

Structures of the Perhalogenated Cyclopentasilanes $\text{Si}_5\text{Br}_{10}$ and Si_5I_{10} at Normal and Low Temperatures

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Abstract. Si_5I_{10} : $M_r = 1409.5$; at $T = 297$ K: monoclinic, $P2_1/n$, $a = 14.304$ (4), $b = 15.782$ (5), $c = 10.966$ (5) Å, $\beta = 101.30$ (4)°, $V = 2427$ Å³, $Z = 4$, $D_x = 3.86$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.71069$ Å, $\mu = 128.6$ cm⁻¹, $F(000) = 2400$, $R = 0.069$ for 1574 reflections; at $T = 96$ K: monoclinic, $P2_1/n$, $a = 14.200$ (5), $b = 15.606$ (3), $c = 10.870$ (2) Å, $\beta = 101.14$ (1)°, $V = 2363.5$ Å³, $Z = 4$, $D_x = 3.96$ g cm⁻³, Mo $K\alpha$ radiation, $\mu = 134.7$ cm⁻¹, $F(000) = 2400$, $R = 0.042$ for 2673 reflections. $\text{Si}_5\text{Br}_{10}$: $M_r = 939.5$, monoclinic, $P2_1/n$, $a = 13.173$ (6), $b = 14.699$ (4), $c = 10.279$ (3) Å, $\beta = 102.85$ (2)°, $V = 1940$ Å³, $Z = 4$, $D_x = 3.22$ g cm⁻³, Mo $K\alpha$ radiation, $\mu = 222$ cm⁻¹, $F(000) = 1680$, $T = 87$ K, $R = 0.055$ for 2202 reflections. The crystal structures are isomorphous and show Si_5 -ring conformations intermediate between envelope and twist. Bond angles within the five-membered rings show positive correlation with the opposite dihedral angle, and bond lengths correlate negatively with the absolute value of the dihedral angle about the same bond.

Introduction. Recent accomplishments in the chemistry of cyclosilanes (Hengge, Schuster & Peter, 1980; Hengge, 1980) have made this class of compounds available to structural investigation. The cyclic Si_4 system shows in the crystal a strong dependence of the ring conformation on the nature of the substituents (Kratky, Schuster & Hengge, 1983). For the cyclic Si_5 system, electron diffraction of cyclo- Si_5I_{10} (Smith, Seip, Hengge & Bauer, 1976) has established a conformation similar to cyclopentane with pseudorotation. So far, the only crystal structure determination on a cyclo- Si_5 compound involved the perphenylated $\text{Si}_5\text{Ph}_{10}$ (Párkányi, Sasvári, Declercq & Germain, 1978), also yielding a highly puckered conformation.

We determined the crystal structures of the halogenides Si_5I_{10} and $\text{Si}_5\text{Br}_{10}$ in view of the scarcity of structural evidence on the cyclo- Si_5 system and in order to see whether a similar dependence between ring conformation and the nature of the substituents exists as with the Si_4 ring.

There is yet another aspect of the two crystal structures: crystallographic evidence on five-membered rings with the full symmetry of a regular isolateral pentagon is limited to arsenomethane As_5Me_5 (Burns & Waser, 1957) and decaphenylcyclopentasilane $\text{Si}_5\text{Ph}_{10}$ (Párkányi *et al.*, 1978). Structural knowledge about such rings is otherwise mainly based on electron diffraction evidence (Kilpatrick, Pitzer & Spitzer, 1947; Adams, Geise & Bartell, 1970; Smith, Seip, Hengge & Bauer, 1976). However, electron diffraction relies on a mathematical description of the pseudorotation, and is unable to distinguish between different conformations along the pseudorotation path. The evidence compiled from the crystal structures of symmetrically substituted five-membered rings is therefore relevant to the validity of mathematical pseudorotation models (Kilpatrick, Pitzer & Spitzer, 1947; Cremer & Pople, 1975; Altona, Geise & Romers, 1968; Adams, Geise & Bartell, 1970; Dunitz, 1972; Herzyk & Rabczenko, 1983).

