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Reaction of difluorocarbene with anions from amides and oximes: Synthesis of *N*-difluoromethyl substituted amides, *O*-difluoromethylimidates or *O*-difluoromethyl substituted oximes under phase-transfer catalysis conditions

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Abstract

N-Aryl substituted amides react with chlorodifluoromethane in the presence of concentrated aqueous sodium hydroxide and benzyltriethylammonium chloride (TEBAC) as a catalyst in benzene (phase-transfer catalysis, PTC), affording mixtures of *N*- and *O*-difluoromethyl substituted derivatives. Amide anions are involved in this process. The reaction carried out with oximes gives *O*-difluoromethyl oxime ethers.

Keywords: Phase-transfer catalysis; Difluorocarbene; Amides; Oximes: *N*- and *O*-difluoromethylation

1. Introduction

Amides treated with strong bases generate ambident anions which react with alkylating agents giving *N*-substituted products [1]. Primary and secondary amides form with reactive alkylating agents (dialkylsulfates, trialkyloxonium salts, etc.) alkoxymethyleneiminium salts which were converted into *O*-substituted products (imidates) after deprotonation with a weak base [2]. Difluorocarbene is an electrophilic species, hence may react principally with amide or oxime anions at both nucleophilic centers but such processes are not reported. However, anions from some cyclic compounds exhibit ambident nature toward difluorocarbene. Thus, chlorodifluoromethane, the precursor of this carbene, afforded with the sodium salt of 2-hydroxyquinoline (carbostyril) [3] or with quinoxalin-2-ones and potassium carbonate or caesium fluoride [4], mixtures of *O*-difluoromethylated (main product) and *N*-difluoromethylated derivatives, in addition to 2-fluoroquinoxalines in the latter case.

Treating oximes with bases generated ambident anions which, depending on the geometry of the substrate, kind of base and type of alkylating agent, afforded *O*- or *N*-substituted derivatives (nitrones) or their mixtures [5–7]. According to one case described, acetoxime gave an *O*-difluoromethyl derivative in 3% yield when reacted with chlorodifluoromethane and sodium methoxide in methanol–ethyl ether mixture [8].

We have reported [9] recently that some nitrogen heterocycles are *N*-difluoromethylated with difluorocarbene generated from chlorodifluoromethane under conditions of phase-transfer catalysis (PTC) [10–12], i.e. in the presence of concentrated aqueous sodium hydroxide and benzyltriethylammonium chloride (TEBAC) as a catalyst.

2. Results and discussion

We studied the reaction of chlorodifluoromethane with amides and oximes under PTC conditions. To select optimal conditions, the amide **1a** was allowed to react with chlorodifluoromethane and sodium or potassium hydroxide in different solvents, with or without a phase-transfer catalyst. These preliminary experiments showed that difluorocarbene reacts with ambident amide anion $\mathbf{1a}^-$ at both nucleophilic centers, giving **2a** and **3a**. Of the basic systems studied, 50% aq. sodium hydroxide/benzene/TEBAC as a catalyst gave the best results, although total yields of difluoromethylated derivatives **2a** and **3a** did not exceed 54%. Potassium hydroxide in aqueous acetone, the system used previously for difluoromethylation of some nitrogen heterocycles with chlorodifluoromethane [13], gave poor yields in this case as did solid–liquid variants of PTC.

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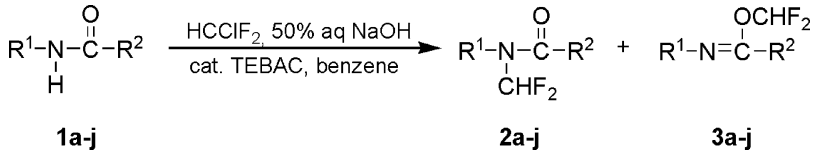
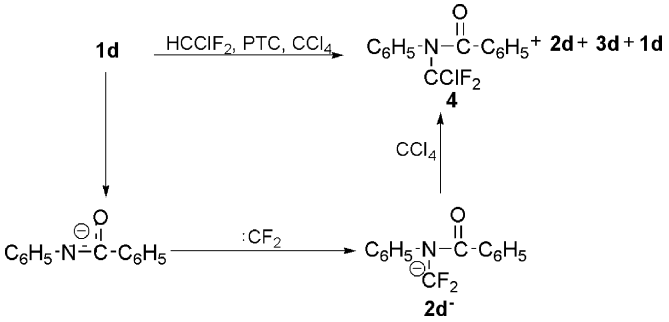


Scheme 1.

