Supporting Information

Synthesis of Disubstituted Imidazo[4,5-*b*]pyridin-2-ones

Jeffrey T. Kuethe, Audrey Wong, and Ian W. Davies*

Department of Process Research, Merck & Co., Inc., P.O. Box 2000, rahway, NJ 070765

Jeffrey_Kuethe@merck.com

Melting points are uncorrected. All solvents and reagents were used as received from commercial sources. Analytical samples were obtained by chromatography on silica gel using an ethyl acetate-hexane mixture as the eluent unless specified otherwise. Water content (KF) was determined by Karl Fisher titration on a Metrohm 737 KF coulometer.

Table of Contents

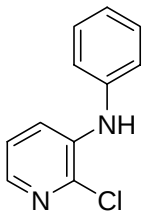
Preparation of 2-Chloro-3-iodopyridine (4). In a 12 L flask was charged 900 mL of 5N HCl and 101.30 g (0.79 mol) of 3-amino-2-chloropyridine. The mixture was cooled to -5 °C and 81.6 g (1.18 mol) of sodium nitrite in 350 mL of water was added dropwise while maintaining the internal temperature below 5 °C. After 10 min, 288.50 g (1.74 mol) of KI in 350 mL of water was added dropwise at -5 °C while maintaining the internal temperature below 10 °C over the course of the addition. The reaction mixture was warmed to rt and 1.50 L of EtOAc was added. The pH of the aqueous layer was adjusted to 11 by the addition of 650 mL of 6N NaOH, the layers were separated, and the organic layer was washed with 1.50 L of 0.3M $Na_2S_2O_3$. The EtOAc layer was concentrated and the residue re-dissolved in 500 mL of DMF. To the reddish/brown mixture was added dropwise 1.50 L of water and the slurry stirred for 30 min and filtered. The filter cake was washed with water (2 X 500 mL) and then dried under vacuum/N_2 sweep to provide 173.3 g (92%) of **4** where the spectroscopic properties were identical to that reported in the literature.[1]

General Procedure for the Preparation of 3-amino-2-chloropyridines: To a slurry of 3.00 g (12.5 mmol) of 2-chloro-3-iodopyridine (**4**) in 31 mL of toluene was added sequentially 10.0 mg (0.376 mmol) of $Pd(OAc)_2$, 84.0 mg (0.376 mmol) of *rac*-BINAP, 1.37 g (62.5 mmol) of solid Cs_2CO_3, 11.9 mmol of the appropriate amine, and 75 mg (0.752 mmol) of triethylamine. The resulting slurry was degassed (2X) by vacuum/N_2 backfills. The mixture was heated to reflux and monitored by TLC (EtOAc/hexane) for the disappearance of starting materials. After 18 h the reaction mixture was cooled to rt and 30 mL of H_2O was added. The layers were separated and the toluene layer was

concentrated under reduced pressure. The residue could be used crude without further purification in the next reaction or purified by silica gel chromatography.

General Procedure for the Preparation of Unsymmetrically Substituted Diamines: To 3.00 mmol of the appropriate 3-amino-2-chloropyridine in 12 mL of toluene was added sequentially 41.0 mg (0.0450 mmol) of $Pd_2(dba)_3$, 84.0 mg (0.135 mmol) of *rac*-BINAP, 404 mg (4.20 mmol) of NaO*t*Bu, and 3.60 mmol of the appropriate aniline. The reaction was degassed (2X) by vacuum/N_2 backfills. The mixture was heated to reflux and monitored by TLC (25% EtOAc/hexane) for the disappearance of starting materials. After 18 h the reaction mixture was cooled to rt and concentrated under reduced pressure. The residue could be used crude without further purification in the next reaction or purified by silica gel chromatography.

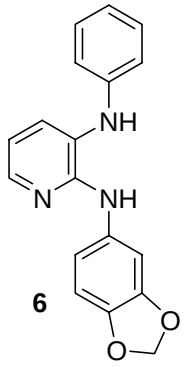
General Procedure for the Preparation of Imidazo[4,5-*b*]pyridin-2-ones: To a solution of 1.0 mmol of the appropriate diamine in 6.7 mL of EtOAc or THF was added 2.5 mmol of triethylamine and 0.4 mmol of triphosgene. The resulting slurry was stirred at rt for 30 min and 7.0 mL of sat $NaHCO_3$ was added. The layers were separated and the organic layer was concentrated under reduced pressure.. The residue was purified by silica gel chromatography or recrystallized from an ethyl acetate/hexane mixture.



5

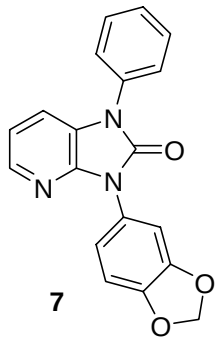
Preparation of 2-Chloro-*N*-phenylpyridin-3-amine (5). According to the general procedure for the preparation of 3-amino-2-chloropyridines, reaction of 7.00 g (29.23

mmol) of **4** with 2.63 g (28.25 mmol) of aniline gave 4.91 g (85%) of **5** which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.15 (br s, 1H), 7.07-7.18 (m, 4H), 7.37 (t, 2H, *J* = 7.7 Hz), 7.49 (d, 1H, *J* = 8.1 Hz), 7.87 (d, 1H, *J* = 4.5 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 121.2, 121.3, 123.9, 124.0, 129.7, 137.8, 139.4, 140.1, 151.8.



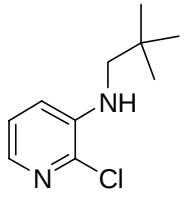
6

Preparation of N^2-1,3-benzodioxol-5-yl-N^3-phenylpyridin-2,3-diamine (6). According to the general procedure for the preparation of unsymmetrically substituted diamines, treatment of 2.20 g (10.8 mmol) of **5** with 1.77 g (12.9 mmol) of 3,4-(methylenedioxy)aniline afforded 2.63 g (80%) of **6** as an oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.22 (br s, 1H), 5.91 (s, 2H), 6.73 (m, 6H), 6.92 (t, 1H, *J* = 7.4 Hz), 7.26 (m, 3H), 7.39 (dd, 1H, *J* = 7.6 and 1.4 Hz), 8.06 (dd, 1H, *J* = 4.9 and 1.6 Hz); ^{13}C NMR ($CDCl_4$, 100 MHz) δ 101.1, 103.0, 108.2, 112.9, 114.9, 115.9, 120.5, 124.8, 129.6, 131.8, 135.0, 143.0, 144.1, 144.8, 147.8, 152.3; Anal. Calcd. For $C_{13}H_{15}N_3O_2$: C, 70.81; H, 4.95; N, 13.76. Found: C, 70.55; H, 5.03; N, 13.88.



7

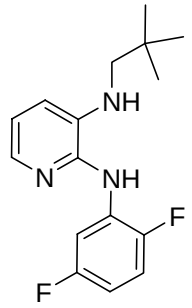
Preparation of 3-(1,3-benzodioxol-5-yl)-1-phenyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (7). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 1.50 g (4.91 mmol) of diamine **6** with 583 mg (1.97 mmol) of triphosgene gave 1.58 g (97%) of **7** as a colorless solid: mp 175-176 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.03 (s, 2H), 6.96 (d, 1H, *J* = 7.7 Hz), 7.05 (dd, 1H, *J* = 7.7 and 5.2 Hz), 7.20 (m, 2H), 7.35 (d, 1H, *J* = 7.7 Hz), 7.44 (m, 1H), 7.57 (m, 4H), 8.11 (d, 1H, *J* = 5.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 101.8, 107.9, 108.6, 115.0, 117.9, 120.3, 123.8, 125.7, 126.7, 128.1, 129.8, 133.9, 141.7, 143.7, 147.4, 148.2, 151.9; Anal. Calcd. For $C_{19}H_{13}N_3O_3$: C, 68.88; H, 3.95; N, 12.68. Found: C, 68.77; H, 3.86; N, 12.73.



8

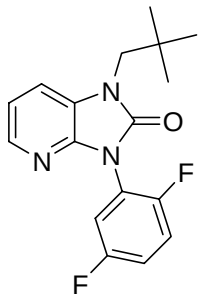
2-Chloro-*N*-(2,2-dimethylpropyl)pyridin-3-amine (8). According to the general procedure for the preparation of 3-amino-2-chloropyridines, reaction of 5.00 g (20.9 mmol) of **4** with 1.73 g (19.8 mmol) of neopentyl amine afforded 2.96 g (75%) of **8** which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.04 (s, 9H), 2.94 (d, 2H, *J* = 6.0 Hz), 4.42 (br s, 1H), 6.91 (dd, 1H, *J* = 8.0 and

1.6 Hz), 7.08 (dd, 1H, *J* = 8.0 and 4.4 Hz), 7.68 (dd, 1H, *J* = 4.8 and 1.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 27.6, 32.1, 50.6, 117.2, 123.4, 135.9, 137.0, 141.5.



9

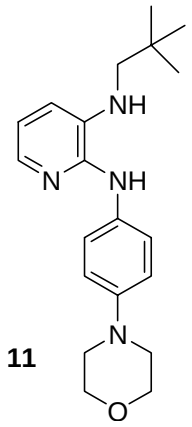
***N*^{2}-(2,5-Difluorophenyl)-*N*^{3}-(2,2-dimethylpropyl)pyridine-2,3-diamine (9).** According to the general procedure for the preparation of unsymmetrically substituted diamines, treatment of 500 mg (2.52 mmol) of **8** with 320 mg (2.5 mmol) of 2,5-difluoroaniline gave 462 mg (63%) of **9** which was used in the next step without further purification. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.04 (s, 9H), 2.86 (d, 2H, *J* = 6.4 Hz), 3.26 (br t, 1H, *J* = 6.0 Hz), 6.54 (m, 1H), 6.90 (dd, 1H, *J* = 8.0 and 4.8 Hz), 7.02 (m, 2H), 7.34 (br s, 1H), 7.78 (m, 1H), 7.84 (dd, 1H, *J* = 4.8 and 1.6 Hz).



10

3-(2,5-Difluorophenyl)-1-(2,2-dimethylpropyl)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (10). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 730 mg (2.51 mmol) of diamine **9** with 430 mg (1.45 mmol) of triphosgene yielded 572 mg (72%) of **10** as a yellow crystalline solid:

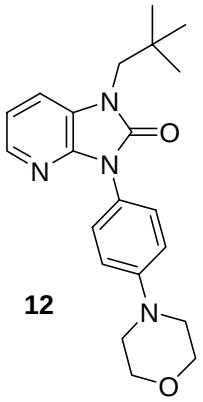
mp 149-150 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.10 (s, 9 H), 3.74 (s, 2H), 7.09 (dd, 1H, *J* = 7.6 and 5.2 Hz), 7.16 (m, 1H), 7.29 (m, 3H), 8.05 (dd, 1H, *J* = 5.2 and 1.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 28.2, 34.6, 53.2, 115.3, 116.9 (d, *J* = 25.7 Hz), 117.0 (dd, *J* = 24.1 and 8.0 Hz), 117.7 (dd, *J* = 22.5 and 8.8 Hz), 118.0, 121.6 (dd, *J* = 15.4 and 10.4 Hz), 125.6, 140.8, 142.9, 152.8, 154.4 (dd, *J* = 249.0 and 2.4 Hz), 158.4 (dd, *J* = 247.4 and 2.8 Hz). ^{19}F NMR ($CDCl_3$, 376 MHz) δ -124.0 (d, *J* = 15.5 Hz), -177.4 (d, *J* = 15.5 Hz); Anal. Calcd. For $C_{17}H_{17}F_2N_3O$: C, 64.34; H, 5.40; N, 13.24. Found: C, 64.20; H, 5.19; N, 13.13.



