Supporting Information

Mono- and double carbonylation of aryl iodides with amine nucleophiles in the presence of recyclable palladium catalysts immobilized on a supported dicationic ionic liquid phase

Máté Papp,[a] Péter Szabó,[b†] Dávid Srankó,[c] György Sáfrán,[d] László Kollár,[e] Rita Skoda-Földes [a]*

[a] University of Pannonia, Institute of Chemistry, Department of Organic Chemistry, H-8201 Veszprém, P.O.Box 158; Hungary

[b] University of Pannonia, Institute of Chemistry, Department of Analytical Chemistry, H-8201 Veszprém, P.O.Box 158; Hungary

[c] Hungarian Academy of Sciences, Centre for Energy Research, Department of Surface Chemistry and Catalysis, H-1525 Budapest 114., P.O. Box 49, Hungary

[d] Hungarian Academy of Sciences, Research Institute for Technical Physics and Materials Science, H-1525 Budapest P.O. Box 49. Hungary

[e] University of Pécs, Department of Inorganic Chemistry and MTA-PTE Research Group for Selective Chemical Syntheses, Ifjúság u. 6. (P.O.Box 266) H-7624 Pécs, Hungary

† Present address: Applied Physics, Division of Materials Science, Department of Engineering Science and Mathematics, Luleå University of Technology, 97187 Luleå, Sweden

---

I. Analytical measurements

Reaction mixtures were analysed by gas chromatography (Hewlett Packard 5890) and GC-MS (Hewlett Packard 5971A GC-MSD, HP-1 column).
The palladium-content of the catalysts and palladium leaching were determined by ICP.
FT-IR spectra were measured on a BRUKER Vertex 70 type spectrometer with a Bruker Platinum ATR adapter without sample preparation. The spectra were recorded at a resolution of 2 cm^{-1} with a room temperature DTGS detector (512 scans were co-added).
Surface compositions of **CAT-1** and **CAT-2** before ("fresh") and after ("spent") the catalytic test reaction were determined by X-ray photoelectron spectroscopy (XPS) performed by a KRATOS XSAM 800 XPS machine. Al Kα characteristic X-ray line, 40 eV pass energy and FAT mode were applied for recording the XPS lines of *Pd 3d*, *C 1s*, *O 1s*, *Br 3d*, *I 3d*, *N 1s* and *Si 2p*. *C 1s* binding energy at 284.8 eV was used as reference for charge compensation. The surface concentrations of the elements were calculated from the integral intensities of the XPS lines using sensitivity factors given by the manufacturer.
Transmission Electron Microscope (TEM) investigations were carried out by a JEOL 3010 high resolution TEM operating at 300 kV, with a point resolution of 0.17 nm. The microscope was equipped with a GATAN Tridiem energy filter used for electron energy loss spectroscopy (EELS) elemental mapping. The samples were suspended in ethanol and drop-dried on carbon-coated microgrids for the measurements of the microstructure of the catalyst particles and their distribution over the support.

II. Preparation of supported Pd catalysts

Preparation of 3-(2-bromoethyl)-1-methylimidazolium bromide

The 3-(2-bromoethyl)-1-methylimidazolium bromide was prepared according to a literature procedure.[33] 1,2-Dibromoethane (3.2 ml, 36.25 mmol) was added to a solution of 1-methylimidazole (500 µl, 6.13 mmol) in diethyl ether (3.5 ml) under Ar atmosphere, and the resulting mixture was stirred at room temperature for 4 days. During this time, a white precipitate was formed. It was collected by filtration and washed with diethyl ether (3×1 ml) and dried *in vacuo*. The filtrate was stirred at room temperature for further 24 hours to give a second crop of crystals. 3-(2-Bromoethyl)-1-methylimidazolium bromide was isolated as a hygroscopic white solid (0.90 g, 53%) and it was stored under argon. ^{1}H NMR (400 MHz, DMSO-d_6) δ 9.23 (s, 1H), 7.87 – 7.81 (m, 1H), 7.78 – 7.73 (m, 1H), 4.63 (t, *J* = 5.8 Hz, 2H), 3.95 (t, *J* = 5.8 Hz, 2H), 3.89 (s, 3H).

Preparation of imidazole functionalized silica

Compound 2 was prepared by a modified method described in the literature.[32] To a solution of (3-aminopropyl)triethoxysilane (2.3 ml, 10 mmol) in inert toluene (36 ml), 5.0 g silica (pre-treated by heating for 5 h at 250 °C) was added. The mixture was refluxed for 72 h. Then the white material was filtered and washed with toluene (3×5 ml) and diethyl ether (3×5 ml). The solid was dried to constant weight (8 h) *in vacuo* to produce **1**. The aminopropyl group content of the silica was determined by measuring the weight increase of the material (0.73 mmol/g modified silica). 1.0 g of **1** was suspended in 10 ml methanol and 2.8 mmol glyoxal (40% solution in water) was added and was stirred overnight. 5.6 mmol NH_4Cl and 5.6 mmol formaldehyde were added and it was diluted with 30 ml methanol. This was refluxed for an hour and H_3PO_4 (0.5 ml, 85%) was slowly added to the mixture before heating to reflux for 12 hours. The resulting material was filtered and washed with methanol (3×10 ml) and diethyl ether (3×10 ml). It was then dried *in vacuo* at 60 °C to constant weight (8 h) to give a yellowish solid product with 0.72 mmol/g imidazole functionality (determined by elemental analysis from the nitrogen content of the solid material: calculated for 0.72 mmol/g $C_8H_{14}N_2OSi$: C: 6.93; H: 1.01; N: 2.02; found C: 6.34; H: 0.90; N: 2.02).

Preparation of SILP phase 3

To a solution of 3-(2-bromoethyl)-1-methylimidazolium bromide (405 mg, 1.5 mmol) in 20 ml acetonitrile, 685 mg of **2** was added and the resulting mixture was refluxed for 5 days. Then the solid was filtered and washed with acetonitrile (3×5 ml), methanol (3×5 ml), acetone (3×5 ml) and diethyl ether (3×5 ml). The solid was dried to constant weight (8 h) *in vacuo* to produce **3**, with 0.40 mmol/g dicationic moieties (determined by measuring the weight increase of the material). Elemental analysis: C: 9.28; H: 1.21; N: 3.11.

Preparation of CAT-1

29.0 µmol (30.0 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 200 mg of **3** was added and the resulting mixture was stirred overnight. The solvents were removed *in vacuo* and the residue was dried *in vacuo* at 35 °C for 3 h and the catalyst was obtained as a dark grey solid. Palladium content of the catalyst: 2.64% (determined by ICP).

Preparation of CAT-2

To a solution of $Pd(OAc)_2$ (13.0 mg, 58.0 µmol) and potassium *tert*-butoxide (6.5 mg, 58.0 µmol) in 4 ml ethanol, 200 mg of **3** was added and the resulting mixture was stirred at room temperature overnight. It was filtered and washed with ethanol (3×2 ml) and diethyl ether (3×2 ml). The catalyst was dried *in vacuo* at 35 °C for 3 h and was obtained as a brown solid. Palladium content of the catalyst: 1.66% (determined by ICP).

III. General procedure for aminocarbonylation reactions

Catalytic reactions at atmospheric pressure

In a typical experiment the catalyst (with 2.8 µmol Pd content) was placed in a Schlenk tube. The atmosphere was changed to carbon monoxide. A solution of 0.2 mmol (22 µl) iodobenzene, 0.5 mmol (44 µl) morpholine and 0.25 mmol (35 µl) triethylamine in 1 ml DMF was added via septum. The mixture was stirred in an oil bath for 8 hours at 100 °C. After cooling to room temperature, the liquid phase was removed with a syringe. The reaction mixture was analysed by GC and the catalyst was reused.

General procedure for solvent-free reactions. The catalyst (with 2.8 µmol Pd content) was placed in a Schlenk tube. The atmosphere was changed to carbon monoxide. 0.4 mmol (45 µl) iodobenzene, 1.0 mmol (87 µl) morpholine and 0.5 mmol (70 µl) triethylamine was added via septum. The mixture was stirred in an oil bath for 8 hours at 100 °C. After cooling to room temperature, the products were extracted with 3×1 ml toluene. The reaction mixture was analysed by GC and the catalyst was reused.

Catalytic reactions at elevated pressure

In a typical experiment the catalyst (with 2.8 µmol Pd content) was placed in a stainless steel autoclave. A solution of 0.2 mmol (22 µl) iodobenzene, 0.5 mmol (44 µl) morpholine and 0.25 mmol (35 µl) triethylamine in 1 ml DMF was added via syringe. The autoclave was charged with carbon monoxide (5-30 bar) and was heated with stirring in an oil bath for 3 and 8 hours at 100 °C. After cooling to room temperature, the liquid phase was removed with a syringe. The reaction mixture was analysed by gas chromatography and the catalyst was reused.

General procedure for solvent-free reactions. In a typical experiment the catalyst (with 2.8 µmol Pd content) was placed in a stainless steel autoclave. 0.4 mmol (45 µl) iodobenzene, 1.0 mmol (87 µl) morpholine and 0.5 mmol (56.1 mg) DABCO was added. It was charged with carbon monoxide (5-30 bar) and was heated with stirring in an oil bath for 3 and 8 hours at 120 °C. After cooling to room temperature, the products were extracted with 3×1 ml toluene. The reaction mixture was analysed by GC and the catalyst was reused. The catalyst was washed with 2×1 ml methanol before recycling in every two runs to remove the ammonium salt formed during the reaction.

IV. Characterization of SILP phase 3

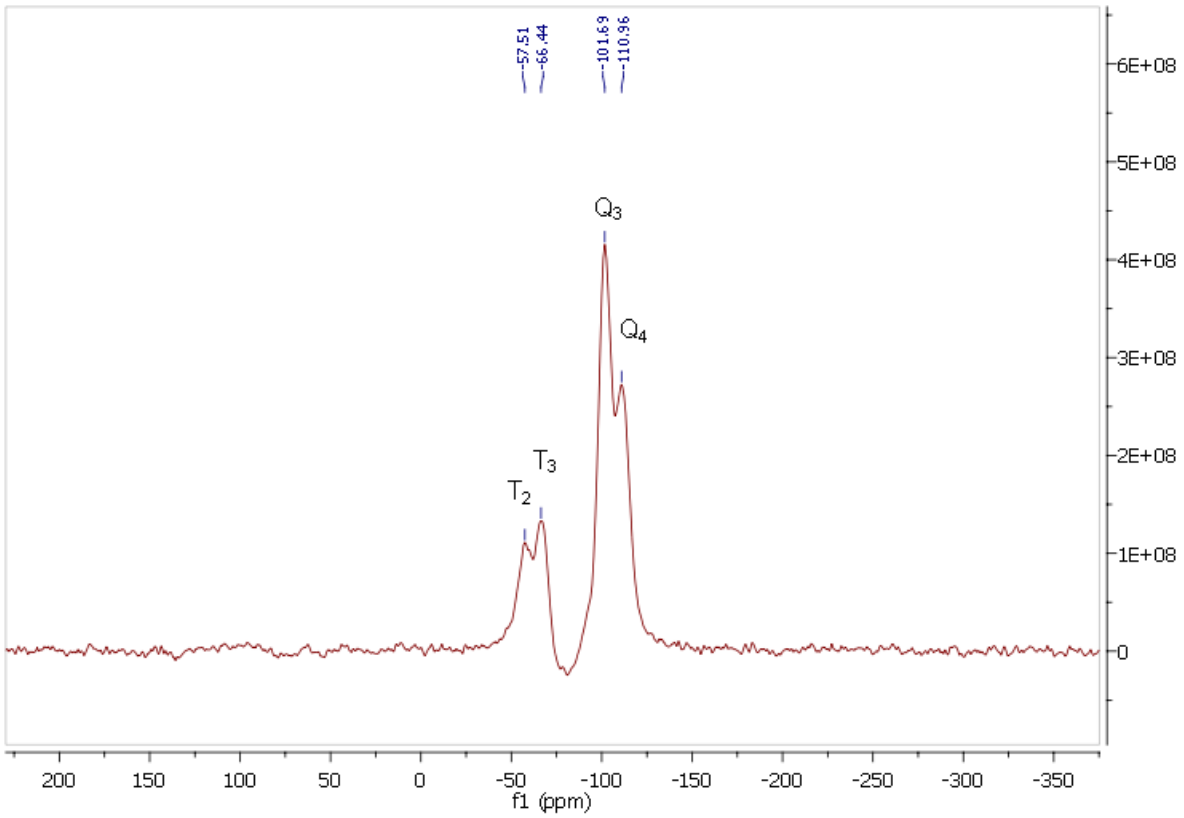


Figure S1. The ^{29}Si CP MAS NMR spectrum of **3** (T_2: $Si(OSi)_2ROH$, T_3: $Si(OSi)_3R$, Q_3 : $Si(OSi)_3OH$, and Q_4: $Si(OSi)_4$)

V. TEM images of fresh and spent catalysts

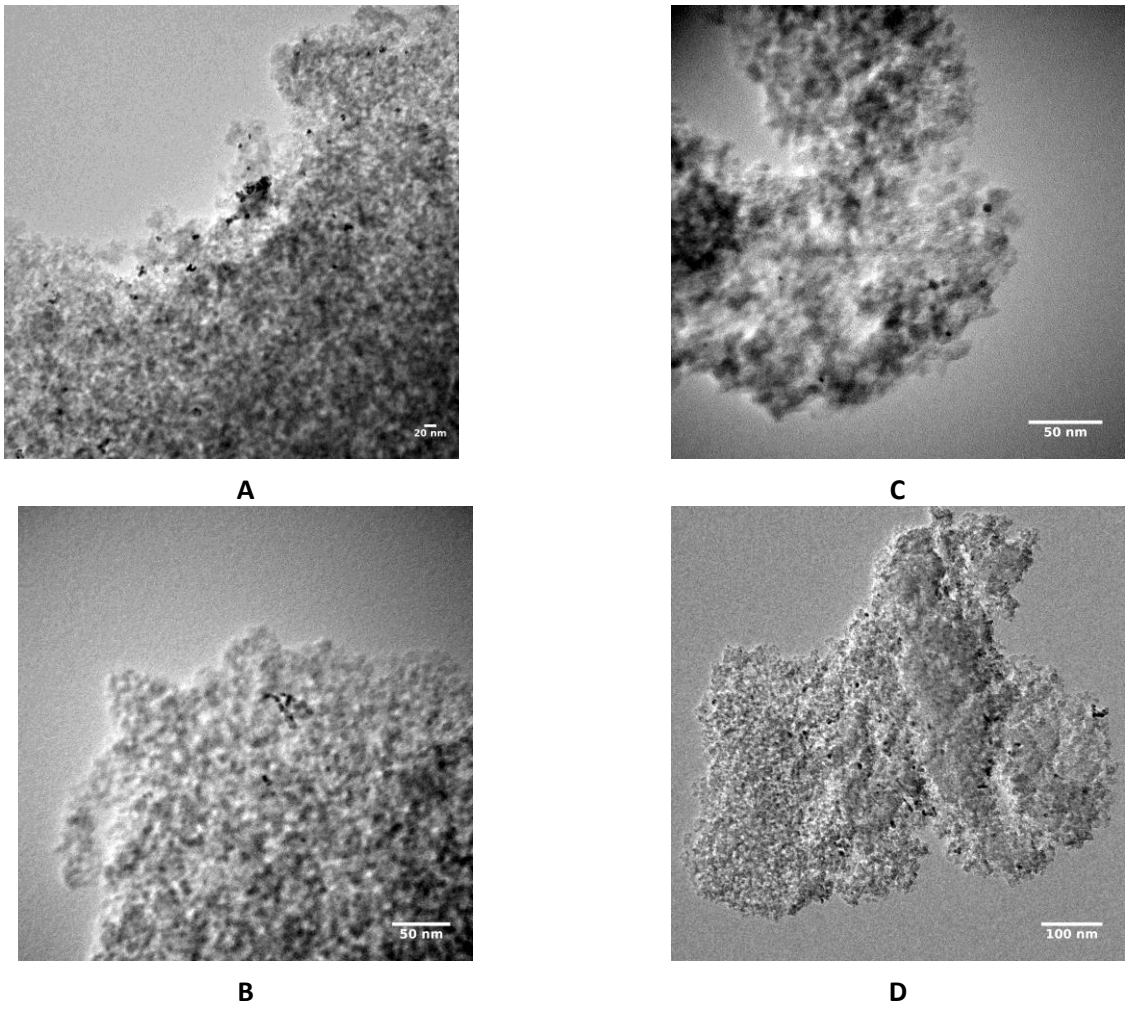


Figure S2. TEM images of **CAT-1** (A: fresh, B: spent) and **CAT-2** (C: fresh, D: spent)

VI. XPS spectra of fresh and spent catalysts

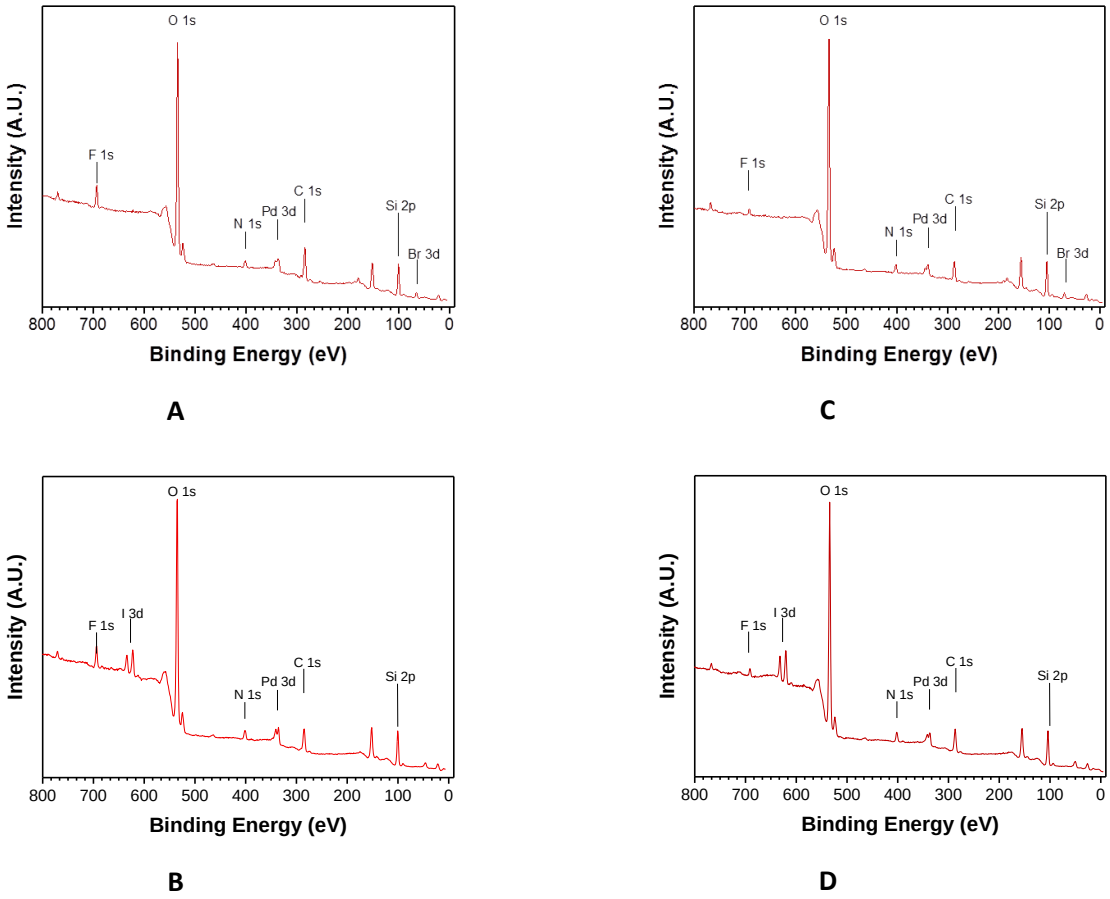


Figure S3. Wide scan XPS spectra of Pd catalyst **CAT-1** (A: fresh, B: spent) and **CAT-2** (C: fresh, D: spent)

VII. Recycle study of catalyst CAT-1

Table S1. Recycling experiments with **CAT-1**

entry	CO pressure [bar]	r. time [h]	solvent	base	conversion of iodobenzene [%][b]									
					run 1	run 2	run 3	run 4	run 5	run 6	run 7	run 8	run 9	run 10
1	30	3	DMF	Et_3N	100	100	100	100	100	97	90	85	66	47
2	1	8	DMF	Et_3N	74	40	26	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3	30	3	acetonitrile	Et_3N	88	87	90	78	76	78	79	73	75	78
4	30	3	toluene	Et_3N	53	51	56	57	50	n.d.	n.d.	n.d.	n.d.	n.d.
5	30	3	toluene	DBU	100	99	90	91	77	n.d.	n.d.	n.d.	n.d.	n.d.
6	30	8	toluene	DBU	100	100	100	100	100	100	100	95	30	29

[a] :Reaction conditions: 0.2 mmol **4a**, 0.5 mmol **5ª**, 0.25 mmol base, catalyst (CAT-1, 2.8 µmol Pd, 1.4 mol%), 100 °C. [b] :determined by GC.

Table S2. Selectivity of aminocarbonylation of iodobenzene with morpholine during recycling experiments in the presence **CAT-1**[a]

entry	CO pressure [bar]	r. time [h]	solvent	base	selectivity for **7a**[b]									
					run 1	run 2	run 3	run 4	run 5	run 6	run 7	run 8	run 9	run 10
1	30	3	DMF	Et_3N	95	94	97	98	96	96	97	98	99	100
2	1	8	DMF	Et_3N	76	61	50	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
3	30	3	acetonitrile	Et_3N	95	94	95	96	98	95	96	95	95	98
4	30	3	toluene	Et_3N	28	29	26	27	30	n.d.	n.d.	n.d.	n.d.	n.d.
5	30	3	toluene	DBU	87	87	86	84	85	n.d.	n.d.	n.d.	n.d.	n.d.
6	30	8	toluene	DBU	95	91	91	89	87	93	88	90	77	56

[a] :Reaction conditions: 0.2 mmol **4a**, 0.5 mmol **5ª**, 0.25 mmol base, catalyst (CAT-1, 2.8 µmol Pd, 1.4 mol%), 100 °C. [b] :determined by GC.

VIII. Recycle study of catalyst CAT-2

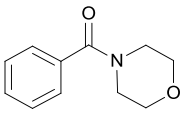
Table S3. Selectivity of aminocarbonylation of iodobenzene with morpholine during recycling experiments in the presence **CAT-2**[a]

entry	CO pressure [bar]	selectivity for **7a**[b]									
		run 1	run 2	run 3	run 4	run 5	run 6	run 7	run 8	run 9	run 10
1	5	65	85	90	90	90	88	92	92	92	90
2	10	86	88	90	89	94	93	95	93	93	93
3	20	95	93	93	96	97	96	96	96	97	96
4	30	97	97	99	98	99	98	98	98	99	97

[a] :Reaction conditions: 0.2 mmol **4a**, 0.5 mmol **5a**, 0.25 mmol Et_3N, 1.0 ml DMF, catalyst (**CAT-2**, 2.8 µmol Pd, 1.4 mol%), 100 °C, 3h, [b] : determined by GC.

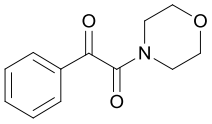
IX. Characterisation of products

Morpholino(phenyl)methanone (**6a, Table 9, entry 1**):



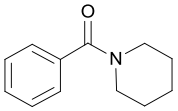
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.44 – 7.36 (m, 5H), 3.92 – 3.56 (m, 6H), 3.56 – 3.34 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 170.4, 135.4, 129.8, 128.6, 127.1, 66.9 (2C), 48.1, 42.6. 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 (7a, Table 6, entry 1):



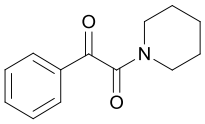
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.96 (dd, J = 8.3, J = 1.3 Hz, 2H), 7.66 (tt, J = 7.4, J = 1.3 Hz, 1H), 7.56 – 7.50 (m, 2H), 3.83 – 3.76 (m, 4H), 3.69 – 3.63 (m, 2H), 3.41 – 3.36 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 191.1, 165.5, 134.9, 133.1, 129.7, 129.1, 66.8, 66.7, 46.3, 41.6. 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 9, entry 2):



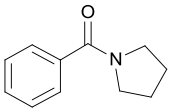
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.41 – 7.32 (m, 5H); 3.79 – 3.58 (m, 2H); 3.43 – 3.22 (m, 2H); 1.75 – 1.57 (m, 4H); 1.57-1.42 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 170.3, 136.6, 129.3, 128.4, 126.8, 48.7, 43.1, 26.5, 25.7, 24.6. 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 (Table 6, entry 2):



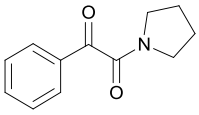
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.97 – 7.88 (m, 2H), 7.65 – 7.58 (m, 1H), 7.53 – 7.46 (m, 2H), 3.73 – 3.64 (m, 2H), 3.31 – 3.23 (m, 2H), 1.74 – 1.63 (m, 4H), 1.59 – 1.49 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 192.1, 165.6, 134.8, 133.4, 129.7, 129.1, 47.2, 42.3, 26.3, 25.6, 24.5. 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 9, entry 3):



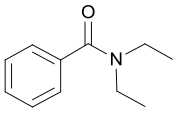
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.55 – 7.44 (m, 2H), 7.42 – 7.32 (m, 3H), 3.62 (t, J = 6.4 Hz, 2H), 3.39 (t, J = 6.4 Hz, 2H), 2.00 – 1.88 (m, 2H), 1.88 – 1.76 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 169.69, 137.30, 129.71, 128.21, 127.05, 49.55, 46.12, 26.36, 24.43. 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 (Table 6, entry 3).



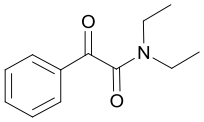
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 8.01 – 7.96 (m, 2H), 7.66 – 7.60 (m, 1H), 7.52 – 7.47 (m, 2H), 3.68 – 3.62 (m, 2H), 3.45 – 3.39 (m, 2H), 1.99 – 1.90 (m, 4H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 191.1, 164.5, 134.1, 132.5, 129.4, 128.5, 46.2, 44.8, 25.4, 23.5. MS(m/z/rel.int.): 203(M^+)/3; 202/6; 105/71; 98/100; 77/52; 70/31; 55/56.

N,N-Diethylbenzamide (Table 9, entry 4):



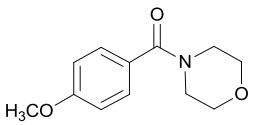
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.39 – 7.35 (m, 5H); 3.54 (brs, 2H); 3.25 (brs, 2H); 1.24 (brs, 3H); 1.10 (brs, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 171.3, 137.3, 129.1, 128.4, 126.3, 43.3, 39.2, 14.2, 12.9. MS(m/z/rel.int.): 177(M^+)/13; 176/36; 105/100; 77/39; 51/11.

N,N-Diethyl-2-oxo-2-phenylacetamide (Table 6, entry 4).



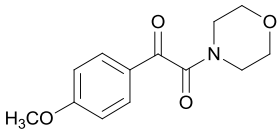
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 8.00 – 7.93 (m, 2H), 7.69 – 7.60 (m, 1H), 7.55 – 7.49 (m, 2H), 3.59 (q, J = 7.2 Hz, 2H), 3.26 (q, J = 7.2 Hz, 2H), 1.31 (t, J = 7.2 Hz, 3H), 1.17 (t, J = 7.2 Hz, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 191.6, 166.7, 134.5, 133.3, 129.6, 128.9, 42.1, 38.8, 14.1, 12.8. MS(m/z/rel.int.): 205(M^+)/5; 105/61; 100/100; 77/42; 72/74; 51/21.

