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Bis(dimethylamido-κN)bis[2-(pyrrol-2-ylmethyleneamino)-2′-methoxy-6,6′-dimethyl-1,1′-biphenyl-$\kappa^2 N,N'$]vanadium(IV) toluene monosolvate

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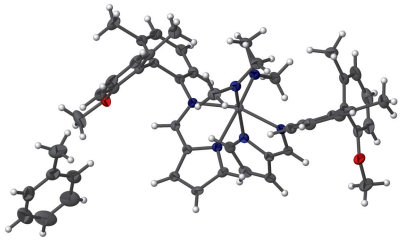
Keywords: crystal structure; vanadium(IV) complex; 2-amino-2′-methoxy-6,6′-dimethyl-1,1′-biphenyl; Schiff base ligand.

CCDC reference: 1502927

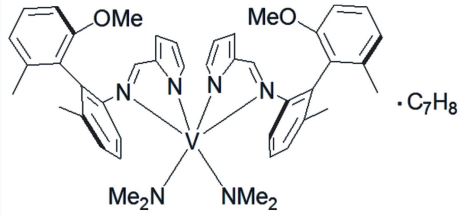
Structural data: full structural data are available from iucrdata.iucr.org

Reaction of the Schiff base ligand (*R*)-2-(pyrrol-2-ylmethyleneamino)-2′-methoxy-6,6′-dimethyl-1,1′-biphenyl with tetrakis(dimethylamido)vanadium in toluene gives the mononuclear V^{IV} title complex, $[V(C_2H_6N)_2(C_{20}H_{19}N_2O)_2]\cdot C_7H_8$, which was isolated as red crystals. The V^{IV} cation is coordinated by two monoanionic bidentate Schiff base ligands and two N atoms of two dimethylamide anions in a VN_6 distorted octahedral coordination geometry, in which two N atoms from the dimethylamide anions are placed in a *cis*-configuration.

3D view



Chemical scheme



Structure description

Crystals of the title compound were obtained by the reaction of the Schiff base ligand (*R*)-2-(pyrrol-2-ylmethyleneamino)-2′-methoxy-6,6′-dimethyl-1,1′-biphenyl and tetrakis(dimethylamido)vanadium in toluene during the synthesis of new vanadium catalysts. The title compound consists of a V^{IV} cation coordinated by two monoanionic bidentate Schiff base ligands and two N atoms of two dimethylamide anions, and one toluene solvent molecule. The V^{IV} coordination sphere can be described as a VN_6 distorted octahedron (Fig. 1), in which two N atoms from the dimethylamide anions are placed in *cis*-configuration. In the title complex, the N-donors from the Schiff base ligands are elongated [V—N = 2.1471 (18)–2.1678 (18) Å], whereas the N-donors from the dimethylamide anions have normal V—N bond lengths [1.8963 (19)–1.8993 (19) Å]. The titanium and zirconium complexes derived from the same Schiff base ligand have been reported, see: Zi *et al.* (2008).

Table 1
Experimental details.

Crystal data	
Chemical formula	$[V(C_2H_6N)_2(C_{20}H_{19}N_2O)_2]\cdot C_7H_8$
M_r	837.97
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	113
a, b, c (Å)	12.140 (1), 19.2980 (15), 19.4490 (16)
V (Å^3)	4556.5 (6)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	2.17
Crystal size (mm)	0.22 × 0.20 × 0.16
Data collection	
Diffractometer	Rigaku Saturn70 CCD camera
Absorption correction	Multi-scan (*CrystalClear-SM Expert*; Rigaku, 2009)
T_{min}, T_{max}	0.66, 0.75
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	49860, 8875, 8286
R_{int}	0.059
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.068, 1.02
No. of reflections	8875
No. of parameters	552
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.18, −0.25
Absolute structure	Flack x determined using 3251 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.0028 (19)

Computer programs: *CrystalClear-SM Expert* (Rigaku, 2009), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *CrystalStructure* (Rigaku, 2010) and *publCIF* (Westrip, 2010).

Synthesis and crystallization

A toluene solution (10 ml) of Schiff base ligand (*R*)-2-(pyrrol-2-ylmethyleneamino)-2′-methoxy-6,6′-dimethyl-1,1′-biphenyl (0.30 g, 1.0 mmol) was slowly added to a toluene solution (10 ml) of tetrakis(dimethylamido)vanadium (0.11 g, 0.5 mmol) with stirring at room temperature. After the resulting red solution had been stirred at room temperature for one day, the solution was filtered. The filtrate was concentrated to about 2 ml under vacuum. Red crystals of the title compound were isolated when this solution was kept at room temperature for three days. Yield: 0.33 g (78%).

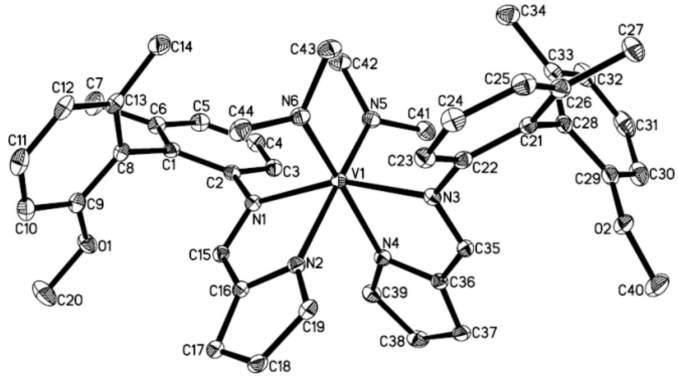


Figure 1
The asymmetric unit of the title compound showing 40% probability displacement ellipsoids (the hydrogen atoms and the solvent toluene molecule are omitted for clarity).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

We thank Professor Guofu Zi for providing the Schiff base ligand and for helpful discussions.

