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Model reactions for preparing poly(imino-tetrafluoro-1,4-phenylene)

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Abstract

2,3,4,5,6-Pentafluoroformanilide was prepared giving, in addition, two new compounds 4,5,6,7-tetrafluoro-1-pentafluorophenyl-benzimidazole and 2,3,4,5-tetrafluoro-6-[(pentafluorophenyl)amino]formanilide. Sodium 2,3,4,5,6-pentafluoro-formanilide was reacted with hexafluorobenzene in a molar ratio of 1:4 to give oligomers of α-pentafluorophenyl-ω-fluoro-poly(imino-tetrafluoro-1,4-phenylene). Some of the oligomers were isolated. The results indicate that poly(imino-tetrafluoro-1,4-phenylene) could be formed. Model reaction on hexafluorobenzene with sodium acetanilide, molar ratio 1:2, gave a low yield of *N*,*N*′-diacetyl-diphenyl-tetrafluoro-1,4-phenylenediamine.

Keywords: Synthesis; 2,3,4,5,6-Pentafluoroformanilide; Oligomers of poly(imino-tetrafluoro-1,4-phenylene)

1. Introduction

Poly(imino-1,4-phenylene) has attracted growing attention as a material used in electrochemical devices and for battery electrodes [1–3]. Reacting hexafluorobenzene with sodium *N*-methylformamide or sodium formanilide gave 1,4-disubstituted compounds in good yields [4,5] indicating that poly(imino-tetrafluoro-1,4-phenylene) ought to be formed by reacting hexafluorobenzene with sodium 2,3,4,5,6-pentafluoroformanilide.

2. Results and discussion

2.1. Preparation of 2,3,4,5,6-pentafluoroformanilide

2,3,4,5,6-Pentafluoroformanilide (**1**) was prepared from pentafluoroaniline and lithium amide in dimethylformamide as previously described (Scheme 1) [6]. The amount of starting materials was increased compared to the previous report, resulting in the isolation of two new compounds, 4,5,6,7-tetrafluoro-1-pentafluorophenyl-benzimidazole (**2**) and 2,3,4,5-tetrafluoro-6-[(pentafluorophenyl)amino]formanilide (**3**). The yield of **3** was better than that of 2,3,5,6-tetrafluoro-4-[(pentafluorophenyl)amino]formanilide (**4**), the only compound isolated together with **1** from the previously described reaction [6] (Scheme 1). **3** and **4** seemed to be formed from a nucleophilic attack by lithium pentafluoroanilide on pentafluoroformanilide. The lithium pentafluoroformanilide will not form a nucleophile strong enough to react with itself. The sodium pentafluoroformanilide did not react with hexafluorobenzene in tetrahydrofuran until dimethylformamide was added (Scheme 3). In contrast, lithium pentafluoroanilide reacted readily with hexafluorobenzene in tetrahydrofuran indicating that lithium pentafluoroanilide is a stronger nucleophilic reagent than lithium pentafluoroformanilide or sodium pentafluoroformanilide [7].

It has previously been shown that some solvents strongly influence the direction of substitution in some pentafluorophenyl compounds [8,9].

Dimethylformamide, as a good dipolar aprotic solvent, may have interfered with the reagents giving an intermediate complex, Scheme 2. Compound 3 has probably been formed through that complex.

Only traces of **2** were isolated. This compound is believed to have been formed by a cyclization of **3**. 1,2,3,4,6,7,8,9-Octafluoro-5-formyl-10-hydrophenazine was not formed from the lithium compound of **3**, believed to be present during the reaction. An attempt at chemical cyclization of 2-aminononafluorodiphenylamine, using sodium hydride and *n*-butyl lithium to give 1,2,3,4,6,7,8,9-octafluoro-5,10-dihydrophenazine, was unsuccessful [10]. Nevertheless, 4,5,6,7-tetrafluoroimidazole was formed by reacting 3,4,5,6-tetrafluoro-1,2-phenylenediamine with formic acid or formaldehyde and hydrochloric acid

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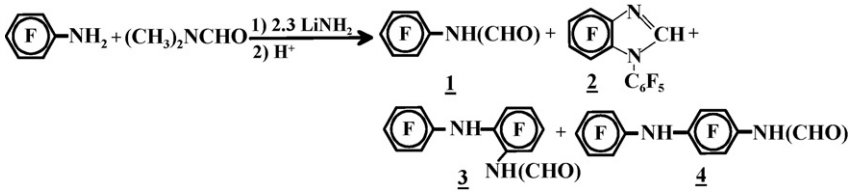
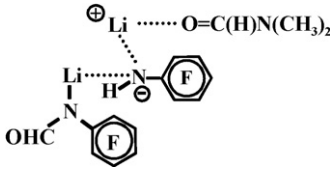


Scheme 1.



