

Copper-Catalyzed Dehydration of Primary Amides to Nitriles

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Received: 6 June 2011 / Accepted: 25 June 2011 / Published online: 13 July 2011
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Abstract In the present study we introduce a copper-catalyzed protocol for the dehydration of primary amides to their corresponding nitriles applying *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (MSTFA) as silylation reagent. For that purpose investigations of various reaction parameters (copper source, solvent, temperature, MSTFA and copper loading) have been carried out to find suitable reaction conditions. Simple copper(I) chloride (2.5 mol%) and MSTFA (2.0 equiv) in toluene allow for the straightforward synthesis of a variety of nitriles (15 examples).

Keywords Copper · Homogeneous catalysis · Dehydration · Amides · Nitriles

1 Introduction

Nitrile groups are abundant functionalities in organic chemistry and in consequence relevant for the production of countless products in chemical industries [1]. Up to now manifold syntheses have been established to access nitriles. Among those protocols the dehydration of amides is an attractive approach and diverse reagent to perform this reaction have been introduced [2–15]. However, the application of stoichiometric amounts of highly acidic and basic compounds disfavors the dehydration of sensitive substrate and reduces the generality of the methods. In this regard the application of transition metal catalyst turned out to be an efficient and flexible alternative and various

successful procedures have been launched so far (e.g., W, Ru, Pd, Rh, V, and U) [16–25]. More recently the potential of abundant, cheap and low toxic metals (e.g., Fe, Zn) as catalysts for the dehydration of primary amides have been demonstrated [26–28]. In various cases hydrosilanes have been applied as dehydration reagent and allow for milder reaction conditions. Nevertheless, difficulties can arise in the presence of functional groups sensitive by side-reactions, due to the reduction abilities of the metal-hydrosilane combination. As the dehydration of primary amides is proposed to proceed via silylation we wonder if the hydrosilanes can be replaced by silylation reagents to avoid additional reduction [25, 27, 28]. In accordance to that we herein present the efficient application of copper precursors in combination with *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (MSTFA) in the dehydration of a variety of primary amides to yield the corresponding nitriles [29].

2 Results and Discussion

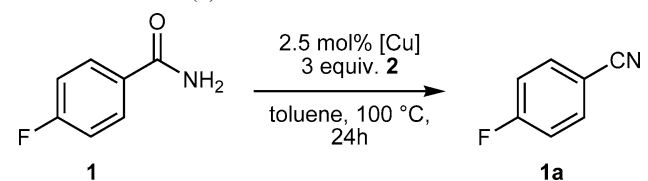
Initial experiments were carried out to find suitable reaction conditions. In this regard the dehydration of 4-fluorobenzamide (**1**) in the presence of catalytic amounts of Cu(acac)₂ and *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (MSTFA, **2**) was chosen as model reaction (Table 1). Excellent yields of nitrile **1a** (>99%) were realized with 5 mol% catalyst and 3.0 equivalents of MSTFA in toluene at 100 °C after 24 h (Table 1, entry 3). In contrast, in the absence of catalytic amounts of copper as well as in the absence of MSTFA no product formation was observed (Table 1, entries 1 and 2). Besides toluene as solvent, *n*-hexane, 1,4-dioxane, THF and dichloromethane were tested, resulting in excellent yields for 1,4-dioxane (Table 1, entries 3–7). In case of *n*-hexane and THF

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moderate to good yields were noticed, while performing the dehydration at lower reaction temperature (70 °C). Moreover, the copper catalyst still demonstrated high activity at lower concentrations, since extraordinary yields (>99%) were monitored at 1.0 mol% Cu (Table 1, entries 8–9). On the other hand the amount of MSTFA was reduced stepwise, showing that at least 2.0 equivalents of MSTFA are required to obtain reasonable amounts of **1a** (Table 1, entries 10–11). In addition the influence of the source of copper on the reaction outcome was examined (Table 2). Various copper precursors were subjected to the dehydration of **1** in the presence of **2** including copper(I) and copper(II) salts. In case of copper(I) precursors best activity was observed for CuCl, while experiments with CuI and Cu₂O gave lower yields (Table 2, entries 1–3). Best performance for copper(II) sources were monitored for Cu(acac)₂, CuCl₂ and Cu(NO₃)₂·5H₂O. For further studies CuCl was applied, because of environmental considerations.

