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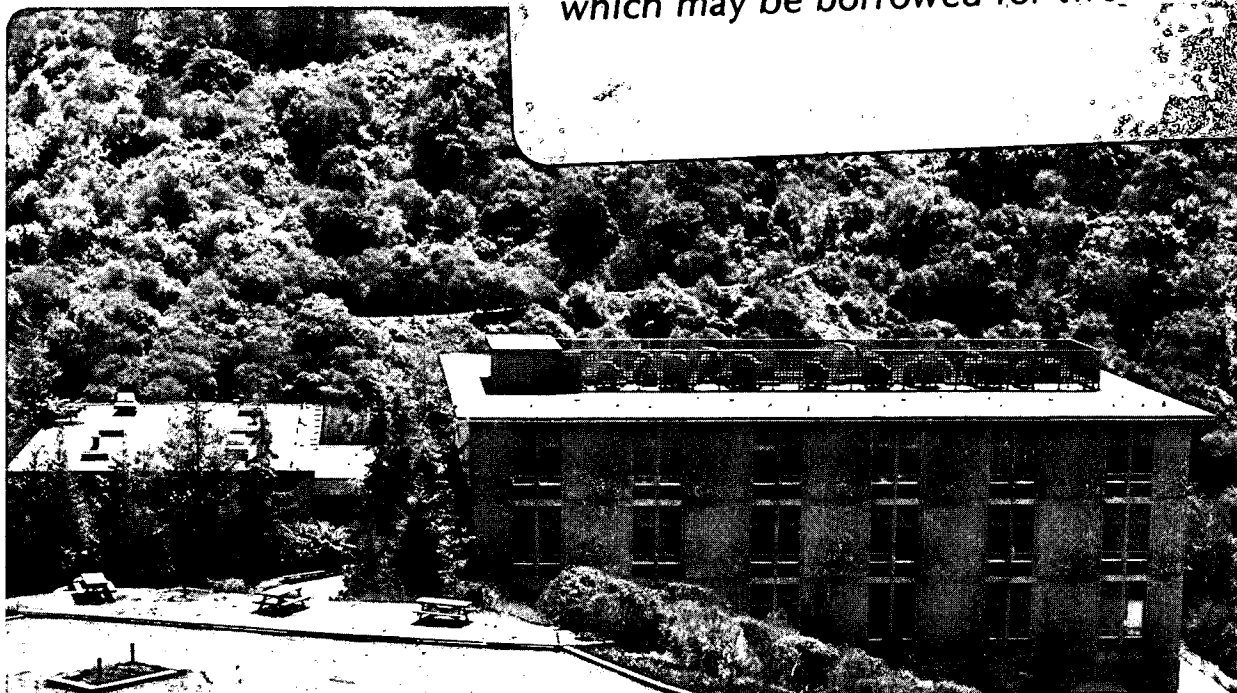
PREPARATION OF THE FIRST MOLECULAR η^2 -ACETYLENE
COMPLEX OF A 4f-TRANSITION METAL,
(Me₅C₅)₂Yb(η^2 -MeC≡CMe)

C.J. Burns and R.A. Andersen

September 1986

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LBL-22127

Preparation of the First Molecular η^2 -Acetylene Complex
of a 4f-Transition Metal, $(\text{Me}_5\text{C}_5)_2\text{Yb}(\eta^2\text{-MeC}\equiv\text{CMe})$

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Abstract

This communication describes the synthesis and crystal structure of the first η^2 -acetylene complex of an f-transition metal, $(\text{Me}_5\text{C}_5)_2\text{Yb}(\eta^2\text{-MeC}\equiv\text{CMe})$. The geometry of the acetylene is not greatly perturbed by coordination to the ytterbium(II) center and the complex can be viewed as a van der Waals molecule.

