Experimental

General Methods. All ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX-400 spectrometer. Chemical shifts are reported in parts per million (ppm). Coupling constants (*J*) are given in Hertz (Hz). Spin multiplicities are indicated by standard notation.

Infrared spectra were recorded on a Nicolet 560 FT-IR spectrophotometer. Band positions are given in reciprocal centimeters (cm^{-1}) and relative intensities are listed as br (broad), s (strong), m (medium), or w (weak).

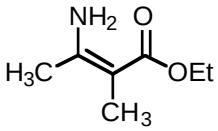
Melting points are corrected and were taken in Kimax soft capillary tubes using a Thomas-Hoover Uni-Melt capillary melting point apparatus equipped with a calibrated thermometer.

Low resolution (LRMS) and high resolution (HRMS) were obtained on a JEOL SX-102A instrument.

Thin layer chromatography (TLC) was performed on 0.25 mm Analtech silica-coated glass plates, with compounds being identified in one or more of the following manners: UV (254 nm) and vanillin/sulfuric acid/ethanol charring. Flash chromatography was performed using glass columns and "medium pressure" silica gel (Sorbent Technologies, 45-70 m).

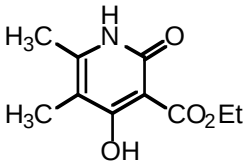
Tetrahydrofuran (THF) was distilled from sodium/benzophenone. Dimethylformamide (DMF) and N,N,N',N'-tetramethylethlenediamine (TMEDA) were distilled from CaH_2 dried over 4Å molecular sieves. Benzene and ethanol were dried over 4Å molecular sieves. Triphenylphosphine was recrystallized from hexanes. Tetraethylorthosilicate was distilled and stored over 4Å molecular sieves. All other reagents were purchased and used as received. Glassware used in the reactions was dried overnight in an oven at 120 °C. All reactions were performed under an atmosphere of argon unless otherwise noted.

Amino ester 8



To a mixture of 25 g (0.17 mol) of ethyl methylacetoacetate absorbed on 42 g of Bentonite K-10 clay was added 16 mL (0.23 mol) of 14.8 M ammonium hydroxide. This mixture was stirred at room temperature under atmospheric conditions for 24 h and washed 4x with 500 mL of CH_2Cl_2. The combined washings were dried over $MgSO_4$ and concentrated *in vacuo* to yield 22.6 g (93%) of amino ester **8** as a white, crystalline solid, m.p. 46-49 °C (lit m.p. 52 °C), which was used without further purification. The ^{1}H NMR spectrum matched that of the reported compound. IR (CCl_4) 3506 (m), 3309 (m), 2975 (m), 2905 (m), 2866 (w), 1666 (s), 1616 (s) cm^{-1}. ^{1}H NMR ($CDCl_3$) d 1.24 (t, *J* = 7 Hz, 3H), 1.72 (s, 3H), 1.91 (s, 3H), 4.10 (q, *J* = 7 Hz, 2 H). ^{13}C ($CDCl_3$) d NMR d 12.8, 15.0, 21.6, 59.3, 89.4, 156.6, 171.2. EI-MS *m/z* 143 (100), 114 (26), 98 (93), 69 (62), 42 (23). HRMS for $C_7H_{13}NO_2$ calcd 143.0946, found 143.0951.

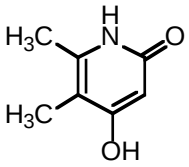
Pyridone 9



To a solution of 3.80 g (0.166 mol) of sodium in 60 mL of ethanol was added a solution of 25.0 mL (0.166 mol) of diethyl malonate in 9 mL of toluene. The resulting yellow solution was stirred for 1 h and a solution of 11.85 g (0.083 mol) of amino ester **8** in 24 mL of toluene was added. The solution was refluxed for 4 d during which time a white precipitate formed. The

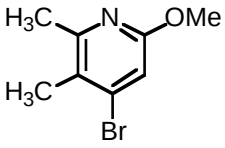
solution was cooled, diluted with 60 mL of water and stirred for 30 min. The phases were separated and the aqueous layer washed with 60 mL of toluene. The ethanol was removed *in vacuo* and the remainder of the aqueous layer acidified with conc. HCl. The precipitate thus obtained filtered, washed with water and dried to yield 11.43 g (65 %) of pyridone **9** as a white, crystalline solid, m.p. 215-220 °C (dec) (lit m.p. 216-221). The ^{1}H NMR spectrum matched that of the reported compound. IR (CCl_4) 3317 (w), 3165 (w), 2986 (m), 2932 (m), 2866 (m), 2803 (m), 1654 (s) cm^{-1}. ^{1}H NMR ($CDCl_3$) d 1.42 (t, *J* = 8 Hz, 3H), 1.65 (s, 3H), 1.95 (s, 3H), 4.41 (q, *J* = 8 Hz, 2H), 12.33 (s, 1 H). ^{13}C NMR ($CDCl_3$) d 9.9, 14.5, 17.9, 62.1, 96.8, 106.5, 149.2, 163.0, 172.9, 175.2. EI-MS *m/z* 211 (93), 165 (100), 139 (48), 137 (47), 109 (64). HRMS for $C_{10}H_{13}NO_4$ calcd 211.0845, found 211.0844.

Pyridone 10



A solution of 13.88 g (0.066 mol) of pyridone **9** and 26.4 g (0.66 mol) of NaOH in 330 mL of water was heated at reflux for 2 h. The solution was cooled to 0 °C, acidified to pH = 7 with conc. HCl and the precipitate filtered and washed with water to yield 9.10 g (99 %) of pyridone **9** as a white solid, m.p. > 350 °C. IR (KBr) 3421 (w), 3266 (w), 3087 (m), 3002 (m), 2928 (m), 2882 (m), 2722 (w), 2680 (w), 2579 (w), 1662 (s), 1616 (s) cm^{-1}. ^{1}H NMR (DMSO-d_6) d 1.79 (s, 3H), 2.09 (s, 3H), 5.49 (s, 1H), 10.53 (br s, 1H), 10.91 (br s, 1H). ^{13}C NMR (DMSO-d_6) d 9.8, 16.7, 96.1, 104.2, 142.2, 163.7, 167.3. EI-MS *m/z* 139 (100), 111, (76), 110 (82), 98 (87), 69 (71), 44 (84). HRMS for $C_7H_9NO_2$ calcd 139.0633, found 139.0638.

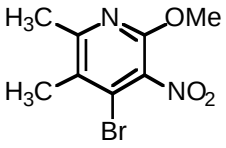
Pyridine 12



2.95 g (21 mmol) of hydroxypyridone **10** and 4.4 g (16 mmol) of $POBr_3$ were weighed out in a dry box. 9 mL DMF was added and the resulting mixture heated at 110 °C for 30 min. After cooling, 10 mL water was added and the resulting solution brought to pH = 7 with Na_2CO_3. The mixture was cooled to 0 °C and the precipitate thus obtained filtered. The precipitate was washed with water and Et_2O to yield 3.21 g of an orange yellow solid. Although the product could be purified, it was more convenient to use the crude material in the next step.

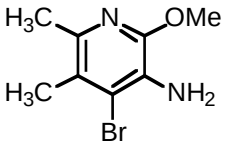
To a solution of the crude bromopyridone and 2.96 g (11 mmol) Ag_2CO_3 in 30 mL benzene was added 1.25 mL (20 mmol) MeI and the resulting mixture heated at 45 °C for 2 d in the dark. After cooling, the mixture was filtered and the precipitate washed with benzene and sat $NaHCO_3$. The layers were separated and the product extracted 3x with CH_2Cl_2. The combined organic extracts were dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (19:1 hexanes:EtOAc, R_f = 0.38) afforded 1.64 g (36% from **10**) of pyridine **12** as a white, crystalline solid, m.p. = 38-41 °C. IR (CCl_4) 3014 (w), 2971 (w), 2944 (w), 2897 (w), (1577 (s), 1577 (s) cm^{-1}. ^1H NMR ($CDCl_3$) d 2.27 (s, 3H), 2.44 (s, 3H), 3.85 (s, 3H), 6.80 (s, 1H). ^{13}C NMR ($CDCl_3$) d 18.0, 24.1, 54.0, 111.6, 124.0, 137.3, 155.4, 161.9. EI-MS *m/z* 216 (98), 214 (100), 187 (37), 185 (32).

Nitropyridine 13



A solution of 1.32 g (6.1 mmol) of pyridine **12**, 0.60 mL (8.6 mmol) of HNO_3 and 15 mL of H_2SO_4 was stirred at room temperature for 14 h. The solution was diluted with 100 mL of water and neutralized with Na_2CO_3. The product was extracted 2x with 100 mL Et_2O and the combined organic extracts dried over $MgSO_4$ and concentrated *in vacuo* to yield 1.55 g (97%) of nitropyridine **13** as a yellow solid, mp 86-89 °C. IR (CCl_4) 3025 (w), 2998 (w), 2951 (w), 2924 (w), 2905 (w), 1581 (s) cm. ^{1}H NMR ($CDCl_3$) d 2.33 (s, 3H), 2.51 (s, 3H), 3.97 (s, 3H). ^{13}C NMR ($CDCl_3$) d 18.5, 24.4, 55.0, 125.2, 127.5, 152.5, 156.9. FAB-MS *m/z* 263 (66), 261 (61), 155 (57), 152 (59), 119 (68), 103 (48), 85 (100). HRMS for $C_8H_9BrN_2O_3$ calcd 260.9875, found 260.9872.

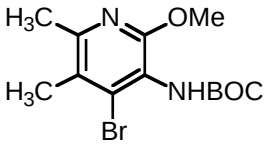
Aminopyridine 13



To a solution of 1.39 g (5.3 mmol) of nitropyridine **13** in 28 mL of EtOH and 7 mL of water was added 3.5 g (63 mmol) of iron and 2 drops of conc. HCl. The resulting solution was heated at reflux for 2 h. After cooling, the solution was filtered and concentrated *in vacuo*. Purification by column chromatography (9:1 hexanes:EtOAc, R_f = 0.40) gave 0.800 g (65 %) of aminopyridine **13** as a yellow crystalline solid, mp 52-54 °C. IR (CCl_4) 3487 (s), 3394 (s), 3014 (w), 2983 (w),

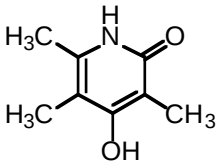
2948 (m), 2920 (w), 2858 (w), 1654 (m), 1612 (s) cm^{-1}. ^{1}H NMR ($CDCl_3$) d 2.29 (s, 3H), 2.41 (s 3H), 3.98 (s, 3H), 4.05 (br s, 2H). ^{13}C NMR ($CDCl_3$) d 18.2, 22.3, 53.3, 119.8, 122.7, 127.1, 140.7, 149.6. EI-MS *m/z* 232 (98), 230 (100), 189 (68), 187 (68). HRMS for $C_8H_{11}BrN_2O$ calcd 232.0034, found 262.0049.

N-BOC protected amine 14



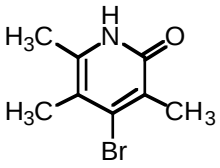
N-BOC-protected amine **14** was obtained directly from nitropyridine **12**. 940 mg (4.3 mmol) of **12** was reduced as described above. The crude product was dissolved in 40 mL THF and 5.6 g (0.026 mol) of BOC_2O and a catalytic amount of DMAP were added. The solution was heated at reflux for 5 h. After cooling, 3.0 g (0.022 mol) of K_2CO_3 and 40 mL MeOH was added and the resulting solution heated at reflux for 24 h. After cooling 100 mL of 0.5 M HCl was added and the product extracted 3x with 100 mL EtOAc. The combined organic extracts were dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (3:1 hexanes:EtOAc R_f = 0.43) afforded 751 mg (52 %) as a white crystalline solid, mp = 101-104 °C. IR (CCl_4) 3429 (m), 3010 (w), 2979 (m), 2951 (w), 2928 (w), 1740 (s) cm^{-1}. ^{1}H NMR ($CDCl_3$) d 1.47 (s, 9H), 2.02 (s, 3H), 2.43 (s, 3H), 3.91 (s, 3H), 5.88 (br s, 1H). ^{13}C NMR ($CDCl_3$) d 16.9, 21.7, 26.5, 52.1, 78.9, 116.0, 122.5, 134.8, 150.5, 151.7, 155.4. EI-MS *m/z* 332 (12), 330 (13), 232 (90), 230 (88), 57 (100). HRMS for $C_{13}H_9BrN_2O_3$ calcd 330.0579, found 330.0579.