Scheme 2.

[11]. The results described in this paper seem to be in agreement with those of Professor Tatlow's group [10,11].

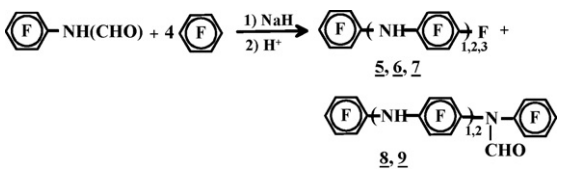
2-Amininonafluorodiphenylamine was not obtained from the hydrolyzed reaction mixture. This indicates that the formyl compound is relatively stable. Attempts to hydrolyze **4** in a mixture of ethanol and sodium hydroxide were unsuccessful.

2.2. Preparation of the oligomers

No reaction seemed to take place between sodium pentafluoroformanilide and hexafluorobenzene in tetrahydrofuran. Dimethylformamide was therefore added to the reaction mixture. The addition of sodium pentafluoroformanilide to hexafluorobenzene seemed to be the rate dependent reaction. When *N*,*N*-decafluorodiphenylformamide was formed, the further substitution reactions seemed to run smoothly. To obtain as large a proportion of oligomers as possible for separation, a four-fold amount of hexafluorobenzene was used to the reaction (Scheme 3).

In an attempt to prevent hydrolysis of the compounds formed, the reaction mixture was cooled to 0 °C and a 20% (v/w) hydrogen chloride solution was added as quickly as possible. It has previously been shown that *N*,*N*-decafluorodiphenylformamide is stable in an acidic solution [12]. When hexafluorobenzene was reacted with the sodium *N*-methylformamide or sodium formanilide, the *N*,*N*′-disubstituted compounds could be obtained along with hydrolyzed and partially hydrolyzed compounds after hydrolysis of the reaction mixture [4,5]. From the hydrolyzed reaction mixture as pure compounds, only decafluorodiphenylamine (**5**) and *N*,*N*′-decafluorodiphenyl-tetrafluoro-1,4-phenylenediamine (**6**) could be isolated together with traces of α-pentafluorophenyl-ω-fluoro-tri(imino-tetrafluoro-1,4-phenylene) (**7**), *N*-formyl-*N*,*N*′-decafluorodiphenyl-tetrafluoro-1,4-phenylenediamine (**8**), and *N*-formyl-4,4′-bis [(pentafluorophenyl)amino]-2,2′,3,3′,5,5′,6,6′-octafluorodiphenylamine (**9**) (Scheme 3). Hydrolyzed or partially hydrolyzed compounds seem to have been formed during the quenching of the reaction mixture. Thin layer chromatography of the rest from the reaction mixture contained **7** and a series of other compounds believed to belong to the same series of compounds as isolated. No 2,3,4,5,6-pentafluoroformanilide was recovered from the reaction mixture.

The results indicate that a poly(imino-tetrafluoro-1,4-phenylene) could be formed.



Scheme 3.

2.3. Hydrolysis of separated formyl compounds

Hydrolysis of **8** and **9** gave the linear compounds **6** and **7** (Scheme 3).

Dissolving a sample of **6** relatively quickly resulted in a strong reddish color of the solution. The discoloration of **6** in solid, dried form was slow. **7** gave a bluish tint to the red. Purification of a colored, dissolved sample of **6** on a silicate column gave almost no red material, which is believed to be an oxidation product.