Most d-block transition metals form well-defined complexes with acetylenes.¹ Even Cu(I) and its congeners yield isolable complexes.² No η^2 -acetylene complexes of the f-block transition metals have been described though lanthanide metal atoms react with acetylenes^{3a,b} and a black substance of unknown structure whose composition is $[(\text{Me}_5\text{C}_5)_2\text{Sm}]_2[\text{PhC}\equiv\text{CPh}]$ has been isolated.^{3c} Monosubstituted acetylenes such as $\text{PhC}\equiv\text{CH}$ react with $(\text{Me}_5\text{C}_5)_2\text{Yb}(\text{OEt}_2)$ to give the mixed-valence phenylacetylide, $(\text{Me}_5\text{C}_5)_4\text{Yb}_3(\mu\text{-C}\equiv\text{CPh})_4$ presumably by way of prior acetylene coordination.^{3d} The bonding in metal-acetylene complexes is similar to that in olefin complexes, viz the filled orbitals of σ -symmetry act as donors and the empty π -symmetry orbitals on the acetylene act as acceptor orbitals.^{4a-c} The $(\eta^6\text{-Me}_6\text{C}_6)\text{Sm}(\text{AlCl}_4)_3$ has been isolated recently.^{4d} Because lanthanide metallocenes can act as Lewis acids though not as π -donors since the filled energy levels are too low in energy,⁵ a reasonable strategy for synthesis of acetylene complexes is to use donor rather than acceptor substituents on the acetylene. The first vertical ionization energy of $\text{MeC}\equiv\text{CMe}$ and tetrahydrofuran are 9.59 eV^{6a} and 9.57 eV,^{6b} respectively, so it is, perhaps, not unexpected that $\text{MeC}\equiv\text{CMe}$ will yield an isolable complex since the thf complex is known.⁷

Addition of 2-butyne to base-free $(\text{Me}_5\text{C}_5)_2\text{Yb}^5$ in pentane results in a color change from orange to red from which dark purple-red needles, mp 170-173 °C, may be isolated on cooling. The infrared spectrum of the complex as a Nujol mull shows no absorptions which may be attributed to $\nu\text{C}\equiv\text{C}$.⁸ The ^1H NMR spectrum in C_6D_6 at 30°C shows that the complex is diamagnetic and that the Me_5C_5 resonance in the complex is slightly deshielded relative to the base-free compound, δ 1.92 vs. 1.99, under identical conditions. The $^{13}\{^1\text{H}\}$ NMR chemical shifts of the Me_5C_5 rings in the complex are also slightly perturbed relative to the base-free complex; the ring carbons are unchanged at δ 113.4

and the methyl carbons are deshielded in the complex, δ 10.6 vs. 10.9. The greatest effect is on the resonances of the 2-butyne ligand. The free ligand at 30°C in C_6D_6 shows a resonance in the 1H NMR spectrum at δ 1.52 which shifts to δ 1.27 on coordination. In the $^{13}C\{^1H\}$ NMR spectrum the methyne and methyl carbons are deshielded on coordination, δ 74.6 to 76.9 and δ 3.08 to 3.76, respectively. The complexed 2-butyne is undergoing chemical exchange at 30°C and -75°C (200 MHz) since at 30°C the 1H NMR resonance of the acetylene in methylcyclohexane- d_{14} with a ten-fold molar excess of acetylene occurs as a sharp singlet at δ 1.63 (relative to the most intense resonance due to residual protons of the solvent at δ 0.82). Cooling to -75°C changes the averaged chemical shift to δ 1.73 with no discernable change in line shape. Both the vibrational spectrum of the solid and solution NMR spectra show that $MeC\equiv CMe$ is only weakly perturbed on coordination to the lanthanide center. This contention is supported by the crystal structure at -80°C.

An ORTEP diagram is shown in the Figure. The geometry of the Me_5C_5 -portion of the complex is not greatly perturbed from that of $(Me_5C_5)_2Yb(thf)$.⁷ The averaged Yb-C and Yb- Me_5C_5 ring centroid distances and the Me_5C_5 ring centroid-Yb- Me_5C_5 ring centroid angle in the acetylene complex are 2.659 ± 0.009 Å, 2.38 Å, and 143.3° , respectively. The equivalent distances and angle in the tetrahydrofuran complex are 2.663 ± 0.011 Å, 2.37 Å, and 143.5° , respectively. The C(1)C(2) and averaged C-C(Me) distances in the complex are 1.154(6) Å and 1.468 ± 0.005 Å, respectively. The equivalent distances in free 2-butyne (-50°C) are 1.21(2) Å and 1.46(1) Å, respectively.¹⁰ The averaged C-C-C(Me) angle in the complex is $177.4 \pm 0.7^\circ$ and the equivalent angle in the free ligand is linear. Comparison of the C-C distance and the C-C-C(Me) angle in $(Me_5C_5)_2Yb(\eta^2-MeC\equiv CMe)$ with the equivalent bond parameters in trans- $[MePt(PMe_2Ph)_2(\eta^2-MeC\equiv CMe)]PF_6$ ^{11a} and $(Ph_3P)_2Pt(\eta^2-$

