Stepwise Approach to the 2,3-Dihydroimidazo[1,2-*a*]pyridine and 5-Oxo-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine Ring Systems

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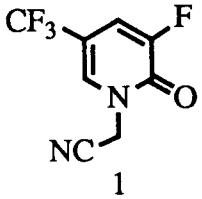
Received February 8, 1991

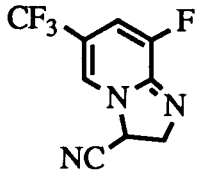
The syntheses of 2,3-dihydro-8-fluoro-6-trifluoromethylimidazo[1,2-*a*]pyridine-3-carbonitrile (**2**), 8-fluoro-6-trifluoromethylimidazo[1,2-*a*]pyridine-3-carbonitrile (**9**) and 5-oxo-8-trifluoromethyl-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine-3-carbonitrile (**3**) are described. These compounds are constructed in a stepwise approach starting from the properly substituted 2-halopyridines.

J. Heterocyclic Chem., **28**, 971 (1991).

A study of the biological activity of cyanomethylpyridone **1** [1] suggested the synthesis of rigid analogues with the nitrile group in a fixed orientation. Two compounds **2** and **3** were targeted for synthesis based on their similarity to the parent compound **1**. A literature search revealed several syntheses of the required 3-cyanoimidazo-[1,2-*a*]pyridine system [2-6]; however, none of these furnished the 2,3-dihydro analogues. The synthesis described here should provide a general route to the imidazo[1,2-*a*]-pyridine system.



1



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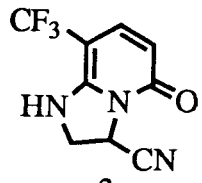
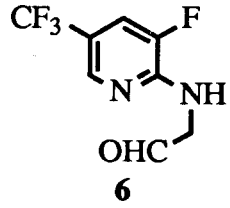


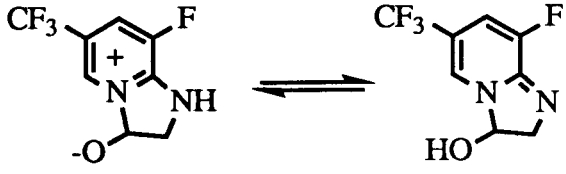
3

The most common approach to the synthesis of imidazo-[1,2-*a*]pyridines [7] involves the condensation of α-halo carbonyl compounds with 2-aminopyridines. Attempted reactions of 2-amino-3-fluoro-5-trifluoromethylpyridine with ethyl 2-bromoacetate, its acid chloride, and 2,3-dibromopropionitrile gave complex mixtures with none of the desired products observed. The electron deficient nature of this aminopyridine appears to result in its unreactive nature towards electrophiles. Other syntheses have been reported [8,9].

The reactivity of halopyridines towards amine and oxygen nucleophiles [10] suggested the approach used for this synthesis (Scheme I). Reaction of 2,3-difluoro-5-trifluoromethylpyridine (**4**) with aminoacetaldehyde dimethyl acetal gave pyridinamine **5**. Hydrolysis of **5** provided aldehyde **6**. This material was a stable solid which gave correct ms and elemental data; however, the nmr spectra was unresolved and the compound was insoluble in most organic solvents. An explanation of this behavior might involve the presence of tautomeric forms. A similiar result has appeared [11].



