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A chelate-stabilized tungsten–bis(imido) complex

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Key indicators

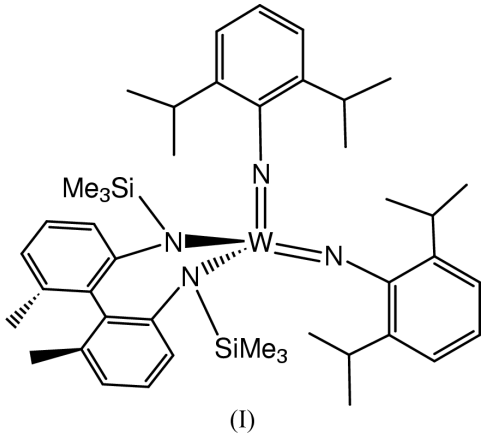
Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
R factor = 0.022
wR factor = 0.047
Data-to-parameter ratio = 21.0

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

The title compound, [*N,N′*-bis(trimethylsilyl)-6,6′-dimethylbipheny]-2,2′-diamido]bis(2,6-diisopropylphenylimido)tungsten(VI), $[W(C_{12}H_{17}N)_2(C_{20}H_{30}N_2Si_2)]$, has been synthesized and its crystal structure determined. The geometry around the W atom, imposed by the biphenyldiamido and bulky 2,6-diisopropylphenylimido ligands, is that of a tetrahedron. As a result of this geometry, the biphenyl group is twisted, with a dihedral angle of 69.65 (8)°. Principal distances include W—N(imido) 1.762 (2) and 1.772 (2) Å, and W—N(amido) 1.976 (2) and 1.984 (2) Å.

Comment

Recently, the search for new catalysts based on high-oxidation-state early transition metal complexes has become a major field of interest. With the ability to significantly alter the properties of a catalyst by changing the ligands surrounding the metal, chelating diamide ligands have become an alternative to the widely used cyclopentadienyl systems (Kempe, 2000; Schrock, 1997). Additional work has focused on the use of C_2-symmetric bis(silylamido) ligands to induce stereocontrol in catalytic reactions (Cloke *et al.*, 1996; Drost *et al.*, 1996; Gountchev & Tilley, 1997). As part of our investigation of the chemistry of chelate stabilized group 6 metal complexes, we have synthesized and determined the structure of the title compound, $[W\{N(2,6\text{-}(^iPr)_2C_6H_3)\}_2\{(NSiMe_3)C_6H_3Me\}_2]$, (I). A displacement ellipsoid drawing of (I) with the atom-labeling scheme is shown in Fig. 1.



(I)

The coordination geometry around the W atom in (I) is distorted tetrahedral, where the N4—W—N2 and N3—W—N1 angles have the largest deviations and N1—W—N2 and N2—W—N3 have the the smallest deviations. The short W—N(imido) distances of 1.762 (2) and 1.772 (2) Å are within

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normal ranges for W^{VI}–bis(imido) complexes (Clark *et al.*, 1988) and indicate that there is significant interaction between the N3 and N4 lone pairs of electrons and the tungsten center. In order to adopt the observed tetrahedral geometry about the W atom, the diaminobiphenyl ligand is twisted, with a dihedral angle of 69.65 (8)° measured between the planes of the two phenyl rings. The W—N1 and W—N2 distances of 1.976 (2) and 1.984 (2) Å are consistent with previously observed W^{VI}–amido complexes (Boncella *et al.*, 1997). In addition, the sums of the angles at N1 and N2 are 360°, with respect to the attached Csp^2, Si(Csp^3) and W atoms, consistent with there being a trigonal–planar environment at the N atoms.

Experimental

2,2′-Diamino-6,6′-dimethylbiphenyl (Kanoh *et al.*, 1987), *N,N*′-bis-(trimethylsilyl)-2,2′-diamino-6,6′-dimethylbiphenyl and its dilithium salt (Gountchev & Tilley, 1997) were prepared according to literature procedures. Using standard Schlenk techniques, $[WCl_2\{N(2,6\text{-}(^iPr)_2C_6H_3)\}_2]$ (DME) was allowed to react with one equivalent of $\{(NSiMe_3)C_6H_3Me\}Li_2$ in Et_2O at 195 K for 30 min. The reaction was stirred at room temperature for an additional 12 h before the mixture was filtered and the solvent removed under reduced pressure. Recrystallization by cooling a pentane solution of $[W\{N(2,6\text{-}(^iPr)_2C_6H_3)\}_2\{(NSiMe_3)C_6H_3Me\}_2]$ to 233 K afforded bright yellow crystals after 7 d. The air and moisture sensitivity of (I) necessitated isolation of the crystals in a dry box. Crystals were placed in oil before being removed from the dry box.

