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Crystal structure of 8-bromo-6-chloro-1′,3′,3′-trimethyl-spiro[2*H*-1-benzopyran-2,2′-indoline], $C_{19}H_{17}BrClNO$

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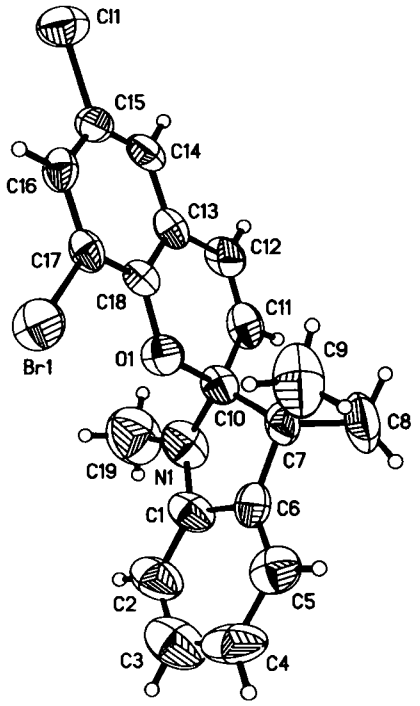

Abstract

$C_{19}H_{17}BrClNO$, tetragonal, $P4_3$ (no. 78), a = 8.1499(8) Å, c = 26.863(5) Å, V = 1784.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.061, $wR_{ref}(F^2)$ = 0.202, T = 294 K.

Source of material

The title compound was synthesized by the method described in [1]. The crude product was purified by silica gel column chromatography using the mixed solvent petroleum ether/ethyl acetate (3:1, *v*:*v*) as the eluent. Single crystals suitable for X-ray crystallography were obtained from an acetone solution by slow evaporation at room temperature.

Discussion

Spiropyrans and spirooxazines are an important classes of photochromic material [2]. The photochromic compounds continue to attract significant attention in view of their general applicability as optical information storage material or switching devices [3-5]. Many modified spiropyrans and spirooxazines had been prepared in order to develop novel photochromic materials [6-9].

In the title structure, the pyrrolidine ring adopts an envelope conformation, while the pyran ring is in a twist-boat conformation. The bond angles at spiro atom C10 deviate from the normal value of 109.5° and lie in the range 103.0(1)° – 116.8(1)°. The dihedral angle between the C1/C2/C3/C4/C5/C6 plane and the C13/C14/C15/C16/C17/C18 plane is 87.4(4)°. The equation of the C10/C11/C12/C13/C18/O1 plane is 5.62(3)x + 5.85(3)y – 2.5(1)z = 2.2(1) with atom C10 lying only –0.0104 Å from the plane. Although atom C10 has sp^3 hybridization and the ring is not an aromatic ring, all the six atoms on the ring lie on the same plane. The dihedral angle between the C10/C11/C12/C13/C18/O1 plane and the C13/C14/C15/C16/C17/C18 plane is 1.7(7)°, which shows the two rings are basically on the same plane. The equation of the C1/C6/C7/N1 plane is 4.99(5)x – 3.90(5)y + 16.9(2)z = 14.0(2). The deviation of atom C10 from the plane being 0.3836 Å is relatively large, which shows the five-membered ring composed of C1,C6,C7,N1,C10 is in an envelope conformation. The dihedral angle between this plane and the C1/C2/C3/C4/C5/C6 plane is 4(1)°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.12 × 0.20 × 0.24 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	24.57 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8965, 2468
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1208
$N(param)_{refined}$:	211
Programs:	SHELXS-97 [10], SHELXL-97 [11], SHELXTL [12]

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

Atom	Site	x	y	z	U_{iso}
H(2)	4*a*	0.2890	0.9315	0.9540	0.156
H(3)	4*a*	0.0989	0.9883	1.0110	0.184
H(4)	4*a*	–0.1001	0.7925	1.0301	0.181
H(5)	4*a*	–0.1161	0.5505	0.9825	0.134
H(8A)	4*a*	–0.1395	0.4738	0.8832	0.259
H(8B)	4*a*	–0.0348	0.3577	0.8492	0.259
H(8C)	4*a*	–0.0212	0.5485	0.8431	0.259
H(9A)	4*a*	0.1621	0.2768	0.9565	0.266
H(9B)	4*a*	0.1170	0.2035	0.9043	0.266
H(9C)	4*a*	–0.0220	0.2660	0.9399	0.266
H(11)	4*a*	0.2385	0.4995	0.8069	0.110
H(12)	4*a*	0.4164	0.3144	0.7821	0.104
H(14)	4*a*	0.6453	0.1107	0.8045	0.096
H(16)	4*a*	0.7661	0.0380	0.9446	0.088
H(19A)	4*a*	0.3943	0.8708	0.8718	0.283
H(19B)	4*a*	0.4666	0.7150	0.8456	0.283
H(19C)	4*a*	0.5139	0.7546	0.9009	0.283

