Supporting Information

Synthesis, Structural Analysis for (Arylimido)vanadium(V) Complexes Containing Phenoxy-imine Ligands: New Efficient Catalyst Precursors for Ethylene Polymerization

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X-ray Structure Report for VCl_2(N-2,6-$Me_2C_6H_3$)[O-2-{(2,6-$^{i}Pr_2C_6H_3$)N=CH}C_6H_4] (**1a**)

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Experimental

Data Collection

A black block crystal of $C_{27}H_{31}Cl_2N_2OV$ having approximate dimensions of 0.20 x 0.15 x 0.07 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 540 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

a = 19.4008(14) Å
b = 9.8601(6) Å β = 121.044(2)^o
c = 16.1374(8) Å
V = 2644.8(3) Å^3

For Z = 4 and F.W. = 521.40, the calculated density is 1.309 g/cm^3. Based on the systematic absences of:

hkl: h+k ± 2n
h0l: l ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

Cc (#9)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 55 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 4.0^o step, at χ=45.0^o and ϕ = 0.0^o. The exposure rate was 280.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 4.0^o step, at χ=45.0^o and ϕ = 180.0^o. The exposure rate was 280.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 12707 reflections that were collected, 3005 were unique (R_{int} = 0.070); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 5.980 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.465 to 0.959. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 1736 observed reflections and 329 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0323$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.0560$$

The standard deviation of an observation of unit weight[4] was 1.02. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.22 and -0.26 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{27}H_{31}Cl_2N_2OV$
Formula Weight	521.40
Crystal Color, Habit	black, block
Crystal Dimensions	0.20 X 0.15 X 0.07 mm
Crystal System	monoclinic
Lattice Type	C-centered
Indexing Images	3 oscillations @ 540.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 19.4008(14) Å b = 9.8601(6) Å c = 16.1374(8) Å β = 121.044(2) ^{o} V = 2644.8(3) $Å^3$
Space Group	Cc (#9)
Z value	4
D_{calc}	1.309 g/cm^3
F_{000}	1088.00
μ(MoKα)	5.980 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	55 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	280.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 160.0^{o}
Exposure Rate	280.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 12707 Unique: 3005 (R_{int} = 0.070)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.465 - 0.959)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.9000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations ($I>2.00\sigma(I)$)	1736
No. Variables	329
Reflection/Parameter Ratio	5.28
Residuals: R1 ($I>2.00\sigma(I)$)	0.0323
Residuals: wR2 ($I>2.00\sigma(I)$)	0.0560
Goodness of Fit Indicator	1.016
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.22 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.26 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
V(1)	0.46521(7)	0.01042(8)	0.99959(7)	2.927(18)
Cl(1)	0.45296(10)	-0.17720(13)	1.06982(10)	4.10(3)
Cl(2)	0.59077(10)	-0.04924(16)	1.03606(11)	4.92(3)
O(1)	0.43869(19)	0.0703(3)	0.8816(2)	3.39(8)
N(1)	0.3344(2)	0.0357(3)	0.9398(2)	2.65(9)
N(2)	0.4909(2)	0.1378(3)	1.0767(2)	3.23(9)
C(1)	0.3918(3)	0.1720(5)	0.8239(3)	3.11(12)
C(2)	0.3179(3)	0.1984(4)	0.8158(3)	2.75(11)
C(3)	0.2684(3)	0.3000(4)	0.7536(3)	3.30(12)
C(4)	0.2932(3)	0.3773(5)	0.7027(3)	4.20(14)
C(5)	0.3656(3)	0.3493(6)	0.7105(3)	4.48(15)
C(6)	0.4155(3)	0.2476(5)	0.7695(3)	4.20(14)
C(7)	0.2918(2)	0.1166(4)	0.8688(2)	2.59(11)
C(8)	0.2867(2)	-0.0291(4)	0.9758(3)	2.74(11)
C(9)	0.2511(2)	-0.1536(4)	0.9399(3)	3.12(12)
C(10)	0.2006(3)	-0.2051(5)	0.9705(3)	4.18(14)
C(11)	0.1878(3)	-0.1341(6)	1.0350(3)	4.35(15)
C(12)	0.2250(3)	-0.0117(6)	1.0709(3)	4.40(15)
C(13)	0.2769(3)	0.0451(4)	1.0448(3)	3.20(12)
C(14)	0.2635(3)	-0.2324(4)	0.8676(3)	3.71(13)
C(15)	0.2837(3)	-0.3807(5)	0.8963(4)	6.01(18)
C(16)	0.1876(3)	-0.2203(5)	0.7656(3)	5.04(16)
C(17)	0.3184(3)	0.1782(5)	1.0847(3)	3.67(13)
C(18)	0.3508(3)	0.1930(6)	1.1954(3)	5.94(18)
C(19)	0.2655(3)	0.3001(5)	1.0323(3)	5.34(16)
C(20)	0.5165(3)	0.2405(5)	1.1449(3)	3.15(12)
C(21)	0.5482(3)	0.2046(5)	1.2428(3)	3.84(14)
C(22)	0.5686(3)	0.3093(7)	1.3096(3)	5.01(16)
C(23)	0.5613(3)	0.4423(6)	1.2815(4)	5.25(17)
C(24)	0.5317(3)	0.4754(5)	1.1862(4)	5.34(16)
C(25)	0.5093(3)	0.3756(5)	1.1157(3)	3.78(13)
C(26)	0.5561(3)	0.0589(5)	1.2721(3)	5.19(16)
C(27)	0.4763(3)	0.4123(5)	1.0116(3)	5.40(15)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.2119	0.3179	0.7456	3.89
H(2)	0.2570	0.4570	0.6584	4.91
H(3)	0.3832	0.4069	0.6698	5.69
H(4)	0.4703	0.2276	0.7737	5.51
H(5)	0.2362	0.1429	0.8469	3.07
H(6)	0.1720	-0.2989	0.9442	4.90
H(7)	0.1499	-0.1738	1.0575	5.53
H(8)	0.2141	0.0412	1.1195	5.73
H(9)	0.3120	-0.1890	0.8642	4.41
H(10)	0.2373	-0.4255	0.8884	7.16
H(11)	0.3002	-0.4228	0.8564	7.16
H(12)	0.3258	-0.3863	0.9620	7.16
H(13)	0.1774	-0.1275	0.7476	6.05
H(14)	0.1958	-0.2682	0.7204	6.06
H(15)	0.1428	-0.2578	0.7663	6.06
H(16)	0.3721	0.1875	1.0831	4.29
H(17)	0.3641	0.2852	1.2142	7.24
H(18)	0.3103	0.1646	1.2080	7.25
H(19)	0.3973	0.1382	1.2313	7.25
H(20)	0.2937	0.3810	1.0630	6.18
H(21)	0.2523	0.2998	0.9669	6.18
H(22)	0.2175	0.2956	1.0344	6.18
H(23)	0.5902	0.2860	1.3824	5.63
H(24)	0.5780	0.5193	1.3334	5.72
H(25)	0.5259	0.5780	1.1663	5.54
H(26)	0.5911	0.0137	1.2563	6.10
H(27)	0.5775	0.0536	1.3399	6.11
H(28)	0.5047	0.0168	1.2390	6.11
H(29)	0.4742	0.5082	1.0052	6.02
H(30)	0.5095	0.3760	0.9897	6.02
H(31)	0.4235	0.3760	0.9738	6.03

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	0.0351(5)	0.0386(5)	0.0390(4)	0.0068(4)	0.0202(3)	0.0022(4)
Cl(1)	0.0515(9)	0.0417(8)	0.0571(7)	0.0065(7)	0.0239(6)	0.0122(6)
Cl(2)	0.0438(10)	0.0827(10)	0.0642(8)	0.0231(9)	0.0304(8)	0.0117(8)
O(1)	0.039(2)	0.051(2)	0.0457(18)	0.0113(18)	0.0272(17)	0.0059(17)
N(1)	0.033(2)	0.037(2)	0.034(2)	0.004(2)	0.019(2)	-0.0005(18)
N(2)	0.035(2)	0.040(2)	0.049(2)	0.003(2)	0.023(2)	-0.002(2)
C(1)	0.043(3)	0.038(3)	0.037(2)	0.005(2)	0.021(2)	-0.001(2)
C(2)	0.037(3)	0.033(2)	0.035(2)	-0.001(2)	0.019(2)	-0.002(2)
C(3)	0.043(3)	0.038(3)	0.042(2)	0.005(2)	0.020(2)	0.004(2)
C(4)	0.060(4)	0.050(3)	0.046(3)	0.004(3)	0.024(3)	0.012(2)
C(5)	0.061(4)	0.065(4)	0.055(3)	0.005(3)	0.037(3)	0.014(2)
C(6)	0.059(4)	0.060(3)	0.056(3)	0.007(3)	0.040(3)	0.013(2)
C(7)	0.032(3)	0.033(2)	0.032(2)	0.004(2)	0.015(2)	-0.004(2)
C(8)	0.031(2)	0.036(3)	0.040(2)	0.009(2)	0.020(2)	0.015(2)
C(9)	0.038(3)	0.033(3)	0.048(2)	0.003(2)	0.022(2)	0.005(2)
C(10)	0.044(3)	0.051(3)	0.060(3)	-0.002(2)	0.024(3)	0.019(2)
C(11)	0.043(3)	0.072(4)	0.060(3)	0.009(3)	0.034(3)	0.025(3)
C(12)	0.057(4)	0.071(4)	0.053(3)	0.012(3)	0.039(3)	0.014(3)
C(13)	0.037(3)	0.049(3)	0.039(2)	0.010(2)	0.022(2)	0.006(2)
C(14)	0.042(3)	0.039(3)	0.058(3)	-0.006(2)	0.025(2)	-0.004(2)
C(15)	0.074(5)	0.050(3)	0.103(4)	-0.003(3)	0.044(4)	-0.011(3)
C(16)	0.065(4)	0.064(4)	0.063(3)	-0.007(3)	0.033(3)	-0.012(3)
C(17)	0.033(3)	0.058(3)	0.045(2)	0.004(2)	0.018(2)	-0.008(2)
C(18)	0.068(5)	0.105(5)	0.056(3)	-0.002(4)	0.035(3)	-0.019(3)
C(19)	0.076(5)	0.053(3)	0.067(3)	0.006(3)	0.032(3)	-0.018(2)
C(20)	0.027(3)	0.044(3)	0.046(2)	-0.002(2)	0.016(2)	-0.013(2)
C(21)	0.037(3)	0.050(3)	0.058(3)	-0.003(2)	0.024(2)	-0.006(2)
C(22)	0.043(3)	0.085(5)	0.050(3)	-0.001(3)	0.015(2)	-0.026(3)
C(23)	0.051(4)	0.060(4)	0.070(4)	-0.004(3)	0.018(3)	-0.029(3)
C(24)	0.045(3)	0.041(3)	0.089(4)	0.007(2)	0.015(3)	-0.016(3)
C(25)	0.027(3)	0.038(3)	0.066(3)	0.001(2)	0.016(2)	-0.007(2)
C(26)	0.070(5)	0.072(4)	0.051(3)	0.003(3)	0.029(3)	-0.002(3)
C(27)	0.070(4)	0.046(3)	0.075(3)	0.002(3)	0.027(3)	0.007(3)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
V(1)	Cl(1)	2.2452(18)	V(1)	Cl(2)	2.269(2)
V(1)	O(1)	1.797(3)	V(1)	N(1)	2.216(4)
V(1)	N(2)	1.654(4)	O(1)	C(1)	1.351(5)
N(1)	C(7)	1.287(4)	N(1)	C(8)	1.468(7)
N(2)	C(20)	1.385(6)	C(1)	C(2)	1.394(8)
C(1)	C(6)	1.398(9)	C(2)	C(3)	1.393(6)
C(2)	C(7)	1.444(8)	C(3)	C(4)	1.377(9)
C(4)	C(5)	1.373(10)	C(5)	C(6)	1.378(7)
C(8)	C(9)	1.382(6)	C(8)	C(13)	1.424(8)
C(9)	C(10)	1.400(9)	C(9)	C(14)	1.520(8)
C(10)	C(11)	1.379(9)	C(11)	C(12)	1.372(8)
C(12)	C(13)	1.394(9)	C(13)	C(17)	1.500(6)
C(14)	C(15)	1.523(7)	C(14)	C(16)	1.546(5)
C(17)	C(18)	1.566(7)	C(17)	C(19)	1.523(6)
C(20)	C(21)	1.414(7)	C(20)	C(25)	1.396(7)
C(21)	C(22)	1.394(8)	C(21)	C(26)	1.495(7)
C(22)	C(23)	1.371(9)	C(23)	C(24)	1.375(9)
C(24)	C(25)	1.393(8)	C(25)	C(27)	1.501(7)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(3)	H(1)	1.050	C(4)	H(2)	1.050
C(5)	H(3)	1.050	C(6)	H(4)	1.050
C(7)	H(5)	0.982	C(10)	H(6)	1.050
C(11)	H(7)	1.050	C(12)	H(8)	1.050
C(14)	H(9)	1.061	C(15)	H(10)	0.950
C(15)	H(11)	0.950	C(15)	H(12)	0.950
C(16)	H(13)	0.950	C(16)	H(14)	0.950
C(16)	H(15)	0.950	C(17)	H(16)	1.061
C(18)	H(17)	0.950	C(18)	H(18)	0.950
C(18)	H(19)	0.950	C(19)	H(20)	0.950
C(19)	H(21)	0.950	C(19)	H(22)	0.950
C(22)	H(23)	1.050	C(23)	H(24)	1.050
C(24)	H(25)	1.050	C(26)	H(26)	0.950
C(26)	H(27)	0.950	C(26)	H(28)	0.950
C(27)	H(29)	0.950	C(27)	H(30)	0.950
C(27)	H(31)	0.950			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	V(1)	Cl(2)	91.97(7)	Cl(1)	V(1)	O(1)	139.35(11)
Cl(1)	V(1)	N(1)	85.23(12)	Cl(1)	V(1)	N(2)	108.36(17)
Cl(2)	V(1)	O(1)	92.43(14)	Cl(2)	V(1)	N(1)	167.87(11)
Cl(2)	V(1)	N(2)	97.77(17)	O(1)	V(1)	N(1)	82.18(16)
O(1)	V(1)	N(2)	111.01(18)	N(1)	V(1)	N(2)	94.31(19)
V(1)	O(1)	C(1)	134.2(3)	V(1)	N(1)	C(7)	121.5(4)
V(1)	N(1)	C(8)	126.3(2)	C(7)	N(1)	C(8)	112.1(4)
V(1)	N(2)	C(20)	176.7(3)	O(1)	C(1)	C(2)	119.8(5)
O(1)	C(1)	C(6)	120.3(5)	C(2)	C(1)	C(6)	119.8(4)
C(1)	C(2)	C(3)	119.7(5)	C(1)	C(2)	C(7)	119.9(4)
C(3)	C(2)	C(7)	120.4(5)	C(2)	C(3)	C(4)	120.4(6)
C(3)	C(4)	C(5)	119.3(4)	C(4)	C(5)	C(6)	122.1(6)
C(1)	C(6)	C(5)	118.8(6)	N(1)	C(7)	C(2)	127.8(5)
N(1)	C(8)	C(9)	120.1(5)	N(1)	C(8)	C(13)	116.8(4)
C(9)	C(8)	C(13)	123.0(5)	C(8)	C(9)	C(10)	117.6(5)
C(8)	C(9)	C(14)	122.2(5)	C(10)	C(9)	C(14)	120.2(4)
C(9)	C(10)	C(11)	120.9(5)	C(10)	C(11)	C(12)	120.3(6)
C(11)	C(12)	C(13)	122.0(6)	C(8)	C(13)	C(12)	116.0(4)
C(8)	C(13)	C(17)	121.8(5)	C(12)	C(13)	C(17)	122.2(5)
C(9)	C(14)	C(15)	112.0(5)	C(9)	C(14)	C(16)	109.7(4)
C(15)	C(14)	C(16)	110.6(3)	C(13)	C(17)	C(18)	112.5(4)
C(13)	C(17)	C(19)	113.2(3)	C(18)	C(17)	C(19)	108.6(4)
N(2)	C(20)	C(21)	118.4(4)	N(2)	C(20)	C(25)	119.8(4)
C(21)	C(20)	C(25)	121.8(4)	C(20)	C(21)	C(22)	117.7(4)
C(20)	C(21)	C(26)	120.6(4)	C(22)	C(21)	C(26)	121.7(5)
C(21)	C(22)	C(23)	120.9(5)	C(22)	C(23)	C(24)	120.6(5)
C(23)	C(24)	C(25)	121.4(5)	C(20)	C(25)	C(24)	117.6(5)
C(20)	C(25)	C(27)	121.2(4)	C(24)	C(25)	C(27)	121.2(4)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(3)	H(1)	119.9	C(4)	C(3)	H(1)	119.8
C(3)	C(4)	H(2)	120.3	C(5)	C(4)	H(2)	120.4
C(4)	C(5)	H(3)	118.8	C(6)	C(5)	H(3)	119.1
C(1)	C(6)	H(4)	120.7	C(5)	C(6)	H(4)	120.6
N(1)	C(7)	H(5)	124.5	C(2)	C(7)	H(5)	107.2
C(9)	C(10)	H(6)	119.8	C(11)	C(10)	H(6)	119.2
C(10)	C(11)	H(7)	120.2	C(12)	C(11)	H(7)	119.5
C(11)	C(12)	H(8)	119.1	C(13)	C(12)	H(8)	118.8
C(9)	C(14)	H(9)	109.3	C(15)	C(14)	H(9)	107.9
C(16)	C(14)	H(9)	107.3	C(14)	C(15)	H(10)	109.2
C(14)	C(15)	H(11)	109.8	C(14)	C(15)	H(12)	109.5
H(10)	C(15)	H(11)	109.5	H(10)	C(15)	H(12)	109.5
H(11)	C(15)	H(12)	109.5	C(14)	C(16)	H(13)	109.5
C(14)	C(16)	H(14)	109.5	C(14)	C(16)	H(15)	109.4
H(13)	C(16)	H(14)	109.5	H(13)	C(16)	H(15)	109.5
H(14)	C(16)	H(15)	109.5	C(13)	C(17)	H(16)	112.4
C(18)	C(17)	H(16)	101.5	C(19)	C(17)	H(16)	108.0
C(17)	C(18)	H(17)	109.6	C(17)	C(18)	H(18)	109.1
C(17)	C(18)	H(19)	109.7	H(17)	C(18)	H(18)	109.5
H(17)	C(18)	H(19)	109.5	H(18)	C(18)	H(19)	109.5
C(17)	C(19)	H(20)	109.4	C(17)	C(19)	H(21)	109.4
C(17)	C(19)	H(22)	109.7	H(20)	C(19)	H(21)	109.5
H(20)	C(19)	H(22)	109.5	H(21)	C(19)	H(22)	109.5
C(21)	C(22)	H(23)	119.6	C(23)	C(22)	H(23)	119.5
C(22)	C(23)	H(24)	119.5	C(24)	C(23)	H(24)	119.9
C(23)	C(24)	H(25)	119.2	C(25)	C(24)	H(25)	119.4
C(21)	C(26)	H(26)	109.7	C(21)	C(26)	H(27)	109.2
C(21)	C(26)	H(28)	109.5	H(26)	C(26)	H(27)	109.5
H(26)	C(26)	H(28)	109.5	H(27)	C(26)	H(28)	109.5
C(25)	C(27)	H(29)	109.3	C(25)	C(27)	H(30)	110.2
C(25)	C(27)	H(31)	108.9	H(29)	C(27)	H(30)	109.5
H(29)	C(27)	H(31)	109.5	H(30)	C(27)	H(31)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle
Cl(1)	V(1)	O(1)	C(1)	118.2(4)
Cl(1)	V(1)	N(1)	C(8)	18.5(3)
Cl(2)	V(1)	O(1)	C(1)	-145.9(4)
Cl(2)	V(1)	N(1)	C(8)	95.5(6)
O(1)	V(1)	N(1)	C(7)	-22.9(3)
N(1)	V(1)	O(1)	C(1)	45.0(4)
N(2)	V(1)	O(1)	C(1)	-46.6(4)
N(2)	V(1)	N(1)	C(7)	87.7(3)
V(1)	O(1)	C(1)	C(2)	-41.3(6)
V(1)	N(1)	C(7)	C(2)	1.8(6)
V(1)	N(1)	C(8)	C(13)	92.8(4)
C(7)	N(1)	C(8)	C(13)	-84.8(4)
V(1)	N(2)	C(20)	C(21)	-47(8)
O(1)	C(1)	C(2)	C(3)	-177.7(3)
O(1)	C(1)	C(6)	C(5)	179.1(4)
C(6)	C(1)	C(2)	C(3)	-0.0(6)
C(1)	C(2)	C(3)	C(4)	-2.2(6)
C(3)	C(2)	C(7)	N(1)	-167.5(4)
C(2)	C(3)	C(4)	C(5)	2.9(7)
C(4)	C(5)	C(6)	C(1)	-0.7(7)
N(1)	C(8)	C(9)	C(14)	3.9(5)
N(1)	C(8)	C(13)	C(17)	-4.6(5)
C(9)	C(8)	C(13)	C(17)	178.1(3)
C(13)	C(8)	C(9)	C(14)	-178.9(3)
C(8)	C(9)	C(14)	C(15)	132.7(4)
C(10)	C(9)	C(14)	C(15)	-48.9(5)
C(14)	C(9)	C(10)	C(11)	-179.1(4)
C(10)	C(11)	C(12)	C(13)	-0.5(7)
C(11)	C(12)	C(13)	C(17)	-179.3(4)
C(8)	C(13)	C(17)	C(19)	96.5(6)
C(12)	C(13)	C(17)	C(19)	-81.8(6)
N(2)	C(20)	C(21)	C(26)	2.0(9)
N(2)	C(20)	C(25)	C(27)	-1.8(9)
C(21)	C(20)	C(25)	C(27)	179.5(6)
C(25)	C(20)	C(21)	C(26)	-179.3(6)
C(26)	C(21)	C(22)	C(23)	179.7(6)
C(22)	C(23)	C(24)	C(25)	-0.9(10)

atom1	atom2	atom3	atom4	angle
Cl(1)	V(1)	N(1)	C(7)	-164.2(3)
Cl(1)	V(1)	N(2)	C(20)	64(8)
Cl(2)	V(1)	N(1)	C(7)	-87.1(6)
Cl(2)	V(1)	N(2)	C(20)	-31(8)
O(1)	V(1)	N(1)	C(8)	159.8(3)
O(1)	V(1)	N(2)	C(20)	-127(8)
N(1)	V(1)	N(2)	C(20)	150(8)
N(2)	V(1)	N(1)	C(8)	-89.6(3)
V(1)	O(1)	C(1)	C(6)	141.1(4)
V(1)	N(1)	C(8)	C(9)	-89.9(4)
C(7)	N(1)	C(8)	C(9)	92.6(4)
C(8)	N(1)	C(7)	C(2)	179.4(4)
V(1)	N(2)	C(20)	C(25)	134(8)
O(1)	C(1)	C(2)	C(7)	-0.2(5)
C(2)	C(1)	C(6)	C(5)	1.5(7)
C(6)	C(1)	C(2)	C(7)	177.4(4)
C(1)	C(2)	C(7)	N(1)	15.1(7)
C(7)	C(2)	C(3)	C(4)	-179.6(4)
C(3)	C(4)	C(5)	C(6)	-1.5(7)
N(1)	C(8)	C(9)	C(10)	-174.5(3)
N(1)	C(8)	C(13)	C(12)	173.8(3)
C(9)	C(8)	C(13)	C(12)	-3.5(6)
C(13)	C(8)	C(9)	C(10)	2.7(6)
C(8)	C(9)	C(10)	C(11)	-0.6(6)
C(8)	C(9)	C(14)	C(16)	-104.2(5)
C(10)	C(9)	C(14)	C(16)	74.2(5)
C(9)	C(10)	C(11)	C(12)	-0.4(7)
C(11)	C(12)	C(13)	C(8)	2.3(6)
C(8)	C(13)	C(17)	C(18)	-140.0(4)
C(12)	C(13)	C(17)	C(18)	41.7(6)
N(2)	C(20)	C(21)	C(22)	-175.8(5)
N(2)	C(20)	C(25)	C(24)	176.4(5)
C(21)	C(20)	C(25)	C(24)	-2.3(9)
C(25)	C(20)	C(21)	C(22)	3.0(9)
C(20)	C(21)	C(22)	C(23)	-2.5(9)
C(21)	C(22)	C(23)	C(24)	1.6(10)
C(23)	C(24)	C(25)	C(20)	1.2(9)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(23)	C(24)	C(25)	C(27)	179.4(6)					

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
V(1)	C(1)	2.905(4)	V(1)	C(2)	3.414(4)
V(1)	C(7)	3.090(4)	V(1)	C(8)	3.305(6)
V(1)	C(20)	3.038(5)	Cl(1)	Cl(2)	3.246(2)
Cl(1)	N(1)	3.020(3)	Cl(1)	N(2)	3.180(4)
Cl(1)	C(8)	3.129(5)	Cl(1)	C(9)	3.364(5)
Cl(1)	C(14)	3.476(4)	Cl(2)	Cl(1)	3.246(2)
Cl(2)	O(1)	2.954(3)	Cl(2)	N(2)	2.983(5)
O(1)	Cl(2)	2.954(3)	O(1)	N(1)	2.656(6)
O(1)	N(2)	2.845(5)	O(1)	C(2)	2.376(6)
O(1)	C(6)	2.385(6)	O(1)	C(7)	2.787(7)
N(1)	Cl(1)	3.020(3)	N(1)	O(1)	2.656(6)
N(1)	N(2)	2.863(5)	N(1)	C(1)	2.948(8)
N(1)	C(2)	2.453(6)	N(1)	C(9)	2.469(6)
N(1)	C(13)	2.464(8)	N(1)	C(14)	2.929(5)
N(1)	C(17)	2.878(7)	N(1)	C(19)	3.588(8)
N(2)	Cl(1)	3.180(4)	N(2)	Cl(2)	2.983(5)
N(2)	O(1)	2.845(5)	N(2)	N(1)	2.863(5)
N(2)	C(1)	3.517(5)	N(2)	C(7)	3.586(5)
N(2)	C(17)	3.438(8)	N(2)	C(21)	2.404(6)
N(2)	C(25)	2.406(6)	N(2)	C(26)	2.834(6)
N(2)	C(27)	2.864(6)	C(1)	V(1)	2.905(4)
C(1)	N(1)	2.948(8)	C(1)	N(2)	3.517(5)
C(1)	C(3)	2.410(7)	C(1)	C(4)	2.780(6)
C(1)	C(5)	2.389(7)	C(1)	C(7)	2.456(9)
C(1)	C(27)	3.514(7)	C(2)	V(1)	3.414(4)
C(2)	O(1)	2.376(6)	C(2)	N(1)	2.453(6)
C(2)	C(4)	2.403(7)	C(2)	C(5)	2.753(9)
C(2)	C(6)	2.415(10)	C(3)	C(1)	2.410(7)
C(3)	C(5)	2.372(10)	C(3)	C(6)	2.778(9)
C(3)	C(7)	2.462(6)	C(3)	C(11)[1)]	3.445(7)
C(4)	C(1)	2.780(6)	C(4)	C(2)	2.403(7)
C(4)	C(6)	2.406(8)	C(4)	C(11)[1)]	3.397(7)
C(5)	C(1)	2.389(7)	C(5)	C(2)	2.753(9)
C(5)	C(3)	2.372(10)	C(6)	O(1)	2.385(6)
C(6)	C(2)	2.415(10)	C(6)	C(3)	2.778(9)
C(6)	C(4)	2.406(8)	C(7)	V(1)	3.090(4)
C(7)	O(1)	2.787(7)	C(7)	N(2)	3.586(5)

Table 9. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(7)	C(1)	2.456(9)	C(7)	C(3)	2.462(6)
C(7)	C(8)	2.288(7)	C(7)	C(9)	3.158(7)
C(7)	C(13)	3.081(8)	C(7)	C(14)	3.483(6)
C(7)	C(17)	3.304(7)	C(7)	C(19)	3.448(8)
C(8)	V(1)	3.305(6)	C(8)	Cl(1)	3.129(5)
C(8)	C(7)	2.288(7)	C(8)	C(10)	2.379(8)
C(8)	C(11)	2.749(9)	C(8)	C(12)	2.390(9)
C(8)	C(14)	2.542(6)	C(8)	C(16)	3.469(6)
C(8)	C(17)	2.555(6)	C(8)	C(19)	3.453(7)
C(9)	Cl(1)	3.364(5)	C(9)	N(1)	2.469(6)
C(9)	C(7)	3.158(7)	C(9)	C(11)	2.418(10)
C(9)	C(12)	2.791(9)	C(9)	C(13)	2.466(6)
C(9)	C(15)	2.523(8)	C(9)	C(16)	2.507(6)
C(10)	C(8)	2.379(8)	C(10)	C(12)	2.386(8)
C(10)	C(13)	2.807(7)	C(10)	C(14)	2.531(10)
C(10)	C(15)	3.003(10)	C(10)	C(16)	3.186(9)
C(11)	C(3)[2)]	3.445(7)	C(11)	C(4)[2)]	3.397(7)
C(11)	C(8)	2.749(9)	C(11)	C(9)	2.418(10)
C(11)	C(13)	2.419(8)	C(12)	C(8)	2.390(9)
C(12)	C(9)	2.791(9)	C(12)	C(10)	2.386(8)
C(12)	C(17)	2.534(8)	C(12)	C(18)	3.002(7)
C(12)	C(19)	3.310(8)	C(13)	N(1)	2.464(8)
C(13)	C(7)	3.081(8)	C(13)	C(9)	2.466(6)
C(13)	C(10)	2.807(7)	C(13)	C(11)	2.419(8)
C(13)	C(18)	2.550(6)	C(13)	C(19)	2.523(7)
C(14)	Cl(1)	3.476(4)	C(14)	N(1)	2.929(5)
C(14)	C(7)	3.483(6)	C(14)	C(8)	2.542(6)
C(14)	C(10)	2.531(10)	C(15)	C(9)	2.523(8)
C(15)	C(10)	3.003(10)	C(15)	C(16)	2.523(7)
C(16)	C(8)	3.469(6)	C(16)	C(9)	2.507(6)
C(16)	C(10)	3.186(9)	C(16)	C(15)	2.523(7)
C(16)	C(24)[3)]	3.553(8)	C(17)	N(1)	2.878(7)
C(17)	N(2)	3.438(8)	C(17)	C(7)	3.304(7)
C(17)	C(8)	2.555(6)	C(17)	C(12)	2.534(8)
C(17)	C(20)	3.499(8)	C(18)	C(12)	3.002(7)
C(18)	C(13)	2.550(6)	C(18)	C(19)	2.508(6)
C(18)	C(21)	3.498(9)	C(19)	N(1)	3.588(8)

Table 9. Distances beyond the asymmetric unit out to 3.60 Å (continued)

atom	atom	distance	atom	atom	distance
C(19)	C(7)	3.448(8)	C(19)	C(8)	3.453(7)
C(19)	C(12)	3.310(8)	C(19)	C(13)	2.523(7)
C(19)	C(18)	2.508(6)	C(20)	V(1)	3.038(5)
C(20)	C(17)	3.499(8)	C(20)	C(22)	2.403(7)
C(20)	C(23)	2.757(8)	C(20)	C(24)	2.386(7)
C(20)	C(26)	2.527(7)	C(20)	C(27)	2.525(7)
C(21)	N(2)	2.404(6)	C(21)	C(18)	3.498(9)
C(21)	C(23)	2.405(8)	C(21)	C(24)	2.786(7)
C(21)	C(25)	2.455(7)	C(22)	C(20)	2.403(7)
C(22)	C(24)	2.385(8)	C(22)	C(25)	2.798(8)
C(22)	C(26)	2.524(8)	C(23)	C(20)	2.757(8)
C(23)	C(21)	2.405(8)	C(23)	C(25)	2.414(8)
C(24)	C(16)[4)]	3.553(8)	C(24)	C(20)	2.386(7)
C(24)	C(21)	2.786(7)	C(24)	C(22)	2.385(8)
C(24)	C(27)	2.522(8)	C(25)	N(2)	2.406(6)
C(25)	C(21)	2.455(7)	C(25)	C(22)	2.798(8)
C(25)	C(23)	2.414(8)	C(26)	N(2)	2.834(6)
C(26)	C(20)	2.527(7)	C(26)	C(22)	2.524(8)
C(27)	N(2)	2.864(6)	C(27)	C(1)	3.514(7)
C(27)	C(20)	2.525(7)	C(27)	C(24)	2.522(8)

