Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Photocatalytic Decarboxylative Alkylation of Silyl Enol Ether and Enamide with N-(Acyloxy)phthalimide using Ammonium Iodide

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1. General Information

A. Materials:

All reactions were carried out in oven-dried Schlenk tubes under argon atmosphere (purity≥99.999%) unless otherwise mentioned. Commercial reagents were purchased from Adamas-beta, TCI and Aldrich. Organic solutions were concentrated under reduced pressure on Buchi rotary evaporator. The LED lamps were purchased from Kessil (PR160-390 nm, 427 nm, 440 nm, 456 nm). The Photo Reaction Setup was purchased from Anhui kemi machinery technology Co., Ltd.

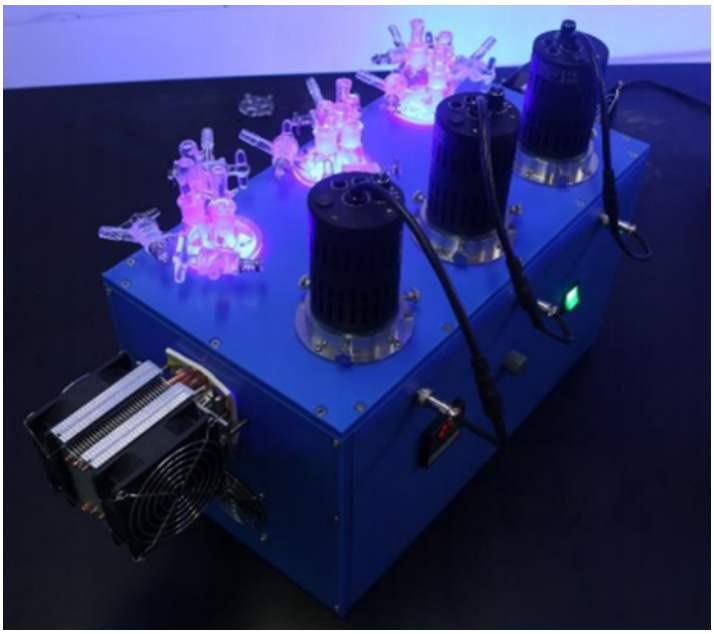


Figure S1. The Photo Reaction Setup and Blue LED lamps

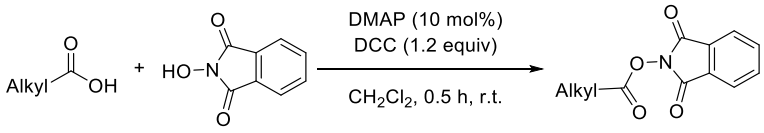
B. Analytical Methods:

^{1}H-NMR, ^{19}F-NMR and ^{13}C-NMR spectra were recorded on a Bruker Avance 400 spectrometer at ambient temperature. Data for ^{1}H-NMR are reported as follows: chemical shift (ppm, scale), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and/or multiplet resonances, br = broad), coupling constant (Hz), and integration. Data for ^{13}C- NMR are reported in terms of chemical shift (ppm, scale), multiplicity, and coupling constant (Hz). HRMS analysis was performed on Finnigan LCQ advantage Max Series MS System. ESI-mass data were acquired using a Thermo LTQ Orbitrap XL Instrument equipped with an ESI source and controlled

by Xcalibur software. UV-Vis spectrum was measured by UV-3600. Flash column chromatographic purification of products was accomplished using forced-flow chromatography on Silica Gel (200-300 mesh). X-ray crystallography was performed on a Nonius Kappa CCD diffractometer or a Bruker D8-QUEST PHOTON-100 diffractometer using CuKa radiation (lambda = 1.5418 Å) at the Cambridge University Chemistry X-Ray Laboratory.

2. Preparation of Substrates

2.1 General Procedure for preparation of redox-active ester



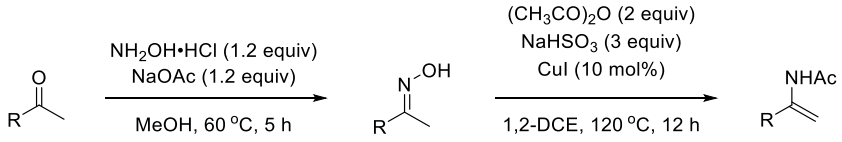
General Procedure 1: Redox-Active Ester.[1] The corresponding alkyl carboxylic acids (10 mmol, 1.0 equiv), *N*-hydroxyphthalimide (12 mmol, 1.2 equiv), and 4-dimethylaminopyridine (1 mmol, 10 mol %) were mixed in a flask equipped with a magnetic stirring bar, and then CH_2Cl_2 (30 mL) was added. A solution of N, N-dicyclohexylcarbodiimide (12 mmol, 1.2 equiv) in CH_2Cl_2 (10 mL) was added slowly at room temperature. The reaction mixture was stirred at room temperature for 0.5 h. After complete conversion of *N*-hydroxyphthalimide traced by TLC, the white precipitate was filtered off and the solution was concentrated on a rotary evaporator. The residue was purified by flash column chromatography to give corresponding redox active esters.

2.2 General Procedure for preparation of silyl enol ether

General Procedure 2: Silyl Enol Ether.[2] The NaI (1.4 equiv) was placed in a tube and dried under vacuum using a heat gun. After cooling to room temperature, the tube was filled with argon. Then, dry CH_3CN (1 M), ketone (1.0 equiv), and Et_3N (1.5

equiv) were successively added. The mixture was cooled with an ice/water bath, and TMSCl (1.3 equiv) was added at 0 °C. The cooling bath was removed, and the mixture was stirred for 12 h at room temperature. Then, volatile components were evaporated under vacuum. The solid residue was washed with petroleum ether (3 × 15 mL) [the petroleum ether layers were decanted and filtered through a cotton plug]. The combined filtrates were concentrated on a rotary evaporator, furnishing silyl enol ether which was used without purification.

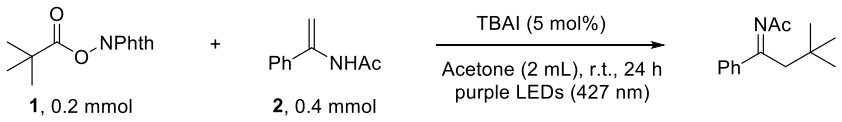
2.3 General Procedure for preparation of phenylvinyl acetamide



General Procedure 3: Phenylvinyl Acetamide.[3,4] To a solution of ketone (10 mmol, 1.0 equiv) in MeOH (30 mL) was added hydroxylamine hydrochloride (15 mmol, 1.5 equiv) and NaOAc (25 mmol, 2.5 equiv). After stirring at 60 °C in oil bath for 5 h, the mixture was concentrated in vacuo and the residue was extracted with EtOAc. The organic layer was separated, washed with brine, dried over Na_2SO_4 and concentrated to give the crude product. A mixture of ketoxime (10 mmol, 1.0 equiv), the anhydride (20 mmol, 2.0 equiv), $NaHSO_3$ (30 mmol, 3.0 equiv), and CuI (1 mmol, 0.1 equiv) was stirred in 1,2-DCE (100 mL) at 120 °C in oil bath under N_2 for 12 h. After completion of the reaction, the reaction mixture was cooled to room temperature, diluted with EtOAc (25 mL), and washed with 2 M NaOH (20 mL) and brine (20 mL). The organic layers were dried over Na_2SO_4 and evaporated in vacuo. The desired enamide was obtained after purification by flash chromatography on silica gel with PE/EA as the eluent.

3. Investigation of the Key Reaction Parameters

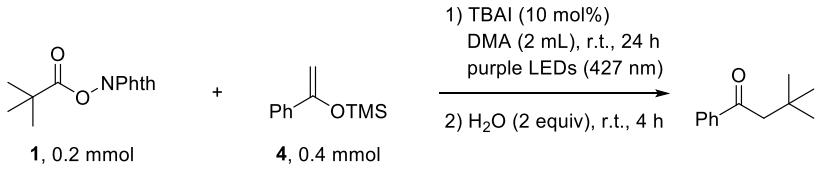
Table S1: Parameters affecting the alkylation of enamine



Entry	Variations from standard conditions	Yield (%)[a]
1	none	96
2	THF instead of Acetone	68
3	MeCN instead of Acetone	32
4	DCM instead of Acetone	27
5	DMA instead of Acetone	78
6	DMF instead of Acetone	56
7	DMSO instead of Acetone	15
8	1,4-Dioxane instead of Acetone	67
9	Toluene instead of Acetone	60
10	NMP instead of Acetone	64
11	**2** (0.3 mmol) instead of **2** (0.4 mmol)	86
12	TBAI (10 mol%) instead of TBAI (5 mol%)	89
13	TBAI (20 mol%) instead of TBAI (5 mol%)	81
14	Acetone (1.5 mL) instead of Acetone (2.0 mL)	95
15	Acetone (1.0 mL) instead of Acetone (2.0 mL)	96
16	Acetone (0.5 mL) instead of Acetone (2.0 mL)	94
17	12 h instead of 24 h	71
18	18 h instead of 24 h	80
19	390 nm instead of 427 nm	75
20	440 nm instead of 427 nm	64
21	456 nm instead of 427 nm	40
22	467 nm instead of 427 nm	30
23	no TBAI	trace
24	no light	0

Reaction condition: 1,3-dioxoisoindolin-2-yl pivalate (0.2 mmol), N-(1-phenylvinyl)acetamide (0.4 mmol), TBAI (0.01 mmol) in Acetone (2 mL), irradiation by purple LEDs (427 nm) at room temperature (25 ± 3 °C) for 24 h in an argon atmosphere. [a]Yield determined by GC using diphenylmethane as an internal standard.

Table S2: Parameters affecting the alkylation of silyl enol ethers



1, 0.2 mmol **4**, 0.4 mmol

Entry	Variations from standard conditions	Yield (%)[a]
1	none	83
2	DMF instead of DMA	20
3	Toluene instead of DMA	54
4	Acetone instead of DMA	trace
5	NMP instead of DMA	18
6	THF instead of DMA	58
7	DMSO instead of DMA	30
8	MeCN instead of DMA	35
9	1,4-Dioxane instead of DMA	38
10	DCM instead of DMA	35
11	**4** (0.3 mmol) instaed of **4** (0.4 mmol)	68
12	TBAI (5 mol%) instead of TBAI (10 mol%)	76
13	TBAI (20 mol%) instead of TBAI (10 mol%)	75
14	DMA (1.5 mL) instead of DMA (2.0 mL)	82
15	DMA (1.0 mL) instead of DMA (2.0 mL)	82
16	DMA (0.5 mL) instead of DMA (2.0 mL)	83
17	12 h instead of 24 h	60
18	18 h instead of 24 h	74
19	390 nm instead of 427 nm	46
20	440 nm instead of 427 nm	68
21	456 nm instead of 427 nm	60
22	467 nm instead of 427 nm	35
23	no TBAI	trace
24	no H_2O	75
25	no light	0
26	NaI instead of TBAI	75
27	TBAI (10%) + PPh_3 (10%) instead of TBAI (10%)	73
28	NaI (10%) + PPh_3 (10%) instead of TBAI (10%)	76

Reaction condition: 1,3-dioxoisoindolin-2-yl pivalate (0.2 mmol), 1-phenyl-1-trimethylsilyloxyethylene (0.4 mmol), TBAI (0.02 mmol) in DMA (2 mL), irradiation by purple LEDs (427 nm) at room temperature (25 ± 3 °C) for 24 h in an argon atmosphere. Then H_2O (2 equiv) was added, stirred at room temperature (25 ± 3 °C) for 4 h.

[a]Yield determined by GC using diphenylmethane as an internal standard.

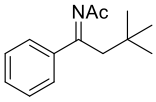
4. Experimental Procedures and Spectral Data

4.1 General Procedure

General Procedure A: Redox-active ester (1.0 equiv, 0.2 mmol) (if solid), Enamide (2.0 equiv, 0.4 mmol) (if solid), TBAI (5 mol%) were placed in a transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, Redox-active ester (1.0 equiv, 0.2 mmol) (if liquid), Enamide (2.0 equiv, 0.4 mmol) (if liquid), and anhydrous Acetone (2 mL) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred under irradiation with purple LEDs (427 nm, distance app. 3.0 cm from the bulb) at room temperature for 24 h. After reaction completed, the mixture was quenched with saturated NaCl solution and extracted with ethyl acetate (3 × 10 mL). The organic layers were combined and concentrated under vacuo. The product was purified by flash column chromatography on silica (petroleum ether/ethyl acetate = 100:1~20:1).

General Procedure B: Redox-active ester (1.0 equiv, 0.2 mmol) (if solid), Silyl Enol Ether (2.0 equiv, 0.4 mmol) (if solid), TBAI (10 mol%) were placed in a transparent Schlenk tube equipped with a stirring bar. The tube was evacuated and filled with argon (three times). To these solids, Redox-active ester (1.0 equiv, 0.2 mmol) (if liquid), Silyl Enol Ether (2.0 equiv, 0.4 mmol) (if liquid), and anhydrous DMA (2 mL) were added via a gastight syringe under argon atmosphere. The reaction mixture was stirred under irradiation with purple LEDs (427 nm, distance app. 3.0 cm from the bulb) at room temperature for 24 h. Then, H_2O (2 equiv) was added via a gastight syringe under air. The reaction mixture was stirred at room temperature for 4 h. After reaction completed, the mixture was quenched with saturated NaCl solution and extracted with ethyl acetate (3 × 10 mL). The organic layers were combined and concentrated under vacuo. The product was purified by flash column chromatography on silica (petroleum ether/ethyl acetate = 50:1~2:1).

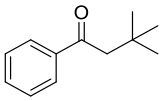
4.2 Spectral Data



N-(3,3-dimethyl-1-phenylbutylidene)acetamide (3): Following the general procedure A, obtained in 96% yield as colorless oil after silica gel chromatography. (41.6 mg, eluent: petroleum ether/triethylamine = 50/1). The compound data was in agreement with the literature (*ACS Catal.*, 2020, **10**, 1334–1343).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 – 7.52 (m, 2H), 7.52 – 7.31 (m, 3H), 2.71 (s, 2H), 2.14 (s, 3H), 0.92 (s, 9H).

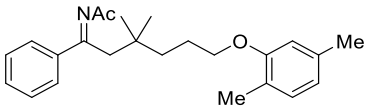
^{13}C NMR (126 MHz, $CDCl_3$) δ 184.4, 167.2, 139.1, 130.7, 128.5, 127.6, 47.9, 32.0, 30.5, 25.6.



3,3-dimethyl-1-phenylbutan-1-one (5): Following the general procedure B, obtained in 80% yield as colorless oil after silica gel chromatography. (28.0 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Chem. Commun.*, 2011, **47**, 2943–2945).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 – 7.91 (m, 1H), 7.59 – 7.50 (m, 2H), 7.49 – 7.40 (m, 2H), 2.86 (s, 2H), 1.07 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.5, 138.6, 132.7, 128.5, 128.2, 50.1, 31.4, 30.1.

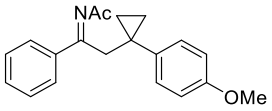


N-(6-(2,5-dimethylphenoxy)-3,3-dimethyl-1-phenylhexylidene)acetamide (6): Following the general procedure A, obtained in 87% yield as colorless oil after silica gel chromatography. (63.6 mg, eluent: petroleum ether/triethylamine = 50/1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.59 – 7.53 (m, 2H), 7.47 – 7.34 (m, 3H), 7.00 (d, J = 7.5 Hz, 1H), 6.65 (d, J = 7.5 Hz, 1H), 6.59 (s, 1H), 3.84 (t, J = 6.5 Hz, 2H), 2.74 (s, 2H), 2.30 (s, 3H), 2.15 (s, 3H), 2.10 (s, 3H), 1.80 – 1.71 (m, 2H), 1.49 – 1.38 (m, 2H), 0.92 (s, 6H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 184.4, 167.2, 156.9, 139.3, 136.5, 130.7, 130.3, 128.6, 127.5, 123.6, 120.7, 112.0, 68.2, 46.8, 39.5, 34.5, 27.8, 25.6, 24.4, 21.4, 15.8.

HRMS (ESI) Calcd for $C_{24}H_{32}O_2N^+$ $[M+H]^+$: 366.2428, found: 366.2425.



