

Table 4. *Hydrogen-bond geometry* (Å, degrees)

Tifluadom hydrochloride					
N(4)—Cl	3.013 (2)	H(4)—Cl	2.09	N(4)—H(4)—Cl	150
N(12)—Cl	3.267 (2)	H(12)—Cl	2.36	N(12)—H(12)—Cl	148
N(4)—F(2')	2.868 (3)	H(4)—F(2')	2.52	N(4)—H(4)—F(2')	99
(+)-(2 <i>S</i> )-Tifluadom <i>p</i> -toluenesulphonate					
N(4)—O(8'')	2.687 (4)	H(4)—O(8'')	1.74	N(4)—H(4)—O(8'')	153
N(12)—O(9'')	2.941 (4)	H(12)—O(9'')	1.99	N(12)—H(12)—O(9'')	155

Crystal packing arrangements are shown in Figs. 3 and 4. In both crystal structures, N(4) and N(12) form intermolecular hydrogen bonds to the anion. In the hydrochloride that from N(4) is bifurcated to Cl and intramolecularly to F(2'); details are given in Table 4.

The crystal structures were determined principally to give some insight into the conformation of this novel opioid. It is reasonable to assume, however, that the solid-state conformation of the side chain is largely determined by the above-mentioned *intermolecular* H bonds. NMR investigations suggest (Milkowski & Finner, 1981) that in organic solvents, the molecule folds so that there is  $\pi$ - $\pi$  stacking of the thienyl and *o*-fluorophenyl rings. Such a conformation can readily be generated from the crystal structures by rotations of  $-120$  and  $180^\circ$  about C(2)—C(11) and C(11)—N(12), respectively, and is certainly energetically not unfavourable. Further experimental and theoretical conformational studies are clearly necessary before these molecules can, with any degree of certainty, be compared with the known conformations of classical opiates.

We acknowledge the skilful experimental contribution of our coworker H. D. Reineke to the chemistry of these compounds.

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*Acta Cryst.* (1985). **C41**, 912–915

## 1,1-Dimethyl-3,3,4,4-tetraphenylgermacyclopentane, C<sub>30</sub>H<sub>30</sub>Ge

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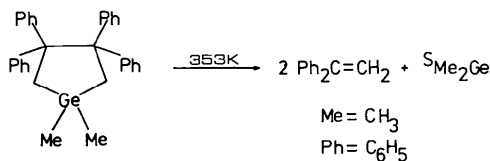
(Received 25 July 1984; accepted 16 January 1985)

**Abstract.**  $M_r = 463.2$ , monoclinic,  $P2_1/c$ ,  $a = 12.234$  (5),  $b = 9.942$  (4),  $c = 19.435$  (6) Å,  $\beta = 97.27$  (5)°,  $V = 2345$  Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.312$  Mg m<sup>-3</sup>, Mo  $K\alpha$ ,  $\lambda = 0.71069$  Å,  $\mu = 1.4$  mm<sup>-1</sup>,  $F(000) = 968$ ,  $T = 292$  (1) K, final  $R = 0.049$  for 2218 [ $I > 2\sigma(I)$ ] unique diffractometer data. The central part of the asymmetric monomeric molecule is a five-membered heterocycle consisting of one Ge and four C atoms. The coordination around Ge is a distorted tetrahedron. An extraordinarily long bond distance between two C atoms of the five-membered heterocycle [1.626 (7) Å] is probably caused by repulsion between the four phenyl groups bound to these C atoms and between the

phenyl groups and C atoms of the heterocycle, which may be the reason for the surprising thermolability of the title compound. The initial step of the thermal decomposition is probably the cleavage of this highly strained and elongated C—C bond.

