IUCrData

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1,3,7,9-Tetrakis[2,6-bis(1-methylethyl)phenyl]-2,2,8,8-tetraphenyl-5,10-dithia-1,3,7,9-tetraaza-2,8-disila-4,6-digermadispiro[3.1.3.1]decane acetonitrile monosolvate

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Keywords: germanium(IV); sulfide; bis(amido)-silane; germanethione dimer; crystal structure.

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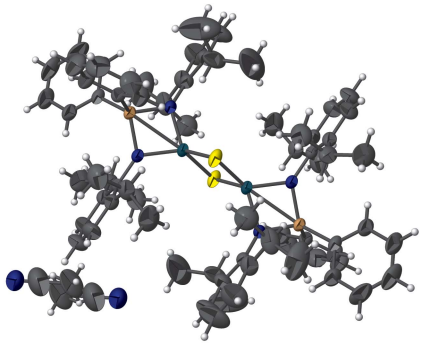
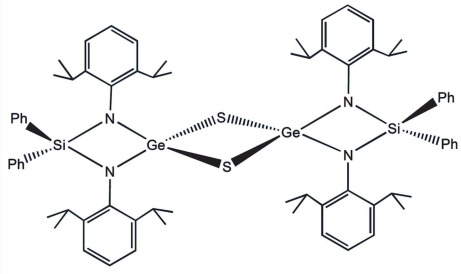
Structural data: full structural data are available from iucrdata.iucr.org

In the compound, $[\eta^2(N,N)\text{-}Ph_2Si(NDipp)_2Ge(\mu\text{-}S)]_2\cdot CH_3CN$ (Dipp = 2,6-$iPr_2C_6H_3$), $C_{72}H_{88}Ge_2N_4S_2Si_2\cdot CH_2CN$, the dimeric germanethione complex lies about an inversion centre with the two Ge^{IV} atoms bridged by two sulfide ligands forming the central Ge_2S_2 ring. Each Ge^{IV} atom is also coordinated by two N atoms from the bulky bis(amido)silane ligands in a slightly distorted tetrahedral coordination geometry. An acetonitrile solvent molecule, disordered about an inversion centre, is also present.

3D view



Chemical scheme



Structure description

Reaction of the arylamido germylene $[Ph_2Si(DippN)_2Ge:]$ with elemental sulfur gave the new germanethione dimer 1,3,7,9-tetrakis[2,6-bis(1-methylethyl)phenyl]-2,2,8,8-tetrakis(1-phenyl)-5,10-dithia-1,3,7,9-tetraaza-2,8-disila-4,6-digermadispiro[3.1.3.1]decane, $[\eta^2(N,N)\text{-}Ph_2Si(DippN)_2Ge(\mu\text{-}S)]_2$ (Dipp = 2,6 − $iPr_2C_6H_3$) which crystallizes as an acetonitrile solvate. A related compound $[\eta^2(N,N)\text{-}Me_2Si(DippN)_2Ge(\mu\text{-}O)]_2$, was prepared previously by a similar process involving the direct reaction of $[\eta^2(N,N)\text{-}Me_2Si(DippN)_2Ge:]$, with dioxygen (Yang *et al.*, 2012).

The Ge^{IV} complex lies about an inversion centre with the two Ge atoms bridged by two sulfido ligands forming the central Ge_2S_2 ring. Each Ge atom is also coordinated by two nitrogen atoms from the bulky bis(amido)silane ligands in a slightly distorted tetrahedral coordination geometry. The molecular structure is dimeric in the solid state and contains planar NSiNGe and Ge_2S_2 arrays that are inclined to one another by 89.42 (8)°, with average endocyclic Ge—S and Ge—N bond lengths of 2.2320 (9) and 1.845 (3) Å,

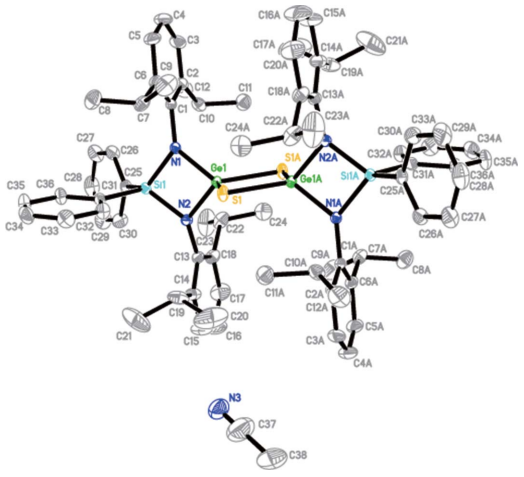


Figure 1
The molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level. Only one component of the disordered acetonitrile solvent molecule is shown and H atoms are omitted for clarity. Atoms labelled A are related to other atoms by the symmetry operation $-x + 1, -y + 1, z$.

respectively (Fig. 1). The Ge—S bond lengths of 2.2590 (10) and 2.2050 (9) Å are reasonably similar to those found in $[\eta^2(N,N)$-*i*Pr$_2$Si(DippN)$_2$Ge(μ-S)]$_2$ [Ge—S = 2.1992 (3) and 2.2577 (3) Å; Al-Rafia *et al.*, 2010] and also those found in other closely related bis-sulfide derivatives (Sen *et al.*, 2011; Bazinet *et al.*, 2001; Wegner *et al.*, 2000). An acetonitrile solvent molecule, disordered about an inversion centre such that each disorder component has equal occupancy, is found in the crystal lattice.

