*Acta Cryst.* (1972). B**28**, 2889

# The Crystal Structure of Tetraphenylgermanium

By Anastas Karipides and David A. Haller*

*Department of Chemistry, Miami University, Oxford, Ohio* 45056, *U.S.A.*

(*Received* 12 *October* 1971 *and in revised form* 10 *January* 1972)

The crystal structure of tetraphenylgermanium has been determined and refined by full-matrix least-squares methods based on 546 reflections recorded on a Picker FACS-1 automated diffractometer. The final $R$ index is 0·026. The crystals are tetragonal, space group $P\bar{4}2_1c$, with cell dimensions $a = 11·656$ (11) $c = 6·928$ (7) Å. There are two molecules in the unit cell; the germanium atoms lie on $\bar{4}$ axes and there is only one independent phenyl ring in the asymmetric unit. The entire molecule is rotated by 7·2° from the $a$ axis and the plane of the phenyl ring is rotated 54·1° (measured clockwise) from the C–Ge–C plane. The observed conformation is different from that calculated by Ismailzade but is in agreement with a more thorough analysis of non-bonded hydrogen–hydrogen interactions.

## Introduction

As part of a general study on the structure and packing of aryl organometallic compounds we have determined the crystal structure of tetraphenylgermanium. Comparison is made with the structure predicted from a geometrical analysis (Ismailzade, 1952).

## Experimental

Crystals of tetraphenylgermanium, $(C_6H_5)_4Ge$, were grown from a benzene solution by slow evaporation at room temperature. The space group was uniquely determined from indexed Weissenberg photographs from a crystal mounted along the $c$ axis. Accurate values of the cell dimensions were obtained by the least-squares refinement of the angular settings of twelve reflections carefully aligned on a Picker FACS-1 diffractometer. The experimental density was measured by flotation in an aqueous potassium iodide solution. The crystal data are summarized in Table 1.

Table 1. *Crystal data for tetraphenylgermanium*

$(C_6H_5)_4Ge$
$Z=2$
Tetragonal
$a=11·656$ (11) Å
$c=6·928$ (7)
(Mo $K\alpha=0·71069$ Å)
$\mu=17·2$ cm$^{-1}$
$d_o=1·31$ g.cm$^{-3}$

M. W. 381·0
$F(000)=392$
Systematic absences:
$h00$: $h$ odd
$hhl$: $l$ odd

Space group $P\bar{4}2_1c$
$d_c=1·34$ g.cm$^{-3}$

Three-dimensional intensity data were collected on a Picker FACS-1 diffractometer equipped with a scintillation counter and a pulse height analyser; zirconium-filtered Mo $K\alpha$ radiation was used. The crystal had dimensions 0·21 × 0·25 × 0·22 mm and was mounted along the longer ($c$) dimension. All reflections in the range 0–55° in $2\theta$ were collected at room temperature using the $\theta$–$2\theta$ scan mode. A scan range of −1 to +1° of the calculated $2\theta$ value of each reflection plus an allowance for the Mo $K(\alpha_1-\alpha_2)$ separation for each reflection was used. The scan rate was 1° per min. A stationary background count was measured for 40 sec on each side of the diffraction peak. A brass attenuator was placed in front of the detector when the count rate exceeded 10,000 counts per sec. The attenuator factor was 8·02. Three standard reflections (040, 321, 202) were monitored every 100 reflections and their intensities indicated no counter or crystal instability throughout the data collection.

Each intensity $I$ was corrected for Lorentz and polarization factors, but not for absorption, to give the observed structure factor amplitudes, $F_o$. From the 684 experimentally determined reflections 55 were discarded because they had a 'negative' intensity. An additional 83 reflections were rejected on the basis of $F_o < \sigma(F_o)$ where $\sigma(F_o)$ is the standard deviation computed from:

$$\sigma^2(|F_o|) = (I + 0·25k^2B/|F_o|^2\mathrm{Lp}^2)$$

and $I$ is the total integrated count from the scanning, $R$ is the ratio of scan time to total background counting time, $B$ is the total background count, and Lp is the Lorentz–polarization correction.

## Structure determination and refinement

There are two germanium atoms per unit cell which are fixed by the space-group symmetry to be at special positions with $\bar{4}$ symmetry. A three-dimensional Patterson map clearly located the germanium atoms at 000 and $\frac{1}{2}\frac{1}{2}\frac{1}{2}$. There is only one phenyl ring in the asymmetric unit and the six independent carbon atoms were found from a three-dimensional electron density map phased by the germanium atom. Full-matrix least-squares refinement with individual isotropic temperature factors led to a conventional $R$ index, $\sum(|F_o - F_c|)/\sum F_o$, of 0·082. The hydrogen atoms representing about 10% of the total electron density in the compound were than included in the following

---

* Based in part on the Master of Science thesis of David Haller, Miami University, 1971.

manner. The positions of the five independent hydrogen atoms were calculated assuming the C–H bond bisects the calculated C–C–C angle. The C–H length was fixed at 1·08 Å. These hydrogen atoms were given the final refined isotropic temperature factors of the carbon atoms to which they were attached.

Full-matrix least-squares refinement was resumed assuming anisotropic motion for the germanium and carbon atoms. The hydrogen atom positions were not varied. A total of 64 parameters including one scale factor were varied. The quantity minimized was $\sum w(|F_o|-|F_c|)^2$ where the weights, $w$, were taken as unity. New hydrogen positions were calculated after each least-squares run and, after four cycles, the structure refinement converged with a conventional $R$ index of 0·026 and a weighted $wR$ index $\{wR=[\sum w(|F_o|-|F_c|)^2/\sum wF_o^2]^{1/2}\}$ of 0·030. A final difference Fourier map showed no electron density maximum greater than 0·18 e.Å$^{-3}$.