$\text{MeC}\equiv\text{CPh}$ ^{11b} in which the C-C distance is 1.22(3) Å and 1.28(3) Å and the C-C-C(Me) angle is $168 \pm 2^\circ$ and $141.8(1.8)^\circ$, respectively, shows that little or no π -backbonding is involved in the ytterbium-acetylene bond. The averaged Yb-C(1,2) distance is 2.850 ± 0.010 Å and the C(1)-Yb-C(2) angle is $23.4(1)^\circ$. The dihedral angle formed by intersection of the plane defined by C(1,2,3,4) and Yb with the plane defined by the Me_5C_5 ring centroid-Yb- Me_5C_5 ring centroid is 91.5° . The Yb to acetylene carbon distance may be compared with the longer, asymmetric Lu-Me distance of 2.756(9) Å in the trivalent lutetium complex, $(\text{Me}_5\text{C}_5)_4\text{Lu}_2(\mu\text{-Me})(\text{Me})$, an interaction that is worth ca. 10 kcal mol^{-1} .^{12a} The Yb to acetylene carbon distance also may be compared to the Yb(II) to semi-bridging methyl distance of 3.04 Å in $\text{Yb}[\text{N}(\text{SiMe}_3)_2]_2(\text{dmpe})$,^{12b} in $\text{NaYb}[\text{N}(\text{SiMe}_3)_2]_3$ which range from 2.86 Å to 3.32 Å,^{12c} and in $\text{Yb}[\text{N}(\text{SiMe}_3)_2]_2[\text{AlMe}_3]_2$ which range from 2.767(4) Å to 2.95(17) Å.^{12d}

Clearly the 2-butyne to $(\text{Me}_5\text{C}_5)_2\text{Yb}$ interaction is weak as judged by spectroscopic and crystallographic data. The complex is best viewed as a Lewis acid-base adduct with little or no π -backbonding.

Acknowledgment. We thank the Fannie and John Hertz Foundation for a fellowship (C.J.B.) and Dr. F. J. Hollander for his help with the X-ray crystallography. This work was supported by the Director, Office of Research, Office of Basic Energy Sciences, Chemical Sciences Division of the U. S. Department of Energy under contract DE-AC03-76SF00098.

Supplementary Material. Atomic positional parameters and thermal parameters (5 pages).

