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An Efficient Zinc-Catalyzed Dehydration of Primary Amides to Nitriles

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Abstract: In the present study, the zinc-catalyzed dehydration of a variety of amides with *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (MSTFA) as a dehydration reagent into the corresponding nitriles has been examined in detail. With the straightforward and commercially available zinc(II)triflate as the precatalyst and MSTFA, an excellent system has been established to afford nitriles in excellent yields and chemoselectivities. After investigation of reaction conditions and the scope and limitations, several efforts were carried out to understand the reaction mechanism.

Keywords: amides · dehydration · homogeneous catalysis · nitriles · zinc

Introduction

Nitrile functionalities are extensively applied in organic chemistry and represent building blocks for the production of pharmaceuticals, agrochemicals, and polymers.[1] Within the various protocols to synthesize nitriles, one convenient and classical way to access nitriles is the dehydration of primary amides. Extensive application was achieved for dehydration with stoichiometric acidic (e.g., $POCl_3$, P_2O_5, $SOCl_2$, $TiCl_4$) as well as basic reagents (e.g., $NaBH_4$) or a combination of reagents.[2] The development of sustainable and selective protocols is one of the fundamental research goals in modern chemistry.[3] Based on these requirements more recently the application of transition metal catalysts became an efficient and flexible alternative.[4] Dehydration in the presence of catalytic amounts of metal precursors (e.g., Ru, Pd, Rh, W, V, Re, U)[5–11] and, for example, silanes as dehydration reagents has been reported, while in the absence of the dehydration reagent high reaction temperatures are required.[12] Primary benzamides were tretaed with $R_3Si{-}H$ to produce the corresponding nitrile, hydrogen, and siloxanes. Nevertheless, present research is focused on the replacement of these reagents with cheaper and less-toxic metals.[13] Currently, the application of more environmental friendly metals (e.g., Fe, Zn) in catalysis has been extensively studied.[14–16] Indeed, cheap, abundant, and non-toxic metals such as iron have also been probed for the dehydration of amides, while the application of zinc has been scarcely reported.[17,18] Herein, we demonstrate the usefulness of an easy-to-adopt zinc catalyst for the dehydration of primary amides by applying *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (MSTFA) as the dehydration reagent.

Results and Discussion

Initially, the dehydration of benzamide (**1**) with *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (**2**) in toluene was studied as a model reaction to evaluate suitable reaction conditions and to study the influence of various reaction parameters (Table 1). As expected when using MSTFA in the absence of any metal source no formation of benzonitrile (**1a**) was observed, while significant amounts of the monosilylated benzamide was detected by GC-MS (Table 1, entry 1). In contrast, in the presence of 5.0 mol% of commercially available $Zn(OTf)_2$ and MSTFA (3.5 equiv) excellent yield (> 99%) and chemoselectivity (>99%) were realized under ambient conditions (Table 1, entry 2). Additionally, the effect of various zinc sources such as ZnF_2, $ZnCl_2$, $Zn(OAc)_2$, $Zn(acac)_2$, and $Zn(ClO_4)_2$ on the benzonitrile formation was investigated (Table 1, entries 3–7). All zinc sources led to a quantitative conversion of benzamide to yield benzonitrile, whereas $Zn(OAc)_2$ gave a decreased yield of 94%. Besides toluene as a solvent dioxane, dichloromethane, and tetrahydrofuran (THF) have been applied. Excellent performance was found for THF with a yield of >99% (Table 1, entry 8). Noteworthy, the reaction temperature was decreased to 70 °C. A further decrease to room temperature resulted in a diminished yield (Table 1, entry 9). The loading of $Zn(OTf)_2$ was decreased to 1.0 mol%, thereby resulting again in excellent yields of **1** (Table 1, entry 10). Moreover, the amount of MSTFA was studied and demonstrated the need for more than three equivalents (Table 1, entries 10–12).[19]

To explore the scope and limitations of the presented zinc catalyst, the dehydration of a variety of primary amides was performed (Table 2). The reaction conditions are in accord-

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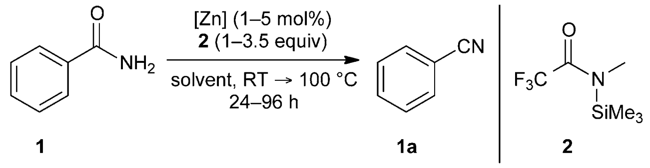
Table 1. Zinc-catalyzed dehydration of benzamide **1**.



