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The Structures of α-Naphthylphenylmethylsilanes, Optically Active Silicon Compounds, and Their Absolute Configurations†

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Optically active α-naphthylphenylmethylsilane, α-$C_{10}H_7 . C_6H_5 . CH_3 . Si^*H$ and its fluoroderivative, α-$C_{10}H_7 . C_6H_5 . CH_3 . Si^*F$, crystallize in the orthorhombic system with space group $P2_12_12_1$; the cell dimensions are: $a=8\cdot72$, $b=19\cdot89$, $c=7\cdot88$ Å for the H compound and $a=8\cdot77$, $b=19\cdot89$, $c=8\cdot10$ Å for the F compound. Three-dimensional integrated intensity data for these crystals were collected on a computer-controlled diffractometer operated by an IBM 1620 machine under a stored data collection program. In order to determine the absolute configuration, Cu *Kα* radiation was used for the dextrorotatory F compound; the data for the H compound were obtained with Mo *Kα* radiation.

The effect of overcrowding around the central asymmetric Si atom is clearly demonstrated in buckling of the α-naphthyl group; the Si – C (of α-naphthyl) bond then bends out of the naphthyl plane by about 0·2 Å. Other features of the conformation of the molecules are also discussed.

The absolute configuration of the F compound was determined by refining the structure in the two possible enantiomorphs; the configuration refining to the lower residuals (by 12%) agrees with that assigned by a chemical method.

Introduction

α-Naphthylphenylmethylsilane, α-$C_{10}H_7C_6H_5CH_3SiH$, and its fluoro and chloro derivatives (hereafter called α-SiH, SiF and SiCl, respectively) are the first optically active silicon compounds with asymmetric silicon atoms synthesized and resolved. The crystal structures of these compounds were studied in order to correlate and confirm results of various chemical studies on their configurations, steric interactions between the substituents and their absolute configurations. The present paper deals with the detailed crystal structures of α-SiH and SiF; unfortunately, no work was done on the chloro compound, which is less stable in the air.

The present study is a complete rework of the previous results (Ashida, Pepinsky & Okaya, 1963). The analysis is based on complete three-dimensional intensity data collected by using CCXD, a computer-con-

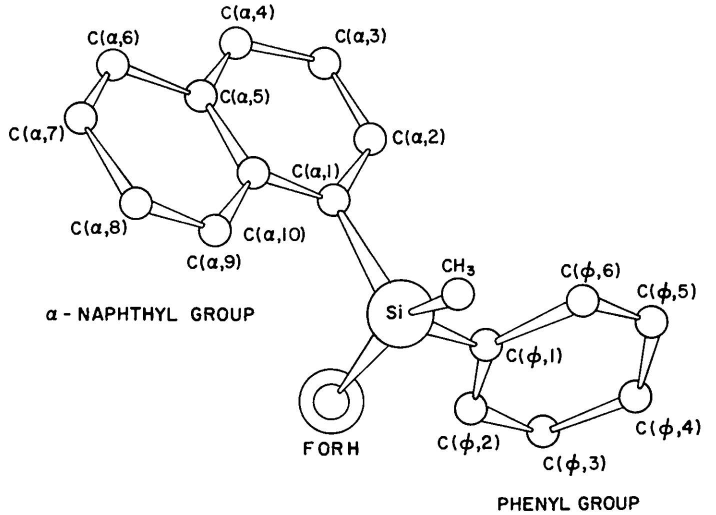

Fig. 1. α-SiX, molecular configuration and numbering of atoms. The absolute configuration of this conformation corresponds to (+)-α-SiH, (+)-α-SiF and (−)-α-SiCl.

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trolled diffractometer (Cole, Okaya & Chambers, 1963), and the refinement was carried out with the use of anisotropic temperature factors; the contribution of hydrogen atoms is also included in the computation. The chemical formula of α-SiF and the numbering of the atoms which will be used in this article are shown in Fig. 1.

An interesting case of isomorphism was found among the crystals of the molecules of the same hand and their mixed crystals (Sommer, Frye, Musolf, Parker, Rodewald, Michael, Okaya & Pepinsky, 1961); the isomorphism was used in confirming the configurations determined by various chemical as well as physico-chemical methods (see, *e.g.* a review article by Sommer, 1962).

These silanes are overcrowded molecules because of the existence of large groups around the central silicon atoms. It is interesting to study the effect of such overcrowding on the conformation; especially, the effect of the hydrogen atom on C(α, 9), to be called an *epi*-hydrogen to the Si atom, on the planarity of the α-naphthyl group.

Experimental

A single crystal of (+)-α-SiF was ground into a sphere and mounted on a General Electric Goniostat which is a part of CCXD, a computer-controlled diffractometer operated by an IBM 1620 machine under a stored data-taking program (Okaya, 1964). Cu $K\alpha$ radiation was used to utilize the imaginary part of Si in establishing the absolute configuration of the molecule. The three-dimensional data for $\sin\theta < 0{\cdot}94$ were recorded by the diffractometer. Only one octant of the Ewald sphere was recorded; however, as discussed later it was sufficient in determining the absolute configuration. For each reflection, the settings for the ω and χ axes were optimized by scanning around the calculated values; the integrated intensity was then recorded by the $(\theta-2\theta)$ step scan method. The computer program chooses the step scan range in such a way that the counts at the starting and the end of the scan represent the background count at the 2θ value of the reflection. In the course of the ω scanning, the maximum and minimum counts of a reflection were recorded, and if the difference between these two counts was less than the statistical fluctuation the reflection was treated as a non-observed one. The three-dimensional data for (−)-α-SiH were also collected in the same manner; the radiation used for this crystal was Mo $K\alpha$. The integrated intensity of a reflection was calculated from the data on count, time, and the attenuator factor for each step; this calculation is performed as a time-shared problem on the IBM 1620 computer (Okaya, 1964). The integrated intensity data thus obtained were corrected for the Lorentz-polarization factors; correction for the absorption effect was made for the data on the fluoro compound ($\mu r = 1{\cdot}2$). The cell dimensions were measured on the diffractometer and used as input data for the intensity collection program; they are shown in Table 1.* For each crystal, about 80% of the reflections measured on the diffractometer (~1400) were observed.

Table 1. *Cell constants of* α-SiX *determined on the diffractometer*

Space group $P2_12_12_1$

	a	b	c
H	$8{\cdot}72_7$ Å	$19{\cdot}89_6$ Å	$7{\cdot}88_4$ Å
F	$8{\cdot}77_4$	$19{\cdot}89_0$	$8{\cdot}10_5$

The refinements of the structures were carried out with a block-diagonal least-squares program on an IBM 7094 computer (Okaya, 1963). The approximate parameters given in the previous report were used as the starting values for the refinements; the previous isotropic temperature factors were transformed into anisotropic temperature factors. For each crystal, after several refinement cycles without hydrogen atoms, their positions were studied by the usual $F_o - F_c$ synthesis method. The atomic coordinates thus obtained for hydrogen atoms with isotropic thermal parameters (5·0 Å^2) were then included in the refinement cycles. After a reasonable number of iterations, when changes in the residual became negligible, the refinement was halted and the parameters at that stage were used for the subsequent discussion. The final error indices, $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$, were about 0·07 for the two crystals, excluding the non-observed reflections. The parameters are listed in Tables 2 and 3; comparison between the observed and calculated structure factors is shown in Table 4. The atomic scattering factors used in the computation were obtained from the tables in *International Tables for X-ray Crystallography*. The weighting schemes used for the two crystals are: $\omega = 1{\cdot}0$ for $|F_{\text{obs}}| \le 20$, $20/|F_{\text{obs}}|$ for larger structure factors and weights of zero for non-observed reflections. The crystals are molecular and no force other than van der Waals force exists in the crystals. The atoms undergo rather strong thermal vibration at room temperature, and unfortunately the standard deviations of atomic coordinates are relatively high. For each crystal, in the course of intensity measurements which took about three weeks, approximately 20% of the original volume was lost by decomposition and/or evaporation. The correction for this decay was made by setting day-to-day decay factors obtained by measuring a standard set of reflections. Nonetheless, these effects contribute to error in the intensity measurement and eventually to an increase in standard deviations.

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* There is a disparity between the cell constants quoted in the earlier work and the values in Table 1. The latter dimensions were used in the data collection on the diffractometer and their errors are estimated to be less than 0·5 %. Since the data used to derive the earlier values are not accessible to the authors, they are unable to discuss the rather large discrepancies.

Table 2

(*a*) SiF. Atomic coordinates in fractions of cell edges and their standard deviations in 10^{-4} Å. These atomic coordinates when used in the left-hand coordinate system correspond to the absolute configuration of (+)-α-SiF.

