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An N-bridged tritungsten compound for the chemical vapor deposition of WN_x thin films

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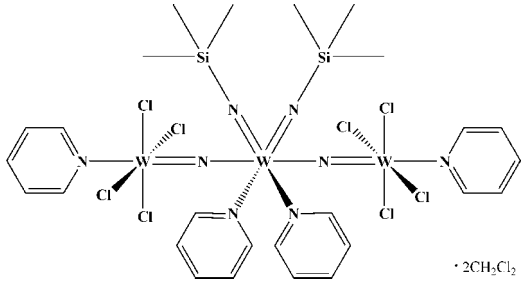
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.008 Å; R factor = 0.029; wR factor = 0.054; data-to-parameter ratio = 23.7.

In the title compound, octachlorido-1κ^4Cl,3κ^4Cl-di-μ_2-nitrido-1:2κ^2N:N;2:3κ^2N:N-tetrapyridine-1κN,2κ^2N,3κN-bis(trimethylsilylimido-2κN)tritungsten(V) dichloromethane disolvate, $[W_3(C_3H_9NSi)_2Cl_8N_2(C_5H_5N)_4]\cdot 2CH_2Cl_2$, the central W metal atom is located on a twofold rotation axis and is coordinated in a distorted octahedral environment by two trimethylsilylimido groups, two pyridine molecules and two μ-N atoms which bridge it to the two other W atoms. These terminal W centers also show a distorted octahedral coordination, the bridging N atom and the pyridine ligand being *trans* with respect to each other. Additionally, four Cl atoms are bound to each terminal W atom, in a square-planar geometry, completing the octahedral coordination.

Related literature

Closely related structures were reported by Dehnicke & Strähle (1992) and Ergezinger *et al.* (1989). For related literature concerning the properties of tungsten nitrides, see: Dehnicke & Strähle (1965). For properties of tungsten–imido complexes and their application in chemical vapor deposition, see: Bradley *et al.*, (1983, 1987) Bchir *et al.* (2005), Orpen *et al.*, (1989) and Rische *et al.* (2006). For related literature, see: Wu *et al.* (2006).



Experimental

Crystal data

$[W_3(C_3H_9NSi)_2Cl_8N_2(C_5H_5N)_4]\cdot 2CH_2Cl_2$
M_r = 1523.83
Monoclinic, $C2/c$
a = 10.0546 (8) Å
b = 19.5493 (15) Å
c = 25.359 (2) Å
β = 94.329 (2)°
V = 4970.3 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 7.65 mm^{-1}
T = 173 (2) K
0.34 × 0.29 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: integration [based on measured indexed crystal faces (*SHELXTL*; Sheldrick, 2000)]
T_{min} = 0.107, T_{max} = 0.736
15825 measured reflections
5688 independent reflections
3802 reflections with $I > 2\sigma(I)$
R_{int} = 0.074

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.054
S = 0.76
5688 reflections
240 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.54 e Å^{-3}
$\Delta\rho_{min}$ = −1.24 e Å^{-3}

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker 1998); data reduction: *SAINT* and *SHELXTL* (Sheldrick, 2000); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZL2070).

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supplementary materials

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An N-bridged tritungsten compound for the chemical vapor deposition of WN_x thin films

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Comment

The title compound is a C_2-symmetric trimetallic tungsten complex incorporating two μ-N moieties which are fairly rare in organometallic chemistry with only a few reported occurrences (Ergezinger *et al.*, 1989). The W1—N2 bond distance of 2.090 (4) Å is in the range of W—N single bonds seen in tungsten amido compounds (Wu *et al.*, 2006), whereas the W2—N2 bond (1.707 (4) Å) more closely resembles a tungsten imido triple bond (Bradley *et al.*, 1987). Both bond lengths are comparable to the bond lengths seen in $[WNCl_3(PhCN)]_4.3CH_2Cl_2$, a compound which also incorporates a μ-N moiety (Ergezinger *et al.*, 1989). This observation is further corroborated by a W2—N2—W1 bond angle of 169.2° which is typical for tungsten imido complexes (Bradley *et al.*, 1983). The trimethylsilyl-imido functionality with a W1—N1 bond distance of 1.773 (4) Å and a W1—N1—Si1 bond angle of 169.3° are similar to reported values (Rische *et al.*, 2006). The W—Cl bonds averaging 2.33 Å are within the expected range for chlorine rich tungsten imido complexes (Orpen *et al.*, 1989). The W1—N3(py) bond length of 2.326 (4) Å is consistent with reported values whereas the W2—N4(py) bond (2.424 (4) Å) seems to be elongated due to the decreased *trans*-effect of the trimethylsilyl imido ligand compared to the bridging μ-N atom (Rische *et al.*, 2006). Although the complex is C_2-symmetric in the solid state, two resonances (δ 0.77 and 0.76 p.p.m.) can be observed in the ^{1}H NMR spectrum.

