Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Visible Light Photoredox Catalyzed Intermolecular Radical Addition of α-Halo Amides to Olefins

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1. General Information

All reactions were performed with oven-dried glassware and under an inert atmosphere (argon) unless otherwise stated.

Acetonitrile was distilled from calcium hydride and stored over 4Å molecular sieves under nitrogen/argon atmosphere. Tetrahydrofuran was distilled from Solvona® / benzophenone. Pentane was distilled in standard distillation apparatus. Other solvents were used as purchased unless otherwise stated.

Commercial reagents were used as purchased without further purification.

Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator. Chromatographic purification of products was carried out using Merck Kieselgel 60 silica gel (230-400 mesh). Thin-layer chromatography was carried out using Merck Kieselgel 60 F254 (230-400 mesh) fluorescent treated silica and were visualized under UV light (254 nm) or by staining with aqueous potassium permanganate solutions or vanillin alcoholic solution.

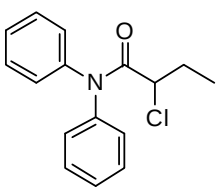
1H NMR spectra were recorded in deuterated solvents on Mercury 300, Inova 400 or Varian 600 spectrometers at 300, 400 or 600 MHz (respectively), with residual protic solvent as the internal standard. 13C NMR spectra were recorded in deuterated solvents on Mercury 300, Inova 400 or Varian 600 spectrometers at 75, 101 or 151 MHz, with the central peak of the deuterated solvent as the internal standard. Chemical shifts (δ) are given in parts per million (ppm), and coupling constants (J) are given in Hertz (Hz) rounded to the nearest 0.1 Hz. The 1H NMR spectra are reported as δ/ppm downfield from tetramethylsilane (multiplicity, coupling constant J/Hz, number of protons). The 13C NMR spectra are reported as δ/ppm. Assignments are aided by the use of DEPT-135, COSY and HMQC spectra where necessary. IR spectra were recorded on a Perkin Elmer 1760 FTIR spectrometer. Low resolution mass spectra (EI/CI) were recorded on a Thermo Finnigan SSQ 7000 mass spectrometer (EI) or a Thermo Finnigan MAT 95 mass spectrometer (CI).

Melting points were recorded on a Büchi Melting Point M-565 apparatus, at ambient pressure and are uncorrected.

2. Preparation of Substrates

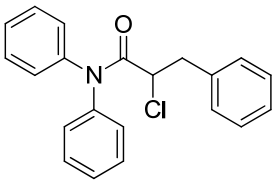
Diarylolefins **3c**−**q** have been synthesized through a Friedel-Crafts acylation and Wittig reaction. The analytical data are in agreement with the literature.[1]

Synthesis of 2-chloro-*N*,*N*-diphenylbutanamide (6c)



To a stirred solution of 2-chlorobutyric acid (5 mmol) and catalytical amount of DMF (0.05 mmol) in 50 mL of DCM was added oxalyl chloride (25 mmol) dropwise at room temperature. After ending of the gas evolution, the reaction mixture was stirred further for 1 hour, before the solvent and remaining oxalyl chloride were distilled off. Then, the residue was diluted with toluene and concentrated one more time to give 2-chlorobutanoyl chloride as a dark oil, which was dissolved in 30 mL of dry toluene. Diphenylamine (10 mmol) was added and the reaction mixture was heated at 100 °C for 16 hours. The solvent was removed under reduced pressure and the residue was subjected to flash column chromatography (Pent:DCM 1:1 to DCM). **yield** 38%; **m. p.** 51-54°C; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.52 – 7.12 (m, 10H), 4.21 (t, *J* = 7.2 Hz, 1H), 2.17 (m, 1H), 1.94 (m, 1H), 0.98 (t, *J* = 7.4 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 169.0, 142.1 (br), 141.8 (br), 130.0 (br), 128.9 (br), 128.6 (br), 128.4 (br), 126.5 (br), 126.1 (br), 56.4, 28.3, 10.9; **FT-IR** ν_{max}(KBr) cm^{-1}: 3342, 2967, 1670, 1591, 1488, 1374, 1322, 1283, 1159, 1076, 1029, 919, 816, 756, 695; **m/z** (EI) 273 ($[M]^{\bullet+}$, 62%), 167 ($[NPh_2-H]^+$, 100%).

Synthesis of 2-chloro-*N*,*N*,3-triphenylpropanamide (6d)

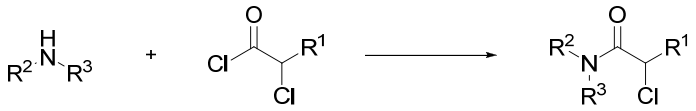


Phenylalanine (10 mmol) was dissolved in concentrated hydrochloric acid (35 mL) and cooled to -5 °C with a NaCl-ice bath. A solution of sodium nitrite (19 mmol) in 5 mL of water was added dropwise over period of 20 min. Then, the reaction mixture was allowed to stir at RT overnight, before 50 mL of EA was added. The organic was separated and the aqueous layer was extracted two more times with EA. The combined organic layers were dried over $MgSO_4$ and concentrated under reduced pressure to afford 2-chloro-3-phenylpropanoyl chloride as a yellow liquid. The residue was dissolved in 20 mL of dry toluene and diphenylamine (20 mmol) was added. The reaction mixture was then heated at 100 °C for 12 hours. The solvent was removed under reduced

[1] **3c**, **3e**, **3g**, **3k**, **3m**: D. Xing, B. Guan, G. Cai, Z. Fang, L. Yang and Z. Shi, *Org. Lett.*, **2006**, 8, 693; **3d**, **3f**, **3h**: C.-L. Sun, Y. Wang, X. Zhou, Z.-H. Wu, B.-J. Li, B.-T. Guan and Z.-J. Shi, *Chem. Eur. J.*, **2010**, 16, 5844; **3i**: Md. A. Jabbar, H. Shimakoshi and Y. Hisaeda, *Chem. Commun.*, **2007**, 1653; **3l**: A. Nuñez, B. Abarca, A. M. Cuadro, J. Alvarez-Builla and J. J. Vaquero, *J. Org. Chem.*, **2009**, 74, 4166; **3n**: Y. Onishi, Y. Yoneda, Y. Nishimoto, M. Yasuda and A. Baba, *Org. Lett.*, **2012**, 14, 5788; **3o**: J.-Y. Yu, R. Shimizu, R. Kuwano, *Angew. Chem. Int. Ed.*, **2010**, 49, 6396; **3p**: E. Boyd, S. Buksha, G. S. Coumbarides, M. Dingjan, J. Eames, R. V. H. Jones, M. Motevalli, R. A. Stenson and M. J. Suggate, *J. Chem. Crystallogr.*, **2007**, 37, 233; **3q**: Y. Onishi, Y. Yoneda, Y. Nishimoto, M. Yasuda and A. Baba, *Org. Lett.*, **2012**, 14, 5788.

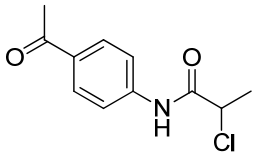
pressure and the residue solid was subjected to column chromatography (Pent:DCM 1:1 to DCM) to afford **6d** as a brownish solid. **yield** 47%; **m.p.** 72-75°C; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.38–7.23 (m, 8H), 7.18–7.15 (m, 3H), 7.06 (d, *J* = 7.8 Hz, 2H), 6.83 (br s, 2H), 4.44 (dd, *J* = 10.2, 5.2 Hz, 1H), 3.54 (dd, *J* = 13.1, 10.2 Hz, 1H), 3.10 (dd, *J* = 13.1, 5.2 Hz, 1H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 168.4, 142.0 (br), 141.5 (br), 136.3, 129.8, 128.9 (br), 128.9 (br), 128.6, 128.4 (br), 128.4 (br), 127.4, 126.5 (br), 126.1 (br), 54.6, 41.4; **FT-IR** ν_{max}(KBr) cm^{-1}: 3376, 3028, 2939, 1674, 1594, 1488, 1358, 1269, 1158, 1073, 1022, 945, 833, 752, 689; **m/z** (EI) 335 ($[M]^{\bullet+}$, 61%), 300 ($[M–Cl]^+$, 100%).

General procedure A for the synthesis of 2-chloro-N,N-diphenylamides



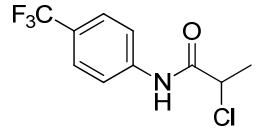
To a stirred solution of acid chloride (5 mmol) in DCM was added the amine (10 mmol) in one portion and stirred at RT overnight. Then, the reaction mixture was washed with 2% aqueous HCl and the organic layer was separated. The aqueous layer was once extracted with DCM and the overall organic layers were dried over MgSO4. The solvent was removed under reduced pressure and the product was isolated as specified below.

***N*-(4-acetylphenyl)-2-chloropropanamide (6h)**



Prepared according to the general procedure **A**. The title compound was isolated as a white solid after recrystallization (Pent/DCM); **yield** 34%; **m.p.** 126-128 °C; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 8.41 (br s, 1H), 7.96 (d, *J* = 8.8 Hz, 2H), 7.66 (d, *J* = 8.8 Hz, 2H), 4.55 (q, *J* = 7.1 Hz, 1H), 2.57 (s, 3H), 1.83 (d, *J* = 7.1 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 196.8, 167.6, 141.1, 133.6, 129.7, 119.2, 56.1, 26.5, 22.6; **FT-IR** ν_{max}(KBr) cm^{-1}: 3323, 2993, 1773, 1669, 1595, 1508, 1404, 1361, 1317, 1244, 1073, 962, 830, 765, 682; **m/z** (EI) 226 ($[M+H]^+$, 78%), 211 ($[M-CH_3]^{\bullet+}$, 100%).

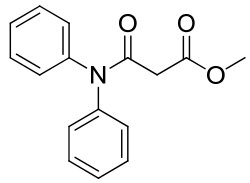
2-chloro-*N*-(4-(trifluoromethyl)phenyl)propanamide (6i)



Prepared according to the general procedure **A**. The title compound was isolated as a white solid after recrystallization (Pent/DCM); **yield** 48%; **m.p.** 127-129 °C; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 8.41 (s, 1H), 7.68 (d, *J* = 8.2 Hz, 2H), 7.60 (d, *J* = 8.1 Hz, 2H), 4.56 (q, *J* = 6.8 Hz, 1H), 1.83 (d, *J* = 6.8 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 167.7, 140.0, 126.8 (q, *J* = 32.9 Hz), 126.3 (q, *J* = 3.6 Hz), 123.9 (q, *J* = 272.8 Hz), 119,6, 56.0, 22.5; **^{19}F-NMR** (376 MHz, $CDCl_3$) δ −62.3 **FT-IR** ν_{max}(KBr) cm^{-1}: 3260, 2932, 1729, 1670, 1602, 1535, 1413, 1320, 1254, 1169, 1111, 1062, 912, 837, 769, 697; **m/z** (EI) 251 ($[M]^{\bullet+}$, 100%), 189 ($[M-C_2H_5Cl]^{\bullet+}$, 35%).

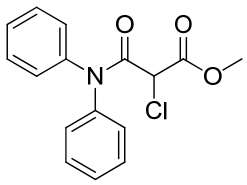
α-Chloro amides **6a**, **6b**, **6f** and **6g** have been prepared following the general method **A**. The corresponding analytical data are in agreement with the literature.[2]

methyl 3-(diphenylamino)-3-oxopropanoate (9)



To a solution of methyl malonyl chloride (5 mmol) in 25 mL of dry THF was added a solution of diphenylamine (5 mmol) in 10 mL of dry THF at 0 °C. After addition, the reaction mixture was stirred at RT for 2 hours, before diluted with water (50 mL) and diethyl ether (50 mL). The organic phase was separated and the aqueous phase was extracted twice with diethylether. The combined organic phases were dried over Na_2SO_4 and the solvent was removed to give **9** as a white solid. **yield** 99%; **m.p.** 83-84 °C; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.54 – 7.10 (m, 10H), 3.68 (s, 3H), 3.40 (s, 2H); **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 167.9, 165.9, 142.4 (br), 142.2 (br), 129.9 (br), 128.9 (br), 128.6 (br), 128.4 (br), 126.4 (br), 126.2 (br), 52.3, 42.5.

methyl 2-chloro-3-(diphenylamino)-3-oxopropanoate (6e)



An oven dried Schlenk flask was charged with **9** and 25 mL of dry THF and cooled to –75 °C. LHMDS (1M in THF) was added slowly and the reaction mixture was stirred at –70 °C for 1 hour, whereby a formation of a white precipitate could be observed. NCS (mmol) was added in one portion, and the reaction was allowed to warm up slowly to RT overnight. After quenching the mixture with sat. NH_4Cl-solution and addition of Et_2O, the organic layer was separated. The aqueous layer was once extracted with EA and the combined organic layers were dried over $MgSO_4$ and the solvent was removed under reduced pressure. The curde oil was purified by flash column chromatography (Pent:EA 4:1) to obtain **6e** as a white solid. **yield** 87%; **m.p.** 98-99 °C; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.51 – 7.15 (m, 3H), 5.02 (s, 1H), 3.83 (s, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 165.3, 164.4, 141.6 (br), 141.4 (br), 130.2 (br), 129.0 (br), 128.9 (br), 128.7 (br), 126.8 (br), 125.9 (br), 54.5, 53.8; **FT-IR** ν_{max}(KBr) cm^{-1}: 3341, 2968, 1755, 1668, 1484, 1355, 1283, 1174, 996, 825, 751, 685; **m/z** (EI) 303 ([M]$^{\bullet+}$, 100%).

[2] **6a**: S. Kumar, A. K. Wahi and R. Singh, *Eur. J. Med. Chem.*, **2011**, 46, 4753; **6b**: M. Murphy, D. Lynch, M. Schaeffer, M. Kissane, J. Chopra, E. O'Brien, A. Ford, G. Ferguson and A. R. Maguire, *Org. Biomol. Chem.* **2007**, 5, 1228; **6f**: K. Weidner, A. Giroult, P. Panchaud and P. Renaud, *J. Am. Chem. Soc.*, **2010**, 132, 17511; **6g**: J. W. Comerford, J. H. Clark, D. J. Macquarrie and S. W Breeden, *Chem. Commun.*, **2009**, 2562.

3. *Full Optimisation Studies*

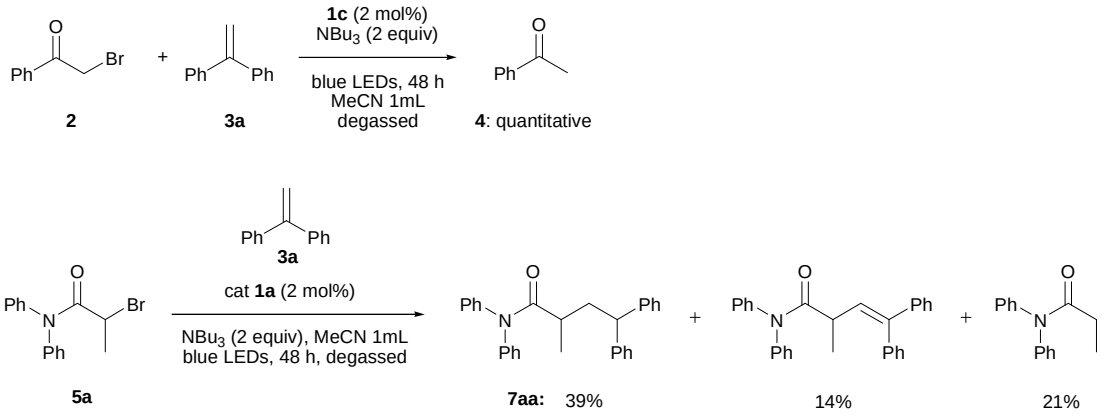
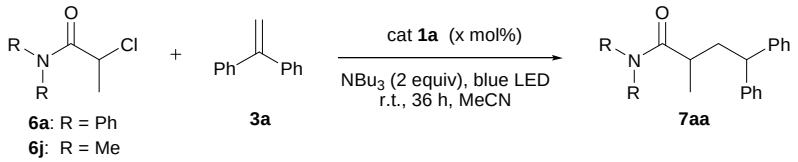


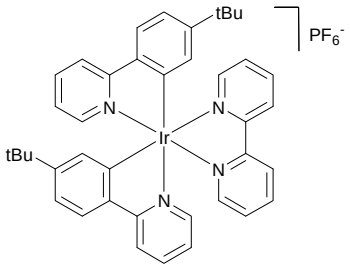
Table 1. Reaction condition screening[a]



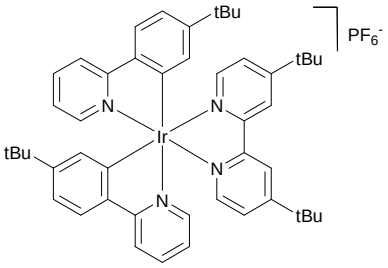
R	Olefin : Amide	Catalyst loading (x mol%)	$Yield^{NMR\ [b]}/Yield^{isol}$
Me	3 : 1	1	- /27
Me	3 : 1	2	51 / 39
Me	3 : 1	3	55 / -
Me	3 : 1	4	50 / -
Me	1 : 1	2	13 / -
Me	1 : 1.5[c]	2	21 / -
Ph	3 : 1[d]	2	81 / 79

[a] The reaction was performed with amide **6** (0.13 mmol), 1,1-diphenylethylene **3a** (0.39 mmol), NBu_3 (0.26 mmol) in 1.0 mL of degassed MeCN. [b] Determined by ^{1}H-NMR analysis of the crude reaction mixture, which was filtered through a short plug of silica using Pent:EA 5:1 as eluent. Phenyl benzyl ether was used as internal standard. [c] The reaction has been carried out with amide (0.13 mmol), 1,1-diphenylethylene (0.2 mmol), NBu_3 (0.26 mmol) in 1.0 mL of degassed MeCN. [d] The reaction was carried out in a 0.13 mmol scale respect to the olefin **3a**.

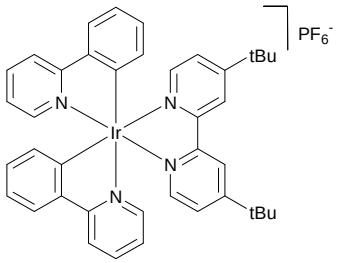
Table 2. Catalyst screening[a]



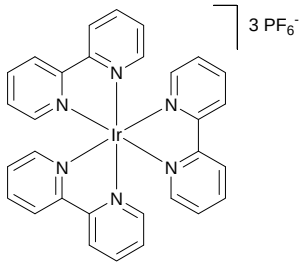
1a 81%



1b 77%



1c 73%



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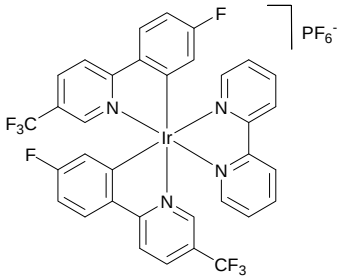

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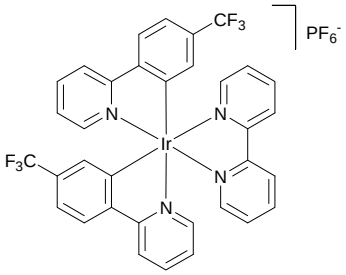

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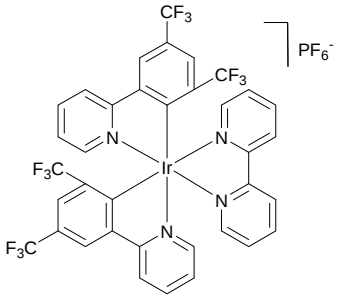

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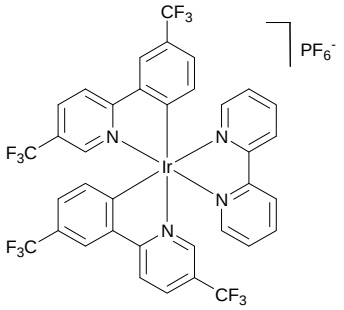
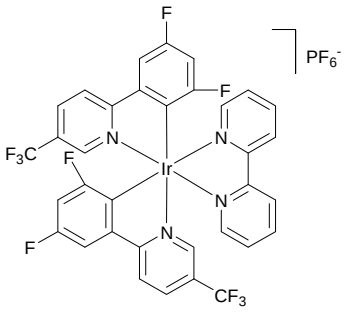
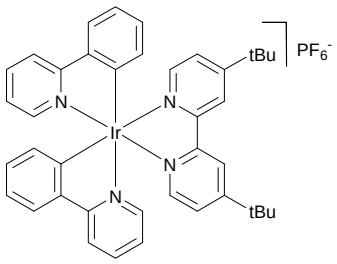


36%



37%



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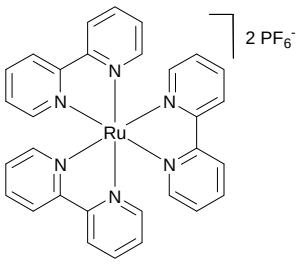
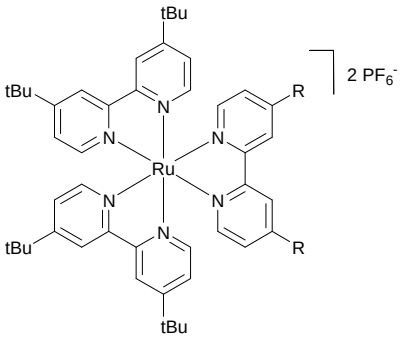


23%



R = H **36%**

R = tBu **24%**

[a] The reaction was performed with amide **6a** (0.13 mmol), 1,1-diphenylethylene **3a** (0.39 mmol), NBu_3 (0.26 mmol) and catalyst (2 mol%) in 1.0 mL of degassed MeCN. [b] Determined by ^{1}H-NMR analysis of the crude reaction mixture, which was filtered through a short plug of silica using Pent:EA 5:1 as eluent. Mesitylene has been used as internal standard.

Table 3. Solvent screening[a]

Solvent	Yield$^{NMR[b]}$
MeCN	81
DMSO	54
DMF	47
$PhCF_3$	5
EA	5
DCM	39
MeOH	46
EtOH	16

[a] The reaction was performed with amide **6a** (0.13 mmol), 1,1-diphenylethylene **3a** (0.39 mmol), NBu_3 (0.26 mmol) and catalyst **1a** (2 mol%) in 1.0 mL of degassed solvent. [b] Determined by ^{1}H-NMR analysis of the crude reaction mixture, which was filtered through a short plug of silica using Pent:EA 5:1 as eluent. Phenyl benzyl ether was used as internal standard.