Pyridone 15



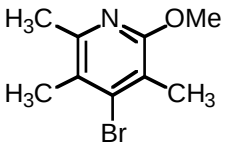
A solution of 27.2 mL (0.158 mol) of diethyl methylmalonate in 8 mL of toluene was added to a solution of 3.65 g (0.158 mol) of sodium in 55 mL of ethanol. The resulting solution was stirred at room temperature for 1 h and a solution of 22.6 g (0.158 mol) of amino ester **8** in 22 mL of toluene was added over a period of 20 min. The reaction was refluxed for 24 h during which time a white precipitate formed. The mixture was cooled, diluted with 105 mL of water, and stirred for 30 min. The phases were separated and the aqueous layer washed with 105 mL toluene. The solution was acidified with conc. HCl to pH 7.0. Filtration and drying of the precipitate yielded 16.8 g (69 %) of pyridone **15** as a white, crystalline solid, m.p. 270-275 °C (dec), (lit m.p. 260 °C). The ^{1}H NMR spectrum matched that of the reported compound. IR (KBr) 3429 (br), 3262 (br), 2955 (m), 2928 (m), 1631 (s) cm^{-1}. ^{1}H NMR (DMSO-d_6) d 1.80 (s, 3H), 1.83 (s, 3H), 2.06 (s, 3H), 9.12 (br s, 1H), 10.85 (br s, 1H). ^{13}NMR d (DMSO-d_6) 6.9, 8.2, 14.0, 101.5, 101.8, 135.8, 160.0, 161.3. EI-MS *m/z* 153 (100), 124 (32), 98 (41), 78 (37), 63 (33). HRMS for $C_8H_{10}NO_2$ calcd 153.0790, found 153.0783.

Bromopyridone 16



A mixture of 5.0 g (.033 mol) of pyridone **12** and 14.10 g (0.049 mol) of $POBr_3$ was weighed out in a dry box and heated at 110° C for 1 h. The solution was allowed to cool to room temperature and ice was added. The resulting precipitate was filtered and washed with water. Recrystallization from ethanol afforded 4.25 g (60 %) of bromopyridone **16** as a white, crystalline solid, m.p. 226-228° C (lit m.p. 227° C). The ^{1}H NMR spectrum matched that of the reported compound. IR (CCl_4) 3289 (w), 3134 (w), 2979 (m), 2928 (m), 2870 (m), 2769 (m), 1647 (s), 1616 (m), 1550 (s) cm^{-1}. ^{1}H NMR (DMSO-d_6) d 2.01 (s, 3H), 2.05 (s, 3H), 2.27 (s, 3H). ^{13}C NMR (DMSO-d_6) d 17.3, 17.5, 17.7, 111.2, 125.6, 139.1, 140.7, 161.0. EI-MS *m/z* 217 (100), 215 (94) 188 (45), 186 (40). HRMS for $C_8H_{10}BrNO_2$ calcd 216.9925, found 216.9917.

Bromopyridine 17

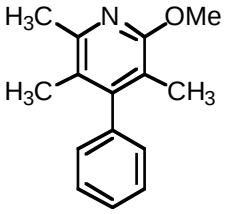


To a solution of 3.45 g (0.011 mol) of Ag_2CO_3 and 3.58 g (0.017 mol) of pyridone **16** in 30 mL of benzene was added 1.25 mL (0.020 mol) of MeI and the resulting solution heated at 45° C for 12 h in the dark. The solution was cooled to 0 °C, filtered, and the filtrate washed with 50 mL of 2 % $NaHCO_3$ and 50 mL water. The solution was concentrated under reduced pressure to 100 mL and extracted 3x with CH_2Cl_2. The combined organic extracts were dried over $MgSO_4$ and

concentrated *in vacuo* to give 3.80 g (100 %) of pyridine **17** as a white, crystalline solid, m.p. 44-45 °C. IR (CCl_4) 3006 (w), 2953 (w), 2951 (m), 2920 (w), 2893 (w), 2866 (w), 1581 (m), 1553 (m) cm^{-1}. ^1H NMR ($CDCl_3$) d 2.19 (s, 3H), 2.22 (s, 3H), 2.37 (s, 3H), 3.83 (s, 3H). ^{13}C NMR ($CDCl_3$) d 14.5, 19.1, 23.8, 53.9, 118.4, 124.0, 139.4, 151.1, 159.9. EI-MS *m/z* 231 (100), 229 (87), 216 (29). HRMS for $C_9BrH_{12}NO_2$ calcd 229.0104, found 229.0102.

General procedure for the synthesis of biaryls. Coupling reactions were performed under identical conditions using 1.0 eq of aryl bromide, 2.0 eq of siloxane, 2.0 eq TBAF, 0.20 eq $Pd(OAc)_2$, 0.40 eq PPh_3. The following example is illustrative.

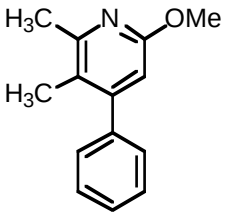
Entry 3



To a solution of 261 mg (1.1 mmol) of bromopyridine **17**, 440 mg (2.2 mmol) of phenyltrimethoxysilane, 52 mg (0.21 mmol) of $Pd(OAc)_2$, and 110 mg (0.42 mmol) of PPh_3 in 10 mL DMF was added 2.2 mL (2.2 mmol) of a 1M solution of TBAF in DMF. The solution was degassed *via* a single freeze-pump-thaw cycle and heated at 80 °C for 12 h. The reaction was quenched with 10 mL water and the product extracted 3x with 20 mL Et_2O. The combined organic extracts were dried over $MgSO_4$ and concentrated *in vacuo*. Purification by column chromatography (hexanes, R_f = 0.14) afforded 230 mg (89 %) as a colorless oil. IR (CCl_4) 3084 (w), 3056 (w), 2967 (s), 2944 (s), 2924 (m), 2858 (w), 1584 (s), 1561 (s) cm^{-1}. ^1H NMR ($CDCl_3$) d 1.82 (s, 3H), 1.83 (s, 3H), 2.42 (s, 3H), 3.03 (s, 3H), 7.05 (d, *J* = 7 Hz, 2 H), 7.33 (d, *J*

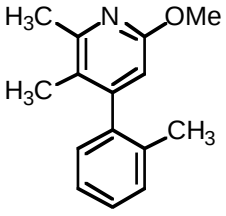
= 7 Hz, 1H), 7.40 (t, *J* = 7 Hz, 2H). ^{13}C NMR ($CDCl_3$) d 13.5, 16.3, 23.1, 53.6, 115.6, 121.9, 127.5, 128.8, 128.9, 140.9, 120.7, 152.0, 160.0. EI-MS *m/z* 227 (70), 226 (100). HRMS for $C_{15}H_{17}NO$ calcd 226.1232, found 226.1228

Entry 1



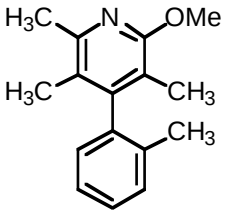
White crystalline solid, mp 52-54 °C. R_f = 0.20 (19:1 hexanes:EtOAc). IR (CCl_4) 3087 (w), 3060 (m), 2948 (m), 2850 (w), 1592, (s). ^{1}H NMR ($CDCl_3$) d 2.06 (s, 3H), 2.46 (s, 3H), 3.90 (s, 3H), 6.45 (s, 1H), 7.25 (dd, 2H, *J* = 10 Hz, *J* = 2 Hz), 7.33-7.24 (m, 3H). ^{13}C NMR ($CDCl_3$) d 14.4, 20.1, 52.3, 106.6, 120.2, 126.2, 127.2, 127.6, 139.2, 151.7, 153.8, 160.3. EI-MS *m/z* 211 (93), 212 (100), 184 (49), 183 (55), 128 (49). HRMS for $C_{14}H_{15}NO$ calcd 212.1075, found 212.1064.

Entry 2



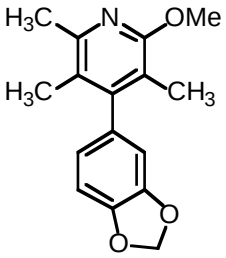
Colorless oil. ^{1}H NMR ($CDCl_3$) d 1.86 (s, 3H), 2.04 (s, 3H), 2.46 (s, 3H), 3.90 (s, 3H), 6.36 (s, 1H), 7.02 (d, 1H), 7.23 (m, 3H).

Entry 4



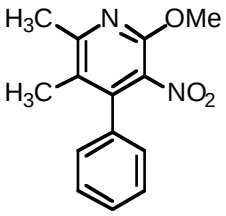
Light yellow oil. R_f = 0.15 (hexanes). IR (CCl_4) 3072 (w), 3002 (m), 2921 (m), 1581 (m). ^1H NMR ($CDCl_3$) d 1.76 (s, 3H), 1.77 (s, 3H), 1.94 (s, 3H), 2.42 (s, 3H), 3.96 (s, 3H), 6.91 (d, 1H), 7.24 (m, 3H); ^{13}C ($CDCl_3$) d 13.1, 15.8, 19.8, 23.0, 53.5, 115.5, 121.9, 126.4, 127.9, 128.6, 130.4, 135.6, 139.6, 150.8, 151.5, 160.1. EI-MS *m/z* 241 (100), 240 (84), 226 (94), 216 (65).

Entry 6



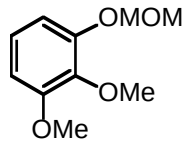
Faint yellow oil. R_f = 0.32 (hexanes). IR (CCl_4) 3072 (w), 3002 (m), 2948 (s), 2920 (s), 2829 (s), 1581 (m). ^1H NMR ($CDCl_3$) d 1.79 (s, 3H), 1.81 (s, 3H), 2.35 (s, 3H), 3.87 (s, 3H), 5.93 (s, 2H), 6.43 (d, *J* = 7 Hz, 1H), 6.44 (s, 1H), 6.79 (d, *J* = 7 Hz, 1 H) ^{13}C NMR ($CDCl_3$), d 13.5, 16.4, 23.1, 53.6, 101.5, 108.8, 109.5, 116.0, 122.1, 122.2, 133.7, 147.0, 148.1, 150.1, 151.6, 160.0. EI-MS *m/z* 271 (74), 270 (100). HRMS for $C_{16}H_{17}NO_3$ calcd 270.1130, found 270.1125.

Entry 7



White, crystalline solid, m.p. = 79-82 °C. R_f = 0.19 (hexanes). IR (CCl_4) 3087 (w), 3064 (w), 3025 (w), 2990 (w), 2901 (w), 2874 (w), 1596 (m), 1557 (s), 1538 (s). ^{1}H NMR ($CDCl_3$) d 1.95 (s, 3H), 2.50 (s, 3H), 4.00 (s, 3H), 7.16 (m, 2H), 7.40 (m, 3H). ^{13}C NMR ($CDCl_3$), d 16.0, 23.7, 54.6, 123.2, 128.6, 129.1, 129.3, 133.9, 134.4, 144.3, 152.1, 157.1. EI-MS *m/z* 258 (100) 211 (57). HRMS for $C_{14}H_{14}N_2O_3$ calcd 258.1004, found 258.0997.