Next, a series of amides **1b–j** was allowed to react with chlorodifluoromethane under the above conditions, affording mixtures of *N*- and *O*-difluoromethylated products, **2b–j** and **3b–j**, respectively, in moderate yields (Scheme 1; Table 1).

To check the stability of difluoromethylated products, a mixture of **2d** and **3d** was stirred in benzene at room temperature for 4–5 h with water, 20% or 50% aq. sodium hydroxide or 5% hydrochloric acid. The products **2d** and **3d** were stable to neutral or basic conditions; hydrochloric acid caused cleavage of both isomers with the formation of **1d**, but **3d** reacted at a much higher rate. Evidently, the difluoromethyl group in **2d** was first converted into formyl, and the product thus formed was easily deformylated giving **1d**. Products **2** and **3** were stored in a refrigerator.

The experiments collected in Table 1 revealed that only those amides which are *N*-substituted with an aryl group (Table 1, Entries 1–7) were able to form the products **2** and **3** in moderate yields, in other cases (Table 1, Entries 8–10), the yields were negligible. This results are due to the increased acidity of *N*-aryl substituted amides **1a–g** which are able to generate anions in sufficiently high concentration (but not exceeding that of the catalyst) for reaction with difluorocarbene. Alternative mechanistic pathway, i.e. reaction of the amide nitrogen electron pair with difluorocarbene is not feasible since amides are too weak bases. To confirm the presence of difluoromethyl carbanion, we carried out the reaction of *N*-phenylbenzamide (**1d**) with chlorodifluoromethane, under PTC conditions in carbon tetrachloride (Scheme 2). This reagent is known to chlorinate anions easily via a halophilic process [14,15].

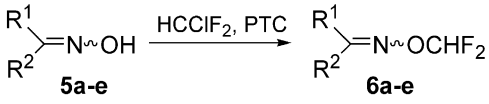


Scheme 2.

Among the products formed, we identified *N*-chlorodifluoro-derivative **4**, which resulted by chlorination of anion **2d** with carbon tetrachloride.

These investigations were completed by reaction of chlorodifluoromethane with oximes **5a–e** under PTC conditions (Scheme 3, Table 2).

In this case we noticed formation of only *O*-difluoromethyl substituted products **6a–e** in a rather low yields. Ketoximes **5a** and **b** (Entries 1, 2) gave the products in higher yields than aldoximes **5c–e**, and oxime of methylethylketone did not react at all. The products **6** are much more stable than **2** and particularly **3**.



Scheme 3.

Table 1
Reaction of amides **1a–j** with chlorodifluoromethane under PTC conditions

Entry	Substrates **1**, products **2**, **3**	R^1	R^2	Time (h)	Temperature (°C)	Yield[a] (%)	
						2	**3**
1	**a**	2-$H_3CC_6H_4$	CH_3	2	20	12	42
2	**b**	C_6H_5	CH_3	3	40	20	6
3	**c**	4-$H_3COC_6H_4$	CH_3	3	20	13	26
4	**d**	C_6H_5	C_6H_5	3	20	26	19
5	**e**	4-$H_3CC_6H_4$	C_6H_5	0.5	20	28	23
6	**f**	4-$H_3COC_6H_4$	C_6H_5	3	20	13[b]	2[b]
7	**g**	4-NCC_6H_4	CH_3	1	20	16	10
8	**h**	CH_3	CH_3	5	20	7[c]	
9	**i**	C_2H_5	CH_3	6	20	6[c]	
10	**j**	CH_3	C_6H_5	5	20	5[c]	

[a] Isolated pure (≥97%) products.
[b] The mixture of **2f** and **3f** was isolated in 41% yield.
[c] Content in the reaction mixture.