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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)	4*a*	0.5605(2)	0.2583(2)	1.00375(6)	0.143(1)	0.172(2)	0.0546(7)	0.046(1)	−0.0243(9)	−0.003(1)
Cl(1)	4*a*	0.8591(5)	−0.0896(4)	0.8549(2)	0.110(3)	0.097(3)	0.138(4)	0.013(2)	0.026(3)	−0.009(2)
O(1)	4*a*	0.384(1)	0.408(1)	0.9175(3)	0.107(6)	0.110(6)	0.048(4)	0.026(5)	−0.020(4)	−0.001(4)
N(1)	4*a*	0.295(2)	0.667(2)	0.8931(5)	0.13(1)	0.098(9)	0.11(1)	−0.045(7)	0.021(8)	0.006(7)
C(1)	4*a*	0.201(2)	0.716(2)	0.9338(6)	0.14(1)	0.059(9)	0.09(1)	0.016(9)	−0.03(1)	0.001(8)
C(2)	4*a*	0.206(3)	0.856(2)	0.9601(8)	0.18(2)	0.08(1)	0.13(2)	0.02(1)	−0.03(2)	−0.02(1)
C(3)	4*a*	0.096(4)	0.889(3)	0.994(1)	0.24(3)	0.09(1)	0.13(2)	0.02(2)	−0.04(2)	0.00(1)
C(4)	4*a*	−0.025(3)	0.773(3)	1.005(1)	0.19(2)	0.15(2)	0.11(1)	0.11(2)	−0.02(2)	−0.01(2)
C(5)	4*a*	−0.034(2)	0.627(2)	0.9764(7)	0.12(1)	0.12(1)	0.10(1)	0.02(1)	−0.00(1)	0.01(1)
C(6)	4*a*	0.078(2)	0.599(2)	0.9410(5)	0.073(8)	0.10(1)	0.081(9)	0.005(8)	−0.023(8)	0.012(8)
C(7)	4*a*	0.102(2)	0.464(2)	0.9045(5)	0.08(1)	0.086(9)	0.083(9)	0.006(7)	−0.038(7)	−0.003(7)
C(8)	4*a*	−0.036(2)	0.461(3)	0.8665(9)	0.13(2)	0.17(2)	0.22(2)	−0.03(1)	−0.09(2)	−0.04(2)
C(9)	4*a*	0.088(2)	0.285(2)	0.9288(9)	0.18(2)	0.11(1)	0.24(3)	−0.04(1)	−0.09(2)	0.08(2)
C(10)	4*a*	0.272(2)	0.496(1)	0.8837(5)	0.10(1)	0.045(7)	0.082(9)	−0.002(6)	−0.022(7)	0.014(6)
C(11)	4*a*	0.301(2)	0.448(2)	0.8311(5)	0.11(1)	0.11(1)	0.059(8)	−0.012(9)	−0.028(8)	0.012(7)
C(12)	4*a*	0.408(2)	0.338(2)	0.8158(4)	0.12(1)	0.11(1)	0.036(6)	0.008(9)	−0.019(7)	−0.007(6)
C(13)	4*a*	0.511(1)	0.255(1)	0.8510(4)	0.089(8)	0.069(7)	0.053(7)	−0.022(7)	−0.009(6)	0.002(6)
C(14)	4*a*	0.628(2)	0.136(2)	0.8379(5)	0.090(9)	0.080(9)	0.070(8)	−0.022(7)	0.013(7)	−0.029(7)
C(15)	4*a*	0.715(1)	0.058(1)	0.8727(5)	0.072(8)	0.067(8)	0.077(9)	−0.003(6)	0.005(7)	−0.010(6)
C(16)	4*a*	0.701(2)	0.091(2)	0.9212(5)	0.073(8)	0.075(8)	0.072(9)	−0.007(6)	−0.018(6)	0.014(6)
C(17)	4*a*	0.585(2)	0.210(1)	0.9359(4)	0.088(8)	0.075(8)	0.052(6)	−0.010(7)	−0.020(6)	0.002(6)
C(18)	4*a*	0.493(1)	0.291(1)	0.9014(4)	0.079(7)	0.060(7)	0.057(7)	−0.010(6)	−0.001(6)	−0.005(6)
C(19)	4*a*	0.428(3)	0.759(3)	0.877(1)	0.19(1)	0.18(1)	0.19(1)	−0.051(9)	0.020(9)	−0.011(9)

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