Morpholino(4-methoxyphenyl)methanone (**Table 9, entry 11**):



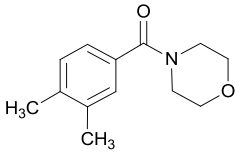
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.38 (d, J = 8.9 Hz, 2H), 6.91 (d, J = 8.9 Hz, 2H), 3.83 (s, 3H), 3.75 – 3.52 (m, 8H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 170.5, 161.0, 129.2, 127.3, 113.8, 66.9 (2C), 55.4, 42.9. MS(m/z/rel.int.): 221(M^+)/10; 220/16; 135/100; 107/8; 92/9; 77/15; 64/5; 56/3.

1-Morpholino-2-(4-methoxyphenyl)ethane-1,2-dione (Table 6, entry 5):



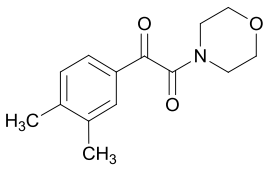
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.96 (d, J = 8.9 Hz, 3H), 7.01 (d, J = 8.9 Hz, 2H), 3.92 (s, 3H), 3.84 – 3.77 (m, 4H), 3.70 – 3.64 (m, 2H), 3.44 – 3.38 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 189.8, 165.8, 165.0, 132.2, 126.2, 114.4, 66.8, 66.7, 55.7, 46.3, 41.6. 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 9, entry 12**):



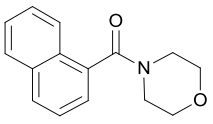
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.19 (brs, 1H), 7.16 – 7.09 (m, 2H), 3.88 – 3.32 (m, 8H), 2.28 (s, 6H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 170.8, 138.7, 137.0, 132.8, 129.6, 128.4, 124.5, 66.9 (2C), 48.3, 42.7, 19.7, 19.7. 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 (Table 6, entry 6):



The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.72 (brs, 1H), 7.68 (dd, J = 7.8, 1.9 Hz, 1H), 7.27 (d, J = 7.8 Hz, 1H), 3.81 – 3.77 (m, 4H), 3.67 – 3.62 (m, 2H), 3.39 – 3.34 (m, 2H), 2.34 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 191.2, 165.8, 145.1, 137.7, 131.0, 130.5, 130.4, 127.5, 66.7, 66.7, 46.3, 41.6, 20.3, 19.7. MS(m/z/rel.int.): 247(M^+)/3; 133/100; 105/24; 79/9; 77/9; 70/9.

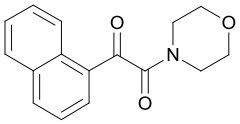
Morpholino(naphth-1-yl)methanone (**Table 9, entry 13**):



The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene: acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.91 – 7.84 (m, 3H), 7.58 – 7.47 (m, 3H), 7.45 – 7.40 (m, 1H), 4.06 – 3.98 (m, 1H), 3.94 – 3.83 (m, 3H), 3.56 – 3.48

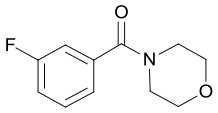
(m, 2H), 3.26 – 3.14 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 169.5, 133.7, 133.5, 129.6, 129.4, 128.5, 127.2, 126.5, 125.2, 124.6, 123.9, 67.1, 67.0, 47.6, 42.2. MS(m/z/rel.int.): 241(M^+)/38; 240/23; 156/23; 155/100; 127/81; 86/8; 77/8.

1-(naphth-1-yl)-2-morpholinoethane-1,2-dione (Table 6, entry 7):



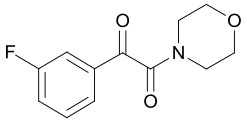
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 9.25 (d, J = 8.7 Hz, 1H), 8.13 (d, J = 8.2 Hz, 1H), 8.04 (dd, J = 7.2, J = 0.9 Hz, 1H), 7.93 (d, J = 8.2 Hz, 1H), 7.75 – 7.68 (m, 1H), 7.64 – 7.53 (m, 2H), 3.89 – 3.80 (m, 4H), 3.72 – 3.64 (m, 2H), 3.49 – 3.42 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 193.6, 166.1, 136.1, 134.5, 134.1, 130.9, 129.5, 128.8, 128.5, 127.1, 125.8, 124.5, 66.7 (2C), 46.4, 41.8. 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 9, entry 14**):



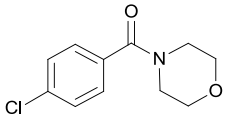
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene: acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.42 – 7.36 (m, 1H), 7.19 – 7.17 (m, 1H), 7.15 – 7.11 (m, 2H), 3.96 – 3.54 (m, 6H), 3.54 – 3.29 (m, 2H). ^{13}C NMR (125.78 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 (2C), 48.1, 42.6. 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 (Table 6, entry 8):



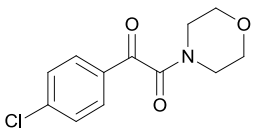
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.74 – 7.72 (m, 1H), 7.67 – 7.64 (m, 1H), 7.50 (td, J = 8.0, J = 5.4 Hz, 1H), 7.35 (tdd, J = 8.2, J = 2.6, J = 0.8 Hz, 1H), 3.80 – 3.76 (m, 4H), 3.69 – 3.63 (m, 2H), 3.41 – 3.34 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 189.7 (d, J = 2.2 Hz), 164.8, 162.9 (d, J = 249.6 Hz), 135.2 (d, J = 6.4 Hz), 130.9 (d, J = 7.6 Hz), 125.7 (d, J = 3.1 Hz), 122.0 (d, J = 21.6 Hz), 116.0 (d, J = 22.6 Hz), 66.7, 66.6, 46.3, 41.7. 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(4-chlorophenyl)methanone (**Table 9, entry 15**):



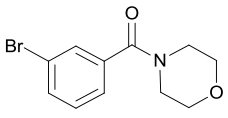
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.38 (d, J = 8.2 Hz, 2H), 7.34 (d, J = 8.2 Hz, 2H), 4.00 – 3.53 (m, 6H), 3.53 – 3.24 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 169.3, 136.0, 133.7, 128.8, 128.7, 66.8 (2C), 48.4, 42.9. MS(m/z/rel.int.): 227(M^+)/3; 226/4; 225(M^+)/10, 224/20;141/32; 140/8; 139/100; 113/13; 111/36; 86/23; 76/7; 75/29; 56/34; 44/29.

1-(4-Chlorophenyl)-2-morpholinoethane-1,2-dione (Table 6, entry 9):



The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 3:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.91 (d, J = 8.5 Hz, 2H), 7.50 (d, J = 8.5 Hz, 2H), 3.85 – 3.76 (m, 4H), 3.72 – 3.65 (m, 2H), 3.44 – 3.38 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 189.7, 164.9, 141.6, 131.5, 131.0, 129.5, 66.8, 66.7, 46.3, 41.7. MS(m/z/rel.int.): 253(M^+)/3; 141/33; 140/8; 139/100; 114/53; 113/9; 111/29; 86/14; 76/6; 75/22; 70/70; 56/8; 50/8; 42/29.

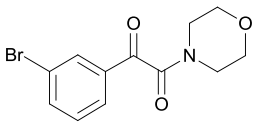
Morpholino(3-bromophenyl)methanone (**Table 9, entry 16**):



The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene: acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.59 – 7.51 (m, 2H), 7.34 – 7.26 (m, 2H), 3.91 – 3.53 (m, 6H), 3.53 – 3.28 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 168.7, 137.2, 133.0, 130.2, 130.1, 125.6, 122.7, 66.8, 66.8, 48.2, 42.6. MS(m/z/rel.int.):

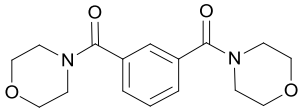
271(M^+)/26; 270/55; 269(M^+)/26; 268/55; 256/16; 254/16; 185/92; 183/92; 157/42; 55/42; 104/14; 86/74; 77/12; 76/63; 75/42; 74/11; 72/16; 56/100; 55/11; 50/30; 42/24.

1-(3-Bromophenyl)-2-morpholinoethane-1,2-dione (Table 6, entry 10):



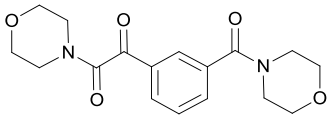
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 8.09 (dd, J = 3.9, 2.1 Hz, 1H), 7.91 – 7.85 (m, 1H), 7.79 – 7.74 (m, 1H), 7.40 (td, J = 7.9, 2.1 Hz, 1H), 3.81 – 3.76 (m, 4H), 3.67 – 3.65 (m, 2H), 3.39 – 3.37 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 189.5, 164.6, 137.7, 134.9, 132.4, 130.6, 128.3, 123.4, 66.7, 66.6, 46.3, 41.8. 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) (8b, Table 9, entry 17):



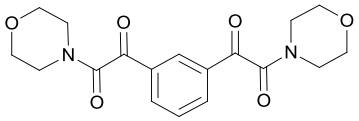
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate:ethanol = 10:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.48 – 7.35 (m, 4H), 3.91 – 3.22 (m, 16H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 169.3, 135.9, 128.9, 128.4, 125.7, 66.8 (4C), 48.2 (2C), 42.6 (2C). 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 (**9b, Table 9, entry 17**):



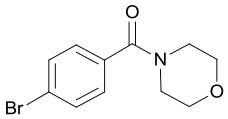
MS(m/z/rel.int.): 332(M^+)/22; 304/14; 218/100; 133/35; 114/86; 104/24; 86/12; 76/27; 70/67; 56/16; 42/25.

1,3-Phenylenebis(2-morpholinoethane-1,2-dione) (**10b, Table 9, entry 17**):



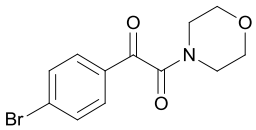
MS(m/z/rel.int.): 360(M^+)/14; 246/100; 133/12; 114/96; 104/17; 78/13; 76/20; 70/84; 45/11; 42/32.

Morpholino(4-bromophenyl)methanone (**Table 9, entry 19**):



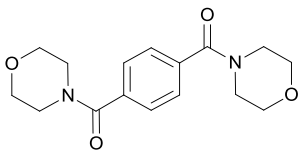
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene: acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.53 (d, J = 8.2 Hz, 2H), 7.27 (d, J = 8.2 Hz, 2H), 3.81 – 3.52 (m, 6H), 3.52 – 3.33 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 169.3, 134.2, 131.8, 128.8, 124.2, 66.8 (2C), 48.2, 42.7. MS(m/z/rel.int.): 271(M^+)/17; 269(M^+)/17; 268/42; 185/99; 183/100; 157/28; 155/22; 104/12; 86/41; 76/34; 75/26; 72/11; 56/54; 50/18; 42/12

1-(4-Bromophenyl)-2-morpholinoethane-1,2-dione (Table 6, entry 11):



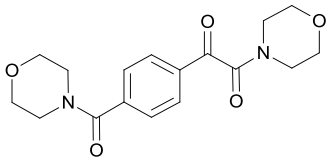
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.83 (d, J = 8.6 Hz, 2H), 7.67 (d, J = 8.6 Hz, 2H), 3.83 – 3.75 (m, 4H), 3.69 – 3.63 (m, 2H), 3.41 – 3.35 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 189.9, 164.9, 132.5, 131.9, 131.1, 130.5, 66.8, 66.7, 46.3, 41.7. MS(m/z/rel.int.): 299(M^+)/6; 297(M^+)/6; 185/100; 183/100; 157/20; 155/20; 114/93; 86/31; 76/22; 75/19; 70/83; 42/27.

1,4-Phenylenebis(morpholinomethanone) (8c, Table 9, entry 19):



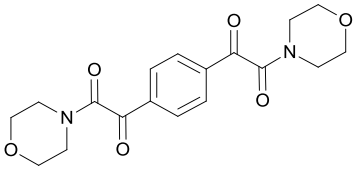
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate:ethanol = 10:1). ^{1}H NMR (400 MHz, $CDCl_3$): 7.44 (s, 4H), 3.86 – 3.34 (m, 16H). ^{13}C NMR (100 MHz, $CDCl_3$): 169.0, 136.4, 126.9, 66.3 (4C), 47.7 (2C), 42.1 (2C). MS(m/z/rel.int.): 304(M^+)/6; 191/11; 190/100; 91/5; 77/6.

1-(4-Morpholinocarbonyl)phenyl-2-morpholinoethane-1,2-dione (**9c, Table 9, entry 19**):



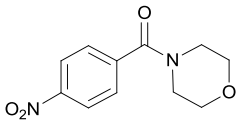
MS(m/z/rel.int.): 332(M^+)/8; 304/10; 219/13; 218/100; 208/7; 114/31; 104/17; 76/9; 70/26; 56/6; 42/10.

1,4-Phenylenebis(2-morpholinoethane-1,2-dione) (**10c, Table 9, entry 19**):



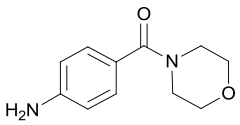
MS(m/z/rel.int.): 360(M^+)/16; 332/12; 247/14; 246/100; 218/8; 114/98; 104/21; 76/12; 70/71; 45/5; 42/22.

Morpholino(4-nitrophenyl)methanone (**Table 6, entry 12**):



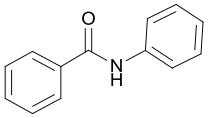
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene: acetone = 4:1). ^{1}H NMR (500.15 MHz, $CDCl_3$): 8.29 (d, J = 8.7 Hz, 2H), 7.58 (d, J = 8.7 Hz, 2H), 3.89 – 3.57 (m, 6H), 3.48 – 3.30 (m, 2H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 168.1, 148.5, 141.4, 128.2, 124.0, 66.7 (2C), 48.0, 42.6. MS(m/z/rel.int.): 236(M^+)/17; 235/29; 221/24; 219/6; 205/9; 189/7; 151/7; 150/67; 134/5; 120/26; 104/46; 92/19; 86/41; 76/44; 75/13; 56/100; 50/20; 42/18.

4-Aminophenyl(morpholino)methanone (**Table 6, entry 12**):



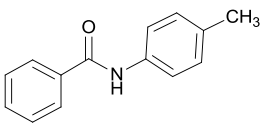
Side product obtained in the aminocarbonylation reaction of 4-iodonitrobenzene and morpholine with a yield of 52% and 44% (GC) in the 1st and 2nd runs, respectively. MS(m/z/rel.int.): 206(M^+)/13; 205/9; 121/8; 120/100; 93/2; 92/18; 91/2; 66/2; 65/17; 64/2; 56/2; 39/5.

N-Phenylbenzamide (Table 7, entry 1):



The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (ethyl acetate). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.90 – 7.85 (m, 2H), 7.82 (s, 1H), 7.67 – 7.62 (m, 2H), 7.58 – 7.53 (m, 1H), 7.52 – 7.46 (m, 2H), 7.40 – 7.35 (m, 2H), 7.19 – 7.13 (m, 1H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 165.9, 137.9, 135.0, 131.8, 129.1, 128.8, 127.0, 124.6, 120.3. MS(m/z/rel.int.): 197(M^+)/42; 105/100; 77/52; 51/14.

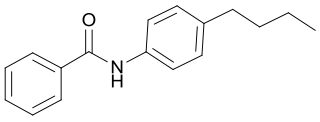
N-(4-Methylphenyl)benzamide (Table 7, entry 2):



The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 5:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.87 (d, J = 8.2 Hz, 2H), 7.77 (brs, 1H), 7.58 – 7.45 (m, 5H), 7.18 (d, J = 8.2 Hz, 2H), 2.34 (s, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 165.6, 135.4, 135.1, 134.3, 131.7, 129.6, 128.8, 127.0, 120.3, 20.9.

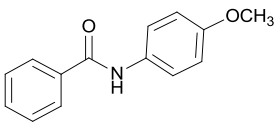
MS(m/z/rel.int.): 211(M+)/39; 150/100; 77/47; 51/9.

N-(4-Butylphenyl)benzamide (Table 7, entry 3):



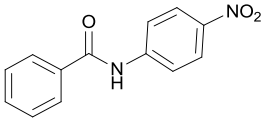
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene: ethyl acetate = 5:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.87 (d, J = 8.3 Hz, 2H), 7.78 (brs, 1H), 7.58 – 7.45 (m, 5H), 7.18 (d, J = 8.3 Hz, 2H), 2.60 (t, J = 7.8 Hz, 2H), 1.64 – 1.54 (m, 4H), 1.41 – 1.30 (m, 2H), 0.93 (t, J = 7.3 Hz, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 165.6, 139.4, 135.5, 135.2, 131.7, 129.0, 128.8, 127.0, 120.3, 35.1, 33.6, 22.3, 13.9. MS(m/z/rel.int.): 253(M^+)/57; 210/32; 105/100; 77/46; 51/5.

N-(4-Methoxyphenyl)benzamide (Table 7, entry 4):



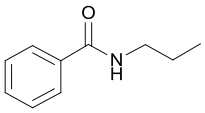
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 5:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.89 – 7.83 (m, 2H), 7.75 (brs, 1H), 7.58 – 7.51 (m, 3H), 7.51 – 7.45 (m, 2H), 6.95 – 6.87 (m, 2H), 3.82 (s, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 165.7, 156.7, 135.1, 131.7, 131.0, 128.7, 127.0, 122.2, 114.3, 55.5. MS(m/z/rel.int.): 227(M^+)/51; 122/5; 105/100; 95/4; 77/38; 51/6.

N-(4-Nitrophenyl)benzamide (Table 7, entry 5):



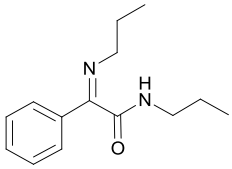
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 5:2). ^{1}H NMR (500.15 MHz, DMSO-d6): 10.80 (s, 1H), 8.29 – 8.25 (m, 2H), 8.09 – 8.05 (m, 2H), 8.02 – 7.94 (m, 2H), 7.67 – 7.60 (m, 1H), 7.60 – 7.52 (m, 2H). ^{13}C NMR (125.78 MHz, DMSO-d6): 166.8, 146.0, 143.0, 134.7, 132.6, 129.0, 128.4, 125.2, 120.3. MS(m/z/rel.int.): 242(M^+)/7; 105/100; 77/51; 51/8.

N-Propylbenzamide (Table 9, entry 5):



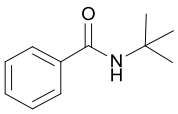
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 5:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.79 – 7.72 (m, 2H), 7.51 – 7.44 (m, 1H), 7.44 – 7.36 (m, 2H), 6.27 (brs, 1H), 3.45 – 3.36 (m, 2H), 1.68 – 1.58 (m, 2H), 0.97 (td, J = 7.4, 1.8 Hz, 3H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 167.6, 134.9, 131.2, 128.5, 126.8, 41.8, 22.9, 11.4. MS(m/z/rel.int.): 163(M^+)/12; 134/6; 105/100; 77/50; 51/22.

N-propyl-benzoylformamide N'-propylimine (11d, Table 9, entry 5):



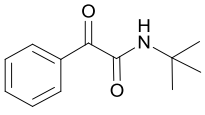
MS(m/z/rel.int.): 232(M^+)/0.8; 203/10; 146/24; 130/4; 104/100; 77/17; 43/22; 41/21.

N-*tert*-Butylbenzamide (Table 9, entry 6):



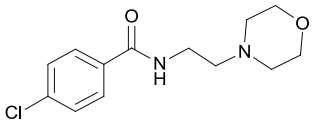
The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (toluene:ethyl acetate = 5:2). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.72 (d, J = 7.5 Hz, 2H), 7.46 (t, J = 7.5 Hz, 1H), 7.40 (t, J = 7.5 Hz, 2H), 5.94 (brs, 1H), 1.47 (s, 9H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 166.9, 136.0, 131.0, 128.5, 126.7, 51.6, 28.9. MS(m/z/rel.int.): 177(M^+)/9; 162/11; 122/10; 105/100; 77/50; 51/21.

N-*tert*-Butyl-2-oxo-2-phenylacetamide (Table 9, entry 6):



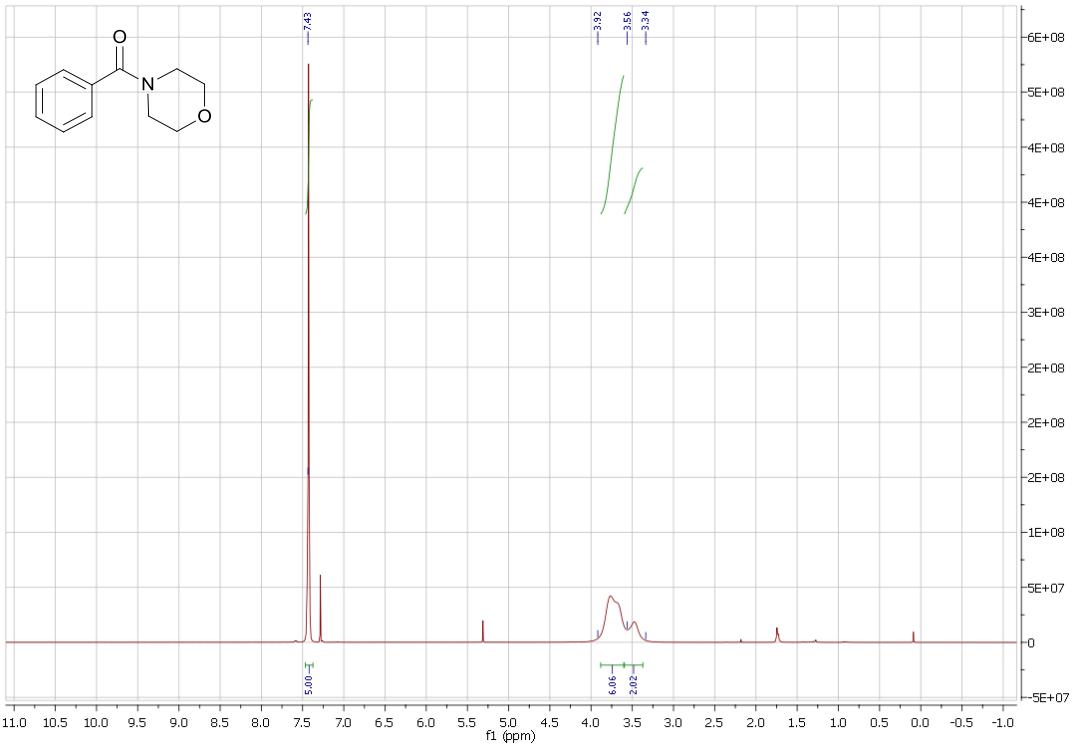
MS(m/z/rel.int.): 205(M^+)/6; 162/3; 149/3; 105/100; 77/37; 57/75; 51/21; 44/21.

4-Chloro-N-(2-morpholinoethyl)benzamide (*Moclobemide,* Table 9, entry 20):

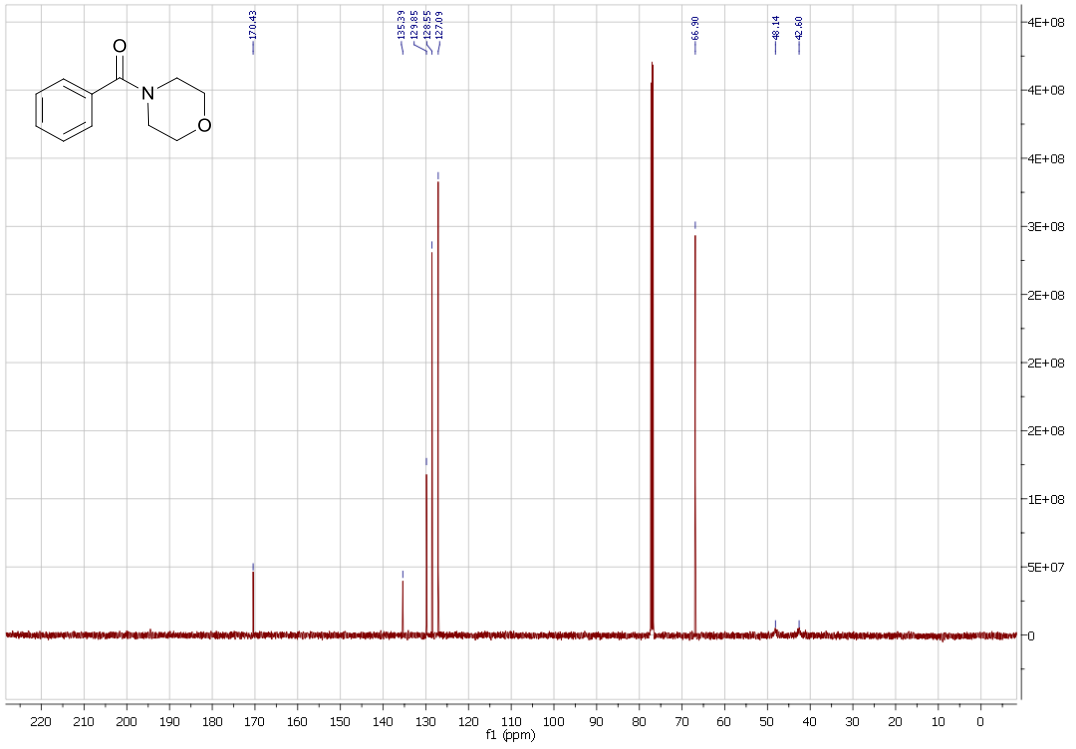


The solvent was evaporated from the crude reaction mixture and it was purified by column chromatography on silica gel (acetone). ^{1}H NMR (500.15 MHz, $CDCl_3$): 7.71 (d, J = 8.5 Hz, 2H), 7.41 (d, J = 8.5 Hz, 2H), 3.78 – 3.69 (m, 4H), 3.54 (dd, J = 11.2, J = 5.6 Hz, 2H), 2.61 (t, J = 6.0 Hz, 2H), 2.56 – 2.47 (m, 4H). ^{13}C NMR (125.78 MHz, $CDCl_3$): 166.3, 137.7, 133.0, 128.8, 128.3, 67.0 (2C), 56.9, 53.3 (2C), 36.1. MS(m/z/rel.int.): 139/7; 113/11; 100/100; 75/5; 70/6; 56/14; 42/9.

