

Palladium and Visible-Light Mediated Carbonylative Suzuki-Miyaura Coupling of Unactivated Alkyl Halides and Aryl Boronic Acids

Sara Roslin^a, Luke R. Odell^{*a}

^aOrganic Pharmaceutical Chemistry, Department of Medicinal Chemistry, Uppsala Biomedical Center, Uppsala University, P.O. Box 574, SE-751 23 Uppsala, Sweden

Contents

General information	S2
General procedure for optimization of carbonylative Suzuki coupling	S2
Synthesis of alkyl iodides	S5
General procedure for carbonylative Suzuki coupling – investigation of scope	S6
Control experiments	S12
Pressure measurement	S13
References	S15
NMR spectra	S16

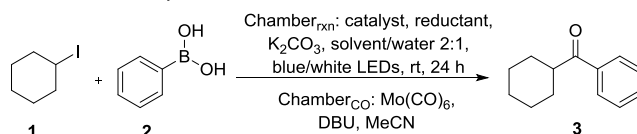
General information

All reagents were purchased at the highest commercial quality and used without further purification. Yields refer to isolated, homogenous and spectroscopically pure material, unless otherwise stated. Blue light-emitting diodes (2 W, $\lambda = 465$ nm) and white light-emitting diodes (2W, 3200K) were used for irradiation of the reaction mixture. Crude reaction mixtures were purified by silica gel chromatography (E. Merck silica gel, particle size 0.043–0.063 mm). The luminous emittance was measured at the surface of the reaction vessel (a distance of 7 cm) to 12 900 lux (all LED lists) and 4500 lux (single LED list) with a lux meter (Lux light meter, CL-J18-11-C3). Thin layer chromatography was carried out using E. Merck silica plates (60F-254) with UV light (254 nm) as the visualization agent. Analytical reversed phase HPLC-MS (ESI) was performed on a Dionex Ultimate 3000 system using MeCN/0.05% HCOOH in H₂O as the mobile phase with MS detection, equipped with a C18 (Phenomenex Kinetex SB-C18 (4.8 × 50 mm)) column using a UV diode array detector. Purity determinations were performed on the same instrument with UV detection at 245 nm. Low resolution GC-MS (EI) analyses were performed with a CP-SIL 8 CB low bleed (30 m × 0.25 mm) capillary column using a 70–300 °C temperature gradient and electron impact ionization at 70 eV. Accurate mass values were determined on a mass spectrometer equipped with an electron-impact ion source and TOF detector. ¹H NMR spectra were recorded at 400 MHz and ¹³C{¹H} NMR spectra at 100 MHz. The chemical shifts for ¹H NMR and ¹³C{¹H} NMR spectra were referenced to tetramethylsilane *via* residual solvent signals (¹H: CHCl₃ δ 7.26, CD₂HOD δ 3.31 ¹³C: CDCl₃ δ 77.16, CD₃OD δ 49.00).

General procedure for optimization of carbonylative Suzuki coupling

At the initiation of the optimization, **1** (0.25 mmol), **2** (0.375 mmol), potassium carbonate (K₂CO₃, 0.25 mmol), Pd(PPh₃)₂Cl₂ (5 mol%), *fac*-Ir(ppy)₃ (1 mol%) and Hantzsch ester (0.5 mmol) was added to the reaction chamber (C_{rxn}), followed by benzene and water (2:1, 2 mL total volume). To the carbon monoxide chamber (C_{CO}), molybdenum hexacarbonyl (Mo(CO)₆, 0.75 mmol) was added followed by acetonitrile (2 mL). All solvents were sonicated for 15 min before addition to the H-tube. After capping of C_{rxn} and C_{CO}, DBU (1.5 mmol) was added to C_{CO}. C_{rxn} was then subjected to visible light radiation for 24 h. Both chambers were held at room temperature. After 24 h, the contents of C_{rxn} was filtered and washed with dichloromethane. The filtrate was concentrated in vacuo. Anisole (1 eq) was used as the internal standard for calculation of the NMR yield. In experiments with anisole as solvent, 1-hexene (1 eq) was used as the internal standard. See table S1 for all experiments.

Table S1. Full optimization of reaction parameters



Entry	Solvent C _{rxn}	Catalyst/ligand	Base	Reductant	Temperature	NMR yield ^a
					C _{CO}	
S1	Benzene	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S2	Benzene + water 100 μ L	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	7%
S3	Benzene/water 9:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	9%
S4	Benzene/water 7:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	15%
S5	Benzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	17%
S6	Benzene/water 1:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S7	Acetonitrile	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-

S8	Acetonitrile + water 100 μ L	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S9	Acetonitrile /water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S10	Methylcyclohexane/water 7:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	Trace ^b
S11	Methylcyclohexane/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	Trace ^b
S12	Perfluorobenzene/water 7:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S13	Perfluorobenzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S14	Benzene/acetonitrile 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S15	Methylcyclohexane/acetonitrile 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S16	Benzene/acetonitrile/water 4:1:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	6%
S17	Water	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S18	Benzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (0.5 eq)	70 °C	32%
S19	Benzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.5 eq)	70 °C	37%
S20	Benzene/water 7:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	7%
S21	Benzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	-
S22	Benzene	Pd(dba) ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	-
S23	Benzene/water 2:1	Pd(dba) ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	-
S24	Benzene + water 100 μ L	Pd(dba) ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	-
S25	Benzene/water 7:1	Pd(dppf)Cl ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	-
S26	Benzene/water 2:1	Pd(dppf)Cl ₂ , <i>fac</i> -Ir(ppy) ₃ /Xantphos ^d	K ₂ CO ₃	HE (2 eq)	RT	Trace ^b
S27	Benzene/acetonitrile/water 4:1:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	6%
S28	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	24%
S29	Benzene/water 2:1	Pd(OAc) ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	-
S30	Benzene/water 2:1	Pd(dppf)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	RT	7%
S31	Benzene/water 2:1	Pd(dba) ₂ , <i>fac</i> -Ir(ppy) ₃ /Xphos ^e	K ₂ CO ₃	HE (2 eq)	RT	-
S32	Benzene/water 2:1	Pd(dba) ₂ , <i>fac</i> -Ir(ppy) ₃ /Sphos ^e	K ₂ CO ₃	HE (2 eq)	RT	Trace ^b
S33	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	CsCO ₃	HE (2 eq)	RT	18%
S34	Benzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	CsCO ₃	HE (2 eq)	RT	-
S35	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	NEt ₃	HE (2 eq)	RT	8%
S36	Benzene/water 2:1	Pd(PPh ₃)Cl ₂ , <i>fac</i> -Ir(ppy) ₃	NEt ₃	HE (2 eq)	RT	Trace ^b
S37	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (2 eq)	70 °C	31%
S38	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (1 eq)	70 °C	19%
S39	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (0.5 eq)	70 °C	21%
S40	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃	K ₂ CO ₃	HE (0.25 eq)	70 °C	37%
S41	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (2 eq)	70 °C	31%
S42	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (1 eq)	70 °C	31%
S43	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.5 eq)	70 °C	46%
S44	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.25 eq)	70 °C	35%
S45	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.1 eq)	70 °C	29%
S46	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE	70 °C	32%

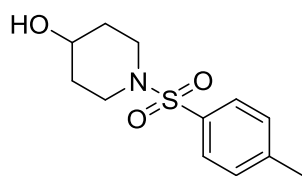
			TBA	(0.25 eq)			
			(2 eq)				
S47	Benzene/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^{c,f}	K ₂ CO ₃	HE (0.5 eq)	70 °C	26%	
S48	Benzene ^g	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.5 eq)	70 °C	24%	
S50	Benzene/water 7:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.5 eq)	70 °C	37%	
S51	Dioxane/water 2:1	Pd(PPh ₃) ₄ , <i>fac</i> -Ir(ppy) ₃ ^c	K ₂ CO ₃	HE (0.5 eq)	70 °C	Trace ^b	
S52	Benzene/water 2:1	NiCl ₂ , <i>fac</i> - Ir(ppy) ₃ ^c /BBBDP ^d	K ₂ CO ₃	HE (0.5 eq)	RT	-	
S53	Dioxane + 5 eq water	NiCl ₂ , <i>fac</i> - Ir(ppy) ₃ ^c /BBBDP ^d	K ₂ CO ₃	HE (0.5 eq)	RT	-	
S54	Benzene/water 2:1	Pd(PPh ₃) ₄	K ₂ CO ₃	-		49%	
S55	Benzene/water 2:1	Pd(PPh ₃)Cl ₂	K ₂ CO ₃	-	70 °C	39%	
S56 ^h	Benzene/water 2:1	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	51%	
S57 ^h	Benzene/water 2:1	Pd(PPh ₃) ₄ ⁱ	K ₂ CO ₃	-	70 °C	63%	
S58 ^h	Benzene/water 2:1	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	56%	
S59 ^h	Benzene/water 2:1 ^k	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	60%	
S59 ^h	Benzene/water 2:1 ^l	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	64%	
S60 ^h	Benzene/water 2:1 ^m	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	61%	
S61 ^h	Benzene/water 2:1 ⁿ	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	41%	
S62 ^h	Benzene/water 2:1 ^{j,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	64%	
S63^h	Benzene/water 2:1^{l,n}	Pd(PPh₃)₄	K₂CO₃	-	70 °C	71%	
S64 ^h	Benzene/water 2:1 ^{m,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	67%	
S65 ^h	Benzene/water 2:1 ^{l,o}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	47%	
S66 ^h	Benzene/water 2:1 ^{l,p}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	37%	
S67 ^h	Benzene/water 2:1 ^{l,q}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	Ester formed ^b	33%
S68 ^h	Benzene/water 2:1 ^{l,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C		
			TBA	(1 eq)			
S69 ^h	Benzene/water 2:1 ^{l,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	43%	
			(2 eq)				
S70 ^h	Benzene/water 2:1 ^{l,n}	Pd(PPh ₃) ₄	NaOH	-	70 °C	41%	
			(2 eq)				
S71 ^h	Anhydrous acetonitrile ^{l,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	68%	
S72 ^h	Benzene ^{l,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	38%	
S73 ^h	Benzene/water 2:1 ^{l,n,r}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	58%	
S74 ^h	Toluene/water 2:1 ^{l,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	70%	
S75 ^h	Anisole/water 2:1 ^{l,n}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	(23%)	
S76 ^h	Benzene/water 2:1 ^{l,n}	Pd(PPh ₃) ₄ /dppp ^s	K ₂ CO ₃	-	70 °C	-	
S77 ^h	Benzene/water 2:1 ^{l,n}	Pd(PPh ₃) ₄ /dppf ^s	K ₂ CO ₃	-	70 °C	-	
S78 ^h	Benzene/water 2:1 ^{l,n,t}	Pd(PPh ₃) ₄	K ₂ CO ₃	-	70 °C	71%	
S79	Benzene/water 2:1	-	K ₂ CO ₃	-	70 °C	-	
S80	Benzene/water 2:1	Pd(PPh ₃) ₄ ^u	-	-		Trace ^b	

Reaction conditions: Chamber_{rxn} 0.25 mmol **1**, 1.5 eq **2**, 5 mol% [Pd], 1 eq K₂CO₃, 1 mol% *fac*-Ir(ppy)₃, 2 ml solvent Chamber_{co} 2.5 eq Mo(CO)₆, 5 eq DBU, 2 ml MeCN. Solvents were sonicated for 15 min before addition to H-tube. ^a Yield determined by ¹H NMR. ^b Determined by visual inspection of GC. ^c 2 mol% *fac*-Ir(ppy)₃ ^d 5 mol% ligand. ^e 10 mol% ligand. ^f 10 mol% Pd(PPh₃)₄. ^g 3 eq water. ^h N₂/vacuum sparging for 5 minutes instead of sonication of solvent. ⁱ 7.5 mol% Pd(PPh₃)₄. ^j 3 mL solvent in C_{rxn} and C_{co}. ^k 3.33 mL solvent in C_{rxn} and C_{co}. ^l 4 mL solvent in C_{rxn} and C_{co}. ^m 5 mL solvent in C_{rxn} and C_{co}. ⁿ Increase in concentration, to 0.3 mmol of cyclohexyl iodide. ^o 3 eq of cyclohexyl iodide. ^p 1 eq phenyl boronic acid. ^q 5 eq phenyl boronic acid. ^r 1 eq of naphthalene. ^s 7 mol% ligand. ^t Heating both chambers to 50 °C. ^u Reaction run without light.

Abbreviations: ppy = 2-phenylpyridinato-C², HE = Hantzsch ester, RT = room temperature, BBBDP = 4,4'-di-*tert*-butyl-2,2'-bipyridine, TBA = tributylamine.

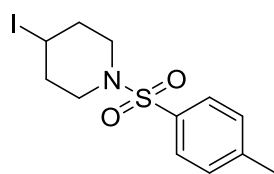
Synthesis of alkyl iodides

1-[(4-Methylphenyl)sulfonyl]piperidin-4-ol¹ CAS: 80213-12-3



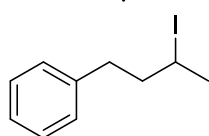
4-Hydroxypiperidine (2.0 mmol) was dissolved in water (5 mL), to which Na_2CO_3 (1.3 eq) and *p*-toluenesulfonyl chloride (1.6 eq) was added. The reaction mixture was diluted with diethylether (15 mL) and left to stir at room temperature for 24 h. After completion, the reaction mixture was extracted with 3x20 mL ethyl acetate. The combined organic phases were washed with 30 mL brine, dried over Na_2SO_4 , filtered and concentrated *in vacuo*. Purified with silica column chromatography using a gradient (3:1 *i*-hexane:ethyl acetate + 1% triethylamine to ethyl acetate + 1% triethylamine). Isolated as a white solid (404.0 mg, 81%). $R_f = 0.59$ (ethyl acetate + 1% triethylamine). Spectral data were in agreement with literature values. ^1H NMR (400 MHz, CDCl_3) δ 7.66 – 7.61 (m, 2H), 7.33 – 7.29 (m, 2H), 3.78 – 3.70 (m, 1H), 3.35 – 3.26 (m, 2H), 2.87 – 2.79 (m, 2H), 2.43 (s, 3H), 1.96 – 1.87 (m, 2H), 1.69 – 1.59 (m, 2H), 1.46 (s, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.7, 133.2, 129.8, 127.8, 66.0, 43.3, 33.4, 21.7. MS-ESI [$\text{M}+\text{H}^+$]: m/z 256.2. HPLC purity > 99%.

4-Iodo-1-[(4-methylphenyl)sulfonyl]piperidine¹ CAS: 289890-80-8



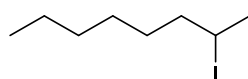
1-[(4-methylphenyl)sulfonyl]piperidin-4-ol (1.6 mmol) was dissolved in dichloromethane (10 mL), before addition of PPh_3 (1.6 eq) and imidazole (1.6 eq). The reaction mixture was stirred at -10°C for 5 minutes before addition of I_2 (2.0 eq). The ice-bath was then removed and the mixture stirred at ambient temperature overnight. The reaction mixture was then diluted with H_2O (10 mL) and extracted with 2x10 mL diethylether. The combined organic phases were dried over Na_2SO_4 , filtered and concentrated *in vacuo*. Purification with silica column chromatography eluting with 3:1 *i*-hexane:ethyl acetate + 1% trimethylamine afforded the title compound as a beige solid (413.6 mg, 72%). $R_f = 0.88$ (3:1 *i*-hexane:ethyl acetate + 1% triethylamine). Spectral data were in agreement with literature values. ^1H NMR (400 MHz, CDCl_3) δ 7.65 (d, $J = 8.3$ Hz, 2H), 7.34 (d, $J = 8.0$ Hz, 2H), 4.33 – 4.25 (m, 1H), 3.21 – 3.12 (m, 2H), 3.02 – 2.95 (m, 2H), 2.45 (s, 3H), 2.16 – 2.08 (m, 4H). ^{13}C NMR (101 MHz, CDCl_3) δ 143.9, 133.3, 129.9, 127.7, 45.8, 36.6, 25.7, 21.7. MS-ESI [$\text{M}+\text{H}^+$]: m/z 365.9. HPLC purity > 99%.

3-Iodobutylbenzene^{2,3} CAS: 59456-20-1



4-Phenyl-2-butanol (6 mmol), PPh_3 (1 eq), imidazole (1.5 eq) and I_2 (1.3 eq) were dissolved in dichloromethane (15 mL) at -10°C and left to stir for 4 h at room temperature. The reaction mixture was concentrated *in vacuo* and purified with silica column chromatography eluting with 7:1 *i*-hexane:ethyl acetate. Isolated as a yellow oil (1.5 g, 96%). $R_f = 0.70$ (29:1 *i*-hexane:ethyl acetate). Spectral data were in agreement with literature values. ^1H NMR (400 MHz, CDCl_3) δ 7.32 – 7.27 (m, 2H), 7.23 – 7.18 (m, 3H), 4.17 – 4.06 (m, 1H), 2.90 – 2.80 (m, 1H), 2.75 – 2.64 (m, 1H), 2.21 – 2.09 (m, 1H), 1.95 (d, $J = 6.8$ Hz, 3H), 1.93 – 1.83 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 140.9, 128.6, 128.6, 126.2, 44.5, 36.0, 29.8, 29.1. EI-MS: m/z 260.0.

2-Iodo-octane^{3,4} CAS: 557-36-8



2-Octanol (7.7 mmol), PPh_3 (1 eq), imidazole (2 eq) and I_2 (1.2 eq) were dissolved in dichloromethane (25 mL) at -10°C and left to stir for 3 days at room temperature. The reaction mixture was filtered and concentrated *in vacuo* and purified with silica column chromatography eluting with 7:1 *i*-hexane:ethyl acetate. Isolated as a colorless oil (1.2 g, 67%). $R_f = 0.89$ (29:1 *i*-hexane:ethyl acetate). Spectral data were in agreement with literature values. ^1H NMR (400 MHz, CDCl_3) δ 4.19 (dq, $J = 8.5, 6.8, 5.1$ Hz, 1H), 1.92 (d, $J = 6.8$ Hz, 3H), 1.89 – 1.79 (m, 1H), 1.65 – 1.56 (m, 1H), 1.54 – 1.44 (m, 1H), 1.40 – 1.25 (m, 7H),

0.93 – 0.85 (m, 3H). ^{13}C NMR (101 MHz, CDCl_3) δ 43.1, 31.8, 31.0, 29.8, 29.1, 28.6, 22.7, 14.2. EI-MS: m/z 240.1.

General procedure for carbonylative Suzuki coupling – investigation of scope

Conditions as in entry S63, table S1. To C_{rxn} was added the appropriate alkyl iodide or alkyl bromide (0.3 mmol), boronic acid (1.5 eq), $\text{Pd}(\text{PPh}_3)_4$ (5 mol%) and K_2CO_3 (1 eq) followed by benzene/water (2:1, 4 mL total volume). To C_{CO} was added $\text{Mo}(\text{CO})_6$ (2.5 eq) followed by acetonitrile (4 mL). After capping, both chambers were subjected to 5 minutes of N_2 /vacuum sparging. When finished, DBU (5 eq) was added to C_{CO} and the double chamber system was positioned in a Dry-Syn heating block with C_{CO} heated at 70°C and C_{rxn} held at room temperature whilst subjected to visible light for 24 h. Thereafter, 5% citric acid (aq.) was added to the reaction mixture and the mixture was extracted with dichloromethane (3x10 mL). The organic phase was washed with brine (10 mL), dried over MgSO_4 , filtered and concentrated *in vacuo*. Extracted product was purified with silica column chromatography, with 0.5%-1% ethyl acetate in *i*-hexane or *n*-pentane as the eluent.

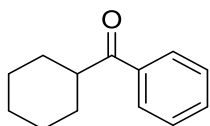
Experimental set-up

The double-chamber system can be seen in figure S1a and the experimental set-up in figure S1b. For a full description of the reaction set-up please see S. Y. Chow et. al. *Chem. Eur. J.* **2016**, *22*, 9155–9161

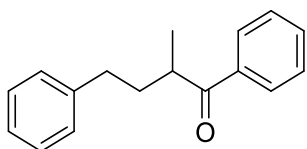


Figure S1. a) Double-chamber system, C_{CO} is the left chamber and C_{rxn} is the right chamber. b) Experimental set-up.

Cyclohexylphenylmethanone⁵ (4) CAS: 712-50-5



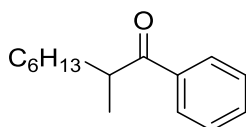
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as crystals (37 mg, 65% from cyclohexyl iodide; 20 mg, 35 % from cyclohexyl bromide; 22 mg, 39% from cyclohexyl bromide (C_{rxn} and C_{Co} heated to 50°C)). $R_f = 0.52$ (29:1 *i*-hexane:ethyl acetate). 1H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.91 (m, 2H), 7.57 – 7.51 (m, 1H), 7.49 – 7.41 (m, 2H), 3.26 (tt, $J = 11.5, 3.2$ Hz, 1H), 1.94 – 1.80 (m, 4H), 1.78 – 1.70 (m, 1H), 1.56 – 1.24 (m, 5H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.1, 136.5, 132.9, 128.7, 128.4, 45.8, 29.6, 26.1, 26.0. EI-MS: m/z 188.1.



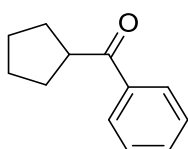
2-Methyl-1,4-diphenylbutan-1-one (5) CAS: 108974-08-9

Synthesized according to the general procedure. Isolated as a colorless oil (45 mg, 65%). $R_f = 0.25$ (29:1 *i*-hexane:ethyl acetate). 1H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.95 (m, 2H), 7.68 – 7.62 (m, 1H), 7.57 – 7.51 (m, 2H), 7.40 – 7.35 (m, 2H), 7.32 – 7.23 (m, 3H), 3.57 (h, $J = 6.8$ Hz, 1H), 2.79 – 2.72 (m, 2H), 2.33 – 2.22 (m, 1H), 1.92 – 1.81 (m, 1H), 1.34 (d, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.2, 141.9, 136.7, 133.0, 128.7, 128.6, 128.5, 128.4, 126.1, 39.9, 35.3, 33.6, 17.4. EI-MS: m/z 238.1. HRMS found: 239.1427 calc: 239.1436.

2-Methyl-1-phenyloctan-1-one⁶ (6) CAS: 132719-12-1

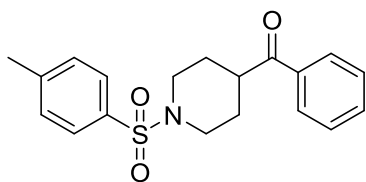


Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (43 mg, 64%). $R_f = 0.1$ (1% EtOAc in *i*-Hexane). 1H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.93 (m, 2H), 7.58 – 7.52 (m, 1H), 7.50 – 7.44 (m, 2H), 3.46 (h, $J = 6.8$ Hz, 1H), 1.85 – 1.74 (m, 1H), 1.49 – 1.37 (m, 1H), 1.35 – 1.21 (m, 8H), 1.19 (d, $J = 6.8$ Hz, 3H), 0.90 – 0.80 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 204.8, 136.9, 132.9, 128.7, 128.4, 40.7, 33.9, 31.8, 29.5, 27.5, 22.7, 17.4, 14.2. EI-MS: m/z 218.2.



Cyclopentylphenylmethanone⁷ (7) CAS: 5422-88-8

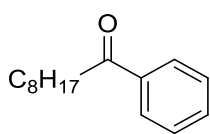
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (34 mg, 64%). $R_f = 0.75$ (7:1 *i*-hexane:EtOAc). 1H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.95 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.41 (m, 2H), 3.72 (p, $J = 7.9$ Hz, 1H), 1.98 – 1.87 (m, 4H), 1.80 – 1.61 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.0, 137.1, 132.8, 128.6, 128.6, 46.5, 30.1, 26.5. EI-MS: m/z 174.1.



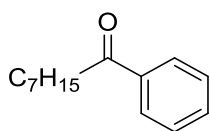
Phenyl(1-tosylpiperidin-4-yl)methanone (8) CAS: 922504-26-5

Synthesized according to the general procedure. Isolated as a yellow solid (63 mg, 60%). $R_f = 0.42$ (3:1 *i*-hexane:ethyl acetate + 1% triethylamine). 1H NMR (400 MHz, $CDCl_3$) δ 7.87 – 7.82 (m, 2H), 7.69 – 7.64 (m, 2H), 7.57 – 7.52 (m, 1H), 7.46 – 7.40 (m, 2H), 7.37 – 7.32 (m, 2H), 3.80 – 3.73 (m, 2H), 3.23 – 3.14 (m, 1H), 2.57 – 2.48 (m, 2H), 2.45 (s, 3H), 1.98 – 1.81 (m, 4H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 201.5, 143.8, 135.7, 133.4, 133.2, 129.8, 128.9, 128.3, 127.9, 45.7, 42.4, 28.0, 21.7. ESI-MS: 344.1 HRMS found: 344.1315 calc: 344.1309. HPLC purity > 99%.

1-Phenylnonan-1-one⁸ (9) CAS: 6008-36-2

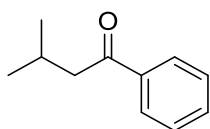


Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (36 mg, 55 % from 1-iodooctane; 20 mg and 27 mg, 30% and 40% from 1-bromooctane (24 h and 55 h irradiation)). $R_f = 0.44$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.07 – 7.83 (m, 2H), 7.58 – 7.52 (m, 1H), 7.48 – 7.41 (m, 2H), 2.96 (t, $J = 7.3$ Hz, 2H), 1.80 – 1.65 (m, 2H), 1.42 – 1.20 (m, 10H), 0.91 – 0.83 (m, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 200.8, 137.2, 133.0, 128.7, 128.2, 38.8, 32.0, 29.6, 29.5, 29.3, 24.5, 22.8, 14.3. EI-MS: m/z 218.2.



1-Phenyloctan-1-one⁹ (10) CAS: 1674-37-8

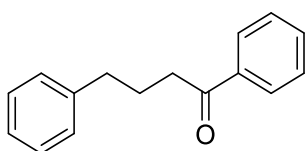
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (44 mg, 71% from 1-iodoheptane, 25 mg, 42% from 1-bromoheptane). $R_f = 0.37$ (29:1 *i*-Hexane:EtOAc). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.98 – 7.94 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.41 (m, 2H), 2.96 (t, $J = 7.4$ Hz, 2H), 1.78 – 1.68 (m, 2H), 1.43 – 1.23 (m, 8H), 0.92 – 0.84 (m, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 200.8, 137.2, 133.0, 128.7, 128.2, 38.8, 31.9, 29.5, 29.3, 24.5, 22.8, 14.3. EI-MS: m/z 204.1.



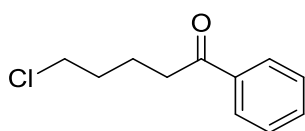
3-Methyl-1-phenylbutan-1-one¹⁰ (11) CAS: 582-62-7

Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (24 mg, 50%). $R_f = 0.16$ (1% EtOAc in *i*-Hexane). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.97 – 7.93 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 2.84 (d, $J = 6.9$ Hz, 2H), 2.30 (dp, $J = 13.5, 6.7$ Hz, 1H), 1.00 (d, $J = 6.7$ Hz, 6H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 200.4, 137.5, 132.3, 128.7, 128.2, 47.7, 25.3, 22.9. EI-MS: m/z 162.1.

1,4-Diphenylbutan-1-one⁸ (12) CAS: 5407-91-0

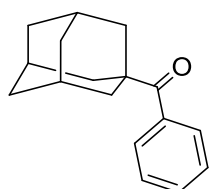


Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (45 mg, 67% from 3-iodo-1-phenylpropane; 28 mg and 33 mg, 41% and 48% from 3-bromo-1-phenylpropane (24 h and 55 h irradiation)). $R_f = 0.31$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 – 7.91 (m, 2H), 7.58 – 7.53 (m, 1H), 7.49 – 7.42 (m, 2H), 7.33 – 7.27 (m, 2H), 7.25 – 7.19 (m, 3H), 2.99 (t, $J = 7.3$ Hz, 2H), 2.73 (t, $J = 7.4$ Hz, 2H), 2.15 – 2.05 (m, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 200.3, 141.8, 137.1, 133.1, 128.7, 128.7, 128.5, 128.1, 126.1, 37.8, 35.3, 25.8. EI-MS: m/z 224.1.



5-Chloro-1-phenylpentan-1-one¹¹ (13) CAS: 942-93-8

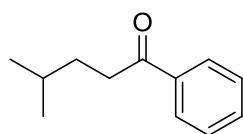
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a white solid (42 mg, 71% from 1-chloro-4-iodobutane; 36 mg, 61% from 1-chloro-4-bromobutane). $R_f = 0.73$ (7:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.98 – 7.93 (m, 2H), 7.60 – 7.53 (m, 1H), 7.50 – 7.43 (m, 2H), 3.64 – 3.54 (m, 2H), 3.07 – 2.97 (m, 2H), 1.98 – 1.82 (m, 4H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 199.8, 137.0, 133.2, 128.8, 128.1, 44.9, 37.7, 32.2, 21.6. EI-MS: m/z 196.1, 198.1.



((3r,5r,7r)-Adamantan-1-yl)(phenyl)methanone¹² (14) CAS: 31919-47-8

Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a white solid (45 mg, 61% from 1-iodoadamantane, 25 mg, 32% from 1-bromoadamantane). $R_f = 0.28$ (29:1 *i*-

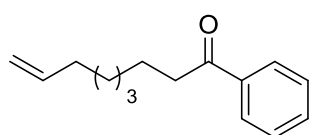
hexane:ethyl acetate). ^1H NMR (400 MHz, CDCl_3) δ 7.56 – 7.52 (m, 2H), 7.46 – 7.36 (m, 3H), 2.10 – 2.04 (m, 3H), 2.03 – 1.98 (m, 6H), 1.81 – 1.69 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 210.4, 139.7, 130.3, 128.1, 127.2, 47.0, 39.2, 36.7, 28.3. EI-MS: m/z 240.2.



4-Methyl-1-phenylpentan-1-one⁹ (18) CAS: 2050-07-9

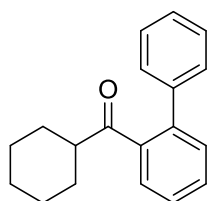
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (18 mg, 34%). R_f = 0.40 (29:1 *i*-hexane:ethyl acetate). ^1H NMR (400 MHz, CDCl_3) δ 7.98 – 7.94 (m, 2H), 7.58 – 7.53 (m, 1H), 7.49 – 7.43 (m, 2H), 2.99 – 2.94 (m, 2H), 1.66 – 1.61 (m, 3H), 0.96 – 0.93 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 201.0, 137.2, 133.0, 128.7, 128.2, 36.8, 33.4, 28.0, 22.6. EI-MS: m/z 176.1.

1-Phenylnon-8-en-1-one (19) CAS: 148056-64-8



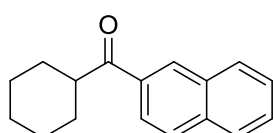
Synthesized according to the general procedure. Isolated as a colorless oil (17 mg, 26%). R_f = 0.42 (29:1 *i*-hexane:ethyl acetate). ^1H NMR (400 MHz, CDCl_3) δ 7.98 – 7.93 (m, 2H), 7.58 – 7.52 (m, 1H), 7.49 – 7.43 (m, 2H), 5.86 – 5.74 (m, 1H), 5.01 – 4.91 (m, 2H), 2.96 (t, J = 7.5 Hz, 2H), 2.07 – 2.01 (m, 2H), 1.78 – 1.70 (m, 2H), 1.44 – 1.34 (m, 6H). ^{13}C NMR (101 MHz, CDCl_3) δ 200.7, 139.2, 137.2, 133.0, 128.7, 128.2, 114.4, 38.7, 33.9, 29.3, 29.1, 28.9, 24.4. EI-MS: m/z 216.1 HRMS found: 216.1520 calc: 216.1514.

[1,1'-Biphenyl]-2-yl(cyclo-hexyl)methanone (20) CAS: 963-92-8

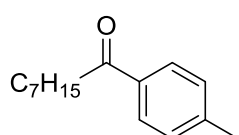


Synthesized according to the general procedure. Isolated as a colorless oil (34 mg, 43%). R_f = 0.41 (29:1 *i*-hexane:ethyl acetate). ^1H NMR (400 MHz, CDCl_3) δ 7.52 – 7.47 (m, 1H), 7.44 – 7.36 (m, 6H), 7.35 – 7.30 (m, 2H), 2.10 (tt, J = 11.4, 3.3 Hz, 1H), 1.64 – 1.54 (m, 2H), 1.53 – 1.45 (m, 3H), 1.29 – 1.16 (m, 2H), 1.13 – 1.00 (m, 1H), 0.93 – 0.80 (m, 2H). ^{13}C NMR (101 MHz, CDCl_3) δ 211.8, 140.8, 140.7, 139.8, 130.1, 129.9, 128.9, 128.6, 128.0, 127.7, 127.3, 50.3, 29.0, 25.7, 25.6. EI-MS: m/z 264.1 HRMS found: 265.1597 calc: 265.1592.

Cyclohexyl(naphthalen-2-yl)-methanone (21) CAS: 10404-26-9



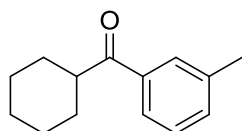
Synthesized according to the general procedure. Isolated as a white solid (43 mg, 59%). R_f = 0.33 (29:1 *i*-hexane:ethyl acetate). ^1H NMR (400 MHz, CDCl_3) δ 8.46 (d, J = 1.7 Hz, 1H), 8.02 (dd, J = 8.6, 1.8 Hz, 1H), 8.00 – 7.95 (m, 1H), 7.92 – 7.85 (m, 2H), 7.62 – 7.52 (m, 2H), 3.43 (tt, J = 11.3, 3.3 Hz, 1H), 1.99 – 1.84 (m, 4H), 1.82 – 1.73 (m, 1H), 1.63 – 1.39 (m, 4H), 1.37 – 1.24 (m, 1H). ^{13}C NMR (101 MHz, CDCl_3) δ 204.0, 135.6, 133.8, 132.7, 129.7, 129.7, 128.6, 128.4, 127.9, 126.8, 124.5, 45.8, 29.7, 26.1, 26.1. EI-MS: m/z 238.1 HRMS found: 239.1436 calc: 239.1436.



1-(*p*-Tolyl)octan-1-one¹³ (22) CAS: 2789-44-8

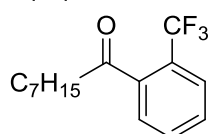
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a white solid (52 mg, 79%). R_f = 0.40 (29:1 *i*-hexane:ethyl acetate). ^1H NMR (400 MHz, CD_3OD) δ 7.89 – 7.85 (m, 2H), 7.32 – 7.27 (m, 2H), 2.97 (t, J = 7.3 Hz, 2H), 2.40 (s, 3H), 1.73 – 1.63 (m, 2H), 1.40 – 1.26 (m, 8H), 0.93 – 0.87 (m, 3H). ^{13}C NMR (101 MHz, CD_3OD) δ 202.6, 145.3, 135.8, 130.3, 129.3, 39.4, 32.9, 30.4, 30.3, 25.7, 23.7, 21.6, 14.4. EI-MS: m/z 218.2.

Cyclohexyl(*m*-tolyl)methanone¹⁴ (23) CAS: 3277-78-9

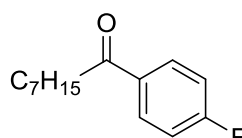


Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (39 mg, 64%). $R_f = 0.42$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.76 – 7.71 (m, 2H), 7.36 – 7.32 (m, 2H), 3.29 – 3.20 (m, 1H), 2.41 (s, 3H), 1.92 – 1.81 (m, 4H), 1.78 – 1.70 (m, 1H), 1.56 – 1.23 (m, 5H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 204.3, 138.5, 136.6, 133.6, 128.9, 128.6, 125.6, 45.8, 29.6, 26.1, 26.0, 21.6. EI-MS: m/z 202.1.

1-(2-(Trifluoromethyl)-phenyl)octan-1-one (24) CAS: 1778855-78-9

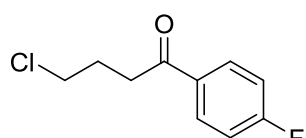


Synthesized according to the general procedure. Isolated as a colorless oil (46 mg, 56%). $R_f = 0.34$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.73 – 7.68 (m, 1H), 7.62 – 7.51 (m, 2H), 7.42 – 7.38 (m, 1H), 2.83 (t, $J = 7.4$ Hz, 2H), 1.70 (p, $J = 7.4$ Hz, 2H), 1.38 – 1.25 (m, 8H), 0.91 – 0.84 (m, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 204.9, 140.8 (q, $J = 1.9$ Hz), 131.9, 130.0, 127.0, 127.0 (q, $J = 32.8$ Hz), 126.8 (q, $J = 5.2$ Hz), 123.8 (q, $J = 274.8$ Hz), 43.4, 31.8, 29.2, 29.1, 23.9, 22.8, 14.2. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -58.1. EI-MS: m/z 272.1 HRMS found: 273.1460 calc: 273.1461.



1-(4-Fluorophenyl)octan-1-one¹⁵ (25) CAS: 111829-15-3

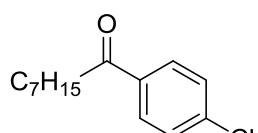
Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a white solid (47 mg, 71%). $R_f = 0.36$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.95 – 7.88 (m, 2H), 7.09 – 7.02 (m, 2H), 2.86 (t, $J = 7.3$ Hz, 2H), 1.65 (q, $J = 7.4$ Hz, 2H), 1.33 – 1.17 (m, 8H), 0.85 – 0.78 (m, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 199.1, 165.8 (d, $J = 254.2$ Hz), 133.6 (d, $J = 3.1$ Hz), 130.8 (d, $J = 9.3$ Hz), 115.7 (d, $J = 21.7$ Hz), 38.7, 31.9, 29.5, 29.3, 24.5, 22.8, 14.2. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -105.7 – -105.8 (m). EI-MS: m/z 222.1.



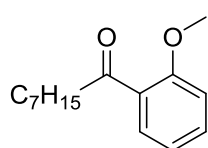
4-Chloro-1-(4-fluorophenyl)butan-1-one¹⁶ (26) CAS: 3874-54-2

Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a colorless oil (41 mg, 68%). $R_f = 0.27$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 8.05 – 7.98 (m, 2H), 7.18 – 7.11 (m, 2H), 3.68 (t, $J = 6.2$ Hz, 2H), 3.16 (t, $J = 7.0$ Hz, 2H), 2.23 (m, 2H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 197.5, 165.9 (d, $J = 254.9$ Hz), 133.3 (d, $J = 3.2$ Hz), 130.8 (d, $J = 9.3$ Hz), 115.9 (d, $J = 21.8$ Hz), 44.8, 35.3, 26.8. $^{19}\text{F NMR}$ (376 MHz, CDCl_3) δ -105.0 – -105.1 (m). EI-MS: m/z 200.0.

1-(4-Chlorophenyl)octan-1-one (27) CAS: 7295-52-5



Synthesized according to the general procedure. Isolated as a white solid (60 mg, 83%). $R_f = 0.49$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.92 – 7.87 (m, 2H), 7.46 – 7.40 (m, 2H), 2.95 – 2.89 (m, 2H), 1.76 – 1.66 (m, 2H), 1.38 – 1.23 (m, 8H), 0.91 – 0.84 (m, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3) δ 199.5, 139.4, 135.5, 129.6, 129.0, 38.8, 31.8, 29.4, 29.3, 24.4, 22.8, 14.2. EI-MS: m/z 238.1, 240.1 HRMS found: 239.1195 calc: 239.1197.

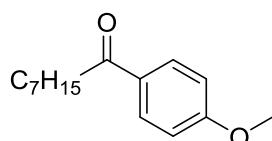


1-(2-Methoxyphenyl)octan-1-one (28) CAS: 153644-77-0

Synthesized according to the general procedure. Isolated as a white solid (19 mg, 26%). $R_f = 0.29$ (29:1 *i*-hexane:ethyl acetate). $^1\text{H NMR}$ (400 MHz, CDCl_3) δ 7.64 (dd, $J = 7.7, 1.8$ Hz, 1H), 7.47 – 7.41 (m, 1H), 7.01 – 6.93 (m, 2H), 3.89 (s, 3H), 2.95 (t, $J = 7.3$ Hz, 2H), 1.71 – 1.62 (m, 2H), 1.37 – 1.23 (m, 8H), 0.91 – 0.84 (m, 3H). $^{13}\text{C NMR}$ (101 MHz, CDCl_3)

δ 203.6, 158.4, 133.2, 130.3, 129.0, 120.8, 111.6, 55.6, 43.9, 31.9, 29.5, 29.3, 24.6, 22.8, 14.3. EI-MS: m/z 234.1 HRMS found: 235.1700 calc: 235.1698.