Crystal data

$[W(C_{12}H_{17}N)_2(C_{20}H_{30}N_2Si_2)]$	D_x = 1.350 Mg m^{-3}
M_r = 889.02	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 268 reflections
a = 17.480 (2) Å	θ = 2.0–27.5°
b = 11.443 (2) Å	μ = 2.73 mm^{-1}
c = 21.889 (3) Å	T = 173 (2) K
β = 91.95 (2)°	Needle, yellow
V = 4376 (1) Å^3	0.23 × 0.11 × 0.08 mm
Z = 4	

Data collection

SMART CCD area-detector diffractometer	29 255 measured reflections
ω scans	10 022 independent reflections
Absorption correction: by integration based on measured indexed crystal faces (*SHELXTL*; Bruker, 1998)	8026 reflections with $I > 2\sigma(I)$
T_{min} = 0.480, T_{max} = 0.829	R_{int} = 0.035
	θ_{max} = 27.5°
	$h = -22 \rightarrow 22$
	$k = -14 \rightarrow 14$
	$l = -21 \rightarrow 28$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0160P)^2 + 1.0307P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.022	$(\Delta/\sigma)_{max}$ = 0.003
$wR(F^2)$ = 0.047	$\Delta\rho_{max}$ = 0.62 e Å^{-3}
S = 1.00	$\Delta\rho_{min}$ = −0.54 e Å^{-3}
10 022 reflections	Extinction correction: *SHELXL*97
477 parameters	Extinction coefficient: 0.00010 (3)
H-atoms parameters constrained	

The H atoms were placed in idealized positions and were refined riding on their parent atoms. C—H distances of 0.98 and 0.95 Å were used for methyl and sp^2 C atoms, respectively. A distance of 1.00 Å was used for the tertiary H atoms on C27 and C30. The H-atom displacement parameters were $1.2U_{eq}$ of the parent C atom and $1.5U_{eq}$ for the methyl atoms. A hemisphere of frames, 0.3° in ω, was collected. The first 50 frames were remeasured at the end of data collection to monitor instrument and crystal stability. Full data collection details are in the relevant _special_details section of the archived CIF and also reported elsewhere (Abboud *et al.*, 1997).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART* and *SAINT* (Bruker, 1998); data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

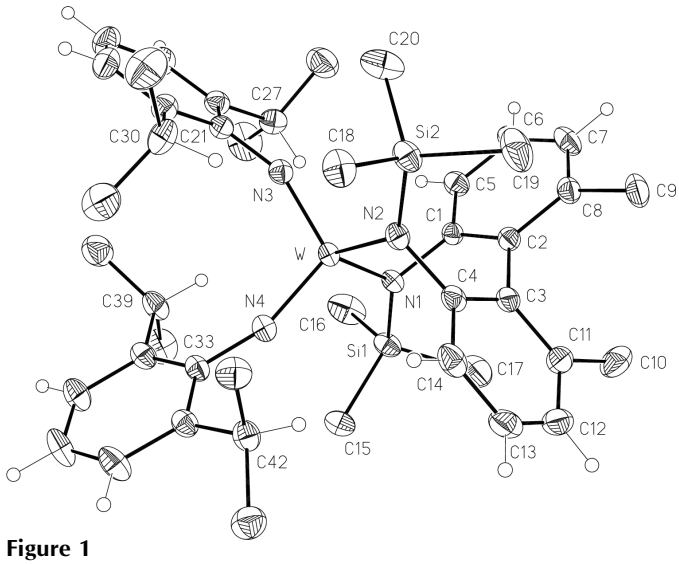

Figure 1
The molecular structure of (I), with 40% probability ellipsoids, showing the atom-labeling scheme. The methyl H atoms have been omitted for clarity.

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