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Crystal structure of 3,8-di-*tert*-butyl-4-methoxy-1-methyl-9,10-diphenylmethylidenedioxy)dibenzo[*b*,*d*]pyran-6-one, $C_6H[O_2C(C_6H_5)_2](C_4H_9)COO(C_6H)(CH_3)(OCH_3)(C_4H_9)$

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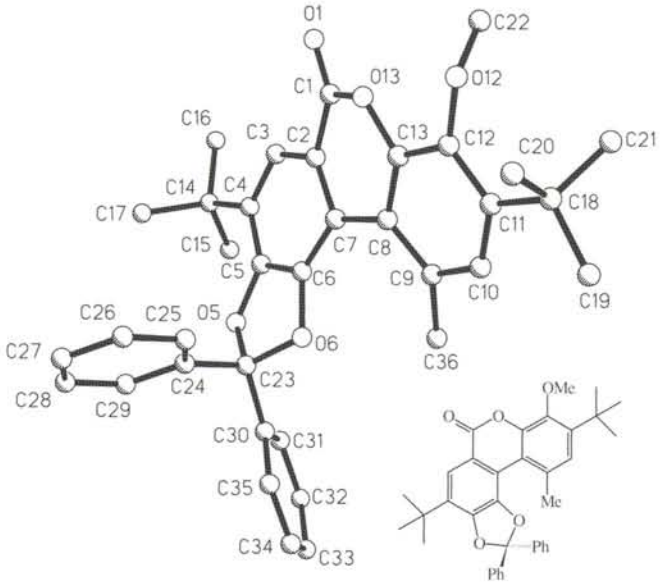

Abstract

$C_{36}H_{36}O_5$, triclinic, $P\bar{1}$ (No. 2), a = 10.7518(9) Å, b = 10.9501(9) Å, c = 14.372(1) Å, α = 72.022(6)°, β = 73.190(6)°, γ = 73.099(7)°, V = 1502.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.061, $wR_{ref}(F^2)$ = 0.182, T = 293 K.

Source of material

The title compound, a configurationally unstable intermediate in the atropo-enantioselective biaryl synthesis of simplified model analogs [1] of nerve-growth stimulating mastigophorenes [2], was prepared by intramolecular biaryl coupling of the corresponding bromo ester [3]. Crystals were grown from petroleum ether, b.range '303-323',/ dichloromethane.

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Table 1. Data collection and handling.

Crystal:	colourless lath, size 0.15 × 0.5 × 1.4 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.80 cm^{-1}
Diffractometer, scan mode:	Bruker AXS, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7911, 6812
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4847
$N(param)_{refined}$:	371
Program:	SHELXL-97 [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.5146	0.0174	0.7413	0.08
H(10)	2*i*	–0.0946	0.0906	1.1750	0.08
H(15A)	2*i*	0.3158	0.3717	0.5381	0.08
H(15B)	2*i*	0.3272	0.2315	0.5252	0.08
H(15C)	2*i*	0.4241	0.3196	0.4511	0.08
H(16A)	2*i*	0.6338	0.0741	0.5974	0.08
H(16B)	2*i*	0.6173	0.1402	0.4869	0.08
H(16C)	2*i*	0.5206	0.0515	0.5604	0.08
H(17A)	2*i*	0.6023	0.3593	0.5106	0.08
H(17B)	2*i*	0.6181	0.2954	0.6215	0.08
H(17C)	2*i*	0.4960	0.4108	0.5987	0.08
H(19A)	2*i*	–0.1387	0.0703	1.3402	0.08
H(19B)	2*i*	–0.1702	–0.0563	1.4221	0.08
H(19C)	2*i*	–0.1904	–0.0319	1.3136	0.08
H(20A)	2*i*	0.1782	–0.1264	1.3695	0.08
H(20B)	2*i*	0.0507	–0.1186	1.4565	0.08
H(20C)	2*i*	0.0832	0.0104	1.3785	0.08
H(21A)	2*i*	0.0944	–0.2895	1.3214	0.08
H(21B)	2*i*	–0.0493	–0.2481	1.3008	0.08
H(21C)	2*i*	–0.0303	–0.2743	1.4099	0.08
H(22A)	2*i*	0.3836	–0.3763	1.2288	0.08
H(22B)	2*i*	0.3826	–0.2930	1.1184	0.08
H(22C)	2*i*	0.2563	–0.3450	1.1858	0.08
H(25)	2*i*	0.2434	0.4127	0.9539	0.08
H(26)	2*i*	0.3789	0.5062	0.9965	0.08
H(27)	2*i*	0.4691	0.6742	0.8826	0.08
H(28)	2*i*	0.4185	0.7546	0.7270	0.08
H(29)	2*i*	0.2813	0.6639	0.6835	0.08
H(31)	2*i*	0.0511	0.5199	0.6255	0.08
H(32)	2*i*	–0.1522	0.6543	0.5969	0.08
H(33)	2*i*	–0.2869	0.7740	0.7096	0.08
H(34)	2*i*	–0.2178	0.7647	0.8490	0.08
H(35)	2*i*	–0.0171	0.6315	0.8782	0.08
H(36A)	2*i*	–0.0106	0.2425	0.9214	0.08
H(36B)	2*i*	–0.1019	0.2818	1.0193	0.08
H(36C)	2*i*	–0.1196	0.1639	0.9879	0.08