**Introduction.** Germacyclopentanes like 1,1-dimethyl-3,4-diphenylgermacyclopentane are normally stable up to at least 473 K (Köcher & Neumann, 1985). 1,1-Dimethyl-3,3,4,4-tetraphenylgermacyclopentane, however, gives a uniform fragmentation even at 353 K, and is a thermal source for dimethylgermylene, Me<sub>2</sub>Ge, a member of (heavy) carbene analogues  $R_2M$ ,  $M$

= Si, Ge, Sn, which have attracted much attention during the last years in both chemical and structural aspects; see, for example, Schriewer & Neumann (1983), Köcher & Lehnig (1984).



This Me<sub>2</sub>Ge species can be identified by means of scavenger experiments which demonstrate its singlet ground state (Me<sub>2</sub>Ge<sup>s</sup>) as in the case of the thermal cleavage of 7-germabicyclo[2.2.1]heptadienes (Schriewer & Neumann, 1983; Köcher & Lehnig, 1984).

We therefore conclude that the reason for this unusual behaviour of the title compound could be an instability of the fully substituted C—C bond resulting from severe steric crowding by the four phenyl groups. A similar situation has been observed in the case of fully substituted ethanes, which show thermal lability because of their weakened central C—C bonds indicated by unusual lengths of up to 1.63 Å (Rüchardt & Beckhaus, 1980). In order better to understand the structural features associated with the surprising thermal instability of the title compound it has been examined by X-ray analysis.

**Experimental.** Colourless plates grown by recrystallization from methylene chloride/methanol, approximate dimensions 0.08 × 0.33 × 0.23 mm. Data collection on CAD-4 diffractometer with graphite-monochromated Mo K $\alpha$  radiation,  $\omega/2\theta$  scan technique, scan speed 3.33° min<sup>-1</sup> in  $\omega$ ; cell parameters from 25 reflexions in range 4.1 ≤  $\theta$  ≤ 11.8°; 4884 reflexions measured in range 1 ≤  $\theta$  ≤ 25°,  $h$ : -14 → +14,  $k$ : 0 → 11,  $l$ : 0 → 23; four standard reflexions recorded every 9000 s remained stable; intensities corrected for Lorentz-polarization effects and absorption *via*  $\psi$  scans; max./min. transmission 1.00/0.87; systematic absences  $h0l$ ,  $l = 2n + 1$ ,  $0k0$ ,  $k = 2n + 1$ , indicated space group  $P2_1/c$ ; structure solved *via* Patterson function,  $\Delta F$  syntheses and full-matrix least squares on  $F$  with 2218 [ $I > 2\sigma(I)$ ] reflexions; 281 refined parameters, anisotropic temperature factors for all non-H atoms, common isotropic temperature factor for all H atoms; H atoms in geometrically calculated positions (C—H 0.95 Å),  $w^{-1} = [\sigma^2(I) + (0.04F_o^2)^2]^{1/2}$ ,  $S = 1.27$ ,  $R = 0.049$ ,  $wR = 0.057$ , max.  $\Delta/\sigma = 0.36$ , largest peak in final  $\Delta F$  map = 0.33 (7) e Å<sup>-3</sup>; complex neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974); programs: *Enraf-Nonius Structure Determination Package* (Frenz, 1981), *ORTEP* (Johnson, 1976), *POP1* (van de Waal, 1976).

**Discussion.** The structure and conformation of the title compound are shown in Fig. 1 (*ORTEP*) and in a stereoscopic view in Fig. 2 (*POP1*). Atomic parameters are given in Table 1,\* selected bond lengths and angles in Table 2. The central part of the molecule is a five-membered heterocycle consisting of one Ge atom and four C atoms. The geometry of this ring indicates a high strain, which is revealed by varying deviations of the bond angles from the ideal tetrahedral angle (109.47°) and by bond elongations. All endocyclic bond angles of the five-membered heterocycle are smaller than the tetrahedral angle. Because of the higher polarizability of the Ge atom the ring angle C(6)—Ge(1)—C(3) shows the maximum deviation from the tetrahedral angle. All C—C bonds in the five-membered heterocycle are significantly lengthened with regard to the single-bond distance of 1.54 Å (Pauling, 1970). The bond distance C(4)—C(5) is one of the longest C—C single-bond distances that have so far been measured.

