Electronic Supporting Information

Palladium-Catalyzed Regio- and Chemoselective *ortho*-Benzylation of C-H Bond Using a Functionalizable Primary Amide Directing Group: A Concise Synthesis of Dibenzo[*b*,*e*]azepin-6-ones

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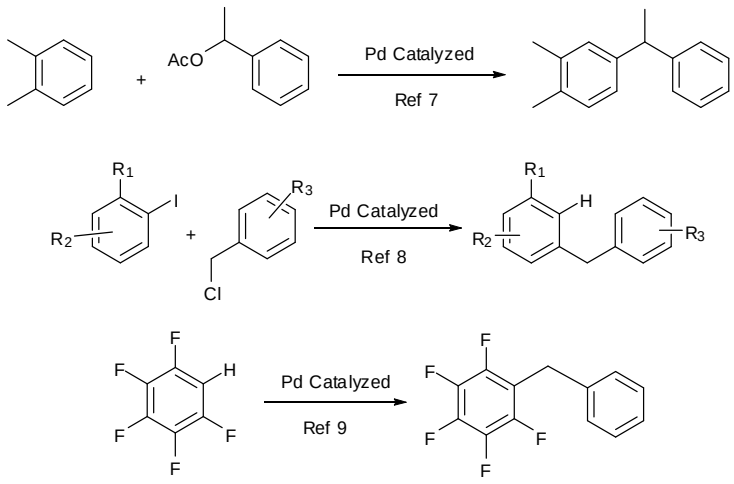
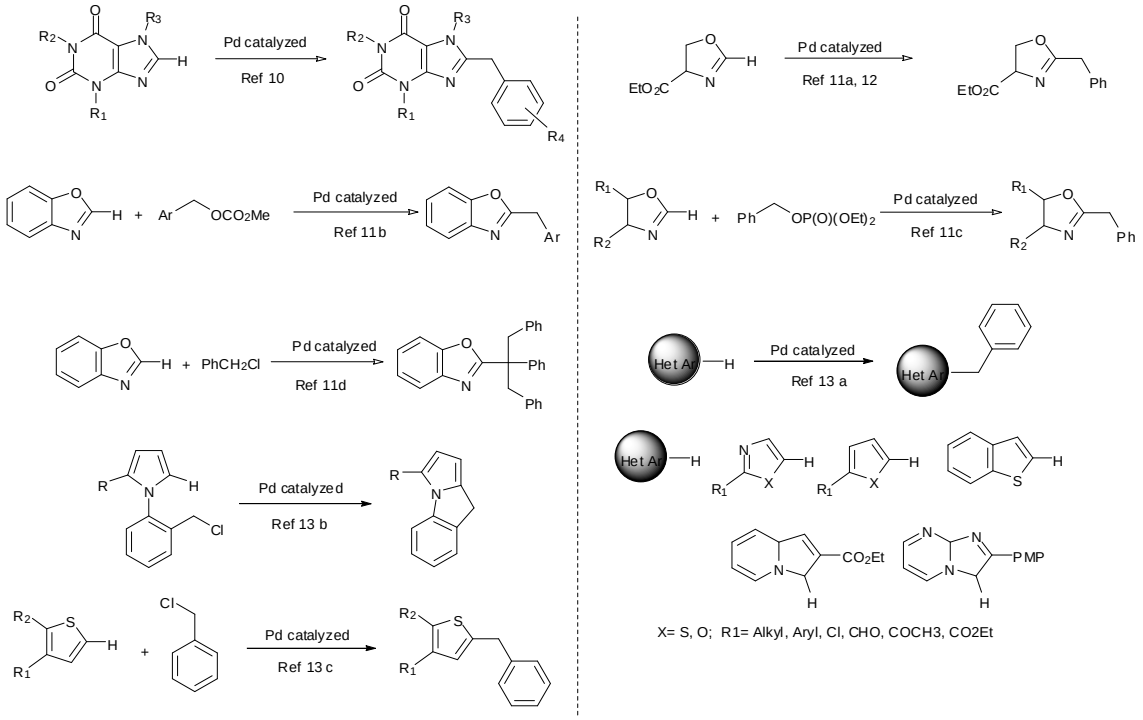
Additional Information on report: Development of an efficient method for the synthesis of functionalized diarylmethanes[1,2] is considered an active research area in direct C-H functionalization. A classical still widely used approach to prepare these structural motifs is Lewis-acid mediated Friedel-Crafts (or S_EAr) reactions of arenes with benzylic electrophiles.[3] While valuable these processes often suffer from several limitations including restrictions to electron-rich arenes, stumpy tolerance to acid-sensitive functional groups, and low chemo- and regioselectivities. Remarkably, some of these limitations have been addressed in the Friedel-Crafts benzylation of activated and deactivated arenes using Lewis acid $BF_3{\cdot}OEt_2$.[4] Recently, transition-metal catalyzed cross-coupling reactions of a stoichiometric organometallic aryl with benzyl halides have emerged as alternatives to the Friedel-Crafts reactions.[5] However, these cross-coupling reactions call for pre-functionalization adding synthetic steps to the preparation of organometallic reagents and demand improved functional groups compatibility. To overcome these drawbacks, transition-metal catalyzed direct benzylation[6] of electron-deficient arenes and heteroarenes have been successfully developed, which includes palladium-catalyzed direct benzylation of arenes,[7,8] pentafluoroarenes (**Scheme 1**),[9] xanthenes,[10] azoles,[11,12] oxazol(in)es,[12] or heteroarenes[7,13] (**Scheme 2**), ruthenium-catalyzed direct benzylation of arenes containing a heterocycle (**Scheme 3**),[14] and cobalt-catalyzed direct benzylation of *N*-pyridinylindole (only one example) with benzyl phosphate (**Scheme 4**).[15]

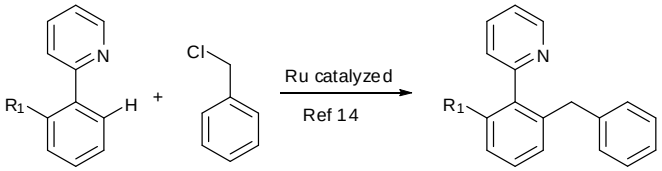
Scheme 1. Pd-catalyzed direct benzylations of arenes



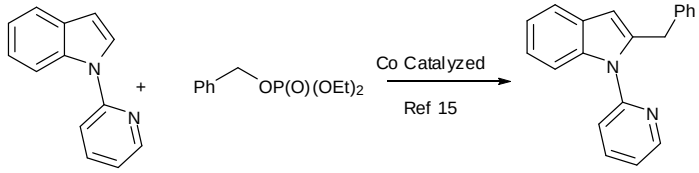
Scheme 2: Pd-catalyzed direct benzylation of heteroarenes



Scheme 3: Ru-catalyzed direct benzylation of arenes

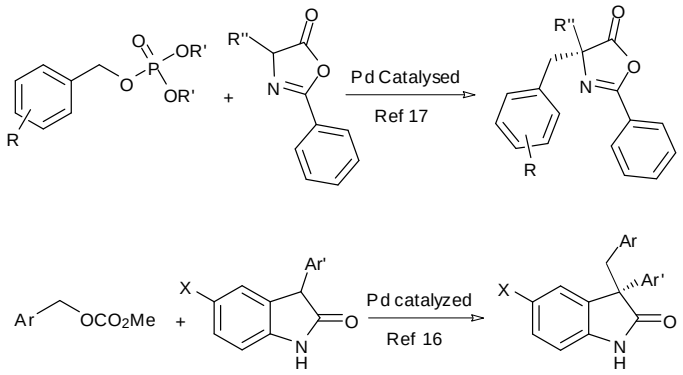


Scheme 4: Co-catalyzed direct benzylation of heteroarenes



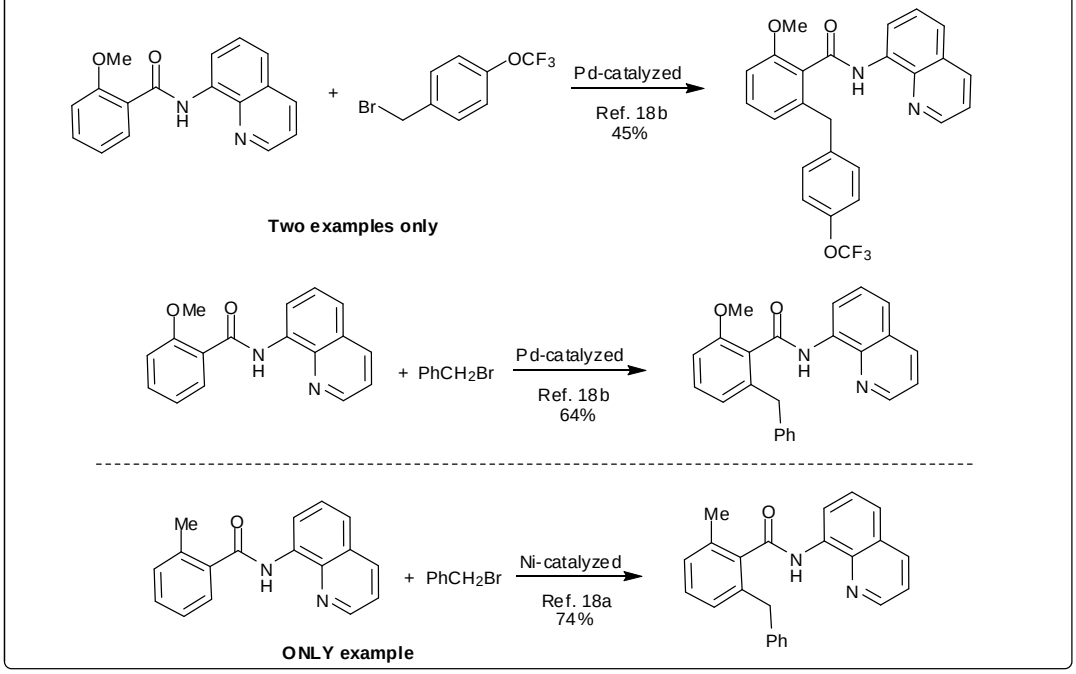
Notably, palladium-catalyzed asymmetric direct benzylation of 3-aryl oxindoles[16] and azlactones[17] have also been realized (**Scheme 5**).

Scheme 5: Asymmetric benzylations of heteroaryls



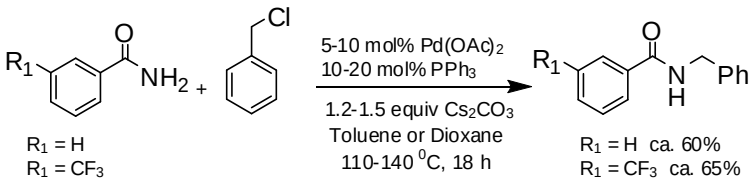
While a wide variety of functional groups have been evaluated as directing group in arene C-H functionalizations, only a few example of *ortho*-benzylation directed by a secondary amide containing an 8-aminoquinoline moiety have appeared. The nickel-[18a] or palladium-[18b] catalyzed direct alkylation of *ortho* C-H bond in secondary benzamides containing an 8-aminoquinoline moiety as a bidentate directing-group has been developed, wherein a few examples of *ortho*-benzylation have been included (**Scheme 6**).

Scheme 6: Pd- or Ni-catalyzed benzylations directed by secondary amide group



At the outset, choice of the appropriate benzylic electrophile for palladium-catalyzed direct benzylation of benzamides was crucial. While transition metal-catalyzed direct benzylation of electron-deficient arenes or (hetero)arenes was successfully achieved with benzyl chloride, only a limited success was documented with benzyl bromide.[9,13a] Initial efforts directed to the development of palladium-catalyzed direct benzylation of benzamides with benzyl chloride invariably gave N-benzylated benzamides as major isolated product (**Scheme 7**).

Scheme 7: Benzylation of primary benzamides with benzyl chlorde

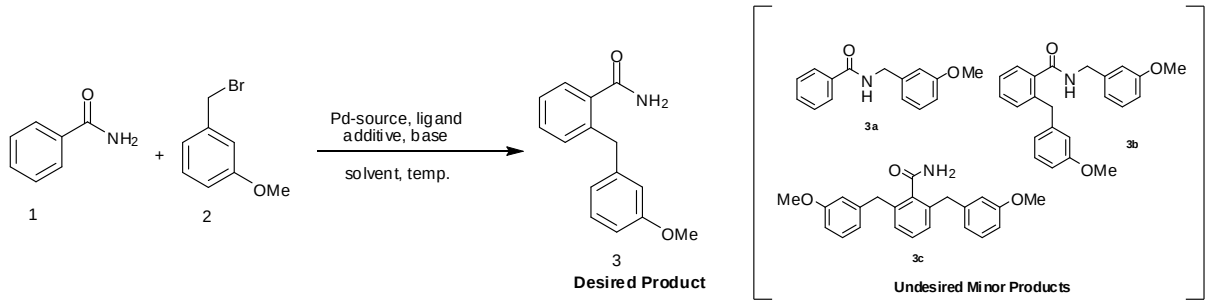


R_1 = H
R_1 = CF_3

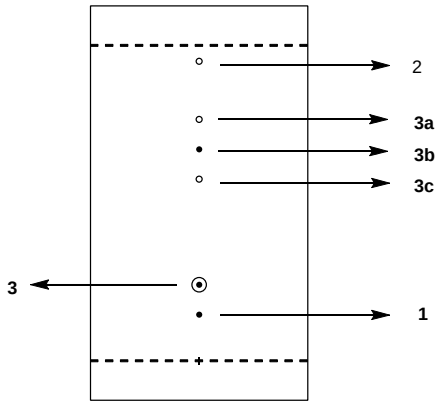
R_1 = H ca. 60%
R_1 = CF_3 ca. 65%

We, therefore, chose 3-methoxybenzyl bromide (**2**) in our optimization study. The reason for choosing **2** as electrophile was two-fold: (1) it is readily available in our laboratory, (2) η^3-benzyl-palladium species generated in situ from **2** by oxidative addition of Pd^0 would be stabilized by the presence of an electron-donating group.[5,6] We began our optimization study using a reaction condition used for the direct benzylation of electron-deficient arenes.[9] Thus, reaction of **1** with **2** did not afford the diarylmethane **3** under the condition described in the literature (Table A, entry 1). However, reaction of **1** with **2** in the presence of 10 mol% $Pd(OAc)_2$, 20 mol% of a trialkylphosphine (PCy_3) or triarylphosphine (PPh_3) ligand, and a strong base (NaOBu-*t*) afforded the desired benzylated product **3** only in detectable amount together with substantial amount of N-benzylated product (30-35%) and other unidentified by-products (entry 2). In addition, use of a mild base Cs_2CO_3 in combination with PCy_3 also produced only a trace amount of diarylmethane **3**, but significantly reduced the amount of N-benzylated product (entry 3). Instead, use of Cs_2CO_3 with PPh_3 produced**3** in 30% yield though a significant amount of starting material remained unreacted even after prolonging the reaction to 36 h (entry 4). Other triarylphosphine ligands [**L1** = P(*o*-Tol)$_3$ or Xant-Phos, BINAP] or biaryldialkylphosphine ligands [**L2** = P(biphen-2-yl)Cy_2, Jhon-Phos, S-Phos, X-Phos, or DPPP] didn't offer any improvement (entry 5-7). The effect of other bases [K_2CO_3, Na_2CO_3, KOAc, K_2HPO_4, TEA, DBU, or DBN] was also examined, but the formation of diarylmethane **3** was observed in 5-20% in these cases (entry 8). Increasing the temperature from 110^0 to 140 ^{0}C using other solvents [*o*-xylene, DMA, DMF, or DME] was detrimental (entry 9). However, the yield of **3**was increased to 45% using dioxane as the solvent (entry 10). A lower catalyst loading (5 mol%) was somewhat better to suppress the formation of undesired products at the cost of unreacted starting material. Thus, compound **3** was best obtained in 60% yield by heating **1** and **2** in the presence of 5 mol% $Pd(OAc)_2$, 10 mol% PPh_3 and 1.2 equiv Cs_2CO_3 in dioxane at 110 ^{0}C (entry 11). It should be noted here in addition to the desired diarylmethane **3**, the formation of N-benzylated, N,C-dibenzylated derivatives, and other unidentified products account for the mass balance of conversion of benzamide into products. For example, 3-trifluoromethylbenzamide produced diarylmethane **6** in 68% yield together with N-benzylated (**SC-1**) and N,C-dibenzylated (**SC-2**) derivatives in 8% and 11% yields, respectively (**Scheme 8**).

Table A: Optimization study for the direct benzylation[a]

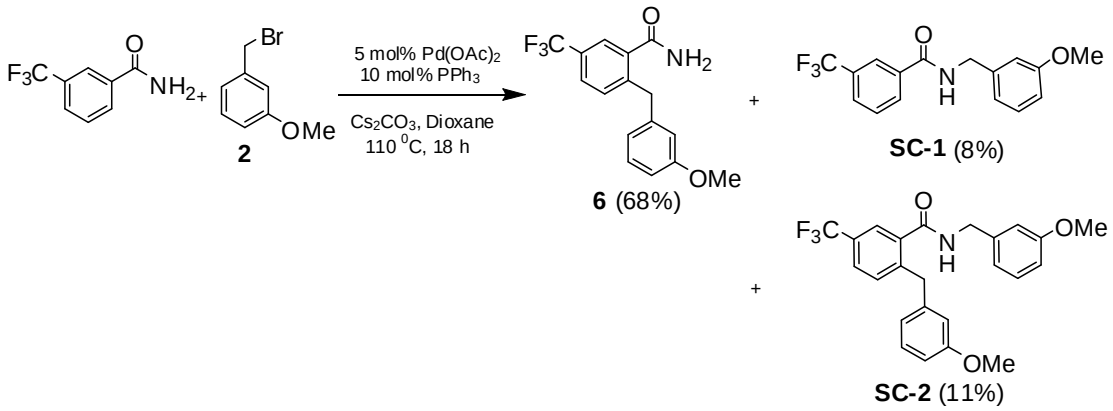


Entry	Metal	Ligand	Base	Solvent	Yield [%]
1[b]	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Toluene	0
2	$Pd(OAc)_2$	PCy_3 or PPh_3	NaOBu-*t*	Toluene	Trace
3	$Pd(OAc)_2$	PCy_3	Cs_2CO_3	Toluene	Trace
4	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Toluene	30
5	$Pd(OAc)_2$	Ligand **L1**	Cs_2CO_3	Toluene	10-15
6	$Pd(OAc)_2$	BINAP	Cs_2CO_3	Toluene	Trace
7	$Pd(OAc)_2$	Ligand **L2**	Cs_2CO_3	Toluene	8-13
8	$Pd(OAc)_2$	PPh_3	Other bases	Toluene	5-20
9[c]	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Other solvents	0-10
10	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Dioxane	45
11[d]	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Dioxane	**60**
12[e]	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Dioxane	38
13[f]	$Pd(OAc)_2$	-	K_2CO_3	t-Amyl-OH	20
14[g]	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Dioxane	30-35
15[h]	$Pd(PPh_3)_4$	-	Cs_2CO_3	Dioxane	52
16	$Pd(OAc)_2$	-	Cs_2CO_3	Dioxane	38
17[i]	$Pd(OAc)_2$	PPh_3	Cs_2CO_3	Dioxane	0

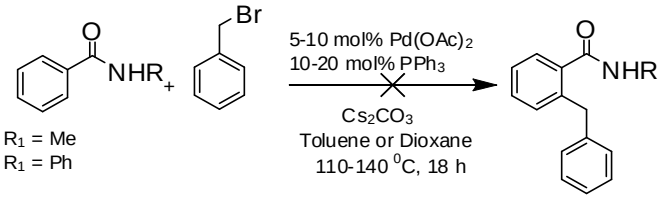


[a] Reagents and conditions: **1** (0.2 mmol), **2** (0.2 mmol), $Pd(OAc)_2$ (0.02 mmol), PPh_3 (0.04 mmol), base (1.2 mmol), solvent (500 mM), temp.110 ^{0}C, 18 h. [b] Pivalic acid (1.2 equiv) as additive. [c] Temp 140 ^{0}C, [d] Used $Pd(OAc)_2$ (0.01 mmol) and PPh_3 (0.02 mmol). [e] Used **2** (2 equiv.), [f] **1** (0.2 mmol), **2** (0.6mmol), base (0.6 mmol), $Pd(OAc)_2$ (5 mol%), Pivalic acid (20 mol%) [g] Used 1.25-2.50 mol% $Pd(OAc)_2$ and 2.5-5.0 mol% PPh_3. [h] Used $Pd(PPh_3)_4$ (0.02 mmol). [i] Employed secondary benzamide (*N*-methylbenzamide or *N*-phenylbenzamide) as one of the coupling partners using 5 mol% $Pd(OAc)_2$ and 10 mol% PPh_3

Schene 8: Products isolated in benzylation of 3-trifluoromethyl benzamide with 3-methoxy benzylbromide



Attempt for complete conversion of starting material into products using excess **2** (2 equiv) was not beneficial (entry 12). However reported experimental condition[18b] resulted in only 20% yield (entry 13). Similarly, further lowering of the catalyst loading to 2.5 mol% or 1.25 mol% was deleterious (entry 14). Replacing $Pd(OAc)_2$by $Pd(PPh_3)_4$ resulted in slightly reduced yield of **3** (entry 15). The optimized condition excluding PPh_3 gave the desired *ortho*-benzylated benzamide in reduced yield (38% vs. 60%) with increased amount of byproduct (C,C-dibenzylated benzamide and C,N-dibenzylated benzamide) (entry 16). A similar observation (reflecting reduced yield) was also recorded in the reaction of 3-trifuorobenzamide and 3-methoxybenzyl bromide under the optimized condition excluding PPh_3 (42% vs. 68%). To understand the role of Ph_3P in this reaction, we performed the following control experiments. An experiment involving reaction of Ph_3P and benzyl bromide in dioxane at 110 ^{0}C for 30 min did not show the presence of Ph_3P on TLC and formed a white colored insoluble material, which indicated the formation of benzyl triphenylphosphonium salt. Another control experiment including $Pd(OAc)_2$, Ph_3P, and benzyl bromide in dioxane at 110 ^{0}C for 1-2 h also did not reveal the presence of Ph_3P. ^{31}P NMR of these reactions also suggested the absence of Ph_3P (^{31}P NMR Spectra Exp-A and Exp-B, respectively). Remarkably, reaction of a secondary benzamide (*N*-methylbenzamide or *N*-phenylbenzamide) with **2** did not give the corresponding direct benzylated product under the best condition (entry 17).



Only a few examples of direct benzylation of activated C-H bonds in (hetero)arenes using 2-, 3- or 4-chlorobenzyl chloride has been documented, which demonstrated chemical selectivity at benzyl chloride over aryl chloride.[13,14] This may be rationalized of the poor reactivity of aryl chloride compared to benzyl chloride leading to chemoselective direct benzylation.

Experimental

General Information: Unless otherwise noted, all reagents and solvents were purchased from commercial sources and used as received. All palladium-catalyzed reactions were degassed with argon and performed in a screw-capped vial. Unless specified, the proton and carbon NMR spectra were obtained in $CDCl_3$ using a 400 MHz spectrometer and are reported in δ units. Coupling constants (*J* values) are reported in Hz. Column chromatography was performed on silica gel (100-200 or 230-400 mesh). High Resolution Mass Spectra (HRMS) were obtained using Bruker-Maxis. IR spectra were obtained using Perkin Elmer-Spectrum II instrument. All melting points were taken using a melting point apparatus equipped with a calibrated thermometer and are uncorrected. New compounds were characterized by melting point, ^{1}H NMR, ^{13}CNMR, IR, and HRMS data.

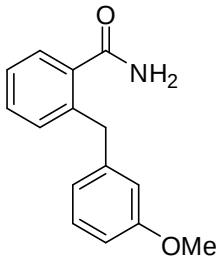
All benzamides and benzyl bromides were purchased from commercial vendors.

General procedure for the palladium-catalyzed direct benzylation of primary benzamides: In an oven-dried screw cap vial equipped with a magnetic stir bar, benzamide (122 mg, 1 mmol) was dissolved in dioxane (2 mL) under nitrogen followed by the addition of Cs_2CO_3 (390 mg, 1.2 mmol), 3-methoxy benzylbromide (201 mg, 1 mmol), $Pd(OAc)_2$ (11.2 mg, 0.05 mmol) and PPh_3 (26 mg, 0.10 mmol). The resulting reaction mixture was heated at 110 ^{o}C for 18 h. The reaction mixture was allowed to cool to room temperature and extracted with ethyl acetate (2 x 50 mL). The organic layers were combined, dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography on silica using(ethyl acetate/ hexane = 1:4) as an eluent to give the desired product.

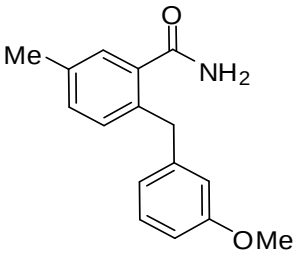
General procedure for the palladium-catalyzed interamolecular *N*-arylation of 2-(2-bromobenzyl) benzamides: In an oven-dried screw cap vial equipped with a magnetic stir bar, 2-(2-bromobenzyl) benzamide (0.25 mmol) was dissolved in dioxane (2 mL) under nitrogen followed by the addition of Cs_2CO_3 (161 mg, 0.5 mmol), $Pd(OAc)_2$ (5.6 mg, 0.025 mmol) and Xant-Phos (20 mg, 0.034 mmol). The resulting reaction mixture was heated at 110 ^{0}C for 18 h. The reaction mixture was allowed to cool to room temperature and extracted with ethyl acetate (2 x 50 mL). The organic layers were combined, dried (Na_2SO_4), concentrated under reduced pressure, and purified by column chromatography on silica gel using (ethyl acetate/ hexane = 1:7) as an eluent to give the cyclized compound.

Characterization Data

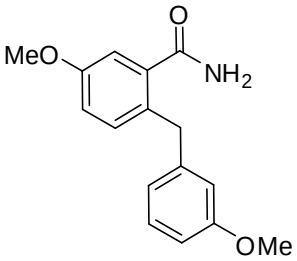
2-(3-Methoxybenzyl)benzamide (**3**): white solid; mp: 109 ^{0}C; ^{1}H NMR: δ 7.50 (d, *J*=7.3 Hz, 1H), 7.37 (d, *J*=7.5 Hz, 1H), 7.16 - 7.32 (m, 3H), 6.72 - 6.77 (m, 3H), 5.90 (br. s, 1H), 5.70 (br. s, 1H), 4.21 (s, 2 H), 3.75 (s, 3H); ^{13}C NMR: δ 172.0, 159.7, 142.3, 138.7, 135.3, 131.2, 130.5, 129.5, 127.4, 126.4, 121.3, 114.8, 111.4, 55.1, 38.8; HRMS obsd 242.1175, calcd 242.1181 for $C_{15}H_{16}NO_2$ (M^++H); IR (KBr): 3371, 3178, 1648, 1626, 1626, 1261, 646, 556 cm^{-1}.



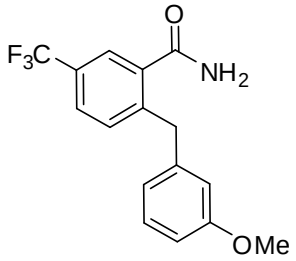
2-(3-Methoxybenzyl)-5-methylbenzamide (4): white solid; ^{1}H NMR: δ 7.32 (s, 1H), 7.18 - 7.21 (m, 2H), 7.11 - 7.13 (m, 1H), 6.74 - 6.80 (m, 3H), 5.65 (br. s., 2H), 4.18 (s, 2H), 3.77 (s, 3H), 2.35 (s, 3H); ^{13}C NMR: δ 171.8, 159.7, 142.6, 136.1, 135.4, 135.1, 131.2, 131.1, 129.4, 128.1, 121.2, 114.6, 111.3, 55.1, 38.4, 20.8; HRMS obsd 255.1257, calcd 255.1259 for $C_{16}H_{17}NO_2$(M^++H); IR (KBr): 3370, 3182, 1654, 1324, 1260, 996, 695, 548 cm^{-1}.



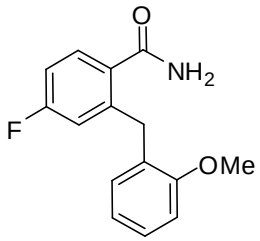
5-Methoxy-2-(3-methoxybenzyl)benzamide (5): white solid; mp: 79 ^{0}C; ^{1}H NMR: δ 7.17 - 7.22 (m, 1H), 7.16 (d, *J*=8.5 Hz, 1H), 7.05 (d, *J*= 3.0 Hz, 1H), 6.93 (dd, *J*=8.4, 2.9 Hz, 1H), 6.68 - 6.79 (m, 3H), 5.58 - 5.83 (m, 2H), 4.15 (s, 2H), 3.82 (s, 3H), 3.77 (s, 3H); ^{13}C NMR: δ 171.5, 159.5, 157.9, 142.0, 136.5,132.3, 130.2, 129.1, 121.1, 116.1, 114.6, 112.8, 111.3, 55.4, 55.1, 38.0; HRMS obsd 295.1140, calcd 295.1140 for $C_{16}H_{17}NNaO_3$ (M^++Na); IR (KBr): 3369, 3184, 2834, 1648, 1334, 1158, 1039, 920, 753, 655, 556 cm^{-1}.



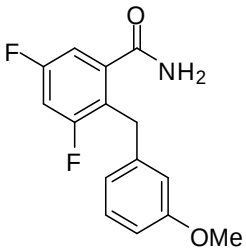
2-(3-Methoxybenzyl)-5-(trifluoromethyl)benzamide (6): pale yellow solid; mp: 133 ^{0}C; ^{1}H NMR: δ 7.75 (s, 1H), 7.63 (d, *J*=8.0 Hz, 1H), 7.38 (d, *J*=7.8 Hz, 1H), 7.23 (t, *J*=7.8 Hz, 1H), 6.73 - 6.83 (m, 3H), 5.81 (br. s, 1H),5.73 (br. s, 1H), 4.27 (s, 2H), 3.79 (s, 3H); ^{13}C NMR: δ 170.3, 159.8, 142.9, 142.9, 141.1, 135.9, 131.6, 129.7, 128.7, 127.1, 124.3, 121.3, 114.9, 111.6, 55.1, 38.6; HRMS obsd 309.0980, calcd 309.0977 for $C_{16}H_{14}F_3NO_2$ (M^+); IR (KBr): 3370, 3182, 1654, 1324, 1260, 996, 695, 548 cm^{-1}.