The atomic form factors for germanium and carbon were taken from Cromer & Waber (1968) and for hydrogen from *International Tables for X-ray Crystallography* (1968). All computations were performed on an IBM 360/50 computer with 256K byte core. Patterson and electron density maps were computed with the A. Zalkin *FORDAP* program. The Busing Martin & Levy (1964) full-matrix least-squares program (*ORFLS*) was used for the structure refinement and their function and error program (*ORFFE*) was used for calculations of bond distances, angles and their respective standard deviations. The final atomic positional and thermal parameters and their estimated standard deviations are given in Table 2. Observed and calculated structure factors are given in Table 3.

## Results and discussion

Tetraphenylgermanium (Fig. 1) has $\bar{4}$ symmetry in the crystalline state; hence there is only one independent phenyl ring in the unit cell and it is convenient to describe the structure in terms of two angular parameters of the tetraphenylgermanium molecule in this space group. These are:

(1) $\varphi$, the angle between the projection of the Ge–C(1) bond on the $xy$ plane and the $a$ axis. This defines the rotation of the entire molecule about the $c$ axis.

(2) $\alpha$, the angle between the phenyl ring plane and the C(1) ($\bar{x}$, $\bar{y}$, $z$)–Ge–C(1) plane. A clockwise rotation is taken as positive looking from the phenyl ring to the germanium atom. This defines the rotation of the phenyl ring about the Ge–C(1) bond.

For tetraphenylgermanium the angles $\varphi$ and $\alpha$ were found to be 7·2 and 54·1° respectively.

Tetraphenylgermanium was first examined by George (1927), who determined cell dimensions and space group but did little other work. Ismailzade (1952) has reported a geometrical analysis on tetraphenylgermanium where he attempted to arrive at $\varphi$ and $\alpha$ by considering the closest approach of phenyl hydrogen atoms. No experimental measurements were made by Ismailzade on $Ge(C_6H_5)_4$. Similar efforts by Ismailzade & Zdanov (1952) were carried out on the tetraphenyl derivatives of silicon, tin and lead, although a recent crystal structure determination of tetraphenyllead (Busetti, Mammi, Signor & Del Pra, 1967) has shown Ismailzade's geometrical analysis of the lead compound to be in error. The results from Ismailzade's (1952) geometrical analysis on tetraphenylgermanium are $\varphi=7$ and $\alpha=37{\cdot}5°$. Note that Ismailzade's indicated $\psi=90-\alpha$. Also we note that the reported calculated coordinates of Ismailzade (1952) are incorrectly coupled. As pointed out by Busetti *et al.* (1967), Ismailzade's (1952) coordinates make sense only if the respective $z$ coordinates of the pairs of atoms C(2), C(6) and C(3), C(5) are reversed.

The wide difference between the value of $\alpha$ obtained from the three-dimensional crystal structure determination (54·1°) and Ismailzade's geometrical analysis (35·5°) is quite surprising. We have re-examined the tetraphenylgermanium structure from the point of

Table 2. *Final parameters and their standard deviations* (× 10$^4$)

The anisotropic coefficients for the heavier atoms are of the form:

$$\exp\left[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta hl+2\beta_{23}kl)\right]$$

and have been multiplied by 10$^4$.
For hydrogen atoms, isotropic $B$'s are given.

| | $x/a$ | $y/b$ | $z/c$ | $\beta_{11}$ | $\beta_{22}$ | $\beta_{33}$ | $\beta_{12}$ | $\beta_{13}$ | $\beta_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Ge | 0 | 0 | 0 | 49 (1) | 49 (1) | 216 (1) | 0 | 0 | 0 |
| C(1) | 1356 (3) | 171 (3) | 1640 (6) | 53 (3) | 57 (3) | 206 (8) | −4 (2) | −0 (4) | 7 (5) |
| C(2) | 1386 (4) | 1019 (4) | 3065 (6) | 80 (3) | 71 (3) | 227 (9) | 4 (3) | 6 (5) | −5 (5) |
| C(3) | 2350 (4) | 1161 (4) | 4236 (6) | 106 (4) | 80 (4) | 207 (9) | −19 (3) | −12 (6) | −11 (5) |
| C(4) | 3295 (4) | 448 (4) | 4004 (7) | 80 (4) | 106 (5) | 238 (11) | −20 (3) | −22 (6) | 29 (6) |
| C(5) | 3272 (4) | −407 (4) | 2626 (7) | 65 (3) | 109 (5) | 268 (11) | 9 (3) | −3 (6) | 18 (5) |
| C(6) | 2309 (4) | −543 (3) | 1433 (6) | 66 (3) | 72 (3) | 237 (10) | 8 (3) | −3 (5) | −7 (5) |
| H(2) | 652 | 1573 | 3264 | 4·1 (2) | | | | | |
| H(3) | 2362 | 1826 | 5322 | 4·5 (2) | | | | | |
| H(4) | 4045 | 562 | 4900 | 4·9 (2) | | | | | |
| H(5) | 3999 | −973 | 2465 | 4·8 (2) | | | | | |
| H(6) | 2304 | −1207 | 345 | 4·2 (2) | | | | | |

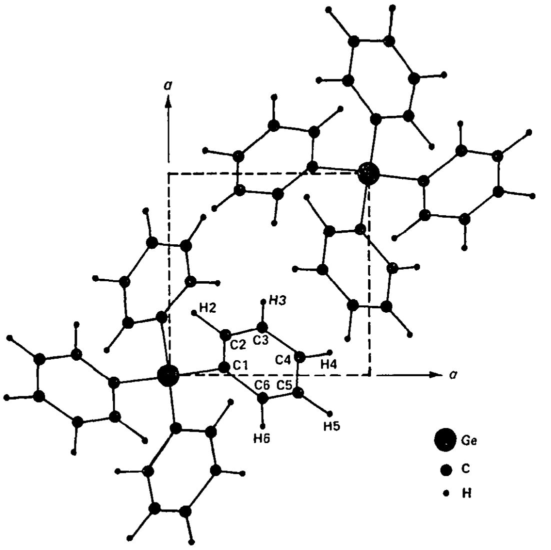

Fig. 1. Tetraphenylgermanium projection in *xy* plane.

view of closest approach of phenyl hydrogen atoms and, in contrast to Ismailzade's results, found there is excellent agreement with the conformation found from the X-ray structure determination.

