

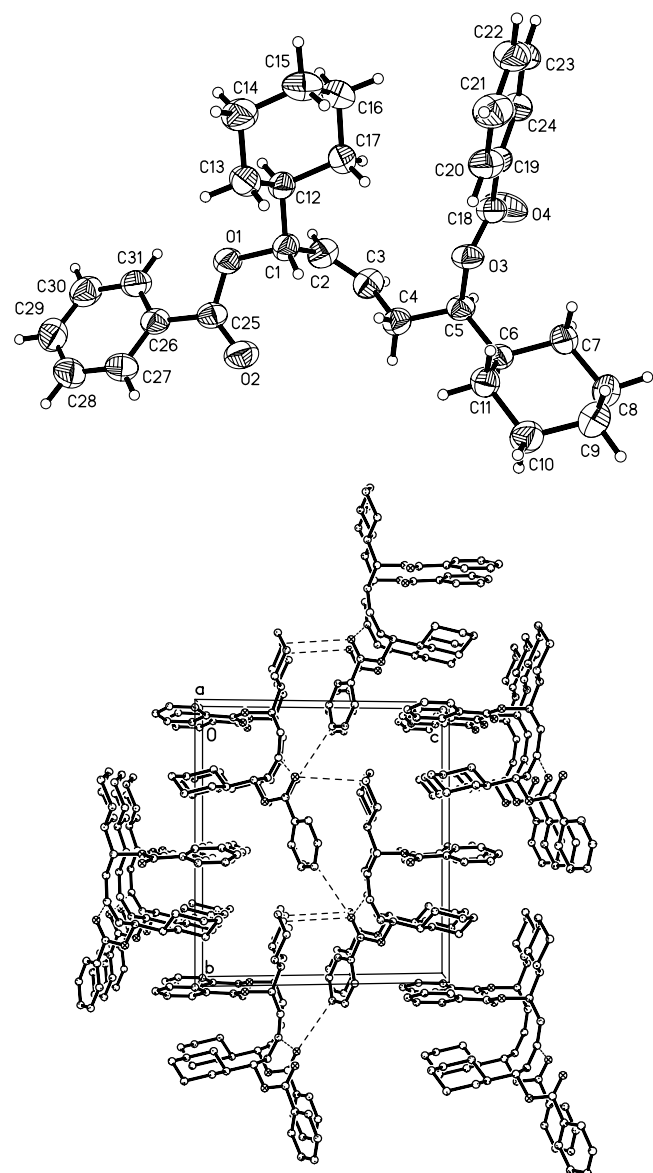
Crystal structure of (1*R*,5*R*,2*Z*)-1,5-dicyclohexylpent-2-ene-1,5-diyl dibenzoate, C₃₁H₃₈O₄

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Abstract

C₃₁H₃₈O₄, monoclinic, *P*2₁ (no. 4), *a* = 6.394(1) Å, *b* = 15.226(3) Å, *c* = 13.891(2) Å, β = 100.71(1)°, *V* = 1328.9 Å³, *Z* = 2, *R*_{gt}(*F*) = 0.045, *wR*_{ref}(*F*²) = 0.119, *T* = 293 K.

Source of material

The title compound was obtained as the major product by the reaction of (1*R*,5*R*,2*Z*)-1,5-dicyclohexylpent-2-ene-1,5-diol [1] with PhCOCl in the presence of pyridine in CH₂Cl₂ as solvent. The crude product was subjected to flash-column chromatography on silica gel (petroleum ether/ethyl acetate, 95:5) and crystallized from heptane and diethyl ether affording the title compound as colourless blocks.

Experimental details

H atoms were located on difference Fourier map, but refined with fixed individual displacement parameters, using a riding model with *d*(C—H) ranging from 0.93 to 0.98 Å. Friedel pairs were not merged.

Discussion

The title compound crystallizes with one molecule in the asymmetric unit of the acentric space group *P*2₁ (figure, top). The double bond C2=C3 is clearly characterized by the distance of 1.326(3) Å. The packing in the crystal structure (figure, bottom) is stabilized by three weak intermolecular interactions, of which always oxygen O2 of the carbonyl function of one benzoate moiety works as acceptor. In contrast, there are three type of donors which interacts with O2. First, the distance H3...O2 of the alkenyl group is 2.51 Å and their angle C3–H3...O2 is 158°. Second, the distance of the interaction C29–H29...O2 of one of the phenyl moieties is 2.77 Å and their relevant angle is 162°. Third, the distance H8A...O2 of a methylene group of one of the cyclohexyl moieties is 2.80 Å and the angle C8–H8A...O2 is 163°. There is also one weak intramolecular hydrogen bond between C13–H13B of one of the cyclohexyl moieties as donor and the oxygen O1 of one of the benzoate functions as acceptor evident. The C13–H13B...O1 angle of this interaction is 101° and the relevant donor-acceptor distance is 2.59 Å.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.6 × 0.7 × 0.9 mm
Wavelength:	Mo <i>K</i> _α radiation (0.71073 Å)
μ:	0.77 cm ⁻¹
Diffractometer, scan mode:	Nicolet P3, Wyckoff scan
2θ _{max} :	56°
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} :	6983, 6437
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 5574
<i>N</i> (<i>param</i>) _{refined} :	317
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL-Plus [4]

