistry of substituted pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecanes, PCUs (Marchand, 1989), four isomeric $C_{22}H_{24}$ alkenes [compound (1) with connecting double bond] were prepared by dimerization of PCU. The structure of one isomer has been reported (Flippen-Anderson, Gilardi, George, Marchand, Jin & Deshpande, 1988). A further series of reactions led to the isolation of the title compound whose structure we now report.

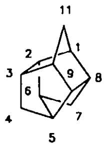
The title compound (1) is composed of a pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane [PCU, (2)] moiety and a pentacyclo[6.3.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane [trishomocubane, (3) and (4)] moiety connected by a single bond. Compounds (2) and (3) are geometric isomers with formula $C_{11}H_{14}$. The PCU moiety (2) can be described as a fusion of four five-membered rings, a four-membered ring and a six-membered ring. Trishomocubane (3) can be described as a fusion of six five-membered rings. Trishomocubane is calculated by molecular mechanics (Allinger, Yuh & Lii, 1989; Technical Utilization Corporation, 1992) to be less strained than PCU [ΔH_f = 6.30 and 3.50 kJ for (2) and (3), respectively] and is formed *via* a carbenium-ion rearrangement of (2) (Kent, Godleski, Osawa & Schleyer, 1977; Dekker, Oliver & Venter, 1980).



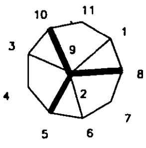
(1)



(2)



(3)



(4)

Trishomocubane has a threefold axis of symmetry through C2 and C9 as indicated by structure (4). The six bonds associated with C2 and C9 are calculated by molecular mechanics to be 1.578 Å. The bonds C1—C8, C5—C6 and C3—C10 are 1.545 Å with the remainder being 1.527 Å. The *ab initio* (1.582, 1.543 and 1.534 Å) and *AM*1 (1.578, 1.565 and 1.532 Å) values have been reported previously (Watson, Kashyap, Krawiec, Marchand, Madhusudhan Reddy & Gadgil, 1992). The parameters in the latest *MM*3 (Technical Utilization Corporation, 1992) program lead to longer bond distances which agree more closely with the *ab initio* values. Ignoring the three bonds associated with C3, which joins the two cage moieties, the three classes of bonds in (1) average 1.564 (5), 1.528 (2) and 1.516 (2) Å, respectively. These data agree well with the X-ray data on three similar compounds [averaged values 1.567 (5), 1.526 (8) and 1.518 (5) Å] and with literature data (Watson *et al.*, 1992). *MM*3 molecular-mechanics distances, r_g, are parameterized to fit electron diffraction data and are approximately 0.006 Å longer than C(sp^3)—C(sp^3) values determined by X-ray diffraction (Jones, 1984; Technical Utilization Corporation, 1992; Sim & Sutton, 1973). The average difference between the observed and calculated distances for the trishomocubane moiety in compound (1) is 0.012 Å with values ranging from 0.003 to 0.023 Å.

There are five different valence angles in the ideal trishomocubane. Molecular mechanics calculates C1—C2—C3 = 103.2, C2—C1—C8 = 99.6, C2—C1—C11 = 105.4, C3—C4—C5 = 95.1 and C1—C8—C7 = 101.9°. The averaged values of these five angles for trishomocubane found in (1) are 104.0 (2), 98.6(7), 105.3(6), 94.6(5) and 103.1(8)°, respectively. The largest deviations are associated with the junction at C3. These data agree well with those reported for similar compounds (Watson *et al.*, 1992). The ideal PCU moiety has mirror (C_s) symmetry. The average deviation between calculated (*MM*3) and observed values for the PCU moiety in (1) is 0.016 Å with values ranging from 0.003 to 0.027 Å. The greatest discrepancy involves the C2′—C3′ and C5′—C6′ bonds which are calculated to be 1.556 and 1.550 Å and are found to be 1.529 (3) and 1.528 (3) Å. This large discrepancy occurs in all of the measured PCU derivatives (Watson, Kashyap, Krawiec, Marchand & Tsay, 1993), and probably indicates a need for modification of the molecular-mechanics parameters for these systems.

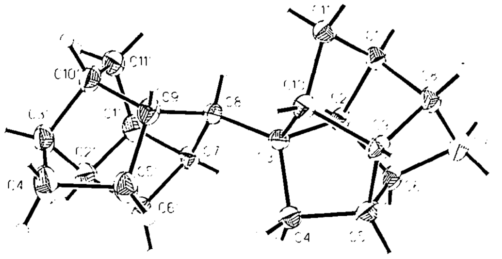


Fig. 1. Drawing of compound (1) with thermal ellipsoids at the 35% probability level and H atoms drawn as spheres of arbitrary size.