Entry[a]	Zinc source [mol %]	MSTFA [equiv]	t [h]	T [°C]	Solvent	Yield [%][b]
1	–	3.5	24	100	toluene	<1
2	$Zn(OTf)_2$ (5)	3.5	24	100	toluene	>99
3	$Zn(OAc)_2$ (5)	3.5	24	100	toluene	94
4	ZnF_2 (5)	3.5	24	100	toluene	>99
5	$ZnCl_2$ (5)	3.5	24	100	toluene	>99
6	$Zn(acac)_2$ (5)	3.5	24	100	toluene	>99
7	$Zn(ClO_4)_2$ (5)	3.5	24	100	toluene	>99
8	$Zn(OTf)_2$ (5)	3.5	24	70	THF	>99
9	$Zn(OTf)_2$ (5)	3.5	96	r.t.	THF	<5
10	$Zn(OTf)_2$ (1)	3.5	24	70	THF	>99
11	$Zn(OTf)_2$ (5)	2.0	24	70	THF	<1
12	$Zn(OTf)_2$ (5)	1.0	24	70	THF	<1

[a] Reaction conditions: **1** (0.72 mmol), zinc source (1–5 mol %), MSTFA (1.0–3.5 equiv), solvent (2.0 mL), RT→100 °C, 24–96 h. [b] Determined by GC methods using biphenyl as an internal standard.

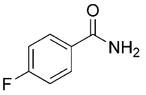
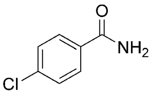
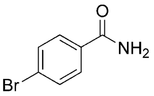
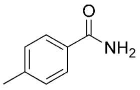
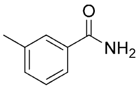
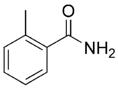
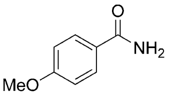
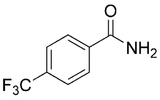
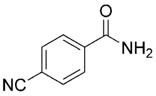
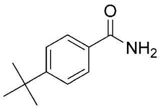
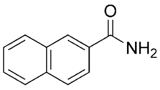
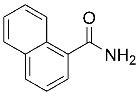
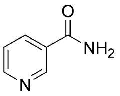
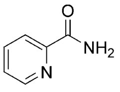
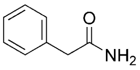
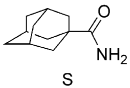
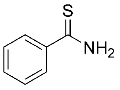
ance to optimized conditions discovered in Table 1; this means the corresponding amides were converted in the presence of 5.0 mol % $Zn(OTf)_2$ and 3.5 equivalents of MSTFA in THF at 70 °C for 24 hours.[20] Firstly, various substituted benzamides were tested. In all cases excellent yields were observed. No significant effect was detected for electron-withdrawing as well as electron-donating groups (Table 2, entries 1–10). Only in the case of strong electron-withdrawing functionalities such as *para*-trifluoromethylbenzamide (**10**) and *para*-nitrilobenzamide (**11**) moderate yields were obtained (Table 2, entries 8–9). Moreover, naphthylamides were dehydrated with excellent yields and selectivities, while for nitrogen-based heteroaromatics the catalyst was completely inactive (Table 2, entries 11–14). Besides, alkyl amides were reacted under optimized conditions, resulting in excellent yields of the corresponding nitriles (Table 2, entries 15–16). Furthermore the catalyst showed high efficiency in the dehydration of thiobenzamide (**19**) (Table 2, entry 17).