Table 4. Amine screening[a]

Amine	Yield$^{NMR[b]}$
NEt_3	74
NBu_3	81
DIPEA	77
NPh_2(Ph-4OMe)	<5

[a] The reaction was performed with amide **6a** (0.13 mmol), 1,1-diphenylethylene **3a** (0.39 mmol), amine (0.26 mmol) and catalyst **1a** (2 mol%) in 1.0 mL of degassed MeCN. [b] Determined by ^{1}H-NMR analysis of the crude reaction mixture, which was filtered through a short plug of silica using Pent:EA 5:1 as eluent. Phenyl benzyl ether was used as internal standard.

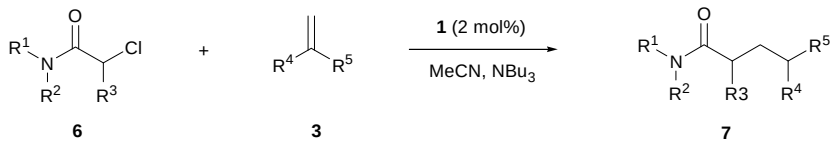
Table 5. Control Experiments[a]

Entry[a]	Deviation from the standard condition	Yield [%][b]
1	-	81
3	No degassing	30
4	No amine	0
5	No catalyst	0
6	No light	0
7	TEMPO as additive[c]	0

[a] Reaction condition: **6a** (0.13 mmol), **3a** (0.39 mmol), NBu_3 (0.26 mmol) and 2 mol% of **1a** in degassed MeCN (1.0 mL) under irradiation of blue LED at RT. [b] NMR yield: benzyl phenyl ether has been used as internal standard. [c] 2.0 equiv of TEMPO has been used.

4. *Preparation and Characterisation of Products*

General procedure B for the reductive alkylation of α-chloroamide and olefin

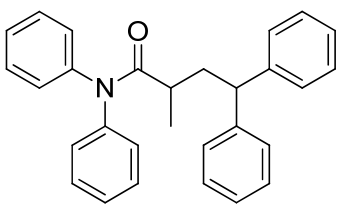


A Schlenk-tube was charged with α-chloroamide **6** (0.13 mmol), diarylalkene **3** (0.39 mmol), catalyst **1a** or **1b** and magnetic stir bar. It was capped with a rubber septum and evacuated and backfilled with argon. Then, degassed MeCN (1.0 mL) and NBu_3 were added via syringe. The vial was placed in a 100 mL beaker with blue LEDs wrapped inside and the reaction mixture was stirred at RT for 48 h. The solvent was removed *in vacuo* and the residue was purified by column chromatography on silica using the specified eluent system.

General procedure C for the reductive alkylation of α-chloroamide and olefin

A Schlenk-tube was charged with α-chloroamide **6** (0.13 mmol), diarylalkene **3** (0.39 mmol), catalyst **1a** or **1b** and magnetic stir bar. It was capped with a rubber septum and evacuated and backfilled with argon. Then, degassed MeCN (1.5 mL), degassed dichlormethane (0.5 mL) and NBu_3 were added via syringe. The vial was placed in a 100 mL beaker with blue LEDs wrapped inside and the reaction mixture was stirred at RT for 48 h. The solvent was removed *in vacuo* and the residue was purified by column chromatography on silica using the specified eluent system. **Note**: In some cases, the product **7** could not be isolated in a pure form, being contaminated with the unconsumed chloro amide **6**, the mixture has been post-treated as follows: The mixture (**6**+**7**), NaI (0.05 mmol) and 0.4 mL of a solution of 4-Methylpiperazine (0. 1 mmol/mL) in MeCN were heated at 80 °C for 6 hours. Then the solvent was removed and the crude mixture was purified as mentioned above.

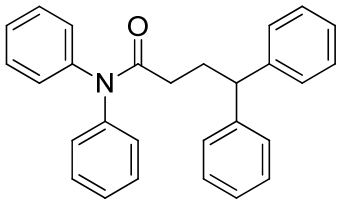
2-methyl-*N*,*N*,4,4-tetraphenylbutanamide (7aa)



The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 79%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.39–7.02 (m, 18H), 6.76 (br s, 2H), 4.12 (dd, *J* = 9.6, 6.7 Hz, 1H), 2.64–2.56 (m, 1H), 2.51 (m, 1H), 2.09 (m, 1H), 1.13 (d, *J* = 6.8 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 176.4, 144.9, 143.7, 142.9 (br), 142.3 (br), 129.4 (br), 128.8 (br), 128.7, 128.5 (br), 128.4, 128.2, 127.6, 127.4 (br), 126.5 (br), 126.3, 126.0, 125.9 (br), 48.6, 39.9, 35.6, 18.4; **FT-IR** ν_{max}(ATR) cm^{1}: 3442, 3060, 3029, 2970, 2927, 1670 (C=O), 1593, 1492, 1451, 1382,

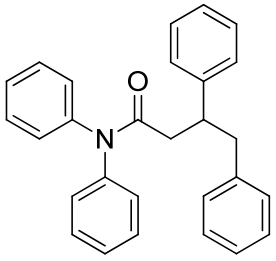
1262, 1160, 1073, 1028, 908, 756, 699; **m/z** (EI) 406 ($[M+H]^+$, 100%), 225 ($[Ph_2N(CO)CH_2CH_3]^+$, 66%).

***N*,*N*,4,4-tetraphenylbutanamide (7ba)**



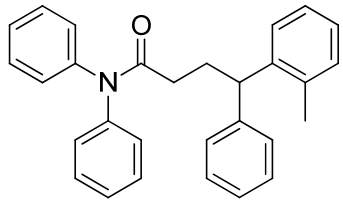
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 90%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.45–6.91 (m, 20H), 3.99 (t, *J* = 8.0 Hz, 1H), 2.43 (td, *J* = 8.0, 7.2 Hz, 2H), 2.27 (t, *J* = 7.2 Hz, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.8, 144.2, 142.8, 129.6 (br), 129.0 (br), 128.4, 127.9, 126.4 (br), 126.2, 50.2, 33.6, 31.2; **FT-IR** ν_{max}(ATR) cm^{-1}: 3031, 2929, 1669, 1593, 1491, 1450, 1373, 1275, 911, 749, 700; **m/z** (EI) 391 ($[M]^{\bullet+}$, 100%), 211 ($[Ph_2N(CO)CH_3]^+$, 57%), 167 ($[Ph_2CHCH_2]^{\bullet+}$, 55%).

***N*,*N*,3,4-tetraphenylbutanamide (7bb)**



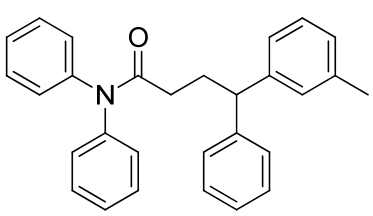
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:EA 5:1 to 3:1). **Yield** 28%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.40–7.07 (m, 3H), 7.04–7.00 (m, 2H), 6.94 (d, *J* = 7.4 Hz, 15H), 3.60–3.53 (m, 1H), 2.89 (m, 2H), 2.60–2.57 (m, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 171.8, 143.6, 142.8, 139.8, 129.6 (br), 129.1, 128.7 (br), 128.2, 128.1, 127.9, 126.5, 126.4 (br), 126.0, 44.6, 42.4, 40.9; **FT-IR** ν_{max}(ATR) cm^{-1}: 3030, 2926, 1670, 1594, 1491, 1450, 1373, 1286, 1153, 1074, 1026, 910, 757, 698; **m/z** (EI) 391 ($[M]^+$, 21%), 211 ($[Ph_2N(CO)CH_3]^+$, 44%), 91 ($[PhCH_2]^{\bullet+}$, 100%).

***N*,*N*,4-triphenyl-4-(o-tolyl)butanamide (7bc)**



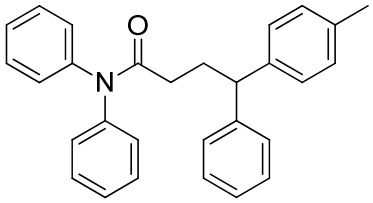
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 82%; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.51–6.94 (m, 17H), 4.16 (t, *J* = 7.7 Hz, 1H), 2.41–2.31 (m, 2H), 2.30–2.22 (m, 2H), 2.21 (s, 3H); **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 172.8, 143.9, 142.8, 141.8, 136.5, 130.5, 129.2 (br), 128.3, 128.2, 128.2 (br), 126.6, 126.2 (br), 126.1, 126.0, 126.0, 45.8, 33.6, 31.5, 19.8; **FT-IR** ν_{max}(ATR) cm^{-1}: 3043, 2934, 1952, 1669, 1597, 1465, 1369, 1163, 1022, 907, 744; **m/z** (EI) 405 ($[M]^{\bullet+}$, 19%), 211 ($[Ph_2N(CO)CH_3]^+$, 100%).

***N*,*N*,4-triphenyl-4-(m-tolyl)butanamide (7bd)**



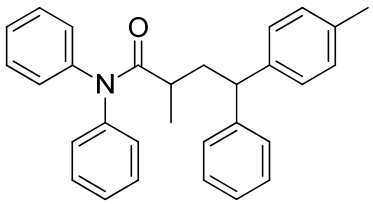
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 74%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ δ 7.38–6.92 (m, 19H), 3.92 (t, *J* = 8.0 Hz, 1H), 2.40 (m, 2H), 2.29 (s, 3H), 2.23 (t, *J* = 7.4 Hz, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.8, 144.3, 144.2, 142.8, 137.9, 129.5 (br), 129.0 (br), 128.7, 128.4, 128.3, 127.9, 126.9, 126.4 (br), 126.1, 124.8, 50.2, 33.6, 31.2, 21.5; **FT-IR** ν_{max}(ATR) cm^{-1}: 3030, 2925, 1671, 1594, 1490, 1450, 1376, 1276, 1157, 1027, 759, 700; **m/z** (EI) 405 ($[M]^{\bullet+}$, 74%), 211 ($[Ph_2N(CO)CH_3]^+$, 100%).

***N*,*N*,4-triphenyl-4-(p-tolyl)butanamide (7be)**



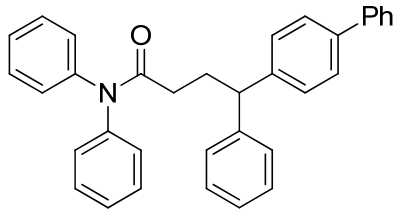
The title compound was prepared to according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 85%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.55–7.28 (m, 19H), 4.14 (t, *J* = 7.8 Hz, 1H), 2.60 (dt, *J* = 7.8, 7.2 Hz, 2H), 2.51 (s, 3H), 2.44 (t, *J* = 7.2 Hz, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.8, 144.5, 142.8, 141.2, 135.6, 129.4 (br), 129.1, 128.9 (br), 128.4, 127.8, 127.7, 126.4 (br), 126.1, 49.8, 33.6, 31.3, 21.0 **FT-IR** ν_{max}(ATR) cm^{-1}: 3030, 2927, 1670, 1593, 1494, 1449, 1375, 1275, 1155, 1075, 1026, 810, 759, 702; **m/z** (EI) 405 ($[M]^{\bullet+}$, 47%), 211 ($[Ph_2N(CO)CH_3]^+$, 100%).

2-methyl-*N*,*N*,4-triphenyl-4-(p-tolyl)butanamide (7ae)



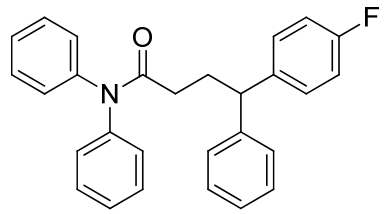
The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:DCM:EA 30:3:1) to give a 1:1 mixture of two diastereomers. **Yield** 73%; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.38 – 6.94 (m, 17H), 6.86 – 6.67 (s, 2H), 4.07 (dd, *J* = 9.4, 6.8 Hz, 1H), 2.64 – 2.53 (m, 1H), 2.53 – 2.42 (m, 1H), 2.35 (s, 1.5H), 2.26 (s, 1.5H), 2.12 – 2.01 (m, 1H), 1.12 (d, J = 6.7 Hz, 3H); **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 176.39, 145.10, 143.93, 142.90, 142.36, 141.95, 140.67, 135.77, 135.43, 129.34, 129.31, 129.04, 128.82, 128.66, 128.52, 128.33, 128.14, 128.07, 127.58, 127.48, 127.30, 126.48, 126.23, 125.94, 125.91, 48.19, 48.16, 40.01, 40.00, 35.54, 35.53, 21.01, 20.93, 18.31, 18.31; **FT-IR** ν_{max}(ATR) cm^{-1}: 2923, 2859, 1669 (C=O), 1590, 1453, 1385, 1260, 1159, 1031, 876, 699; **m/z** (EI) 419 ($[M+H]^+$, 5%), 225 ($[Ph_2N(CO)CH_2CH_3]^+$, 100%).

4-([1,1'-biphenyl]-4-yl)-*N*,*N*,4-triphenylbutanamide (7bf)



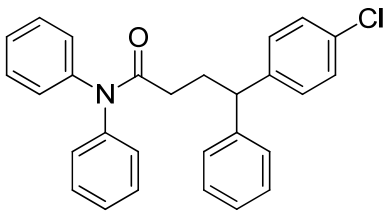
The title compound was prepared according to the general method **C** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 45:3:1). **Yield** 72%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.55 (ddd, J = 7.9, 5.0, 3.1 Hz, 2H), 7.49–7.45 (m, 2H), 7.45–7.40 (m, 2H), 7.36–6.99 (m, 18H), 4.03 (t, J = 8.0 Hz, 1H), 2.45 (m, 2H), 2.27 (t, J = 7.3 Hz, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.7, 144.2, 143.3, 142.7, 140.9, 139.1, 129.5 (br), 128.9 (br), 128.7, 128.5, 128.3, 127.9, 127.2, 127.1, 127.0, 126.3 (br), 126.3, 49.9, 33.5, 31.2; **FT-IR** ν_{max}(ATR) cm^{-1}: 3029, 2929, 1953, 1891, 1804, 1669, 1594, 1489, 1450, 1376, 1274, 1157, 1075, 1008, 908, 837, 753; **m/z** (EI) 467 ($[M]^{\bullet+}$, 17%), 211 ($[Ph_2N(CO)CH_3]^+$, 100%).

4-(4-fluorophenyl)-*N*,*N*,4-triphenylbutanamide (7bg)



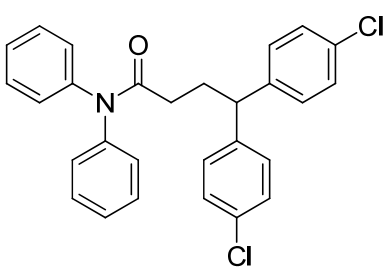
The title compound was prepared according to the general method **B** employing catalyst **1b** and 0.65 mmol of **3g**. After purification on column chromatography (Pent:DCM:EA 18:3:1) a white semi solid was obtained. **Yield** 68%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.35–7.07 (m, 17H), 6.95–6.88 (m, 2H), 3.97 (m, 1H), 2.42–2.34 (m, 2H), 2.22 (m, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.6, 161.3 (d, J_F = 244.3 Hz), 144.0, 142.7, 140.0 (d, J_F = 3.0 Hz), 129.5 (br), 129.2 (d, J_F = 7.8 Hz), 129.0 (br), 128.5, 127.8, 126.3, 126.2 (br), 115.2 (d, J_F = 21.2 Hz), 49.4, 33.4, 31.3; **^{19}F-NMR** (594 MHz, $CDCl_3$) δ –117.2; **FT-IR** ν_{max}(ATR) cm^{-1}: 3061, 2926, 1667, 1594, 1493, 1451, 1374, 1271, 1221, 1157, 1075, 1006, 907, 832, 757, 696; **m/z** (EI) 409 ($[M]^+$, 8%), 210 ($[Ph_2N(CO)CH_2]^{\bullet+}$, 44%), 168 ($[Ph_2N]^{\bullet+}$, 100%).

4-(4-chlorophenyl)-*N*,*N*,4-triphenylbutanamide (7bh)



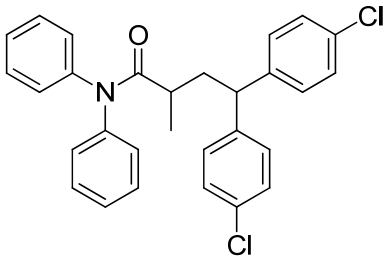
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 18:3:1 to 15:3:1). **Yield** 68%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 8.10–6.62 (m, 19H), 3.96 (t, J = 8.0 Hz, 1H), 2.42–2.33 (m, 2H), 2.22 (t, J = 7.3 Hz, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.5, 143.7, 142.8, 142.7, 131.9, 129.6 (br), 129.2, 129.0 (br), 128.5, 128.5, 127.8, 126.4, 126.2 (br), 49.5, 33.4, 31.1; **FT-IR** ν_{max}(ATR) cm^{-1}: 3051, 2926, 1668, 1595, 1449, 1381, 1277, 1158, 1084, 824, 755, 699; **m/z** (EI) 425 ($[M]^{\bullet+}$, 17%), 210 ($[Ph_2N(CO)CH_2]^{\bullet+}$, 100%).

4,4-bis(4-chlorophenyl)-*N*,*N*-diphenylbutanamide (7bi)



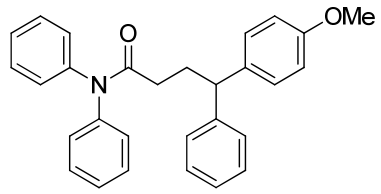
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 18:3:1 to 15:3:1). **Yield** 65%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.39–6.98 (m, 17H), 3.95 (t, *J* = 7.9 Hz, 1H), 2.33 (m, 2H), 2.20 (t, *J* = 7.2 Hz, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.3, 142.6, 142.2, 132.2, 129.6 (br), 129.1, 128.9 (br), 128.7, 126.2 (br), 48.8, 33.2, 31.0; **FT-IR** ν_{max}(ATR) cm^{-1}: 3060, 2926, 1666, 1596, 1454, 1384, 1274, 1075, 753, 699; **m/z** (EI) 460 ($[M+H]^+$, 5%), 211 ($[Ph_2N(CO)CH_3]^+$, 100%).

4,4-bis(4-chlorophenyl)-2-methyl-*N*,*N*-diphenylbutanamide (7ai)



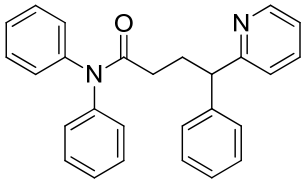
The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:DCM:EA 18:3:1 to 15:3:1). **Yield** 61%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.33–7.10 (m, 14H), 6.98 (d, *J* = 8.5 Hz, 2H), 6.77 (br s, 2H), 4.06 (dd, *J* = 9.5, 6.8 Hz, 1H), 2.56-2.49 (m, 1H), 2.43 (m, 1H), 2.01 (m, 1H), 1.14 (d, *J* = 6.8 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 176.0, 142.9, 142.7, 142.2, 141.6, 132.4, 132.0, 129.5 (br), 129.5, 128.9 128.9, 128.9 (br), 128.6, 128.4 (br), 127.6 (br), 126.4 (br), 126.1 (br), 47.3, 39.8, 35.4, 18.3; **FT-IR** ν_{max}(ATR) cm^{-1}: 3051, 2931, 2245, 1897, 1667, 1594, 1481, 1384, 1270, 1166, 1091, 1016, 912, 822, 704, 631, 521; **m/z** (EI) 474 ($[M]^{\bullet+}$, 5%), 225 ($[Ph_2N(CO)CH_2CH_3]^+$, 100%).

4-(4-methoxyphenyl)-*N*,*N*,4-triphenylbutanamide (7bk)



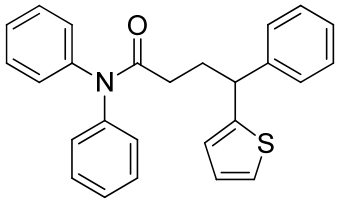
The title compound was prepared according to the general method **C** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 86%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.37–6.99 (m, 17H), 6.82–6.75 (m, 2H), 3.95–3.88 (m, 1H), 3.76 (s, 3H), 2.41–2.34 (m, 2H), 2.27–2.19 (m, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.8, 157.9, 144.6, 142.7, 136.4, 129.5 (br), 128.8, 128.6 (br), 128.4, 127.8, 126.4 (br), 126.1, 113.8, 55.2, 49.4, 33.6, 31.4; **FT-IR** ν_{max}(ATR) cm^{-1}: 3032, 2939, 1667, 1596, 1496, 1368, 1252, 1175, 1030, 909, 826, 755, 697; **m/z** (EI) 421 ($[M]^{\bullet+}$, 17%), 211 ($[Ph_2N(CO)CH_3]^+$, 74%).

***N*,*N*,4-triphenyl-4-(pyridin-2-yl)butanamide (7bl)**



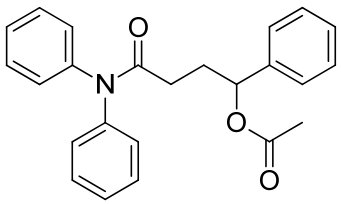
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:EA 3:1 to 1:1). **Yield** 73%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 8.50 (d, *J* = 3.9 Hz, 1H), 7.52 (td, *J* = 7.7, 1.8 Hz, 1H), 7.36–7.02 (m, 17H), 4.14 (t, *J* = 7.9 Hz, 1H), 2.57 (m, 1H), 2.46 (m, 1H), 2.27–2.18 (m, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 172.8, 163.2, 149.1, 143.2, 142.7, 136.4, 129.4 (br), 128.9 (br), 128.5, 128.0, 126.42, 126.3 (br), 122.8, 121.3, 52.4, 33.3, 30.6; **FT-IR** ν_{max}(ATR) cm^{-1}: 3050, 2931, 1956, 1885, 1667, 1589, 1453, 1377, 1278, 1157, 1015, 906, 755, 702; **m/z** (EI) 393 ($[M]^{\bullet+}$, 2%), 224 ($[Ph_2N(CO)CH_2CH_2]^{\bullet+}$, 74%).

***N*,*N*,4-triphenyl-4-(thiophen-2-yl)butanamide (7bm)**



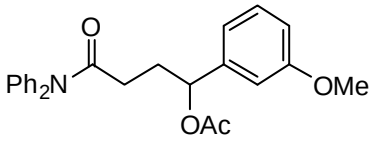
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 48%; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.34–7.05 (m, 16H), 6.87 (dd, *J* = 5.1, 3.5 Hz, 1H), 6.76 (dt, *J* = 3.5, 1.0 Hz, 1H), 4.22 (t, *J* = 7.9 Hz, 1H), 2.52–2.31 (m, 2H), 2.23 (t, *J* = 7.2 Hz, 2H); **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 172.5, 148.6, 143.8, 142.7, 129.2 (br), 128.5, 128.3 (br), 127.7, 126.7 (br), 126.6, 126.5, 123.9, 123.6, 45.7, 33.3, 32.8; **FT-IR** ν_{max}(ATR) cm^{-1}: 3059, 2932, 1735, 1667, 1593, 1490, 1370, 1272, 1139, 1072, 757, 694; **m/z** (EI) 397 ($[M]^{\bullet+}$, 20%), 211 ($[Ph_2N(CO)CH_3]^{+}$, 100%).

