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## Key indicators

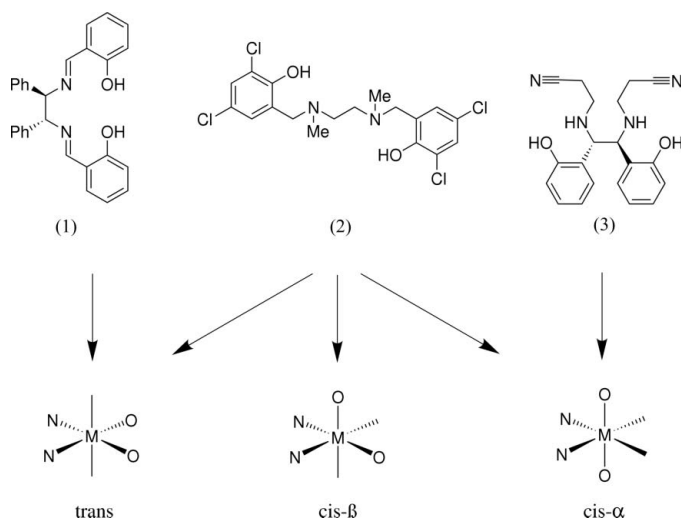
Single-crystal X-ray study  
 $T = 150\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$   
 $R$  factor = 0.039  
 $wR$  factor = 0.096  
Data-to-parameter ratio = 9.9For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.*(S,S)*-*N,N'*-Dicyanoethyl-1,2-(2-hydroxyphenyl)-  
ethylenediamine

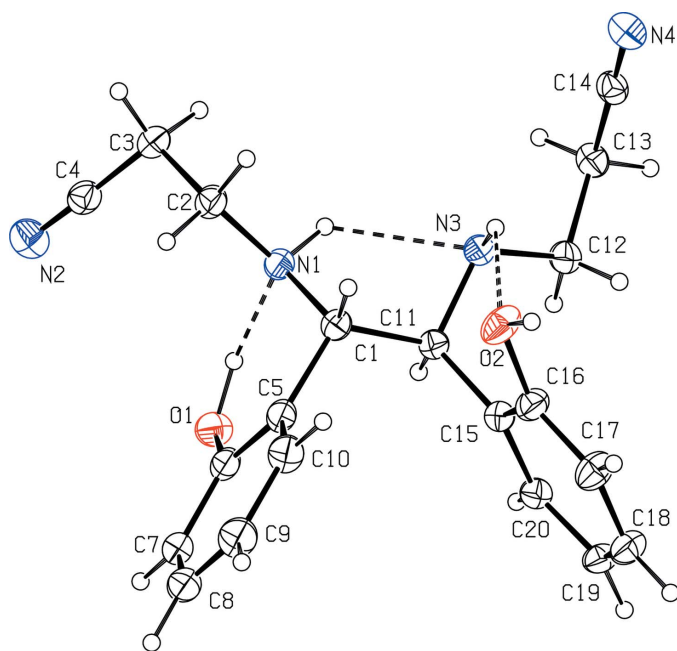
In the title molecule {systematic name: 3,3'-[1,2-bis(2-hydroxyphenyl)ethane-1,2-diyl-diimino]propanenitrile},  $\text{C}_{20}\text{H}_{22}\text{N}_4\text{O}_2$ , there are three different types of intramolecular hydrogen bonds, one of each of the types  $\text{N}-\text{H}\cdots\text{O}$  [ $\text{H}\cdots\text{O} = 2.51(3)\text{ \AA}$ ],  $\text{N}-\text{H}\cdots\text{N}$  [ $\text{H}\cdots\text{N} = 2.21(3)\text{ \AA}$ ] and  $\text{O}-\text{H}\cdots\text{N}$  [ $\text{H}\cdots\text{O} = 1.71(3)\text{ \AA}$ ]. In the crystal structure, molecules are linked into one-dimensional chains in the  $a$ -axis direction *via* intermolecular  $\text{O}-\text{H}\cdots\text{N}$  [ $\text{H}\cdots\text{N} = 1.95(4)\text{ \AA}$ ] hydrogen bonds.

