Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2,2,6,6-Tetramethyl-3,5-bis(2,6-diisopropylphenylimino)heptane at 150 K

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Received 28 June 2001
Accepted 11 July 2001
Online 17 August 2001

Key indicators

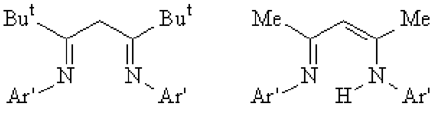
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.124
Data-to-parameter ratio = 16.6

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

The title compound, $C_{35}H_{54}N_2$, crystallizes as the E/Z and E/E isomers. There are two independent molecules in the crystal; the first occupies a general position, adopts a non-symmetrical conformation and represents the E/Z isomer with respect to the C═N bonds; the second exhibits exact C_2 symmetry (E/E isomer). The observed bond lengths and disposition of the H atoms in both molecules are consistent with a diimine. This contrasts with the related compound 2-(2,6-diisopropylphenylamino)-4-(2,6-diisopropylphenylimino)-2-pentene, which crystallizes as the imine–enamine tautomer.

Comment

Diimine ligands such as the title compound, (1), have attracted attention as useful and versatile sources of monoanionic ligands for early transition metals in olefin polymerization (Budzelaar *et al.*, 1998). Recently, we have been interested in the application of (1) for olefin polymerization by nitrogen ligated organomagnesium species (Bailey, Coxall *et al.*, 2001). However, whilst there are now a growing number of metal complexes involving this ligand in the deprotonated monoanionic form as chelates, there are no examples to date of the free protonated parent 1,3-diimine ligand (1) with the intact C—N═C(R)–CH_2–C(R)═N—C chain. Following on from our earlier report of the closely related β-diketimine compound (2) (Bailey, Liddle & Parsons, 2001), we report here the crystal structure of (1), which was synthesized *via* a coupling–elimination reaction of 1-chloro-1-(2,6-diisopropylphenylimino)-2,2-dimethylpropane and lithium-2-(2,6-diisopropylphenylimino)-3,3-dimethylbutane (Knorr & Weiss, 1982).

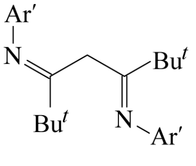


(1) (2)

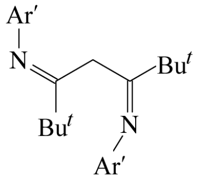
Ar' = 2,6-diisopropylphenyl

The crystal structure is composed of discrete molecules of (1) exhibiting no significant intermolecular contacts. There are two independent molecules in the crystal; the first occupies a general position and adopts a non-symmetrical conformation (Fig. 1); the second exhibits exact C_2 symmetry (Fig. 2). Examination of bond lengths and the disposition of the H atoms within the N—C—C—C—N backbones of both independent molecules (Table 1) reveals that (1) crystallizes as the diimine tautomer. This contrasts with (2), which crystallizes as the imine–enamine tautomer.

In the crystal under study, the two independent molecules exhibit different isomeric forms with respect to the C═N bonds; the non-symmetrical conformer adopts the *E*/*Z* form, whereas the C_2 conformer adopts the *E*/*E* form. In both independent molecules, the *tert*-butyl groups (C1, C8 and C36) adopt an *anti* orientation with respect to each other; this is clearly a result of the steric bulk of the *tert*-butyl groups, as the equivalent methyl groups in (2) adopt a *syn* conformation in the solid state.



(1)-*E/Z*



(1)-*E/E*

Experimental

Compound (1) was synthesized by a coupling reaction of 1-chloro-1-(2,6-diisopropylphenylimino)-2,2-dimethylpropane and 1-lithium-2-(2,6-diisopropylphenylimino)-3,3-dimethylbutane (Knorr & Weiss, 1982). Crystals were obtained from a saturated solution in hexane at 243 K.

Crystal data

$C_{35}H_{54}N_2$
$M_r = 502.80$
Monoclinic, $C2/c$
$a = 41.471$ (4) Å
$b = 9.3431$ (9) Å
$c = 24.989$ (3) Å
$\beta = 102.077$ (2)°
$V = 9468.0$ (16) Å^3
$Z = 12$

$D_x = 1.058$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 7323 reflections
$\theta = 2.2$–$26.3°$
$\mu = 0.06$ mm^{-1}
$T = 150$ (2) K
Block, colourless
0.42 × 0.26 × 0.19 mm