4-(diphenylamino)-4-oxo-1-phenylbutyl acetate (7bn)



The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:EA 4:1). **Yield** 70%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.48–7.01 (m, 16H), 5.76 (dd, *J* = 8.0, 5.2 Hz, 1H), 2.30–2.15 (m, 4H), 1.99 (s, *J* = 3.7 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 171.9, 170.2, 142.7, 140.1, 129.6 (br), 128.9 (br), 128.4, 127.9, 126.4, 126.2 (br), 75.0, 31.9, 31.3, 21.2; **FT-IR** ν_{max}(ATR) cm^{-1}:, 2933, 1733, 1669, 1593, 1491, 1371, 1234, 1025, 757, 696; **m/z** (EI) 373 ($[M]^{+}$, 28%), 211 ($[Ph_2N(CO)CH_3]^{+}$, 19%), 168 ($[Ph_2N]^{\bullet+}$, 100%).

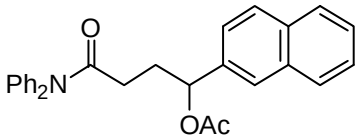
4-(diphenylamino)-1-(3-methoxyphenyl)-4-oxobutyl acetate (7bo)



The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:EA 5:1 to 3:1). **Yield** 86%; **^{1}H-NMR** (400

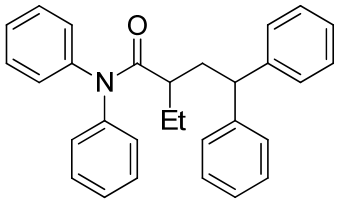
MHz, $CDCl_3$) δ 7.40–7.12 (m, 11H), 6.84 (d, *J* = 7.7 Hz, 1H), 6.79 (dd, *J* = 7.7, 1.3 Hz, 2H), 5.73 (dd, *J* = 7.7, 5.2 Hz, 1H), 3.77 (s, 3H), 2.29–2.11 (m, 4H), 1.98 (s, 3H); **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 171.9, 170.1, 159.6, 142.7, 141.8, 129.5, 129.1 (br), 128.8 (br), 126.4 (br), 118.6, 113.3, 111.9, 74.8, 55.2, 32.0, 31.3, 21.1; **FT-IR** ν_{max}(ATR) cm^{-1}: 3061, 2933, 1736, 1672, 1594, 1491, 1453, 1376, 1239, 1161, 1036, 759, 700; **m/z** (EI) 403 ($[M+H]^+$, 6%), 211 ($[Ph_2N(CO)CH_3]^+$, 37%), 169 ($[Ph_2NH]^+$, 100%).

4-(diphenylamino)-1-(naphthalen-2-yl)-4-oxobutyl acetate (7bp)



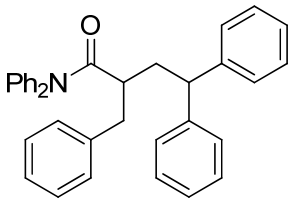
The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:EA 5:1 to 3:1). **Yield** 73%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.83–7.78 (m, 3H), 7.72 (br s, 1H), 7.51–7.45 (m, 2H), 7.41 (dd, *J* = 8.5, 1.6 Hz, 1H), 7.36–7.25 (m, 5H), 7.19 (d, *J* = 6.9 Hz, 4H), 5.94 (m, 1H), 2.35–2.26 (m, 4H), 2.03 (s, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 171.9, 170.2, 142.6, 137.4, 133.1, 129.7 (br), 128.9 (br), 128.6 (br), 128.3, 128.0, 127.6, 126.4 (br), 126.2, 126.1, 125.6, 124.2, 75.2, 31.8, 31.3, 21.2; **FT-IR** ν_{max}(ATR) cm^{-1}: 3058, 2930, 1735, 1668, 1593, 1491, 1450, 1372, 1233, 1026, 948, 903, 858, 820, 752, 697; **m/z** (EI) 423 ($[M]^{\bullet+}$, 6%), 211 ($[Ph_2N(CO)CH_3]^+$,74%), 169 ($[Ph_2NH]^+$, 100%).

2-ethyl-*N*,*N*,4,4-tetraphenylbutanamide (7ca)



The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:DCM:EA 30:3:1). **Yield** 51%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.42–7.00 (m, 18H), 6.75 (br s, 2H), 4.11 (dd, *J* = 9.4, 6.5 Hz, 1H), 2.49 (m, 2H), 2.22–2.16 (m, 1H), 1.70–1.62 (m, 1H), 1.53 (tt, *J* = 13.9, 6.9 Hz, 1H), 0.85 (t, *J* = 7.4 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 175.6, 145.1, 143.7, 143.0 (br), 142.3 (br), 129.3 (br), 128.8 (br), 128.7 (br), 128.7, 128.4, 128.2, 127.7, 127.3 (br), 126.5 (br), 126.3, 126.0, 125.9 (br), 48.6, 42.0, 37.5, 26.0, 11.6; **FT-IR** ν_{max}(ATR) cm^{-1}: 3032, 2925, 1951, 1666, 1592, 1450, 1384, 1267, 1159, 1073, 1028753, 700; **m/z** (EI) 419 ($[M]^{\bullet+}$, 6%), 211 ($[Ph_2N(CO)C_3H_7]^{\bullet+}$, 81%), 168 ($[Ph_2N]^{\bullet+}$, 100%).

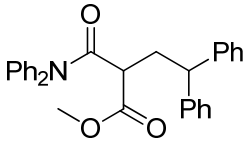
2-benzyl-*N*,*N*,4,4-tetraphenylbutanamide (7da)



The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:EA 15:1 to 10:1). **Yield** 72%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.36–7.11 (m, 16H), 7.10–7.03 (m, 5H), 6.80–6.77 (m, 2H), 6.46 (d, *J* = 7.5 Hz,

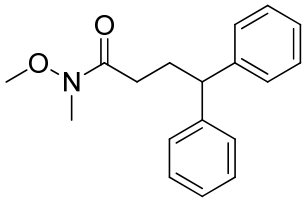
2H), 4.12 (dd, *J* = 9.1, 7.2 Hz, 1H), 3.07–2.99 (m, 1H), 2.85–2.79 (m, 1H), 2.71 (dd, *J* = 13.0, 7.5 Hz, 1H), 2.53-2.47 (m, 1H), 2.22-2.17 (m, 1H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 174.9, 144.8, 143.4, 142.8 (br), 142.0 (br), 139.3, 129.3, 129.2, 128.8, 128.6 (br), 128.6, 128.4, 128.3, 128.3, 127.8, 127.4, 126.5, 126.4, 126.3, 126.1, 126.1 (br), 48.5, 43.3, 38.8, 37.4; **FT-IR** ν_{max}(ATR) cm^{-1}: 3059, 2926, 1667, 1592, 1490, 1448, 1383, 1290, 1262, 1157, 1075, 752, 696; **m/z** (EI) 482 ($[M]^{\bullet+}$, 2%), 301 ($[Ph_2N(CO)(CH_2)_2C_6H_6]^+$, 100%), 181 ($[Ph_2CHCH_2]^{\bullet+}$, 20%).

methyl 2-(diphenylcarbamoyl)-4,4-diphenylbutanoate (7ea)



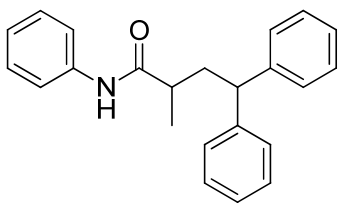
The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:EA 15:1 to 10:1). **Yield** 58%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.41–7.06 (m, 18H), 6.80 (br s, 2H), 4.18 (dd, *J* = 9.3, 7.1 Hz, 1H), 3.67 (s, 3H), 3.54 (dd, *J* = 8.5, 6.1 Hz, 1H), 2.69 (m, 2H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 170.5, 168.6, 144.1, 142.8, 142.5 (br), 141.8 (br), 129.5 (br), 128.9 (br), 128.8, 128.5 (br), 128.5 (br), 128.1, 127.9 (br), 127.7, 126.6, 126.3 (br), 126.3(br), 126.3, 52.4, 48.4, 48.4, 35.1; **FT-IR** ν_{max}(ATR) cm^{-1}: 3040, 2940, 1959, 1737, 1674, 1595, 1448, 1368, 1273, 1019, 911, 704; **m/z** (EI) 450 ($[M+H]^+$, 2%), 269 ($[Ph_2N(CO)CH_2COOMe]^+$, 100%).

***N*-methoxy-*N*-methyl-4,4-diphenylbutanamide (7fa)**



The title compound was prepared according to the general method **B** employing catalyst **1b** and purified by flash column chromatography (Pent:EA 10:1 to 8:1). **Yield** 73%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.30–7.12 (m, 10H), 3.97 (dd, *J* = 8.9, 6.1 Hz, 1H), 3.48 (s, 3H), 3.12 (s, 3H), 2.46-2.30 (m, 4H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 174.2 (br), 144.4, 128.4, 127.9, 126.2, 61.0, 50.5, 32.1 (br), 30.4 (br), 30.2; **FT-IR** ν_{max}(ATR) cm^{-1}: 3026, 2938, 1662, 1449, 1178, 1108, 996, 749, 703; **m/z** (EI) 284 ($[M+H]^+$, 14%), 164 ($[Me(OMe)N(CO)(CH_2)_3C_6H_6+H]^+$, 100%).

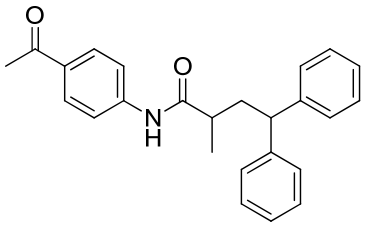
2-methyl-*N*,4,4-triphenylbutanamide (7ga)



The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:DCM:EA 20:3:1). **Yield** 77%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.6 Hz, 2H), 7.36–7.29 (m, 4H), 7.29–7.20 (m, 7H), 7.15 (m, 1H), 7.13–7.08 (m, 1H), 6.81 (s, 1H), 4.00 (dd, *J* = 9.9, 6.3 Hz, 1H), 2.53–2.47 (m, 1H), 2.18 (m, 2H), 1.26 (d, *J* = 6.6 Hz, 3H); **^{13}C-NMR** (151 MHz, $CDCl_3$) δ 174.2, 144.5, 143.7, 137.8, 1298.0, 128.7, 128.5, 128.1, 127.6, 126.6, 126.3, 124.2, 119.6, 49.0, 40.6, 40.1,

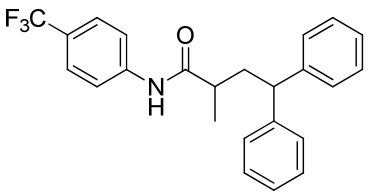
18.5; **FT-IR** ν_{max}(ATR) cm^{-1}: 3301, 2927, 2322, 2015, 1740, 1663, 1600, 1537, 1493, 1437, 1370, 1306, 1226, 1068, 902, 752; **m/z** (EI) 329 ($[M]^{\bullet+}$, 100%).

***N*-(4-acetylphenyl)-2-methyl-4,4-diphenylbutanamide (7ha)**



The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:EA 3:1). **Yield** 83%; **^{1}H-NMR** (600 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.6 Hz, 2H), 7.57 (d, *J* = 8.6 Hz, 2H), 7.34–7.30 (m, 2H), 7.27–7.20 (m, 6H), 7.18 (br s, 1H), 7.16–7.12 (m, 1H), 3.98 (dd, *J* = 9.4, 6.6 Hz, 1H), 2.56 (s, 3H), 2.54–2.48 (m, 1H), 2.28–2.18 (m, 1H), 1.26 (d, *J* = 6.6 Hz, 3H); **^{13}C-NMR (151 MHz, $CDCl_3$)** δ 196.9, 174.7, 144.3, 143.6, 142.2, 132.8, 129.7, 128.8, 128.5, 128.0, 127.6, 126.7, 126.4, 118.8, 49.0, 40.7, 40.0, 26.4, 18.4; **FT-IR** ν_{max}(ATR) cm^{-1}: 3319, 2944, 2246, 1947, 1688, 1251, 1170, 1033, 923, 836, 727; **m/z** (EI) 372 ($[M+H]^+$, 22%), 191 ($[AcPhNH(CO)CH_2CH_3]^+$, 100%).

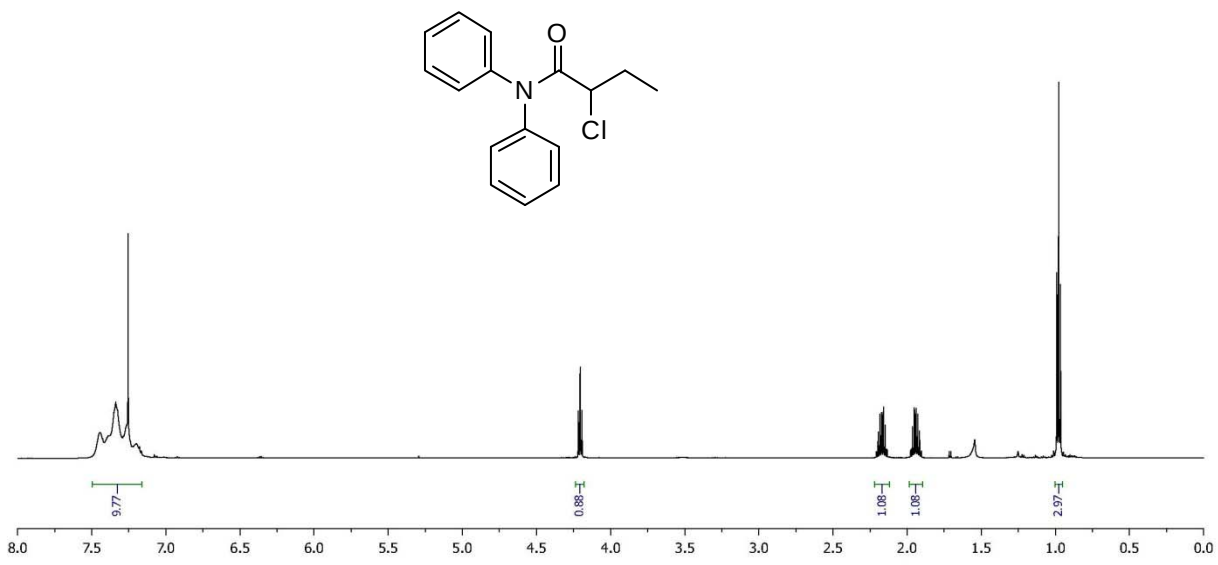
2-methyl-4,4-diphenyl-*N*-(4-(trifluoromethyl)phenyl)butanamide (7ia)

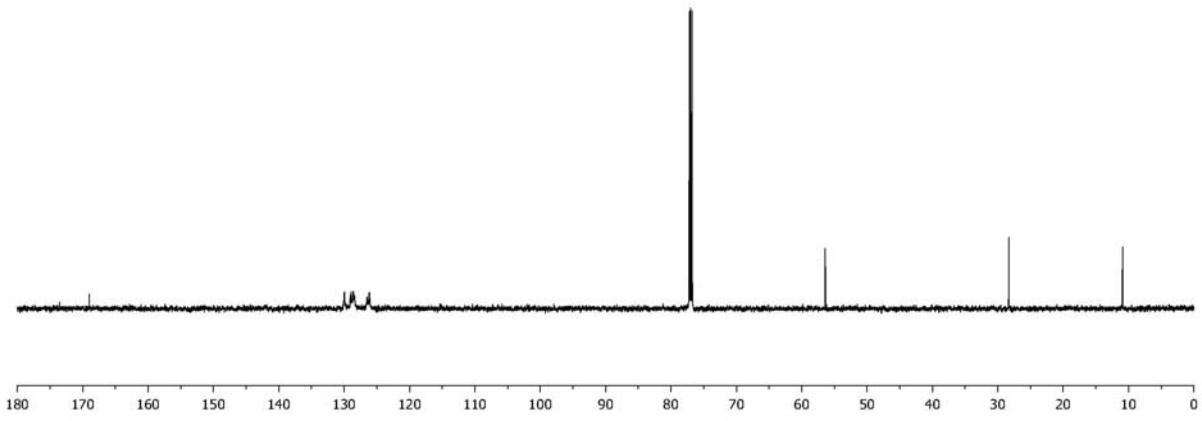


The title compound was prepared according to the general method **B** employing catalyst **1a** and purified by flash column chromatography (Pent:EA 6:1). **Yield** 79%; **^{1}H-NMR** (400 MHz, $CDCl_3$) δ 7.59–7.53 (m, 4H), 7.35–7.30 (m, 2H), 7.27–7.19 (m, 6H), 7.17–7.12 (m, 1H), 6.95 (s, 1H), 3.97 (dd, J_F = 9.5, 6.4 Hz, 1H), 2.49 (m, 1H), 2.27–2.15 (m, 2H), 1.26 (d, J_F = 6.5 Hz, 3H); **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 174.6, 144.3, 143.5, 140.8, 128.8, 128.5, 128.0, 127.6, 126.7, 126.4, 126.2 (q, J_F = 3.7 Hz), 124.0 (q, J_F = 271 Hz), 119.2, 49.0, 40.7, 40.0, 18.4; **^{19}F NMR** (376 MHz) δ 62.1; **FT-IR** ν_{max}(ATR) cm^{-1}: 3245, 1665, 1602, 1534, 1405, 1319, 1256, 1162, 1109, 1062, 1016, 832, 695; **m/z** (EI) 397 ($[M+H]^{\bullet+}$, 24%), 216 ($[CF_3PhNH(CO)CH_2CH_2]^{\bullet+}$, 100%).

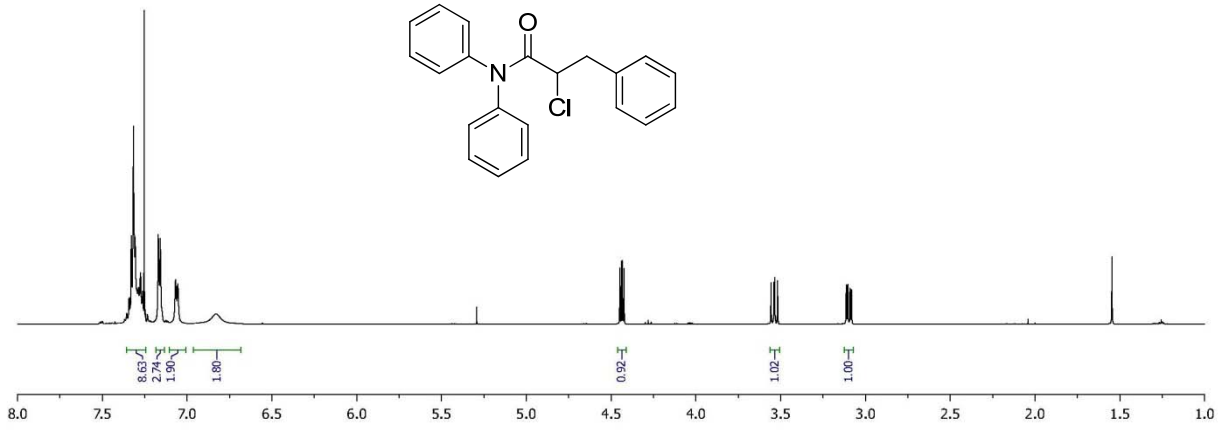
5. NMR-Spectra

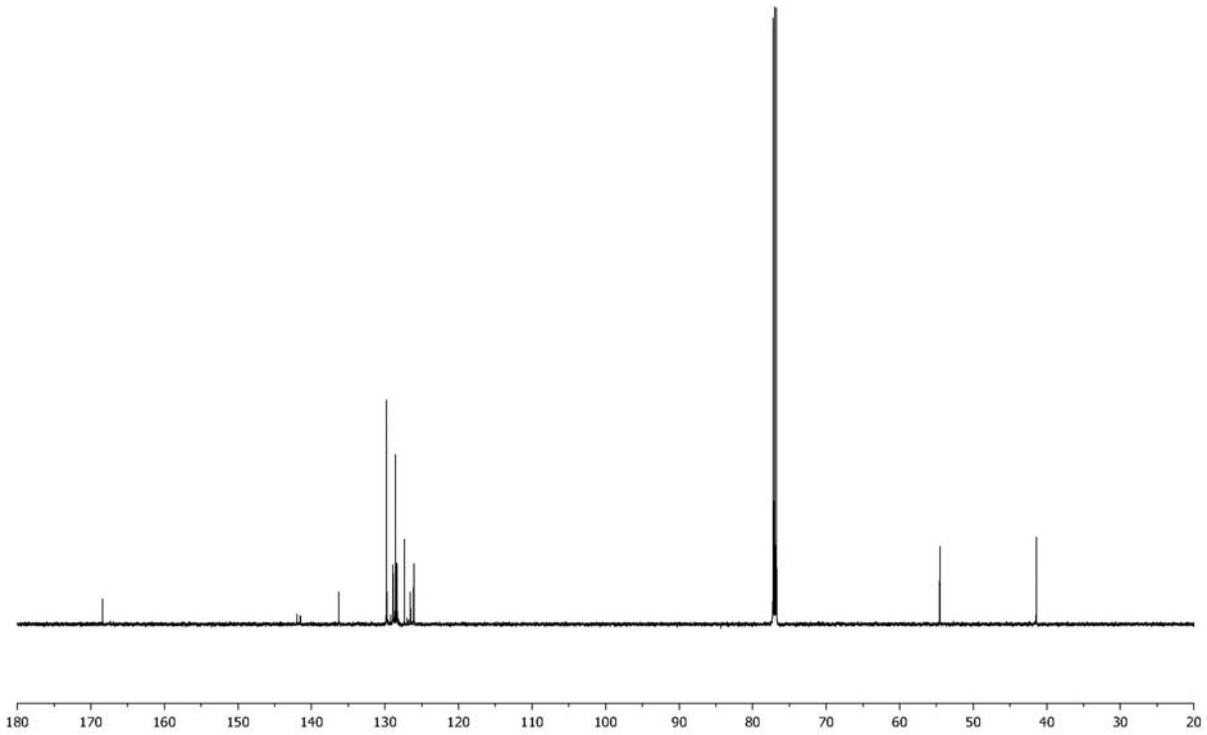
2-chloro-*N*,*N*-diphenylbutanamide (6c)



