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REACTIONS OF HEXAFLUOROBENZENE AND PENTAFLUOROPHENYLAMINO COMPOUNDS WITH AMIDES AND ANILIDES (STERIC EFFECTS)

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SUMMARY

Contrary to N-lithium-N-methylanilide and N-lithium-diphenylamide, trimethyl-N-lithium-N-phenyl-silylamide gave with hexafluorobenzene in molar ratio 2:1, only the monosubstituted compound. Competitive reactions gave the following order of reactivity toward N-lithiumanilide: 2,3,4,5,6-pentafluorotriphenylamine > 2,3,4,5,6-pentafluoro-N-methyl-diphenylamine > hexafluorobenzene >> trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine. A reason for the low reactivity of trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine against aromatic amides has been proposed.

Some new aromatic amines have been synthesized, among them hexamethyl-N-pentafluorophenyl-disilylamine.

INTRODUCTION

Direct nucleophilic disubstitution in hexafluorobenzene with amino compounds has been described by several workers for ammonia [1], methylamine [1,2] dimethylamine [2,3], cyclic amines [4], hydrazine [1,5], N-lithium or sodium anilide [6,7,8], N-lithium-N-methyl-anilide [8], and N-lithium or sodium diphenylamide [6,9].

Disubstituted compounds were obtained with the higher cyclic amines by refluxing the reaction mixture without solvent [4] and for the acyclic amines by running the reactions in sealed tubes at 100-250° [1,2,3]. At lower temperatures the monosubstituted compounds were the only product, or only minor yields of the disubstituted compounds were obtained [4,10,11,12]. Of the anilides pentafluoro-N-lithium-anilide gave with hexafluorobenzene only the monosubstituted compound [7], while N-lithium and sodium anilide gave small yields of the para-disubstituted compound under more severe conditions [6,8]. The orthodisubstituted compound, however, was obtained in good yield between N-lithium anilide and hexafluorobenzene in hexamethyl phosphoric triamide [7]. Both N-lithium-N-methylanilide [8] and N-lithium or sodium diphenylamide readily gave the para-disubstituted compounds [6,9]. With 2,3,4,5,6-pentafluoro-N-lithium-N-methyl or N-ethyl anilide [10,11] and 2,3,4,5,6-pentafluoro-N-lithium or sodium diphenylamide [6,9] a series of oligomers and polymers were formed depending upon the amount of hexafluorobenzene present.

This paper deals with model reactions performed on hexafluorobenzene or its amino compounds aiming at the preparation of poly(imino-perfluoro-1,4-phenylene).

RESULTS AND DISCUSSION

No reaction occured when trimethyl-N-phenyl-silylamine was refluxed with hexafluorobenzene in tetrahydrofuran, but a 38% yield of trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine, I, could be isolated by employing trimethyl-N-lithium-N-phenyl-silylamide and hexafluorobenzene in molar ratio 1:1. The yield of I (isolated after hydrolysis as 2,3,4,5,6-pentafluorodiphenylamine) increased to 85% with the reactants in molar ratio 2:1. No disubstituted compound was obtained. These results were unexpected as a disubstitution was preferred for monosubstitution in hexafluorobenzene with N-lithium-N-methylanilide and N-lithium-diphenylamide already with the reactants in molar ratio 1:1* [8,9

The ^{19}F NMR spectra of the monosubstituted compounds, I, 2,3,4,5,6-pentafluoro-N-methyldiphenylamine, II [8], and 2,3,4,5,6-pentafluorotriphenylamine, III [9], gave approximately the same shifts for the para-fluorine downfield to hexafluorobenzene, 4.0, 3.8, and 3.8 respectively, see Table 1. These results indicate nearly the same electron density around the para-position in the pentafluorophenyl group in I, II, and III [13,14,15].

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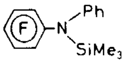
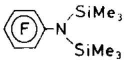
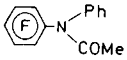
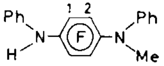
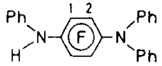
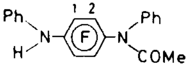
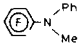
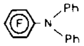
*N-lithium-N-methyl-anilide gave a yield of 40.2% of the monosubstituted a 48.5% of the para-disubstituted compound, and N-lithium-diphenylamide 29.9% and 43.6% respectively. Yields were calculated with respect to consumed anilide and amide.

Table 1.

