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J. Am. Chem. Soc., 1998, 120(25), 6287-6305, DOI:10.1021/ja973769t

where: No = number of observations

Nv = 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) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{60}H_{15}F_{28}Al$
Formula Weight	1294.72
Crystal Color, Habit	yellow, platy
Crystal Dimensions	0.40 X 0.20 X 0.08 mm
Crystal System	triclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25 (17.1 - 23.1°)
Omega Scan Peak Width at Half-height	0.30°
Lattice Parameters	a = 12.179(5)Å b = 12.473(5) Å c = 18.334(5) Å α = 99.21(3)° β = 94.88(3)° γ = 108.82(3)° V = 2574(1) Å^3
Space Group	$P\bar{1}$ (#2)
Z value	2
D_{calc}	1.670 g/cm^3
F_{000}	1280.00
μ(MoKα)	1.84 cm^{-1}

B. Intensity Measurements

Diffractometer	CAD4
Radiation	MoKα ($\lambda = 0.71069$ Å) graphite monochromated
Attenuator	Zr foil (factor = 22.25)
Take-off Angle	2.8°
Detector Aperture	2.0 - 2.5 mm horizontal 2.0 mm vertical
Crystal to Detector Distance	21 mm
Temperature	-120.0°C
Scan Type	ω-θ
Scan Rate	3.0°/min (in ω) (up to 0 scans)
Scan Width	$(1.00 + 0.35 \tan\theta)°$
$2\theta_{max}$	47.9°
No. of Reflections Measured	Total: 8343 Unique: 8057 ($R_{int} = 0.088$)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9604 - 0.9834)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SHELXS86)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$\frac{1}{\sigma^2(Fo)} = \frac{4Fo^2}{\sigma^2(Fo^2)}$
p-factor	0.0000
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>3.00σ(I))	3109
No. Variables	514
Reflection/Parameter Ratio	6.05

Residuals: R; Rw	0.072 ; 0.053
Goodness of Fit Indicator	2.25
Max Shift/Error in Final Cycle	0.16
Maximum peak in Final Diff. Map	0.72 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.35 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}
Al	0.6348(3)	0.2809(3)	0.7371(2)	2.96(9)
F(1)	0.6192(5)	0.0364(5)	0.7792(3)	5.2(2)
F(2)	0.7267(6)	-0.0173(6)	0.8932(3)	6.2(2)
F(3)	0.8674(5)	0.1528(6)	1.0075(3)	6.9(2)
F(4)	0.9033(6)	0.3764(6)	1.0028(3)	7.5(2)
F(5)	0.9844(5)	0.4613(5)	0.8432(3)	5.8(2)
F(6)	1.0325(6)	0.6840(6)	0.8348(3)	7.3(2)
F(7)	0.8757(6)	0.7902(5)	0.8697(3)	7.0(2)
F(8)	0.6734(6)	0.6725(5)	0.9154(3)	7.1(2)
F(9)	0.6295(5)	0.4507(5)	0.9284(3)	4.9(2)
F(10)	0.8512(5)	0.1979(5)	0.7080(3)	5.6(2)
F(11)	0.9941(5)	0.2270(5)	0.6052(3)	5.8(2)
F(12)	0.9739(5)	0.3575(6)	0.5022(4)	6.5(2)
F(13)	0.8104(5)	0.4634(5)	0.5077(3)	5.8(2)
F(14)	0.7686(5)	0.5983(5)	0.7087(3)	4.9(2)
F(15)	0.5914(6)	0.6835(5)	0.7172(3)	6.1(2)
F(16)	0.3889(5)	0.5793(5)	0.6200(3)	5.6(2)
F(17)	0.3619(5)	0.3880(5)	0.5179(3)	4.5(2)
F(18)	0.5332(5)	0.2961(5)	0.5144(3)	3.8(2)
F(19)	0.6014(5)	0.0595(5)	0.6232(3)	4.2(2)
F(20)	0.4170(5)	-0.0976(5)	0.5329(3)	4.9(2)
F(21)	0.2002(5)	-0.0813(5)	0.5348(3)	4.7(2)
F(22)	0.1675(4)	0.0858(4)	0.6326(3)	3.5(2)
F(23)	0.2577(5)	0.3195(5)	0.6386(3)	4.1(2)

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

atom	x	y	z	B_{eq}
F(24)	0.2040(6)	0.4702(5)	0.7370(4)	7.5(2)
F(25)	0.2640(6)	0.4890(6)	0.8877(4)	9.5(3)
F(26)	0.3781(5)	0.3526(6)	0.9344(3)	6.6(2)
F(27)	0.4288(5)	0.1998(5)	0.8360(3)	4.5(2)
F(28)	0.5986(4)	0.3983(4)	0.7660(3)	3.0(1)
C(1)	0.7048(8)	0.2375(8)	0.8275(5)	3.0(2)
C(2)	0.6879(9)	0.1264(9)	0.8336(6)	3.5(2)
C(3)	0.7431(10)	0.094(1)	0.8926(6)	4.3(3)
C(4)	0.8139(9)	0.1813(10)	0.9496(6)	4.3(3)
C(5)	0.8312(10)	0.2944(10)	0.9461(6)	4.2(3)
C(6)	0.7791(9)	0.3253(9)	0.8861(5)	3.2(2)
C(7)	0.8033(9)	0.4487(9)	0.8862(5)	3.3(2)
C(8)	0.907(1)	0.5122(10)	0.8623(6)	3.8(3)
C(9)	0.932(1)	0.627(1)	0.8572(6)	4.5(3)
C(10)	0.854(1)	0.678(1)	0.8760(6)	4.2(3)
C(11)	0.750(1)	0.621(1)	0.8999(6)	4.3(3)
C(12)	0.7306(10)	0.5061(9)	0.9051(6)	3.6(2)
C(13)	0.7535(8)	0.3185(8)	0.6662(5)	2.8(2)
C(14)	0.8385(9)	0.2678(9)	0.6599(6)	3.9(3)
C(15)	0.9149(10)	0.2801(9)	0.6073(6)	4.0(3)
C(16)	0.904(1)	0.3488(9)	0.5566(6)	4.4(3)
C(17)	0.8203(10)	0.3992(9)	0.5591(6)	3.9(2)
C(18)	0.7463(8)	0.3883(8)	0.6139(5)	2.8(2)
C(19)	0.6557(8)	0.4431(8)	0.6121(5)	2.5(2)

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

atom	x	y	z	B_{eq}
C(20)	0.6685(9)	0.5439(9)	0.6613(6)	3.2(2)
C(21)	0.5800(9)	0.5889(9)	0.6657(6)	3.4(2)
C(22)	0.4790(10)	0.5367(9)	0.6185(6)	3.7(2)
C(23)	0.4625(9)	0.4396(9)	0.5669(6)	3.1(2)
C(24)	0.5502(8)	0.3926(8)	0.5641(5)	2.6(2)
C(25)	0.4871(8)	0.1616(8)	0.6786(5)	2.7(2)
C(26)	0.4952(9)	0.0717(8)	0.6271(5)	3.1(2)
C(27)	0.4020(9)	-0.0105(9)	0.5795(5)	2.9(2)
C(28)	0.2926(9)	-0.0026(9)	0.5803(5)	3.1(2)
C(29)	0.2791(8)	0.0846(8)	0.6304(5)	2.5(2)
C(30)	0.3723(8)	0.1653(8)	0.6788(5)	2.4(2)
C(31)	0.3450(8)	0.2534(8)	0.7335(5)	2.6(2)
C(32)	0.2895(9)	0.3254(8)	0.7114(6)	3.2(2)
C(33)	0.2618(10)	0.4042(10)	0.7626(6)	4.5(3)
C(34)	0.292(1)	0.416(1)	0.8367(7)	4.7(3)
C(35)	0.3477(9)	0.3476(9)	0.8608(6)	4.2(3)
C(36)	0.3763(9)	0.2687(9)	0.8108(6)	3.3(2)
C(37)	1.0378(8)	0.9241(8)	0.7347(5)	2.8(2)
C(38)	1.0576(8)	1.0137(8)	0.7995(5)	2.5(2)
C(39)	1.1069(9)	1.0083(9)	0.8707(6)	3.6(2)
C(40)	1.1225(9)	1.0966(9)	0.9306(6)	4.1(3)
C(41)	1.0934(9)	1.1908(9)	0.9214(6)	4.6(3)
C(42)	1.0476(9)	1.2001(9)	0.8520(6)	3.7(2)
C(43)	1.0299(8)	1.1136(8)	0.7916(5)	2.9(2)