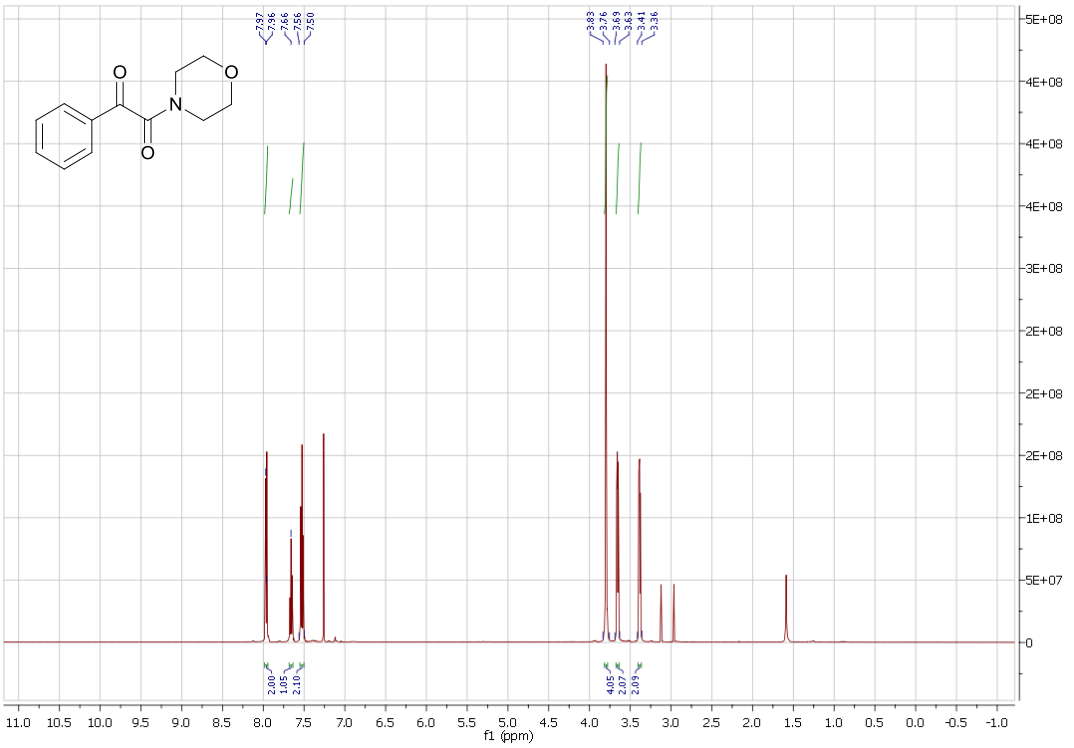
X. NMR spectra of isolated products



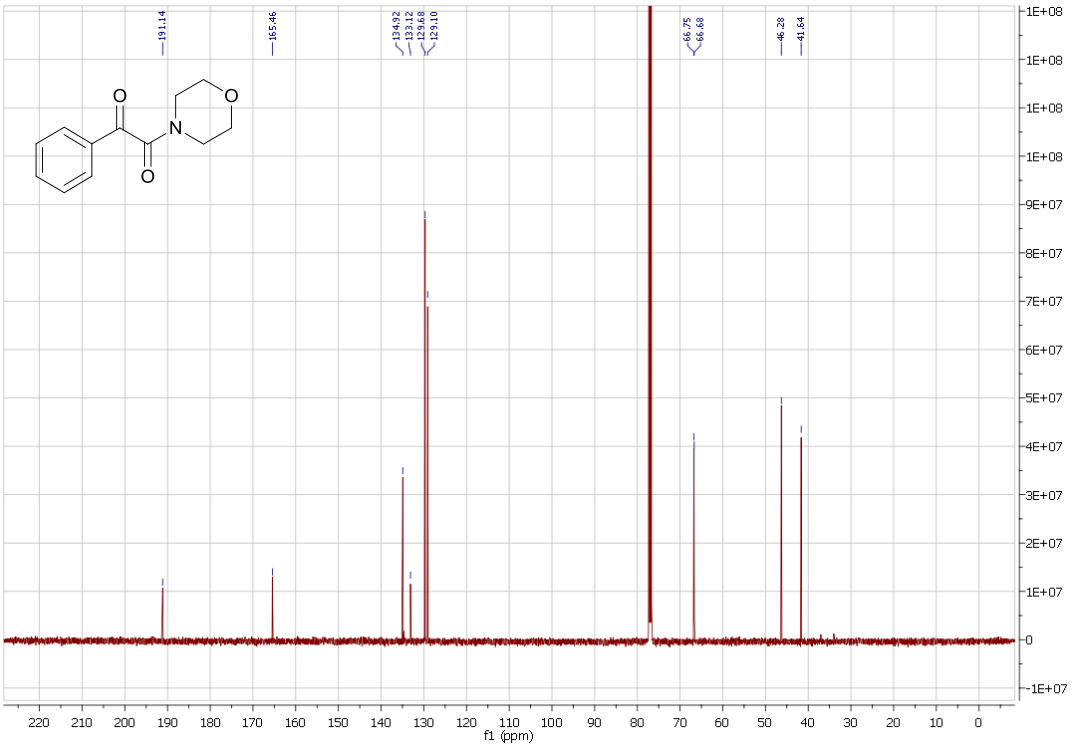
^{1}H NMR spectrum of **6a**



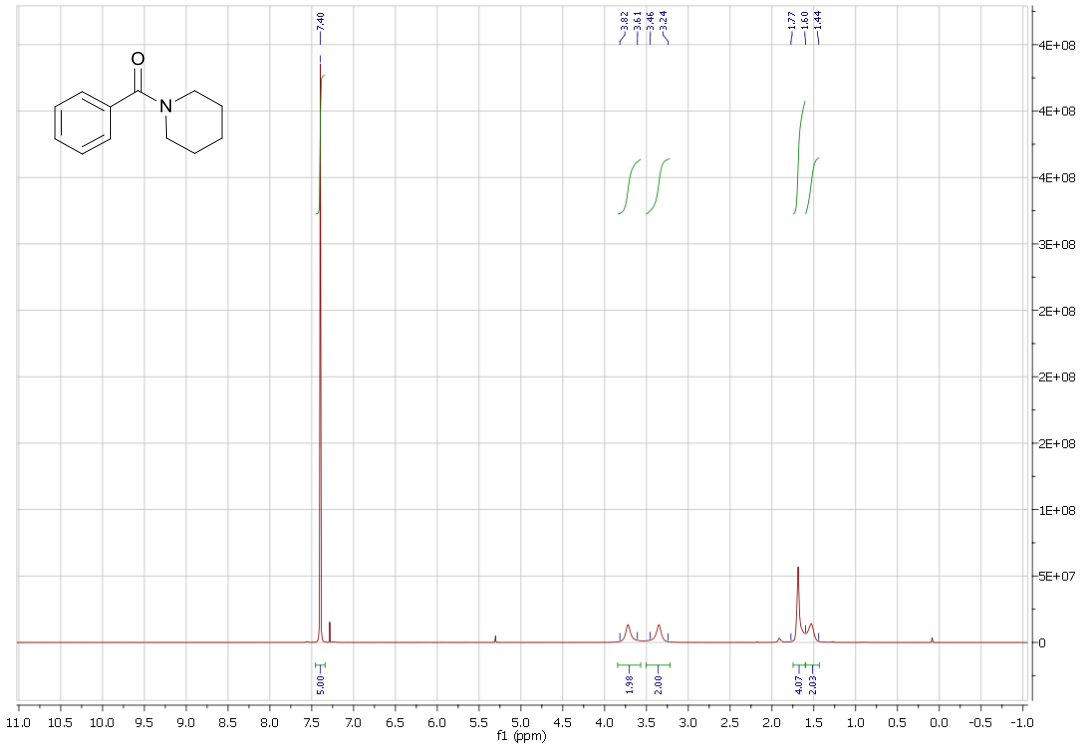
^{13}C NMR spectrum of **6a**



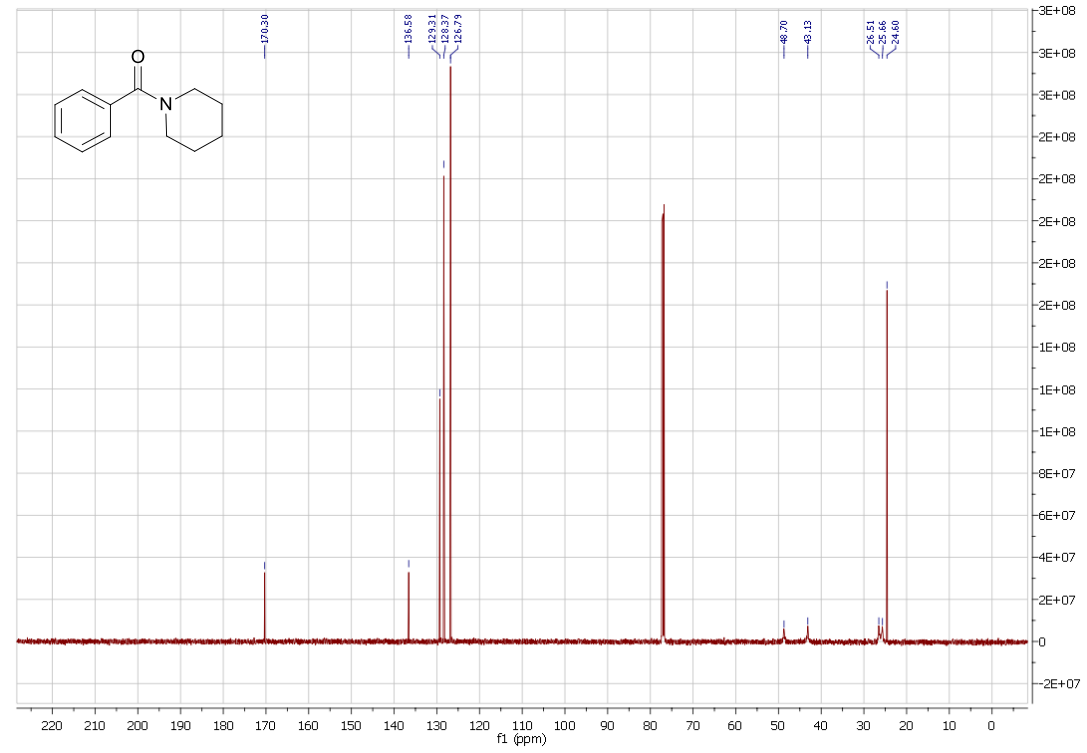
^{1}H NMR spectrum of **7a**



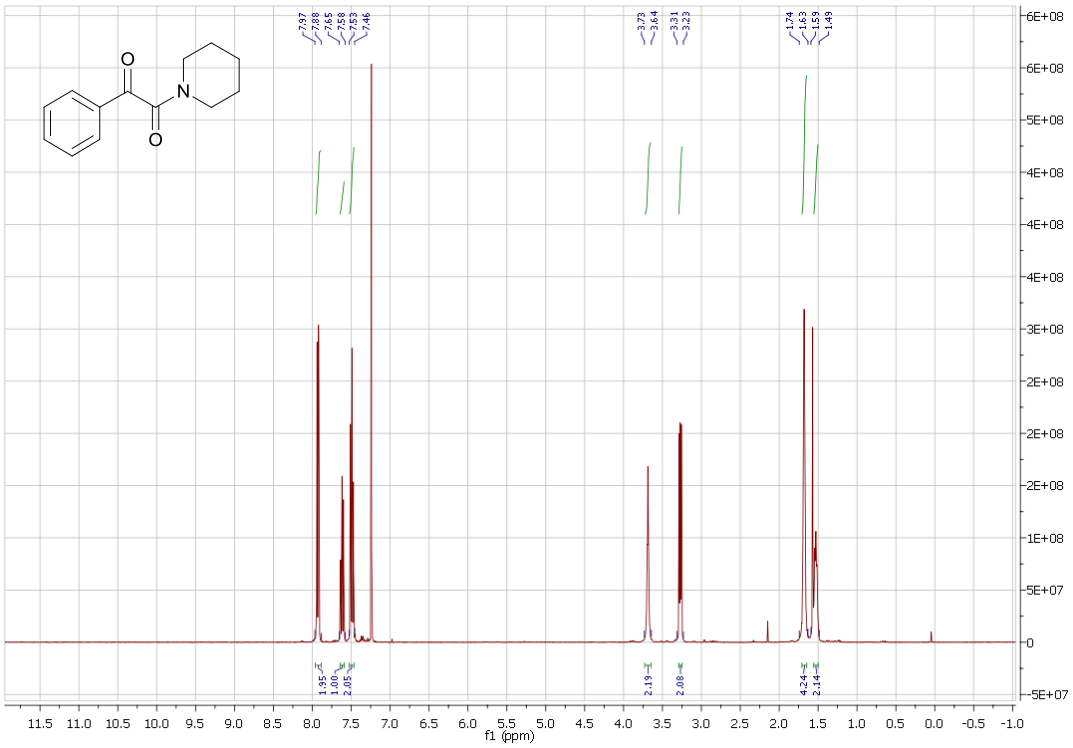
^{13}C NMR spectrum of **7a**



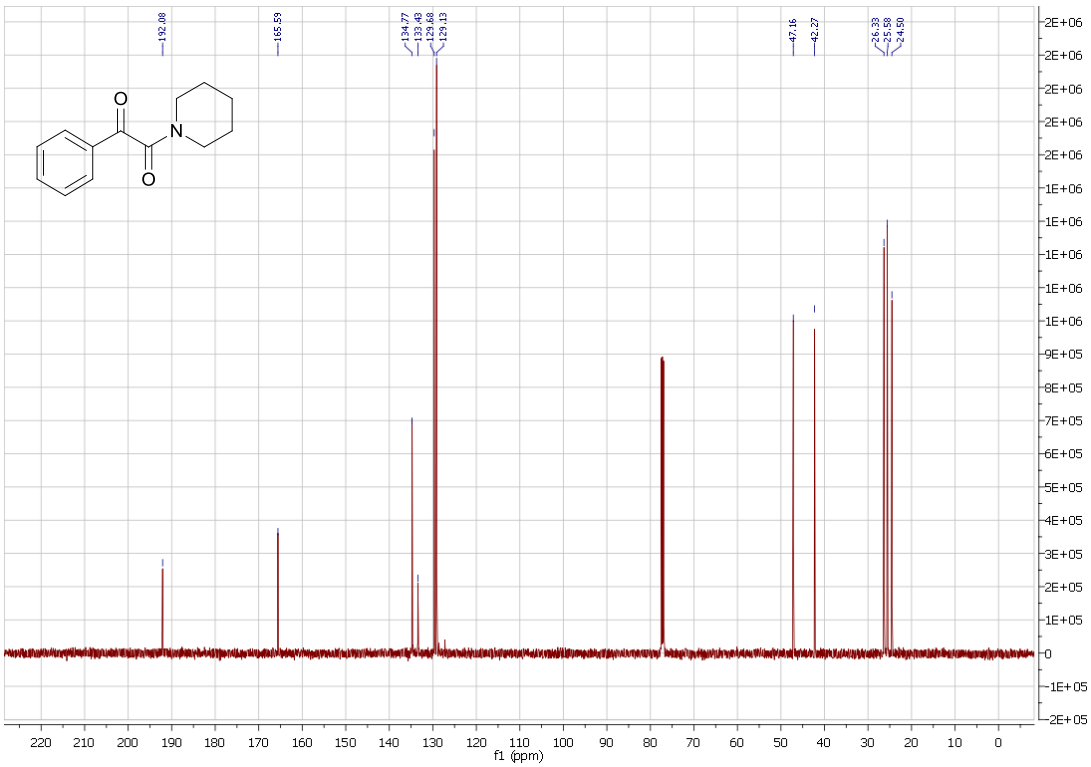
^{1}H NMR spectrum of Phenyl(piperidin-1-yl)methanone



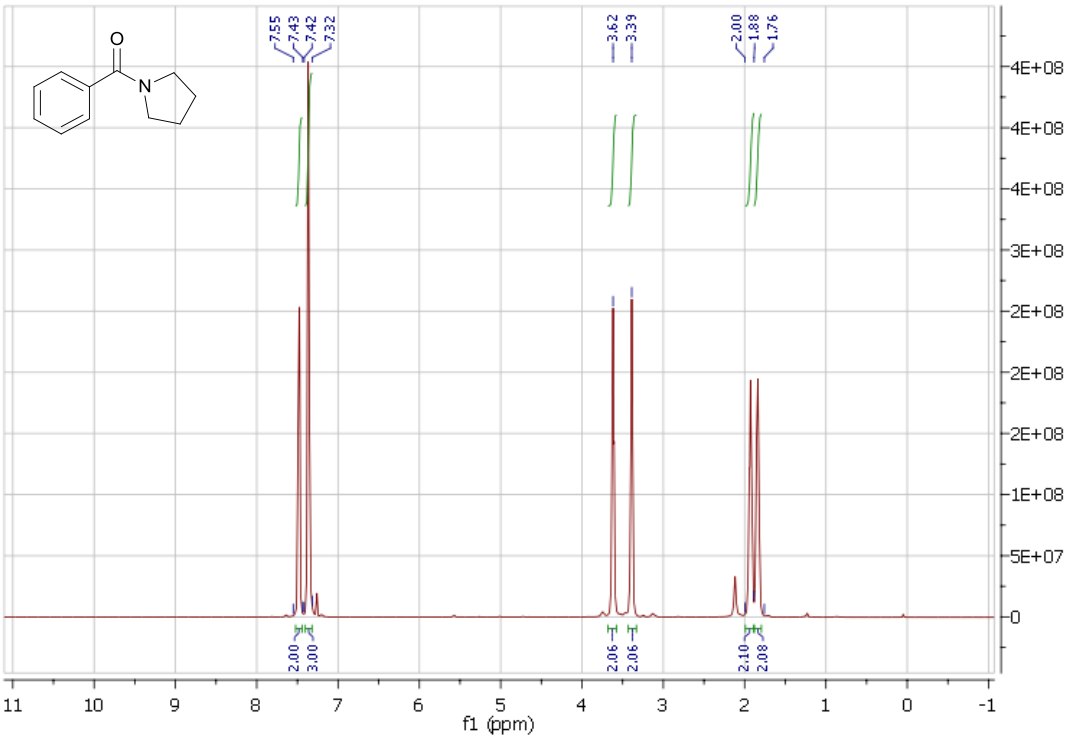
^{13}C NMR spectrum of Phenyl(piperidin-1-yl)methanone



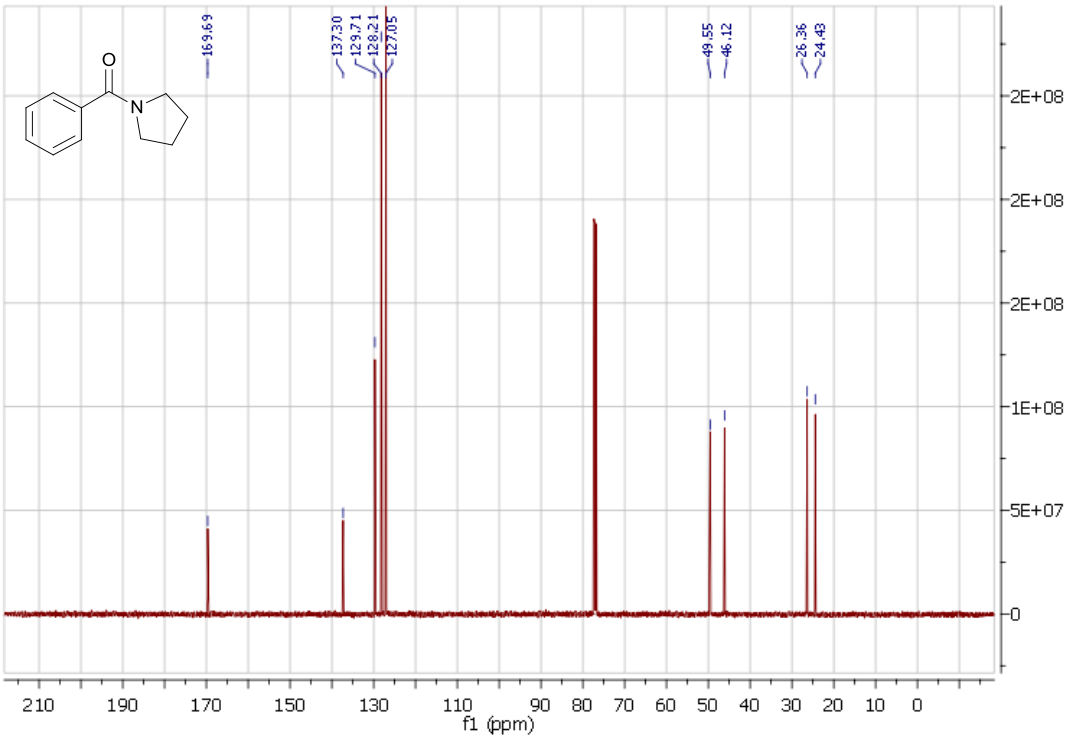
^{1}H NMR spectrum of 1-Phenyl-2-(piperidin-1-yl)ethane-1,2-dione



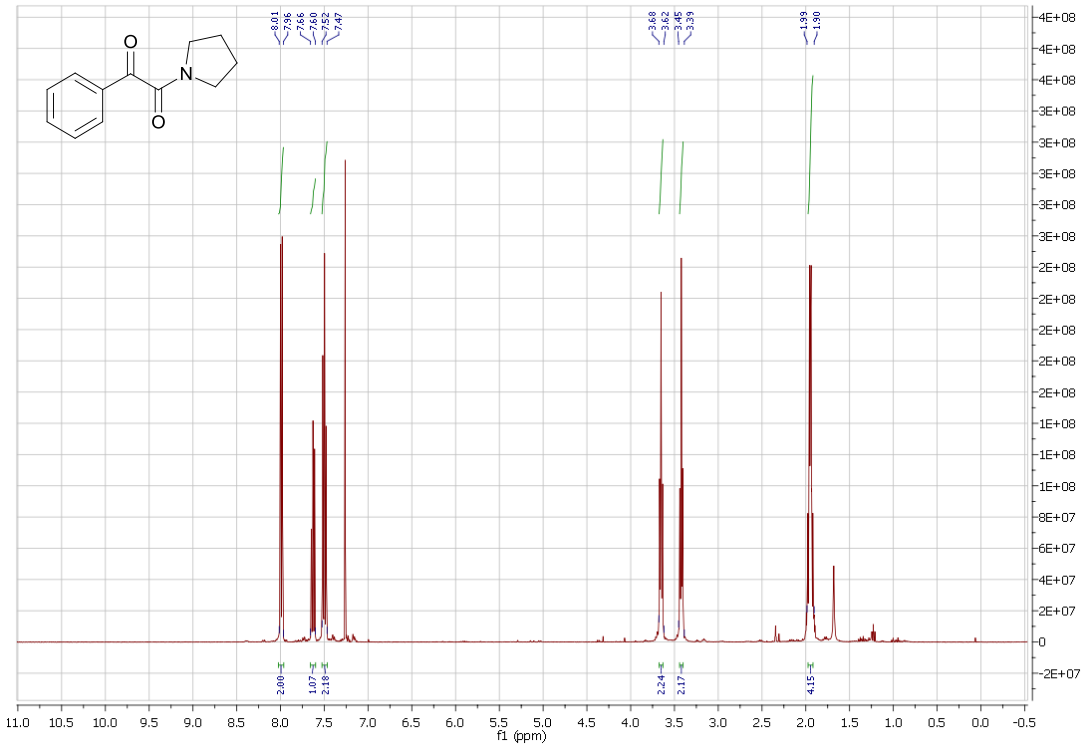
^{13}C NMR spectrum of 1-Phenyl-2-(piperidin-1-yl)ethane-1,2-dione



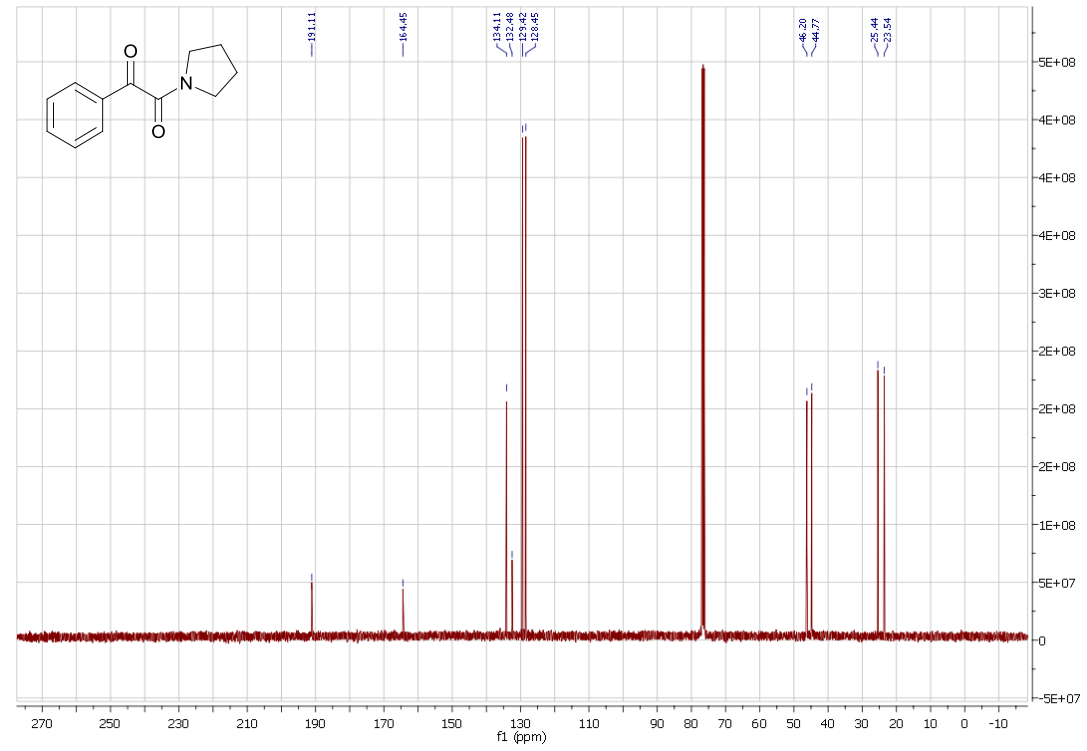
^{1}H NMR spectrum of Phenyl(pyrrolidin-1-yl)methanone



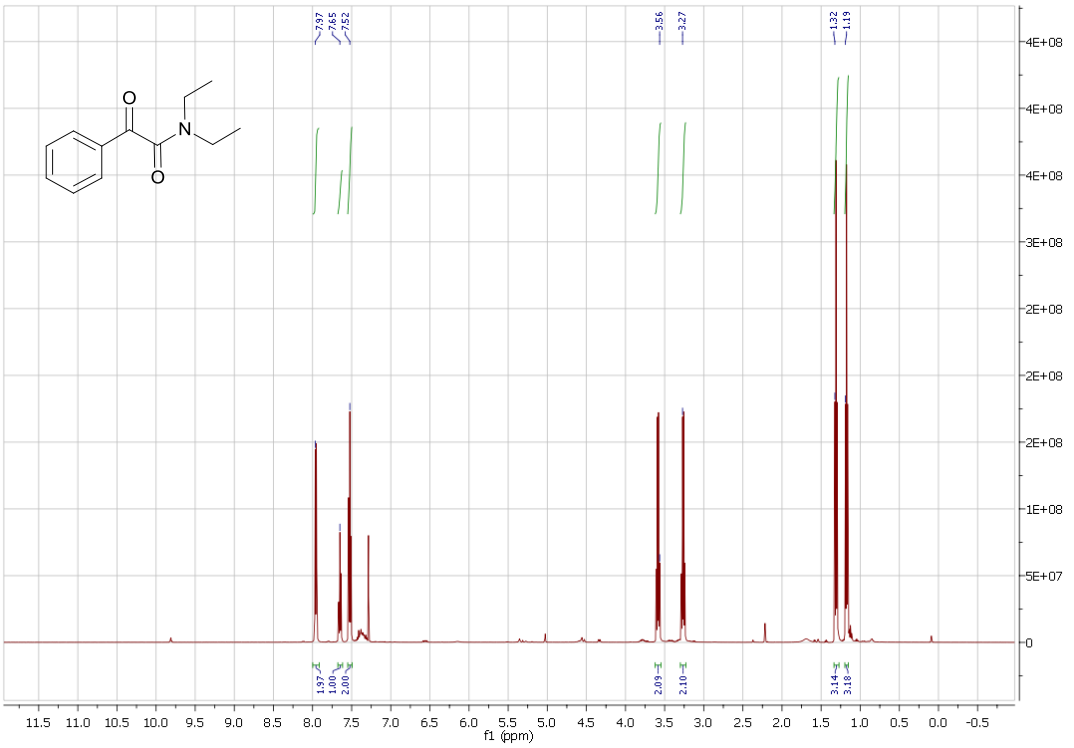
^{13}C NMR spectrum of Phenyl(pyrrolidin-1-yl)methanone



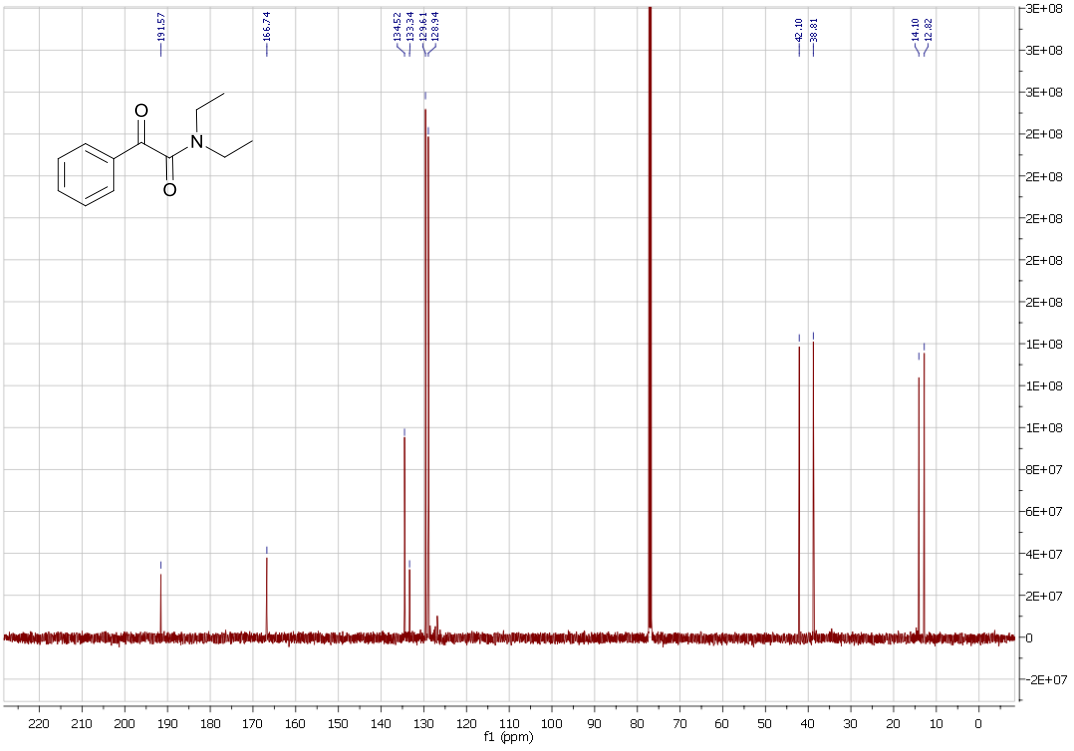
^{1}H NMR spectrum of 1-Phenyl-2-(pyrrolidin-1-yl)ethane-1,2-dione