Spectral data

Compound		^{19}F Chemical shifts in $CDCl_3$ with C_6F_6 as internal standard at 94.1 and 56.45*MHz Shifts at (ppm), down-field to C_6F_6	(Posi-tion)	^1H chemical shifts in $CDCl_3$ with TMS as internal standard at 60 MHz Shifts at NH	(ppm) $-CH_3$	IR (cm^{-1})
I 	a)	15.80	o		0.28	1515(m), 1500(s), 1255(m), 1245(m),
		4.00	p			1014(s), 985(s), 922(m), 837(s).
		-0.95	m			
IV 		15.00	o		0.12	1516(s), 1502(s), 1267(w), 1257(s)
		0.15	p			1029(m), 1010(s), 987(s), 927(s),
		-2.50	m			872(m), 840(m), 822(m).
V 		17.40	o		2.08	1692(s), 1654(m), 1519(s), 1302(s),
		7.05	p			1150(m), 1040(m), 1029(m), 994(s),
		-0.20	m			974(s)
VI 		11.70*	1	5.51	3.27	3390(m), 1500(s), 1093(m), 982(s),
		15.00*	2			962(m)
VII 		11.80*	1	5.54		3390(m), 1512(sh), 1500(s), 1481(w),
		15.15*	2			1082(m), 1037(m), 1016(m), 978(s)
VIII 		11.45*	1	5.82	2.08	3310(s), 1682(s), 1644(m), 1498(s),
		15.35*	2			1325(s), 1050(m), 995(m), 977(s)

a) II  16.9(o), 3.8(p), -0.3(m) [8]

III  17.6(o), 3.8(p), -0.15(m) [9]

The U.V. spectra, Fig 1, may indicate a higher coplanarity for II and III than for I (λ_{max} = 290.5 nm, 282 nm and 236.5 nm respectively). As a result a better stabilization of the carbanion with a quinoid structure by further substitutions may be proposed for II and III than for I. The results from the spectroscopic data, N.M.R. and U.V., therefore seem to indicate a somewhat higher reactivity for II and III than for I, but the N.M.R. data do not indicated any dramatical differences in the reactivity. Competitive reactions of hexafluorobenzene, compounds I, II, and III with N-lithium anilide qualitatively gave the following order of reactivity in tetrahydrofuran: III > II > hexafluorobenzene >> I (see Experimental section).

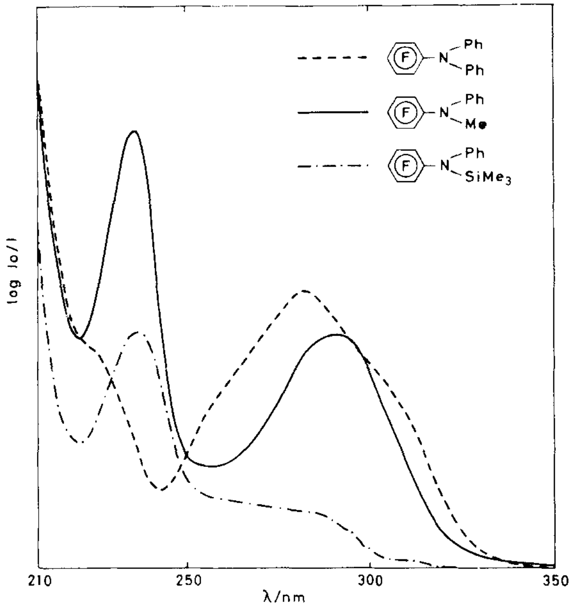


Fig. 1. U.V. spectra run in cyclohexane.

When trimethyl-N-lithium N-phenyl-silylamide was reacted with 2,3,4,5,6-pentafluoro-N-methyldiphenylamine, or N-lithium-N-methyl-anilide with compound I, 2,3,5,6-tetrafluo N-methyl-N,N'-diphenyl-1,4-phenylenediamine, VI, could b isolated after hydrolysis. Th reactions were, however, sluggish compared with reactions N-lithium-N-methylanilide wit 2,3,4,5,6-pentafluoro-N-methyadiphenylamine, which readily gave the disubstituted compound [8]. The low reactivity of I against N-lithiumanilide, N-lithium-N-methylanilide and the unreactivity against trimethyl-N-lithium-N-phenylsilyamide may be caused by steriohinderance and do not seem to result from molecular association between the nucphile and the silicon atom in I [16], nor a reduced possibility for charge delocalization in the transition state of I.