The structure of Si_5I_{10} was determined independently in Graz and in Delaware. When we became aware of this duplication, the experimental work had been finished in both laboratories, and we decided to report our results together. The structure of the bromide $\text{Si}_5\text{Br}_{10}$ was determined only in Graz. Both compounds are highly reactive; at ambient conditions, crystals hydrolyze instantaneously. While the work in Graz was therefore carried out in a cold N_2 stream, the Delaware group determined the Si_5I_{10} structure at room temperature under argon.

Table 1. *Experimental data*

	RT-Si ₅ I ₁₀	LT-Si ₅ I ₁₀	LT-Si ₅ Br ₁₀
Temperature (K)	297 (2)	96 (1)*	87 (1)*
Crystal size (mm)	0.2 × 0.3 × 0.3†	0.13	0.5 × 0.5 × 0.4
Number and 2θ range (°) of reflections used to determine lattice parameters	25	20	15
	22 ≤ 2θ ≤ 30	19 ≤ 2θ ≤ 21	15 ≤ 2θ ≤ 21
Max./min. transmission factor	0.147/0.066†	0.29/0.28	0.018/0.006
Max. sinθ/λ(Å ⁻¹) for intensity data collection	0.481	0.595	0.595
Range of indices	-13 ≤ h ≤ 13 0 ≤ k ≤ 13 0 ≤ l ≤ 15	-16 ≤ h ≤ 16 0 ≤ k ≤ 18 -12 ≤ l ≤ 0	-15 ≤ h ≤ 15 -17 ≤ k ≤ 0 -12 ≤ l ≤ 0
Number/frequency of standard reflections	3/97	3/100	2/100
Intensity scan technique	ω/2θ	ω	ω
Scan width (°)	2.0 + Δ(α ₁ - α ₂)	1.2	1.0
Number of recorded reflections	2298	4481	4990
Number of unique reflections	2068	3974	3417
Number of significant reflections	1574	2673	2202
R _{int}	0.03	0.007	0.022
Criterion for significant reflections	F _o ² > 3σ(F _o ²)	F _o > 4σ(F _o)	F _o > 4σ(F _o)
R	0.069	0.042	0.055
wR	0.067	0.040	0.055
Coefficient g of weighting‡ system	0.008	0.0	0.0011
Max. Δ/σ in last refinement cycle	0.154	0.05	0.001
Max./min. height in final ΔF Fourier synthesis (e Å ⁻³)	1.4/-0.5	1.6/-1.4	1.4/-1.6

* Recorded temperature of N₂ stream at nozzle of low-temperature device, about 12 mm in front of crystal.

† Value for first out of three crystals used for data collection - see text.

‡ Weighting system: $w_i = [\sigma^2(F_{o,i}) + gF_{o,i}^2]^{-1}$.

Experimental. Relevant data-collection conditions and structure-refinement details are given in Table 1. All three structures involved the refinement of F magnitudes; anisotropic temperature coefficients for all atoms (137 parameters); scattering factors from *International Tables for X-ray Crystallography* (1972).

Room-temperature cyclo-Si₅I₁₀ (RT-Si₅I₁₀). Nicolet R3 diffractometer, crystal mounted in argon-filled 0.3 mm capillary. Decrease in standard intensities (25% in 12 h exposure) required use of three crystals. Weak reflections determined by application of a profile-fitting procedure using learned peak profiles. Data reduction (decay, Lp and absorption with empirical ψ scan, using a refined pseudo-ellipsoid model) for each crystal separately, merging of the three data sets using standard intensities. Structure solved with direct methods and ΔF Fourier syntheses, blocked-cascade refinement. Computer programs: *SHELXTL*, *P3* and *XP* from the Nicolet program packages.