6

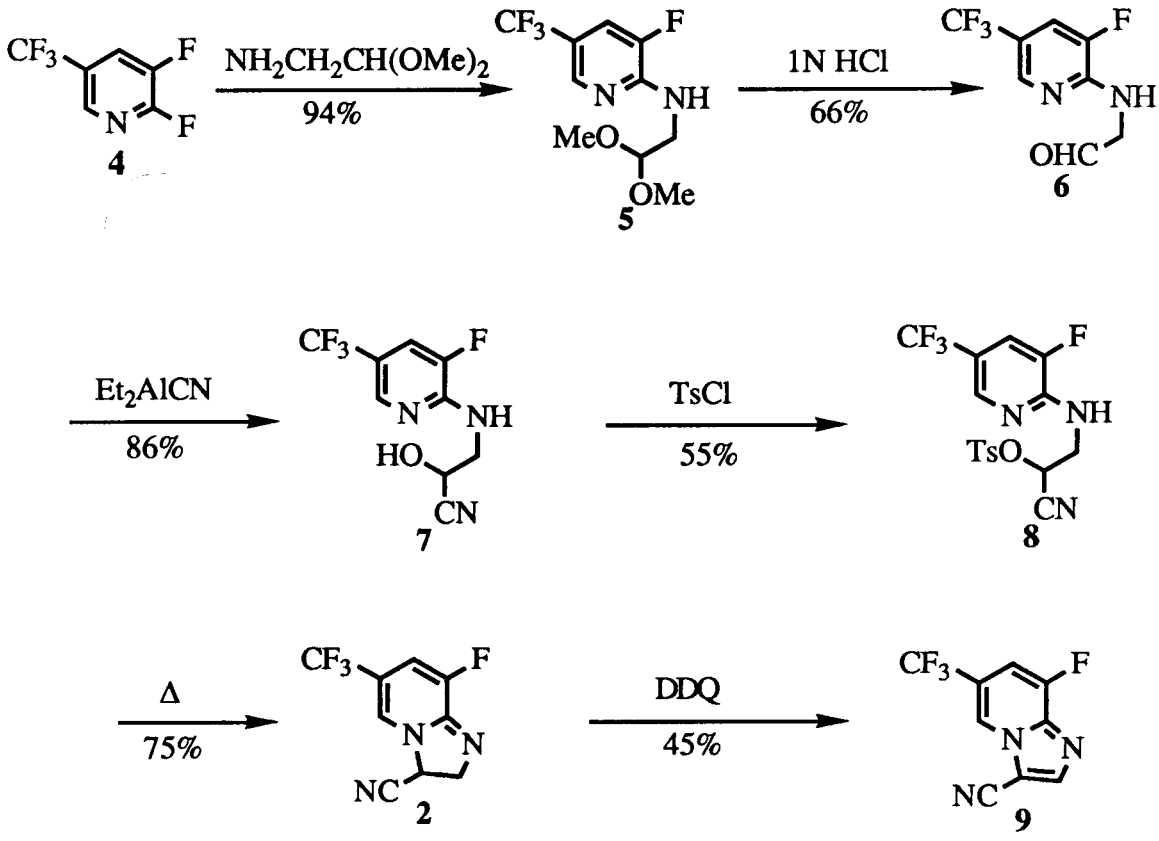


Reaction of **6** with diethylaluminum cyanide gave the desired cyanohydrin **7**. Tosylation followed by heating in acetonitrile afforded 2,3-dihydro-3-cyanoimidazo[1,2-*a*]-pyridine **2**. This compound could be dehydrogenated with DDQ in dioxane to yield 3-cyanoimidazo[1,2-*a*]pyridine **9**.

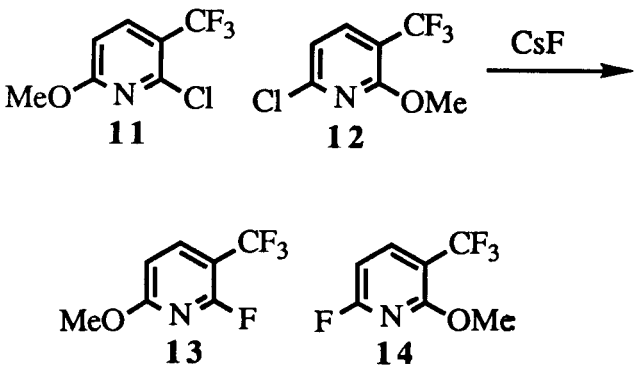
Although several syntheses of the 5-oxoimidazo[1,2-*a*]-pyridine system have been reported [12-15], the route used for the synthesis of **3** is shown in Scheme II. Reaction of 2,6-dichloro-3-trifluoromethylpyridine (**10**) with sodium methoxide gave a 97:3 ratio of 6-methoxy to 2-methoxy pyridines **11** and **12** by gc-ms analysis. The major isomer resulting from displacement of the 6-chlorine was confirmed by ^{19}F nmr spectroscopy. Reaction of the **11** and **12** mixture with cesium fluoride in DMSO at 120° for 4 hours gave fluorinated compounds **13** and **14**. The ^{19}F nmr showed a doublet for the trifluoromethyl resonance and a broad multiplet for the 2-fluorine, confirming assignment of **11** as the major methoxide displacement product.