Abstract in German: Im Rahmen dieser Arbeit wird die Zink-katalysierte Dehydration von primären Amiden zu den entsprechenden Nitrilen mit Hilfe von *N*-Methyl-*N*-(trimethylsilyl)trifluoroacetamid (MSTFA) vorgestellt. Hierbei konnten exzellente Ausbeuten und Selektivitäten (>99 %) durch Anwendung von einfachen und kommerziell verfügbaren Zinksalzen beobachtet werden. Nach eingehender Untersuchung verschiedenster Reaktionsparameter wurden die hervorragenden Eigenschaften des Katalysatorsystems in der Dehydration zahlreicher primärer Amide gezeigt. Zum tieferen Verständnis der Reaktion wurden verschiedene mechanistische Experimente durchgeführt.

Having found a suitable system for the dehydration of amides into the corresponding nitrile functionality, we became interested in the evaluation of the reaction mechanism. Two different reaction pathways can be considered (Scheme 1). The first one is the "bissilylation" pathway, which has been proposed recently for the application of Si−H compounds.[5a,17] In accordance to that the primary amide can be activated by the Lewis acidic $Zn(OTf)_2$ and allow therefore the attack of the silylation reagent MSTFA and the transfer of $SiMe_3$ to the amide functionality. This process takes place two times, and results in the formation of *N*-methyltrifluoroacetamide (**21**) and the double silylated intermediate **23**. As reported, this type of compound can easily rearrange into the *N*,*O*-bis(trimethylsilyl)imidate.[21] The zinc catalyst activates this intermediate and forces the abstraction of the siloxane **25** to yield the desired nitrile and provides the free $Zn(OTf)_2$, which can then react again. The second is the "monosilylation" pathway as proposed in Scheme 1. In agreement to the bissilylation process the amide will be activated by the zinc and thus allow the transfer of the $SiMe_3$ group to the amide functionality to form the monosilylated amide **22**. This silylated amide can undergo a rearrangement into the *O*-(trimethylsilyl)imidate **26**, which after activation by the zinc catalyst eliminates the desired nitrile and trimethylsilanol (**27**). The trimethylsilanol can react with an excess of MSTFA to form the siloxane **25** and **21**.

To elucidate the underlying mechanism of the dehydration, various experiments have been carried out. For preliminary mechanistic investigations the dehydration in the absence of MSTFA was studied. As a model substrate *para*-fluorobenzamide (**3**) was employed, because it is an excellent probe for ^{19}F NMR spectroscopy measurements. The role of the MSTFA was clarified by dissolving substrate **3** and catalytic amounts of $Zn(OTf)_2$ (10 mol %) in $[D_8]THF$. At room temperature as well as after heating for 24 hours no product formation was observed, while the signals for compound **3** ($\delta = -111.0$ ppm, sept, $J = 4.69$ Hz) and $Zn(OTf)_2$ ($\delta = -79.3$ ppm) are unchanged. The signal for the $Zn(OTf)_2$ is slightly shifted ($\delta = -79.1$ ppm). To clarify the function of the zinc precursor, MSTFA and **3** were mixed in $[D_8]THF$ in the absence of zinc, but no product formation was detected, which clearly indicates that the zinc acts as a catalyst. In addition, even after heating to 70 °C for 24 hours no monosilylation or bissilylation of **3** was observed, which is in contrast to substrate **1** (vide supra). This fact probably points to a dual function of zinc, on the one hand it acts as a silylation catalyst and on the other hand as an activator for the elimination step. Obviously, for dehydration of primary amides all three compounds are necessary. After addition of MSTFA to this mixture three new peaks appeared, which could be attributed to MSTFA and the hydrolyzed product **21**. After heating for several hours, a ^{19}F NMR spectrum was recorded, which revealed the formation of *para*-fluoro-

Table 2. Scope and limitations of the zinc-catalyzed dehydration of primary amides.

