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3,4-Dimethyl-1,1-bis[4-(trifluoromethyl)phenyl]-2,5-dihydro-1*H*-germole

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3,4-Dimethyl-1,1-bis[4-(trifluoromethyl)-phenyl]-2,5-dihydro-1H-germole

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Key indicators

Single-crystal X-ray study

$T = 173\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$

Disorder in main residue

R factor = 0.040

w R factor = 0.109

Data-to-parameter ratio = 24.9

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

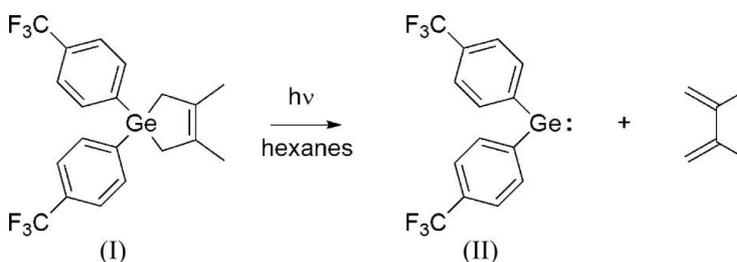
The internal C—Ge—C bond angle in the germacyclopentene ring of the title compound, $\text{C}_{20}\text{H}_{18}\text{F}_6\text{Ge}$ or $[\text{Ge}(\text{C}_6\text{H}_9\text{F}_3)(\text{C}_7\text{H}_4\text{F}_3)_2]$, is $93.17(9)^\circ$.

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Comment

Aryl-substituted 1-germacyclopent-3-ene derivatives are useful for the photochemical generation of a variety of different germynes, the properties and reactivities of which can be studied *via* laser flash photolysis methods (Leigh *et al.*, 2006). For example, photolysis of the title compound, (I), in a hydrocarbon solution efficiently ($\Phi = 0.55 \pm 0.12$) and cleanly (>90% chemical yield) generates the diarylgermylene (II) and 2,3-dimethyl-1,3-butadiene (Huck & Leigh, 2007).



In (I) (Fig. 1), the endocyclic C—Ge—C bond angle of 93.17° (Fig. 2) is slightly larger than the calculated [(B3LYP/6-311G(*d,p*))] angle in the parent 1,1-diphenyl analogue of 91.8° (Lemierre *et al.*, 2004). This ring strain may partially explain the high efficiency of germylene extrusion.

The five-membered germacyclopentene ring is not planar; instead the C atoms in the ring (C1–C4) bend towards one of the aromatic rings (C11), making an angle of $12(1)^\circ$ with the plane defined by atoms C1—Ge—C4 (Fig. 1). The endocyclic Ge—C bond distances are essentially identical to the exocyclic

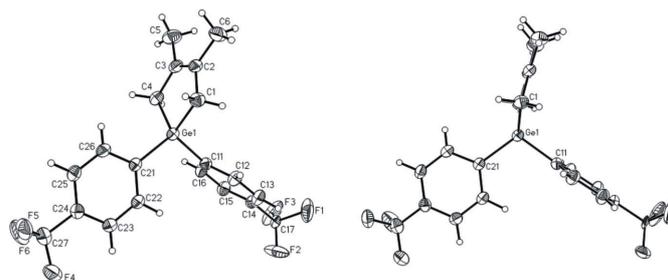


Figure 1

The molecular structure of (I), drawn with 50% probability ellipsoids (two different views are shown). Atoms C17, F1, F2 and F3 are disordered but the disorder is not shown for clarity.

Ge—C(aryl) bond distances, and the endocyclic C=C bond distance of 1.345 (3) Å is quite similar to those found in cyclopentene derivatives (Guan *et al.*, 2005). The bond distances and angles for (I) are comparable to those found in the considerably more structurally complex 1-germacyclopent-3-ene derivatives that have been previously characterized by crystallographic methods (Fröhlich *et al.*, 1994; Fukaya *et al.*, 2001; Lei *et al.*, 1990, 1991; Stender *et al.*, 2002; Sugiyama *et al.*, 2006).