Synthesis and crystallization

A solution of [Ph$_2$Si(DippN)$_2$Ge:] (0.600 g, 1.0 mmol) (Yang *et al.*, 2012) in 10 ml dry ether was slowly added drop-wise to a stirred solution of sulfur (0.037 g, 1.2 mmol) in dry ether (30 ml) at room temperature. After the mixture was stirred for 36 h at room temperature, the clear yellow solution was filtered through Celite. The volatile components of the filtrate were removed under reduced pressure to afford the title complex as colorless crystals at −30°C (0.45 g, 71%). M.p. 320 K (decomposition). ^{1}H NMR (298 K, CDCl$_3$): δ = 7.29–6.96 (*m*, 24 H, Ar-*H*), 3.64 (*m*, 8 H, Me$_2$C*H*), 0.98 (*d*, 24 H, C*H*$_3$), 0.55 (*d*, 24 H, C*H*$_3$) p.p.m.; ^{13}C{^{1}H} NMR (298 K, CDCl$_3$): δ = 26.0 (*s*, (*C*H$_3$)$_2$C), 28.3 (*s*, (CH$_3$)$_2$*C*), 123.7, 125.1, 127.3, 130.0, 134.7, 135.5, 136.6, 147.6 (8 *s*, *C* for Ph ring) p.p.m. Elemental analysis (%): Calculated for C$_{72}$H$_{88}$Ge$_2$N$_4$S$_2$Si$_4$ (the solvent molecule was removed under high vacuum): C 67.76, H 6.90, N 4.39; found: C 67.74, H 6.88, N 4.38.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1. A difference Fourier synthesis following location of all the non-hydrogen atoms of the germanethione complex revealed additional peaks consistent with the presence of an acetonitrile solvent molecule. This solvent molecule was disordered about an inversion centre and was refined with equal occupancy for the atoms of each disorder component.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{72}H_{88}Ge_2N_4S_2Si_2\cdot C_2H_3N$
M_r	1316.00
Crystal system, space group	Triclinic, $P\overline{1}$
Temperature (K)	153
a, b, c (Å)	11.0447 (5), 13.4587 (6), 13.9291 (6)
α, β, γ (°)	64.297 (4), 82.371 (4), 85.629 (4)
V (Å^3)	1848.74 (14)
Z	1
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.94
Crystal size (mm)	0.15 × 0.13 × 0.11
Data collection	
Diffractometer	Agilent SuperNova Dual Source diffractometer with an Eos detector
Absorption correction	Multi-scan (*CrysAlis PRO*; Agilent, 2012)
T_{min}, T_{max}	0.871, 0.903
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	13757, 7010, 5534
R_{int}	0.037
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.609
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.046, 0.138, 1.08
No. of reflections	7010
No. of parameters	405
No. of restraints	5
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.75, −0.38

Computer programs: *CrysAlis PRO* (Agilent, 2012), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *SHELXTL* (Sheldrick, 2008), *publCIF* (Westrip, 2010).

Acknowledgements

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References

Agilent (2012). *CrysAlis PRO*. Agilent Technologies, Yarnton, England.
Al-Rafia, S. M. I., Lummis, P. A., Ferguson, M. J., McDonald, R. & Rivard, E. (2010). *Inorg. Chem.* **49**, 9709–9717.
Bazinet, P., Yap, G. P. A. & Richeson, D. S. (2001). *J. Am. Chem. Soc.* **123**, 11162–11167.
Sen, S. S., Ghadwal, R. S., Kratzert, D., Stern, D., Roesky, H. W. & Stalke, D. (2011). *Organometallics*, **30**, 1030–1033.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.
Wegner, G. L., Jockisch, A., Schier, A. & Schmidbaur, H. (2000). *Z. Naturforsch. Teil B*, **55**, 347–351.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Yang, D., Guo, J., Wu, H., Ding, Y. & Zheng, W. (2012). *Dalton Trans.* **41**, 2187–2194.

full crystallographic data

IUCrData (2016). **1**, x160299 [https://doi.org/10.1107/S2414314616002996]

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Crystal data

$C_{72}H_{88}Ge_2N_4S_2Si_2·C_2H_3N$
M_r = 1316.00
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 11.0447 (5) Å
b = 13.4587 (6) Å
c = 13.9291 (6) Å
α = 64.297 (4)°
β = 82.371 (4)°
γ = 85.629 (4)°
V = 1848.74 (14) Å^3
Z = 1
$F(000)$ = 694
D_x = 1.182 Mg m^{-3}
Melting point: 320 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 3142 reflections
θ = 2.2–27.0°
μ = 0.94 mm^{-1}
T = 153 K
Block, colorless
0.15 × 0.13 × 0.11 mm