4-Fluoro-2-(3-methoxybenzyl)benzamide (7): white solid; mp: 87 ^{0}C; ^{1}H NMR: δ 7.50 (dd, *J*=8.3, 5.8 Hz, 1H), 7.22 (t, *J*=7.8 Hz, 1H), 6.89 - 6.99 (m, 2H), 6.74 - 6.78 (m, 3H), 5.93 (br. s, 1H), 5.73 (br. s, 1H), 4.22 (s, 2H), 3.78 (d, *J*=0.8 Hz, 3H); ^{13}C NMR: δ 171.0, 164.9, 162.4, 159.8, 142.3, 141.4, 131.4, 129.6, 121.3, 118.0, 115.0, 113.5, 111.6, 55.2, 38.7; HRMS obsd 260.1082, calcd 260.1087 for $C_{15}H_{15}FNO_2$ (M^++H); IR (KBr): 3367, 3184, 2834, 1647, 1584, 1401, 1261, 968, 694, 556 cm^{-1}.

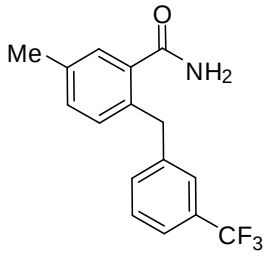


3,5-difluoro-2-(3-methoxybenzyl)benzamide (8): white solid; ^{1}H NMR: δ 7.16 (t, *J*= 8.0 Hz, 1H), 7.02 (d, *J*= 7.3 Hz, 1H), 6.93 - 6.88 (m, 1H), 6.76- 6.70 (3, 3H), 6.20 (br. S, 1H), 5.72 (br. S, 1H), 4.16 (s, 2H), 3.75 (s, 3H); ^{13}C NMR: δ 169.5, 162.4, 159.8, 141.0, 138.3, 129.6, 122.0, 120.6, 114.3, 111.4, 110.5, 105.8, 55.1, 30.8; HRMS obsd 300.0819 calcd 300.0812 for $C_{15}H_{13}F_2NNaO_2$ (M^++Na); IR (KBr): 3369, 3092, 2927, 1647, 1618, 1460, 1129, 879, 753, 609 cm^{-1}

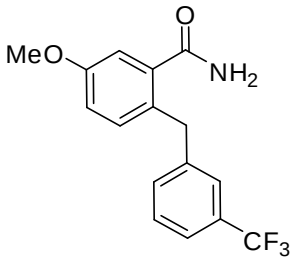


5-Methyl-2-(3-(trifluoromethyl)benzyl)benzamide (9): white solid; mp: 157 ^{0}C; ^{1}H NMR: δ7.42 - 7.44 (m, 2H), 7.34 - 7.38 (m, 2H), 7.29 (s, 1H), 7.20 (d, *J*=8.0 Hz, 1H), 7.12 (d, *J*=7.8 Hz, 1H), 5.74 (br. s, 1H),

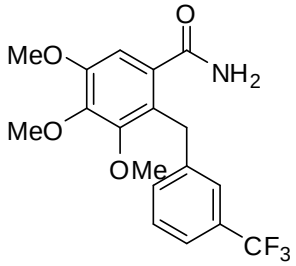
5.66 (br. s, 1H), 4.27 (s, 2H), 2.36 (s, 3H); ^{13}C NMR: δ 171.8, 142.0, 136.5, 135.5, 134.9, 132.4, 131.4, 131.1, 128.8, 127.9, 125.6, 125.5, 123.0, 122.9, 38.1, 20.9; HRMS: obsd 294.1107, calcd 294.1106 for $C_{16}H_{15}F_3NO$ (M^++ H); IR (KBr): 3372, 3187, 2919, 1647, 1334, 1111, 872, 656, 584 cm^{-1}.



5-Methoxy-2-(3-(trifluoromethyl)benzyl)benzamide (10): white solid; mp:135^{0}C; ^{1}H NMR: δ 7.45 (s, 2H), 7.37 - 7.39 (m, 2H), 7.14 (d, *J*=8.5 Hz, 1H), 7.03 (d, *J*=2.5 Hz, 1H), 6.94 (dd, *J*=8.5, 2.5 Hz, 1H), 5.76 (s, 1H), 5.67 (s, 1H), 4.24 (s, 2H), 3.81 (s, 3H); ^{13}C NMR: δ 171.3, 158.0, 142.2, 136.0, 132.3, 130.2, 128.8, 125.4, 122.9, 116.0, 113.0, 55.5, 37.7; HRMS obsd 333.0912, calcd 333.0908 for $C_{16}H_{14}F_3NNaO_2$ (M^++Na); IR (KBr): 3374, 3188, 1646, 1336, 1115, 701, 661, 537 cm^{-1}.

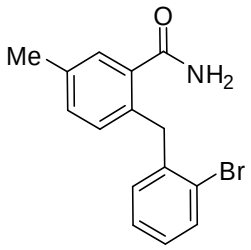


3,4,5-trimethoxy-2-(3-(trifluoromethyl)benzyl)benzamide (11): white solid; ^{1}H NMR: δ 7.49 (s, 1H), 7.40-7.34 (m, 3H), 6.84 (s, 1H), 5.5 (br.s, 2H), 4.23 (s, 2H), 3.89 (s, 6H), 3.70 (s, 3H); ^{13}C NMR: δ 171.2, 152.6, 152.2, 144.1, 142.4, 131.9, 131.1, 128.6, 125.3, 125.2, 124.6, 122.6, 106.5, 60.8, 56.1, 32.1; HRMS obsd 392.1086 calcd 392.1086 for $C_{18}H_{18}F_3NNaO_4$ (M^++Na); IR (KBr): 3371, 3186, 1647, 1340, 1112, 703, 667, 541 cm^{-1}.

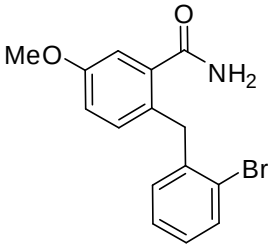


2-(2-Bromobenzyl)-5-methylbenzamide(12): white solid; mp:141^{0}C; ^{1}H NMR: δ 7.58 (d, *J*=8.3 Hz, 1H), 7.35 (s, 1 H), 7.21 – 7.25 (m, 1H), 7.18 (d, *J*=1H), 7.07 - 7.12 (m, 2H), 6.98 (d, *J*=7.8 Hz, 1H), 5.66 (br. s, 1H), 5.71 (br. s, 1H), 4.31 (s, 2H), 2.36 (s, 3H); ^{13}C NMR: δ 171.8, 140.2, 136.2, 135.3, 134.5, 132.8,

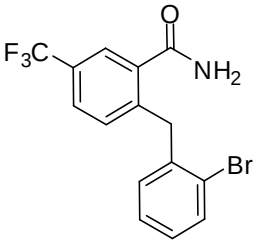
131.3, 130.6, 128.9, 128.5, 127.9, 127.5, 125.0, 38.6, 20.9; HRMS obsd 303.0256, calcd 303.0259 for $C_{15}H_{14}BrNO$ (M^+); IR (KBr): 3372, 3178, 1647, 1604, 1598, 742, 654, 608 cm^{-1}.



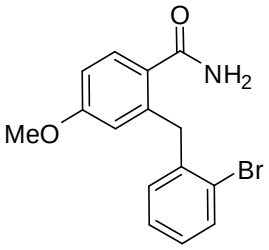
2-(2-Bromobenzyl)-5-methoxybenzamide (13): white solid; mp: 152 ^{0}C; ^{1}H NMR: δ 6.56 (d, *J*=7.7 Hz, 1H), 7.20 - 7.24 (m, 1H), 7.05 - 7.10 (m, 3H), 7.01 (d, *J*=8.5 Hz, 1H), 6.91 (dd, *J*=8.5, 2.8 Hz, 1H) , 5.83 (br. s, 1H), 5.74 (br. s, 1H), 4.26 (s, 2H), 3.81 (s, 3H); ^{13}C NMR: δ 171.4, 157.9, 140.3, 136.4, 132.8, 131.9, 130.9, 129.2, 127.9, 127.5, 124.9, 116.1, 112.8, 55.4, 38.3; HRMS obsd 343.0109, calcd 342.0106 for $C_{15}H_{14}BrNNaO_2$ (M^++Na); IR (KBr): 3375, 3186, 1646, 1504, 1241, 1030, 745, 544 cm^{-1}.



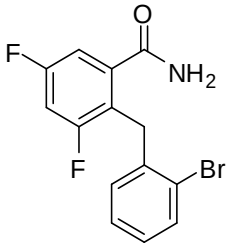
2-(2-Bromobenzyl)-5-(trifluoromethyl)benzamide (14): white solid; mp:140 ^{0}C; ^{1}H NMR: δ 7.77 (s, 1H), 7.60 (d, *J*=8.0 Hz, 2H), 7.25 - 7.29 (m, 1H), 7.13 - 7.20 (m, 3H), 5.93 (br. s, 1H), 5.83(br. s, 1H), 4.41 (s, 2H); ^{13}C NMR: δ170.2, 138.9, 135.9, 133.1, 131.3, 130.9, 129.0, 128.4, 127.7, 127.1, 127.0, 125.0, 124.1, 124.0, 38.9; HRMS obsd 356.9971, calcd 356.9976 for $C_{15}H_{11}BrF_3NO$ (M^+); IR (KBr): 3379, 3186, 1648, 1610, 1323, 1123, 712, 656 cm^{-1}.



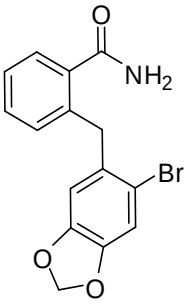
2-(2-Bromobenzyl)-4-methoxybenzamide (15): white solid; mp: 155 ^{0}C;^{1}H NMR: δ 7.58 (dd, *J*=8.3, 1.3 Hz, 1H), 7.53 (d, *J*=8.5 Hz, 1H), 7.22 - 7.26 (m, 1H), 7.09 - 7.12 (m, 2H), 6.80 (dd, *J*=8.5, 2.5 Hz, 1H), 6.61 (d, *J*=2.5 Hz, 1H), 5.66 (br. s, 2H), 4.39 (s, 2H), 3.77 (s, 3H); ^{13}C NMR: δ 171.4, 161.2, 140.4, 140.0, 132.8, 131.1, 129.3, 128.0, 128.5, 127.6, 125.0, 116.5, 111.3, 55.3, 39.3; HRMS obsd 342.0107, calcd 342.0106 for $C_{15}H_{14}BrNNaO_2$ (M^++Na); IR (KBr): 3184, 1641, 1541, 1275, 1260, 764, 749 cm^{-1}.



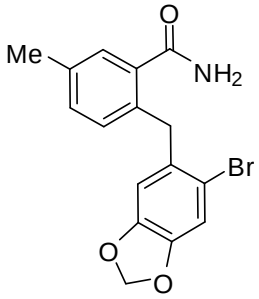
2-(2-bromobenzyl)-3,5-difluorobenzamide (16): white solid; ^{1}H NMR: δ 7.55 (d, *J*= 7.3 Hz, 1H), 7.15 (t, *J*= 7.6 Hz, 1H), 7.10- 7.03 (m, 2H), 6.97-6.92 (m, 1H), 6.79 (d, *J*= 8 Hz, 1H), 6.16 (br.s, 1H), 5.66 (br.s, 1H), 4.24 (s, 2H); ^{13}C NMR: δ 169.1, 162.7, 160.2, 138.5, 132.8, 129, 128.0, 127.5, 124.5, 120.9, 110.9, 105.9, 53.4, 31.8; HRMS obsd 347.9812 calcd 347.9812 for $C_{14}H_{10}BrF_2NaO$ (M^++Na); IR (KBr): 3371, 3082, 2922, 1650, 1611, 1473, 1119, 859, 680, 607 cm^{-1}.



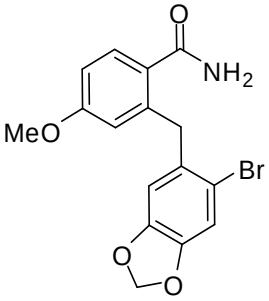
2-(6-Bromobenzo[*d*][1,3]dioxol-5-yl)methyl)benzamide (17): white solid; mp: 182 ^{0}C; ^{1}H NMR: δ 7.52 (dd, *J*=7.7, 1.4 Hz, 1H), 7.37 (td, *J*=7.5, 1.5 Hz, 1H), 7.28 - 7.32 (m, 1H), 7.11 (d, *J*=7.0 Hz, 1H), 7.04 (s, 1H), 6.62 (s, 1H), 5.95 (s, 2H), 5.80 (br. s, 2H), 4.27(s, 2H); ^{13}C NMR:δ 171.6, 147.5, 146.9, 138.0, 135.2, 133.0, 130.6, 130.5, 127.2, 126.5, 115.0, 112.7, 110.8, 101.6, 38.6; HRMS obsd 355.9898, calcd 355.9898 for $C_{15}H_{12}BrNNaO_3$ (M^++Na); IR (KBr): 3360, 3190, 1645, 1617, 1468, 1261, 935, 764, 642, 595 cm^{-1}.



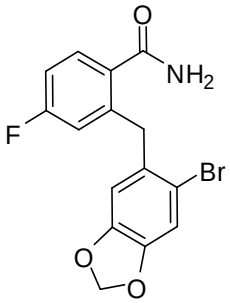
2-(6-Bromobenzo[*d*][1,3]dioxol-5-yl)methyl)-5-methylbenzamide (18): white solid; mp: 208 ^{0}C; ^{1}H NMR: δ 7.34 (s, 1H), 7.18 (d, *J*=7.5 Hz, 1H), 7.04 (s, 1H), 7.00 (d, *J*=8.0 Hz, 1H), 6.60 (s,1H), 5.94 (s, 2H), 5.72 (br. s, 1H), 5.72 (br. s, 1H**)**,5.67 (br. s, 1H), 4.21 (s, 2H), 2.36 (s, 3H); ^{13}C NMR (DMSO-d_6, 400 MHz): δ 171.6, 147.6, 147.1, 137.1, 136.3, 134.6, 133.7, 130.5, 129.8, 128.3, 114.5, 112.6, 111.1, 102.2, 37.8, 20.9; HRMS obsd 370.0057, calcd 370.0055 for $C_{16}H_{14}BrNNaO_3$ (M^++Na); IR (KBr): 3370, 3186, 2921, 2362, 1648, 1478, 1245, 935, 680, 585 cm^{-1}.



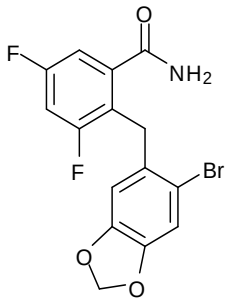
2-(6-Bromobenzo[*d*][1,3]dioxol-5-yl)methyl)-4-methoxybenzamide (19): white solid; mp: 186 ^{0}C; ^{1}H NMR (DMSO-d_6, 400 MHz): δ 7.83 (s, 1H), 7.40 (s, 1H), 7.19 (s, 1H), 6.98 (t, *J*=1.5 Hz, 1H), 6.92 (d, *J*=1.5 Hz, 2H), 6.67 (s, 1H), 6.02 (s, 2H), 4.06 (s, 2H), 3.76 (m, 3H); ^{13}C NMR (DMSO-d_6, 400 MHz): δ 171.1, 157.6, 147.6, 147.0, 138.1, 133.9, 131.2, 129.5, 115.6, 114.4, 113.3, 112.6, 111.0, 102.2, 55.6, 37.5; HRMS obsd 364.0177, calcd 364.0184 for $C_{16}H_{15}BrNO_4$ (M^++H); IR (KBr): 3361, 3180, 2925, 1652, 1517, 1485, 1244, 803, 702, 546 cm^{-1}.



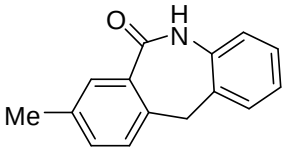
2-(6-Bromobenzo[*d*][1,3]dioxol-5-yl)methyl)-4-fluorobenzamide (20): white solid; mp: 195 ^{0}C; ^{1}H NMR: δ 7.51 (s, 1H), 6.96 - 7.03 (m, 2H), 6.75 - 6.65 (m, 2H), 5.96 (s, 2H), 5.72 (br. s, 2H), 4.26 (s, 2H); ^{13}C NMR: δ 170.6, 162.5, 147.6, 147.2, 141.8, 132.3, 131.1, 129.3, 129.2, 117.2, 115.1, 113.5, 112.8, 110.8, 101.7, 38.6; HRMS obsd 373.9800, calcd 373.9804 for $C_{15}H_{11}BrFNNaO_3$ (M^++Na); IR (KBr): 3364, 3184, 1645, 1617, 1479, 1275, 920, 650, 588 cm^{-1}.



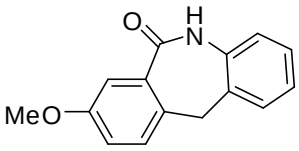
2-((6-bromobenzo[d][1,3]dioxol-5-yl)methyl)-3,5-difluorobenzamide (21): white solid; ^{1}H NMR: δ 7.10 (d, *J*= 7.2 Hz, 1H), 7.02 (s, 1H), 6.95 (t, *J*= 8.0 Hz, 1H), 6.33 (s, 1H), 5.91 (br. s, 3H), 5.65 (br. s, 1H), 4.15 (s, 2H); ^{13}C NMR: δ 168.7, 147.5, 146.8, 138.7, 131.5, 114.5, 112.7, 110.9, 110.6, 109, 106.2, 106, 105.7, 101.6, 31.6, 31.5; HRMS obsd 391.9711 calcd 391.9710 for $C_{15}H_{10}BrF_2NNaO_3$ (M^++Na); IR (KBr): 3372, 3185, 1647, 1615, 1460, 1285, 921, 657, 589 cm^{-1}.



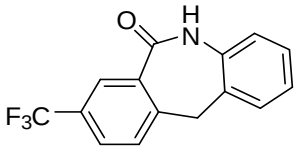
8-Methyl-5,11-Dihydrodibenzo[*b,e*]azepin-6-one (22): white solid; mp:150 ^{0}C; ^{1}H NMR: δ 8.51 (br. s, 1H), 7.75 (s, 1H), 7.25 - 7.29 (m, 2H), 7.21 (td, *J*=7.6, 1.6 Hz, 1H), 7.17 (d, *J*=7.8 Hz, 1H),7.09 - 7.14 (m, 1H), 7.07 (dd, *J*=7.8, 1.3 Hz, 1H), 3.93 (s, 2H), 2.35 (s, 3H); ^{13}C NMR δ: 169.7, 138.6, 136.8, 136.1, 133.4, 133.3, 131.1, 128.2, 127.4, 127.2, 125.4, 120.8, 38.8, 20.9; HRMS obsd 224.1073, calcd 224.1075 for $C_{15}H_{14}NO$ (M^{+}+H); IR (KBr): 3435, 3173, 1671, 1363, 1139, 750, 561, 512, 488 cm^{-1}.



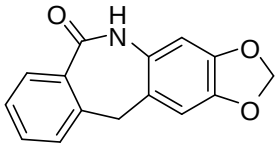
8-Methoxy-5,11-Dihydrodibenzo[*b,e*]azepin-6-one (23): pale yellow solid; mp:147 ^{0}C; ^{1}H NMR: δ 8.6 (s, 1H), 7.46 (d, *J*= 2.8 Hz, 1H), 7.27 – 7.28 (m, 2H), 7.22 (dd, *J*=7.56, 1.4 Hz, 1H), 7.18 (d, *J*=8 Hz, 1H), 7.12 (td, *J*=7.4, 1.08 Hz, 1H), 7.08 (d, *J*=7.8 Hz, 1H), 7.00 (dd, *J*= 8.3, 2.8 Hz, 1H), 3.90 (s, 2H), 3.82 (s, 3H); ^{13}C NMR: δ 169.5, 158.6, 135.9, 133.9, 133.6, 132.2, 128.5, 128.1, 127.4, 125.5, 120.8, 119.6, 114.3, 55.5, 38.3; HRMS obsd 240.1017, calcd 240.1025 for $C_{15}H_{14}NO_2$ (M^{+}+H); IR (KBr): 3172, 3037, 2918, 1647, 1492, 1236, 1035, 846, 650, 539 cm^{-1}.



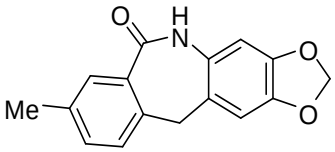
8-(Trifluoromethyl)-5,11-Dihydrodibenzo[*b,e*]azepin-6-one (24): off-whitesolid; ^{1}H NMR: δ 8.38 (br. s, 1H). 8.21 (s, 1H), 7.69 - 7.71 (m, 1H), 7.43 (d, *J*=8.0 Hz, 1 H), 7.31 (d, *J*=7.5 Hz, 1H), 7.25 (dd, *J*=7.7, 1.4 Hz, 1H), 7.14 - 7.18 (m, 1H), 7.09 (d, *J*=7.8 Hz, 1H), 4.03 (s, 2H); ^{13}C NMR (DMSO-d_6, 400 MHz): δ 169.0, 145.0, 135.9, 132.3, 132.0, 129.9. 129.5, 129.0, 128.3, 128.0, 127.8, 125.8, 121.2, 39.0; HRMS obsd 300.0609, calcd 300.0612 for $C_{15}H_{10}F_3NNaO$ (M^{+}+Na).



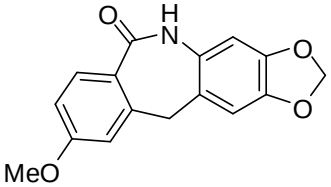
5,11-Dihydrodibenzo[2, 3-*d*]1,3-dioxole[*b,e*]azepin-6-one (25): off white solid; ^{1}H NMR: δ 8.14 (br. s, 1H), 7.91 (d, *J*=7.7, 1H), 7.45 (td, *J*= 7.5, 1.28 Hz, 1H), 7.33 (t, *J*=7.5, 1H), 7.24 (d, *J*=7.5, 1H), 6.75 (s, 1H), 6.59 (s, 1H), 5.93 (s, 2H), 3.84 (s, 2H); ^{13}C NMR: δ 169.4, 146,8, 145.4, 141.9, 132.5, 131.3, 130.8, 127.1, 126.8, 107.9, 102.4, 101.5, 38.8; HRMS obsd 254.0810, calcd 254.0817for $C_{15}H_{12}NO_3$ (M^++H).



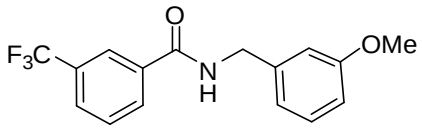
8-Methyl-5,11-Dihydrodibenzo[2, 3-*d*]1,3-dioxole[*b,e*]azepin-6-one (26): off white solid; ^{1}H NMR: δ 8.06 (br. s, 1H), 7.72 (s, 1H), 7.25 (s, 1H), 7.12 (d, *J*=7.72, 1H), 6.74 (s, 1H), 6.57 (s, 1H), 5.92 (s, 2H), 3.79 (s, 2H), 2.35 (s, 3H); ^{13}C NMR: δ 170.0, 146.8, 145.2, 139.2, 136.9, 133.6, 131.3, 129.9, 126.5, 108.0, 102.7, 101.5, 38.6, 20.5; HRMS obsd 267.0894, calcd 267.0895 for $C_{16}H_{14}NO_3$ (M^++H); IR (KBr): 3150, 1656, 1482, 1482, 1356, 1275, 750 cm^{-1}.



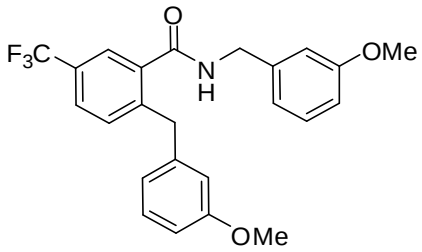
9-Methoxy-5,11-Dihydrodibenzo[2,3-*d*]1,3-dioxole[*b,e*]azepin-6-one (27): white solid; ^{1}H NMR: δ 8.21 (br. s, 1H), 7.41 (d, *J*=2.7 Hz, 1H), 7.13 (d, *J*=8.4 Hz, 1H), 6.98 (dd, *J*=8.3, 2.7 Hz, 1H), 6.72 (s, 1H), 6.57 (s, 1H), 5.91 (s, 2H), 3.81 (s, 3H), 3.76(s, 2H); ^{13}C NMR (DMSO-d_6, 400 MHz): δ 167.2, 161.9, 145.9, 144.0, 143.4, 131.8, 130.7, 125.7, 124.4, 112.2, 111.6, 107.7, 102.2, 101.1, 55.3, 37.4; HRMS obsd 306.0741, calcd 306.0742 for $C_{16}H_{13}NNaO_4$ (M^++Na).



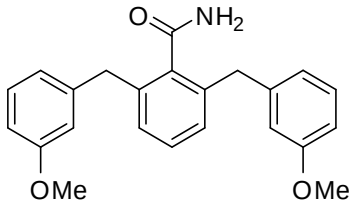
N-(3-methoxybenzyl)-3-(trifluoromethyl)benzamide (SC-1): white yellow solid; ^{1}H NMR: δ 8.06 (s, 1H), 7.99 (d, *J*=7.8 Hz, 1H), 7.78 (d, *J*=7.8 Hz, 1H), 7.59 (t, *J*=7.8 Hz, 1H), 7.29 - 7.33 (m, 1H), 6.96 (d, *J*=7.5 Hz, 1H), 6.92 (s, 1H), 6.87 (dd, *J*=8.3, 2.5 Hz, 1H), 6.42 (br. s, 1H), 4.66 (d, *J*=5.8 Hz, 2H), 3.83 (s, 3H).



N,2-bis(3-methoxybenzyl)-5-(trifluoromethyl)benzamide (SC-2): light yellow solid, ^{1}H NMR: δ 7.66 (s, 1H), 7.61 (d, *J*=8.3 Hz, 1H), 7.37 (d, *J*=8.0 Hz, 1H), 7.25 (t, *J*=8.2 Hz, 1H), 7.15 - 7.21 (m, 1H), 6.82 - 6.87 (m, 1H), 6.78 - 6.81 (m, 2H), 6.73 - 6.78 (m, 1H), 6.67 - 6.72 (m, 2H), 5.92 (br. s, 1H), 4.50 (d, *J*=5.8 Hz, 2H), 4.23 (s, 2H), 3.80 (s, 3H), 3.76 (s, 3H).



2,6-bis(3-methoxybenzyl)benzamide (3c): off white solid; ^{1}H NMR: δ 7.26-7.18 (m, 3H), 7.06-7.04 (d, *J*= 8 Hz, 2H), 6.79-6.74 (m, 6H), 5.84 (br. s, 1H), 5.39 (br. s, 1H), 4.064 (s, 4H), 3.77 (s, 6H).