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Si	0·7749	20	0·1707	19	0·7112	18
F	0·8649	38	0·1282	40	0·8501	39
C(α, 1)	0·7766	59	0·1192	56	0·5156	56
C(α, 2)	0·6410	57	0·1115	55	0·4361	57
C(α, 3)	0·6340	68	0·0784	70	0·2762	78
C(α, 4)	0·7637	74	0·0564	62	0·2034	73
C(α, 5)	0·9072	65	0·0647	54	0·2832	71
C(α, 6)	1·0462	76	0·0422	67	0·2044	79
C(α, 7)	1·1800	77	0·0484	69	0·2793	73
C(α, 8)	1·1860	67	0·0767	69	0·4453	78
C(α, 9)	1·0565	63	0·0989	60	0·5209	66
C(α, 10)	0·9115	54	0·0944	52	0·4407	59
C(φ, 1)	0·5770	52	0·1818	58	0·7836	59
C(φ, 2)	0·4938	70	0·1271	68	0·8400	71
C(φ, 3)	0·3419	79	0·1337	80	0·8887	76
C(φ, 4)	0·2726	72	0·1935	82	0·8810	68
C(φ, 5)	0·3506	82	0·2486	78	0·8272	79
C(φ, 6)	0·5029	71	0·2437	64	0·7761	77
CH_3	0·8770	70	0·2499	64	0·6823	75
H(α, 2)	0·520		0·135		0·481	
H(α, 3)	0·550		0·067		0·242	
H(α, 4)	0·774		0·029		0·102	
H(α, 6)	1·006		0·029		0·070	
H(α, 7)	1·286		0·035		0·241	
H(α, 8)	1·285		0·090		0·526	
H(α, 9)	1·064		0·126		0·634	
H(φ, 2)	0·523		0·082		0·853	
H(φ, 3)	0·322		0·095		0·908	
H(φ, 4)	0·167		0·200		0·925	
H(φ, 5)	0·335		0·287		0·823	
H(φ, 6)	0·572		0·288		0·744	

(*b*) Thermal parameters. The β's are used in the expression: $\exp\{-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+hk\beta_{12}+hl\beta_{13}+kl\beta_{23})\}$.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Si	0·019913	0·004094	0·020395	−0·000647	−0·000175	0·000032
F	0·025115	0·005969	0·022151	0·002524	−0·004779	0·000704
C(α, 1)	0·019249	0·003706	0·019422	−0·000455	0·001772	0·001026
C(α, 2)	0·016066	0·004326	0·021316	0·000783	0·001941	0·000671
C(α, 3)	0·017064	0·005894	0·030966	−0·000989	−0·003797	−0·002246
C(α, 4)	0·024799	0·004131	0·023185	−0·004022	0·001879	−0·001098
C(α, 5)	0·021239	0·003246	0·022780	−0·001193	0·000899	0·000013
C(α, 6)	0·023329	0·004581	0·030835	−0·000183	0·009702	−0·001434
C(α, 7)	0·020839	0·005292	0·032394	0·003797	0·006533	0·000326
C(α, 8)	0·018025	0·005003	0·032425	0·001256	−0·002852	0·001485
C(α, 9)	0·019813	0·004284	0·023527	0·002197	0·002010	0·001752
C(α, 10)	0·018994	0·003235	0·022206	0·001769	0·002909	0·001215
C(φ, 1)	0·022381	0·003776	0·017410	0·000595	−0·006686	−0·002023
C(φ, 2)	0·022893	0·004128	0·030759	0·001337	0·009174	0·001318
C(φ, 3)	0·025265	0·005410	0·027502	−0·001141	0·005065	0·000705
C(φ, 4)	0·021259	0·006631	0·022123	0·001735	0·000450	−0·003648
C(φ, 5)	0·028342	0·005254	0·030633	0·005514	−0·000097	−0·001106
C(φ, 6)	0·022710	0·004457	0·024805	0·002389	0·000473	−0·000517
CH_3	0·024762	0·004373	0·032713	−0·002705	0·007439	−0·001498

Table 3

(*a*) Atomic coordinates of SiH. For explanation of the coordinate system, see the legend of Table 2.

	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
Si	0·7685	17	0·1714	18	0·7207	16
C(α, 1)	0·7674	50	0·1199	45	0·5232	48
C(α, 2)	0·6329	50	0·1100	55	0·4372	48
C(α, 3)	0·6305	58	0·0761	62	0·2779	55
C(α, 4)	0·7599	62	0·0541	55	0·2082	56
C(α, 5)	0·9011	52	0·0630	47	0·2877	49
C(α, 6)	1·0388	55	0·0397	58	0·2168	56
C(α, 7)	1·1717	54	0·0472	60	0·2951	62
C(α, 8)	1·1799	53	0·0768	59	0·4542	59
C(α, 9)	1·0495	59	0·1003	54	0·5292	49
C(α, 10)	0·9063	49	0·0948	43	0·4498	50
C(φ, 1)	0·5662	48	0·1833	42	0·7899	52
C(φ, 2)	0·4858	43	0·1296	48	0·8537	50
C(φ, 3)	0·3339	44	0·1356	59	0·8975	46
C(φ, 4)	0·2630	59	0·1965	59	0·8809	52
C(φ, 5)	0·3408	57	0·2508	51	0·8230	49
C(φ, 6)	0·4924	62	0·2448	51	0·7770	51
CH_3	0·8685	62	0·2517	59	0·6802	61
H(α, 2)	0·527		0·129		0·480	
H(α, 3)	0·536		0·077		0·231	
H(α, 4)	0·769		0·029		0·096	
H(α, 6)	1·028		0·020		0·087	
H(α, 7)	1·262		0·029		0·244	
H(α, 8)	1·275		0·086		0·502	
H(α, 9)	1·050		0·119		0·652	
H(φ, 2)	0·542		0·079		0·871	
H(φ, 3)	0·259		0·096		0·952	
H(φ, 4)	0·163		0·199		0·909	
H(φ, 5)	0·292		0·290		0·805	
H(φ, 6)	0·563		0·280		0·751	

(*b*) Thermal parameters

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Si	0·013256	0·003142	0·015323	−0·000535	−0·000253	−0·000304
C(α, 1)	0·010306	0·002369	0·016670	−0·000374	0·003274	0·000686
C(α, 2)	0·010951	0·003343	0·017988	−0·001269	0·000861	0·001154
C(α, 3)	0·014144	0·004872	0·020297	−0·002472	−0·006327	−0·001358
C(α, 4)	0·017265	0·003043	0·020966	−0·002627	−0·000421	−0·003189
C(α, 5)	0·013254	0·002299	0·019193	−0·001738	0·005073	−0·001087
C(α, 6)	0·014266	0·003075	0·028229	0·000675	0·008664	−0·002052
C(α, 7)	0·011990	0·003953	0·033266	0·001255	0·015123	0·001266
C(α, 8)	0·012590	0·003826	0·029296	0·000992	−0·000937	0·002325
C(α, 9)	0·013951	0·003367	0·018209	−0·000655	−0·000858	0·003144
C(α, 10)	0·012639	0·002182	0·019104	−0·000827	−0·001276	0·001415
C(φ, 1)	0·016172	0·003243	0·012723	0·000446	−0·002290	−0·001325
C(φ, 2)	0·016609	0·003244	0·021373	0·000691	0·003543	0·001112
C(φ, 3)	0·014934	0·004257	0·019599	−0·001735	0·004727	−0·001804
C(φ, 4)	0·013190	0·006285	0·015049	0·000220	0·003290	−0·004811
C(φ, 5)	0·018778	0·004008	0·021427	0·001989	−0·000681	−0·000230
C(φ, 6)	0·017151	0·003364	0·018104	0·000740	−0·000718	−0·000418
CH_3	0·026755	0·003588	0·020712	−0·005855	0·006592	−0·001356

Discussion

Bond distances, bond angles and other important intra and inter-molecular approaches have been calculated from the atomic coordinates in Tables 2 and 3; they are shown in Figs. 2, 3 and 4. Fig. 2 shows the general molecular conformation with some important interatomic distances between the substituent groups. It is apparent from these figures that C(α, 9) and especially its *epi*-hydrogen try to turn away from the α-naphthyl or methyl groups and come close to the hydrogen (or fluorine) atom which is the smallest of the four substituents. However, this conformation does not yet solve the problem of overcrowding, the effect