Table 2
Reaction of oximes **5a–e** with chlorodifluoromethane under PTC conditions

Entry	**5** and **6**	R^1	R^2	Time (h)	Content of **6** in the reaction mixture determined by GC (%)	Yield[a] (%)
1	**a**	C_6H_5	C_6H_5	1.5	48	22
2	**b**	C_6H_5	CH_3	3	45	13
3	**c**[b]	4-$CH_3OC_6H_4$	H	5	27	7
4	**d**[b]	C_6H_5	H	3	24	6
5	**e**	$-(CH_2)_5-$		5	13	–

[a] Purity ≥ 99% (by GC).
[b] *syn*-Oximes **5c** and **d** were used.

We have shown that difluorocarbene (generated from chlorodifluoromethane) react with ambident amide anions under typical PTC conditions giving mixtures of *N*- **2** and *O*-difluoromethylated **3** products, in low to moderate yields. A similar process applicable to oximes **5** led to formation of only *O*-difluoromethylated derivatives **6**, in a rather low yield. Taking into account availability of difluorocarbene precursor and amides or oximes as well as the simplicity of PTC procedures, these processes may be applied for synthesis of difluoromethyl substituted derivatives **2**, **3** and **6**. However, similar retention times of **2** and **3** create some problems during their separation by column chromatography.

3. Experimental

3.1. General

Melting points were measured on a capillary melting point apparatus and are uncorrected. ^{1}H NMR spectra were recorded on a Varian Mercury (at 400 MHz) or Varian Gemini (at 200 MHz), ^{13}C NMR spectra on a Varian Mercury (at 100 MHz) spectrometers in $CDCl_3$ with tetramethylsilane as internal reference. ^{19}F NMR spectra were recorded on a Varian Mercury (at 376 MHz) in $CDCl_3$ with trifluoroacetic acid as external reference (d = −77.00 ppm). All chemical shifts are reported in ppm. The abbreviations used are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Gas chromatography (GC) analyses were carried out on Agilent 6850 Series GC System equipped with HP-50+ (30 m) column. Microanalyses were obtained using a CHN/S Perkin-Elmer 2400 element analyzer. Column chromatography was performed using Merck basic aluminum oxide 90 (70–230 mesh) or silica gel with hexane and ethyl acetate mixtures (gradient) as eluents. Chlorodifluoromethane and amides **1b**, **1h–j** were commercial materials while amides **1a** [16], **1c** [17], **1d** [18], **1e**, **1f** [19], **1g** [20] and oximes **5a** [21], **5b** [22], **5c** [23], **5d** [24], **5e** [25] were prepared by literature procedures.

3.2. General procedure for difluoromethylation of amides *1a–j*

Into a three-necked, round bottomed flask equipped with a reflux condenser, mechanical stirrer and glass pipe for introducing chlorodifluoromethane, amide **1a–j** (10 mmol), 50% aq. NaOH (1.6 ml, 2.40 g, 30 mmol), TEBAC (0.11 g, 0.5 mmol) and benzene (20 ml) were placed. The content of the flask was stirred for ca. 2 min, then chlorodifluoromethane was bubbled through the mixture. The progress of the reaction was monitored by GC, the reaction was diluted with benzene (50 ml) when content of **2** and **3** in the reaction mixture did not increase. The organic phase was decanted from semi-solid material and dried over $MgSO_4$, the solvent was evaporated and the residue was purified by column chromatography on aluminum oxide.

2a, solid, mp 37–40 °C, ^{1}H NMR δ 1.80 (s, 3H, CH_3), 2.27 (s, 3H, CH_3), 7.20–7.38 (m, 4H, aromatic C), 7.57 (t, *J* = 61 Hz, 1H, CHF_2). ^{13}C NMR δ 17.9 (CH_3), 22.6 (CH_3), 108.5 (t, *J* = 241 Hz, CHF_2), 127.3, 130.0, 130.9, 131.6, 133.4, 138.2, 171.8 (C=O). ^{19}F NMR δ −94.1, −102.5 (part AB of ABX, J_{F-F} = 230 Hz, J_{H-F} = 61 Hz, 2F). HRMS calcd. for $C_{10}H_{11}NOF_2$: 199.0809. Found: 199.0815.

