Supporting Information for

# Synthesis of (Arylimido)vanadium(V) Complexes Containing (2-Anilidomethyl)pyridine Ligands and their Use as the Catalyst Precursors for Olefin Polymerization

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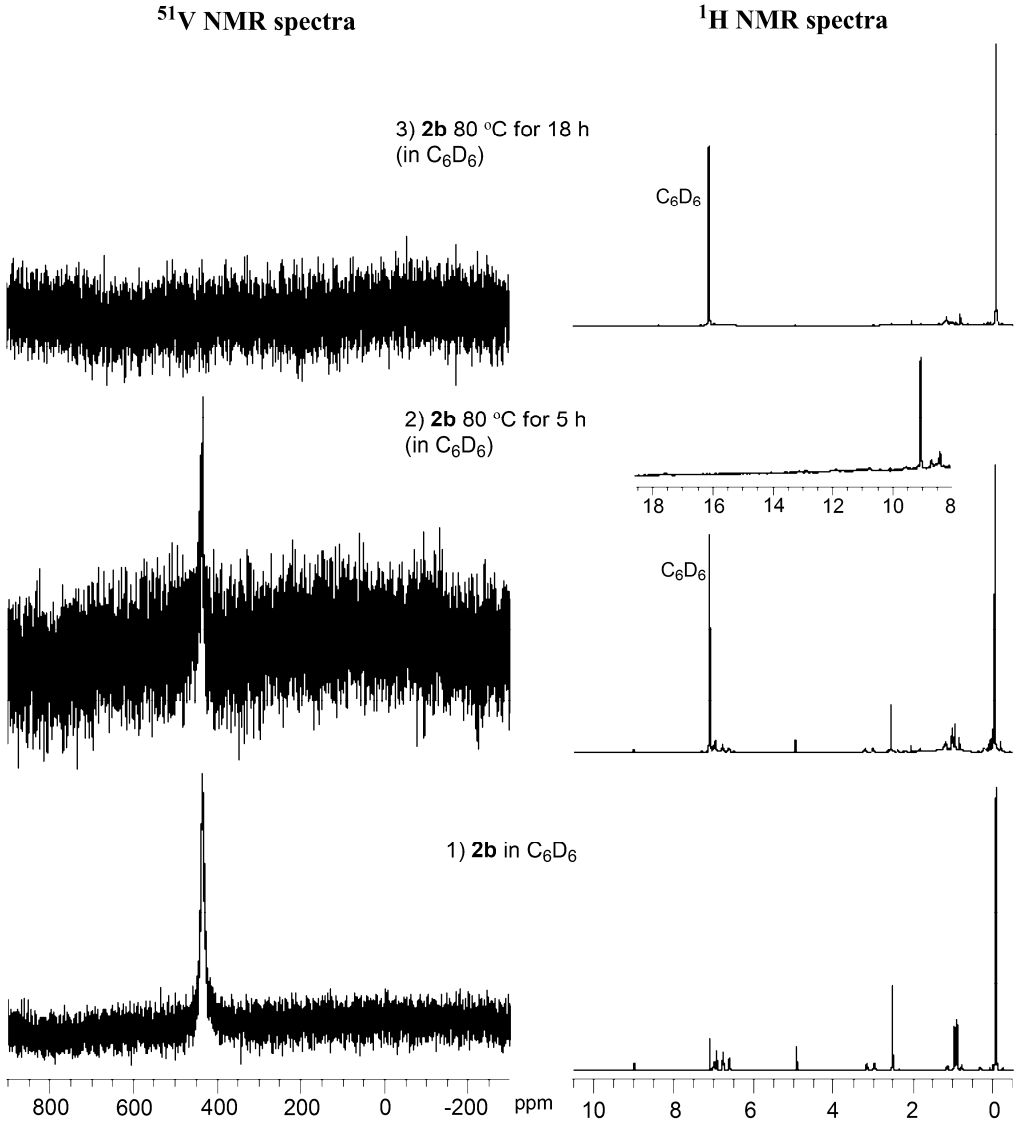
## 1. $^{1}$H and $^{51}$V NMR spectra for monitoring reactions of V(N-2,6-$Me_2C_6H_3$)($CH_2SiMe_3$)$_2$-[2-(2,6-$^{i}Pr_2C_6H_3$)$NCH_2(C_5H_3N)$)] (2b) in $C_6D_6$ at 80 ºC in the presence/absence of $PMe_3$.

### 1-1. Reaction (thermolysis) of 2b in $C_6D_6$ at 80 ºC

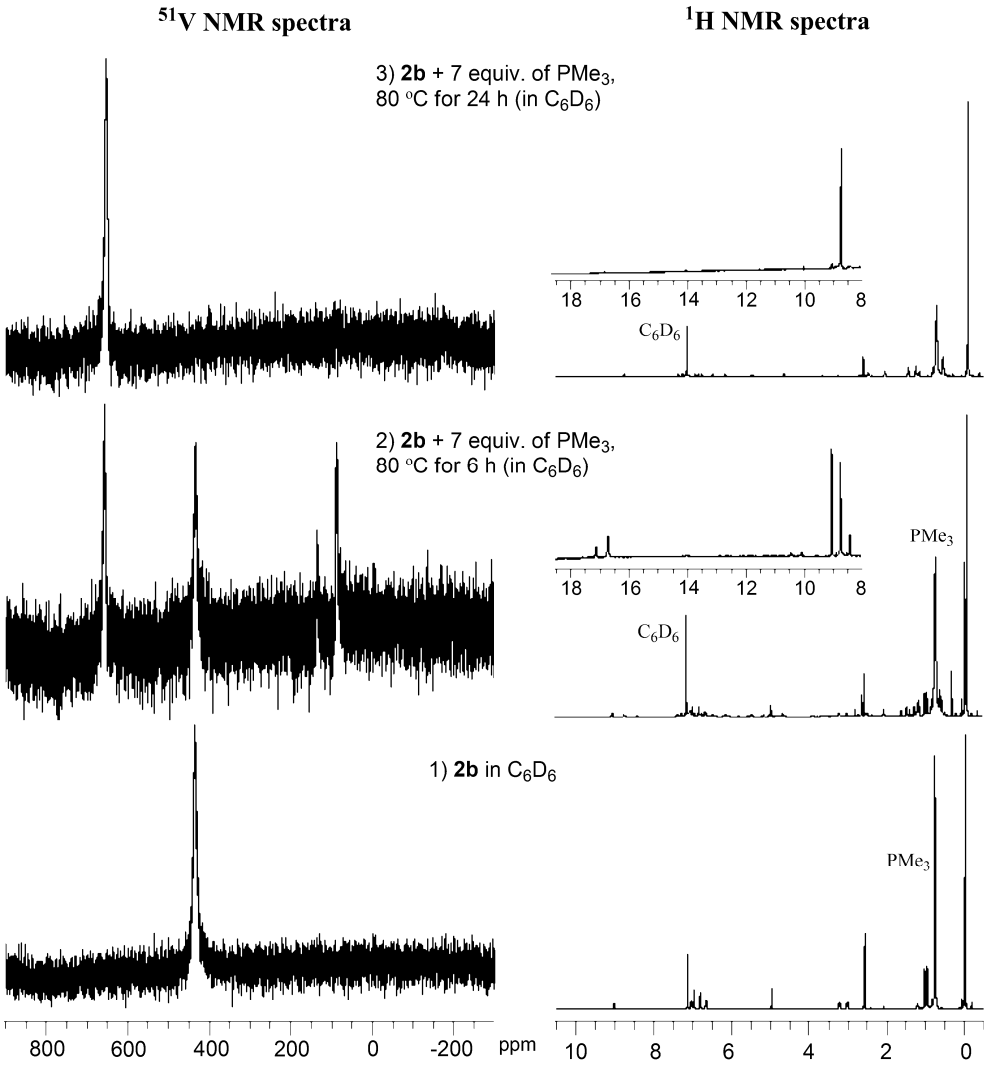
Procedure: The $C_6D_6$ solution (0.5 mL) containing **2b** (31 mg) was heated at 80 ºC, and the reaction was monitored by both $^{1}$H and $^{51}$V NMR spectra.




Additional note: No resonances in the $^{51}$V NMR spectrum were seen after 18 h, probably suggesting that the reaction may give certain paramagnetic species [vanadium(III) or vanadium(IV)].

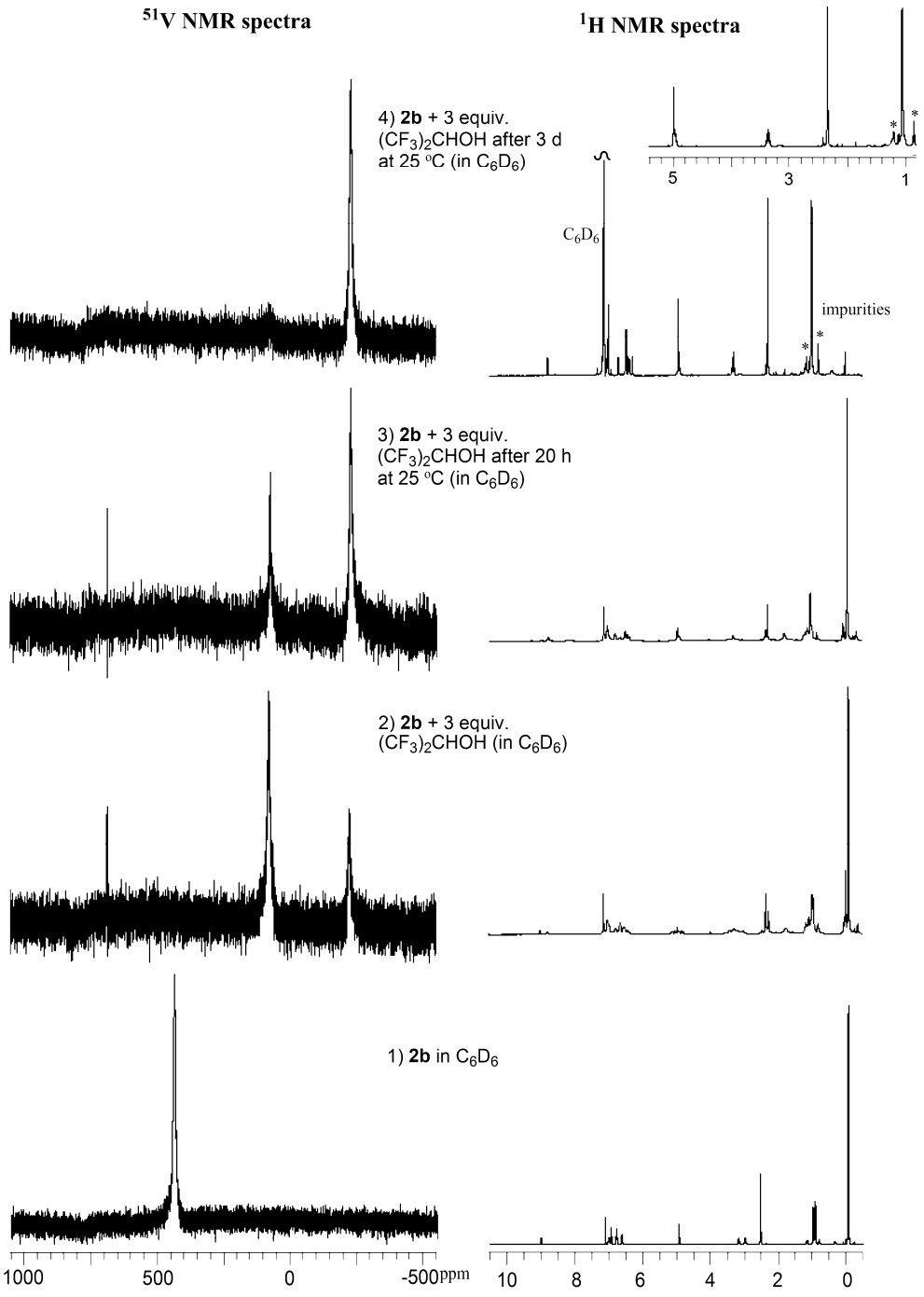
## 1-2. Reaction of 2b in $C_6D_6$ in the presence of $PMe_3$ (7 equiv.).

Procedure: The $C_6D_6$ solution (0.5 mL) containing complex **2b** (31 mg) and $PMe_3$ (27 mg, 7 equiv.) was heated at 80 ºC, and the reaction was monitored by $^1H$ and $^{51}V$ NMR spectra.

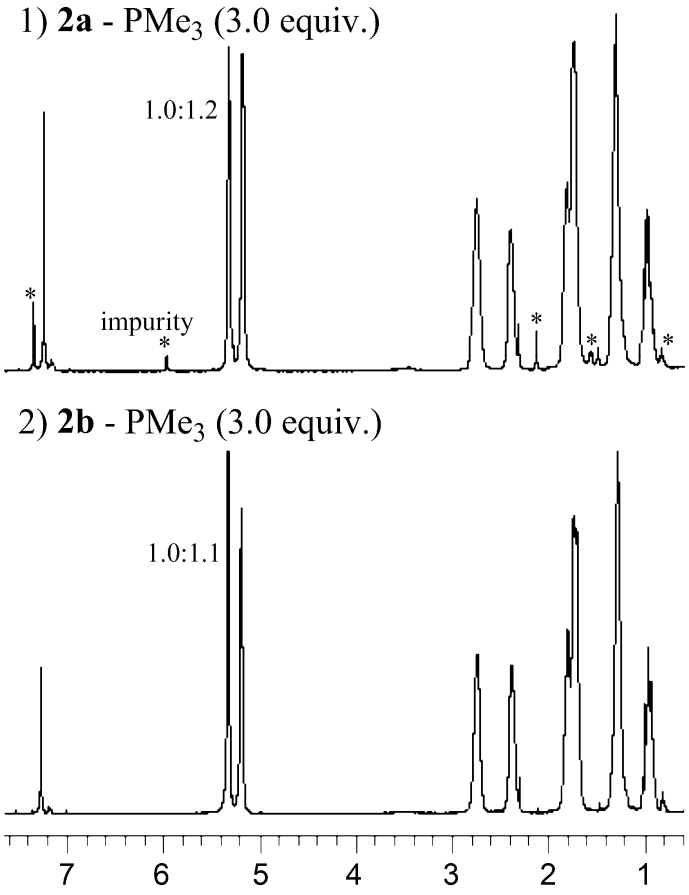



**Additional note**: Resonances seen at δ 16.7 and 17.1 ppm (after 6 h, in the $^1H$ NMR spectrum) presumably ascribed to the protons of the vanadium(V)-alkylidene(s) disappeared after 24 h to afford another (unidentified) species. For comparison, δ 14.52 ppm in V(C*H*SiMe$_3$)(NAr)(N=C$^t$Bu$_2$)(PMe$_3$) (reference 14a,c), δ 16.09 and 16.38 ppm in V(C*H*SiMe$_3$)(NAr)(O-2,6-Me$_2$C$_6$H$_3$)(PMe$_3$)$_{0.89}$ (reference 14b). Although only one resonance was seen in the $^{51}V$ NMR spectrum after 24 h, the isolation from the mixture was not successful (due to a difficulty to isolate as microcrystals from the mixture probably containing certain amount of decomposed and/or paramagnetic products.)

**2. $^{1}$H and $^{51}$V NMR spectra for monitoring reactions of 2b with 3 equiv. of $(CF_3)_2CHOH$ in $C_6D_6$ at 25 ºC.** Experimental details are shown in the Experimental Section.



**3. $^{1}$H NMR spectra in $CDCl_3$ for ring-opened poly(norbornene) prepared by 2b in the presence of $PMe_3$ (Table 4).**




**Additional note**: The spectra clearly indicates that the resultant polymer possessed ring-opened structure containing a mixture of *cis*/*trans* olefinic double bonds.

**4) Crystal data and collection parameters of complexes V(N-2,6-$Me_2C_6H_3$)$Cl_2$[2-(2,6-$R_2C_6H_3$)$NCH_2(C_5H_3N)$] [R = $^i$Pr (1b), F (1c)] and V(N-2,6-$Me_2C_6H_3$)$(CH_2SiMe_3)_2$[2-(2,6-$R_2C_6H_3$)$NCH_2(C_5H_3N)$] [R = Me (2a), $^i$Pr (2b)].**

| | 1b | 1c | 2a | 2b |
|---|---|---|---|---|
| Formula | $C_{26}H_{32}Cl_2N_3V$ | $C_{23}H_{18}Cl_2F_2N_3V$ | $C_{30}H_{46}N_3Si_2V$ | $C_{34}H_{54}N_3Si_2V$ |
| Formula Weight | 508.41 | 496.26 | 555.83 | 611.93 |
| Crystal color, Habit | red, block | red, block | red, block | red, block |
| Crystal size (mm) | 0.25 × 0.25 × 0.10 | 0.40 × 0.30 × 0.20 | 0.50 × 0.40 × 0.40 | 0.20 × 0.15 × 0.10 |
| Crystal system | monoclinic | triclinic | monoclinic | triclinic |
| Space group | $P2_1/n$ (#14) | $P$-1 (#2) | $P2_1/c$ (#14) | $P$-1 (#2) |
| a (Å) | 11.3290(7) | 7.3016(4) | 15.9173(4) | 10.1893(9) |
| b (Å) | 16.2926(7) | 10.2681(6) | 11.9182(3) | 11.0729(9) |
| c (Å) | 14.8872(8) | 15.9162(12) | 17.7030(4) | 16.2502(16) |
| α (deg) | | 99.261(3) | | 97.395(3) |
| β (deg) | 106.8908(18) | 92.447(3) | 103.6830(7) | 95.290(3) |
| γ (deg) | | 97.1083(15) | | 96.446(2) |
| V ($Å^3$) | 2629.3(2) | 1166.29(12) | 3263.05(13) | 1796.1(3) |
| Z value | 4 | 2 | 4 | 2 |
| $D_{calcd}$ ($g/cm^3$) | 1.284 | 1.413 | 1.131 | 1.131 |
| F000 | 1064.00 | 504.00 | 1192.00 | 660.00 |
| Temp (K) | 193 | 193 | 193 | 153 |
| μ (MoKα) ($cm^{-1}$) | 5.980 | 6.843 | 3.985 | 3.677 |
| No. of reflections measured | Total: 25405<br>Unique: 5988 | Total: 9482<br>Unique: 4207 | Total: 26522<br>Unique: 5936 | Total: 13412<br>Unique: 5866 |
| No. of observations (I > 2.00σ(I)) | 3126 | 4207 | 5233 | 5866 |
| No. of Variables | 321 | 281 | 371 | 362 |
| R1( I > 2.00σ(I)) | 0.0398 | 0.0404 | 0.0372 | 0.0637 |
| wR2 (I > 2.00σ(I)) | 0.0954 | 0.1316 | 0.1323 | 0.2134 |
| Goodness of Fit | 1.003 | 1.154 | 1.008 | 1.181 |

X-ray Structure Report for V(N=2,6-$Me_2C_6H_3$)$Cl_2$[2-(2,6-$^{i}Pr_2C_6H_3$)$CH_2$($C_5H_4N$)] (**1b**)

July 14, 2009

*Experimental*

Data Collection

A red block crystal of $C_{26}H_{32}Cl_2N_3V$ having approximate dimensions of 0.25 x 0.25 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K$\alpha$ radiation.

Indexing was performed from 3 oscillations that were exposed for 420 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 = 11.3290(7) Å
b = 16.2926(7) Å $\beta$ = 106.8908(18)$^o$
c = 14.8872(8) Å
V = 2629.3(2) Å$^3$

For Z = 4 and F.W. = 508.41, the calculated density is 1.284 g/cm$^3$. The systematic absences of:

h0l: h+l ± 2n
0k0: k ± 2n

uniquely determine the space group to be:

$P2_1/n$ (#14)

The data were collected at a temperature of -80 ± 1$^o$C to a maximum 2$\theta$ value of 54.9$^o$. A total of 55 oscillation images were collected. A sweep of data was done using $\omega$ scans from 130.0 to 190.0$^o$ in 4.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 0.0$^o$. The exposure rate was 240.0 [sec./$^o$]. A second sweep was performed using $\omega$ scans from 0.0 to 160.0$^o$ in 4.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 180.0$^o$. The exposure rate was 240.0 [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 25405 reflections that were collected, 5988 were unique ($R_{int}$ = 0.062); equivalent reflections were merged.

The linear absorption coefficient, $\mu$, for Mo-K$\alpha$ radiation is 5.980 $cm^{-1}$. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.568 to 0.942. 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 3126 observed reflections and 321 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0398$$

$$wR2 = [ \Sigma \, ( w \, (Fo^2 - Fc^2)^2 ) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.0954$$

The standard deviation of an observation of unit weight[4] was 1.00. A Sheldrick weighting scheme was used. Plots of $\Sigma$ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin $\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.70 and -0.27 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta$f' and $\Delta$f'' 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 | $C_{26}H_{32}Cl_2N_3V$ |
| Formula Weight | 508.41 |
| Crystal Color, Habit | red, block |
| Crystal Dimensions | 0.25 X 0.25 X 0.10 mm |
| Crystal System | monoclinic |
| Lattice Type | Primitive |
| Indexing Images | 3 oscillations @ 420.0 seconds |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| Lattice Parameters | a = 11.3290(7) Å<br>b = 16.2926(7) Å<br>c = 14.8872(8) Å<br>$\beta$ = 106.8908(18) $^{o}$<br>V = 2629.3(2) Å$^3$ |
| Space Group | $P2_1/n$ (#14) |
| Z value | 4 |
| $D_{calc}$ | 1.284 g/cm$^3$ |
| $F_{000}$ | 1064.00 |
| $\mu$(MoK$\alpha$) | 5.980 cm$^{-1}$ |

## B. Intensity Measurements

| | |
|---|---|
| Diffractometer | Rigaku RAXIS-RAPID |
| Radiation | MoK$\alpha$ ($\lambda$ = 0.71075 Å)<br>graphite monochromated |
| Detector Aperture | 280 mm x 256 mm |
| Data Images | 55 exposures |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=0.0) | 130.0 - 190.0$^{o}$ |
| Exposure Rate | 240.0 sec./$^{o}$ |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=180.0) | 0.0 - 160.0$^{o}$ |
| Exposure Rate | 240.0 sec./$^{o}$ |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| $2\theta_{max}$ | 54.9$^{o}$ |
| No. of Reflections Measured | Total: 25405<br>Unique: 5988 ($R_{int}$ = 0.062) |
| Corrections | Lorentz-polarization<br>Absorption<br>(trans. factors: 0.568 - 0.942) |

C. Structure Solution and Refinement

| | |
|---|---|
| Structure Solution | Direct Methods (SIR92) |
| Refinement | Full-matrix least-squares on $F^2$ |
| Function Minimized | $\Sigma$ w $(Fo^2 - Fc^2)^2$ |
| Least Squares Weights | $1/[0.0008Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$ |
| $2\theta_{max}$ cutoff | $54.9^o$ |
| Anomalous Dispersion | All non-hydrogen atoms |
| No. Observations (I>2.00$\sigma$(I)) | 3126 |
| No. Variables | 321 |
| Reflection/Parameter Ratio | 9.74 |
| Residuals: R1 (I>2.00$\sigma$(I)) | 0.0398 |
| Residuals: wR2 (I>2.00$\sigma$(I)) | 0.0954 |
| Goodness of Fit Indicator | 1.003 |
| Max Shift/Error in Final Cycle | 0.000 |
| Maximum peak in Final Diff. Map | 0.70 $e^-/Å^3$ |
| Minimum peak in Final Diff. Map | -0.27 $e^-/Å^3$ |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| V(1) | 0.52187(5) | 0.21060(3) | 0.71452(3) | 2.464(12) |
| Cl(1) | 0.38770(10) | 0.10409(5) | 0.68731(6) | 4.38(2) |
| Cl(2) | 0.72897(8) | 0.19223(5) | 0.78155(6) | 4.12(2) |
| N(1) | 0.4744(2) | 0.29724(13) | 0.63156(16) | 2.29(5) |
| N(2) | 0.5608(2) | 0.16298(14) | 0.58699(16) | 2.52(5) |
| N(3) | 0.4926(2) | 0.25352(14) | 0.80834(16) | 2.60(5) |
| C(1) | 0.4497(3) | 0.28513(17) | 0.52960(19) | 2.74(6) |
| C(2) | 0.5066(2) | 0.20718(17) | 0.5105(2) | 2.32(6) |
| C(3) | 0.5023(3) | 0.18079(19) | 0.4210(2) | 2.95(7) |
| C(4) | 0.5570(3) | 0.1063(2) | 0.4115(2) | 3.33(8) |
| C(5) | 0.6160(3) | 0.06240(19) | 0.4903(2) | 3.45(8) |
| C(6) | 0.6168(3) | 0.09144(18) | 0.5763(2) | 3.12(7) |
| C(7) | 0.4608(2) | 0.38220(17) | 0.65577(18) | 2.30(6) |
| C(8) | 0.5618(2) | 0.43534(17) | 0.66305(19) | 2.57(6) |
| C(9) | 0.5517(3) | 0.51566(19) | 0.6937(2) | 3.44(7) |
| C(10) | 0.4474(3) | 0.5413(2) | 0.7150(2) | 3.86(8) |
| C(11) | 0.3475(3) | 0.4894(2) | 0.7030(2) | 3.63(8) |
| C(12) | 0.3515(3) | 0.40864(18) | 0.6720(2) | 2.86(7) |
| C(13) | 0.6762(2) | 0.41004(19) | 0.6363(2) | 2.98(7) |
| C(14) | 0.7947(3) | 0.4224(2) | 0.7147(2) | 4.55(9) |
| C(15) | 0.6848(3) | 0.4569(2) | 0.5498(2) | 4.94(10) |
| C(16) | 0.2372(3) | 0.3551(2) | 0.6505(2) | 3.51(8) |
| C(17) | 0.1659(3) | 0.3635(2) | 0.7213(2) | 5.24(11) |
| C(18) | 0.1501(3) | 0.3753(2) | 0.5523(2) | 4.62(9) |
| C(19) | 0.4720(3) | 0.28444(18) | 0.8893(2) | 2.76(7) |
| C(20) | 0.5513(3) | 0.3462(2) | 0.9415(2) | 3.43(8) |
| C(21) | 0.5279(4) | 0.3753(2) | 1.0229(2) | 4.93(10) |
| C(22) | 0.4328(4) | 0.3456(2) | 1.0521(2) | 5.41(11) |
| C(23) | 0.3564(3) | 0.2843(2) | 1.0020(2) | 4.30(9) |
| C(24) | 0.3749(3) | 0.2528(2) | 0.9203(2) | 3.20(7) |
| C(25) | 0.6558(3) | 0.3783(2) | 0.9107(2) | 4.69(9) |
| C(26) | 0.2940(3) | 0.1857(2) | 0.8679(2) | 4.45(9) |

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

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| H(1) | 0.4835 | 0.3297 | 0.5042 | 3.21 |
| H(2) | 0.3631 | 0.2829 | 0.5009 | 3.20 |
| H(3) | 0.4633 | 0.2175 | 0.3618 | 3.52 |
| H(4) | 0.5486 | 0.0872 | 0.3413 | 4.13 |
| H(5) | 0.6527 | 0.0073 | 0.4893 | 4.24 |
| H(6) | 0.6522 | 0.0562 | 0.6442 | 3.87 |
| H(7) | 0.6267 | 0.5587 | 0.7012 | 3.82 |
| H(8) | 0.4399 | 0.6013 | 0.7425 | 4.36 |
| H(9) | 0.2705 | 0.5069 | 0.7309 | 4.36 |
| H(10) | 0.6614 | 0.3465 | 0.6160 | 3.53 |
| H(11) | 0.8080 | 0.4794 | 0.7269 | 5.38 |
| H(12) | 0.8618 | 0.4002 | 0.6965 | 5.38 |
| H(13) | 0.7887 | 0.3955 | 0.7697 | 5.37 |
| H(14) | 0.7527 | 0.4368 | 0.5309 | 6.05 |
| H(15) | 0.6108 | 0.4495 | 0.5002 | 6.06 |
| H(16) | 0.6964 | 0.5137 | 0.5643 | 6.05 |
| H(17) | 0.2665 | 0.2923 | 0.6506 | 4.39 |
| H(18) | 0.1285 | 0.4162 | 0.7155 | 6.54 |
| H(19) | 0.1039 | 0.3224 | 0.7102 | 6.55 |
| H(20) | 0.2205 | 0.3572 | 0.7828 | 6.55 |
| H(21) | 0.1922 | 0.3668 | 0.5064 | 5.56 |
| H(22) | 0.0798 | 0.3406 | 0.5393 | 5.56 |
| H(23) | 0.1247 | 0.4310 | 0.5507 | 5.55 |
| H(24) | 0.5928 | 0.4221 | 1.0641 | 5.81 |
| H(25) | 0.4186 | 0.3671 | 1.1073 | 6.51 |
| H(26) | 0.2749 | 0.2613 | 1.0254 | 5.18 |
| H(27) | 0.6890 | 0.3353 | 0.8823 | 5.56 |
| H(28) | 0.6274 | 0.4213 | 0.8666 | 5.58 |
| H(29) | 0.7179 | 0.3987 | 0.9635 | 5.57 |
| H(30) | 0.3415 | 0.1372 | 0.8704 | 5.62 |
| H(31) | 0.2298 | 0.1754 | 0.8956 | 5.62 |
| H(32) | 0.2592 | 0.2017 | 0.8043 | 5.61 |

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

Table 3. Anisotropic displacement parameters

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| V(1) | 0.0379(3) | 0.0249(2) | 0.0293(2) | -0.0014(2) | 0.0075(2) | -0.0009(2) |
| Cl(1) | 0.0764(7) | 0.0417(4) | 0.0584(5) | -0.0265(4) | 0.0356(5) | -0.0169(4) |
| Cl(2) | 0.0454(5) | 0.0499(5) | 0.0499(5) | 0.0140(4) | -0.0044(4) | -0.0086(4) |
| N(1) | 0.0284(14) | 0.0271(12) | 0.0293(12) | 0.0013(11) | 0.0052(10) | -0.0021(10) |
| N(2) | 0.0343(15) | 0.0266(12) | 0.0347(14) | -0.0004(11) | 0.0098(12) | 0.0002(11) |
| N(3) | 0.0378(16) | 0.0274(12) | 0.0311(13) | -0.0020(11) | 0.0061(12) | 0.0009(11) |
| C(1) | 0.040(2) | 0.0331(16) | 0.0287(15) | -0.0006(15) | 0.0057(14) | -0.0027(13) |
| C(2) | 0.0274(17) | 0.0306(15) | 0.0305(16) | -0.0012(14) | 0.0086(13) | -0.0023(13) |
| C(3) | 0.040(2) | 0.0405(17) | 0.0318(17) | -0.0023(15) | 0.0098(15) | -0.0028(14) |
| C(4) | 0.046(2) | 0.0452(19) | 0.0394(19) | -0.0032(17) | 0.0186(16) | -0.0127(15) |
| C(5) | 0.050(2) | 0.0354(18) | 0.049(2) | 0.0014(16) | 0.0192(18) | -0.0086(16) |
| C(6) | 0.045(2) | 0.0331(17) | 0.0433(19) | 0.0042(15) | 0.0177(16) | 0.0003(14) |
| C(7) | 0.0355(19) | 0.0267(14) | 0.0228(15) | 0.0053(14) | 0.0045(13) | 0.0007(12) |
| C(8) | 0.0331(19) | 0.0284(15) | 0.0310(16) | 0.0003(14) | 0.0014(14) | 0.0024(13) |
| C(9) | 0.046(2) | 0.0313(17) | 0.0437(19) | -0.0012(16) | -0.0025(16) | -0.0023(14) |
| C(10) | 0.059(2) | 0.0319(17) | 0.047(2) | 0.0081(18) | 0.0006(18) | -0.0111(15) |
| C(11) | 0.053(2) | 0.0422(19) | 0.0422(19) | 0.0123(18) | 0.0137(17) | -0.0041(16) |
| C(12) | 0.039(2) | 0.0370(17) | 0.0325(17) | 0.0058(15) | 0.0105(15) | 0.0012(14) |
| C(13) | 0.0330(19) | 0.0395(18) | 0.0389(18) | -0.0057(15) | 0.0076(15) | -0.0008(14) |
| C(14) | 0.040(2) | 0.076(2) | 0.054(2) | -0.0002(19) | 0.0088(18) | 0.006(2) |
| C(15) | 0.060(2) | 0.077(2) | 0.055(2) | 0.001(2) | 0.023(2) | 0.013(2) |
| C(16) | 0.036(2) | 0.0423(19) | 0.060(2) | 0.0041(16) | 0.0221(17) | 0.0033(16) |
| C(17) | 0.053(2) | 0.080(2) | 0.074(2) | 0.013(2) | 0.032(2) | 0.020(2) |
| C(18) | 0.044(2) | 0.069(2) | 0.063(2) | -0.0143(19) | 0.0164(19) | -0.0063(19) |
| C(19) | 0.041(2) | 0.0352(16) | 0.0262(15) | 0.0043(15) | 0.0055(14) | 0.0010(14) |
| C(20) | 0.044(2) | 0.0487(19) | 0.0350(18) | -0.0021(17) | 0.0069(16) | -0.0058(15) |
| C(21) | 0.058(2) | 0.082(2) | 0.044(2) | -0.006(2) | 0.009(2) | -0.027(2) |
| C(22) | 0.055(2) | 0.109(3) | 0.042(2) | 0.005(2) | 0.015(2) | -0.025(2) |
| C(23) | 0.045(2) | 0.080(2) | 0.0384(19) | 0.004(2) | 0.0129(17) | -0.0090(19) |
| C(24) | 0.040(2) | 0.0450(19) | 0.0353(18) | 0.0014(16) | 0.0088(15) | 0.0039(15) |
| C(25) | 0.067(2) | 0.061(2) | 0.048(2) | -0.022(2) | 0.012(2) | -0.0202(18) |
| C(26) | 0.063(2) | 0.058(2) | 0.057(2) | -0.014(2) | 0.031(2) | -0.0035(18) |