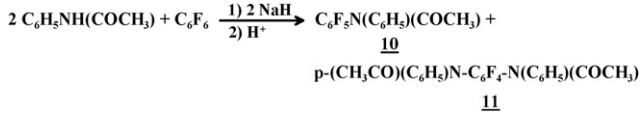
2.4. Reaction of hexafluorobenzene with acetanilide

A reaction between hexafluorobenzene and sodium acetanilide, molar ratio 1:2, resulted in 2.8% 2,3,4,5,6-pentafluoro-*N*-phenylacetanilide (**10**) and 14.4% *N*,*N*′-diacetyl-*N*,*N*′-diphenyl-tetrafluoro-1,4-phenylenediamine (**11**) (Scheme 4). No reaction occurred between lithium acetanilide and hexafluorobenzene in tetrahydrofuran [13].

The use of sodium 2,3,4,5,6-pentafluoro-acetanilide to obtain poly(imino-tetrafluoro-1,4-phenylene) after hydrolysis, therefore seems little fruitful.

2.5. Spectroscopic results

Mass spectra of the formanilides concurred with the normal pattern [$M^+ - CO$]. **3** gave a strong peak at *m*/*z* 355.9989



Scheme 4.

indicating the presence of **2**[+] (*m/z* calc.: 355.9996) and the strongest peak at *m/z* 326.0093, which is believed to indicate the presence of 5,10-dihydrooctafluorophenazine [+] (*m/z* calc.: 326.0085) (obtained by electrolysis of 2-aminononafluorodiphenylamine [10]) in the mass spectrum.

^{1}H NMR of pentafluoroformanilide did not show any presence of *cis* and *trans* isomers in acetone-d_6 [6,14]. This has been attributed to a very strong hydrogen bonding between the acetone-d_6 and the solute [6]. The same situation is possible with the new formyl compounds. The fluorine shifts of the formyl compounds showed two isomers indicative of the presence of *cis* and *trans* isomers. Based on previous findings [4,6], the signals at the lowest field seemed to be the *trans* isomer, i.e. when the carbonyl and the heaviest groups have a *trans* relationship to each other. For compound **9**, ^{19}F NMR indicated that only one of the isomers was present. It has previously been reported that only one of the isomers could be observed in the solution when the solubility of a formanilide was too low [15]. In one report only one of the isomers of **4** was identified [6]. From a new ^{19}F NMR-spectrum of **4**, the presence of both *cis* and *trans* isomers at room temperature could be shown. NMR analyses at different concentrations were not performed, as the focus only was to confirm that the formyl compounds, **8** and **9**, were linear, which was also demonstrated for the hydrolyzed compounds **6** and **7**.

3. Experimental

The NMR spectra were obtained with a Varian Gemini 200 and a Varian VXR 300 S spectrometer at 24 °C. The ^{19}F chemical shifts are reported with respect to CCl_3F [with C_6F_6 and $C_6H_6CF_3$ as secondary references (−164.9 and −63.732 ppm[16])]. The accuracy of the shifts is ±0.01. The ^{1}H chemical shifts are reported with reference to $SiMe_4$. All spectra were recorded in deuterated acetone unless otherwise stated. The fluorines in the *ortho* positions belonging to AA′XX′M and AA′XX′ spin systems have been given as doublets (d), as shown in the spectra, and not as pseudo-doublets (ps-d). Mass spectroscopy data were obtained with a VG Micromass 7070E instrument with data system VG 11–250J. IR spectra were obtained from a FT-IR spectrometer, Bio-Rad Laboratories, Digilab Merlin 3.4.

3.1. General methods

All reactions were performed under oxygen-free nitrogen. Reaction mixtures were cooled on an ice bath, and diethyl ether was added (30–50 ml), followed by hydrolysis with HCl (10% or 20% v/w) to give an acidic mixture, extracted with diethyl ether, dried with anhydrous sodium sulfate, concentrated and chromatographed on silica (230–400 mesh) starting with light petroleum (bp 60 °C) as the elution solvent. When no more could be eluted from the mixture, increasing amounts diethyl ether or acetone were added to the light petroleum. When necessary, additional chromatography or recrystallization was performed using a mixture of light petroleum and diethyl ether. Hydrolysis of **8** and **9** was performed by a slow elution from a neutral alumina column (approximately 30 cm high and 2 cm in diameter) with a mixture of acetone and light petroleum (3:1). When hydrolysis was not complete, for **9**, the material was added to the same column again.