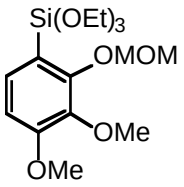
MOM-protected phenol



To 620 mg (0.026 mol) of NaH, which had been washed 2x with 4 mL of hexanes, was added 40 mL of DMF and the resulting suspension cooled to 0° C. A solution of 2.87 g (0.19 mol) of the phenol in 10 mL of DMF was added and the resulting solution stirred at 0° C for 30 min. 2.0 mL (0.026 mol) of MOM-Cl was added which caused an immediate evolution of gas. The solution was allowed to warm to room temperature, stirred for 1 h, and quenched with 50 mL of water. The product was extracted 3x with 100 mL of Et_2O and the combined organic extracts dried and concentrated *in vacuo* to afford 2.70 g (76 %) of MOM-protected phenol as a pale yellow oil which was used without further purification. The spectral data matched that of the

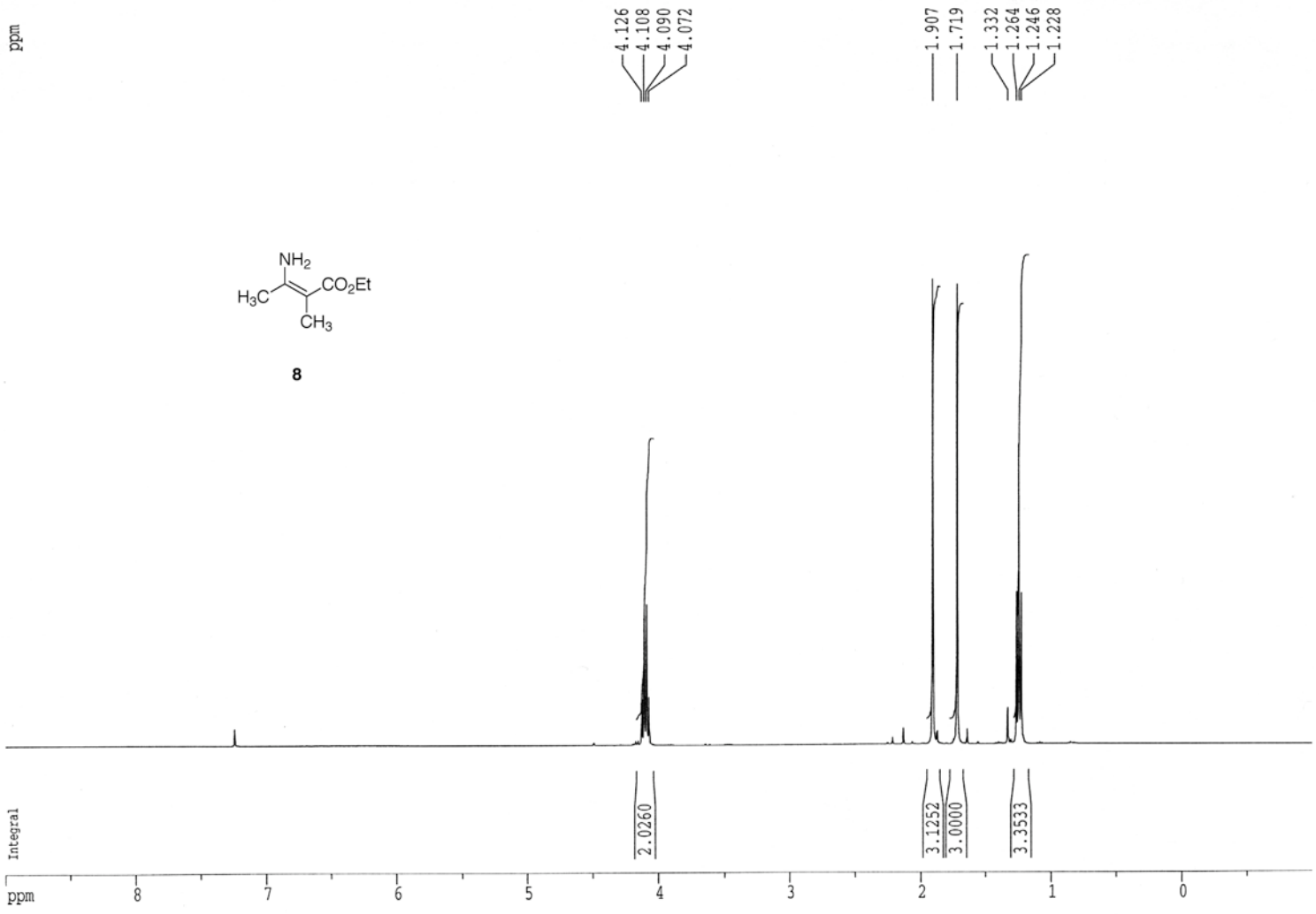
reported compound. IR (CCl_4) 2998 (m), 2955 (s), 2932 (s), 2834 (m), 1596 (s). ^{1}H NMR ($CDCl_3$) d 3.47 (s, 3H), 3.82 (s, 3H), 3.83 (s, 3H), 5.18 (s, 2H), 6.58 (d, *J* = 8 Hz, 1H), 6.74 (d, *J* = 8 Hz, 1H), 6.92 (t, *J* = 8 Hz, 1 H). ^{13}C NMR $CDCl_3$ d 56.4, 56.6, 61.3, 95.7, 106.7, 109.9, 124.1, 139.6, 151.4, 154.1.

Siloxane

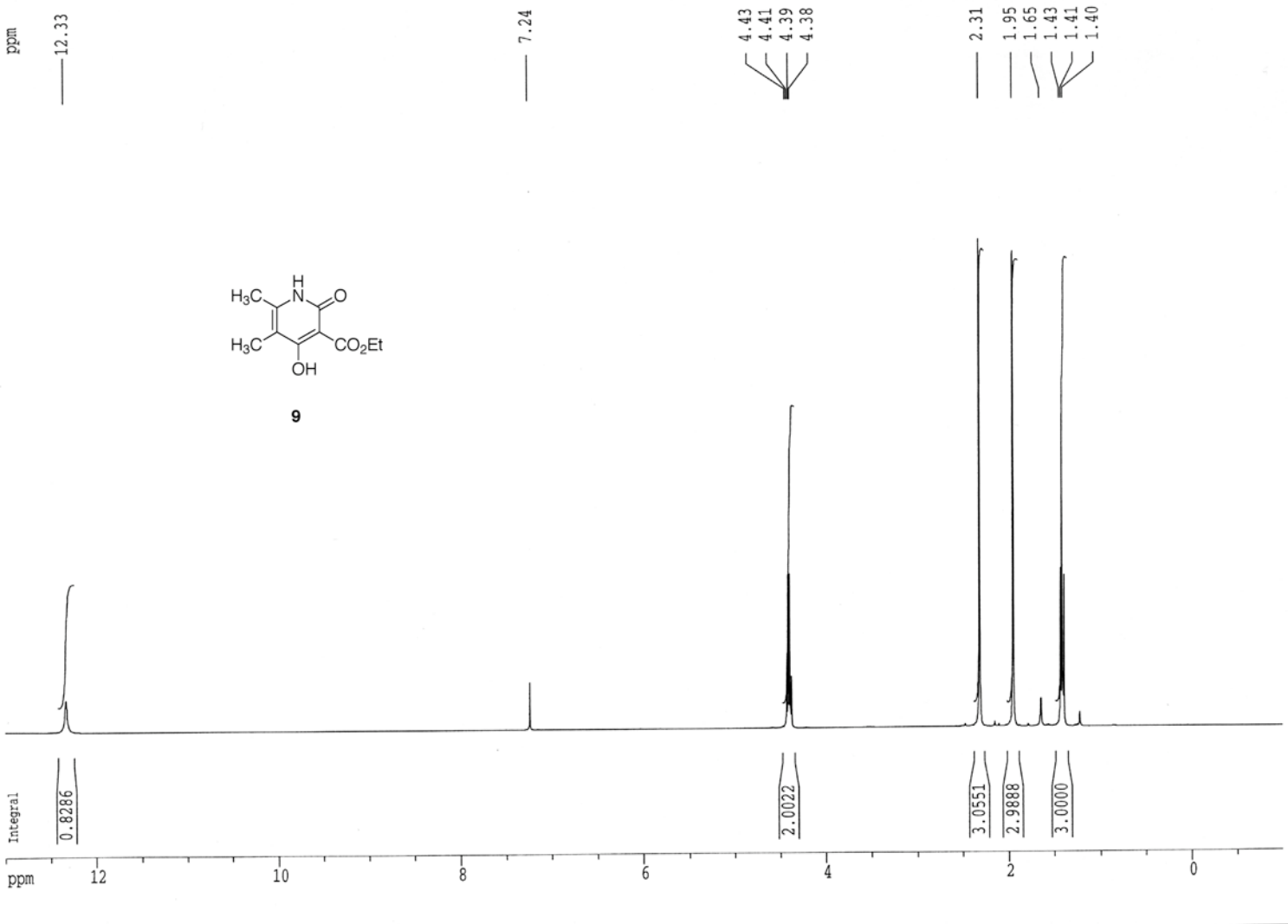


15.3 mL (0.014 mol) of a 0.8M solution of BuLi in hexanes was added dropwise to a solution of 1.88 g (0.0095 mol) of the MOM-protected phenol and 2.2 mL (0.014 mol) of TMEDA in 40 mL of THF at -78 °C. The resulting solution was stirred at -78 °C for 10 min and then allowed to warm to 0 °C and stirred an additional 2 h. This solution was added over 30 min to 4.3 mL (0.019 mol) of $Si(OEt)_4$ in 40 mL of THF at –78 °C. The resulting solution was allowed to warm to room temperature, quenched with 80 mL of water, and extracted 3x with 160 mL of Et_2O. The combined organic extracts were dried and concentrated *in vacuo*. Column chromatography (19:1 hexanes:EtOAc) afforded 850 mg (25 %) (R_f = 0.24) of the siloxane as a pale yellow oil. IR (CCl_4) 2971 (s), 2924 (s), 2889 (s), 2835 (m), 1581 (s). ^{1}H NMR ($CDCl_3$) d 1.22 (t, *J* = 7 Hz, 9 H), 3.62 (s, 3H), 3.81 (s, 3 H), 3.85 (s, 3 H), 3.86 (q, *J* = 7 Hz, 6H), 5.17 (s, 2 H), 6.67 (d, *J* = 8 Hz, 1 H), 7.32 (d, *J* = 8 Hz, 1 H). ^{13}C NMR $CDCl_3$ d 18.6, 56.3, 57.9, 59.0, 61.1, 99.6, 108.0, 117.2, 123.6, 141.9, 155.5, 156.6. MS *m/z* (EI) 360 (87), 271 (95), 270 (100), 255 (53), 166 (56), 45 (53). HRMS for $C_{15}H_{28}O_7Si$ calcd 360.1590, found 360.1604.