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

atom	x	y	z	B_{eq}
C(44)	0.9358(8)	0.8968(8)	0.6797(5)	2.4(2)
C(45)	0.9392(8)	0.8619(8)	0.6035(5)	2.8(2)
C(46)	0.8429(9)	0.8457(8)	0.5521(6)	3.8(2)
C(47)	0.7412(9)	0.8592(9)	0.5750(6)	4.3(3)
C(48)	0.7366(9)	0.8908(9)	0.6498(6)	4.5(3)
C(49)	0.8318(9)	0.9092(8)	0.7015(5)	3.4(2)
C(50)	1.1209(9)	0.8646(8)	0.7252(5)	3.1(2)
C(51)	1.2401(9)	0.9223(8)	0.7576(5)	3.5(2)
C(52)	1.3228(10)	0.866(1)	0.7478(6)	5.3(3)
C(53)	1.283(1)	0.756(1)	0.7069(7)	5.9(3)
C(54)	1.171(1)	0.697(1)	0.6754(6)	6.0(3)
C(55)	1.0871(9)	0.7511(10)	0.6836(6)	4.6(3)
C(56)	0.573(1)	-0.061(1)	0.0385(7)	5.3(3)
C(57)	0.545(3)	-0.145(3)	-0.004(2)	9.3(10)
C(58)	0.434(3)	-0.238(3)	-0.079(2)	9(1)
C(59)	0.390(1)	-0.210(1)	-0.0817(8)	5.0(4)
C(60)	0.308(2)	-0.252(2)	-0.086(1)	3.4(5)
C(61)	0.472(2)	-0.287(2)	-0.140(2)	8.0(8)
C(62)	0.447(3)	-0.070(3)	-0.043(2)	14(1)
C(63)	0.470(2)	-0.213(2)	-0.010(2)	5.8(7)
H(1)	1.129	0.944	0.877	4.310
H(2)	1.154	1.092	0.979	4.894
H(3)	1.105	1.251	0.963	5.475
H(4)	1.028	1.267	0.846	4.389

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

atom	x	y	z	B_{eq}
H(5)	0.999	1.120	0.744	3.527
H(6)	1.007	0.850	0.588	3.381
H(7)	0.846	0.825	0.500	4.600
H(8)	0.675	0.847	0.539	5.112
H(9)	0.667	0.900	0.665	5.390
H(10)	0.828	0.931	0.753	4.042
H(11)	1.264	0.999	0.786	4.148
H(12)	1.403	0.904	0.769	6.387
H(13)	1.339	0.718	0.700	7.131
H(14)	1.149	0.620	0.648	7.216
H(15)	1.008	0.712	0.661	5.543

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Al	0.039(2)	0.049(2)	0.027(2)	0.016(2)	0.001(2)	0.014(2)
F(1)	0.094(5)	0.052(4)	0.055(4)	0.028(4)	0.000(4)	0.013(3)
F(2)	0.111(6)	0.079(5)	0.075(5)	0.053(5)	0.022(4)	0.047(4)
F(3)	0.076(5)	0.122(6)	0.071(5)	0.026(5)	-0.011(4)	0.070(5)
F(4)	0.093(6)	0.093(6)	0.055(5)	-0.023(5)	-0.042(4)	0.038(4)
F(5)	0.046(4)	0.093(5)	0.076(5)	0.004(4)	0.004(4)	0.045(4)
F(6)	0.075(5)	0.102(6)	0.077(5)	-0.013(5)	0.005(4)	0.048(4)
F(7)	0.129(7)	0.052(5)	0.063(5)	0.002(4)	0.006(4)	0.015(4)
F(8)	0.128(7)	0.071(5)	0.070(5)	0.034(5)	0.026(5)	0.004(4)
F(9)	0.058(5)	0.073(5)	0.045(4)	0.006(4)	0.014(3)	0.012(4)
F(10)	0.064(5)	0.109(6)	0.072(5)	0.058(4)	0.018(4)	0.049(4)
F(11)	0.052(4)	0.104(6)	0.086(5)	0.051(4)	0.014(4)	0.021(4)
F(12)	0.069(5)	0.104(6)	0.088(5)	0.033(4)	0.049(4)	0.035(5)
F(13)	0.075(5)	0.094(5)	0.079(5)	0.039(4)	0.042(4)	0.058(4)
F(14)	0.071(5)	0.058(4)	0.041(4)	0.006(4)	-0.003(4)	0.008(3)
F(15)	0.124(6)	0.044(4)	0.063(5)	0.026(4)	0.033(4)	0.004(4)
F(16)	0.079(5)	0.079(5)	0.077(5)	0.046(4)	0.032(4)	0.029(4)
F(17)	0.046(4)	0.063(4)	0.065(4)	0.022(4)	0.003(3)	0.020(3)
F(18)	0.044(4)	0.045(4)	0.049(4)	0.016(3)	-0.003(3)	-0.003(3)
F(19)	0.054(4)	0.061(4)	0.052(4)	0.035(4)	0.005(3)	0.009(3)
F(20)	0.079(5)	0.055(4)	0.053(4)	0.028(4)	0.016(4)	-0.001(3)
F(21)	0.057(4)	0.054(4)	0.044(4)	-0.004(4)	0.000(3)	-0.005(3)
F(22)	0.038(4)	0.044(4)	0.048(4)	0.010(3)	0.005(3)	0.011(3)
F(23)	0.043(4)	0.063(4)	0.059(4)	0.019(3)	0.012(3)	0.032(3)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(24)	0.101(6)	0.068(5)	0.142(7)	0.054(5)	0.042(5)	0.031(5)
F(25)	0.119(7)	0.105(6)	0.122(7)	0.036(5)	0.054(6)	-0.030(5)
F(26)	0.072(5)	0.111(6)	0.039(4)	0.008(5)	0.013(4)	-0.015(4)
F(27)	0.063(4)	0.080(5)	0.026(4)	0.024(4)	0.001(3)	0.008(3)
F(28)	0.045(4)	0.036(4)	0.034(3)	0.012(3)	0.005(3)	0.010(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 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
Al	F(28)	1.682(5)	Al	C(1)	2.027(9)
Al	C(13)	2.019(10)	Al	C(25)	2.009(9)
F(1)	C(2)	1.36(1)	F(2)	C(3)	1.34(1)
F(3)	C(4)	1.35(1)	F(4)	C(5)	1.35(1)
F(5)	C(8)	1.34(1)	F(6)	C(9)	1.34(1)
F(7)	C(10)	1.37(1)	F(8)	C(11)	1.32(1)
F(9)	C(12)	1.35(1)	F(10)	C(14)	1.37(1)
F(11)	C(15)	1.34(1)	F(12)	C(16)	1.36(1)
F(13)	C(17)	1.35(1)	F(14)	C(20)	1.34(1)
F(15)	C(21)	1.35(1)	F(16)	C(22)	1.36(1)
F(17)	C(23)	1.36(1)	F(18)	C(24)	1.332(9)
F(19)	C(26)	1.36(1)	F(20)	C(27)	1.34(1)
F(21)	C(28)	1.34(1)	F(22)	C(29)	1.368(10)
F(23)	C(32)	1.34(1)	F(24)	C(33)	1.35(1)
F(25)	C(34)	1.35(1)	F(26)	C(35)	1.35(1)
F(27)	C(36)	1.34(1)	C(1)	C(2)	1.36(1)
C(1)	C(6)	1.41(1)	C(2)	C(3)	1.40(1)
C(3)	C(4)	1.38(1)	C(4)	C(5)	1.37(1)
C(5)	C(6)	1.39(1)	C(6)	C(7)	1.47(1)
C(7)	C(8)	1.40(1)	C(7)	C(12)	1.34(1)
C(8)	C(9)	1.38(1)	C(9)	C(10)	1.34(1)
C(10)	C(11)	1.38(1)	C(11)	C(12)	1.39(1)
C(13)	C(14)	1.38(1)	C(13)	C(18)	1.41(1)
C(14)	C(15)	1.39(1)	C(15)	C(16)	1.39(1)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(16)	C(17)	1.36(1)	C(17)	C(18)	1.40(1)
C(18)	C(19)	1.47(1)	C(19)	C(20)	1.38(1)
C(19)	C(24)	1.39(1)	C(20)	C(21)	1.37(1)
C(21)	C(22)	1.35(1)	C(22)	C(23)	1.36(1)
C(23)	C(24)	1.38(1)	C(25)	C(26)	1.38(1)
C(25)	C(30)	1.41(1)	C(26)	C(27)	1.38(1)
C(27)	C(28)	1.37(1)	C(28)	C(29)	1.37(1)
C(29)	C(30)	1.38(1)	C(30)	C(31)	1.50(1)
C(31)	C(32)	1.38(1)	C(31)	C(36)	1.40(1)
C(32)	C(33)	1.39(1)	C(33)	C(34)	1.35(1)
C(34)	C(35)	1.35(1)	C(35)	C(36)	1.39(1)
C(37)	C(38)	1.44(1)	C(37)	C(44)	1.44(1)
C(37)	C(50)	1.44(1)	C(38)	C(39)	1.41(1)
C(38)	C(43)	1.42(1)	C(39)	C(40)	1.38(1)
C(40)	C(41)	1.36(1)	C(41)	C(42)	1.38(1)
C(42)	C(43)	1.37(1)	C(44)	C(45)	1.40(1)
C(44)	C(49)	1.41(1)	C(45)	C(46)	1.38(1)
C(46)	C(47)	1.39(1)	C(47)	C(48)	1.38(1)
C(48)	C(49)	1.37(1)	C(50)	C(51)	1.42(1)
C(50)	C(55)	1.41(1)	C(51)	C(52)	1.41(1)
C(52)	C(53)	1.37(1)	C(53)	C(54)	1.36(1)
C(54)	C(55)	1.40(1)	C(56)	C(57)	1.13(3)
C(56)	C(62)	1.72(4)	C(57)	C(58)	1.81(5)
C(57)	C(63)	1.01(4)	C(58)	C(59)	0.72(4)