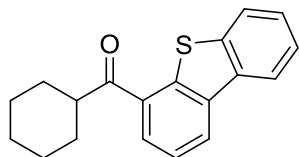
1-(4-Methoxyphenyl)octan-1-one¹⁷ (29) CAS: 62170-25-6



Synthesized according to the general procedure. Spectral data were in agreement with literature values. Isolated as a white solid (57 mg, 80%). R_f = 0.30 (29:1 *i*-hexane:ethyl acetate). ¹H NMR (400 MHz, CDCl₃) δ 7.97 – 7.91 (m, 2H), 6.95 – 6.90 (m, 2H), 3.86 (s, 3H), 2.90 (t, J = 7.6 Hz, 2H), 1.76 – 1.66 (m, 2H), 1.39 – 1.24 (m, 8H), 0.91 – 0.85 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 199.4, 163.4, 130.5, 130.3, 113.8, 55.6, 38.5, 31.9, 29.5, 29.3, 24.8, 22.8, 14.2. EI-MS: m/z 234.2.

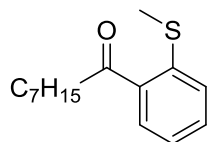
Compound **29** was also synthesized using another CO-surrogate. Reactants and reagents were added according to the general procedure apart from the reactants in C_{CO}. Instead of Mo(CO)₆ followed by DBU, 0.16 mL of H₂SO₄ (10 eq) was added to C_{CO}, followed by capping of the vial. No solvent was used in C_{CO}. After 5 minutes of N₂/vacuum sparging, 0.12 mL of HCOOH (10.5 eq) was added and the double chamber system was positioned in a Dry-Syn heating block with C_{CO} heated at 70°C and C_{rxn} held at room temperature whilst subjected to visible light for 24 h. Work-up and purification according to the general procedure. Spectral data were in agreement with literature values. Isolated as a white solid (19 mg, 27%). R_f = 0.33 (29:1 *i*-hexane:ethyl acetate). ¹H NMR (400 MHz, CDCl₃) δ 7.97 – 7.91 (m, 2H), 6.96 – 6.90 (m, 2H), 3.87 (s, 3H), 2.94 – 2.86 (m, 2H), 1.72 (p, J = 7.5 Hz, 2H), 1.38 – 1.27 (m, 8H), 0.90 – 0.85 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 199.4, 163.4, 130.5, 130.4, 113.8, 55.6, 38.5, 31.9, 29.6, 29.3, 24.8, 22.8, 14.2. EI-MS: m/z 234.2.

Cyclohexyl(dibenzo[b,d]thio-phen-4-yl)methanone (30)



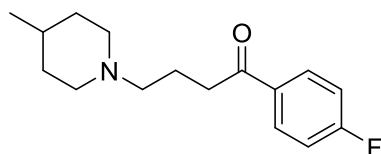
Synthesized according to the general procedure. Isolated as an oil (55 mg, 62%). R_f = 0.40 (0.5% ethyl acetate in *n*-pentane). ¹H NMR (400 MHz, CDCl₃) δ 8.33 (dd, J = 7.7, 1.1 Hz, 1H), 8.15 – 8.08 (m, 2H), 7.91 – 7.86 (m, 1H), 7.53 (t, J = 7.7 Hz, 1H), 7.46 – 7.39 (m, 2H), 3.43 (tt, J = 11.5, 3.3 Hz, 1H), 1.95 – 1.80 (m, 4H), 1.75 – 1.68 (m, 1H), 1.58 (qd, J = 12.8, 3.2 Hz, 2H), 1.39 (qt, J = 12.7, 3.3 Hz, 2H), 1.27 (tt, J = 12.5, 3.3 Hz, 1H). ¹³C NMR (101 MHz, CDCl₃) δ 203.2, 142.6, 140.0, 137.7, 133.9, 129.6, 128.3, 127.3, 126.0, 124.6, 124.2, 122.9, 121.5, 45.5, 29.9, 26.1, 26.1. EI-MS: m/z 294.1 HRMS found: 295.1150 calc: 295.1130.

1-(2-(Methylthio)phenyl)octan-1-one (31) CAS: 1778855-66-5



Synthesized according to the general procedure. Isolated as a colorless oil (40 mg, 53%). R_f = 0.21 (29:1 *i*-hexane:ethyl acetate). ¹H NMR (400 MHz, CDCl₃) δ 7.81 (dd, J = 7.8, 1.5 Hz, 1H), 7.48 – 7.43 (m, 1H), 7.34 – 7.31 (m, 1H), 7.21 – 7.16 (m, 1H), 2.94 (t, J = 7.3 Hz, 2H), 2.43 (s, 3H), 1.77 – 1.69 (m, 2H), 1.38 – 1.25 (m, 8H), 0.90 – 0.84 (m, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 202.0, 142.3, 134.8, 132.1, 130.2, 125.3, 123.6, 40.2, 31.8, 29.4, 29.3, 24.6, 22.8, 16.2, 14.2. EI-MS: m/z 250.1 HRMS found: 251.1472 calc: 251.1470.

1-(4-Fluorophenyl)-4-(4-methylpiperidin-1-yl)butan-1-one (Melperone) CAS: 3575-80-2



26 (0.20 mmol), 4-methylpiperidine (2.0 eq), KI (0.2 eq) and NaHCO₃ (2.0 eq) were dissolved in toluene (5 mL). The reaction mixture was heated to 100°C for 22 h. After completion, the reaction mixture was concentrated *in vacuo* and purified with silica column chromatography using a gradient (3:1 *i*-hexane:ethyl acetate + 1% triethylamine to ethyl acetate + 1% triethylamine). Isolated as a red solid (29 mg, 55%). R_f = 0.16

(3:1 *i*-hexane:ethyl acetate + 1% trimethylamine). ¹H NMR (400 MHz, CDCl₃) δ 8.02 – 7.96 (m, 2H), 7.15 – 7.08 (m, 2H), 2.95 (t, *J* = 7.2 Hz, 2H), 2.88 – 2.80 (m, 2H), 2.36 (t, *J* = 7.1 Hz, 2H), 1.97 – 1.84 (m, 4H), 1.61 – 1.54 (m, 2H), 1.38 – 1.23 (m, 1H), 1.21 – 1.08 (m, 2H), 0.89 (d, *J* = 6.5 Hz, 3H). ¹³C NMR (101 MHz, CDCl₃) δ 198.6, 166.7 (d, *J* = 254.1 Hz), 133.6 (d, *J* = 2.7 Hz), 130.7 (d, *J* = 9.3 Hz), 115.6 (d, *J* = 21.7 Hz), 58.2, 54.0, 36.4, 34.3, 30.8, 21.9 (2 carbons). MS-ESI [M+H⁺]: *m/z* 264.2. HPLC purity > 99%.

Control experiments

To learn more about the reaction, a set of control experiments were conducted (table S2).

Table S2. Control experiments for the visible-light mediated carbonylative Suzuki-Miyaura cross-coupling

Reaction scheme: Cyclohexane-X + Phenyl-B(OH)₂ (2) → Cyclohexane-CO-Phenyl (3)

Chamber_{rxn}: catalyst, additive, K₂CO₃, benzene/water 2:1, blue/white LEDs, rt, 24 h
 Chamber_{CO}: Mo(CO)₆, DBU, 70°C, MeCN

X = I entries 1-3, 5-6
 X = Br entries 4 and 7

Entry	Catalyst	Additive	Irradiation	Outcome ^a
1	-	-	LED	No product
2	Pd(PPh ₃) ₄	TEMPO (1 eq)	LED	No product
3	-	TEMPO (1 eq)	LED	No product
4	Pd(PPh ₃) ₄	TEMPO (1 eq)	LED	No product
5 ^b	Pd(PPh ₃) ₄	-	-	Trace
6	Pd(PPh ₃) ₄	-	Ambient light	Trace
7 ^b	Pd(PPh ₃) ₄	TEMPO (1 eq)	-	No product
8 ^b	Pd(PPh ₃) ₄	-	-	No product
9 ^c	Pd(PPh ₃) ₄	-	LED	Trace (of coupling product)

Reaction conditions: As in entry S63, table S1. ^a Outcome determined by GCMS- and LCMS-analysis. ^b Reaction performed in the dark. ^c No Mo(CO)₆.

Pressure measurement

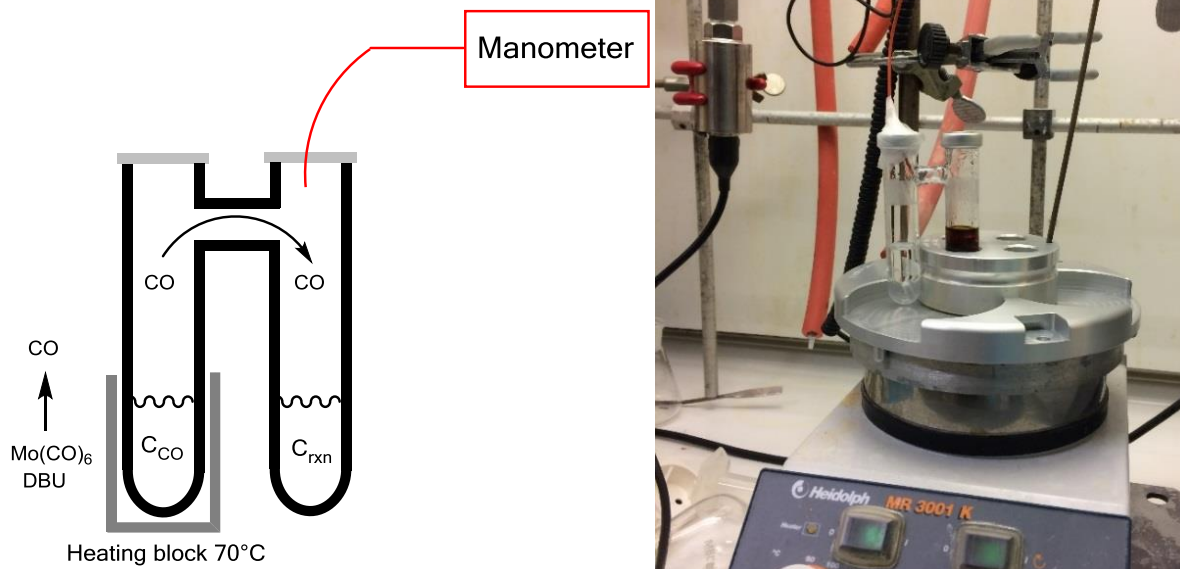


Figure S2. Pressure measurement set-up. To the left, a schematic illustration and to the right, the actual set-up.

To C_{rxn} was added benzene/water 2:1 (4 mL) and to C_{CO} was added $Mo(CO)_6$ (0.75 mmol) followed by acetonitrile (4 mL). After capping, both chambers were subjected to 5 minute of N_2 /vacuum sparging. C_{rxn} was pierced with a capillary connected to a manometer and DBU (1.5 mmol) was added to C_{CO} and the H-tube was positioned in a Dry-Syn heating block with C_{CO} heated at $70^\circ C$ (figure S2). The temperature in C_{rxn} was measured to $34^\circ C$. The resulting pressure increase due to the CO release was monitored at regular time points (see table S3 and figure S3). Maximum pressure measured was 2.6 bar, reached within 20-25 minutes of the start of the reaction.

Table S3. Pressure measurements.

Time (min)	Pressure		Average pressure (bar)
	Measurement 1 (bar)	Measurement 2 (bar)	
0	1,1	1,1	1,1
5	1,7	1,8	1,75
10	2,3	2,3	2,3
15	2,5	2,5	2,5
20	2,5	2,6	2,55
25	2,6	2,6	2,6
30	2,6	2,6	2,6
40	2,6	2,6	2,6
50	2,6	2,6	2,6
60	2,6	2,5	2,55
210	2,5	2,2	2,35

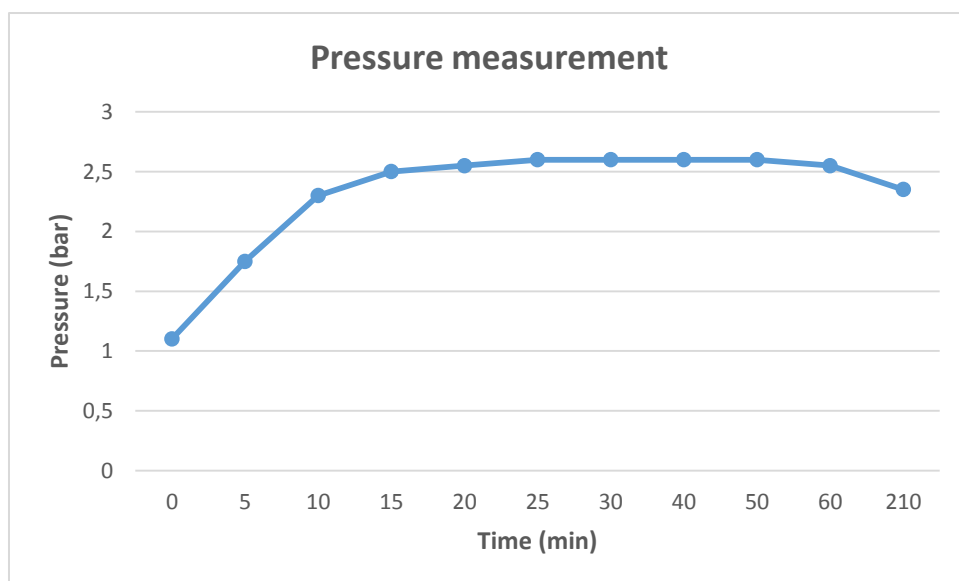
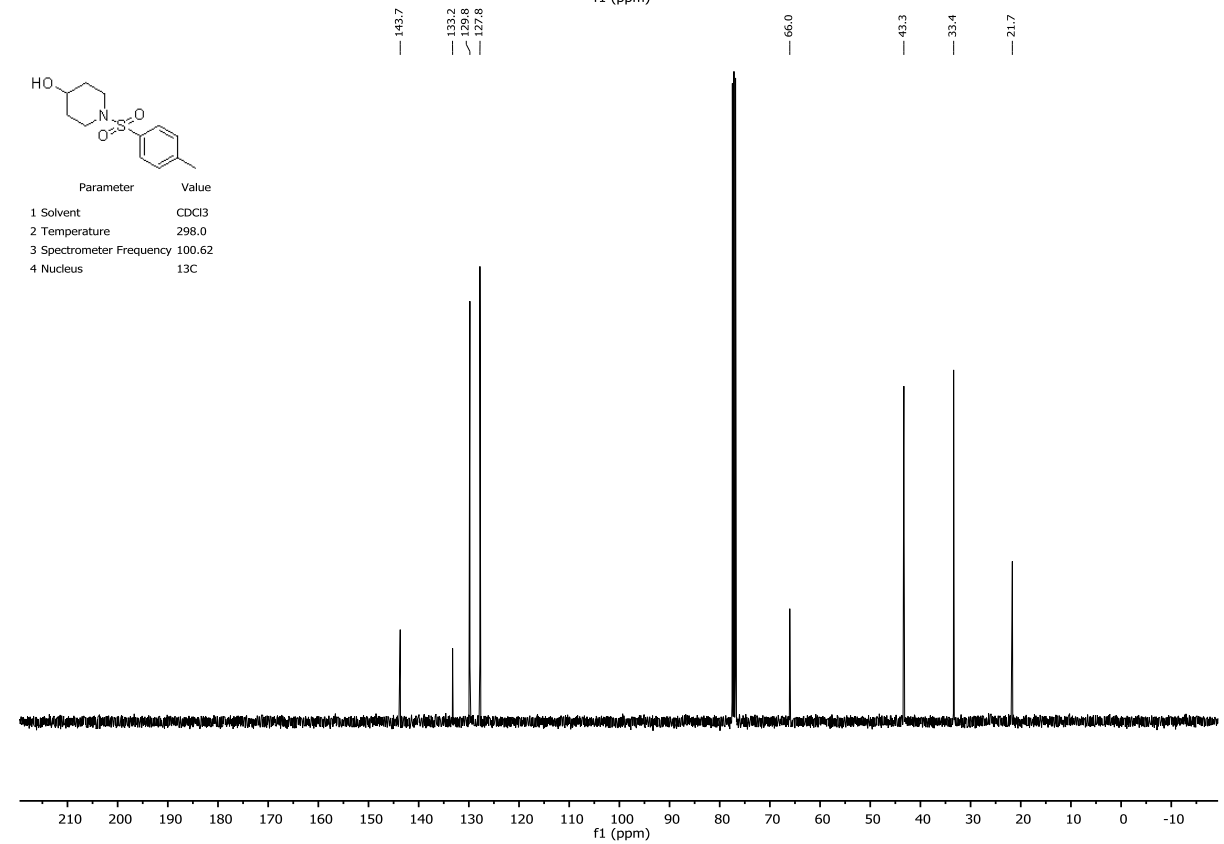
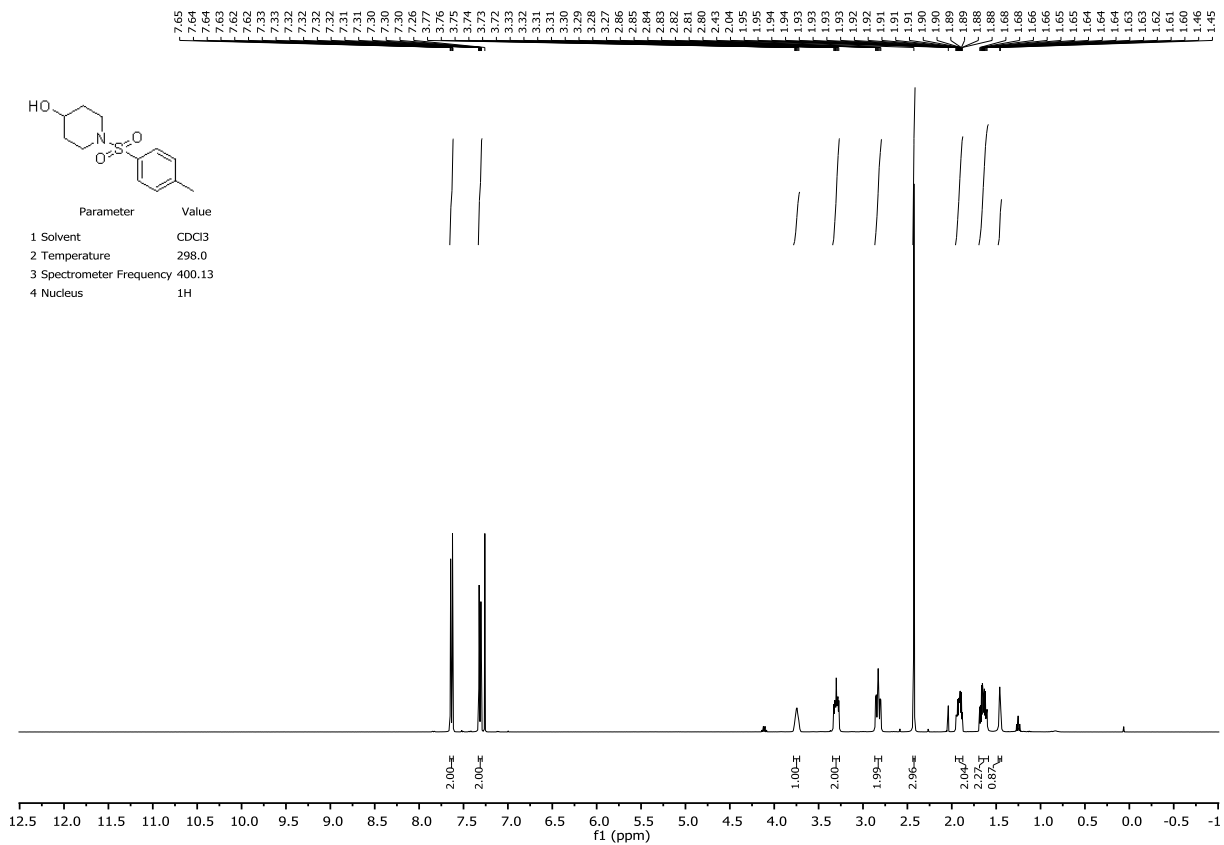


Figure S3. Graphic representation of pressure measurements. Maximum pressure reached within 20-25 minutes of the start of the reaction.

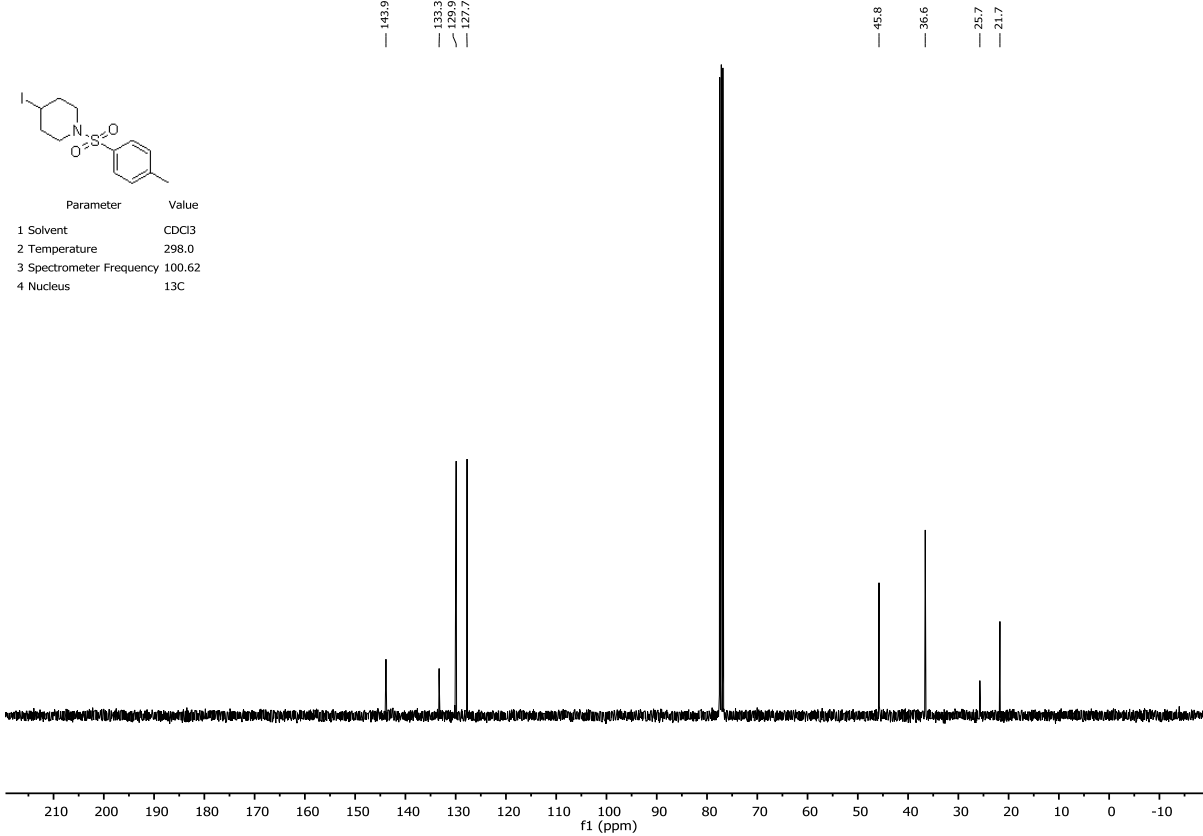
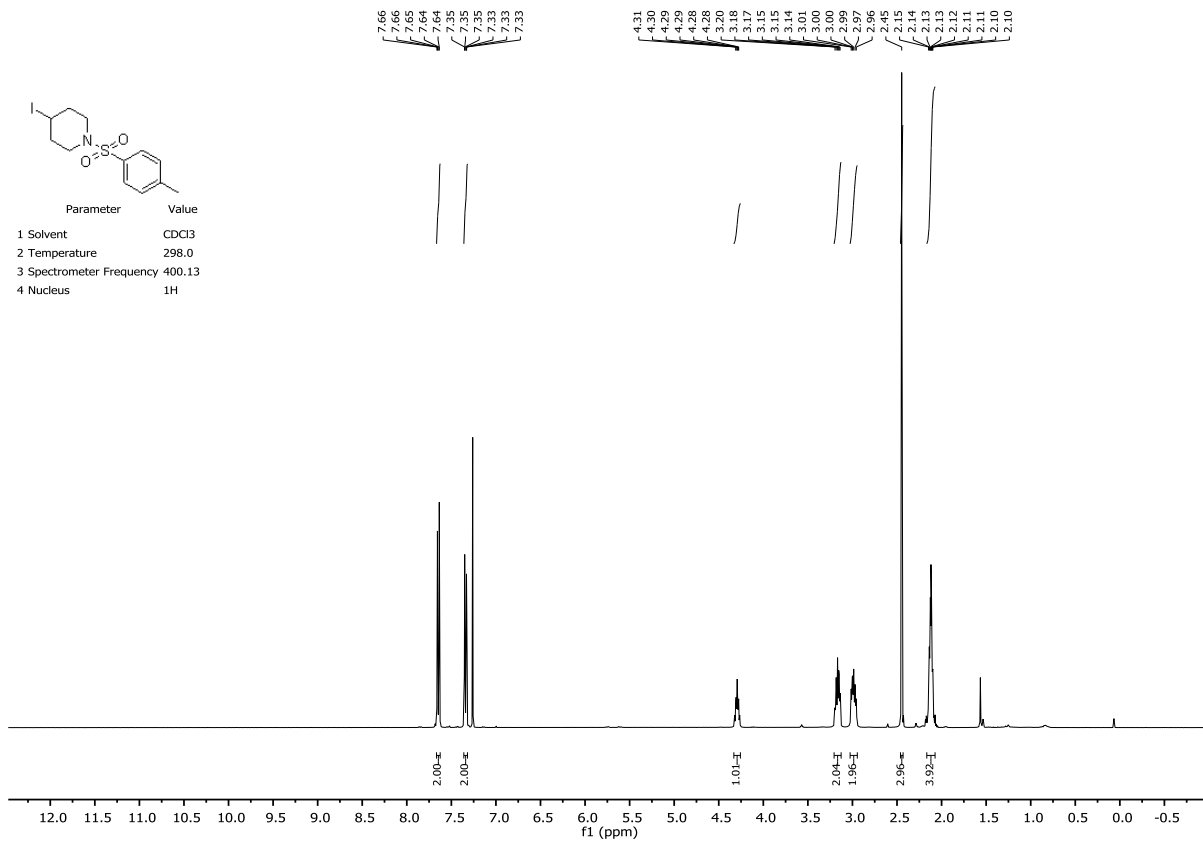
References

- (1) Gonnard, L.; Guérintot, A.; Cossy, J. *Chem. - A Eur. J.* **2015**, *21*, 12797–12803.
- (2) Ellwood, A. R.; Porter, M. J. *J. Org. Chem.* **2009**, *74*, 7982–7985.
- (3) Vurchio, C.; Cordero, F. M.; Faggi, C.; Macchi, B.; Frezza, C.; Grelli, S.; Brandi, A. *Tetrahedron* **2015**, *71*, 5806–5813.
- (4) Kawaguchi, S. I.; Ogawa, A. *Org. Lett.* **2010**, *12*, 1893–1895.
- (5) Hsieh, J. C.; Chen, Y. C.; Cheng, A. Y.; Tseng, H. C. *Org. Lett.* **2012**, *14*, 1282–1285.
- (6) Sumino, S.; Ui, T.; Ryu, I. *Org. Lett.* **2013**, *15*, 2011–2014.
- (7) Li, L.; Cai, P.; Guo, Q.; Xue, S. *J. Org. Chem.* **2008**, *73*, 3516–3522.
- (8) Vautravers, N. R.; Regent, D. D.; Breit, B. *Chem. Commun.* **2011**, *47*, 6635–6637.
- (9) Cho, C. S. *J. Mol. Catal. A Chem.* **2005**, *240*, 55–60.
- (10) Malosh, C. F.; Ready, J. M. *J. Am. Chem. Soc.* **2004**, *126*, 10240–10241.
- (11) Komissarov, V. V.; Kritzyn, A. M. *Russ. J. Bioorganic Chem.* **2010**, *36*, 477–487.
- (12) Lo Fiego, M. J.; Lockhart, M. T.; Chopra, A. B. *J. Organomet. Chem.* **2009**, *694*, 3674–3678.
- (13) Fausett, B. W.; Liebeskind, L. S. *J. Org. Chem.* **2005**, *70*, 4851–4853.
- (14) Gooßen, L. J.; Mamone, P.; Oppel, C. *Adv. Synth. Catal.* **2011**, *353*, 57–63.
- (15) Döbele, M.; Vanderheiden, S.; Jung, N.; Bräse, S. *Angew. Chemie - Int. Ed.* **2010**, *49*, 5986–5988.
- (16) Gowda, M. S.; Pande, S. S.; Ramakrishna, R. A.; Prabhu, K. R. *Org. Biomol. Chem.* **2011**, *9*, 5365–5368.
- (17) Ruan, J.; Saidi, O.; Iggo, J. A.; Xiao, J. *J. Am. Chem. Soc.* **2008**, *130*, 10510–10511.

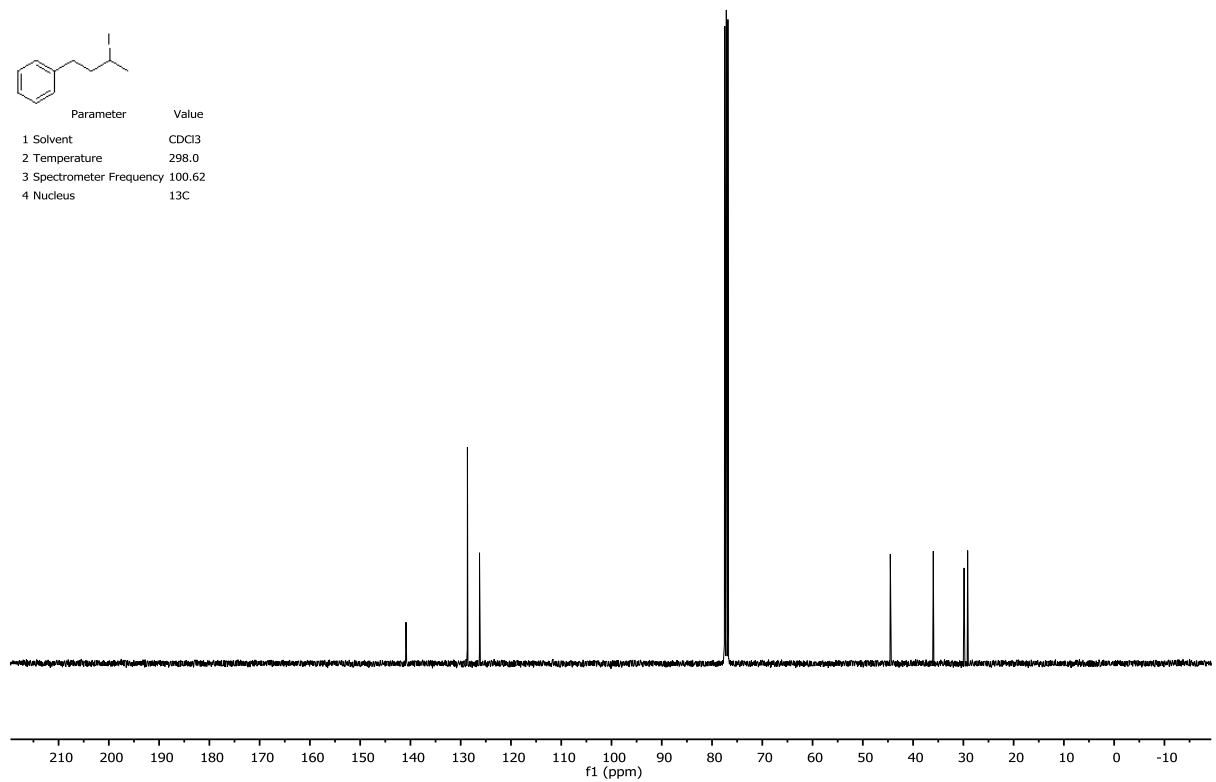
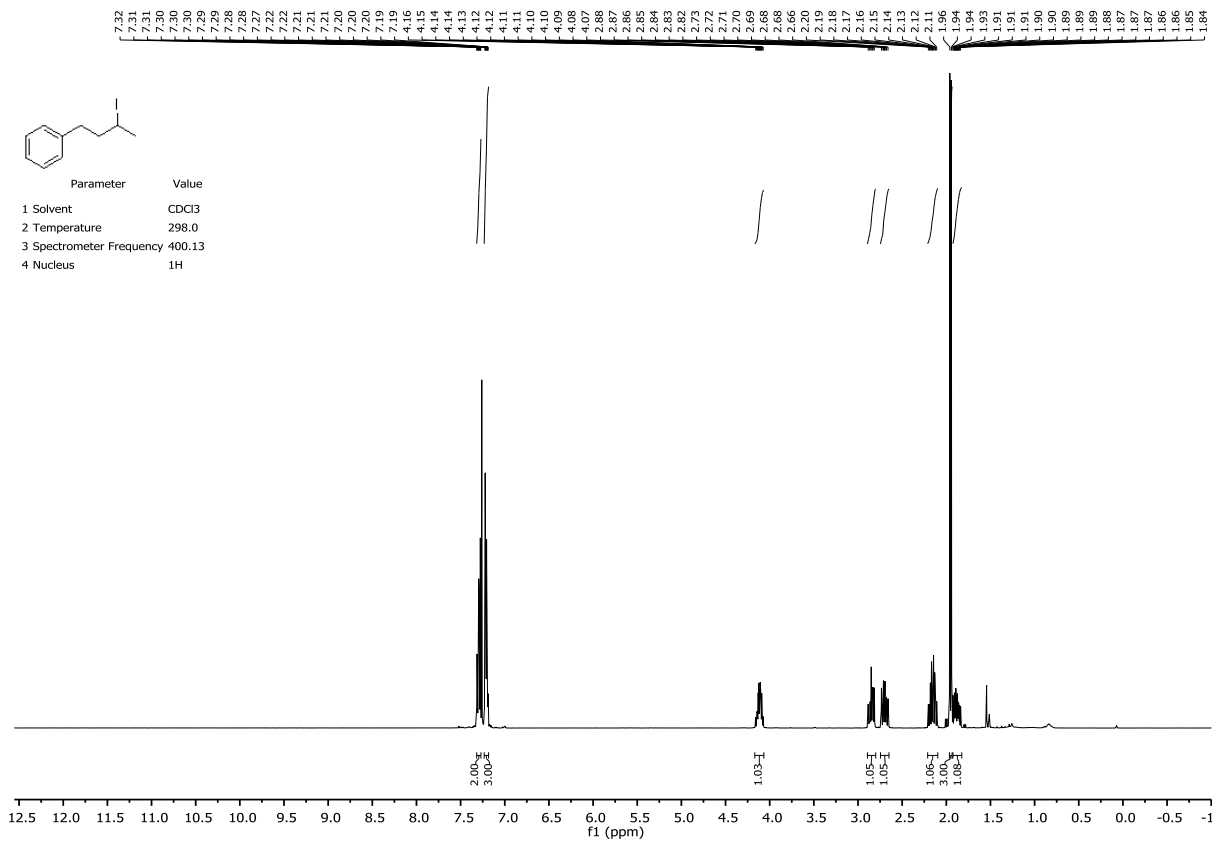
NMR spectra



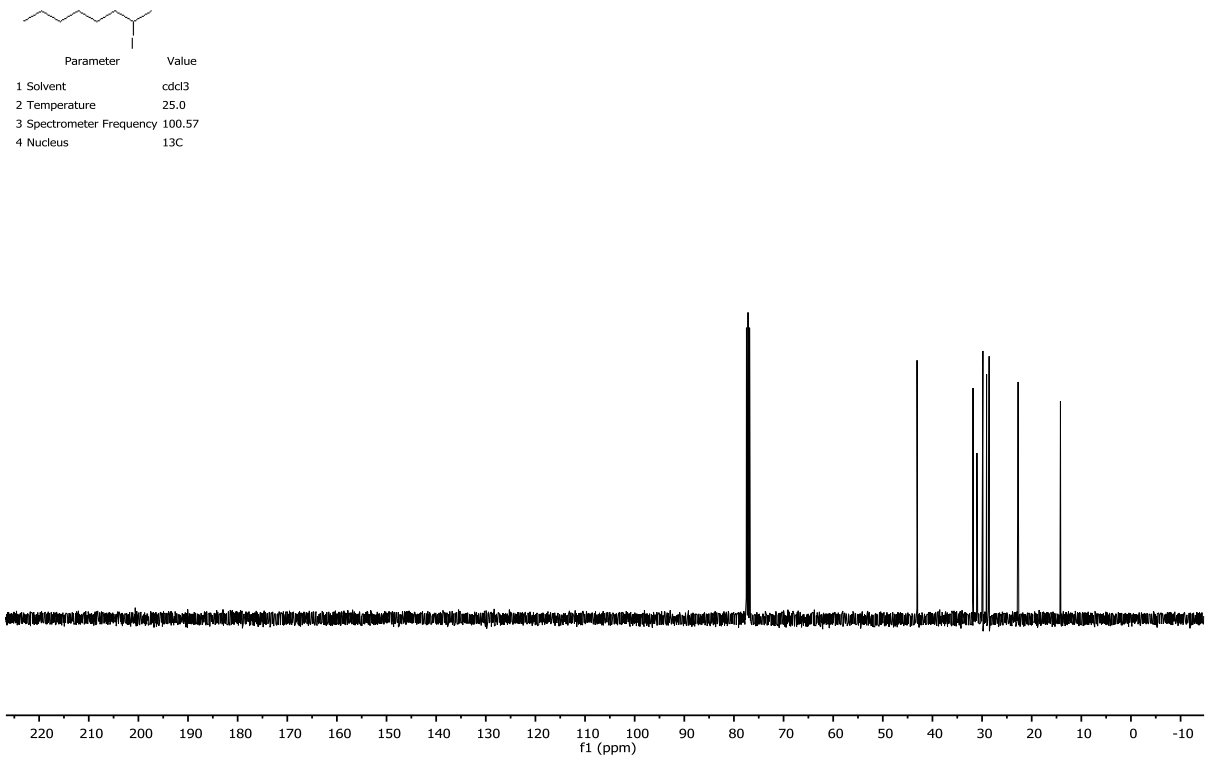
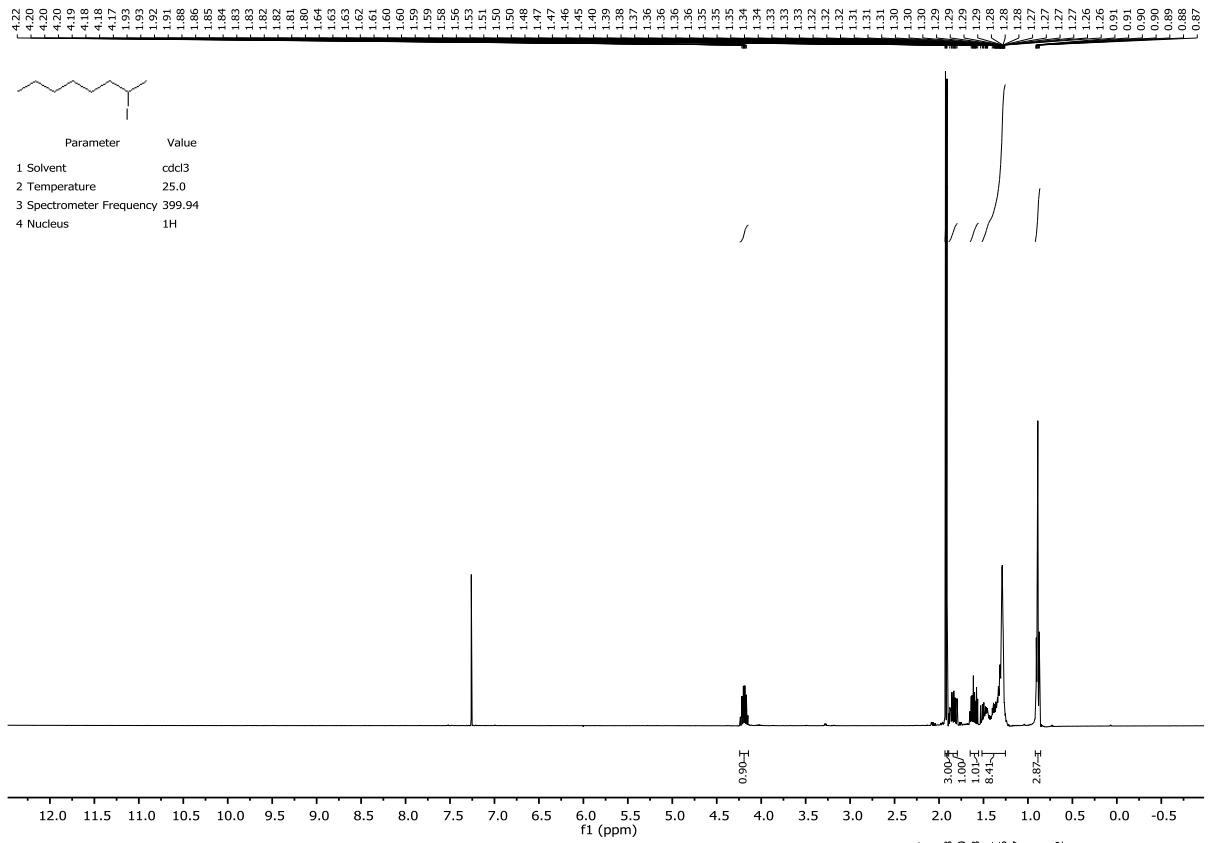
1-[(4-Methylphenyl)sulfonyl]piperidin-4-ol¹ CAS: 80213-12-3