References

Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Rigaku (2009). *CrystalClear-SM Expert*. Rigaku Corporation, Tokyo, Japan.
Rigaku (2010). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* A**71**, 3–8.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zi, G., Wang, Q., Xiang, L. & Song, H. (2008). *Dalton Trans.* pp. 5930–5944.

full crystallographic data

IUCrData (2016). **1**, x161421 [doi:10.1107/S2414314616014218]

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Crystal data

$[V(C_2H_6N)_2(C_{20}H_{19}N_2O)_2]\cdot C_7H_8$
M_r = 837.97
Orthorhombic, $P2_12_12_1$
a = 12.140 (1) Å
b = 19.2980 (15) Å
c = 19.4490 (16) Å
V = 4556.5 (6) Å^3
Z = 4
$F(000)$ = 1780
D_x = 1.222 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54187 Å
Cell parameters from 5675 reflections
θ = 27.5–72.3°
μ = 2.17 mm^{-1}
T = 113 K
Prism, red
0.22 × 0.20 × 0.16 mm

Data collection

Rigaku Saturn70 CCD camera diffractometer
Radiation source: fine-focus sealed tube
Multilayer monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear-SM Expert*; Rigaku, 2009)
T_{min} = 0.66, T_{max} = 0.75
49860 measured reflections
8875 independent reflections
8286 reflections with $I > 2\sigma(I)$
R_{int} = 0.059
θ_{max} = 72.5°, θ_{min} = 3.2°
h = −11→14
k = −23→22
l = −24→23

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.068
S = 1.02
8875 reflections
552 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0253P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}
Absolute structure: Flack x determined using 3251 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.0028 (19)

Special details

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
V1	0.21029 (3)	0.22412 (2)	0.34762 (2)	0.01745 (9)
O1	0.27959 (16)	0.49262 (8)	0.20219 (8)	0.0300 (4)
O2	−0.01883 (14)	0.01342 (9)	0.50417 (9)	0.0318 (4)
N1	0.22032 (16)	0.31292 (9)	0.28127 (9)	0.0190 (4)
N2	0.16986 (16)	0.30961 (10)	0.41538 (9)	0.0202 (4)
N3	0.14730 (16)	0.15604 (9)	0.42558 (9)	0.0194 (4)
N4	0.03559 (16)	0.22780 (10)	0.33013 (9)	0.0205 (4)
N5	0.22971 (16)	0.15586 (10)	0.27833 (9)	0.0225 (4)
N6	0.35897 (16)	0.22403 (10)	0.37752 (9)	0.0227 (4)
C1	0.33105 (19)	0.35760 (12)	0.18341 (11)	0.0212 (5)
C2	0.24777 (19)	0.31447 (11)	0.20978 (11)	0.0202 (5)
C3	0.18745 (19)	0.27192 (12)	0.16559 (11)	0.0240 (5)
H3	0.1315	0.2427	0.1836	0.029*
C4	0.2087 (2)	0.27206 (12)	0.09597 (11)	0.0266 (5)
H4	0.1677	0.2427	0.0662	0.032*
C5	0.2895 (2)	0.31492 (12)	0.06947 (11)	0.0281 (5)
H5	0.3034	0.3150	0.0214	0.034*
C6	0.3509 (2)	0.35797 (12)	0.11232 (12)	0.0254 (5)
C7	0.4367 (2)	0.40532 (15)	0.08186 (13)	0.0377 (7)
H7A	0.4439	0.3958	0.0326	0.057*
H7B	0.5076	0.3973	0.1046	0.057*
H7C	0.4144	0.4536	0.0886	0.057*
C8	0.40022 (19)	0.40236 (12)	0.22961 (11)	0.0219 (5)
C9	0.3708 (2)	0.47248 (12)	0.23822 (12)	0.0254 (5)
C10	0.4315 (2)	0.51559 (13)	0.28132 (12)	0.0312 (6)
H10	0.4120	0.5630	0.2865	0.037*
C11	0.5209 (2)	0.48849 (15)	0.31650 (13)	0.0337 (6)
H11	0.5607	0.5171	0.3476	0.040*
C12	0.5531 (2)	0.42038 (14)	0.30702 (12)	0.0299 (6)
H12	0.6156	0.4029	0.3307	0.036*
C13	0.4937 (2)	0.37708 (13)	0.26256 (12)	0.0256 (5)
C14	0.5321 (2)	0.30421 (14)	0.25033 (14)	0.0344 (6)
H14A	0.5369	0.2956	0.2008	0.052*
H14B	0.4797	0.2717	0.2710	0.052*
H14C	0.6048	0.2978	0.2712	0.052*