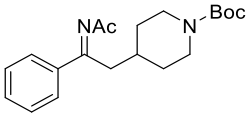
N-(2-(1-(4-methoxyphenyl)cyclopropyl)-1-phenylethylidene)acetamide (7):

Following the general procedure A, obtained in 76% yield as colorless oil after silica gel chromatography. (46.7 mg, eluent: petroleum ether/triethylamine = 20/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.52 (m, 2H), 7.45 – 7.39 (m, 1H), 7.36 (m, 2H), 7.18 – 7.02 (m, 2H), 6.75 (m, 2H), 3.75 (s, 3H), 3.03 (s, 2H), 1.74 (s, 3H), 0.99 – 0.55 (m, 4H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 185.1, 166.1, 158.3, 138.0, 136.2, 130.7, 130.0, 128.3, 127.9, 113.7, 55.3, 44.2, 24.8, 23.6 13.4.

HRMS (ESI) Calcd for $C_{20}H_{22}O_2N^+$ $[M+H]^+$: 308.1645, found: 308.1642.



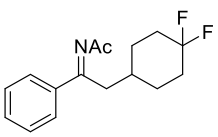
tert-butyl 4-(2-(acetylimino)-2-phenylethyl)piperidine-1-carboxylate (8):

Following the general procedure A, obtained in 45% yield as colorless oil after silica gel chromatography. (31.0 mg, eluent: petroleum ether/triethylamine = 10/1).

^{1}H NMR (400 MHz, C_6D_6) δ 7.36 – 7.27 (m, 2H), 6.86 – 6.79 (m, 3H), 2.14 (q, J = 7.1 Hz, 1H), 1.98 (s, 4H), 1.63 (s, 3H), 1.34 – 1.22 (m, 2H), 1.19 (s, 9H), 1.03 (d, J = 3.4 Hz, 2H), 0.70 (t, J = 7.1 Hz, 2H).

^{13}C NMR (101 MHz, C_6D_6) δ 182.6, 163.4, 153.3, 136.2, 129.9, 127.5, 126.8, 77.7, 45.5, 39.8, 33.6, 31.0, 27.3, 24.1.

HRMS (ESI) Calcd for $C_{20}H_{29}O_3N_2^+$ $[M+H]^+$: 345.2173, found: 345.2174.



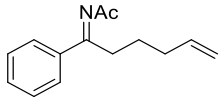
N-(2-(4,4-difluorocyclohexyl)-1-phenylethylidene)acetamide (9): Following the general procedure A, obtained in 50% yield as colorless oil after silica gel chromatography. (27.9 mg, eluent: petroleum ether/triethylamine = 50/1).

^{1}H NMR (400 MHz, C_6D_6) δ 7.56 (dd, J = 7.6, 2.0 Hz, 2H), 7.15 – 7.04 (m, 3H), 2.25 (d, J = 7.0 Hz, 2H), 1.90 (s, 3H), 1.85 – 1.73 (m, 2H), 1.49 – 1.41 (m, 1H), 1.40 – 1.34 (m, 2H), 1.12 (dt, J = 18.3, 10.0 Hz, 4H).

^{13}C NMR (101 MHz, C_6D_6) δ 182.8, 163.4, 136.1, 130.1, 127.6, 126.8, 122.1(dd, J = 255.5, 226.3 Hz), 39.1 (d, J = 2.5 Hz), 33.2 (d, J = 1.5 Hz), 32.2 (dd, J = 25.3, 22.9 Hz), 27.9 (d, J = 9.7 Hz), 24.1.

^{19}F NMR (376 MHz, C_6D_6) δ -91.53 (d, J = 236.2 Hz), -101.93 (d, J = 236.5 Hz).

HRMS (ESI) Calcd for $C_{16}H_{20}ONF_2^+$ $[M+H]^+$: 280.1508, found: 280.1515.

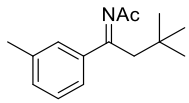


N-(1-phenylhex-5-en-1-ylidene)acetamide (10): Following the general procedure A, obtained in 56% yield as colorless oil after silica gel chromatography. (24.2 mg, eluent: petroleum ether/triethylamine = 50/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.80 – 7.69 (m, 2H), 7.53 – 7.37 (m, 3H), 5.77 (ddt, J = 17.0, 10.3, 6.7 Hz, 1H), 5.15 – 4.94 (m, 2H), 2.77 – 2.64 (m, 2H), 2.21 (s, 3H), 2.14 (td, J = 7.2, 1.2 Hz, 2H), 1.79 – 1.66 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 185.6, 166.2, 137.4, 136.3, 131.2, 128.6, 127.6, 115.7, 34.1, 33.5, 26.9, 25.5.

HRMS (ESI) Calcd for $C_{14}H_{18}ON^+$ $[M+H]^+$: 216.1283, found: 216.1289.

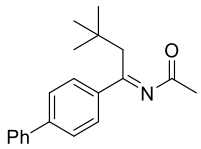


N-(3,3-dimethyl-1-(m-tolyl)butylidene)acetamide (11): Following the general procedure A, obtained in 95% yield as colorless oil after silica gel chromatography. (43.9 mg, eluent: petroleum ether/triethylamine = 50/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 (s, 1H), 7.27 (d, J = 7.7 Hz, 1H), 6.98 (t, J = 7.6 Hz, 1H), 6.91 (d, J = 7.6 Hz, 1H), 2.53 (s, 2H), 2.03 (s, 3H), 1.91 (s, 3H), 0.84 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.5, 167.5, 139.1, 138.3, 131.5, 128.3, 128.1, 124.8, 47.9, 32.1, 30.6, 25.7, 21.4.

HRMS (ESI) Calcd for $C_{15}H_{22}ON^+$ $[M+H]^+$: 232.1696, found: 232.1701.



(*E*)-N-(1-([1,1'-biphenyl]-4-yl)-3,3-dimethylbutylidene)acetamide (12): Following the general procedure A, obtained in 96% yield as colorless oil after silica gel chromatography. (56.2 mg, eluent: petroleum ether/triethylamine = 50/1). The compound data was in agreement with the literature (*ACS Catal.*, 2020, **10**, 1334−1343).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 – 7.67 (m, 2H), 7.62 (ddd, J = 7.8, 2.4, 1.1 Hz, 4H), 7.49 – 7.43 (m, 2H), 7.40 – 7.35 (m, 1H), 2.75 (s, 2H), 2.18 (s, 3H), 0.95 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 184.5, 166.8, 143.6, 139.9, 137.8, 128.9, 128.3, 127.9, 127.2, 127.1, 47.6, 32.2, 30.7, 25.7.

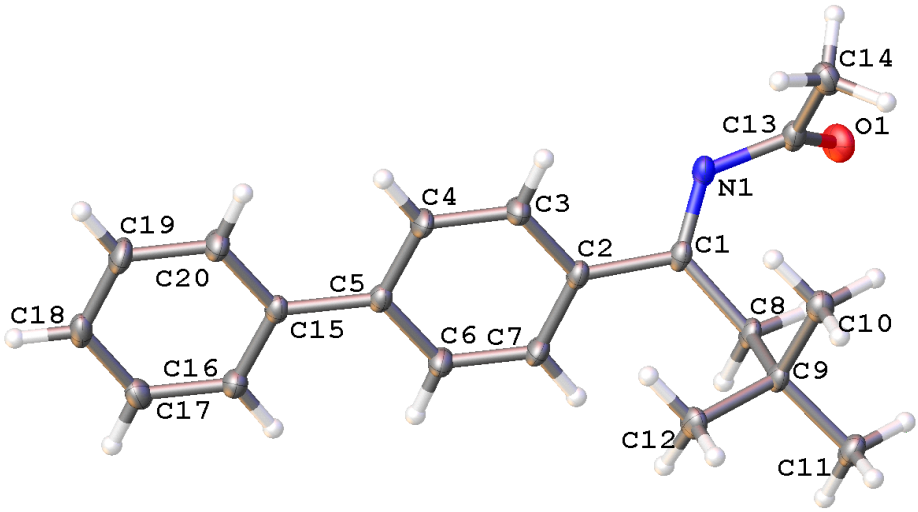
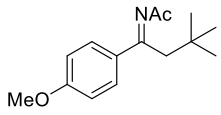


Table S3: Crystal data and structure refinement for (E)-N-(1-([1,1'-biphenyl]-4-yl)-3,3-dimethylbutylidene)acetamide.

Empirical formula	$C_{20}H_{23}NO$
Formula weight	293.39
Temperature/K	173.0
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	5.5518(2)
b/Å	8.4841(3)
c/Å	34.8351(12)
α/°	90
β/°	90.852(2)
γ/°	90
Volume/Å^3	1640.62(10)
Z	4
ρ_{calc}g/cm^3	1.188
μ/mm^{-1}	0.359
F(000)	632.0
Crystal size/mm^3	0.05 × 0.01 × 0.01
Radiation	GaKα (λ = 1.34139)
2Θ range for data collection/°	8.834 to 109.842
Index ranges	$-6 \leq h \leq 6$, $-10 \leq k \leq 10$, $-42 \leq l \leq 42$
Reflections collected	15306
Independent reflections	3089 [R_{int} = 0.0805, R_{sigma} = 0.0626]
Data/restraints/parameters	3089/0/203
Goodness-of-fit on F^2	1.078
Final R indexes [I>=2σ (I)]	R_1 = 0.0595, wR_2 = 0.1582
Final R indexes [all data]	R_1 = 0.0801, wR_2 = 0.1747
Largest diff. peak/hole / e Å^{-3}	0.28/-0.26

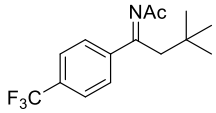


N-(1-(4-methoxyphenyl)-3,3-dimethylbutylidene)acetamide (13): Following the general procedure A, obtained in 90% yield as colorless oil after silica gel chromatography. (44.5 mg, eluent: petroleum ether/triethylamine = 50/1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.69 – 7.52 (m, 2H), 6.96 – 6.82 (m, 2H), 3.84 (s, 3H), 2.70 (s, 2H), 2.16 (s, 3H), 0.92 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 184.5, 166.2, 161.8, 130.6, 129.7, 113.7, 55.4, 47.1, 30.6, 30.2, 25.7.

HRMS (ESI) Calcd for $C_{15}H_{22}O_2N^+$ $[M+H]^+$: 248.1645, found: 248.1644.



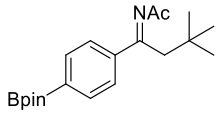
N-(3,3-dimethyl-1-(4-(trifluoromethyl)phenyl)butylidene)acetamide (14): Following the general procedure A, obtained in 60% yield as colorless oil after silica gel chromatography. (34.2 mg, eluent: petroleum ether/triethylamine = 50/1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.74 (d, J = 8.2 Hz, 2H), 7.67 (d, J = 8.2 Hz, 2H), 2.74 (s, 2H), 2.19 (s, 3H), 0.92 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 184.2, 166.1, 142.4, 132.5 (q, J = 32.7 Hz), 128.0, 128.8 – 122.1 (m), 125.5 (q, J = 3.6 Hz), 47.6, 32.2, 30.6, 25.6.

^{19}F NMR (376 MHz, $CDCl_3$) δ -62.88 (s).

HRMS (ESI) Calcd for $C_{15}H_{19}ONF_3^+$ $[M+H]^+$: 286.1413, found: 286.1417.

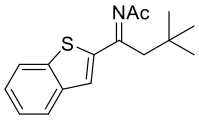


N-(3,3-dimethyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)butylidene)acetamide (15): Following the general procedure A, obtained in 96% yield as colorless oil after silica gel chromatography. (65.8 mg, eluent: petroleum ether/triethylamine = 3/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.0 Hz, 2H), 7.58 (d, J = 8.0 Hz, 2H), 2.71 (s, 2H), 2.14 (s, 3H), 1.35 (s, 12H), 0.90 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 184.5, 167.4, 141.5, 134.8, 126.8, 123.6, 84.1, 48.0, 32.1, 30.6, 25.6, 24.9.

HRMS (ESI) Calcd for $C_{20}H_{31}O_3NB^+$ $[M+H]^+$: 344.2396, found: 344.2394.

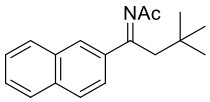


N-(1-(benzo[b]thiophen-2-yl)-3,3-dimethylbutylidene)acetamide (16): Following the general procedure A, obtained in 56% yield as colorless oil after silica gel chromatography. (30.6 mg, eluent: petroleum ether/triethylamine = 50/3).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, J = 7.9 Hz, 1H), 7.18 (d, J = 8.0 Hz, 1H), 7.13 (s, 1H), 6.91 – 6.86 (m, 1H), 6.85 – 6.79 (m, 1H), 2.48 (s, 2H), 1.82 (s, 3H), 0.66 (s, 9H).

^{13}C NMR (101 MHz, CDCl3) δ 183.7, 161.7, 144.2, 141.6, 139.3, 134.7, 128.5, 126.7, 125.1, 122.5, 45.7, 32.5, 30.8 27.0.

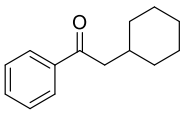
HRMS (ESI) Calcd for $C_{16}H_{20}ONS^+$ $[M+H]^+$: 274.1260, found: 274.1262.



N-(3,3-dimethyl-1-(naphthalen-2-yl)butylidene)acetamide (17): Following the general procedure A, obtained in 93% yield as colorless oil after silica gel chromatography. (49.6 mg, eluent: petroleum ether/triethylamine = 50/1). The compound data was in agreement with the literature (*ACS Catal.*, 2020, **10**, 1334−1343).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (d, J = 1.2 Hz, 1H), 7.87 – 7.80 (m, 1H), 7.80 – 7.76 (m, 2H), 7.66 (dd, J = 8.6, 1.8 Hz, 1H), 7.52 – 7.41 (m, 2H), 2.77 (s, 2H), 2.11 (s, 3H), 0.87 (s, 9H).

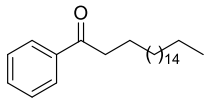
^{13}C NMR (126 MHz, $CDCl_3$) δ 184.5, 167.2, 136.5, 134.3, 132.6, 128.9, 128.4, 128.2, 127.7, 127.6, 126.8, 124.6, 47.7, 32.2, 30.7, 25.7.



2-cyclohexyl-1-phenylethan-1-one (18): Following the general procedure B, obtained in 79% yield as colorless oil after silica gel chromatography. (31.9 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Chem. Commun.*, 2015, **51**, 7546−7549).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 – 7.87 (m, 2H), 7.60 – 7.50 (m, 1H), 7.50 – 7.41 (m, 2H), 2.82 (d, J = 6.8 Hz, 2H), 1.97 (ddd, J = 14.8, 7.7, 3.9 Hz, 1H), 1.86 – 1.59 (m, 5H), 1.28 (ddd, J = 12.8, 8.2, 3.1 Hz, 2H), 1.23 – 1.09 (m, 1H), 1.03 (td, J = 12.1, 2.7 Hz, 2H).

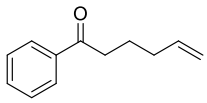
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 137.5, 132.8, 128.5, 128.2, 46.2, 34.6, 33.5, 26.3, 26.2.



1-phenylhexan-1-one (19): Following the general procedure B, obtained in 68% yield as white solid after silica gel chromatography. (23.8 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Org. Lett.*, 2018, **20**, 349–352).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.91 (m, 2H), 7.61 – 7.51 (m, 1H), 7.45 (dd, *J* = 10.4, 4.7 Hz, 2H), 3.17 – 2.83 (m, 2H), 1.73 (dt, *J* = 14.8, 7.4 Hz, 2H), 1.26 (s, 30H), 0.88 (t, *J* = 6.8 Hz, 3H).

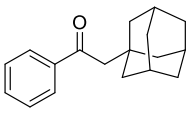
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.6, 137.1, 132.8, 128.5, 128.0, 38.6, 31.9, 29.7, 29.5, 29.4, 24.4, 22.7, 14.1.