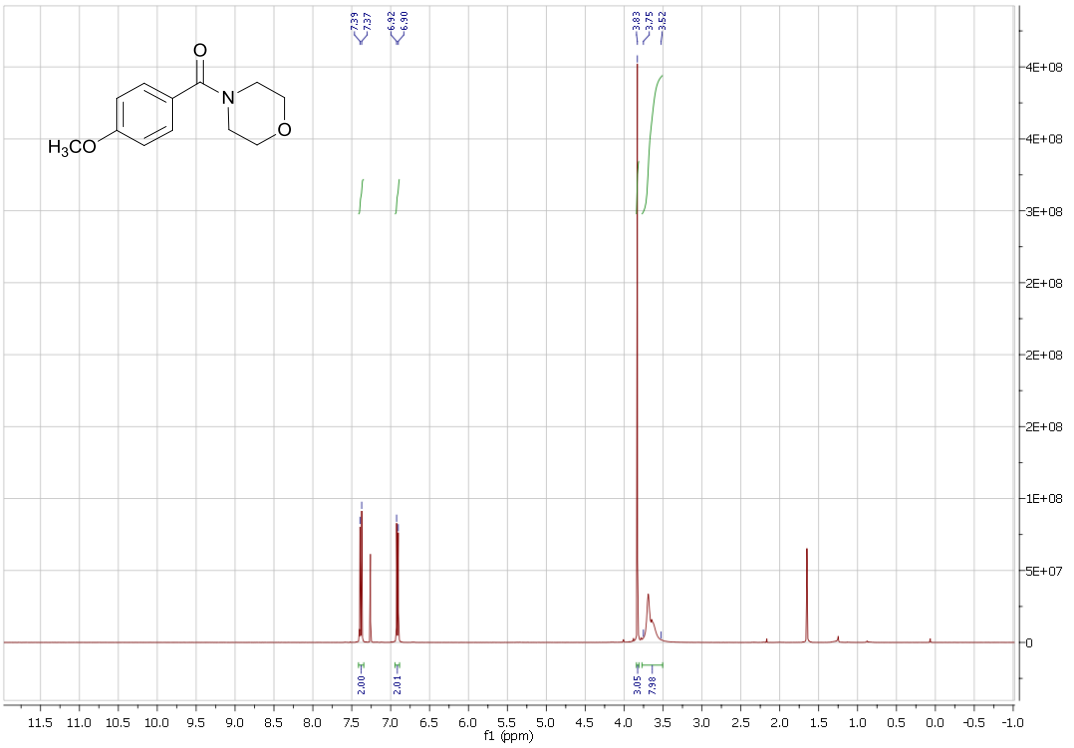
^{13}C NMR spectrum of 1-Phenyl-2-(pyrrolidin-1-yl)ethane-1,2-dione



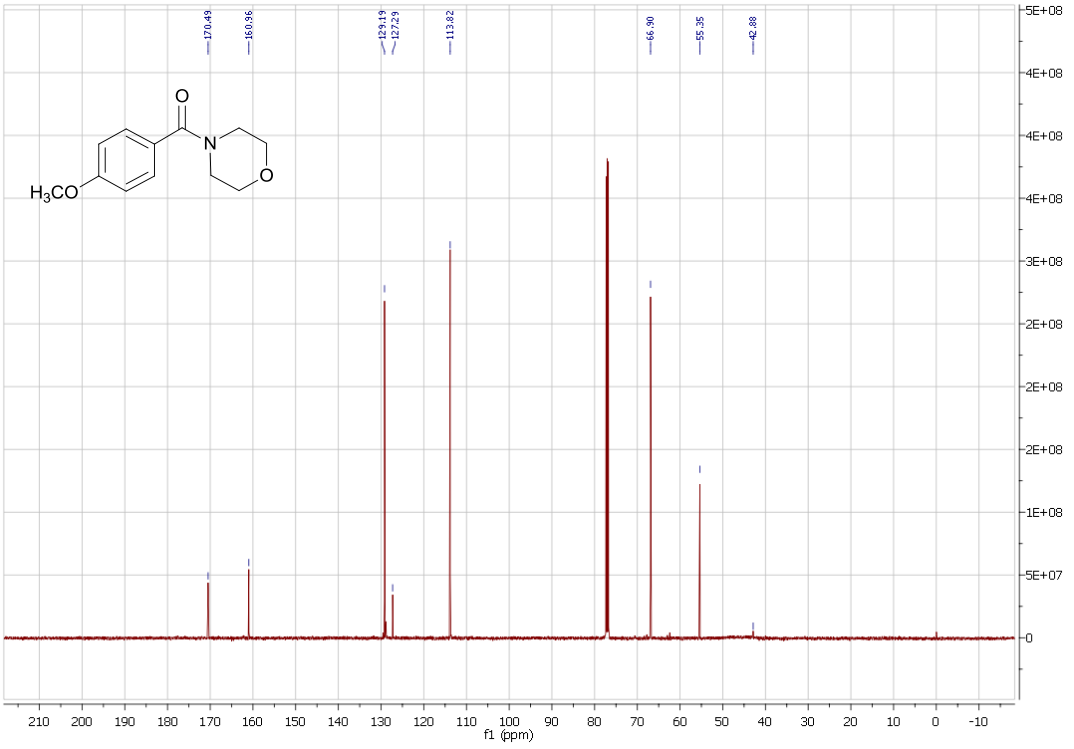
^{1}H NMR spectrum of N,N-Diethyl-2-oxo-2-phenylacetamide



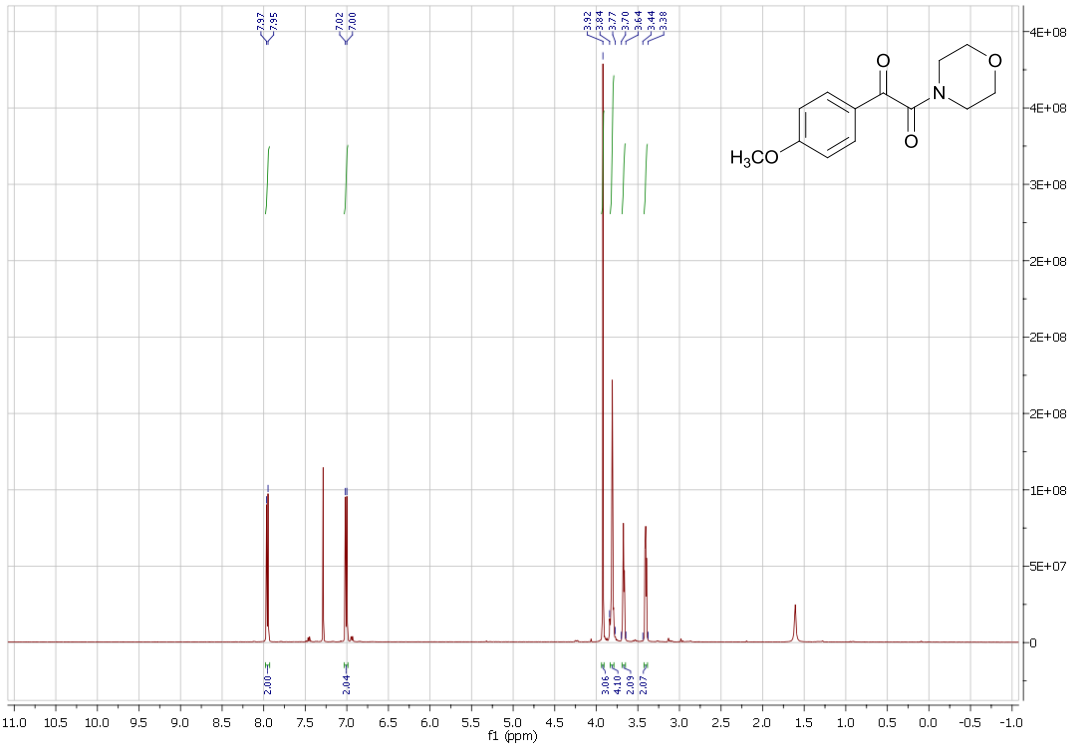
^{13}C NMR spectrum of N,N-Diethyl-2-oxo-2-phenylacetamide



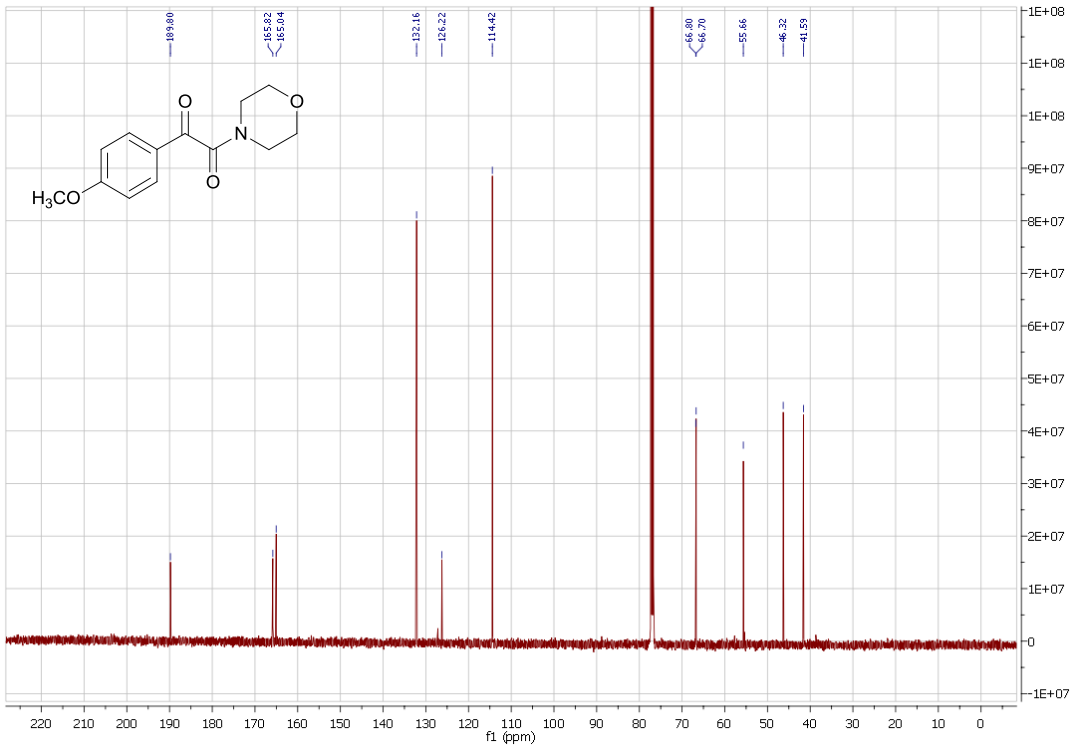
^{1}H NMR spectrum of morpholino(4-methoxyphenyl)methanone



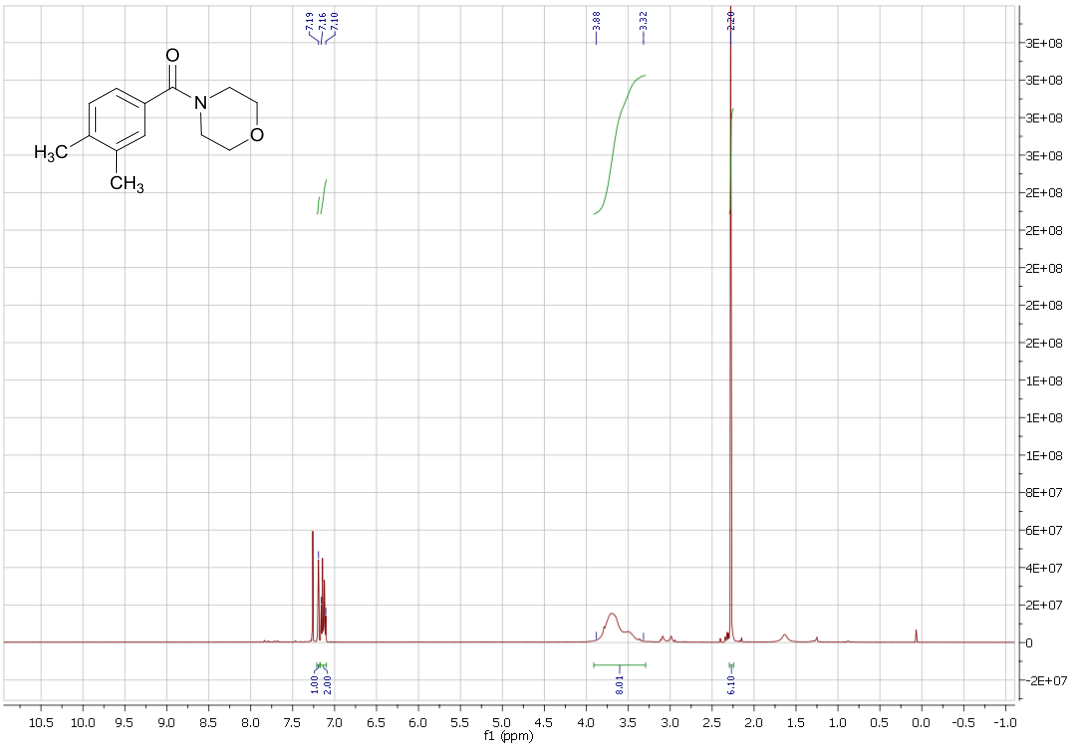
^{13}C NMR spectrum of morpholino(4-methoxyphenyl)methanone



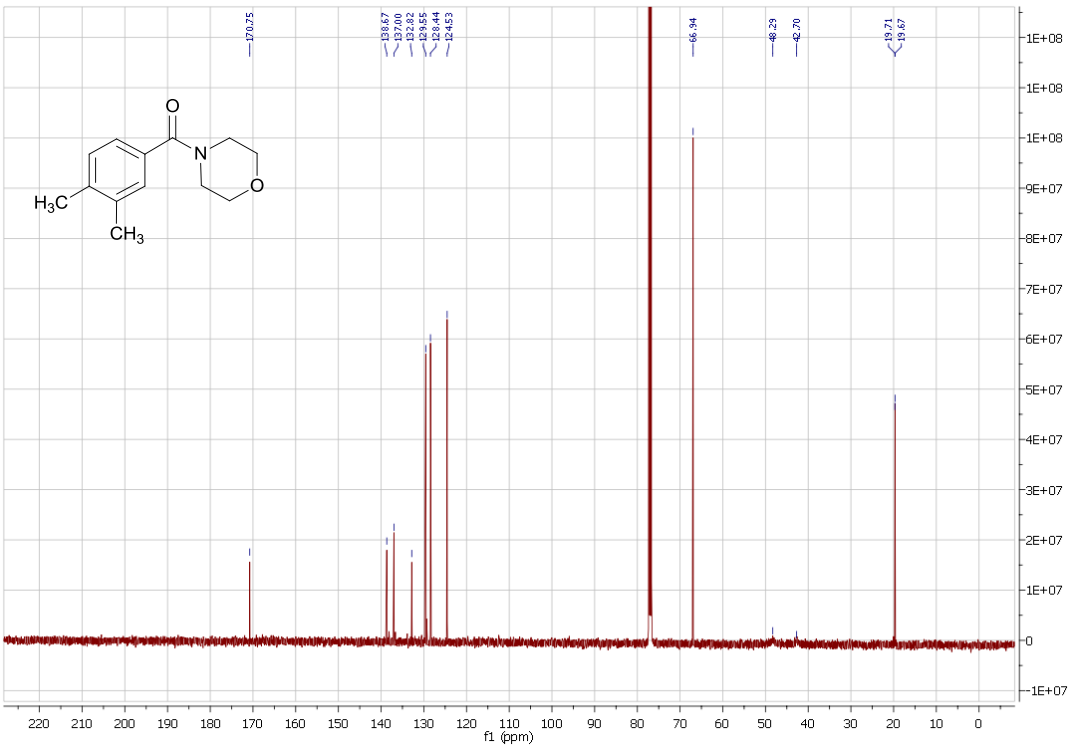
^{1}H NMR spectrum of 1-morpholino-2-(4-methoxyphenyl)ethane-1,2-dione



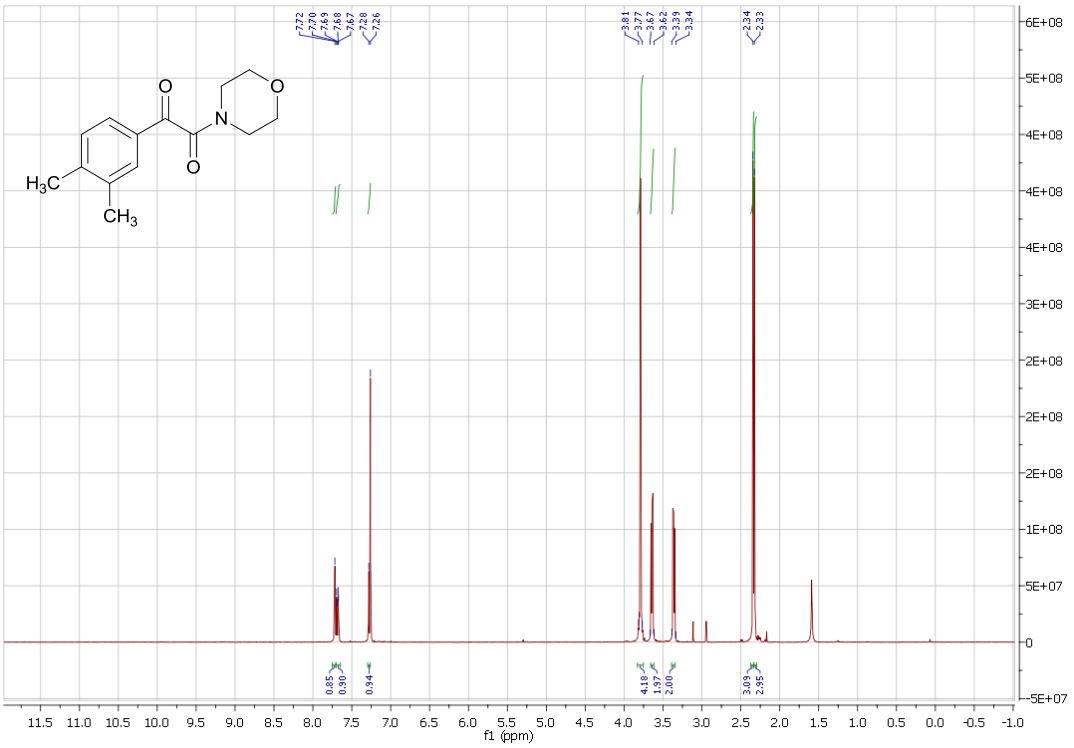
^{13}C NMR spectrum of 1-morpholino-2-(4-methoxyphenyl)ethane-1,2-dione



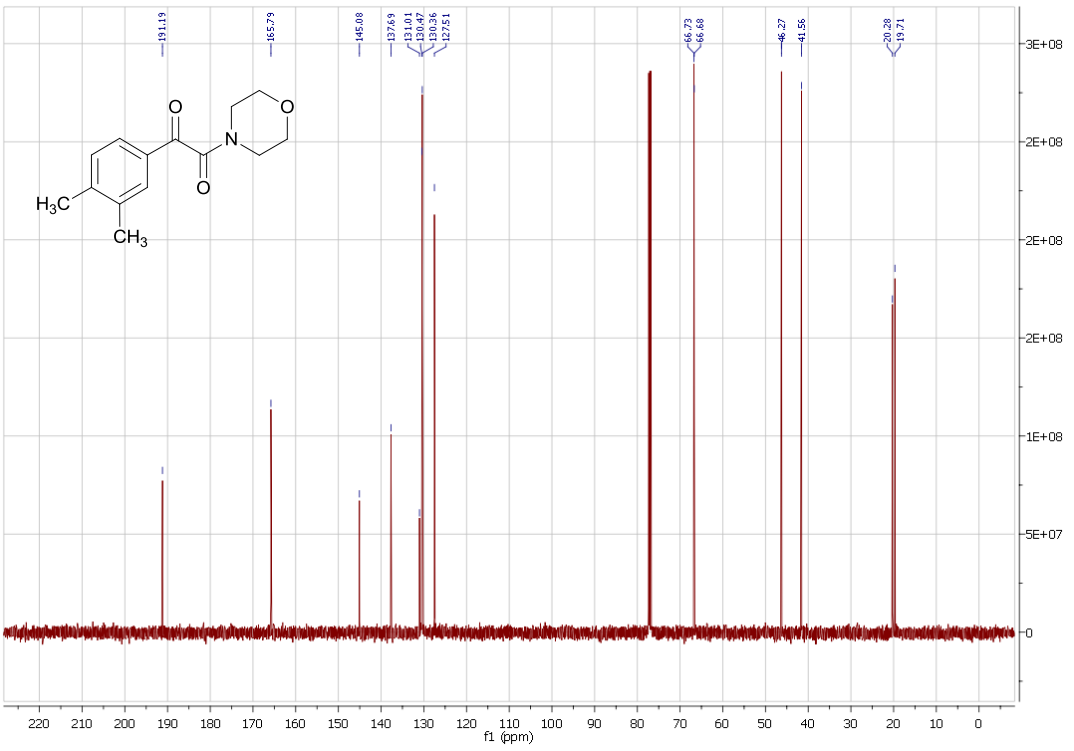
^{1}H NMR spectrum of morpholino(3,4-dimethylphenyl)methanone



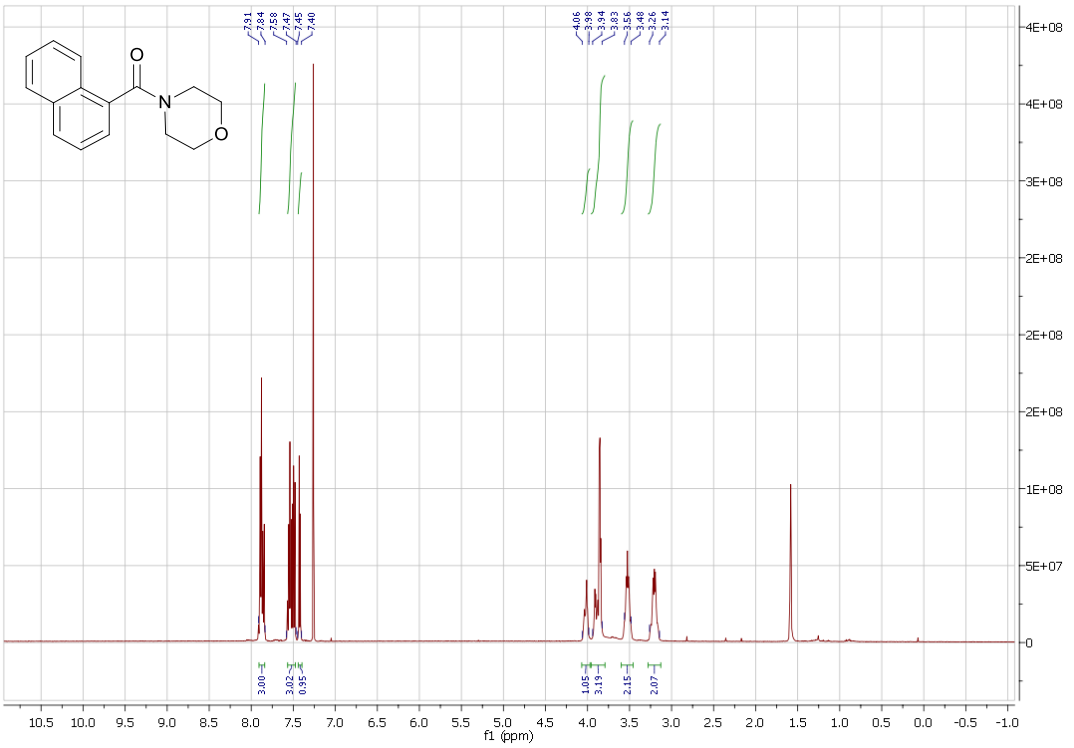
^{13}C NMR spectrum of morpholino(3,4-dimethylphenyl)methanone



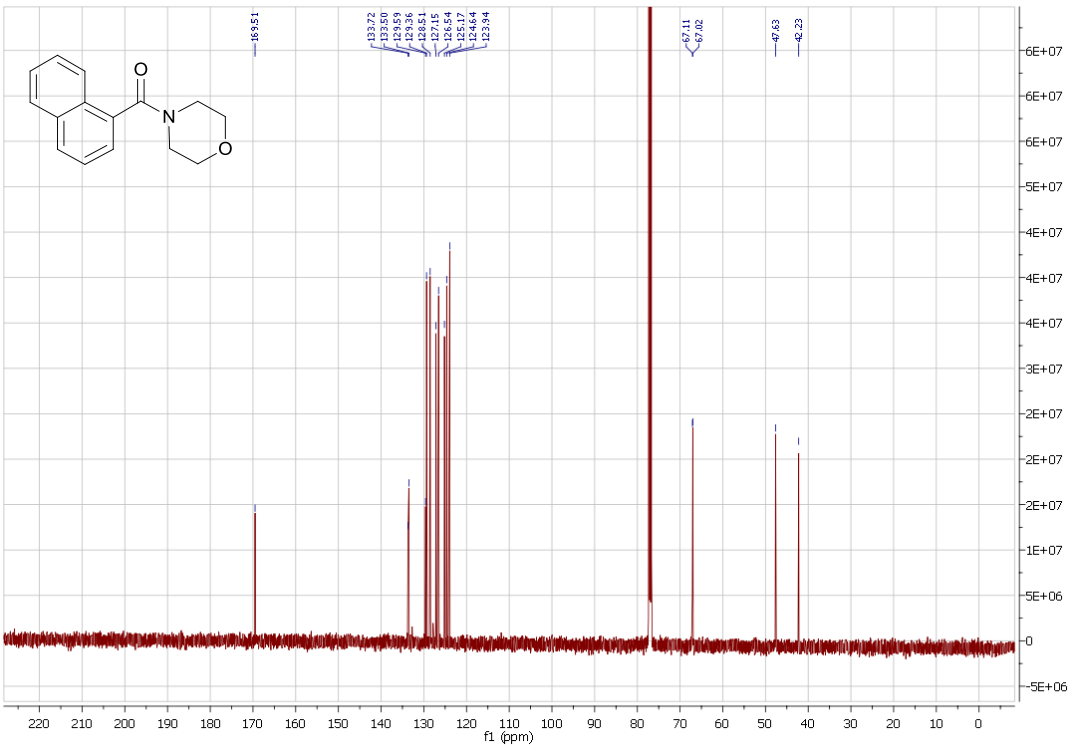
^{1}H NMR spectrum of 1-(3,4-dimethylphenyl)-2-morpholinoethane-1,2-dione



