Preparation of N,N,N'-tris(trimethylsilyl)amidines; a convenient route to unsubstituted amidines

René T. Boeré, Richard T. Oakley and Robert W. Reed

Guelph-Waterloo Centre for Graduate Work in Chemistry, Department of Chemistry and Biochemistry, University of Guelph, Guelph, Ontario N1G 2W1 (Canada)
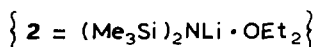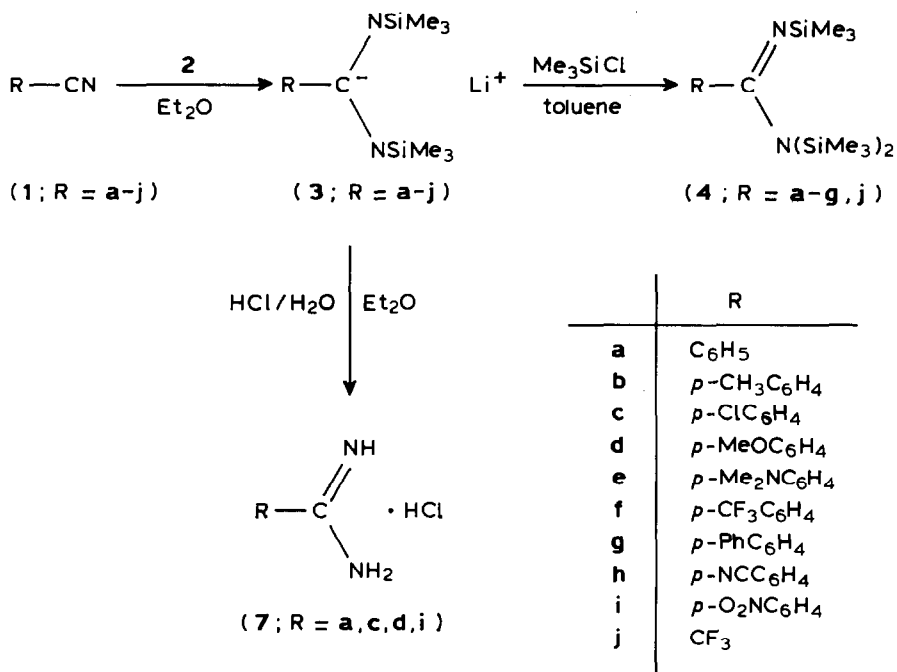
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Abstract

The tris(trimethylsilyl)amidines $RC(NSiMe_3)N(SiMe_3)_2$ ($R = C_6H_5$, p-$CH_3C_6H_4$, p-ClC_6H_4, p-$MeOC_6H_4$, p-$Me_2NC_6H_4$, p-$CF_3C_6H_4$, p-$C_6H_5C_6H_4$ and CF_3) are prepared by the reaction of the respective nitriles with $(Me_3Si)_2NLi \cdot OEt_2$ in ether to give intermediates $RC(NLi)N(SiMe_3)_2$. Heating these intermediates with $ClSiMe_3$ in toluene affords the products, which are isolated by vacuum distillation, in high yield. With 1,4-dicyanobenzene, two equivalents of reagents affords the per(trimethylsilyl)-1,4-diamidine. Hydrolysis of the intermediates with $6N$ ethanolic HCl affords the unsubstituted amidine hydrochlorides $RC(NH)NH_2 \cdot HCl$ ($R = C_6H_5$, p-$MeOC_6H_4$, p-ClC_6H_4, p-$O_2NC_6H_4$) in high yield.

Introduction

We have recently reported the novel application of benzamidine [1] as well as its tris(trimethylsilyl) analogue [2] in the preparation in high yield of heterocyclic thiazenes incorporating an RC(N–)N$\langle$ moiety. In the course of extending this work we have prepared a number of new tris(trimethylsilyl) amidines, and have used a similar route to obtain some desired unsubstituted amidines, which can be very difficult to prepare by the literature methods. We now report the results of these investigations.

