Inorganic Chemistry
including bioinorganic chemistry
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Table 1. Data Collection and Processing Parameters

Space group	$R\bar{3}c$ (rhombohedral)
Cell constants	a = 19.294(3)Å
	$\alpha = 31.89(1)^{o}$
	$V = 1781Å^{3}$
Molecular formula	$C_{18}H_{54}Si_{6}Ge_{3}$
Formula weight	657.03
Formula units per cell	Z = 2
Density	$\rho = 1.23$ g-cm^{-3}
Absorption coefficient	$\mu = 26.9$ cm^{-1}
Temperature	$T = -50^{o}C$
Radiation (Mo Kα)	$\lambda = 0.71073Å$
Collection range	$4^{o} \leq 2\theta \leq 50^{o}$
Scan width	$\Delta\theta = 1.25+(K\alpha_{2}-K\alpha_{1})^{o}$
Scan speed range	1.5 to 15.0^{o}-min^{-1}
Total data collected	1262
Independent data, I>3σ(I)	818
Total variables	43
$R=\Sigma\|\|F_{o}\|-\|F_{c}\|\|/\Sigma\|F_{o}\|$	0.026
$R_{w}=[\Sigma w(\|F_{o}\|-\|F_{c}\|)^{2}/\Sigma w\|F_{o}\|^{2}]^{\frac{1}{2}}$	0.027
Weights	$w = \sigma(F)^{-2}$

Table 2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2 x10^3$)

	x	y	z	U(eq)
Ge	1160(1)	3840(1)	2500	37(1)
Si	134(1)	5517(1)	2893(1)	47(3)
C(1)	1488(5)	5487(5)	2434(5)	71(18)
C(2)	-782(5)	7471(4)	1679(5)	67(15)
C(3)	-1315(4)	5128(5)	4872(5)	67(15)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor

Table 3. Bond lengths (Å)

Ge-Si	2.388 (4)	Ge-Ge'	2.460 (1)
Si-C(1)	1.874 (4)	Si-C(2)	1.882 (5)
Si-C(3)	1.879 (4)		

Table 4. Bond angles (°)

Si-Ge-Ge'	120.7(1)	Si-Ge-Ge"	121.9(1)
Ge'-Ge-Ge"	60.0	Si-Ge-Si'	106.3(1)
Ge-Si-C(1)	115.7(3)	Ge-Si-C(2)	109.7(4)
C(1)-Si-C(2)	104.8(4)	Ge-Si-C(3)	107.8(4)
C(1)-Si-C(3)	110.3(5)	C(2)-Si-C(3)	108.3(2)

Table 5. Anisotropic displacement parameters ($Å^2 x 10^3$)

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Ge	34(1)	34(1)	40(1)	-19(1)	-19(1)	-11(1)
Si	49(1)	40(1)	44(1)	-22(1)	-20(1)	-18(1)
C(1)	82(4)	75(4)	90(4)	-43(3)	-39(3)	-35(3)
C(2)	74(4)	41(3)	59(3)	-19(3)	-35(3)	-23(3)
C(3)	62(3)	61(3)	48(3)	-32(3)	-19(3)	-23(3)

The anisotropic displacement exponent takes the form:

$-2\pi^2(h^2a^{*2}U_{11} + ... + 2hka^*b^*U_{12})$

Table 6. H-Atom coordinates ($x10^4$) and isotropic displacement parameters ($Å^2 x10^3$)

	x	y	z	U
H(1A)	2024	4474	3042	93(6)
H(1B)	949	6211	2652	93(6)
H(1C)	2214	5760	1362	93(6)
H(2A)	-1528	7575	1863	93(6)
H(2B)	-20	7713	619	93(6)
H(2C)	-1285	8163	1909	93(6)
H(3A)	-849	4093	5536	93(6)
H(3B)	-2088	5252	5074	93(6)
H(3C)	-1781	5833	5061	93(6)

TORSION ANGLES FOR K321

GE'	GE	SI	C1	60.8
GE'	GE	SI	C2	179.1
GE'	GE	SI	C3	-63.2
GE"	GE	SI	C1	-10.8
GE"	GE	SI	C2	107.5
GE"	GE	SI	C3	-134.8
SI'	GE	SI	C1	-154.8
SI'	GE	SI	C2	-36.5
SI'	GE	SI	C3	81.3
GE"	GE'	GE	SI	-111.5
GE"	GE'	GE	GE"	0.0
GE"	GE'	GE	SI'	109.6
GE'	GE"	GE	SI	109.6
GE'	GE"	GE	GE'	0.0
GE'	GE"	GE	SI'	-111.5
GE	GE"	GE'	GE	0.0

LEAST-SQUARES PLANE NUMBER 1 (XO = OTHOGONAL, X = CRYSTAL COORDINATES)

0.1650 XO + 0.2710 YO + 0.9484 ZO = 13.7232

18.298 X + 18.298 Y + 18.298 Z = 13.7232

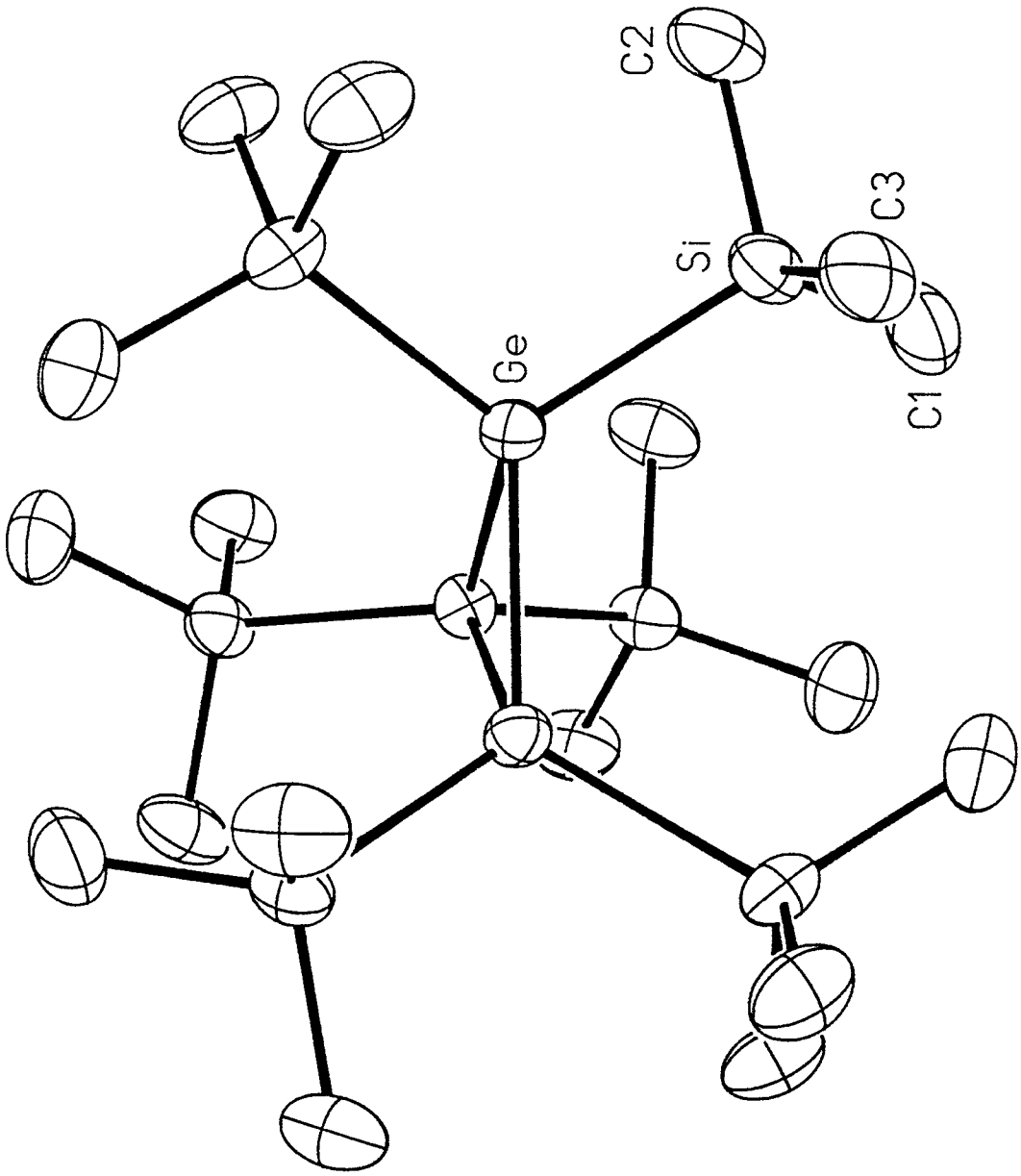
	DEVIATIONS	WEIGHT
GE	0.0000	1.0000+
SI	1.9101	1.0000
C1	3.4923	1.0000
C2	1.5871	1.0000
C3	2.1686	1.0000
SI'	-1.9101	1.0000
C1'	-3.4923	1.0000
C2'	-1.5871	1.0000
C3'	-2.1686	1.0000
GE'	0.0000	1.0000+
GE"	0.0000	1.0000+