Symmetry Operators:

(1) X,-Y,Z+1/2-1 (2) X,-Y,Z+1/2
(3) X+1/2-1,-Y+1/2,Z+1/2-1 (4) X+1/2,-Y+1/2,Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
V(1)	H(9)	3.282	V(1)	H(16)	3.260
V(1)	H(26)	3.563	V(1)	H(28)	3.530
Cl(1)	H(3)[1)]	3.438	Cl(1)	H(4)[1)]	3.169
Cl(1)	H(9)	3.024	Cl(1)	H(12)	2.989
Cl(1)	H(15)[2)]	3.462	Cl(1)	H(25)[3)]	2.825
Cl(1)	H(26)	3.386	Cl(1)	H(28)	3.049
Cl(1)	H(29)[3)]	3.365	Cl(2)	H(2)[4)]	2.926
Cl(2)	H(14)[2)]	3.160	Cl(2)	H(22)[5)]	2.907
Cl(2)	H(23)[6)]	3.401	Cl(2)	H(27)[6)]	3.042
O(1)	H(4)	2.629	O(1)	H(9)	3.456
O(1)	H(18)[6)]	3.496	O(1)	H(19)[6)]	2.958
O(1)	H(27)[6)]	3.327	O(1)	H(28)[6)]	3.273
O(1)	H(30)	3.398	O(1)	H(31)	3.441
N(1)	H(5)	2.013	N(1)	H(9)	2.458
N(1)	H(13)	3.427	N(1)	H(16)	2.524
N(1)	H(21)	3.197	N(2)	H(16)	2.408
N(2)	H(26)	2.804	N(2)	H(28)	2.762
N(2)	H(30)	2.853	N(2)	H(31)	2.783
C(1)	H(1)	3.360	C(1)	H(3)	3.339
C(1)	H(4)	2.134	C(1)	H(5)	3.239
C(1)	H(19)[6)]	3.430	C(1)	H(30)	3.181
C(1)	H(31)	2.957	C(2)	H(1)	2.121
C(2)	H(2)	3.355	C(2)	H(4)	3.371
C(2)	H(5)	1.972	C(2)	H(21)	3.429
C(2)	H(31)	2.895	C(3)	H(2)	2.111
C(3)	H(3)	3.318	C(3)	H(5)	2.455
C(3)	H(7)[6)]	3.057	C(3)	H(11)[7)]	3.090
C(3)	H(31)	3.349	C(4)	H(1)	2.105
C(4)	H(3)	2.092	C(4)	H(4)	3.354
C(4)	H(7)[6)]	3.245	C(4)	H(11)[7)]	3.116
C(4)	H(17)[8)]	3.570	C(4)	H(20)[8)]	3.283
C(5)	H(1)	3.323	C(5)	H(2)	2.108
C(5)	H(4)	2.115	C(5)	H(20)[8)]	3.355
C(6)	H(2)	3.353	C(6)	H(3)	2.100
C(6)	H(24)[8)]	3.597	C(6)	H(28)[6)]	3.304
C(6)	H(30)	3.297	C(6)	H(31)	3.459
C(7)	H(1)	2.667	C(7)	H(9)	3.045

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(7)	H(13)	3.173	C(7)	H(16)	3.052
C(7)	H(21)	2.757	C(7)	H(31)	3.388
C(8)	H(5)	2.462	C(8)	H(6)	3.335
C(8)	H(8)	3.348	C(8)	H(9)	2.624
C(8)	H(13)	3.309	C(8)	H(16)	2.709
C(8)	H(21)	3.299	C(8)	H(24)[9)]	3.476
C(9)	H(5)	3.231	C(9)	H(6)	2.127
C(9)	H(7)	3.368	C(9)	H(9)	2.122
C(9)	H(10)	2.778	C(9)	H(11)	3.331
C(9)	H(12)	2.638	C(9)	H(13)	2.677
C(9)	H(14)	3.325	C(9)	H(15)	2.691
C(9)	H(24)[9)]	3.167	C(10)	H(7)	2.112
C(10)	H(8)	3.331	C(10)	H(9)	3.386
C(10)	H(10)	2.821	C(10)	H(12)	3.075
C(10)	H(13)	3.473	C(10)	H(15)	2.928
C(10)	H(24)[9)]	2.912	C(11)	H(6)	2.102
C(11)	H(8)	2.095	C(11)	H(24)[9)]	3.043
C(11)	H(30)[10)]	3.148	C(12)	H(6)	3.330
C(12)	H(7)	2.098	C(12)	H(16)	3.386
C(12)	H(18)	2.623	C(12)	H(19)	3.337
C(12)	H(22)	3.076	C(12)	H(23)[9)]	3.583
C(12)	H(24)[9)]	3.399	C(13)	H(5)	3.025
C(13)	H(7)	3.361	C(13)	H(8)	2.111
C(13)	H(16)	2.142	C(13)	H(17)	3.342
C(13)	H(18)	2.644	C(13)	H(19)	2.847
C(13)	H(20)	3.327	C(13)	H(21)	2.738
C(13)	H(22)	2.695	C(14)	H(6)	2.715
C(14)	H(10)	2.043	C(14)	H(11)	2.050
C(14)	H(12)	2.046	C(14)	H(13)	2.067
C(14)	H(14)	2.068	C(14)	H(15)	2.067
C(14)	H(18)[6)]	3.211	C(15)	H(6)	2.769
C(15)	H(9)	2.107	C(15)	H(13)	3.338
C(15)	H(14)	2.683	C(15)	H(15)	2.725
C(15)	H(20)[3)]	3.501	C(15)	H(21)[3)]	3.508
C(15)	H(26)[11)]	3.475	C(15)	H(29)[3)]	3.354
C(15)	H(31)[3)]	3.344	C(16)	H(6)	3.145
C(16)	H(8)[6)]	3.194	C(16)	H(9)	2.120

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(16)	H(10)	2.641	C(16)	H(11)	2.758
C(16)	H(12)	3.339	C(16)	H(18)[6)]	3.019
C(16)	H(24)[9)]	3.469	C(16)	H(25)[9)]	3.031
C(16)	H(26)[11)]	3.407	C(17)	H(5)	3.328
C(17)	H(8)	2.722	C(17)	H(17)	2.086
C(17)	H(18)	2.080	C(17)	H(19)	2.088
C(17)	H(20)	2.044	C(17)	H(21)	2.045
C(17)	H(22)	2.048	C(17)	H(28)	3.537
C(18)	H(8)	2.725	C(18)	H(9)[1)]	3.177
C(18)	H(14)[1)]	3.317	C(18)	H(16)	2.059
C(18)	H(20)	2.606	C(18)	H(21)	3.331
C(18)	H(22)	2.745	C(18)	H(28)	3.203
C(19)	H(2)[12)]	3.201	C(19)	H(5)	3.150
C(19)	H(8)	3.307	C(19)	H(10)[7)]	3.422
C(19)	H(16)	2.107	C(19)	H(17)	2.550
C(19)	H(18)	2.835	C(19)	H(19)	3.306
C(19)	H(23)[9)]	3.107	C(20)	H(1)[4)]	3.315
C(20)	H(16)	2.497	C(20)	H(19)	3.411
C(20)	H(23)	3.359	C(20)	H(25)	3.341
C(20)	H(26)	2.764	C(20)	H(27)	3.295
C(20)	H(28)	2.754	C(20)	H(29)	3.288
C(20)	H(30)	2.779	C(20)	H(31)	2.743
C(21)	H(1)[4)]	3.162	C(21)	H(5)[4)]	3.474
C(21)	H(16)	3.042	C(21)	H(17)	3.448
C(21)	H(19)	2.909	C(21)	H(23)	2.119
C(21)	H(24)	3.351	C(21)	H(26)	2.024
C(21)	H(27)	2.018	C(21)	H(28)	2.022
C(22)	H(5)[4)]	3.021	C(22)	H(17)	3.445
C(22)	H(19)	3.341	C(22)	H(21)[4)]	3.312
C(22)	H(22)[4)]	3.430	C(22)	H(24)	2.097
C(22)	H(25)	3.328	C(22)	H(26)	3.131
C(22)	H(27)	2.557	C(22)	H(28)	3.114
C(23)	H(3)[12)]	3.312	C(23)	H(5)[4)]	3.104
C(23)	H(13)[4)]	3.160	C(23)	H(15)[4)]	3.555
C(23)	H(23)	2.097	C(23)	H(25)	2.098
C(24)	H(3)[12)]	2.987	C(24)	H(13)[4)]	2.888
C(24)	H(15)[4)]	3.347	C(24)	H(23)	3.331

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(24)	H(24)	2.106	C(24)	H(29)	2.554
C(24)	H(30)	3.129	C(24)	H(31)	3.113
C(25)	H(7)[13)]	3.346	C(25)	H(16)	3.058
C(25)	H(24)	3.361	C(25)	H(25)	2.116
C(25)	H(29)	2.025	C(25)	H(30)	2.035
C(25)	H(31)	2.020	C(26)	H(1)[4)]	3.484
C(26)	H(4)[1)]	3.285	C(26)	H(6)[2)]	3.591
C(26)	H(10)[2)]	3.287	C(26)	H(15)[2)]	3.436
C(26)	H(16)	3.524	C(26)	H(19)	2.905
C(26)	H(23)	2.722	C(27)	H(7)[13)]	3.168
C(27)	H(11)[7)]	3.429	C(27)	H(12)[7)]	3.270
C(27)	H(16)	3.567	C(27)	H(25)	2.711
H(1)	C(1)	3.360	H(1)	C(2)	2.121
H(1)	C(4)	2.105	H(1)	C(5)	3.323
H(1)	C(7)	2.667	H(1)	C(20)[9)]	3.315
H(1)	C(21)[9)]	3.162	H(1)	C(26)[9)]	3.484
H(1)	H(2)	2.428	H(1)	H(5)	2.253
H(1)	H(7)[6)]	2.986	H(1)	H(10)[7)]	3.283
H(1)	H(11)[7)]	3.083	H(1)	H(21)	3.242
H(1)	H(26)[9)]	2.949	H(2)	Cl(2)[9)]	2.926
H(2)	C(2)	3.355	H(2)	C(3)	2.111
H(2)	C(5)	2.108	H(2)	C(6)	3.353
H(2)	C(19)[8)]	3.201	H(2)	H(1)	2.428
H(2)	H(3)	2.410	H(2)	H(6)[6)]	3.349
H(2)	H(7)[6)]	3.359	H(2)	H(11)[7)]	3.091
H(2)	H(14)[7)]	3.313	H(2)	H(17)[8)]	3.108
H(2)	H(20)[8)]	2.561	H(2)	H(22)[8)]	2.993
H(3)	Cl(1)[6)]	3.438	H(3)	C(1)	3.339
H(3)	C(3)	3.318	H(3)	C(4)	2.092
H(3)	C(6)	2.100	H(3)	C(23)[8)]	3.312
H(3)	C(24)[8)]	2.987	H(3)	H(2)	2.410
H(3)	H(4)	2.423	H(3)	H(12)[6)]	2.946
H(3)	H(17)[8)]	3.184	H(3)	H(20)[8)]	2.697
H(3)	H(24)[8)]	3.389	H(3)	H(25)[8)]	2.802
H(4)	Cl(1)[6)]	3.169	H(4)	O(1)	2.629
H(4)	C(1)	2.134	H(4)	C(2)	3.371
H(4)	C(4)	3.354	H(4)	C(5)	2.115

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(4)	C(26)[6)]	3.285	H(4)	H(3)	2.423
H(4)	H(6)[13)]	3.441	H(4)	H(15)[13)]	3.411
H(4)	H(24)[8)]	3.073	H(4)	H(25)[8)]	3.126
H(4)	H(26)[6)]	3.451	H(4)	H(27)[6)]	3.296
H(4)	H(28)[6)]	2.636	H(4)	H(30)	3.484
H(5)	N(1)	2.013	H(5)	C(1)	3.239
H(5)	C(2)	1.972	H(5)	C(3)	2.455
H(5)	C(8)	2.462	H(5)	C(9)	3.231
H(5)	C(13)	3.025	H(5)	C(17)	3.328
H(5)	C(19)	3.150	H(5)	C(21)[9)]	3.474
H(5)	C(22)[9)]	3.021	H(5)	C(23)[9)]	3.104
H(5)	H(1)	2.253	H(5)	H(9)	3.540
H(5)	H(13)	3.015	H(5)	H(16)	3.363
H(5)	H(21)	2.369	H(5)	H(22)	3.562
H(5)	H(23)[9)]	3.242	H(5)	H(24)[9)]	3.365
H(6)	C(8)	3.335	H(6)	C(9)	2.127
H(6)	C(11)	2.102	H(6)	C(12)	3.330
H(6)	C(14)	2.715	H(6)	C(15)	2.769
H(6)	C(16)	3.145	H(6)	C(26)[11)]	3.591
H(6)	H(2)[1)]	3.349	H(6)	H(4)[10)]	3.441
H(6)	H(7)	2.420	H(6)	H(10)	2.270
H(6)	H(12)	2.975	H(6)	H(15)	2.654
H(6)	H(24)[9)]	3.280	H(6)	H(26)[11)]	3.352
H(6)	H(27)[11)]	3.052	H(7)	C(3)[1)]	3.057
H(7)	C(4)[1)]	3.245	H(7)	C(9)	3.368
H(7)	C(10)	2.112	H(7)	C(12)	2.098
H(7)	C(13)	3.361	H(7)	C(25)[10)]	3.346
H(7)	C(27)[10)]	3.168	H(7)	H(1)[1)]	2.986
H(7)	H(2)[1)]	3.359	H(7)	H(6)	2.420
H(7)	H(8)	2.400	H(7)	H(24)[9)]	3.485
H(7)	H(29)[10)]	3.547	H(7)	H(30)[10)]	2.404
H(8)	C(8)	3.348	H(8)	C(10)	3.331
H(8)	C(11)	2.095	H(8)	C(13)	2.111
H(8)	C(16)[1)]	3.194	H(8)	C(17)	2.722
H(8)	C(18)	2.725	H(8)	C(19)	3.307
H(8)	H(7)	2.400	H(8)	H(13)[1)]	2.649
H(8)	H(14)[1)]	2.896	H(8)	H(17)	3.465

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(8)	H(18)	2.063	H(8)	H(19)	3.192
H(8)	H(22)	2.877	H(9)	V(1)	3.282
H(9)	Cl(1)	3.024	H(9)	O(1)	3.456
H(9)	N(1)	2.458	H(9)	C(7)	3.045
H(9)	C(8)	2.624	H(9)	C(9)	2.122
H(9)	C(10)	3.386	H(9)	C(15)	2.107
H(9)	C(16)	2.120	H(9)	C(18)[6)]	3.177
H(9)	H(5)	3.540	H(9)	H(10)	2.877
H(9)	H(11)	2.314	H(9)	H(12)	2.431
H(9)	H(13)	2.379	H(9)	H(14)	2.384
H(9)	H(15)	2.897	H(9)	H(17)[6)]	3.210
H(9)	H(18)[6)]	2.514	H(9)	H(19)[6)]	3.354
H(10)	C(9)	2.778	H(10)	C(10)	2.821
H(10)	C(14)	2.043	H(10)	C(16)	2.641
H(10)	C(19)[3)]	3.422	H(10)	C(26)[11)]	3.287
H(10)	H(1)[3)]	3.283	H(10)	H(6)	2.270
H(10)	H(9)	2.877	H(10)	H(11)	1.551
H(10)	H(12)	1.551	H(10)	H(13)	3.525
H(10)	H(14)	2.855	H(10)	H(15)	2.500
H(10)	H(20)[3)]	3.098	H(10)	H(21)[3)]	2.940
H(10)	H(26)[11)]	2.666	H(10)	H(27)[11)]	3.052
H(11)	C(3)[3)]	3.090	H(11)	C(4)[3)]	3.116
H(11)	C(9)	3.331	H(11)	C(14)	2.050
H(11)	C(16)	2.758	H(11)	C(27)[3)]	3.429
H(11)	H(1)[3)]	3.083	H(11)	H(2)[3)]	3.091
H(11)	H(9)	2.314	H(11)	H(10)	1.551
H(11)	H(12)	1.551	H(11)	H(13)	3.595
H(11)	H(14)	2.581	H(11)	H(15)	3.084
H(11)	H(17)[6)]	3.404	H(11)	H(18)[6)]	3.569
H(11)	H(29)[3)]	3.043	H(11)	H(31)[3)]	2.928
H(12)	Cl(1)	2.989	H(12)	C(9)	2.638
H(12)	C(10)	3.075	H(12)	C(14)	2.046
H(12)	C(16)	3.339	H(12)	C(27)[3)]	3.270
H(12)	H(3)[1)]	2.946	H(12)	H(6)	2.975
H(12)	H(9)	2.431	H(12)	H(10)	1.551
H(12)	H(11)	1.551	H(12)	H(14)	3.574
H(12)	H(15)	3.553	H(12)	H(20)[3)]	3.059

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(12)	H(21)[3)]	3.426	H(12)	H(25)[3)]	3.586
H(12)	H(29)[3)]	2.790	H(12)	H(31)[3)]	2.958
H(13)	N(1)	3.427	H(13)	C(7)	3.173
H(13)	C(8)	3.309	H(13)	C(9)	2.677
H(13)	C(10)	3.473	H(13)	C(14)	2.067
H(13)	C(15)	3.338	H(13)	C(23)[9)]	3.160
H(13)	C(24)[9)]	2.888	H(13)	H(5)	3.015
H(13)	H(8)[6)]	2.649	H(13)	H(9)	2.379
H(13)	H(10)	3.525	H(13)	H(11)	3.595
H(13)	H(14)	1.551	H(13)	H(15)	1.551
H(13)	H(18)[6)]	2.980	H(13)	H(24)[9)]	3.087
H(13)	H(25)[9)]	2.574	H(14)	Cl(2)[11)]	3.160
H(14)	C(9)	3.325	H(14)	C(14)	2.068
H(14)	C(15)	2.683	H(14)	C(18)[6)]	3.317
H(14)	H(2)[3)]	3.313	H(14)	H(8)[6)]	2.896
H(14)	H(9)	2.384	H(14)	H(10)	2.855
H(14)	H(11)	2.581	H(14)	H(12)	3.574
H(14)	H(13)	1.551	H(14)	H(15)	1.551
H(14)	H(17)[6)]	3.322	H(14)	H(18)[6)]	2.544
H(14)	H(22)[6)]	3.249	H(14)	H(25)[9)]	3.490
H(14)	H(26)[11)]	3.397	H(15)	Cl(1)[11)]	3.462
H(15)	C(9)	2.691	H(15)	C(10)	2.928
H(15)	C(14)	2.067	H(15)	C(15)	2.725
H(15)	C(23)[9)]	3.555	H(15)	C(24)[9)]	3.347
H(15)	C(26)[11)]	3.436	H(15)	H(4)[10)]	3.411
H(15)	H(6)	2.654	H(15)	H(9)	2.897
H(15)	H(10)	2.500	H(15)	H(11)	3.084
H(15)	H(12)	3.553	H(15)	H(13)	1.551
H(15)	H(14)	1.551	H(15)	H(24)[9)]	3.111
H(15)	H(25)[9)]	2.668	H(15)	H(26)[11)]	2.689
H(15)	H(28)[11)]	3.560	H(16)	V(1)	3.260
H(16)	N(1)	2.524	H(16)	N(2)	2.408
H(16)	C(7)	3.052	H(16)	C(8)	2.709
H(16)	C(12)	3.386	H(16)	C(13)	2.142
H(16)	C(18)	2.059	H(16)	C(19)	2.107
H(16)	C(20)	2.497	H(16)	C(21)	3.042
H(16)	C(25)	3.058	H(16)	C(26)	3.524

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(16)	C(27)	3.567	H(16)	H(5)	3.363
H(16)	H(17)	2.402	H(16)	H(18)	2.838
H(16)	H(19)	2.234	H(16)	H(20)	2.357
H(16)	H(21)	2.377	H(16)	H(22)	2.886
H(16)	H(28)	3.017	H(16)	H(31)	3.061
H(17)	C(4)[12)]	3.570	H(17)	C(13)	3.342
H(17)	C(17)	2.086	H(17)	C(19)	2.550
H(17)	C(21)	3.448	H(17)	C(22)	3.445
H(17)	H(2)[12)]	3.108	H(17)	H(3)[12)]	3.184
H(17)	H(8)	3.465	H(17)	H(9)[1)]	3.210
H(17)	H(11)[1)]	3.404	H(17)	H(14)[1)]	3.322
H(17)	H(16)	2.402	H(17)	H(18)	1.551
H(17)	H(19)	1.551	H(17)	H(20)	2.296
H(17)	H(21)	3.424	H(17)	H(22)	2.830
H(18)	O(1)[1)]	3.496	H(18)	C(12)	2.623
H(18)	C(13)	2.644	H(18)	C(14)[1)]	3.211
H(18)	C(16)[1)]	3.019	H(18)	C(17)	2.080
H(18)	C(19)	2.835	H(18)	H(8)	2.063
H(18)	H(9)[1)]	2.514	H(18)	H(11)[1)]	3.569
H(18)	H(13)[1)]	2.980	H(18)	H(14)[1)]	2.544
H(18)	H(16)	2.838	H(18)	H(17)	1.551
H(18)	H(19)	1.551	H(18)	H(20)	3.059
H(18)	H(22)	2.749	H(19)	O(1)[1)]	2.958
H(19)	C(1)[1)]	3.430	H(19)	C(12)	3.337
H(19)	C(13)	2.847	H(19)	C(17)	2.088
H(19)	C(19)	3.306	H(19)	C(20)	3.411
H(19)	C(21)	2.909	H(19)	C(22)	3.341
H(19)	C(26)	2.905	H(19)	H(8)	3.192
H(19)	H(9)[1)]	3.354	H(19)	H(16)	2.234
H(19)	H(17)	1.551	H(19)	H(18)	1.551
H(19)	H(20)	3.394	H(19)	H(23)	3.558
H(19)	H(27)	3.110	H(19)	H(28)	2.349
H(20)	C(4)[12)]	3.283	H(20)	C(5)[12)]	3.355
H(20)	C(13)	3.327	H(20)	C(15)[7)]	3.501
H(20)	C(17)	2.044	H(20)	C(18)	2.606
H(20)	H(2)[12)]	2.561	H(20)	H(3)[12)]	2.697
H(20)	H(10)[7)]	3.098	H(20)	H(12)[7)]	3.059

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(20)	H(16)	2.357	H(20)	H(17)	2.296
H(20)	H(18)	3.059	H(20)	H(19)	3.394
H(20)	H(21)	1.552	H(20)	H(22)	1.551
H(20)	H(31)	3.486	H(21)	N(1)	3.197
H(21)	C(2)	3.429	H(21)	C(7)	2.757
H(21)	C(8)	3.299	H(21)	C(13)	2.738
H(21)	C(15)[7)]	3.508	H(21)	C(17)	2.045
H(21)	C(18)	3.331	H(21)	C(22)[9)]	3.312
H(21)	H(1)	3.242	H(21)	H(5)	2.369
H(21)	H(10)[7)]	2.940	H(21)	H(12)[7)]	3.426
H(21)	H(16)	2.377	H(21)	H(17)	3.424
H(21)	H(20)	1.552	H(21)	H(22)	1.551
H(21)	H(23)[9)]	2.837	H(21)	H(27)[9)]	3.259
H(21)	H(31)	3.350	H(22)	Cl(2)[14)]	2.907
H(22)	C(12)	3.076	H(22)	C(13)	2.695
H(22)	C(17)	2.048	H(22)	C(18)	2.745
H(22)	C(22)[9)]	3.430	H(22)	H(2)[12)]	2.993
H(22)	H(5)	3.562	H(22)	H(8)	2.877
H(22)	H(14)[1)]	3.249	H(22)	H(16)	2.886
H(22)	H(17)	2.830	H(22)	H(18)	2.749
H(22)	H(20)	1.551	H(22)	H(21)	1.551
H(22)	H(23)[9)]	2.553	H(22)	H(27)[9)]	3.263
H(23)	Cl(2)[1)]	3.401	H(23)	C(12)[4)]	3.583
H(23)	C(19)[4)]	3.107	H(23)	C(20)	3.359
H(23)	C(21)	2.119	H(23)	C(23)	2.097
H(23)	C(24)	3.331	H(23)	C(26)	2.722
H(23)	H(5)[4)]	3.242	H(23)	H(19)	3.558
H(23)	H(21)[4)]	2.837	H(23)	H(22)[4)]	2.553
H(23)	H(24)	2.404	H(23)	H(26)	3.374
H(23)	H(27)	2.369	H(23)	H(28)	3.346
H(24)	C(6)[12)]	3.597	H(24)	C(8)[4)]	3.476
H(24)	C(9)[4)]	3.167	H(24)	C(10)[4)]	2.912
H(24)	C(11)[4)]	3.043	H(24)	C(12)[4)]	3.399
H(24)	C(16)[4)]	3.469	H(24)	C(21)	3.351
H(24)	C(22)	2.097	H(24)	C(24)	2.106
H(24)	C(25)	3.361	H(24)	H(3)[12)]	3.389
H(24)	H(4)[12)]	3.073	H(24)	H(5)[4)]	3.365

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(24)	H(6)[4)]	3.280	H(24)	H(7)[4)]	3.485
H(24)	H(13)[4)]	3.087	H(24)	H(15)[4)]	3.111
H(24)	H(23)	2.404	H(24)	H(25)	2.412
H(24)	H(30)[12)]	3.558	H(25)	Cl(1)[7)]	2.825
H(25)	C(16)[4)]	3.031	H(25)	C(20)	3.341
H(25)	C(22)	3.328	H(25)	C(23)	2.098
H(25)	C(25)	2.116	H(25)	C(27)	2.711
H(25)	H(3)[12)]	2.802	H(25)	H(4)[12)]	3.126
H(25)	H(12)[7)]	3.586	H(25)	H(13)[4)]	2.574
H(25)	H(14)[4)]	3.490	H(25)	H(15)[4)]	2.668
H(25)	H(24)	2.412	H(25)	H(29)	2.355
H(25)	H(30)	3.354	H(25)	H(31)	3.346
H(26)	V(1)	3.563	H(26)	Cl(1)	3.386
H(26)	N(2)	2.804	H(26)	C(15)[2)]	3.475
H(26)	C(16)[2)]	3.407	H(26)	C(20)	2.764
H(26)	C(21)	2.024	H(26)	C(22)	3.131
H(26)	H(1)[4)]	2.949	H(26)	H(4)[1)]	3.451
H(26)	H(6)[2)]	3.352	H(26)	H(10)[2)]	2.666
H(26)	H(14)[2)]	3.397	H(26)	H(15)[2)]	2.689
H(26)	H(23)	3.374	H(26)	H(27)	1.551
H(26)	H(28)	1.551	H(27)	Cl(2)[1)]	3.042
H(27)	O(1)[1)]	3.327	H(27)	C(20)	3.295
H(27)	C(21)	2.018	H(27)	C(22)	2.557
H(27)	H(4)[1)]	3.296	H(27)	H(6)[2)]	3.052
H(27)	H(10)[2)]	3.052	H(27)	H(19)	3.110
H(27)	H(21)[4)]	3.259	H(27)	H(22)[4)]	3.263
H(27)	H(23)	2.369	H(27)	H(26)	1.551
H(27)	H(28)	1.551	H(28)	V(1)	3.530
H(28)	Cl(1)	3.049	H(28)	O(1)[1)]	3.273
H(28)	N(2)	2.762	H(28)	C(6)[1)]	3.304
H(28)	C(17)	3.537	H(28)	C(18)	3.203
H(28)	C(20)	2.754	H(28)	C(21)	2.022
H(28)	C(22)	3.114	H(28)	H(4)[1)]	2.636
H(28)	H(15)[2)]	3.560	H(28)	H(16)	3.017
H(28)	H(19)	2.349	H(28)	H(23)	3.346
H(28)	H(26)	1.551	H(28)	H(27)	1.551
H(29)	Cl(1)[7)]	3.365	H(29)	C(15)[7)]	3.354

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(29)	C(20)	3.288	H(29)	C(24)	2.554
H(29)	C(25)	2.025	H(29)	H(7)[13)]	3.547
H(29)	H(11)[7)]	3.043	H(29)	H(12)[7)]	2.790
H(29)	H(25)	2.355	H(29)	H(30)	1.551
H(29)	H(31)	1.551	H(30)	O(1)	3.398
H(30)	N(2)	2.853	H(30)	C(1)	3.181
H(30)	C(6)	3.297	H(30)	C(11)[13)]	3.148
H(30)	C(20)	2.779	H(30)	C(24)	3.129
H(30)	C(25)	2.035	H(30)	H(4)	3.484
H(30)	H(7)[13)]	2.404	H(30)	H(24)[8)]	3.558
H(30)	H(25)	3.354	H(30)	H(29)	1.551
H(30)	H(31)	1.551	H(31)	O(1)	3.441
H(31)	N(2)	2.783	H(31)	C(1)	2.957
H(31)	C(2)	2.895	H(31)	C(3)	3.349
H(31)	C(6)	3.459	H(31)	C(7)	3.388
H(31)	C(15)[7)]	3.344	H(31)	C(20)	2.743
H(31)	C(24)	3.113	H(31)	C(25)	2.020
H(31)	H(11)[7)]	2.928	H(31)	H(12)[7)]	2.958
H(31)	H(16)	3.061	H(31)	H(20)	3.486
H(31)	H(21)	3.350	H(31)	H(25)	3.346
H(31)	H(29)	1.551	H(31)	H(30)	1.551

Symmetry Operators:

(1) X,-Y,Z+1/2
(2) X+1/2,-Y+1/2-1,Z+1/2
(3) X,Y-1,Z
(4) X+1/2,-Y+1/2,Z+1/2
(5) X+1/2,Y+1/2-1,Z
(6) X,-Y,Z+1/2-1
(7) X,Y+1,Z
(8) X,-Y+1,Z+1/2-1
(9) X+1/2-1,-Y+1/2,Z+1/2-1
(10) X+1/2-1,Y+1/2-1,Z
(11) X+1/2-1,-Y+1/2-1,Z+1/2-1
(12) X,-Y+1,Z+1/2
(13) X+1/2,Y+1/2,Z
(14) X+1/2-1,Y+1/2,Z

X-ray Structure Report for VCl_2(N-2,6-$Me_2C_6H_3$)[O-2-Me-6-{(2,6-$^iPr_2C_6H_3$)N=CH}C_6H_3]
(**1b**)

February 25, 2008

Experimental

Data Collection

A black block crystal of $C_{28}H_{33}Cl_2N_2OV$ having approximate dimensions of 0.60 x 0.20 x 0.14 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 240 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 11.2178(4) Å
b = 14.2133(4) Å
c = 16.9951(6) Å
V = 2709.72(16) Å^3

