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4-(4-Hydroxyphenyldiazenyl)-benzonitrile

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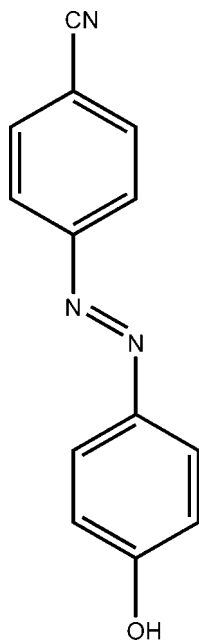
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Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å;
R factor = 0.054; wR factor = 0.145; data-to-parameter ratio = 8.3.

The molecule of the title compound, $\text{C}_{13}\text{H}_9\text{N}_3\text{O}$, is achiral but forms a chiral arrangement in the crystal structure. The molecule adopts an *E* configuration with respect to the $\text{N}=\text{N}$ bond and is almost planar, with an r.m.s. deviation of 0.0439 Å from the plane through all atoms in the molecule. The dihedral angle between the two benzene rings is $2.2(2)^\circ$. In the crystal structure, intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonding generates a chain.

Related literature

For the preparation of tetrazole derivatives from nitrile compounds, see: Dunica *et al.* (1991); Wittenberger & Donner (1993). For the general chemistry of tetrazole compounds, see: Xiong *et al.* (2002). For a similar structure, see: Harada *et al.* (1997).



Experimental

Crystal data

$\text{C}_{13}\text{H}_9\text{N}_3\text{O}$
 $M_r = 223.23$
Monoclinic, *Cc*
 $a = 6.5307(13)$ Å
 $b = 10.747(2)$ Å
 $c = 15.851(3)$ Å
 $\beta = 93.54(3)^\circ$
 $V = 1110.4(4)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 293(2)$ K
 $0.18 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2 diffractometer
Absorption correction: multi-scan
(*CrystalClear*; Rigaku, 2005)
 $T_{\min} = 0.781$, $T_{\max} = 1$
(expected range = 0.778–0.996)
5599 measured reflections
2532 independent reflections
1296 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.073$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.145$
 $S = 0.97$
1278 reflections
154 parameters
2 restraints
H-atom parameters constrained
 $\Delta\rho_{\max} = 0.17$ e Å⁻³
 $\Delta\rho_{\min} = -0.19$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O1}-\text{H1A}\cdots\text{N1}^i$	0.85	2.01	2.817 (5)	159

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

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KP2153).

References

- Dunica, J. V., Pierce, M. E. & Santella, J. B. III (1991). *J. Org. Chem.*, **56**, 2395–2400.
Harada, J., Ogawa, K. & Tomoda, S. (1997). *Acta Cryst.* **B53**, 662–672.
Rigaku (2005). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
Wittenberger, S. J. & Donner, B. G. (1993). *J. Org. Chem.*, **58**, 4139–4141.
Xiong, R.-G., Xue, X., Zhao, H., You, X.-Z., Abrahams, B. F. & Xue, Z.-L. (2002). *Angew. Chem. Int. Ed.* **41**, 3800–3803.

supporting information

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4-(4-Hydroxyphenyldiazenyl)benzonitrile

Chao Zhi Zhang

S1. Comment

Nitrile compounds are the precursor of tetrazole derivatives (Dunica, *et al.*, 1991; Wittenberger, *et al.*, 1993) and can be used for the design of noncentrosymmetric bulk materials (Xiong, *et al.*(2002)). We report here the crystal structure of the title compound, 4-(4-hydroxyphenylazo)benzonitrile, (I) (Fig. 1).

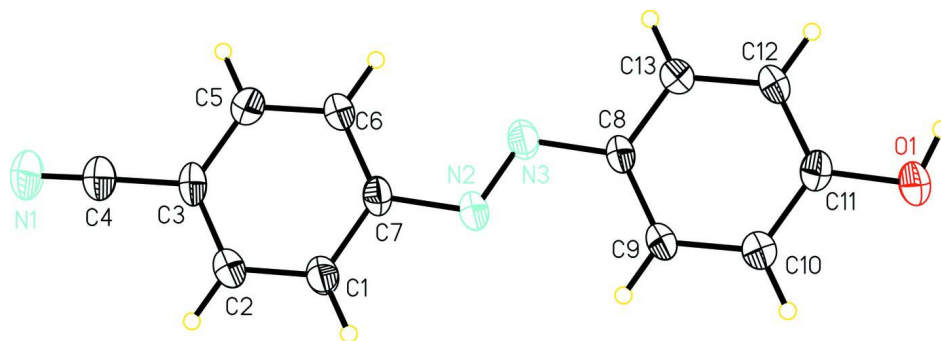
In I, the N=N double bond [1.257 (4) Å] is in the range found in other similar azo complexes with a *trans*-configuration (Harada, *et al.*, 1997). The torsion angle C7 - N2 - N3 - C8 is -179.30 (0.32)°. The dihedral angle between the two benzene rings is 2.18 (1/5)°. The crystal structure involves O—H···N hydrogen bond resulting in the formation of a chain.