Results and discussion

In a communication published 15 years ago [3], Sanger reported that the reaction of lithium bis(trimethylsilyl)amide with benzonitrile (**1a**) in ether allowed the isolation of a lithiated benzamidine derivative (**3a**) whereas the reaction in petroleum gave only *sym*-2,4,6-triphenyltriazine. The base-catalysed trimerization of nitriles can thus be avoided by using ether as solvent. The reaction of **3a** with

$$R\text{---}CN \xrightarrow[\text{Et}_2\text{O}]{2} R\text{---}\overset{\overset{\displaystyle NSiMe_3}{\diagup}}{\underset{\underset{\displaystyle NSiMe_3}{\diagdown}}{C^-}} Li^+ \xrightarrow[\text{toluene}]{Me_3SiCl} R\text{---}\overset{\overset{\displaystyle NSiMe_3}{/\!/}}{\underset{\underset{\displaystyle N(SiMe_3)_2}{\diagdown}}{C}}$$

(1 ; R = a–j)　　　　　(3 ; R = a–j)　　　　　　　(4 ; R = a–g , j)

HCl/H₂O | Et₂O

$$R\text{---}\overset{\overset{\displaystyle NH}{/\!/}}{\underset{\underset{\displaystyle NH_2}{\diagdown}}{C}} \cdot HCl$$

(7 ; R = a,c,d,i)

	R
a	C_6H_5
b	$p\text{-}CH_3C_6H_4$
c	$p\text{-}ClC_6H_4$
d	$p\text{-}MeOC_6H_4$
e	$p\text{-}Me_2NC_6H_4$
f	$p\text{-}CF_3C_6H_4$
g	$p\text{-}PhC_6H_4$
h	$p\text{-}NCC_6H_4$
i	$p\text{-}O_2NC_6H_4$
j	CF_3

$\{\, 2 = (Me_3Si)_2NLi \cdot OEt_2 \,\}$

Scheme 1

Me₃SiCl afforded tris(trimethylsilyl)benzamidine (**4a**) [3]. Full experimental details were not reported, and we found that this last step is not complete in ether at 25°C after 18 h; work-up of the reaction mixture afforded **3a**, identified by ¹H NMR. However the reaction could be driven to completion by replacing most of the ether with toluene at this stage of the reaction, and refluxing the reaction mixture for 5–6 h (¹H NMR could be used to monitor the progress of the reaction, see Table 1).

Essentially the same synthetic procedure has now been applied in the preparation of a range of ring-substituted tris(trimethylsilyl)benzamidine derivatives from the respective nitriles, as well as N,N,N'-tris(trimethylsilyl)-1,1,1-trifluoroacetamidine (**4j**) from CF₃CN. The synthetic route, which is outlined in Scheme 1, is applicable to any nitrile lacking an α-hydrogen atom. The presence of an α-hydrogen atom leads via a ketimine-enamine tautomerism to a carbanion of the form [4]:

$$R'\text{---}CH(Li)\text{---}\overset{\overset{\displaystyle NH}{/\!/}}{\underset{\underset{\displaystyle N(SiMe_3)_2}{\diagdown}}{C}}$$
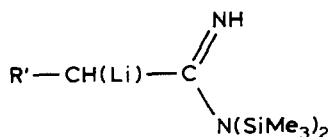
The reaction of trichloroacetonitrile fails for a related reason, the elimination of LiCl from the intermediate.

The preparations are best done on a large scale, and in order to maintain reasonable solvent volumes, we have found the use of the crystalline ether adduct of

Table 1

NMR data (δ in ppm; J in Hz)

R =		3[a] δ(SiMe$_3$)	4[b] δ(SiMe$_3$)	δ(substit.)	δ(A)[c]	δ(B)	J(AB)
C_6H_5	(a)	−0.31	0.02	s 7.23	−	−	−
p-CH$_3$C$_6$H$_4$	(b)	−0.33	0.09	2.37	7.08	7.23	7.94
p-ClC$_6$H$_4$	(c)	−0.32	0.07	−	7.22	7.29	8.23
p-MeOC$_6$H$_4$	(d)	−0.30	0.09	3.85	6.64	7.45	8.65
p-Me$_2$NC$_6$H$_4$	(e)	−	0.09	2.99	6.31	7.48	8.75
p-CF$_3$C$_6$H$_4$	(f)	−0.33	0.11	−63.09[d]	7.36	7.68	8.15
p-PhC$_6$H$_4$	(g)	−0.28	0.14	(7.4–7.7)	7.32	7.67	8.04
p-NCC$_6$H$_4$	(h)	−0.31[e]	−	−	−	−	−
p-O$_2$NC$_6$H$_4$	(i)	−0.33	−	−	−	−	−
CF$_3$	(j)	−	0.20	−75.62[d,f]	−	−	−

