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SUPPLEMENTARY INFORMATION

Palladium-Catalyzed Decarboxylative, Decarbonylative and Dehydrogenative C(sp^2)-H Acylation at *Room Temperature*

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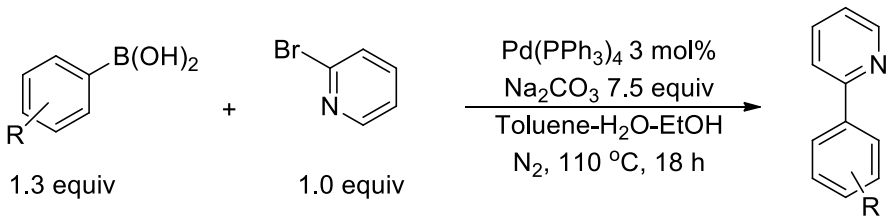
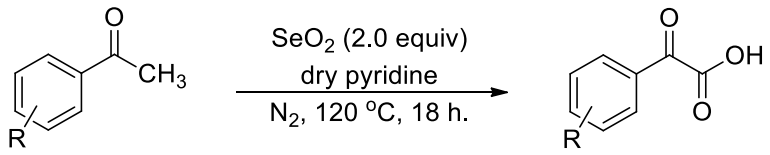
General experimental procedure for the preparation of 2-phenylpyridines.[1]



To a solution of arylboronic acid (2.6 mmol, 1.3 equiv) in toluene (7.0 mL), ethanol (1.5 mL) and H_2O (7.0 mL) Na_2CO_3 (1.6 g, 15 mmol, 7.5 equiv) was added followed by $Pd(PPh_3)_4$ (69 mg, 0.060 mmol) and 2-bromopyridine (0.2 mL, 2.0 mmol, 1.0 equiv) were added in a 50 mL round bottom flask. The reaction mixture was evacuated and refilled with nitrogen three times and stirred at 110 ^oC for 18 h. After the completion of the reaction as indicated by TLC, the reaction mixture was cooled to room temperature. To the reaction mixture, aqueous NH_4CL (15 mL) was added, extracted with ethyl acetate for three times, dried over Na_2SO_4 and evaporated in vacuum to afford the crude product. The crude product was purified by column chromatography on silica gel with using hexane/EtOAc (9/1) to afford pure 2-arylpyridines.

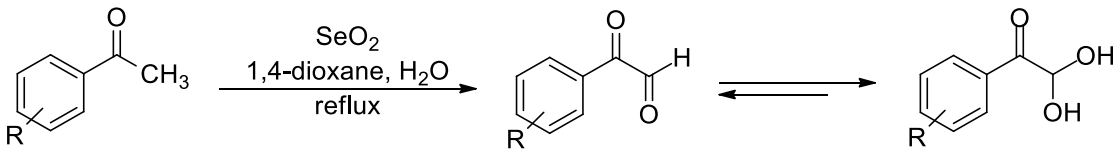
General Experimental procedure for the preparation of arylglyoxylic acids.[2]



A mixture of acetophenones (8.0 mmol, 1.0 equiv) and selenium dioxide (1.8 g, 16 mmol, 2.0 equiv) in dry pyridine (4.0 mL) was stirred at 120 ^oC under N_2 for 18 h. After completion of the reaction, as determined by TLC, the solution containing precipitated selenium was filtered using a Buckner funnel and the residue was washed with ethyl acetate (40 mL). The combined filtrate was treated with 1N HCl (60 mL), the organic layer was separated, and the remaining aqueous layer was extracted with ethyl acetate (2x20). The organic layers were combined and treated with

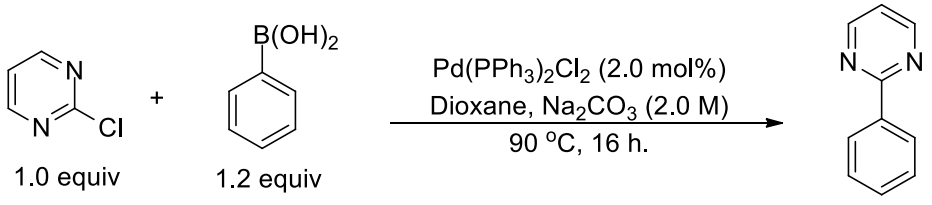
1N NaOH (2x50), and the aqueous layer was separated. Then the aqueous layer was acidified using 1N HCl to about pH 1.5. The mixture was extracted with ethyl acetate (3x40), and the combined organic layers were dried over Na_2SO_4 and evaporated in vacuum to afford the crude product. The crude product was purified by column chromatography on silica gel with using hexane/EtOAc (7/3) to give the pure corresponding arylglyoxylic acids.

General experimental procedure for the preparation of phenylglyoxals.[3]



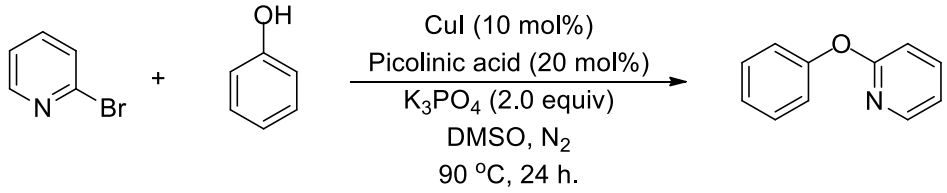
To a 50 mL two-neck round bottom flask fitted with a condenser, was added 1,4-dioxane (13.0 mL), SeO_2 (2.8 g, 25.0 mmol) and water (0.5 mL). The mixture was heated at 50-55 ^oC and stirred under N_2 atmosphere until the solid was dissolved, then the corresponding acetophenone (25.0 mmol, 1.0 equiv) was added and the reaction mixture was stirred at 110 ^oC for 4 h. Then the reaction mixture was cooled to room temperature. The solid was removed by filtration; the filtrate was evaporated to afford a crude product. The crude product was purified by silica gel column chromatography (hexane:ethyl acetate/8:2) to give the product as a yellow liquid. This liquid was dissolved in hot water (10 mL) and allowed to crystallize to afford the desired products, arylglyoxal monohydrate as a white solid.

Experimental procedure for the preparation of 2-phenylpyrimidine.[4]



To a round-bottom flask was added 2-chloropyrimidine (229.0 mg, 2.0 mmol, 1.0 equiv), phenylboronic acid (293mg, 2.4 mmol, 1.2 equiv), $Pd(PPh_3)_2Cl_2$ (28mg, 0.04 mmol, 0.02 equiv) and Na_2CO_3 (2.0 M, 5.0 mL) in dioxane (5.0 mL). The reaction mixture was heated to 90 ^oC for 16 h under N_2 atmosphere. After completion of the reaction (as indicated by TLC), the heterogeneous aqueous was concentrated under reduced pressure and the residue was extracted with EtOAc (30 mL), washed by H_2O (30 mL) and brine (30 mL). The organic layer was dried over Na_2SO_4, concentrated and purified by column chromatography on silica gel using ethyl acetate/hexane as eluent to afford the desired product, 2-phenylpyrimidine as colorless oil. ^1H NMR (300 MHz, $CDCl_3$): δ 8.81 (d, *J* = 4.8 Hz, 2H), 8.43-8.46 (m, 2H), 7.48-7.52 (m, 3H), 7.19 (t, *J* = 4.8 Hz, 1H).

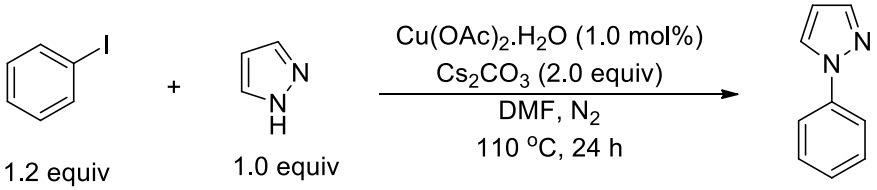
Experimental procedure for the preparation of 2-phenoxypyridine.[5]



To an oven-dried 50 mL round bottomed flask, CuI (122.0 mg, 0.64 mmol, 0.1 equiv), picolinic acid (158 mg, 1.3 mmol, 0.2 equiv), phenol (715 mg, 7.6 mmol, 1.2 equiv) and K_3PO_4 (2.7 g, 12.8 mmol, 2.0 equiv) was added then the flask was evacuated and back-filled with N_2. To this reaction mixture, 2-bromopyridine (0.6 mL, 6.4 mmol, 1.0 equiv) and DMSO (12 mL) was added via syringe. The reaction mixture was stirred at 90 ^oC for 24 h under N_2. Then the reaction mixture was cooled to room temperature. The mixture was poured into water (30 mL) and extracted with ethyl acetate (30 mL). The organic layer was washed with water (20x2 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced

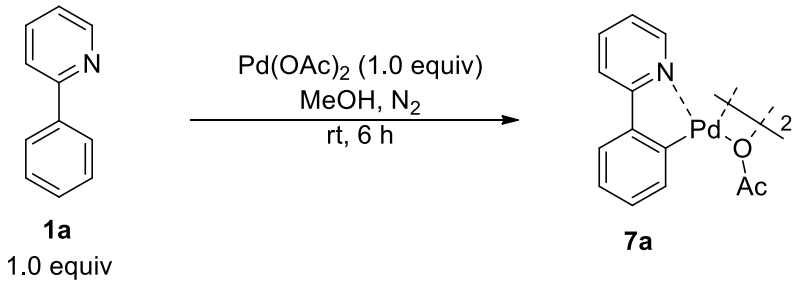
pressure. The crude product was purified by silica gel column chromatography using ethyl acetate/hexane as eluent to afford the desired product, 2-phenoxypyridine as a white solid. ^{1}H NMR (300 MHz, $CDCl_3$): δ 8.21 (dd, *J* = 4.8 Hz, 1.2 Hz, 1H), 7.65-7.71 (m, 1H), 7.41 (t, *J* = 7.8 Hz, 2H), 7.22 (d, *J* = 7.5 Hz, 1H), 7.15 (d, *J* = 7.8 Hz, 2H), 6.99 (dd, *J* = 5.1 Hz, 5.4 Hz, 1H), 6.91 (d, *J* = 8.4 Hz, 1H).

Experimental procedure for the preparation of 1-phenyl-1*H*-pyrazole.[6]



To a solution of $Cu(OAc)_2.H_2O$ (6.0 mg, 0.03 mmol, 0.01 equiv) in DMF (6.0 mL) were added iodobenzene (0.4 mL, 3.6 mmol, 1.2 equiv), 1*H*-pyrazole (204 mg, 3.0 mmol, 1.0 equiv), and Cs_2CO_3 (2.0 g, 6.0 mmol, 2.0 equiv) under N_2. The mixture was stirred at 110 ^{o}C for 24 h. Then the reaction mixture was cooled to room temperature. The mixture was poured into water (20 mL) and extracted with ethyl acetate (30 mL). The organic layer was washed with water (10x2 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by silica gel column chromatography using ethyl acetate/hexane as eluent to afford the desired product, 1-phenyl-1*H*-pyrazole as colorless oil. ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.94 (d, *J* = 2.1 Hz, 1H), 7.69-7.74 (m, 3H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.2 Hz, 1H), 6.48 (t, *J* = 1.8 Hz, 1H).

Experimental procedure for the preparation of 2-phenylpyridine palladacyclo dimer complex (7a).[7]

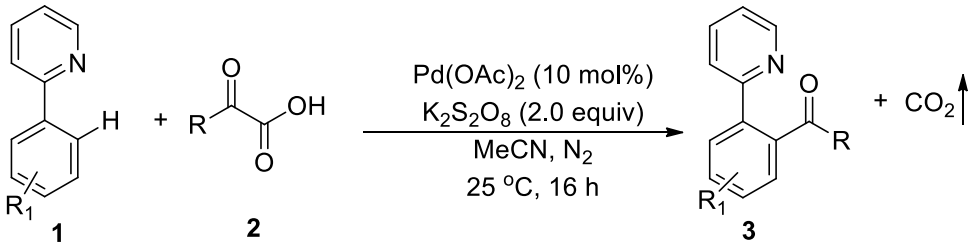


1a
1.0 equiv

7a

To an oven-dried 50 mL round bottomed flask, 2-Phenylpyridine (155 mg, 1.0 mmol, 1.0 equiv) was added to a solution of $Pd(OAc)_2$ (225 mg, 1.0 mol, 1.0 equiv) in MeOH (16 mL) and stirred at room temperature for 6 h under N_2, during which time a yellow solid precipitated was formed in the solution. The precipitate was collected at the top of a plug of celite, and the solids were washed with hexanes (3 x 10 mL). The yellow residue at the top of the celite plug was then washed through with CH_2Cl_2 (2 x 30 mL). The solvent was evaporated under reduced pressure, and the resulting solid was recrystallized from CH_2Cl_2/hexanes to afford the desired product as a yellow solid (82% yield). ^{1}H NMR (300 MHz, $CDCl_3$): δ 7.87 (d, *J* = 5.4 Hz, 2H), 7.38 (t, *J* = 7.8 Hz, 2H), 7.09 (d, *J* = 8.1 Hz, 2H), 6.79-6.94 (m, 8H), 6.45 (t, *J* = 6.6 Hz, 2H), 2.29 (s, 6H).

General experimental procedure for the decarboxylative acylation reaction between 2-phenylpyridines and α-ketocarboxylic acids, Scheme 2.



1
1.0 equiv

2
1.5-2.0 equiv

3

To an oven-dried 10 mL sealed tube, a mixture of 2-phenylpyridines (0.2 mmol, 1.0 equiv), α-ketocarboxylic acids (0.3-0.4 mmol, 1.5-2.0 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II)acetate (4.6 mg, 0.02 mmol, 0.1 equiv) was taken and dry MeCN (3.0 mL) was added to it. After flushing with nitrogen for 30 seconds, immediately the vessel was sealed with a screw cap. The reaction mixture was allowed to stir for 16 h at room temperature. After that the reaction mixture was quenched with $NaHCO_3$ to remove the unreacted acids. Then the reaction mixture was poured into water (40 mL) and extracted with ethyl acetate (40 mL). The organic layer was washed with water (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate/hexane as eluent to afford the desired product.

To note: During optimization, it was found that α-ketocarboxylic acids are hygroscopic in nature thus water or moisture is detrimental to the reaction outcome. Therefore, so after flushing with nitrogen the reaction vessel was immediately sealed with a screw cap.

SPECTRAL DATA

[1,1'-Biphenyl]-4-yl(2-(pyridin-2-yl)phenyl)methanone, 3f, Scheme 2.[8] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-([1,1'-biphenyl]-4-yl)-2-oxoacetic acid (68 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a light yellow oil, (55.0 mg, 82%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.41-8.42 (m, 1H), 7.78-7.82 (m, 3H), 7.64 (td, *J* = 7.2 Hz, 1.2 Hz, 1H),7.52-7.61 (m, 8H), 7.45 (t, *J* = 7.2, Hz, 2H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.03-7.05 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 197.8, 156.8, 149.1, 144.9, 139.9, 139.6, 139.5, 136.6, 136.3, 130.2, 130.1, 129.0, 128.84, 128.82, 128.5, 128.0, 127.2,

126.7, 122.7, 122.0; IR (neat): υ_{max} 1662, 1595, 1280, 926, 745 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{24}H_{17}NO$ $[M]^+$: 335.1310; found: 335.1314.

(4-Fluorophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3g, Scheme 2.[9] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(4-fluorophenyl)-2-oxoacetic acid (51.5 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (44.0 mg, 80%). ^1H NMR (600 MHz, $CDCl_3$): δ 8.38 (d, *J* = 4.8 Hz, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.70-7.73 (m, 2H), 7.59-7.65 (m, 2H), 7.54 (d, *J* = 4.2 Hz, 2H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.04-7.06 (m, 1H), 6.92-6.96 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 196.7, 165.1 (d, *J* = 252.0 Hz), 156.6, 149.0, 139.4, 139.2, 136.4, 134.3 (d, *J* = 3.0 Hz), 131.9 (d, *J* = 9.0 Hz), 130.3, 129.0, 128.7, 128.6, 122.6, 122.0, 115.1 (d, *J* = 21.0 Hz); IR (neat): υ_{max} 1666, 1594, 1280, 1232, 1151, 752 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{12}FNONa$ $[M + Na]^+$: 300.0801; found: 300.0806.

(4-Chlorophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3h, Scheme 2.[10] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(4-chlorophenyl)-2-oxoacetic acid (55.5 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (37.5 mg, 64%). ^1H NMR (600 MHz, $CDCl_3$): δ 8.35-8.36 (m, 1H), 7.79 (d, *J* = 7.8 Hz, 1H), 7.60-7.64 (m, 4H), 7.52-7.56 (m, 3H), 7.24-7.28 (m, 2H), 7.05-7.07 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 197.0, 156.4, 149.0, 139.4, 139.1, 138.6, 136.45, 136.41, 130.7,

130.3, 129.0, 128.6, 128.3, 122.4, 122.1; IR (neat): υ_{max} 2925, 1669, 1587, 1280, 1089, 928, 750 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{12}ClNONa$ $[M + Na]^+$: 316.0505; found: 316.0503.

(4-Bromophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3i, Scheme 2.[9] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(4-bromophenyl)-2-oxoacetic acid (69 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish solid, (54.5 mg, 81%), mp 102-104 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.35-8.36 (m, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.60-7.64 (m, 2H), 7.52-7.57 (m, 5H), 7.40-7.42 (m, 2H), 7.05-7.07 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ 196.9, 156.4, 148.9, 139.4, 139.0, 136.8, 136.5, 131.3, 130.8, 130.3, 129.0, 128.64, 128.58, 127.2, 122.4, 122.1; IR (neat): υ_{max} 1670, 1584, 1279, 1067, 927, 750 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{12}BrNONa$ $[M + Na]^+$: 360.0000; found: 360.0002.

(2-(Pyridin-2-yl)phenyl)(4-(trifluoromethyl)phenyl)methanone, 3j, Scheme 2.[8] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-(4-(trifluoromethyl)phenyl)acetic acid (65.5 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (37.0 mg, 57%), mp 76-78 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.30-8.31 (m, 1H), 7.78-7.81 (m, 3H), 7.54-7.66 (m, 5H), 7.53 (d, *J* = 7.8 Hz, 2H), 7.02-7.04 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 196.9, 156.0, 148.8, 141.0, 139.4, 138.8, 136.6, 133.2 (q, *J* = 31.5 Hz), 130.5, 129.3, 129.1, 128.8, 128.4, 125.0 (q, *J* = 3.0 Hz), 123.6 (q, *J* = 271.5 Hz),

122.2, 122.1; IR (neat): υ_{max} 1675, 1587, 1322, 1130, 753 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{19}H_{12}F_3NO$ $[M]^+$: 327.0871; found: 327.0867.

Naphthalen-1-yl(2-(pyridin-2-yl)phenyl)methanone, 3k, Scheme 2.[9] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(naphthalen-1-yl)-2-oxoacetic acid (60 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish solid, (34.5 mg, 56%), mp 101-103 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.93 (d, *J* = 8.4 Hz, 1H), 8.14 (d, *J* = 4.8 Hz, 1H), 7.75-7.78 (m, 3H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.63-7.66 (m, 2H), 7.56 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.52 (t, *J* = 7.2 Hz, 1H), 7.35-7.43 (m, 3H), 7.17 (t, *J* = 7.8 Hz, 1H), 6.76-6.78 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 199.6, 157.0, 148.9, 141.0, 140.2, 136.3, 136.0, 133.5, 132.3, 131.0, 130.6, 130.1, 129.6, 128.8, 128.5, 128.0, 127.6, 126.5, 126.2, 123.7, 122.2, 121.4; IR (neat): υ_{max} 1662, 1586, 1432, 1279, 750 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{22}H_{15}NO$ $[M]^+$: 309.1154; found: 309.1133.

(2-Bromophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3l, Scheme 2.[11] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(2-bromophenyl)-2-oxoacetic acid (69 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (54.0 mg, 80%). ^1H NMR (600 MHz, $CDCl_3$): δ 8.52-8.54 (m, 1H), 7.70-7.71 (m, 1H), 7.61-7.66 (m, 2H), 7.57 (td, *J* = 7.8 Hz, 1.8 Hz, 1H), 7.53 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.44-7.46 (m, 2H), 7.23-7.26 (m, 1H), 7.06-7.09 (m, 2H), 7.01-7.03 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 196.7, 157.1, 149.1, 140.7, 139.4, 138.7, 136.2, 139.9, 131.6, 131.3, 131.2, 130.6,

129.3, 128.6, 126.5, 122.8, 121.8, 121.4; IR (neat): υ_{max} 1673, 1586, 1464, 1432, 1292, 928, 750 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{18}H_{12}BrNO$ $[M]^+$: 337.0102; found: 337.0101.

(5-Methyl-2-(pyridin-2-yl)phenyl)(phenyl)methanone, 3o, Scheme 2.[12] The same general procedure was followed by using 2-(*p*-tolyl)pyridine (34.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetic acid (60 mg, 0.4 mmol, 2.0 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (40.0 mg, 73%), mp 137-139 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.35 (d, *J* = 4.8 Hz, 1H), 7.68-7.70 (m, 3 H), 7.55 (td, *J* = 7.8 Hz, 1.8 Hz, 1H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 7.8 Hz, 1H), 7.36-7.40 (m, 2H), 7.26-7.28 (m, 2H), 6.98-7.00 (m, 1H), 2.47 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.4, 156.6, 148.9, 139.4, 138.6, 137.9, 136.8, 136.2, 132.2, 130.9, 129.6, 129.4, 128.5, 127.9, 122.4, 121.6, 21.2; IR (neat): υ_{max} 1666, 1588, 1461, 1250, 698 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{19}H_{15}NONa$ $[M + Na]^+$: 296.1051; found: 296.1051.

(5-(*tert*-Butyl)-2-(pyridin-2-yl)phenyl)(phenyl)methanone, 3p, Scheme 2.[13] The same general procedure was followed by using 2-(4-(*tert*-butyl)phenyl)pyridine (42.5 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetic acid (60 mg, 0.4 mmol, 2.0 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (38.0 mg, 60%). ^1H NMR (600 MHz, $CDCl_3$): δ 8.35 (d, *J* = 4.2 Hz, 1H), 7.70-7.73 (m, 3H), 7.64 (dd, *J* = 7.8 Hz, 1.8 Hz, 1H), 7.53-7.56 (m, 2H), 7.48 (d, *J* = 7.8 Hz, 1H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.26-7.28 (m, 2H), 6.98-7.00 (m, 1H), 1.39 (s, 9H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.7, 156.7, 151.8, 148.9, 139.0, 138.0, 136.8, 136.2, 132.2, 129.4, 128.4, 127.9,

127.3, 126.0, 122.4, 121.6, 34.8, 31.2; IR (neat): υ_{max} 2962, 1667, 1590, 1467, 1249, 751 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{22}H_{21}NONa$ $[M + Na]^+$: 338.1521; found: 338.1521.

(4-Methyl-2-(pyridin-2-yl)phenyl)(phenyl)methanone, 3q, Scheme 2.[9] The same general procedure was followed by using 2-(*m*-tolyl)pyridine (34.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetic acid (60 mg, 0.4 mmol, 2.0 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (38.0 mg, 70%), mp 106-108 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.40 (d, *J* = 4.2 Hz, 1H), 7.68 (d, *J* = 7.2 Hz, 2H), 7.58 (s, 1H), 7.52-7.55 (m, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.38 (t, *J* = 7.2 Hz, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.24-7.28 (m, 2H), 7.00-7.02 (m, 1H), 2.51 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.2, 157.1, 149.0, 140.6, 139.9, 138.0, 136.6, 136.1, 132.2, 129.6, 129.5, 129.4, 129.1, 127.9, 123.0, 121.8, 21.5; IR (neat): υ_{max} 1665, 1588, 1281, 932, 702 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{19}H_{15}NONa$ $[M + Na]^+$: 296.1051; found: 296.1050.

(2-(Pyridin-2-yl)phenyl)(*m*-tolyl)methanone, 3r, Scheme 2.[14] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-(*m*-tolyl)acetic acid (49 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (38.0 mg, 70%). ^1H NMR (600 MHz, $CDCl_3$): δ 8.39 (d, *J* = 4.8 Hz, 1H), 7.77 (d, *J* = 7.8 Hz, 1H), 7.61 (td, *J* = 7.2 Hz, 2.4 Hz, 1H), 7.48-7.58 (m, 6H), 7.22 (d, *J* = 7.2 Hz, 1H), 7.16 (t, *J* = 7.8 Hz, 1H), 7.02-7.04 (m, 1H), 2.29 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.4, 156.9, 149.0, 139.7, 139.6, 137.7, 136.2, 133.2, 130.1, 129.9, 129.1, 128.8,128.4, 127.9, 127.0, 122.7, 121.9, 21.2; IR (neat):

υ_{max} 1666, 1588, 1432, 1284, 752 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{19}H_{15}NO$ $[M]^+$: 273.1154; found: 273.1150.

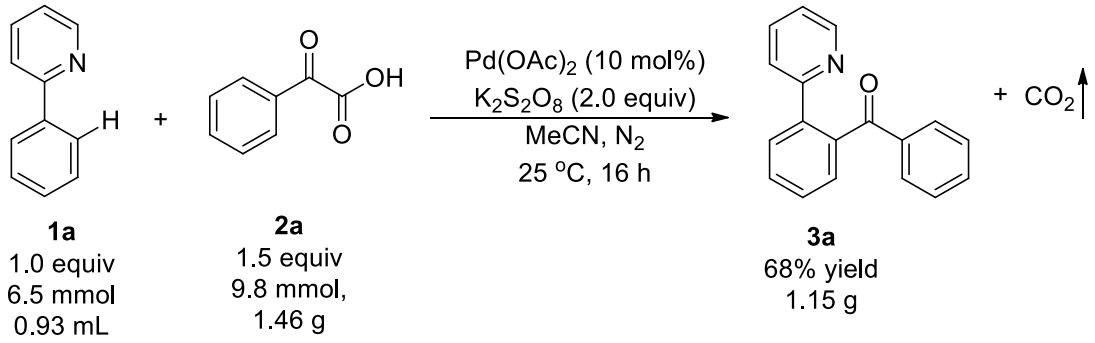
(5-Methoxy-2-(pyridin-2-yl)phenyl)(m-tolyl)methanone, 3s, Scheme 2. The same general procedure was followed by using 2-(4-methoxyphenyl)pyridine (37.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-(m-tolyl)acetic acid (66 mg, 0.4 mmol, 2.0 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (42.5 mg, 70%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.34 (d, *J* = 4.8 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.55 (s, 1H), 7.51-7.54 (m, 1H), 7.48 (d, *J* = 7.2 Hz, 1H), 7.44 (d, *J* = 7.8 Hz, 1H), 7.20 (d, *J* = 7.8 Hz, 1H), 7.12-7.16 (m, 2H), 7.05 (d, *J* = 2.4 Hz, 1H), 6.96-6.98 (m, 1H), 3.88 (s, 3H), 2.28 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.1, 159.7, 156.4, 148.8, 140.8, 137.7, 137.6, 136.1, 133.2, 132.0, 130.0, 129.8, 127.9, 126.8, 122.1, 121.3, 116.0, 114.0, 55.5, 21.2; IR (neat): υ_{max} 1667, 1595, 1464, 1288, 1234, 753 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{20}H_{17}NO_2Na$ $[M + Na]^+$: 326.1157; found: 326.1161.

3-Methyl-1-(2-(pyridin-2-yl)phenyl)butan-1-one, 3w, Scheme 2.[8] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 4-methyl-2-oxopentanoic acid (52 mg, 0.4 mmol, 2.0 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (34.0 mg, 71%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.62 (d, *J* = 4.2 Hz, 1H), 7.76 (td, *J* = 7.8 Hz, 1.8 Hz, 1H), 7.61 (d, *J* = 7.2 Hz, 1H), 7.56 (d, *J* = 8.4 Hz, 1H), 7.44-7.52 (m, 3H), 7.24-7.28 (m, 1H), 2.40 (d, *J* = 6.6 Hz, 2H), 2.09-2.16 (m, 1H), 0.87 (d, *J* = 7.2 Hz, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 206.6, 157.6, 149.2, 142.0, 138.6, 136.7, 130.0, 129.2, 128.5, 127.5, 122.6, 122.2, 51.7, 24.8,

22.6; IR (neat): υ_{max} 2956, 1693, 1587, 1465, 1208, 754 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{16}H_{17}NONa$ $[M + Na]^+$: 262.1208; found: 262.1207.

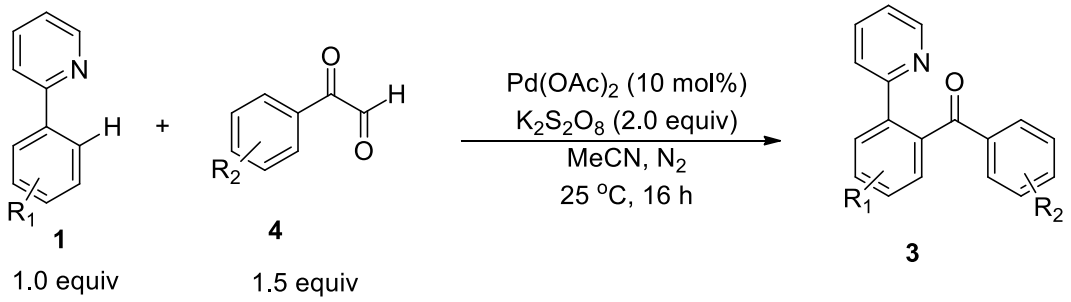
Phenyl(2-(pyrimidin-2-yl)phenyl)methanone, 3x, Scheme 2.[15] The same general procedure was followed by using 2-phenylpyrimidine (31.5 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetic acid (45 mg, 0.4 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (33 mg, 63%), mp 129-131 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.56 (d, *J* = 4.8 Hz, 2H), 8.38 (d, J = 7.2 Hz, 1H), 7.73 (d, *J* = 7.2 Hz, 2H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.61 (t, *J* = 7.8 Hz, 1H), 7.52 (d, *J* = 7.2 Hz, 1H), 7.42 (t, *J* = 7.8 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 2H), 7.00 (t, *J* = 4.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 197.8, 163.9, 156.6, 140.5, 138.1, 136.9, 132.2, 130.3, 129.9, 129.4, 129.0, 128.6, 128.1, 118.7; IR (neat): υ_{max} 1664, 1557, 1413, 1281, 752 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{17}H_{12}N_2ONa$ $[M + Na]^+$: 283.0847; found: 283.0841.

Decarboxylative acylation reaction in gram scale (Synthesis of phenyl(2-(pyridin-2-yl)phenyl)methanone, 3a, Scheme 2).



To an oven-dried 100 mL sealed tube, a mixture of 2-phenylpyridines (0.93 mL, 6.5 mmol, 1.0 equiv), 2-oxo-2-phenylacetic acid (1.46g, 9.8 mmol, 1.5 equiv), potassium persulfate (3.51g, 13.0 mmol, 2.0 equiv) and palladium(II)acetate (146 mg, 0.65 mmol, 0.1 equiv) was taken and dry MeCN (50 mL) was added to it. After flushing with nitrogen for 30 seconds, immediately the vessel was sealed with a screw cap. The reaction mixture was allowed to stir for 16 h at room temperature. After that the reaction mixture was quenched with $NaHCO_3$ to remove the unreacted acids. Then the reaction mixture was poured into water (80 mL) and extracted with ethyl acetate (60x2 mL). The organic layer was washed with water (30x2 mL) and brine (30 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The pure phenyl(2-(pyridin-2-yl)phenyl)methanone (**3a**) was obtained as a white solid in 68% (1.15 g) yield after column chromatography of the crude reaction mixture using ethyl acetate/hexane (7:3) as eluent.