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	2*i*	0.5428(1)	–0.1442(1)	0.9060(1)	0.0424(7)	0.0561(8)	0.076(1)	0.0105(6)	–0.0029(7)	–0.0232(7)
C(1)	2*i*	0.4388(2)	–0.0701(2)	0.9304(2)	0.0350(9)	0.047(1)	0.061(1)	–0.0008(7)	–0.0065(8)	–0.0228(9)
C(2)	2*i*	0.3723(2)	0.0442(2)	0.8637(1)	0.0360(9)	0.0440(9)	0.052(1)	–0.0007(7)	–0.0073(7)	–0.0220(8)
C(3)	2*i*	0.4398(2)	0.0777(2)	0.7643(1)	0.0365(9)	0.054(1)	0.055(1)	0.0015(8)	–0.0044(8)	–0.0300(9)
C(4)	2*i*	0.4016(2)	0.1964(2)	0.6977(1)	0.0378(9)	0.056(1)	0.0458(9)	–0.0036(8)	–0.0056(7)	–0.0256(8)
C(5)	2*i*	0.2949(2)	0.2823(2)	0.7424(1)	0.0409(9)	0.047(1)	0.0446(9)	–0.0020(8)	–0.0091(7)	–0.0183(8)
O(5)	2*i*	0.2430(1)	0.4113(1)	0.69887(9)	0.0489(7)	0.0500(7)	0.0414(7)	0.0008(6)	–0.0043(5)	–0.0169(6)
C(6)	2*i*	0.2233(2)	0.2484(2)	0.8405(1)	0.0347(8)	0.0444(9)	0.0458(9)	0.0005(7)	–0.0057(7)	–0.0216(8)
O(6)	2*i*	0.1263(1)	0.3567(1)	0.86122(9)	0.0425(7)	0.0416(7)	0.0454(7)	0.0018(5)	–0.0023(5)	–0.0153(5)
C(7)	2*i*	0.2524(2)	0.1260(2)	0.9044(1)	0.0334(8)	0.0436(9)	0.0466(9)	–0.0021(7)	–0.0083(7)	–0.0202(7)
C(8)	2*i*	0.1800(2)	0.0752(2)	1.0067(1)	0.0355(8)	0.0405(9)	0.0467(9)	–0.0029(7)	–0.0097(7)	–0.0179(7)
C(9)	2*i*	0.0433(2)	0.1163(2)	1.0482(1)	0.0342(8)	0.0442(9)	0.050(1)	–0.0004(7)	–0.0095(7)	–0.0201(8)
C(10)	2*i*	–0.0054(2)	0.0596(2)	1.1478(1)	0.0376(9)	0.052(1)	0.049(1)	–0.0019(8)	–0.0056(8)	–0.0206(8)
C(11)	2*i*	0.0686(2)	–0.0404(2)	1.2105(1)	0.045(1)	0.052(1)	0.046(1)	–0.0056(8)	–0.0086(8)	–0.0194(8)
C(12)	2*i*	0.2004(2)	–0.0904(2)	1.1662(1)	0.046(1)	0.046(1)	0.048(1)	0.0004(8)	–0.0166(8)	–0.0160(8)
O(12)	2*i*	0.2802(2)	–0.1933(1)	1.2201(1)	0.0575(8)	0.0604(9)	0.0526(8)	0.0100(7)	–0.0218(7)	–0.0163(6)
C(13)	2*i*	0.2525(2)	–0.0315(2)	1.0670(1)	0.0336(8)	0.0456(9)	0.050(1)	0.0013(7)	–0.0104(7)	–0.0203(8)
O(13)	2*i*	0.3836(1)	–0.0897(1)	1.0309(1)	0.0364(7)	0.0541(8)	0.0558(8)	0.0063(6)	–0.0103(6)	–0.0165(6)
C(14)	2*i*	0.4755(2)	0.2351(2)	0.5884(1)	0.043(1)	0.073(1)	0.044(1)	–0.0044(9)	–0.0033(8)	–0.0261(9)
C(15)	2*i*	0.3764(2)	0.2951(3)	0.5192(2)	0.058(1)	0.128(2)	0.049(1)	–0.006(1)	–0.010(1)	–0.035(1)
