Vanadium (beta-(dimethylamino)ethyl)cyclopentadienyl complexes with diphenylacetylene ligands

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Publication date:
2008

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   3 -x+y,-x,z
   4 -x,-y,-z
   5 y,-x+y,-z
   6 x-y,x,-z
   7 1/3+x,2/3+y,2/3+z
   8 1/3-y,2/3+x-y,2/3+z
   9 1/3-x+y,2/3-x,2/3+z
  10 1/3-x,2/3-y,2/3-z
  11 1/3+y,2/3-x+y,2/3-z
  12 1/3+x-y,2/3+x,2/3-z
  13 2/3+x,1/3+y,1/3+z
  14 2/3-y,1/3+x-y,1/3+z
  15 2/3-x+y,1/3-x,1/3+z
  16 2/3-x,1/3-y,1/3-z
  17 2/3+y,1/3-x+y,1/3-z
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_computing_structure_solution     'SHELXS-97 (Sheldrick, 1997)'
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
 goodness of fit S are based on F^2^, conventional R-factors R are based
 on F, with F set to zero for negative F^2^. The threshold expression of
 F^2^ > 2sigma(F^2^) is used only for calculating R-factors(gt) etc. and is
 not relevant to the choice of reflections for refinement.  R-factors based
 on F^2^ are statistically about twice as large as those based on F, and R-
 factors based on ALL data will be even larger.
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Cl Cl Uani 0.13373(2) 0.22855(2) -0.12984(4) 1.000 0.0241(16) . .
N N Uani 0.17751(8) 0.20036(8) 0.10452(17) 1.000(4) 0.0274(6) . .
C1 C Uani 0.24031(8) 0.31703(9) 0.14132(2) 1.000(4) 0.0251(7) . .
C2 C Uani 0.22343(8) 0.34547(9) 0.08376(2) 1.000 0.0257(7) . .
C3 C Uani 0.22404(8) 0.33874(8) -0.04188(2) 1.000 0.0242(7) . .
C4 C Uani 0.24190(8) 0.30673(8) -0.06130(2) 1.000 0.0218(7) . .
C5 C Uani 0.25210(8) 0.29333(9) 0.05073(15) 1.000 0.0228(6) . .
C6 C Uani 0.26497(9) 0.25478(10) 0.06965(2) 1.000 0.0291(8) . .
C7 C Uani 0.22136(10) 0.20627(10) 0.03869(2) 1.000 0.0295(8) . .
C8 C Uani 0.13566(12) 0.15337(10) 0.06903(3) 1.000 0.0377(9) . .
C9 C Uani 0.18543(12) 0.19781(13) 0.23668(2) 1.000 0.0382(10) . .
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C12 C Uani 0.07693(9) 0.38656(9) 0.14167(2) 1.000 0.0275(7) . .
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C17 C Uani 0.11902(8) 0.24560(8) 0.17975(18) 1.000 0.0195(6) . .
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C20 C Uani 0.08299(12) 0.19711(10) 0.49544(2) 1.000 0.0367(9) . .
C21 C Uani 0.03704(12) 0.15848(11) 0.47796(3) 1.000 0.0423(10) . .
C22 C Uani 0.01762(11) 0.14791(12) 0.36269(3) 1.000 0.0446(9) . .
C23 C Uani 0.04416(9) 0.17589(10) 0.26485(2) 1.000 0.0344(8) . .
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H3 H Uiso 0.21387(-) 0.35410(-) -0.10219(-) 1.000 0.0448(11) . .
H4 H Uiso 0.24469(-) 0.29538(-) -0.13457(-) 1.000 0.0229(9) . .
H6 H Uiso 0.29163(-) 0.26086(-) 0.02153(-) 1.000 0.0282(10) . .
H6' H Uiso 0.27566(-) 0.25600(-) 0.15572(-) 1.000 0.0374(10) . .
H7 H Uiso 0.21493(-) 0.20569(-) -0.05076(-) 1.000 0.0365(10) . .
H7' H Uiso 0.22746(-) 0.17998(-) 0.06271(-) 1.000 0.0353(10) . .
H8 H Uiso 0.12930(-) 0.15429(-) -0.01613(-) 1.000 0.0288(10) . .
H8' H Uiso 0.10905(-) 0.14987(-) 0.10798(-) 1.000 0.0401(12) . .
H8" H Uiso 0.14219(-) 0.12843(-) 0.09112(-) 1.000 0.0417(11) . .
H9 H Uiso 0.19843(-) 0.17632(-) 0.24959(-) 1.000 0.0414(11) . .
H9' H Uiso 0.21031(-) 0.23032(-) 0.26554(-) 1.000 0.0588(14) . .
H9" H Uiso 0.15309(-) 0.18522(-) 0.28252(-) 1.000 0.0343(10) . .