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) | 2.2645(10) | V(1) | Cl(2) | 2.2868(9) |
| V(1) | N(1) | 1.850(2) | V(1) | N(2) | 2.211(2) |
| V(1) | N(3) | 1.679(2) | N(1) | C(1) | 1.475(3) |
| N(1) | C(7) | 1.450(3) | N(2) | C(2) | 1.336(3) |
| N(2) | C(6) | 1.358(4) | N(3) | C(19) | 1.387(4) |
| C(1) | C(2) | 1.488(4) | C(2) | C(3) | 1.387(4) |
| C(3) | C(4) | 1.389(4) | C(4) | C(5) | 1.371(4) |
| C(5) | C(6) | 1.362(4) | C(7) | C(8) | 1.414(4) |
| C(7) | C(12) | 1.397(4) | C(8) | C(9) | 1.401(4) |
| C(8) | C(13) | 1.519(4) | C(9) | C(10) | 1.374(5) |
| C(10) | C(11) | 1.382(5) | C(11) | C(12) | 1.400(4) |
| C(12) | C(16) | 1.517(4) | C(13) | C(14) | 1.515(4) |
| C(13) | C(15) | 1.525(5) | C(16) | C(17) | 1.509(6) |
| C(16) | C(18) | 1.543(4) | C(19) | C(20) | 1.421(4) |
| C(19) | C(24) | 1.409(5) | C(20) | C(21) | 1.397(5) |
| C(20) | C(25) | 1.483(5) | C(21) | C(22) | 1.362(6) |
| C(22) | C(23) | 1.389(5) | C(23) | C(24) | 1.390(5) |
| C(24) | C(26) | 1.494(4) | | | |

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(3) | H(3) | 1.050 | C(4) | H(4) | 1.067 |
| C(5) | H(5) | 0.992 | C(6) | H(6) | 1.131 |
| C(9) | H(7) | 1.082 | C(10) | H(8) | 1.073 |
| C(11) | H(9) | 1.106 | C(13) | H(10) | 1.078 |
| C(14) | H(11) | 0.950 | C(14) | H(12) | 0.950 |
| C(14) | H(13) | 0.950 | C(15) | H(14) | 0.950 |
| C(15) | H(15) | 0.950 | C(15) | H(16) | 0.950 |
| C(16) | H(17) | 1.075 | C(17) | H(18) | 0.950 |
| C(17) | H(19) | 0.950 | C(17) | H(20) | 0.950 |
| C(18) | H(21) | 0.950 | C(18) | H(22) | 0.950 |
| C(18) | H(23) | 0.950 | C(21) | H(24) | 1.112 |
| C(22) | H(25) | 0.950 | C(23) | H(26) | 1.141 |
| C(25) | H(27) | 0.950 | C(25) | H(28) | 0.950 |
| C(25) | H(29) | 0.950 | C(26) | H(30) | 0.950 |
| C(26) | H(31) | 0.950 | C(26) | H(32) | 0.950 |

Table 6. Bond angles (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| Cl(1) | V(1) | Cl(2) | 121.87(3) | Cl(1) | V(1) | N(1) | 114.55(7) |
| Cl(1) | V(1) | N(2) | 82.45(7) | Cl(1) | V(1) | N(3) | 100.56(9) |
| Cl(2) | V(1) | N(1) | 116.98(8) | Cl(2) | V(1) | N(2) | 83.87(6) |
| Cl(2) | V(1) | N(3) | 96.74(8) | N(1) | V(1) | N(2) | 77.66(9) |
| N(1) | V(1) | N(3) | 98.52(11) | N(2) | V(1) | N(3) | 175.93(10) |
| V(1) | N(1) | C(1) | 121.10(17) | V(1) | N(1) | C(7) | 126.44(17) |
| C(1) | N(1) | C(7) | 112.4(2) | V(1) | N(2) | C(2) | 112.7(2) |
| V(1) | N(2) | C(6) | 128.2(2) | C(2) | N(2) | C(6) | 118.5(2) |
| V(1) | N(3) | C(19) | 176.2(2) | N(1) | C(1) | C(2) | 110.3(2) |
| N(2) | C(2) | C(1) | 114.5(2) | N(2) | C(2) | C(3) | 122.0(2) |
| C(1) | C(2) | C(3) | 123.5(2) | C(2) | C(3) | C(4) | 118.5(2) |
| C(3) | C(4) | C(5) | 119.3(3) | C(4) | C(5) | C(6) | 119.5(3) |
| N(2) | C(6) | C(5) | 122.1(2) | N(1) | C(7) | C(8) | 117.5(2) |
| N(1) | C(7) | C(12) | 120.1(2) | C(8) | C(7) | C(12) | 122.4(2) |
| C(7) | C(8) | C(9) | 117.1(3) | C(7) | C(8) | C(13) | 123.1(2) |
| C(9) | C(8) | C(13) | 119.7(2) | C(8) | C(9) | C(10) | 121.1(3) |
| C(9) | C(10) | C(11) | 120.7(3) | C(10) | C(11) | C(12) | 120.9(3) |
| C(7) | C(12) | C(11) | 117.6(3) | C(7) | C(12) | C(16) | 122.1(2) |
| C(11) | C(12) | C(16) | 120.2(3) | C(8) | C(13) | C(14) | 113.3(2) |
| C(8) | C(13) | C(15) | 110.8(2) | C(14) | C(13) | C(15) | 108.9(2) |
| C(12) | C(16) | C(17) | 113.4(2) | C(12) | C(16) | C(18) | 110.4(2) |
| C(17) | C(16) | C(18) | 108.5(2) | N(3) | C(19) | C(20) | 119.6(3) |
| N(3) | C(19) | C(24) | 119.7(2) | C(20) | C(19) | C(24) | 120.7(3) |
| C(19) | C(20) | C(21) | 117.7(3) | C(19) | C(20) | C(25) | 121.0(3) |
| C(21) | C(20) | C(25) | 121.3(3) | C(20) | C(21) | C(22) | 121.6(3) |
| C(21) | C(22) | C(23) | 120.8(3) | C(22) | C(23) | C(24) | 120.4(3) |
| C(19) | C(24) | C(23) | 118.8(2) | C(19) | C(24) | C(26) | 121.1(3) |
| C(23) | C(24) | C(26) | 120.1(3) | | | | |

Table 7. Bond angles involving hydrogens (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| N(1) | C(1) | H(1) | 109.3 | N(1) | C(1) | H(2) | 109.3 |
| C(2) | C(1) | H(1) | 109.3 | C(2) | C(1) | H(2) | 109.3 |
| H(1) | C(1) | H(2) | 109.5 | C(2) | C(3) | H(3) | 121.2 |
| C(4) | C(3) | H(3) | 120.2 | C(3) | C(4) | H(4) | 116.1 |
| C(5) | C(4) | H(4) | 124.6 | C(4) | C(5) | H(5) | 124.1 |
| C(6) | C(5) | H(5) | 116.2 | N(2) | C(6) | H(6) | 112.8 |
| C(5) | C(6) | H(6) | 124.6 | C(8) | C(9) | H(7) | 120.2 |
| C(10) | C(9) | H(7) | 118.8 | C(9) | C(10) | H(8) | 122.3 |
| C(11) | C(10) | H(8) | 117.0 | C(10) | C(11) | H(9) | 120.0 |
| C(12) | C(11) | H(9) | 118.0 | C(8) | C(13) | H(10) | 105.0 |
| C(14) | C(13) | H(10) | 112.0 | C(15) | C(13) | H(10) | 106.7 |
| C(13) | C(14) | H(11) | 109.5 | C(13) | C(14) | H(12) | 109.5 |
| C(13) | C(14) | H(13) | 109.5 | H(11) | C(14) | H(12) | 109.5 |
| H(11) | C(14) | H(13) | 109.5 | H(12) | C(14) | H(13) | 109.5 |
| C(13) | C(15) | H(14) | 109.5 | C(13) | C(15) | H(15) | 109.5 |
| C(13) | C(15) | H(16) | 109.5 | H(14) | C(15) | H(15) | 109.5 |
| H(14) | C(15) | H(16) | 109.5 | H(15) | C(15) | H(16) | 109.5 |
| C(12) | C(16) | H(17) | 107.6 | C(17) | C(16) | H(17) | 108.2 |
| C(18) | C(16) | H(17) | 108.5 | C(16) | C(17) | H(18) | 109.5 |
| C(16) | C(17) | H(19) | 109.5 | C(16) | C(17) | H(20) | 109.5 |
| H(18) | C(17) | H(19) | 109.5 | H(18) | C(17) | H(20) | 109.5 |
| H(19) | C(17) | H(20) | 109.5 | C(16) | C(18) | H(21) | 109.5 |
| C(16) | C(18) | H(22) | 109.5 | C(16) | C(18) | H(23) | 109.5 |
| H(21) | C(18) | H(22) | 109.5 | H(21) | C(18) | H(23) | 109.5 |
| H(22) | C(18) | H(23) | 109.5 | C(20) | C(21) | H(24) | 116.7 |
| C(22) | C(21) | H(24) | 121.7 | C(21) | C(22) | H(25) | 119.6 |
| C(23) | C(22) | H(25) | 119.6 | C(22) | C(23) | H(26) | 120.3 |
| C(24) | C(23) | H(26) | 119.2 | C(20) | C(25) | H(27) | 109.5 |
| C(20) | C(25) | H(28) | 109.5 | C(20) | C(25) | H(29) | 109.5 |
| H(27) | C(25) | H(28) | 109.5 | H(27) | C(25) | H(29) | 109.5 |
| H(28) | C(25) | H(29) | 109.5 | C(24) | C(26) | H(30) | 109.5 |
| C(24) | C(26) | H(31) | 109.5 | C(24) | C(26) | H(32) | 109.5 |
| H(30) | C(26) | H(31) | 109.5 | H(30) | C(26) | H(32) | 109.5 |
| H(31) | C(26) | H(32) | 109.5 | | | | |

Table 8. Torsion Angles(°)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| Cl(1) | V(1) | N(1) | C(1) | -57.6(2) |
| Cl(1) | V(1) | N(2) | C(2) | 102.2(2) |
| Cl(1) | V(1) | N(3) | C(19) | 70(3) |
| Cl(2) | V(1) | N(1) | C(7) | -83.3(2) |
| Cl(2) | V(1) | N(2) | C(6) | 54.8(2) |
| N(1) | V(1) | N(2) | C(2) | -15.1(2) |
| N(2) | V(1) | N(1) | C(1) | 18.1(2) |
| N(1) | V(1) | N(3) | C(19) | -173(3) |
| N(3) | V(1) | N(1) | C(7) | 18.8(2) |
| N(3) | V(1) | N(2) | C(2) | -35.7(15) |
| V(1) | N(1) | C(1) | C(2) | -18.5(3) |
| V(1) | N(1) | C(7) | C(12) | -83.6(2) |
| C(1) | N(1) | C(7) | C(12) | 98.3(3) |
| V(1) | N(2) | C(2) | C(1) | 9.3(3) |
| V(1) | N(2) | C(6) | C(5) | 168.1(2) |
| C(6) | N(2) | C(2) | C(1) | -179.0(2) |
| V(1) | N(3) | C(19) | C(20) | 101(3) |
| N(1) | C(1) | C(2) | N(2) | 3.5(3) |
| N(2) | C(2) | C(3) | C(4) | -0.4(4) |
| C(2) | C(3) | C(4) | C(5) | -1.7(5) |
| C(4) | C(5) | C(6) | N(2) | 0.1(4) |
| N(1) | C(7) | C(8) | C(13) | 7.1(3) |
| N(1) | C(7) | C(12) | C(16) | -9.9(3) |
| C(8) | C(7) | C(12) | C(16) | 170.9(2) |
| C(12) | C(7) | C(8) | C(13) | -173.7(2) |
| C(7) | C(8) | C(13) | C(14) | -125.0(3) |
| C(9) | C(8) | C(13) | C(14) | 57.2(3) |
| C(13) | C(8) | C(9) | C(10) | 177.6(2) |
| C(9) | C(10) | C(11) | C(12) | 2.3(4) |
| C(10) | C(11) | C(12) | C(16) | -174.3(2) |
| C(7) | C(12) | C(16) | C(18) | -95.2(3) |
| C(11) | C(12) | C(16) | C(18) | 80.2(3) |
| N(3) | C(19) | C(20) | C(25) | 0.0(3) |
| N(3) | C(19) | C(24) | C(26) | 0.6(4) |
| C(20) | C(19) | C(24) | C(26) | -177.7(2) |
| C(24) | C(19) | C(20) | C(25) | 178.4(2) |
| C(25) | C(20) | C(21) | C(22) | -179.4(3) |

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| Cl(1) | V(1) | N(1) | C(7) | 124.6(2) |
| Cl(1) | V(1) | N(2) | C(6) | -68.5(2) |
| Cl(2) | V(1) | N(1) | C(1) | 94.6(2) |
| Cl(2) | V(1) | N(2) | C(2) | -134.5(2) |
| Cl(2) | V(1) | N(3) | C(19) | -54(3) |
| N(1) | V(1) | N(2) | C(6) | 174.2(2) |
| N(2) | V(1) | N(1) | C(7) | -159.8(2) |
| N(3) | V(1) | N(1) | C(1) | -163.3(2) |
| N(2) | V(1) | N(3) | C(19) | -152(2) |
| N(3) | V(1) | N(2) | C(6) | 153.7(14) |
| V(1) | N(1) | C(7) | C(8) | 95.6(3) |
| C(1) | N(1) | C(7) | C(8) | -82.4(2) |
| C(7) | N(1) | C(1) | C(2) | 159.7(2) |
| V(1) | N(2) | C(2) | C(3) | -169.4(2) |
| C(2) | N(2) | C(6) | C(5) | -2.1(4) |
| C(6) | N(2) | C(2) | C(3) | 2.2(4) |
| V(1) | N(3) | C(19) | C(24) | -77(3) |
| N(1) | C(1) | C(2) | C(3) | -177.8(2) |
| C(1) | C(2) | C(3) | C(4) | -179.0(3) |
| C(3) | C(4) | C(5) | C(6) | 1.8(5) |
| N(1) | C(7) | C(8) | C(9) | -175.1(2) |
| N(1) | C(7) | C(12) | C(11) | 174.6(2) |
| C(8) | C(7) | C(12) | C(11) | -4.6(4) |
| C(12) | C(7) | C(8) | C(9) | 4.1(3) |
| C(7) | C(8) | C(9) | C(10) | -0.4(4) |
| C(7) | C(8) | C(13) | C(15) | 112.3(3) |
| C(9) | C(8) | C(13) | C(15) | -65.5(3) |
| C(8) | C(9) | C(10) | C(11) | -2.8(4) |
| C(10) | C(11) | C(12) | C(7) | 1.3(4) |
| C(7) | C(12) | C(16) | C(17) | 142.8(2) |
| C(11) | C(12) | C(16) | C(17) | -41.8(3) |
| N(3) | C(19) | C(20) | C(21) | -179.6(2) |
| N(3) | C(19) | C(24) | C(23) | 179.6(2) |
| C(20) | C(19) | C(24) | C(23) | 1.3(4) |
| C(24) | C(19) | C(20) | C(21) | -1.3(4) |
| C(19) | C(20) | C(21) | C(22) | 0.2(5) |
| C(20) | C(21) | C(22) | C(23) | 0.8(5) |

Table 8. Torsion angles (°) (continued)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| C(21) | C(22) | C(23) | C(24) | -0.8(5) |
| C(22) | C(23) | C(24) | C(26) | 178.7(3) |
| C(22) | C(23) | C(24) | C(19) | -0.2(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 |
|---|---|---|---|---|---|
| C(4) | C(14)[1)] | 3.546(4) | C(4) | C(17)[2)] | 3.436(6) |
| C(5) | C(5)[3)] | 3.400(5) | C(5) | C(6)[3)] | 3.564(4) |
| C(6) | C(5)[3)] | 3.564(4) | C(10) | C(26)[4)] | 3.546(5) |
| C(11) | C(26)[4)] | 3.595(4) | C(14) | C(4)[5)] | 3.546(4) |
| C(17) | C(4)[6)] | 3.436(6) | C(26) | C(10)[7)] | 3.546(5) |
| C(26) | C(11)[7)] | 3.595(4) | | | |

Symmetry Operators:

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

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| Cl(1) | H(4)[1)] | 3.254 | Cl(1) | H(5)[1)] | 3.116 |
| Cl(1) | H(9)[2)] | 2.911 | Cl(1) | H(18)[2)] | 3.414 |
| Cl(1) | H(24)[3)] | 3.338 | Cl(1) | H(29)[3)] | 3.328 |
| Cl(2) | H(2)[4)] | 3.202 | Cl(2) | H(3)[4)] | 2.966 |
| Cl(2) | H(7)[5)] | 2.689 | Cl(2) | H(11)[5)] | 3.490 |
| N(2) | H(26)[6)] | 3.088 | C(2) | H(26)[6)] | 3.027 |
| C(2) | H(29)[3)] | 3.583 | C(3) | H(12)[3)] | 3.515 |
| C(3) | H(13)[3)] | 3.050 | C(3) | H(26)[6)] | 3.171 |
| C(3) | H(27)[3)] | 3.437 | C(3) | H(31)[6)] | 3.583 |
| C(4) | H(6)[1)] | 3.489 | C(4) | H(11)[3)] | 3.600 |
| C(4) | H(12)[3)] | 3.322 | C(4) | H(13)[3)] | 3.157 |
| C(4) | H(18)[6)] | 3.268 | C(4) | H(19)[6)] | 3.396 |
| C(4) | H(20)[6)] | 3.084 | C(4) | H(26)[6)] | 3.345 |
| C(5) | H(5)[1)] | 3.344 | C(5) | H(25)[6)] | 3.555 |
| C(5) | H(26)[6)] | 3.350 | C(5) | H(29)[5)] | 3.221 |
| C(6) | H(5)[1)] | 3.337 | C(6) | H(11)[5)] | 3.345 |
| C(6) | H(25)[6)] | 3.382 | C(6) | H(26)[6)] | 3.213 |
| C(7) | H(15)[7)] | 3.532 | C(8) | H(15)[7)] | 3.239 |
| C(9) | H(6)[8)] | 3.579 | C(9) | H(15)[7)] | 2.993 |
| C(9) | H(25)[9)] | 3.458 | C(10) | H(15)[7)] | 3.082 |
| C(10) | H(24)[9)] | 3.499 | C(10) | H(25)[9)] | 3.032 |
| C(10) | H(30)[10)] | 3.515 | C(10) | H(31)[10)] | 3.097 |
| C(10) | H(32)[10)] | 3.464 | C(11) | H(14)[7)] | 3.545 |
| C(11) | H(15)[7)] | 3.346 | C(11) | H(30)[10)] | 3.196 |
| C(11) | H(31)[10)] | 3.370 | C(12) | H(15)[7)] | 3.568 |
| C(13) | H(26)[6)] | 3.584 | C(14) | H(3)[4)] | 3.349 |
| C(14) | H(4)[4)] | 2.950 | C(14) | H(6)[8)] | 2.965 |
| C(15) | H(15)[7)] | 3.557 | C(15) | H(21)[7)] | 3.401 |
| C(15) | H(23)[7)] | 3.479 | C(15) | H(31)[6)] | 3.293 |
| C(17) | H(4)[11)] | 2.643 | C(17) | H(12)[12)] | 3.409 |
| C(18) | H(14)[7)] | 3.593 | C(18) | H(16)[7)] | 3.326 |
| C(19) | H(21)[4)] | 3.579 | C(19) | H(22)[4)] | 3.007 |
| C(20) | H(22)[4)] | 3.349 | C(21) | H(8)[9)] | 3.425 |
| C(21) | H(22)[4)] | 3.563 | C(22) | H(8)[9)] | 3.101 |
| C(22) | H(22)[4)] | 3.492 | C(23) | H(22)[4)] | 3.168 |
| C(24) | H(22)[4)] | 2.907 | C(25) | H(2)[4)] | 3.520 |
| C(25) | H(5)[8)] | 3.076 | C(26) | H(8)[2)] | 3.013 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(26) | H(9)[2)] | 3.249 | C(26) | H(14)[11)] | 3.278 |
| C(26) | H(22)[4)] | 3.518 | H(1) | H(7)[7)] | 3.463 |
| H(1) | H(16)[7)] | 3.240 | H(1) | H(26)[6)] | 3.548 |
| H(2) | Cl(2)[3)] | 3.202 | H(2) | C(25)[3)] | 3.520 |
| H(2) | H(16)[7)] | 3.464 | H(2) | H(27)[3)] | 2.950 |
| H(2) | H(29)[3)] | 3.354 | H(3) | Cl(2)[3)] | 2.966 |
| H(3) | C(14)[3)] | 3.349 | H(3) | H(12)[3)] | 3.068 |
| H(3) | H(13)[3)] | 2.758 | H(3) | H(19)[6)] | 3.184 |
| H(3) | H(27)[3)] | 3.323 | H(3) | H(31)[6)] | 3.395 |
| H(4) | Cl(1)[1)] | 3.254 | H(4) | C(14)[3)] | 2.950 |
| H(4) | C(17)[6)] | 2.643 | H(4) | H(6)[1)] | 3.310 |
| H(4) | H(11)[3)] | 2.969 | H(4) | H(12)[3)] | 2.554 |
| H(4) | H(13)[3)] | 2.839 | H(4) | H(18)[6)] | 2.306 |
| H(4) | H(19)[6)] | 2.662 | H(4) | H(20)[6)] | 2.522 |
| H(5) | Cl(1)[1)] | 3.116 | H(5) | C(5)[1)] | 3.344 |
| H(5) | C(6)[1)] | 3.337 | H(5) | C(25)[5)] | 3.076 |
| H(5) | H(5)[1)] | 3.575 | H(5) | H(24)[5)] | 3.493 |
| H(5) | H(27)[5)] | 3.567 | H(5) | H(28)[5)] | 3.109 |
| H(5) | H(29)[5)] | 2.274 | H(6) | C(4)[1)] | 3.489 |
| H(6) | C(9)[5)] | 3.579 | H(6) | C(14)[5)] | 2.965 |
| H(6) | H(4)[1)] | 3.310 | H(6) | H(7)[5)] | 2.867 |
| H(6) | H(11)[5)] | 2.223 | H(6) | H(12)[5)] | 3.511 |
| H(6) | H(13)[5)] | 2.906 | H(6) | H(25)[6)] | 3.454 |
| H(6) | H(28)[5)] | 3.365 | H(6) | H(29)[5)] | 3.562 |
| H(7) | Cl(2)[8)] | 2.689 | H(7) | H(1)[7)] | 3.463 |
| H(7) | H(6)[8)] | 2.867 | H(7) | H(15)[7)] | 3.400 |
| H(7) | H(25)[9)] | 3.271 | H(8) | C(21)[9)] | 3.425 |
| H(8) | C(22)[9)] | 3.101 | H(8) | C(26)[10)] | 3.013 |
| H(8) | H(15)[7)] | 3.587 | H(8) | H(24)[9)] | 3.031 |
| H(8) | H(25)[9)] | 2.398 | H(8) | H(30)[10)] | 3.196 |
| H(8) | H(31)[10)] | 2.662 | H(8) | H(32)[10)] | 2.708 |
| H(9) | Cl(1)[10)] | 2.911 | H(9) | C(26)[10)] | 3.249 |
| H(9) | H(24)[9)] | 3.210 | H(9) | H(30)[10)] | 2.699 |
| H(9) | H(31)[10)] | 3.329 | H(9) | H(32)[10)] | 3.219 |
| H(10) | H(26)[6)] | 2.751 | H(11) | Cl(2)[8)] | 3.490 |
| H(11) | C(4)[4)] | 3.600 | H(11) | C(6)[8)] | 3.345 |
| H(11) | H(4)[4)] | 2.969 | H(11) | H(6)[8)] | 2.223 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(12) | C(3)[4)] | 3.515 | H(12) | C(4)[4)] | 3.322 |
| H(12) | C(17)[13)] | 3.409 | H(12) | H(3)[4)] | 3.068 |
| H(12) | H(4)[4)] | 2.554 | H(12) | H(6)[8)] | 3.511 |
| H(12) | H(18)[13)] | 2.963 | H(12) | H(19)[13)] | 2.973 |
| H(12) | H(26)[6)] | 3.594 | H(13) | C(3)[4)] | 3.050 |
| H(13) | C(4)[4)] | 3.157 | H(13) | H(3)[4)] | 2.758 |
| H(13) | H(4)[4)] | 2.839 | H(13) | H(6)[8)] | 2.906 |
| H(14) | C(11)[7)] | 3.545 | H(14) | C(18)[7)] | 3.593 |
| H(14) | C(26)[6)] | 3.278 | H(14) | H(21)[7)] | 3.337 |
| H(14) | H(23)[7)] | 3.005 | H(14) | H(26)[6)] | 3.240 |
| H(14) | H(30)[6)] | 3.094 | H(14) | H(31)[6)] | 2.675 |
| H(15) | C(7)[7)] | 3.532 | H(15) | C(8)[7)] | 3.239 |
| H(15) | C(9)[7)] | 2.993 | H(15) | C(10)[7)] | 3.082 |
| H(15) | C(11)[7)] | 3.346 | H(15) | C(12)[7)] | 3.568 |
| H(15) | C(15)[7)] | 3.557 | H(15) | H(7)[7)] | 3.400 |
| H(15) | H(8)[7)] | 3.587 | H(15) | H(15)[7)] | 3.001 |
| H(15) | H(16)[7)] | 3.385 | H(15) | H(31)[6)] | 3.098 |
| H(16) | C(18)[7)] | 3.326 | H(16) | H(1)[7)] | 3.240 |
| H(16) | H(2)[7)] | 3.464 | H(16) | H(15)[7)] | 3.385 |
| H(16) | H(21)[7)] | 2.696 | H(16) | H(23)[7)] | 3.142 |
| H(18) | Cl(1)[10)] | 3.414 | H(18) | C(4)[11)] | 3.268 |
| H(18) | H(4)[11)] | 2.306 | H(18) | H(12)[12)] | 2.963 |
| H(19) | C(4)[11)] | 3.396 | H(19) | H(3)[11)] | 3.184 |
| H(19) | H(4)[11)] | 2.662 | H(19) | H(12)[12)] | 2.973 |
| H(20) | C(4)[11)] | 3.084 | H(20) | H(4)[11)] | 2.522 |
| H(21) | C(15)[7)] | 3.401 | H(21) | C(19)[3)] | 3.579 |
| H(21) | H(14)[7)] | 3.337 | H(21) | H(16)[7)] | 2.696 |
| H(22) | C(19)[3)] | 3.007 | H(22) | C(20)[3)] | 3.349 |
| H(22) | C(21)[3)] | 3.563 | H(22) | C(22)[3)] | 3.492 |
| H(22) | C(23)[3)] | 3.168 | H(22) | C(24)[3)] | 2.907 |
| H(22) | C(26)[3)] | 3.518 | H(22) | H(30)[3)] | 3.130 |
| H(23) | C(15)[7)] | 3.479 | H(23) | H(14)[7)] | 3.005 |
| H(23) | H(16)[7)] | 3.142 | H(23) | H(23)[14)] | 3.583 |
| H(23) | H(30)[10)] | 3.543 | H(24) | Cl(1)[4)] | 3.338 |
| H(24) | C(10)[9)] | 3.499 | H(24) | H(5)[8)] | 3.493 |
| H(24) | H(8)[9)] | 3.031 | H(24) | H(9)[9)] | 3.210 |
| H(24) | H(24)[9)] | 3.491 | H(25) | C(5)[11)] | 3.555 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(25) | C(6)[11)] | 3.382 | H(25) | C(9)[9)] | 3.458 |
| H(25) | C(10)[9)] | 3.032 | H(25) | H(6)[11)] | 3.454 |
| H(25) | H(7)[9)] | 3.271 | H(25) | H(8)[9)] | 2.398 |
| H(25) | H(28)[9)] | 3.525 | H(26) | N(2)[11)] | 3.088 |
| H(26) | C(2)[11)] | 3.027 | H(26) | C(3)[11)] | 3.171 |
| H(26) | C(4)[11)] | 3.345 | H(26) | C(5)[11)] | 3.350 |
| H(26) | C(6)[11)] | 3.213 | H(26) | C(13)[11)] | 3.584 |
| H(26) | H(1)[11)] | 3.548 | H(26) | H(10)[11)] | 2.751 |
| H(26) | H(12)[11)] | 3.594 | H(26) | H(14)[11)] | 3.240 |
| H(27) | C(3)[4)] | 3.437 | H(27) | H(2)[4)] | 2.950 |
| H(27) | H(3)[4)] | 3.323 | H(27) | H(5)[8)] | 3.567 |
| H(28) | H(5)[8)] | 3.109 | H(28) | H(6)[8)] | 3.365 |
| H(28) | H(25)[9)] | 3.525 | H(29) | Cl(1)[4)] | 3.328 |
| H(29) | C(2)[4)] | 3.583 | H(29) | C(5)[8)] | 3.221 |
| H(29) | H(2)[4)] | 3.354 | H(29) | H(5)[8)] | 2.274 |
| H(29) | H(6)[8)] | 3.562 | H(30) | C(10)[2)] | 3.515 |
| H(30) | C(11)[2)] | 3.196 | H(30) | H(8)[2)] | 3.196 |
| H(30) | H(9)[2)] | 2.699 | H(30) | H(14)[11)] | 3.094 |
| H(30) | H(22)[4)] | 3.130 | H(30) | H(23)[2)] | 3.543 |
| H(31) | C(3)[11)] | 3.583 | H(31) | C(10)[2)] | 3.097 |
| H(31) | C(11)[2)] | 3.370 | H(31) | C(15)[11)] | 3.293 |
| H(31) | H(3)[11)] | 3.395 | H(31) | H(8)[2)] | 2.662 |
| H(31) | H(9)[2)] | 3.329 | H(31) | H(14)[11)] | 2.675 |
| H(31) | H(15)[11)] | 3.098 | H(32) | C(10)[2)] | 3.464 |
| H(32) | H(8)[2)] | 2.708 | H(32) | H(9)[2)] | 3.219 |

Symmetry Operators:

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

X-ray Structure Report for V(N=2,6-$Me_2C_6H_3$)$Cl_2$[2-(2,6-$F_2C_6H_3$)$CH_2$($C_5H_4N$)] (**1c**)

June 24, 2009

*Experimental*

Data Collection

A red block crystal of $C_{23}H_{18}Cl_2F_2N_3V$ having approximate dimensions of 0.40 x 0.30 x 0.20 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K$\alpha$ 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 triclinic cell with dimensions:

| | | | |
|---|---|---|---|
| a | = 7.3016(4) Å | $\alpha$ | = 99.261(3)$^o$ |
| b | = 10.2681(6) Å | $\beta$ | = 92.447(3)$^o$ |
| c | = 15.9162(12) Å | $\gamma$ | = 97.1083(15)$^o$ |
| V | = 1166.29(12) Å$^3$ | | |

For Z = 2 and F.W. = 496.26, the calculated density is 1.413 g/cm$^3$. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -80 $\pm$ 1$^o$C to a maximum 2$\theta$ value of 50.7$^o$. A total of 55 oscillation images were collected. A sweep of data was done using $\omega$ scans from 130.0 to 190.0$^o$ in 4.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 30.0$^o$. The exposure rate was 140.0 [sec./$^o$]. A second sweep was performed using $\omega$ scans from 0.0 to 160.0$^o$ in 4.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 180.0$^o$. 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 9482 reflections that were collected, 4207 were unique ($R_{int}$ = 0.022); equivalent reflections were merged.

The linear absorption coefficient, $\mu$, for Mo-K$\alpha$ radiation is 6.843 $cm^{-1}$. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.432 to 0.872. 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 4207 observed reflections and 281 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0404$$

$$wR2 = [ \; \Sigma \; ( \; w \; (Fo^2 - Fc^2)^2 \; ) / \; \Sigma \; w(Fo^2)^2]^{1/2} = 0.1316$$

The standard deviation of an observation of unit weight[4] was 1.15. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.75 and -0.42 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta$f' and $\Delta$f'' 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] crystallographic software package except for refinement, which was performed using SHELXL-97[10].

*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: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ 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

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

(10) SHELX97: Sheldrick, G.M. (1997).