With suitable reaction parameters in hand (2.5 mol% CuCl, 2.0 equiv. MSTFA, toluene, 100 °C, 24 h) we started to explore the scope and limitation of the system in the dehydration of various aromatic and aliphatic primary amides to the corresponding nitriles (Table 3). First the dehydration of substituted benzamides was investigated to clarify the influence of electronic and steric factors (Table 3, entries 1–10). Substitution with alkyl groups in *para*- as well as *meta*-position resulted in excellent yields, while *ortho*-substituted substrate **5** a reduced yield was

Table 2 Influence of the copper source on the dehydration of 4-fluorobenzamide (**1**)



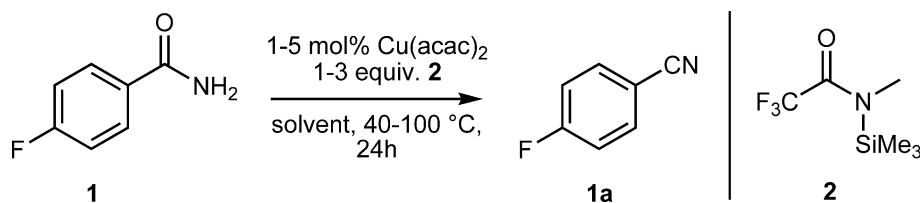
Entry	Copper source	Yield [%] ^a
1	CuCl	>99
2	CuI	48
3	Cu ₂ O	50
4	Cu(acac) ₂	99
5	CuCl ₂	>99
6	CuO	85
7	Cu(NO ₃) ₂ ·5H ₂ O	>99

Reaction conditions: **1** (0.72 mmol), catalyst (2.5 mol%), MSTFA (3 equiv.), toluene (2.0 mL), 24 h, 100 °C

^a Determined by GC methods

noticed (Table 3, entries 1–4). Next the effect of electron withdrawing halide functionalities were studied (Table 3, entries 5–7). In all cases excellent yields were achieved, only for *p*-bromobenzamide (**8**) 80% yield was observed. Also with CF₃, OMe, and NO₂ lower yields were attained (Table 3, entries 8–10). Moreover, a challenging heteroaromatic primary amide was converted in excellent yield (Table 3, entries 12). Noteworthy, the presented system is

Table 1 Copper-catalyzed dehydration of 4-fluorobenzamide (**1**)



Entry	Copper loading [mol%]	Solvent	MSTFA loading [equiv]	T [°C]	Yield [%] ^a
1	–	Toluene	3	100	<1
2	5	Toluene	–	100	<1
3	5	Toluene	3	100	>99
4	5	Hexane	3	70	39
5	5	THF	3	70	92
6	5	1,4-dioxane	3	100	>99
7	5	Dichloromethane	3	40	<1
8	2.5	Toluene	3	100	>99
9	1	Toluene	3	100	>99
10	5	Toluene	2	100	>99
11	5	Toluene	1	100	43

Reaction conditions: **1** (0.72 mmol), catalyst (1–5 mol%), MSTFA (1–3 equiv.), solvent (2.0 mL), 24 h, 40–100 °C

^a Determined by GC methods

Table 3 Scope and limitation of the copper-catalyzed dehydration of amides

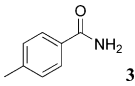
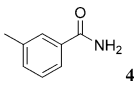
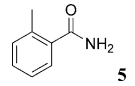
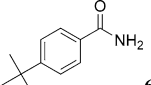
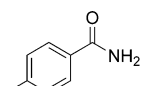
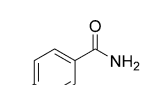
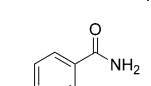
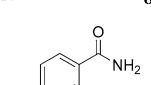
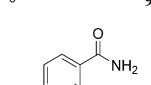
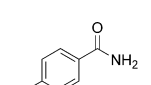
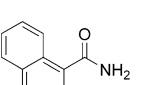
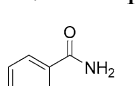
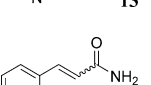
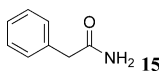
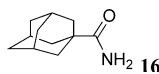
Entry	Substrate	Yield ^a
1		>99
2		>99
3		77
4		>99
5		>99
6		>99
7		80
8		94
9		83
10		81
11		6
12		>99
13		92