4-Iodo-1-[(4-methylphenyl)sulfonyl]piperidine¹ CAS: 289890-80-8

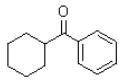


3-Iodobutylbenzene^{2,3} CAS: 59456-20-1

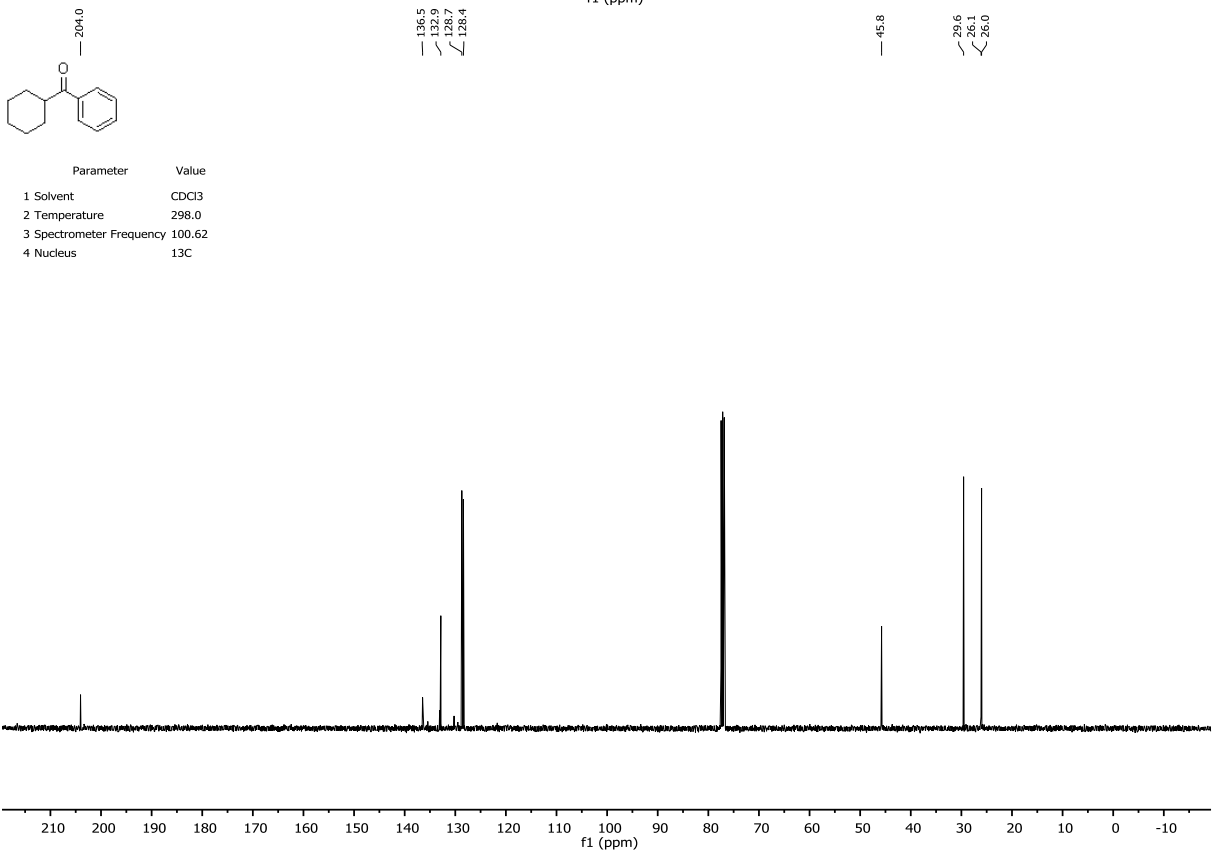
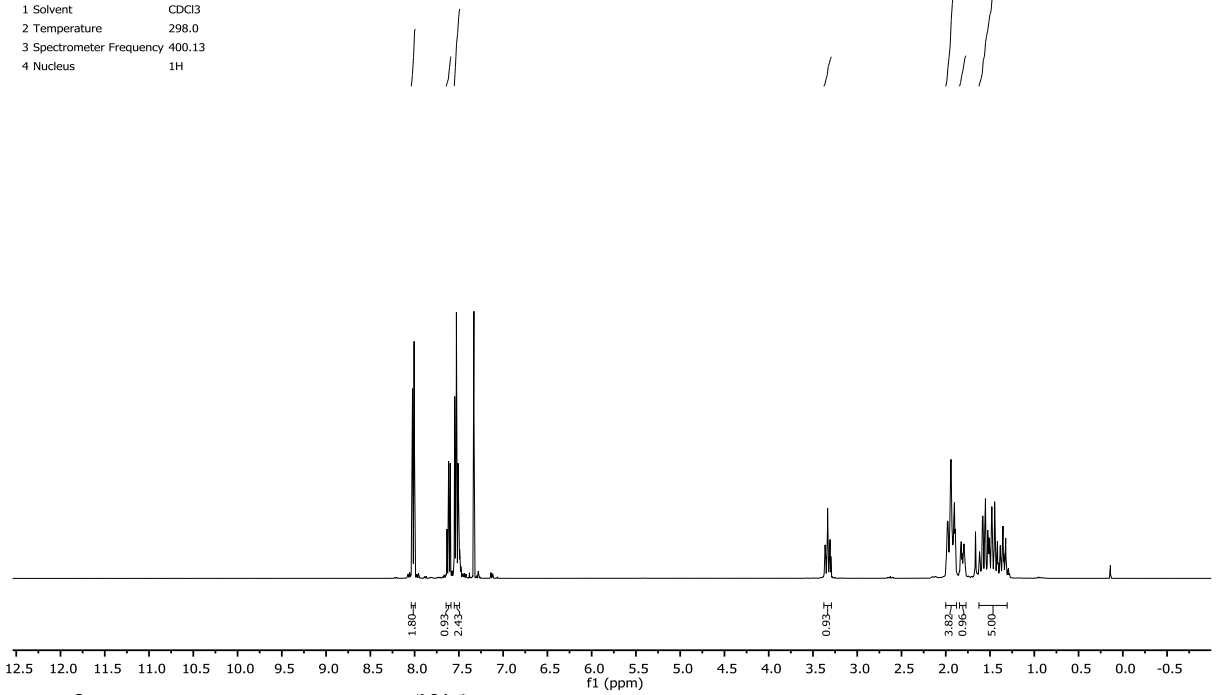


2-Iodoctane^{3,4} CAS: 557-36-8

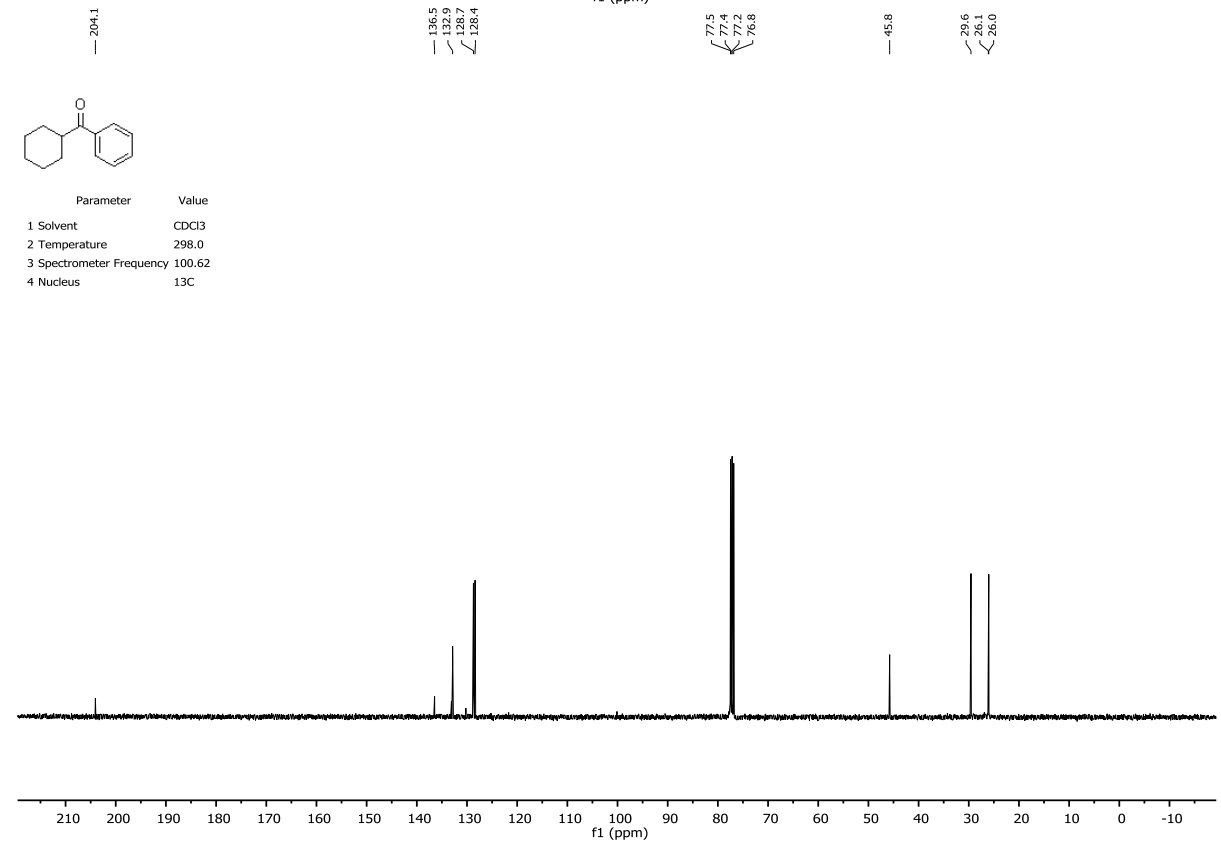
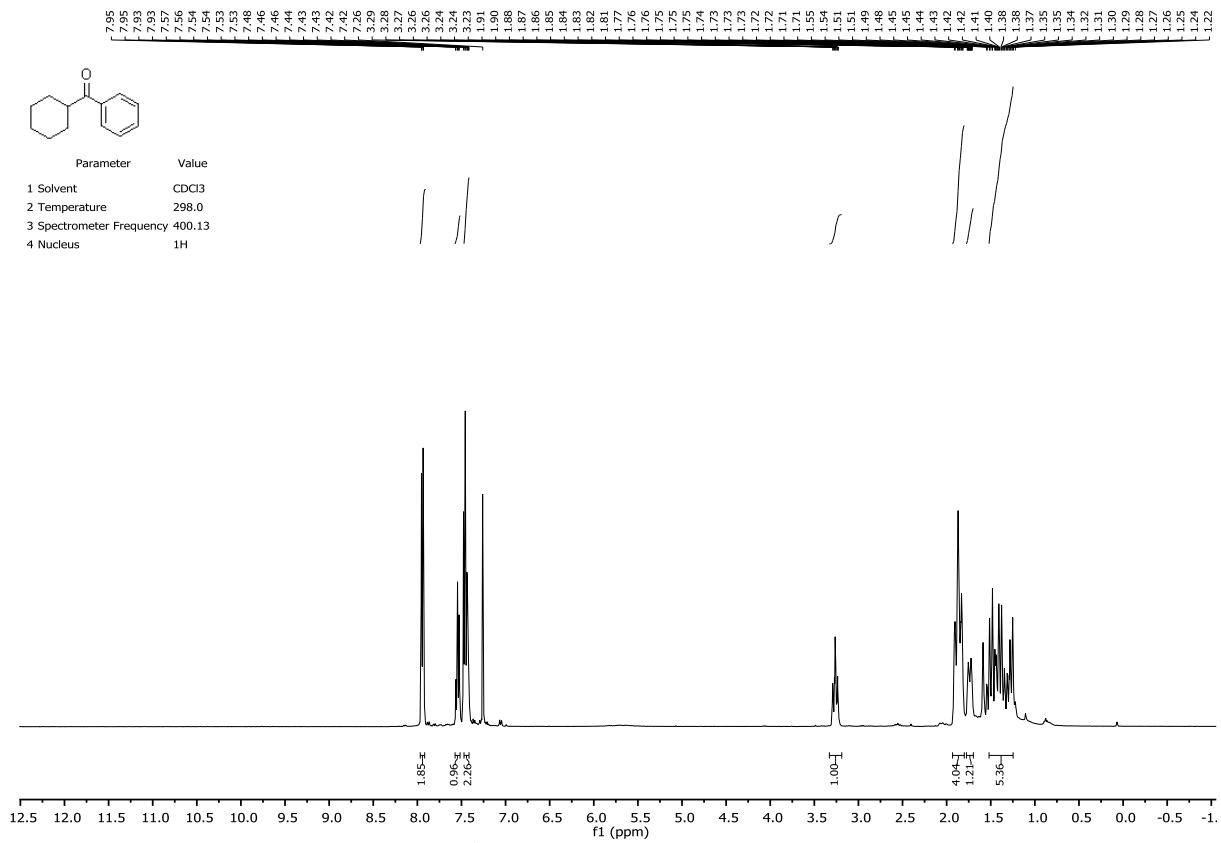
7.06
7.05
7.04
7.03
7.02
7.01
7.00
6.99
6.98
6.97
6.96
6.95
6.94
6.93
6.92
6.91
6.90
6.89
6.88
6.87
6.86
6.85
6.84
6.83
6.82
6.81
6.80
6.79
6.78
6.77
6.76
6.75
6.74
6.73
6.72
6.71
6.70
6.69
6.68
6.67
6.66
6.65
6.64
6.63
6.62
6.61
6.60
6.59
6.58
6.57
6.56
6.55
6.54
6.53
6.52
6.51
6.50
6.49
6.48
6.47
6.46
6.45
6.44
6.43
6.42
6.41
6.40
6.39
6.38
6.37
6.36
6.35
6.34
6.33
6.32
6.31
6.30
6.29
6.28
6.27
6.26
6.25
6.24
6.23
6.22
6.21
6.20
6.19
6.18
6.17
6.16
6.15
6.14
6.13
6.12
6.11
6.10
6.09
6.08
6.07
6.06
6.05
6.04
6.03
6.02
6.01
6.00
5.99
5.98
5.97
5.96
5.95
5.94
5.93
5.92
5.91
5.90
5.89
5.88
5.87
5.86
5.85
5.84
5.83
5.82
5.81
5.80
5.79
5.78
5.77
5.76
5.75
5.74
5.73
5.72
5.71
5.70
5.69
5.68
5.67
5.66
5.65
5.64
5.63
5.62
5.61
5.60
5.59
5.58
5.57
5.56
5.55
5.54
5.53
5.52
5.51
5.50
5.49
5.48
5.47
5.46
5.45
5.44
5.43
5.42
5.41
5.40
5.39
5.38
5.37
5.36
5.35
5.34
5.33
5.32
5.31
5.30
5.29
5.28
5.27
5.26
5.25
5.24
5.23
5.22
5.21
5.20
5.19
5.18
5.17
5.16
5.15
5.14
5.13
5.12
5.11
5.10
5.09
5.08
5.07
5.06
5.05
5.04
5.03
5.02
5.01
5.00
4.99
4.98
4.97
4.96
4.95
4.94
4.93
4.92
4.91
4.90
4.89
4.88
4.87
4.86
4.85
4.84
4.83
4.82
4.81
4.80
4.79
4.78
4.77
4.76
4.75
4.74
4.73
4.72
4.71
4.70
4.69
4.68
4.67
4.66
4.65
4.64
4.63
4.62
4.61
4.60
4.59
4.58
4.57
4.56
4.55
4.54
4.53
4.52
4.51
4.50
4.49
4.48
4.47
4.46
4.45
4.44
4.43
4.42
4.41
4.40
4.39
4.38
4.37
4.36
4.35
4.34
4.33
4.32
4.31
4.30
4.29
4.28
4.27
4.26
4.25
4.24
4.23
4.22
4.21
4.20
4.19
4.18
4.17
4.16
4.15
4.14
4.13
4.12
4.11
4.10
4.09
4.08
4.07
4.06
4.05
4.04
4.03
4.02
4.01
4.00
3.99
3.98
3.97
3.96
3.95
3.94
3.93
3.92
3.91
3.90
3.89
3.88
3.87
3.86
3.85
3.84
3.83
3.82
3.81
3.80
3.79
3.78
3.77
3.76
3.75
3.74
3.73
3.72
3.71
3.70
3.69
3.68
3.67
3.66
3.65
3.64
3.63
3.62
3.61
3.60
3.59
3.58
3.57
3.56
3.55
3.54
3.53
3.52
3.51
3.50
3.49
3.48
3.47
3.46
3.45
3.44
3.43
3.42
3.41
3.40
3.39
3.38
3.37
3.36
3.35
3.34
3.33
3.32
3.31
3.30
3.29
3.28
3.27
3.26
3.25
3.24
3.23
3.22
3.21
3.20
3.19
3.18
3.17
3.16
3.15
3.14
3.13
3.12
3.11
3.10
3.09
3.08
3.07
3.06
3.05
3.04
3.03
3.02
3.01
3.00
2.99
2.98
2.97
2.96
2.95
2.94
2.93
2.92
2.91
2.90
2.89
2.88
2.87
2.86
2.85
2.84
2.83
2.82
2.81
2.80
2.79
2.78
2.77
2.76
2.75
2.74
2.73
2.72
2.71
2.70
2.69
2.68
2.67
2.66
2.65
2.64
2.63
2.62
2.61
2.60
2.59
2.58
2.57
2.56
2.55
2.54
2.53
2.52
2.51
2.50
2.49
2.48
2.47
2.46
2.45
2.44
2.43
2.42
2.41
2.40
2.39
2.38
2.37
2.36
2.35
2.34
2.33
2.32
2.31
2.30
2.29
2.28
2.27
2.26
2.25
2.24
2.23
2.22
2.21
2.20
2.19
2.18
2.17
2.16
2.15
2.14
2.13
2.12
2.11
2.10
2.09
2.08
2.07
2.06
2.05
2.04
2.03
2.02
2.01
2.00
1.99
1.98
1.97
1.96
1.95
1.94
1.93
1.92
1.91
1.90
1.89
1.88
1.87
1.86
1.85
1.84
1.83
1.82
1.81
1.80
1.79
1.78
1.77
1.76
1.75
1.74
1.73
1.72
1.71
1.70
1.69
1.68
1.67
1.66
1.65
1.64
1.63
1.62
1.61
1.60
1.59
1.58
1.57
1.56
1.55
1.54
1.53
1.52
1.51
1.50
1.49
1.48
1.47
1.46
1.45
1.44
1.43
1.42
1.41
1.40
1.39
1.38
1.37
1.36
1.35
1.34
1.33
1.32
1.31
1.30
1.29
1.28



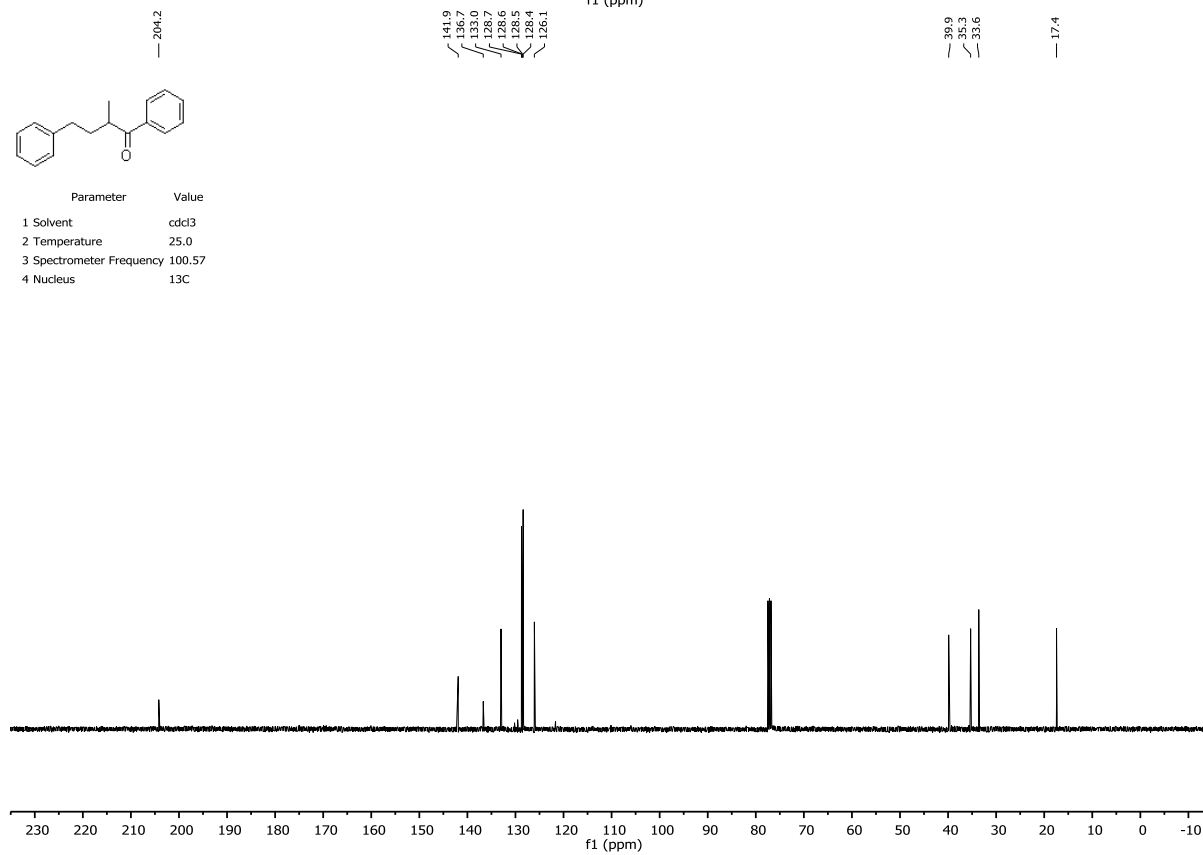
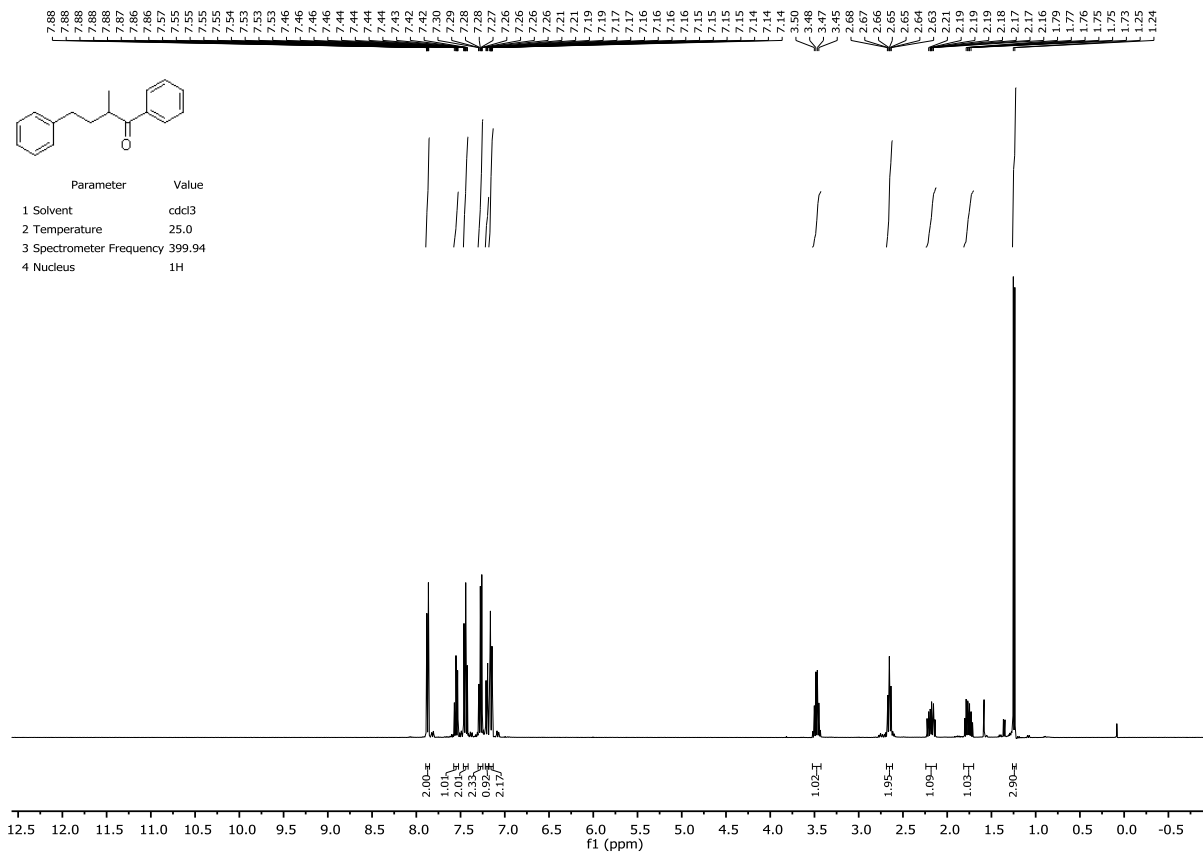
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



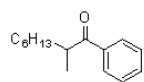
Cyclohexylphenylmethanone⁵ (4) CAS: 712-50-5 (from cyclohexyl iodide)



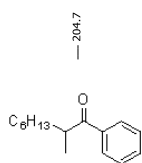
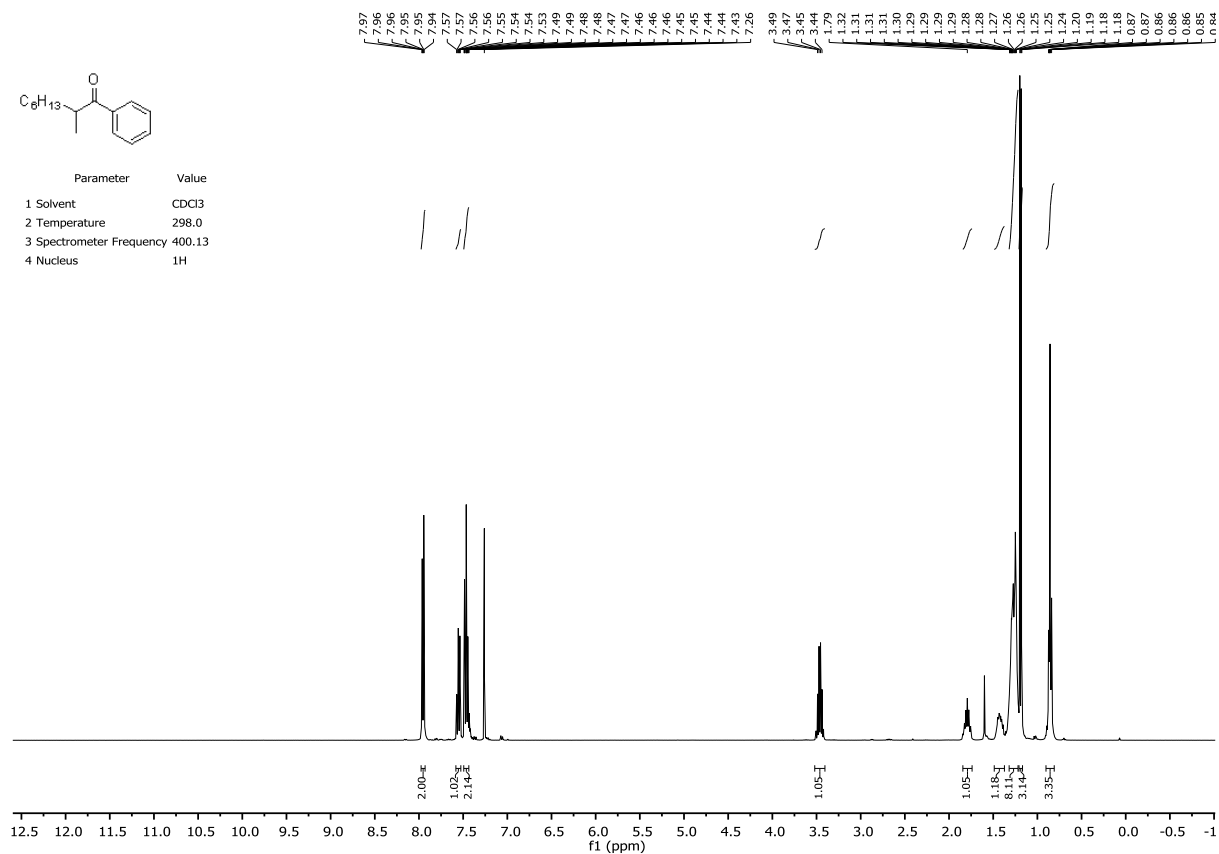
Cyclohexylphenylmethanone⁵ (4) CAS: 712-50-5 (from cyclohexyl bromide)



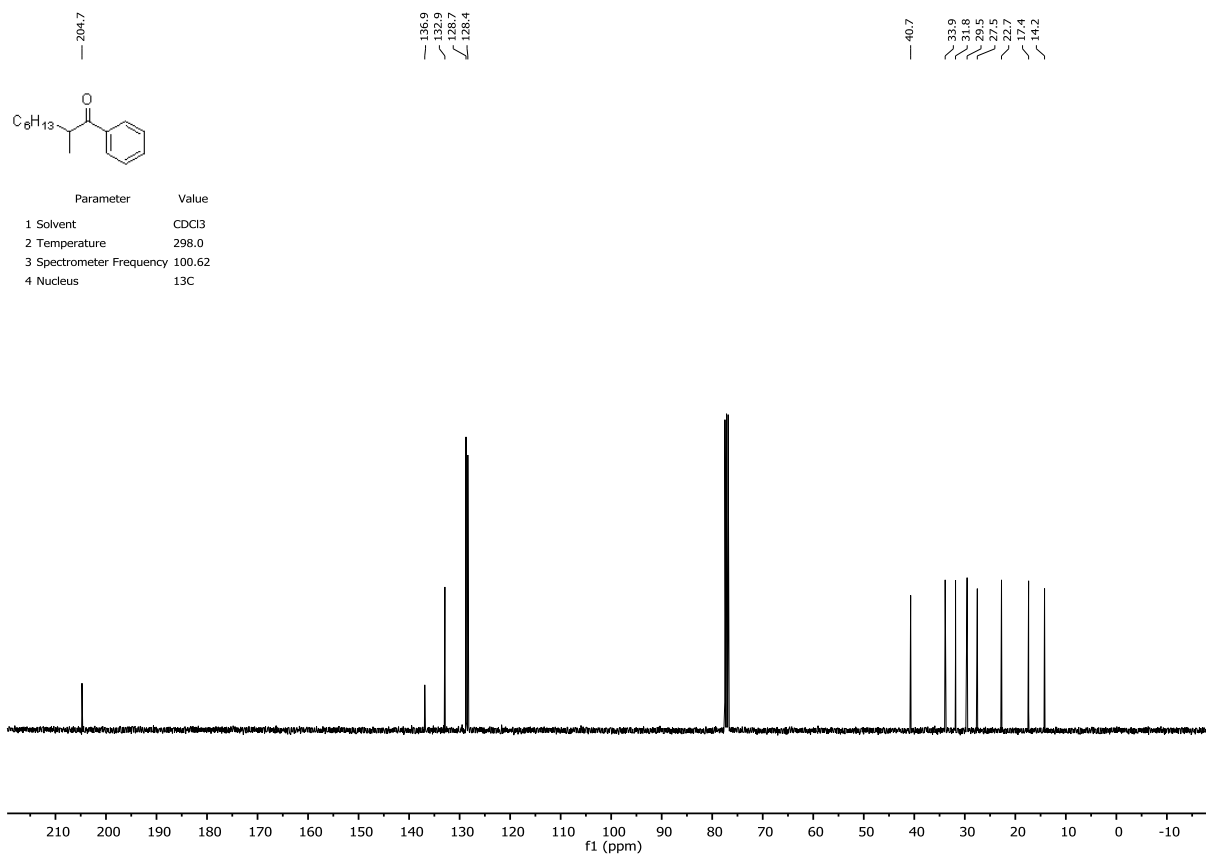
2-Methyl-1,4-diphenylbutan-1-one (5) CAS: 108974-08-9



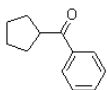
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	1H



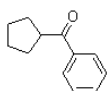
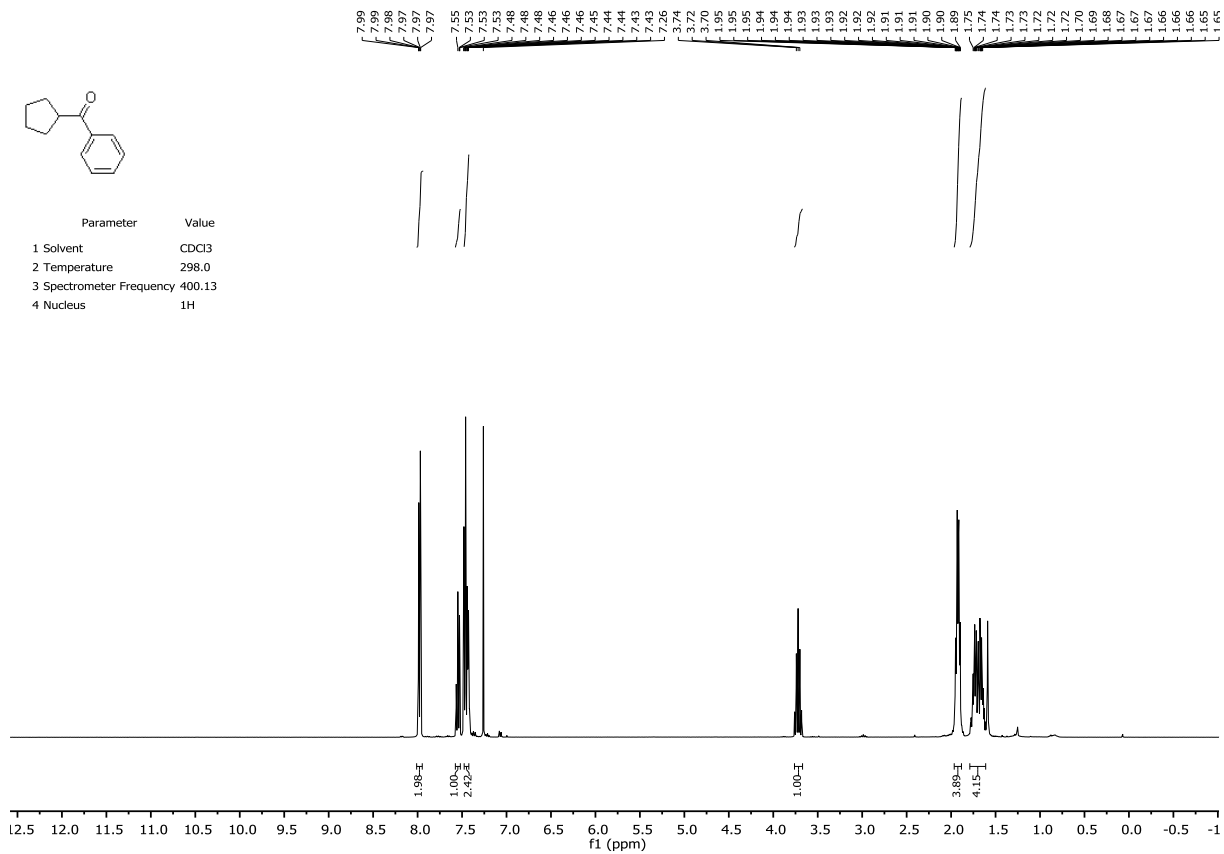
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	13C



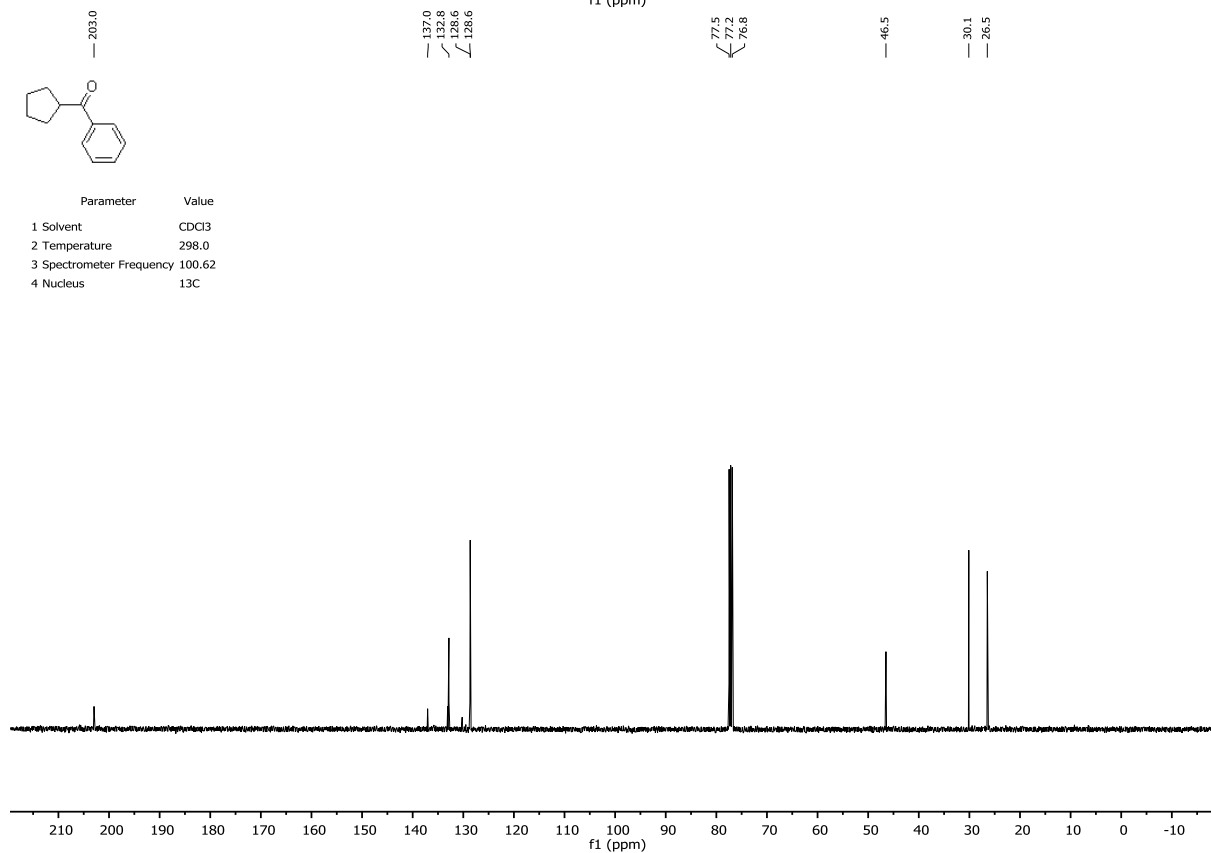
2-Methyl-1-phenyloctan-1-one⁶ (6) CAS: 132719-12-1