2b, solid, mp 74–77 °C, ^{1}H NMR δ 1.88 (s, 3H, CH_3), 7.2–7.5 (m, 5H, aromatic H), 7.55 (t, *J* = 61 Hz, 1H, CHF_2). ^{13}C NMR δ 22.6 (CH_3), 107.9 (t, *J* = 241.15 Hz, CHF_2), 129.3, 129.4, 130.0, 134.0, 171.2 (C=O). ^{19}F NMR δ −97.5 (d, *J* = 61 Hz, 2F). HRMS calcd. for $C_9H_9NOF_2$: 185.0652. Found: 185. 0655.

2c, solid, mp 58–60 °C, ^{1}H NMR δ 1.86 (s, 3H, CH_3), 3.83, (s, 3H, CH_3–O), 6.93–7.20 (m, 4H, aromatic H), 7.52 (t, *J* = 62 Hz, 1H, CHF_2). ^{13}C NMR δ 22.9 (–CH_3), 55.4 (–O–CH_3), 107.9 (t, *J* = 240.4 Hz), 114.7, 126.6, 131.2, 160.3, 171.9. ^{19}F NMR δ −97.4 (d, *J* = 62 Hz, 2F). HRMS calcd. for $C_{10}H_{11}NO_2F_2$: 215.0758. Found: 215.0749.

2d, solid, mp 92–95 °C, ^{1}H NMR δ 7.20–7.41 (m, 10H, aromatic H), 7.62 (t, *J* = 60 Hz, 1H, CHF_2). ^{13}C NMR δ 109.4 (t, *J* = 243 Hz, 1C, CHF_2), 128.0, 128.7, 128.8, 129.1, 130.0, 131.1, 133.5, 134.9, 170.6 (C=O). ^{19}F NMR δ −94.9 (d, *J* = 53 Hz, 2F). HRMS calcd. for $C_{14}H_{11}NOF_2$: 247.0809. Found: 247.0803.

2e, solid, mp 37–39 °C, ^{1}H NMR δ 2.31 (s, 3H, CH_3), 7.00–7.42 (m, 9H, aromatic H), 7.59 (t, *J* = 61 Hz, 1H, CHF_2). ^{13}C NMR δ 21.2 (CH_3), 109.6 (t, *J* = 242.4 Hz, CHF_2), 125.1, 128.2, 128.9, 129.0, 131.2, 132.1, 134.0, 138.9, 170.9 (C=O). ^{19}F NMR δ −96.1 (br s, 2F) HRMS calcd. for $C_{15}H_{13}NOF_2$: 261.0965. Found: 261.0969.

2f, solid, mp 68–71 °C, ^{1}H NMR δ 3.81 (s, 3H, CH_3–O), 6.85–7.49 (m, 9H, aromatic H), 7.66 (t, *J* = 61 Hz, CHF_2). ^{13}C NMR δ 55.4 (CH_3), 109.5 (t, *J* = 243.1 Hz), 114.4, 127.4, 128.1,

128.9, 131.2, 131.4, 133.8, 159.7, 171.0 (C=O). ^{19}F NMR δ −96.5 (br s, 2F). HRMS calcd. for $C_{15}H_{13}NO_2F_2$: 277.0914. Found: 277.0901.

2g, solid, mp 115–118 °C, ^{1}H NMR δ 1.89 (s, 3H, CH_3), 7.42–7.79 (m, 4H, aromatic H), 7.49 (t, J = 61 Hz, 1H, CHF_2). ^{13}C NMR δ 22.9 (CH_3), 108.0 (t, J = 242.6 Hz, 1C, CHF_2), 113.8, 117.5, 131.1, 133.5, 138.2, 169.9. ^{19}F NMR δ −106.1 (br s, 2F) HRMS calcd. for $C_{10}H_8N_2OF_2$: 210.0605. Found: 210.0599.

3a, liquid, ^{1}H NMR δ 1.90 (s, 3H, CH_3), 2.11 (s, 3H, CH_3), 6.65–7.21 (m, 4H, aromatic H), 7.42 (t, J = 73 Hz, 1H, CHF_2). ^{13}C NMR δ 15.8 (CH_3), 17.8 (CH_3), 113.2 (t, J = 254 Hz, 1C, CHF_2), 119.8, 124.3, 126.7, 128.8, 130.7, 145.1, 156.8. ^{19}F NMR δ −86.9 (d, J = 73 Hz, 2F). HRMS calcd. for $C_{10}H_{11}NOF_2$: 199.0809. Found: 199.0814.

