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Supporting information

Solvent-free aminocarbonylation of iodobenzene in the presence of SILP-palladium catalysts

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I. Preparation of catalysts

Preparation of silica support with grafted imidazolium ions (SILP-2)[19]

A mixture of 15.1 mmol (1.24 g, 1.2 ml) 1-methylimidazole and 15.0 mmol (3,6 ml) (3-chloropropyl)-triethoxysilane was reacted in dry toluene under argon at room temperature for 24 h. Toluene was removed in vacuo , the residue was washed with diethyl ether, dried in vacuo to yield 4.59 g 1-methyl-3-[(triethoxysilyl)propyl] imidazolium chloride (yield:95 %).
A mixture of 10 mmol (3.23 g) of 1-methyl-3-[(triethoxysilyl)propyl] imidazolium chloride and 10 mmol (1.05 g) ammonium-tetrafluoroborate and 50 ml acetonitrile was stirred at room temperature for 5 days. The precipitated solid was removed by filtration through alumina and the solvent was evaporated. The residue was dissolved in dichloromethane (50 ml) and the solution was filtered through activated charcoal and alumina. After removal of the solvent in vacuo, 1-methyl-3-[(triethoxysilyl)propyl] imidazolium tetrafluoroborate was obtained in 64% yield. (Elemental analysis: Calc. for $C_{13}H_{27}N_2O_3SiBF_4$: C, 41.72; H, 7.27; N, 7.49; Found: C, 41.95; H, 7.35; N, 7.63.)
4.8 mmol (1.8 g) of 1-methyl-3-[(triethoxysilyl)propyl] imidazolium tetrafluoroborate was dissolved in 50 ml chloroform and 3.0 g silica (pre-treated by heating for 5 h at 250 °C) was added. The mixture was refluxed for 24 h. Then the SILP material was filtered and washed with pentane (50 ml), acetonitrile (100 ml) and diethylether (100 ml) and was dried in vacuo to produce the **SILP-2** phase. The amount of ionic liquid supported on silica was determined by measuring the weight increase after heating the material to constant weight at 150 ^{o}C in vacuo (180 mg 1-methyl-3-[(triethoxysilyl)propyl] imidazolium tetrafluoroborate on 1 g silica).

Preparation of silica supported palladium catalysts

Preparation of CAT-1. 0.02 mmol (20.7 mg) $Pd_2(dba)_3.CHCl_3$ was dissolved in a mixture of 2 ml acetonitrile and 2 ml THF. The mixture was stirred for 15 min at room temperature. Then 550 mg silica (Kieselgel 60 (0.040-0.063 mm), Merck, pre-treated by heating for 5 h at 250 ^{o}C) was added and the resulting mixture was stirred for 24 h. The solvents were removed in vacuo and the catalyst was dried at 35 ^{o}C in vacuo for 3 h and was stored under argon until use. Palladium content of the catalyst: 0.55% (determined by ICP).
Preparation of CAT-2. 0.04 mmol (8.7 mg) $Pd(OAc)_2$ and 0.12 mmol (36.8 mg) 4-diphenylphosphino-benzoic acid (DPPBA) were dissolved in a mixture of 2 ml acetonitrile and 2 ml THF. The mixture was stirred for 15 min at room temperature. Then 550 mg silica (Kieselgel 60 (0.040-0.063 mm), Merck, pre-treated by heating for 5 h at 250 ^{o}C) was added and the resulting mixture was stirred for 24 h. The solvents were removed in vacuo and the catalyst was dried at 35 ^{o}C in vacuo for 3 h and was stored under argon until use. Palladium content of the catalyst: 0.64% (determined by ICP).
Preparation of CAT-3. 200 mg [BMIM][BF_4] and 0.02 mmol (20.7 mg) $Pd_2(dba)_3.CHCl_3$ were dissolved in a mixture of 2 ml acetonitrile and 2 ml THF. The mixture was stirred for 15 min at room temperature. Then 550 mg silica (Kieselgel 60 (0.040-0.063 mm), Merck, pre-treated by heating for 5 h at 250 ^{o}C) was added under stirring and the resulting mixture was stirred for 24 h. The solvents were removed in vacuo and the catalyst was dried at 35 ^{o}C in vacuo for 3 h and was stored under argon until use. Palladium content of the catalyst: 0.40% (determined by ICP).
Preparation of CAT-4. 200 mg [BMIM][BF_4], 0.04 mmol (8.7 mg) $Pd(OAc)_2$ and 0.12 mmol (36.8 mg) 4-diphenylphosphino-benzoic acid (DPPBA) were dissolved in a mixture of 2 ml acetonitrile and 2 ml THF. The mixture was stirred for 15 min at room temperature. Then 550 mg silica (Kieselgel 60 (0.040-0.063 mm), Merck, pre-treated by heating for 5 h at 250 ^{o}C) was added under stirring and the resulting mixture was stirred for 24 h. The solvents were removed in vacuo and the catalyst was dried at 35 ^{o}C in vacuo for 3 h and was stored under argon until use. Palladium content of the catalyst: 0.51% (determined by ICP).
Preparation of CAT-5. A solution of 8.95 µmol (9.3 mg) $Pd_2(dba)_3.CHCl_3$ in 2 ml acetonitrile and 2 ml THF was stirred for 15 min. Then 500 mg of **SILP-2** was added and the resulting mixture was stirred for 24 h at room temperature. After evaporation of the solvents, the catalyst was dried at 35 ^{o}C in vacuo for 3 h. Palladium content of the catalyst: 0.29% (determined by ICP).
Preparation of CAT-6. 17.9 µmol (4.0 mg) $Pd(OAc)_2$ and 53.7 µmol (16.4 mg) 4-diphenylphosphino-benzoic acid (DPPBA) were dissolved in a mixture of 2 ml acetonitrile and 2 ml THF. The mixture was stirred for 15 min at room temperature. Then 500 mg of **SILP-2** was added and the resulting mixture was stirred for 24 h at room temperature. After evaporation of the solvents, the catalyst was dried at 35 ^{o}C in vacuo for 3 h. Palladium content of the catalyst: 0.58% (determined by ICP).

