

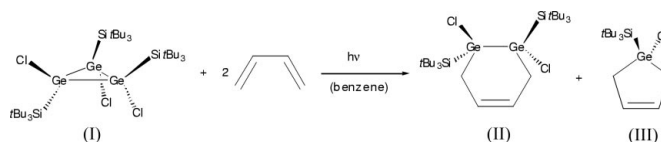
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Dürner<sup>b</sup> and Michael Bolte<sup>b\*</sup><sup>a</sup>Institut für Anorganische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and <sup>b</sup>Institut für Organische Chemie, J. W. Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, GermanyCorrespondence e-mail:  
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## Key indicators

Single-crystal X-ray study  
T = 173 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$   
R factor = 0.027  
wR factor = 0.059  
Data-to-parameter ratio = 28.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*trans*- $\mu$ -But-2-ene-1,4-diyl-dichlorobis-(tri-*tert*-butylsilyl)digermanium(Ge–Ge)The title compound,  $[\text{Ge}_2\text{Cl}_2(\text{C}_4\text{H}_6)(\text{C}_{12}\text{H}_{27}\text{Si})_2]$ , is located on a twofold rotation axis. As a result of the crystal symmetry, the butenyl chain is disordered over two sites.Received 12 August 2003  
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## Comment

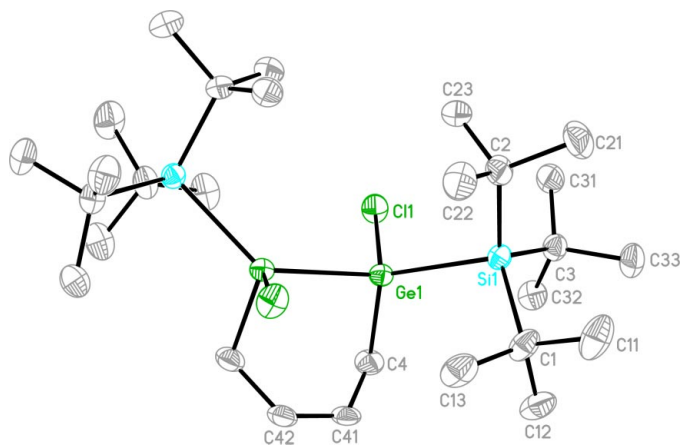
Since cyclotrimeranes can be conveniently transformed into germlyenes and digermenes, we have become interested in the properties of *cis,trans*-cyclotrimerane,  $(\text{Bu}_3\text{SiGeCl})_3$ . In this context, the photolysis of  $(\text{Bu}_3\text{SiGeCl})_3$  is of particular interest to us. Chlorosupersilylgermylene,  $\text{Bu}_3\text{SiGeCl}$ , and *trans*-1,2-dichloro-1,2-disupersilyldigermene,  $\text{Bu}_3\text{SiGeCl}=\text{ClGeSiBu}_3$ , were formed almost quantitatively by photolysis of *cis,trans*-cyclotrimerane  $(\text{Bu}_3\text{SiGeCl})_3$  in benzene. The reaction of chlorosupersilylgermylene,  $\text{Bu}_3\text{SiGeCl}$ , and *trans*-1,2-dichloro-1,2-disupersilyldigermene,  $\text{Bu}_3\text{SiGeCl}=\text{ClGeSiBu}_3$ , with 1,3-butadiene gave the [4+1] and the [4+2] cycloadducts. X-ray quality crystals of the [4+2] cycloadduct, the title compound, (II), were obtained from benzene.



Compound (II) is located on a twofold rotation axis running through the Ge–Ge bond. Unfortunately, this imposed symmetry leads to a disordered butenyl chain. Two different orientations of the central C=C double bond can be found. The geometric parameters (Table 1) are comparable with those of *trans*-( $\mu_2$ -2,3-dimethylbut-2-ene-1,4-diyl)dichloro-disupersilyldigermene (Ichinohe *et al.*, 2000).

## Experimental

A mixture of 1,3-butadiene (0.5 mmol),  $(\text{Bu}_3\text{SiGeCl})_3$  (0.12 mmol), and  $\text{C}_6\text{D}_6$  (0.6 ml) was photolyzed for one week in a sealed NMR tube. The NMR ( $^1\text{H}$ ,  $^{13}\text{C}$  and  $^{29}\text{Si}$ ) spectra showed only the signals from (II) and (III), and thus indicated the formation of the cycloadducts (II) and (III) in quantitative yield. Cycloadducts (II) and (III) were separated by HPLC using hexane as eluent (stationary phase, Macherey–Nagel nucleosil 50  $\text{Å}$ , 5 mm), and showed peaks with retention times of 17.18 min for (II) and 20.07 min for (III). X-ray quality crystals of (II) were grown from a benzene solution at ambient temperature. NMR data for (I), (II) and (III) are listed in Table 2.