General experimental procedure for the decarbonylative acylation reaction between 2-phenylpyridines and phenylglyoxals, Scheme 3.



To an oven-dried 10 mL sealed tube, a mixture of 2-phenylpyridines (0.2 mmol, 1.0 equiv), phenylglyoxals (0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II)acetate (4.6 mg, 0.02 mmol, 0.1 equiv) was taken and dry MeCN (3.0 mL) was added to it. After flushing with nitrogen, immediately the vessel was sealed with a screw cap. The reaction mixture was allowed to stir for 16 h at room temperature. After that the reaction

mixture was quenched with $NaHCO_3$. Then the reaction mixture was poured into water (40 mL) and extracted with ethyl acetate (40 mL). The organic layer was washed with water (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate/hexane as eluent to afford the desired product.

SPECTRAL DATA

Phenyl(2-(pyridin-2-yl)phenyl)methanone, 3a, Scheme 3. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetaldehyde (40 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (36.5 mg, 70%), mp 105-107 ^oC. The spectral data of compound **3a** is in Scheme 2.

(2-(Pyridin-2-yl)phenyl)(*p*-tolyl)methanone, 3b, Scheme 3. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-(*p*-tolyl)acetaldehyde (45 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (26 mg, 48%), mp 97-99 ^oC. The spectral data of compound **3b** is in Scheme 2.

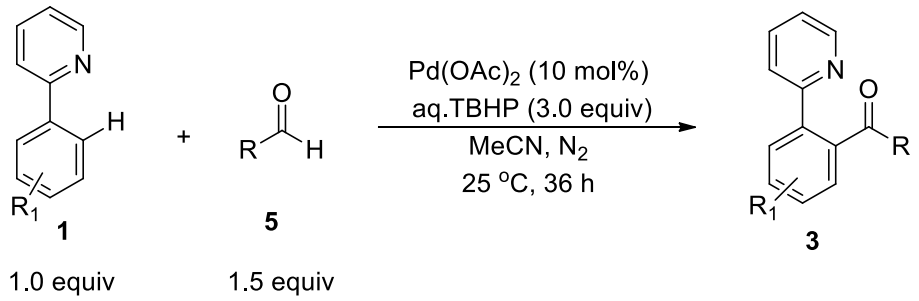
(4-Fluorophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3g, Scheme 3. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(4-fluorophenyl)-2-oxoacetaldehyde (46 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (31.0 mg, 56%). The spectral data of compound **3g** is in Scheme 2.

(4-Chlorophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3h, Scheme 3. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(4-chlorophenyl)-2-oxoacetaldehyde (51.0 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (33.5 mg, 57%). The spectral data of compound **3h** is in Scheme 2.

(2-Bromophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3l, Scheme 3. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-(2-bromophenyl)-2-oxoacetaldehyde (64.0 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (40.5 mg, 60%). The spectral data of compound **3l** is in Scheme 2.

(2-(Pyridin-2-yl)phenyl)(m-tolyl)methanone, 3r, Scheme 3. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-(*m*-tolyl)acetaldehyde (45 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv) and palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil (26.0 mg, 47%). The spectral data of compound **3r** is in Scheme 2.

General experimental procedure for the dehydrogenative acylation reaction between 2-phenylpyridines and aldehydes, Scheme 4.



To an oven-dried 7 mL clear vial, a mixture of 2-phenylpyridines (0.2 mmol, 1.0 equiv) and palladium(II)acetate (4.6 mg, 0.02 mmol, 0.1 equiv) was taken and dry MeCN (3.0 mL) was added to it. The corresponding aldehydes (0.3 mmol 1.5 equiv) were added to the reaction mixture. After flushing with nitrogen for 30 seconds, immediately the vessel was sealed with a screw cap. Then aq. TBHP (82 µL, 0.6 mmol, 3.0 equiv) was added to the reaction mixture via micro-litter syringe. The reaction mixture was allowed to stir for 36 h at room temperature. After completion (as indicated by TLC), the reaction mixture was quenched with $NaHCO_3$. Then the reaction mixture was poured into water (40 mL) and extracted with ethyl acetate (40 mL). The organic layer was washed with water (10 mL) and brine (10 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The crude product was purified by column chromatography using ethyl acetate/hexane as eluent to afford the desired product.

SPECTRAL DATA

Phenyl(2-(pyridin-2-yl)phenyl)methanone, 3a, Scheme 4. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), benzaldehyde (32 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography

(SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (45.0 mg, 87%), mp 105-107 ^oC. The spectral data of compound **3a** is in Scheme 2.

(2-(Pyridin-2-yl)phenyl)(*p*-tolyl)methanone, 3b, Scheme 4. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 4-methylbenzaldehyde (36 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (42.5 mg, 78%), 97-99 ^oC. The spectral data of compound **3b** is in Scheme 2.

(4-Methoxyphenyl)(2-(pyridin-2-yl)phenyl)methanone, 3e, Scheme 4. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 4-methoxybenzaldehyde (36.5 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a yellowish oil, (39.0 mg, 68%). The spectral data of compound **3e** is in Scheme 2.

1-(4-(2-(Pyridin-2-yl)benzoyl)phenyl)ethanone, 3ac, Scheme 4. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 4-acetylbenzaldehyde (44.5 mg, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (44.0 mg, 73%), 80-82 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.30 (d, J =

4.8 Hz, 1H), 7.83 (d, *J* = 8.4 Hz, 2H), 7.80 (d, *J* = 7.8 Hz, 1H), 7.75 (d, *J* = 8.4 Hz, 2H), 7.62-7.66 (m, 1H), 7.60 (td, *J* = 7.8 Hz, 1.2 Hz, 1H), 7.56-7.58 (m, 3H), 7.00-7.02 (m, 1H), 2.57 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 197.6, 197.3, 156.1, 148.8, 141.6, 139.3, 139.2, 139.0, 136.5, 130.5, 129.2, 129.1, 128.8, 128.4, 127.9, 122.2, 122.1, 26.8; IR (neat): υ_{max} 1682, 1433, 1259, 930, 753 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{20}H_{15}NO_2$ $[M]^+$: 301.1103; found: 301.1104.

4-(2-(Pyridin-2-yl)benzoyl)benzonitrile, 3ae, Scheme 4.[9] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 4-formylbenzonitrile (40 mg, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (26.5 mg, 47%), mp 106-108 ^oC. ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.26 (d, *J* = 4.2 Hz, 1H), 7.81 (d, *J* = 7.2 Hz, 1H), 7.74-7.75 (m, 2H), 7.54-7.67 (m, 7H), 7.02-7.04 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 196.4, 155.7, 148.7, 141.6, 139.2, 138.4, 136.7, 131.9, 130.7, 129.2, 129.0, 128.2, 122.3, 122.0, 118.2, 115.0; IR (neat): υ_{max} 2225, 1671, 1285, 929, 744 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{19}H_{12}N_2O$ $[M]^+$: 284.0950; found: 284.0958.

(3-Nitrophenyl)(2-(pyridin-2-yl)phenyl)methanone, 3af, Scheme 4.[9] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 3-nitrobenzaldehyde (45 mg, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (34.0 mg, 56%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.44 (s, 1H), 8.26 (d, *J* = 4.2 Hz, 1H), 8.22 (d, *J* = 7.8 Hz, 1H), 8.03 (d, *J* = 7.8 Hz, 1H), 7.83 (d, *J* = 7.2 Hz, 1H), 7.58-7.69 (m, 5H), 7.47 (t, *J* = 7.8 Hz,

1H), 7.00-7.02 (m,1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 195.5, 155.8, 148.7, 147.9, 139.8, 139.3, 138.2, 136.8, 134.4, 130.8, 129.20, 129.17, 129.0, 128.4, 126.2, 123.6, 122.2, 122.0; IR (neat): υ_{max} 1674, 1531, 1349, 1277, 753 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{12}N_2O_3Na$ $[M + Na]^+$: 327.0746; found: 327.0752.

Naphthalen-2-yl(2-(pyridin-2-yl)phenyl)methanone, 3ag, Scheme 4.[8] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 2-naphthaldehyde (47 mg, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (37.0 mg, 60%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.33 (d, *J* =4.8 Hz, 1H), 8.09 (s, 1H), 7.94 (dd, *J* = 9.0 Hz, 1.8 Hz, 1H), 7.83 (d, *J* = 7.2 Hz, 1H), 7.77-7.81 (m, 3H), 7.65-7.67 (m, 1H), 7.62 (dd, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.51-7.58 (m, 4H), 7.46 (t, *J* = 7.2 Hz, 1H), 6.93-6.95 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.2, 156.7, 149.0, 139.7, 139.6, 136.3, 135.3, 135.2, 132.2, 131.5, 130.2, 129.4, 129.1, 128.8, 128.5, 128.1, 128.0, 127.6, 126.4, 124.9, 122.5, 121.9; IR (neat): υ_{max} 1662, 1588, 1465, 1289, 753 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{22}H_{15}NO$ $[M]^+$: 309.1154; found: 309.1150.

(5-Methyl-2-(pyridin-2-yl)phenyl)(*p*-tolyl)methanone, 3al, Scheme 4.[14] The same general procedure was followed by using 2-(p-tolyl)pyridine (34.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 4-methylbenzaldehyde (36 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (40.0 mg, 70%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.37-8.38 (m, 1H), 7.68 (d, *J* = 8.4 Hz, 1H), 7.61 (d, *J* = 8.4 Hz, 2H), 7.53-7.55 (m, 1H), 7.46 (d, *J* = 7.8 Hz, 1H), 7.40 (d, *J* =

7.8 Hz, 1H), 7.32 (s, 1H), 7.07 (d, *J* = 8.4 Hz, 2H), 6.99-7.01 (m, 1H), 2.45 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.2, 156.8, 149.0, 143.0, 139.6, 138.5, 136.7, 136.1, 135.3, 130.7, 129.7, 129.4, 128.72, 128.70, 122.6, 121.6, 21.6, 21.2; IR (neat): υ_{max} 2923, 1663, 1603, 1463, 1290, 757 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{20}H_{17}NO$ $[M]^+$: 287.1310; found: 287.1316.

Phenyl(2-(quinolin-2-yl)phenyl)methanone, 3ao, Scheme 4.[16] The same general procedure was followed by using 2-phenylquinoline (41.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), benzaldehyde (32 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (28.5 mg, 46%), mp 118-120 ^{o}C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.07 (d, *J* = 8.4 Hz, 1H), 7.94 (d, *J* = 7.8 Hz, 1H), 7.76-7.77 (m, 2H), 7.65-7.74 (m, 4H), 7.57-7.61 (m, 3H), 7.43 (d, *J* = 7.2 Hz, 1H), 7.31-7.33 (m, 1H), 7.23 (t, *J* = 7.8 Hz, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.2, 156.1, 147.3, 140.3, 139.4, 138.2,136.6, 132.1, 130.1, 129.5, 129.2, 129.04, 128.99, 128.82, 128.80, 127.9, 127.2, 126.54, 126.46, 119.9; IR (neat): υ_{max} 1665, 1596, 1282, 931, 768 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{22}H_{15}NONa$ $[M + Na]^+$: 332.1051; found: 332.1050.

Furan-2-yl(2-(pyridin-2-yl)phenyl)methanone, 3aq, Scheme 4.[17] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), furan-2-carbaldehyde (25 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (21.0 mg, 42%). ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.49 (d, *J* = 4.8 Hz, 1H), 7.78 (d, *J* = 7.8 Hz, 1H), 7.61-7.66 (m, 3H), 7.52-7.54 (m, 2H), 7.43 (s, 1H), 7.10-7.12 (m, 1H), 6.82 (d, *J* = 3.0 Hz, 1H), 6.34-6.35 (m, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 185.4, 157.1, 152.8, 149.2, 146.5, 139.8, 138.3, 136.4,

130.6, 129.1, 129.0, 128.4, 122.7, 121.9, 119.3, 111.9; IR (neat): υ_{max} 1656, 1566, 1465, 1301, 1019, 753 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{16}H_{11}NO_2Na$ $[M + Na]^+$: 272.0687; found: 272.0634.

3-Methyl-1-(2-(pyridin-2-yl)phenyl)butan-1-one, 3w, Scheme 4. The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), 3-methylbutanal (33 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (31.0 mg, 65%). The spectral data of compound **3w** is in Scheme 2.

Cyclohexyl(2-(pyridin-2-yl)phenyl)methanone, 3ar, Scheme 4.[18] The same general procedure was followed by using 2-phenylpyridine (31.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), cyclohexanecarbaldehyde (36.5 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (46.5 mg, 88%), mp 86-88 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.64 (d, *J* = 4.2 Hz, 1H), 7.76 (t, *J* = 7.2 Hz, 1H), 7.68 (d, *J* = 7.8 Hz, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.51 (t, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 1H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.23-7.25 (m, 1H), 2.18-2.23 (m, 1H), 1.73 (d, *J* = 13.2 Hz, 2H), 1.66-1.69 (m, 2H), 1.55 (d, *J* = 13.2 Hz, 1H), 1.35-1.42 (m, 2H), 1.10-1.17 (m, 1H), 0.94-1.02 (m, 2H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 210.8, 157.2, 149.3, 141.3, 138.1, 136.8, 129.8, 128.7, 128.6, 128.1, 122.3, 122.3, 50.8, 29.1, 25.9, 25.8; IR (neat): υ_{max} 2928, 1687, 1586, 1438, 976, 750 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{19}NONa$ $[M + Na]^+$: 288.1364; found: 288.1361.

Benzo[h]quinolin-10-yl(phenyl)methanone, 3at, Scheme 4.[9] The same general procedure was followed by using benzo[*h*]quinoline (36.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), benzaldehyde (32 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (51.5 mg, 91%), mp 148-150 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.51 (d, *J* = 4.2 Hz, 1.8 Hz, 1H), 8.04-8.07 (m, 2H), 7.88 (d, *J* = 8.4 Hz, 1H), 7.77-7,80 (m, 3H), 7.72 (d, *J* = 9.0 Hz, 1H), 7.65 (dd, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.40-7.43 (m, 1H), 7.29-7.32 (m, 3H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 198.6, 147.0, 144.6, 139.2, 138.9, 135.3, 133.8, 131.7, 129.2, 129.0, 128.7, 128.1, 127.8, 127.7, 127.0, 126.4, 126.1, 121.6; IR (neat): υ_{max} 1672, 1417, 1272, 841, 708 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{20}H_{13}NO$ $[M]^+$: 283.0997; found: 283.0993.

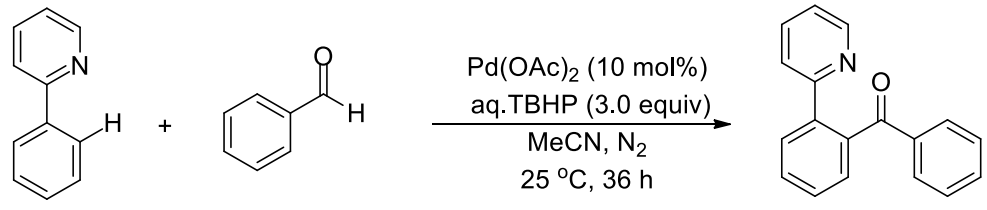
Phenyl(2-(pyrimidin-2-yl)phenyl)methanone, 3x, Scheme 4. The same general procedure was followed by using 2-phenylpyrimidine (31.5 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), benzaldehyde (32 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (34.0 mg, 65%), mp 129-131 ^oC. The spectral data of compound **3x** is in Scheme 2.

Phenyl(2-(pyridin-2-yloxy)phenyl)methanone, 3au, Scheme 4.[19] The same general procedure was followed except the reaction was run for 72 hours by using 2-phenoxypyridine (34.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), benzaldehyde (32 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a colourless oil, (12.0 mg, 22%). ^1H NMR (600 MHz, $CDCl_3$): δ 8.00-8.02 (m, 1H),

7.76 (d, *J* = 8.4 Hz, 2H), 7.56-7.59 (m, 2H), 7.46-7.52 (m, 2H), 7.30-7.34 (m, 3H), 7.27 (d, *J* = 8.4 Hz, 1H), 6.86-6.88 (m, 1H), 6.60 (d, *J* = 8.4 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 195.3, 163.0, 151.6, 147.0, 139.3, 137.5, 132.8, 132.22, 132.15, 130.3, 129.8, 128.0, 124.7, 122.8, 118.5, 111.5, IR (neat): υ_{max} 1666, 1590, 1432, 1250, 766 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{18}H_{13}NO_2Na$ $[M + Na]^+$: 298.0844; found: 298.0861.

(2-(1*H*-Pyrazol-1-yl)phenyl)(phenyl)methanone, 3av, Scheme 4.[19] The same general procedure was followed by using 1-phenyl-1*H*-pyrazole (29.0 mg, 0.2 mmol, 1.0 equiv), palladium(II) acetate (4.5 mg, 0.02 mmol, 0.1 equiv), benzaldehyde (32 µL, 0.3 mmol, 1.5 equiv), and aq. *tert*-butyl hydroperoxide (82 µL, 0.6 mmol, 3.0 equiv). Column chromatography (SiO_2, eluting with 7:3 hexane/ethyl acetate) afforded the desired product as a white solid, (16.0 mg, 32%), mp 82-84 ^{o}C. ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.58-7.66 (m, 6H), 7.50 (td, *J* = 7.2 Hz, 1.2 Hz, 1H), 7.43-7.46 (m, 1H), 7.41 (d, *J* = 1.8 Hz, 1H), 7.30 (t, *J* = 8.4 Hz, 2H), 6.19 (t, *J* = 1.8 Hz, 1H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 195.8, 141.2, 138.6, 136.7, 133.8, 132.9, 131.2, 129.8, 129.6, 129.0, 128.1, 127.5, 123.2, 107.6; IR (neat): υ_{max} 1669, 1590, 1264, 931, 762 cm^{-1}; HRMS (EI, m/z) calcd. for $C_{16}H_{12}N_2O$ $[M]^+$: 248.0950; found: 248.0947.

Dehydrogenative acylation reaction in gram scale (Synthesis of phenyl(2-(pyridin-2-yl)phenyl)methanone, 3a, Scheme 4).



1a
1.0 equiv
6.5 mmol
0.93 mL

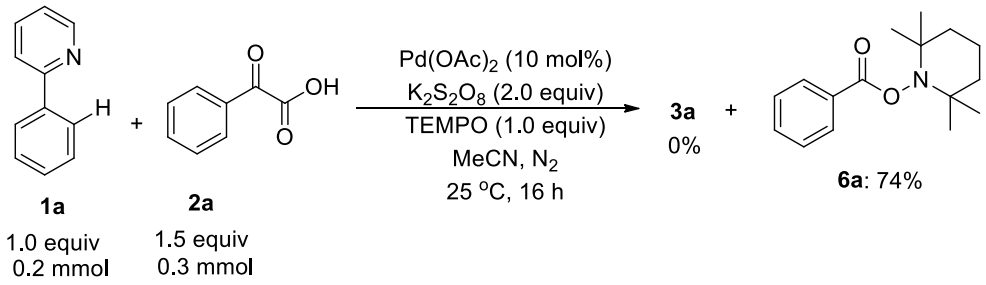
5a
1.5 equiv
9.8 mmol
1.0 mL

3a
72% yield
1.21 g

To an oven-dried 100 mL sealed tube, a mixture of 2-phenylpyridines (0.93 mL, 6.5 mmol, 1.0 equiv) and palladium(II)acetate (146 mg, 0. 65 mmol, 0.1 equiv) was taken and dry MeCN (50 mL) was added to it. Then benzaldehyde (1.0 mL, 9.8 mmol 1.5 equiv) was added to the reaction mixture. After few seconds flushing with nitrogen, immediately 70% aq. TBHP (2.6 mL, 19.5 mmol, 3.0 equiv) was added to the reaction mixture via syringe. The vessel was sealed with a screw cap. The reaction mixture was allowed to stir for 36 h at room temperature. After completion (as indicated by TLC), the reaction mixture was quenched with $NaHCO_3$. Then the reaction mixture was poured into water (80 mL) and extracted with ethyl acetate (60x2 mL). The organic layer was washed with water (30x2 mL) and brine (30 mL), dried over anhydrous Na_2SO_4 and the solvent was evaporated under reduced pressure. The pure phenyl(2-(pyridin-2-yl)phenyl)methanone (**3a**) was obtained as a white solid in 72% (1.21 g) yield after column chromatography of the crude reaction mixture using ethyl acetate/hexane (7:3) as eluent.

Control experiments, Scheme 5.

The standard decarboxylative acylation reaction with radical scavenger (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO), Scheme 5a.



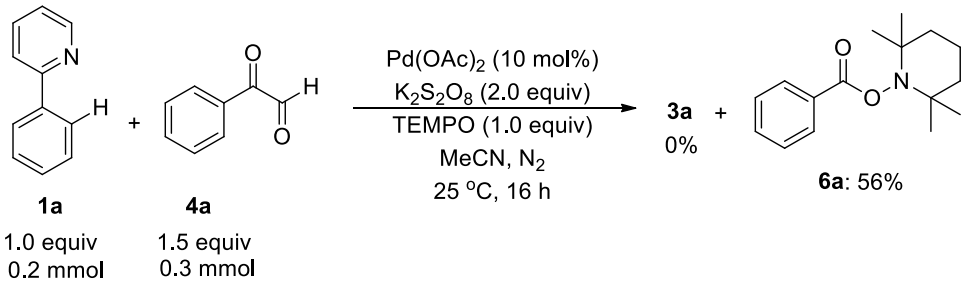
To an oven-dried 10 mL sealed tube, a mixture of 2-phenylpyridine (31.5 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetic acid (45 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv), palladium(II)acetate (4.6 mg, 0.02 mmol, 0.1 equiv) and (2,2,6,6-

Tetramethylpiperidin-1-yl)oxyl (TEMPO) (31.5 mg, 0.2 mmol, 1.0 equiv) was taken and dry MeCN (3.0 mL) was added to it. After few seconds flushing with nitrogen, the vessel was sealed with a screw cap. The reaction mixture was allowed to stir for 16 h at room temperature. After that the reaction mixture was quenched with $NaHCO_3$. We did not detect any desired acylation product (**3a**) by TLC. However, we isolated TEMPO-acyl adduct, 2,2,6,6-tetramethylpiperidin-1-yl benzoate (**6a**) in 74% yield as a white solid from the reaction mixture.

This control experiment suggested that the reaction may proceed via radical pathway and the acyl radical intermediate may form from the corresponding 2-oxo-2-phenylacetic acids.

2,2,6,6-Tetramethylpiperidin-1-yl benzoate 6a, Scheme 5a.[20] Column chromatography (SiO_2, eluting with 95:5 hexane/ethyl acetate) afforded the desired product as a white solid, (39.0 mg, 74%), mp 85-87 ^oC. ^1H NMR (600 MHz, $CDCl_3$): δ 8.08-8.10 (m, 2H), 7.59 (t, *J* = 7.2 Hz, 1H), 7.47 (t, *J* = 7.8 Hz, 2H), 1.77-1.82 (m, 2H), 1.67-1.74 (m, 1H), 1.59-1.62 (m, 2H), 1.46-1.49 (m, 1H), 1.29 (s, 6H), 1.13 (s, 6H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 166.4, 132.8, 129.7, 129.5, 128.4, 60.4, 39.0, 31.9, 20.8, 17.0; IR (neat): υ_{max} 2932, 1742, 1452, 1248, 1067, 712 cm^{-1}; HRMS (ESI, m/z) calcd. for $C_{16}H_{23}NO_2Na$ $[M + Na]^+$: 284.1626; found: 284.1628.

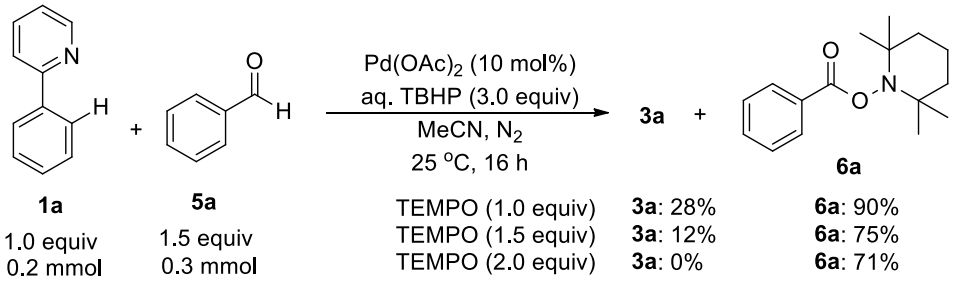
The standard decarbonylative acylation reaction with radical scavenger (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO), Scheme 5a.



To an oven-dried 10 mL sealed tube, a mixture of 2-phenylpyridine (31.5 mg, 0.2 mmol, 1.0 equiv), 2-oxo-2-phenylacetaldehyde (40 mg, 0.3 mmol, 1.5 equiv), potassium persulfate (108 mg, 0.4 mmol, 2.0 equiv), palladium(II)acetate (4.6 mg, 0.02 mmol, 0.1 equiv) and (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO) (31.5 mg, 0.2 mmol, 1.0 equiv) was taken and dry MeCN (3.0 mL) was added to it. After few seconds flushing with nitrogen, the vessel was sealed with a screw cap. The reaction mixture was allowed to stir for 16 h at room temperature. After that the reaction mixture was quenched with $NaHCO_3$. We did not detect any desired acylation product (**3a**) as indicated by TLC. But we isolated TEMPO-acyl adduct, 2,2,6,6-tetramethylpiperidin-1-yl benzoate (**6a**) in 56% as a white solid from the reaction mixture.

This control experiment suggested that the reaction may proceed via radical pathway and the acyl radical intermediate may form from the corresponding arylglyoxals.

The standard dedrogenative acylation reaction with radical scavenger (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO), Scheme 5a.

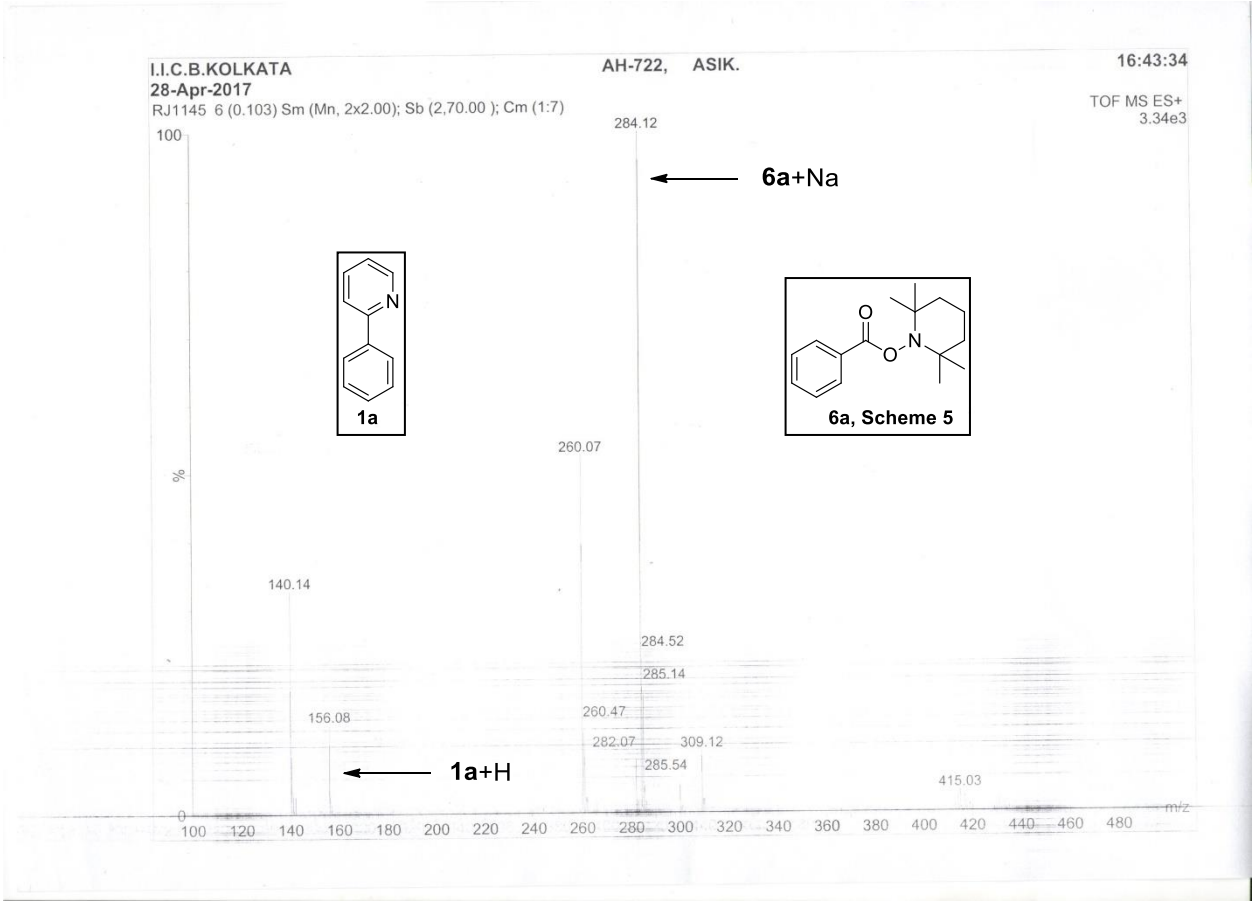


To an oven-dried 7 mL clear vial, a mixture of 2-phenylpyridine (31.5 mg, 0.2 mmol, 1.0 equiv) and palladium(II)acetate (4.6 mg, 0.02 mmol, 0.1 equiv) was taken and dry MeCN (3.0 mL) was added to it. Then benzaldehyde (32.0 µL, 0.3 mmol 1.5 equiv) and (2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO) (63.0 mg, 0.4 mmol, 2.0 equiv) was added to the reaction mixture. After flushing with nitrogen, immediately the vessel was sealed with a screw cap. Then aq. TBHP (82 µL, 0.6 mmol, 3.0 equiv) was added to the reaction mixture via micro-

litter syringe. The reaction mixture was allowed to stir for 36 h at room temperature. After that the reaction mixture was quenched with $NaHCO_3$. We did not detect any desired acylation product (**3a**) as indicated by TLC. But we isolated TEMPO-acyl adduct, 2,2,6,6-tetramethylpiperidin-1-yl benzoate (**6a**) in 71% as a white solid from the reaction mixture.

When the same experiment was performed with 1.0 equiv of 2,2,6,6-Tetramethylpiperidin-1-yl)oxyl (TEMPO) (31.5 mg, 0.2 mmol), we isolated the desired acylation product (**3a**) in 28% yield and also TEMPO-acyl adduct, 2,2,6,6-tetramethylpiperidin-1-yl benzoate (**6a**) in 90% as a white solid from the reaction mixture. In addition, when the same experiment was performed with 1.5 equiv of (2,2,6,6-tetramethylpiperidin-1-yl)oxyl (TEMPO) (47.0 mg, 0.3 mmol), we isolated the desired acylation product (**3a**) in 12% yield and also TEMPO-acyl adduct, 2,2,6,6-tetramethylpiperidin-1-yl benzoate (**6a**) in 75% as a white solid from the reaction mixture. This control experiment suggested that the reaction may proceed via radical pathway and the acyl radical intermediate may form from the corresponding aldehydes.

