

{1-Methyl-2-[3-(trimethylsilylethynyl)phenyl-imino]propylidene}[3-(trimethylsilylethynyl)-phenyl]amine**Hyeon Mo Cho, Jeffrey S. Moore and Scott R. Wilson***

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Key indicatorsSingle-crystal X-ray study
 $T = 193$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
Disorder in main residue
 R factor = 0.056
 wR factor = 0.166
Data-to-parameter ratio = 14.8For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

In the crystal structure of the title compound, $\text{C}_{26}\text{H}_{32}\text{N}_2\text{Si}_2$, there are two independent half-molecules in the asymmetric unit, each molecule lying on a crystallographic inversion center, and hence, in both, the two imine groups are mutually *trans*. One of the molecules is disordered.

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Comment

Molecules containing the 1,4-diaza-1,3-butadiene skeleton are interesting because of their versatile coordination behavior and the properties of their metal complexes (van Koten & Vrieze, 1982). The central diimine group of the title compound, (I), is planar. The angle between the planes of the ordered diimine group and each benzene ring is 72.0 (1)°.

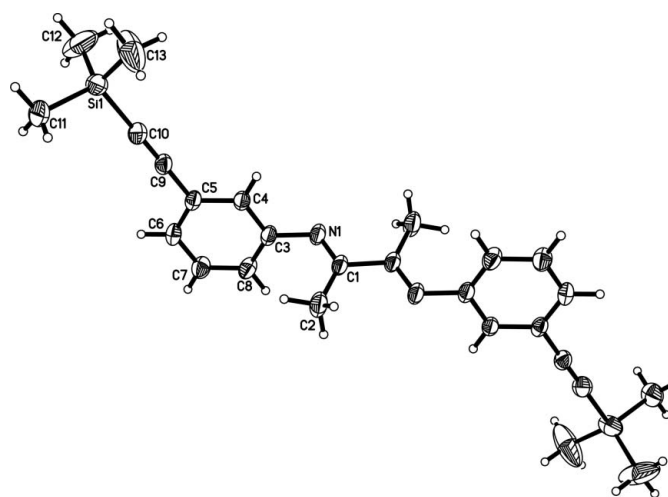
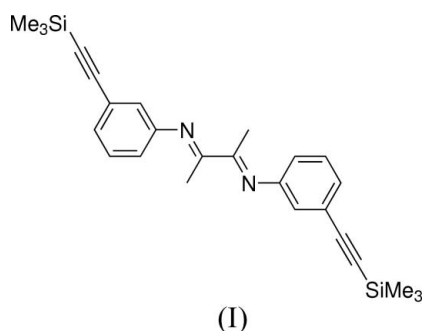


Figure 1
The molecular structure of the title compound, showing the ordered molecule. Displacement ellipsoids are drawn at the 35% probability level. The unlabeled atoms are related by the symmetry operator $(-x, 2 - y, 2 - z)$.

Experimental

The title compound was prepared by the reaction of (3-iodophenyl)-[2-(3-iodophenylimino)-1-methylpropylidene]amine with 2 equivalents of trimethylsilylacetylene in the presence of tetrakis(tri-2-furylphosphine)palladium, CuI and triethylamine in tetrahydrofuran (Sonogashira *et al.*, 1975). The product was separated by silica-gel column chromatography (ethyl acetate–*n*-hexane, 1:20) with 3% triethylamine and was recrystallized from diethyl ether at room temperature. Single crystals suitable for X-ray diffraction were grown at room temperature by evaporation of a diethyl ether solution. ¹H NMR (500 MHz, THF-*d*₈, δ, p.p.m.): 7.32 (*t*, 2H), 7.18 (*t*, 1H), 7.17 (*t*, 1H), 6.88 (*t*, 2H), 6.77 (*q*, 1H), 6.75 (*q*, 1H), 2.11 (*s*, 6H), 0.24 (*s*, 18H). ¹³C NMR (126 MHz, THF-*d*₈, δ, p.p.m.): 169.3, 152.2, 129.8, 127.9, 124.9, 122.7, 119.9, 105.9, 94.3, 15.2, 0.0. LRMS calculated for C₂₆H₃₂N₂Si₂: 428.2; found: 428.1.