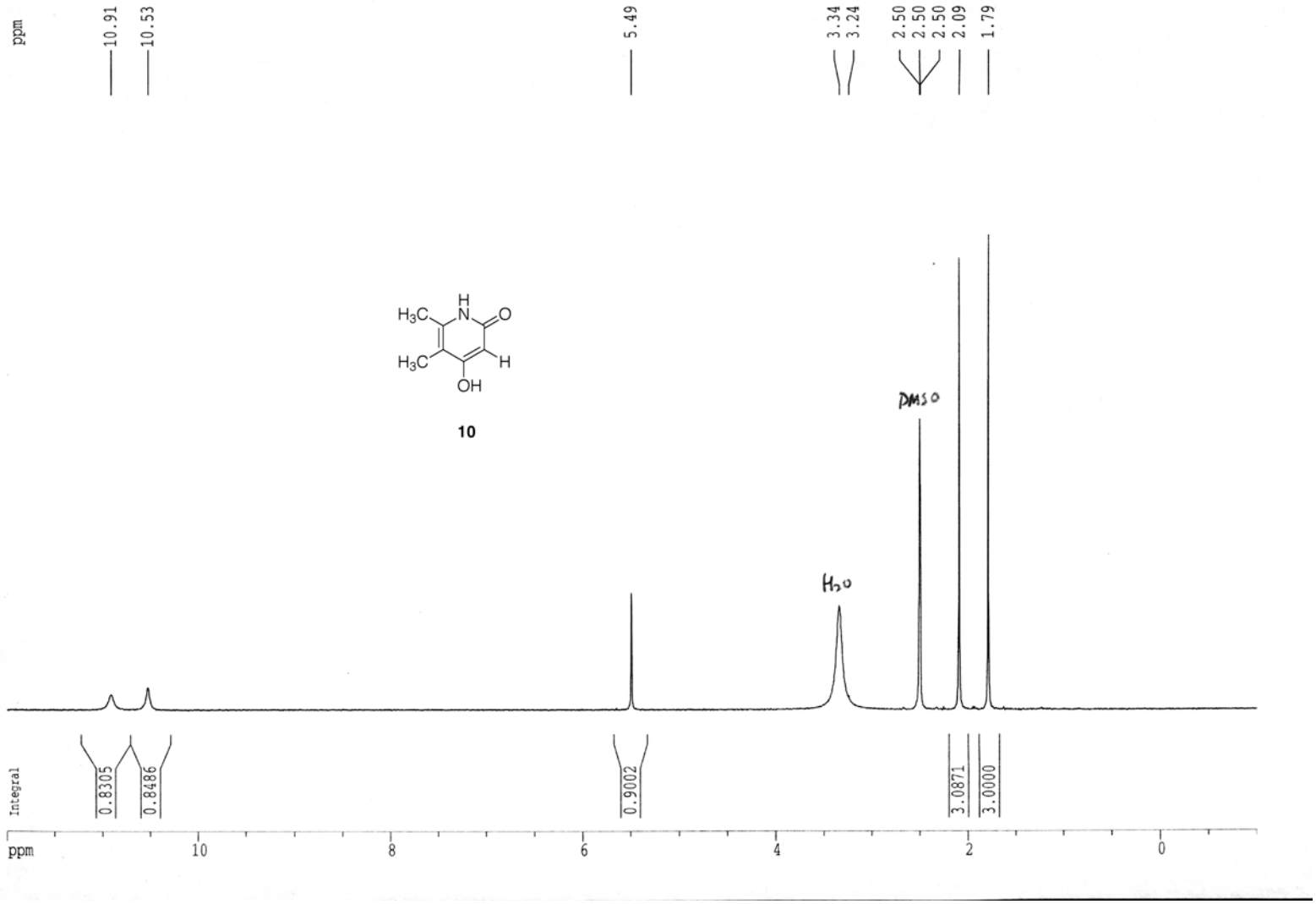
A routine proton parameter from DRX400 MHZ, UMCP.9/1/00