Reaction of **11** with aminoacetaldehyde dimethyl acetal gave pyridinamine **15**. Hydrolysis of **15** provided aldehyde **16**, which showed none of the unusual properties of aldehyde **6**. Conversion to the cyanohydrin, followed by tosylation and cyclization as before afforded 5-oxo-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine **3**. An initial product was

Scheme I



formed in the final cyclization which disappeared with continued heating. This appeared to be the 5-methoxy compound **19** by ms and nmr analysis of an isolated sample. Attempted dehydrogenation of **3** with DDQ, 5% Pd/C, or benzeneseleninic anhydride failed to give compound **20**.



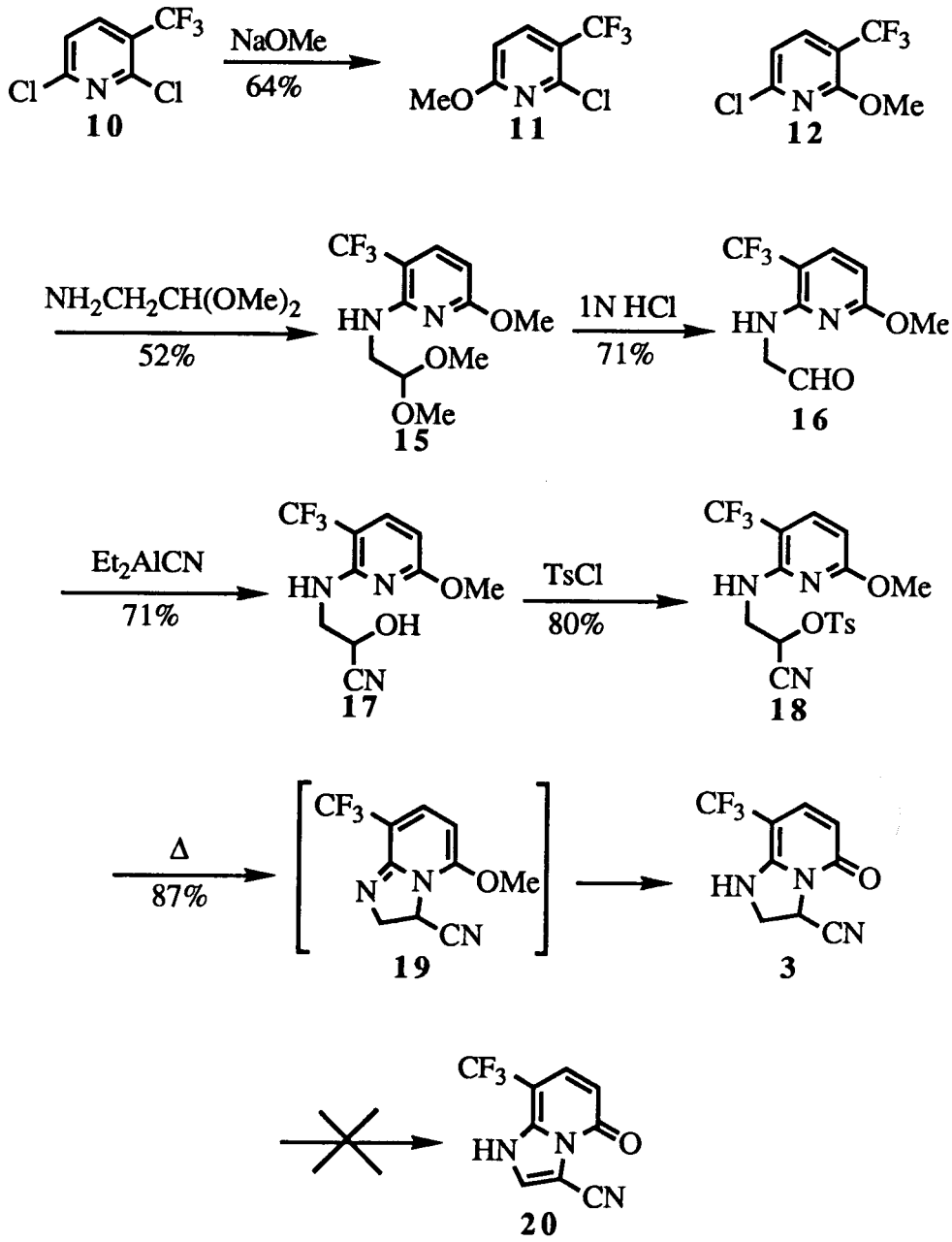
EXPERIMENTAL

Melting points were measured with a Thomas Hoover Capillary Melting Point Apparatus and are uncorrected. The ir spectra were recorded on a Perkin-Elmer 1310 spectrometer, and ^{1}H nmr spectra were obtained with a Varian EM390 spectrometer using tetramethylsilane as an internal standard. Mass spectra (DEP, 70 eV) were determined on a Finnigan Model 4615 spectrometer or a Hewlett Packard 5971A gc-ms system. Elemental analyses were provided by Spang Microanalytical Laboratories or DowElanco, Walnut Creek, CA. Gas chromatographic analyses were obtained from a Hewlett Packard 5980 Series II gas chromatograph with a 3396A Integrator. Whatman 230-400 mesh silica gel was used for flash chromatography. All reactions were carried out under a nitrogen atmosphere.

N-(2,2-Dimethoxyethyl)-3-fluoro-5-trifluoromethyl-2-pyridinamine (**5**).

A solution of 5.99 ml (55 mmoles) of aminoacetaldehyde dimethyl acetal in 4 ml of DMSO was added dropwise over 5 minutes to a slurry of 9.15 g (50 mmoles) of 2,3-difluoro-5-trifluoromethylpyridine (**4**) [16] and 7.59 g (55 mmoles) of potassium carbonate in 100 ml of DMSO. Some warming was noted. After stirring 12 hours at room temperature, water was added, and