

**(E)-2-Hydroxy-3-methoxy-5-[(3-methoxyphenyl)diazenyl]benzaldehyde**Nevzat Karadayı,<sup>a</sup> Çiğdem Albayrak,<sup>b</sup> Mustafa Odabaşoğlu<sup>b</sup> and Orhan Büyükgüngör<sup>c\*</sup><sup>a</sup>Industrial Electronics Program, Samsun Vocational School, Ondokuz Mayıs University, TR-55139 Samsun, Turkey, <sup>b</sup>Department of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139 Samsun, Turkey, and <sup>c</sup>Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, TR-55139, Samsun, Turkey

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**Key indicators**Single-crystal X-ray study  
*T* = 296 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
*R* factor = 0.046  
*wR* factor = 0.078  
Data-to-parameter ratio = 12.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

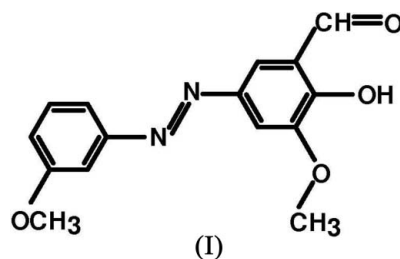
The molecule of the title compound,  $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_4$ , is approximately planar and displays a *trans* configuration with respect to the  $\text{N}=\text{N}$  double bond. The molecules are linked by intermolecular  $\text{C}-\text{H}\cdots\text{O}$  and  $\text{C}-\text{H}\cdots\pi$  interactions, forming a three-dimensional network.

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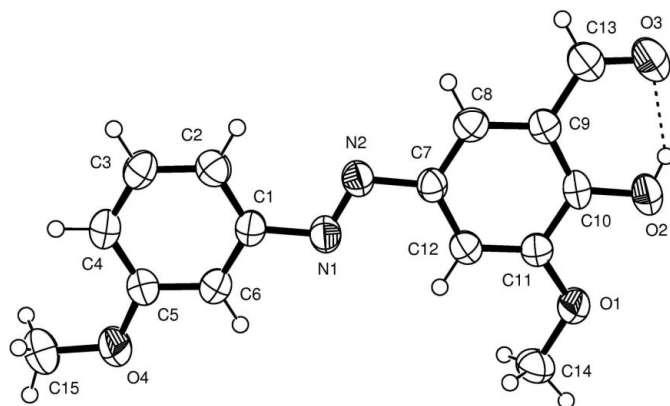
**Comment**

Azobenzenes have a wide variety of potential applications for organic non-linear optics, optical storage media, chemosensors, and photochemical switches because of their characteristic colour and photoresponsive properties (Kim *et al.*, 2004). In addition, azo compounds represent the dominant class of synthetic colourants employed in the textile, printing, agrochemical and pharmaceutical industries. As a result of the presence of the stable chromophoric azo group ( $-\text{N}=\text{N}-$ ) linking different aromatic units carrying electron-donating and/or electron-withdrawing groups, the dyes can be designed to resist chemical or photochemical degradation processes (Evans *et al.*, 1980; Griffiths & Allen, 1980; Leaver *et al.*, 1980; Sharma *et al.*, 2002; Neta & Levanon, 1977; Flamingi & Monti, 1985). We are interested in the synthesis of functionalized azobenzenes for the development of the crystal chemistry of dyes.



An *ORTEP*-3 (Farrugia, 1997) view of the molecule of (I) is shown in Fig. 1. The  $\text{C}1-\text{N}1=\text{N}2-\text{C}7$  azo linkage is coplanar with the multiply-substituted benzene ring ( $\text{C}17-\text{C}12$ ) [dihedral angle =  $0.8(3)^\circ$ ]. The dihedral angle between the mean planes of the azo linkage and the methoxyphenyl ( $\text{C}1-\text{C}6$ ) ring is  $5.5(3)^\circ$ . The dihedral angle between the planes of the two aromatic rings is  $6.0(1)^\circ$ . The aromatic rings are in a *trans* configuration with respect to the azo functional group.

All the  $\text{C}-\text{C}$  bond distances in the benzene rings have typical  $\text{Csp}^2-\text{Csp}^2$  values. The average  $\text{C}-\text{C}$  bond distances within these two rings are  $1.381(3)$  and  $1.389(3)$  Å. The  $\text{N}1-\text{C}1$ ,  $\text{N}2-\text{C}7$  and  $\text{N}1=\text{N}2$  bond lengths (Table 1) are comparable to those found for similar compounds (Karadayı *et al.*, 2004; Odabaşoğlu *et al.*, 2005; Filip *et al.*, 2001). The  $\text{O}1-\text{C}14$  and  $\text{O}4-\text{C}15$  bond lengths are  $1.432(2)$  and  $1.424(3)$  Å,


**Figure 1**

A view of (I), showing the atomic numbering scheme and 50% probability displacement ellipsoids. The intramolecular hydrogen bond is shown as a dashed line.

respectively. These values are within the expected ranges for structures with multiple methoxy substituents [1.419 (6)–1.434 (4) Å (Crano *et al.*, 1994) and 1.436 (3)–1.413 (4) Å (Gales *et al.*, 2001)].

An intramolecular O–H···O hydrogen bond is observed in the molecular structure (Fig. 1). The crystal packing is stabilized by C–H···O hydrogen bonds (Table 2). There is also a weak C–H··· $\pi$  interaction between C14–H14*B* and the C1–C6 ring.

## Experimental

Compound (I) was prepared according to the literature method of Odabaşoğlu *et al.* (2003), using *o*-vanillin and 3-methoxyaniline as starting materials. The product was crystallized from acetic acid to obtain well shaped crystals (yield 89%; m.p. 388–389 K).