A white compound usualy is formed when the first drops of hexafluorobenze are applied to an addition coloumn containing minor remnants of aniline. The compound is previously described [1] and is believed to be a charge transfer compound

[17, 18]. This may indicate that the nucleophilic reactions described proceed through a weak charge-transfer complex [19], with the nucleophile acting as a n- or π-donor giving a n-π or π-π complex [17, 20, 21, 22]. This might explain why the bulky group in compound I make the compound so little attractive for further reactions with anilides, as it may obstruct the orientation of the electron donor in forming the charge transfer complex, which may for aromatic amines be the first step in the reaction mechanism [20, 23].

The anticipated reaction products from the competitive reactions (see Experimental section) were produced by reacting N-lithium anilide with the compounds I, II, and III. The molecular mass determination of the obtained para-substituted 2,3,5,6-tetrafluoro-N,N,N'-triphenyl-1,4-phenylenediamine, VII, showed a broad peak above the molecular ion. A recheck of the compound led to the conclusion that a relative large amount of a metastable ion with a higher m/e-value than the molecular ion was present in the spectrum. To the best of my knowledge, a direct observation of such a metastable ion in a routine spectrum has hitherto not been reported. The results will, together with results from analysis of different phenylenediamines, be published in a separated paper.

Preparation of trimethyl-N,N-decafluorodiphenyl-silylamine has, been attempted. From a reaction between N-metal-decafluoro-diphenylamine and trimethylchlorosilane no evidence of reaction was observed in tetrahydrofuran. From trimethyl-N-lithium-N-pentafluorophenyl-silylamide and hexafluorobenzene, hexamethyl-N-pentafluorophenyl-disilylamine, IV, was the main compound obtained from a vacuum distillation. This compound was also obtained from trimethyl-N-lithium-N-pentafluorophenyl-silylamide and trimethylchlorosilane, but not from hexamethyl-N-lithium-disilylamide and hexafluorobenzene in tetrahydrofuran. This may be a result of steric hinderance [24] as the N-lithium-disilylamide is a stronger base than N-lithium anilide [25] which reacts readily with hexafluorobenzene. The Stuart-Briegleb model of IV showed hindered rotation around the N-C bond and the model of trimethyl-N,N-decafluorodiphenyl-silylamine showed a very rigid molecule indicating why the reactions above did not proceed as expected.

2,3,4,5,6-Pentafluoro-N-phenylacetanilide, V, was obtained from 2,3,4,5,6-pentafluorodiphenylamine and acetic anhydride [6] and from the N-lithium or N-magnesium iodide compound and acetyl chloride. No reaction occurred between N-lithium-acetanilide and hexafluorobenzene in tetrahydofuran nor between 2,3,4,5,6,-pentafluorodiphenylamine and acetyl chloride in the presence of triethylamine. Further reaction on V with N-lithium anilide gave N-(4-anilino-2,3,5,6-tetrafluorophenylene) acetanilide in low yield.

From the model reactions described above and previously described reactions [7,8] no easy way for preparing poly (iminotetrafluoro-1,4-phenylene) in common solvents has been established.

EXPERIMENTAL

All reactions were carried out under an athmosphere of dry oxygen-free nitrogen. Tetrahydrofuran was dried and distilled from sodium benzophenone ketyl prior to use. Infrared and NMR spectra were determined by employing Perkin-Elmer 457-IR and 100 and 60 MHz NMR instruments, respectively. Mass spectra were recorded on an AEI MS 902 instrument attached to an AEI DS30 data system. General reaction [8] and purification [8] procedures were followed.

Reactions between trimethyl-N-lithium-N-phenyl-silylamide and hexafluorobenzene

Molar ratio 1:1 in THF

To trimethyl-N-phenyl-silylamine (8.3 g, 0.05 mol) in THF (30 ml) at -75°, n-Buli (0.05 mol in hexane) was added. A heavy white precipitate disappeared when the mixture was warmed to room temperature. To the cooled mixture (-75°) hexafluorobenzene (9.3 g, 0.05 mol) was added. This gave a red liquid. The mixture was stirred at ambient temperature over night, and ordinary work up of the unhydrolyzed mixture gave 6.30 g (38.1%) trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine, (n.c.), m.p. 58-60°. Found: C, 54.56 H, 4.24, m/e, 332 (15) 331 (52), 220 (31.5), 77 (51), 74 (16), 73 (100). Calc. for $C_{15}H_{14}F_5NS$: C, 54.37, H, 4.26, M, 331.3.

