Yong Hong*, Gaozhang Gou, Wei Liu* and Heping Yan

Crystal structure of (E)-2,4-dibromo-6-(((4-methyl-2-nitrophenyl)imino)methyl)phenol, C_{10}H_{14}Br_{2}N_{2}O_{3}

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Abstract

C_{14}H_{10}Br_{2}N_{2}O_{3}, monoclinic, P2_{1}/c (no. 14), a = 10.4183(3) Å, b = 11.5741(3) Å, c = 14.0587(4) Å, β = 89.011(3)°, V = 1455.0(5) Å^3, Z = 4, R_{gt}(F) = 0.0474, wR_{ref}(F^2) = 0.1314, T = 293 K.

CCDC no.: 1465500

The asymmetric unit of the title 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-Methyl-2-nitroaniline (1 mmol, 0.152 g) and 3,5-dibromosalicylaldehyde (1 mmol, 0.180 g) were added to ethanol (10 mL). The mixture was refluxed for 4 h. The solution was evaporated slowly at room temperature to obtain colorless prismatic crystals of the title compound.

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Table 1: Data collection and handling.

| Crystal: | Colourless prism |
| Size: | 0.28 × 0.24 × 0.21 mm |
| Wavelength: | Cu Kα radiation (1.54178 Å) |
| μ: | 71.8 cm^{-1} |
| Diffractometer, scan mode: | Bruker APEX-II, ω-scans |
| 2θ_{max}, completeness: | 136.4°, >97% |
| N(hkl)_{measured}, N(hkl)_{unique}, R_{int}: | 8305, 2601, 0.078 |
| Criterion for I_{obs}, N(hkl)_{gt}: | I_{obs} > 2σ(I_{obs}), 2188 |
| N(param)_{refined}: | 192 |
| Programs: | Bruker programs [1], SHELX [2], OLEX2 [3] |

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

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>U_{iso}/U_{eq}</th>
</tr>
</thead>
<tbody>
<tr>
<td>Br1</td>
<td>0.42470(3)</td>
<td>−0.08083(7)</td>
<td>0.22617(3)</td>
<td>0.0662(2)</td>
</tr>
<tr>
<td>Br2</td>
<td>0.33951(4)</td>
<td>0.12776(8)</td>
<td>0.57074(3)</td>
<td>0.0762(2)</td>
</tr>
<tr>
<td>C1</td>
<td>0.3315(2)</td>
<td>−0.0009(5)</td>
<td>0.3031(2)</td>
<td>0.0437(7)</td>
</tr>
<tr>
<td>C2</td>
<td>0.3642(2)</td>
<td>0.0345(5)</td>
<td>0.3912(2)</td>
<td>0.0482(8)</td>
</tr>
<tr>
<td>H2</td>
<td>0.4317</td>
<td>0.0244</td>
<td>0.4127</td>
<td>0.058*</td>
</tr>
<tr>
<td>C3</td>
<td>0.2948(3)</td>
<td>0.0856(5)</td>
<td>0.4480(2)</td>
<td>0.0462(8)</td>
</tr>
<tr>
<td>C4</td>
<td>0.2312(2)</td>
<td>0.0149(5)</td>
<td>0.2679(2)</td>
<td>0.0401(7)</td>
</tr>
<tr>
<td>C5</td>
<td>0.1950(3)</td>
<td>0.1060(4)</td>
<td>0.4165(2)</td>
<td>0.0435(7)</td>
</tr>
<tr>
<td>H5</td>
<td>0.1496</td>
<td>0.1431</td>
<td>0.4551</td>
<td>0.052*</td>
</tr>
<tr>
<td>C6</td>
<td>0.1624(2)</td>
<td>0.0706(4)</td>
<td>0.3265(2)</td>
<td>0.0378(7)</td>
</tr>
<tr>
<td>C7</td>
<td>0.0566(2)</td>
<td>0.0962(4)</td>
<td>0.2943(2)</td>
<td>0.0387(7)</td>
</tr>
<tr>
<td>H7</td>
<td>0.0129</td>
<td>0.1364</td>
<td>0.3337</td>
<td>0.046*</td>
</tr>
<tr>
<td>C8</td>
<td>−0.0794(2)</td>
<td>0.0806(4)</td>
<td>0.1820(2)</td>
<td>0.0372(6)</td>
</tr>
<tr>
<td>C9</td>
<td>−0.1097(2)</td>
<td>0.1342(4)</td>
<td>0.0934(2)</td>
<td>0.0355(7)</td>
</tr>
<tr>
<td>C10</td>
<td>−0.2087(2)</td>
<td>0.1283(4)</td>
<td>0.0550(2)</td>
<td>0.0420(7)</td>
</tr>
<tr>
<td>H10</td>
<td>−0.2256</td>
<td>0.1612</td>
<td>−0.0047</td>
<td>0.050*</td>
</tr>
<tr>
<td>C11</td>
<td>−0.2819(2)</td>
<td>0.0731(5)</td>
<td>0.1063(2)</td>
<td>0.0437(7)</td>
</tr>
<tr>
<td>C12</td>
<td>−0.2538(2)</td>
<td>0.0242(5)</td>
<td>0.1948(2)</td>
<td>0.0469(8)</td>
</tr>
<tr>
<td>H12</td>
<td>−0.3029</td>
<td>−0.0099</td>
<td>0.2302</td>
<td>0.056*</td>
</tr>
<tr>
<td>C13</td>
<td>−0.1542(2)</td>
<td>0.0247(5)</td>
<td>0.2324(2)</td>
<td>0.0420(7)</td>
</tr>
<tr>
<td>H13</td>
<td>−0.1375</td>
<td>0.0126</td>
<td>0.2915</td>
<td>0.050*</td>
</tr>
<tr>
<td>C14</td>
<td>−0.3897(3)</td>
<td>0.0651(6)</td>
<td>0.0655(3)</td>
<td>0.0591(10)</td>
</tr>
<tr>
<td>H14A</td>
<td>−0.4132</td>
<td>−0.0599</td>
<td>0.0665</td>
<td>0.089*</td>
</tr>
<tr>
<td>H14B</td>
<td>−0.3950</td>
<td>0.1082</td>
<td>0.0048</td>
<td>0.089*</td>
</tr>
<tr>
<td>H14C</td>
<td>−0.4296</td>
<td>0.1419</td>
<td>0.0994</td>
<td>0.089*</td>
</tr>
<tr>
<td>N1</td>
<td>0.02343(19)</td>
<td>0.0638(4)</td>
<td>0.21228(18)</td>
<td>0.0376(6)</td>
</tr>
</tbody>
</table>