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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)	2a	-0.0389	0.7265	0.7660	0.054
H(2)	2a	0.3171	0.8191	0.7205	0.062
H(3)	2a	0.4097	0.7026	0.6522	0.062
H(4A)	2a	0.1194	0.5989	0.6033	0.058
H(4B)	2a	0.0710	0.6089	0.7094	0.058
H(5)	2a	0.4780	0.5496	0.6871	0.050
H(6)	2a	0.2571	0.4457	0.5987	0.047
H(7A)	2a	0.4652	0.3725	0.7777	0.060
H(7B)	2a	0.5784	0.3953	0.6899	0.060
H(8A)	2a	0.3865	0.2869	0.5930	0.065
H(8B)	2a	0.4962	0.2433	0.6920	0.065
H(9A)	2a	0.1345	0.2003	0.6476	0.071
H(9B)	2a	0.1803	0.2458	0.7508	0.071
H(10A)	2a	-0.1322	0.3038	0.6613	0.069
H(10B)	2a	-0.0164	0.3284	0.5748	0.069
H(11A)	2a	-0.0498	0.4553	0.6670	0.055
H(11B)	2a	0.0693	0.4089	0.7627	0.055
H(12)	2a	0.1719	0.8629	0.8866	0.052
H(13A)	2a	-0.1467	0.7458	0.9261	0.066

Table 2. Continued.

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(13B)	2a	-0.1859	0.8453	0.8994	0.066
H(14A)	2a	0.0319	0.8878	1.0463	0.077
H(14B)	2a	-0.1450	0.8245	1.0707	0.077
H(15A)	2a	0.1941	0.7860	1.1608	0.073
H(15B)	2a	0.0928	0.7077	1.0945	0.073
H(16A)	2a	0.4445	0.7227	1.0765	0.070
H(16B)	2a	0.4090	0.8223	1.0495	0.070
H(17A)	2a	0.2252	0.6811	0.9296	0.062
H(17B)	2a	0.4052	0.7428	0.9053	0.062
H(20)	2a	0.2774	0.5038	0.9716	0.064
H(21)	2a	0.3054	0.4986	1.1404	0.077
H(22)	2a	0.6222	0.5345	1.2421	0.079
H(23)	2a	0.9114	0.5752	1.1768	0.075
H(24)	2a	0.8899	0.5780	1.0088	0.063
H(27)	2a	-0.6044	0.8685	0.5472	0.065
H(28)	2a	-0.7772	0.9964	0.4892	0.078
H(29)	2a	-0.6029	1.1297	0.5104	0.080
H(30)	2a	-0.2568	1.1354	0.5941	0.080
H(31)	2a	-0.0863	1.0093	0.6602	0.066