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H11 H Uiso 0.03095(-) 0.34013(-) 0.26559(-) 1.000 0.0323(10) . .
H12 H Uiso 0.06483(-) 0.40916(-) 0.14750(-) 1.000 0.0396(11) . .
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H21 H Uiso 0.01797(-) 0.14062(-) 0.54280(-) 1.000 0.0487(12) . .
H22 H Uiso -0.01592(-) 0.12384(-) 0.35035(-) 1.000 0.0547(14) . .
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Cl 0.0231(3) 0.0298(3) 0.0162(2) -0.0050(2) -0.0013(19) 0.0110(2)
N 0.0375(12) 0.0306(11) 0.0224(9) 0.0045(8) 0.0087(8) 0.0232(10)
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C3 0.0170(11) 0.0244(12) 0.0296(12) 0.0048(9) 0.0034(9) 0.0092(9)
C4 0.0178(10) 0.0265(12) 0.0204(10) -0.0012(9) 0.0016(8) 0.0105(9)
C5 0.0174(10) 0.0313(12) 0.0218(10) -0.0053(9) -0.0020(8) 0.0138(10)
C6 0.0321(13) 0.0473(16) 0.0207(11) -0.0004(10) 0.0011(10) 0.0295(12)
C7 0.0413(15) 0.0371(14) 0.0252(11) 0.0047(10) 0.0095(10) 0.0308(13)
C8 0.0457(17) 0.0270(14) 0.0450(16) 0.0066(12) 0.0156(14) 0.0217(13)
C9 0.0576(19) 0.0573(19) 0.0242(12) 0.0157(12) 0.0130(12) 0.0472(18)
C10 0.0250(12) 0.0322(12) 0.0146(9) -0.0013(9) 0.0002(8) 0.0165(10)
C11 0.0326(13) 0.0464(15) 0.0182(10) -0.0053(10) -0.0010(9) 0.0289(12)
```

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C12 0.0352(13) 0.0379(14) 0.0219(10) -0.0077(10) -0.0075(9) 0.0276(12)
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C14 0.0190(1) 0.0268(12) 0.0171(9) -0.0023(8) -0.0019(8) 0.0121(9)
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C17 0.0196(10) 0.0238(11) 0.0153(9) -0.0030(8) -0.0010(8) 0.0109(9)
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C19 0.0362(14) 0.0302(13) 0.0183(10) -0.0022(9) 0.0021(10) 0.0182(12)
C20 0.0597(19) 0.0405(15) 0.0181(11) 0.0031(10) 0.0098(11) 0.0312(14)
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 Bond distances, angles etc. have been calculated using the
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 from the variances of the (full) variance-covariance matrix.
 The cell esds are taken into account in the estimation of
 distances, angles and torsion angles
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 V       C2          2.284(3)                      .       .   yes
 V       C3          2.351(2)                      .       .   yes
 V       C4          2.360(3)                      .       .   yes
 V       C5          2.305(3)                      .       .   yes
 V       C16         1.969(3)                      .       .   yes
 V       C17         2.003(2)                      .       .   yes
 N       C7          1.505(4)                      .       .   yes
 N       C8          1.479(4)                      .       .   yes
 N       C9          1.495(2)                      .       .   yes
 C1      C2          1.419(4)                      .       .   no
 C1      C5          1.417(3)                      .       .   no
 C2      C3          1.410(1)                      .       .   no
 C3      C4          1.415(4)                      .       .   no
 C4      C5          1.403(3)                      .       .   no