The first nearest neighbor contacts are those involving the hydrogen atoms on the phenyl rings. The cumulative effect of all non-bonded van der Waals interactions will, of course, determine the structure and hence the angle $\alpha$. The critical requirement is that at the equilibrium conformation, there must be at least one $H\cdots H$ non-bonded intermolecular contact of 2·37–2·40 Å (Bondi, 1964) and there cannot be, obviously, any $H\cdots H$ contacts significantly less than 2·37 Å. In our analysis, the variation of all possible non-bonded inter- and intramolecular $H\cdots H$ contact distances were calculated as a function of the angle $\alpha$ using a standard phenyl ring geometry, C–C = 1·395 Å, C–H = 1·08 Å, a Ge–C distance of 1·957 Å and $\varphi=7{\cdot}2°$. From the results six $H\cdot\cdot H$ contacts were found which could be used to determine the structure. The variation of these six intermolecular $H\cdots H$ contacts with $\alpha$ are given in Fig. 2. At the stable conformation there cannot be any $H\cdots H$ contacts less than 2·37 Å and from Fig. 2 we can see that at all $\alpha$ angles except 49 and 54° there is at least one $H\cdots H$ intermolecular distance less than 2·37 Å. All these conformations can thus be ruled out. The conformations between 49 and 54° can also be excluded since all $H\cdots H$ contacts in this region are substantially greater than 2·37 Å. Only at two angles, $\alpha=54$ and $\alpha=49°$, are there $H\cdots H$ contacts of 2·37 Å and none less than this critical value. This result is in excellent agreement with the $\alpha=54{\cdot}1°$ found from our X-ray structure determination. The conformation at $\alpha=49°$ must be energetically less stable. The fact that two rather close conformations are allowed by the simple consideration of van der Waals contacts suggests a limitation of this method of analysis alone. The crystal lattice (packing) energy is a more sensitive probe in predicting a probable structure.

The critical $H\cdots H$ contacts are intermolecular and not intramolecular, since it is the intermolecular inter-

Table 3. *Observed and calculated structure factors* ($\times 10^2$)