A routine proton parameter from DRX400 MHZ, UMCP.9/1/00

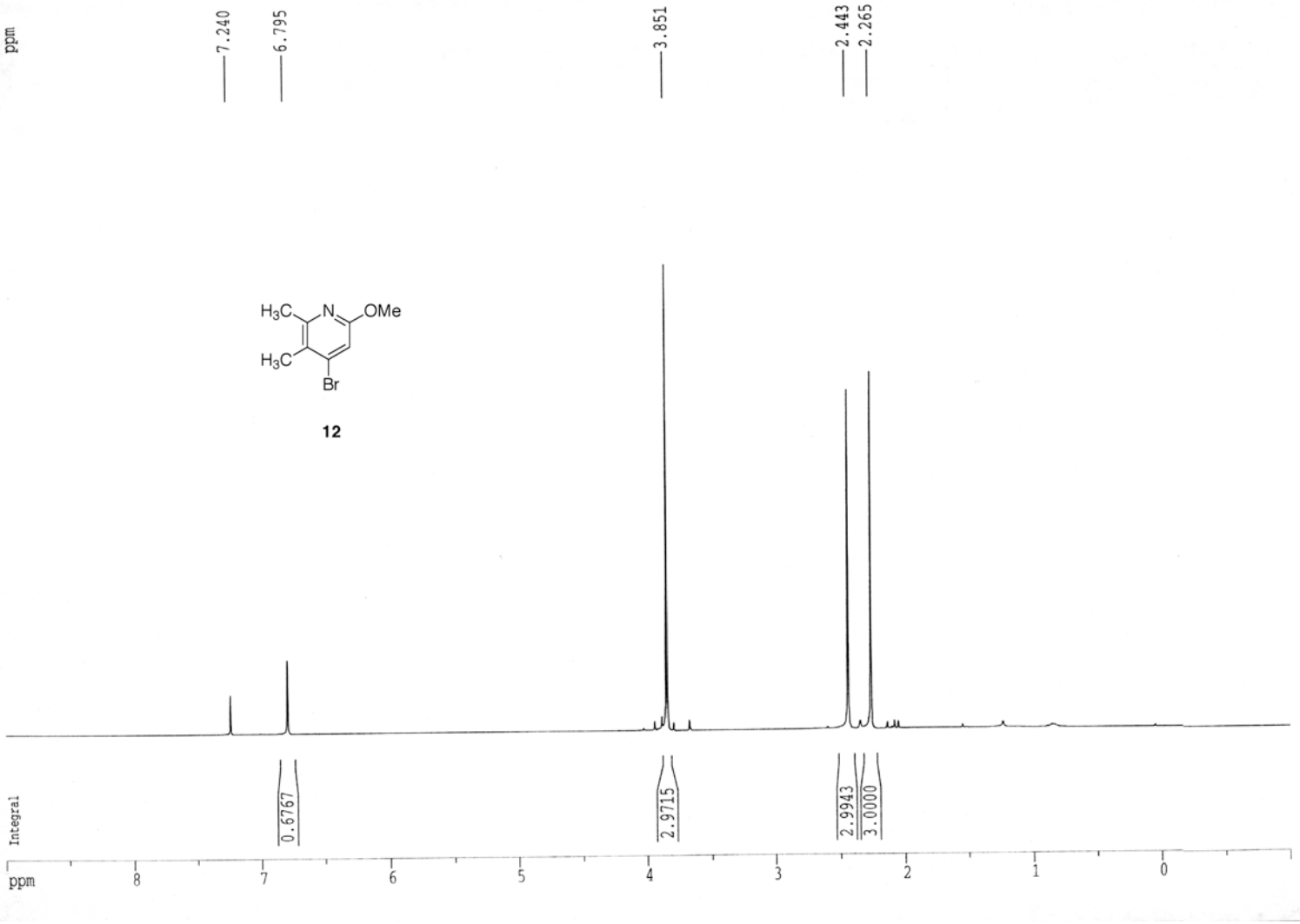


BM - 4 - 01
DMSO - d6

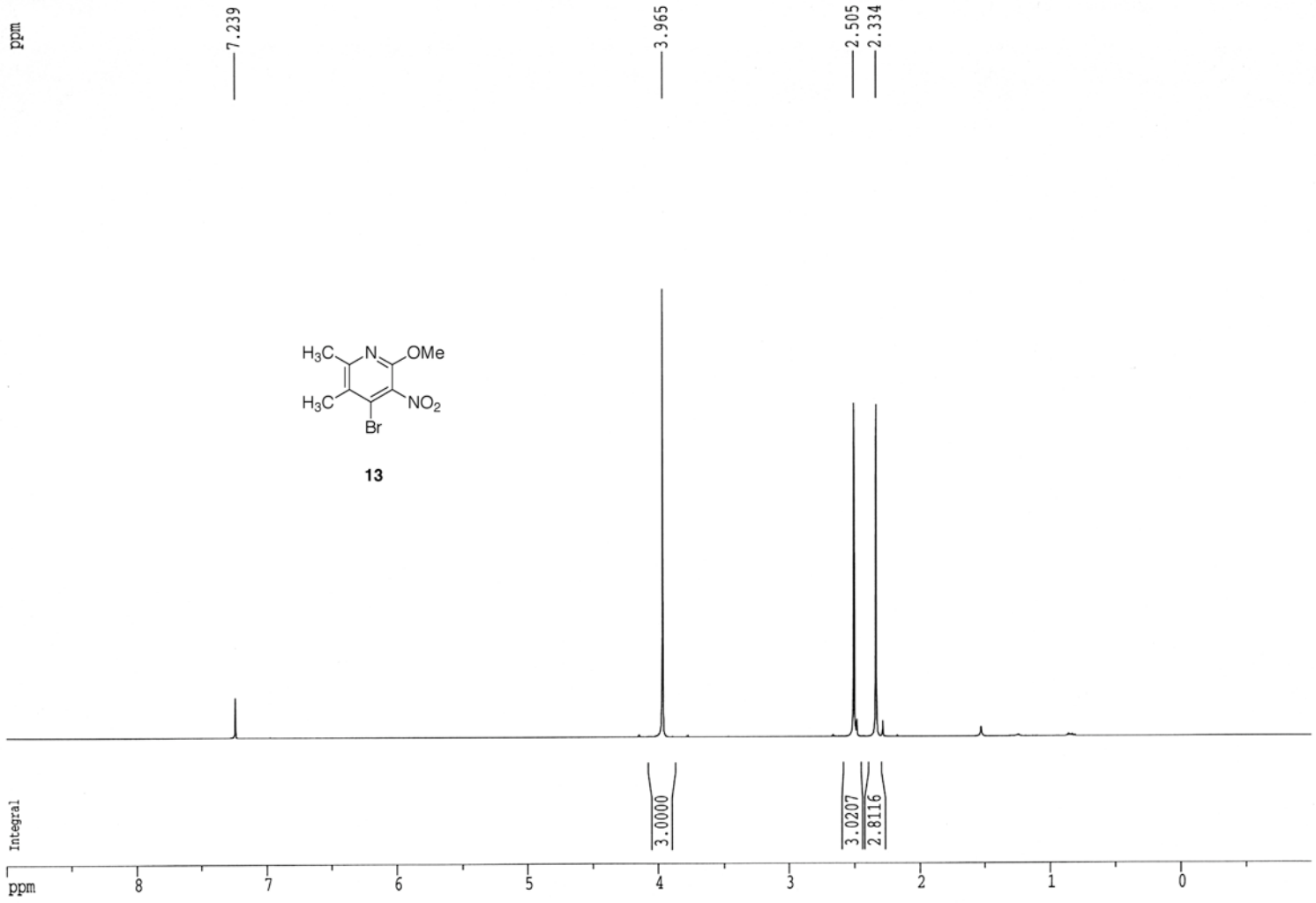


10

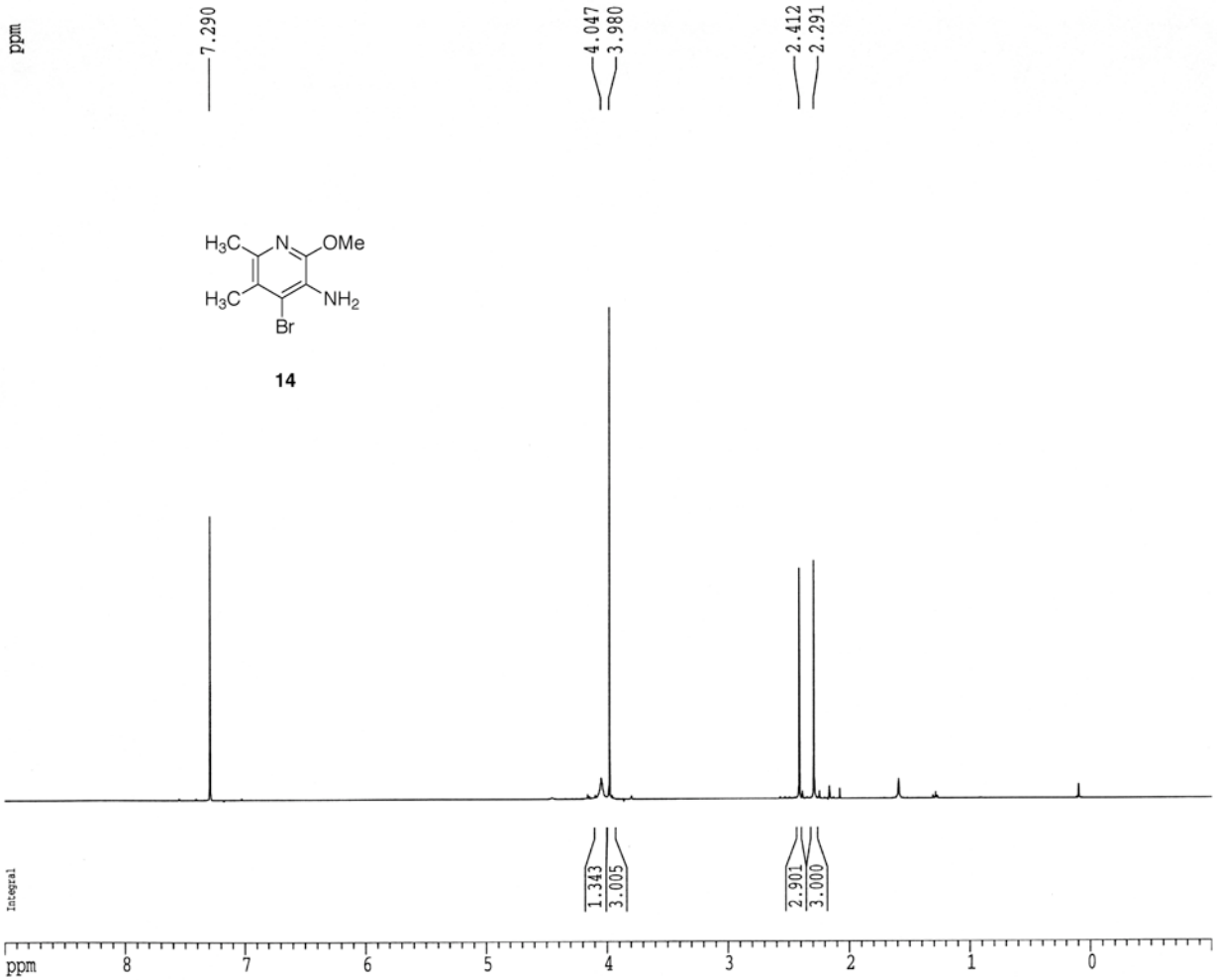
BM - 04 - 18
CDCl3



BM - 04 - 84
CDCl3



PROTON_16_no_plot CDCl3 u Mcelroy 6



14

Current Data Parameters

NAME	bm-06-54
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20030718
Time	9.44
INSTRUM	spect
PROBHD	5 mm QNP 1H/1
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	4
SWH	8223.685 Hz
FIDRES	0.125483 Hz
AQ	3.9846387 sec
RG	406.4
DW	60.800 usec
DE	6.00 usec
TE	300.0 K
D1	1.00000000 sec

======== CHANNEL f1 ========

NUC1	1H
P1	9.00 usec
PL1	-4.00 dB
SFO1	399.7324685 MHz

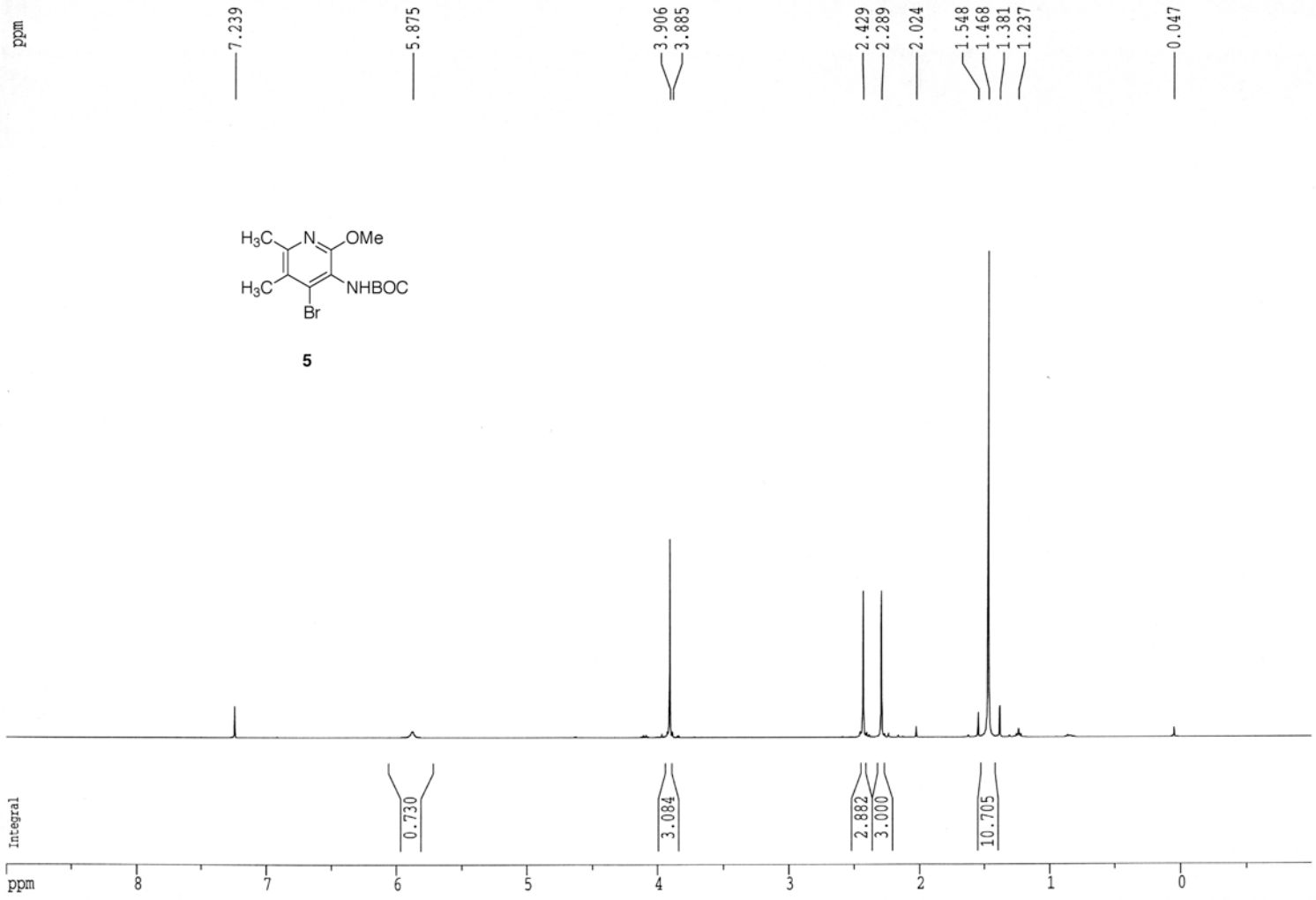
F2 - Processing parameters

SI	32768
SF	399.7300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	2.00

1D NMR plot parameters

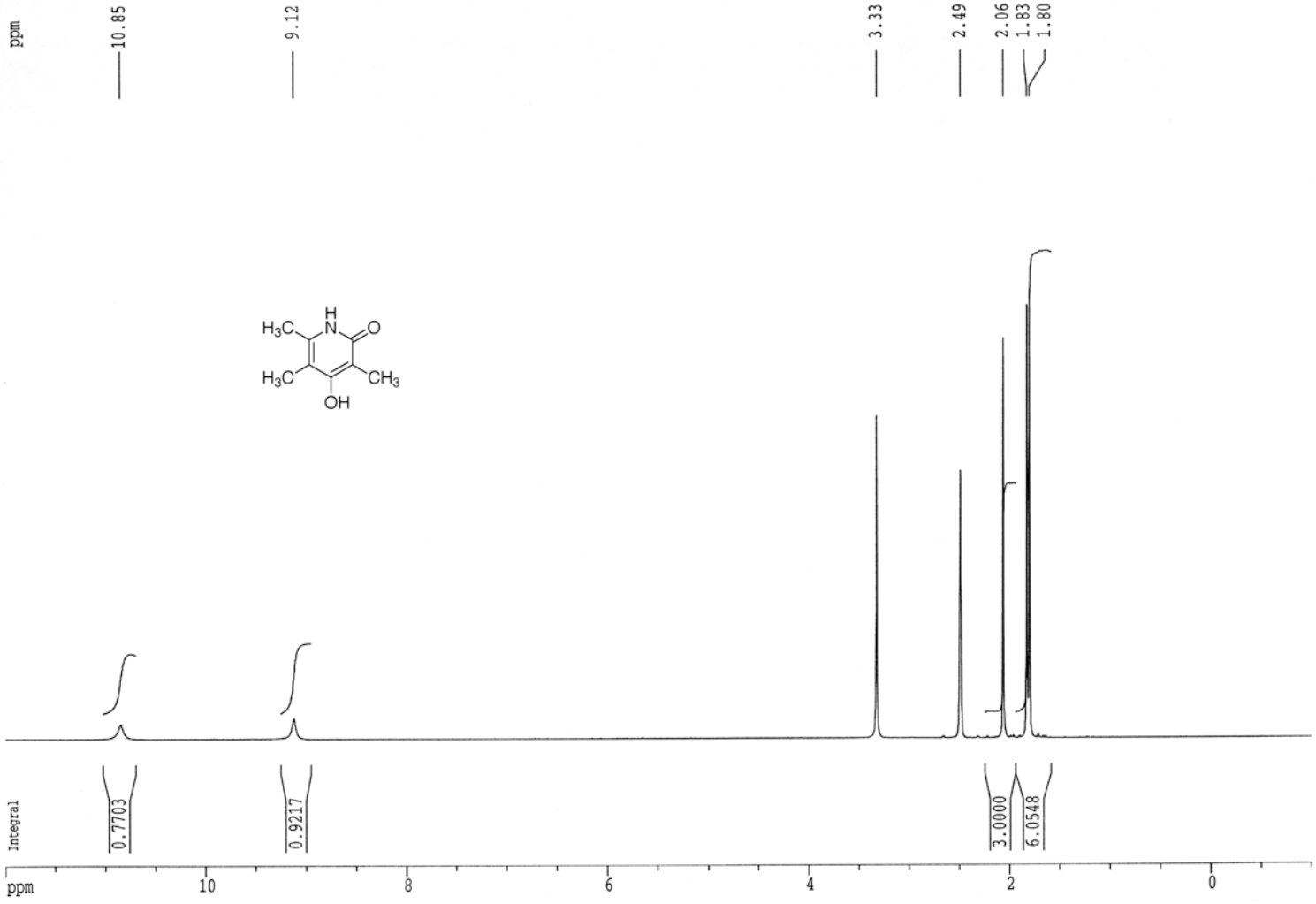
CX	21.00 cm
CY	8.59 cm
F1P	9.000 ppm
F1	3597.57 Hz
F2P	-1.000 ppm
F2	-399.73 Hz
PPMCM	0.47619 ppm/cm
HZCM	190.34763 Hz/cm

