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Contribution from the Department of Chemistry,

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and

The Corporate Research Laboratories and

the Analytical Technology Division,

Eastman Kodak Company,

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Organometallic compounds of gallium with bonds to group 15 elements such as $R_3Ga\bullet ER'_3$[2] (E=N, P, As) and $(R_2GaER'_2)_2$[3] (E=P, As) have been receiving increasing attention due to their potential usefulness as precursors for the preparation of films of group 13-15 materials.[4] In contrast, similar types of compounds with bonds to the heavier group 16 elements have been the subject of only limited research. The early work of G. E. Coates[5] revealed that $GaMe_3$ reacted with EMe_2 (E=Se, Te) to form 1:1 adducts which were extensively dissociated in the vapor phase. Thus, the simple adduct $Me_3Ga\bullet TeMe_2$ is the only example of a reported compound containing a Ga-Te bond prior to the present work. When selenols[6] were used, elimination reactions occurred at room temperature and dimeric derivatives of the type $(Me_2GaER)_2$ (E=Se; R=Me, Ph) were formed. These dimers were stable to dissociation in the gas phase at moderate temperatures but the gallium-chalcogen bond was cleaved by NMe_3 to give monomeric species of the type $Me_2GaER\bullet NMe_3$.[6] The only other examples of derivatives containing Ga-Se bonds are $[Et_2GaSe(SiEt_3)]_2$,[7] $[i\text{-}Pr(Br)GaSeEt]_3$,[8] and $(EtGaSe)_n$.[7]

The first example of an organometallic gallium-tellurium compound $(Me_3CCH_2)_2GaTePh$, which is not a simple adduct, has been prepared by the reaction of $Ga(CH_2CMe_3)_2Cl$ with LiTePh in Et_2O. The new compound has been fully characterized by partial elemental analyses (C and H), physical

$$Ga(CH_2CMe_3)_2Cl + LiTePh \xrightarrow{Et_2O} (Me_3CCH_2)_2GaTePh + LiCl$$

properties, infrared spectroscopy, ^1H and ^{125}Te NMR spectroscopies and an X-ray structural study. The tellurium reagent[9] LiTePh was generated by the reductive cleavage of Te_2Ph_2 by $LiBEt_3H$ in THF/Et_2O. The use of a slight excess of Te_2Ph_2, which was readily removed by washing the LiTePh with pentane, insured that no $LiBEt_3H$ would be available to react with $Ga(CH_2CMe_3)_2Cl$ in the second step of the synthesis. The reagent $LiBEt_3H$ has been observed previously to ethylate $GeCl_4$.[10] The product $(Me_3CCH_2)_2GaTePh$ was purified by repetitive low temperature (-20 °C) crystallizations from pentane and was isolated in low yield (20%) as an off-white, extremely air-sensitive solid. An unidentified orange, oily product, which was shown to contain tellurium by ^{125}Te NMR spectroscopy, prevented the isolation of the above product in higher yield.

The structure of $(Me_3CCH_2)_2GaTePh$ consists of discrete dimeric units with the formula $[(Me_3CCH_2)_2GaTePh]_2$. There were no abnormally close contacts between dimeric units in the unit cell. The labelling of the atoms in the molecule is shown in Figure 1. The geometry of the Ga_2Te_2 butterfly ring is depicted in Figure 2. The interatomic distances and angles are collected in Tables I and II.

The Ga-Te bond distances in $[(Me_3CCH_2)_2GaTePh]_2$ are in the range of 2.7435(8) to 2.7623(8) Å. The average Ga-Te bond of 2.755 Å is 0.14 Å longer than the sum of the Ga and Te covalent radii, 2.62 Å.[11] Although no molecular compounds which contain Ga-Te bonds have been structurally characterized, Ga-Te bond distances have been reported for four GaTe based solid state phases. The average Ga-Te bond distances for the solid state phases GaTe (monoclinic),[12,13] GaTe (hexagonal)[14] and GaTeCl[15] are 2.665Å, 2.61Å and 2.634Å, respectively. The Ga_2Te_5 phase contains $GaTe_4$ tetrahedra with a Ga-Te bond distance of 2.641Å[3].