Entry[a]	Substrate		Yield [%][b]
1		**3**	**3a**: >99 (94)
2		**4**	**4a**: >99 (94)
3		**5**	**5a**: 97 (91)
4		**6**	**6a**: >99 (93)
5		**7**	**7a**: >99 (90)
6		**8**	**8a**: >99 (90)
7		**9**	**9a**: >99 (95)
8		**10**	**10a**: 63
9		**11**	**11a**: 47
10		**12**	**12a**: >99 (92)
11		**13**	**13a**: >99 (93)
12		**14**	**14a**: >99 (92)
13		**15**	**15a**: <1
14		**16**	**16a**: <1
15		**17**	**17a**: >99 (87)
16		**18**	**18a**: >99 (91)
17		**19**	**1a**: >99[c]

[a] Reaction conditions: amide (1.0 mmol), Zn(OTf)$_2$ (5 mol %), MSTFA (3.5 equiv), THF (2.0 mL), 70 °C, 24 h. [b] Determined by GC methods and ^{1}H NMR spectroscopy. The yield of isolated product is in brackets. [c] Determined by GC methods using biphenyl as an internal standard.

benzonitrile (**3a**, $\delta = -104.9$ ppm, sept, $J = 4.56$ Hz). Noteworthy, the signal for Zn(OTf)$_2$ was unchanged and no monosilylated or bissilylated products of **3** were observed on the NMR time scale.

In addition, the ^{29}Si NMR spectrum revealed the formation of Me$_3$SiOSiMe$_3$ (**25**, $\delta = 7.1$ ppm), while the signals for MSTFA (**2**, $\delta = 25.6$ ppm) disappeared.[22] To distinguish between the mono- and bissilylation pathway the monosilylated compound **30** and the bissilylated compound **29** were prepared (Scheme 2). Compound **29** was synthesized according to the procedure reported by Nagashima and co-workers.[5] The benzoyl chloride **28** was treated with lithium hexamethyldisilazane (LiHMDS) at low temperature to yield compound **29**, which was later on heated in [D$_8$]THF in the presence of catalytic amounts of Zn(OTf)$_2$ to obtain **3a** with a yield of 49 % after six hours. However, the monosilylated benzamide was accessible by reaction of benzamide **3** with chlorotrimethylsilane in the presence of triethylamine. In contrast to **29** no formation of **3a** was observed.

Based on these results the reaction mechanism by the bissilylation pathway is more likely. To prove these assumptions, theoretical investigations were carried out. Quantum chemical calculations using density functional theory at the B3LYP/6-31G(d) level were performed.[23] First the starting material MSTFA was examined, as different states are assumed, for example, *N*- and *O*-silylation. Two isomers of the *N*-silylated product are possible; these isomers are the trimethylsilyl group *E*- or *Z*-positioned to the oxygen (Figure 1, **2** and **2a**). As shown in Figure 1, the rotation energy of the C–N(SiMe$_3$)(Me) bond in MSTFA (**2**) is 13.8 kcal mol^{-1} and a lower energy for **2a** was observed. Furthermore, we assume for the transfer of the silyl group from the nitrogen to the oxygen an intramolecular process through a 4-membered transition state **2a**-**TS** (Figure 1). The transition state **2a**-**TS** was found in this pathway with a barrier of 16.9 kcal mol^{-1}. The *O*-silylated compound **2c** was comparable in energy with **2**. By judging from the relative energy of **2**-**TS** and **2a**-**TS**, it is reasonable to expect that the reaction occurs at room temperature. Due to the attached *N*-methyl group in MSTFA, an internal elimination of the trimethylsilyl group is hampered. Based on this fact and in cooperation with activation by the CF$_3$ functionality, the silyl group is transferred into a primary amide. As a model for primary amides acetamide was chosen, because of simplicity. Initially the bissilylation was studied. In Figure 2, the pathway for the formation of acetonitrile (**33**) and