[a] In neat $(C_2H_5)_2O$, ref. to the methyl triplet at 1.12 ppm. [b] In CDCl$_3$ ref. to CH$_2$Cl$_2$ at 5.32 ppm. [c] C$_6$H$_4$ group gives rise to an AB doublet signal. [d] ^{19}F NMR; positive shift is to low field of external CFCl$_3$. [e] The diadduct has a singlet at −0.21 ppm. [f] For ^{13}C NMR, see Experimental.

lithium bis(trimethylsilyl)amide, $(Me_3Si)_2NLi \cdot OEt_2$ (2) particularly advantageous [5] (see Experimental). Silylated amidines 4 with the following substituents have been prepared: C$_6$H$_5$, p-CH$_3$C$_6$H$_4$, p-ClC$_6$H$_4$, p-CH$_3$OC$_6$H$_4$, p-(CH$_3$)$_2$NC$_6$H$_4$, p-CF$_3$C$_6$H$_4$, p-C$_6$H$_5$C$_6$H$_4$ and CF$_3$. They may be purified by vacuum distillation, but with the exception of 4j, they solidify to waxy solids, although this may occur only after several days in a refrigerator. Melting points for the solid derivatives range from 25 to 78°C. All new compounds have been fully characterized by mass spectrometry, NMR and microanalysis. (See Table 1 and Experimental.) A precise analysis for 4g could not be obtained, due to persistent contamination by the nitrile. Attempts to further purify the compound by recrystallization resulted only in hydrolysis. The structure of 4j was confirmed by ^{13}C NMR, since it did not show a parent ion in the mass spectrum. The observation of two quartet patterns at 116.1 and 150.4 ppm, the former with a large coupling to ^{19}F, and the latter with a smaller coupling, along with signals due to the trimethylsilyl carbon atoms, is consistent with the formulation $CF_3C(NSiMe_3)N(SiMe_3)_2$.

For R = p-NCC$_6$H$_4$- and p-NO$_2$C$_6$H$_4$-, the formation of the intermediates 3h and 3i was quantitative by ^{1}H NMR; however the addition of Me$_3$SiCl to form 4h and 4i was not successful. We attribute this to extensive delocalization of the negative charge over the ring in the intermediates, which significantly lowers their nucleophilicity. During reflux of the intermediates with Me$_3$SiCl, ^{1}H NMR showed that the reaction was not progressing, and extensive decomposition also occurred. Although t-butylcyanide reacts with 2, the chemical shift of the product was quite different from that of all the others in Table 1. Furthermore, reaction with Me$_3$SiCl did not lead to a t-butyl derivative of 4. We were able to isolate a reactive, lithium-containing material by vacuum distillation, possibly an intermediate of type 3, but could not get a satisfactory analysis of this hydrolytically unstable species.

Further investigation of the tar obtained from the attempt to prepare the p-cyano substituted amidine indicated the presence of small quantities of both the 1/1 and 2/1 trisilylated amidines in the mixture, identified by mass spectroscopy. The addition of two equivalents of 2 to 1,4-dicyanobenzene proceeds smoothly to the

intermediate **5**, $\text{LiN}\{((\text{Me}_3\text{Si})_2\text{N}\}\text{CC}_6\text{H}_4\text{C}\{\text{N(SiMe}_3)_2\}\text{NLi}$. Subsequent reaction with an excess of Me_3SiCl provides $p\text{-C}_6\text{H}_4[\text{C}\{\text{NSiMe}_3\}\text{N(SiMe}_3)_2]_2$ **(6)** in reasonable yield. **6** sublimes in vacuo, forming large colourless blocks.

Hydrolysis of the persilylated amidines with ethanolic HCl leads cleanly to the respective unsubstituted amidines, some of which are difficult to prepare by the conventional routes. It is more convenient to hydrolyse the intermediates **3** directly using ethanolic HCl (see Scheme 1). The amidines are isolated as their hydrochlorides **(7)** by precipitation. This is a rapid and convenient synthesis of unsubstituted amidines derived from nitriles lacking an α-hydrogen or chlorine atom. Some of these have not been reported previously; others were made by diverse routes, which in our hands gave poor yields of the desired materials [6–12].

Conclusion

The reaction of lithium bis(trimethylsilyl)amide with nitriles provides a convenient and versatile route for the synthesis of a wide range of persilylated amidines with both electron-withdrawing and electron-donating substituents. Hydrolysis of the intermediates leads directly to the related amidine hydrochlorides.