Table 3 continued

Entry	Substrate	Yield ^a
14		38
15		92

Reaction conditions: substrate (0.72 mmol), CuCl (2.5 mol%), MSTFA (2.0 equiv.), toluene (2.0 mL), 24 h, 100 °C

^a Determined by GC-MS methods and ¹H NMR

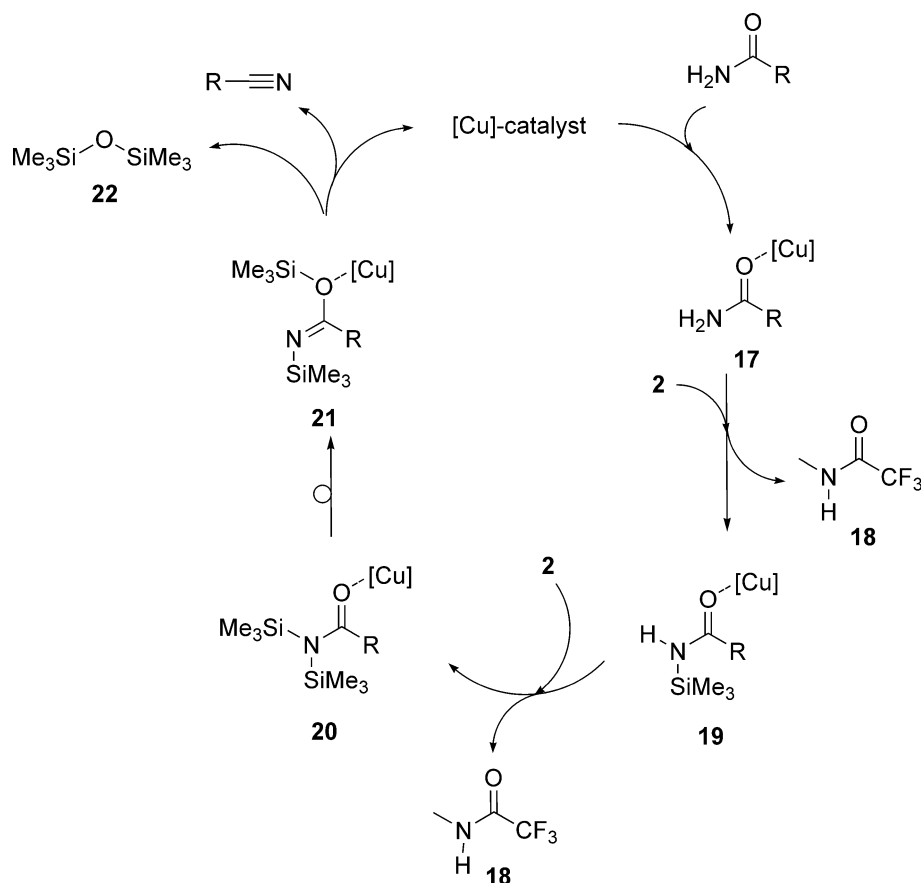
highly selective for the transformation of amides to nitriles in the presence of functional groups sensitive to reduction, e.g., nitro-groups and olefins. Finally, alkyl based amides were dehydrated in moderate to good yields (Table 3, entries 14 and 15).

With respect to the reaction mechanism we propose a similar pathway as introduced by the groups of Nagashima for ruthenium and Beller for iron (Scheme 1) [16, 25–28]. Initially, the copper catalyst activates the primary amide by coordination of the carbonyl oxygen and provides the possibility for MSTFA to transfer the trimethylsilyl group to yield the monosilylated species **19** and *N*-methyltrifluoroacetamide (**18**). Noteworthy, the monosilylated compound of substrate **1** was synthesized and mixed with catalytic amounts of CuCl in the absence of additional MSTFA. After work-up only marginal amounts of the nitrile **1a** were observed. This experiment clearly indicates the necessity of 2.0 equivalents of MSTFA for successful nitrile formation. Further on, to the monosilylated species **19** a second equivalent of [Me₃Si] is transferred by MSTFA resulting in the intermediate **20** and **18**. The intermediate **20** is rearranged to the corresponding *N,O*-bis(trimethylsilyl)imidate **21**, which eliminates in the presence of copper the nitrile and the siloxane **22**. Finally, the copper catalyst is recovered and can convert additional substrate molecules. To prove the pathway via the bisilylated species **20** the bisilylated compound of **1** was synthesized and reacted with catalytic amounts of copper(I) chloride. An excellent yield (>99%) of nitrile **1a** was noticed after 24 h, while for the monosilylated compound a yield of 7% was monitored (Scheme 2).