The four membered ring in $[(Me_3CCH_2)_2GaTePh]_2$ has a butterfly arrangement as found in $[I_2GaS(i\text{-}Pr)]_2$.[16] The angle of fold about the gallium atoms in $[(Me_3CCH_2)_2GaTePh]_2$ is 150.21° and about the tellurium atoms is 148.01°. The angle of fold about the gallium atoms in $[I_2GaS(i\text{-}Pr)]_2$ is 143.3(2)°.[16] Interestingly, the ring systems in the dimers $[Ph_2GaSEt]_2$[17,18] and $[I_2GaSMe]_2$[17,19] are planar. The phenyl groups in $[(Me_3CCH_2)_2GaTePh]_2$ are in the anti-position relative to the ring. The methyl groups in $[I_2GaSMe]_2$[17,19] are also in the anti-position relative to the planar ring as are the ethyl groups in $[Ph_2GaSEt]_2$.[17,18] However, $[I_2GaS(i\text{-}Pr)]_2$, which has a butterfly ring geometry, has the i-propyl groups in the syn-position.[16]

Each gallium and tellurium atom in $[(Me_3CCH_2)_2GaTePh]_2$ has a distorted tetrahedral coordination environment. The Te-Ga-Te angles are 91.92° and 92.12°. These angles are similar to the S-Ga-S angles of 94.3° in $[I_2GaSMe]_2$, 93.6° in $[I_2GaS(i\text{-}Pr)]_2$ and 89.1° in $[Ph_2GaSEt]_2$.[17] The Ga-Te-Ga angles in $[(Me_3CCH_2)_2GaTePh]_2$ are 83.51° and 83.04°. These angles are similar to the Ga-S-Ga angles of 86.4° in $[Ph_2GaSEt]_2$, 85.6° in $[I_2GaSMe]_2$ and 84.7° in $[I_2GaS(i\text{-}Pr)]_2$.[16]

The NMR spectral properties of the new gallium-tellurium compound are consistent with the presence of dimeric molecules $[(Me_3CCH_2)_2GaTePh]_2$ in solution. The ^{125}Te NMR spectrum, the most useful for identifying components, exhibits only one line at 345.4 ppm. For comparison, a solution of LiTePh in THF gave a line at 133.9 ppm whereas the unknown impurity which was an orange oil had a line at 370.1 ppm. The ^{1}H NMR spectrum in benzene-d_6 had a broadened line at 1.30 ppm for the methylene protons of the neopentyl group and at least six lines of decreasing intensity from 1.11 to 0.81 ppm for the methyl protons of the neopentyl group. The phenyl lines were obscured by those due to the solvent. The occurrence of multiple and/or broadened lines for the neopentyl group protons suggests isomers but their presence could not be confirmed. Since the X-ray structural study revealed only the trans isomer with the phenyl rings in the anti-position relative to the ring, the gallium-tellurium bonds in the dimer must have broken at least partially for isomerization to have occurred.