11

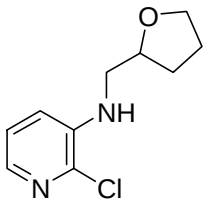
***N*^{3}-(2,2-Dimethylpropyl)-*N*^{2}-(4-morpholin-4-ylphenyl)pyridine-2,3-diamine (11).** According to the general procedure for the preparation of unsymmetrically substituted diamines, treatment of 450 mg (2.26 mmol) of **8** with 540 mg (3.00 mmol) of 4-morpholinoaniline gave 520 mg (67%) of **11** which was used in the next step without further purification. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 0.97 (s, 9H), 2.83 (d, 2H, *J* = 5.2 Hz), 3.09 (m, 4H), 3.32 (br t, 1H, *J* = 5.6 Hz), 3.87 (m, 4H), 6.13 (br s, 1H), 6.81 (dd, 1H, *J* = 7.8 and 5.0 Hz), 6.90 (m, 2H), 6.93 (dd, 1H, *J* = 7.8 and 1.4 Hz), 7.15 (m, 2H), 7.72 (dd,

1H, *J* = 5.0 and 1.4 Hz);); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 27.7, 31.7, 50.6, 56.1, 67.1, 117.2, 117.3, 118.3, 120.6, 134.5, 135.0, 136.8, 146.0, 146.8.



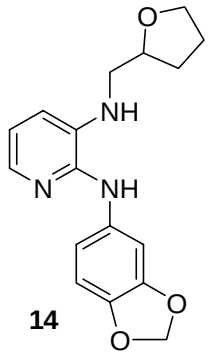
12

1-(2,2-Dimethylpropyl)-3-(4-morpholin-4-ylphenyl)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (12). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 380 mg (1.11 mmol) of diamine **11** with 200 mg (0.674 mmol) of triphosgene gave 370 mg (90%) of **12** as a white solid: mp 172 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.09 (s, 9H), 3.22 (m, 4H), 3.73 (s, 2H), 3.90 (m, 4H), 7.03 (dd, 1H, *J* = 8.0 and 5.2 Hz), 7.07 (m, 2H), 7.27 (dd, 1H, *J* = 7.8 and 1.4 Hz), 7.59 (m, 2H), 8.05 (dd, 1H, *J* = 5.0 and 1.4 Hz); ^{13}C ($CDCl_3$, 100 MHz) δ 28.3, 34.6, 49.4, 53.0, 66.8, 114.6, 116.2, 116.3, 117.2, 125.3, 126.9, 140.6, 143.6, 150.4, 153.8; Anal. Calcd. For $C_{21}H_{26}N_4O_2$: C, 68.83; H, 7.15; N, 15.29. Found: C, 68.43; H, 7.04; N, 15.05.



13

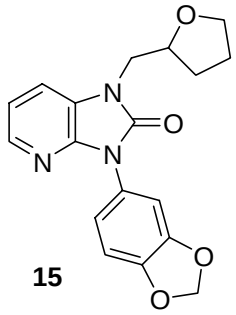
2-Chloro-*N*-(tetrahydrofuran-2-ylmethyl)pyridin-3-amine (13). According to the general procedure for the preparation of 3-amino-2-chloropyridines, reaction of 5.00 g (20.9 mmol) of **4** with 2.01 g (19.9 mmol) of tetrahydrofurfuryl amine afforded 3.12 g (74%) of **13** as a yellow oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.68 (m, 1H), 1.06 (m, 2H), 2.07 (m, 1H), 3.15 (m, 1H), 3.30 (m, 1H), 3.81 (m, 1H), 3.92 (m, 1H), 4.16 (m, 1H), 4.68 (br t, 1H, *J* = 6.0 Hz), 6.92 (dd, 1H, *J* = 8.0 and 1.6 Hz), 7.08 (dd, 1H, *J* = 8.0 and 4.4 Hz), 7.72 (dd, 1H, *J* = 4.8 and 1.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 25.9, 29.2, 47.4, 68.4, 77.3, 117.5, 123.3, 136.5, 137.4, 141.1. HRMS (EI) calcd for (M + H^+) $C_{10}H_{13}ClNO$: 213.0795. Found: 213.0795.



14

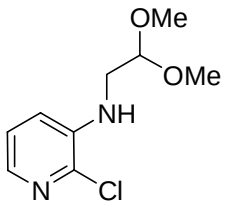
***N*^{2}-1,3-Benzodioxol-5-yl-*N*^{3}-(tetrahydrofuran-2-ylmethyl)pyridin-2,3-diamine (14).** According to the general procedure for the preparation of unsymmetrically substituted diamines, reaction of 500 mg (2.35 mmol) of **13** with 310 mg (2.30 mmol) of 3,4-(methylenedioxy)aniline gave 574 mg (78%) of **14** which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.67 (m, 1H), 1.95 (quint, 2H, *J* = 6.8 Hz), 2.06 (m, 1H), 3.05 (dd, 1H, *J* = 12.0 and 7.6 Hz), 3.19 (dd, 1H, *J* = 12.2 and 3.4 Hz), 3.68 (br s, 1H), 3.81 (m, 1H), 3.89 (m, 1H), 4.18 (m, 1H), 5.93 (s, 2H), 6.34 (br s, 1H), 6.66 (dd, 1H, *J* = 8.4 and 2.0 Hz), 6.74 (d, 1H, *J* = 8.0 Hz), 6.77 (dd, 1H, *J* = 7.6 and 5.2 Hz), 6.94 (dd, 1H, *J* = 7.6 and 1.6 Hz), 7.04 (d, 1H, *J* = 2.4 Hz), 7.77 (dd, 1H, *J*

= 4.8 and 1.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 25.9, 29.2, 48.9, 68.2, 77.5, 101.0, 102.5, 108.2, 112.1, 116.5, 119.3, 133.0, 136.1, 137.8, 142.6, 146.8, 147.9.



15

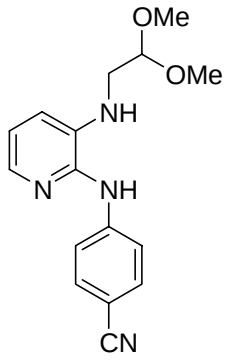
3-(1,3-Benzodioxol-5-yl)-1-(tetrahydrofuran-2-yl-methyl)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (15). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 740 mg (2.36 mmol) of diamine **14** with 280 mg (0.943 mmol) of triphosgene gave 270 mg (65%) of **15** as an oil: ^{1}H NMR ($CDCl_3$, 400MHz) δ 1.67 (m, 1H), 1.81 (quint, 2H), 2.00 (m, 1H), 3.66 (q, 1H, *J* = 7.4 Hz), 3.76 (q, 1H, *J* = 6.8 Hz), 3.85 (dd, 1H, *J* = 14.9 and 3.8 Hz), 4.03 (m, 1H), 4.20 (m, 1H), 5.92 (s, 2H), 6.85 (d, 1H, *J* = 8.8 Hz), 6.96 (dd, 1H, *J* = 7.63 and 5.2 Hz), 7.06 (m, 2H), 7.38 (dd, 1H, *J* = 7.6 and 1.2 Hz), 7.95 (dd, 1H, *J* = 5.2 and 1.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 25.8, 28.8, 45.4, 68.2, 77.8, 101.7, 107.7, 108.3, 115.5, 117.8, 120.0, 124.5, 127.0, 140.8, 143.4, 147.1, 148.0, 153.1; HRMS (EI) calcd for ($M + H^+$) for 340.1295. Found: 340.1297.



16

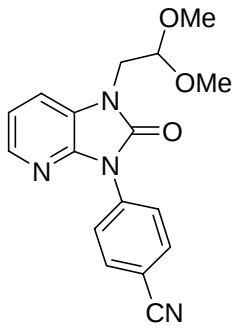
2-Chloro-*N*-(2,2-dimethoxyethyl)pyridin-3-amine (16). According to the general procedure for 3-amino-2-chloropyridines, reaction of 5.00 g (20.9 mmol) of **4** with 2.08 g

(19.9 mmol) of aminoacetaldehyde dimethyl acetal afforded 3.18 g (74%) of **16** as a yellow oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.29 (t, 2H, *J* = 5.6 Hz), 3.45 (s, 6H), 4.58 (br s, 1H), 4.59 (t, 1H, *J* = 5.4 Hz), 6.93 (dd, 1H, *J* = 7.6 and 1.4 Hz), 7.10 (dd, 1H, *J* = 8.0 and 4.8 Hz), 7.74 (dd, 1H, *J* = 4.8 and 1.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 45.0, 54.2, 102.5, 117.7, 123.4, 136.7, 137.4, 140.6; Anal. Calcd. for $C_9H_{13}ClN_2O_2$: C, 49.89; H, 6.05; N, 12.93. Found: C, 50.22; H, 5.80; N, 12.71.



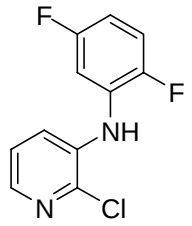
17

4-({3-[(2,2-Dimethoxyethyl)amino]pyridin-2-yl}amino)benzonitrile (17). According to the general procedure for preparation of unsymmetrically substituted diamines, treatment of 600 mg (2.77mmol) of **16** with 340 mg (2.88 mmol) of 4-aminobenzonitrile gave 719 mg (87%) of **17** which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400MHz) δ 3.20 (t, 2H, *J* = 5.6 Hz), 3.40 (s, 6H), 3.72 (br t, 1H, *J* = 5.6 Hz), 4.56 (t, 1H, *J* = 5.2 Hz), 6.90 (dd, 1H, *J* = 8.0 and 4.8 Hz), 6.92 (br s, 1H), 7.00 (dd, 1H, *J* = 7.6 and 1.2 Hz), 7.30 (dt, 2H, *J* = 8.8 and 2.2 Hz), 7.45 (dt, 2H, *J* = 8.8 and 2.2 Hz), 7.81 (dd, 1H, *J*= 4.8 and 1.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 46.0, 54.2, 102.6, 102.8, 117.4, 119.1, 120.0, 120.4, 133.2, 134.3, 137.7, 143.9, 146.1.



18

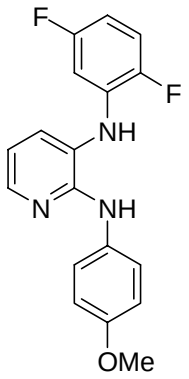
4-[1-(2,2-Dimethoxyethyl)-2-oxo-1,2-dihydro-3*H*-imidazo[4,5-*b*]pyridin-3-yl]benzonitrile (18). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 830 mg (2.77 mmol) of **17** with 330 mg (1.11 mmol) of triphosgene gave 720 mg (80%) of **18** as an off-white solid. mp 113-114 °C, ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.46 (s, 6H), 4.06 (d, 2H, *J* = 4.8 Hz), 4.65 (t, 1H, *J* = 4.8 Hz), 7.14 (dd, 1H, *J* = 8.0 and 5.2 Hz), 7.46 (dd, 1H, *J* = 7.8 and 1.2 Hz), 7.81 (dt, 2H, *J* = 9.2 and 2.0 Hz), 8.09 (dd, 1H, *J* = 5.0 and 1.6 Hz), 8.12 (dt, 2H, *J* = 8.8 and 2.0 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 43.6, 55.3, 102.9, 110.4, 116.0, 118.5, 118.8, 124.6, 125.3, 133.0, 138.0, 141.0, 142.3, 152.2; Anal. Calcd. for $C_{17}H_{16}N_4O_3$: C, 62.95; H, 4.95; N, 17.27. Found: C, 62.84; H, 4.80; N 17.14.