Experimental

In a 250 ml Schlenk flask WCl_6 (3.75 g, 9.46 mmol) was suspended in 100 ml of toluene. 1,1,1,3,3,3-Hexamethyldisilazane (2.76 ml, 13.2 mmol) and of pyridine (4.08 ml, 50.4 mmol) were added *via* syringe at room temperature. The reaction mixture was stirred for an additional 24 h. The resulting red solution was filtered and the solvent removed *in vacuo*. The remaining solid was extracted with 2 *x* 10 ml of methylene chloride. The extracts were combined and filtered. The solution was layered with an equal volume of pentane and cooled to 248 K (−25 °C) to yield the pure title compound as a red crystalline solid. Yield 411 mg (10%, 3.15 mmol). ^{1}H NMR (Benzene-d_6, 298 K): δ 9.70 (br s, 4H, aromatic), 9.42 (d, 4H, aromatic), 6.95 (br s, 6H, aromatic), 6.73 (t, 2H, aromatic), 6.48 (t, 4H, aromatic), 0.77 (s, 9H, (SiCH_3)$_3$), 0.76 (s, 9H, (SiCH_3)$_3$). ^{13}C NMR (Benzene-d_6, 298 K): δ 3.59 (Si(*C*H$_3$)$_3$), 124.36 (aromatic), 125.07 (aromatic), 138.10 (aromatic), 139.35 (aromatic), 152.11 (aromatic), 153.26 (aromatic).

Refinement

The H atoms were placed in idealized positions and were refined riding on their parent atoms. C—H distances of 0.98 and 0.95Å were used for aromatic and methyl atoms respectively. The H atoms thermal parameters were 1.2U_{eq} of the parent C; 1.5U~eq~ for the methyl atoms.

Figures

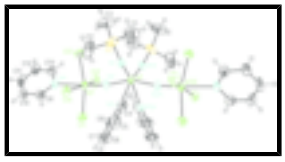


Fig. 1. : The molecular structure with 50% probability ellipsoids, showing the atom labeling scheme. Subscript "A" in the atomic labels denotes atoms created by the symmetry operation i = (x + 1, y, z + 1/2).

octachlorido-1κ^4*Cl*,3κ^4*Cl*-di-μ$_2$-nitrido-1:2κ^2*N*:*N*;2:3κ^2*N*:*N*-\ tetrapyridine-1κ*N*,2κ^2*N*,3κ*N*-bis(trimethylsilylimido-2κ*N*)tritungsten(V) dichloromethane disolvate

Crystal data

$[W_3(C_3H_9NSi)_2Cl_8N_2(C_5H_5N)_4]\cdot 2CH_2Cl_2$	F_{000} = 2880
M_r = 1523.83	D_x = 2.036 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
a = 10.0546 (8) Å	Cell parameters from 4105 reflections
b = 19.5493 (15) Å	θ = 2.0–28.0º
c = 25.359 (2) Å	μ = 7.65 mm^{-1}
β = 94.329 (2)º	*T* = 173 (2) K
V = 4970.3 (7) Å^3	Plate, red
Z = 4	0.34 × 0.29 × 0.04 mm