II. Charaterisation of catalysts

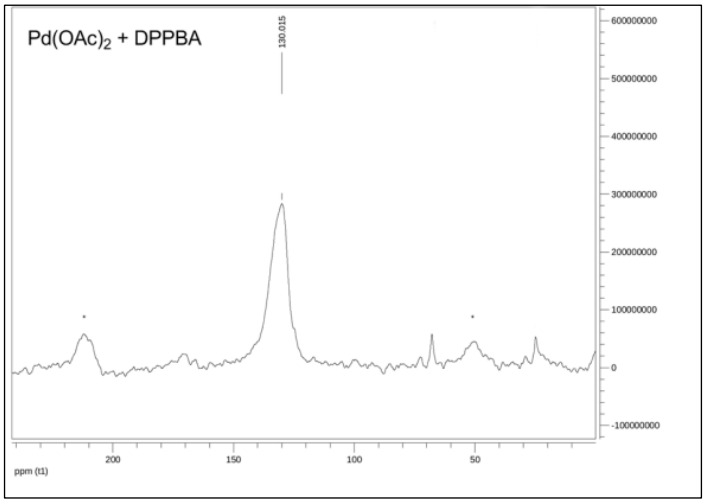


Figure S1 ^{13}C CP MAS NMR spectrum of **CAT-2** (* indicates rotational sidebands)

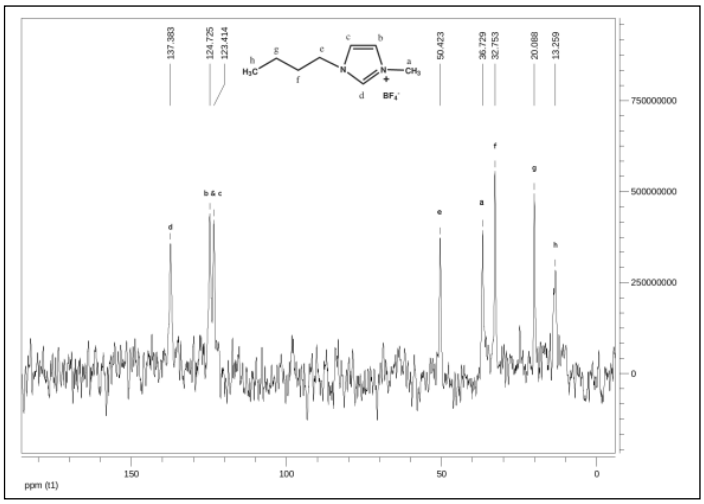


Figure S2 HPDEC ^{13}C NMR spectrum of **CAT-3**

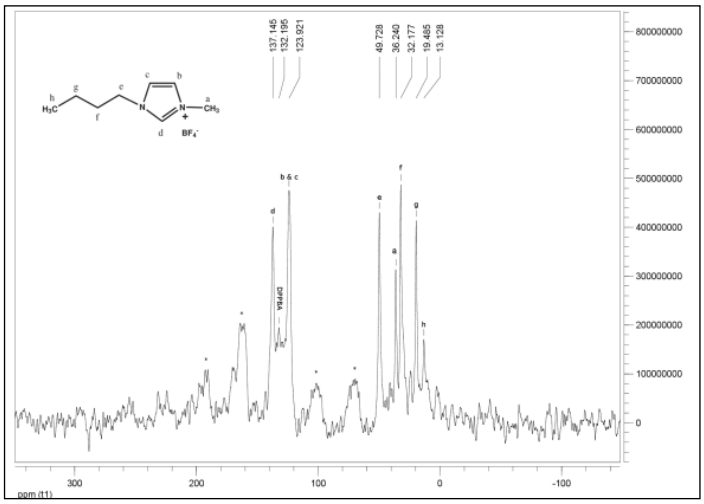


Figure S3 ^{13}C CP MAS NMR spectrum of **CAT-4** (* indicates rotational sidebands)

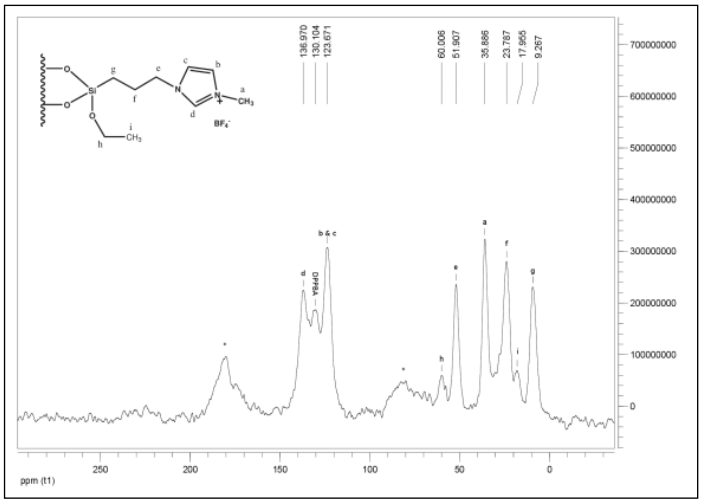


Figure S4 ^{13}C CP MAS NMR spectrum of **CAT-6** (* indicates rotational sidebands)

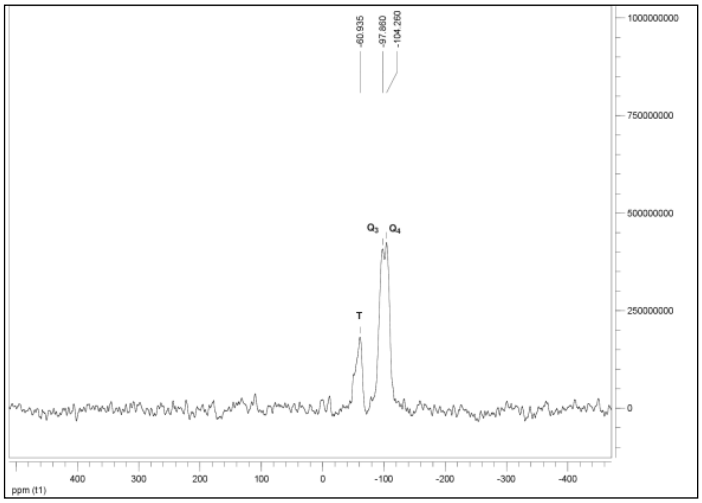


Figure S5 ^{29}Si CP MAS NMR spectrum of **CAT-6** (T: $Si(OSi)_3R$, $Si(OSi)_2ROH$, $Si(OSi)R(OH)_2$, Q_3: $Si(OSi)_3OH$, and Q_4: $Si(OSi)_4$)