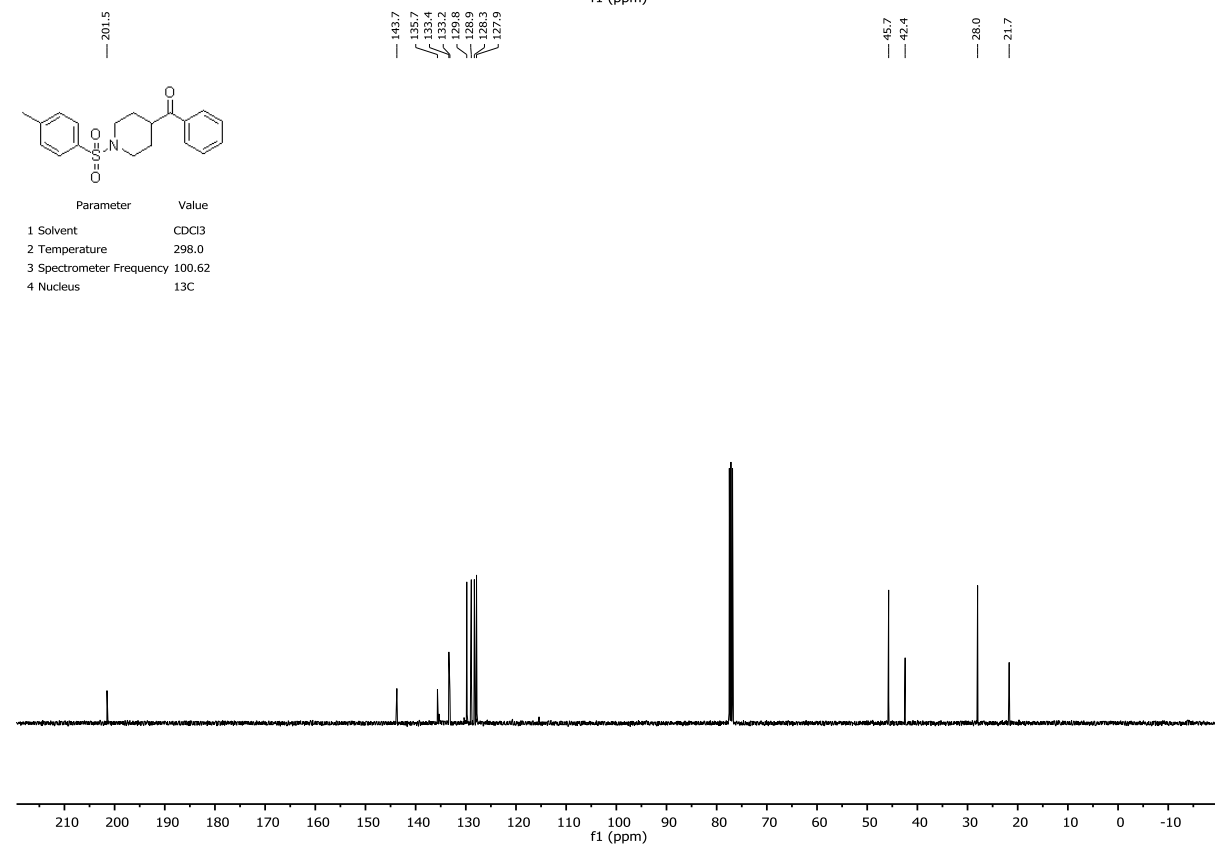
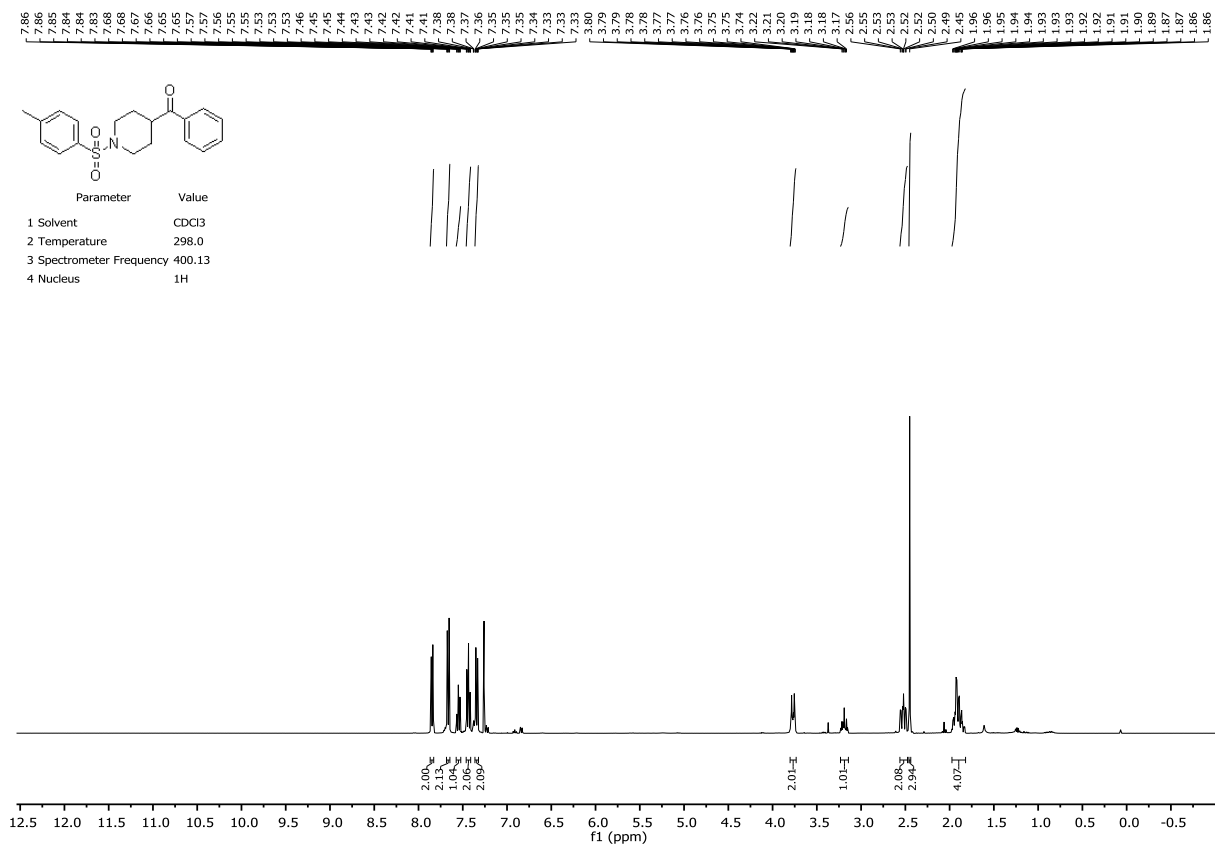
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



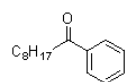
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	¹³ C



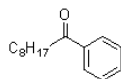
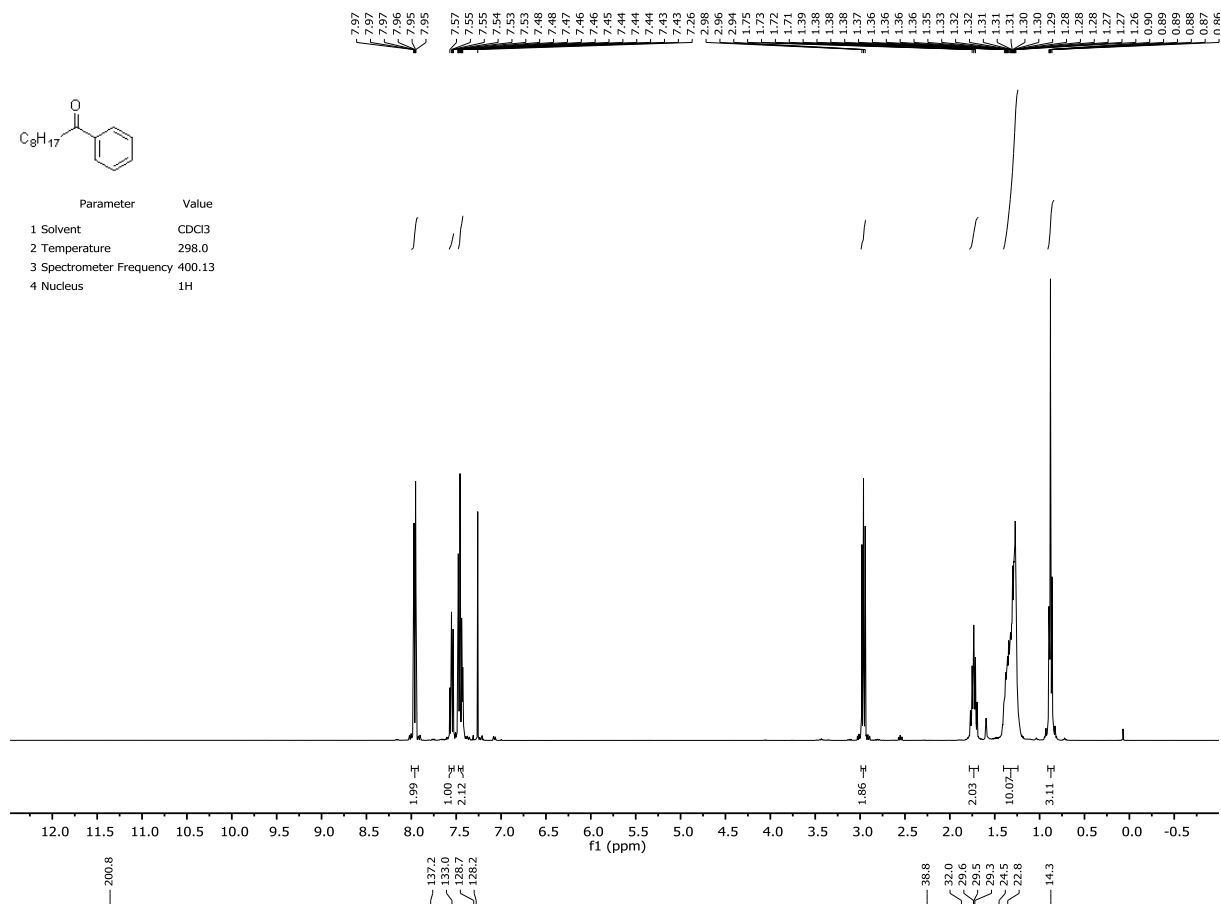
Cyclopentylphenylmethanone⁷ (7) CAS: 5422-88-8



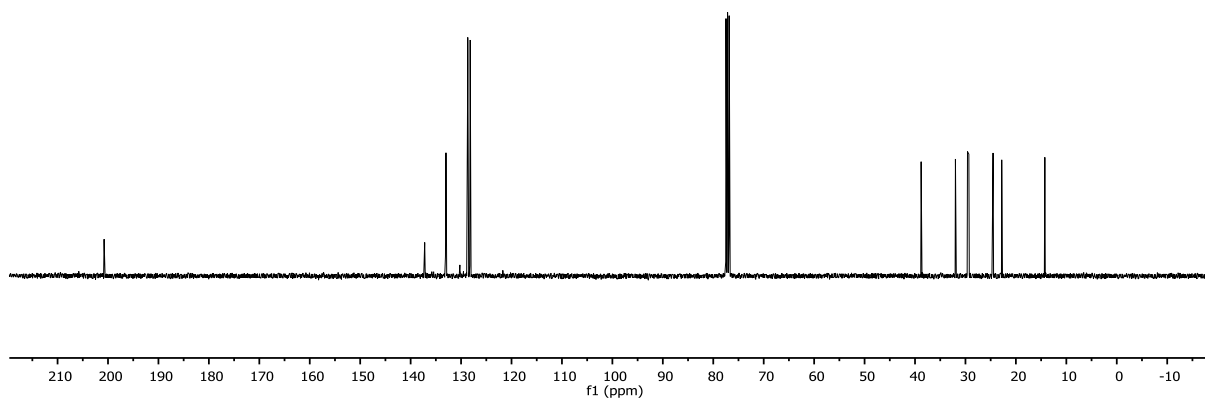
Phenyl(1-tosylpiperidin-4-yl)methanone (8) CAS: 922504-26-5



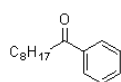
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



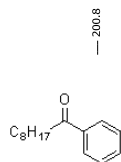
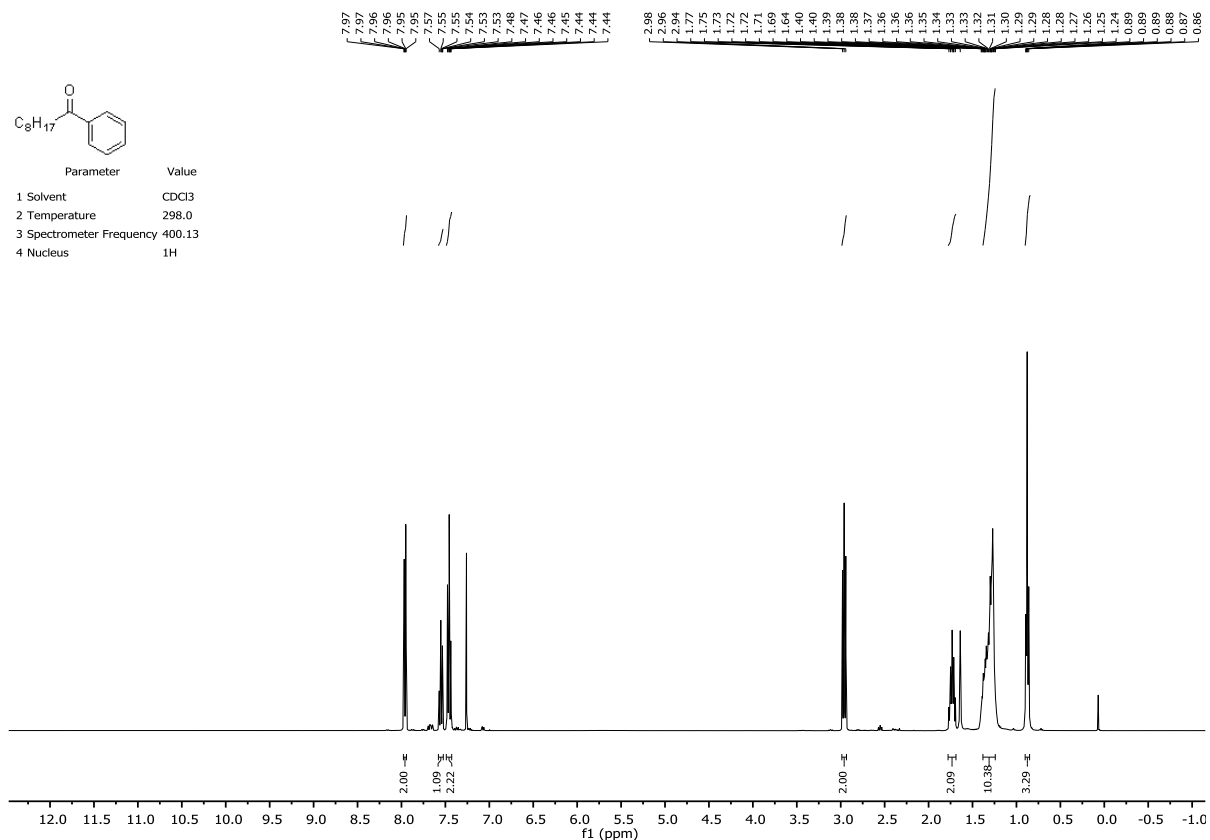
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	¹³ C



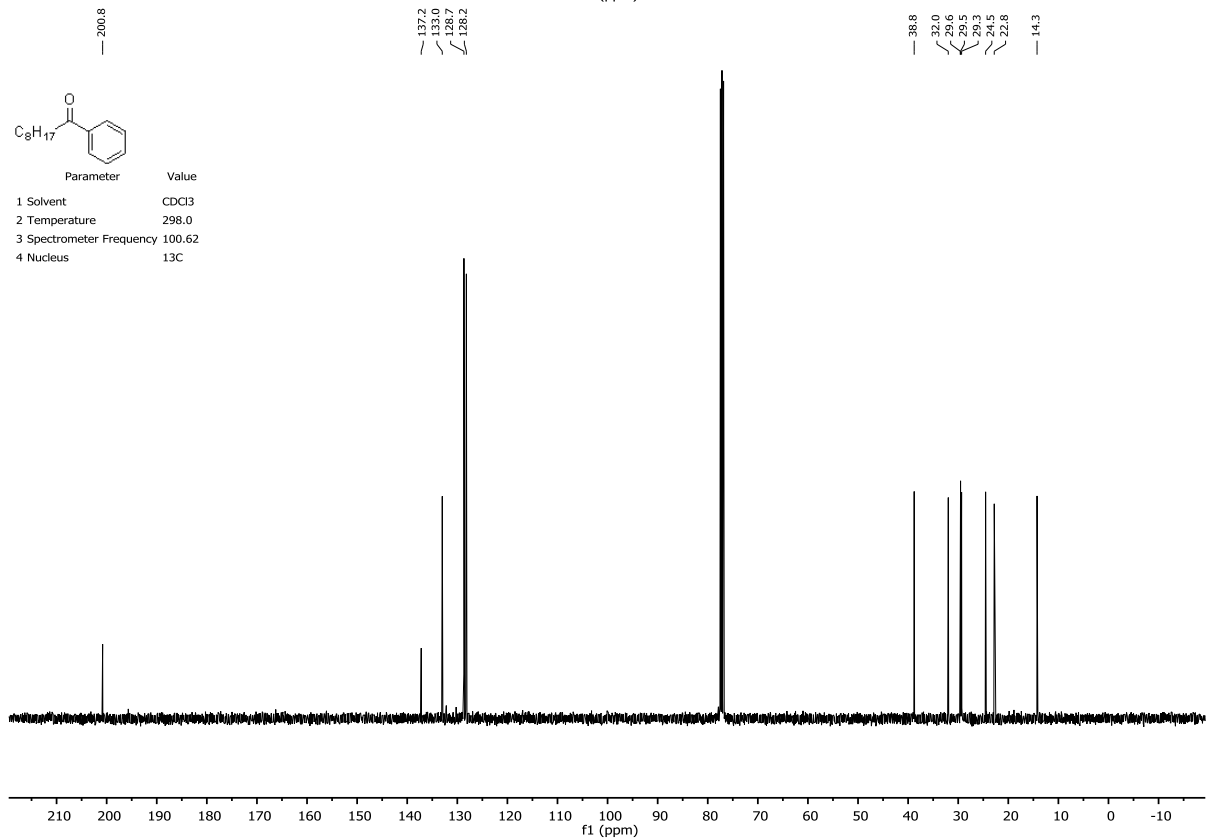
1-Phenylnonan-1-one⁸ (9) CAS: 6008-36-2 (from 1-iodooctane)



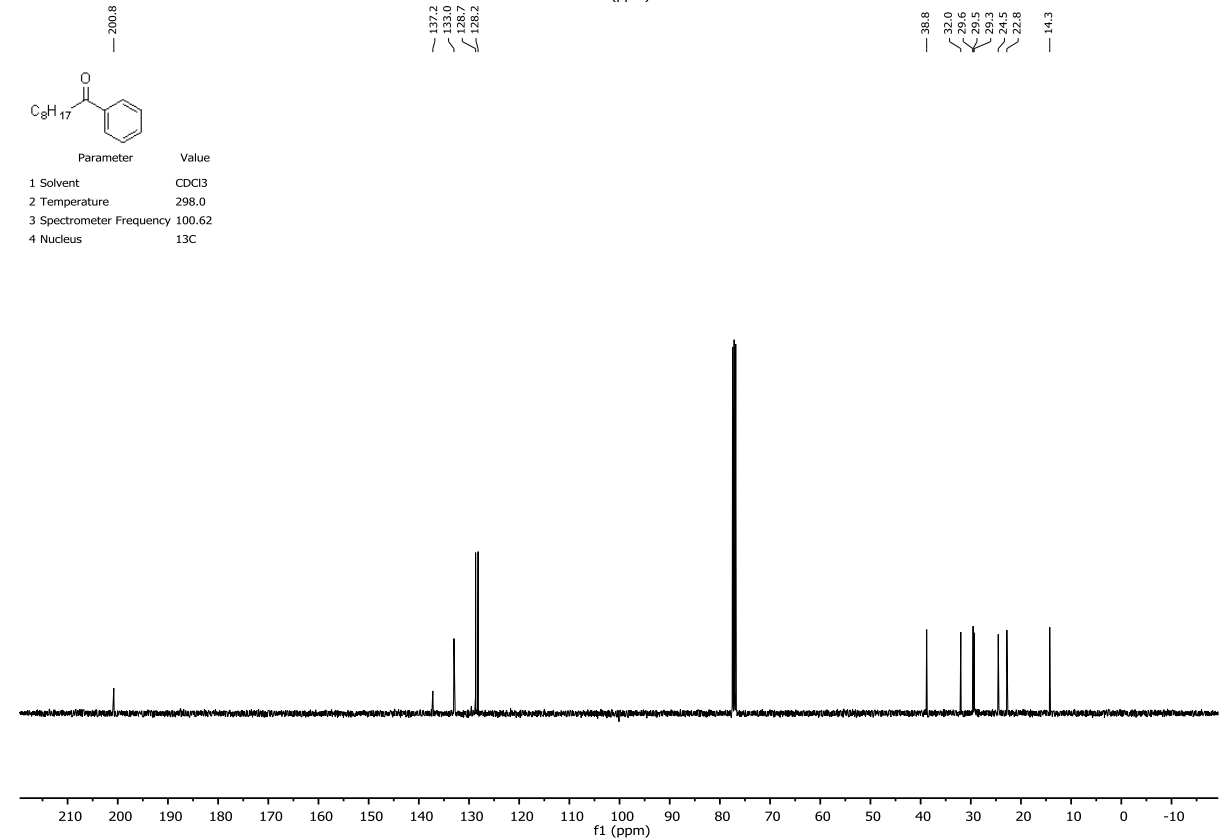
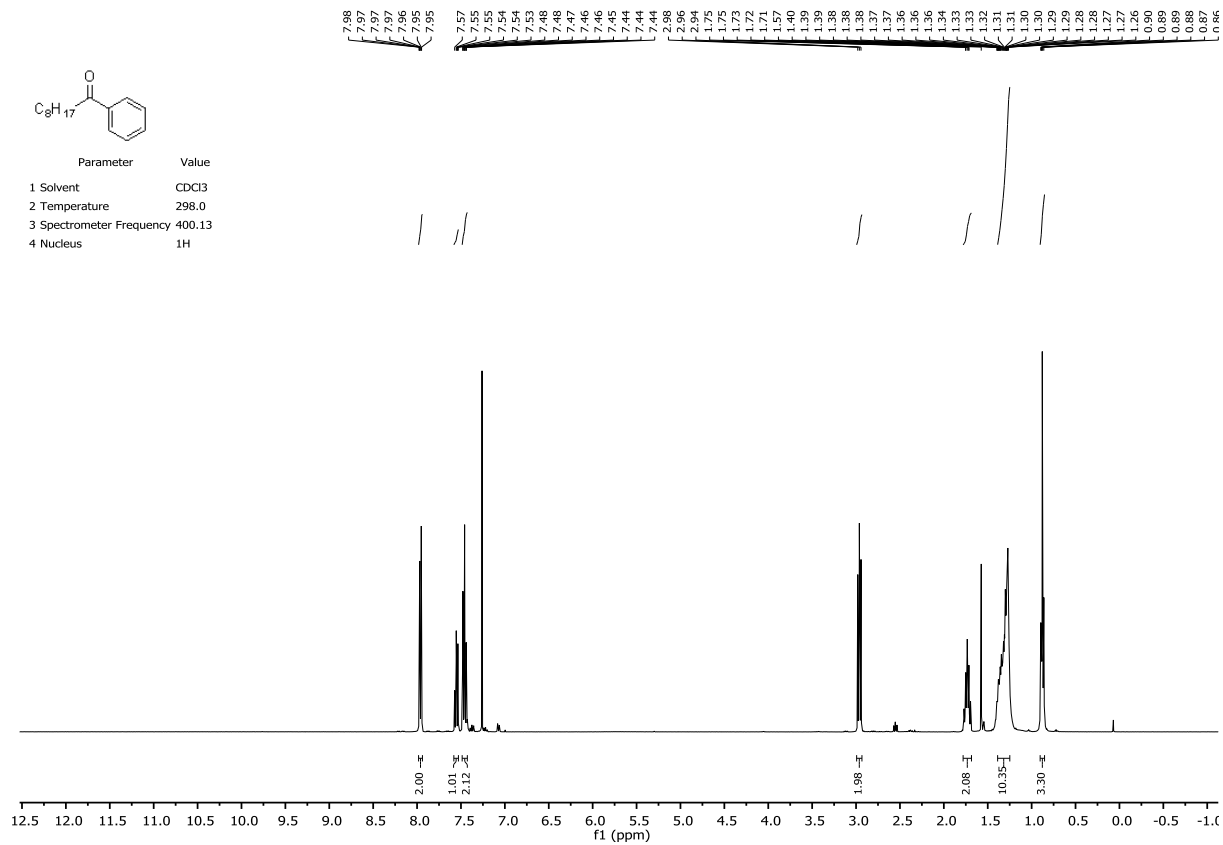
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



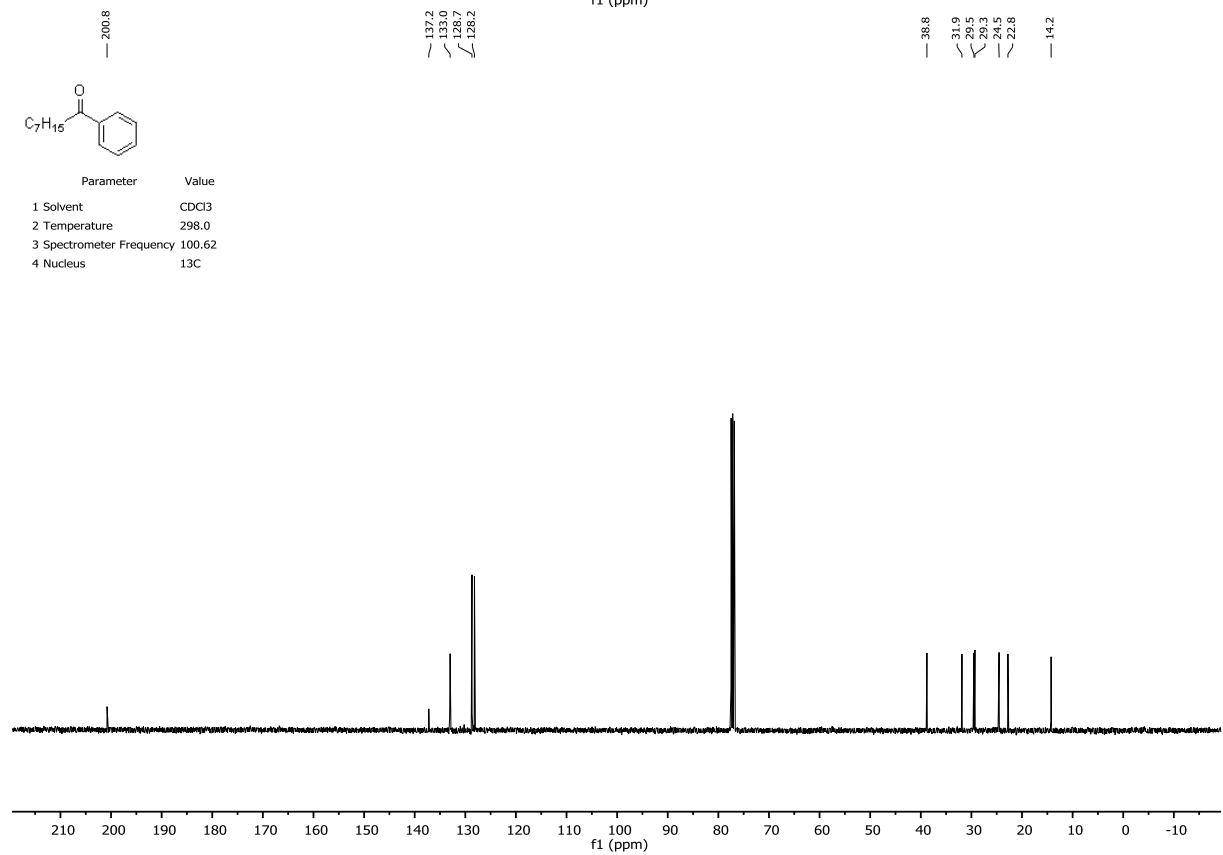
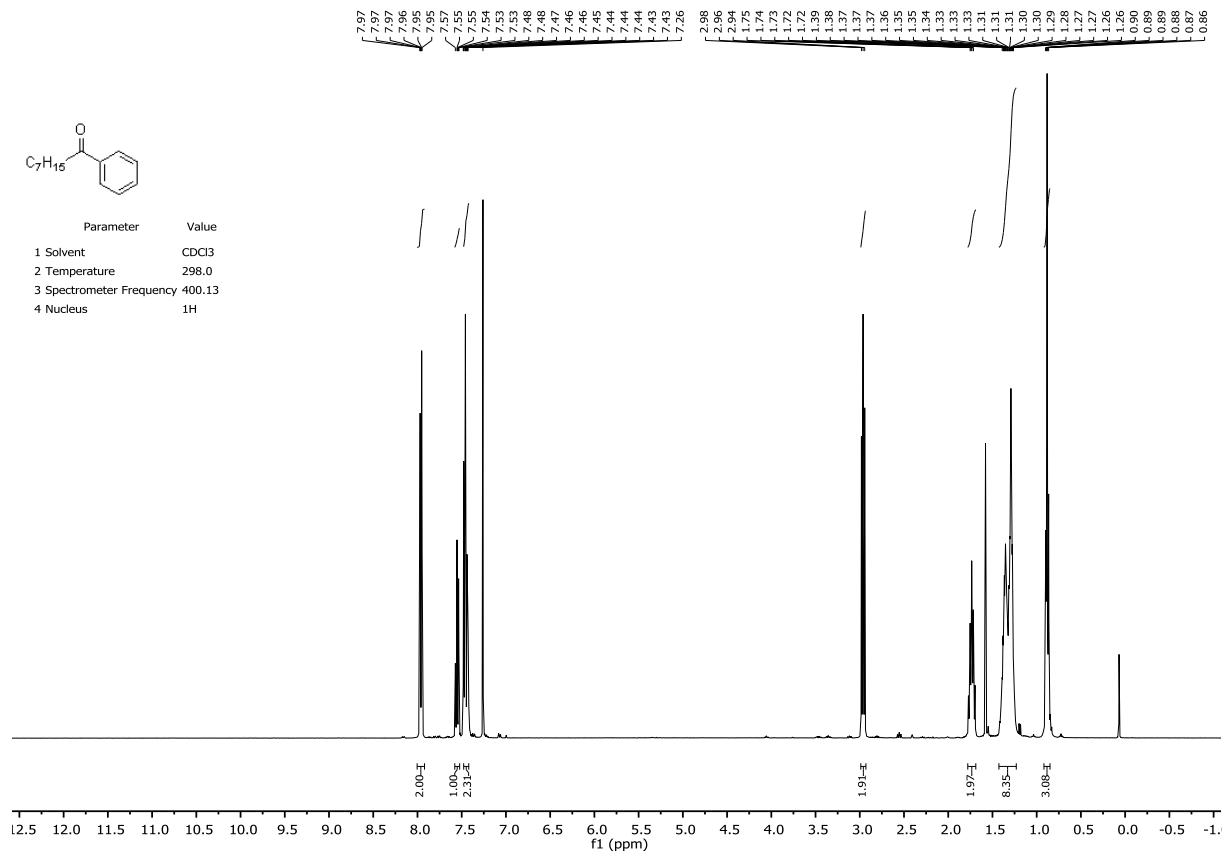
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	¹³ C



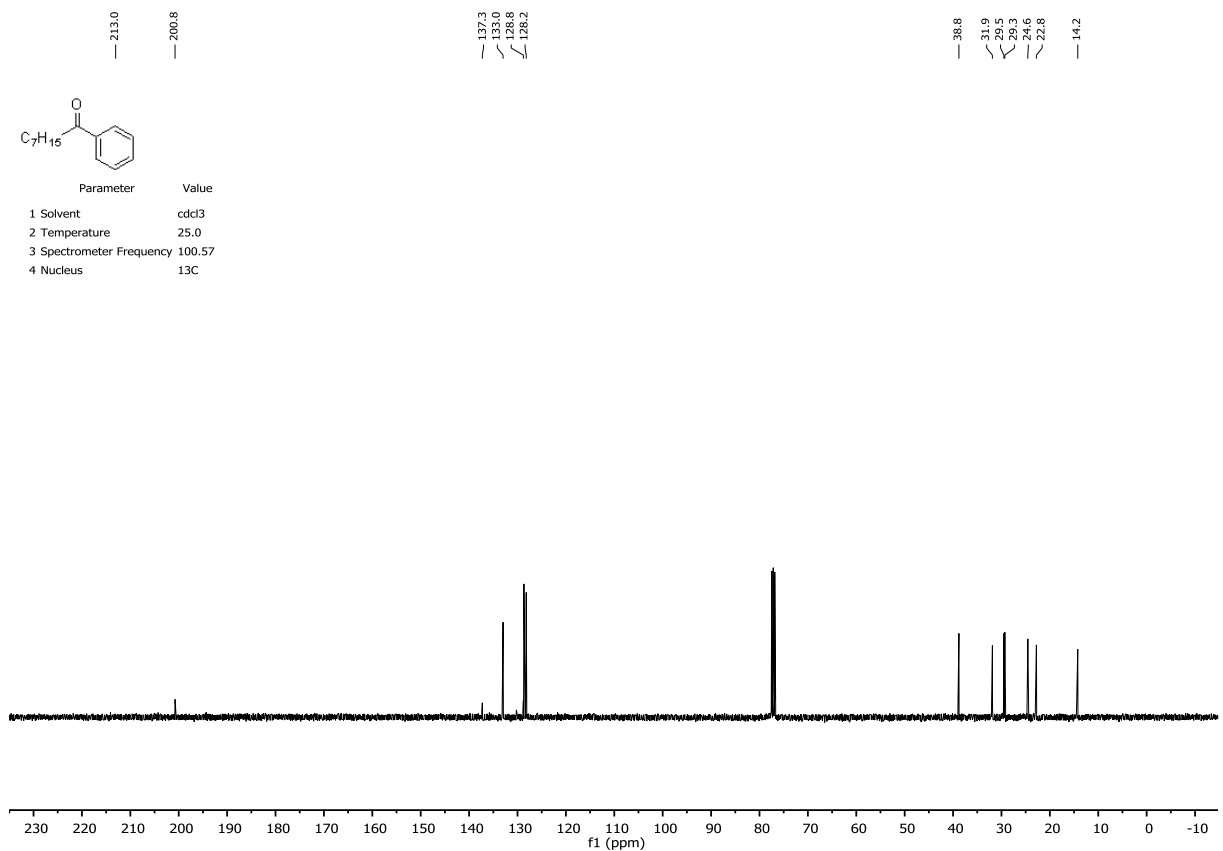
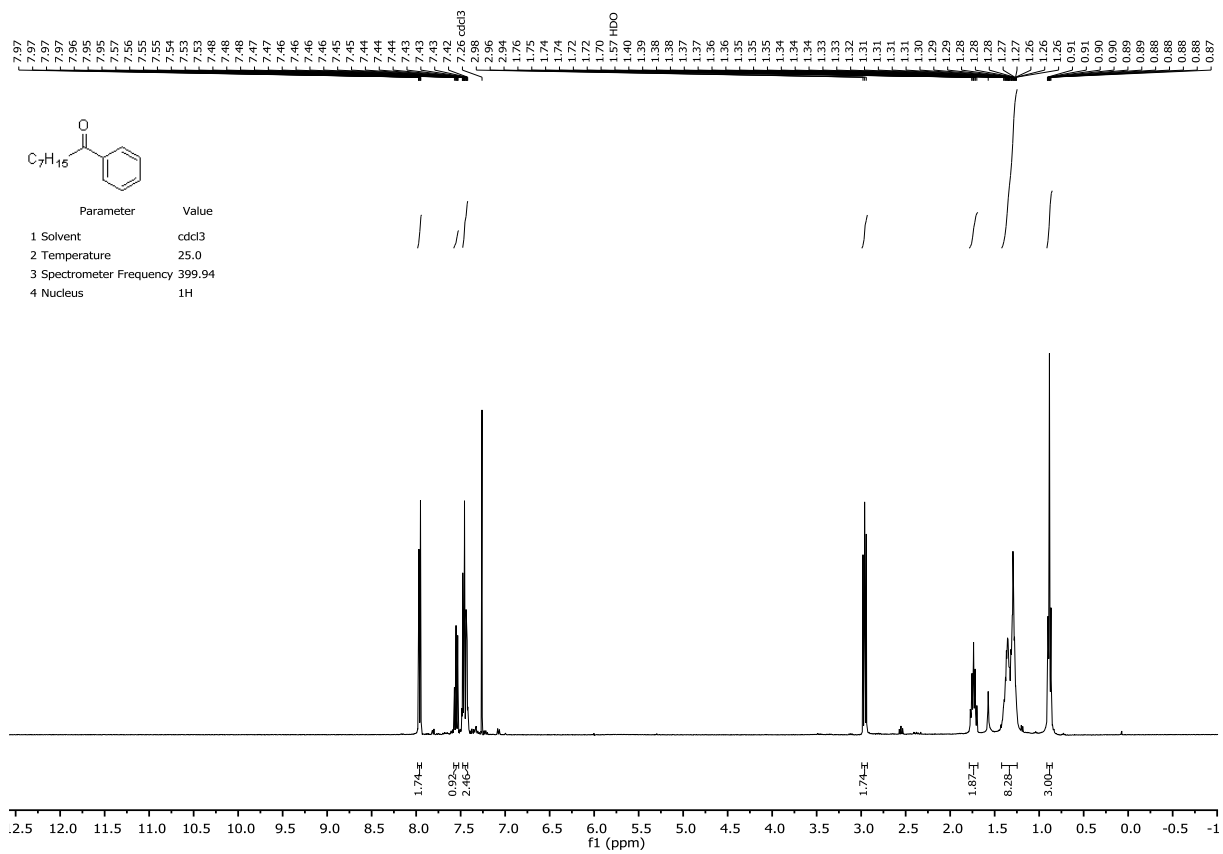
1-Phenylnonan-1-one⁸ (9) CAS: 6008-36-2 (from 1-bromooctane, 24 h irradiation)