| H | K | FO | FC |
|---|---|---|---|
| | L = 0 | | |
| 0 | 2 | 6912 | 6786 |
| 0 | 4 | 3492 | 3539 |
| 0 | 6 | 4171 | 4135 |
| 0 | 8 | 4060 | 4044 |
| 0 | 12 | 1325 | 1351 |
| 0 | 10 | 1275 | 1210 |
| 0 | 14 | 768 | 786 |
| 1 | 2 | 975 | 859 |
| 1 | 3 | 4433 | 4453 |
| 1 | 5 | 3215 | 3204 |
| 1 | 6 | 1032 | 1005 |
| 1 | 7 | 4100 | 4032 |
| 1 | 8 | 650 | 651 |
| 1 | 9 | 4729 | 4787 |
| 1 | 10 | 434 | 429 |
| 1 | 11 | 778 | 788 |
| 1 | 12 | 782 | 802 |
| 1 | 14 | 327 | 287 |
| 1 | 15 | 1103 | 1111 |
| 2 | 2 | 1503 | 1217 |
| 2 | 3 | 1209 | 1138 |
| 2 | 4 | 3628 | 3494 |
| 2 | 5 | 728 | 681 |
| 2 | 6 | 5740 | 5804 |
| 2 | 8 | 3497 | 3500 |
| 2 | 9 | 347 | 335 |
| 2 | 10 | 1902 | 1858 |
| 2 | 11 | 752 | 750 |
| 2 | 12 | 2100 | 2147 |
| 2 | 13 | 557 | 640 |
| 2 | 14 | 1142 | 1200 |
| 3 | 3 | 7168 | 7193 |
| 3 | 4 | 1830 | 1709 |
| 3 | 5 | 3719 | 3726 |
| 3 | 6 | 2515 | 2443 |
| 3 | 7 | 2579 | 2893 |
| 3 | 9 | 2157 | 2158 |
| 3 | 11 | 647 | 725 |
| 3 | 12 | 231 | 44 |
| 3 | 13 | 1081 | 1115 |
| 3 | 14 | 271 | 256 |
| 4 | 4 | 1525 | 1629 |
| 4 | 5 | 1884 | 1769 |
| 4 | 6 | 4050 | 4000 |
| 4 | 8 | 1938 | 1956 |
| 4 | 9 | 460 | 461 |
| 4 | 10 | 1460 | 1468 |
| 4 | 11 | 369 | 375 |
| 4 | 12 | 1216 | 1229 |
| 4 | 14 | 1006 | 1010 |
| 5 | 5 | 2289 | 2383 |
| 5 | 7 | 2906 | 2880 |
| 5 | 8 | 835 | 843 |
| 5 | 9 | 2196 | 2173 |
| 5 | 11 | 1534 | 1575 |
| 5 | 12 | 328 | 373 |
| 5 | 13 | 917 | 948 |
| 5 | 14 | 263 | 103 |
| 6 | 6 | 3448 | 3468 |
| 6 | 7 | 546 | 576 |
| 6 | 8 | 2113 | 2161 |
| 6 | 9 | 232 | 183 |
| 6 | 10 | 1019 | 1078 |
| 6 | 11 | 332 | 313 |
| 6 | 12 | 1129 | 1131 |
| 7 | 7 | 2806 | 2877 |
| 7 | 9 | 2146 | 2136 |
| 7 | 11 | 1826 | 1817 |
| 7 | 12 | 515 | 515 |
| 7 | 13 | 891 | 926 |
| 8 | 8 | 2647 | 2734 |
| 8 | 10 | 1557 | 1582 |
| 8 | 12 | 842 | 865 |
| 9 | 9 | 847 | 818 |
| 9 | 11 | 725 | 718 |
| 9 | 12 | 403 | 434 |
| 10 | 10 | 738 | 740 |
| 10 | 11 | 326 | 291 |
| | L = 1 | | |
| 0 | 1 | 5491 | 5401 |
| 0 | 2 | 9539 | 10069 |
| 0 | 3 | 4954 | 4968 |
| 0 | 4 | 2306 | 2153 |
| 0 | 5 | 5939 | 5943 |
| 0 | 6 | 1568 | 1546 |
| 0 | 7 | 3775 | 3767 |
| 0 | 8 | 601 | 572 |
| 0 | 9 | 2101 | 2015 |
| 0 | 10 | 727 | 725 |
| 0 | 11 | 1931 | 1942 |
| 0 | 12 | 427 | 413 |
| 0 | 13 | 1542 | 1565 |
| 0 | 15 | 854 | 865 |
| 1 | 2 | 7600 | 8128 |
| 1 | 3 | 4530 | 4416 |
| 1 | 4 | 6094 | 6016 |
| 1 | 5 | 853 | 791 |
| 1 | 6 | 4094 | 3960 |
| 1 | 7 | 193 | 97 |
| 1 | 8 | 3321 | 3285 |
| 1 | 9 | 606 | 633 |
| 1 | 10 | 2068 | 2050 |
| 1 | 11 | 1416 | 1428 |
| 1 | 12 | 1265 | 1300 |
| 1 | 13 | 357 | 290 |
| 1 | 14 | 1203 | 1233 |
| 1 | 15 | 469 | 395 |
| 2 | 3 | 3713 | 3684 |
| 2 | 4 | 1243 | 1156 |
| 2 | 5 | 4130 | 4042 |
| 2 | 6 | 1655 | 1565 |
| 2 | 7 | 3742 | 3743 |
| 2 | 9 | 2900 | 2957 |
| 2 | 10 | 975 | 1010 |
| 2 | 11 | 1704 | 1759 |
| 2 | 12 | 302 | 295 |
| 2 | 13 | 1177 | 1219 |
| 2 | 14 | 333 | 201 |
| 3 | 2 | 3704 | 3684 |
| 3 | 4 | 3571 | 3526 |
| 3 | 5 | 1585 | 1503 |
| 3 | 6 | 2546 | 2527 |
| 3 | 7 | 563 | 484 |
| 3 | 8 | 3807 | 3844 |
| 3 | 9 | 1419 | 1418 |
| 3 | 10 | 2115 | 2118 |
| 3 | 12 | 1305 | 1318 |
| 3 | 14 | 1077 | 1102 |
| 4 | 5 | 3879 | 3813 |
| 4 | 6 | 1089 | 1052 |
| 4 | 7 | 2529 | 2540 |
| 4 | 8 | 691 | 728 |
| 4 | 9 | 1808 | 1806 |
| 4 | 10 | 514 | 525 |
| 4 | 11 | 1180 | 1155 |
| 4 | 12 | 315 | 294 |
| 4 | 13 | 1380 | 1334 |
| 5 | 6 | 2852 | 2773 |
| 5 | 7 | 308 | 281 |
| 5 | 8 | 2433 | 2406 |
| 5 | 9 | 325 | 320 |
| 5 | 10 | 1425 | 1400 |
| 5 | 11 | 270 | 284 |
| 5 | 12 | 1141 | 1165 |
| 5 | 13 | 322 | 220 |
| 5 | 14 | 975 | 976 |
| 6 | 7 | 3338 | 3322 |
| 6 | 8 | 533 | 541 |
| 6 | 9 | 2282 | 2340 |
| 6 | 10 | 233 | 190 |
| 6 | 11 | 1427 | 1453 |
| 6 | 13 | 1086 | 1071 |
| 7 | 8 | 2250 | 2288 |
| 7 | 9 | 754 | 769 |
| 7 | 10 | 1597 | 1613 |
| 7 | 11 | 271 | 303 |
| 7 | 12 | 948 | 1007 |
| 7 | 13 | 242 | 170 |
| 8 | 9 | 1108 | 1125 |
| 8 | 10 | 466 | 489 |
| 8 | 11 | 1021 | 1052 |
| 9 | 10 | 1240 | 1268 |
| 9 | 11 | 267 | 236 |
| 9 | 12 | 962 | 954 |
| 10 | 11 | 960 | 956 |
| | L = 2 | | |
| 0 | 0 | 653 | 704 |
| 0 | 1 | 1763 | 1666 |
| 0 | 2 | 2852 | 2768 |
| 0 | 3 | 3848 | 3619 |
| 0 | 4 | 5563 | 5565 |
| 0 | 5 | 563 | 912 |
| 0 | 6 | 2311 | 2308 |
| 0 | 8 | 2696 | 2656 |
| 0 | 9 | 276 | 280 |
| 0 | 10 | 2215 | 2212 |
| 0 | 12 | 1413 | 1419 |
| 0 | 14 | 985 | 970 |
| 1 | 1 | 5682 | 5508 |
| 1 | 2 | 2751 | 2572 |