Data collection

Agilent SuperNova Dual Source diffractometer with an Eos detector
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (CrysAlis PRO; Agilent, 2012)
T_{min} = 0.871, T_{max} = 0.903
13757 measured reflections
7010 independent reflections
5534 reflections with $I > 2\sigma(I)$
R_{int} = 0.037
θ_{max} = 25.7°, θ_{min} = 2.7°
h = −13→13
k = −16→16
l = −14→16
5839 standard reflections every 1 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.138
S = 1.08
7010 reflections
405 parameters
5 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 1.4505P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.5999 (3)	0.8105 (3)	0.8258 (3)	0.0343 (8)	
C2	0.7166 (4)	0.8406 (3)	0.8332 (3)	0.0432 (9)	
C3	0.7231 (5)	0.9309 (4)	0.8568 (4)	0.0664 (13)	
H3	0.7992	0.9523	0.8620	0.080*	
C4	0.6212 (6)	0.9890 (4)	0.8726 (4)	0.0758 (15)	
H4	0.6284	1.0488	0.8880	0.091*	
C5	0.5104 (5)	0.9585 (4)	0.8656 (4)	0.0649 (13)	
H5	0.4415	0.9978	0.8770	0.078*	
C6	0.4960 (4)	0.8698 (3)	0.8419 (3)	0.0455 (9)	
C7	0.3688 (4)	0.8415 (3)	0.8343 (3)	0.0526 (10)	
H7	0.3753	0.7737	0.8239	0.063*	
C8	0.3135 (5)	0.9325 (4)	0.7373 (4)	0.0771 (15)	
H8A	0.3124	1.0014	0.7425	0.116*	
H8B	0.2316	0.9138	0.7364	0.116*	
H8C	0.3621	0.9390	0.6724	0.116*	
C9	0.2836 (5)	0.8191 (5)	0.9380 (5)	0.0907 (19)	
H9A	0.2061	0.7952	0.9328	0.136*	
H9B	0.2721	0.8855	0.9481	0.136*	
H9C	0.3195	0.7627	0.9979	0.136*	
C10	0.8328 (4)	0.7778 (4)	0.8211 (3)	0.0529 (10)	
H10	0.8167	0.7357	0.7819	0.063*	
C11	0.8684 (5)	0.6954 (4)	0.9310 (4)	0.0730 (14)	
H11A	0.8005	0.6490	0.9711	0.109*	
H11B	0.8902	0.7347	0.9693	0.109*	
H11C	0.9368	0.6507	0.9216	0.109*	
C12	0.9418 (5)	0.8525 (5)	0.7581 (5)	0.0852 (17)	
H12A	1.0091	0.8086	0.7455	0.128*	
H12B	0.9655	0.8884	0.7988	0.128*	
H12C	0.9187	0.9069	0.6907	0.128*	
C13	0.6486 (4)	0.4640 (3)	0.7428 (3)	0.0407 (8)	
C14	0.5671 (4)	0.3995 (4)	0.7257 (4)	0.0573 (11)	
C15	0.6130 (6)	0.3145 (5)	0.7004 (6)	0.0928 (19)	
H15	0.5597	0.2724	0.6876	0.111*	
C16	0.7335 (8)	0.2918 (6)	0.6940 (7)	0.117 (3)	
H16	0.7626	0.2342	0.6768	0.140*	

C17	0.8142 (6)	0.3521 (5)	0.7124 (5)	0.0923 (19)	
H17	0.8972	0.3347	0.7076	0.111*	
C18	0.7741 (4)	0.4396 (3)	0.7384 (4)	0.0558 (11)	
C19	0.4309 (5)	0.4185 (5)	0.7355 (5)	0.0757 (15)	
H19	0.4156	0.4803	0.7553	0.091*	
C20	0.3639 (9)	0.3199 (7)	0.8245 (8)	0.170 (5)	
H20A	0.3710	0.2591	0.8054	0.254*	
H20B	0.2792	0.3393	0.8342	0.254*	
H20C	0.3993	0.2992	0.8901	0.254*	
C21	0.3781 (7)	0.4528 (9)	0.6297 (7)	0.151 (4)	
H21A	0.4010	0.3988	0.6024	0.227*	
H21B	0.4095	0.5231	0.5789	0.227*	
H21C	0.2907	0.4580	0.6411	0.227*	
C22	0.8674 (4)	0.4998 (4)	0.7626 (4)	0.0694 (14)	
H22	0.8293	0.5689	0.7602	0.083*	
C23	0.9825 (6)	0.5286 (7)	0.6815 (6)	0.135 (3)	
H23A	1.0348	0.4643	0.6977	0.203*	
H23B	1.0252	0.5858	0.6857	0.203*	
H23C	0.9596	0.5535	0.6105	0.203*	
C24	0.9062 (5)	0.4330 (5)	0.8741 (5)	0.0804 (15)	
H24A	0.9575	0.4766	0.8906	0.121*	
H24B	0.9505	0.3679	0.8765	0.121*	
H24C	0.8350	0.4121	0.9257	0.121*	
C25	0.7362 (3)	0.7545 (3)	0.5812 (3)	0.0386 (8)	
C26	0.7889 (4)	0.8533 (4)	0.5569 (3)	0.0545 (11)	
H26	0.7620	0.8915	0.5978	0.065*	
C27	0.8806 (5)	0.8973 (5)	0.4733 (4)	0.0737 (15)	
H27	0.9140	0.9646	0.4584	0.088*	
C28	0.9219 (5)	0.8424 (5)	0.4131 (4)	0.0829 (17)	
H28	0.9851	0.8708	0.3581	0.100*	
C29	0.8699 (6)	0.7446 (5)	0.4338 (4)	0.0883 (19)	
H29	0.8971	0.7076	0.3918	0.106*	
C30	0.7771 (5)	0.7006 (4)	0.5170 (3)	0.0645 (13)	
H30	0.7420	0.6347	0.5299	0.077*	
C31	0.4652 (3)	0.7543 (3)	0.6081 (3)	0.0380 (8)	
C32	0.3480 (4)	0.7169 (4)	0.6525 (3)	0.0536 (10)	
H32	0.3362	0.6614	0.7219	0.064*	
C33	0.2480 (4)	0.7609 (4)	0.5951 (4)	0.0670 (13)	
H33	0.1702	0.7349	0.6262	0.080*	
C34	0.2645 (5)	0.8431 (4)	0.4920 (4)	0.0695 (14)	
H34	0.1976	0.8722	0.4536	0.083*	
C35	0.3779 (5)	0.8816 (4)	0.4464 (4)	0.0703 (14)	
H35	0.3886	0.9373	0.3770	0.084*	
C36	0.4777 (4)	0.8376 (3)	0.5037 (3)	0.0528 (10)	
H36	0.5550	0.8643	0.4716	0.063*	
C38	−0.0273 (16)	−0.0191 (14)	0.9955 (15)	0.128 (6)	0.50
H38A	−0.1136	−0.0196	0.9925	0.193*	0.50
H38B	0.0120	−0.0783	0.9814	0.193*	0.50