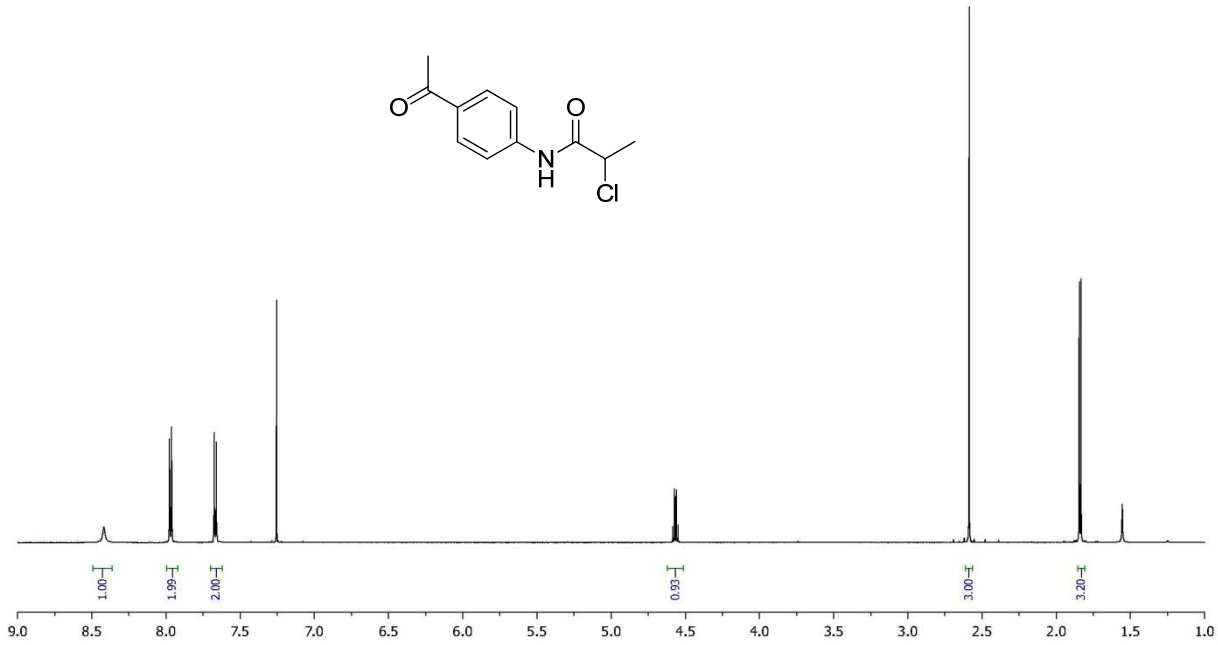


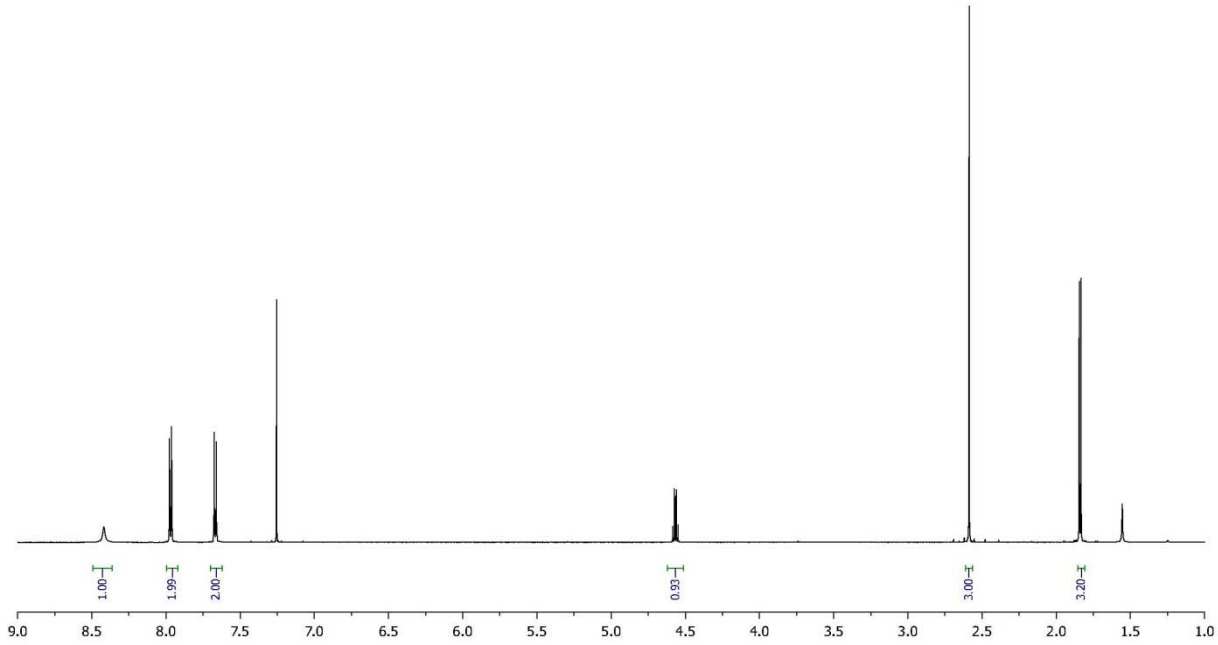
2-chloro-*N*,*N*,3-triphenylpropanamide (6d)



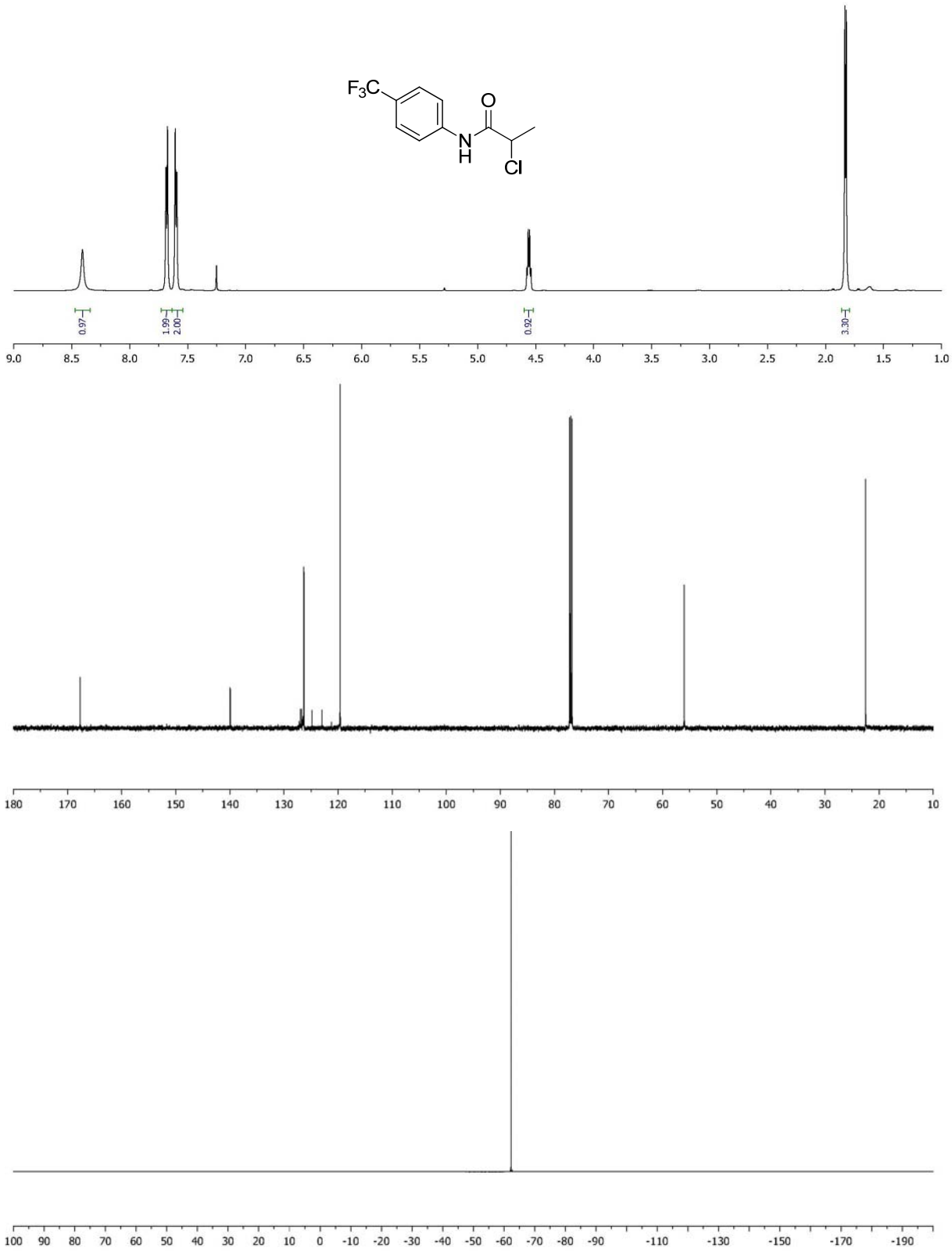


***N*-(4-acetylphenyl)-2-chloropropanamide (6h)**

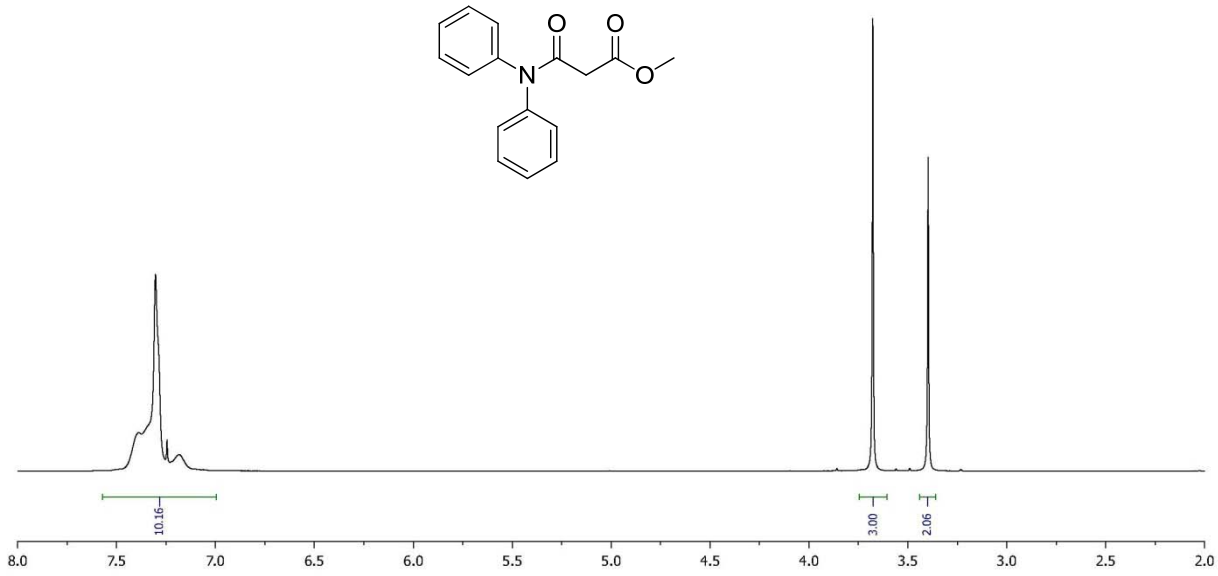


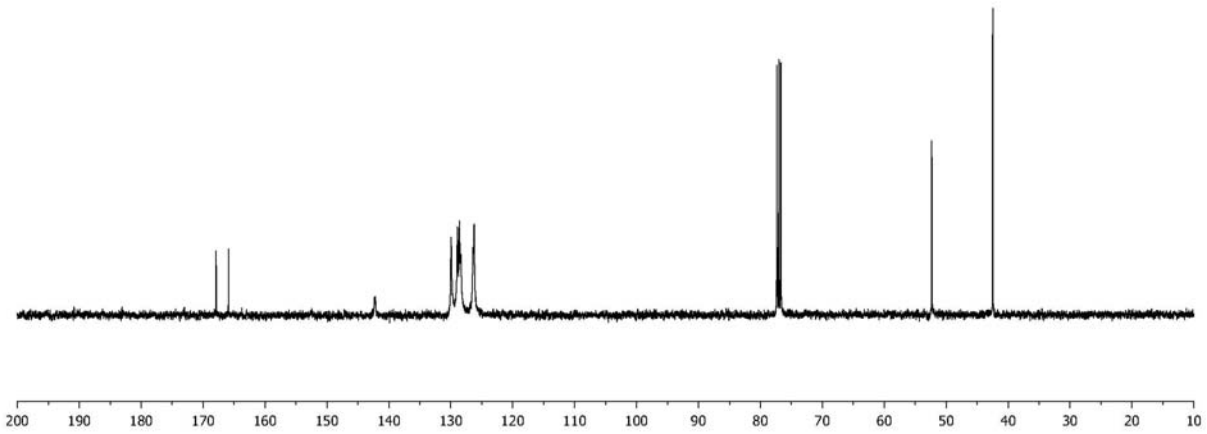


2-chloro-*N*-(4-(trifluoromethyl)phenyl)propanamide (6i)

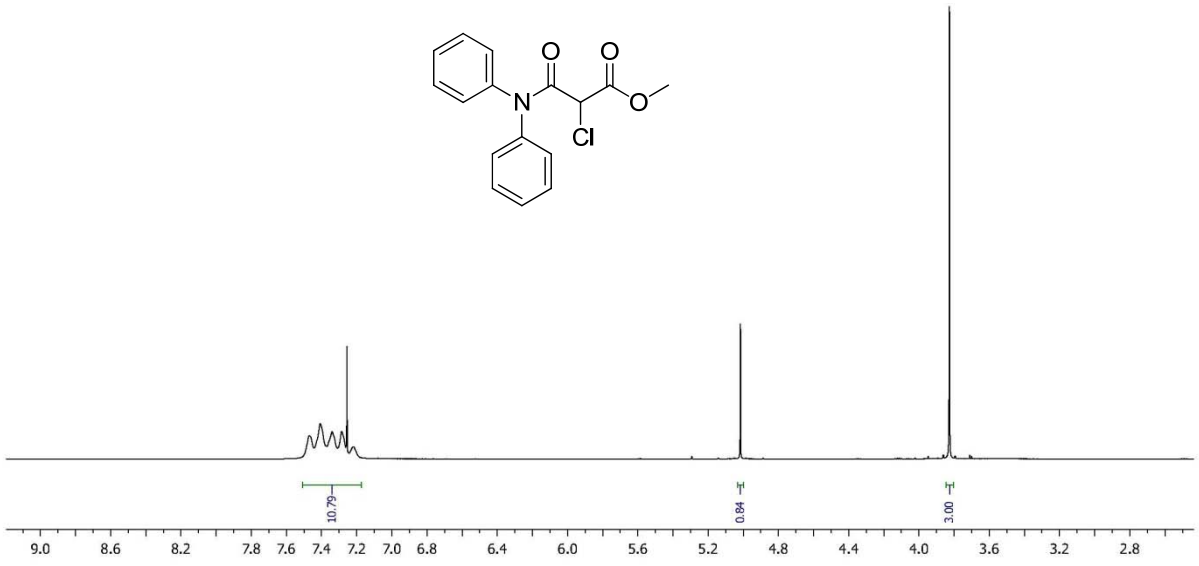


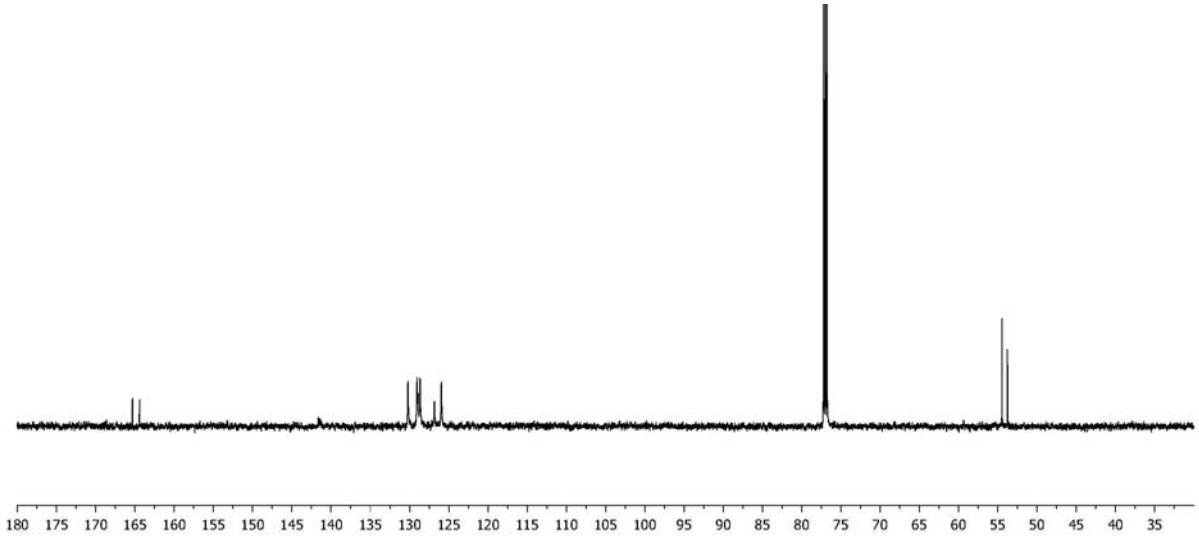
methyl 3-(diphenylamino)-3-oxopropanoate (9)



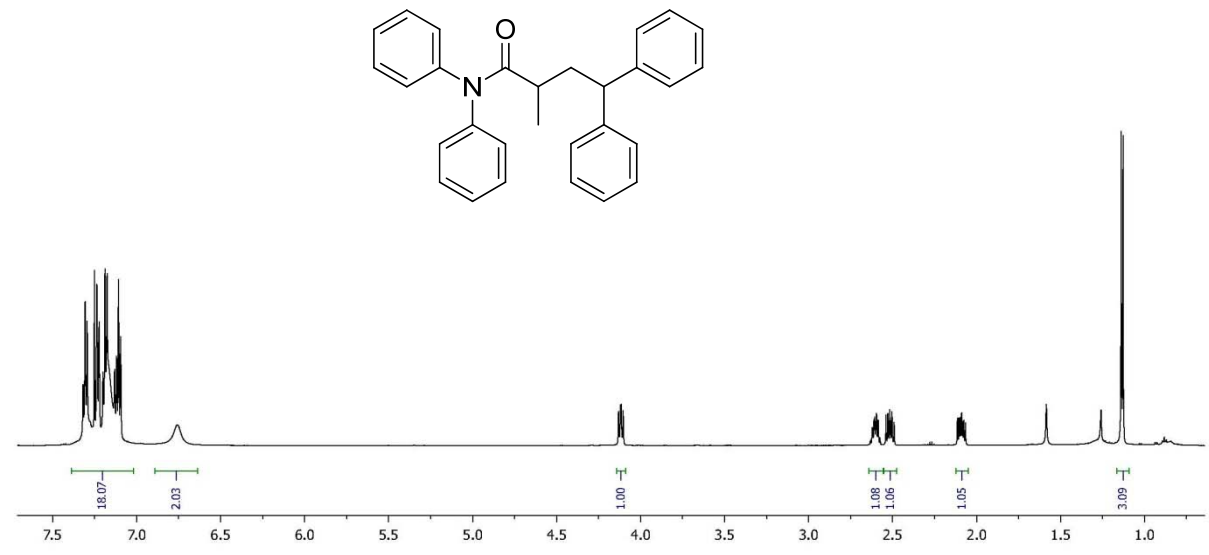


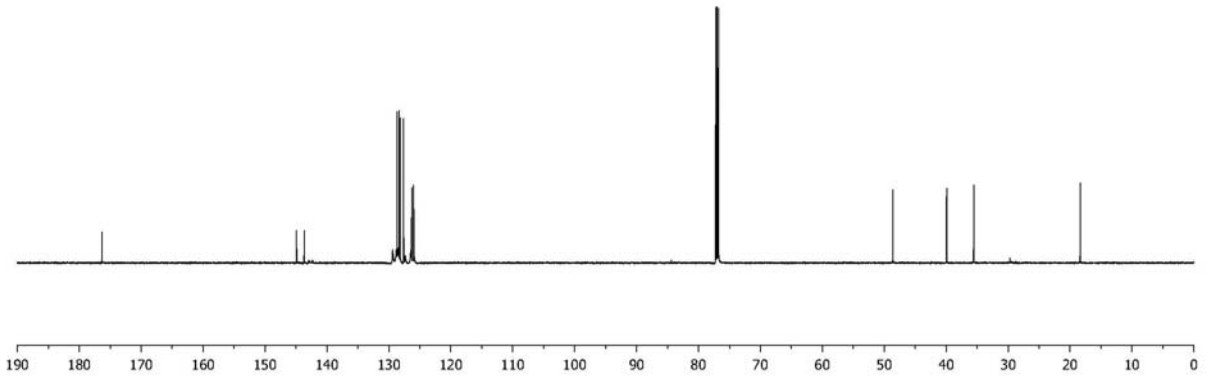
methyl 2-chloro-3-(diphenylamino)-3-oxopropanoate (6e)