*EXPERIMENTAL DETAILS*

A. Crystal Data

| | |
|---|---|
| Empirical Formula | $C_{23}H_{18}Cl_2F_2N_3V$ |
| Formula Weight | 496.26 |
| Crystal Color, Habit | red, block |
| Crystal Dimensions | 0.40 X 0.30 X 0.20 mm |
| Crystal System | triclinic |
| Lattice Type | Primitive |
| Indexing Images | 3 oscillations @ 240.0 seconds |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| Lattice Parameters | a = 7.3016(4) Å<br>b = 10.2681(6) Å<br>c = 15.9162(12) Å<br>$\alpha$ = 99.261(3) $^o$<br>$\beta$ = 92.447(3) $^o$<br>$\gamma$ = 97.1083(15) $^o$<br>V = 1166.29(12) Å$^3$ |
| Space Group | P-1 (#2) |
| Z value | 2 |
| $D_{calc}$ | 1.413 g/cm$^3$ |
| $F_{000}$ | 504.00 |
| $\mu$(MoK$\alpha$) | 6.843 cm$^{-1}$ |

B. Intensity Measurements

| | |
|---|---|
| Diffractometer | Rigaku RAXIS-RAPID |
| Radiation | MoK$\alpha$ ($\lambda$ = 0.71075 Å)<br>graphite monochromated |
| Detector Aperture | 280 mm x 256 mm |
| Data Images | 55 exposures |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=30.0) | 130.0 - 190.0$^{o}$ |
| Exposure Rate | 140.0 sec./$^{o}$ |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=180.0) | 0.0 - 160.0$^{o}$ |
| Exposure Rate | 140.0 sec./$^{o}$ |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| $2\theta_{max}$ | 50.7$^{o}$ |
| No. of Reflections Measured | Total: 9482<br>Unique: 4207 ($R_{int}$ = 0.022) |
| Corrections | Lorentz-polarization<br>Absorption<br>(trans. factors: 0.432 - 0.872) |

C. Structure Solution and Refinement

| | |
|---|---|
| Structure Solution | Direct Methods (SIR92) |
| Refinement | Full-matrix least-squares on $F^2$ |
| Function Minimized | $\Sigma$ w $(Fo^2 - Fc^2)^2$ |
| Least Squares Weights | $w = 1/ [ \sigma^2(Fo^2) + (0.0619 \cdot P)^2 + 1.1383 \cdot P ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$ |
| $2\theta_{max}$ cutoff | $50.7^o$ |
| Anomalous Dispersion | All non-hydrogen atoms |
| No. Observations (All reflections) | 4207 |
| No. Variables | 281 |
| Reflection/Parameter Ratio | 14.97 |
| Residuals: R1 (I>2.00$\sigma$(I)) | 0.0404 |
| Residuals: R (All reflections) | 0.0467 |
| Residuals: wR2 (All reflections) | 0.1316 |
| Goodness of Fit Indicator | 1.154 |
| Max Shift/Error in Final Cycle | 0.001 |
| Maximum peak in Final Diff. Map | 0.75 $e^-/Å^3$ |
| Minimum peak in Final Diff. Map | -0.42 $e^-/Å^3$ |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| V(1) | 0.12474(6) | 0.27661(4) | 0.80762(3) | 2.266(12) |
| Cl(1) | 0.00338(10) | 0.29282(8) | 0.93700(5) | 3.345(16) |
| Cl(2) | 0.00762(12) | 0.40235(7) | 0.72110(5) | 3.639(17) |
| F(1) | 0.4885(3) | 0.0546(2) | 0.89366(12) | 4.62(4) |
| F(2) | 0.3193(3) | 0.1292(2) | 0.61993(12) | 4.34(4) |
| N(1) | 0.3618(3) | 0.2269(2) | 0.79371(15) | 2.56(4) |
| N(2) | 0.3088(3) | 0.4595(2) | 0.86434(15) | 2.68(4) |
| N(3) | 0.0156(3) | 0.1254(2) | 0.76558(14) | 2.40(3) |
| C(1) | 0.3983(3) | 0.0971(2) | 0.75840(18) | 2.64(4) |
| C(2) | 0.4585(4) | 0.0098(3) | 0.8083(2) | 3.42(5) |
| C(3) | 0.4849(4) | -0.1176(3) | 0.7763(2) | 4.11(6) |
| C(4) | 0.4503(4) | -0.1626(3) | 0.6898(2) | 4.22(6) |
| C(5) | 0.3960(4) | -0.0807(3) | 0.6372(2) | 4.07(6) |
| C(6) | 0.3720(4) | 0.0475(3) | 0.6718(2) | 3.14(5) |
| C(7) | 0.5335(3) | 0.3184(3) | 0.8197(2) | 3.12(5) |
| C(8) | 0.4900(3) | 0.4483(3) | 0.86354(18) | 2.72(4) |
| C(9) | 0.2567(4) | 0.5735(2) | 0.90314(19) | 2.93(5) |
| C(10) | 0.3817(4) | 0.6801(3) | 0.94179(19) | 3.15(5) |
| C(11) | 0.5682(4) | 0.6682(3) | 0.9418(2) | 3.27(5) |
| C(12) | 0.6245(4) | 0.5517(3) | 0.9020(2) | 3.22(5) |
| C(13) | -0.0416(3) | -0.0060(2) | 0.73089(19) | 2.66(4) |
| C(14) | -0.0319(4) | -0.1050(3) | 0.7814(2) | 3.31(5) |
| C(15) | -0.0680(5) | -0.2368(3) | 0.7420(2) | 4.23(6) |
| C(16) | -0.1177(5) | -0.2694(3) | 0.6563(2) | 4.79(8) |
| C(17) | -0.1384(4) | -0.1710(3) | 0.6076(2) | 4.03(6) |
| C(18) | -0.1032(4) | -0.0375(3) | 0.6434(2) | 3.23(5) |
| C(19) | 0.0138(5) | -0.0698(3) | 0.8762(2) | 4.12(6) |
| C(20) | -0.1336(5) | 0.0706(3) | 0.5933(2) | 3.88(6) |
| C(21) | -0.4369(16) | -0.4494(7) | 0.4422(5) | 11.5(3) |
| C(22) | -0.319(2) | -0.5362(9) | 0.4753(7) | 17.6(5) |
| C(23) | -0.364(2) | -0.6115(8) | 0.5454(9) | 27.0(11) |

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

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| H(1) | 0.5262 | -0.1742 | 0.8128 | 4.93 |
| H(2) | 0.4647 | -0.2518 | 0.6669 | 5.06 |
| H(3) | 0.3751 | -0.1112 | 0.5776 | 4.89 |
| H(4) | 0.6024 | 0.3314 | 0.7688 | 3.75 |
| H(5) | 0.6127 | 0.2796 | 0.8584 | 3.75 |
| H(6) | 0.1285 | 0.5811 | 0.9041 | 3.52 |
| H(7) | 0.3406 | 0.7602 | 0.9679 | 3.78 |
| H(8) | 0.6569 | 0.7397 | 0.9691 | 3.93 |
| H(9) | 0.7521 | 0.5421 | 0.9007 | 3.86 |
| H(10) | -0.0583 | -0.3056 | 0.7747 | 5.07 |
| H(11) | -0.1381 | -0.3603 | 0.6300 | 5.75 |
| H(12) | -0.1772 | -0.1955 | 0.5489 | 4.83 |
| H(13) | -0.0772 | -0.0159 | 0.9024 | 4.95 |
| H(14) | 0.1376 | -0.0190 | 0.8871 | 4.95 |
| H(15) | 0.0105 | -0.1515 | 0.9009 | 4.95 |
| H(16) | -0.1686 | 0.0312 | 0.5336 | 4.66 |
| H(17) | -0.0193 | 0.1327 | 0.5963 | 4.66 |
| H(18) | -0.2326 | 0.1185 | 0.6171 | 4.66 |

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

Table 3. Anisotropic displacement parameters

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| V(1) | 0.0252(2) | 0.0323(2) | 0.0280(2) | 0.00555(19) | -0.00032(18) | 0.00248(19) |
| Cl(1) | 0.0360(3) | 0.0539(4) | 0.0335(3) | 0.0008(3) | 0.0070(2) | -0.0010(3) |
| Cl(2) | 0.0518(4) | 0.0378(4) | 0.0485(4) | 0.0081(3) | -0.0140(3) | 0.0094(3) |
| F(1) | 0.0683(13) | 0.0688(13) | 0.0427(11) | 0.0195(11) | -0.0041(9) | 0.0164(9) |
| F(2) | 0.0706(13) | 0.0610(12) | 0.0366(10) | 0.0233(10) | -0.0019(9) | 0.0079(8) |
| N(1) | 0.0274(11) | 0.0370(12) | 0.0324(12) | 0.0076(9) | 0.0014(9) | 0.0023(9) |
| N(2) | 0.0305(11) | 0.0407(13) | 0.0290(11) | 0.0031(10) | 0.0009(9) | 0.0023(9) |
| N(3) | 0.0286(11) | 0.0361(12) | 0.0274(11) | 0.0064(9) | 0.0009(8) | 0.0060(9) |
| C(1) | 0.0259(13) | 0.0387(14) | 0.0368(15) | 0.0090(11) | 0.0034(11) | 0.0060(12) |
| C(2) | 0.0377(16) | 0.0518(18) | 0.0424(17) | 0.0109(14) | 0.0022(13) | 0.0096(14) |
| C(3) | 0.0434(18) | 0.0467(18) | 0.072(2) | 0.0151(15) | 0.0065(16) | 0.0206(17) |
| C(4) | 0.0484(19) | 0.0396(17) | 0.072(2) | 0.0138(15) | 0.0080(17) | 0.0013(16) |
| C(5) | 0.0497(19) | 0.0515(19) | 0.0499(19) | 0.0137(16) | 0.0022(15) | -0.0072(15) |
| C(6) | 0.0350(15) | 0.0457(16) | 0.0392(16) | 0.0119(13) | 0.0029(12) | 0.0042(13) |
| C(7) | 0.0258(13) | 0.0467(17) | 0.0433(16) | 0.0044(12) | 0.0022(11) | -0.0007(13) |
| C(8) | 0.0307(14) | 0.0427(15) | 0.0302(14) | 0.0043(12) | 0.0012(11) | 0.0080(11) |
| C(9) | 0.0345(14) | 0.0387(15) | 0.0380(15) | 0.0088(12) | 0.0021(12) | 0.0029(12) |
| C(10) | 0.0442(16) | 0.0375(15) | 0.0359(15) | 0.0027(13) | 0.0008(12) | 0.0020(12) |
| C(11) | 0.0405(16) | 0.0394(16) | 0.0405(16) | -0.0060(13) | -0.0045(12) | 0.0052(12) |
| C(12) | 0.0287(14) | 0.0452(16) | 0.0468(17) | -0.0006(12) | -0.0009(12) | 0.0080(13) |
| C(13) | 0.0277(13) | 0.0337(14) | 0.0392(15) | 0.0048(11) | 0.0023(11) | 0.0033(11) |
| C(14) | 0.0343(15) | 0.0402(16) | 0.0535(19) | 0.0080(12) | 0.0060(13) | 0.0108(13) |
| C(15) | 0.0509(19) | 0.0349(16) | 0.077(2) | 0.0106(15) | 0.0085(17) | 0.0112(16) |
| C(16) | 0.058(2) | 0.0330(17) | 0.085(2) | 0.0033(15) | 0.0077(19) | -0.0067(17) |
| C(17) | 0.0463(18) | 0.0448(18) | 0.055(2) | 0.0042(15) | -0.0014(15) | -0.0103(15) |
| C(18) | 0.0369(15) | 0.0436(16) | 0.0385(16) | 0.0026(13) | 0.0019(12) | -0.0021(13) |
| C(19) | 0.058(2) | 0.0514(19) | 0.052(2) | 0.0088(16) | 0.0049(16) | 0.0227(16) |
| C(20) | 0.0539(19) | 0.055(2) | 0.0346(16) | 0.0046(16) | -0.0069(14) | 0.0013(14) |
| C(21) | 0.241(10) | 0.062(3) | 0.111(5) | -0.027(4) | -0.078(6) | 0.002(3) |
| C(22) | 0.363(17) | 0.094(6) | 0.154(9) | -0.081(8) | -0.067(10) | -0.046(5) |
| C(23) | 0.54(2) | 0.073(5) | 0.342(19) | -0.015(10) | -0.35(2) | -0.030(8) |

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) | 2.2661(9) | V(1) | Cl(2) | 2.2449(9) |
| V(1) | N(1) | 1.877(2) | V(1) | N(2) | 2.208(2) |
| V(1) | N(3) | 1.676(2) | F(1) | C(2) | 1.362(3) |
| F(2) | C(6) | 1.346(4) | N(1) | C(1) | 1.422(3) |
| N(1) | C(7) | 1.471(3) | N(2) | C(8) | 1.343(3) |
| N(2) | C(9) | 1.339(3) | N(3) | C(13) | 1.378(3) |
| C(1) | C(2) | 1.387(4) | C(1) | C(6) | 1.386(4) |
| C(2) | C(3) | 1.366(4) | C(3) | C(4) | 1.382(5) |
| C(4) | C(5) | 1.359(5) | C(5) | C(6) | 1.378(4) |
| C(7) | C(8) | 1.481(4) | C(8) | C(12) | 1.395(3) |
| C(9) | C(10) | 1.378(3) | C(10) | C(11) | 1.382(4) |
| C(11) | C(12) | 1.379(4) | C(13) | C(14) | 1.401(4) |
| C(13) | C(18) | 1.418(4) | C(14) | C(15) | 1.385(4) |
| C(14) | C(19) | 1.507(4) | C(15) | C(16) | 1.374(6) |
| C(16) | C(17) | 1.386(5) | C(17) | C(18) | 1.385(4) |
| C(18) | C(20) | 1.500(5) | C(21) | C(22) | 1.456(16) |
| C(21) | C(23)[1)] | 1.66(2) | C(22) | C(23) | 1.484(18) |

Symmetry Operators:

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

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) | 0.990 |
| C(7) | H(5) | 0.990 | C(9) | H(6) | 0.950 |
| C(10) | H(7) | 0.950 | C(11) | H(8) | 0.950 |
| C(12) | H(9) | 0.950 | C(15) | H(10) | 0.950 |
| C(16) | H(11) | 0.950 | C(17) | H(12) | 0.950 |
| C(19) | H(13) | 0.980 | C(19) | H(14) | 0.980 |
| C(19) | H(15) | 0.980 | C(20) | H(16) | 0.980 |
| C(20) | H(17) | 0.980 | C(20) | H(18) | 0.980 |

Table 6. Bond angles (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| Cl(1) | V(1) | Cl(2) | 114.03(3) | Cl(1) | V(1) | N(1) | 120.40(8) |
| Cl(1) | V(1) | N(2) | 85.09(6) | Cl(1) | V(1) | N(3) | 97.67(8) |
| Cl(2) | V(1) | N(1) | 120.51(8) | Cl(2) | V(1) | N(2) | 85.86(7) |
| Cl(2) | V(1) | N(3) | 100.97(8) | N(1) | V(1) | N(2) | 76.93(9) |
| N(1) | V(1) | N(3) | 94.10(10) | N(2) | V(1) | N(3) | 170.71(10) |
| V(1) | N(1) | C(1) | 124.67(16) | V(1) | N(1) | C(7) | 123.54(18) |
| C(1) | N(1) | C(7) | 111.8(2) | V(1) | N(2) | C(8) | 114.57(18) |
| V(1) | N(2) | C(9) | 126.56(19) | C(8) | N(2) | C(9) | 118.7(2) |
| V(1) | N(3) | C(13) | 169.3(2) | N(1) | C(1) | C(2) | 122.4(2) |
| N(1) | C(1) | C(6) | 122.4(2) | C(2) | C(1) | C(6) | 115.1(2) |
| F(1) | C(2) | C(1) | 117.3(2) | F(1) | C(2) | C(3) | 119.3(3) |
| C(1) | C(2) | C(3) | 123.4(3) | C(2) | C(3) | C(4) | 118.7(3) |
| C(3) | C(4) | C(5) | 120.7(3) | C(4) | C(5) | C(6) | 118.8(3) |
| F(2) | C(6) | C(1) | 117.8(2) | F(2) | C(6) | C(5) | 119.0(2) |
| C(1) | C(6) | C(5) | 123.2(3) | N(1) | C(7) | C(8) | 110.1(2) |
| N(2) | C(8) | C(7) | 114.7(2) | N(2) | C(8) | C(12) | 121.9(2) |
| C(7) | C(8) | C(12) | 123.5(2) | N(2) | C(9) | C(10) | 122.6(2) |
| C(9) | C(10) | C(11) | 118.7(2) | C(10) | C(11) | C(12) | 119.5(2) |
| C(8) | C(12) | C(11) | 118.6(2) | N(3) | C(13) | C(14) | 119.7(2) |
| N(3) | C(13) | C(18) | 118.6(2) | C(14) | C(13) | C(18) | 121.7(2) |
| C(13) | C(14) | C(15) | 118.1(3) | C(13) | C(14) | C(19) | 121.2(2) |
| C(15) | C(14) | C(19) | 120.7(3) | C(14) | C(15) | C(16) | 120.9(3) |
| C(15) | C(16) | C(17) | 120.7(3) | C(16) | C(17) | C(18) | 121.0(3) |
| C(13) | C(18) | C(17) | 117.4(3) | C(13) | C(18) | C(20) | 120.6(2) |
| C(17) | C(18) | C(20) | 122.0(2) | C(22) | C(21) | C(23)[1)] | 143.2(9) |
| C(21) | C(22) | C(23) | 124.6(12) | C(21)[1)] | C(23) | C(22) | 92.2(10) |

Symmetry Operators:

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

Table 7. Bond angles involving hydrogens (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| C(2) | C(3) | H(1) | 120.7 | C(4) | C(3) | H(1) | 120.7 |
| C(3) | C(4) | H(2) | 119.6 | C(5) | C(4) | H(2) | 119.6 |
| C(4) | C(5) | H(3) | 120.6 | C(6) | C(5) | H(3) | 120.6 |
| N(1) | C(7) | H(4) | 109.6 | N(1) | C(7) | H(5) | 109.6 |
| C(8) | C(7) | H(4) | 109.6 | C(8) | C(7) | H(5) | 109.6 |
| H(4) | C(7) | H(5) | 108.2 | N(2) | C(9) | H(6) | 118.7 |
| C(10) | C(9) | H(6) | 118.7 | C(9) | C(10) | H(7) | 120.6 |
| C(11) | C(10) | H(7) | 120.6 | C(10) | C(11) | H(8) | 120.3 |
| C(12) | C(11) | H(8) | 120.3 | C(8) | C(12) | H(9) | 120.7 |
| C(11) | C(12) | H(9) | 120.7 | C(14) | C(15) | H(10) | 119.5 |
| C(16) | C(15) | H(10) | 119.5 | C(15) | C(16) | H(11) | 119.7 |
| C(17) | C(16) | H(11) | 119.7 | C(16) | C(17) | H(12) | 119.5 |
| C(18) | C(17) | H(12) | 119.5 | C(14) | C(19) | H(13) | 109.5 |
| C(14) | C(19) | H(14) | 109.5 | C(14) | C(19) | H(15) | 109.5 |
| H(13) | C(19) | H(14) | 109.5 | H(13) | C(19) | H(15) | 109.5 |
| H(14) | C(19) | H(15) | 109.5 | C(18) | C(20) | H(16) | 109.5 |
| C(18) | C(20) | H(17) | 109.5 | C(18) | C(20) | H(18) | 109.5 |
| H(16) | C(20) | H(17) | 109.5 | H(16) | C(20) | H(18) | 109.5 |
| H(17) | C(20) | H(18) | 109.5 | | | | |

Table 8. Torsion Angles(°)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| Cl(1) | V(1) | N(1) | C(1) | 100.8(2) |
| Cl(1) | V(1) | N(2) | C(8) | 121.6(2) |
| Cl(1) | V(1) | N(3) | C(13) | -115.1(11) |
| Cl(2) | V(1) | N(1) | C(7) | 75.1(2) |
| Cl(2) | V(1) | N(2) | C(9) | 60.9(2) |
| N(1) | V(1) | N(2) | C(8) | -1.2(2) |
| N(2) | V(1) | N(1) | C(1) | 177.0(2) |
| N(1) | V(1) | N(3) | C(13) | 6.3(11) |
| N(3) | V(1) | N(1) | C(7) | -179.6(2) |
| N(3) | V(1) | N(2) | C(8) | 13.9(7) |
| V(1) | N(1) | C(1) | C(2) | -104.3(2) |
| V(1) | N(1) | C(7) | C(8) | 4.5(3) |
| C(7) | N(1) | C(1) | C(2) | 74.7(3) |
| V(1) | N(2) | C(8) | C(7) | 3.9(3) |
| V(1) | N(2) | C(9) | C(10) | 175.6(2) |
| C(9) | N(2) | C(8) | C(7) | 179.6(2) |
| V(1) | N(3) | C(13) | C(14) | 70.7(12) |
| N(1) | C(1) | C(2) | F(1) | -2.2(4) |
| N(1) | C(1) | C(6) | F(2) | 3.6(4) |
| C(2) | C(1) | C(6) | F(2) | -177.9(2) |
| C(6) | C(1) | C(2) | F(1) | 179.2(2) |
| F(1) | C(2) | C(3) | C(4) | 178.7(2) |
| C(2) | C(3) | C(4) | C(5) | 1.8(5) |
| C(4) | C(5) | C(6) | F(2) | 179.6(3) |
| N(1) | C(7) | C(8) | N(2) | -5.2(3) |
| N(2) | C(8) | C(12) | C(11) | 0.1(3) |
| N(2) | C(9) | C(10) | C(11) | -1.1(4) |
| C(10) | C(11) | C(12) | C(8) | -0.8(5) |
| N(3) | C(13) | C(14) | C(19) | 8.9(4) |
| N(3) | C(13) | C(18) | C(20) | -9.4(4) |
| C(14) | C(13) | C(18) | C(20) | 172.8(2) |
| C(18) | C(13) | C(14) | C(19) | -173.2(3) |
| C(19) | C(14) | C(15) | C(16) | 177.1(3) |
| C(15) | C(16) | C(17) | C(18) | 2.3(5) |
| C(16) | C(17) | C(18) | C(20) | -176.9(3) |
| C(23)[1)] | C(21) | C(22) | C(23) | 1.6(18) |

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| Cl(1) | V(1) | N(1) | C(7) | -78.2(2) |
| Cl(1) | V(1) | N(2) | C(9) | -53.7(2) |
| Cl(2) | V(1) | N(1) | C(1) | -105.9(2) |
| Cl(2) | V(1) | N(2) | C(8) | -123.8(2) |
| Cl(2) | V(1) | N(3) | C(13) | 128.5(11) |
| N(1) | V(1) | N(2) | C(9) | -176.5(2) |
| N(2) | V(1) | N(1) | C(7) | -2.0(2) |
| N(3) | V(1) | N(1) | C(1) | -0.6(2) |
| N(2) | V(1) | N(3) | C(13) | -8.4(16) |
| N(3) | V(1) | N(2) | C(9) | -161.4(5) |
| V(1) | N(1) | C(1) | C(6) | 74.1(3) |
| C(1) | N(1) | C(7) | C(8) | -174.6(2) |
| C(7) | N(1) | C(1) | C(6) | -106.8(3) |
| V(1) | N(2) | C(8) | C(12) | -175.6(2) |
| C(8) | N(2) | C(9) | C(10) | 0.5(4) |
| C(9) | N(2) | C(8) | C(12) | 0.1(3) |
| V(1) | N(3) | C(13) | C(18) | -107.2(11) |
| N(1) | C(1) | C(2) | C(3) | 176.4(2) |
| N(1) | C(1) | C(6) | C(5) | -176.0(2) |
| C(2) | C(1) | C(6) | C(5) | 2.5(4) |
| C(6) | C(1) | C(2) | C(3) | -2.1(4) |
| C(1) | C(2) | C(3) | C(4) | 0.1(4) |
| C(3) | C(4) | C(5) | C(6) | -1.4(5) |
| C(4) | C(5) | C(6) | C(1) | -0.8(5) |
| N(1) | C(7) | C(8) | C(12) | 174.3(2) |
| C(7) | C(8) | C(12) | C(11) | -179.4(3) |
| C(9) | C(10) | C(11) | C(12) | 1.3(4) |
| N(3) | C(13) | C(14) | C(15) | -172.0(2) |
| N(3) | C(13) | C(18) | C(17) | 172.3(2) |
| C(14) | C(13) | C(18) | C(17) | -5.5(4) |
| C(18) | C(13) | C(14) | C(15) | 5.9(4) |
| C(13) | C(14) | C(15) | C(16) | -2.0(5) |
| C(14) | C(15) | C(16) | C(17) | -2.0(5) |
| C(16) | C(17) | C(18) | C(13) | 1.4(5) |
| C(22) | C(21) | C(23)[1)] | C(22)[1)] | -1.3(15) |
| C(21) | C(22) | C(23) | C(21)[1)] | -1.0(11) |

Symmetry Operators:

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

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) | F(2) | 3.560(2) | V(1) | C(1) | 2.929(2) |
| V(1) | C(7) | 2.956(2) | V(1) | C(8) | 3.024(2) |
| V(1) | C(9) | 3.192(2) | V(1) | C(13) | 3.041(2) |
| Cl(1) | N(2) | 3.025(2) | Cl(1) | N(3) | 2.993(2) |
| Cl(1) | C(9) | 3.359(3) | Cl(1) | C(9)[1)] | 3.449(3) |
| Cl(1) | C(10)[1)] | 3.491(3) | Cl(1) | C(11)[2)] | 3.549(3) |
| Cl(2) | N(1) | 3.583(2) | Cl(2) | N(2) | 3.033(2) |
| Cl(2) | N(3) | 3.046(2) | Cl(2) | C(9) | 3.451(2) |
| F(1) | N(1) | 2.775(3) | F(1) | C(1) | 2.347(3) |
| F(1) | C(3) | 2.353(4) | F(1) | C(6) | 3.583(3) |
| F(1) | C(7) | 3.110(4) | F(1) | C(10)[2)] | 3.469(3) |
| F(1) | C(19) | 3.526(4) | F(2) | V(1) | 3.560(2) |
| F(2) | N(1) | 2.777(2) | F(2) | N(3) | 3.277(3) |
| F(2) | C(1) | 2.339(3) | F(2) | C(2) | 3.571(4) |
| F(2) | C(4) | 3.581(4) | F(2) | C(5) | 2.347(4) |
| F(2) | C(13) | 3.503(3) | F(2) | C(18) | 3.404(3) |
| F(2) | C(20) | 3.284(4) | F(2) | C(21)[3)] | 3.597(8) |
| N(1) | Cl(2) | 3.583(2) | N(1) | F(1) | 2.775(3) |
| N(1) | F(2) | 2.777(2) | N(1) | N(2) | 2.554(3) |
| N(1) | N(3) | 2.604(3) | N(1) | C(2) | 2.462(4) |
| N(1) | C(6) | 2.461(3) | N(1) | C(8) | 2.419(3) |
| N(1) | C(13) | 3.554(3) | N(2) | Cl(1) | 3.025(2) |
| N(2) | Cl(2) | 3.033(2) | N(2) | N(1) | 2.554(3) |
| N(2) | C(7) | 2.378(3) | N(2) | C(10) | 2.383(3) |
| N(2) | C(11) | 2.765(3) | N(2) | C(12) | 2.394(3) |
| N(3) | Cl(1) | 2.993(2) | N(3) | Cl(2) | 3.046(2) |
| N(3) | F(2) | 3.277(3) | N(3) | N(1) | 2.604(3) |
| N(3) | C(1) | 2.850(3) | N(3) | C(6) | 3.178(3) |
| N(3) | C(14) | 2.404(4) | N(3) | C(18) | 2.404(3) |
| N(3) | C(19) | 2.872(4) | N(3) | C(20) | 2.850(3) |
| C(1) | V(1) | 2.929(2) | C(1) | F(1) | 2.347(3) |
| C(1) | F(2) | 2.339(3) | C(1) | N(3) | 2.850(3) |
| C(1) | C(3) | 2.423(4) | C(1) | C(4) | 2.792(4) |
| C(1) | C(5) | 2.431(4) | C(1) | C(7) | 2.396(3) |
| C(1) | C(13) | 3.243(3) | C(2) | F(2) | 3.571(4) |
| C(2) | N(1) | 2.462(4) | C(2) | C(4) | 2.363(4) |
| C(2) | C(5) | 2.732(4) | C(2) | C(6) | 2.341(4) |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(2) | C(7) | 3.122(4) | C(2) | C(19) | 3.509(4) |
| C(3) | F(1) | 2.353(4) | C(3) | C(1) | 2.423(4) |
| C(3) | C(5) | 2.382(5) | C(3) | C(6) | 2.730(5) |
| C(3) | C(14)[4)] | 3.512(4) | C(4) | F(2) | 3.581(4) |
| C(4) | C(1) | 2.792(4) | C(4) | C(2) | 2.363(4) |
| C(4) | C(6) | 2.357(5) | C(4) | C(16)[4)] | 3.499(5) |
| C(4) | C(17)[4)] | 3.331(5) | C(4) | C(18)[4)] | 3.506(4) |
| C(5) | F(2) | 2.347(4) | C(5) | C(1) | 2.431(4) |
| C(5) | C(2) | 2.732(4) | C(5) | C(3) | 2.382(5) |
| C(6) | F(1) | 3.583(3) | C(6) | N(1) | 2.461(3) |
| C(6) | N(3) | 3.178(3) | C(6) | C(2) | 2.341(4) |
| C(6) | C(3) | 2.730(5) | C(6) | C(4) | 2.357(5) |
| C(6) | C(7) | 3.393(4) | C(6) | C(13) | 3.209(4) |
| C(6) | C(18) | 3.463(4) | C(7) | V(1) | 2.956(2) |
| C(7) | F(1) | 3.110(4) | C(7) | N(2) | 2.378(3) |
| C(7) | C(1) | 2.396(3) | C(7) | C(2) | 3.122(4) |
| C(7) | C(6) | 3.393(4) | C(7) | C(12) | 2.533(4) |
| C(8) | V(1) | 3.024(2) | C(8) | N(1) | 2.419(3) |
| C(8) | C(9) | 2.307(4) | C(8) | C(10) | 2.726(4) |
| C(8) | C(11) | 2.385(4) | C(8) | C(11)[2)] | 3.520(4) |
| C(9) | V(1) | 3.192(2) | C(9) | Cl(1) | 3.359(3) |
| C(9) | Cl(1)[1)] | 3.449(3) | C(9) | Cl(2) | 3.451(2) |
| C(9) | C(8) | 2.307(4) | C(9) | C(11) | 2.374(4) |
| C(9) | C(12) | 2.723(4) | C(10) | Cl(1)[1)] | 3.491(3) |
| C(10) | F(1)[2)] | 3.469(3) | C(10) | N(2) | 2.383(3) |
| C(10) | C(8) | 2.726(4) | C(10) | C(12) | 2.384(4) |
| C(11) | Cl(1)[2)] | 3.549(3) | C(11) | N(2) | 2.765(3) |
| C(11) | C(8) | 2.385(4) | C(11) | C(8)[2)] | 3.520(4) |
| C(11) | C(9) | 2.374(4) | C(12) | N(2) | 2.394(3) |
| C(12) | C(7) | 2.533(4) | C(12) | C(9) | 2.723(4) |
| C(12) | C(10) | 2.384(4) | C(13) | V(1) | 3.041(2) |
| C(13) | F(2) | 3.503(3) | C(13) | N(1) | 3.554(3) |
| C(13) | C(1) | 3.243(3) | C(13) | C(6) | 3.209(4) |
| C(13) | C(15) | 2.389(4) | C(13) | C(16) | 2.752(4) |
| C(13) | C(17) | 2.394(4) | C(13) | C(19) | 2.533(4) |
| C(13) | C(20) | 2.535(4) | C(14) | N(3) | 2.404(4) |
| C(14) | C(3)[5)] | 3.512(4) | C(14) | C(16) | 2.400(4) |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(14) | C(17) | 2.793(4) | C(14) | C(18) | 2.461(4) |
| C(15) | C(13) | 2.389(4) | C(15) | C(17) | 2.398(5) |
| C(15) | C(18) | 2.800(5) | C(15) | C(19) | 2.514(4) |
| C(16) | C(4)[5)] | 3.499(5) | C(16) | C(13) | 2.752(4) |
| C(16) | C(14) | 2.400(4) | C(16) | C(18) | 2.412(4) |
| C(17) | C(4)[5)] | 3.331(5) | C(17) | C(13) | 2.394(4) |
| C(17) | C(14) | 2.793(4) | C(17) | C(15) | 2.398(5) |
| C(17) | C(20) | 2.523(5) | C(18) | F(2) | 3.404(3) |
| C(18) | N(3) | 2.404(3) | C(18) | C(4)[5)] | 3.506(4) |
| C(18) | C(6) | 3.463(4) | C(18) | C(14) | 2.461(4) |
| C(18) | C(15) | 2.800(5) | C(18) | C(16) | 2.412(4) |
| C(19) | F(1) | 3.526(4) | C(19) | N(3) | 2.872(4) |
| C(19) | C(2) | 3.509(4) | C(19) | C(13) | 2.533(4) |
| C(19) | C(15) | 2.514(4) | C(20) | F(2) | 3.284(4) |
| C(20) | N(3) | 2.850(3) | C(20) | C(13) | 2.535(4) |
| C(20) | C(17) | 2.523(5) | C(21) | F(2)[3)] | 3.597(8) |
| C(21) | C(21)[6)] | 2.417(12) | C(21) | C(22)[6)] | 2.266(17) |
| C(21) | C(23) | 2.603(16) | C(22) | C(21)[6)] | 2.266(17) |
| C(22) | C(22)[6)] | 2.94(2) | C(22) | C(23)[6)] | 2.95(2) |
| C(23) | C(21) | 2.603(16) | C(23) | C(22)[6)] | 2.95(2) |

