Crystal structure of 1-bromo-2-methyl-dinaphtho[2,1-*b*:1′,2′-*d*]pyran-4-one, $C_{21}H_{10}O_2Br(CH_3)$

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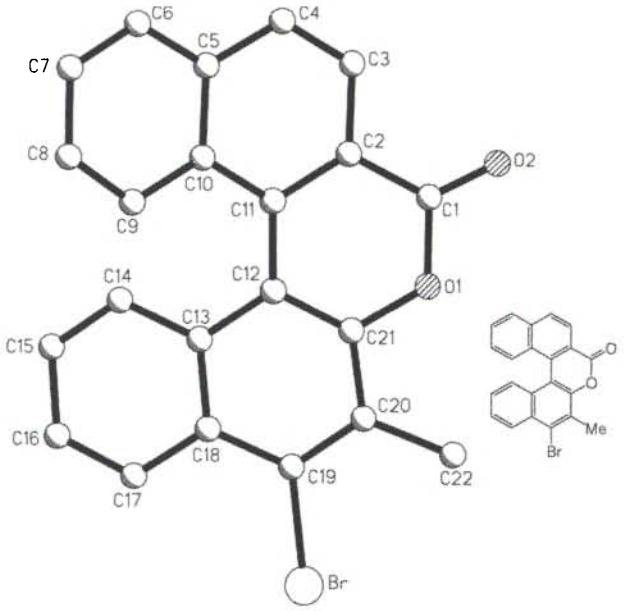

Abstract

$C_{22}H_{13}BrO_2$, orthorhombic, *Pbca* (No. 61), $a = 17.968(4)$ Å, $b = 8.171(1)$ Å, $c = 21.721(5)$ Å, $V = 3189.0$ Å^3, $Z = 8$, $R_{gt}(F) = 0.057$, $wR(F) = 0.046$, $T = 293$ K.

Source of material

2-Methyl-dinaphtho[2,1-*b*:1′2′-*d*]pyran-4-one, the synthetic precursor of the title compound was prepared *via* the lactone-methodology [1]. Subsequent bromination (Br_2, CH_2Cl_2, 273 K) gave the bromo compound in 42% yield.

Table 1. Data collection and handling.