\* Lists of structure factors, thermal parameters, H-atom parameters and a full list of bond lengths and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42062 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

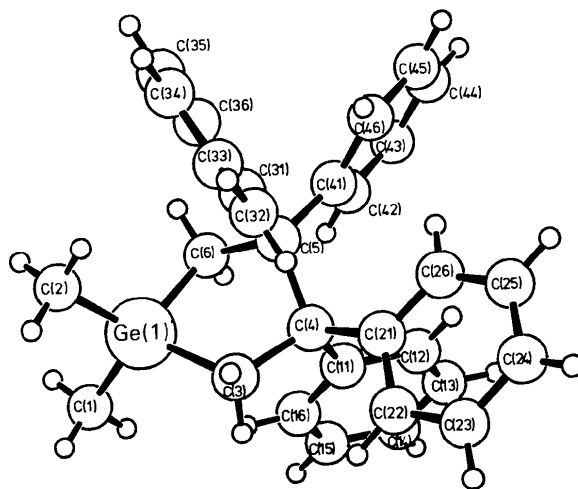


Fig. 1. General view of the molecule.

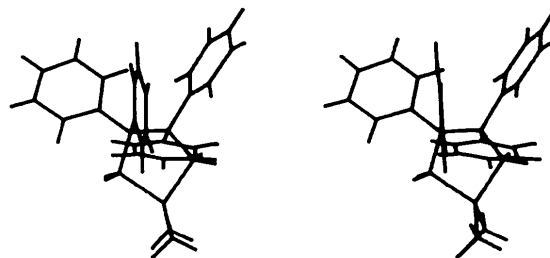


Fig. 2. Stereoscopic view of the molecule.

Table 1. Atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ )
$$B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i a_j$$

	x	y	z	$B_{eq}$
Ge(1)	0.18768 (6)	0.23039 (7)	0.20548 (3)	3.43 (1)
C(1)	0.0560 (6)	0.2225 (8)	0.1380 (3)	5.4 (2)
C(2)	0.3116 (6)	0.1951 (7)	0.1555 (3)	4.7 (2)
C(3)	0.2010 (5)	0.3937 (6)	0.2624 (3)	2.8 (1)
C(4)	0.1993 (5)	0.3531 (5)	0.3404 (3)	2.7 (1)
C(5)	0.2532 (4)	0.2035 (5)	0.3477 (3)	2.5 (1)
C(6)	0.1856 (5)	0.1208 (6)	0.2886 (3)	2.9 (1)
C(11)	0.0800 (5)	0.3477 (6)	0.3582 (3)	2.8 (1)
C(12)	0.0568 (5)	0.3549 (6)	0.4262 (3)	3.4 (1)
C(13)	-0.0496 (5)	0.3510 (6)	0.4431 (3)	3.9 (2)
C(14)	-0.1370 (5)	0.3378 (7)	0.3917 (4)	4.1 (2)
C(15)	-0.1166 (5)	0.3299 (7)	0.3242 (4)	4.1 (2)
C(16)	-0.0102 (5)	0.3349 (6)	0.3074 (3)	3.5 (1)
C(21)	0.2571 (5)	0.4642 (6)	0.3879 (3)	3.0 (1)
C(22)	0.2421 (5)	0.5963 (7)	0.3678 (4)	4.3 (2)
C(23)	0.2877 (7)	0.7014 (6)	0.4099 (4)	6.0 (2)
C(24)	0.3488 (6)	0.6746 (7)	0.4721 (4)	5.6 (2)
C(25)	0.3627 (5)	0.5456 (7)	0.4932 (4)	4.4 (2)
C(26)	0.3156 (5)	0.4419 (6)	0.4518 (3)	3.9 (2)
C(31)	0.3746 (4)	0.2016 (5)	0.3317 (3)	2.7 (1)
C(32)	0.4416 (5)	0.3117 (5)	0.3276 (3)	2.8 (1)
C(33)	0.5494 (5)	0.3008 (6)	0.3132 (3)	3.5 (1)
C(34)	0.5925 (5)	0.1768 (6)	0.3025 (3)	3.6 (1)
C(35)	0.5284 (5)	0.0633 (6)	0.3076 (3)	3.4 (1)
C(36)	0.4230 (5)	0.0765 (6)	0.3222 (3)	3.1 (1)
C(41)	0.2479 (5)	0.1286 (5)	0.4164 (3)	2.5 (1)
C(42)	0.1537 (5)	0.0668 (6)	0.4333 (3)	3.4 (1)
C(43)	0.1510 (5)	-0.0064 (6)	0.4929 (4)	4.5 (2)
C(44)	0.2450 (6)	-0.0192 (7)	0.5397 (3)	4.3 (2)
C(45)	0.3397 (5)	0.0396 (6)	0.5249 (3)	3.8 (2)
C(46)	0.3420 (5)	0.1124 (6)	0.4650 (3)	3.1 (1)