19

2-Chloro-*N*-(2,5-difluorophenyl)pyridin-3-amine (19). According to the general procedure for the preparation of 3-amino-2-chloropyridines, reaction of 700 mg (2.9 mmol) of **4** with 370 mg (2.87mmol) of 2,5-difluoroaniline afforded 580 mg (84%) of **19** as a white solid: mp 109-110 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.22 (br s, 1H), 6.69 (m,

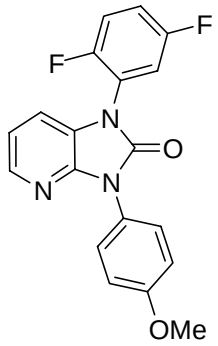
1H), 6.99 (m, 1H), 7.10 (m, 1H), 7.18 (m, 1H), 7.54 (dd, 1H, *J* = 8.0 and 1.2 Hz), 7.9 (dd, 1H, *J* = 4.8 and 1.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 106.3 (d, *J* = 27.3 Hz), 109.1 (dd, *J* = 24.1 and 7.2 Hz), 116.6 (dd, *J* = 22.1 and 10.0 Hz), 123.1, 123.3, 130.0 (dd, *J* = 12.8 and 11.2 Hz), 135.6, 140.4, 141.2, 150.3 (dd, *J* = 240.1 and 2.4 Hz), 158.9 (dd, *J* = 242.8 and 2.4 Hz); ^{19}F NMR ($CDCl_3$, 376 MHz) δ -135.0 (d, *J* = 15.5 Hz), -117.0 (d, *J* = 15.5 Hz). Anal. Calcd. for $C_{11}H_7ClF_2N_2$: C, 54.90; H, 2.93; N, 11.64. Found: C, 54.81; H, 2.74; N, 11.47.



20

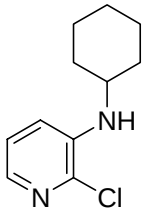
***N*^{3}-(2,5-Difluorophenyl)-*N*^{2}-(4-methoxyphenyl)pyridine-2,3-diamine (20).** According to the general procedure for the preparation of unsymmetrically substituted diamines, treatment of 250 mg (1.04 mmol) of **19** with 130 mg (2.5 mmol) of *p*-anisidine provided 190 mg (56%) of **20** as a fluffy white solid: mp 116-117 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.79 (s, 3H), 5.47 (br s, 1H), 6.38 (m, 1H), 6.47 (m, 1H), 6.74 (dd, 1H, *J* = 7.6 and 4.8 Hz), 6.78 (br s, 1H), 6.87 (m, 2H), 7.03 (m, 1H), 7.39 (dd, 1H, *J* = 7.8 and 1.2 Hz), 7.43 (m, 2H), 8.12 (dd, 1H, *J* = 4.8 and 1.6 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 55.6, 102.5 (dd, *J* = 28.9 and 2.1 Hz), 105.3 (dd, *J* = 24.0 and 7.2 Hz), 114.3 114.6, 115.7 (dd, *J* = 20.9 and 10.4 Hz), 121.9, 122.3, 133.3, 133.5, 124.9 (dd, *J* = 11.4 and 12.8), 145.6, 148.3 (d, *J* = 235.0 Hz), 153.2, 155.6, 159.5 (d, *J* = 240.9 Hz); ^{19}F NMR ($CDCl_3$,

376 MHz) δ -140.9 (d, *J* = 15.5 Hz), -117.3 (d, *J* = 17.2 Hz); Anal. Calcd for $C_{18}H_{15}F_2N_3O$: C, 66.05; H, 4.62; N, 12.84. Found: C, 65.82; H, 4.45; N 12.60.



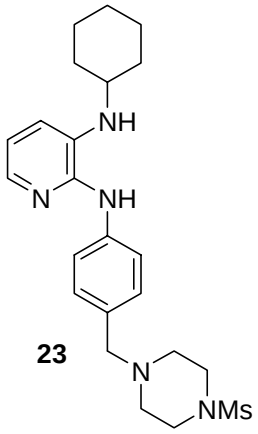
21

1-(2,5-Difluorophenyl)-3-(4-methoxyphenyl)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (21). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 160 mg (0.49 mmol) of **20** with 58.0 mg (0.195 mmol) of triphosgene gave 150 mg (88%) of **21** as a fluffy white solid: mp 172-173 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.88 (s, 3H), 7.08 (m, 3H), 7.19 (m, 2H), 7.31 (m, 1H), 7.38 (m, 1H), 7.64 (m, 2H), 8.15 (dd, 1H, *J* = 5.0 and 1.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 55.6, 114.7, 115.4, 116.3 (d, *J* = 25.7 Hz), 117.1 (dd, *J* = 23.7 and 7.6 Hz), 117.99 (dd, *J* = 22.9 and 9.2 Hz), 118.01, 123.4, 125.6, 127.6, 137.2, 142.1, 143.7, 151.2, 153.8, (dd, 249.0 and 3.2 Hz), 158.5 (dd, *J* = 248.0 and 2.4 Hz), 159.3; ^{19}F NMR ($CDCl_3$, 376 MHz) δ -123.9 (d, *J* = 15.5), -116.4 (d, *J* = 15.5 Hz). Anal. Calcd. for $C_{19}H_{13}F_2N_3O_2$: C, 64.59; H, 3.71; N, 11.89. Found: C, 64.38; H, 3.39; N, 11.81.



22

Preparation of 2-Chloro-*N*-cyclohexylpyridin-3-amine (22). According to the general procedure for the preparation of 3-amino-2-chloropyridines, reaction of 8.00 g (33.4 mmol) of **4** with 3.14 g (31.7 mmol) of cyclohexylamine gave 5.67 g (85%) of **22** as a colorless solid: mp 44-45 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.26 (m, 5H), 1.63 (m, 1H), 1.75 (m, 2H), 2.02 (m, 2H), 3.24 (m, 1H), 4.25 (br s, 1H), 6.86 (dd, 1H, *J* = 8.1 and 1.5 Hz), 7.04 (dd, 1H, *J* = 8.1 and 4.6 Hz), 7.64 (dd, 1H, *J* = 4.6 and 1.5 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.7, 25.7, 32.8, 51.1, 117.6, 123.3, 135.7, 136.9, 139.9; Anal. Calcd. For $C_{11}H_{15}ClN_2$: C, 62.70; H, 7.18; N, 13.30. Found: C, 63.00; H, 7.28; N, 13.14.



23

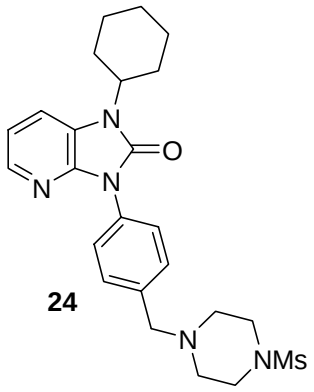
Preparation of *N*^{3}-Cyclohexyl-*N*^{2}-(4-{[4-(methylsulfonyl)piperazin-1-yl]methyl}phenyl)pyridine-2,3-diamine (23). A solution of 1.00 g (4.63 mmol) of 4-nitrobenzyl bromide in 5 mL of isopropyl acetate was added dropwise over a period of 20 min to a stirred solution of 4 g (46.4 mmol) of piperazine in 8 mL of 95% EtOH. The resulting suspension was allowed to stir at rt for 30 min, filtered over a pad of celite and concentrated under reduced pressure. The residue was dissolved in 30 mL of isopropyl acetate and washed with 25 mL of water. The isopropyl acetate layer was separated and concentrated to a final volume of 20 mL. To the solution was added 610 mg (6.01 mmol)

of NEt_3 followed by 560 mg (4.86 mmol) of methanesulfonyl chloride. After stirring for 30 min the reaction mixture was filtered over a pad of celite and concentrated to a final volume of 20 mL. To the resulting mixture was added 20 mL of heptane and the slurry of the product was stirred for 20 min and filtered affording 900 mg (65%) of 1-methanesulfonyl-4-(4-nitrobenzyl)-piperazine as a colorless solid: mp 116-117 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.58 (m, 4H), 2.80 (s, 3H), 3.27 (m, 4H), 3.65 (s, 2H), 7.51 (d, 2H, *J* = 8.8 Hz), 8.18 (d, 2H, *J* = 8.8 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 34.5, 46.0, 52.6, 61.8, 123.8, 129.6, 145.7, 147.5; Anal. Calcd. For $C_{12}H_{17}N_3O_4S$: C, 48.15; H, 5.72; N, 14.04. Found: C, 48.16; H, 5.52; N, 13.91.

To a solution of 25.0 g (83.5 mmol) of the above nitrocompound in 1.50 L of EtOAc was added 5.00 g of 5% Pd/C. The resulting mixture stirred at rt under an atmosphere of hydrogen (15 psi) for 4 h. The reaction mixture was filtered over a pad of celite and concentrated to a solid under reduced pressure to give 19.0 g (84%) of 4-(4-methanesulfonyl-piperazin-1-ylmethyl)aniline as a colorless solid: mp 161-162 ºC; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.52 (m, 4H), 2.76 (s, 3H), 3.22 (m, 4H), 3.42 (s, 2H), 3.67 (br s, 2H), 6.63 (d, 2H, *J* = 8.4 Hz), 7.06 (d, 2H, *J* = 8.4 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz)) δ 34.1, 46.0, 52.1, 62.3, 115.0, 127.2, 130.4, 145.8; Anal. Calcd. For $C_{12}H_{19}N_3O_2S$: C, 53.51; H, 7.11; N, 15.60. Found: C, 53.71; H, 7.09; N, 15.48.

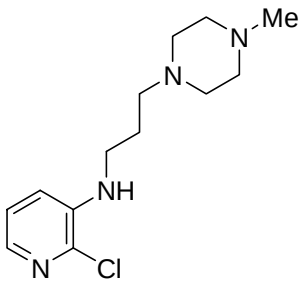
According to the general procedure for the preparation of unsymmetrically substituted diamines, treatment of 250 mg (1.19 mmol) of **22** with 380 mg (1.41 mmol) of the above aniline afforded 504 mg (95%) of **23** as an oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.21 (m, 3H), 1.31 (m, 2H), 1.65 (m, 1H), 1.73 (m, 2H), 1.99 (m, 2H), 2.54 (m, 4H), 2.76 (s, 3H), 3.22 (m, 6H), 3.47 (s, 2H), 6.24 (br s, 1H), 6.82 (m, 1H), 6.95 (dd, 1H, *J* = 7.7 and 1.3

Hz), 7.19 (s, 4H), 7.75 (dd, 1H, *J* = 4.9 and 1.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.8, 25.9, 33.2, 34.1, 45.9, 52.0, 51.1, 62.2, 117.6, 118.1, 119.8, 129.9, 133.0, 136.9, 141.1, 145.5.



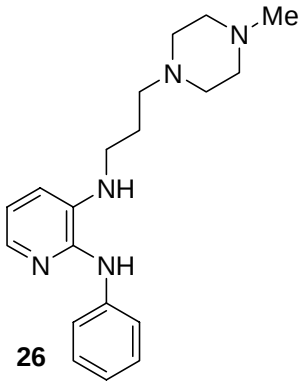
24

Preparation of 1-Cyclohexyl-3-(4-{[4-(methylsulfonyl)piperazin-1-yl]methyl}phenyl)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (24). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 410 mg (0.924 mmol) of **23** with 110 mg (0.370 mmol) of triphosgene gave 410 mg (94%) of **24** as a yellow foam: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.29 (m, 1H), 1.46 (m, 2H), 1.76 (m, 1H), 1.96 (m, 4H), 2.10 (m, 2H), 2.58 (m, 4H), 2.77 (s, 3H), 3.24 (m, 4H), 3.58 (s, 2H), 4.37 (m, 1H), 7.03 (dd, 1H, *J* = 8.0 and 5.2 Hz), 7.45 (m, 3H), 7.65 (d, 2H, *J* = 8.4 Hz), 8.02 (dd, 1H, *J* = 5.2 and 1.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 25.4, 25.9, 30.3, 34.3, 46.0, 52.4, 53.2, 62.2, 115.4, 117.4, 123.0, 126.0, 129.7, 132.7, 136.9, 140.3, 143.3, 152.3; Anal. Calcd. For $C_{24}H_{31}N_5O_3S$: C, 61.38; H, 6.65; N, 14.91. Found: C, 61.57; H, 6.87; N, 14.79.