### Crystal data

$C_{15}H_{14}N_2O_4$	$D_x = 1.382 \text{ Mg m}^{-3}$
$M_r = 286.28$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 8411 reflections
$a = 11.8486$ (14) Å	$\theta = 2.7\text{--}27.5^\circ$
$b = 9.9295$ (9) Å	$\mu = 0.10 \text{ mm}^{-1}$
$c = 12.6574$ (15) Å	$T = 296$ (2) K
$\beta = 112.441$ (9)°	Plate, brown
$V = 1376.4$ (3) Å <sup>3</sup>	$0.36 \times 0.27 \times 0.11 \text{ mm}$
$Z = 4$	

### Data collection

Stoe IPDS-2 diffractometer	$R_{\text{int}} = 0.083$
$\omega$ scans	$\theta_{\text{max}} = 25.0^\circ$
Absorption correction: none	$h = -14 \rightarrow 14$
16858 measured reflections	$k = -11 \rightarrow 11$
2414 independent reflections	$l = -14 \rightarrow 14$
1298 reflections with $I > 2\sigma(I)$	

### Refinement

Refinement on $F^2$	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.046$	$w = 1/[\sigma^2(F_o^2) + (0.026P)^2]$
$wR(F^2) = 0.078$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.99$	$(\Delta/\sigma)_{\text{max}} = 0.001$
2414 reflections	$\Delta\rho_{\text{max}} = 0.12 \text{ e \AA}^{-3}$
190 parameters	$\Delta\rho_{\text{min}} = -0.13 \text{ e \AA}^{-3}$

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

C1–C6	1.371 (3)	C7–C12	1.400 (3)
C1–C2	1.392 (3)	C7–N2	1.423 (3)
C1–N1	1.433 (3)	C9–C13	1.447 (3)
C5–O4	1.361 (3)	N1–N2	1.255 (2)
C7–C8	1.375 (3)		
C6–C1–N1	114.9 (2)	C12–C7–N2	123.8 (2)
C2–C1–N1	124.8 (2)	O1–C11–C12	125.5 (2)
O4–C5–C4	125.1 (2)	O1–C11–C10	114.8 (2)
O4–C5–C6	115.5 (2)	O3–C13–C9	123.4 (2)
C8–C7–N2	116.4 (2)		
C10–C9–C13–O3	−1.5 (4)	C12–C11–O1–C14	1.5 (3)
C1–N1–N2–C7	179.80 (17)	C4–C5–O4–C15	−1.9 (3)

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

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
O2–H1···O3	0.82	1.89	2.606 (2)	146
C14–H14 <i>A</i> ···O4 <sup>i</sup>	0.96	2.49	3.188 (3)	130
C14–H14 <i>C</i> ···O3 <sup>ii</sup>	0.96	2.54	3.398 (3)	148
C14–H14 <i>B</i> ···Cg1 <sup>iii</sup>	0.96	2.83	3.667 (2)	147

Symmetry codes: (i)  $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$ ; (ii)  $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$ ; (iii)  $-x + 1, -y + 1, -z + 1$ . Cg1 is the centroid of the C1–C6 ring.

All C-bound H atoms were refined using a riding model, with C–H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  for aromatic and C–H = 0.96 Å and  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$  for methyl H atoms. The H atom of the hydroxyl group was refined with a O–H distance of 0.82 Å and with  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ .

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) and *PLATON* (Spek, 2003); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

## References

- Crano, J., Knowles, D. & Kwiatkowski, P. (1994). *Acta Cryst.* **B50**, 772–779.
- Evans, N. A., Allen, N. S. & McKellar, J. F. (1980). *Photochemistry of Dyed and Pigmented Polymers*, pp. 93–159. London: Elsevier.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Filip, L. F., Caira, M. R., Fărcaş, S. I. & Bojită, M. T. (2001). *Acta Cryst.* **C57**, 435–436.
- Flamingi, L. & Monti, S. (1985). *J. Phys. Chem.* **89**, 3702–3707.
- Gales, L., De Sousa, M. E., Pinto, M. M. M., Kijjoa, A. & Damas, A. M. (2001). *Acta Cryst.* **C57**, 1319–1323.
- Griffiths, J. & Allen, N. S. (1980). *Developments in Polymer Photochemistry*, Vol. 1, pp. 145–189. London: Applied Science Publishers.
- Karadayı, N., Turgut, G., Odabaşoğlu, M. & Büyükgüngör, O. (2004). *Acta Cryst.* **E60**, o176–o178.
- Kim, K.-Y., Shin, J.-T., Lee, K.-S. & Cho, C.-G. (2004). *Tetrahedron Lett.* **45**, 117–120.
- Leaver, I. H., Allen, N. S. & McKellar, J. F. (1980). *Photochemistry of Dyed and Pigmented Polymers*, pp. 161–245. London: Elsevier.
- Neta, P. & Levanon, H. (1977). *J. Phys. Chem.* **81**, 2288–2292.
- Odabaşoğlu, M., Albayrak, C., Büyükgüngör, O. & Goesmann, H. (2003). *Acta Cryst.* **C59**, o234–o236.
- Odabaşoğlu, M., Turgut, G., Karadayı, N. & Büyükgüngör, O. (2005). *Dyes Pigments*, **64**, 271–278.

Sharma, K. K., Rao, B. S. M., Mohan, H., Mittal, J. P., Oakes, J. & O'Neill, P. (2002). *J. Phys. Chem. A*, **106**, 2915–2923.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie, Darmstadt, Germany.