Experimental Section

All compounds described in this investigation were extremely sensitive to oxygen and moisture and were manipulated in a standard vacuum line or in a purified argon atmosphere. The alkyl gallium chloride, $Ga(CH_2CMe_3)_2Cl$ was prepared and purified by literature methods.[20a] The reagent $LiBEt_3H$ (1.0M in THF) was obtained from Aldrich Chemical Co. whereas Te_2Ph_2 was prepared as previously described.[20b] Solvents were dried by conventional procedures. Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY. Infrared spectra of Nujol mulls between CsI plates were recorded by means of a Perkin-Elmer 683 spectrometer. Absorption intensities are reported with the abbreviations vs (very strong), s (strong), m (medium), w (weak) and sh (shoulder). The ^{1}H NMR spectra were recorded at 90 MHz by using a Varian EM-390 spectrometer. Proton chemical shifts are reported in δ units (ppm) and are referenced to $SiMe_4$ at δ 0.00 and benzene as δ 7.13 ppm. The ^{125}Te NMR spectra were recorded at 94.70 MHz by using a Varian XLAA-300 spectrometer. The proton decoupled ^{125}Te NMR spectra are reported relative to diphenyl telluride ($TePh_2$) as δ = 688 ppm, which was referenced to external dimethyl telluride ($TeMe_2$) as δ = 0.00 ppm. The samples were run unlocked and the chemical shifts were measured by tube interchange. All samples for NMR spectra were contained in sealed NMR tubes. Melting points were observed in sealed capillaries.

Synthesis of $(Me_3CCH_2)_2GaTePh$.

In a glove box, a side-arm dumper was charged with 0.749 g of LiTePh (ca. 3 mmol) which had been prepared[9] from Te_2Ph_2 and $LiBEt_3H$ in THF/Et_2O and connected to a two-neck round-bottom flask charged with 0.871 g (3.52 mmol) of $Ga(CH_2CMe_3)_2Cl$. Diethyl ether (50 mL) was vacuum distilled into the reaction vessel and the contents were warmed to ambient temperature with stirring. The LiTePh was slowly added over a 5 min time period to the ether solution. The colorless solution slowly became yellow in color. The mixture was allowed to stir 16 h and the Et_2O was removed by vacuum distillation. An orange colored oil remained in the flask. The vessel was fitted with a medium frit and a Schlenk receiving flask in the glove box. Anhydrous pentane (20 mL) was distilled into the flask and repetitive low temperature crystallizations (-10 to -20 °C) provided 0.336 g (0.807 mmol, 22.9% yield) of an off-white solid which was identified as $(Me_3CCH_2)_2GaTePh$. Recrystallization of $(Me_3CCH_2)_2GaTePh$ from pentane at -20 °C afforded crystallographic quality crystals.

$(Me_3CCH_2)_2GaTePh$. mp 109.0-110.5 °C dec (sample begins to decompose and darken at 96 °C and melts to an orange liquid at 109.0-110.5 °C). ^1H NMR (C_6D_6, δ): 0.81, 0.89, 0.96, 1.01, 1.05, 1.11 (s, combined lines 18 H, $-CMe_3$); 1.30 (s, 3.7 H, $-CH_2-$). $^{125}Te\{^1H\}$ (C_6D_6, δ): 345.4 (s). The ^{125}Te NMR spectrum of the orange oil from the reaction gave a resonance in the $^{125}Te\{^1H\}$ NMR spectrum at 370.1 ppm. The ^{125}Te NMR spectrum of a sample of LiTePh in THF gave a resonance in the $^{125}Te\{^1H\}$ NMR spectrum at 133.9 ppm. Anal. Calcd. for $C_{32}H_{54}Ga_2Te_2$: C, 46.12; H, 6.53. Found: C, 46.41, H, 6.63. IR (Nujol, cm^{-1}): 3070 (vw), 3055 (vw), 1571 (m), 1430 (m), 1358 (m), 1232 (m), 1133 (m), 1099 (m), 1062 (vw), 1055 (vw), 1011 (m), 999 (w,sh), 994 (m), 795 (vw,br), 740 (w), 720 (s), 648 (w), 618 (m), 449 (w).

Crystallography. A colorless crystal of $(Me_3CCH_2)_2GaTePh$ was selected and sealed in a glass capillary under an argon atmosphere for examination by X-ray diffraction procedures. Data were collected on an Enraf-Nonius CAD-4 diffractometer by using graphite monochromated Mo Kα radiation. Table III summarizes the unit cell data and the refinement parameters. Unit cell dimensions were determined by least-squares refinement of 25 reflections ($20.0° < 2\theta < 26.8°$). Three standard reflections were remeasured at every hour of X-ray exposure. The data were corrected for a slow, steady decrease of intensity over time. The maximum correction was 1.230 on I. An empirical absorption correction[21] was applied. Correction factors ranged from 0.85 to 1.31.