25

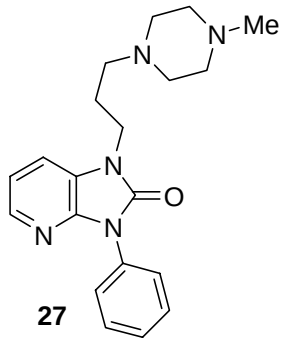
Preaparion of 2-Chloro-*N*-[3-(4-methylpiperazin-1-yl)propyl]pyridin-3-amine (25). According to the general procedure for the preparation of 3-amino-2-chloropyridines, reaction of 8.01 g (33.45 mmol) of **4** with 5.00 g (31.79 mmol) of 1-(3-aminopropyl)-4-methylpiperazine gave 7.09 g (83%) of **25** as an oil which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.83 (m, 2H), 2.28 (s, 3H), 2.49 (m, 10H), 3.18 (m, 2H), 5.68 (br s, 1H), 6.82 (m, 1H), 7.04 (m, 1H), 7.65 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.8, 43.4, 46.1, 53.5, 54.9, 57.5, 116.9, 123.3, 135.7, 137.1, 141.4.



26

Preparation of N^3-[3-(4-Methylpiperazin-1-yl)propyl]-N^2-phenylpyridine-2,3-diamine (26). According to the general procedure for the preparation of unsymmetrically substituted diamines, treatment of 1.40 g (5.21 mmol) of **25** with 613 mg (6.58 mmol) of aniline afforded 1.34 g (79%) of **26** as a clear oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 185 (m, 2H), 2.11 (s, 3H), 2.50 (m, 10H), 3.18 (t, 2H, J = 6.0 Hz), 5.09 (br s,

1H), 6.03 (br s, 1H), 6.81 (m, 1H), 6.87 (m, 1H), 6.93 (m, 1H), 7.27 (m, 4H), 7.73 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 25.2, 44.5, 45.8, 53.5, 55.1, 58.1, 117.1, 117.4, 118.9, 121.4, 128.9, 134.5, 136.2, 141.5, 145.2.

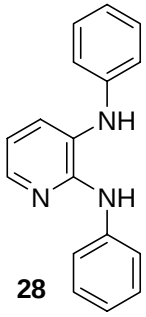


27

Preparation of 1-[3-(4-methylpiperazin-1-yl)propyl]-3-phenyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (27)[2]. According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 1.00 g (3.07 mmol) of **26** with 364 mg (1.23 mmol) of triphosgene gave 993 mg (92%) of **27**[2] as an oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.98 (m, 2H), 2.31 (s, 3H), 2.44 (m, 10H), 4.01 (t, 2H, *J* = 6.8 Hz), 7.04 (dd, 1H, *J* = 7.8 and 5.2 Hz), 7.23 (m, 1H), 7.41 (m, 1H), 7.51 (t, 2H, *J* = 8.0 Hz), 7.73 (m, 2H), 8.04 (dd, 1H, *J* = 5.2 and 1.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 25.3, 39.1, 45.7, 52.6, 54.9, 55.0, 113.9, 117.6, 124.1, 125.7, 127.5, 129.1, 133.4, 140.6, 143.2, 152.6; Anal. Calcd. For $C_{20}H_{25}N_5O$: C, 68.35; H, 7.17; N, 19.93. Found: C, 68.14; H, 6.98; N, 19.92.

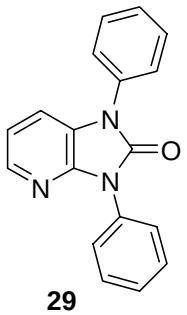
General Procedure for Symmetrically Substituted Diamines: To 3.00 g (12.5 mmol) of 2-chloro-3-iodopyridine (**4**) in 31 mL of toluene was added sequentually 10 mg (0.376 mmol) of $Pd(OAc)_2$, 84 mg (0.376 mmol) of *rac*-BINAP, 1.37 g (62.5 mmol) of Cs_2CO_3, 31.3 mmol of the appropriate aniline, and 75.0 mg (0.752 mmol) of triethylamine. The resulting slurry was degassed (2X) by vacuum/N_2 backfills. The mixture was heated to

reflux and monitored by TLC (25% EtOAc/hexane) for the dissappearance of starting materials. After 18 h the reaction mixture was cooled to rt and 30 mL of H_2O was added. The layers were separated and the toluene layer was concentrated under reduced pressure. The residue could be used crude without further purification in the next step or purified by silica gel chromatography.



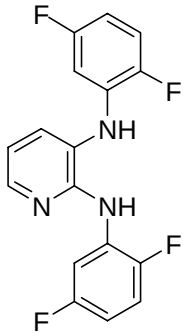
28

Preparation of *N,N'*-Diphenylpyridine-2,3-diamine (28). According to the general procedure for the preparation of symmetrically substituted diamines, reaction of 4.00 g (16.7 mmol) of **4** with 3.85 g (41.4 mmol) of aniline afforded 3.63 g (83%) of **28** as a colorless solid: mp 127-128 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.18 (br s, 1H), 6.81 (m, 3H), 6.94 (m, 2H), 7.01 (t, 1H, *J* = 7.4 Hz), 7.32 (m, 4H), 7.42 (m, 1H), 7.56 (m, 2H), 8.13 (dd, 1H, *J* = 4.9 and 1.5 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 115.2, 116.0, 119.2, 120.5, 121.9, 125.2, 128.9, 129.4, 131.7, 140.6, 143.9, 144.8, 151.8; Anal. Calcd. For $C_{17}H_{15}N_3$: C, 78.13; H, 5.79; H, 16.08. Found: C, 78.35; H, 5.46; N, 16.01.



29

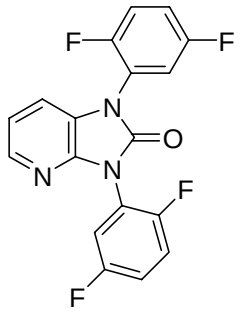
Preparation of 1,3-Diphenyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (29). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 1.50 g (5.74 mmol) of **28** with 681 mg (2.30 mmol) of triphosgene yielded 1.60 g (97%) of **29** as colorless solid: mp 119-120 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.07 (dd, 1H, *J* = 7.8 and 5.2 Hz), 7.38 (dd, 1H, *J* = 7.8 and 1.5 Hz), 7.45 (m, 2H), 7.61 (m, 6H), 7.80 (m, 2H), 8.14 (dd, 1H, *J* = 5.2 and 1.5 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 115.0, 118.0, 123.9, 125.8, 126.2, 127.8, 128.1, 129.3, 129.8, 133.3, 133.9, 141.7, 143.4, 151.8; Anal. Calcd. For $C_{18}H_{13}N_3O$: C, 75.25; H, 4.56; N, 14.63. Found: C, 75.17; H, 4.36; N, 14.50.



30

***N, N*-Bis(2,5-difluorophenyl)pyridine,2,3-diamine** (**30).** According to the general procedure for the preparation of symmetrically substituted diamines, reaction of 700 mg (2.92 mmol) of **4** with 940 mg (7.30 mmol) of 2,5-difluoroaniline gave 867 mg (89%) of

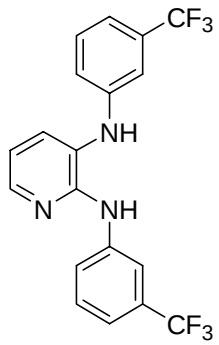
30 which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.42 (br s, 1H), 6.34 (m, 1H), 6.50 (m, 1H), 6.60 (m, 1H), 6.90 (dd, 1H, *J* = 7.6 and 4.8 Hz), 7.00 (m, 1H), 7.06 (m, 1H), 7.37 (br s, 1H), 7.50 (dd, 1H, *J* = 7.6 and 1.6 Hz), 8.24 (dd, 1H, *J* = 5.2 and 1.6 Hz), 8.50 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 102.6 (dd, *J* = 28.7 and 2.4 Hz), 105.9 (dd, *J* = 24.1 and 7.2 Hz), 106.9 (dd, *J* = 24.9 and 7.2 Hz), 107.0 (d, *J* = 30.5 Hz), 114.7 (dd, *J* = 21.7 and 9.6 Hz), 115.9 (dd, *J* = 20.9 and 10.4 Hz), 116.2, 123.2, 129.8 (dd, *J* = 12.0 and 11.2 Hz), 133.9, 134.5 (dd, *J* = 13.7 and 11.2 Hz), 145.4, 148.2 (dd, *J* = 237.7 and 3.2 Hz), 148.3 (dd, *J* = 236.9 and 3.2 Hz), 151.5, 159.3 (dd, *J* = 239.1 and 2.0 Hz), 159.5 (dd, *J* = 24.9 and 1.6 Hz).



33

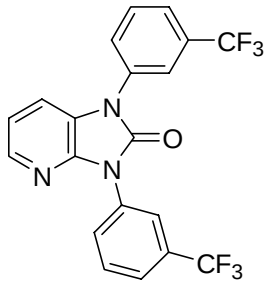
1,3-Bis(2,5-difluorophenyl)-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (33). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 970 mg (2.91 mmol) of **30** with 340 mg (1.14 mmol) of triphosgene gave 850 mg (81%) of **33** as an off-white solid: mp 130-132 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.13 (dd, 1H, *J* = 8.0 and 5.2 Hz), 7.21 (m, 3H), 7.34 (m, 4H), 8.15 (dd, 1H, *J* = 5.2 and 1.2 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 115.9 (d, *J* = 3.2 Hz), 116.2 (d, *J* = 25.7 Hz), 116.9 (d, *J* = 25.7 Hz), 117.4 (dd, *J* = 24.1 and 8.8 Hz), 117.5 (dd, *J* = 24.1, 8.8 Hz), 117.9 (dd, *J* = 22.5 and 9.6 Hz), 118.1 (dd, *J* = 22.5 and 8.8 Hz), 118.6, 120.9 (dd, *J* = 15.8 and 12.0 Hz), 121.7 (dd, *J* = 14.5 and 11.4 Hz), 123.7, 142.3, 142.9, 150.4, 153.8

(dd, *J* = 249.8 and 3.2 Hz), 154.4 (dd, *J* = 249.8 and 3.2 Hz), 158.4 (dd, *J* = 249.8 and 3.2 Hz), 158.6 (dd, *J* = 249.0 and 3.2 Hz); ^{19}F NMR ($CDCl_3$, 376 MHz) δ -123.9 (d, *J* = 15.5 Hz), -123.8 (d, *J* = 17.2 Hz), -117.1 (d, *J* = 17.2 Hz), -116.2 (d, *J* = 15.5 Hz); Anal. Caldc. For $C_{18}H_9F_4N_3O \cdot 1/4\ H_2O$: C, 59.43; H, 2.23; N, 11.50. Found: C, 59.66; H, 2.25; N, 11.55.



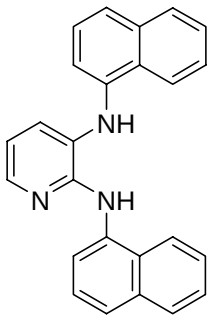
31

Preparation of *N,N'*-Bis[3-(trifluoromethyl)phenyl]pyridine-2,3-diamine (31). According to the general procedure for the preparation of symmetrically substituted diamines, reaction of 4.07 g (17.0 mmol) of **4** with 6.85 g (42.5 mmol) of 3-(trifluoromethyl)aniline afforded 5.98 g (89%) of **31** as an oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.51 (br s, 1H), 6.86 (m, 2H), 7.03 (d, 2H, *J* = 10.6 Hz), 7.17 (d, 1H, *J* = 7.7 Hz), 7.23 (d, 1H, *J* = 7.7 Hz), 7.35 (q, 2H, *J* = 8.1 Hz), 7.43 (d, 1H, *J* = 7.7 Hz), 7.69 (d, 1H, *J* = 8.1 Hz), 7.89 (s, 1H), 8.17 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 112.3 (d, *J* = 4.0 Hz), 115.6 (d, *J* = 4.0 Hz), 116.3, 117.1 (d, *J* = 4.0 Hz), 118.4 (d, *J* = 4.0 Hz), 122.1, 124.1 (q, *J* = 271.0 Hz), 124.2, 124.3 (q, *J* = 271.0 Hz), 129.4, 130.2, 131.4 (q, *J* = 31.0 Hz), 132.0 (q, *J* = 31.0 Hz), 133.0, 140.9, 144.8, 145.4, 151.6; ^{19}F NMR ($CDCl_3$, 376 MHz) δ -63.2, -63.3.