ESI mass of the crude reaction mixture for TEMPO experiment



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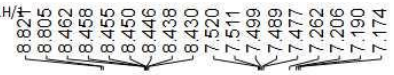
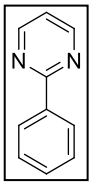
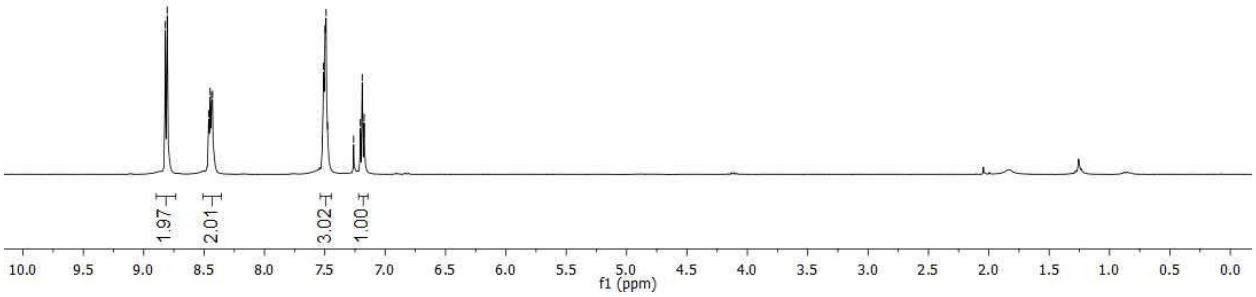
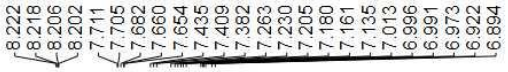
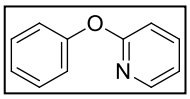


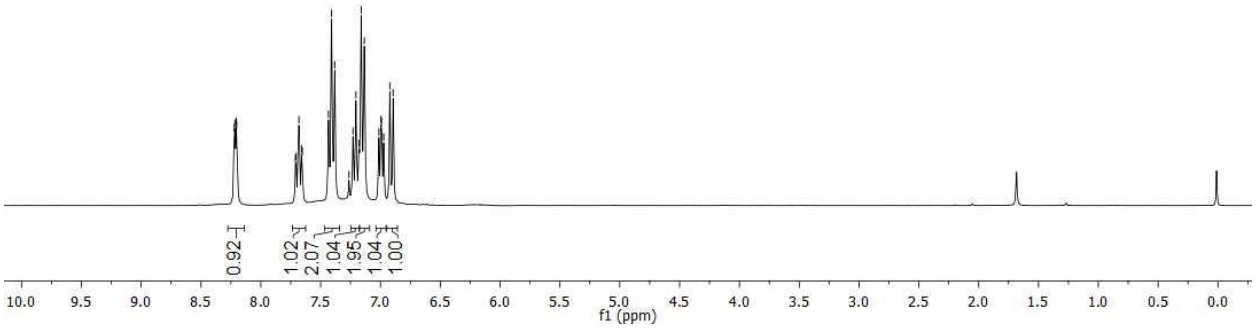




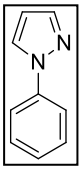
AH-652-1H/1

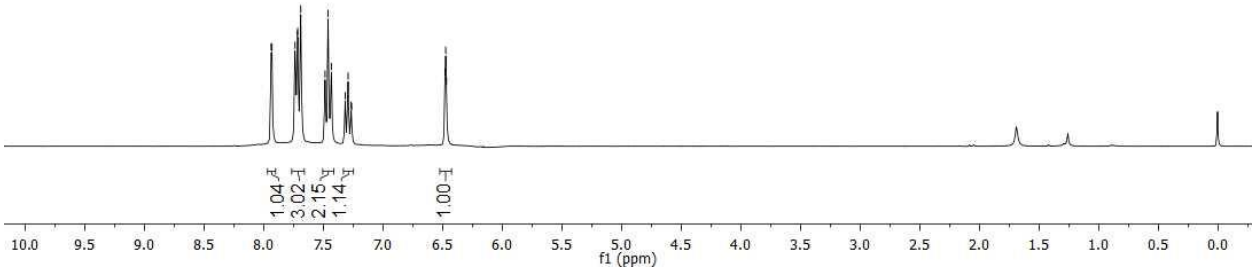




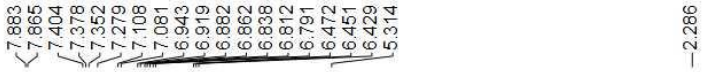


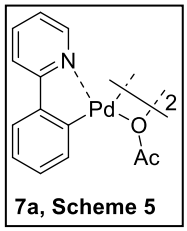
AH-654-1H/1



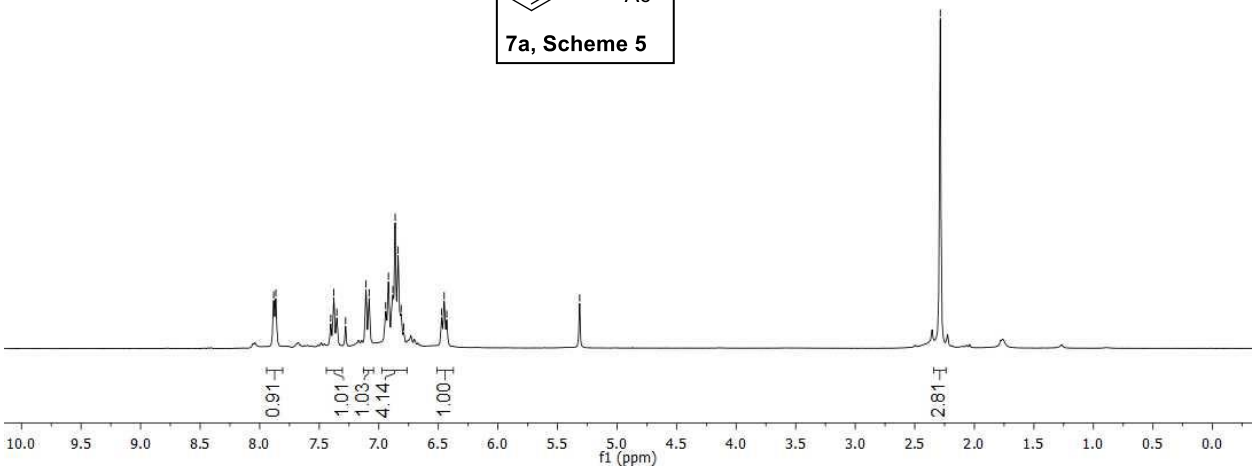


AH-677-1H/1

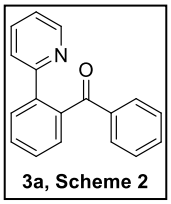


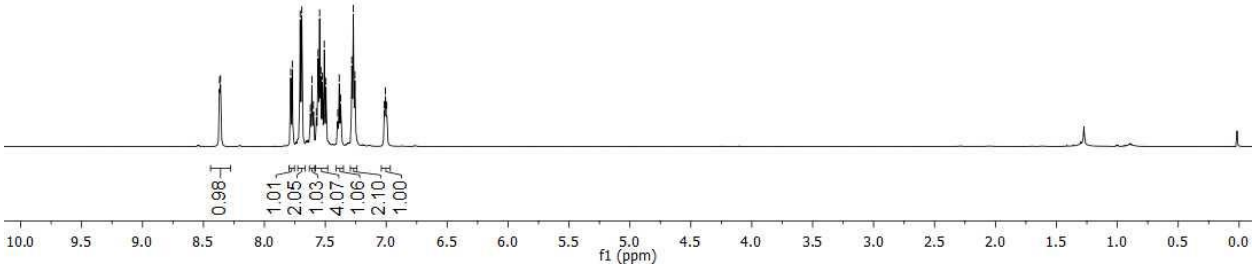


7a, Scheme 5

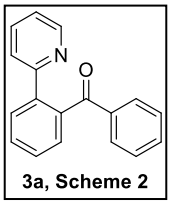


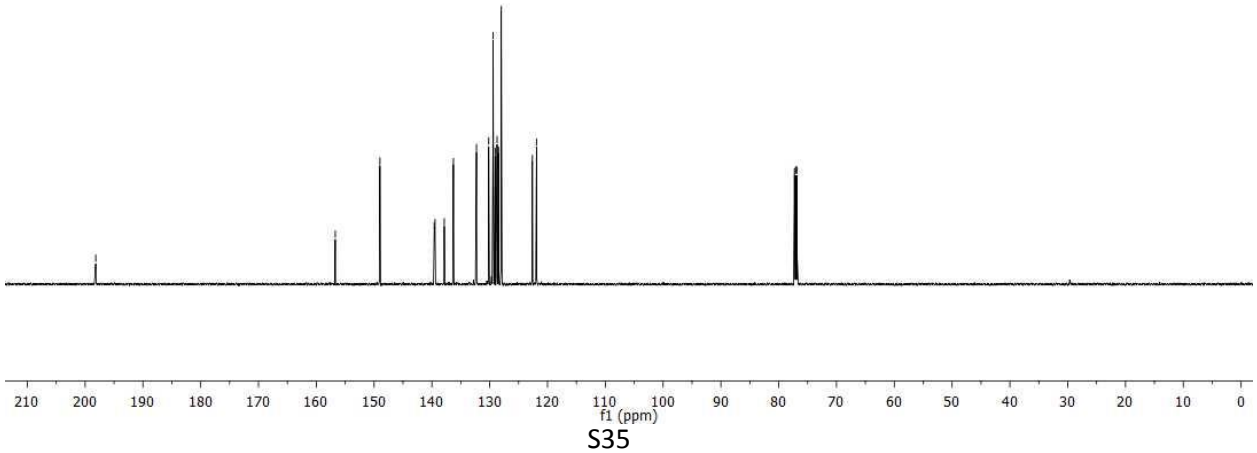

acylation NMR/AH-607 P
AH-607 1H-NMR in CDCl3
8.367
8.360
7.784
7.771
7.706
7.693
7.622
7.610
7.598
7.574
7.559
7.547
7.536
7.524
7.508
7.495
7.398
7.386
7.373
7.282
7.270
7.257
7.015
7.007
6.996

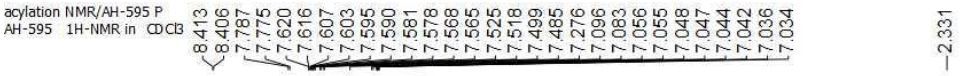

N
O
3a, Scheme 2

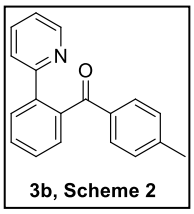

0.98
1.01
2.05
1.03
4.07
1.06
2.10
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


acylation NMR/AH-607 C
AH-607 13C-NMR in CDCl3
198.71
156.72
148.99
139.60
139.46
137.86
136.28
132.29
130.17
129.41
129.06
128.72
128.47
128.00
122.62
121.90
77.28
77.07
76.85

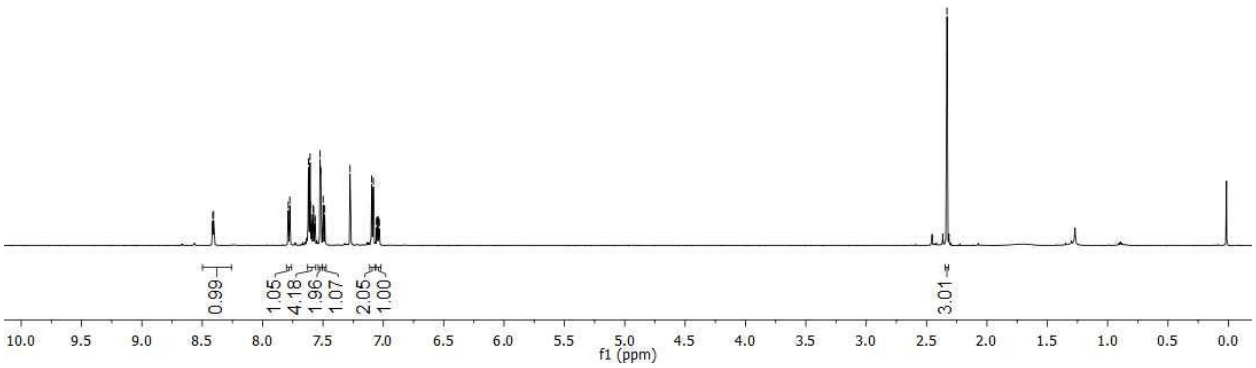

N
O
3a, Scheme 2

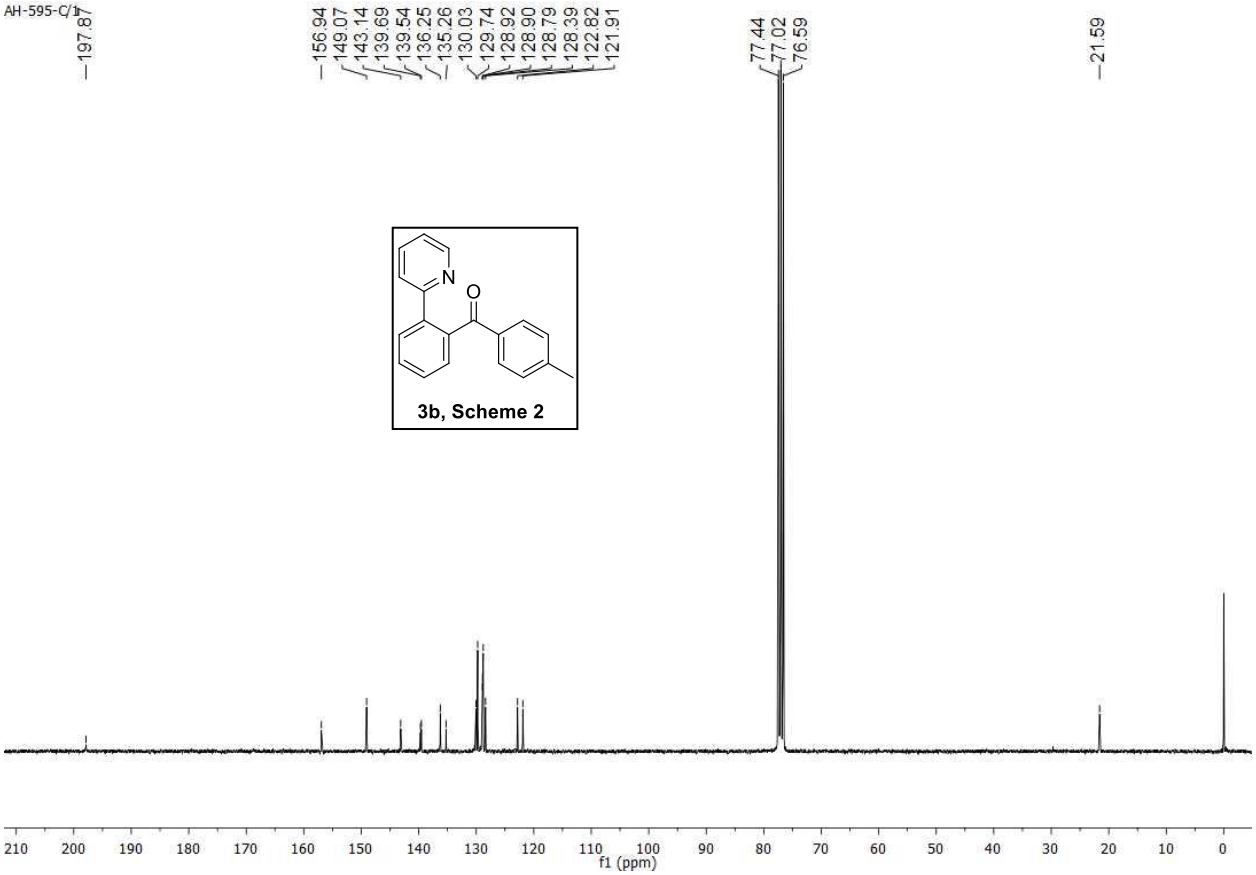

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



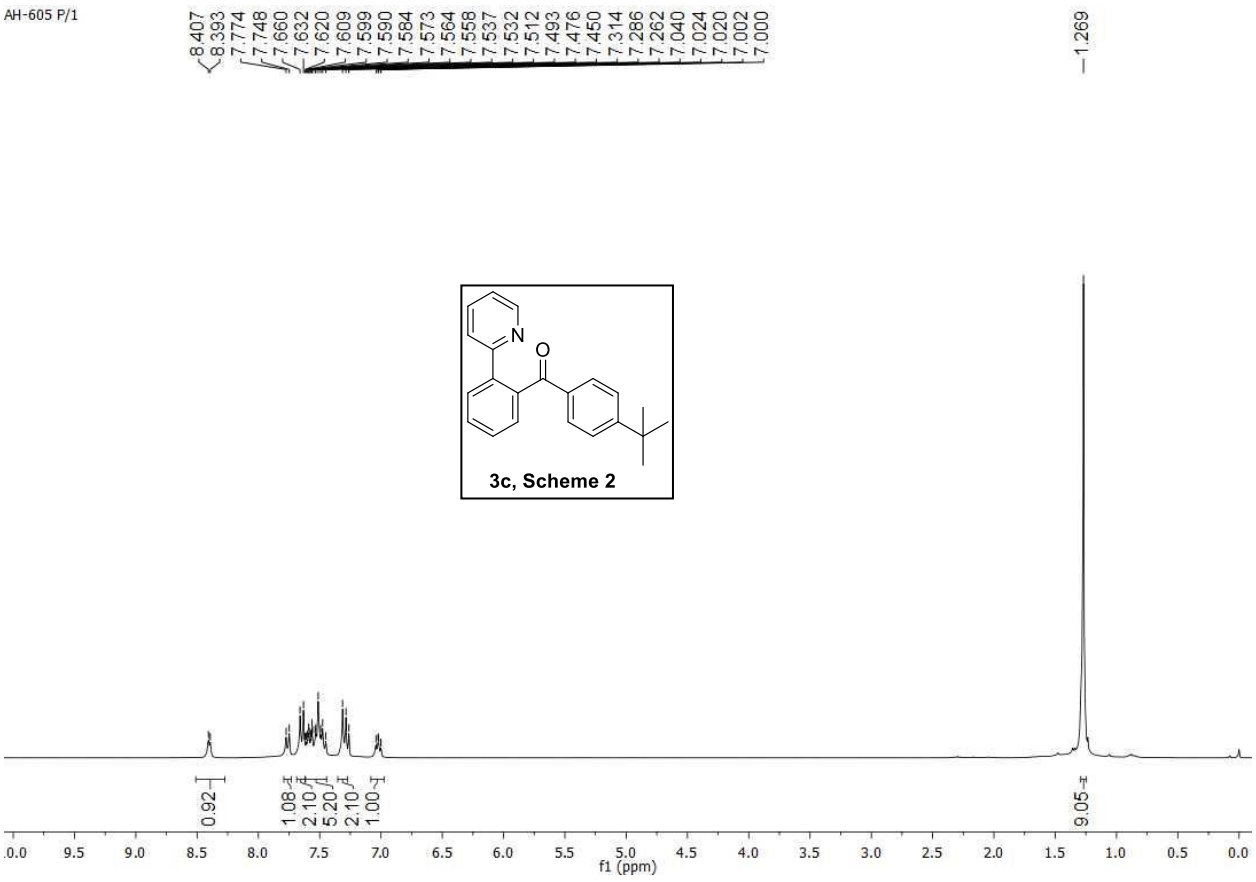


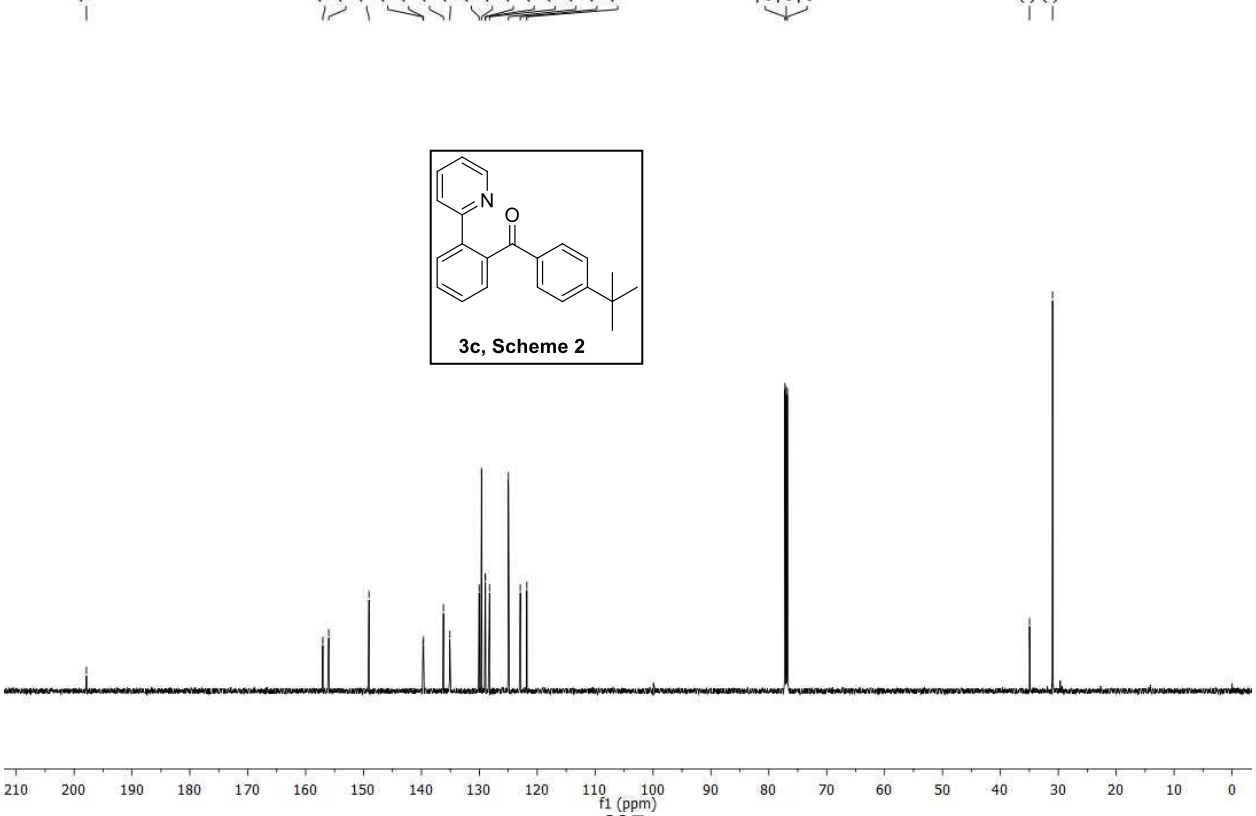
3b, Scheme 2

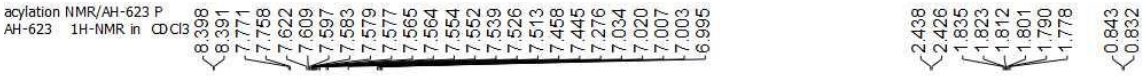


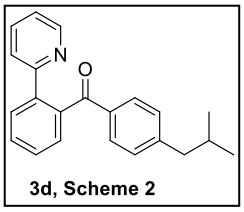


3b, Scheme 2

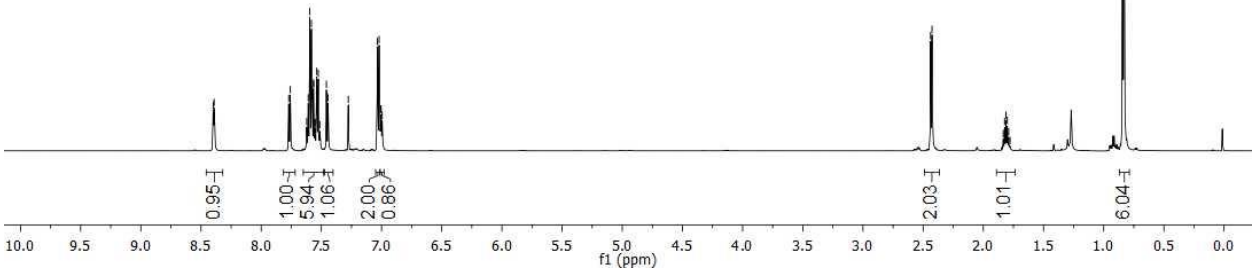

AH-605 P/1
8.407
8.393
7.774
7.748
7.660
7.632
7.620
7.609
7.599
7.590
7.584
7.573
7.564
7.558
7.537
7.532
7.512
7.493
7.476
7.450
7.314
7.286
7.262
7.040
7.024
7.020
7.002
7.000
1.269
3c, Scheme 2
0.92
1.08
2.10
5.20
2.10
1.00
9.05
f1 (ppm)

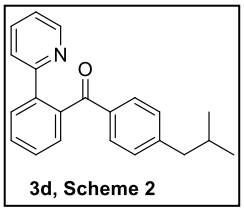

acylation NMR/AH-605 C
AH-605 13C-NMR in CDCl3
197.
157.04
156.02
149.06
139.74
139.64
136.20
135.11
130.03
129.62
129.00
128.94
128.24
124.98
122.93
121.80
77.21
77.00
76.79
34.97
30.99
3c, Scheme 2
f1 (ppm)