The structure of $(Me_3CCH_2)_2GaTePh$ was solved by direct methods by using MULTAN 11/82.[22] An E-map phased with the best phase set gave the four heavy atom positions. Subsequent difference electron density maps allowed location of the remaining atoms. All hydrogen atoms were included in the refinement except that those on methyl groups were fixed at calculated positions riding on the parent carbon atoms. Positional parameters, excluding hydrogen atoms, are given in Table IV.

Acknowledgment. This work was supported in part by the Office of Naval Research and by a generous grant from Eastman Kodak Company.

Supplementary Material Available. Table of refined temperature factors for $[(Me_3CCH_2)_2GaTePh]_2$ (2 pages); table of hydrogen atom positional parameters (3 pages); tables of carbon-carbon bond distances and angles (2 pages); structure factors (29 pages). Ordering information is given on any current masthead page.

References

1. a) State University of New York at Buffalo

   b) Corporate Research Laboratories, Eastman Kodak Company

   c) Analytical Technology Division, Eastman Kodak Company

2. Moss, R. H. J. Crystal Growth 1984, 68, 78.

3. Cowley, A. H.; Benac, B. L.; Ekerdt, J. G.; Jones, R. A.; Kidd, K. B.; Lee, J. Y.; Miller, J. E. J. Am. Chem. Soc. 1988, 110, 6248.

4. Cowley, A. H.; Jones, R. A. Angew. Chem. Int. Ed. 1989, 28, 1208.

5. Coates, G. E. J. Chem. Soc. 1951, 2003.

6. Coates, G. E.; Hayter, R. G. J. Chem. Soc. 1953, 2519.

7. Vyazankin, N. S.; Bochkarev, M. N.; Charov, A. I. J. Organomet. Chem. 1971, 27, 175.

8. Hoffmann, G. G.; Fischer, R. Inorg. Chem. 1989, 28, 4165.

9. Rauchfuss, T. B.; Weatherill, T. D. Inorg. Chem. 1982, 21, 827.

10. Gysling, H. J.; Luss, H. R. Organometallics 1989, 8, 363.

11. Pauling, L. The Nature of the Chemical Bond, 3rd ed., Cornell University Press, Ithaca, NY, 1960.

12. Julien-Pouzol, M.; Jaulmes, S.; Guittard, M.; Alapini, F. Acta Cryst. 1979, 35B, 2848.

13. Alapini, F.; Flahaut, J.; Guittard, M.; Jaulmes, S.; Julien-Pouzol, M. J. Solid State Chem. 1979, 28, 309.

14. Semiletov, S. A.; Vlasov, V. A. Kristallografiya 1963, 8, 877.

15. Wilms, A.; Kniep, R. Z. Naturforsch. 1981, 36B, 1658.

16. Hoffmann, G. G.; Burschka, C. Angew. Chem. Int. Ed. 1985, 24, 970.

17. Hoffmann, G. G. Phosphorus and Sulfur 1986, 28, 167.

18. Hoffmann, G. G.; Burschka, C. J. Organomet. Chem. 1984, 267, 229.

19. Boardman, A.; Jeffs, S. E.; Small, R. W. H.; Worrall, I. J. Inorg. Chem. Acta 1985, 99, L39.

20. a) Beachley, O. T., Jr.; Pazik, J. C. Organometallics 1988, 7, 1516.

b) Haller, W. S.; Irgolic, K. J. J. Organometal. Chem. 1972, 38, 97.

21. Walker, N.; Stuart, D. Acta Cryst. 1983, A39, 159.

22. Programs used in this study were from the Structure Determination Package, SDP-PLUS, V3.0 (1985). Enraf-Nonius Corp., Delft, Holland.