For Z = 4 and F.W. = 535.43, the calculated density is 1.312 g/cm^3. The systematic absences of:

h00: h ± 2n
0k0: k ± 2n
00l: l ± 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -30 ± 1°C to a maximum 2θ value of 54.8°. A total of 44 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 5.0° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 100.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 160.0° in 5.0° step, at χ=45.0° and ϕ = 210.0°. The exposure rate was 100.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 26395 reflections that were collected, 6163 were unique (R_{int} = 0.032); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 5.856 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.653 to 0.921. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4535 observed reflections and 341 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0293$$

$$wR2 = [\, \Sigma \, (\, w \, (Fo^2 - Fc^2)^2 \,) / \, \Sigma \, w(Fo^2)^2]^{1/2} = 0.0594$$

The standard deviation of an observation of unit weight[4] was 1.01. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.37 and -0.31 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.003(19), refined using 2707 Friedel pairs.[5]

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10,11] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$$\Sigma w(F_o^2-F_c^2)^2$$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$$

where: N_o = number of observations
N_v = number of variables

(5) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{28}H_{33}Cl_2N_2OV$
Formula Weight	535.43
Crystal Color, Habit	black, block
Crystal Dimensions	0.60 X 0.20 X 0.14 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 240.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 11.2178(4) Å b = 14.2133(4) Å c = 16.9951(6) Å V = 2709.72(16) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.312 g/cm^3
F_{000}	1120.00
μ(MoKα)	5.856 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	44 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	100.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=210.0)	0.0 - 160.0^{o}
Exposure Rate	100.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.8^{o}
No. of Reflections Measured	Total: 26395 Unique: 6163 (R_{int} = 0.032) Friedel pairs: 2707
Corrections	Lorentz-polarization Absorption (trans. factors: 0.653 - 0.921)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0001Fo^2+1.0500\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.8^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	4535
No. Variables	341
Reflection/Parameter Ratio	13.30
Residuals: R1 (I>2.00σ(I))	0.0293
Residuals: wR2 (I>2.00σ(I))	0.0594
Goodness of Fit Indicator	1.008
Flack Parameter (Friedel pairs = 2707)	0.003(19)
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.37 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.31 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
V(1)	0.28873(4)	-0.03957(2)	0.83775(2)	2.952(8)
Cl(1)	0.37051(7)	-0.17953(4)	0.86674(4)	4.793(16)
Cl(2)	0.11540(7)	-0.10954(5)	0.80121(4)	4.986(17)
O(1)	0.20860(15)	0.06147(10)	0.87722(9)	3.56(3)
N(1)	0.34177(17)	-0.00642(11)	0.75236(11)	3.08(4)
N(2)	0.43346(16)	0.01974(11)	0.91139(10)	2.70(4)
C(1)	0.3731(2)	0.01976(14)	0.67673(13)	3.05(5)
C(2)	0.3863(2)	0.11665(15)	0.65826(15)	3.45(5)
C(3)	0.4137(2)	0.13943(18)	0.58114(15)	4.06(6)
C(4)	0.4271(2)	0.07145(19)	0.52546(16)	4.19(6)
C(5)	0.4144(2)	-0.0228(2)	0.54363(15)	4.23(6)
C(6)	0.3862(2)	-0.05076(16)	0.61887(14)	3.52(5)
C(7)	0.3686(2)	0.19019(16)	0.71909(15)	4.73(7)
C(8)	0.3651(3)	-0.15219(16)	0.63856(16)	5.01(7)
C(9)	0.2299(2)	0.15050(15)	0.89794(13)	3.11(5)
C(10)	0.3377(2)	0.17218(15)	0.93284(13)	3.09(5)
C(11)	0.3578(2)	0.26485(16)	0.95927(16)	4.43(6)
C(12)	0.2713(3)	0.33259(18)	0.94820(18)	5.44(8)
C(13)	0.1691(3)	0.3097(2)	0.91075(17)	4.82(7)
C(14)	0.1426(2)	0.21943(19)	0.88501(15)	4.24(6)
C(15)	0.4309(2)	0.10338(14)	0.94015(13)	3.01(5)
C(16)	0.0285(2)	0.1936(2)	0.84321(19)	6.27(8)
C(17)	0.5438(2)	-0.03085(15)	0.93157(13)	2.98(5)
C(18)	0.6413(2)	-0.02514(16)	0.88127(14)	3.48(5)
C(19)	0.7476(2)	-0.06608(17)	0.90607(17)	4.35(6)
C(20)	0.7544(2)	-0.1112(2)	0.97841(19)	4.98(7)
C(21)	0.6566(2)	-0.11749(17)	1.02698(17)	3.99(6)
C(22)	0.5475(2)	-0.07845(15)	1.00385(14)	3.17(5)
C(23)	0.6365(2)	0.02611(16)	0.80257(15)	4.04(6)
C(24)	0.6682(2)	-0.0369(2)	0.73416(17)	6.08(8)
C(25)	0.7174(3)	0.1117(2)	0.8019(2)	7.41(9)
C(26)	0.4402(2)	-0.08845(15)	1.05761(14)	3.45(5)
C(27)	0.4290(2)	-0.18785(17)	1.09038(18)	5.23(7)
C(28)	0.4454(2)	-0.0185(2)	1.12513(18)	5.44(7)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.4232	0.2036	0.5668	4.84
H(2)	0.4457	0.0892	0.4730	5.01
H(3)	0.4252	-0.0688	0.5037	5.07
H(4)	0.4228	0.1802	0.7612	5.69
H(5)	0.3824	0.2504	0.6967	5.71
H(6)	0.2892	0.1870	0.7383	5.68
H(7)	0.4319	-0.1750	0.6671	6.03
H(8)	0.2953	-0.1582	0.6698	6.04
H(9)	0.3560	-0.1877	0.5916	6.03
H(10)	0.4306	0.2806	0.9846	5.32
H(11)	0.2835	0.3949	0.9668	6.51
H(12)	0.1123	0.3580	0.9015	5.81
H(13)	0.4987	0.1219	0.9698	3.59
H(14)	-0.0186	0.1556	0.8771	7.55
H(15)	-0.0143	0.2490	0.8299	7.54
H(16)	0.0466	0.1594	0.7967	7.55
H(17)	0.8160	-0.0631	0.8732	5.22
H(18)	0.8279	-0.1381	0.9947	5.98
H(19)	0.6631	-0.1484	1.0763	4.77
H(20)	0.5571	0.0474	0.7949	4.81
H(21)	0.6619	-0.0036	0.6859	7.28
H(22)	0.7478	-0.0584	0.7408	7.27
H(23)	0.6156	-0.0893	0.7335	7.30
H(24)	0.7146	0.1411	0.7517	8.89
H(25)	0.6927	0.1553	0.8411	8.91
H(26)	0.7965	0.0917	0.8126	8.90
H(27)	0.3707	-0.0754	1.0276	4.12
H(28)	0.3653	-0.1901	1.1272	6.28
H(29)	0.4140	-0.2310	1.0489	6.30
H(30)	0.5014	-0.2042	1.1160	6.31
H(31)	0.5181	-0.0264	1.1530	6.53
H(32)	0.4413	0.0437	1.1048	6.55
H(33)	0.3802	-0.0289	1.1597	6.54

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	0.0423(2)	0.03591(18)	0.0340(2)	-0.00496(19)	-0.0024(2)	0.00028(17)
Cl(1)	0.0856(5)	0.0349(2)	0.0616(4)	0.0006(3)	-0.0212(4)	-0.0027(2)
Cl(2)	0.0604(4)	0.0744(4)	0.0546(4)	-0.0302(3)	-0.0111(3)	0.0060(3)
O(1)	0.0365(9)	0.0553(9)	0.0436(9)	0.0035(8)	-0.0001(8)	-0.0073(7)
N(1)	0.0434(12)	0.0381(9)	0.0356(11)	-0.0044(8)	-0.0027(9)	-0.0021(7)
N(2)	0.0332(11)	0.0333(9)	0.0361(11)	0.0024(8)	-0.0008(8)	-0.0025(7)
C(1)	0.0370(13)	0.0485(12)	0.0303(13)	-0.0042(11)	-0.0008(10)	0.0003(10)
C(2)	0.0442(14)	0.0461(12)	0.0409(14)	-0.0077(11)	-0.0002(13)	0.0022(11)
C(3)	0.0561(19)	0.0565(15)	0.0415(16)	-0.0135(13)	0.0026(13)	0.0097(12)
C(4)	0.0533(18)	0.0739(17)	0.0319(14)	-0.0139(14)	-0.0005(12)	0.0028(12)
C(5)	0.0516(17)	0.0726(18)	0.0365(15)	-0.0027(14)	0.0003(12)	-0.0113(13)
C(6)	0.0435(14)	0.0513(13)	0.0390(14)	-0.0027(12)	-0.0028(12)	-0.0067(11)
C(7)	0.081(2)	0.0452(13)	0.0532(18)	-0.0095(15)	0.0094(15)	0.0036(12)
C(8)	0.090(2)	0.0474(13)	0.0535(18)	0.0006(15)	-0.0009(17)	-0.0120(11)
C(9)	0.0430(15)	0.0435(12)	0.0317(12)	0.0086(11)	0.0123(12)	0.0022(10)
C(10)	0.0445(15)	0.0355(11)	0.0375(14)	0.0047(11)	0.0052(11)	-0.0008(10)
C(11)	0.066(2)	0.0408(12)	0.0620(18)	-0.0001(13)	0.0090(16)	-0.0098(12)
C(12)	0.093(2)	0.0370(13)	0.077(2)	0.0133(16)	0.026(2)	-0.0004(13)
C(13)	0.074(2)	0.0537(16)	0.0557(19)	0.0323(15)	0.0235(16)	0.0144(13)
C(14)	0.0491(18)	0.0723(16)	0.0395(16)	0.0233(14)	0.0120(13)	0.0112(12)
C(15)	0.0363(14)	0.0382(11)	0.0398(14)	-0.0060(11)	-0.0021(11)	-0.0012(10)
C(16)	0.059(2)	0.123(2)	0.0565(19)	0.0361(18)	-0.0021(17)	0.0065(19)
C(17)	0.0324(13)	0.0378(11)	0.0430(14)	0.0048(11)	-0.0060(10)	-0.0092(11)
C(18)	0.0408(15)	0.0439(12)	0.0476(15)	0.0019(12)	0.0034(12)	-0.0061(11)
C(19)	0.0354(15)	0.0655(16)	0.0645(19)	0.0088(11)	0.0038(13)	-0.0100(14)
C(20)	0.0457(18)	0.0625(16)	0.081(2)	0.0177(13)	-0.0123(16)	-0.0051(16)
C(21)	0.0455(16)	0.0533(14)	0.0528(17)	0.0125(13)	-0.0051(13)	0.0015(12)
C(22)	0.0380(14)	0.0409(12)	0.0417(15)	0.0034(11)	-0.0030(11)	-0.0076(10)
C(23)	0.0455(16)	0.0535(14)	0.0544(15)	-0.0001(13)	0.0100(13)	-0.0037(12)
C(24)	0.100(2)	0.0743(17)	0.0568(19)	0.0050(19)	0.0131(16)	-0.0053(16)
C(25)	0.118(3)	0.0596(16)	0.104(2)	-0.021(2)	-0.009(2)	0.0083(18)
C(26)	0.0423(16)	0.0493(13)	0.0393(14)	-0.0015(12)	-0.0044(12)	0.0009(11)
C(27)	0.070(2)	0.0535(15)	0.075(2)	-0.0031(15)	0.0113(17)	0.0127(14)
C(28)	0.062(2)	0.0789(18)	0.066(2)	-0.0097(16)	0.0172(16)	-0.0191(16)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
V(1)	Cl(1)	2.2454(6)	V(1)	Cl(2)	2.2705(8)
V(1)	O(1)	1.8222(15)	V(1)	N(1)	1.6377(18)
V(1)	N(2)	2.2165(17)	O(1)	C(9)	1.335(2)
N(1)	C(1)	1.384(2)	N(2)	C(15)	1.286(2)
N(2)	C(17)	1.472(2)	C(1)	C(2)	1.420(2)
C(1)	C(6)	1.412(3)	C(2)	C(3)	1.384(3)
C(2)	C(7)	1.484(3)	C(3)	C(4)	1.361(3)
C(4)	C(5)	1.383(3)	C(5)	C(6)	1.376(3)
C(6)	C(8)	1.499(3)	C(9)	C(10)	1.382(3)
C(9)	C(14)	1.402(3)	C(10)	C(11)	1.410(3)
C(10)	C(15)	1.437(3)	C(11)	C(12)	1.380(4)
C(12)	C(13)	1.351(4)	C(13)	C(14)	1.387(3)
C(14)	C(16)	1.509(4)	C(17)	C(18)	1.390(3)
C(17)	C(22)	1.403(3)	C(18)	C(19)	1.393(3)
C(18)	C(23)	1.524(3)	C(19)	C(20)	1.388(4)
C(20)	C(21)	1.376(4)	C(21)	C(22)	1.400(3)
C(22)	C(26)	1.518(3)	C(23)	C(24)	1.510(3)
C(23)	C(25)	1.518(4)	C(26)	C(27)	1.524(3)
C(26)	C(28)	1.519(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(3)	H(1)	0.950	C(4)	H(2)	0.950
C(5)	H(3)	0.950	C(7)	H(4)	0.950
C(7)	H(5)	0.950	C(7)	H(6)	0.950
C(8)	H(7)	0.950	C(8)	H(8)	0.950
C(8)	H(9)	0.950	C(11)	H(10)	0.950
C(12)	H(11)	0.950	C(13)	H(12)	0.950
C(15)	H(13)	0.950	C(16)	H(14)	0.950
C(16)	H(15)	0.950	C(16)	H(16)	0.950
C(19)	H(17)	0.950	C(20)	H(18)	0.950
C(21)	H(19)	0.950	C(23)	H(20)	0.950
C(24)	H(21)	0.950	C(24)	H(22)	0.950
C(24)	H(23)	0.950	C(25)	H(24)	0.950
C(25)	H(25)	0.950	C(25)	H(26)	0.950
C(26)	H(27)	0.950	C(27)	H(28)	0.950
C(27)	H(29)	0.950	C(27)	H(30)	0.950
C(28)	H(31)	0.950	C(28)	H(32)	0.950
C(28)	H(33)	0.950			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	V(1)	Cl(2)	91.25(2)	Cl(1)	V(1)	O(1)	144.99(5)
Cl(1)	V(1)	N(1)	107.51(6)	Cl(1)	V(1)	N(2)	85.05(4)
Cl(2)	V(1)	O(1)	91.34(5)	Cl(2)	V(1)	N(1)	101.23(6)
Cl(2)	V(1)	N(2)	161.49(5)	O(1)	V(1)	N(1)	106.18(7)
O(1)	V(1)	N(2)	81.59(6)	N(1)	V(1)	N(2)	97.17(8)
V(1)	O(1)	C(9)	139.12(15)	V(1)	N(1)	C(1)	173.19(17)
V(1)	N(2)	C(15)	123.36(15)	V(1)	N(2)	C(17)	124.18(12)
C(15)	N(2)	C(17)	112.45(18)	N(1)	C(1)	C(2)	119.52(19)
N(1)	C(1)	C(6)	118.85(18)	C(2)	C(1)	C(6)	121.6(2)
C(1)	C(2)	C(3)	117.3(2)	C(1)	C(2)	C(7)	121.0(2)
C(3)	C(2)	C(7)	121.6(2)	C(2)	C(3)	C(4)	121.1(2)
C(3)	C(4)	C(5)	121.4(2)	C(4)	C(5)	C(6)	120.7(2)
C(1)	C(6)	C(5)	117.8(2)	C(1)	C(6)	C(8)	120.7(2)
C(5)	C(6)	C(8)	121.4(2)	O(1)	C(9)	C(10)	118.7(2)
O(1)	C(9)	C(14)	119.7(2)	C(10)	C(9)	C(14)	121.5(2)
C(9)	C(10)	C(11)	119.0(2)	C(9)	C(10)	C(15)	121.48(19)
C(11)	C(10)	C(15)	119.5(2)	C(10)	C(11)	C(12)	119.7(2)
C(11)	C(12)	C(13)	119.5(2)	C(12)	C(13)	C(14)	123.6(2)
C(9)	C(14)	C(13)	116.5(2)	C(9)	C(14)	C(16)	119.7(2)
C(13)	C(14)	C(16)	123.7(2)	N(2)	C(15)	C(10)	127.8(2)
N(2)	C(17)	C(18)	119.31(19)	N(2)	C(17)	C(22)	117.66(19)
C(18)	C(17)	C(22)	122.9(2)	C(17)	C(18)	C(19)	117.6(2)
C(17)	C(18)	C(23)	122.7(2)	C(19)	C(18)	C(23)	119.7(2)
C(18)	C(19)	C(20)	120.5(2)	C(19)	C(20)	C(21)	121.2(2)
C(20)	C(21)	C(22)	120.2(2)	C(17)	C(22)	C(21)	117.6(2)
C(17)	C(22)	C(26)	123.3(2)	C(21)	C(22)	C(26)	119.1(2)
C(18)	C(23)	C(24)	112.6(2)	C(18)	C(23)	C(25)	111.6(2)
C(24)	C(23)	C(25)	109.2(2)	C(22)	C(26)	C(27)	111.9(2)
C(22)	C(26)	C(28)	111.3(2)	C(27)	C(26)	C(28)	109.5(2)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(3)	H(1)	119.4	C(4)	C(3)	H(1)	119.4
C(3)	C(4)	H(2)	119.3	C(5)	C(4)	H(2)	119.3
C(4)	C(5)	H(3)	119.6	C(6)	C(5)	H(3)	119.6
C(2)	C(7)	H(4)	109.5	C(2)	C(7)	H(5)	109.5
C(2)	C(7)	H(6)	109.4	H(4)	C(7)	H(5)	109.5
H(4)	C(7)	H(6)	109.5	H(5)	C(7)	H(6)	109.5
C(6)	C(8)	H(7)	108.6	C(6)	C(8)	H(8)	110.0
C(6)	C(8)	H(9)	109.8	H(7)	C(8)	H(8)	109.5
H(7)	C(8)	H(9)	109.5	H(8)	C(8)	H(9)	109.5
C(10)	C(11)	H(10)	120.1	C(12)	C(11)	H(10)	120.1
C(11)	C(12)	H(11)	120.2	C(13)	C(12)	H(11)	120.2
C(12)	C(13)	H(12)	118.2	C(14)	C(13)	H(12)	118.2
N(2)	C(15)	H(13)	116.1	C(10)	C(15)	H(13)	116.1
C(14)	C(16)	H(14)	109.0	C(14)	C(16)	H(15)	109.8
C(14)	C(16)	H(16)	109.6	H(14)	C(16)	H(15)	109.5
H(14)	C(16)	H(16)	109.5	H(15)	C(16)	H(16)	109.5
C(18)	C(19)	H(17)	119.7	C(20)	C(19)	H(17)	119.7
C(19)	C(20)	H(18)	119.4	C(21)	C(20)	H(18)	119.4
C(20)	C(21)	H(19)	119.9	C(22)	C(21)	H(19)	119.9
C(18)	C(23)	H(20)	107.7	C(24)	C(23)	H(20)	107.7
C(25)	C(23)	H(20)	107.7	C(23)	C(24)	H(21)	110.5
C(23)	C(24)	H(22)	108.7	C(23)	C(24)	H(23)	109.1
H(21)	C(24)	H(22)	109.5	H(21)	C(24)	H(23)	109.5
H(22)	C(24)	H(23)	109.5	C(23)	C(25)	H(24)	109.8
C(23)	C(25)	H(25)	110.2	C(23)	C(25)	H(26)	108.5
H(24)	C(25)	H(25)	109.5	H(24)	C(25)	H(26)	109.5
H(25)	C(25)	H(26)	109.5	C(22)	C(26)	H(27)	108.0
C(27)	C(26)	H(27)	108.0	C(28)	C(26)	H(27)	108.0
C(26)	C(27)	H(28)	109.5	C(26)	C(27)	H(29)	110.0
C(26)	C(27)	H(30)	108.9	H(28)	C(27)	H(29)	109.5
H(28)	C(27)	H(30)	109.5	H(29)	C(27)	H(30)	109.5
C(26)	C(28)	H(31)	109.4	C(26)	C(28)	H(32)	109.4
C(26)	C(28)	H(33)	109.7	H(31)	C(28)	H(32)	109.5
H(31)	C(28)	H(33)	109.5	H(32)	C(28)	H(33)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle
Cl(1)	V(1)	O(1)	C(9)	103.8(2)
Cl(1)	V(1)	N(2)	C(15)	-162.21(17)
Cl(2)	V(1)	O(1)	C(9)	-162.1(2)
Cl(2)	V(1)	N(2)	C(15)	-83.1(2)
O(1)	V(1)	N(1)	C(1)	-79.2(12)
O(1)	V(1)	N(2)	C(15)	-14.67(17)
N(2)	V(1)	O(1)	C(9)	35.1(2)
N(1)	V(1)	N(2)	C(17)	-88.00(16)
V(1)	O(1)	C(9)	C(10)	-36.4(3)
V(1)	N(1)	C(1)	C(2)	103.2(12)
V(1)	N(2)	C(15)	C(10)	1.5(3)
V(1)	N(2)	C(17)	C(22)	-95.6(2)
C(15)	N(2)	C(17)	C(22)	85.5(2)
N(1)	C(1)	C(2)	C(3)	-177.8(2)
N(1)	C(1)	C(6)	C(5)	178.4(2)
C(2)	C(1)	C(6)	C(5)	1.1(3)
C(6)	C(1)	C(2)	C(3)	-0.5(3)
C(1)	C(2)	C(3)	C(4)	0.1(3)
C(2)	C(3)	C(4)	C(5)	-0.2(3)
C(4)	C(5)	C(6)	C(1)	-1.2(3)
O(1)	C(9)	C(10)	C(11)	-176.1(2)
O(1)	C(9)	C(14)	C(13)	177.6(2)
C(10)	C(9)	C(14)	C(13)	-1.5(3)
C(14)	C(9)	C(10)	C(11)	3.0(3)
C(9)	C(10)	C(11)	C(12)	-1.6(3)
C(11)	C(10)	C(15)	N(2)	-171.2(2)
C(10)	C(11)	C(12)	C(13)	-1.3(4)
C(12)	C(13)	C(14)	C(9)	-1.5(4)
N(2)	C(17)	C(18)	C(19)	173.75(19)
N(2)	C(17)	C(22)	C(21)	-172.96(19)
C(18)	C(17)	C(22)	C(21)	3.2(3)
C(22)	C(17)	C(18)	C(19)	-2.3(3)
C(17)	C(18)	C(19)	C(20)	0.3(3)
C(17)	C(18)	C(23)	C(25)	114.3(2)
C(19)	C(18)	C(23)	C(25)	-63.8(3)
C(18)	C(19)	C(20)	C(21)	0.7(4)
C(20)	C(21)	C(22)	C(17)	-2.1(3)

atom1	atom2	atom3	atom4	angle
Cl(1)	V(1)	N(1)	C(1)	110.5(12)
Cl(1)	V(1)	N(2)	C(17)	19.07(15)
Cl(2)	V(1)	N(1)	C(1)	15.5(12)
Cl(2)	V(1)	N(2)	C(17)	98.2(2)
N(1)	V(1)	O(1)	C(9)	-60.0(2)
O(1)	V(1)	N(2)	C(17)	166.62(16)
N(1)	V(1)	N(2)	C(15)	90.72(18)
N(2)	V(1)	N(1)	C(1)	-162.5(12)
V(1)	O(1)	C(9)	C(14)	144.4(2)
V(1)	N(1)	C(1)	C(6)	-74.1(12)
V(1)	N(2)	C(17)	C(18)	88.1(2)
C(15)	N(2)	C(17)	C(18)	-90.7(2)
C(17)	N(2)	C(15)	C(10)	-179.7(2)
N(1)	C(1)	C(2)	C(7)	0.8(3)
N(1)	C(1)	C(6)	C(8)	0.7(3)
C(2)	C(1)	C(6)	C(8)	-176.5(2)
C(6)	C(1)	C(2)	C(7)	178.0(2)
C(7)	C(2)	C(3)	C(4)	-178.5(2)
C(3)	C(4)	C(5)	C(6)	0.7(4)
C(4)	C(5)	C(6)	C(8)	176.4(2)
O(1)	C(9)	C(10)	C(15)	6.1(3)
O(1)	C(9)	C(14)	C(16)	-3.8(3)
C(10)	C(9)	C(14)	C(16)	177.1(2)
C(14)	C(9)	C(10)	C(15)	-174.7(2)
C(9)	C(10)	C(15)	N(2)	6.5(3)
C(15)	C(10)	C(11)	C(12)	176.2(2)
C(11)	C(12)	C(13)	C(14)	2.9(4)
C(12)	C(13)	C(14)	C(16)	180.0(2)
N(2)	C(17)	C(18)	C(23)	-4.3(3)
N(2)	C(17)	C(22)	C(26)	6.4(3)
C(18)	C(17)	C(22)	C(26)	-177.4(2)
C(22)	C(17)	C(18)	C(23)	179.6(2)
C(17)	C(18)	C(23)	C(24)	-122.4(2)
C(19)	C(18)	C(23)	C(24)	59.5(3)
C(23)	C(18)	C(19)	C(20)	178.5(2)
C(19)	C(20)	C(21)	C(22)	0.2(3)
C(20)	C(21)	C(22)	C(26)	178.5(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C(17)	C(22)	C(26)	C(27)	136.2(2)	C(17)	C(22)	C(26)	C(28)	-100.9(2)
C(21)	C(22)	C(26)	C(27)	-44.4(3)	C(21)	C(22)	C(26)	C(28)	78.5(2)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Cl(2)	C(28)[1)]	3.568(3)	O(1)	C(4)[2)]	3.498(3)
O(1)	C(5)[2)]	3.194(2)	C(4)	O(1)[1)]	3.498(3)
C(5)	O(1)[1)]	3.194(3)	C(5)	C(9)[1)]	3.470(3)
C(9)	C(5)[2)]	3.470(3)	C(28)	Cl(2)[2)]	3.568(3)

Symmetry Operators:

(1) -X+1/2,-Y,Z+1/2-1 (2) -X+1/2,-Y,Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
V(1)	H(2)[1)]	3.563	Cl(1)	H(1)[2)]	3.064
Cl(1)	H(5)[2)]	3.136	Cl(1)	H(18)[3)]	3.535
Cl(1)	H(19)[3)]	3.511	Cl(1)	H(24)[2)]	3.387
Cl(2)	H(2)[1)]	3.012	Cl(2)	H(15)[4)]	3.208
Cl(2)	H(30)[3)]	3.259	Cl(2)	H(31)[5)]	3.510
Cl(2)	H(32)[5)]	3.525	Cl(2)	H(33)[5)]	3.107
O(1)	H(2)[1)]	3.198	O(1)	H(3)[1)]	2.624
N(1)	H(33)[5)]	2.988	C(1)	H(33)[5)]	2.859
C(2)	H(28)[5)]	3.056	C(2)	H(33)[5)]	3.239
C(3)	H(18)[6)]	3.250	C(3)	H(27)[5)]	3.440
C(3)	H(28)[5)]	3.305	C(3)	H(29)[7)]	3.466
C(4)	H(18)[6)]	2.954	C(4)	H(27)[5)]	3.342
C(4)	H(29)[7)]	3.558	C(5)	H(10)[2)]	3.325
C(5)	H(11)[2)]	3.590	C(5)	H(14)[5)]	3.597
C(5)	H(27)[5)]	3.500	C(6)	H(33)[5)]	3.270
C(7)	H(7)[7)]	3.524	C(7)	H(23)[7)]	3.242
C(7)	H(28)[5)]	3.054	C(8)	H(10)[2)]	3.247
C(8)	H(24)[2)]	3.593	C(8)	H(25)[2)]	2.833
C(9)	H(3)[1)]	2.757	C(9)	H(9)[1)]	3.469
C(10)	H(3)[1)]	3.508	C(10)	H(9)[1)]	3.471
C(11)	H(3)[7)]	3.451	C(11)	H(7)[7)]	3.303
C(11)	H(9)[7)]	3.392	C(11)	H(9)[1)]	3.466
C(11)	H(14)[8)]	3.306	C(12)	H(9)[1)]	3.496
C(12)	H(13)[9)]	3.422	C(12)	H(21)[7)]	3.344
C(12)	H(22)[7)]	3.572	C(12)	H(23)[7)]	3.518
C(13)	H(9)[1)]	3.540	C(13)	H(10)[9)]	3.459
C(13)	H(13)[9)]	2.953	C(13)	H(22)[7)]	3.319
C(13)	H(32)[9)]	3.308	C(14)	H(3)[1)]	3.038
C(14)	H(9)[1)]	3.539	C(14)	H(10)[9)]	3.250
C(15)	H(12)[8)]	3.418	C(16)	H(3)[1)]	3.294
C(16)	H(10)[9)]	3.147	C(16)	H(26)[10)]	3.024
C(18)	H(5)[2)]	3.464	C(18)	H(11)[8)]	3.555
C(19)	H(5)[2)]	3.461	C(19)	H(11)[8)]	3.279
C(19)	H(29)[11)]	3.520	C(20)	H(1)[2)]	3.390
C(20)	H(2)[12)]	3.380	C(20)	H(11)[8)]	3.228
C(20)	H(28)[11)]	3.570	C(20)	H(29)[11)]	2.907
C(21)	H(1)[2)]	3.132	C(21)	H(11)[8)]	3.471

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(22)	H(1)[2)]	3.339	C(22)	H(12)[8)]	3.596
C(24)	H(5)[2)]	3.292	C(24)	H(11)[2)]	3.591
C(25)	H(7)[7)]	3.502	C(25)	H(8)[7)]	3.308
C(25)	H(9)[7)]	3.476	C(25)	H(14)[13)]	3.285
C(27)	H(1)[2)]	3.503	C(27)	H(6)[1)]	3.510
C(27)	H(16)[1)]	3.540	C(27)	H(18)[3)]	3.081
C(28)	H(12)[8)]	2.986	C(28)	H(16)[1)]	3.538
H(1)	Cl(1)[7)]	3.064	H(1)	C(20)[7)]	3.390
H(1)	C(21)[7)]	3.132	H(1)	C(22)[7)]	3.339
H(1)	C(27)[7)]	3.503	H(1)	H(18)[6)]	3.189
H(1)	H(19)[7)]	3.359	H(1)	H(28)[5)]	3.400
H(1)	H(29)[7)]	2.840	H(1)	H(30)[7)]	3.476
H(2)	V(1)[5)]	3.563	H(2)	Cl(2)[5)]	3.012
H(2)	O(1)[5)]	3.198	H(2)	C(20)[6)]	3.380
H(2)	H(17)[6)]	3.187	H(2)	H(18)[6)]	2.659
H(2)	H(29)[7)]	3.025	H(2)	H(30)[7)]	3.355
H(3)	O(1)[5)]	2.624	H(3)	C(9)[5)]	2.757
H(3)	C(10)[5)]	3.508	H(3)	C(11)[2)]	3.451
H(3)	C(14)[5)]	3.038	H(3)	C(16)[5)]	3.294
H(3)	H(10)[2)]	2.690	H(3)	H(11)[2)]	3.347
H(3)	H(14)[5)]	2.693	H(4)	H(7)[7)]	2.894
H(4)	H(23)[7)]	3.307	H(5)	Cl(1)[7)]	3.136
H(5)	C(18)[7)]	3.464	H(5)	C(19)[7)]	3.461
H(5)	C(24)[7)]	3.292	H(5)	H(7)[7)]	3.289
H(5)	H(22)[7)]	3.262	H(5)	H(23)[7)]	2.569
H(5)	H(28)[5)]	3.139	H(6)	C(27)[5)]	3.510
H(6)	H(23)[7)]	3.388	H(6)	H(28)[5)]	2.565
H(6)	H(33)[5)]	3.232	H(7)	C(7)[2)]	3.524
H(7)	C(11)[2)]	3.303	H(7)	C(25)[2)]	3.502
H(7)	H(4)[2)]	2.894	H(7)	H(5)[2)]	3.289
H(7)	H(10)[2)]	3.070	H(7)	H(24)[2)]	3.381
H(7)	H(25)[2)]	2.791	H(8)	C(25)[2)]	3.308
H(8)	H(15)[4)]	3.417	H(8)	H(24)[2)]	3.151
H(8)	H(25)[2)]	2.660	H(8)	H(32)[5)]	3.304
H(8)	H(33)[5)]	3.313	H(9)	C(9)[5)]	3.469
H(9)	C(10)[5)]	3.471	H(9)	C(11)[2)]	3.392
H(9)	C(11)[5)]	3.466	H(9)	C(12)[5)]	3.496