Symmetry Operators:

| | | | |
|---|---|---|---|
| (1) | -X,-Y+1,-Z+2 | (2) | -X+1,-Y+1,-Z+2 |
| (3) | -X,-Y,-Z+1 | (4) | X+1,Y,Z |
| (5) | X-1,Y,Z | (6) | -X-1,-Y-1,-Z+1 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| V(1) | H(4) | 3.560 | V(1) | H(6) | 3.250 |
| V(1) | H(14) | 3.482 | V(1) | H(17) | 3.510 |
| Cl(1) | H(5)[1)] | 3.044 | Cl(1) | H(6) | 3.121 |
| Cl(1) | H(6)[2)] | 2.909 | Cl(1) | H(7)[2)] | 3.024 |
| Cl(1) | H(8)[3)] | 2.924 | Cl(1) | H(9)[1)] | 3.435 |
| Cl(1) | H(9)[3)] | 3.191 | Cl(1) | H(13) | 3.106 |
| Cl(1) | H(14) | 3.445 | Cl(1) | H(15)[4)] | 3.157 |
| Cl(2) | H(4)[1)] | 3.114 | Cl(2) | H(6) | 3.207 |
| Cl(2) | H(10)[5)] | 3.086 | Cl(2) | H(11)[5)] | 3.290 |
| Cl(2) | H(17) | 3.117 | Cl(2) | H(18) | 3.354 |
| F(1) | H(1) | 2.544 | F(1) | H(5) | 2.532 |
| F(1) | H(7)[6)] | 3.486 | F(1) | H(7)[3)] | 2.803 |
| F(1) | H(8)[3)] | 3.103 | F(1) | H(13)[7)] | 3.341 |
| F(1) | H(14) | 2.573 | F(2) | H(3) | 2.540 |
| F(2) | H(4) | 3.336 | F(2) | H(12)[8)] | 3.047 |
| F(2) | H(16)[8)] | 2.815 | F(2) | H(17) | 2.490 |
| F(2) | H(18)[7)] | 3.289 | N(1) | H(4) | 2.030 |
| N(1) | H(5) | 2.030 | N(1) | H(14) | 3.415 |
| N(2) | H(4) | 2.996 | N(2) | H(5) | 3.054 |
| N(2) | H(6) | 1.979 | N(2) | H(7) | 3.234 |
| N(2) | H(9) | 3.246 | N(3) | H(13) | 2.864 |
| N(3) | H(14) | 2.799 | N(3) | H(17) | 2.710 |
| N(3) | H(18) | 2.902 | C(1) | H(1) | 3.276 |
| C(1) | H(3) | 3.283 | C(1) | H(4) | 2.645 |
| C(1) | H(5) | 2.569 | C(1) | H(14) | 3.131 |
| C(1) | H(18)[7)] | 3.591 | C(2) | H(1) | 2.022 |
| C(2) | H(2) | 3.222 | C(2) | H(4) | 3.502 |
| C(2) | H(5) | 2.831 | C(2) | H(14) | 2.712 |
| C(3) | H(2) | 2.027 | C(3) | H(3) | 3.243 |
| C(3) | H(14) | 3.318 | C(4) | H(1) | 2.038 |
| C(4) | H(3) | 2.016 | C(5) | H(1) | 3.240 |
| C(5) | H(2) | 2.007 | C(5) | H(16)[8)] | 3.267 |
| C(5) | H(18)[7)] | 3.247 | C(6) | H(2) | 3.217 |
| C(6) | H(3) | 2.033 | C(6) | H(4) | 3.290 |
| C(6) | H(16)[8)] | 3.463 | C(6) | H(17) | 3.322 |
| C(6) | H(18)[7)] | 3.086 | C(7) | H(9) | 2.729 |
| C(8) | H(4) | 2.039 | C(8) | H(5) | 2.039 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(8) | H(6) | 3.162 | C(8) | H(8) | 3.250 |
| C(8) | H(9) | 2.050 | C(9) | H(1)[5)] | 3.582 |
| C(9) | H(7) | 2.033 | C(9) | H(8) | 3.237 |
| C(9) | H(9)[3)] | 3.514 | C(9) | H(10)[5)] | 3.484 |
| C(9) | H(15)[5)] | 3.534 | C(10) | H(1)[5)] | 2.890 |
| C(10) | H(5)[3)] | 3.137 | C(10) | H(6) | 2.014 |
| C(10) | H(8) | 2.033 | C(10) | H(9) | 3.248 |
| C(10) | H(15)[5)] | 3.483 | C(11) | H(1)[5)] | 2.841 |
| C(11) | H(5)[3)] | 3.482 | C(11) | H(6) | 3.228 |
| C(11) | H(7) | 2.037 | C(11) | H(9) | 2.035 |
| C(12) | H(1)[5)] | 3.486 | C(12) | H(4) | 2.824 |
| C(12) | H(5) | 2.758 | C(12) | H(7) | 3.247 |
| C(12) | H(8) | 2.031 | C(12) | H(10)[9)] | 3.483 |
| C(13) | H(10) | 3.251 | C(13) | H(12) | 3.259 |
| C(13) | H(13) | 2.770 | C(13) | H(14) | 2.790 |
| C(13) | H(15) | 3.324 | C(13) | H(16) | 3.330 |
| C(13) | H(17) | 2.757 | C(13) | H(18) | 2.798 |
| C(14) | H(1)[1)] | 3.294 | C(14) | H(10) | 2.029 |
| C(14) | H(11) | 3.253 | C(14) | H(13) | 2.053 |
| C(14) | H(14) | 2.053 | C(14) | H(15) | 2.053 |
| C(15) | H(1)[1)] | 3.315 | C(15) | H(2)[1)] | 3.544 |
| C(15) | H(11) | 2.020 | C(15) | H(12) | 3.249 |
| C(15) | H(13) | 3.139 | C(15) | H(14) | 3.130 |
| C(15) | H(15) | 2.555 | C(16) | H(2)[1)] | 3.087 |
| C(16) | H(10) | 2.019 | C(16) | H(12) | 2.030 |
| C(17) | H(2)[1)] | 3.141 | C(17) | H(10) | 3.251 |
| C(17) | H(11) | 2.032 | C(17) | H(16) | 2.574 |
| C(17) | H(16)[8)] | 3.580 | C(17) | H(17) | 3.168 |
| C(17) | H(17)[8)] | 3.554 | C(17) | H(18) | 3.115 |
| C(18) | H(11) | 3.264 | C(18) | H(12) | 2.029 |
| C(18) | H(16) | 2.047 | C(18) | H(16)[8)] | 3.520 |
| C(18) | H(17) | 2.047 | C(18) | H(18) | 2.047 |
| C(19) | H(7)[6)] | 3.525 | C(19) | H(8)[10)] | 3.578 |
| C(19) | H(10) | 2.665 | C(19) | H(13)[4)] | 3.488 |
| C(20) | H(3)[8)] | 3.288 | C(20) | H(12) | 2.685 |
| C(20) | H(12)[8)] | 3.545 | C(20) | H(16)[8)] | 3.211 |
| C(21) | H(2)[11)] | 3.251 | C(21) | H(11) | 3.550 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(21) | H(11)[12)] | 3.499 | C(21) | H(12) | 3.218 |
| C(22) | H(2)[11)] | 2.937 | C(22) | H(3)[11)] | 3.550 |
| C(22) | H(11) | 2.961 | C(22) | H(12) | 3.516 |
| C(23) | H(2)[11)] | 3.454 | C(23) | H(3)[11)] | 3.175 |
| C(23) | H(11) | 2.972 | C(23) | H(18)[6)] | 3.392 |
| H(1) | F(1) | 2.544 | H(1) | C(1) | 3.276 |
| H(1) | C(2) | 2.022 | H(1) | C(4) | 2.038 |
| H(1) | C(5) | 3.240 | H(1) | C(9)[6)] | 3.582 |
| H(1) | C(10)[6)] | 2.890 | H(1) | C(11)[6)] | 2.841 |
| H(1) | C(12)[6)] | 3.486 | H(1) | C(14)[7)] | 3.294 |
| H(1) | C(15)[7)] | 3.315 | H(1) | H(2) | 2.336 |
| H(1) | H(7)[6)] | 2.997 | H(1) | H(8)[6)] | 2.932 |
| H(1) | H(10)[7)] | 3.507 | H(1) | H(13)[7)] | 3.286 |
| H(1) | H(14) | 3.576 | H(2) | C(2) | 3.222 |
| H(2) | C(3) | 2.027 | H(2) | C(5) | 2.007 |
| H(2) | C(6) | 3.217 | H(2) | C(15)[7)] | 3.544 |
| H(2) | C(16)[7)] | 3.087 | H(2) | C(17)[7)] | 3.141 |
| H(2) | C(21)[11)] | 3.251 | H(2) | C(22)[11)] | 2.937 |
| H(2) | C(23)[11)] | 3.454 | H(2) | H(1) | 2.336 |
| H(2) | H(3) | 2.312 | H(2) | H(11)[7)] | 3.276 |
| H(2) | H(12)[7)] | 3.330 | H(3) | F(2) | 2.540 |
| H(3) | C(1) | 3.283 | H(3) | C(3) | 3.243 |
| H(3) | C(4) | 2.016 | H(3) | C(6) | 2.033 |
| H(3) | C(20)[8)] | 3.288 | H(3) | C(22)[11)] | 3.550 |
| H(3) | C(23)[11)] | 3.175 | H(3) | H(2) | 2.312 |
| H(3) | H(12)[7)] | 3.513 | H(3) | H(16)[8)] | 2.574 |
| H(3) | H(18)[7)] | 3.444 | H(3) | H(18)[8)] | 3.212 |
| H(4) | V(1) | 3.560 | H(4) | Cl(2)[7)] | 3.114 |
| H(4) | F(2) | 3.336 | H(4) | N(1) | 2.030 |
| H(4) | N(2) | 2.996 | H(4) | C(1) | 2.645 |
| H(4) | C(2) | 3.502 | H(4) | C(6) | 3.290 |
| H(4) | C(8) | 2.039 | H(4) | C(12) | 2.824 |
| H(4) | H(5) | 1.603 | H(4) | H(9) | 2.836 |
| H(4) | H(18)[7)] | 3.355 | H(5) | Cl(1)[7)] | 3.044 |
| H(5) | F(1) | 2.532 | H(5) | N(1) | 2.030 |
| H(5) | N(2) | 3.054 | H(5) | C(1) | 2.569 |
| H(5) | C(2) | 2.831 | H(5) | C(8) | 2.039 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(5) | C(10)[3)] | 3.137 | H(5) | C(11)[3)] | 3.482 |
| H(5) | C(12) | 2.758 | H(5) | H(4) | 1.603 |
| H(5) | H(7)[3)] | 2.870 | H(5) | H(8)[3)] | 3.462 |
| H(5) | H(9) | 2.726 | H(6) | V(1) | 3.250 |
| H(6) | Cl(1) | 3.121 | H(6) | Cl(1)[2)] | 2.909 |
| H(6) | Cl(2) | 3.207 | H(6) | N(2) | 1.979 |
| H(6) | C(8) | 3.162 | H(6) | C(10) | 2.014 |
| H(6) | C(11) | 3.228 | H(6) | H(7) | 2.318 |
| H(6) | H(9)[1)] | 2.724 | H(6) | H(10)[5)] | 2.894 |
| H(6) | H(15)[5)] | 2.984 | H(7) | Cl(1)[2)] | 3.024 |
| H(7) | F(1)[5)] | 3.486 | H(7) | F(1)[3)] | 2.803 |
| H(7) | N(2) | 3.234 | H(7) | C(9) | 2.033 |
| H(7) | C(11) | 2.037 | H(7) | C(12) | 3.247 |
| H(7) | C(19)[5)] | 3.525 | H(7) | H(1)[5)] | 2.997 |
| H(7) | H(5)[3)] | 2.870 | H(7) | H(6) | 2.318 |
| H(7) | H(8) | 2.345 | H(7) | H(14)[5)] | 3.255 |
| H(7) | H(15)[5)] | 2.898 | H(8) | Cl(1)[3)] | 2.924 |
| H(8) | F(1)[3)] | 3.103 | H(8) | C(8) | 3.250 |
| H(8) | C(9) | 3.237 | H(8) | C(10) | 2.033 |
| H(8) | C(12) | 2.031 | H(8) | C(19)[9)] | 3.578 |
| H(8) | H(1)[5)] | 2.932 | H(8) | H(5)[3)] | 3.462 |
| H(8) | H(7) | 2.345 | H(8) | H(9) | 2.343 |
| H(8) | H(13)[9)] | 3.320 | H(8) | H(14)[3)] | 3.499 |
| H(8) | H(15)[9)] | 3.002 | H(9) | Cl(1)[7)] | 3.435 |
| H(9) | Cl(1)[3)] | 3.191 | H(9) | N(2) | 3.246 |
| H(9) | C(7) | 2.729 | H(9) | C(8) | 2.050 |
| H(9) | C(9)[3)] | 3.514 | H(9) | C(10) | 3.248 |
| H(9) | C(11) | 2.035 | H(9) | H(4) | 2.836 |
| H(9) | H(5) | 2.726 | H(9) | H(6)[7)] | 2.724 |
| H(9) | H(8) | 2.343 | H(9) | H(10)[9)] | 3.016 |
| H(9) | H(15)[9)] | 3.462 | H(10) | Cl(2)[6)] | 3.086 |
| H(10) | C(9)[6)] | 3.484 | H(10) | C(12)[10)] | 3.483 |
| H(10) | C(13) | 3.251 | H(10) | C(14) | 2.029 |
| H(10) | C(16) | 2.019 | H(10) | C(17) | 3.251 |
| H(10) | C(19) | 2.665 | H(10) | H(1)[1)] | 3.507 |
| H(10) | H(6)[6)] | 2.894 | H(10) | H(9)[10)] | 3.016 |
| H(10) | H(11) | 2.313 | H(10) | H(13) | 3.343 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(10) | H(14) | 3.309 | H(10) | H(15) | 2.341 |
| H(11) | Cl(2)[6)] | 3.290 | H(11) | C(14) | 3.253 |
| H(11) | C(15) | 2.020 | H(11) | C(17) | 2.032 |
| H(11) | C(18) | 3.264 | H(11) | C(21) | 3.550 |
| H(11) | C(21)[12)] | 3.499 | H(11) | C(22) | 2.961 |
| H(11) | C(23) | 2.972 | H(11) | H(2)[1)] | 3.276 |
| H(11) | H(10) | 2.313 | H(11) | H(12) | 2.325 |
| H(12) | F(2)[8)] | 3.047 | H(12) | C(13) | 3.259 |
| H(12) | C(15) | 3.249 | H(12) | C(16) | 2.030 |
| H(12) | C(18) | 2.029 | H(12) | C(20) | 2.685 |
| H(12) | C(20)[8)] | 3.545 | H(12) | C(21) | 3.218 |
| H(12) | C(22) | 3.516 | H(12) | H(2)[1)] | 3.330 |
| H(12) | H(3)[1)] | 3.513 | H(12) | H(11) | 2.325 |
| H(12) | H(16) | 2.372 | H(12) | H(16)[8)] | 3.300 |
| H(12) | H(17) | 3.376 | H(12) | H(17)[8)] | 2.889 |
| H(12) | H(18) | 3.312 | H(13) | Cl(1) | 3.106 |
| H(13) | F(1)[1)] | 3.341 | H(13) | N(3) | 2.864 |
| H(13) | C(13) | 2.770 | H(13) | C(14) | 2.053 |
| H(13) | C(15) | 3.139 | H(13) | C(19)[4)] | 3.488 |
| H(13) | H(1)[1)] | 3.286 | H(13) | H(8)[10)] | 3.320 |
| H(13) | H(10) | 3.343 | H(13) | H(13)[4)] | 3.212 |
| H(13) | H(14) | 1.600 | H(13) | H(14)[4)] | 3.366 |
| H(13) | H(15) | 1.600 | H(13) | H(15)[4)] | 3.306 |
| H(14) | V(1) | 3.482 | H(14) | Cl(1) | 3.445 |
| H(14) | F(1) | 2.573 | H(14) | N(1) | 3.415 |
| H(14) | N(3) | 2.799 | H(14) | C(1) | 3.131 |
| H(14) | C(2) | 2.712 | H(14) | C(3) | 3.318 |
| H(14) | C(13) | 2.790 | H(14) | C(14) | 2.053 |
| H(14) | C(15) | 3.130 | H(14) | H(1) | 3.576 |
| H(14) | H(7)[6)] | 3.255 | H(14) | H(8)[3)] | 3.499 |
| H(14) | H(10) | 3.309 | H(14) | H(13) | 1.600 |
| H(14) | H(13)[4)] | 3.366 | H(14) | H(15) | 1.600 |
| H(15) | Cl(1)[4)] | 3.157 | H(15) | C(9)[6)] | 3.534 |
| H(15) | C(10)[6)] | 3.483 | H(15) | C(13) | 3.324 |
| H(15) | C(14) | 2.053 | H(15) | C(15) | 2.555 |
| H(15) | H(6)[6)] | 2.984 | H(15) | H(7)[6)] | 2.898 |
| H(15) | H(8)[10)] | 3.002 | H(15) | H(9)[10)] | 3.462 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(15) | H(10) | 2.341 | H(15) | H(13) | 1.600 |
| H(15) | H(13)[4)] | 3.306 | H(15) | H(14) | 1.600 |
| H(16) | F(2)[8)] | 2.815 | H(16) | C(5)[8)] | 3.267 |
| H(16) | C(6)[8)] | 3.463 | H(16) | C(13) | 3.330 |
| H(16) | C(17) | 2.574 | H(16) | C(17)[8)] | 3.580 |
| H(16) | C(18) | 2.047 | H(16) | C(18)[8)] | 3.520 |
| H(16) | C(20)[8)] | 3.211 | H(16) | H(3)[8)] | 2.574 |
| H(16) | H(12) | 2.372 | H(16) | H(12)[8)] | 3.300 |
| H(16) | H(16)[8)] | 2.831 | H(16) | H(17) | 1.600 |
| H(16) | H(17)[8)] | 2.947 | H(16) | H(18) | 1.600 |
| H(17) | V(1) | 3.510 | H(17) | Cl(2) | 3.117 |
| H(17) | F(2) | 2.490 | H(17) | N(3) | 2.710 |
| H(17) | C(6) | 3.322 | H(17) | C(13) | 2.757 |
| H(17) | C(17) | 3.168 | H(17) | C(17)[8)] | 3.554 |
| H(17) | C(18) | 2.047 | H(17) | H(12) | 3.376 |
| H(17) | H(12)[8)] | 2.889 | H(17) | H(16) | 1.600 |
| H(17) | H(16)[8)] | 2.947 | H(17) | H(18) | 1.600 |
| H(18) | Cl(2) | 3.354 | H(18) | F(2)[1)] | 3.289 |
| H(18) | N(3) | 2.902 | H(18) | C(1)[1)] | 3.591 |
| H(18) | C(5)[1)] | 3.247 | H(18) | C(6)[1)] | 3.086 |
| H(18) | C(13) | 2.798 | H(18) | C(17) | 3.115 |
| H(18) | C(18) | 2.047 | H(18) | C(23)[5)] | 3.392 |
| H(18) | H(3)[1)] | 3.444 | H(18) | H(3)[8)] | 3.212 |
| H(18) | H(4)[1)] | 3.355 | H(18) | H(12) | 3.312 |
| H(18) | H(16) | 1.600 | H(18) | H(17) | 1.600 |

Symmetry Operators:

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

X-ray Structure Report for V(N=2,6-$Me_2C_6H_3$)($CH_2SiMe_3$)$_2$[2-(2,6-$Me_2C_6H_3$)$CH_2$($C_5H_4N$)] (**2a**)

December 8, 2008

*Experimental*

Data Collection

A red block crystal of $C_{30}H_{46}N_3Si_2V$ having approximate dimensions of 0.50 x 0.40 x 0.40 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K$\alpha$ radiation.

Indexing was performed from 3 oscillations that were exposed for 140 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 = 15.9173(4) Å
b = 11.9182(3) Å  $\beta$ = 103.6830(7)$^o$
c = 17.7030(4) Å
V = 3263.05(13) Å$^3$

For Z = 4 and F.W. = 555.83, the calculated density is 1.131 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 -80 ± 1$^o$C to a maximum 2$\theta$ value of 50.7$^o$. A total of 112 oscillation images were collected. A sweep of data was done using $\omega$ scans from 130.0 to 190.0$^o$ in 2.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 0.0$^o$. The exposure rate was 190.0 [sec./$^o$]. A second sweep was performed using $\omega$ scans from 0.0 to 164.0$^o$ in 2.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 210.0$^o$. The exposure rate was 190.0 [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 26522 reflections that were collected, 5936 were unique ($R_{int}$ = 0.022); equivalent reflections were merged.

The linear absorption coefficient, $\mu$, for Mo-K$\alpha$ radiation is 3.985 $cm^{-1}$. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.581 to 0.853. 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 5233 observed reflections and 371 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0372$$

$$wR2 = [ \Sigma \, ( w \, (Fo^2 - Fc^2)^2 ) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.1323$$

The standard deviation of an observation of unit weight[4] was 1.01. A Sheldrick weighting scheme was used. Plots of $\Sigma$ w $(|Fo| - |Fc|)^2$ versus |Fo|, reflection order in data collection, sin $\theta/\lambda$ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.51 and -0.30 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta$f' and $\Delta$f'' 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).

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(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 | $C_{30}H_{46}N_3Si_2V$ |
| Formula Weight | 555.83 |
| Crystal Color, Habit | red, block |
| Crystal Dimensions | 0.50 X 0.40 X 0.40 mm |
| Crystal System | monoclinic |
| Lattice Type | Primitive |
| Indexing Images | 3 oscillations @ 140.0 seconds |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| Lattice Parameters | a = 15.9173(4) Å<br>b = 11.9182(3) Å<br>c = 17.7030(4) Å<br>$\beta$ = 103.6830(7) $^o$<br>V = 3263.05(13) Å$^3$ |
| Space Group | $P2_1/c$ (#14) |
| Z value | 4 |
| $D_{calc}$ | 1.131 g/cm$^3$ |
| $F_{000}$ | 1192.00 |
| $\mu$(MoK$\alpha$) | 3.985 cm$^{-1}$ |

B. Intensity Measurements

| | |
|---|---|
| Diffractometer | Rigaku RAXIS-RAPID |
| Radiation | MoK$\alpha$ ($\lambda$ = 0.71075 Å)<br>graphite monochromated |
| Detector Aperture | 280 mm x 256 mm |
| Data Images | 112 exposures |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=0.0) | 130.0 - 190.0$^{o}$ |
| Exposure Rate | 190.0 sec./$^{o}$ |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=210.0) | 0.0 - 164.0$^{o}$ |
| Exposure Rate | 190.0 sec./$^{o}$ |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| $2\theta_{max}$ | 50.7$^{o}$ |
| No. of Reflections Measured | Total: 26522<br>Unique: 5936 ($R_{int}$ = 0.022) |
| Corrections | Lorentz-polarization<br>Absorption<br>(trans. factors: 0.581 - 0.853) |

C. Structure Solution and Refinement

| | |
|---|---|
| Structure Solution | Direct Methods (SIR92) |
| Refinement | Full-matrix least-squares on $F^2$ |
| Function Minimized | $\Sigma$ w $(Fo^2 - Fc^2)^2$ |
| Least Squares Weights | $1/[0.0035Fo^2+1.0000\sigma(Fo^2)]/(4Fo^2)$ |
| $2\theta_{max}$ cutoff | $50.7^o$ |
| Anomalous Dispersion | All non-hydrogen atoms |
| No. Observations (I>2.00$\sigma$(I)) | 5233 |
| No. Variables | 371 |
| Reflection/Parameter Ratio | 14.11 |
| Residuals: R1 (I>2.00$\sigma$(I)) | 0.0372 |
| Residuals: wR2 (I>2.00$\sigma$(I)) | 0.1323 |
| Goodness of Fit Indicator | 1.008 |
| Max Shift/Error in Final Cycle | 0.000 |
| Maximum peak in Final Diff. Map | 0.51 $e^-/Å^3$ |
| Minimum peak in Final Diff. Map | -0.30 $e^-/Å^3$ |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| V(1) | 0.73681(2) | 0.05304(2) | 0.716240(10) | 2.142(7) |
| Si(1) | 0.85832(4) | -0.05171(4) | 0.58422(3) | 3.300(12) |
| Si(2) | 0.82580(4) | -0.02203(5) | 0.90525(3) | 3.642(12) |
| N(1) | 0.62918(9) | 0.08907(12) | 0.64756(8) | 2.56(2) |
| N(2) | 0.68027(9) | -0.11734(11) | 0.67387(8) | 2.68(2) |
| N(3) | 0.77042(9) | 0.18497(12) | 0.73959(8) | 2.61(2) |
| C(1) | 0.57561(12) | 0.00630(15) | 0.59725(12) | 3.47(4) |
| C(2) | 0.60810(12) | -0.10997(14) | 0.61729(11) | 2.99(3) |
| C(3) | 0.56784(14) | -0.20393(16) | 0.57782(13) | 4.06(4) |
| C(4) | 0.60300(15) | -0.30765(17) | 0.59744(13) | 4.25(4) |
| C(5) | 0.67739(15) | -0.31690(16) | 0.65716(12) | 3.93(4) |
| C(6) | 0.71307(12) | -0.22076(15) | 0.69357(11) | 3.22(3) |
| C(7) | 0.58860(11) | 0.19635(14) | 0.63618(11) | 2.88(3) |
| C(8) | 0.52098(14) | 0.22050(19) | 0.67268(15) | 4.73(5) |
| C(9) | 0.48100(18) | 0.3241(2) | 0.65976(19) | 6.44(7) |
| C(10) | 0.50636(17) | 0.4038(2) | 0.61308(18) | 5.73(6) |
| C(11) | 0.57207(16) | 0.38000(16) | 0.57820(13) | 4.40(4) |
| C(12) | 0.61365(12) | 0.27666(15) | 0.58836(10) | 3.08(3) |
| C(13) | 0.4930(2) | 0.1363(2) | 0.7252(2) | 7.69(9) |
| C(14) | 0.68454(16) | 0.25185(18) | 0.54750(13) | 4.35(4) |
| C(15) | 0.79475(13) | 0.29628(15) | 0.75570(10) | 3.28(3) |
| C(16) | 0.75058(18) | 0.36307(18) | 0.80007(12) | 4.81(5) |
| C(17) | 0.7774(2) | 0.4756(2) | 0.81391(15) | 6.53(7) |
| C(18) | 0.8442(2) | 0.5210(2) | 0.7853(2) | 7.27(7) |
| C(19) | 0.88455(19) | 0.4549(2) | 0.7432(2) | 6.54(7) |
| C(20) | 0.86270(14) | 0.34339(18) | 0.72756(16) | 4.77(5) |
| C(21) | 0.6810(2) | 0.3172(2) | 0.83238(18) | 7.63(9) |
| C(22) | 0.91254(18) | 0.2735(2) | 0.6825(2) | 7.10(8) |
| C(23) | 0.84375(11) | -0.01064(15) | 0.68256(10) | 2.81(3) |
| C(24) | 0.75669(15) | -0.1002(2) | 0.51494(12) | 4.31(4) |
| C(25) | 0.90224(19) | 0.0675(2) | 0.53573(16) | 5.49(6) |
| C(26) | 0.93643(17) | -0.1718(2) | 0.59607(16) | 5.37(6) |
| C(27) | 0.72712(11) | 0.00143(16) | 0.82648(10) | 3.01(3) |
| C(28) | 0.7956(2) | -0.0434(2) | 1.00012(14) | 6.51(7) |
| C(29) | 0.88107(16) | -0.1567(2) | 0.88725(13) | 5.18(5) |
| C(30) | 0.9009(2) | 0.0979(3) | 0.9135(2) | 9.97(10) |

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

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| H(1) | 0.5131 | 0.0122 | 0.6037 | 3.89 |
| H(2) | 0.5765 | 0.0228 | 0.5403 | 3.83 |
| H(3) | 0.5128 | -0.1955 | 0.5348 | 4.53 |
| H(4) | 0.5756 | -0.3760 | 0.5693 | 4.87 |
| H(5) | 0.7044 | -0.3925 | 0.6732 | 4.64 |
| H(6) | 0.7650 | -0.2273 | 0.7360 | 3.77 |
| H(7) | 0.4301 | 0.3427 | 0.6861 | 8.00 |
| H(8) | 0.4768 | 0.4784 | 0.6049 | 6.71 |
| H(9) | 0.5910 | 0.4391 | 0.5440 | 5.08 |
| H(10) | 0.5388 | 0.0835 | 0.7436 | 10.04 |
| H(11) | 0.4796 | 0.1745 | 0.7687 | 10.06 |
| H(12) | 0.4426 | 0.0972 | 0.6969 | 10.05 |
| H(13) | 0.6828 | 0.1747 | 0.5338 | 5.43 |
| H(14) | 0.6765 | 0.2964 | 0.5018 | 5.44 |
| H(15) | 0.7390 | 0.2689 | 0.5811 | 5.45 |
| H(16) | 0.7471 | 0.5247 | 0.8456 | 7.26 |
| H(17) | 0.8635 | 0.6083 | 0.7967 | 7.67 |
| H(18) | 0.9347 | 0.4894 | 0.7214 | 7.22 |
| H(19) | 0.6717 | 0.3659 | 0.8736 | 9.76 |
| H(20) | 0.6282 | 0.3125 | 0.7917 | 9.77 |
| H(21) | 0.6970 | 0.2429 | 0.8532 | 9.79 |
| H(22) | 0.8728 | 0.2269 | 0.6455 | 8.86 |
| H(23) | 0.9438 | 0.3223 | 0.6549 | 8.90 |
| H(24) | 0.9532 | 0.2261 | 0.7181 | 8.89 |
| H(25) | 0.8901 | 0.0476 | 0.7012 | 3.45 |
| H(26) | 0.8585 | -0.0811 | 0.7146 | 3.42 |
| H(27) | 0.7149 | -0.0384 | 0.5045 | 5.24 |
| H(28) | 0.7700 | -0.1243 | 0.4662 | 5.21 |
| H(29) | 0.7320 | -0.1632 | 0.5379 | 5.17 |
| H(30) | 0.9591 | 0.0920 | 0.5691 | 7.05 |
| H(31) | 0.9103 | 0.0423 | 0.4840 | 7.09 |
| H(32) | 0.8607 | 0.1317 | 0.5282 | 7.09 |
| H(33) | 0.9137 | -0.2360 | 0.6217 | 6.42 |
| H(34) | 0.9437 | -0.1958 | 0.5438 | 6.48 |
| H(35) | 0.9936 | -0.1476 | 0.6290 | 6.46 |
| H(36) | 0.6912 | 0.0616 | 0.8465 | 3.74 |
| H(37) | 0.6936 | -0.0733 | 0.8190 | 3.72 |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| H(38) | 0.8524 | -0.0535 | 1.0450 | 7.95 |
| H(39) | 0.7606 | 0.0273 | 1.0123 | 7.94 |
| H(40) | 0.7566 | -0.1162 | 0.9968 | 7.95 |
| H(41) | 0.9325 | -0.1702 | 0.9312 | 6.19 |
| H(42) | 0.8397 | -0.2208 | 0.8836 | 6.22 |
| H(43) | 0.9001 | -0.1503 | 0.8375 | 6.17 |
| H(44) | 0.8693 | 0.1726 | 0.9258 | 10.45 |
| H(45) | 0.9567 | 0.0826 | 0.9594 | 10.49 |
| H(46) | 0.9201 | 0.1082 | 0.8599 | 10.49 |

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

Table 3. Anisotropic displacement parameters

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| V(1) | 0.02481(19) | 0.0288(2) | 0.0272(2) | 0.00015(10) | 0.00512(13) | -0.00051(9) |
| Si(1) | 0.0439(3) | 0.0446(3) | 0.0411(3) | 0.0060(2) | 0.0184(2) | -0.0033(2) |
| Si(2) | 0.0540(3) | 0.0465(3) | 0.0325(2) | -0.0058(2) | -0.0006(2) | 0.0065(2) |
| N(1) | 0.0289(7) | 0.0273(7) | 0.0389(7) | -0.0005(5) | 0.0037(5) | 0.0025(5) |
| N(2) | 0.0330(7) | 0.0300(7) | 0.0366(7) | 0.0003(5) | 0.0040(6) | 0.0024(5) |
| N(3) | 0.0348(7) | 0.0345(7) | 0.0279(6) | -0.0003(6) | 0.0037(5) | -0.0030(5) |
| C(1) | 0.0321(9) | 0.0335(9) | 0.0564(12) | -0.0039(7) | -0.0092(8) | 0.0044(8) |
| C(2) | 0.0343(9) | 0.0304(8) | 0.0449(9) | -0.0044(7) | 0.0010(7) | 0.0044(7) |
| C(3) | 0.0502(11) | 0.0364(10) | 0.0561(11) | -0.0110(8) | -0.0104(9) | 0.0039(8) |
| C(4) | 0.0625(13) | 0.0313(9) | 0.0603(12) | -0.0102(8) | -0.0006(10) | -0.0006(8) |
| C(5) | 0.0619(12) | 0.0305(8) | 0.0537(11) | 0.0053(8) | 0.0072(9) | 0.0049(8) |
| C(6) | 0.0416(9) | 0.0349(9) | 0.0426(10) | 0.0052(7) | 0.0038(8) | 0.0058(7) |
| C(7) | 0.0315(8) | 0.0308(8) | 0.0440(9) | 0.0030(6) | 0.0030(7) | 0.0015(7) |
| C(8) | 0.0443(11) | 0.0533(12) | 0.0887(17) | 0.0166(9) | 0.0289(11) | 0.0174(11) |
| C(9) | 0.0647(16) | 0.0710(17) | 0.118(2) | 0.0343(13) | 0.0389(16) | 0.0136(15) |
| C(10) | 0.0726(16) | 0.0422(11) | 0.098(2) | 0.0245(11) | 0.0093(14) | 0.0073(12) |
| C(11) | 0.0734(14) | 0.0288(9) | 0.0581(12) | 0.0005(9) | 0.0017(11) | 0.0033(8) |
| C(12) | 0.0452(10) | 0.0297(8) | 0.0382(9) | -0.0040(7) | 0.0018(7) | -0.0006(6) |
| C(13) | 0.0775(19) | 0.090(2) | 0.150(3) | 0.0252(16) | 0.079(2) | 0.039(2) |
| C(14) | 0.0769(15) | 0.0453(11) | 0.0493(11) | -0.0057(10) | 0.0271(11) | 0.0052(9) |
| C(15) | 0.0492(10) | 0.0325(9) | 0.0330(9) | 0.0016(7) | -0.0100(7) | -0.0046(6) |
| C(16) | 0.1005(18) | 0.0437(11) | 0.0306(9) | 0.0184(11) | -0.0006(10) | -0.0052(8) |
| C(17) | 0.134(2) | 0.0444(12) | 0.0501(14) | 0.0293(16) | -0.0176(16) | -0.0165(10) |
| C(18) | 0.100(2) | 0.0384(12) | 0.105(2) | 0.0009(14) | -0.042(2) | -0.0071(14) |
| C(19) | 0.0605(16) | 0.0430(13) | 0.124(2) | -0.0148(11) | -0.0204(16) | 0.0020(13) |
| C(20) | 0.0424(11) | 0.0415(11) | 0.0863(16) | -0.0130(9) | -0.0072(10) | -0.0012(10) |
| C(21) | 0.164(3) | 0.0727(17) | 0.0721(18) | 0.0323(19) | 0.065(2) | -0.0040(14) |
| C(22) | 0.0532(14) | 0.0659(16) | 0.162(3) | -0.0205(12) | 0.0481(17) | -0.0037(18) |
| C(23) | 0.0318(8) | 0.0391(9) | 0.0362(9) | 0.0018(7) | 0.0089(7) | -0.0025(7) |
| C(24) | 0.0645(13) | 0.0644(13) | 0.0354(10) | 0.0030(10) | 0.0129(9) | -0.0051(9) |
| C(25) | 0.0805(18) | 0.0758(16) | 0.0678(15) | -0.0079(13) | 0.0481(14) | 0.0008(12) |
| C(26) | 0.0672(15) | 0.0695(15) | 0.0675(15) | 0.0219(12) | 0.0164(12) | -0.0171(12) |
| C(27) | 0.0372(9) | 0.0446(9) | 0.0343(9) | 0.0010(7) | 0.0119(7) | 0.0039(7) |
| C(28) | 0.138(2) | 0.0758(17) | 0.0373(12) | 0.0444(16) | 0.0275(15) | 0.0095(10) |
| C(29) | 0.0566(13) | 0.0940(19) | 0.0451(12) | 0.0266(13) | 0.0097(10) | 0.0132(11) |
| C(30) | 0.130(3) | 0.101(2) | 0.101(2) | -0.069(2) | -0.067(2) | 0.045(2) |