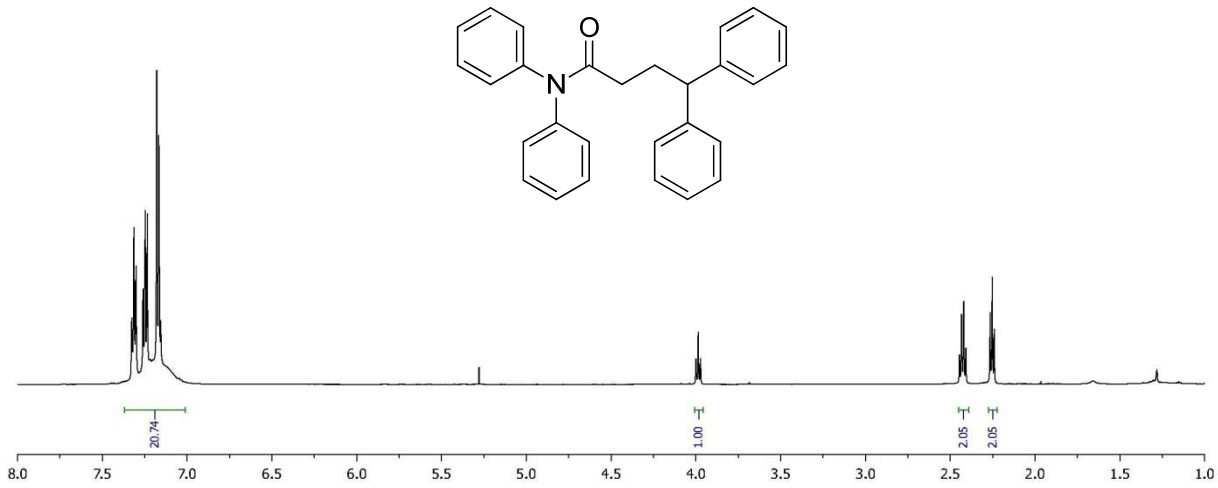


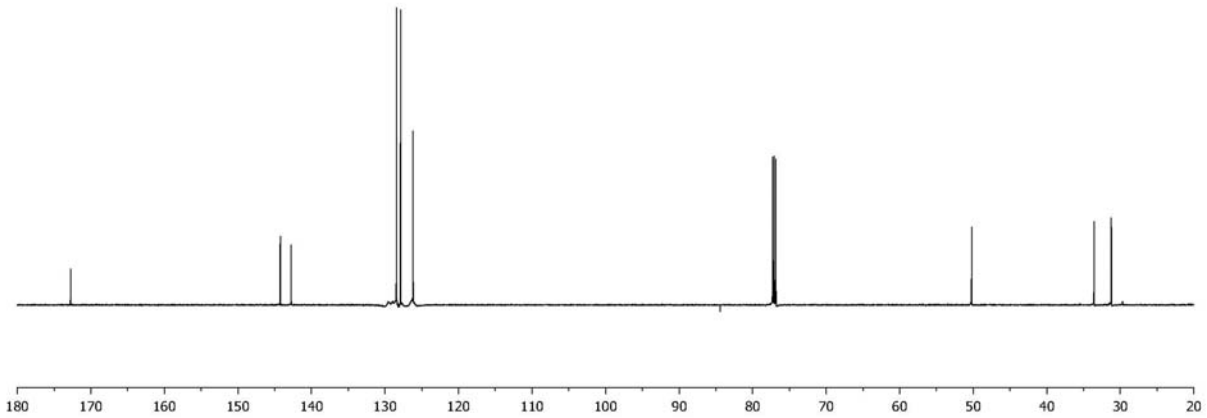
2-methyl-*N*,*N*,4,4-tetraphenylbutanamide (7aa)



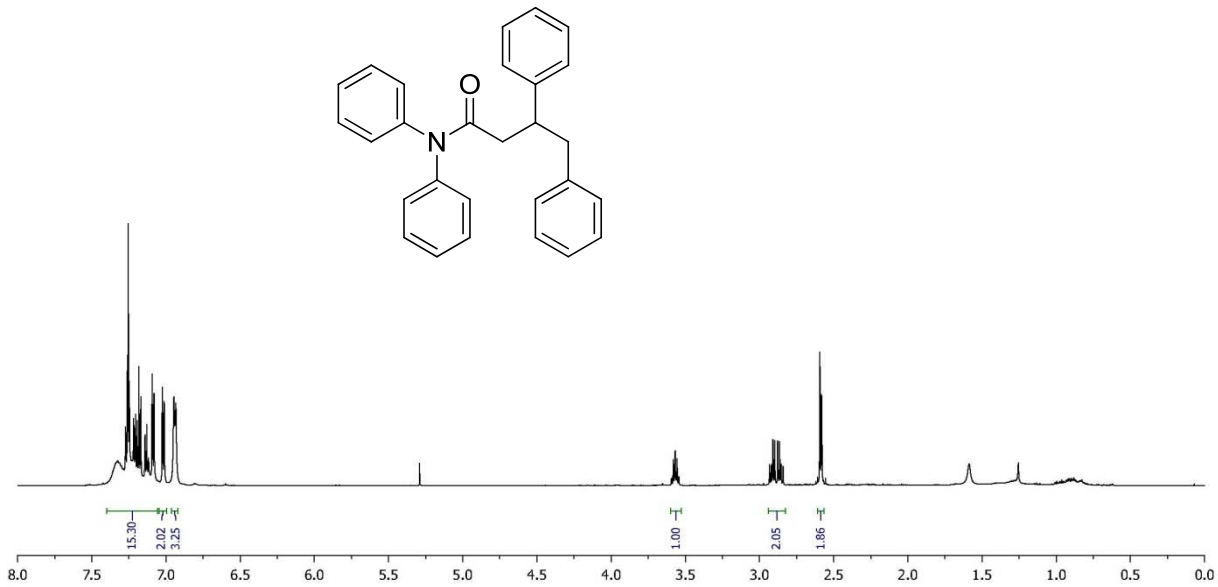


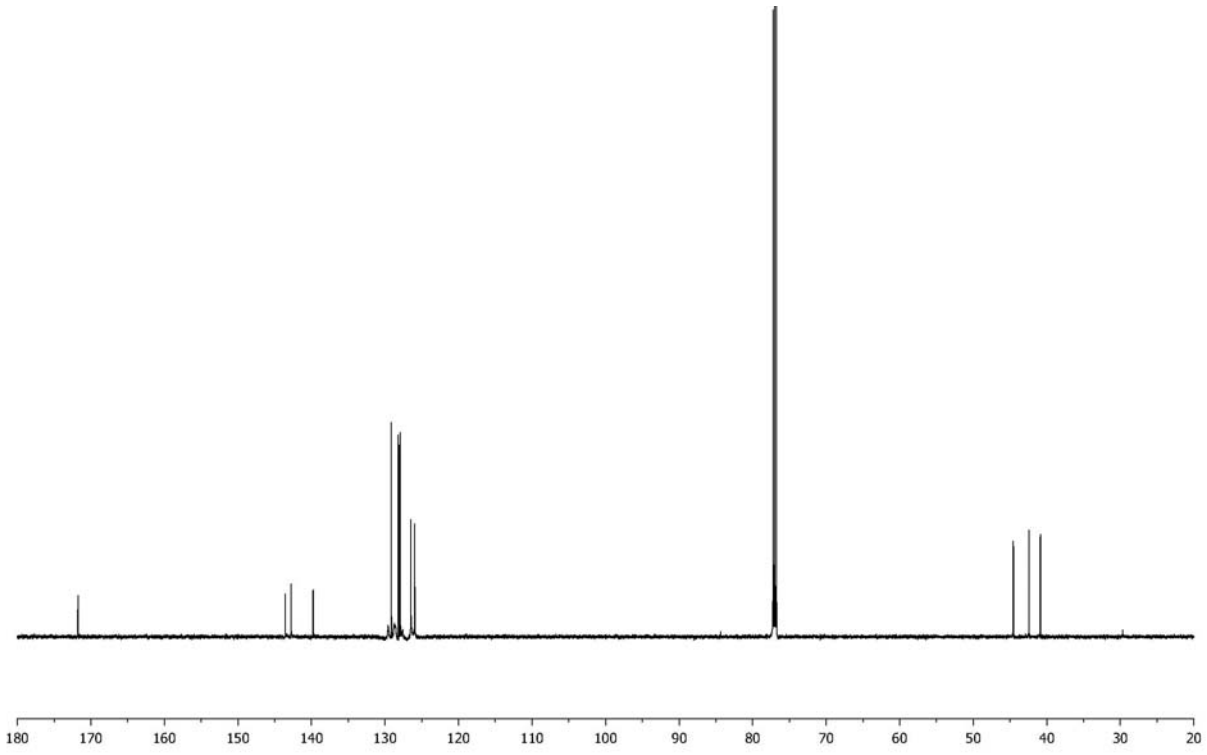
***N*,*N*,4,4-tetraphenylbutanamide (7ba)**



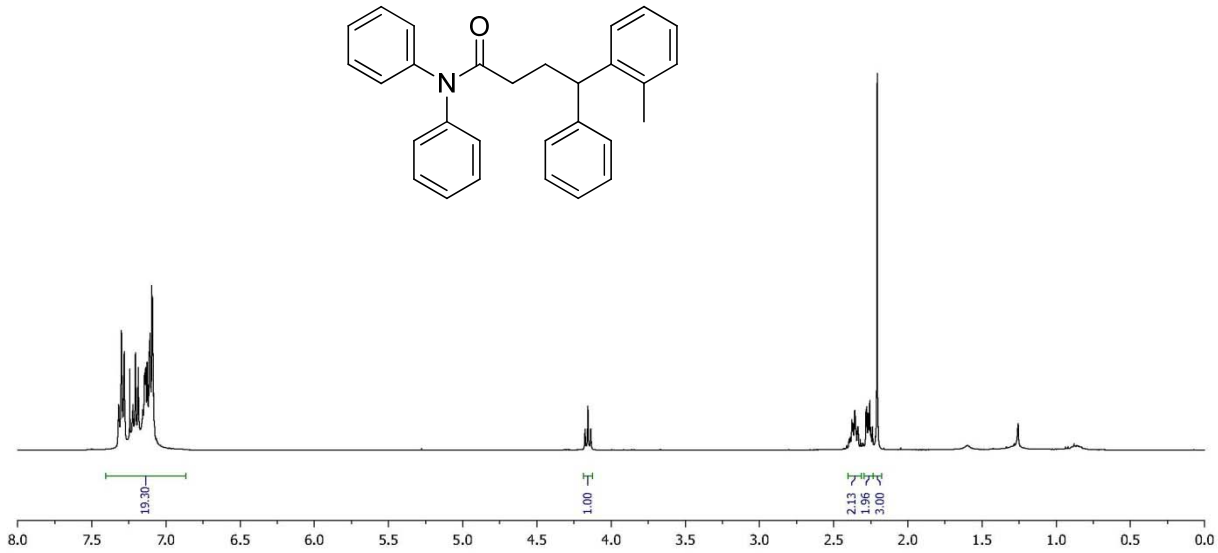


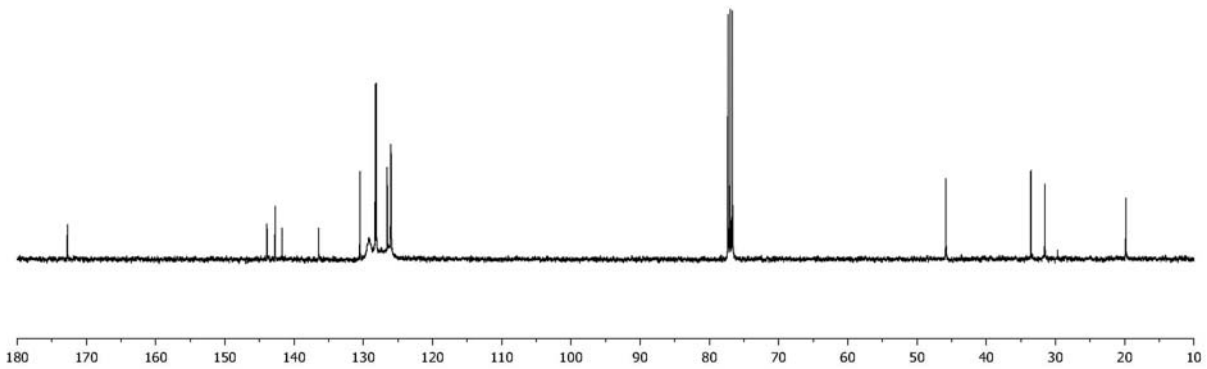
***N*,*N*,3,4-tetraphenylbutanamide (7bb)**



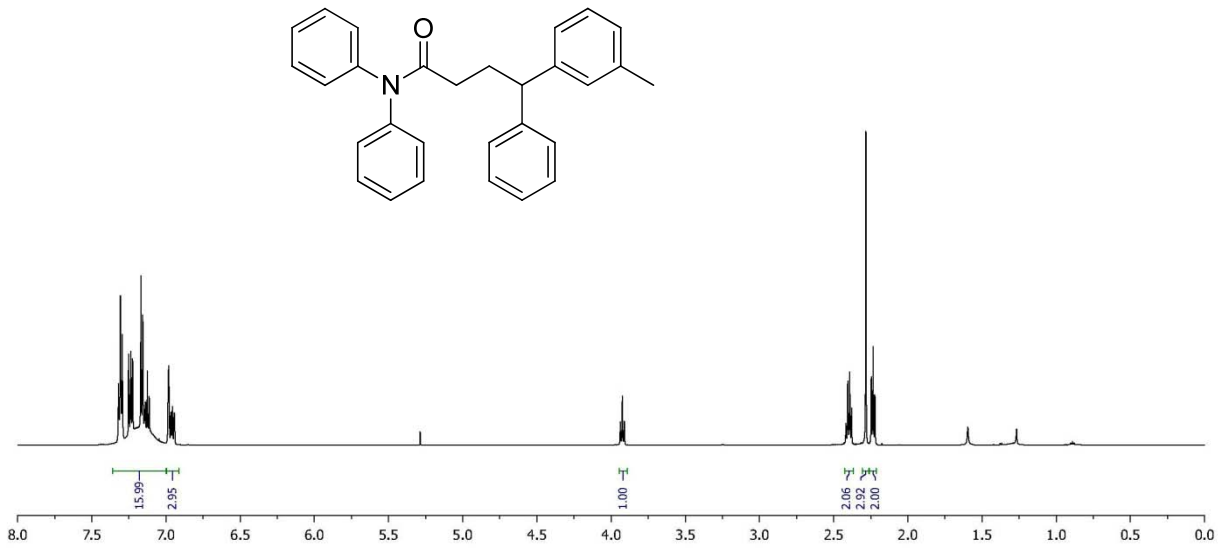


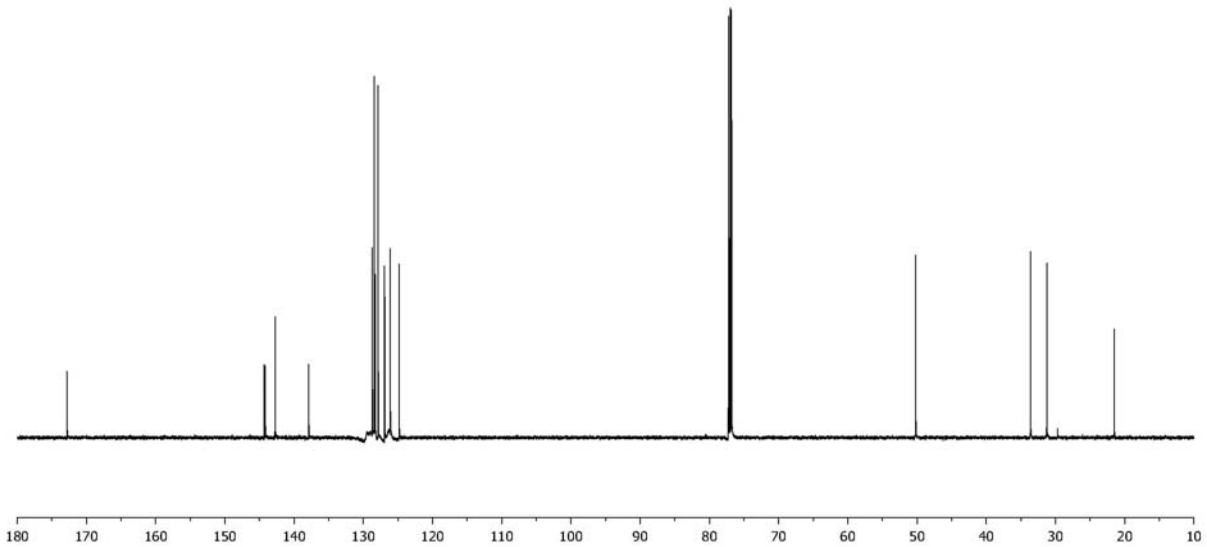
***N*,*N*,4-triphenyl-4-(*o*-tolyl)butanamide (7bc)**



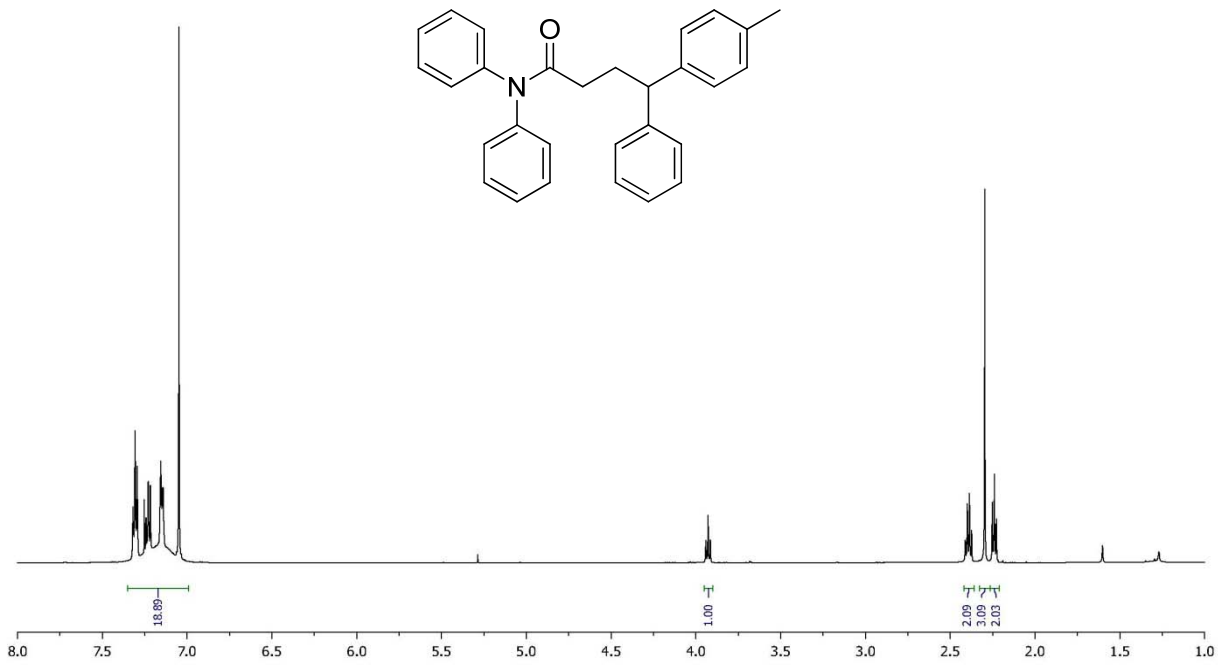


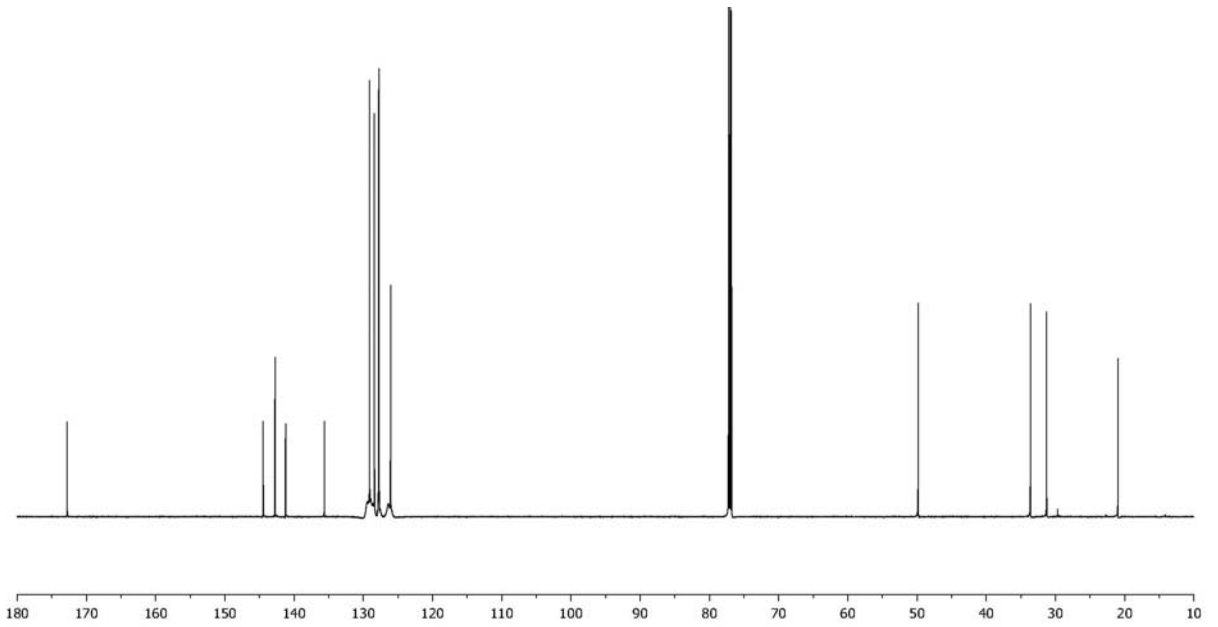
***N*,*N*,4-triphenyl-4-(*m*-tolyl)butanamide (7bd)**



