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A Novel One-Pot Synthesis of *N*,*N*-Dimethylaminopyridines by Diazotization of Aminopyridines in Dimethylformamide in the Presence of Trifluoromethanesulfonic Acid

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Abstract—Diazotization of aminopyridines in the presence of trifluoromethanesulfonic acid gives the corresponding pyridinyl trifluoromethanesulfonates instead of expected diazonium salts. Pyridinyl trifluoromethanesulfonates can be converted to *N*,*N*-dimethylaminopyridines on heating in dimethylformamide via replacement of the trifluoromethanesulfonyloxy group. The reaction is accelerated under microwave irradiation. A novel one-pot procedure has been proposed for the synthesis of 2- and 4-(dimethylamino)pyridines from commercially available aminopyridines. The procedure provides high yields of the target products, and it can be regarded as an alternative to the known methods of synthesis of *N*,*N*-dimethylpyridin-4-amine (DMAP) widely used as base catalyst in organic synthesis.

Keywords: aminopyridines, diazotization, pyridyl trifluoromethanesulfonates, *N*,*N*-dimethylaminopyridines

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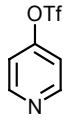
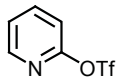
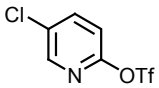
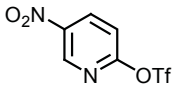
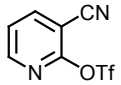
N,*N*-Dimethylaminopyridines are widely used in the synthesis of biologically active compounds of the pyridine series [1–4] and are also of interest as such. Of particular importance is *N*,*N*-dimethylpyridin-4-amine (DMAP) which catalyzes numerous organic reactions [5–7]. However, despite practical significance of *N*,*N*-dimethylaminopyridines, methods of their preparation are few in number. For example, alkylation with methyl iodide, dimethyl sulfate, or methanol in the presence of Lewis acids leads to the formation of mixtures of mono- and dialkylamino derivatives. Successful reactions with the systems based on formaldehyde [8] or formic acid [9] have been reported only for 2-aminopyridine. At present, the most appropriate method of synthesis of *N*,*N*-dimethylaminopyridines is the amination of 2- and 4-halopyridines with dimethylamine [10–14]; the target products are thus obtained in 83–91% yield. The amination of 3-iodo- and 3-bromopyridines was accomplished only in the presence of copper iodide [14]. In some cases [15–18], *N*,*N*-dimethylformamide was used as a source of dimethylamino group. For example, 2- and 4-halopyridines and quinolines were converted to the corresponding *N*,*N*-dimethylamino derivatives in DMF solution in the presence of a base or acid under microwave irradiation [17] or on prolonged heating [18].

We previously found that diazotization of aminopyridines and aminoquinolines in the presence of sulfonic acids such as *p*-toluenesulfonic acid or trifluoromethanesulfonic acid leads to the formation of the corresponding heteroaryl sulfonates [19, 20]. The observed peculiarity in the behavior of aminopyridines and aminoquinolines allowed us to develop a convenient procedure for the synthesis of pyridyl and quinolyl *p*-toluenesulfoates and trifluoromethanesulfonates that are important intermediate products in organic synthesis. It is known that trifluoromethanesulfonyloxy group is a good nucleofuge [21]. However, substitution of that group in pyridines by dimethylamino group has not been reported. Thus, the goal of the present work was to study the reactivity of pyridyl trifluoromethanesulfonates toward dimethylformamide with a view to obtaining *N*,*N*-dimethylaminopyridines.

We were the first to find out that pyridin-2-yl and pyridin-4-yl trifluoromethanesulfonates **1a**–**1c**, **1e**, and **1f** can be converted to *N*,*N*-dimethylaminopyridines **2a**–**2c**, **2e**, and **2f** in good yields on heating in dimethylformamide (method *a*; Table 1). The reaction was faster than with chloro- or bromopyridines. For example, dimethylaminopyridine **2a** was obtained from 4-chloropyridine in 69% yield by heating in boiling dimethylformamide for 34 h [18], whereas trifluoro-

Table 1. Synthesis of *N*,*N*-dimethylaminopyridines **2a**–**2c**, **2e**, and **2f** from pyridyl trifluoromethanesulfonates **1a**–**1c**, **1e**, and **1f** and DMF at 160°C (method *a*)

Substrate	Reaction time, h	Yield of **2**, %
 **1a**	5 (34 [18])	89
 **1b**	5.5 (29 [26])	90
 **1c**	5 (24 [27])	90
 **1e**	6 (24 [18])	58
 **1f**	5 (22 [18])	90

methanesulfonate **1a** was converted to **2a** in 5 h with 89% yield (Table 1).