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) | N(1) | 1.9006(13) | V(1) | N(2) | 2.2752(13) |
| V(1) | N(3) | 1.6807(14) | V(1) | C(23) | 2.0758(19) |
| V(1) | C(27) | 2.0864(18) | Si(1) | C(23) | 1.8749(19) |
| Si(1) | C(24) | 1.875(2) | Si(1) | C(25) | 1.878(2) |
| Si(1) | C(26) | 1.875(2) | Si(2) | C(27) | 1.8584(16) |
| Si(2) | C(28) | 1.871(3) | Si(2) | C(29) | 1.894(2) |
| Si(2) | C(30) | 1.847(3) | N(1) | C(1) | 1.461(2) |
| N(1) | C(7) | 1.425(2) | N(2) | C(2) | 1.337(2) |
| N(2) | C(6) | 1.352(2) | N(3) | C(15) | 1.392(2) |
| C(1) | C(2) | 1.492(2) | C(2) | C(3) | 1.393(2) |
| C(3) | C(4) | 1.367(2) | C(4) | C(5) | 1.393(2) |
| C(5) | C(6) | 1.370(2) | C(7) | C(8) | 1.410(3) |
| C(7) | C(12) | 1.397(2) | C(8) | C(9) | 1.384(3) |
| C(8) | C(13) | 1.506(4) | C(9) | C(10) | 1.380(4) |
| C(10) | C(11) | 1.364(4) | C(11) | C(12) | 1.390(2) |
| C(12) | C(14) | 1.507(3) | C(15) | C(16) | 1.416(3) |
| C(15) | C(20) | 1.410(3) | C(16) | C(17) | 1.411(3) |
| C(16) | C(21) | 1.468(4) | C(17) | C(18) | 1.391(5) |
| C(18) | C(19) | 1.348(5) | C(19) | C(20) | 1.385(3) |
| C(20) | C(22) | 1.503(4) | | | |

Table 5. Bond lengths involving hydrogens (Å)

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(1) | H(1) | 1.030 | C(1) | H(2) | 1.030 |
| C(3) | H(3) | 1.020 | C(4) | H(4) | 1.000 |
| C(5) | H(5) | 1.010 | C(6) | H(6) | 0.980 |
| C(9) | H(7) | 1.050 | C(10) | H(8) | 1.000 |
| C(11) | H(9) | 1.020 | C(13) | H(10) | 0.960 |
| C(13) | H(11) | 0.960 | C(13) | H(12) | 0.960 |
| C(14) | H(13) | 0.950 | C(14) | H(14) | 0.950 |
| C(14) | H(15) | 0.950 | C(17) | H(16) | 1.010 |
| C(18) | H(17) | 1.090 | C(19) | H(18) | 1.050 |
| C(21) | H(19) | 0.970 | C(21) | H(20) | 0.970 |
| C(21) | H(21) | 0.970 | C(22) | H(22) | 0.970 |
| C(22) | H(23) | 0.970 | C(22) | H(24) | 0.970 |
| C(23) | H(25) | 1.010 | C(23) | H(26) | 1.010 |
| C(24) | H(27) | 0.980 | C(24) | H(28) | 0.980 |
| C(24) | H(29) | 0.980 | C(25) | H(30) | 1.000 |
| C(25) | H(31) | 1.000 | C(25) | H(32) | 1.000 |
| C(26) | H(33) | 1.000 | C(26) | H(34) | 1.000 |
| C(26) | H(35) | 1.000 | C(27) | H(36) | 1.030 |
| C(27) | H(37) | 1.030 | C(28) | H(38) | 1.060 |
| C(28) | H(39) | 1.060 | C(28) | H(40) | 1.060 |
| C(29) | H(41) | 1.000 | C(29) | H(42) | 1.000 |
| C(29) | H(43) | 1.000 | C(30) | H(44) | 1.070 |
| C(30) | H(45) | 1.070 | C(30) | H(46) | 1.070 |

Table 6. Bond angles (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| N(1) | V(1) | N(2) | 76.37(5) | N(1) | V(1) | N(3) | 97.60(6) |
| N(1) | V(1) | C(23) | 125.11(6) | N(1) | V(1) | C(27) | 114.21(6) |
| N(2) | V(1) | N(3) | 173.78(5) | N(2) | V(1) | C(23) | 82.32(6) |
| N(2) | V(1) | C(27) | 86.23(6) | N(3) | V(1) | C(23) | 100.24(7) |
| N(3) | V(1) | C(27) | 97.78(7) | C(23) | V(1) | C(27) | 114.08(6) |
| C(23) | Si(1) | C(24) | 114.25(9) | C(23) | Si(1) | C(25) | 111.59(10) |
| C(23) | Si(1) | C(26) | 108.65(10) | C(24) | Si(1) | C(25) | 106.91(10) |
| C(24) | Si(1) | C(26) | 106.54(10) | C(25) | Si(1) | C(26) | 108.66(13) |
| C(27) | Si(2) | C(28) | 110.05(12) | C(27) | Si(2) | C(29) | 110.19(9) |
| C(27) | Si(2) | C(30) | 111.02(12) | C(28) | Si(2) | C(29) | 105.30(12) |
| C(28) | Si(2) | C(30) | 109.51(15) | C(29) | Si(2) | C(30) | 110.61(15) |
| V(1) | N(1) | C(1) | 123.04(11) | V(1) | N(1) | C(7) | 127.08(10) |
| C(1) | N(1) | C(7) | 109.88(12) | V(1) | N(2) | C(2) | 113.03(10) |
| V(1) | N(2) | C(6) | 129.07(10) | C(2) | N(2) | C(6) | 117.67(13) |
| V(1) | N(3) | C(15) | 176.96(11) | N(1) | C(1) | C(2) | 111.26(14) |
| N(2) | C(2) | C(1) | 115.30(14) | N(2) | C(2) | C(3) | 122.34(15) |
| C(1) | C(2) | C(3) | 122.33(15) | C(2) | C(3) | C(4) | 119.15(17) |
| C(3) | C(4) | C(5) | 119.19(18) | C(4) | C(5) | C(6) | 118.31(17) |
| N(2) | C(6) | C(5) | 123.32(15) | N(1) | C(7) | C(8) | 119.21(17) |
| N(1) | C(7) | C(12) | 121.04(17) | C(8) | C(7) | C(12) | 119.74(17) |
| C(7) | C(8) | C(9) | 118.5(2) | C(7) | C(8) | C(13) | 121.1(2) |
| C(9) | C(8) | C(13) | 120.4(2) | C(8) | C(9) | C(10) | 121.7(2) |
| C(9) | C(10) | C(11) | 119.5(2) | C(10) | C(11) | C(12) | 121.2(2) |
| C(7) | C(12) | C(11) | 119.36(19) | C(7) | C(12) | C(14) | 120.66(16) |
| C(11) | C(12) | C(14) | 119.98(18) | N(3) | C(15) | C(16) | 119.90(19) |
| N(3) | C(15) | C(20) | 120.43(18) | C(16) | C(15) | C(20) | 119.67(18) |
| C(15) | C(16) | C(17) | 117.5(2) | C(15) | C(16) | C(21) | 121.8(2) |
| C(17) | C(16) | C(21) | 120.8(2) | C(16) | C(17) | C(18) | 122.2(2) |
| C(17) | C(18) | C(19) | 118.6(2) | C(18) | C(19) | C(20) | 122.8(3) |
| C(15) | C(20) | C(19) | 119.3(2) | C(15) | C(20) | C(22) | 120.8(2) |
| C(19) | C(20) | C(22) | 119.9(2) | V(1) | C(23) | Si(1) | 130.70(8) |
| V(1) | C(27) | Si(2) | 120.65(9) | | | | |

Table 7. Bond angles involving hydrogens (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| N(1) | C(1) | H(1) | 109.0 | N(1) | C(1) | H(2) | 109.0 |
| C(2) | C(1) | H(1) | 109.0 | C(2) | C(1) | H(2) | 109.0 |
| H(1) | C(1) | H(2) | 109.5 | C(2) | C(3) | H(3) | 120.4 |
| C(4) | C(3) | H(3) | 120.4 | C(3) | C(4) | H(4) | 120.4 |
| C(5) | C(4) | H(4) | 120.4 | C(4) | C(5) | H(5) | 120.8 |
| C(6) | C(5) | H(5) | 120.8 | N(2) | C(6) | H(6) | 118.3 |
| C(5) | C(6) | H(6) | 118.3 | C(8) | C(9) | H(7) | 119.2 |
| C(10) | C(9) | H(7) | 119.2 | C(9) | C(10) | H(8) | 120.3 |
| C(11) | C(10) | H(8) | 120.3 | C(10) | C(11) | H(9) | 119.4 |
| C(12) | C(11) | H(9) | 119.4 | C(8) | C(13) | H(10) | 109.5 |
| C(8) | C(13) | H(11) | 109.5 | C(8) | C(13) | H(12) | 109.5 |
| H(10) | C(13) | H(11) | 109.5 | H(10) | C(13) | H(12) | 109.5 |
| H(11) | C(13) | H(12) | 109.5 | C(12) | C(14) | H(13) | 109.5 |
| C(12) | C(14) | H(14) | 109.5 | C(12) | C(14) | H(15) | 109.5 |
| H(13) | C(14) | H(14) | 109.5 | H(13) | C(14) | H(15) | 109.5 |
| H(14) | C(14) | H(15) | 109.5 | C(16) | C(17) | H(16) | 118.9 |
| C(18) | C(17) | H(16) | 118.9 | C(17) | C(18) | H(17) | 120.7 |
| C(19) | C(18) | H(17) | 120.7 | C(18) | C(19) | H(18) | 118.6 |
| C(20) | C(19) | H(18) | 118.6 | C(16) | C(21) | H(19) | 109.5 |
| C(16) | C(21) | H(20) | 109.5 | C(16) | C(21) | H(21) | 109.5 |
| H(19) | C(21) | H(20) | 109.5 | H(19) | C(21) | H(21) | 109.5 |
| H(20) | C(21) | H(21) | 109.5 | C(20) | C(22) | H(22) | 109.5 |
| C(20) | C(22) | H(23) | 109.5 | C(20) | C(22) | H(24) | 109.5 |
| H(22) | C(22) | H(23) | 109.5 | H(22) | C(22) | H(24) | 109.5 |
| H(23) | C(22) | H(24) | 109.5 | V(1) | C(23) | H(25) | 103.9 |
| V(1) | C(23) | H(26) | 103.9 | Si(1) | C(23) | H(25) | 103.9 |
| Si(1) | C(23) | H(26) | 103.9 | H(25) | C(23) | H(26) | 109.5 |
| Si(1) | C(24) | H(27) | 109.5 | Si(1) | C(24) | H(28) | 109.5 |
| Si(1) | C(24) | H(29) | 109.5 | H(27) | C(24) | H(28) | 109.5 |
| H(27) | C(24) | H(29) | 109.5 | H(28) | C(24) | H(29) | 109.5 |
| Si(1) | C(25) | H(30) | 109.5 | Si(1) | C(25) | H(31) | 109.5 |
| Si(1) | C(25) | H(32) | 109.5 | H(30) | C(25) | H(31) | 109.5 |
| H(30) | C(25) | H(32) | 109.5 | H(31) | C(25) | H(32) | 109.5 |
| Si(1) | C(26) | H(33) | 109.5 | Si(1) | C(26) | H(34) | 109.5 |
| Si(1) | C(26) | H(35) | 109.5 | H(33) | C(26) | H(34) | 109.5 |
| H(33) | C(26) | H(35) | 109.5 | H(34) | C(26) | H(35) | 109.5 |
| V(1) | C(27) | H(36) | 106.6 | V(1) | C(27) | H(37) | 106.6 |

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

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| Si(2) | C(27) | H(36) | 106.6 | Si(2) | C(27) | H(37) | 106.6 |
| H(36) | C(27) | H(37) | 109.5 | Si(2) | C(28) | H(38) | 109.5 |
| Si(2) | C(28) | H(39) | 109.5 | Si(2) | C(28) | H(40) | 109.5 |
| H(38) | C(28) | H(39) | 109.5 | H(38) | C(28) | H(40) | 109.5 |
| H(39) | C(28) | H(40) | 109.5 | Si(2) | C(29) | H(41) | 109.5 |
| Si(2) | C(29) | H(42) | 109.5 | Si(2) | C(29) | H(43) | 109.5 |
| H(41) | C(29) | H(42) | 109.5 | H(41) | C(29) | H(43) | 109.5 |
| H(42) | C(29) | H(43) | 109.5 | Si(2) | C(30) | H(44) | 109.5 |
| Si(2) | C(30) | H(45) | 109.5 | Si(2) | C(30) | H(46) | 109.5 |
| H(44) | C(30) | H(45) | 109.5 | H(44) | C(30) | H(46) | 109.5 |
| H(45) | C(30) | H(46) | 109.5 | | | | |

Table 8. Torsion Angles(°)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| N(1) | V(1) | N(2) | C(2) | 7.37(13) |
| N(2) | V(1) | N(1) | C(1) | -9.50(14) |
| N(1) | V(1) | N(3) | C(15) | -12(2) |
| N(3) | V(1) | N(1) | C(7) | -10.68(16) |
| C(23) | V(1) | N(1) | C(1) | 60.70(17) |
| N(1) | V(1) | C(27) | Si(2) | -173.09(9) |
| C(27) | V(1) | N(1) | C(7) | 91.43(16) |
| N(3) | V(1) | N(2) | C(2) | -7.0(7) |
| N(2) | V(1) | C(23) | Si(1) | 64.30(11) |
| C(23) | V(1) | N(2) | C(6) | 52.55(17) |
| C(27) | V(1) | N(2) | C(2) | 123.40(14) |
| N(3) | V(1) | C(23) | Si(1) | -109.95(12) |
| N(3) | V(1) | C(27) | Si(2) | -71.09(11) |
| C(23) | V(1) | C(27) | Si(2) | 33.83(13) |
| C(24) | Si(1) | C(23) | V(1) | -28.67(16) |
| C(26) | Si(1) | C(23) | V(1) | -147.46(13) |
| C(29) | Si(2) | C(27) | V(1) | -73.93(14) |
| V(1) | N(1) | C(1) | C(2) | 10.2(2) |
| V(1) | N(1) | C(7) | C(12) | 80.6(2) |
| C(1) | N(1) | C(7) | C(12) | -99.07(18) |
| V(1) | N(2) | C(2) | C(1) | -4.2(2) |
| V(1) | N(2) | C(6) | C(5) | -172.20(16) |
| C(6) | N(2) | C(2) | C(1) | -179.15(18) |
| V(1) | N(3) | C(15) | C(16) | 82(2) |
| N(1) | C(1) | C(2) | N(2) | -2.5(2) |
| N(2) | C(2) | C(3) | C(4) | 0.1(2) |
| C(2) | C(3) | C(4) | C(5) | 1.0(3) |
| C(4) | C(5) | C(6) | N(2) | -0.8(3) |
| N(1) | C(7) | C(8) | C(13) | 1.9(2) |
| N(1) | C(7) | C(12) | C(14) | -0.1(2) |
| C(8) | C(7) | C(12) | C(14) | -178.78(17) |
| C(12) | C(7) | C(8) | C(13) | -179.4(2) |
| C(13) | C(8) | C(9) | C(10) | 178.8(2) |
| C(9) | C(10) | C(11) | C(12) | 0.4(3) |
| C(10) | C(11) | C(12) | C(14) | 178.5(2) |
| N(3) | C(15) | C(16) | C(21) | 2.2(2) |
| N(3) | C(15) | C(20) | C(22) | -2.5(3) |
| N(1) | V(1) | N(2) | C(6) | -178.41(17) |
| N(2) | V(1) | N(1) | C(7) | 170.87(16) |
| N(3) | V(1) | N(1) | C(1) | 168.94(15) |
| N(1) | V(1) | C(23) | Si(1) | -3.02(16) |
| C(23) | V(1) | N(1) | C(7) | -118.92(15) |
| C(27) | V(1) | N(1) | C(1) | -88.95(16) |
| N(2) | V(1) | N(3) | C(15) | 2(2) |
| N(3) | V(1) | N(2) | C(6) | 167.2(5) |
| C(23) | V(1) | N(2) | C(2) | -121.68(14) |
| N(2) | V(1) | C(27) | Si(2) | 113.68(10) |
| C(27) | V(1) | N(2) | C(6) | -62.38(17) |
| C(23) | V(1) | N(3) | C(15) | 115(2) |
| C(27) | V(1) | N(3) | C(15) | -128(2) |
| C(27) | V(1) | C(23) | Si(1) | 146.66(11) |
| C(25) | Si(1) | C(23) | V(1) | 92.75(14) |
| C(28) | Si(2) | C(27) | V(1) | 170.38(11) |
| C(30) | Si(2) | C(27) | V(1) | 48.97(18) |
| V(1) | N(1) | C(7) | C(8) | -100.68(18) |
| C(1) | N(1) | C(7) | C(8) | 79.7(2) |
| C(7) | N(1) | C(1) | C(2) | -170.13(17) |
| V(1) | N(2) | C(2) | C(3) | 173.50(17) |
| C(2) | N(2) | C(6) | C(5) | 1.8(3) |
| C(6) | N(2) | C(2) | C(3) | -1.4(3) |
| V(1) | N(3) | C(15) | C(20) | -98(2) |
| N(1) | C(1) | C(2) | C(3) | 179.8(2) |
| C(1) | C(2) | C(3) | C(4) | 177.6(2) |
| C(3) | C(4) | C(5) | C(6) | -0.7(3) |
| N(1) | C(7) | C(8) | C(9) | -178.66(19) |
| N(1) | C(7) | C(12) | C(11) | 179.48(15) |
| C(8) | C(7) | C(12) | C(11) | 0.8(2) |
| C(12) | C(7) | C(8) | C(9) | 0.1(2) |
| C(7) | C(8) | C(9) | C(10) | -0.7(3) |
| C(8) | C(9) | C(10) | C(11) | 0.5(4) |
| C(10) | C(11) | C(12) | C(7) | -1.0(2) |
| N(3) | C(15) | C(16) | C(17) | -179.35(18) |
| N(3) | C(15) | C(20) | C(19) | 178.8(2) |
| C(16) | C(15) | C(20) | C(19) | -0.6(3) |

Table 8. Torsion angles (°) (continued)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| C(16) | C(15) | C(20) | C(22) | 178.0(2) |
| C(20) | C(15) | C(16) | C(21) | -178.4(2) |
| C(21) | C(16) | C(17) | C(18) | 178.8(2) |
| C(17) | C(18) | C(19) | C(20) | -0.4(4) |
| C(18) | C(19) | C(20) | C(22) | -177.9(2) |

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| C(20) | C(15) | C(16) | C(17) | 0.1(2) |
| C(15) | C(16) | C(17) | C(18) | 0.3(3) |
| C(16) | C(17) | C(18) | C(19) | -0.2(3) |
| C(18) | C(19) | C(20) | C(15) | 0.8(4) |