the mixture extracted twice with ether. The combined organic layers were washed with brine, dried over magnesium sulfate and concentrated under reduced pressure to give 12.67 g (94%) of a white, crystalline solid, mp 40-42°; ir (film): 3450, 3360, 2940, 2835, 1630, 1580, 1525, 1420, 1345, 1270, 1140, 1110, 930 cm^{-1}; ^{1}H nmr (deuteriochloroform): δ 8.20 (d, J = 1.5 Hz, 1 H), 7.35 (dd, J = 1.5 Hz and 10.0 Hz, 1 H), 5.23 (br s, 1H), 4.58 (t, J = 5 Hz, 1

Scheme II



H), 3.73 (m, 2 H), 3.48 (s, 6 H); eims: m/z (relative intensity) 268 (2.7), 237 (10.1), 217 (2.9), 205 (11.0), 193 (7.1), 164 (5.7), 75 (100).

Anal. Calcd. for $C_{10}H_{12}F_4N_2O_2$: C, 44.78; H, 4.51; N, 10.45. Found: C, 44.55; H, 4.36; N, 10.39.

((3-Fluoro-5-trifluoromethyl-2-pyridinyl)amino)acetaldehyde (**6**).

A solution of 2.68 g (10 mmoles) of *N*-(2,2-dimethoxymethyl)-3-fluoro-5-trifluoromethyl-2-pyridinamine (**5**) in 20 ml of glacial acetic acid and 10 ml of 1*N* hydrochloric acid was stirred at room temperature for 12 hours. After concentration under reduced pressure, the residue was dissolved in water, made basic with saturated sodium bicarbonate solution, and extracted twice with ethyl acetate. The organic extracts were combined, washed with water and brine, dried over magnesium sulfate, and concentrated under reduced pressure to give a yellow solid. The solid was recrystallized from 3:2 ethyl acetate/hexanes and dried in a vacuum oven at 60° to give 1.48 g (66%) of a white solid, mp 133-135°; ir (film): 3350 (br), 1620, 1520, 1425, 1315, 1260, 1105, 920, 890 cm^{-1}; ^1H nmr (DMSO-d_6): unresolved; eims: m/z (relative intensity) 223 (4.9), 222 (7.1), 203 (6.1), 194 (35.4), 193 (100), 166 (11.3), 165 (6.4), 164 (18.3), 146 (14.7).

Anal. Calcd. for $C_8H_6F_4N_2O$: C, 43.25; H, 2.72; N, 12.61.

Found: C, 43.12; H, 2.56; N, 12.62.

3-((3-Fluoro-5-trifluoromethyl-2-pyridinyl)amino)-2-hydroxypropanenitrile (**7**).