1-phenylhex-5-en-1-one (20): Following the general procedure B, obtained in 55% yield as colorless liquid after silica gel chromatography. (19.2 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*J. Am. Chem. Soc.*, 2013, **135**, 10022−10025).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.46 (t, *J* = 7.6 Hz, 2H), 5.92 – 5.71 (m, 1H), 5.11 – 4.94 (m, 2H), 2.98 (t, *J* = 7.4 Hz, 2H), 2.17 (dd, *J* = 14.2, 7.1 Hz, 2H), 1.92 – 1.79 (m, 2H).

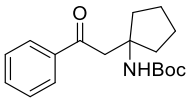
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.3, 138.1, 137.1, 132.9, 128.6, 128.0, 115.3, 37.7, 33.2, 23.3.



2-((3r,5r,7r)-adamantan-1-yl)-1-phenylethan-1-one (21): Following the general procedure B, obtained in 59% yield as colorless liquid after silica gel chromatography. (30.0 mg, eluent: petroleum ether/ethyl acetate = 25/1). The compound data was in agreement with the literature (*Angew. Chem. Int. Ed.*, 2016, **55**, 9969–9973).

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.87 (m, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 2.72 (s, 2H), 1.94 (s, 3H), 1.75 – 1.54 (m, 12H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 200.4, 138.9, 132.7, 128.5, 128.4, 51.2, 43.0, 36.8, 33.9, 28.7.

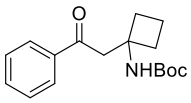


***tert*-butyl (1-(2-oxo-2-phenylethyl)cyclopentyl)carbamate (22):** Following the general procedure B, obtained in 57% yield as white solid after silica gel chromatography. (34.6 mg, eluent: petroleum ether/ethyl acetate = 10/1).

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 – 7.90 (m, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.44 (m, *J* = 7.7 Hz, 2H), 4.83 (s, 1H), 3.51 (s, 2H), 1.71 (m, *J* = 22.2, 11.3, 7.3 Hz, 8H), 1.35 (s, 9H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 199.7, 155.0, 137.9, 132.9, 128.5, 128.2, 78.9, 61.8, 44.5, 38.6, 28.4, 23.3.

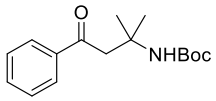
HRMS (ESI) Calcd for $C_{18}H_{25}O_3NNa^+$ $[M+Na]^+$: 326.1727 found: 326.1730.



***tert*-butyl (1-(2-oxo-2-phenylethyl)cyclobutyl)carbamate (23):** Following the general procedure B, obtained in 80% yield as white solid after silica gel chromatography. (46.3 mg, eluent: petroleum ether/ethyl acetate = 10/1). The compound data was in agreement with the literature (*Science*, 2019, **363**, 1429–1434).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.6 Hz, 2H), 7.56 (t, *J* = 7.3 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 5.22 (s, 1H), 3.56 (s, 2H), 2.43 – 2.14 (m, 4H), 1.98 (dt, *J* = 9.4, 8.0 Hz, 1H), 1.86 (dt, *J* = 11.3, 8.6 Hz, 1H), 1.38 (s, 9H).

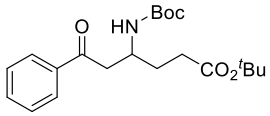
^{13}C NMR (101 MHz, $CDCl_3$) δ 199.2, 154.5, 137.5, 133.1, 128.6, 128.1, 79.1, 54.7, 44.5, 33.4, 28.4, 15.5.



***tert*-butyl (2-methyl-4-oxo-4-phenylbutan-2-yl)carbamate (24):** Following the general procedure B, obtained in 74% yield as white solid after silica gel chromatography. (41.1 mg, eluent: petroleum ether/ethyl acetate = 10/1). The compound data was in agreement with the literature (*Science*, 2019, **363**, 1429–1434).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (d, *J* = 7.3 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.6 Hz, 2H), 5.01 (s, 1H), 3.38 (s, 2H), 1.44 (s, 6H), 1.39 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.6, 154.7, 137.9, 133.0, 128.5, 128.3, 78.9, 51.8, 46.2, 28.4, 27.9.

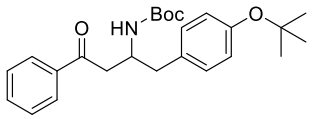


***tert*-butyl 4-((tert-butoxycarbonyl)amino)-6-oxo-6-phenylhexanoate (25):** Following the general procedure B, obtained in 59% yield as white solid after silica gel chromatography. (44.5 mg, eluent: petroleum ether/ethyl acetate = 5/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.95 (d, J = 7.6 Hz, 1H), 7.57 (t, J = 7.3 Hz, 2H), 7.46 (t, J = 7.7 Hz, 2H), 5.17 (d, J = 7.7 Hz, 1H), 4.05 (td, J = 8.7, 4.9 Hz, 1H), 3.33 (d, J = 16.4 Hz, 1H), 3.12 (dd, J = 16.7, 6.2 Hz, 1H), 2.33 (t, J = 7.3 Hz, 2H), 2.05 – 1.75 (m, 2H), 1.44 (s, 9H), 1.41 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 198.8, 172.8, 155.5, 136.9, 133.3, 128.7, 128.1, 80.5, 79.2, 47.7, 43.1, 32.6, 29.2, 28.4, 28.1.

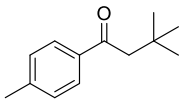
HRMS (ESI) Calcd for $C_{21}H_{31}O_5NNa^+$ $[M+Na]^+$: 400.2095, found: 400.2099.



***tert*-butyl (1-(4-(tert-butoxy)phenyl)-4-oxo-4-phenylbutan-2-yl)carbamate (26):** Following the general procedure B, obtained in 82% yield as white solid after silica gel chromatography. (67.5 mg, eluent: petroleum ether/ethyl acetate = 5/1). The compound data was in agreement with the literature (*Science*, 2019, **363**, 1429–1434).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (m, *J* = 5.2, 3.3 Hz, 2H), 7.61 – 7.51 (m, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 7.14 – 7.00 (m, 2H), 6.90 (t, *J* = 5.5 Hz, 2H), 5.24 (s, 1H), 4.43 – 4.01 (m, 1H), 3.27 – 2.72 (m, 4H), 1.39 (s, 9H), 1.32 (s, 9H).

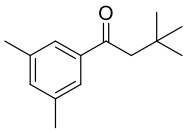
^{13}C NMR (101 MHz, $CDCl_3$) δ 199.4, 155.3, 153.9, 136.9, 133.3, 133.2, 129.7, 128.6, 128.1, 124.3, 79.2, 78.3, 49.3, 40.9, 39.4, 28.8, 28.4.



3,3-dimethyl-1-(p-tolyl)butan-1-one (27): Following the general procedure B, obtained in 83% yield as colorless oil after silica gel chromatography. (31.6 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Angew. Chem. Int. Ed.*, 2015, **54**, 7929–7933).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 8.0 Hz, 2H), 2.83 (s, 2H), 2.40 (s, 3H), 1.06 (s, 9H).

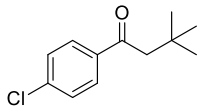
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.2, 143.4, 136.1, 129.2, 128.4, 49.9, 31.4, 30.1, 21.6.



1-(3,5-dimethylphenyl)-3,3-dimethylbutan-1-one (28): Following the general procedure B, obtained in 84% yield as colorless oil after silica gel chromatography. (34.3 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*ACS Catal.*, 2020, **10**, 1334–1343).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.54 (s, 2H), 7.17 (s, 1H), 2.83 (s, 2H), 2.36 (s, 6H), 1.06 (s, 9H).

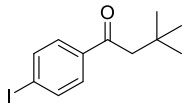
^{13}C NMR (101 MHz, $CDCl_3$) δ 200.9, 138.8, 138.0, 134.3, 126.0, 50.2, 31.4, 30.1, 21.3.



1-(4-chlorophenyl)-3,3-dimethylbutan-1-one (29): Following the general procedure B, obtained in 88% yield as colorless oil after silica gel chromatography. (37.1 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Org. Lett.*, 2014, **16**, 3064–3067).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 – 7.82 (m, 2H), 7.46 – 7.33 (m, 2H), 2.83 (s, 2H), 1.06 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.1, 139.2, 136.9, 129.7, 128.8, 50.0, 31.5, 30.0.

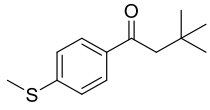


1-(4-iodophenyl)-3,3-dimethylbutan-1-one (30): Following the general procedure B, obtained in 81% yield as colorless oil after silica gel chromatography. (49.0 mg, eluent: petroleum ether/ethyl acetate = 50/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 – 7.76 (m, 2H), 7.68 – 7.61 (m, 2H), 2.81 (s, 2H), 1.05 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.6, 137.8, 129.7, 100.7, 49.9, 31.5, 30.1.

HRMS (ESI) Calcd for $C_{12}H_{16}OI^+$ $[M+H]^+$: 303.0240, found: 303.0245.

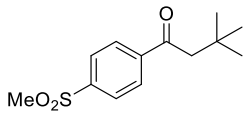


3,3-dimethyl-1-(4-(methylthio)phenyl)butan-1-one (31): Following the general procedure B, obtained in 85% yield as colorless oil after silica gel chromatography. (37.8 mg, eluent: petroleum ether/ethyl acetate = 50/1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.40 (m, 2H), 7.04 – 6.99 (m, 2H), 2.58 (s, 2H), 2.28 (s, 3H), 0.82 (s, 9H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 199.4, 145.4, 134.9, 128.7, 124.9, 49.8, 31.5, 30.1, 14.8.

HRMS (ESI) Calcd for $C_{13}H_{19}OS^+$ $[M+H]^+$: 223.1151, found: 223.1155.



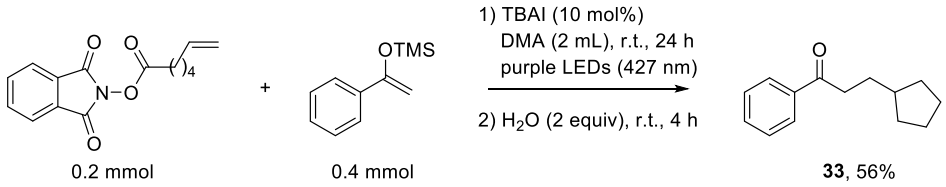
3,3-dimethyl-1-(4-(methylsulfonyl)phenyl)butan-1-one (32): Following the general procedure B, obtained in 83% yield as white solid after silica gel chromatography. (42.2 mg, eluent: petroleum ether/ethyl acetate = 5/1). The compound data was in agreement with the literature (*ACS Catal.*, 2020, **10**, 1334−1343).

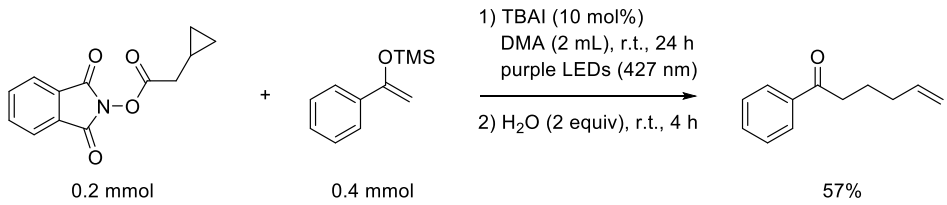
^{1}H NMR (400 MHz, $CDCl_3$) δ 8.11 (m, 2H), 8.04 (m, 2H), 3.09 (s, 3H), 2.91 (s, 2H), 1.08 (s, 9H).

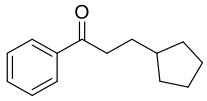
^{13}C NMR (101 MHz, $CDCl_3$) δ 199.1, 143.8, 142.5, 129.0, 127.7, 50.6, 44.3, 31.6, 29.9.

5. Experimental Studies on Mechanism

5.1 Radical clock experiments





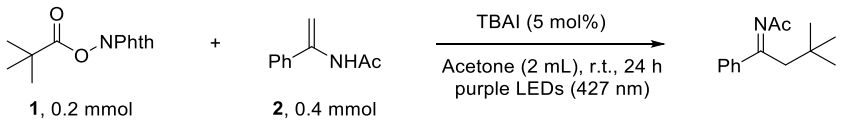


3-cyclopentyl-1-phenylpropan-1-one (33): Following the general procedure A, obtained in 56% yield as white solid after silica gel chromatography. (22.6 mg, eluent: petroleum ether/ethyl acetate = 50/1). The compound data was in agreement with the literature (*Science*, 2019, **363**, 1429–1434).

^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 – 7.77 (m, 2H), 7.52 – 7.44 (m, 1H), 7.51-7.36 (m, 2H), 3.00 – 2.83 (m, 2H), 1.82 – 1.62 (m, 5H), 1.61 – 1.36 (m, 4H), 1.21 – 1.07 (m, 2H).

^{13}C NMR (101 MHz, $CDCl_3$) δ 200.7, 137.1, 132.8, 128.5, 128.1, 39.8, 37.9, 32.6, 30.7, 25.2.

5.2 UV-vis absorption spectroscopic measurements



Stock solutions of **1**, **2**, TBAI were prepared with the same concentration used in the reaction. The solutions were prepared in the presence of air using Acetone as solvent.

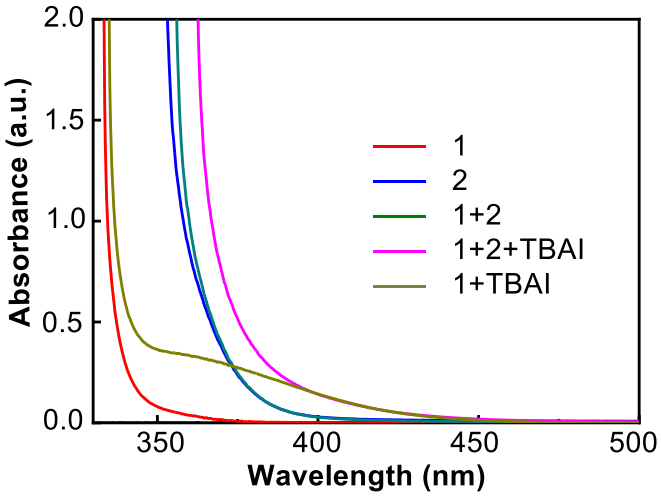
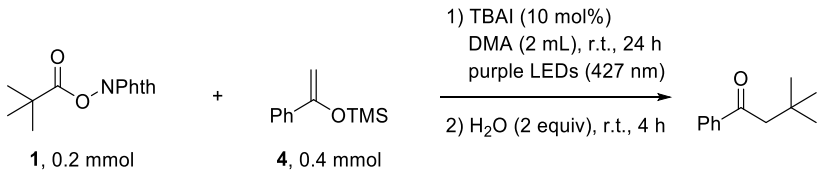


Figure S1: UV/vis absorption spectra of the combination between different starting materials recorded in Acetone as solvent.



Stock solutions of **1**, **4**, TBAI were prepared with the same concentration used in the reaction. The solutions were prepared in the presence of air using DMA as solvent.