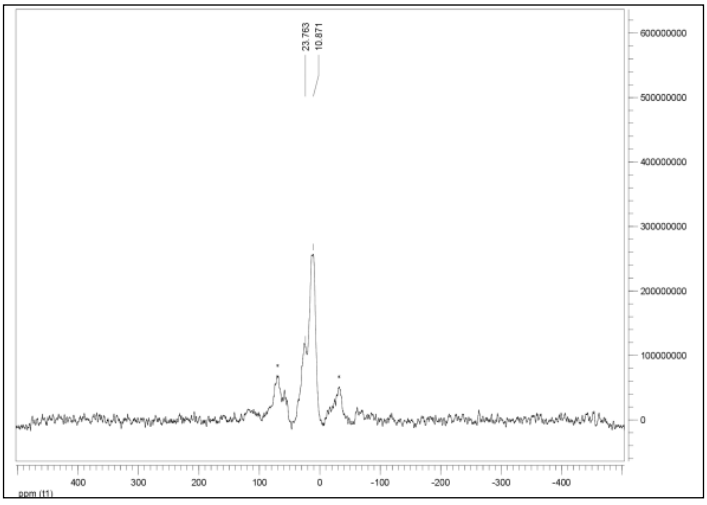


Figure S6 ^{31}P CP MAS NMR spectrum of **CAT-6** (* indicates rotational sidebands)

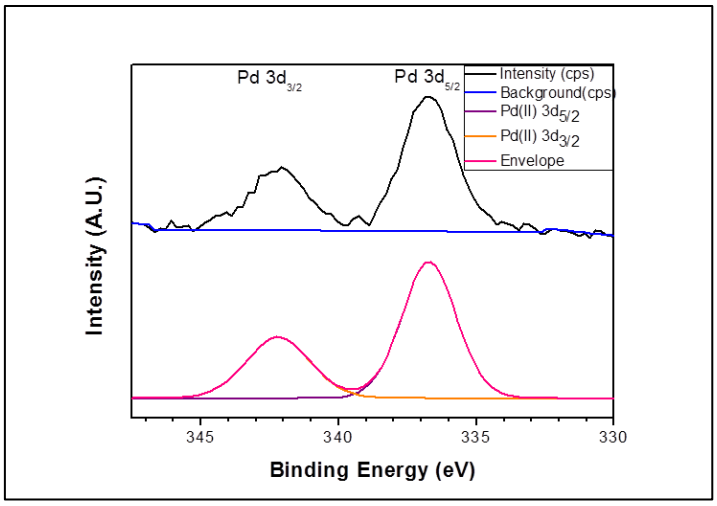


Figure S7 Pd 3d XPS spectrum of **CAT-6**

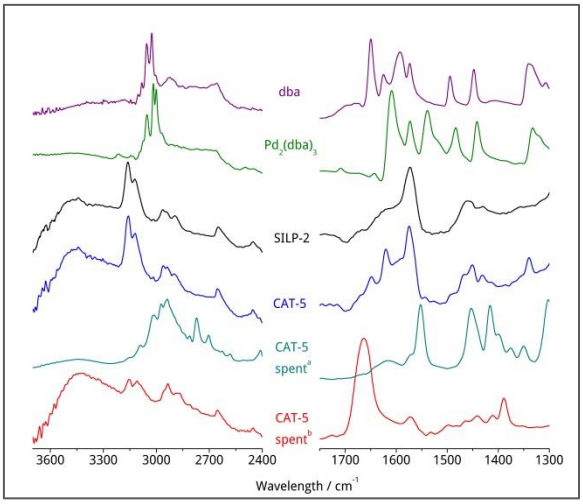


Figure S8 FT-IR spectra of ligand, Pd precursor, supported ionic liquid phase, fresh and spent **CAT-5** catalyst ([a]: washed with toluene, [b]: washed with DMF)

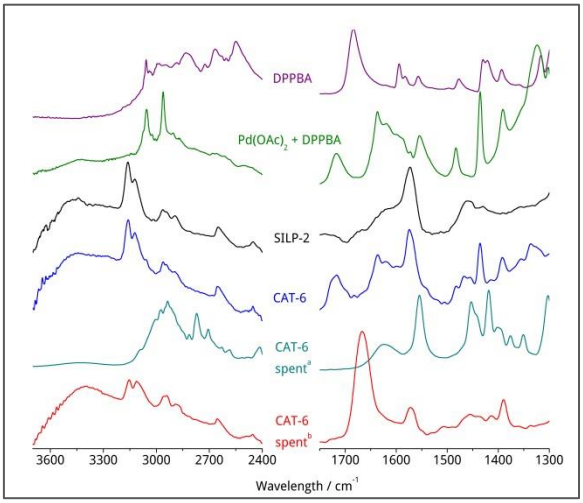


Figure S9 FT-IR spectra of ligand, Pd precursor, supported ionic liquid phase, fresh and spent **CAT-6** catalyst ([a]: washed with toluene, [b]: washed with DMF)

III. General procedure for aminocarbonylation reactions

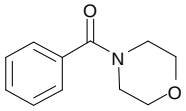
Catalytic reactions at atmospheric pressure. In a typical experiment a solution containing the palladium catalyst (with 3.6 µmol Pd-content) was placed in a Schlenk-tube. Under argon, 0.4 mmol (45 µl) iodobenzene (**1a**), 1.0 mmol (88 µl) morpholine (**2a**) and 0.5 mmol (70 µl) triethylamine were added and the atmosphere was changed to carbon monoxide. The reaction was conducted for 3 hours at 100^{o}C. After cooling to room temperature, the products were extracted with 2x1 ml toluene. The reaction mixture was analysed by GC and GC-MS.