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) in  $C_{30}H_{30}Ge$ 

The C—C distances and the C—C—C angles in the phenyl groups are in the range 1.350 (9) to 1.403 (7)  $\text{\AA}$  [mean: 1.378 (8)  $\text{\AA}$ ] and 115.3 (5) to 123.1 (5) $^\circ$  [mean: 120.0 (5) $^\circ$ ]. The C—C distances and the C—C—C angles around the phenyl group positions are in the range 1.537 (7) to 1.557 (6)  $\text{\AA}$  [mean: 1.547 (7)  $\text{\AA}$ ] and 117.9 (4) to 124.8 (5) $^\circ$  [mean: 122.0 (5) $^\circ$ ].

Ge(1)—C(1)	1.946 (6)	Ge(1)—C(2)	1.933 (6)
Ge(1)—C(3)	1.960 (6)	Ge(1)—C(6)	1.951 (6)
C(3)—C(4)	1.572 (8)	C(4)—C(11)	1.543 (7)
C(4)—C(21)	1.551 (8)	C(4)—C(5)	1.626 (7)
C(5)—C(31)	1.557 (6)	C(5)—C(41)	1.537 (7)
C(5)—C(6)	1.562 (7)		
C(2)—Ge(1)—C(1)	106.8 (3)	C(21)—C(4)—C(3)	109.3 (4)
C(2)—Ge(1)—C(6)	114.0 (2)	C(21)—C(4)—C(5)	116.7 (4)
C(2)—Ge(1)—C(3)	114.8 (2)	C(3)—C(4)—C(5)	105.4 (4)
C(1)—Ge(1)—C(6)	116.1 (2)	C(41)—C(5)—C(31)	108.3 (4)
C(1)—Ge(1)—C(3)	114.6 (3)	C(41)—C(5)—C(6)	107.9 (4)
C(6)—Ge(1)—C(3)	90.3 (2)	C(41)—C(5)—C(4)	117.3 (4)
C(4)—C(3)—Ge(1)	108.7 (3)	C(31)—C(5)—C(6)	106.0 (4)
C(11)—C(4)—C(21)	105.3 (4)	C(31)—C(5)—C(4)	112.4 (4)
C(11)—C(4)—C(3)	110.7 (4)	C(6)—C(5)—C(4)	104.3 (4)
C(11)—C(4)—C(5)	109.5 (4)	C(5)—C(6)—Ge(1)	104.7 (3)