Low-temperature cyclo-Si₅I₁₀ (LT-Si₅I₁₀). Locally modified Stoe four-circle diffractometer inside glove box, Nonius low-temperature attachment. Approximately spherical crystal mounted on glass fiber. Standard intensities constant within $\pm 3\%$. Structure solved with direct methods (*MULTAN*, Germain, Main & Woolfson, 1971) and ΔF Fourier syntheses, empirical extinction correction [$F_{\text{corr}} = F_o(1 - aF_o^2/\sin\theta)$, $a = 9 \times 10^{-9}$ (Sheldrick, 1976)], spherical absorption correction ($\mu r = 0.9$), exclusion of the two strongest reflections in last refinement cycles.

Low-temperature cyclo-Si₅Br₁₀ (LT-Si₅Br₁₀). Procedure analogous to LT-Si₅I₁₀. During data collection, a failure in the low-temperature device led to a several-hours-long rise of the temperature to ambient values. After re-cooling, standard intensities were down by 7 and 9%, respectively. Apart from that, they remained constant within counting statistics. Standard-intensity drop corrected by application of a scaling factor obtained from the average of the two standards. Refinement started from the LT-Si₅I₁₀ coordinates. Empirical extinction correction (see above, $a = 8 \times 10^{-9}$), spherical absorption correction ($\mu r = 5.2$), exclusion of the two strongest reflections.

Computer programs used for LT-Si₅I₁₀ and LT-Si₅Br₁₀: *MULTAN* (Germain, Main & Woolfson, 1971), *XRAY* (Stewart, 1976), *SHELX76* (Sheldrick, 1976), *ORTEP* (Johnson, 1976).*

Discussion. Table 2 lists atomic coordinates and equivalent isotropic temperature coefficients from the three refinements, Table 3 gives intramolecular bonding parameters involving the Si₅-ring atoms, and Fig. 1 shows a superposition of the asymmetric units of RT-Si₅I₁₀ and LT-Si₅I₁₀. The similarity in the shapes of the vibration ellipsoids of corresponding atoms argues against the presence of disorder in any of the two Si₅I₁₀ structures.

Bond lengths within the Si₅ ring are intermediate between the values reported for cyclopentasilane in the gas phase [2.342 (3) Å] and the crystalline Si₅Ph₁₀ [2.396 (8) Å]. Deviations from the average of the five bonds [RT-Si₅I₁₀: 2.362 (10); LT-Si₅I₁₀: 2.366 (10); LT-Si₅Br₁₀: 2.353 (8) Å] far exceed the corresponding e.s.d.'s. Fig. 2 shows a plot of the Si-Si bond lengths r_{jk} vs $[1 + \cos(3\tau_{ijk})]^{1/2}$ [τ_{ijk} is the torsion angle about the jk bond (Adams, Geise & Bartell, 1970)], and the two quantities are evidently correlated ($r = 0.83$ for RT-Si₅I₁₀, 0.97 for LT-Si₅I₁₀ and 0.87 for LT-Si₅Br₁₀). Unlike bond angles, bond lengths are not subject to geometrical constraints, but it is not surprising that the most completely eclipsed bonds are associated with the longest bond lengths.

The conformations of five-membered rings are frequently described in terms of a puckering amplitude $q = \left(\sum_{i=1}^5 z_i^2 \right)^{1/2}$ (z_i = perpendicular out-of-plane deviations of atom i from a least-squares plane through the ring) and a phase angle φ [$z_i(\text{calc.}) = \sqrt{(2/5)} q \cos(4\pi i/5 + \varphi)$ (Kilpatrick, Pitzer & Spitzer, 1947)]. Values for q and φ for the five known crystal structures of symmetrically substituted five-membered rings are

* Lists of structure factors, anisotropic thermal parameters, bond lengths and bond angles for the three structures have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42048 (51 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Atomic coordinates and equivalent isotropic temperature coefficients ($\text{\AA}^2 \times 10^4$) for the three crystal structure investigations [(A) RT-Si₅I₁₀, (B) LT-Si₅I₁₀, (C) LT-Si₅Br₁₀]

E.s.d.'s are in units of the last figure. Values for U_{eq} were obtained as one third of the trace of the orthogonalized U_{ij} tensor. $X=I$ for (A) and (B), $X=Br$ for (C).