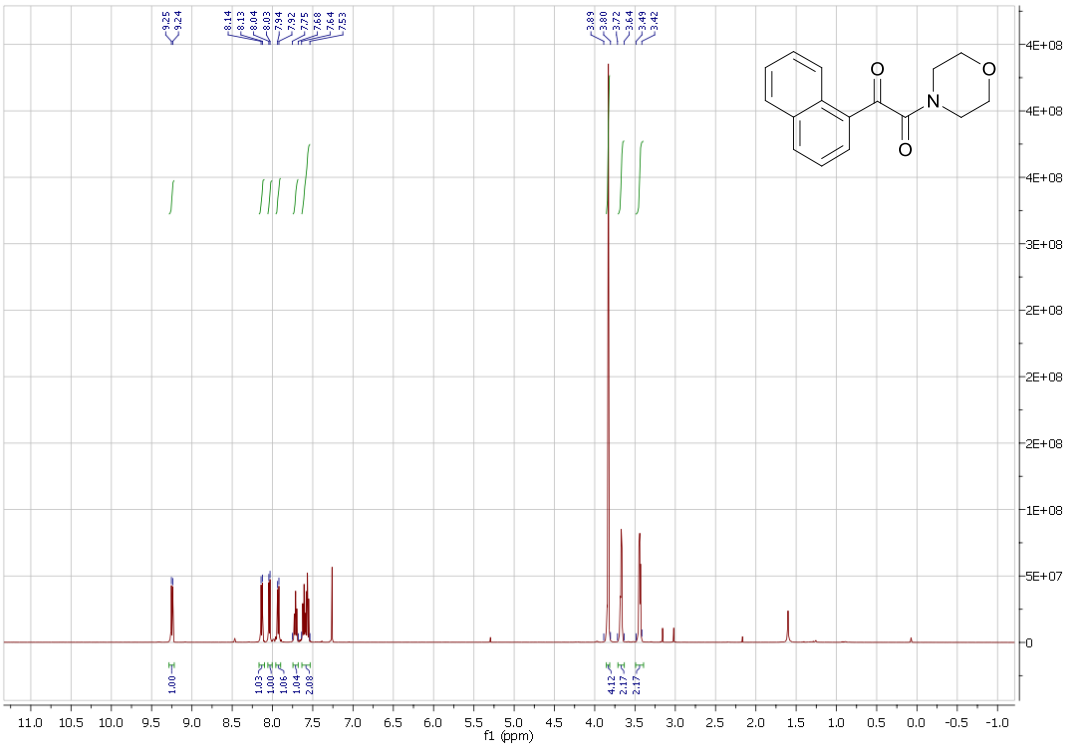
^{13}C NMR spectrum of 1-(3,4-dimethylphenyl)-2-morpholinoethane-1,2-dione



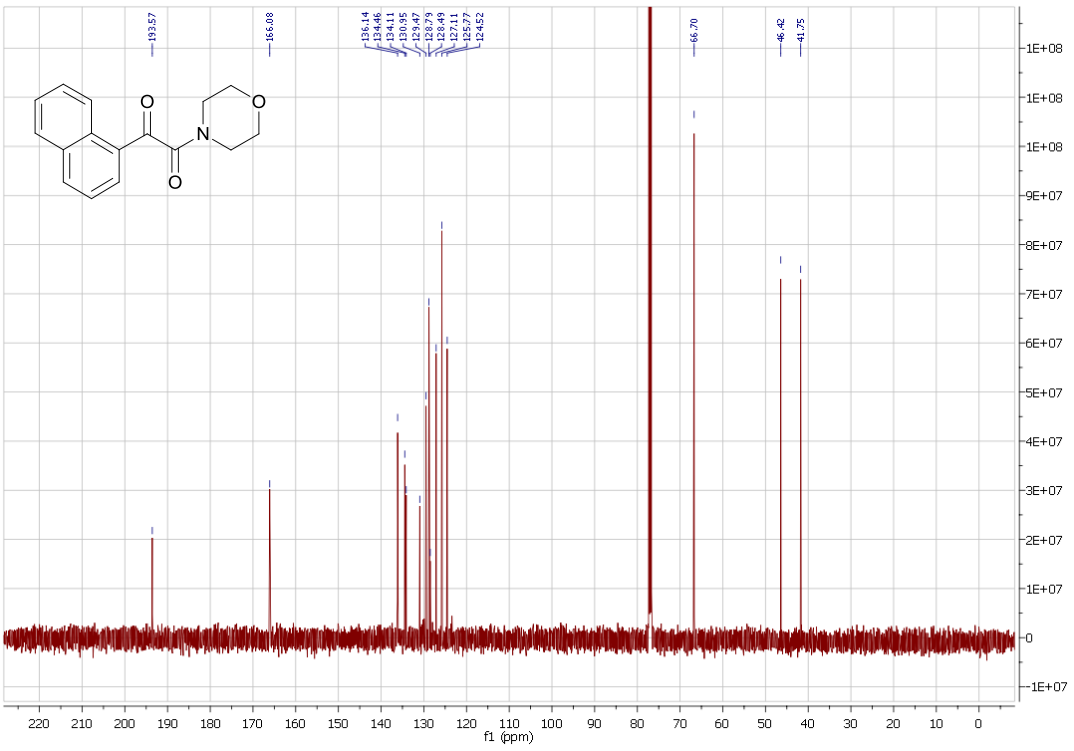
^{1}H NMR spectrum of morpholino(naphth-1-yl)methanone



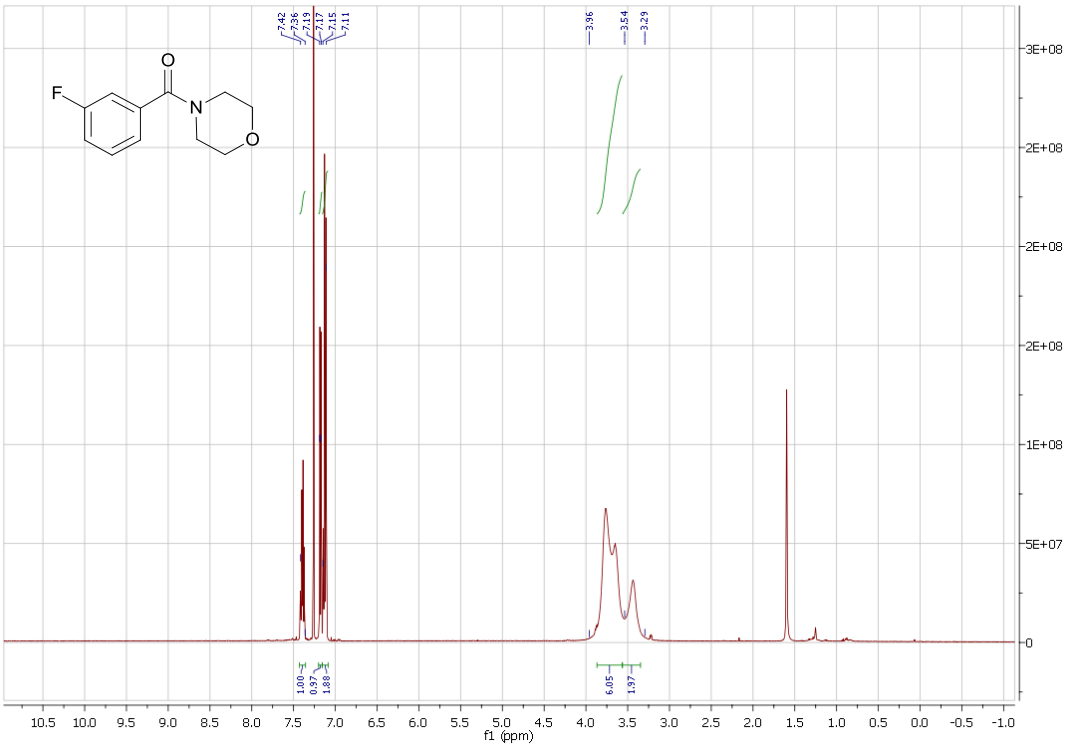
^{13}C NMR spectrum of morpholino(naphth-1-yl)methanone



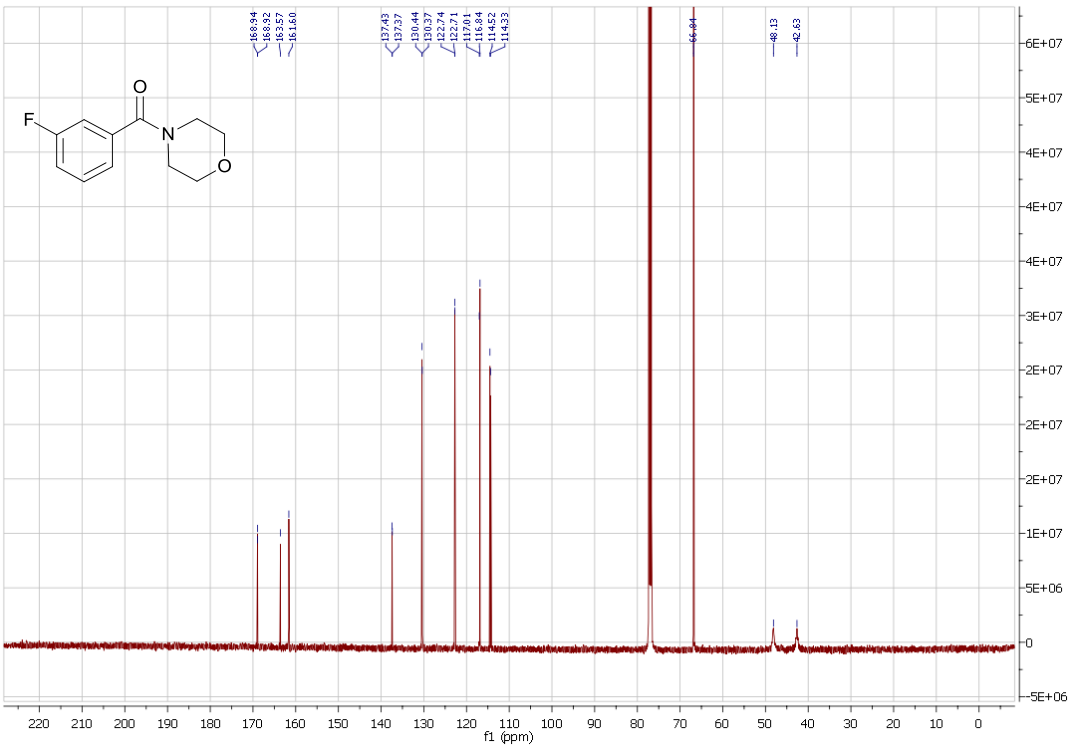
^{1}H NMR spectrum of 1-(naphth-1-yl)-2-morpholinoethane-1,2-dione



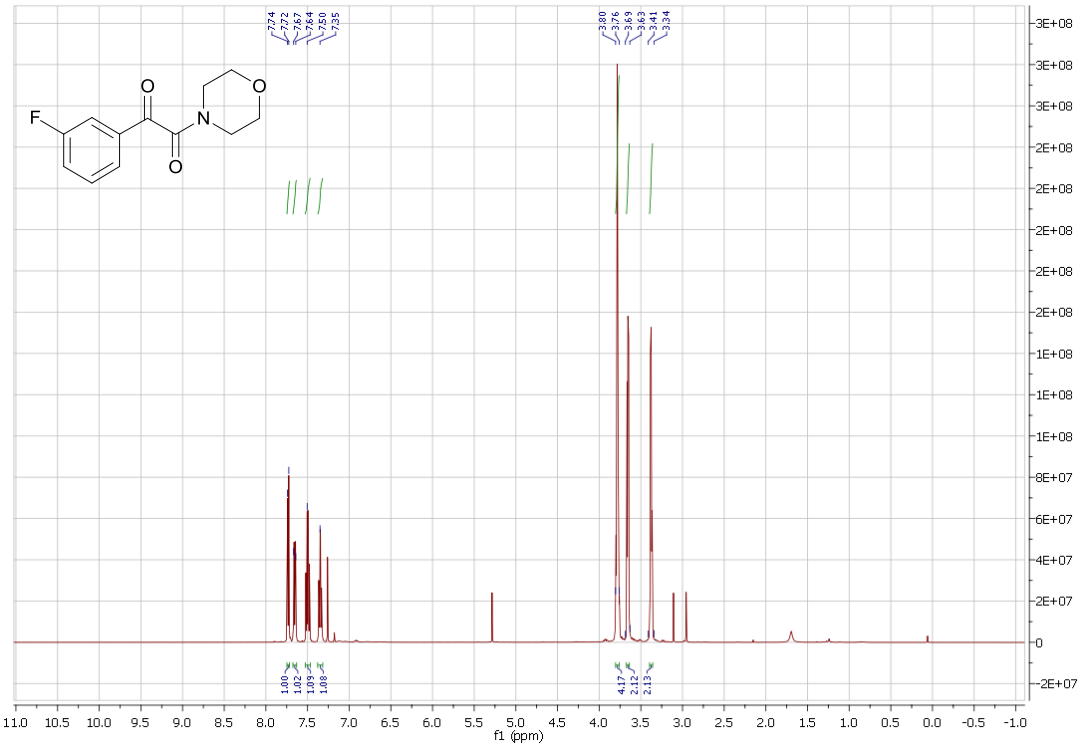
^{13}C NMR spectrum of 1-(naphth-1-yl)-2-morpholinoethane-1,2-dione



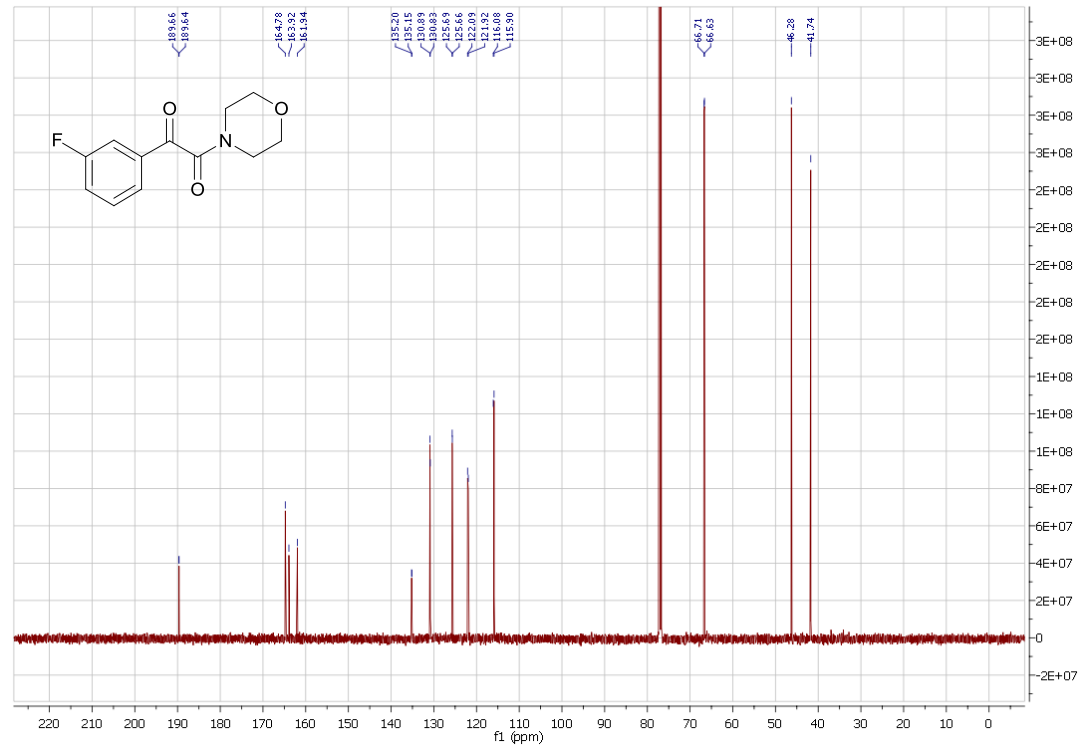
^{1}H NMR spectrum of morpholino(3-fluorophenyl)methanone



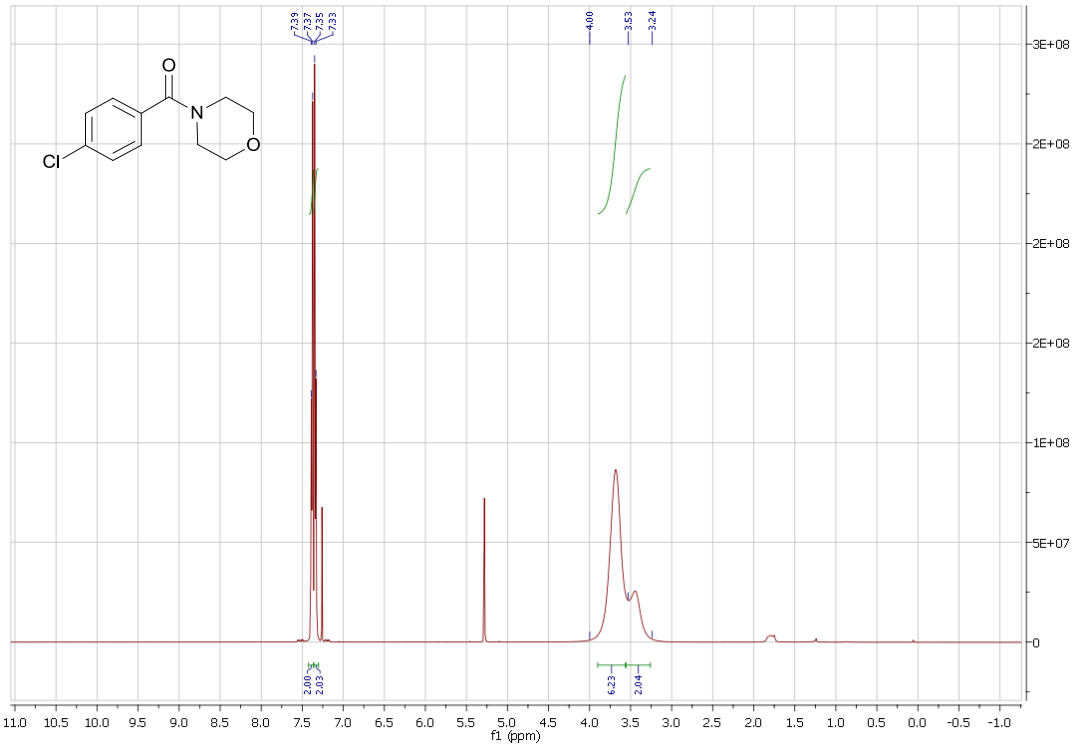
^{13}C NMR spectrum of morpholino(3-fluorophenyl)methanone



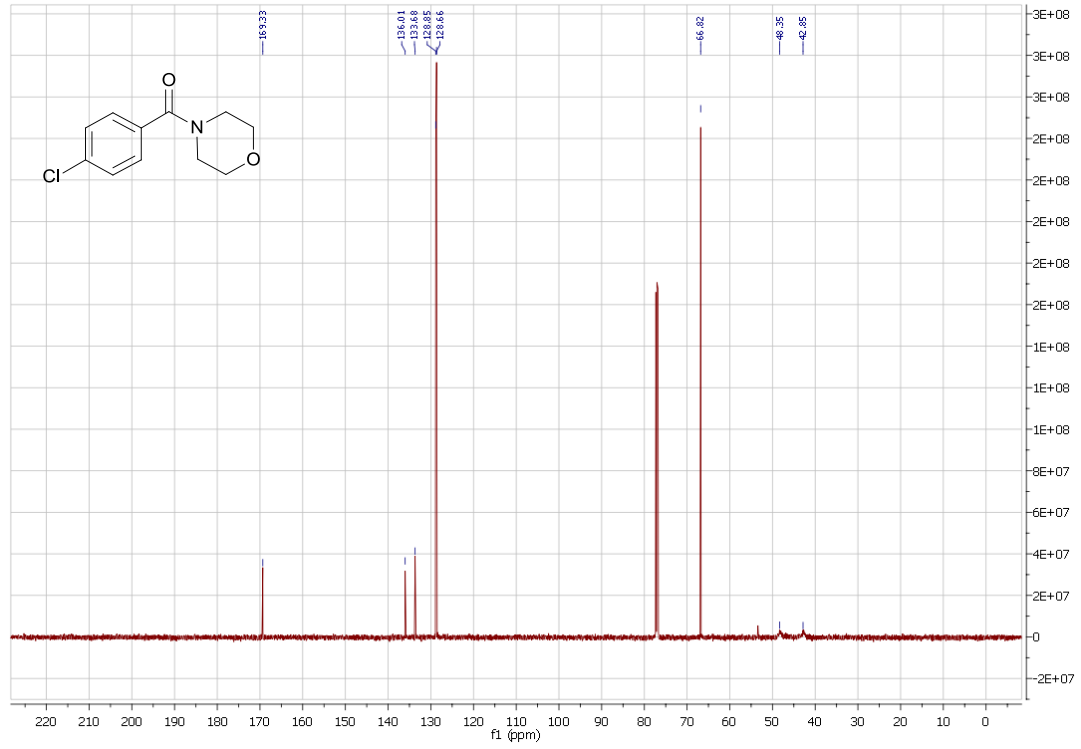
^{1}H NMR spectrum of 1-(3-fluorophenyl)-2-morpholinoethane-1,2-dione



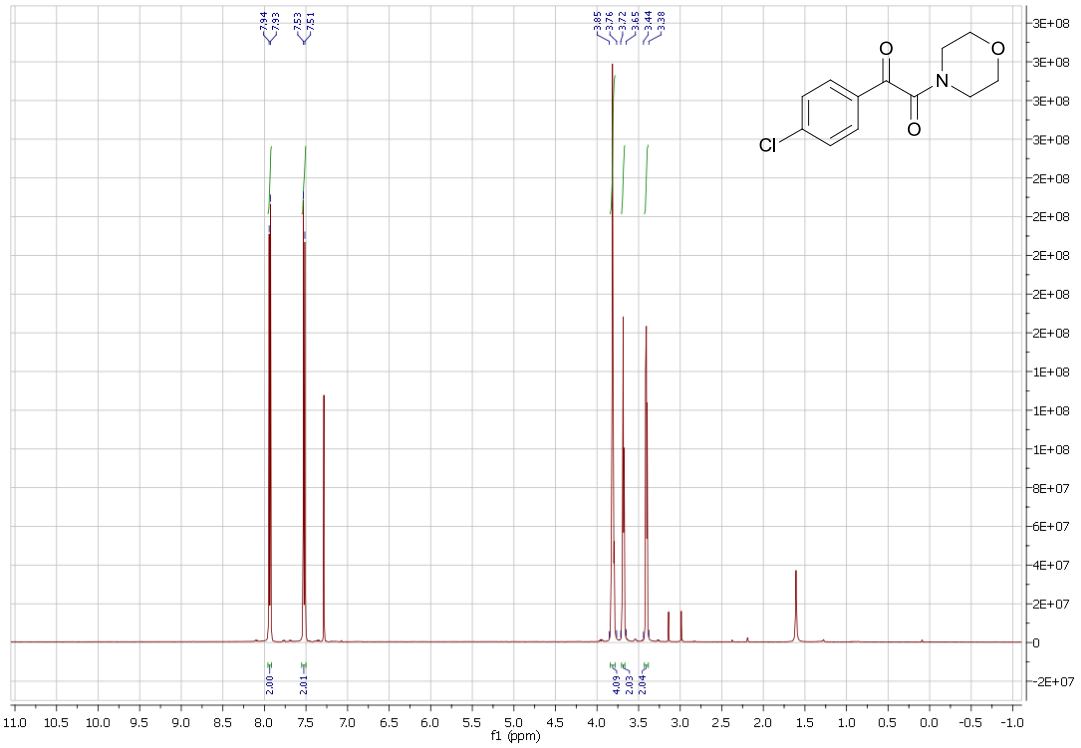
^{13}C NMR spectrum of 1-(3-fluorophenyl)-2-morpholinoethane-1,2-dione



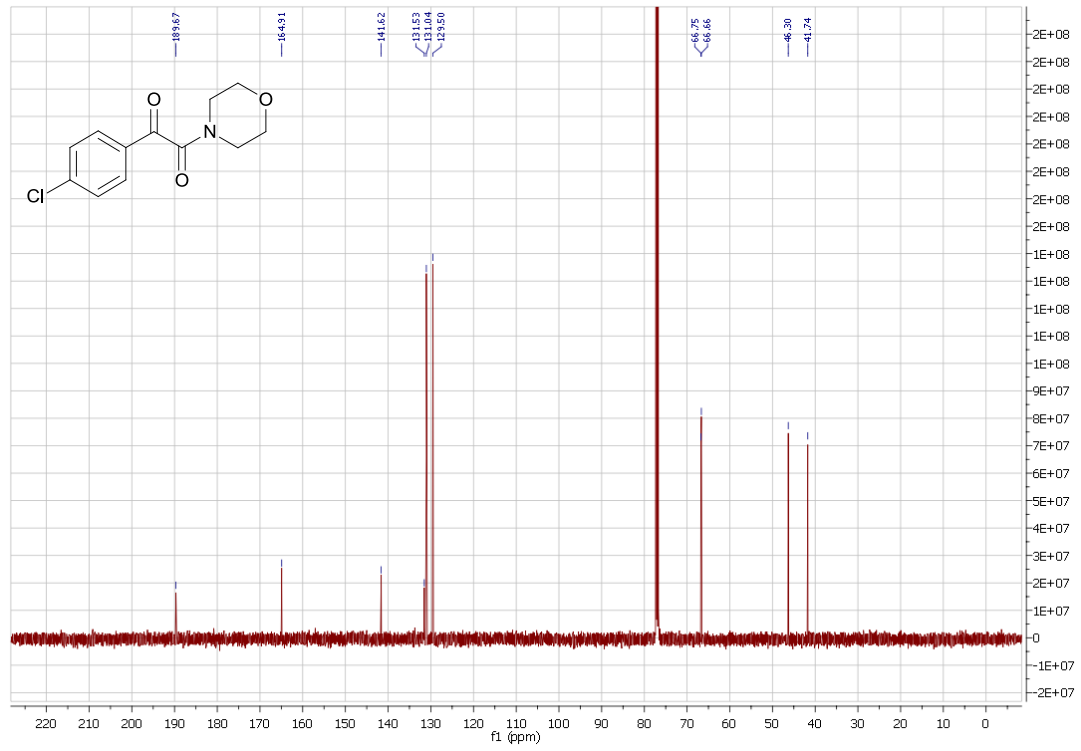
^{1}H NMR spectrum of morpholino(4-chlorophenyl)methanone



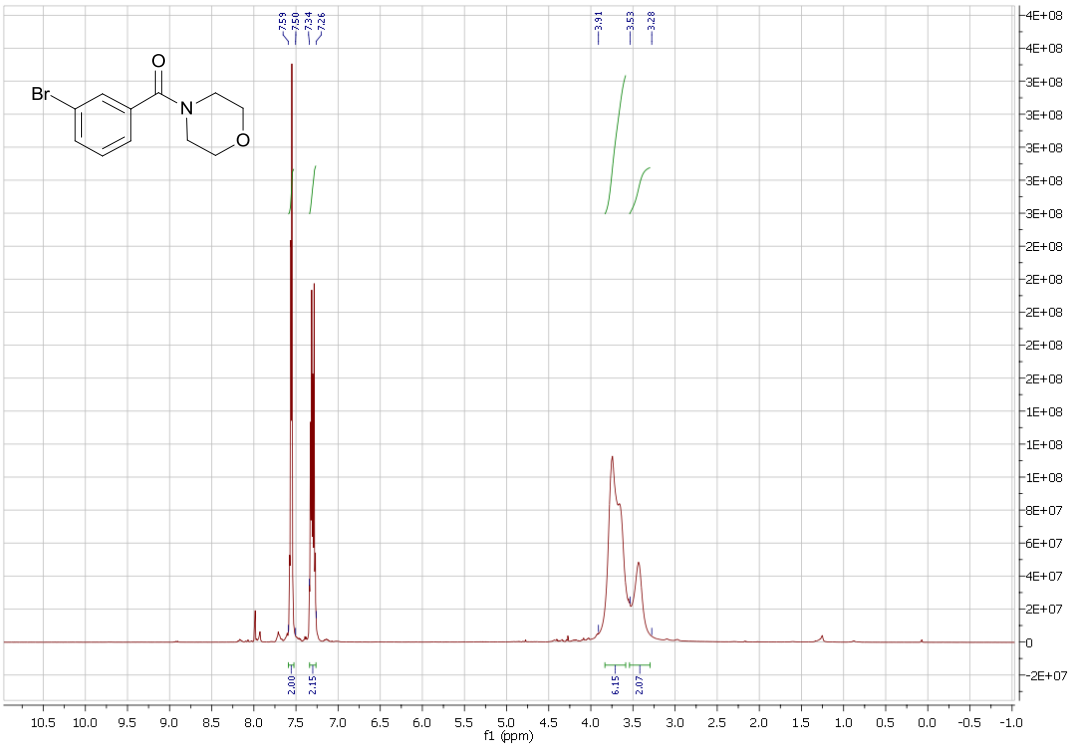
^{13}C NMR spectrum of morpholino(4-chlorophenyl)methanone



