Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

4,4′-(*p*-Phenylenediimino)dipent-3-en-2-one

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.101
Data-to-parameter ratio = 16.6

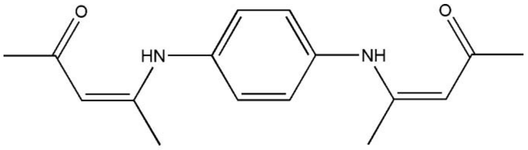
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The molecule of the title Schiff base compound, $C_{16}H_{20}N_2O_2$, lies on a crystallographic twofold rotation axis. The crystal structure is stabilized by weaker intermolecular C—H···O and stronger intramolecular N—H···O hydrogen bonds.

Received 9 March 2007
Accepted 20 March 2007

Comment

The title compound, (I), has been reported to be a good polydentate ligand which can be a building block for larger molecules and supramolecular assemblies and coordinate with various metals with different properties (Shauib *et al.*, 2006). It can be obtained *via* two different methods in high yield (Shauib *et al.*, 2006). We obtained (I) in over 90% yield from the reaction of *p*-phenylenediamine with two equivalents of 2,4-pentanedione *via* a method slightly different from those in the literature (Shauib *et al.*, 2006) (see *Experimental*).



(I)

A crystallographic twofold rotation axis passes through the mid-points of the C7—C7A and C8—C8A bonds (Fig. 1). The title compound is generally believed to exist in three main forms: dione–diimine, dione–dienamine and dienol–diimine (Shauib *et al.*, 2006). The crystal structure indicates that (I) exists in the dione–dienamine form in the solid state, according to the bond lengths C2—O1 [1.2528 (15) Å], C2—C3 [1.4285 (17) Å], C3—C4 [1.3813 (17) Å] and C4—N1 [1.3548 (15) Å]. Atoms C2, C3, C4, O1 and N1 are nearly coplanar. The N1—C4—C3—C2 and O1—C2—C3—C4 torsion angles are 0.45 (19) and −2.9 (2)°, respectively. The dihedral angle between the benzene plane C6/C6a/C7/C7a/C8/C8a and the plane formed by atoms C2/C3/C4/O1/N1 is 45.9°.

The crystal structure (Fig. 2) is stabilized by weaker intermolecular C—H···O hydrogen bonds (the upper limit of the C···O distance in C—H···O hydrogen bonds is 4.0 Å; Desiraju, 1996; Steed & Atwood, 2000), forming an S-shaped sheet, and stronger intramolecular N—H···O hydrogen bonds (Table 1).

Experimental

A mixture of 2,4-pentanedione (5.00 ml, 48.9 mmol), *p*-phenylenediamine (2.64 g, 24.5 mmol) and catalytic concentrated HCl (1% mol ratio) was refluxed in absolute ethanol (20 ml). The crude product precipitated from the solution and was recrystallized from ethanol to give yellow crystals (yield 6.07 g, 91%).

Crystal data

$C_{16}H_{20}N_2O_2$
$M_r = 272.34$
Orthorhombic, *Pbcn*
$a = 10.675$ (2) Å
$b = 8.8373$ (18) Å
$c = 14.995$ (3) Å

$V = 1414.5$ (5) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 295$ (2) K
$0.1 \times 0.1 \times 0.1$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: none
12933 measured reflections

1610 independent reflections
1343 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.101$
$S = 1.06$
1610 reflections
97 parameters

H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C7—H7$\cdots$O1$^{\rm i}$	0.93	3.08	3.9803 (16)	163
N1—H1$\cdots$O1	0.920 (18)	1.913 (17)	2.6776 (14)	139.3 (15)

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

C-bound H atoms were positioned geometrically, with C—H = 0.93–0.96 Å, and allowed to ride on their parent atoms, with $U_{\rm iso}$(H) = 1.2 or $1.5U_{\rm eq}$(C). Atom H1 was located in a difference Fourier map and refined freely with an isotropic displacement parameter.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

We thank the National Natural Science Foundation of China (grant Nos. 20674024 and 20374023) for support.

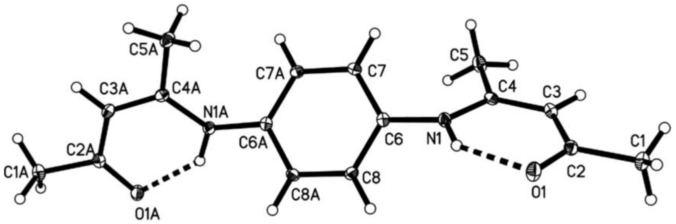


Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level and H atoms are represented by spheres of arbitrary radius. Intramolecular hydrogen bonds are indicated by dashed lines. [Symmetry code: (A) $1-x, y, \frac{3}{2}-z$.]

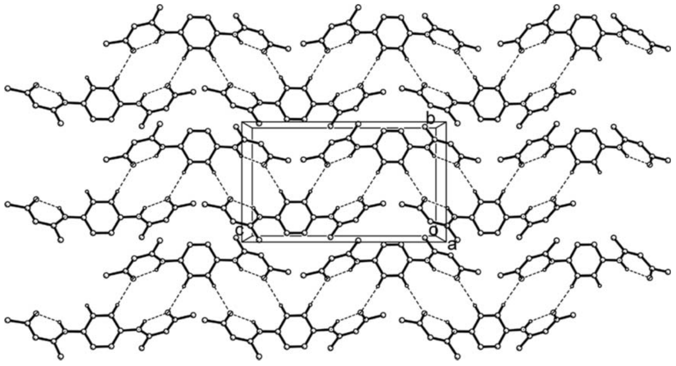


Figure 2
The packing of (I). Hydrogen bonds are indicated by dashed lines.

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Acta Cryst. (2007). E**63**, o2052–o2053 Su *et al.* • $C_{16}H_{20}N_2O_2$ **o2053**