Table 3. Bond Lengths($\AA$) (continued)

atom	atom	distance	atom	atom	distance
C(58)	C(60)	1.48(4)	C(58)	C(61)	1.37(4)
C(58)	C(63)	1.26(4)	C(59)	C(60)	0.96(2)
C(59)	C(62)	1.67(4)	C(59)	C(63)	1.58(3)

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
C(39)	H(1)	0.95	C(40)	H(2)	0.95
C(41)	H(3)	0.95	C(42)	H(4)	0.95
C(43)	H(5)	0.95	C(45)	H(6)	0.95
C(46)	H(7)	0.95	C(47)	H(8)	0.95
C(48)	H(9)	0.95	C(49)	H(10)	0.95
C(51)	H(11)	0.95	C(52)	H(12)	0.95
C(53)	H(13)	0.95	C(54)	H(14)	0.95
C(55)	H(15)	0.95			

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
F(28)	Al	C(1)	108.6(3)	F(28)	Al	C(13)	109.0(3)
F(28)	Al	C(25)	105.4(3)	C(1)	Al	C(13)	110.0(4)
C(1)	Al	C(25)	115.9(4)	C(13)	Al	C(25)	107.7(4)
Al	C(1)	C(2)	123.1(8)	Al	C(1)	C(6)	119.7(7)
C(2)	C(1)	C(6)	117.2(9)	F(1)	C(2)	C(1)	121.2(9)
F(1)	C(2)	C(3)	114.5(10)	C(1)	C(2)	C(3)	124(1)
F(2)	C(3)	C(2)	121(1)	F(2)	C(3)	C(4)	121(1)
C(2)	C(3)	C(4)	117(1)	F(3)	C(4)	C(3)	118(1)
F(3)	C(4)	C(5)	121(1)	C(3)	C(4)	C(5)	119(1)
F(4)	C(5)	C(4)	117(1)	F(4)	C(5)	C(6)	120.5(10)
C(4)	C(5)	C(6)	122(1)	C(1)	C(6)	C(5)	119.1(10)
C(1)	C(6)	C(7)	122.2(9)	C(5)	C(6)	C(7)	118.7(9)
C(6)	C(7)	C(8)	119(1)	C(6)	C(7)	C(12)	124(1)
C(8)	C(7)	C(12)	116(1)	F(5)	C(8)	C(7)	119(1)
F(5)	C(8)	C(9)	118(1)	C(7)	C(8)	C(9)	121(1)
F(6)	C(9)	C(8)	119(1)	F(6)	C(9)	C(10)	121(1)
C(8)	C(9)	C(10)	118(1)	F(7)	C(10)	C(9)	119(1)
F(7)	C(10)	C(11)	117(1)	C(9)	C(10)	C(11)	122(1)
F(8)	C(11)	C(10)	121(1)	F(8)	C(11)	C(12)	122(1)
C(10)	C(11)	C(12)	116(1)	F(9)	C(12)	C(7)	119(1)
F(9)	C(12)	C(11)	116(1)	C(7)	C(12)	C(11)	124(1)
Al	C(13)	C(14)	122.5(8)	Al	C(13)	C(18)	121.7(7)
C(14)	C(13)	C(18)	115.4(9)	F(10)	C(14)	C(13)	119.5(10)
F(10)	C(14)	C(15)	114.9(10)	C(13)	C(14)	C(15)	125(1)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
F(11)	C(15)	C(14)	121(1)	F(11)	C(15)	C(16)	121(1)
C(14)	C(15)	C(16)	117(1)	F(12)	C(16)	C(15)	118(1)
F(12)	C(16)	C(17)	121(1)	C(15)	C(16)	C(17)	119(1)
F(13)	C(17)	C(16)	118(1)	F(13)	C(17)	C(18)	119.4(10)
C(16)	C(17)	C(18)	122(1)	C(13)	C(18)	C(17)	119.8(9)
C(13)	C(18)	C(19)	121.0(9)	C(17)	C(18)	C(19)	119.0(9)
C(18)	C(19)	C(20)	121.5(9)	C(18)	C(19)	C(24)	121.9(9)
C(20)	C(19)	C(24)	116.5(9)	F(14)	C(20)	C(19)	119.8(9)
F(14)	C(20)	C(21)	118.0(10)	C(19)	C(20)	C(21)	122(1)
F(15)	C(21)	C(20)	121.3(10)	F(15)	C(21)	C(22)	119(1)
C(20)	C(21)	C(22)	119(1)	F(16)	C(22)	C(21)	121(1)
F(16)	C(22)	C(23)	117.2(10)	C(21)	C(22)	C(23)	121(1)
F(17)	C(23)	C(22)	121.7(10)	F(17)	C(23)	C(24)	119.1(9)
C(22)	C(23)	C(24)	119(1)	F(18)	C(24)	C(19)	119.3(9)
F(18)	C(24)	C(23)	119.2(9)	C(19)	C(24)	C(23)	121.5(10)
Al	C(25)	C(26)	119.0(7)	Al	C(25)	C(30)	126.3(7)
C(26)	C(25)	C(30)	114.4(9)	F(19)	C(26)	C(25)	119.0(9)
F(19)	C(26)	C(27)	116.1(9)	C(25)	C(26)	C(27)	124(1)
F(20)	C(27)	C(26)	121.0(9)	F(20)	C(27)	C(28)	120.0(9)
C(26)	C(27)	C(28)	119(1)	F(21)	C(28)	C(27)	120.4(10)
F(21)	C(28)	C(29)	120.8(9)	C(27)	C(28)	C(29)	118(1)
F(22)	C(29)	C(28)	117.1(9)	F(22)	C(29)	C(30)	120.8(9)
C(28)	C(29)	C(30)	122.0(10)	C(25)	C(30)	C(29)	121.0(9)
C(25)	C(30)	C(31)	122.1(8)	C(29)	C(30)	C(31)	116.9(8)

Table 5. Bond Angles(°) (continued)