C15	0.19212 (19)	0.37112 (11)	0.31069 (11)	0.0207 (5)
H15	0.1905	0.4128	0.2848	0.025*
C16	0.16427 (19)	0.37189 (11)	0.38074 (11)	0.0206 (5)
C17	0.1300 (2)	0.42506 (12)	0.42513 (12)	0.0244 (5)
H17	0.1192	0.4725	0.4139	0.029*
C18	0.1151 (2)	0.39398 (12)	0.48859 (12)	0.0259 (5)
H18	0.0927	0.4161	0.5299	0.031*
C19	0.1395 (2)	0.32343 (12)	0.48011 (11)	0.0230 (5)
H19	0.1351	0.2898	0.5157	0.028*
C20	0.2464 (3)	0.56310 (14)	0.20580 (17)	0.0485 (8)
H20A	0.3063	0.5928	0.1892	0.073*
H20B	0.2291	0.5751	0.2536	0.073*
H20C	0.1810	0.5701	0.1771	0.073*
C21	0.1974 (2)	0.04326 (11)	0.47941 (11)	0.0210 (5)
C22	0.2066 (2)	0.11598 (11)	0.47583 (10)	0.0210 (4)
C23	0.2746 (2)	0.15131 (12)	0.52159 (11)	0.0243 (5)
H23	0.2809	0.2003	0.5185	0.029*
C24	0.3332 (2)	0.11615 (13)	0.57141 (12)	0.0280 (5)
H24	0.3800	0.1405	0.6022	0.034*
C25	0.3227 (2)	0.04441 (13)	0.57602 (12)	0.0282 (5)
H25	0.3623	0.0201	0.6106	0.034*
C26	0.2560 (2)	0.00801 (12)	0.53132 (12)	0.0250 (5)
C27	0.2482 (2)	−0.07008 (13)	0.53692 (14)	0.0338 (6)
H27A	0.2879	−0.0857	0.5780	0.051*
H27B	0.2809	−0.0914	0.4960	0.051*
H27C	0.1706	−0.0838	0.5404	0.051*
C28	0.1304 (2)	0.00239 (11)	0.42885 (12)	0.0222 (5)
C29	0.0185 (2)	−0.01111 (12)	0.44244 (13)	0.0275 (5)
C30	−0.0460 (2)	−0.04779 (14)	0.39565 (15)	0.0367 (6)
H30	−0.1221	−0.0554	0.4044	0.044*
C31	0.0019 (2)	−0.07291 (13)	0.33646 (15)	0.0399 (7)
H31	−0.0419	−0.0973	0.3040	0.048*
C32	0.1125 (2)	−0.06315 (13)	0.32358 (13)	0.0343 (6)
H32	0.1444	−0.0820	0.2831	0.041*
C33	0.1780 (2)	−0.02576 (12)	0.36964 (12)	0.0260 (5)
C34	0.2985 (2)	−0.01619 (13)	0.35456 (13)	0.0336 (6)
H34A	0.3087	0.0255	0.3265	0.050*
H34B	0.3262	−0.0567	0.3295	0.050*
H34C	0.3390	−0.0111	0.3978	0.050*
C35	0.0396 (2)	0.15476 (11)	0.42837 (11)	0.0209 (5)
H35	0.0035	0.1284	0.4630	0.025*
C36	−0.02257 (19)	0.19223 (11)	0.38036 (11)	0.0204 (5)
C37	−0.1364 (2)	0.20096 (12)	0.37040 (12)	0.0260 (5)
H37	−0.1940	0.1823	0.3978	0.031*
C38	−0.1474 (2)	0.24234 (12)	0.31248 (13)	0.0293 (6)
H38	−0.2145	0.2572	0.2921	0.035*
C39	−0.0406 (2)	0.25794 (12)	0.28985 (12)	0.0246 (5)
H39	−0.0242	0.2861	0.2511	0.030*

C40	−0.1352 (2)	0.01588 (17)	0.51440 (18)	0.0470 (8)
H40A	−0.1649	−0.0313	0.5145	0.071*
H40B	−0.1694	0.0425	0.4772	0.071*
H40C	−0.1513	0.0382	0.5586	0.071*
C41	0.1426 (2)	0.10869 (12)	0.25604 (12)	0.0282 (5)
H41A	0.1742	0.0629	0.2469	0.042*
H41B	0.1084	0.1267	0.2140	0.042*
H41C	0.0868	0.1049	0.2923	0.042*
C42	0.3251 (2)	0.14854 (13)	0.23374 (12)	0.0296 (6)
H42A	0.3023	0.1537	0.1857	0.044*
H42B	0.3580	0.1026	0.2404	0.044*
H42C	0.3795	0.1843	0.2451	0.044*
C43	0.4299 (2)	0.16340 (14)	0.38204 (13)	0.0299 (5)
H43A	0.4461	0.1535	0.4304	0.045*
H43B	0.4987	0.1723	0.3573	0.045*
H43C	0.3924	0.1235	0.3614	0.045*
C44	0.4124 (2)	0.28268 (14)	0.41150 (12)	0.0299 (5)
H44A	0.4881	0.2872	0.3946	0.045*
H44B	0.4135	0.2749	0.4613	0.045*
H44C	0.3714	0.3252	0.4014	0.045*
C45	0.4571 (3)	0.6708 (2)	0.42065 (18)	0.0586 (9)
H45A	0.4767	0.6323	0.3902	0.088*
H45B	0.4639	0.6560	0.4687	0.088*
H45C	0.5068	0.7099	0.4122	0.088*
C46	0.3405 (3)	0.69278 (16)	0.40673 (14)	0.0414 (7)
C47	0.3080 (3)	0.76147 (15)	0.41459 (14)	0.0500 (9)
H47	0.3612	0.7955	0.4265	0.060*
C48	0.1999 (4)	0.78084 (19)	0.40534 (15)	0.0605 (10)
H48	0.1790	0.8278	0.4121	0.073*
C49	0.1226 (4)	0.7331 (2)	0.38658 (16)	0.0679 (12)
H49	0.0481	0.7468	0.3801	0.081*
C50	0.1529 (3)	0.6649 (2)	0.37704 (15)	0.0554 (9)
H50	0.0994	0.6317	0.3632	0.066*
C51	0.2618 (3)	0.64452 (15)	0.38760 (13)	0.0405 (7)
H51	0.2821	0.5974	0.3816	0.049*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
V1	0.01700 (19)	0.01853 (17)	0.01684 (16)	0.00097 (15)	−0.00101 (15)	−0.00021 (15)
O1	0.0339 (10)	0.0216 (8)	0.0346 (9)	0.0034 (8)	−0.0082 (8)	0.0014 (7)
O2	0.0197 (10)	0.0331 (10)	0.0427 (10)	0.0006 (7)	0.0072 (7)	0.0081 (8)
N1	0.0183 (10)	0.0209 (9)	0.0177 (8)	0.0000 (8)	−0.0006 (8)	0.0014 (7)
N2	0.0185 (10)	0.0211 (9)	0.0211 (9)	−0.0002 (7)	0.0003 (7)	−0.0021 (7)
N3	0.0206 (11)	0.0182 (9)	0.0193 (9)	0.0012 (7)	−0.0013 (8)	0.0000 (7)
N4	0.0191 (10)	0.0201 (9)	0.0224 (9)	0.0004 (8)	−0.0025 (7)	−0.0012 (7)
N5	0.0231 (11)	0.0230 (9)	0.0215 (9)	0.0024 (8)	−0.0013 (8)	−0.0005 (7)
N6	0.0208 (11)	0.0253 (10)	0.0220 (9)	0.0016 (9)	−0.0018 (7)	0.0004 (8)

