

The Synthesis and Molecular Structure of Tetra(isopropyl)silane

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Tetra(isopropyl)silane has been prepared using literature methods, with the individual steps improved by changes in some of the experimental conditions. The key reagent 2-lithiopropene, which can now be obtained in good yields from 1-methacrylic acid via 1,2-dibromo-1-methylpropionic acid and 2-bromopropene by treatment of the latter with ultrasound-activated lithium metal, was shown to contain mono- and dilithiopropene. The reaction with chlorotrimethylsilane led to the corresponding silylated derivatives, while with silicon tetrachloride tetra(isopropenyl)silane was obtained, which after purification is easily converted into the title compound by catalytic hydrogenation. — The gas phase molecular structure of $[(\text{CH}_3)_2\text{CH}]_4\text{Si}$ has been determined by electron diffraction. The parameters could be successfully refined for a model of S_4 symmetry. Bond distances Si—C, C—C, and C—H as well as bond angles Si—C—C and C—C—H show the steric compression of the four isopropyl substituents. Steric strain is minimized by twists of the methyl groups and the isopropyl groups away from the fully staggered conformations, but also by an increase of two of the C—Si—C angles as compared to the remaining four, which are decreased relative to the tetrahedral standard. The structure differs strongly (mainly in the twist angles) from that of the isoelectronic tetra(isopropyl)phosphonium cation in $[(\text{CH}_3)_2\text{CH}]_4\text{P}^+ [\text{B}(\text{C}_6\text{H}_5)_4]^-$, but is very similar to those of tetra(cyclohexyl)silane and of tri(isopropyl)phosphonium isopropylide, where the pyramidal configuration of the ylidic carbon atoms leads to a pseudo homoleptic array of the substituents at phosphorus.

Ground state conformations of simple molecules with a tetrahedrally coordinated central atom and a homoleptic set of ligands of various symmetry characteristics are an area of current interest¹⁾. The results of pertinent studies allow a rationalisation of physical properties, crystal packing, and reactivity of these molecules.

Work in this laboratory has originally focused on species where four substituents with C_3 symmetry (threefold rotors) are present at the tetrahedral center. The tetrakis(*tert*-butyl)phosphonium cation $[(\text{CH}_3)_3\text{C}]_4\text{P}^+$ served as a unique example²⁾ for these studies, since attempts to synthesize other (tBu)₄M compounds were not successful. By X-ray crystallography of the iodide salt it was found that the maximum possible symmetry T_d was reduced to the point group T as predicted by theory based on force-field calculations³⁾.

Darstellung und Molekülstruktur von Tetra(isopropyl)silan

Tetra(isopropyl)silan wurde nach Literaturvorschriften hergestellt, deren einzelne Stufen aber durch veränderte Reaktionsbedingungen verbessert werden konnten. Das Schlüsselreagens 2-Lithiopropen wurde ausgehend von 1-Methacrylsäure über 1,3-Dibrom-1-methylpropionsäure und 2-Brompropen durch Metallierung des letzteren mit ultraschallaktiviertem Lithium erhalten. Die Lösungen enthalten noch Mono- und Dilithiopropin, wie durch Silylierung mit Chlorotrimethylsilan bestätigt werden konnte. Mit SiCl_4 entsteht Tetra(isopropenyl)silan, das nach Reinigung katalytisch zur Titelverbindung hydriert werden kann. — Die Molekülstruktur von $[(\text{CH}_3)_2\text{CH}]_4\text{Si}$ in der Gasphase wurde durch Elektronenbeugung bestimmt. Eine Verfeinerung der Parameter war mit einem Modell der Punktgruppe S_4 erfolgreich. Die Bindungslängen Si—C, C—C und C—H zeigen ebenso wie die Winkel Si—C—C und C—C—H die Beugung der vier Substituenten an. Die sterische Spannung wird durch Herausdrehen der Methylgruppen und der Isopropylgruppen aus den jeweiligen gestaffelten Konformationen sowie durch Aufweitung zweier C—Si—C-Winkel vermindert. Die vier restlichen C—Si—C-Winkel sind entsprechend kleiner als der Tetraederwinkel. Die Gesamtstruktur unterscheidet sich in den Drehwinkeln C—Si—C—H deutlich von der des Tetra(isopropyl)phosphonium-Kations in $[(\text{CH}_3)_2\text{CH}]_4\text{P}^+ [\text{B}(\text{C}_6\text{H}_5)_4]^-$, die viel größere Winkel C—P—C—H aufweist. Sie ist aber den Strukturen des Tetra(cyclohexyl)silans und sogar des Tri(isopropyl)phosphonium-isopropylids sehr ähnlich, in dem die pyramidale Konfiguration am ylidischen C-Atom zu einer pseudo-homoleptischen Ligandenanordnung führt.