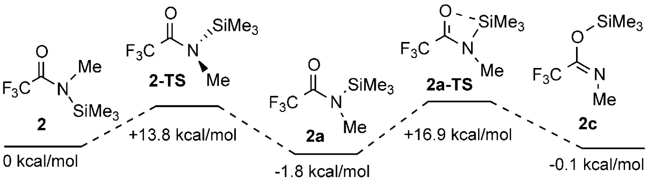
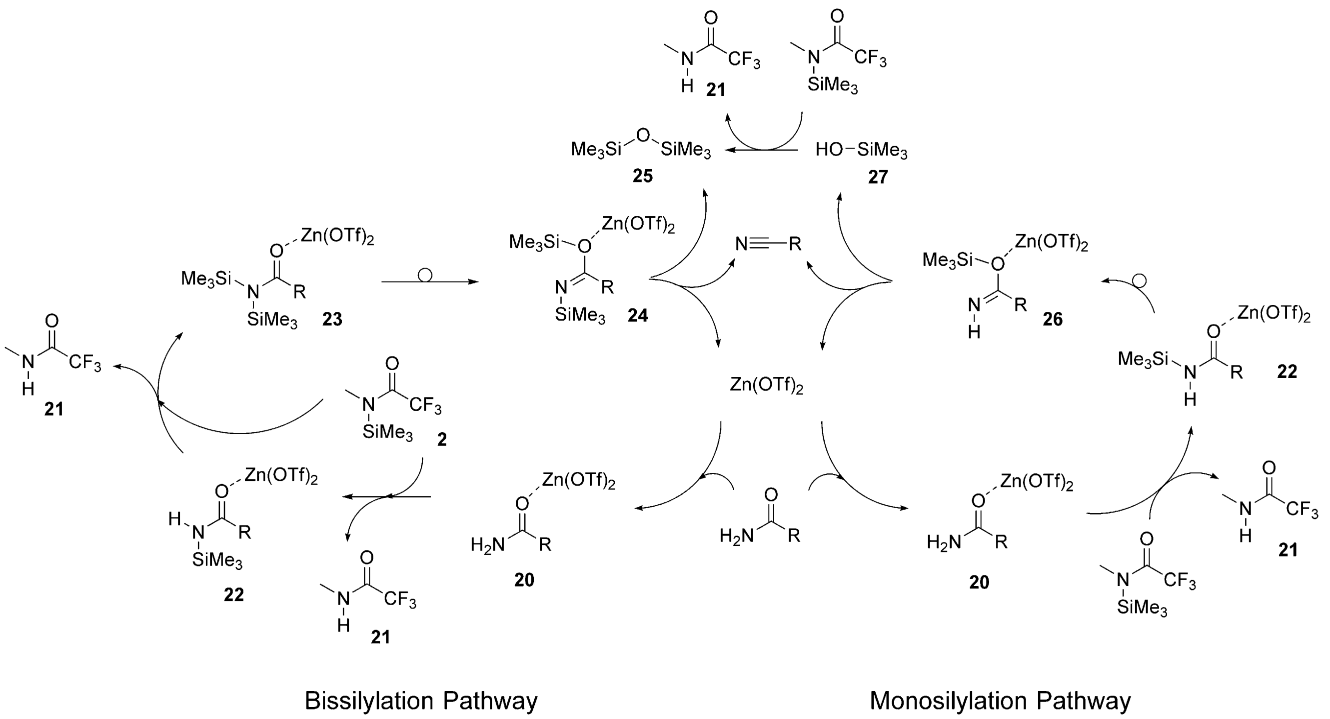
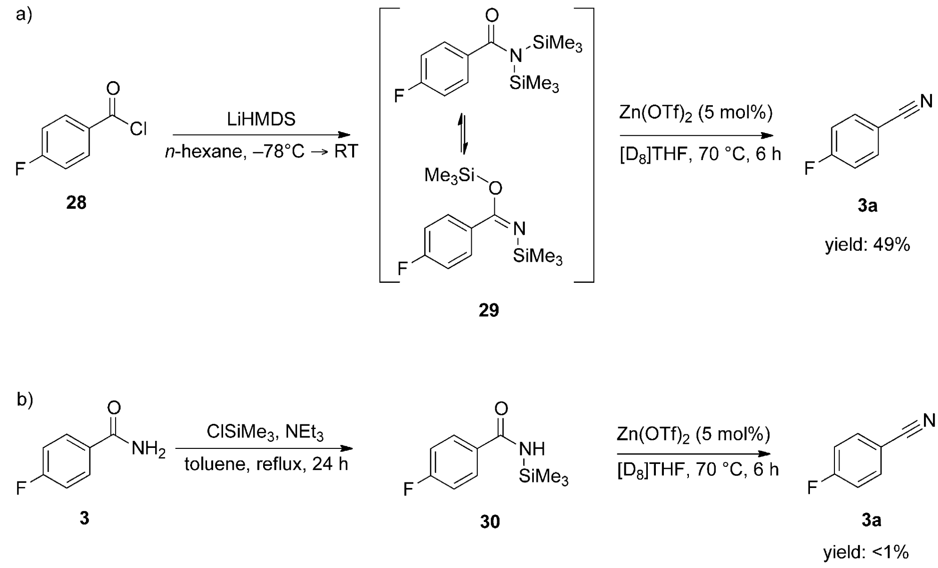


