Crystal structure of 6-bromochromone, $C_9H_5BrO_2$

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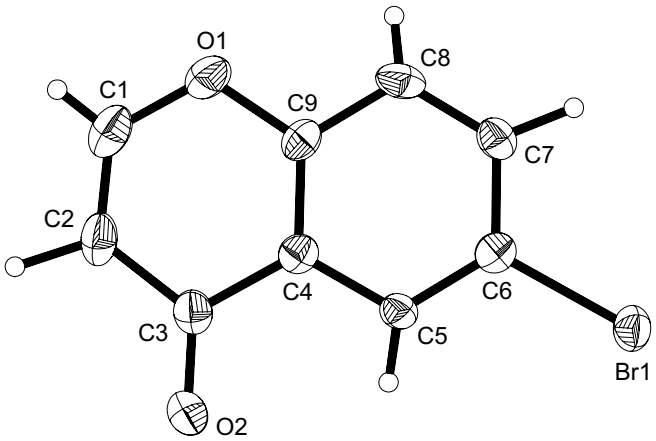

Abstract

$C_9H_5BrO_2$, monoclinic, $P12_11$ (no. 4), a = 3.922(1) Å, b = 5.723(2) Å, c = 17.208(5) Å, β = 95.447(6)°, V = 384.5 Å^3, Z = 2, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.106, T = 193 K.

Source of material

The title compound was purchased from Aldrich and crystals were grown from a solution of dichloromethane which was layered with hexanes. A mixture of 3-bromochromone and 6-bromochromone was dissolved in dichloromethane and layered with hexanes. The result was two crystal types, plates and needles.

Discussion

In the course of studying crystallization properties of substituted chromones [1], the title compound, 6-bromochromone, was crystallized in the same manner as 3-bromochromone. It was hoped that the two compounds would crystallize in the same crystal system or even in the same space group. This was not the case, 6-bromochromone crystallized as large plate-like crystals in monoclinic, where 3-bromochromone forms needles in orthorhombic crystal system. Co-crystallization resulted in two crystal types, plates and needles, both having cell dimensions consistent with the previously solved corresponding compounds.

Table 1. Data collection and handling.

Crystal:	colorless plate, size 0.02 × 0.12 × 0.12 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	52.91 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, ω/φ
$2\theta_{max}$:	55.78°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2588, 1670
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1592
$N(param)_{refined}$:	129
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL [4]

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

Atom	Site	x	y	z	U_{iso}
H(1)	2*a*	0.93(1)	0.01(2)	0.577(4)	0.03(2)
H(2)	2*a*	0.64(1)	0.30(1)	0.517(4)	0.03(2)
H(5)	2*a*	0.43(1)	0.69(1)	0.753(3)	0.03(2)
H(7)	2*a*	0.91(2)	0.31(1)	0.929(4)	0.04(2)
H(8)	2*a*	1.02(2)	0.04(1)	0.823(4)	0.03(2)

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)	2*a*	0.55602(9)	0.7295(2)	0.93012(2)	0.0318(2)	0.0329(2)	0.0222(2)	0.0007(2)	0.0042(1)	–0.0033(2)
O(1)	2*a*	0.948(1)	0.0659(7)	0.6832(2)	0.037(2)	0.023(2)	0.037(2)	0.003(1)	0.006(2)	–0.005(2)
O(2)	2*a*	0.402(1)	0.6499(7)	0.6089(2)	0.055(2)	0.033(2)	0.024(2)	0.006(2)	0.001(2)	0.002(1)
C(1)	2*a*	0.852(2)	0.108(1)	0.6062(4)	0.039(3)	0.029(3)	0.038(3)	–0.006(2)	0.015(2)	–0.009(2)
C(2)	2*a*	0.676(2)	0.2932(9)	0.5791(3)	0.044(3)	0.031(3)	0.024(2)	–0.010(2)	0.009(2)	–0.007(2)
C(3)	2*a*	0.569(1)	0.4745(8)	0.6309(3)	0.031(2)	0.025(2)	0.024(2)	–0.006(2)	0.004(2)	–0.000(2)
C(4)	2*a*	0.671(1)	0.4272(8)	0.7139(3)	0.024(2)	0.020(2)	0.026(2)	–0.004(2)	0.003(2)	–0.001(2)
C(5)	2*a*	0.588(1)	0.5824(8)	0.7724(3)	0.026(2)	0.021(2)	0.021(2)	0.002(2)	0.004(2)	0.004(2)
C(6)	2*a*	0.680(1)	0.5268(8)	0.8495(3)	0.022(2)	0.022(2)	0.028(2)	–0.004(2)	0.003(2)	–0.001(2)
C(7)	2*a*	0.857(1)	0.3229(9)	0.8709(3)	0.028(2)	0.027(2)	0.023(2)	–0.003(2)	0.001(2)	0.004(2)
C(8)	2*a*	0.949(1)	0.1719(8)	0.8150(3)	0.030(2)	0.020(3)	0.036(3)	0.001(1)	–0.004(2)	0.007(2)
C(9)	2*a*	0.8520(9)	0.226(1)	0.7362(2)	0.026(2)	0.022(2)	0.028(2)	–0.002(3)	0.004(1)	–0.006(3)

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