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

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> ₁₁	<i>U</i> ₂₂	<i>U</i> ₃₃	<i>U</i> ₁₂	<i>U</i> ₁₃	<i>U</i> ₂₃
C(1)	2a	0.0487(3)	0.7798(1)	0.7739(1)	0.057(1)	0.0358(8)	0.0411(8)	0.0025(7)	0.0053(7)	-0.0005(7)
O(1)	2a	-0.0769(2)	0.85590(8)	0.7292(1)	0.0713(9)	0.0402(7)	0.0428(6)	0.0063(6)	-0.0056(6)	-0.0016(5)
O(2)	2a	-0.2621(3)	0.77100(9)	0.6106(1)	0.0783(9)	0.0475(7)	0.0543(8)	-0.0085(7)	-0.0017(7)	-0.0036(6)
C(2)	2a	0.2327(3)	0.7697(1)	0.7228(1)	0.067(1)	0.0429(9)	0.048(1)	-0.0069(8)	0.0149(9)	0.0030(8)
O(3)	2a	0.3764(2)	0.53553(8)	0.81438(8)	0.0474(6)	0.0582(7)	0.0307(5)	-0.0031(5)	0.0067(5)	-0.0069(5)
C(3)	2a	0.2889(3)	0.6976(1)	0.6802(1)	0.063(1)	0.051(1)	0.0447(9)	-0.0021(9)	0.0182(8)	0.0025(8)
O(4)	2a	0.7263(2)	0.5637(2)	0.8294(1)	0.0500(8)	0.134(2)	0.0500(8)	-0.0208(9)	0.0099(6)	-0.0045(9)
C(4)	2a	0.1835(3)	0.6094(1)	0.6713(1)	0.056(1)	0.047(1)	0.0408(9)	0.0026(8)	0.0086(8)	-0.0044(7)
C(5)	2a	0.3426(3)	0.5359(1)	0.7071(1)	0.0454(8)	0.0492(9)	0.0305(7)	0.0009(7)	0.0094(6)	-0.0037(6)
C(6)	2a	0.2727(3)	0.4441(1)	0.6702(1)	0.0424(8)	0.0456(9)	0.0299(7)	0.0027(7)	0.0071(6)	-0.0030(6)
C(7)	2a	0.4452(3)	0.3762(1)	0.7069(2)	0.0416(9)	0.054(1)	0.054(1)	0.0077(8)	0.0047(8)	-0.0052(8)
C(8)	2a	0.3884(3)	0.2852(1)	0.6630(2)	0.054(1)	0.051(1)	0.058(1)	0.0111(8)	0.0100(9)	-0.0061(8)
C(9)	2a	0.1724(3)	0.2555(1)	0.6812(2)	0.066(1)	0.047(1)	0.066(1)	-0.0004(9)	0.015(1)	-0.0024(9)
C(10)	2a	0.0019(3)	0.3232(1)	0.6455(2)	0.048(1)	0.056(1)	0.068(1)	-0.0051(8)	0.0091(9)	-0.0080(9)
C(11)	2a	0.0603(3)	0.4130(1)	0.6923(1)	0.0408(8)	0.051(1)	0.0457(9)	0.0038(7)	0.0101(7)	-0.0024(7)
C(12)	2a	0.1115(3)	0.8035(1)	0.8819(1)	0.0508(9)	0.0369(8)	0.0410(8)	0.0012(7)	0.0051(7)	-0.0002(6)
C(13)	2a	-0.0817(3)	0.8036(2)	0.9321(2)	0.050(1)	0.069(1)	0.047(1)	0.0097(9)	0.0056(8)	-0.0028(9)
C(14)	2a	-0.0202(4)	0.8279(2)	1.0404(2)	0.063(1)	0.082(2)	0.050(1)	0.003(1)	0.0151(9)	-0.012(1)
C(15)	2a	0.1505(4)	0.7666(2)	1.0935(1)	0.070(1)	0.070(1)	0.0405(9)	-0.013(1)	0.0054(9)	-0.0018(9)
C(16)	2a	0.3419(3)	0.7650(2)	1.0437(2)	0.057(1)	0.066(1)	0.046(1)	0.003(1)	-0.0034(8)	-0.0008(9)
C(17)	2a	0.2801(3)	0.7406(1)	0.9355(1)	0.053(1)	0.054(1)	0.045(1)	0.0109(8)	0.0038(8)	-0.0005(8)
C(18)	2a	0.5733(3)	0.5484(1)	0.8648(1)	0.0450(9)	0.052(1)	0.0406(8)	-0.0022(8)	0.0050(7)	-0.0057(7)
C(19)	2a	0.5800(3)	0.5423(1)	0.9729(1)	0.0502(9)	0.0374(8)	0.0399(8)	-0.0001(7)	0.0024(7)	-0.0040(6)
C(20)	2a	0.4052(3)	0.5180(1)	1.0124(1)	0.059(1)	0.055(1)	0.0439(9)	-0.0066(9)	0.0071(8)	-0.0031(8)
C(21)	2a	0.4223(4)	0.5151(2)	1.1135(2)	0.083(2)	0.065(1)	0.047(1)	-0.009(1)	0.020(1)	0.0010(9)
C(22)	2a	0.6121(4)	0.5365(2)	1.1744(2)	0.095(2)	0.062(1)	0.0381(9)	0.002(1)	0.005(1)	0.0034(9)
C(23)	2a	0.7845(4)	0.5604(2)	1.1356(2)	0.074(1)	0.061(1)	0.045(1)	0.002(1)	-0.0102(9)	-0.0015(9)
C(24)	2a	0.7712(3)	0.5627(1)	1.0350(1)	0.053(1)	0.050(1)	0.0492(9)	-0.0008(8)	-0.0018(8)	-0.0022(8)
C(25)	2a	-0.2224(3)	0.8419(1)	0.6480(1)	0.054(1)	0.0473(9)	0.0343(8)	-0.0044(7)	0.0083(7)	-0.0004(7)
C(26)	2a	-0.3282(3)	0.9254(1)	0.6099(1)	0.057(1)	0.051(1)	0.0287(7)	0.0008(8)	0.0091(7)	0.0005(7)
C(27)	2a	-0.5344(3)	0.9222(2)	0.5581(1)	0.060(1)	0.063(1)	0.0395(9)	-0.0031(9)	0.0059(8)	-0.0001(8)
C(28)	2a	-0.6369(4)	0.9987(2)	0.5224(2)	0.067(1)	0.081(2)	0.044(1)	0.014(1)	-0.0008(9)	-0.000(1)
C(29)	2a	-0.5337(4)	1.0786(2)	0.5355(2)	0.089(2)	0.064(1)	0.046(1)	0.025(1)	0.009(1)	0.007(1)
C(30)	2a	-0.3277(4)	1.0819(2)	0.5859(2)	0.091(2)	0.048(1)	0.058(1)	0.002(1)	0.008(1)	-0.0010(9)
C(31)	2a	-0.2244(4)	1.0062(1)	0.6246(2)	0.064(1)	0.051(1)	0.047(1)	-0.0021(9)	0.0023(9)	-0.0034(8)

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