C1	0.0183 (12)	0.0229 (12)	0.0223 (11)	0.0023 (9)	0.0002 (9)	0.0012 (8)
C2	0.0197 (12)	0.0205 (11)	0.0203 (11)	0.0037 (8)	−0.0022 (8)	0.0010 (8)
C3	0.0236 (13)	0.0237 (11)	0.0247 (11)	0.0000 (10)	−0.0026 (9)	0.0017 (9)
C4	0.0292 (13)	0.0275 (11)	0.0230 (10)	−0.0004 (11)	−0.0045 (10)	−0.0032 (9)
C5	0.0320 (14)	0.0339 (13)	0.0185 (10)	0.0016 (11)	0.0014 (10)	−0.0009 (9)
C6	0.0250 (14)	0.0279 (13)	0.0234 (11)	0.0006 (10)	0.0001 (10)	0.0006 (9)
C7	0.0409 (18)	0.0458 (16)	0.0264 (13)	−0.0114 (13)	0.0071 (12)	0.0026 (11)
C8	0.0208 (13)	0.0250 (12)	0.0200 (11)	−0.0042 (9)	0.0009 (9)	0.0021 (8)
C9	0.0253 (13)	0.0275 (12)	0.0234 (11)	−0.0045 (10)	0.0006 (10)	0.0027 (9)
C10	0.0361 (16)	0.0277 (13)	0.0297 (13)	−0.0089 (11)	0.0026 (11)	−0.0015 (10)
C11	0.0346 (16)	0.0416 (15)	0.0248 (12)	−0.0166 (12)	−0.0001 (11)	−0.0012 (11)
C12	0.0210 (14)	0.0438 (15)	0.0250 (12)	−0.0105 (11)	−0.0023 (10)	0.0065 (10)
C13	0.0206 (13)	0.0336 (13)	0.0225 (11)	−0.0055 (10)	0.0019 (9)	0.0046 (9)
C14	0.0246 (14)	0.0375 (14)	0.0411 (15)	0.0021 (11)	−0.0049 (11)	0.0033 (11)
C15	0.0165 (13)	0.0211 (11)	0.0244 (11)	−0.0005 (9)	−0.0017 (9)	0.0026 (9)
C16	0.0160 (12)	0.0211 (11)	0.0246 (11)	−0.0011 (9)	0.0004 (9)	−0.0008 (9)
C17	0.0197 (13)	0.0225 (11)	0.0312 (12)	−0.0020 (9)	0.0034 (10)	−0.0027 (9)
C18	0.0215 (13)	0.0292 (12)	0.0269 (12)	−0.0043 (10)	0.0062 (10)	−0.0095 (9)
C19	0.0199 (13)	0.0282 (12)	0.0208 (11)	−0.0040 (9)	0.0024 (9)	0.0003 (9)
C20	0.059 (2)	0.0257 (14)	0.0605 (19)	0.0089 (13)	−0.0165 (16)	−0.0023 (13)
C21	0.0185 (13)	0.0227 (11)	0.0218 (11)	0.0016 (9)	0.0031 (9)	0.0013 (8)
C22	0.0200 (12)	0.0249 (11)	0.0181 (10)	0.0017 (10)	0.0034 (9)	0.0023 (8)
C23	0.0266 (14)	0.0246 (12)	0.0217 (11)	0.0012 (10)	0.0002 (9)	−0.0012 (9)
C24	0.0281 (14)	0.0327 (13)	0.0231 (11)	−0.0009 (10)	−0.0038 (10)	−0.0034 (10)
C25	0.0264 (14)	0.0349 (14)	0.0232 (11)	0.0055 (10)	−0.0029 (10)	0.0048 (9)
C26	0.0245 (13)	0.0274 (12)	0.0232 (11)	0.0042 (9)	0.0033 (9)	0.0041 (9)
C27	0.0400 (16)	0.0264 (13)	0.0351 (13)	0.0034 (11)	−0.0049 (11)	0.0079 (10)
C28	0.0216 (13)	0.0184 (11)	0.0265 (11)	0.0002 (9)	−0.0029 (9)	0.0041 (8)
C29	0.0245 (14)	0.0206 (12)	0.0374 (13)	0.0004 (10)	−0.0023 (10)	0.0068 (10)
C30	0.0276 (16)	0.0284 (14)	0.0541 (17)	−0.0057 (11)	−0.0124 (13)	0.0081 (12)
C31	0.0474 (18)	0.0238 (13)	0.0485 (17)	−0.0047 (12)	−0.0235 (14)	0.0006 (11)
C32	0.0481 (19)	0.0233 (13)	0.0315 (13)	0.0009 (12)	−0.0092 (12)	−0.0013 (10)
C33	0.0312 (15)	0.0193 (11)	0.0276 (11)	0.0040 (9)	−0.0035 (9)	0.0028 (9)
C34	0.0342 (15)	0.0367 (13)	0.0299 (12)	0.0047 (12)	0.0054 (12)	−0.0028 (10)
C35	0.0210 (13)	0.0186 (11)	0.0232 (11)	−0.0018 (9)	0.0030 (9)	−0.0024 (8)
C36	0.0198 (13)	0.0184 (11)	0.0231 (11)	0.0001 (9)	−0.0001 (9)	−0.0032 (8)
C37	0.0184 (13)	0.0236 (12)	0.0358 (13)	0.0000 (9)	0.0031 (10)	−0.0059 (9)
C38	0.0193 (14)	0.0313 (14)	0.0373 (13)	0.0061 (10)	−0.0077 (10)	−0.0047 (10)
C39	0.0256 (14)	0.0244 (12)	0.0240 (11)	0.0031 (9)	−0.0047 (10)	0.0008 (9)
C40	0.0236 (16)	0.0528 (18)	0.065 (2)	0.0028 (13)	0.0131 (14)	0.0157 (15)
C41	0.0333 (15)	0.0236 (12)	0.0276 (12)	−0.0003 (10)	−0.0036 (11)	−0.0047 (9)
C42	0.0318 (15)	0.0331 (13)	0.0238 (11)	0.0071 (11)	0.0020 (10)	−0.0031 (10)
C43	0.0219 (14)	0.0377 (14)	0.0301 (13)	0.0064 (11)	−0.0029 (10)	0.0028 (10)
C44	0.0223 (13)	0.0367 (14)	0.0306 (12)	−0.0051 (11)	−0.0037 (10)	−0.0051 (11)
C45	0.043 (2)	0.074 (2)	0.058 (2)	0.0005 (17)	0.0117 (16)	−0.0063 (17)
C46	0.0487 (19)	0.0467 (17)	0.0287 (14)	0.0052 (14)	0.0112 (13)	0.0031 (11)
C47	0.080 (3)	0.0380 (17)	0.0320 (14)	−0.0026 (16)	0.0139 (15)	0.0080 (11)
C48	0.095 (3)	0.051 (2)	0.0358 (15)	0.031 (2)	0.0007 (18)	0.0118 (14)

