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STRUCTURE OF TETRA-*o*-TOLYLGERMANIUM

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Summary

A full X-ray structure analysis of tetra-*o*-tolylgermanium, $C_{28}H_{28}Ge$, has been performed. Unlike the corresponding tetraphenyl derivative, this molecule loses its D_{2d} symmetry due to steric hindrance. The mean value of the Ge–C bond is 2.166 Å.

Introduction

It was found [1] that in a series of methyl-substituted derivatives of tetraphenyltin, the SnR_4 molecules retain their own D_{2d} symmetry regardless of the position of the methyl group in the ring. It was interesting to check whether this phenomenon occurs in the corresponding germanium compounds, considering that the covalent radius of Ge (1.225 Å) is substantially less than that of Sn (1.405 Å) [2].

Experimental

Crystals of I are triclinic, a 8.245(2), b 8.210(2), c 17.072(6) Å, α 79.48(2), β 84.20(2), 91.55(2)°, d_{calc} 1.26 g cm^{-3}, μ(Mo) 14.2 cm^{-1}, $Z = 2$, space group $P\bar{1}$. Experimental data were collected on an automatic four-circle diffractometer (Syntex $P\bar{1}$), employing Mo-K_α radiation, a graphite monochromator, and θ–2θ scanning. The intensities of 2460 reflections with $I > 3\sigma(I)$ were corrected for Lorentz and polarization effects but not for absorption. The structure was solved using the heavy-atom technique, and was refined using the method of anisotropic full-matrix least-squares. The coordinates of the H atoms were obtained from difference synthesis; their positional and isotropic thermal parameters were also refined. The final R-value was 0.026. All calculations were performed with the SHELXTL (G.M. Sheldrick) set of programs on a NOVA 3 computer incorporated in a NICOLET R3 crystallographic system. The atomic coordinates for the non-hydrogen atoms and values of U_{eq} (Å^2) are given in Table 1 *.

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* Tables of positional and thermal parameters of the hydrogen atoms, bond lengths and angles, anisotropic thermal parameters for Ge and C atoms, and mean planes may be obtained from the authors.

TABLE 1

ATOMIC COORDINATES FOR NON-HYDROGEN ATOMS AND THEIR e.s.d.'s ($\times 10^4$) AND EQUIVALENT THERMAL PARAMETERS ($U_{eq} \times 10^3$ Å^2)

Atom	x	y	z	U_{eq}
Ge	6170(1)	3260(1)	7482(1)	42(1)
C(1)	4719(4)	1795(3)	7052(2)	43(1)
C(2)	5287(4)	909(4)	6466(2)	52(1)
C(3)	4164(5)	−53(4)	6182(2)	63(1)
C(4)	2530(5)	−132(5)	6444(3)	68(2)
C(5)	1970(4)	765(4)	7004(2)	62(1)
C(6)	3072(4)	1711(4)	7308(2)	51(1)
C(7)	7041(5)	992(5)	6116(3)	76(2)
C(8)	4886(4)	4445(4)	8210(2)	44(1)
C(9)	4174(4)	3648(4)	8952(2)	47(1)
C(10)	3343(4)	4595(4)	9439(2)	59(1)
C(11)	3200(5)	6277(5)	9195(3)	68(2)
C(12)	3889(5)	7057(4)	8472(3)	65(2)
C(13)	4739(4)	6132(4)	7986(2)	53(1)
C(14)	4303(4)	1821(4)	9265(2)	61(1)
C(15)	7720(4)	1917(4)	8076(2)	46(1)
C(16)	8831(4)	2572(4)	8512(2)	54(1)
C(17)	9815(4)	1508(5)	8948(2)	67(2)
C(18)	9729(5)	−164(5)	8961(2)	79(2)
C(19)	8629(5)	−830(4)	8554(2)	73(2)
C(20)	7637(4)	207(4)	8110(2)	56(1)
C(21)	8965(5)	4387(5)	8545(2)	75(2)
C(22)	7303(4)	4847(4)	6595(2)	45(1)
C(23)	6479(4)	5761(4)	6004(2)	49(1)
C(24)	7377(5)	6809(4)	5379(2)	62(1)
C(25)	9046(5)	6966(5)	5329(3)	74(2)
C(26)	9863(4)	6047(5)	5893(3)	70(2)
C(27)	9009(4)	4992(4)	6521(2)	55(1)
C(28)	4654(4)	5610(4)	5997(2)	62(1)

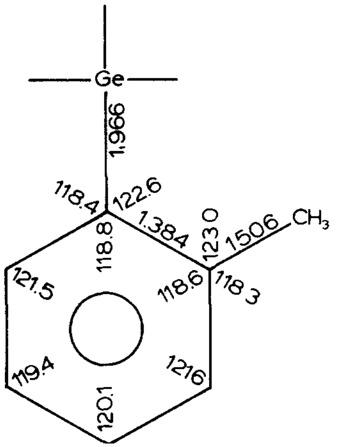

Fig. 1. Mean interatomic distances and bond angles.