Figure 1. *N*-methyl-*N*-(trimethylsilyl)trifluoroacetamide (**2**) versus *N*-methyl-*O*-(trimethylsilyl)trifluoroacetoimidate (**2c**).



Scheme 1. Mechanistic proposal for the dehydration of amides.



Scheme 2. Synthesis of mono- and bissilylated benzamide **29** and **30**.

$Me_3SiOSiMe_3$ (**25**) from the bissilylated compound **30** are presented with the energy profile.

First a rearrangement takes place via the 4-membered transition state **31-TS** to form the more stable *N*,*O*-bis(trimethylsilyl)imidate **32** with a barrier of 10.1 kcal mol^{-1}. Next, a concerted bond cleavage of the C–O and N–Si bonds of **32** proceeds via transition state **32-TS**, with a barrier of 54.6 kcal mol^{-1}, to afford **25** and **33**. This barrier can probably be influenced by the catalyst. The overall pathway is exothermic by 12.3 kcal mol^{-1}.

The monosilylation pathway was investigated by theoretical methods. A schematic potential-energy surface for the formation of trimethylsilylanol **27** and acetonitrile **33** from the monosilylated compound **34** is given in Figure 3. This pathway includes the rearrangement of **34** into **35** via transition state **34-TS**, formation of *O*-silylated species **36** via transition state **35-TS**, and cleavage of the Si–O and N–H bonds of **36** to give **33** and **27**. Interestingly, this process is endothermic by 14.3 kcal mol^{-1}. This is contrary to the bissilylated pathway (vide supra). Accordingly, we have assumed that the bissilylated pathway (Figure 2) is more favorable than the monosilylated pathyway (Figure 3).

Conclusions

In summary, we have developed an efficient method for the dehydration of primary amides into the corresponding ni-

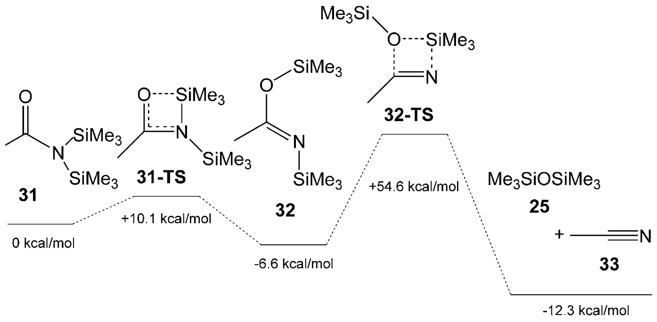


Figure 2. Mechanistic proposal for the dehydration of amides—bissilyation pathway.

triles with MSTFA as the silylation reagent in the presence of simple zinc salts under mild reaction conditions. Furthermore, mechanistic investigations indicated, that zinc(II) triflate acts as a Lewis acid catalyst.