**Figure 1**

A perspective view of (II) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Only one of the two orientations of the butenyl chain is shown. H atoms have been omitted for clarity.

#### Crystal data

$[\text{Ge}_2\text{Cl}_2(\text{C}_4\text{H}_6)(\text{C}_{12}\text{H}_{27}\text{Si})_2]$

$M_r = 669.02$

Orthorhombic, *Pbcn*

$a = 25.236$  (1) Å

$b = 9.6427$  (5) Å

$c = 14.0761$  (6) Å

$V = 3425.3$  (3) Å<sup>3</sup>

$Z = 4$

$D_x = 1.297$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

Cell parameters from 25 280

reflections

$\theta = 2.6\text{--}29.2^\circ$

$\mu = 2.00$  mm<sup>-1</sup>

$T = 173$  (2) K

Plate, colourless

$0.17 \times 0.14 \times 0.08$  mm

#### Data collection

Stoe IPDS II two-circle  
diffractometer

$\omega$  scans

Absorption correction: multi-scan  
(*MULABS*; Spek, 1990; Blessing,  
1995)

$T_{\min} = 0.728$ ,  $T_{\max} = 0.857$

47 335 measured reflections

4663 independent reflections

3368 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.061$

$\theta_{\max} = 29.3^\circ$

$h = -34 \rightarrow 34$

$k = -13 \rightarrow 13$

$l = -19 \rightarrow 19$

#### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.059$

$S = 0.87$

4663 reflections

164 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0338P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$

$\Delta\rho_{\max} = 0.60$  e Å<sup>-3</sup>

$\Delta\rho_{\min} = -0.50$  e Å<sup>-3</sup>

Extinction correction: *SHELXL97*  
(Sheldrick, 1997)

Extinction coefficient: 0.00146 (12)

**Table 1**

Selected geometric parameters (Å, °).

|         |             |                      |            |
|---------|-------------|----------------------|------------|
| Ge1—C4  | 2.0062 (18) | Ge1—Ge1 <sup>i</sup> | 2.4994 (4) |
| Ge1—Cl1 | 2.2214 (5)  | C4—C41               | 1.445 (4)  |
| Ge1—Si1 | 2.4613 (5)  | C41—C42              | 1.325 (5)  |

Symmetry code: (i)  $1 - x, y, \frac{1}{2} - z$ .

**Table 2**

Selected NMR data for (I), (II) and (III).

Solvent is C<sub>6</sub>D<sub>6</sub> in all cases;  $\delta$  values are in p.p.m.

| Compound | <sup>1</sup> H NMR                          | <sup>13</sup> C NMR          | <sup>29</sup> Si NMR                        |
|----------|---|------------------------------|---|
| (I)      | 1.39 (s, 2Si <sup>i</sup> Bu <sub>3</sub> ) | 25.6 (s, CMe <sub>3</sub> )  | 46.5 (s, Si <sup>i</sup> Bu <sub>3</sub> )  |
|          | 1.40 (s, 2Si <sup>i</sup> Bu <sub>3</sub> ) | 26.1 (s, 2CMe <sub>3</sub> ) | 48.4 (s, 2Si <sup>i</sup> Bu <sub>3</sub> ) |
|          |   | 31.7 (s, CMe <sub>3</sub> )  |   |
| (II)     | 1.31 (s, 2Si <sup>i</sup> Bu <sub>3</sub> ) | 25.1 (s, CMe <sub>3</sub> )  | 37.1 (s, 2Si <sup>i</sup> Bu <sub>3</sub> ) |
|          | 2.53 (s, 2CH <sub>2</sub> )                 | 31.5 (s, CMe <sub>3</sub> )  |   |
|          | 5.83 (m, 2 =CH)                             | 27.8 (s, 2CH <sub>2</sub> )  |   |
| (III)    |   | 125.3 (s, 2 =CH)             |   |
|          | 1.14 (s, Si <sup>i</sup> Bu <sub>3</sub> )  | 23.9 (s, CMe <sub>3</sub> )  | 19.2 (s, Si <sup>i</sup> Bu <sub>3</sub> )  |
|          | 2.07 (s, 2CH <sub>2</sub> )                 | 30.8 (s, CMe <sub>3</sub> )  |   |
|          | 6.02 (m, 2 =CH)                             | 29.2 (s, 2CH <sub>2</sub> )  |   |
|          |   | 130.4 (s, 2 =CH)             |   |

The two central atoms of the butenyl chain are disordered over two sites. Therefore, they were refined with site occupancy factors of 0.5. H atoms were refined with fixed individual displacement parameters [ $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$  or  $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{C}_{\text{methyl}})$ ], using a riding model with methyl C—H = 0.98, methylene C—H = 0.99 and C( $sp^2$ )—H = 0.95 Å.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *SHELXL97*.

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