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) | Si(1) | 3.5917(6) | V(1) | Si(2) | 3.4294(5) |
| V(1) | C(1) | 2.9621(17) | V(1) | C(2) | 3.0565(16) |
| V(1) | C(6) | 3.2986(18) | V(1) | C(7) | 2.9849(16) |
| V(1) | C(15) | 3.0721(18) | Si(1) | V(1) | 3.5917(6) |
| Si(2) | V(1) | 3.4294(5) | N(1) | N(2) | 2.5982(19) |
| N(1) | N(3) | 2.6985(18) | N(1) | C(2) | 2.438(2) |
| N(1) | C(8) | 2.445(2) | N(1) | C(12) | 2.457(2) |
| N(1) | C(13) | 2.884(4) | N(1) | C(14) | 2.901(2) |
| N(1) | C(23) | 3.530(2) | N(1) | C(27) | 3.349(2) |
| N(2) | N(1) | 2.5982(19) | N(2) | C(1) | 2.391(2) |
| N(2) | C(3) | 2.392(2) | N(2) | C(4) | 2.775(2) |
| N(2) | C(5) | 2.396(2) | N(2) | C(23) | 2.868(2) |
| N(2) | C(24) | 3.325(2) | N(2) | C(27) | 2.984(2) |
| N(3) | N(1) | 2.6985(18) | N(3) | C(7) | 3.040(2) |
| N(3) | C(12) | 3.381(2) | N(3) | C(14) | 3.445(2) |
| N(3) | C(16) | 2.431(2) | N(3) | C(20) | 2.432(2) |
| N(3) | C(21) | 2.884(4) | N(3) | C(22) | 2.886(3) |
| N(3) | C(23) | 2.894(2) | N(3) | C(27) | 2.851(2) |
| N(3) | C(30) | 3.441(3) | C(1) | V(1) | 2.9621(17) |
| C(1) | N(2) | 2.391(2) | C(1) | C(3) | 2.528(2) |
| C(1) | C(7) | 2.362(2) | C(1) | C(8) | 3.099(3) |
| C(1) | C(12) | 3.289(2) | C(1) | C(13) | 3.267(4) |
| C(2) | V(1) | 3.0565(16) | C(2) | N(1) | 2.438(2) |
| C(2) | C(4) | 2.381(2) | C(2) | C(5) | 2.726(2) |
| C(2) | C(6) | 2.300(2) | C(2) | C(24) | 3.304(3) |
| C(3) | N(2) | 2.392(2) | C(3) | C(1) | 2.528(2) |
| C(3) | C(5) | 2.381(2) | C(3) | C(6) | 2.710(2) |
| C(4) | N(2) | 2.775(2) | C(4) | C(2) | 2.381(2) |
| C(4) | C(6) | 2.372(2) | C(5) | N(2) | 2.396(2) |
| C(5) | C(2) | 2.726(2) | C(5) | C(3) | 2.381(2) |
| C(6) | V(1) | 3.2986(18) | C(6) | C(2) | 2.300(2) |
| C(6) | C(3) | 2.710(2) | C(6) | C(4) | 2.372(2) |
| C(6) | C(23) | 3.291(2) | C(6) | C(27) | 3.514(2) |
| C(7) | V(1) | 2.9849(16) | C(7) | N(3) | 3.040(2) |
| C(7) | C(1) | 2.362(2) | C(7) | C(9) | 2.401(3) |
| C(7) | C(10) | 2.782(2) | C(7) | C(11) | 2.405(2) |
| C(7) | C(13) | 2.540(4) | C(7) | C(14) | 2.524(3) |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(8) | N(1) | 2.445(2) | C(8) | C(1) | 3.099(3) |
| C(8) | C(10) | 2.414(3) | C(8) | C(11) | 2.778(3) |
| C(8) | C(12) | 2.428(3) | C(8) | C(21) | 3.521(3) |
| C(9) | C(7) | 2.401(3) | C(9) | C(11) | 2.371(4) |
| C(9) | C(12) | 2.764(3) | C(9) | C(13) | 2.508(4) |
| C(10) | C(7) | 2.782(2) | C(10) | C(8) | 2.414(3) |
| C(10) | C(12) | 2.400(3) | C(11) | C(7) | 2.405(2) |
| C(11) | C(8) | 2.778(3) | C(11) | C(9) | 2.371(4) |
| C(11) | C(14) | 2.509(3) | C(12) | N(1) | 2.457(2) |
| C(12) | N(3) | 3.381(2) | C(12) | C(1) | 3.289(2) |
| C(12) | C(8) | 2.428(3) | C(12) | C(9) | 2.764(3) |
| C(12) | C(10) | 2.400(3) | C(13) | N(1) | 2.884(4) |
| C(13) | C(1) | 3.267(4) | C(13) | C(7) | 2.540(4) |
| C(13) | C(9) | 2.508(4) | C(14) | N(1) | 2.901(2) |
| C(14) | N(3) | 3.445(2) | C(14) | C(7) | 2.524(3) |
| C(14) | C(11) | 2.509(3) | C(15) | V(1) | 3.0721(18) |
| C(15) | C(17) | 2.416(3) | C(15) | C(18) | 2.806(3) |
| C(15) | C(19) | 2.411(3) | C(15) | C(21) | 2.520(4) |
| C(15) | C(22) | 2.533(4) | C(16) | N(3) | 2.431(2) |
| C(16) | C(18) | 2.453(4) | C(16) | C(19) | 2.788(4) |
| C(16) | C(20) | 2.444(3) | C(17) | C(15) | 2.416(3) |
| C(17) | C(19) | 2.355(5) | C(17) | C(20) | 2.765(4) |
| C(17) | C(21) | 2.503(4) | C(18) | C(15) | 2.806(3) |
| C(18) | C(16) | 2.453(4) | C(18) | C(20) | 2.400(3) |
| C(19) | C(15) | 2.411(3) | C(19) | C(16) | 2.788(4) |
| C(19) | C(17) | 2.355(5) | C(19) | C(22) | 2.501(4) |
| C(20) | N(3) | 2.432(2) | C(20) | C(16) | 2.444(3) |
| C(20) | C(17) | 2.765(4) | C(20) | C(18) | 2.400(3) |
| C(21) | N(3) | 2.884(4) | C(21) | C(8) | 3.521(3) |
| C(21) | C(15) | 2.520(4) | C(21) | C(17) | 2.503(4) |
| C(22) | N(3) | 2.886(3) | C(22) | C(15) | 2.533(4) |
| C(22) | C(19) | 2.501(4) | C(22) | C(23) | 3.559(3) |
| C(22) | C(25) | 3.550(4) | C(23) | N(1) | 3.530(2) |
| C(23) | N(2) | 2.868(2) | C(23) | N(3) | 2.894(2) |
| C(23) | C(6) | 3.291(2) | C(23) | C(22) | 3.559(3) |
| C(23) | C(24) | 3.149(2) | C(23) | C(25) | 3.104(3) |
| C(23) | C(26) | 3.046(3) | C(23) | C(27) | 3.492(2) |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(24) | N(2) | 3.325(2) | C(24) | C(2) | 3.304(3) |
| C(24) | C(23) | 3.149(2) | C(24) | C(25) | 3.016(3) |
| C(24) | C(26) | 3.005(3) | C(25) | C(22) | 3.550(4) |
| C(25) | C(23) | 3.104(3) | C(25) | C(24) | 3.016(3) |
| C(25) | C(26) | 3.049(3) | C(26) | C(23) | 3.046(3) |
| C(26) | C(24) | 3.005(3) | C(26) | C(25) | 3.049(3) |
| C(27) | N(1) | 3.349(2) | C(27) | N(2) | 2.984(2) |
| C(27) | N(3) | 2.851(2) | C(27) | C(6) | 3.514(2) |
| C(27) | C(23) | 3.492(2) | C(27) | C(28) | 3.056(2) |
| C(27) | C(29) | 3.077(3) | C(27) | C(30) | 3.054(4) |
| C(28) | C(27) | 3.056(2) | C(28) | C(29) | 2.993(4) |
| C(28) | C(30) | 3.036(5) | C(29) | C(27) | 3.077(3) |
| C(29) | C(28) | 2.993(4) | C(29) | C(30) | 3.076(4) |
| C(30) | N(3) | 3.441(3) | C(30) | C(27) | 3.054(4) |
| C(30) | C(28) | 3.036(5) | C(30) | C(29) | 3.076(4) |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| V(1) | H(2) | 3.547 | V(1) | H(6) | 3.378 |
| V(1) | H(10) | 3.320 | V(1) | H(13) | 3.458 |
| V(1) | H(15) | 3.519 | V(1) | H(21) | 3.483 |
| V(1) | H(22) | 3.439 | V(1) | H(25) | 2.518 |
| V(1) | H(26) | 2.518 | V(1) | H(36) | 2.577 |
| V(1) | H(37) | 2.577 | V(1) | H(46) | 3.449 |
| Si(1) | H(22) | 3.484 | Si(1) | H(25) | 2.334 |
| Si(1) | H(26) | 2.334 | Si(1) | H(27) | 2.388 |
| Si(1) | H(28) | 2.388 | Si(1) | H(29) | 2.388 |
| Si(1) | H(30) | 2.404 | Si(1) | H(31) | 2.404 |
| Si(1) | H(32) | 2.404 | Si(1) | H(33) | 2.401 |
| Si(1) | H(34) | 2.401 | Si(1) | H(35) | 2.401 |
| Si(2) | H(36) | 2.368 | Si(2) | H(37) | 2.368 |
| Si(2) | H(38) | 2.438 | Si(2) | H(39) | 2.438 |
| Si(2) | H(40) | 2.438 | Si(2) | H(41) | 2.418 |
| Si(2) | H(42) | 2.418 | Si(2) | H(43) | 2.418 |
| Si(2) | H(44) | 2.423 | Si(2) | H(45) | 2.423 |
| Si(2) | H(46) | 2.423 | N(1) | H(1) | 2.043 |
| N(1) | H(2) | 2.044 | N(1) | H(10) | 2.474 |
| N(1) | H(12) | 3.290 | N(1) | H(13) | 2.577 |
| N(1) | H(15) | 3.163 | N(1) | H(27) | 3.495 |
| N(1) | H(36) | 3.441 | N(1) | H(37) | 3.541 |
| N(2) | H(1) | 3.072 | N(2) | H(2) | 3.043 |
| N(2) | H(3) | 3.305 | N(2) | H(5) | 3.303 |
| N(2) | H(6) | 2.011 | N(2) | H(7)[1)] | 3.393 |
| N(2) | H(26) | 2.791 | N(2) | H(27) | 3.310 |
| N(2) | H(29) | 2.776 | N(2) | H(37) | 2.582 |
| N(3) | H(13) | 3.582 | N(3) | H(15) | 2.908 |
| N(3) | H(20) | 3.046 | N(3) | H(21) | 2.646 |
| N(3) | H(22) | 2.638 | N(3) | H(24) | 3.061 |
| N(3) | H(25) | 2.718 | N(3) | H(26) | 3.538 |
| N(3) | H(36) | 2.908 | N(3) | H(44) | 3.303 |
| N(3) | H(46) | 2.940 | C(1) | H(1)[2)] | 3.511 |
| C(1) | H(2)[2)] | 3.021 | C(1) | H(3) | 2.735 |
| C(1) | H(3)[2)] | 3.313 | C(1) | H(10) | 2.936 |
| C(1) | H(12) | 3.246 | C(1) | H(13) | 3.018 |
| C(1) | H(27) | 3.101 | C(1) | H(29) | 3.552 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(2) | H(1) | 2.071 | C(2) | H(2) | 2.071 |
| C(2) | H(3) | 2.103 | C(2) | H(4) | 3.292 |
| C(2) | H(6) | 3.182 | C(2) | H(10) | 3.564 |
| C(2) | H(27) | 3.035 | C(2) | H(29) | 2.753 |
| C(2) | H(37) | 3.536 | C(3) | H(1) | 2.791 |
| C(3) | H(2) | 2.794 | C(3) | H(2)[2)] | 3.470 |
| C(3) | H(4) | 2.063 | C(3) | H(5) | 3.300 |
| C(3) | H(11)[1)] | 3.320 | C(3) | H(27) | 3.535 |
| C(3) | H(29) | 2.904 | C(4) | H(3) | 2.079 |
| C(4) | H(5) | 2.098 | C(4) | H(6) | 3.255 |
| C(4) | H(8)[3)] | 3.268 | C(4) | H(9)[3)] | 3.155 |
| C(4) | H(11)[1)] | 2.977 | C(4) | H(29) | 3.052 |
| C(4) | H(40)[4)] | 3.469 | C(5) | H(3) | 3.311 |
| C(5) | H(4) | 2.086 | C(5) | H(6) | 2.028 |
| C(5) | H(11)[1)] | 3.087 | C(5) | H(17)[3)] | 3.495 |
| C(5) | H(29) | 3.071 | C(5) | H(40)[4)] | 3.461 |
| C(6) | H(4) | 3.283 | C(6) | H(5) | 2.078 |
| C(6) | H(7)[1)] | 3.549 | C(6) | H(11)[1)] | 3.519 |
| C(6) | H(17)[3)] | 3.339 | C(6) | H(26) | 2.804 |
| C(6) | H(29) | 2.923 | C(6) | H(37) | 2.906 |
| C(6) | H(42) | 3.489 | C(6) | H(43) | 3.532 |
| C(7) | H(1) | 2.503 | C(7) | H(2) | 2.653 |
| C(7) | H(3)[2)] | 3.076 | C(7) | H(7) | 3.353 |
| C(7) | H(9) | 3.327 | C(7) | H(10) | 2.601 |
| C(7) | H(11) | 3.242 | C(7) | H(12) | 3.023 |
| C(7) | H(13) | 2.625 | C(7) | H(14) | 3.257 |
| C(7) | H(15) | 2.921 | C(7) | H(20) | 3.012 |
| C(8) | H(1) | 2.757 | C(8) | H(2) | 3.578 |
| C(8) | H(3)[2)] | 3.597 | C(8) | H(7) | 2.106 |
| C(8) | H(8) | 3.314 | C(8) | H(10) | 2.038 |
| C(8) | H(11) | 2.038 | C(8) | H(12) | 2.038 |
| C(8) | H(20) | 2.618 | C(9) | H(8) | 2.073 |
| C(9) | H(9) | 3.293 | C(9) | H(10) | 3.260 |
| C(9) | H(10)[5)] | 3.583 | C(9) | H(11) | 2.631 |
| C(9) | H(12) | 2.883 | C(9) | H(20) | 2.894 |
| C(9) | H(37)[5)] | 3.139 | C(10) | H(3)[2)] | 3.569 |
| C(10) | H(4)[6)] | 3.018 | C(10) | H(4)[2)] | 3.200 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(10) | H(7) | 2.103 | C(10) | H(9) | 2.066 |
| C(10) | H(9)[7)] | 3.406 | C(10) | H(10)[5)] | 3.522 |
| C(10) | H(20) | 3.470 | C(11) | H(3)[2)] | 3.064 |
| C(11) | H(4)[6)] | 2.913 | C(11) | H(4)[2)] | 3.072 |
| C(11) | H(5)[6)] | 3.597 | C(11) | H(7) | 3.317 |
| C(11) | H(8) | 2.058 | C(11) | H(8)[7)] | 3.573 |
| C(11) | H(13) | 3.220 | C(11) | H(14) | 2.578 |
| C(11) | H(15) | 2.958 | C(12) | H(1) | 3.575 |
| C(12) | H(2) | 3.160 | C(12) | H(3)[2)] | 2.769 |
| C(12) | H(8) | 3.303 | C(12) | H(9) | 2.089 |
| C(12) | H(13) | 2.032 | C(12) | H(14) | 2.032 |
| C(12) | H(15) | 2.031 | C(12) | H(20) | 3.578 |
| C(13) | H(1) | 2.692 | C(13) | H(7) | 2.684 |
| C(13) | H(8)[1)] | 3.483 | C(13) | H(20) | 3.038 |
| C(13) | H(36) | 3.486 | C(14) | H(2) | 3.212 |
| C(14) | H(3)[2)] | 3.206 | C(14) | H(9) | 2.676 |
| C(14) | H(19)[8)] | 3.346 | C(14) | H(21)[8)] | 3.492 |
| C(14) | H(22) | 3.101 | C(14) | H(27) | 3.599 |
| C(14) | H(32) | 3.237 | C(14) | H(39)[8)] | 3.023 |
| C(15) | H(15) | 3.025 | C(15) | H(16) | 3.331 |
| C(15) | H(18) | 3.357 | C(15) | H(19) | 3.288 |
| C(15) | H(20) | 2.876 | C(15) | H(21) | 2.660 |
| C(15) | H(22) | 2.679 | C(15) | H(23) | 3.304 |
| C(15) | H(24) | 2.880 | C(15) | H(25) | 3.566 |
| C(15) | H(35)[9)] | 3.565 | C(15) | H(44) | 3.310 |
| C(15) | H(46) | 3.266 | C(16) | H(16) | 2.095 |
| C(16) | H(17) | 3.439 | C(16) | H(19) | 2.011 |
| C(16) | H(20) | 2.011 | C(16) | H(21) | 2.011 |
| C(16) | H(44) | 3.419 | C(17) | H(5)[6)] | 2.942 |
| C(17) | H(17) | 2.161 | C(17) | H(18) | 3.305 |
| C(17) | H(19) | 2.549 | C(17) | H(20) | 3.022 |
| C(17) | H(21) | 3.199 | C(17) | H(28)[10)] | 3.251 |
| C(17) | H(31)[10)] | 3.253 | C(18) | H(5)[6)] | 2.804 |
| C(18) | H(6)[6)] | 3.290 | C(18) | H(16) | 2.077 |
| C(18) | H(18) | 2.067 | C(18) | H(31)[10)] | 3.507 |
| C(18) | H(35)[9)] | 3.340 | C(18) | H(42)[6)] | 3.544 |
| C(19) | H(5)[6)] | 3.375 | C(19) | H(16) | 3.263 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(19) | H(17) | 2.122 | C(19) | H(22) | 3.202 |
| C(19) | H(23) | 2.555 | C(19) | H(24) | 3.009 |
| C(19) | H(35)[9)] | 2.880 | C(20) | H(15) | 2.995 |
| C(20) | H(17) | 3.386 | C(20) | H(18) | 2.100 |
| C(20) | H(22) | 2.042 | C(20) | H(23) | 2.042 |
| C(20) | H(24) | 2.042 | C(20) | H(25) | 3.596 |
| C(20) | H(35)[9)] | 2.990 | C(21) | H(11) | 3.567 |
| C(21) | H(13)[10)] | 3.560 | C(21) | H(14)[10)] | 3.307 |
| C(21) | H(16) | 2.677 | C(21) | H(36) | 3.058 |
| C(21) | H(44) | 3.513 | C(22) | H(15) | 2.917 |
| C(22) | H(18) | 2.665 | C(22) | H(25) | 2.746 |
| C(22) | H(30) | 3.157 | C(22) | H(32) | 3.151 |
| C(22) | H(35)[9)] | 3.448 | C(22) | H(41)[9)] | 3.596 |
| C(22) | H(43)[9)] | 3.216 | C(23) | H(6) | 3.114 |
| C(23) | H(18)[11)] | 3.534 | C(23) | H(22) | 2.967 |
| C(23) | H(24) | 3.297 | C(23) | H(27) | 3.345 |
| C(23) | H(29) | 3.297 | C(23) | H(30) | 3.263 |
| C(23) | H(32) | 3.281 | C(23) | H(33) | 3.191 |
| C(23) | H(35) | 3.213 | C(23) | H(43) | 3.152 |
| C(23) | H(46) | 3.396 | C(24) | H(2) | 3.345 |
| C(24) | H(13) | 3.523 | C(24) | H(16)[8)] | 3.098 |
| C(24) | H(26) | 3.530 | C(24) | H(31) | 3.130 |
| C(24) | H(32) | 3.201 | C(24) | H(33) | 3.194 |
| C(24) | H(34) | 3.114 | C(24) | H(40)[4)] | 3.395 |
| C(25) | H(22) | 2.836 | C(25) | H(25) | 2.990 |
| C(25) | H(27) | 3.164 | C(25) | H(28) | 3.153 |
| C(25) | H(31)[12)] | 3.351 | C(25) | H(34) | 3.203 |
| C(25) | H(34)[12)] | 3.460 | C(25) | H(35) | 3.206 |
| C(26) | H(24)[11)] | 3.558 | C(26) | H(25) | 3.390 |
| C(26) | H(26) | 2.888 | C(26) | H(28) | 3.121 |
| C(26) | H(29) | 3.174 | C(26) | H(30) | 3.213 |
| C(26) | H(31) | 3.198 | C(26) | H(31)[12)] | 3.461 |
| C(26) | H(38)[4)] | 3.572 | C(26) | H(41)[4)] | 3.462 |
| C(26) | H(46)[11)] | 3.444 | C(27) | H(6) | 3.289 |
| C(27) | H(7)[1)] | 3.103 | C(27) | H(10) | 3.164 |
| C(27) | H(21) | 2.974 | C(27) | H(26) | 3.351 |
| C(27) | H(39) | 3.220 | C(27) | H(40) | 3.257 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(27) | H(42) | 3.224 | C(27) | H(43) | 3.262 |
| C(27) | H(44) | 3.239 | C(27) | H(46) | 3.246 |
| C(28) | H(14)[10)] | 3.506 | C(28) | H(36) | 3.096 |
| C(28) | H(37) | 3.252 | C(28) | H(41) | 3.129 |
| C(28) | H(42) | 3.147 | C(28) | H(44) | 3.235 |
| C(28) | H(45) | 3.199 | C(29) | H(6) | 2.991 |
| C(29) | H(17)[3)] | 3.205 | C(29) | H(23)[11)] | 3.061 |
| C(29) | H(26) | 3.125 | C(29) | H(34)[13)] | 3.234 |
| C(29) | H(37) | 3.106 | C(29) | H(38) | 3.182 |
| C(29) | H(40) | 3.121 | C(29) | H(45) | 3.239 |
| C(29) | H(45)[14)] | 3.391 | C(29) | H(46) | 3.276 |
| C(30) | H(34)[9)] | 3.443 | C(30) | H(36) | 3.295 |
| C(30) | H(38) | 3.182 | C(30) | H(39) | 3.257 |
| C(30) | H(41) | 3.239 | C(30) | H(41)[14)] | 3.448 |
| C(30) | H(43) | 3.249 | C(30) | H(45)[14)] | 3.524 |
| H(1) | N(1) | 2.043 | H(1) | N(2) | 3.072 |
| H(1) | C(1)[2)] | 3.511 | H(1) | C(2) | 2.071 |
| H(1) | C(3) | 2.791 | H(1) | C(7) | 2.503 |
| H(1) | C(8) | 2.757 | H(1) | C(12) | 3.575 |
| H(1) | C(13) | 2.692 | H(1) | H(2) | 1.682 |
| H(1) | H(2)[2)] | 2.645 | H(1) | H(3) | 2.758 |
| H(1) | H(3)[2)] | 3.238 | H(1) | H(10) | 2.558 |
| H(1) | H(12) | 2.426 | H(1) | H(19)[1)] | 3.523 |
| H(2) | V(1) | 3.547 | H(2) | N(1) | 2.044 |
| H(2) | N(2) | 3.043 | H(2) | C(1)[2)] | 3.021 |
| H(2) | C(2) | 2.071 | H(2) | C(3) | 2.794 |
| H(2) | C(3)[2)] | 3.470 | H(2) | C(7) | 2.653 |
| H(2) | C(8) | 3.578 | H(2) | C(12) | 3.160 |
| H(2) | C(14) | 3.212 | H(2) | C(24) | 3.345 |
| H(2) | H(1) | 1.682 | H(2) | H(1)[2)] | 2.645 |
| H(2) | H(2)[2)] | 2.573 | H(2) | H(3) | 2.785 |
| H(2) | H(3)[2)] | 2.670 | H(2) | H(13) | 2.499 |
| H(2) | H(27) | 2.540 | H(2) | H(29) | 3.331 |
| H(3) | N(2) | 3.305 | H(3) | C(1) | 2.735 |
| H(3) | C(1)[2)] | 3.313 | H(3) | C(2) | 2.103 |
| H(3) | C(4) | 2.079 | H(3) | C(5) | 3.311 |
| H(3) | C(7)[2)] | 3.076 | H(3) | C(8)[2)] | 3.597 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(3) | C(10)[2)] | 3.569 | H(3) | C(11)[2)] | 3.064 |
| H(3) | C(12)[2)] | 2.769 | H(3) | C(14)[2)] | 3.206 |
| H(3) | H(1) | 2.758 | H(3) | H(1)[2)] | 3.238 |
| H(3) | H(2) | 2.785 | H(3) | H(2)[2)] | 2.670 |
| H(3) | H(4) | 2.390 | H(3) | H(9)[2)] | 3.468 |
| H(3) | H(13)[2)] | 3.074 | H(3) | H(14)[2)] | 3.166 |
| H(3) | H(29) | 3.497 | H(4) | C(2) | 3.292 |
| H(4) | C(3) | 2.063 | H(4) | C(5) | 2.086 |
| H(4) | C(6) | 3.283 | H(4) | C(10)[3)] | 3.018 |
| H(4) | C(10)[2)] | 3.200 | H(4) | C(11)[3)] | 2.913 |
| H(4) | C(11)[2)] | 3.072 | H(4) | H(3) | 2.390 |
| H(4) | H(5) | 2.417 | H(4) | H(8)[3)] | 2.520 |
| H(4) | H(8)[2)] | 3.236 | H(4) | H(9)[3)] | 2.272 |
| H(4) | H(9)[2)] | 3.018 | H(4) | H(11)[1)] | 3.249 |
| H(4) | H(40)[4)] | 3.420 | H(5) | N(2) | 3.303 |
| H(5) | C(3) | 3.300 | H(5) | C(4) | 2.098 |
| H(5) | C(6) | 2.078 | H(5) | C(11)[3)] | 3.597 |
| H(5) | C(17)[3)] | 2.942 | H(5) | C(18)[3)] | 2.804 |
| H(5) | C(19)[3)] | 3.375 | H(5) | H(4) | 2.417 |
| H(5) | H(6) | 2.353 | H(5) | H(9)[3)] | 3.249 |
| H(5) | H(11)[1)] | 3.420 | H(5) | H(16)[3)] | 3.125 |
| H(5) | H(17)[3)] | 2.929 | H(5) | H(39)[4)] | 3.564 |
| H(5) | H(40)[4)] | 3.418 | H(6) | V(1) | 3.378 |
| H(6) | N(2) | 2.011 | H(6) | C(2) | 3.182 |
| H(6) | C(4) | 3.255 | H(6) | C(5) | 2.028 |
| H(6) | C(18)[3)] | 3.290 | H(6) | C(23) | 3.114 |
| H(6) | C(27) | 3.289 | H(6) | C(29) | 2.991 |
| H(6) | H(5) | 2.353 | H(6) | H(16)[3)] | 3.583 |
| H(6) | H(17)[3)] | 2.579 | H(6) | H(26) | 2.380 |
| H(6) | H(29) | 3.506 | H(6) | H(33) | 3.461 |
| H(6) | H(37) | 2.759 | H(6) | H(42) | 2.603 |
| H(6) | H(43) | 2.620 | H(7) | N(2)[5)] | 3.393 |
| H(7) | C(6)[5)] | 3.549 | H(7) | C(7) | 3.353 |
| H(7) | C(8) | 2.106 | H(7) | C(10) | 2.103 |
| H(7) | C(11) | 3.317 | H(7) | C(13) | 2.684 |
| H(7) | C(27)[5)] | 3.103 | H(7) | H(8) | 2.397 |
| H(7) | H(10) | 3.569 | H(7) | H(10)[5)] | 3.121 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(7) | H(11) | 2.497 | H(7) | H(12) | 2.936 |
| H(7) | H(20) | 3.283 | H(7) | H(36)[5)] | 3.216 |
| H(7) | H(37)[5)] | 2.192 | H(8) | C(4)[6)] | 3.268 |
| H(8) | C(8) | 3.314 | H(8) | C(9) | 2.073 |
| H(8) | C(11) | 2.058 | H(8) | C(11)[7)] | 3.573 |
| H(8) | C(12) | 3.303 | H(8) | C(13)[5)] | 3.483 |
| H(8) | H(4)[6)] | 2.520 | H(8) | H(4)[2)] | 3.236 |
| H(8) | H(7) | 2.397 | H(8) | H(9) | 2.369 |
| H(8) | H(9)[7)] | 2.781 | H(8) | H(10)[5)] | 3.023 |
| H(8) | H(11)[5)] | 3.196 | H(8) | H(36)[5)] | 3.157 |
| H(8) | H(37)[5)] | 3.359 | H(9) | C(4)[6)] | 3.155 |
| H(9) | C(7) | 3.327 | H(9) | C(9) | 3.293 |
| H(9) | C(10) | 2.066 | H(9) | C(10)[7)] | 3.406 |
| H(9) | C(12) | 2.089 | H(9) | C(14) | 2.676 |
| H(9) | H(3)[2)] | 3.468 | H(9) | H(4)[6)] | 2.272 |
| H(9) | H(4)[2)] | 3.018 | H(9) | H(5)[6)] | 3.249 |
| H(9) | H(8) | 2.369 | H(9) | H(8)[7)] | 2.781 |
| H(9) | H(9)[7)] | 3.286 | H(9) | H(13) | 3.497 |
| H(9) | H(14) | 2.405 | H(9) | H(15) | 3.060 |
| H(9) | H(39)[8)] | 2.911 | H(10) | V(1) | 3.320 |
| H(10) | N(1) | 2.474 | H(10) | C(1) | 2.936 |
| H(10) | C(2) | 3.564 | H(10) | C(7) | 2.601 |
| H(10) | C(8) | 2.038 | H(10) | C(9) | 3.260 |
| H(10) | C(9)[1)] | 3.583 | H(10) | C(10)[1)] | 3.522 |
| H(10) | C(27) | 3.164 | H(10) | H(1) | 2.558 |
| H(10) | H(7) | 3.569 | H(10) | H(7)[1)] | 3.121 |
| H(10) | H(8)[1)] | 3.023 | H(10) | H(11) | 1.568 |
| H(10) | H(12) | 1.568 | H(10) | H(20) | 3.102 |
| H(10) | H(21) | 3.377 | H(10) | H(36) | 2.680 |
| H(10) | H(37) | 3.129 | H(11) | C(3)[5)] | 3.320 |
| H(11) | C(4)[5)] | 2.977 | H(11) | C(5)[5)] | 3.087 |
| H(11) | C(6)[5)] | 3.519 | H(11) | C(7) | 3.242 |
| H(11) | C(8) | 2.038 | H(11) | C(9) | 2.631 |
| H(11) | C(21) | 3.567 | H(11) | H(4)[5)] | 3.249 |
| H(11) | H(5)[5)] | 3.420 | H(11) | H(7) | 2.497 |
| H(11) | H(8)[1)] | 3.196 | H(11) | H(10) | 1.568 |
| H(11) | H(12) | 1.568 | H(11) | H(20) | 2.830 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(11) | H(21) | 3.525 | H(11) | H(36) | 3.586 |
| H(12) | N(1) | 3.290 | H(12) | C(1) | 3.246 |
| H(12) | C(7) | 3.023 | H(12) | C(8) | 2.038 |
| H(12) | C(9) | 2.883 | H(12) | H(1) | 2.426 |
| H(12) | H(7) | 2.936 | H(12) | H(10) | 1.568 |
| H(12) | H(11) | 1.568 | H(12) | H(16)[1)] | 3.058 |
| H(12) | H(19)[1)] | 3.375 | H(12) | H(20)[1)] | 3.596 |
| H(13) | V(1) | 3.458 | H(13) | N(1) | 2.577 |
| H(13) | N(3) | 3.582 | H(13) | C(1) | 3.018 |
| H(13) | C(7) | 2.625 | H(13) | C(11) | 3.220 |
| H(13) | C(12) | 2.032 | H(13) | C(21)[8)] | 3.560 |
| H(13) | C(24) | 3.523 | H(13) | H(2) | 2.499 |
| H(13) | H(3)[2)] | 3.074 | H(13) | H(9) | 3.497 |
| H(13) | H(14) | 1.551 | H(13) | H(15) | 1.551 |
| H(13) | H(19)[8)] | 2.841 | H(13) | H(21)[8)] | 3.402 |
| H(13) | H(22) | 3.258 | H(13) | H(27) | 2.665 |
| H(13) | H(32) | 2.900 | H(14) | C(7) | 3.257 |
| H(14) | C(11) | 2.578 | H(14) | C(12) | 2.032 |
| H(14) | C(21)[8)] | 3.307 | H(14) | C(28)[8)] | 3.506 |
| H(14) | H(3)[2)] | 3.166 | H(14) | H(9) | 2.405 |
| H(14) | H(13) | 1.551 | H(14) | H(15) | 1.551 |
| H(14) | H(19)[8)] | 2.969 | H(14) | H(21)[8)] | 2.766 |
| H(14) | H(32) | 3.466 | H(14) | H(36)[8)] | 3.285 |
| H(14) | H(39)[8)] | 2.475 | H(15) | V(1) | 3.519 |
| H(15) | N(1) | 3.163 | H(15) | N(3) | 2.908 |
| H(15) | C(7) | 2.921 | H(15) | C(11) | 2.958 |
| H(15) | C(12) | 2.031 | H(15) | C(15) | 3.025 |
| H(15) | C(20) | 2.995 | H(15) | C(22) | 2.917 |
| H(15) | H(9) | 3.060 | H(15) | H(13) | 1.551 |
| H(15) | H(14) | 1.551 | H(15) | H(22) | 2.222 |
| H(15) | H(23) | 3.275 | H(15) | H(32) | 2.856 |
| H(15) | H(39)[8)] | 2.775 | H(16) | C(15) | 3.331 |
| H(16) | C(16) | 2.095 | H(16) | C(18) | 2.077 |
| H(16) | C(19) | 3.263 | H(16) | C(21) | 2.677 |
| H(16) | C(24)[10)] | 3.098 | H(16) | H(5)[6)] | 3.125 |
| H(16) | H(6)[6)] | 3.583 | H(16) | H(12)[5)] | 3.058 |
| H(16) | H(17) | 2.436 | H(16) | H(19) | 2.356 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(16) | H(20) | 3.168 | H(16) | H(21) | 3.462 |
| H(16) | H(27)[10)] | 2.980 | H(16) | H(28)[10)] | 2.392 |
| H(16) | H(31)[10)] | 3.223 | H(16) | H(42)[6)] | 3.370 |
| H(17) | C(5)[6)] | 3.495 | H(17) | C(6)[6)] | 3.339 |
| H(17) | C(16) | 3.439 | H(17) | C(17) | 2.161 |
| H(17) | C(19) | 2.122 | H(17) | C(20) | 3.386 |
| H(17) | C(29)[6)] | 3.205 | H(17) | H(5)[6)] | 2.929 |
| H(17) | H(6)[6)] | 2.579 | H(17) | H(16) | 2.436 |
| H(17) | H(18) | 2.407 | H(17) | H(24)[9)] | 3.304 |
| H(17) | H(30)[9)] | 3.237 | H(17) | H(41)[6)] | 3.549 |
| H(17) | H(42)[6)] | 2.632 | H(17) | H(43)[6)] | 2.989 |
| H(18) | C(15) | 3.357 | H(18) | C(17) | 3.305 |
| H(18) | C(18) | 2.067 | H(18) | C(20) | 2.100 |
| H(18) | C(22) | 2.665 | H(18) | C(23)[9)] | 3.534 |
| H(18) | H(17) | 2.407 | H(18) | H(22) | 3.454 |
| H(18) | H(23) | 2.335 | H(18) | H(24) | 3.153 |
| H(18) | H(24)[9)] | 3.375 | H(18) | H(25)[9)] | 2.886 |
| H(18) | H(26)[9)] | 3.325 | H(18) | H(35)[9)] | 3.091 |
| H(18) | H(38)[8)] | 3.180 | H(18) | H(43)[9)] | 3.473 |
| H(18) | H(46)[9)] | 3.315 | H(19) | C(14)[10)] | 3.346 |
| H(19) | C(15) | 3.288 | H(19) | C(16) | 2.011 |
| H(19) | C(17) | 2.549 | H(19) | H(1)[5)] | 3.523 |
| H(19) | H(12)[5)] | 3.375 | H(19) | H(13)[10)] | 2.841 |
| H(19) | H(14)[10)] | 2.969 | H(19) | H(16) | 2.356 |
| H(19) | H(20) | 1.584 | H(19) | H(21) | 1.584 |
| H(19) | H(27)[10)] | 3.053 | H(19) | H(32)[10)] | 3.557 |
| H(20) | N(3) | 3.046 | H(20) | C(7) | 3.012 |
| H(20) | C(8) | 2.618 | H(20) | C(9) | 2.894 |
| H(20) | C(10) | 3.470 | H(20) | C(12) | 3.578 |
| H(20) | C(13) | 3.038 | H(20) | C(15) | 2.876 |
| H(20) | C(16) | 2.011 | H(20) | C(17) | 3.022 |
| H(20) | H(7) | 3.283 | H(20) | H(10) | 3.102 |
| H(20) | H(11) | 2.830 | H(20) | H(12)[5)] | 3.596 |
| H(20) | H(16) | 3.168 | H(20) | H(19) | 1.584 |
| H(20) | H(21) | 1.584 | H(20) | H(36) | 3.229 |
| H(21) | V(1) | 3.483 | H(21) | N(3) | 2.646 |
| H(21) | C(14)[10)] | 3.492 | H(21) | C(15) | 2.660 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(21) | C(16) | 2.011 | H(21) | C(17) | 3.199 |
| H(21) | C(27) | 2.974 | H(21) | H(10) | 3.377 |
| H(21) | H(11) | 3.525 | H(21) | H(13)[10)] | 3.402 |
| H(21) | H(14)[10)] | 2.766 | H(21) | H(16) | 3.462 |
| H(21) | H(19) | 1.584 | H(21) | H(20) | 1.584 |
| H(21) | H(36) | 2.165 | H(21) | H(44) | 2.864 |
| H(22) | V(1) | 3.439 | H(22) | Si(1) | 3.484 |
| H(22) | N(3) | 2.638 | H(22) | C(14) | 3.101 |
| H(22) | C(15) | 2.679 | H(22) | C(19) | 3.202 |
| H(22) | C(20) | 2.042 | H(22) | C(23) | 2.967 |
| H(22) | C(25) | 2.836 | H(22) | H(13) | 3.258 |
| H(22) | H(15) | 2.222 | H(22) | H(18) | 3.454 |
| H(22) | H(23) | 1.584 | H(22) | H(24) | 1.584 |
| H(22) | H(25) | 2.342 | H(22) | H(30) | 2.681 |
| H(22) | H(32) | 2.334 | H(23) | C(15) | 3.304 |
| H(23) | C(19) | 2.555 | H(23) | C(20) | 2.042 |
| H(23) | C(29)[9)] | 3.061 | H(23) | H(15) | 3.275 |
| H(23) | H(18) | 2.335 | H(23) | H(22) | 1.584 |
| H(23) | H(24) | 1.584 | H(23) | H(25) | 3.528 |
| H(23) | H(30) | 3.174 | H(23) | H(32) | 3.246 |
| H(23) | H(38)[8)] | 3.487 | H(23) | H(41)[9)] | 2.760 |
| H(23) | H(43)[9)] | 2.477 | H(24) | N(3) | 3.061 |
| H(24) | C(15) | 2.880 | H(24) | C(19) | 3.009 |
| H(24) | C(20) | 2.042 | H(24) | C(23) | 3.297 |
| H(24) | C(26)[9)] | 3.558 | H(24) | H(17)[11)] | 3.304 |
| H(24) | H(18) | 3.153 | H(24) | H(18)[11)] | 3.375 |
| H(24) | H(22) | 1.584 | H(24) | H(23) | 1.584 |
| H(24) | H(25) | 2.341 | H(24) | H(30) | 3.105 |
| H(24) | H(32) | 3.520 | H(24) | H(33)[9)] | 3.146 |
| H(24) | H(35)[9)] | 3.037 | H(24) | H(43)[9)] | 3.111 |
| H(24) | H(46) | 3.030 | H(25) | V(1) | 2.518 |
| H(25) | Si(1) | 2.334 | H(25) | N(3) | 2.718 |
| H(25) | C(15) | 3.566 | H(25) | C(20) | 3.596 |
| H(25) | C(22) | 2.746 | H(25) | C(25) | 2.990 |
| H(25) | C(26) | 3.390 | H(25) | H(18)[11)] | 2.886 |
| H(25) | H(22) | 2.342 | H(25) | H(23) | 3.528 |
| H(25) | H(24) | 2.341 | H(25) | H(26) | 1.649 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(25) | H(30) | 2.858 | H(25) | H(32) | 3.149 |
| H(25) | H(35) | 3.279 | H(25) | H(43) | 3.351 |
| H(25) | H(46) | 2.830 | H(26) | V(1) | 2.518 |
| H(26) | Si(1) | 2.334 | H(26) | N(2) | 2.791 |
| H(26) | N(3) | 3.538 | H(26) | C(6) | 2.804 |
| H(26) | C(24) | 3.530 | H(26) | C(26) | 2.888 |
| H(26) | C(27) | 3.351 | H(26) | C(29) | 3.125 |
| H(26) | H(6) | 2.380 | H(26) | H(18)[11)] | 3.325 |
| H(26) | H(25) | 1.649 | H(26) | H(29) | 3.438 |
| H(26) | H(33) | 2.751 | H(26) | H(35) | 3.015 |
| H(26) | H(37) | 3.552 | H(26) | H(42) | 3.498 |
| H(26) | H(43) | 2.274 | H(26) | H(46) | 3.387 |
| H(27) | Si(1) | 2.388 | H(27) | N(1) | 3.495 |
| H(27) | N(2) | 3.310 | H(27) | C(1) | 3.101 |
| H(27) | C(2) | 3.035 | H(27) | C(3) | 3.535 |
| H(27) | C(14) | 3.599 | H(27) | C(23) | 3.345 |
| H(27) | C(25) | 3.164 | H(27) | H(2) | 2.540 |
| H(27) | H(13) | 2.665 | H(27) | H(16)[8)] | 2.980 |
| H(27) | H(19)[8)] | 3.053 | H(27) | H(28) | 1.600 |
| H(27) | H(29) | 1.600 | H(27) | H(31) | 3.357 |
| H(27) | H(32) | 3.034 | H(28) | Si(1) | 2.388 |
| H(28) | C(17)[8)] | 3.251 | H(28) | C(25) | 3.153 |
| H(28) | C(26) | 3.121 | H(28) | H(16)[8)] | 2.392 |
| H(28) | H(27) | 1.600 | H(28) | H(29) | 1.600 |
| H(28) | H(31) | 2.948 | H(28) | H(32) | 3.442 |
| H(28) | H(33) | 3.406 | H(28) | H(34) | 2.908 |
| H(28) | H(40)[4)] | 3.156 | H(28) | H(42)[4)] | 2.747 |
| H(29) | Si(1) | 2.388 | H(29) | N(2) | 2.776 |
| H(29) | C(1) | 3.552 | H(29) | C(2) | 2.753 |
| H(29) | C(3) | 2.904 | H(29) | C(4) | 3.052 |
| H(29) | C(5) | 3.071 | H(29) | C(6) | 2.923 |
| H(29) | C(23) | 3.297 | H(29) | C(26) | 3.174 |
| H(29) | H(2) | 3.331 | H(29) | H(3) | 3.497 |
| H(29) | H(6) | 3.506 | H(29) | H(26) | 3.438 |
| H(29) | H(27) | 1.600 | H(29) | H(28) | 1.600 |
| H(29) | H(33) | 3.047 | H(29) | H(34) | 3.368 |
| H(29) | H(40)[4)] | 2.780 | H(30) | Si(1) | 2.404 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(30) | C(22) | 3.157 | H(30) | C(23) | 3.263 |
| H(30) | C(26) | 3.213 | H(30) | H(17)[11)] | 3.237 |
| H(30) | H(22) | 2.681 | H(30) | H(23) | 3.174 |
| H(30) | H(24) | 3.105 | H(30) | H(25) | 2.858 |
| H(30) | H(31) | 1.633 | H(30) | H(31)[12)] | 2.948 |
| H(30) | H(32) | 1.633 | H(30) | H(34) | 3.460 |
| H(30) | H(34)[12)] | 3.064 | H(30) | H(35) | 3.051 |
| H(30) | H(41)[9)] | 3.318 | H(31) | Si(1) | 2.404 |
| H(31) | C(17)[8)] | 3.253 | H(31) | C(18)[8)] | 3.507 |
| H(31) | C(24) | 3.130 | H(31) | C(25)[12)] | 3.351 |
| H(31) | C(26) | 3.198 | H(31) | C(26)[12)] | 3.461 |
| H(31) | H(16)[8)] | 3.223 | H(31) | H(27) | 3.357 |
| H(31) | H(28) | 2.948 | H(31) | H(30) | 1.633 |
| H(31) | H(30)[12)] | 2.948 | H(31) | H(31)[12)] | 2.953 |
| H(31) | H(32) | 1.633 | H(31) | H(34) | 3.031 |
| H(31) | H(34)[12)] | 3.088 | H(31) | H(35) | 3.443 |
| H(31) | H(35)[12)] | 3.061 | H(31) | H(44)[8)] | 3.566 |
| H(32) | Si(1) | 2.404 | H(32) | C(14) | 3.237 |
| H(32) | C(22) | 3.151 | H(32) | C(23) | 3.281 |
| H(32) | C(24) | 3.201 | H(32) | H(13) | 2.900 |
| H(32) | H(14) | 3.466 | H(32) | H(15) | 2.856 |
| H(32) | H(19)[8)] | 3.557 | H(32) | H(22) | 2.334 |
| H(32) | H(23) | 3.246 | H(32) | H(24) | 3.520 |
| H(32) | H(25) | 3.149 | H(32) | H(27) | 3.034 |
| H(32) | H(28) | 3.442 | H(32) | H(30) | 1.633 |
| H(32) | H(31) | 1.633 | H(32) | H(44)[8)] | 2.977 |
| H(33) | Si(1) | 2.401 | H(33) | C(23) | 3.191 |
| H(33) | C(24) | 3.194 | H(33) | H(6) | 3.461 |
| H(33) | H(24)[11)] | 3.146 | H(33) | H(26) | 2.751 |
| H(33) | H(28) | 3.406 | H(33) | H(29) | 3.047 |
| H(33) | H(34) | 1.633 | H(33) | H(35) | 1.633 |
| H(33) | H(38)[4)] | 2.909 | H(33) | H(40)[4)] | 3.409 |
| H(33) | H(45)[11)] | 3.520 | H(33) | H(46)[11)] | 3.186 |
| H(34) | Si(1) | 2.401 | H(34) | C(24) | 3.114 |
| H(34) | C(25) | 3.203 | H(34) | C(25)[12)] | 3.460 |
| H(34) | C(29)[4)] | 3.234 | H(34) | C(30)[11)] | 3.443 |
| H(34) | H(28) | 2.908 | H(34) | H(29) | 3.368 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(34) | H(30) | 3.460 | H(34) | H(30)[12)] | 3.064 |
| H(34) | H(31) | 3.031 | H(34) | H(31)[12)] | 3.088 |
| H(34) | H(33) | 1.633 | H(34) | H(35) | 1.633 |
| H(34) | H(38)[4)] | 3.325 | H(34) | H(41)[4)] | 2.529 |
| H(34) | H(42)[4)] | 3.091 | H(34) | H(44)[11)] | 3.295 |
| H(34) | H(45)[11)] | 3.088 | H(34) | H(46)[11)] | 3.365 |
| H(35) | Si(1) | 2.401 | H(35) | C(15)[11)] | 3.565 |
| H(35) | C(18)[11)] | 3.340 | H(35) | C(19)[11)] | 2.880 |
| H(35) | C(20)[11)] | 2.990 | H(35) | C(22)[11)] | 3.448 |
| H(35) | C(23) | 3.213 | H(35) | C(25) | 3.206 |
| H(35) | H(18)[11)] | 3.091 | H(35) | H(24)[11)] | 3.037 |
| H(35) | H(25) | 3.279 | H(35) | H(26) | 3.015 |
| H(35) | H(30) | 3.051 | H(35) | H(31) | 3.443 |
| H(35) | H(31)[12)] | 3.061 | H(35) | H(33) | 1.633 |
| H(35) | H(34) | 1.633 | H(35) | H(44)[11)] | 3.361 |
| H(35) | H(46)[11)] | 3.205 | H(36) | V(1) | 2.577 |
| H(36) | Si(2) | 2.368 | H(36) | N(1) | 3.441 |
| H(36) | N(3) | 2.908 | H(36) | C(13) | 3.486 |
| H(36) | C(21) | 3.058 | H(36) | C(28) | 3.096 |
| H(36) | C(30) | 3.295 | H(36) | H(7)[1)] | 3.216 |
| H(36) | H(8)[1)] | 3.157 | H(36) | H(10) | 2.680 |
| H(36) | H(11) | 3.586 | H(36) | H(14)[10)] | 3.285 |
| H(36) | H(20) | 3.229 | H(36) | H(21) | 2.165 |
| H(36) | H(37) | 1.682 | H(36) | H(39) | 2.910 |
| H(36) | H(40) | 3.368 | H(36) | H(44) | 3.142 |
| H(37) | V(1) | 2.577 | H(37) | Si(2) | 2.368 |
| H(37) | N(1) | 3.541 | H(37) | N(2) | 2.582 |
| H(37) | C(2) | 3.536 | H(37) | C(6) | 2.906 |
| H(37) | C(9)[1)] | 3.139 | H(37) | C(28) | 3.252 |
| H(37) | C(29) | 3.106 | H(37) | H(6) | 2.759 |
| H(37) | H(7)[1)] | 2.192 | H(37) | H(8)[1)] | 3.359 |
| H(37) | H(10) | 3.129 | H(37) | H(26) | 3.552 |
| H(37) | H(36) | 1.682 | H(37) | H(39) | 3.543 |
| H(37) | H(40) | 3.111 | H(37) | H(42) | 2.924 |
| H(37) | H(43) | 3.353 | H(38) | Si(2) | 2.438 |
| H(38) | C(26)[13)] | 3.572 | H(38) | C(29) | 3.182 |
| H(38) | C(30) | 3.182 | H(38) | H(18)[10)] | 3.180 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(38) | H(23)[10)] | 3.487 | H(38) | H(33)[13)] | 2.909 |
| H(38) | H(34)[13)] | 3.325 | H(38) | H(39) | 1.731 |
| H(38) | H(40) | 1.731 | H(38) | H(41) | 2.974 |
| H(38) | H(42) | 3.451 | H(38) | H(44) | 3.472 |
| H(38) | H(45) | 2.979 | H(38) | H(45)[14)] | 3.078 |
| H(39) | Si(2) | 2.438 | H(39) | C(14)[10)] | 3.023 |
| H(39) | C(27) | 3.220 | H(39) | C(30) | 3.257 |
| H(39) | H(5)[13)] | 3.564 | H(39) | H(9)[10)] | 2.911 |
| H(39) | H(14)[10)] | 2.475 | H(39) | H(15)[10)] | 2.775 |
| H(39) | H(36) | 2.910 | H(39) | H(37) | 3.543 |
| H(39) | H(38) | 1.731 | H(39) | H(40) | 1.731 |
| H(39) | H(44) | 3.097 | H(39) | H(45) | 3.528 |
| H(40) | Si(2) | 2.438 | H(40) | C(4)[13)] | 3.469 |
| H(40) | C(5)[13)] | 3.461 | H(40) | C(24)[13)] | 3.395 |
| H(40) | C(27) | 3.257 | H(40) | C(29) | 3.121 |
| H(40) | H(4)[13)] | 3.420 | H(40) | H(5)[13)] | 3.418 |
| H(40) | H(28)[13)] | 3.156 | H(40) | H(29)[13)] | 2.780 |
| H(40) | H(33)[13)] | 3.409 | H(40) | H(36) | 3.368 |
| H(40) | H(37) | 3.111 | H(40) | H(38) | 1.731 |
| H(40) | H(39) | 1.731 | H(40) | H(41) | 3.339 |
| H(40) | H(42) | 2.928 | H(41) | Si(2) | 2.418 |
| H(41) | C(22)[11)] | 3.596 | H(41) | C(26)[13)] | 3.462 |
| H(41) | C(28) | 3.129 | H(41) | C(30) | 3.239 |
| H(41) | C(30)[14)] | 3.448 | H(41) | H(17)[3)] | 3.549 |
| H(41) | H(23)[11)] | 2.760 | H(41) | H(30)[11)] | 3.318 |
| H(41) | H(34)[13)] | 2.529 | H(41) | H(38) | 2.974 |
| H(41) | H(40) | 3.339 | H(41) | H(42) | 1.633 |
| H(41) | H(43) | 1.633 | H(41) | H(44)[14)] | 3.548 |
| H(41) | H(45) | 3.064 | H(41) | H(45)[14)] | 2.516 |
| H(41) | H(46) | 3.538 | H(42) | Si(2) | 2.418 |
| H(42) | C(6) | 3.489 | H(42) | C(18)[3)] | 3.544 |
| H(42) | C(27) | 3.224 | H(42) | C(28) | 3.147 |
| H(42) | H(6) | 2.603 | H(42) | H(16)[3)] | 3.370 |
| H(42) | H(17)[3)] | 2.632 | H(42) | H(26) | 3.498 |
| H(42) | H(28)[13)] | 2.747 | H(42) | H(34)[13)] | 3.091 |
| H(42) | H(37) | 2.924 | H(42) | H(38) | 3.451 |
| H(42) | H(40) | 2.928 | H(42) | H(41) | 1.633 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(42) | H(43) | 1.633 | H(43) | Si(2) | 2.418 |
| H(43) | C(6) | 3.532 | H(43) | C(22)[11)] | 3.216 |
| H(43) | C(23) | 3.152 | H(43) | C(27) | 3.262 |
| H(43) | C(30) | 3.249 | H(43) | H(6) | 2.620 |
| H(43) | H(17)[3)] | 2.989 | H(43) | H(18)[11)] | 3.473 |
| H(43) | H(23)[11)] | 2.477 | H(43) | H(24)[11)] | 3.111 |
| H(43) | H(25) | 3.351 | H(43) | H(26) | 2.274 |
| H(43) | H(37) | 3.353 | H(43) | H(41) | 1.633 |
| H(43) | H(42) | 1.633 | H(43) | H(45) | 3.501 |
| H(43) | H(46) | 3.113 | H(44) | Si(2) | 2.423 |
| H(44) | N(3) | 3.303 | H(44) | C(15) | 3.310 |
| H(44) | C(16) | 3.419 | H(44) | C(21) | 3.513 |
| H(44) | C(27) | 3.239 | H(44) | C(28) | 3.235 |
| H(44) | H(21) | 2.864 | H(44) | H(31)[10)] | 3.566 |
| H(44) | H(32)[10)] | 2.977 | H(44) | H(34)[9)] | 3.295 |
| H(44) | H(35)[9)] | 3.361 | H(44) | H(36) | 3.142 |
| H(44) | H(38) | 3.472 | H(44) | H(39) | 3.097 |
| H(44) | H(41)[14)] | 3.548 | H(44) | H(45) | 1.747 |
| H(44) | H(46) | 1.747 | H(45) | Si(2) | 2.423 |
| H(45) | C(28) | 3.199 | H(45) | C(29) | 3.239 |
| H(45) | C(29)[14)] | 3.391 | H(45) | C(30)[14)] | 3.524 |
| H(45) | H(33)[9)] | 3.520 | H(45) | H(34)[9)] | 3.088 |
| H(45) | H(38) | 2.979 | H(45) | H(38)[14)] | 3.078 |
| H(45) | H(39) | 3.528 | H(45) | H(41) | 3.064 |
| H(45) | H(41)[14)] | 2.516 | H(45) | H(43) | 3.501 |
| H(45) | H(44) | 1.747 | H(45) | H(45)[14)] | 2.626 |
| H(45) | H(46) | 1.747 | H(46) | V(1) | 3.449 |
| H(46) | Si(2) | 2.423 | H(46) | N(3) | 2.940 |
| H(46) | C(15) | 3.266 | H(46) | C(23) | 3.396 |
| H(46) | C(26)[9)] | 3.444 | H(46) | C(27) | 3.246 |
| H(46) | C(29) | 3.276 | H(46) | H(18)[11)] | 3.315 |
| H(46) | H(24) | 3.030 | H(46) | H(25) | 2.830 |
| H(46) | H(26) | 3.387 | H(46) | H(33)[9)] | 3.186 |
| H(46) | H(34)[9)] | 3.365 | H(46) | H(35)[9)] | 3.205 |
| H(46) | H(41) | 3.538 | H(46) | H(43) | 3.113 |
| H(46) | H(44) | 1.747 | H(46) | H(45) | 1.747 |