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Table 2 (continued)

<table>
<thead>
<tr>
<th>Atom</th>
<th>x</th>
<th>y</th>
<th>z</th>
<th>Uiso*/Ueq</th>
</tr>
</thead>
<tbody>
<tr>
<td>N2</td>
<td>-0.03684(19)</td>
<td>0.2107(4)</td>
<td>0.03842(18)</td>
<td>0.0416(6)</td>
</tr>
<tr>
<td>O1</td>
<td>0.20455(17)</td>
<td>-0.0221(4)</td>
<td>0.18116(16)</td>
<td>0.0517(6)</td>
</tr>
<tr>
<td>H1</td>
<td>0.14554</td>
<td>0.00454</td>
<td>0.1677</td>
<td>0.077*</td>
</tr>
<tr>
<td>O2</td>
<td>0.03179(18)</td>
<td>0.3047(6)</td>
<td>0.07455(17)</td>
<td>0.0596(7)</td>
</tr>
<tr>
<td>O3</td>
<td>-0.0484(2)</td>
<td>0.1801(6)</td>
<td>-0.04213(17)</td>
<td>0.0613(7)</td>
</tr>
</tbody>
</table>

Experimental details

The H atoms were positioned geometrically with d(C–H) = 0.93–0.98 Å and refined as riding with Uiso(H) = 1.2 Ueq(carrier) or 1.5 Ueq(methyl).

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]. Compared with crystal structure of the related Schiff base [5], the title compound with electron-withdrawing bromo substituents (cf. the figure) clearly shows the enol form.

The molecule displays an E-configuration at the central C6=N2 bond. The dihedral angle between the two substitutated phenyl moieties (C1–C2–C3–C4–C5–C6 and C8–C9–C10–C11–C12–C13) is 35.34°. There is an intramolecular O–H···N hydrogen bond molecular structure of the title compound. All bond lengths and angles are in the expected ranges.

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References