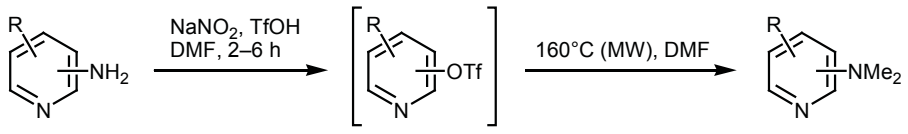
We previously showed that DMF is a good solvent for the diazotization of 2-aminopyridine with $NaNO_2$ in the presence of TfOH. However, the reaction is accompanied by formation of a strong complex of DMF with pyridin-4-yl trifluoromethanesulfonate (**1a**), which complicates isolation of **1a** [20].

In this work we have found that the diazotization of a number of 2- and 4-aminopyridines **3a**–**3i** with $NaNO_2$/TfOH in DMF at 20°C is characterized by complete conversion of the substrate. Further heating of the reaction mixtures without isolation of intermediate pyridyl trifluoromethanesulfonates leads to the formation of *N*,*N*-dimethylaminopyridines **2a**–**2i**. Furthermore, microwave activation at the stage of transformation of **1a**–**1i** to **2a**–**2i** by the action of DMF significantly shortens the reaction time (method *b*; Scheme 1, Table 2).

For example, the transformation of **1b** to **2b** without microwave irradiation (method *a*) takes 5.5 h (Table 1), whereas the complete conversion of **1b** under microwave irradiation (method *b*) is achieved in 0.83 h (Table 2). The yields of *N*,*N*-dimethylaminopyridines **2a**–**2i** obtained by methods *a* and *b* generally exceed those reported for the synthesis of the same compounds from halopyridines [17, 18]. As shown with *N*,*N*-dimethylpyridin-4-amine (**2a**, DMAP) as an example, the yield did not decrease in enlarged synthesis (10 mmol; Table 2).

The developed one-pot procedure for the transformation of aminopyridines to *N*,*N*-dimethylamino-

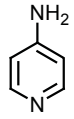
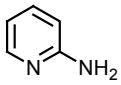
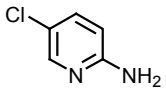
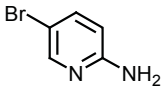
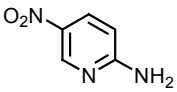
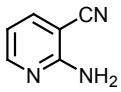
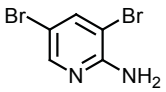
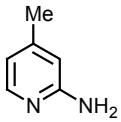
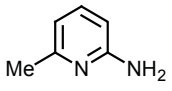
Scheme 1.



3a–3i **1a–1i** **2a–2i**

R = H, 4-Me_2N (**a**); R = H, 2-Me_2N (**b**); R = 5-Cl, 2-Me_2N (**c**); R = 5-Br, 2-Me_2N (**d**); R = 5-O_2N, 2-Me_2N (**e**); R = 3-CN, 2-Me_2N (**f**); R = 3,5-Br, 2-Me_2N (**g**); R = 4-Me, 2-Me_2N (**h**); R = 6-Me, 2-Me_2N (**i**).

Table 2. Synthesis of *N*,*N*-dimethylaminopyridines **2a**–**2i** by diazotization of aminopyridines **3a**–**3i** in DMF in the presence of trifluoromethanesulfonic aid and subsequent heating under microwave irradiation (method *b*)

Substrate	Reaction time,[a] h	Product	Yield, %
 **3a**	0.83	**2a**	92[b] (69 [18])
 **3b**	0.83	**2b**	90 (92 [26])
 **3c**	3.3	**2c**	91 (85 [27])
 **3d**	3	**2d**	87 (82 [28])
 **3e**	0.83	**2e**	98 (76 [18])
 **3f**	0.83	**2f**	94 (73 [18])
 **3g**	2	**2g**	78 (77 [29])
 **3h**	0.83	**2h**	92 (80 [13])
 **3i**	1.6	**2i**	81 (63 [30])

[a] Second stage; diazotization of aminopyridines **3a**–**3i** (first stage) was carried out for 3 h.
[b] Reaction with 10 mmol of 4-aminopyridine.

pyridines through intermediate pyridyl trifluoromethanesulfonates makes it possible to successfully obtain *N*,*N*-dimethylpyridin-2- and -4-amines with both electron-donating and electron-withdrawing substituents in the pyridine ring (Table 2). However, the amino group in 3-aminopyridine was not replaced by dimethylamino group. In this case, pyridin-3-yl trifluoromethanesulfonate formed in the first diazotization stage remained unchanged after heating in DMF for 10 h. It is known that the halogen atom in 3-halopyridines cannot be replaced by dimethylamino via reaction with DMF without a catalyst [15–18, 22].