In a related study⁴⁾ the conformation of the tetrakis(isopropyl)phosphonium cation $[(\text{CH}_3)_2\text{CH}]_4\text{P}^+$ was investigated⁵⁾, where the four substituents at the phosphonium center have only local mirror symmetry (C_s). X-ray crystallography of the $[\text{B}(\text{C}_6\text{H}_5)_4]^-$ salt showed that the cation approached S_4 symmetry in this lattice, though crystallographically only a twofold axis is required. This result was in agreement with findings⁶⁾ for molecules of the type $\text{C}(\text{CH}_2\text{X})_4$, and also for tetra(cyclohexyl)silane⁷⁾ where, after reconsideration of the data, this point group was confirmed⁸⁾.

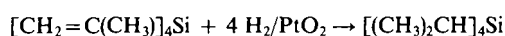
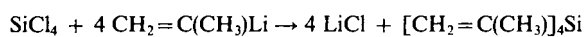
In the crystals of the $[\text{B}(\text{C}_6\text{H}_5)_4]^-$ salt the isopropyl groups of the $[(\text{CH}_3)_2\text{CH}]_4\text{P}^+$ cation are rotated pairwise in opposite directions away from the fully staggered conformation, which would represent D_{2d} symmetry. It remained

an open question whether this distortion could be a consequence of the non-isotropic environment of the cation in the crystal, or whether the intrinsic force field actually required the lowering of the symmetry. This problem is obviously related to the distortion $T_d \rightarrow T$ in the *tert*-butyl analogue. However, while the non-existence of a neutral molecule like $[(CH_3)_3C]_4Si$ has precluded an experimental study of this problem in the gas phase, the availability of the isopropyl silicon analogue $[(CH_3)_2CH]_4Si$ makes such a study amenable for the isopropyl case. We have therefore prepared pure tetrakis(isopropyl)silane and determined its gas phase structure by electron diffraction.

Synthesis and NMR Spectra

As observed previously by other workers⁸⁻¹¹, the preparation of the title compound is not easily accomplished. The obvious "classical" synthesis from a silicon tetrahalide and an isopropyl Grignard or lithium reagent proves very difficult probably owing to the steric hindrance of the transition state of the last substitution step. Yields are generally low and purification of the product is tedious.

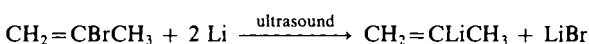
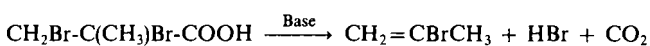
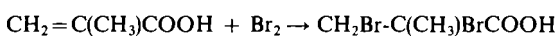
For this study the method first developed by Seyferth et al. proved most satisfactory, where sterically less demanding isopropenyl groups are first introduced at silicon, and then hydrogenated to give the tetra(isopropyl)silane. Yields are 60 and 85%, respectively.



There are problems in this synthesis, however, connected with the preparation of the isopropenyllithium required in the process. Solutions of this compound are generally obtained through the reaction of 2-bromopropene with lithium metal in diethyl ether solvent¹¹. The success of this procedure strongly depends on a number of parameters, and sometimes yields and quality of the products are very unsatisfactory. Experimental conditions were therefore sought under which most of the difficulties can be overcome, including also an improved preparation of the 2-bromopropene starting material.