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(9)	C(13)[5)]	3.540	H(9)	C(14)[5)]	3.539
H(9)	C(25)[2)]	3.476	H(9)	H(10)[2)]	2.758
H(9)	H(13)[2)]	3.327	H(9)	H(25)[2)]	2.567
H(10)	C(5)[7)]	3.325	H(10)	C(8)[7)]	3.247
H(10)	C(13)[8)]	3.459	H(10)	C(14)[8)]	3.250
H(10)	C(16)[8)]	3.147	H(10)	H(3)[7)]	2.690
H(10)	H(7)[7)]	3.070	H(10)	H(9)[7)]	2.758
H(10)	H(12)[8)]	3.431	H(10)	H(14)[8)]	2.583
H(10)	H(15)[8)]	3.240	H(11)	C(5)[7)]	3.590
H(11)	C(18)[9)]	3.555	H(11)	C(19)[9)]	3.279
H(11)	C(20)[9)]	3.228	H(11)	C(21)[9)]	3.471
H(11)	C(24)[7)]	3.591	H(11)	H(3)[7)]	3.347
H(11)	H(13)[9)]	3.379	H(11)	H(14)[8)]	3.533
H(11)	H(18)[9)]	3.554	H(11)	H(21)[7)]	3.032
H(11)	H(23)[7)]	3.594	H(11)	H(25)[9)]	3.494
H(12)	C(15)[9)]	3.418	H(12)	C(22)[9)]	3.596
H(12)	C(28)[9)]	2.986	H(12)	H(10)[9)]	3.431
H(12)	H(13)[9)]	2.546	H(12)	H(21)[7)]	3.536
H(12)	H(22)[7)]	3.119	H(12)	H(31)[9)]	2.776
H(12)	H(32)[9)]	2.376	H(13)	C(12)[8)]	3.422
H(13)	C(13)[8)]	2.953	H(13)	H(9)[7)]	3.327
H(13)	H(11)[8)]	3.379	H(13)	H(12)[8)]	2.546
H(14)	C(5)[1)]	3.597	H(14)	C(11)[9)]	3.306
H(14)	C(25)[10)]	3.285	H(14)	H(3)[1)]	2.693
H(14)	H(10)[9)]	2.583	H(14)	H(11)[9)]	3.533
H(14)	H(25)[10)]	3.295	H(14)	H(26)[10)]	2.515
H(15)	Cl(2)[14)]	3.208	H(15)	H(8)[14)]	3.417
H(15)	H(10)[9)]	3.240	H(15)	H(25)[10)]	3.551
H(15)	H(26)[10)]	3.097	H(15)	H(32)[9)]	3.189
H(16)	C(27)[5)]	3.540	H(16)	C(28)[5)]	3.538
H(16)	H(26)[10)]	2.978	H(16)	H(28)[5)]	3.077
H(16)	H(30)[5)]	3.182	H(16)	H(31)[5)]	3.171
H(16)	H(33)[5)]	3.087	H(17)	H(2)[12)]	3.187
H(17)	H(28)[11)]	3.551	H(17)	H(29)[11)]	3.394
H(18)	Cl(1)[11)]	3.535	H(18)	C(3)[12)]	3.250
H(18)	C(4)[12)]	2.954	H(18)	C(27)[11)]	3.081
H(18)	H(1)[12)]	3.189	H(18)	H(2)[12)]	2.659

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(18)	H(11)[8)]	3.554	H(18)	H(28)[11)]	3.229
H(18)	H(29)[11)]	2.223	H(18)	H(30)[11)]	3.513
H(19)	Cl(1)[11)]	3.511	H(19)	H(1)[2)]	3.359
H(19)	H(21)[12)]	3.463	H(19)	H(24)[12)]	3.283
H(21)	C(12)[2)]	3.344	H(21)	H(11)[2)]	3.032
H(21)	H(12)[2)]	3.536	H(21)	H(19)[6)]	3.463
H(22)	C(12)[2)]	3.572	H(22)	C(13)[2)]	3.319
H(22)	H(5)[2)]	3.262	H(22)	H(12)[2)]	3.119
H(22)	H(31)[6)]	3.252	H(23)	C(7)[2)]	3.242
H(23)	C(12)[2)]	3.518	H(23)	H(4)[2)]	3.307
H(23)	H(5)[2)]	2.569	H(23)	H(6)[2)]	3.388
H(23)	H(11)[2)]	3.594	H(24)	Cl(1)[7)]	3.387
H(24)	C(8)[7)]	3.593	H(24)	H(7)[7)]	3.381
H(24)	H(8)[7)]	3.151	H(24)	H(19)[6)]	3.283
H(25)	C(8)[7)]	2.833	H(25)	H(7)[7)]	2.791
H(25)	H(8)[7)]	2.660	H(25)	H(9)[7)]	2.567
H(25)	H(11)[8)]	3.494	H(25)	H(14)[13)]	3.295
H(25)	H(15)[13)]	3.551	H(26)	C(16)[13)]	3.024
H(26)	H(14)[13)]	2.515	H(26)	H(15)[13)]	3.097
H(26)	H(16)[13)]	2.978	H(26)	H(31)[6)]	3.542
H(27)	C(3)[1)]	3.440	H(27)	C(4)[1)]	3.342
H(27)	C(5)[1)]	3.500	H(28)	C(2)[1)]	3.056
H(28)	C(3)[1)]	3.305	H(28)	C(7)[1)]	3.054
H(28)	C(20)[3)]	3.570	H(28)	H(1)[1)]	3.400
H(28)	H(5)[1)]	3.139	H(28)	H(6)[1)]	2.565
H(28)	H(16)[1)]	3.077	H(28)	H(17)[3)]	3.551
H(28)	H(18)[3)]	3.229	H(29)	C(3)[2)]	3.466
H(29)	C(4)[2)]	3.558	H(29)	C(19)[3)]	3.520
H(29)	C(20)[3)]	2.907	H(29)	H(1)[2)]	2.840
H(29)	H(2)[2)]	3.025	H(29)	H(17)[3)]	3.394
H(29)	H(18)[3)]	2.223	H(30)	Cl(2)[11)]	3.259
H(30)	H(1)[2)]	3.476	H(30)	H(2)[2)]	3.355
H(30)	H(16)[1)]	3.182	H(30)	H(18)[3)]	3.513
H(31)	Cl(2)[1)]	3.510	H(31)	H(12)[8)]	2.776
H(31)	H(16)[1)]	3.171	H(31)	H(22)[12)]	3.252
H(31)	H(26)[12)]	3.542	H(32)	Cl(2)[1)]	3.525
H(32)	C(13)[8)]	3.308	H(32)	H(8)[1)]	3.304

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(32)	H(12)[8)]	2.376	H(32)	H(15)[8)]	3.189
H(33)	Cl(2)[1)]	3.107	H(33)	N(1)[1)]	2.988
H(33)	C(1)[1)]	2.859	H(33)	C(2)[1)]	3.239
H(33)	C(6)[1)]	3.270	H(33)	H(6)[1)]	3.232
H(33)	H(8)[1)]	3.313	H(33)	H(16)[1)]	3.087

Symmetry Operators:

(1) -X+1/2,-Y,Z+1/2
(2) -X+1,Y+1/2-1,-Z+1/2+1
(3) X+1/2-1,-Y+1/2-1,-Z+2
(4) -X,Y+1/2-1,-Z+1/2+1
(5) -X+1/2,-Y,Z+1/2-1
(6) -X+1/2+1,-Y,Z+1/2-1
(7) -X+1,Y+1/2,-Z+1/2+1
(8) X+1/2,-Y+1/2,-Z+2
(9) X+1/2-1,-Y+1/2,-Z+2
(10) X-1,Y,Z
(11) X+1/2,-Y+1/2-1,-Z+2
(12) -X+1/2+1,-Y,Z+1/2
(13) X+1,Y,Z
(14) -X,Y+1/2,-Z+1/2+1

X-ray Structure Report for VCl_2(N-2,6-$Me_2C_6H_3$)[O-2-^{t}Bu-6-{(2,6-^{i}$Pr_2C_6H_3$)N=CH}C_6H_3]
(**1c**)

February 13, 2007

Experimental

Data Collection

A black block crystal of $C_{31}H_{39}Cl_2N_2OV$ having approximate dimensions of 0.34 x 0.30 x 0.15 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 240 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive monoclinic cell with dimensions:

a = 18.7923(3) Å
b = 19.5289(4) Å β = 119.0630(7)^o
c = 19.3387(4) Å
V = 6203.5(2) Å^3

For Z = 8 and F.W. = 577.51, the calculated density is 1.237 g/cm^3. The systematic absences of:

h0l: l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.8^o. A total of 72 oscillation images were collected. A sweep of data was done using ω scans from 0.0 to 180.0^o in 5.0^o step, at χ=45.0^o and ϕ = 30.0^o. The exposure rate was 140.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 180.0^o in 5.0^o step, at χ=45.0^o and ϕ = 210.0^o. The exposure rate was 140.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 93174 reflections that were collected, 14073 were unique (R_{int} = 0.037); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 5.165 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.707 to 0.925. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 9952 observed reflections and 745 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0332$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.0951$$

The standard deviation of an observation of unit weight[4] was 1.00. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.27 and -0.29 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku/MSC (2000-2006). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{31}H_{39}Cl_2N_2OV$
Formula Weight	577.51
Crystal Color, Habit	black, block
Crystal Dimensions	0.34 X 0.30 X 0.15 mm
Crystal System	monoclinic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 240.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 18.7923(3) Å b = 19.5289(4) Å c = 19.3387(4) Å β = 119.0630(7) ^{o} V = 6203.5(2) $Å^3$
Space Group	$P2_1/c$ (#14)
Z value	8
D_{calc}	1.237 g/cm^3
F_{000}	2432.00
μ(MoKα)	5.165 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	72 exposures
ω oscillation Range (χ=45.0, ϕ=30.0)	0.0 - 180.0^{o}
Exposure Rate	140.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=210.0)	0.0 - 180.0^{o}
Exposure Rate	140.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.8^{o}
No. of Reflections Measured	Total: 93174 Unique: 14073 (R_{int} = 0.037)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.707 - 0.925)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0010Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.8^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	9952
No. Variables	745
Reflection/Parameter Ratio	13.36
Residuals: R1 (I>2.00σ(I))	0.0332
Residuals: wR2 (I>2.00σ(I))	0.0951
Goodness of Fit Indicator	1.001
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.27 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.29 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
V(1)	0.46311(2)	0.24917(2)	0.20405(2)	2.033(6)
V(2)	0.96280(2)	0.20605(2)	0.21636(2)	2.163(6)
Cl(1)	0.52993(3)	0.32818(3)	0.29952(3)	3.110(11)
Cl(2)	0.45247(4)	0.17931(3)	0.29324(3)	3.398(12)
Cl(3)	1.05356(4)	0.27858(3)	0.30963(3)	3.709(12)
Cl(4)	0.98829(4)	0.12390(3)	0.30721(4)	4.244(13)
O(1)	0.47948(8)	0.17724(7)	0.15421(8)	2.40(2)
O(2)	0.89497(8)	0.27607(7)	0.16386(8)	2.57(2)
N(1)	0.51317(10)	0.30610(8)	0.13900(10)	2.28(3)
N(2)	0.36823(10)	0.27499(9)	0.14956(10)	2.53(3)
N(3)	0.85230(10)	0.14542(8)	0.14210(10)	2.22(3)
N(4)	1.01361(10)	0.18712(9)	0.16972(10)	2.71(3)
C(1)	0.46595(11)	0.16264(10)	0.08088(11)	2.10(3)
C(2)	0.48520(12)	0.21418(11)	0.04180(12)	2.42(4)
C(3)	0.47911(14)	0.20046(12)	-0.03246(13)	3.02(4)
C(4)	0.45131(14)	0.13746(12)	-0.06668(13)	3.33(5)
C(5)	0.42964(13)	0.08782(12)	-0.02811(13)	2.98(4)
C(6)	0.43640(11)	0.09780(10)	0.04604(12)	2.33(4)
C(7)	0.51267(12)	0.28075(11)	0.07658(12)	2.56(4)
C(8)	0.41167(13)	0.04314(11)	0.08689(13)	2.65(4)
C(9)	0.37774(15)	-0.02140(12)	0.03531(15)	3.55(5)
C(10)	0.48505(14)	0.02198(12)	0.16602(14)	3.39(5)
C(11)	0.34314(14)	0.07103(13)	0.10050(16)	3.71(5)
C(12)	0.54931(13)	0.37408(10)	0.16054(12)	2.54(4)
C(13)	0.63400(13)	0.37893(11)	0.20897(13)	2.81(4)
C(14)	0.66811(15)	0.44418(13)	0.22629(16)	3.69(5)
C(15)	0.62023(16)	0.50200(13)	0.19702(16)	3.99(6)
C(16)	0.53725(16)	0.49587(12)	0.15082(16)	3.80(5)
C(17)	0.49938(14)	0.43192(11)	0.13145(14)	3.05(4)
C(18)	0.68716(14)	0.31574(13)	0.24037(15)	3.48(5)
C(19)	0.75024(18)	0.32190(17)	0.32709(19)	5.98(7)
C(20)	0.7276(2)	0.30091(16)	0.1900(2)	5.87(8)
C(21)	0.40775(15)	0.42717(13)	0.08103(15)	3.67(5)
C(22)	0.3781(2)	0.4537(2)	-0.00275(19)	6.60(9)
C(23)	0.36525(16)	0.46333(14)	0.12130(19)	4.84(6)
C(24)	0.28857(12)	0.29859(11)	0.10893(13)	2.71(4)
C(25)	0.25228(13)	0.32354(13)	0.15277(14)	3.43(5)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(26)	0.17369(14)	0.35091(14)	0.11074(16)	4.05(5)
C(27)	0.13411(14)	0.35337(13)	0.02989(16)	4.04(5)
C(28)	0.16985(13)	0.32752(13)	-0.01268(15)	3.57(5)
C(29)	0.24643(13)	0.29792(11)	0.02527(13)	2.97(4)
C(30)	0.29601(17)	0.32073(18)	0.24142(16)	5.60(7)
C(31)	0.28251(14)	0.26578(14)	-0.02150(14)	3.95(5)
C(32)	0.83235(11)	0.29197(10)	0.09256(12)	2.27(4)
C(33)	0.77628(12)	0.23992(10)	0.05208(12)	2.41(4)
C(34)	0.70559(13)	0.25520(12)	-0.01899(14)	3.37(4)
C(35)	0.69369(15)	0.32072(13)	-0.04859(15)	4.23(5)
C(36)	0.75183(14)	0.37106(13)	-0.00893(14)	3.75(5)
C(37)	0.82294(12)	0.35926(11)	0.06246(13)	2.74(4)
C(38)	0.78868(12)	0.17074(11)	0.08160(12)	2.37(4)
C(39)	0.88652(14)	0.41556(11)	0.10518(15)	3.48(5)
C(40)	0.86142(18)	0.48308(13)	0.05832(19)	5.52(7)
C(41)	0.89572(19)	0.42975(14)	0.18689(17)	4.89(6)
C(42)	0.96894(15)	0.39452(14)	0.11351(18)	4.66(6)
C(43)	0.84418(12)	0.07271(10)	0.15496(12)	2.39(4)
C(44)	0.80600(13)	0.05351(11)	0.19884(14)	2.91(4)
C(45)	0.79666(15)	-0.01664(12)	0.20697(16)	3.81(5)
C(46)	0.82459(16)	-0.06477(12)	0.17375(17)	4.17(6)
C(47)	0.86350(15)	-0.04466(12)	0.13226(16)	3.51(5)
C(48)	0.87398(12)	0.02430(11)	0.12155(13)	2.64(4)
C(49)	0.77453(15)	0.10547(13)	0.23586(16)	3.81(6)
C(50)	0.68149(17)	0.11299(17)	0.1855(2)	6.39(9)
C(51)	0.7987(2)	0.08820(19)	0.3213(2)	7.00(10)
C(52)	0.91426(13)	0.04571(11)	0.07336(13)	2.83(4)
C(53)	0.85662(16)	0.03629(14)	-0.01510(15)	4.16(6)
C(54)	0.99473(15)	0.00840(14)	0.09890(16)	4.01(6)
C(55)	1.06175(12)	0.17782(11)	0.13449(14)	2.82(4)
C(56)	1.14169(14)	0.15187(13)	0.18143(16)	3.65(5)
C(57)	1.18886(15)	0.14358(14)	0.14435(19)	4.42(6)
C(58)	1.15897(17)	0.15895(14)	0.06613(19)	4.48(7)
C(59)	1.08052(16)	0.18360(13)	0.02033(17)	3.89(6)
C(60)	1.03065(13)	0.19434(11)	0.05386(14)	2.98(5)
C(61)	1.17360(16)	0.13415(19)	0.26677(17)	5.66(7)
C(62)	0.94663(15)	0.22250(14)	0.00486(15)	4.10(6)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.4911	0.2365	-0.0626	3.76
H(2)	0.4547	0.1252	-0.1155	4.10
H(3)	0.4099	0.0436	-0.0566	3.49
H(4)	0.5355	0.3115	0.0481	3.13
H(5)	0.4179	-0.0397	0.0241	4.22
H(6)	0.3640	-0.0545	0.0628	4.23
H(7)	0.3306	-0.0098	-0.0128	4.23
H(8)	0.5055	0.0611	0.1991	3.90
H(9)	0.4688	-0.0115	0.1912	3.92
H(10)	0.5263	0.0035	0.1566	3.91
H(11)	0.3618	0.1109	0.1326	4.76
H(12)	0.2974	0.0824	0.0511	4.75
H(13)	0.3280	0.0373	0.1263	4.75
H(14)	0.7282	0.4457	0.2598	4.60
H(15)	0.6432	0.5493	0.2067	5.07
H(16)	0.5028	0.5383	0.1296	4.74
H(17)	0.6525	0.2725	0.2394	4.13
H(18)	0.7805	0.2806	0.3445	6.24
H(19)	0.7860	0.3587	0.3337	6.23
H(20)	0.7237	0.3304	0.3573	6.23
H(21)	0.7589	0.2601	0.2079	7.74
H(22)	0.6872	0.2957	0.1362	7.76
H(23)	0.7622	0.3382	0.1949	7.72
H(24)	0.3927	0.3767	0.0784	4.07
H(25)	0.3219	0.4440	-0.0354	7.36
H(26)	0.3865	0.5018	-0.0006	7.37
H(27)	0.4086	0.4322	-0.0240	7.37
H(28)	0.3083	0.4550	0.0925	5.53
H(29)	0.3862	0.4467	0.1738	5.53
H(30)	0.3751	0.5111	0.1226	5.54
H(31)	0.1480	0.3701	0.1407	4.68
H(32)	0.0749	0.3730	-0.0042	4.37
H(33)	0.1368	0.3253	-0.0711	3.81
H(34)	0.3292	0.2809	0.2580	6.61
H(35)	0.3292	0.3602	0.2622	6.61
H(36)	0.2578	0.3189	0.2602	6.62
H(37)	0.2404	0.2556	-0.0734	4.48

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(38)	0.3200	0.2968	-0.0243	4.50
H(39)	0.3100	0.2248	0.0038	4.46
H(40)	0.6641	0.2167	-0.0475	3.58
H(41)	0.6424	0.3299	-0.0963	4.19
H(42)	0.7433	0.4215	-0.0309	3.96
H(43)	0.7423	0.1385	0.0492	2.84
H(44)	0.9019	0.5169	0.0856	5.62
H(45)	0.8561	0.4756	0.0075	5.63
H(46)	0.8109	0.4983	0.0527	5.61
H(47)	0.9347	0.4649	0.2126	5.49
H(48)	0.8446	0.4437	0.1809	5.49
H(49)	0.9130	0.3891	0.2178	5.49
H(50)	0.9878	0.3544	0.1452	5.41
H(51)	0.9627	0.3854	0.0626	5.42
H(52)	1.0073	0.4304	0.1378	5.41
H(53)	0.7654	-0.0310	0.2364	4.94
H(54)	0.8176	-0.1153	0.1813	5.38
H(55)	0.8875	-0.0805	0.1113	4.43
H(56)	0.8027	0.1541	0.2359	5.05
H(57)	0.6623	0.1452	0.2094	8.26
H(58)	0.6683	0.1283	0.1340	8.28
H(59)	0.6565	0.0698	0.1818	8.26
H(60)	0.7800	0.1230	0.3429	9.66
H(61)	0.7745	0.0457	0.3222	9.67
H(62)	0.8562	0.0846	0.3518	9.67
H(63)	0.9264	0.0967	0.0815	3.59
H(64)	0.8818	0.0520	-0.0445	5.00
H(65)	0.8437	-0.0109	-0.0257	5.02
H(66)	0.8082	0.0616	-0.0300	5.02
H(67)	1.0291	0.0141	0.1542	5.09
H(68)	0.9841	-0.0389	0.0871	5.09
H(69)	1.0208	0.0265	0.0714	5.10
H(70)	1.2508	0.1249	0.1799	5.56
H(71)	1.1849	0.1481	0.0338	6.11
H(72)	1.0535	0.1948	-0.0447	5.20
H(73)	1.1709	0.1735	0.2943	6.39
H(74)	1.1413	0.0987	0.2709	6.39

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(75)	1.2285	0.1192	0.2893	6.42
H(76)	0.9083	0.1922	0.0071	5.00
H(77)	0.9432	0.2660	0.0252	5.01
H(78)	0.9351	0.2274	-0.0485	5.02

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	0.02741(17)	0.02634(18)	0.02164(17)	0.00196(14)	0.01046(14)	-0.00165(14)
V(2)	0.02522(17)	0.02667(18)	0.02546(18)	0.00083(14)	0.00851(14)	0.00098(14)
Cl(1)	0.0470(3)	0.0399(3)	0.0311(2)	-0.0091(2)	0.0189(2)	-0.0114(2)
Cl(2)	0.0635(3)	0.0363(3)	0.0357(3)	-0.0011(2)	0.0291(2)	0.0025(2)
Cl(3)	0.0465(3)	0.0374(3)	0.0352(3)	-0.0065(2)	0.0027(2)	-0.0037(2)
Cl(4)	0.0512(3)	0.0472(3)	0.0392(3)	-0.0106(2)	0.0034(2)	0.0156(2)
O(1)	0.0402(7)	0.0261(7)	0.0274(7)	0.0010(6)	0.0185(6)	-0.0014(5)
O(2)	0.0272(6)	0.0271(7)	0.0325(7)	0.0009(5)	0.0060(6)	-0.0004(6)
N(1)	0.0307(8)	0.0256(9)	0.0290(9)	0.0008(6)	0.0133(7)	-0.0009(7)
N(2)	0.0313(9)	0.0345(9)	0.0271(9)	0.0007(7)	0.0117(7)	-0.0016(7)
N(3)	0.0299(8)	0.0250(8)	0.0322(9)	0.0004(7)	0.0172(7)	-0.0014(7)
N(4)	0.0271(8)	0.0362(10)	0.0343(10)	-0.0023(7)	0.0106(7)	-0.0044(8)
C(1)	0.0242(9)	0.0299(10)	0.0245(10)	0.0047(8)	0.0109(8)	-0.0020(8)
C(2)	0.0328(10)	0.0324(11)	0.0272(10)	0.0041(8)	0.0149(8)	0.0003(8)
C(3)	0.0466(12)	0.0422(13)	0.0301(11)	0.0044(10)	0.0219(10)	0.0019(9)
C(4)	0.0517(13)	0.0484(14)	0.0296(11)	0.0051(11)	0.0221(10)	-0.0048(10)
C(5)	0.0418(12)	0.0359(12)	0.0330(12)	0.0002(9)	0.0162(10)	-0.0101(9)
C(6)	0.0256(9)	0.0310(11)	0.0295(10)	0.0043(8)	0.0116(8)	-0.0034(8)
C(7)	0.0356(10)	0.0326(11)	0.0306(11)	0.0024(9)	0.0173(9)	0.0036(9)
C(8)	0.0339(10)	0.0285(11)	0.0392(12)	-0.0022(8)	0.0186(9)	-0.0059(9)
C(9)	0.0456(13)	0.0358(12)	0.0526(15)	-0.0066(10)	0.0233(11)	-0.0111(11)
C(10)	0.0455(12)	0.0340(12)	0.0443(13)	-0.0019(10)	0.0179(11)	0.0046(10)
C(11)	0.0434(13)	0.0453(14)	0.0616(16)	-0.0089(11)	0.0330(12)	-0.0125(12)
C(12)	0.0396(11)	0.0285(11)	0.0332(11)	-0.0034(9)	0.0215(9)	-0.0018(9)
C(13)	0.0378(11)	0.0336(11)	0.0411(12)	-0.0021(9)	0.0237(10)	-0.0049(9)
C(14)	0.0434(13)	0.0449(14)	0.0579(16)	-0.0117(11)	0.0293(12)	-0.0109(12)
C(15)	0.0627(16)	0.0316(13)	0.0672(17)	-0.0118(11)	0.0393(14)	-0.0084(12)
C(16)	0.0618(16)	0.0295(12)	0.0595(16)	0.0005(11)	0.0346(14)	0.0014(11)
C(17)	0.0476(12)	0.0298(11)	0.0402(13)	0.0016(10)	0.0229(11)	0.0020(10)
C(18)	0.0339(11)	0.0428(13)	0.0540(15)	0.0006(10)	0.0202(11)	-0.0020(11)
C(19)	0.0537(17)	0.074(2)	0.069(2)	0.0038(15)	0.0058(15)	0.0024(17)
C(20)	0.073(2)	0.068(2)	0.104(2)	0.0176(16)	0.060(2)	-0.0000(18)
C(21)	0.0461(13)	0.0364(13)	0.0469(14)	0.0054(10)	0.0145(11)	0.0085(11)
C(22)	0.076(2)	0.099(2)	0.0579(19)	0.0049(19)	0.0185(16)	0.0236(19)
C(23)	0.0487(15)	0.0506(16)	0.076(2)	0.0115(12)	0.0232(14)	0.0024(14)
C(24)	0.0274(10)	0.0327(11)	0.0347(11)	0.0010(8)	0.0088(9)	0.0004(9)
C(25)	0.0351(11)	0.0475(14)	0.0436(13)	0.0046(10)	0.0159(10)	-0.0069(11)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(26)	0.0359(12)	0.0529(15)	0.0600(17)	0.0070(11)	0.0192(12)	-0.0065(13)
C(27)	0.0315(11)	0.0449(14)	0.0613(17)	0.0073(10)	0.0101(11)	0.0026(12)
C(28)	0.0315(11)	0.0471(14)	0.0414(13)	-0.0014(10)	0.0055(10)	0.0050(11)
C(29)	0.0323(11)	0.0358(12)	0.0347(12)	-0.0015(9)	0.0085(9)	0.0014(9)
C(30)	0.0552(16)	0.107(2)	0.0469(16)	0.0240(16)	0.0216(13)	-0.0149(16)
C(31)	0.0405(13)	0.0659(17)	0.0341(12)	0.0014(12)	0.0105(10)	-0.0011(12)
C(32)	0.0260(9)	0.0294(10)	0.0286(10)	0.0053(8)	0.0113(8)	-0.0010(8)
C(33)	0.0273(9)	0.0297(11)	0.0306(11)	0.0016(8)	0.0111(8)	-0.0024(8)
C(34)	0.0345(11)	0.0391(13)	0.0390(12)	-0.0020(10)	0.0059(10)	-0.0046(10)
C(35)	0.0433(13)	0.0482(15)	0.0405(14)	0.0058(11)	-0.0023(11)	0.0035(11)
C(36)	0.0467(13)	0.0341(13)	0.0442(14)	0.0061(10)	0.0084(11)	0.0069(11)
C(37)	0.0336(10)	0.0281(11)	0.0362(11)	0.0040(8)	0.0121(9)	0.0007(9)
C(38)	0.0261(9)	0.0315(11)	0.0318(11)	-0.0030(8)	0.0135(8)	-0.0055(9)
C(39)	0.0429(12)	0.0273(12)	0.0482(14)	-0.0019(9)	0.0113(11)	0.0018(10)
C(40)	0.0692(19)	0.0348(15)	0.073(2)	-0.0070(13)	0.0093(16)	0.0072(14)
C(41)	0.0728(18)	0.0413(15)	0.0595(17)	-0.0096(13)	0.0226(15)	-0.0157(13)
C(42)	0.0471(14)	0.0476(15)	0.077(2)	-0.0098(12)	0.0257(14)	0.0039(14)
C(43)	0.0280(9)	0.0252(10)	0.0349(11)	-0.0015(8)	0.0133(9)	-0.0005(8)
C(44)	0.0376(11)	0.0321(11)	0.0455(13)	-0.0010(9)	0.0236(10)	0.0000(10)
C(45)	0.0543(14)	0.0398(13)	0.0622(16)	-0.0030(11)	0.0374(13)	0.0084(12)
C(46)	0.0649(16)	0.0283(12)	0.0767(19)	-0.0009(11)	0.0433(15)	0.0075(12)
C(47)	0.0511(13)	0.0273(11)	0.0623(16)	0.0032(10)	0.0332(12)	0.0008(11)
C(48)	0.0329(10)	0.0292(11)	0.0386(12)	0.0002(8)	0.0176(9)	-0.0013(9)
C(49)	0.0539(14)	0.0445(14)	0.0625(16)	-0.0039(11)	0.0412(13)	-0.0036(12)
C(50)	0.0607(18)	0.070(2)	0.132(3)	0.0013(15)	0.062(2)	-0.018(2)
C(51)	0.137(3)	0.084(2)	0.085(2)	-0.004(2)	0.086(2)	-0.0080(19)
C(52)	0.0422(11)	0.0290(11)	0.0430(12)	-0.0025(9)	0.0260(10)	-0.0047(9)
C(53)	0.0539(15)	0.0565(17)	0.0479(15)	-0.0026(12)	0.0248(12)	-0.0006(12)
C(54)	0.0469(13)	0.0574(16)	0.0575(16)	0.0060(12)	0.0326(13)	-0.0007(13)
C(55)	0.0305(10)	0.0343(11)	0.0441(13)	-0.0051(9)	0.0195(10)	-0.0085(10)
C(56)	0.0338(11)	0.0457(14)	0.0576(16)	0.0017(10)	0.0208(11)	-0.0044(12)
C(57)	0.0412(13)	0.0546(16)	0.080(2)	0.0048(12)	0.0357(14)	-0.0061(15)
C(58)	0.0606(16)	0.0471(15)	0.086(2)	-0.0017(12)	0.0541(16)	-0.0098(14)
C(59)	0.0634(16)	0.0394(13)	0.0618(17)	-0.0092(12)	0.0436(14)	-0.0087(12)
C(60)	0.0424(12)	0.0311(12)	0.0453(13)	-0.0068(9)	0.0255(11)	-0.0052(10)
C(61)	0.0413(14)	0.101(2)	0.0593(18)	0.0233(15)	0.0137(13)	0.0030(17)
C(62)	0.0539(14)	0.0598(17)	0.0435(14)	0.0023(12)	0.0248(12)	0.0051(12)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
V(1)	Cl(1)	2.2614(6)	V(1)	Cl(2)	2.2828(7)
V(1)	O(1)	1.8124(16)	V(1)	N(1)	2.203(2)
V(1)	N(2)	1.6488(15)	V(2)	Cl(3)	2.2759(6)
V(2)	Cl(4)	2.2522(7)	V(2)	O(2)	1.8078(13)
V(2)	N(3)	2.2089(15)	V(2)	N(4)	1.643(2)
O(1)	C(1)	1.344(2)	O(2)	C(32)	1.344(2)
N(1)	C(7)	1.300(3)	N(1)	C(12)	1.457(2)
N(2)	C(24)	1.389(2)	N(3)	C(38)	1.298(2)
N(3)	C(43)	1.462(2)	N(4)	C(55)	1.384(3)
C(1)	C(2)	1.407(3)	C(1)	C(6)	1.415(2)
C(2)	C(3)	1.410(3)	C(2)	C(7)	1.439(2)
C(3)	C(4)	1.374(3)	C(4)	C(5)	1.399(3)
C(5)	C(6)	1.390(3)	C(6)	C(8)	1.528(3)
C(8)	C(9)	1.540(3)	C(8)	C(10)	1.538(2)
C(8)	C(11)	1.534(4)	C(12)	C(13)	1.404(2)
C(12)	C(17)	1.400(2)	C(13)	C(14)	1.392(3)
C(13)	C(18)	1.517(3)	C(14)	C(15)	1.382(3)
C(15)	C(16)	1.375(3)	C(16)	C(17)	1.396(3)
C(17)	C(21)	1.514(3)	C(18)	C(19)	1.517(3)
C(18)	C(20)	1.526(5)	C(21)	C(22)	1.525(4)
C(21)	C(23)	1.532(5)	C(24)	C(25)	1.408(4)
C(24)	C(29)	1.414(3)	C(25)	C(26)	1.400(3)
C(25)	C(30)	1.500(3)	C(26)	C(27)	1.368(3)
C(27)	C(28)	1.386(4)	C(28)	C(29)	1.385(3)
C(29)	C(31)	1.506(4)	C(32)	C(33)	1.398(2)
C(32)	C(37)	1.413(2)	C(33)	C(34)	1.403(2)
C(33)	C(38)	1.441(2)	C(34)	C(35)	1.375(3)
C(35)	C(36)	1.391(3)	C(36)	C(37)	1.397(2)
C(37)	C(39)	1.536(2)	C(39)	C(40)	1.539(3)
C(39)	C(41)	1.529(4)	C(39)	C(42)	1.533(4)
C(43)	C(44)	1.402(4)	C(43)	C(48)	1.406(3)
C(44)	C(45)	1.400(3)	C(44)	C(49)	1.518(4)
C(45)	C(46)	1.378(4)	C(46)	C(47)	1.379(5)
C(47)	C(48)	1.391(3)	C(48)	C(52)	1.517(4)
C(49)	C(50)	1.540(3)	C(49)	C(51)	1.524(5)
C(52)	C(53)	1.528(3)	C(52)	C(54)	1.528(3)
C(55)	C(56)	1.419(2)	C(55)	C(60)	1.411(3)