34

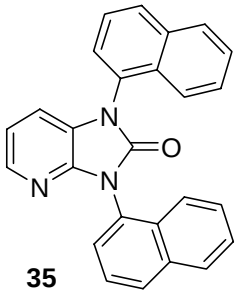
Preparation of 1,3-Bis[3-trifluoromethyl)phenyl]-1,3-dihydro-2*H*-imadazo[4,5-*b*]pyridin-2-one (34). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatement of 1.50 g (3.79 mmol) of **31** with 450 mg (1.52 mmol) of triphosgene gave 1.53 g (95%) of **34** as a colorless solid: mp 181-182 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.16 (dd, 1H, *J* = 7.8 and 5.2 Hz), 7.42 (dd, 1H, *J* = 7.8 and 1.4 Hz), 7.73 (m, 4H), 7.84 (m, 1H), 7.91 (s, 1H), 8.07 (m, 1H), 8.20 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz,) δ 115.3, 118.7, 122.5 (d, *J* = 4.0 Hz), 122.8 (d, *J* = 4.0 Hz), 123.3, 123.5 (q, *J* = 271.0 Hz), 123.7 (q, *J* = 271.0 Hz), 124.4 (d, *J* = 4.0 Hz), 125.0 (d, *J* = 4.0 Hz), 128.9, 129.0, 130.5, 131.7 (q, *J* = 33.0 Hz), 132.5 (q, *J* = 33.0 Hz), 133.8, 134.3, 142.2, 142.9, 151.2; ^{19}F NMR ($CDCl_3$, 376 MHz) δ -63.14, -63.26; Anal. Calcd. For $C_{20}H_{11}F_6N_3O$: C, 56.75; H, 2.62; N, 9.93. Found: C, 56.47; H, 2.41; N, 9.82.



32

Preparation of *N,N*-Di-1-naphthylpyridine-2,3-diamine (32). According to the general procedure for the preparation of symmetrically substituted diamines, reaction of

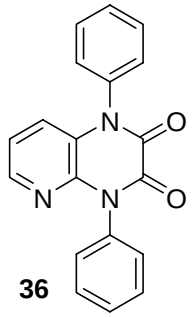
2.00 g (8.35 mmol) of **4** with 2.99 g (20.9 mmol) of 1-aminonaphthalene gave 2.41 g (80%) of **32** which was used in the next step without further purification: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 5.98 (br s, 1H), 6.81 (dd, 1H, *J* = 7.6 and 4.8 Hz), 6.90 (d, 1H, *J* = 7.2 Hz), 7.22 – 7.60 (m, 10 H), 7.64 (d, 1H, *J* = 8.0 Hz), 7.83 (d, 1H, *J* = 8.0 Hz), 7.93 (d, 1H, *J* = 8.0 Hz), 7.97 (d, 1H, *J* = 7.6 Hz), 8.03 (dd, 1H, *J* = 7.6 and 1.2 Hz), 8.12 (dd, 1H, *J* = 4.8 and 1.6 Hz) ^{13}C NMR ($CDCl_3$, 100 MHz) δ 112.8, 116.0, 116.8, 121.1, 122.2, 123.2, 125.6, 125.7, 125.8, 126.0, 126.2, 126.3, 126.95, 127.04, 128.3, 128.6, 128.8, 129.1, 130.5, 134.4, 134.7, 135.9, 139.4, 143.1, 150.7.



35

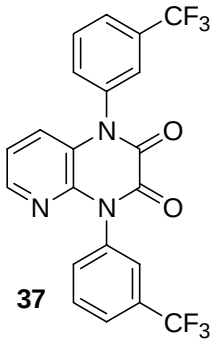
1,3-Di-(1-naphthyl-1,3-dihydro-2*H*-imidazo[4,5-*b*]pyridin-2-one (35). According to the general procedure for the preparation of imidazo[4,5-*b*]pyridin-2-ones, treatment of 1.92 g (5.31 mmol) of **32** with 630 mg (2.1 mmol) of triphosgene gave 1.73 g (84%) of **35** as a light purple solid. mp: 231-232 °C; ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.00 (m, 2H), 7.52-7.82 (m, 10H), 8.03 (m, 5H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 115.0 and 115.1 (due to rotomers), 118.2 and 118.1 (due to rotomers), 122.5, 122.91 and 122.93 (due to rotomers), 123.2, 125.4 and 125.5 (due to rotomers), 125.74 and 125.76 (due to rotomers), 125.97 and 126.01 (due to rotomers), 126.55 and 126.59 (due to rotomers), 126.81 and 126.83 (due to rotomers), 127.02 and 127.07 (due to rotomers), 127.2 and 127.25 (due to rotomers), 127.33, 127.55 and 127.60 (due to rotomers), 127.88, 128.3 and 128.4 (due to rotomers), 128.5 and 128.6 (due to rotomers), 129.44 and 129.46 (due

to rotomers), 129.65 and 129.70 (due to rotomers), 129.74 and 129.77 (due to rotomers), 130.27 and 130.28 (due to rotomers), 133.99 and 134.00 (due to rotomers), 134.2, 140.8 and 140.9 (due to rotomers), 144.3 and 144.4 (due to rotomers), 152.1 and 152.2 (due to rotomers); Anal. Calcd for $C_{26}H_{17}N_3O$: C, 80.60; H, 4.42; N, 10.85. Found: C, 80.27; H, 4.28; N, 10.77.



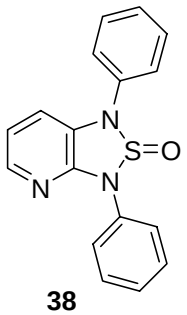
36

Preparation of 1,4-Diphenyl-1,4-dihydropyrido[2,3-*b*]pyrazine-2,3-diamine (36). To a stirred solution of 350 mg (1.34 mmol) of **28** in 8 mL of THF was added 312 mg (3.09 mmol) of triethylamine followed by 189 mg (1.49 mmol) of oxalyl chloride. After stirring for 15 min at rt, the reaction was diluted with EtOAc and then quenched with 8 mL of sat. aqueous $NaHCO_3$. The organic layer was dried over $MgSO_4$ and the solvent removed under reduced pressure. The residual solid was recrystallized from EtOAc/hexane to give 400 mg (95%) of **36** as a tan solid: mp 300 °C (decomp); ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.76 (dd, 1H, *J* = 8.1 and 1.3 Hz), 7.05 (dd, 1H, *J* = 8.1 and 4.6 Hz), 7.35 (d, 2H, *J* = 7.4 Hz), 7.45 (m, 3H), 7.55 (m, 3H), 7.66 (m, 2H), 7.95 (dd, 1H, *J* = 4.6 and 1.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 119.6, 123.4, 125.9, 128.8, 128.9, 129.3, 129.7, 129.9, 130.9, 136.3, 136.9, 140.8, 142.0, 154.5, 155.7; Anal. Calcd. For $C_{19}H_{13}N_3O_2$: C, 72.37; H, 4.16; N, 13.33. Found: C, 72.61; H, 4.08; N, 13.13.



37

Preparation of 1,4-Bis[3-(Trifluoromethyl)phenyl]-1,4-dihydropyrido[2,3-*b*]pyrazine-2,3-dione (37). To a stirred solution of 1.15 mg (2.91 mmol) of **31** in 25 mL of THF was added 675 mg (6.67 mmol) of triethylamine followed by 407 mg (3.21 mmol) of oxalyl chloride. After stirring for 15 min at rt, the reaction was diluted with EtOAc and then quenched with 25 mL of sat. aqueous $NaHCO_3$. The organic layer was dried over $MgSO_4$ and the solvent removed under reduced pressure. The residual solid was recrystallized from EtOAc/hexane to give 1.27 g (97%) of **37** as a yellow solid: mp 319-320 °C; ^{1}H NMR (DMSO-d_6, 400 MHz) δ 6.87 (dd, 1H, J = 8.2 and 1.3 Hz), 7.12 (dd, 1H, J = 8.2 and 4.7 Hz), 7.71 (m, 1H), 7.76 (m, 2H), 7.86 (m, 3H), 7.92 (m, 1H), 8.01 (m, 2H); ^{13}C NMR (DMSO-d_6, 100 MHz) δ 119.9, 123.6, 124.1 (q, J = 272.0 Hz), 124.3 (q, J = 272.0 Hz), 125.8, 125.9, 126.1, 126.9, 130.6 (q, J = 30.0 Hz), 131.2, 131.8 (q, J = 30.0 Hz), 133.5, 133.8, 136.9, 137.5, 140.6, 142.2, 154.5, 155.4; ^{19}F NMR (DMSO-d_6, 376 MHz) -61.6, -61.8; Anal. Calcd. For $C_{21}H_{11}F_6N_3O_2$: C, 55.89; H, 2.46; N, 9.31. Found: C, 55.64; N, 2.24; N, 9.26.



38

Preparation of 1,3-Diphenyl-1,3-dihydro[1,2,5]thiadiazolo[3,4-*b*]pyridine-2-oxide (38). To a stirred solution of 371 mg (1.42 mmol) of **28** in 8 mL of THF was added 334 mg (3.30 mmol) of triethylamine followed by 179 mg (1.51 mmol) of thionyl chloride. After stirring at rt for 15 min the reaction was diluted with EtOAc and then quenched with 10 mL of sat. aqueous $NaHCO_3$. The organic layer was dried over $MgSO_4$ and the solvent removed under reduced pressure. The residual was purified by silica gel chromatography to give 388 mg (89%) of **38** as an oil: ^{1}H NMR ($CDCl_3$, 400 MHz) δ 6.91 (dd, 1H, *J* = 7.8 and 5.1 Hz), 7.13 (dd, 1H, *J* = 7.8 and 1.3 Hz), 7.45 (m, 2H), 7.56 (m, 6H), 7.69 (m, 2H), 7.98 (dd, 1H, *J* = 5.1 and 1.3 Hz); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 116.6, 116.7, 126.8, 126.9, 127.6, 128.4, 128.6, 129.9, 130.3, 134.5, 135.7, 140.5, 146.8; Anal. Calcd. For $C_{17}H_{13}N_3OS$: C, 66.43; H, 4.26; N, 13.67. Found: C, 66.40; H, 4.12; N, 13.49.

References

1. Sakamoto, T.; Kondo, Y.; Yamanaka, H. *Chem. Pharm. Bull.* **1985**, *33*, 4764.

2. Robinson, M. M.; Finch, N. U. S. Patent 3719683 (1973).

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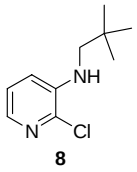

8


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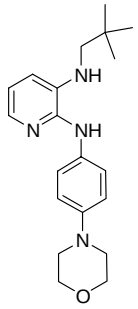
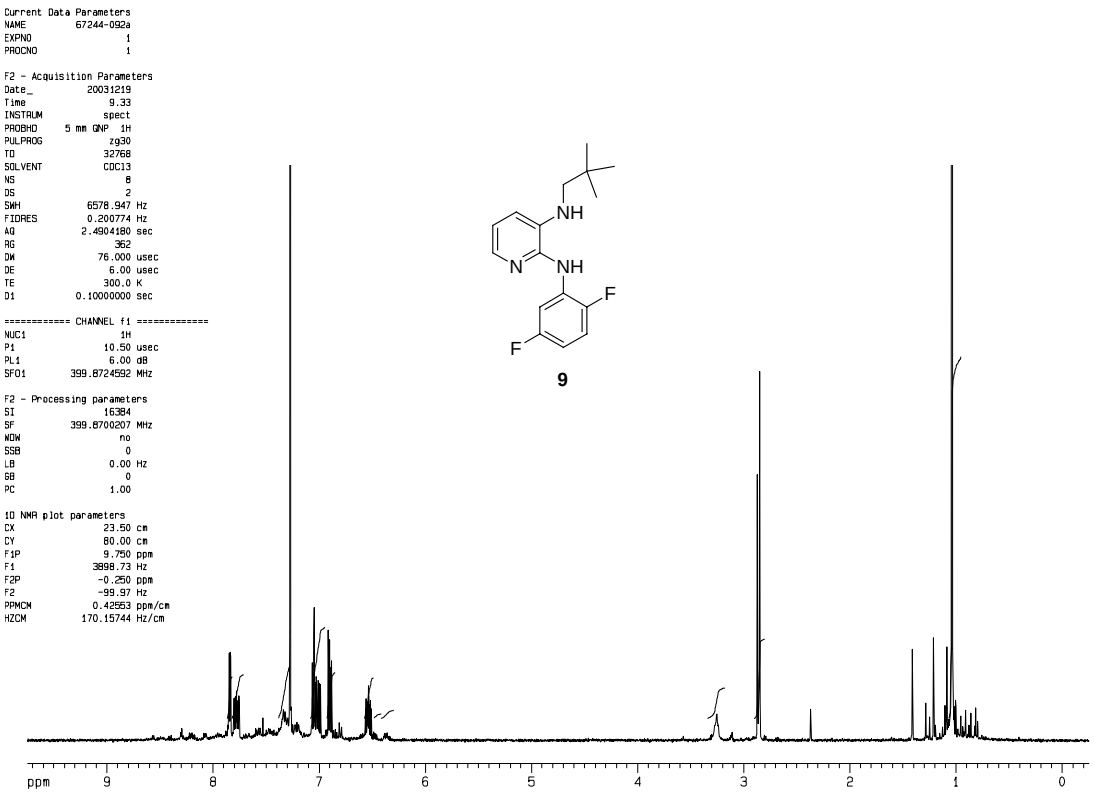