Molar ratio 2:1 in THF/triethyleneglycol dimethyl ether

To trimethyl-N-phenyl-silylamine (9.9 g, 0.06 mol) in THF (15 ml) triethyleneglycol dimethyl ether (10 ml), and lithium amide (1.5 g) were added. The mixture was placed on a 70° bath, in which the temperature was raised to 90° in 50 min. The mixture was then cooled to -10°. On addition of hexafluorobenzene (5.58 g, 0.03 mol) a reaction giving a viscous oil took place. The -10° bath was removed and more THF (10 ml) was added. The temperature was elevated to 90° in 1 h and maintained for 2 h. Work up of the hydrolyzed mixture afforded 6.62 g (85.3%) 2,3,4,5,6-pentafluoro-diphenylamine, m.p. 70-72° which was identical with an authentic sample [8]. Thin layer chromatography of the crude did not show any traces of the disubstituted compound, 2,3,5,6-tetrafluoro-N,N'-diphenyl-1,4-phenylenediamine [8].

2,3,5,6-Tetrafluoro-N,N,N' triphenyl-1,4-phenylenediamine

2,3,4,5,6-Pentafluoro-triphenylamine (1.68 g, 0.005 mol) was added to a reaction mixture from aniline (0.47 g, 0.005 mol) and lithium amide (0.30 g) in THF (10 ml) at 0°. The mixture was stirred over night at ambient temperature and then at 60° for 2 h. The hydrolyzed material gave after sublimation 1.91 g (93.5%) 2,3,5,6-tetrafluoro-N,N,N'-triphenyl-1,4-phenylenediamine (n.c.) m.p. 143-144°. Found: C, 70.42, H, 4.13, m/e, 409 (33), 408 (100), 388 (6.5), 368 (6), 316 (4.5), 311 (4) 291 (4), 204 (8.8), 77 (25), 51 (12). Calc. for $C_{24}H_{16}F_4N_2$: C, 70.58, H, 3.95, M, 408.4.

2,3,5,6-Tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine

(1) 2,3,4,5,6-Pentafluoro-N-methyl-diphenylamine (2.73 g, 0.01 mol) in THF (10 ml) was added to a reaction mixture of aniline (0.93 g, 0.01 mol) and lithium amide (0.52 g) in THF (10 ml) at 0°, and stirred over night at ambient temperature. Sublimation of the hydrolyzed mixture gave 2.24 g (64.7%) 2,3,5,6-tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine, (n.c.), m.p. 57-61°. Found C, 65.61, H, 4.24, m/e, 347 (22), 346 (100), 331 (9), 326 (13), 311 (5), 306 (6), 305 (6.5), 291 (10), 254 (7), 349 (4), 240 (2), 173 (7), 77 (34), 51 (16). Calc. for $C_{19}H_{14}F_4N_2$: C, 65.90, H, 4.08, M, 346.3.

(2) To trimethyl-N-lithium-N-phenyl-silylamide (0.02 mol) prepared as previously described with n-BuLi in THF (30 ml), 2,3,4,5,6-pentafluoro-N-methyl-diphenylamine (5.46 g, 0.02 mol) was added. After 1 h at ambient temperature. thin layer chromatography of an unhydrolyzed sample with light petroleum (40-60°) showed traces of a new compound. The mixture was refluxed for 2 h, stirred over night and worked up. This gave 1.47 g recovered 2,3,4,5,6-pentafluoro-N-methyl-diphenylamine and 4.06 g (58.6%) 2,3,5,6-tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine, m.p. 57-61°.

(3) To N-lithium-N-methylanilide, prepared from N-methylaniline (0.75 g, 0.007 mol) and lithium amide (0.20 g) in THF (10 ml), trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine (2.32 g, 0.007 mol) was added. The mixture was refluxed for 2 h and stirred over night at ambient temperature. The hydrolyzed crude, which was analyzed by G.L.C., contained from the peak area 26% 2,3,4,5,6-pentafluorodiphenylamine and 65% 2,3,5,6-tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine.