	(A)				(B)				(C)			
	x	y	z	U_{eq}	x	y	z	U_{eq}	x	y	z	U_{eq}
Si(1)	436 (4)	1684 (3)	963 (5)	803 (32)	428 (3)	1669 (3)	931 (4)	123 (25)	436 (3)	1638 (3)	866 (4)	257 (23)
Si(2)	363 (4)	3193 (3)	890 (5)	742 (33)	375 (3)	3199 (3)	889 (4)	127 (25)	375 (3)	3238 (3)	830 (4)	278 (24)
Si(3)	198 (3)	3598 (3)	2909 (3)	783 (28)	199 (3)	3615 (3)	2908 (4)	125 (25)	215 (3)	3686 (3)	2969 (4)	276 (24)
Si(4)	-718 (4)	2528 (4)	3550 (5)	802 (33)	-739 (3)	2533 (3)	3552 (4)	126 (24)	-782 (3)	2530 (3)	3618 (4)	299 (25)
Si(5)	-219 (3)	1270 (3)	2708 (5)	805 (29)	-222 (3)	1259 (3)	2714 (4)	142 (25)	-236 (3)	1190 (3)	2736 (4)	274 (24)
X(11)	2067 (1)	1210 (1)	1173 (1)	940 (9)	2087 (1)	1201 (1)	1158 (1)	166 (6)	2054 (1)	1180 (1)	1128 (2)	309 (9)
X(12)	-515 (1)	1131 (1)	-978 (1)	953 (9)	-526 (1)	1115 (1)	-1009 (1)	164 (6)	-526 (1)	1119 (1)	-1020 (2)	317 (9)
X(21)	-1057 (1)	3623 (1)	-545 (1)	1064 (9)	-1067 (1)	3627 (1)	-583 (1)	203 (7)	-1044 (1)	3648 (1)	-610 (2)	366 (10)
X(22)	1804 (1)	3791 (1)	382 (2)	1130 (14)	1825 (1)	3798 (1)	378 (1)	225 (7)	1785 (1)	3823 (1)	352 (2)	378 (10)
X(31)	1773 (1)	3457 (1)	4205 (2)	1277 (14)	1778 (1)	3472 (1)	4217 (1)	274 (7)	1765 (1)	3562 (1)	4264 (2)	441 (11)
X(32)	-452 (1)	5005 (1)	3013 (2)	1208 (14)	-476 (1)	5031 (1)	3009 (1)	243 (7)	-442 (2)	5053 (1)	3035 (2)	413 (11)
X(41)	-2368 (1)	2827 (1)	2581 (1)	1005 (9)	-2397 (1)	2823 (1)	2570 (1)	184 (6)	-2419 (1)	2788 (1)	2623 (2)	349 (10)
X(42)	-499 (1)	2453 (1)	5812 (1)	1033 (9)	-502 (1)	2441 (1)	5836 (1)	197 (6)	-550 (1)	2436 (1)	5800 (2)	345 (10)
X(51)	1049 (1)	633 (1)	4245 (1)	1032 (9)	1060 (1)	624 (1)	4259 (1)	193 (6)	1057 (1)	603 (1)	4226 (2)	339 (10)
X(52)	-1571 (1)	321 (1)	2214 (1)	962 (9)	-1592 (1)	299 (1)	2208 (1)	168 (6)	-1557 (1)	245 (1)	2226 (2)	321 (9)

Table 3. Geometrical parameters of the Si₅ rings in the three structures [(A) RT-Si₅I₁₀, (B) LT-Si₅I₁₀, (C) LT-Si₅Br₁₀]

Out-of-plane deviations refer to a least-squares plane through the Si₅ ring.