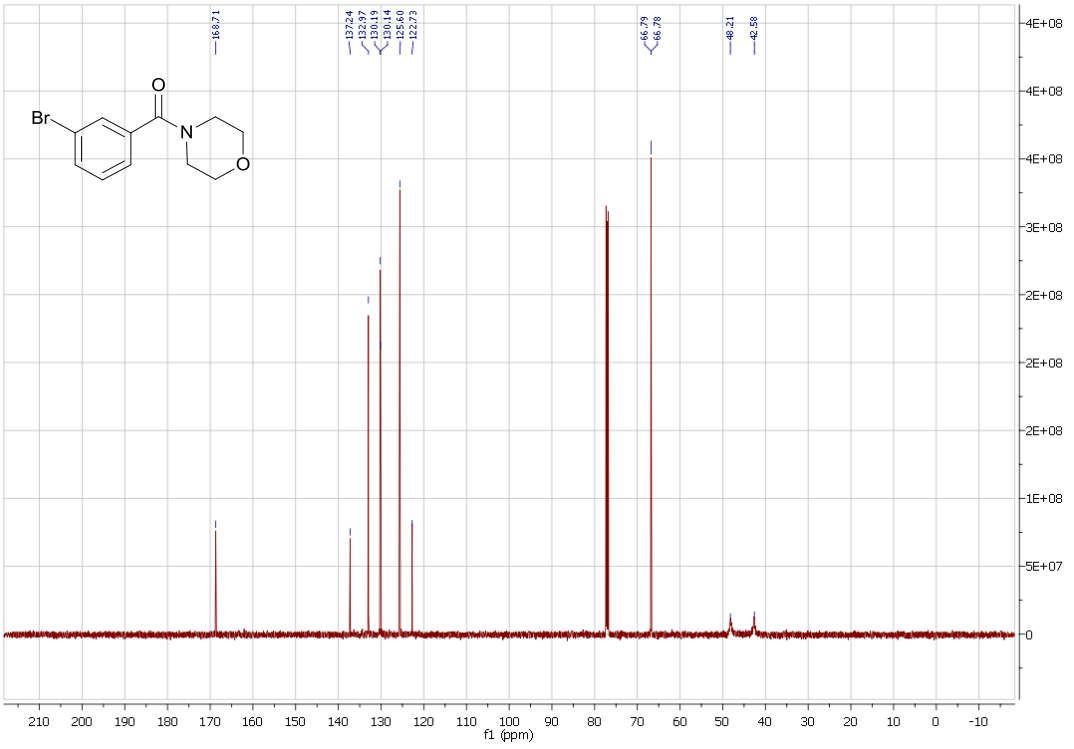
^{1}H NMR spectrum of 1-(4-Chlorophenyl)-2-morpholinoethane-1,2-dione



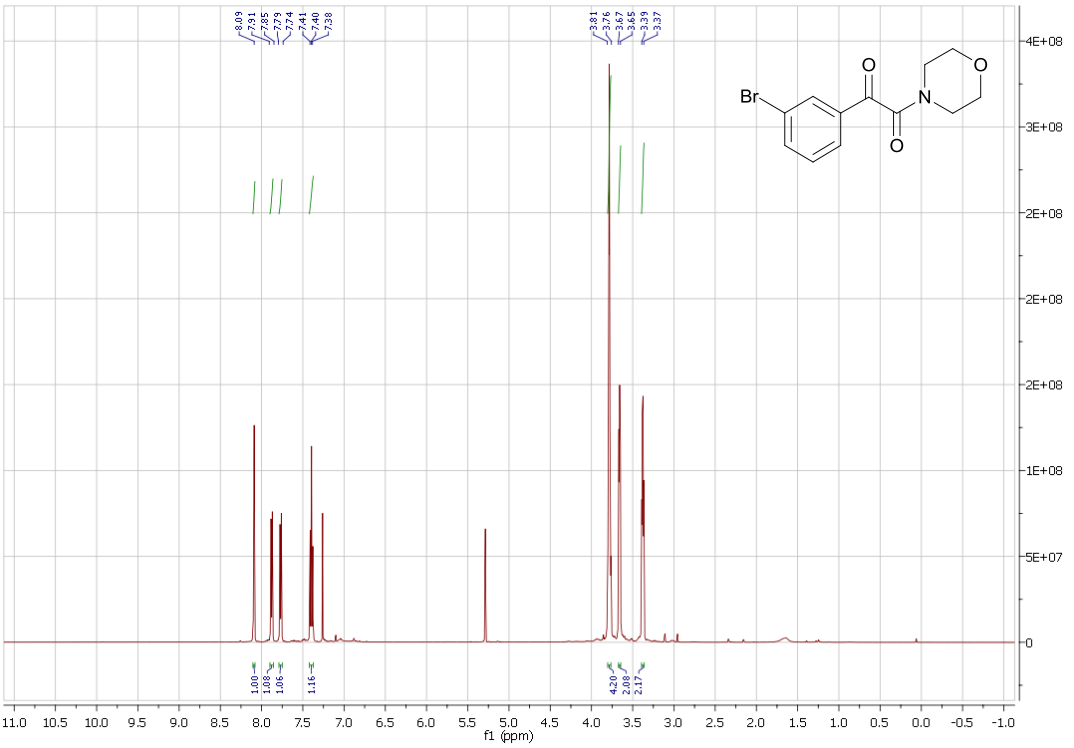
^{13}C NMR spectrum of 1-(4-Chlorophenyl)-2-morpholinoethane-1,2-dione



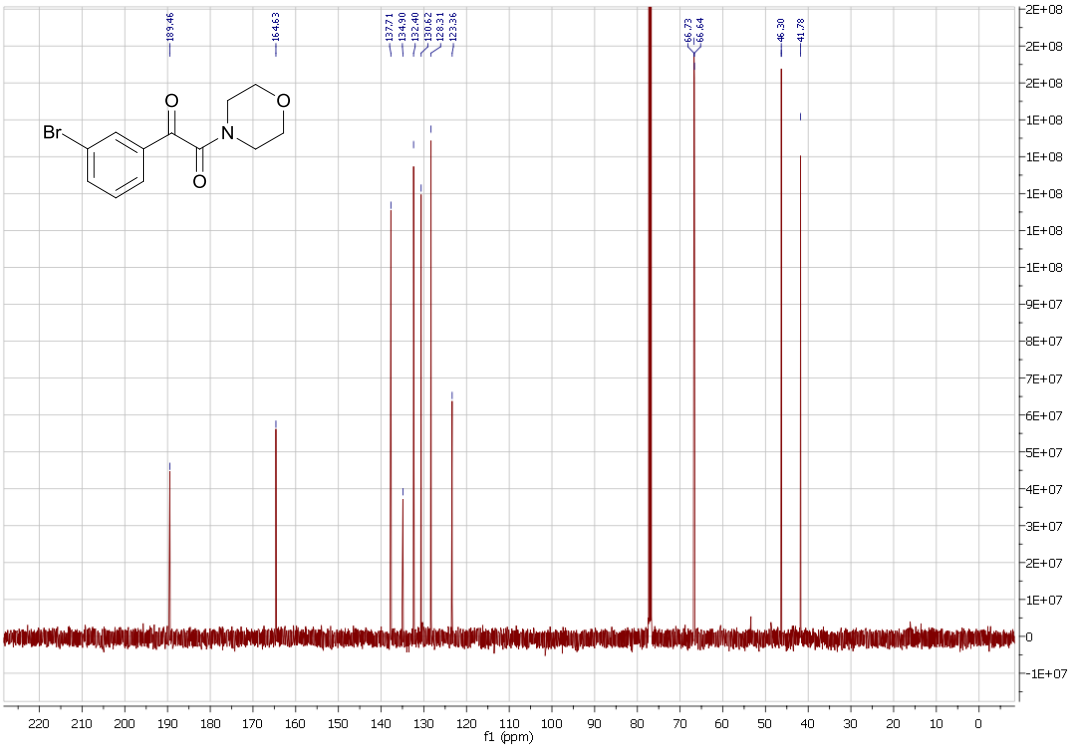
^{1}H NMR spectrum of morpholino(3-bromophenyl)methanone



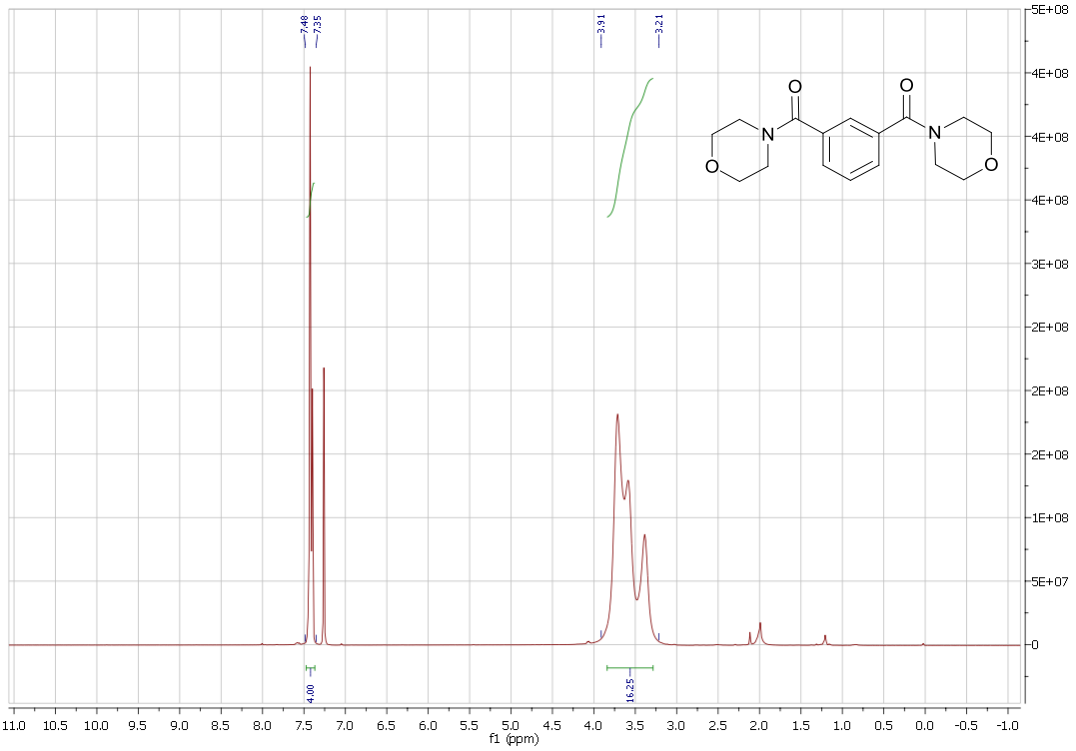
^{13}C NMR spectrum of morpholino(3-bromophenyl)methanone



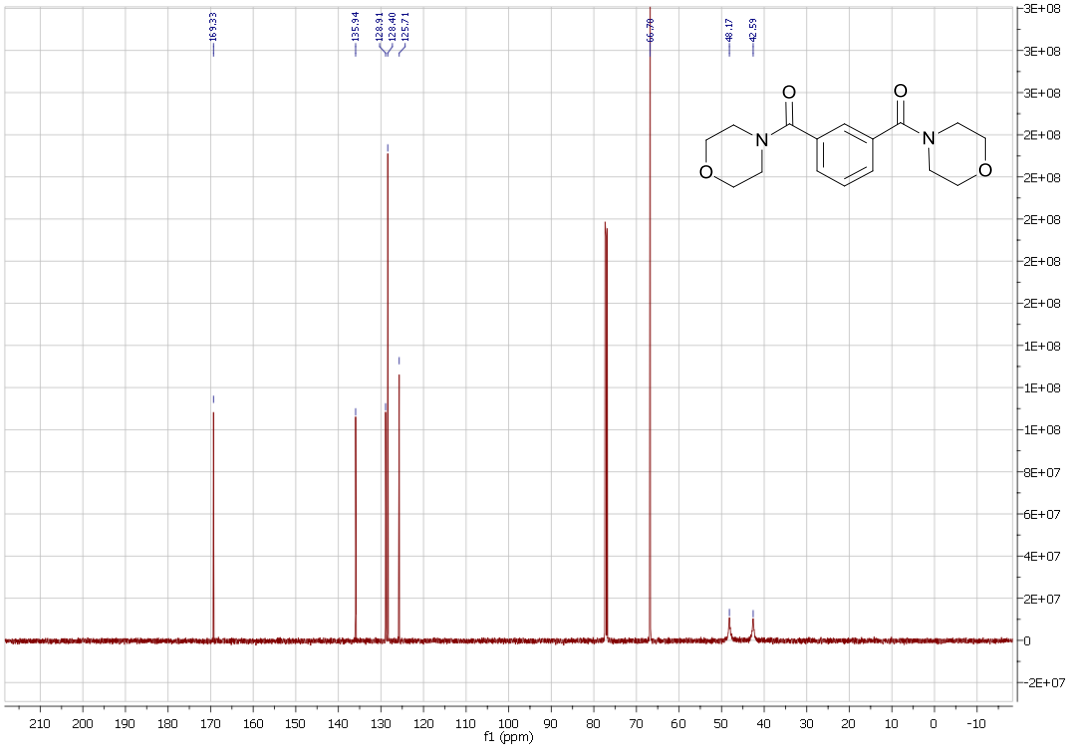
^{1}H NMR spectrum of 1-(3-bromophenyl)-2-morpholinoethane-1,2-dione



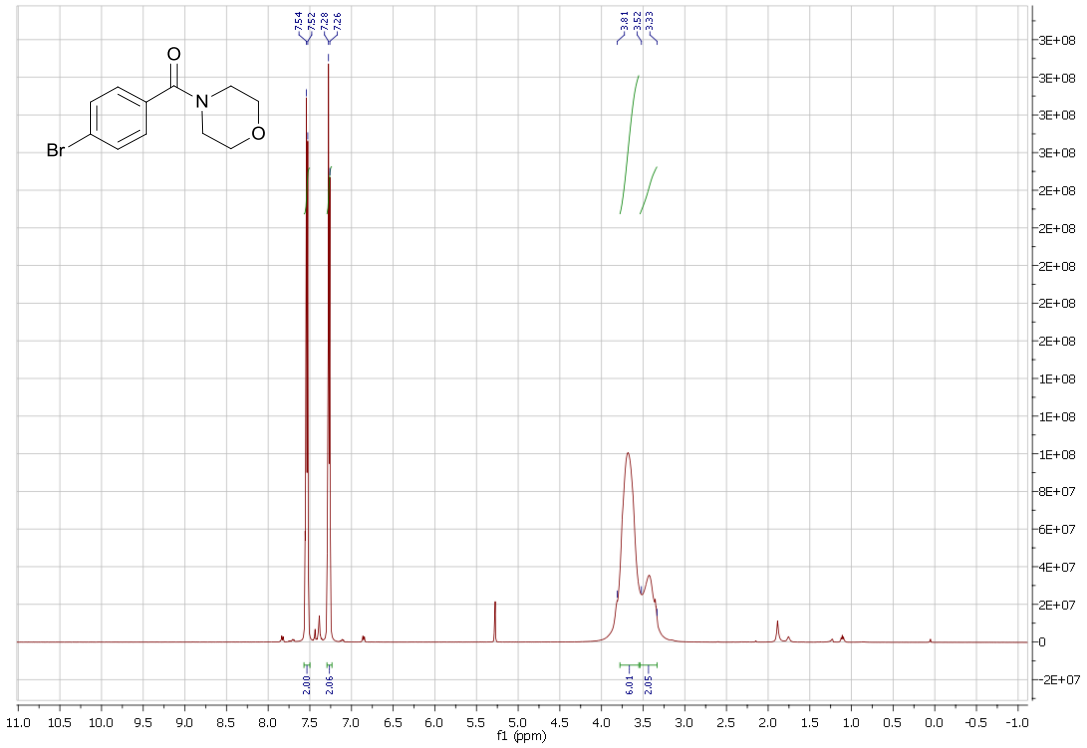
^{13}C NMR spectrum of 1-(3-bromophenyl)-2-morpholinoethane-1,2-dione



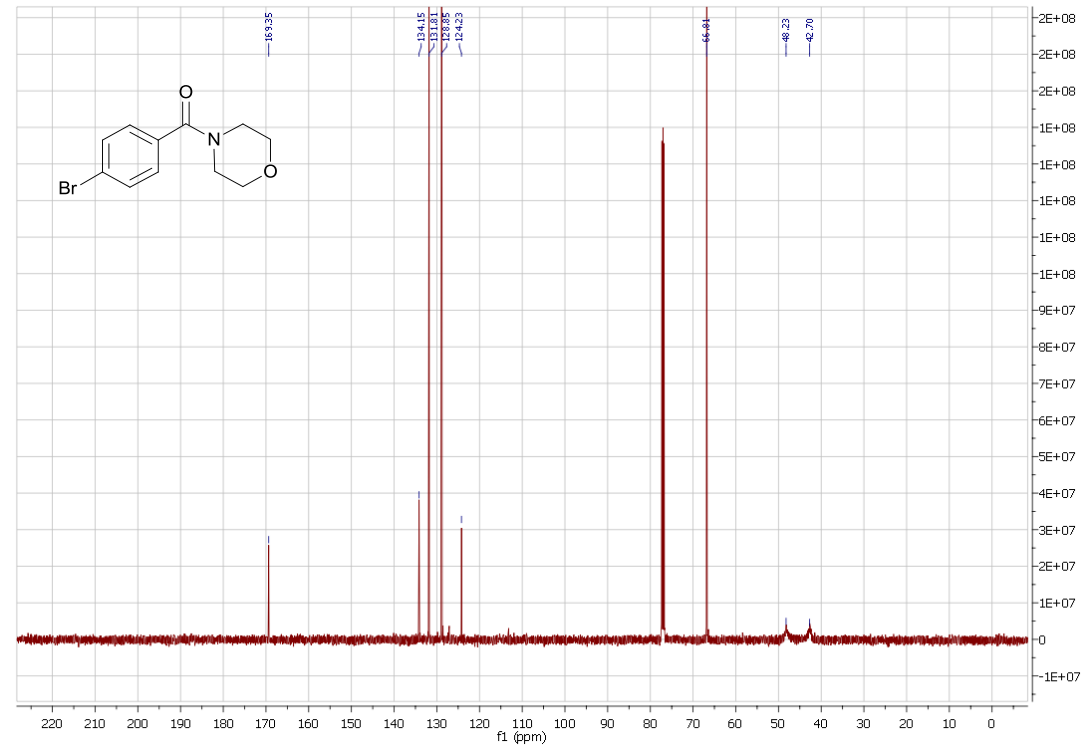
^{1}H NMR spectrum of **8b**



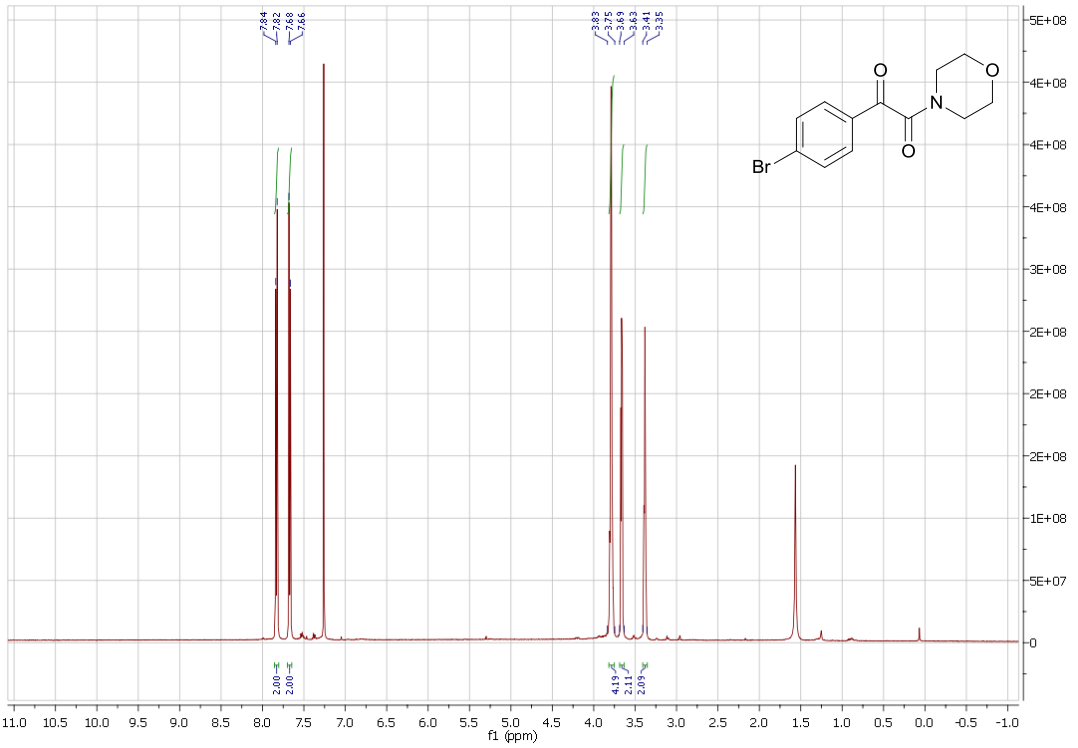
^{13}C NMR spectrum of **8b**



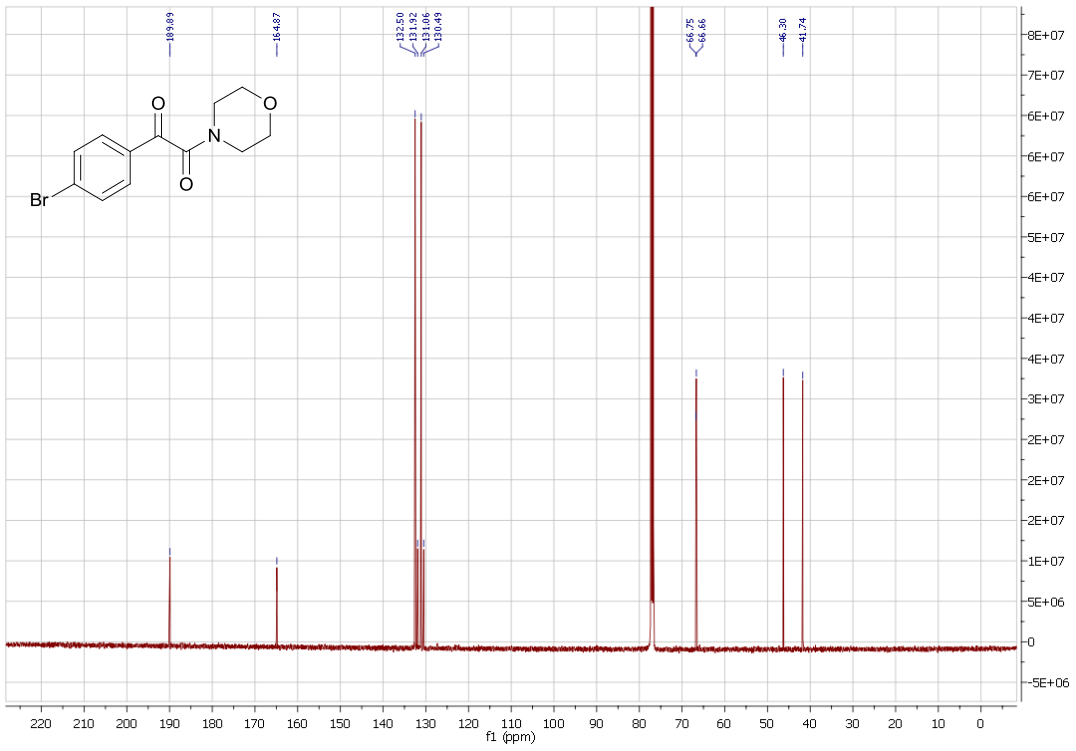
^{1}H NMR spectrum of morpholino(4-bromophenyl)methanone



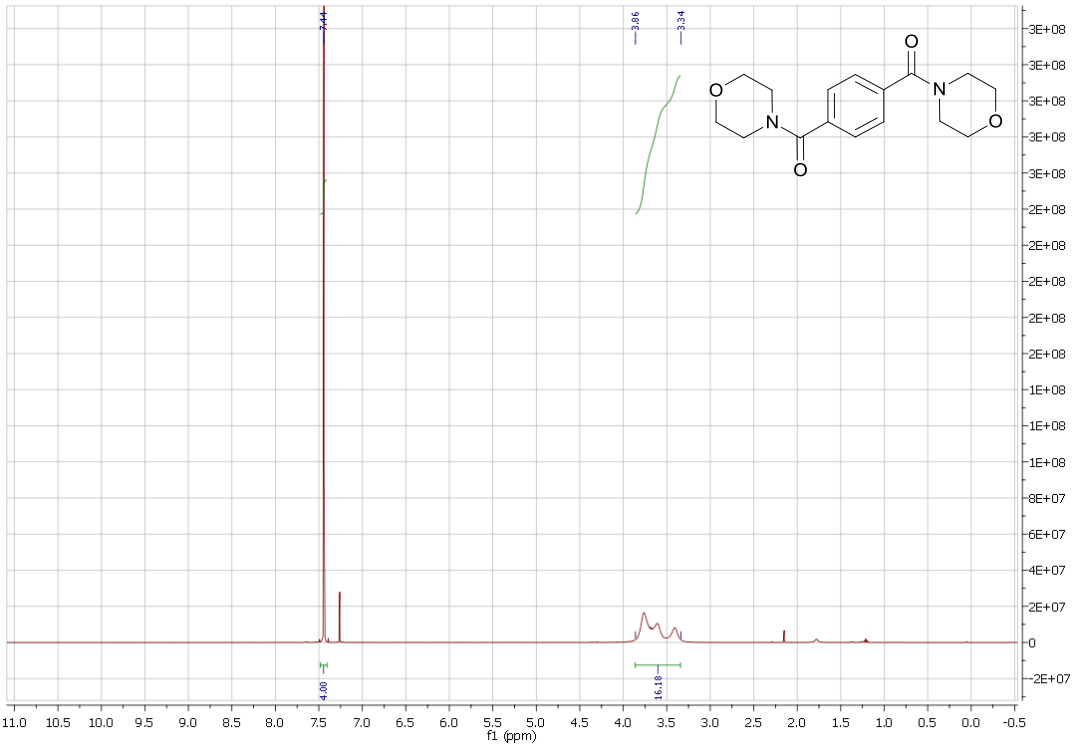
^{13}C NMR spectrum of morpholino(4-bromophenyl)methanone