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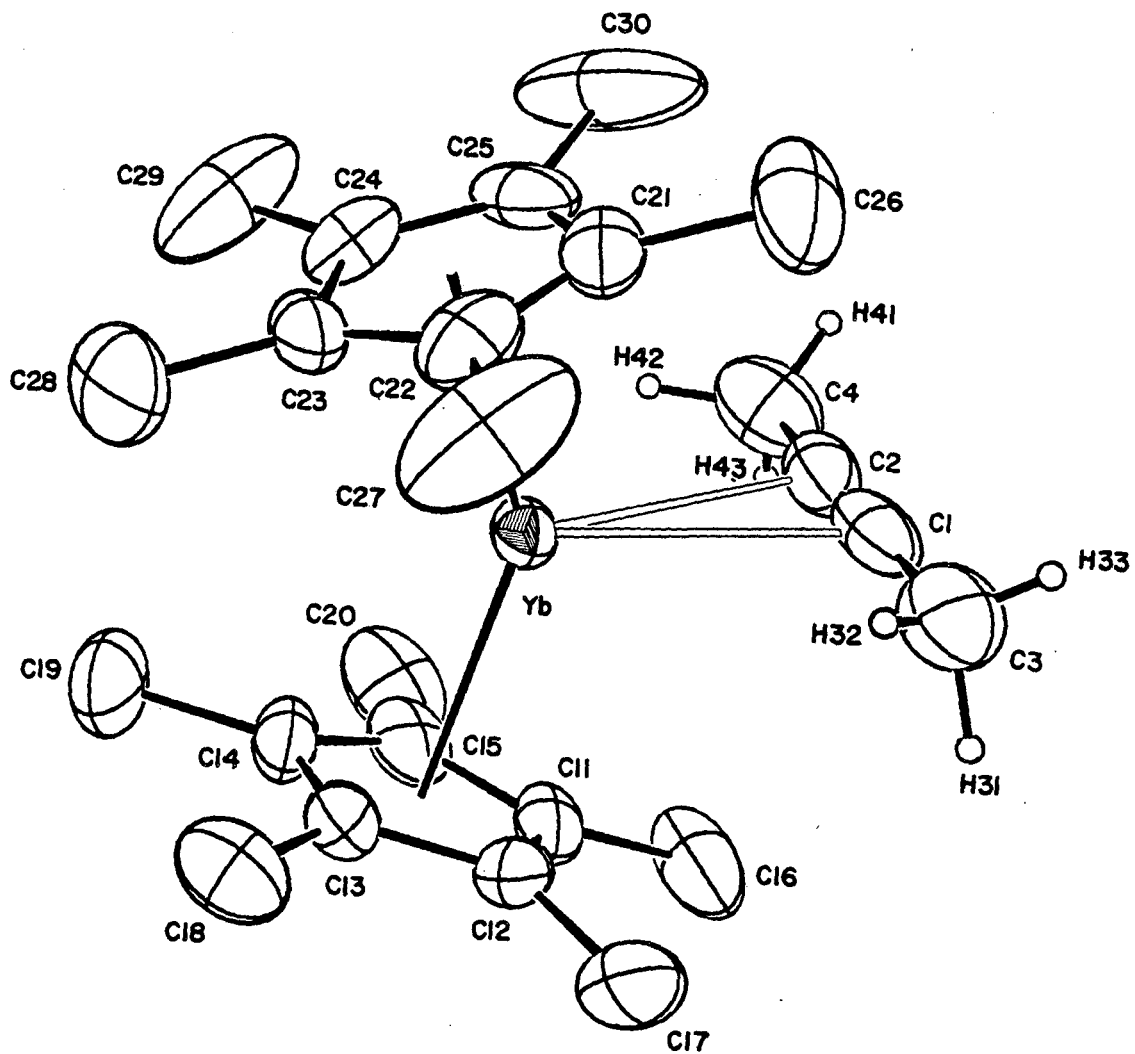
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8. We have isolated the unsymmetrical acetylene complexes, $(\text{Me}_5\text{C}_5)_2\text{Yb}(\text{L})$, $\text{L} = \text{PhC}\equiv\text{CMe}$ or $\text{Me}_3\text{CC}\equiv\text{CMe}$, though the $\nu\text{C}\equiv\text{C}$ is not observable in the infrared. We have not been able to observe the Raman spectrum of the $\text{MeC}\equiv\text{CMe}$ complex in the solid state nor in a methylcyclohexane solution. We thank Professor H.L. Struass and R.J. Sension for their help with these nonproductive experiments.
9. The compound crystallizes in the monoclinic crystal system in space group $\text{P}2_1/n$ with cell dimensions $a=15.738(2)\text{\AA}$, $b=15.299(2)\text{\AA}$, and $c=9.724(1)\text{\AA}$, $\beta=103.90(1)^\circ$, $V=2272.6(8)\text{\AA}^3$ with $Z=4$, and $d(\text{calc})=1.45\text{g cm}^{-3}$. The data were collected on a Nonius CAD4 automated diffractometer with MoK_α X-rays ($\lambda=0.71073\text{\AA}$) at -80°C . The structure was solved from Patterson and electron density maps and refined by full-matrix least squares to a conventional R-factor of 0.022 ($R_w=0.029$ and $\text{GOF}=1.505$) by using 2449 absorption corrected data, where $F_o^2 > 3\sigma(F_o^2)$ against 251 variables. The R-value for all 2971 unique data was 0.044. All non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms on the

2-butyne ligand were located in a difference map and they were refined with isotropic thermal parameters. All other hydrogen atoms were located in a difference Fourier map, then placed in calculated positions included in the structure factor calculations, but were not refined.

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Figure Caption: ORTEP Diagram of $(\text{Me}_5\text{C}_5)_2\text{Yb}(\eta^2\text{-MeC}\equiv\text{CMe})$. Thermal Ellipsoids represent 50% probability surfaces except those of the hydrogen atoms which are arbitrary.



XBL 868-2898

Supplementary Material for

Preparation of the First Molecular η^2 -Acetylene Complex of a 4f-Transition
Metal, $(\text{Me}_5\text{C}_5)_2\text{Yb}(\eta^2\text{-MeC}\equiv\text{CMe})$.