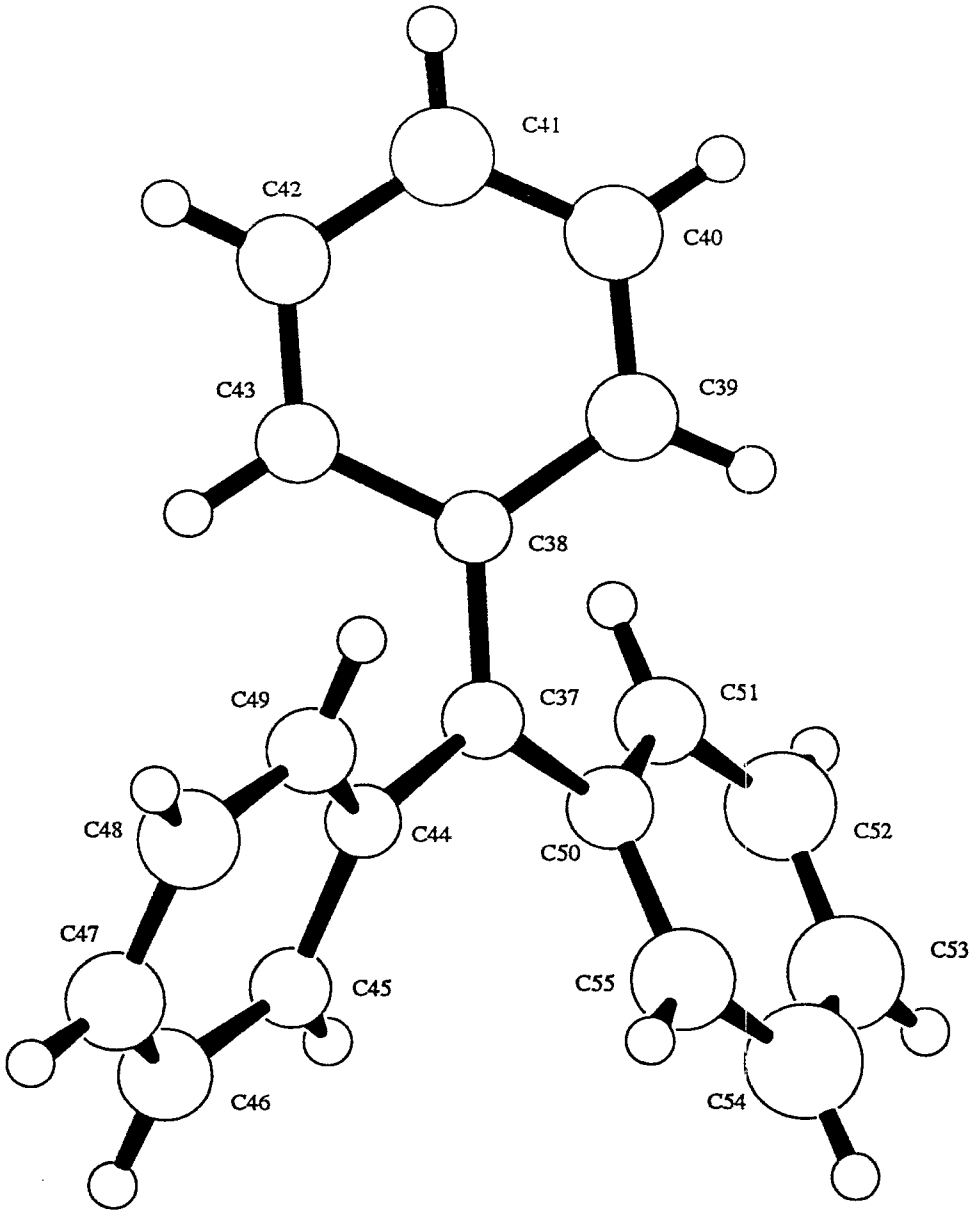
atom	atom	atom	angle	atom	atom	atom	angle
C(30)	C(31)	C(32)	122.6(9)	C(30)	C(31)	C(36)	122.0(9)
C(32)	C(31)	C(36)	115.4(9)	F(23)	C(32)	C(31)	120.2(9)
F(23)	C(32)	C(33)	117.7(9)	C(31)	C(32)	C(33)	122(1)
F(24)	C(33)	C(32)	119(1)	F(24)	C(33)	C(34)	120(1)
C(32)	C(33)	C(34)	120(1)	F(25)	C(34)	C(33)	122(1)
F(25)	C(34)	C(35)	118(1)	C(33)	C(34)	C(35)	119(1)
F(26)	C(35)	C(34)	122(1)	F(26)	C(35)	C(36)	116.9(10)
C(34)	C(35)	C(36)	121(1)	F(27)	C(36)	C(31)	118.4(9)
F(27)	C(36)	C(35)	120.0(10)	C(31)	C(36)	C(35)	121(1)
C(38)	C(37)	C(44)	119.3(9)	C(38)	C(37)	C(50)	119.6(9)
C(44)	C(37)	C(50)	121.2(9)	C(37)	C(38)	C(39)	122.9(9)
C(37)	C(38)	C(43)	119.0(8)	C(39)	C(38)	C(43)	118.1(9)
C(38)	C(39)	C(40)	119.9(9)	C(39)	C(40)	C(41)	120(1)
C(40)	C(41)	C(42)	121(1)	C(41)	C(42)	C(43)	119.9(10)
C(38)	C(43)	C(42)	120.5(9)	C(37)	C(44)	C(45)	121.1(9)
C(37)	C(44)	C(49)	120.2(9)	C(45)	C(44)	C(49)	118.7(9)
C(44)	C(45)	C(46)	119.4(9)	C(45)	C(46)	C(47)	120.9(10)
C(46)	C(47)	C(48)	119(1)	C(47)	C(48)	C(49)	120(1)
C(44)	C(49)	C(48)	121.1(9)	C(37)	C(50)	C(51)	119.4(9)
C(37)	C(50)	C(55)	121.5(10)	C(51)	C(50)	C(55)	119.1(10)
C(50)	C(51)	C(52)	120.2(10)	C(51)	C(52)	C(53)	117(1)
C(52)	C(53)	C(54)	124(1)	C(53)	C(54)	C(55)	118(1)
C(50)	C(55)	C(54)	119(1)	C(57)	C(56)	C(62)	135(2)
C(56)	C(57)	C(58)	144(2)	C(56)	C(57)	C(63)	128(4)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(58)	C(57)	C(63)	42(2)	C(57)	C(58)	C(59)	104(4)
C(57)	C(58)	C(60)	125(2)	C(57)	C(58)	C(61)	116(3)
C(57)	C(58)	C(63)	32(1)	C(59)	C(58)	C(60)	33(2)
C(59)	C(58)	C(61)	124(5)	C(59)	C(58)	C(63)	101(5)
C(60)	C(58)	C(61)	117(3)	C(60)	C(58)	C(63)	105(3)
C(61)	C(58)	C(63)	132(3)	C(58)	C(59)	C(60)	122(4)
C(58)	C(59)	C(62)	111(4)	C(58)	C(59)	C(63)	51(3)
C(60)	C(59)	C(62)	121(2)	C(60)	C(59)	C(63)	116(2)
C(62)	C(59)	C(63)	78(1)	C(58)	C(60)	C(59)	24(2)
C(56)	C(62)	C(59)	143(2)	C(57)	C(63)	C(58)	105(3)
C(57)	C(63)	C(59)	107(3)	C(58)	C(63)	C(59)	26(1)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
C(38)	C(39)	H(1)	120.0	C(40)	C(39)	H(1)	120.1
C(39)	C(40)	H(2)	119.7	C(41)	C(40)	H(2)	119.8
C(40)	C(41)	H(3)	119.5	C(42)	C(41)	H(3)	119.5
C(41)	C(42)	H(4)	120.0	C(43)	C(42)	H(4)	120.1
C(38)	C(43)	H(5)	119.8	C(42)	C(43)	H(5)	119.8
C(44)	C(45)	H(6)	120.3	C(46)	C(45)	H(6)	120.3
C(45)	C(46)	H(7)	119.6	C(47)	C(46)	H(7)	119.5
C(46)	C(47)	H(8)	120.1	C(48)	C(47)	H(8)	120.0
C(47)	C(48)	H(9)	120.0	C(49)	C(48)	H(9)	120.0
C(44)	C(49)	H(10)	119.4	C(48)	C(49)	H(10)	119.5
C(50)	C(51)	H(11)	119.8	C(52)	C(51)	H(11)	120.0
C(51)	C(52)	H(12)	121.2	C(53)	C(52)	H(12)	121.3
C(52)	C(53)	H(13)	117.6	C(54)	C(53)	H(13)	117.7
C(53)	C(54)	H(14)	120.5	C(55)	C(54)	H(14)	120.5
C(50)	C(55)	H(15)	120.2	C(54)	C(55)	H(15)	120.1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C47
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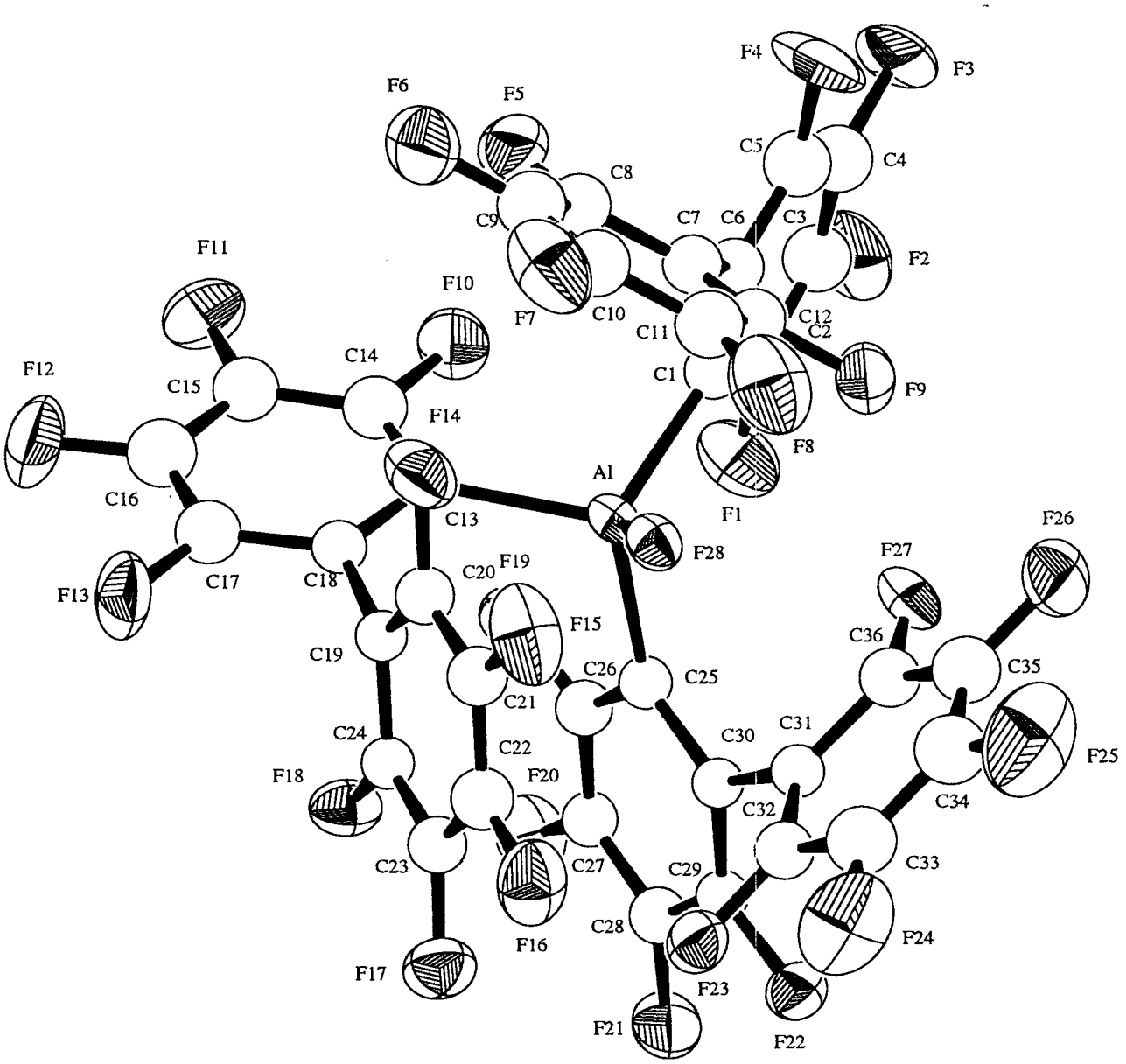
C46
C54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Supporting Information (IV)