In the preparation of 2-bromopropene, the literature method was modified to give better yields: For this purpose the bromination of 1-methacrylic acid was carried out at $-15^\circ C$, which led to an increase of the yield from 32¹² or 44%¹³ to 90%. For the decarboxylation/dehydrobromination of the dibrominated acid 4-picolin was employed instead of pyridine¹³, which also improved the yield (from 52% to 77%).

The preparation of isopropenyllithium could be improved by the application of ultrasound activation of the lithium metal. Yields of 85% have thus been obtained, but the resulting solutions contain a mixture of organolithium compounds.



In test runs, the reaction of the $LiC(CH_3)=CH_2$ solution in diethyl ether with chlorotrimethylsilane has produced three major components: $(CH_3)_3SiC(CH_3)=CH_2$, $(CH_3)_3SiC \equiv CCH_3$, and $(CH_3)_3SiC \equiv CCH_2SiC(CH_3)_3$, indicating 1-lithio-1-propyne and 1,3-dilithiopropyne as the main impurities.

A set of mixed substituted products was thus also to be expected in the reaction with $SiCl_4$, and great care was therefore taken to produce a pure tetraisopropenylsilane precursor for hydrogenation. Fractional distillation proved sufficient for the separation of $[CH_2=C(CH_3)]_4Si$ from the by-products. GLC analysis and the NMR spectra (see Experimental below) showed no significant contamination of the final samples.

Pure tetrakis(isopropyl)silane crystallizes from ethanol in colorless needles, m.p. $27^\circ C$, the crystal quality of which has not yet allowed a structure determination by X-ray diffraction. Efforts in this direction are continued. The vapour pressure of the compound at slightly elevated temperatures is sufficient, however, to carry out the gas phase structure determination by electron diffraction.

The Molecular Structure of Tetra(isopropyl)silane

It was assumed that tetra(isopropyl)silane contained four structurally identical isopropyl groups, and had these groups oriented in such a way that the overall molecular symmetry was S_4 , D_2 or, as a special case of these, D_{2d} . Torsional angles about the Si-C bonds were allowed to vary widely, and two structures fitting the data fairly well were identified. In one of these the symmetry was D_2 , and the isopropyl groups were twisted ca. 80° away from D_{2d} configuration, while the second had S_4 symmetry and a twist angle of ca. 15° . After careful refinement of both these models the D_2 conformation was rejected, as it led to a higher R_G factor (0.165 as opposed to 0.109 for the S_4 symmetry), and it also had an unreasonably short C...C contact between two methyl groups of 2.99 Å, as well as two amplitudes of vibration which were unacceptable.

The molecular dimensions for the S_4 model $[(CH_3)_2CH]_4Si$ are summarized in Table 1. The Si-C distance of 1.919(2) Å is significantly longer than the standard value of 1.87 Å, clearly due to the steric crowding in the molecule. Even the C-C distance of 1.549(1) Å is on the long side of the normal range, as the congestion is still noticeable in the periphery of the molecule. It should be noted that the standard deviations of these parameters are small and that the values can thus be assumed to be of relatively high precision, as no constrictions have been applied for these parameters. On the other hand, all C-H bonds were assumed to be equal, the methyl groups were assigned local C_{3v} symmetry, and no tilting was allowed, which makes the parameters relating to the hydrogen atoms less accurate.

The Si-CC₂ groups were assumed to have local mirror symmetry, and thus the angles Si-C9-C1 and Si-C9-C5 etc. are equal. (See Figure 1 for the atomic numbering.) The value for Si-C-C of $113.3(3)^\circ$ again reflects the steric crowding, and, accordingly, the C-C-C angles of $108.5(5)^\circ$ are smaller than the tetrahedral angle. The angle

C—C—H of $112.4(7)^\circ$ in the methyl groups is larger than the tetrahedral angle, obviously also as a consequence of compression. The angle Si—C—H at the unique CH group of the isopropyl substituents was not refined.