H38C	−0.0128	−0.0286	1.0654	0.193*	0.50
Ge1	0.55890 (3)	0.57094 (3)	0.88889 (3)	0.03020 (13)	
N1	0.5904 (3)	0.7178 (2)	0.8016 (2)	0.0311 (6)	
N2	0.6031 (3)	0.5576 (2)	0.7626 (2)	0.0343 (6)	
N3	0.1041 (12)	0.1467 (8)	0.8636 (8)	0.114 (4)	0.50
S1	0.36302 (8)	0.52913 (8)	0.95938 (7)	0.0412 (2)	
Si1	0.60204 (9)	0.69914 (7)	0.68380 (7)	0.0309 (2)	
C37	0.0482 (11)	0.0770 (9)	0.9190 (9)	0.158 (9)	0.50

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.045 (2)	0.0297 (17)	0.0255 (16)	−0.0063 (15)	−0.0003 (14)	−0.0094 (13)
C2	0.051 (2)	0.044 (2)	0.0345 (19)	−0.0137 (17)	−0.0031 (17)	−0.0147 (16)
C3	0.081 (4)	0.063 (3)	0.067 (3)	−0.026 (3)	−0.012 (3)	−0.033 (2)
C4	0.106 (5)	0.057 (3)	0.081 (4)	−0.011 (3)	−0.008 (3)	−0.046 (3)
C5	0.087 (4)	0.048 (3)	0.067 (3)	0.003 (2)	0.000 (3)	−0.034 (2)
C6	0.058 (3)	0.037 (2)	0.0371 (19)	−0.0023 (17)	0.0035 (17)	−0.0134 (16)
C7	0.046 (3)	0.041 (2)	0.062 (3)	0.0037 (18)	0.0064 (19)	−0.0183 (19)
C8	0.066 (4)	0.063 (3)	0.091 (4)	0.009 (3)	−0.019 (3)	−0.021 (3)
C9	0.079 (4)	0.081 (4)	0.092 (4)	−0.005 (3)	0.036 (3)	−0.030 (3)
C10	0.045 (2)	0.066 (3)	0.052 (2)	−0.016 (2)	−0.0043 (19)	−0.027 (2)
C11	0.066 (3)	0.071 (3)	0.080 (3)	0.000 (3)	−0.024 (3)	−0.026 (3)
C12	0.058 (3)	0.113 (5)	0.085 (4)	−0.036 (3)	0.006 (3)	−0.041 (3)
C13	0.050 (2)	0.0343 (19)	0.0366 (19)	−0.0032 (16)	0.0024 (16)	−0.0155 (15)
C14	0.066 (3)	0.052 (2)	0.063 (3)	−0.009 (2)	0.000 (2)	−0.035 (2)
C15	0.104 (5)	0.081 (4)	0.130 (5)	0.001 (3)	−0.014 (4)	−0.078 (4)
C16	0.124 (7)	0.102 (5)	0.170 (8)	0.028 (5)	−0.019 (5)	−0.105 (6)
C17	0.073 (4)	0.089 (4)	0.126 (5)	0.025 (3)	−0.004 (4)	−0.063 (4)
C18	0.050 (3)	0.048 (2)	0.065 (3)	0.0047 (19)	0.001 (2)	−0.023 (2)
C19	0.065 (3)	0.091 (4)	0.098 (4)	−0.026 (3)	0.003 (3)	−0.065 (3)
C20	0.151 (8)	0.147 (7)	0.220 (11)	−0.093 (6)	0.100 (8)	−0.107 (8)
C21	0.083 (5)	0.271 (11)	0.178 (8)	0.031 (6)	−0.054 (5)	−0.162 (9)
C22	0.041 (3)	0.057 (3)	0.097 (4)	0.002 (2)	−0.007 (2)	−0.021 (3)
C23	0.058 (4)	0.160 (7)	0.119 (6)	−0.027 (4)	0.002 (4)	0.005 (5)
C24	0.060 (3)	0.090 (4)	0.093 (4)	0.004 (3)	−0.009 (3)	−0.041 (3)
C25	0.037 (2)	0.042 (2)	0.0282 (17)	−0.0048 (15)	0.0030 (14)	−0.0084 (15)
C26	0.056 (3)	0.062 (3)	0.040 (2)	−0.023 (2)	0.0070 (19)	−0.0171 (19)
C27	0.064 (3)	0.091 (4)	0.052 (3)	−0.044 (3)	0.007 (2)	−0.015 (3)
C28	0.053 (3)	0.112 (5)	0.050 (3)	−0.017 (3)	0.020 (2)	−0.008 (3)
C29	0.097 (5)	0.101 (4)	0.051 (3)	0.011 (4)	0.028 (3)	−0.030 (3)
C30	0.080 (4)	0.060 (3)	0.047 (2)	−0.011 (2)	0.020 (2)	−0.023 (2)
C31	0.040 (2)	0.0387 (19)	0.0339 (18)	−0.0034 (15)	−0.0050 (15)	−0.0139 (15)
C32	0.042 (2)	0.059 (3)	0.050 (2)	−0.0059 (19)	−0.0017 (19)	−0.015 (2)
C33	0.037 (3)	0.079 (3)	0.085 (4)	−0.005 (2)	−0.009 (2)	−0.034 (3)
C34	0.059 (3)	0.086 (4)	0.070 (3)	0.012 (3)	−0.032 (3)	−0.034 (3)
C35	0.070 (4)	0.078 (3)	0.046 (3)	0.008 (3)	−0.019 (2)	−0.009 (2)
C36	0.048 (3)	0.061 (3)	0.038 (2)	−0.004 (2)	−0.0073 (18)	−0.0090 (19)

