

Yanhua Feng,^a Xianhong Yin,^{b†}
Lin Chen,^c Minyu Tan^{a*} and
Kaibei Yu^d

^aCollege of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China, ^bChemistry Department, Guangxi University for Nationalities, Nanning 530006, People's Republic of China, ^cOSSO, Lanzhou Institute of Chemical Physics Chinese Academy of Sciences, Lanzhou 730000, People's Republic of China, and ^dAnalysis Center, Chengdu Branch of Chinese Academy of Science, Chengdu 610041, People's Republic of China

† Current address: College of Chemistry and Chemical Engineering, Lanzhou University, Lanzhou 730000, People's Republic of China

Correspondence e-mail:
eagletiger@vip.sina.com

Key indicators

Single-crystal X-ray study

$T = 296\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

R factor = 0.038

wR factor = 0.106

Data-to-parameter ratio = 14.1

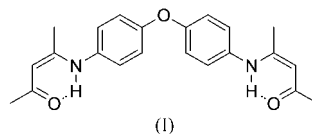
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

(Z)-4-(4-{4-[(Z)-1-Methyl-3-oxobut-1-enylamino]-phenoxy}phenylamino)pent-3-en-2-one

In the structure of the title compound, $\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_3$, short intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts influence the crystal packing and molecular ladders are formed. Intramolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds make the *Z* configuration stable by formation of six-membered rings.

Comment

Schiff bases are becoming increasingly important as biological, analytical, pharmacological and antimicrobial reagents in coordination chemistry. There are many reports of Schiff bases obtained by condensation between the NH_2 group of an amine and the carbonyl group of aldehydes/ketones (Ismail, 2000; Raman *et al.*, 2001; Daniel Thangadurai & Natarajan, 2000). A literature survey revealed fewer results involving isomers of Schiff bases. We obtained the title compound, (I), by the usual type of condensation; however, the expected $\text{C}=\text{C}=\text{N}$ group is, in fact, $\text{C}=\text{C}-\text{N}$.



A view of the molecule of (I) is shown in Fig. 1. The dihedral angle between the two planes defined by O1 and each of the two phenyl rings is $73.39(7)^\circ$. The $\text{N1}-\text{H1N}\cdots\text{O2}$ and $\text{N2}-\text{H2N}\cdots\text{O3}$ hydrogen bonds stabilize the *Z* configuration of the $\text{C}=\text{C}$ bonds by forming six-membered rings (Table 2). The torsion angles $\text{C7}-\text{C9}-\text{C10}-\text{O2}$ [$1.3(3)^\circ$] and $\text{C7}-\text{C9}-\text{C10}-\text{C11}$ [$-177.81(19)^\circ$] indicate that the group $\text{N1}/\text{C7}/\text{C8}/\text{C9}/\text{C10}/\text{C11}/\text{O2}$ is nearly planar; the group $\text{N2}/\text{C18}/\text{C19}/\text{C20}/\text{C21}/\text{C22}/\text{O3}$ is slightly more non-planar, as is evident from the torsion angles $\text{C18}-\text{C20}-\text{C21}-\text{O3}$ [$-2.3(3)^\circ$] and $\text{C18}-\text{C20}-\text{C21}-\text{C22}$ [$175.70(18)^\circ$]. The dihedral angle between these two groups is $88.21(6)^\circ$. The two halves of the molecule resemble each other in geometry (Table 1).