BM - 06 - 50
CDC13

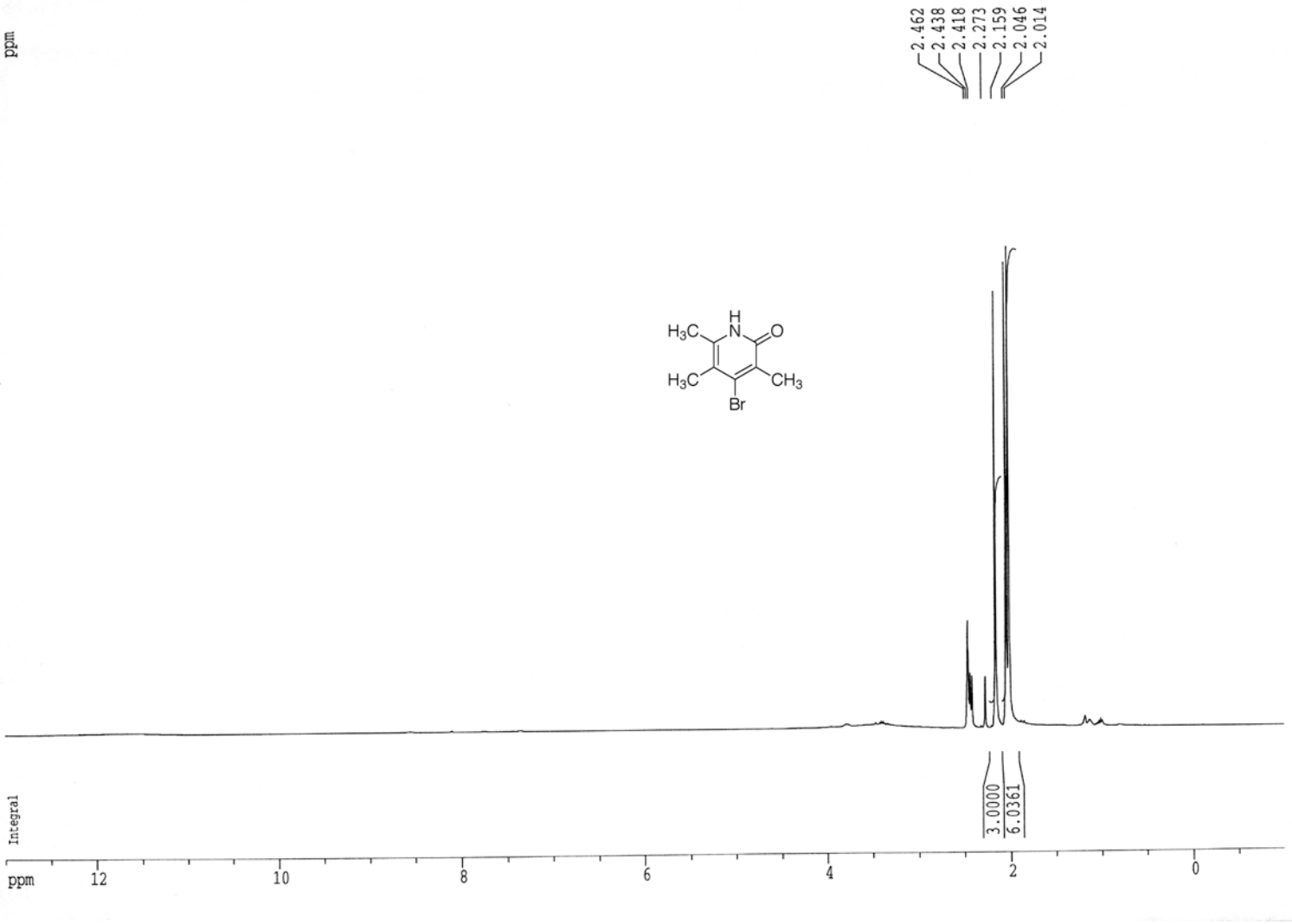


5

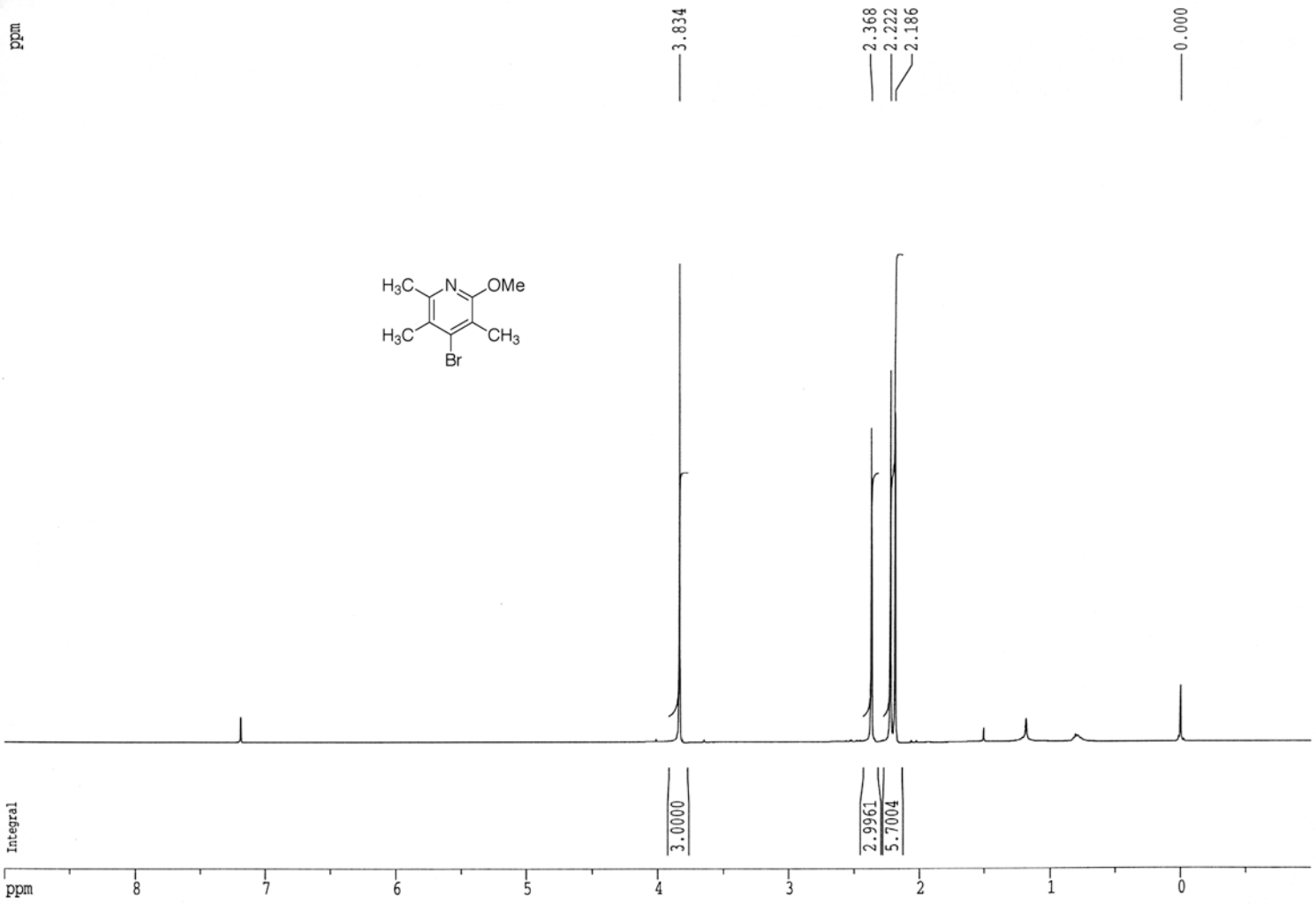
A routine proton parameter from DRX400 MHZ, UMCP.9/1/00