Table 4. *Comparison between the observed and calculated structure factors*

(*a*) α–SiH

H	K	L	FOBS	FCAL
0	2	0	171	171
	4	0	532	525
	6	0	761	772
	8	0	115	135
	10	0	248	254
	12	0	155	158
	14	0	340	333
	16	0	192	177
	18	0	35	36
	20	0	14	17
	22	0	0	5
	24	0	63	45
1	1	0	400	393
	2	0	0	17
	3	0	394	364
	4	0	627	644
	5	0	148	149
	6	0	333	341
	7	0	93	104
	8	0	0	10
	9	0	250	243
	10	0	84	88
	11	0	239	225
	12	0	40	40
	13	0	27	25
	14	0	53	56
	15	0	142	127
	16	0	54	60
	17	0	214	201
	18	0	126	115
	19	0	0	33
	20	0	10	25
	21	0	91	81
	22	0	39	12
	24	0	0	16
2	0	0	485	462
	1	0	308	333
	2	0	124	135
	3	0	582	550
	4	0	138	138
	5	0	0	3
	6	0	347	336
	7	0	250	254
	8	0	367	361
	9	0	304	281
	10	0	62	61
	11	0	197	174
	12	0	70	83
	13	0	26	40
	14	0	31	37
	15	0	27	27
	16	0	45	39
	17	0	56	55
	18	0	136	124
	19	0	0	8
	20	0	93	72
	21	0	36	32
	22	0	0	21
	23	0	0	2
	24	0	45	33
3	1	0	13	37
	2	0	126	118
	3	0	116	118
	4	0	45	59
	5	0	145	138
	6	0	120	135
	7	0	368	353
	8	0	105	107
	9	0	124	145
	10	0	133	134
	11	0	111	89
	12	0	68	79
	13	0	64	75
	14	0	16	[illegible]
	15	0	153	132
	16	0	9	27
	17	0	118	101
	18	0	58	67
	19	0	0	20
	20	0	0	4
	21	0	57	57
	22	0	0	26
	23	0	65	52
4	0	0	279	251
	1	0	379	377
	2	0	163	187
	3	0	183	173
	4	0	67	63
	5	0	252	240
	6	0	457	443
	7	0	99	102
	8	0	101	104
	9	0	93	95
	10	0	50	62
	11	0	64	57
	12	0	27	28
	13	0	122	113
	14	0	119	112
	15	0	85	84
	16	0	0	20
	17	0	0	20
	19	0	77	63
	20	0	75	63
	21	0	0	23
	22	0	0	20
5	1	0	98	116
	2	0	96	84
	3	0	189	192
	4	0	48	32
	5	0	152	143
	6	0	93	92
	7	0	77	79
	8	0	120	124
	9	0	268	253
	10	0	106	114
	11	0	0	26
	12	0	84	81
	13	0	0	26
	14	0	0	5
	15	0	0	12
	16	0	69	76
	17	0	109	101
	18	0	0	7
	19	0	27	22
	20	0	0	28
6	0	0	110	101
	1	0	89	91
	2	0	22	25
	3	0	0	5
	4	0	129	130
	5	0	211	203
	6	0	196	190
	7	0	51	49
	8	0	246	233
	9	0	0	6
	10	0	133	120
	11	0	24	9
	12	0	58	52
	13	0	0	10
	14	0	71	67
	15	0	0	25
	16	0	0	7
	17	0	25	14
	18	0	93	85
	19	0	75	67
7	1	0	364	362
	2	0	262	265
	3	0	80	74
	4	0	0	43
	5	0	137	138
	6	0	100	104
	7	0	58	47
	8	0	20	7
	9	0	83	78
	10	0	0	8
	11	0	0	13
	12	0	0	5
	13	0	0	10
	14	0	0	23
	15	0	48	43
	16	0	63	58
	17	0	51	55
	18	0	0	20
8	0	0	141	127
	1	0	149	127
	2	0	92	78
	3	0	178	172
	4	0	58	36
	5	0	0	15
	6	0	0	14
	7	0	107	109
	8	0	36	27
	9	0	90	81
	10	0	0	15
	11	0	0	7
	12	0	0	19
	13	0	38	38
	15	0	0	34
	16	0	55	44
9	1	0	28	34
	2	0	63	58
	3	0	42	30
	4	0	113	92
	5	0	32	28
	6	0	37	24
	7	0	8	6
	8	0	52	52
	10	0	40	48
10	0	0	84	66
	1	0	84	77
	2	0	0	18
	3	0	11	10
	4	0	0	13
	5	0	0	31
	6	0	0	37
0	1	1	302	257
	2	1	154	161
	3	1	768	827
	4	1	1818	1606
	5	1	131	108
	6	1	600	607
	7	1	483	485
	8	1	157	164
	9	1	252	251
	10	1	107	119
	11	1	0	34
	12	1	250	242
	13	1	166	177
	14	1	106	115
	15	1	0	18
	16	1	58	65
	17	1	89	86
	18	1	121	118
	19	1	0	36
	20	1	13	17
	21	1	31	22
	22	1	67	64
	24	1	41	24
1	0	1	827	807
	1	1	413	422
	2	1	461	464
	3	1	374	389
	4	1	343	343
	5	1	499	485
	6	1	300	307
	7	1	375	412
	8	1	65	64
	9	1	76	71
	10	1	249	230
	11	1	124	119
	12	1	165	145
	13	1	266	247
	14	1	75	83
	15	1	76	73
	16	1	87	82
	17	1	98	86
	18	1	29	40
	19	1	76	63
	20	1	23	25
	21	1	86	73
	24	1	0	0
2	0	1	316	350
	1	1	288	293
	2	1	177	170
	3	1	121	134
	4	1	487	461
	5	1	150	161
	6	1	84	100
	7	1	90	85
	8	1	209	221
	9	1	88	98
	10	1	218	210
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	9	2	114	146
	10	2	626	573
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	12	2	191	192
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	14	2	148	141
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	1	2	661	627
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	4	2	186	183
	5	2	202	198
	6	2	162	159
	7	2	256	263
	8	2	312	297
	9	2	524	510
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	11	2	242	234
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	13	2	172	169
	14	2	105	107
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	16	2	79	74
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	7	2	230	216
	8	2	250	246
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	10	2	80	80
	11	2	105	114
	12	2	176	171
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	15	2	44	43
	16	2	89	87
	17	2	81	73
	18	2	103	103
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	20	2	0	22
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	4	2	216	236
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	6	2	16	44
	7	2	116	121
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	9	2	175	193
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	11	2	71	75
	12	2	86	88
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Table 4 (*cont.*)

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	18	3	35	39
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	22	3	29	24
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	4	3	281	276
	5	3	397	407
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	7	3	240	244
	8	3	0	42
	9	3	57	65
	10	3	214	210
	11	3	96	101
	12	3	65	64
	13	3	134	132
	14	3	130	147
	15	3	100	102
	16	3	51	56
	17	3	86	102
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	19	3	0	33
	20	3	0	24
	21	3	44	36
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	7	3	143	127
	8	3	164	161
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	2	5	88	99
	3	5	93	95
	4	5	32	24
	5	5	0	57
	6	5	55	50
	7	5	44	45
	8	5	0	16
	9	5	35	37
	10	5	21	15
8	1	5	56	49
	3	5	63	53
	4	5	31	24
	5	5	0	47
	7	5	51	63
	8	5	33	45
	9	5	38	26
9	4	5	41	32
0	0	6	97	86
	1	6	53	47
	2	6	0	20
	3	6	205	180
	4	6	54	57
	5	6	134	122
	6	6	109	99
	7	6	139	136
	8	6	125	115
	9	6	51	60
	10	6		22
	12	6	31	39
	13	6	43	46
	15	6	27	41
	17	6	23	27
	19	6	22	14
1	0	6	112	96
	1	6	133	100
	2	6	56	47
	3	6	91	86
	4	6	25	15
	5	6	13	33
	6	6	101	94
	8	6	55	63
	9	6	51	55
	10	6	97	105
	11	6	61	63
	12	6	70	69
	13	6	77	83
	14	6	55	60
	15	6	79	75
	16	6	0	17
2	0	6	62	63
	1	6	104	102
	2	6	97	101
	3	6	224	219
	4	6	122	113
	5	6	161	158
	6	6	108	104
	7	6	54	61
	8	6	70	71
	9	6	110	115
	10	6	0	17
	11	6	65	58
	12	6	0	25
	13	6	0	20
	15	6	27	4
	17	6	67	73
	18	6	35	34
3	0	6	319	308
	1	6	38	24
	2	6	78	79
	3	6	28	19
	4	6	150	150
	5	6	122	131
	6	6	67	65
	7	6	35	24
	8	6	118	116
	9	6	79	79
	10	6	47	42
	11	6	0	5
	12	6	57	58
	14	6	79	78
	15	6	34	31
	18	6	57	58
4	0	6	12	1
	1	6	227	218
	2	6	179	178
	3	6	125	124
	4	6	130	136
	5	6	55	55
	6	6	0	48
	7	6	81	82
	8	6	0	31
	9	6	80	[illegible]
	10	6	54	61
	12	6	76	71
	13	6	38	37
	14	6	54	43
	15	6	16	17
	16	6	30	26
5	0	6	31	37
	1	6	40	34
	2	6	16	26
	3	6	44	43
	4	6	76	77
	5	6	32	33
	6	6	51	51
	7	6	0	32
	8	6	0	12
	9	6	34	34
	10	6	0	35
	12	6	32	30
6	0	6	26	23
	1	6	60	62
	2	6	52	62
	3	6	0	40
	4	6	77	78
	5	6	0	46
	6	6	0	35
	8	6	42	52
	9	6	50	43
	10	6	0	49
	12	6	27	28
7	0	6	25	13
	1	6	0	35
	2	6	0	35
	3	6	0	16
	4	6	41	28
	5	6	76	74
	6	6	73	73
	7	6	56	64
	8	6	17	15
	9	6	26	22
8	1	6	45	33
	3	6	47	42
	4	6	57	58
	5	6	50	49
	1	7	129	107
	2	7	73	69
	3	7	82	74
	4	7	56	43
	5	7	94	86
	6	7	47	35
	7	7	40	57
	9	7	62	68
	10	7	42	40
	12	7	21	19
1	0	7	32	17
	1	7	0	22
	2	7	113	107
	3	7	46	32
	4	7	79	71
	5	7	66	69
	6	7	0	24
	8	7	57	61
	9	7	48	46
	10	7	101	90
	11	7	37	37
	12	7	21	31
	13	7	33	43
	14	7	78	67
	16	7	26	36
2	0	7	40	35
	1	7	135	134
	2	7	72	76
	3	7	90	91
	4	7	52	43
	5	7	49	54
	6	7	59	58
	7	7	118	117
	8	7	51	49
	9	7	0	37
	10	7	43	48
	11	7	48	53
	12	7	0	30
	13	7	89	99
	15	7	33	27
3	0	7	27	16
	1	7	0	44
	2	7	22	9
	3	7	68	62
	4	7	197	193
	5	7	0	13
	6	7	50	41
	7	7	63	53
	8	7	101	98
	9	7	56	45
	10	7	81	74
	11	7	60	65
	12	7	58	61
4	0	7	62	60
	1	7	40	45
	2	7	56	54
	3	7	84	86
	4	7	48	45
	5	7	149	145
	6	7	82	77
	7	7	97	96
	8	7	11	31
	9	7	26	26
	10	7	42	51
	14	7	8	9
5	1	7	7	13
	2	7	0	5
	3	7	49	54
	4	7	50	44
	5	7	32	21
	7	7	0	15
	8	7	95	59
	10	7	36	49
	12	7	12	13
6	0	7	81	79
	2	7	0	21
	3	7	0	45
	5	7	19	24
	6	7	40	50
	9	7	26	18
	10	7	41	36
7	1	7	32	37
	2	7	45	42
	3	7	60	60
	5	7	28	11
0	0	8	48	37
	5	8	57	59
	6	8	62	67
	7	8	35	37
	9	8	64	69
1	0	8	74	76
	1	8	47	41
	2	8	41	40
	3	8	44	35
	4	8	41	45
	6	8	112	105
	8	8	40	52
	11	8	22	14
	12	8	48	39
2	0	8	27	26
	1	8	46	37
	2	8	46	36
	3	8	64	72
	4	8	29	29
	5	8	76	83
	9	8	84	82
	11	8	67	67
	12	8	41	34
3	0	8	111	110
	1	8	30	39
	2	8	34	35
	3	8	51	61
	4	8	8	5
	5	8	77	71
	6	8	86	88
	8	8	112	111
	10	8	40	46
4	0	8	84	78
	1	8	66	65
	3	8	39	52
	6	8	47	47
	8	8	48	50
	9	8	68	85
5	1	8	37	27
	2	8	27	27
	5	8	19	25
	7	8	52	50
6	2	8	36	29
	3	8	26	23

