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## Key indicators

Single-crystal X-ray study  
 $T = 296\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$   
 $R$  factor = 0.030  
 $wR$  factor = 0.083  
Data-to-parameter ratio = 8.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**(E)-5-[(2-Fluorophenyl)diazenyl]-2-hydroxy-3-methoxybenzaldehyde**

The title compound,  $\text{C}_{14}\text{H}_{11}\text{FN}_2\text{O}_3$ , displays the characteristic features of azobenzene derivatives. Intramolecular  $\text{O}-\text{H}\cdots\text{O}$  and weak  $\pi-\pi$  interactions influence the conformation of the molecules and the crystal packing. The azobenzene moiety of the molecule has a *trans* configuration and the dihedral angle between the planes of the two aromatic rings is  $10.13(8)^\circ$ .

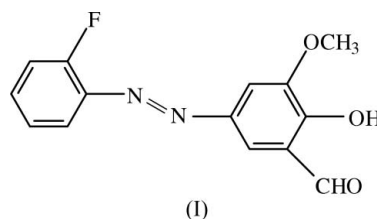
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## Comment

Azo compounds are among the largest group of dyes, with over one thousand compounds commercially available. Azo dyes have been developed for coloring fibers, both natural and synthetic, and for the coloration of solvents and a wide range of non-textile substrates (Kumar & Neckers, 1989). Moreover, azobenzene and some azobenzene derivatives can undergo *cis-trans* isomerization under photochemical stimulation, and may be involved in energy-transfer processes (Murakami *et al.*, 1997).



The molecular structure of (I) is shown in Fig. 1 with the atom-numbering scheme. Selected bond distances and angles are given in Table 1. The dihedral angle  $\Theta_1$  between the mean planes of the fluorophenyl ring (C1–C6) and the C1–N1=N2–C7 azo bridge is  $7.58(7)^\circ$ , and the angle  $\Theta_2$  between the C1–N1=N2–C7 azo group and the multiply substituted

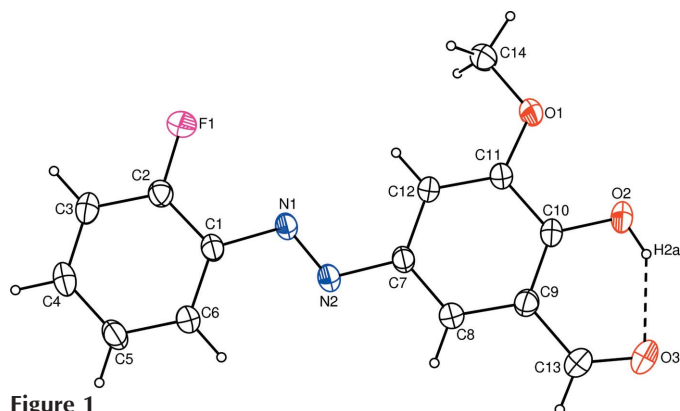
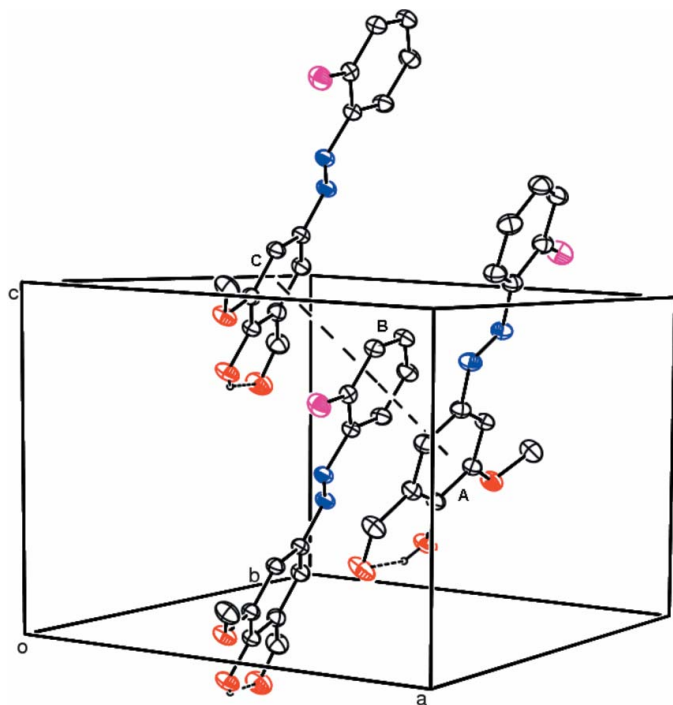


Figure 1

A view of (I) with the atom-numbering scheme and 30% probability displacement ellipsoids. The dashed line indicates the intramolecular hydrogen bond.