S2. Experimental

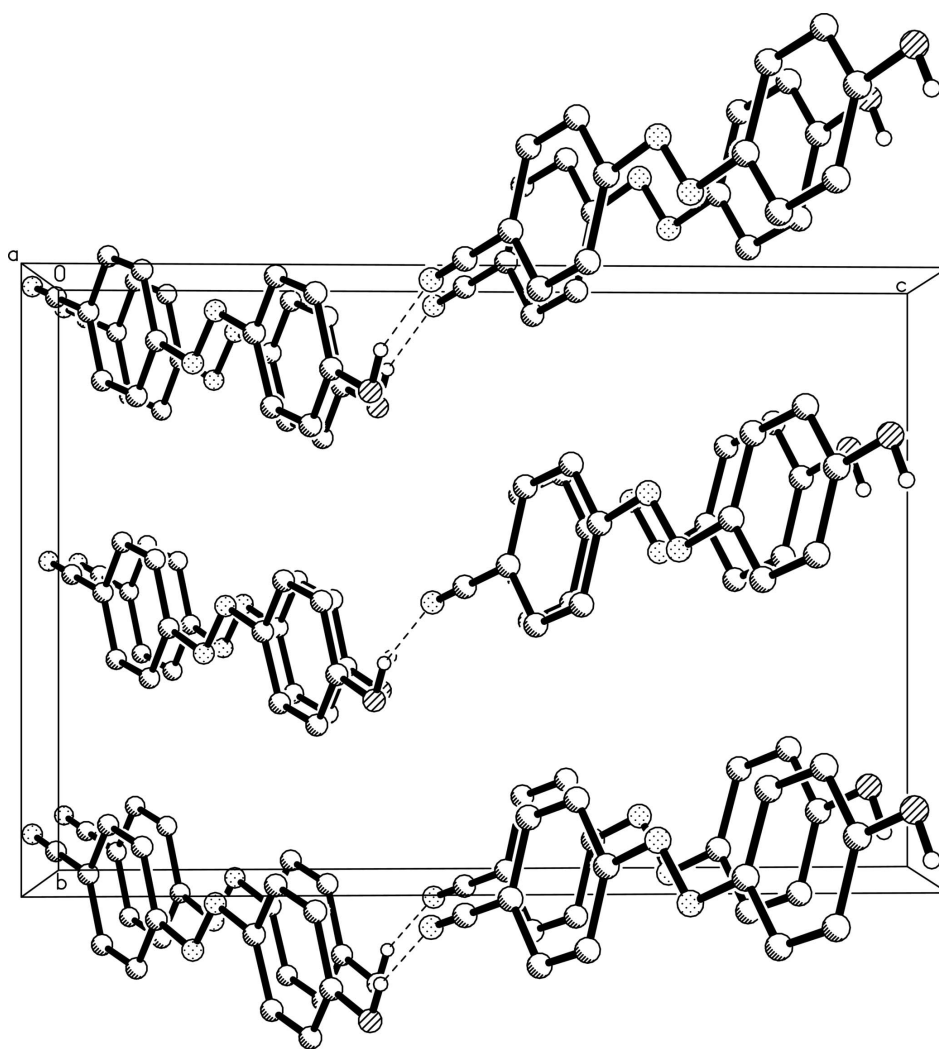
A solution of 4-cyanoaniline (0.71 g, 6 mmol) in a solution of hydrochloric acid (6 ml, 4M) was added to a solution of sodium nitrite (0.42 g, 6.1 mmol) in 2 ml water, and the mixture was stirred for 4 h under N₂ atmosphere at 273–278 K. Then urea (0.01 g, 0.2 mmol) was added to decompose excessive nitrous acid, and the mixture was further stirred for 30 min. The solution of the diazonium salt was added to a aqueous phenol (0.62 g, 6.6 mmol), sodium carbonate (3 g), baking soda (0.2 g) and ice (15 g) at 273–278 K. The mixture was stirred for 7 h. After the reaction solution was neutralized with a solution of hydrochloric acid (13.6 ml, 3 M), the mixture was filtrated. A yellow block-like crystals (1.27 g, 5.7 mmol, 95%), which is suitable for X-ray analysis, were obtained by recrystallization from ethyl acetate (18 ml).