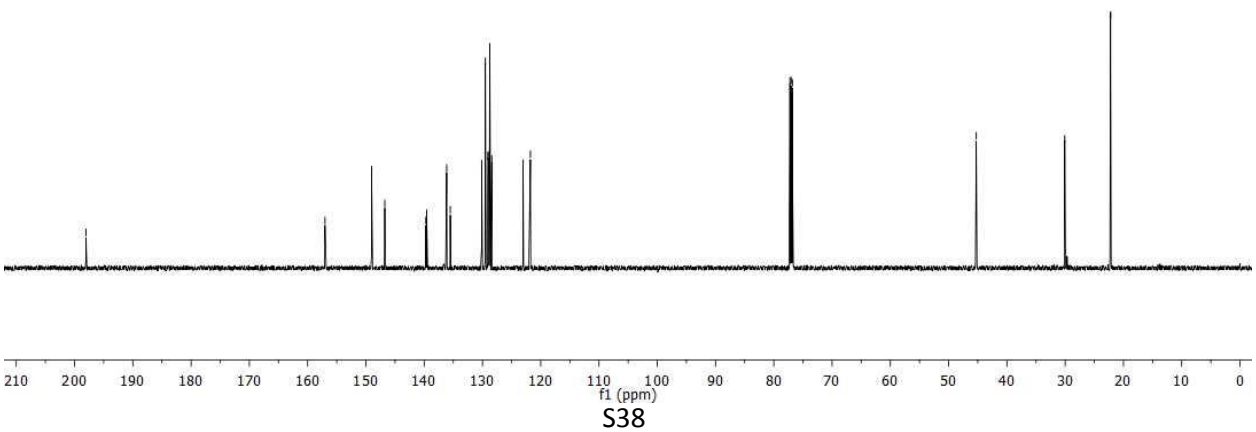


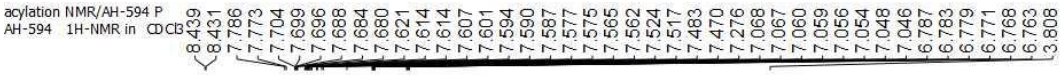
3d, Scheme 2

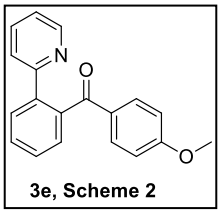




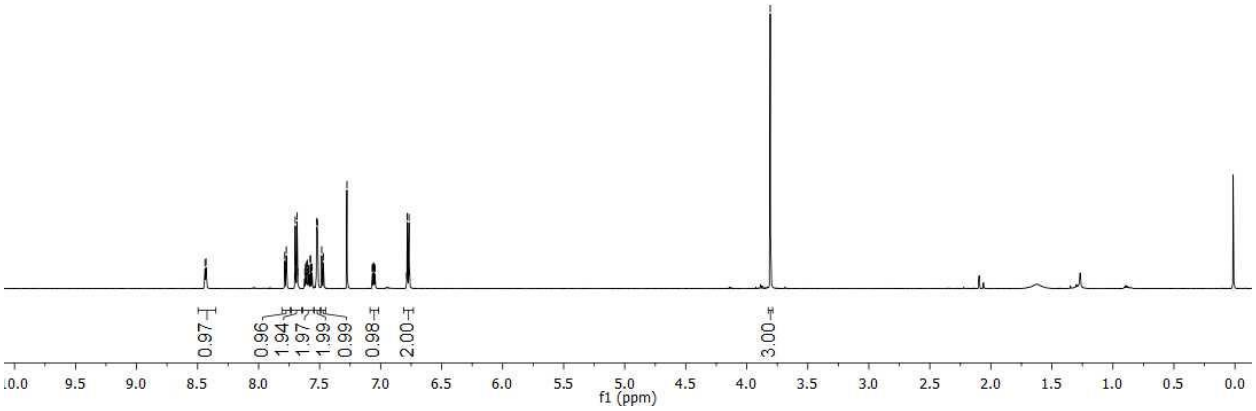
3d, Scheme 2

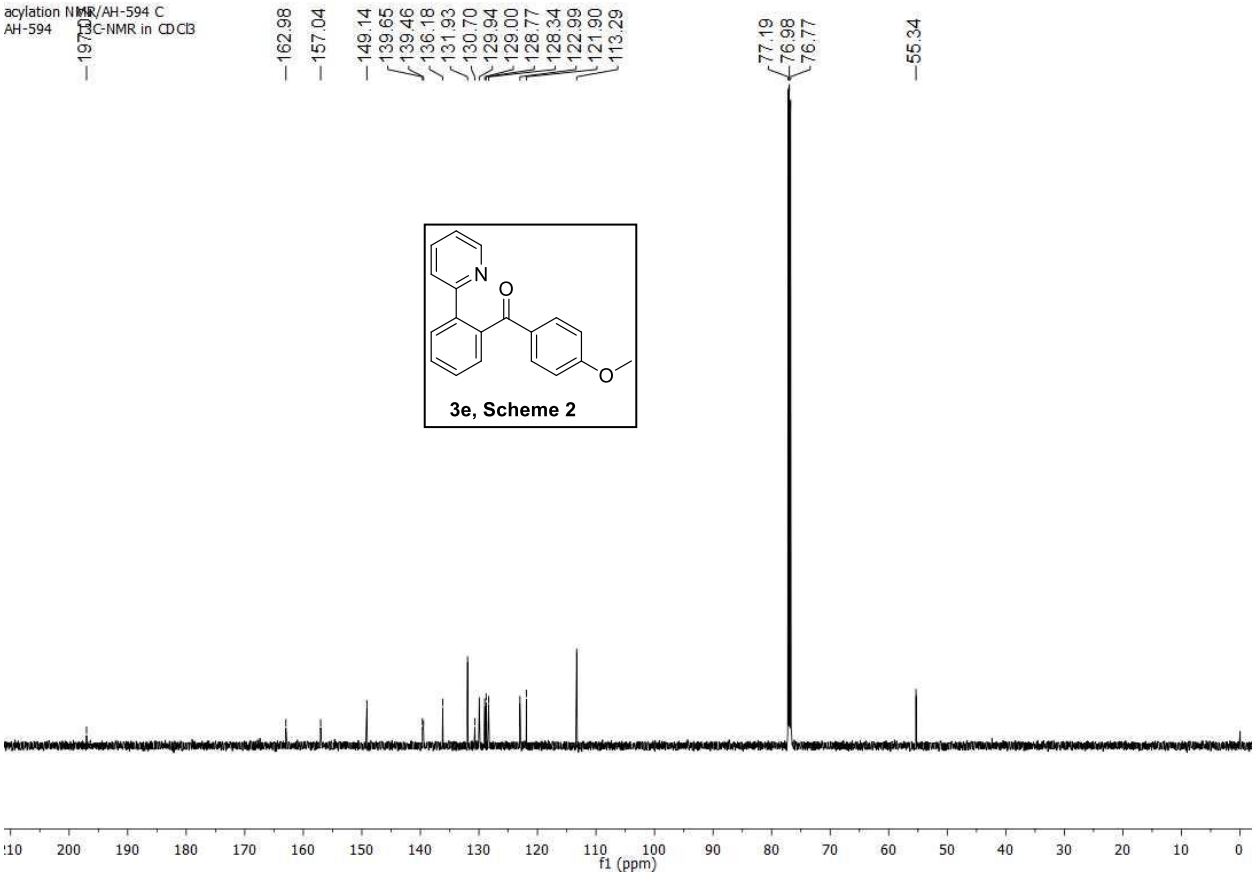




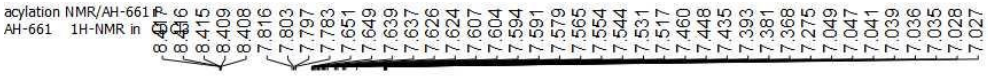


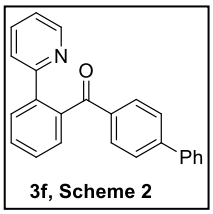
3e, Scheme 2



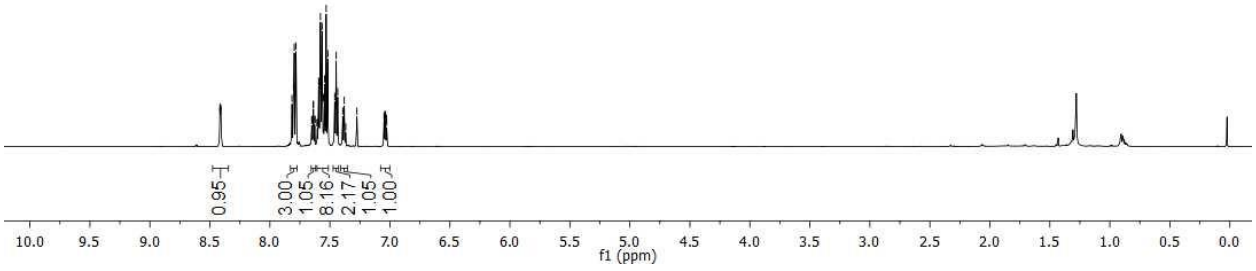


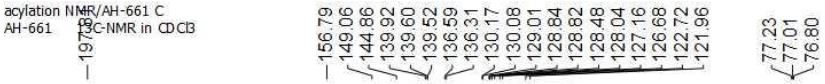
3e, Scheme 2

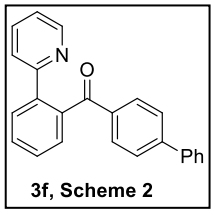




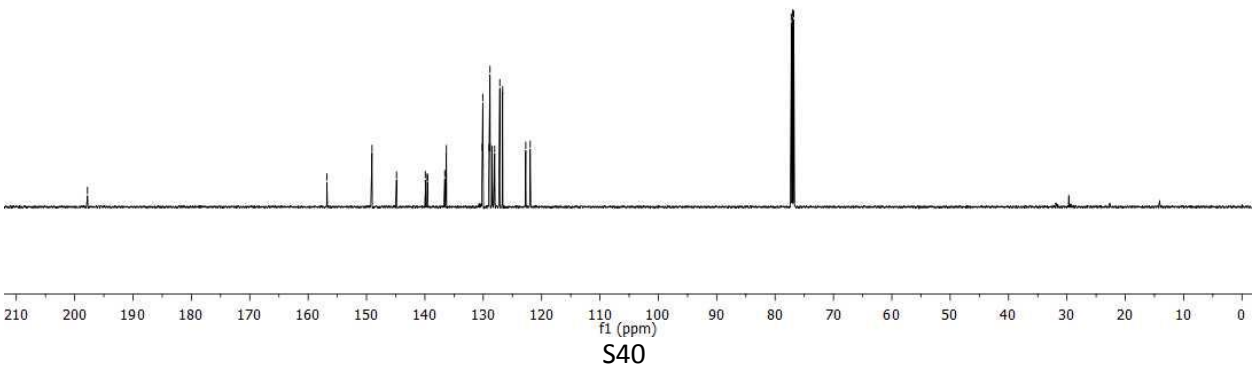
3f, Scheme 2

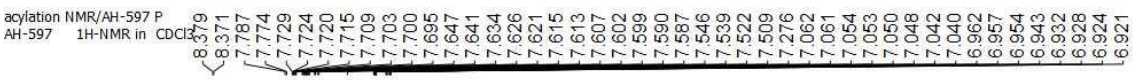






3f, Scheme 2




acylation NMR/AH-597 P
AH-597 1H-NMR in CDCl3
8.379
8.371
7.787
7.774
7.729
7.724
7.720
7.715
7.709
7.703
7.700
7.695
7.647
7.641
7.634
7.626
7.621
7.615
7.613
7.607
7.602
7.599
7.590
7.587
7.546
7.539
7.522
7.509
7.276
7.062
7.061
7.054
7.053
7.050
7.048
7.042
7.040
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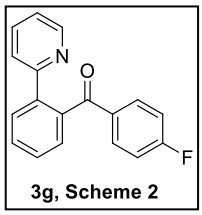
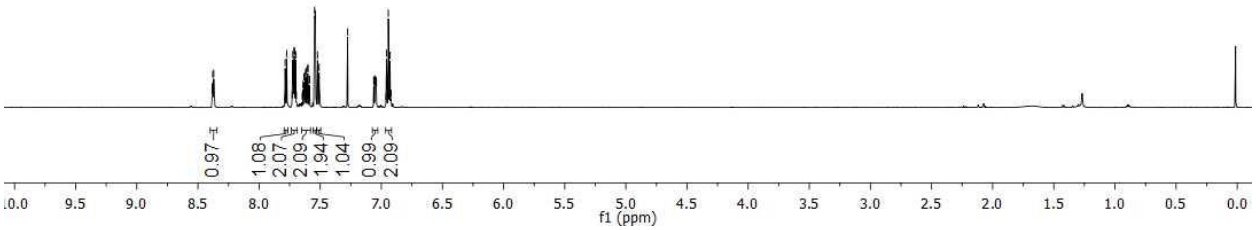
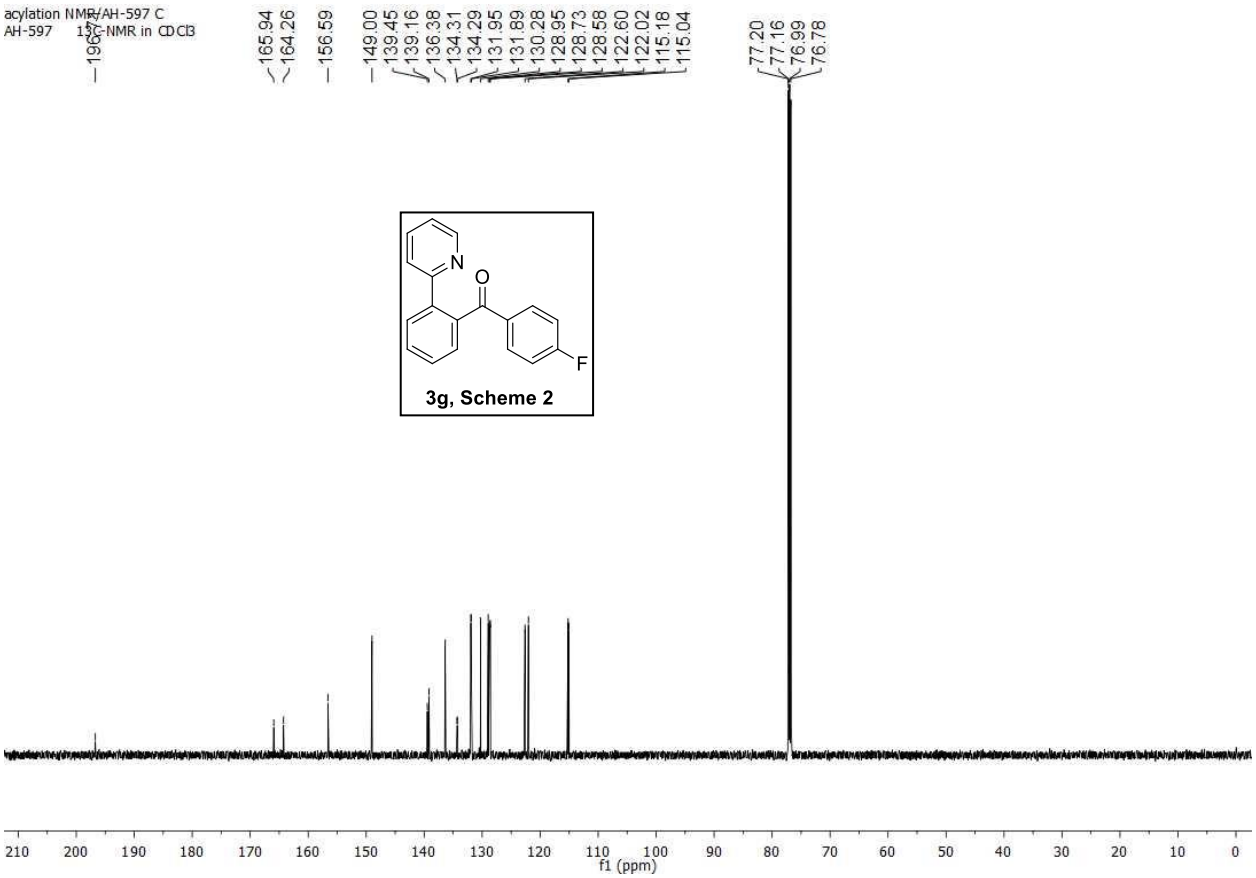
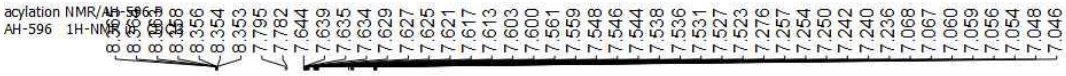
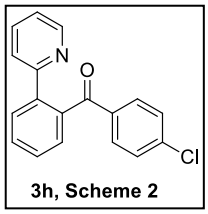
6.954
6.943
6.932
6.928
6.924
6.921

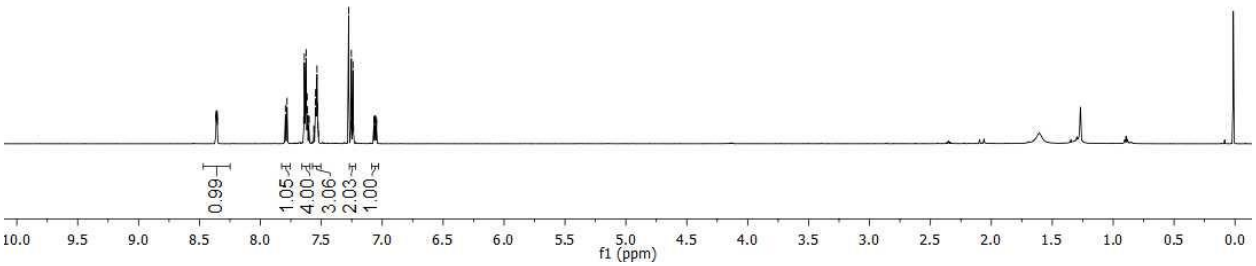

N
O
F
3g, Scheme 2

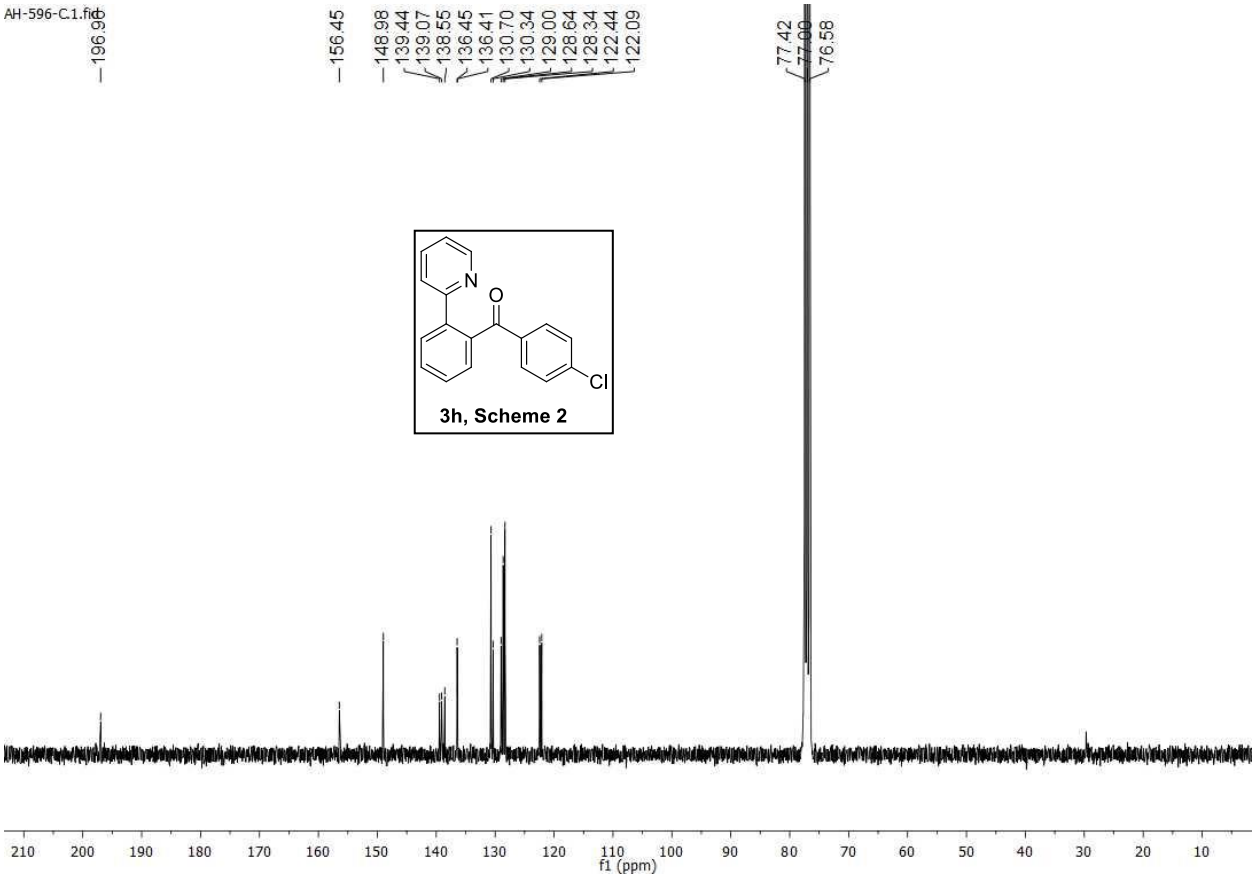

0.97
1.08
2.07
2.09
1.94
1.04
0.99
2.09
f1 (ppm)

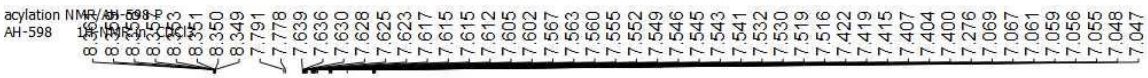

acylation NMR/AH-597 C
AH-597 13C-NMR in CDCl3
196.57
165.94
164.26
156.59
149.00
139.45
139.16
136.38
134.31
134.29
131.95
131.89
130.28
128.95
128.73
128.58
122.60
122.02
115.18
115.04
77.20
77.16
76.99
76.78
N
O
F
3g, Scheme 2
f1 (ppm)

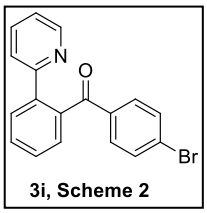

acylation NMR/AH-596-5
AH-596 1H-NMR
8.360
8.356
8.354
8.353
7.795
7.782
7.644
7.639
7.635
7.634
7.629
7.627
7.625
7.621
7.617
7.613
7.603
7.600
7.561
7.559
7.548
7.546
7.544
7.538
7.536
7.531
7.527
7.523
7.276
7.257
7.254
7.250
7.242
7.240
7.236
7.068
7.067
7.060
7.059
7.056
7.054
7.048
7.046

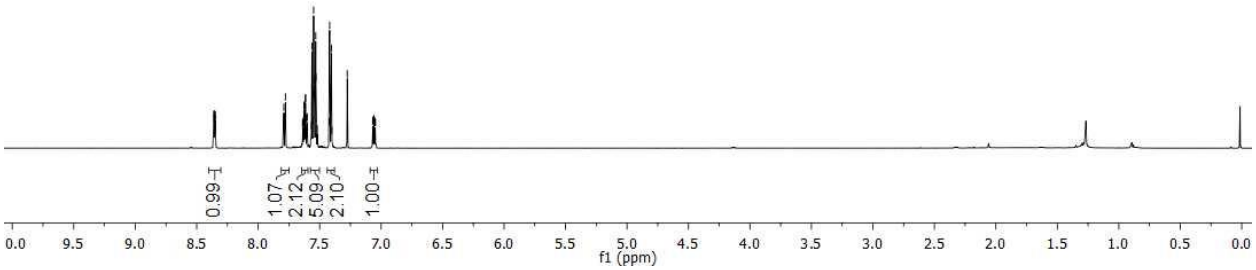

N
O
Cl
3h, Scheme 2

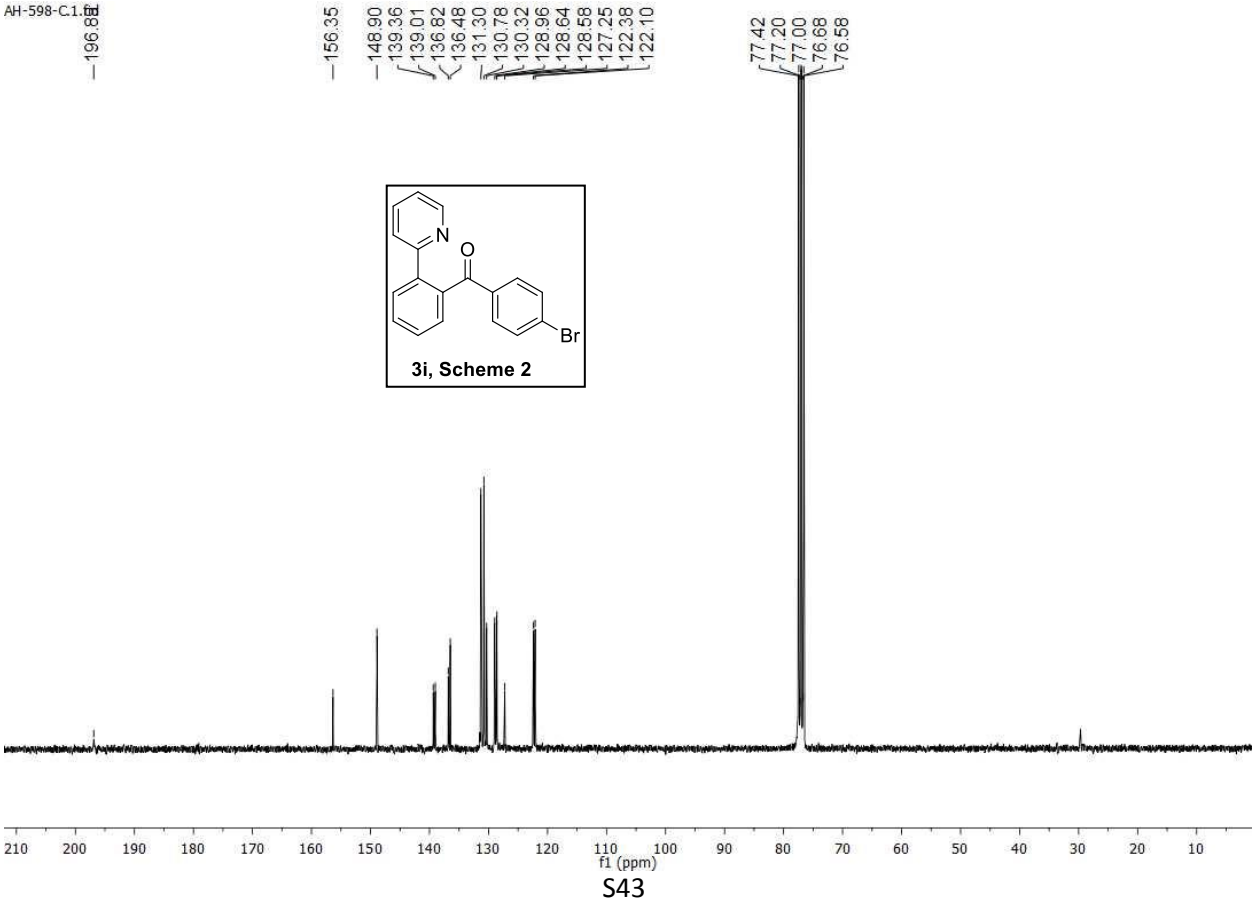

0.99
1.05
4.00
3.06
2.03
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

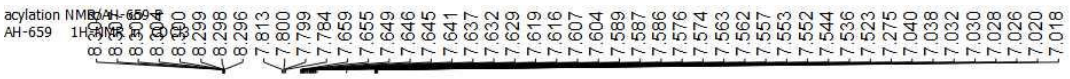

AH-596-C.1.fid
196.96
156.45
148.98
139.44
139.07
138.55
136.45
136.41
130.70
130.34
129.00
128.64
128.34
122.44
122.09
77.42
77.00
76.58
N
O
Cl
3h, Scheme 2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

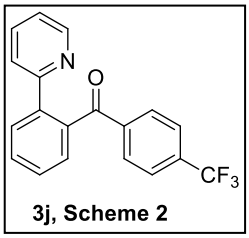

acylation NMR/AH-598-P
AH-598
8.353
8.351
8.350
8.349
7.791
7.778
7.639
7.636
7.630
7.628
7.625
7.623
7.617
7.615
7.615
7.612
7.605
7.602
7.567
7.563
7.560
7.555
7.552
7.549
7.546
7.545
7.543
7.541
7.532
7.530
7.519
7.516
7.422
7.419
7.415
7.407
7.404
7.400
7.276
7.069
7.067
7.061
7.059
7.056
7.055
7.048
7.047


N
O
Br
3i, Scheme 2

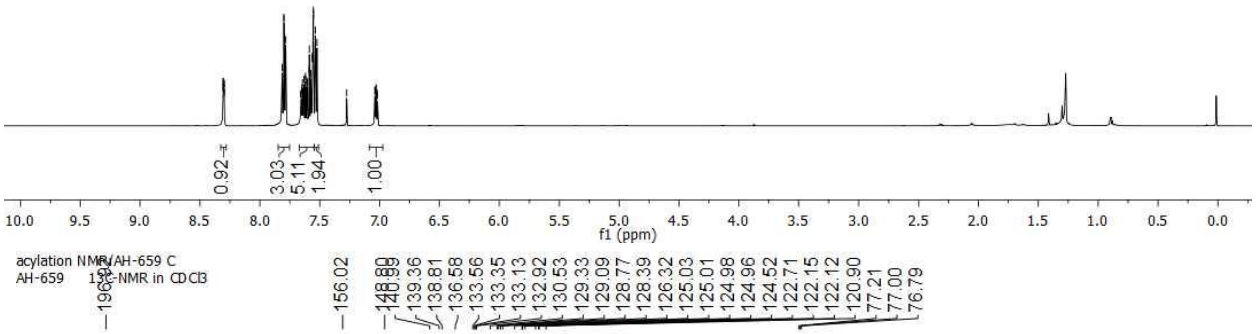

0.99
1.07
2.12
5.09
2.10
1.00
0.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

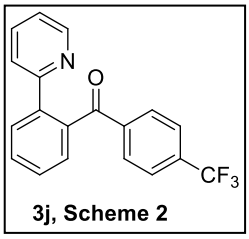

AH-598-C.1.fid
196.80
156.35
148.90
139.36
139.01
136.82
136.48
131.30
130.78
130.32
128.96
128.64
128.58
127.25
122.38
122.10
77.42
77.20
77.00
76.68
76.58
N
O
Br
3i, Scheme 2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)



