Crystal structure of (1 α ,8 α ,9 β ,12 β)-1,8-diphenyl-13-oxatetracyclo-[6.4.1.0^{2,7}.0^{9,12}]trideca-2,4,6,10-tetraene, C₂₄H₁₈O

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Abstract

C₂₄H₁₈O, orthorhombic, *Pbca* (No. 61), *a* = 19.870(4) Å, *b* = 16.78(2) Å, *c* = 10.520(6) Å, *V* = 3507.6 Å³, *Z* = 8, *R*_{gt}(*F*) = 0.057, *wR*_{ref}(*F*²) = 0.147, *T* = 293 K.

Source of material

Colourless crystals were obtained from the slow evaporation of a methanol solution of the compound [1].

Experimental details

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation.

Discussion

The structure determination shows that the molecule approximates *mm* symmetry and that the phenyl groups are oriented in to the same side of the molecule as the O3 atom.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.16 × 0.40 × 0.40 mm
Wavelength:	Mo <i>K</i> _α radiation (0.7107 Å)
<i>μ</i> :	0.73 cm ⁻¹
Diffractionmeter, scan mode:	Rigaku AFC6R, <i>ω</i> / <i>2θ</i>
<i>2θ</i> _{max} :	50.2°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	3512, 3107
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 <i>σ</i> (<i>I</i> _{obs}), 1163
<i>N</i> (<i>param</i>) _{refined} :	227
Programs:	SIR92 [2], teXsan [3], SHELXL-97 [4], DIBABS [5]

Table 2. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1)	8c	-0.0084	-0.0028	-0.1725	0.098(4)
H(1a)	8c	-0.0193	0.0608	-0.4076	0.078(3)
H(2)	8c	0.0096	0.1275	-0.0779	0.098
H(2a)	8c	0.0000	0.1988	-0.3057	0.078
H(4)	8c	0.1527	0.1152	0.1292	0.078
H(5)	8c	0.1665	-0.0007	0.2495	0.078
H(6)	8c	0.1510	-0.1235	0.1576	0.078
H(7)	8c	0.1185	-0.1347	-0.0540	0.078
H(32)	8c	0.2188	0.1785	-0.2650	0.078
H(33)	8c	0.2694	0.3021	-0.2496	0.078
H(34)	8c	0.2306	0.3946	-0.1063	0.078
H(35)	8c	0.1377	0.3664	0.0170	0.078
H(36)	8c	0.0857	0.2441	-0.0008	0.078
H(82)	8c	0.1925	-0.0097	-0.4011	0.078
H(83)	8c	0.2150	-0.1084	-0.5498	0.078
H(84)	8c	0.1485	-0.2203	-0.5593	0.078
H(85)	8c	0.0601	-0.2350	-0.4220	0.078
H(86)	8c	0.0353	-0.1374	-0.2753	0.078

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Table 3. Atomic coordinates and displacement parameters (in Å²).