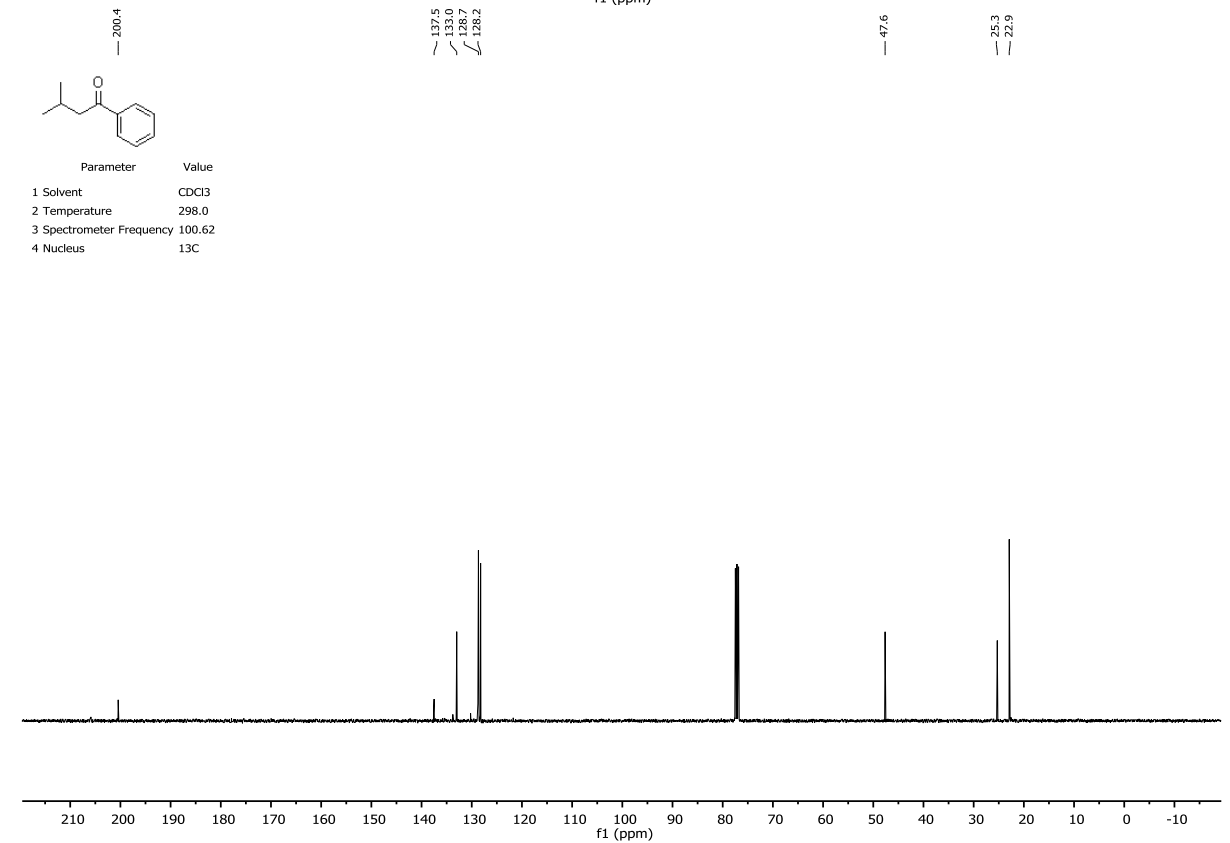
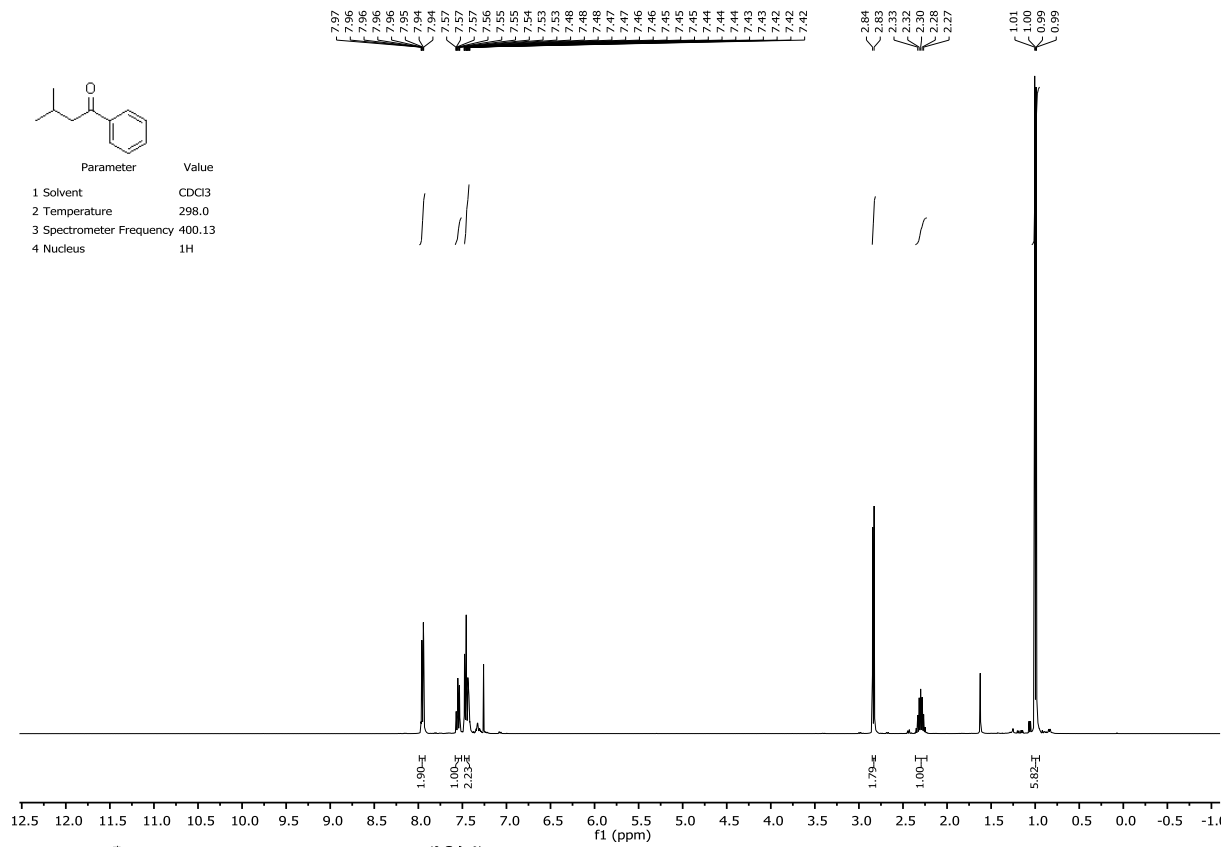
1-Phenylnonan-1-one⁸ (9) CAS: 6008-36-2 (from 1-bromooctane, 55 h irradiation)



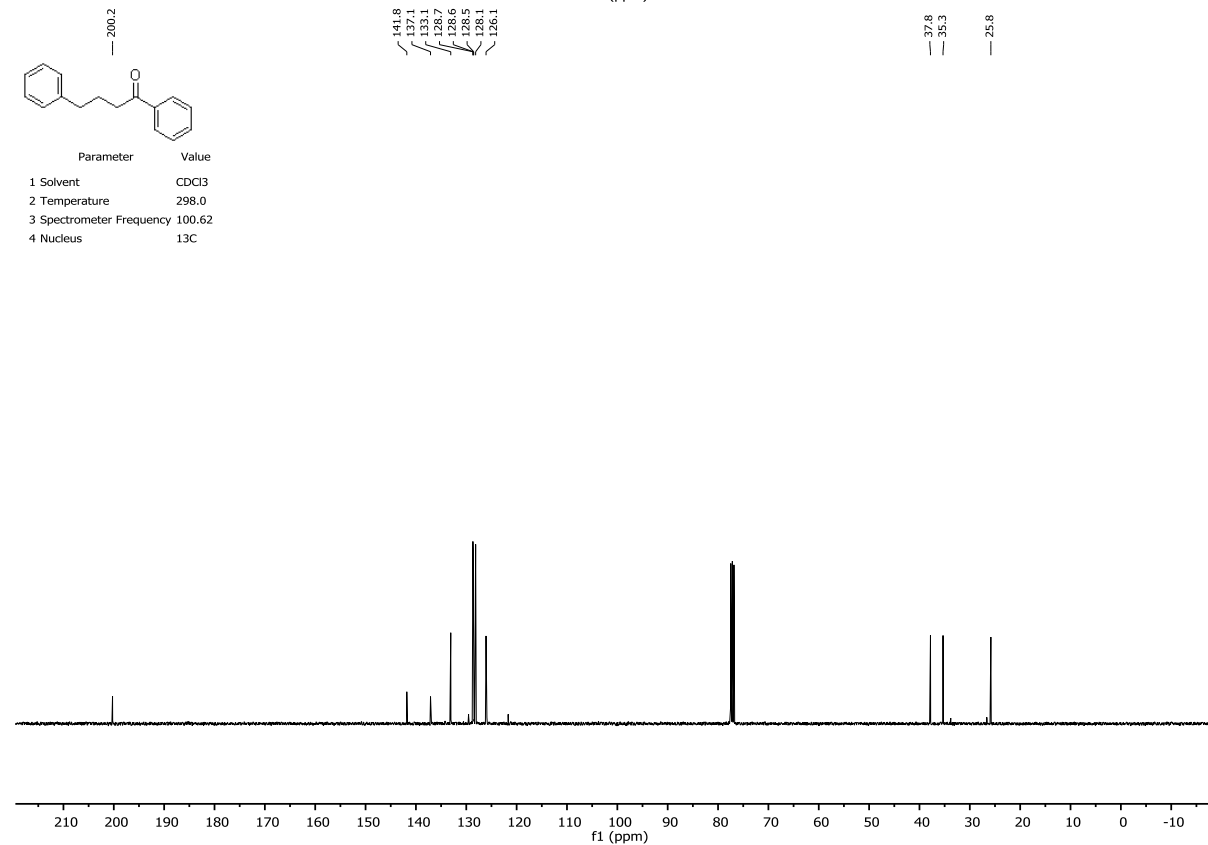
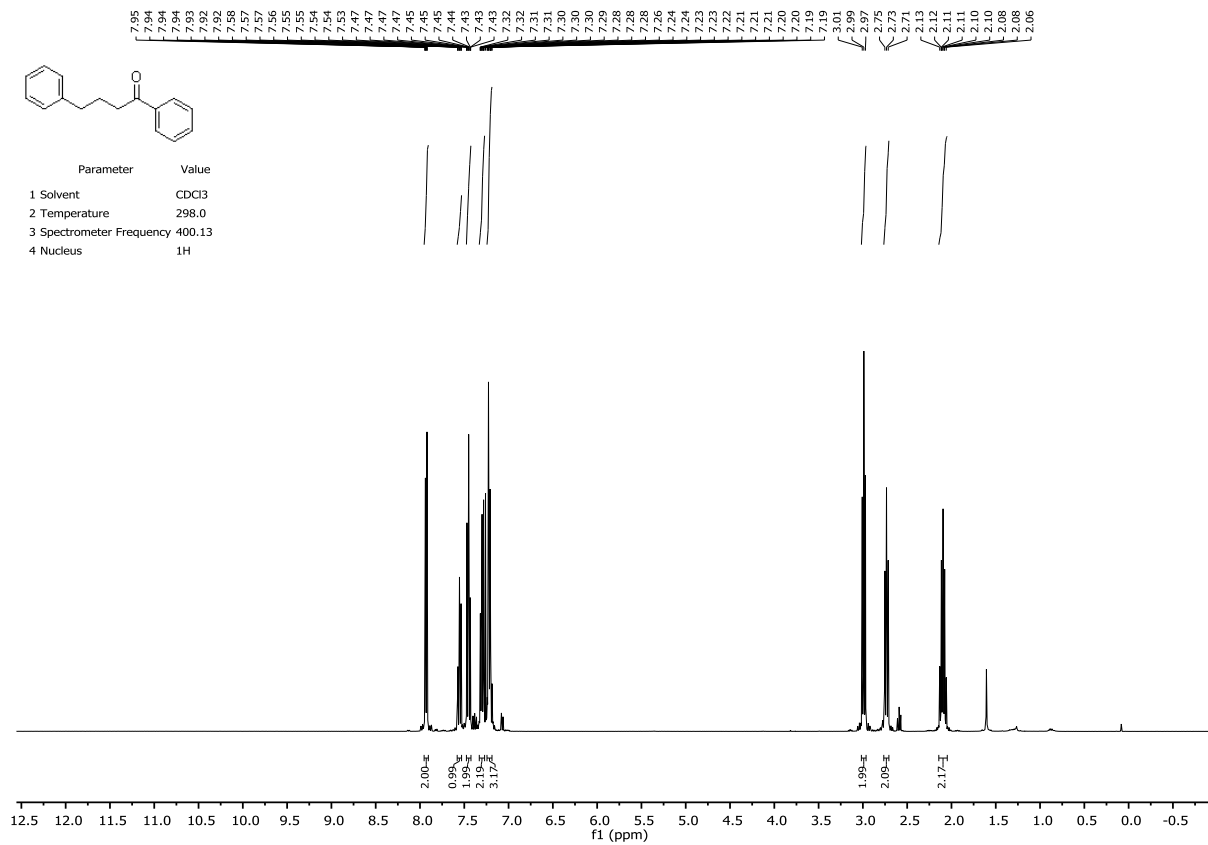
1-Phenyloctan-1-one⁹ (10) CAS: 1674-37-8 (from 1-iodoheptane)



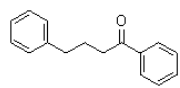
1-Phenyloctan-1-one⁹ (10) CAS: 1674-37-8 (from 1-bromoheptane)



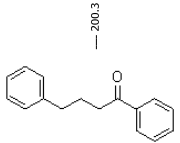
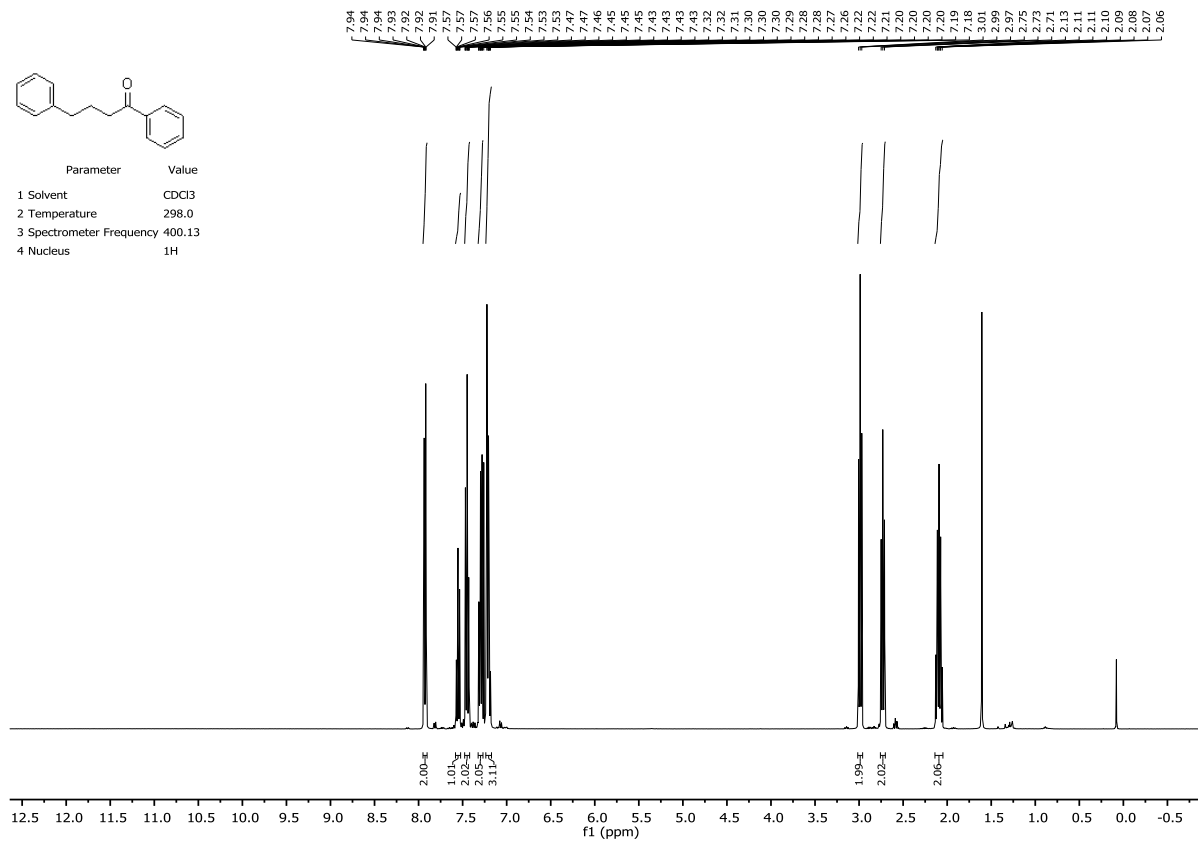
3-Methyl-1-phenylbutan-1-one¹⁰ (11) CAS: 582-62-7



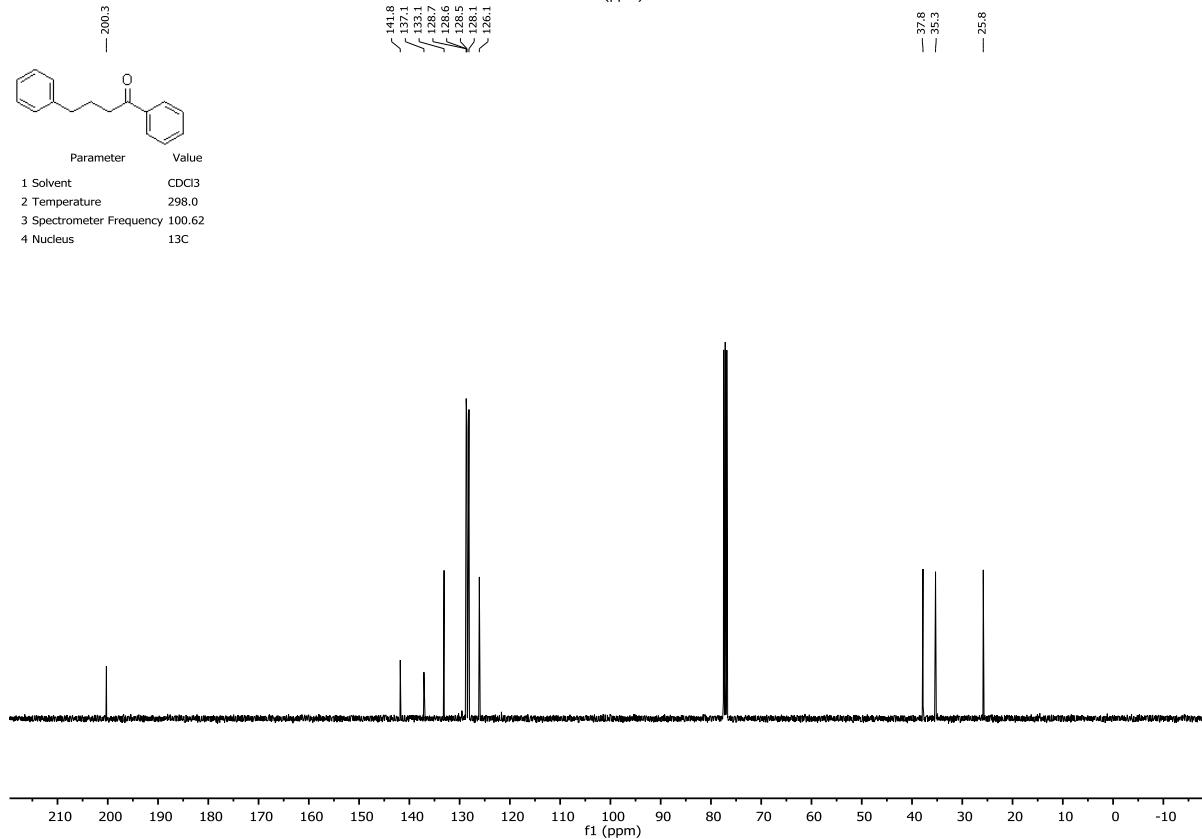
1,4-Diphenylbutan-1-one⁸ (12) CAS: 5407-91-0 (from 3-iodo-1-phenylpropane)



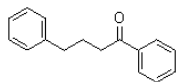
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	1H



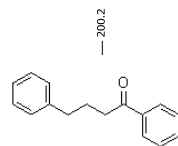
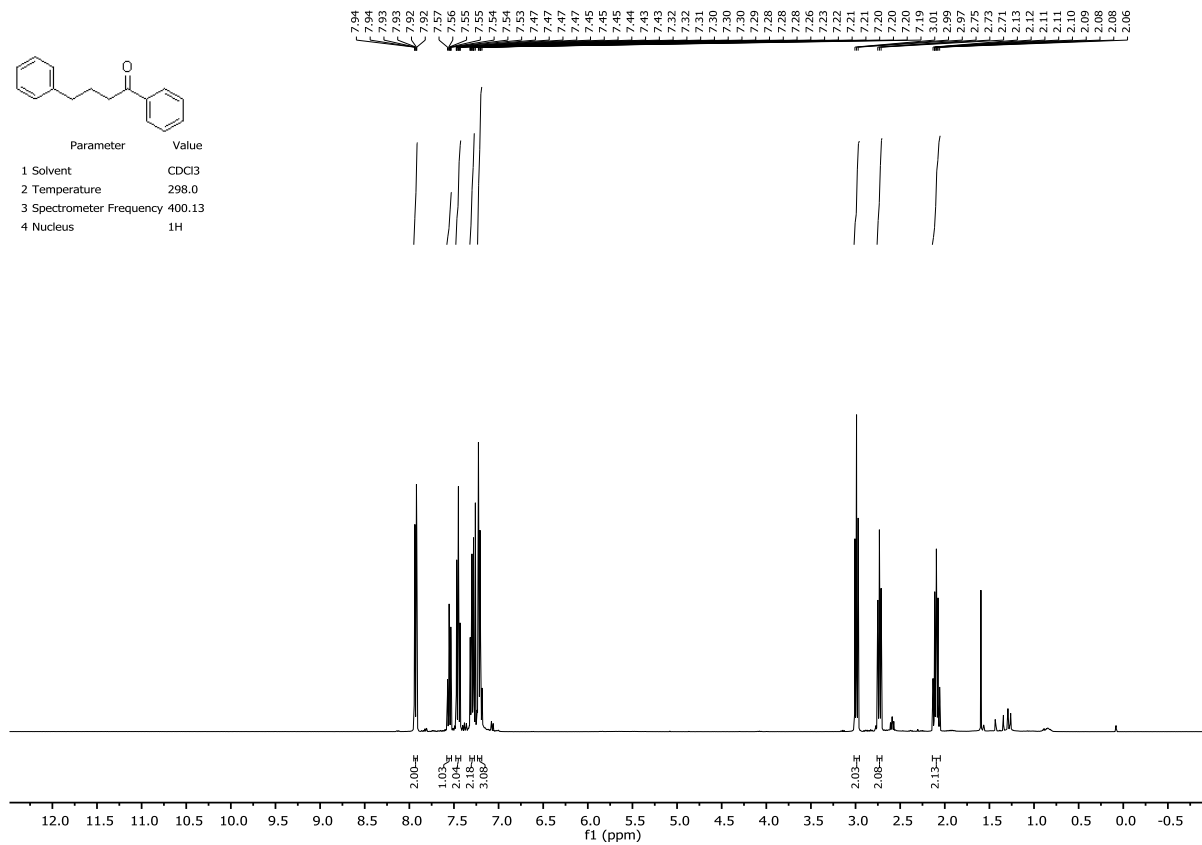
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	13C



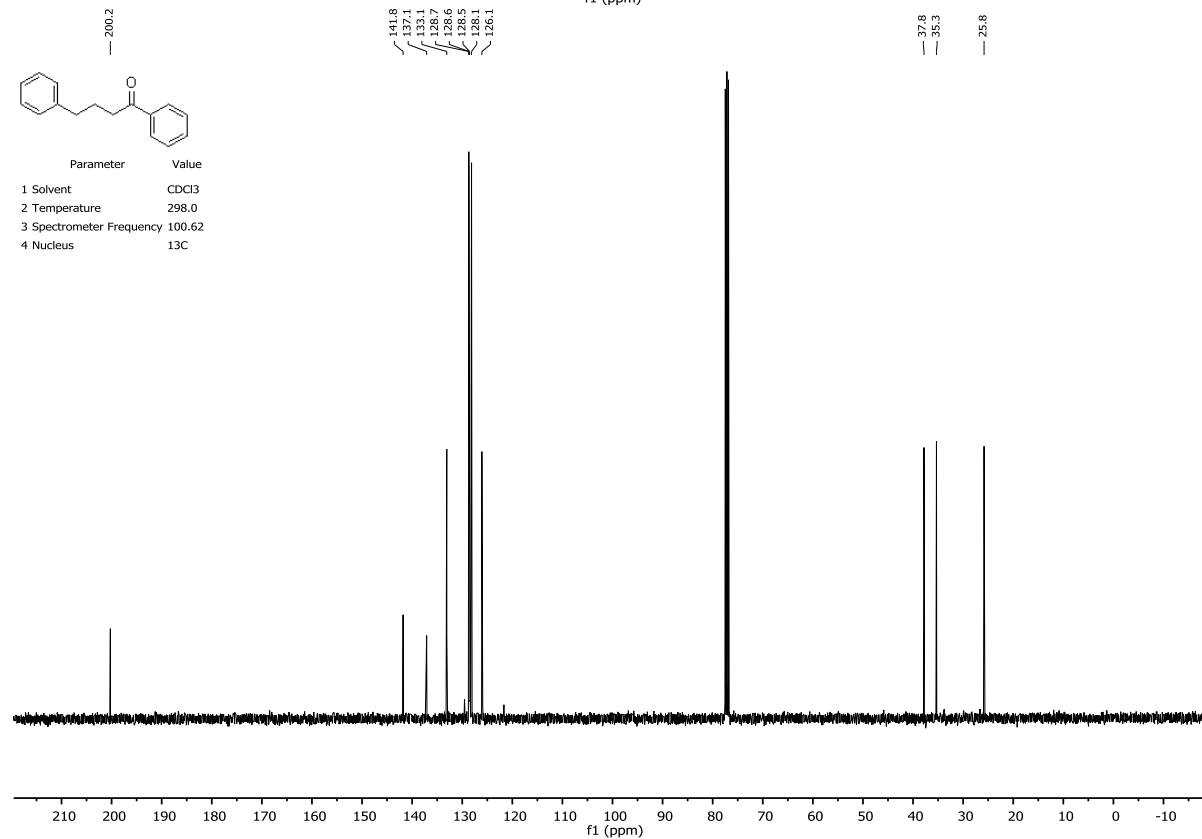
1,4-Diphenylbutan-1-one⁸ (12) CAS: 5407-91-0 (from 3-bromo-1-phenylpropane, 24 h irradiation)



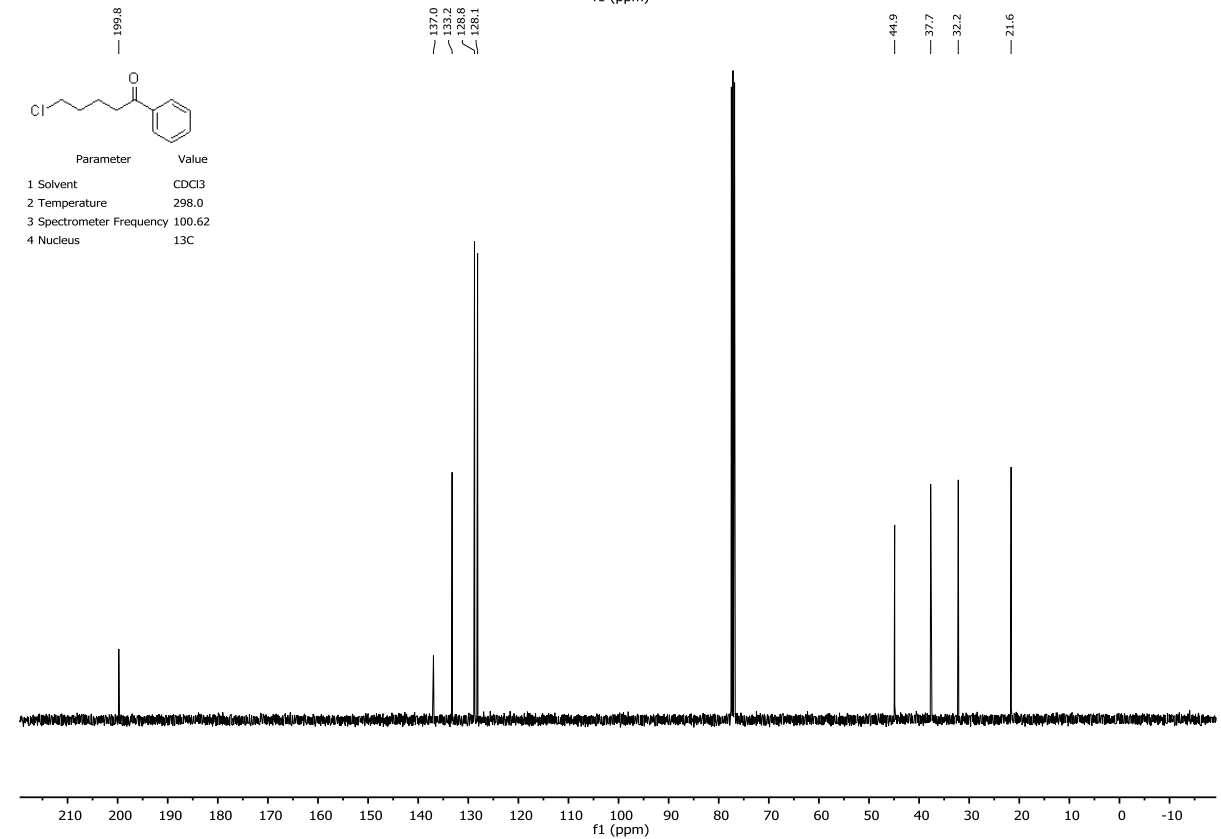
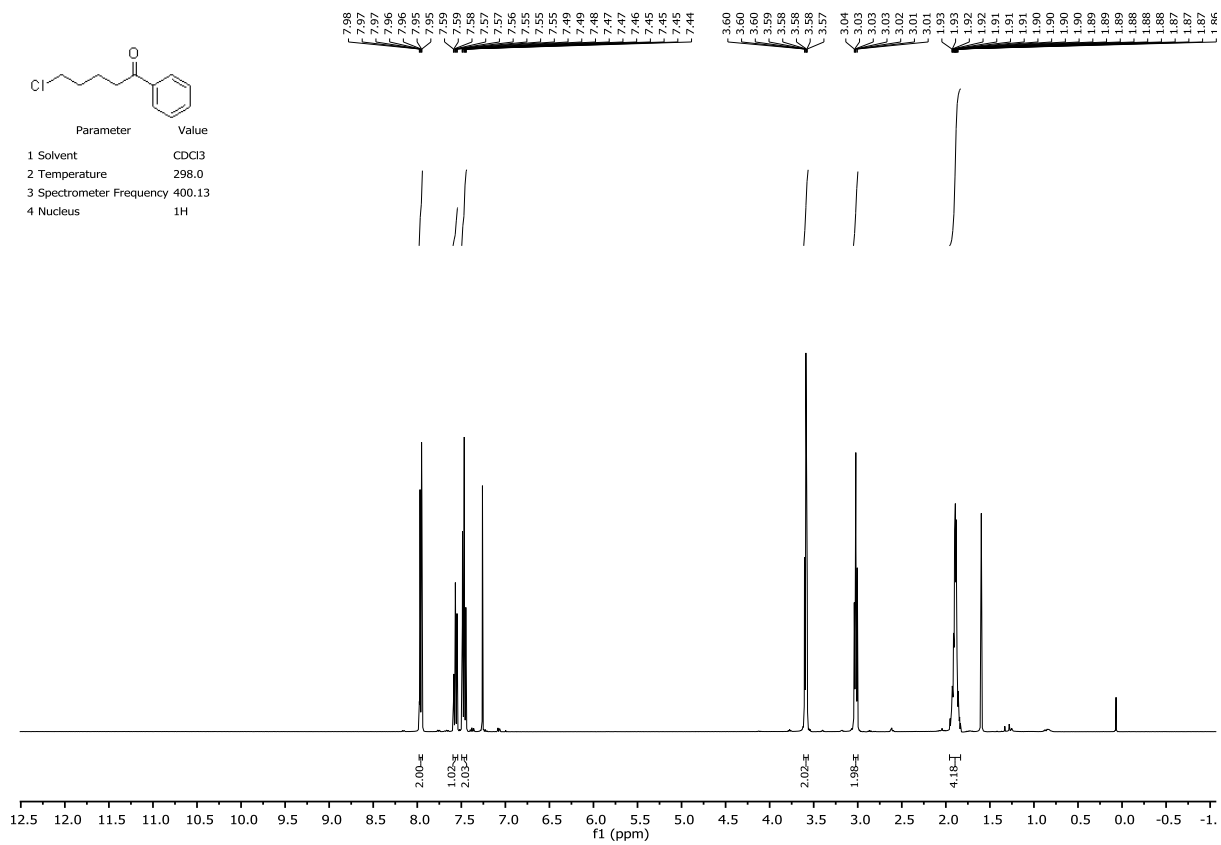
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



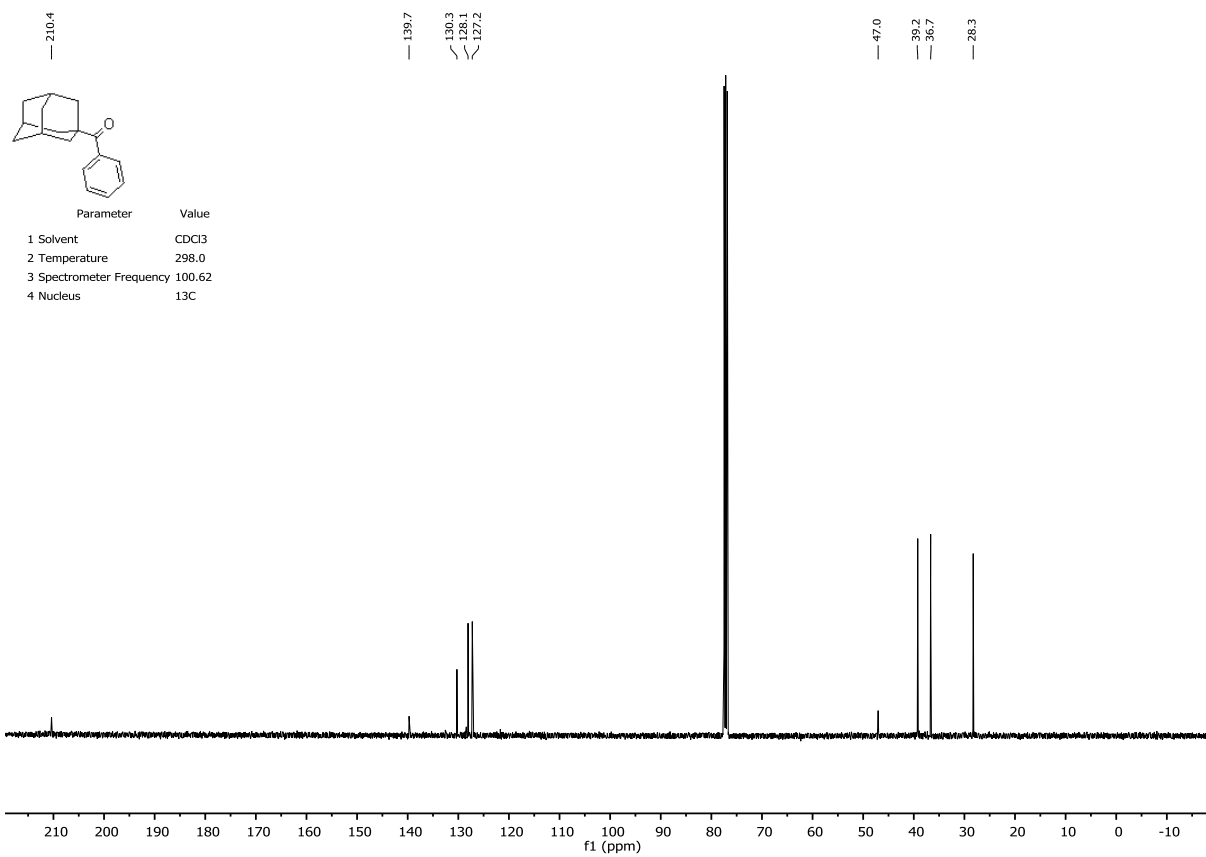
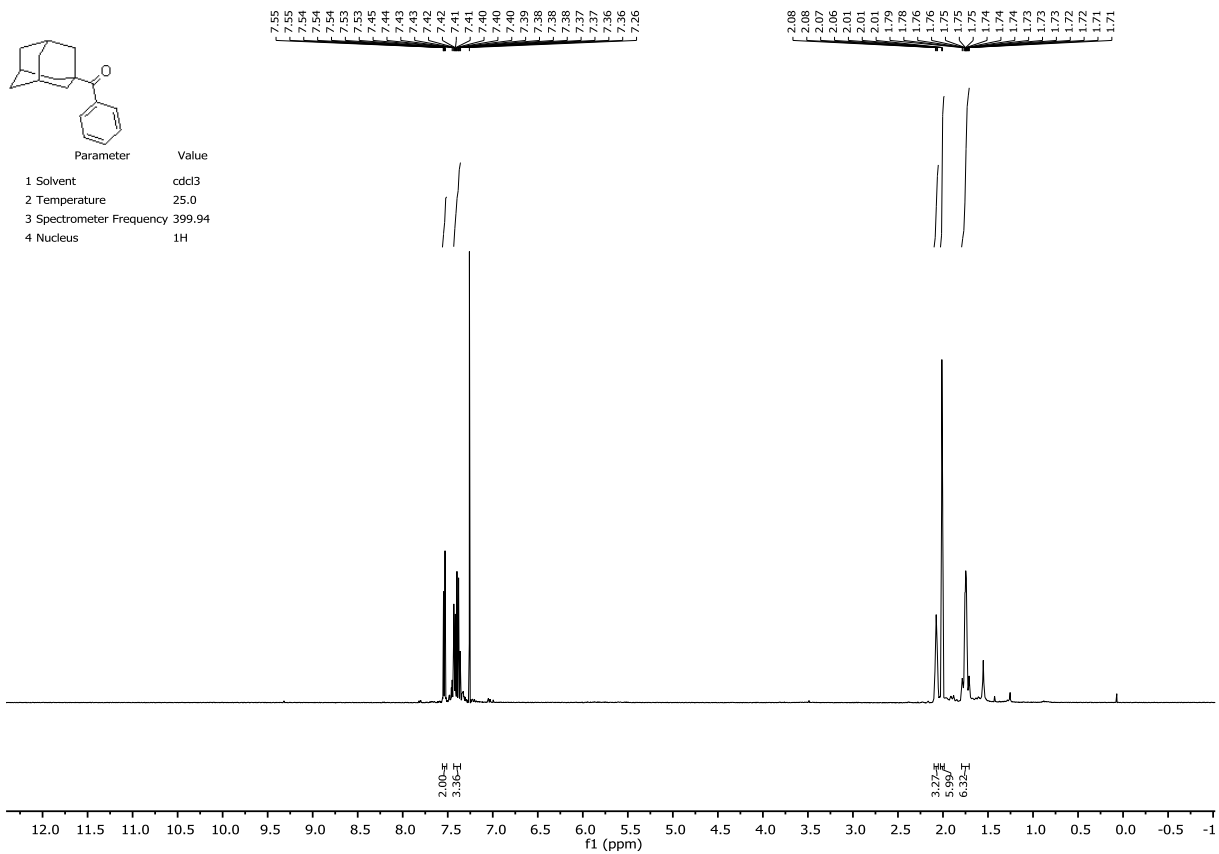
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	¹³ C