X-ray Structure Report for $CGCZrCH_3^+PBA^-$ (19)

Sterically Encumbered (Perfluoroaryl) Borane and Aluminate Cocatalysts. Tuning Cation-Anion Ion Pair Structure and Reactivity in Metallocene Polymerization Through a Synthetic, Structural, and Polymerization Study

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Experimental

Data Collection

A colorless, transparent columnar crystal of $C_{59}H_{30}F_{28}NSiAlZr$ having approximate dimensions of 0.55 x 0.18 x 0.16 mm was mounted using oil, (Paratone-N, Exxon), on a glass fiber. All measurements were made on an Enraf-Nonius CAD4 diffractometer with graphite monochromated Mo-Kα radiation.

Cell constants and an orientation matrix for data collection , obtained from a least-squares refinement using the setting angles of 25 carefully centered reflections, (The whole crystal did not seem to diffract and was noticed when optically centering the crystal) in the range $20.0 < 2\theta < 23.6°$ *correspondedtoamonocliniccellwithdimensio*

a = 18.461(9) Å
b = 13.934(6) Å β = 108.34(4)°
c = 23.85(1) Å
V = 5822(4) Å^3

For Z = 4 and F.W. = 1431.13, the calculated density is 1.63 g/cm^3. The systematic absences of:

h0l: l $\neq$ 2n
0k0: k $\neq$ 2n

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -120 $\pm$ 1°C using the ω-θ scan technique to a maximum 2θ value of 52.0°. Omega scans of several intense reflections, made prior to data collection, had an average width at half-height of 0.35° with a take-off angle of 2.8°. Scans of (1.00 + 0.35 tan θ)° were made at a variable speed of 3.0-16.0°/min (in omega). Moving-crystal moving counter background measurements were made by scanning an additional 25% above and below the scan range. The counter aperture consisted of a variable horizontal slit with a width ranging from 2.0 to 2.5 mm and a vertical slit set to 2.0 mm. The diameter of the incident beam collimator was 0.7 mm and the crystal to detector distance was 21 cm. For intense reflections an attenuator was automatically inserted in front of the detector.

Data Reduction

Of the 12225 reflections which were collected, 12068 were unique (R_{int} = 0.089). The intensities of three representative reflection were measured after every 90 minutes of X-ray exposure time. No decay correction was applied.

The linear absorption coefficient, μ, for Mo-Kα radiation is 3.5 cm^{-1}. An analytical absorption correction was applied which resulted in transmission factors ranging from 0.92 to 0.95. The data were corrected for Lorentz and polarization effects. A correction for secondary extinction was applied (coefficient = 8.47714e-08).

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The disordered toluene carbon atoms were refined isotropically while the remaining non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included in idealized positions but not refined. The were not included in the structure factors for the disordered toluene. The methyl group for the disordered toluene refined to occupancy of 69C59 and 31distances to 1.4 angstroms. The final cycle of full-matrix least-squares refinement[3] was based on 4445 observed reflections ($I > 2.50\sigma(I)$) and 786 variable parameters and converged (largest parameter shift was 0.06 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.071$$

$$R_w = \sqrt{(\Sigma w(|Fo| - |Fc|)^2/\Sigma wFo^2)} = 0.056$$

The standard deviation of an observation of unit weight[4] was 2.05. The weighting scheme was based on counting statistics. 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 0.74 and -0.85 $e^-/\AA^3$, respectively.