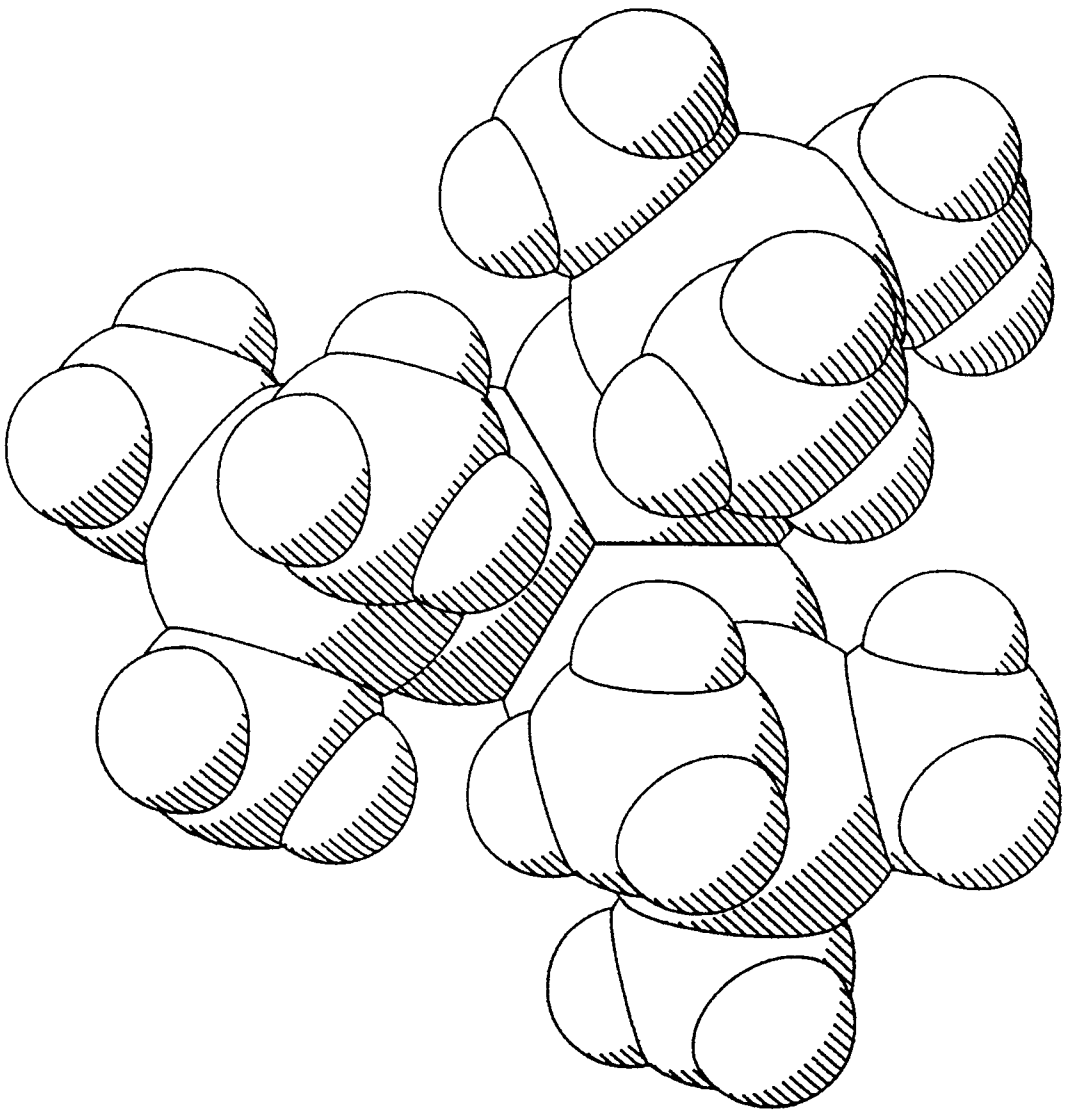
MEAN DEVIATION FROM PLANE = 0.0000 ANGSTROMS

Fig. 1 -- View of the molecule showing the atom numbering scheme. Thermal ellipsoids are 40% equiprobability envelopes, with hydrogens omitted.

Fig. 2 -- Space-filling view of the top half of the molecule along the 3-fold axis, showing the mutual twists of the trimethylsilyl ligands.

Fig. 3 -- Packing of the molecules in the unit cell.


C2
C3
Si
Ge
C1



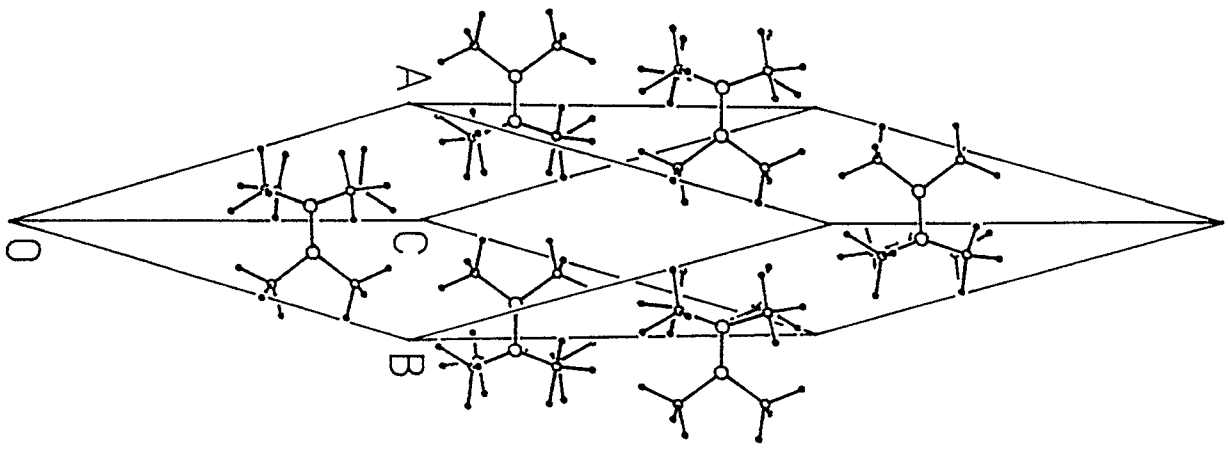

A
O
C
B

Table 1. Crystal data, data collection, and solution and refinement for 96346.

