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2,2-Diphenyl-1,3,2-dithiagermole-4,5-dicarbonitrile

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.045
wR factor = 0.100
Data-to-parameter ratio = 21.0

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

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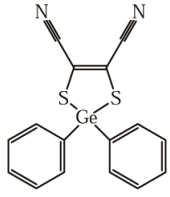
The title compound, $(C_6H_5)_2GeS_2C_2(CN)_2$, crystallizes with two molecules in the asymmetric unit. Each five-membered ring has an envelope conformation.

Comment

The X-ray investigation of the title compound, (I), was undertaken as part of our study of the structure and conformation of new germanium complexes.



(I)

In the title compound, (I), the average Ge—S and Ge—C bond lengths are 2.2530 (13) and 1.926 (5) Å, respectively, in agreement with the literature values (Allen *et al.*, 1987; Liu *et al.*, 2002).

The title compound crystallizes with two molecules (denoted *A* and *B*) in the asymmetric unit. In molecule *A*, the dihedral angle between the five-membered ring and phenyl ring C1*A*–C6*A* is 82.4 (1)°, that between the five-membered ring and phenyl ring C7*A*–C12*A* is 66.1 (1)° and the angle between the two phenyl rings is 55.8 (2)°. The corresponding angles in molecule *B* are 73.1 (1), 79.1 (1) and 58.9 (1)°, respectively. Each five-membered ring has an envelope conformation, the flaps Ge1*A* and Ge1*B* being displaced from the planes of the other ring atoms by 0.4115 (4) and 0.3467 (5) Å, respectively. In 2,2-dimethyl-[1,3,2]dithiagermole-4,5-dicarbonitrile, $Me_2GeS_2C_2(CN)_2$ (Ewert, 1983), the five-membered ring also has an envelope conformation.

Experimental

Under an argon atmosphere, a stirred aqueous solution of $Na_2S_2C_2(CN)_2$ was added dropwise to a solution of Ph_2GeBr_2 in CH_3Cl (1:1). This mixture was stirred at room temperature for 1 h. After filtration, the coloured solid was dried over anhydrous $MgSO_4$. The solvent was then evaporated and the product crystallized from absolute toluene. Analysis calculated for $C_{16}H_{10}GeN_2S_2$: C 52.10, H 2.82, Ge 19.00, S 17.39%; found: C 52.32, H 2.74, Ge, 19.78, S 17.47%. M.p.: 416 K. IR, ν (cm^{-1}): 2220 (C—N), 700 (C—S), 405 (Ge—S), 310 (Ge—C). ^{1}H NMR (δ, p.p.m., 200 MHz): 7.56. ^{13}C NMR (δ, p.p.m., 200 MHz): 132.25 (α), 133.84 (β), 129.60 (γ), 132.44 (δ), 119.63 (C═C), 112.96 (CN).

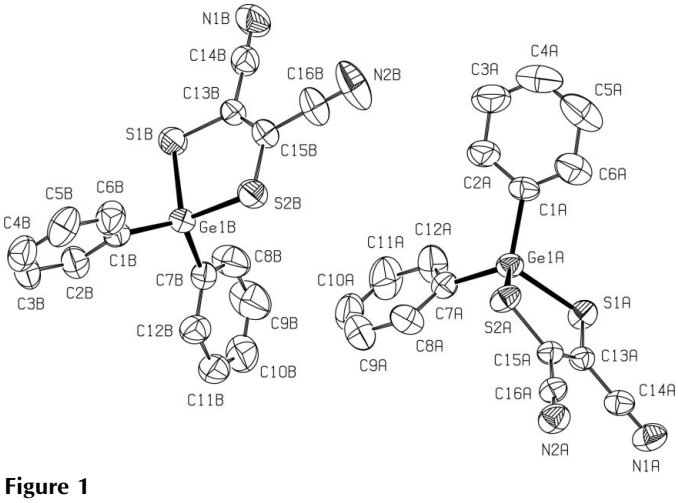


Figure 1
Views of the two independent molecules of (I), with the atom-numbering scheme and displacement ellipsoids drawn at the 50% probability level. H atoms have been omitted.

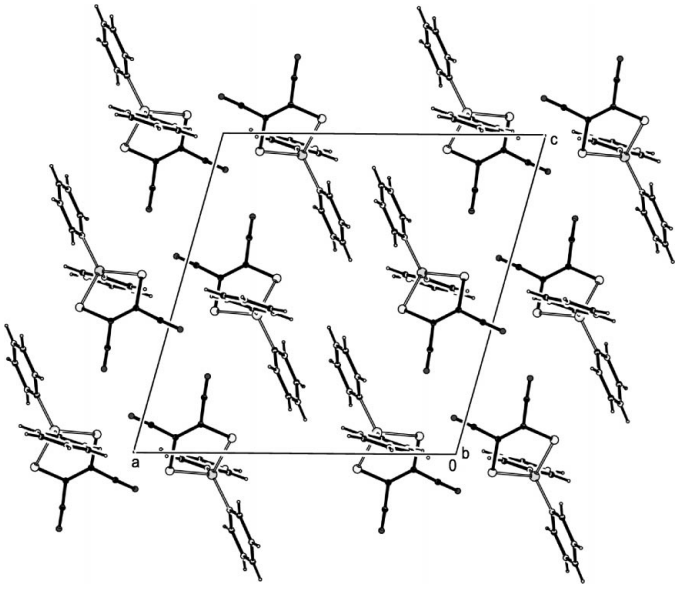


Figure 2
A packing diagram of the title compound, (I), viewed along the *b* axis.