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

References

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

(2) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least-Squares:

Function minimized: $\Sigma w(|Fo| - |Fc|)^2$

where w = $\frac{1}{\sigma^2(Fo)} = \frac{4Fo^2}{\sigma^2(Fo^2)}$

$\sigma^2(Fo^2) = \frac{S^2(C+R^2B)+(pFo^2)^2}{Lp^2}$

S = Scan rate

C = Total integrated peak count

R = Ratio of scan time to background counting time

B = Total background count

Lp = Lorentz-polarization factor

p = p-factor

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

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = 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) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1992).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{59}H_{30}F_{28}NSiAlZr$
Formula Weight	1431.13
Crystal Color, Habit	colorless, columnar
Crystal Dimensions	0.55 X 0.18 X 0.16 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit	
Cell Determination (2θ range)	25 (20.0 - 23.6°)
Omega Scan Peak Width	
at Half-height	0.35°
Lattice Parameters	a = 18.461(9) Å b = 13.934(6) Å c = 23.85(1) Å β = 108.34(4)°
	V = 5822(4) Å^3
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.632 g/cm^3
F_{000}	2840.00
μ(MoKα)	3.55 cm^{-1}

B. Intensity Measurements

Diffractometer	CAD4

Radiation	MoKα (λ = 0.71069 Å) graphite monochromated
Attenuator	Zr foil (factor = 22.25)
Take-off Angle	2.8°
Detector Aperture	2.0 - 2.5 mm horizontal 2.0 mm vertical
Crystal to Detector Distance	21 mm
Temperature	-120.0°C
Scan Type	ω-θ
Scan Rate	3.0-16.0°/min (in ω) (up to 0 scans)
Scan Width	(1.00 + 0.35 tan θ)°
$2\theta_{max}$	52.0°
No. of Reflections Measured	Total: 12225 Unique: 12068 (R_{int} = 0.089)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.9186 - 0.9486) Secondary Extinction (coefficient: 8.47714e-08)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(\|Fo\| - \|Fc\|)^2$
Least Squares Weights	$\frac{1}{\sigma^2(Fo)} = \frac{4Fo^2}{\sigma^2(Fo^2)}$
p-factor	0.0000
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>2.50σ(I))	4445
No. Variables	786
Reflection/Parameter Ratio	5.66

Residuals: R; Rw	0.071 : 0.056
Goodness of Fit Indicator	2.05
Max Shift/Error in Final Cycle	0.06
Maximum peak in Final Diff. Map	0.74 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.85 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}
Zr	0.22076(5)	0.40390(6)	0.59761(4)	2.45(2)
Si	0.1419(1)	0.5389(2)	0.4955(1)	3.09(6)
Al	0.3143(1)	0.1507(2)	0.6317(1)	2.74(6)
F(1)	0.2692(2)	0.2650(3)	0.6181(2)	3.2(1)
F(2)	0.2532(3)	0.2315(3)	0.7259(2)	4.7(1)
F(3)	0.2910(3)	0.2133(4)	0.8421(2)	6.3(2)
F(4)	0.4199(3)	0.1161(4)	0.9027(2)	7.4(2)
F(5)	0.5029(3)	0.0265(4)	0.8434(2)	5.8(2)
F(6)	0.4059(2)	-0.1171(3)	0.7230(2)	5.0(1)
F(7)	0.4979(3)	-0.2099(3)	0.6740(2)	6.1(2)
F(8)	0.6136(3)	-0.1160(4)	0.6507(2)	5.4(2)
F(9)	0.6364(2)	0.0732(3)	0.6768(2)	4.9(1)
F(10)	0.5478(2)	0.1653(3)	0.7306(2)	4.3(1)
F(11)	0.4365(2)	0.2909(3)	0.6699(2)	3.8(1)
F(12)	0.5614(3)	0.3350(4)	0.6401(2)	5.5(2)
F(13)	0.5887(3)	0.2437(4)	0.5483(2)	6.3(2)
F(14)	0.4914(3)	0.1105(4)	0.4872(2)	5.0(1)
F(15)	0.3231(3)	0.1703(4)	0.4340(2)	5.3(2)
F(16)	0.2295(3)	0.0430(5)	0.3597(2)	7.9(2)
F(17)	0.2152(3)	-0.1381(4)	0.3979(2)	8.7(2)
F(18)	0.2944(3)	-0.1892(4)	0.5098(2)	6.8(2)
F(19)	0.3878(3)	-0.0638(3)	0.5827(2)	4.7(1)
F(20)	0.2197(2)	0.1797(3)	0.5092(2)	3.7(1)
F(21)	0.1028(3)	0.0896(4)	0.4327(2)	5.6(1)

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

atom	x	y	z	B_{eq}
F(22)	0.0403(3)	-0.0693(4)	0.4660(2)	5.9(2)
F(23)	0.0976(3)	-0.1369(3)	0.5761(2)	4.9(1)
F(24)	0.2630(3)	-0.1979(4)	0.6264(2)	5.2(2)
F(25)	0.2954(3)	-0.2831(3)	0.7318(2)	6.2(2)
F(26)	0.2702(3)	-0.1917(4)	0.8245(2)	7.2(2)
F(27)	0.2095(3)	-0.0120(4)	0.8093(2)	6.7(2)
F(28)	0.1700(3)	0.0695(3)	0.7024(2)	4.9(1)
N	0.2286(3)	0.4825(4)	0.5282(2)	2.6(1)
C(1)	0.0988(4)	0.4802(5)	0.5490(3)	2.5(2)
C(2)	0.1173(4)	0.5077(6)	0.6090(3)	2.6(2)
C(3)	0.1151(4)	0.4247(6)	0.6443(3)	3.0(2)
C(4)	0.0946(4)	0.3462(6)	0.6055(3)	2.6(2)
C(5)	0.0851(4)	0.3763(5)	0.5482(3)	2.5(1)
C(6)	0.1332(4)	0.6086(6)	0.6352(3)	3.9(2)
C(7)	0.1253(5)	0.4243(6)	0.7091(3)	4.5(2)
C(8)	0.0799(5)	0.2464(6)	0.6233(4)	4.2(2)
C(9)	0.0637(4)	0.3159(6)	0.4937(3)	3.3(2)
C(10)	0.1424(5)	0.6743(6)	0.4963(4)	5.0(2)
C(11)	0.0931(5)	0.5057(7)	0.4169(3)	4.9(2)
C(12)	0.3002(5)	0.4939(7)	0.5137(4)	4.8(2)
C(13)	0.3412(6)	0.5883(8)	0.5426(6)	9.3(4)
C(14)	0.2879(6)	0.502(1)	0.4494(5)	11.9(4)
C(15)	0.3528(5)	0.4116(7)	0.5422(4)	5.0(3)
C(16)	0.2962(4)	0.4739(6)	0.6778(3)	4.0(2)

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

atom	x	y	z	B_{eq}
C(17)	0.3545(4)	0.1350(5)	0.7198(3)	2.6(2)
C(18)	0.3151(5)	0.1790(6)	0.7530(4)	3.1(2)
C(19)	0.3334(6)	0.1744(8)	0.8126(4)	4.9(3)
C(20)	0.3970(6)	0.1223(7)	0.8427(4)	4.7(3)
C(21)	0.4405(5)	0.0750(6)	0.8119(4)	4.0(2)
C(22)	0.4193(4)	0.0793(6)	0.7511(3)	2.8(2)
C(23)	0.4707(4)	0.0290(6)	0.7246(3)	2.8(2)
C(24)	0.4614(5)	-0.0690(6)	0.7116(4)	3.6(2)
C(25)	0.5091(5)	-0.1169(6)	0.6879(4)	3.9(2)
C(26)	0.5673(5)	-0.0694(7)	0.6752(4)	4.0(2)
C(27)	0.5793(5)	0.0274(7)	0.6881(4)	3.5(2)
C(28)	0.5319(5)	0.0729(6)	0.7134(3)	3.1(2)
C(29)	0.3981(4)	0.1724(5)	0.5981(3)	2.5(2)
C(30)	0.4498(4)	0.2422(6)	0.6252(4)	3.2(2)
C(31)	0.5126(5)	0.2659(6)	0.6097(4)	3.7(2)
C(32)	0.5277(5)	0.2213(6)	0.5648(4)	4.0(2)
C(33)	0.4767(5)	0.1516(6)	0.5330(4)	3.6(2)
C(34)	0.4148(4)	0.1289(5)	0.5499(3)	2.9(2)
C(35)	0.3605(4)	0.0560(6)	0.5092(3)	2.9(2)
C(36)	0.3182(5)	0.0796(7)	0.4528(4)	3.6(2)
C(37)	0.2675(5)	0.0141(8)	0.4147(4)	4.6(2)
C(38)	0.2631(6)	-0.0768(9)	0.4342(5)	5.5(3)
C(39)	0.3017(5)	-0.1023(8)	0.4907(4)	4.3(2)
C(40)	0.3493(5)	-0.0352(6)	0.5270(4)	3.3(2)