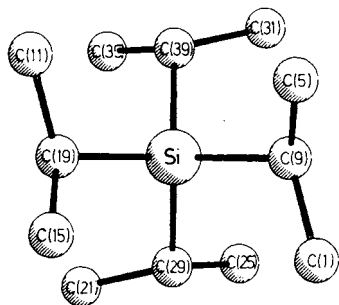


Figure 1. Projection of the tetra(isopropyl)silane molecule down the S_4 axis with atomic numbering

The “C—C twist” defines the conformation of the methyl groups: 0° is fully eclipsed, and 60° is fully staggered. The 29° twist found in the analysis minimizes the $H \cdots H$ interactions, which would be much less favorable in the staggered arrangement.

Regarding the angles C—Si—C, the refining parameter was C9—Si—C19, which equals C29—Si—C39 at $115.7(7)^\circ$. The four remaining C—Si—C angles are dependent parameters at $106.6(4)^\circ$. It is this distortion which is clearly most important in reducing steric strain in the molecule by bringing non-bonding carbon atoms as far apart as possible (3.65 to 3.90 Å). (See Figure 2, where hydrogen atoms have been included).

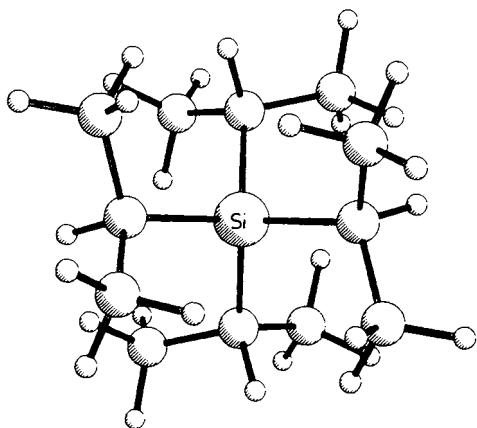


Figure 2. The same projection as in Figure 1 including hydrogen atoms

Finally, the “Si—C twist” refers to the conformation of the isopropyl groups: 0° would be fully staggered, but again this arrangement involves unfavorable interactions, such as between C5 and C11 or C1 and C15 in Figure 1. The 15° twist makes the $C1 \cdots C15$ and $C5 \cdots C15$ contacts comparable. This twist of ca. 15° is also found in molecules of the type $M(XR_3)_4$ and related compounds¹¹. Replacing the unique CH_3 hydrogen atoms in $[(CH_3)_2CH]_4Si$ by methyl

groups would result in a predicted structure of the (unknown) molecule $[(CH_3)_2C]_4Si$ with an Si—C twist similar to that found in $[(CH_3)_2C]_4P^+$ cations². The *tert*-butyl groups in *tert*-Bu₃PO and *tert*-Bu₃PNH also have similar twist angles¹⁴.

The “Si—C” twist in $[(CH_3)_2CH]_4Si$ is much smaller, however, than the large “P—C” twist in the $[(CH_3)_2CH]_4P^+$ cation in the crystals of the $[B(C_6H_5)_4]^-$ salt⁵. The two dihedral angles C—P—C'—H' (C_2 symmetry) are 71.8 and -66.4° , as compared to C—Si—C'—H' = -14.8° in the title compound (e.g. H—C9—Si—C19).

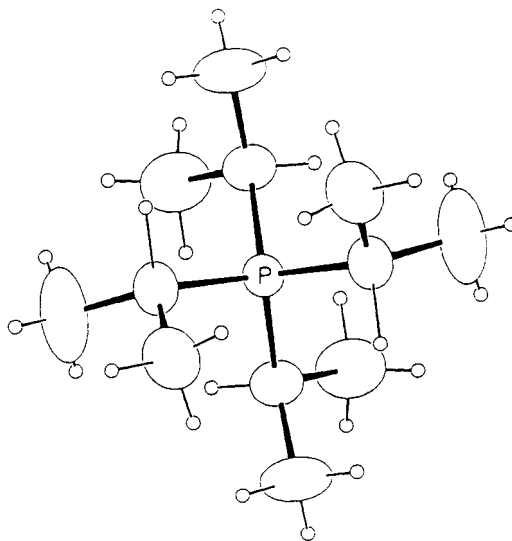


Figure 3. Structure of the tetra(isopropyl)phosphonium cation in crystals of its $[B(C_6H_5)_4]^-$ salt