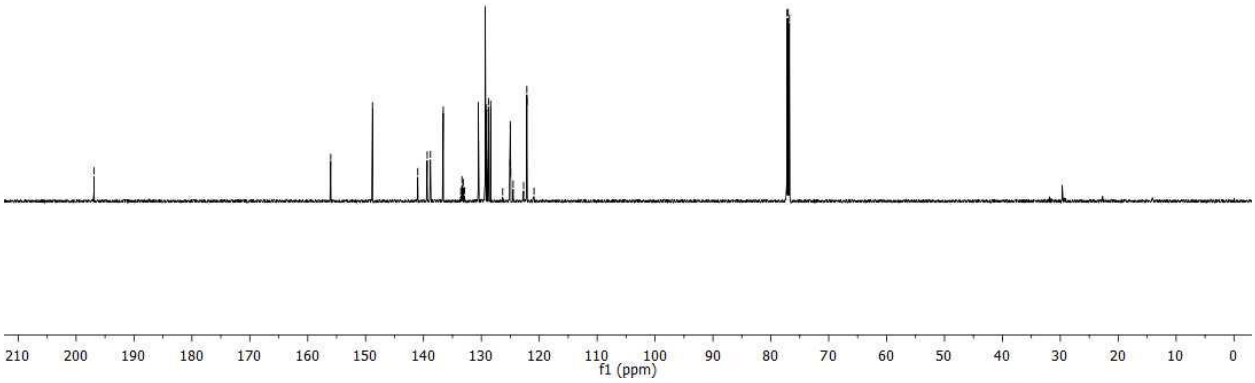


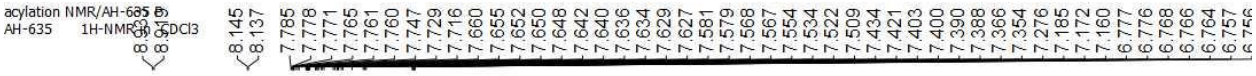
3j, Scheme 2

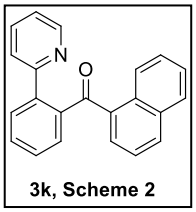




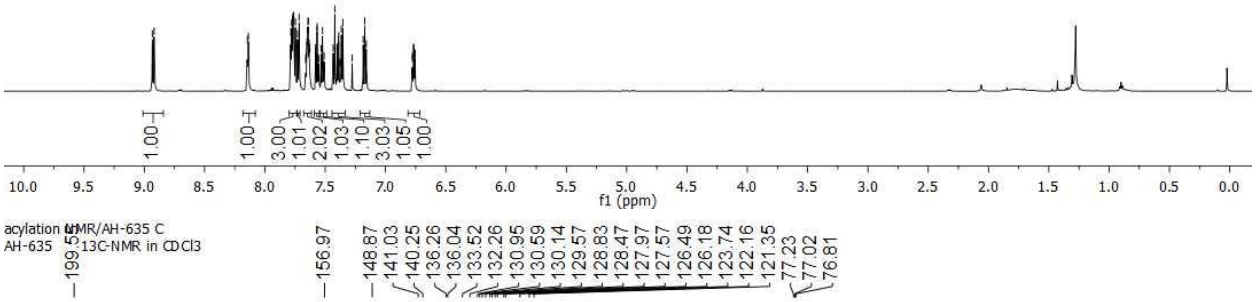
3j, Scheme 2

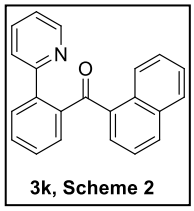




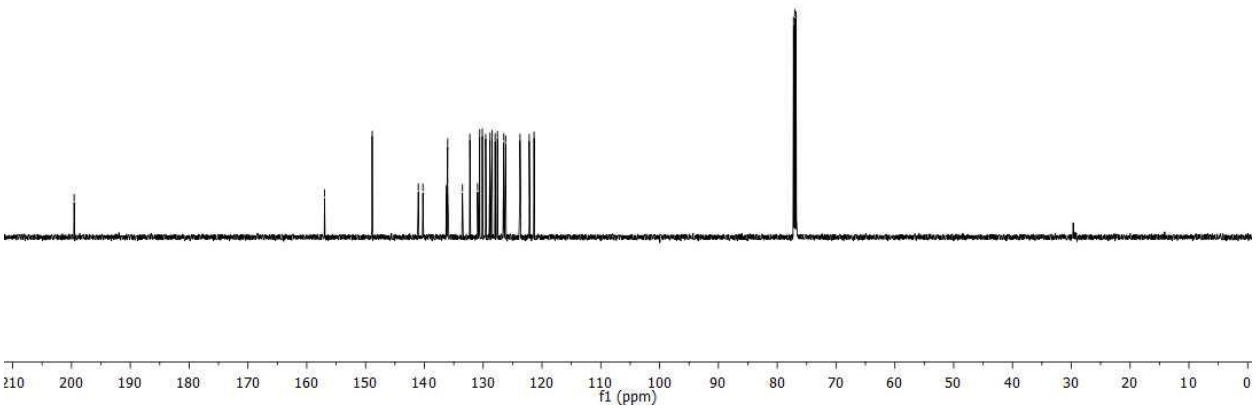


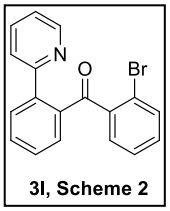
3k, Scheme 2



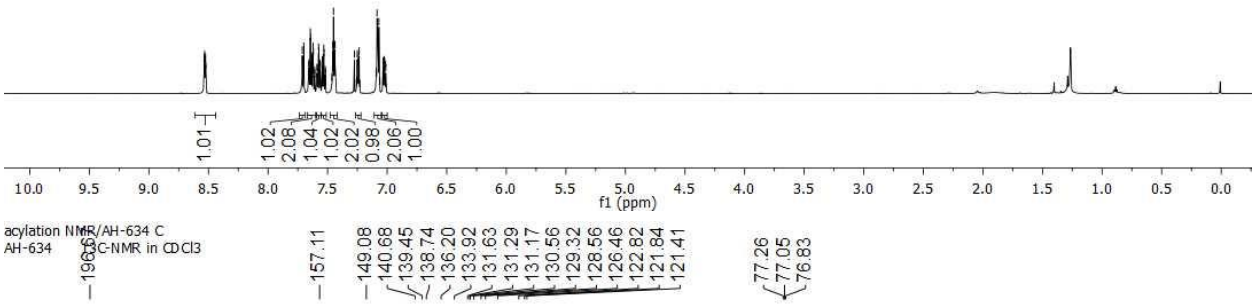


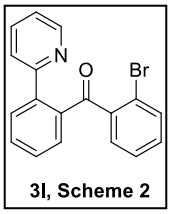
3k, Scheme 2



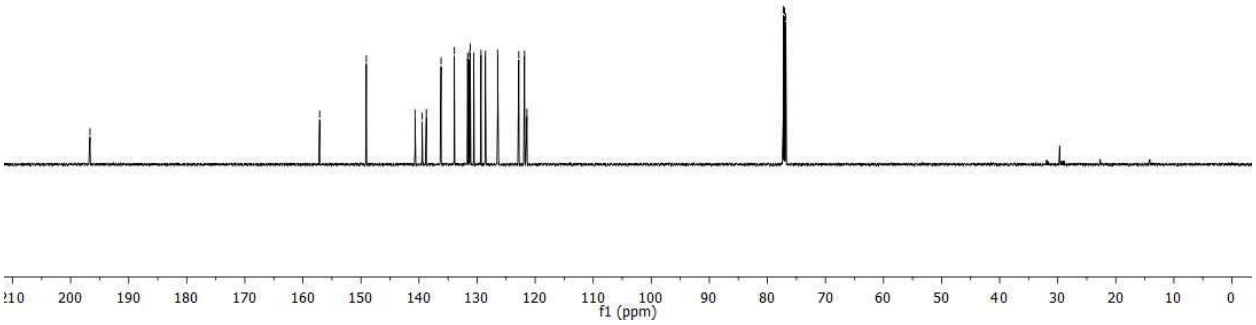


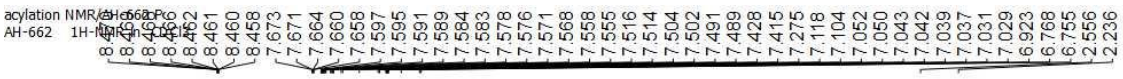
3l, Scheme 2

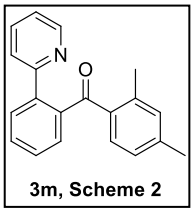




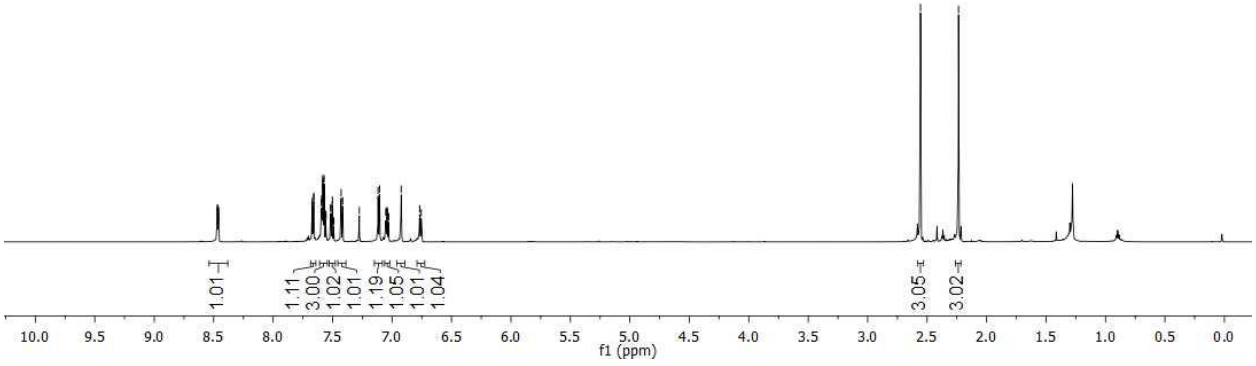
3l, Scheme 2

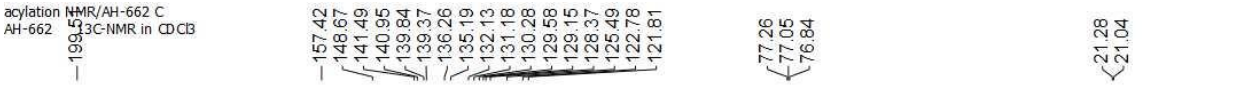


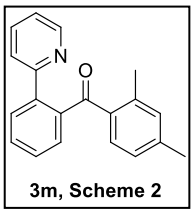




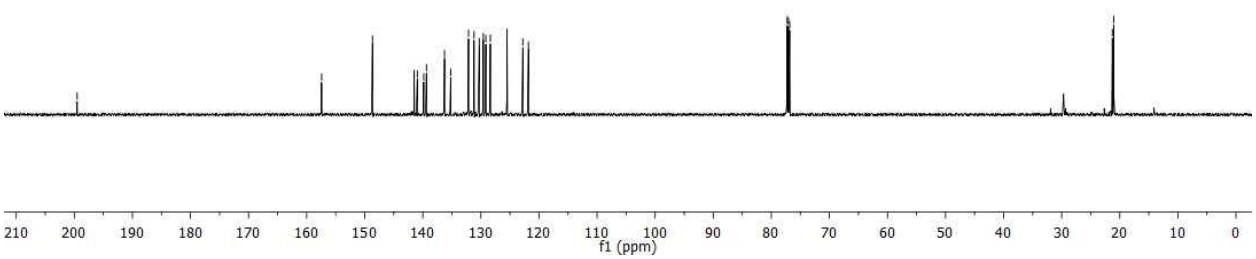
3m, Scheme 2

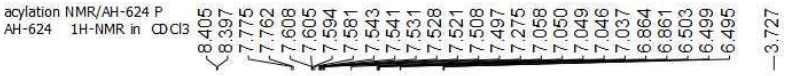


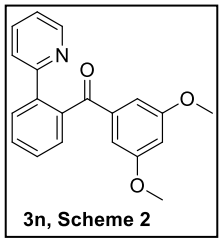


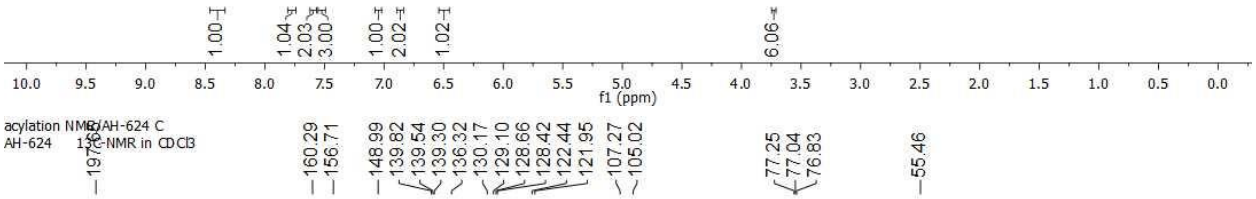


3m, Scheme 2

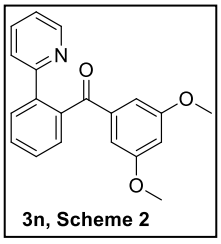


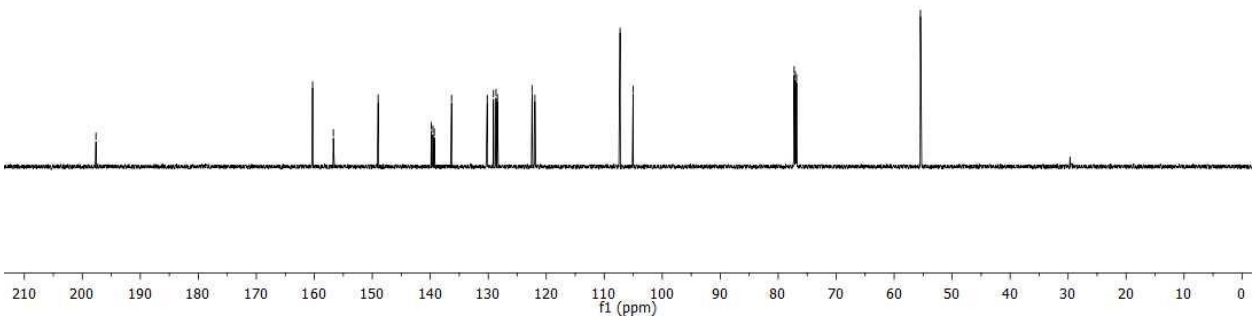

acylation NMR/AH-624 P
AH-624 1H-NMR in CDCl3
8.405
8.397
7.775
7.762
7.608
7.605
7.594
7.581
7.543
7.541
7.531
7.528
7.521
7.508
7.497
7.275
7.058
7.050
7.049
7.046
7.037
6.864
6.861
6.503
6.499
6.495
3.727

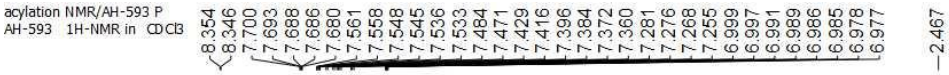

N
O
O
O
3n, Scheme 2

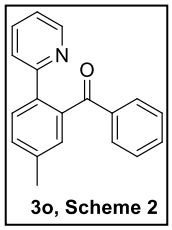

1.00
1.04
2.03
3.00
1.00
2.02
1.02
6.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

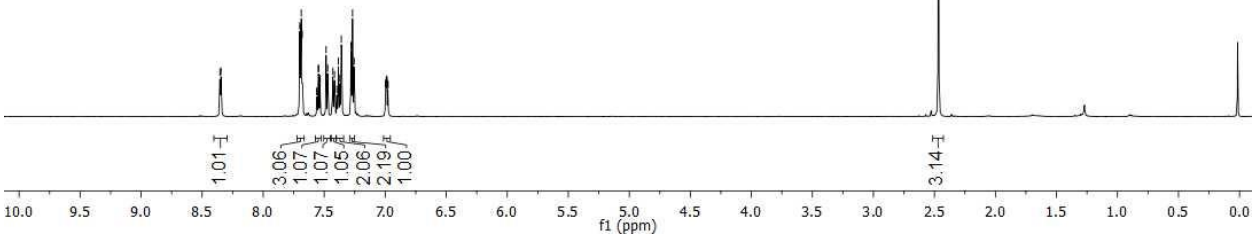
acylation NMR/AH-624 C
AH-624 13C-NMR in CDCl3
197.48
160.29
156.71
148.99
139.82
139.54
139.30
136.32
130.17
129.10
128.66
128.42
122.44
121.95
107.27
105.02
77.25
77.04
76.83
55.46

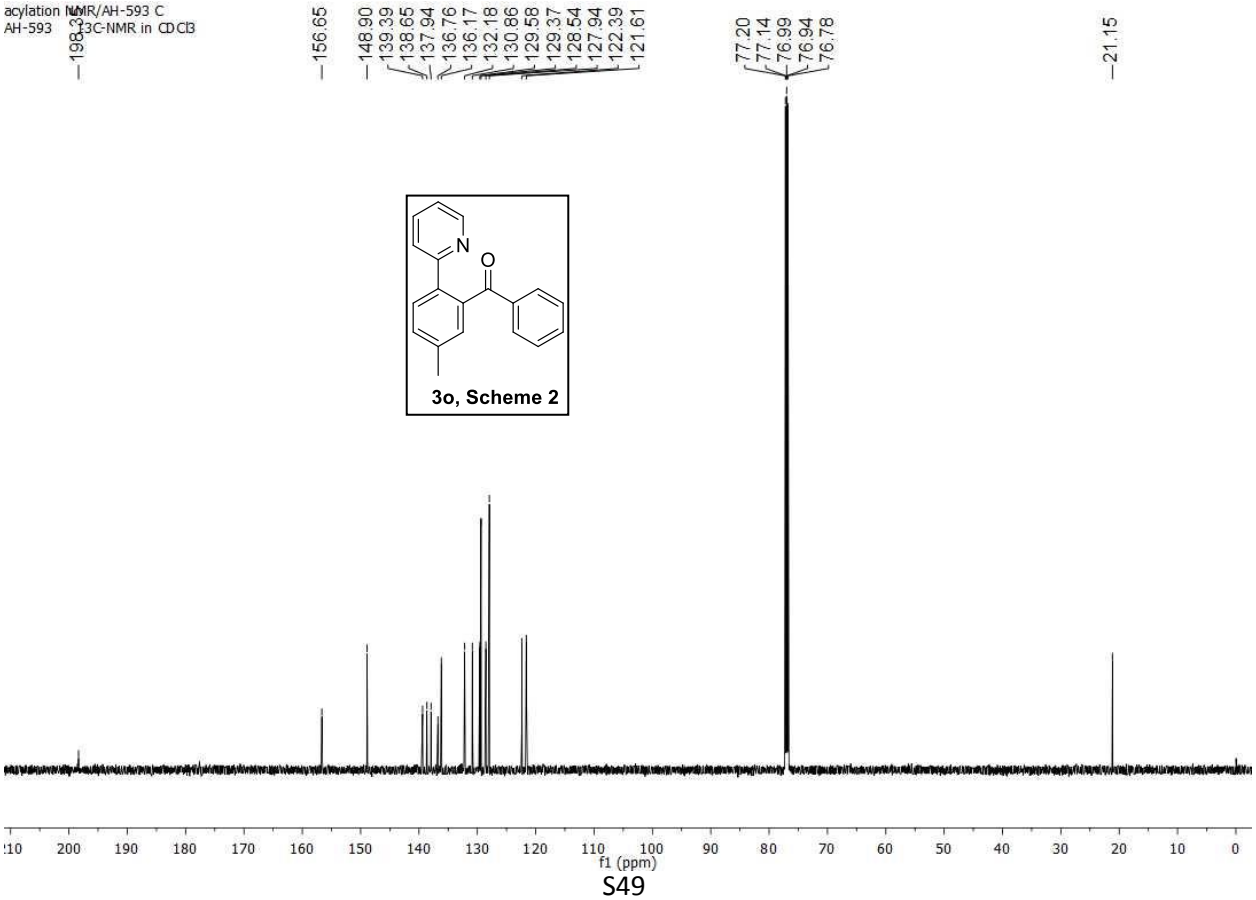

N
O
O
O
3n, Scheme 2

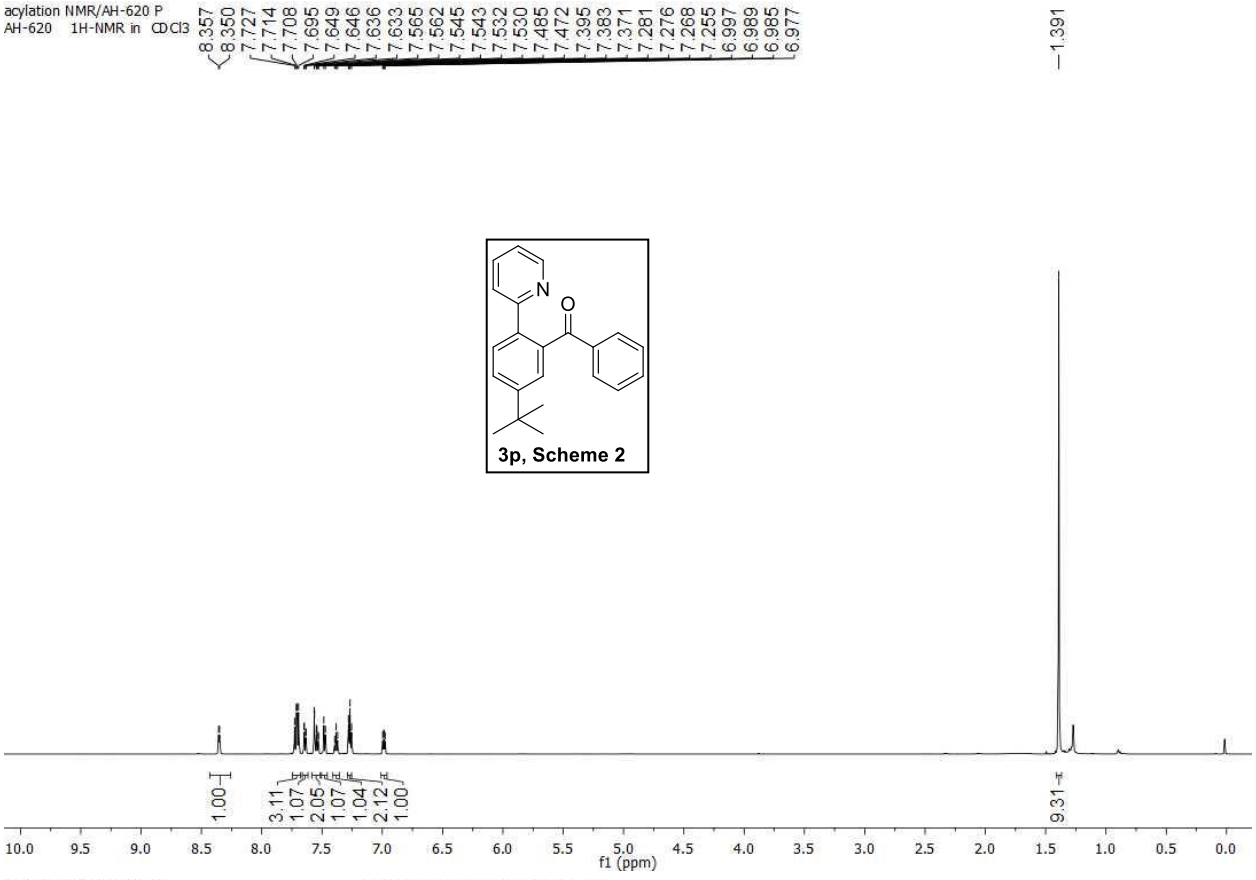

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

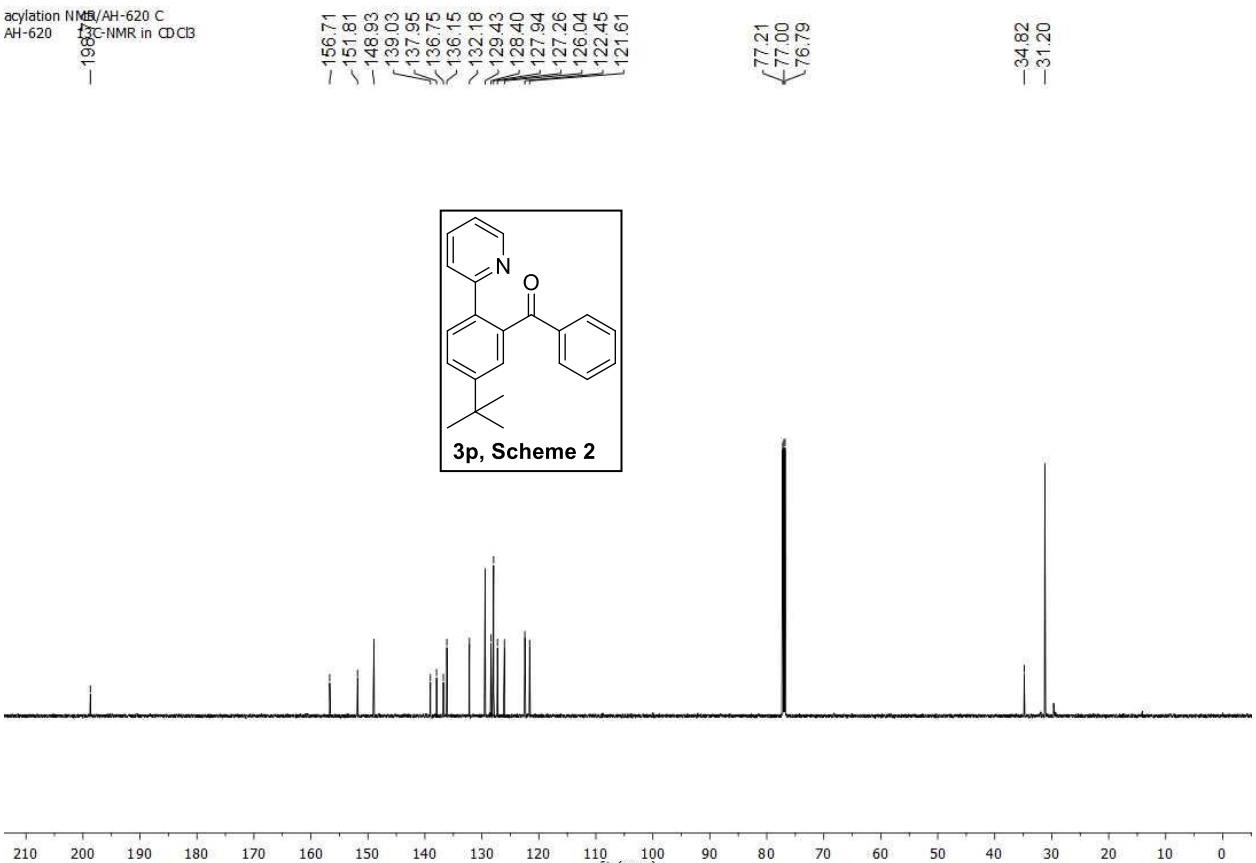

acylation NMR/AH-593 P
AH-593 1H-NMR in CDCl3
8.354
8.346
7.700
7.693
7.688
7.686
7.680
7.561
7.558
7.548
7.545
7.536
7.533
7.484
7.471
7.429
7.416
7.396
7.384
7.372
7.360
7.281
7.276
7.268
7.255
6.999
6.997
6.991
6.989
6.986
6.985
6.978
6.977
2.467

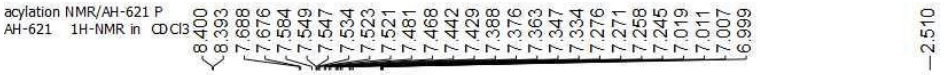

N
O
3o, Scheme 2

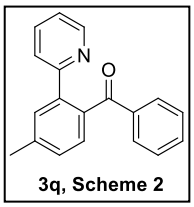

1.01
3.06
1.07
1.07
1.05
2.06
2.19
1.00
3.14
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


acylation NMR/AH-593 C
AH-593 13C-NMR in CDCl3
198.31
156.65
148.90
139.39
138.65
137.94
136.76
136.17
132.18
130.86
129.58
129.37
128.54
127.94
122.39
121.61
77.20
77.14
76.99
76.94
76.78
21.15
N
O
3o, Scheme 2
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

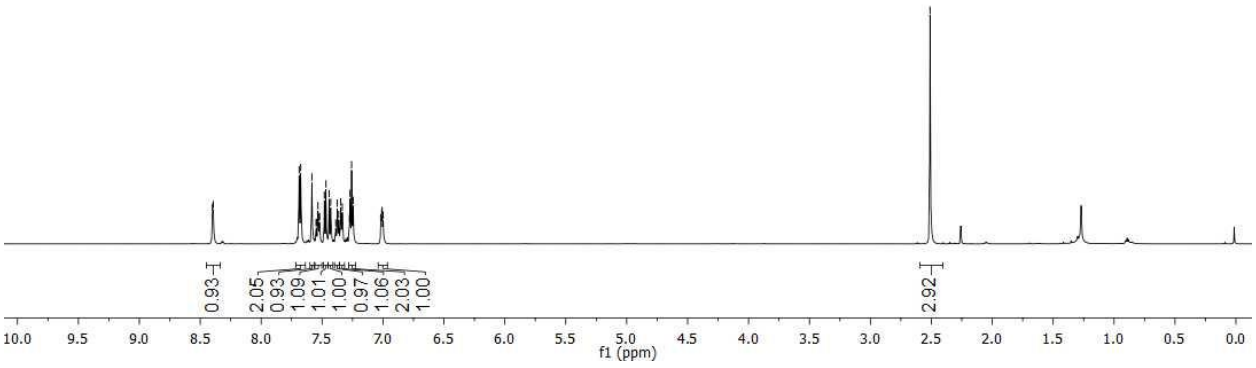

acylation NMR/AH-620 P
AH-620 1H-NMR in CDCl3
8.357
8.350
7.727
7.714
7.708
7.695
7.649
7.646
7.636
7.633
7.565
7.562
7.545
7.543
7.532
7.530
7.485
7.472
7.395
7.383
7.371
7.281
7.276
7.268
7.255
6.997
6.989
6.985
6.977
1.391
3p, Scheme 2
1.00
3.11
1.07
2.05
1.07
1.04
2.12
1.00
9.31
f1 (ppm)

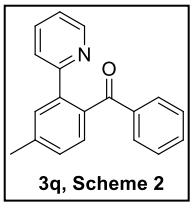

acylation NMR/AH-620 C
AH-620 13C-NMR in CDCl3
198.47
156.71
151.81
148.93
139.03
137.95
136.75
136.15
132.18
129.43
128.40
127.94
127.26
126.04
122.45
121.61
77.21
77.00
76.79
34.82
31.20
3p, Scheme 2
f1 (ppm)



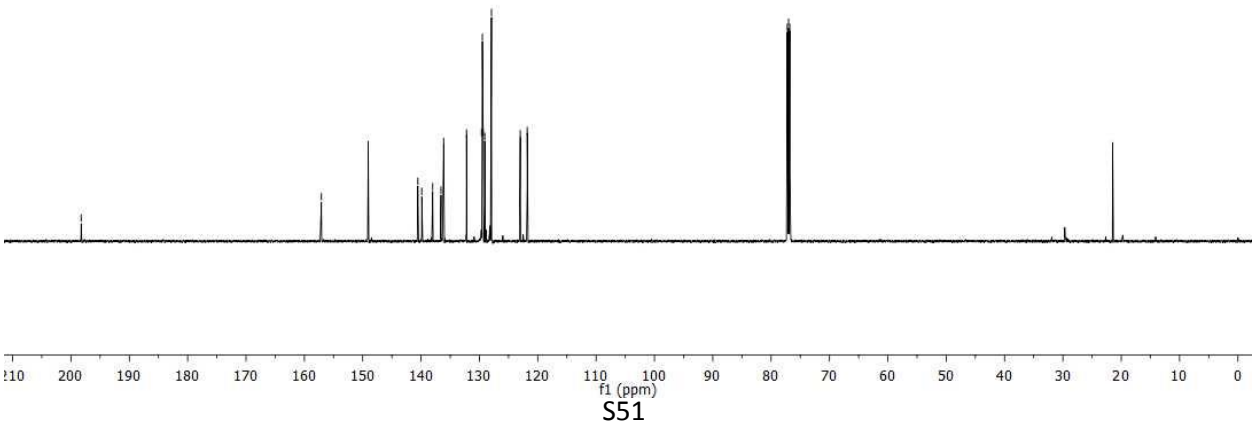


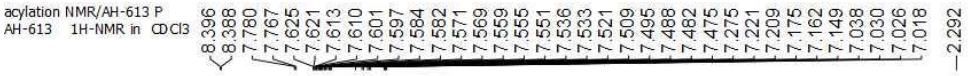
3q, Scheme 2

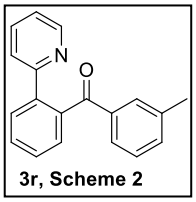




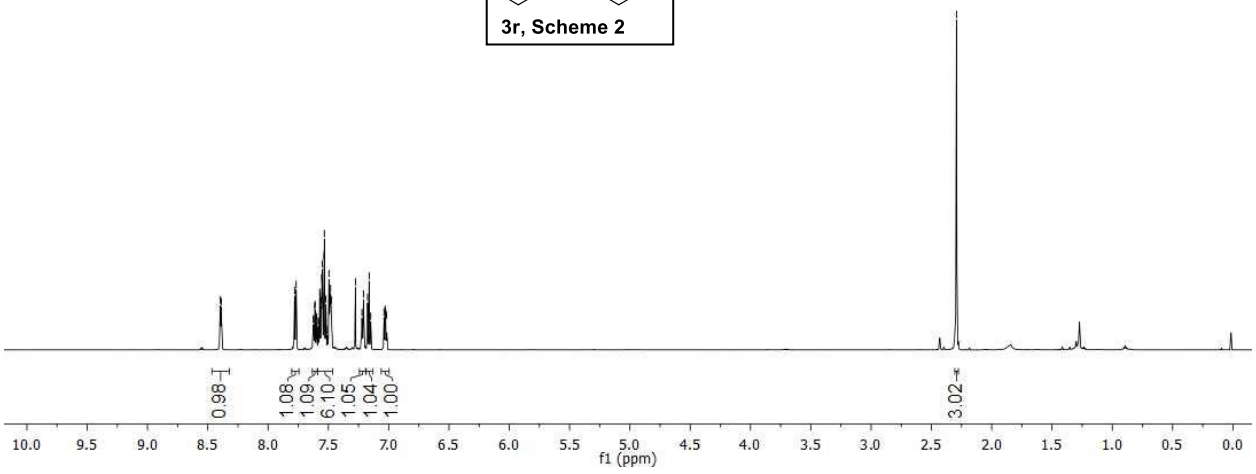
3q, Scheme 2

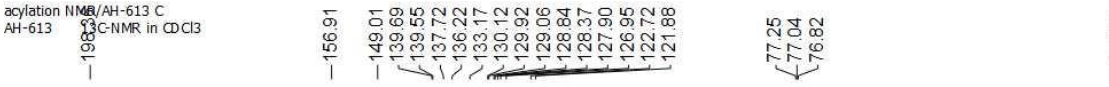


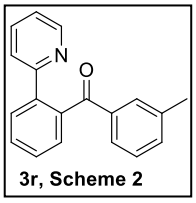




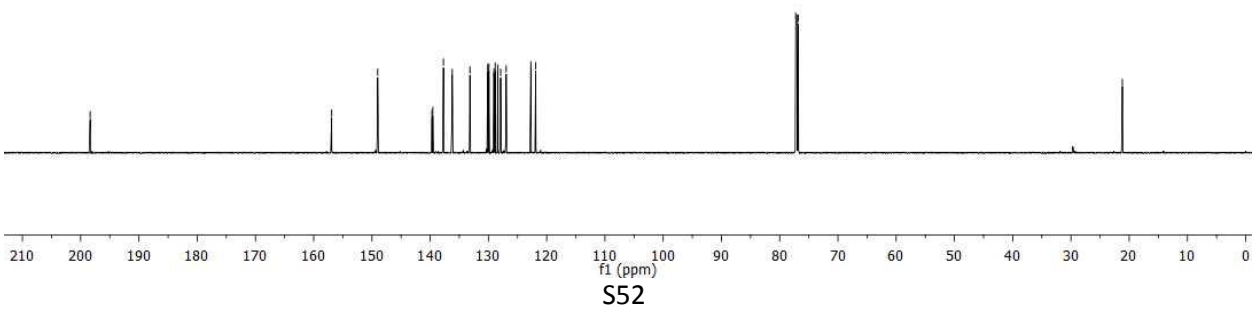
3r, Scheme 2



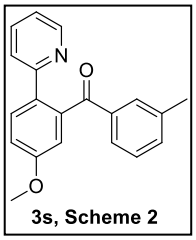


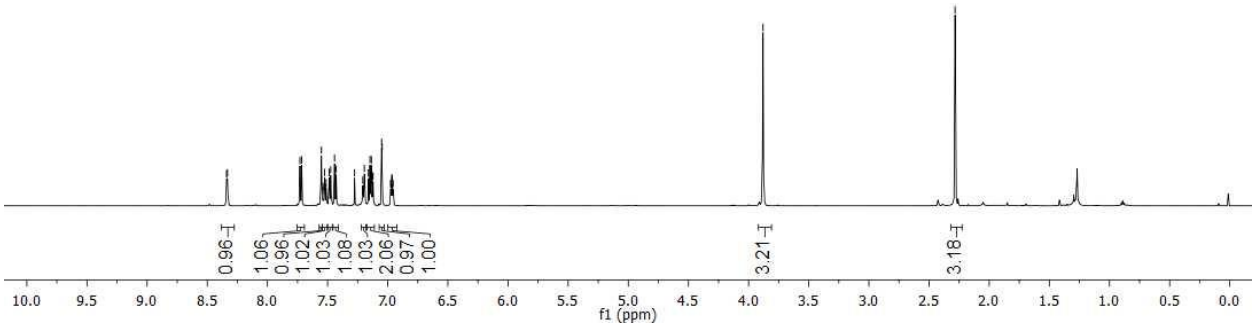


3r, Scheme 2

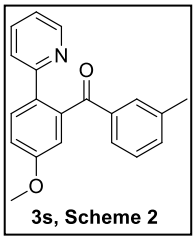


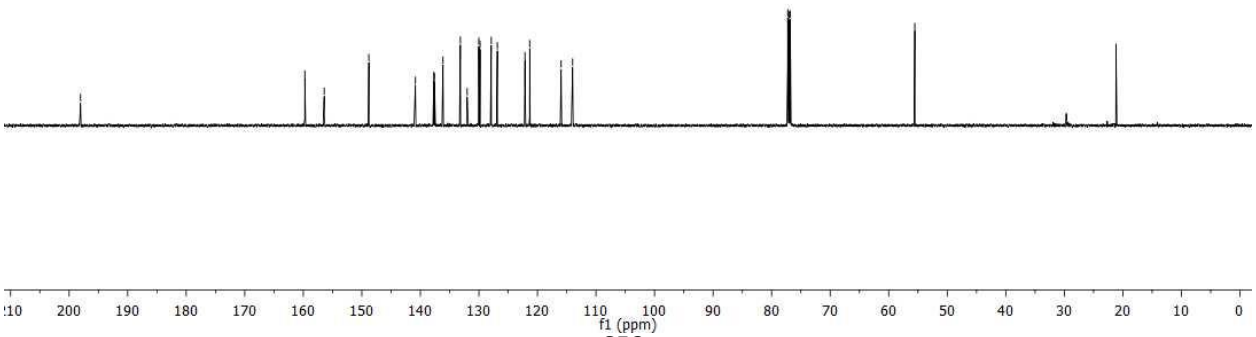

acylation NMR/AH-619 P
AH-619 1H-NMR in CDCl3
8.340
8.332
7.729
7.715
7.551
7.540
7.538
7.525
7.515
7.512
7.486
7.474
7.442
7.429
7.276
7.207
7.194
7.160
7.148
7.140
7.135
7.125
7.122
7.050
7.046
6.975
6.974
6.967
6.965
6.963
6.955
3.880
2.284