 C5      C6          1.496(4)                      .       .   no
 C6      C7          1.517(4)                      .       .   no
 C10     C11         1.389(4)                      .       .   no
 C10     C15         1.403(4)                      .       .   no
 C11     C12         1.390(4)                      .       .   no
 C12     C13         1.388(4)                      .       .   no
 C13     C14         1.389(4)                      .       .   no
 C14     C15         1.403(3)                      .       .   no
 C15     C16         1.461(4)                      .       .   no
 C16     C17         1.312(3)                      .       .   no
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 C18     C19         1.400(3)                      .       .   no
 C18     C23         1.399(4)                      .       .   no
 C19     C20         1.391(4)                      .       .   no
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C21	C22	1.386(2)	.	.	no	
C22	C23	1.391(4)	.	.	no	
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C3	H3	0.9800(-)	.	.	no	
C4	H4	0.9100(-)	.	.	no	
C6	H6	0.9400(-)	.	.	no	
C6	H6'	1.0100(-)	.	.	no	
C7	H7	1.0100(-)	.	.	no	
C7	H7'	0.9900(-)	.	.	no	
C8	H8	0.9700(-)	.	.	no	
C8	H8'	0.9100(-)	.	.	no	
C8	H8"	0.9500(-)	.	.	no	
C9	H9	0.9700(-)	.	.	no	
C9	H9'	0.9900(-)	.	.	no	
C9	H9"	1.0400(-)	.	.	no	
C10	H10	0.9700(-)	.	.	no	
C11	H11	0.9200(-)	.	.	no	
C12	H12	0.9800(-)	.	.	no	
C13	H13	0.9600(-)	.	.	no	
C14	H14	0.9500(-)	.	.	no	
C19	H19	0.9500(-)	.	.	no	
C20	H20	0.9500(-)	.	.	no	
C21	H21	0.9300(-)	.	.	no	
C22	H22	0.9700(-)	.	.	no	
C23	H23	0.9300(-)	.	.	no	

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Cl	V	C2	126.74(6)	.	.	.	yes
Cl	V	C3	92.71(6)	.	.	.	yes
Cl	V	C4	85.29(6)	.	.	.	yes
Cl	V	C5	111.71(6)	.	.	.	yes
Cl	V	C16	103.43(7)	.	.	.	yes
Cl	V	C17	109.09(7)	.	.	.	yes
N	V	C1	92.34(9)	.	.	.	yes
N	V	C2	128.75(10)	.	.	.	yes
N	V	C3	131.6(1)	.	.	.	yes
N	V	C4	97.57(9)	.	.	.	yes
N	V	C5	75.01(9)	.	.	.	yes
N	V	C16	127.19(9)	.	.	.	yes
N	V	C17	88.66(9)	.	.	.	yes
C1	V	C2	36.55(10)	.	.	.	yes
C1	V	C3	59.70(8)	.	.	.	yes
C1	V	C4	59.16(8)	.	.	.	yes
C1	V	C5	36.29(9)	.	.	.	yes
C1	V	C16	102.76(10)	.	.	.	yes
C1	V	C17	106.43(9)	.	.	.	yes
C2	V	C3	35.38(3)	.	.	.	yes
C2	V	C4	58.74(9)	.	.	.	yes
C2	V	C5	60.05(10)	.	.	.	yes

C2	V	C16	81.35(10)	.	.	.	yes
C2	V	C17	106.30(9)	.	.	.	yes
C3	V	C4	34.94(9)	.	.	.	yes
C3	V	C5	59.01(9)	.	.	.	yes
C3	V	C16	98.73(9)	.	.	.	yes
C3	V	C17	134.58(9)	.	.	.	yes
C4	V	C5	34.98(8)	.	.	.	yes
C4	V	C16	133.65(9)	.	.	.	yes
C4	V	C17	164.34(8)	.	.	.	yes
C5	V	C16	138.36(9)	.	.	.	yes
C5	V	C17	135.92(9)	.	.	.	yes
C16	V	C17	38.56(9)	.	.	.	yes
V	N	C7	106.13(15)	.	.	.	yes
V	N	C8	113.9(2)	.	.	.	yes
V	N	C9	113.18(19)	.	.	.	yes
C7	N	C8	108.1(2)	.	.	.	yes
C7	N	C9	108.0(3)	.	.	.	yes
C8	N	C9	107.3(2)	.	.	.	yes
V	C1	C2	73.44(15)	.	.	.	yes
V	C1	C5	74.40(15)	.	.	.	yes
C2	C1	C5	108.2(2)	.	.	.	no
V	C2	C1	70.01(14)	.	.	.	yes
V	C2	C3	74.94(14)	.	.	.	yes
C1	C2	C3	107.9(2)	.	.	.	no
V	C3	C2	69.68(14)	.	.	.	yes
V	C3	C4	72.87(13)	.	.	.	yes
C2	C3	C4	107.6(2)	.	.	.	no
V	C4	C3	72.19(14)	.	.	.	yes
V	C4	C5	70.36(15)	.	.	.	yes