C49	0.074 (3)	0.099 (3)	0.0309 (16)	0.045 (3)	−0.0140 (16)	−0.0013 (18)
C50	0.055 (2)	0.078 (2)	0.0326 (15)	0.0082 (19)	−0.0114 (14)	−0.0125 (15)
C51	0.053 (2)	0.0414 (16)	0.0269 (13)	0.0078 (13)	−0.0004 (12)	−0.0056 (11)

Geometric parameters (Å, º)

V1—N6	1.8963 (19)	C22—C23	1.393 (3)
V1—N5	1.8993 (19)	C23—C24	1.380 (3)
V1—N3	2.1471 (18)	C23—H23	0.9500
V1—N1	2.1487 (17)	C24—C25	1.393 (4)
V1—N4	2.1491 (19)	C24—H24	0.9500
V1—N2	2.1678 (18)	C25—C26	1.381 (4)
O1—C9	1.367 (3)	C25—H25	0.9500
O1—C20	1.420 (3)	C26—C27	1.514 (4)
O2—C29	1.368 (3)	C27—H27A	0.9800
O2—C40	1.428 (3)	C27—H27B	0.9800
N1—C15	1.306 (3)	C27—H27C	0.9800
N1—C2	1.430 (3)	C28—C33	1.398 (3)
N2—C19	1.339 (3)	C28—C29	1.409 (4)
N2—C16	1.379 (3)	C29—C30	1.394 (4)
N3—C35	1.309 (3)	C30—C31	1.378 (4)
N3—C22	1.439 (3)	C30—H30	0.9500
N4—C39	1.344 (3)	C31—C32	1.378 (4)
N4—C36	1.387 (3)	C31—H31	0.9500
N5—C42	1.454 (3)	C32—C33	1.398 (3)
N5—C41	1.461 (3)	C32—H32	0.9500
N6—C43	1.455 (3)	C33—C34	1.503 (4)
N6—C44	1.462 (3)	C34—H34A	0.9800
C1—C6	1.404 (3)	C34—H34B	0.9800
C1—C2	1.406 (3)	C34—H34C	0.9800
C1—C8	1.503 (3)	C35—C36	1.401 (3)
C2—C3	1.396 (3)	C35—H35	0.9500
C3—C4	1.378 (3)	C36—C37	1.405 (3)
C3—H3	0.9500	C37—C38	1.387 (4)
C4—C5	1.383 (4)	C37—H37	0.9500
C4—H4	0.9500	C38—C39	1.403 (4)
C5—C6	1.393 (3)	C38—H38	0.9500
C5—H5	0.9500	C39—H39	0.9500
C6—C7	1.507 (4)	C40—H40A	0.9800
C7—H7A	0.9800	C40—H40B	0.9800
C7—H7B	0.9800	C40—H40C	0.9800
C7—H7C	0.9800	C41—H41A	0.9800
C8—C13	1.391 (3)	C41—H41B	0.9800
C8—C9	1.410 (3)	C41—H41C	0.9800
C9—C10	1.392 (3)	C42—H42A	0.9800
C10—C11	1.385 (4)	C42—H42B	0.9800
C10—H10	0.9500	C42—H42C	0.9800
C11—C12	1.384 (4)	C43—H43A	0.9800