Symmetry Operators:

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

X-ray Structure Report for V(N=2,6-$Me_2C_6H_3$)($CH_2SiMe_3$)$_2$[2-(2,6-$^{i}Pr_2C_6H_3$)$CH_2$($C_5H_4N$)] (**2b**)

June 24, 2009

*Experimental*

Data Collection

A red block crystal of $C_{34}H_{54}N_3Si_2V$ having approximate dimensions of 0.20 x 0.15 x 0.10 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-K$\alpha$ 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 primitive triclinic cell with dimensions:

| | | | |
|---|---|---|---|
| a | = 10.1893(9) Å | $\alpha$ | = 97.395(3)$^o$ |
| b | = 11.0729(9) Å | $\beta$ | = 95.290(3)$^o$ |
| c | = 16.2502(16) Å | $\gamma$ | = 96.446(2)$^o$ |
| V | = 1796.1(3) Å$^3$ | | |

For Z = 2 and F.W. = 611.93, the calculated density is 1.131 g/cm$^3$. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -120 $\pm$ 1$^o$C to a maximum 2$\theta$ value of 48.8$^o$. A total of 74 oscillation images were collected. A sweep of data was done using $\omega$ scans from 130.0 to 190.0$^o$ in 3.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 0.0$^o$. The exposure rate was 240.0 [sec./$^o$]. A second sweep was performed using $\omega$ scans from 0.0 to 162.0$^o$ in 3.0$^o$ step, at $\chi$=45.0$^o$ and $\phi$ = 180.0$^o$. The exposure rate was 240.0 [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 13412 reflections that were collected, 5866 were unique ($R_{int}$ = 0.054); equivalent reflections were merged.

The linear absorption coefficient, $\mu$, for Mo-K$\alpha$ radiation is 3.677 $cm^{-1}$. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.576 to 0.964. 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 5866 observed reflections and 362 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0637$$

$$wR2 = [ \; \Sigma \; ( \; w \; (Fo^2 - Fc^2)^2 \; ) / \; \Sigma \; w(Fo^2)^2]^{1/2} = 0.2134$$

The standard deviation of an observation of unit weight[4] was 1.18. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.66 and -1.09 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for $\Delta$f' and $\Delta$f'' 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] crystallographic software package except for refinement, which was performed using SHELXL-97[10].

*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: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ 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) SHELX97: Sheldrick, G.M. (1997).

*EXPERIMENTAL DETAILS*

A. Crystal Data

| | |
|---|---|
| Empirical Formula | $C_{34}H_{54}N_3Si_2V$ |
| Formula Weight | 611.93 |
| Crystal Color, Habit | red, block |
| Crystal Dimensions | 0.20 X 0.15 X 0.10 mm |
| Crystal System | triclinic |
| Lattice Type | Primitive |
| Indexing Images | 3 oscillations @ 320.0 seconds |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| Lattice Parameters | a = 10.1893(9) Å<br>b = 11.0729(9) Å<br>c = 16.2502(16) Å<br>$\alpha$ = 97.395(3) $^o$<br>$\beta$ = 95.290(3) $^o$<br>$\gamma$ = 96.446(2) $^o$<br>V = 1796.1(3) Å$^3$ |
| Space Group | P-1 (#2) |
| Z value | 2 |
| $D_{calc}$ | 1.131 g/cm$^3$ |
| $F_{000}$ | 660.00 |
| $\mu$(MoK$\alpha$) | 3.677 cm$^{-1}$ |

B. Intensity Measurements

| | |
|---|---|
| Diffractometer | Rigaku RAXIS-RAPID |
| Radiation | MoK$\alpha$ ($\lambda$ = 0.71075 Å)<br>graphite monochromated |
| Detector Aperture | 280 mm x 256 mm |
| Data Images | 74 exposures |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=0.0) | 130.0 - 190.0$^{o}$ |
| Exposure Rate | 240.0 sec./$^{o}$ |
| $\omega$ oscillation Range ($\chi$=45.0, $\phi$=180.0) | 0.0 - 162.0$^{o}$ |
| Exposure Rate | 240.0 sec./$^{o}$ |
| Detector Position | 127.40 mm |
| Pixel Size | 0.100 mm |
| $2\theta_{max}$ | 48.8$^{o}$ |
| No. of Reflections Measured | Total: 13412<br>Unique: 5866 ($R_{int}$ = 0.054) |
| Corrections | Lorentz-polarization<br>Absorption<br>(trans. factors: 0.576 - 0.964) |

C. Structure Solution and Refinement

| | |
|---|---|
| Structure Solution | Direct Methods (SIR92) |
| Refinement | Full-matrix least-squares on $F^2$ |
| Function Minimized | $\Sigma$ w $(Fo^2 - Fc^2)^2$ |
| Least Squares Weights | $w = 1/ [ \sigma^2(Fo^2) + (0.0585 \cdot P)^2 + 5.5510 \cdot P ]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$ |
| $2\theta_{max}$ cutoff | $48.8^o$ |
| Anomalous Dispersion | All non-hydrogen atoms |
| No. Observations (All reflections) | 5866 |
| No. Variables | 362 |
| Reflection/Parameter Ratio | 16.20 |
| Residuals: R1 (I>2.00$\sigma$(I)) | 0.0637 |
| Residuals: R (All reflections) | 0.1097 |
| Residuals: wR2 (All reflections) | 0.2134 |
| Goodness of Fit Indicator | 1.181 |
| Max Shift/Error in Final Cycle | 0.000 |
| Maximum peak in Final Diff. Map | 0.66 $e^-/Å^3$ |
| Minimum peak in Final Diff. Map | -1.09 $e^-/Å^3$ |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| V(1) | 0.30575(9) | 0.17342(8) | 0.22466(6) | 2.45(2) |
| Si(1) | 0.20614(17) | 0.31577(15) | 0.04828(10) | 3.25(3) |
| Si(2) | 0.38487(16) | -0.11858(13) | 0.18168(10) | 3.07(3) |
| N(1) | 0.3381(4) | 0.3111(3) | 0.3099(2) | 2.50(7) |
| N(2) | 0.4946(4) | 0.2792(3) | 0.1956(2) | 2.67(8) |
| N(3) | 0.1675(3) | 0.1034(3) | 0.2526(2) | 2.31(7) |
| C(1) | 0.4320(5) | 0.4189(4) | 0.3045(3) | 2.48(9) |
| C(2) | 0.5248(5) | 0.3907(4) | 0.2403(3) | 2.46(9) |
| C(3) | 0.6320(5) | 0.4711(4) | 0.2284(3) | 3.04(10) |
| C(4) | 0.7129(5) | 0.4376(5) | 0.1684(3) | 3.36(11) |
| C(5) | 0.6821(5) | 0.3207(5) | 0.1220(3) | 3.62(11) |
| C(6) | 0.5739(5) | 0.2449(4) | 0.1371(3) | 2.87(9) |
| C(7) | 0.2930(5) | 0.3176(4) | 0.3924(3) | 2.47(9) |
| C(8) | 0.3813(5) | 0.2971(4) | 0.4596(3) | 2.39(9) |
| C(9) | 0.3384(5) | 0.3052(4) | 0.5385(3) | 2.87(10) |
| C(10) | 0.2110(5) | 0.3303(5) | 0.5516(3) | 3.35(11) |
| C(11) | 0.1265(5) | 0.3497(5) | 0.4851(3) | 3.25(10) |
| C(12) | 0.1630(5) | 0.3448(4) | 0.4045(3) | 2.70(9) |
| C(13) | 0.5224(5) | 0.2652(4) | 0.4491(3) | 2.71(9) |
| C(14) | 0.0675(5) | 0.3712(5) | 0.3342(3) | 3.16(10) |
| C(15) | 0.0507(5) | 0.0450(4) | 0.2778(3) | 2.89(10) |
| C(16) | 0.0579(5) | -0.0045(5) | 0.3536(3) | 3.12(10) |
| C(17) | -0.0607(6) | -0.0553(5) | 0.3807(4) | 3.98(12) |
| C(18) | -0.1820(6) | -0.0587(6) | 0.3339(4) | 4.36(13) |
| C(19) | -0.1865(5) | -0.0130(5) | 0.2587(4) | 4.03(12) |
| C(20) | -0.0716(5) | 0.0386(5) | 0.2290(3) | 3.28(10) |
| C(21) | 0.1864(5) | -0.0062(5) | 0.4050(3) | 3.80(12) |
| C(22) | -0.0806(6) | 0.0846(6) | 0.1458(4) | 4.17(12) |
| C(23) | 0.2462(5) | 0.1808(4) | 0.0983(3) | 2.86(10) |
| C(24) | 0.3405(8) | 0.4443(6) | 0.0645(5) | 7.4(2) |
| C(25) | 0.0549(10) | 0.3772(9) | 0.0831(6) | 9.9(3) |
| C(26) | 0.1711(9) | 0.2706(6) | -0.0656(4) | 6.6(2) |
| C(27) | 0.4238(5) | 0.0353(4) | 0.2460(3) | 2.71(9) |
| C(28) | 0.4640(6) | -0.2359(5) | 0.2357(4) | 4.26(13) |
| C(29) | 0.4563(8) | -0.1168(6) | 0.0808(4) | 5.59(17) |
| C(30) | 0.2062(7) | -0.1738(6) | 0.1588(5) | 5.96(18) |
| C(31) | 0.6287(5) | 0.3621(5) | 0.4979(3) | 3.58(11) |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| C(32) | 0.5400(6) | 0.1407(4) | 0.4755(3) | 3.56(11) |
| C(33) | 0.0859(6) | 0.5079(5) | 0.3257(4) | 4.03(12) |
| C(34) | -0.0775(5) | 0.3295(5) | 0.3441(3) | 3.93(12) |

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

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| H(1) | 0.4838 | 0.4465 | 0.3595 | 2.98 |
| H(2) | 0.3825 | 0.4864 | 0.2897 | 2.98 |
| H(3) | 0.6501 | 0.5495 | 0.2615 | 3.65 |
| H(4) | 0.7872 | 0.4920 | 0.1589 | 4.03 |
| H(5) | 0.7359 | 0.2940 | 0.0802 | 4.34 |
| H(6) | 0.5541 | 0.1656 | 0.1054 | 3.45 |
| H(7) | 0.3977 | 0.2933 | 0.5847 | 3.44 |
| H(8) | 0.1826 | 0.3340 | 0.6058 | 4.02 |
| H(9) | 0.0396 | 0.3672 | 0.4946 | 3.90 |
| H(10) | 0.5354 | 0.2608 | 0.3886 | 3.26 |
| H(11) | 0.0892 | 0.3250 | 0.2812 | 3.79 |
| H(12) | -0.0575 | -0.0880 | 0.4321 | 4.78 |
| H(13) | -0.2614 | -0.0921 | 0.3534 | 5.23 |
| H(14) | -0.2698 | -0.0168 | 0.2264 | 4.83 |
| H(15) | 0.2212 | 0.0775 | 0.4310 | 4.56 |
| H(16) | 0.2503 | -0.0388 | 0.3692 | 4.56 |
| H(17) | 0.1723 | -0.0584 | 0.4485 | 4.56 |
| H(18) | -0.0435 | 0.1714 | 0.1531 | 5.01 |
| H(19) | -0.1738 | 0.0757 | 0.1224 | 5.01 |
| H(20) | -0.0303 | 0.0368 | 0.1078 | 5.01 |
| H(21) | 0.3167 | 0.1475 | 0.0675 | 3.43 |
| H(22) | 0.1664 | 0.1192 | 0.0836 | 3.43 |
| H(23) | 0.3241 | 0.5003 | 0.0237 | 8.91 |
| H(24) | 0.4254 | 0.4128 | 0.0574 | 8.91 |
| H(25) | 0.3436 | 0.4885 | 0.1211 | 8.91 |
| H(26) | 0.0614 | 0.3889 | 0.1442 | 11.93 |
| H(27) | -0.0237 | 0.3191 | 0.0605 | 11.93 |
| H(28) | 0.0474 | 0.4561 | 0.0629 | 11.93 |
| H(29) | 0.1327 | 0.3364 | -0.0906 | 7.87 |
| H(30) | 0.1081 | 0.1953 | -0.0773 | 7.87 |
| H(31) | 0.2539 | 0.2561 | -0.0894 | 7.87 |
| H(32) | 0.5164 | 0.0670 | 0.2394 | 3.25 |
| H(33) | 0.4217 | 0.0231 | 0.3052 | 3.25 |
| H(34) | 0.4515 | -0.3145 | 0.1987 | 5.12 |
| H(35) | 0.4226 | -0.2462 | 0.2869 | 5.12 |
| H(36) | 0.5592 | -0.2086 | 0.2498 | 5.12 |
| H(37) | 0.4750 | -0.1995 | 0.0600 | 6.71 |

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

| atom | x | y | z | $B_{eq}$ |
|---|---|---|---|---|
| H(38) | 0.5389 | -0.0600 | 0.0889 | 6.71 |
| H(39) | 0.3927 | -0.0898 | 0.0401 | 6.71 |
| H(40) | 0.1922 | -0.2394 | 0.1112 | 7.16 |
| H(41) | 0.1579 | -0.1058 | 0.1455 | 7.16 |
| H(42) | 0.1733 | -0.2054 | 0.2077 | 7.16 |
| H(43) | 0.6189 | 0.3672 | 0.5576 | 4.30 |
| H(44) | 0.6190 | 0.4419 | 0.4796 | 4.30 |
| H(45) | 0.7168 | 0.3397 | 0.4879 | 4.30 |
| H(46) | 0.6194 | 0.1118 | 0.4539 | 4.27 |
| H(47) | 0.4619 | 0.0819 | 0.4531 | 4.27 |
| H(48) | 0.5503 | 0.1481 | 0.5366 | 4.27 |
| H(49) | 0.1736 | 0.5301 | 0.3077 | 4.84 |
| H(50) | 0.0790 | 0.5563 | 0.3796 | 4.84 |
| H(51) | 0.0169 | 0.5249 | 0.2841 | 4.84 |
| H(52) | -0.0881 | 0.2423 | 0.3507 | 4.71 |
| H(53) | -0.1340 | 0.3416 | 0.2944 | 4.71 |
| H(54) | -0.1036 | 0.3777 | 0.3935 | 4.71 |

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

Table 3. Anisotropic displacement parameters

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| V(1) | 0.0329(5) | 0.0281(4) | 0.0322(5) | 0.0040(3) | 0.0049(3) | 0.0043(3) |
| Si(1) | 0.0451(9) | 0.0407(8) | 0.0384(9) | 0.0050(7) | 0.0024(7) | 0.0098(7) |
| Si(2) | 0.0454(9) | 0.0300(7) | 0.0399(9) | 0.0082(6) | -0.0007(7) | 0.0000(6) |
| N(1) | 0.039(2) | 0.032(2) | 0.028(2) | 0.0086(19) | 0.0117(19) | 0.0068(18) |
| N(2) | 0.035(2) | 0.034(2) | 0.036(2) | 0.0050(19) | 0.011(2) | 0.009(2) |
| N(3) | 0.026(2) | 0.027(2) | 0.032(2) | -0.0040(17) | 0.0014(18) | 0.0026(18) |
| C(1) | 0.031(2) | 0.026(2) | 0.038(3) | -0.002(2) | 0.005(2) | 0.009(2) |
| C(2) | 0.036(2) | 0.029(2) | 0.028(2) | 0.002(2) | 0.004(2) | 0.007(2) |
| C(3) | 0.037(3) | 0.033(2) | 0.044(3) | -0.007(2) | 0.006(2) | 0.010(2) |
| C(4) | 0.039(3) | 0.047(3) | 0.041(3) | -0.002(2) | 0.001(2) | 0.017(2) |
| C(5) | 0.039(3) | 0.058(3) | 0.044(3) | 0.008(2) | 0.012(2) | 0.014(3) |
| C(6) | 0.035(3) | 0.032(2) | 0.041(3) | 0.000(2) | 0.010(2) | 0.000(2) |
| C(7) | 0.039(3) | 0.018(2) | 0.037(3) | 0.002(2) | 0.006(2) | 0.003(2) |
| C(8) | 0.035(2) | 0.024(2) | 0.030(2) | -0.001(2) | 0.000(2) | 0.003(2) |
| C(9) | 0.048(3) | 0.032(2) | 0.027(2) | -0.001(2) | 0.002(2) | 0.003(2) |
| C(10) | 0.049(3) | 0.041(3) | 0.035(3) | -0.002(2) | 0.010(2) | 0.001(2) |
| C(11) | 0.042(3) | 0.046(3) | 0.037(3) | 0.007(2) | 0.015(2) | -0.000(2) |
| C(12) | 0.032(2) | 0.033(2) | 0.037(3) | 0.006(2) | 0.006(2) | 0.001(2) |
| C(13) | 0.038(3) | 0.028(2) | 0.037(3) | 0.005(2) | 0.003(2) | 0.005(2) |
| C(14) | 0.038(3) | 0.042(3) | 0.040(3) | 0.013(2) | 0.003(2) | 0.000(2) |
| C(15) | 0.036(3) | 0.032(2) | 0.042(3) | 0.010(2) | 0.005(2) | 0.003(2) |
| C(16) | 0.037(3) | 0.037(3) | 0.044(3) | -0.000(2) | 0.006(2) | 0.008(2) |
| C(17) | 0.046(3) | 0.056(3) | 0.048(3) | -0.007(2) | 0.009(3) | 0.011(3) |
| C(18) | 0.043(3) | 0.066(4) | 0.054(4) | -0.006(3) | 0.013(3) | 0.008(3) |
| C(19) | 0.028(3) | 0.056(3) | 0.063(4) | -0.005(2) | 0.002(2) | 0.000(3) |
| C(20) | 0.035(3) | 0.042(3) | 0.049(3) | 0.009(2) | 0.005(2) | 0.010(2) |
| C(21) | 0.044(3) | 0.051(3) | 0.050(3) | -0.003(2) | 0.005(2) | 0.018(3) |
| C(22) | 0.043(3) | 0.058(3) | 0.057(4) | 0.002(3) | -0.003(3) | 0.014(3) |
| C(23) | 0.040(3) | 0.037(2) | 0.030(2) | 0.009(2) | 0.002(2) | -0.001(2) |
| C(24) | 0.102(6) | 0.059(4) | 0.112(7) | -0.011(4) | -0.053(5) | 0.041(4) |
| C(25) | 0.143(9) | 0.143(8) | 0.152(9) | 0.105(7) | 0.102(7) | 0.112(8) |
| C(26) | 0.134(7) | 0.056(4) | 0.055(4) | 0.006(4) | -0.018(4) | 0.015(3) |
| C(27) | 0.031(2) | 0.032(2) | 0.037(3) | -0.004(2) | -0.001(2) | 0.007(2) |
| C(28) | 0.073(4) | 0.027(2) | 0.057(4) | 0.004(2) | -0.008(3) | 0.001(2) |
| C(29) | 0.112(6) | 0.044(3) | 0.056(4) | 0.020(4) | 0.009(4) | -0.001(3) |
| C(30) | 0.066(4) | 0.045(3) | 0.104(6) | 0.002(3) | -0.005(4) | -0.020(3) |
| C(31) | 0.045(3) | 0.037(3) | 0.052(3) | 0.002(2) | 0.000(2) | 0.003(2) |

Table 3. Anisotropic displacement parameters (continued)

| atom | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|
| C(32) | 0.052(3) | 0.030(2) | 0.051(3) | 0.007(2) | -0.003(2) | 0.001(2) |
| C(33) | 0.048(3) | 0.054(3) | 0.054(4) | 0.020(3) | 0.002(3) | 0.010(3) |
| C(34) | 0.043(3) | 0.055(3) | 0.049(3) | 0.011(2) | 0.003(2) | -0.003(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) | N(1) | 1.900(3) | V(1) | N(2) | 2.259(4) |
| V(1) | N(3) | 1.659(4) | V(1) | C(23) | 2.101(5) |
| V(1) | C(27) | 2.093(5) | Si(1) | C(23) | 1.858(5) |
| Si(1) | C(24) | 1.835(7) | Si(1) | C(25) | 1.858(11) |
| Si(1) | C(26) | 1.844(7) | Si(2) | C(27) | 1.862(4) |
| Si(2) | C(28) | 1.873(6) | Si(2) | C(29) | 1.856(7) |
| Si(2) | C(30) | 1.842(7) | N(1) | C(1) | 1.460(6) |
| N(1) | C(7) | 1.453(7) | N(2) | C(2) | 1.337(6) |
| N(2) | C(6) | 1.349(7) | N(3) | C(15) | 1.410(6) |
| C(1) | C(2) | 1.498(7) | C(2) | C(3) | 1.374(7) |
| C(3) | C(4) | 1.375(8) | C(4) | C(5) | 1.399(8) |
| C(5) | C(6) | 1.370(8) | C(7) | C(8) | 1.407(7) |
| C(7) | C(12) | 1.417(7) | C(8) | C(9) | 1.389(7) |
| C(8) | C(13) | 1.537(7) | C(9) | C(10) | 1.387(8) |
| C(10) | C(11) | 1.373(8) | C(11) | C(12) | 1.390(8) |
| C(12) | C(14) | 1.511(7) | C(13) | C(31) | 1.522(7) |
| C(13) | C(32) | 1.521(7) | C(14) | C(33) | 1.530(8) |
| C(14) | C(34) | 1.527(8) | C(15) | C(16) | 1.411(8) |
| C(15) | C(20) | 1.403(7) | C(16) | C(17) | 1.407(8) |
| C(16) | C(21) | 1.490(8) | C(17) | C(18) | 1.385(8) |
| C(18) | C(19) | 1.379(10) | C(19) | C(20) | 1.399(8) |
| C(20) | C(22) | 1.503(9) | | | |

Table 5. Bond lengths involving hydrogens (Å)

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(1) | H(1) | 0.990 | C(1) | H(2) | 0.990 |
| C(3) | H(3) | 0.950 | C(4) | H(4) | 0.950 |
| C(5) | H(5) | 0.950 | C(6) | H(6) | 0.950 |
| C(9) | H(7) | 0.950 | C(10) | H(8) | 0.950 |
| C(11) | H(9) | 0.950 | C(13) | H(10) | 1.000 |
| C(14) | H(11) | 1.000 | C(17) | H(12) | 0.950 |
| C(18) | H(13) | 0.950 | C(19) | H(14) | 0.950 |
| C(21) | H(15) | 0.980 | C(21) | H(16) | 0.980 |
| C(21) | H(17) | 0.980 | C(22) | H(18) | 0.980 |
| C(22) | H(19) | 0.980 | C(22) | H(20) | 0.980 |
| C(23) | H(21) | 0.990 | C(23) | H(22) | 0.990 |
| C(24) | H(23) | 0.980 | C(24) | H(24) | 0.980 |
| C(24) | H(25) | 0.980 | C(25) | H(26) | 0.980 |
| C(25) | H(27) | 0.980 | C(25) | H(28) | 0.980 |
| C(26) | H(29) | 0.980 | C(26) | H(30) | 0.980 |
| C(26) | H(31) | 0.980 | C(27) | H(32) | 0.990 |
| C(27) | H(33) | 0.990 | C(28) | H(34) | 0.980 |
| C(28) | H(35) | 0.980 | C(28) | H(36) | 0.980 |
| C(29) | H(37) | 0.980 | C(29) | H(38) | 0.980 |
| C(29) | H(39) | 0.980 | C(30) | H(40) | 0.980 |
| C(30) | H(41) | 0.980 | C(30) | H(42) | 0.980 |
| C(31) | H(43) | 0.980 | C(31) | H(44) | 0.980 |
| C(31) | H(45) | 0.980 | C(32) | H(46) | 0.980 |
| C(32) | H(47) | 0.980 | C(32) | H(48) | 0.980 |
| C(33) | H(49) | 0.980 | C(33) | H(50) | 0.980 |
| C(33) | H(51) | 0.980 | C(34) | H(52) | 0.980 |
| C(34) | H(53) | 0.980 | C(34) | H(54) | 0.980 |

Table 6. Bond angles (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| N(1) | V(1) | N(2) | 75.88(17) | N(1) | V(1) | N(3) | 99.74(19) |
| N(1) | V(1) | C(23) | 125.0(2) | N(1) | V(1) | C(27) | 113.17(19) |
| N(2) | V(1) | N(3) | 175.60(17) | N(2) | V(1) | C(23) | 82.61(18) |
| N(2) | V(1) | C(27) | 85.45(18) | N(3) | V(1) | C(23) | 100.4(2) |
| N(3) | V(1) | C(27) | 96.1(2) | C(23) | V(1) | C(27) | 114.8(2) |
| C(23) | Si(1) | C(24) | 114.3(3) | C(23) | Si(1) | C(25) | 112.9(4) |
| C(23) | Si(1) | C(26) | 109.6(3) | C(24) | Si(1) | C(25) | 107.2(4) |
| C(24) | Si(1) | C(26) | 106.4(4) | C(25) | Si(1) | C(26) | 106.0(4) |
| C(27) | Si(2) | C(28) | 110.1(2) | C(27) | Si(2) | C(29) | 110.1(2) |
| C(27) | Si(2) | C(30) | 114.5(2) | C(28) | Si(2) | C(29) | 106.5(3) |
| C(28) | Si(2) | C(30) | 107.3(3) | C(29) | Si(2) | C(30) | 107.9(3) |
| V(1) | N(1) | C(1) | 122.5(3) | V(1) | N(1) | C(7) | 126.2(3) |
| C(1) | N(1) | C(7) | 110.5(3) | V(1) | N(2) | C(2) | 114.2(3) |
| V(1) | N(2) | C(6) | 127.6(3) | C(2) | N(2) | C(6) | 118.1(4) |
| V(1) | N(3) | C(15) | 179.0(3) | N(1) | C(1) | C(2) | 111.1(3) |
| N(2) | C(2) | C(1) | 113.9(4) | N(2) | C(2) | C(3) | 122.7(4) |
| C(1) | C(2) | C(3) | 123.4(4) | C(2) | C(3) | C(4) | 119.7(4) |
| C(3) | C(4) | C(5) | 117.7(5) | C(4) | C(5) | C(6) | 119.6(5) |
| N(2) | C(6) | C(5) | 122.1(4) | N(1) | C(7) | C(8) | 118.6(4) |
| N(1) | C(7) | C(12) | 120.3(4) | C(8) | C(7) | C(12) | 121.1(4) |
| C(7) | C(8) | C(9) | 118.4(4) | C(7) | C(8) | C(13) | 122.9(4) |
| C(9) | C(8) | C(13) | 118.7(4) | C(8) | C(9) | C(10) | 121.4(5) |
| C(9) | C(10) | C(11) | 119.1(5) | C(10) | C(11) | C(12) | 122.7(5) |
| C(7) | C(12) | C(11) | 117.2(5) | C(7) | C(12) | C(14) | 122.3(4) |
| C(11) | C(12) | C(14) | 120.4(5) | C(8) | C(13) | C(31) | 112.2(4) |
| C(8) | C(13) | C(32) | 111.0(4) | C(31) | C(13) | C(32) | 109.5(4) |
| C(12) | C(14) | C(33) | 110.6(4) | C(12) | C(14) | C(34) | 113.0(5) |
| C(33) | C(14) | C(34) | 110.0(4) | N(3) | C(15) | C(16) | 119.1(4) |
| N(3) | C(15) | C(20) | 120.4(5) | C(16) | C(15) | C(20) | 120.5(5) |
| C(15) | C(16) | C(17) | 118.5(5) | C(15) | C(16) | C(21) | 122.3(5) |
| C(17) | C(16) | C(21) | 119.2(5) | C(16) | C(17) | C(18) | 121.2(6) |
| C(17) | C(18) | C(19) | 119.5(6) | C(18) | C(19) | C(20) | 121.7(5) |
| C(15) | C(20) | C(19) | 118.7(5) | C(15) | C(20) | C(22) | 121.4(5) |
| C(19) | C(20) | C(22) | 119.9(5) | V(1) | C(23) | Si(1) | 128.4(2) |
| V(1) | C(27) | Si(2) | 119.6(2) | | | | |

Table 7. Bond angles involving hydrogens (°)

