Crystal structure of 1,4-dimethyl-6*H*-benzo[*b*]naphtho[1,2-*d*]pyran-6-one, $C_{10}H_6(COO)C_6H_2(CH_3)_2$

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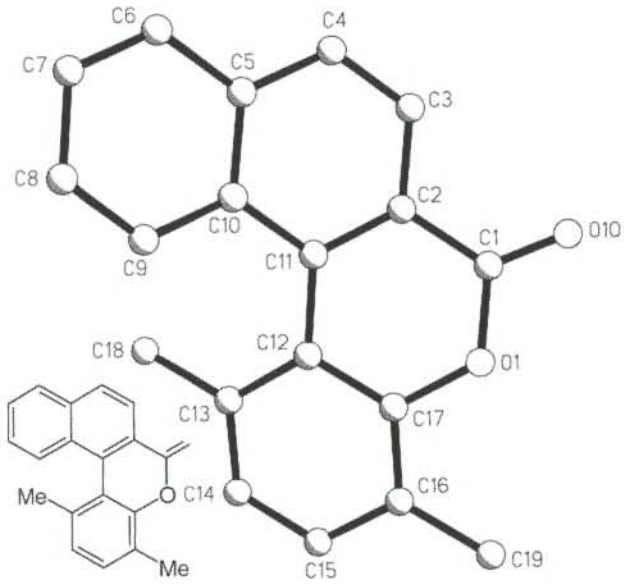

Abstract

$C_{19}H_{14}O_2$, orthorhombic, *Pbca* (No. 61), $a = 13.526(1)$ Å, $b = 9.523(1)$ Å, $c = 21.421(2)$ Å, $V = 2759.2$ Å^3, $Z = 8$, $R_{gt}(F) = 0.059$, $wR(F) = 0.056$, $T = 293$ K.

Source of material

The configurationally unstable [1] lactone-bridged title compound was prepared from the corresponding bromonaphthoic aryl ester in 72% yield, by intramolecular biaryl coupling [$Pd(OAc)_2$, PPh_3, NaOAc, DMA, 403 K , 17 h]; it constitutes a precursor for the synthesis of a fluorenone [2].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.3 × 0.45 × 0.65 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.80 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4533, 3173
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2277
$N(param)_{refined}$:	190
Program:	SHELXTL-plus [3]