Data collection

Bruker SMART CCD area-detector diffractometer	5688 independent reflections
Radiation source: fine-focus sealed tube	3802 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.074
T = 173(2) K	θ_{max} = 27.5º
ω scans	θ_{min} = 1.6º
Absorption correction: integration [based on measured indexed crystal faces (SHELXTL; Sheldrick, 2000)]	*h* = −12→12
T_{min} = 0.107, T_{max} = 0.736	*k* = −23→25
15825 measured reflections	*l* = −30→32

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.029	H-atom parameters constrained
$wR(F^2)$ = 0.054	$w = 1/[\sigma^2(F_o^2) + (0.0063P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.76	$(\Delta/\sigma)_{max}$ = 0.002

5688 reflections	$\Delta\rho_{max}$ = 1.54 e Å^{-3}
240 parameters	$\Delta\rho_{min}$ = −1.24 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

The asymmetric unit consists of a half complex and a dichloromethane molecule.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
W1	0.5000	0.579772 (14)	0.2500	0.02631 (8)
W2	0.700778 (18)	0.558005 (11)	0.130958 (9)	0.02924 (6)
Cl1	0.75725 (13)	0.44150 (7)	0.13040 (6)	0.0456 (4)
Cl2	0.91447 (12)	0.57892 (8)	0.17020 (6)	0.0528 (4)
Cl3	0.67204 (14)	0.67298 (7)	0.11095 (6)	0.0488 (4)
Cl4	0.51146 (11)	0.53051 (7)	0.07504 (5)	0.0388 (3)
Cl5	0.14238 (18)	0.81650 (10)	0.01595 (9)	0.0922 (7)
Cl6	−0.12476 (16)	0.79117 (9)	0.04710 (7)	0.0704 (5)
Si1	0.29608 (13)	0.70110 (8)	0.18333 (7)	0.0444 (4)
N1	0.3864 (3)	0.6351 (2)	0.21422 (16)	0.0335 (11)
N2	0.6182 (3)	0.5604 (2)	0.18735 (16)	0.0310 (10)
N3	0.3784 (3)	0.4851 (2)	0.21409 (16)	0.0295 (10)
N4	0.8088 (4)	0.5616 (2)	0.04871 (17)	0.0350 (11)
C1	0.1684 (5)	0.7262 (3)	0.2266 (3)	0.075 (2)
H1A	0.2109	0.7445	0.2597	0.112*
H1B	0.1142	0.6863	0.2343	0.112*
H1C	0.1113	0.7614	0.2092	0.112*
C2	0.2213 (6)	0.6674 (4)	0.1205 (2)	0.086 (3)
H2A	0.1617	0.6293	0.1275	0.129*
H2B	0.2919	0.6512	0.0990	0.129*
H2C	0.1703	0.7037	0.1015	0.129*
C3	0.4096 (6)	0.7721 (3)	0.1717 (3)	0.091 (3)
H3A	0.4485	0.7895	0.2057	0.136*
H3B	0.3601	0.8088	0.1526	0.136*
H3C	0.4810	0.7559	0.1506	0.136*
C4	0.2439 (5)	0.4848 (3)	0.2134 (2)	0.0410 (15)
H4A	0.2010	0.5232	0.2276	0.049*