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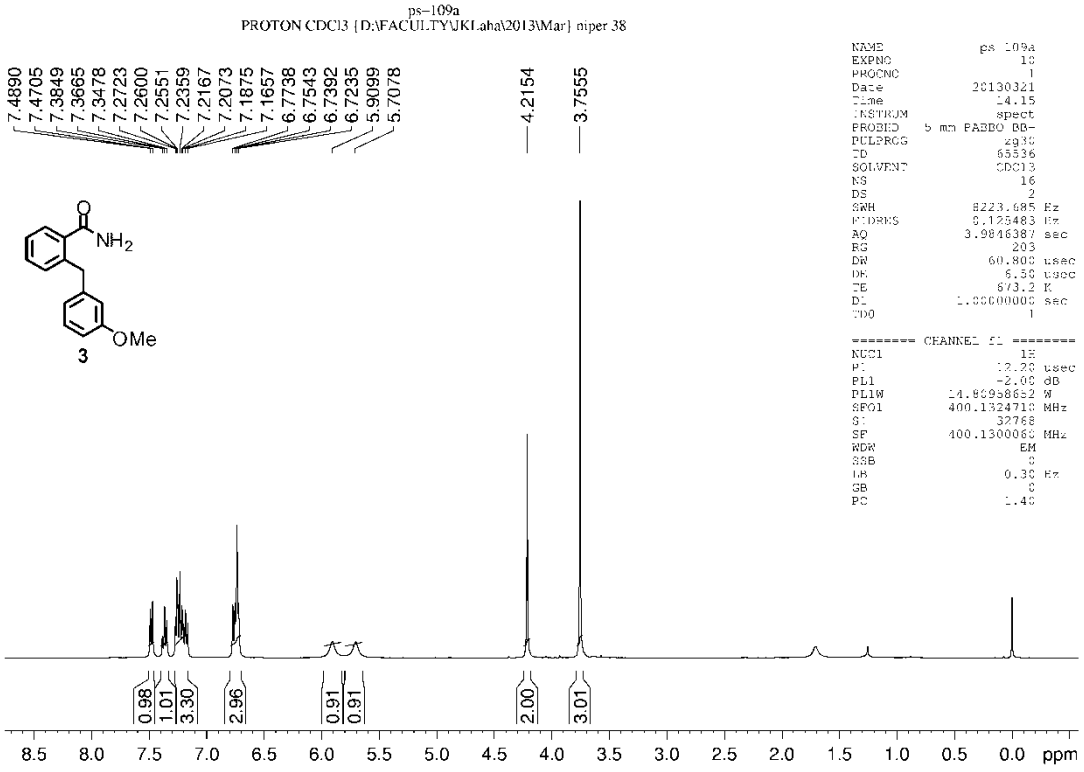

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RG 203
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DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
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PL1 -2.00 dB
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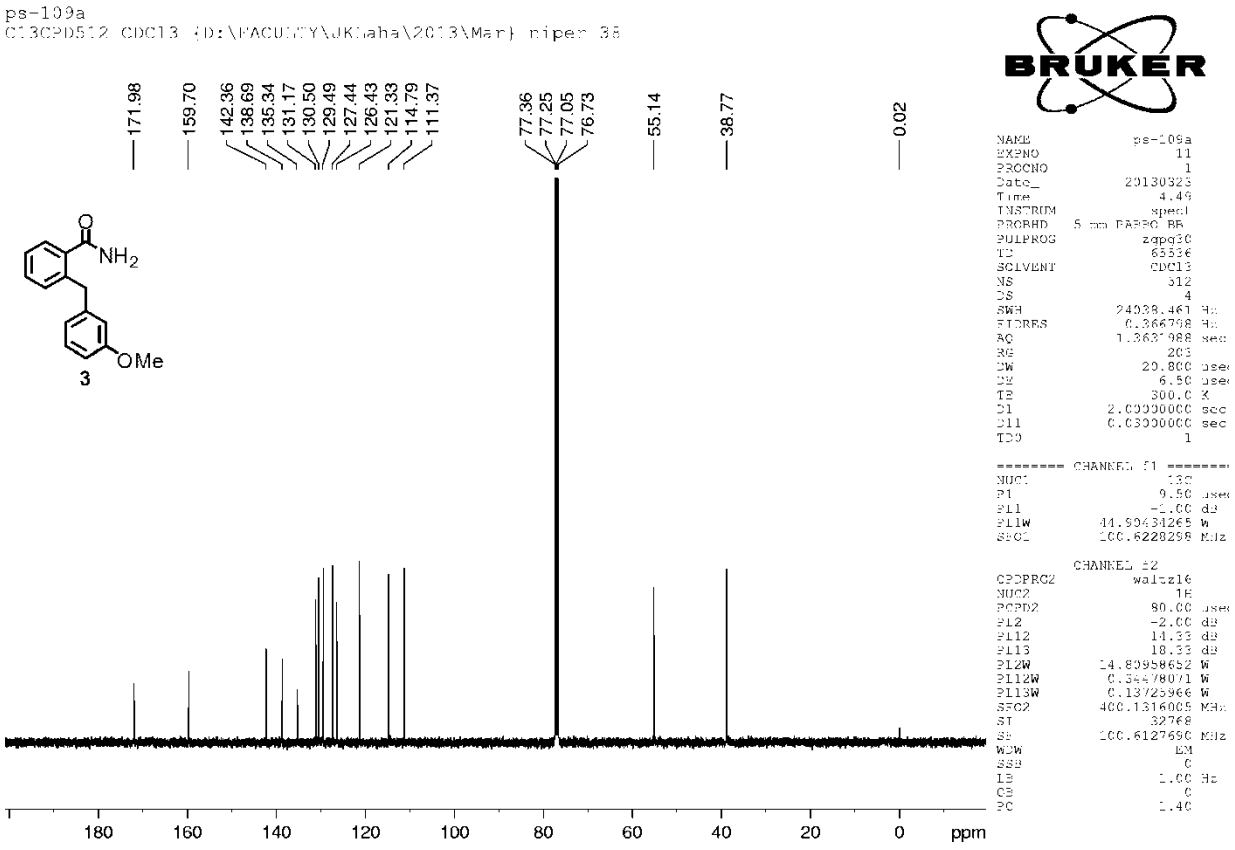

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RG 203
DW 20.800 usec
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TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
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PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
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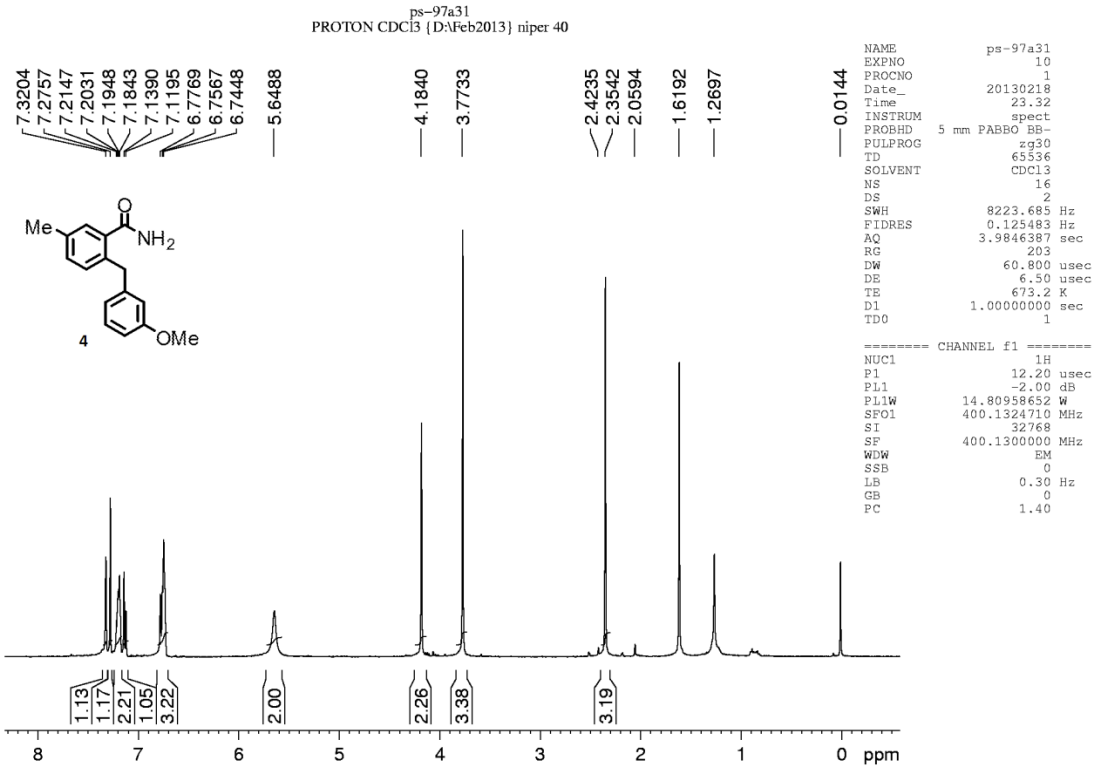
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TE 673.2 K
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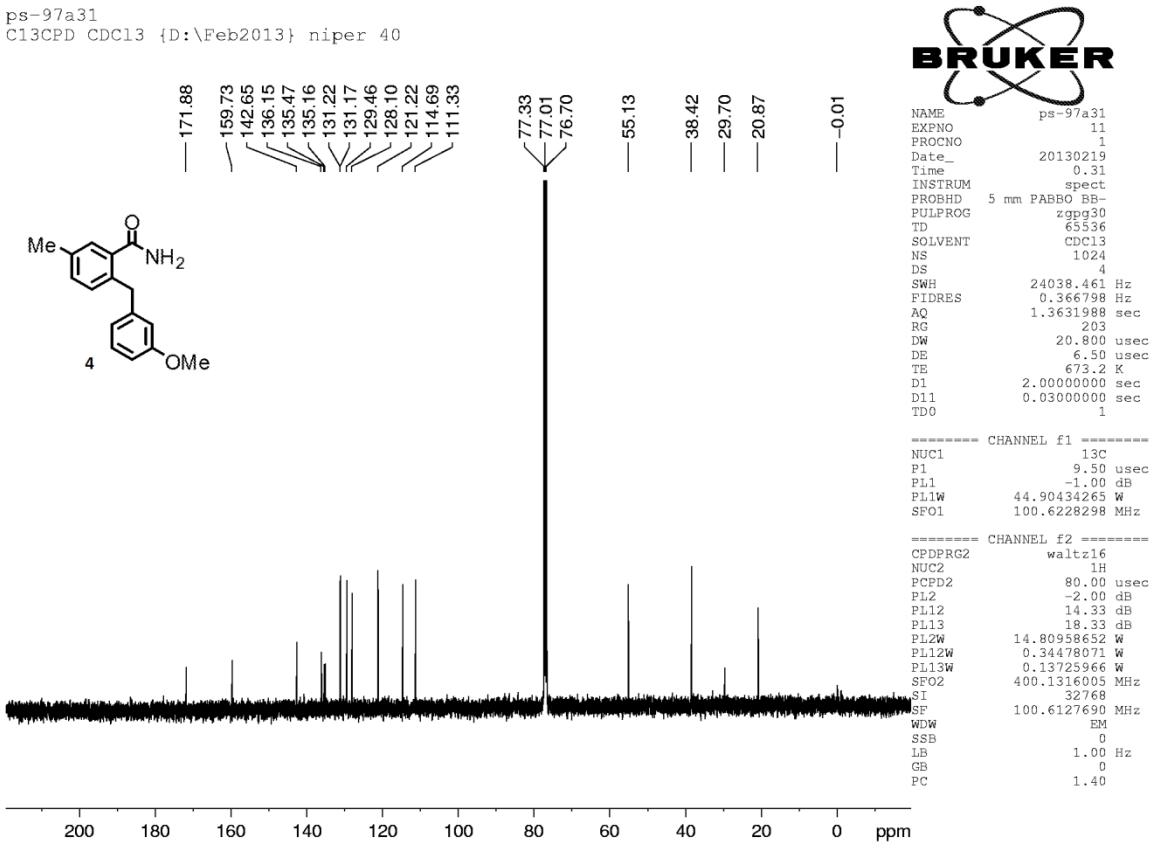
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29.70
20.87
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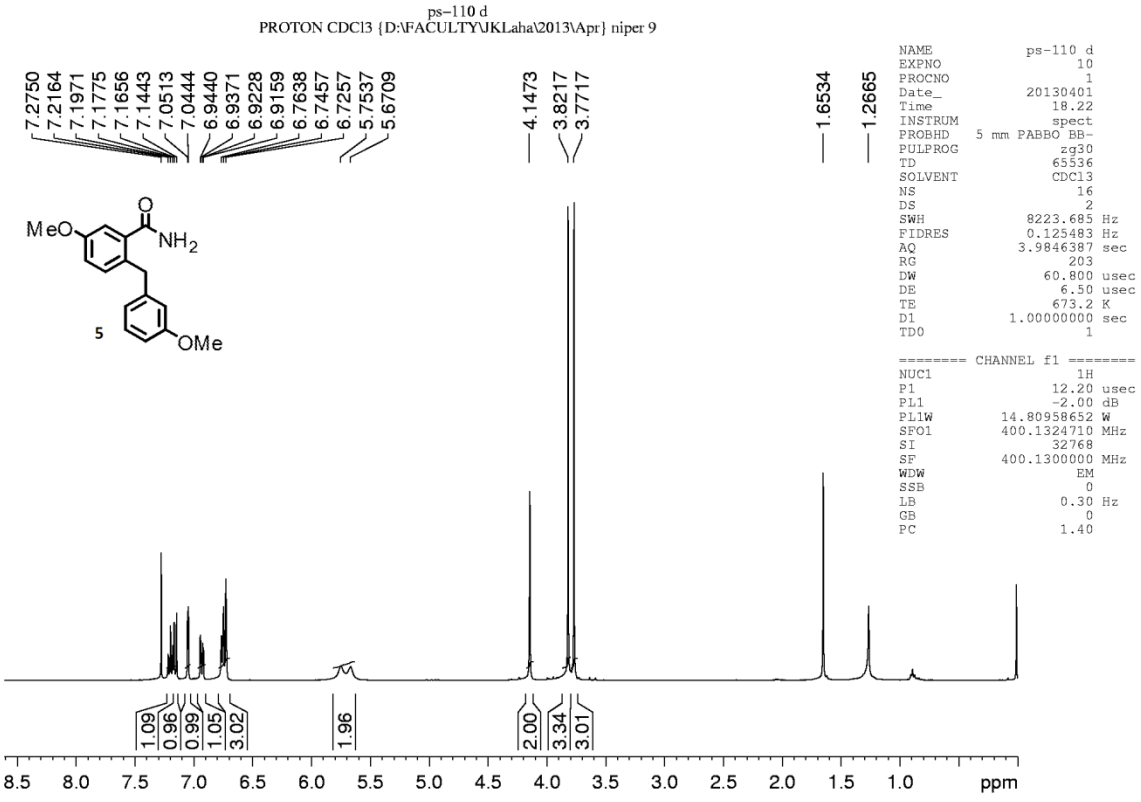
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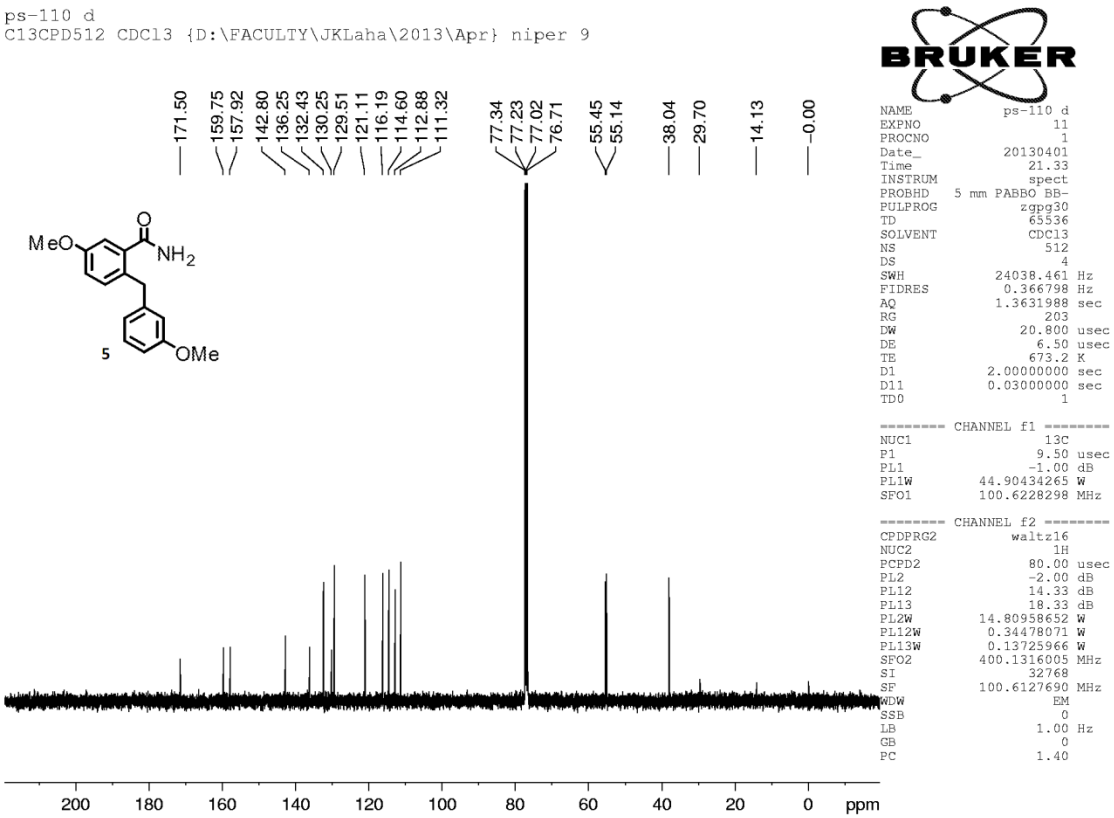
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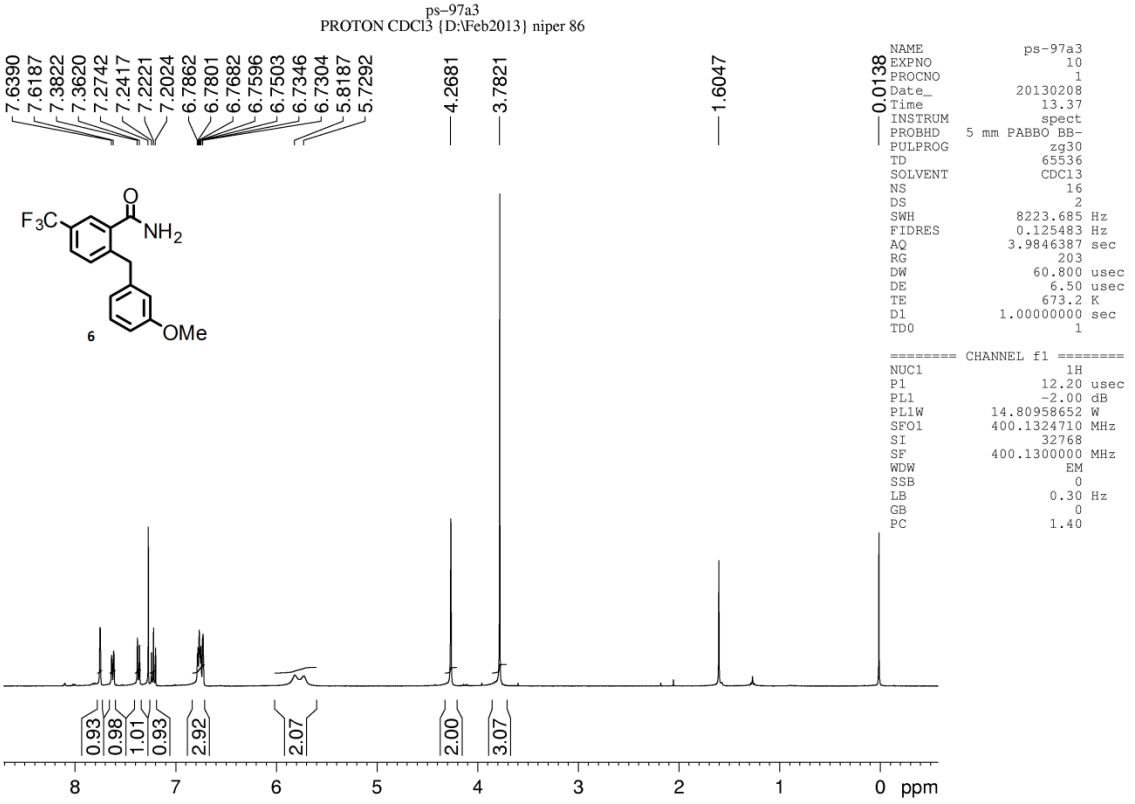
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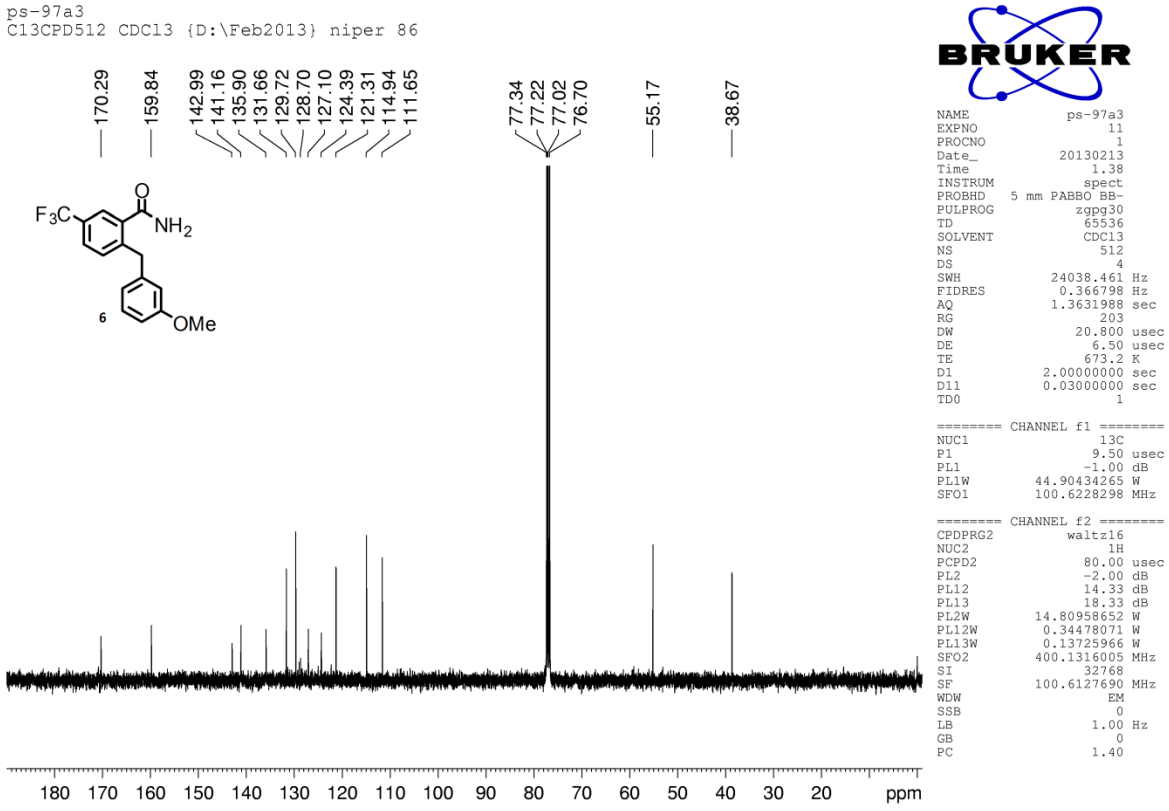
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PL13 18.33 dB
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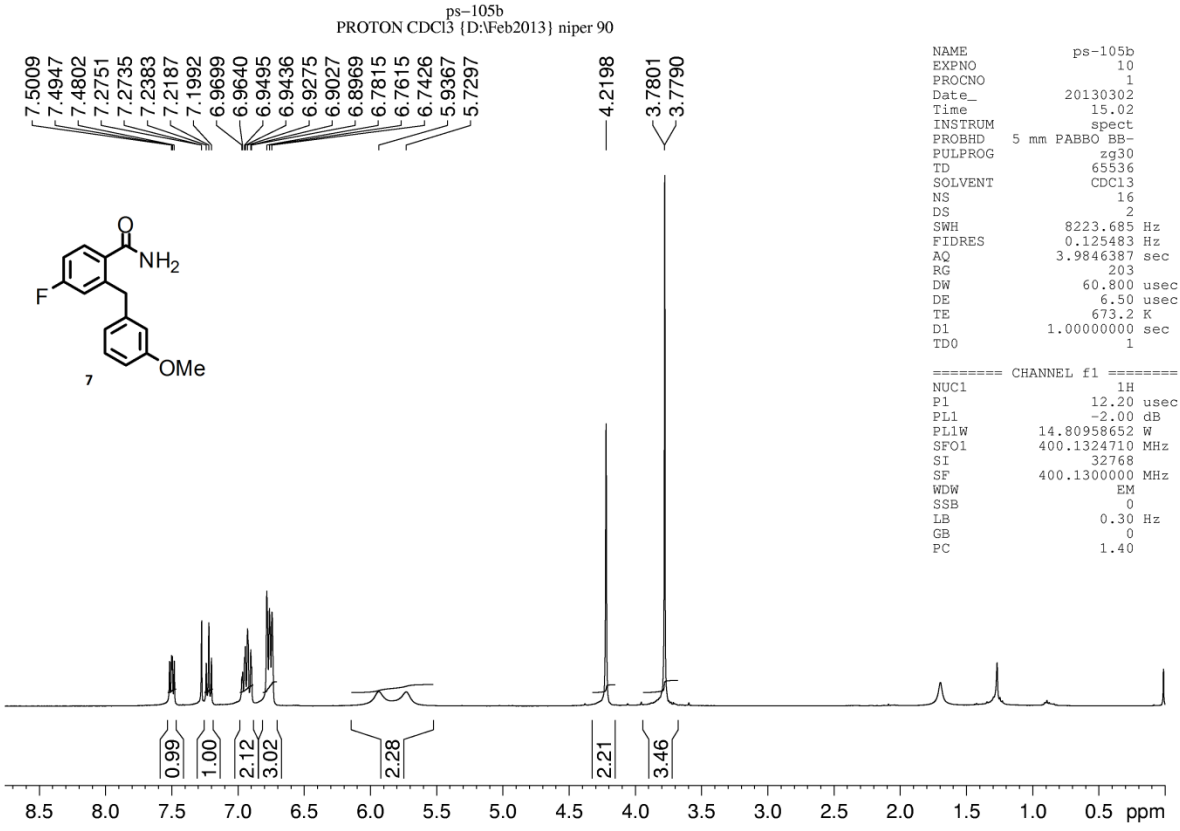


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PC                 1.40
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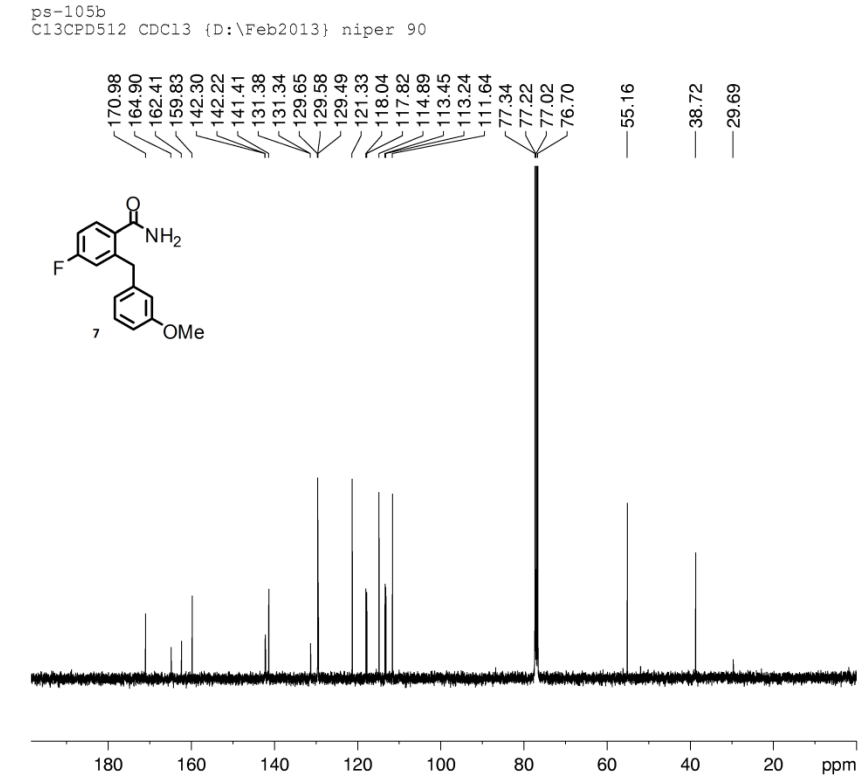
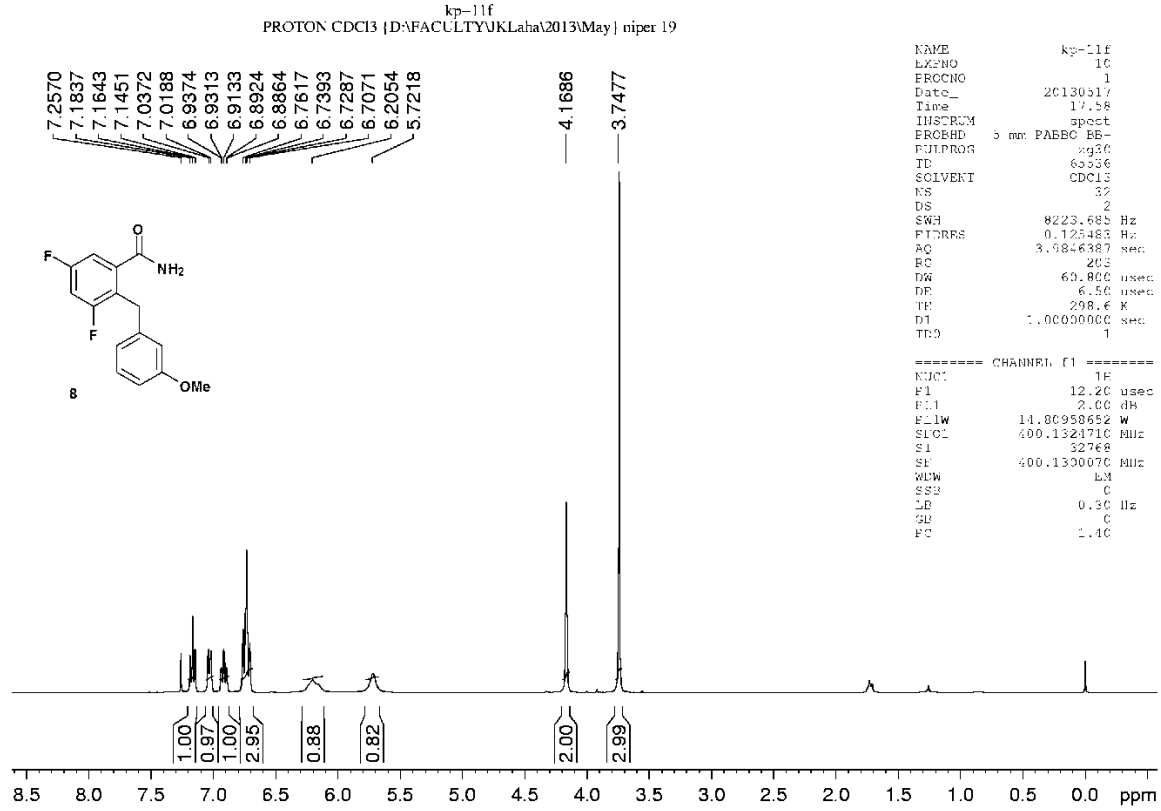
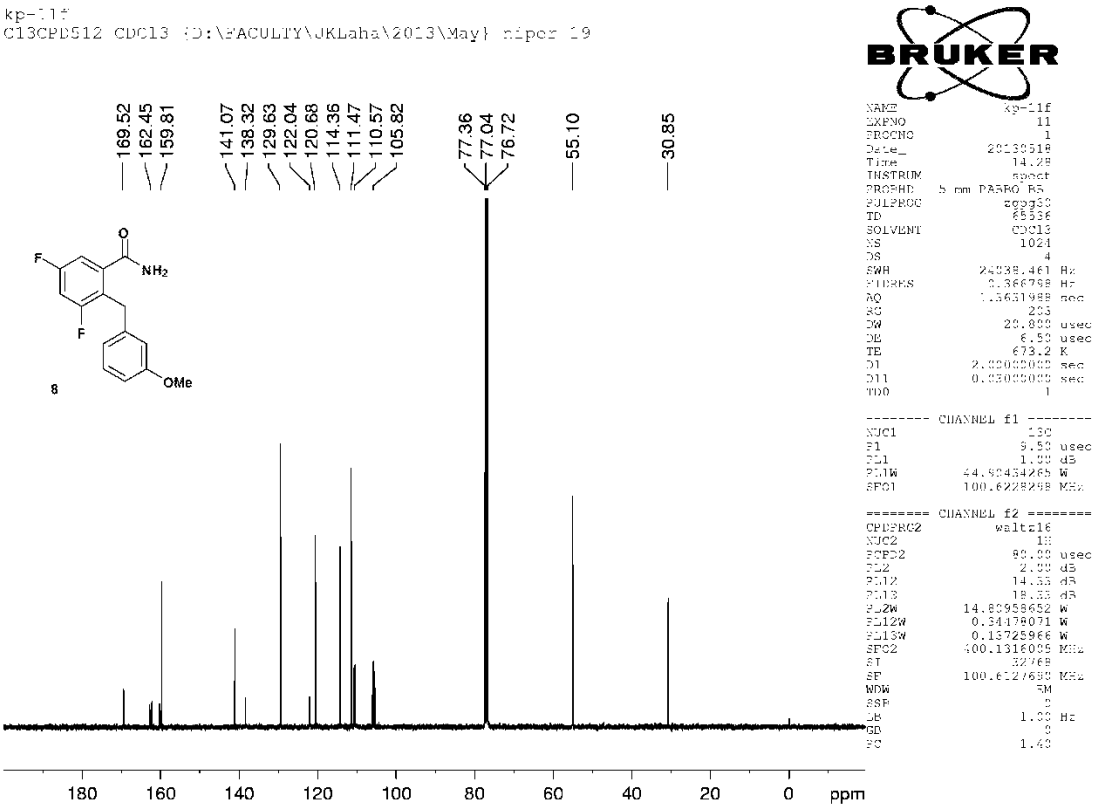
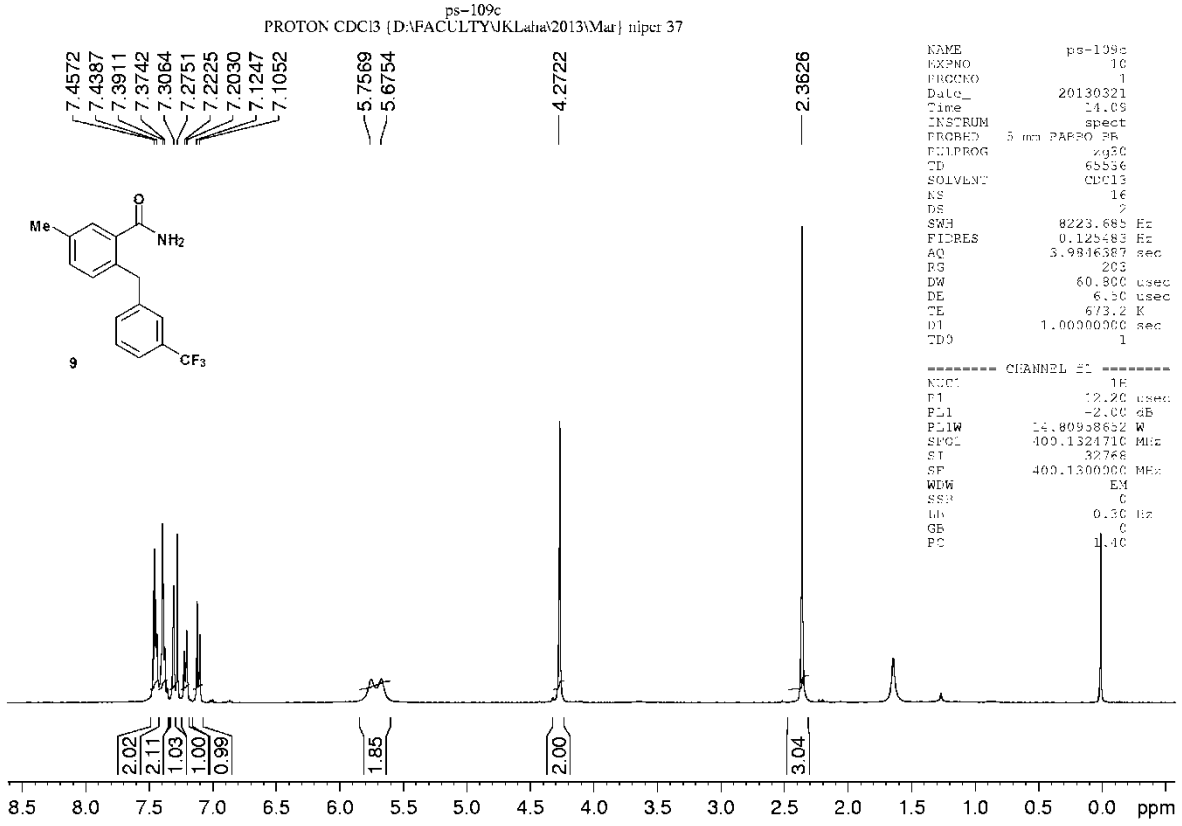


