

**(E)-5-(4-Bromophenyldiazenyl)salicylaldehyde****Onur Şahin,<sup>a</sup> Çiğdem Albayrak,<sup>b</sup>  
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**Key indicators**

Single-crystal X-ray study

T = 296 K

Mean  $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$ 

R factor = 0.033

wR factor = 0.080

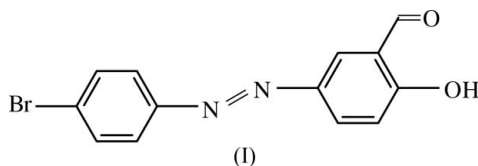
Data-to-parameter ratio = 16.4

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

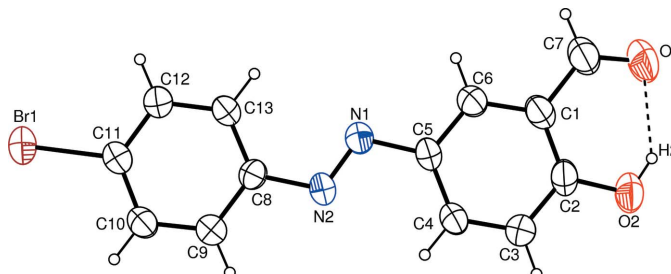
The molecule of the title compound,  $\text{C}_{13}\text{H}_9\text{BrN}_2\text{O}_2$ , is approximately planar and displays a *trans* configuration with respect to the  $\text{N}=\text{N}$  double bond. The dihedral angle between the two aromatic rings is  $3.65 (16)^\circ$ . The molecules are linked by intermolecular  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds, forming a two-dimensional network.

**Comment**

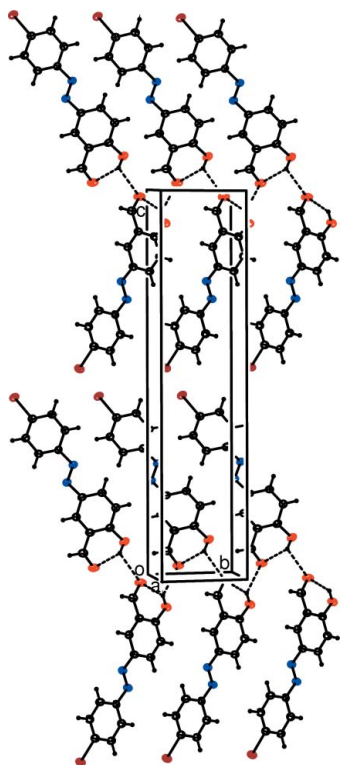
Optically active azobenzene polymers are very important functional materials because of their photoresponsive properties. The pendant azobenzene groups in these materials behave as both photoresponsive chromophores and mesogens (Labarthe *et al.*, 1999). Recently, the formation of holographic gratings with polymeric azobenzene liquid crystals containing only azobenzene groups, each group being mesogenic and photoresponsive, was reported (Yamamoto *et al.*, 2001).



An *ORTEP3* (Farrugia, 1997) view of the molecule of the title compound, (I), and a packing diagram are shown in Figs. 1 and 2, respectively. Compound (I) is isostructural with the corresponding Cl-containing compound, (*E*)-5-(4-chlorophenyldiazenyl)salicylaldehyde (Şahin *et al.*, 2005), with small differences for some bond lengths due to the Br attached to the C8–C13 benzene ring. In the azo group, the N1–C5 and N2–C8 bond lengths (Table 1) indicate significant single-bond character, whereas the N1=N2 bond length is indicative of significant double-bond character. Similar values have been observed in other *trans*-azo compounds (*e.g.* Şahin *et al.*, 2005).

**Figure 1**

A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids for non-H atoms. The intramolecular hydrogen bond is shown as a dashed line.



**Figure 2**  
A packing diagram for (I), showing hydrogen bonds as dashed lines.

The C7=O1 and C2–O2 bond lengths agree with the corresponding distances in the chloro compound [1.223 (4) and 1.347 (3) Å, respectively; Şahin *et al.*, 2005].

Compound (I) consists of benzene rings *A* (C1–C6) and *B* (C8–C13), their substituents, and the azo unit *C* (C5–N1=N2–C8). Benzene rings *A* and *B* adopt a *trans* configuration about the azo functional group, as observed in the crystal structures of other previously studied azo compounds. In (I), the dihedral angle between *A* and *B* rings is 3.65 (16)°, the dihedral angle for *A/C* is 3.3 (3)°, and 0.6 (4)° for *B/C*. Compound (I) also displays intramolecular and weak intermolecular O–H...O hydrogen bonds (Table 2), giving a two-dimensional network for the crystal structure (Fig. 2).

