

N,N'*-Bis(2,6-dimethylphenyl)-2-methyl-1-(trimethylsilyl)ethane-1,2-diimine*D. J. Loroño-González**

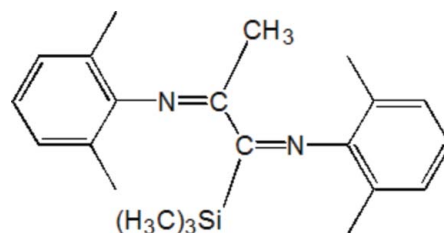
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The dimerization of 2,6-dimethylphenyl isocyanide by a double insertion of 2,6-dimethylphenyl isocyanide with MeLi, followed by treatment with Me₃SiCl, gave the title compound, C₂₂H₃₀N₂Si, which adopts the *trans* configuration with respect to the position of the 2,6-dimethylphenylamine groups about the central C—C bond. The molecule is stabilized by C—H···π interactions.

Received 26 April 2006
Accepted 2 June 2006**Key indicators**Single-crystal X-ray study
T = 150 K
Mean σ (C—C) = 0.007 Å
R factor = 0.076
wR factor = 0.218
Data-to-parameter ratio = 12.3For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.**Comment**

The addition of organolithium compounds to isonitriles is subject to various side reactions, notably α -deprotonation (Walborsky *et al.*, 1970, 1971; Niznik *et al.*, 1974; Hirowatari & Walborsky, 1974; Niznik & Walborsky, 1974). In the absence of α -H atoms, however, isonitriles show carbene-like reactivity (Barluenga *et al.*, 1988; Ito *et al.*, 1984), giving lithioimines; subsequent reaction with electrophilic reagents gives the corresponding imine derivatives. The title compound, (I) (Fig. 1), was obtained as the main product of the reaction of lithium aldimine obtained from the α -addition of methyl lithium to 2,6-dimethylphenyl isocyanide with Me₃SiCl.



(I)

The molecule adopts a *trans* configuration with respect to the position of the 2,6-dimethylphenylamine groups about the central C1—C2 bond. The C1—C2 single bond length of 1.511 (6) Å does not indicate an effective conjugation between the two azomethine systems. The bond angle sums about C1 and C2 of 360° suggests that all these atoms are *sp*²-hybridized. The conformation of (I) is described by the torsion angles N1—C1—C2—N2 and Si—C1—C2—C3, which are −170.8 (4) and −170.8 (3)°, respectively, and the geometry about the Si atom is tetrahedral. The dihedral angle between the benzene rings is 6.4 (2)°. There is no significant π – π interaction. However, the molecule is stabilized by C—H··· π interactions C4—H4B···(C7—C12) and C4—H4C···(C7—C12) of 2.92 Å, where the angles about the H atoms are 105 and 107°, respectively.

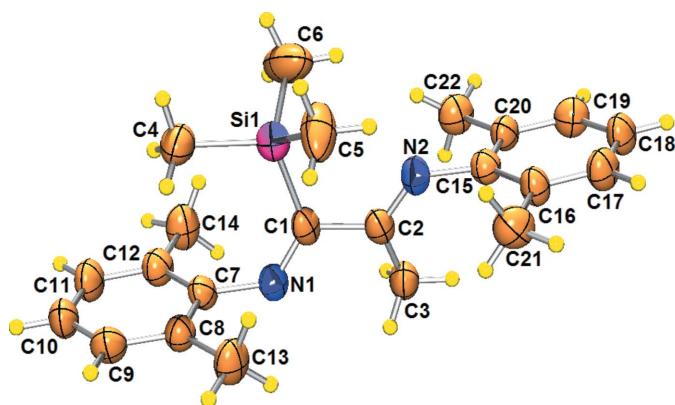


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids.

Experimental

For the preparation of (I), a solution of 2,6-dimethylphenyl isonitrile (0.260 g, 1.91 mmol) in diethyl ether (10 ml) was treated with methyllithium (1.2 ml, 1.6 M in hexanes, 1.91 mmol) at 195 K. After 30 min, a solution of trimethylsilyl chloride (0.24 ml, 1.91 mmol) was added dropwise and the solution was allowed to warm to room temperature overnight. The solution was filtered under a nitrogen atmosphere and the LiCl removed by filtration through Celite 521. The solvent and volatile components were removed under vacuum to provide an orange solid. The product, (I), was filtered off and crystallized from a concentrated ether solution at room temperature to give orange plate-shaped crystals (0.175 g, yield 25%). The melting point is 398 K and CHN analysis of (I) found: C 77.55, H 8.31, N 7.80%; calculated: C 79.29; H 8.64; N 7.99%.

Crystal data

$C_{22}H_{30}N_2Si$	$Z = 4$
$M_r = 350.57$	$D_x = 1.106 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 8.159 (5) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$b = 12.612 (8) \text{ \AA}$	$T = 150 (2) \text{ K}$
$c = 20.486 (12) \text{ \AA}$	Plate, light orange
$\beta = 92.692 (8)^\circ$	$0.18 \times 0.16 \times 0.09 \text{ mm}$
$V = 2106 (2) \text{ \AA}^3$	

Data collection

Bruker SMART APEX CCD diffractometer with an Oxford Cryosystems low-temperature device	11514 measured reflections
φ and ω scans	4258 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	3308 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.967$, $T_{\max} = 0.997$	$R_{\text{int}} = 0.122$
	$\theta_{\text{max}} = 26.4^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0932P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.077$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.218$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 0.93$	$\Delta\rho_{\text{max}} = 0.35 \text{ e \AA}^{-3}$
3308 reflections	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$
269 parameters	Extinction correction: SHELXL97
H-atom parameters constrained	Extinction coefficient: 0.021 (3)

Table 1

Selected geometric parameters (\AA , $^\circ$).

Si1—C1	1.926 (5)	N2—C15	1.414 (5)
N1—C1	1.288 (5)	C1—C2	1.511 (6)
N1—C7	1.428 (5)	C2—C3	1.474 (6)
N2—C2	1.281 (5)		
C4—Si1—C1	113.2 (2)	C2—C1—Si1	116.4 (3)
C1—N1—C7	121.8 (4)	N2—C2—C3	126.0 (4)
C2—N2—C15	123.2 (4)	N2—C2—C1	114.1 (4)
N1—C1—C2	113.6 (4)	C3—C2—C1	119.9 (4)
N1—C1—Si1	130.1 (4)		
C7—N1—C1—C2	179.6 (3)	C15—N2—C2—C1	179.0 (4)
C7—N1—C1—Si1	−0.6 (6)	Si1—C1—C2—N2	9.4 (5)
C15—N2—C2—C3	−0.8 (7)	N1—C1—C2—C3	9.0 (6)

Table 2

Hydrogen-bond geometry (\AA , $^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4C \cdots C7	0.96	2.85	3.110 (7)	97
C4—H4B \cdots C8	0.96	2.80	3.415 (7)	123
C4—H4B \cdots C9	0.96	3.15	3.844 (7)	131
C4—H4C \cdots C11	0.96	3.08	3.794 (7)	132
C4—H4C \cdots C12	0.96	2.70	3.353 (7)	126

The H atoms were included in the refinement at calculated positions, in the riding-model approximation, with C—H distances of 0.93 and 0.96 \AA . The $U_{\text{iso}}(\text{H})$ values were set equal to 1.5 (methyl H atoms) or 1.2 (other H atoms) times $U_{\text{eq}}(\text{C})$.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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