C38	0.085 (13)	0.118 (15)	0.194 (19)	−0.046 (10)	−0.002 (13)	−0.074 (13)
Ge1	0.0324 (2)	0.0288 (2)	0.02414 (18)	−0.00556 (14)	0.00189 (13)	−0.00704 (14)
N1	0.0337 (16)	0.0311 (14)	0.0255 (13)	−0.0047 (11)	0.0001 (11)	−0.0096 (11)
N2	0.0418 (18)	0.0314 (15)	0.0276 (14)	−0.0092 (12)	0.0045 (12)	−0.0116 (12)
N3	0.185 (14)	0.074 (7)	0.072 (7)	−0.027 (8)	0.007 (7)	−0.023 (5)
S1	0.0318 (5)	0.0476 (5)	0.0295 (4)	−0.0063 (4)	−0.0011 (4)	−0.0027 (4)
Si1	0.0323 (5)	0.0330 (5)	0.0241 (4)	−0.0062 (4)	0.0015 (4)	−0.0094 (4)
C37	0.20 (2)	0.103 (16)	0.16 (2)	−0.043 (15)	0.008 (18)	−0.046 (14)

Geometric parameters (Å, º)

C1—C6	1.397 (5)	C21—H21C	0.9600
C1—C2	1.410 (5)	C22—C24	1.520 (7)
C1—N1	1.441 (4)	C22—C23	1.535 (8)
C2—C3	1.399 (6)	C22—H22	0.9800
C2—C10	1.513 (6)	C23—H23A	0.9600
C3—C4	1.368 (7)	C23—H23B	0.9600
C3—H3	0.9300	C23—H23C	0.9600
C4—C5	1.349 (7)	C24—H24A	0.9600
C4—H4	0.9300	C24—H24B	0.9600
C5—C6	1.395 (6)	C24—H24C	0.9600
C5—H5	0.9300	C25—C26	1.377 (5)
C6—C7	1.511 (6)	C25—C30	1.391 (5)
C7—C9	1.539 (6)	C25—Si1	1.864 (4)
C7—C8	1.539 (6)	C26—C27	1.383 (6)
C7—H7	0.9800	C26—H26	0.9300
C8—H8A	0.9600	C27—C28	1.358 (8)
C8—H8B	0.9600	C27—H27	0.9300
C8—H8C	0.9600	C28—C29	1.374 (8)
C9—H9A	0.9600	C28—H28	0.9300
C9—H9B	0.9600	C29—C30	1.388 (7)
C9—H9C	0.9600	C29—H29	0.9300
C10—C11	1.533 (6)	C30—H30	0.9300
C10—C12	1.538 (6)	C31—C32	1.388 (6)
C10—H10	0.9800	C31—C36	1.395 (5)
C11—H11A	0.9600	C31—Si1	1.877 (4)
C11—H11B	0.9600	C32—C33	1.391 (6)
C11—H11C	0.9600	C32—H32	0.9300
C12—H12A	0.9600	C33—C34	1.379 (7)
C12—H12B	0.9600	C33—H33	0.9300
C12—H12C	0.9600	C34—C35	1.357 (7)
C13—C18	1.400 (6)	C34—H34	0.9300
C13—C14	1.405 (6)	C35—C36	1.388 (6)
C13—N2	1.444 (4)	C35—H35	0.9300
C14—C15	1.380 (6)	C36—H36	0.9300
C14—C19	1.505 (7)	C38—C38^{i}	0.88 (3)
C15—C16	1.343 (9)	C38—C37^{i}	1.11 (3)
C15—H15	0.9300	C38—C37	1.495 (9)