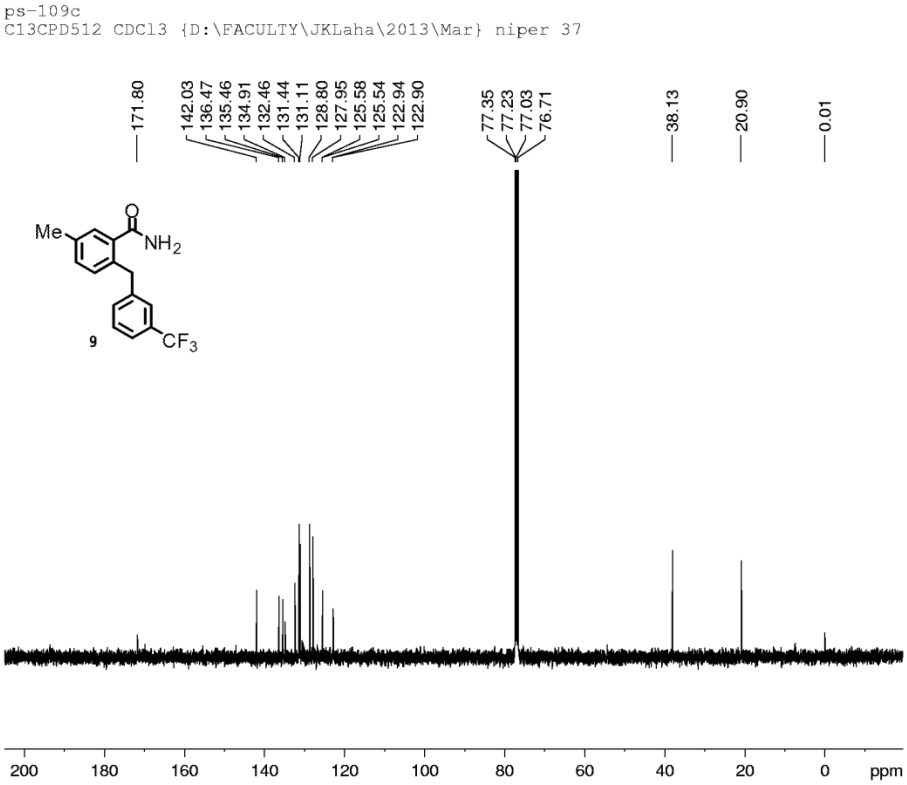

```
N.
EXPNO                  11
PROCNO                  1
Date_            20130302
Time                15.32
INSTRUM             spect
PROBHD   5 mm PABBO BB-
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                    203
DW                20.800 usec
DE                 6.50 usec
TE                673.2 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                 9.50 usec
PL1               -1.00 dB
PL1W         44.90434265 W
SFO1         100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2              80.00 usec
PL2                -2.00 dB
PL12               14.33 dB
PL13               18.33 dB
PL2W         14.80958652 W
PL12W         0.34478071 W
PL13W         0.13725966 W
SFO2         400.1316005 MHz
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                 1.40
```


kp-11f
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\May} niper 19
7.2570
7.1837
7.1643
7.1451
7.0372
7.0188
6.9374
6.9313
6.9133
6.8924
6.8864
6.7617
6.7393
6.7287
6.7071
6.2054
5.7218
4.1686
3.7477
F
O
NH2
F
OMe
8
1.00
0.97
1.00
2.95
0.88
0.82
2.00
2.99
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
NAME kp-11f
EXPNO 10
PROCNO 1
Date_ 20130517
Time 17.58
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

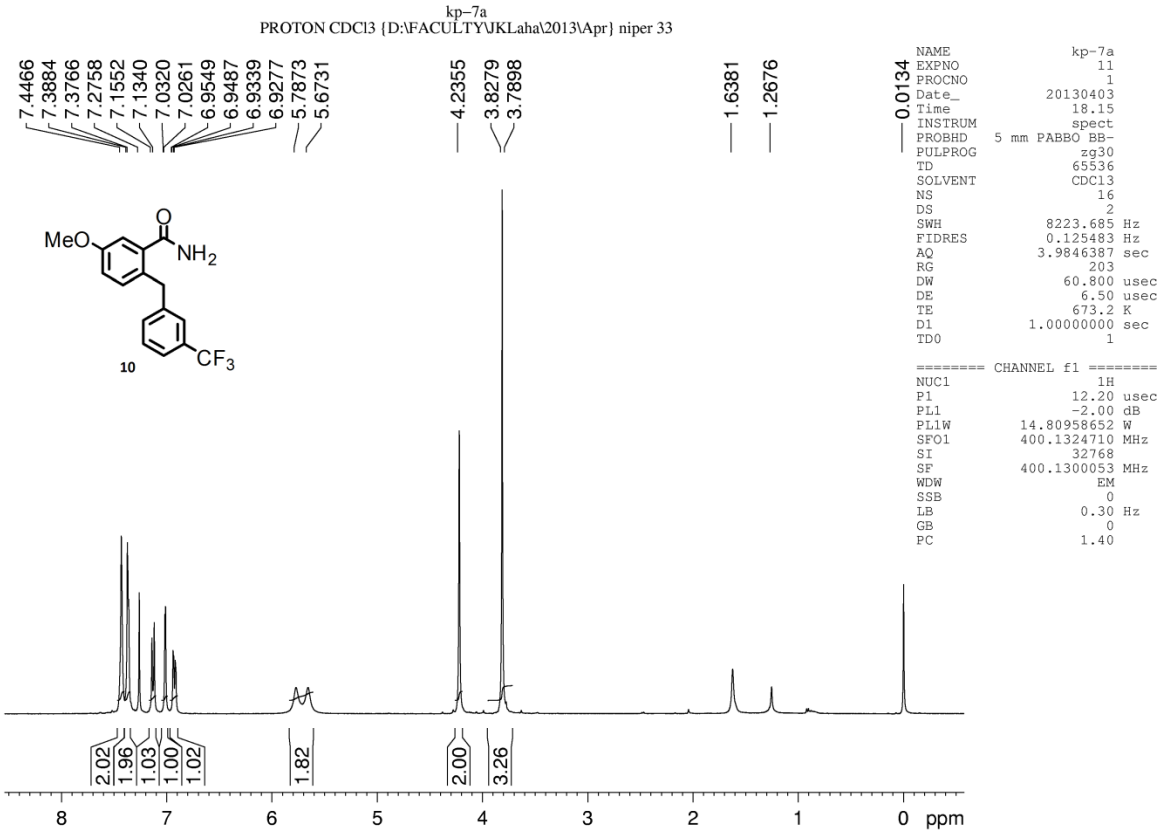

kp-11f
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\May} niper 19
169.52
162.45
159.81
141.07
138.32
129.63
122.04
120.68
114.36
111.47
110.57
105.82
77.36
77.04
76.72
55.10
30.85
F
O
NH2
F
OMe
8
180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
NAME kp-11f
EXPNO 11
PROCNO 1
Date_ 20130518
Time 14.28
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 9.50 usec
PL1 1.00 dB
PL1W 44.92434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 14.55 dB
PL13 18.55 dB
PL2W 14.80958652 W
PL12W 0.34178071 W
PL13W 0.15725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 2
LB 1.00 Hz
GB 0
PC 1.40

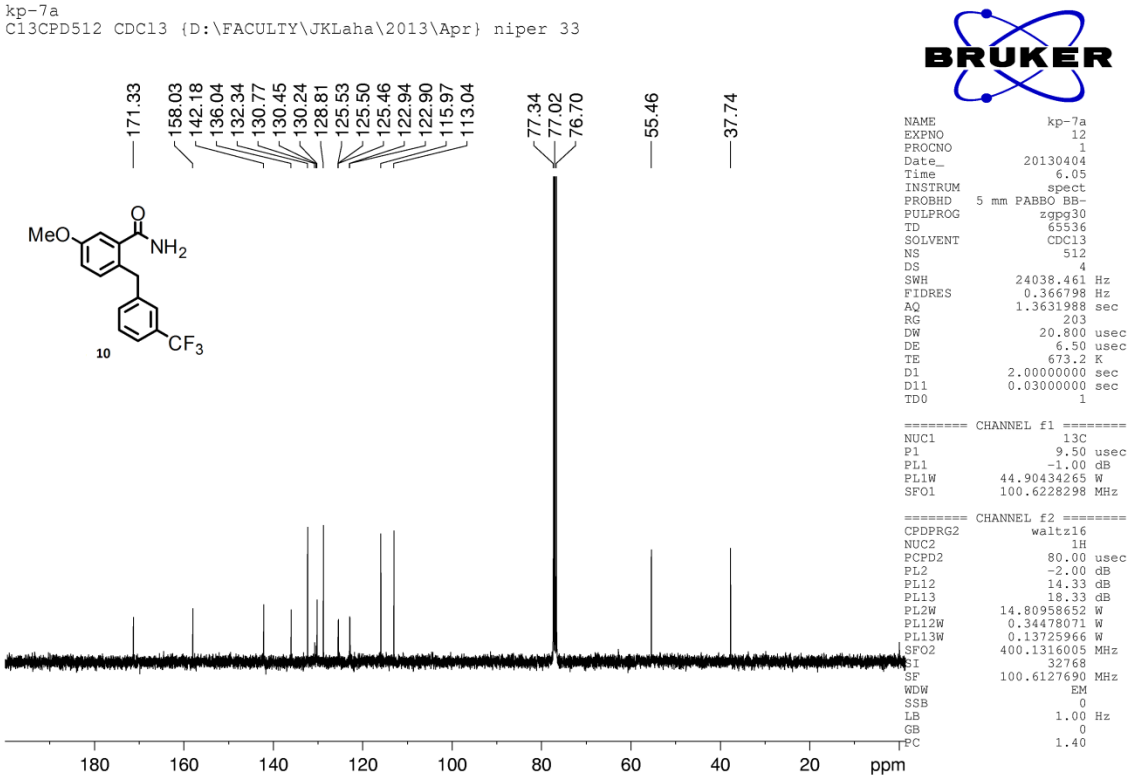


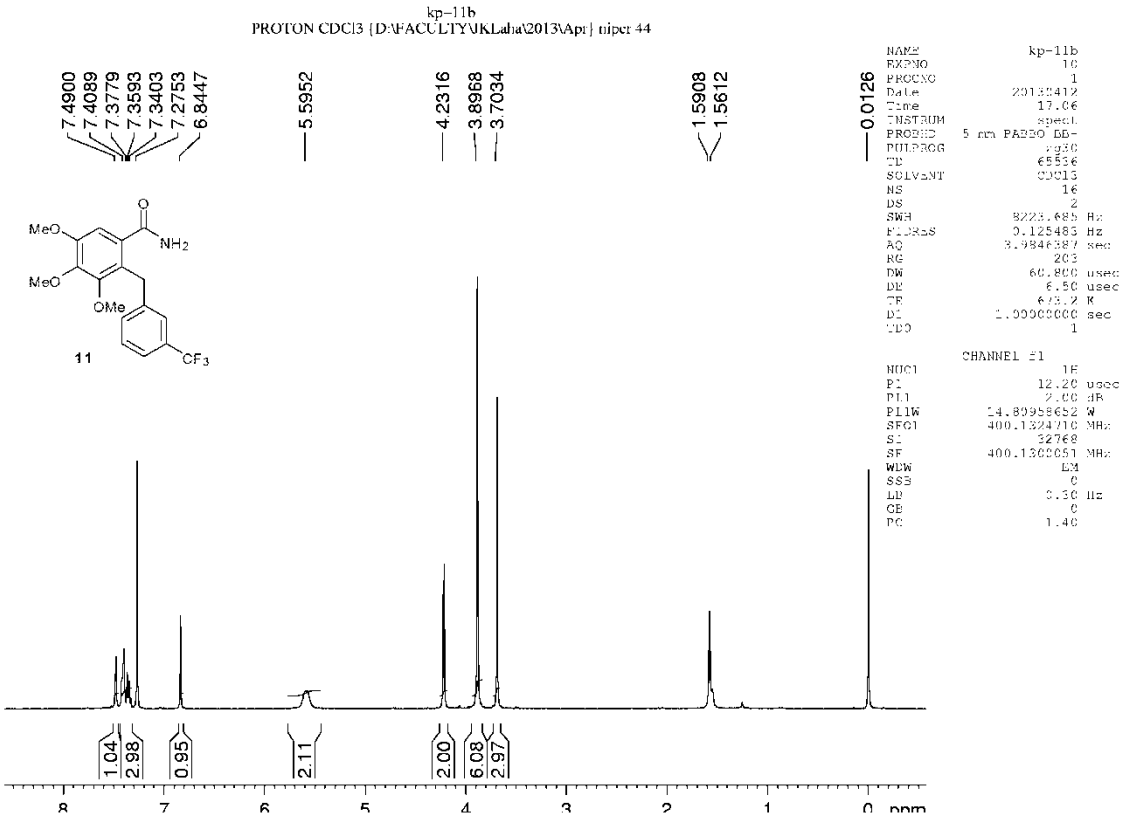

```
NAME                ps-109c
EXPNO                    10
PROCNO                    1
Date_              20130321
Time                  14.09
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       16
DS                        2
SWH               8223.685 Hz
FIDRES            0.125483 Hz
AQ                3.9846387 sec
RG                      203
DW                 60.800 usec
DE                  6.50 usec
TE                 673.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    12.20 usec
PL1                   -2.00 dB
PL1W            14.80958652 W
SFO1            400.1324710 MHz
SI                    32768
SF              400.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.40
```