Experimental Section

General

All compounds were used as received without further purification. THF and toluene were dried by applying standard procedures. ^{1}H, ^{19}F, and ^{13}C NMR spectra were recorded on a Bruker AFM 200 spectrometer (^{1}H: 200.13 MHz; ^{13}C: 50.32 MHz; ^{19}F: 188.31 MHz) using the proton signals of the deuterated solvents as a 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.

General Procedure for the Dehydration of Amides

A pressure tube was charged with an appropriate amount of $Zn(OTf)_2$ (0.05 mmol, 5.0 mol %), the corresponding amide (1.0 mmol), and MSTFA (3.5 equivalents, 3.5 mmol). After addition of THF (2.0 mL), the reaction mixture was stirred in a preheated oil bath at 70 °C for 24 h. The mixture was cooled in an ice bath and biphenyl (internal standard) was added. The solution was diluted with dichloromethane and an aliquot was taken for GC-analysis (30 m Rxi-5 ms column, 40–300 °C). The solvent was carefully removed and the residue was purified by column chromatography (*n*-hexane/ethyl acetate 5:1). The analytical properties of the corresponding nitriles are in agreement with literature.[24,25]

*4-Fluorobenzonitrile (**3a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.57$–7.67 (m, 2H), 7.05–7.18 ppm (m, 2H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 167.6$, 162.5, 134.8, 134.6, 118.0, 117.1, 116.6, 108.6, 108.5 ppm; MS (EI) $m/z = 121$ (100, M^+), 94 (39); t(GC) = 5.192 min.

*4-Chlorobenzonitrile (**4a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.39$–7.59 ppm (m, 4H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 139.7$, 133.4, 129.7, 118.0, 110.7 ppm; MS (EI) $m/z = 137$ (100, M^+), 102 (38), 75 (17), 50 (15); t(GC) = 8.142 min.

*4-Bromobenzonitrile (**5a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.45$–7.64 ppm (m, 4H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 133.4$, 132.7, 128.1, 118.1, 111.2 ppm; MS (EI) $m/z = 181$ (60, M^+), 102 (100), 75 (31), 50 (24); t(GC) = 9.583 min.

*4-Methylbenzonitrile (**6a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.47$–7.55 (m, 2H), 7.19–7.28 (m, 2H), 2.39 ppm (s, 3H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 143.7$, 132.1, 129.8, 119.2, 109.8, 21.8 ppm; MS (EI) $m/z = 117$ (100, M^+), 90 (47), 63 (14); t(GC) = 7.467 min.

*3-Methylbenzonitrile (**7a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.23$–7.43 (m, 4H), 2.34 ppm (s, 3H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 139.3$, 133.7, 132.4, 132.1, 129.2, 119.0, 112.2, 21.1 ppm; MS (EI) $m/z = 117$ (100, M^+), 90 (50); t(GC) = 7.250 min.

*2-Methylbenzonitrile (**8a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.39$–7.57 (m, 2H), 7.15–7.33 (m, 2H), 2.50 ppm (s, 3H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 141.9$, 132.7, 132.5, 130.2, 126.2, 118.1, 112.8, 20.4 ppm; MS (EI) $m/z = 117$ (100, M^+), 90 (56), 63 (14); t(GC) = 6.842 min.

*4-Methoxybenzonitrile (**9a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.50$–7.58 (m, 2H), 6.86–6.95 (m, 2H), 3.81 ppm (s, 3H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 162.8$, 133.9, 119.1, 114.7, 103.8, 55.5 ppm; MS (EI) $m/z = 133$ (100, M^+), 103 (46), 90 (47), 76 (12), 63 (17); t(GC) = 10.150 min.

*4-Trifluoromethylbenzonitrile (**10a**)*

^{1}H NMR (CDCl$_3$, 200 MHz): $\delta = 7.68$–7.82 ppm (m, 4H); ^{13}C NMR (CDCl$_3$, 50 MHz): $\delta = 135.5$, 134.9, 134.2, 133.5, 133.3, 132.7, 131.2, 126.29, 126.22, 126.14, 126.07, 125.8, 120.4, 117.4, 116.11, 116.08, 114.9 ppm; ^{19}F NMR (CDCl$_3$, 188 MHz) $\delta = -63.6$ ppm; MS (ESI) $m/z = 171$ (100, M^+), 152 (40), 121 (70), 75 (20), 50 (11); t(GC) = 5.008 min.

