Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

2-[2-(2,6-Dimethylphenylamino)phenylmethyleneamino]-1,3-diisopropylbenzene

Haiyang Gao,[a] Qing Wu[a] and Seik Weng Ng[b]*

[a]Institute of Polymer Science, School of Chemistry and Chemical Engineering, Sun Yat-Sen University, Guangzhou 510275, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: seikweng@um.edu.my

Received 5 January 2004
Accepted 7 January 2004
Online 17 January 2004

Key indicators

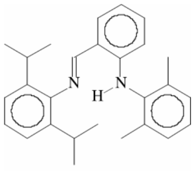
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.052
wR factor = 0.179
Data-to-parameter ratio = 19.3

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

The *ortho*-disubstituted aromatic ring in the title compound, $C_{27}H_{32}N_2$, is twisted by 76.1 (1)° with respect to the isopropyl-substituted ring and by 89.3 (1)° with respect to the methyl-substituted ring. The amino N atom is linked intramolecularly to the imino N by a hydrogen bond [N···N = 2.686 (2) Å].

Comment

Multidentate complexes of the late transition metals show high catalytic activity for olefin polymerization (Ittle *et al.*, 2000); for the nickel complexes in particular, the salicylaldehyde–imine type of Schiff base complexes are particularly useful catalysts (Wang *et al.*, 1998; Younkin *et al.*, 2000). A plethora of such Schiff bases and their complexes has been structurally authenticated. The Schiff bases are readily synthesized by condensing the salicylaldehyde reagent with a primary amine; on the other hand, an arylamino benzaldehyde–arylimine has yet not been documented in the Cambridge Structural Database (Allen, 2002). The Ni complex of the present compound (Fig. 1) is being investigated for catalytic activity in a continuation of related investigations (He, Yao, Luo, Zhang, Liu *et al.*, 2003; He, Yao, Luo, Zhang & Wu, 2003). The arylamino entity is introduced into the benzaldehyde-2,6-diisopropylphenylimine system by the nucleophilic displacement of the *ortho*-substituted fluoride (Sprung, 1940).



(I)

The title compound, (I), is envisaged as consisting of three aromatic portions, a dimethylphenyl part that is linked to the phenylene part through the amino group [N2—C19 = 1.368 (2) Å and N2—C20 = 1.424 (2) Å], along with a diisopropylphenyl part that is connected to the phenylene part through the methyleneimine group [N1—C1 = 1.418 (2) Å and N1—C13 = 1.265 (2) Å]. The aromatic rings in the two portions are severely twisted with respect to the phenylene ring, probably to avoid crowding by their 2,6-substituents. The amino group is linked to the imino group by an intramolecular hydrogen bond [N···N = 2.686 (2) Å].

Experimental

A xylene (15 ml) solution of 2-fluorobenzaldehyde (3.28 ml, 3.83 g, 30.9 mmol) and 2,6-diisopropylphenylamine (6.42 ml, 6.03 g,

DOI: 10.1107/S1600536804000492

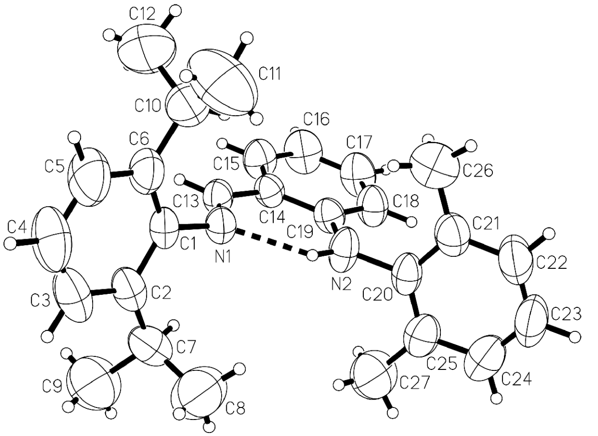


Figure 1
*ORTEP*II (Johnson, 1976) plot of (I); displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as spheres of arbitrary radii.

34.0 mmol) was stirred for 2 h, after which magnesium sulfate was added. The mixture was filtered and the yellow solution cooled to 263 K to furnish 4.49 g of yellow 2,6-$(C_3H_7)_2C_6H_3N{=}CH\text{-}2C_6H_4F$; the removal of the solvent yielded an additional 1.43 g of the compound (approximate yield of 70%). Elemental analysis calculated for $C_{19}H_{22}FN$: C 80.53, H 7.83, N 8.94%; found: C 80.37, H 7.54, N 4.73%. An *n*-hexane solution (6.5 ml) of *n*-butyllithium (2.6 *M*) was added to 2,6-dimethylaniline (2.2 ml, 2.10 g, 1.74 mmol) dissolved in THF (40 ml) at 199 K; after the addition, the solution was allowed to warm to room temperature over several hours. It was transferred into a solution of $C_{19}H_{22}NF$ (4.40 g, 1.55 mmol) dissolved in thf (50 ml). The mixture was stirred for 1 h and was then quenched with water (20 ml). The desired product was extracted with *n*-hexane and the solvent removed to give an oil. The compound was obtained as pale-yellow crystals upon recrystallization from methanol in 50% yield. Elemental analysis calculated for $C_{27}H_{32}N_2$: C 84.26, H 8.26, N 7.28%; found C 84.34, H 8.47, N 7.11%.