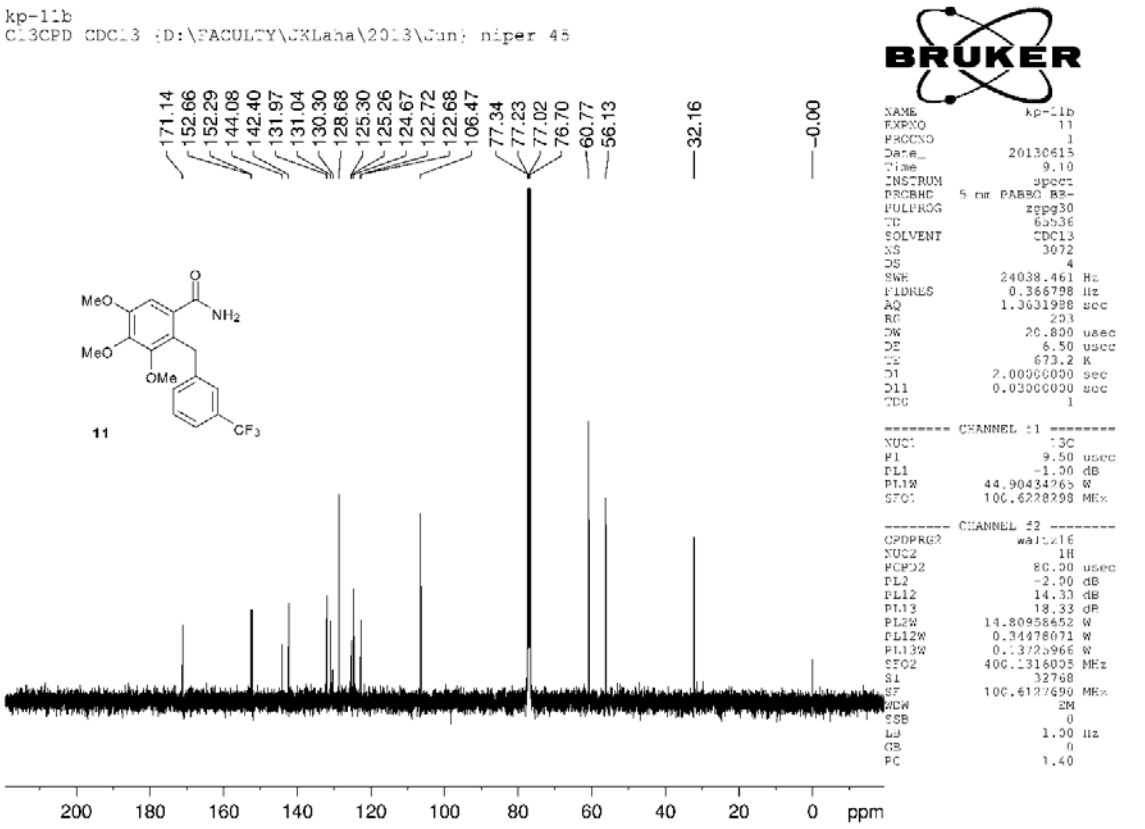


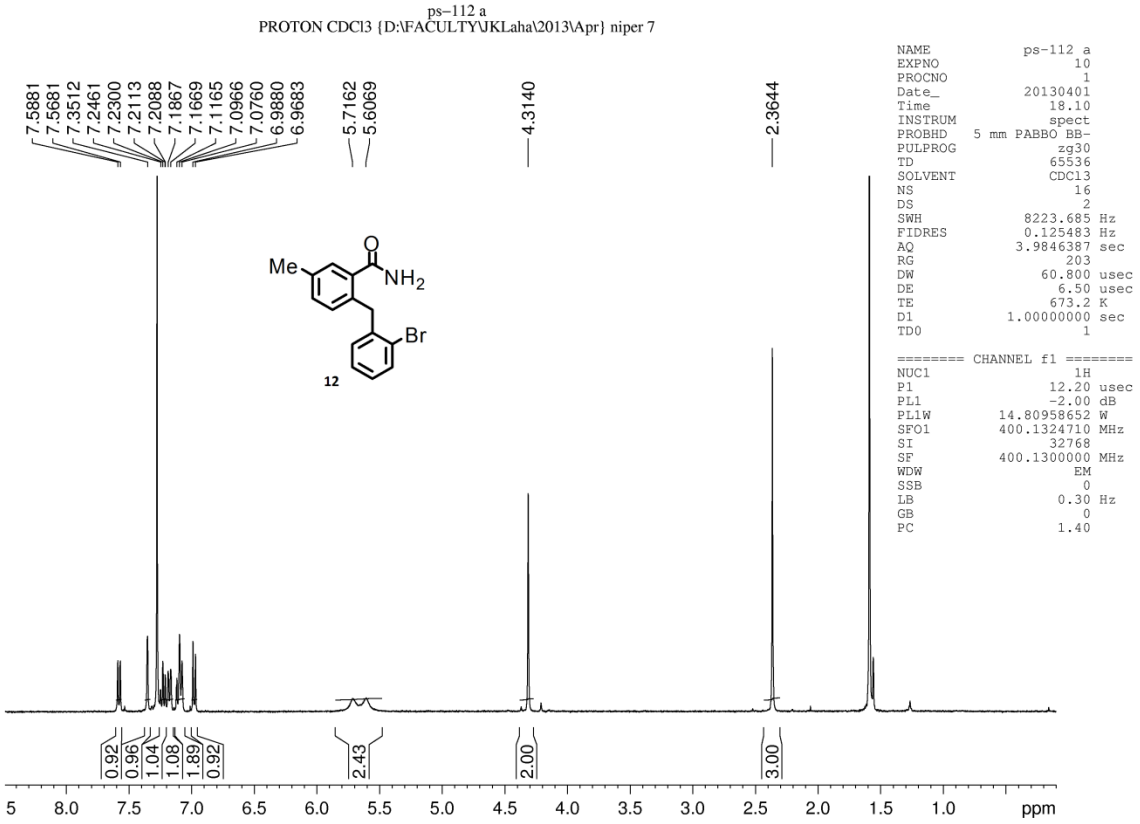

```
NAME                ps-109c
EXPNO                    11
PROCNO                    1
Date_              20130323
Time                   4.15
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      512
DS                        4
SWH              24038.461 Hz
FIDRES            0.366798 Hz
AQ                1.3631988 sec
RG                      203
DW                 20.800 usec
DE                  6.50 usec
TE                 673.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                    9.50 usec
PL1                  -1.00 dB
PL1W            44.90434265 W
SFO1            100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 14.33 dB
PL13                 18.33 dB
PL2W            14.80958652 W
PL12W            0.34478071 W
PL13W            0.13725966 W
SFO2            400.1316005 MHz
SI                    32768
SF              100.6127690 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

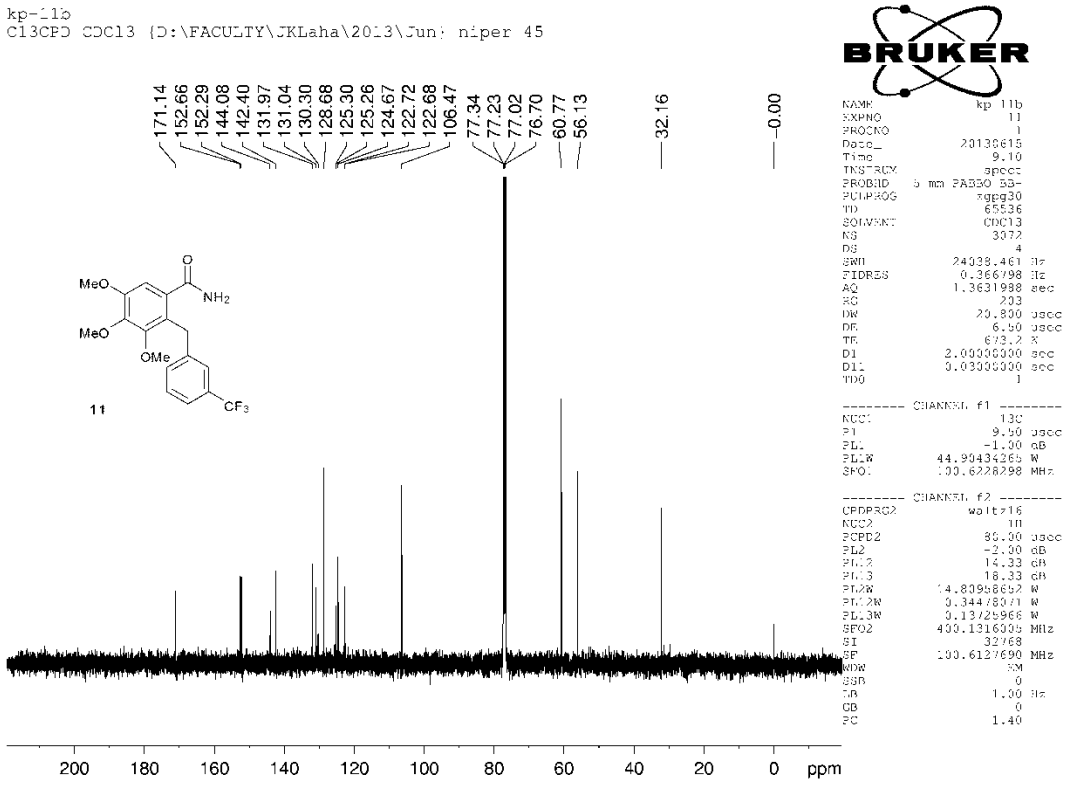


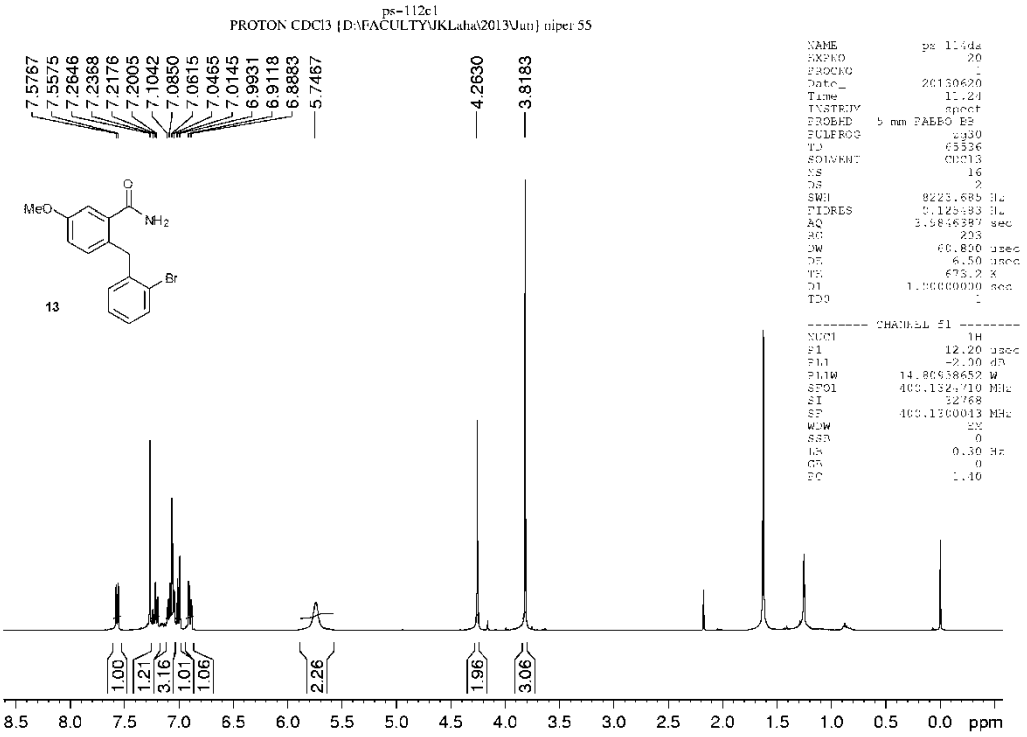


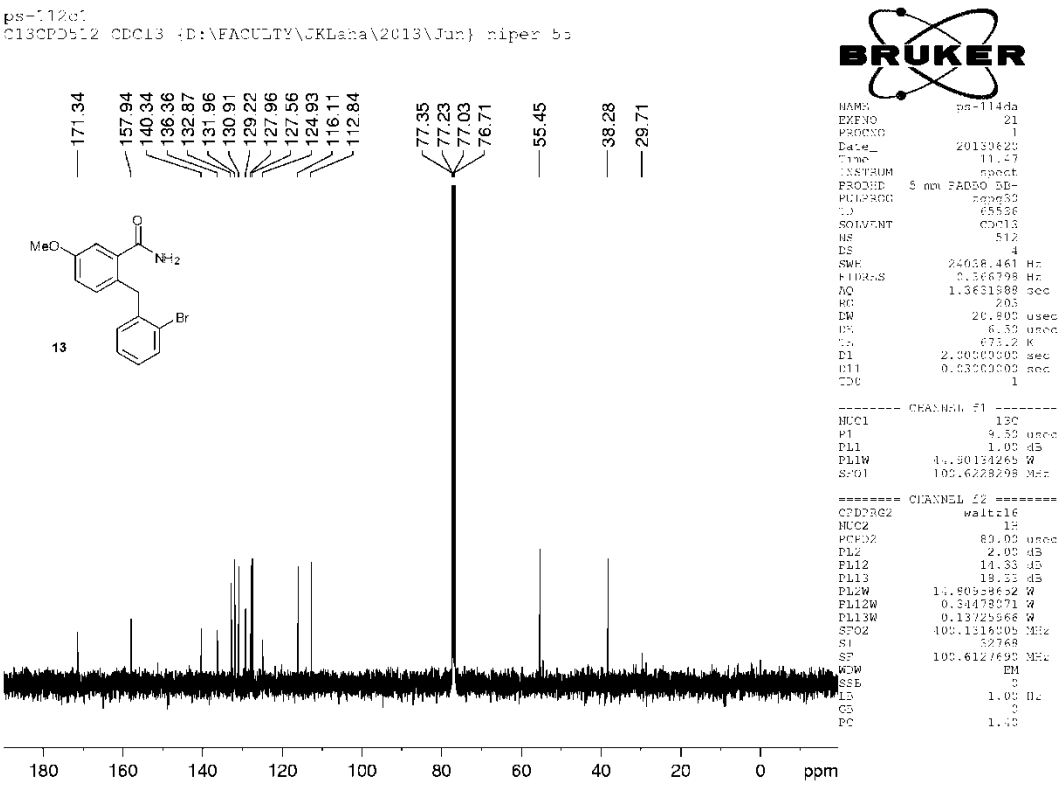

kp-11b
PROTON CDCl3 [D:\FACULTY\KLaha\2013\Apr] niper 44
7.4900
7.4089
7.3779
7.3593
7.3403
7.2753
6.8447
5.5952
4.2316
3.8968
3.7034
1.5908
1.5612
0.0126
MeO
MeO
OMe
NH2
CF3
11
1.04
2.98
0.95
2.11
2.00
6.08
2.97
ppm

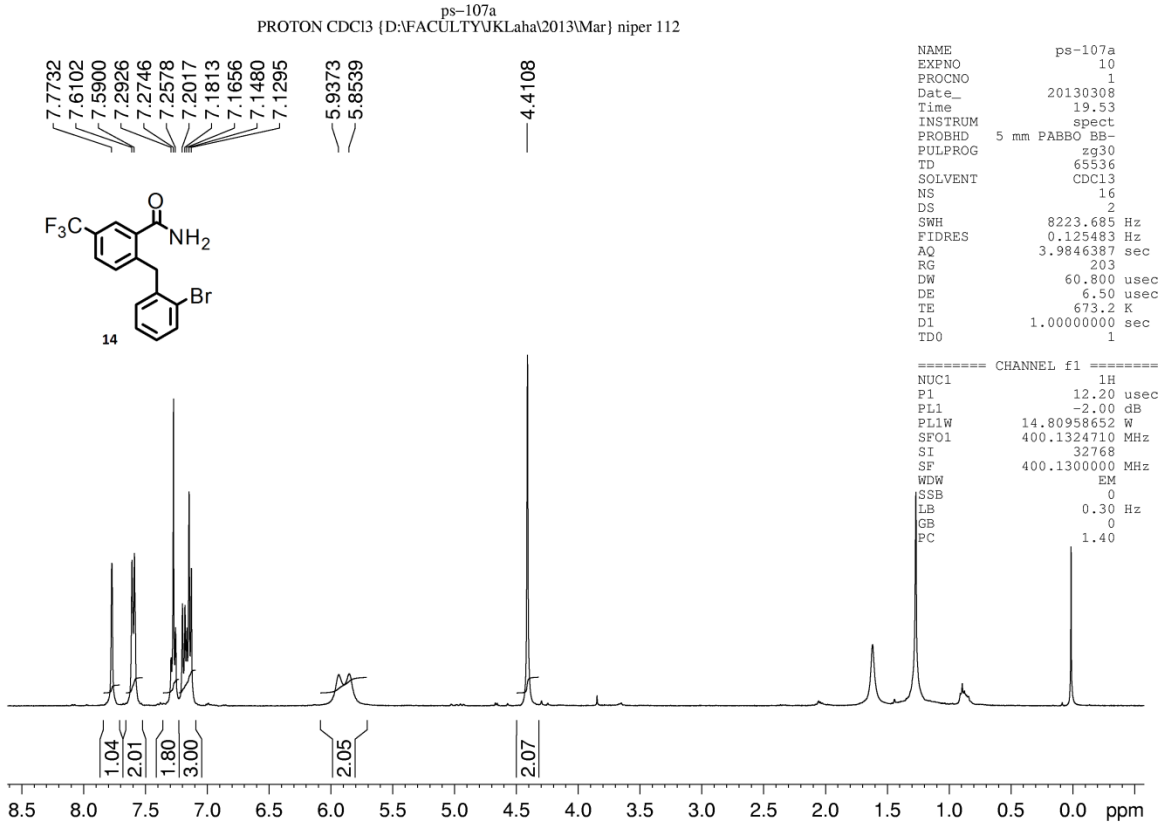

kp-11b
C13CPD CDCl3 [D:\FACULTY\KLaha\2013\Jun] niper 45
171.14
152.66
152.29
144.08
142.40
131.97
131.04
130.30
128.68
125.30
125.26
124.67
122.72
122.68
106.47
77.34
77.23
77.02
76.70
60.77
56.13
32.16
0.00
MeO
MeO
OMe
NH2
CF3
11
ppm

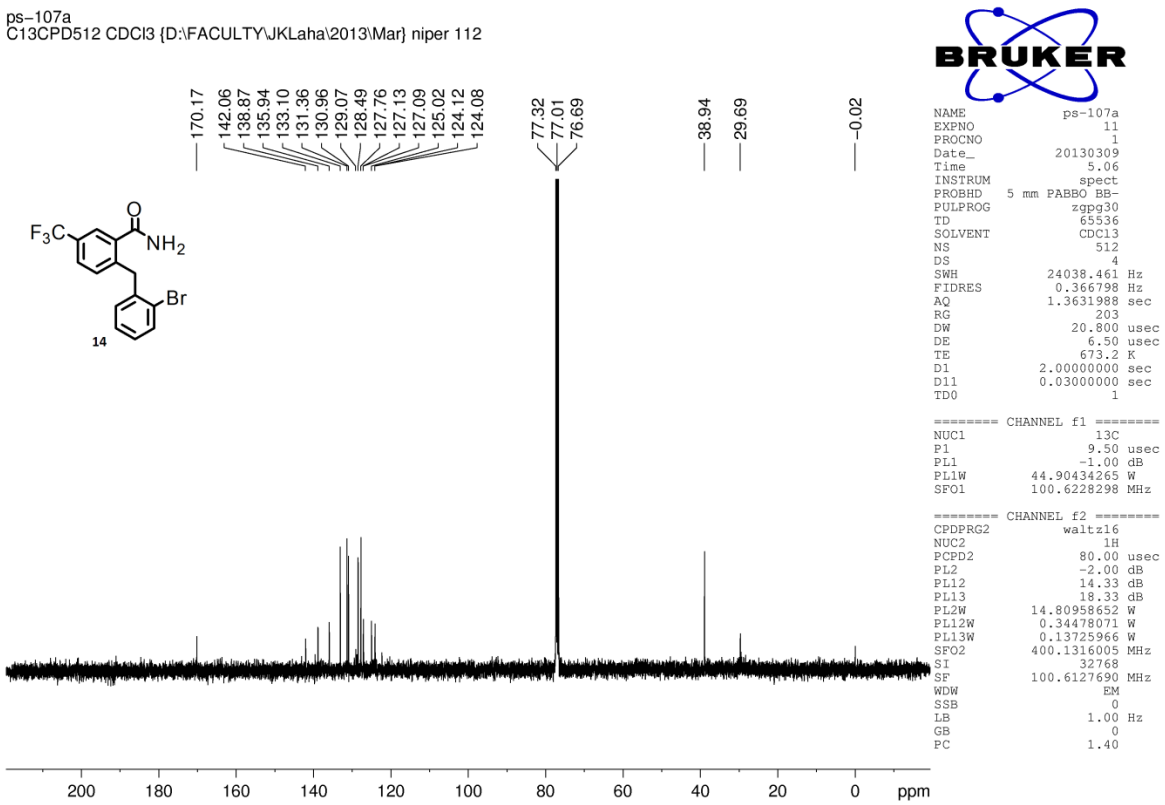

ps-112 a
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 7
7.5881
7.5681
7.3512
7.2461
7.2300
7.2113
7.2088
7.1867
7.1669
7.1165
7.0966
7.0760
6.9880
6.9683
5.7162
5.6069
4.3140
2.3644
Me
NH2
O
Br
12
0.92
0.96
1.04
1.08
1.89
0.92
2.43
2.00
3.00
ppm
NAME ps-112 a
EXPNO 10
PROCNO 1
Date_ 20130401
Time 18.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

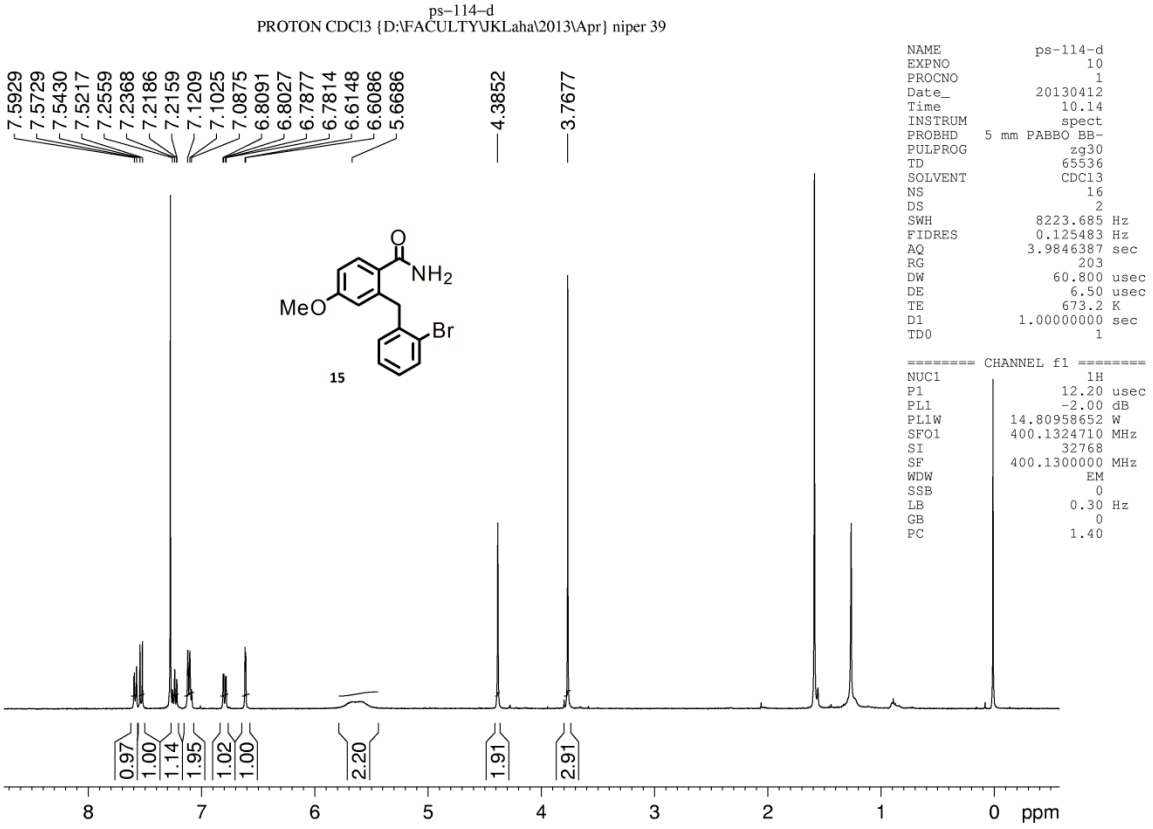

kp-11b
C13CPD CDCl3 {D:\FACULTY\JKLaha\2013\Jun; niper 45
171.14
152.66
152.29
144.08
142.40
131.97
131.04
130.30
128.68
125.30
125.26
124.67
122.72
122.68
106.47
77.34
77.23
77.02
76.70
60.77
56.13
32.16
0.00
MeO
MeO
OMe
NH2
O
CF3
11
ppm
NAME kp 11b
EXPNO 11
PROCNO 1
Date_ 20130615
Time 9.10
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13729968 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

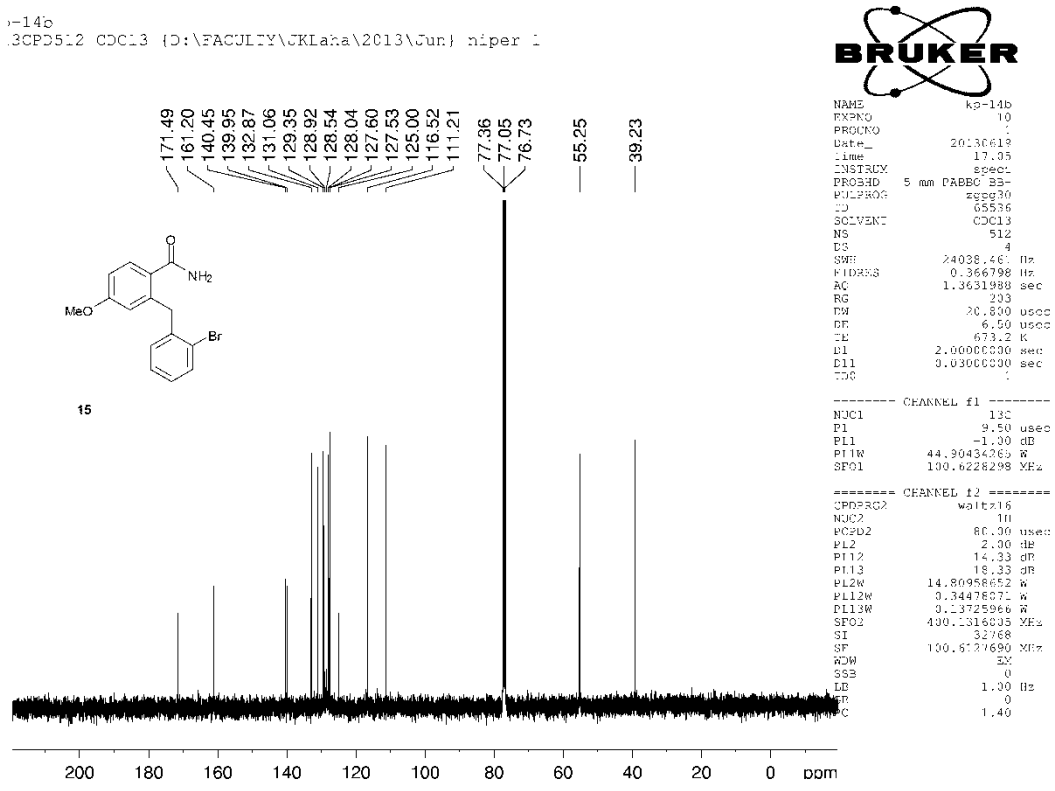

ps-112c1
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Jun} niper 55
7.5767
7.5575
7.2646
7.2368
7.2176
7.2005
7.1042
7.0850
7.0615
7.0465
7.0145
6.9931
6.9118
6.8883
5.7467
4.2630
3.8183
MeO
NH2
Br
13
1.00
1.21
3.16
1.01
1.06
2.26
1.96
3.06
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm

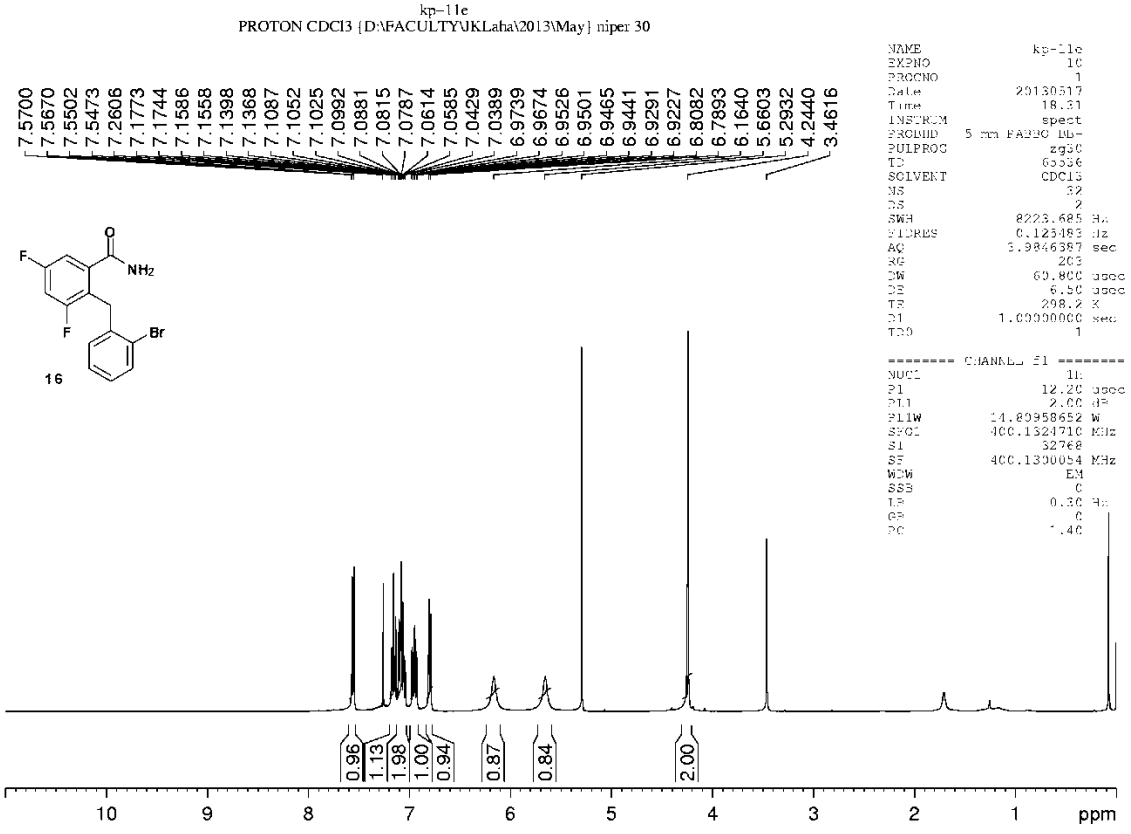

ps-112c1
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Jun} niper 55
171.34
157.94
140.34
136.36
132.87
131.96
130.91
129.22
127.96
127.56
124.93
116.11
112.84
77.35
77.23
77.03
76.71
55.45
38.28
29.71
MeO
NH2
Br
13
180 160 140 120 100 80 60 40 20 0 ppm

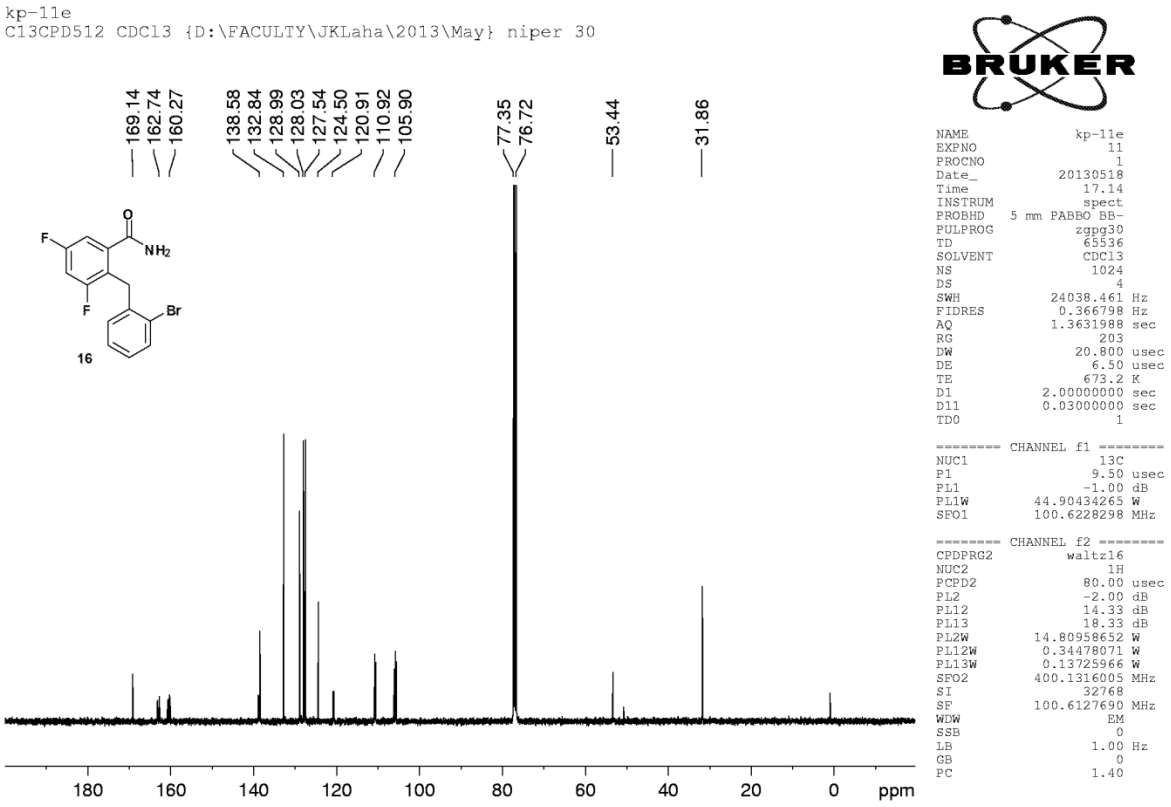


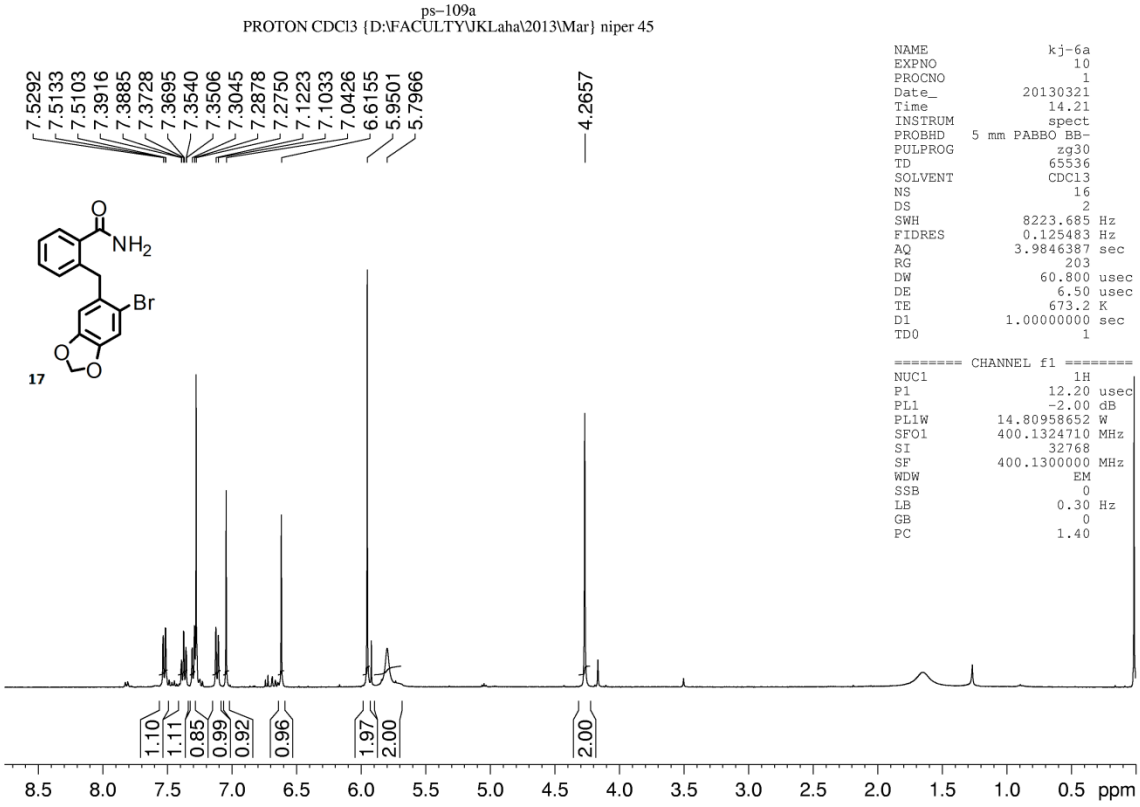


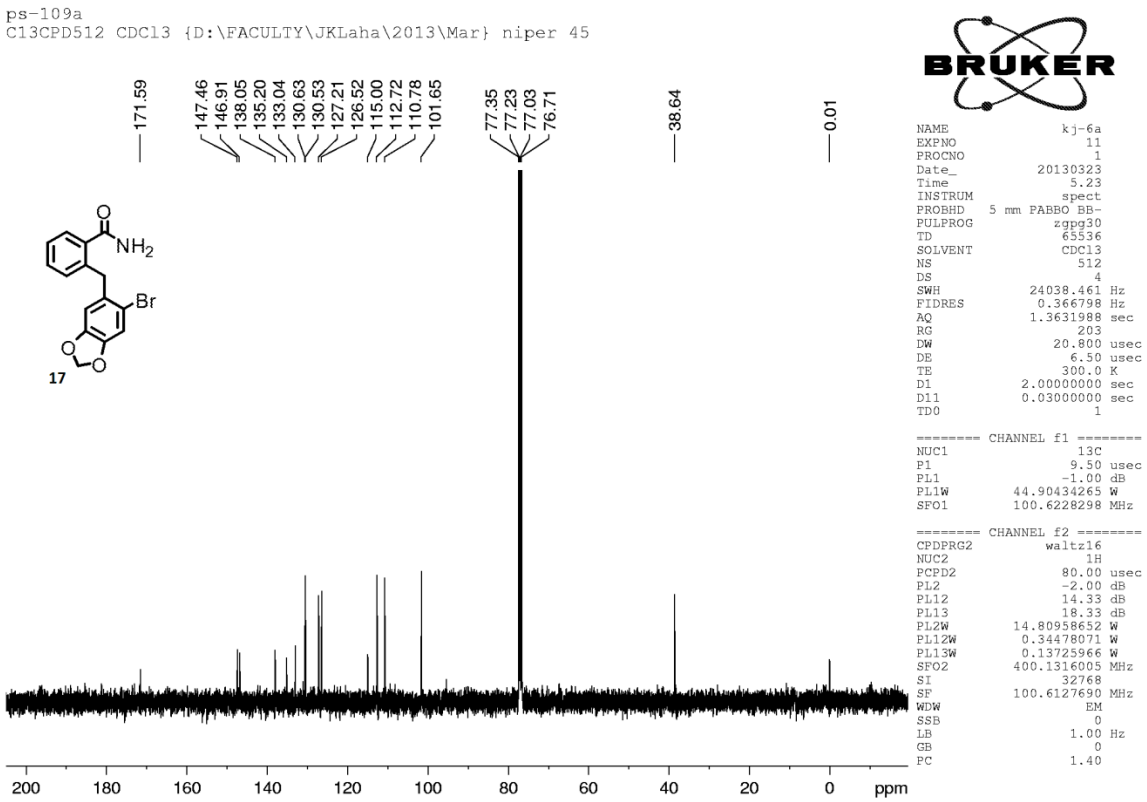

ps–114–d
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 39
7.5929
7.5729
7.5430
7.5217
7.2559
7.2368
7.2186
7.2159
7.1209
7.1025
7.0875
6.8091
6.8027
6.7877
6.7814
6.6148
6.6086
5.6686
4.3852
3.7677
NAME ps-114-d
EXPNO 10
PROCNO 1
Date_ 20130412
Time 10.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
NH2
MeO
Br
15
0.97
1.00
1.14
1.95
1.02
1.00
2.20
1.91
2.91
8
7
6
5
4
3
2
1
0
ppm

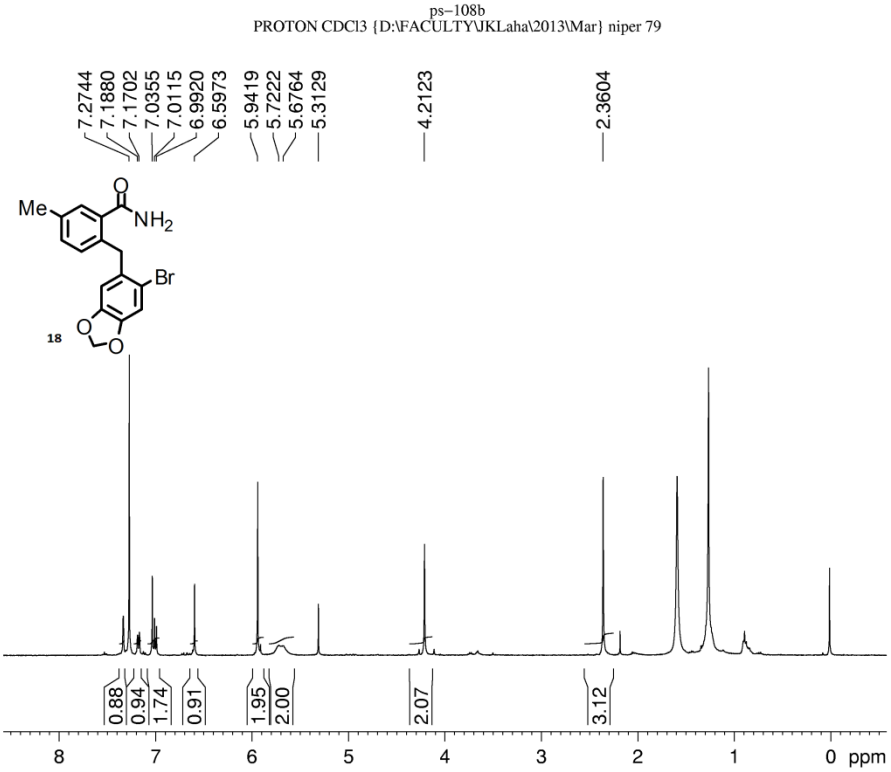

p-14b
13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Jun} niper 1
171.49
161.20
140.45
139.95
132.87
131.06
129.35
128.92
128.54
128.04
127.60
127.53
125.00
116.52
111.21
77.36
77.05
76.73
55.25
39.23
BRUKER
NAME kp-14b
EXPNO 10
PROCNO 1
Date_ 20130619
Time 17.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
-------- CHANNEL f1 --------
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478671 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NH2
MeO
Br
15
200
180
160
140
120
100
80
60
40
20
0
ppm


kp-11e
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\May} niper 30
7.5700
7.5670
7.5502
7.5473
7.2606
7.1773
7.1744
7.1586
7.1558
7.1398
7.1368
7.1087
7.1052
7.1025
7.0992
7.0881
7.0815
7.0787
7.0614
7.0585
7.0429
7.0389
6.9739
6.9674
6.9526
6.9501
6.9465
6.9441
6.9291
6.9227
6.8082
6.7893
6.1640
5.6603
5.2932
4.2440
3.4616
NAME kp-11e
EXPNO 10
PROCNO 1
Date 20130517
Time 18.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300054 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
F
NH2
O
F
Br
16
0.96
1.13
1.98
1.00
0.94
0.87
0.84
2.00
10
9
8
7
6
5
4
3
2
1
ppm

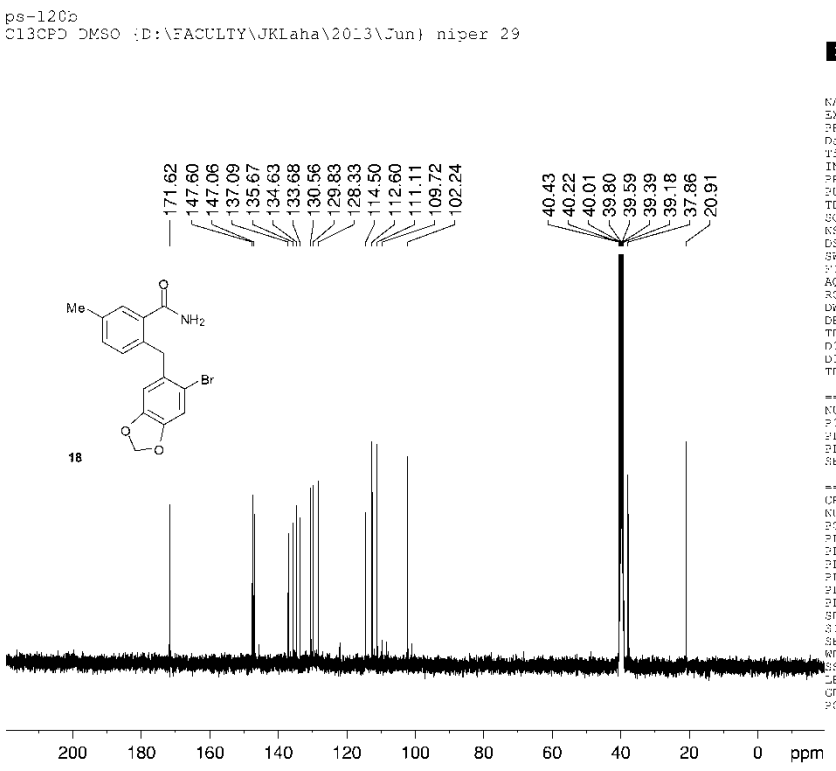

kp-11e
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\May} niper 30
169.14
162.74
160.27
138.58
132.84
128.99
128.03
127.54
124.50
120.91
110.92
105.90
77.35
76.72
53.44
31.86
BRUKER
NAME kp-11e
EXPNO 11
PROCNO 1
Date_ 20130518
Time 17.14
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
F
NH2
O
F
Br
16
180
160
140
120
100
80
60
40
20
0
ppm


ps-109a
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Mar} niper 45
7.5292
7.5133
7.5103
7.3916
7.3885
7.3728
7.3695
7.3540
7.3506
7.3045
7.2878
7.2750
7.1223
7.1033
7.0426
6.6155
5.9501
5.7966
4.2657
NH2
Br
17
1.10
1.11
0.85
0.99
0.92
0.96
1.97
2.00
2.00
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
NAME kj-6a
EXPNO 10
PROCNO 1
Date_ 20130321
Time 14.21
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

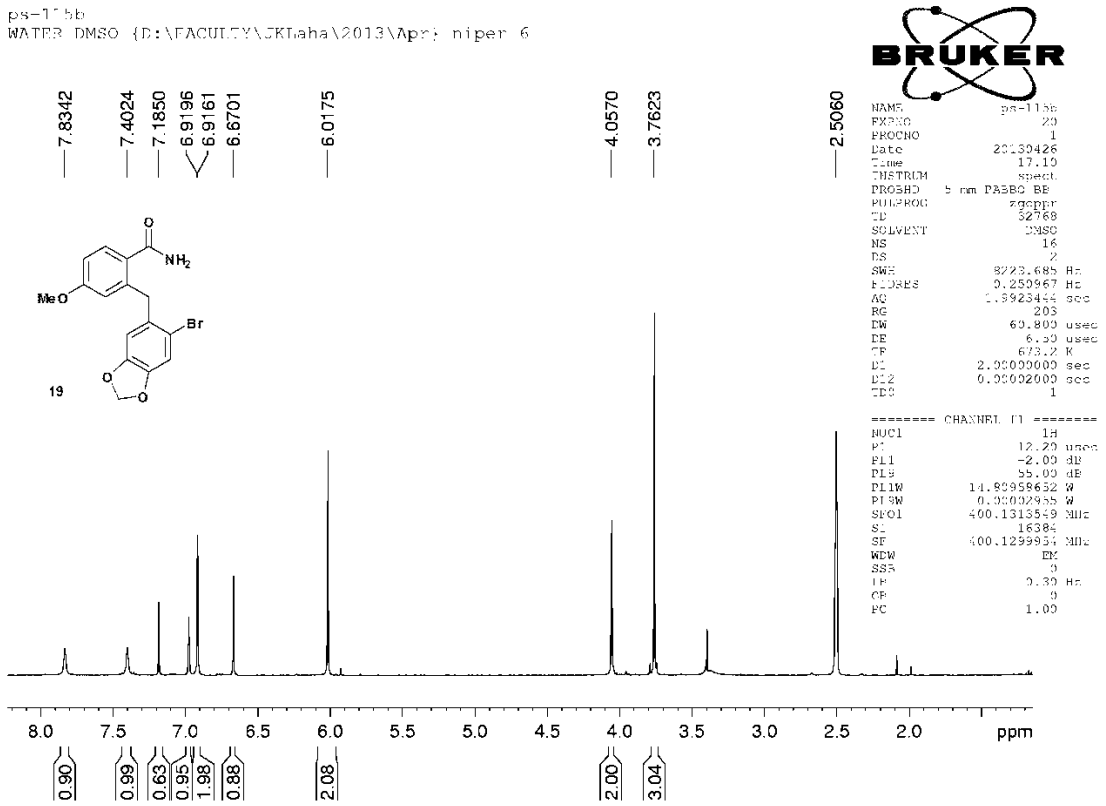

ps-109a
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Mar} niper 45
171.59
147.46
146.91
138.05
135.20
133.04
130.63
130.53
127.21
126.52
115.00
112.72
110.78
101.65
77.35
77.23
77.03
76.71
38.64
0.01
NH2
Br
17
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME kj-6a
EXPNO 11
PROCNO 1
Date_ 20130323
Time 5.23
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

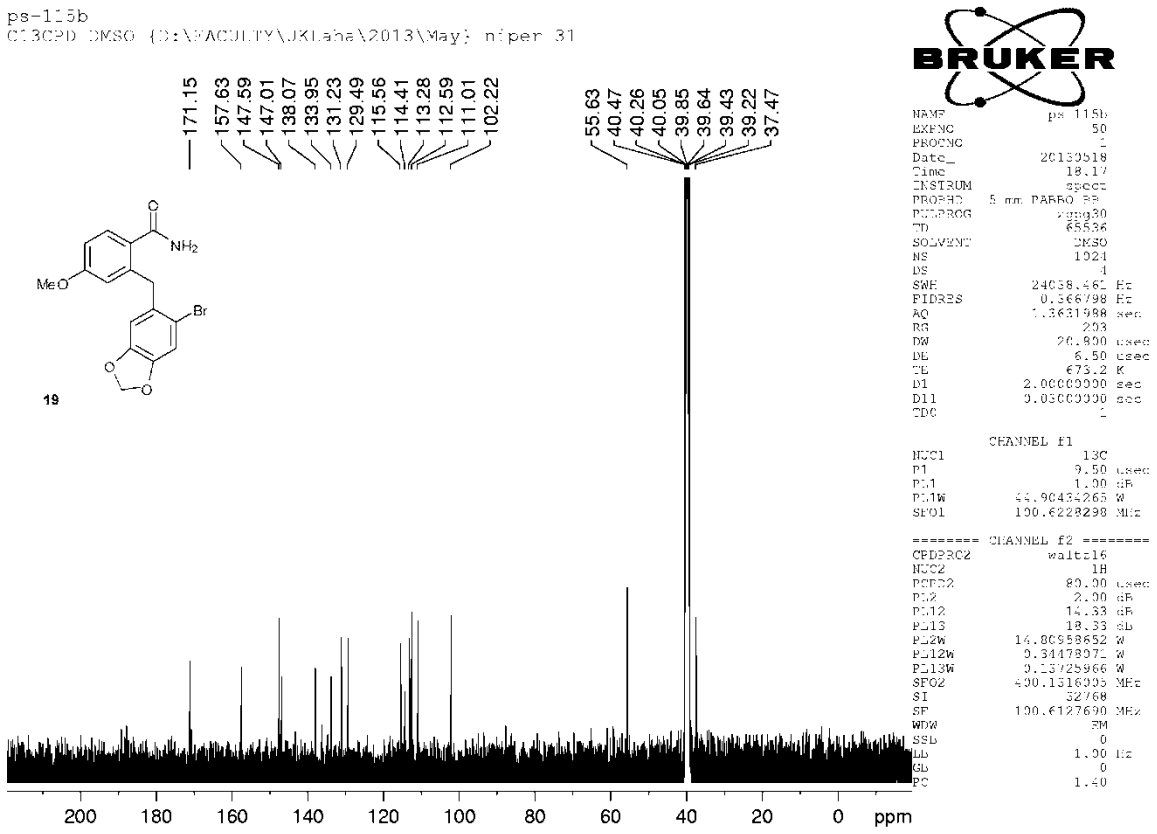


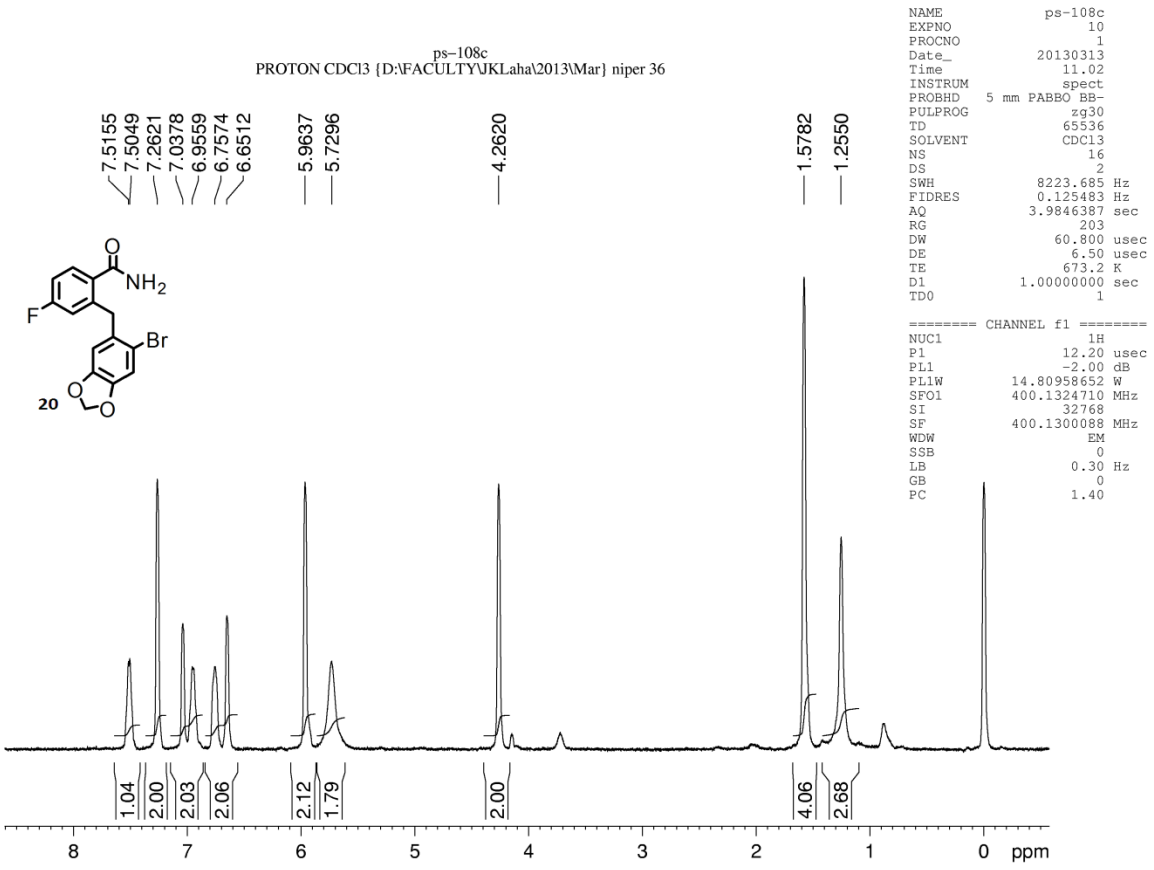