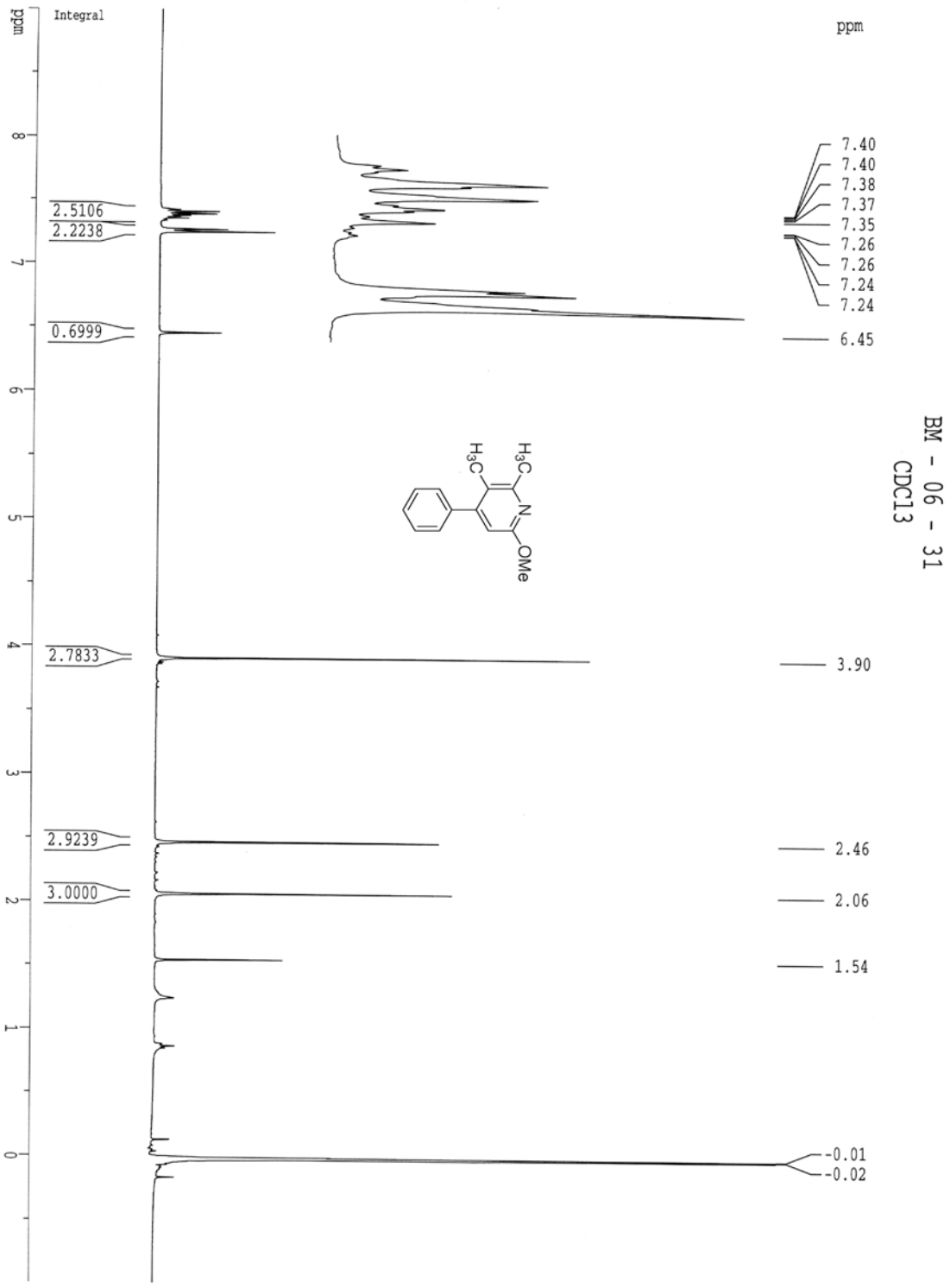


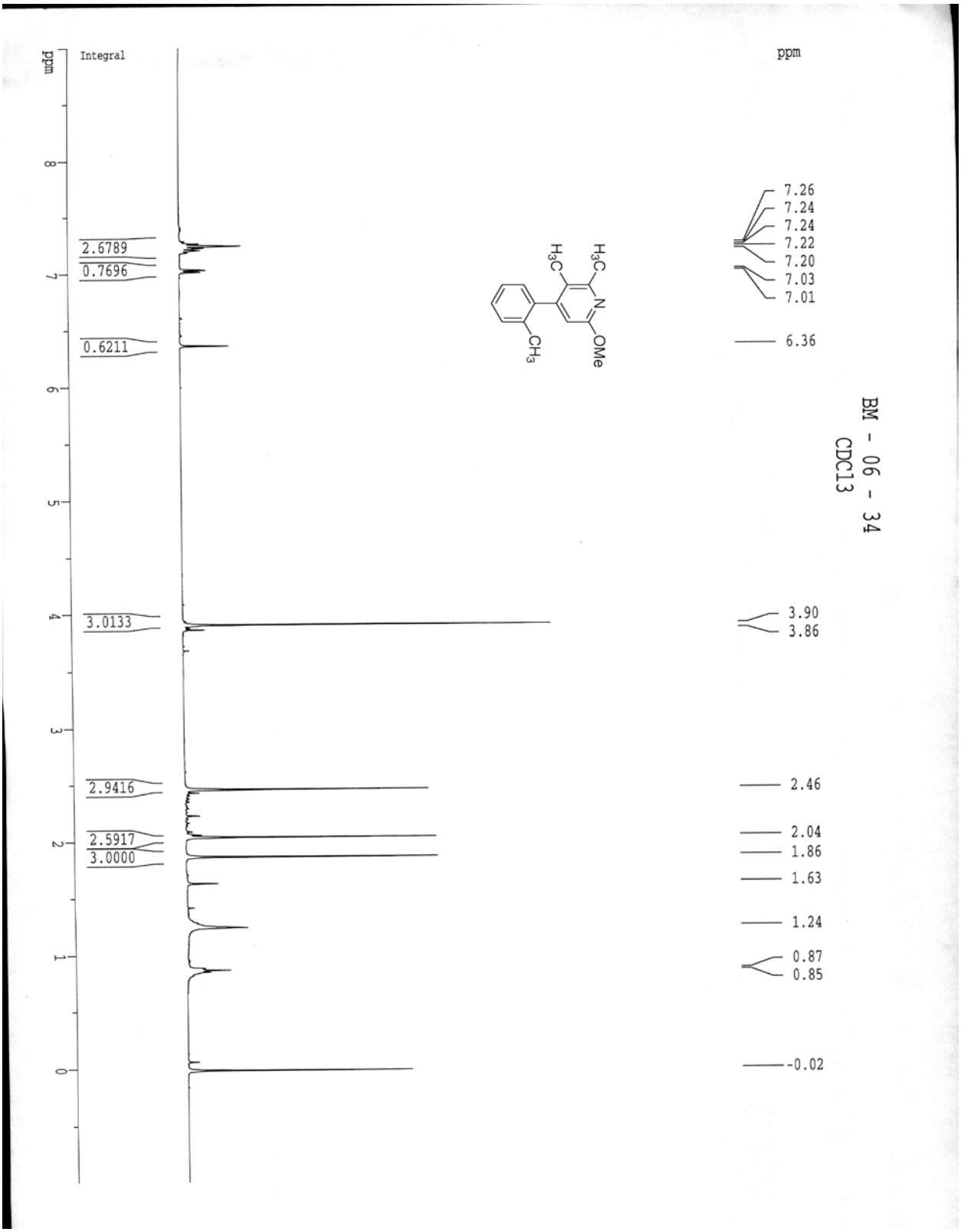
A routine proton parameter from DRX400 MHZ, UMCP.9/1/00

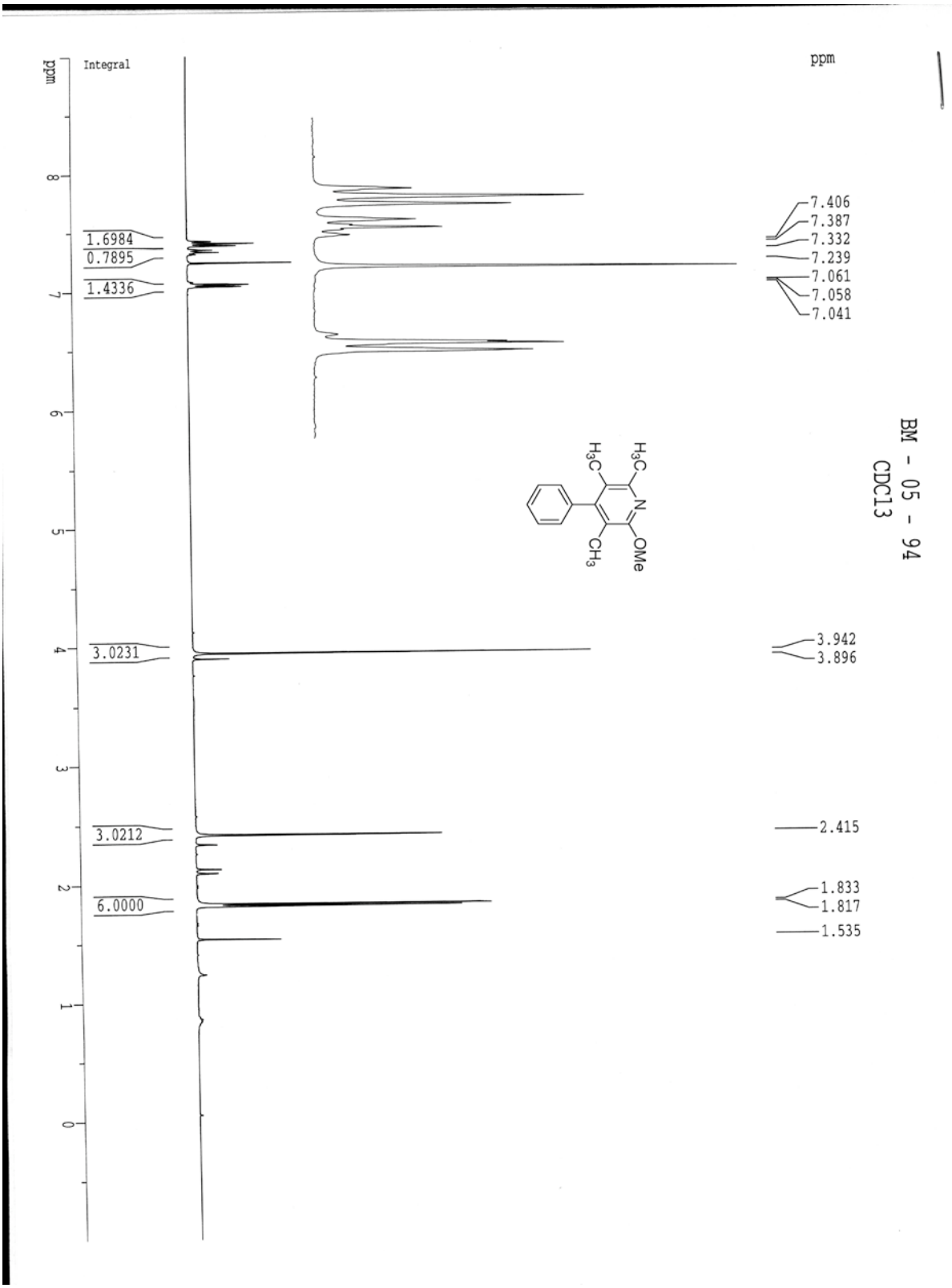


A routine proton parameter from DRX400 MHZ, UMCP.9/1/00

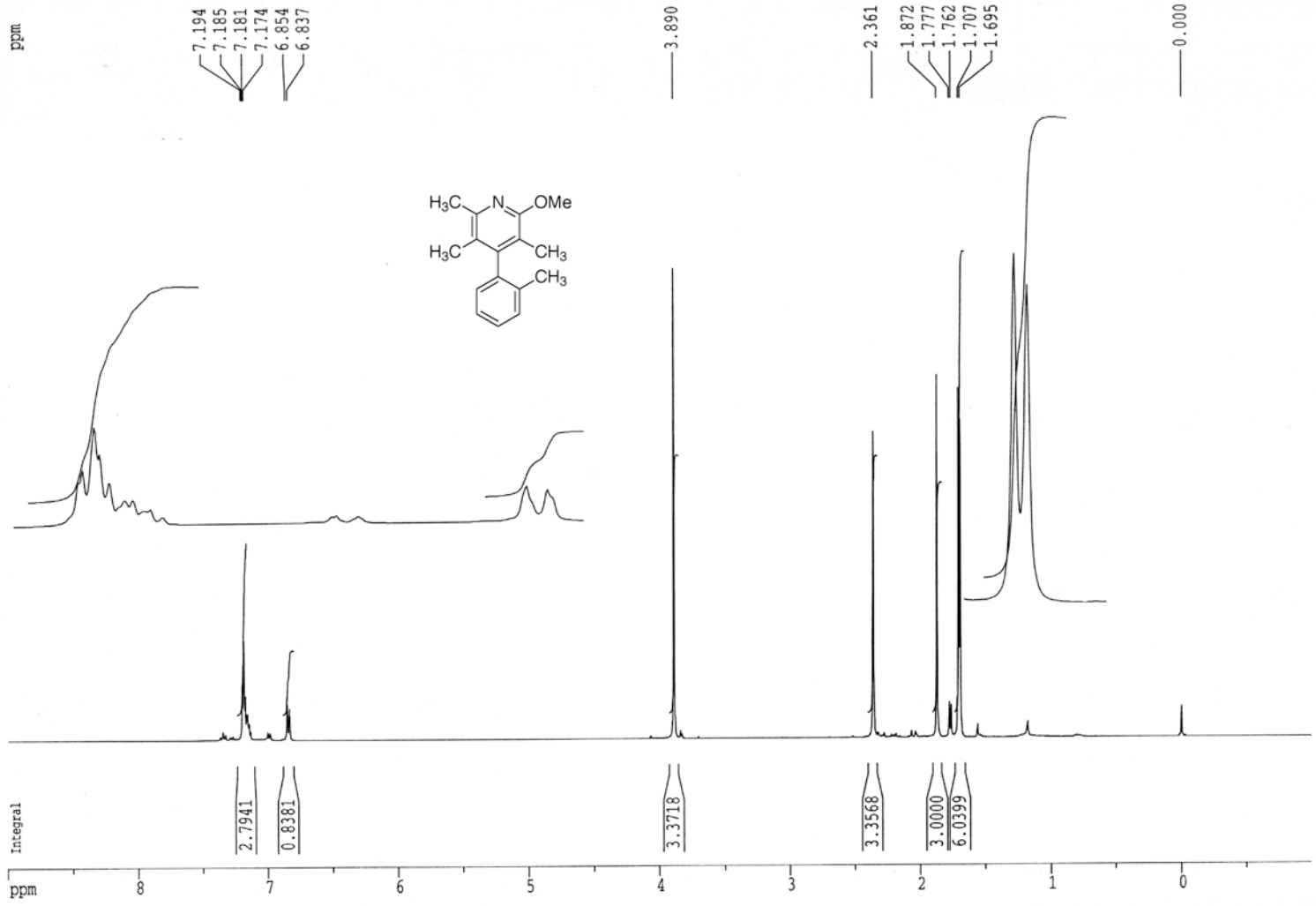



ppm
Integral
ppm
2.5106
2.2238
0.6999
2.7833
2.9239
3.0000
7.40
7.40
7.38
7.37
7.35
7.26
7.26
7.24
7.24
6.45
3.90
2.46
2.06
1.54
-0.01
-0.02
H3C
H3C
N
OMe
BM - 06 - 31
CDCl3

