Crystal structure of (2,5-dimethyl)phenyl 1-bromonaphthyl-2-carboxylate, $C_{10}H_6Br(COO)C_6H_3(CH_3)_2$

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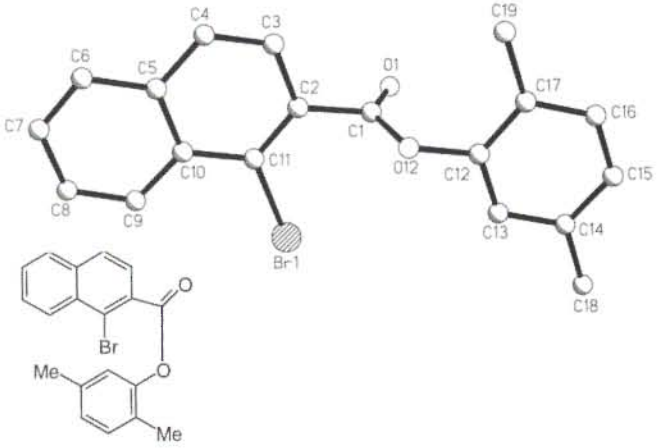

Abstract

$C_{19}H_{15}BrO_2$, monoclinic, $P12_1/c1$ (No. 14), $a = 6.795(1)$ Å, $b = 7.651(1)$ Å, $c = 30.024(3)$ Å, $\beta = 95.60(1)°$, $V = 1553.5$ Å^3, $Z = 4$, $R_{gt}(F) = 0.064$, $wR(F) = 0.061$, $T = 293$ K.

Source of material

The title compound, a precursor to biaryl lactones [1] and to fluorenones, developped recently in a new pathway [2], was obtained in 96% yield by esterification of 1-bromo-2-naphthoic acid chloride with 2,5-dimethylphenol (NaH, THF, room temperature, 64 h).

Table 1. Data collection and handling.

Crystal:	colourless plate, size 0.6 × 1.4 × 0.06 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	26.50 cm^{-1}
Diffractometer, scan mode:	Siemens P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3802, 3292
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\ \sigma(F_{obs})$, 2472
$N(param)_{refined}$:	199
Program:	SHELXTL-plus [3]