3.2. Reaction of pentafluoroanilide with dimethylformamide

Lithium amide (0.28 mol, 6.4 g) was added to pentafluoroaniline (0.12 mol, 22.0 g) in dried tetrahydrofuran (40 ml) at 0 °C, and then dimethylformamide (30 ml) was added after 20 min. The mixture was slowly heated and refluxed for 4 h, cooled and worked up.

Pentafluoroformanilide was extracted from the crude products with hot water, extracted with diethyl ether, dried and chromatographed giving in the following equation.

3.2.1. 10.8 g (49.0%) 2,3,4,5,6-Pentafluoroformanilide (*1*) [6]

IR (KBr): *ν* 3212(s,br), 1682(s), 1651(m), 1550(m), 1526(s), 1499(s), 1395(m), 1211(s), 1117(s), 1018(w), 1000(s), 972(s), 865(s), 694(m).

Remaining compounds on the column was washed off with acetone, dried and added to the rest of the dried crude materials.

Further work-up on silica gel columns gave yet three more compounds.

3.2.2. (0.15 g) 4,5,6,7-Tetrafluoro-1-pentafluorophenylbenzimidazole (*2*) (nc) mp 92–93.5 °C (sublimed)

EIMS 70 eV, *m/z* (rel. int.): 357(15), 356(100), 337(7), 310(13), 279(7), 156(13), 117(11). HRMS: $C_{13}H_1F_9N_2$ requires 355.9996, Found 355.9990.

^{19}F NMR: *δ* −144.09(d), −149.57(t), −151.61(t), −159.30(m), −159.85(t), −162.58(t), rel. int.: 2:1:1:3:1:1. ^{1}H NMR: *δ* 8.51.

IR (KBr): *ν* 3140(w), 3093(w), 1547(s), 1520(s), 1478(m), 1314(w), 1288(w), 1223(m), 1155(w), 1056(m), 995(s), 835(w), 810(m).

3.2.3. 1.9 g (8.5%) 2,3,4,5-Tetrafluoro-6-[(pentafluorophenyl)amino]formanilide (*3*) (nc) mp 160–161.5 °C

EIMS 70 eV, *m/z* (rel. int.): 375(20), 374(85), 357(32), 356(96), 346(32), 327(24), 326(100), 325(55), 307(30), 306(32), 179(23), 156(21), 152(19). HRMS: $C_{13}H_3F_9N_2O$ requires 374.0102, Found 374.0102.

^{19}F NMR: (*trans*) *δ* −147.89(m), −153.95(m), −156.74(d), −161.75(t), −166.43(m), −169.14(t), rel. int.: 1:1:2:1:3:1 (2F from the *cis* isomer is included in the multiplet centered at −166.43), (*cis*) *δ* −151.48(m), −153.18(m), −156.17(d), −162.11(t), −165.34(t),−166.43(m),−168.65(t), rel. int.: 1:1:2:1:1:2:1 (3F from the *trans* isomer is included in the multiplet centered at −166.43). ^{1}H NMR: *δ* (>NH) 7.3 (broad), (–CHO) 8.39, 8.31, (–NHCHO) 9.3 (broad).

IR (KBr): ν 3289(s), 1675(s), 1521(s), 1497(w), 1431(m), 1395(m), 1220(w), 1116(m), 1034(s), 1007(s), 976(w), 954(w), 870(m).

3.2.4. 1.1 g (4.9 %) 2,3,5,6-Tetrafluoro-4-[(pentafluorophenyl)amino]formanilide (**4**) [6]

^{19}F NMR: (*trans*) δ −148.35(m), −154.52(d), −157(m), −166.09(m) rel. int.: 2:2:2:3 (3F from the *cis* isomer is included in the multiplet centered at −166.09), (*cis*) δ −153.48(m), −154.70(d), −156.28(m), −166.09(m), rel. int.: 2:2:2:3 (3F from the *trans* isomer is included in the multiplet centered at −166.09).

^{1}H NMR: δ (>NH) 7.9 (broad), (–CHO) 8.47, (–NHCHO) 9.1 (broad).