Crystal Data	
Empirical formula	$C_{30}H_{90}Cl_2Ge_5Si_{10}$
Crystal Habit, color	block, yellow
Crystal size	0.40 x 0.32 x 0.30 mm
Crystal system	Cubic
Space group	F23
	a = 22.7731(3) Å α = 90°
	b = 22.7731(3) Å β = 90°
	c = 22.7731(3) Å γ = 90°
Volume	11810.5(3) $Å^3$
Z	8
Formula weight	1165.77
Density (calculated)	1.311 Mg/m^3
Absorption coefficient	2.828 mm^{-1}
F(000)	4832
Data Collection	
Diffractometer	Siemens SMART Platform CCD
Wavelength	0.71073 Å
Temperature	173(2) K
θ range for data collection	1.55 to 24.95°
Index ranges	$-18 \le h \le 18$, $0 \le k \le 19$, $1 \le \ell \le 27$
Reflections collected	25664
Independent reflections	1759 (R_{int} = 0.0336)
Solution and Refinement	
System used	SHELXTL-V5.0
Solution	Direct methods
Refinement method	Full-matrix least-squares on F^2
Weighting scheme	$w = [\sigma^2(F_o^2)+(AP)^2+(BP)]^{-1}$, where P = $(Fo^2+2Fc^2))/3$, A = 0.1344, and B = 187.6490
Absorption correction	SADABS (Sheldrick, 1996)
Max. and min. transmission	1.0000 and 0.7396
Absolute structure parameter	0.02(5)
Data / restraints / parameters	1759 / 6 / 83
R indices (I>2σ(I) = 1561)	R1 = 0.0849, wR2 = 0.2208
R indices (all data)	R1 = 0.0941, wR2 = 0.2318
Goodness-of-fit on F^2	1.074
Largest diff. peak and hole	1.397 and -0.993 $eÅ^{-3}$

Table 2. Atomic coordinates [$x_3 10^4$] and equivalent isotropic displacement parameters [$Å^2$ x 10^3] for 96346. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)	SOF
Ge(1)	6958(1)	7411(1)	8043(1)	31(1)	0.33
Ge(2)	6488(1)	6488(1)	8512(1)	68(1)	1
Ge(3)	2500	7500	7500	65(1)	1
Ge(4)	5000	0	0	56(1)	1
Cl(1)	6647(1)	8353(1)	8353(1)	92(2)	1
Si(1)	5655(2)	7012(2)	8894(2)	79(1)	1
Si(2)	1894(2)	8106(2)	8106(2)	90(2)	1
Si(3)	4397(1)	-603(1)	-603(1)	67(2)	1
C(1)	5930(9)	7492(7)	9531(6)	105(5)	1
C(2)	5273(8)	7426(7)	8290(7)	95(5)	1
C(3)	5107(6)	6450(6)	9136(8)	93(4)	1
C(4)	1593(8)	7664(10)	8735(8)	119(6)	1
C(5)	4096(7)	-173(8)	-1229(7)	99(5)	1

Table 3. Bond lengths [Å] and angles [°] for 96346.

Ge(1)-Ge(1)#1	1.459(4)	Ge(1)-Ge(1)#2	1.460(4)
Ge(1)-Ge(1)#3	2.035(4)	Ge(1)-Ge(1)#4	2.035(4)
Ge(1)-Cl(1)	2.367(5)	Ge(1)-Ge(1)#5	2.503(5)
Ge(1)-Ge(1)#6	2.506(5)	Ge(1)-Ge(2)	2.589(3)
Ge(2)-Si(1)	2.404(4)	Ge(2)-Si(1)#1	2.404(4)
Ge(2)-Si(1)#2	2.404(4)	Ge(2)-Ge(1)#1	2.589(3)
Ge(2)-Ge(1)#2	2.589(3)	Ge(3)-Si(2)#7	2.392(7)
Ge(3)-Si(2)#6	2.392(8)	Ge(3)-Si(2)#8	2.392(7)
Ge(3)-Si(2)	2.392(7)	Ge(4)-Si(3)#9	2.378(6)
Ge(4)-Si(3)#10	2.378(6)	Ge(4)-Si(3)	2.378(6)
Ge(4)-Si(3)#11	2.378(6)	Cl(1)-Ge(1)#3	2.367(5)
Cl(1)-Ge(1)#4	2.367(5)	Si(1)-C(3)	1.870(14)
Si(1)-C(2)	1.881(14)	Si(1)-C(1)	1.92(2)
Si(2)-C(4)#12	1.88(2)	Si(2)-C(4)#13	1.88(2)
Si(2)-C(4)	1.88(2)	Si(3)-C(5)#14	1.86(2)
Si(3)-C(5)	1.86(2)	Si(3)-C(5)#15	1.86(2)

Ge(1)#1-Ge(1)-Ge(1)#2	60.0	Ge(1)#1-Ge(1)-Ge(1)#3	90.1(2)
Ge(1)#2-Ge(1)-Ge(1)#3	120.009(14)	Ge(1)#1-Ge(1)-Ge(1)#4	119.994(14)
Ge(1)#2-Ge(1)-Ge(1)#4	89.9(2)	Ge(1)#3-Ge(1)-Ge(1)#4	60.0
Ge(1)#1-Ge(1)-Cl(1)	148.5(2)	Ge(1)#2-Ge(1)-Cl(1)	148.3(2)
Ge(1)#3-Ge(1)-Cl(1)	64.53(6)	Ge(1)#4-Ge(1)-Cl(1)	64.53(6)
Ge(1)#1-Ge(1)-Ge(1)#5	54.4(2)	Ge(1)#2-Ge(1)-Ge(1)#5	96.67(14)
Ge(1)#3-Ge(1)-Ge(1)#5	35.67(12)	Ge(1)#4-Ge(1)-Ge(1)#5	83.44(12)
Cl(1)-Ge(1)-Ge(1)#5	98.47(13)	Ge(1)#1-Ge(1)-Ge(1)#6	96.54(14)
Ge(1)#2-Ge(1)-Ge(1)#6	54.3(2)	Ge(1)#3-Ge(1)-Ge(1)#6	83.35(12)
Ge(1)#4-Ge(1)-Ge(1)#6	35.62(12)	Cl(1)-Ge(1)-Ge(1)#6	98.44(13)
Ge(1)#5-Ge(1)-Ge(1)#6	88.49(4)	Ge(1)#1-Ge(1)-Ge(2)	73.63(5)
Ge(1)#2-Ge(1)-Ge(2)	73.63(5)	Ge(1)#3-Ge(1)-Ge(2)	150.1(2)
Ge(1)#4-Ge(1)-Ge(2)	149.9(2)	Cl(1)-Ge(1)-Ge(2)	119.29(11)
Ge(1)#5-Ge(1)-Ge(2)	122.68(13)	Ge(1)#6-Ge(1)-Ge(2)	122.62(13)
Si(1)-Ge(2)-Si(1)#1	106.62(11)	Si(1)-Ge(2)-Si(1)#2	106.62(11)
Si(1)#1-Ge(2)-Si(1)#2	106.62(11)	Si(1)-Ge(2)-Ge(1)#1	125.81(11)
Si(1)#1-Ge(2)-Ge(1)#1	94.15(11)	Si(1)#2-Ge(2)-Ge(1)#1	114.45(11)
Si(1)-Ge(2)-Ge(1)#2	114.45(11)	Si(1)#1-Ge(2)-Ge(1)#2	125.81(11)
Si(1)#2-Ge(2)-Ge(1)#2	94.15(11)	Ge(1)#1-Ge(2)-Ge(1)#2	32.75(9)
Si(1)-Ge(2)-Ge(1)	94.15(11)	Si(1)#1-Ge(2)-Ge(1)	114.45(11)
Si(1)#2-Ge(2)-Ge(1)	125.81(11)	Ge(1)#1-Ge(2)-Ge(1)	32.75(9)
Ge(1)#2-Ge(2)-Ge(1)	32.75(9)	Si(2)#7-Ge(3)-Si(2)#6	109.471(1)
Si(2)#7-Ge(3)-Si(2)#8	109.471(1)	Si(2)#6-Ge(3)-Si(2)#8	109.470(1)
Si(2)#7-Ge(3)-Si(2)	109.5	Si(2)#6-Ge(3)-Si(2)	109.5
Si(2)#8-Ge(3)-Si(2)	109.471(1)	Si(3)#9-Ge(4)-Si(3)#10	109.5
Si(3)#9-Ge(4)-Si(3)	109.5	Si(3)#10-Ge(4)-Si(3)	109.5
Si(3)#9-Ge(4)-Si(3)#11	109.5	Si(3)#10-Ge(4)-Si(3)#11	109.5
Si(3)-Ge(4)-Si(3)#11	109.5	Ge(1)-Cl(1)-Ge(1)#3	50.94(13)
Ge(1)-Cl(1)-Ge(1)#4	50.94(13)	Ge(1)#3-Cl(1)-Ge(1)#4	50.94(13)
C(3)-Si(1)-C(2)	104.5(8)	C(3)-Si(1)-C(1)	112.6(8)
C(2)-Si(1)-C(1)	114.7(8)	C(3)-Si(1)-Ge(2)	107.0(5)
C(2)-Si(1)-Ge(2)	110.5(5)	C(1)-Si(1)-Ge(2)	107.3(6)
C(4)#12-Si(2)-C(4)#13	109.1(7)	C(4)#12-Si(2)-C(4)	109.1(7)
C(4)#13-Si(2)-C(4)	109.1(6)	C(4)#12-Si(2)-Ge(3)	109.9(6)
C(4)#13-Si(2)-Ge(3)	109.9(6)	C(4)-Si(2)-Ge(3)	109.9(6)
C(5)#14-Si(3)-C(5)	108.4(6)	C(5)#14-Si(3)-C(5)#15	108.4(6)
C(5)-Si(3)-C(5)#15	108.3(6)	C(5)#14-Si(3)-Ge(4)	110.6(5)
C(5)-Si(3)-Ge(4)	110.6(5)	C(5)#15-Si(3)-Ge(4)	110.6(5)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters [Å^2 x 10^3] for 96346._2 The anisotropic displacement factor exponent takes the form: $-2\pi^2$ [$(ha^*)^2 U_{11}$ + ... + 2hka* b* U_{12}]