of which can be seen in deviations from the planarity of the α-naphthyl group. The deviations of atoms from the α-naphthyl group are shown in Fig. 5. Here in evaluating the equations of the α-naphthyl planes, the atoms placed in dotted regions were not included in calculations. As shown in the figure, the C(α, 8)–C(α, 9) and C(α, 1)–C(α, 2) edges buckle from the α-naphthyl plane in opposite directions. The Si–C(α, 1) bond then bends out of the plane in the direction of the buckling of the C(α, 1)–C(α, 2) bond. The bending of the Si–C(α, 1) bond is about 7° and represents a relatively large shift of the Si atom from the plane. The angles around C(α, 1) and C(α, 10) also contribute in increasing the separation between Si and the C(α, 8)–

Table 4 (*cont.*)

(*b*)α-SiF

H	K	L	FOBS	FCAL
0	2	0	165	173
	4	0	798	854
	6	0	746	756
	8	0	8	20
	10	0	220	234
	12	0	91	87
	14	0	261	247
	16	0	184	177
	18	0	23	24
	20	0	21	30
	24	0	34	27
1	1	0	486	527
	2	0	217	225
	3	0	539	552
	4	0	632	613
	5	0	249	257
	6	0	169	183
	7	0	230	232
	8	0	26	33
	9	0	148	154
	10	0	45	46
	11	0	129	135
	12	0	0	2
	13	0	0	13
	14	0	0	14
	15	0	75	79
	16	0	36	27
	17	0	158	143
	18	0	64	74
	19	0	3	11
	20	0	0	21
	21	0	62	58
	22	0	0	8
	24	0	8	10
2	0	0	447	462
	1	0	485	523
	2	0	63	71
	3	0	368	375
	4	0	195	195
	5	0	150	158
	6	0	342	350
	7	0	299	305
	8	0	340	336
	9	0	327	333
	10	0	39	34
	11	0	109	104
	12	0	51	56
	13	0	0	9
	14	0	0	30
	15	0	25	31
	16	0	12	21
	17	0	43	56
	18	0	81	82
	19	0	0	1
	20	0	47	42
	21	0	13	13
	22	0	0	16
	23	0	7	7
	24	0	12	13
3	1	0	71	61
	2	0	274	277
	3	0	10	19
	4	0	65	71
	5	0	159	165
	6	0	17	22
	7	0	236	241
	8	0	143	140
	9	0	122	121
	10	0	0	27
	11	0	51	55
	12	0	71	77
	13	0	0	39
	14	0	23	29
	15	0	114	111
	16	0	0	31
	17	0	59	57
	18	0	42	56
	19	0	22	34
	20	0	0	7
	21	0	35	32
	22	0	1	23
	23	0	29	20
4	0	0	0	2
	1	0	387	390
	2	0	67	80
	3	0	125	134
	4	0	71	67
	5	0	281	266
	6	0	312	323
	7	0	110	115
	8	0	186	173
	9	0	86	82
	10	0	34	44
	11	0	41	48
	12	0	31	18
	13	0	67	84
	14	0	62	61
	15	0	86	71
	16	0	0	2
	17	0	0	19
	18	0	58	50
	19	0	0	8
	20	0	38	26
	21	0	0	19
	22	0	0	6
5	1	0	0	3
	2	0	52	52
	3	0	82	72
	4	0	59	52
	5	0	152	161
	6	0	107	116
	7	0	0	12
	8	0	140	134
	9	0	201	195
	10	0	156	143
	11	0	23	32
	12	0	0	45
	13	0	40	45
	14	0	0	19
	15	0	0	17
	16	0	66	59
	17	0	62	47
	18	0	0	5
	19	0	0	6
	20	0	16	20
	21	0	0	10
6	0	0	36	37
	1	0	35	29
	2	0	0	5
	3	0	0	14
	4	0	46	43
	5	0	155	158
	6	0	129	118
	7	0	122	113
	8	0	210	183
	9	0	86	76
	10	0	66	61
	11	0	0	20
	12	0	60	59
	13	0	31	39
	14	0	0	39
	15	0	0	13
	16	0	0	11
	17	0	40	33
	18	0	0	35
	19	0	54	41
	20	0	5	2
7	1	0	241	210
	2	0	249	218
	3	0	34	33
	4	0	55	65
	5	0	0	68
	6	0	0	49
	7	0	0	54
	8	0	0	13
	9	0	57	54
	10	0	26	24
	11	0	0	2
	12	0	0	1
	13	0	0	6
	14	0	19	24
	15	0	10	11
	16	0	51	44
	17	0	0	16
	18	0	0	7
8	0	0	61	63
	1	0	116	101
	2	0	66	65
	3	0	92	88
	4	0	27	14
	5	0	0	4
	6	0	0	1
	7	0	51	49
	8	0	68	53
	9	0	47	41
	10	0	0	23
	11	0	0	20
	12	0	24	10
	13	0	35	28
	14	0	0	4
	15	0	21	13
	16	0	35	26
9	1	0	30	36
	2	0	0	30
	3	0	0	5
	4	0	65	48
	5	0	39	34
	6	0	0	14
	7	0	21	5
	8	0	46	35
	10	0	0	26
	12	0	0	1
10	1	0	58	50
	3	0	0	7
	5	0	38	24
0	1	1	149	158
	2	1	375	368
	3	1	632	664
	4	1	1639	1622
	5	1	141	142
	6	1	688	727
	7	1	380	396
	8	1	219	209
	9	1	277	286
	10	1	36	31
	11	1	21	13
	12	1	189	195
	13	1	186	186
	14	1	62	62
	15	1	0	13
	16	1	62	67
	17	1	80	73
	18	1	99	100
	19	1	0	19
	20	1	0	24
	21	1	0	16
	23	1	9	4
	24	1	0	11
1	0	1	656	673
	1	1	421	402
	2	1	354	346
	3	1	251	267
	4	1	383	392
	5	1	463	483
	6	1	203	197
	7	1	342	355
	8	1	147	143
	9	1	136	131
	10	1	154	142
	11	1	137	140
	12	1	124	114
	13	1	160	167
	14	1	36	48
	15	1	66	72
	16	1	75	68
	17	1	60	60
	18	1	0	16
	19	1	0	42
	20	1	0	14
	21	1	0	43
	22	1	0	13
2	0	1	447	448
	1	1	443	436
	2	1	156	147
	3	1	198	202
	4	1	517	515
	5	1	231	233
	6	1	76	79
	7	1	167	166
	8	1	248	242
	9	1	88	82
	10	1	180	185
	11	1	119	111
	12	1	124	131
	13	1	0	29
	14	1	91	86
	15	1	34	38
	16	1	61	60
	17	1	21	10
	18	1	0	9
	19	1	32	47
	20	1	0	24
	21	1	0	20
	22	1	34	28
	23	1	35	29
3	0	1	435	411
	1	1	36	50
	2	1	220	219
	3	1	233	225
	4	1	353	343
	5	1	178	172
	6	1	118	104
	7	1	53	62
	8	1	146	151
	9	1	179	181
	10	1	41	40
	11	1	131	134
	12	1	73	76
	13	1	113	105
	14	1	46	71
	15	1	0	36
	16	1	22	32
	17	1	0	38
	18	1	0	28
	19	1	62	61
	20	1	0	29
	23	1	0	13
4	0	1	207	197
	1	1	296	272
	2	1	227	227
	3	1	296	288
	4	1	136	131
	5	1	192	177
	6	1	106	104
	7	1	13	10
	8	1	77	71
	9	1	151	146
	10	1	177	164
	11	1	105	108
	12	1	0	37
	13	1	0	28
	14	1	86	87
	15	1	0	11
	16	1	43	39
	17	1	47	49
	18	1	50	37
	19	1	0	20
	20	1	0	12
	21	1	15	18
	22	1	15	19
5	0	1	80	89
	1	1	138	150
	2	1	39	41
	3	1	108	121
	4	1	82	92
	5	1	79	84
	6	1	208	204
	7	1	106	108
	8	1	184	180
	9	1	0	42
	10	1	0	49
	11	1	66	60
	12	1	93	87
	13	1	108	103
	14	1	0	25
	15	1	0	26
	16	1	0	24
	17	1	35	28
	18	1	47	43
	19	1	0	19
	20	1	0	25
	21	1	31	26
6	0	1	51	53
	1	1	79	83
	2	1	0	32
	3	1	81	79
	4	1	154	160
	5	1	139	139
	6	1	82	83
	7	1	79	79
	8	1	76	86
	9	1	115	112
	10	1	87	88
	11	1	28	38
	12	1	75	78
	13	1	0	18
	14	1	24	32
	15	1	0	37
	16	1	31	34
	17	1	0	20
	18	1	0	20
7	0	1	0	9
	1	1	115	113
	2	1	108	114
	3	1	125	129
	4	1	31	34
	5	1	135	139
	6	1	76	83
	7	1	12	23
	8	1	54	61
	9	1	49	49
	10	1	59	54
	11	1	36	40
	12	1	51	49
	13	1	46	47
	14	1	33	32
	17	1	0	13
	18	1	0	8
8	0	1	92	90
	1	1	54	48
	2	1	35	29
	3	1	88	81
	4	1	81	78
	5	1	63	67
	6	1	0	24
	7	1	0	34
	8	1	41	47
	9	1	0	25
	10	1	0	25
	11	1	48	41
	12	1	0	7
	13	1	0	15
	14	1	0	16
9	0	1	0	48
	1	1	0	8
	2	1	0	23
	3	1	0	26
	4	1	0	12
	6	1	52	41
	8	1	22	21
	10	1	0	15
	12	1	0	20
10	1	1	0	24
	3	1	66	49
	5	1	0	12
0	0	2	1136	1071
	1	2	619	597
	2	2	346	327
	3	2	565	539
	4	2	647	616
	5	2	457	433
	6	2	178	171
	7	2	13	10
	8	2	418	423
	9	2	292	277
	10	2	524	502
	11	2	68	66
	12	2	134	143
	13	2	11	30
	14	2	94	86
	15	2	125	119
	16	2	104	117
	17	2	2	12
	18	2	0	35
	19	2	51	51
	20	2	33	28
	21	2	0	16
	23	2	16	3
	24	2	22	14
1	0	2	585	515
	1	2	578	524
	2	2	394	375
	3	2	387	361
	4	2	329	307
	5	2	165	154
	6	2	152	143
	7	2	174	163
	8	2	330	315
	9	2	410	397
	10	2	143	150
	11	2	189	200
	12	2	135	145
	13	2	142	145
	14	2	97	97
	15	2	97	78
	16	2	35	34
	17	2	51	55
	18	2	0	5
	19	2	0	18
	20	2	0	33
	21	2	32	24
	22	2	0	9
2	0	2	48	37
	1	2	208	201
	2	2	243	231
	3	2	153	154
	4	2	72	77
	5	2	23	25
	6	2	343	321
	7	2	225	204
	8	2	235	229
	9	2	132	120
	10	2	138	153
	11	2	81	83
	12	2	122	134
	13	2	0	39
	14	2	42	62
	15	2	37	43
	16	2	68	78
	17	2	56	48
	18	2	61	58
	19	2	25	32
	20	2	21	21
	21	2	34	33
	22	2	0	16
3	0	2	627	642
	1	2	81	71
	2	2	214	212
	3	2	281	251
	4	2	297	302
	5	2	176	170
	6	2	57	74
	7	2	49	45
	8	2	182	192
	9	2	134	148
	10	2	42	52
	11	2	59	63
	12	2	0	20
	13	2	76	81
	14	2	21	33
	15	2	96	95
	16	2	56	72
	17	2	28	13
	18	2	0	29
	19	2	0	22
	20	2	0	16
	23	2	12	21
4	0	2	73	68
	1	2	311	321
	2	2	125	134
	3	2	129	145
	4	2	169	173
	5	2	145	151
	6	2	151	156
	7	2	156	163
	8	2	62	68
	9	2	56	58
	10	2	23	61
	11	2	58	53
	12	2	33	51
	13	2	81	86
	14	2	66	66
	15	2	21	24
	16	2	0	41
	17	2	36	28
	18	2	39	43
	19	2	17	8
	20	2	0	14
	21	2	30	20
	22	2	15	7
5	0	2	64	55
	1	2	150	154
	2	2	61	72
	3	2	98	121
	4	2	159	166
	5	2	125	134
	6	2	109	115
	7	2	0	20
	8	2	70	91
	9	2	96	101
	10	2	114	122
	11	2	57	79
	12	2	49	65
	13	2	0	10
	14	2	0	41
	15	2	56	50
	16	2	45	41
	17	2	31	38
	18	2	37	37
	19	2	13	17
	20	2	0	18
	21	2	13	7
6	0	2	0	49
	1	2	98	103
	2	2	67	75
	3	2	75	70
	4	2	40	36
	5	2	86	91
	6	2	116	112
	7	2	80	95
	8	2	122	141
	9	2	81	87
	10	2	29	32
	11	2	72	75
	12	2	0	39
	13	2	46	48
	14	2	0	12
	15	2	0	38
	16	2	0	20
	17	2	7	27
	18	2	26	24
7	0	2	12	12
	1	2	36	52
	2	2	0	33
	3	2	0	21
	4	2	47	52
	5	2	86	90
	6	2	85	97
	7	2	103	94
	8	2	60	70
	9	2	57	78
	10	2	60	69
	11	2	0	29
	12	2	35	49
	13	2	0	10
	14	2	0	30
	17	2	30	28
	18	2	7	11
8	0	2	58	62
	1	2	0	22
	2	2	35	29
	3	2	21	25
	4	2	17	29
	6	2	51	47
	7	2	100	91
	8	2	47	43
	9	2	40	35
	10	2	0	5
	11	2	66	57
	12	2	39	43
	13	2	0	12
	14	2	0	0
9	0	2	0	6
	1	2	18	27
	2	2	44	34
	3	2	0	12
	4	2	38	36
	6	2	0	10
	10	2	51	44
	12	2	17	12
10	1	2	28	25
	3	2	0	15

Table 4 (*cont.*)