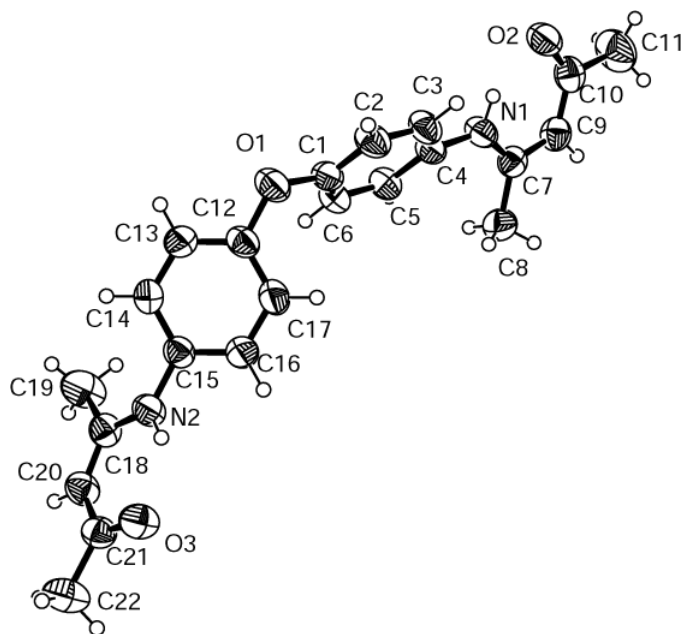
The short intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts (Table 2) involving both ketone functions are respectively 0.19, 0.18 and 0.12 \AA shorter than the sum of van der Waals radii (Bondi, 1964). They may be classified as weak to very weak hydrogen bonds (Desiraju & Steiner, 1999), and the second is near-linear (Jeffrey *et al.*, 1985). Examination of the structure with *PLATON* (Spek, 2002) shows that there are no solvent-accessible voids.

The intermolecular $\text{C}-\text{H}\cdots\text{O}$ contacts link molecules into dimers, dimers face-to-face to form four-molecule aggregates, and aggregates into ladders, to give a two-dimensional network (Fig. 2).

Received 27 August 2002

Accepted 23 September 2002

Online 27 September 2002

**Figure 1**

A view of the molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

Experimental

The title compound was prepared by the condensation in ethanol of 4,4'-diamino-diphenyl ether (0.5 mol) with pentane-2,4-dione (1.0 mol). The solution was stirred for 2 h at 433 K. Crystals suitable for diffraction study were obtained after one month (m.p. 428 K). Spectroscopic analysis, $^1\text{H NMR}$ (CDCl_3 , δ , p.p.m.): 1.97 (*s*, 6H), 2.10 (*s*, 6H), 5.19 (*s*, 1H), 7.04 (*m*, 8H), 12.39 (*s*, 1H).

Crystal data

$\text{C}_{22}\text{H}_{24}\text{N}_2\text{O}_3$
 $M_r = 364.43$
 Monoclinic, $P2_1/c$
 $a = 8.769$ (1) Å
 $b = 20.073$ (4) Å
 $c = 11.928$ (2) Å
 $\beta = 107.53$ (1)°
 $V = 2002.1$ (6) Å³
 $Z = 4$

$D_x = 1.209$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 26 reflections
 $\theta = 3.2\text{--}15.2^\circ$
 $\mu = 0.08$ mm⁻¹
 $T = 296$ (2) K
 Block, yellow
 $0.54 \times 0.52 \times 0.44$ mm

Data collection

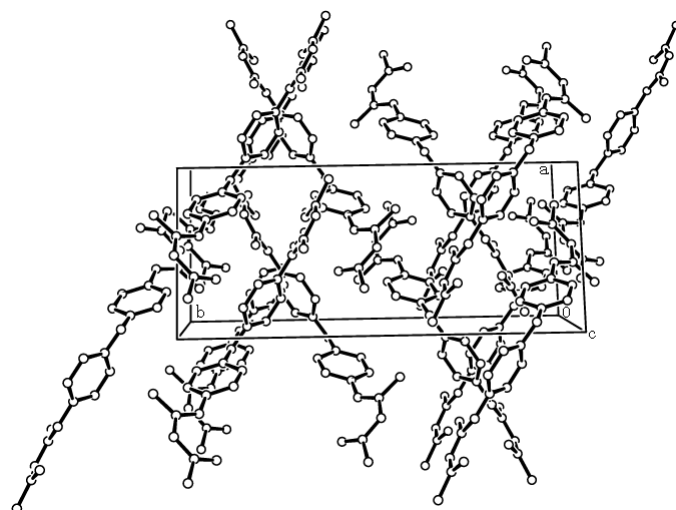
Siemens P4 diffractometer
 ω scans
 Absorption correction: none
 3762 measured reflections
 3520 independent reflections
 2338 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.010$

