$(CH_3NH_3)_2TeCl_6$ exceptionally forms an *anti*-CdI_2-type arrangement (Kitahama & Kiriyama, 1978).

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1,3,7,9-Tetra-*tert*-butyl-2,2,8,8-tetramethyl-5,10-diphenyl-1,3,5,7,9,10-hexaaza-2,8-disila-4,6-digermadispiro[3.1.3.1]decane

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Abstract. $C_{32}H_{58}Ge_2N_6Si_2$, $M_r = 728.20$, triclinic, $P\bar{1}$, $a = 9.182$ (11), $b = 9.774$ (9), $c = 11.784$ (10) Å, $\alpha = 107.27$ (7), $\beta = 100.16$ (8), $\gamma = 98.05$ (9)°, $V = 973$ (2) Å^3, $Z = 1$, $D_x = 1.243$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.61$ mm^{-1}, $F(000) = 384$, $T = 291$ (1) K, final $R = 0.0443$ for 2186 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The crystal consists of discrete molecular units. The central part of the centrosymmetric molecule is a planar four-membered Ge—N—Ge—N— ring [Ge—N 1.851 (4), 1.852 (4); Ge···Ge 2.784 (1); N···N 2.441 (4) Å; Ge—N—Ge 97.5 (1); N—Ge—N 82.5 (1)°]. A phenyl group is bound to each N atom of this ring and each Ge atom of this central ring is also a member of an outer four-membered Ge—N—Si—N— ring [Ge—N 1.829 (4), 1.824 (4); Si—N 1.713 (4), 1.735 (4); Ge···Si 2.608 (4); N···N 2.441 (4) Å; N—Ge—N 82.5 (2); Ge—N—Si 94.8 (3), 94.2 (3); N—Si—N 88.6 (3)°]. The dihedral angle between the plane of the central ring and the least-squares plane through the outer ring is 90.6 (2)°. A distorted tetrahedron [angles between 82.5 (1) and 125.1 (1)°] around Ge is formed by the atoms bound to Ge. The phenyl rings bound to the N atoms of the Ge—N—Ge—N— ring and this central four-membered ring are nearly [4.0 (2)°] coplanar. The coordination around Si is a distorted tetrahedron with angles between 88.6 (3) and 115.5 (3)°.

Experimental. The title compound has been obtained as the major product from the known germylene (Veith & Grosser, 1982) and phenyl azide at 298 K. Colourless crystals were obtained from a saturated THF solution by slow cooling to 253 K. A crystal platelet of approximate size 0.86 × 0.28 × 0.10 mm was used. Its quality was checked with optical polarizing microscopy. The crystal was mounted on the tip of a glass fibre with shellac. D_m was not determined. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 4.5–14.6° min^{-1} in θ, scan width 1.2° + dispersion. A Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation was used for preliminary examinations and data collection. The lattice parameters were determined from a least-squares fit of the angular settings for ten reflections with $2\theta_{max} = 24.8°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections ($4\bar{4}2$, 050, $0\bar{2}7$, $\bar{4}4\bar{2}$, $0\bar{5}0$, $02\bar{7}$) were recorded every 300 reflections; only random deviations were detected during 38.4 h of X-ray exposure. 4902 reflections with $2.0 \leq 2\theta \leq 45.0°$, $-10 \leq h \leq 6$, $-11 \leq k \leq 11$, $-13 \leq l \leq 13$ were measured. The

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data were corrected for Lorentz–polarization but not for absorption effects, and averaged ($R_{int} = 0.0415$) to 2563 unique reflections, 2186 of which had $F \geq 4.0\sigma(F)$. The structure was solved *via* direct methods and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a common isotropic displacement parameter for the H atoms, which were placed in geometrically calculated positions (C—H 0.96 Å). The phenyl ring was refined as a rigid body (C—C 1.396 Å, C—C—C 120°). 179 parameters were refined. Weights $w^{-1} = \sigma^2(F) + 0.0037F^2$, led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. The refinement converged to $S = 1.37$, $R = 0.0443$, $wR = 0.0588$, $(\Delta/\sigma)_{max} = 0.003$ (no extinction correction). The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in final $\Delta\rho$ map were ± 0.7 (2) e Å^{-3}. Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELXTL-Plus* (Sheldrick, 1987), *PCK*83 (Williams, 1984), *PLATON* (Spek, 1982), *SADIAN* (Baur & Wenninger, 1969), and *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Interatomic distances, bond angles, least-squares planes and dihedral angles are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55026 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA0016]

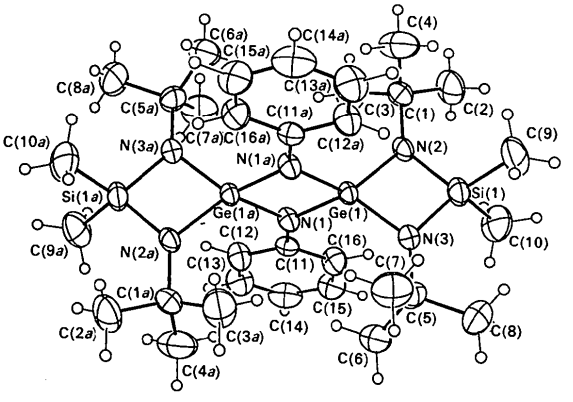

Fig. 1. General view (*SHELXTL-Plus;* Sheldrick, 1987) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries. H atoms are represented as spheres of arbitrary radii.