Table 3. Short intramolecular distances ( $\text{\AA}$ ) in  $C_{30}H_{30}Ge$ 

C(22)...C(3)	2.872 (9)	C(32)...C(3)	3.161 (7)
C(16)...C(3)	2.890 (7)	C(26)...C(12)	3.259 (8)
C(36)...C(6)	2.928 (7)	C(16)...C(6)	3.259 (8)
C(42)...C(6)	2.937 (8)	C(32)...C(26)	3.288 (8)
C(46)...C(36)	3.083 (8)	C(46)...C(26)	3.298 (8)
C(42)...C(12)	3.097 (8)		

The elongation of this bond is mainly caused by the repulsion of the conflicting phenyl groups. In Table 3 selected non-bonding C...C distances are given that are markedly shorter than the sum of the van der Waals radii of 3.60  $\text{\AA}$  (Kitaigorodskii, 1979) and which indicate highly repulsive van der Waals forces. The widening of the bond angles C(4)—C(5)—C(41), C(5)—C(4)—C(21) supports this conclusion, too. Systematic investigations on symmetric ethane derivatives (Rüchardt & Beckhaus, 1980) have shown that steric pressure of the substituents causes, apart from bond-angle widenings, elongations of the central C—C and neighbouring bonds. Similar long C—C bonds were found in 5,6-dibutyl-5,6-diphenyldecane (Littke & Drück, 1979) (1.638  $\text{\AA}$ ), 3,4-dicyclohexyl-3,4-dimethylhexane (Beckhaus, Kratt, Lay, Geiselmann, Rüchardt, Kitschke & Lindner, 1980) [1.626 (7)  $\text{\AA}$ ], *meso*-3,4-dicyclohexyl-2,2,5,5-tetramethylhexane (Beckhaus, Hellmann, Rüchardt, Kitschke, Lindner & Fritz, 1978) [1.602 (6)  $\text{\AA}$ ] and pentaphenylethane (Bernardinelli & Gerdil, 1981) [1.612 (3)  $\text{\AA}$ ].

The dihedral angle between the least-squares plane through atoms Ge(1), C(3), C(4), C(6) [ $-0.922x + 0.071y - 0.106z = 2.044 \text{\AA}$ , maximum deviation  $-0.013 \text{\AA}$ ;  $xyz$  are orthogonalized coordinates,  $x$  along  $a$ ,  $y$  in the  $ab$  plane,  $z$  along  $c^*$ ] and the plane through atoms C(4), C(5) and C(6) ( $0.736x + 0.258y - 0.626z = 2.021 \text{\AA}$ ) is 49.8 (5) $^\circ$ .

The coordination around the Ge atom is a distorted tetrahedron, and the Ge—C bond lengths are in good agreement with those in compounds with tetrahedrally coordinated Ge, such as 1.945 (3)  $\text{\AA}$  in  $Ge(CH_3)_4$  (Hencher & Mustoe, 1975) (gas-phase electron diffraction) and values between 1.95 (2) and 2.00 (2)  $\text{\AA}$  in  $\mu$ -thio-bis[tribenzylgermanium(IV)] (Glidewell & Liles, 1982).

The initial step in the thermal decomposition is probably the cleavage of the highly strained and elongated C(4)—C(5) bond. The higher instability of the title compound compared with open-chain hexa-substituted ethanes (Rüchardt & Beckhaus, 1980) seems to be caused by the additional strain of the five-membered heterocycle, thus leading to the total cleavage of the molecule.

Intermolecular distances do not indicate interactions that exceed van der Waals forces.