Table 4. Bond lengths (Å) (continued)

atom	atom	distance	atom	atom	distance
C(56)	C(57)	1.394(5)	C(56)	C(61)	1.497(4)
C(57)	C(58)	1.367(4)	C(58)	C(59)	1.386(3)
C(59)	C(60)	1.390(4)	C(60)	C(62)	1.497(3)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(9)	H(5)	0.950	C(9)	H(6)	0.950
C(9)	H(7)	0.950	C(10)	H(8)	0.950
C(10)	H(9)	0.950	C(10)	H(10)	0.950
C(11)	H(11)	0.950	C(11)	H(12)	0.950
C(11)	H(13)	0.950	C(19)	H(18)	0.950
C(19)	H(19)	0.950	C(19)	H(20)	0.950
C(20)	H(21)	0.950	C(20)	H(22)	0.950
C(20)	H(23)	0.950	C(22)	H(25)	0.950
C(22)	H(26)	0.950	C(22)	H(27)	0.950
C(23)	H(28)	0.950	C(23)	H(29)	0.950
C(23)	H(30)	0.950	C(30)	H(34)	0.950
C(30)	H(35)	0.950	C(30)	H(36)	0.950
C(31)	H(37)	0.950	C(31)	H(38)	0.950
C(31)	H(39)	0.950	C(40)	H(44)	0.950
C(40)	H(45)	0.950	C(40)	H(46)	0.950
C(41)	H(47)	0.950	C(41)	H(48)	0.950
C(41)	H(49)	0.950	C(42)	H(50)	0.950
C(42)	H(51)	0.950	C(42)	H(52)	0.950
C(50)	H(57)	0.950	C(50)	H(58)	0.950
C(50)	H(59)	0.950	C(51)	H(60)	0.950
C(51)	H(61)	0.950	C(51)	H(62)	0.950
C(53)	H(64)	0.950	C(53)	H(65)	0.950
C(53)	H(66)	0.950	C(54)	H(67)	0.950
C(54)	H(68)	0.950	C(54)	H(69)	0.950
C(61)	H(73)	0.950	C(61)	H(74)	0.950
C(61)	H(75)	0.950	C(62)	H(76)	0.950
C(62)	H(77)	0.950	C(62)	H(78)	0.950

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	V(1)	Cl(2)	90.49(2)	Cl(1)	V(1)	O(1)	142.43(4)
Cl(1)	V(1)	N(1)	84.27(4)	Cl(1)	V(1)	N(2)	108.20(6)
Cl(2)	V(1)	O(1)	92.20(5)	Cl(2)	V(1)	N(1)	162.08(4)
Cl(2)	V(1)	N(2)	101.61(7)	O(1)	V(1)	N(1)	81.81(7)
O(1)	V(1)	N(2)	107.90(7)	N(1)	V(1)	N(2)	96.31(8)
Cl(3)	V(2)	Cl(4)	91.81(2)	Cl(3)	V(2)	O(2)	91.54(4)
Cl(3)	V(2)	N(3)	162.70(6)	Cl(3)	V(2)	N(4)	99.41(5)
Cl(4)	V(2)	O(2)	143.29(6)	Cl(4)	V(2)	N(3)	84.37(4)
Cl(4)	V(2)	N(4)	107.93(6)	O(2)	V(2)	N(3)	81.76(5)
O(2)	V(2)	N(4)	107.53(8)	N(3)	V(2)	N(4)	97.82(8)
V(1)	O(1)	C(1)	137.01(12)	V(2)	O(2)	C(32)	141.09(13)
V(1)	N(1)	C(7)	121.69(14)	V(1)	N(1)	C(12)	124.45(16)
C(7)	N(1)	C(12)	113.9(2)	V(1)	N(2)	C(24)	175.63(19)
V(2)	N(3)	C(38)	123.05(13)	V(2)	N(3)	C(43)	123.56(10)
C(38)	N(3)	C(43)	113.38(15)	V(2)	N(4)	C(55)	173.39(15)
O(1)	C(1)	C(2)	116.65(17)	O(1)	C(1)	C(6)	121.4(2)
C(2)	C(1)	C(6)	121.9(2)	C(1)	C(2)	C(3)	119.75(19)
C(1)	C(2)	C(7)	121.5(2)	C(3)	C(2)	C(7)	118.7(2)
C(2)	C(3)	C(4)	119.0(2)	C(3)	C(4)	C(5)	120.3(2)
C(4)	C(5)	C(6)	123.3(2)	C(1)	C(6)	C(5)	115.8(2)
C(1)	C(6)	C(8)	121.8(2)	C(5)	C(6)	C(8)	122.46(18)
N(1)	C(7)	C(2)	128.1(2)	C(6)	C(8)	C(9)	111.8(2)
C(6)	C(8)	C(10)	110.56(18)	C(6)	C(8)	C(11)	109.39(18)
C(9)	C(8)	C(10)	108.28(17)	C(9)	C(8)	C(11)	106.48(19)
C(10)	C(8)	C(11)	110.3(2)	N(1)	C(12)	C(13)	118.18(17)
N(1)	C(12)	C(17)	119.47(17)	C(13)	C(12)	C(17)	122.34(18)
C(12)	C(13)	C(14)	117.55(19)	C(12)	C(13)	C(18)	121.67(18)
C(14)	C(13)	C(18)	120.77(19)	C(13)	C(14)	C(15)	121.1(2)
C(14)	C(15)	C(16)	120.1(2)	C(15)	C(16)	C(17)	121.5(2)
C(12)	C(17)	C(16)	117.3(2)	C(12)	C(17)	C(21)	122.67(19)
C(16)	C(17)	C(21)	120.0(2)	C(13)	C(18)	C(19)	112.8(2)
C(13)	C(18)	C(20)	109.5(2)	C(19)	C(18)	C(20)	110.9(2)
C(17)	C(21)	C(22)	112.2(2)	C(17)	C(21)	C(23)	110.8(2)
C(22)	C(21)	C(23)	111.8(2)	N(2)	C(24)	C(25)	118.59(19)
N(2)	C(24)	C(29)	119.7(2)	C(25)	C(24)	C(29)	121.74(19)
C(24)	C(25)	C(26)	117.7(2)	C(24)	C(25)	C(30)	121.3(2)
C(26)	C(25)	C(30)	121.0(2)	C(25)	C(26)	C(27)	120.8(3)

Table 6. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(26)	C(27)	C(28)	121.0(2)	C(27)	C(28)	C(29)	121.1(2)
C(24)	C(29)	C(28)	117.6(2)	C(24)	C(29)	C(31)	121.72(19)
C(28)	C(29)	C(31)	120.7(2)	O(2)	C(32)	C(33)	116.91(17)
O(2)	C(32)	C(37)	120.63(15)	C(33)	C(32)	C(37)	122.41(15)
C(32)	C(33)	C(34)	119.42(18)	C(32)	C(33)	C(38)	121.80(15)
C(34)	C(33)	C(38)	118.78(17)	C(33)	C(34)	C(35)	119.39(19)
C(34)	C(35)	C(36)	120.19(19)	C(35)	C(36)	C(37)	123.1(2)
C(32)	C(37)	C(36)	115.38(18)	C(32)	C(37)	C(39)	122.35(15)
C(36)	C(37)	C(39)	122.3(2)	N(3)	C(38)	C(33)	127.98(17)
C(37)	C(39)	C(40)	111.15(17)	C(37)	C(39)	C(41)	110.7(2)
C(37)	C(39)	C(42)	110.3(2)	C(40)	C(39)	C(41)	107.6(2)
C(40)	C(39)	C(42)	107.3(2)	C(41)	C(39)	C(42)	109.8(2)
N(3)	C(43)	C(44)	119.3(2)	N(3)	C(43)	C(48)	118.5(2)
C(44)	C(43)	C(48)	122.23(19)	C(43)	C(44)	C(45)	117.3(2)
C(43)	C(44)	C(49)	122.5(2)	C(45)	C(44)	C(49)	120.2(2)
C(44)	C(45)	C(46)	121.2(3)	C(45)	C(46)	C(47)	120.4(2)
C(46)	C(47)	C(48)	121.0(2)	C(43)	C(48)	C(47)	117.8(2)
C(43)	C(48)	C(52)	121.73(19)	C(47)	C(48)	C(52)	120.5(2)
C(44)	C(49)	C(50)	110.4(2)	C(44)	C(49)	C(51)	112.6(2)
C(50)	C(49)	C(51)	110.8(3)	C(48)	C(52)	C(53)	111.15(19)
C(48)	C(52)	C(54)	112.38(19)	C(53)	C(52)	C(54)	110.9(2)
N(4)	C(55)	C(56)	118.3(2)	N(4)	C(55)	C(60)	119.68(18)
C(56)	C(55)	C(60)	122.0(2)	C(55)	C(56)	C(57)	116.9(2)
C(55)	C(56)	C(61)	121.4(2)	C(57)	C(56)	C(61)	121.7(2)
C(56)	C(57)	C(58)	121.4(2)	C(57)	C(58)	C(59)	121.4(3)
C(58)	C(59)	C(60)	120.2(2)	C(55)	C(60)	C(59)	118.0(2)
C(55)	C(60)	C(62)	121.8(2)	C(59)	C(60)	C(62)	120.2(2)

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
C(2)	C(3)	H(1)	121.8	C(4)	C(3)	H(1)	119.1
C(3)	C(4)	H(2)	118.9	C(5)	C(4)	H(2)	120.2
C(4)	C(5)	H(3)	115.7	C(6)	C(5)	H(3)	121.0
N(1)	C(7)	H(4)	117.0	C(2)	C(7)	H(4)	115.0
C(8)	C(9)	H(5)	109.3	C(8)	C(9)	H(6)	109.5
C(8)	C(9)	H(7)	109.6	H(5)	C(9)	H(6)	109.5
H(5)	C(9)	H(7)	109.5	H(6)	C(9)	H(7)	109.5
C(8)	C(10)	H(8)	109.2	C(8)	C(10)	H(9)	109.8
C(8)	C(10)	H(10)	109.4	H(8)	C(10)	H(9)	109.5
H(8)	C(10)	H(10)	109.5	H(9)	C(10)	H(10)	109.5
C(8)	C(11)	H(11)	109.2	C(8)	C(11)	H(12)	109.6
C(8)	C(11)	H(13)	109.6	H(11)	C(11)	H(12)	109.5
H(11)	C(11)	H(13)	109.5	H(12)	C(11)	H(13)	109.5
C(13)	C(14)	H(14)	115.5	C(15)	C(14)	H(14)	123.4
C(14)	C(15)	H(15)	122.9	C(16)	C(15)	H(15)	116.9
C(15)	C(16)	H(16)	119.6	C(17)	C(16)	H(16)	118.9
C(13)	C(18)	H(17)	111.1	C(19)	C(18)	H(17)	103.1
C(20)	C(18)	H(17)	109.2	C(18)	C(19)	H(18)	109.7
C(18)	C(19)	H(19)	109.4	C(18)	C(19)	H(20)	109.4
H(18)	C(19)	H(19)	109.5	H(18)	C(19)	H(20)	109.5
H(19)	C(19)	H(20)	109.5	C(18)	C(20)	H(21)	109.9
C(18)	C(20)	H(22)	109.8	C(18)	C(20)	H(23)	108.7
H(21)	C(20)	H(22)	109.5	H(21)	C(20)	H(23)	109.5
H(22)	C(20)	H(23)	109.5	C(17)	C(21)	H(24)	107.0
C(22)	C(21)	H(24)	109.0	C(23)	C(21)	H(24)	105.7
C(21)	C(22)	H(25)	110.6	C(21)	C(22)	H(26)	108.8
C(21)	C(22)	H(27)	109.0	H(25)	C(22)	H(26)	109.5
H(25)	C(22)	H(27)	109.5	H(26)	C(22)	H(27)	109.5
C(21)	C(23)	H(28)	109.8	C(21)	C(23)	H(29)	109.8
C(21)	C(23)	H(30)	108.8	H(28)	C(23)	H(29)	109.5
H(28)	C(23)	H(30)	109.5	H(29)	C(23)	H(30)	109.5
C(25)	C(26)	H(31)	118.8	C(27)	C(26)	H(31)	120.4
C(26)	C(27)	H(32)	123.5	C(28)	C(27)	H(32)	115.5
C(27)	C(28)	H(33)	118.5	C(29)	C(28)	H(33)	120.0
C(25)	C(30)	H(34)	108.7	C(25)	C(30)	H(35)	109.6
C(25)	C(30)	H(36)	110.1	H(34)	C(30)	H(35)	109.5
H(34)	C(30)	H(36)	109.5	H(35)	C(30)	H(36)	109.5

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(29)	C(31)	H(37)	109.6	C(29)	C(31)	H(38)	109.4
C(29)	C(31)	H(39)	109.5	H(37)	C(31)	H(38)	109.5
H(37)	C(31)	H(39)	109.5	H(38)	C(31)	H(39)	109.5
C(33)	C(34)	H(40)	118.7	C(35)	C(34)	H(40)	121.9
C(34)	C(35)	H(41)	116.9	C(36)	C(35)	H(41)	122.9
C(35)	C(36)	H(42)	120.9	C(37)	C(36)	H(42)	115.9
N(3)	C(38)	H(43)	117.5	C(33)	C(38)	H(43)	114.5
C(39)	C(40)	H(44)	109.5	C(39)	C(40)	H(45)	109.1
C(39)	C(40)	H(46)	109.8	H(44)	C(40)	H(45)	109.5
H(44)	C(40)	H(46)	109.5	H(45)	C(40)	H(46)	109.5
C(39)	C(41)	H(47)	110.0	C(39)	C(41)	H(48)	109.1
C(39)	C(41)	H(49)	109.3	H(47)	C(41)	H(48)	109.5
H(47)	C(41)	H(49)	109.5	H(48)	C(41)	H(49)	109.5
C(39)	C(42)	H(50)	109.2	C(39)	C(42)	H(51)	109.3
C(39)	C(42)	H(52)	109.9	H(50)	C(42)	H(51)	109.5
H(50)	C(42)	H(52)	109.5	H(51)	C(42)	H(52)	109.5
C(44)	C(45)	H(53)	117.5	C(46)	C(45)	H(53)	121.2
C(45)	C(46)	H(54)	119.4	C(47)	C(46)	H(54)	120.2
C(46)	C(47)	H(55)	119.9	C(48)	C(47)	H(55)	119.0
C(44)	C(49)	H(56)	106.5	C(50)	C(49)	H(56)	108.7
C(51)	C(49)	H(56)	107.7	C(49)	C(50)	H(57)	109.8
C(49)	C(50)	H(58)	109.4	C(49)	C(50)	H(59)	109.3
H(57)	C(50)	H(58)	109.5	H(57)	C(50)	H(59)	109.5
H(58)	C(50)	H(59)	109.5	C(49)	C(51)	H(60)	109.9
C(49)	C(51)	H(61)	108.7	C(49)	C(51)	H(62)	109.8
H(60)	C(51)	H(61)	109.5	H(60)	C(51)	H(62)	109.5
H(61)	C(51)	H(62)	109.5	C(48)	C(52)	H(63)	108.4
C(53)	C(52)	H(63)	106.0	C(54)	C(52)	H(63)	107.8
C(52)	C(53)	H(64)	110.0	C(52)	C(53)	H(65)	109.1
C(52)	C(53)	H(66)	109.3	H(64)	C(53)	H(65)	109.5
H(64)	C(53)	H(66)	109.5	H(65)	C(53)	H(66)	109.5
C(52)	C(54)	H(67)	109.4	C(52)	C(54)	H(68)	109.1
C(52)	C(54)	H(69)	109.9	H(67)	C(54)	H(68)	109.5
H(67)	C(54)	H(69)	109.5	H(68)	C(54)	H(69)	109.5
C(56)	C(57)	H(70)	117.8	C(58)	C(57)	H(70)	120.8
C(57)	C(58)	H(71)	126.2	C(59)	C(58)	H(71)	111.7
C(58)	C(59)	H(72)	122.9	C(60)	C(59)	H(72)	116.9

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(56)	C(61)	H(73)	109.2	C(56)	C(61)	H(74)	109.3
C(56)	C(61)	H(75)	109.9	H(73)	C(61)	H(74)	109.5
H(73)	C(61)	H(75)	109.5	H(74)	C(61)	H(75)	109.5
C(60)	C(62)	H(76)	109.5	C(60)	C(62)	H(77)	109.2
C(60)	C(62)	H(78)	109.8	H(76)	C(62)	H(77)	109.5
H(76)	C(62)	H(78)	109.5	H(77)	C(62)	H(78)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Cl(1)	V(1)	O(1)	C(1)	-114.40(17)	Cl(1)	V(1)	N(1)	C(7)	165.82(14)
Cl(1)	V(1)	N(1)	C(12)	-14.17(13)	Cl(1)	V(1)	N(2)	C(24)	-37(2)
Cl(2)	V(1)	O(1)	C(1)	151.92(19)	Cl(2)	V(1)	N(1)	C(7)	92.22(19)
Cl(2)	V(1)	N(1)	C(12)	-87.8(2)	Cl(2)	V(1)	N(2)	C(24)	57(2)
O(1)	V(1)	N(1)	C(7)	20.81(14)	O(1)	V(1)	N(1)	C(12)	-159.18(14)
N(1)	V(1)	O(1)	C(1)	-45.05(19)	O(1)	V(1)	N(2)	C(24)	153(2)
N(2)	V(1)	O(1)	C(1)	49.0(2)	N(1)	V(1)	N(2)	C(24)	-124(2)
N(2)	V(1)	N(1)	C(7)	-86.44(15)	N(2)	V(1)	N(1)	C(12)	93.57(14)
Cl(3)	V(2)	O(2)	C(32)	161.1(2)	Cl(3)	V(2)	N(3)	C(38)	81.8(2)
Cl(3)	V(2)	N(3)	C(43)	-99.7(2)	Cl(3)	V(2)	N(4)	C(55)	-17.9(12)
Cl(4)	V(2)	O(2)	C(32)	-103.8(2)	Cl(4)	V(2)	N(3)	C(38)	159.6(2)
Cl(4)	V(2)	N(3)	C(43)	-21.82(19)	Cl(4)	V(2)	N(4)	C(55)	-112.9(12)
O(2)	V(2)	N(3)	C(38)	13.7(2)	O(2)	V(2)	N(3)	C(43)	-167.7(2)
N(3)	V(2)	O(2)	C(32)	-34.9(2)	O(2)	V(2)	N(4)	C(55)	76.8(12)
N(4)	V(2)	O(2)	C(32)	60.7(2)	N(3)	V(2)	N(4)	C(55)	160.5(12)
N(4)	V(2)	N(3)	C(38)	-93.0(2)	N(4)	V(2)	N(3)	C(43)	85.6(2)
V(1)	O(1)	C(1)	C(2)	43.4(2)	V(1)	O(1)	C(1)	C(6)	-138.62(17)
V(2)	O(2)	C(32)	C(33)	35.9(3)	V(2)	O(2)	C(32)	C(37)	-146.6(2)
V(1)	N(1)	C(7)	C(2)	-3.1(2)	V(1)	N(1)	C(12)	C(13)	93.6(2)
V(1)	N(1)	C(12)	C(17)	-87.6(2)	C(7)	N(1)	C(12)	C(13)	-86.3(2)
C(7)	N(1)	C(12)	C(17)	92.4(2)	C(12)	N(1)	C(7)	C(2)	176.88(18)
V(1)	N(2)	C(24)	C(25)	-7(2)	V(1)	N(2)	C(24)	C(29)	172(2)
V(2)	N(3)	C(38)	C(33)	-1.0(4)	V(2)	N(3)	C(43)	C(44)	96.0(2)
V(2)	N(3)	C(43)	C(48)	-85.1(2)	C(38)	N(3)	C(43)	C(44)	-85.4(2)
C(38)	N(3)	C(43)	C(48)	93.6(2)	C(43)	N(3)	C(38)	C(33)	-179.7(2)
V(2)	N(4)	C(55)	C(56)	75.8(13)	V(2)	N(4)	C(55)	C(60)	-104.0(12)
O(1)	C(1)	C(2)	C(3)	174.66(18)	O(1)	C(1)	C(2)	C(7)	-4.1(2)
O(1)	C(1)	C(6)	C(5)	-176.28(17)	O(1)	C(1)	C(6)	C(8)	5.2(2)
C(2)	C(1)	C(6)	C(5)	1.6(2)	C(2)	C(1)	C(6)	C(8)	-177.00(18)
C(6)	C(1)	C(2)	C(3)	-3.3(2)	C(6)	C(1)	C(2)	C(7)	177.92(18)
C(1)	C(2)	C(3)	C(4)	2.5(3)	C(1)	C(2)	C(7)	N(1)	-10.2(3)
C(3)	C(2)	C(7)	N(1)	171.01(19)	C(7)	C(2)	C(3)	C(4)	-178.7(2)
C(2)	C(3)	C(4)	C(5)	-0.1(2)	C(3)	C(4)	C(5)	C(6)	-1.6(3)
C(4)	C(5)	C(6)	C(1)	0.9(3)	C(4)	C(5)	C(6)	C(8)	179.4(2)
C(1)	C(6)	C(8)	C(9)	177.25(18)	C(1)	C(6)	C(8)	C(10)	-62.1(2)
C(1)	C(6)	C(8)	C(11)	59.6(2)	C(5)	C(6)	C(8)	C(9)	-1.2(2)
C(5)	C(6)	C(8)	C(10)	119.5(2)	C(5)	C(6)	C(8)	C(11)	-118.9(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
N(1)	C(12)	C(13)	C(14)	177.3(2)
N(1)	C(12)	C(17)	C(16)	-177.2(2)
C(13)	C(12)	C(17)	C(16)	1.5(4)
C(17)	C(12)	C(13)	C(14)	-1.4(4)
C(12)	C(13)	C(14)	C(15)	0.2(3)
C(12)	C(13)	C(18)	C(20)	100.1(3)
C(14)	C(13)	C(18)	C(20)	-78.8(3)
C(13)	C(14)	C(15)	C(16)	0.9(5)
C(15)	C(16)	C(17)	C(12)	-0.4(4)
C(12)	C(17)	C(21)	C(22)	-114.6(3)
C(16)	C(17)	C(21)	C(22)	65.6(3)
N(2)	C(24)	C(25)	C(26)	176.2(2)
N(2)	C(24)	C(29)	C(28)	-174.4(2)
C(25)	C(24)	C(29)	C(28)	4.8(3)
C(29)	C(24)	C(25)	C(26)	-2.9(3)
C(24)	C(25)	C(26)	C(27)	-0.3(3)
C(25)	C(26)	C(27)	C(28)	1.4(4)
C(27)	C(28)	C(29)	C(24)	-3.5(3)
O(2)	C(32)	C(33)	C(34)	173.9(2)
O(2)	C(32)	C(37)	C(36)	-174.6(2)
C(33)	C(32)	C(37)	C(36)	2.7(4)
C(37)	C(32)	C(33)	C(34)	-3.5(4)
C(32)	C(33)	C(34)	C(35)	1.6(4)
C(34)	C(33)	C(38)	N(3)	174.1(2)
C(33)	C(34)	C(35)	C(36)	0.9(4)
C(35)	C(36)	C(37)	C(32)	-0.1(3)
C(32)	C(37)	C(39)	C(40)	178.8(2)
C(32)	C(37)	C(39)	C(42)	60.0(3)
C(36)	C(37)	C(39)	C(41)	118.4(2)
N(3)	C(43)	C(44)	C(45)	177.50(16)
N(3)	C(43)	C(48)	C(47)	-178.04(15)
C(44)	C(43)	C(48)	C(47)	0.9(2)
C(48)	C(43)	C(44)	C(45)	-1.4(2)
C(43)	C(44)	C(45)	C(46)	0.6(2)
C(43)	C(44)	C(49)	C(51)	-133.8(2)
C(45)	C(44)	C(49)	C(51)	47.0(3)
C(44)	C(45)	C(46)	C(47)	0.8(3)

atom1	atom2	atom3	atom4	angle
N(1)	C(12)	C(13)	C(18)	-1.6(4)
N(1)	C(12)	C(17)	C(21)	3.0(4)
C(13)	C(12)	C(17)	C(21)	-178.3(2)
C(17)	C(12)	C(13)	C(18)	179.6(2)
C(12)	C(13)	C(18)	C(19)	-135.9(2)
C(14)	C(13)	C(18)	C(19)	45.2(4)
C(18)	C(13)	C(14)	C(15)	179.1(3)
C(14)	C(15)	C(16)	C(17)	-0.8(5)
C(15)	C(16)	C(17)	C(21)	179.5(3)
C(12)	C(17)	C(21)	C(23)	119.6(2)
C(16)	C(17)	C(21)	C(23)	-60.2(3)
N(2)	C(24)	C(25)	C(30)	-4.0(3)
N(2)	C(24)	C(29)	C(31)	6.2(3)
C(25)	C(24)	C(29)	C(31)	-174.7(2)
C(29)	C(24)	C(25)	C(30)	176.9(2)
C(30)	C(25)	C(26)	C(27)	180.0(2)
C(26)	C(27)	C(28)	C(29)	0.5(4)
C(27)	C(28)	C(29)	C(31)	175.9(2)
O(2)	C(32)	C(33)	C(38)	-5.7(3)
O(2)	C(32)	C(37)	C(39)	5.5(4)
C(33)	C(32)	C(37)	C(39)	-177.2(2)
C(37)	C(32)	C(33)	C(38)	176.9(2)
C(32)	C(33)	C(38)	N(3)	-6.4(4)
C(38)	C(33)	C(34)	C(35)	-178.8(2)
C(34)	C(35)	C(36)	C(37)	-1.7(5)
C(35)	C(36)	C(37)	C(39)	179.8(3)
C(32)	C(37)	C(39)	C(41)	-61.7(3)
C(36)	C(37)	C(39)	C(40)	-1.1(4)
C(36)	C(37)	C(39)	C(42)	-119.9(2)
N(3)	C(43)	C(44)	C(49)	-1.8(2)
N(3)	C(43)	C(48)	C(52)	0.3(2)
C(44)	C(43)	C(48)	C(52)	179.25(16)
C(48)	C(43)	C(44)	C(49)	179.29(16)
C(43)	C(44)	C(49)	C(50)	101.8(2)
C(45)	C(44)	C(49)	C(50)	-77.4(2)
C(49)	C(44)	C(45)	C(46)	179.87(19)
C(45)	C(46)	C(47)	C(48)	-1.3(3)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(46)	C(47)	C(48)	C(43)	0.5(2)
C(43)	C(48)	C(52)	C(53)	-103.3(2)
C(47)	C(48)	C(52)	C(53)	75.0(2)
N(4)	C(55)	C(56)	C(57)	-179.5(2)
N(4)	C(55)	C(60)	C(59)	-179.3(2)
C(56)	C(55)	C(60)	C(59)	0.9(3)
C(60)	C(55)	C(56)	C(57)	0.3(3)
C(55)	C(56)	C(57)	C(58)	-0.8(3)
C(56)	C(57)	C(58)	C(59)	0.2(3)
C(58)	C(59)	C(60)	C(55)	-1.5(3)

atom1	atom2	atom3	atom4	angle
C(46)	C(47)	C(48)	C(52)	-177.86(18)
C(43)	C(48)	C(52)	C(54)	131.81(19)
C(47)	C(48)	C(52)	C(54)	-49.9(2)
N(4)	C(55)	C(56)	C(61)	0.6(3)
N(4)	C(55)	C(60)	C(62)	0.6(3)
C(56)	C(55)	C(60)	C(62)	-179.2(2)
C(60)	C(55)	C(56)	C(61)	-179.6(2)
C(61)	C(56)	C(57)	C(58)	179.1(2)
C(57)	C(58)	C(59)	C(60)	1.1(3)
C(58)	C(59)	C(60)	C(62)	178.6(2)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
O(2)	C(20)	3.454(4)	C(4)	C(30)[1)]	3.526(3)
C(20)	O(2)	3.454(4)	C(20)	C(32)	3.326(5)
C(20)	C(33)	3.427(5)	C(24)	C(58)[2)]	3.478(3)
C(27)	C(59)[2)]	3.444(3)	C(28)	C(59)[2)]	3.488(4)
C(29)	C(58)[2)]	3.457(4)	C(30)	C(4)[3)]	3.526(3)
C(32)	C(20)	3.326(5)	C(33)	C(20)	3.427(5)
C(58)	C(24)[4)]	3.478(3)	C(58)	C(29)[4)]	3.457(4)
C(59)	C(27)[4)]	3.444(3)	C(59)	C(28)[4)]	3.488(4)

Symmetry Operators:

(1) X,-Y+1/2,Z+1/2-1 (2) X-1,Y,Z
(3) X,-Y+1/2,Z+1/2 (4) X+1,Y,Z

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
V(1)	H(17)	3.313	V(1)	H(24)	3.276
V(2)	H(56)	3.367	V(2)	H(63)	3.177
Cl(1)	H(1)[1)]	3.336	Cl(1)	H(2)[1)]	2.790
Cl(1)	H(6)[2)]	3.339	Cl(1)	H(9)[2)]	3.136
Cl(1)	H(17)	3.231	Cl(1)	H(40)[1)]	2.938
Cl(2)	H(1)[1)]	3.006	Cl(2)	H(15)[3)]	3.112
Cl(2)	H(16)[3)]	3.049	Cl(2)	H(41)[1)]	3.151
Cl(2)	H(70)[4)]	3.497	Cl(3)	H(33)[5)]	2.895
Cl(3)	H(37)[5)]	3.189	Cl(3)	H(54)[6)]	3.127
Cl(3)	H(55)[6)]	3.081	Cl(3)	H(72)[1)]	2.864
Cl(4)	H(32)[5)]	3.192	Cl(4)	H(33)[5)]	2.816
Cl(4)	H(44)[7)]	2.961	Cl(4)	H(47)[7)]	3.523
Cl(4)	H(56)	3.121	O(1)	H(17)	3.397
O(1)	H(57)	3.125	O(2)	H(21)	3.078
O(2)	H(23)	3.084	N(1)	H(4)	1.998
N(1)	H(17)	2.473	N(1)	H(24)	2.412
N(2)	H(24)	2.582	N(3)	H(21)	3.448
N(3)	H(43)	1.978	N(3)	H(56)	2.416
N(3)	H(63)	2.408	N(4)	H(63)	2.449
C(1)	H(1)	3.359	C(1)	H(3)	3.296
C(1)	H(4)	3.372	C(1)	H(57)	3.316
C(1)	H(58)	3.488	C(2)	H(1)	2.121
C(2)	H(2)	3.303	C(2)	H(4)	2.100
C(2)	H(57)	3.593	C(2)	H(58)	3.445
C(3)	H(1)	1.007	C(3)	H(2)	2.058
C(3)	H(3)	3.272	C(3)	H(4)	2.579
C(4)	H(1)	2.062	C(4)	H(2)	1.006
C(4)	H(3)	2.038	C(4)	H(5)[8)]	2.898
C(4)	H(10)[8)]	3.390	C(4)	H(34)[9)]	3.427
C(4)	H(35)[9)]	2.974	C(5)	H(1)	3.311
C(5)	H(2)	2.094	C(5)	H(3)	0.995
C(5)	H(5)[8)]	2.980	C(5)	H(10)[8)]	3.470
C(6)	H(2)	3.348	C(6)	H(3)	2.085
C(7)	H(1)	2.664	C(7)	H(4)	1.038
C(7)	H(17)	2.967	C(7)	H(24)	2.944
C(8)	H(3)	2.759	C(9)	H(2)[8)]	3.417
C(9)	H(3)	2.485	C(9)	H(43)[8)]	3.067

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(9)	H(66)[8)]	3.533	C(10)	H(2)[8)]	3.401
C(10)	H(29)[3)]	3.214	C(10)	H(59)	3.225
C(11)	H(65)[8)]	3.297	C(11)	H(66)[8)]	3.590
C(11)	H(70)[4)]	3.011	C(11)	H(71)[4)]	3.008
C(12)	H(4)	2.396	C(12)	H(14)	3.268
C(12)	H(16)	3.301	C(12)	H(17)	2.673
C(12)	H(24)	2.577	C(13)	H(4)	3.040
C(13)	H(14)	2.028	C(13)	H(15)	3.333
C(13)	H(17)	2.142	C(14)	H(13)[2)]	3.352
C(14)	H(14)	0.993	C(14)	H(15)	2.098
C(14)	H(16)	3.299	C(14)	H(17)	3.385
C(15)	H(9)[2)]	3.324	C(15)	H(13)[2)]	3.141
C(15)	H(14)	2.099	C(15)	H(15)	0.998
C(15)	H(16)	2.068	C(15)	H(27)[10)]	3.372
C(15)	H(70)[6)]	3.420	C(15)	H(75)[6)]	3.558
C(16)	H(8)[2)]	3.599	C(16)	H(9)[2)]	3.115
C(16)	H(14)	3.307	C(16)	H(15)	2.032
C(16)	H(16)	1.008	C(16)	H(24)	3.326
C(16)	H(27)[10)]	3.383	C(17)	H(4)	3.106
C(17)	H(9)[2)]	3.369	C(17)	H(15)	3.295
C(17)	H(16)	2.079	C(17)	H(24)	2.058
C(18)	H(4)	3.415	C(18)	H(14)	2.627
C(18)	H(17)	1.061	C(18)	H(57)	3.376
C(19)	H(14)	2.682	C(19)	H(17)	2.038
C(19)	H(40)[1)]	3.590	C(19)	H(64)[1)]	3.518
C(19)	H(66)[1)]	3.328	C(19)	H(76)[1)]	3.312
C(19)	H(78)[1)]	3.265	C(20)	H(4)	3.314
C(20)	H(14)	3.131	C(20)	H(17)	2.126
C(20)	H(56)	3.128	C(20)	H(57)	3.367
C(20)	H(58)	3.549	C(21)	H(4)	3.571
C(21)	H(16)	2.674	C(21)	H(24)	1.020
C(22)	H(16)	2.991	C(22)	H(24)	2.092
C(22)	H(46)[10)]	3.330	C(23)	H(9)[2)]	3.483
C(23)	H(16)	2.906	C(23)	H(24)	2.058
C(23)	H(42)[10)]	2.968	C(23)	H(46)[10)]	3.467
C(24)	H(24)	2.762	C(24)	H(31)	3.299
C(24)	H(33)	3.298	C(24)	H(71)[4)]	3.434

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(25)	H(31)	2.069	C(25)	H(32)	3.381
C(26)	H(31)	0.991	C(26)	H(32)	2.136
C(26)	H(33)	3.275	C(26)	H(53)[2)]	3.475
C(27)	H(31)	2.056	C(27)	H(32)	1.052
C(27)	H(33)	2.052	C(27)	H(44)[10)]	3.225
C(27)	H(45)[10)]	3.440	C(27)	H(72)[4)]	3.441
C(28)	H(31)	3.293	C(28)	H(32)	2.070
C(28)	H(33)	0.991	C(28)	H(44)[10)]	3.346
C(28)	H(46)[10)]	3.546	C(28)	H(71)[4)]	3.594
C(28)	H(72)[4)]	3.250	C(29)	H(24)	2.870
C(29)	H(32)	3.329	C(29)	H(33)	2.067
C(29)	H(71)[4)]	3.179	C(30)	H(2)[1)]	3.107
C(30)	H(31)	2.683	C(30)	H(53)[2)]	3.222
C(30)	H(54)[2)]	3.384	C(31)	H(24)	2.971
C(31)	H(33)	2.686	C(31)	H(71)[4)]	3.420
C(31)	H(73)[11)]	3.352	C(32)	H(21)	3.202
C(32)	H(22)	3.224	C(32)	H(23)	3.000
C(32)	H(40)	3.343	C(32)	H(42)	3.320
C(32)	H(43)	3.342	C(33)	H(21)	3.209
C(33)	H(22)	3.046	C(33)	H(23)	3.477
C(33)	H(40)	2.101	C(33)	H(41)	3.262
C(33)	H(43)	2.073	C(34)	H(20)[9)]	3.070
C(34)	H(22)	3.280	C(34)	H(40)	1.029
C(34)	H(41)	2.013	C(34)	H(42)	3.357
C(34)	H(43)	2.554	C(35)	H(30)[10)]	3.564
C(35)	H(40)	2.109	C(35)	H(41)	0.974
C(35)	H(42)	2.134	C(35)	H(60)[9)]	3.397
C(36)	H(30)[10)]	3.274	C(36)	H(40)	3.342
C(36)	H(41)	2.087	C(36)	H(42)	1.055
C(36)	H(60)[9)]	3.158	C(37)	H(23)	3.297
C(37)	H(41)	3.339	C(37)	H(42)	2.087
C(38)	H(21)	3.264	C(38)	H(22)	3.559
C(38)	H(40)	2.615	C(38)	H(43)	1.009
C(38)	H(56)	2.885	C(38)	H(63)	2.964
C(39)	H(42)	2.702	C(40)	H(25)[10)]	3.551
C(40)	H(28)[10)]	3.332	C(40)	H(32)[10)]	3.412
C(40)	H(42)	2.372	C(41)	H(23)	3.146

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(41)	H(67)[6)]	3.152	C(41)	H(74)[6)]	3.547
C(42)	H(31)[12)]	3.179	C(43)	H(7)[8)]	3.329
C(43)	H(43)	2.387	C(43)	H(53)	3.323
C(43)	H(55)	3.317	C(43)	H(56)	2.602
C(43)	H(63)	2.598	C(44)	H(7)[8)]	3.359
C(44)	H(43)	3.033	C(44)	H(53)	2.091
C(44)	H(54)	3.331	C(44)	H(56)	2.102
C(45)	H(3)[8)]	3.583	C(45)	H(7)[8)]	3.370
C(45)	H(31)[3)]	3.416	C(45)	H(36)[3)]	3.522
C(45)	H(52)[7)]	3.593	C(45)	H(53)	1.036
C(45)	H(54)	2.074	C(45)	H(55)	3.311
C(45)	H(56)	3.374	C(46)	H(7)[8)]	3.389
C(46)	H(31)[3)]	3.600	C(46)	H(36)[3)]	3.334
C(46)	H(52)[7)]	3.476	C(46)	H(53)	2.110
C(46)	H(54)	1.015	C(46)	H(55)	2.083
C(47)	H(7)[8)]	3.403	C(47)	H(12)[8)]	3.441
C(47)	H(47)[7)]	3.516	C(47)	H(53)	3.337
C(47)	H(54)	2.083	C(47)	H(55)	1.018
C(47)	H(63)	3.332	C(47)	H(71)[13)]	3.520
C(48)	H(7)[8)]	3.380	C(48)	H(43)	3.114
C(48)	H(54)	3.328	C(48)	H(55)	2.084
C(48)	H(63)	2.075	C(49)	H(21)	3.057
C(49)	H(43)	3.419	C(49)	H(53)	2.671
C(49)	H(56)	1.086	C(50)	H(8)	3.589
C(50)	H(10)	3.436	C(50)	H(17)	3.412
C(50)	H(21)	3.154	C(50)	H(43)	3.379
C(50)	H(53)	3.139	C(50)	H(56)	2.150
C(51)	H(42)[1)]	3.488	C(51)	H(45)[1)]	3.450
C(51)	H(53)	2.740	C(51)	H(56)	2.123
C(52)	H(43)	3.533	C(52)	H(55)	2.688
C(52)	H(63)	1.017	C(53)	H(12)[8)]	3.503
C(53)	H(13)[8)]	3.388	C(53)	H(19)[9)]	3.277
C(53)	H(43)	3.571	C(53)	H(55)	3.184
C(53)	H(63)	2.055	C(53)	H(69)[13)]	3.230
C(54)	H(47)[7)]	3.328	C(54)	H(55)	2.757
C(54)	H(63)	2.078	C(54)	H(64)[13)]	3.203
C(54)	H(69)[13)]	3.234	C(55)	H(63)	2.739

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(55)	H(70)	3.380	C(55)	H(72)	3.410
C(56)	H(70)	2.130	C(56)	H(71)	3.326
C(57)	H(11)[12)]	3.426	C(57)	H(12)[12)]	3.523
C(57)	H(13)[12)]	3.485	C(57)	H(15)[7)]	3.580
C(57)	H(65)[13)]	3.314	C(57)	H(70)	1.087
C(57)	H(71)	2.104	C(57)	H(72)	3.436
C(58)	H(11)[12)]	3.508	C(58)	H(12)[12)]	3.134
C(58)	H(55)[13)]	3.459	C(58)	H(65)[13)]	2.989
C(58)	H(70)	2.138	C(58)	H(71)	0.984
C(58)	H(72)	2.211	C(59)	H(55)[13)]	3.518
C(59)	H(68)[13)]	3.365	C(59)	H(70)	3.389
C(59)	H(71)	1.975	C(59)	H(72)	1.127
C(60)	H(63)	2.964	C(60)	H(71)	3.234
C(60)	H(72)	2.149	C(61)	H(37)[5)]	3.463
C(61)	H(70)	2.706	C(62)	H(18)[9)]	3.156
C(62)	H(19)[9)]	3.588	C(62)	H(63)	2.988
C(62)	H(72)	2.668	H(1)	Cl(1)[9)]	3.336
H(1)	Cl(2)[9)]	3.006	H(1)	C(1)	3.359
H(1)	C(2)	2.121	H(1)	C(3)	1.007
H(1)	C(4)	2.062	H(1)	C(5)	3.311
H(1)	C(7)	2.664	H(1)	H(2)	2.359
H(1)	H(4)	2.385	H(1)	H(34)[9)]	3.340
H(1)	H(40)	3.143	H(2)	Cl(1)[9)]	2.790
H(2)	C(2)	3.303	H(2)	C(3)	2.058
H(2)	C(4)	1.006	H(2)	C(5)	2.094
H(2)	C(6)	3.348	H(2)	C(9)[8)]	3.417
H(2)	C(10)[8)]	3.401	H(2)	C(30)[9)]	3.107
H(2)	H(1)	2.359	H(2)	H(3)	2.340
H(2)	H(5)[8)]	2.743	H(2)	H(6)[8)]	3.343
H(2)	H(9)[8)]	3.344	H(2)	H(10)[8)]	2.710
H(2)	H(34)[9)]	3.051	H(2)	H(35)[9)]	2.413
H(2)	H(36)[9)]	3.467	H(3)	C(1)	3.296
H(3)	C(3)	3.272	H(3)	C(4)	2.038
H(3)	C(5)	0.995	H(3)	C(6)	2.085
H(3)	C(8)	2.759	H(3)	C(9)	2.485
H(3)	C(45)[8)]	3.583	H(3)	H(2)	2.340
H(3)	H(3)[8)]	3.457	H(3)	H(5)	2.209

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(3)	H(5)[8)]	2.983	H(3)	H(6)	3.418
H(3)	H(7)	2.291	H(3)	H(10)[8)]	2.876
H(3)	H(35)[9)]	3.599	H(3)	H(53)[8)]	3.447
H(3)	H(59)[8)]	3.064	H(4)	N(1)	1.998
H(4)	C(1)	3.372	H(4)	C(2)	2.100
H(4)	C(3)	2.579	H(4)	C(7)	1.038
H(4)	C(12)	2.396	H(4)	C(13)	3.040
H(4)	C(17)	3.106	H(4)	C(18)	3.415
H(4)	C(20)	3.314	H(4)	C(21)	3.571
H(4)	H(1)	2.385	H(4)	H(17)	3.346
H(4)	H(22)	2.531	H(4)	H(24)	3.269
H(4)	H(27)	3.155	H(4)	H(38)	3.596
H(5)	C(4)[8)]	2.898	H(5)	C(5)[8)]	2.980
H(5)	H(2)[8)]	2.743	H(5)	H(3)	2.209
H(5)	H(3)[8)]	2.983	H(5)	H(43)[8)]	3.263
H(5)	H(58)[8)]	3.187	H(5)	H(59)[8)]	3.571
H(6)	Cl(1)[3)]	3.339	H(6)	H(2)[8)]	3.343
H(6)	H(3)	3.418	H(6)	H(20)[3)]	3.552
H(6)	H(40)[8)]	3.200	H(6)	H(43)[8)]	2.677
H(6)	H(66)[8)]	2.981	H(7)	C(43)[8)]	3.329
H(7)	C(44)[8)]	3.359	H(7)	C(45)[8)]	3.370
H(7)	C(46)[8)]	3.389	H(7)	C(47)[8)]	3.403
H(7)	C(48)[8)]	3.380	H(7)	H(3)	2.291
H(7)	H(43)[8)]	2.785	H(7)	H(58)[8)]	3.302
H(7)	H(59)[8)]	3.589	H(7)	H(66)[8)]	3.256
H(8)	C(16)[3)]	3.599	H(8)	C(50)	3.589
H(8)	H(16)[3)]	3.419	H(8)	H(29)[3)]	3.211
H(8)	H(30)[3)]	3.218	H(8)	H(57)	3.293
H(8)	H(59)	3.020	H(9)	Cl(1)[3)]	3.136
H(9)	C(15)[3)]	3.324	H(9)	C(16)[3)]	3.115
H(9)	C(17)[3)]	3.369	H(9)	C(23)[3)]	3.483
H(9)	H(2)[8)]	3.344	H(9)	H(16)[3)]	3.382
H(9)	H(29)[3)]	2.828	H(9)	H(30)[3)]	3.392
H(10)	C(4)[8)]	3.390	H(10)	C(5)[8)]	3.470
H(10)	C(50)	3.436	H(10)	H(2)[8)]	2.710
H(10)	H(3)[8)]	2.876	H(10)	H(29)[3)]	3.074
H(10)	H(57)	3.562	H(10)	H(59)	2.597

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(11)	C(57)[4)]	3.426	H(11)	C(58)[4)]	3.508
H(11)	H(15)[3)]	3.376	H(11)	H(70)[4)]	2.669
H(11)	H(71)[4)]	3.010	H(12)	C(47)[8)]	3.441
H(12)	C(53)[8)]	3.503	H(12)	C(57)[4)]	3.523
H(12)	C(58)[4)]	3.134	H(12)	H(55)[8)]	3.366
H(12)	H(65)[8)]	2.820	H(12)	H(66)[8)]	3.349
H(12)	H(70)[4)]	3.127	H(12)	H(71)[4)]	2.354
H(13)	C(14)[3)]	3.352	H(13)	C(15)[3)]	3.141
H(13)	C(53)[8)]	3.388	H(13)	C(57)[4)]	3.485
H(13)	H(14)[3)]	3.380	H(13)	H(15)[3)]	3.013
H(13)	H(65)[8)]	2.892	H(13)	H(66)[8)]	3.019
H(13)	H(70)[4)]	2.750	H(13)	H(71)[4)]	3.230
H(14)	C(12)	3.268	H(14)	C(13)	2.028
H(14)	C(14)	0.993	H(14)	C(15)	2.099
H(14)	C(16)	3.307	H(14)	C(18)	2.627
H(14)	C(19)	2.682	H(14)	C(20)	3.131
H(14)	H(13)[2)]	3.380	H(14)	H(15)	2.471
H(14)	H(18)	3.534	H(14)	H(19)	2.145
H(14)	H(20)	2.966	H(14)	H(23)	2.678
H(14)	H(48)	3.217	H(14)	H(64)[1)]	3.471
H(14)	H(66)[1)]	3.588	H(15)	Cl(2)[2)]	3.112
H(15)	C(13)	3.333	H(15)	C(14)	2.098
H(15)	C(15)	0.998	H(15)	C(16)	2.032
H(15)	C(17)	3.295	H(15)	C(57)[6)]	3.580
H(15)	H(11)[2)]	3.376	H(15)	H(13)[2)]	3.013
H(15)	H(14)	2.471	H(15)	H(16)	2.326
H(15)	H(27)[10)]	3.196	H(15)	H(70)[6)]	2.591
H(15)	H(75)[6)]	2.739	H(16)	Cl(2)[2)]	3.049
H(16)	C(12)	3.301	H(16)	C(14)	3.299
H(16)	C(15)	2.068	H(16)	C(16)	1.008
H(16)	C(17)	2.079	H(16)	C(21)	2.674
H(16)	C(22)	2.991	H(16)	C(23)	2.906
H(16)	H(8)[2)]	3.419	H(16)	H(9)[2)]	3.382
H(16)	H(15)	2.326	H(16)	H(26)	2.506
H(16)	H(27)	3.339	H(16)	H(27)[10)]	3.252
H(16)	H(29)	3.248	H(16)	H(30)	2.395
H(16)	H(41)[10)]	3.574	H(17)	V(1)	3.313

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(17)	Cl(1)	3.231	H(17)	O(1)	3.397
H(17)	N(1)	2.473	H(17)	C(7)	2.967
H(17)	C(12)	2.673	H(17)	C(13)	2.142
H(17)	C(14)	3.385	H(17)	C(18)	1.061
H(17)	C(19)	2.038	H(17)	C(20)	2.126
H(17)	C(50)	3.412	H(17)	H(4)	3.346
H(17)	H(18)	2.279	H(17)	H(19)	2.831
H(17)	H(20)	2.303	H(17)	H(21)	2.369
H(17)	H(22)	2.425	H(17)	H(23)	2.891
H(17)	H(57)	2.580	H(17)	H(58)	3.572
H(18)	C(62)[1)]	3.156	H(18)	H(14)	3.534
H(18)	H(17)	2.279	H(18)	H(56)	3.396
H(18)	H(60)	3.077	H(18)	H(76)[1)]	2.932
H(18)	H(77)[1)]	3.464	H(18)	H(78)[1)]	2.628
H(19)	C(53)[1)]	3.277	H(19)	C(62)[1)]	3.588
H(19)	H(14)	2.145	H(19)	H(17)	2.831
H(19)	H(64)[1)]	2.776	H(19)	H(66)[1)]	2.911
H(19)	H(76)[1)]	3.165	H(19)	H(78)[1)]	3.109
H(20)	C(34)[1)]	3.070	H(20)	H(6)[2)]	3.552
H(20)	H(14)	2.966	H(20)	H(17)	2.303
H(20)	H(40)[1)]	2.737	H(20)	H(64)[1)]	3.497
H(20)	H(66)[1)]	2.891	H(20)	H(76)[1)]	3.295
H(21)	O(2)	3.078	H(21)	N(3)	3.448
H(21)	C(32)	3.202	H(21)	C(33)	3.209
H(21)	C(38)	3.264	H(21)	C(49)	3.057
H(21)	C(50)	3.154	H(21)	H(17)	2.369
H(21)	H(56)	2.197	H(21)	H(57)	2.896
H(21)	H(58)	3.033	H(22)	C(32)	3.224
H(22)	C(33)	3.046	H(22)	C(34)	3.280
H(22)	C(38)	3.559	H(22)	H(4)	2.531
H(22)	H(17)	2.425	H(22)	H(56)	3.467
H(22)	H(57)	3.393	H(22)	H(58)	3.286
H(23)	O(2)	3.084	H(23)	C(32)	3.000
H(23)	C(33)	3.477	H(23)	C(37)	3.297
H(23)	C(41)	3.146	H(23)	H(14)	2.678
H(23)	H(17)	2.891	H(23)	H(48)	2.670
H(23)	H(49)	2.829	H(24)	V(1)	3.276

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(24)	N(1)	2.412	H(24)	N(2)	2.582
H(24)	C(7)	2.944	H(24)	C(12)	2.577
H(24)	C(16)	3.326	H(24)	C(17)	2.058
H(24)	C(21)	1.020	H(24)	C(22)	2.092
H(24)	C(23)	2.058	H(24)	C(24)	2.762
H(24)	C(29)	2.870	H(24)	C(31)	2.971
H(24)	H(4)	3.269	H(24)	H(25)	2.344
H(24)	H(26)	2.853	H(24)	H(27)	2.400
H(24)	H(28)	2.313	H(24)	H(29)	2.348
H(24)	H(30)	2.830	H(24)	H(38)	2.369
H(24)	H(39)	3.335	H(25)	C(40)[10)]	3.551
H(25)	H(24)	2.344	H(25)	H(42)[10)]	3.402
H(25)	H(46)[10)]	2.607	H(25)	H(75)[11)]	3.212
H(26)	H(16)	2.506	H(26)	H(24)	2.853
H(26)	H(42)[10)]	3.164	H(26)	H(46)[10)]	3.337
H(27)	C(15)[10)]	3.372	H(27)	C(16)[10)]	3.383
H(27)	H(4)	3.155	H(27)	H(15)[10)]	3.196
H(27)	H(16)	3.339	H(27)	H(16)[10)]	3.252
H(27)	H(24)	2.400	H(28)	C(40)[10)]	3.332
H(28)	H(24)	2.313	H(28)	H(42)[10)]	2.654
H(28)	H(45)[10)]	3.052	H(28)	H(46)[10)]	2.760
H(28)	H(61)[2)]	3.282	H(29)	C(10)[2)]	3.214
H(29)	H(8)[2)]	3.211	H(29)	H(9)[2)]	2.828
H(29)	H(10)[2)]	3.074	H(29)	H(16)	3.248
H(29)	H(24)	2.348	H(30)	C(35)[10)]	3.564
H(30)	C(36)[10)]	3.274	H(30)	H(8)[2)]	3.218
H(30)	H(9)[2)]	3.392	H(30)	H(16)	2.395
H(30)	H(24)	2.830	H(30)	H(41)[10)]	3.136
H(30)	H(42)[10)]	2.448	H(30)	H(46)[10)]	3.497
H(30)	H(61)[2)]	3.523	H(31)	C(24)	3.299
H(31)	C(25)	2.069	H(31)	C(26)	0.991
H(31)	C(27)	2.056	H(31)	C(28)	3.293
H(31)	C(30)	2.683	H(31)	C(42)[4)]	3.179
H(31)	C(45)[2)]	3.416	H(31)	C(46)[2)]	3.600
H(31)	H(32)	2.451	H(31)	H(34)	3.503
H(31)	H(35)	3.063	H(31)	H(36)	2.443
H(31)	H(50)[4)]	3.070	H(31)	H(51)[4)]	3.064

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(31)	H(52)[4)]	2.869	H(31)	H(53)[2)]	2.875
H(31)	H(54)[2)]	3.191	H(32)	Cl(4)[11)]	3.192
H(32)	C(25)	3.381	H(32)	C(26)	2.136
H(32)	C(27)	1.052	H(32)	C(28)	2.070
H(32)	C(29)	3.329	H(32)	C(40)[10)]	3.412
H(32)	H(31)	2.451	H(32)	H(33)	2.314
H(32)	H(44)[10)]	2.821	H(32)	H(45)[10)]	3.241
H(32)	H(51)[4)]	2.970	H(32)	H(72)[4)]	3.546
H(32)	H(77)[4)]	3.490	H(33)	Cl(3)[11)]	2.895
H(33)	Cl(4)[11)]	2.816	H(33)	C(24)	3.298
H(33)	C(26)	3.275	H(33)	C(27)	2.052
H(33)	C(28)	0.991	H(33)	C(29)	2.067
H(33)	C(31)	2.686	H(33)	H(32)	2.314
H(33)	H(37)	2.393	H(33)	H(38)	3.155
H(33)	H(39)	3.459	H(33)	H(44)[10)]	3.148
H(33)	H(46)[10)]	3.552	H(33)	H(72)[4)]	3.159
H(33)	H(73)[11)]	2.968	H(33)	H(74)[11)]	3.435
H(34)	C(4)[1)]	3.427	H(34)	H(1)[1)]	3.340
H(34)	H(2)[1)]	3.051	H(34)	H(31)	3.503
H(34)	H(70)[4)]	3.402	H(35)	C(4)[1)]	2.974
H(35)	H(2)[1)]	2.413	H(35)	H(3)[1)]	3.599
H(35)	H(31)	3.063	H(35)	H(53)[2)]	2.779
H(35)	H(54)[2)]	3.460	H(36)	C(45)[2)]	3.522
H(36)	C(46)[2)]	3.334	H(36)	H(2)[1)]	3.467
H(36)	H(31)	2.443	H(36)	H(53)[2)]	2.970
H(36)	H(54)[2)]	2.549	H(36)	H(73)[4)]	3.494
H(37)	Cl(3)[11)]	3.189	H(37)	C(61)[11)]	3.463
H(37)	H(33)	2.393	H(37)	H(54)[8)]	3.293
H(37)	H(71)[4)]	3.449	H(37)	H(73)[11)]	2.631
H(37)	H(75)[11)]	3.536	H(38)	H(4)	3.596
H(38)	H(24)	2.369	H(38)	H(33)	3.155
H(38)	H(73)[11)]	3.308	H(38)	H(75)[11)]	3.553
H(39)	H(24)	3.335	H(39)	H(33)	3.459
H(39)	H(71)[4)]	3.071	H(40)	Cl(1)[9)]	2.938
H(40)	C(19)[9)]	3.590	H(40)	C(32)	3.343
H(40)	C(33)	2.101	H(40)	C(34)	1.029
H(40)	C(35)	2.109	H(40)	C(36)	3.342

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(40)	C(38)	2.615	H(40)	H(1)	3.143
H(40)	H(6)[8)]	3.200	H(40)	H(20)[9)]	2.737
H(40)	H(41)	2.361	H(40)	H(43)	2.306
H(41)	Cl(2)[9)]	3.151	H(41)	C(33)	3.262
H(41)	C(34)	2.013	H(41)	C(35)	0.974
H(41)	C(36)	2.087	H(41)	C(37)	3.339
H(41)	H(16)[10)]	3.574	H(41)	H(30)[10)]	3.136
H(41)	H(40)	2.361	H(41)	H(42)	2.463
H(41)	H(60)[9)]	3.444	H(42)	C(23)[10)]	2.968
H(42)	C(32)	3.320	H(42)	C(34)	3.357
H(42)	C(35)	2.134	H(42)	C(36)	1.055
H(42)	C(37)	2.087	H(42)	C(39)	2.702
H(42)	C(40)	2.372	H(42)	C(51)[9)]	3.488
H(42)	H(25)[10)]	3.402	H(42)	H(26)[10)]	3.164
H(42)	H(28)[10)]	2.654	H(42)	H(30)[10)]	2.448
H(42)	H(41)	2.463	H(42)	H(44)	3.302
H(42)	H(45)	2.152	H(42)	H(46)	2.116
H(42)	H(60)[9)]	2.969	H(42)	H(61)[9)]	3.229
H(43)	N(3)	1.978	H(43)	C(9)[8)]	3.067
H(43)	C(32)	3.342	H(43)	C(33)	2.073
H(43)	C(34)	2.554	H(43)	C(38)	1.009
H(43)	C(43)	2.387	H(43)	C(44)	3.033
H(43)	C(48)	3.114	H(43)	C(49)	3.419
H(43)	C(50)	3.379	H(43)	C(52)	3.533
H(43)	C(53)	3.571	H(43)	H(5)[8)]	3.263
H(43)	H(6)[8)]	2.677	H(43)	H(7)[8)]	2.785
H(43)	H(40)	2.306	H(43)	H(56)	3.231
H(43)	H(58)	2.622	H(43)	H(63)	3.305
H(43)	H(66)	2.824	H(44)	Cl(4)[6)]	2.961
H(44)	C(27)[10)]	3.225	H(44)	C(28)[10)]	3.346
H(44)	H(32)[10)]	2.821	H(44)	H(33)[10)]	3.148
H(44)	H(42)	3.302	H(45)	C(27)[10)]	3.440
H(45)	C(51)[9)]	3.450	H(45)	H(28)[10)]	3.052
H(45)	H(32)[10)]	3.241	H(45)	H(42)	2.152
H(45)	H(60)[9)]	3.386	H(45)	H(61)[9)]	3.166
H(45)	H(62)[9)]	3.232	H(46)	C(22)[10)]	3.330
H(46)	C(23)[10)]	3.467	H(46)	C(28)[10)]	3.546