To a slurry of 5.42 g (24.4 mmoles) of ((3-fluoro-5-trifluoromethyl-2-pyridinyl)amino)acetaldehyde (**6**) in 125 ml of toluene was added dropwise over 10 minutes with ice-bath cooling 27 ml (26.8 mmoles) a 1*M* solution of diethylaluminum cyanide in toluene. After stirring at room temperature for 14 hours, the mixture was concentrated under reduced pressure, water and ethyl acetate added, and filtered through Celite. The layers were separated, the organic layer washed with saturated sodium bicarbonate solution, brine, dried over magnesium sulfate, filtered and cencentrated under reduced pressure. Flash chromatography of the residue on silica gel (3:1 ethyl acetate/hexanes) gave 5.49 g (90%) of a dark orange oil which was used without further purification; ir (film): 3430 (br), 2905, 1615, 1570, 1515, 1410, 1315, 1255, 1135, 1095, 1070, 920, 890, 740 cm^{-1}; ^1H nmr (deuteriochloroform): δ 8.18 (d, J = 1.5 Hz, 1H), 7.48 (dd, J = 1.5 Hz and 10.0 Hz, 1 H), 6.22 (br s, 1 H), 5.74 (br s, 1 H), 4.81 (br s, 1 H), 3.97 (m, 2 H); eims: m/z (relative intensity) 249 (4.8), 194 (17.3), 193 (100), 165 (5.1), 164 (13.3), 146 (10.3).

3-((3-Fluoro-5-trifluoromethyl-2-pyridinyl)amino)-2-hydroxypropanenitrile, 4-methylbenzenesulfonate (**8**).

A solution of 4.62 g (24.2 mmoles) of *p*-toluenesulfonyl chloride in 10 ml of dichloromethane was added dropwise over 10 minutes to a solution of 5.49 g (22 mmoles) of 3-((3-fluoro-5-trifluoromethyl-2-pyridinyl)amino)-2-hydroxypropanenitrile (**7**) and 4.2 ml (24.2 mmoles) of diisopropylethylamine in 60 ml of dichloromethane at ice-bath temperature. After warming to room temperature, the mixture was concentrated under reduced pressure, water and ether added, and the layers separated. The organic layer was washed successively with 1*N* hydrochloric acid, saturated sodium bicarbonate and brine, dried over magnesium sulfate, filtered and concentrated under reduced pressure to a moist, yellow solid. The residue was recrystallized from 4:1 hexanes/ethyl acetate and dried in a vacuum oven at room temperature to give 4.84 g (55%) of a light tan powder, mp 101-103°; ir (nujol): 3440, 1630, 1530, 1335, 1180, 1150, 1120, 1020, 930, 905, 815, 760 cm^{-1}; ^1H nmr (deuteriochloroform): δ 8.08 (s, 1 H), 7.73 (d, J = 9 Hz, 2 H), 7.38-7.16 (m, 3 H), 5.45-5.18 (m, 2 H), 4.15-3.65 (m, 2 H), 2.33 (s, 3 H); eims: m/z (relative intensity) 403 (1.6), 384 (1.4), 348 (1.4), 205 (1.6), 194 (8.2), 193 (100), 165 (2.0), 164 (5.8), 146 (1.9).

Anal. Calcd. for $C_{16}H_{13}F_4N_3O_3S$: C, 47.64; H, 3.25; N, 10.42. Found: C, 47.55; H, 3.17; N, 10.34.

2,3-Dihydro-8-fluoro-6-trifluoromethylimidazo[1,2-*a*]pyridine-3-carbonitrile (**2**).

A solution of 4.84 g (12 mmoles) of 3-((3-fluoro-5-trifluoromethyl-2-pyridinyl)amino)-2-hydroxypropanenitrile, 4-methylbenzenesulfonate (**8**) in 100 ml of acetonitrile was heated at reflux for 12 hours. After cooling to room temperature, the reaction was concentrated under reduced pressure, ethyl acetate added, the mixture washed with saturated sodium bicarbonate and brine, dried over magnesium sulfate, filtered and concentrated to a brown liquid. Flash chromatography on silica gel (2:1 ethyl acetate/hexanes) yielded a dark orange, partially crystalline oil. This was dissolved in hot 4:1 hexanes/ethyl acetate, cooled, the precipitate collected and dried in a vacuum oven at 60° to give 2.07 g (75%) of a light yellow powder, mp 89-91°; ir (film): 3050, 2920, 1665, 1600, 1415, 1350, 1325, 1210, 1145, 1100, 1040, 905, 865 cm^{-1}; ^1H nmr (deuteriochloroform): δ 7.38 (s, 1 H), 6.76 (d, J = 10 Hz, 1 H), 5.18 (m, 1 H), 4.49 (m, 2 H); eims: m/z (relative intensity) 231 (80), 230 (50), 212 (30), 205 (35), 204 (75), 164 (100).