Catalytic reactions at elevated pressure. In a typical experiment the catalyst (containing 3.6 µmol Pd) was placed in a stainless steel autoclave. The aryl iodide (0.4 mmol), the amine (0.5 mmol) and 0.5 mmol (70 µl) triethylamine were transferred into it under an inert atmosphere. It was charged with carbon monoxide (5-30 bar) and heated with stirring in an oil bath at 100 or 120 ^{o}C for 3 or 8 h. After cooling to room temperature, the products were extracted with 2x1 ml toluene. The reaction mixture was analysed by GC and GC-MS and the catalyst was reused.

IV. Characterisation of products

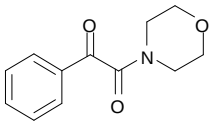
NMR spectra of isolated products correspond well to those reported previously. [6, 7, 17, 20, 22, 32] α-Ketoamides formed as minor components were characterised using GC-MS spectra.

Morpholino(phenyl)methanone (3a)[7]



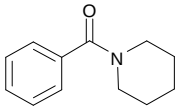
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.41-7.37 (m, 5H); 3.86-3.54 (m, 6H); 3.54-3.36 (m, 2H). MS(m/z/rel.int.): 191(M^+)/11; 190/34; 176/9; 160/6; 105/100; 86/12; 77/68; 51/24

1-Morpholino-2-phenylethane-1,2-dione (4a)[6]



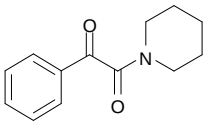
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.96 (dd, J=8.2 Hz, J=1.1 Hz, 2H); 7.66 (tt, J=7.4 Hz, J=1.1 Hz, 1H); 7.54-7.51 (m, 2H); 3.82-3.78 (m, 4H); 3.67-3.65 (m, 2H); 3.40-3.38 (m, 2H). MS(m/z/rel.int.): 219 (M^+)/6; 114/11; 105/100; 86/4; 77/54; 70/26; 51/22.

Phenyl(piperidin-1-yl)methanone (Table 6, entry 1).[7]



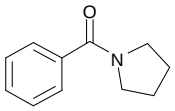
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.37-7.36 (m, 5H); 3.78-3.59 (m, 2H); 3.42-3.22 (m, 2H); 1.69-1.63 (m, 4H); 1.56-1.44 (m, 2H). MS(m/z/rel.int.): 189 (M^+)/36; 188/100; 106/10; 105/98; 84/9; 77/56; 51/12.

1-Phenyl-2-(piperidin-1-yl)ethane-1,2-dione.[6]



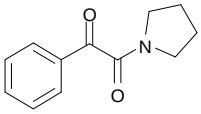
MS(m/z/rel.int.): 217(M^+)/5; 112/100; 105/54; 84/10; 77/33; 69/61; 51/11; 41/29.

Phenyl(pyrrolidin-1-yl)methanone (Table 6, entry 2).[7]



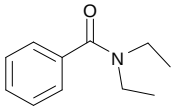
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.50-7.47 (m, 2H); 7.39-7.34 (m, 3H); 3.63 (t, J=6.8 Hz, 2H); 3.40 (t, J=6.8 Hz, 2H); 1.97-1.91 (m, 2H); 1.88-1.81 (m, 2H). MS(m/z/rel.int.): 175 (M^+)/44; 174/28; 146/28; 105/100; 77/57; 51/16.

1-Phenyl-2-(pyrrolidin-1-yl)ethane-1,2-dione.[20]



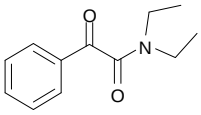
MS(m/z/rel.int.): 203(M^+)/3; 202/6; 105/71; 98/100; 77/52; 70/31; 55/56.

N,N-Diethyl-benzamide (Table 6, entry 3).[7]



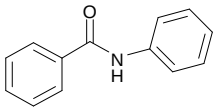
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.39-7.35 (m, 5H); 3.54 (brs, 2H); 3.25 (brs, 2H); 1.24 (brs, 3H); 1.10 (brs, 3H).
MS(m/z/rel.int.): 177(M^+)/13; 176/36; 105/100; 77/39; 51/11.

N,N-Diethyl-2-oxo-2-phenylacetamide.[6]



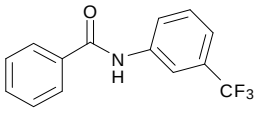
MS(m/z/rel.int.): 205(M^+)/5; 105/61; 100/100; 77/42; 72/74; 51/21.

N-Phenyl-benzamide (Table 6, entry 4).[7]



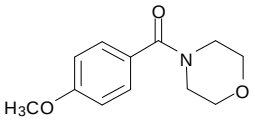
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.87-7.83 (m, 2H); 7.80 (brs, 1H); 7.66-7.61 (m, 2H); 7.56-7.52 (m, 1H); 7.50-7.45 (m, 2H); 7.38-7.33 (m, 2H); 7.16-7.12 (m, 1H). MS(m/z/rel.int.): 197(M^+)/42; 105/100; 77/52; 51/14.

N-(3-Trifluoromethyl-phenyl)benzamide (Table 6, entry 5).[22]



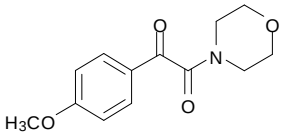
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.93 (brs, 1H); 7.89-7.85 (m, 4H); 7.59-7.55 (m, 1H); 7.52-7.46 (m, 3H); 7.41-7.38 (m, 1H).
MS(m/z/rel.int.): 265(M^+)/6; 246/8; 105/100; 77/47; 51/11.

Morpholino(4-methoxyphenyl)methanone (Table 6, entry 6).[17]



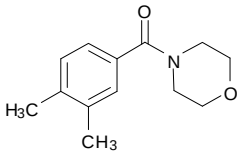
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.38 (d, J=8.9 Hz, 2H); 6.91 (d, J=8.9 Hz, 2H); 3.83 (s, 3H); 3.76-3.54 (m, 8H). MS(m/z/rel.int.): 221(M^+)/10; 220/16; 135/100; 107/8; 92/9; 77/15; 64/5; 56/3.

1-(4-Methoxyphenyl)-2-morpholinoethane-1,2-dione.[32]



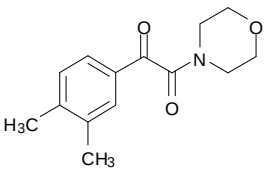
MS(m/z/rel.int.): 249(M^+)/3; 136/12; 135/100; 114/3; 107/11; 92/10; 77/14; 70/8; 64/5.