H	K	L	FOBS	FCAL
0	1	3	546	536
	2	3	92	92
	3	3	473	472
	4	3	395	408
	5	3	84	84
	6	3	106	111
	7	3	431	404
	8	3	0	12
	9	3	133	126
	10	3	183	170
	11	3	257	235
	12	3	166	160
	13	3	107	96
	14	3	184	174
	15	3	62	57
	16	3	96	84
	17	3	40	40
	18	3	0	17
	19	3	26	33
	21	3	28	8
1	0	3	230	219
	1	3	408	391
	2	3	82	88
	3	3	163	156
	4	3	249	246
	5	3	324	325
	6	3	175	174
	7	3	215	211
	8	3	51	52
	9	3	69	69
	10	3	230	249
	11	3	48	55
	12	3	62	72
	13	3	157	150
	14	3	136	128
	15	3	81	71
	16	3	53	56
	17	3	80	73
	18	3	0	20
	19	3	21	25
	20	3	0	5
	22	3	20	15
2	0	3	102	100
	1	3	164	157
	2	3	43	42
	3	3	150	155
	4	3	155	150
	5	3	133	132
	6	3	37	34
	7	3	138	143
	8	3	199	204
	9	3	47	49
	10	3	124	116
	11	3	84	80
	12	3	121	115
	13	3	138	126
	14	3	87	80
	15	3	91	79
	16	3	31	32
	17	3	64	55
	18	3	0	71
	19	3	21	16
	20	3	27	26
	21	3	34	27
	22	3	30	28
3	0	3	195	202
	1	3	75	65
	2	3	144	149
	3	3	120	113
	4	3	358	365
	5	3	170	178
	6	3	35	42
	7	3	196	194
	8	3	108	95
	9	3	117	114
	10	3	87	90
	11	3	135	121
	12	3	139	138
	13	3	0	7
	14	3	62	58
	15	3	40	63
	16	3	0	14
	17	3	14	11
	18	3	23	29
	19	3	60	50
	20	3	0	17
4	0	3	200	194
	1	3	83	89
	2	3	79	88
	3	3	51	53
	4	3	70	76
	5	3	262	281
	6	3	85	91
	7	3	53	59
	8	3	86	89
	9	3	144	153
	10	3	49	57
	11	3	20	29
	12	3	0	88
	13	3	51	62
	14	3	48	45
	15	3	0	15
	16	3	24	28
	17	3	46	47
	18	3	34	26
	20	3	23	15
	21	3	13	17
5	0	3	95	102
	1	3	112	124
	2	3	103	105
	3	3	129	129
	4	3	105	103
	5	3	40	44
	6	3	130	135
	7	3	108	107
	8	3	103	110
	9	3	47	68
	10	3	0	14
	11	3	43	36
	12	3	36	44
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	19	3	0	6
	20	3	31	26
6	0	3	98	100
	1	3	61	83
	2	3	37	56
	3	3	69	73
	4	3	88	96
	5	3	36	40
	6	3	43	49
	7	3	54	65
	8	3	49	55
	9	3	43	50
	10	3	0	30
	11	3	41	36
	12	3	56	58
	13	3	0	16
	14	3	32	23
	15	3	35	30
	16	3	36	23
	17	3	31	29
7	0	3	55	56
	1	3	38	33
	2	3	40	65
	3	3	58	59
	4	3	44	48
	5	3	88	88
	6	3	41	43
	7	3	0	0
	8	3	20	23
	9	3	72	79
	10	3	28	32
	11	3	0	27
	13	3	52	43
	14	3	41	32
8	0	3	0	15
	1	3	0	8
	2	3	32	36
	3	3	58	52
	4	3	0	6
	6	3	0	39
	7	3	34	39
	8	3	0	19
	9	3	41	35
	10	3	27	31
	12	3	40	26
	13	3	37	33
	14	3	13	2
9	0	3	47	40
	1	3	0	12
	3	3	7	21
	4	3	0	13
	6	3	30	33
10	3	3	12	18
	5	3	15	13
0	0	4	310	301
	1	4	123	120
	2	4	125	133
	3	4	90	83
	4	4	39	46
	5	4	71	68
	6	4	95	84
	7	4	41	41
	8	4	249	246
	9	4	129	136
	10	4	22	25
	11	4	116	115
	12	4	136	132
	13	4	26	19
	14	4	80	89
	15	4	89	94
	16	4	0	9
	17	4	11	2
	18	4	49	54
	19	4	27	33
1	0	4	231	229
	1	4	212	231
	2	4	0	173
	3	4	0	294
	4	4	132	133
	5	4	159	164
	6	4	42	46
	7	4	23	23
	8	4	23	36
	9	4	0	88
	10	4	63	67
	11	4	114	114
	12	4	105	104
	13	4	81	89
	14	4	105	114
	15	4	0	21
	16	4	0	20
	17	4	28	52
	18	4	47	51
	19	4	28	28
	20	4	0	19
2	0	4	21	7
	1	4	278	268
	2	4	80	79
	3	4	278	267
	4	4	168	168
	5	4	78	75
	6	4	136	130
	7	4	59	62
	8	4	0	29
	9	4	146	140
	10	4	115	111
	11	4	87	94
	12	4	47	47
	13	4	14	35
	14	4	63	65
	15	4	0	24
	16	4	0	18
	17	4	57	64
	18	4	55	49
	20	4	32	35
	21	4	25	24
3	0	4	193	182
	1	4	106	104
	2	4	0	13
	3	4	218	213
	4	4	78	74
	5	4	64	74
	6	4	211	217
	7	4	82	85
	8	4	230	221
	9	4	107	84
	10	4	121	116
	11	4	131	120
	12	4	0	24
	13	4	88	80
	14	4	0	2
	15	4	54	51
	16	4	41	45
	18	4	40	41
	20	4	41	30
4	0	4	141	132
	1	4	95	84
	2	4	78	77
	3	4	55	51
	4	4	65	79
	5	4	109	117
	6	4	78	99
	7	4	38	38
	8	4	180	174
	9	4	164	177
	10	4	65	59
	11	4	31	22
	12	4	51	54
	13	4	73	65
	14	4	0	43
	15	4	0	15
	16	4	29	22
	17	4	34	38
5	0	4	70	57
	1	4	88	87
	2	4	144	136
	3	4	120	105
	4	4	142	131
	5	4	76	72
	6	4	50	52
	7	4	58	55
	8	4	30	64
	9	4	45	42
	10	4	0	21
	11	4	38	38
	12	4	39	40
	13	4	37	33
	14	4	34	30
	15	4	0	11
	16	4	0	15
	17	4	29	33
	19	4	34	26
6	0	4	102	102
	1	4	61	62
	2	4	86	73
	3	4	30	34
	4	4	105	110
	5	4	62	66
	6	4	26	31
	8	4	43	37
	9	4	39	36
	10	4	0	29
	12	4	21	23
	13	4	0	8
	15	4	41	27
	16	4	46	36
7	1	4	0	69
	2	4	0	79
	3	4	0	40
	5	4	0	73
	6	4	0	5
	7	4	0	60
	8	4	0	24
	9	4	0	35
	10	4	0	15
	11	4	20	20
8	0	4	0	21
	1	4	52	42
	2	4	62	58
	3	4	41	45
	4	4	21	29
	6	4	0	0
	7	4	0	9
	8	4	26	14
	9	4	14	19
	10	4	13	18
	12	4	29	23
9	1	4	34	28
	4	4	31	27
0	1	5	197	192
	2	5	269	259
	3	5	92	108
	4	5	209	213
	5	5	191	185
	6	5	94	97
	7	5	75	73
	8	5	60	70