Anal. Calcd. for $C_9H_5F_4N_3$: C, 46.76; H, 2.18; N, 18.17. Found: C, 46.67; H, 2.02; N, 18.32.

8-Fluoro-6-trifluoromethylimidazo[1,2-*a*]pyridine-3-carbonitrile (**9**).

A mixture of 846 mg (3.66 mmoles) of 2,3-dihydro-8-fluoro-6-trifluoromethylimidazo[1,2-*a*]pyridine-3-carbonitrile (**2**) and 910 mg (4.0 mmoles) of 2,3-dichloro-5,6-dicyano-1,4-benzoquinone in 25 ml of dioxane was heated at 90° for 3 hours. After cooling, ether was added, and the mixture filtered through Celite. The filtrate was washed with saturated sodium bicarbonate and brine, dried over magnesium sulfate and carbon black, filtered and concentrated under reduced pressure to a yellow oil which slowly crystallized. This material was recrystallized from 2:1 hexanes/ethyl acetate and dried at 60° in a vacuum oven to give 375 mg (45%) of a yellow powder, mp 91-93°; ir (film): 3080, 3040, 2200, 1555, 1420, 1370, 1335, 1305, 1260, 1220, 1175, 1150, 1055, 985, 870, 735, 695 cm^{-1}; ^1H nmr (deuteriochloroform): δ 8.62 (d, J = 1.5 Hz, 1 H), 8.30 (s, 1 H), 7.38 (dd, J = 1.5 Hz, and 9 Hz, 1 H); eims: m/z (relative intensity) 230 (9.8), 229 (100), 210 (13.7), 179 (7.9), 164 (6.7), 150 (5.7), 114 (3.5).

Anal. Calcd. for $C_9H_3F_4N_3$: C, 47.17; H, 1.32; N, 18.34. Found: C, 46.89; H, 1.24; N, 18.39.

2-Chloro-6-methoxy-3-trifluoromethylpyridine (**11**).

To a solution of 21.6 g (0.10 mole) of 2,6-dichloro-3-trifluoromethylpyridine (**10**) [17] in 200 ml of methanol was added dropwise over 20 minutes 25.2 ml (0.11 mole) of a 25% by weight solution of sodium methoxide in methanol. After stirring at room temperature for 1 hour, the mixture was heated at 60° for 2 hours. After cooling and concentration under reduced pressure, ether and water were added, the layers separated, the organic layer washed with brine, dried over magnesium sulfate, filtered and concentrated under reduced pressure to give a moist white solid. This material was heated and dissolved in 15 ml of hot hexanes, cooled and collected to give 13.48 g (64%) of white crystals, mp 46-49°, a 97:3 ratio of 6-methoxy to 2-methoxy isomers by gc analysis; ir (nujol): 1600, 1560, 1480, 1310, 1270, 1145, 1110, 1015, 895, 830, 760, 645 cm^{-1}; ^1H nmr (deuteriochloroform): δ 7.79 (d, J = 9 Hz, 1 H), 6.68 (d, J = 9 Hz, 1 H), 3.94 (s, 3 H); eims: m/z (relative intensity) 213 (26.4), 212 (38.2), 211 (84.5), 210 (100), 194 (5.9), 192 (19.2), 184 (13.1), 183 (25.1), 182 (41.3), 181 (66.4), 161 (22.4), 147 (7.5), 146 (74.6), 133 (14.3), 126 (33.7).

Anal. Calcd. for $C_7H_5F_3ClNO$: C, 39.74; H, 2.38; N, 6.62. Found: C, 39.70; H, 2.39; N, 6.63.

N-(2,2-Dimethoxyethyl)-6-methoxy-3-trifluoromethyl-2-pyridinamine (**15**).