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(46)	H(25)[10)]	2.607	H(46)	H(26)[10)]	3.337
H(46)	H(28)[10)]	2.760	H(46)	H(30)[10)]	3.497
H(46)	H(33)[10)]	3.552	H(46)	H(42)	2.116
H(47)	Cl(4)[6)]	3.523	H(47)	C(47)[6)]	3.516
H(47)	C(54)[6)]	3.328	H(47)	H(55)[6)]	3.535
H(47)	H(67)[6)]	2.514	H(47)	H(68)[6)]	3.405
H(47)	H(74)[6)]	3.067	H(48)	H(14)	3.217
H(48)	H(23)	2.670	H(48)	H(67)[6)]	3.215
H(48)	H(74)[6)]	3.139	H(49)	H(23)	2.829
H(49)	H(67)[6)]	3.264	H(49)	H(68)[6)]	3.586
H(50)	H(31)[12)]	3.070	H(51)	H(31)[12)]	3.064
H(51)	H(32)[12)]	2.970	H(52)	C(45)[6)]	3.593
H(52)	C(46)[6)]	3.476	H(52)	H(31)[12)]	2.869
H(52)	H(54)[6)]	3.560	H(53)	C(26)[3)]	3.475
H(53)	C(30)[3)]	3.222	H(53)	C(43)	3.323
H(53)	C(44)	2.091	H(53)	C(45)	1.036
H(53)	C(46)	2.110	H(53)	C(47)	3.337
H(53)	C(49)	2.671	H(53)	C(50)	3.139
H(53)	C(51)	2.740	H(53)	H(3)[8)]	3.447
H(53)	H(31)[3)]	2.875	H(53)	H(35)[3)]	2.779
H(53)	H(36)[3)]	2.970	H(53)	H(54)	2.413
H(53)	H(59)	2.661	H(53)	H(60)	3.579
H(53)	H(61)	2.180	H(53)	H(62)	3.047
H(54)	Cl(3)[7)]	3.127	H(54)	C(30)[3)]	3.384
H(54)	C(44)	3.331	H(54)	C(45)	2.074
H(54)	C(46)	1.015	H(54)	C(47)	2.083
H(54)	C(48)	3.328	H(54)	H(31)[3)]	3.191
H(54)	H(35)[3)]	3.460	H(54)	H(36)[3)]	2.549
H(54)	H(37)[8)]	3.293	H(54)	H(52)[7)]	3.560
H(54)	H(53)	2.413	H(54)	H(55)	2.398
H(55)	Cl(3)[7)]	3.081	H(55)	C(43)	3.317
H(55)	C(45)	3.311	H(55)	C(46)	2.083
H(55)	C(47)	1.018	H(55)	C(48)	2.084
H(55)	C(52)	2.688	H(55)	C(53)	3.184
H(55)	C(54)	2.757	H(55)	C(58)[13)]	3.459
H(55)	C(59)[13)]	3.518	H(55)	H(12)[8)]	3.366
H(55)	H(47)[7)]	3.535	H(55)	H(54)	2.398

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(55)	H(65)	2.725	H(55)	H(67)	3.007
H(55)	H(68)	2.236	H(55)	H(71)[13)]	2.785
H(55)	H(72)[13)]	3.042	H(56)	V(2)	3.367
H(56)	Cl(4)	3.121	H(56)	N(3)	2.416
H(56)	C(20)	3.128	H(56)	C(38)	2.885
H(56)	C(43)	2.602	H(56)	C(44)	2.102
H(56)	C(45)	3.374	H(56)	C(49)	1.086
H(56)	C(50)	2.150	H(56)	C(51)	2.123
H(56)	H(18)	3.396	H(56)	H(21)	2.197
H(56)	H(22)	3.467	H(56)	H(43)	3.231
H(56)	H(57)	2.437	H(56)	H(58)	2.383
H(56)	H(59)	2.924	H(56)	H(60)	2.385
H(56)	H(61)	2.899	H(56)	H(62)	2.385
H(57)	O(1)	3.125	H(57)	C(1)	3.316
H(57)	C(2)	3.593	H(57)	C(18)	3.376
H(57)	C(20)	3.367	H(57)	H(8)	3.293
H(57)	H(10)	3.562	H(57)	H(17)	2.580
H(57)	H(21)	2.896	H(57)	H(22)	3.393
H(57)	H(56)	2.437	H(58)	C(1)	3.488
H(58)	C(2)	3.445	H(58)	C(20)	3.549
H(58)	H(5)[8)]	3.187	H(58)	H(7)[8)]	3.302
H(58)	H(17)	3.572	H(58)	H(21)	3.033
H(58)	H(22)	3.286	H(58)	H(43)	2.622
H(58)	H(56)	2.383	H(59)	C(10)	3.225
H(59)	H(3)[8)]	3.064	H(59)	H(5)[8)]	3.571
H(59)	H(7)[8)]	3.589	H(59)	H(8)	3.020
H(59)	H(10)	2.597	H(59)	H(53)	2.661
H(59)	H(56)	2.924	H(60)	C(35)[1)]	3.397
H(60)	C(36)[1)]	3.158	H(60)	H(18)	3.077
H(60)	H(41)[1)]	3.444	H(60)	H(42)[1)]	2.969
H(60)	H(45)[1)]	3.386	H(60)	H(53)	3.579
H(60)	H(56)	2.385	H(61)	H(28)[3)]	3.282
H(61)	H(30)[3)]	3.523	H(61)	H(42)[1)]	3.229
H(61)	H(45)[1)]	3.166	H(61)	H(53)	2.180
H(61)	H(56)	2.899	H(62)	H(45)[1)]	3.232
H(62)	H(53)	3.047	H(62)	H(56)	2.385
H(63)	V(2)	3.177	H(63)	N(3)	2.408

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(63)	N(4)	2.449	H(63)	C(38)	2.964
H(63)	C(43)	2.598	H(63)	C(47)	3.332
H(63)	C(48)	2.075	H(63)	C(52)	1.017
H(63)	C(53)	2.055	H(63)	C(54)	2.078
H(63)	C(55)	2.739	H(63)	C(60)	2.964
H(63)	C(62)	2.988	H(63)	H(43)	3.305
H(63)	H(64)	2.328	H(63)	H(65)	2.829
H(63)	H(66)	2.324	H(63)	H(67)	2.380
H(63)	H(68)	2.845	H(63)	H(69)	2.325
H(63)	H(76)	2.278	H(63)	H(77)	3.543
H(64)	C(19)$^{9)}$	3.518	H(64)	C(54)$^{13)}$	3.203
H(64)	H(14)$^{9)}$	3.471	H(64)	H(19)$^{9)}$	2.776
H(64)	H(20)$^{9)}$	3.497	H(64)	H(63)	2.328
H(64)	H(67)$^{13)}$	3.526	H(64)	H(68)$^{13)}$	3.018
H(64)	H(69)$^{13)}$	2.627	H(65)	C(11)$^{8)}$	3.297
H(65)	C(57)$^{13)}$	3.314	H(65)	C(58)$^{13)}$	2.989
H(65)	H(12)$^{8)}$	2.820	H(65)	H(13)$^{8)}$	2.892
H(65)	H(55)	2.725	H(65)	H(63)	2.829
H(65)	H(69)$^{13)}$	3.088	H(65)	H(70)$^{13)}$	3.443
H(65)	H(71)$^{13)}$	2.723	H(66)	C(9)$^{8)}$	3.533
H(66)	C(11)$^{8)}$	3.590	H(66)	C(19)$^{9)}$	3.328
H(66)	H(6)$^{8)}$	2.981	H(66)	H(7)$^{8)}$	3.256
H(66)	H(12)$^{8)}$	3.349	H(66)	H(13)$^{8)}$	3.019
H(66)	H(14)$^{9)}$	3.588	H(66)	H(19)$^{9)}$	2.911
H(66)	H(20)$^{9)}$	2.891	H(66)	H(43)	2.824
H(66)	H(63)	2.324	H(67)	C(41)$^{7)}$	3.152
H(67)	H(47)$^{7)}$	2.514	H(67)	H(48)$^{7)}$	3.215
H(67)	H(49)$^{7)}$	3.264	H(67)	H(55)	3.007
H(67)	H(63)	2.380	H(67)	H(64)$^{13)}$	3.526
H(68)	C(59)$^{13)}$	3.365	H(68)	H(47)$^{7)}$	3.405
H(68)	H(49)$^{7)}$	3.586	H(68)	H(55)	2.236
H(68)	H(63)	2.845	H(68)	H(64)$^{13)}$	3.018
H(68)	H(69)$^{13)}$	3.031	H(68)	H(71)$^{13)}$	3.590
H(68)	H(72)$^{13)}$	3.143	H(69)	C(53)$^{13)}$	3.230
H(69)	C(54)$^{13)}$	3.234	H(69)	H(63)	2.325
H(69)	H(64)$^{13)}$	2.627	H(69)	H(65)$^{13)}$	3.088
H(69)	H(68)$^{13)}$	3.031	H(69)	H(69)$^{13)}$	2.686

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(70)	Cl(2)[12)]	3.497	H(70)	C(11)[12)]	3.011
H(70)	C(15)[7)]	3.420	H(70)	C(55)	3.380
H(70)	C(56)	2.130	H(70)	C(57)	1.087
H(70)	C(58)	2.138	H(70)	C(59)	3.389
H(70)	C(61)	2.706	H(70)	H(11)[12)]	2.669
H(70)	H(12)[12)]	3.127	H(70)	H(13)[12)]	2.750
H(70)	H(15)[7)]	2.591	H(70)	H(34)[12)]	3.402
H(70)	H(65)[13)]	3.443	H(70)	H(71)	2.514
H(70)	H(73)	3.357	H(70)	H(74)	3.333
H(70)	H(75)	2.349	H(71)	C(11)[12)]	3.008
H(71)	C(24)[12)]	3.434	H(71)	C(28)[12)]	3.594
H(71)	C(29)[12)]	3.179	H(71)	C(31)[12)]	3.420
H(71)	C(47)[13)]	3.520	H(71)	C(56)	3.326
H(71)	C(57)	2.104	H(71)	C(58)	0.984
H(71)	C(59)	1.975	H(71)	C(60)	3.234
H(71)	H(11)[12)]	3.010	H(71)	H(12)[12)]	2.354
H(71)	H(13)[12)]	3.230	H(71)	H(37)[12)]	3.449
H(71)	H(39)[12)]	3.071	H(71)	H(55)[13)]	2.785
H(71)	H(65)[13)]	2.723	H(71)	H(68)[13)]	3.590
H(71)	H(70)	2.514	H(71)	H(72)	2.365
H(72)	Cl(3)[9)]	2.864	H(72)	C(27)[12)]	3.441
H(72)	C(28)[12)]	3.250	H(72)	C(55)	3.410
H(72)	C(57)	3.436	H(72)	C(58)	2.211
H(72)	C(59)	1.127	H(72)	C(60)	2.149
H(72)	C(62)	2.668	H(72)	H(32)[12)]	3.546
H(72)	H(33)[12)]	3.159	H(72)	H(55)[13)]	3.042
H(72)	H(68)[13)]	3.143	H(72)	H(71)	2.365
H(72)	H(76)	3.334	H(72)	H(77)	3.282
H(72)	H(78)	2.282	H(73)	C(31)[5)]	3.352
H(73)	H(33)[5)]	2.968	H(73)	H(36)[12)]	3.494
H(73)	H(37)[5)]	2.631	H(73)	H(38)[5)]	3.308
H(73)	H(70)	3.357	H(74)	C(41)[7)]	3.547
H(74)	H(33)[5)]	3.435	H(74)	H(47)[7)]	3.067
H(74)	H(48)[7)]	3.139	H(74)	H(70)	3.333
H(75)	C(15)[7)]	3.558	H(75)	H(15)[7)]	2.739
H(75)	H(25)[5)]	3.212	H(75)	H(37)[5)]	3.536
H(75)	H(38)[5)]	3.553	H(75)	H(70)	2.349

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(76)	C(19)[9)]	3.312	H(76)	H(18)[9)]	2.932
H(76)	H(19)[9)]	3.165	H(76)	H(20)[9)]	3.295
H(76)	H(63)	2.278	H(76)	H(72)	3.334
H(77)	H(18)[9)]	3.464	H(77)	H(32)[12)]	3.490
H(77)	H(63)	3.543	H(77)	H(72)	3.282
H(78)	C(19)[9)]	3.265	H(78)	H(18)[9)]	2.628
H(78)	H(19)[9)]	3.109	H(78)	H(72)	2.282

Symmetry Operators:

(1) X,-Y+1/2,Z+1/2
(2) -X+1,Y+1/2,-Z+1/2
(3) -X+1,Y+1/2-1,-Z+1/2
(4) X-1,Y,Z
(5) X+1,-Y+1/2,Z+1/2
(6) -X+2,Y+1/2,-Z+1/2
(7) -X+2,Y+1/2-1,-Z+1/2
(8) -X+1,-Y,-Z
(9) X,-Y+1/2,Z+1/2-1
(10) -X+1,-Y+1,-Z
(11) X-1,-Y+1/2,Z+1/2-1
(12) X+1,Y,Z
(13) -X+2,-Y,-Z

X-ray Structure Report for V(NAr)Me(N=C^{t}Bu$_2$)[O-2-Me-6-{(2,6-^{i}Pr$_2$C$_6$H$_3$)N=CH}C$_6$H$_3$]
(**2**)

January 11, 2008

Experimental

Data Collection

A red block crystal of $C_{38}H_{54}ON_3V$ having approximate dimensions of 0.30 x 0.30 x 0.25 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 320 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a C-centered monoclinic cell with dimensions:

a = 37.8086(15) Å
b = 9.6536(4) Å β = 94.3971(12)^o
c = 20.2844(7) Å
V = 7381.8(5) Å^3

For Z = 8 and F.W. = 619.81, the calculated density is 1.115 g/cm^3. Based on the systematic absences of:

hkl: h+k ± 2n
h0l: l ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

C2/c (#15)

The data were collected at a temperature of -30 ± 1^{o}C to a maximum 2θ value of 54.9^o. A total of 55 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 4.0^o step, at χ=45.0^o and ϕ = 90.0^o. The exposure rate was 220.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 160.0^o in 4.0^o step, at χ=45.0^o and ϕ = 270.0^o. The exposure rate was 220.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 34784 reflections that were collected, 8411 were unique (R_{int} = 0.060); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 2.992 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.582 to 0.928. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4150 observed reflections and 442 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0542$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1536$$

The standard deviation of an observation of unit weight[4] was 1.00. A Sheldrick weighting scheme was used. Plots of Σ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.75 and -0.35 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9,10] crystallographic software package.

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized:

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(8) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) CRYSTALS Issue 11: Carruthers, J.R., Rollett,J.S., Betteridge, P.W., Kinna, D., Pearce, L., Larsen, A., and Gabe, E. Chemical Crystallography Laboratory, Oxford, UK. (1999)

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{38}H_{54}ON_3V$
Formula Weight	619.81
Crystal Color, Habit	red, block
Crystal Dimensions	0.30 X 0.30 X 0.25 mm
Crystal System	monoclinic
Lattice Type	C-centered
Indexing Images	3 oscillations @ 320.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 37.8086(15) Å b = 9.6536(4) Å c = 20.2844(7) Å β = 94.3971(12) ^{o} V = 7381.8(5) Å^3
Space Group	C2/c (#15)
Z value	8
D_{calc}	1.115 g/cm^3
F_{000}	2672.00
μ(MoKα)	2.992 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	55 exposures
ω oscillation Range (χ=45.0, ϕ=90.0)	130.0 - 190.0^{o}
Exposure Rate	220.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=270.0)	0.0 - 160.0^{o}
Exposure Rate	220.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 34784 Unique: 8411 (R_{int} = 0.060)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.582 - 0.928)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$1/[0.0035Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.00σ(I))	4150
No. Variables	442
Reflection/Parameter Ratio	9.39
Residuals: R1 (I>2.00σ(I))	0.0542
Residuals: wR2 (I>2.00σ(I))	0.1536
Goodness of Fit Indicator	1.000
Max Shift/Error in Final Cycle	0.000
Maximum peak in Final Diff. Map	0.75 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.35 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
V(1)	0.11430(2)	0.09810(7)	0.56883(3)	2.764(14)
O(1)	0.10263(6)	0.1832(2)	0.47925(11)	3.33(5)
N(1)	0.16400(7)	0.1914(2)	0.55719(12)	2.43(6)
N(2)	0.12647(7)	0.0092(3)	0.63841(13)	3.21(6)
N(3)	0.07964(8)	0.2026(3)	0.59958(14)	3.47(7)
C(1)	0.09228(12)	-0.0775(4)	0.5203(2)	4.90(11)
C(2)	0.11061(9)	0.3060(4)	0.45769(17)	3.18(8)
C(3)	0.14206(9)	0.3757(3)	0.48202(16)	2.91(8)
C(4)	0.14978(11)	0.5089(4)	0.45826(19)	4.03(9)
C(5)	0.12768(12)	0.5733(4)	0.4119(2)	4.69(11)
C(6)	0.09738(12)	0.5019(5)	0.3865(2)	4.90(11)
C(7)	0.08851(10)	0.3708(4)	0.40671(17)	3.87(9)
C(8)	0.16807(9)	0.3061(3)	0.52370(16)	2.73(7)
C(9)	0.05666(12)	0.2935(5)	0.3766(2)	6.02(13)
C(10)	0.19715(8)	0.1275(3)	0.58226(16)	2.45(7)
C(11)	0.21590(9)	0.0498(3)	0.53785(17)	2.88(7)
C(12)	0.24760(10)	-0.0105(4)	0.56113(19)	3.66(9)
C(13)	0.26075(11)	0.0073(4)	0.6270(2)	4.25(10)
C(14)	0.24165(10)	0.0825(4)	0.66956(18)	3.66(9)
C(15)	0.20965(9)	0.1438(3)	0.64884(16)	2.84(7)
C(16)	0.20212(10)	0.0260(4)	0.46595(17)	3.22(8)
C(17)	0.22710(13)	0.0885(5)	0.4188(2)	5.64(12)
C(18)	0.19649(16)	-0.1270(5)	0.4508(2)	6.47(14)
C(19)	0.18956(10)	0.2319(4)	0.69555(16)	3.40(8)
C(20)	0.19922(13)	0.3852(4)	0.6899(2)	4.80(11)
C(21)	0.19518(12)	0.1868(5)	0.76769(18)	5.04(11)
C(22)	0.13526(10)	-0.0689(4)	0.69481(17)	3.29(8)
C(23)	0.15807(11)	-0.1835(4)	0.69173(19)	3.78(9)
C(24)	0.16713(13)	-0.2556(4)	0.7497(2)	4.89(11)
C(25)	0.15454(14)	-0.2199(5)	0.8084(2)	5.39(12)
C(26)	0.13167(12)	-0.1085(5)	0.8120(2)	4.97(11)
C(27)	0.12179(11)	-0.0303(4)	0.75584(19)	4.19(10)
C(28)	0.17239(12)	-0.2259(4)	0.6277(2)	4.96(11)
C(29)	0.09748(13)	0.0912(5)	0.7595(2)	6.02(13)
C(30)	0.05421(10)	0.2798(4)	0.61313(17)	3.87(9)
C(31)	0.06223(11)	0.4305(4)	0.63517(19)	4.18(10)
C(32)	0.04867(19)	0.5293(5)	0.5801(3)	8.78(19)

Table 1. Atomic coordinates and B_{iso}/B_{eq} (continued)

atom	x	y	z	B_{eq}
C(33)	0.10182(13)	0.4509(5)	0.6473(2)	6.44(14)
C(34)	0.04619(15)	0.4678(6)	0.6998(2)	7.57(16)
C(35)	0.01642(11)	0.2180(5)	0.6011(2)	5.02(11)
C(36)	-0.01422(17)	0.2947(9)	0.6222(5)	16.0(3)
C(37)	0.00948(18)	0.2065(9)	0.5243(3)	11.6(2)
C(38)	0.01637(17)	0.0700(7)	0.6230(4)	11.2(2)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	0.0851	-0.1418	0.5522	5.83
H(2)	0.0723	-0.0508	0.4919	5.84
H(3)	0.1095	-0.1195	0.4950	5.85
H(4)	0.1731	0.5593	0.4768	4.86
H(5)	0.1332	0.6737	0.3956	5.62
H(6)	0.0805	0.5508	0.3501	5.86
H(7)	0.1936	0.3443	0.5248	3.28
H(8)	0.0569	0.2015	0.3932	7.13
H(9)	0.0356	0.3389	0.3878	7.13
H(10)	0.0573	0.2915	0.3299	7.13
H(11)	0.2645	-0.0717	0.5310	4.42
H(12)	0.2854	-0.0429	0.6483	5.07
H(13)	0.2517	0.0985	0.7194	4.34
H(14)	0.1775	0.0761	0.4581	3.87
H(15)	0.2167	0.0817	0.3747	6.75
H(16)	0.2490	0.0396	0.4224	6.76
H(17)	0.2312	0.1831	0.4297	6.77
H(18)	0.1890	-0.1381	0.4053	7.80
H(19)	0.2181	-0.1756	0.4604	7.80
H(20)	0.1789	-0.1628	0.4771	7.80
H(21)	0.1623	0.2204	0.6811	4.07
H(22)	0.1924	0.4171	0.6465	5.77
H(23)	0.1872	0.4373	0.7210	5.77
H(24)	0.2241	0.3963	0.6988	5.78
H(25)	0.2183	0.2127	0.7849	6.02
H(26)	0.1781	0.2307	0.7926	6.02
H(27)	0.1926	0.0892	0.7705	6.03
H(28)	0.1843	-0.3408	0.7482	5.82
H(29)	0.1622	-0.2771	0.8512	6.43
H(30)	0.1217	-0.0828	0.8574	5.98
H(31)	0.1860	-0.3081	0.6342	5.98
H(32)	0.1532	-0.2423	0.5955	5.99
H(33)	0.1870	-0.1540	0.6127	5.98
H(34)	0.0797	0.0874	0.7239	7.29
H(35)	0.0866	0.0889	0.8003	7.29
H(36)	0.1107	0.1745	0.7568	7.30
H(37)	0.0514	0.6222	0.5952	10.42

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} (continued)

atom	x	y	z	B_{eq}
H(38)	0.0619	0.5161	0.5427	10.42
H(39)	0.0243	0.5111	0.5683	10.43
H(40)	0.1128	0.4363	0.6072	7.79
H(41)	0.1065	0.5426	0.6625	7.78
H(42)	0.1110	0.3866	0.6796	7.79
H(43)	0.0574	0.5485	0.7181	9.23
H(44)	0.0215	0.4848	0.6913	9.23
H(45)	0.0497	0.3932	0.7302	9.23
H(46)	-0.0327	0.2316	0.6301	19.35
H(47)	-0.0075	0.3442	0.6616	19.35
H(48)	-0.0223	0.3580	0.5885	19.35
H(49)	-0.0153	0.1998	0.5130	13.81
H(50)	0.0186	0.2863	0.5041	13.81
H(51)	0.0210	0.1262	0.5092	13.81
H(52)	0.0366	0.0242	0.6086	13.41
H(53)	0.0170	0.0663	0.6699	13.42
H(54)	-0.0045	0.0255	0.6045	13.41

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
V(1)	0.0314(3)	0.0366(3)	0.0370(3)	0.0003(2)	0.0027(2)	0.0031(2)
O(1)	0.0372(13)	0.0519(17)	0.0365(13)	-0.0043(12)	-0.0026(10)	-0.0018(12)
N(1)	0.0321(14)	0.0319(17)	0.0279(14)	0.0011(12)	0.0000(11)	-0.0003(12)
N(2)	0.0359(16)	0.0447(19)	0.0414(17)	-0.0004(14)	0.0038(12)	0.0065(14)
N(3)	0.0390(17)	0.051(2)	0.0423(17)	0.0095(16)	0.0051(13)	0.0046(14)
C(1)	0.060(2)	0.055(2)	0.069(2)	-0.006(2)	-0.005(2)	-0.003(2)
C(2)	0.041(2)	0.046(2)	0.0344(19)	0.0068(19)	0.0054(15)	0.0012(17)
C(3)	0.042(2)	0.037(2)	0.0321(17)	0.0056(17)	0.0038(15)	0.0050(15)
C(4)	0.055(2)	0.044(2)	0.055(2)	0.008(2)	0.0070(19)	0.0087(19)
C(5)	0.067(2)	0.051(2)	0.060(2)	0.014(2)	0.004(2)	0.020(2)
C(6)	0.068(3)	0.068(3)	0.049(2)	0.027(2)	-0.000(2)	0.022(2)
C(7)	0.043(2)	0.070(3)	0.0335(19)	0.012(2)	-0.0033(16)	0.0055(19)
C(8)	0.0342(18)	0.035(2)	0.0343(18)	0.0001(16)	0.0042(14)	-0.0023(16)
C(9)	0.056(2)	0.106(4)	0.063(2)	0.017(2)	-0.019(2)	0.010(2)
C(10)	0.0269(17)	0.029(2)	0.0364(18)	0.0016(14)	0.0001(13)	0.0026(14)
C(11)	0.0352(19)	0.0303(19)	0.044(2)	-0.0017(16)	0.0026(15)	-0.0008(15)
C(12)	0.039(2)	0.040(2)	0.061(2)	0.0055(18)	0.0058(17)	0.0033(18)
C(13)	0.043(2)	0.054(2)	0.064(2)	0.011(2)	-0.0048(19)	0.016(2)
C(14)	0.043(2)	0.053(2)	0.042(2)	0.005(2)	-0.0082(16)	0.0095(18)
C(15)	0.039(2)	0.034(2)	0.0343(18)	-0.0045(16)	-0.0020(15)	0.0043(15)
C(16)	0.041(2)	0.043(2)	0.0391(19)	0.0007(18)	0.0064(15)	-0.0043(17)
C(17)	0.080(3)	0.084(3)	0.050(2)	-0.024(2)	0.003(2)	0.008(2)
C(18)	0.129(4)	0.065(3)	0.053(2)	-0.033(3)	0.014(2)	-0.014(2)
C(19)	0.046(2)	0.051(2)	0.0319(19)	-0.0026(19)	0.0019(15)	-0.0050(17)
C(20)	0.079(3)	0.051(2)	0.053(2)	-0.008(2)	0.008(2)	-0.015(2)
C(21)	0.076(3)	0.077(3)	0.038(2)	-0.009(2)	-0.001(2)	-0.000(2)
C(22)	0.039(2)	0.044(2)	0.042(2)	-0.0074(17)	0.0041(16)	0.0082(17)
C(23)	0.053(2)	0.038(2)	0.053(2)	-0.006(2)	0.0053(18)	0.0086(18)
C(24)	0.075(3)	0.043(2)	0.067(3)	0.004(2)	-0.004(2)	0.016(2)
C(25)	0.082(3)	0.063(3)	0.058(2)	-0.004(2)	-0.002(2)	0.024(2)
C(26)	0.069(2)	0.074(3)	0.046(2)	-0.013(2)	0.008(2)	0.017(2)
C(27)	0.050(2)	0.062(2)	0.048(2)	-0.001(2)	0.0085(18)	0.009(2)
C(28)	0.073(3)	0.041(2)	0.074(3)	0.006(2)	0.008(2)	0.011(2)
C(29)	0.076(3)	0.097(4)	0.058(2)	0.022(3)	0.018(2)	0.006(2)
C(30)	0.047(2)	0.066(2)	0.035(2)	0.011(2)	0.0062(17)	0.0041(18)
C(31)	0.057(2)	0.056(2)	0.048(2)	0.014(2)	0.0131(19)	-0.0010(19)
C(32)	0.168(6)	0.062(3)	0.100(4)	0.021(4)	-0.014(4)	0.013(3)

Table 3. Anisotropic displacement parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(33)	0.076(3)	0.064(3)	0.106(4)	-0.005(2)	0.017(2)	-0.018(3)
C(34)	0.090(4)	0.113(4)	0.089(3)	-0.012(3)	0.035(3)	-0.040(3)
C(35)	0.035(2)	0.081(3)	0.075(2)	0.008(2)	0.005(2)	0.006(2)
C(36)	0.058(3)	0.190(9)	0.365(13)	-0.014(5)	0.040(5)	-0.153(9)
C(37)	0.086(4)	0.224(9)	0.128(5)	-0.029(5)	-0.021(4)	-0.034(6)
C(38)	0.067(4)	0.123(6)	0.235(9)	-0.032(4)	-0.000(4)	0.014(6)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
V(1)	O(1)	2.012(2)	V(1)	N(1)	2.113(2)
V(1)	N(2)	1.685(2)	V(1)	N(3)	1.803(3)
V(1)	C(1)	2.100(4)	O(1)	C(2)	1.307(4)
N(1)	C(8)	1.314(4)	N(1)	C(10)	1.454(3)
N(2)	C(22)	1.389(4)	N(3)	C(30)	1.263(5)
C(2)	C(3)	1.421(4)	C(2)	C(7)	1.423(5)
C(3)	C(4)	1.411(5)	C(3)	C(8)	1.417(4)
C(4)	C(5)	1.360(5)	C(5)	C(6)	1.401(6)
C(6)	C(7)	1.380(6)	C(7)	C(9)	1.505(6)
C(10)	C(11)	1.405(4)	C(10)	C(15)	1.405(4)
C(11)	C(12)	1.383(5)	C(11)	C(16)	1.528(4)
C(12)	C(13)	1.399(5)	C(13)	C(14)	1.375(5)
C(14)	C(15)	1.383(5)	C(15)	C(19)	1.519(5)
C(16)	C(17)	1.520(6)	C(16)	C(18)	1.520(6)
C(19)	C(20)	1.531(5)	C(19)	C(21)	1.526(4)
C(22)	C(23)	1.407(5)	C(22)	C(27)	1.424(5)
C(23)	C(24)	1.387(5)	C(23)	C(28)	1.502(5)
C(24)	C(25)	1.360(6)	C(25)	C(26)	1.385(7)
C(26)	C(27)	1.394(6)	C(27)	C(29)	1.496(6)
C(30)	C(31)	1.545(6)	C(30)	C(35)	1.550(5)
C(31)	C(32)	1.527(7)	C(31)	C(33)	1.511(6)
C(31)	C(34)	1.529(6)	C(35)	C(36)	1.466(9)
C(35)	C(37)	1.563(7)	C(35)	C(38)	1.497(9)

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	0.950	C(1)	H(2)	0.950
C(1)	H(3)	0.950	C(4)	H(4)	1.050
C(5)	H(5)	1.050	C(6)	H(6)	1.050
C(8)	H(7)	1.032	C(9)	H(8)	0.950
C(9)	H(9)	0.950	C(9)	H(10)	0.950
C(12)	H(11)	1.090	C(13)	H(12)	1.110
C(14)	H(13)	1.063	C(16)	H(14)	1.050
C(17)	H(15)	0.950	C(17)	H(16)	0.950
C(17)	H(17)	0.950	C(18)	H(18)	0.950
C(18)	H(19)	0.950	C(18)	H(20)	0.950
C(19)	H(21)	1.056	C(20)	H(22)	0.950
C(20)	H(23)	0.950	C(20)	H(24)	0.950
C(21)	H(25)	0.950	C(21)	H(26)	0.950
C(21)	H(27)	0.950	C(24)	H(28)	1.050
C(25)	H(29)	1.050	C(26)	H(30)	1.050
C(28)	H(31)	0.950	C(28)	H(32)	0.950
C(28)	H(33)	0.950	C(29)	H(34)	0.950
C(29)	H(35)	0.950	C(29)	H(36)	0.950
C(32)	H(37)	0.950	C(32)	H(38)	0.950
C(32)	H(39)	0.950	C(33)	H(40)	0.950
C(33)	H(41)	0.950	C(33)	H(42)	0.950
C(34)	H(43)	0.950	C(34)	H(44)	0.950
C(34)	H(45)	0.950	C(36)	H(46)	0.950
C(36)	H(47)	0.950	C(36)	H(48)	0.950
C(37)	H(49)	0.950	C(37)	H(50)	0.950
C(37)	H(51)	0.950	C(38)	H(52)	0.950
C(38)	H(53)	0.950	C(38)	H(54)	0.950