	(A)	(B)	(C)
Bond lengths (Å)			
Si(1)–Si(2)	2.384 (7)	2.389 (6)	2.354 (6)
Si(2)–Si(3)	2.360 (7)	2.348 (7)	2.349 (6)
Si(3)–Si(4)	2.328 (8)	2.340 (6)	2.334 (6)
Si(4)–Si(5)	2.358 (8)	2.362 (7)	2.346 (6)
Si(5)–Si(1)	2.380 (7)	2.390 (7)	2.380 (6)
Bond angles (°)			
Si(1)–Si(2)–Si(3)	104.5 (3)	105.5 (2)	106.0 (2)
Si(2)–Si(3)–Si(4)	104.6 (3)	104.4 (2)	103.7 (2)
Si(3)–Si(4)–Si(5)	105.1 (3)	104.8 (2)	105.1 (2)
Si(4)–Si(5)–Si(1)	106.3 (3)	106.6 (2)	106.3 (2)
Si(5)–Si(1)–Si(2)	106.2 (3)	105.5 (2)	105.7 (2)
Torsion angles (°)			
Si(1)–Si(2)–Si(3)–Si(4)	-34.3 (3)	-33.7 (3)	-33.6 (3)
Si(2)–Si(3)–Si(4)–Si(5)	36.1 (3)	36.3 (3)	36.2 (3)
Si(3)–Si(4)–Si(5)–Si(1)	-23.8 (3)	-25.0 (3)	-25.1 (3)
Si(4)–Si(5)–Si(1)–Si(2)	2.5 (3)	4.3 (3)	4.3 (3)
Si(5)–Si(1)–Si(2)–Si(3)	19.3 (3)	17.9 (3)	18.0 (3)
Out-of-plane deviations (Å)			
Si(1)	0.081 (6)	0.064 (6)	0.065 (5)
Si(2)	-0.260 (6)	-0.249 (6)	-0.249 (6)
Si(3)	0.351 (5)	0.348 (5)	0.346 (5)
Si(4)	-0.292 (6)	-0.300 (5)	-0.300 (5)
Si(5)	0.120 (5)	0.137 (6)	0.138 (5)

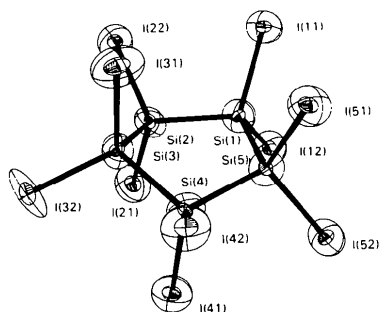


Fig. 1. ORTEP drawing (Johnson, 1976) of the crystal structures of RT-Si₅I₁₀ and LT-Si₅I₁₀.

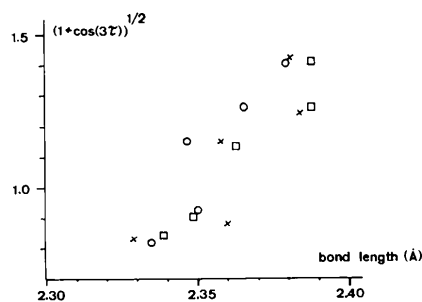


Fig. 2. Plot of $[1 + \cos(3\tau_{ijk})]^{1/2}$ vs r_{jk} for the crystal structures of LT-Si₅I₁₀ (squares), LT-Si₅Br₁₀ (circles) and RT-Si₅I₁₀ (crosses).

summarized in Table 4. The value of φ (mod 36) allows a classification of the ring in terms of envelope [φ (mod 36) = 0°] and twist [φ (mod 36) = 18°] forms, and the data in Table 2 show the three Si₅X₁₀ structures to be intermediate but closer to the envelope than the twist form. While the puckering amplitudes are the same for Si₅Br₁₀ and Si₅I₁₀, the Si₅ ring of the perphenylated derivative Si₅Ph₁₀ is significantly more puckered.