A mixture of 10.58 g (50 mmoles) of 2-chloro-6 methoxy-3-trifluoromethylpyridine (**11**), 6.6 ml (60 mmoles) of aminoacetaldehyde dimethyl acetal and 8.28 g (60 mmoles) of potassium carbonate in 150 ml of DMSO was heated at 120° for 14 hours. The reaction was cooled, poured onto ice, and extracted twice with ethyl acetate. The combined organic extracts were washed with brine, dried over magnesium sulfate and carbon black, filtered and concentrated under reduced pressure to a light orange liquid. This material was subjected to flash chromatography on

silica gel. A small amount of unreacted starting material was eluted first with 5% ethyl acetate in hexanes. The product was next collected with 10% ethyl acetate in hexanes to give 7.34 g (52%) of a clear liquid which crystallized upon standing, mp 37-40°; ir (film): 3480, 2955, 2840, 1595, 1500, 1440, 1415, 1320, 1290, 1245, 1090, 1015, 800, 775 cm^{-1}; ^1H nmr (deuteriochloroform): δ 7.58 (d, J = 9 Hz, 1 H), 6.07 (d, J = 9 Hz, 1 H), 5.15 (br s, 1 H), 4.61 (t, J = 6 Hz, 1 H), 3.96 (s, 3 H), 3.72 (m, 2 H), 3.48 (s, 6 H); eims: m/z (relative intensity) 280 (12.6), 249 (13.6), 217 (5.9), 185 (9.6), 158 (9.5), 75 (100).

Anal. Calcd. for $C_{11}H_{15}F_3N_2O_3$: C, 47.17; H, 5.40; N, 10.00. Found: C, 47.06; H, 5.49; N, 9.99.

((6-Methoxy-3-trifluoromethyl-2-pyridinyl)amino)acetaldehyde (**16**).

A mixture of 5.6 g (20 mmoles) of *N*-(2,2-dimethylethyl)-6-methoxy-3-trifluoromethyl-2-pyridinamine (**15**) in 60 ml of methanol and 30 ml of 1*N* hydrochloric acid was heated at 60° for 12 hours. After cooling and concentration under reduced pressure, water was added and the mixture made basic with saturated sodium bicarbonate. Following extraction twice with ethyl acetate, the organic extracts were washed with brine, dried over magnesium sulfate, filtered and concentrated under reduced pressure to a yellow oil. Flash chromatography on silica gel (3:1 hexanes/ethyl acetate) and drying in a vacuum oven at room temperature gave 3.31 g (71%) of a white solid, mp 62-64°; ir (nujol): 3450, 1725, 1595, 1500, 1320, 1255, 1145, 1095, 1015, 970, 805 cm^{-1}; ^1H nmr (deuteriochloroform): δ 9.78 (s, 1 H), 7.66 (d, J = 8 Hz, 1 H), 6.17 (d, J = 8 Hz, 1 H), 5.68 (br s, 1 H), 4.27 (d, J = 5 Hz, 2 H), 4.88 (s, 3 H); eims: m/z (relative intensity) 234 (30.2), 206 (47.1), 205 (100), 185 (72.8), 177 (13.2), 161 (16.2), 158 (88.0), 142 (13.5), 138 (34.4), 133 (14.2), 114 (18.6).

Anal. Calcd. for $C_9H_9F_3N_2O_2$: C, 46.16; H, 3.87; N, 11.97. Found: C, 45.89; H, 3.87; N, 11.82.

2-Hydroxy-3-((6-methoxy-3-trifluoromethyl-2-pyridinyl)amino)propanenitrile (**17**).

A 1M solution of diethylaluminum cyanide in toluene (15 ml, 15 mmoles) was added dropwise over 3 minutes to 2.27 g (9.6 mmoles) of ((6-methoxy-3-trifluoromethyl-2-pyridinyl)amino)acetaldehyde (**16**) in 30 ml of toluene with ice-bath cooling. After stirring for 10 minutes, the reaction was poured onto ice, ethyl acetate added, and the mixture filtered through Celite. The organic layer was washed with saturated sodium bicarbonate, brine, dried over magnesium sulfate and carbon black, filtered and concentrated under reduced pressure. Flash chromatography of the residue on silica gel, eluting with 4:1 hexanes/ethyl acetate gave 1.77 g (71%) of a light yellow solid, mp 99-101°; ir (nujol): 3440, 3400, 1595, 1520, 1420, 1315, 1280, 1250, 1150, 1130, 1105, 1065, 1010, 815, 780 cm^{-1}; ^1H nmr (deuteriochloroform): δ 7.65 (d, J = 8 Hz, 1 H), 6.19 (d, J = 8 Hz, 1 H), 5.50 (br s, 1 H), 5.25 (d, J = 6 Hz, 1H), 4.78 (m, 1 H), 4.00 (m, 2H), 3.90 (s, 3 H); eims: m/z (relative intensity) 261 (19.0), 206 (16.2), 205 (100), 185 (62.9), 158 (48.1), 149 (8.1), 138 (13.8).