Competitive reactions

(1) To N-lithiumanilide, from aniline (0.65 g, 0.007 mol) and lithium amide (0.17 g) [22] in THF (5 ml) at -75°, hexafluorobenzene (1.8583 g, 0.01 mol) 2,3,4,5,6-pentafluoro-N-methyldiphenylamine (2.7332 g, 0.01 mol), and N,N-dimethylaniline (1.2116 g, 0.01 mol), as the internal standard, in THF (15 ml), were added after withdrawal of a sample. The mixture was stirred at 20° over night and hydrolyzed with 5% HCl to acidic reaction, then extracted with ether, to which N,N-dimethylaniline (1.2118 g, 0.01 mol) was added. The mixtures was analyzed by G.L.C. on a 8 feet SE-30 glass column. Differences in amount of consumed starting material and in amount of new compounds, 2,3,4,5,6-pentafluoro-diphenylamine [8] and 2,3,5,6-tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine, showed the highest reactivity for 2,3,4,5-pentafluoro-N-methyl-diphenylamine.

(2) A reaction was performed with N-lithiumanilide as in 1. and with 2,3,4,5,6-pentafluoro-N-methyl-diphenylamine (2.7310 g, 0.01 mol) and trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine (3.3136 g, 0.01 mol) as the reactants and biphenyl (1.5419 g, 0.01 mol) as the internal standard. G.L.C. was performed on the starting mixture without N-lithiumanilide, and on the unhydrolyzed and the hydrolyzed reaction mixture. Analyses of the unhydrolyzed mixture showed the presence of 2,3,4,5,6-pentafluorodiphenylamine together with the silylamine. Three new compounds were present in the reaction mixture in the ratio 5.5 : 4 : 1 from the peak areas. The first was 2,3,5,6-tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine. G.L.C. -M.S. gave for the two others: m/e 512 (100) and m/e 498 (100). Both were also present in the reaction mixture from 1. in low concentrations. Only traces of 2,3,5,6-tetrafluoro-N,N'-diphenyl-1,4-phenylenediamine [8] (which was the main product in the hydrolyzed mixture from a reaction between trimethyl-2,3,4,5,6-pentafluoro-N,N-diphenyl-silylamine and N-lithium anilide) could be detected.

(3) The reaction was performed as in 1. with 2,3,4,5,6-pentafluoro-N-methyldiphenylamine (2.7321 g, 0.01 mol) and 2,3,4,5,6-pentafluoro-triphenylamine (2.9923 g, 0.01 mol). Comparison of G.L.C. diagrams for the starting mixture with biphenyl (1.5421 g, 0.01 mol) as the internal standard, and the hydrolyzed reaction mixture, showed some higher consumption of triphenylamine than of the diphenylamine. This was also reflected in the amounts of the substitution product 2,3,5,6-tetrafluoro-N-methyl-N,N'-diphenyl-1,4-phenylenediamine and 2,3,5,6-tetrafluoro-N,N,N'-triphenyl-1,4-phenylenediamine.

2,3,4,5,6-pentafluoro-N-phenylacetanilide

(1) To 2,3,4,5,6-pentafluoro-N-lithium-diphenylamide, prepared from 2,3,4,5,6-pentafluoro-diphenylamine (5.18 g, 0.02 mol) and lithium amide (0.6 g) under a flow of purified N_2 in THF (20 ml) more THF (15 ml) and acetyl chloride (3.3 ml) were added at 0°. The mixture was stirred over night at ambient temperature, hydrolyzed with water and then with 2% NaOH to neutral reaction on a pH paper. Work up gave a light brown oil, 5.0 g containing two main compounds in approximately equal amounts. By chromatography, sublimation and recrystallization from light petroleum (40-60°) pure samples of recovered 2,3,4,5,6-pentafluoro-diphenylamine (1.3 g) and 2,3,4,5,6-pentafluoro-N-phenylacetanilide (1.55 g) m.p. 93.5 - 95.5° (lit. [6] m.p. 91-93°) were obtained. Found: C, 56.09, H, 3.10, m/e, 301 (5), 260 (13), 259 (100). Calc. for $C_{14}H_8F_5NO$: C, 55.82, H, 2.68, M, 301.2.

(2) To 2,3,4,5,6-pentafluoro-diphenylamine (10.4 g, 0.04 mol) in ether (20 ml) at 0° methylmagnesium iodide (0.04 mol) was added. At negative Gilman Color Test I [27] acetyl chloride (3.44 ml, 0.05 mol) was added. The mixture was stirred over night at ambient temperature and worked up to give a light brown oil, 9.80 g, which, as determined by G.L.C., contained 6.5 g (54%) 2,3,4,5,6-pentafluoro-N-phenylacetanilide, half of which crystallized from the oil on the addition of light petroleum and a crystal from the previous reaction.