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

atom	x	y	z	B_{eq}
C(41)	0.2267(4)	0.0687(5)	0.5853(3)	2.6(2)
C(42)	0.1929(4)	0.1010(6)	0.5285(3)	3.0(2)
C(43)	0.1317(5)	0.0544(7)	0.4875(4)	4.0(2)
C(44)	0.1007(5)	-0.0248(7)	0.5048(4)	4.0(2)
C(45)	0.1321(5)	-0.0607(6)	0.5614(4)	3.6(2)
C(46)	0.1926(4)	-0.0157(6)	0.6018(3)	2.6(2)
C(47)	0.2180(4)	-0.0602(6)	0.6618(3)	2.7(2)
C(48)	0.2492(5)	-0.1528(7)	0.6708(4)	3.8(2)
C(49)	0.2665(5)	-0.1951(7)	0.7247(4)	4.4(2)
C(50)	0.2554(6)	-0.1498(8)	0.7721(5)	5.2(3)
C(51)	0.2238(6)	-0.0603(8)	0.7649(4)	5.0(3)
C(52)	0.2067(5)	-0.0166(7)	0.7106(4)	3.5(2)
C(53)	0.9766(9)	0.151(1)	0.7303(7)	10.8(4)
C(54)	1.0465(8)	0.1547(10)	0.7752(6)	10.5(4)
C(55)	1.0630(10)	0.233(1)	0.8144(7)	11.0(4)
C(56)	1.0089(9)	0.304(1)	0.8090(6)	11.7(4)
C(57)	0.9474(9)	0.300(1)	0.7573(7)	14.8(5)
C(58)	0.9277(9)	0.222(1)	0.7176(6)	13.1(5)
C(59)	0.9735(9)	0.072(1)	0.6929(7)	9.4(6)
C(60)	1.131(3)	0.235(3)	0.838(2)	10(1)
H(1)	0.1817	0.6097	0.6648	4.7384
H(2)	0.1330	0.6530	0.6049	4.7384
H(3)	0.0949	0.6258	0.6522	4.7384
H(4)	0.0785	0.4074	0.7152	5.3846

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

atom	x	y	z	B_{eq}
H(5)	0.1634	0.3789	0.7283	5.3846
H(6)	0.1405	0.4863	0.7250	5.3846
H(7)	0.0280	0.2299	0.6042	5.0933
H(8)	0.1120	0.2023	0.6120	5.0933
H(9)	0.0903	0.2439	0.6649	5.0933
H(10)	0.0158	0.2863	0.4888	3.9400
H(11)	0.0603	0.3552	0.4604	3.9400
H(12)	0.1014	0.2679	0.4972	3.9400
H(13)	0.1665	0.6963	0.5355	5.9743
H(14)	0.1696	0.6973	0.4712	5.9743
H(15)	0.0914	0.6973	0.4827	5.9743
H(16)	0.0429	0.5314	0.4046	5.9029
H(17)	0.1209	0.5310	0.3928	5.9029
H(18)	0.0907	0.4378	0.4133	5.9029
H(19)	0.3511	0.5855	0.5841	11.1580
H(20)	0.3880	0.5948	0.5342	11.1580
H(21)	0.3094	0.6418	0.5270	11.1580
H(22)	0.2569	0.5564	0.4342	14.2343
H(23)	0.3357	0.5091	0.4427	14.2343
H(24)	0.2631	0.4458	0.4301	14.2343
H(25)	0.3287	0.3523	0.5277	5.9837
H(26)	0.3991	0.4167	0.5328	5.9837
H(27)	0.3637	0.4142	0.5838	5.9837
H(28)	0.2896	0.4445	0.7118	4.8517

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

atom	x	y	z	B_{eq}
H(29)	0.3478	0.4674	0.6788	4.8517
H(30)	0.2836	0.5401	0.6773	4.8517

