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Crystal structure of (*E*)-2-bromo-4-chloro-6-(4-methoxy-2-nitrostyryl)phenol, C₁₄H₁₀BrClN₂O₄

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Abstract

C₁₄H₁₀BrClN₂O₄, monoclinic, *P*2₁/*c* (no. 14), *a* = 14.2726(16) Å, *b* = 6.9919(9) Å, *c* = 15.1927(15) Å, β = 107.308(5)°, *V* = 1447.5(3) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0362, *wR*_{ref}(*F*²) = 0.0831, *T* = 150(2) K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

4-Methoxy-2-nitroaniline (1 mmol, 0.168 g) and 3-bromo-5-chlorosalicylaldehyde (1 mmol, 0.384 g) were added to the solution of ethanol (10 mL). The mixture was refluxed for 4 h. After filtering the residue was dissolved in dichloromethane and the solution was evaporated slowly at room temperature to obtain colorless prismatic crystals suitable for X-ray structure determination.

Experimental details

The H atoms were positioned geometrically with *d*(C–H) = 0.93–0.98 Å and refined as riding with

Table 1: Data collection and handling.

Crystal:	Yellow stick
Size:	0.32 × 0.21 × 0.14 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	3.04 mm ^{−1}
Diffractometer, scan mode:	Bruker SMART, φ and ω-scans
θ _{max} , completeness:	26°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	11197, 2858, 0.073
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2049
<i>N</i> (<i>param</i>) _{refined} :	201
Programs:	Bruker programs [1], SHELX [2], OLEX2 [3]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso} */ <i>U</i> _{eq}
Cl1	0.19654(7)	0.37859(14)	−0.12533(5)	0.0336(2)
N1	0.54689(18)	0.3026(4)	0.46732(15)	0.0170(6)
N2	0.48356(18)	0.4400(3)	0.28106(15)	0.0149(5)
O1	0.88182(16)	0.4503(3)	0.47843(14)	0.0265(5)
O2	0.56274(17)	0.3315(3)	0.55027(13)	0.0263(5)
O3	0.47666(16)	0.2090(3)	0.42055(13)	0.0241(5)
O4	0.29951(16)	0.5089(3)	0.27384(13)	0.0225(5)
H4	0.359548	0.489763	0.299512	0.034*
Br1	0.08251(2)	0.55111(6)	0.18204(2)	0.03328(13)
C1	0.9132(3)	0.4177(5)	0.5762(2)	0.0313(9)
H1A	0.895878	0.287220	0.588983	0.047*
H1B	0.880726	0.509322	0.606267	0.047*
H1C	0.984500	0.434410	0.599999	0.047*
C2	0.7836(2)	0.4399(5)	0.43497(19)	0.0192(7)
C3	0.7548(2)	0.4838(4)	0.3410(2)	0.0215(7)
H3	0.803281	0.515879	0.312072	0.026*
C4	0.6576(2)	0.4813(4)	0.2896(2)	0.0192(7)
H4A	0.639887	0.516769	0.226436	0.023*
C5	0.5841(2)	0.4273(4)	0.32864(19)	0.0145(6)
C6	0.6166(2)	0.3786(4)	0.42246(19)	0.0150(6)
C7	0.7131(2)	0.3883(4)	0.47610(19)	0.0157(7)
H7	0.730751	0.360137	0.540121	0.019*
C8	0.4537(2)	0.4041(4)	0.1938(2)	0.0167(7)
H8	0.499459	0.362285	0.163488	0.020*
C9	0.3510(2)	0.4269(4)	0.14118(19)	0.0149(6)
C10	0.3244(2)	0.3946(4)	0.04569(19)	0.0179(7)
H10	0.373004	0.361762	0.017075	0.021*
C11	0.2279(2)	0.4108(4)	−0.00620(19)	0.0198(7)
C12	0.1550(2)	0.4543(4)	0.03389(19)	0.0207(7)
H12	0.088260	0.461469	−0.002474	0.025*
C13	0.1814(2)	0.4871(4)	0.1278(2)	0.0200(7)
C14	0.2788(2)	0.4757(4)	0.18352(19)	0.0160(6)

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$U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{carrier})$ or $1.5 U_{\text{eq}}(\text{methyl})$. Solving, refining and finishing of the crystal structure were finished by OLEX2 [2] and SHELXS [3].

Discussion

Schiff bases of salicylaldehyde with amines(anils) comprise a chemical system undergoing hydrogen-atom tautomerism between enol and keto forms and show the phenomena of solid state photochromism and thermochromism [4]. Similar to the crystal structure of the related Schiff base [5, 6], the title compound with electron-withdrawing a chloro substituent and a bromo substituent (*cf.* the figure) clearly shows the enol form. The molecule displays a *E*-configuration at the central C8=N2 bond. The dihedral angle between the two substituted phenyl moieties (C2—C3—C4—C5—C6—C7 and C9—C10—C11—C12—C13—C14) is 30.7°. There is an intramolecular O—H...N hydrogen bond in the molecular structure of the title compound. All bond lengths and angles are very similar to the halogene-free parent compound [7].

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