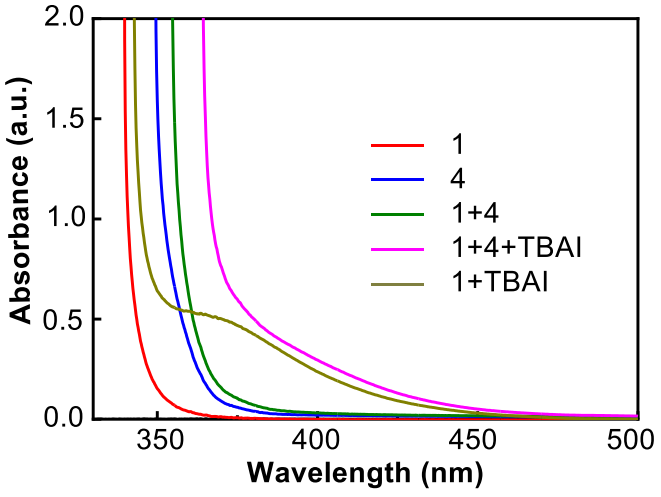


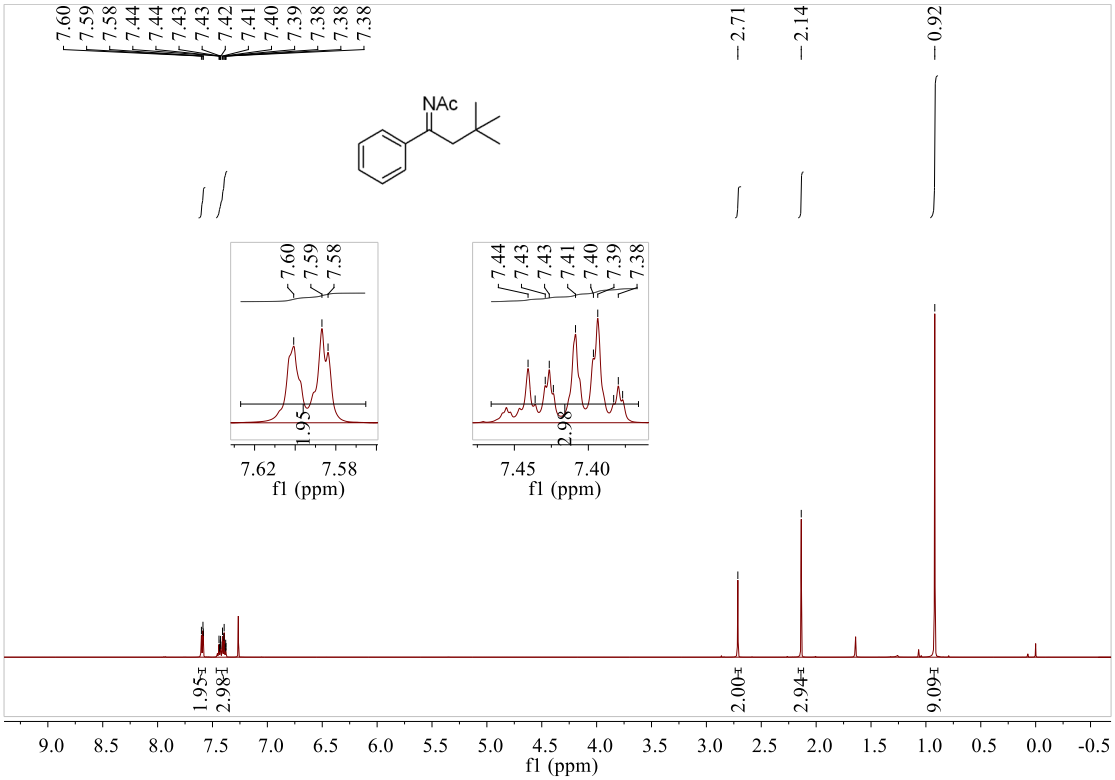
Figure S2: UV/vis absorption spectra of the combination between different starting materials recorded in DMA as solvent.

6. References

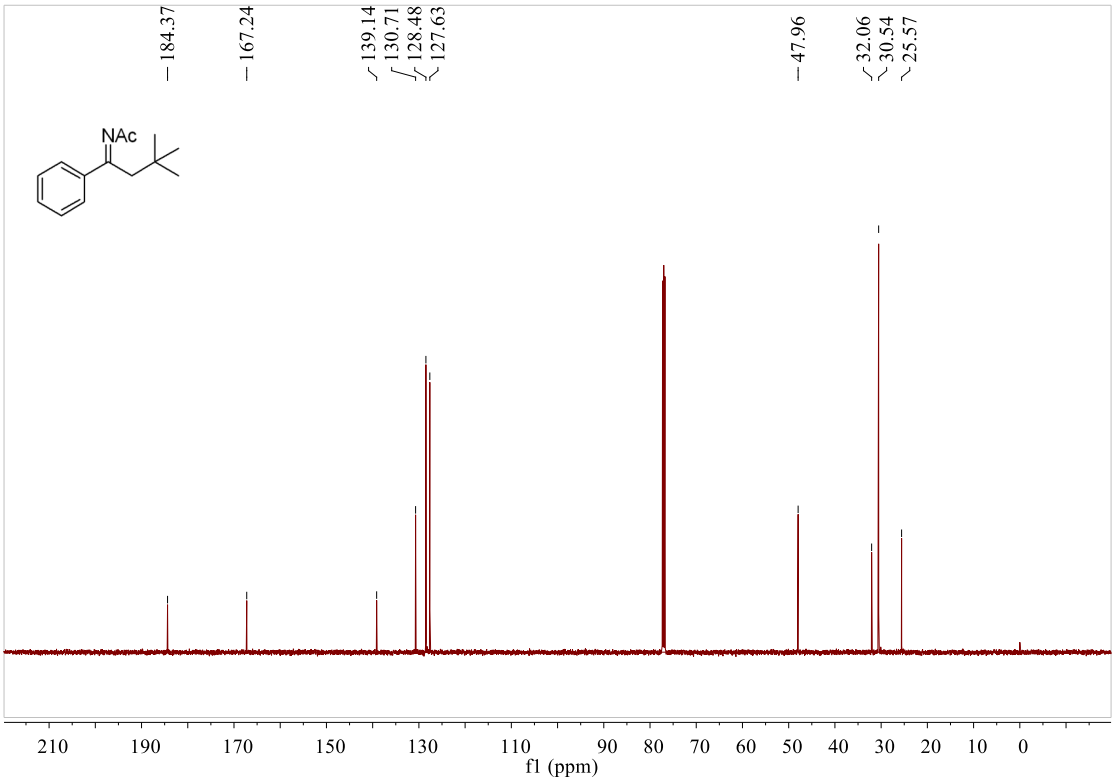
1. J. Cornella, J. T. Edwards, T. Qin, S. Kawamura, J. Wang, C.-M. Pan, R. Gianatassio, M. Schmidt, M. D. Eastgate and P. S. Baran, Practical Ni-Catalyzed Aryl–Alkyl Cross-Coupling of Secondary Redox-Active Esters, *J. Am. Chem. Soc.*, 2016, **138**, 2174–2177.
2. O. V. Fedorov, M. D. Kosobokov, V. V. Levin, M. I. Struchkova and A. D. Dilman, Halogenative Difluorohomologation of Ketones, *J. Org. Chem.*, 2015, **80**, 5870–5876.
3. C. Feng, D. Feng and T.-P. Loh, Rhodium(III)-catalyzed olefinic C–H alkynylation of enamides at room temperature, *Chem. Commun.*, 2014, **50**, 9865–9868.
4. R.-H. Liu, Z.-Y. Shen, C. Wang, T.-P Loh and X.-H. Hu, Selective Dehydrogenative Acylation of Enamides with Aldehydes Leading to Valuable β-Ketoenamides, *Org. Lett.*, 2020, **22**, 944–949.

7. ^{1}H NMR and ^{13}C NMR spectra

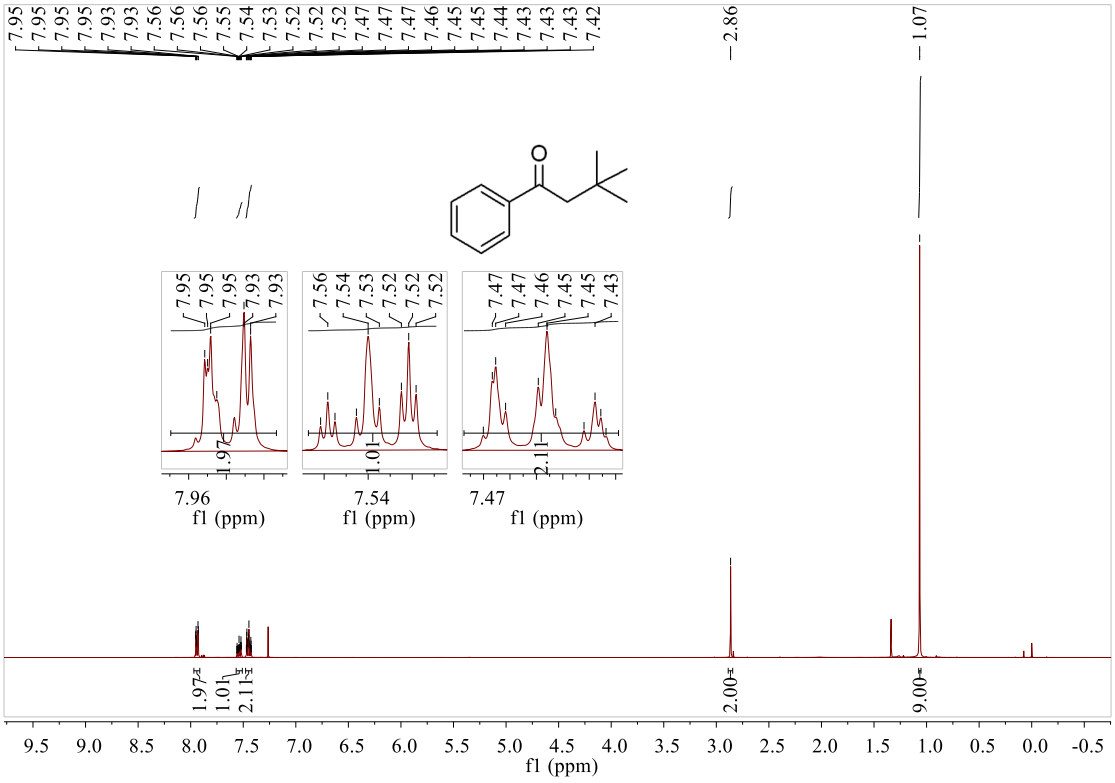
^{1}H NMR spectrum of **N-(3,3-dimethyl-1-phenylbutylidene)acetamide (3)**



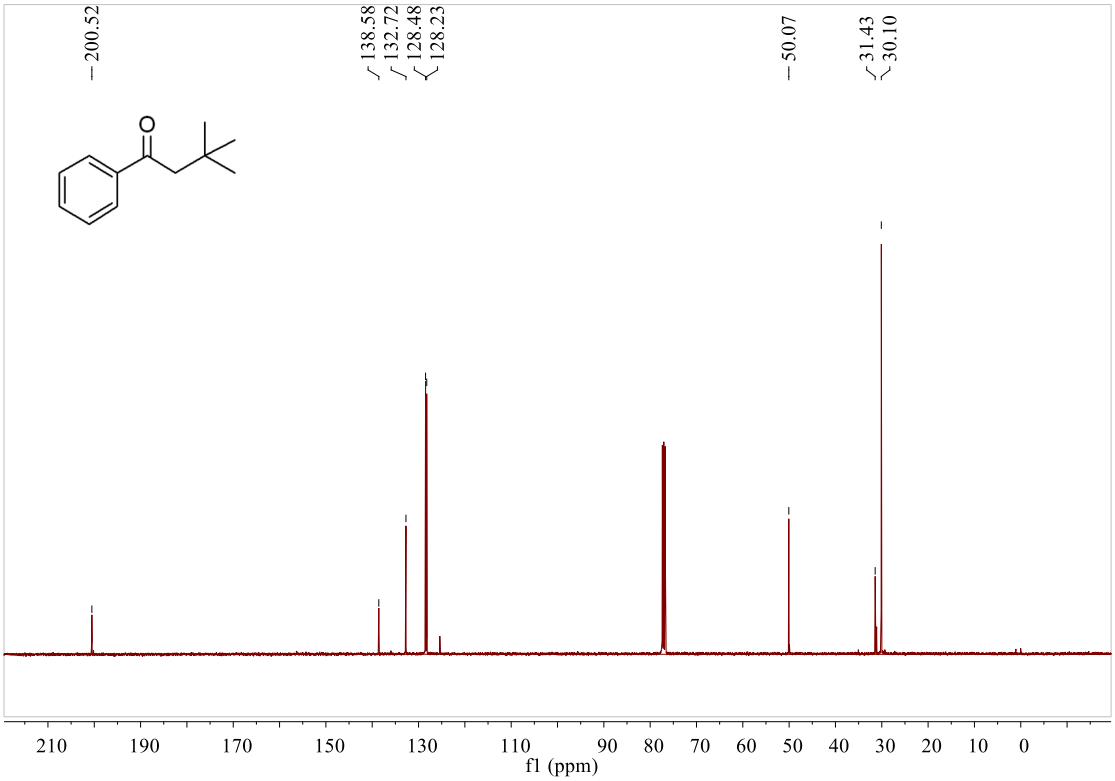
^{13}C NMR spectrum of **N-(3,3-dimethyl-1-phenylbutylidene)acetamide (3)**



^{1}H NMR spectrum of **3,3-dimethyl-1-phenylbutan-1-one (5)**

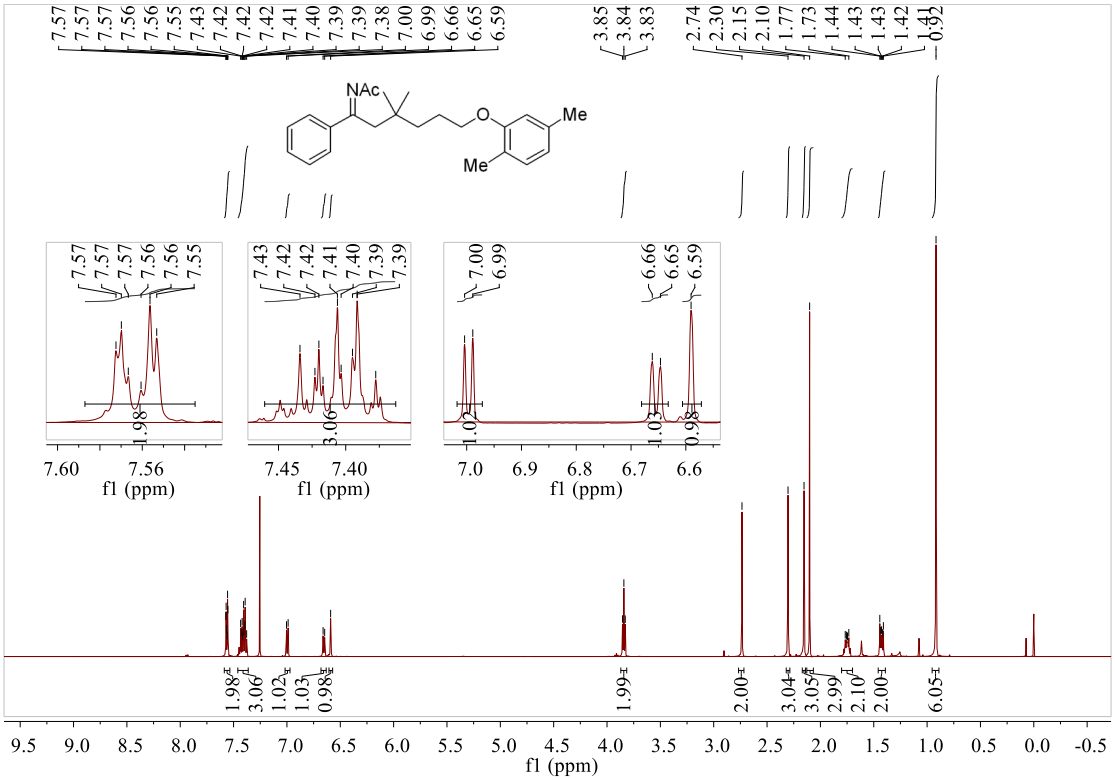


^{13}C NMR spectrum of **3,3-dimethyl-1-phenylbutan-1-one (5)**



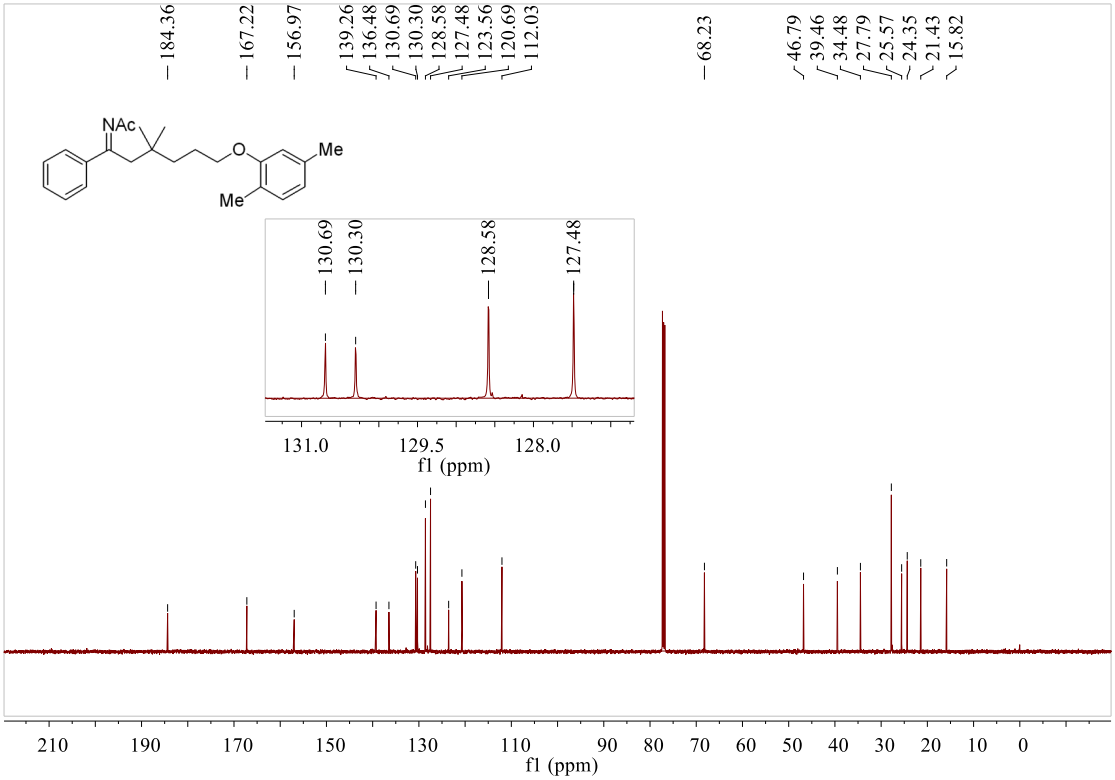
^{1}H NMR spectrum of

N-(6-(2,5-dimethylphenoxy)-3,3-dimethyl-1-phenylhexylidiene)acetamide (6)