```
NAME                ps-108b
EXPNO                    10
PROCNO                    1
Date_              20130314
Time                  17.42
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      203
DW                   60.800 usec
DE                    6.50 usec
TE                   673.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                     12.20 usec
PL1                    -2.00 dB
PL1W            14.80958652 W
SFO1           400.1324710 MHz
SI                     32768
SF              400.1300000 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.40
```

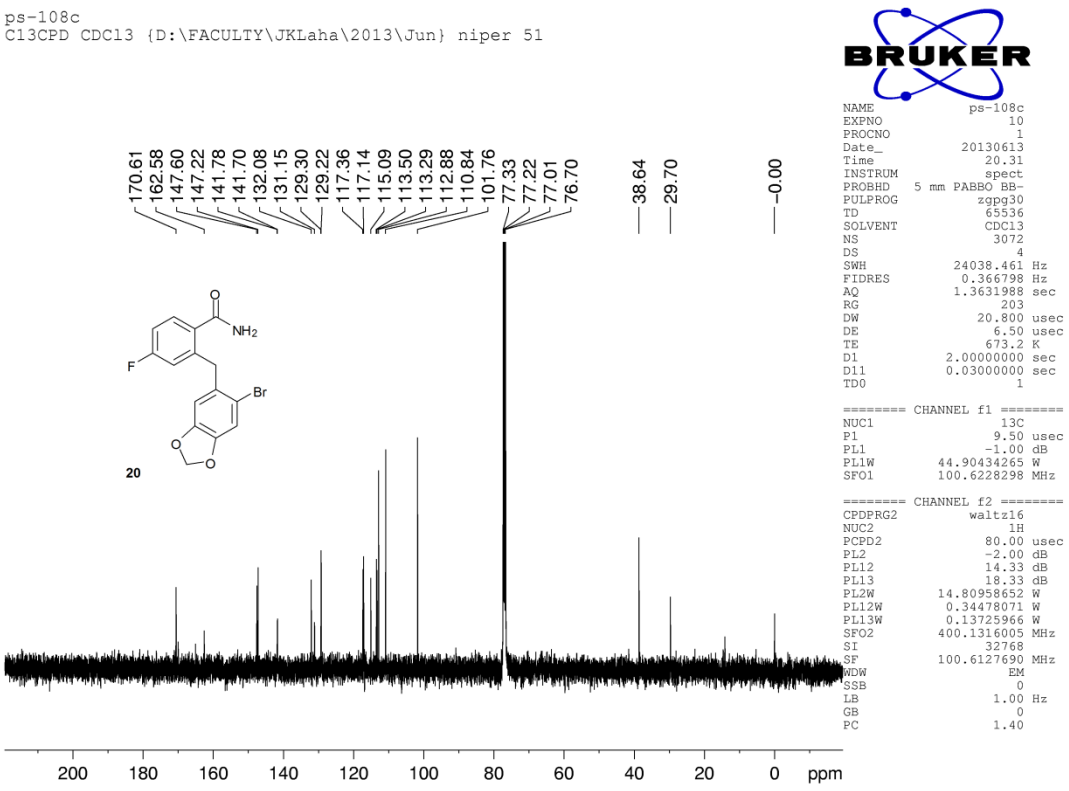


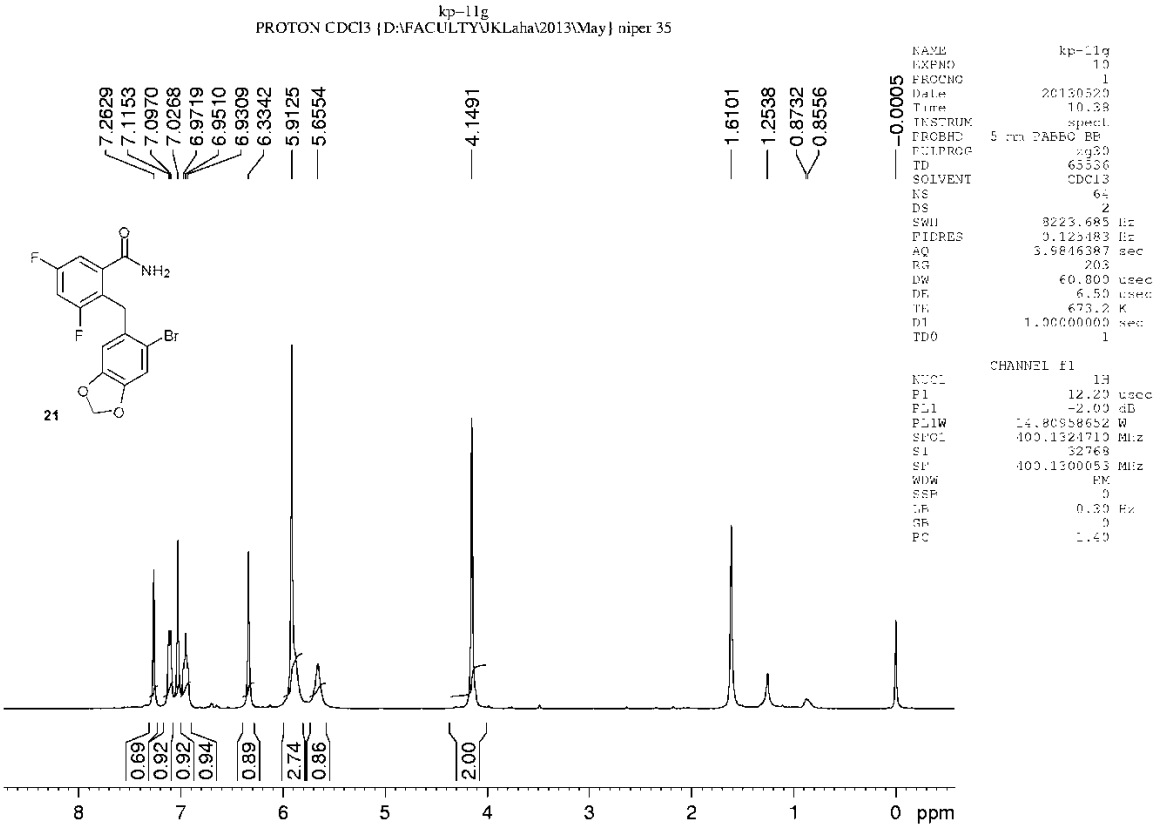

```
NAME                ps-120b
EXPNO                    11
PROCNO                    1
Date_              20130615
Time                  20.53
INSTRUM               spect
PROBHD   5 mm PABBO BB-
PULPROG             zgpg30
TD                    65536
SOLVENT                DMSO
NS                     1024
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                      203
DW                   20.800 usec
DE                    6.50 usec
TE                   673.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                     9.50 usec
PL1                    1.00 dB
PL1W            44.90434265 W
SFO1           100.6228298 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                      1H
PCPD2                  80.00 usec
PL2                    -2.00 dB
PL12                   14.33 dB
PL13                   18.33 dB
PL2W            14.80958653 W
PL12W            0.34478071 W
PL13W            0.13725966 W
SFO2           400.1316005 MHz
SI                     32768
SF              100.6127690 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

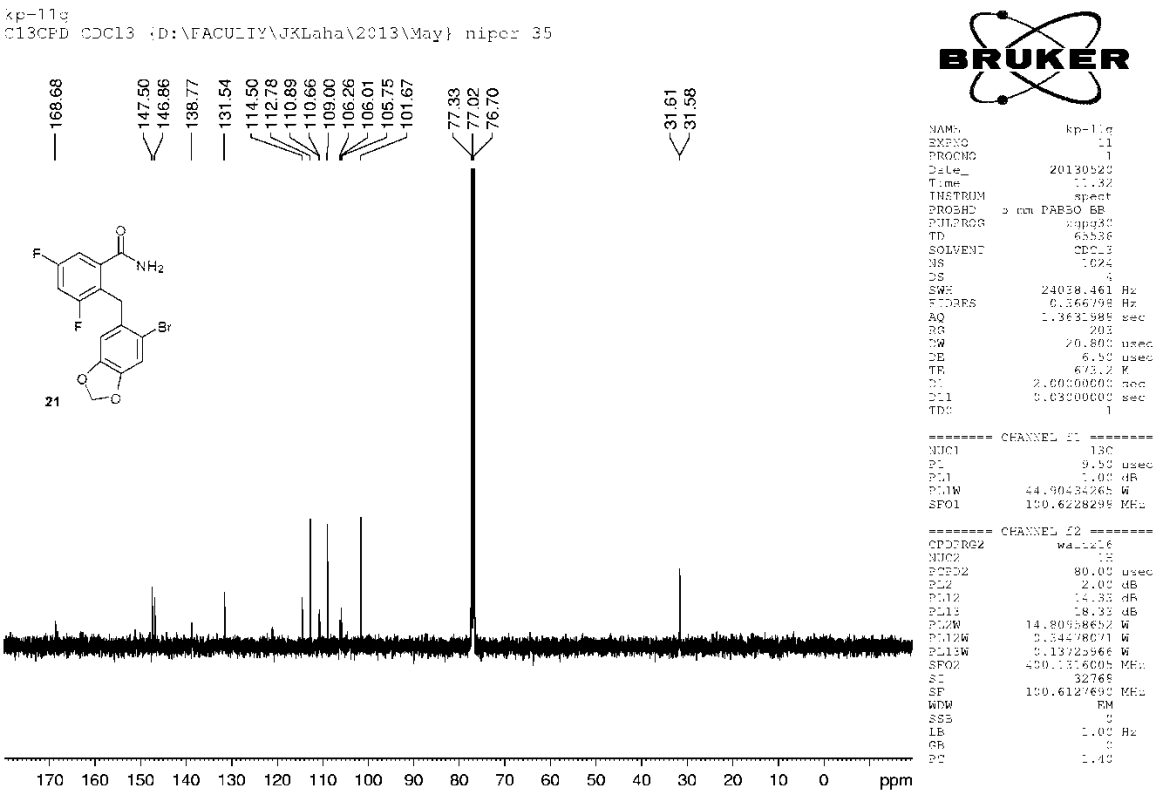

ps-115b
WATER DMSO (D:\FACULTY\JKLaha\2013\Apr) niper 6
7.8342
7.4024
7.1850
6.9196
6.9161
6.6701
6.0175
4.0570
3.7623
2.5060
MeO
NH2
Br
O
O
19
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
ppm
0.90
0.99
0.63
0.95
1.98
0.88
2.08
2.00
3.04
BRUKER
NAME ps-115b
EXPNO 20
PROCNO 1
Date 20130426
Time 17.10
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgppr
TD 32768
SOLVENT DMSO
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.250967 Hz
AQ 1.9923444 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D12 0.00002000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL9 55.00 dB
PL1W 14.89998652 W
PL9W 0.00002955 W
SFO1 400.1313549 MHz
SI 16384
SF 400.1299954 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

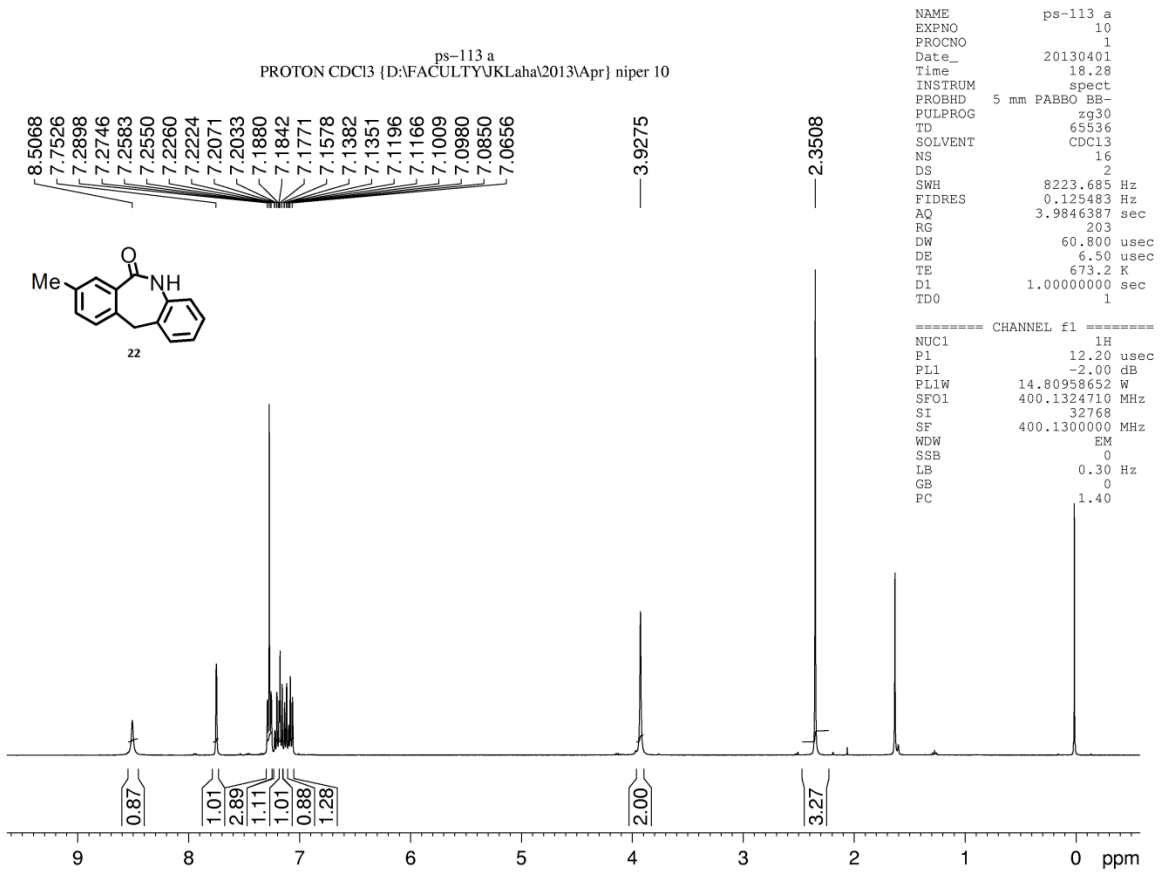

ps-115b
C13CPD DMSO (D:\FACULTY\JKLaha\2013\May) niper 31
171.15
157.63
147.59
147.01
138.07
133.95
131.23
129.49
115.56
114.41
113.28
112.59
111.01
102.22
55.63
40.47
40.26
40.05
39.85
39.64
39.43
39.22
37.47
MeO
NH2
Br
O
O
19
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME ps-115b
EXPNO 50
PROCNO 1
Date_ 20130518
Time 18.17
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
CHANNEL f1
NUC1 13C
P1 9.50 usec
PL1 1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.89958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