Morpholino(3,4-dimethylphenyl)methanone (Table 6, entry 7).[17]



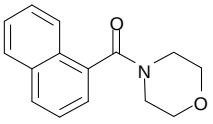
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.19 (brs, 1H); 7.16-7.10 (m, 2H); 3.88-3.38 (m, 8H); 2.28 (s, 6H). MS(m/z/rel.int.): 219(M^+)/11; 218/17; 133/100; 105/20; 79/11; 77/12.

1-(3,4-Dimethylphenyl)-2-morpholinoethane-1,2-dione.[20]



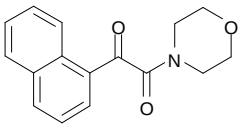
MS(m/z/rel.int.): 247(M^+)/3; 133/100; 105/24; 79/9; 77/9; 70/9.

Morpholino(naphth-1-yl)methanone (Table 6, entry 8).



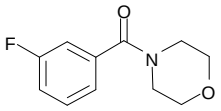
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.91-7.83 (m, 3H); 7.60-7.48 (m, 3H); 7.45-7.42 (m, 1H); 4.05-4.00 (m, 1H); 3.92-3.83 (m, 3H); 3.56-3.48 (m, 2H); 3.26-3.17 (m, 2H). ^{13}C NMR (100.62 MHz, $CDCl_3$): 169.5; 133.7; 133.5; 129.6; 129.4; 128.5; 127.2; 126.5; 125.2; 124.6; 123.9; 67.1; 67.0; 47.6; 42.2. MS(m/z/rel.int.): 241(M^+)/38; 240/23; 156/23; 155/100; 127/81; 86/8; 77/8

1-(Napht-1-yl)-2-morpholinoethane-1,2-dione.



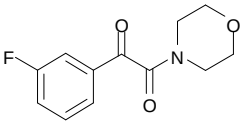
MS(m/z/rel.int.): 269(M^+)/10; 156/12; 155/100; 128/6; 127/53; 126/8; 77/5; 70/10; 42/5.

Morpholino(3-fluorophenyl)methanone (Table 6, entry 9).



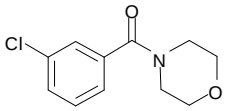
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.42-7.37 (m, 1H); 7.19-7.17 (m, 1H); 7.15-7.11 (m, 4H); 4.00-3.33 (m, 8H). ^{13}C NMR (100.62 MHz, $CDCl_3$): 168.9 (d, J=2.4 Hz); 162.6 (d, J=248.3 Hz); 137.4 (d, J=6.6 Hz); 130.4 (d, J=7.9 Hz); 122.7 (d, J=2.9 Hz); 116.9 (d, J=21.0 Hz); 114.4 (d, J=22.9 Hz); 66.8; 48.2; 42.7. MS(m/z/rel.int.): 209(M^+)/15; 208/28; 194/12; 178/5; 166/3; 123/100; 95/53; 86/24; 75/18; 56/41; 42/6.

1-(3-Fluorophenyl)-2-morpholinoethane-1,2-dione.[20]



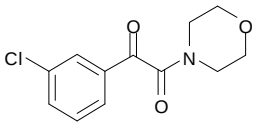
MS(m/z/rel.int.): 237(M^+)/9; 123/70; 114/100; 95/39; 86/12; 75/15; 70/77; 56/7; 45/6; 42/22.

Morpholino(3-chlorophenyl)methanone (Table 6, entry 10).



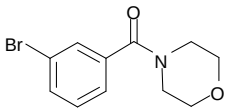
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.41-7.33 (m, 3H); 7.28-7.26 (m, 1H); 3.86-3.35 (m, 8H). MS(m/z/rel.int.): 227(M^+)/5; 226/13; 225(M^+)/16; 224/36; 212/4; 210/12; 141/32; 139/100; 113/16; 111/48; 86/36; 75/26; 56/50; 42/9.

1-(3-Chlorophenyl)-2-morpholinoethane-1,2-dione.[20]



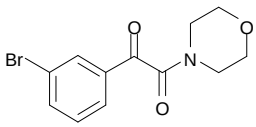
MS(m/z/rel.int.): 255(M^+)/3; 253(M^+)/9; 141/20; 139/61; 114/100; 111/26; 86/13; 75/16; 70/69; 42/20.

Morpholino(3-bromophenyl)methanone (Table 6, entry 11).



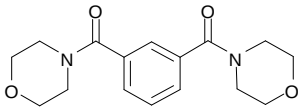
^{1}H NMR (400.13 MHz, $CDCl_3$): 7.55-8.52 (m, 2H); 7.32-7.22 (m, 2H); 3.89-3.30 (m, 8H). MS(m/z/rel.int.): 271(M^+)/26; 270/55; 269(M^+)/26; 268/55; 256/16; 254/16; 185/92; 183/92; 157/42; 55/42; 104/14; 86/74; 77/12; 76/63; 75/42; 74/11; 72/16; 56/100; 55/11; 50/30; 42/24.

1-(3-Bromophenyl)-2-morpholinoethane-1,2-dione.[20]



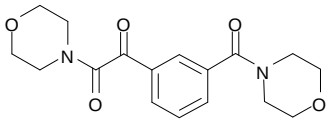
MS(m/z/rel.int.): 299(M^+)/5; 297(M^+)/5; 185/28; 183/28; 157/11; 155/11; 114/100; 86/14; 76/15; 75/12; 70/63; 42/20.

1,3-Phenylenebis(morpholinomethanone)



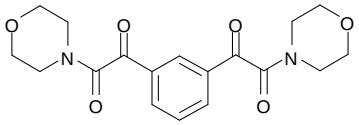
MS(m/z/rel.int.): 304(M^+)/26; 303/18; 219/16; 218/100; 189/11; 160/10; 133/33; 114/10; 105/12; 104/27; 86/86; 77/11; 76/37; 70/18; 56/42; 42/15.

1-(3-Morpholinocarbonyl)phenyl-2-morpholinoethane-1,2-dione



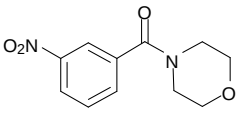
MS(m/z/rel.int.): 332(M^+)/22; 304/14; 218/100; 133/35; 114/86; 104/24; 86/12; 76/27; 70/67; 56/16; 42/25.

