Further spectroscopic investigations will be carried out in order to explain the nature of the hydrogen bond in solution.

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SHORT COMMUNICATIONS

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Acta Cryst. (1978). B**34**, 3494

Refinement of Nb_3Si and Nb_3As: errata.

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In Table 5 of the paper by Waterstrat, Yvon, Flack & Parthé [*Acta Cryst.* (1975), B**31**, 2765–2769] the positional parameters which are presented for the compound Ta_3P (reference 11) are, in fact, those of the compound Zr_3P.

All the relevant information is contained in the Abstract.

Acta Cryst. (1978). B**34**, 3494–3496

The crystal structure of tetrakis(pentafluorophenyl)silane.

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The crystal structure of $(C_6F_5)_4Si$ has been determined from three-dimensional single-crystal X-ray data collected on a computer-automated diffractometer. The compound crystallizes in space group $I4_1/a$ with cell dimensions of a = 17·165 (12), c = 8·125 (8) Å and Z = 4. The $(C_6F_5)_4Si$ molecules have S_4 crystallographically imposed symmetry. Full-matrix least-squares refinement yielded a conventional R factor of 0·070.

The molecular symmetry, crystal packing and energetics of tetra(aryl) Group IV*a* compounds have been the object of numerous recent investigations (Karipides & Oertel, 1977; Hutchings, Andose & Mislow, 1975; Robbins, Jeffrey, Chesick, Donohue, Cotton, Frenz & Murillo, 1975; Hutchings, Nourse & Mislow, 1974; Ahmed, Kitaigorodsky & Mirskaya, 1971). As part of our program to study the structural characteristics of such compounds we have determined the crystal structures of the pentafluorophenyl derivatives of Ge and Sn (Karipides, Forman, Thomas & Reed, 1974) and in this paper report on the crystal structure of tetrakis(pentafluorophenyl)silane.

The title compound was kindly supplied by Dr C. Tamborski (Tamborski, Soloski & Dec, 1965) and suitable crystals were obtained by recrystallization from benzene. Indexed Weissenberg photographs revealed tetragonal symmetry and the observed absences $h + k + l = 2n + 1$ for hkl; h, (k) = $2n + 1$ for $hk0$; $l = 4n + 1$ for $00l$ uniquely determined the space group to be $I4_1/a$. The pertinent crystal data are given in Table 1.

Table 1. *Crystal and structure-analysis data for* $(C_6F_5)_4Si$

λ(Mo $K\alpha$) = 0·71069 Å	M_r = 696·3
a = 17·165 (12) Å	$F(000)$ = 1352
c = 8·125 (8)	μ(Mo $K\alpha$) = 2·90 cm^{-1}
V = 2394 Å^3	D_o = 2·0 g cm^{-3}
Number of independent data, $I > \sigma(I)$ = 923	D_c = 1·93
	R = 0·070
Crystal size (mm), 0·14 × 0·14 × 0·32	R_w = 0·058
	Goodness of fit = 1·59

Table 2. *Final positional parameters and their estimated standard deviations* (× 10^4)

	x	y	z
Si	0	0	0
C(1)	653 (2)	−578 (2)	1412 (4)
C(2)	760 (2)	−1385 (2)	1382 (5)
C(3)	1229 (2)	−1775 (2)	2487 (5)
C(4)	1597 (2)	−1366 (3)	3720 (5)
C(5)	1510 (2)	−566 (3)	3812 (5)
C(6)	1048 (2)	−201 (2)	2683 (4)
F(2)	414 (1)	−1824 (1)	214 (3)
F(3)	1319 (2)	−2550 (1)	2378 (3)
F(4)	2044 (2)	−1740 (2)	4627 (3)
F(5)	1873 (1)	−167 (2)	5013 (3)
F(6)	978 (1)	584 (1)	2802 (3)

Three-dimensional X-ray intensity data out to 52° in 2θ were collected using Zr-filtered Mo $K\alpha$ radiation on a Picker FACS-1 diffractometer and reduced to values of F_o and $\sigma(F_o)$ in the manner previously described (Karipides, Forman, Thomas & Reed, 1974). Absorption corrections were not applied. The space group, morphology and cell dimensions suggested that $(C_6F_5)_4Si$ was isostructural with the corresponding Ge compound (Karipides, Forman, Thomas & Reed, 1974). Hence the coordinates of $(C_6F_5)_4Ge$ along with appropriate isotropic temperature factors were used as the starting point in the solution of the structure. After initial isotropic refinement the complete trial structure was refined by a full-matrix least-squares procedure assuming anisotropic thermal motion for all atoms. The function minimized was $\sum w(F_o - F_c)^2$ where the weight, w, was taken to be $[1/\sigma(F_o)]^2$. The final agreement indices, $R = \sum(|F_o| - |F_c|)/\sum |F_o|$ and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2}$, are given in Table 1. There were 923 data and 102 variable parameters yielding a data:parameter ratio of 9·0:1. An analysis of $w(|F_o| - |F_c|)^2$ as a function of $|F_o|$ or $\sin\theta/\lambda$ revealed no dependence on these quantities suggesting the weighting scheme used was adequate. A final difference Fourier map showed no residual electron density greater or less than 0·20 e Å^{-3}. The atomic scattering factors for all elements were taken from Cromer & Mann (1968); corrections for f' and f'' for Si were made (Cromer & Liberman, 1970). The final atomic positional parameters are presented in Table 2.*