C16—C17	1.367 (9)	C38—H38A	0.9600
C16—H16	0.9300	C38—H38B	0.9599
C17—C18	1.400 (7)	C38—H38C	0.9600
C17—H17	0.9300	Ge1—N2	1.843 (3)
C18—C22	1.514 (7)	Ge1—N1	1.847 (3)
C19—C21	1.524 (9)	Ge1—S1^{ii}	2.2050 (9)
C19—C20	1.525 (9)	Ge1—S1	2.2590 (10)
C19—H19	0.9800	Ge1—Si1	2.6179 (9)
C20—H20A	0.9600	N1—Si1	1.752 (3)
C20—H20B	0.9600	N2—Si1	1.738 (3)
C20—H20C	0.9600	N3—C37	1.091 (9)
C21—H21A	0.9600	S1—Ge1^{ii}	2.2050 (9)
C21—H21B	0.9600	C37—C38^{i}	1.11 (3)

C6—C1—C2	120.3 (3)	C23—C22—H22	107.9
C6—C1—N1	121.0 (3)	C22—C23—H23A	109.5
C2—C1—N1	118.7 (3)	C22—C23—H23B	109.5
C3—C2—C1	117.5 (4)	H23A—C23—H23B	109.5
C3—C2—C10	119.2 (4)	C22—C23—H23C	109.5
C1—C2—C10	123.3 (3)	H23A—C23—H23C	109.5
C4—C3—C2	122.3 (5)	H23B—C23—H23C	109.5
C4—C3—H3	118.9	C22—C24—H24A	109.5
C2—C3—H3	118.9	C22—C24—H24B	109.5
C5—C4—C3	119.3 (4)	H24A—C24—H24B	109.5
C5—C4—H4	120.3	C22—C24—H24C	109.5
C3—C4—H4	120.3	H24A—C24—H24C	109.5
C4—C5—C6	122.1 (5)	H24B—C24—H24C	109.5
C4—C5—H5	119.0	C26—C25—C30	117.4 (4)
C6—C5—H5	119.0	C26—C25—Si1	123.3 (3)
C5—C6—C1	118.6 (4)	C30—C25—Si1	118.8 (3)
C5—C6—C7	118.9 (4)	C25—C26—C27	121.9 (4)
C1—C6—C7	122.5 (3)	C25—C26—H26	119.0
C6—C7—C9	111.8 (4)	C27—C26—H26	119.0
C6—C7—C8	111.4 (4)	C28—C27—C26	120.0 (5)
C9—C7—C8	110.4 (4)	C28—C27—H27	120.0
C6—C7—H7	107.7	C26—C27—H27	120.0
C9—C7—H7	107.7	C27—C28—C29	119.7 (4)
C8—C7—H7	107.7	C27—C28—H28	120.2
C7—C8—H8A	109.5	C29—C28—H28	120.2
C7—C8—H8B	109.5	C28—C29—C30	120.5 (5)
H8A—C8—H8B	109.5	C28—C29—H29	119.7
C7—C8—H8C	109.5	C30—C29—H29	119.7
H8A—C8—H8C	109.5	C29—C30—C25	120.4 (5)
H8B—C8—H8C	109.5	C29—C30—H30	119.8
C7—C9—H9A	109.5	C25—C30—H30	119.8
C7—C9—H9B	109.5	C32—C31—C36	117.0 (4)
H9A—C9—H9B	109.5	C32—C31—Si1	122.5 (3)
C7—C9—H9C	109.5	C36—C31—Si1	120.4 (3)