| 1 | 3 | 5320 | 5239 |
| 1 | 4 | 1328 | 1216 |
| 1 | 5 | 5109 | 4998 |
| 1 | 6 | 1468 | 1368 |
| 1 | 7 | 2465 | 2496 |
| 1 | 8 | 1077 | 1034 |
| 1 | 9 | 1834 | 1830 |
| 1 | 10 | 206 | 158 |
| 1 | 11 | 1940 | 1957 |
| 1 | 12 | 346 | 335 |
| 1 | 13 | 1112 | 1160 |
| 1 | 14 | 354 | 313 |
| 2 | 0 | 2855 | 2768 |
| 2 | 2 | 6375 | 6402 |
| 2 | 3 | 1625 | 1560 |
| 2 | 4 | 5706 | 5734 |
| 2 | 5 | 2045 | 2026 |
| 2 | 6 | 2843 | 2889 |
| 2 | 7 | 427 | 443 |
| 2 | ·8 | 2487 | 2495 |
| 2 | 9 | 532 | 543 |
| 2 | 10 | 1964 | 1965 |
| 2 | 11 | 397 | 381 |
| 2 | 12 | 1364 | 1434 |
| 2 | 13 | 465 | 553 |
| 2 | 14 | 927 | 927 |
| 3 | 3 | 4821 | 4814 |
| 3 | 4 | 2164 | 2155 |
| 3 | 5 | 3394 | 3377 |
| 3 | 6 | 537 | 478 |
| 3 | 7 | 3014 | 3041 |
| 3 | 8 | 817 | 856 |
| 3 | 9 | 2076 | 2080 |
| 3 | 10 | 707 | 753 |
| 3 | 11 | 2093 | 2121 |
| 3 | 12 | 261 | 355 |
| 3 | 13 | 1117 | 1119 |
| 3 | 14 | 273 | 250 |
| 4 | 4 | 2496 | 2512 |
| 4 | 5 | 916 | 897 |
| 4 | 6 | 3135 | 3088 |
| 4 | 7 | 1351 | 1381 |
| 4 | 8 | 2820 | 2838 |
| 4 | 9 | 802 | 838 |
| 4 | 10 | 2262 | 2262 |
| 4 | 11 | 266 | 343 |
| 4 | 12 | 1121 | 1165 |
| 4 | 13 | 336 | 332 |
| 4 | 14 | 822 | 779 |
| 5 | 5 | 3123 | 3026 |
| 5 | 6 | 664 | 711 |
| 5 | 7 | 2560 | 2908 |
| 5 | 8 | 1142 | 1171 |
| 5 | 9 | 1860 | 1872 |
| 5 | 11 | 1422 | 1431 |
| 5 | 12 | 309 | 350 |
| 5 | 13 | 927 | 900 |
| 6 | 6 | 2763 | 2715 |
| 6 | 7 | 750 | 745 |
| 6 | 8 | 1777 | 1817 |
| 6 | 9 | 604 | 594 |
| 6 | 10 | 1428 | 1433 |
| 6 | 11 | 518 | 603 |
| 6 | 12 | 1108 | 1120 |
| 7 | 7 | 1845 | 1784 |
| 7 | 8 | 562 | 576 |
| 7 | 9 | 1320 | 1331 |
| 7 | 11 | 983 | 1006 |
| 7 | 13 | 985 | 950 |
| 8 | 8 | 1564 | 1600 |
| 8 | 10 | 1135 | 1166 |
| 8 | 12 | 937 | 887 |
| 9 | 9 | 1522 | 1556 |
| 9 | 10 | 251 | 222 |
| 9 | 11 | 955 | 985 |
| 10 | 10 | 1041 | 1040 |
| | L = 3 | | |
| 0 | 1 | 2975 | 2971 |
| 0 | 2 | 1850 | 1776 |
| 0 | 3 | 3459 | 3417 |
| 0 | 4 | 1855 | 1773 |
| 0 | 5 | 1535 | 1650 |
| 0 | 6 | 664 | 609 |
| 0 | 7 | 2652 | 2627 |
| 0 | 9 | 2085 | 2031 |
| 0 | 11 | 1843 | 1858 |
| 0 | 12 | 334 | 268 |
| 0 | 13 | 738 | 762 |
| 1 | 2 | 3940 | 3840 |
| 1 | 3 | 743 | 752 |
| 1 | 4 | 2295 | 2275 |
| 1 | 5 | 602 | 612 |
| 1 | 6 | 3085 | 3044 |
| 1 | 7 | 645 | 618 |
| 1 | 8 | 1681 | 1670 |
| 1 | 9 | 519 | 543 |
| 1 | 10 | 1923 | 1959 |
| 1 | 12 | 1375 | 1415 |
| 1 | 14 | 652 | 660 |
| 2 | 3 | 5514 | 5407 |
| 2 | 4 | 482 | 453 |
| 2 | 5 | 3135 | 3061 |
| 2 | 6 | 803 | 738 |
| 2 | 7 | 2158 | 2082 |
| 2 | 8 | 625 | 682 |
| 2 | 9 | 1619 | 1608 |
| 2 | 10 | 501 | 494 |
| 2 | 11 | 1583 | 1651 |
| 2 | 13 | 861 | 867 |
| 2 | 14 | 238 | 173 |
| 3 | 4 | 5105 | 5038 |
| 3 | 5 | 861 | 837 |
| 3 | 6 | 3607 | 3572 |
| 3 | 7 | 575 | 584 |
| 3 | 8 | 1561 | 1534 |
| 3 | 9 | 517 | 518 |
| 3 | 10 | 1631 | 1653 |
| 3 | 12 | 1311 | 1320 |
| 4 | 5 | 4016 | 4032 |
| 4 | 6 | 841 | 840 |
| 4 | 7 | 2510 | 2563 |
| 4 | 8 | 433 | 488 |
| 4 | 9 | 2027 | 1993 |
| 4 | 11 | 1718 | 1756 |
| 4 | 13 | 987 | 975 |
| 5 | 6 | 2907 | 2870 |
| 5 | 7 | 938 | 944 |
| 5 | 8 | 1832 | 1801 |
| 5 | 9 | 417 | 444 |
| 5 | 10 | 1770 | 1822 |
| 5 | 12 | 1251 | 1297 |
| 6 | 7 | 1037 | 1016 |
| 6 | 8 | 1009 | 1002 |
| 6 | 9 | 1433 | 1420 |
| 6 | 10 | 237 | 171 |
| 6 | 11 | 1178 | 1169 |
| 7 | 8 | 1159 | 1211 |
| 7 | 9 | 311 | 322 |
| 7 | 10 | 1230 | 1229 |
| 7 | 12 | 915 | 901 |
| 8 | 9 | 1396 | 1430 |
| 8 | 11 | 1041 | 1012 |
| 9 | 10 | 944 | 971 |
| | L = 4 | | |
| 0 | 0 | 1345 | 1318 |
| 0 | 1 | 1206 | 1137 |
| 0 | 2 | 3095 | 3105 |
| 0 | 3 | 840 | 735 |
| 0 | 4 | 2081 | 2066 |
| 0 | 6 | 2563 | 2575 |
| 0 | 7 | 403 | 507 |
| 0 | 8 | 1459 | 1430 |
| 0 | 9 | 382 | 440 |
| 0 | 10 | 1658 | 1767 |
| 1 | 1 | 2369 | 2354 |
| 1 | 2 | 1284 | 1250 |
| 1 | 3 | 3229 | 3185 |
| 1 | 4 | 179 | 176 |
| 1 | 5 | 2304 | 2229 |
| 1 | 6 | 712 | 721 |
| 1 | 7 | 2146 | 2133 |
| 1 | 8 | 325 | 375 |
| 1 | 9 | 1387 | 1403 |
| 1 | 10 | 355 | 285 |
| 1 | 11 | 1404 | 1409 |
| 1 | 13 | 650 | 704 |
| 2 | 2 | 4236 | 4225 |
| 2 | 3 | 773 | 769 |
| 2 | 4 | 2571 | 2560 |
| 2 | 5 | 355 | 469 |
| 2 | 6 | 2292 | 2270 |
| 2 | 7 | 619 | 593 |
| 2 | 8 | 1360 | 1289 |
| 2 | 10 | 1408 | 1430 |
| 2 | 11 | 413 | 393 |
| 2 | 12 | 926 | 932 |
| 3 | 3 | 3663 | 3693 |
| 3 | 4 | 403 | 378 |
| 3 | 5 | 2739 | 2677 |
| 3 | 6 | 1101 | 1123 |
| 3 | 7 | 2111 | 2061 |
| 3 | 8 | 408 | 516 |
| 3 | 9 | 1389 | 1401 |
| 3 | 10 | 290 | 283 |