1,3-Phenylenebis(2-morpholinoethane-1,2-dione)



MS(m/z/rel.int.): 360(M^+)/14; 246/100; 133/12; 114/96; 104/17; 78/13; 76/20; 70/84; 45/11; 42/32.

Morpholino(3-nitrophenyl)methanone (Table 6, entry 12).



^{1}H NMR (400.13 MHz, $CDCl_3$): 8.30-8.24 (m, 2H); 7.76-7.71 (m, 1H); 7.64-7.160 (m, 4H); 3.91-3.31 (m, 8H). MS(m/z/rel.int.): 236(M^+)/19; 235/28; 221/26; 205/10; 151/7; 150/85; 134/8; 104/47; 92/7; 86/50; 77/9; 76/58; 56/100; 50/19; 42/20.

V. NMR spectra of isolated products

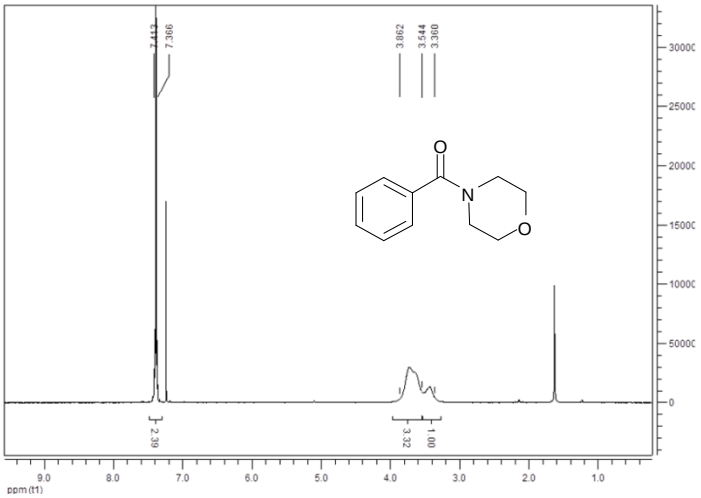


Figure S10 ^{1}H NMR spectrum of morpholino(phenyl)methanone (**3a**)

Figure S11 ^{1}H NMR spectrum of 1-morpholino-2-phenylethane-1,2-dione (**4a**)