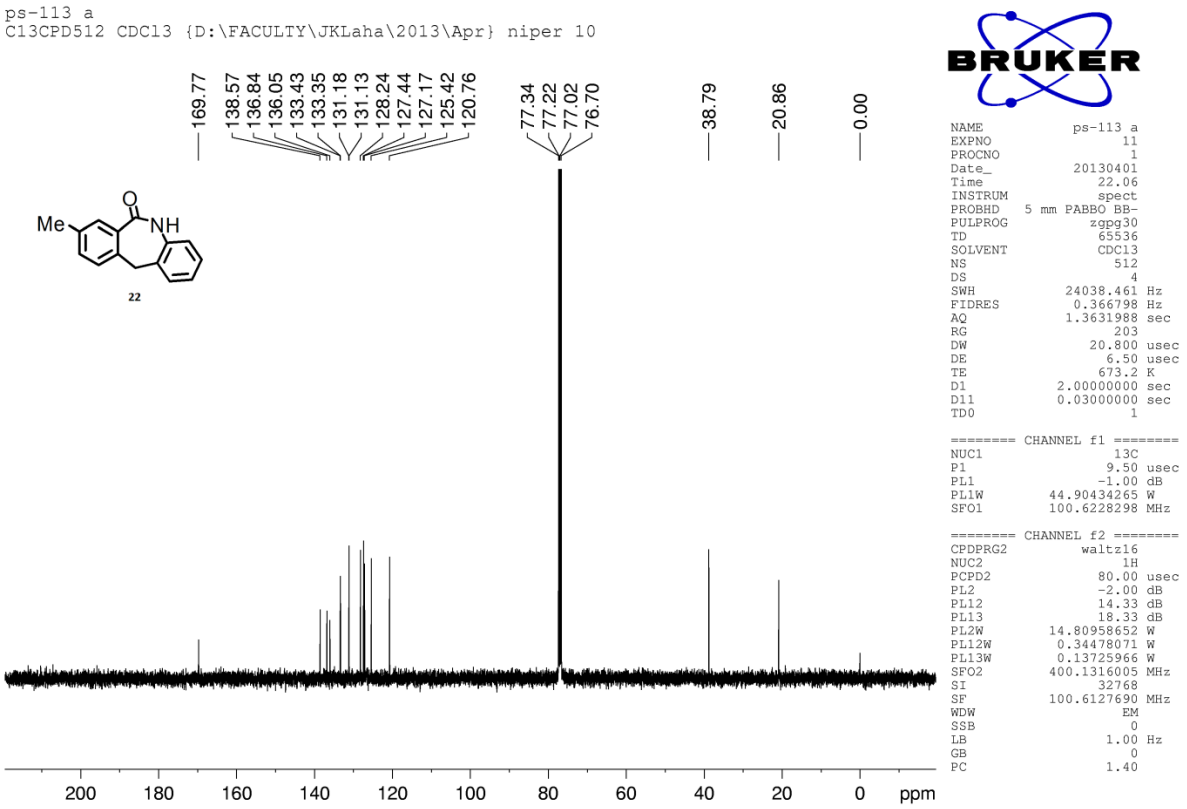

ps-108c
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Mar} niper 36
7.5155
7.5049
7.2621
7.0378
6.9559
6.7574
6.6512
5.9637
5.7296
4.2620
1.5782
1.2550
NAME ps-108c
EXPNO 10
PROCNO 1
Date_ 20130313
Time 11.02
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300088 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40
NH2
F
Br
O
O
O
20
1.04
2.00
2.03
2.06
2.12
1.79
2.00
4.06
2.68
8
7
6
5
4
3
2
1
0
ppm

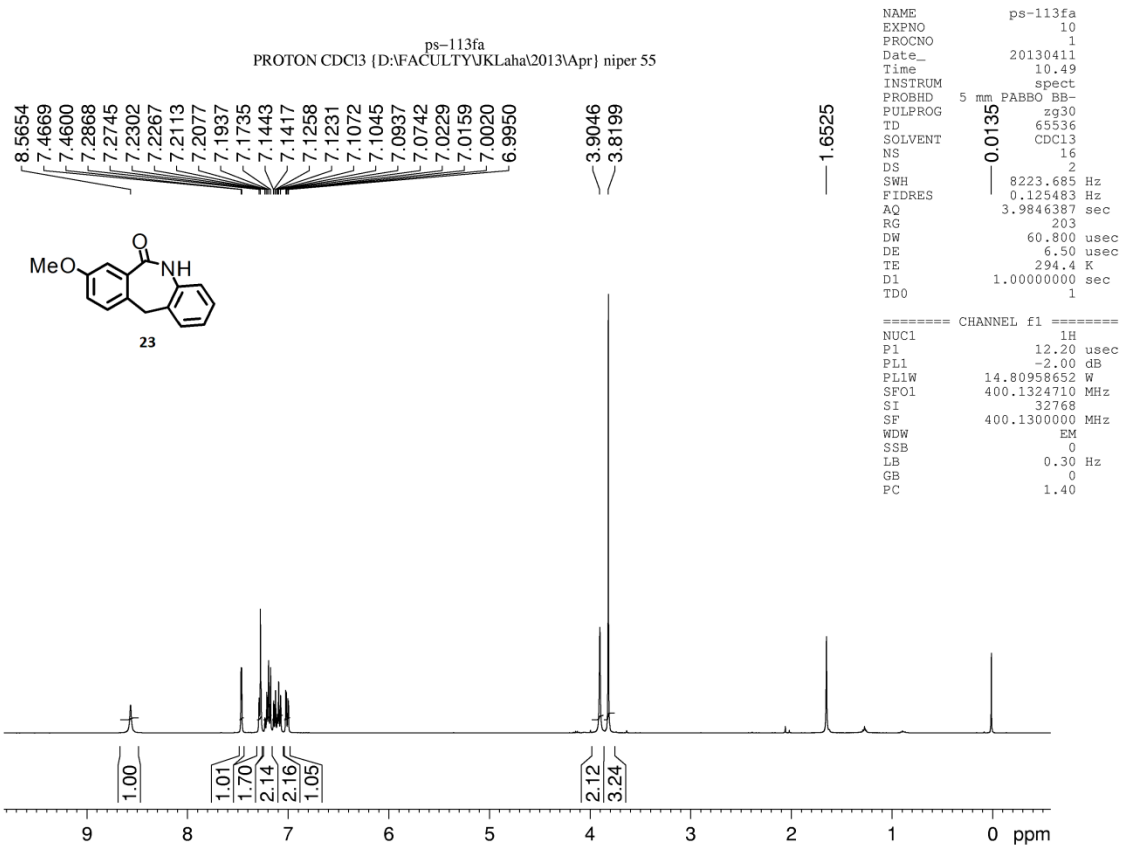

ps-108c
C13CPD CDCl3 {D:\FACULTY\JKLaha\2013\Jun} niper 51
BRUKER
170.61
162.58
147.60
147.22
141.78
141.70
132.08
131.15
129.30
129.22
117.36
117.14
115.09
113.50
113.29
112.88
110.84
101.76
77.33
77.22
77.01
76.70
38.64
29.70
0.00
NAME ps-108c
EXPNO 10
PROCNO 1
Date_ 20130613
Time 20.31
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3072
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
NH2
F
Br
O
O
O
20
200
180
160
140
120
100
80
60
40
20
0
ppm

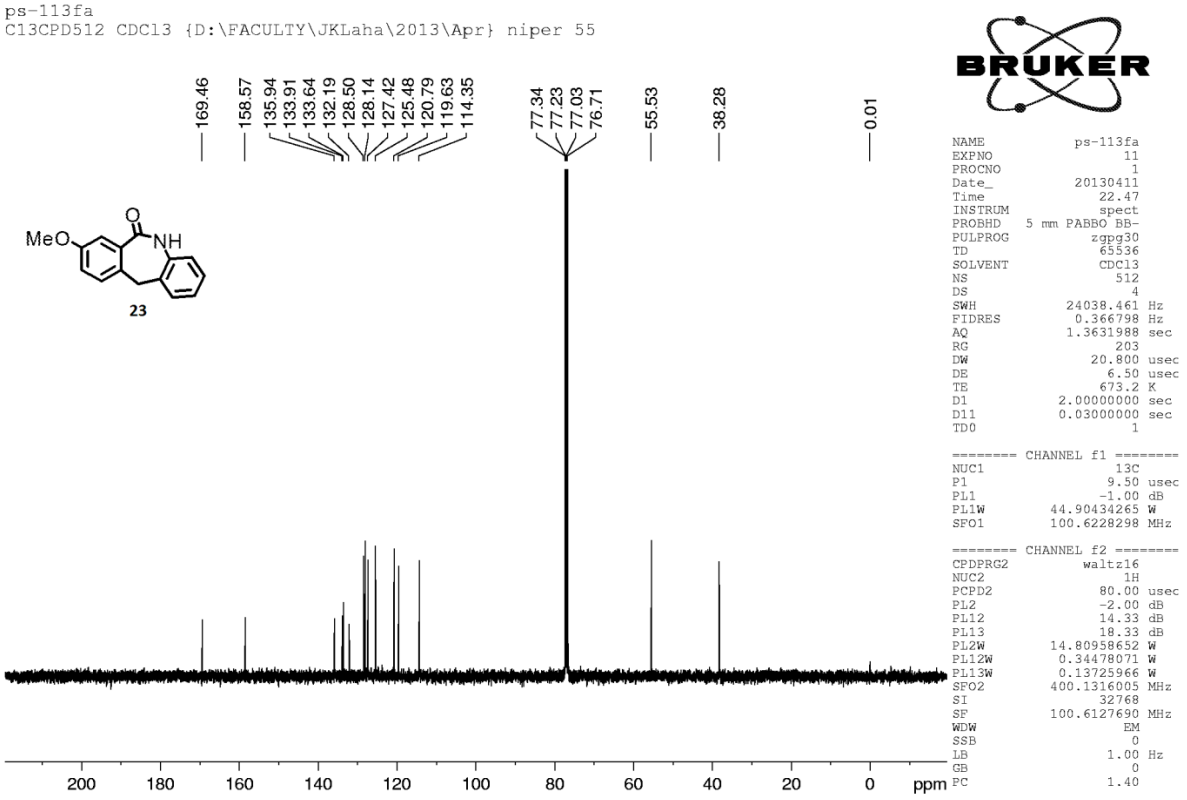

kp-11g
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\May} niper 35
7.2629
7.1153
7.0970
7.0268
6.9719
6.9510
6.9309
6.3342
5.9125
5.6554
4.1491
1.6101
1.2538
0.8732
0.8556
0.0005
0.69
0.92
0.92
0.94
0.89
2.74
0.86
2.00
8
7
6
5
4
3
2
1
0 ppm
21

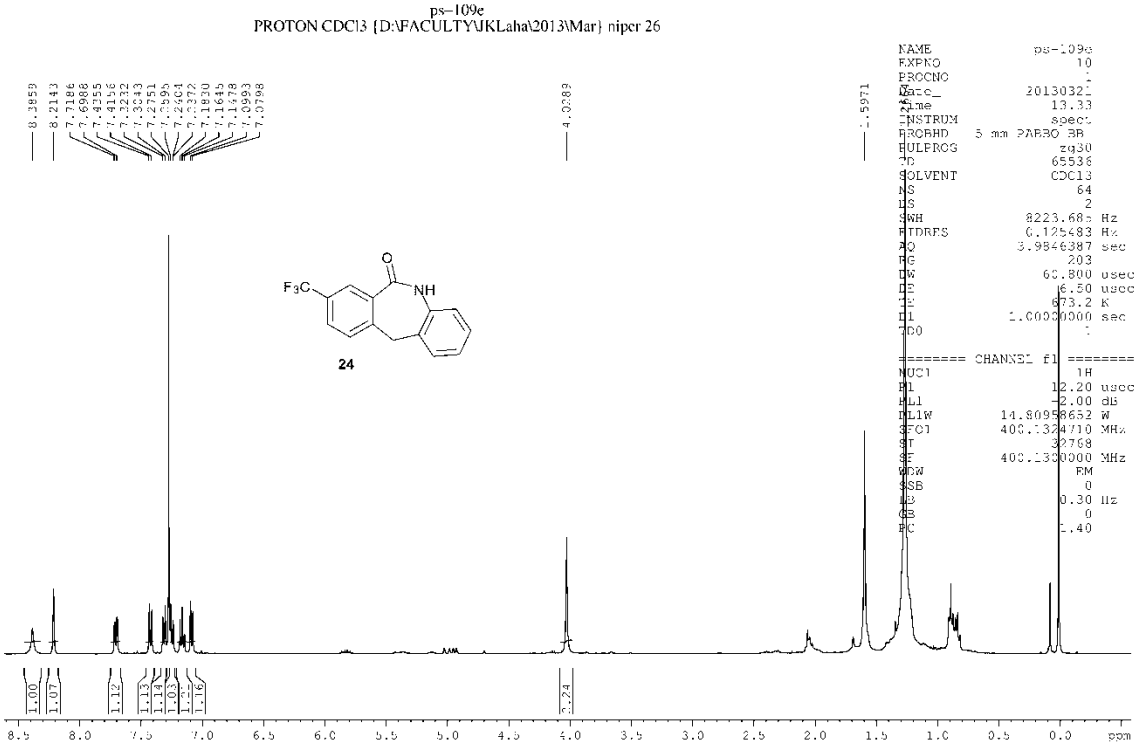

kp-11g
C13CPD CDCl3 {D:\FACULTY\JKLaha\2013\May} niper 35
168.68
147.50
146.86
138.77
131.54
114.50
112.78
110.89
110.66
109.00
106.26
106.01
105.75
101.67
77.33
77.02
76.70
31.61
31.58
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
21

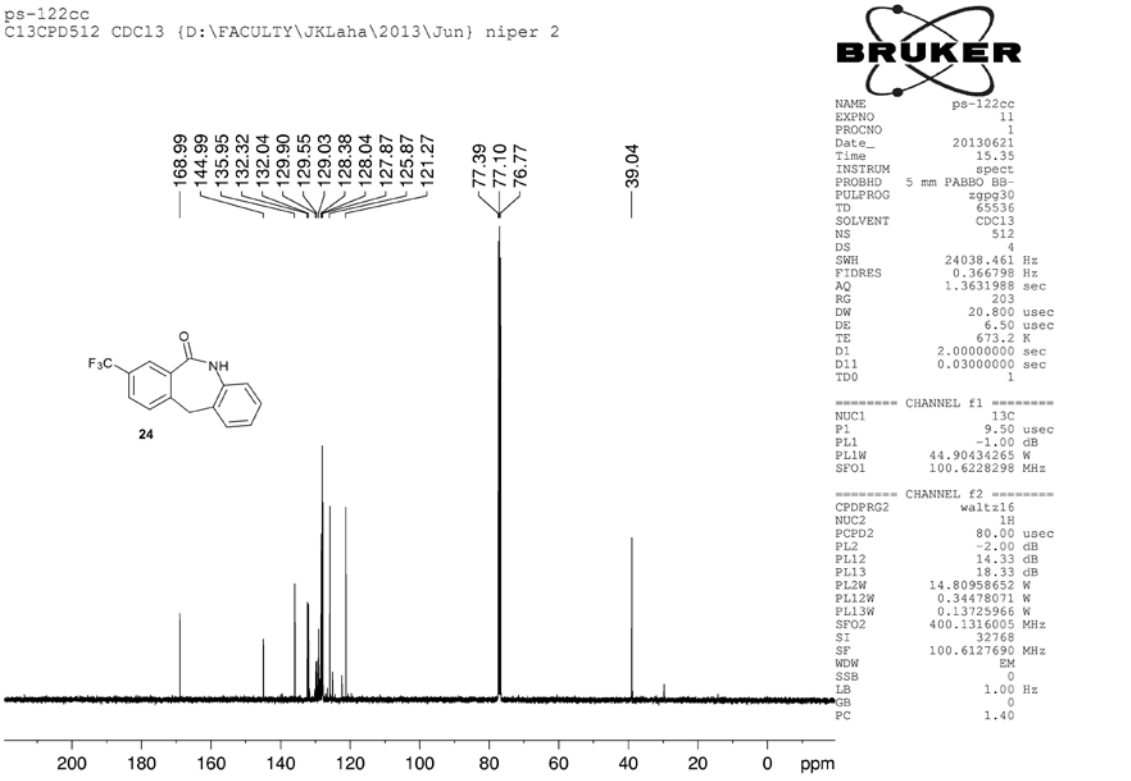

ps-113 a
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 10
8.5068
7.7526
7.2898
7.2746
7.2583
7.2550
7.2260
7.2224
7.2071
7.2033
7.1880
7.1842
7.1771
7.1578
7.1382
7.1351
7.1196
7.1166
7.1009
7.0980
7.0850
7.0656
3.9275
2.3508
Me
NH
O
22
0.87
1.01
2.89
1.11
1.01
0.88
1.28
2.00
3.27
9
8
7
6
5
4
3
2
1
0
ppm
NAME ps-113 a
EXPNO 10
PROCNO 1
Date_ 20130401
Time 18.28
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

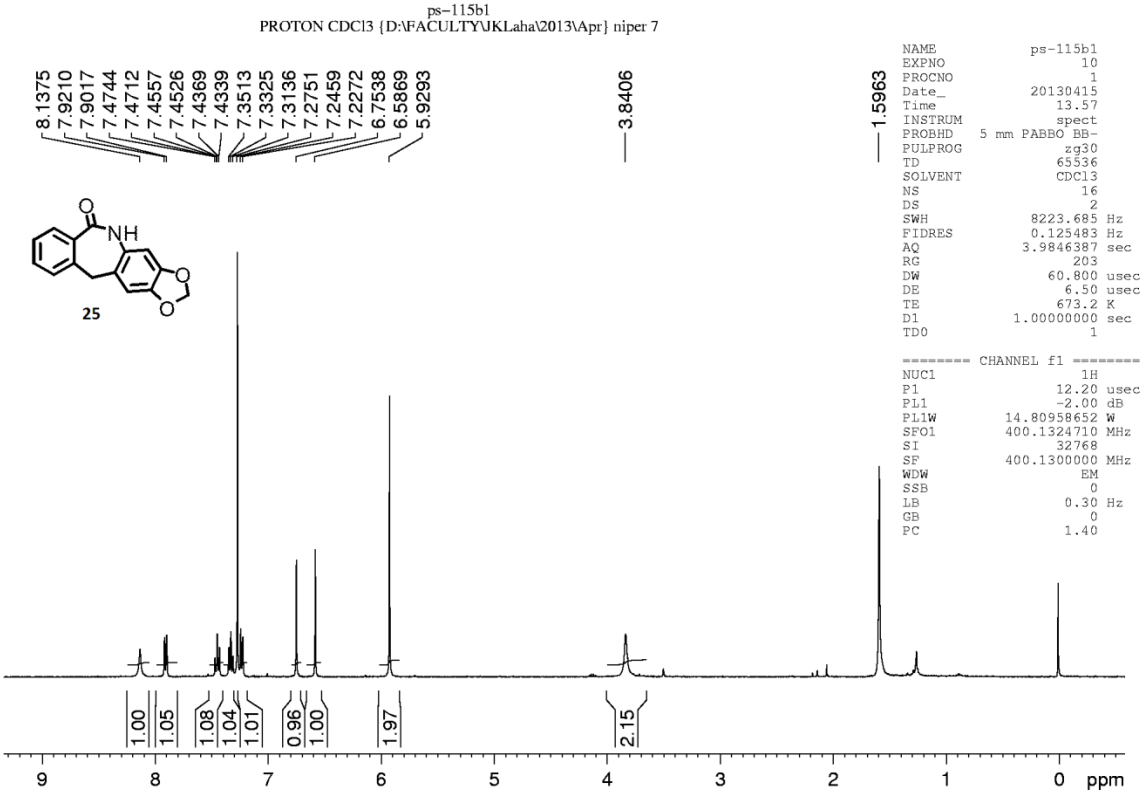

ps-113 a
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 10
169.77
138.57
136.84
136.05
133.43
133.35
131.18
131.13
128.24
127.44
127.17
125.42
120.76
77.34
77.22
77.02
76.70
38.79
20.86
0.00
Me
NH
O
22
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME ps-113 a
EXPNO 11
PROCNO 1
Date_ 20130401
Time 22.06
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

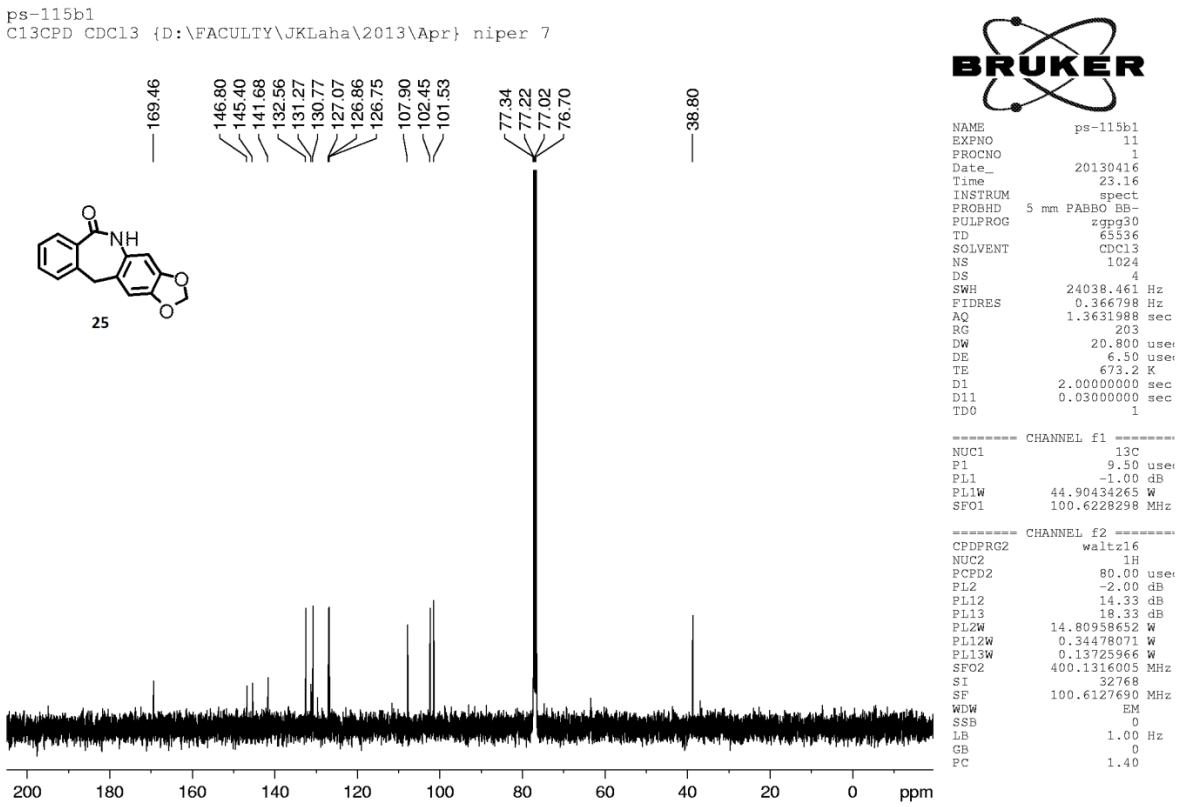

ps-113fa
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 55
8.5654
7.4669
7.4600
7.2868
7.2745
7.2302
7.2267
7.2113
7.2077
7.1937
7.1735
7.1443
7.1417
7.1258
7.1231
7.1072
7.1045
7.0937
7.0742
7.0229
7.0159
7.0020
6.9950
3.9046
3.8199
1.6525
0.0135
MeO
NH
O
23
1.00
1.01
1.70
2.14
2.16
1.05
2.12
3.24
9
8
7
6
5
4
3
2
1
0 ppm
NAME ps-113fa
EXPNO 10
PROCNO 1
Date_ 20130411
Time 10.49
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 294.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

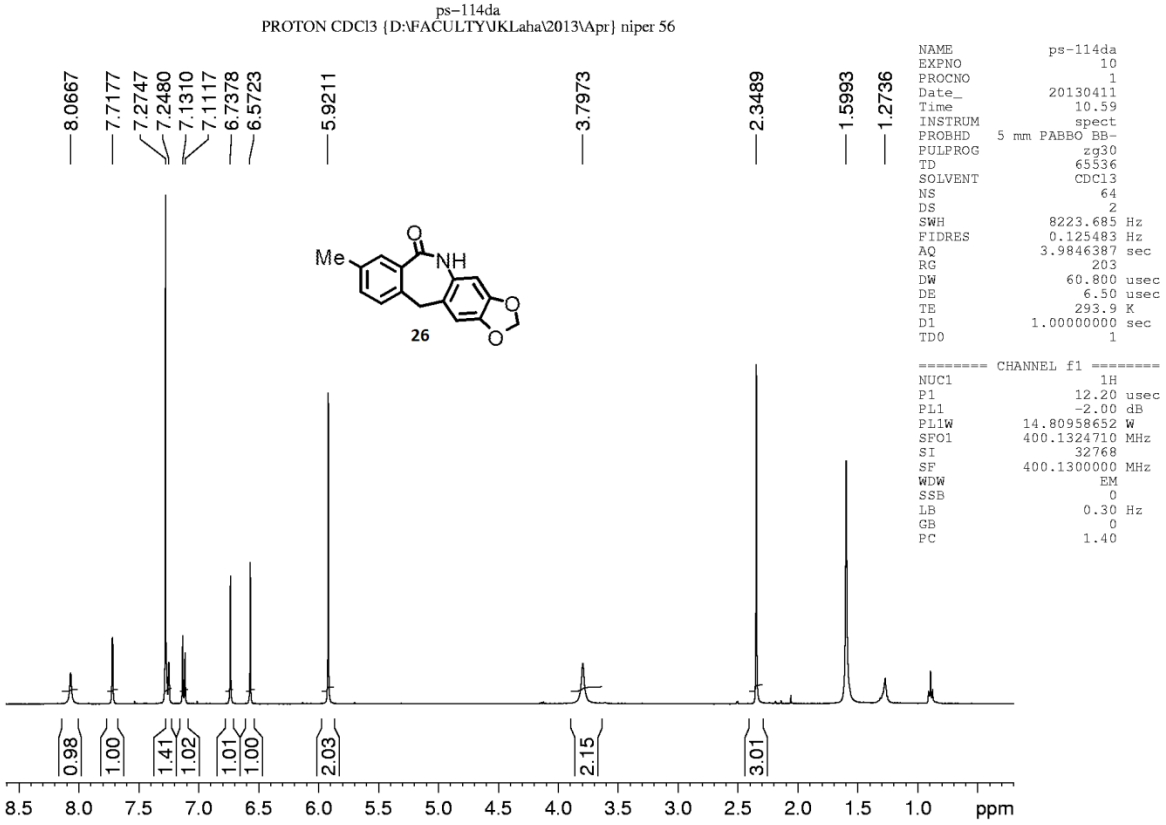

ps-113fa
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 55
169.46
158.57
135.94
133.91
133.64
132.19
128.50
128.14
127.42
125.48
120.79
119.63
114.35
77.34
77.23
77.03
76.71
55.53
38.28
0.01
MeO
NH
O
23
200
180
160
140
120
100
80
60
40
20
0 ppm
BRUKER
NAME ps-113fa
EXPNO 11
PROCNO 1
Date_ 20130411
Time 22.47
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

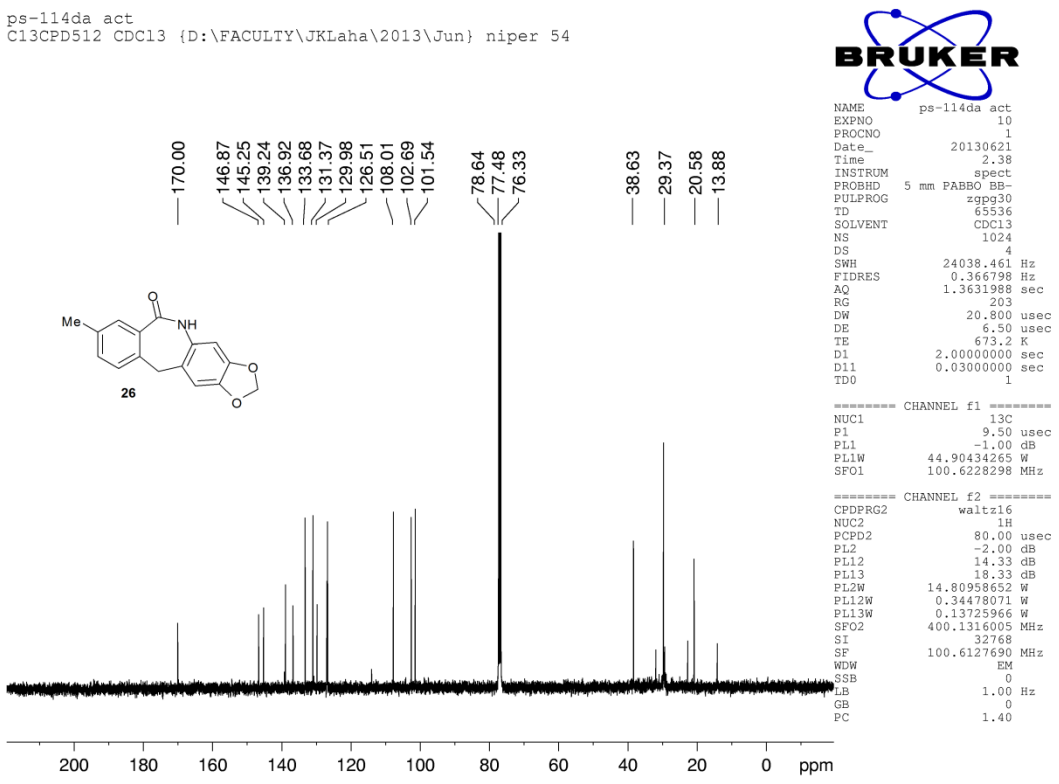

ps-109e
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Mar} niper 26
24
NAME ps-109e
EXPNO 10
Date_ 20130321
Time 13.33
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