| 3 | 11 | 1246 | 1255 |
| 3 | 13 | 771 | 772 |
| 4 | 4 | 3362 | 3313 |
| 4 | 5 | 1618 | 1658 |
| 4 | 6 | 2376 | 2354 |
| 4 | 7 | 915 | 911 |
| 4 | 8 | 1608 | 1637 |
| 4 | 9 | 316 | 375 |
| 4 | 10 | 1464 | 1498 |
| 4 | 11 | 226 | 228 |
| 4 | 12 | 1128 | 1121 |
| 5 | 5 | 2659 | 2661 |
| 5 | 6 | 793 | 813 |
| 5 | 7 | 1675 | 1688 |
| 5 | 8 | 478 | 509 |
| 5 | 9 | 1521 | 1525 |
| 5 | 10 | 353 | 325 |
| 5 | 11 | 1129 | 1146 |
| 6 | 6 | 1852 | 1870 |
| 6 | 7 | 926 | 979 |
| 6 | 8 | 1218 | 1211 |
| 6 | 10 | 973 | 986 |
| 6 | 11 | 254 | 150 |
| 6 | 12 | 750 | 736 |
| 7 | 7 | 1192 | 1112 |
| 7 | 8 | 373 | 362 |
| 7 | 9 | 1039 | 1023 |
| 7 | 11 | 688 | 718 |
| 8 | 8 | 1175 | 1121 |
| 8 | 10 | 903 | 929 |
| 9 | 9 | 1118 | 1107 |
| | L = 5 | | |
| 0 | 1 | 2360 | 2346 |
| 0 | 2 | 364 | 351 |
| 0 | 3 | 2880 | 2868 |
| 0 | 4 | 761 | 758 |
| 0 | 5 | 2125 | 2153 |
| 0 | 6 | 437 | 411 |
| 0 | 7 | 1480 | 1508 |
| 0 | 8 | 481 | 553 |
| 0 | 9 | 1337 | 1385 |
| 0 | 11 | 772 | 726 |
| 0 | 12 | 336 | 309 |
| 1 | 2 | 1917 | 1955 |
| 1 | 3 | 1475 | 1455 |
| 1 | 4 | 2522 | 2490 |
| 1 | 6 | 2043 | 2038 |
| 1 | 7 | 366 | 438 |
| 1 | 8 | 1439 | 1444 |
| 1 | 9 | 386 | 406 |
| 1 | 10 | 953 | 983 |
| 1 | 11 | 246 | 225 |
| 1 | 12 | 940 | 877 |
| 2 | 3 | 2418 | 2468 |
| 2 | 4 | 708 | 657 |
| 2 | 5 | 1957 | 1990 |
| 2 | 6 | 297 | 361 |
| 2 | 7 | 1545 | 1465 |
| 2 | 8 | 476 | 507 |
| 2 | 9 | 1166 | 1169 |
| 2 | 10 | 292 | 325 |
| 2 | 11 | 875 | 890 |
| 2 | 12 | 259 | 146 |
| 3 | 4 | 1496 | 1425 |
| 3 | 5 | 400 | 432 |
| 3 | 6 | 1645 | 1651 |
| 3 | 7 | 204 | 172 |
| 3 | 8 | 1266 | 1243 |
| 3 | 10 | 914 | 927 |
| 3 | 12 | 640 | 669 |
| 4 | 5 | 905 | 892 |
| 4 | 6 | 219 | 226 |
| 4 | 7 | 1784 | 1801 |
| 4 | 8 | 825 | 861 |
| 4 | 9 | 1341 | 1343 |
| 4 | 10 | 260 | 252 |
| 4 | 11 | 802 | 821 |
| 5 | 6 | 1769 | 1793 |
| 5 | 7 | 944 | 912 |
| 5 | 8 | 1421 | 1460 |
| 5 | 9 | 406 | 380 |
| 5 | 10 | 828 | 901 |
| 6 | 7 | 1328 | 1319 |
| 6 | 8 | 312 | 317 |
| 6 | 9 | 708 | 700 |
| 6 | 10 | 391 | 395 |
| 6 | 11 | 513 | 535 |
| 7 | 8 | 1056 | 1053 |
| 7 | 9 | 383 | 350 |
| 7 | 10 | 686 | 622 |
| 8 | 9 | 858 | 867 |
| | L = 6 | | |
| 0 | 0 | 1512 | 1491 |
| 0 | 2 | 2007 | 2056 |
| 0 | 3 | 390 | 414 |
| 0 | 4 | 1392 | 1395 |
| 0 | 5 | 336 | 327 |
| 0 | 6 | 1291 | 1315 |
| 0 | 8 | 1489 | 1475 |
| 0 | 10 | 851 | 823 |
| 0 | 11 | 297 | 262 |
| 1 | 1 | 2658 | 2778 |
| 1 | 2 | 501 | 538 |
| 1 | 3 | 1050 | 1034 |
| 1 | 5 | 1625 | 1657 |
| 1 | 6 | 351 | 345 |
| 1 | 7 | 1585 | 1605 |
| 1 | 9 | 708 | 741 |
| 1 | 11 | 658 | 613 |
| 2 | 2 | 1813 | 1842 |
| 2 | 3 | 370 | 367 |
| 2 | 4 | 1090 | 1102 |
| 2 | 5 | 534 | 582 |
| 2 | 6 | 946 | 975 |
| 2 | 8 | 1513 | 1525 |
| 2 | 10 | 708 | 689 |
| 2 | 11 | 288 | 192 |
| 3 | 3 | 575 | 554 |
| 3 | 4 | 273 | 290 |
| 3 | 5 | 1281 | 1264 |
| 3 | 6 | 434 | 364 |
| 3 | 7 | 1291 | 1260 |
| 3 | 8 | 315 | 238 |
| 3 | 9 | 926 | 896 |
| 3 | 10 | 237 | 203 |
| 4 | 4 | 1655 | 1623 |
| 4 | 5 | 347 | 286 |
| 4 | 6 | 772 | 783 |
| 4 | 8 | 829 | 816 |
| 4 | 10 | 322 | 397 |
| 5 | 5 | 1151 | 1151 |
| 5 | 7 | 807 | 787 |
| 5 | 9 | 535 | 552 |
| 5 | 10 | 246 | 265 |
| 6 | 6 | 1022 | 1031 |
| 6 | 8 | 1159 | 1168 |
| 6 | 9 | 245 | 257 |
| 7 | 7 | 1165 | 1177 |
| | L = 7 | | |
| 0 | 1 | 2009 | 2075 |
| 0 | 3 | 1009 | 1009 |
| 0 | 5 | 705 | 732 |
| 0 | 7 | 729 | 753 |
| 0 | 9 | 953 | 954 |
| 1 | 2 | 675 | 716 |
| 1 | 3 | 784 | 782 |
| 1 | 4 | 1001 | 1021 |
| 1 | 5 | 243 | 212 |
| 1 | 6 | 858 | 847 |
| 1 | 7 | 358 | 361 |
| 1 | 8 | 538 | 555 |
| 2 | 3 | 927 | 969 |
| 2 | 4 | 220 | 171 |
| 2 | 5 | 991 | 1008 |
| 2 | 6 | 243 | 150 |
| 2 | 7 | 867 | 848 |
| 2 | 8 | 275 | 272 |
| 2 | 9 | 793 | 777 |
| 3 | 4 | 941 | 905 |
| 3 | 5 | 313 | 353 |
| 3 | 6 | 906 | 916 |
| 3 | 7 | 231 | 255 |
| 3 | 8 | 533 | 540 |
| 4 | 5 | 568 | 586 |
| 4 | 6 | 244 | 291 |
| 4 | 7 | 683 | 678 |
| 4 | 8 | 264 | 280 |
| 5 | 6 | 797 | 794 |
| 5 | 8 | 514 | 522 |
| 6 | 7 | 719 | 704 |
| | L = 8 | | |
| 0 | 0 | 362 | 496 |
| 0 | 1 | 500 | 500 |
| 0 | 2 | 775 | 784 |
| 0 | 3 | 246 | 264 |
| 0 | 4 | 927 | 972 |
| 0 | 6 | 657 | 678 |
| 1 | 1 | 352 | 392 |
| 1 | 2 | 417 | 405 |
| 1 | 3 | 909 | 904 |
| 1 | 4 | 237 | 280 |
| 1 | 5 | 646 | 647 |
| 2 | 2 | 515 | 527 |
| 2 | 3 | 225 | 235 |
| 2 | 4 | 553 | 539 |
| 2 | 5 | 310 | 164 |
| 2 | 6 | 544 | 545 |
| 3 | 3 | 775 | 765 |
| 3 | 5 | 569 | 550 |
| 4 | 4 | 645 | 650 |