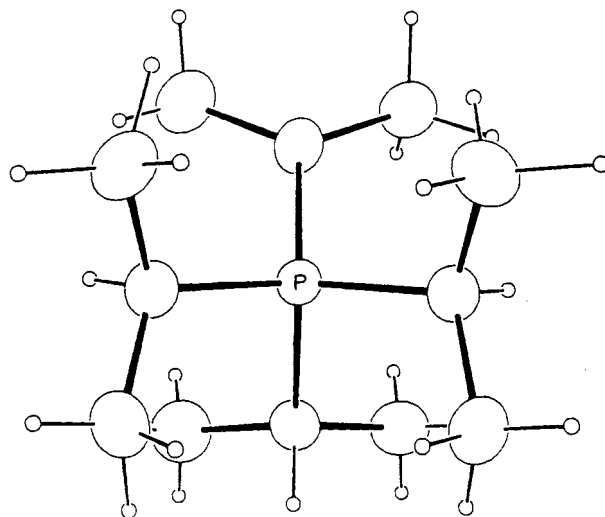


Figure 4. Molecular structure of tri(isopropyl)phosphonium isopropylide

A comparison of Figures 2 and 3 illustrates this difference in the conformations of the two isoelectronic species. On the other hand it turns out that the corresponding phosphorus ylide, tri(isopropyl)phosphonium propylide $[(CH_3)_2CH]_3P=C(CH_3)_2$, has almost the same structure as

tetra(isopropyl)silane, as shown in Figures 2 and 4. It was noted in the discussion of the structure of this ylide⁴⁾ that the pyramidal configuration of the isopropylidene carbon atom renders isopropyl and isopropylidene groups sterically very similar which leads to an almost homoleptic environment at phosphorus. In other words, after deprotonation of one of the isopropyl groups in $[(\text{CH}_3)_2\text{CH}]_4\text{P}^+$ at the α -carbon atom with retention of the pyramidal configuration at this atom (i.e. leaving a lone pair of electrons in the position of the hydrogen atom), the four isopropyl(idene) groups are in positions which approach D_{2d} symmetry. And it is this conformation, characterized by only small "Si/P—C" twist angles, which is also found in tetra(isopropyl)silane. It therefore appears, that the much stronger distortions in the tetra(isopropyl)phosphonium cation observed in the crystal of the tetraphenylborate salt are to a large extent induced by packing forces, while the structures of gaseous $[(\text{CH}_3)_2\text{CH}]_4\text{Si}$ and of $[(\text{CH}_3)_2\text{CH}]_3\text{P}=\text{C}(\text{CH}_3)_2$ in crystals with weak intermolecular forces between almost ball-shaped molecules represent true unperturbed molecular ground state conformations.

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Experimental

General: The experiments were carried out under an atmosphere of pure dry nitrogen. Solvents were purified, dried, and saturated with nitrogen, and glassware was oven-dried and filled with nitrogen. Spectrometers: Jeol GX 270 and GX 400 (^1H , ^{13}C , and ^{29}Si NMR).

1,2-Dibromo-1-methylpropionic Acid: A solution of 1-methacrylic acid (982 g, 11.4 mol) in 2 l of carbon disulfide was cooled to -30°C , irradiated with a 300 W lamp (VIS), stirred vigorously, and slowly treated with bromine (1.73 kg, 10.8 mol). During the addition (3 h) the temperature did not exceed -15°C . The end of the addition was noted by the persistence of the bromine colour in the solution. The precipitate was filtered at -60°C , recrystallized from hot pentane and kept in a vacuum for 1 h at 100°C to remove traces of CS_2 . The yield was 2.39 kg (90%), m.p. $47.5-49^\circ\text{C}$ (ref.¹³); yield 44%, m.p. $46-48^\circ\text{C}$.