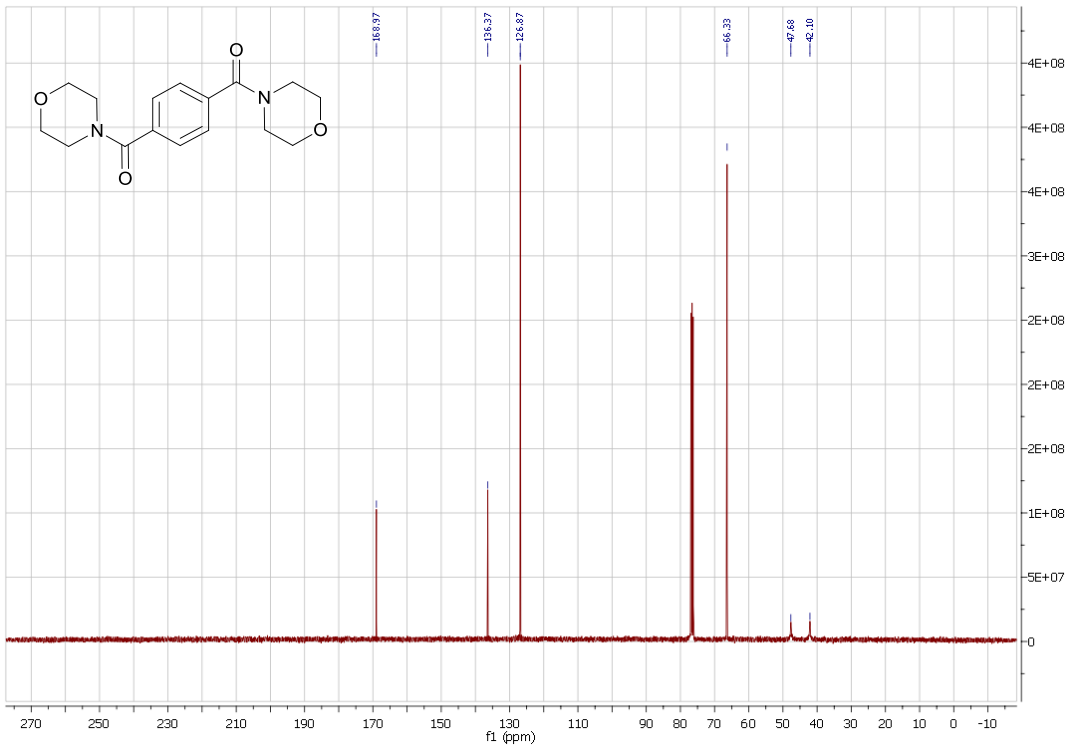
^{1}H NMR spectrum of 1-(4-bromophenyl)-2-morpholinoethane-1,2-dione



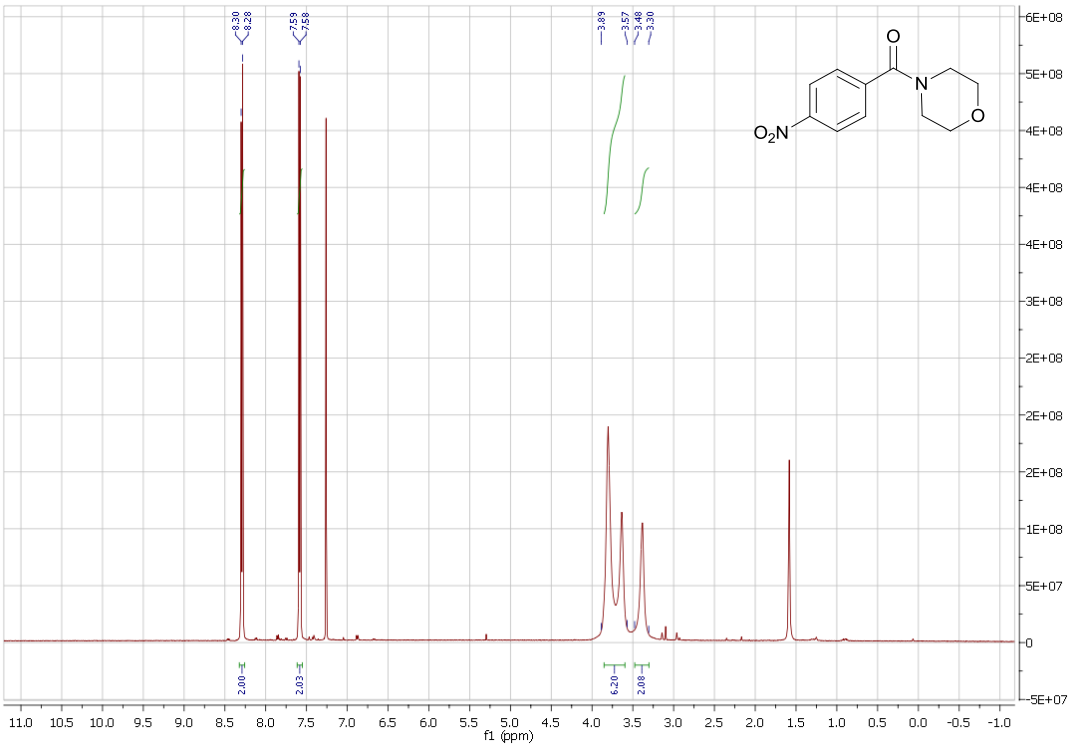
^{13}C NMR spectrum of 1-(4-bromophenyl)-2-morpholinoethane-1,2-dione



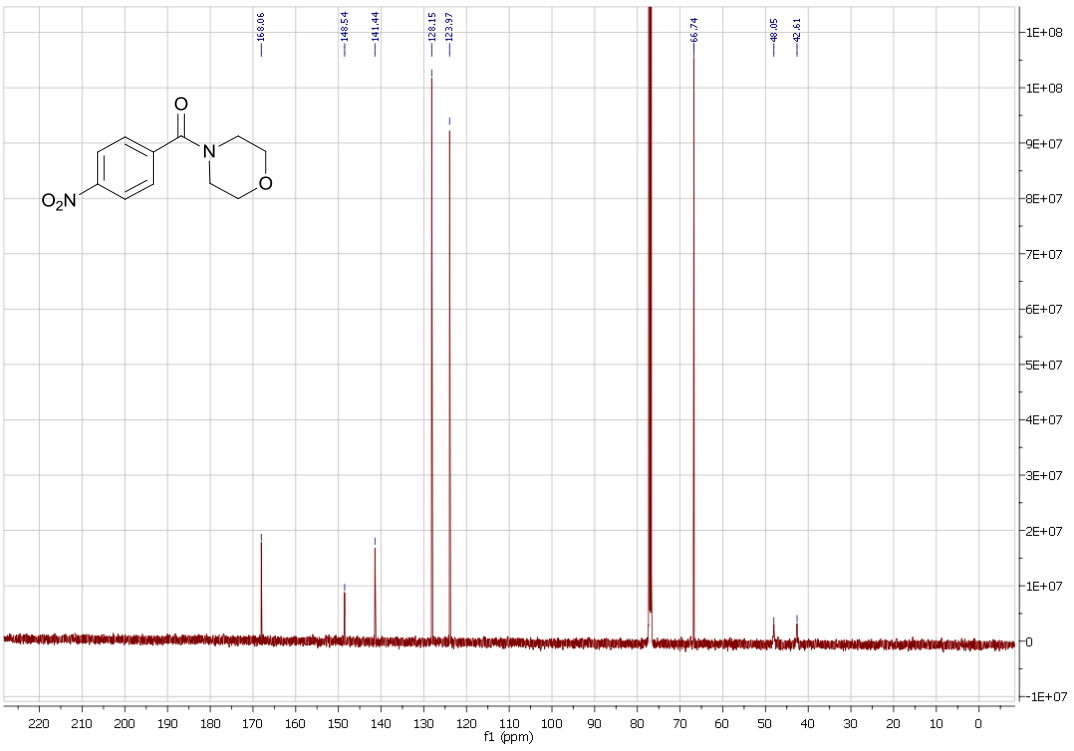
^{1}H NMR spectrum of **8c**



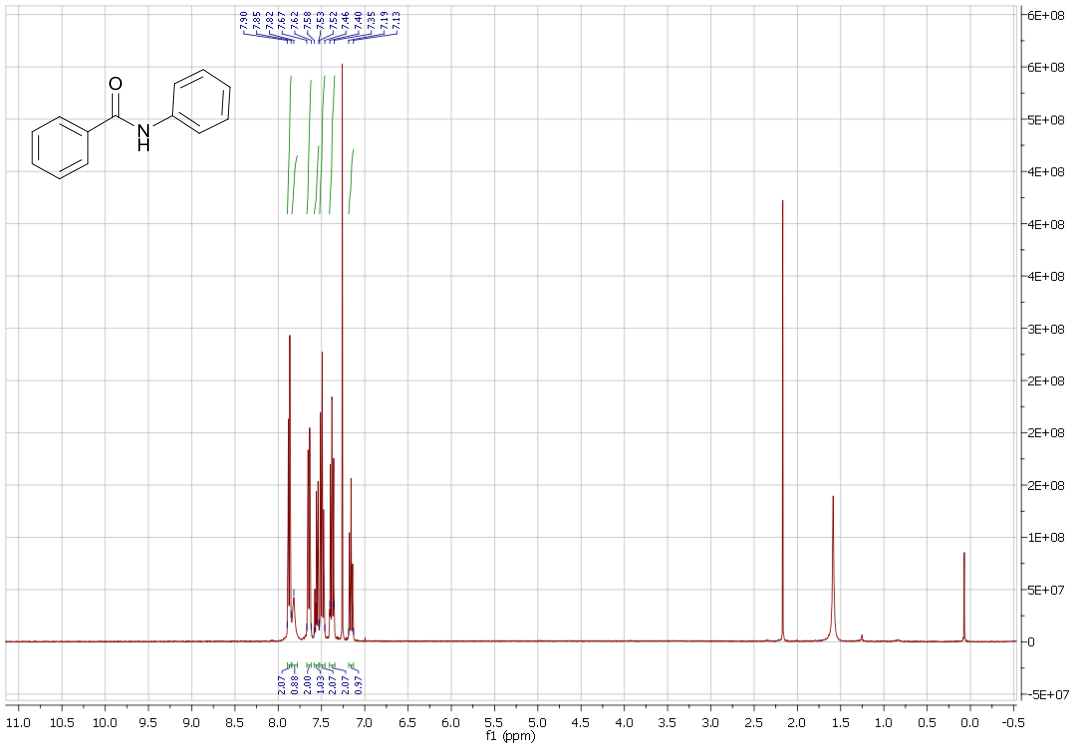
^{13}C NMR spectrum of **8c**



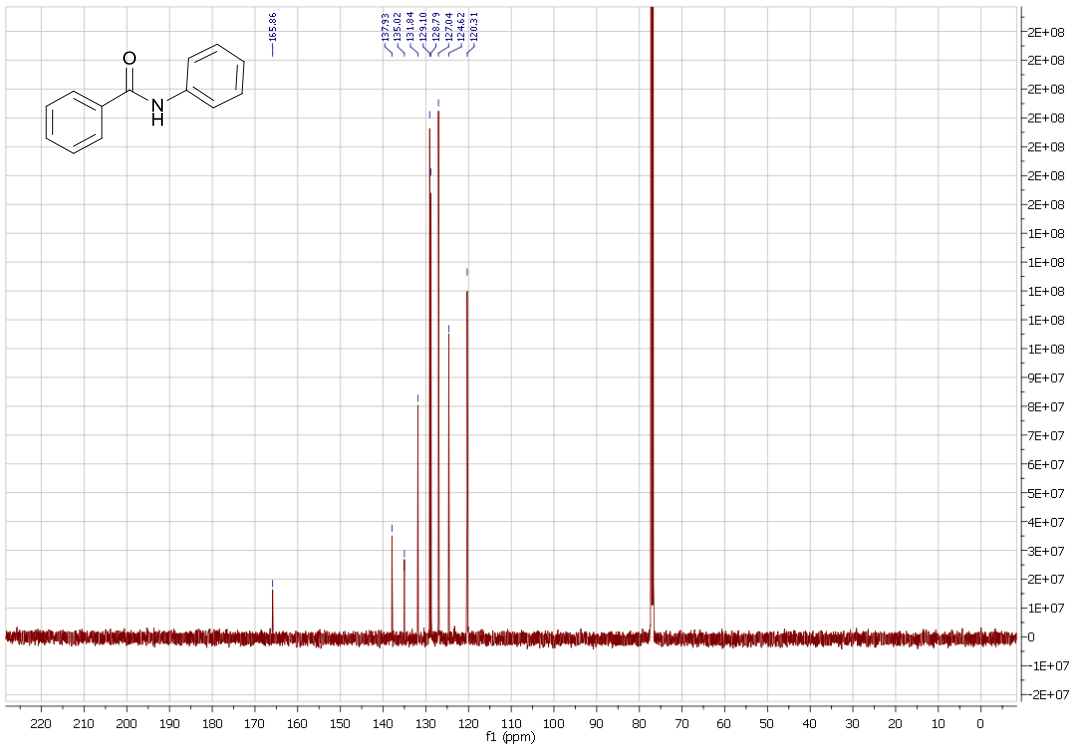
^{1}H NMR spectrum of morpholino(4-nitrophenyl)methanone



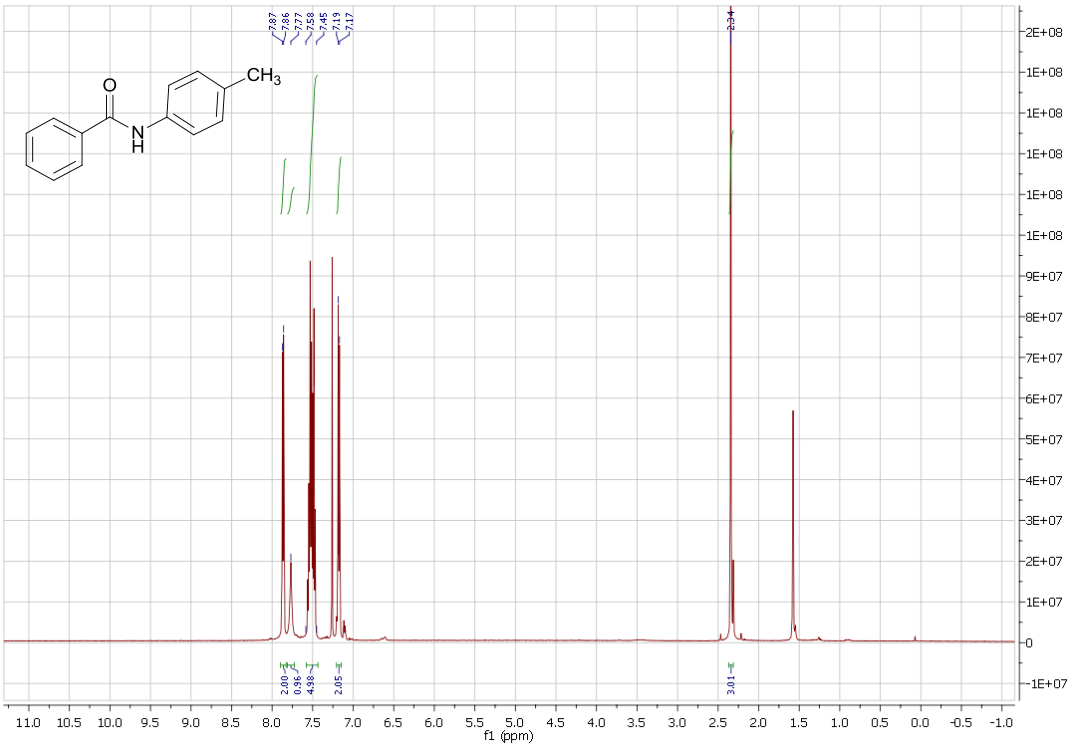
^{13}C NMR spectrum of morpholino(4-nitrophenyl)methanone



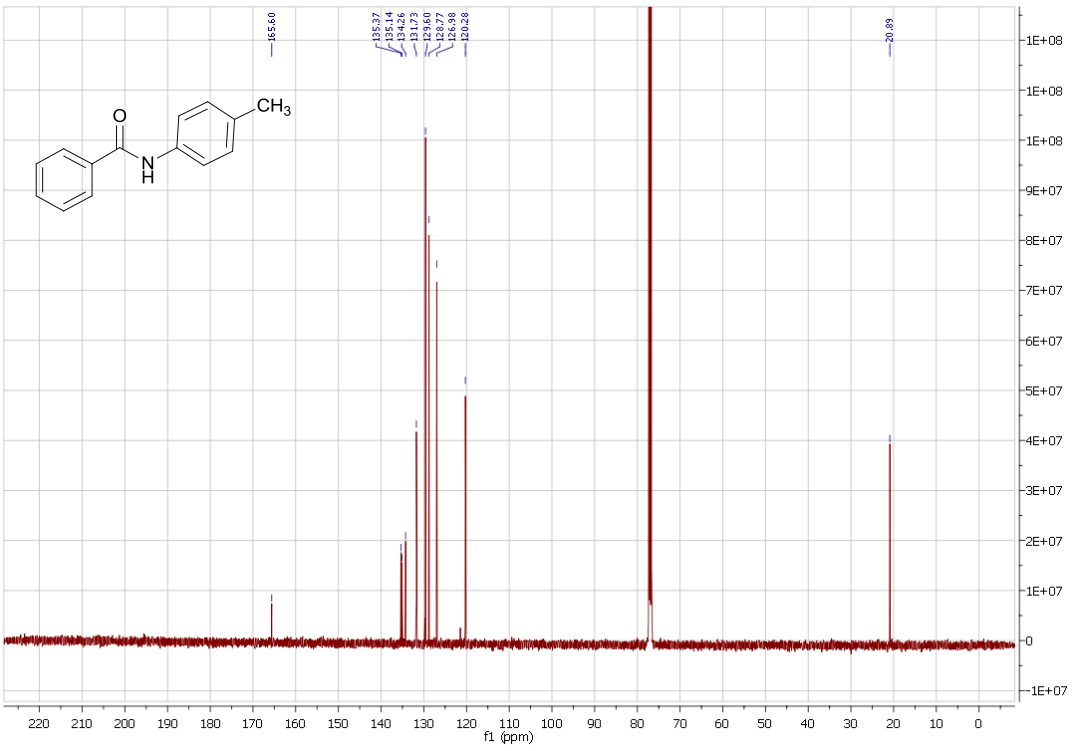
^{1}H NMR spectrum of N-phenylbenzamide



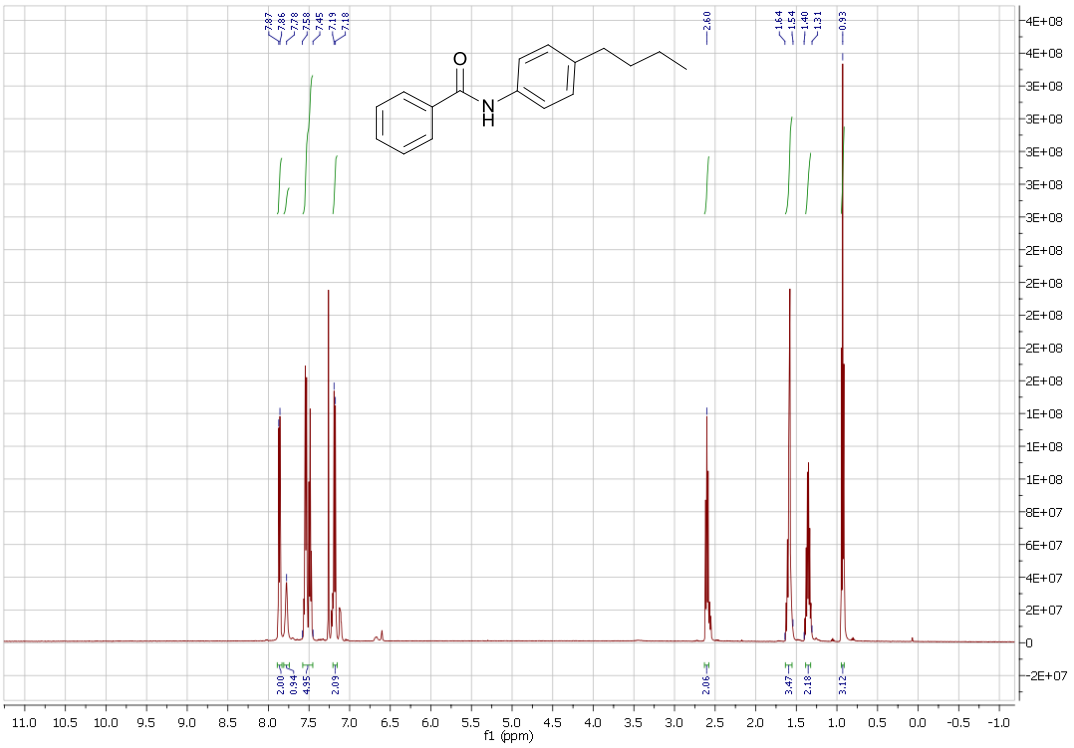
^{13}C NMR spectrum of N-phenylbenzamide



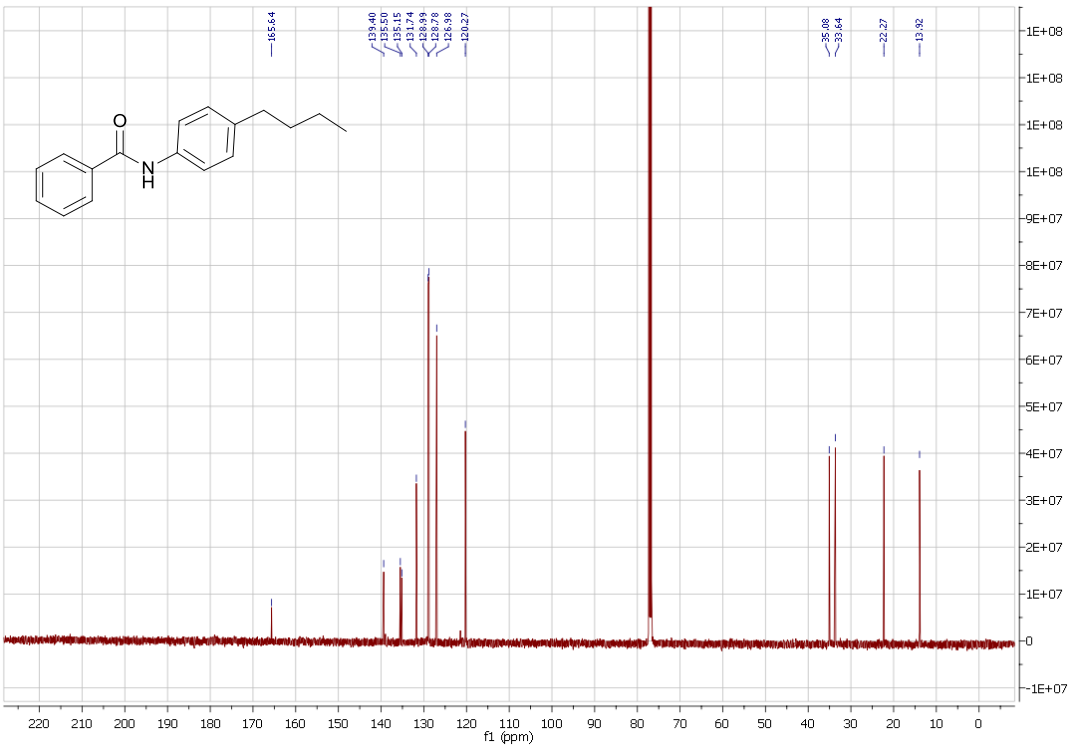
^{1}H NMR spectrum of N-(4-methylphenyl)benzamide



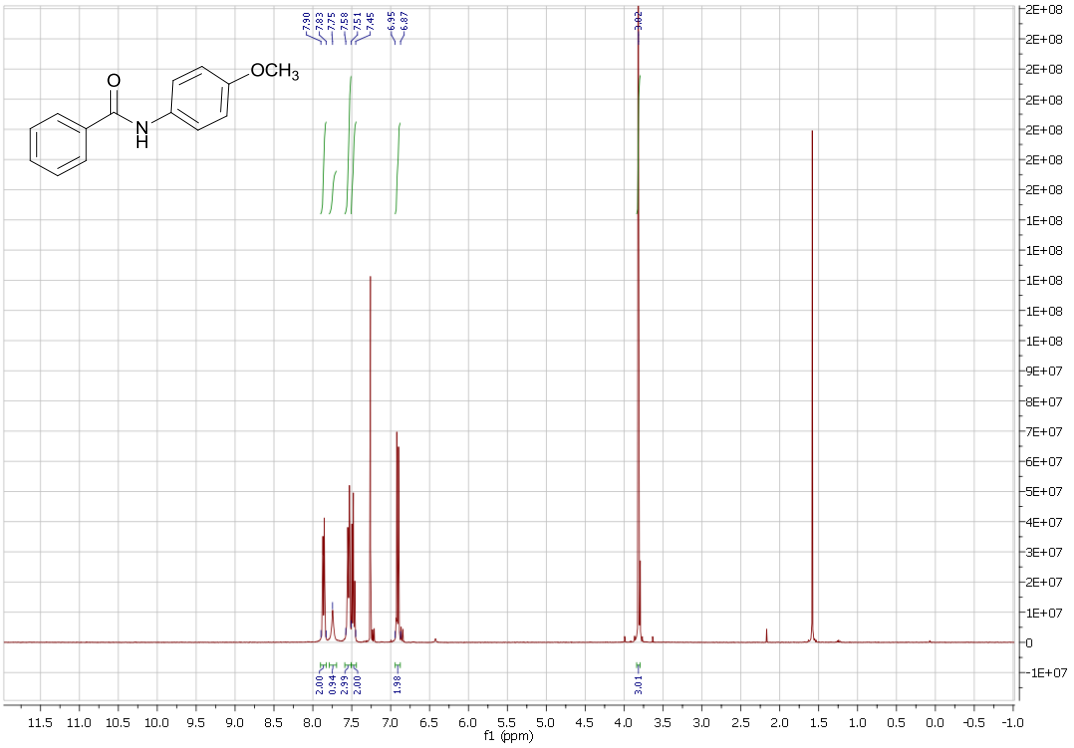
^{13}C NMR spectrum of N-(4-methylphenyl)benzamide



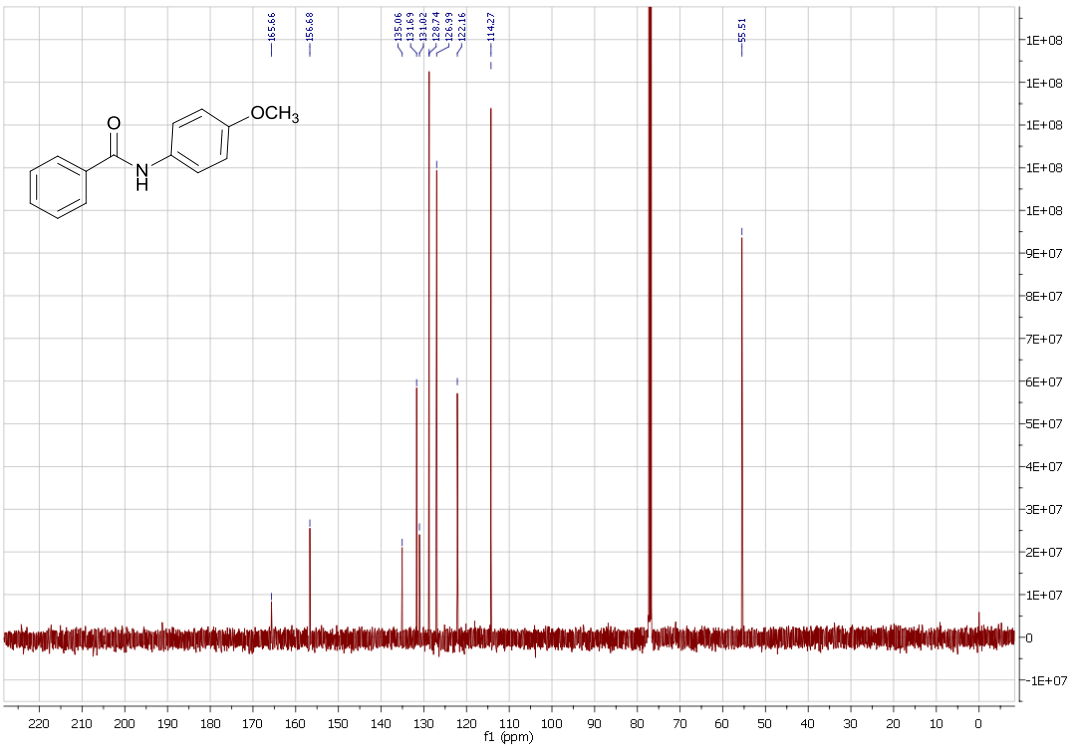
^{1}H NMR spectrum of N-(4-butylphenyl)benzamide