S3. Refinement

Positional parameters of all the H atoms were calculated geometrically and were allowed to ride on the C atoms to which they are bonded, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, or $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{O})$. In the absence of significant anomalous scattering effects the Friedel pairs were merged.

**Figure 1**

A view of the title compound with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

**Figure 2**

The crystal packing of the title compound viewed along the *a* axis. Hydrogen atoms not involved in hydrogen bonding (dashed lines) were omitted for clarity.

4-(4-Hydroxyphenyldiazenyl)benzonitrile

Crystal data

C₁₃H₉N₃O $M_r = 223.23$ Monoclinic, *Cc*

Hall symbol: C -2yc

 $a = 6.5307$ (13) Å $b = 10.747$ (2) Å $c = 15.851$ (3) Å $\beta = 93.54$ (3)° $V = 1110.4$ (4) Å³ $Z = 4$ $F(000) = 464$ $D_x = 1.335$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4352 reflections

 $\theta = 3.7$ – 27.7 ° $\mu = 0.09$ mm⁻¹ $T = 293$ K

BLOCK, yellow

 $0.18 \times 0.05 \times 0.05$ mm

Data collection

Rigaku Mercury2

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 13.6612 pixels mm⁻¹ ω scans

Absorption correction: multi-scan

(CrystalClear; Rigaku, 2005)

 $T_{\min} = 0.781$, $T_{\max} = 1$

5599 measured reflections

2532 independent reflections

1296 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.073$ $\theta_{\max} = 27.5$ °, $\theta_{\min} = 3.7$ ° $h = -8 \rightarrow 8$ $k = -13 \rightarrow 13$ $l = -20 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.053$ $wR(F^2) = 0.145$ $S = 0.98$

1278 reflections

154 parameters

2 restraints

Primary atom site location: structure-invariant

direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

 $w = 1/[\sigma^2(F_o^2) + (0.0762P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} < 0.001$ $\Delta\rho_{\max} = 0.17$ e Å⁻³ $\Delta\rho_{\min} = -0.19$ e Å⁻³

Absolute structure: indeterminate

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N3	0.2824 (5)	0.0455 (4)	0.1896 (2)	0.0497 (10)
N2	0.1650 (5)	0.1302 (4)	0.1608 (2)	0.0526 (11)
C8	0.4581 (6)	0.0892 (5)	0.2376 (3)	0.0447 (11)

O1	0.9855 (5)	0.1997 (4)	0.3729 (2)	0.0717 (11)
H1A	1.0618	0.1372	0.3848	0.108*
C3	-0.3689 (6)	0.0230 (5)	0.0211 (3)	0.0493 (12)
C7	-0.0130 (6)	0.0862 (4)	0.1136 (3)	0.0457 (11)
C12	0.7767 (7)	0.0358 (5)	0.3110 (3)	0.0502 (11)
H12A	0.8709	-0.0242	0.3303	0.060*
C4	-0.5570 (7)	-0.0106 (5)	-0.0251 (3)	0.0572 (13)
C6	-0.0538 (7)	-0.0375 (5)	0.0957 (3)	0.0543 (13)
H6A	0.0385	-0.0988	0.1146	0.065*
C2	-0.3273 (7)	0.1461 (5)	0.0391 (3)	0.0583 (14)
H2B	-0.4188	0.2075	0.0197	0.070*
C5	-0.2315 (6)	-0.0695 (5)	0.0497 (3)	0.0565 (14)
H5A	-0.2600	-0.1526	0.0377	0.068*
C9	0.4922 (6)	0.2126 (4)	0.2580 (3)	0.0577 (13)
H9A	0.3959	0.2725	0.2407	0.069*
C11	0.8129 (7)	0.1589 (4)	0.3289 (3)	0.0513 (12)
C13	0.6011 (7)	0.0010 (5)	0.2644 (3)	0.0519 (13)
H13A	0.5788	-0.0823	0.2511	0.062*
C10	0.6682 (7)	0.2471 (5)	0.3040 (3)	0.0622 (15)
H10A	0.6898	0.3302	0.3183	0.075*
N1	-0.7085 (7)	-0.0361 (5)	-0.0604 (3)	0.0712 (12)
C1	-0.1502 (7)	0.1783 (5)	0.0860 (3)	0.0580 (14)
H1B	-0.1228	0.2613	0.0990	0.070*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N3	0.041 (2)	0.055 (3)	0.051 (2)	0.000 (2)	-0.0098 (18)	0.0033 (19)
N2	0.043 (2)	0.054 (3)	0.059 (3)	0.002 (2)	-0.0175 (19)	0.001 (2)
C8	0.031 (2)	0.051 (3)	0.051 (3)	0.0005 (19)	-0.008 (2)	0.000 (2)
O1	0.055 (2)	0.071 (3)	0.084 (3)	0.0087 (18)	-0.0356 (19)	-0.014 (2)
C3	0.035 (2)	0.070 (4)	0.041 (2)	-0.005 (2)	-0.0080 (19)	0.000 (2)
C7	0.042 (3)	0.055 (3)	0.039 (3)	-0.002 (2)	-0.010 (2)	0.003 (2)
C12	0.039 (3)	0.055 (3)	0.055 (3)	0.008 (2)	-0.012 (2)	0.003 (2)
C4	0.046 (3)	0.072 (4)	0.052 (3)	-0.006 (3)	-0.006 (2)	-0.001 (2)
C6	0.045 (3)	0.053 (3)	0.063 (3)	0.007 (2)	-0.017 (2)	-0.004 (3)
C2	0.054 (3)	0.057 (3)	0.061 (3)	0.006 (2)	-0.019 (2)	0.003 (2)
C5	0.053 (3)	0.054 (3)	0.060 (3)	0.000 (2)	-0.009 (2)	-0.012 (2)
C9	0.045 (3)	0.049 (3)	0.076 (4)	0.007 (2)	-0.023 (2)	0.001 (3)
C11	0.046 (3)	0.057 (3)	0.049 (3)	-0.003 (2)	-0.010 (2)	-0.006 (2)
C13	0.045 (3)	0.049 (3)	0.061 (3)	0.008 (2)	-0.007 (2)	-0.007 (2)
C10	0.055 (3)	0.050 (3)	0.078 (4)	0.005 (2)	-0.021 (3)	-0.004 (3)
N1	0.048 (2)	0.091 (3)	0.072 (3)	-0.013 (2)	-0.017 (2)	-0.007 (2)
C1	0.051 (3)	0.049 (3)	0.071 (4)	-0.004 (2)	-0.021 (3)	0.008 (2)