Experimental

Crystal data

$C_{22}H_{26}$
M_r = 290.45
Triclinic
$P\bar{1}$
a = 6.357 (1) Å
b = 11.429 (1) Å
c = 11.706 (1) Å
α = 61.87 (1)°
β = 85.52 (1)°
γ = 87.56 (1)°
V = 747.7 (1) Å^3
Z = 2

D_x = 1.290 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 9.69–14.74°
μ = 0.067 mm^{-1}
T = 295 K
Prism
0.50 × 0.50 × 0.13 mm
Colorless

Data collection

Syntex $P2_1$ $R3m/\mu$ diffractometer

$R_{\rm int}$ = 0.032
$\theta_{\rm max}$ = 50°

ω scans
Absorption correction:
ψ scan
$T_{\min} = 0.831$, $T_{\max} = 0.909$
2710 measured reflections
2639 independent reflections
2211 observed reflections
$[I \geq 3\sigma(I)]$

$h = -7 \rightarrow 7$
$k = -11 \rightarrow 13$
$l = 0 \rightarrow -13$
2 standard reflections
monitored every 100 reflections
intensity variation: 2%

Refinement

Refinement on F
Final $R = 0.0504$
$wR = 0.0651$
$S = 1.371$
2211 reflections
303 parameters
Only coordinates of H atoms refined
$w = 1/[\sigma^2(F) - 0.00139F^2]$

$(\Delta/\sigma)_{\max} = 0.009$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.29$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Refinement was by block-cascade least-squares method. All computer programs were supplied by Nicolet Instrument Corporation (1986) for Desktop 30 Microeclipse and NOVA4/*C* configuration. Data collection: *R*3*m*/μ diffractometer control program. Cell refinement: *R*3*m*/μ diffractometer control program. Data reduction: *SHELXTL* (Sheldrick, 1986). Program(s) used to solve structure: *SHELXTL*. Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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

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

	x	y	z	$U_{\rm eq}$
C1	0.3890 (3)	0.1692 (2)	0.5411 (2)	0.033 (1)
C2	0.1873 (3)	0.2107 (2)	0.4608 (2)	0.032 (1)
C3	0.2794 (3)	0.2748 (2)	0.3151 (2)	0.030 (1)
C4	0.1803 (3)	0.4139 (2)	0.2521 (2)	0.042 (1)
C5	0.2124 (3)	0.4415 (2)	0.3641 (2)	0.042 (1)
C6	0.0931 (3)	0.3298 (2)	0.4796 (2)	0.041 (1)
C7	0.1960 (3)	0.3220 (2)	0.5956 (2)	0.045 (1)
C8	0.4155 (3)	0.2963 (2)	0.5510 (2)	0.038 (1)
C9	0.4425 (3)	0.3959 (2)	0.4022 (2)	0.036 (1)
C10	0.5023 (3)	0.3051 (2)	0.3376 (2)	0.032 (1)
C11	0.5716 (3)	0.1744 (2)	0.4477 (2)	0.034 (1)
C1′	0.0225 (3)	0.0776 (2)	0.1621 (1)	0.047 (1)
C2′	0.0401 (3)	0.1862 (2)	0.0184 (2)	0.050 (1)
C3′	0.2621 (4)	0.1768 (2)	−0.0374 (2)	0.053 (1)
C4′	0.3427 (4)	0.3175 (2)	−0.0982 (2)	0.068 (1)
C5′	0.2953 (4)	0.3333 (2)	0.0237 (2)	0.050 (2)
C6′	0.0634 (3)	0.2944 (2)	0.0616 (2)	0.046 (1)
C7′	0.0466 (3)	0.1860 (2)	0.2058 (2)	0.037 (1)
C8′	0.2635 (3)	0.1821 (2)	0.2552 (2)	0.032 (1)
C9′	0.4015 (3)	0.2129 (2)	0.1316 (2)	0.040 (1)
C10′	0.3785 (3)	0.1023 (2)	0.0884 (2)	0.047 (1)
C11′	0.2215 (4)	−0.0047 (2)	0.1775 (2)	0.050 (1)