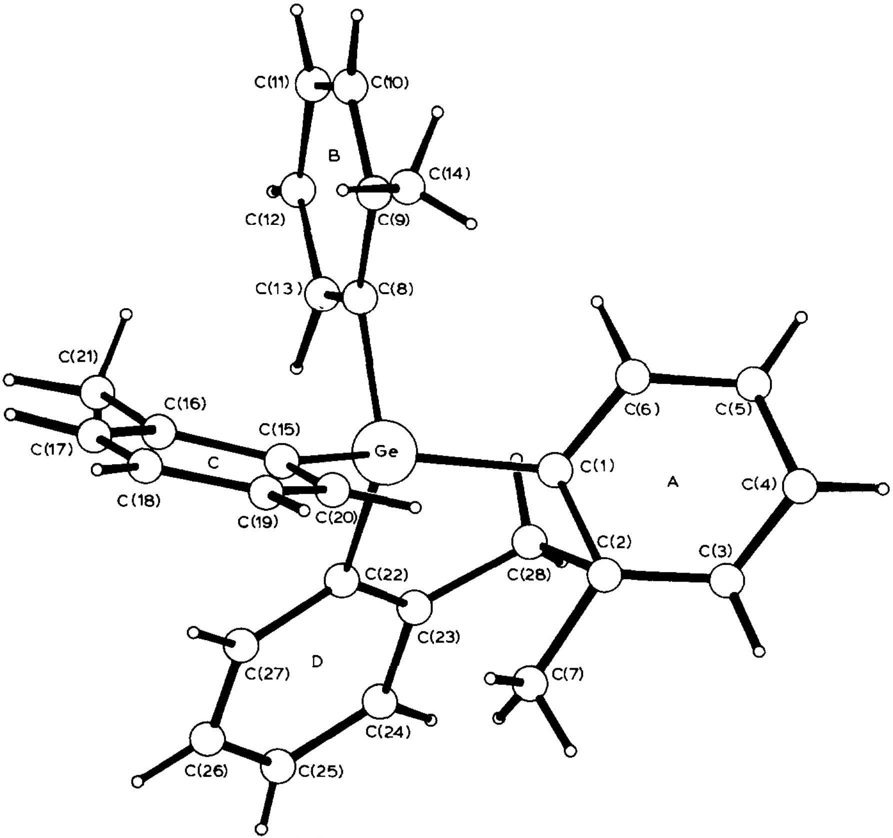

Fig. 2. Conformation of molecule I.

Figure 1 represents the interatomic distances and bond angles averaged for four independent *o*-tolyl groups. The e.s.d.'s are as follows: Ge–C 0.003, C–C 0.004-0.006 Å; CGeC 0.01, GeCC 0.02, CCC 0.03°. The mean value for the Ge–C bond (1.966 Å) is somewhat longer than that found [3] in tetraphenylgermanium (1.957 Å). This elongation may be explained (as for tin derivatives [1]) by steric hindrance caused by the presence of methyl groups in the *ortho* position. This factor is probably responsible for the loss of the own D_{2d} symmetry of the molecule. As can be seen from Fig. 2, the directions of the C–CH_3 bonds correspond to the own symmetry, C_2, of the molecule. The angles between the mean ring planes (maximum deviations for C atoms less than 0.01 Å, displacements of the Ge and CH_3 groups are in the limits 0.06–0.09 Å) are equal in pairs: A–B 108.6 and C–D 117.1, A–C 66.6 and B–D 66.7, A–D 78.3 and B–C 77.1°. The steric factor also influences the geometry of the *o*-tolyl groups. As Fig. 1 shows the system Ge–C–C–CH_3, the exocyclic angles are more, and the endocyclic angles less, than 120°. Intermolecular contacts correspond to normal Van der Waals interactions.

References

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