Carol J. Burns and Richard A. Andersen

Positional and Thermal Parameters (5 pages)

Table of Positional Parameters and Their Estimated Standard Deviations

Atom	x	y	z	B(A ²)
Yb	0.37121(1)	0.23167(1)	0.16790(2)	2.495(4)
C1	0.3473(3)	0.1184(3)	0.3854(5)	4.1(1)
C2	0.2794(3)	0.1118(4)	0.3078(5)	4.1(1)
C3	0.4335(4)	0.1285(4)	0.4858(7)	5.7(2)
C4	0.1916(4)	0.1002(4)	0.2180(7)	6.0(2)
C11	0.3091(3)	0.3492(3)	0.3225(5)	4.2(1)
C12	0.3977(4)	0.3697(3)	0.3412(5)	4.1(1)
C13	0.4108(3)	0.4012(3)	0.2115(5)	3.5(1)
C14	0.3292(3)	0.3985(3)	0.1119(5)	3.3(1)
C15	0.2663(3)	0.3662(4)	0.1777(5)	4.0(1)
C16	0.2636(5)	0.3206(4)	0.4359(6)	8.0(2)
C17	0.4667(6)	0.3686(5)	0.4799(7)	7.8(2)
C18	0.4941(4)	0.4409(4)	0.1908(7)	5.3(2)
C19	0.3094(4)	0.4357(4)	-0.0385(6)	5.4(2)
C20	0.1696(4)	0.3577(5)	0.1106(8)	6.5(2)
C21	0.4588(4)	0.0957(4)	0.1014(5)	4.8(1)
C22	0.5057(3)	0.1687(4)	0.0810(5)	4.1(1)
C23	0.4564(3)	0.2154(3)	-0.0353(5)	3.6(1)
C24	0.3774(3)	0.1696(4)	-0.0840(5)	4.0(1)
C25	0.3782(3)	0.0966(4)	0.0002(6)	4.8(1)
C26	0.4892(5)	0.0204(5)	0.2020(7)	10.1(2)

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	B(A ²)
C27	0.5965(4)	0.1941(7)	0.1631(7)	8.3(2)
C28	0.4890(5)	0.2901(5)	-0.1091(7)	7.6(2)
C29	0.3063(5)	0.1948(7)	-0.2153(7)	8.7(2)
C30	0.3114(5)	0.0246(5)	-0.0244(8)	12.2(2)
H31	0.437(4)	0.174(5)	0.564(7)	7(2)*
H32	0.482(4)	0.151(5)	0.453(7)	8(2)*
H33	0.439(5)	0.081(6)	0.526(8)	10(3)*
H41	0.167(3)	0.041(3)	0.207(5)	3(1)*
H42	0.151(4)	0.132(5)	0.248(6)	6(2)*
H43	0.180(3)	0.133(4)	0.121(6)	4(1)*
H161	0.2461	0.3709	0.4797	9*
H162	0.2137	0.2866	0.3942	9*
H163	0.3029	0.2868	0.5051	9*
H171	0.4663	0.4230	0.5266	9*
H172	0.4540	0.3229	0.5381	9*
H173	0.5225	0.3590	0.4616	9*
H181	0.4949	0.5015	0.2120	6*
H182	0.5427	0.4131	0.2521	6*
H183	0.4972	0.4330	0.0953	6*
H191	0.2958	0.4960	-0.0360	6*
H192	0.3590	0.4284	-0.0767	6*

Table of Positional Parameters and Their Estimated Standard Deviations (cont.)

Atom	x	y	z	B(A2)
H193	0.2608	0.4055	-0.0960	6*
H201	0.1408	0.4106	0.1226	7*
H202	0.1612	0.3455	0.0124	7*
H203	0.1460	0.3112	0.1547	7*
H261	0.5192	-0.0211	0.1584	12*
H262	0.5279	0.0417	0.2861	12*
H263	0.4403	-0.0065	0.2254	12*
H271	0.6384	0.1671	0.1211	9*
H272	0.6026	0.2555	0.1612	9*
H273	0.6050	0.1747	0.2583	9*
H281	0.5173	0.2675	-0.1776	9*
H282	0.4410	0.3255	-0.1547	9*
H283	0.5293	0.3239	-0.0416	9*
H291	0.3179	0.1671	-0.2964	10*
H292	0.2509	0.1760	-0.2035	10*
H293	0.3059	0.2562	-0.2273	10*
H301	0.3255	-0.0170	-0.0878	14*
H302	0.3109	-0.0025	0.0627	14*
H303	0.2551	0.0486	-0.0651	14*