C11—H11	0.9500	C43—H43B	0.9800
C12—C13	1.402 (3)	C43—H43C	0.9800
C12—H12	0.9500	C44—H44A	0.9800
C13—C14	1.501 (4)	C44—H44B	0.9800
C14—H14A	0.9800	C44—H44C	0.9800
C14—H14B	0.9800	C45—C46	1.503 (5)
C14—H14C	0.9800	C45—H45A	0.9800
C15—C16	1.404 (3)	C45—H45B	0.9800
C15—H15	0.9500	C45—H45C	0.9800
C16—C17	1.404 (3)	C46—C51	1.385 (4)
C17—C18	1.384 (3)	C46—C47	1.391 (4)
C17—H17	0.9500	C47—C48	1.376 (6)
C18—C19	1.403 (3)	C47—H47	0.9500
C18—H18	0.9500	C48—C49	1.365 (6)
C19—H19	0.9500	C48—H48	0.9500
C20—H20A	0.9800	C49—C50	1.380 (5)
C20—H20B	0.9800	C49—H49	0.9500
C20—H20C	0.9800	C50—C51	1.395 (5)
C21—C22	1.410 (3)	C50—H50	0.9500
C21—C26	1.410 (3)	C51—H51	0.9500
C21—C28	1.500 (3)		

N6—V1—N5	95.67 (8)	C24—C23—H23	119.6
N6—V1—N3	97.00 (8)	C22—C23—H23	119.6
N5—V1—N3	96.95 (8)	C23—C24—C25	119.2 (2)
N6—V1—N1	97.53 (8)	C23—C24—H24	120.4
N5—V1—N1	96.88 (7)	C25—C24—H24	120.4
N3—V1—N1	158.81 (7)	C26—C25—C24	121.2 (2)
N6—V1—N4	171.07 (7)	C26—C25—H25	119.4
N5—V1—N4	91.89 (8)	C24—C25—H25	119.4
N3—V1—N4	77.32 (7)	C25—C26—C21	120.1 (2)
N1—V1—N4	86.25 (7)	C25—C26—C27	119.9 (2)
N6—V1—N2	91.71 (8)	C21—C26—C27	120.0 (2)
N5—V1—N2	170.87 (8)	C26—C27—H27A	109.5
N3—V1—N2	87.47 (7)	C26—C27—H27B	109.5
N1—V1—N2	76.76 (7)	H27A—C27—H27B	109.5
N4—V1—N2	81.24 (7)	C26—C27—H27C	109.5
C9—O1—C20	118.5 (2)	H27A—C27—H27C	109.5
C29—O2—C40	117.5 (2)	H27B—C27—H27C	109.5
C15—N1—C2	117.97 (18)	C33—C28—C29	118.7 (2)
C15—N1—V1	114.06 (14)	C33—C28—C21	121.4 (2)
C2—N1—V1	127.87 (14)	C29—C28—C21	119.8 (2)
C19—N2—C16	105.80 (18)	O2—C29—C30	124.3 (2)
C19—N2—V1	141.78 (16)	O2—C29—C28	114.8 (2)
C16—N2—V1	112.17 (14)	C30—C29—C28	120.9 (2)
C35—N3—C22	117.48 (19)	C31—C30—C29	119.1 (3)
C35—N3—V1	113.36 (15)	C31—C30—H30	120.4
C22—N3—V1	129.06 (15)	C29—C30—H30	120.4