H9A—C9—H9C	109.5	C31—C32—C33	121.2 (4)
H9B—C9—H9C	109.5	C31—C32—H32	119.4
C2—C10—C11	110.7 (4)	C33—C32—H32	119.4
C2—C10—C12	113.7 (4)	C34—C33—C32	119.9 (4)
C11—C10—C12	109.0 (4)	C34—C33—H33	120.0
C2—C10—H10	107.7	C32—C33—H33	120.0
C11—C10—H10	107.7	C35—C34—C33	120.3 (4)
C12—C10—H10	107.7	C35—C34—H34	119.9
C10—C11—H11A	109.5	C33—C34—H34	119.9
C10—C11—H11B	109.5	C34—C35—C36	119.8 (4)
H11A—C11—H11B	109.5	C34—C35—H35	120.1
C10—C11—H11C	109.5	C36—C35—H35	120.1
H11A—C11—H11C	109.5	C35—C36—C31	121.8 (4)
H11B—C11—H11C	109.5	C35—C36—H36	119.1
C10—C12—H12A	109.5	C31—C36—H36	119.1
C10—C12—H12B	109.5	C38^{i}—C38—C37^{i}	97 (3)
H12A—C12—H12B	109.5	C38^{i}—C38—C37	47 (2)
C10—C12—H12C	109.5	C37^{i}—C38—C37	144.4 (16)
H12A—C12—H12C	109.5	C38^{i}—C38—H38A	140.5
H12B—C12—H12C	109.5	C37^{i}—C38—H38A	85.0
C18—C13—C14	120.5 (4)	C37—C38—H38A	121.2
C18—C13—N2	119.6 (3)	C38^{i}—C38—H38B	110.0
C14—C13—N2	119.9 (4)	C37^{i}—C38—H38B	89.8
C15—C14—C13	119.1 (5)	C37—C38—H38B	101.7
C15—C14—C19	118.5 (5)	H38A—C38—H38B	109.5
C13—C14—C19	122.4 (4)	C38^{i}—C38—H38C	58.0
C16—C15—C14	120.9 (6)	C37^{i}—C38—H38C	39.9
C16—C15—H15	119.6	C37—C38—H38C	104.9
C14—C15—H15	119.6	H38A—C38—H38C	109.5
C15—C16—C17	121.0 (5)	H38B—C38—H38C	109.5
C15—C16—H16	119.5	N2—Ge1—N1	82.41 (12)
C17—C16—H16	119.5	N2—Ge1—S1^{ii}	124.77 (9)
C16—C17—C18	121.2 (6)	N1—Ge1—S1^{ii}	125.33 (9)
C16—C17—H17	119.4	N2—Ge1—S1	114.90 (10)
C18—C17—H17	119.4	N1—Ge1—S1	116.58 (9)
C13—C18—C17	117.3 (5)	S1^{ii}—Ge1—S1	94.97 (3)
C13—C18—C22	124.1 (4)	N2—Ge1—Si1	41.47 (9)
C17—C18—C22	118.6 (5)	N1—Ge1—Si1	41.92 (8)
C14—C19—C21	112.4 (5)	S1^{ii}—Ge1—Si1	146.78 (4)
C14—C19—C20	112.5 (6)	S1—Ge1—Si1	118.22 (3)
C21—C19—C20	110.9 (6)	C1—N1—Si1	135.0 (2)
C14—C19—H19	106.8	C1—N1—Ge1	131.7 (2)
C21—C19—H19	106.8	Si1—N1—Ge1	93.30 (12)
C20—C19—H19	106.8	C13—N2—Si1	133.6 (2)
C19—C20—H20A	109.5	C13—N2—Ge1	131.2 (2)
C19—C20—H20B	109.5	Si1—N2—Ge1	93.92 (13)
H20A—C20—H20B	109.5	Ge1^{ii}—S1—Ge1	85.03 (3)
C19—C20—H20C	109.5	N2—Si1—N1	88.31 (13)

H20A—C20—H20C	109.5	N2—Si1—C25	116.31 (15)
H20B—C20—H20C	109.5	N1—Si1—C25	119.45 (15)
C19—C21—H21A	109.5	N2—Si1—C31	114.79 (15)
C19—C21—H21B	109.5	N1—Si1—C31	113.02 (15)
H21A—C21—H21B	109.5	C25—Si1—C31	104.96 (16)
C19—C21—H21C	109.5	N2—Si1—Ge1	44.61 (9)
H21A—C21—H21C	109.5	N1—Si1—Ge1	44.78 (9)
H21B—C21—H21C	109.5	C25—Si1—Ge1	138.43 (12)
C18—C22—C24	111.4 (4)	C31—Si1—Ge1	116.59 (12)
C18—C22—C23	113.1 (5)	N3—C37—C38^{i}	144.0 (16)
C24—C22—C23	108.3 (5)	N3—C37—C38	179.4 (4)
C18—C22—H22	107.9	C38^{i}—C37—C38	35.6 (16)
C24—C22—H22	107.9		

C6—C1—C2—C3	0.0 (5)	Si1—Ge1—N1—C1	179.5 (4)
N1—C1—C2—C3	180.0 (3)	N2—Ge1—N1—Si1	10.54 (13)
C6—C1—C2—C10	177.6 (3)	S1^{ii}—Ge1—N1—Si1	138.19 (8)
N1—C1—C2—C10	−2.3 (5)	S1—Ge1—N1—Si1	−103.55 (10)
C1—C2—C3—C4	0.1 (7)	C18—C13—N2—Si1	76.5 (5)
C10—C2—C3—C4	−177.7 (4)	C14—C13—N2—Si1	−102.1 (4)
C2—C3—C4—C5	0.2 (8)	C18—C13—N2—Ge1	−86.7 (4)
C3—C4—C5—C6	−0.5 (8)	C14—C13—N2—Ge1	94.7 (4)
C4—C5—C6—C1	0.6 (7)	N1—Ge1—N2—C13	157.2 (3)
C4—C5—C6—C7	−179.4 (4)	S1^{ii}—Ge1—N2—C13	29.1 (4)
C2—C1—C6—C5	−0.3 (5)	S1—Ge1—N2—C13	−87.0 (3)
N1—C1—C6—C5	179.7 (3)	Si1—Ge1—N2—C13	167.9 (4)
C2—C1—C6—C7	179.7 (3)	N1—Ge1—N2—Si1	−10.64 (13)
N1—C1—C6—C7	−0.4 (5)	S1^{ii}—Ge1—N2—Si1	−138.79 (8)
C5—C6—C7—C9	−56.7 (5)	S1—Ge1—N2—Si1	105.20 (11)
C1—C6—C7—C9	123.4 (4)	N2—Ge1—S1—Ge1^{ii}	132.18 (10)
C5—C6—C7—C8	67.3 (5)	N1—Ge1—S1—Ge1^{ii}	−133.84 (9)
C1—C6—C7—C8	−112.7 (4)	S1^{ii}—Ge1—S1—Ge1^{ii}	0.0
C3—C2—C10—C11	81.3 (5)	Si1—Ge1—S1—Ge1^{ii}	178.67 (3)
C1—C2—C10—C11	−96.3 (4)	C13—N2—Si1—N1	−156.2 (3)
C3—C2—C10—C12	−41.8 (5)	Ge1—N2—Si1—N1	11.13 (13)
C1—C2—C10—C12	140.6 (4)	C13—N2—Si1—C25	−34.0 (4)
C18—C13—C14—C15	−2.5 (7)	Ge1—N2—Si1—C25	133.41 (15)
N2—C13—C14—C15	176.1 (4)	C13—N2—Si1—C31	89.1 (4)
C18—C13—C14—C19	176.5 (5)	Ge1—N2—Si1—C31	−103.53 (16)
N2—C13—C14—C19	−4.8 (6)	C13—N2—Si1—Ge1	−167.4 (4)
C13—C14—C15—C16	1.2 (10)	C1—N1—Si1—N2	169.4 (3)
C19—C14—C15—C16	−177.9 (7)	Ge1—N1—Si1—N2	−11.09 (13)
C14—C15—C16—C17	0.1 (12)	C1—N1—Si1—C25	49.9 (4)
C15—C16—C17—C18	−0.1 (12)	Ge1—N1—Si1—C25	−130.60 (15)
C14—C13—C18—C17	2.4 (7)	C1—N1—Si1—C31	−74.3 (4)
N2—C13—C18—C17	−176.2 (4)	Ge1—N1—Si1—C31	105.21 (15)
C14—C13—C18—C22	−175.9 (4)	C1—N1—Si1—Ge1	−179.5 (4)
N2—C13—C18—C22	5.4 (6)	C26—C25—Si1—N2	−141.4 (3)