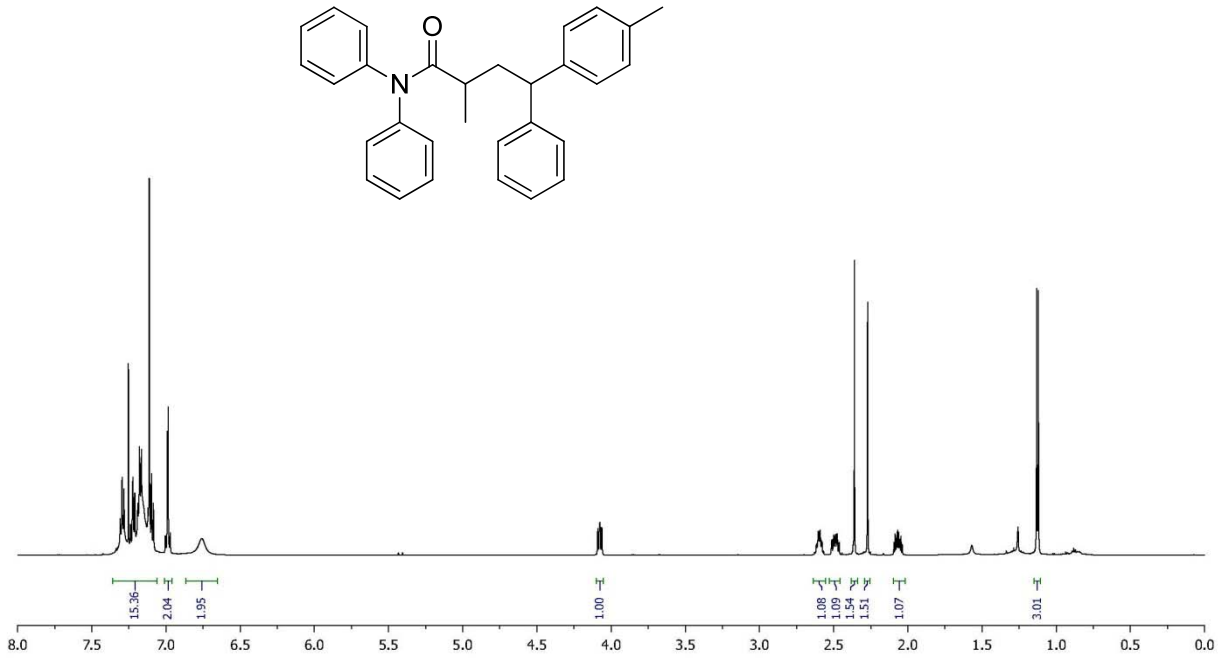


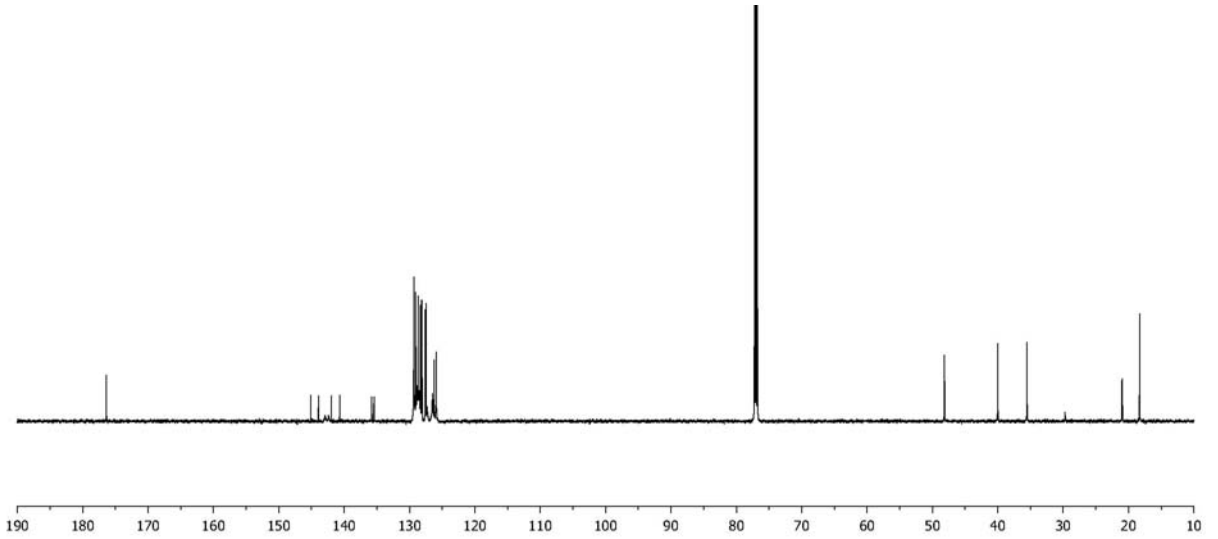
***N*,*N*,4-triphenyl-4-(*p*-tolyl)butanamide (7be)**



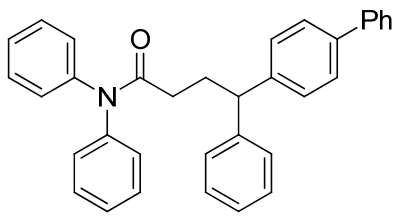


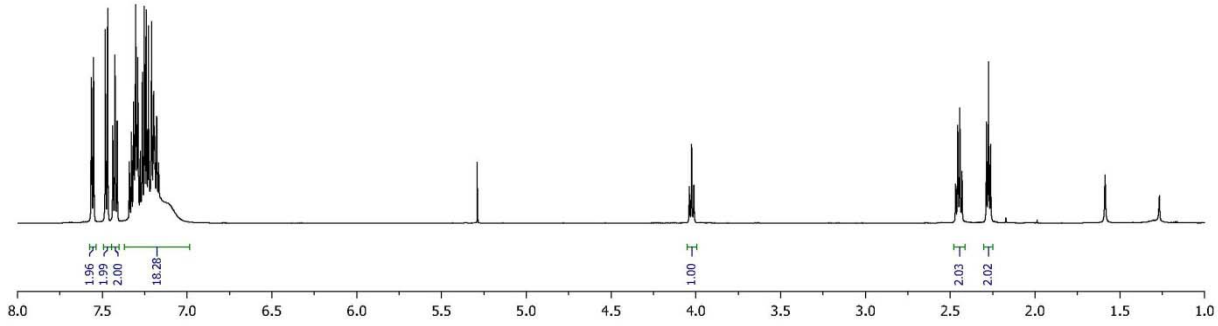
2-methyl-*N*,*N*,4-triphenyl-4-(*p*-tolyl)butanamide (7ae)

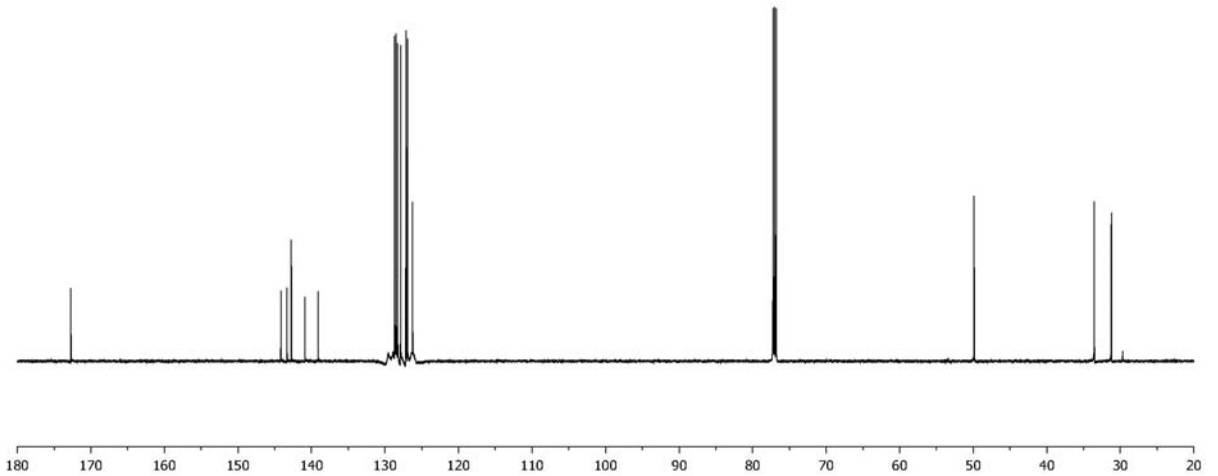




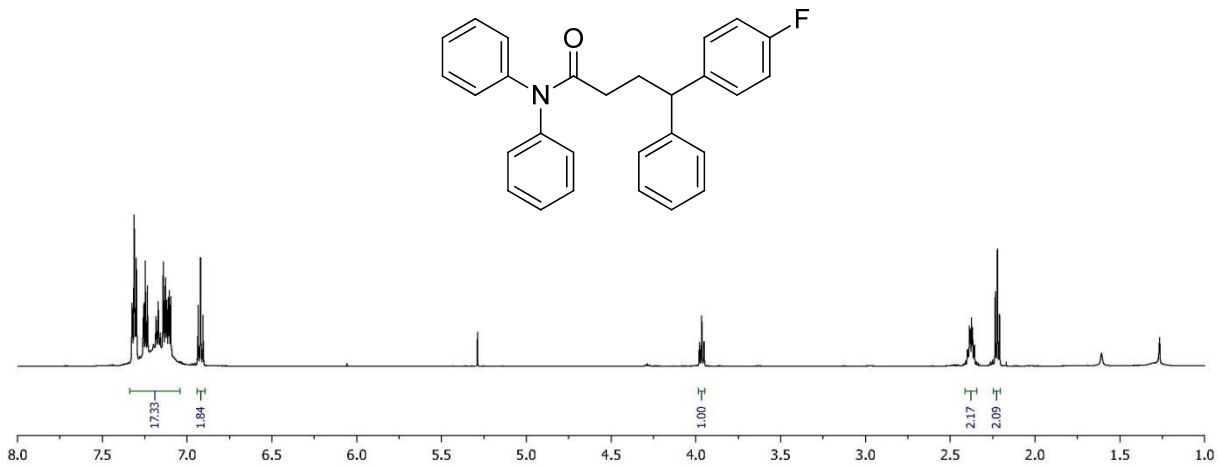
4-([1,1'-biphenyl]-4-yl)-*N*,*N*,4-triphenylbutanamide (7bf)

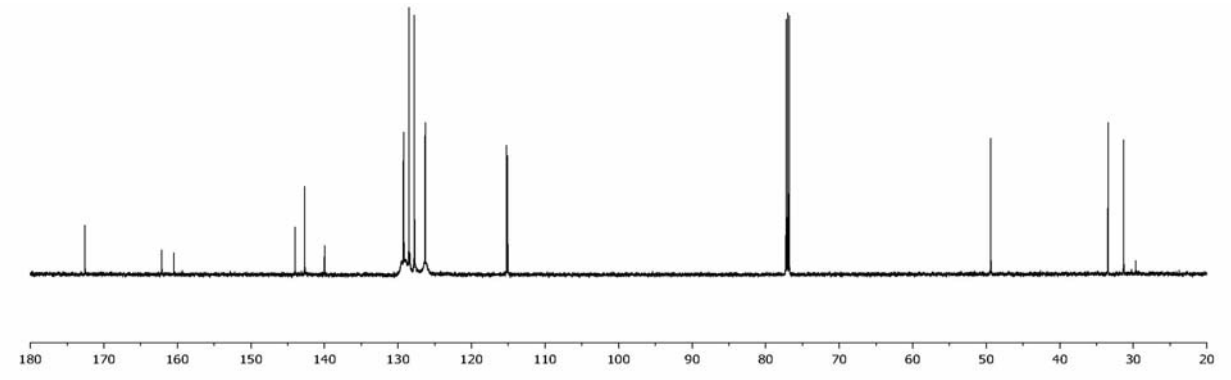


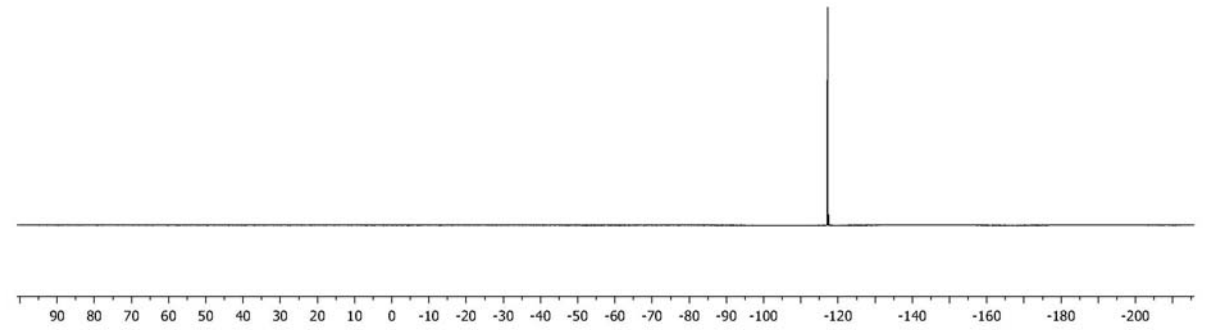




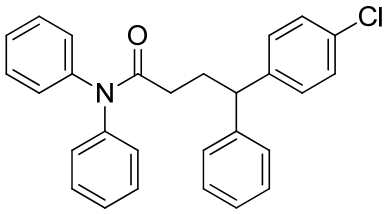
4-(4-fluorophenyl)-*N*,*N*,4-triphenylbutanamide (7bg)

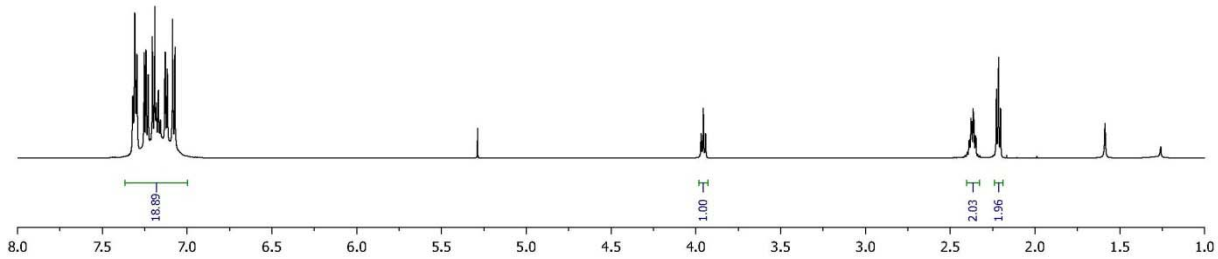


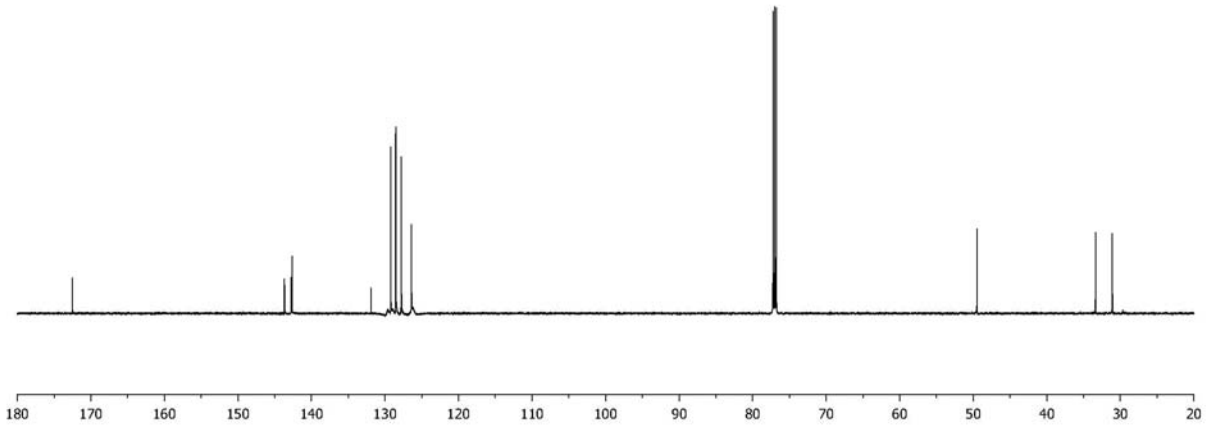




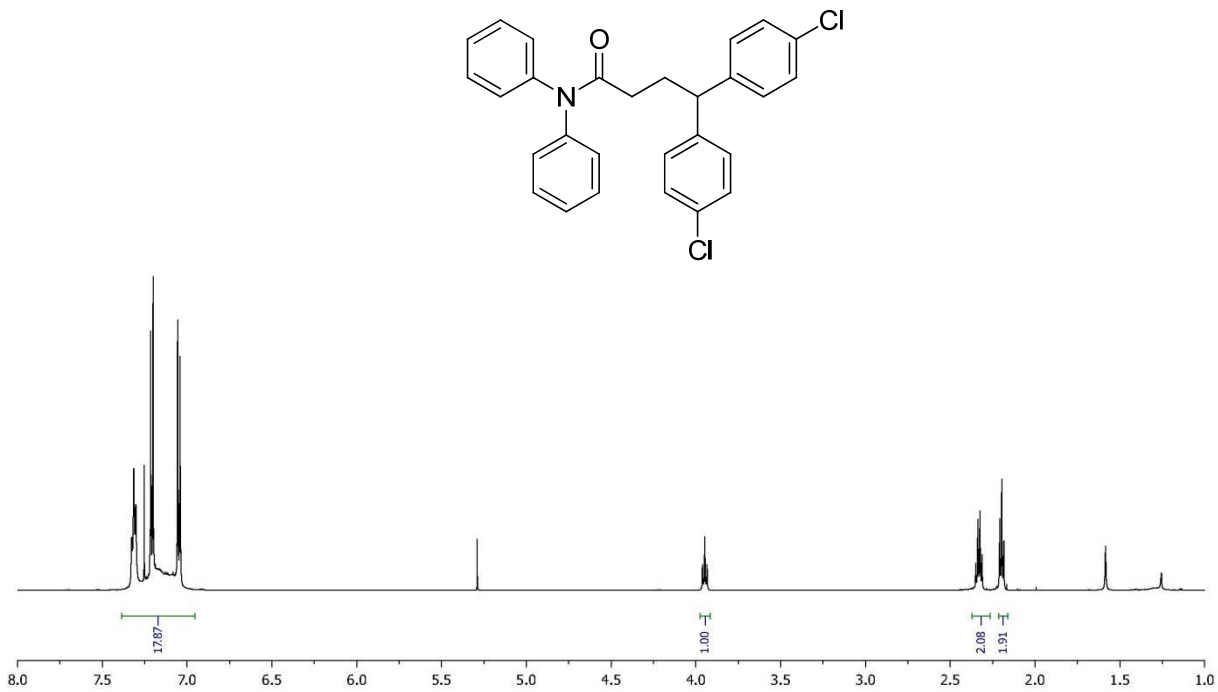
4-(4-chlorophenyl)-*N*,*N*,4-triphenylbutanamide (7bh)

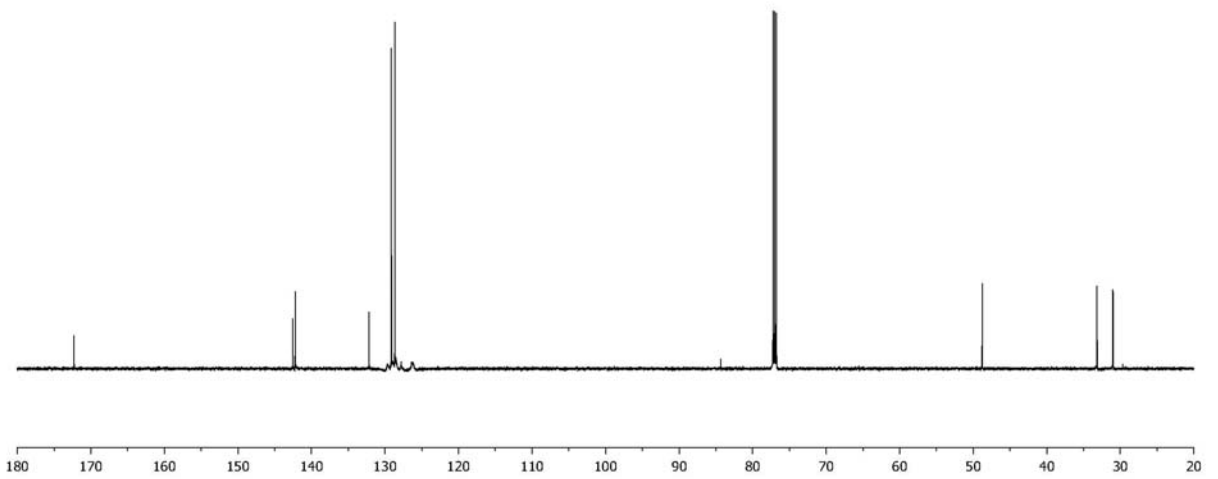




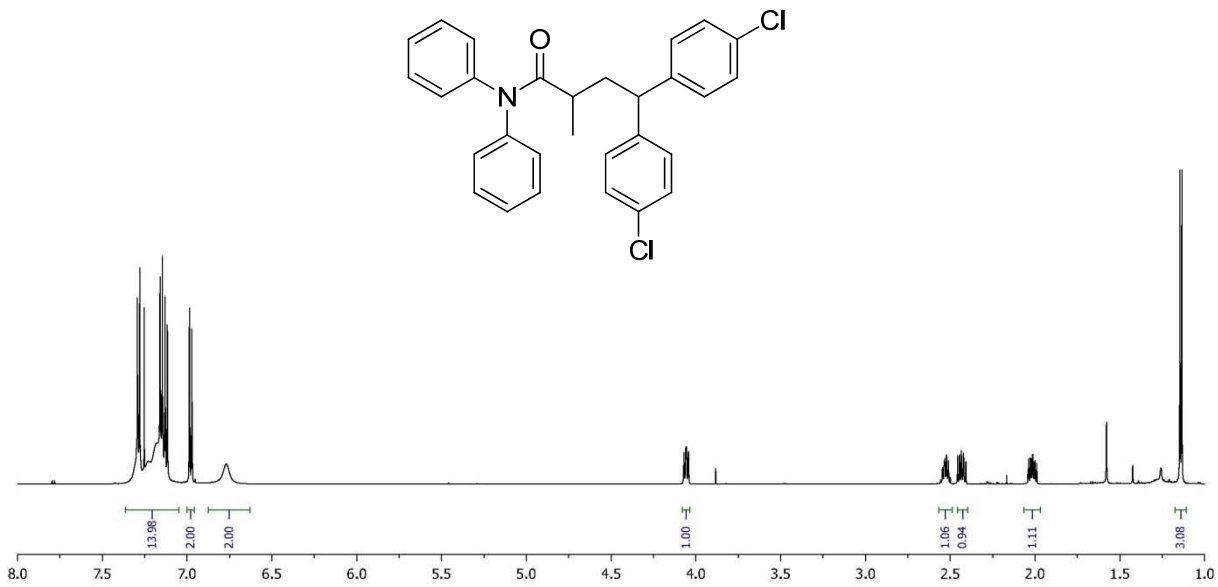


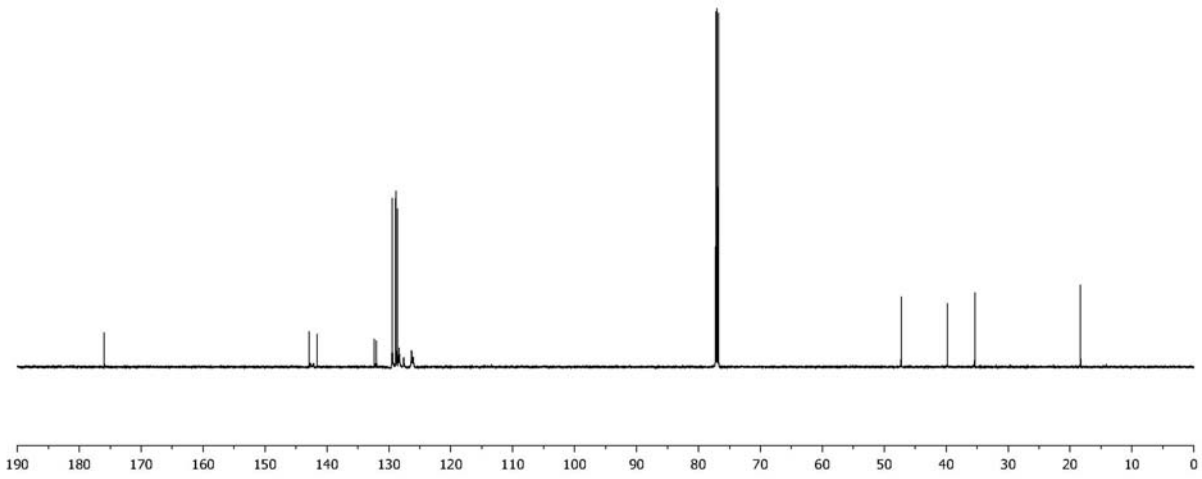
4,4-bis(4-chlorophenyl)-*N*,*N*-diphenylbutanamide (7bi)