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* (Å$^2 \times 10^4$)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Ge(1)	0.07505 (5)	0.11369 (4)	0.10650 (4)	261
N(1)	0.0769 (4)	0.0520 (4)	−0.0576 (3)	306
C(12)	0.1325 (3)	0.0497 (3)	−0.2528 (2)	411
C(13)	0.2175 (3)	0.1100 (3)	−0.3199 (2)	508
C(14)	0.3317 (3)	0.2343 (3)	−0.2604 (2)	534
C(15)	0.3609 (3)	0.2984 (3)	−0.1339 (2)	479
C(16)	0.2760 (3)	0.2382 (3)	−0.0667 (2)	426
C(11)	0.1618 (3)	0.1138 (3)	−0.1262 (2)	304
Si(1)	0.2153 (2)	0.3289 (1)	0.3047 (1)	389
C(9)	0.1789 (7)	0.3586 (6)	0.4603 (5)	658
C(10)	0.3698 (6)	0.4815 (5)	0.3154 (6)	691
N(2)	0.0579 (4)	0.2985 (3)	0.1909 (3)	308
C(1)	−0.0655 (5)	0.3790 (5)	0.1745 (4)	398
C(2)	−0.0027 (7)	0.5427 (5)	0.2406 (5)	599
C(3)	−0.1240 (7)	0.3514 (6)	0.0398 (5)	701
C(4)	−0.1935 (6)	0.3279 (6)	0.2288 (6)	716
N(3)	0.2415 (4)	0.1552 (4)	0.2306 (3)	356
C(5)	0.3561 (5)	0.0688 (5)	0.2541 (5)	428
C(6)	0.4256 (6)	0.0254 (6)	0.1441 (5)	568
C(7)	0.2825 (7)	−0.0657 (6)	0.2787 (6)	661
C(8)	0.4806 (7)	0.1622 (7)	0.3650 (6)	807

Table 2. *Interatomic distances* (Å), *bond angles* (°), *least-squares planes and dihedral angles* (°)

Ge(1)—N(1)	1.851 (4)	Si(1)—N(3)	1.735 (4)
Ge(1)—N(1*a*)	1.852 (4)	N(2)—C(1)	1.484 (6)
Ge(1)···Si(1)	2.608 (4)	N(2)···N(3)	2.408 (3)
Ge(1)—N(2)	1.829 (4)	C(1)—C(2)	1.531 (6)
Ge(1)—N(3)	1.824 (4)	C(1)—C(3)	1.512 (8)
Ge(1)···Ge(1*a*)	2.784 (1)	C(1)—C(4)	1.526 (9)
N(1)—C(11)	1.417 (5)	N(3)—C(5)	1.477 (7)
N(1)···N(1*a*)	2.441 (4)	C(5)—C(6)	1.519 (8)
Si(1)—C(9)	1.867 (6)	C(5)—C(7)	1.521 (8)
Si(1)—C(10)	1.867 (6)	C(5)—C(8)	1.523 (7)
Si(1)—N(2)	1.713 (4)		

N(1)—Ge(1)—N(1*a*)	82.5 (1)	Si(1)—N(2)—C(1)	133.6 (4)
N(1)—Ge(1)—N(2)	123.6 (2)	Ge(1)—N(2)—C(1)	130.9 (4)
N(1)—Ge(1)—N(3)	124.4 (3)	N(2)—C(1)—C(4)	110.0 (5)
N(1*a*)—Ge(1)—N(2)	125.1 (1)	N(2)—C(1)—C(3)	109.2 (5)
N(1*a*)—Ge(1)—N(3)	124.6 (1)	N(2)—C(1)—C(2)	108.8 (5)
N(2)—Ge(1)—N(3)	82.5 (2)	C(3)—C(1)—C(4)	109.9 (5)
Ge(1)—N(1)—Ge(1*a*)	97.5 (1)	C(2)—C(1)—C(4)	109.1 (5)
Ge(1)—N(1)—C(11)	131.8 (3)	C(2)—C(1)—C(3)	109.8 (5)
N(1)—C(11)—C(16)	119.8 (3)	Ge(1)—N(3)—Si(1)	94.2 (3)
N(1)—C(11)—C(12)	120.2 (4)	Si(1)—N(3)—C(5)	134.4 (4)
N(2)—Si(1)—N(3)	88.6 (3)	Ge(1)—N(3)—C(5)	131.4 (4)
C(10)—Si(1)—N(3)	114.7 (4)	N(3)—C(5)—C(8)	109.4 (5)
C(10)—Si(1)—N(2)	114.6 (3)	N(3)—C(5)—C(7)	109.5 (5)
C(9)—Si(1)—N(3)	115.5 (3)	N(3)—C(5)—C(6)	108.9 (5)
C(9)—Si(1)—N(2)	115.0 (3)	C(7)—C(5)—C(8)	109.4 (5)
C(9)—Si(1)—C(10)	107.9 (3)	C(6)—C(5)—C(8)	108.5 (5)
Ge(1)—N(2)—Si(1)	94.8 (3)	C(6)—C(5)—C(7)	111.0 (5)