C39—N4—C36	105.93 (19)	C32—C31—C30	121.0 (2)
C39—N4—V1	141.74 (16)	C32—C31—H31	119.5
C36—N4—V1	112.00 (14)	C30—C31—H31	119.5
C42—N5—C41	109.82 (18)	C31—C32—C33	120.5 (3)
C42—N5—V1	126.13 (16)	C31—C32—H32	119.7
C41—N5—V1	123.56 (15)	C33—C32—H32	119.7
C43—N6—C44	109.4 (2)	C28—C33—C32	119.6 (2)
C43—N6—V1	125.61 (16)	C28—C33—C34	121.0 (2)
C44—N6—V1	124.06 (16)	C32—C33—C34	119.4 (2)
C6—C1—C2	119.0 (2)	C33—C34—H34A	109.5
C6—C1—C8	119.4 (2)	C33—C34—H34B	109.5
C2—C1—C8	121.59 (19)	H34A—C34—H34B	109.5
C3—C2—C1	120.0 (2)	C33—C34—H34C	109.5
C3—C2—N1	117.6 (2)	H34A—C34—H34C	109.5
C1—C2—N1	122.31 (19)	H34B—C34—H34C	109.5
C4—C3—C2	120.4 (2)	N3—C35—C36	120.0 (2)
C4—C3—H3	119.8	N3—C35—H35	120.0
C2—C3—H3	119.8	C36—C35—H35	120.0
C3—C4—C5	120.0 (2)	N4—C36—C35	116.8 (2)
C3—C4—H4	120.0	N4—C36—C37	110.1 (2)
C5—C4—H4	120.0	C35—C36—C37	133.1 (2)
C4—C5—C6	120.9 (2)	C38—C37—C36	106.0 (2)
C4—C5—H5	119.6	C38—C37—H37	127.0
C6—C5—H5	119.6	C36—C37—H37	127.0
C5—C6—C1	119.7 (2)	C37—C38—C39	106.8 (2)
C5—C6—C7	119.8 (2)	C37—C38—H38	126.6
C1—C6—C7	120.6 (2)	C39—C38—H38	126.6
C6—C7—H7A	109.5	N4—C39—C38	111.1 (2)
C6—C7—H7B	109.5	N4—C39—H39	124.4
H7A—C7—H7B	109.5	C38—C39—H39	124.4
C6—C7—H7C	109.5	O2—C40—H40A	109.5
H7A—C7—H7C	109.5	O2—C40—H40B	109.5
H7B—C7—H7C	109.5	H40A—C40—H40B	109.5
C13—C8—C9	119.2 (2)	O2—C40—H40C	109.5
C13—C8—C1	122.0 (2)	H40A—C40—H40C	109.5
C9—C8—C1	118.8 (2)	H40B—C40—H40C	109.5
O1—C9—C10	124.6 (2)	N5—C41—H41A	109.5
O1—C9—C8	114.7 (2)	N5—C41—H41B	109.5
C10—C9—C8	120.7 (2)	H41A—C41—H41B	109.5
C11—C10—C9	119.1 (2)	N5—C41—H41C	109.5
C11—C10—H10	120.4	H41A—C41—H41C	109.5
C9—C10—H10	120.4	H41B—C41—H41C	109.5
C12—C11—C10	120.9 (2)	N5—C42—H42A	109.5
C12—C11—H11	119.5	N5—C42—H42B	109.5
C10—C11—H11	119.5	H42A—C42—H42B	109.5
C11—C12—C13	120.2 (2)	N5—C42—H42C	109.5
C11—C12—H12	119.9	H42A—C42—H42C	109.5
C13—C12—H12	119.9	H42B—C42—H42C	109.5

C8—C13—C12	119.7 (2)	N6—C43—H43A	109.5
C8—C13—C14	120.6 (2)	N6—C43—H43B	109.5
C12—C13—C14	119.7 (2)	H43A—C43—H43B	109.5
C13—C14—H14A	109.5	N6—C43—H43C	109.5
C13—C14—H14B	109.5	H43A—C43—H43C	109.5
H14A—C14—H14B	109.5	H43B—C43—H43C	109.5
C13—C14—H14C	109.5	N6—C44—H44A	109.5
H14A—C14—H14C	109.5	N6—C44—H44B	109.5
H14B—C14—H14C	109.5	H44A—C44—H44B	109.5
N1—C15—C16	119.8 (2)	N6—C44—H44C	109.5
N1—C15—H15	120.1	H44A—C44—H44C	109.5
C16—C15—H15	120.1	H44B—C44—H44C	109.5
N2—C16—C15	116.9 (2)	C46—C45—H45A	109.5
N2—C16—C17	110.55 (19)	C46—C45—H45B	109.5
C15—C16—C17	132.5 (2)	H45A—C45—H45B	109.5
C18—C17—C16	105.7 (2)	C46—C45—H45C	109.5
C18—C17—H17	127.2	H45A—C45—H45C	109.5
C16—C17—H17	127.2	H45B—C45—H45C	109.5
C17—C18—C19	106.7 (2)	C51—C46—C47	118.4 (3)
C17—C18—H18	126.6	C51—C46—C45	120.6 (3)
C19—C18—H18	126.6	C47—C46—C45	121.0 (3)
N2—C19—C18	111.2 (2)	C48—C47—C46	121.0 (3)
N2—C19—H19	124.4	C48—C47—H47	119.5
C18—C19—H19	124.4	C46—C47—H47	119.5
O1—C20—H20A	109.5	C49—C48—C47	120.5 (3)
O1—C20—H20B	109.5	C49—C48—H48	119.7
H20A—C20—H20B	109.5	C47—C48—H48	119.7
O1—C20—H20C	109.5	C48—C49—C50	119.7 (4)
H20A—C20—H20C	109.5	C48—C49—H49	120.1
H20B—C20—H20C	109.5	C50—C49—H49	120.1
C22—C21—C26	118.4 (2)	C49—C50—C51	120.1 (4)
C22—C21—C28	122.3 (2)	C49—C50—H50	119.9
C26—C21—C28	119.28 (19)	C51—C50—H50	119.9
C23—C22—C21	120.2 (2)	C46—C51—C50	120.2 (3)
C23—C22—N3	117.90 (19)	C46—C51—H51	119.9
C21—C22—N3	121.9 (2)	C50—C51—H51	119.9
C24—C23—C22	120.9 (2)		

N6—V1—N5—C42	−37.95 (19)	C16—C17—C18—C19	−0.7 (3)
N3—V1—N5—C42	−135.74 (18)	C16—N2—C19—C18	−0.6 (3)
N1—V1—N5—C42	60.36 (19)	V1—N2—C19—C18	−173.84 (19)
N4—V1—N5—C42	146.81 (18)	C17—C18—C19—N2	0.8 (3)
N6—V1—N5—C41	150.91 (18)	C26—C21—C22—C23	1.8 (3)
N3—V1—N5—C41	53.12 (18)	C28—C21—C22—C23	−176.4 (2)
N1—V1—N5—C41	−110.78 (18)	C26—C21—C22—N3	−178.3 (2)
N4—V1—N5—C41	−24.33 (18)	C28—C21—C22—N3	3.5 (3)
N5—V1—N6—C43	−37.05 (19)	C35—N3—C22—C23	−117.3 (2)
N3—V1—N6—C43	60.69 (18)	V1—N3—C22—C23	58.9 (3)