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
O(1)	V(1)	N(1)	81.97(9)	O(1)	V(1)	N(2)	172.34(13)
O(1)	V(1)	N(3)	88.22(11)	O(1)	V(1)	C(1)	81.68(14)
N(1)	V(1)	N(2)	97.04(11)	N(1)	V(1)	N(3)	118.43(13)
N(1)	V(1)	C(1)	128.02(15)	N(2)	V(1)	N(3)	98.87(13)
N(2)	V(1)	C(1)	93.10(15)	N(3)	V(1)	C(1)	109.96(16)
V(1)	O(1)	C(2)	129.2(2)	V(1)	N(1)	C(8)	123.9(2)
V(1)	N(1)	C(10)	122.0(2)	C(8)	N(1)	C(10)	114.0(2)
V(1)	N(2)	C(22)	177.2(2)	V(1)	N(3)	C(30)	172.3(2)
O(1)	C(2)	C(3)	121.4(3)	O(1)	C(2)	C(7)	120.2(3)
C(3)	C(2)	C(7)	118.2(3)	C(2)	C(3)	C(4)	120.1(3)
C(2)	C(3)	C(8)	120.4(3)	C(4)	C(3)	C(8)	118.9(3)
C(3)	C(4)	C(5)	121.4(3)	C(4)	C(5)	C(6)	118.1(4)
C(5)	C(6)	C(7)	123.5(3)	C(2)	C(7)	C(6)	118.5(3)
C(2)	C(7)	C(9)	118.9(3)	C(6)	C(7)	C(9)	122.6(3)
N(1)	C(8)	C(3)	127.2(3)	N(1)	C(10)	C(11)	117.8(2)
N(1)	C(10)	C(15)	120.4(2)	C(11)	C(10)	C(15)	121.8(2)
C(10)	C(11)	C(12)	118.2(3)	C(10)	C(11)	C(16)	122.7(2)
C(12)	C(11)	C(16)	119.1(3)	C(11)	C(12)	C(13)	120.6(3)
C(12)	C(13)	C(14)	120.0(3)	C(13)	C(14)	C(15)	121.6(3)
C(10)	C(15)	C(14)	117.8(3)	C(10)	C(15)	C(19)	121.0(2)
C(14)	C(15)	C(19)	121.2(2)	C(11)	C(16)	C(17)	111.0(3)
C(11)	C(16)	C(18)	111.8(3)	C(17)	C(16)	C(18)	110.0(3)
C(15)	C(19)	C(20)	111.2(3)	C(15)	C(19)	C(21)	113.5(3)
C(20)	C(19)	C(21)	109.3(3)	N(2)	C(22)	C(23)	119.9(3)
N(2)	C(22)	C(27)	119.7(3)	C(23)	C(22)	C(27)	120.4(3)
C(22)	C(23)	C(24)	118.0(3)	C(22)	C(23)	C(28)	121.0(3)
C(24)	C(23)	C(28)	121.0(3)	C(23)	C(24)	C(25)	122.3(4)
C(24)	C(25)	C(26)	120.3(4)	C(25)	C(26)	C(27)	120.4(4)
C(22)	C(27)	C(26)	118.5(3)	C(22)	C(27)	C(29)	120.7(3)
C(26)	C(27)	C(29)	120.7(3)	N(3)	C(30)	C(31)	118.8(3)
N(3)	C(30)	C(35)	116.5(3)	C(31)	C(30)	C(35)	124.5(3)
C(30)	C(31)	C(32)	109.2(3)	C(30)	C(31)	C(33)	109.9(3)
C(30)	C(31)	C(34)	112.8(3)	C(32)	C(31)	C(33)	108.1(4)
C(32)	C(31)	C(34)	110.3(4)	C(33)	C(31)	C(34)	106.5(3)
C(30)	C(35)	C(36)	119.9(4)	C(30)	C(35)	C(37)	105.5(4)
C(30)	C(35)	C(38)	110.0(3)	C(36)	C(35)	C(37)	104.5(5)
C(36)	C(35)	C(38)	112.1(5)	C(37)	C(35)	C(38)	103.0(5)

Table 6. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
V(1)	C(1)	H(1)	109.5	V(1)	C(1)	H(2)	109.5
V(1)	C(1)	H(3)	109.5	H(1)	C(1)	H(2)	109.5
H(1)	C(1)	H(3)	109.5	H(2)	C(1)	H(3)	109.5
C(3)	C(4)	H(4)	119.3	C(5)	C(4)	H(4)	119.3
C(4)	C(5)	H(5)	121.0	C(6)	C(5)	H(5)	120.9
C(5)	C(6)	H(6)	118.2	C(7)	C(6)	H(6)	118.2
N(1)	C(8)	H(7)	115.9	C(3)	C(8)	H(7)	116.6
C(7)	C(9)	H(8)	109.5	C(7)	C(9)	H(9)	109.5
C(7)	C(9)	H(10)	109.5	H(8)	C(9)	H(9)	109.5
H(8)	C(9)	H(10)	109.5	H(9)	C(9)	H(10)	109.5
C(11)	C(12)	H(11)	124.5	C(13)	C(12)	H(11)	114.9
C(12)	C(13)	H(12)	123.0	C(14)	C(13)	H(12)	116.9
C(13)	C(14)	H(13)	120.5	C(15)	C(14)	H(13)	117.9
C(11)	C(16)	H(14)	108.0	C(17)	C(16)	H(14)	108.0
C(18)	C(16)	H(14)	108.0	C(16)	C(17)	H(15)	109.5
C(16)	C(17)	H(16)	109.5	C(16)	C(17)	H(17)	109.5
H(15)	C(17)	H(16)	109.5	H(15)	C(17)	H(17)	109.5
H(16)	C(17)	H(17)	109.5	C(16)	C(18)	H(18)	109.5
C(16)	C(18)	H(19)	109.5	C(16)	C(18)	H(20)	109.5
H(18)	C(18)	H(19)	109.5	H(18)	C(18)	H(20)	109.5
H(19)	C(18)	H(20)	109.5	C(15)	C(19)	H(21)	107.0
C(20)	C(19)	H(21)	108.3	C(21)	C(19)	H(21)	107.3
C(19)	C(20)	H(22)	109.5	C(19)	C(20)	H(23)	109.5
C(19)	C(20)	H(24)	109.5	H(22)	C(20)	H(23)	109.5
H(22)	C(20)	H(24)	109.5	H(23)	C(20)	H(24)	109.5
C(19)	C(21)	H(25)	109.5	C(19)	C(21)	H(26)	109.5
C(19)	C(21)	H(27)	109.5	H(25)	C(21)	H(26)	109.5
H(25)	C(21)	H(27)	109.5	H(26)	C(21)	H(27)	109.5
C(23)	C(24)	H(28)	118.8	C(25)	C(24)	H(28)	118.8
C(24)	C(25)	H(29)	119.8	C(26)	C(25)	H(29)	119.8
C(25)	C(26)	H(30)	119.8	C(27)	C(26)	H(30)	119.8
C(23)	C(28)	H(31)	109.5	C(23)	C(28)	H(32)	109.5
C(23)	C(28)	H(33)	109.5	H(31)	C(28)	H(32)	109.5
H(31)	C(28)	H(33)	109.5	H(32)	C(28)	H(33)	109.5
C(27)	C(29)	H(34)	109.5	C(27)	C(29)	H(35)	109.5
C(27)	C(29)	H(36)	109.5	H(34)	C(29)	H(35)	109.5
H(34)	C(29)	H(36)	109.5	H(35)	C(29)	H(36)	109.5

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(31)	C(32)	H(37)	109.5	C(31)	C(32)	H(38)	109.5
C(31)	C(32)	H(39)	109.5	H(37)	C(32)	H(38)	109.5
H(37)	C(32)	H(39)	109.5	H(38)	C(32)	H(39)	109.5
C(31)	C(33)	H(40)	109.5	C(31)	C(33)	H(41)	109.5
C(31)	C(33)	H(42)	109.5	H(40)	C(33)	H(41)	109.5
H(40)	C(33)	H(42)	109.5	H(41)	C(33)	H(42)	109.5
C(31)	C(34)	H(43)	109.5	C(31)	C(34)	H(44)	109.5
C(31)	C(34)	H(45)	109.5	H(43)	C(34)	H(44)	109.5
H(43)	C(34)	H(45)	109.5	H(44)	C(34)	H(45)	109.5
C(35)	C(36)	H(46)	109.5	C(35)	C(36)	H(47)	109.5
C(35)	C(36)	H(48)	109.5	H(46)	C(36)	H(47)	109.5
H(46)	C(36)	H(48)	109.5	H(47)	C(36)	H(48)	109.5
C(35)	C(37)	H(49)	109.5	C(35)	C(37)	H(50)	109.5
C(35)	C(37)	H(51)	109.5	H(49)	C(37)	H(50)	109.5
H(49)	C(37)	H(51)	109.5	H(50)	C(37)	H(51)	109.5
C(35)	C(38)	H(52)	109.5	C(35)	C(38)	H(53)	109.5
C(35)	C(38)	H(54)	109.5	H(52)	C(38)	H(53)	109.5
H(52)	C(38)	H(54)	109.5	H(53)	C(38)	H(54)	109.5

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle
O(1)	V(1)	N(1)	C(8)	30.7(2)
N(1)	V(1)	O(1)	C(2)	-42.5(2)
N(2)	V(1)	O(1)	C(2)	-125.6(9)
N(3)	V(1)	O(1)	C(2)	76.6(2)
N(1)	V(1)	N(2)	C(22)	-150(5)
N(2)	V(1)	N(1)	C(10)	27.4(2)
N(3)	V(1)	N(1)	C(8)	-52.9(2)
C(1)	V(1)	N(1)	C(8)	103.5(2)
N(2)	V(1)	N(3)	C(30)	-167(2)
C(1)	V(1)	N(2)	C(22)	-21(5)
V(1)	O(1)	C(2)	C(3)	30.4(4)
V(1)	N(1)	C(8)	C(3)	-10.2(4)
V(1)	N(1)	C(10)	C(15)	-82.8(3)
C(8)	N(1)	C(10)	C(15)	101.2(3)
V(1)	N(2)	C(22)	C(23)	81(5)
V(1)	N(3)	C(30)	C(31)	-99(2)
O(1)	C(2)	C(3)	C(4)	-179.4(3)
O(1)	C(2)	C(7)	C(6)	178.4(3)
C(3)	C(2)	C(7)	C(6)	-4.3(5)
C(7)	C(2)	C(3)	C(4)	3.4(5)
C(2)	C(3)	C(4)	C(5)	-0.2(5)
C(4)	C(3)	C(8)	N(1)	170.2(3)
C(3)	C(4)	C(5)	C(6)	-2.0(6)
C(5)	C(6)	C(7)	C(2)	2.2(6)
N(1)	C(10)	C(11)	C(12)	179.5(3)
N(1)	C(10)	C(15)	C(14)	-178.9(3)
C(11)	C(10)	C(15)	C(14)	1.1(5)
C(15)	C(10)	C(11)	C(12)	-0.5(5)
C(10)	C(11)	C(12)	C(13)	-0.9(5)
C(10)	C(11)	C(16)	C(18)	-118.9(4)
C(12)	C(11)	C(16)	C(18)	59.1(4)
C(11)	C(12)	C(13)	C(14)	1.6(6)
C(13)	C(14)	C(15)	C(10)	-0.3(5)
C(10)	C(15)	C(19)	C(20)	-83.9(4)
C(14)	C(15)	C(19)	C(20)	92.9(4)
N(2)	C(22)	C(23)	C(24)	177.8(3)
N(2)	C(22)	C(27)	C(26)	-178.5(3)
O(1)	V(1)	N(1)	C(10)	-144.9(2)
O(1)	V(1)	N(2)	C(22)	-68(5)
O(1)	V(1)	N(3)	C(30)	10(2)
C(1)	V(1)	O(1)	C(2)	-173.0(3)
N(2)	V(1)	N(1)	C(8)	-156.9(2)
N(1)	V(1)	N(3)	C(30)	90(2)
N(3)	V(1)	N(1)	C(10)	131.5(2)
C(1)	V(1)	N(1)	C(10)	-72.1(2)
N(3)	V(1)	N(2)	C(22)	89(5)
C(1)	V(1)	N(3)	C(30)	-71(2)
V(1)	O(1)	C(2)	C(7)	-152.4(2)
V(1)	N(1)	C(10)	C(11)	97.2(3)
C(8)	N(1)	C(10)	C(11)	-78.8(3)
C(10)	N(1)	C(8)	C(3)	165.7(3)
V(1)	N(2)	C(22)	C(27)	-101(5)
V(1)	N(3)	C(30)	C(35)	77(2)
O(1)	C(2)	C(3)	C(8)	8.7(5)
O(1)	C(2)	C(7)	C(9)	-2.1(5)
C(3)	C(2)	C(7)	C(9)	175.2(3)
C(7)	C(2)	C(3)	C(8)	-168.5(3)
C(2)	C(3)	C(8)	N(1)	-17.8(5)
C(8)	C(3)	C(4)	C(5)	171.9(3)
C(4)	C(5)	C(6)	C(7)	1.0(6)
C(5)	C(6)	C(7)	C(9)	-177.3(4)
N(1)	C(10)	C(11)	C(16)	-2.4(4)
N(1)	C(10)	C(15)	C(19)	-1.9(4)
C(11)	C(10)	C(15)	C(19)	178.1(3)
C(15)	C(10)	C(11)	C(16)	177.6(3)
C(10)	C(11)	C(16)	C(17)	117.8(3)
C(12)	C(11)	C(16)	C(17)	-64.1(4)
C(16)	C(11)	C(12)	C(13)	-179.0(3)
C(12)	C(13)	C(14)	C(15)	-1.0(6)
C(13)	C(14)	C(15)	C(19)	-177.3(3)
C(10)	C(15)	C(19)	C(21)	152.4(3)
C(14)	C(15)	C(19)	C(21)	-30.8(5)
N(2)	C(22)	C(23)	C(28)	-1.8(5)
N(2)	C(22)	C(27)	C(29)	1.7(5)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(23)	C(22)	C(27)	C(26)	-0.1(4)
C(27)	C(22)	C(23)	C(24)	-0.7(5)
C(22)	C(23)	C(24)	C(25)	0.6(6)
C(23)	C(24)	C(25)	C(26)	0.3(7)
C(25)	C(26)	C(27)	C(22)	1.0(6)
N(3)	C(30)	C(31)	C(32)	109.0(4)
N(3)	C(30)	C(31)	C(34)	-128.0(3)
N(3)	C(30)	C(35)	C(37)	-69.2(5)
C(31)	C(30)	C(35)	C(36)	-11.4(7)
C(31)	C(30)	C(35)	C(38)	-143.6(4)
C(35)	C(30)	C(31)	C(33)	175.5(3)

atom1	atom2	atom3	atom4	angle
C(23)	C(22)	C(27)	C(29)	-179.9(2)
C(27)	C(22)	C(23)	C(28)	179.8(3)
C(28)	C(23)	C(24)	C(25)	-179.9(3)
C(24)	C(25)	C(26)	C(27)	-1.1(7)
C(25)	C(26)	C(27)	C(29)	-179.2(4)
N(3)	C(30)	C(31)	C(33)	-9.4(4)
N(3)	C(30)	C(35)	C(36)	173.4(5)
N(3)	C(30)	C(35)	C(38)	41.2(5)
C(31)	C(30)	C(35)	C(37)	106.0(5)
C(35)	C(30)	C(31)	C(32)	-66.1(5)
C(35)	C(30)	C(31)	C(34)	56.9(5)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
O(1)	H(30)[1)]	2.801	C(1)	H(49)[2)]	3.165
C(2)	H(29)[1)]	3.033	C(2)	H(30)[1)]	3.015
C(3)	H(29)[1)]	2.973	C(4)	H(11)[3)]	3.289
C(4)	H(12)[3)]	3.404	C(4)	H(20)[4)]	3.367
C(4)	H(29)[1)]	3.179	C(5)	H(3)[4)]	3.506
C(5)	H(20)[4)]	3.405	C(5)	H(29)[1)]	3.413
C(6)	H(29)[1)]	3.391	C(6)	H(46)[5)]	3.548
C(6)	H(48)[5)]	3.218	C(7)	H(29)[1)]	3.214
C(7)	H(30)[1)]	3.240	C(8)	H(16)[3)]	3.568
C(8)	H(29)[1)]	3.501	C(9)	H(30)[1)]	3.239
C(9)	H(43)[6)]	3.561	C(10)	H(17)[3)]	3.292
C(11)	H(17)[3)]	3.298	C(12)	H(4)[3)]	3.188
C(12)	H(7)[3)]	3.339	C(12)	H(17)[3)]	3.263
C(12)	H(19)[7)]	3.338	C(13)	H(4)[3)]	3.451
C(13)	H(17)[3)]	3.225	C(13)	H(23)[8)]	3.594
C(13)	H(25)[8)]	3.420	C(13)	H(28)[9)]	3.474
C(13)	H(29)[9)]	3.581	C(14)	H(17)[3)]	3.246
C(14)	H(24)[8)]	3.393	C(14)	H(28)[9)]	3.232
C(15)	H(17)[3)]	3.298	C(17)	H(7)[3)]	3.193
C(17)	H(22)[3)]	3.412	C(17)	H(24)[3)]	3.128
C(17)	H(29)[1)]	3.271	C(18)	H(4)[10)]	3.211
C(18)	H(5)[10)]	3.206	C(18)	H(11)[7)]	3.271
C(18)	H(26)[1)]	3.381	C(20)	H(12)[9)]	3.361
C(20)	H(13)[9)]	3.247	C(20)	H(15)[3)]	3.544
C(20)	H(16)[3)]	3.200	C(20)	H(28)[4)]	2.969
C(20)	H(31)[4)]	3.194	C(21)	H(12)[9)]	3.171
C(21)	H(15)[11)]	3.439	C(21)	H(18)[11)]	2.858
C(23)	H(41)[10)]	3.311	C(24)	H(13)[8)]	3.391
C(24)	H(15)[11)]	3.470	C(24)	H(23)[10)]	3.125
C(24)	H(41)[10)]	3.398	C(25)	H(14)[11)]	3.391
C(25)	H(15)[11)]	2.938	C(26)	H(8)[11)]	3.495
C(26)	H(10)[11)]	3.364	C(26)	H(14)[11)]	3.331
C(26)	H(15)[11)]	3.376	C(28)	H(22)[10)]	3.543
C(28)	H(41)[10)]	3.459	C(32)	H(1)[4)]	3.524
C(32)	H(9)[5)]	3.538	C(32)	H(49)[5)]	3.410
C(32)	H(50)[5)]	3.448	C(34)	H(6)[12)]	3.225
C(34)	H(10)[12)]	3.518	C(34)	H(44)[13)]	3.511

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C(34)	H(47)[13)]	3.478	C(36)	H(6)[5)]	3.007
C(36)	H(45)[13)]	3.505	C(37)	H(2)[2)]	3.431
C(37)	H(39)[5)]	3.497	C(37)	H(51)[2)]	3.461
C(37)	H(54)[2)]	3.435	C(38)	H(51)[2)]	3.492
H(1)	C(32)[10)]	3.524	H(1)	H(37)[10)]	2.783
H(1)	H(38)[10)]	3.418	H(1)	H(49)[2)]	2.912
H(2)	C(37)[2)]	3.431	H(2)	H(46)[2)]	3.293
H(2)	H(49)[2)]	2.586	H(2)	H(51)[2)]	3.599
H(2)	H(54)[2)]	3.112	H(3)	C(5)[10)]	3.506
H(3)	H(5)[10)]	3.022	H(3)	H(30)[1)]	3.467
H(4)	C(12)[3)]	3.188	H(4)	C(13)[3)]	3.451
H(4)	C(18)[4)]	3.211	H(4)	H(11)[3)]	2.382
H(4)	H(12)[3)]	3.087	H(4)	H(16)[3)]	3.586
H(4)	H(18)[4)]	3.337	H(4)	H(19)[4)]	3.106
H(4)	H(20)[4)]	2.692	H(4)	H(31)[4)]	3.441
H(4)	H(32)[4)]	3.210	H(5)	C(18)[4)]	3.206
H(5)	H(3)[4)]	3.022	H(5)	H(12)[3)]	3.504
H(5)	H(18)[4)]	2.780	H(5)	H(20)[4)]	2.788
H(5)	H(26)[6)]	2.939	H(5)	H(35)[6)]	3.400
H(5)	H(36)[6)]	3.228	H(6)	C(34)[6)]	3.225
H(6)	C(36)[5)]	3.007	H(6)	H(36)[6)]	3.499
H(6)	H(43)[6)]	2.913	H(6)	H(45)[6)]	2.671
H(6)	H(46)[5)]	2.820	H(6)	H(47)[5)]	2.934
H(6)	H(48)[5)]	2.754	H(7)	C(12)[3)]	3.339
H(7)	C(17)[3)]	3.193	H(7)	H(11)[3)]	2.981
H(7)	H(16)[3)]	2.600	H(7)	H(17)[3)]	2.933
H(8)	C(26)[1)]	3.495	H(8)	H(30)[1)]	2.848
H(8)	H(54)[2)]	2.958	H(9)	C(32)[5)]	3.538
H(9)	H(37)[5)]	3.354	H(9)	H(39)[5)]	2.887
H(9)	H(44)[5)]	3.100	H(9)	H(47)[5)]	3.367
H(9)	H(48)[5)]	3.014	H(10)	C(26)[1)]	3.364
H(10)	C(34)[6)]	3.518	H(10)	H(30)[1)]	3.177
H(10)	H(43)[6)]	2.744	H(11)	C(4)[3)]	3.289
H(11)	C(18)[7)]	3.271	H(11)	H(4)[3)]	2.382
H(11)	H(7)[3)]	2.981	H(11)	H(18)[7)]	3.503
H(11)	H(19)[7)]	2.530	H(11)	H(20)[7)]	3.353
H(12)	C(4)[3)]	3.404	H(12)	C(20)[8)]	3.361

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(12)	C(21)[8)]	3.171	H(12)	H(4)[3)]	3.087
H(12)	H(5)[3)]	3.504	H(12)	H(18)[7)]	3.430
H(12)	H(19)[7)]	3.496	H(12)	H(23)[8)]	2.778
H(12)	H(24)[8)]	3.205	H(12)	H(25)[8)]	2.731
H(12)	H(26)[8)]	2.804	H(12)	H(28)[9)]	3.025
H(12)	H(29)[9)]	3.241	H(13)	C(20)[8)]	3.247
H(13)	C(24)[9)]	3.391	H(13)	H(23)[8)]	2.966
H(13)	H(24)[8)]	2.678	H(13)	H(28)[9)]	2.526
H(14)	C(25)[1)]	3.391	H(14)	C(26)[1)]	3.331
H(14)	H(29)[1)]	2.935	H(14)	H(30)[1)]	2.823
H(15)	C(20)[3)]	3.544	H(15)	C(21)[1)]	3.439
H(15)	C(24)[1)]	3.470	H(15)	C(25)[1)]	2.938
H(15)	C(26)[1)]	3.376	H(15)	H(22)[3)]	3.496
H(15)	H(24)[3)]	2.790	H(15)	H(25)[1)]	3.379
H(15)	H(27)[1)]	2.779	H(15)	H(29)[1)]	2.808
H(15)	H(30)[1)]	3.582	H(16)	C(8)[3)]	3.568
H(16)	C(20)[3)]	3.200	H(16)	H(4)[3)]	3.586
H(16)	H(7)[3)]	2.600	H(16)	H(22)[3)]	2.742
H(16)	H(24)[3)]	2.801	H(16)	H(31)[7)]	3.576
H(17)	C(10)[3)]	3.292	H(17)	C(11)[3)]	3.298
H(17)	C(12)[3)]	3.263	H(17)	C(13)[3)]	3.225
H(17)	C(14)[3)]	3.246	H(17)	C(15)[3)]	3.298
H(17)	H(7)[3)]	2.933	H(17)	H(17)[3)]	3.347
H(17)	H(22)[3)]	3.514	H(17)	H(24)[3)]	3.302
H(17)	H(29)[1)]	3.088	H(18)	C(21)[1)]	2.858
H(18)	H(4)[10)]	3.337	H(18)	H(5)[10)]	2.780
H(18)	H(11)[7)]	3.503	H(18)	H(12)[7)]	3.430
H(18)	H(25)[1)]	2.852	H(18)	H(26)[1)]	2.459
H(18)	H(27)[1)]	2.788	H(18)	H(30)[1)]	3.403
H(19)	C(12)[7)]	3.338	H(19)	H(4)[10)]	3.106
H(19)	H(11)[7)]	2.530	H(19)	H(12)[7)]	3.496
H(19)	H(19)[7)]	3.141	H(19)	H(25)[1)]	3.580
H(20)	C(4)[10)]	3.367	H(20)	C(5)[10)]	3.405
H(20)	H(4)[10)]	2.692	H(20)	H(5)[10)]	2.788
H(20)	H(11)[7)]	3.353	H(22)	C(17)[3)]	3.412
H(22)	C(28)[4)]	3.543	H(22)	H(15)[3)]	3.496
H(22)	H(16)[3)]	2.742	H(22)	H(17)[3)]	3.514

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(22)	H(28)[4)]	3.148	H(22)	H(31)[4)]	2.674
H(23)	C(13)[9)]	3.594	H(23)	C(24)[4)]	3.125
H(23)	H(12)[9)]	2.778	H(23)	H(13)[9)]	2.966
H(23)	H(28)[4)]	2.217	H(23)	H(31)[4)]	3.021
H(24)	C(14)[9)]	3.393	H(24)	C(17)[3)]	3.128
H(24)	H(12)[9)]	3.205	H(24)	H(13)[9)]	2.678
H(24)	H(15)[3)]	2.790	H(24)	H(16)[3)]	2.801
H(24)	H(17)[3)]	3.302	H(24)	H(28)[4)]	3.153
H(24)	H(31)[4)]	3.412	H(25)	C(13)[9)]	3.420
H(25)	H(12)[9)]	2.731	H(25)	H(15)[11)]	3.379
H(25)	H(18)[11)]	2.852	H(25)	H(19)[11)]	3.580
H(26)	C(18)[11)]	3.381	H(26)	H(5)[12)]	2.939
H(26)	H(12)[9)]	2.804	H(26)	H(18)[11)]	2.459
H(27)	H(15)[11)]	2.779	H(27)	H(18)[11)]	2.788
H(28)	C(13)[8)]	3.474	H(28)	C(14)[8)]	3.232
H(28)	C(20)[10)]	2.969	H(28)	H(12)[8)]	3.025
H(28)	H(13)[8)]	2.526	H(28)	H(22)[10)]	3.148
H(28)	H(23)[10)]	2.217	H(28)	H(24)[10)]	3.153
H(28)	H(41)[10)]	3.486	H(29)	C(2)[11)]	3.033
H(29)	C(3)[11)]	2.973	H(29)	C(4)[11)]	3.179
H(29)	C(5)[11)]	3.413	H(29)	C(6)[11)]	3.391
H(29)	C(7)[11)]	3.214	H(29)	C(8)[11)]	3.501
H(29)	C(13)[8)]	3.581	H(29)	C(17)[11)]	3.271
H(29)	H(12)[8)]	3.241	H(29)	H(14)[11)]	2.935
H(29)	H(15)[11)]	2.808	H(29)	H(17)[11)]	3.088
H(30)	O(1)[11)]	2.801	H(30)	C(2)[11)]	3.015
H(30)	C(7)[11)]	3.240	H(30)	C(9)[11)]	3.239
H(30)	H(3)[11)]	3.467	H(30)	H(8)[11)]	2.848
H(30)	H(10)[11)]	3.177	H(30)	H(14)[11)]	2.823
H(30)	H(15)[11)]	3.582	H(30)	H(18)[11)]	3.403
H(31)	C(20)[10)]	3.194	H(31)	H(4)[10)]	3.441
H(31)	H(16)[7)]	3.576	H(31)	H(22)[10)]	2.674
H(31)	H(23)[10)]	3.021	H(31)	H(24)[10)]	3.412
H(31)	H(41)[10)]	3.420	H(32)	H(4)[10)]	3.210
H(32)	H(40)[10)]	3.476	H(32)	H(41)[10)]	3.106
H(35)	H(5)[12)]	3.400	H(35)	H(46)[13)]	2.914
H(36)	H(5)[12)]	3.228	H(36)	H(6)[12)]	3.499

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(37)	H(1)$^{4)}$	2.783	H(37)	H(9)$^{5)}$	3.354
H(37)	H(49)$^{5)}$	3.027	H(37)	H(50)$^{5)}$	3.318
H(38)	H(1)$^{4)}$	3.418	H(38)	H(48)$^{5)}$	3.191
H(38)	H(49)$^{5)}$	3.405	H(39)	C(37)$^{5)}$	3.497
H(39)	H(9)$^{5)}$	2.887	H(39)	H(39)$^{5)}$	3.214
H(39)	H(48)$^{5)}$	3.418	H(39)	H(49)$^{5)}$	3.246
H(39)	H(50)$^{5)}$	2.872	H(40)	H(32)$^{4)}$	3.476
H(41)	C(23)$^{4)}$	3.311	H(41)	C(24)$^{4)}$	3.398
H(41)	C(28)$^{4)}$	3.459	H(41)	H(28)$^{4)}$	3.486
H(41)	H(31)$^{4)}$	3.420	H(41)	H(32)$^{4)}$	3.106
H(43)	C(9)$^{12)}$	3.561	H(43)	H(6)$^{12)}$	2.913
H(43)	H(10)$^{12)}$	2.744	H(44)	C(34)$^{13)}$	3.511
H(44)	H(9)$^{5)}$	3.100	H(44)	H(44)$^{13)}$	2.985
H(44)	H(45)$^{13)}$	3.349	H(44)	H(47)$^{13)}$	3.356
H(45)	C(36)$^{13)}$	3.505	H(45)	H(6)$^{12)}$	2.671
H(45)	H(44)$^{13)}$	3.349	H(45)	H(46)$^{13)}$	3.340
H(45)	H(47)$^{13)}$	2.849	H(46)	C(6)$^{5)}$	3.548
H(46)	H(2)$^{2)}$	3.293	H(46)	H(6)$^{5)}$	2.820
H(46)	H(35)$^{13)}$	2.914	H(46)	H(45)$^{13)}$	3.340
H(47)	C(34)$^{13)}$	3.478	H(47)	H(6)$^{5)}$	2.934
H(47)	H(9)$^{5)}$	3.367	H(47)	H(44)$^{13)}$	3.356
H(47)	H(45)$^{13)}$	2.849	H(47)	H(47)$^{13)}$	3.586
H(48)	C(6)$^{5)}$	3.218	H(48)	H(6)$^{5)}$	2.754
H(48)	H(9)$^{5)}$	3.014	H(48)	H(38)$^{5)}$	3.191
H(48)	H(39)$^{5)}$	3.418	H(49)	C(1)$^{2)}$	3.165
H(49)	C(32)$^{5)}$	3.410	H(49)	H(1)$^{2)}$	2.912
H(49)	H(2)$^{2)}$	2.586	H(49)	H(37)$^{5)}$	3.027
H(49)	H(38)$^{5)}$	3.405	H(49)	H(39)$^{5)}$	3.246
H(49)	H(51)$^{2)}$	3.184	H(49)	H(52)$^{2)}$	3.332
H(49)	H(54)$^{2)}$	3.353	H(50)	C(32)$^{5)}$	3.448
H(50)	H(37)$^{5)}$	3.318	H(50)	H(39)$^{5)}$	2.872
H(51)	C(37)$^{2)}$	3.461	H(51)	C(38)$^{2)}$	3.492
H(51)	H(2)$^{2)}$	3.599	H(51)	H(49)$^{2)}$	3.184
H(51)	H(51)$^{2)}$	2.915	H(51)	H(52)$^{2)}$	3.430
H(51)	H(54)$^{2)}$	2.763	H(52)	H(49)$^{2)}$	3.332
H(52)	H(51)$^{2)}$	3.430	H(53)	H(53)$^{13)}$	3.584
H(54)	C(37)$^{2)}$	3.435	H(54)	H(2)$^{2)}$	3.112

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(54)	H(8)[2)]	2.958	H(54)	H(49)[2)]	3.353
H(54)	H(51)[2)]	2.763			

Symmetry Operators:

(1) X,-Y,Z+1/2-1
(2) -X,-Y,-Z+1
(3) -X+1/2,-Y+1/2,-Z+1
(4) X,Y+1,Z
(5) -X,-Y+1,-Z+1
(6) X,-Y+1,Z+1/2-1
(7) -X+1/2,-Y+1/2-1,-Z+1
(8) -X+1/2,Y+1/2-1,-Z+1/2+1
(9) -X+1/2,Y+1/2,-Z+1/2+1
(10) X,Y-1,Z
(11) X,-Y,Z+1/2
(12) X,-Y+1,Z+1/2
(13) -X,Y,-Z+1/2+1