Crystal data

C ₂₆ H ₃₂ N ₂ Si ₂	Z = 2
M _r = 428.72	D _x = 1.039 Mg m ⁻³
Triclinic, P $\bar{1}$	Mo K α radiation
a = 10.5351 (3) Å	Cell parameters from 7708 reflections
b = 11.4008 (3) Å	θ = 2.3–27.2°
c = 11.8287 (4) Å	μ = 0.14 mm ⁻¹
α = 80.458 (2)°	T = 193 (2) K
β = 78.076 (2)°	Prism, colorless
γ = 89.318 (2)°	0.48 × 0.34 × 0.18 mm
V = 1370.47 (7) Å ³	

Data collection

Bruker Kappa/APEX-II CCD diffractometer	6809 independent reflections
φ and ω scans	5162 reflections with $I > 2\sigma(I)$
Absorption correction: integration (SHELXTL/XPREP; Bruker, 2001)	R _{int} = 0.022
T _{min} = 0.948, T _{max} = 0.975	θ_{\max} = 28.3°
38376 measured reflections	h = -14 → 14
	k = -15 → 15
	l = -15 → 15

Refinement

Refinement on F ²	$w = 1/[\sigma^2(F_o^2) + (0.0772P)^2 + 0.4863P]$
R[F ² > 2σ(F ²)] = 0.056	where $P = (F_o^2 + 2F_c^2)/3$
wR(F ²) = 0.166	(Δ/σ) _{max} = 0.031
S = 1.03	$\Delta\rho_{\max}$ = 0.55 e Å ⁻³
6809 reflections	$\Delta\rho_{\min}$ = -0.61 e Å ⁻³
460 parameters	
H-atom parameters constrained	

The proposed model imposes inversion symmetry on the two independent *trans* diimine molecules. One of the two independent centrosymmetric molecules is ordered, while the other is disordered. The disorder model includes two superimposed molecules in the same plane rotated 180° roughly along the axis of the imine bonds. Relative primary site occupancy converged at 0.7145 (7). The trimethyl groups of these superimposed molecules suffered from additional rotational disorder about the ethynyl–Si bond. The imine and all ordered methyl H-atom positions, R–CH₃, were optimized by rotation about the R–C bonds with idealized C–H (0.98 Å). The

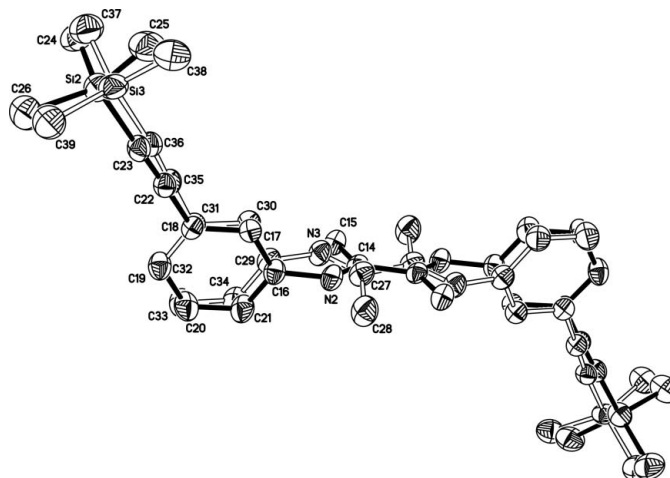


Figure 2

The molecular structure of the title compound, showing the disordered molecule with solid bonds for the major site. H atoms and minor site disordered methyl groups have been omitted for clarity. Displacement ellipsoids are drawn at the 35% probability level. The unlabeled atoms are related by the symmetry operator ($-x, 1 - y, -z$).

remaining H atoms were included as riding idealized contributors (C–H = 0.95 Å). $U_{\text{iso}}(\text{H})$ values were assigned as 1.2 times $U_{\text{eq}}(\text{carrier atom})$. Overlapping disordered groups were restrained to have a similar geometry using effective standard deviations of 0.01 Å for bond lengths and 0.02 Å for 1,3-distances. Rigid bond restraints with similar amplitudes were imposed on displacement parameters for disordered sites (s.u. = 0.01 Å²). The highest peaks in the final difference Fourier map were in the vicinity of atom Si1, suggesting additional rotational disorder for this group; the final map had no other significant features.

Data collection: APEX-II (Bruker, 2004); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2001); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: XCIF (Bruker, 2001).

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