Table I. Interatomic Distances (Å) for $[(Me_3CCH_2)_2GaTePh]_2$.

(A) Gallium-Tellurium Distances

Ga(1)-Te(1)	2.7623(8)	Ga(2)-Te(2)	2.7525(8)
Ga(1)-Te(2)	2.7435(8)	Ga(2)-Te(1)	2.7623(8)

(B) Gallium-Carbon Distances

Ga(1)-C(7)	1.975(7)	Ga(2)-C(23)	1.988(7)
Ga(1)-C(12)	1.962(8)	Ga(2)-C(28)	1.979(8)

(C) Tellurium-Carbon Distances

Te(1)-C(1)	2.125(6)	Te(2)-C(17)	2.125(6)

Table II. Angles (in deg) for $[(Me_3CCH_2)_2GaTePh]_2$.

(A) Angles around the Gallium Atom

Te(1)-Ga(1)-Te(2)	92.1(2)	Te(1)-Ga(2)-Te(2)	91.92(2)
Te(1)-Ga(1)-C(7)	103.5(2)	Te(1)-Ga(2)-C(23)	104.4(2)
Te(1)-Ga(1)-C(12)	111.6(4)	Te(2)-Ga(2)-C(28)	114.2(3)
Te(2)-Ga(1)-C(7)	108.9(3)	Te(1)-Ga(2)-C(23)	103.7(3)
Te(2)-Ga(1)-C(12)	103.7(3)	Te(1)-Ga(2)-C(28)	105.8(2)
C(7)-Ga(1)-C(12)	130.4(4)	C(23)-Ga(2)-C(28)	129.7(4)

(B) Angles around the Tellurium Atom

Ga(1)-Te(1)-Ga(2)	83.51(2)	Ga(1)-Te(2)-Ga(2)	84.04(2)
Ga(1)-Te(1)-C(1)	102.6(2)	Ga(1)-Te(2)-C(17)	99.1(2)
Ga(2)-Te(1)-C(1)	106.8(2)	Ga(2)-Te(2)-C(17)	103.8(2)

(C) Gallium-Carbon-Carbon Angles

Ga(1)-C(7)-C(8)	121.6(5)	Ga(2)-C(23)-C(24)	121.3(6)
Ga(1)-C(12)-C(13)	123.1(6)	Ga(2)-C(28)-C(29)	122.7(6)

(D) Tellurium-Carbon-Carbon Angles

Te(1)-C(1)-C(2)	121.1(5)	Te(2)-C(17)-C(18)	122.7(5)
Te(1)-C(1)-C(6)	119.4(5)	Te(2)-C(17)-C(21)	118.2(6)

Table III. Summary of Crystal Data and Refinement Parameters

Formula		$(GaTeC_{16}H_{27})_2$
M.W.		833.428
Crystal system		Monoclinic
Space group		$P2_1/C$
Cell constants at 23(1)°C:	a, Å	18.864(7)
	b	10.041(5)
	c	19.869(9)
	β, deg	101.80(3)
	V, $Å^3$	3684(5)
No. molecules/unit cell (Z)		4
D(calc), g cm^{-3}		1.505
Crystal dimensions, mm		0.18 x 0.25 x 0.38
Absorption coeff. (μ, Mo Kα) cm^{-1}		30.4
Scan technique		ω-2θ
Scan rate, deg 2θ min^{-1}		2.1 to 20
2θ limit, deg		46
hkl range		-20 to +20, 0 to 11, 0 to 21
No. of unique data measured		5100
No. of data used in refinement (I>σ(I))		3954
No. of parameters		397
$R = \Sigma\|\|F_o\| - K\|F_c\|\|/\Sigma\|F_o\|$		0.030
$R_w = (\Sigma w(\|F_o\| - K\|F_c\|)^2/\Sigma wF_o^2)^{1/2}$		0.035
$S = [(\Sigma w(\|F_o\| - K\|F_c\|)^2/(n_o-n_v)]^{1/2}$		0.94
Wtg. parameters ($w^{-1} = \sigma^2(F_o) + (pF_o)^2 + q$):	p	0.02
	q	1.0
Scale factor, K		0.6497(2)
Maximum shift in final cycle, (Δ/σ)		0.03
Residual electron density in final difference Fourier synthesis (e/$Å^3$)		-0.33 to +0.44

Table IV. Positional Parameters (Esd's in Parentheses) for $[(Me_3CCH_2)_2GaTePh]_2$[a].