C(16)	2*i*	0.5707(3)	0.1139(3)	0.5552(2)	0.066(1)	0.083(2)	0.060(1)	–0.008(1)	0.007(1)	–0.039(1)
C(17)	2*i*	0.5553(2)	0.3343(2)	0.5790(2)	0.060(1)	0.067(1)	0.053(1)	–0.012(1)	–0.008(1)	–0.011(1)
C(18)	2*i*	0.0067(2)	–0.0953(2)	1.3212(2)	0.056(1)	0.067(1)	0.045(1)	–0.004(1)	–0.0076(9)	–0.0166(9)
C(19)	2*i*	–0.1364(3)	–0.0214(3)	1.3521(2)	0.066(2)	0.100(2)	0.058(1)	0.001(1)	0.006(1)	–0.011(1)
C(20)	2*i*	0.0872(3)	–0.0812(3)	1.3876(2)	0.087(2)	0.108(2)	0.056(1)	–0.007(2)	–0.017(1)	–0.038(1)
C(21)	2*i*	0.0052(3)	–0.2403(3)	1.3401(2)	0.086(2)	0.078(2)	0.063(1)	–0.021(1)	–0.004(1)	–0.012(1)
C(22)	2*i*	0.3298(3)	–0.3117(2)	1.1854(2)	0.095(2)	0.058(1)	0.067(2)	0.018(1)	–0.021(1)	–0.016(1)
C(23)	2*i*	0.1627(2)	0.4676(2)	0.7822(1)	0.0427(9)	0.0440(9)	0.0400(9)	–0.0022(7)	–0.0040(7)	–0.0142(7)
C(24)	2*i*	0.2493(2)	0.5280(2)	0.8138(2)	0.0389(9)	0.051(1)	0.054(1)	0.0016(8)	–0.0070(8)	–0.0222(8)
C(25)	2*i*	0.2786(2)	0.4816(2)	0.9077(2)	0.062(1)	0.070(1)	0.064(1)	–0.004(1)	–0.021(1)	–0.024(1)
C(26)	2*i*	0.3600(3)	0.5374(3)	0.9330(2)	0.073(2)	0.097(2)	0.095(2)	–0.003(2)	–0.039(2)	–0.043(2)
C(27)	2*i*	0.4131(3)	0.6380(3)	0.8656(3)	0.060(2)	0.094(2)	0.138(3)	–0.008(1)	–0.027(2)	–0.068(2)
C(28)	2*i*	0.3833(3)	0.6854(3)	0.7727(3)	0.075(2)	0.079(2)	0.118(2)	–0.030(2)	–0.011(2)	–0.032(2)
C(29)	2*i*	0.3014(3)	0.6310(3)	0.7466(2)	0.071(2)	0.068(1)	0.072(2)	–0.023(1)	–0.010(1)	–0.016(1)
C(30)	2*i*	0.0366(2)	0.5607(2)	0.7557(1)	0.0422(9)	0.0430(9)	0.048(1)	–0.0041(8)	–0.0073(8)	–0.0110(8)
C(31)	2*i*	–0.0030(3)	0.5684(3)	0.6712(2)	0.071(2)	0.086(2)	0.062(1)	0.013(1)	–0.026(1)	–0.028(1)
C(32)	2*i*	–0.1249(3)	0.6491(3)	0.6541(2)	0.081(2)	0.116(2)	0.080(2)	0.013(2)	–0.046(2)	–0.022(2)
C(33)	2*i*	–0.2051(3)	0.7211(3)	0.7209(2)	0.051(1)	0.077(2)	0.095(2)	0.011(1)	–0.026(1)	–0.011(1)
C(34)	2*i*	–0.1643(2)	0.7150(3)	0.8040(2)	0.053(1)	0.085(2)	0.093(2)	0.016(1)	–0.015(1)	–0.040(2)
C(35)	2*i*	–0.0442(2)	0.6353(3)	0.8214(2)	0.049(1)	0.082(2)	0.071(1)	0.012(1)	–0.017(1)	–0.038(1)
C(36)	2*i*	–0.0563(2)	0.2096(2)	0.9888(2)	0.0333(9)	0.055(1)	0.056(1)	–0.0003(8)	–0.0104(8)	–0.0158(9)

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