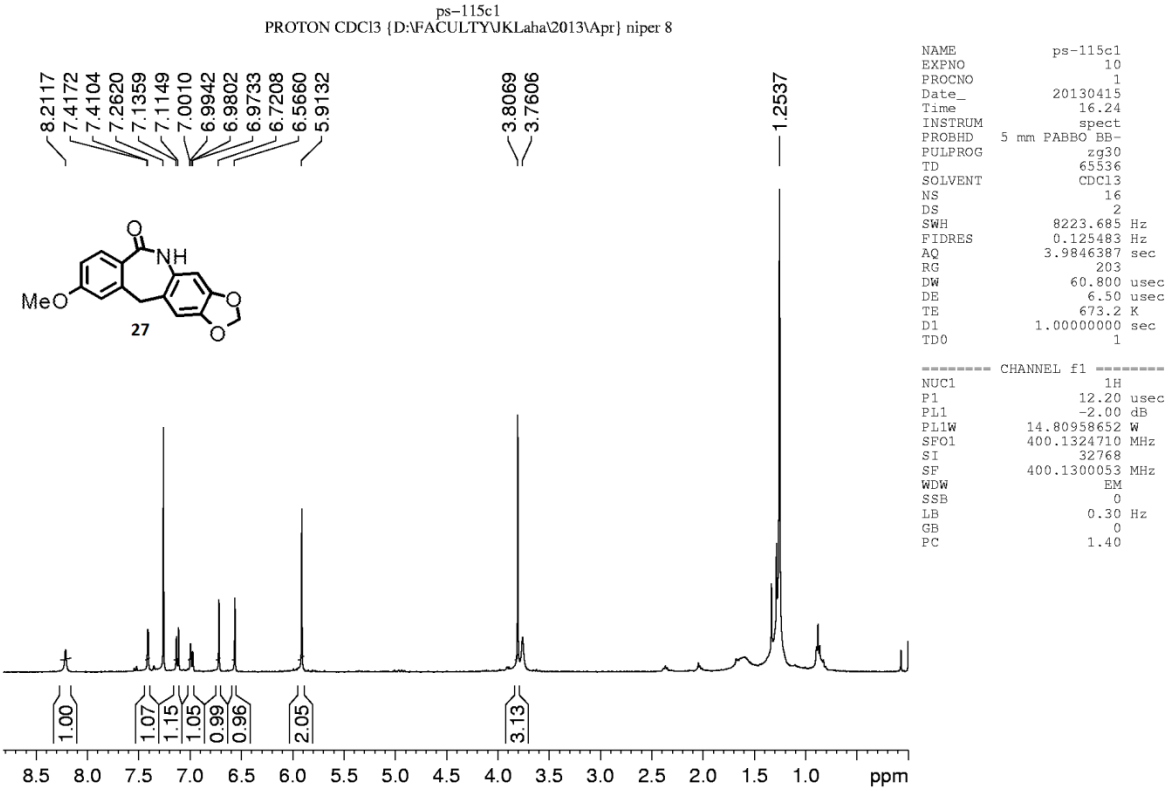

ps-122cc
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Jun} niper 2
24
NAME ps-122cc
EXPNO 11
PROCNO 1
Date_ 20130621
Time 15.35
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

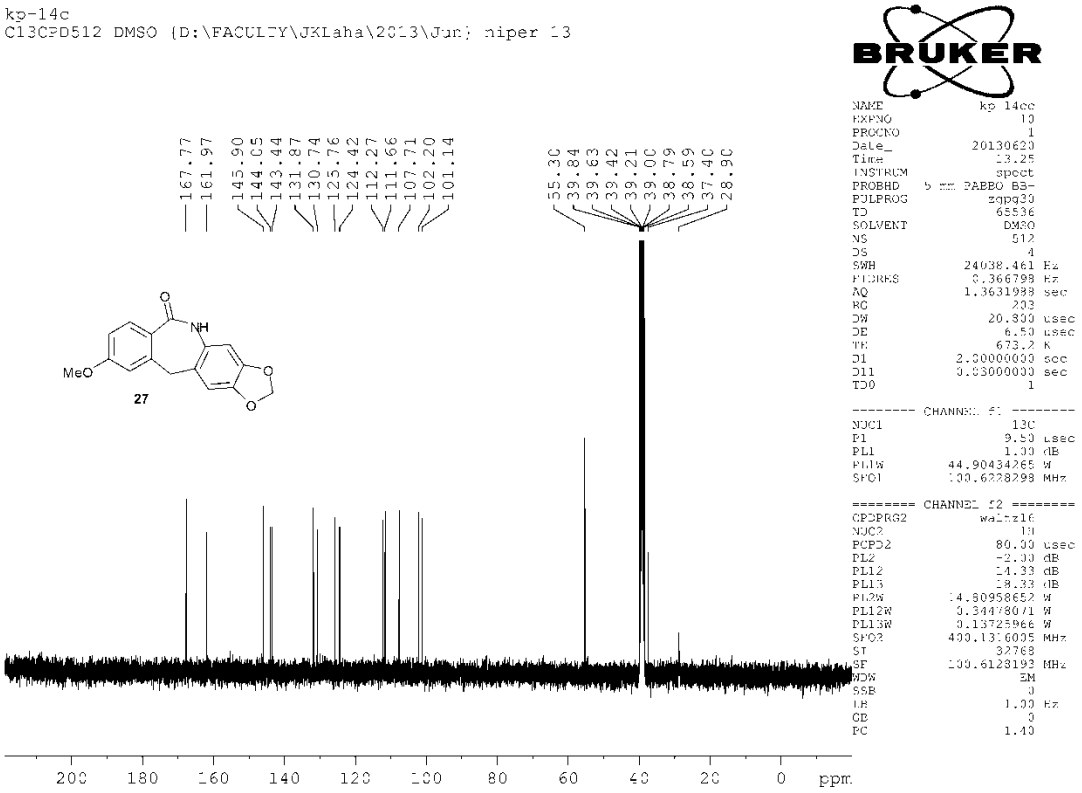

ps-115b1
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 7
8.1375
7.9210
7.9017
7.4744
7.4712
7.4557
7.4526
7.4369
7.4339
7.3513
7.3325
7.3136
7.2751
7.2459
7.2272
6.7538
6.5869
5.9293
3.8406
1.5963
NH
O
O
O
25
1.00
1.05
1.08
1.04
1.01
0.96
1.00
1.97
2.15
9
8
7
6
5
4
3
2
1
0
ppm
NAME ps-115b1
EXPNO 10
PROCNO 1
Date_ 20130415
Time 13.57
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

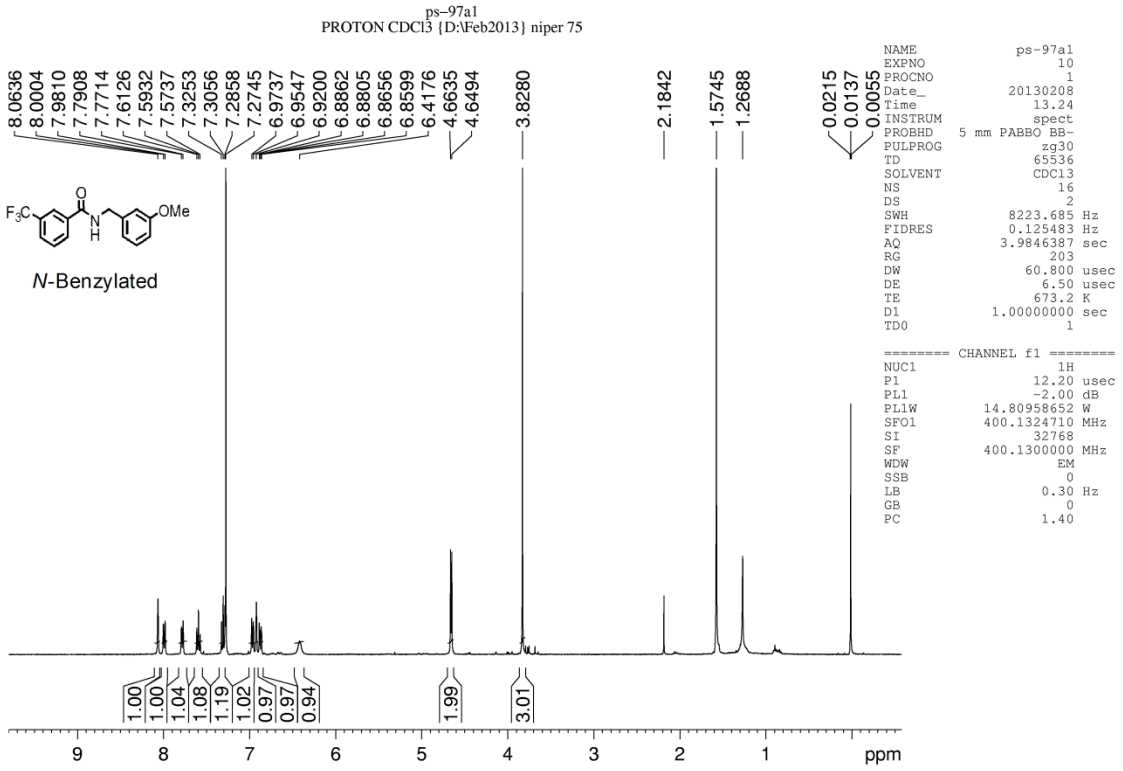

ps-115b1
C13CPD CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 7
169.46
146.80
145.40
141.68
132.56
131.27
130.77
127.07
126.86
126.75
107.90
102.45
101.53
77.34
77.22
77.02
76.70
38.80
NH
O
O
O
25
200
180
160
140
120
100
80
60
40
20
0
ppm
NAME ps-115b1
EXPNO 11
PROCNO 1
Date_ 20130416
Time 23.16
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 use
DE 6.50 use
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 use
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 use
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

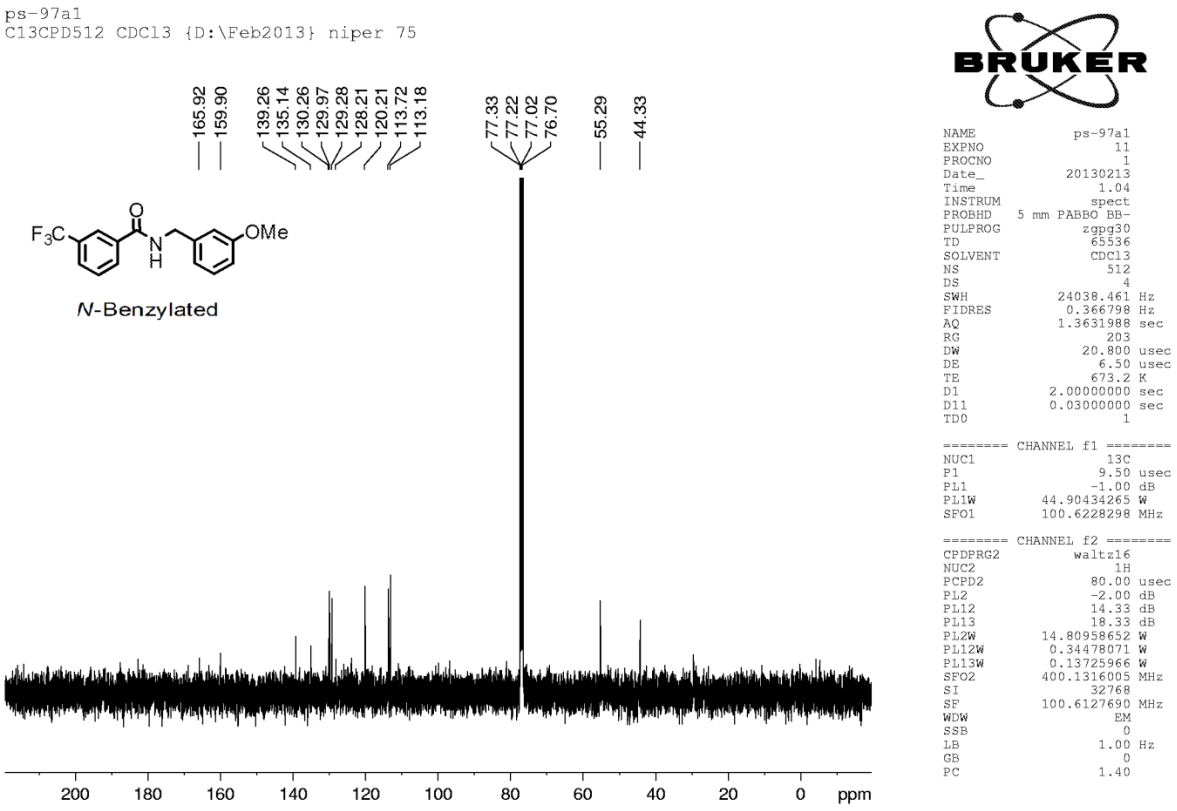

ps-114da
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 56
8.0667
7.7177
7.2747
7.2480
7.1310
7.1117
6.7378
6.5723
5.9211
3.7973
2.3489
1.5993
1.2736
Me
NH
O
O
O
26
0.98
1.00
1.41
1.02
1.01
1.00
2.03
2.15
3.01
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
NAME ps-114da
EXPNO 10
PROCNO 1
Date_ 20130411
Time 10.59
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 293.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

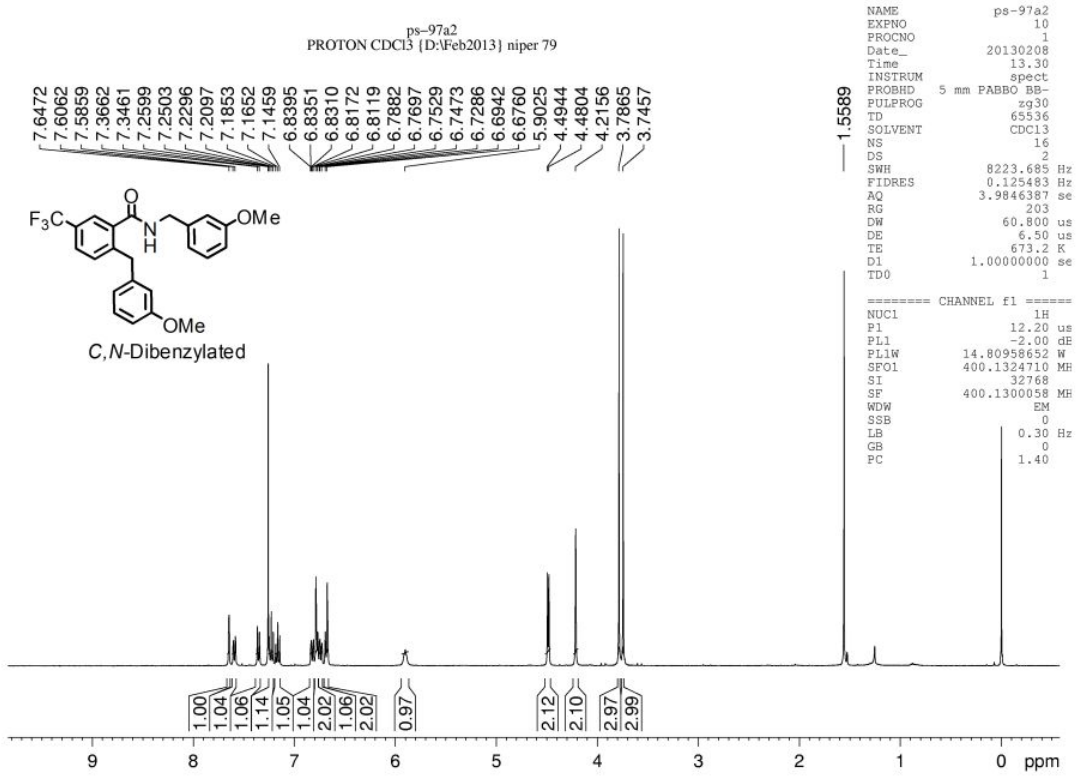

ps-114da act
C13CPD512 CDCl3 {D:\FACULTY\JKLaha\2013\Jun} niper 54
BRUKER
170.00
146.87
145.25
139.24
136.92
133.68
131.37
129.98
126.51
108.01
102.69
101.54
78.64
77.48
76.33
38.63
29.37
20.58
13.88
Me
NH
O
O
O
26
200 180 160 140 120 100 80 60 40 20 0 ppm
NAME ps-114da act
EXPNO 10
PROCNO 1
Date_ 20130621
Time 2.38
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

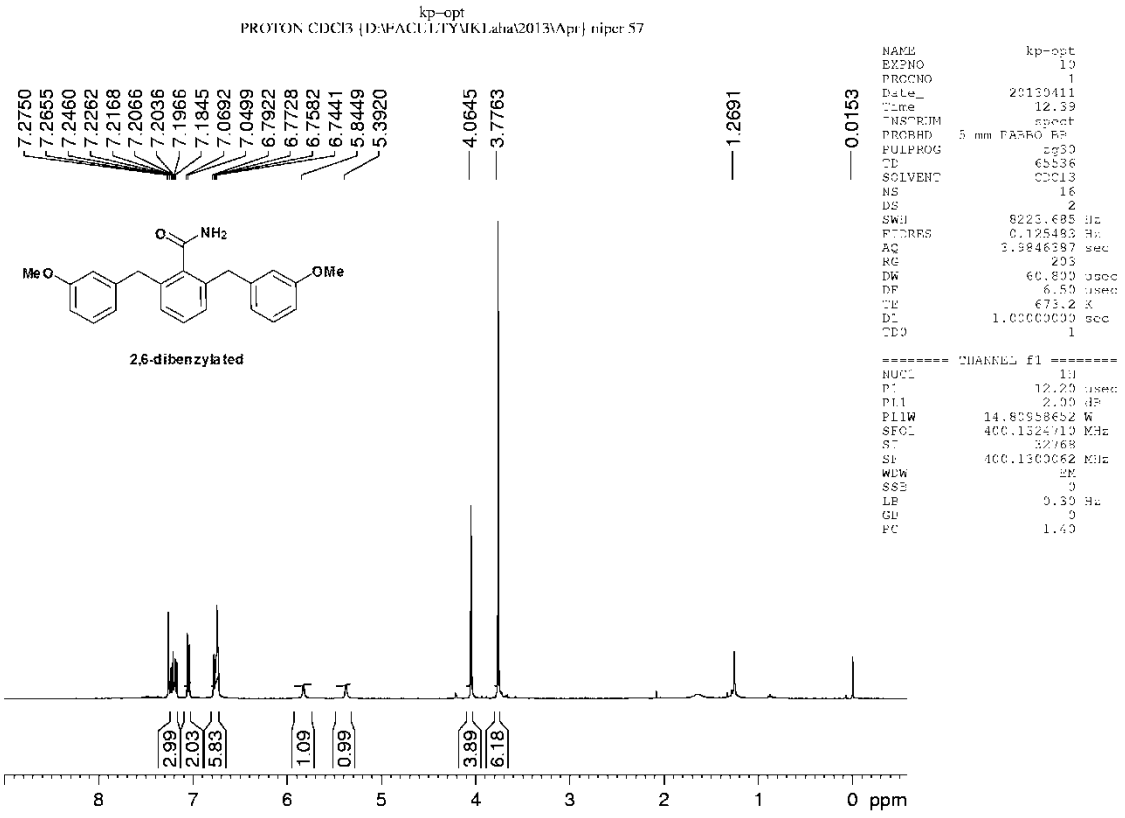

ps-115c1
PROTON CDCl3 {D:\FACULTY\JKLaha\2013\Apr} niper 8
8.2117
7.4172
7.4104
7.2620
7.1359
7.1149
7.0010
6.9942
6.9802
6.9733
6.7208
6.5660
5.9132
3.8069
3.7606
1.2537
NH
MeO
O
O
27
1.00
1.07
1.15
1.05
0.99
0.96
2.05
3.13
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
NAME ps-115c1
EXPNO 10
PROCNO 1
Date_ 20130415
Time 16.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300053 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


kp-14c
C13CPD512 DMSO {D:\FACULTY\JKLaha\2013\Jun} niper 13
167.77
161.97
145.90
144.05
143.44
131.87
130.74
125.76
124.42
112.27
111.66
107.71
102.20
101.14
55.30
39.84
39.63
39.42
39.21
39.00
38.79
38.59
37.40
28.90
NH
MeO
O
O
27
200 180 160 140 120 100 80 60 40 20 0 ppm
BRUKER
NAME kp 14cc
EXPNO 10
PROCNO 1
Date_ 20130620
Time 13.25
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6128193 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

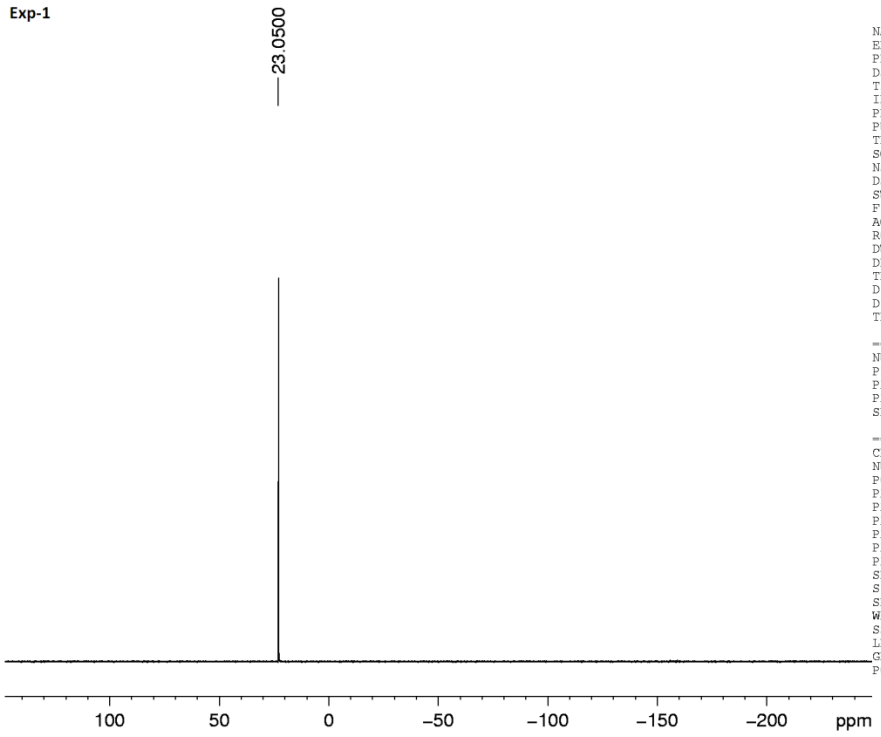

ps–97a1
PROTON CDCl3 {D:\Feb2013} niper 75
8.0636
8.0004
7.9810
7.7908
7.7714
7.6126
7.5932
7.5737
7.3253
7.3056
7.2858
7.2745
6.9737
6.9547
6.9200
6.8862
6.8805
6.8656
6.8599
6.4176
4.6635
4.6494
3.8280
2.1842
1.5745
1.2688
0.0215
0.0137
0.0055
F3C
O
N
H
OMe
N-Benzylated
1.00
1.00
1.04
1.08
1.19
1.02
0.97
0.97
0.94
1.99
3.01
9
8
7
6
5
4
3
2
1
ppm
NAME ps-97a1
EXPNO 10
PROCNO 1
Date_ 20130208
Time 13.24
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40


ps–97a1
C13CPD512 CDCl3 {D:\Feb2013} niper 75
165.92
159.90
139.26
135.14
130.26
129.97
129.28
128.21
120.21
113.72
113.18
77.33
77.22
77.02
76.70
55.29
44.33
F3C
O
N
H
OMe
N-Benzylated
200
180
160
140
120
100
80
60
40
20
0
ppm
BRUKER
NAME ps-97a1
EXPNO 11
PROCNO 1
Date_ 20130213
Time 1.04
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 203
DW 20.800 usec
DE 6.50 usec
TE 673.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 9.50 usec
PL1 -1.00 dB
PL1W 44.90434265 W
SFO1 100.6228298 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -2.00 dB
PL12 14.33 dB
PL13 18.33 dB
PL2W 14.80958652 W
PL12W 0.34478071 W
PL13W 0.13725966 W
SFO2 400.1316005 MHz
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


ps–97a2
PROTON CDCl3 {D:\Feb2013} niper 79
7.6472
7.6062
7.5859
7.3662
7.3461
7.2599
7.2503
7.2296
7.2097
7.1853
7.1652
7.1459
6.8395
6.8351
6.8310
6.8172
6.8119
6.7882
6.7697
6.7529
6.7473
6.7286
6.6942
6.6760
5.9025
4.4944
4.4804
4.2156
3.7865
3.7457
1.5589
F3C
O
N
H
OMe
OMe
C,N-Dibenzylated
1.00
1.04
1.06
1.14
1.05
1.04
2.02
1.06
2.02
0.97
2.12
2.10
2.97
2.99
9
8
7
6
5
4
3
2
1
0 ppm
NAME ps-97a2
EXPNO 10
PROCNO 1
Date_ 20130208
Time 13.30
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 se
RG 203
DW 60.800 us
DE 6.50 us
TE 673.2 K
D1 1.00000000 se
TD0 1
======== CHANNEL f1 ======
NUC1 1H
P1 12.20 us
PL1 -2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MH
SI 32768
SF 400.1300058 MH
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

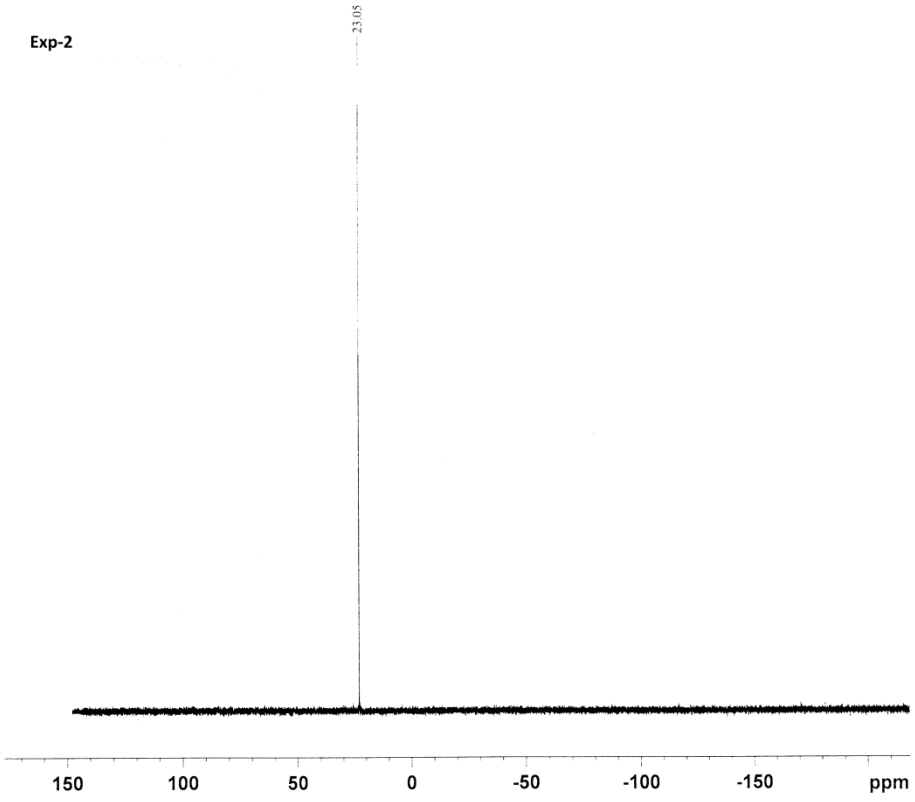

kp–opt
PROTON CDCl3 {D:\PACULTY\KLaha\2013\Apr} niper 57
7.2750
7.2655
7.2460
7.2262
7.2168
7.2066
7.2036
7.1966
7.1845
7.0692
7.0499
6.7922
6.7728
6.7582
6.7441
5.8449
5.3920
4.0645
3.7763
1.2691
0.0153
O
NH2
MeO
OMe
2,6-dibenzylated
2.99
2.03
5.83
1.09
0.99
3.89
6.18
8
7
6
5
4
3
2
1
0 ppm
NAME kp-opt
EXPNO 10
PROCNO 1
Date_ 20130411
Time 12.39
INSTRUM spect
PROBHD 5 mm PABBO BB
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 203
DW 60.800 usec
DE 6.50 usec
TE 673.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 12.20 usec
PL1 2.00 dB
PL1W 14.80958652 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300062 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.40

jkp-19a
P31CPD DMSO {D:\FACULTY\JKLaha\2013\Jul} niper 35




```
NAME                jkp-19a
EXPNO                    10
PROCNO                    1
Date_              20130705
Time                   2.02
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                       512
DS                         4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ                0.5112308 sec
RG                       203
DW                   7.800 usec
DE                   6.50 usec
TE                   673.2 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     31P
P1                   12.68 usec
PL1                   3.00 dB
PL1W            12.96693134 W
SFO1           161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 14.33 dB
PL13                 18.33 dB
PL2W            14.80958652 W
PL12W            0.34478071 W
PL13W            0.13725966 W
SFO2           400.1316005 MHz
SI                    32768
SF             161.9755930 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

jkp-19b
P31CPD DMSO {D:\FACULTY\JKLaha\2013\Jul} niper 36




```
NAME                jkp-19b
EXPNO                    11
PROCNO                    1
Date_              20130705
Time                  12.45
INSTRUM               spect
PROBHD    5 mm PABBO BB-
PULPROG             zgpg30
TD                   65536
SOLVENT                DMSO
NS                       16
DS                         4
SWH              64102.563 Hz
FIDRES            0.978127 Hz
AQ                0.5112308 sec
RG                       203
DW                   7.800 usec
DE                   6.50 usec
TE                   673.2 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                     31P
P1                   12.68 usec
PL1                   3.00 dB
PL1W            12.96693134 W
SFO1           161.9674942 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -2.00 dB
PL12                 14.33 dB
PL13                 18.33 dB
PL2W            14.80958652 W
PL12W            0.34478071 W
PL13W            0.13725966 W
SFO2           400.1316005 MHz
SI                    32768
SF             161.9755930 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```