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8-Bromo-6-chloro-1′,3′,3′,5′-tetramethyl-spiro[2*H*-1-benzopyran-2,2′-indoline]

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Key indicators

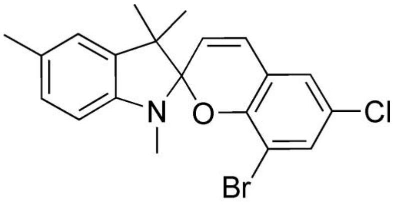
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.029
wR factor = 0.069
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

In the title compound, $C_{20}H_{19}BrClNO$, the pyrrolidine ring adopts an envelope conformation, while the pyran ring is in a twist-boat form.

Comment

Spiropyrans and spirooxazines are important classes of photochromic compounds (Winkler *et al.*, 1998). Photochromic compounds continue to attract significant attention in view of their general applicability as optical information storage materials or switching devices (Duerr, 1989; Duerr & Bouas-Laurent, 1990; Ichi, 2000). Many modified spiropyrans and spirooxazines have been prepared in order to develop novel photochromic materials. The synthesis and the properties of certain spiropyrans and spirooxazines containing various functional groups have been studied recently (Li *et al.*, 1999; Zou *et al.*, 2003; Song *et al.*, 2003; Guo *et al.*, 2005). The title compound, (I), a spiropyran with Br and Cl substituents, was prepared for the first time in our laboratory. Compound (I) has been characterized by ^{1}H NMR, mass spectroscopy and X-ray analysis. We report its crystal structure here.



(I)

A perspective view of (I), with the atom-labeling scheme, is shown in Fig. 1. The pyrrolidine ring is in an envelope conformation, with atom C9 lying 0.408 (4) Å from the C10–C12/N1 plane. The pyran ring adopts a twist-boat conformation, the deviations of atoms O1 and C6 from the C5/C7–C9 plane being 0.264 (5) and 0.132 (5) Å, respectively. The dihedral angle between the C10–C12/N1 and C5/C7–C9 planes is 81.8 (1)°. Some of the bond angles at spiro atom C9 deviate from the normal value of 109.5°; the angles lie in the range 105.8 (2)–115.9 (2)° (Table 1).

Experimental

Compound (I) was synthesized by the method described by Ono *et al.* (1971). The crude product was purified by silica gel column chromatography using the mixed solvent petroleum ether/ethyl acetate (3:1 *v*/*v*) as eluant. Single crystals of (I) suitable for X-ray study were obtained from an acetone solution by slow evaporation at room temperature.

Crystal data

$C_{20}H_{19}BrClNO$
M_r = 404.72
Tetragonal, $P4_3$
a = 8.2117 (6) Å
c = 27.440 (3) Å
V = 1850.3 (3) Å^3
Z = 4
D_x = 1.453 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3221 reflections
θ = 2.5–22.0°
μ = 2.37 mm^{-1}
T = 293 (2) K
Prism, colorless
0.38 × 0.16 × 0.12 mm

Data collection

Bruker APEX-II CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min}$ = 0.521, $T_{\max}$ = 0.752
10 024 measured reflections
3243 independent reflections
2646 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.030
$\theta_{\max}$ = 25.0°
$h = -8 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.069
S = 1.02
3243 reflections
221 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.028P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0.001
$\Delta\rho_{\max}$ = 0.27 e Å^{-3}
$\Delta\rho_{\min}$ = −0.30 e Å^{-3}
Extinction correction: none
Absolute structure: Flack (1983), 1578 Friedel pairs
Flack parameter: 0.014 (7)

Table 1
Selected bond angles (°).

N1—C9—O1	105.8 (2)	N1—C9—C10	103.6 (2)
N1—C9—C8	112.0 (2)	O1—C9—C10	108.0 (2)
O1—C9—C8	110.9 (2)	C8—C9—C10	115.9 (2)

All H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances of 0.93 and 0.96 Å, and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(C) for methyl H atoms and 1.2$U_{\rm eq}$(C) for other H atoms. Each methyl group was allowed to rotate freely about its C—C bond.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

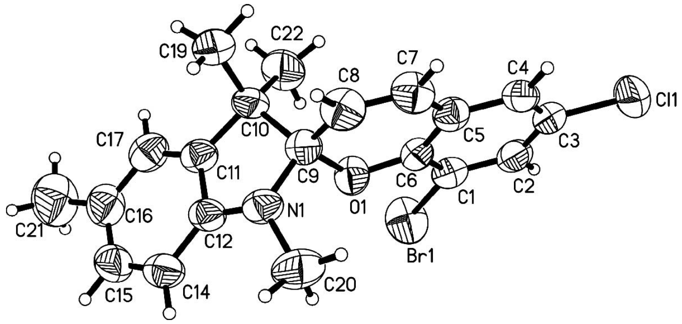


Figure 1
Structure of (I), showing 30% probability displacement ellipsoids and the atom-labeling scheme.

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