C5	0.1662 (6)	0.4319 (3)	0.1934 (2)	0.0494 (16)
H5A	0.0720	0.4338	0.1941	0.059*
C6	0.2253 (6)	0.3769 (3)	0.1726 (2)	0.0560 (18)
H6A	0.1729	0.3395	0.1592	0.067*
C7	0.3623 (6)	0.3757 (3)	0.1709 (2)	0.0441 (15)
H7A	0.4060	0.3384	0.1555	0.053*
C8	0.4322 (5)	0.4299 (2)	0.1920 (2)	0.0363 (13)
H8A	0.5264	0.4288	0.1911	0.044*
C9	0.7786 (5)	0.5171 (3)	0.0082 (2)	0.0414 (15)
H9A	0.7151	0.4820	0.0128	0.050*
C10	0.8371 (5)	0.5209 (3)	−0.0397 (2)	0.0456 (15)
H10A	0.8128	0.4895	−0.0673	0.055*
C11	0.9305 (5)	0.5707 (3)	−0.0464 (2)	0.0495 (16)
H11A	0.9723	0.5737	−0.0787	0.059*
C12	0.9635 (5)	0.6164 (3)	−0.0061 (2)	0.0450 (15)
H12A	1.0274	0.6514	−0.0103	0.054*
C13	0.9011 (5)	0.6101 (3)	0.0410 (2)	0.0425 (15)
H13A	0.9245	0.6413	0.0688	0.051*
C14	−0.0070 (6)	0.8512 (3)	0.0316 (3)	0.0626 (19)
H14A	−0.0442	0.8788	0.0013	0.075*
H14B	0.0100	0.8824	0.0621	0.075*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.02293 (14)	0.02997 (17)	0.02613 (19)	0.000	0.00256 (12)	0.000
W2	0.02455 (10)	0.03619 (13)	0.02732 (13)	−0.00144 (9)	0.00412 (8)	0.00063 (10)
Cl1	0.0468 (7)	0.0413 (8)	0.0502 (10)	0.0111 (6)	0.0139 (7)	0.0082 (7)
Cl2	0.0292 (7)	0.0874 (12)	0.0414 (10)	−0.0116 (7)	−0.0003 (6)	0.0000 (8)
Cl3	0.0599 (9)	0.0359 (8)	0.0531 (11)	−0.0014 (7)	0.0215 (7)	0.0034 (7)
Cl4	0.0298 (6)	0.0512 (8)	0.0349 (9)	−0.0032 (6)	−0.0007 (6)	−0.0006 (7)
Cl5	0.0739 (12)	0.0853 (15)	0.120 (2)	0.0036 (11)	0.0277 (12)	0.0040 (13)
Cl6	0.0784 (11)	0.0629 (11)	0.0714 (14)	−0.0211 (9)	0.0153 (9)	−0.0163 (9)
Si1	0.0259 (7)	0.0428 (10)	0.0640 (13)	0.0041 (7)	0.0001 (7)	0.0170 (9)
N1	0.032 (2)	0.037 (3)	0.031 (3)	0.0010 (19)	0.005 (2)	0.005 (2)
N2	0.0173 (19)	0.048 (3)	0.027 (3)	−0.0022 (18)	−0.0010 (17)	−0.006 (2)
N3	0.024 (2)	0.036 (3)	0.027 (3)	−0.0002 (18)	0.0007 (18)	0.003 (2)
N4	0.029 (2)	0.047 (3)	0.029 (3)	0.001 (2)	0.0058 (19)	0.002 (2)
C1	0.052 (4)	0.064 (5)	0.109 (7)	0.021 (3)	0.018 (4)	0.000 (4)
C2	0.063 (4)	0.146 (7)	0.047 (5)	−0.004 (5)	−0.017 (4)	0.027 (5)
C3	0.051 (4)	0.047 (4)	0.176 (9)	0.005 (3)	0.021 (5)	0.033 (5)
C4	0.030 (3)	0.057 (4)	0.035 (4)	−0.008 (3)	−0.005 (2)	0.006 (3)
C5	0.040 (3)	0.064 (4)	0.043 (4)	−0.014 (3)	−0.003 (3)	−0.001 (3)
C6	0.064 (4)	0.049 (4)	0.053 (5)	−0.024 (3)	−0.009 (3)	0.004 (3)
C7	0.062 (4)	0.034 (3)	0.037 (4)	−0.008 (3)	0.006 (3)	−0.004 (3)
C8	0.039 (3)	0.038 (3)	0.032 (3)	−0.003 (2)	0.006 (3)	0.003 (3)
C9	0.033 (3)	0.045 (4)	0.045 (4)	−0.004 (2)	−0.004 (3)	−0.004 (3)
C10	0.039 (3)	0.068 (4)	0.029 (4)	0.008 (3)	0.002 (3)	−0.009 (3)

C11	0.036 (3)	0.084 (5)	0.030 (4)	0.010 (3)	0.013 (3)	0.016 (3)
C12	0.037 (3)	0.062 (4)	0.038 (4)	0.000 (3)	0.010 (3)	0.006 (3)
C13	0.031 (3)	0.053 (4)	0.043 (4)	−0.003 (3)	0.006 (3)	0.001 (3)
C14	0.081 (4)	0.042 (4)	0.067 (5)	−0.002 (3)	0.024 (4)	0.009 (3)