Geometric parameters (Å, °)

N3—N2	1.258 (4)	C12—H12A	0.9300
N3—C8	1.417 (5)	C4—N1	1.140 (6)
N2—C7	1.424 (5)	C6—C5	1.375 (7)
C8—C13	1.380 (6)	C6—H6A	0.9300
C8—C9	1.380 (6)	C2—C1	1.380 (6)
O1—C11	1.361 (5)	C2—H2B	0.9300
O1—H1A	0.8501	C5—H5A	0.9300
C3—C2	1.377 (7)	C9—C10	1.373 (6)
C3—C5	1.395 (7)	C9—H9A	0.9300
C3—C4	1.437 (6)	C11—C10	1.379 (7)
C7—C6	1.382 (6)	C13—H13A	0.9300
C7—C1	1.388 (6)	C10—H10A	0.9300
C12—C11	1.371 (7)	C1—H1B	0.9300
C12—C13	1.377 (7)		
N2—N3—C8	114.2 (3)	C3—C2—H2B	120.0
N3—N2—C7	114.2 (3)	C1—C2—H2B	120.0
C13—C8—C9	119.4 (4)	C6—C5—C3	119.9 (4)
C13—C8—N3	116.6 (4)	C6—C5—H5A	120.0
C9—C8—N3	124.0 (4)	C3—C5—H5A	120.0
C11—O1—H1A	108.4	C10—C9—C8	120.1 (4)
C2—C3—C5	120.1 (4)	C10—C9—H9A	119.9
C2—C3—C4	119.9 (4)	C8—C9—H9A	119.9
C5—C3—C4	119.9 (5)	O1—C11—C12	122.9 (4)
C6—C7—C1	120.6 (4)	O1—C11—C10	117.2 (4)
C6—C7—N2	124.6 (4)	C12—C11—C10	119.9 (4)
C1—C7—N2	114.8 (4)	C12—C13—C8	120.3 (5)
C11—C12—C13	120.0 (4)	C12—C13—H13A	119.8
C11—C12—H12A	120.0	C8—C13—H13A	119.8
C13—C12—H12A	120.0	C9—C10—C11	120.2 (4)
N1—C4—C3	178.5 (6)	C9—C10—H10A	119.9
C5—C6—C7	119.7 (4)	C11—C10—H10A	119.9
C5—C6—H6A	120.2	C2—C1—C7	119.6 (5)
C7—C6—H6A	120.2	C2—C1—H1B	120.2
C3—C2—C1	120.0 (4)	C7—C1—H1B	120.2
C7—N2—N3—C8	-179.2 (4)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1A \cdots N1 ⁱ	0.85	2.01	2.817 (5)	159

Symmetry code: (i) $x+2, -y, z+1/2$.