Anal. Calcd. for $C_{10}H_{10}F_3N_3O_2$: C, 45.98; H, 3.86; N, 16.09. Found: C, 46.12; H, 3.96; N, 15.93.

2-Hydroxy-3-((6-methoxy-3-trifluoromethyl-2-pyridinyl)amino)propanenitrile, 4-Methylbenzenesulfonate (**18**).

To a solution of 2.0 g (7.7 mmoles) of 2-hydroxy-3-((6-methoxy-3-trifluoromethyl-2-pyridinyl)amino)propanenitrile (**17**) and 1.5 ml (8.4 mmoles) of diisopropylethylamine in 50 ml of dichloromethane was added 1.61 g (8.4 mmoles) of *p*-toluenesulfonyl chloride with ice-bath cooling. After stirring at room temperature for 12 hours, water and ethyl acetate were added and the layers separated. The organic layer was washed with 1N hydrochloric acid, water, saturated sodium bicarbonate, brine, dried over magnesium sulfate, filtered and concentrated under reduced pressure to a yellow solid. Recrystallization from 1:1 ethyl acetate/hexanes gave 2.55 g (80%) of a white powder, mp 148-150°; ir (nujol): 3470, 1595, 1315, 1290, 1250, 1190, 1175, 1125, 1090, 1010, 900, 805, 760 cm^{-1}; ^1H nmr (deuteriochloroform): δ 7.86-7.19 (m, 5 H), 6.14 (d, J = 9 Hz, 1 H), 5.48 (t, J = 6 Hz, 1 H), 5.20 (br s, 1 H), 3.98 (m, 2 H), 3.88 (s, 3 H), 2.38 (s, 3 H); eims: m/z (relative intensity) 415 (6.6), 206 (9.0), 205 (100), 185 (26.7), 158 (13.4), 138 (3.6).

Anal. Calcd. for $C_{17}H_{16}F_3N_3O_4S$: C, 49.15; H, 3.88; N, 10.12. Found: C, 49.17; H, 4.08; N, 9.95.

5-Oxo-8-trifluoromethyl-1,2,3,5-tetrahydroimidazo[1,2-*a*]pyridine-3-carbonitrile (**3**).

A solution of 2.3 g (5.5 mmoles) of 2-hydroxy-3-((6-methoxy-3-trifluoromethyl-2-pyridinyl)amino)propanenitrile, 4-methylbenzenesulfonate (**18**) in 60 ml of acetonitrile was heated at reflux for 24 hours. After cooling and concentration under reduced pressure, water and ethyl acetate were added, and the layers separated. The organic layer was washed with saturated sodium bicarbonate and brine, dried over magnesium sulfate, filtered and concentrated under reduced pressure to a yellow, partially crystalline oil. Flash chromatography on silica gel (1:1 ethyl acetate/hexanes) gave 1.10 g (87%) of a light tan powder, mp 162-164°; ir (nujol): 3260 (br), 1670, 1530, 1325, 1250, 1200, 1145, 1070, 795 cm^{-1}; ^1H nmr (deuteriochloroform with 2 drops DMSO-d_6) δ 7.30 (d, J = 9 Hz, 1 H), 7.18 (br s, 1 H), 5.82 (d, J = 9 Hz, 1 H), 5.34 (t, J = 7 Hz, 1 H), 4.03 (d, J = 7 Hz, 2 H); eims: m/z (relative intensity) 229 (100), 210 (17.5), 208 (11.5), 201 (33.9), 182 (10.4), 181 (25.9), 155 (14.5), 154 (33.6), 107 (28.6).

Anal. Calcd. for $C_9H_6F_3N_3O$: C, 47.17; H, 2.64; H, 18.34. Found: C, 46.91; H, 2.61; N, 18.53.

Acknowledgment.

Susan Wollowitz of DowElanco, Walnut Creek, CA is thanked for many helpful discussions and suggestions.

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