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| N(1) | C(1) | H(1) | 109.4 | N(1) | C(1) | H(2) | 109.4 |
| C(2) | C(1) | H(1) | 109.4 | C(2) | C(1) | H(2) | 109.4 |
| H(1) | C(1) | H(2) | 108.0 | C(2) | C(3) | H(3) | 120.1 |
| C(4) | C(3) | H(3) | 120.1 | C(3) | C(4) | H(4) | 121.2 |
| C(5) | C(4) | H(4) | 121.2 | C(4) | C(5) | H(5) | 120.2 |
| C(6) | C(5) | H(5) | 120.2 | N(2) | C(6) | H(6) | 119.0 |
| C(5) | C(6) | H(6) | 119.0 | C(8) | C(9) | H(7) | 119.3 |
| C(10) | C(9) | H(7) | 119.3 | C(9) | C(10) | H(8) | 120.4 |
| C(11) | C(10) | H(8) | 120.4 | C(10) | C(11) | H(9) | 118.6 |
| C(12) | C(11) | H(9) | 118.6 | C(8) | C(13) | H(10) | 108.0 |
| C(31) | C(13) | H(10) | 108.0 | C(32) | C(13) | H(10) | 108.0 |
| C(12) | C(14) | H(11) | 107.7 | C(33) | C(14) | H(11) | 107.7 |
| C(34) | C(14) | H(11) | 107.7 | C(16) | C(17) | H(12) | 119.4 |
| C(18) | C(17) | H(12) | 119.4 | C(17) | C(18) | H(13) | 120.3 |
| C(19) | C(18) | H(13) | 120.3 | C(18) | C(19) | H(14) | 119.2 |
| C(20) | C(19) | H(14) | 119.2 | C(16) | C(21) | H(15) | 109.5 |
| C(16) | C(21) | H(16) | 109.5 | C(16) | C(21) | H(17) | 109.5 |
| H(15) | C(21) | H(16) | 109.5 | H(15) | C(21) | H(17) | 109.5 |
| H(16) | C(21) | H(17) | 109.5 | C(20) | C(22) | H(18) | 109.5 |
| C(20) | C(22) | H(19) | 109.5 | C(20) | C(22) | H(20) | 109.5 |
| H(18) | C(22) | H(19) | 109.5 | H(18) | C(22) | H(20) | 109.5 |
| H(19) | C(22) | H(20) | 109.5 | V(1) | C(23) | H(21) | 105.2 |
| V(1) | C(23) | H(22) | 105.2 | Si(1) | C(23) | H(21) | 105.2 |
| Si(1) | C(23) | H(22) | 105.2 | H(21) | C(23) | H(22) | 105.9 |
| Si(1) | C(24) | H(23) | 109.5 | Si(1) | C(24) | H(24) | 109.5 |
| Si(1) | C(24) | H(25) | 109.5 | H(23) | C(24) | H(24) | 109.5 |
| H(23) | C(24) | H(25) | 109.5 | H(24) | C(24) | H(25) | 109.5 |
| Si(1) | C(25) | H(26) | 109.5 | Si(1) | C(25) | H(27) | 109.5 |
| Si(1) | C(25) | H(28) | 109.5 | H(26) | C(25) | H(27) | 109.5 |
| H(26) | C(25) | H(28) | 109.5 | H(27) | C(25) | H(28) | 109.5 |
| Si(1) | C(26) | H(29) | 109.5 | Si(1) | C(26) | H(30) | 109.5 |
| Si(1) | C(26) | H(31) | 109.5 | H(29) | C(26) | H(30) | 109.5 |
| H(29) | C(26) | H(31) | 109.5 | H(30) | C(26) | H(31) | 109.5 |
| V(1) | C(27) | H(32) | 107.4 | V(1) | C(27) | H(33) | 107.4 |
| Si(2) | C(27) | H(32) | 107.4 | Si(2) | C(27) | H(33) | 107.4 |
| H(32) | C(27) | H(33) | 106.9 | Si(2) | C(28) | H(34) | 109.5 |
| Si(2) | C(28) | H(35) | 109.5 | Si(2) | C(28) | H(36) | 109.5 |

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

| atom | atom | atom | angle | atom | atom | atom | angle |
|---|---|---|---|---|---|---|---|
| H(34) | C(28) | H(35) | 109.5 | H(34) | C(28) | H(36) | 109.5 |
| H(35) | C(28) | H(36) | 109.5 | Si(2) | C(29) | H(37) | 109.5 |
| Si(2) | C(29) | H(38) | 109.5 | Si(2) | C(29) | H(39) | 109.5 |
| H(37) | C(29) | H(38) | 109.5 | H(37) | C(29) | H(39) | 109.5 |
| H(38) | C(29) | H(39) | 109.5 | Si(2) | C(30) | H(40) | 109.5 |
| Si(2) | C(30) | H(41) | 109.5 | Si(2) | C(30) | H(42) | 109.5 |
| H(40) | C(30) | H(41) | 109.5 | H(40) | C(30) | H(42) | 109.5 |
| H(41) | C(30) | H(42) | 109.5 | C(13) | C(31) | H(43) | 109.5 |
| C(13) | C(31) | H(44) | 109.5 | C(13) | C(31) | H(45) | 109.5 |
| H(43) | C(31) | H(44) | 109.5 | H(43) | C(31) | H(45) | 109.5 |
| H(44) | C(31) | H(45) | 109.5 | C(13) | C(32) | H(46) | 109.5 |
| C(13) | C(32) | H(47) | 109.5 | C(13) | C(32) | H(48) | 109.5 |
| H(46) | C(32) | H(47) | 109.5 | H(46) | C(32) | H(48) | 109.5 |
| H(47) | C(32) | H(48) | 109.5 | C(14) | C(33) | H(49) | 109.5 |
| C(14) | C(33) | H(50) | 109.5 | C(14) | C(33) | H(51) | 109.5 |
| H(49) | C(33) | H(50) | 109.5 | H(49) | C(33) | H(51) | 109.5 |
| H(50) | C(33) | H(51) | 109.5 | C(14) | C(34) | H(52) | 109.5 |
| C(14) | C(34) | H(53) | 109.5 | C(14) | C(34) | H(54) | 109.5 |
| H(52) | C(34) | H(53) | 109.5 | H(52) | C(34) | H(54) | 109.5 |
| H(53) | C(34) | H(54) | 109.5 | | | | |

Table 8. Torsion Angles(°)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| N(1) | V(1) | N(2) | C(2) | -10.1(3) |
| N(2) | V(1) | N(1) | C(1) | 14.9(3) |
| N(1) | V(1) | N(3) | C(15) | -23(22) |
| N(3) | V(1) | N(1) | C(7) | 25.1(4) |
| C(23) | V(1) | N(1) | C(1) | -55.3(4) |
| N(1) | V(1) | C(27) | Si(2) | 168.1(2) |
| C(27) | V(1) | N(1) | C(7) | -75.9(4) |
| N(3) | V(1) | N(2) | C(2) | -15(2) |
| N(2) | V(1) | C(23) | Si(1) | -70.6(3) |
| C(23) | V(1) | N(2) | C(6) | -57.9(4) |
| C(27) | V(1) | N(2) | C(2) | -125.4(3) |
| N(3) | V(1) | C(23) | Si(1) | 106.2(3) |
| N(3) | V(1) | C(27) | Si(2) | 64.8(3) |
| C(23) | V(1) | C(27) | Si(2) | -39.7(3) |
| C(24) | Si(1) | C(23) | V(1) | 56.8(5) |
| C(26) | Si(1) | C(23) | V(1) | 176.1(4) |
| C(29) | Si(2) | C(27) | V(1) | 82.2(4) |
| V(1) | N(1) | C(1) | C(2) | -17.4(5) |
| V(1) | N(1) | C(7) | C(12) | -83.3(5) |
| C(1) | N(1) | C(7) | C(12) | 106.3(4) |
| V(1) | N(2) | C(2) | C(1) | 4.0(5) |
| V(1) | N(2) | C(6) | C(5) | 175.9(4) |
| C(6) | N(2) | C(2) | C(1) | -179.0(4) |
| V(1) | N(3) | C(15) | C(16) | -44(22) |
| N(1) | C(1) | C(2) | N(2) | 6.7(6) |
| N(2) | C(2) | C(3) | C(4) | 0.1(6) |
| C(2) | C(3) | C(4) | C(5) | -0.5(8) |
| C(4) | C(5) | C(6) | N(2) | 0.3(7) |
| N(1) | C(7) | C(8) | C(13) | -1.5(6) |
| N(1) | C(7) | C(12) | C(14) | -1.6(6) |
| C(8) | C(7) | C(12) | C(14) | 178.0(4) |
| C(12) | C(7) | C(8) | C(13) | 179.0(4) |
| C(7) | C(8) | C(13) | C(31) | 117.7(5) |
| C(9) | C(8) | C(13) | C(31) | -62.8(6) |
| C(13) | C(8) | C(9) | C(10) | -178.2(4) |
| C(9) | C(10) | C(11) | C(12) | 0.3(6) |
| C(10) | C(11) | C(12) | C(14) | -177.9(4) |
| N(1) | V(1) | N(2) | C(6) | 173.2(4) |
| N(2) | V(1) | N(1) | C(7) | -154.5(4) |
| N(3) | V(1) | N(1) | C(1) | -165.5(3) |
| N(1) | V(1) | C(23) | Si(1) | -3.6(4) |
| C(23) | V(1) | N(1) | C(7) | 135.2(4) |
| C(27) | V(1) | N(1) | C(1) | 93.6(4) |
| N(2) | V(1) | N(3) | C(15) | -18(24) |
| N(3) | V(1) | N(2) | C(6) | 169(2) |
| C(23) | V(1) | N(2) | C(2) | 118.8(3) |
| N(2) | V(1) | C(27) | Si(2) | -119.4(3) |
| C(27) | V(1) | N(2) | C(6) | 57.9(4) |
| C(23) | V(1) | N(3) | C(15) | -151(22) |
| C(27) | V(1) | N(3) | C(15) | 92(22) |
| C(27) | V(1) | C(23) | Si(1) | -152.0(3) |
| C(25) | Si(1) | C(23) | V(1) | -66.1(4) |
| C(28) | Si(2) | C(27) | V(1) | -160.6(3) |
| C(30) | Si(2) | C(27) | V(1) | -39.6(4) |
| V(1) | N(1) | C(7) | C(8) | 97.2(4) |
| C(1) | N(1) | C(7) | C(8) | -73.3(5) |
| C(7) | N(1) | C(1) | C(2) | 153.5(4) |
| V(1) | N(2) | C(2) | C(3) | -176.5(4) |
| C(2) | N(2) | C(6) | C(5) | -0.7(8) |
| C(6) | N(2) | C(2) | C(3) | 0.5(7) |
| V(1) | N(3) | C(15) | C(20) | 135(22) |
| N(1) | C(1) | C(2) | C(3) | -172.8(4) |
| C(1) | C(2) | C(3) | C(4) | 179.5(5) |
| C(3) | C(4) | C(5) | C(6) | 0.3(8) |
| N(1) | C(7) | C(8) | C(9) | 179.0(4) |
| N(1) | C(7) | C(12) | C(11) | -179.9(3) |
| C(8) | C(7) | C(12) | C(11) | -0.3(6) |
| C(12) | C(7) | C(8) | C(9) | -0.5(6) |
| C(7) | C(8) | C(9) | C(10) | 1.3(7) |
| C(7) | C(8) | C(13) | C(32) | -119.4(5) |
| C(9) | C(8) | C(13) | C(32) | 60.1(5) |
| C(8) | C(9) | C(10) | C(11) | -1.2(7) |
| C(10) | C(11) | C(12) | C(7) | 0.5(7) |
| C(7) | C(12) | C(14) | C(33) | -87.9(6) |

Table 8. Torsion angles (°) (continued)

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| C(7) | C(12) | C(14) | C(34) | 148.2(4) |
| C(11) | C(12) | C(14) | C(34) | -33.5(6) |
| N(3) | C(15) | C(16) | C(21) | -5.0(7) |
| N(3) | C(15) | C(20) | C(22) | 4.8(8) |
| C(16) | C(15) | C(20) | C(22) | -176.7(5) |
| C(20) | C(15) | C(16) | C(21) | 176.4(5) |
| C(21) | C(16) | C(17) | C(18) | -178.3(5) |
| C(17) | C(18) | C(19) | C(20) | -1.0(9) |
| C(18) | C(19) | C(20) | C(22) | 178.6(5) |

| atom1 | atom2 | atom3 | atom4 | angle |
|---|---|---|---|---|
| C(11) | C(12) | C(14) | C(33) | 90.3(6) |
| N(3) | C(15) | C(16) | C(17) | 175.9(4) |
| N(3) | C(15) | C(20) | C(19) | -175.9(4) |
| C(16) | C(15) | C(20) | C(19) | 2.6(8) |
| C(20) | C(15) | C(16) | C(17) | -2.6(8) |
| C(15) | C(16) | C(17) | C(18) | 0.8(8) |
| C(16) | C(17) | C(18) | C(19) | 1.0(9) |
| C(18) | C(19) | C(20) | C(15) | -0.7(8) |

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 |
|---|---|---|---|---|---|
| C(10) | C(17)[1)] | 3.598(8) | C(17) | C(10)[1)] | 3.598(8) |
| C(32) | C(32)[2)] | 3.345(7) | | | |

Symmetry Operators:

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

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| Si(2) | H(14)[1)] | 3.551 | C(1) | H(34)[2)] | 3.599 |
| C(1) | H(43)[3)] | 3.166 | C(2) | H(34)[2)] | 3.563 |
| C(3) | H(8)[3)] | 3.483 | C(3) | H(34)[2)] | 3.220 |
| C(3) | H(53)[1)] | 3.096 | C(4) | H(23)[4)] | 3.280 |
| C(4) | H(29)[4)] | 3.260 | C(4) | H(51)[1)] | 3.442 |
| C(4) | H(53)[1)] | 2.846 | C(5) | H(18)[1)] | 3.435 |
| C(5) | H(19)[1)] | 3.226 | C(5) | H(23)[4)] | 3.279 |
| C(5) | H(27)[1)] | 3.248 | C(5) | H(37)[5)] | 3.277 |
| C(5) | H(39)[5)] | 3.393 | C(5) | H(53)[1)] | 3.191 |
| C(6) | H(19)[1)] | 3.354 | C(6) | H(37)[5)] | 3.163 |
| C(6) | H(39)[5)] | 3.225 | C(7) | H(43)[3)] | 3.476 |
| C(7) | H(44)[3)] | 3.149 | C(8) | H(44)[3)] | 2.932 |
| C(9) | H(1)[3)] | 3.277 | C(9) | H(3)[3)] | 3.420 |
| C(9) | H(13)[6)] | 3.190 | C(9) | H(44)[3)] | 2.842 |
| C(10) | H(3)[3)] | 3.277 | C(10) | H(12)[6)] | 3.006 |
| C(10) | H(13)[6)] | 3.288 | C(10) | H(44)[3)] | 3.022 |
| C(10) | H(50)[7)] | 3.555 | C(10) | H(54)[7)] | 3.561 |
| C(11) | H(12)[6)] | 3.381 | C(11) | H(44)[3)] | 3.228 |
| C(11) | H(45)[3)] | 3.581 | C(11) | H(50)[7)] | 3.324 |
| C(11) | H(54)[7)] | 3.430 | C(12) | H(44)[3)] | 3.312 |
| C(16) | H(12)[6)] | 3.502 | C(17) | H(8)[6)] | 3.236 |
| C(17) | H(12)[6)] | 3.298 | C(17) | H(17)[6)] | 3.253 |
| C(18) | H(8)[6)] | 3.319 | C(18) | H(17)[6)] | 3.595 |
| C(18) | H(36)[8)] | 3.057 | C(18) | H(46)[8)] | 3.479 |
| C(19) | H(30)[9)] | 3.559 | C(19) | H(31)[9)] | 3.553 |
| C(19) | H(32)[8)] | 3.247 | C(19) | H(36)[8)] | 3.165 |
| C(20) | H(30)[9)] | 3.301 | C(21) | H(12)[6)] | 3.173 |
| C(21) | H(46)[10)] | 3.304 | C(21) | H(48)[10)] | 3.381 |
| C(22) | H(5)[8)] | 3.355 | C(22) | H(30)[9)] | 3.128 |
| C(24) | H(24)[4)] | 3.598 | C(24) | H(34)[2)] | 3.246 |
| C(25) | H(4)[8)] | 3.402 | C(25) | H(5)[8)] | 3.272 |
| C(25) | H(28)[11)] | 3.351 | C(26) | H(4)[4)] | 3.207 |
| C(26) | H(20)[9)] | 3.498 | C(27) | H(14)[1)] | 3.275 |
| C(28) | H(2)[12)] | 3.341 | C(28) | H(3)[12)] | 3.247 |
| C(28) | H(7)[10)] | 3.291 | C(28) | H(13)[1)] | 3.354 |
| C(28) | H(14)[1)] | 3.459 | C(28) | H(25)[12)] | 3.400 |
| C(29) | H(5)[5)] | 3.377 | C(29) | H(6)[5)] | 2.994 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| C(29) | H(14)[1)] | 3.468 | C(29) | H(21)[5)] | 3.503 |
| C(29) | H(31)[5)] | 3.482 | C(29) | H(38)[5)] | 3.582 |
| C(29) | H(39)[5)] | 3.520 | C(30) | H(30)[9)] | 3.321 |
| C(31) | H(1)[3)] | 3.308 | C(31) | H(44)[3)] | 3.523 |
| C(31) | H(49)[3)] | 3.575 | C(31) | H(50)[3)] | 3.389 |
| C(31) | H(54)[1)] | 3.342 | C(32) | H(16)[10)] | 3.533 |
| C(32) | H(17)[10)] | 3.342 | C(32) | H(46)[10)] | 3.452 |
| C(32) | H(47)[10)] | 2.854 | C(32) | H(48)[10)] | 3.202 |
| C(33) | H(8)[7)] | 3.595 | C(33) | H(9)[7)] | 3.482 |
| C(33) | H(43)[3)] | 3.449 | C(33) | H(45)[3)] | 3.588 |
| C(34) | H(45)[8)] | 3.279 | H(1) | C(9)[3)] | 3.277 |
| H(1) | C(31)[3)] | 3.308 | H(1) | H(7)[3)] | 2.987 |
| H(1) | H(43)[3)] | 2.681 | H(1) | H(44)[3)] | 3.069 |
| H(2) | C(28)[2)] | 3.341 | H(2) | H(7)[3)] | 3.433 |
| H(2) | H(34)[2)] | 2.868 | H(2) | H(35)[2)] | 2.950 |
| H(2) | H(43)[3)] | 2.786 | H(3) | C(9)[3)] | 3.420 |
| H(3) | C(10)[3)] | 3.277 | H(3) | C(28)[2)] | 3.247 |
| H(3) | H(7)[3)] | 2.968 | H(3) | H(8)[3)] | 2.707 |
| H(3) | H(34)[2)] | 2.847 | H(3) | H(35)[2)] | 3.431 |
| H(3) | H(36)[2)] | 2.954 | H(3) | H(53)[1)] | 3.415 |
| H(4) | C(25)[1)] | 3.402 | H(4) | C(26)[4)] | 3.207 |
| H(4) | H(23)[4)] | 3.093 | H(4) | H(26)[1)] | 3.152 |
| H(4) | H(27)[1)] | 3.247 | H(4) | H(28)[1)] | 3.234 |
| H(4) | H(29)[4)] | 2.422 | H(4) | H(31)[4)] | 3.196 |
| H(4) | H(51)[1)] | 2.914 | H(4) | H(53)[1)] | 3.031 |
| H(5) | C(22)[1)] | 3.355 | H(5) | C(25)[1)] | 3.272 |
| H(5) | C(29)[5)] | 3.377 | H(5) | H(18)[1)] | 2.985 |
| H(5) | H(19)[1)] | 2.823 | H(5) | H(23)[4)] | 3.086 |
| H(5) | H(26)[1)] | 3.389 | H(5) | H(27)[1)] | 2.489 |
| H(5) | H(28)[1)] | 3.520 | H(5) | H(37)[5)] | 2.981 |
| H(5) | H(39)[5)] | 2.894 | H(5) | H(40)[5)] | 3.257 |
| H(5) | H(53)[1)] | 3.560 | H(6) | C(29)[5)] | 2.994 |
| H(6) | H(14)[1)] | 3.514 | H(6) | H(19)[1)] | 3.050 |
| H(6) | H(37)[5)] | 2.756 | H(6) | H(38)[5)] | 3.237 |
| H(6) | H(39)[5)] | 2.533 | H(7) | C(28)[10)] | 3.291 |
| H(7) | H(1)[3)] | 2.987 | H(7) | H(2)[3)] | 3.433 |
| H(7) | H(3)[3)] | 2.968 | H(7) | H(13)[6)] | 2.837 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(7) | H(35)[10)] | 2.788 | H(7) | H(36)[10)] | 2.972 |
| H(7) | H(44)[3)] | 3.253 | H(8) | C(3)[3)] | 3.483 |
| H(8) | C(17)[6)] | 3.236 | H(8) | C(18)[6)] | 3.319 |
| H(8) | C(33)[7)] | 3.595 | H(8) | H(3)[3)] | 2.707 |
| H(8) | H(12)[6)] | 2.841 | H(8) | H(13)[6)] | 3.016 |
| H(8) | H(44)[3)] | 3.529 | H(8) | H(50)[7)] | 3.067 |
| H(8) | H(51)[7)] | 3.230 | H(8) | H(54)[7)] | 3.378 |
| H(9) | C(33)[7)] | 3.482 | H(9) | H(9)[7)] | 3.123 |
| H(9) | H(12)[6)] | 3.472 | H(9) | H(45)[8)] | 3.259 |
| H(9) | H(50)[7)] | 2.568 | H(9) | H(54)[7)] | 3.123 |
| H(12) | C(10)[6)] | 3.006 | H(12) | C(11)[6)] | 3.381 |
| H(12) | C(16)[6)] | 3.502 | H(12) | C(17)[6)] | 3.298 |
| H(12) | C(21)[6)] | 3.173 | H(12) | H(8)[6)] | 2.841 |
| H(12) | H(9)[6)] | 3.472 | H(12) | H(12)[6)] | 2.819 |
| H(12) | H(15)[6)] | 2.901 | H(12) | H(17)[6)] | 2.792 |
| H(13) | C(9)[6)] | 3.190 | H(13) | C(10)[6)] | 3.288 |
| H(13) | C(28)[8)] | 3.354 | H(13) | H(7)[6)] | 2.837 |
| H(13) | H(8)[6)] | 3.016 | H(13) | H(15)[6)] | 3.472 |
| H(13) | H(17)[6)] | 3.422 | H(13) | H(32)[8)] | 3.553 |
| H(13) | H(35)[8)] | 3.478 | H(13) | H(36)[8)] | 2.486 |
| H(13) | H(46)[8)] | 3.045 | H(14) | Si(2)[8)] | 3.551 |
| H(14) | C(27)[8)] | 3.275 | H(14) | C(28)[8)] | 3.459 |
| H(14) | C(29)[8)] | 3.468 | H(14) | H(6)[8)] | 3.514 |
| H(14) | H(30)[9)] | 3.567 | H(14) | H(31)[9)] | 3.264 |
| H(14) | H(32)[8)] | 2.478 | H(14) | H(33)[8)] | 3.553 |
| H(14) | H(36)[8)] | 2.682 | H(14) | H(38)[8)] | 2.781 |
| H(15) | H(12)[6)] | 2.901 | H(15) | H(13)[6)] | 3.472 |
| H(15) | H(46)[10)] | 3.427 | H(16) | C(32)[10)] | 3.533 |
| H(16) | H(46)[10)] | 3.288 | H(16) | H(48)[10)] | 2.897 |
| H(17) | C(17)[6)] | 3.253 | H(17) | C(18)[6)] | 3.595 |
| H(17) | C(32)[10)] | 3.342 | H(17) | H(12)[6)] | 2.792 |
| H(17) | H(13)[6)] | 3.422 | H(17) | H(46)[10)] | 2.697 |
| H(17) | H(48)[10)] | 3.099 | H(18) | C(5)[8)] | 3.435 |
| H(18) | H(5)[8)] | 2.985 | H(19) | C(5)[8)] | 3.226 |
| H(19) | C(6)[8)] | 3.354 | H(19) | H(5)[8)] | 2.823 |
| H(19) | H(6)[8)] | 3.050 | H(19) | H(30)[9)] | 3.157 |
| H(19) | H(38)[8)] | 3.103 | H(19) | H(39)[9)] | 3.327 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(20) | C(26)[9)] | 3.498 | H(20) | H(22)[9)] | 3.445 |
| H(20) | H(30)[9)] | 2.573 | H(21) | C(29)[5)] | 3.503 |
| H(21) | H(37)[5)] | 3.159 | H(21) | H(38)[5)] | 3.153 |
| H(22) | H(20)[9)] | 3.445 | H(23) | C(4)[4)] | 3.280 |
| H(23) | C(5)[4)] | 3.279 | H(23) | H(4)[4)] | 3.093 |
| H(23) | H(5)[4)] | 3.086 | H(23) | H(24)[4)] | 3.093 |
| H(23) | H(34)[2)] | 3.346 | H(23) | H(37)[2)] | 3.455 |
| H(23) | H(40)[2)] | 3.507 | H(24) | C(24)[4)] | 3.598 |
| H(24) | H(23)[4)] | 3.093 | H(24) | H(24)[4)] | 3.211 |
| H(24) | H(34)[2)] | 3.519 | H(24) | H(37)[5)] | 3.160 |
| H(25) | C(28)[2)] | 3.400 | H(25) | H(34)[2)] | 2.462 |
| H(25) | H(40)[2)] | 3.550 | H(26) | H(4)[8)] | 3.152 |
| H(26) | H(5)[8)] | 3.389 | H(27) | C(5)[8)] | 3.248 |
| H(27) | H(4)[8)] | 3.247 | H(27) | H(5)[8)] | 2.489 |
| H(27) | H(28)[11)] | 3.408 | H(27) | H(40)[9)] | 3.107 |
| H(28) | C(25)[11)] | 3.351 | H(28) | H(4)[8)] | 3.234 |
| H(28) | H(5)[8)] | 3.520 | H(28) | H(27)[11)] | 3.408 |
| H(28) | H(28)[11)] | 2.534 | H(28) | H(29)[11)] | 3.111 |
| H(28) | H(40)[2)] | 3.485 | H(29) | C(4)[4)] | 3.260 |
| H(29) | H(4)[4)] | 2.422 | H(29) | H(28)[11)] | 3.111 |
| H(29) | H(40)[9)] | 3.337 | H(29) | H(42)[9)] | 3.554 |
| H(30) | C(19)[9)] | 3.559 | H(30) | C(20)[9)] | 3.301 |
| H(30) | C(22)[9)] | 3.128 | H(30) | C(30)[9)] | 3.321 |
| H(30) | H(14)[9)] | 3.567 | H(30) | H(19)[9)] | 3.157 |
| H(30) | H(20)[9)] | 2.573 | H(30) | H(40)[9)] | 3.162 |
| H(30) | H(41)[9)] | 2.849 | H(30) | H(42)[9)] | 3.426 |
| H(31) | C(19)[9)] | 3.553 | H(31) | C(29)[5)] | 3.482 |
| H(31) | H(4)[4)] | 3.196 | H(31) | H(14)[9)] | 3.264 |
| H(31) | H(36)[5)] | 3.391 | H(31) | H(37)[5)] | 2.912 |
| H(31) | H(38)[5)] | 3.195 | H(32) | C(19)[1)] | 3.247 |
| H(32) | H(13)[1)] | 3.553 | H(32) | H(14)[1)] | 2.478 |
| H(33) | H(14)[1)] | 3.553 | H(33) | H(48)[10)] | 3.395 |
| H(34) | C(1)[12)] | 3.599 | H(34) | C(2)[12)] | 3.563 |
| H(34) | C(3)[12)] | 3.220 | H(34) | C(24)[12)] | 3.246 |
| H(34) | H(2)[12)] | 2.868 | H(34) | H(3)[12)] | 2.847 |
| H(34) | H(23)[12)] | 3.346 | H(34) | H(24)[12)] | 3.519 |
| H(34) | H(25)[12)] | 2.462 | H(35) | H(2)[12)] | 2.950 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(35) | H(3)[12)] | 3.431 | H(35) | H(7)[10)] | 2.788 |
| H(35) | H(13)[1)] | 3.478 | H(35) | H(43)[10)] | 3.050 |
| H(35) | H(48)[10)] | 2.910 | H(35) | H(49)[12)] | 3.414 |
| H(36) | C(18)[1)] | 3.057 | H(36) | C(19)[1)] | 3.165 |
| H(36) | H(3)[12)] | 2.954 | H(36) | H(7)[10)] | 2.972 |
| H(36) | H(13)[1)] | 2.486 | H(36) | H(14)[1)] | 2.682 |
| H(36) | H(31)[5)] | 3.391 | H(37) | C(5)[5)] | 3.277 |
| H(37) | C(6)[5)] | 3.163 | H(37) | H(5)[5)] | 2.981 |
| H(37) | H(6)[5)] | 2.756 | H(37) | H(21)[5)] | 3.159 |
| H(37) | H(23)[12)] | 3.455 | H(37) | H(24)[5)] | 3.160 |
| H(37) | H(31)[5)] | 2.912 | H(38) | C(29)[5)] | 3.582 |
| H(38) | H(6)[5)] | 3.237 | H(38) | H(14)[1)] | 2.781 |
| H(38) | H(19)[1)] | 3.103 | H(38) | H(21)[5)] | 3.153 |
| H(38) | H(31)[5)] | 3.195 | H(38) | H(38)[5)] | 3.398 |
| H(38) | H(39)[5)] | 2.922 | H(39) | C(5)[5)] | 3.393 |
| H(39) | C(6)[5)] | 3.225 | H(39) | C(29)[5)] | 3.520 |
| H(39) | H(5)[5)] | 2.894 | H(39) | H(6)[5)] | 2.533 |
| H(39) | H(19)[9)] | 3.327 | H(39) | H(38)[5)] | 2.922 |
| H(39) | H(39)[5)] | 3.265 | H(40) | H(5)[5)] | 3.257 |
| H(40) | H(23)[12)] | 3.507 | H(40) | H(25)[12)] | 3.550 |
| H(40) | H(27)[9)] | 3.107 | H(40) | H(28)[12)] | 3.485 |
| H(40) | H(29)[9)] | 3.337 | H(40) | H(30)[9)] | 3.162 |
| H(41) | H(30)[9)] | 2.849 | H(42) | H(29)[9)] | 3.554 |
| H(42) | H(30)[9)] | 3.426 | H(42) | H(49)[12)] | 3.528 |
| H(43) | C(1)[3)] | 3.166 | H(43) | C(7)[3)] | 3.476 |
| H(43) | C(33)[3)] | 3.449 | H(43) | H(1)[3)] | 2.681 |
| H(43) | H(2)[3)] | 2.786 | H(43) | H(35)[10)] | 3.050 |
| H(43) | H(44)[3)] | 3.452 | H(43) | H(49)[3)] | 2.919 |
| H(43) | H(50)[3)] | 3.138 | H(44) | C(7)[3)] | 3.149 |
| H(44) | C(8)[3)] | 2.932 | H(44) | C(9)[3)] | 2.842 |
| H(44) | C(10)[3)] | 3.022 | H(44) | C(11)[3)] | 3.228 |
| H(44) | C(12)[3)] | 3.312 | H(44) | C(31)[3)] | 3.523 |
| H(44) | H(1)[3)] | 3.069 | H(44) | H(7)[3)] | 3.253 |
| H(44) | H(8)[3)] | 3.529 | H(44) | H(43)[3)] | 3.452 |
| H(44) | H(44)[3)] | 2.958 | H(44) | H(54)[1)] | 3.373 |
| H(45) | C(11)[3)] | 3.581 | H(45) | C(33)[3)] | 3.588 |
| H(45) | C(34)[1)] | 3.279 | H(45) | H(9)[1)] | 3.259 |

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

| atom | atom | distance | atom | atom | distance |
|---|---|---|---|---|---|
| H(45) | H(49)[3)] | 3.476 | H(45) | H(50)[3)] | 2.879 |
| H(45) | H(52)[1)] | 3.280 | H(45) | H(54)[1)] | 2.528 |
| H(46) | C(18)[1)] | 3.479 | H(46) | C(21)[10)] | 3.304 |
| H(46) | C(32)[10)] | 3.452 | H(46) | H(13)[1)] | 3.045 |
| H(46) | H(15)[10)] | 3.427 | H(46) | H(16)[10)] | 3.288 |
| H(46) | H(17)[10)] | 2.697 | H(46) | H(47)[10)] | 2.869 |
| H(46) | H(48)[10)] | 3.215 | H(47) | C(32)[10)] | 2.854 |
| H(47) | H(46)[10)] | 2.869 | H(47) | H(47)[10)] | 2.651 |
| H(47) | H(48)[10)] | 2.563 | H(48) | C(21)[10)] | 3.381 |
| H(48) | C(32)[10)] | 3.202 | H(48) | H(16)[10)] | 2.897 |
| H(48) | H(17)[10)] | 3.099 | H(48) | H(33)[10)] | 3.395 |
| H(48) | H(35)[10)] | 2.910 | H(48) | H(46)[10)] | 3.215 |
| H(48) | H(47)[10)] | 2.563 | H(48) | H(48)[10)] | 3.352 |
| H(49) | C(31)[3)] | 3.575 | H(49) | H(35)[2)] | 3.414 |
| H(49) | H(42)[2)] | 3.528 | H(49) | H(43)[3)] | 2.919 |
| H(49) | H(45)[3)] | 3.476 | H(50) | C(10)[7)] | 3.555 |
| H(50) | C(11)[7)] | 3.324 | H(50) | C(31)[3)] | 3.389 |
| H(50) | H(8)[7)] | 3.067 | H(50) | H(9)[7)] | 2.568 |
| H(50) | H(43)[3)] | 3.138 | H(50) | H(45)[3)] | 2.879 |
| H(51) | C(4)[8)] | 3.442 | H(51) | H(4)[8)] | 2.914 |
| H(51) | H(8)[7)] | 3.230 | H(52) | H(45)[8)] | 3.280 |
| H(53) | C(3)[8)] | 3.096 | H(53) | C(4)[8)] | 2.846 |
| H(53) | C(5)[8)] | 3.191 | H(53) | H(3)[8)] | 3.415 |
| H(53) | H(4)[8)] | 3.031 | H(53) | H(5)[8)] | 3.560 |
| H(54) | C(10)[7)] | 3.561 | H(54) | C(11)[7)] | 3.430 |
| H(54) | C(31)[8)] | 3.342 | H(54) | H(8)[7)] | 3.378 |
| H(54) | H(9)[7)] | 3.123 | H(54) | H(44)[8)] | 3.373 |
| H(54) | H(45)[8)] | 2.528 | | | |

Symmetry Operators:

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