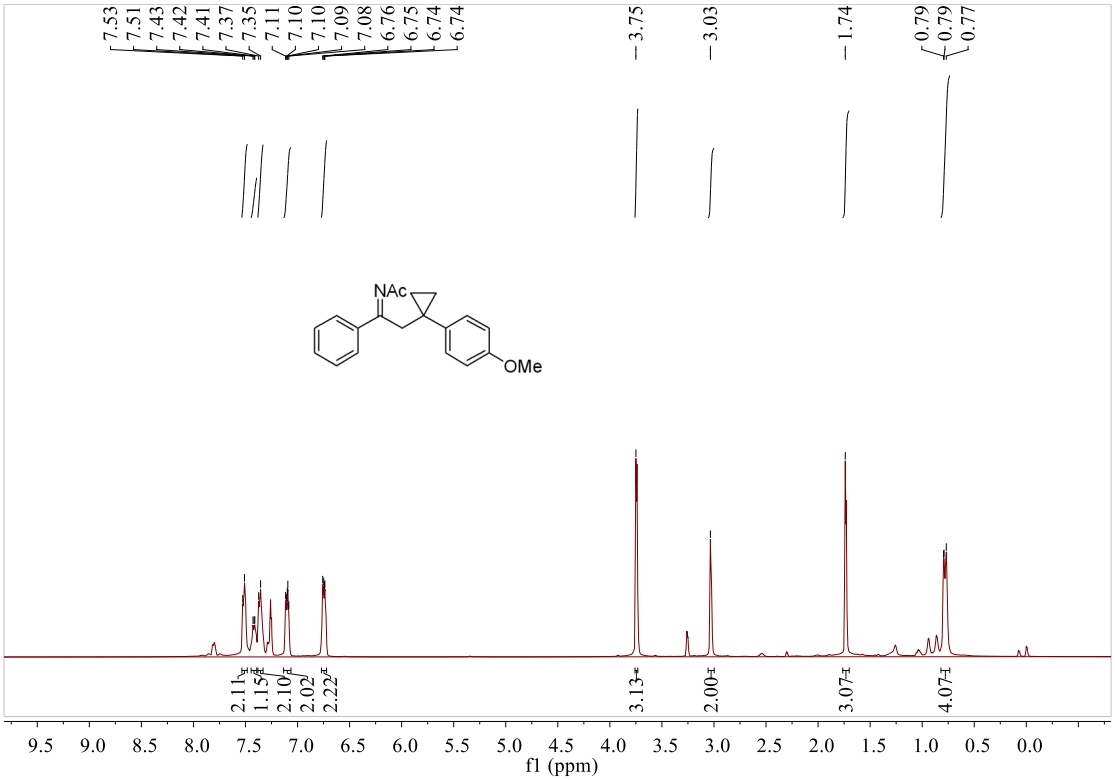
^{13}C NMR spectrum of

N-(6-(2,5-dimethylphenoxy)-3,3-dimethyl-1-phenylhexylidiene)acetamide (6)



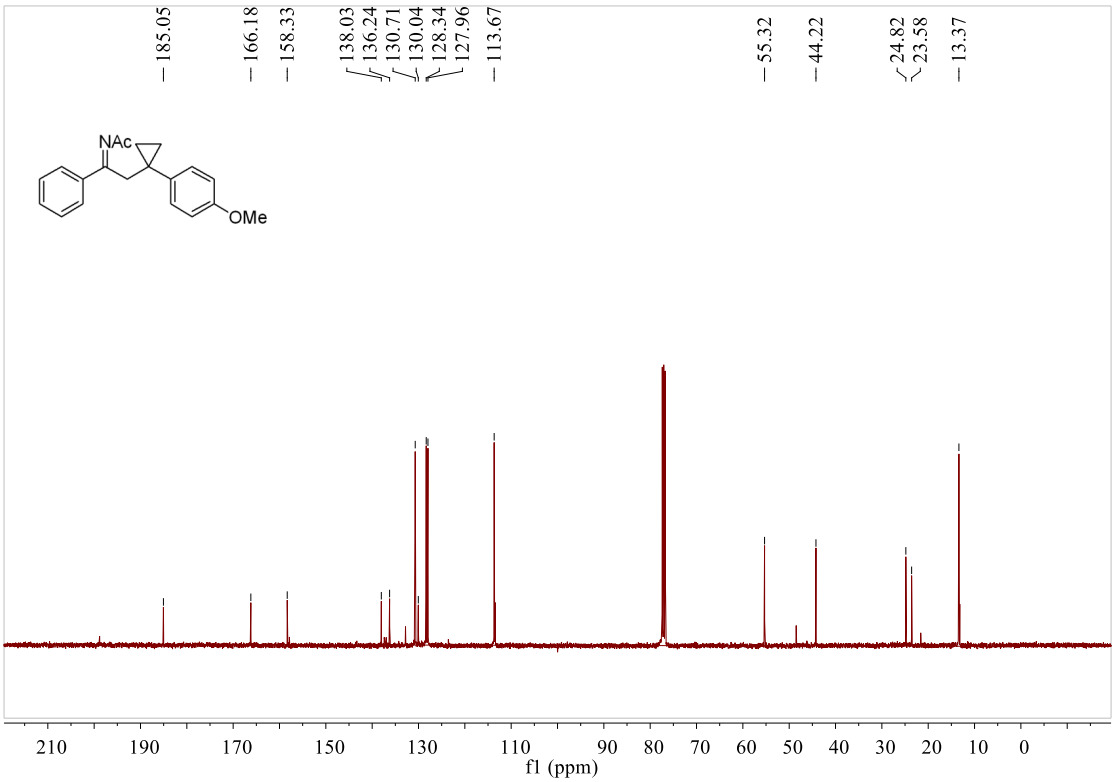
^{1}H NMR spectrum of

N-(2-(1-(4-methoxyphenyl)cyclopropyl)-1-phenylethylidene)acetamide (7)

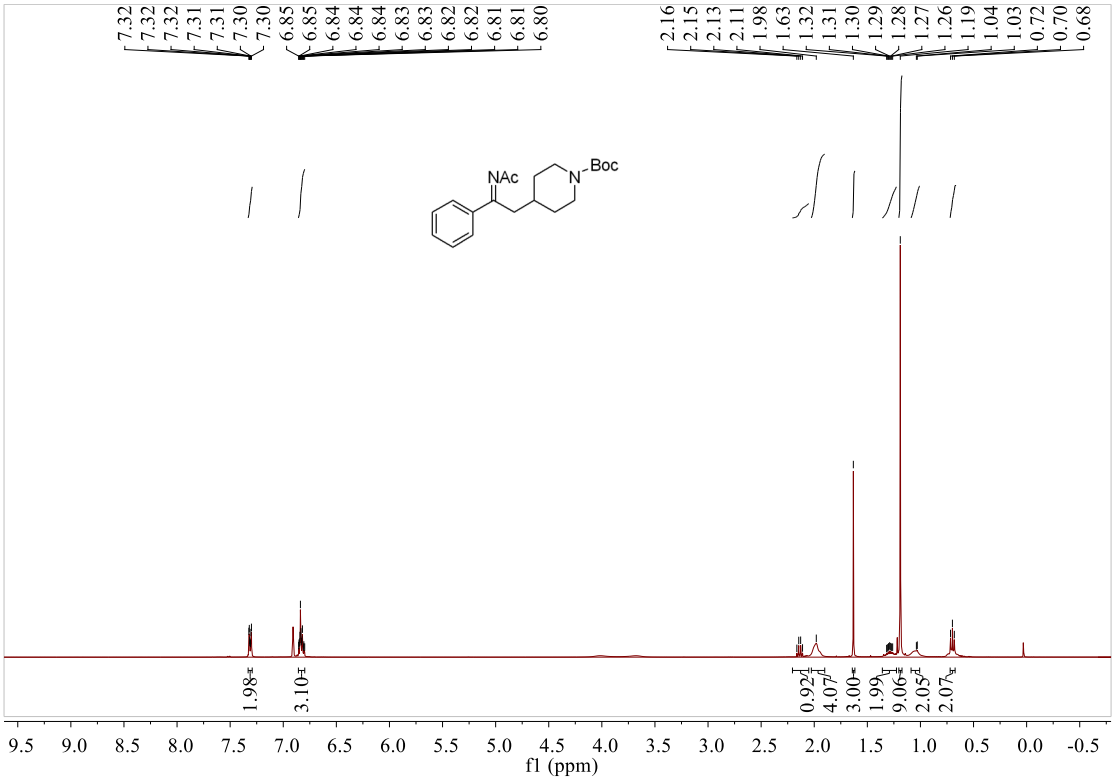


^{13}C NMR spectrum of

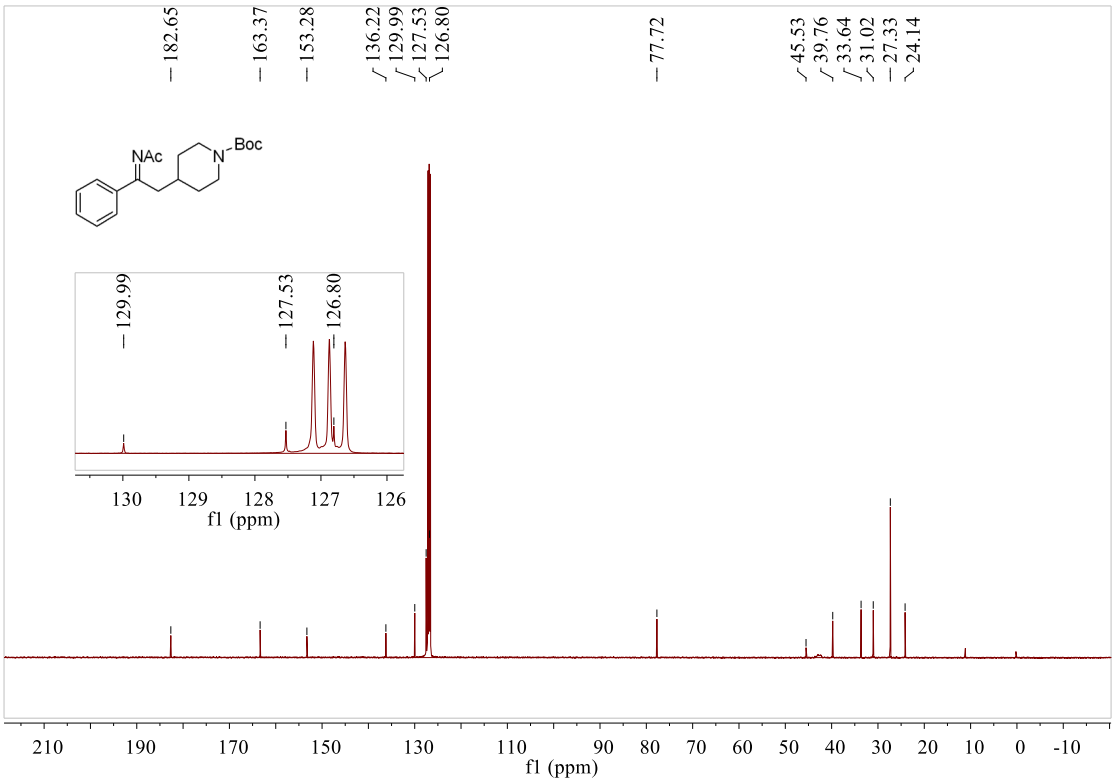
N-(2-(1-(4-methoxyphenyl)cyclopropyl)-1-phenylethylidene)acetamide (7)



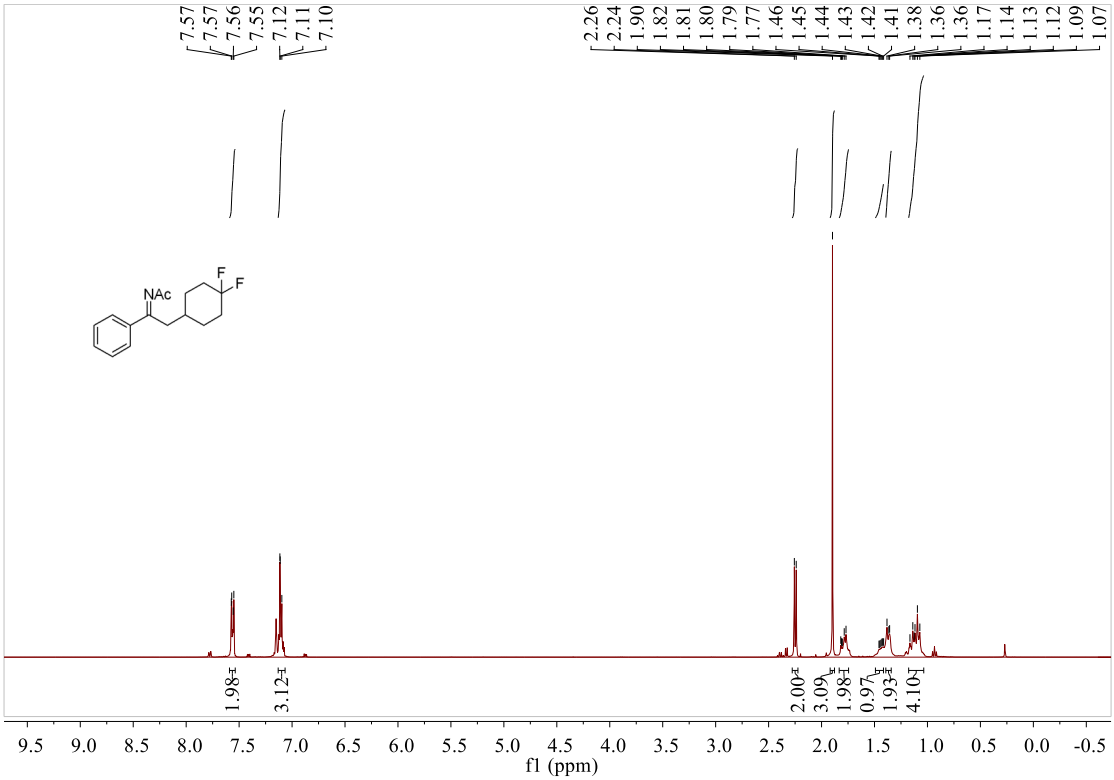
^{1}H NMR spectrum of **tert-butyl 4-(2-(acetylimino)-2-phenylethyl)piperidine-1-carboxylate (8)**