The mechanism of the described transformation of trifluoromethanesulfonates **1a**–**1i** to dimethylamino derivatives **2a**–**2i** in DMF was not specially studied; nevertheless, it is likely to be consistent with that proposed in [18] where *N*,*N*-dimethylaminopyridines were synthesized by prolonged heating of chloropyridines in dimethylformamide.

In summary, we have demonstrated for the first time that trifluoromethanesulfonyloxy group on C^2 or C^4 of the pyridine ring is readily replaced by dimethylamino group on heating in DMF to produce the corresponding *N*,*N*-dimethylaminopyridines. This reaction is faster than analogous transformation of halopyridines. These findings allowed us to develop a one-pot procedure for the synthesis of 2- and 4-(dimethylamino)pyridines by diazotization of 2- and 4-aminopyridines in DMF in the presence of trifluoromethanesulfonic acid at room temperature and subsequent fast heating of the reaction mixture under microwave irradiation without isolation of intermediate pyridyl trifluoromethanesulfonates. The proposed procedure provides high yields of the target products and is an efficient alternative to the known methods of synthesis of 4-(dimethylamino)-pyridine (DMAP), a widely used catalyst in organic synthesis.

EXPERIMENTAL

The mass spectra were recorded on an Agilent Technologies 7890A/5975C GC/MS system (electron impact, 70 eV; carrier gas helium). The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker AC-400 spectrometer at 400 and 100 MHz, respectively, using tetramethylsilane as internal standard. The melting points were measured on a Mettler Toledo MP50 melting point apparatus. The microwave-assisted reactions were carried out in a CEM Discover Labmate microwave reactor (irradiation frequency 2455 MHz).

The progress of reactions was monitored, and the purity of the isolated compounds was checked, by TLC on silica gel 60 F_{254} plates (Merck). Spots were visualized under UV light (λ 254 nm). The products were isolated by flash chromatography using 15×1-cm columns packed with silica gel (40–60 μm). Aminopyridines **3a**–**3i** were commercial products (Aldrich); pyridyl trifluoromethanesulfonates **1a**–**1e** were synthesized as described in [20].

General procedure for the synthesis of *N*,*N*-dimethylpyridin-2- and -4-amines from pyridyl trifluoromethanesulfonates in DMF at 160°C. *a*. A solution of pyridyl trifluoromethanesulfonate **1a**–**1c**, **1e**, or **1f** (2 mmol) in 0.5 mL of dimethylformamide was heated at 160°C with continuous stirring for a time indicated in Table 1. The progress of the reaction was monitored by TLC (eluent ethyl acetate–hexane, 1:3, R_f 0.2–0.3) and GC/MS. The mixture was then poured into 40 mL of water, neutralized with a 25% aqueous solution of Na_2CO_3 (4 mL), and extracted with ethyl acetate (50×3 mL). The combined extracts were washed with 40 mL of cold water and dried over Na_2SO_4, the solvent was distilled off, and the residue was purified by flash chromatography using methylene chloride as eluent.

General procedure for the synthesis of *N*,*N*-dimethylpyridin-2- and -4-amines 2a–2i by diazotization of aminopyridines 3a–3i in DMF in the presence of trifluoromethanesulfonic acid and subsequent heating under microwave irradiation. *b*. A solution of trifluoromethanesulfonic acid (6 mmol, 0.54 mL) in dimethylformamide (0.5 mL) was cooled to 5°C, preliminarily ground mixture of aminopyridine **3a–3i** (2 mmol) and sodium nitrite (5 mmol, 0.35 g) was added over a period of 10 min with continuous stirring, and the mixture was stirred for 3 h at room temperature. The progress of the reaction was monitored by TLC (eluent ethyl acetate–hexane, 3:1, R_f 0.68–0.72) and GC/MS. An additional amount of DMF (0.5 mL) was then added, and the mixture was stirred under microwave irradiation (50 W) at a pressure of 3 bar for a time indicated in Table 2. The progress of the reaction was monitored by TLC (eluent ethyl acetate–hexane, 1:3, R_f 0.2–0.3) and GC/MS. The products were isolated as described above in *a*.

***N*,*N*-Dimethylpyridin-4-amine (2a).** Yield 0.224 g (92%), white crystals, mp 108–110°C (mp 109–111°C [18]). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.89 s [6H, $N(CH_3)_2$], 6.97 d (2H, 3-H, 5-H, J = 8.0), 8.20 d (2H, 2-H, 6-H, J = 8.0). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 36.56 [$N(CH_3)_2$], 107.01 (C^3, C^5), 139.27 (C^2, C^6), 162.65 (C^4).