	9	5	49	54
	10	5	74	73
	11	5	26	41
	12	5	36	38
	13	5	44	43
	14	5	44	47
	16	5	43	44
	17	5	0	7
	19	5	30	31
1	0	5	81	71
	1	5	63	59
	2	5	69	72
	3	5	151	137
	4	5	117	121
	5	5	16	18
	6	5	24	39
	7	5	134	148
	8	5	88	84
	9	5	0	22
	10	5	77	77
	11	5	56	62
	12	5	0	20
	13	5	0	16
	14	5	55	53
	15	5	12	21
	16	5	0	37
	17	5	33	36
	18	5	29	31
	20	5	14	23
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	2	5	77	77
	3	5	81	88
	4	5	48	64
	5	5	159	159
	6	5	115	104
	7	5	145	134
	8	5	46	48
	9	5	37	34
	10	5	24	29
	11	5	0	12
	12	5	0	9
	13	5	81	79
	15	5	56	55
	16	5	28	35
	17	5	13	11
	18	5	0	15
	20	5	24	16
3	0	5	17	25
	1	5	158	147
	2	5	145	143
	3	5	130	143
	4	5	193	199
	5	5	67	56
	6	5	91	99
	7	5	62	66
	8	5	48	33
	9	5	58	62
	10	5	27	45
	12	5	103	93
	13	5	31	30
	14	5	64	66
	15	5	27	26
	16	5	53	45
	18	5	19	14
4	0	5	57	54
	1	5	116	121
	2	5	152	141
	3	5	43	39
	4	5	126	131
	5	5	172	172
	6	5	80	74
	7	5	76	78
	8	5	46	49
	9	5	47	47
	10	5	53	52
	11	5	56	59
	12	5	28	30
	13	5	61	64
	14	5	14	15
	15	5	0	21
	16	5	21	27
	17	5	26	24
5	0	5	95	94
	1	5	76	73
	2	5	55	64
	3	5	68	55
	4	5	83	77
	5	5	65	65
	6	5	56	55
	7	5	34	38
	8	5	61	61
	9	5	17	14
	10	5	0	26
	11	5	22	23
	13	5	0	8
	15	5	21	36
	16	5	34	24
6	0	5	72	70
	1	5	56	55
	2	5	66	70
	3	5	33	41
	4	5	0	23
	6	5	70	72
	8	5	51	46
	9	5	0	23
	10	5	0	4
	12	5	25	24
	13	5	24	27
7	1	5	37	40
	2	5	30	40
	3	5	68	66
	5	5	0	26
	6	5	25	29
	7	5	0	26
	8	5	12	9
	9	5	33	32
	10	5	21	22
	11	5	30	27
8	0	5	58	55
	2	5	35	34
	4	5	0	16
	6	5	42	36
	7	5	24	33
	8	5	19	22
	9	5	6	7
0	0	6	7	18
	1	6	58	69
	2	6	40	37
	3	6	185	173
	4	6	31	40
	5	6	81	85
	6	6	14	19
	7	6	119	114
	8	6	117	122
	9	6	52	53
	10	6	34	27
	13	6	14	26
	14	6	0	0
	17	6	26	17
1	0	6	134	114
	1	6	103	100
	2	6	67	59
	3	6	46	46
	4	6	14	36
	5	6	20	28
	6	6	91	93
	7	6	29	37
	8	6	46	38
	9	6	0	34
	10	6	70	84
	11	6	43	47
	12	6	55	47
	13	6	42	48
	14	6	27	28
	15	6	42	46
	16	6	7	11
	17	6	0	8
	18	6	35	30
2	0	6	14	18
	1	6	134	127
	2	6	127	129
	3	6	198	192
	4	6	90	92
	5	6	98	93
	6	6	0	40
	7	6	54	50
	8	6	38	46
	9	6	82	92
	11	6	48	49
	12	6	0	8
	13	6	0	4
	15	6	41	37
	17	6	48	47
	18	6	30	22
3	0	6	264	230
	1	6	63	62
	2	6	88	83
	3	6	0	15
	4	6	88	86
	5	6	99	101
	6	6	95	90
	7	6	36	43
	8	6	104	104
	9	6	15	19
	10	6	46	45
	12	6	57	55
	14	6	53	51
	18	6	38	36
4	0	6	27	25
	1	6	120	117
	2	6	179	166
	3	6	90	89
	4	6	82	84
	5	6	37	34
	6	6	0	13
	7	6	63	60
	8	6	0	19
	9	6	72	74
	10	6	40	41
	11	6	54	54
	12	6	19	25
	15	6	19	15
	16	6	14	19
	17	6	19	22
5	1	6	25	31
	2	6	0	25
	3	6	0	10
	4	6	38	41
	5	6	0	18
	6	6	15	33
	7	6	0	17
	8	6	14	13
	9	6	0	9
	10	6	13	16
	11	6	33	26
	13	6	26	17
6	0	6	0	5
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	2	6	0	23
	3	6	0	7
	5	6	38	42
	6	6	15	24
	7	6	51	49
	10	6	23	13
8	2	6	13	16
	4	6	44	35
0	1	7	110	112
	3	7	77	81
	5	7	82	79
	6	7	0	39
	7	7	0	21
	13	7	17	13
	14	7	17	6
1	0	7	22	29
	1	7	51	46
	2	7	93	100
	3	7	43	45
	4	7	56	62
	5	7	36	49
	6	7	0	40
	8	7	24	35
	9	7	30	33
	10	7	47	44
	11	7	0	14
	14	7	38	40
	17	7	13	18
2	0	7	91	88
	1	7	114	104
	2	7	34	41
	3	7	47	44
	4	7	29	30
	5	7	29	50
	6	7	29	38
	7	7	82	81
	9	7	0	16
	11	7	33	35
	12	7	13	19
	13	7	56	55
3	0	7	37	35
	1	7	46	54
	2	7	31	35
	3	7	17	29
	4	7	159	152
	5	7	15	24
	6	7	12	39
	7	7	27	19
	8	7	76	71
	9	7	43	43
	10	7	55	56
	12	7	38	36
	14	7	24	28
4	0	7	47	50
	1	7	22	14
	2	7	51	54
	3	7	57	70
	5	7	76	67
	6	7	0	67
	7	7	90	84
	8	7	0	12
	9	7	28	27
	10	7	0	12
	11	7	20	23
5	2	7	7	10
	3	7	18	25
	5	7	0	25
	6	7	21	25
	7	7	0	25
	8	7	34	25
	9	7	14	23
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	3	7	24	29
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	2	7	11	16
	3	7	27	30
0	0	8	25	39
	3	8	48	41
	5	8	3	26
	6	8	24	15
	7	8	31	45
	14	8	23	18
1	0	8	62	66
	1	8	31	42
	4	8	32	38
	5	8	32	26
	6	8	64	62
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	3	8	40	51
	5	8	46	47
	9	8	43	45
	11	8	35	36
	12	8	31	28
3	0	8	73	66
	1	8	11	25
	2	8	25	21
	3	8	41	39
	5	8	64	66
	6	8	38	45
	8	8	69	74
	9	8	29	34
	10	8	40	31
	12	8	34	33
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	5	8	17	19
	6	8	28	38
	7	8	36	33
	8	8	28	36
	9	8	42	43
	10	8	27	20
5	2	8	11	12
	3	8	15	16
	5	8	15	17
	6	8	15	11
	7	8	21	19
	8	8	27	21
6	1	8	12	2