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

C1—C2	1.571 (2)	C1′—C2′	1.549 (2)
C1—C8	1.528 (3)	C1′—C7′	1.563 (4)
C1—C11	1.514 (3)	C1′—C11′	1.514 (3)
C2—C3	1.579 (2)	C2′—C3′	1.529 (3)
C2—C6	1.566 (3)	C2′—C6′	1.557 (4)
C3—C4	1.532 (2)	C3′—C4′	1.513 (3)
C3—C10	1.544 (2)	C3′—C10′	1.541 (3)
C3—C8′	1.532 (3)	C4′—C5′	1.527 (4)
C4—C5	1.516 (4)	C5′—C6′	1.528 (3)
C5—C6	1.525 (2)	C5′—C9′	1.536 (2)
C5—C9	1.559 (3)	C6′—C7′	1.551 (2)
C6—C7	1.516 (3)	C7′—C8′	1.527 (3)
C7—C8	1.517 (3)	C8′—C9′	1.527 (3)
C8—C9	1.568 (2)	C9′—C10′	1.583 (4)
C9—C10	1.568 (3)	C10′—C11′	1.522 (3)
C10—C11	1.518 (2)		
C2—C1—C8	98.5 (1)	C2′—C1′—C7′	89.8 (2)
C2—C1—C11	106.1 (1)	C2′—C1′—C11′	104.3 (2)
C8—C1—C11	103.0 (1)	C7′—C1′—C11′	112.0 (2)
C1—C2—C3	103.9 (1)	C1′—C2′—C3′	107.4 (2)
C1—C2—C6	103.9 (2)	C1′—C2′—C6′	90.3 (2)
C3—C2—C6	105.0 (1)	C3′—C2′—C6′	103.1 (2)
C2—C3—C4	104.1 (1)	C2′—C3′—C4′	103.6 (2)
C2—C3—C10	97.6 (1)	C2′—C3′—C10′	100.5 (2)
C4—C3—C10	101.4 (1)	C4′—C3′—C10′	104.6 (2)
C2—C3—C8′	112.4 (1)	C3′—C4′—C5′	94.9 (2)
C4—C3—C8′	120.7 (2)	C4′—C5′—C6′	103.8 (2)
C10—C3—C8′	117.3 (1)	C4′—C5′—C9′	104.9 (2)
C3—C4—C5	95.2 (1)	C6′—C5′—C9′	100.4 (1)
C4—C5—C6	103.7 (2)	C2′—C6′—C5′	102.7 (2)
C4—C5—C9	105.6 (2)	C2′—C6′—C7′	89.9 (1)
C6—C5—C9	99.1 (1)	C5′—C6′—C7′	107.8 (2)
C2—C6—C5	98.2 (2)	C1′—C7′—C6′	90.0 (2)
C2—C6—C7	105.5 (1)	C1′—C7′—C8′	112.5 (2)
C5—C6—C7	103.4 (2)	C6′—C7′—C8′	105.1 (1)
C6—C7—C8	94.3 (2)	C3—C8′—C7′	113.0 (2)
C1—C8—C7	103.4 (2)	C3—C8′—C9′	117.5 (1)
C1—C8—C9	98.3 (2)	C7′—C8′—C9′	99.5 (1)
C7—C8—C9	105.7 (1)	C5′—C9′—C8′	104.9 (2)
C5—C9—C8	103.7 (1)	C5′—C9′—C10′	102.0 (2)
C5—C9—C10	103.7 (2)	C8′—C9′—C10′	111.0 (1)
C8—C9—C10	104.0 (1)	C3′—C10′—C9′	102.6 (2)
C3—C10—C9	99.6 (1)	C3′—C10′—C11′	102.7 (2)
C3—C10—C11	103.3 (1)	C9′—C10′—C11′	112.5 (2)
C9—C10—C11	104.9 (2)	C1′—C11′—C10′	100.7 (1)
C1—C11—C10	94.4 (1)		

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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 71263 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH1068]

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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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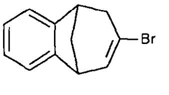
Abstract

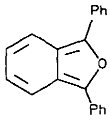
3,7-Diphenyl-5,6;10,11-dibenzotricyclo$[7.2.1.0^{3,8}]$dodeca-5,7,10-trien-4-one consists of a fused ring system containing four six-membered rings and a five-membered ring with two pendant phenyl groups. The four phenyl rings are planar while the cyclohexadienone ring exhibits a flattened 1,3-diplanar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



(1)



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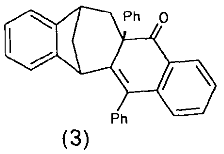

(3)

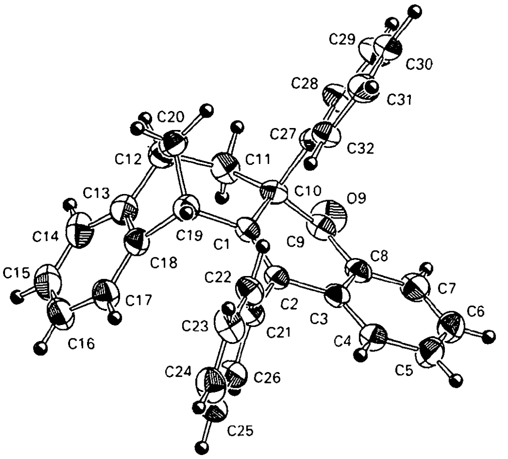

Fig. 1. Thermal ellipsoid drawing of compound (3). Thermal ellipsoids are drawn at the 35% prbability level while H atoms are represented by spheres of arbitrary size.

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
$\beta = 114.55$ (1)°
$V = 2207$ (2) Å^3
$Z = 4$

$D_x = 1.278$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 25 reflections
$\theta = 12.5\text{-}27.5°$
$\mu = 5.44$ 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 $[I \geq 3\sigma(I)]$

$R_{\rm int} = 0.027$
$\theta_{\max} = 65.10°$
$h = 0 \rightarrow 17$
$k = 0 \rightarrow 9$
$l = -21 \rightarrow 21$
3 standard reflections monitored every 150 reflections intensity variation: none

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