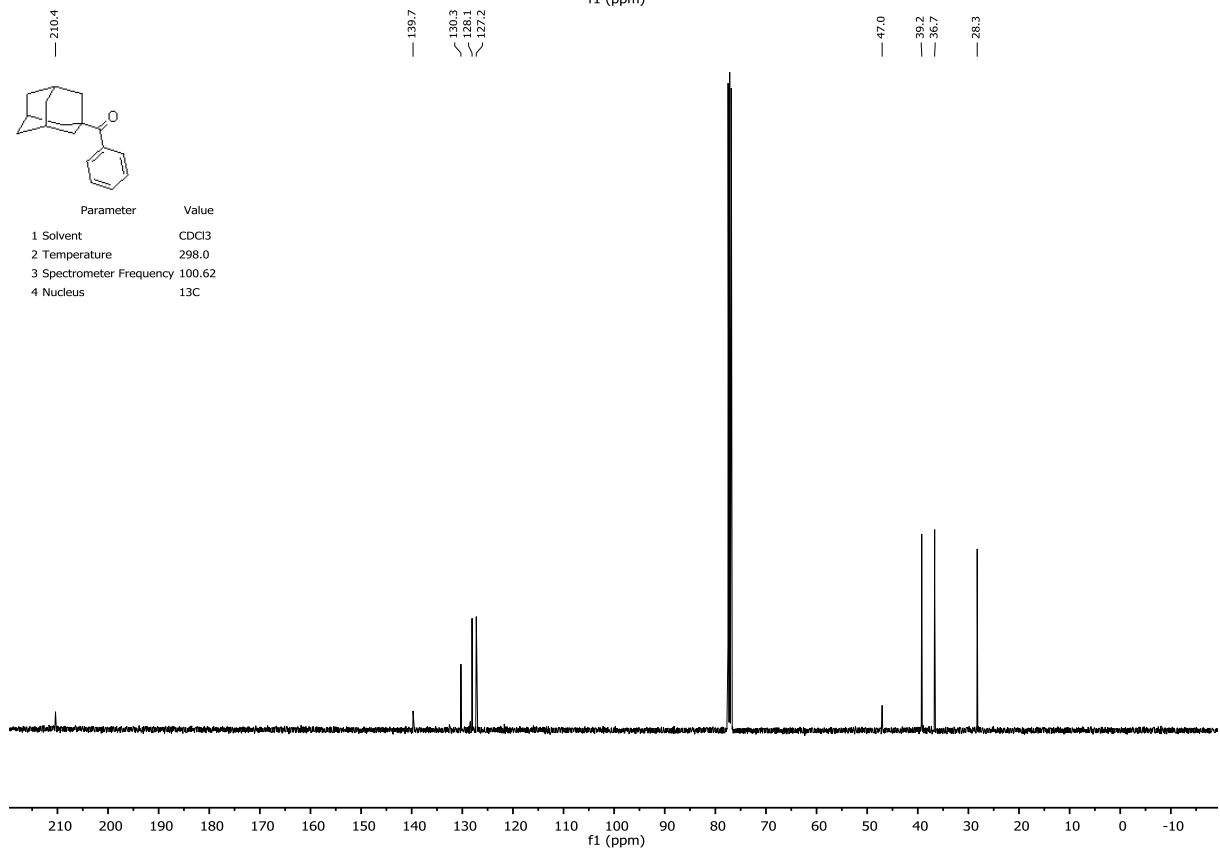
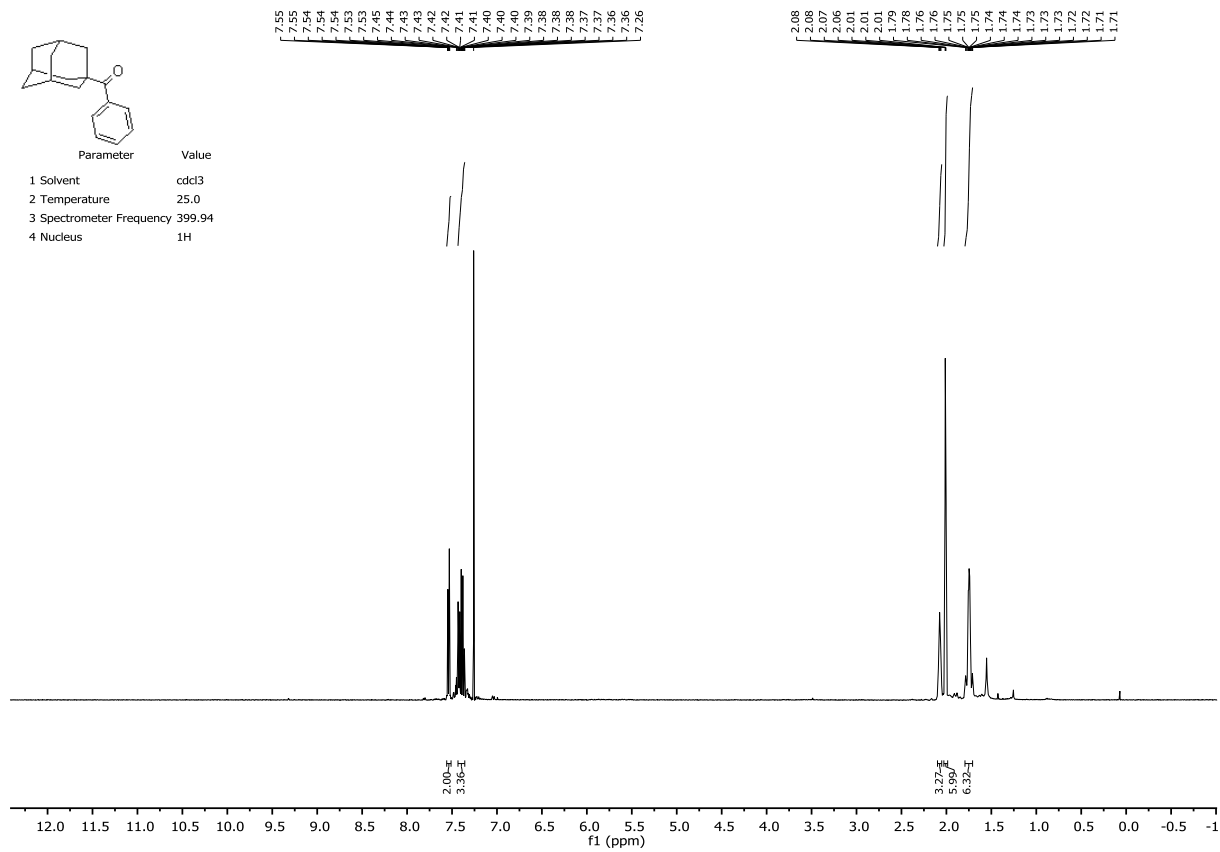
1,4-Diphenylbutan-1-one⁸ (12) CAS: 5407-91-0 (from 3-bromo-1-phenylpropane, 55 h irradiation)



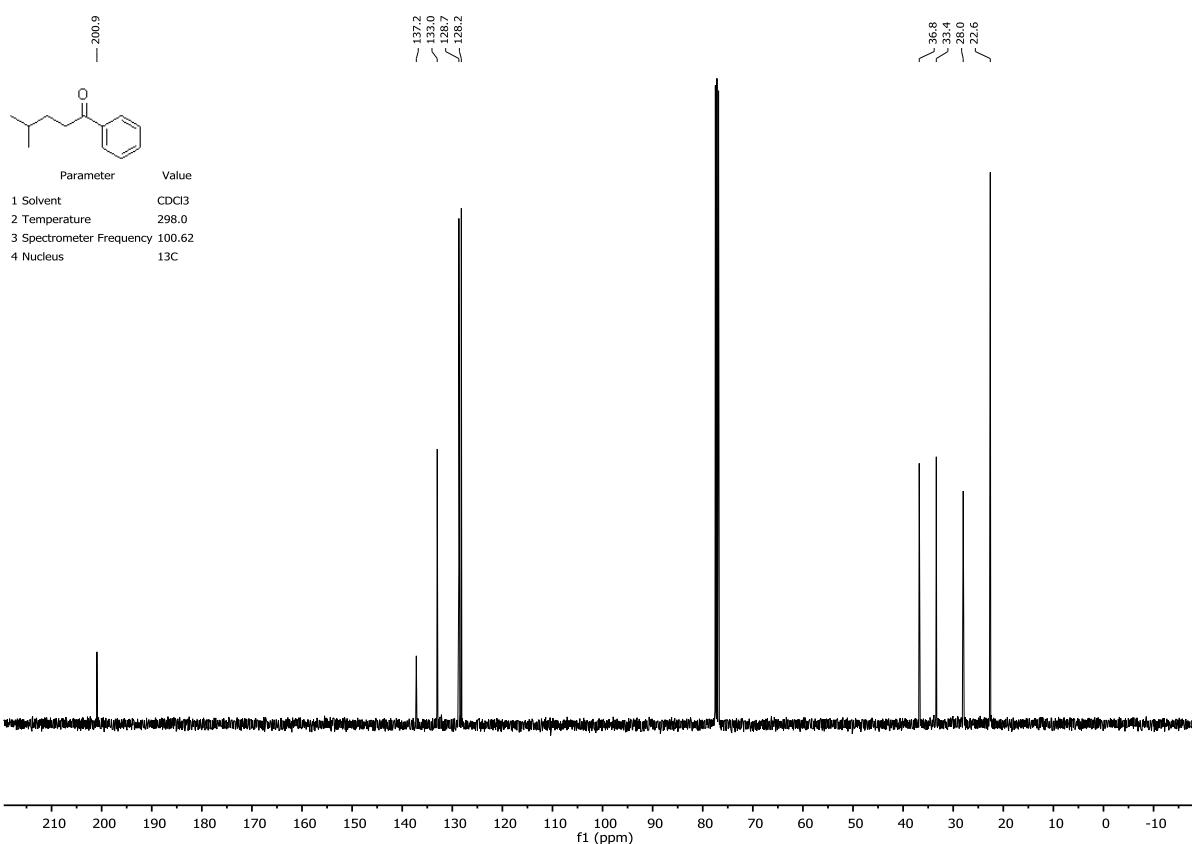
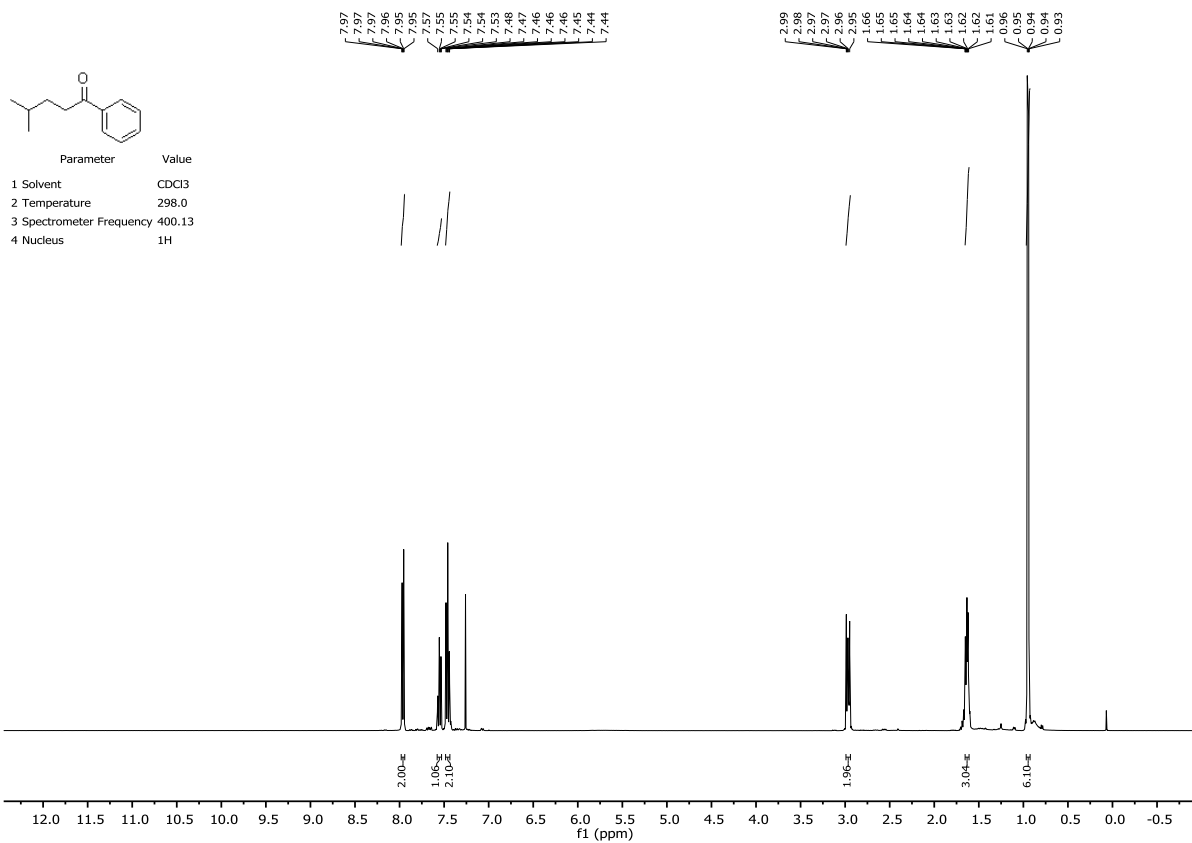
5-Chloro-1-phenylpentan-1-one¹¹ (13) CAS: 942-93-8 (from 4-bromo-1-chlorobutane)



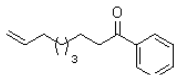
((3r,5r,7r)-Adamantan-1-yl)(phenyl)methanone¹² (14) CAS: 31919-47-8 (from 1-iodoadamantane)



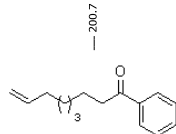
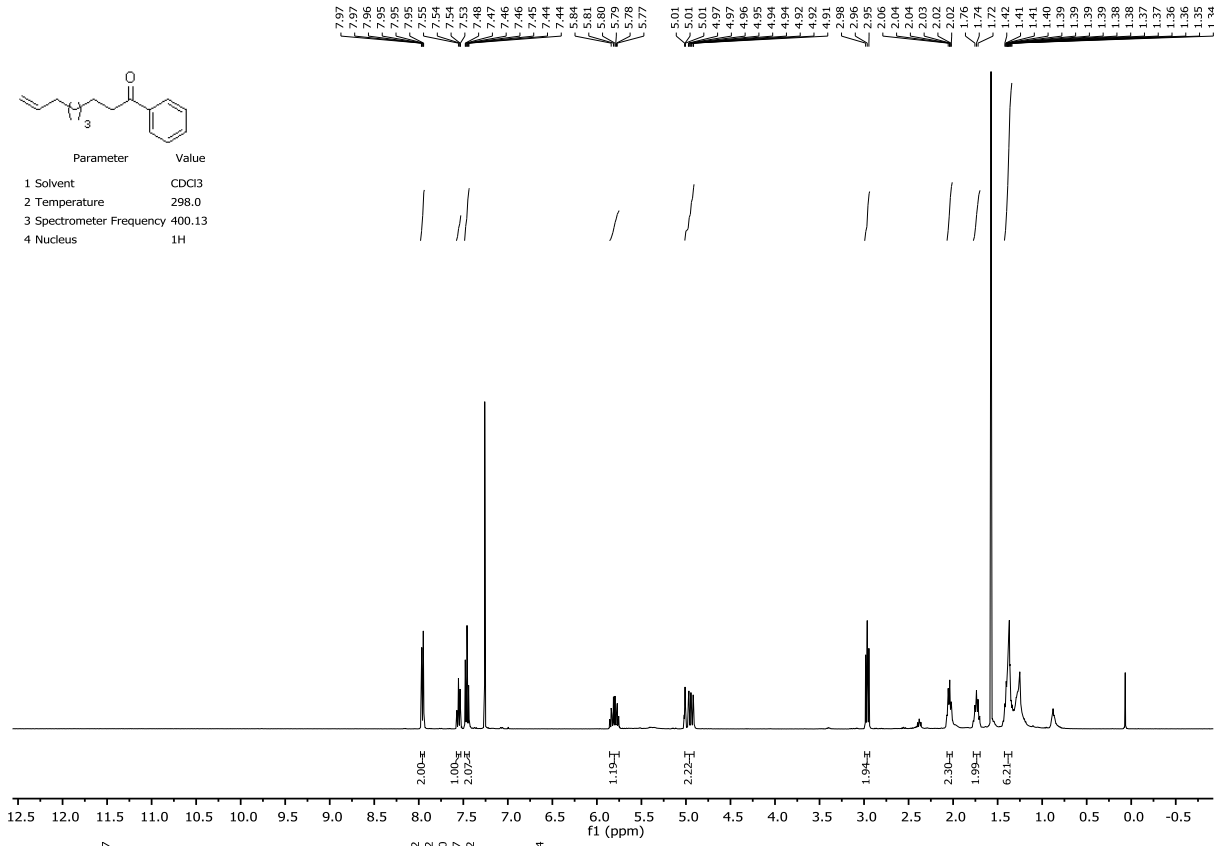
((3r,5r,7r)-Adamantan-1-yl)(phenyl)methanone¹² (14) CAS: 31919-47-8 (from 1-bromoadamantane)



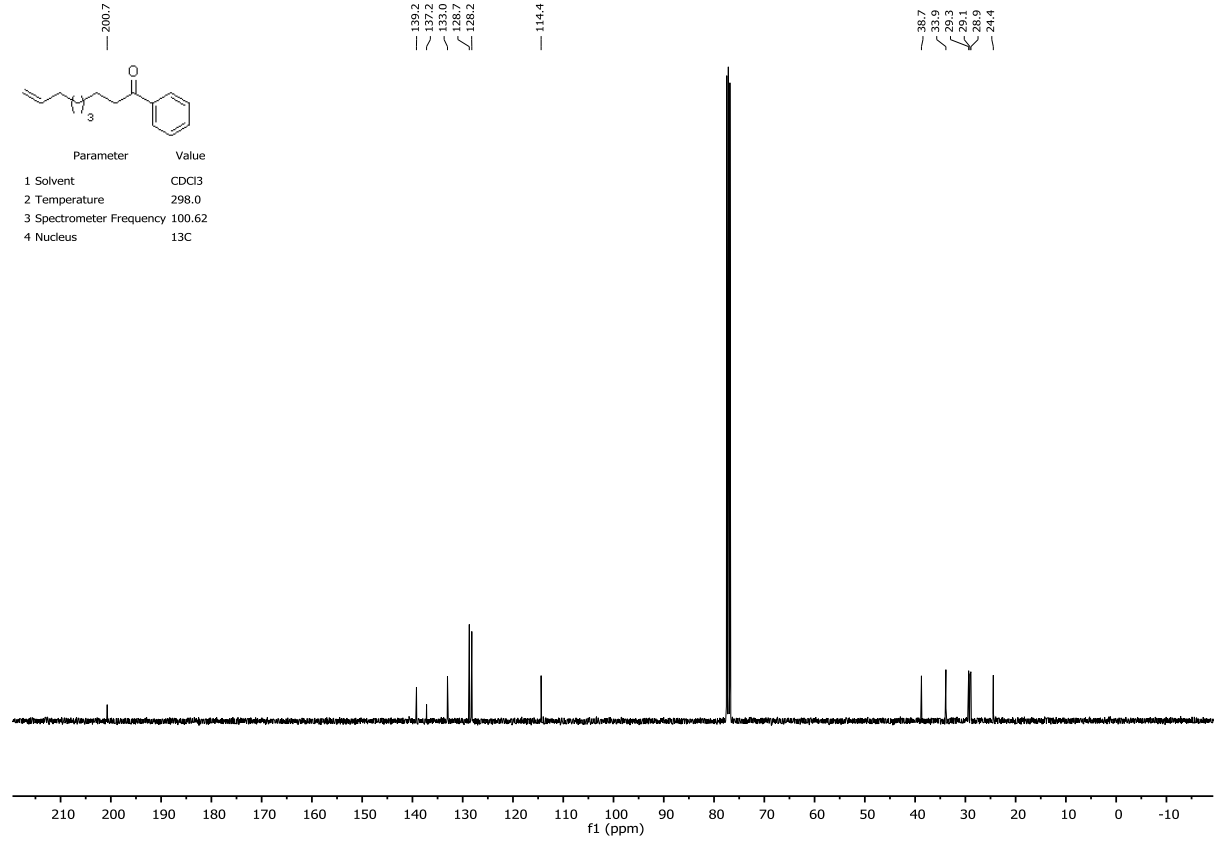
4-Methyl-1-phenylpentan-1-one⁹ (18) CAS: 2050-07-9



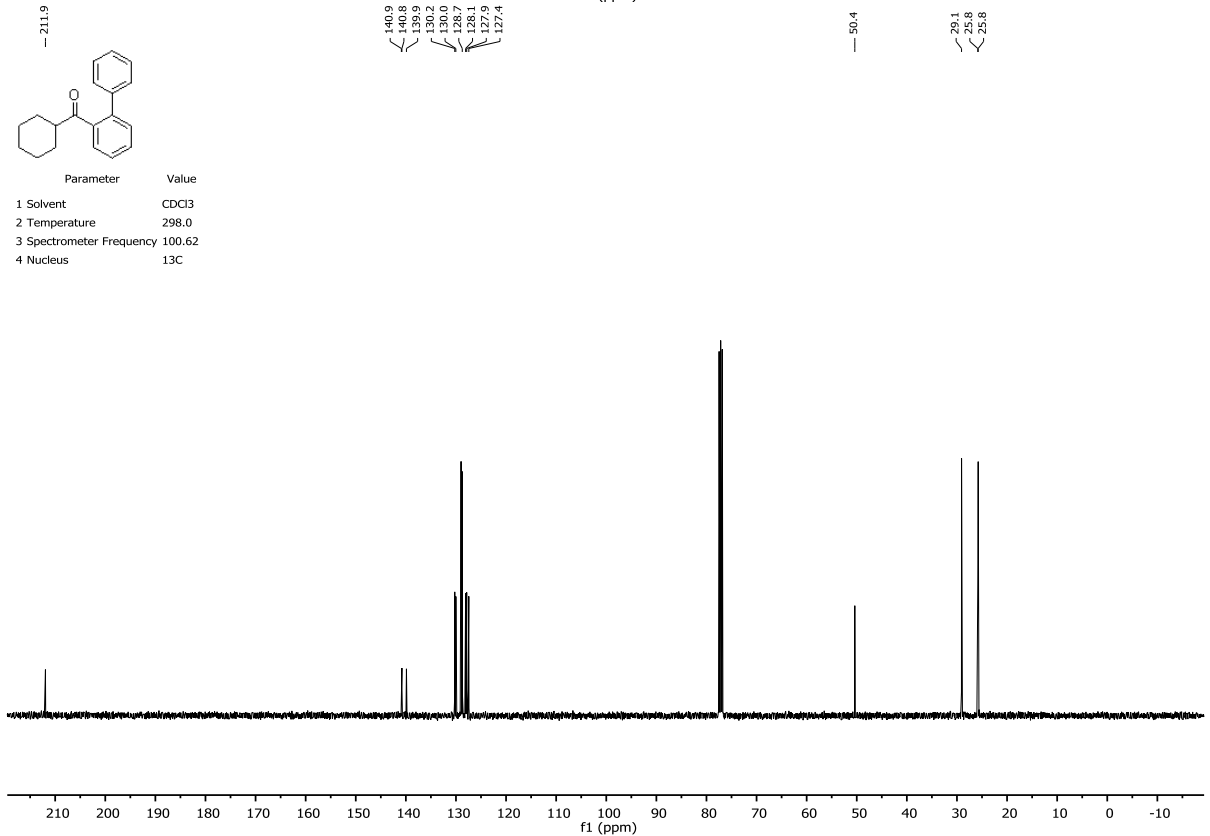
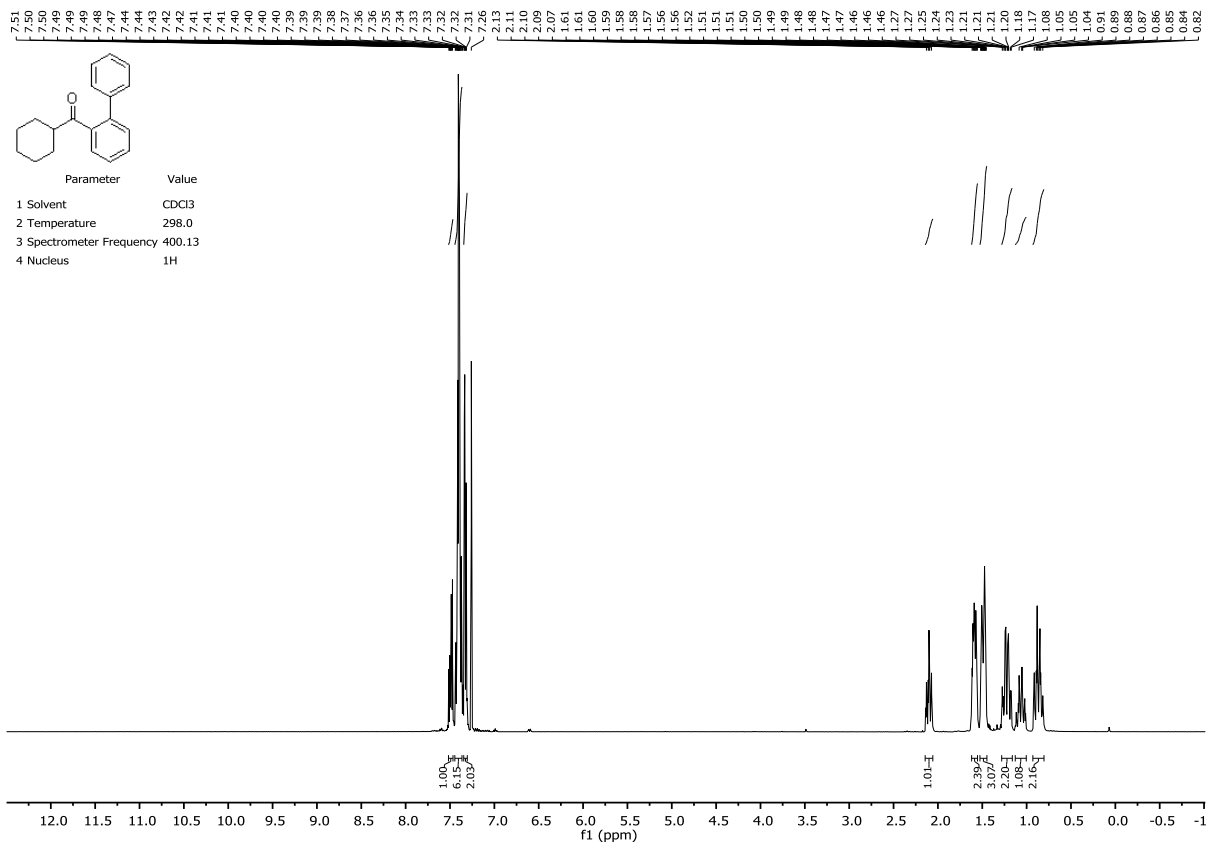
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	1H



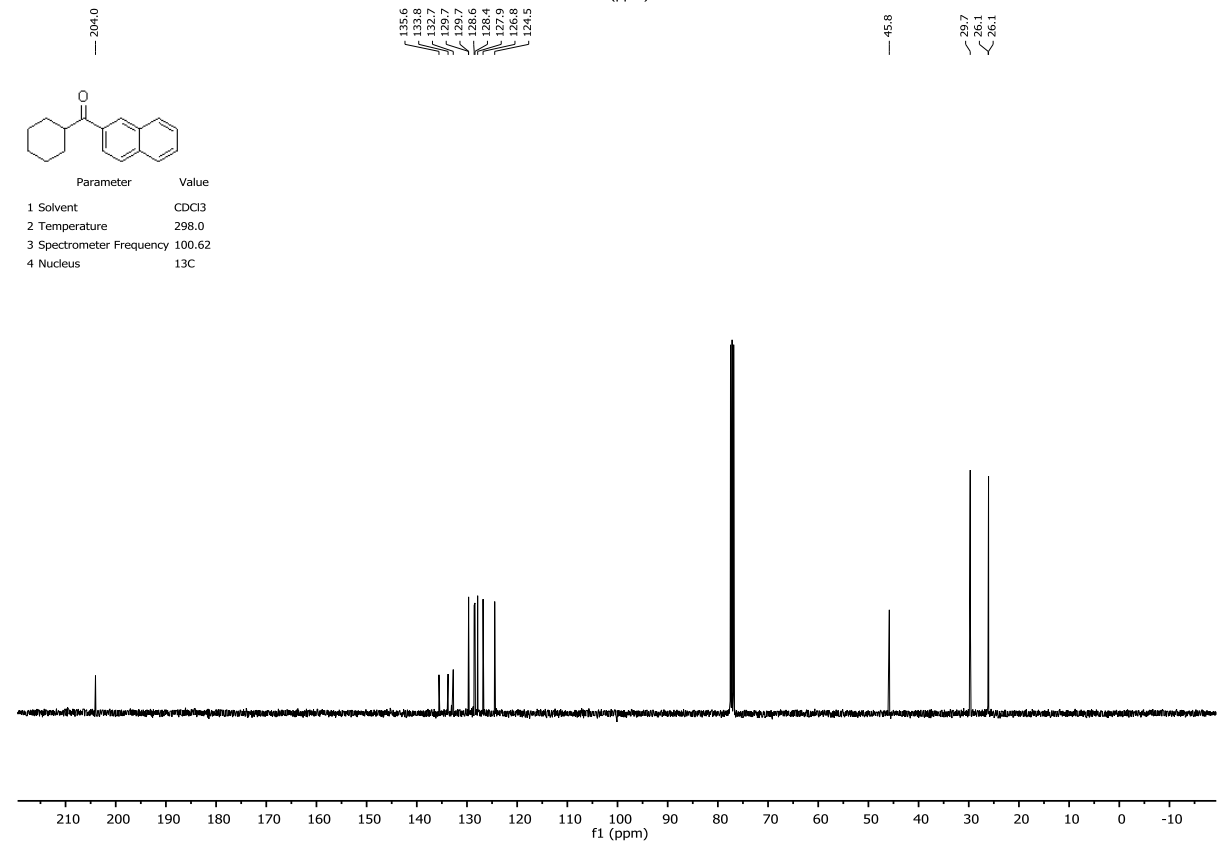
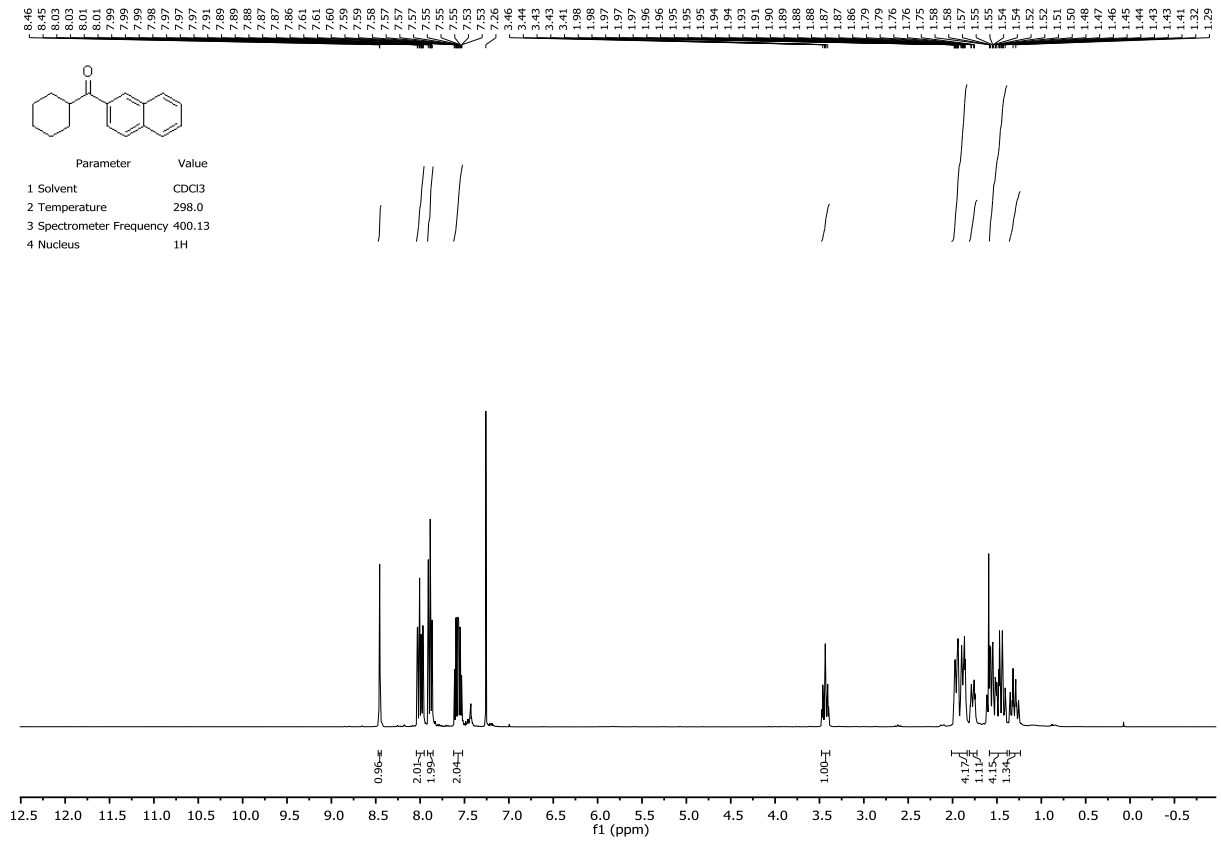
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	13C



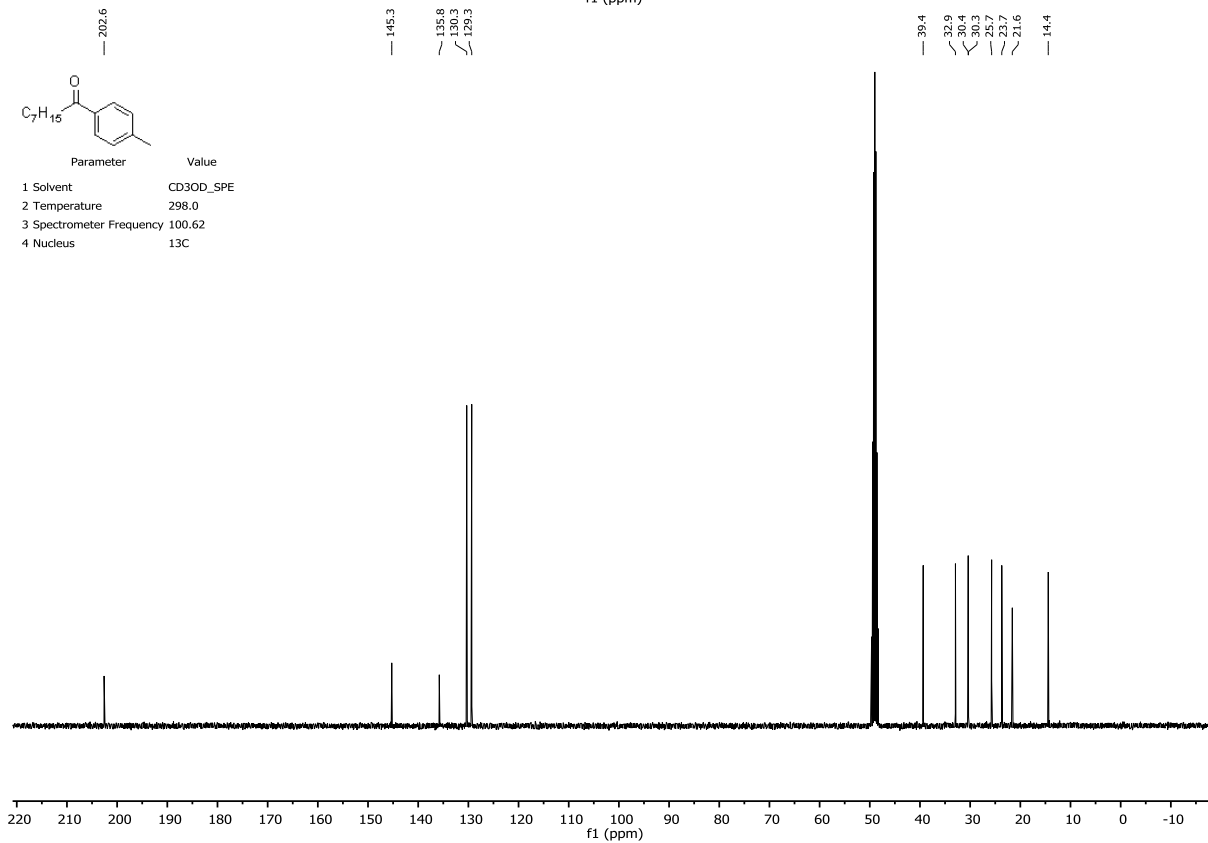
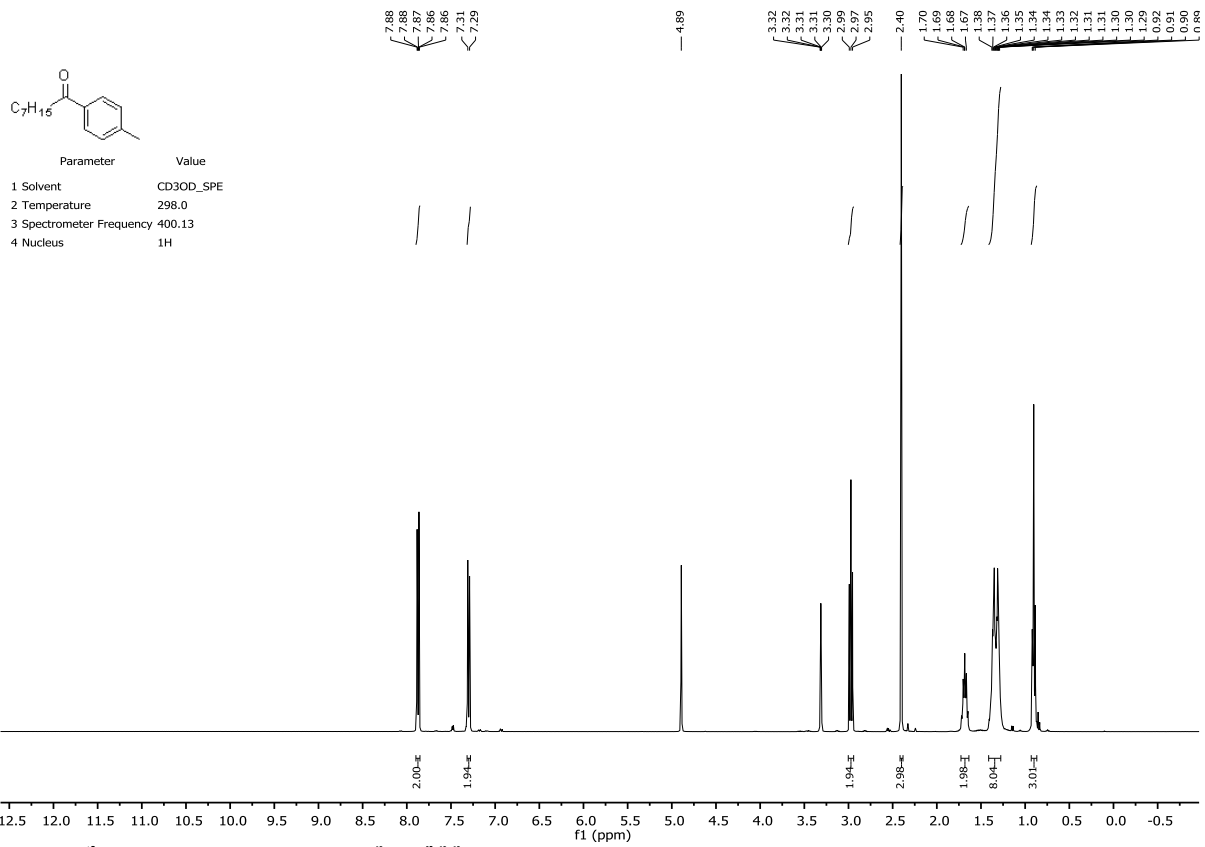
1-Phenylnon-8-en-1-one (19) CAS: 148056-64-8



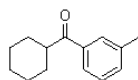
[1,1'-Biphenyl]-2-yl(cyclo-hexyl)methanone (20) CAS: 963-92-8



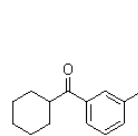
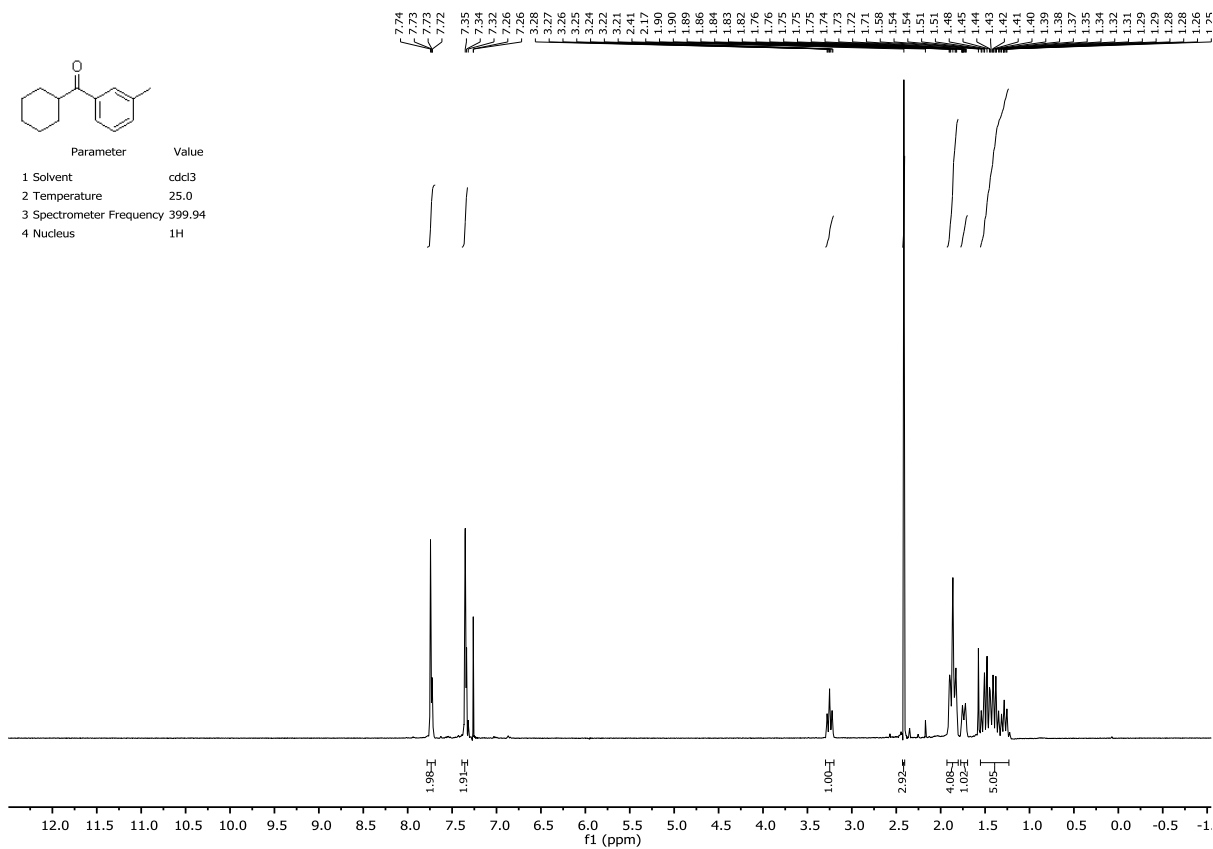
Cyclohexyl(naphthalen-2-yl)-methanone (21) CAS: 10404-26-9