11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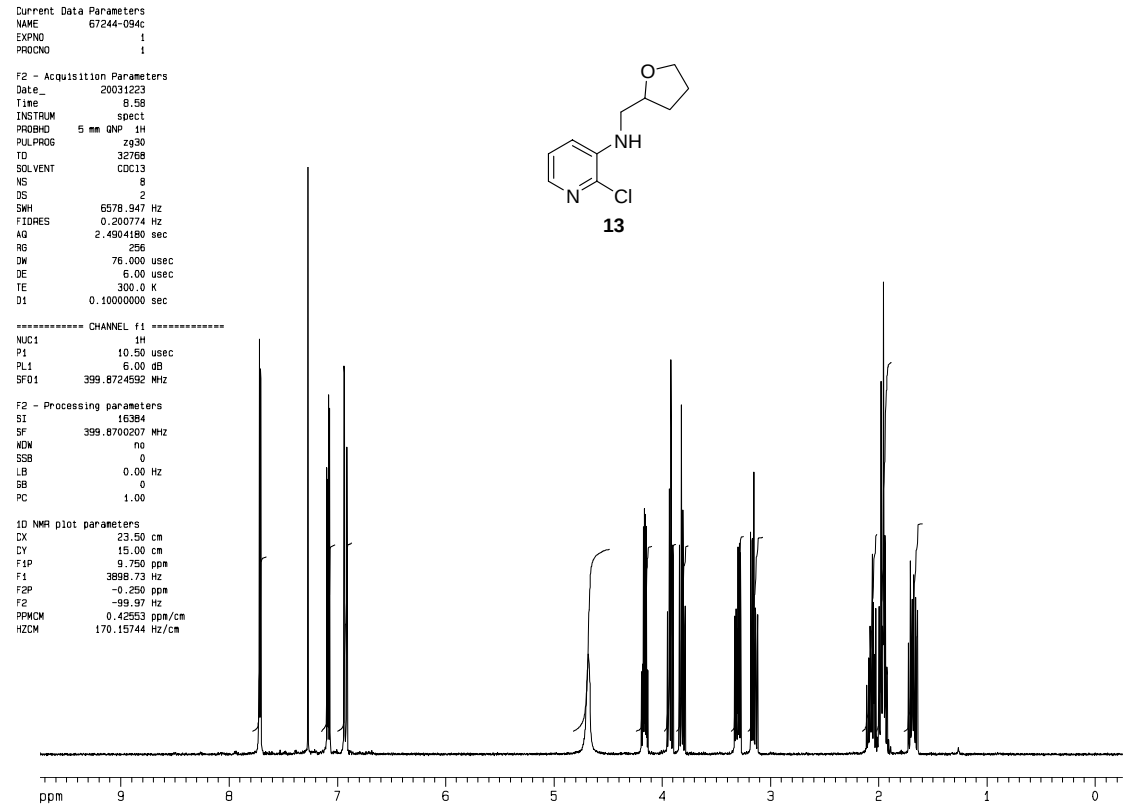
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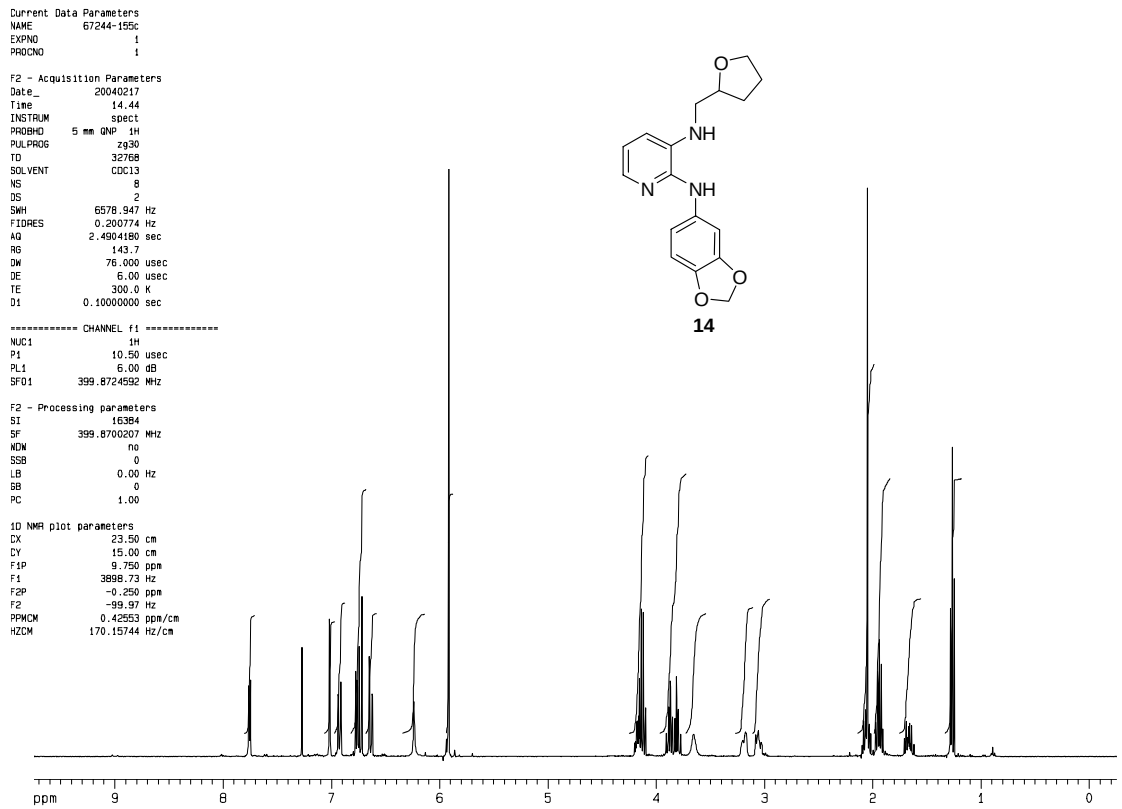
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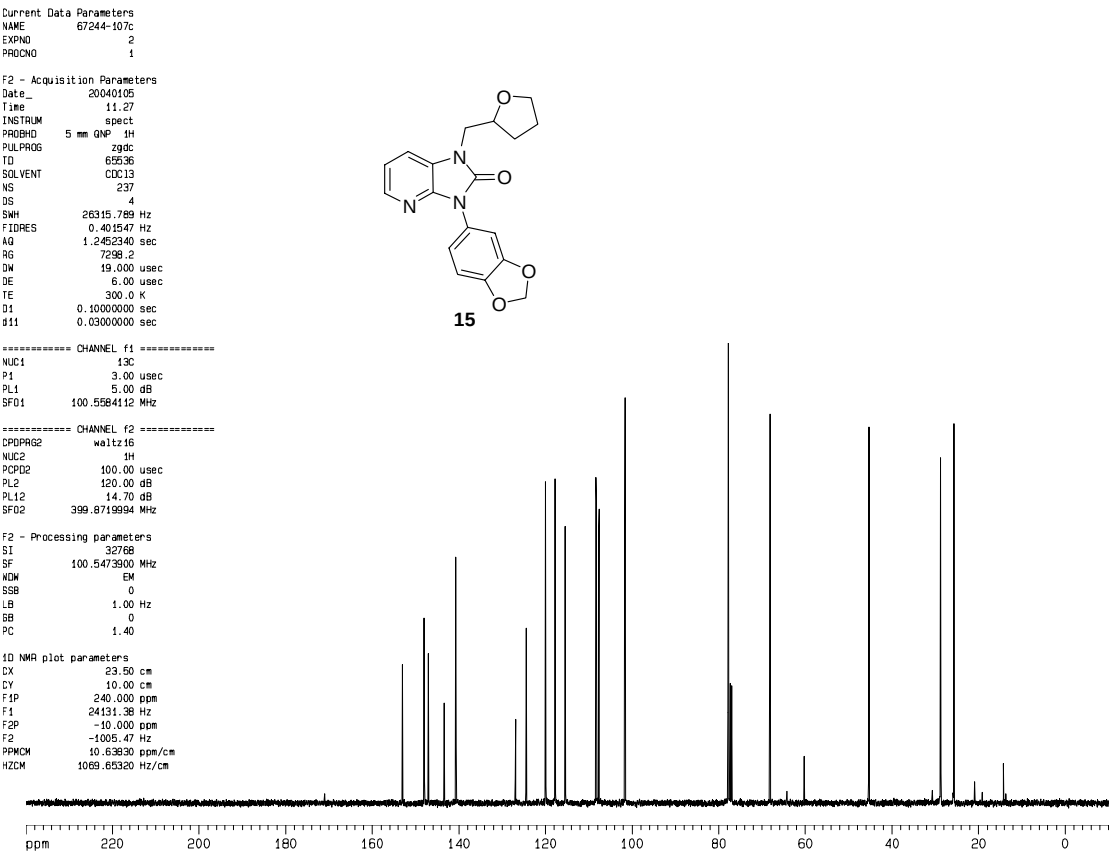
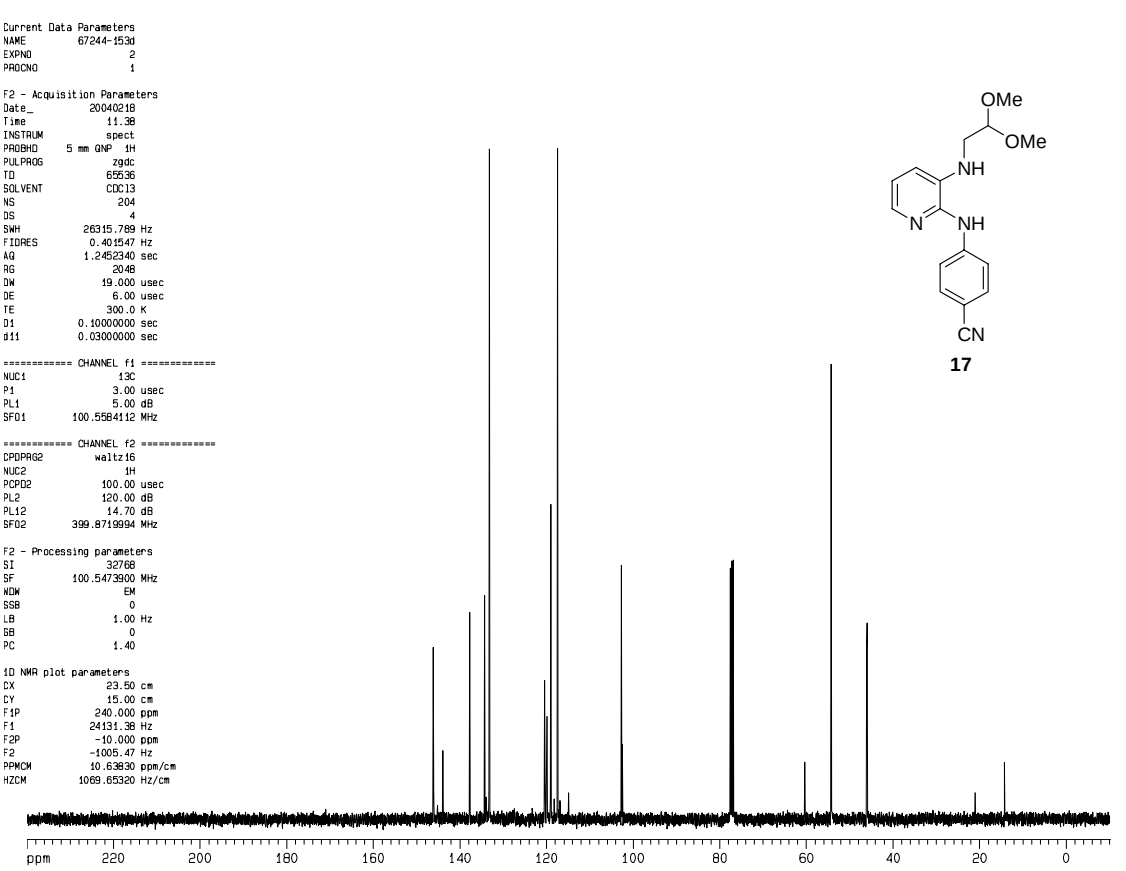
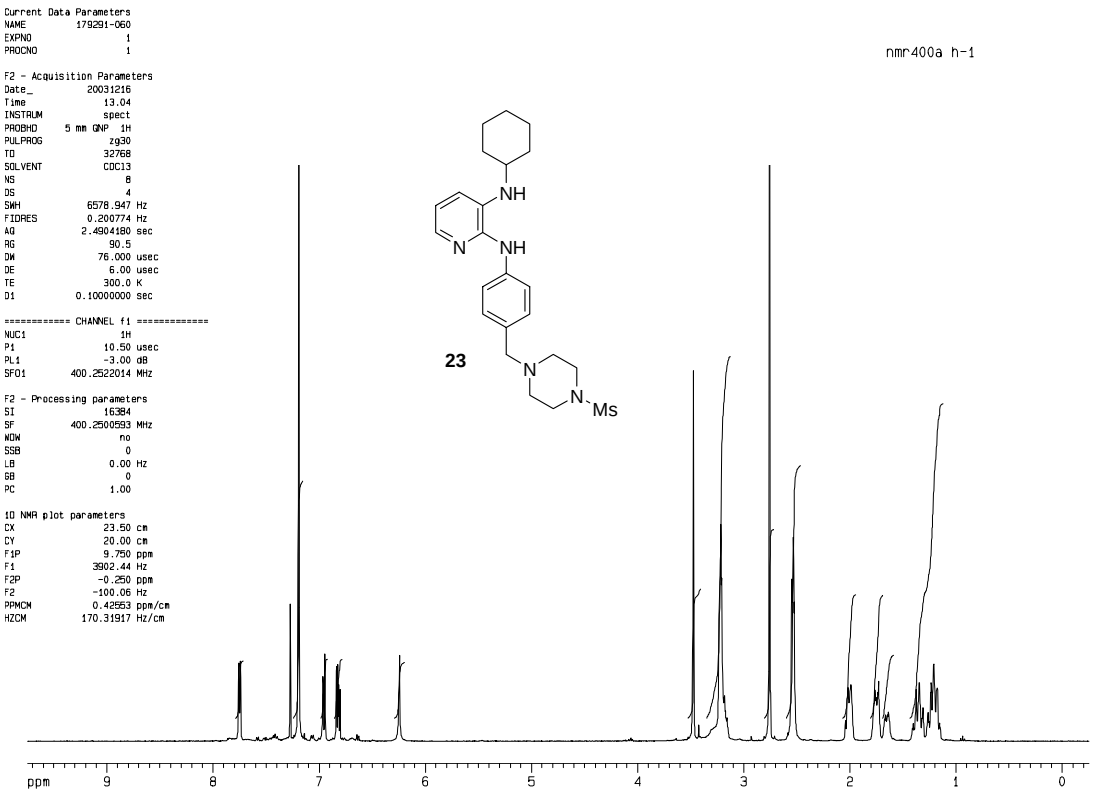
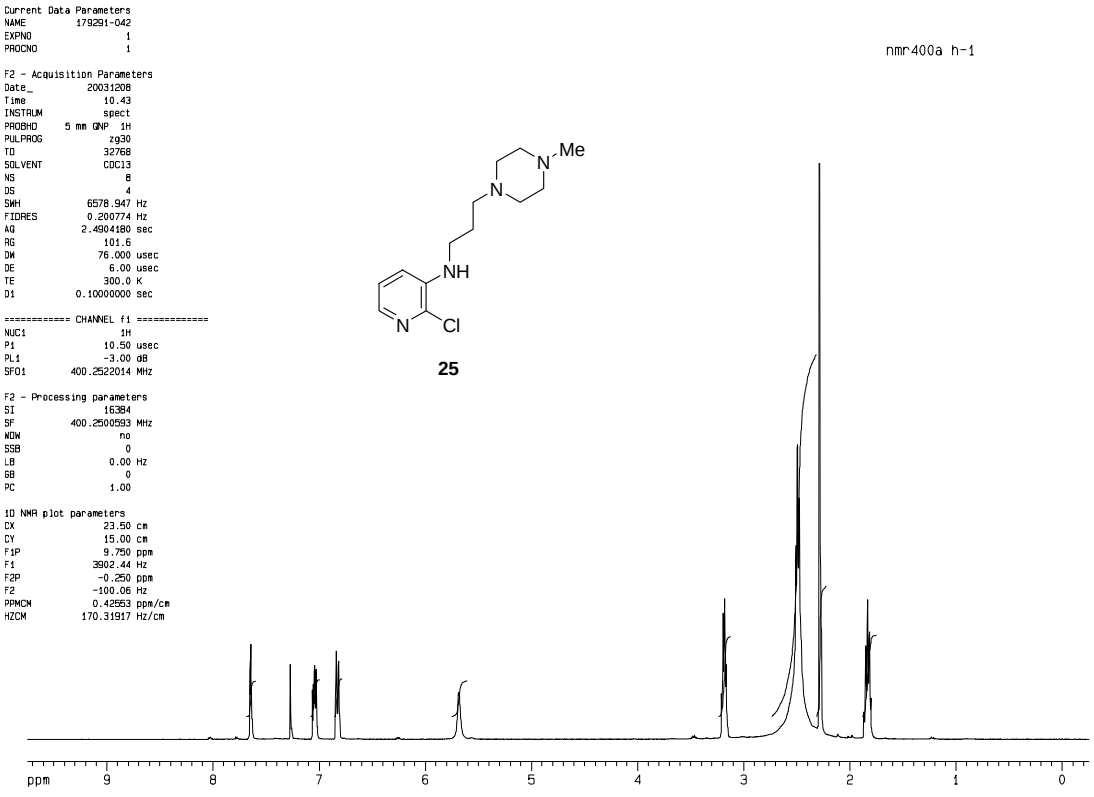
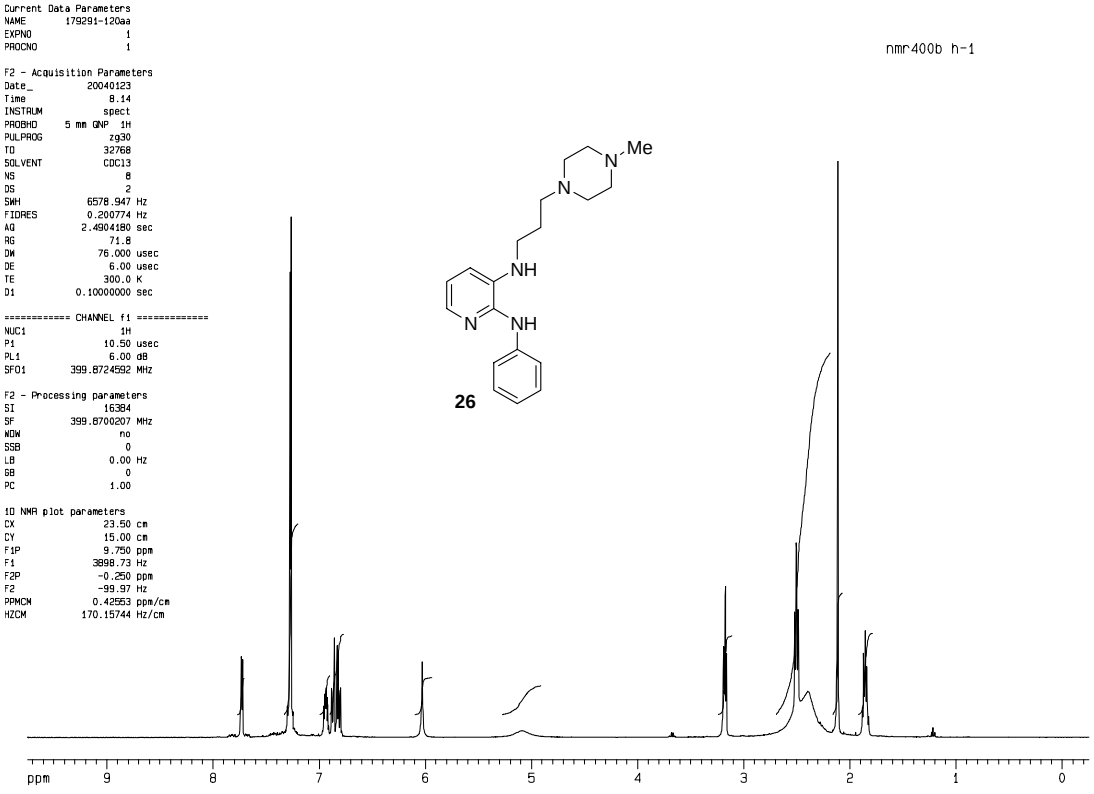
F2 -99.97 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.15744 Hz/cm
O
NH
N
NH
O
O
14
ppm
9
8
7
6
5
4
3
2
1
0