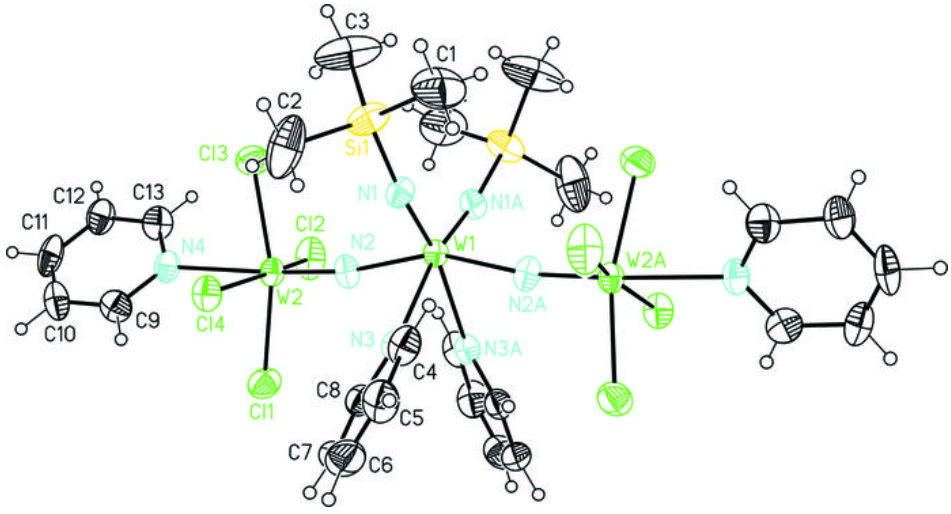
Geometric parameters (Å, °)

W1—N1^{i}	1.773 (4)	C2—H2B	0.9800
W1—N1	1.773 (4)	C2—H2C	0.9800
W1—N2	2.090 (4)	C3—H3A	0.9800
W1—N2^{i}	2.090 (4)	C3—H3B	0.9800
W1—N3^{i}	2.362 (4)	C3—H3C	0.9800
W1—N3	2.362 (4)	C4—C5	1.370 (7)
W2—N2	1.707 (4)	C4—H4A	0.9500
W2—Cl3	2.3174 (14)	C5—C6	1.356 (8)
W2—Cl2	2.3347 (12)	C5—H5A	0.9500
W2—Cl1	2.3475 (13)	C6—C7	1.382 (7)
W2—Cl4	2.3486 (12)	C6—H6A	0.9500
W2—N4	2.424 (4)	C7—C8	1.358 (6)
Cl5—C14	1.721 (6)	C7—H7A	0.9500
Cl6—C14	1.733 (6)	C8—H8A	0.9500
Si1—N1	1.730 (4)	C9—C10	1.390 (7)
Si1—C1	1.819 (6)	C9—H9A	0.9500
Si1—C2	1.831 (6)	C10—C11	1.373 (7)
Si1—C3	1.835 (6)	C10—H10A	0.9500
N3—C8	1.349 (6)	C11—C12	1.379 (8)
N3—C4	1.351 (6)	C11—H11A	0.9500
N4—C13	1.351 (6)	C12—C13	1.396 (7)
N4—C9	1.364 (6)	C12—H12A	0.9500
C1—H1A	0.9800	C13—H13A	0.9500
C1—H1B	0.9800	C14—H14A	0.9900
C1—H1C	0.9800	C14—H14B	0.9900
C2—H2A	0.9800		
N1^{i}—W1—N1	104.8 (2)	H1B—C1—H1C	109.5
N1^{i}—W1—N2	96.79 (16)	Si1—C2—H2A	109.5
N1—W1—N2	95.93 (16)	Si1—C2—H2B	109.5
N1^{i}—W1—N2^{i}	95.93 (17)	H2A—C2—H2B	109.5
N1—W1—N2^{i}	96.79 (16)	Si1—C2—H2C	109.5
N2—W1—N2^{i}	159.1 (2)	H2A—C2—H2C	109.5
N1^{i}—W1—N3^{i}	89.20 (15)	H2B—C2—H2C	109.5
N1—W1—N3^{i}	165.99 (15)	Si1—C3—H3A	109.5
N2—W1—N3^{i}	80.98 (14)	Si1—C3—H3B	109.5
N2^{i}—W1—N3^{i}	82.67 (14)	H3A—C3—H3B	109.5
N1^{i}—W1—N3	165.99 (15)	Si1—C3—H3C	109.5
N1—W1—N3	89.20 (15)	H3A—C3—H3C	109.5
N2—W1—N3	82.67 (14)	H3B—C3—H3C	109.5