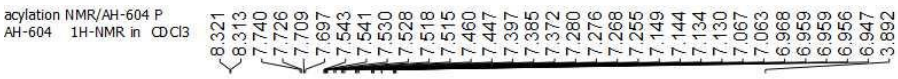

N
O
O
3s, Scheme 2

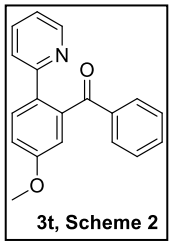

0.96
1.06
0.96
1.02
1.03
1.08
1.03
2.06
0.97
1.00
3.21
3.18
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

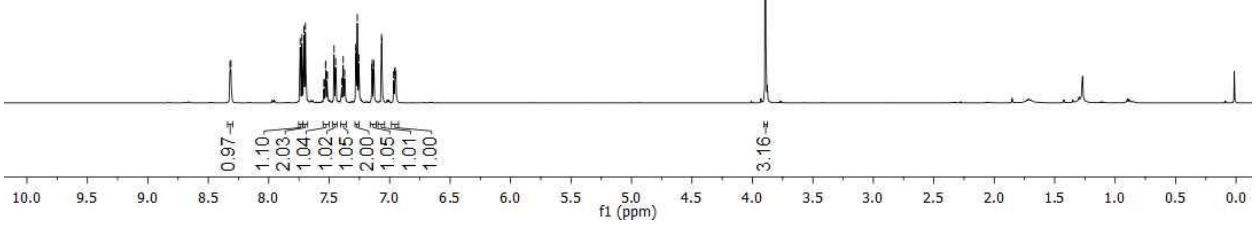

acylation NMR/AH-619 C
AH-619 13C-NMR in CDCl3
198.03
159.74
156.42
148.80
140.85
137.71
137.59
136.14
133.17
132.01
130.02
129.81
127.89
126.85
122.13
121.32
115.96
113.99
77.24
77.03
76.82
55.54
21.16


N
O
O
3s, Scheme 2

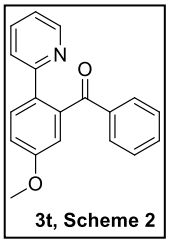

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

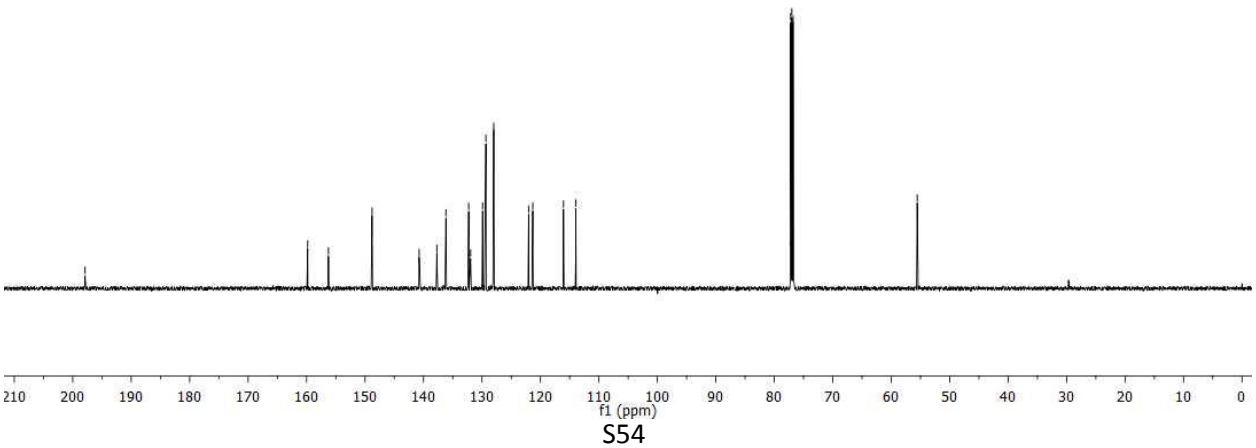

acylation NMR/AH-604 P
AH-604 1H-NMR in CDCl3
8.321
8.313
7.740
7.726
7.709
7.697
7.543
7.541
7.530
7.528
7.518
7.515
7.460
7.447
7.397
7.385
7.372
7.280
7.276
7.268
7.255
7.149
7.144
7.134
7.130
7.067
7.063
6.968
6.959
6.959
6.956
6.947
3.892


N
O
O
3t, Scheme 2

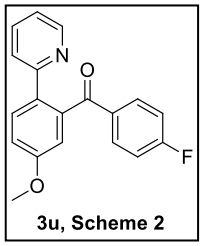

0.97
1.10
2.03
1.04
1.02
1.05
2.00
1.05
1.01
1.00
3.16
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

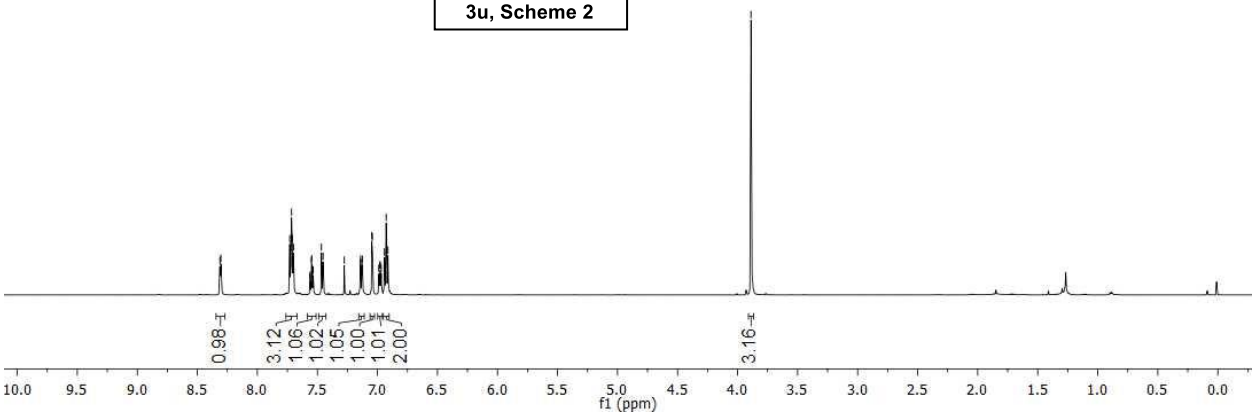

acylation NMR/AH-604 C
AH-604 13C-NMR in CDCl3
197.88
159.82
156.26
148.80
140.75
137.72
136.17
132.27
131.95
129.90
129.33
127.98
122.02
121.31
116.06
113.95
77.21
77.17
77.00
76.79
55.55


N
O
O
3t, Scheme 2

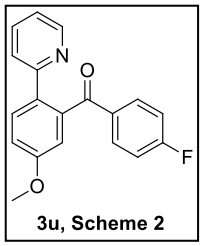

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

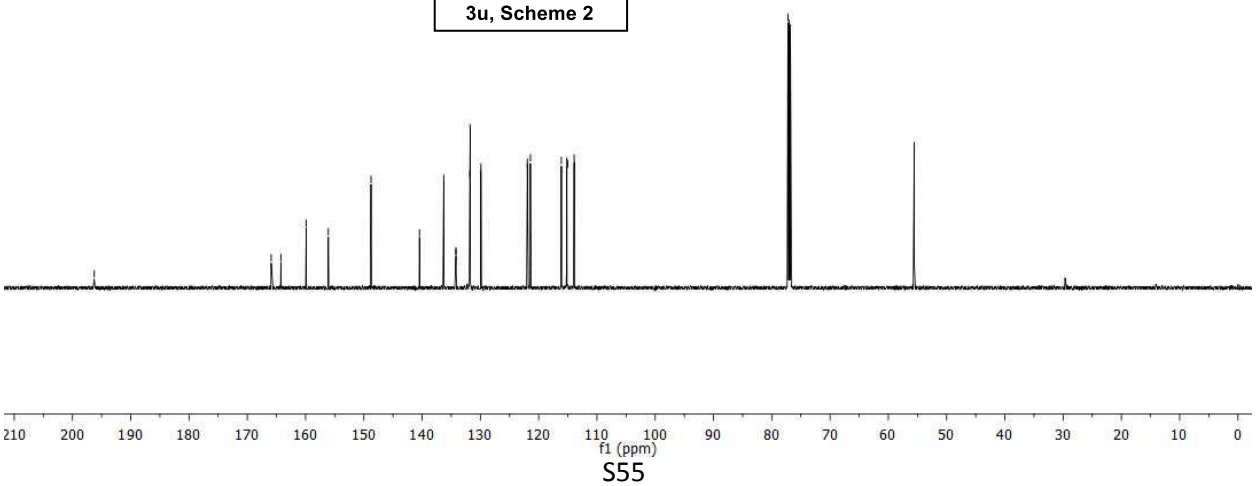

acylation NMR/AH-617 P
AH-617 1H-NMR in CDCl3
8.312
8.304
7.730
7.723
7.715
7.709
7.699
7.563
7.560
7.550
7.548
7.537
7.535
7.466
7.453
7.276
7.144
7.140
7.130
7.125
7.047
7.043
6.988
6.980
6.976
6.968
6.941
6.927
6.913
3.888

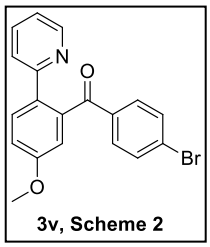

N
O
F
O
3u, Scheme 2


0.98
3.12
1.06
1.02
1.05
1.00
1.01
2.00
3.16
10.0 9.5 9.0 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
f1 (ppm)

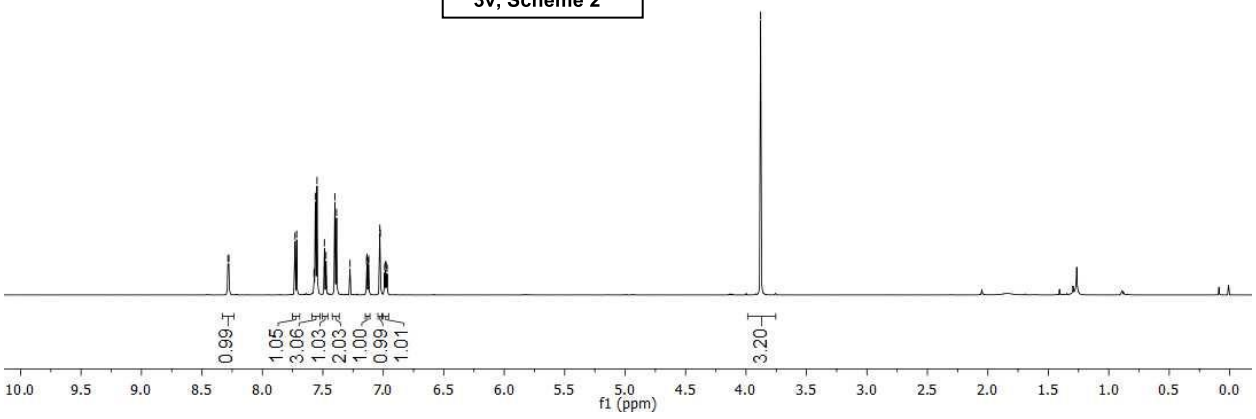

acylation NMR/AH-617 C
AH-617 13C-NMR in CDCl3
196.52
165.91
164.23
159.90
156.11
148.77
140.46
136.29
134.22
134.20
131.82
131.76
129.91
121.94
121.43
116.10
115.17
115.03
113.91
77.22
77.01
76.80
55.55

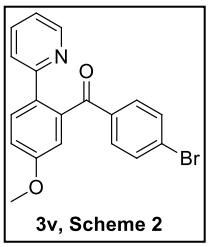

N
O
F
O
3u, Scheme 2


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

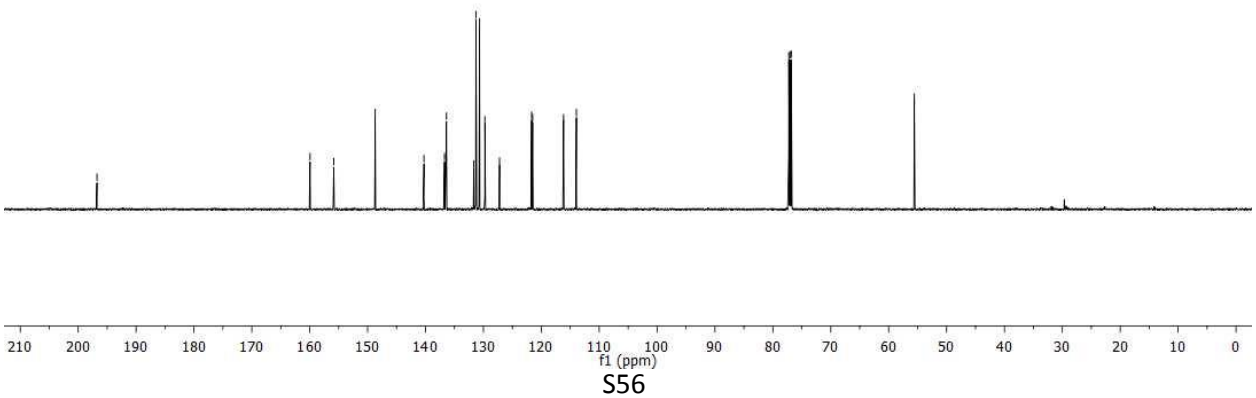


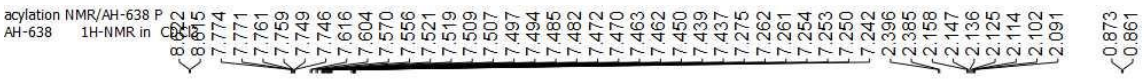
3v, Scheme 2

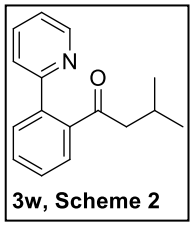




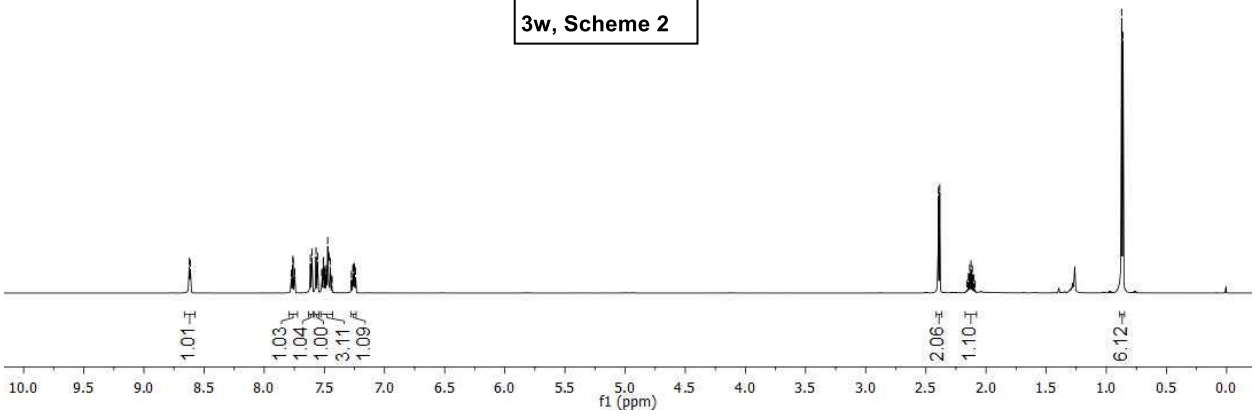
3v, Scheme 2

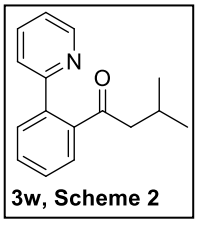




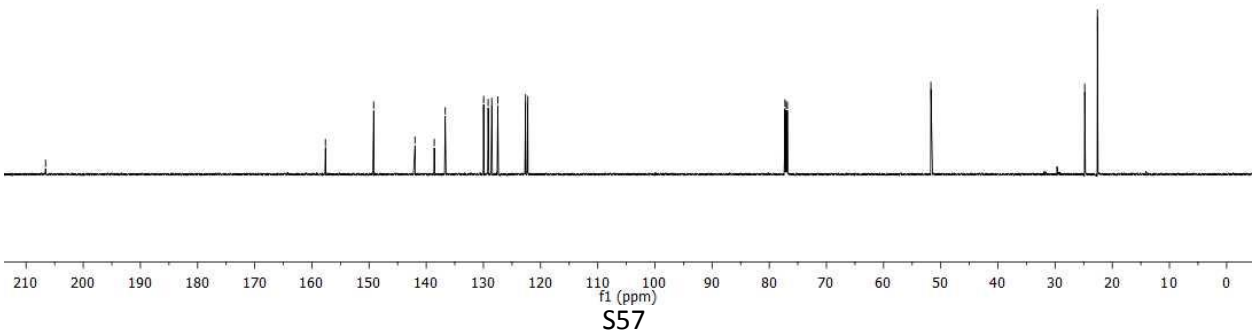


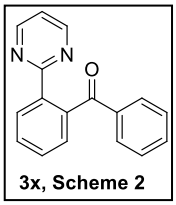
3w, Scheme 2



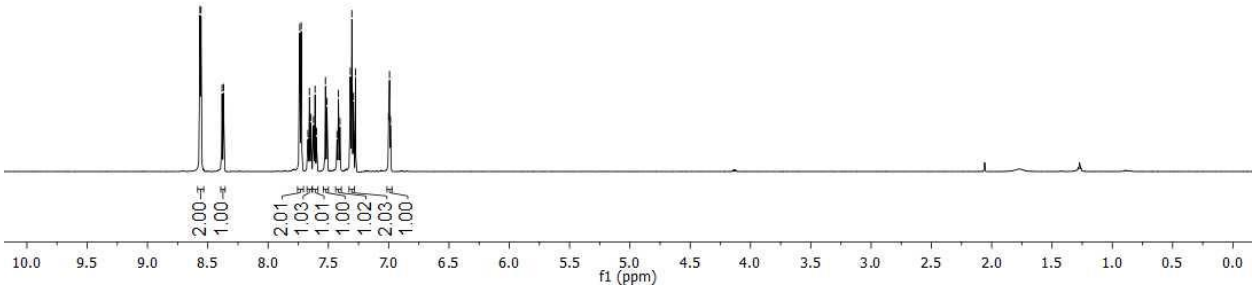


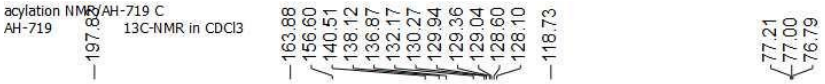
3w, Scheme 2

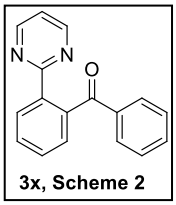




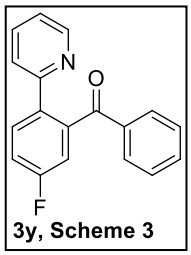
3x, Scheme 2



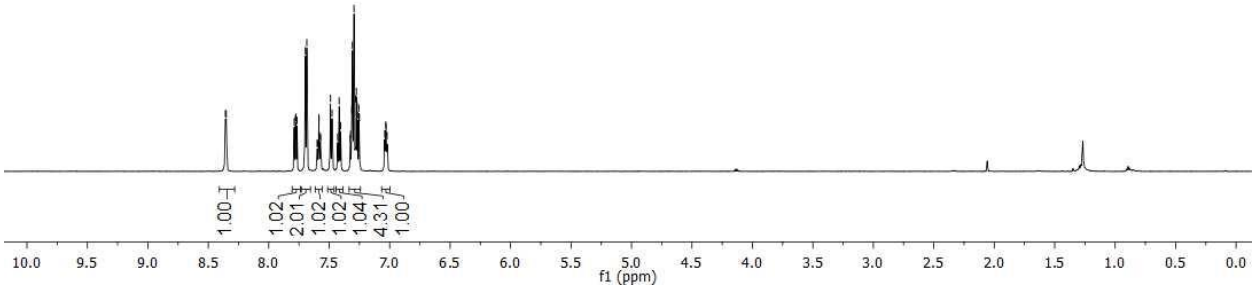


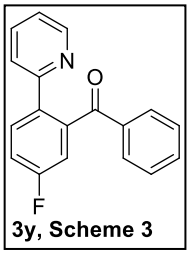


3x, Scheme 2

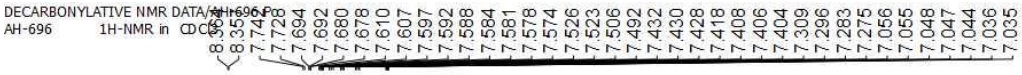


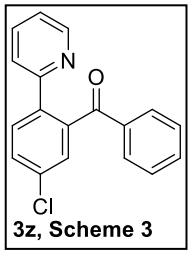
3y, Scheme 3



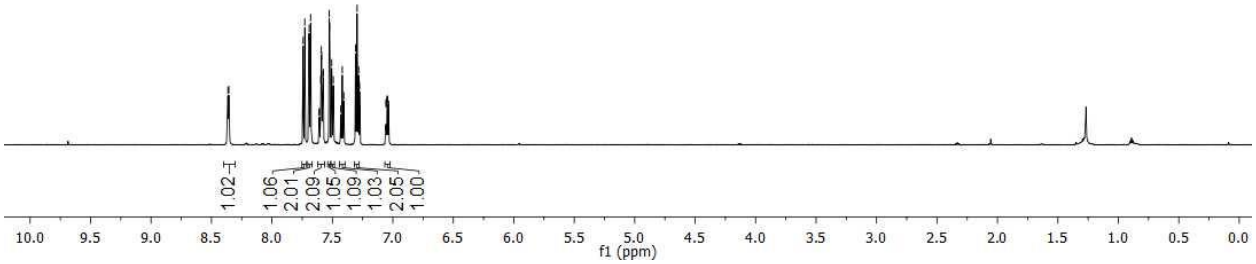


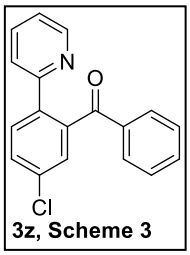
3y, Scheme 3



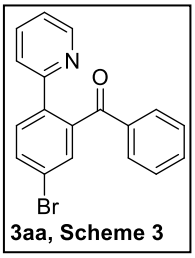


3z, Scheme 3

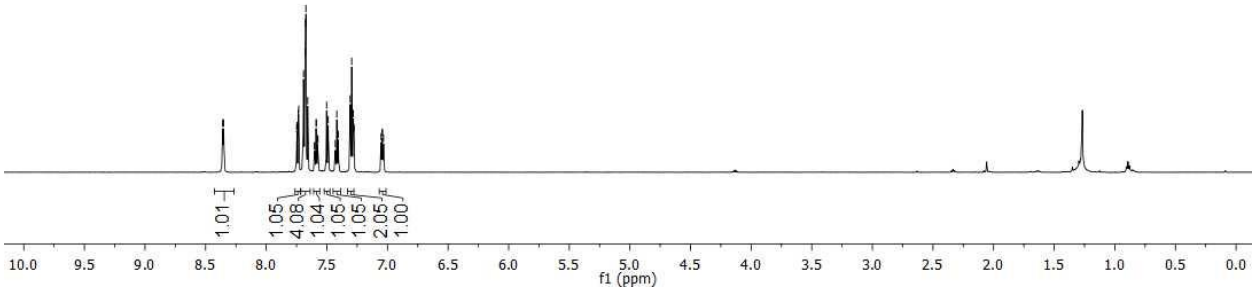


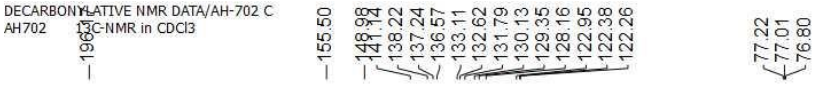


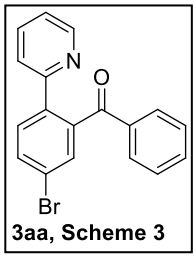
3z, Scheme 3



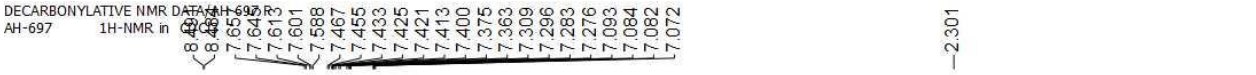
3aa, Scheme 3

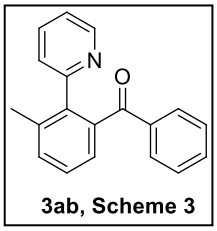




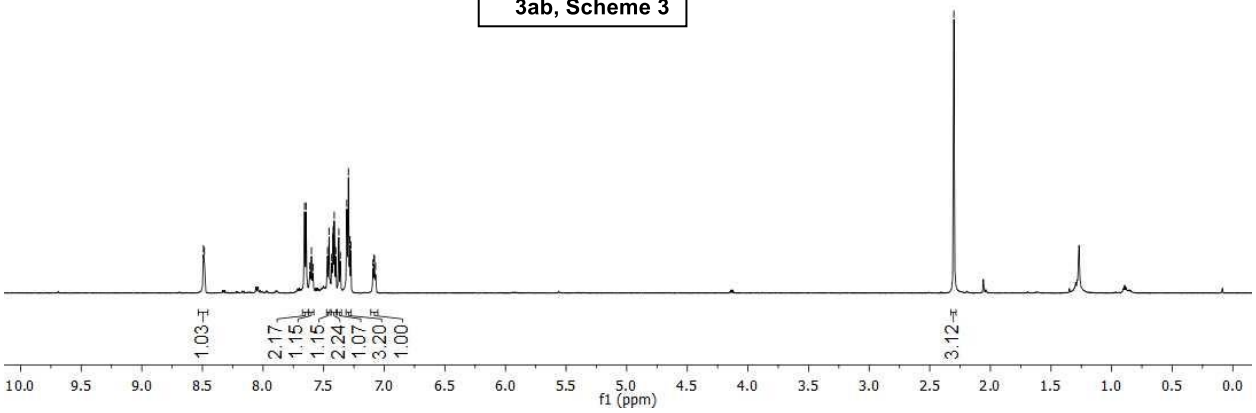


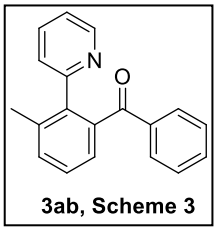
3aa, Scheme 3





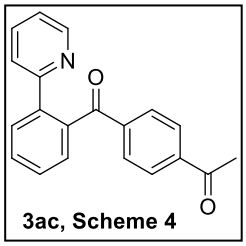
3ab, Scheme 3

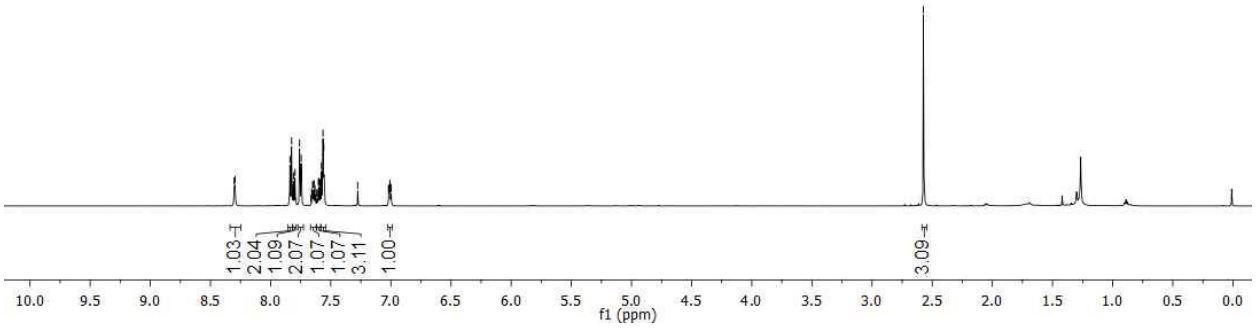




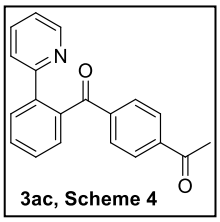
3ab, Scheme 3

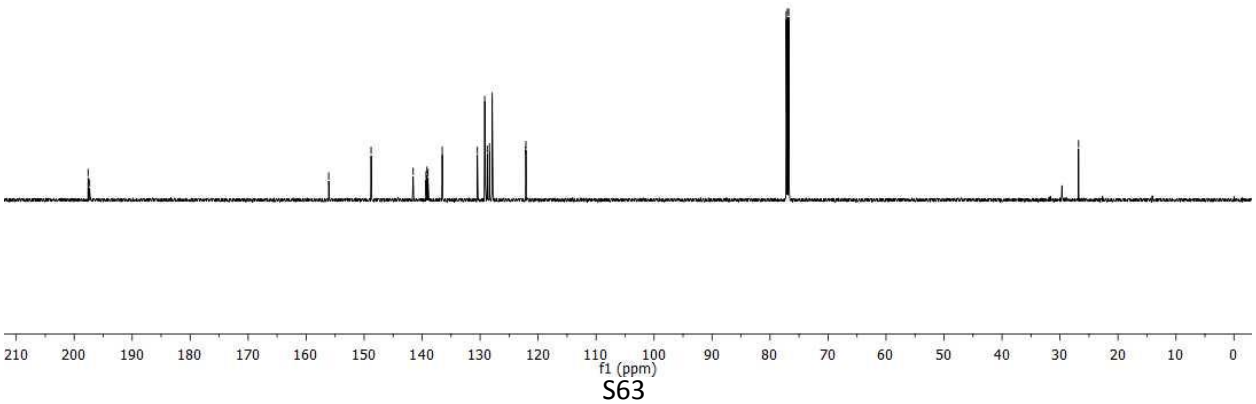

acylation NMR/AH-639 P
AH-639 1H-NMR in CDCl3
8.303
8.295
7.839
7.825
7.810
7.797
7.759
7.745
7.664
7.658
7.651
7.644
7.638
7.631
7.624
7.615
7.613
7.602
7.600
7.590
7.587
7.577
7.564
7.558
7.276
7.019
7.018
7.008
7.006
7.000
6.998
2.573


N
O
O
3ac, Scheme 4


1.03
2.04
1.09
2.07
1.07
3.11
1.00
3.09
10.0
9.5
9.0
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
f1 (ppm)


acylation NMR/AH-639 C
AH-639 13C-NMR in CDCl3
197.61
197.54
156.10
148.80
141.55
139.33
139.17
138.99
136.53
130.48
129.21
129.12
128.76
128.38
127.91
122.16
122.10
77.21
77.00
76.79
26.80