Crystal:	pale yellow plate, size 0.4 × 0.45 × 0.1 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	25.90 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4814, 3415
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 2102
$N(param)_{refined}$:	226
Program:	SHELXTL-plus [2]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(3)	8*c*	0.7807(4)	–0.3791(7)	0.9738(3)	0.08
H(4)	8*c*	0.8975(4)	–0.3995(7)	1.0170(3)	0.08
H(6)	8*c*	1.0267(4)	–0.2940(7)	1.0248(2)	0.08
H(7)	8*c*	1.1203(3)	–0.1286(8)	0.9902(3)	0.08
H(8)	8*c*	1.1012(3)	0.0439(7)	0.9056(2)	0.08
H(9)	8*c*	0.9932(3)	0.0173(6)	0.8449(2)	0.08
H(14)	8*c*	0.9948(3)	–0.2417(6)	0.7929(2)	0.08
H(15)	8*c*	1.0765(3)	–0.2321(6)	0.7109(2)	0.08
H(16)	8*c*	1.0501(3)	–0.0696(6)	0.6251(2)	0.08
H(17)	8*c*	0.9328(3)	0.0488(6)	0.6145(2)	0.08
H(22A)	8*c*	0.6737(2)	0.0812(6)	0.6840(2)	0.08
H(22B)	8*c*	0.6629(2)	0.1029(6)	0.7552(2)	0.08
H(22C)	8*c*	0.6527(2)	–0.0699(6)	0.7249(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	8*c*	0.78727(3)	0.12629(7)	0.61498(3)	0.0590(3)	0.0728(4)	0.0455(3)	–0.0000(3)	–0.0083(3)	0.0199(4)
O(1)	8*c*	0.7264(2)	–0.1179(4)	0.8274(2)	0.049(2)	0.060(2)	0.040(2)	–0.002(2)	0.014(2)	0.008(2)
C(1)	8*c*	0.7364(3)	–0.2214(7)	0.8767(2)	0.063(3)	0.054(4)	0.042(4)	–0.009(3)	0.018(3)	–0.002(3)
O(2)	8*c*	0.6816(2)	–0.2759(5)	0.9005(2)	0.069(2)	0.097(3)	0.066(3)	–0.018(3)	0.022(2)	0.020(3)
C(2)	8*c*	0.8127(3)	–0.2425(6)	0.8984(2)	0.067(3)	0.043(3)	0.036(3)	0.001(3)	0.013(3)	0.005(3)
C(3)	8*c*	0.8225(4)	–0.3270(7)	0.9545(3)	0.086(4)	0.062(4)	0.037(3)	0.005(3)	0.018(3)	0.011(3)
C(4)	8*c*	0.8903(4)	–0.3344(7)	0.9807(3)	0.105(5)	0.064(4)	0.032(3)	0.023(4)	0.010(3)	0.015(3)
C(5)	8*c*	0.9516(3)	–0.2458(7)	0.9562(2)	0.084(4)	0.055(4)	0.021(3)	0.026(3)	0.002(3)	0.002(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}
C(6)	8*c*	1.0188(4)	–0.2320(7)	0.9879(2)	0.094(5)	0.089(5)	0.024(3)	0.045(4)	0.004(3)	0.003(3)
C(7)	8*c*	1.0739(3)	–0.1304(8)	0.9684(3)	0.070(4)	0.108(6)	0.032(3)	0.049(4)	–0.014(3)	–0.019(4)
C(8)	8*c*	1.0635(3)	–0.0336(7)	0.9170(2)	0.055(3)	0.076(4)	0.038(3)	0.012(3)	–0.004(3)	–0.017(3)
C(9)	8*c*	0.9993(3)	–0.0461(6)	0.8818(2)	0.057(3)	0.057(3)	0.033(3)	0.010(3)	–0.004(3)	–0.012(3)
C(10)	8*c*	0.9422(3)	–0.1559(6)	0.8998(2)	0.059(3)	0.045(3)	0.028(3)	0.014(3)	0.005(2)	–0.007(3)
C(11)	8*c*	0.8725(3)	–0.1713(6)	0.8667(2)	0.057(3)	0.039(3)	0.030(2)	0.008(2)	0.004(2)	0.001(2)
C(12)	8*c*	0.8578(2)	–0.1123(6)	0.8043(2)	0.045(3)	0.032(3)	0.029(2)	–0.001(2)	0.002(2)	0.003(3)
C(13)	8*c*	0.9112(2)	–0.1023(6)	0.7547(2)	0.043(2)	0.031(3)	0.028(2)	–0.006(2)	–0.002(2)	0.001(2)
C(14)	8*c*	0.9811(3)	–0.1807(6)	0.7569(2)	0.049(3)	0.032(3)	0.034(3)	–0.002(2)	–0.005(2)	–0.001(2)
C(15)	8*c*	1.0302(3)	–0.1738(6)	0.7086(2)	0.043(3)	0.043(3)	0.036(3)	–0.000(2)	–0.000(2)	–0.009(3)
C(16)	8*c*	1.0135(3)	–0.0824(6)	0.6569(2)	0.046(3)	0.055(3)	0.033(3)	–0.010(3)	0.005(2)	–0.008(3)
C(17)	8*c*	0.9447(3)	–0.0126(6)	0.6508(2)	0.054(3)	0.046(3)	0.025(2)	–0.010(3)	–0.003(2)	0.003(3)
C(18)	8*c*	0.8911(2)	–0.0256(5)	0.6984(2)	0.046(3)	0.031(3)	0.025(2)	–0.009(2)	0.001(2)	0.001(2)
C(19)	8*c*	0.8157(2)	0.0268(6)	0.6906(2)	0.046(3)	0.038(3)	0.030(2)	–0.001(2)	–0.006(2)	0.007(3)
C(20)	8*c*	0.7620(2)	–0.0044(6)	0.7340(2)	0.043(2)	0.034(3)	0.041(3)	0.003(2)	–0.003(2)	–0.000(3)
C(21)	8*c*	0.7852(3)	–0.0780(6)	0.7890(2)	0.049(3)	0.037(3)	0.037(3)	–0.004(2)	0.012(2)	–0.005(2)
C(22)	8*c*	0.6804(2)	0.0305(6)	0.7235(2)	0.045(3)	0.062(4)	0.052(3)	0.005(3)	–0.001(3)	0.006(3)

References

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2. Sheldrick, G. M.: Program Package SHELXTL-plus. Release 4.1. Siemens Analytical X-Ray Instruments Inc. Madison (WI53719) USA 1990.