3b, liquid, ^{1}H NMR δ 2.03 (s, 3H, CH_3), 6.86–7.46 (m, 5H, aromatic H), 7.46 (t, J = 72 Hz, 1H, CHF_2). ^{13}C NMR δ 15.6 (CH_3), 112.9 (t, J = 253.7 Hz, CHF_2), 120.5, 124.1, 129.2, 146.3, 157.2. ^{19}F NMR δ −86.7 (d, J = 72 Hz, 2F). HRMS calcd. for $C_9H_9NOF_2$: 185.0652. Found: 185. 0655.

3c, liquid, ^{1}H NMR δ 1.96 (s, 3H, CH_3), 3.80 (s, 3H, CH_3–O), 6.72–6.88 (m, 4H, aromatic H), 7.37 (t, J = 72 Hz, 1H, CHF_2). ^{13}C NMR δ 19.4 (–CH_3), 55.5 (–O–CH_3), 113.1 (t, J = 253, 6 Hz, CHF_2), 114.0, 114.5, 121.7, 124.6, 130.5, 139.6, 156.5. ^{19}F NMR δ −86.7 (d, J = 72 Hz, 2F). HRMS calcd. for $C_{10}H_{11}NO_2F_2$: 215.0758. Found: 215.0757.

3d, solid, mp 158–160 °C, ^{1}H NMR δ 6.82–7.45 (m, 10H, aromatic H), 7.53 (t, J = 72 Hz, 1H, CHF_2). ^{13}C NMR δ 113.8 (t, J = 254.6 Hz, 1C, CHF_2), 121.0, 124.1, 128.4, 129.4, 129.7, 131.4, 146.2. ^{19}F NMR δ −85.4 (d, J = 72 Hz, 2F). HRMS calcd. for $C_{14}H_{11}NOF_2$: 247.0809. Found: 247.0812.

3e, liquid, ^{1}H NMR δ 2.32 (s, 3H, CH_3), 6.70–7.42, (m, 9H, aromatic H), 7.52 (t, J = 72 Hz, 1H, CHF_2). ^{13}C NMR δ 20.9 (CH_3), 113.8 (t, J = 254,4 Hz), 120.9, 128.4, 129.6, 129.9, 131.2, 133.6, 143.5. ^{19}F NMR δ −86.3 (d, J = 72 Hz, 2F). HRMS calcd. for $C_{15}H_{13}NOF_2$: 261.0965. Found: 261.0974.

3f, liquid, ^{1}H NMR δ 3.77 (s, 3H, CH_3O), 6.73–7.38 (m, 9H, aromatic H), 7.48 (t, J = 76 Hz, C, 1H, CHF_2). ^{13}C NMR δ 55.5 (CH_3–O), 113.8 (t, J = 261.3 Hz, CHF_2), 114.6, 122.3, 128.4, 129.6, 131.2, 139.0, 156.5. ^{19}F NMR δ −86.2 (d, J = 73 Hz, 2F). HRMS calcd. for $C_{15}H_{13}NO_2F_2$: 277.0914. Found: 277.0906.

3g, solid, mp 32–35 °C, ^{1}H NMR δ 1.96 (s, 3H, CH_3), 6.86–7.64 (m, 4H, aromatic H), 7.30 (t, J = 71 Hz, 1H, CHF_2). ^{13}C NMR δ 15.9, 107.8, 112.9 (t, J = 255.1 Hz, 1C, CHF_2), 118.8 (CN), 121.4, 133.4, 150.4, 157.8. ^{19}F NMR δ −96.0 (d, J = 71 Hz, 2F). HRMS calcd. for $C_{10}H_8N_2OF_2$: 210.0605. Found: 210.0609.