N
O
O
3ac, Scheme 4


210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


acylation NMR/AH-646-P
AH-646 1H-NMR in CDCl3
8.286
8.282
8.280
7.914
7.900
7.791
7.779
7.719
7.705
7.644
7.640
7.638
7.627
7.620
7.616
7.614
7.593
7.590
7.588
7.577
7.575
7.568
7.562
7.559
7.556
7.547
7.545
7.543
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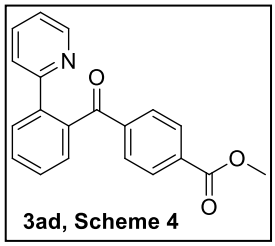
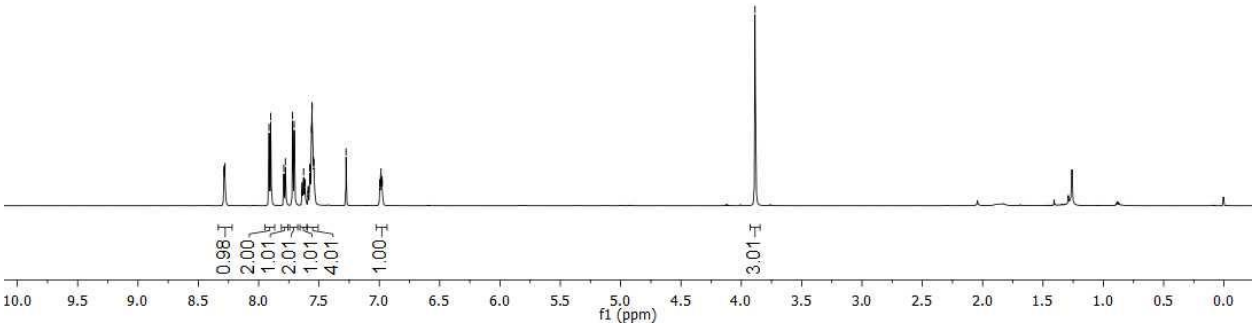
6.995
6.992
6.989
6.986
6.980
6.978
6.975
3.887


N
O
O
O
3ad, Scheme 4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2.00
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2.01
1.01
4.01
1.00
3.01
10.0
9.5
9.0
8.5
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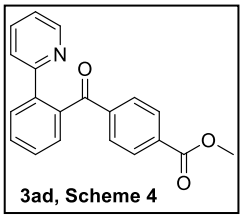
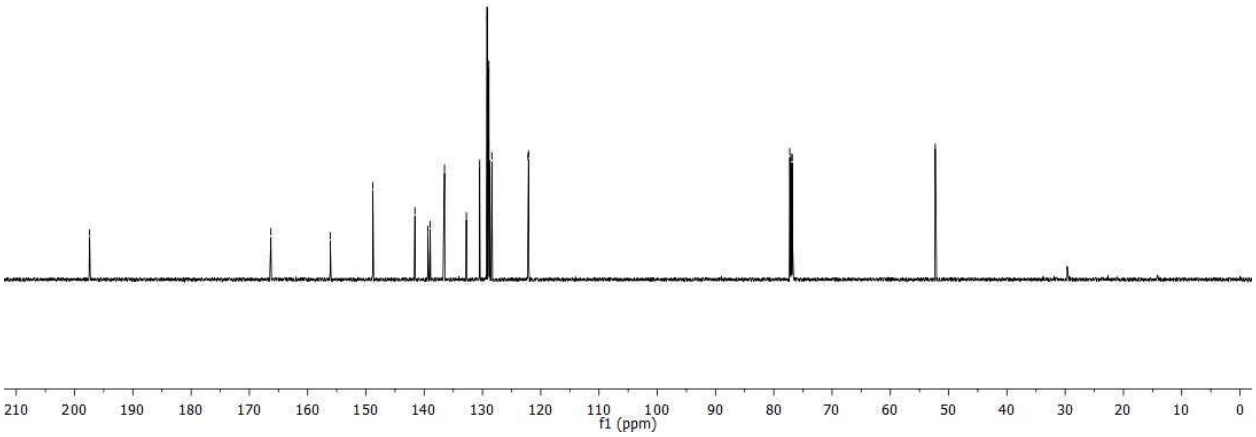
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


acylation NMR/AH-646 C
AH-646 13C-NMR in CDCl3
197.41
166.31
156.12
148.81
141.59
139.37
138.97
136.51
132.76
130.48
129.20
128.90
128.76
128.37
122.18
122.09
77.25
77.04
76.83
52.29


N
O
O
O
3ad, Scheme 4


210
200
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150
140
130
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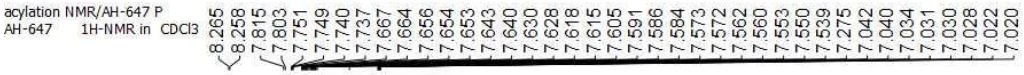
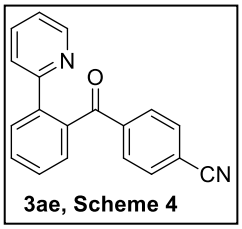
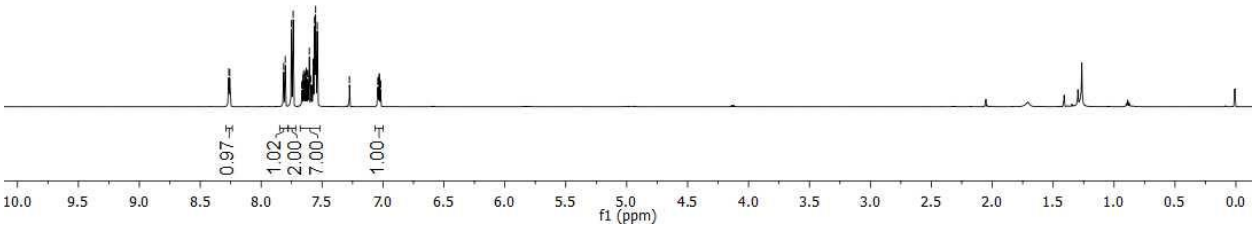
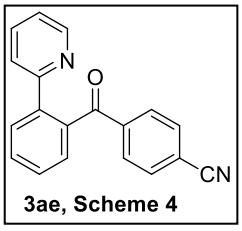
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



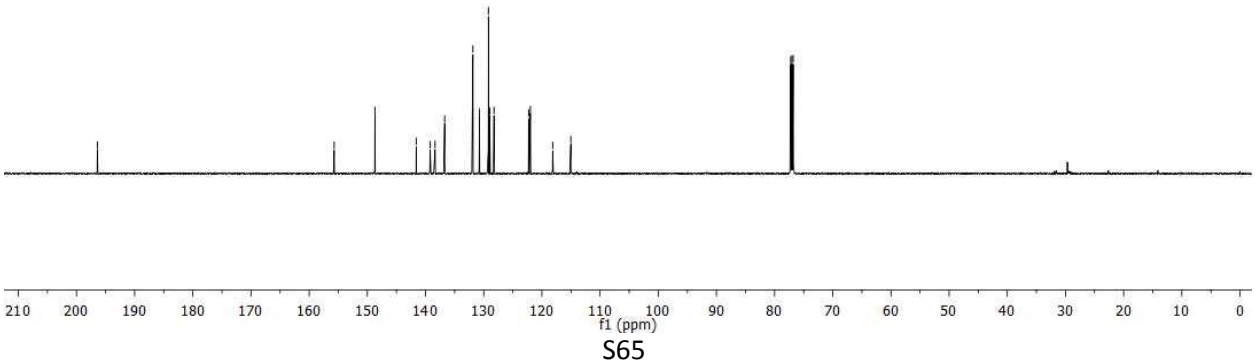


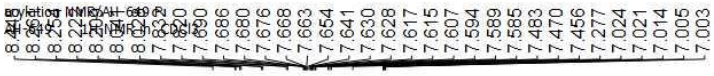
3ae, Scheme 4

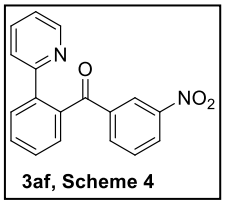




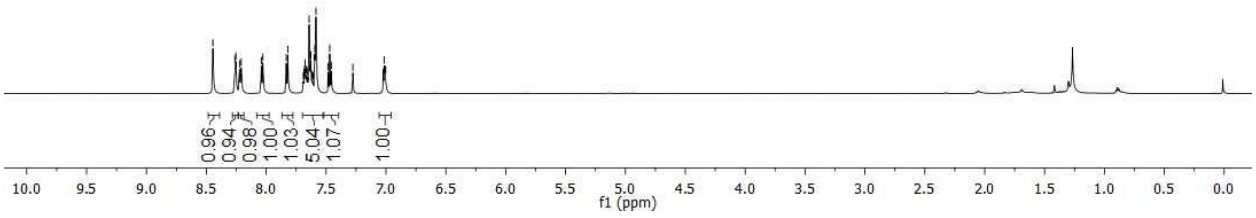
3ae, Scheme 4

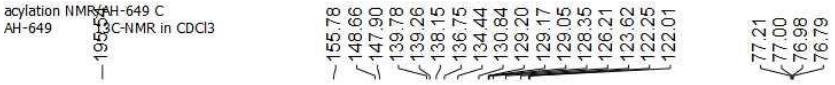


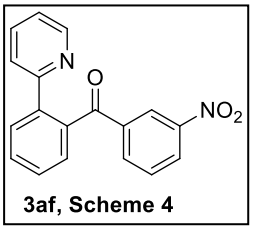




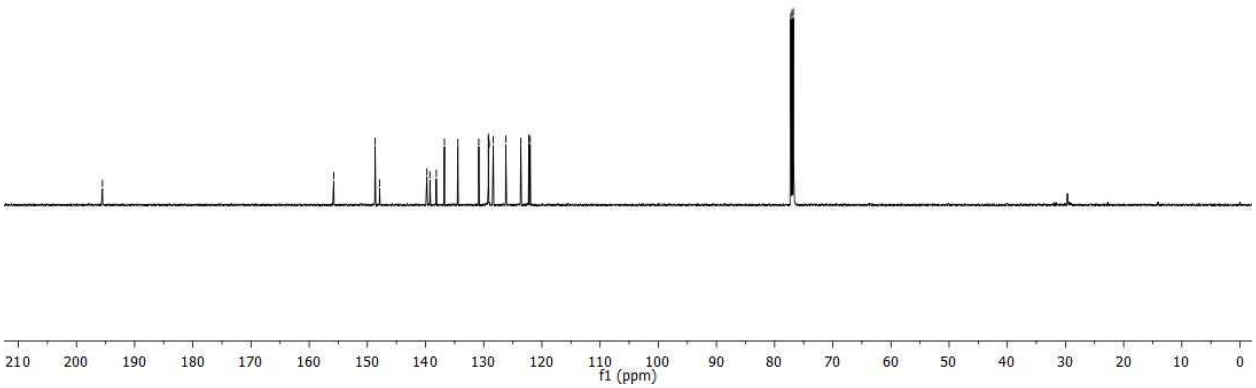
3af, Scheme 4

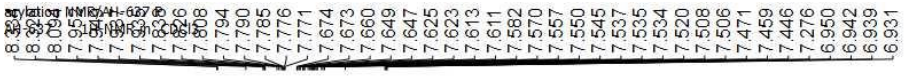


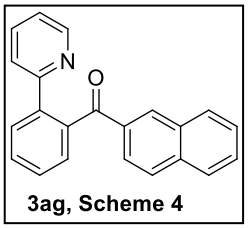




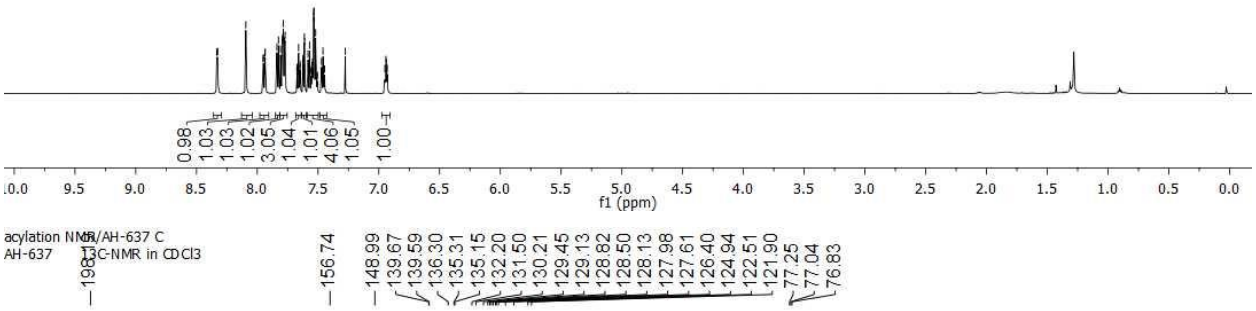
3af, Scheme 4

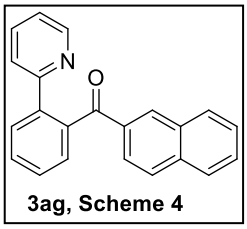




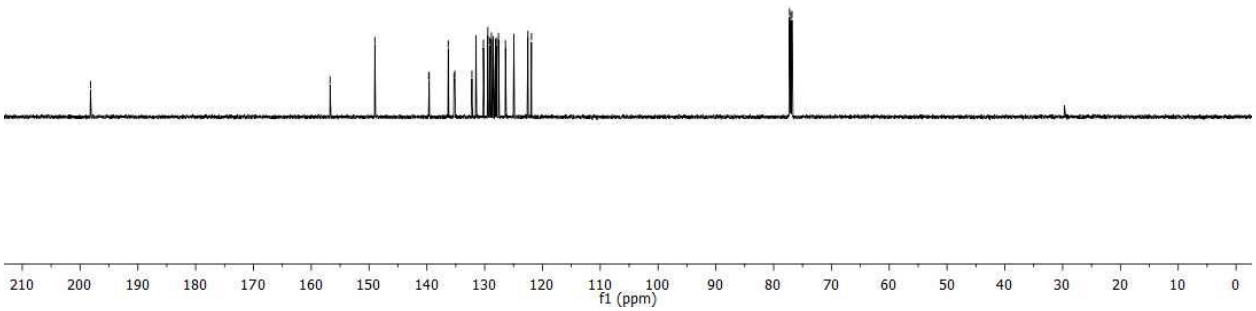


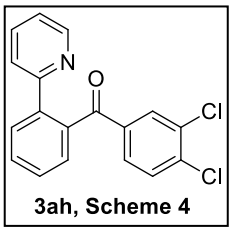
3ag, Scheme 4



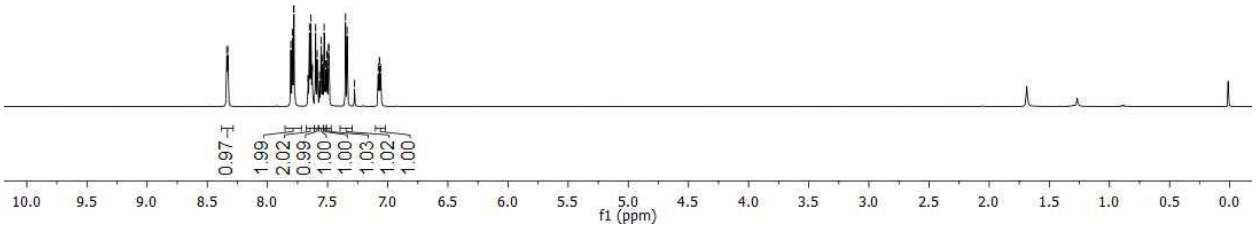


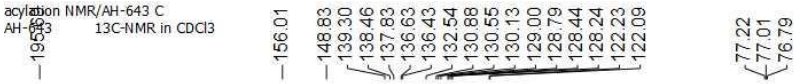
3ag, Scheme 4

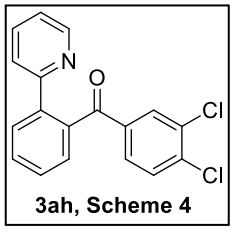




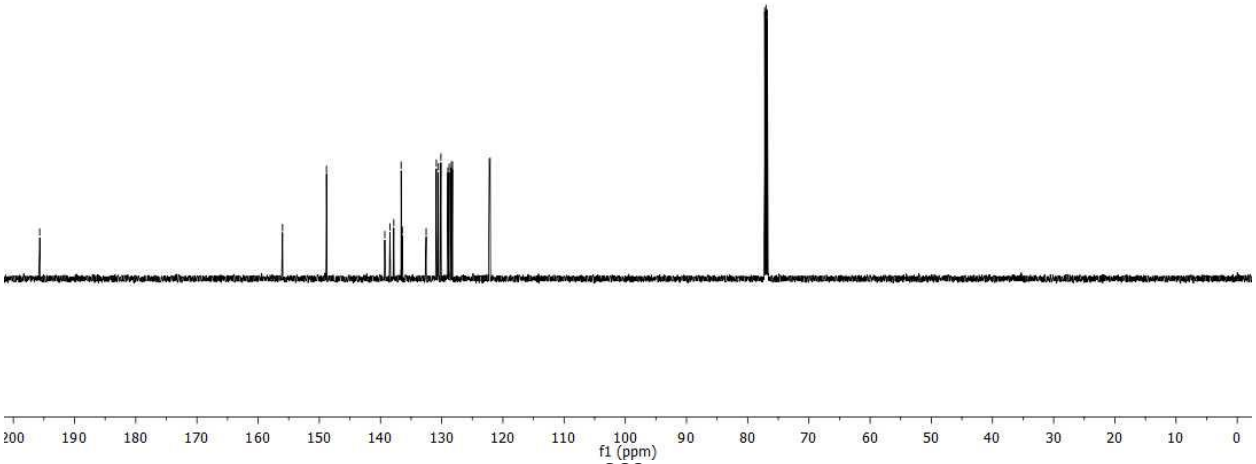
3ah, Scheme 4

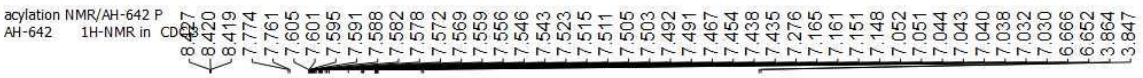


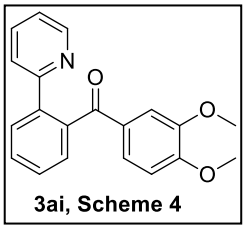




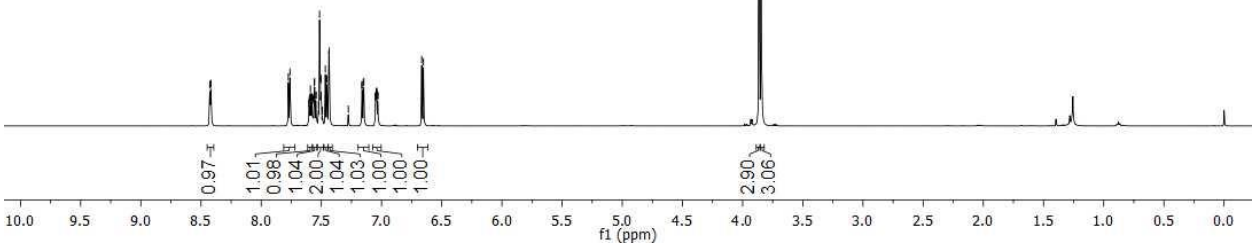
3ah, Scheme 4

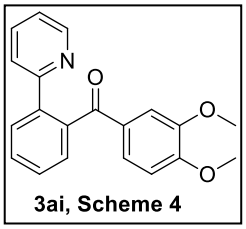




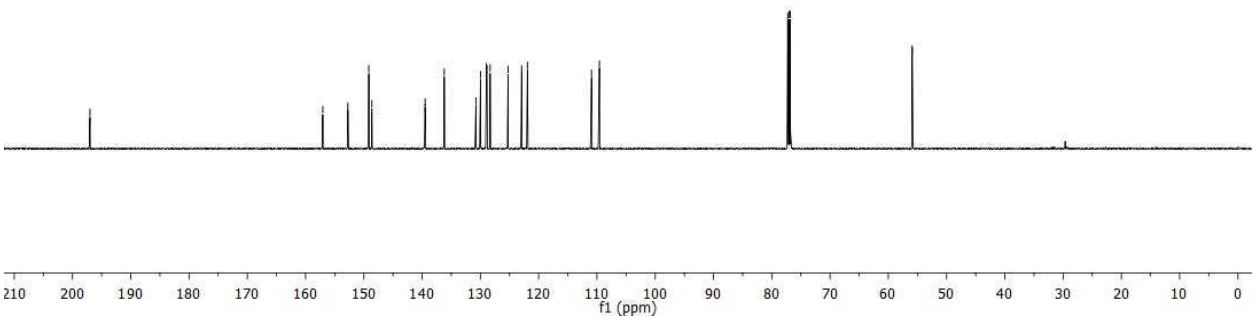


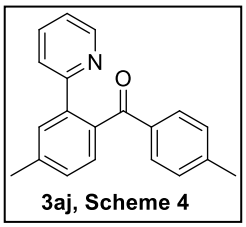
3ai, Scheme 4



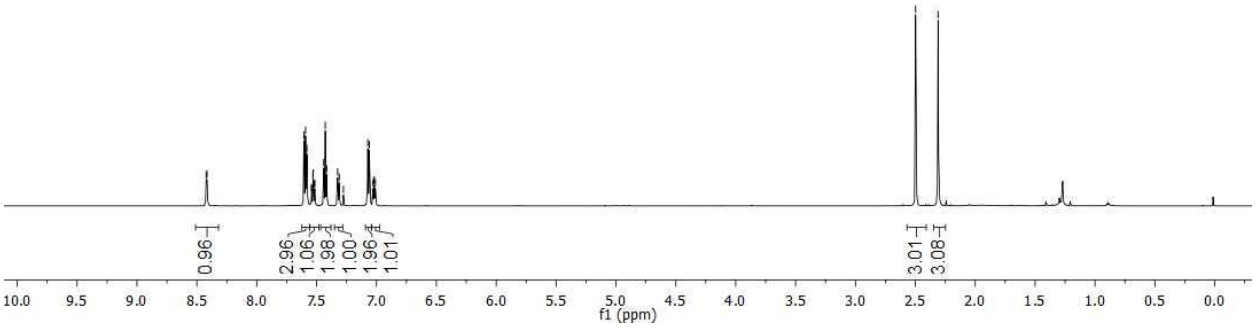


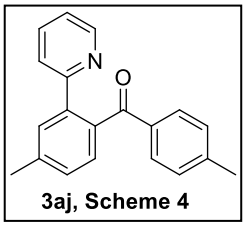
3ai, Scheme 4



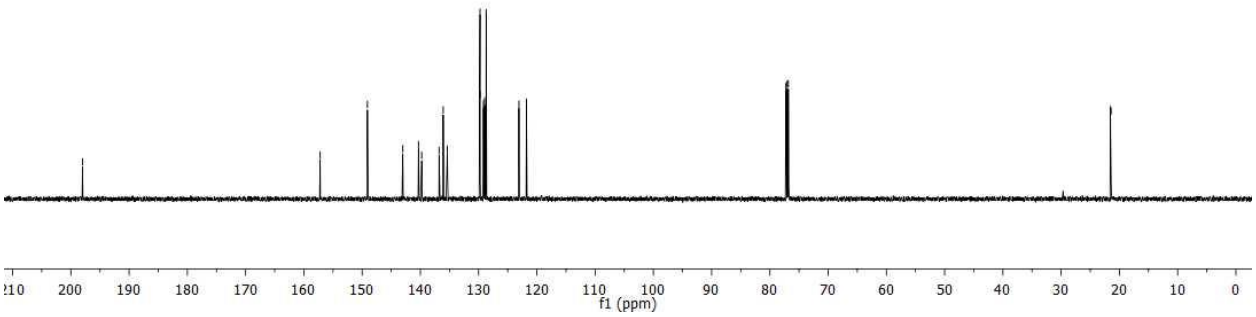


3aj, Scheme 4





3aj, Scheme 4




acylation NMR/AH-625 P
AH-625 1H-NMR in CDCl3
8.439
8.437
8.434
7.592
7.578
7.547
7.544
7.534
7.529
7.521
7.518
7.515
7.394
7.381
7.276
7.263
7.258
7.070
7.057
7.046
7.044
7.042
7.040
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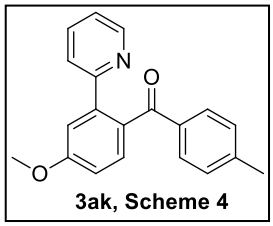
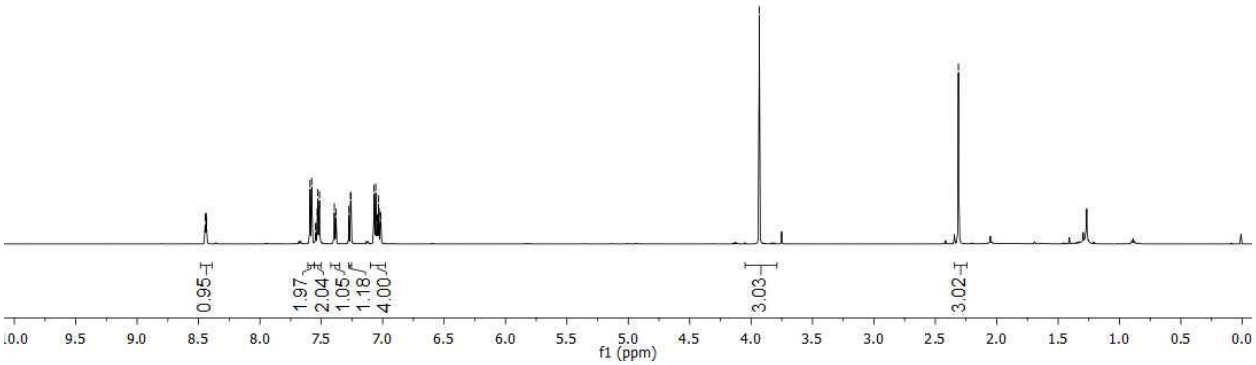
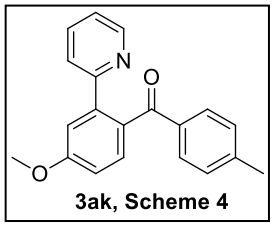
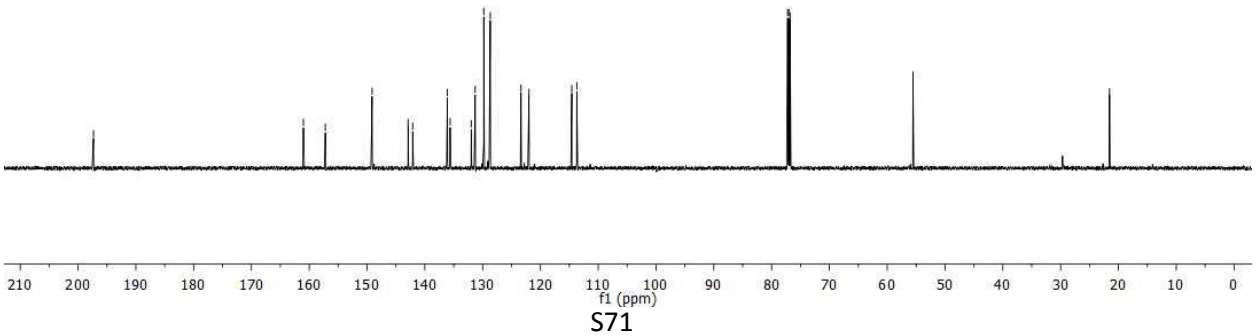
7.029
7.020
7.015
3.933
2.312

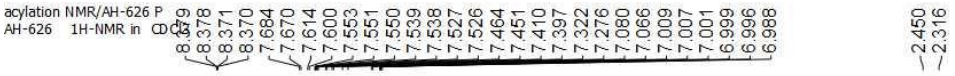

N
O
O
3ak, Scheme 4

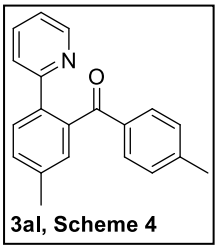

0.95
1.97
2.04
1.05
1.18
4.00
3.03
3.02
f1 (ppm)


acylation NMR/AH-625 C
AH-625 13C-NMR in CDCl3
161.01
157.22
149.13
142.90
142.11
136.10
135.62
131.97
131.29
129.80
128.67
123.36
121.98
114.57
113.71
77.24
77.02
76.81
55.54
21.53

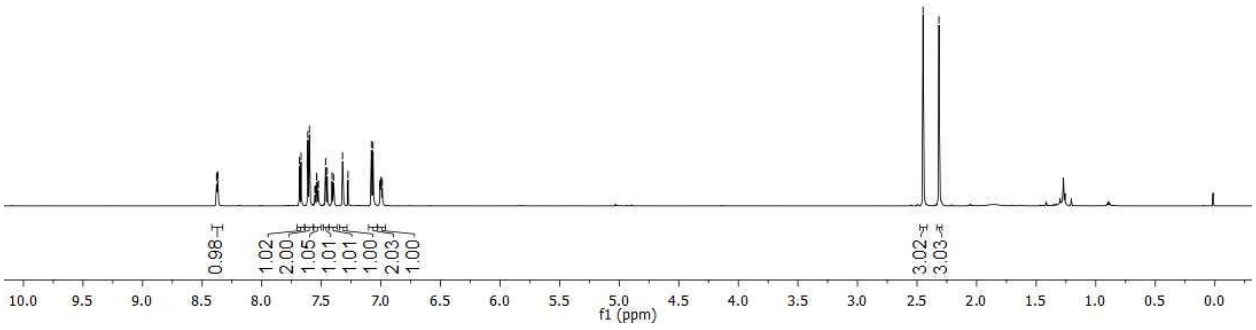

N
O
O
3ak, Scheme 4

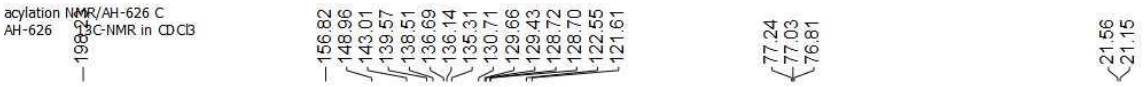

f1 (ppm)