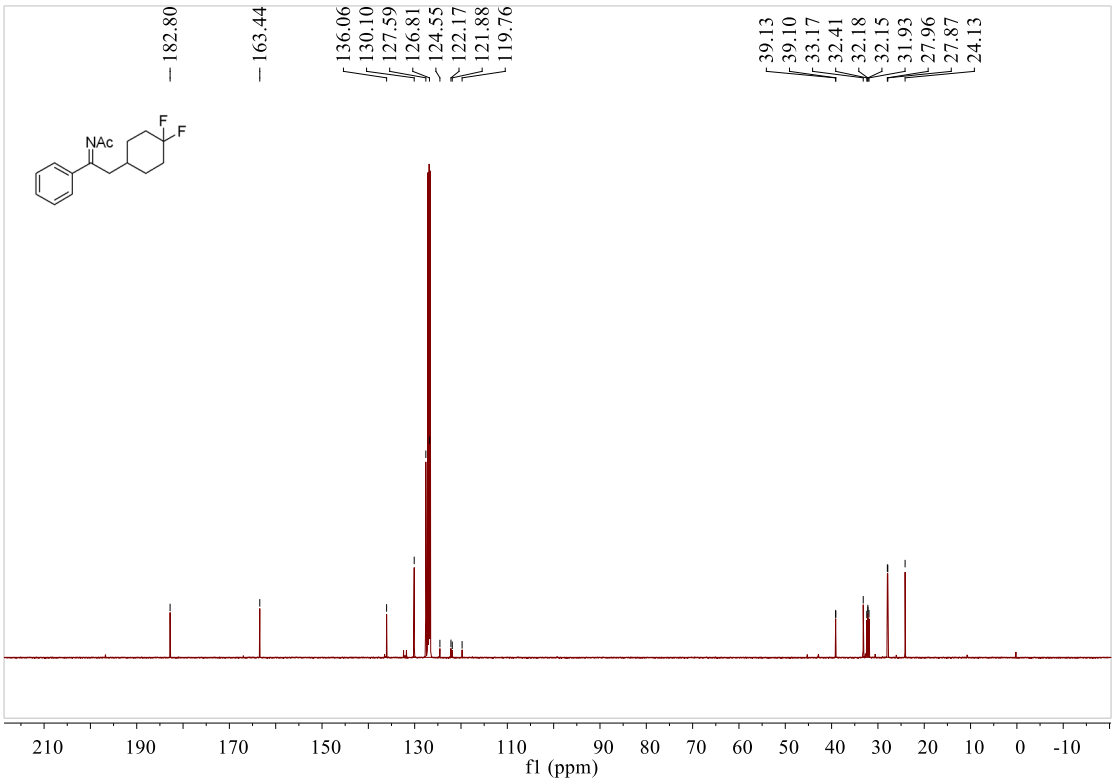
^{1}H NMR spectrum of **tert-butyl 4-(2-(acetylimino)-2-phenylethyl)piperidine-1-carboxylate (8)**



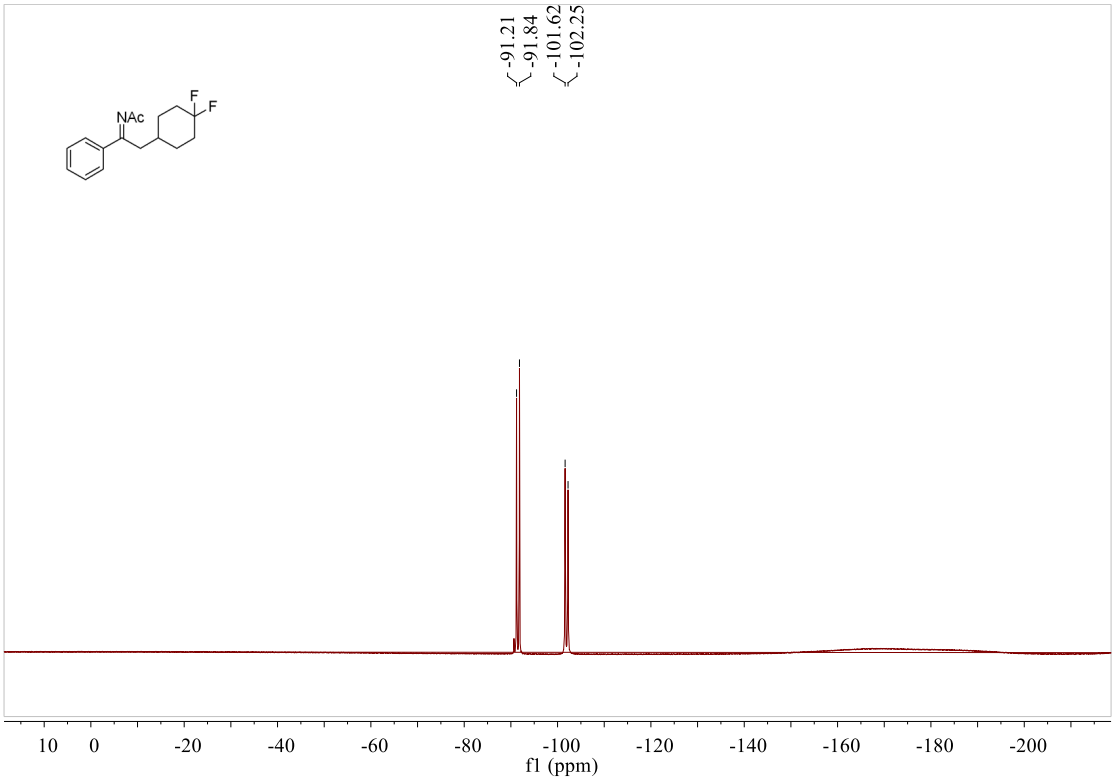
^{1}H NMR spectrum of **N-(2-(4,4-difluorocyclohexyl)-1-phenylethylidene)acetamide (9)**



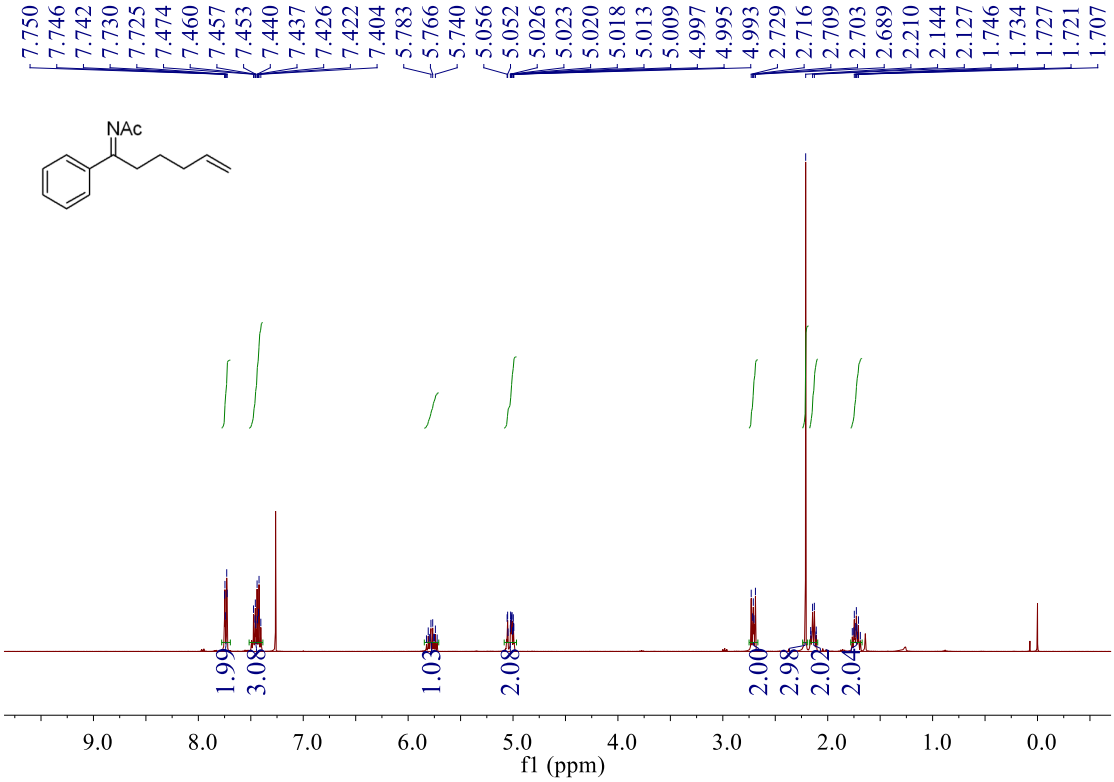
^{13}C NMR spectrum of **N-(2-(4,4-difluorocyclohexyl)-1-phenylethylidene)acetamide (9)**



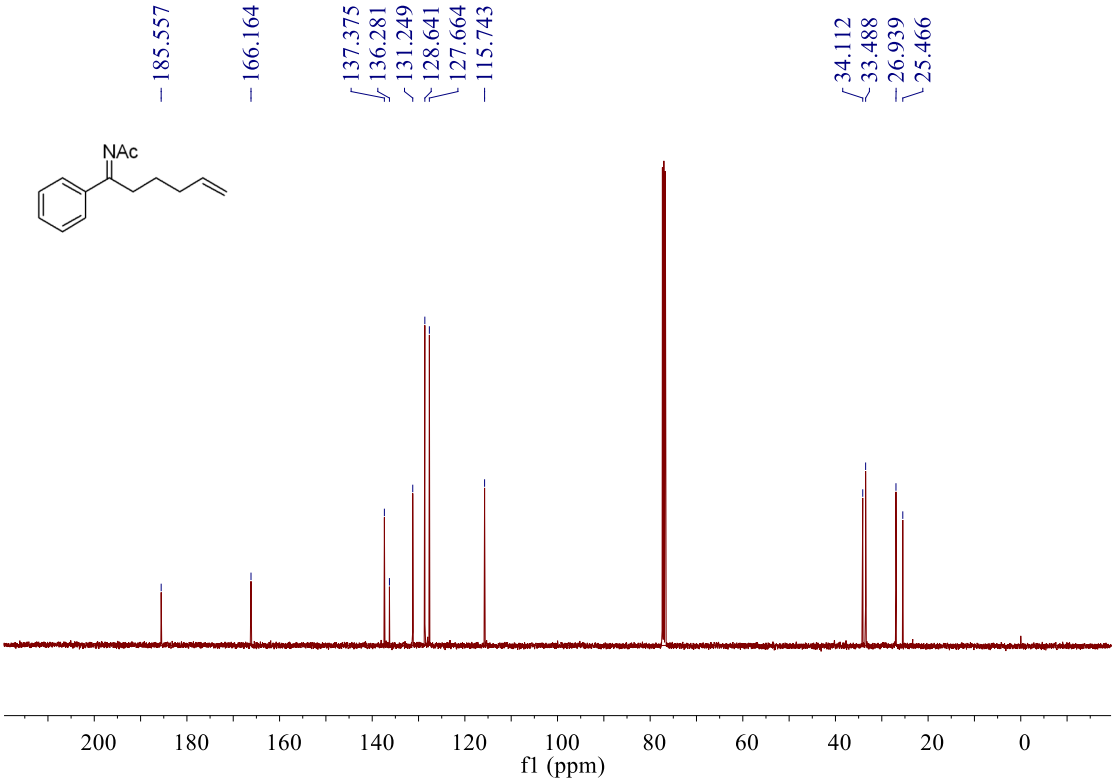
^{19}F NMR spectrum of **N-(2-(4,4-difluorocyclohexyl)-1-phenylethylidene)acetamide (9)**



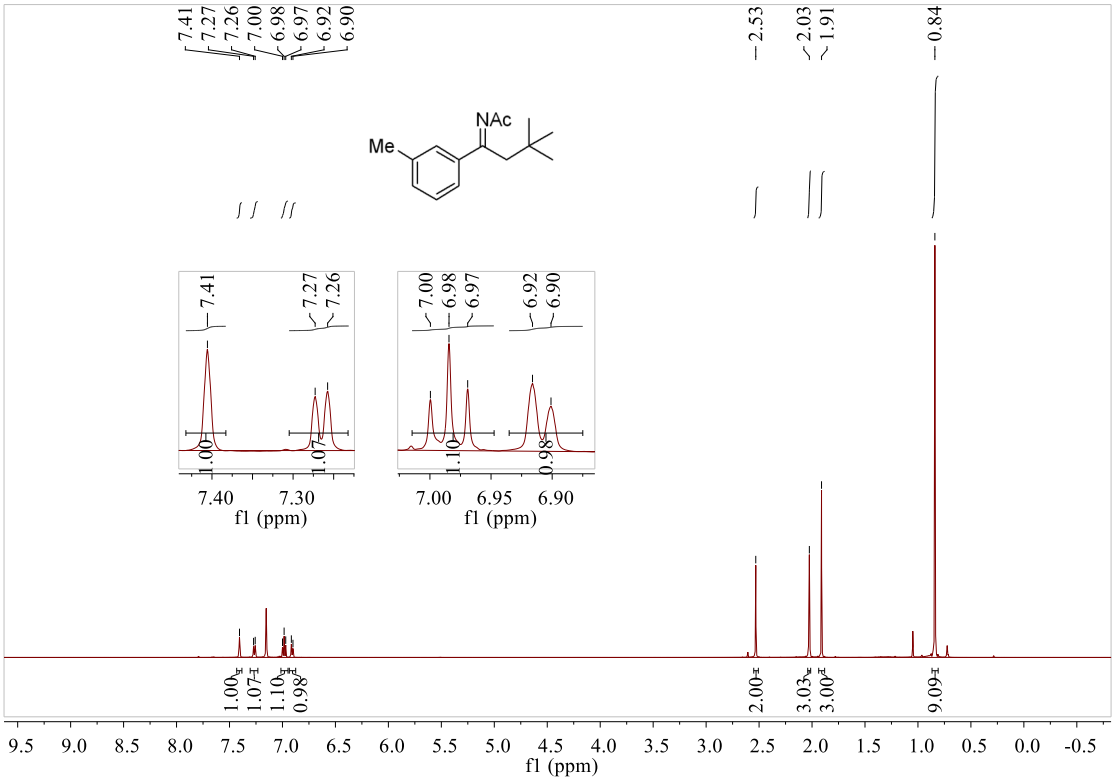
^{1}H NMR spectrum of **N-(1-phenylhex-5-en-1-ylidene)acetamide (10)**



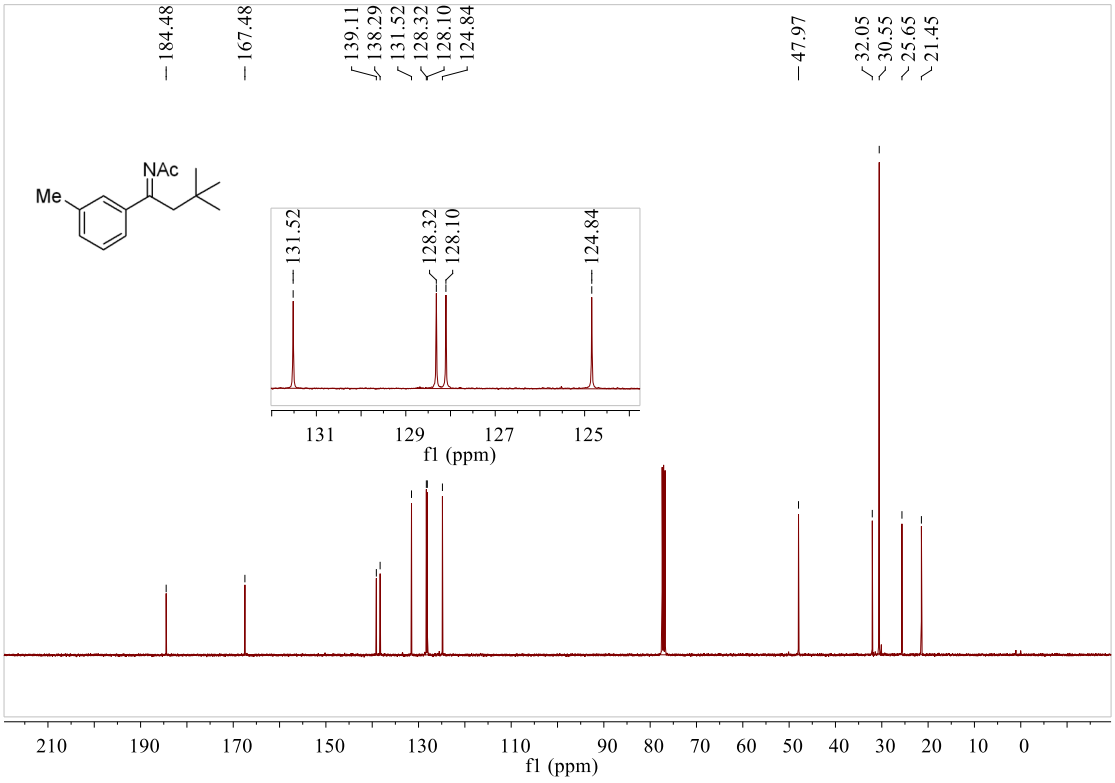
^{13}C NMR spectrum of **N-(1-phenylhex-5-en-1-ylidene)acetamide (10)**