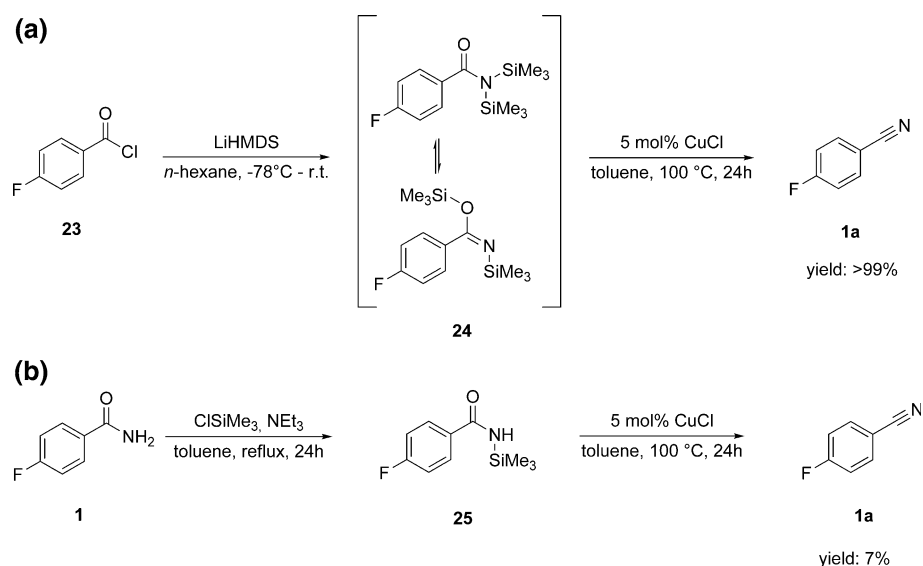
3 Conclusion

In summary, we have verified the value of a novel system for the dehydration of amides to nitriles composed of catalytic amounts of CuCl and 2.0 equivalents of *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide as dehydration reagent. Excellent reactivity and selectivity was observed in the dehydration of different amides.

Scheme 1 Mechanistic proposal for the dehydration of amides in the presence of a copper catalyst



Scheme 2 Synthesis of mono- and bisilylated benzamide **24** and **25** and application in the dehydration protocol



4 Experimental Section

4.1 General

All compounds were used as received without further purification. THF and toluene were dried applying standard procedures. ^1H , and ^{13}C NMR spectra were

recorded on a Bruker AFM 200 spectrometer (^1H : 200.13 MHz; ^{19}F : 188.31 MHz ^{13}C : 50.32 MHz) using the proton signals of the deuterated solvents as reference. GC-MS measurements were carried out on a Shimadzu GC-2010 gas chromatograph (30 m Rxi-5 ms column) linked with a Shimadzu GCMA-QP 2010 Plus mass spectrometer.

4.2 General Procedure for the Dehydration of Amides

A pressure tube was charged with an appropriate amount of copper(I) chloride (0.018 mmol, 2.5 mol%), and the corresponding amide (0.72 mmol). After addition of toluene (2.0 mL) 2.0 equivalents of MSTFA (1.44 mmol) were added and the reaction mixture was stirred in a preheated oil bath at 100 °C for 24 h. The mixture was cooled to room temperature and an aliquot was taken for GC-analysis (30 m Rxi-5 ms column, 40–300 °C). The reaction mixture was dissolved in diethyl ether and purified by column chromatography after that the solvents were removed under reduced pressure. The analytical properties of the corresponding nitriles are in agreement with literature.