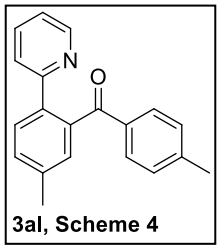




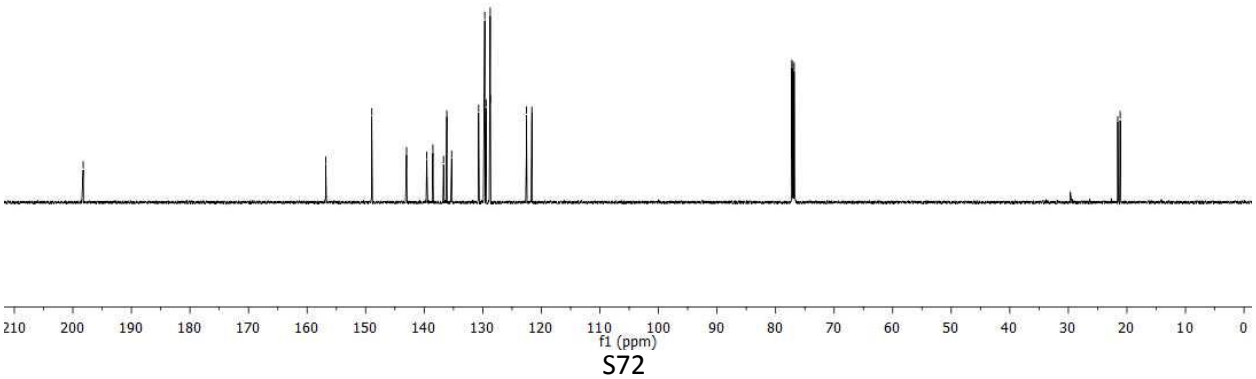
3al, Scheme 4

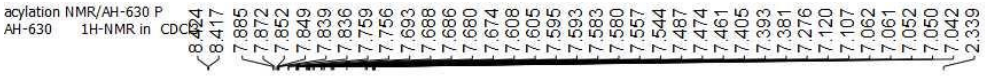


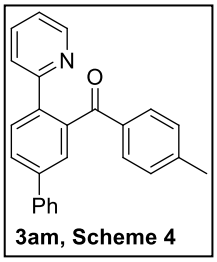




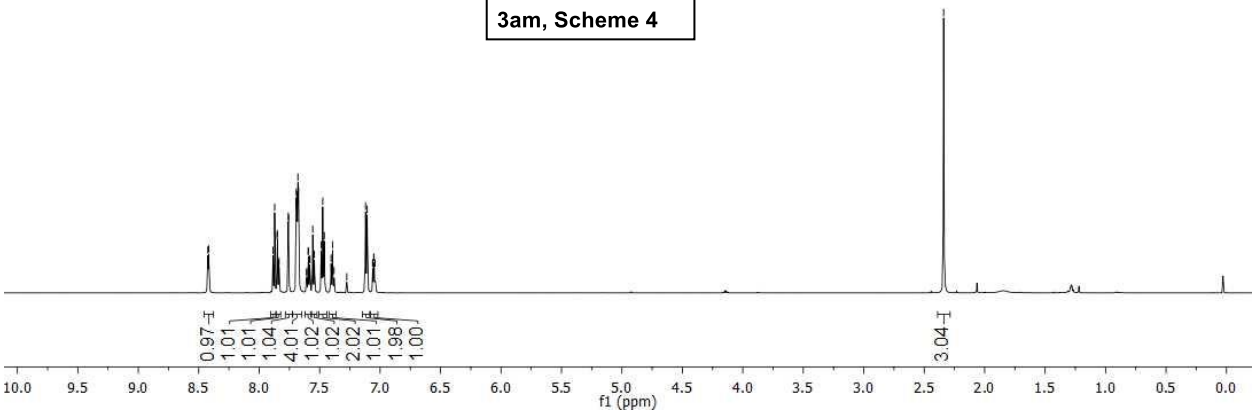
3al, Scheme 4

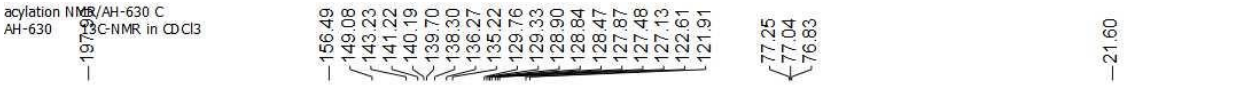


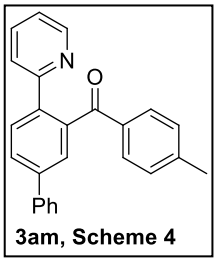




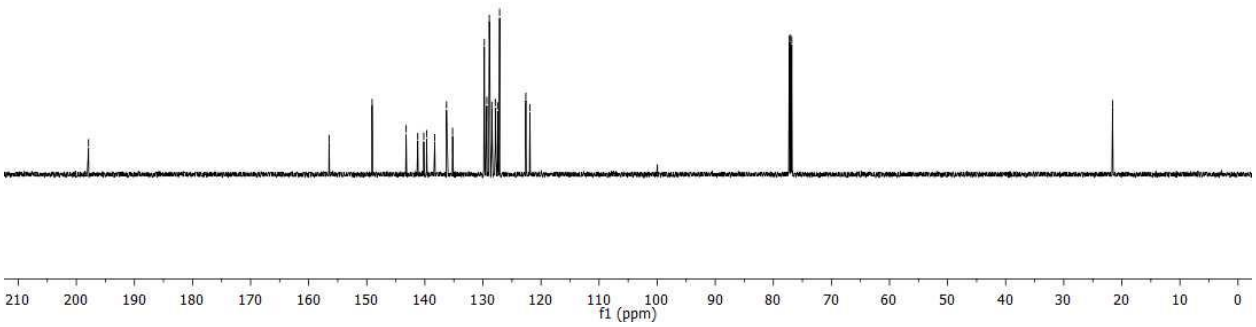
3am, Scheme 4

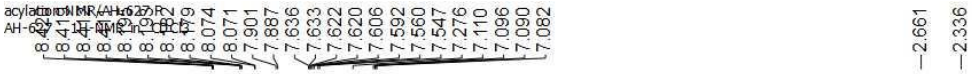


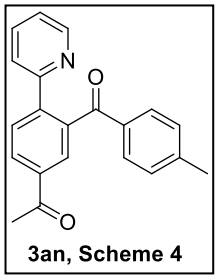




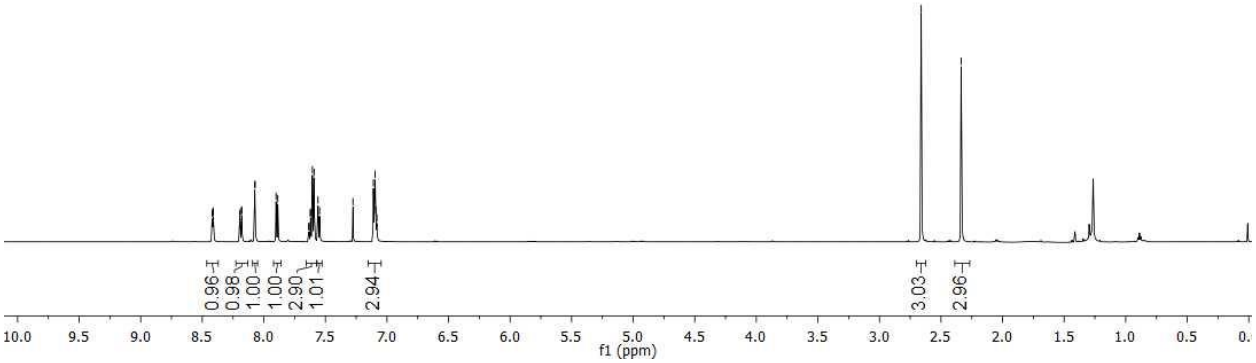
3am, Scheme 4

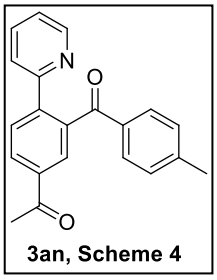




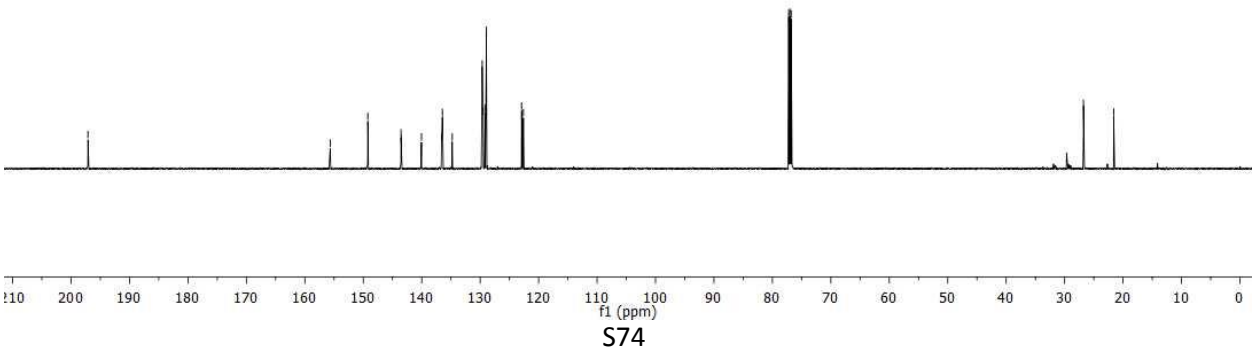


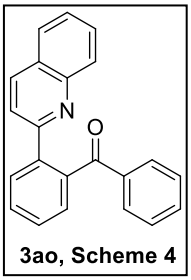
3an, Scheme 4



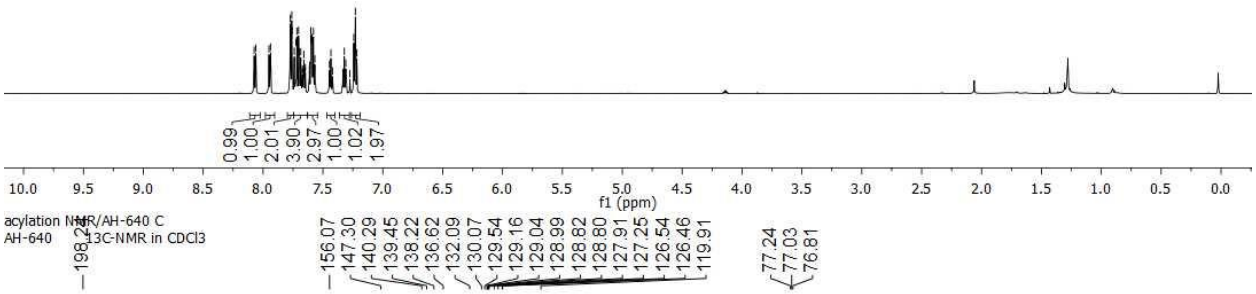


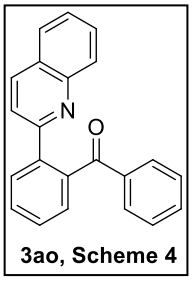
3an, Scheme 4



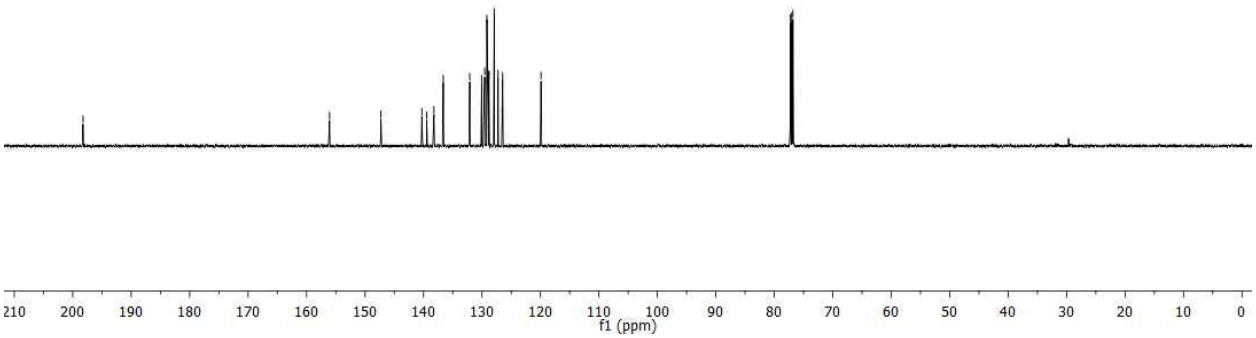


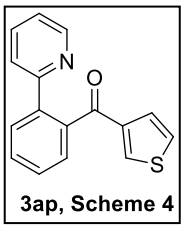
3ao, Scheme 4



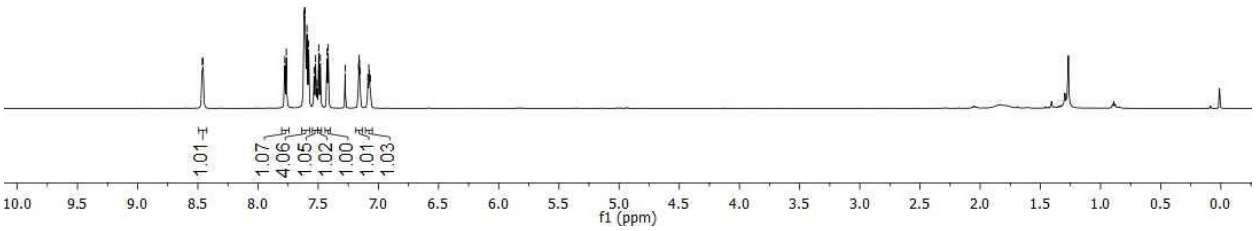


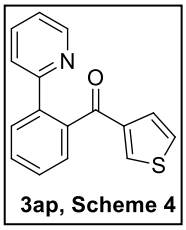
3ao, Scheme 4



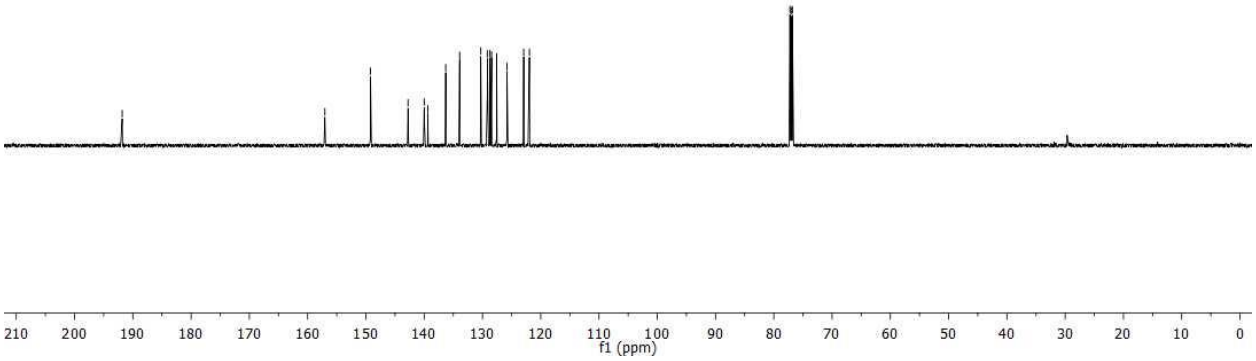


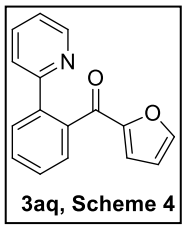
3ap, Scheme 4



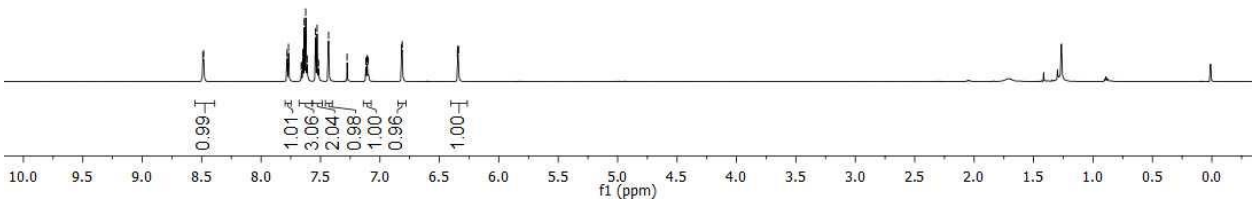


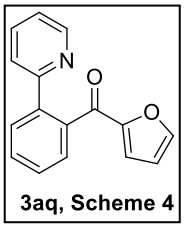
3ap, Scheme 4



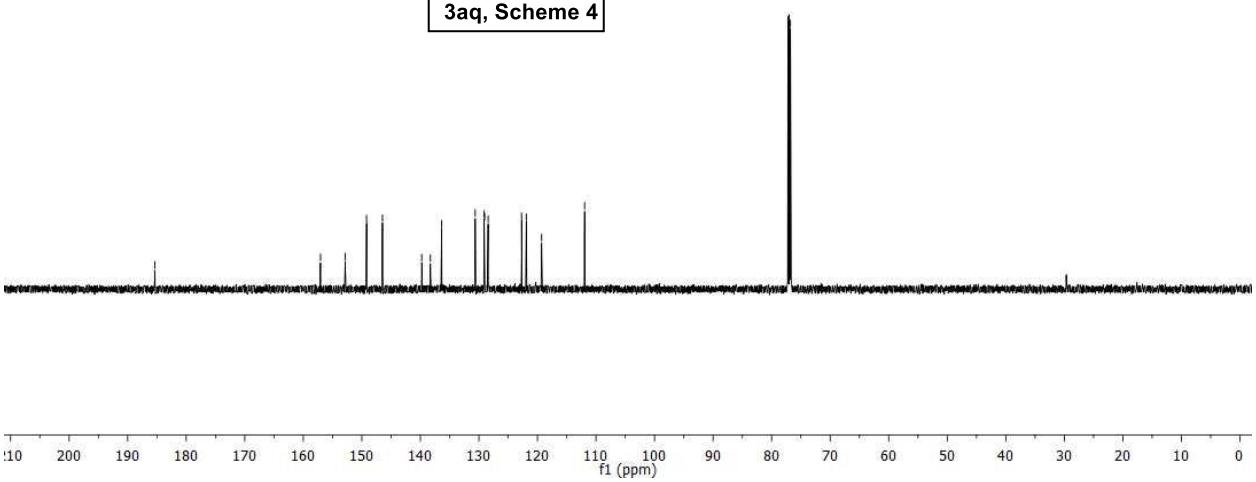


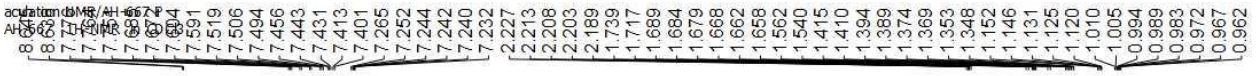
3aq, Scheme 4

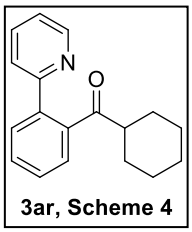




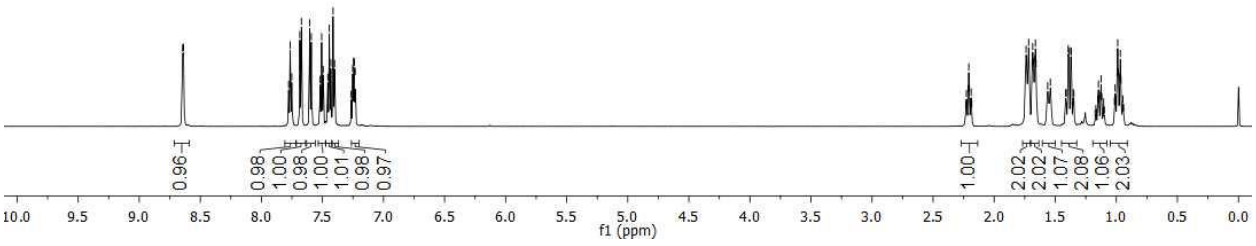
3aq, Scheme 4

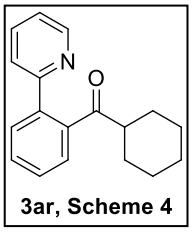




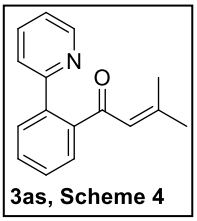


3ar, Scheme 4

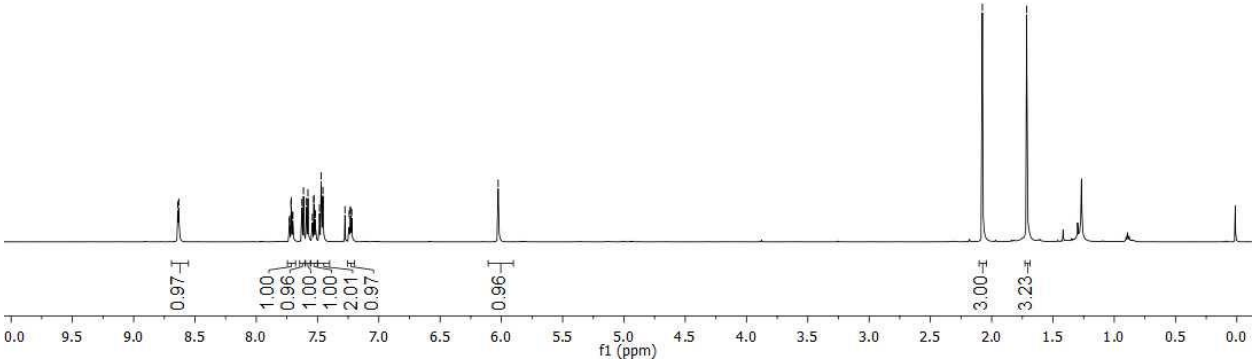


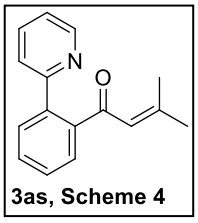


3ar, Scheme 4

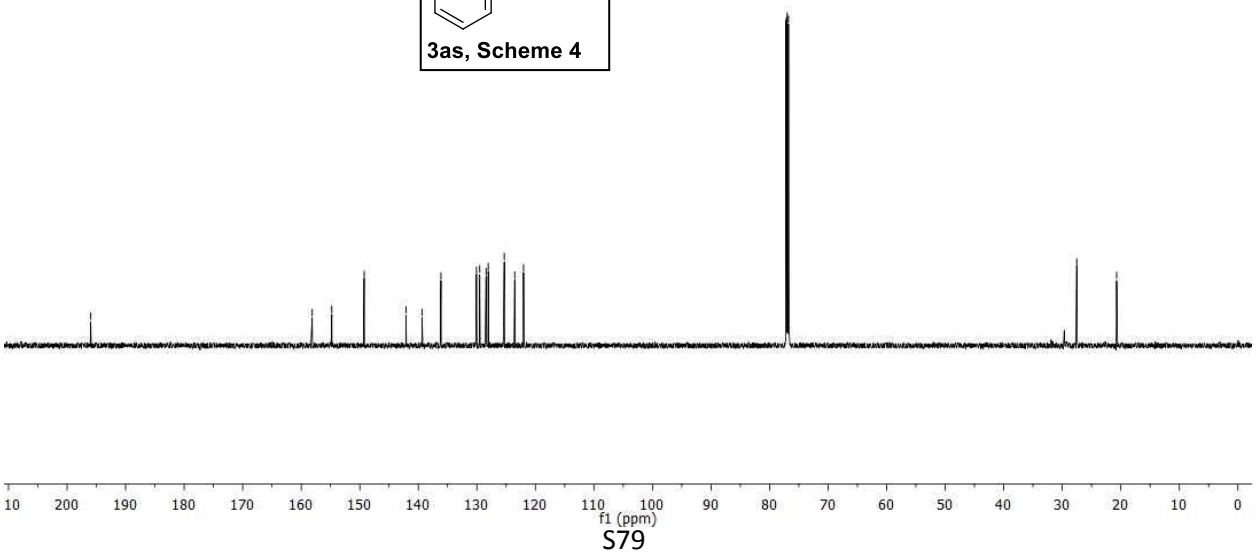


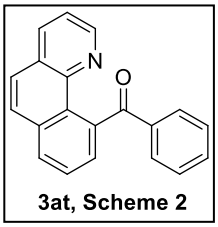
3as, Scheme 4



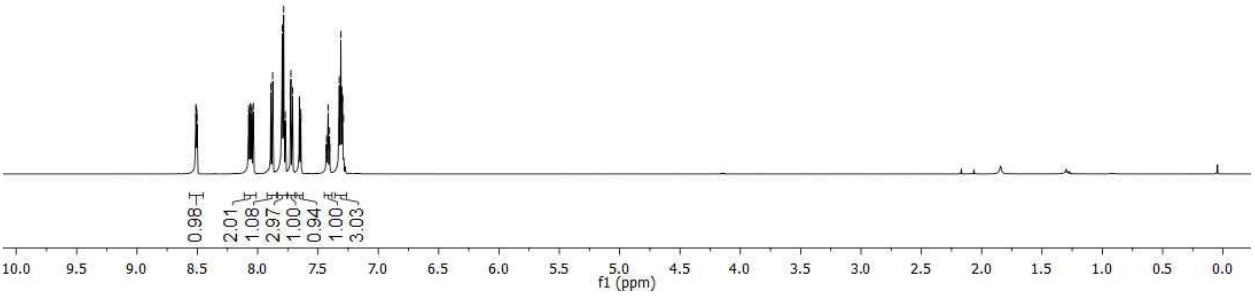


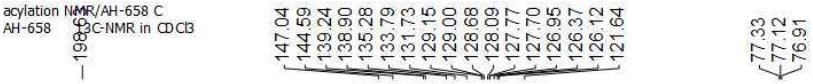
3as, Scheme 4

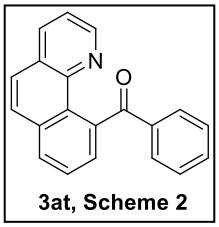




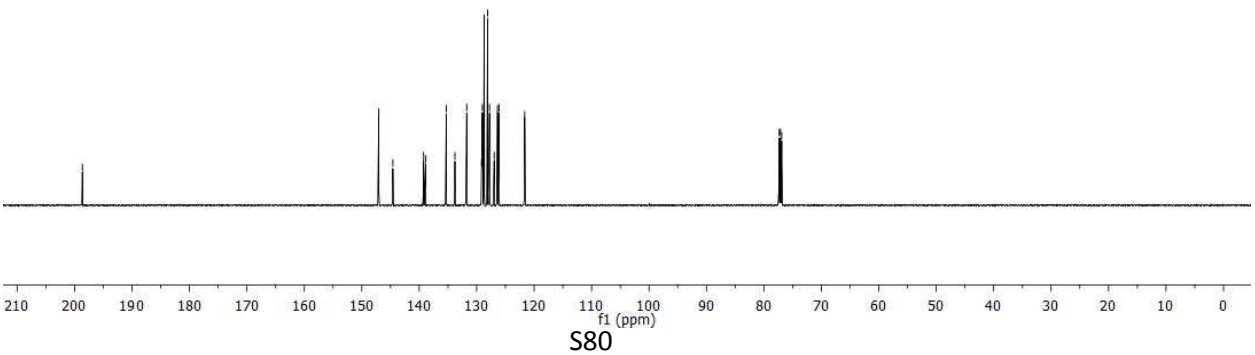
3at, Scheme 2

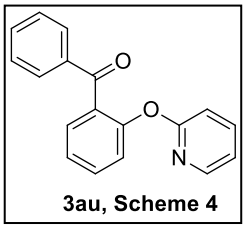




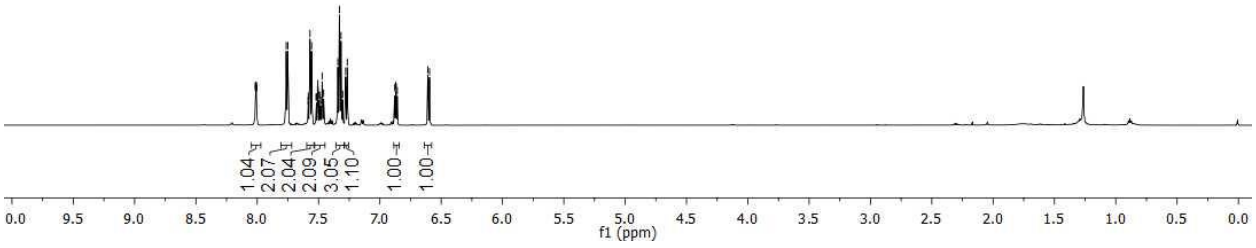


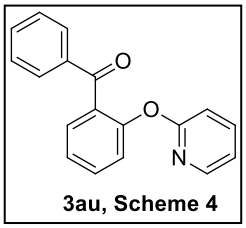
3at, Scheme 2



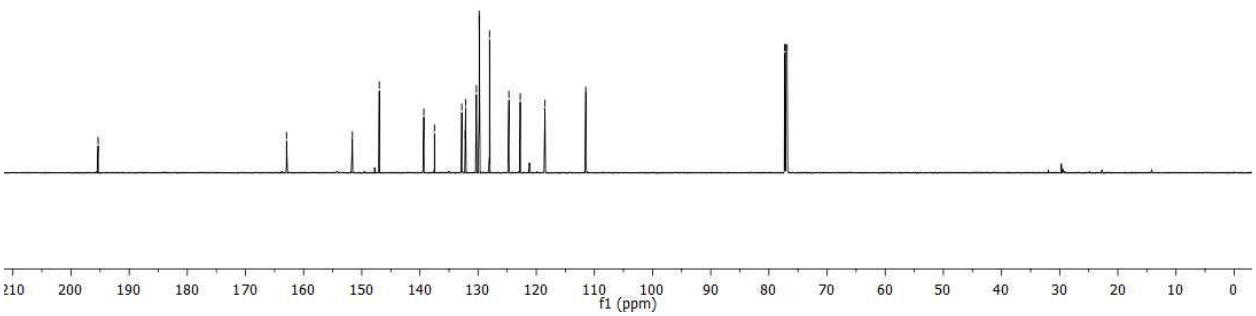


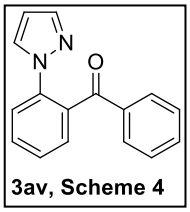
3au, Scheme 4

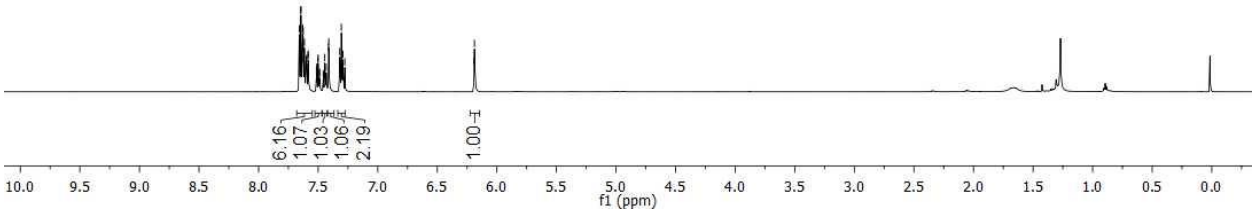




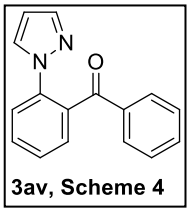
3au, Scheme 4

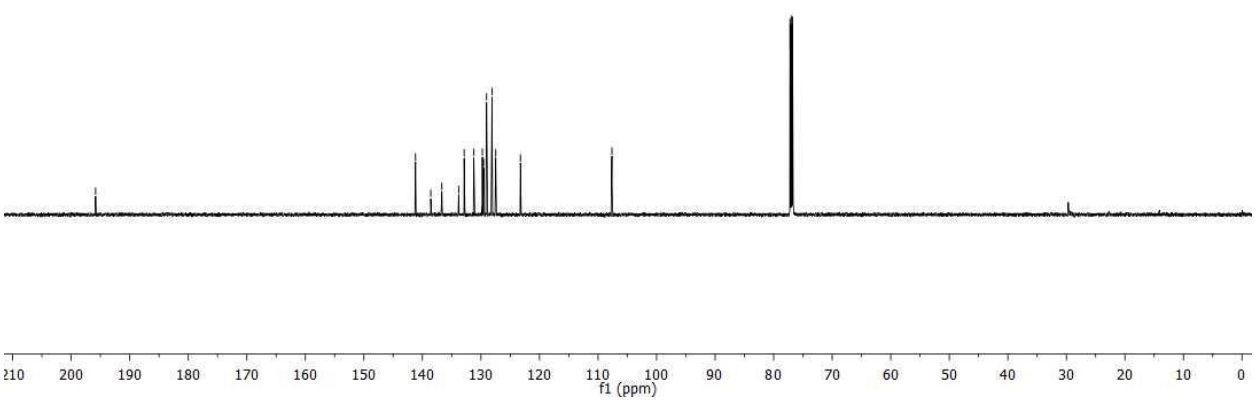



3av, Scheme 4

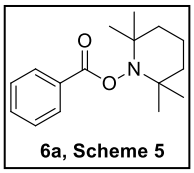

6.16
1.07
1.03
1.06
2.19
1.00
f1 (ppm)


acylation NMR/AH-656 C
AH-656 13C-NMR in CDCl3
195.58
141.18
138.58
136.69
133.80
132.87
131.22
129.76
129.55
129.04
128.11
127.48
123.25
107.63
77.21
76.99
76.78


3av, Scheme 4

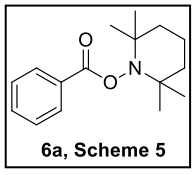

f1 (ppm)

RJ-0517/3
AH-723 1H-NMR in CDCl3



6a, Scheme 5

RJ-0517/4
AH-723 13C-NMR in CDCl3



6a, Scheme 5