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
O(3)	8c	0.1353(1)	0.0722(1)	-0.2492(2)	0.054(2)	0.045(1)	0.051(1)	-0.006(1)	0.012(1)	-0.005(1)
C(1)	8c	0.0241(2)	0.0318(2)	-0.2159(3)	0.042(2)	0.061(3)	0.056(3)	0.000(2)	-0.005(2)	-0.002(2)
C(1a)	8c	-0.0022(2)	0.0768(3)	-0.3293(4)	0.060(3)	0.081(3)	0.058(3)	0.008(3)	-0.012(2)	-0.005(3)
C(2)	8c	0.0358(2)	0.1159(2)	-0.1545(4)	0.048(3)	0.053(2)	0.054(3)	0.000(2)	0.000(2)	-0.001(2)
C(2a)	8c	0.0078(2)	0.1473(3)	-0.2767(4)	0.060(3)	0.069(3)	0.065(3)	0.008(2)	-0.006(3)	0.008(3)
C(3)	8c	0.1131(2)	0.1177(2)	-0.1388(3)	0.052(3)	0.049(2)	0.040(2)	0.003(2)	0.007(2)	-0.007(2)
C(4)	8c	0.1462(2)	0.0653(3)	0.0927(4)	0.064(3)	0.075(3)	0.048(3)	-0.013(3)	-0.003(2)	0.003(3)
C(4a)	8c	0.1284(2)	0.0582(2)	-0.0324(4)	0.044(3)	0.054(2)	0.048(2)	-0.003(2)	0.000(2)	0.002(2)
C(5)	8c	0.1543(2)	-0.0041(3)	0.1642(4)	0.066(3)	0.095(4)	0.060(3)	-0.011(3)	-0.004(3)	0.016(3)
C(6)	8c	0.1448(2)	-0.0777(3)	0.1091(4)	0.070(3)	0.073(3)	0.071(3)	0.002(3)	-0.001(3)	0.027(3)
C(7)	8c	0.1259(2)	-0.0849(2)	-0.0177(4)	0.057(3)	0.058(3)	0.071(3)	0.001(2)	0.003(3)	0.007(2)
C(8)	8c	0.0974(2)	-0.0005(2)	-0.2243(4)	0.044(2)	0.045(2)	0.058(3)	-0.009(2)	0.005(2)	0.000(2)
C(8a)	8c	0.1187(2)	-0.0161(2)	-0.0883(4)	0.044(3)	0.057(3)	0.057(3)	-0.002(2)	0.001(2)	0.004(2)
C(31)	8c	0.1473(2)	0.1978(2)	-0.1339(3)	0.058(3)	0.048(2)	0.043(2)	0.000(2)	-0.002(2)	0.003(2)
C(32)	8c	0.2021(2)	0.2160(2)	-0.2080(3)	0.056(3)	0.058(3)	0.056(3)	-0.005(2)	0.003(2)	0.007(2)
C(33)	8c	0.2327(2)	0.2900(3)	-0.1983(4)	0.065(3)	0.070(3)	0.069(3)	-0.012(3)	-0.006(3)	0.017(3)
C(34)	8c	0.2092(3)	0.3455(3)	-0.1137(4)	0.092(4)	0.056(3)	0.070(3)	-0.015(3)	-0.019(3)	0.003(3)
C(35)	8c	0.1542(3)	0.3287(3)	-0.0398(4)	0.098(4)	0.060(3)	0.076(3)	-0.011(3)	0.000(3)	-0.016(3)
C(36)	8c	0.1233(2)	0.2552(2)	-0.0505(4)	0.088(4)	0.063(3)	0.068(3)	-0.014(3)	0.024(3)	-0.010(3)
C(81)	8c	0.1117(2)	-0.0630(2)	-0.3225(4)	0.052(3)	0.048(2)	0.055(3)	0.004(2)	-0.001(2)	0.000(2)
C(82)	8c	0.1654(2)	-0.0548(2)	-0.4050(4)	0.062(3)	0.053(3)	0.059(3)	0.004(2)	-0.005(3)	-0.005(2)
C(83)	8c	0.1790(2)	-0.1141(3)	-0.4941(4)	0.065(3)	0.074(3)	0.062(3)	0.006(3)	0.001(3)	-0.006(3)
C(84)	8c	0.1394(3)	-0.1808(3)	-0.4997(4)	0.086(4)	0.063(3)	0.078(3)	0.000(3)	-0.002(3)	-0.016(3)
C(85)	8c	0.0866(3)	-0.1893(2)	-0.4181(4)	0.091(4)	0.057(3)	0.089(3)	-0.019(3)	0.008(3)	-0.016(3)
C(86)	8c	0.0718(2)	-0.1311(2)	-0.3298(4)	0.072(3)	0.058(3)	0.075(3)	-0.016(3)	0.013(3)	-0.009(2)

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