actions which are responsible for holding the molecules in the solid state. The shortest H···H intramolecular contact, H(2) ($xyz$)···H(6) ($\bar{y}x\bar{z}$), at $\alpha$= 54·1° is 2·72 Å which is substantially greater than the 2·37 Å non-bonded hydrogen contact distance. In this regard we disagree with Busetti *et al.* (1967) who used only intramolecular contacts as a simple means of predicting the ring conformation in tetraphenylmethane. Furthermore the method of Ismailzade (1952) in considering only one intermolecular interaction is clearly not adequate. Ismailzade (1952) did not report his hydrogen atom positions although he used a C–H distance of 1·08 Å. We have calculated hydrogen atom coordinates based on his reported carbon atom positions and C–H length 1·08 Å in the same manner already indicated. Using these calculated hydrogen coordinates all non-bonded H···H contact distances were computed. At Ismailzade's conformation ($\alpha$= 35·5°) there are two contacts which are well below the allowed van der Waals closest approach for two hydrogen atoms. These are H(4) ($xyz$)···H(8) ($\frac{1}{2}-y$, $\frac{1}{2}-x$, $\frac{1}{2}+z$) at 2·09 Å and H(5) ($\bar{x}y\bar{z}$)···H(4) ($\frac{1}{2}-x$, $\frac{1}{2}-y$, $\frac{1}{2}-z$) at 1·92 Å. It is clear that in such geometrical analyses all distances must be considered at a given conformation to insure no violation of the allowed contact distances.