In any non-planar isogonal five-membered ring, individual endocyclic bond angles have to deviate from the mean over the five angles (Table 4) for the ring to be geometrically possible (Dunitz, 1972; Dunitz & Waser, 1972). We note a positive correlation between observed bond angles and the absolute value of the torsion angle about the opposite bond for each of the five structures. That this is an inherent property of any pseudorotating five-membered ring can be illustrated by the following line of arguments (Dunitz, 1984).

In a five-membered ring, every 1–3 distance is also a 1–4 distance. Thus, for an equilateral pentagon with side R , the square of the distance between, say, atoms 1 and 3 can be computed from R and the bond angle $\theta(=\theta_2)$ as

$$d^2 = 4R^2 \sin^2(\theta/2).$$

Table 4. Summary of five-membered-ring parameters for five crystal structures

For a definition of q and φ see text. $\langle\theta\rangle$ is the average of the five endocyclic bond angles, $r(\theta, |\tau|)$ the correlation coefficient between endocyclic bond angles and the absolute value of the opposite torsion angles.

	RT-Si ₃ I ₁₀	LT-Si ₃ I ₁₀	LT-Si ₃ Br ₁₀	RT-Si ₃ Ph ₁₀	RT-As ₃ Me ₁₀
q	0.545 (6)	0.544 (6)	0.543 (5)	0.634 (4)	0.865 (5)
φ	-69.4 (9)	-66.3 (9)	-66.1 (8)	-97.2 (6)	-152.7 (4)
$\varphi \pmod{36}$	2.6 (9)	5.7 (9)	5.9 (8)	10.8 (6)	28.0 (4)
$\langle\theta\rangle$	105.3 (4)	105.4 (4)	105.4 (5)	104.5 (6)	101.8 (16)
$r(\theta, \tau)$	0.80	0.83	0.94	0.88	0.75

The same quantity can also be obtained from the 1-4 distance, using the dihedral angle $\tau(=\tau_{3456})$ and assuming that $\theta_4 = \theta_5 = \bar{\theta}$, the average over all five bond angles,

$$d^2 = R^2(1 - 2\cos\bar{\theta})^2 + 4R^2\sin^2\bar{\theta}\sin^2(\tau/2).$$

Equating the two right sides, squaring and rearranging yields:

$$\cos\theta = \frac{1}{2} - (1 - \cos\bar{\theta})^2 + \sin^2\bar{\theta}\cos\tau.$$

With $\bar{\theta} = 105.35^\circ$, the value observed in the three halogenide structures, we obtain

$$\cos\theta = 0.93 \cos\tau - 1.1.$$

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The Structure of Lazurite, Ideally $\text{Na}_6\text{Ca}_2(\text{Al}_6\text{Si}_6\text{O}_{24})\text{S}_2$, a Member of the Sodalite Group

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Abstract. Cubic, $P\bar{4}3n$, $Z = 1$, two different specimens: (1) Afghanistan, $\text{Na}_{8.56}(\text{Al}_6\text{Si}_6\text{O}_{24})(\text{SO}_4)_{1.56}\text{S}_{0.44}$, $M_r = 1075.14$, $a = 9.105$ (2) Å, $V = 754.8$ Å³, $D_x = 2.39$ Mg m⁻³, graphite-monochromatized Mo $K\alpha$ radiation, $\lambda K\alpha = 0.71069$ Å,

$\mu = 1.18$ mm⁻¹, $F(000) = 530$; (2) Baffin Island, $\text{Na}_{8.16}(\text{Al}_6\text{Si}_6\text{O}_{24})(\text{SO}_4)_{1.14}\text{S}_{0.86}$, $M_r = 1039.06$, $a = 9.054$ (1) Å, $V = 742.2$ Å³, $D_x = 2.42$ Mg m⁻³ graphite-monochromatized Cu $K\alpha$ radiation, $\lambda K\alpha = 1.54178$ Å, $\mu = 9.31$ mm⁻¹, $F(000) = 512$. R factors

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