4.3 4-Fluorobenzonitrile (**1a**) [30]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.57–7.67 (m, 2H), 7.05–7.18 (m, 2H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 167.6, 162.5, 134.8, 134.6, 118.0, 117.1, 116.6, 108.6, 108.5 ppm; ^{19}F NMR (THF-d_8 , 188 MHz) δ = –111.0 (m) ppm; MS (ESI) m/z = 121 (100, M^+), 94 (39).

4.4 4-Methylbenzonitrile (**3a**) [31]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.55–7.63 (m, 2H), 7.04–7.14 (m, 2H), 2.87 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 137.5, 132.8, 127.6, 118.8, 109.6, 26.2 ppm; MS (ESI) m/z = 117 (100, M^+), 90 (47), 63 (14).

4.5 3-Methylbenzonitrile (**4a**) [31]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.23–7.43 (m, 4H), 2.34 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 139.3, 133.7, 132.4, 132.1, 129.2, 119.0, 112.2, 21.1 ppm; MS (ESI) m/z = 117 (100, M^+), 90 (50).

4.6 2-Methylbenzonitrile (**5a**) [31]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.35–7.60 (m, 2H), 7.15–7.34 (m, 2H), 2.48 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 141.9, 132.7, 132.4, 130.2, 126.3, 118.7, 112.6, 20.4 ppm; MS (ESI) m/z = 117 (100, M^+), 90 (56), 63 (14).

4.7 4-tert-Butylbenzonitrile (**6a**) [31]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.38–7.64 (m, 4H), 1.28 (s, 9H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 156.7, 132.3, 126.2, 109.4, 35.3, 30.9 ppm; MS (ESI) m/z = 159 (21, M^+), 144 (100), 116 (71), 104 (15).

4.8 4-Chlorobenzonitrile (**7a**) [32]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.21–7.46 (m, 4H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 139.4, 133.7, 129.1, 118.9, 112.1 ppm; MS (ESI) m/z = 137 (100, M^+), 102 (38), 75 (17), 50 (15).

4.9 4-Bromobenzonitrile (**8a**) [33]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.43–7.65 (m, 4H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 133.4, 132.7, 128.0, 118.0, 111.2 ppm; MS (ESI) m/z = 181 (60, M^+), 102 (100), 75 (31), 50 (24).

4.10 4-(Trifluoromethyl)benzonitrile (**9a**) [32]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.62–7.84 (m, 4H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 134.2, 132.7, 126.2, 125.7, 117.5, 116.0 ppm; ^{19}F NMR (CDCl_3 , 188 MHz) δ = –63.6 ppm; MS (ESI) m/z = 171 (100, M^+), 152 (40), 121 (70), 75 (20), 50 (11).

4.11 4-Methoxybenzonitrile (**10a**) [34]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.48–7.60 (m, 2H), 6.86–6.96 (m, 2H), 3.82 (s, 3H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 162.9, 134.0, 119.2, 114.8, 103.8, 55.5 ppm; MS (ESI) m/z = 133 (100, M^+), 103 (46), 90 (47), 76 (12), 63 (17).

4.12 4-Nitrobenzonitrile (**11a**) [34]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.78–8.40 (m, 4H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 150.1, 133.4, 124.5, 118.4, 116.7 ppm; MS (ESI) m/z = 148 (53, M^+), 118 (11), 102 (100), 90 (29), 75 (42), 51 (25).

4.13 1-Naphthonitrile (**12a**) [35]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.32–8.24 (m, 7H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 133.4, 132.9, 132.6, 132.3, 129.0, 128.7, 128.6, 125.3, 124.9, 117.8, 109.9 ppm; MS (ESI) m/z = 153 (100, M^+), 126 (28), 63 (11).

4.14 Nicotinonitrile (**13a**) [36]

^1H NMR (CDCl_3 , 200 MHz) δ = 7.35–8.83 (m, 4H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) δ = 152.9, 152.3, 139.4, 123.8, 116.5, 110.2 ppm; MS (ESI) m/z = 104 (100, M^+), 77 (58), 50 (24).

4.15 Cinnamoylnitrile (**14a**) [33]

^1H NMR (CDCl_3 , 200 MHz) $\delta = 7.35\text{--}7.48$ (m, 6H), 5.78–5.92 (m, 1H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) $\delta = 150.7$, 133.5, 131.3, 129.2, 127.4, 118.2, 96.2 ppm; MS (ESI) $m/z = 129$ (100, M^+), 102 (44), 76 (12), 51 (18).