Atom	x	y	z	B(Å^2)
TE1	0.23526(2)	0.46017(4)	0.36194(2)	4.466(8)
TE2	0.19798(2)	0.76885(4)	0.23521(2)	4.453(8)
GA1	0.21570(3)	0.73101(6)	0.37440(3)	4.23(1)
GA2	0.27140(3)	0.53118(6)	0.23890(3)	4.51(1)
C1	0.3341(3)	0.4253(5)	0.4331(3)	4.5(1)
C2	0.3915(4)	0.3614(6)	0.4131(3)	5.9(2)
C3	0.4536(4)	0.3314(7)	0.4628(4)	6.9(2)
C4	0.4585(4)	0.3626(7)	0.5299(4)	7.5(2)
C5	0.4015(4)	0.4245(8)	0.5486(4)	7.6(2)
C6	0.3391(4)	0.4567(6)	0.5012(3)	6.0(2)
C7	0.3127(3)	0.7953(6)	0.4196(3)	5.2(1)
C8	0.3333(3)	0.9419(6)	0.4185(3)	5.2(1)
C9	0.2822(5)	1.0242(8)	0.4459(7)	17.2(4)
C10	0.4083(5)	0.9634(8)	0.4601(5)	11.5(3)
C11	0.3362(6)	0.9864(9)	0.3475(5)	15.9(3)
C12	0.1199(3)	0.7718(8)	0.3933(3)	6.8(2)
C13	0.0990(3)	0.7417(6)	0.4618(3)	5.7(2)
C14	0.0880(7)	0.604(1)	0.4688(6)	25.1(4)
C15	0.0313(5)	0.807(2)	0.4689(5)	20.1(5)
C16	0.1528(6)	0.783(2)	0.5171(5)	24.7(6)
C17	0.0874(3)	0.7130(6)	0.2050(3)	4.8(1)
C18	0.0615(3)	0.5929(6)	0.2226(4)	6.1(2)
C19	-0.0115(4)	0.5641(8)	0.2035(4)	7.9(2)

C20	-0.0573(4)	0.6533(9)	0.1660(4)	8.7(2)
C21	-0.0322(4)	0.7726(8)	0.1474(5)	8.4(2)
C22	0.0401(3)	0.8023(7)	0.1673(4)	6.3(2)
C23	0.3749(3)	0.5842(7)	0.2647(3)	5.9(2)
C24	0.4163(3)	0.6338(7)	0.2106(4)	6.6(2)
C25	0.4936(4)	0.669(1)	0.2451(5)	12.3(3)
C26	0.4235(5)	0.517(1)	0.1624(5)	11.2(3)
C27	0.3777(5)	0.7415(9)	0.1699(5)	15.5(3)
C28	0.2202(4)	0.4281(6)	0.1586(3)	5.8(2)
C29	0.2182(4)	0.2763(6)	0.1577(3)	6.2(2)
C30	0.2924(5)	0.2199(8)	0.1877(5)	9.9(3)
C31	0.1643(5)	0.2253(8)	0.1994(4)	9.5(2)
C32	0.1925(5)	0.2272(8)	0.0834(4)	9.4(2)

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

Captions for Figures

Figure 1. ORTEP Plot of $[(Me_3CCH_2)_2GaTePh]_2$. The thermal ellipsoids were drawn at the 40% probability level.

Figure 2. Geometry of Ga_2Te_2 Ring in $[(Me_3CCH_2)_2GaTePh]_2$.