N-(4-anilino-2,3,5,6-tetrafluorophenylene)acetanilide

(1) N-(pentafluorophenyl)acetanilide (3.01 g, 0.01 mol) in THF (10 ml) was added in 5 min at 0° to N-lithium anilide, prepared from aniline (0.95 g, 0.01 mol) and lithium amide (0.5 g) in THF (15 ml). The mixture was stirred at ambient temperature for 1 h and worked up to a black solid mixture. Thin layer chromatography showed three compounds present in the crude product. The mixture was treated with light petroleum (40-60°) from which 0.1 g 2,3,4,5,6-pentafluoro-diphenylamine was obtained. The rest of the crude was sublimed. This gave 0.3 g acetanilide, m.p. 115-117°, identified by M.S. and I.R., and 0.92 g (24.6%) N-(4-anilino-2,3,5,6-tetrafluorophenylene)acetanilide, (n.c.), m.p. 154-155.5°. Found: C, 64.02, H, 3.75, m/e, 374 (22), 333 (21.5), 332 (100), 331 (7), 293 (11), 77 (17.5), 43 (28.5). Calc. for $C_{20}H_{14}F_4N_2O$: C, 64.17, H, 3.77, M, 374.3.

(2) The reaction was performed as above, but the N-lithium anilide was added to the N-(pentafluorophenyl)acetanilide dissolved in THF (40 ml). The work up gave 0.2 g 2,3,4,5,6-pentafluoro-diphenylamine and 1.18 g (31.5%) N-(4-anilino-2,3,5,6-tetrafluorophenylene)acetanilide, m.p. 153.5-155.5°.

Trimethyl-N-pentafluorophenyl-silylamine

Pentafluoroaniline (14.65 g, 0.08 mol) in THF (20 ml) was added slowly to lithium amide (1.9 g) in THF (5 ml) on a 20° water bath. The mixture was stirred at 60° for 1 h under a stream of nitrogen and cooled, whereupon trimethylchlorosilane (10 ml) was added all at once and still more (2 ml) after 5 min. The mixture was stirred at ambient temperature over night, then at 60° for 1 h and distilled under vacuum to give 15.9 g of a crude material which contained pentafluoroaniline (15,9%) and trimethyl-N-pentafluorophenyl-silylamine (84.1%). An analytical sample of the silylamine was obtained by preparative C.L.C. NMR and IR were in agreement with reported data [28].

Hexamethyl-N-pentafluorophenyl-disilylamine

To trimethyl-N-pentafluorophenyl-silylamine, prepared as above from 0.05 mol of starting materials, but not distilled, lithium amide (1.3 g) was added and the mixture stirred at 70° for 2 h under a stream of nitrogen. To the cooled mixture trimethylchlorosilane (10 ml) was added all at once, whereupon the mixture was stirred at ambient temperature over night. Vaccum distillation gave 12.95 g of a crude product containing trimethyl-N-pentafluorophenyl-silylamine (44.7%) and hexamethyl-pentafluorophenyl-disilylamine, (n.c.), (55.3%), m.p. 25°. Found: C, 43.72, H. 5.74, m/e, 328(6), 327(25), 314(4), 313(11), 312(46), 221(5), 216(4), 120(6), 113(5), 81(20, 77(56), 73(100).
Calc. for $C_{12}H_{18}F_5NSi_2$: C. 44.02, 5.53, M, 327.4.

Analytical samples were obtained by preparative G.L.C.

Attempt to prepare trimethyl-N,N-decafluorodiphenyl-silylamine

Trimethyl-N-pentafluorophenyl-silylamine (6.4 g containing approximately 8% pentafluoroaniline) in THF (20 ml) was added slowly to lithium amide (0.7 g) in THF (5 ml) and stirred at 65° for 30 min, then cooled to 0°, whereupon hexafluorobenzene (4.65 g, 0.025 mol) was added slowly. The mixture was stirred over night at ambient temperature and then refluxed for 2 h. The solvent was distilled off and the remaining oil vacuum distilled. This gave trimethyl-N-pentafluorophenyl-silylamine and hexamethyl-N-pentafluorophenyl-disilylamine as the main compounds and some decafluorodiphenylamine.

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