$\theta_{\text{max}} = 25.0^\circ$
 $h = 0 \rightarrow 10$
 $k = 0 \rightarrow 23$
 $l = -14 \rightarrow 13$
 3 standard reflections every 97 reflections
 intensity decay: 4.4%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.106$
 $S = 0.98$
 3520 reflections
 249 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.16$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³
 Extinction correction: *SHELXL97*
 Extinction coefficient: 0.0223 (19)

**Figure 2**

A packing diagram for (I). H atoms have been omitted for clarity.

Table 1

Selected geometric parameters (Å, °).

O1—C1	1.3911 (19)	N1—C4	1.417 (2)
O1—C12	1.3926 (19)	N2—C18	1.335 (2)
O2—C10	1.245 (2)	N2—C15	1.419 (2)
O3—C21	1.2406 (19)	C7—C9	1.368 (2)
N1—C7	1.339 (2)	C18—C20	1.373 (2)
C1—O1—C12	117.75 (12)	N1—C7—C8	119.99 (15)
C7—N1—C4	130.90 (14)	C14—C15—N2	123.45 (15)
C18—N2—C15	131.37 (14)	C16—C15—N2	117.30 (15)
C5—C4—N1	122.85 (15)	N2—C18—C20	119.66 (15)
C3—C4—N1	118.10 (14)	N2—C18—C19	119.41 (16)
N1—C7—C9	119.59 (15)	C4—N1—C7—C9	175.96 (17)
C15—N2—C18—C20	−178.18 (16)	N1—C7—C9—C10	−3.6 (3)
N2—C18—C20—C21	3.3 (3)	C7—C9—C10—O2	1.3 (3)
C18—C20—C21—O3	−2.3 (3)	C7—C9—C10—C11	−177.81 (19)
C18—C20—C21—C22	175.70 (18)		

Table 2

Contact distances (Å).

H8C···O2 ⁱ	2.54	H16···O2 ⁱⁱ	2.60
-----------------------	------	------------------------	------

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

Table 3

Hydrogen-bonding geometry (Å, °).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
N1—H1N···O2	0.86	1.94	2.643 (2)	139
N2—H2N···O3	0.86	1.92	2.637 (2)	139
C6—H6···O3 ⁱ	0.93	2.53	3.175 (2)	127

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

All H atoms were generated geometrically and refined using a riding model, with C—H = 0.93 Å for aromatic H atoms and 0.96 Å for methyl H atoms, N—H = 0.86 Å, and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}$ of the parent atoms.

Data collection: *XSCANS* (Fait, 1991); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997b); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *ORTEP-3* for Windows (Farrugia, 1997); software used to prepare material for publication: *SHELXTL*.

References

- Bondi, A. (1964). *J. Phys. Chem.* **68**, 441.
- Daniel Thangadurai, T. & Natarajan, K. (2000). *Transition Met. Chem.* **25**, 347–351.
- Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond*. Oxford University Press.
- Fait, J. (1991). *XSCANS*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Ismail, K. Z. (2000). *Transition Met. Chem.* **25**, 522–528.
- Jeffrey, G. A., Maluszynska, H. & Mitra, J. (1985). *Int. J. Biol. Macromol.* **7**, 336–348.
- Raman, N., Kulandaisamy, A. & Shunmugasundaram, A. (2001). *Transition Met. Chem.* **26**, 131–135.
- Sheldrick, G. M. (1990). *Acta Cryst. A* **46**, 467–473.
- Sheldrick, G. M. (1997a). *SHELXL97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Spek, A. L. (2002). *PLATON*. Utrecht University, The Netherlands.