4.16 Benzyl Cyanide (**15a**) [31]

^1H NMR (CDCl_3 , 200 MHz) $\delta = 7.42\text{--}7.17$ (m, 5H), 3.71 (s, 2H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) $\delta = 129.4$, 129.1, 128.0, 127.9, 23.6 ppm; MS (ESI) $m/z = 117$ (100, M^+), 90 (62), 63 (13), 51 (14).

4.17 1-Adamantyl nitrile (**16a**) [26]

^1H NMR (CDCl_3 , 200 MHz) $\delta = 2.05\text{--}1.57$ (m, 15H) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) $\delta = 126.2$, 39.9, 35.7, 27.1, 26.3 ppm; MS (ESI) $m/z = 161$ (44, M^+), 146 (20), 134 (100), 119 (12), 104 (16), 93 (53), 79 (25), 69 (23).

4.18 Synthesis of the Bissilylated Compound **24**

The protocol has been carried out in accordance to the work of Nagashima and co-workers [16]. 4-Fluorobenzoyl chloride (**23**, 12.0 mmol) was added to a solution of lithium bis(trimethylsilyl)amide (12.0 mmol) in *n*-hexane (20 mL) at -78 °C. After complete addition the mixture was slowly warmed to room temperature and stirring was continued for 24 h. The mixture was filtered and the solvent was removed to yield yellow oil, which was used without further purifications. Yield: 87%; ^1H NMR (CDCl_3 , 200 MHz) $\delta = 0.12$ (s, 9H, SiMe_3), 0.27 (s, 9H, SiMe_3), 6.94–7.07 (m, 2H, Ar), 7.51–7.64 (m, 2H, Ar) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) $\delta = 171.0$, 168.3, 161.3, 155.1, 134.8, 134.6, 132.9, 130.6, 130.4, 129.8, 129.6, 129.0, 115.7, 115.2, 114.5, 1.9, 1.3, 0.3, -0.7 ppm; ^{19}F NMR (CDCl_3 , 188 MHz) $\delta = -110.7$ (m) ppm.

4.19 Synthesis of the Monosilylated Compound **25**

The protocol has been carried out in accordance to literature [37]. To a solution of **1** (22.0 mmol) and triethylamine (22.0 mmol) in toluene (20 mL) was added trimethylsilyl chloride (22.0 mmol) at room temperature under an atmosphere of dinitrogen. The mixture was refluxed for 24 h. The solvent was removed in vacuum and the residue was extracted with *n*-hexane to yield the monosilylated compound **25**. Yield: 63%; ^1H NMR (CDCl_3 , 200 MHz) $\delta = 0.30$ (s, 9H, SiMe_3), 6.97–7.14 (m, 2H, Ar), 7.72–7.86 (m, 2H, Ar) ppm; ^{13}C NMR (CDCl_3 , 50 MHz) $\delta = 171.2$, 168.4, 167.3, 162.4, 162.3, 131.3, 131.2, 130.0, 129.9,

129.8, 129.6, 115.7, 115.6, 115.2, 115.1, 1.9, -0.7 ppm; ^{19}F NMR (CDCl_3 , 188 MHz) $\delta = -108.2$ (m) ppm.

4.20 General Procedure for the Dehydration of Amides

A pressure tube was charged with an appropriate amount of copper(I) chloride (0.018 mmol, 2.5 mol%), and the corresponding amide (0.72 mmol). After addition of toluene (2.0 mL) 2.0 equivalents of MSTFA (1.44 mmol) were added and the reaction mixture was stirred in a preheated oil bath at 100 °C for 24 h. The mixture was cooled to room temperature and an aliquot was taken for GC-analysis (30 m Rxi-5 ms column, 40–300 °C). The reaction mixture was dissolved in diethyl ether and purified by column chromatography after that the solvents were removed under reduced pressure. The analytical properties of the corresponding nitriles are in agreement with literature.

Acknowledgment Financial support from the Cluster of Excellence “Unifying Concepts in Catalysis” (funded by the Deutsche Forschungsgemeinschaft and administered by the Technische Universität Berlin) is gratefully acknowledged.

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