Table 3. *Bond distances* (Å) *and angles* (°)

Si–C(1)	1·866 (4)	C(6)–C(1)	1·394 (5)
C(1)–C(2)	1·398 (5)	C(2)–F(2)	1·350 (4)
C(2)–C(3)	1·379 (5)	C(3)–F(3)	1·343 (4)
C(3)–C(4)	1·377 (6)	C(4)–F(4)	1·345 (4)
C(4)–C(5)	1·383 (6)	C(5)–F(5)	1·346 (5)
C(5)–C(6)	1·365 (5)	C(6)–F(6)	1·357 (4)
C(1)–C(2)–C(3)	123·1 (4)	F(2)–C(2)–C(1)	120·5 (3)
C(2)–C(3)–C(4)	119·6 (4)	F(2)–C(2)–C(3)	116·3 (3)
C(3)–C(4)–C(5)	119·7 (4)	F(3)–C(3)–C(2)	120·4 (4)
C(4)–C(5)–C(6)	118·9 (4)	F(3)–C(3)–C(4)	120·0 (4)
C(5)–C(6)–C(1)	124·5 (4)	F(4)–C(4)–C(3)	120·4 (4)
C(6)–C(1)–C(2)	114·1 (3)	F(4)–C(4)–C(5)	120·0 (4)
C(1)–Si–C(1)($\bar{x}\bar{y}z$)	105·1 (2)	F(5)–C(5)–C(4)	119·6 (4)
C(1)–Si–C(1)($\bar{y}x\bar{z}$)	111·7 (1)	F(5)–C(5)–C(6)	121·5 (4)
Si–C(1)–C(2)	126·1 (3)	F(6)–C(6)–C(5)	117·4 (4)
Si–C(1)–C(6)	119·7 (3)	F(6)–C(6)–C(1)	118·1 (3)

The molecular symmetry of $(C_6F_5)_4Si$ in the crystalline state is S_4. The molecular S_4 axis is coincident with the crystal c axis. The crystal and molecular structure resembles that of the corresponding Ge and Sn compounds as shown in Figs. 1, 2 and 3 of Karipides, Forman, Thomas & Reed (1974), and represents another example of the preference of S_4 molecular symmetry for symmetrical Ar$_4M$ compounds in the crystalline state (Hutchings, Andose & Mislow, 1975; Karipides & Oertel, 1977). Intramolecular bond distances and angles and given in Table 3.

There is only one pentafluorophenyl ring in the asymmetric unit. This ring is planar within experimental error; the equation of the unit-weighted least-squares plane is $0{\cdot}7912X + 0{\cdot}1235Y - 0{\cdot}5989Z - 0{\cdot}0746 = 0$. The largest deviations of any of the six C atoms or five F atoms from this plane are 0·009 Å (C3) and 0·02 Å (F2), respectively. The Si atom lies −0·07 Å from this plane. However, the aryl ring is distorted from idealized geometry as indicated by the large variation in bond lengths and angles, although the individual values cited in Table 3 are in close agreement with those observed in other pentafluorophenyl compounds (Karipides, Forman, Thomas & Reed, 1974). An important exception is the apical C(2)–C(1)–C(6) angle of 114·1 (3)°, which is significantly smaller than the 116·1 (7)° found for the corresponding angle in tetraphenylsilane (Glidewell & Sheldrick, 1971). This decrease in the magnitude of the apical angle in the perfluorophenylsilane is consistent with the strong σ-electron withdrawing properties of a pentafluorophenyl ring and supports the arguments of Domenicano, Vaciago & Coulson (1975*a*,*b*) concerning the skeletal deformations of substituted benzenes.