$N2^{i}$—W1—N3	80.98 (14)	N3—C4—C5	123.4 (5)
$N3^{i}$—W1—N3	76.87 (18)	N3—C4—H4A	118.3
N2—W2—Cl3	95.54 (14)	C5—C4—H4A	118.3
N2—W2—Cl2	97.36 (11)	C6—C5—C4	119.3 (5)
Cl3—W2—Cl2	91.09 (5)	C6—C5—H5A	120.4
N2—W2—Cl1	99.47 (14)	C4—C5—H5A	120.4
Cl3—W2—Cl1	164.98 (5)	C5—C6—C7	119.6 (5)
Cl2—W2—Cl1	87.55 (5)	C5—C6—H6A	120.2
N2—W2—Cl4	95.10 (11)	C7—C6—H6A	120.2
Cl3—W2—Cl4	90.45 (5)	C8—C7—C6	117.5 (6)
Cl2—W2—Cl4	167.24 (5)	C8—C7—H7A	121.3
Cl1—W2—Cl4	87.69 (5)	C6—C7—H7A	121.3
N2—W2—N4	175.95 (16)	N3—C8—C7	125.2 (5)
Cl3—W2—N4	80.80 (10)	N3—C8—H8A	117.4
Cl2—W2—N4	84.49 (10)	C7—C8—H8A	117.4
Cl1—W2—N4	84.18 (10)	N4—C9—C10	122.6 (5)
Cl4—W2—N4	83.25 (10)	N4—C9—H9A	118.7
N1—Si1—C1	107.3 (3)	C10—C9—H9A	118.7
N1—Si1—C2	107.0 (3)	C11—C10—C9	119.1 (5)
C1—Si1—C2	111.1 (3)	C11—C10—H10A	120.5
N1—Si1—C3	109.0 (2)	C9—C10—H10A	120.5
C1—Si1—C3	111.6 (3)	C10—C11—C12	119.7 (6)
C2—Si1—C3	110.6 (3)	C10—C11—H11A	120.2
Si1—N1—W1	169.3 (3)	C12—C11—H11A	120.2
W2—N2—W1	169.2 (2)	C11—C12—C13	118.6 (6)
C8—N3—C4	115.1 (4)	C11—C12—H12A	120.7
C8—N3—W1	125.1 (3)	C13—C12—H12A	120.7
C4—N3—W1	119.8 (3)	N4—C13—C12	122.9 (5)
C13—N4—C9	117.1 (5)	N4—C13—H13A	118.5
C13—N4—W2	120.1 (4)	C12—C13—H13A	118.5
C9—N4—W2	122.8 (4)	Cl5—C14—Cl6	114.1 (3)
Si1—C1—H1A	109.5	Cl5—C14—H14A	108.7
Si1—C1—H1B	109.5	Cl6—C14—H14A	108.7
H1A—C1—H1B	109.5	Cl5—C14—H14B	108.7
Si1—C1—H1C	109.5	Cl6—C14—H14B	108.7
H1A—C1—H1C	109.5	H14A—C14—H14B	107.6

Symmetry codes: (i) $-x+1, y, -z+1/2$.

Fig. 1


C3
C1
C2
Si1
Cl3
N1
N1A
C12
C13
C11
Cl2
N2
W1
W2A
N4
W2
N2A
Cl4
C10
C9
N3
N3A
C4
C8
C5
Cl1
C7
C6