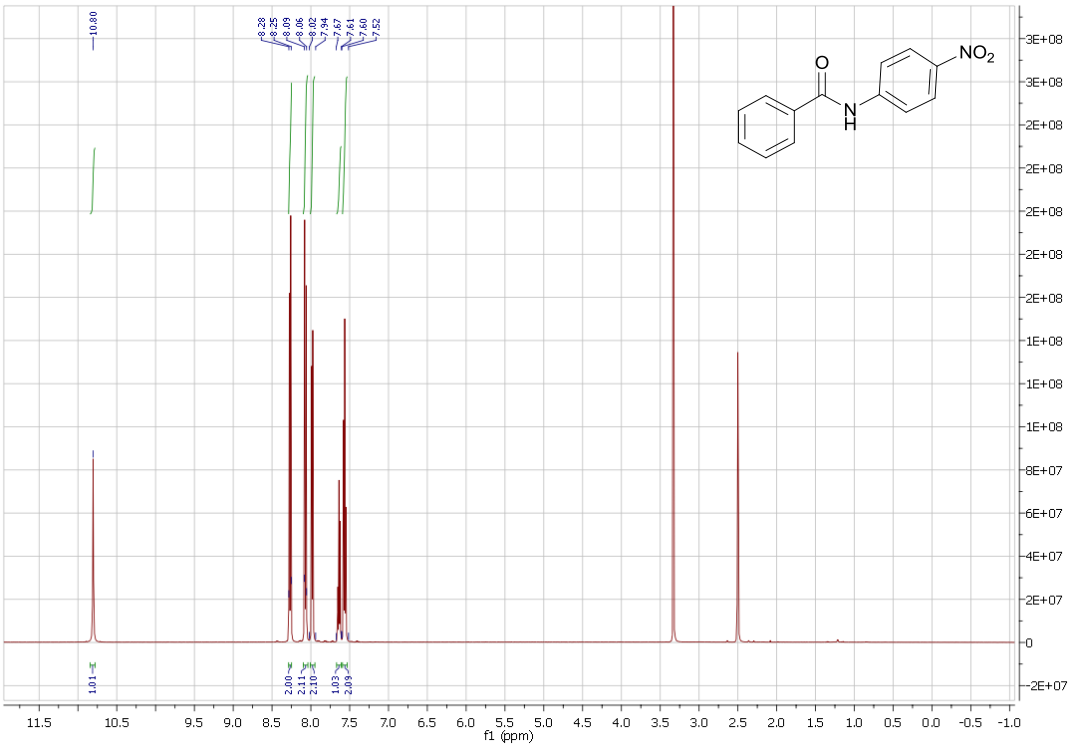
^{13}C NMR spectrum of N-(4-butylphenyl)benzamide



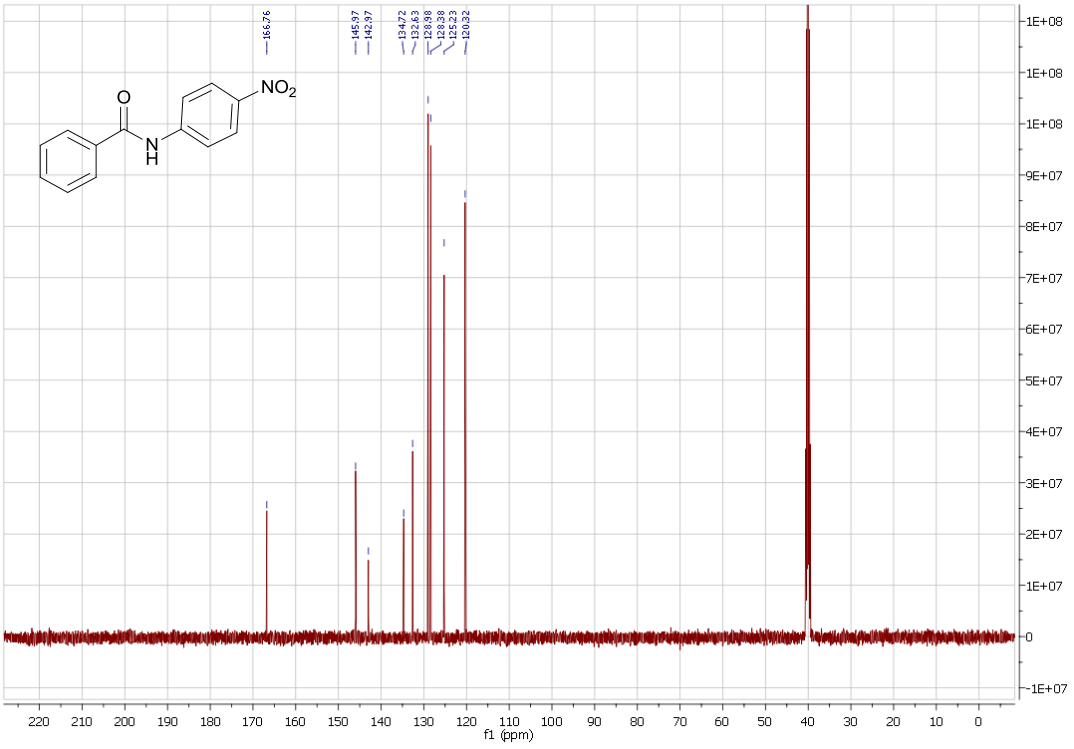
^{1}H NMR spectrum of N-(4-methoxyphenyl)benzamide



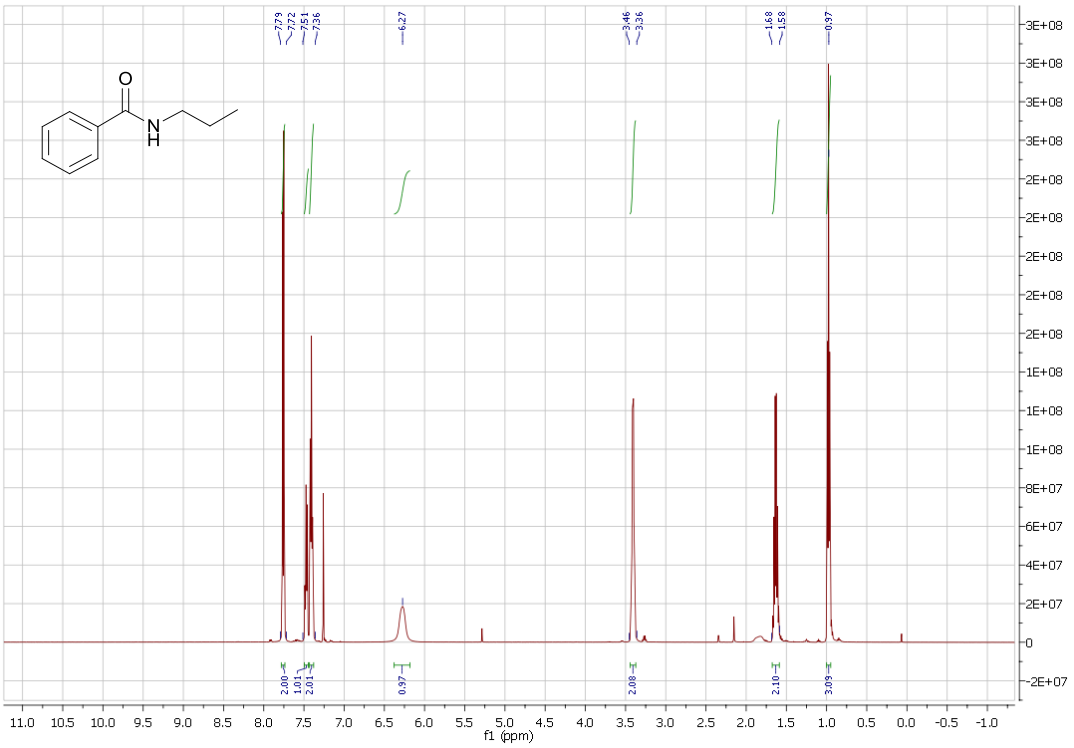
^{13}C NMR spectrum of N-(4-methoxyphenyl)benzamide



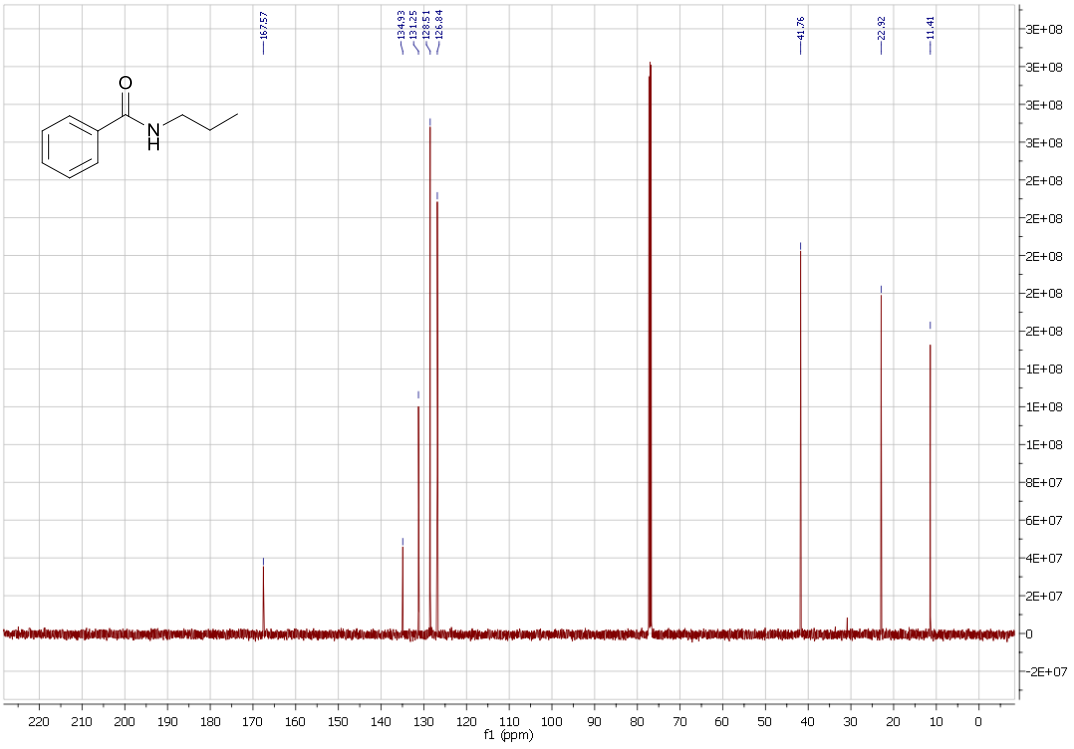
^{1}H NMR spectrum of N-(4-nitrophenyl)benzamide



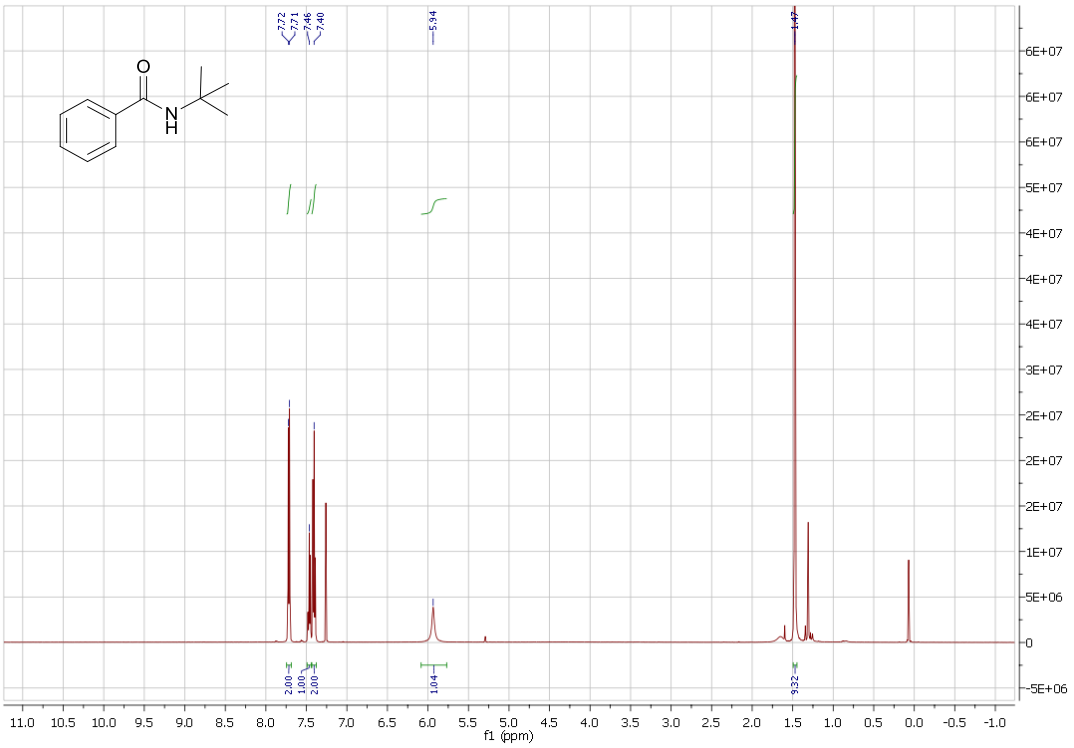
^{13}C NMR spectrum of N-(4-nitrophenyl)benzamide



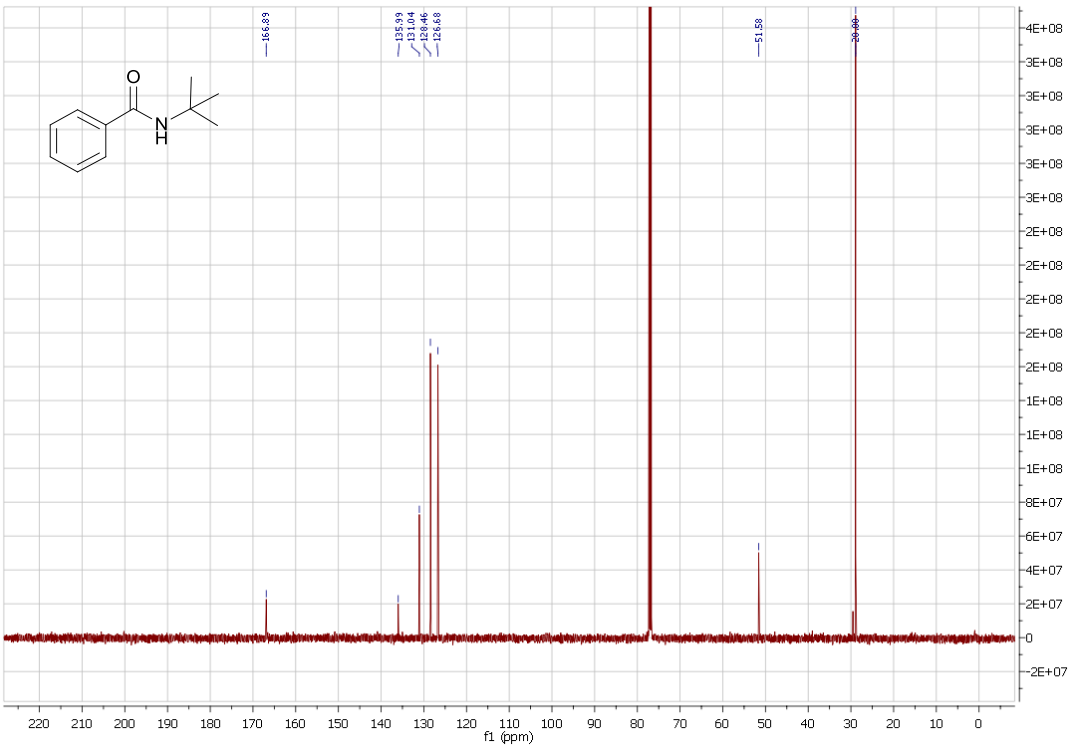
^{1}H NMR spectrum of N-propylbenzamide



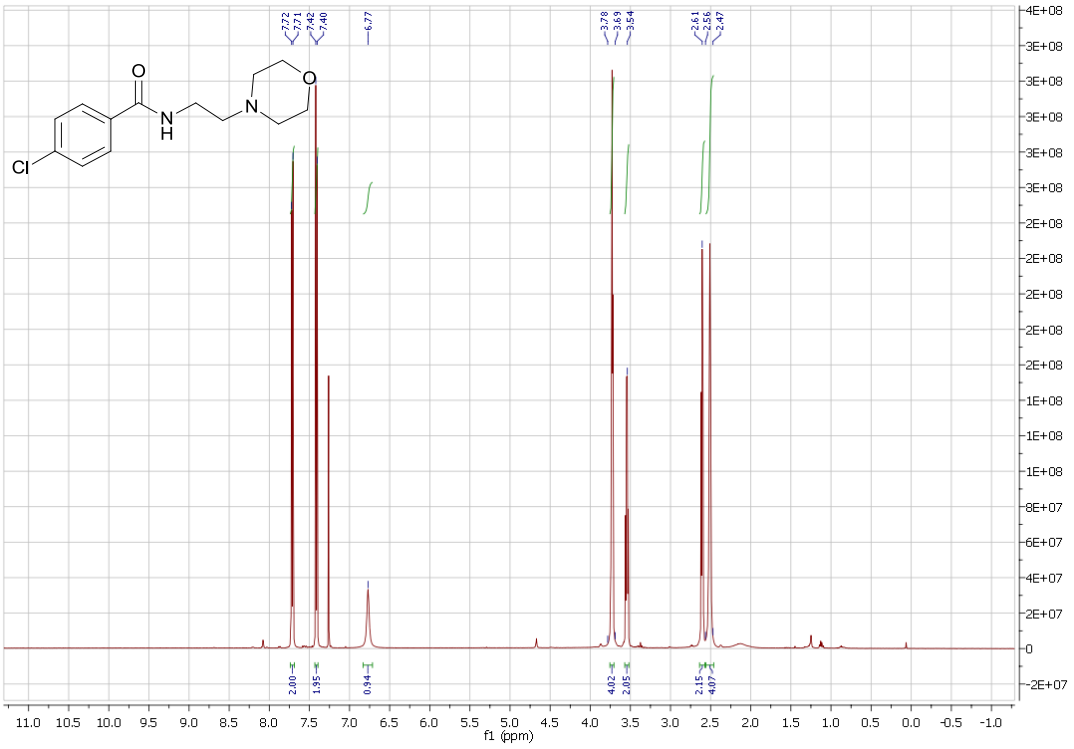
^{13}C NMR spectrum of N-propylbenzamide



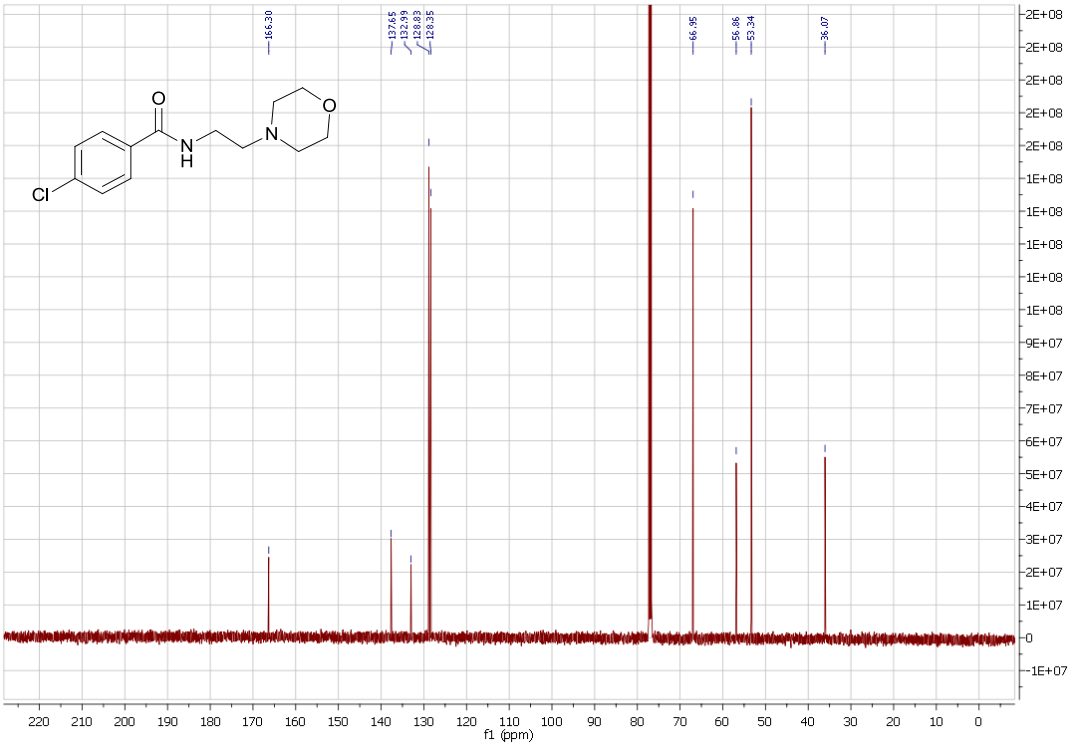
^{1}H NMR spectrum of N-*tert*-butylbenzamide



^{13}C NMR spectrum of N-*tert*-butylbenzamide



^{1}H NMR spectrum of 4-Chloro-N-(2-morpholinoethyl)benzamide



^{13}C NMR spectrum of 4-Chloro-N-(2-morpholinoethyl)benzamide

XI. References

1 Skoda-Földes, R *Top. Organomet. Chem.* **2015**, 51, 145–162.
2 Barnard, C. F. J. *Organometallics* **2008**, 27, 5402–5422.
3 Brennführer, A.; Neumann, H.; Beller, M. *Angew. Chem., Int. Ed.* **2009**, 48, 4114–4133.
4 Grigg, R.; Mutton, S. P. *Tetrahedron* **2010**, 66, 5515–5548.
5 Kumar, D.; Vemula, S. R.; Cook, G. R. *ACS Catal.* **2016**, 6, 4920–4945.
6 Roy, S.; Roy, S.; Gribble, G. W. *Tetrahedron*, **2012**, 68, 9867-9923.
7 De Risi, C.; Pollini, G. P.; Zanirato, V. *Chem. Rev.* **2016**, 116, 3241-3305.
8 Dang, T. T.; Zhu, Y.; Ghosh, S. C.; Chen, A.; Chai, C. L. L.; Seayad, A. M. *Chem. Commun.* **2012**, 48, 1805-1807.
9 Dang, T. T.; Zhu, Y.; Ngiam, J. S. Y.; Ghosh, S. C.; Chen, A.; Seayad, A. M. *ACS Catal.* **2013**, 3, 1406−1410.
10 Dang, T. T.; Chen, A; Seayad, A. M. *RSC Adv.*, **2014**, 4, 30019–30027.
11 Biying, A. O.; Yuanting, K. T.; Hosmane, N. S.; Yinghuai, Z. *J. Organomet. Chem.* **2013**, 747, 184-188.
12 Tinnis, F.; Verho, O.; Gustafson, K. P. J.; Tai, C.-W.; Bäckvall, J.-E.; Adolfsson, H. *Chem. Eur. J.* **2014**, 20, 5885–5889.
13 Mane, R. S.; Sasaki, T.; Bhanage, B. M. *RSC Adv.* **2015**, 5, 94776–94785.
14 Islam, S. M.; Ghosh, K.; Roy, A. S.; Molla, R. A. *RSC Adv.* **2014**, 4, 38986–38999.
15 Ibrahim, M. B.; Suleiman, R.; Fettouhi, M.; Ali, B. E. *RSC Adv.* **2016**, 6, 78826–78837.
16 Mei, H.; Hu, J.; Xiao, S.; Lei, Y.; Li, G. *Appl. Catal. A* **2014**, 475, 40–47.
17 Urbán, B.; Papp, M.; Srankó, D.; Skoda-Földes, R. *J. Mol. Catal. A*, **2015**, 397, 150–157.
18 Khedkar, M. V.; Sasaki, T.; Bhanage, B. M. *ACS Catal.* **2013**, 3, 287–293.
19 Yan, Y.; Zuo, H. P.; Jin, Z. L. *React. Func. Polym.***1997**, 32, 21–24.
20 Liu, J.; Zheng, S.; Su, W.; Xia, C. *Chin. J. Chem.* **2009**, 27, 623–627.
21 Genelot, M.; Villandier, N.; Bendjeriou, A.; Jaithong, P.; Djakovitch, L.; Dufaudz, V. *Catal. Sci. Technol.* **2012**, 2, 1886–1893.
22 Chen, B.; Li, F.; Huang, Z.; Lu, T.; Yuan, G. *Appl. Catal. A* **2014**, 481, 54–63.
23 Wang, Z.; Liu, C.; Huang, Y.; Hu, Y.; Zhang, B. *Chem. Commun.* **2016**, 52, 2960–2963.
24 Takács, E.; Varga, Cs.; Skoda-Földes, R.; Kollár, L. *Tetrahedron Lett.* **2007**, 48, 2453–2456.
25 Molla, R. A.; Iqubal, M. A.; Ghosh, K.; Roy, A. S.; Kamaluddin; Islam, S. M. *RSC Adv.* **2014**, 4, 48177–48190.
26 Papp, M.; Szabó, P.; Srankó, D.; Skoda-Földes, R. *RSC Adv.* **2016**, 6, 45349–45356.
27 Papp, M.; Skoda-Földes, R. *J. Mol. Catal. A*, **2013**, 378, 193–199.
28 D'Anna, F.; Noto, R. *Eur. J. Org. Chem.* **2014**, 4201–4223.
29 Sans, V.; Gelat, F.; Burguete, M. I.; Garcia-Verdugo, E.; Luis, S. V. *Catal. Today* **2012**, 196, 137–147.
30 Schwarz, J.; Bohm, V. P.; Gardiner, M. G.; Grosche, M. Herrmann, W. A.; Hieringer, W.; Raudaschl-Sieber, G. *Chem. Eur. J.* **2000**, 6, 1773–1780.
31 Massaro, M.; Riela, S.; Cavallaro, G.; Colletti, C. G.; Milioto, S.; Noto, R.; Parisi, F.; Lazzara, G. *J. Mol. Catal. A* **2015**, 408, 12–19.
32 Bayero, A. A *J. Pure Appl. Sci.* **2009**, 2, 61-69.
33 Field, L. D.; Messerle, B. A.; Vuong, K. Q.; Turner, P. *Organometallics* **2005**, 24, 4241-4250.
34 Jiang, Y.; Gao, Q.; Yu, H.; Chen, Y.; Deng, F. *Microporous and Mesoporous Mater.* **2007**, 103, 316–324.
35 Gushikem, Y.; Moreira, J. C. *J. Colloid Interface Sci.* **1985**, 107, 70-74.
36 Aher, S. B.; Bhagat, P. R. *Res. Chem. Intermed.* **2016**, 42, 5587–5596.
37 Han, L.; Park, M. S.; Choi, S. J.; Kim, Y. J.; Lee, S. M.; Park, D. W. *Catal. Lett.* **2012**, 142, 259–266.
38 Brun, M.; Berthet, A.; Bertolini, J. C. *J. Electron. Spectrosc. Relat. Phenom.* **1999**, 104, 55-60.
39 Pavia, C.; Ballerini, E.; Bivona, L. A.; Giacalone, F.; Aprile, C.; Vaccaro, L.; Gruttadauria, M. *Adv. Synth. Catal*. **2013**, 335, 2007-2018.
40 Urbán, B.; Srankó, D.; Sáfrán, G.; Ürge, L.; Darvas, F.; Bakos, J.; Skoda-Földes, R. *J. Mol. Catal. A* **2014**, 395, 364-372
41 Skoda-Földes, R.; Kollár, L. *Curr. Org. Chem*. **2002**, *6*, 1097-1119.
42 Wagner, C. D.; Riggs, W. M.; David, L. E.; Moulder, J. F; Muilenberg, G. T. *Handbook of X-Ray Photoelectron Spectroscopy*, Perkin-Elmer Corporation, Physical Electronics Division, Eden Prairie, Minn. 55344, **1979**.
43 Eremin, D. B.; Ananikov, V. P. *Coord. Chem. Rev.* **2017**, 346, 2-19.
44 Fitton, A.; Faulds, D.; Goa, K. L. *Drugs* **1992**, 43, 561–596.