The conformation of the pentafluorophenyl ring in the molecule is specified by the angle between the aryl ring plane and the C(1)–Si–C(1)($\bar{x}\bar{y}z$) plane. This angle is 51·9° in $(C_6F_5)_4Si$ and corresponds closely to that found in $(C_6F_5)_4Ge$ (51·9°) and $(C_6F_5)_4Sn$ (53·2°) indicating that this torsion angle is independent of the central-atom size. The Si–C(1) bond length is 1·866 (4) Å.

The crystal structure consists of discrete $(C_6F_5)_4Si$ molecules separated by normal van der Waals distances. The shortest intermolecular F···F and F···C nonbonded contacts in the structure are F(4)···F(4)($\bar{y}$, $x-\frac{1}{2}$, $\frac{1}{4}+z$) 2·959 (3) Å and F(3)···C(3)($\bar{y}$, $x-\frac{1}{2}$, $\frac{1}{4}+z$) 3·080 (5) Å.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33760 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The shortest intramolecular nonbonded distances are 2·863 (3) and 2·903 (4) Å for the F(2)···F(6)($y\bar{x}\bar{z}$) and F(2)···C(6)($y\bar{x}\bar{z}$) contacts, respectively.

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Acta Cryst. (1978). B**34**, 3496–3497

Comments on *A study of the crystal structure of* $(CH_3NH_3)_2MnCl_4$ *by neutron diffraction at* 188 ± 1 K and *A study of the crystal structure of* $(CH_3NH_3)_2MnCl_4$ *by neutron diffraction at* 404 K by I. Mikhail.

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The new refinements reported by Mikhail [*Acta Cryst.* (1977), B33, 1317–1321, 1321–1325] of the tetragonal low-temperature and tetragonal high-temperature phases of $(CH_3NH_3)_2MnCl_4$ and the arguments leading to the assignment of space groups different from those proposed earlier seem to be doubtful.

The two papers by Mikhail (1977*a*,*b*) show a variety of discrepancies with respect to our previous results on $(CH_3NH_3)_2MnCl_4$ and $(CH_3NH_3)_2CdCl_4$. In this comment we would like to discuss some specific points which lead us to doubt the conclusions given by Mikhail (1977*a*,*b*).

(1) It should first be mentioned that the same neutron diffraction data have already been used for complete structure determinations (Heger, Mullen & Knorr, 1975, 1976).

(2) In his papers, Mikhail has not mentioned the dominant role of the incoherent neutron scattering by hydrogen and the necessity for correcting the measured intensity data for absorption. With an effective linear absorption coefficient of μ = 2·46 cm^{-1} for $(CH_3NH_3)_2MnCl_4$ and crystal dimensions of approximately 4·60 × 4·95 × 1·35 mm a correction for absorption is necessary (Heger *et al.*, 1975). Mikhail based his argument and calculations on absorption-corrected *and* averaged data, which were handed to him.

(3) Mikhail describes a desymmetrization of the published structures, assuming subgroups of the space groups used earlier: for $(CH_3NH_3)_2MnCl_4$ at 404 K: *I*4/*m* instead of *I*4/*mmm* (HTT phase), and at 188 K: *Pccn* instead of $P4_2/ncm$ (LTT phase). The author supports this by noting 'marked inconsistencies' with the diffraction symmetry 4/*mmm*. However, the maximum differences between the absorption-corrected intensities of symmetry-related reflections were within 10%, but the corresponding value for the uncorrected measured intensities was 150% (Heger *et al.*, 1975). Moreover, Mikhail used for his refinements independent intensities with respect to the higher symmetry averaged according to 4/*mmm* (the same data as Heger *et al.*, 1975, 1976).

(4) In the re-refinement of the LTT phase in space group *Pccn*, which is a subgroup of the previously assumed symmetry $P4_2/ncm$, the ratio of the number of observations to the number of refined parameters is only 2·5. 183 independent reflections with respect to tetragonal symmetry were taken from Heger *et al.* (1976). This represents only half of the data required for orthorhombic symmetry. A refinement as described by Mikhail is admissible only if the crystal is, in fact, an orthorhombic single crystal and if the intensities used are not averaged over equivalent reflections with respect to tetragonal symmetry. In this case, a loss of information due to the incompleteness of the data is still to be taken into account. In all other cases, the least-squares program must be modified since the intensity of a reflection is given by $I = q|F_{hkl}|^2 + (1-q)|F_{khl}|^2$, q and $(1-q)$ being the relative amounts of the two twin components contributing to the measured or averaged value. Derivatives of I with respect to the structural parameters and q are easily calculated. In the absence of any such modification, the new parameters cannot describe the real orthorhombic structure, even if it should exist.

(5) The re-refinement of the HTT phase in space group *I*4/*m* may be questioned on the basis of group-theoretical