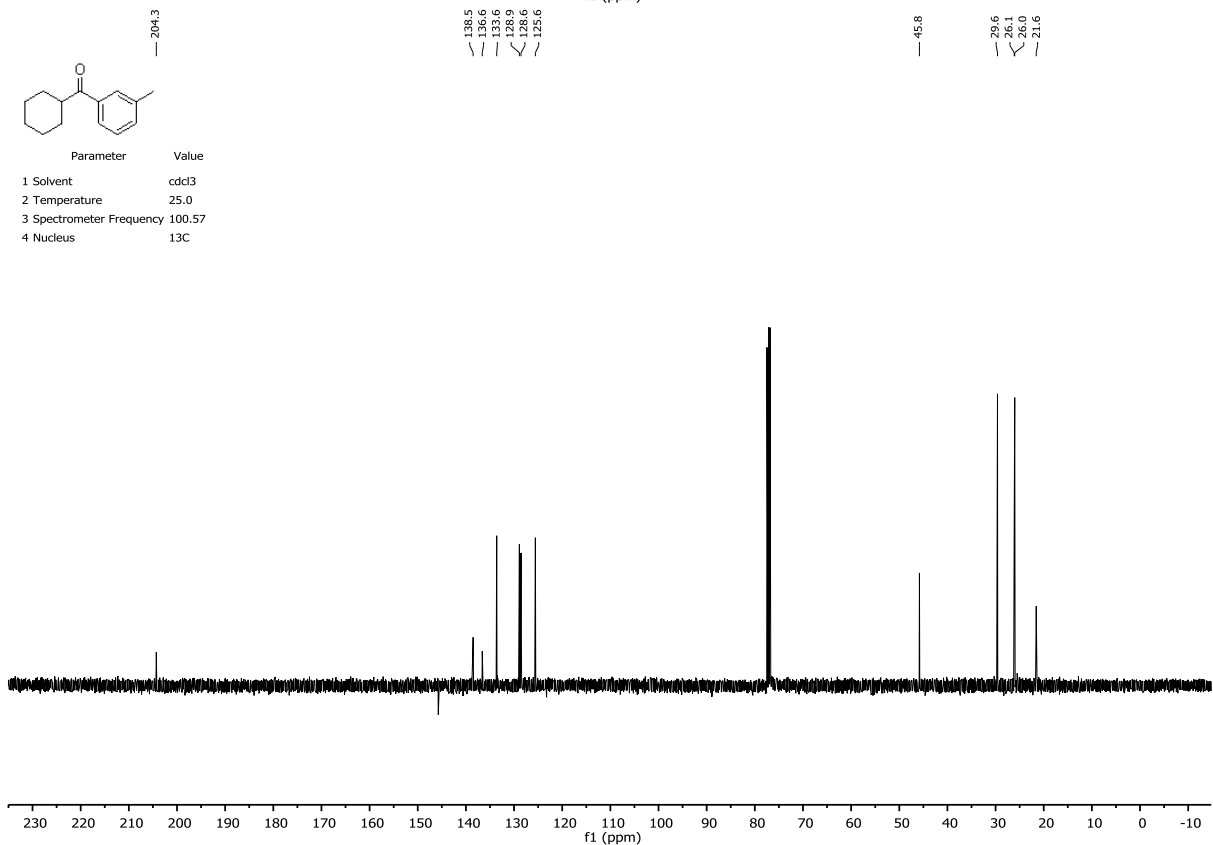
1-(*p*-Tolyl)octan-1-one¹³ (22) CAS: 2789-44-8



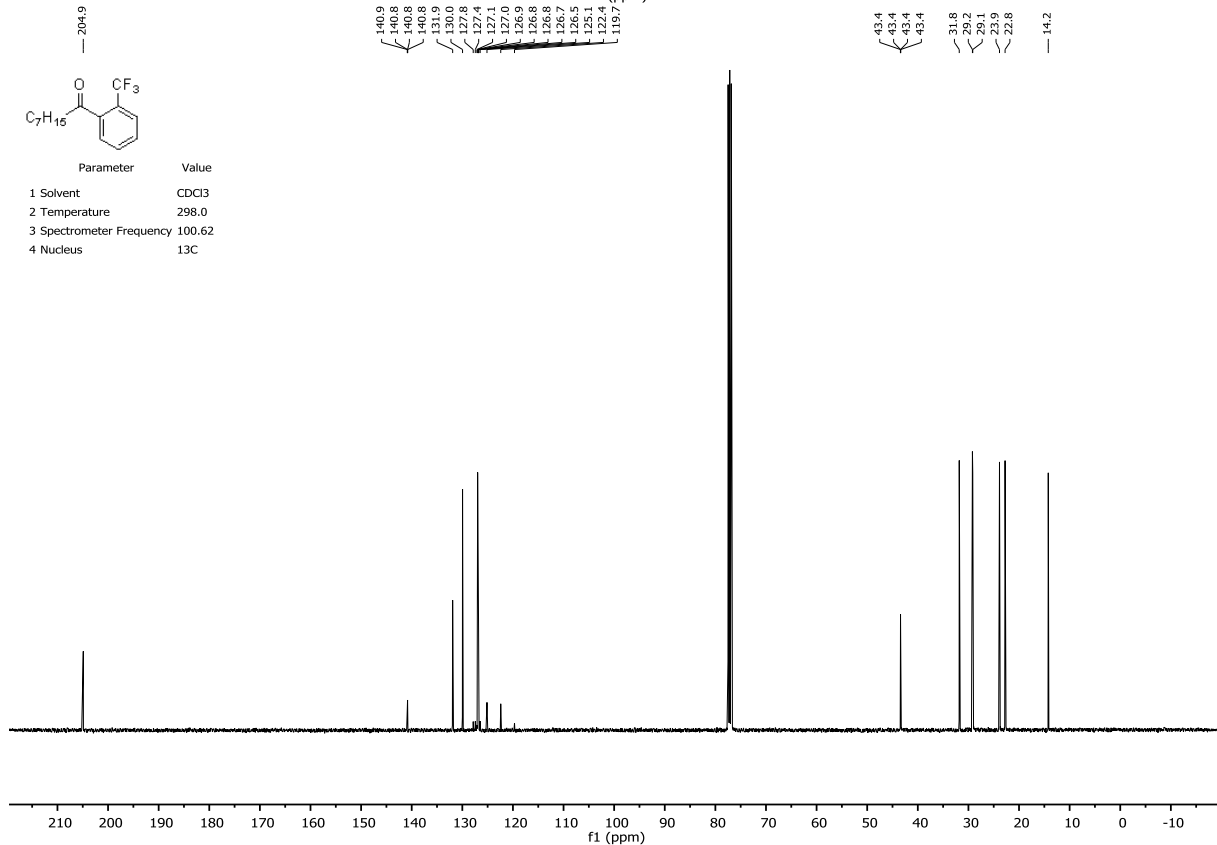
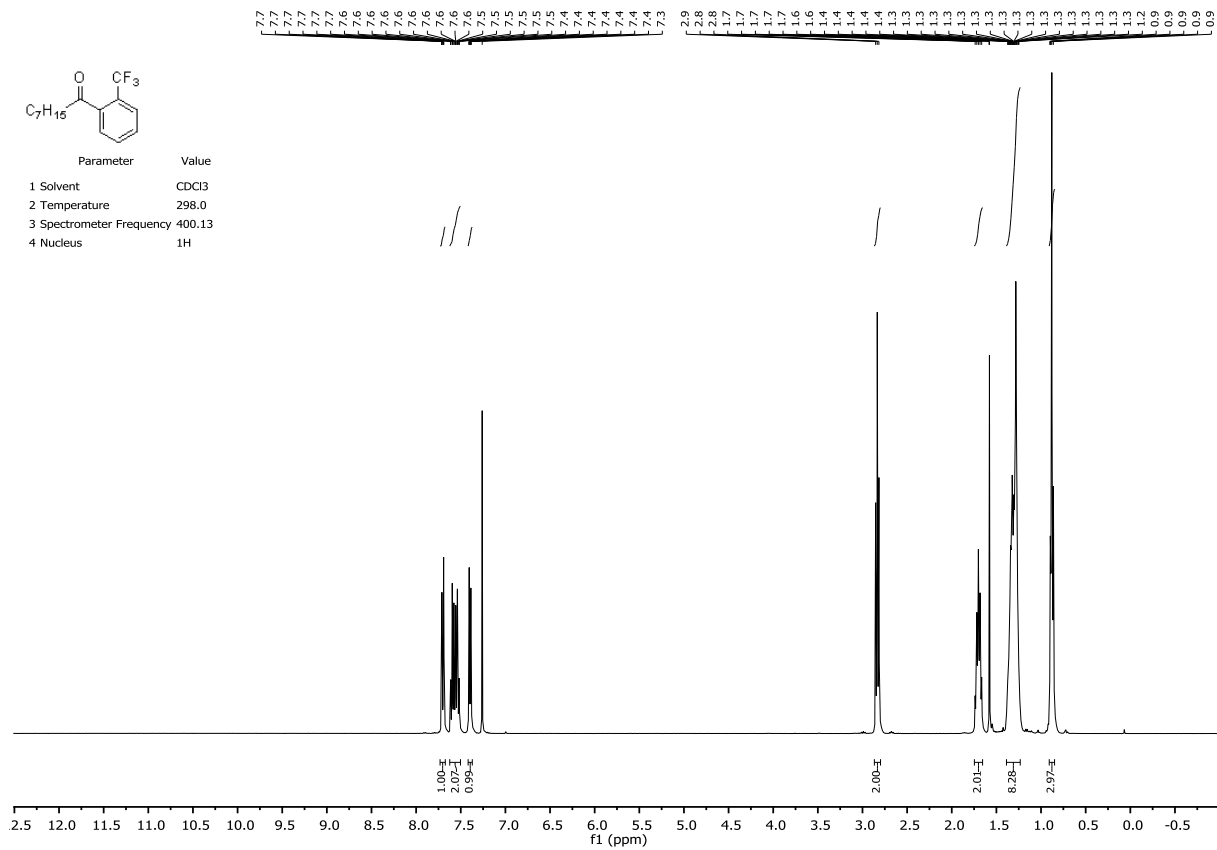
Parameter	Value
1 Solvent	cdcl3
2 Temperature	25.0
3 Spectrometer Frequency	399.94
4 Nucleus	1H



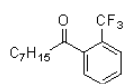
Parameter	Value
1 Solvent	cdcl3
2 Temperature	25.0
3 Spectrometer Frequency	100.57
4 Nucleus	13C



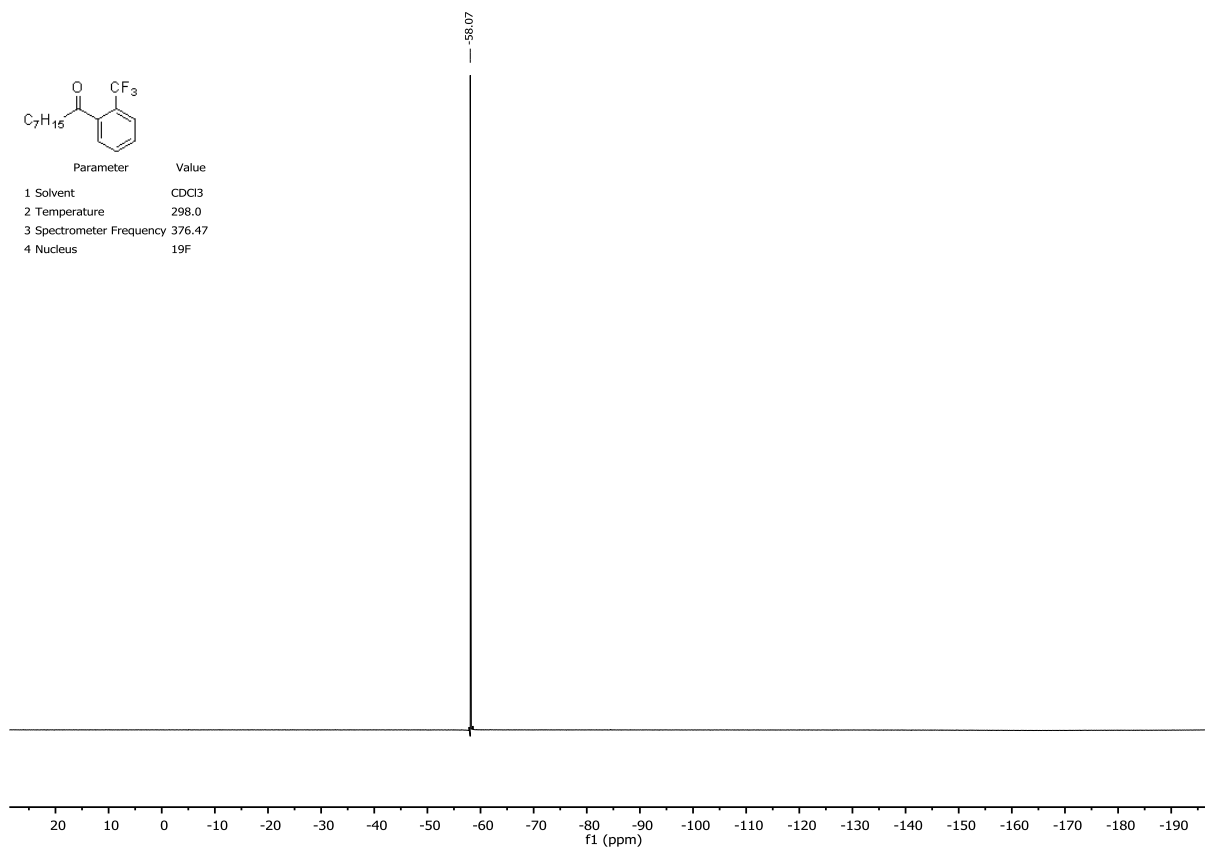
Cyclohexyl(*m*-tolyl)methanone¹⁴ (23) CAS: 3277-78-9



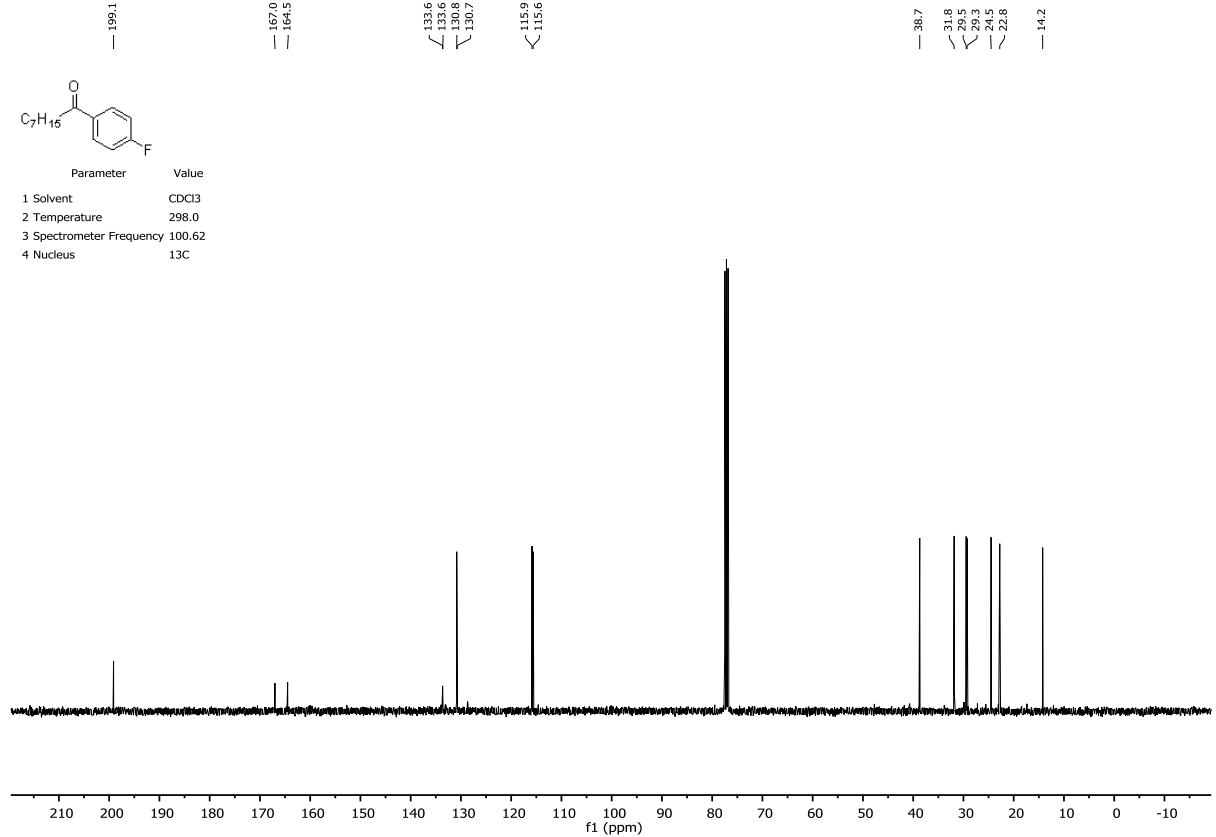
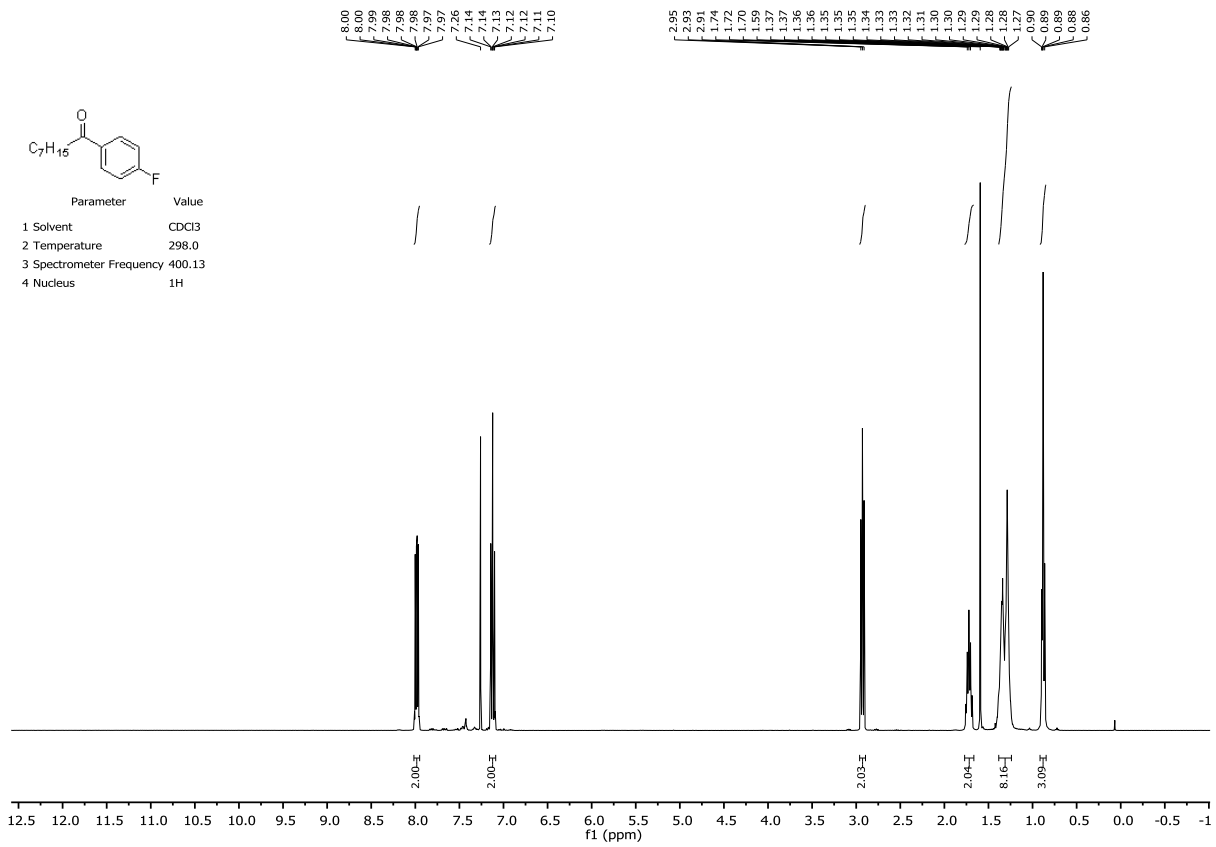
1-(2-(Trifluoromethyl)phenyl)octan-1-one (24) CAS: 1778855-78-9



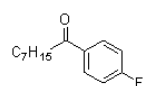
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	376.47
4 Nucleus	¹⁹ F



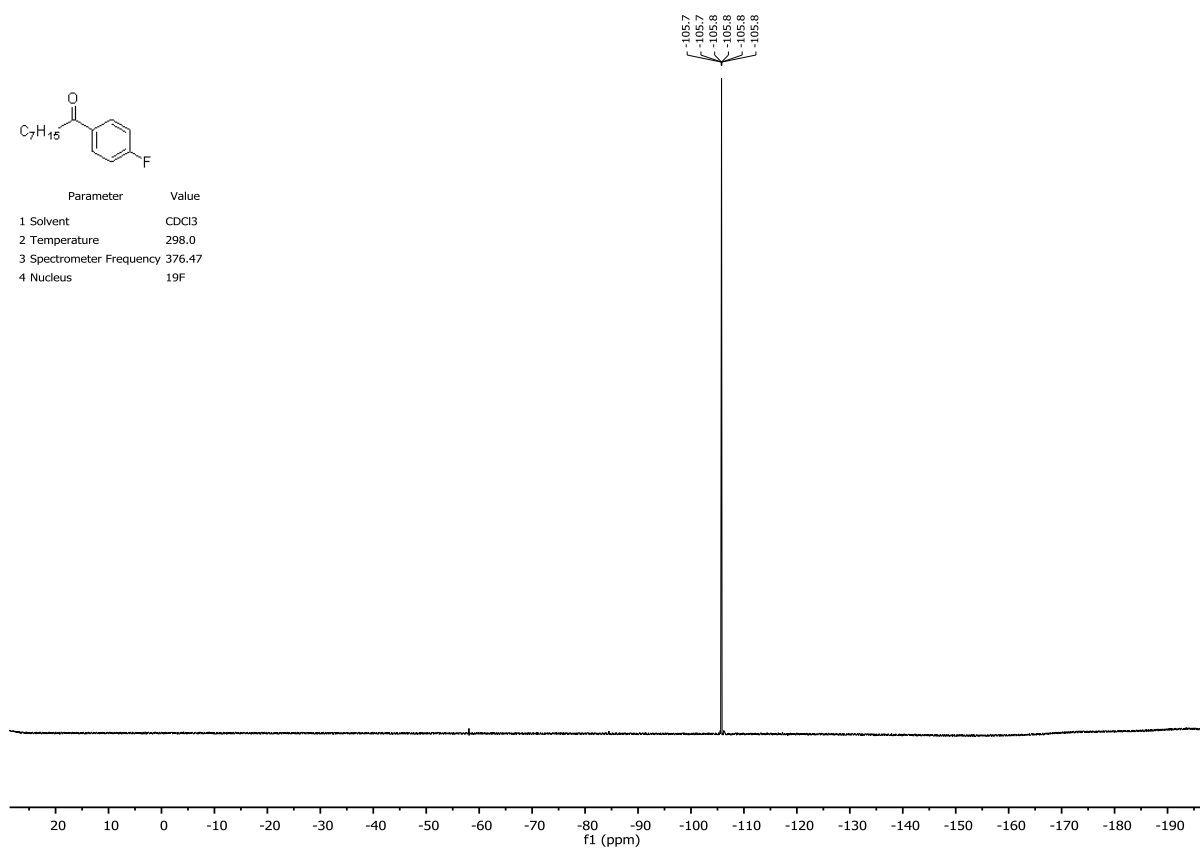
1-(2-(Trifluoromethyl)-phenyl)octan-1-one (24) CAS: 1778855-78-9



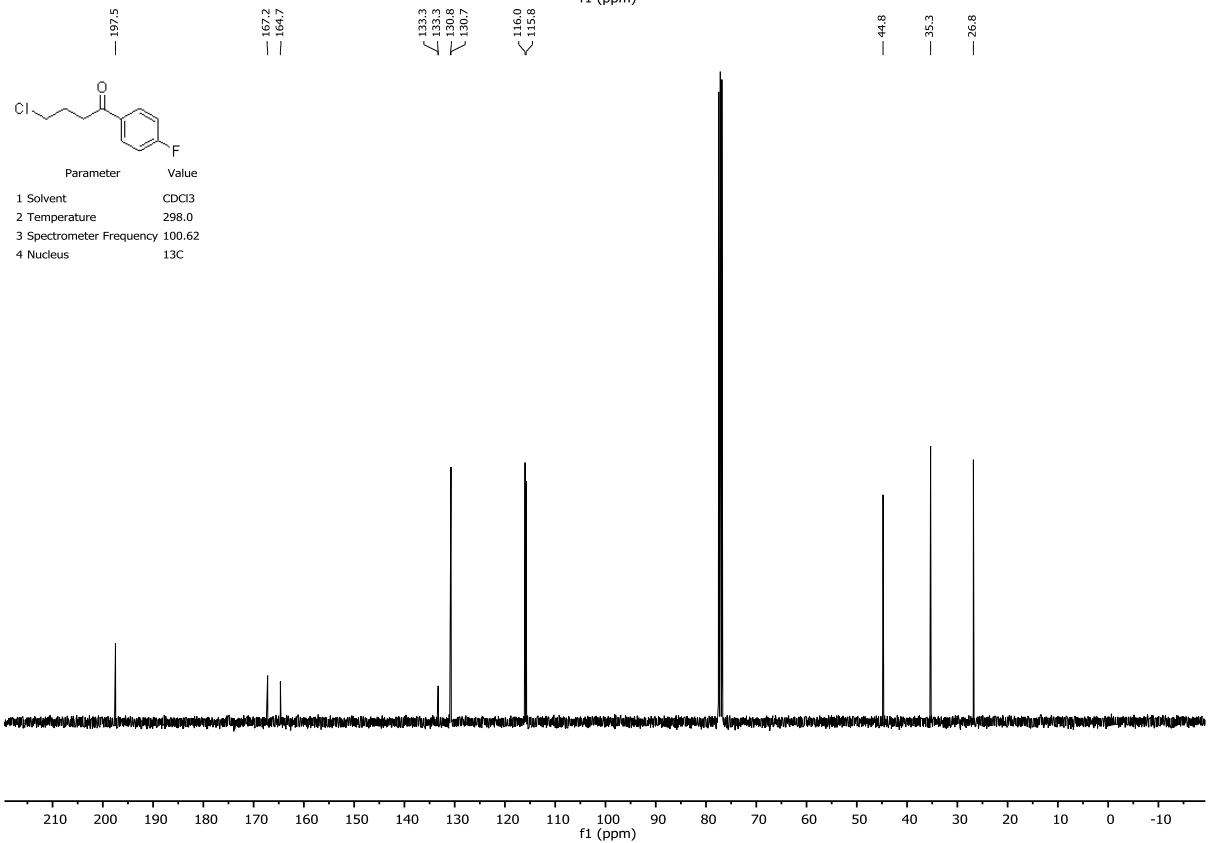
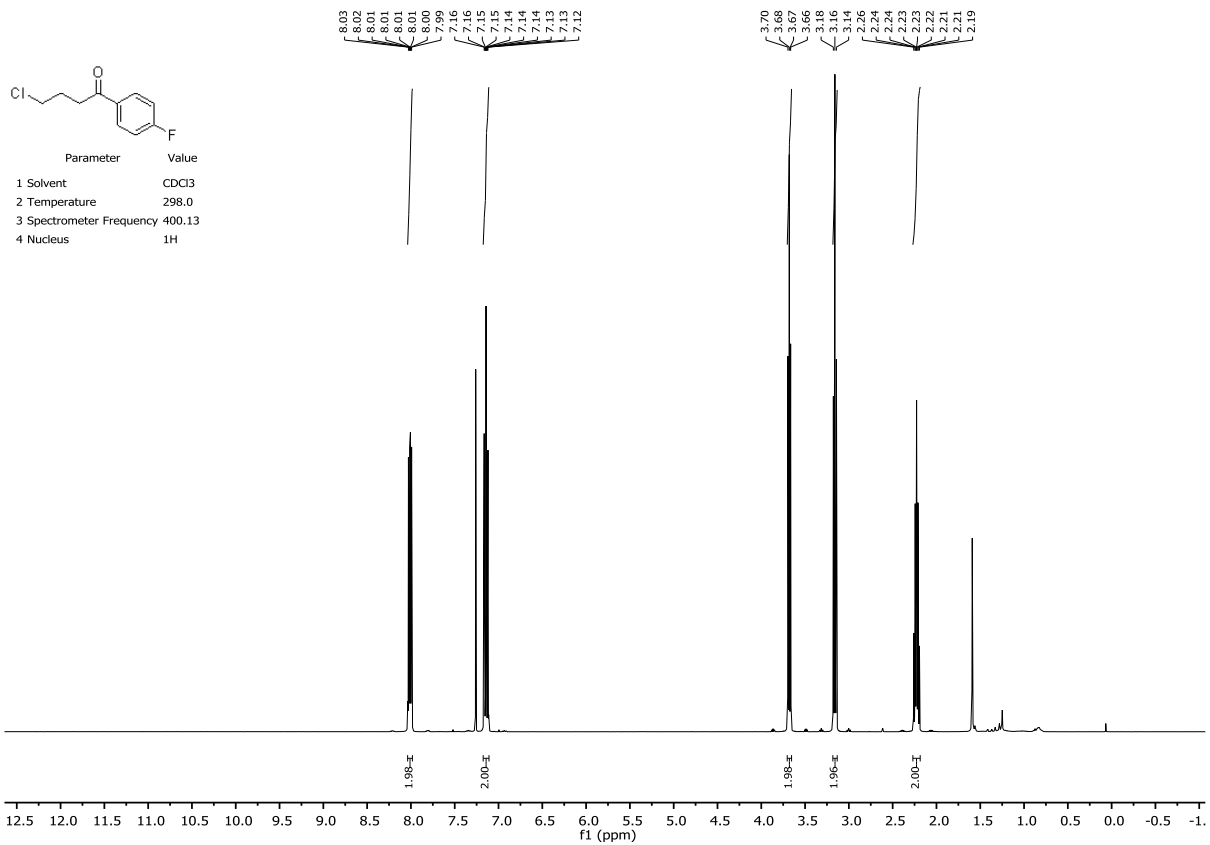
1-(4-Fluorophenyl)octan-1-one¹⁵ (25) CAS: 111829-15-3



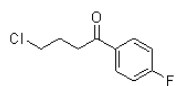
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	376.47
4 Nucleus	19F



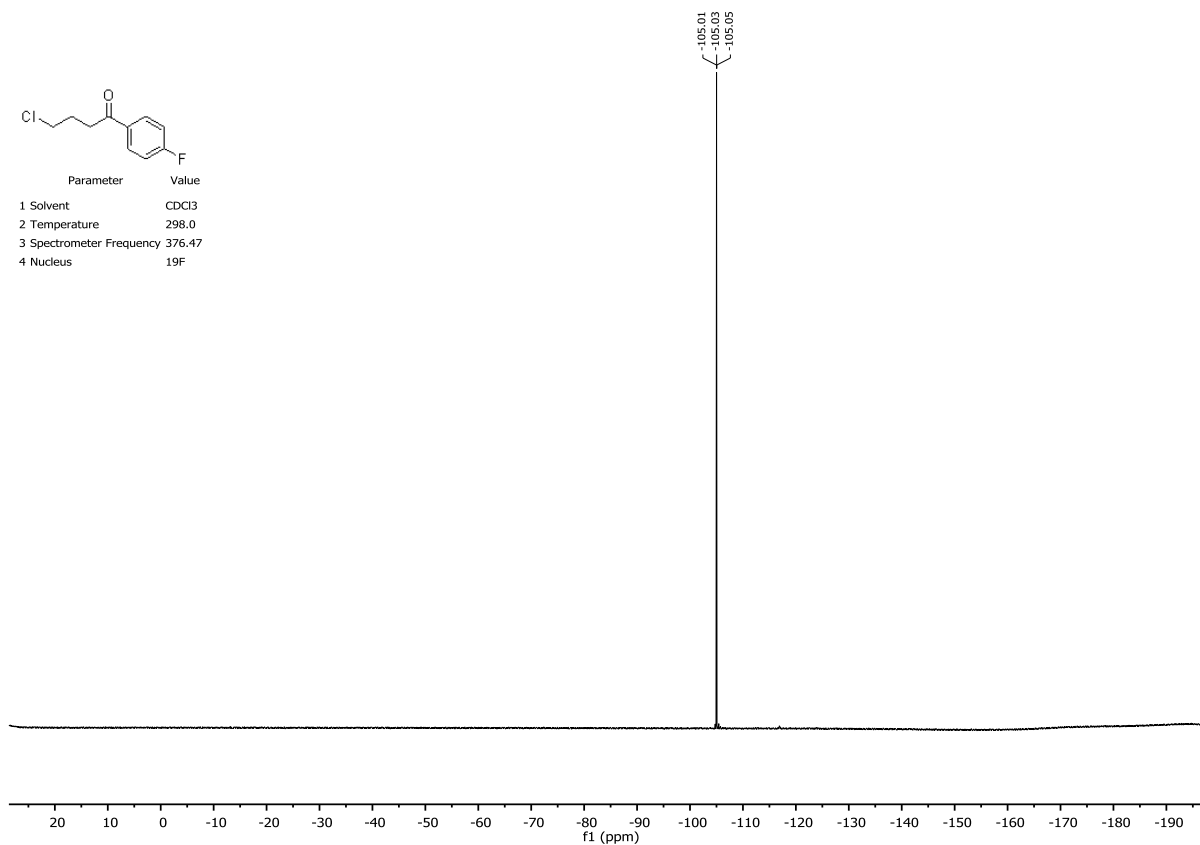
1-(4-Fluorophenyl)octan-1-one¹⁵ (25) CAS: 111829-15-3



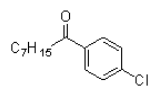
4-Chloro-1-(4-fluorophenyl)-butan-1-one¹⁶ (26) CAS: 3874-54-2



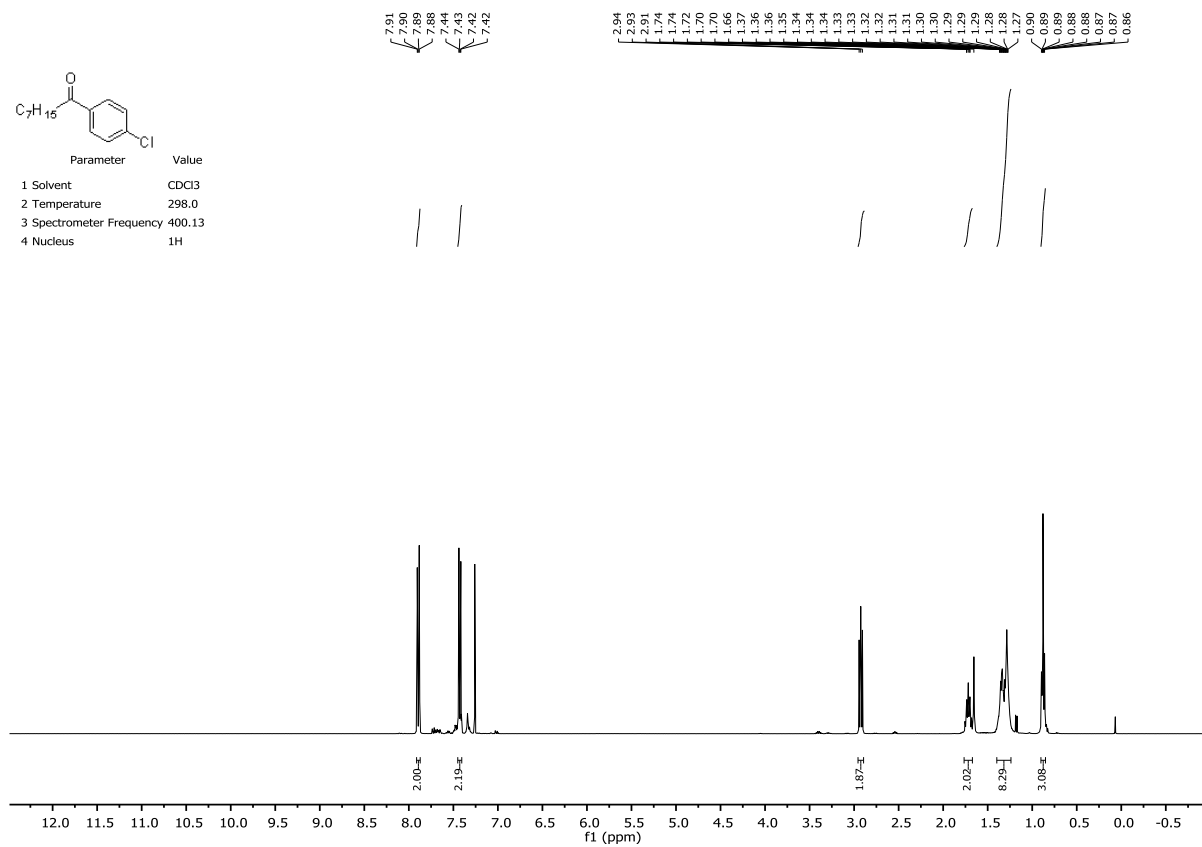
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	376.47
4 Nucleus	¹⁹ F



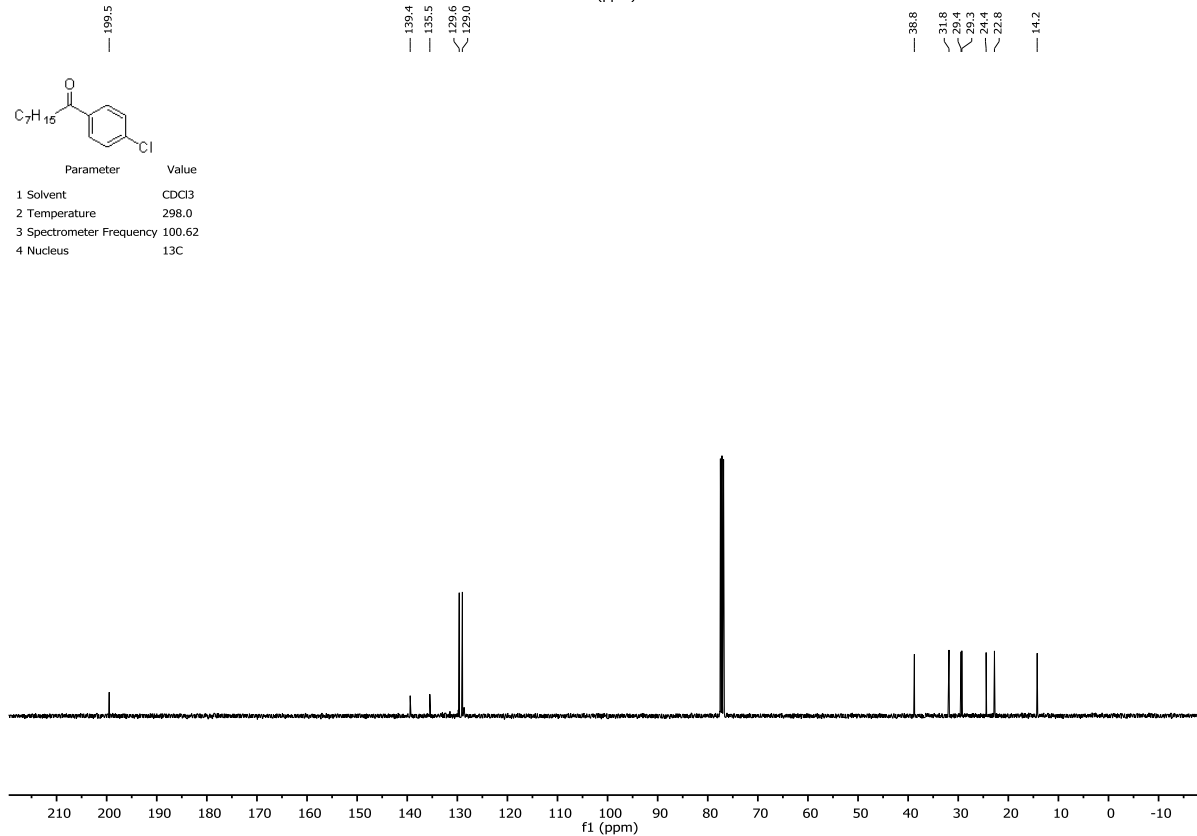
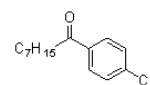
4-Chloro-1-(4-fluorophenyl)-butan-1-one¹⁶ (26) CAS: 3874-54-2