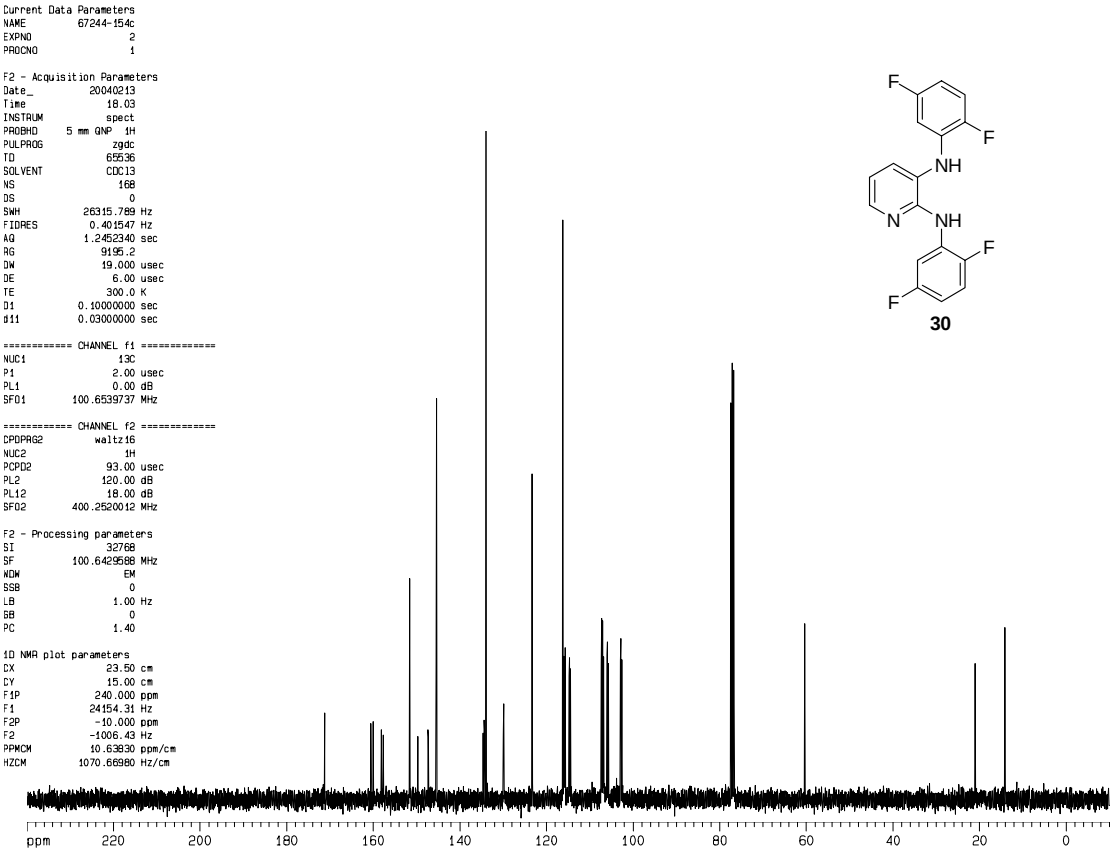

Current Data Parameters
NAME 67244-107c
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040105
Time 11.27
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 237
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 7298.2
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz
F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PPMCM 10.63830 ppm/cm
HZCM 1069.65320 Hz/cm
15
ppm 220 200 180 160 140 120 100 80 60 40 20 0