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Zr	0.0328(5)	0.0237(5)	0.0347(5)	0.0040(5)	0.0076(4)	-0.0003(5)
Si	0.040(2)	0.035(2)	0.043(2)	0.007(1)	0.013(1)	0.005(1)
Al	0.035(2)	0.026(1)	0.046(2)	0.007(1)	0.017(1)	0.004(1)
F(1)	0.032(3)	0.028(2)	0.060(3)	0.014(2)	0.010(2)	0.006(2)
F(2)	0.054(3)	0.049(4)	0.086(4)	0.012(3)	0.035(3)	-0.012(3)
F(3)	0.111(5)	0.072(4)	0.082(4)	-0.026(3)	0.067(4)	-0.047(3)
F(4)	0.113(5)	0.132(6)	0.039(3)	-0.038(4)	0.026(3)	-0.014(4)
F(5)	0.065(4)	0.094(5)	0.046(3)	-0.005(3)	-0.004(3)	0.021(3)
F(6)	0.053(3)	0.041(4)	0.103(4)	-0.008(3)	0.035(3)	0.004(3)
F(7)	0.079(4)	0.032(3)	0.125(5)	0.005(3)	0.039(4)	-0.004(3)
F(8)	0.067(4)	0.065(4)	0.085(4)	0.030(3)	0.040(3)	0.003(3)
F(9)	0.043(3)	0.072(4)	0.074(4)	-0.005(3)	0.025(3)	0.006(3)
F(10)	0.049(3)	0.049(3)	0.066(3)	-0.013(3)	0.019(3)	-0.009(3)
F(11)	0.051(3)	0.037(3)	0.058(3)	-0.002(2)	0.018(3)	-0.012(3)
F(12)	0.051(4)	0.051(4)	0.110(5)	-0.015(3)	0.030(3)	-0.021(3)
F(13)	0.065(4)	0.069(4)	0.131(5)	-0.011(3)	0.068(3)	-0.003(4)
F(14)	0.076(4)	0.063(4)	0.075(4)	0.002(3)	0.056(3)	-0.001(3)
F(15)	0.091(4)	0.059(4)	0.057(4)	0.024(3)	0.030(3)	0.034(3)
F(16)	0.101(5)	0.157(6)	0.032(3)	0.001(4)	0.007(3)	0.004(4)
F(17)	0.110(5)	0.117(6)	0.097(5)	-0.039(4)	0.023(4)	-0.058(4)
F(18)	0.114(5)	0.037(4)	0.119(5)	-0.024(4)	0.051(4)	-0.014(3)
F(19)	0.076(4)	0.041(4)	0.058(3)	0.010(3)	0.013(3)	0.020(3)
F(20)	0.054(3)	0.037(3)	0.052(3)	0.008(2)	0.020(3)	0.019(2)
F(21)	0.070(4)	0.090(4)	0.044(3)	0.011(4)	0.004(3)	0.007(3)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(22)	0.057(4)	0.084(5)	0.072(4)	-0.026(3)	0.002(3)	-0.024(3)
F(23)	0.063(4)	0.044(4)	0.082(4)	-0.027(3)	0.025(3)	-0.006(3)
F(24)	0.069(4)	0.054(4)	0.080(4)	0.013(3)	0.030(3)	-0.011(3)
F(25)	0.072(4)	0.040(4)	0.107(4)	0.004(3)	0.006(3)	0.023(3)
F(26)	0.093(4)	0.107(5)	0.069(4)	-0.009(4)	0.016(3)	0.050(4)
F(27)	0.117(5)	0.100(5)	0.046(3)	-0.001(4)	0.038(3)	0.000(3)
F(28)	0.073(4)	0.060(4)	0.061(3)	0.006(3)	0.032(3)	-0.007(3)
N	0.041(3)	0.023(4)	0.031(4)	0.004(3)	0.008(3)	0.002(2)
C(1)	0.033(4)	0.025(4)	0.036(4)	0.005(3)	0.007(3)	-0.005(3)
C(2)	0.026(4)	0.036(4)	0.035(4)	-0.001(3)	0.005(4)	-0.003(3)
C(3)	0.039(5)	0.036(5)	0.036(4)	0.006(4)	0.009(4)	0.006(3)
C(4)	0.027(5)	0.034(4)	0.037(4)	0.007(4)	0.011(4)	0.005(3)
C(5)	0.031(2)	0.026(4)	0.038(4)	0.004(4)	0.009(3)	-0.001(3)
C(6)	0.064(6)	0.033(5)	0.049(6)	0.023(5)	0.012(5)	-0.008(4)
C(7)	0.073(6)	0.063(7)	0.037(4)	0.021(6)	0.021(5)	0.005(5)
C(8)	0.066(6)	0.033(5)	0.063(6)	-0.005(5)	0.021(5)	0.011(5)
C(9)	0.043(6)	0.028(5)	0.047(5)	0.003(4)	0.004(4)	-0.011(4)
C(10)	0.068(7)	0.035(2)	0.088(8)	0.009(5)	0.027(6)	0.015(6)
C(11)	0.056(6)	0.090(8)	0.041(4)	0.002(6)	0.016(4)	0.004(5)
C(12)	0.041(6)	0.075(7)	0.079(6)	0.027(4)	0.035(5)	0.036(5)
C(13)	0.071(8)	0.069(8)	0.21(1)	-0.010(6)	0.045(9)	0.036(8)
C(14)	0.070(8)	0.32(2)	0.083(7)	0.08(1)	0.051(7)	0.10(1)
C(15)	0.047(6)	0.072(7)	0.073(7)	0.028(5)	0.022(5)	0.024(6)
C(16)	0.051(6)	0.047(6)	0.042(5)	0.006(5)	-0.006(4)	-0.008(4)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(17)	0.032(5)	0.023(5)	0.046(2)	-0.010(3)	0.017(3)	-0.007(4)
C(18)	0.042(5)	0.038(6)	0.050(5)	-0.014(3)	0.030(4)	-0.014(4)
C(19)	0.073(7)	0.065(8)	0.050(5)	-0.040(5)	0.021(4)	-0.020(5)
C(20)	0.074(7)	0.075(9)	0.037(3)	-0.037(5)	0.026(4)	-0.019(5)
C(21)	0.054(5)	0.050(7)	0.043(4)	-0.028(4)	0.009(4)	0.001(4)
C(22)	0.033(4)	0.033(6)	0.041(4)	-0.009(3)	0.010(3)	-0.006(4)
C(23)	0.036(5)	0.040(4)	0.031(5)	0.006(4)	0.008(4)	0.009(4)
C(24)	0.046(5)	0.039(5)	0.046(6)	0.010(4)	0.007(4)	0.016(4)
C(25)	0.058(6)	0.035(4)	0.057(6)	0.007(4)	0.021(5)	0.002(5)
C(26)	0.048(6)	0.062(6)	0.036(6)	0.006(4)	0.002(4)	0.007(5)
C(27)	0.036(5)	0.059(5)	0.038(6)	0.009(4)	0.012(4)	0.011(5)
C(28)	0.038(5)	0.043(4)	0.035(5)	0.001(4)	0.009(4)	0.007(4)
C(29)	0.034(4)	0.022(5)	0.037(5)	0.010(3)	0.010(3)	0.013(3)
C(30)	0.031(5)	0.031(5)	0.053(6)	0.009(4)	0.006(4)	0.000(4)
C(31)	0.039(5)	0.032(6)	0.071(7)	0.005(3)	0.021(4)	0.014(4)
C(32)	0.056(6)	0.030(6)	0.074(7)	0.007(4)	0.032(5)	0.012(4)
C(33)	0.049(6)	0.036(6)	0.064(6)	0.008(4)	0.034(5)	0.018(4)
C(34)	0.044(5)	0.032(6)	0.033(5)	0.011(3)	0.010(4)	0.018(3)
C(35)	0.043(5)	0.046(5)	0.032(4)	0.000(4)	0.028(3)	0.003(3)
C(36)	0.050(6)	0.058(5)	0.036(4)	0.010(5)	0.022(4)	0.006(4)
C(37)	0.054(7)	0.095(7)	0.027(4)	-0.004(6)	0.015(4)	-0.022(4)
C(38)	0.057(7)	0.093(8)	0.066(6)	0.000(6)	0.028(5)	-0.012(5)
C(39)	0.063(7)	0.040(4)	0.066(5)	-0.002(5)	0.029(5)	-0.021(4)
C(40)	0.036(6)	0.039(5)	0.051(4)	0.009(4)	0.015(4)	0.001(4)

Table 2. Anisotropic Displacement Parameters (continued)

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(41)	0.030(4)	0.034(5)	0.040(4)	0.011(3)	0.018(3)	0.001(3)
C(42)	0.043(5)	0.036(5)	0.041(4)	0.012(4)	0.023(3)	0.004(4)
C(43)	0.041(6)	0.073(8)	0.039(4)	0.002(4)	0.017(4)	-0.002(4)
C(44)	0.046(6)	0.056(7)	0.048(5)	0.002(4)	0.011(4)	-0.019(4)
C(45)	0.045(6)	0.045(6)	0.049(5)	-0.007(4)	0.020(4)	-0.012(4)
C(46)	0.041(5)	0.025(5)	0.037(4)	0.013(3)	0.017(4)	-0.003(3)
C(47)	0.034(5)	0.033(5)	0.036(4)	-0.012(4)	0.010(4)	-0.001(4)
C(48)	0.036(6)	0.039(5)	0.058(5)	0.002(5)	0.001(5)	0.014(4)
C(49)	0.049(6)	0.043(5)	0.065(5)	0.003(5)	0.004(6)	0.027(5)
C(50)	0.065(8)	0.069(7)	0.059(5)	-0.028(6)	0.014(6)	0.014(5)
C(51)	0.070(8)	0.075(7)	0.047(6)	-0.012(6)	0.023(6)	0.014(5)

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 3. Bond Lengths(Å)

atom	atom	distance	atom	atom	distance
Zr	Si	3.059(4)	Zr	F(1)	2.123(6)
Zr	N	2.027(8)	Zr	C(1)	2.43(1)
Zr	C(2)	2.48(1)	Zr	C(3)	2.55(1)
Zr	C(4)	2.53(1)	Zr	C(5)	2.44(1)
Zr	C(16)	2.21(1)	Si	N	1.733(8)
Si	C(1)	1.89(1)	Si	C(10)	1.89(1)
Si	C(11)	1.86(1)	Al	F(1)	1.780(6)
Al	C(17)	2.01(1)	Al	C(29)	1.98(1)
Al	C(41)	2.00(1)	F(2)	C(18)	1.34(1)
F(3)	C(19)	1.32(1)	F(4)	C(20)	1.36(1)
F(5)	C(21)	1.34(1)	F(6)	C(24)	1.32(1)
F(7)	C(25)	1.34(1)	F(8)	C(26)	1.34(1)
F(9)	C(27)	1.33(1)	F(10)	C(28)	1.35(1)
F(11)	C(30)	1.35(1)	F(12)	C(31)	1.36(1)
F(13)	C(32)	1.34(1)	F(14)	C(33)	1.33(1)
F(15)	C(36)	1.35(1)	F(16)	C(37)	1.34(1)
F(17)	C(38)	1.33(2)	F(18)	C(39)	1.32(1)
F(19)	C(40)	1.35(1)	F(20)	C(42)	1.34(1)
F(21)	C(43)	1.34(1)	F(22)	C(44)	1.35(1).
F(23)	C(45)	1.34(1)	F(24)	C(48)	1.32(1)
F(25)	C(49)	1.33(1)	F(26)	C(50)	1.33(1)
F(27)	C(51)	1.35(1)	F(28)	C(52)	1.36(1)
N	C(12)	1.48(1)	C(1)	C(2)	1.41(1)
C(1)	C(5)	1.47(1)	C(2)	C(3)	1.44(1)