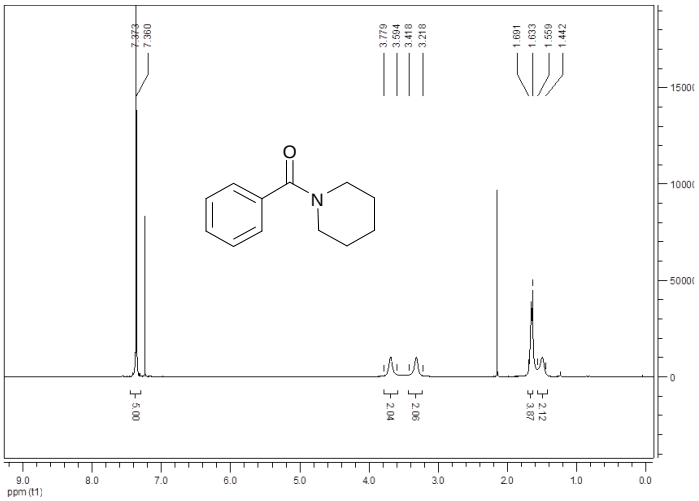


Figure S12 ^{1}H NMR spectrum of phenyl(piperidin-1-yl)methanone

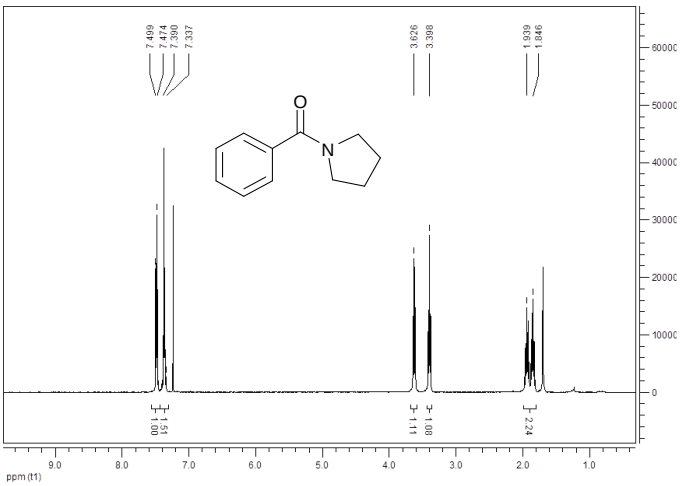


Figure S13 ^{1}H NMR spectrum of phenyl(pyrrolidin-1-yl)methanone

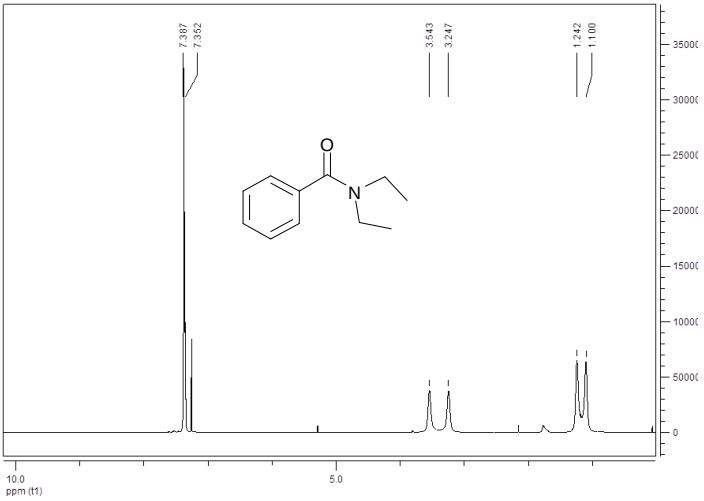


Figure S14 ^{1}H NMR spectrum of N,N-diethyl-benzamide

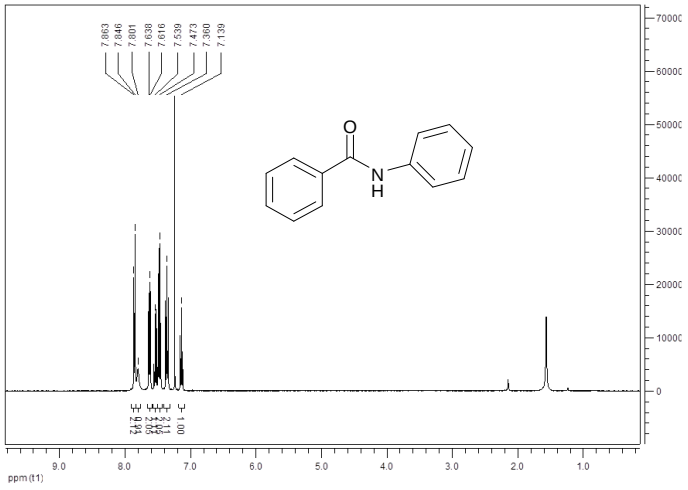


Figure S15 ^{1}H NMR spectrum of N-phenyl-benzamide

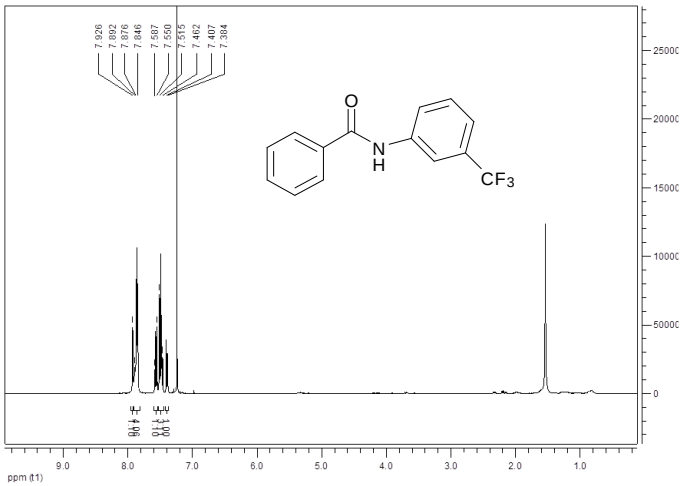


Figure S16 ^{1}H NMR spectrum of N-(3-trifluoromethyl-phenyl)benzamide

Figure S17 ^{1}H NMR spectrum of morpholino(4-methoxyphenyl)methanone

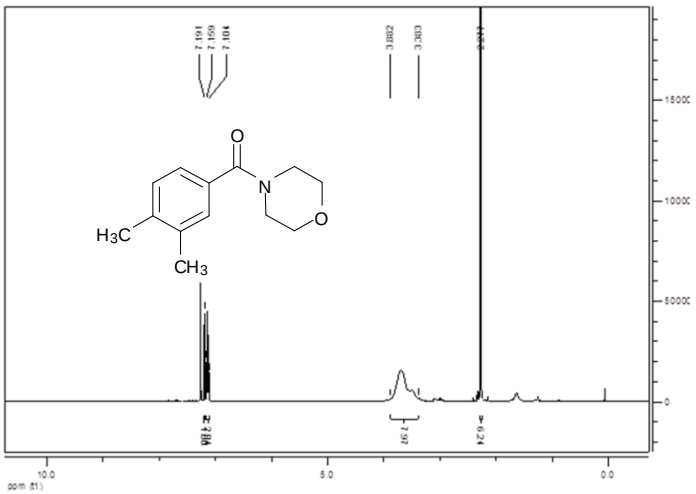


Figure S18 ^{1}H NMR spectrum of morpholino(3,4-dimethylphenyl)methanone

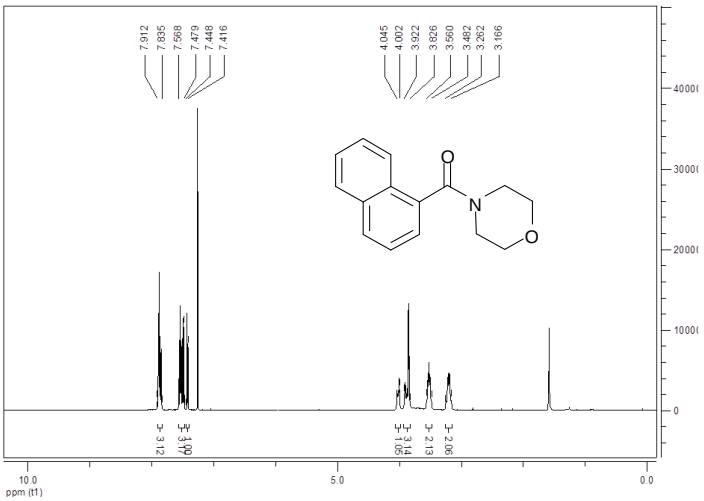