No.	Plane through atoms	Equation of plane	χ^2	Dihedral angle
1	Ge(1),N(1),Ge(1*a*),N(1*a*)	$-0.754x + 0.634y - 0.175z = 0.0$ Å	0.0	1,2 90.6 (2)
2	Ge(1),N(2),N(3),Si(1)	$0.624x + 0.582y - 0.522z = -0.009$ Å	1.0	1,3 4.0 (2)
3	C(11),C(12),C(13),C(14), C(15),C(16)	$-0.750x + 0.652y - 0.108z = -0.011$ Å	0.0	2,3 91.8 (2)

Related literature. The crystal structures of the Sn analogue (Preut, Obloh & Neumann, 1987), and that of a comparable 5,7-digermadispiro[4.1.4.1]dodecane (Pfeiffer, Maringgele, Noltemeyer & Meller, 1989) have been published. For further results and details of the above mentioned reaction see Preut, Obloh & Neumann (1988) and Obloh & Neumann (1991).

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Structure of a Twisted Styrylbenzothiazole, 2-(α-Acetoxy-2-chloro-5-nitrostyryl)-benzothiazole

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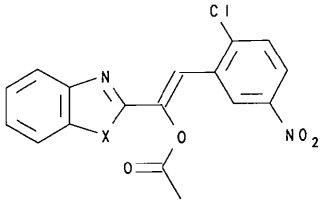
Abstract. α-(1,3-Benzothiazol-2-yl)-2-chloro-5-nitrostyryl α-acetate (ac-nsb), $C_{17}H_{11}ClN_2O_4S$, M_r = 374.81, triclinic, $P\bar{1}$, a = 9.762 (4), b = 10.689 (5), c = 8.387 (3) Å, α = 96.97 (4), β = 102.26 (3), γ = 76.74 (4)°, V = 830 (1) Å^3, Z = 2, D_x = 1.500 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 3.72 cm^{-1}, $F(000)$ = 384, T = 298 K, final R = 0.047 for 2463 unique observed reflections. The molecule is distinctly non-planar, with the benzothiazole and phenyl rings rotated with respect to the olefin to give a dihedral angle of 51.1 (1)° between the planes of those rings.

Experimental. The benzothiazole derivative ac-nsb (I) (X = S) was prepared as part of a study of substitution in the azole ring (X = O, S, Se and NR, $R = C_2H_5$). It was synthesized by refluxing 2-hydroxymethylbenzothiazole and 2-chloro-5-nitrobenzaldehyde in acetic anhydride. The solid product, m.p. 428–430 K, formed on vacuum evaporation. Crystals were obtained by slow evaporation from hexane. Analysis for $C_{17}H_{11}ClN_2O_4S$: calculated C 55.48, H 2.96, N 7.47%; found C 54.43, H 2.97, N 7.47%.



(I)

Light-brown plate (D_m not determined), 0.30 × 0.25 × 0.20 mm, used for data collection on an Enraf–Nonius CAD-4 diffractometer with monochromated Mo $K\alpha$ radiation, ω–2θ scan, scan width $(0.9 + 0.34\tan\theta)°$, $2\theta_{max}$ = 53°. Lattice parameters were determined from least-squares fit of 24 reflections in the range $10 < \theta < 15°$. No systematic absences; $-h$, $\pm k$, $\pm l$, $-12 \le h \le 0$, $-13 \le k \le 13$, $-10 \le l \le 10$. Three standard reflections ($\bar{4}\bar{1}4$, 422 and $2\bar{4}1$) showed total intensity loss of 1.1% during 51.1 h of exposure. R_{int} = 0.02. Total number of reflections measured was 5094, 2463 of these being unique with $F^2 > 3\sigma^2(F_o)$, $\sigma(F_o) = [\sigma^2(I_{raw}) + (0.04F_o^2)^2]^{1/2}/2F_o$. The structure was solved with the Enraf–Nonius *Sructure Determination Package* (Frenz, 1986), heavy-atom and difference Fourier methods. Isotropic then anisotropic refinement was performed on F of all non-H atoms, H atoms were placed at calculated positions using a riding model [C—H 0.95 Å, U(H) 1.3U(C)]. 227 parameters were refined. R = 0.047, wR = 0.060, $w = 1/[\sigma^2(F_o)]$, S = 1.501; difference syntheses showed no densities above 0.37 e Å^{-3} (minimum −0.39 e Å^{-3}); maximum Δ/σ = 0.01. Neutral-atom scattering factors were used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B, 2.3.1), and were corrected for anomalous dispersion (Cromer & Liberman, 1970). No absorption correction was applied. An extinction parameter which refined to 1.10 (1) × 10^{-5} was applied and the calculated structure factor became $F_c = 1/(1 + xI_c)$ (maximum correction 55 for the 012 reflection).

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