	U11	U22	U33	U23	U13	U12
Ge(1)	27(1)	37(1)	28(1)	2(1)	2(1)	-4(1)
Ge(2)	68(1)	68(1)	68(1)	19(1)	19(1)	-19(1)
Ge(3)	65(1)	65(1)	65(1)	0	0	0
Ge(4)	56(1)	56(1)	56(1)	0	0	0
Cl(1)	92(2)	92(2)	92(2)	-29(2)	29(2)	29(2)
Si(1)	103(3)	62(2)	72(2)	7(2)	2(2)	-3(2)
Si(2)	90(2)	90(2)	90(2)	-8(2)	8(2)	8(2)
Si(3)	67(2)	67(2)	67(2)	-8(1)	-8(1)	-8(1)
C(1)	172(17)	70(8)	72(8)	-16(7)	5(8)	4(9)
C(2)	125(13)	68(8)	94(9)	13(6)	-7(8)	28(8)
C(3)	67(8)	84(8)	129(12)	29(8)	18(8)	6(6)
C(4)	115(14)	141(17)	102(13)	-8(12)	17(11)	9(12)
C(5)	103(11)	102(12)	91(10)	5(9)	-42(9)	-1(9)

Table 5. Torsion angles [°] for 96346.

Ge(1)#1-Ge(1)-Ge(2)-Si(1)	166.3(2)	Ge(1)#2-Ge(1)-Ge(2)-Si(1)	-130.9(2)
Ge(1)#3-Ge(1)-Ge(2)-Si(1)	106.83(14)	Ge(1)#4-Ge(1)-Ge(2)-Si(1)	-71.52(14)
Cl(1)-Ge(1)-Ge(2)-Si(1)	17.61(14)	Ge(1)#5-Ge(1)-Ge(2)-Si(1)	141.70(13)
Ge(1)#6-Ge(1)-Ge(2)-Si(1)	-106.38(13)	Ge(1)#1-Ge(1)-Ge(2)-Si(1)#1	56.0(2)
Ge(1)#2-Ge(1)-Ge(2)-Si(1)#1	118.8(2)	Ge(1)#3-Ge(1)-Ge(2)-Si(1)#1	-3.5(2)
Ge(1)#4-Ge(1)-Ge(2)-Si(1)#1	178.11(13)	Cl(1)-Ge(1)-Ge(2)-Si(1)#1	-92.76(14)
Ge(1)#5-Ge(1)-Ge(2)-Si(1)#1	31.3(2)	Ge(1)#6-Ge(1)-Ge(2)-Si(1)#1	143.26(13)
Ge(1)#1-Ge(1)-Ge(2)-Si(1)#2	-79.7(2)	Ge(1)#2-Ge(1)-Ge(2)-Si(1)#2	-16.9(2)
Ge(1)#3-Ge(1)-Ge(2)-Si(1)#2	-139.21(14)	Ge(1)#4-Ge(1)-Ge(2)-Si(1)#2	42.4(2)
Cl(1)-Ge(1)-Ge(2)-Si(1)#2	131.57(14)	Ge(1)#5-Ge(1)-Ge(2)-Si(1)#2	-104.34(14)
Ge(1)#6-Ge(1)-Ge(2)-Si(1)#2	7.6(2)	Ge(1)#1-Ge(1)-Ge(2)-Ge(1)#1	0.0
Ge(1)#2-Ge(1)-Ge(2)-Ge(1)#1	62.82(2)	Ge(1)#3-Ge(1)-Ge(2)-Ge(1)#1	-59.5(2)
Ge(1)#4-Ge(1)-Ge(2)-Ge(1)#1	122.2(2)	Cl(1)-Ge(1)-Ge(2)-Ge(1)#1	-148.7(2)
Ge(1)#5-Ge(1)-Ge(2)-Ge(1)#1	-24.6(2)	Ge(1)#6-Ge(1)-Ge(2)-Ge(1)#1	87.3(2)
Ge(1)#1-Ge(1)-Ge(2)-Ge(1)#2	-62.82(2)	Ge(1)#2-Ge(1)-Ge(2)-Ge(1)#2	0.0
Ge(1)#3-Ge(1)-Ge(2)-Ge(1)#2	-122.3(2)	Ge(1)#4-Ge(1)-Ge(2)-Ge(1)#2	59.3(2)
Cl(1)-Ge(1)-Ge(2)-Ge(1)#2	148.5(2)	Ge(1)#5-Ge(1)-Ge(2)-Ge(1)#2	-87.4(2)
Ge(1)#6-Ge(1)-Ge(2)-Ge(1)#2	24.5(2)	Ge(1)#1-Ge(1)-Cl(1)-Ge(1)#3	39.2(2)
Ge(1)#2-Ge(1)-Cl(1)-Ge(1)#3	-106.44(10)	Ge(1)#3-Ge(1)-Cl(1)-Ge(1)#3	0.0
Ge(1)#4-Ge(1)-Cl(1)-Ge(1)#3	-67.26(4)	Ge(1)#5-Ge(1)-Cl(1)-Ge(1)#3	11.27(10)
Ge(1)#6-Ge(1)-Cl(1)-Ge(1)#3	-78.45(9)	Ge(2)-Ge(1)-Cl(1)-Ge(1)#3	146.5(2)
Ge(1)#1-Ge(1)-Cl(1)-Ge(1)#4	106.41(11)	Ge(1)#2-Ge(1)-Cl(1)-Ge(1)#4	-39.18(14)
Ge(1)#3-Ge(1)-Cl(1)-Ge(1)#4	67.26(4)	Ge(1)#4-Ge(1)-Cl(1)-Ge(1)#4	0.0
Ge(1)#5-Ge(1)-Cl(1)-Ge(1)#4	78.53(9)	Ge(1)#6-Ge(1)-Cl(1)-Ge(1)#4	-11.19(10)
Ge(2)-Ge(1)-Cl(1)-Ge(1)#4	-146.3(2)	Si(1)#1-Ge(2)-Si(1)-C(3)	-75.3(6)
Si(1)#2-Ge(2)-Si(1)-C(3)	38.4(6)	Ge(1)#1-Ge(2)-Si(1)-C(3)	176.8(6)
Ge(1)#2-Ge(2)-Si(1)-C(3)	141.0(6)	Ge(1)-Ge(2)-Si(1)-C(3)	167.7(6)
Si(1)#1-Ge(2)-Si(1)-C(2)	171.5(6)	Si(1)#2-Ge(2)-Si(1)-C(2)	-74.9(7)
Ge(1)#1-Ge(2)-Si(1)-C(2)	63.5(6)	Ge(1)#2-Ge(2)-Si(1)-C(2)	27.8(6)
Ge(1)-Ge(2)-Si(1)-C(2)	54.5(6)	Si(1)#1-Ge(2)-Si(1)-C(1)	45.8(6)
Si(1)#2-Ge(2)-Si(1)-C(1)	159.4(6)	Ge(1)#1-Ge(2)-Si(1)-C(1)	-62.2(6)
Ge(1)#2-Ge(2)-Si(1)-C(1)	-97.9(6)	Ge(1)-Ge(2)-Si(1)-C(1)	-71.2(6)
Si(2)#7-Ge(3)-Si(2)-C(4)#12	77.4(6)	Si(2)#6-Ge(3)-Si(2)-C(4)#12	-162.6(6)
Si(2)#8-Ge(3)-Si(2)-C(4)#12	-42.6(6)	Si(2)#7-Ge(3)-Si(2)-C(4)#13	-162.6(6)
Si(2)#6-Ge(3)-Si(2)-C(4)#13	-42.6(6)	Si(2)#8-Ge(3)-Si(2)-C(4)#13	77.4(6)
Si(2)#7-Ge(3)-Si(2)-C(4)	-42.6(6)	Si(2)#6-Ge(3)-Si(2)-C(4)	77.4(6)
Si(2)#8-Ge(3)-Si(2)-C(4)	-162.6(6)	Si(3)#9-Ge(4)-Si(3)-C(5)#14	-42.5(6)
Si(3)#10-Ge(4)-Si(3)-C(5)#14	77.5(6)	Si(3)#11-Ge(4)-Si(3)-C(5)#1	-162.5(6)
Si(3)#9-Ge(4)-Si(3)-C(5)	77.5(6)	Si(3)#10-Ge(4)-Si(3)-C(5)	-162.5(6)
Si(3)#11-Ge(4)-Si(3)-C(5)	-42.5(6)	Si(3)#9-Ge(4)-Si(3)-C(5)#15	-162.5(6)
Si(3)#10-Ge(4)-Si(3)-C(5)#15	-42.5(6)	Si(3)#11-Ge(4)-Si(3)-C(5)#15	77.5(6)