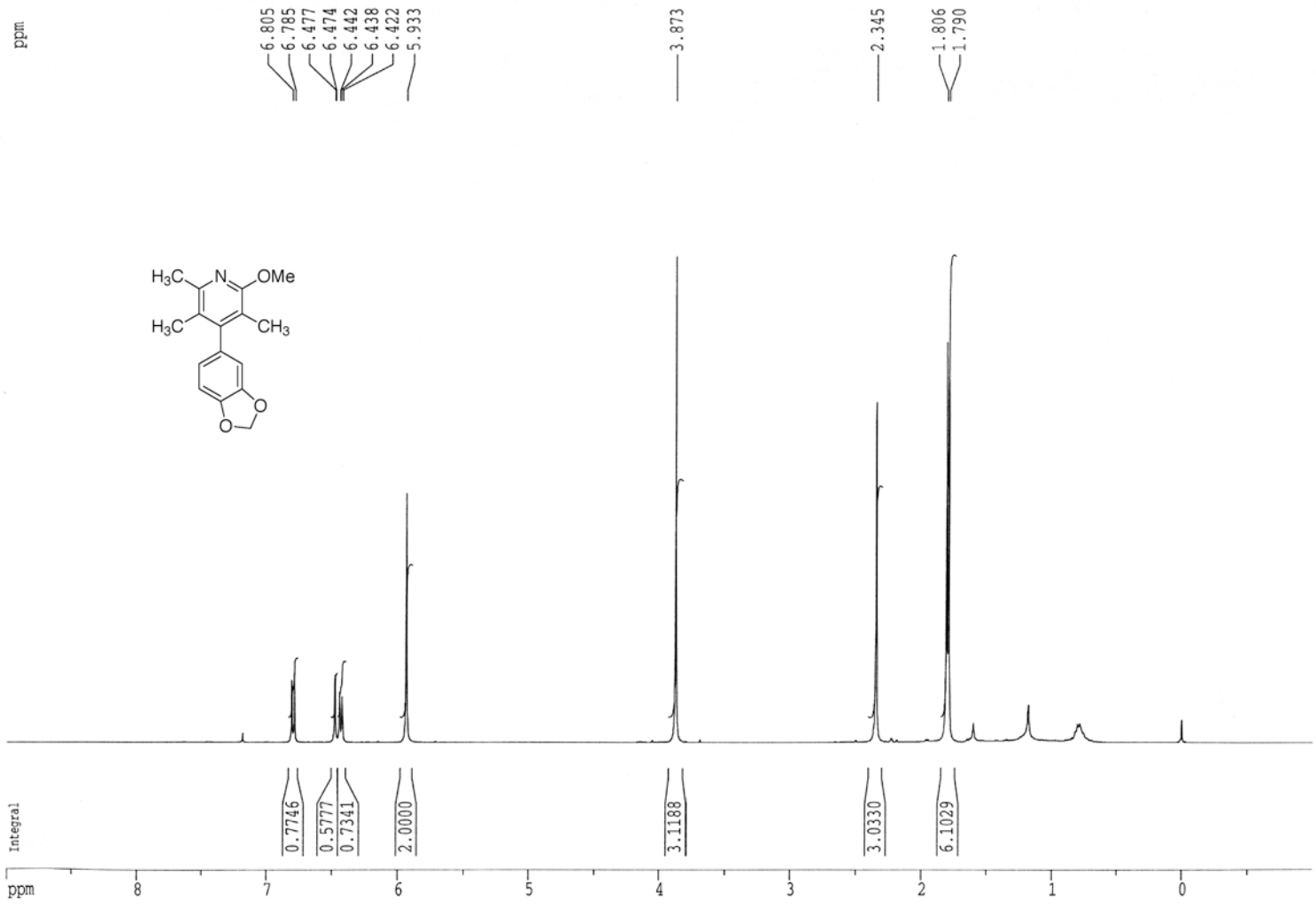

ppm
Integral
8
7
6
5
4
3
2
1
0
2.6789
0.7696
0.6211
3.0133
2.9416
2.5917
3.0000
H3C
H3C
N
OMe
CH3
ppm
7.26
7.24
7.24
7.22
7.20
7.03
7.01
6.36
3.90
3.86
2.46
2.04
1.86
1.63
1.24
0.87
0.85
-0.02
BM - 06 - 34
CDCl3

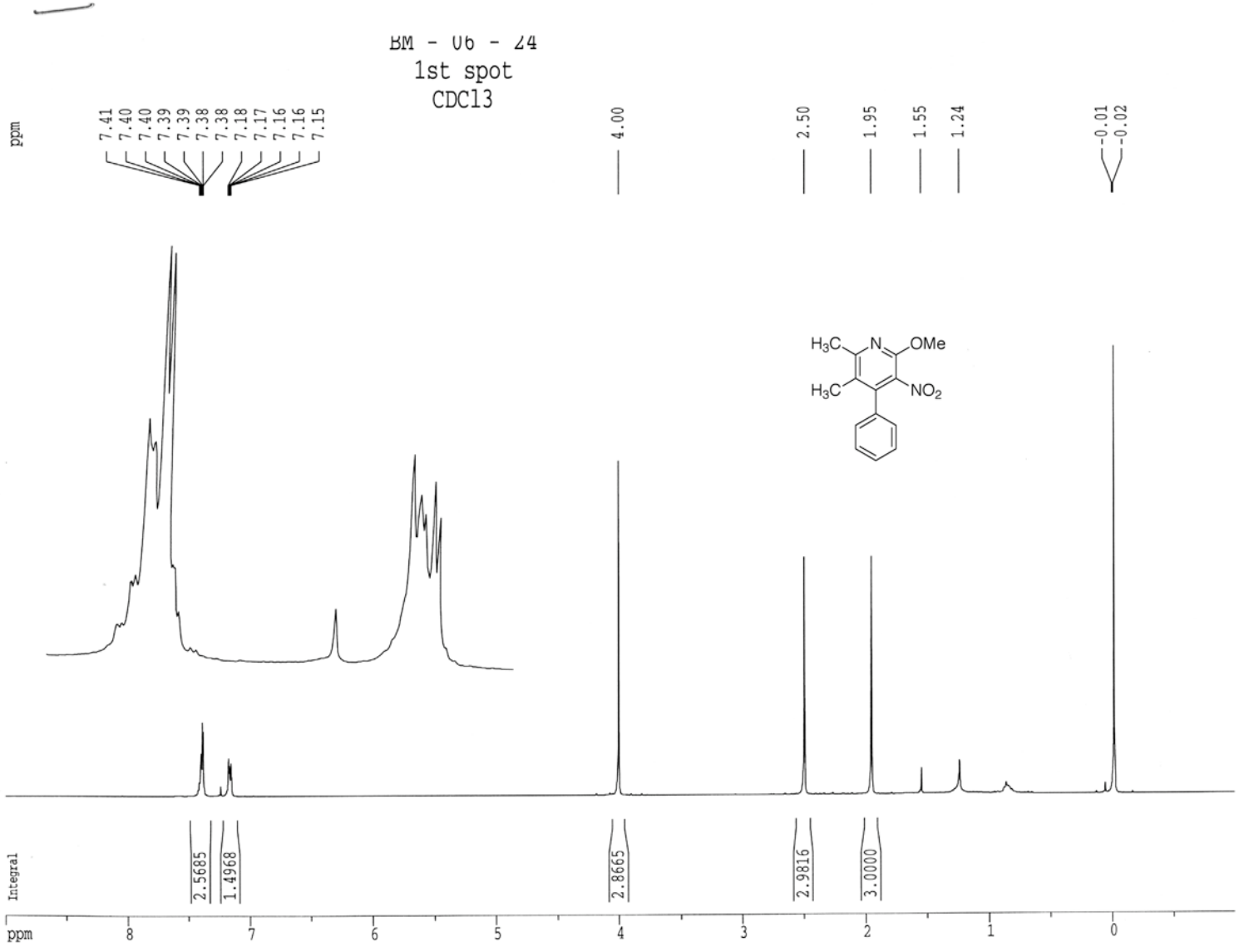

ppm
Integral
1.6984
0.7895
1.4336
3.0231
3.0212
6.0000
ppm
7.406
7.387
7.332
7.239
7.061
7.058
7.041
3.942
3.896
2.415
1.833
1.817
1.535
H3C
H3C
N
OMe
CH3
BM - 05 - 94
CDC13

A routine proton parameter from DRX400 MHZ, UMCP.9/1/00

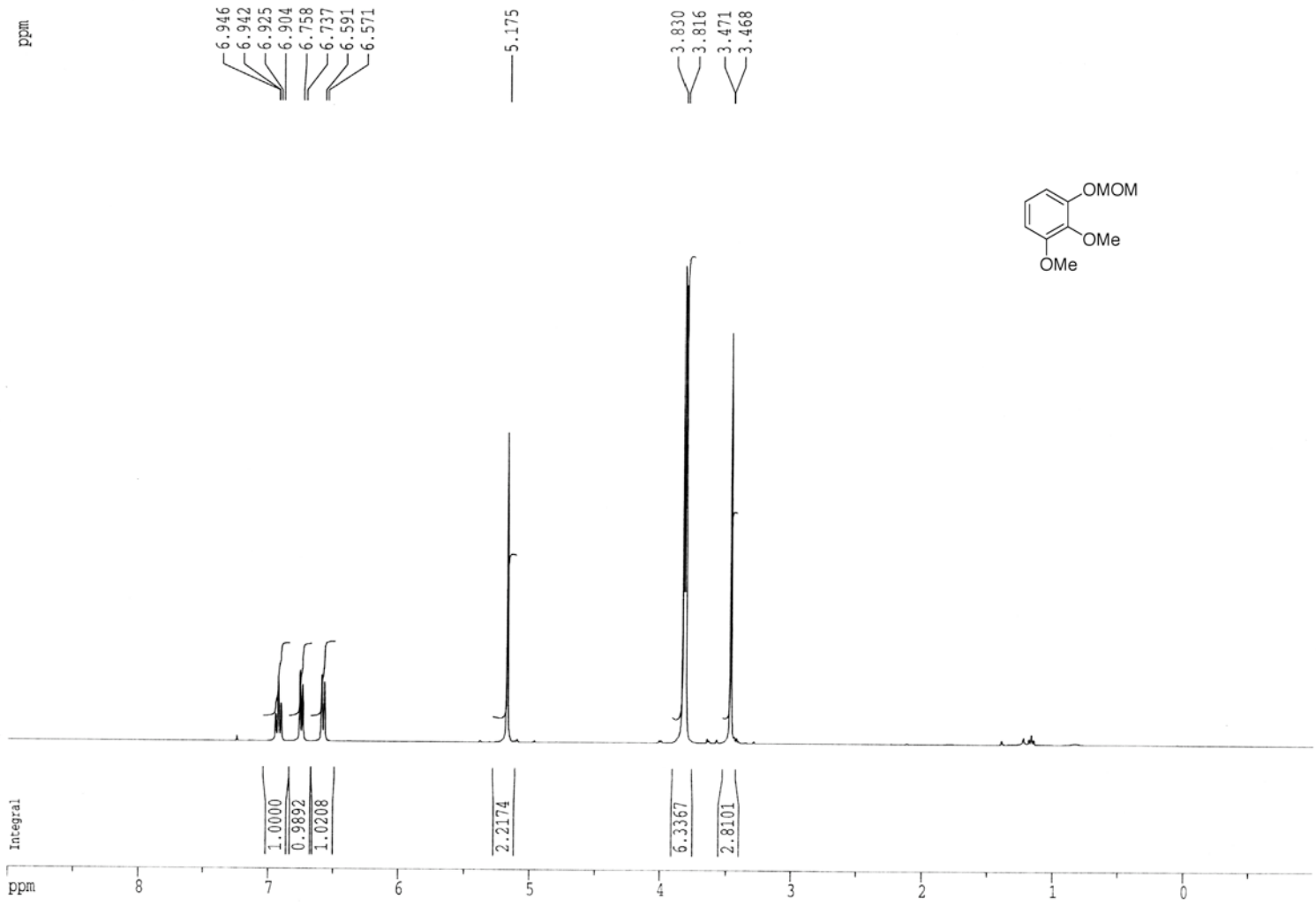


A routine proton parameter from DRX400 MHZ, UMCP.9/1/00




BM - Ub - 24
1st spot
CDCl3
ppm
7.41
7.40
7.40
7.39
7.39
7.38
7.38
7.18
7.17
7.16
7.16
7.15
4.00
2.50
1.95
1.55
1.24
-0.01
-0.02
H3C
N
OMe
H3C
NO2
Integral
2.5685
1.4968
2.8665
2.9816
3.0000
ppm
8
7
6
5
4
3
2
1
0

BM - 04 - 08


ppm
6.946
6.942
6.925
6.904
6.758
6.737
6.591
6.571
5.175
3.830
3.816
3.471
3.468
OMOM
OMe
OMe
Integral
1.0000
0.9892
1.0208
2.2174
6.3367
2.8101
ppm
8
7
6
5
4
3
2
1
0

BM - 04 - 16
CDCl3