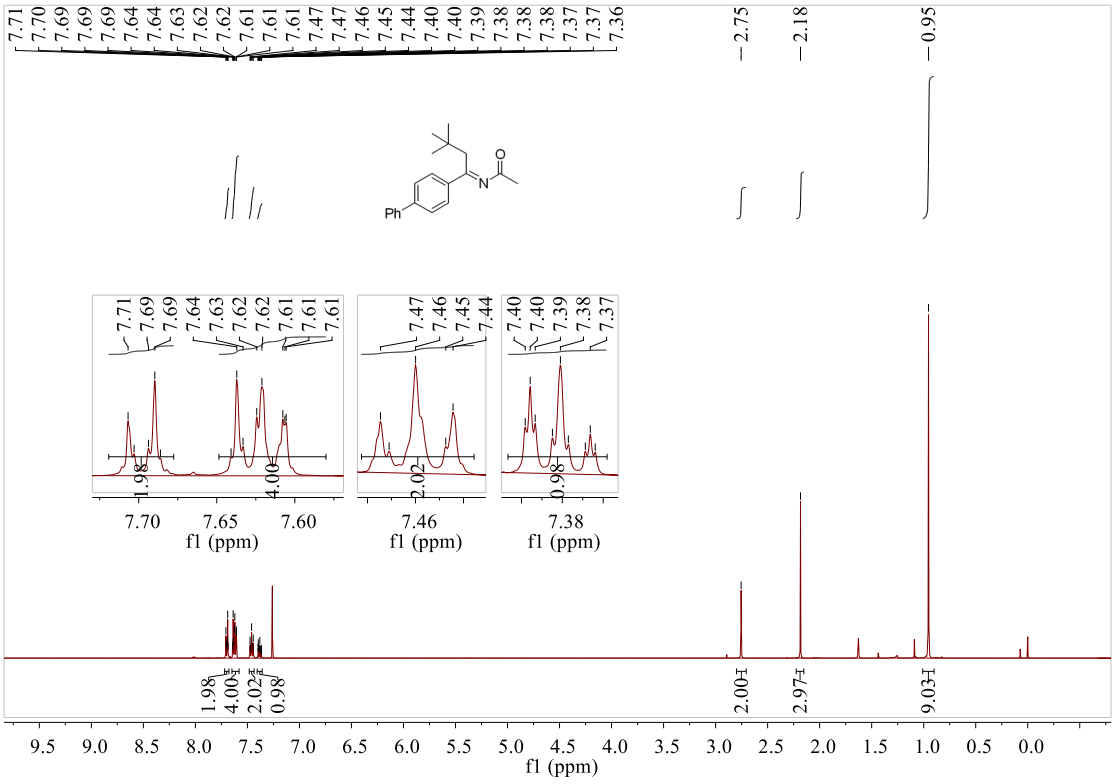
^{1}H NMR spectrum of **N-(3,3-dimethyl-1-(m-tolyl)butylidene)acetamide (11)**



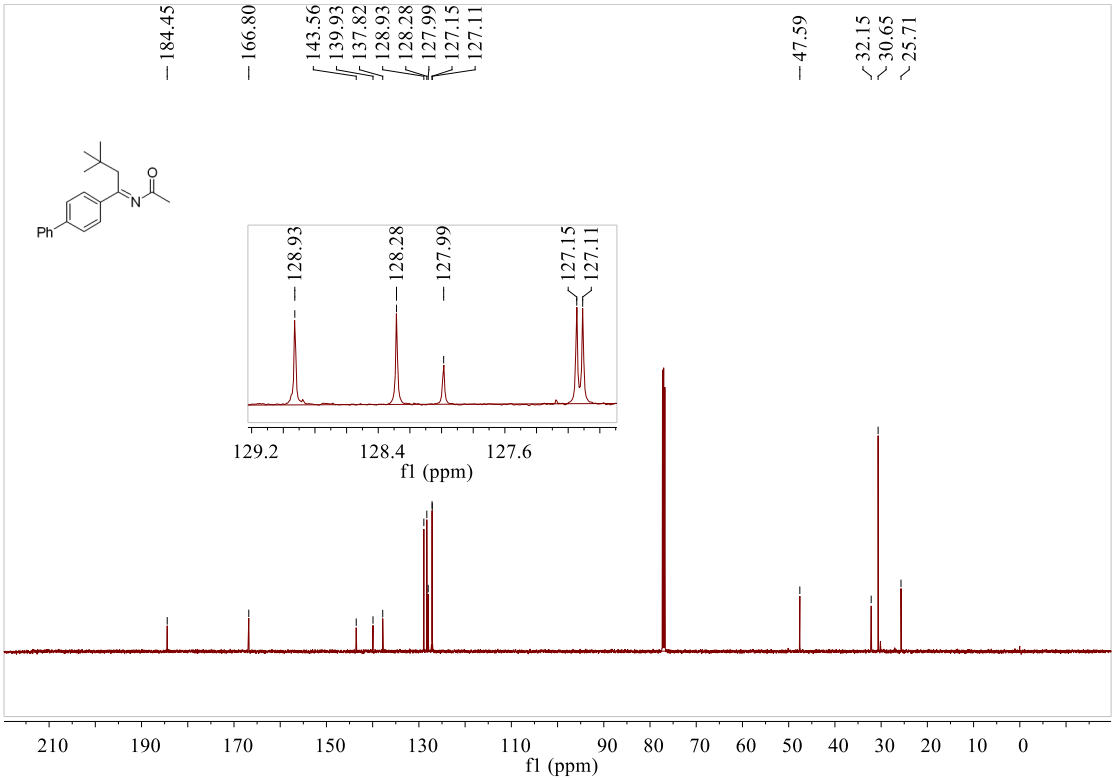
^{13}C NMR spectrum of **N-(3,3-dimethyl-1-(m-tolyl)butylidene)acetamide (11)**



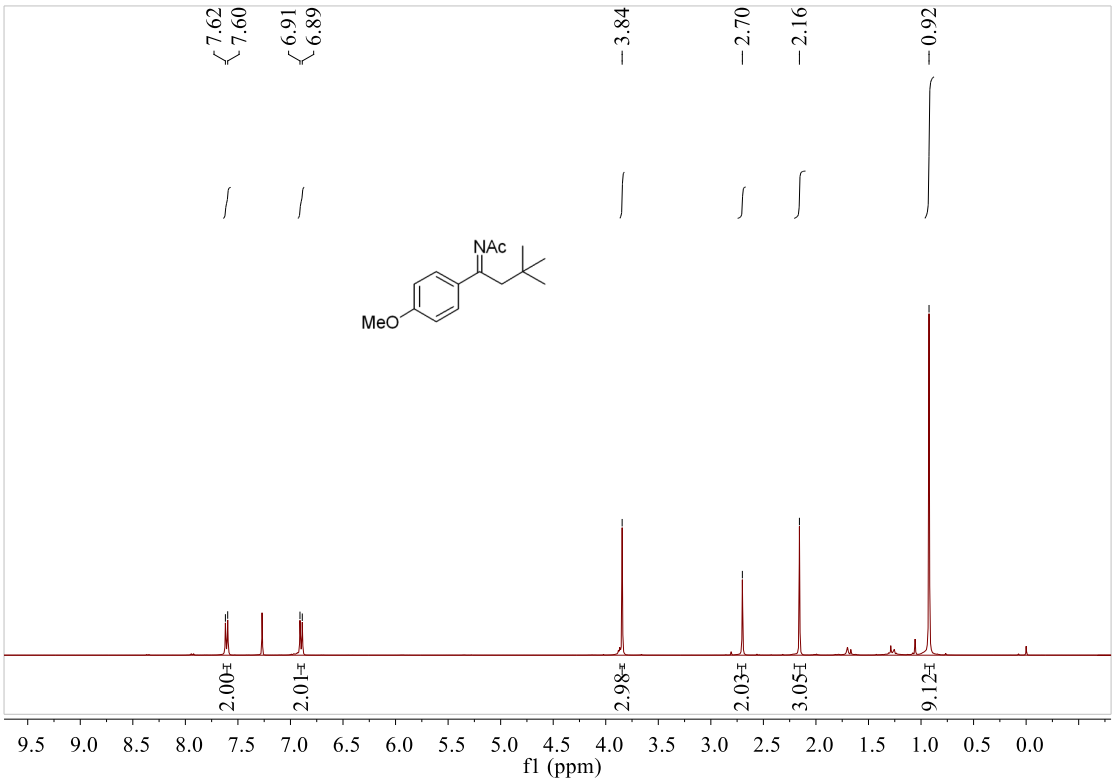
^{1}H NMR spectrum of **(*E*)-N-(1-([1,1'-biphenyl]-4-yl)-3,3-dimethylbutylidene)acetamide (12)**



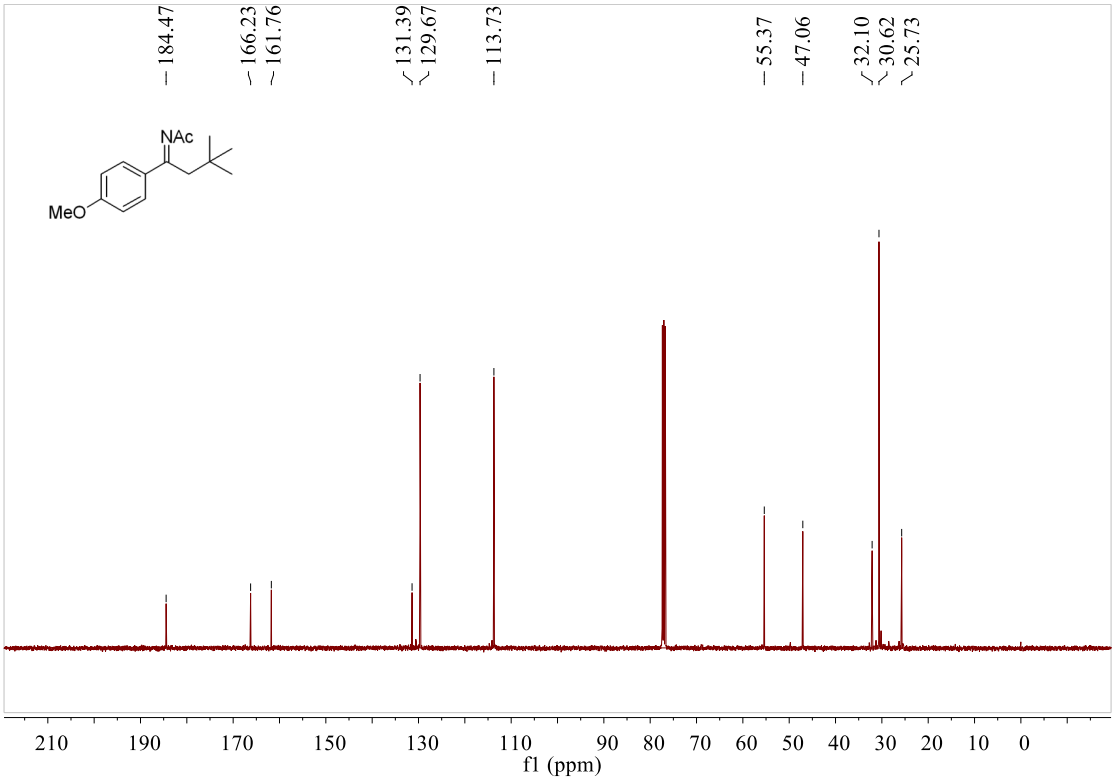
^{13}C NMR spectrum of **(*E*)-N-(1-([1,1'-biphenyl]-4-yl)-3,3-dimethylbutylidene)acetamide (12)**



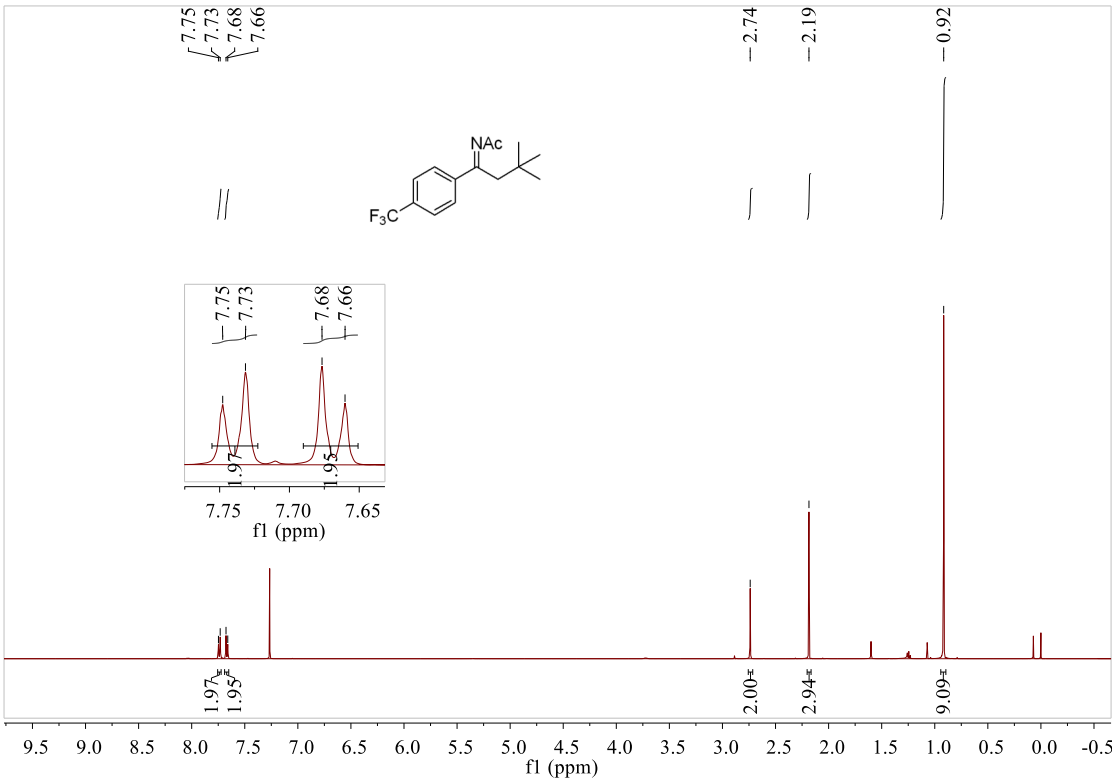
^{1}H NMR spectrum of **N-(1-(4-methoxyphenyl)-3,3-dimethylbutylidene)acetamide (13)**



^{13}C NMR spectrum of **N-(1-(4-methoxyphenyl)-3,3-dimethylbutylidene)acetamide (13)**