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## Comment

There has been much interest in  $C_2$ -symmetric  $\text{N}_2\text{O}_2$  ligands and their metal complexes. Metal complexes based on the chiral ligand bis(salicylidene)-1,2-diaminoethane, (1), have been used to catalyze a wide variety of organic reactions stereoselectively (Larrow & Jacobsen, 2004). Other  $C_2$ -symmetric  $\text{N}_2\text{O}_2$  ligands with electron-withdrawing groups, (2), have recently been used as Ziegler–Natta catalysts in the synthesis of polyethylenes (Mason & Coates, 2004; Segal *et al.*, 2005). However, compound (2) can, in principle, form three different isomers of octahedral complexes (*cis- $\alpha$* , *cis- $\beta$*  and *trans*). More desirable are ligands which can form highly rigid single-isomer metal complexes. These ligands are particularly attractive for the development of catalysts and the investigation of stereoselective recognition of bidentate substrates. Here, we report the crystal structure of the title chiral  $C_2$ -symmetric  $\text{N}_2\text{O}_2$  ligand, (3), which has two electron-withdrawing groups attached.





**Figure 1**  
The structure of (3), showing 30% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

the octahedral complex is possible upon deprotonation of the phenoxy group. A view of (3) is shown in Fig. 1 and selected torsion angles, describing the different orientations of the two dicyanoethyl groups, are given in Table 1.

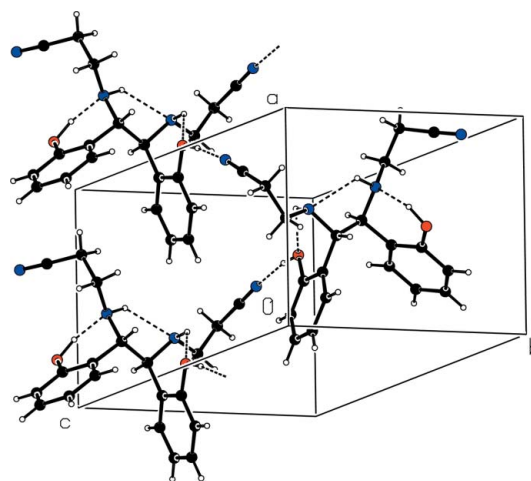
In the molecule of (3), there are three intramolecular hydrogen bonds (Table 2), forming one *S*(5) and two *S*(6) rings (Bernstein *et al.*, 1995). In the crystal structure, molecules related by  $2_1$  screw axes are linked *via* O—H...N hydrogen bonds into one-dimensional *C*(10) chains in the *a*-axis direction (Fig. 2). These chains are further linked by weak C—H...O hydrogen bonds to form a three-dimensional network.

## Experimental

1,2-(2-Hydroxyphenyl)-1,2-ethylenediamine was synthesized according to the literature procedure of Müller *et al.* (1989). Acrylonitrile (0.67 ml, 10.2 mmol) was added to a solution of (*S,S*)-1,2-(2-hydroxyphenyl)-1,2-ethylenediamine (1 g, 4.1 mmol) dissolved in methanol (14 ml). The mixture was stirred for 48 h at room temperature. After removal of the solvent, diethyl ether was added and the resulting solution was allowed to stand at room temperature for 3 d to give yellow crystals of (3). Spectroscopic analysis:  $^1\text{H NMR}$  (400 MHz,  $\text{CDCl}_3$ ,  $\delta$ , p.p.m.): 2.50 (*m*, 4H), 2.84 (*m*, 4H), 3.99 (*s*, 2H), 6.74 (*t*, 2H), 6.81 (*d*, 2H), 6.87 (*d*, 2H), 7.18 (*t*, 2H).