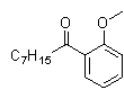
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



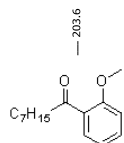
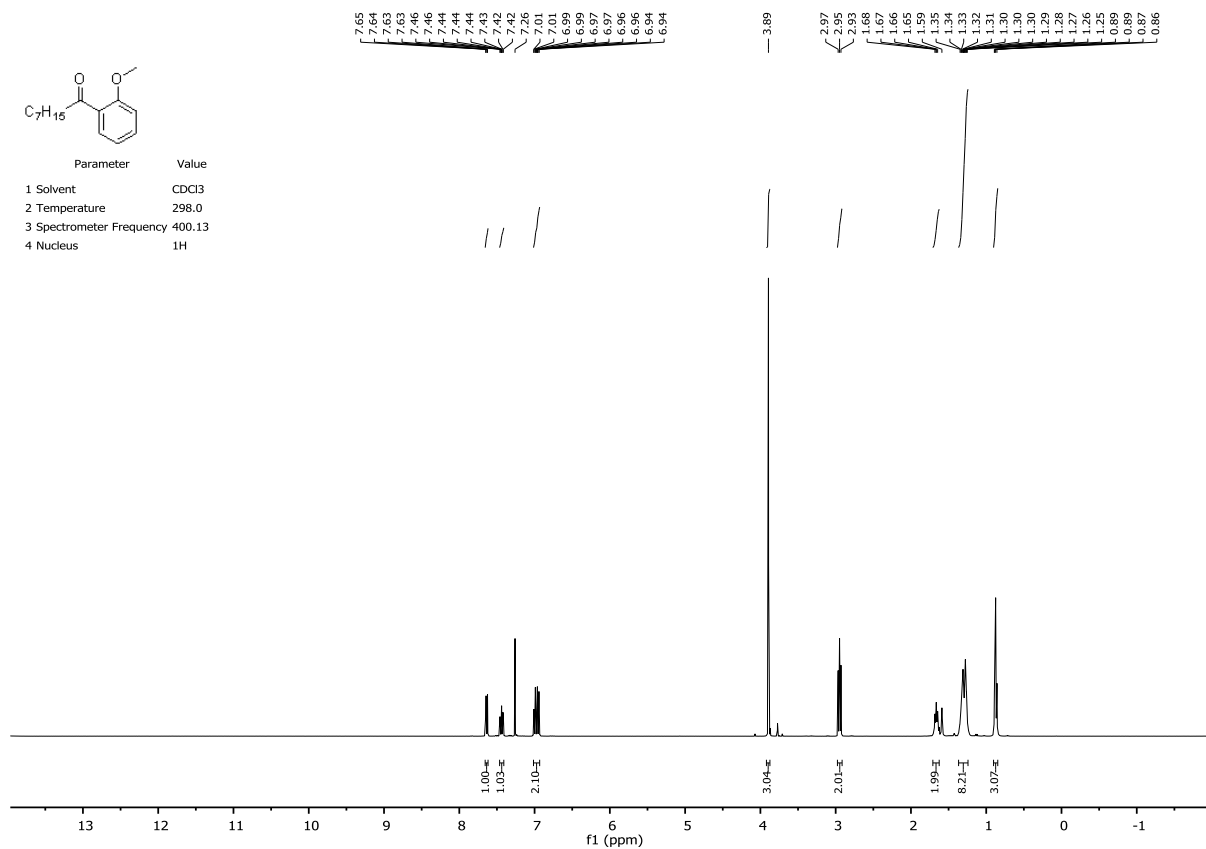
Parameter	Value
1 Solvent	CDCl ₃
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	¹³ C



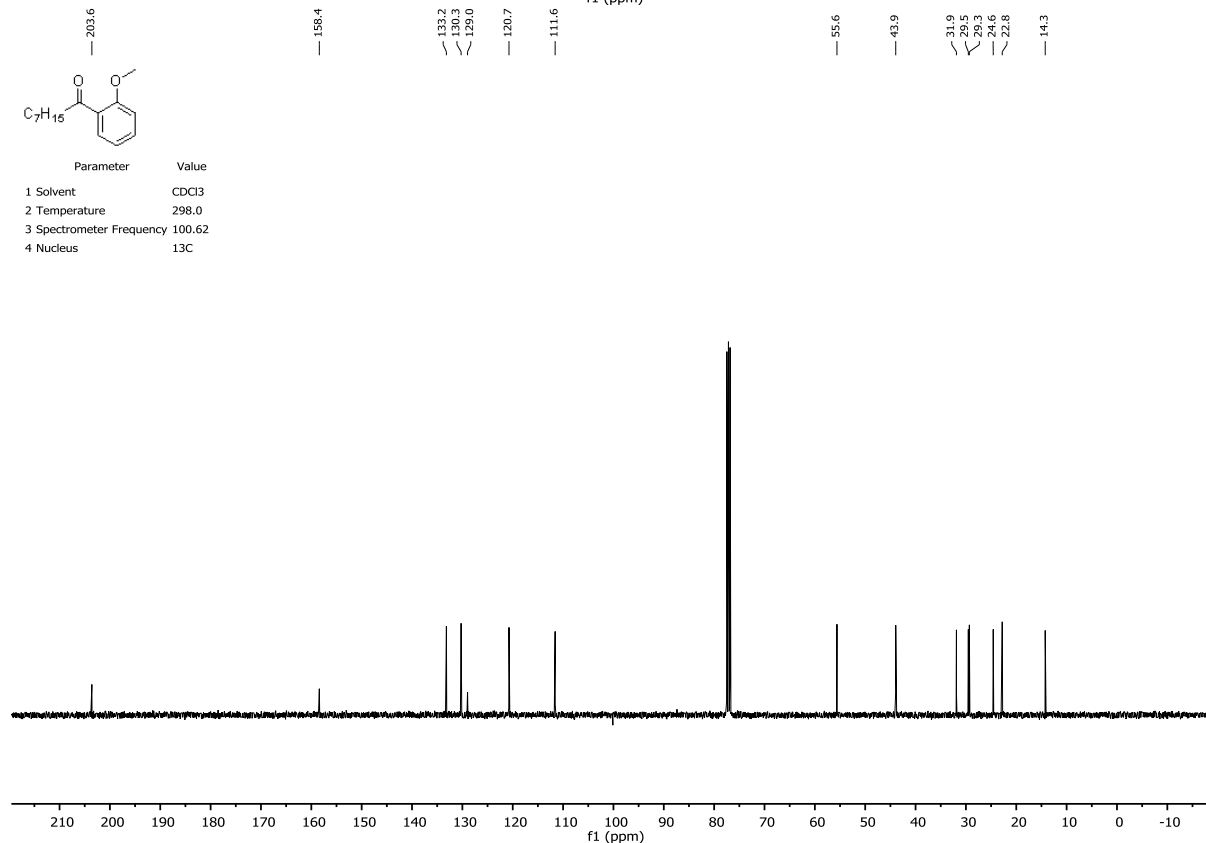
1-(4-Chlorophenyl)octan-1-one (27) CAS: 7295-52-5



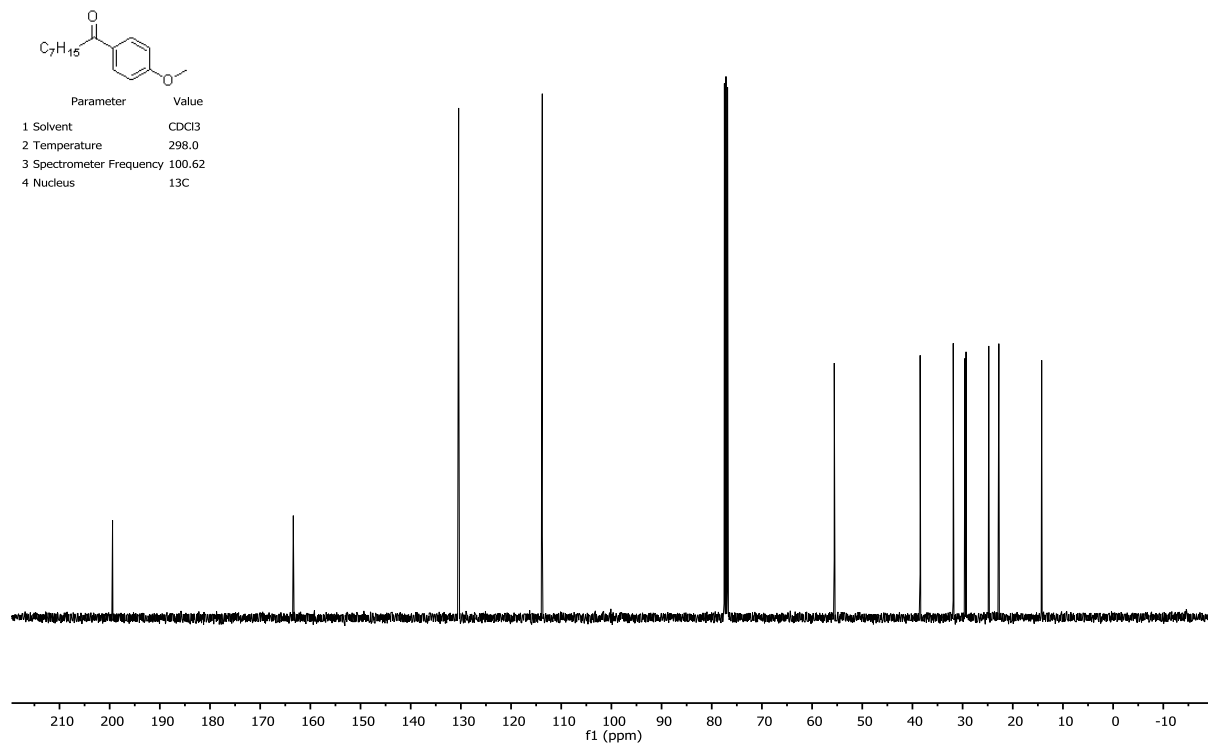
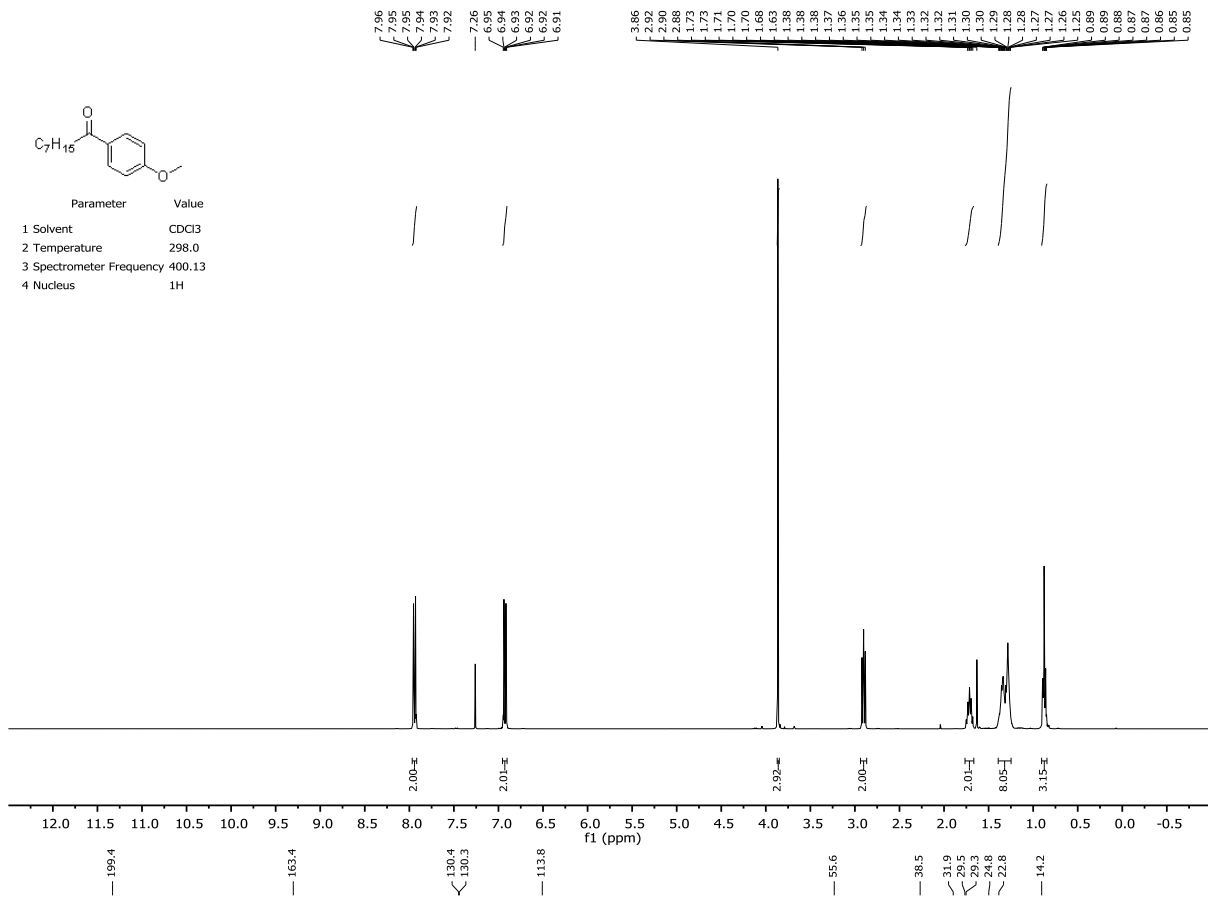
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	400.13
4 Nucleus	¹ H



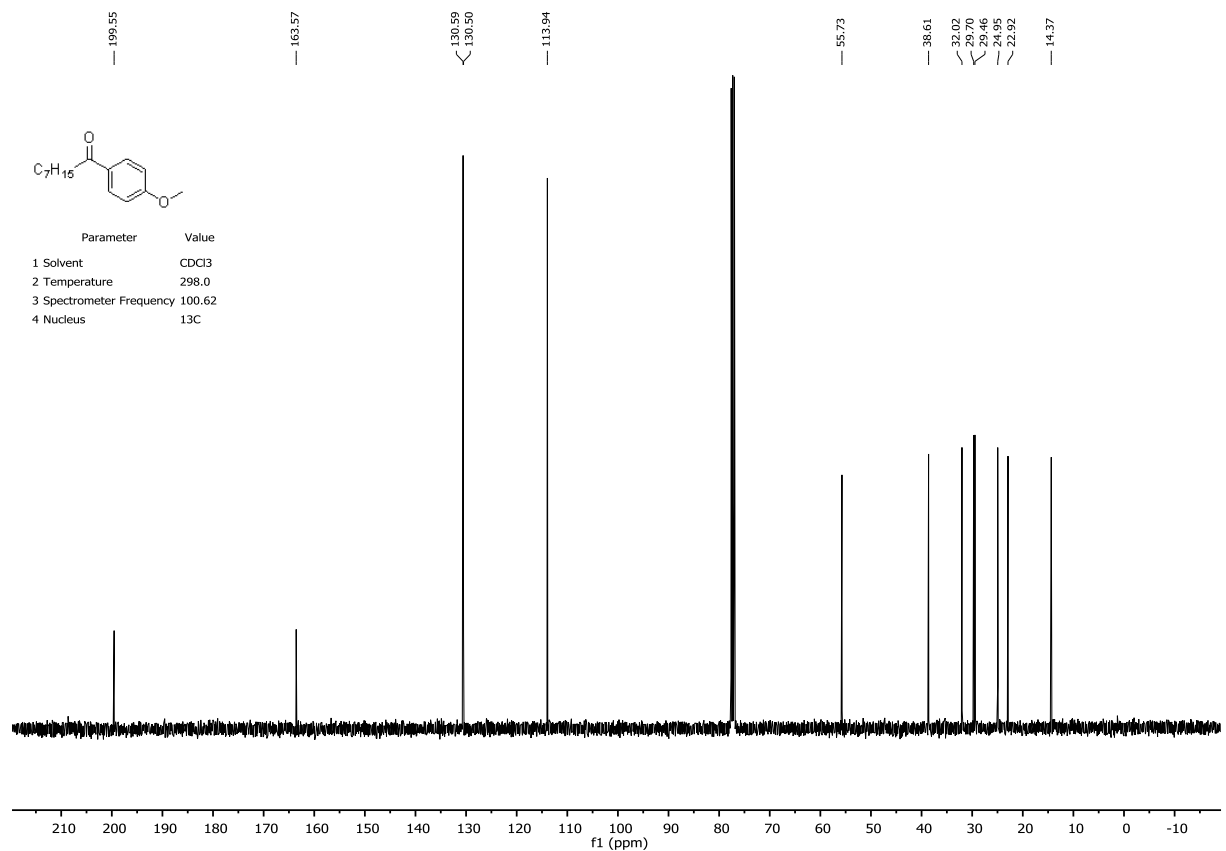
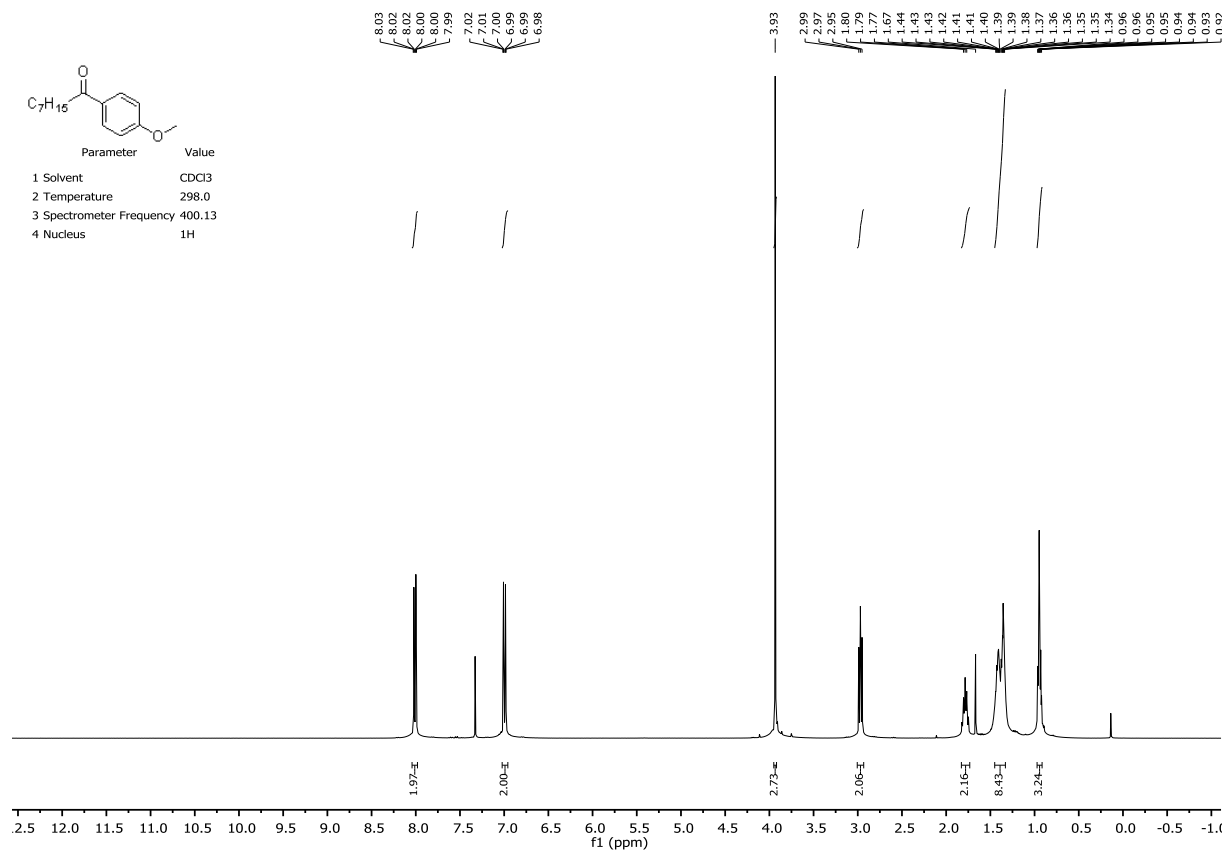
Parameter	Value
1 Solvent	CDCl3
2 Temperature	298.0
3 Spectrometer Frequency	100.62
4 Nucleus	¹³ C



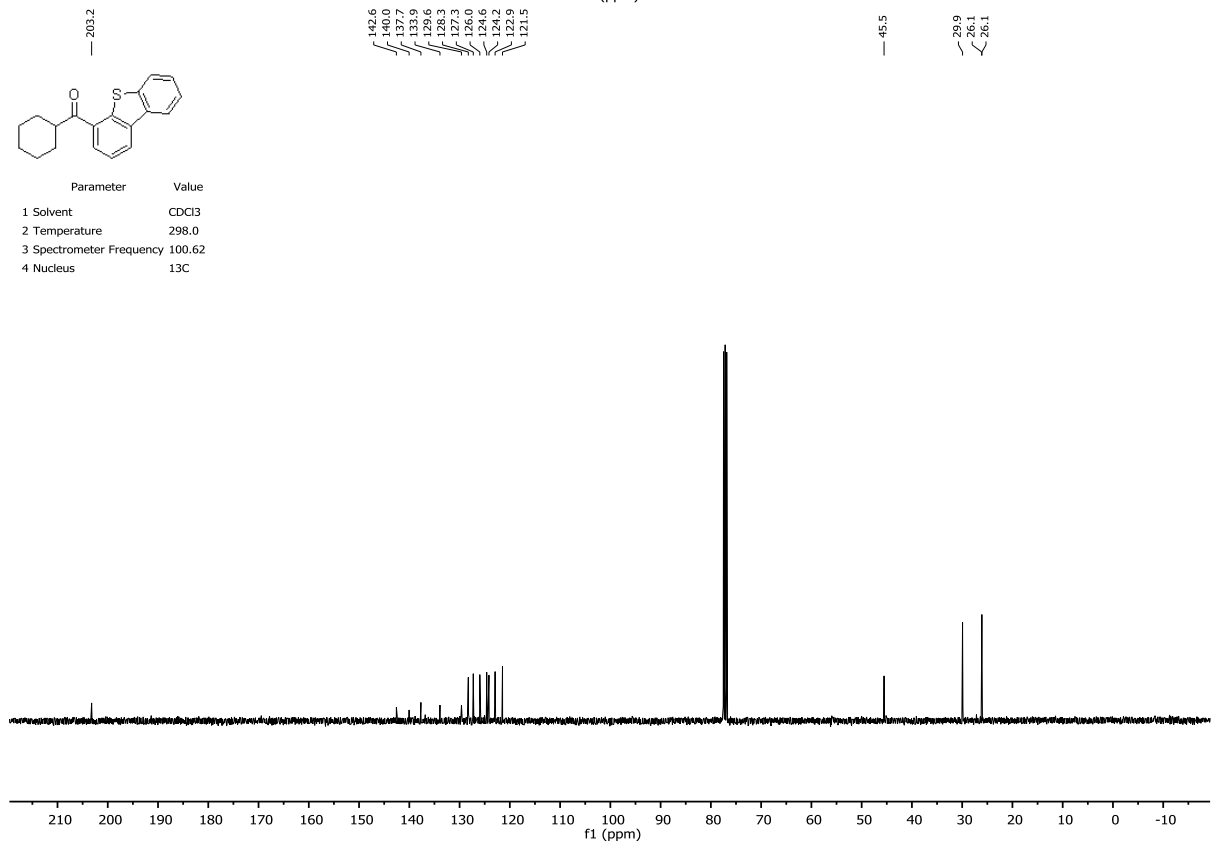
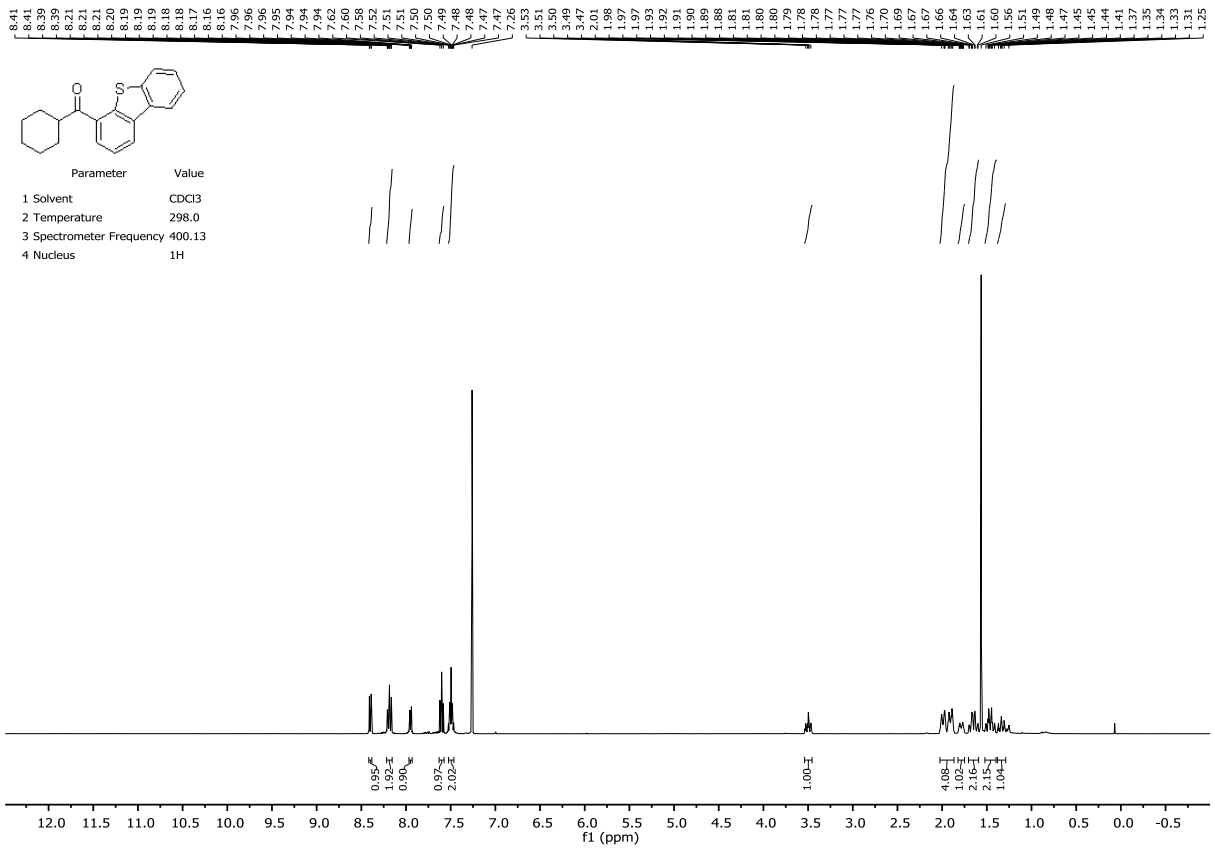
1-(2-Methoxyphenyl)octan-1-one (28) CAS: 153644-77-0



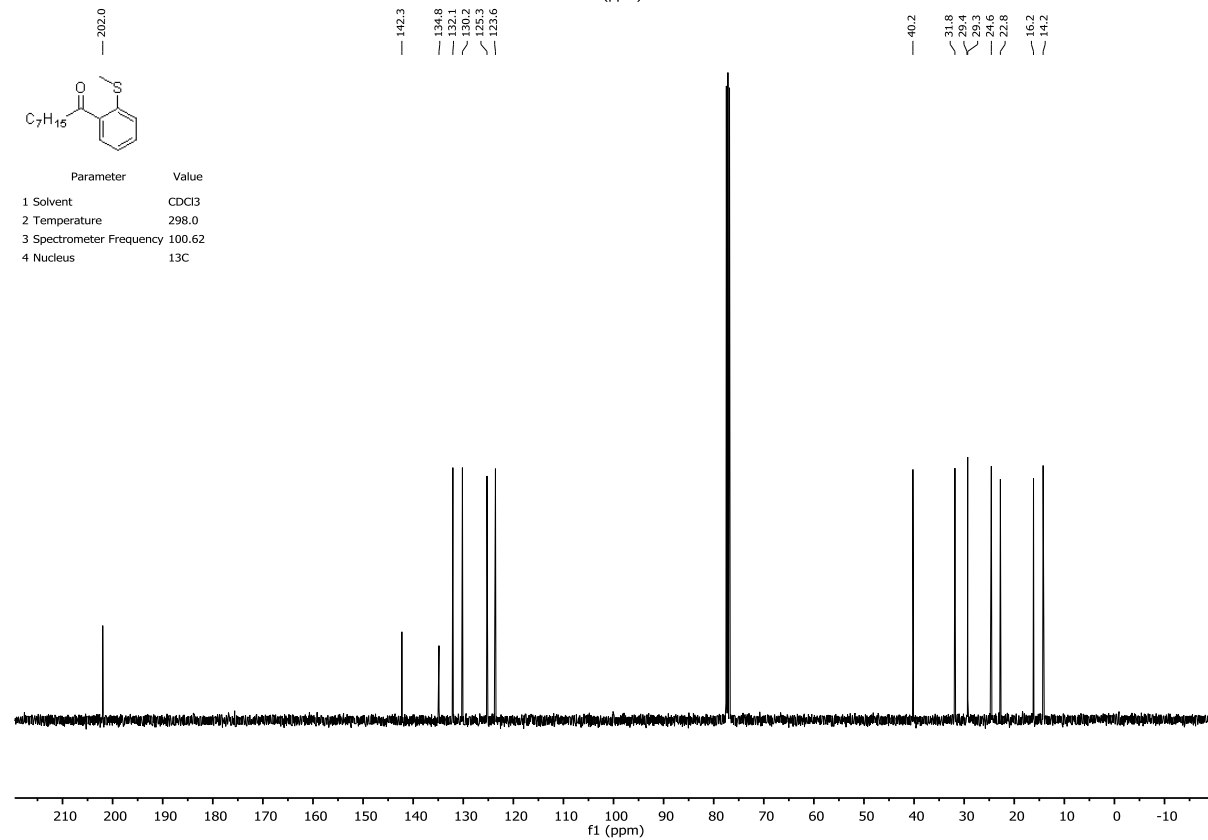
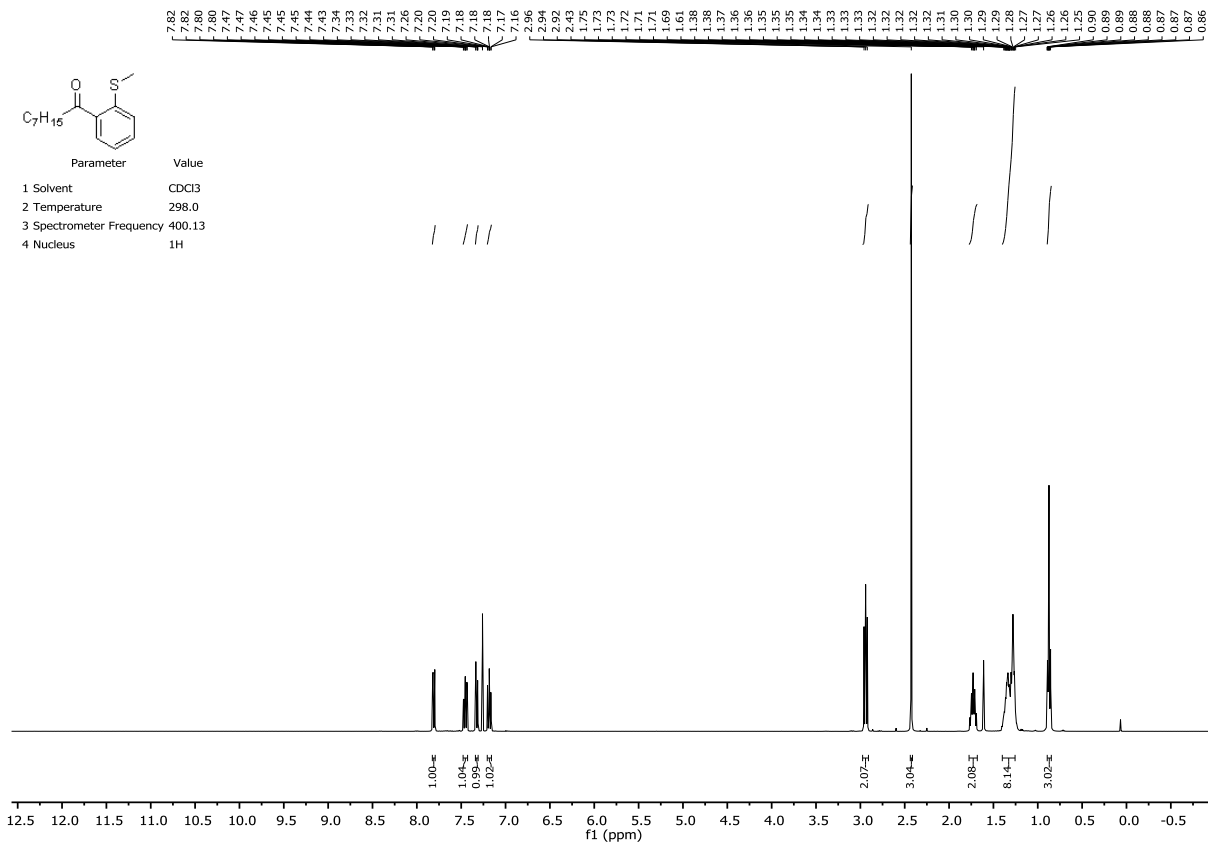
1-(4-Methoxyphenyl)octan-1-one¹⁷ (29) CAS: 62170-25-6



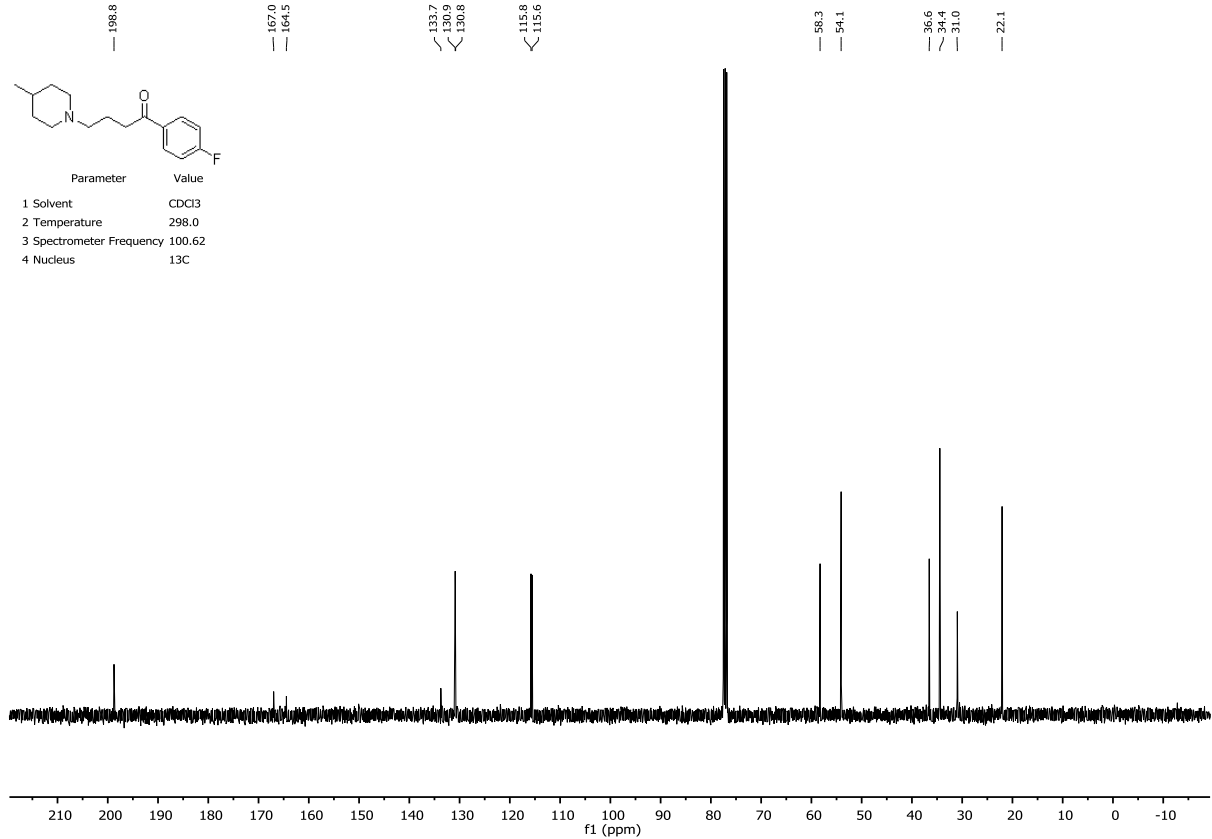
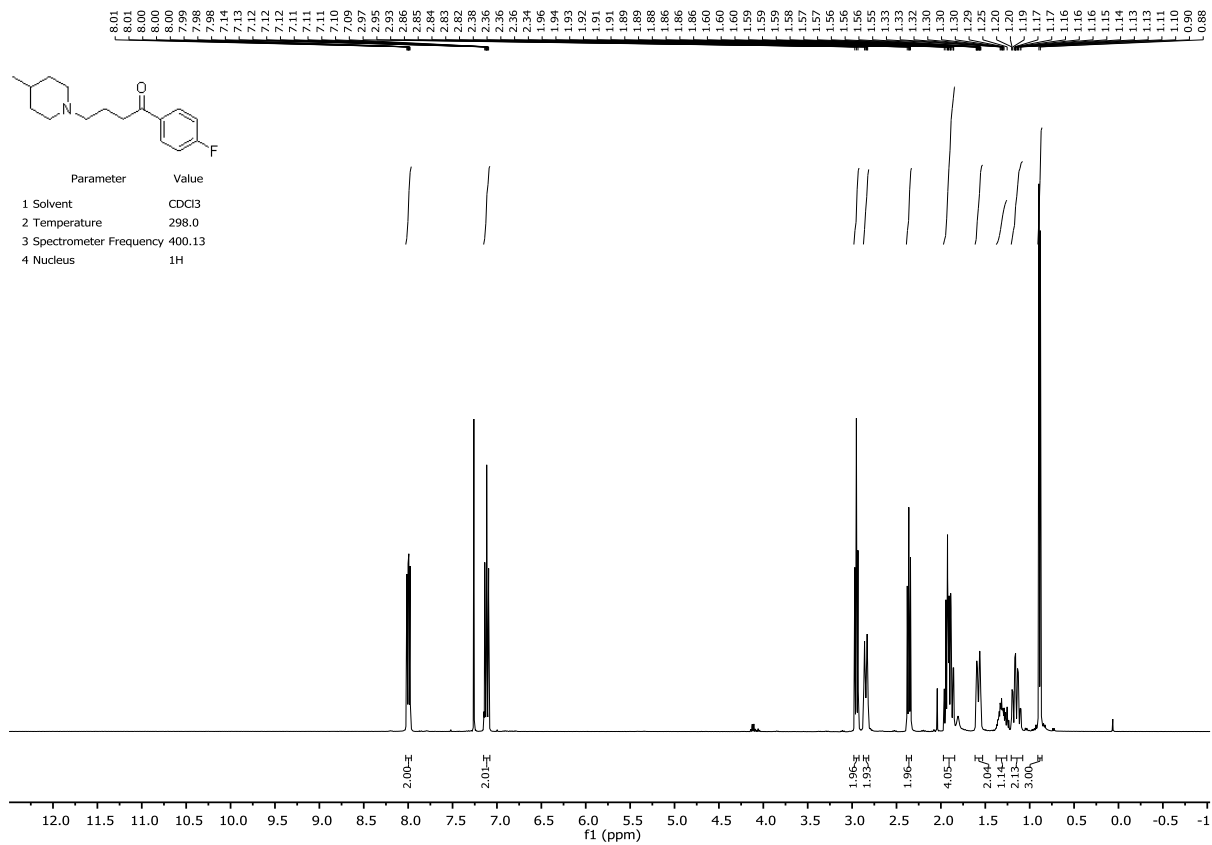
1-(4-Methoxyphenyl)octan-1-one¹⁷ (29) CAS: 62170-25-6 (CO-surrogate synthesis)



Cyclohexyl(dibenzo[b,d]thio-phen-4-yl)methanone (30) CAS: -



1-(2-(Methylthio)phenyl)octan-1-one (31) CAS: 1778855-66-5



1-(4-Fluorophenyl)-4-(4-methylpiperidin-1-yl)butan-1-one (Melperone) CAS: 3575-80-2