***N*,*N*-Dimethylpyridin-2-amine (2b).** Yield 0.219 g (90%), light yellow oil (bp 196°C [32]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 2.99 s [6H, $N(CH_3)_2$], 6.41–6.46 m (2H, 3-H, 5-H), 7.33–7.37 m (1H, 4-H), 8.075 d (1H, 6-H, J = 4.0). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 38.14 [$N(CH_3)_2$], 105.91 (C^3), 111.42 (C^5), 137.17 (C^4), 147.72 (C^6), 159.26 (C^2).

5-Chloro-*N*,*N*-dimethylpyridin-2-amine (2c). Yield 0.284 g (91%), yellow oil (mp 26–27°C [31]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 3.08 s [6H, $N(CH_3)_2$], 6.46 d (1H, 3-H, J = 8.0), 7.4 d (1H,

4-H, J = 8.0), 8.10 s (1H, 6-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 38.33 [$N(CH_3)_2$], 106.63 (C^3), 118.54 (C^5), 136.93 (C^4), 145.87 (C^6), 157.53 (C^2).

5-Bromo-*N*,*N*-dimethylpyridin-2-amine (2d). Yield 0.349 g (87%), yellow crystals, mp 40–41°C (mp 39–41°C [12, 13]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (J, Hz): 3.04 s [6H, $N(CH_3)_2$], 6.38 d (1H, 3-H, J = 9.0), 7.47 d.d (1H, 4-H, J = 9.0, 2.5), 8.15 d (1H, 6-H, J = 2.3). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 38.2 [$N(CH_3)_2$], 106.0 (C^5), 107.3 (C^3), 139.3 (C^4), 148.3 (C^6), 157.8 (C^2).

***N*,*N*-Dimethyl-5-nitropyridin-2-amin (2e).** Yield 0.328 g (98%), yellow crystals, mp 156–157°C (mp 155–157°C [18]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (J, Hz): 3.26 s [6H, $N(CH_3)_2$], 6.49 d (1H, 3-H, J = 8.0), 8.22 d (1H, 4-H, J = 12.0), 9.07 s (1H, 6-H). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 38.55 [$N(CH_3)_2$], 104.33 (C^3), 132.76 (C^4), 134.60 (C^5), 146.33 (C^6), 160.63 (C^2).

2-(Dimethylamino)pyridine-3-carbonitrile (2f). Yield 0.278 g (94%), dark yellow oil (bp 265°C [23]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (J, Hz): 3.22 s [6H, $N(CH_3)_2$], 6.55 d.d (1H, 5-H, J = 8.0, 4.0), 7.65 d.d (1H, 4-H, J = 8.0, 4.0), 8.22 d.d (1H, 6-H, J = 4.0, 4.0). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 40.37 [$N(CH_3)_2$], 91.09 (C^3), 111.99 (C^5), 119.17 (CN), 144.56 (C^4), 151.72 (C^6), 159.33 (C^2).

3,5-Dibromo-*N*,*N*-dimethylpyridin-2-amine (2g). Yield 0.436 (78%), yellow crystals, mp 42–43°C (mp 41–43°C [24]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (J, Hz): 2.97 s [6H, $N(CH_3)_2$], 7.86 d (1H, 4-H, J = 2.4), 8.19 d (1H, 6-H, J = 2.4). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 41.93 [$N(CH_3)_2$], 110.55 (C^5), 110.68 (C^3), 144.10 (C^4), 146.64 (C^6), 158.98 (C^2).

***N*,*N*,4-Trimethylpyridin-2-amine (2h).** Yield 0.251 (92%), light yellow oil (bp 225°C [3]). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (J, Hz): 2.24 s (3H, CH_3), 3.05 s [6H, $N(CH_3)_2$], 6.31 s (1H, 3-H), 6.38 d (1H, 5-H, J = 4.8), 8.03 d (1H, 6-H, J = 5.2). ^{13}C NMR spectrum ($CDCl_3$), δ_C, ppm: 21.5 (CH_3), 38.2 [$N(CH_3)_2$], 106.3 (C^3), 113.2 (C^5), 147.6 (C^6), 148.0 (C^4), 159.8 (C^2).

***N*,*N*,6-Trimethylpyridin-2-amine (2i).** Yield 0.222 g (81%), light yellow oil (bp 198–200°C [25]). ^{1}H NMR spectrum (500 MHz, $CDCl_3$), δ, ppm (J, Hz): 2.41 s (3H, CH_3), 3.07 s [6H, $N(CH_3)_2$], 6.32 d (1H, 3-H, J = 8.3), 6.41 d (1H, 5-H, J = 7.5), 7.34 d.d (1H, 4-H, J = 8.3, 7.5). ^{13}C NMR spectrum (125 MHz, $CDCl_3$), δ_C, ppm: 24.6 (CH_3), 37.9 [$N(CH_3)_2$], 102.4 (C^3), 110.6 (C^5), 137.3 (C^4), 156.5 (C^6), 159.1 (C^2).

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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