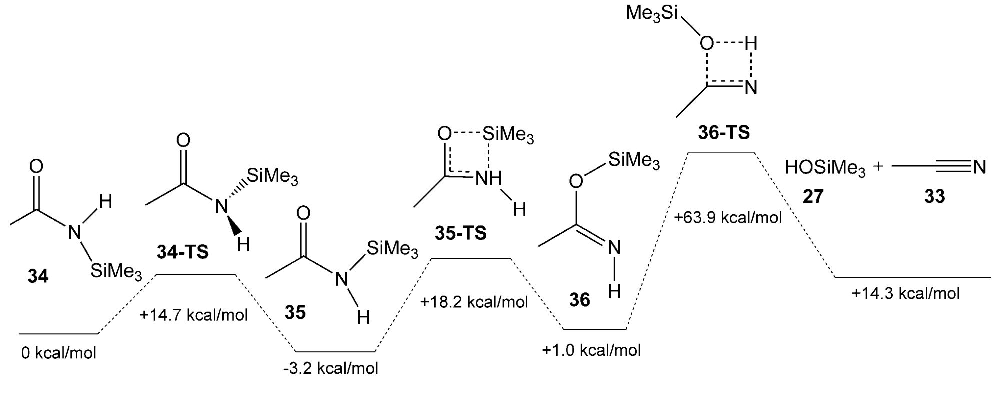


Figure 3. Potential-energy surface of the proposed pathway for the formation of trimethylsilylanol **27** and acetonitrile **33** from *N*-trimethylsilyl acetamide **34**—monosilyation pathway.

*4-Benzodinitrile (**11 a**)*

(EI) $m/z = 128$ (100, M^+), 101 (27), 75 (12), 50 (11); t(GC) = 10.083 min.

*4-tert-Butylbenzonitrile (**12 a**)*

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 7.38$–7.56 (m, 4H), 1.26 ppm (s, 9H); ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = 156.8$, 131.9, 126.2, 119.2, 109.1, 35.2, 30.8 ppm; MS (EI) $m/z = 159$ (21, M^+), 144 (100), 116 (71), 104 (15); t(GC) = 10.950 min.

*Naphthalene-2-carbonitrile (**13 a**)*

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 7.38$–8.30 ppm (m, 7H); ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = 133.3$, 132.9, 132.6, 128.7, 128.6, 127.6, 125.1, 124.9, 117.8, 110.2 ppm; MS (EI) $m/z = 153$ (100, M^+), 126 (25); t(GC) = 13.533 min.

*Naphthalene-1-carbonitrile (**14 a**)*

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 7.47$–8.25 ppm (m, 7H); ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = 133.4$, 133.0, 132.7, 132.5, 128.9, 128.7, 128.6, 125.4, 124.9, 117.9, 110.3 ppm; MS (EI) $m/z = 153$ (100, M^+), 126 (28), 63 (11); t(GC) = 13.175 min.

*Benzyl cyanide (**15 a**)*

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 7.31$–7.42 (m, 5H), 3.83 ppm (s, 2H, CH_2); ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = 130.1$, 129.2, 127.8, 127.7, 23.7 ppm; MS (EI) $m/z = 117$ (100, M^+), 90 (62), 63 (13), 51/14); t(GC) = 8.17083 min.

*1-Adamantylnitrile (**16 a**)*

^{1}H NMR ($CDCl_3$, 200 MHz): $\delta = 1.50$–2.10 ppm (m, 15H); ^{13}C NMR ($CDCl_3$, 50 MHz): $\delta = 125.2$,39.9, 35.7, 27.1, 26.5 ppm; MS (ESI) $m/z = 161$ (44, M^+), 146 (20), 134 (100), 119 (12), 104 (16), 93 (53), 79 (25), 69 (23); t(GC) = 12.117 min.

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