Experimental

General. Solvents were rigorously dried before use; diethyl ether by distillation from lithium aluminum hydride and toluene by distillation from sodium. ^1H NMR spectra were recorded on Varian EM-360 and Bruker WH-400 instruments, ^{19}F on a Varian XL-300, IR on a Perkin–Elmer 1330 grating spectrometer as neat liquids or Nujol mulls, and mass spectra on a VG 7070 EF mass spectrometer at 70 eV by electron impact. Only the major fragments are reported, using the most abundant isotopes of each element. Chemical analyses were performed by MHW laboratories, Phoenix, Arizona. All procedures were conducted under an atmosphere of dry nitrogen. **2** was prepared directly from n-butyllithium and 1,1,1,3,3,3-hexamethyldisilazane by the method of Wannagat and Niederprum [5]; it crystallizes as large, colorless crystals after distillation of ca. one half the solvent volume and cooling to room temperature, and can be isolated (by filtration) in a yield of 87% (from a 1.4 mole scale reaction). ^1H NMR, δ: s, -0.13 ppm in CCl_4 or -0.01 ppm in ether.

Preparation of $p\text{-CH}_3\text{C}_6\text{H}_4\text{C}\{\text{NSiMe}_3\}\text{N(SiMe}_3)_2$ (4b)

A solution of p-tolunitrile (50.0 g, 0.427 mol) in 50 ml of ether was added dropwise to a slurry of $(\text{Me}_3\text{Si})_2\text{NLi} \cdot \text{OEt}_2$ **(2)** (103.0 g, 0.427 mol) in 400 ml ether in a 1 l round bottom flask. After 24 h the formation of **3b** was shown to be quantitative by NMR (δ, s, 0.38 ppm in ether solution). The ether was distilled off and 350 ml of toluene was added. 46.4 g of ClSiMe_3 (0.427 mmol) in 50 ml of toluene was then added and the mixture refluxed for 5 h. After cooling, LiCl was separated from the light orange solution by decanting and filtration, and the solvent was removed by distillation. High vacuum distillation using an air-cooled condenser removed some p-tolunitrile as a first cut, and gave **4b** as a pale yellow liquid, b.p. $80°\text{C}/0.03$ mmHg. Yield 100.0 g, 67%. **4b** solidifies on standing or refrigeration to give a waxy solid, m.p. $90°$ C; m/z 350 (26% M^+), 335 (34% $[M - \text{Me}]^+$), 277 (23% $[M - \text{SiMe}_3]^+$), 245 (85% $[(\text{Me}_3\text{Si})_2\text{CNSiMe}_3]^+$), 190 (72% $[M - \text{N(SiMe}_3)_2)$;

ν(C=N) 1615, δ(SiMe$_3$) 1240 cm^{-1} (Found: C, 58.22; H, 9.81; N, 8.06; C$_{17}$H$_{34}$N$_2$Si$_3$ calc: C, 58.22; H, 9.77; N, 7.99%).

Preparation of p-ClC$_6$H$_4$C{NSiMe$_3$}N(SiMe$_3$)$_2$ (4c)

Procedure as for **4b** with 27.5 g (0.200mol) of 4-chlorobenzonitrile, 48.3 g of (Me$_3$Si)$_2$NLi · OEt$_2$ (0.200 mol) and 21.7 g (0.200 mol) of ClSiMe$_3$, except that the nitrile and lithium silylamide were stirred together as a slurry. The product distils as a pale yellow liquid at 100°C/0.11 mmHg, which solidifies on cooling, yield 40.0 g, 54%. M.p. 47–48°C; m/z 370 (9%, M^+), 355 (14%, $[M - Me]^+$), 297 (12%, $[M - SiMe_3]^+$), 245 (45%, $[(Me_3Si)_2CNSiMe_3]^+$), 210 (19%, $[M - N(SiMe_3)_2]^+$); ν(C=N) 1620, δ(SiMe$_3$) 1245 cm^{-1} (Found: C, 51.62; H, 8.53; N, 7.50; C$_{16}$H$_{31}$ClN$_2$Si$_3$ calc: C, 51.78; H, 8.42; N, 7.55%).