3.3. Preparation of N-chlorodifluoromethylbenzanilide (*4*)

To a three-necked, round bottomed flask equipped with reflux condenser, mechanical stirrer and glass pipe for introducing chlorodifluoromethane, benzanilide (**1d**, 3.94 g, 20 mmol), 50% aq. NaOH (3.2 ml, 4.80 g, 60 mmol), TEBAC (0.23 g, 1 mmol) and CCl_4 (30 ml) were placed. The content of the flask was stirred for ca. 2 min, then chlorodifluoromethane was bubbled through the reaction mixture for 3 h. The progress of the reaction was monitored by GC, the reaction was diluted with CCl_4, when content of **4** in the reaction mixture did not increase. The organic phase was decanted (GC indicated 14% of **4**, 15% of **2d**, 17% of **3d** and unreacted **1d**), the product **4** was isolated with 2% yield by column chromatography on silica gel.

Stirring of **2d** with carbon tetrachloride under the same conditions did not afford **4**.

4, solid, mp 40–43 °C, ^{1}H NMR δ 7.19–7.33 (m, 8H, aromatic H), 7.48–7.51 (m, 2H, aromatic H). ^{13}C NMR δ 123.2 (t, J = 285.1, 1C, CHF_2), 128.0, 129.2, 129.3, 130.3, 131.6, 133.6, 136.7, 170.1. ^{19}F NMR δ −39.8 (s, 2F). Anal. calcd. for $C_{14}H_{10}NOClF_2$: C, 59.7, H, 3.6, N, 5.0, Cl, 12.6. Found: C, 59.5, H, 3.4, N, 5.2, Cl, 12.3.

3.4. General procedure for difluoromethylation of oximes *5a–e*

The reactions were carried out as described in Section 3.2, but products were isolated by column chromatography on silica gel.

6a, liquid, ^{1}H NMR δ 6.76 (t, J = 73 Hz, 1H, CHF_2), 7.36–7.51 (m, 10H, aromatic H). ^{13}C NMR δ 118.8 (t, J = 259 Hz, 1C, CHF_2), 128.2, 128.4, 128.6, 129.1, 129.7, 130.6, 131.9, 134.8, 162.2. ^{19}F NMR δ −91.6 (d, J = 73 Hz, 2F). Anal. calcd. for $C_{14}H_{11}NOF_2$: C, 68.0, H, 4.5, N, 5.7. Found: C, 67.9, H, 4.5, N, 5.8.

6b, liquid, ^{1}H NMR δ 2.36 (s, 3H, CH_3), 6.78 (t, J = 73 Hz, 1H, CHF_2), 7.41–7.46 (m, 3H, aromatic H), 7.66–7.71 (m, 2H, aromatic H). ^{13}C NMR δ 13.9, 119.2 (t, J = 256.4 Hz, 1C, CHF_2), 126.7, 128.7, 130.5, 134.9, 160.5. ^{19}F NMR δ −91.4 (d, J = 73 Hz, 2F). Anal. calcd. for $C_9H_9NOF_2$: C, 58.4, H, 4.9, N, 7.6. Found: C, 58.6, H, 4.7, N, 7.6.

6c, solid, mp 26–28 °C, ^{1}H NMR δ 3.84 (s, 3H, CH_3O), 6.69 (t, J = 73 Hz, 1H, CHF_2), 6.91–6.94 (m, 2H, aromatic H), 7.56–7.59 (m, 2H, aromatic H), 8.17 (s, 1H). ^{13}C NMR δ 55.3, 114.3, 118.5 (t, J = 257.1 Hz, 1C, CHF_2), 122.7, 129.5, 153.5, 162.1. ^{19}F NMR δ −91.8 (d, J = 73 Hz, 2F). Anal. calcd. for $C_9H_9NO_2F_2$: C, 53.7, H, 4.5, N, 6.9. Found: C, 53.8, H, 4.5, N, 6.8.

6d, liquid, ^{1}H NMR δ 6.72 (t, J = 72 Hz, 1H, CHF_2), 7.40–7.47 (m, 3H, aromatic H), 7.63–7.66 (m, 2H, aromatic H), 8.24 (s, 1H). ^{13}C NMR δ 118.4, 118.5 (t, J = 256.6 Hz, 1C, CHF_2), 124.7, 127.8, 128.9, 130.2, 131.3, 153.9. ^{19}F NMR δ −91.9 Hz (d, J = 72 Hz, 2F). Anal. calcd. for $C_8H_7NOF_2$: C, 56.1, H, 4.1, N, 8.2. Found: C, 56.2, H, 4.3, N, 8.2.

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