2-Bromopropene: Molten 1,2-dibromo-1-methylpropionic acid (1.40 kg, 5.70 mol) was added with stirring to 2.3 l of γ -picolin (23 mol) kept at 80°C . Gas was evolved and passed through traps kept at -20 and -78°C . After the addition the reaction mixture was heated briefly to 125°C to ensure completion. The product collected in the traps was redistilled to give 530 g (77%) of product, b.p. $47-49^\circ\text{C}$ (ref.¹³); yield 52%, b.p. $48-49^\circ\text{C}$.

2-Lithiopropene: Lithium turnings (37 g, 5.4 mol) were covered with 1 l of diethyl ether and activated by ultrasound. This mixture was then treated with 2-bromopropene (150 ml, 1.71 mol) while ultrasound irradiation was continued for a total of 2.5 h. Slow reflux was maintained during the addition and the mixture finally exposed to ultrasound for an additional 0.5 h. The brown reaction mixture was kept at -25°C over night, filtered, and the concentration of the orange-coloured filtrate determined by double titration¹⁵. Yield 1.3 mol in 1 l of the solution (85%).

Trimethylsilylation of the 2-Lithiopropene Solution: Aliquots of the solution were treated with a small excess of chlorotrimethylsilane, the precipitate filtered, and the filtrate distilled and analyzed by GLC and NMR spectroscopy.

2-(Trimethylsilyl)propene: Yield 45%, b.p. $79-83^\circ\text{C}$. — ^1H NMR (CDCl_3): $\delta = 0.06$ (s, 9H, CH_3Si), 1.81 (br. s, 3H, CH_3C), 5.24 and 5.55 (m, CH_2). — $\{^1\text{H}\}^{13}\text{C}$ NMR (C_6D_6): $\delta = -2.41$ (s, CH_3Si), 22.13 (s, CH_3C), 124.87 and 147.79 (s, $\text{C}=\text{C}$).

1-Trimethylsilyl-1-propyne: Yield 8%, b.p. $33-34^\circ\text{C}/60$ Torr. — ^1H NMR (CDCl_3): $\delta = 0.11$ (s, 9H, CH_3Si), 1.83 (s, 3H, CH_3C). — $\{^1\text{H}\}^{13}\text{C}$ NMR (C_6D_6): $\delta = -0.06$ (s, CH_3Si), 4.23 (s, CH_3C), 83.9 and 103.05 (s, $\text{C}\equiv\text{C}$). — $\{^1\text{H}\}^{29}\text{Si}$ NMR (C_6D_6): $\delta = 4.80$.

1,2-Bis(trimethylsilyl)propyne: Yield 4%, b.p. $83-84^\circ\text{C}/50$ Torr. — ^1H NMR: $\delta = 0.126$ (s, 18H, CH_3Si), 1.51 (s, 2H, CH_2). — $\{^1\text{H}\}^{13}\text{C}$ NMR (C_6D_6): $\delta = -2.18$ and 0.55 (s, CH_3Si), 8.86 (s, CH_2), 83.20 and 105.67 (s, $\text{C}\equiv\text{C}$). — $\{^1\text{H}\}^{29}\text{Si}$ NMR (C_6H_6): $\delta = -19.92$ and 2.59 (s).

Tetra(2-propenyl)silane: The preparation followed the literature method¹¹. Yield 55%, b.p. $74-76^\circ\text{C}/11$ Torr (ref.¹¹) $111-115^\circ\text{C}/55$ Torr). — ^1H NMR (CDCl_3): $\delta = 1.87$ (m, 3H), 5.41 and 5.81 (m, 2H). — $\{^1\text{H}\}^{13}\text{C}$ NMR (CDCl_3): $\delta = 23.31$ (s, CH_3), 129.75 (s, CH_2), 141.80 (s, $\text{Si}-\text{C}=\text{C}$). — IR (film, cm^{-1}): $\tilde{\nu} = 1615$ and 1600 , $\nu(\text{C}=\text{C})$.