C16—C17—C18—C13	−1.2 (9)	C30—C25—Si1—N2	46.3 (4)
C16—C17—C18—C22	177.3 (6)	C26—C25—Si1—N1	−37.4 (4)
C15—C14—C19—C21	−64.4 (8)	C30—C25—Si1—N1	150.2 (3)
C13—C14—C19—C21	116.5 (6)	C26—C25—Si1—C31	90.6 (4)
C15—C14—C19—C20	61.7 (7)	C30—C25—Si1—C31	−81.8 (4)
C13—C14—C19—C20	−117.4 (6)	C26—C25—Si1—Ge1	−91.1 (4)
C13—C18—C22—C24	101.9 (5)	C30—C25—Si1—Ge1	96.5 (3)
C17—C18—C22—C24	−76.4 (6)	C32—C31—Si1—N2	40.0 (4)
C13—C18—C22—C23	−135.8 (5)	C36—C31—Si1—N2	−141.3 (3)
C17—C18—C22—C23	45.9 (7)	C32—C31—Si1—N1	−59.3 (4)
C30—C25—C26—C27	−1.2 (7)	C36—C31—Si1—N1	119.4 (3)
Si1—C25—C26—C27	−173.6 (4)	C32—C31—Si1—C25	168.9 (3)
C25—C26—C27—C28	−0.6 (8)	C36—C31—Si1—C25	−12.4 (4)
C26—C27—C28—C29	1.8 (9)	C32—C31—Si1—Ge1	−9.8 (4)
C27—C28—C29—C30	−1.2 (9)	C36—C31—Si1—Ge1	168.9 (3)
C28—C29—C30—C25	−0.6 (9)	N1—Ge1—Si1—N2	164.10 (19)
C26—C25—C30—C29	1.7 (7)	S1^{ii}—Ge1—Si1—N2	81.03 (15)
Si1—C25—C30—C29	174.5 (4)	S1—Ge1—Si1—N2	−96.56 (15)
C36—C31—C32—C33	−0.1 (6)	N2—Ge1—Si1—N1	−164.10 (19)
Si1—C31—C32—C33	178.7 (3)	S1^{ii}—Ge1—Si1—N1	−83.07 (14)
C31—C32—C33—C34	0.1 (7)	S1—Ge1—Si1—N1	99.34 (13)
C32—C33—C34—C35	−0.3 (8)	N2—Ge1—Si1—C25	−78.9 (2)
C33—C34—C35—C36	0.3 (8)	N1—Ge1—Si1—C25	85.2 (2)
C34—C35—C36—C31	−0.3 (8)	S1^{ii}—Ge1—Si1—C25	2.1 (2)
C32—C31—C36—C35	0.1 (6)	S1—Ge1—Si1—C25	−175.49 (18)
Si1—C31—C36—C35	−178.7 (4)	N2—Ge1—Si1—C31	99.21 (19)
C6—C1—N1—Si1	95.0 (4)	N1—Ge1—Si1—C31	−96.68 (18)
C2—C1—N1—Si1	−85.0 (4)	S1^{ii}—Ge1—Si1—C31	−179.76 (13)
C6—C1—N1—Ge1	−84.3 (4)	S1—Ge1—Si1—C31	2.66 (14)
C2—C1—N1—Ge1	95.7 (4)	C38^{i}—C38—C37—N3	45 (39)
N2—Ge1—N1—C1	−170.0 (3)	C37^{i}—C38—C37—N3	45 (39)
S1^{ii}—Ge1—N1—C1	−42.3 (3)	C37^{i}—C38—C37—C38^{i}	0.000 (8)
S1—Ge1—N1—C1	76.0 (3)		

Symmetry codes: (i) −*x*, −*y*, −*z*+2; (ii) −*x*+1, −*y*+1, −*z*+2.