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

Atom	Site	x	y	z	U_{iso}
H(3)	4e	0.9306(8)	0.3903(8)	0.0536(2)	0.08
H(4)	4e	0.8425(8)	0.3768(8)	-0.0222(2)	0.08
H(6)	4e	0.605(1)	0.2936(8)	-0.0866(2)	0.08
H(7)	4e	0.303(1)	0.1707(9)	-0.1133(2)	0.08
H(8)	4e	0.0727(9)	0.0877(9)	-0.0642(2)	0.08
H(9)	4e	0.1431(8)	0.1200(8)	0.0118(2)	0.08
H(13)	4e	0.4701(8)	0.3675(8)	0.2072(2)	0.08
H(15)	4e	0.845(1)	0.5643(9)	0.3080(2)	0.08
H(16)	4e	1.0668(9)	0.6376(9)	0.2576(2)	0.08
H(18A)	4e	0.372(1)	0.362(1)	0.2796(2)	0.08
H(18B)	4e	0.449(1)	0.518(1)	0.3101(2)	0.08
H(18C)	4e	0.547(1)	0.334(1)	0.3168(2)	0.08
H(19A)	4e	0.999(1)	0.558(1)	0.1391(2)	0.08
H(19B)	4e	1.175(1)	0.532(1)	0.1764(2)	0.08
H(19C)	4e	1.076(1)	0.716(1)	0.1690(2)	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}
Br(1)	4e	0.29656(9)	0.1861(1)	0.09802(2)	0.0507(3)	0.0941(5)	0.0463(3)	-0.0033(4)	0.0087(3)	0.0022(4)
O(1)	4e	0.8420(7)	0.1959(6)	0.1397(2)	0.080(3)	0.071(3)	0.045(2)	0.026(3)	-0.017(2)	-0.005(2)
C(1)	4e	0.7352(8)	0.3021(8)	0.1226(2)	0.041(3)	0.051(4)	0.037(3)	-0.002(3)	-0.005(2)	-0.006(3)
C(2)	4e	0.6652(8)	0.2984(7)	0.0731(2)	0.043(3)	0.039(3)	0.037(3)	0.001(2)	-0.003(2)	0.001(3)
C(3)	4e	0.8013(8)	0.3497(8)	0.0425(2)	0.038(3)	0.053(4)	0.054(4)	-0.003(3)	0.002(2)	0.003(3)
C(4)	4e	0.7501(8)	0.3383(8)	-0.0020(2)	0.047(3)	0.057(4)	0.050(3)	0.000(3)	0.015(3)	0.009(3)
C(5)	4e	0.5633(8)	0.2749(7)	-0.0194(2)	0.045(3)	0.048(4)	0.039(3)	0.008(2)	0.007(2)	0.004(3)
C(6)	4e	0.513(1)	0.2559(8)	-0.0662(2)	0.067(4)	0.058(4)	0.035(3)	0.012(3)	0.012(3)	0.003(3)
C(7)	4e	0.335(1)	0.1856(9)	-0.0817(2)	0.076(4)	0.062(4)	0.035(3)	0.014(4)	-0.003(3)	-0.006(3)
C(8)	4e	0.1975(9)	0.1366(9)	-0.0527(2)	0.052(3)	0.066(4)	0.050(4)	-0.004(3)	-0.011(3)	-0.006(3)
C(9)	4e	0.2395(8)	0.1547(8)	-0.0078(2)	0.046(3)	0.058(4)	0.045(3)	-0.005(3)	0.001(3)	0.002(3)
C(10)	4e	0.4236(7)	0.2235(7)	0.0106(2)	0.038(3)	0.044(3)	0.035(3)	0.005(2)	0.001(2)	0.001(3)
C(11)	4e	0.4811(8)	0.2444(8)	0.0573(2)	0.036(3)	0.048(3)	0.039(3)	0.003(2)	0.009(2)	0.003(3)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(12)	4*e*	0.6681(6)	0.4437(6)	0.1422(1)	0.061(2)	0.056(3)	0.039(2)	0.013(2)	–0.012(2)	–0.003(2)
C(12)	4*e*	0.7266(8)	0.4676(8)	0.1883(2)	0.053(3)	0.046(3)	0.040(3)	0.007(3)	–0.011(3)	–0.003(3)
C(13)	4*e*	0.5918(8)	0.4241(8)	0.2178(2)	0.045(3)	0.051(4)	0.054(4)	0.003(3)	–0.007(3)	–0.009(3)
C(14)	4*e*	0.6330(9)	0.4608(8)	0.2633(2)	0.057(3)	0.048(4)	0.051(4)	0.005(3)	–0.002(3)	–0.005(3)
C(15)	4*e*	0.812(1)	0.5397(9)	0.2768(2)	0.073(4)	0.056(4)	0.046(3)	–0.001(3)	–0.009(3)	–0.008(3)
C(16)	4*e*	0.9450(9)	0.5813(9)	0.2469(2)	0.051(3)	0.064(4)	0.063(4)	–0.002(3)	–0.015(3)	–0.013(4)
C(17)	4*e*	0.9066(9)	0.5464(9)	0.2008(2)	0.054(3)	0.050(4)	0.063(4)	0.009(3)	–0.004(3)	–0.007(3)
C(18)	4*e*	0.487(1)	0.414(1)	0.2952(2)	0.078(5)	0.076(5)	0.056(4)	–0.001(4)	0.005(3)	–0.012(4)
C(19)	4*e*	1.053(1)	0.592(1)	0.1685(2)	0.064(4)	0.082(5)	0.068(5)	–0.008(4)	0.004(4)	–0.005(4)

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