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

Atom	Site	x	y	z	U_{iso}
H(3)	8*c*	-0.0905(2)	-0.1698(3)	0.6515(1)	0.08
H(4)	8*c*	-0.0349(2)	-0.2681(3)	0.7440(1)	0.08
H(6)	8*c*	0.0704(2)	-0.2448(3)	0.8388(1)	0.08
H(7)	8*c*	0.1905(2)	-0.1265(3)	0.8935(1)	0.08
H(8)	8*c*	0.2713(2)	0.0675(3)	0.8490(1)	0.08
H(9)	8*c*	0.2463(2)	0.1226(3)	0.7448(1)	0.08
H(14)	8*c*	0.4059(2)	0.1868(3)	0.5836(1)	0.08
H(15)	8*c*	0.3202(3)	0.3316(3)	0.5155(1)	0.08
H(18A)	8*c*	0.3082(2)	-0.0658(3)	0.6861(1)	0.08
H(18B)	8*c*	0.3960(2)	0.0417(3)	0.6854(1)	0.08
H(18C)	8*c*	0.3857(2)	-0.0692(3)	0.6317(1)	0.08
H(19A)	8*c*	0.1737(3)	0.3989(3)	0.4643(1)	0.08
H(19B)	8*c*	0.0810(3)	0.4000(3)	0.5082(1)	0.08
H(19C)	8*c*	0.0942(3)	0.2797(3)	0.4589(1)	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)	8*c*	0.0353(1)	0.1460(2)	0.56213(6)	0.070(1)	0.063(1)	0.0467(8)	0.0106(9)	-0.0046(8)	0.0000(8)
C(1)	8*c*	-0.0148(2)	0.0334(3)	0.5847(1)	0.053(1)	0.063(2)	0.046(1)	0.010(1)	0.001(1)	-0.011(1)
C(2)	8*c*	0.0276(2)	-0.0366(2)	0.6392(1)	0.045(1)	0.049(1)	0.048(1)	0.006(1)	0.004(1)	-0.008(1)
C(3)	8*c*	-0.0299(2)	-0.1378(3)	0.6699(1)	0.044(1)	0.062(1)	0.068(1)	-0.006(1)	0.006(1)	-0.008(1)
C(4)	8*c*	0.0001(2)	-0.1908(3)	0.7255(1)	0.057(1)	0.056(1)	0.076(2)	-0.008(1)	0.015(1)	0.006(1)
C(5)	8*c*	0.0825(2)	-0.1325(2)	0.7565(1)	0.051(1)	0.050(1)	0.057(1)	0.004(1)	0.010(1)	0.006(1)
C(6)	8*c*	0.1054(2)	-0.1690(3)	0.8192(1)	0.075(2)	0.070(2)	0.064(1)	0.006(2)	0.012(1)	0.020(1)
C(7)	8*c*	0.1752(2)	-0.0986(3)	0.8515(1)	0.085(2)	0.086(2)	0.049(1)	0.022(2)	0.001(1)	0.014(1)
C(8)	8*c*	0.2256(2)	0.0136(3)	0.8245(1)	0.064(2)	0.077(2)	0.052(1)	0.013(1)	-0.007(1)	-0.003(1)
C(9)	8*c*	0.2098(2)	0.0470(3)	0.7633(1)	0.051(1)	0.055(1)	0.052(1)	0.006(1)	-0.002(1)	-0.000(1)

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Table 3. Continued.

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(10)	8*c*	0.1400(2)	–0.0278(2)	0.7267(1)	0.043(1)	0.042(1)	0.045(1)	0.008(1)	0.003(1)	–0.000(1)
O(10)	8*c*	–0.0944(1)	0.0088(2)	0.56224(8)	0.056(1)	0.106(1)	0.062(1)	0.007(1)	–0.0118(9)	–0.007(1)
C(11)	8*c*	0.1182(2)	0.0071(2)	0.66287(9)	0.045(1)	0.036(1)	0.044(1)	0.004(1)	0.0039(9)	–0.0033(9)
C(12)	8*c*	0.1824(2)	0.0897(2)	0.62229(9)	0.056(1)	0.040(1)	0.043(1)	–0.002(1)	0.005(1)	–0.007(1)
C(13)	8*c*	0.2878(2)	0.0913(3)	0.6233(1)	0.054(1)	0.054(1)	0.051(1)	–0.011(1)	0.008(1)	–0.012(1)
C(14)	8*c*	0.3351(2)	0.1817(3)	0.5826(1)	0.076(2)	0.076(2)	0.065(2)	–0.031(2)	0.016(1)	–0.016(2)
C(15)	8*c*	0.2841(3)	0.2656(3)	0.5407(1)	0.120(3)	0.066(2)	0.051(1)	–0.038(2)	0.020(2)	–0.006(1)
C(16)	8*c*	0.1834(2)	0.2562(3)	0.5343(1)	0.107(2)	0.053(1)	0.040(1)	–0.014(2)	0.007(1)	–0.003(1)
C(17)	8*c*	0.1357(2)	0.1642(2)	0.57366(9)	0.072(2)	0.047(1)	0.040(1)	–0.001(1)	0.004(1)	–0.008(1)
C(18)	8*c*	0.3500(2)	–0.0095(3)	0.6599(1)	0.047(1)	0.077(2)	0.073(2)	–0.003(1)	0.006(1)	–0.015(1)
C(19)	8*c*	0.1282(3)	0.3413(3)	0.4872(1)	0.174(3)	0.069(2)	0.052(1)	–0.013(2)	–0.006(2)	0.009(1)

References

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