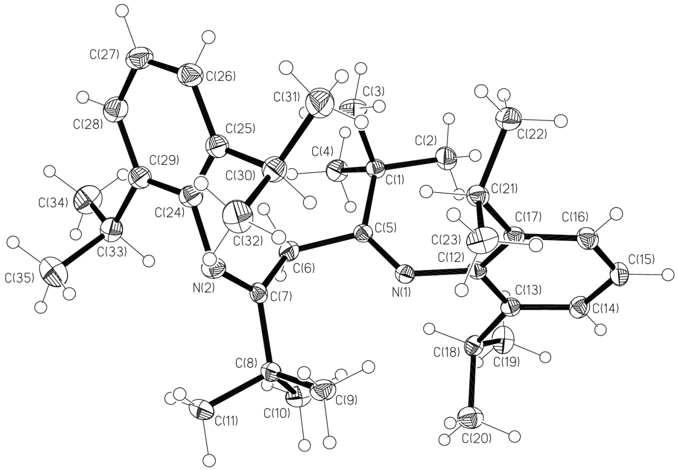


Figure 1
A view of the non-symmetrical conformer of (1) with the atom-numbering scheme. Displacement ellipsoids enclose 30% probability surfaces.

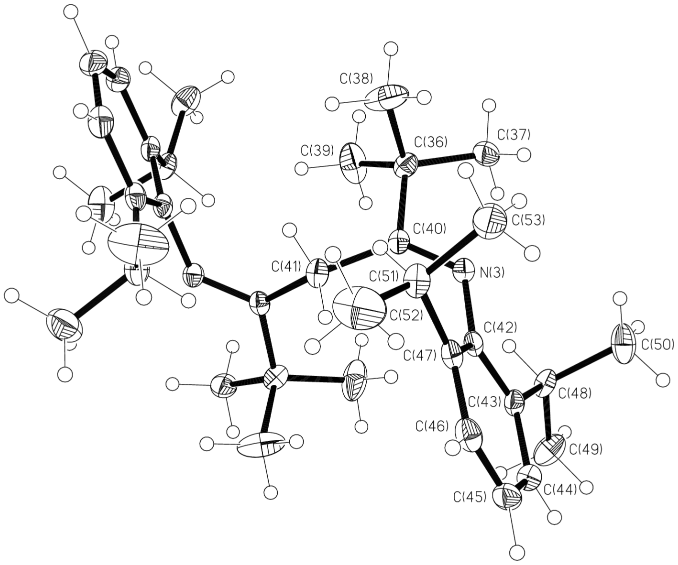


Figure 2
A view of the C_2-symmetric conformer of (1) with a selected atom-numbering scheme. Displacement ellipsoids enclose 30% probability surfaces.

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.975$, $T_{\max} = 0.989$
33 069 measured reflections

8317 independent reflections
5827 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.043$
$\theta_{\max} = 25.0°$
$h = -49 \rightarrow 49$
$k = -11 \rightarrow 11$
$l = -29 \rightarrow 29$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.124$
$S = 1.02$
8317 reflections
501 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0566P)^2 + 4.2336P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.49$ e Å^{-3}
$\Delta\rho_{\min} = -0.25$ e Å^{-3}

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

N1—C5	1.270 (2)	C6—C7	1.509 (3)
N1—C12	1.423 (2)	N3—C40	1.275 (2)
N2—C7	1.283 (2)	N3—C42	1.416 (2)
N2—C24	1.420 (2)	C40—C41	1.527 (2)
C5—C6	1.530 (2)		
C5—N1—C12	128.60 (15)	N2—C7—C8	115.91 (15)
C7—N2—C24	124.08 (15)	C6—C7—C8	121.23 (16)
N1—C5—C6	117.63 (15)	C40—N3—C42	126.12 (15)
N1—C5—C1	129.83 (15)	N3—C40—C41	124.14 (15)
C6—C5—C1	112.51 (14)	N3—C40—C36	115.65 (15)
C7—C6—C5	120.35 (16)	C41—C40—C36	119.37 (13)
N2—C7—C6	122.46 (16)	C40—C41—C40$^{\rm i}$	126.6 (2)

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

Analysis of Fourier difference maps, based on the refinement of non-H atoms, clearly showed the location of the H atoms at C6 (two H atoms) and C41 (one independent H atom). No peaks corresponding to H atoms in the vicinity of N atoms N1, N2 or N3 were observed. After inspection of the difference map, all H atoms were placed in calculated positions and subsequently allowed to ride on

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their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$. Methyl groups were treated as rotating rigid groups with $U_{iso}(H) = 1.5U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1999); 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).

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