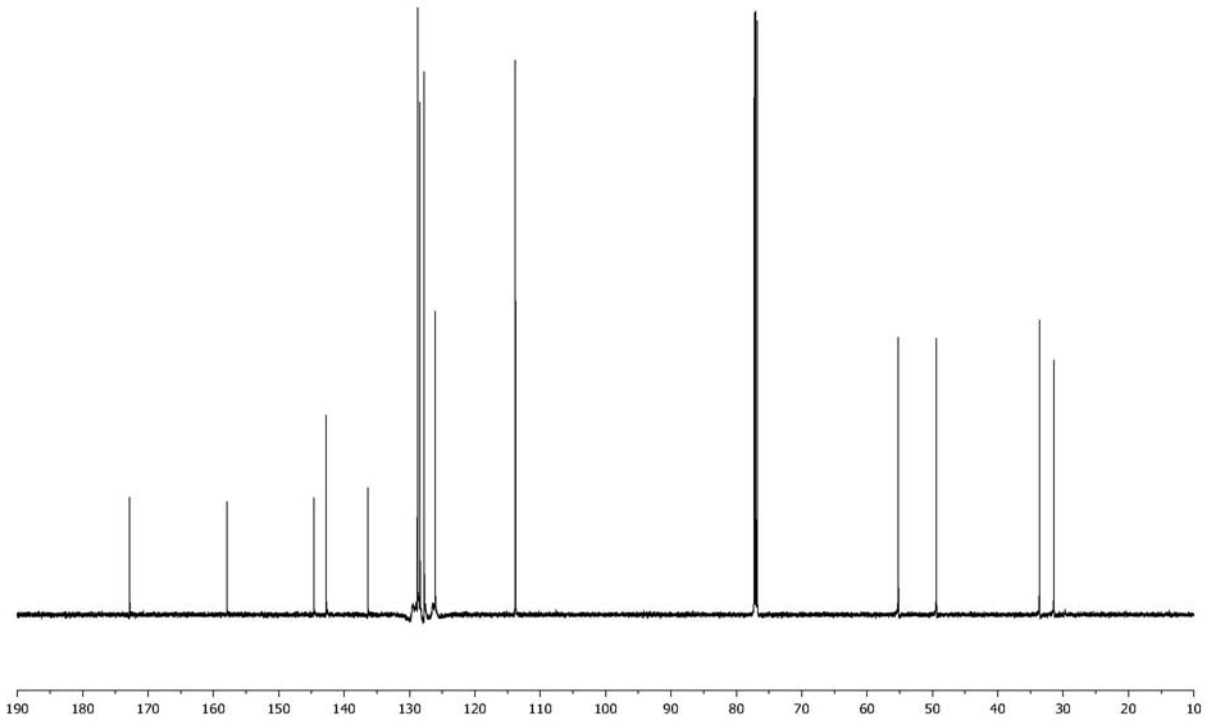


4,4-bis(4-chlorophenyl)-2-methyl-*N*,*N*-diphenylbutanamide (7ai)

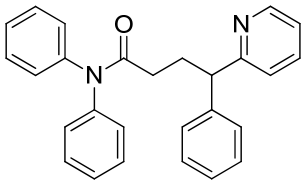


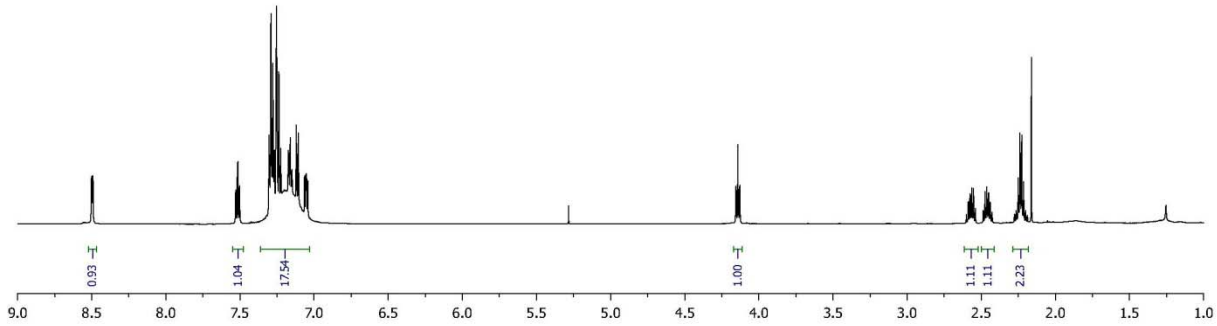


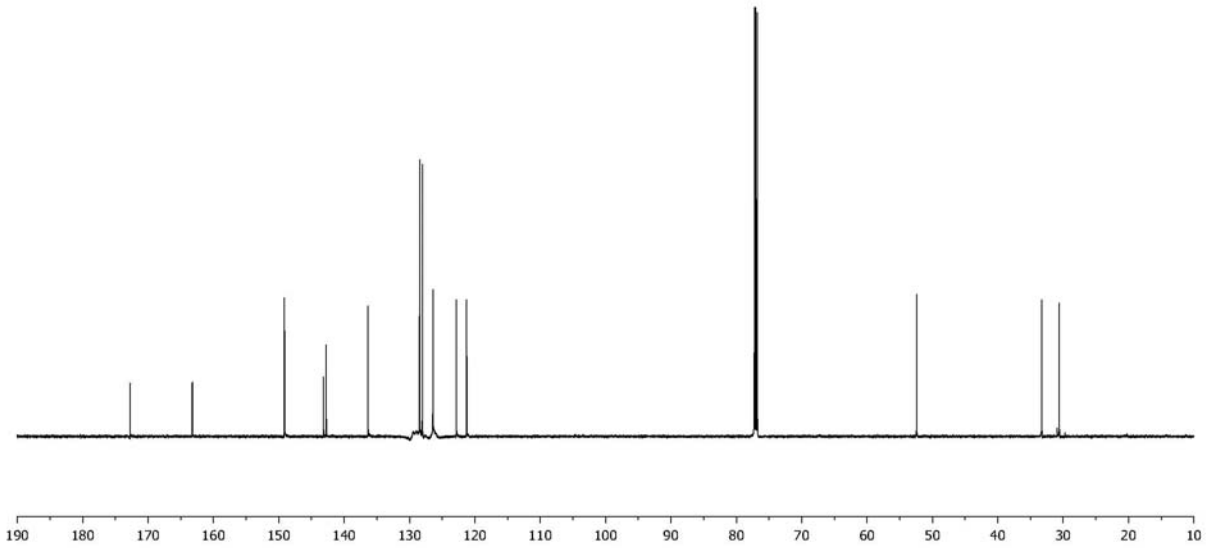
4-(4-methoxyphenyl)-*N*,*N*,4-triphenylbutanamide (7bk)



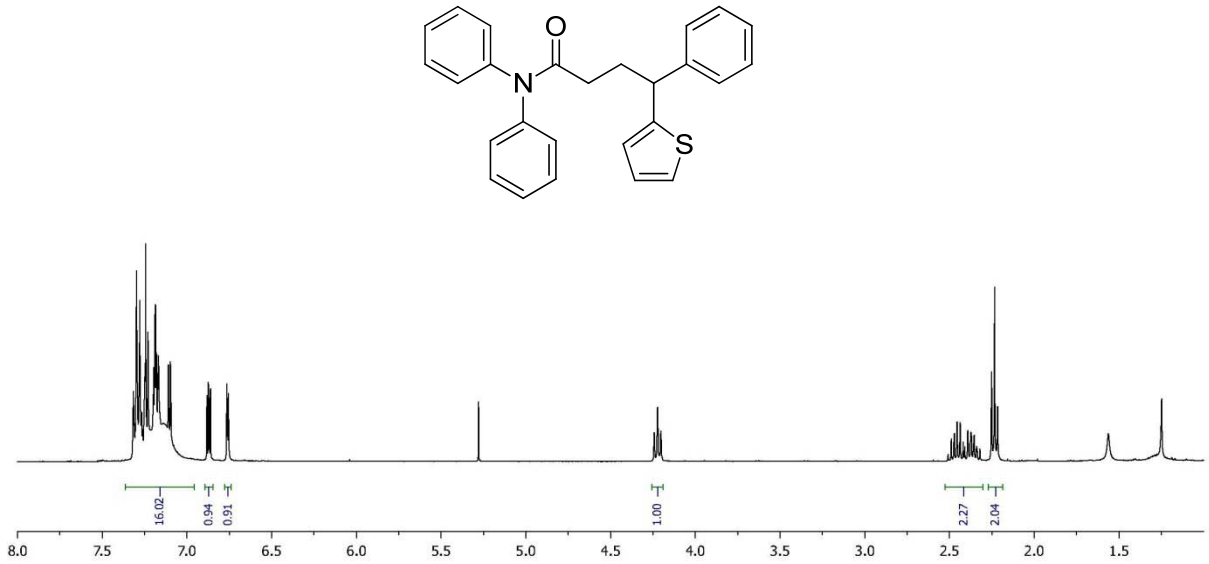
***N*,*N*,4-triphenyl-4-(pyridin-2-yl)butanamide (7bl)**

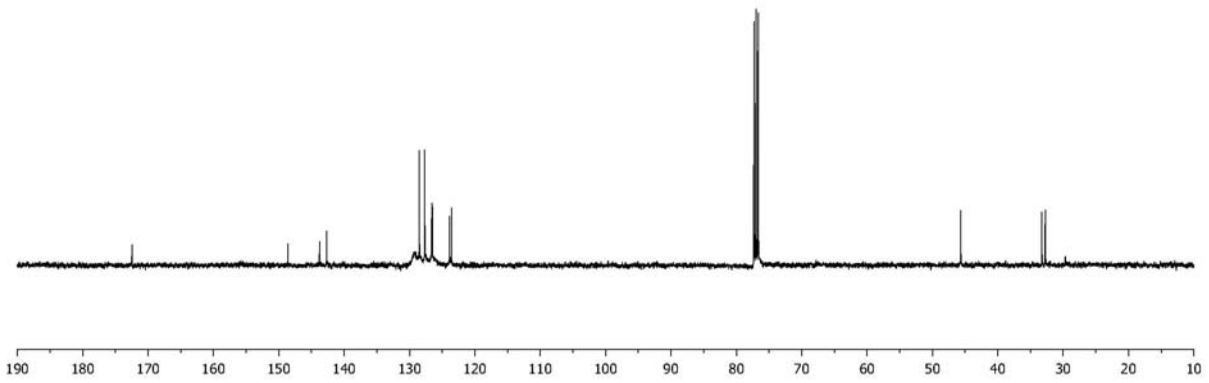




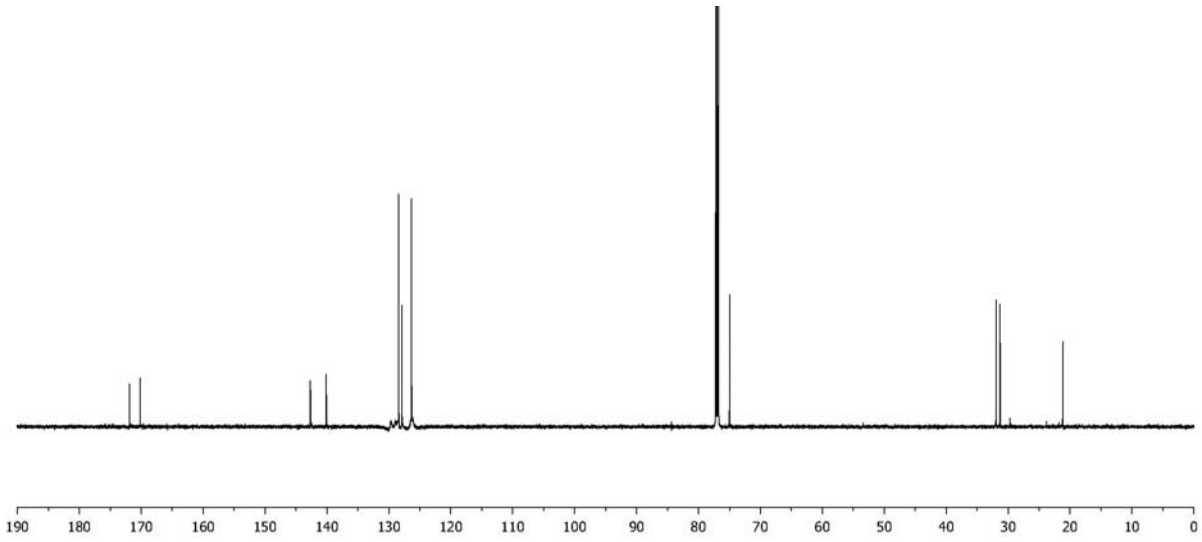


***N*,*N*,4-triphenyl-4-(thiophen-2-yl)butanamide (7bm)**

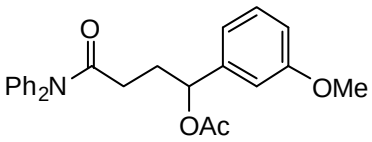


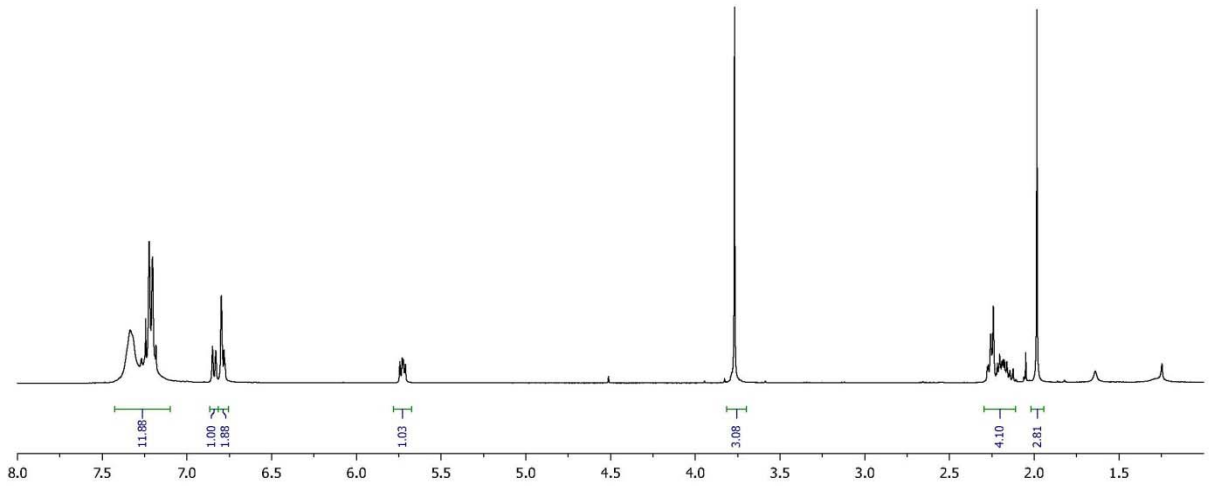


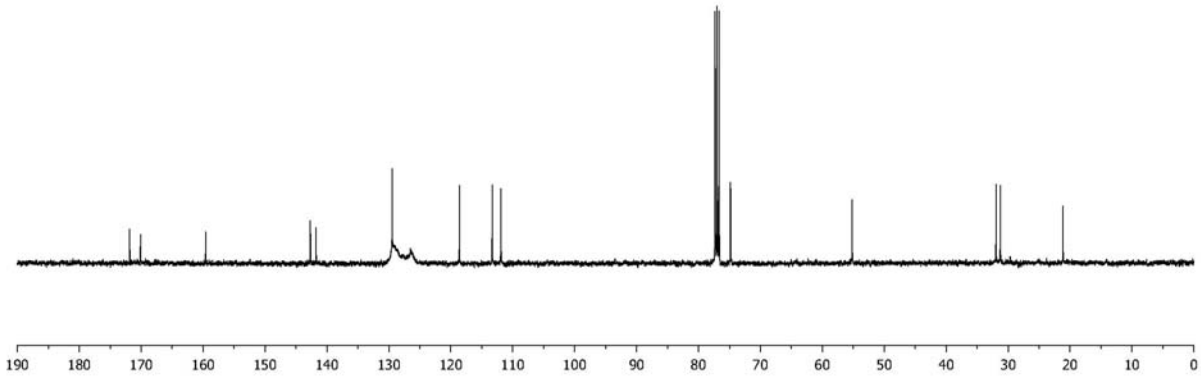
4-(diphenylamino)-4-oxo-1-phenylbutyl acetate (7bn)



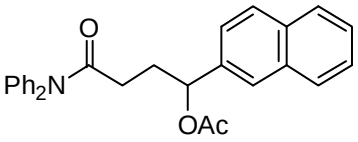
4-(diphenylamino)-1-(3-methoxyphenyl)-4-oxobutyl acetate (7bo)

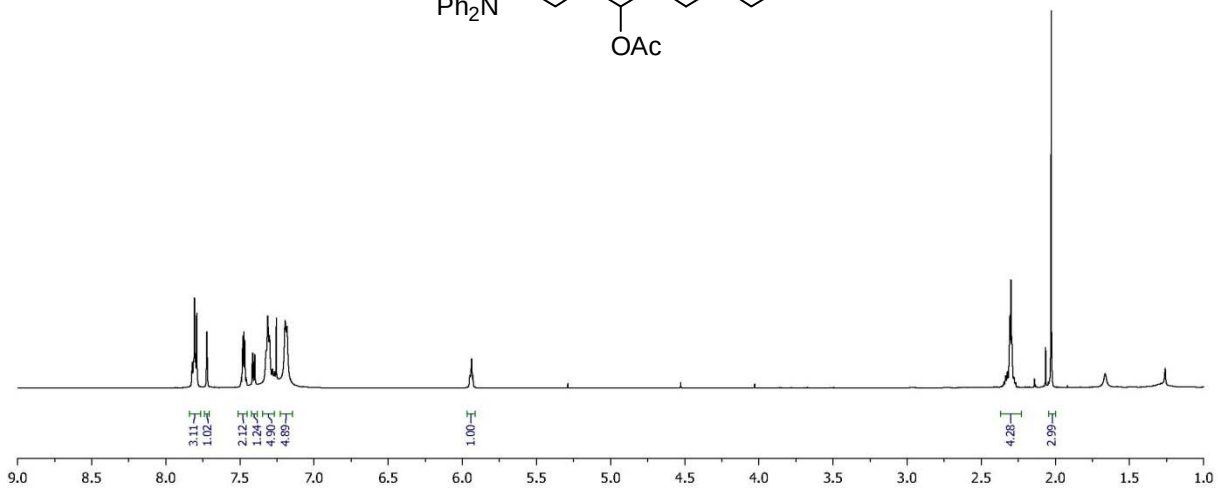


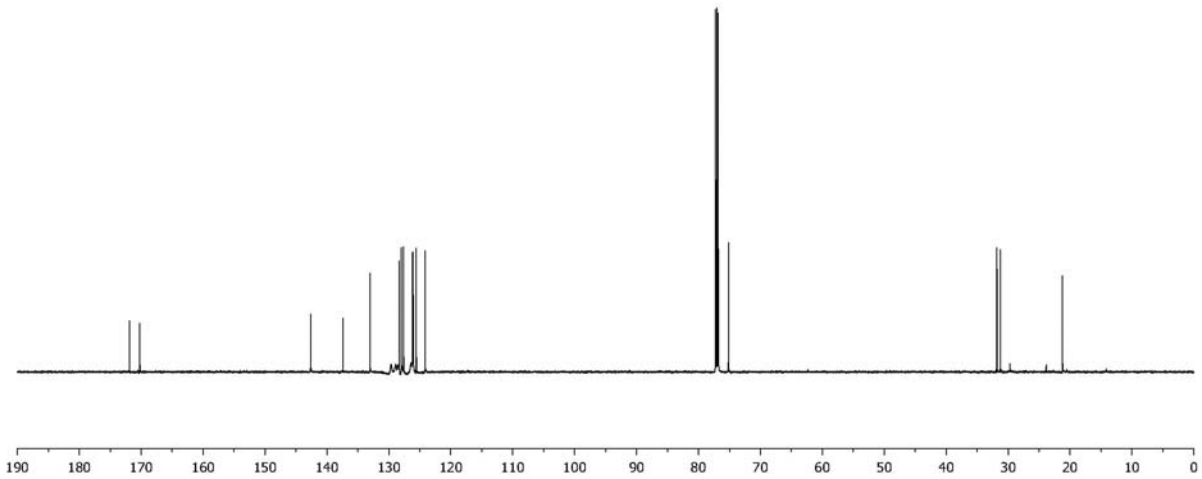




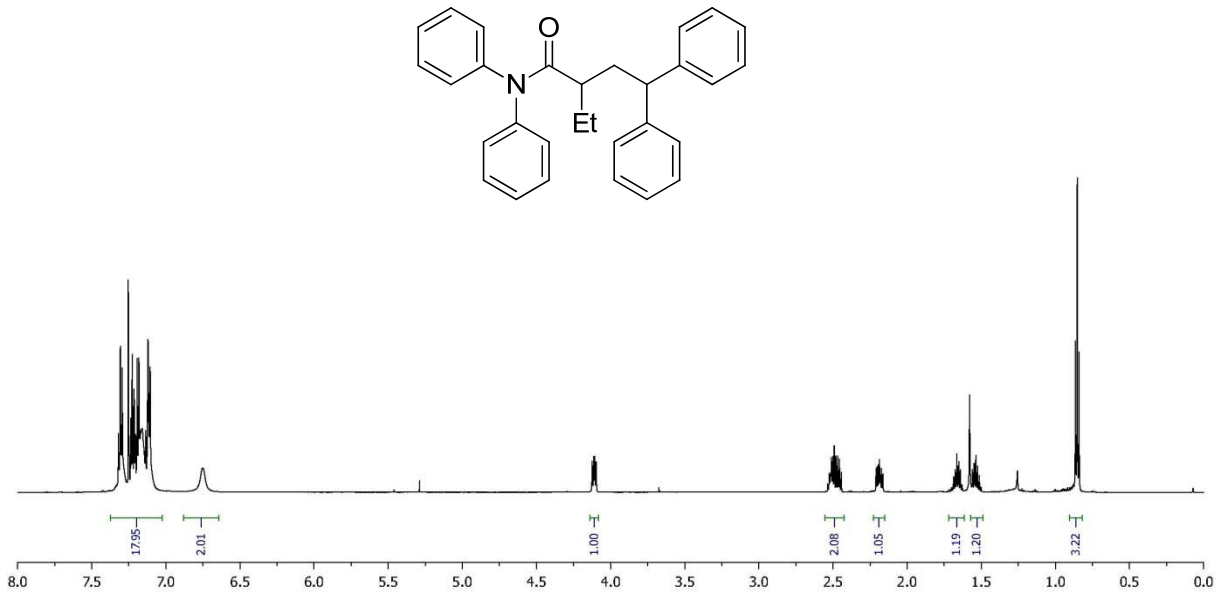
4-(diphenylamino)-1-(naphthalen-2-yl)-4-oxobutyl acetate (7bp)