IR (KBr): ν 3429(m), 3208(m), 1682(s), 1655(m), 1549(s), 1530(s), 1515(s), 1495(s), 1445(m), 1397(m), 1212(m), 1182 (m), 1117(m), 1034(s), 1001(m), 976(m), 959(m), 861(m), 814(w).

3.3. Reaction of pentafluoroformanilide with hexafluorobenzene

Sodium hydride (0.022 mol, 0.96 g, 55% in oil) was added to pentafluoroformanilide (0.02 mol, 4.22 g) in tetrahydrofuran (35 ml) at 0 °C and the mixture was stirred overnight at ambient temperature. Hexafluorobenzene (0.08 mol, 14.88 g) in tetrahydrofuran (5 ml) was added in a single portion at 0 °C. The mixture was stirred for 1 h followed by the addition of dimethylformamide (10 ml). The temperature was raised to light reflux and maintained for 3.5 h. The mixture was cooled to 0 °C, diethyl ether (50 ml) was added, followed by 20% HCl (50 ml) in one portion, and worked up. From the reaction mixture some of the lower oligomers were isolated.

3.3.1. 0.32 g (9.2%) Decafluorodiphenylamine (**5**)

This compound has previously been described [7].

3.3.2. 0.60 g (17.6%) N,N′-decafluorodiphenyl-tetrafluoro-1,4-phenylenediamine (**6**) (nc) mp 183–184 °C

EIMS 70 eV, *m/z* (rel. int.): 513(23), 512(100), 345(32), 256(12). HRMS: $C_{18}H_2F_{14}N_2$ requires 511.9994, Found 512.0003.

^{19}F NMR (Gemini 200): δ −157.41(s), −158.49(m), −168.29(m), −170.24 (t of t), rel. int.: 4:4:4:2. ^{1}H NMR (tetrahydrofuran$_D$): δ 7.77.

IR (KBr): ν 3430(m), 1559(m), 1522(s), 1511(s), 1470(w), 1445(m), 1313(w), 1260(w),1056(m), 1034(m), 967(m), 800 (w), 650(w).

3.3.3. Traces of α-pentafluorophenyl-ω-fluoro-tri(imino-tetrafluoro-1,4-phenylene) (**7**) (nc)

mp and IR were in accordance with the hydrolyzed compound obtained from *N*-formyl-4,4′-bis[(pentafluorophenyl)amino]-2,2′,3,3′,5,5′,6,6′-octafluorodiphenylamine (**9**).

3.3.4. 0.97 g (27.2%) N-Formyl-N,N′-decafluorodiphenyl-tetrafluoro-1,4-phenylene-diamine (**8**) (nc) mp 143.5–145.5 °C

EIMS 70 eV, *m/z* (rel. int.): 541(11), 540(51), 513(21), 512(100), 345(30), 256(11). HRMS: $C_{19}H_2F_{14}N_2O$ requires 539.9944, Found 539.9969. (Ionization with methane gave a trace signal at 569(0.67) which could indicate *N*,*N*′-diformyl-*N*,*N*′- dipentafluorophenyl-tetrafluoro-1,4-phenylenediamine.)

^{19}F NMR: (*trans*) δ −141.88(d), −146.56(d), −148.78(d), −151.86(t), −153.46(d), −159.63(t), −160.35(t), −161.86(m), rel. int.: 2:2:2:1:2:1:2:2, (*cis*) δ −143.89(d), −144.64(d), −148.97(d), −152.10(t), −153.73(d), −159.92(m), −161.86 (m), rel. int.: 2:2:2:1:2:3:2. ^{1}H NMR: δ (>NH) 8.63, (–CHO) 8.66.

IR (KBr): ν 3240(br,m), 3005(w), 1714(s), 1653(m), 1555 (w), 1514(br,s), 1467(w), 1329(m),1256(s), 1162(m), 1113(m), 1065(s), 1030(m), 981(s), 924(m), 807(m).