## Experimental

The title compound was prepared according to the literature method of Odabaşoğlu *et al.* (2003), using *p*-bromoaniline and salicylaldehyde as starting materials. The product was crystallized from toluene to obtain well shaped crystals (yield 74%; m.p. 489–491 K).

### Crystal data

|  |   |
|--|---|
| C <sub>13</sub> H <sub>9</sub> BrN <sub>2</sub> O <sub>2</sub> | $D_x = 1.744 \text{ Mg m}^{-3}$           |
| $M_r = 305.13$   | Mo $K\alpha$ radiation                    |
| Monoclinic, $P2_1$   | Cell parameters from 9332 reflections     |
| $a = 3.8914$ (6) Å   | $\theta = 1.6$ – $28.0^\circ$             |
| $b = 5.8043$ (5) Å   | $\mu = 3.53 \text{ mm}^{-1}$              |
| $c = 25.745$ (4) Å   | $T = 296 \text{ K}$                       |
| $\beta = 92.101$ (12)°   | Plate, orange                             |
| $V = 581.11$ (13) Å <sup>3</sup>                               | $0.50 \times 0.29 \times 0.04 \text{ mm}$ |
| $Z = 2$  |   |

### Data collection

|  |  |
|--|--|
| Stoe IPDS-2 diffractometer   | 2547 reflections with $I > 2\sigma(I)$ |
| $\omega$ scans   | $R_{\text{int}} = 0.082$               |
| Absorption correction: integration<br>( <i>X-RED32</i> ; Stoe & Cie, 2002) | $\theta_{\text{max}} = 28.0^\circ$     |
| $T_{\text{min}} = 0.258$ , $T_{\text{max}} = 0.854$                        | $h = -5 \rightarrow 5$                 |
| 9332 measured reflections  | $k = -7 \rightarrow 7$                 |
| 2744 independent reflections   | $l = -33 \rightarrow 33$               |

### Refinement

|  |  |
|--|--|
| Refinement on $F^2$  | $w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.0931P]$    |
| $R[F^2 > 2\sigma(F^2)] = 0.033$  | where $P = (F_o^2 + 2F_c^2)/3$                       |
| $wR(F^2) = 0.080$  | $(\Delta/\sigma)_{\text{max}} < 0.001$               |
| $S = 1.07$   | $\Delta\rho_{\text{max}} = 0.31 \text{ e \AA}^{-3}$  |
| 2744 reflections   | $\Delta\rho_{\text{min}} = -0.99 \text{ e \AA}^{-3}$ |
| 167 parameters   | Absolute structure: Flack (1983),                    |
| H atoms treated by a mixture of independent and constrained refinement | with 1213 Friedel pairs                              |
|  | Flack parameter = 0.140 (11)                         |

**Table 1**  
Selected geometric parameters (Å, °).

|             |            |           |           |
|-------------|------------|-----------|-----------|
| C2–O2       | 1.357 (4)  | C8–N2     | 1.426 (4) |
| C5–N1       | 1.426 (4)  | N1–N2     | 1.259 (4) |
| C7–O1       | 1.227 (5)  |           |           |
| O2–C2–C3    | 116.6 (4)  | C4–C5–N1  | 124.4 (3) |
| O2–C2–C1    | 123.5 (3)  | C9–C8–N2  | 115.5 (3) |
| C6–C5–N1    | 117.0 (3)  | C13–C8–N2 | 124.4 (3) |
| C5–N1–N2–C8 | –179.1 (3) |           |           |

**Table 2**  
Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$           | $D-H$    | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-------------------------|----------|-------------|-------------|---------------|
| O2–H2...O1              | 0.84 (6) | 1.99 (6)    | 2.693 (4)   | 141 (5)       |
| O2–H2...O1 <sup>i</sup> | 0.84 (6) | 2.41 (6)    | 2.929 (4)   | 121 (5)       |

Symmetry code: (i)  $-x, y + \frac{1}{2}, -z + 2$ .

All C-bonded H atoms were refined using a riding model, with C–H distances constrained to 0.93 Å and  $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{C})$ . The H atom of the hydroxyl group was found in a difference map and refined with an O–H distance restrained to 0.83 (5) Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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