Symmetry transformations used to generate equivalent atoms:

#1 y,-z+3/2,-x+3/2 #2 -z+3/2,x,-y+3/2 #3 -y+3/2,z,-x+3/2
#4 -z+3/2,-x+3/2,y #5 -x+3/2,-y+3/2,z #6 x,-y+3/2,-z+3/2
#7 -x+1/2,-y+3/2,z #8 -x+1/2,y,-z+3/2 #9 x,-y,-z
#10 -x+1,y,-z #11 -x+1,-y,z #12 -y+1,z,-x+1 #13 -z+1,-x+1,y
#14 z+1/2,x-1/2,y #15 y+1/2,z,x-1/2

```
#1 y,-z+3/2,-x+3/2     #2 -z+3/2,x,-y+3/2     #3 -y+3/2,z,-x+3/2
#4 -z+3/2,-x+3/2,y     #5 -x+3/2,-y+3/2,z     #6 x,-y+3/2,-z+3/2
#7 -x+1/2,-y+3/2,z     #8 -x+1/2,y,-z+3/2     #9 x,-y,-z
#10 -x+1,y,-z     #11 -x+1,-y,z     #12 -y+1,z,-x+1     #13 -z+1,-x+1,y
#14 z+1/2,x-1/2,y     #15 y+1/2,z,x-1/2
```

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for 96346.

	x	y	z	U(eq)	SOF
H(1A)	6154(56)	7824(38)	9375(7)	157	1
H(1B)	6182(55)	7258(19)	9789(37)	157	1
H(1C)	5594(10)	7640(54)	9756(40)	157	1
H(2A)	5539(22)	7724(38)	8130(39)	143	1
H(2B)	4921(33)	7618(48)	8447(15)	143	1
H(2C)	5159(51)	7153(11)	7978(28)	143	1
H(3A)	5276(22)	6212(38)	9453(41)	140	1
H(3B)	5006(43)	6196(36)	8804(17)	140	1
H(3C)	4753(23)	6648(7)	9279(53)	140	1
H(4A)	1536(69)	7256(20)	8609(23)	179	1
H(4B)	1216(37)	7829(47)	8860(49)	179	1
H(4C)	1871(36)	7675(60)	9063(28)	179	1
H(5A)	3988(51)	221(21)	-1094(14)	148	1
H(5B)	3748(33)	-372(31)	-1385(36)	148	1
H(5C)	4394(21)	-142(47)	-1538(24)	148	1

DATA COLLECTION

A crystal of the compound was attached to a glass fiber and mounted on the Siemens SMART system for a data collection at 173(2) K. An initial set of cell constants was calculated from reflections harvested from three sets of 20 frames. These initial sets of frames are oriented such that orthogonal wedges of reciprocal space were surveyed. This produces orientation matrices determined from 211 reflections. Final cell constants are calculated from a set of 4306 strong reflections from the actual data collection. Final cell constants reported in this manner usually are about one order of magnitude better in precision than reported from four-circle diffractometers. Please refer to Table 1 for additional crystal and refinement information.

The data collection technique used for this specimen is generally known as a hemisphere collection. Here a randomly oriented region of reciprocal space is surveyed to the extent of 1.3 hemispheres to a resolution of 0.84 Å. Three major swaths of frames are collected with 0.30° steps in ω.

STRUCTURE SOLUTION AND REFINEMENT

The space group F23 was determined based on systematic absences and intensity statistics.[1] A successful direct-methods solution was calculated which provided most non-hydrogen atoms from the E-map. Several full-matrix least squares / difference Fourier cycles were performed which located the remainder of the non-hydrogen atoms. All non-hydrogen atoms were refined with anisotropic displacement parameters unless stated otherwise. All hydrogen atoms were placed in ideal positions and refined as riding atoms with group isotropic displacement parameters.

Two molecules of Ge(SiMe3)4 were found in addition to the expected complex. The central Ge4 core of the complex is disordered over three positions related by three-fold symmetry [ie Ge(1) is pulled off the crystallographic 3_1 axis]. It is this statistical disorder which likely results in the higher than expected residuals (R_{int} = 0.0336). The largest difference feature was located in the vicinity of Si(1). A second data set collected from a different sample did not yield a better result (R1 for that refinement was 10.36%).

Data collection and structure solution were conducted at the X-Ray Crystallographic Laboratory, 160 Kolthoff Hall, Chemistry Department, The University of Minnesota. All calculations were preformed using SGI INDY R4400-SC or Pentium computers using the SHELXTL V5.0 suite of programs. All publications arising from this report MUST either 1)include Victor G. Young, Jr. as a coauthor or 2)acknowledge both Victor G. Young, Jr. and the X-Ray

Crystallographic Laboratory.

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1. SHELXTL-Plus V5.0, Siemens Industrial Automation, Inc., Madison, WI.

Some equations of interest:

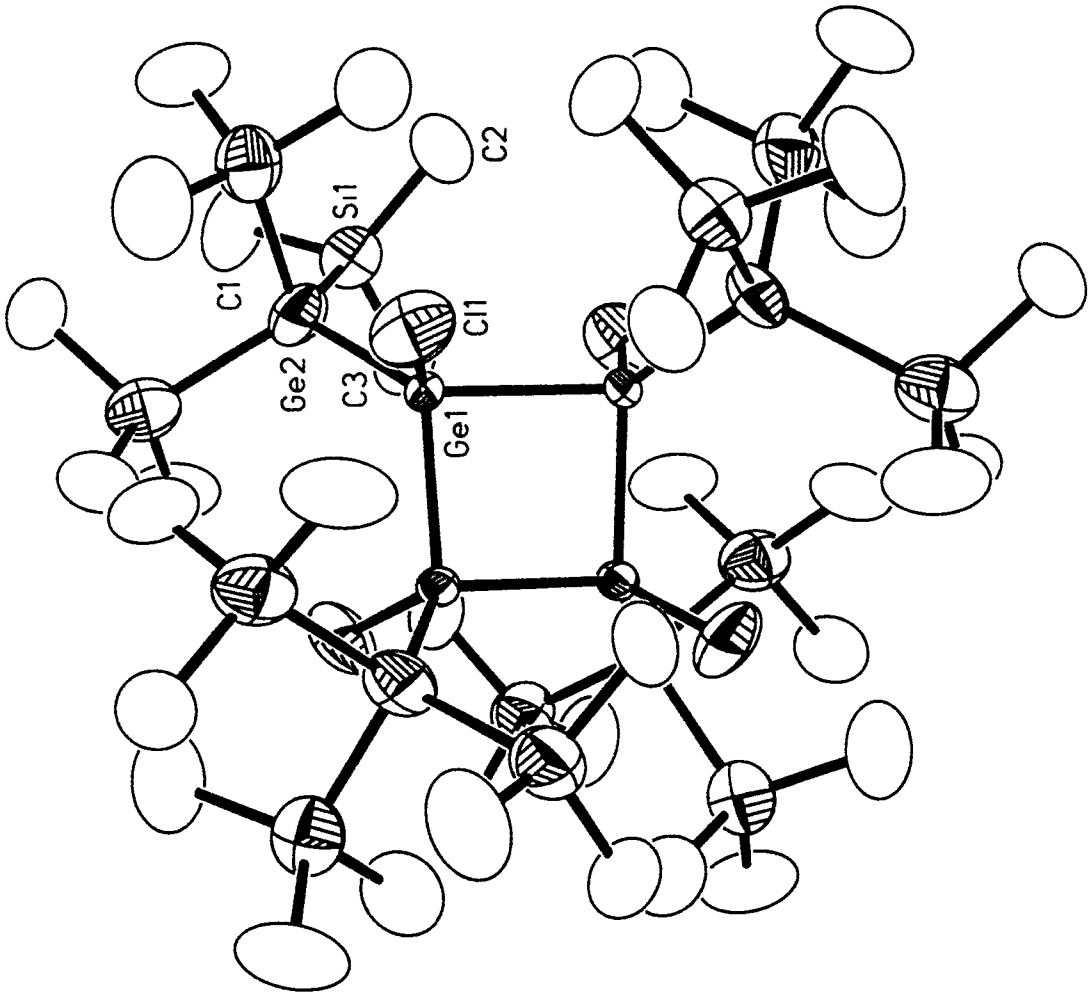
$$R_{int}=\sum|F_o^2-\langle F_o^2\rangle|/\sum|F_o^2|$$