3.3.5. 0.36 g (10.2%) N-Formyl-4,4′-bis[(pentafluorophenyl)amino]-2,2′,3,3′,5,5′,6,6′-octafluorodiphenylamine (**9**) (nc) mp 142–144 °C

EIMS 70 eV, *m/z* (rel. int.): 705(2), 704(12), 703(41,), 677(4), 676(27), 675(100), 674(7), 508(9), 507(1), 493(2), 474(1), 473(2),347(2), 346(2), 345(13), 340(1.6), 338(4.4), 337.4(16), 311(2.3), 310(3.2), 280(2.0), 279(2.0)248(2.4). HRMS: $C_{25}H_3F_{18}N_3O$ requires 702.9989, Found 702.9961.

^{19}F NMR: δ −144.05(d), −146.82(d), −148.87(d), −149.09 (d), −153.36(d), −153.67(d), −159.87(t), −160.25(t), −161.88 (m), rel. int.: 2:2:2:2:2:2:1:1:4. ^{1}H NMR: δ (>NH) 8.11, (–CHO) 8.65.

IR (KBr): ν 3432(m), 3315(m), 1715(s), 1652(m), 1514(s), 1247(m), 1175(w), 1071(s), 1021(s), 1021(s), 978(s), 822(w), 793(w).

3.4. Hydrolysis of isolated formyl compounds

Hydrolysis of *N*-formyl-*N*,*N*′-dipentafluorophenyl-1,4-tetrafluorophenylenediamine (0.14 g) gave (0.08 g) *N*,*N*′-dipentafluorophenyl-1,4-tetrafluorophenylene-diamine (**6**).

Hydrolysis of *N*-formyl-4,4′-bis[(pentafluorophenyl)amino]-2,2′,3,3′,5,5′,6,6′-octafluorodiphenylamine (0.15 g) gave (0.08 g) α-pentafluorophenyl-ω-fluoro-tri(imino-tetrafluoro-1,4-phenylene) (**7**) (nc) mp 245–246 °C.

^{19}F NMR: δ −151.81(m), −152.70(d), −152.86(d), −162.28 (t), −164.31(t), rel. int.: 4:4:4:4:2. ^{1}H NMR: δ (>NH) 7.58.

IR (KBr): ν 3429(m), 1561(m), 1522(s), 1511(s), 1478(w), 1447(m), 1314(w), 1258(w),1062(m), 1040(m), 968(m), 788 (w), 651(w).

3.5. Reaction of acetanilide with hexafluorobenzene

To acetanilide (0.042 mol, 5.68 g) in tetrahydrofuran (30 ml) at 0 °C sodium hydride (0.042 mol, 2.03 g, 55% in oil) was added giving sodium acetanilide. The temperature was raised slowly to ambient whereupon the mixture was cooled in a bath with tap water. Then hexafluorobenzene (0.02 mol, 3.72 g) in tetrahydrofuran (10 ml) and dimethylformamide (10 ml) was added in one

portion to the sodium acetanilide. The temperature in the mixture was slowly raised (1 h) to light reflux and the mixture was refluxed for 3 h, cooled, hydrolyzed and worked up to give the following.

*3.5.1. 0.17 g, (2.8%) 2,3,4,5,6-Pentafluoro-N-phenylacetanilide (**10**)*

This compound has previously been described [14].

*3.5.2. 1.2 g, (14.4%) N,N'-diacetyl-N,N'-diphenyl-tetrafluoro-1,4-phenylenediamine (**11**) (nc) mp 252–252.5 °C.*

EIMS 70 eV, *m/z* (rel. int.): 418(2), 417(5), 416(24), 375(16), 374(69), 334(3), 333(22), 332(100),331(8), 292(4), 291(7), 255(3), 254(5), 241(3), 240(6), 43(20). HRMS: $C_{22}H_{16}N_2O_2F_4$ requires 416.1148, Found, 416.1132.

^{19}F NMR ($CDCl_3$): δ −144.10(br. band), −145.97(s), −146.34(br. band) (amount: 1.00:4.84:1.00) ^1H NMR ($CDCl_3$): δ ($-C_6H_5$) 7.40, 7.37, 7.24, ($-CH_3$) 2.04(s) (amount 5:3).

IR (KBr): ν 1691(s), 1595(m), 1497(s), 1371(m), 1325(w), 1302(s), 1248(w), 1067(w), 1030(w), 977(m), 969(m), 751(m), 700(s), 646(w), 572(m), 426(w).

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