Figure S19 ^{1}H NMR spectrum of morpholino(naphth-1-yl)methanone

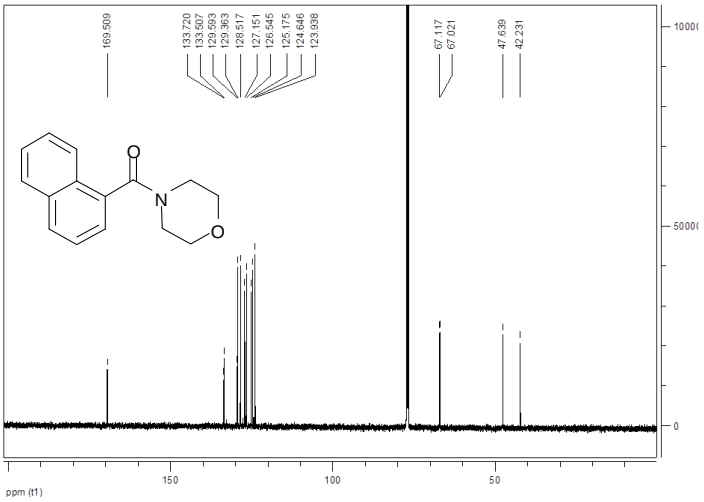


Figure S20 ^{13}C NMR spectrum of morpholino(naphth-1-yl)methanone

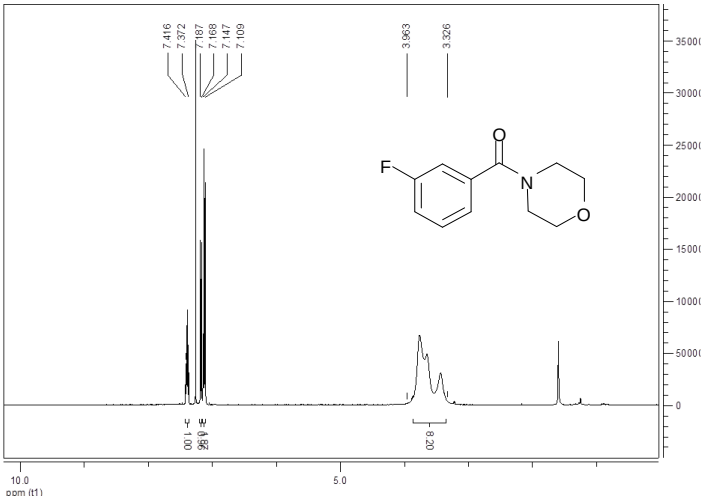


Figure S21 ^{1}H NMR spectrum of morpholino(3-fluorophenyl)methanone

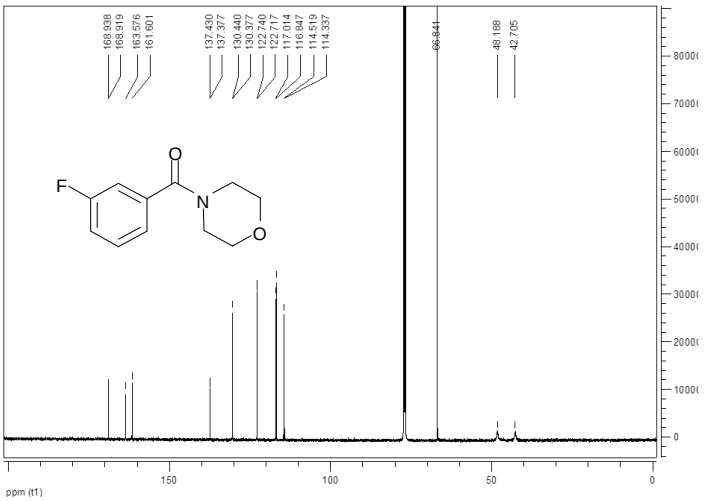


Figure S22 ^{13}C NMR spectrum of morpholino(3-fluorophenyl)methanone

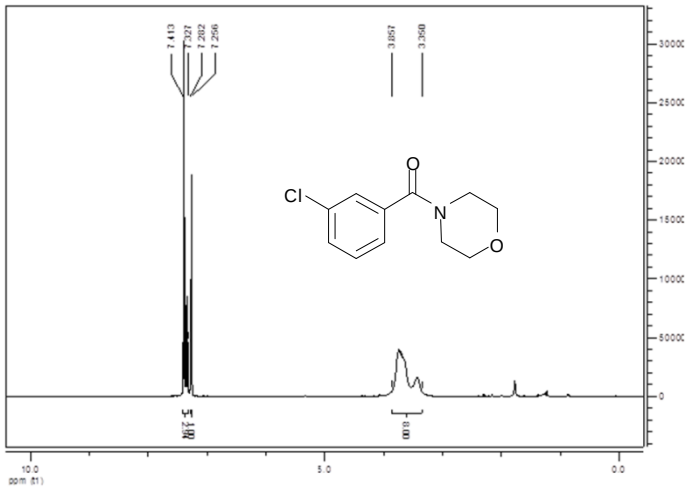


Figure S23 ^{1}H NMR spectrum of morpholino(3-chlorophenyl)methanone

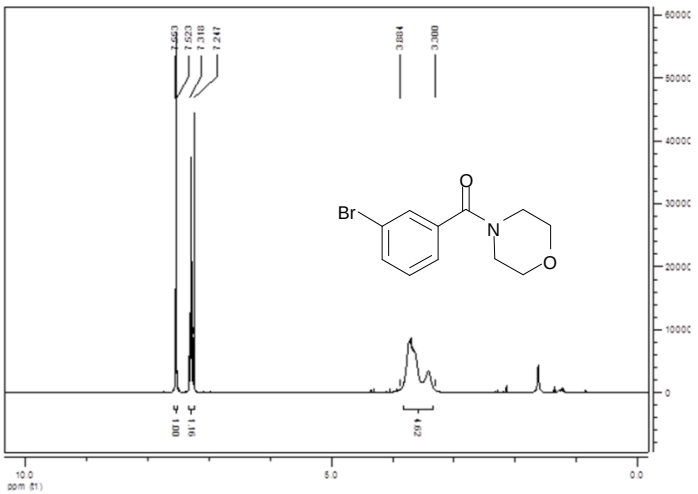


Figure S24 ^{1}H NMR spectrum of morpholino(3-bromophenyl)methanone

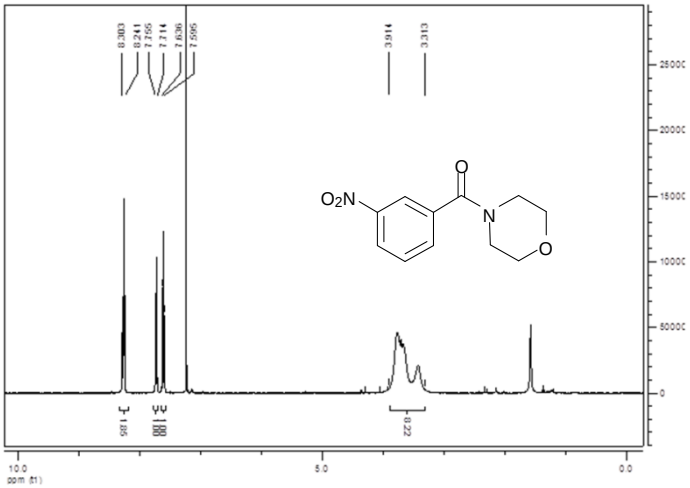


Figure S25 ^{1}H NMR spectrum of morpholino(3-nitrophenyl)methanone

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