**Figure 2**  
Part of the crystal structure of (I). Dashed lines indicate  $\pi$ - $\pi$  interactions [symmetry codes: (i)  $x, y, 1 + z$ ; (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ]. H atoms have been omitted for clarity.

benzene ring (C7–C12) is  $177.36(9)^\circ$ . The angle  $\Theta_3$  between the planes of the two aromatic rings is  $10.13(8)^\circ$ . All the C–C bond distances in the benzene rings have typical  $Csp^2$ – $Csp^2$  values. The average C–C bond distances within these two rings are  $1.374(2) \text{ \AA}$  and  $1.415(2) \text{ \AA}$ . The N1–C1 and N2–C7 bond lengths [ $1.4213(18) \text{ \AA}$  and  $1.4158(19) \text{ \AA}$ , respectively] and the N=N bond length [ $1.247(2) \text{ \AA}$ ] are comparable to those found for similar compounds (Şahin *et al.*, 2005).

The crystal packing in (I) is governed by intramolecular and  $\pi$ - $\pi$  (Fig. 2) interactions. An intermolecular  $\pi$ - $\pi$  contact occurs between  $CgA$ – $CgB$  and  $CgB$ – $CgC$ , where  $A$  is the ring (C7–C12),  $B$  ring (C1–C6)<sup>i</sup> [symmetry code: (i)  $x, y, 1 + z$ ] and  $C$  ring (C7–C12)<sup>ii</sup> [symmetry code: (ii)  $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$ ]. The perpendicular distance from  $A$  to  $B$  is  $3.390 \text{ \AA}$  and  $B$  to  $C$  is  $3.343 \text{ \AA}$ . Compound (I) also has an intramolecular O–H...O hydrogen bond (Table 2).

## Experimental

A mixture of 2-fluoroaniline (0.98 g, 8.8 mmol), water (50 ml) and concentrated hydrochloric acid (2.2 ml, 26.4 mmol) was heated with stirring until a clear solution was obtained. This solution was cooled to 273–278 K, and a solution of sodium nitrite (0.85 g, 12.3 mmol) in water was added dropwise while the temperature was maintained below 278 K. The resulting mixture was stirred for 30 min in an ice bath. An *o*-vanillin (1.19 g, 8.8 mmol) solution (pH 9) was added gradually to the solution of cooled 2-fluorobenzenediazonium chloride, prepared as described above, and the resulting mixture was stirred at 273–278 K for 60 min in an ice bath. The product was recrystallized from glacial acetic acid to obtain solid (*E*)-5-[(2-fluorophenyl)diazenyl]-2-hydroxy-3-methoxybenzaldehyde. The

product, (I), was recrystallized from acetic acid (yield 82%; m.p. 441–442 K).

### Crystal data

$C_{14}H_{11}FN_2O_3$   
 $M_r = 274.25$   
Monoclinic,  $Cc$   
 $a = 10.7956(8) \text{ \AA}$   
 $b = 13.7521(13) \text{ \AA}$   
 $c = 8.3078(6) \text{ \AA}$   
 $\beta = 92.274(6)^\circ$   
 $V = 1232.42(17) \text{ \AA}^3$   
 $Z = 4$

$D_x = 1.478 \text{ Mg m}^{-3}$   
Mo  $K\alpha$  radiation  
Cell parameters from 8358 reflections  
 $\theta = 2.4$ – $28.0^\circ$   
 $\mu = 0.12 \text{ mm}^{-1}$   
 $T = 296 \text{ K}$   
Prism, brown  
 $0.50 \times 0.45 \times 0.38 \text{ mm}$

### Data collection

Stoe IPDS-2 diffractometer  
 $\omega$  scans  
Absorption correction: integration  
(*X-RED32*; Stoe & Cie, 2002)  
 $T_{\min} = 0.939$ ,  $T_{\max} = 0.967$   
8358 measured reflections  
1456 independent reflections

1399 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.151$   
 $\theta_{\max} = 27.9^\circ$   
 $h = -14 \rightarrow 14$   
 $k = -18 \rightarrow 17$   
 $l = -10 \rightarrow 10$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.030$   
 $wR(F^2) = 0.083$   
 $S = 1.06$   
1456 reflections  
182 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.0699P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\max} < 0.001$   
 $\Delta\rho_{\max} = 0.15 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\min} = -0.18 \text{ e \AA}^{-3}$

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

C2–F1	1.342 (2)	C10–O2	1.3385 (19)
C1–N1	1.4213 (18)	C13–O3	1.219 (3)
C7–N2	1.4158 (19)	N1–N2	1.247 (2)
O3–C13–C9	124.7 (2)		
C13–C9–C10–O2	−0.9 (3)	C1–N1–N2–C7	177.97 (13)

**Table 2**

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D$ –H... $A$	$D$ –H	H... $A$	$D$ ... $A$	$D$ –H... $A$
O2–H2A...O3	0.82	1.91	2.629 (2)	146

The crystal of (I) used for data collection was not of good quality, hence the high value of  $R_{\text{int}}$  above. In the absence of significant anomalous scattering effects, Friedel pairs were merged before the final refinement. All H-atoms bound to carbon were refined using a riding model with C–H =  $0.93 \text{ \AA}$  [ $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent atom})$ ] for aromatic carbon and C–H =  $0.96 \text{ \AA}$  [ $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{parent atom})$ ] for methyl carbon atoms. The H atom of the hydroxyl O atom was refined with O–H =  $0.82 \text{ \AA}$  [ $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$ ].

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA* (Stoe & Cie, 2002); 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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