The valency angle C(1) ($\bar{x}\bar{y}z$)–Ge–C(1) of 109·0 (2)° is very near the tetrahedral value. The refined value of the Ge–C length is 1·957 (4) Å which compares with the values of 1·956 (4) Å found by Elder (1969) in $[Ge(C_6H_5)_2]_2Fe(CO)$ and 1·945 Å in $Ge(C_6H_5)_3(COCH_3)$ by Harrison & Trotter (1968). The phenyl ring is planar within experimental error. The sum of the interior angles of the phenyl ring is 720·0 (4)° and the C–C lengths in the ring vary from 1·381 (6) to 1·403 (6) Å with an average of 1·393 (6) Å. The angle between the Ge–C(1) bond vector and the C(1)–C(4) vector is 0° indicating the absence of any ring tilt.

The pertinent bond distances and angles are given in Table 4. The shortest non-bonded distances less than 3·5 Å are given in Table 5.

Table 4. *Bond distances and angles with their standard deviations*

| | |
|---|---|
| Ge—C(1) | 1·957 (4) Å |
| C(1)–C(2) | 1·397 (6) |
| C(2)–C(3) | 1·396 (6) |
| C(3)–C(4) | 1·388 (7) |
| C(4)–C(5) | 1·381 (6) |
| C(5)–C(6) | 1·403 (6) |
| C(6)–C(1) | 1·395 (5) |
| C(1) ($\bar{x}\bar{y}z$)–Ge–C(1) | 109·0 (2)° |
| Ge—C(1)–C(2) | 120·0 (4) |
| Ge—C(1)–C(6) | 122·0 (4) |
| C(1)–C(2)–C(3) | 120·9 (4) |
| C(2)–C(3)–C(4) | 120·0 (4) |
| C(3)–C(4)–C(5) | 119·8 (4) |
| C(4)–C(5)–C(6) | 120·3 (4) |
| C(5)–C(6)–C(1) | 120·6 (4) |
| C(6)–C(1)–C(2) | 118·4 (4) |

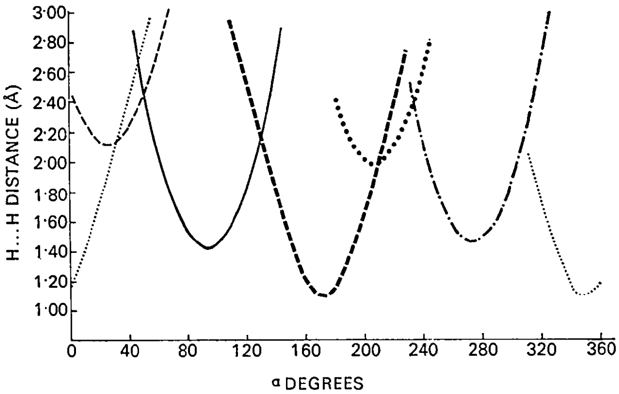

Fig. 2. H···H intermolecular contact distances.

| | |
|---|---|
| ············· | H(4) ($xyz$)···H(5) ($\frac{1}{2}-y$, $\frac{1}{4}-x$, $\frac{1}{2}-z$) |
| - - - - - - - - - | H(5) ($\bar{y}x\bar{z}$)···H(4) ($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) |
| ———————— | H(3) ($xyz$)···H(6) ($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) |
| ■ ■ ■ ■ ■ ■ | H(4) ($xyz$)···H(3) ($\frac{1}{2}-y$, $\frac{1}{2}-x$, $\frac{1}{2}+z$) |
| ● ● ● ● ● ● | H(3) ($\bar{y}x\bar{z}$)···H(4) ($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) |
| -·-·-·-·-·-·- | H(5) ($xyz$)···H(2) ($\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$). |

Table 5. *Intermolecular distances less than* 3·5 Å

| Atom | Asymmetric unit | Atom | Asymmetric unit | Distance |
|---|---|---|---|---|
| H(3) | $x, y, z$ | H(6) | $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$ | 2·37 Å |
| H(2) | $x, y, z$ | H(5) | $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$ | 2·93 |
| H(4) | $x, y, z$ | H(5) | $\frac{1}{2}-y, \frac{1}{2}-x, \frac{1}{2}+x$ | 2·91 |
| H(2) | $x, y, z$ | H(6) | $\bar{y}, x, \bar{z}$ | 2·72 |

*Note added in proof:* A structure determination of tetraphenylgermanium by Chieh (1971) has secently appeared. Although small differences are noted, there is little change from the structure reported here.

Grateful acknowledgement is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, Grant No. 1453–G3 and the Miami University Faculty Research Fund for support of this work.

## References

BONDI, A. (1964). *J. Phys. Chem.* **48**, 441.

BUSETTI, V., MAMMI, M., SIGNOR, A. & DEL PRA, A. (1967). *Inorg. Chemica Acta*, **1**, 424.

BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1964). *ORFLS*, report ORNL-TM-305; and *ORFFE*, report ORNL-TM-306, Oak Ridge National Laboratory, Oak Ridge, Tennessee.

CHIEH, P. C. (1971). *J. Chem. Soc.* (*A*), p. 3243.

CROMER, D. T. & WABER, J. T. (1968). *Acta Cryst.* A**24**, 321.

ELDER, M. (1969). *Inorg. Chem.* **8**, 2703.

GEORGE, W. H. (1927). *Proc. Roy. Soc.* A**113**, 585.

HARRISON, R. W. & TROTTER, J. (1968). *J. Chem. Soc.* (*A*), p. 258.

*International Tables for X-ray Crystallography* (1968). Vol. III. Table 3.3.1*A*. Birmingham: Kynoch Press.

ISMAILZADE, I. G. (1952). *Zh. Fiz. Khim. SSSR*, **26**, 1139.

ISMAILZADE, I. G. & ZDANOV, G. S. (1952). *Zh. Fiz. Khim. SSSR*, **26**, 1619.