Preparation of p-MeOC$_6$H$_4$C{NSiMe$_3$}N(SiMe$_3$)$_2$ (4d)

By the same method as **4c** with 17.0 g of 4-methoxybenzonitrile (0.128 mol), 31.7 g of (Me$_3$Si)$_2$NLi · OEt$_2$ (0.131 mol) and 15.6 g of ClSiMe$_3$ (10% excess). **4d** distils as a pale yellow liquid, b.p. 108–112°C/0.2 mmHg, which solidifies on cooling, yield 28.1 g, 60%. M.p. 35–40°C; m/z 366 (8%, M^+), 351 (21%, $[M - Me]^+$), 293 (6%, $[M - SiMe_3]^+$), 245 (76%, $[(Me_3Si)_2CNSiMe_3]^+$), 206 (55%, $[M - N(SiMe_3)_2]^+$); ν(C=N) 1620, δ(SiMe$_3$) 1245 cm^{-1} (Found: C, 55.70; H, 9.17; N, 7.66; C$_{17}$H$_{34}$N$_2$OSi$_3$ calc: C, 55.89; H, 9.34; N, 7.64%).

Preparation of p-Me$_2$NC$_6$H$_4$C{NSiMe$_3$}N(SiMe$_3$)$_2$ (4e)

By the same method as **4c** with 50.0 g of 4-(dimethylamino)-benzonitrile (0.342 mol), 82.6 g of (Me$_3$Si)$_2$NLi · OEt$_2$ (0.342 mol) and 40.9 g of ClSiMe$_3$ (10% excess). **4e** distils as a yellow liquid, b.p. 115–135°C/0.2 mmHg, which solidifies on cooling to a waxy solid, yield 93.5 g, 72%. Mp. 51–55°C; m/z 379 (4%, M^+), 364 (28%, $[M - Me]^+$), 306 (4%, $[M - SiMe_3]^+$), 245 (80%, $[(Me_3Si)_2CNSiMe_3]^+$), 219 (64%, $[M - N(SiMe_3)_2]^+$); ν(C=N) 1605, δ (SiMe$_3$) 1245 cm^{-1} (Found: C, 56.71; H, 9.71; N, 11.20; C$_{18}$H$_{37}$N$_3$Si$_3$ calc: C, 56.93; H, 9.82; N, 11.06%).

Preparation of p-F$_3$CC$_6$H$_4$C{NSiMe$_3$}N(SiMe$_3$)$_2$ (4f)

By the same method as **4b** with 20.0 g of 4-trifluoromethylbenzonitrile (0.117 mol), 28.2 g of (Me$_3$Si)$_2$NLi · OEt$_2$ (0.117 mol) and 13.9 g of ClSiMe$_3$ (10% excess). **4h** distils as a yellow liquid, b.p. 95°C/0.1 mmHg, which solidifies on cooling, yield 28.1 g, 59%. M.p. 26–31°C; m/z 404 (4%, M^+), 389 (4%, $[M - Me]^+$), 331 (5%, $[M - SiMe_3]^+$), 147 (42%, $[CF_3C_6H_5]^+$); ν(C=N) 1630, δ(SiMe$_3$) 1250 cm^{-1} (Found: C, 50.51; H, 7.84; N, 7.06; C$_{17}$H$_{31}$F$_3$N$_2$Si$_3$ calc: C, 50.45; H, 7.72; N, 6.92%).

Preparation of p-C$_6$H$_5$C$_6$H$_4$C{NSiMe$_3$}N(SiMe$_3$)$_2$ (4g)

By the same method as **4c** with 6.12 g of 4-biphenylcarbonitrile (34.2 mmol), 8.25 g of (Me$_3$Si)$_2$NLi · OEt$_2$ (34.2 mmol) and 4.09 g of ClSiMe$_3$ (10% excess.) **4g** can only be distilled using an ultra-short path-length still, b.p. ~ 134°C/0.1 mmHg. It solidifies readily. M.p. 75–78°C, yield ~ 7 g, ~ 50%, with the nitrile as an impurity. m/z 412 (15%, M^+), 397 (21% $[M - Me]^+$), 339 (12%, $[M - SiMe_3]^+$), 252 (100%, $[M - N(SiMe_3)_2]^+$), 245 (96%, $[(Me_3Si)_2CNSiMe_3]^+$); ν(C=N) 1640, δ(SiMe$_3$) 1245 cm^{-1} (Found: C, 65.6; H, 8.51: N, 7.29; C$_{22}$H$_{36}$N$_2$Si$_3$ calc: C, 64.01; H, 8.79; N, 6.79%).