C3	C4	C5	109.0(2)	.	.	.	no
V	C5	C1	69.31(16)	.	.	.	yes
V	C5	C4	74.66(16)	.	.	.	yes
V	C5	C6	113.92(18)	.	.	.	yes
C1	C5	C4	107.4(2)	.	.	.	no
C1	C5	C6	126.6(2)	.	.	.	no
C4	C5	C6	125.2(2)	.	.	.	no
C5	C6	C7	108.6(3)	.	.	.	no
N	C7	C6	109.9(2)	.	.	.	yes
C11	C10	C15	120.6(2)	.	.	.	no
C10	C11	C12	120.3(3)	.	.	.	no
C11	C12	C13	120.0(3)	.	.	.	no
C12	C13	C14	119.7(2)	.	.	.	no
C13	C14	C15	121.2(2)	.	.	.	no
C10	C15	C14	118.2(2)	.	.	.	no
C10	C15	C16	120.5(2)	.	.	.	no
C14	C15	C16	121.3(2)	.	.	.	no
V	C16	C15	148.42(17)	.	.	.	yes
V	C16	C17	72.13(16)	.	.	.	yes
C15	C16	C17	139.1(2)	.	.	.	no
V	C17	C16	69.31(14)	.	.	.	yes
V	C17	C18	152.3(2)	.	.	.	yes
C16	C17	C18	138.2(3)	.	.	.	no
C17	C18	C19	120.2(2)	.	.	.	no
C17	C18	C23	121.3(2)	.	.	.	no
C19	C18	C23	118.5(2)	.	.	.	no
C18	C19	C20	120.4(3)	.	.	.	no
C19	C20	C21	120.7(3)	.	.	.	no
C20	C21	C22	119.6(3)	.	.	.	no
C21	C22	C23	120.4(3)	.	.	.	no
C18	C23	C22	120.5(2)	.	.	.	no
V	C1	H1	118.00(-)	.	.	.	no

C2	C1	H1	129.00(-)	.	.	.	no
C5	C1	H1	123.00(-)	.	.	.	no
V	C2	H2	122.00(-)	.	.	.	no
C1	C2	H2	126.00(-)	.	.	.	no
C3	C2	H2	126.00(-)	.	.	.	no
V	C3	H3	123.00(-)	.	.	.	no
C2	C3	H3	124.00(-)	.	.	.	no
C4	C3	H3	128.00(-)	.	.	.	no
V	C4	H4	121.00(-)	.	.	.	no
C3	C4	H4	125.00(-)	.	.	.	no
C5	C4	H4	126.00(-)	.	.	.	no
C5	C6	H6	110.00(-)	.	.	.	no
C5	C6	H6'	109.00(-)	.	.	.	no
C7	C6	H6	111.00(-)	.	.	.	no
C7	C6	H6'	112.00(-)	.	.	.	no
H6	C6	H6'	106.00(-)	.	.	.	no
N	C7	H7	108.00(-)	.	.	.	no
N	C7	H7'	109.00(-)	.	.	.	no
C6	C7	H7	108.00(-)	.	.	.	no
C6	C7	H7'	110.00(-)	.	.	.	no
H7	C7	H7'	112.00(-)	.	.	.	no
N	C8	H8	109.00(-)	.	.	.	no
N	C8	H8'	108.00(-)	.	.	.	no
N	C8	H8"	108.00(-)	.	.	.	no
H8	C8	H8'	106.00(-)	.	.	.	no
H8	C8	H8"	114.00(-)	.	.	.	no
H8'	C8	H8"	111.00(-)	.	.	.	no
N	C9	H9	109.00(-)	.	.	.	no
N	C9	H9'	109.00(-)	.	.	.	no
N	C9	H9"	110.00(-)	.	.	.	no
H9	C9	H9'	107.00(-)	.	.	.	no
H9	C9	H9"	110.00(-)	.	.	.	no
H9'	C9	H9"	112.00(-)	.	.	.	no
C11	C10	H10	121.00(-)	.	.	.	no
C15	C10	H10	118.00(-)	.	.	.	no
C10	C11	H11	120.00(-)	.	.	.	no
C12	C11	H11	120.00(-)	.	.	.	no
C11	C12	H12	118.00(-)	.	.	.	no
C13	C12	H12	122.00(-)	.	.	.	no
C12	C13	H13	120.00(-)	.	.	.	no
C14	C13	H13	120.00(-)	.	.	.	no
C13	C14	H14	119.00(-)	.	.	.	no
C15	C14	H14	120.00(-)	.	.	.	no
C18	C19	H19	123.00(-)	.	.	.	no
C20	C19	H19	117.00(-)	.	.	.	no
C19	C20	H20	117.00(-)	.	.	.	no
C21	C20	H20	123.00(-)	.	.	.	no
C20	C21	H21	121.00(-)	.	.	.	no
C22	C21	H21	119.00(-)	.	.	.	no
C21	C22	H22	120.00(-)	.	.	.	no
C23	C22	H22	119.00(-)	.	.	.	no
C18	C23	H23	120.00(-)	.	.	.	no
C22	C23	H23	119.00(-)	.	.	.	no

```
loop_
_geom_hbond_atom_site_label_D
_geom_hbond_atom_site_label_H
_geom_hbond_atom_site_label_A
_geom_hbond_distance_DH
_geom_hbond_distance_HA
```

```
_geom_hbond_distance_DA
_geom_hbond_angle_DHA
_geom_hbond_site_symmetry_A
_geom_hbond_publ_flag
#
#D   H   A   D - H  H...A   D...A    D - H...A  symm(A)
#
C8      H8      Cl     0.9700     2.6300     3.282(2)  125.00     .   yes


_diffrn_measured_fraction_theta_max    2.21
_diffrn_reflns_theta_full              28.28
_diffrn_measured_fraction_theta_full   1.876
_refine_diff_density_max    0.430
_refine_diff_density_min   -0.280


#===END
```