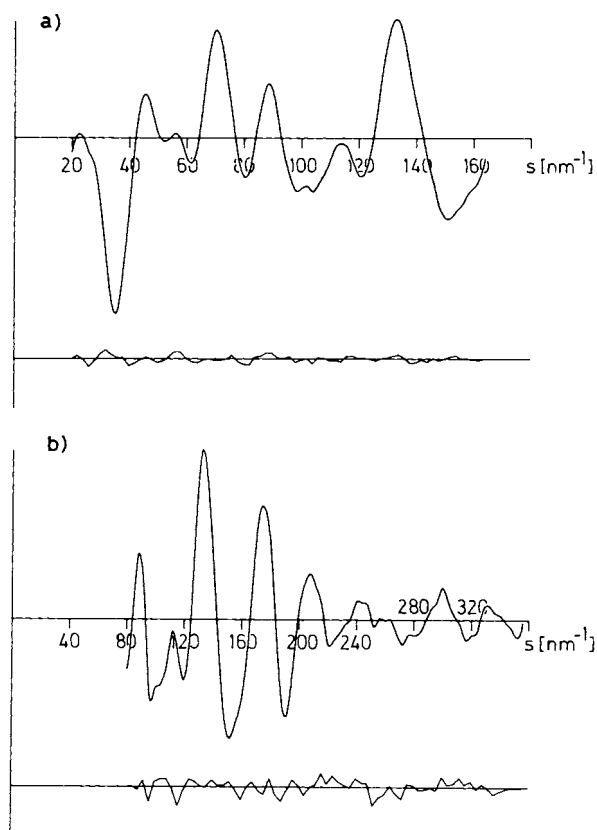


Figure 5. Observed and final weighted difference molecular scattering intensities for nozzle-to-plate distances of a) 258.5 mm and b) 95.5 mm

Tetra(2-propenyl)silane: The preparation followed the literature method¹¹. Yield 72%, b.p. $78-80^\circ\text{C}/7$ Torr (ref.¹¹) $104-106^\circ\text{C}/20$ Torr). — ^1H NMR (C_6D_6): $\delta = -1.10$ and 1.08 [AB_6 , $J(\text{AB}) = 7.4$ Hz]. — $\{^1\text{H}\}^{13}\text{C}$ NMR (CDCl_3): $\delta = 19.27$ (s, CH_3), 11.33 (CH_2).

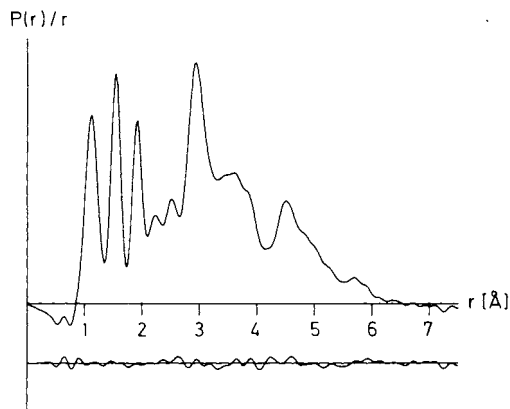


Figure 6. Observed and difference radial distribution curves, $P(r)/r$. Before Fourier inversion the data were multiplied by $s \cdot \exp(-0.00002s^2)/(Z_{Si} - f_{Si})(Z_C - f_C)$.

Table 1. Molecular parameters (distances [Å], angles [°]) of tetra(isopropyl)silane. Note: Errors quoted in this and the following table are estimated standard deviations, increased to allow for systematic errors

p_1	$r(\text{Si}-\text{C})$	1.919(2)
p_2	$r(\text{C}-\text{C})$	1.549(1)
p_3	$r(\text{C}-\text{H})$	1.120(2)
p_4	$\angle \text{SiCC}$	113.3(3)
p_5	$\angle \text{CCC}$	108.5(5)
p_6	$\angle \text{CCH}(\text{methyl})$	112.4(7)
p_7	$\angle \text{SiCH}$	110.0(fixed)
p_8	$\text{C}-\text{C}$ twist ^{a)}	29.0(2)
p_9	$\angle \text{CSiC}^{\text{a)}$	115.7(7)
	$\angle \text{CSiC}^{\text{a)}$	106.4(4) ^{b)}
p_{10}	$\text{Si}-\text{C}$ twist ^{a)}	14.8(7)

^{a)} For definition see text. — ^{b)} Dependent parameter.