### Crystal data

$\text{C}_{20}\text{H}_{22}\text{N}_4\text{O}_2$	Mo $K\alpha$ radiation
$M_r = 350.42$	Cell parameters from 16844 reflections
Orthorhombic, $P2_12_12_1$	$\theta = 2.6\text{--}27.5^\circ$
$a = 8.4115$ (3) Å	$\mu = 0.08\text{ mm}^{-1}$
$b = 9.6525$ (3) Å	$T = 150$ (1) K
$c = 23.3886$ (8) Å	Plate, colourless
$V = 1898.97$ (11) Å <sup>3</sup>	$0.22 \times 0.20 \times 0.02\text{ mm}$
$Z = 4$	
$D_x = 1.226\text{ Mg m}^{-3}$	



**Figure 2**  
The crystal packing of (3), showing hydrogen bonds as dashed lines.

### Data collection

Nonius KappaCCD diffractometer	1958 reflections with $I > 2\sigma(I)$
$\varphi$ scans, and $\omega$ scans with $\kappa$ offsets	$R_{\text{int}} = 0.070$
Absorption correction: multi-scan (SORTAV; Blessing, 1995)	$\theta_{\text{max}} = 27.5^\circ$
$T_{\text{min}} = 0.723$ , $T_{\text{max}} = 0.999$	$h = -9 \rightarrow 10$
16844 measured reflections	$k = -11 \rightarrow 12$
2492 independent reflections	$l = -30 \rightarrow 30$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.1038P]$
$R[F^2 > 2\sigma(F^2)] = 0.039$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.14\text{ e \AA}^{-3}$
2492 reflections	$\Delta\rho_{\text{min}} = -0.17\text{ e \AA}^{-3}$
252 parameters	Extinction correction: <i>SHELXTL/PC</i> (Sheldrick, 2001)
H atoms treated by a mixture of independent and constrained refinement	Extinction coefficient: 0.025 (3)

**Table 1**

Selected torsion angles ( $^\circ$ ).

C1—N1—C2—C3	−179.39 (17)	C11—N3—C12—C13	162.36 (18)
N1—C2—C3—C4	70.5 (2)	N3—C12—C13—C14	60.0 (3)
C5—C1—C11—N3	−178.44 (16)		

**Table 2**

Hydrogen-bond geometry (Å,  $^\circ$ ).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1—H1O...N1	1.00 (3)	1.71 (3)	2.644 (2)	154 (3)
O2—H2O...N4 <sup>i</sup>	0.93 (4)	1.95 (4)	2.883 (3)	173 (3)
N1—H1N...N3	0.93 (3)	2.21 (3)	2.787 (3)	119 (2)
N3—H3N...O2	0.91 (3)	2.51 (3)	3.133 (3)	126 (2)
C2—H2B...N4 <sup>ii</sup>	0.99	2.60	3.489 (3)	149
C12—H12A...O1 <sup>iii</sup>	0.99	2.48	3.450 (3)	167
C17—H17A...N4 <sup>i</sup>	0.95	2.56	3.308 (3)	135

Symmetry codes: (i)  $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 1$ ; (ii)  $x, y + 1, z$ ; (iii)  $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$ .

H atoms bonded to N and O atoms were located in a difference map and refined isotropically. H atoms bonded to C atoms were

placed in calculated positions, with C—H distances ranging from 0.95 to 1.00 Å. They were included in the refinement in the riding-model approximation, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ . In the absence of significant anomalous dispersion effects, Friedel pairs were merged before the refinement. The enantiomer was assigned from the known absolute configuration of the starting material.

Data collection: *COLLECT* (Nonius, 2003); cell refinement: *DENZO-SMN* (Otwinowski & Minor, 1997); data reduction: *DENZO-SMN*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXTL/PC* (Sheldrick, 2001); molecular graphics: *SHELXTL/PC* and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXTL/PC*.

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