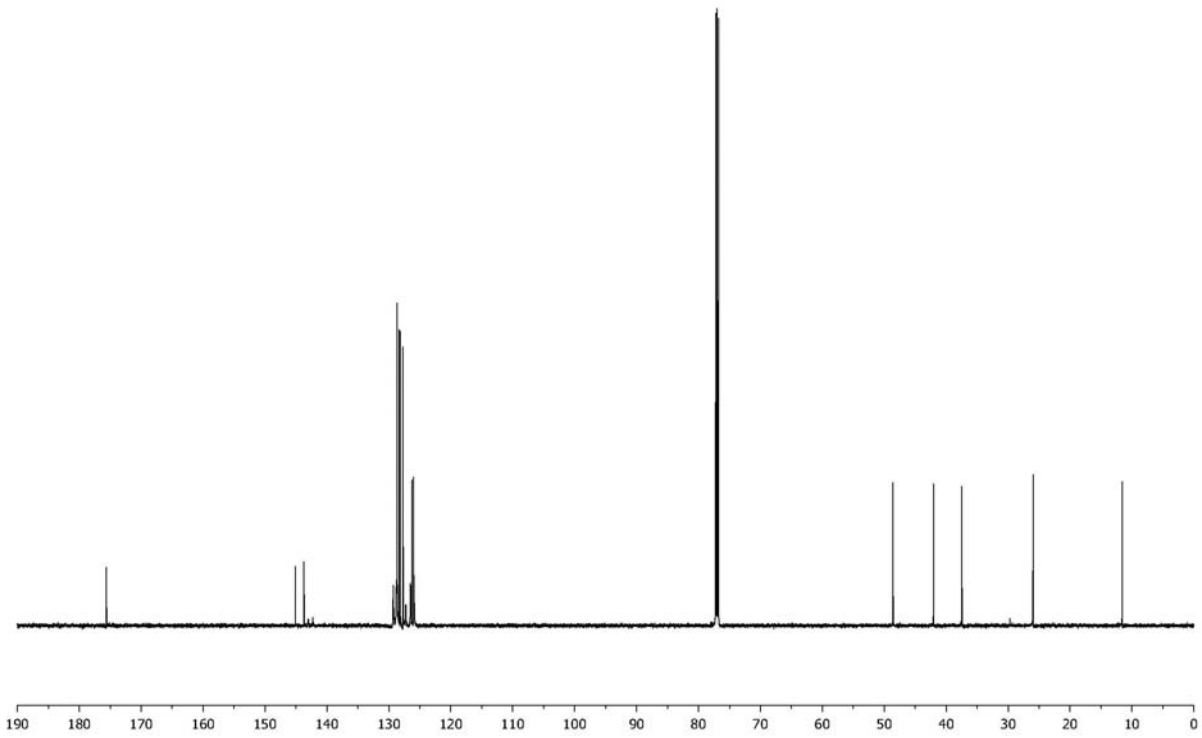




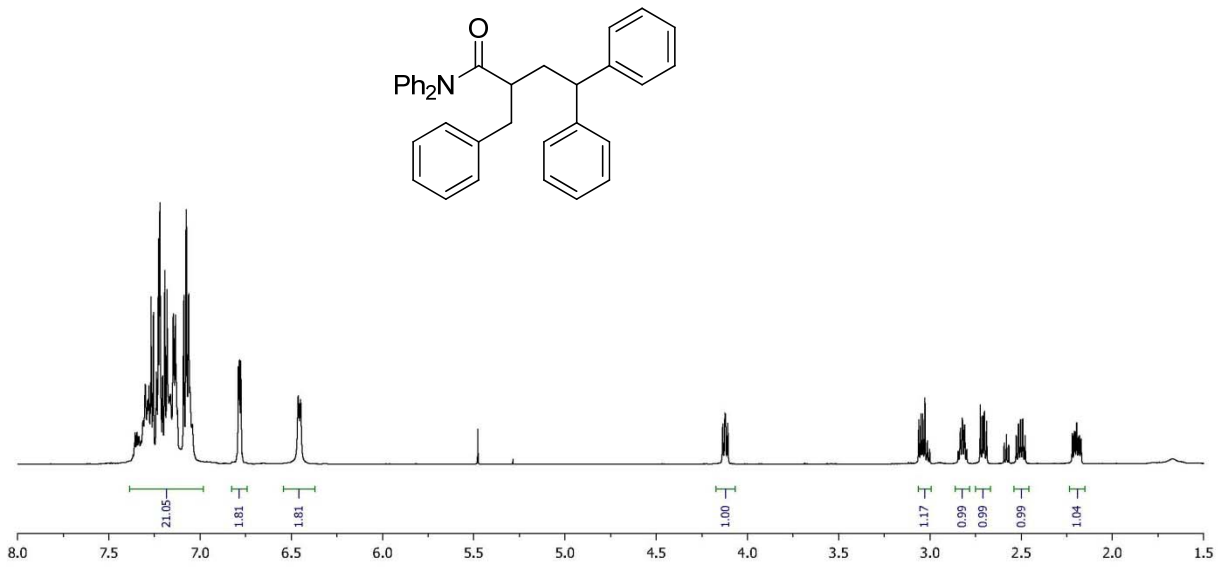


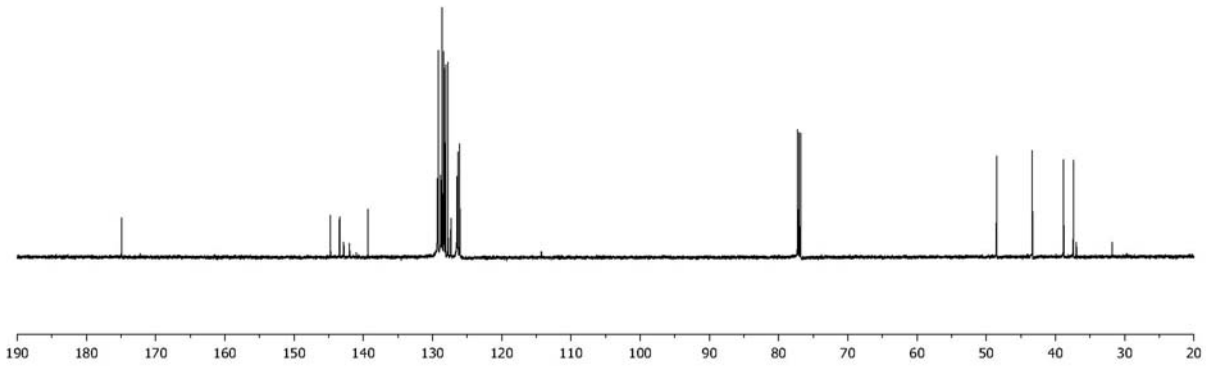
2-ethyl-*N*,*N*,4,4-tetraphenylbutanamide (7ca)



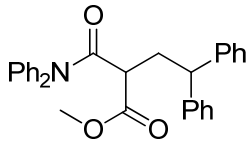


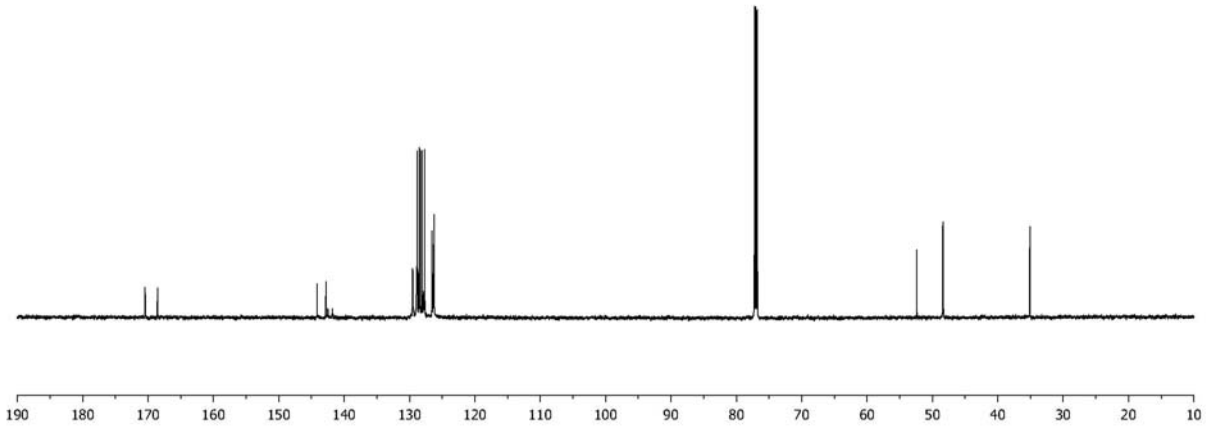
2-benzyl-*N*,*N*,4,4-tetraphenylbutanamide (7da)



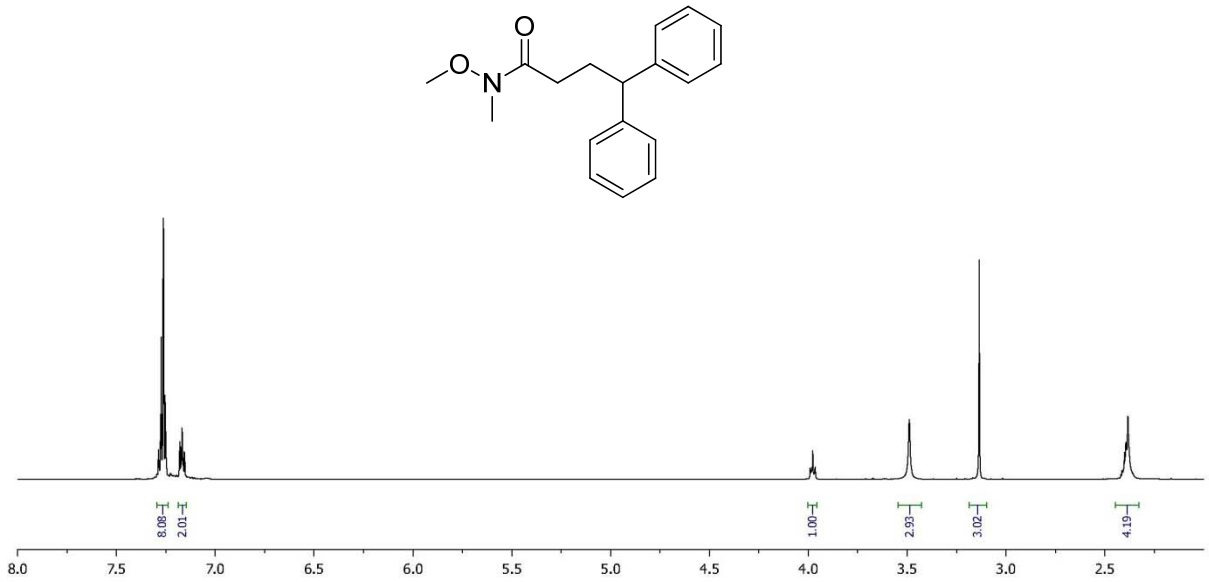


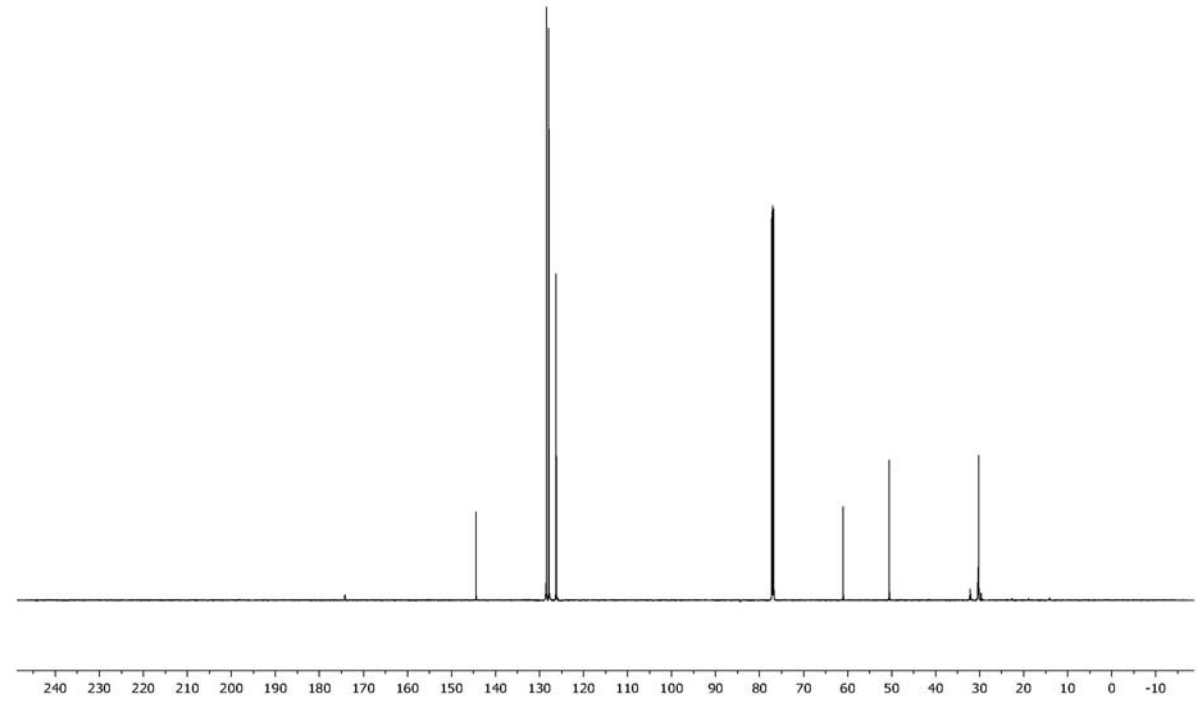
methyl 2-(diphenylcarbamoyl)-4,4-diphenylbutanoate (7ea)



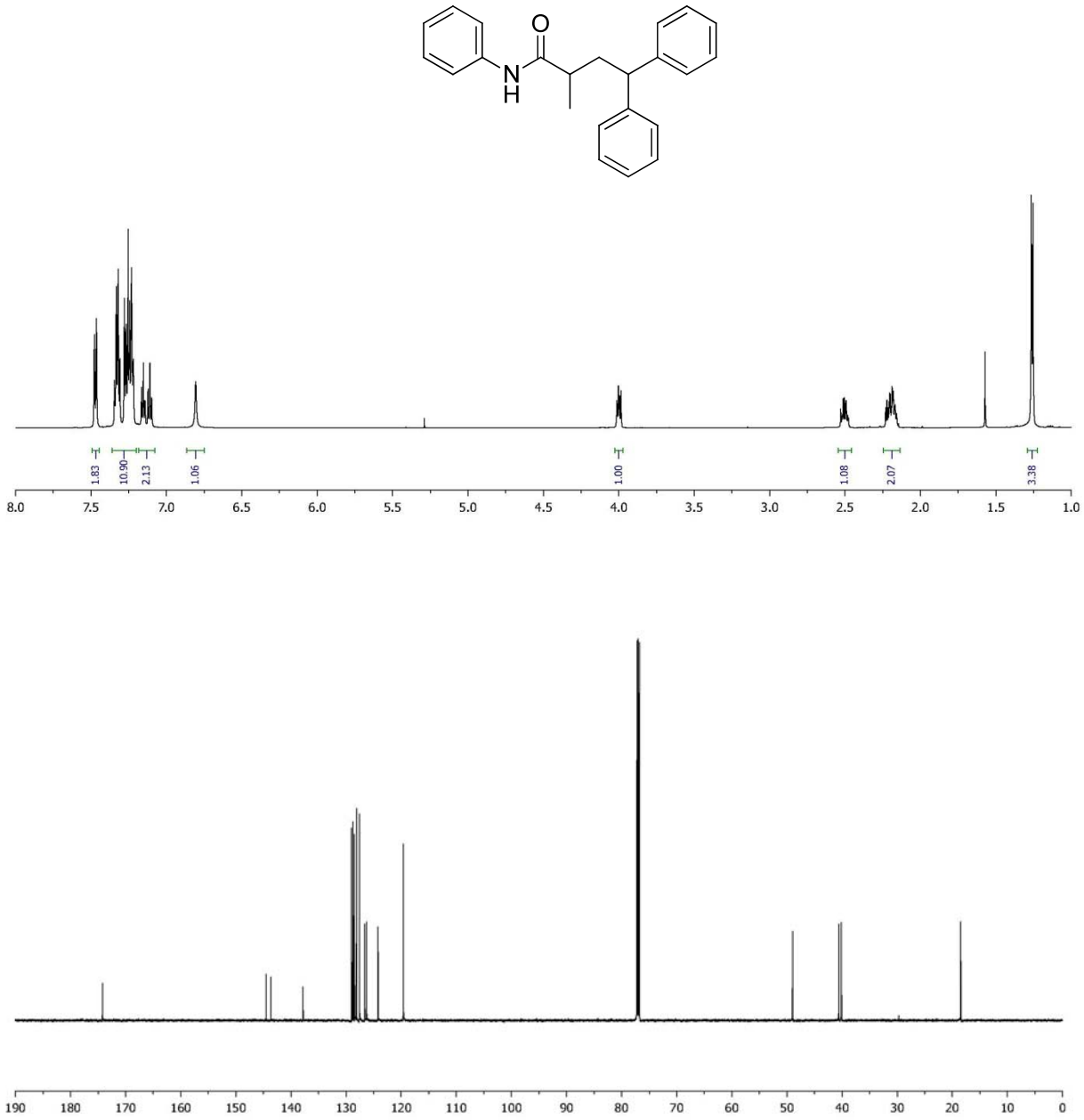


***N*-methoxy-*N*-methyl-4,4-diphenylbutanamide (7fa)**

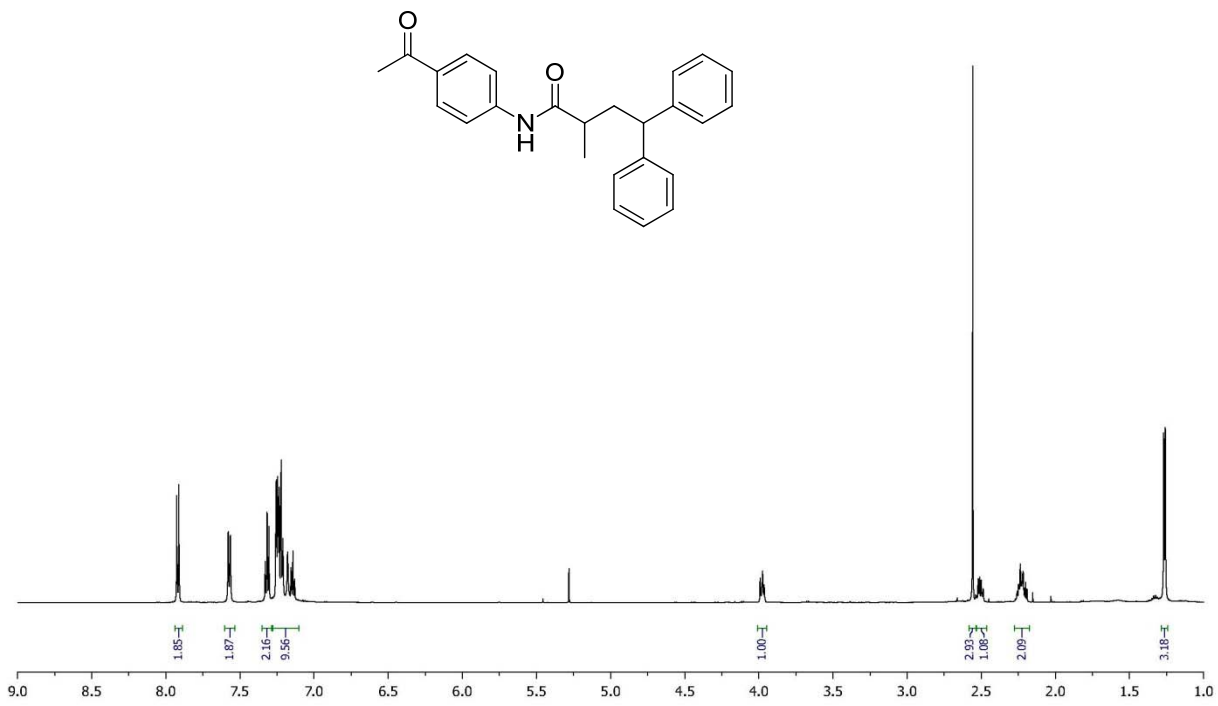


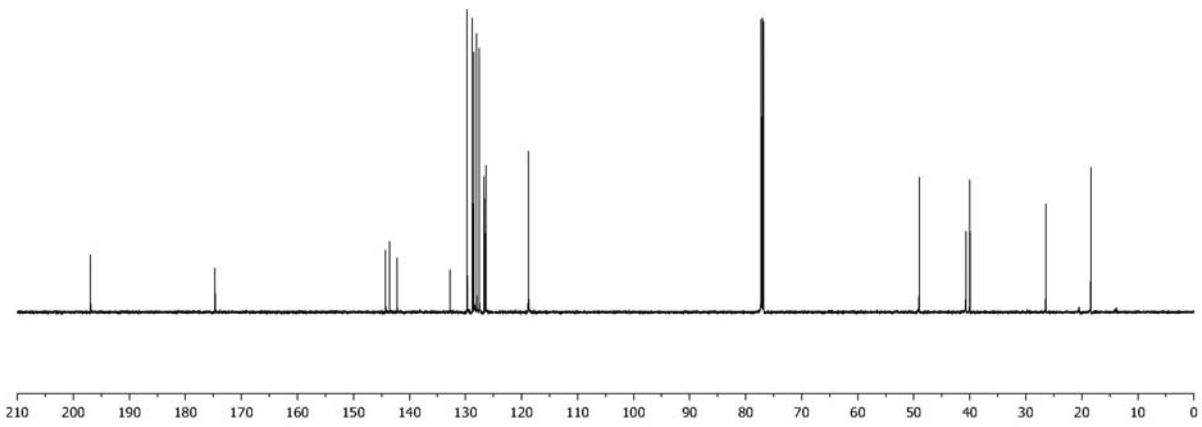


2-methyl-*N*,4,4-triphenylbutanamide (7ga)



***N*-(4-acetylphenyl)-2-methyl-4,4-diphenylbutanamide (7ha)**





2-methyl-4,4-diphenyl-*N*-(4-(trifluoromethyl)phenyl)butanamide (7ia)