Crystal data

$C_{16}H_{10}GeN_2S_2$
M_r = 367.01
Monoclinic, $P2_1/c$
a = 15.3121 (11) Å
b = 14.0064 (10) Å
c = 15.7730 (11) Å
β = 105.3900 (10)°
V = 3261.5 (4) Å^3
Z = 8
D_x = 1.495 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5356 reflections
θ = 2.4–24.7°
μ = 2.13 mm^{-1}
T = 293 K
Slab, light brown
0.40 × 0.40 × 0.20 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.483, T_{max} = 0.676
19 939 measured reflections
7949 independent reflections
4421 reflections with $I > 2\sigma(I)$
R_{int} = 0.046
θ_{max} = 28.3°
$h = -16 \rightarrow 20$
$k = -18 \rightarrow 13$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.100
S = 1.04
7949 reflections
379 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0286P)^2 + 1.6083P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ge1*A*—S1*A*	2.2484 (11)	S1*A*—C13*A*	1.742 (3)
Ge1*A*—S2*A*	2.2616 (12)	S2*A*—C15*A*	1.751 (3)
Ge1*A*—C1*A*	1.926 (3)	S1*B*—C13*B*	1.737 (4)
Ge1*A*—C7*A*	1.931 (4)	S2*B*—C15*B*	1.742 (5)
Ge1*B*—C1*B*	1.922 (3)	N1*A*—C14*A*	1.136 (6)
Ge1*B*—C7*B*	1.926 (5)	N2*A*—C16*A*	1.135 (6)
Ge1*B*—S2*B*	2.2480 (11)	N1*B*—C14*B*	1.123 (7)
Ge1*B*—S1*B*	2.2539 (13)	N2*B*—C16*B*	1.136 (6)
S1*A*—Ge1*A*—S2*A*	95.16 (4)	Ge1*A*—C7*A*—C8*A*	121.6 (3)
S1*A*—Ge1*A*—C1*A*	112.76 (11)	Ge1*A*—C7*A*—C12*A*	121.4 (3)
S1*A*—Ge1*A*—C7*A*	113.55 (11)	S1*A*—C13*A*—C14*A*	114.1 (3)
S2*A*—Ge1*A*—C1*A*	110.94 (11)	S1*A*—C13*A*—C15*A*	124.8 (2)
S2*A*—Ge1*A*—C7*A*	108.90 (14)	N1*A*—C14*A*—C13*A*	178.2 (5)
C1*A*—Ge1*A*—C7*A*	113.94 (16)	S2*A*—C15*A*—C16*A*	114.5 (2)
S1*B*—Ge1*B*—S2*B*	95.40 (4)	S2*A*—C15*A*—C13*A*	124.3 (2)
S1*B*—Ge1*B*—C1*B*	109.02 (11)	N2*A*—C16*A*—C15*A*	177.6 (4)
S1*B*—Ge1*B*—C7*B*	109.22 (14)	Ge1*B*—C1*B*—C2*B*	120.0 (3)
S2*B*—Ge1*B*—C1*B*	114.03 (12)	Ge1*B*—C1*B*—C6*B*	121.9 (3)
S2*B*—Ge1*B*—C7*B*	111.25 (13)	Ge1*B*—C7*B*—C8*B*	119.6 (3)
C1*B*—Ge1*B*—C7*B*	115.88 (17)	Ge1*B*—C7*B*—C12*B*	121.4 (4)
Ge1*A*—S1*A*—C13*A*	96.94 (11)	S1*B*—C13*B*—C14*B*	114.0 (3)
Ge1*A*—S2*A*—C15*A*	96.45 (11)	S1*B*—C13*B*—C15*B*	124.8 (3)
Ge1*B*—S1*B*—C13*B*	96.71 (12)	N1*B*—C14*B*—C13*B*	178.2 (5)
Ge1*B*—S2*B*—C15*B*	96.78 (15)	S2*B*—C15*B*—C13*B*	124.6 (3)
Ge1*A*—C1*A*—C2*A*	119.4 (3)	S2*B*—C15*B*—C16*B*	115.3 (3)
Ge1*A*—C1*A*—C6*A*	121.2 (3)	N2*B*—C16*B*—C15*B*	179.3 (5)
Ge1*A*—S1*A*—C13*A*—C14*A*	171.5 (2)	Ge1*B*—S1*B*—C13*B*—C15*B*	−8.9 (3)
Ge1*A*—S1*A*—C13*A*—C15*A*	−9.3 (3)	Ge1*B*—S1*B*—C13*B*—C14*B*	174.4 (3)
Ge1*A*—S2*A*—C15*A*—C13*A*	11.8 (3)	Ge1*B*—S2*B*—C15*B*—C13*B*	9.0 (4)
Ge1*A*—S2*A*—C15*A*—C16*A*	−171.7 (2)	Ge1*B*—S2*B*—C15*B*—C16*B*	−170.5 (3)

All H atoms were placed in geometrically idealized positions, with C—H distances of 0.93 Å, and refined in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}$ of their parent atoms.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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