Table 2. Weighting functions and other experimental data pertinent to the electron diffraction structure determination

Camera distance [mm]	Δs	s_{min}	sw_1 [nm ⁻¹]	sw_2	s_{max}	Correlation parameter	Scale factor	Electron wavelength [Å]
258.5	2	20	40	140	164	0.376	0.762(13)	0.05686
95.5	4	80	100	304	356	0.203	0.535(17)	0.05685

Electron Diffraction: Electron diffraction scattering data were recorded on Kodak Electron Image photographic plates using the Edinburgh gas diffraction apparatus¹⁶⁾, with the sample held at 75°C and the nozzle at 81°C, and an accelerating voltage of 44.5 kV. Two plates were obtained at the long camera distance (258.5 mm) and four at the short distance (95.5 mm), and data were also recorded for benzene to provide calibration of the distances and electron wavelength. A Joyce-Loebl MDM6 microdensitometer¹⁷⁾ at the SERC Daresbury Laboratory was used to give the data in digital form, and subsequent data reduction¹⁷⁾ and least-squares refinement¹⁸⁾ were performed using standard programs and scattering factors¹⁹⁾. Parameters used to define the off-diagonal weight matrices used in the refinements are given in Table 2, with other experimental data.

The geometrical parameters obtained in the refinements are listed in Table 1, while Table 3 gives interatomic distances and vibrational parameters, and Table 4 contains the most significant elements of

the least-squares correlation matrix. The observed and final difference molecular scattering intensities and radial distribution curves are shown in Figures 5 and 6, respectively.

Table 3. Interatomic distances [Å] and amplitudes of vibration [Å] of tetra(isopropyl)silane. Note: Other Si...H, C...H and H...H distances were also included in the refinements, but are not listed here

		Distance [Å]	Amplitude [Å]
r_1	Si-C	1.919(2)	0.056(3)
r_2	C-C	1.549(1)	0.051(3)
r_3	C-H	1.120(2)	0.080(4)
r_4	Si...C	2.904(3)	0.109(6)
r_5	C(1)...C(5)	2.515(5)	0.067(8)
r_6	C(9)...C(19)	3.251(10)	0.086(25)
r_7	C(9)...C(29)	3.075(5)	0.086(25)
r_8	C(1)...C(19)	3.900(10)	0.272(14)
r_9	C(5)...C(19)	3.605(14)	0.272(14)
r_{10}	C(1)...C(29)	3.305(8)	0.272(14)
r_{11}	C(9)...C(25)	3.624(11)	0.272(14)
r_{12}	C(1)...C(15)	3.657(16)	0.272(14)
r_{13}	C(5)...C(15)	3.873(28)	0.272(14)
r_{14}	C(1)...C(25)	3.903(14)	0.272(14)
r_{15}	C(5)...C(29)	4.465(5)	0.094(7)
r_{16}	C(1)...C(11)	4.819(14)	0.136
r_{17}	C(1)...C(21)	4.703(7)	0.136
r_{18}	C(5)...C(21)	5.702(6)	0.185
r_{19}	C(5)...C(25)	5.121(11)	0.185

Table 4. Least-squares correlation matrix ($\times 100$)^{a)}

p_4	p_5	u_6	k_1	k_2	
-57					p_1
		66			p_4
	60				p_6
				57	u_1
				66	u_2
			53	53	u_3
	-55	75	57		u_4

^{a)} Only elements with absolute values above 50 are listed.

CAS Registry Numbers

CH₂=C(CH₃)COOH: 79-41-4 / CH₂BrC(CH₃)BrCOOH: 33673-74-4 / SiCl₄: 10026-04-7 / CH₂=CBrCH₃: 557-93-7 / CH₂=CLi-CH₃: 6386-71-6 / (CH₃)₃SiC(CH₃)=CH₂: 18163-07-0 / (CH₃)₃Si-C≡CCH₃: 6224-91-5 / (CH₃)₃SiC≡CCH₂Si(CH₃)₃: 21752-80-7 / [CH₂=C(CH₃)₂]₄Si: 76240-85-2 / [(CH₃)₂CH]₄Si: 3429-55-8

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