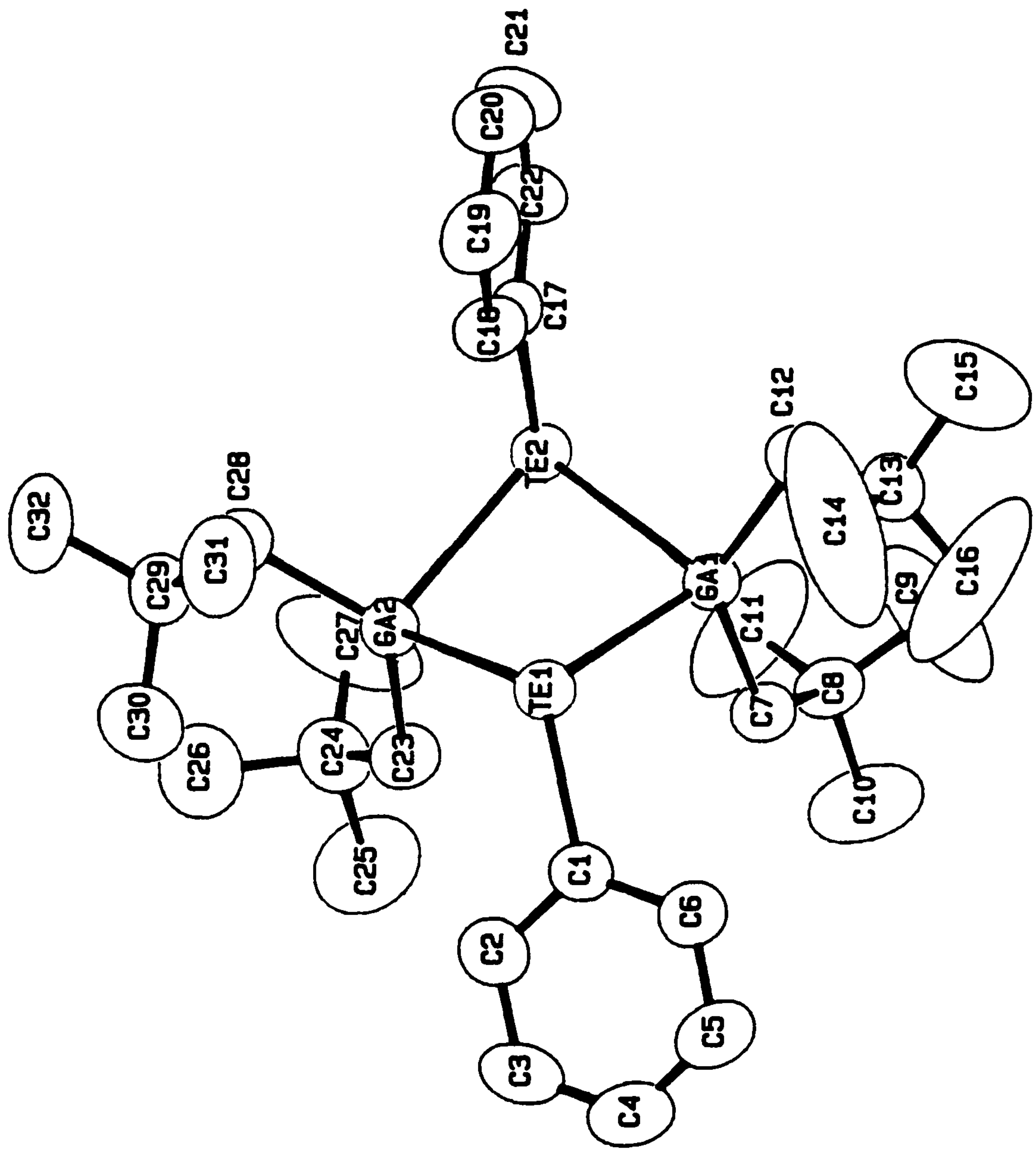
TE1
TE2
GA1
GA2
C1
C2
C3
C4
C5
C6
C7
C8
C9
C10
C11
C12
C13
C14
C15
C16
C17
C18
C19
C20
C21
C22
C23
C24
C25
C26
C27
C28
C29
C30
C31
C32