N1—V1—N6—C43	−134.78 (18)	C35—N3—C22—C21	62.8 (3)
N2—V1—N6—C43	148.34 (18)	V1—N3—C22—C21	−121.0 (2)
N5—V1—N6—C44	154.99 (17)	C21—C22—C23—C24	−0.6 (4)
N3—V1—N6—C44	−107.27 (18)	N3—C22—C23—C24	179.5 (2)
N1—V1—N6—C44	57.26 (18)	C22—C23—C24—C25	−0.6 (4)
N2—V1—N6—C44	−19.62 (18)	C23—C24—C25—C26	0.7 (4)
C6—C1—C2—C3	1.1 (3)	C24—C25—C26—C21	0.5 (4)
C8—C1—C2—C3	−177.9 (2)	C24—C25—C26—C27	178.8 (2)
C6—C1—C2—N1	−177.9 (2)	C22—C21—C26—C25	−1.7 (3)
C8—C1—C2—N1	3.0 (3)	C28—C21—C26—C25	176.5 (2)
C15—N1—C2—C3	−122.5 (2)	C22—C21—C26—C27	179.9 (2)
V1—N1—C2—C3	53.7 (3)	C28—C21—C26—C27	−1.8 (3)
C15—N1—C2—C1	56.5 (3)	C22—C21—C28—C33	92.8 (3)
V1—N1—C2—C1	−127.2 (2)	C26—C21—C28—C33	−85.4 (3)
C1—C2—C3—C4	−0.3 (3)	C22—C21—C28—C29	−90.3 (3)
N1—C2—C3—C4	178.8 (2)	C26—C21—C28—C29	91.5 (3)
C2—C3—C4—C5	−0.5 (4)	C40—O2—C29—C30	−15.1 (3)
C3—C4—C5—C6	0.4 (4)	C40—O2—C29—C28	165.9 (2)
C4—C5—C6—C1	0.4 (4)	C33—C28—C29—O2	174.7 (2)
C4—C5—C6—C7	−178.6 (2)	C21—C28—C29—O2	−2.2 (3)
C2—C1—C6—C5	−1.2 (4)	C33—C28—C29—C30	−4.3 (3)
C8—C1—C6—C5	177.9 (2)	C21—C28—C29—C30	178.7 (2)
C2—C1—C6—C7	177.9 (2)	O2—C29—C30—C31	−176.9 (2)
C8—C1—C6—C7	−3.1 (4)	C28—C29—C30—C31	2.1 (4)
C6—C1—C8—C13	−94.4 (3)	C29—C30—C31—C32	1.0 (4)
C2—C1—C8—C13	84.7 (3)	C30—C31—C32—C33	−1.7 (4)
C6—C1—C8—C9	84.0 (3)	C29—C28—C33—C32	3.5 (3)
C2—C1—C8—C9	−97.0 (3)	C21—C28—C33—C32	−179.6 (2)
C20—O1—C9—C10	3.2 (4)	C29—C28—C33—C34	−176.7 (2)
C20—O1—C9—C8	−177.7 (2)	C21—C28—C33—C34	0.2 (3)
C13—C8—C9—O1	178.2 (2)	C31—C32—C33—C28	−0.6 (4)
C1—C8—C9—O1	−0.2 (3)	C31—C32—C33—C34	179.7 (2)
C13—C8—C9—C10	−2.6 (3)	C22—N3—C35—C36	−179.39 (19)
C1—C8—C9—C10	179.0 (2)	V1—N3—C35—C36	3.8 (3)
O1—C9—C10—C11	178.3 (2)	C39—N4—C36—C35	178.62 (19)
C8—C9—C10—C11	−0.8 (4)	V1—N4—C36—C35	−6.5 (2)
C9—C10—C11—C12	2.8 (4)	C39—N4—C36—C37	0.0 (2)
C10—C11—C12—C13	−1.5 (4)	V1—N4—C36—C37	174.89 (15)
C9—C8—C13—C12	3.9 (3)	N3—C35—C36—N4	1.9 (3)
C1—C8—C13—C12	−177.8 (2)	N3—C35—C36—C37	−179.9 (2)
C9—C8—C13—C14	−175.4 (2)	N4—C36—C37—C38	0.5 (3)
C1—C8—C13—C14	2.9 (3)	C35—C36—C37—C38	−177.9 (2)
C11—C12—C13—C8	−1.9 (3)	C36—C37—C38—C39	−0.7 (3)
C11—C12—C13—C14	177.4 (2)	C36—N4—C39—C38	−0.4 (3)
C2—N1—C15—C16	179.8 (2)	V1—N4—C39—C38	−172.80 (18)
V1—N1—C15—C16	3.1 (3)	C37—C38—C39—N4	0.7 (3)
C19—N2—C16—C15	−179.7 (2)	C51—C46—C47—C48	1.6 (4)
V1—N2—C16—C15	−4.2 (2)	C45—C46—C47—C48	−176.5 (3)

C19—N2—C16—C17	0.1 (3)	C46—C47—C48—C49	−1.6 (4)
V1—N2—C16—C17	175.62 (16)	C47—C48—C49—C50	0.2 (5)
N1—C15—C16—N2	0.8 (3)	C48—C49—C50—C51	1.0 (5)
N1—C15—C16—C17	−179.0 (2)	C47—C46—C51—C50	−0.4 (4)
N2—C16—C17—C18	0.4 (3)	C45—C46—C51—C50	177.8 (3)
C15—C16—C17—C18	−179.8 (2)	C49—C50—C51—C46	−0.9 (5)