Crystal data

$C_{27}H_{32}N_2$
$M_r = 384.55$
Monoclinic, $P2_1/n$
$a = 12.013$ (2) Å
$b = 7.789$ (1) Å
$c = 25.542$ (3) Å
$\beta = 97.802$ (3)°
$V = 2367.9$ (5) Å^3
$Z = 4$

$D_x = 1.079$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 854 reflections
$\theta = 2.7$–23.1°
$\mu = 0.06$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.49 × 0.48 × 0.43 mm

Data collection

Bruker SMART 1K area-detector diffractometer
φ and ω scans
Absorption correction: none
13131 measured reflections
5184 independent reflections

2681 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.024$
$\theta_{\rm max} = 27.1°$
$h = -15 \rightarrow 13$
$k = -4 \rightarrow 9$
$l = -32 \rightarrow 32$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.179$
$S = 1.01$
5184 reflections
268 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0863P)^2 + 0.2352P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.16$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1	1.418 (2)	C13—C14	1.444 (2)
N1—C13	1.265 (2)	C14—C15	1.392 (2)
N2—C19	1.368 (2)	C14—C19	1.413 (2)
N2—C20	1.424 (2)	C15—C16	1.367 (3)
C1—C6	1.393 (3)	C16—C17	1.374 (3)
C1—C2	1.400 (3)	C17—C18	1.362 (3)
C2—C3	1.381 (3)	C18—C19	1.396 (2)
C2—C7	1.507 (3)	C20—C25	1.390 (3)
C3—C4	1.369 (4)	C20—C21	1.391 (3)
C4—C5	1.365 (4)	C21—C22	1.378 (3)
C5—C6	1.381 (3)	C21—C26	1.496 (3)
C6—C10	1.523 (3)	C22—C23	1.352 (4)
C7—C8	1.502 (4)	C23—C24	1.366 (4)
C7—C9	1.511 (4)	C24—C25	1.394 (3)
C10—C12	1.487 (4)	C25—C27	1.498 (3)
C10—C11	1.513 (4)		
C1—N1—C13	121.4 (2)	C13—C14—C19	122.8 (2)
C19—N2—C20	123.8 (2)	C15—C14—C19	118.4 (2)
C2—C1—C6	121.8 (2)	C14—C15—C16	121.9 (2)
C6—C1—N1	120.0 (2)	C15—C16—C17	119.3 (2)
C2—C1—N1	118.0 (2)	C16—C17—C18	120.9 (2)
C1—C2—C3	117.6 (2)	C17—C18—C19	121.0 (2)
C1—C2—C7	120.6 (2)	N2—C19—C14	120.3 (2)
C3—C2—C7	121.8 (2)	N2—C19—C18	121.1 (2)
C2—C3—C4	121.4 (2)	C18—C19—C14	118.6 (2)
C3—C4—C5	119.9 (2)	C21—C20—C25	121.9 (2)
C4—C5—C6	121.7 (2)	C25—C20—N2	119.3 (2)
C1—C6—C5	117.6 (2)	C21—C20—N2	118.8 (2)
C1—C6—C10	122.0 (2)	C20—C21—C22	118.1 (2)
C5—C6—C10	120.4 (2)	C20—C21—C26	121.1 (2)
C2—C7—C8	112.3 (2)	C22—C21—C26	120.8 (2)
C2—C7—C9	114.2 (2)	C21—C22—C23	121.1 (2)
C8—C7—C9	110.5 (2)	C22—C23—C24	120.7 (2)
C6—C10—C11	112.2 (2)	C23—C24—C25	121.0 (2)
C6—C10—C12	112.4 (2)	C20—C25—C24	117.2 (2)
C11—C10—C12	111.9 (3)	C20—C25—C27	120.9 (2)
N1—C13—C14	124.7 (2)	C24—C25—C27	121.9 (2)
C13—C14—C15	118.9 (2)		

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···N1	0.89 (2)	1.98 (2)	2.686 (2)	136 (2)

The H atoms were placed at calculated positions in the riding-model approximation (C—H = 0.93 Å for the H atoms on Csp^2 atoms, C—H = 0.98 Å for methine H atoms and 0.96 Å for methyl H atoms). The displacement parameters of the H atoms were set to 1.2 times those of the equivalent isotropic displacement parameters of the carrier atoms, except for the methyl H atoms, for which the factor was 1.5. The N-bound H atom was located and refined freely. Furthermore, the torsion angles of the two methyl groups directly bound to the aromatic ring were refined.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); 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: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

The authors thank the National Natural Science Foundation of China, the Natural Science Foundation of Guangdong

Province, Sun Yat-Sen University and the University of Malaya for supporting this work.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Bruker (1999). *SAINT* and *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.

He, X.-H., Yao, Y.-Z., Luo, X., Zhang, J., Liu, Y., Zhang, L. & Wu, Q. (2003). *Organometallics*, **22**, 4952–4957.

He, X.-H., Yao, Y.-Z., Luo, X., Zhang, L. & Wu, Q. (2003). *Acta Cryst.* E**59**, m1196–m1197.

Ittle, S. D., Johnson, L. K. & Brookhart, M. (2000). *Chem. Rev.* **100**, 1169–1203.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Sprung, M. M. (1940). *Chem. Rev.* **26**, 297–338.

Wang, C., Friedrich, S., Younkin, T. R., Li, R. T., Grubbs, R. H., Bansleben, D. A. & Day, M. W. (1998). *Organometallics*, **17**, 3149–3151.

Younkin, T. R., Connor, E. F., Henderson, J. I., Friedrich, S. K., Grubbs, R. H. & Bansleben, D. A. (2000). *Science*, **287**, 460–461.