Fig 1

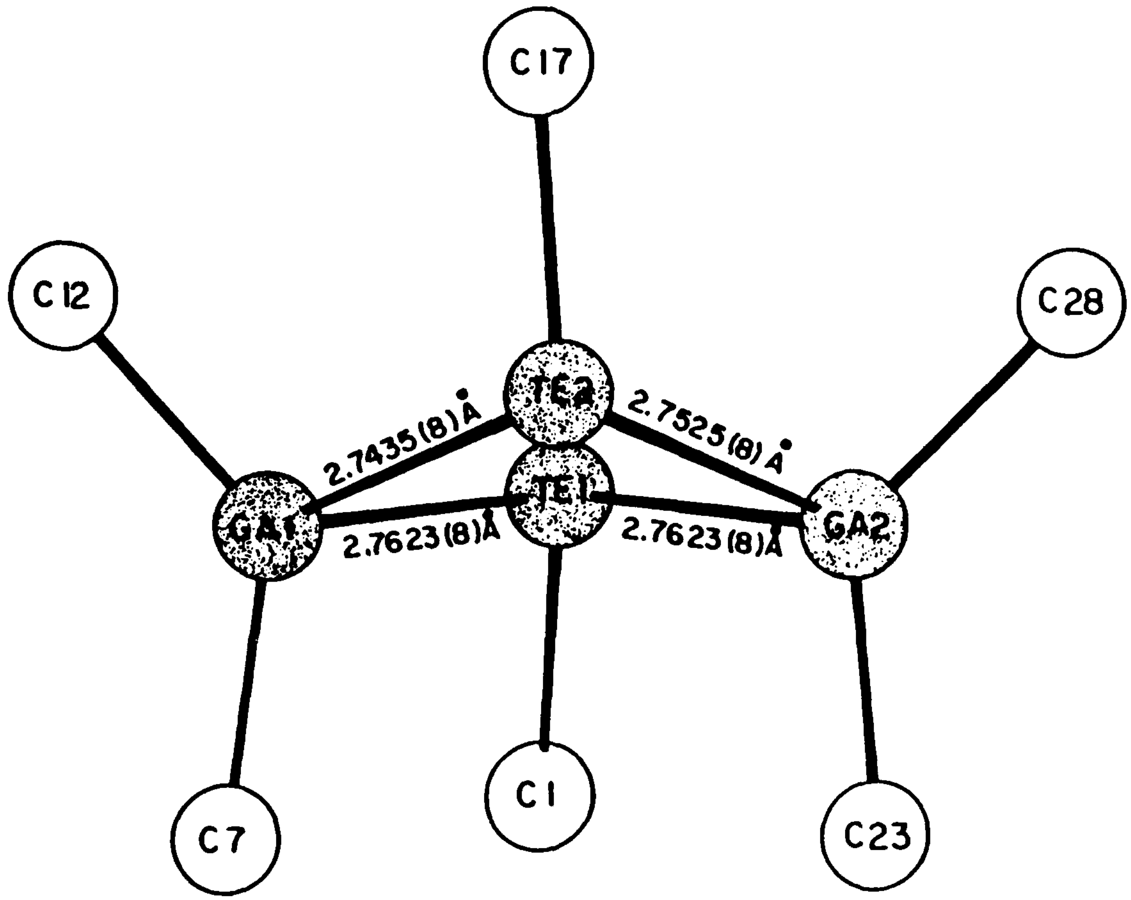
C17
C12
C28
TE2
TE1
GA1
GA2
2.7435(8)Å
2.7525(8)Å
2.7623(8)Å
2.7623(8)Å
C7
C1
C23

Fig 2