C(α, 9) edge. A similar bending of a bond between Si and an aromatic carbon atom is found in the cyclic dimer of tetramethyl-*p*-silphenylenesiloxane (Smith, 1964). In this example, the Si atom comes out of the phenyl plane by about 0·22 Å. As is obvious from the general conformation of the molecules, the deviations of atoms from the α-naphthyl plane of α-SiF are larger than those of α-SiH. It is expected that α-SiCl might exhibit further deviations from the planarity of the naphthyl group, and the amount of such distortions may have some significance in relation to the stability of the compounds. For example, the stability of the molecules against the attack by water decreases from α-SiH to α-SiCl and the Br compound is too unstable to exist out of solution.

Aside from these anomalies in the planarity of the α-naphthyl rings, other features of the molecules are more or less normal. The bond distances and angles in the α-naphthyl group are quite similar to the corresponding values in free naphthalene (Cruickshank, 1957). For comparison, the bond distances in the latter molecule are listed here; they are: C(α, 2)–C(α, 3), 1·421 Å; C(α, 3)–C(α, 4), 1·361 Å; C(α, 4)–C(α, 5), 1·425 Å and C(α, 5)–C(α, 10), 1·410 Å. The phenyl groups are slightly distorted hexagons; for α-SiF the two bonds around C(φ, 4) are shorter than the normal aromatic C–C distance. It is apparent from the angles around C(φ, 1) and C(φ, 4) that these short bonds are not caused by an error in the C(φ, 4) position, but by a decrease in the C(φ, 2)–C(φ, 1)–C(φ, 6) angle from the ideal one of 120°. In contrast to the configuration in α-SiF, there is no such specific shortening of the bonds around C(φ, 4) for α-SiH, although in the latter molecule all the C–C bond distances in the phenyl group are shorter than the usual aromatic bond distance. It might be conjectured that the difference in the polarizability of H and F creates some change in the contribution of the *d* level of the central Si atoms and eventually influences the electronic state of the phenyl groups causing such a difference in the shape of these molecules. The Si atom is also slightly off the plane of the phenyl group by about 0·08 Å.

The Si–F distance of 1·61 Å observed in α-SiF is quite normal as that of a compound with a silicon atom associated with one fluorine atom. Shortening of the Si–F distances according to the number of fluorine atoms

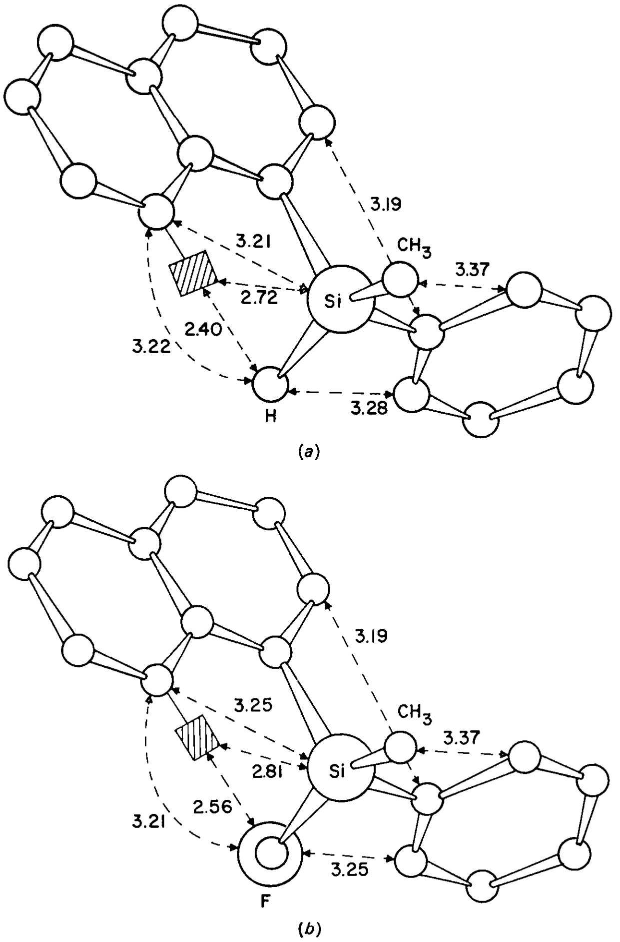

Fig. 2. General conformation of the molecules. Some important intramolecular separations are shown. A shaded square represents the *epi*-hydrogen on C(α, 9).