The atoms of one trifluoromethyl group (C17, F1, F2, and F3) pack in a staggered fashion with the same trifluoromethyl group of a neighbouring molecule, resulting in substantial disorder (Fig. 2).

Experimental

Compound (I) was synthesized as described elsewhere (Huck & Leigh, 2007). Single crystals of (I) suitable for X-ray diffraction were obtained by slow evaporation of a hexane solution at room temperature. X-ray crystallographic analyses were performed at the McMaster Analytical X-Ray (MAX) Diffraction Facility. The data were collected at 173 K on a single crystal coated with Paratone oil and mounted in a cryoloop.

Crystal data

[Ge(C ₆ H ₁₀)(C ₇ H ₄ F ₃) ₂]	$V = 1880.4 (10) \text{ \AA}^3$
$M_r = 444.93$	$Z = 4$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.535 (2) \text{ \AA}$	$\mu = 1.69 \text{ mm}^{-1}$
$b = 18.367 (6) \text{ \AA}$	$T = 173 (2) \text{ K}$
$c = 15.674 (5) \text{ \AA}$	$0.20 \times 0.20 \times 0.08 \text{ mm}$
$\beta = 91.726 (6)^\circ$	

Data collection

Bruker APEXII CCD area-detector diffractometer	43214 measured reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1997)	8127 independent reflections
$T_{\min} = 0.733$, $T_{\max} = 0.87$	5493 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.059$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.109$	$\Delta\rho_{\text{max}} = 0.55 \text{ e \AA}^{-3}$
$S = 1.03$	$\Delta\rho_{\text{min}} = -0.41 \text{ e \AA}^{-3}$
8127 reflections	
326 parameters	
42 restraints	

The two positions of the disordered trifluoromethyl group were determined from the difference map and refined anisotropically with occupancies of 0.54 (2) (C17, F1, F2 and F3) and 0.46 (2) (C17A, F1A, F2A, and F3A). H atoms were located in a difference map and refined isotropically, except for the positions of the H atoms bonded to C5, C6 and C25, which were calculated (C—H = 0.95 or 0.98 Å) and refined as riding on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The position of H25 was refined using a riding model (C—H = 0.95 Å) but the U_{iso} was allowed to refine.

Data collection: APEXII (Bruker, 2006); cell refinement: APEXII; data reduction: SAINT (Sheldrick, 2003); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics:

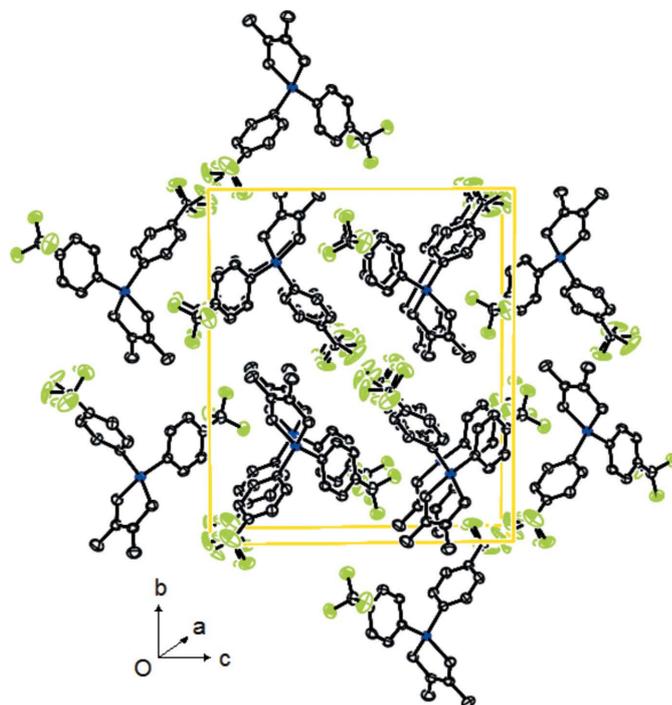


Figure 2

Packing of (I) viewed down the a axis. H atoms have been omitted for clarity. Both disorder components are shown.

ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXTL (Sheldrick, 2000).

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