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*Acta Cryst.* (1985). **C41**, 915–917

## Methyl 3,3,7,7-Tetramethyl-2-oxocycloheptanesulphinate, C<sub>12</sub>H<sub>22</sub>O<sub>3</sub>S

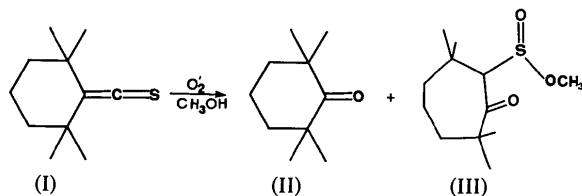
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(Received 7 September 1984; accepted 28 January 1985)

**Abstract.**  $M_r = 246.37$ , triclinic,  $P\bar{1}$ ,  $a = 8.680$  (2),  $b = 10.643$  (2),  $c = 8.306$  (2) Å,  $\alpha = 110.45$  (1),  $\beta = 111.20$  (1),  $\gamma = 79.97$  (2)°,  $V = 669.3$  (3) Å<sup>3</sup>,  $Z = 2$ ,  $D_m$ (flotation, aqueous KI) = 1.224,  $D_x = 1.222$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 2.2$  cm<sup>-1</sup>,  $F(000) = 268$ ,  $T = 295$  K,  $R = 0.036$  for 2971 observed reflections. The cycloheptane ring has a twist-chair conformation, with mean C–C = 1.538 (6) Å, mean C–C–C = 113.5 (16)°, C=O = 1.215 (2) Å. In the methyl sulphinate group, S has a pyramidal configuration, C–S = 1.824 (1), S=O = 1.466 (1), S–OCH<sub>3</sub> = 1.631 (1) Å. Packing of the molecules is governed by normal van der Waals contacts.

**Introduction.** In a study of the reactions of thioketones with singlet oxygen (Wasserman & Murray, 1979; Ramnath, Ramesh & Ramamurthy, 1983; Jayathirtha Rao, Ramamurthy, Schaumann & Nimmesgern, 1984), oxidation of the cyclohexane derivative (I) in methanol gave the ketone (II) and a novel product with proposed structure (III). The present X-ray analysis was undertaken to establish the structure of (III) and to determine the conformation of the cycloheptane ring.



**Experimental.** Colourless crystals, 0.05 × 0.38 × 0.26 mm, CAD-4 diffractometer, graphite-monochromatized Mo  $K\alpha$  radiation,  $\theta \leq 32.5^\circ$ ,  $\omega - 2\theta$

scan,  $\omega$ -scan width  $(0.90 + 0.35 \tan \theta)^\circ$  extended by 25% on either side of the peak for background measurement, horizontal aperture  $(2 + \tan \theta)$  mm, vertical aperture 4 mm. Cell parameters by least-squares fit to observed  $\sin^2 \theta$  values for 21 centred reflections with  $17 < \theta < 21^\circ$ , intensity and orientation controls regularly; decay of three standard intensities necessitated application of an isotropic decay correction. 4289 independent reflections ( $h - 12$  to 12,  $k - 15$  to 15,  $l$  0 to 12), 2971 with  $I \geq 3\sigma(I)$ ,  $\sigma^2(I) = S + 2B + [0.04(S - B)]^2$ ,  $S$  = total peak count and  $B$  = time-averaged background,  $L_p$  corrections (no absorption corrections). Structure solved by direct methods with *SHELX76* (Sheldrick, 1976), refined by full-matrix least squares on  $F$  with anisotropic thermal parameters, H atoms identified on a difference map and included in refinement with isotropic temperature factors; final  $R = 0.036$ ,  $wR = 0.039$  for 2971 observed reflections, 233 variables,  $R = 0.076$  for all data,  $w = 1/\sigma^2(F)$ ;  $(\Delta/\sigma)_{\max} = 0.05$ , max. height in final difference map  $0.3 \text{ e } \text{Å}^{-3}$ . Scattering factors from Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965), local adaptations of standard computer programs *ORFFE* (Busing, Martin & Levy, 1964) and *PLUTO* (Motherwell & Clegg, 1978).

**Discussion.** Final atomic parameters are in Table 1, bond lengths and angles in Table 2.\*

\* Lists of structure factors, anisotropic thermal parameters, H coordinates, bond distances involving H atoms, torsion angles and the packing diagram have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42017 (32 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.