Starred atoms were refined isotropically.
 Anisotropically refined atoms are given in the form of the
 isotropic equivalent displacement parameter defined as:
 $(4/3) * [a^2*B(1,1) + b^2*B(2,2) + c^2*B(3,3) + ab(\cos \gamma)*B(1,2) + ac(\cos \beta)*B(1,3) + bc(\cos \alpha)*B(2,3)]$

Table of General Displacement Parameter Expressions - B's

Name	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	Beqv
Yb	3.094(8)	2.117(9)	2.594(7)	0.260(7)	1.312(6)	0.253(7)	2.495(4)
C1	5.2(2)	2.3(2)	5.8(2)	0.5(2)	3.2(2)	0.3(2)	4.1(1)
C2	5.4(2)	3.0(2)	4.4(2)	0.8(2)	2.2(2)	0.6(2)	4.1(1)
C3	6.3(3)	4.7(3)	5.9(3)	1.1(3)	0.7(3)	0.2(3)	5.7(2)
C4	4.9(3)	4.6(3)	8.6(4)	-0.5(2)	2.2(3)	-0.1(3)	6.0(2)
C11	7.7(3)	2.2(2)	3.9(2)	1.6(2)	3.5(2)	0.5(2)	4.2(1)
C12	6.9(3)	2.5(2)	2.5(2)	0.5(2)	0.6(2)	-0.2(2)	4.1(1)
C13	5.1(2)	2.4(2)	3.2(2)	0.1(2)	1.4(2)	0.2(2)	3.5(1)
C14	4.4(2)	3.1(2)	2.9(2)	0.7(2)	1.7(2)	0.7(2)	3.3(1)
C15	4.2(2)	2.8(2)	5.4(2)	1.5(2)	1.9(2)	0.6(2)	4.0(1)
C16	14.6(3)	3.6(3)	9.0(3)	1.8(3)	9.3(2)	1.0(2)	8.0(2)
C17	13.3(5)	4.2(3)	4.1(3)	-0.3(4)	-1.4(3)	-0.6(3)	7.8(2)
C18	5.2(3)	4.1(3)	6.5(3)	-1.2(2)	1.4(2)	-0.2(3)	5.3(2)
C19	7.2(3)	4.9(3)	4.0(2)	1.5(3)	1.4(2)	1.7(2)	5.4(2)
C20	5.1(3)	4.9(3)	10.0(4)	1.3(3)	3.0(2)	1.3(3)	6.5(2)
C21	8.0(3)	3.6(2)	4.2(2)	2.8(2)	3.8(2)	0.8(2)	4.8(1)
C22	3.5(2)	5.5(3)	3.4(2)	1.0(2)	1.4(2)	-0.4(2)	4.1(1)
C23	5.3(2)	2.4(2)	3.9(2)	0.3(2)	2.6(2)	0.2(2)	3.6(1)
C24	3.7(2)	5.7(3)	2.5(2)	0.2(2)	0.8(2)	-0.7(2)	4.0(1)
C25	6.9(2)	3.1(2)	6.1(2)	-1.6(2)	4.8(2)	-1.9(2)	4.8(1)
C26	19.2(5)	6.2(3)	8.1(3)	7.7(3)	9.2(2)	3.8(3)	10.1(2)

Table of General Displacement Parameter Expressions -- B's (Continued)

Name	B(1,1)	B(2,2)	B(3,3)	B(1,2)	B(1,3)	B(2,3)	Beqv
C27	3.7(2)	15.5(6)	5.8(3)	1.7(4)	1.2(2)	-2.2(4)	8.3(2)
C28	11.9(3)	5.2(4)	8.5(3)	-0.9(3)	8.0(2)	0.4(3)	7.6(2)
C29	7.4(4)	14.8(6)	3.6(3)	2.9(4)	0.6(3)	-1.4(4)	8.7(2)
C30	16.0(4)	9.2(4)	15.7(4)	-8.5(3)	12.1(3)	-8.9(3)	12.2(2)

The form of the anisotropic displacement parameter is:
 $\exp[-0.25(h^2a^2B(1,1) + k^2b^2B(2,2) + l^2c^2B(3,3) + 2hkabB(1,2) + 2hlacB(1,3) + 2klbcB(2,3))]$ where a, b, and c are reciprocal lattice constants.

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