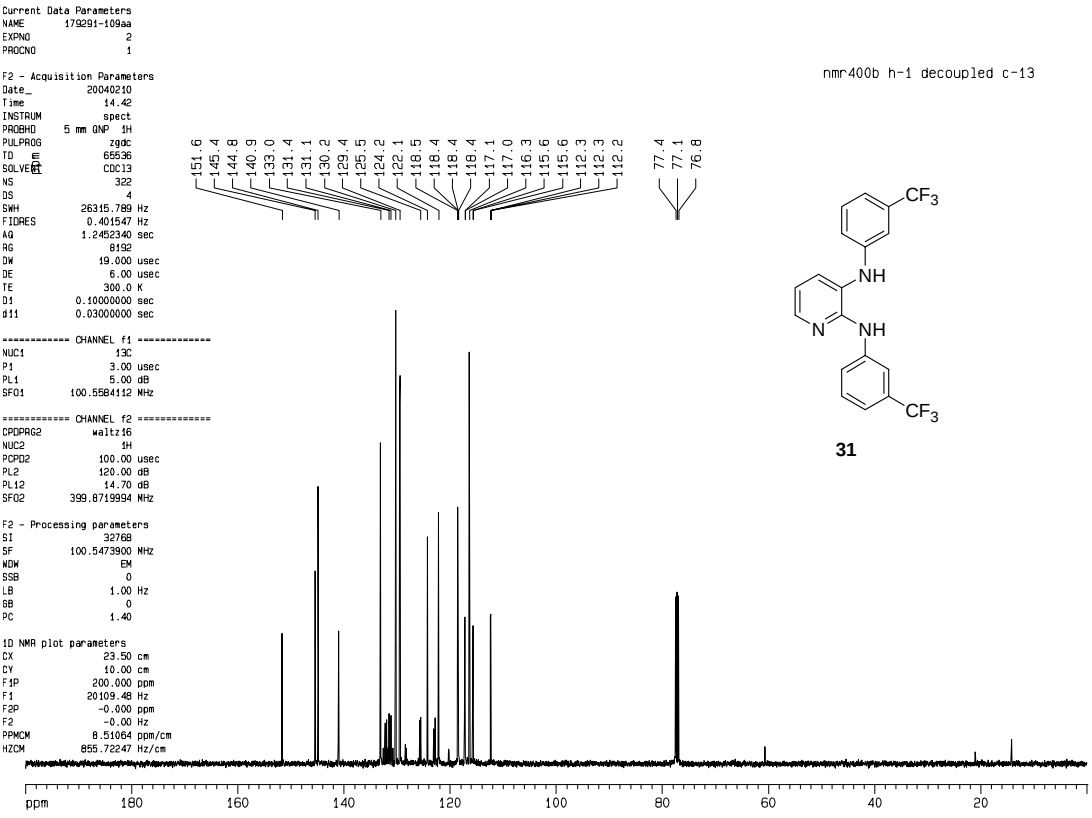

Current Data Parameters
NAME 67244-153d
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040218
Time 11.38
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 204
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 2048
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz
F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 240.000 ppm
F1 24131.38 Hz
F2P -10.000 ppm
F2 -1005.47 Hz
PPMCM 10.63830 ppm/cm
HZCM 1069.65320 Hz/cm
OMe
OMe
NH
NH
N
CN
17
ppm 220 200 180 160 140 120 100 80 60 40 20 0

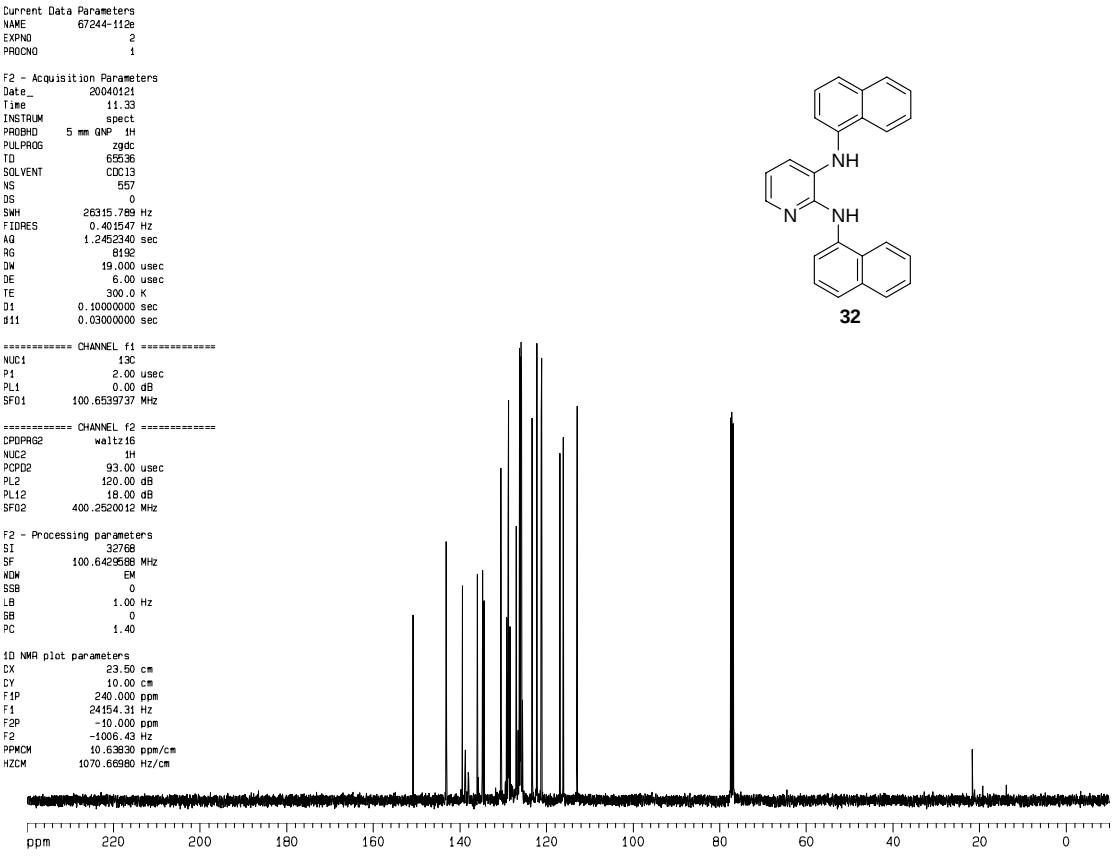

Current Data Parameters
NAME 179291-060
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20031216
Time 13.04
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 90.5
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
========== CHANNEL f1 ==========
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.50 cm
CY 20.00 cm
F1P 9.750 ppm
F1 3902.44 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.31917 Hz/cm
nmr400a h-1
NH
N
NH
23
N
N
Ms
ppm
9
8
7
6
5
4
3
2
1
0


Current Data Parameters
NAME 179291-042
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20031208
Time 10.43
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 4
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 101.6
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 10.50 usec
PL1 -3.00 dB
SFO1 400.2522014 MHz
F2 - Processing parameters
SI 16384
SF 400.2500593 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 9.750 ppm
F1 3902.44 Hz
F2P -0.250 ppm
F2 -100.06 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.31917 Hz/cm
nmr400a h-1
Me
N
N
NH
N
Cl
25
ppm
9
8
7
6
5
4
3
2
1
0


Current Data Parameters
NAME 179291-120aa
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040123
Time 8.14
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 2
SWH 6578.947 Hz
FIDRES 0.200774 Hz
AQ 2.4904180 sec
RG 71.8
DW 76.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
CHANNEL f1
NUC1 1H
P1 10.50 usec
PL1 6.00 dB
SFO1 399.8724592 MHz
F2 - Processing parameters
SI 16384
SF 399.8700207 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 9.750 ppm
F1 3898.73 Hz
F2P -0.250 ppm
F2 -99.97 Hz
PPMCM 0.42553 ppm/cm
HZCM 170.15744 Hz/cm
nmr400b h-1
Me
N
N
NH
N
NH
26
ppm
9
8
7
6
5
4
3
2
1
0


Current Data Parameters
NAME 67244-154c
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040213
Time 18.03
INSTRUM spect
PROBHD 5 mm GNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 168
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 9195.2
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6539737 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz
F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 23.50 cm
CY 15.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.43 Hz
PPMCM 10.63830 ppm/cm
HZCM 1070.66980 Hz/cm
F
F
NH
N
NH
F
F
30
ppm
220
200
180
160
140
120
100
80
60
40
20
0


Current Data Parameters
NAME 179291-109aa
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040210
Time 14.42
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 322
DS 4
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
CHANNEL f1
NUC1 13C
P1 3.00 usec
PL1 5.00 dB
SFO1 100.5584112 MHz
CHANNEL f2
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 14.70 dB
SFO2 399.8719994 MHz
F2 - Processing parameters
SI 32768
SF 100.5473900 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 200.000 ppm
F1 20109.48 Hz
F2P -0.000 ppm
F2 -0.00 Hz
PPMCM 8.51064 ppm/cm
HZCM 855.72247 Hz/cm
nmr400b h-1 decoupled c-13
151.6
145.4
144.8
140.9
133.0
131.4
131.1
130.2
129.4
125.5
124.2
122.1
118.5
118.4
118.4
118.4
117.1
117.0
116.3
115.6
115.6
112.3
112.3
112.2
77.4
77.1
76.8
CF3
NH
N
NH
CF3
31
ppm
180
160
140
120
100
80
60
40
20


Current Data Parameters
NAME 67244-112e
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040121
Time 11.33
INSTRUM spect
PROBHD 5 mm QNP 1H
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 557
DS 0
SWH 26315.789 Hz
FIDRES 0.401547 Hz
AQ 1.2452340 sec
RG 8192
DW 19.000 usec
DE 6.00 usec
TE 300.0 K
D1 0.10000000 sec
d11 0.03000000 sec
========== CHANNEL f1 ==========
NUC1 13C
P1 2.00 usec
PL1 0.00 dB
SFO1 100.6539737 MHz
========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 93.00 usec
PL2 120.00 dB
PL12 18.00 dB
SFO2 400.2520012 MHz
F2 - Processing parameters
SI 32768
SF 100.6429588 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 23.50 cm
CY 10.00 cm
F1P 240.000 ppm
F1 24154.31 Hz
F2P -10.000 ppm
F2 -1006.43 Hz
PPMCM 10.63830 ppm/cm
HZCM 1070.66980 Hz/cm
NH
N
NH
32
ppm
220
200
180
160
140
120
100
80
60
40
20
0