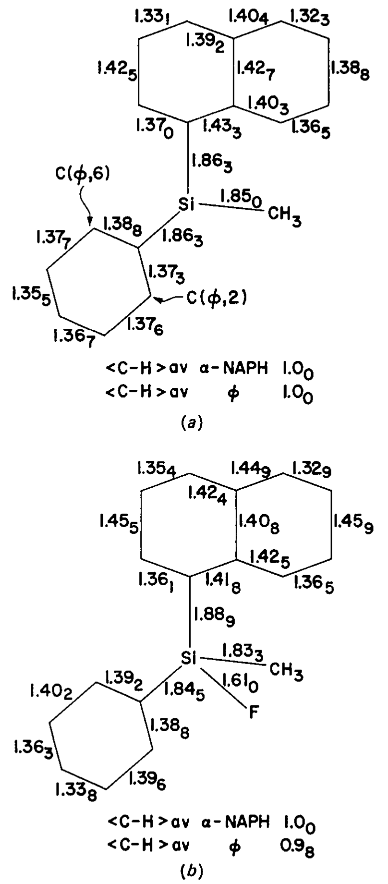

Fig. 3. Bond distances. Standard deviations of these distances are about 0·01–2 Å for C–C, 0·01 Å for Si–C and less than 0·01 Å for Si–F. (*a*) α-SiH, (*b*) α-SiF.

attached to Si is well established (see Pauling, 1960). All the C–H distances in the aromatic systems are around the usual value of 1·0 Å; the methyl groups exhibit rotation or statistical distribution around the Si–C bonds as seen by difference syntheses, and their hydrogen atoms were not included in the lists.

The integrated intensity of (+)-α-SiF was collected by using Cu $K\alpha$ which gives $\Delta f'' \simeq 0{\cdot}3$. The structure was refined by starting from the coordinates representing the molecules of one hand and also from those of the opposite hand. The difference in the two sets of coordinates is a change from the right-hand system to the left-hand system in the crystal structure. One of the two refinements converged with a smaller residual than the other by about 12% (or 1·07 as the ratio in R's); the structure with the smaller residual was therefore chosen to represent the correct hand of the molecule, the absolute configuration.* The absolute configuration thus obtained agrees with that deduced chemically by using Cram's asymmetric induction method (Brook & Limburg, 1963) and also with our previous result (Ashida, Pepinsky & Okaya, 1963). The result is used in interpreting the atomic coordinates in Table 2 for α-SiF, and since it has been established by various methods that α-SiF and α-SiH of the same rotation direction have the same configuration (Sommer, 1962), the same interpretation can be applied to the atomic coordinates of α-SiH in Table 3. In contrast to our previous determination of the absolute configurations in which certain pairs of hkl and $\bar{h}\bar{k}\bar{l}$ reflections were studied, the present study used only the reflections in one octant. This is an example of determination of absolute configuration without the need to collect extra reflections (Ibers & Hamilton, 1964). The absolute configuration of (+)-α-SiX is shown in Fig. 6.

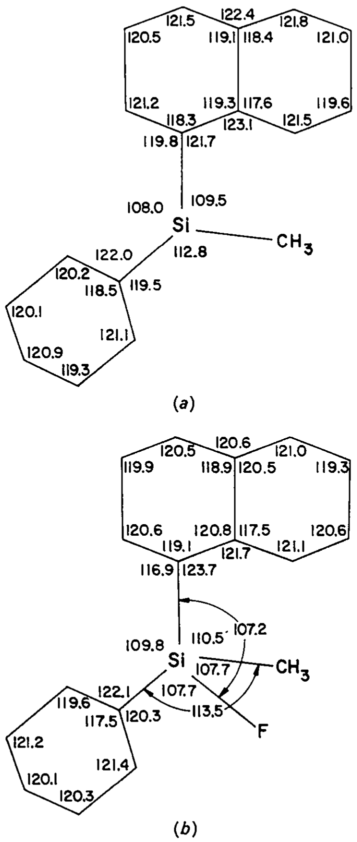

Fig. 4. Bond angles. (*a*) α-SiH, (*b*) α-SiF.

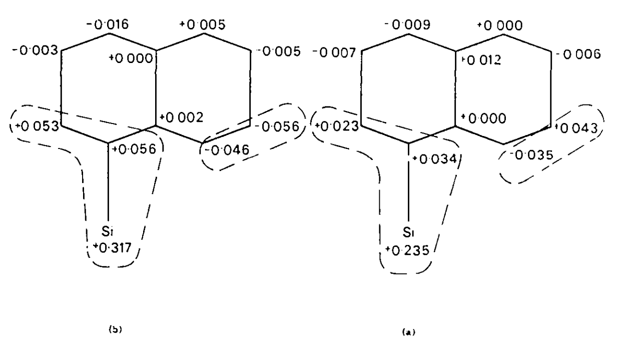

Fig. 5. Deviations of atoms from the α-naphthyl group showing buckling of the plane. (*a*) α-SiH, (*b*) α-SiF.

The crystal structures of these compounds are normal molecular ones; the molecules are packed in the structure by the usual van der Waals force and all intermolecular separations exhibit normal values; also normal is the mode of contact between aromatic rings. A perspective drawing of the structure of (+)-α-SiF is shown in Fig. 7.

The anisotropic thermal parameters listed in Tables 2 and 3 were decoded into the corresponding vibration

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* There is hardly any need to consult the table of Hamilton (1965) in deciding the correctness of the deduction.

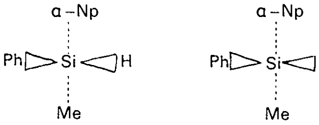

Fig. 6. Absolute configuration of (+)-α-SiX, where X=H and F.

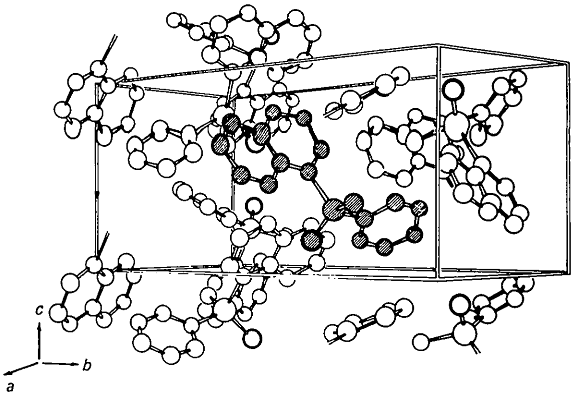

Fig. 7. (+)-α-Naphthylphenylmethylfluorosilane: A view of the structure. Fluorine atoms are shown by double circles.

ellipsoids. Inspection of the result indicates that the carbon atoms which are not involved in the crowding around the central Si atom undergo stronger vibration; they are C(α, 3), C(α, 4), C(α, 6), C(α, 7) and the three outside carbon atoms in the phenyl ring. The directions of the maximum amplitudes are almost in the ring planes. The characteristics of the thermal motion in the two crystals are quite similar and the *F* compound has larger amplitude than the *H* compound; this is reasonable in view of the stability of these two compounds.

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The Crystal Structure of Tetraphenylarsonium 3-Fluoro-1,1,4,5,5-pentacyano-2-azapentadienide

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One of the two carbanions produced by the reaction of sodium or potassium cyanide with 1,2-dichlorohexafluorocyclopentene has been identified by X-ray diffraction as 3-fluoro-1,1,4,5,5-pentacyano-2-azapentadienide. The crystal structure analysis of the tetraphenylarsonium salt was completed without any chemical assumptions.

Tetraphenylarsonium 3-fluoro-1,1,4,5,5-pentacyano-2-azapentadienide forms deep red orthorhombic crystals with $a=9{\cdot}789$, $b=24{\cdot}601$, $c=23{\cdot}918$ Å. The space group is *Pcab* with 8 molecules per unit cell. The structure was refined by least-squares methods and the final residual (*R*) was 5·7% for the 2835 reflections included in the analysis.

In the cation the arsenic atom is bonded to four carbon atoms at the corners of a tetrahedron with an average As–C bond distance of $1{\cdot}897 \pm 0{\cdot}009$ Å. The cation's ideal $\bar{4}$ symmetry is destroyed by rotation of the four planar phenyl groups around the As–C bond. The average C–C bond distance in the four phenyl groups is $1{\cdot}389 \pm 0{\cdot}012$ Å.

The deep red carbanion is nonplanar, probably the result of internal steric effects. The average of the five $C \equiv N$ bonds is $1{\cdot}146 \pm 0{\cdot}012$ Å, in agreement with other reported cyano compounds.

Introduction

The reaction of sodium or potassium cyanide with an excess of 1,2-dichlorohexafluorocyclopentene produces deeply colored solutions. From this reaction mixture, Carpenter (1965) found that two strongly colored carbanions could be isolated as the tetraalkylammonium, -phosphonium or -arsonium salts. The microanalyses indicated that the red carbanion had an approximate empirical formula of $C_{9-11}N_6OF$. The magenta carbanion was formulated as $C_{11}N_6F$. Since the chemical and physical properties did not suggest a structure, an X-ray crystal structure determination appeared essential. The tetraphenylarsonium salt of the red anion (henceforth $TPAS^+$ RD^-) was chosen for the X-ray investigation since it was the most unusual of the two carbanions. The presence of the tetraphenylarsonium cation suggested that the structure of the anion could