Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-(2,6-Diisopropylphenylamino)-4-(2,6-diisopropylphenylimino)-2-pentene toluene hemisolvate at 150 K

Philip J. Bailey, Stephen T. Liddle* and Simon Parsons

Department of Chemistry, The University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

Correspondence e-mail:
pjb06@holyrood.ed.ac.uk

Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
Some non-H atoms missing
R factor = 0.060
wR factor = 0.141
Data-to-parameter ratio = 18.0

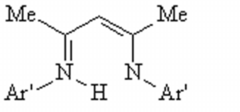
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 22 May 2001
Accepted 4 June 2001
Online 29 June 2001

The title compound 2-(2,6-diisopropylphenylamino)-4-(2,6-diisopropylphenylimino)pent-2-ene crystallizes as a toluene solvate, $C_{29}H_{42}N_2\cdot0.5C_7H_8$; the observed bond lengths are consistent with delocalization of the imino and alkene double bonds across the N—C—C—C—N backbone.

Comment

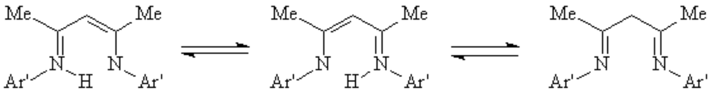
β-Diketimine ligands such as the title compound, (I), are proving to be versatile ligands in the monoanionic form for early transition metals in olefin polymerization (Budzelaar *et al.*, 1998). Recently, we have been interested in the application of (I) for olefin polymerization by nitrogen-ligated organomagnesium species (Bailey *et al.*, 2000). However, whilst there are now many examples of this ligand in the deprotonated monoanionc form as chelates, there are no examples to date of the free protonated parent ligand (I) itself. We report here the crystal structure of (I), which was synthesized *via* a standard procedure of acid-catalysed condensation of 2,4-pentanedione and 2,6-diisopropylaniline (Parks & Holm, 1968).



(I)

Ar' = 2,6-diisopropylphenyl

The crystal structure is composed of discrete molecules of (I) (Fig. 1) exhibiting no significant intermolecular contacts. The N—C [N1—C2 1.326 (2) and N2—C4 1.327 (2) Å] and C—C [C2—C3 1.398 (2) and C3—C4 1.401 (2) Å] bond lengths of the N—C—C—C—N backbone are in the range associated with aromatic N—C and C—C bonds (Allen *et al.*, 1992). Variation between the N—C (N1—C2 and N2—C4) and C—C (C2—C3 and C3—C4) bonds are statistically insignificant. Compound (I) thus crystallizes as a mixture of the two possible imine–enamine tautomers (see Scheme below); inspection of the difference map around C3 gives no evidence to suggest the presence of the third tautomer. As a consequence, the amine proton is disordered in 50:50%



Ar' = 2,6-diisopropylphenyl

DOI: 101107/S1600536801009163

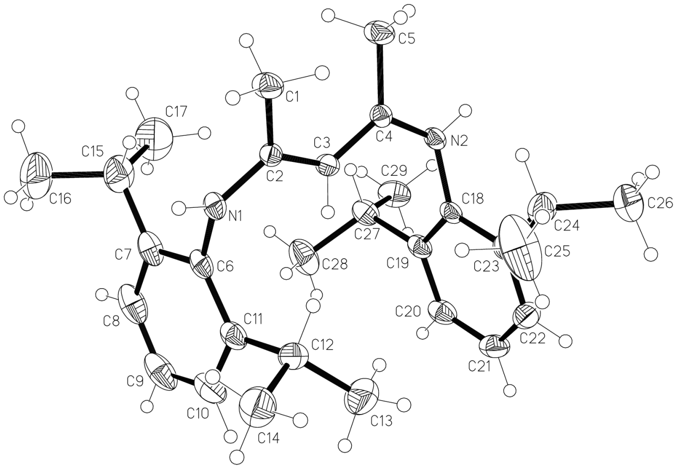


Figure 1
A view of (I) with the atom-numbering scheme. Displacement ellipsoids are at the 30% probability level.

occupancy over both N1 and N2 with bond lengths of 0.90 (3) and 0.89 (3) Å, respectively.

The two backbone methyl groups (C1 and C5) adopt a *syn* orientation; this is surprising as an *anti* conformation would lead to a less congested conformer. It is likely that small steric, electronic and entropic factors, or crystal-packing forces, are responsible for this. The two phenyl rings adopt a synperiplanar arrangement in order to minimize steric crowding.

Experimental

Compound (I) was synthesized by acid-catalysed condensation of 2,4-pentanedione and 2,6-diisopropylaniline in ethanol (Parks & Holm, 1968). Crystals were obtained from a saturated solution in toluene at 243 K.

Crystal data

$C_{29}H_{42}N_2{\cdot}0.5C_7H_8$
$M_r = 464.71$
Monoclinic, $C2/c$
$a = 26.655\ (5)$ Å
$b = 18.004\ (3)$ Å
$c = 12.623\ (2)$ Å
$\beta = 91.972\ (3)^\circ$
$V = 6054.0\ (19)$ Å^3
$Z = 8$

$D_x = 1.020$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 10829 reflections
$\theta = 2.2$–26.4°
$\mu = 0.06$ mm^{-1}
$T = 150\ (2)$ K
Block, colourless
0.19 × 0.16 × 0.07 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.989$, $T_{\max} = 0.996$
21 824 measured reflections

5322 independent reflections
4677 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.025$
$\theta_{\max} = 25.0^\circ$
$h = -31 \rightarrow 31$
$k = -21 \rightarrow 21$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.141$
$S = 1.14$
5322 reflections
296 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0507P)^2 + 4.1159P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °) for (section).

C1—C2	1.502 (2)	C4—N2	1.327 (2)
C2—N1	1.326 (2)	C4—C5	1.505 (2)
C2—C3	1.398 (2)	N1—C6	1.424 (2)
C3—C4	1.401 (2)	N2—C18	1.428 (2)
N1—C2—C3	121.92 (15)	N2—C4—C5	114.06 (13)
N1—C2—C1	114.12 (14)	C3—C4—C5	124.05 (14)
C3—C2—C1	123.89 (14)	C2—N1—C6	123.66 (13)
C2—C3—C4	129.70 (15)	C4—N2—C18	122.77 (13)
N2—C4—C3	121.84 (14)		

The toluene solvent molecule, located over a centre of inversion, was too highly disordered for individual atoms to be resolved; this was treated with the *SQUEEZE* procedure of *PLATON* (Spek, 1990). H atoms attached to C atoms were placed in calculated positions and subsequently allowed to ride on their parent atoms with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$. Methyl groups were treated as rotating rigid groups with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}({\rm C})$. H atoms attached to N atoms were located in a difference synthesis and were freely refined, each with 50% occupancy.

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

We thank the EPSRC for provision of a Bruker SMART APEX CCD Area Detector (SP) and for funding (STL).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1992). *International Tables for Crystallography*, Vol. C, edited by A. J. C. Wilson. Dordrecht: Kluwer Academic Publishers.

Bailey, P. J., Dick, C. M. E., Fabre, S. & Parsons, S. (2000). *J. Chem. Soc. Dalton Trans.* pp. 1655–1661.

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.

Budzelaar, P. H. M., B. van Oort, A. & Orpen, A. G. (1998). *Eur. J. Inorg. Chem.* pp. 1485–1494.

Parks, J. E. & Holm, R. H. (1968). *Inorg. Chem.* **7**, 1408–1416.

Sheldrick, G. M. (1997). *SADABS* and *SHELXTL* (Version 5.1). Bruker AXS Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.