$$R1=\sum||F_o|-|F_c||/\sum|F_o|$$

$$wR2=[\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2},$$

where $w=1/\sigma^2(F_o^2)+(a*P)^2+b*P$

$$GooF=S=[\sum[w(F_o^2-F_c^2)^2]/(n-p)]^{1/2}$$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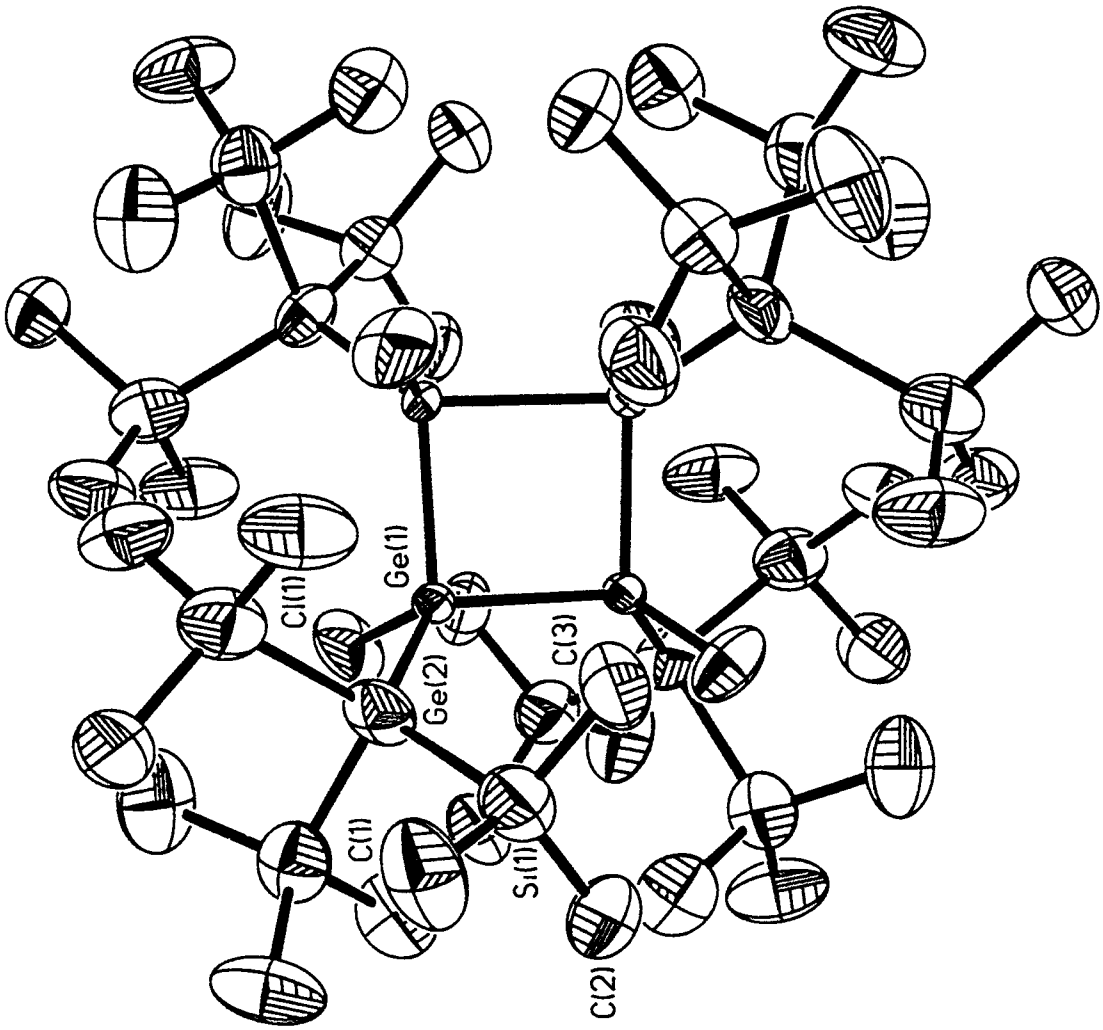
Si1
C1
Cl1
Ge2
C3
Ge1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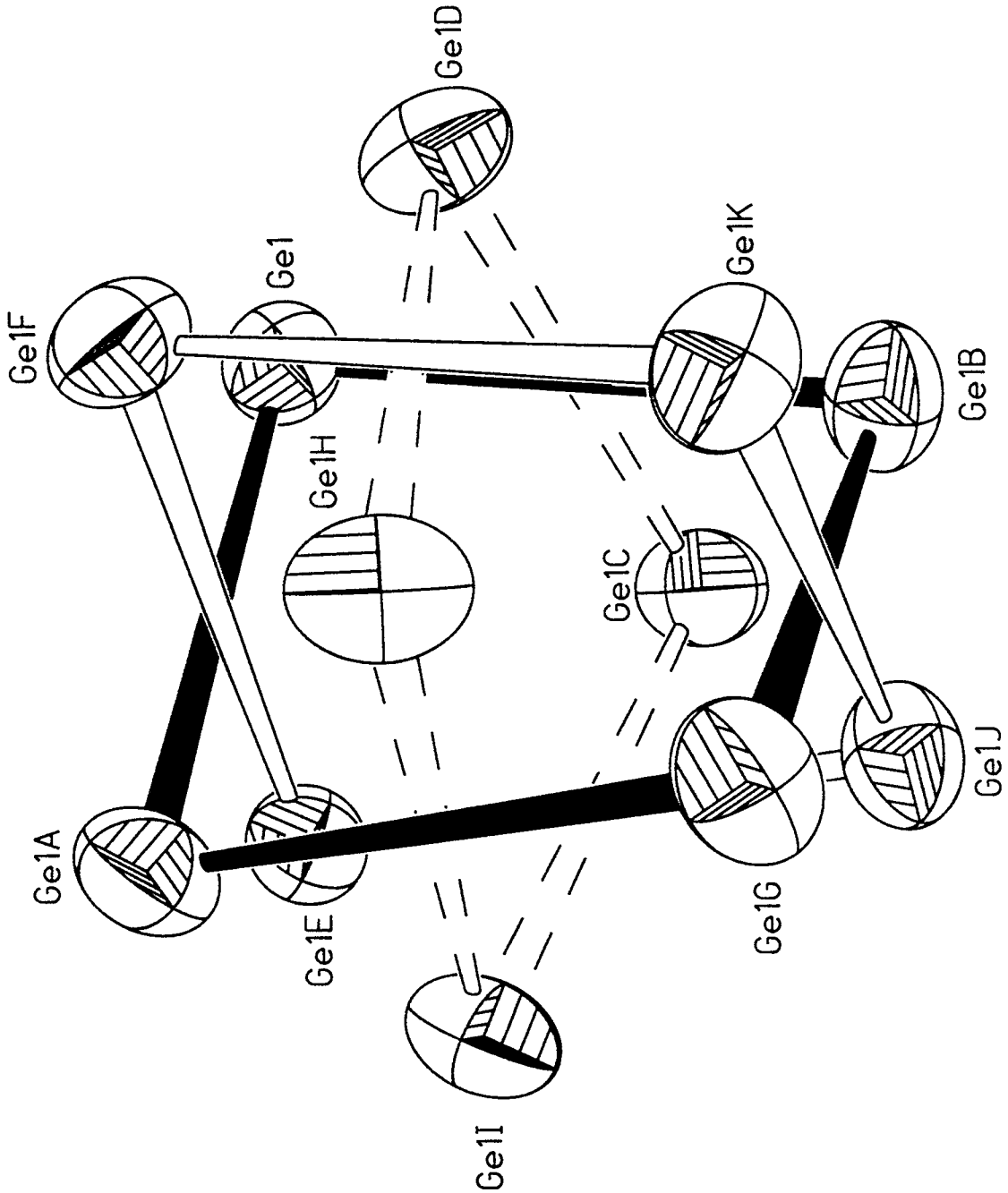
Ge(1)
Ge(2)
C(3)
C(1)
Si(1)
C(2)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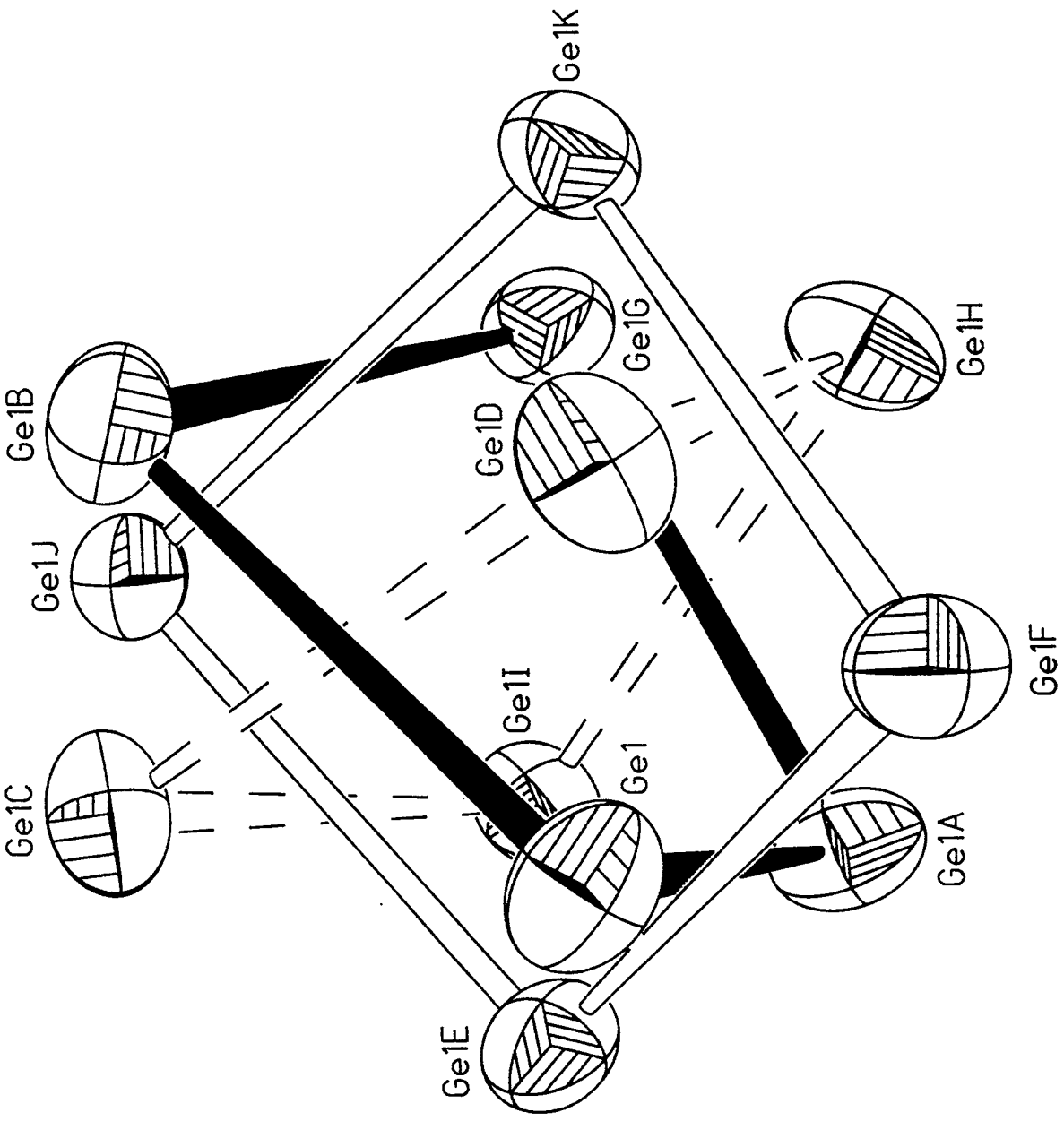