Preparation of F₃CC{NSiMe₃}N(SiMe₃)₂ (4j)

5.65 g of CF_3CN (60 mmol) was introduced as a vapor via a stream of dry nitrogen to 13.5 g of $(Me_3Si)_2NLi \cdot OEt_2$ (56 mmol) suspended in 80 ml ether in a 250 ml three-necked flask equipped with a dry-ice condenser. The flask was cooled to 0 °C during addition. After stirring the mixture at room temperature for 2 h and at reflux for 1 h, the solvent was removed and replaced by 75 ml toluene. 6.07 g of $ClSiMe_3$ in 5 ml of toluene was then added, and the mixture refluxed for 7 h. Worked up as for **4b**, except that a water-cooled condenser was required, b.p. 40 °C/0.20 mmHg, yield 8.9 g, 46%. $\delta(^{13}C)$ 0.7 (s, $=NSi(CH_3)_3$), 2.1 (s, $N\{Si(CH_3)_3\}_2$), 116.1 (q, CF_3, $^1J(FC)$ 289.0 Hz), 150.4 (q, CF_3C, $^2J(FC)$ 35.4 Hz); m/z 313 (6%, $[M - Me]^+$), 259 (100%, $[M - CF_3]^+$), 171 (94%, $[(Me_3Si)_2CN]^+$); $\nu(C=N)$ 1640, $\delta(SiMe_3)$ 1255 cm^{-1} (Found: C, 40.15; H, 8.46; N, 8.52; $C_{11}H_{27}F_3N_2Si_3$ calc: C, 40.21; H, 8.28; N, 8.53%).

Preparation of p-C₆H₄[C{NSiMe₃}N(SiMe₃)₂]₂ (6)

1,4-Dicyanobenzene (12.8 g, 0.100 mol) and $(Me_3Si)_2NLi \cdot OEt_2$ (48.3 g, 0.200 mol) were mixed together in 150 ml of ether, and the resulting slurry was stirred at room temperature overnight. The ^1H NMR spectrum of the reaction mixture indicated a quantitative conversion to $p\text{-}C_6H_4[C\{=NLi\}N(SiMe_3)_2]_2$, $\delta(SiMe_3)$ −0.26 ppm (the one-to-one adduct is at −0.36 ppm, cf. Table 1). The ether was removed and replaced by 125 ml of toluene, containing 22.0 g (0.202 mol) of $ClSiMe_3$, and the mixture was refluxed for 5 h. A hot filtration under N_2 removed the precipitated LiCl. On cooling the filtrate, **6** precipitated and was washed twice with 20 ml toluene to give 25.9 g of off-white solid (43%). Recrystallization from 30 ml of toluene afforded the product as a white, crystalline solid, m.p. 162–167 °C. Alternatively, **6** could be sublimed readily at 150–160 °C/0.07 mmHg, yielding large clear crystals, m.p. 162–166 °C. $\delta(^1H)$ s 7.27 (aromatic, s 0.068 (SiMe₃); m/z 594 (9%, M^+), 579 (20%, $[M - Me]^+$), 434 (26%, $[M - N(SiMe_3)_2]^+$), 245 (100%, $[(Me_3Si)_2CNSiMe_3]^+$); $\nu(C=N)$ 1630, $\delta(SiMe_3)$ 1245 cm^{-1} (Found: C, 52.28; H, 9.70; N, 9.55; $C_{26}H_{58}N_4Si_6$ calc: C, 52.46; H, 9.82; N, 9.41%).

Preparation of amidines

Benzamidine hydrochloride (7a)

9.3 g of benzonitrile (0.090 mol) was added to 21.7 g (0.090 mol) of **2** suspended in 100 ml dry ether. Formation of **3a** was monitored by NMR; when complete (~ 1 h), the solution was cooled to 0 °C, four equivalents of $6N$ ethanolic HCl were added, and the mixture set aside for several hours. The precipitate was then filtered, washed with ether and recrystallized from ethanol/water to give 10.74 g of **7a** (78%). M.p. 172–176 °C (lit. 166–168 °C [7]).

Preparation of other amidine hydrochlorides

The following amidine hydrochlorides were prepared in a manner entirely analogous to **7a**: **7c** from 10.0 g of 4-chlorobenzonitrile and 17.6 g of **2** to give 10.7 g of product (76%), m.p. 249–251 °C (lit. 241–242 [7,12]); **7d** from 18.1 g of 4-methoxybenzonitrile and 33.1 g of **2** to give 22.3 g of product (88%), m.p. 222–226 °C (**7d** was characterized as the free base by microanalysis: Found: C, 63.89; H, 6.61; N, 18.69; $C_8H_{10}N_2O$ calc: C, 63.98; H, 6.71; N, 18.65%);

7i from 8.3 g 4-nitrobenzonitrile and 13.9 g of **2** to give 8.5 g product (75%), m.p. 293–295°C (lit. 285–287°C [6]).

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