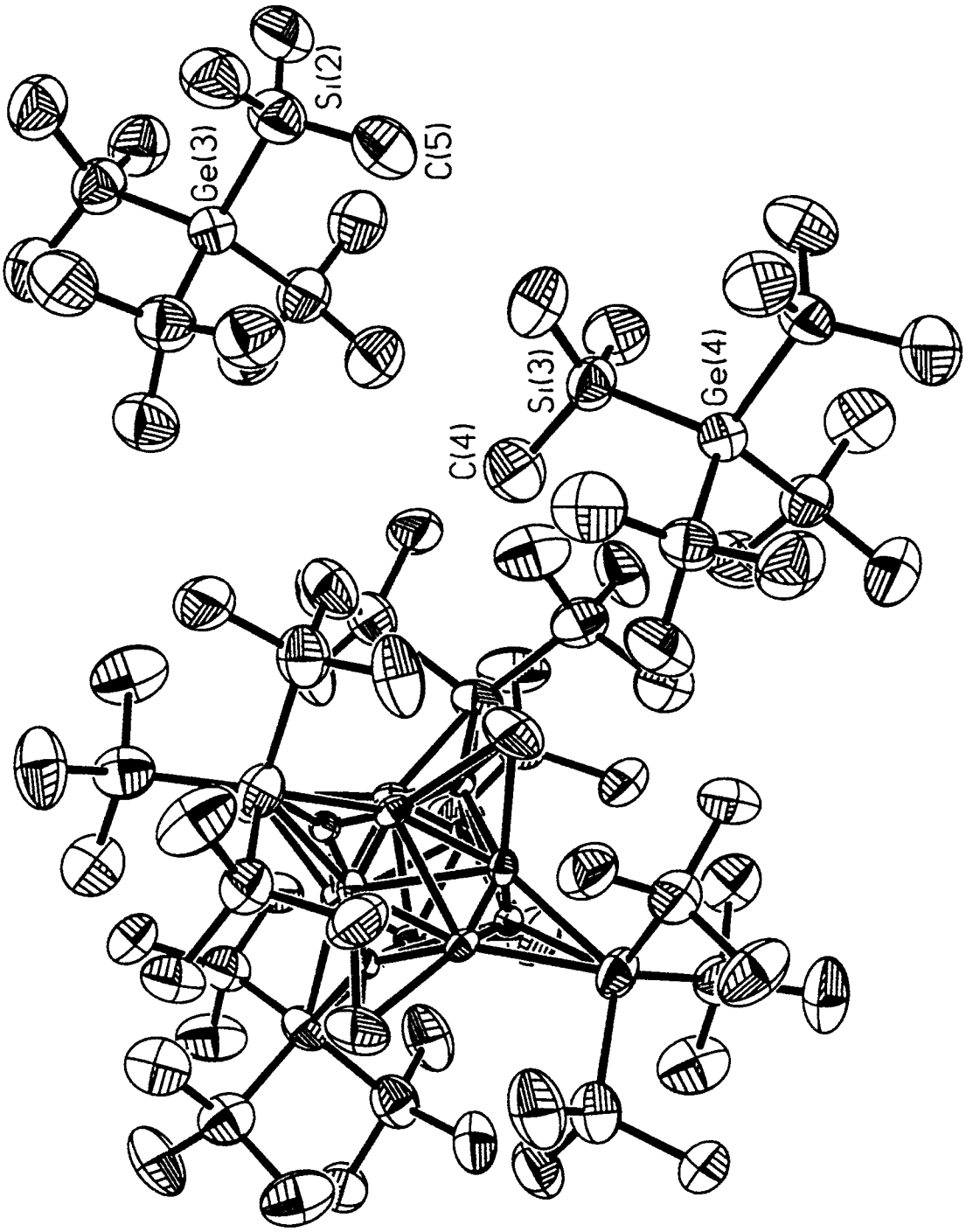
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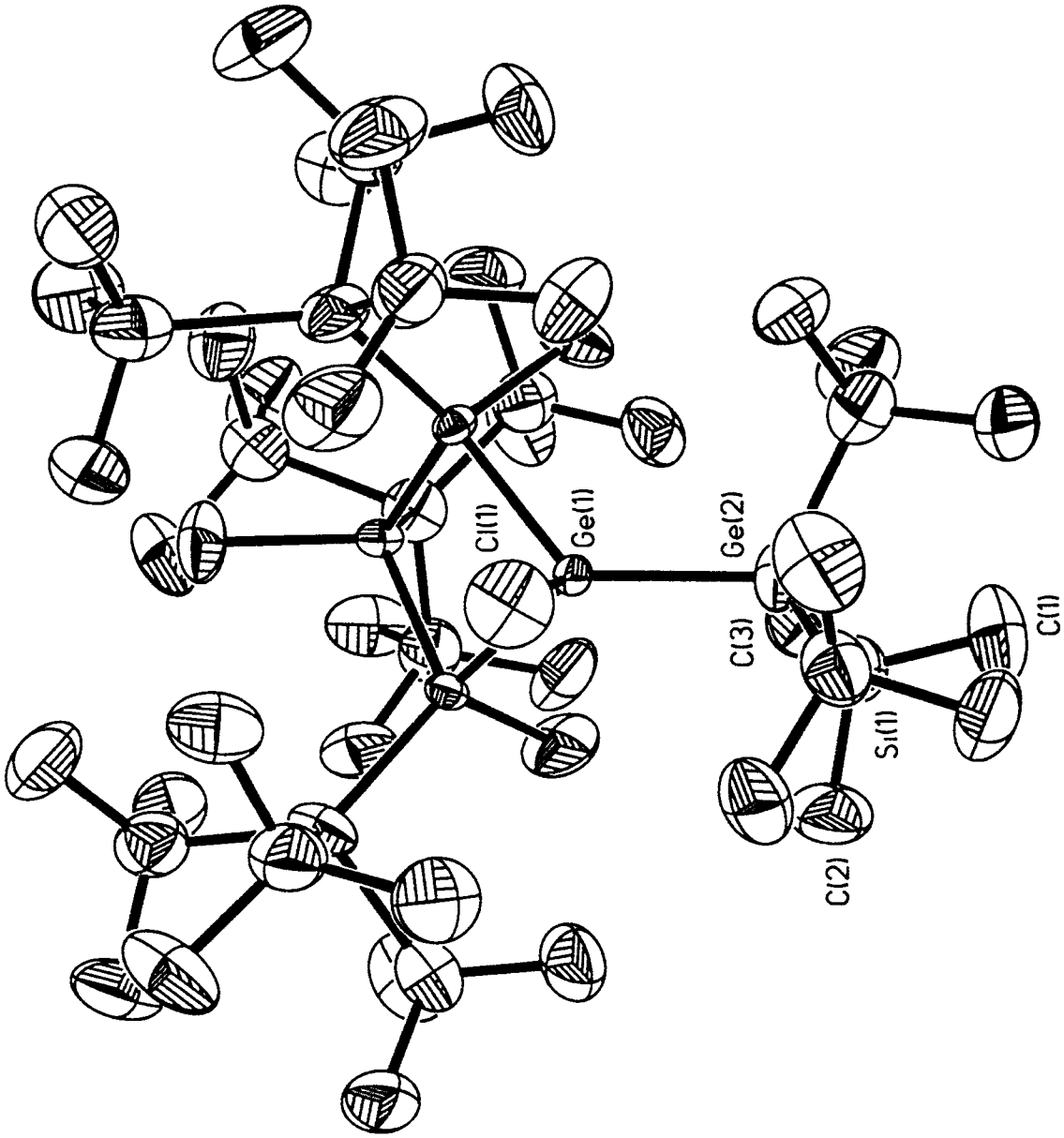
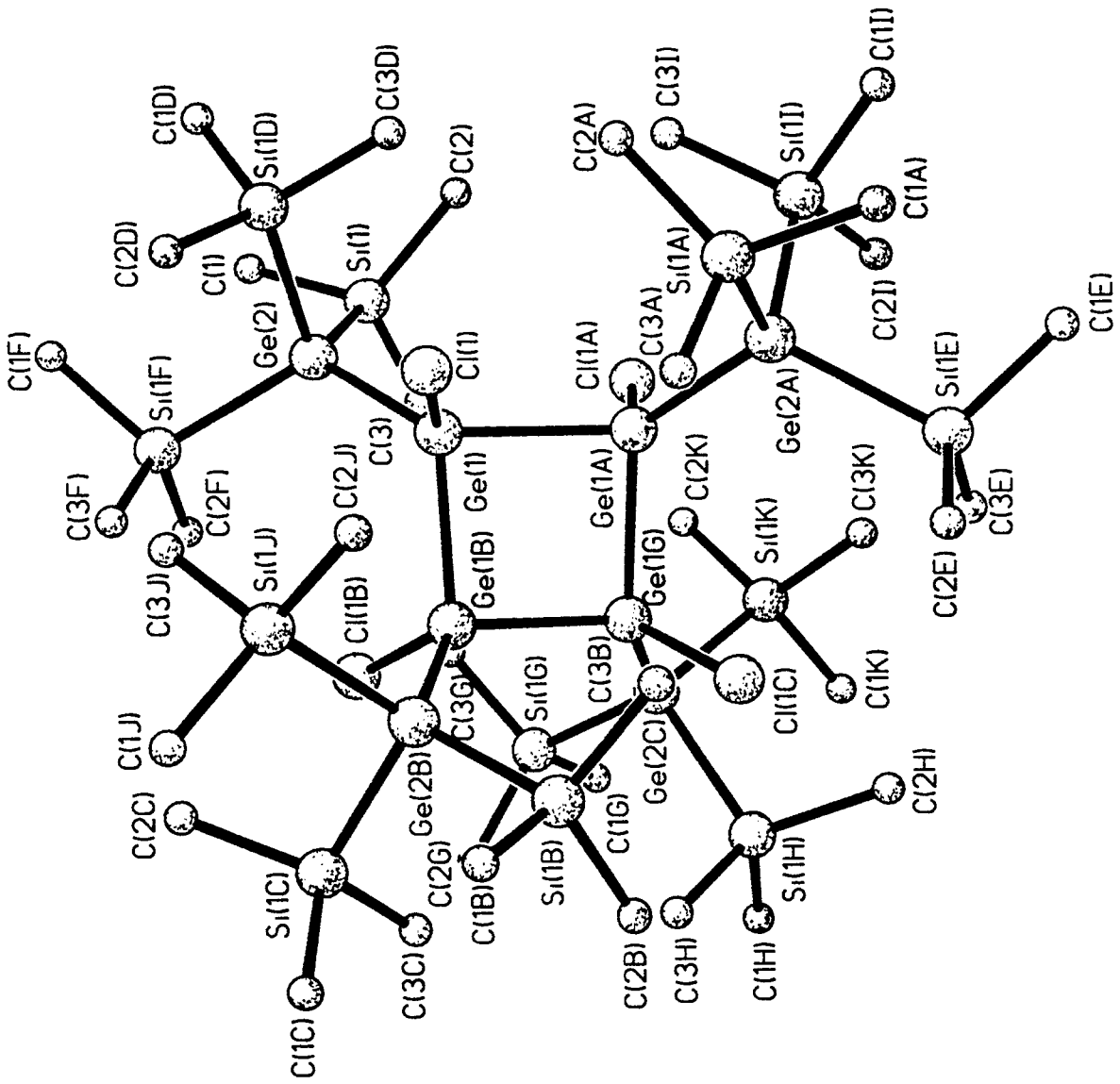
Ge(3)
C(5)
C(4)
Si(3)
Ge(4)

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C(3D)
C(2D)
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C(3F)
C(2F)
C(3)
Cl(1)
C(2J)
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C(3J)
Cl(1B)
Ge(1B)
C(1J)
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Si(1G)
C(2C)
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C(2G)
C(3C)
C(1B)
C(1C)
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C(1I)
C(1A)
Si(1A)
C(2I)
C(3A)
Ge(2A)
C(1E)
Si(1E)
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C(3E)