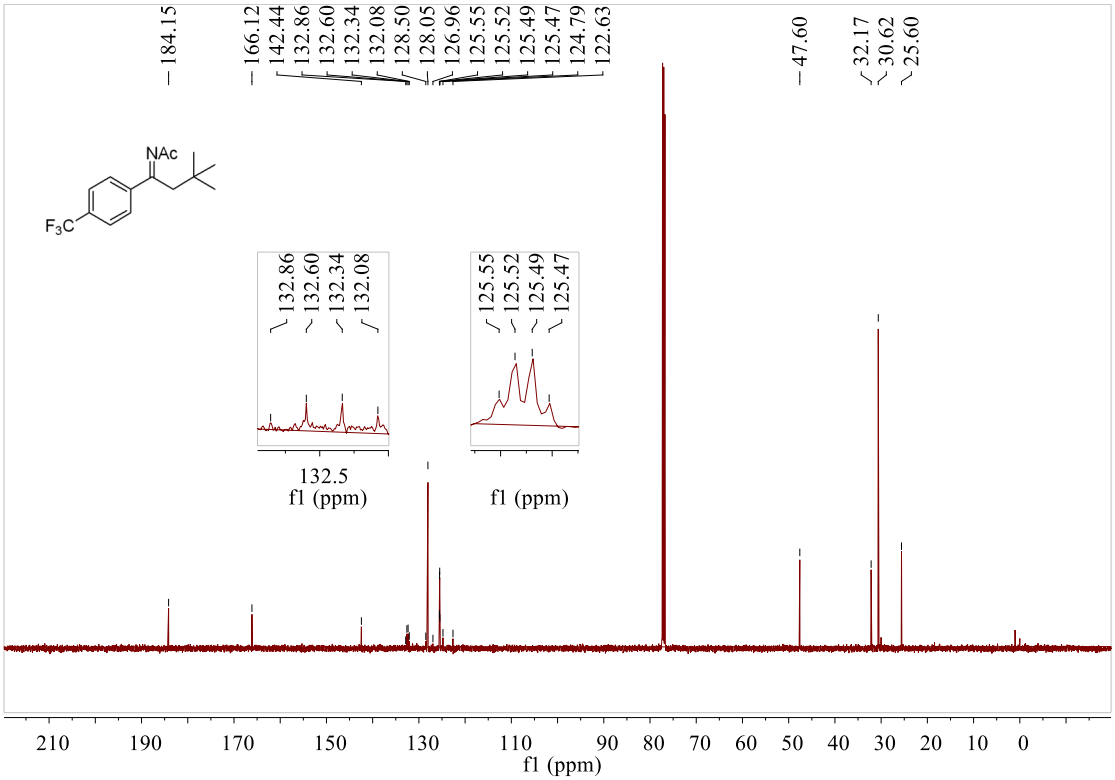


^{1}H NMR spectrum of **N-(3,3-dimethyl-1-(4-(trifluoromethyl)phenyl)butylidene)acetamide (14)**



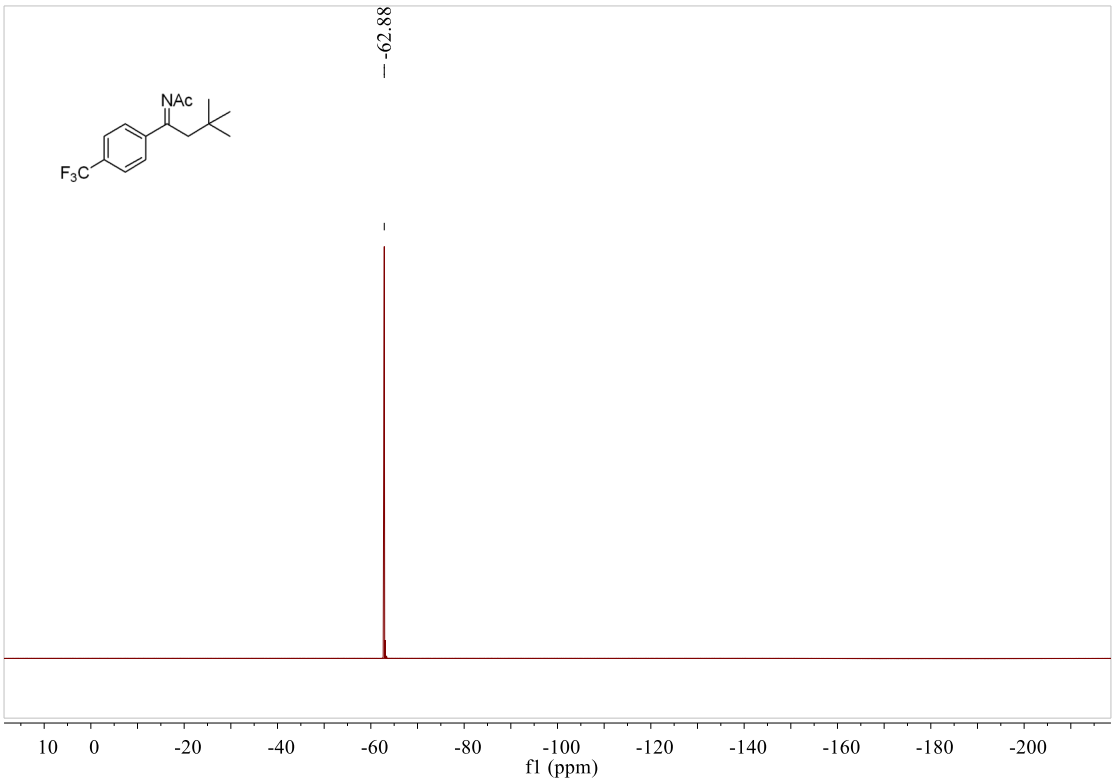
^{13}C NMR spectrum of

N-(3,3-dimethyl-1-(4-(trifluoromethyl)phenyl)butylidene)acetamide (14)

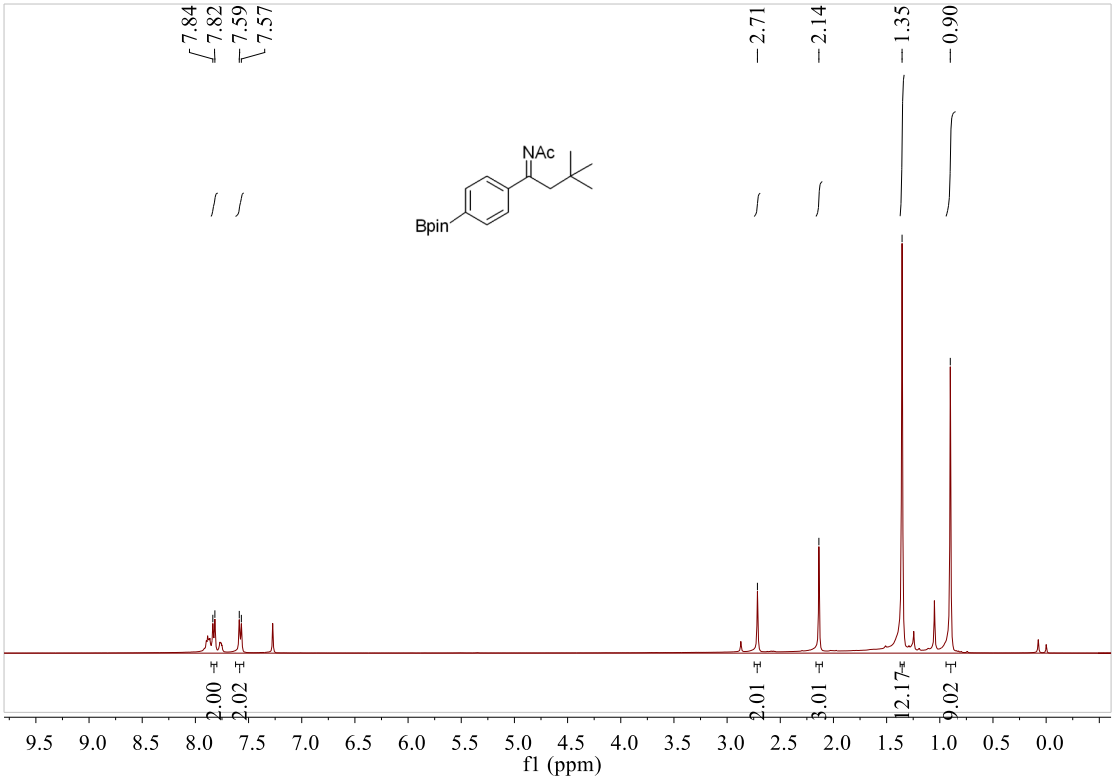


^{19}F NMR spectrum of

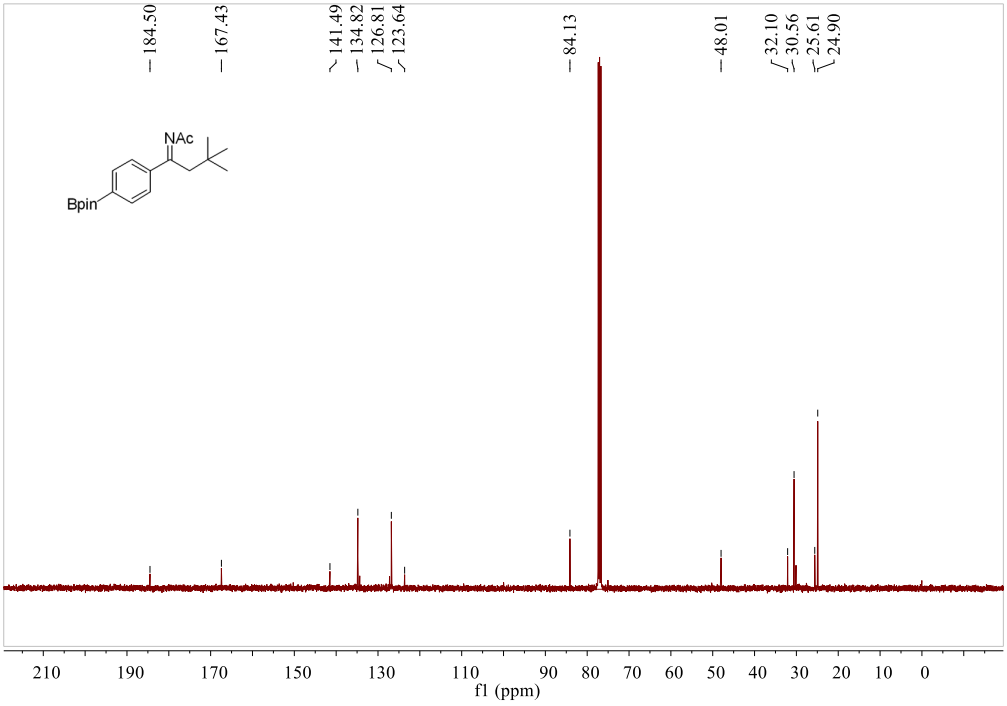
N-(3,3-dimethyl-1-(4-(trifluoromethyl)phenyl)butylidene)acetamide (14)



^{1}H NMR spectrum of **N-(3,3-dimethyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)butylidene)acetamide (15)**

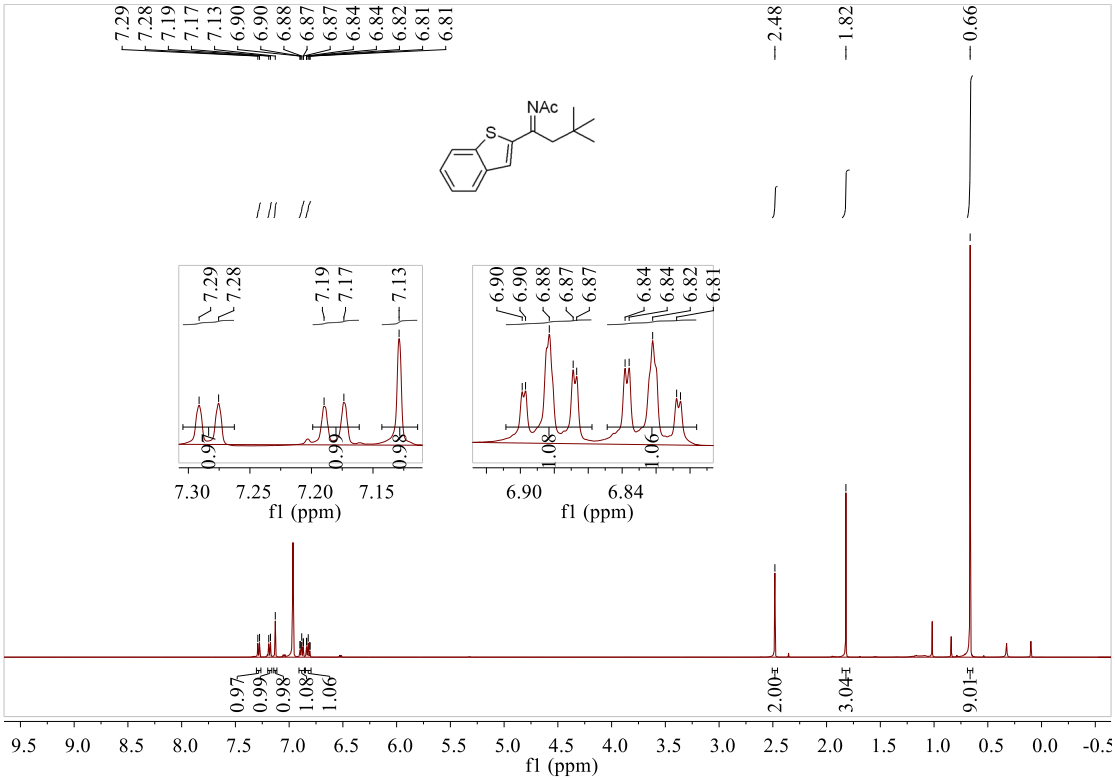


^{13}C NMR spectrum of **N-(3,3-dimethyl-1-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl)phenyl)butylidene)acetamide (15)**



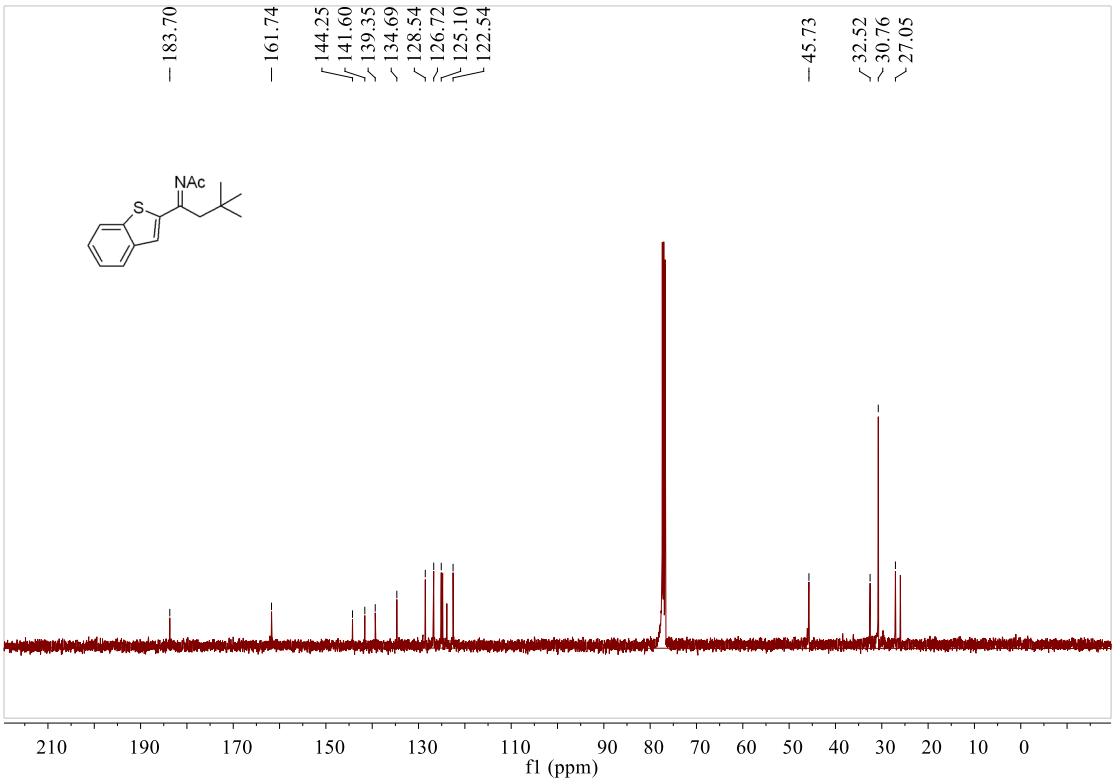
^{1}H NMR spectrum of

N-(1-(benzo[b]thiophen-2-yl)-3,3-dimethylbutylidene)acetamide (16)

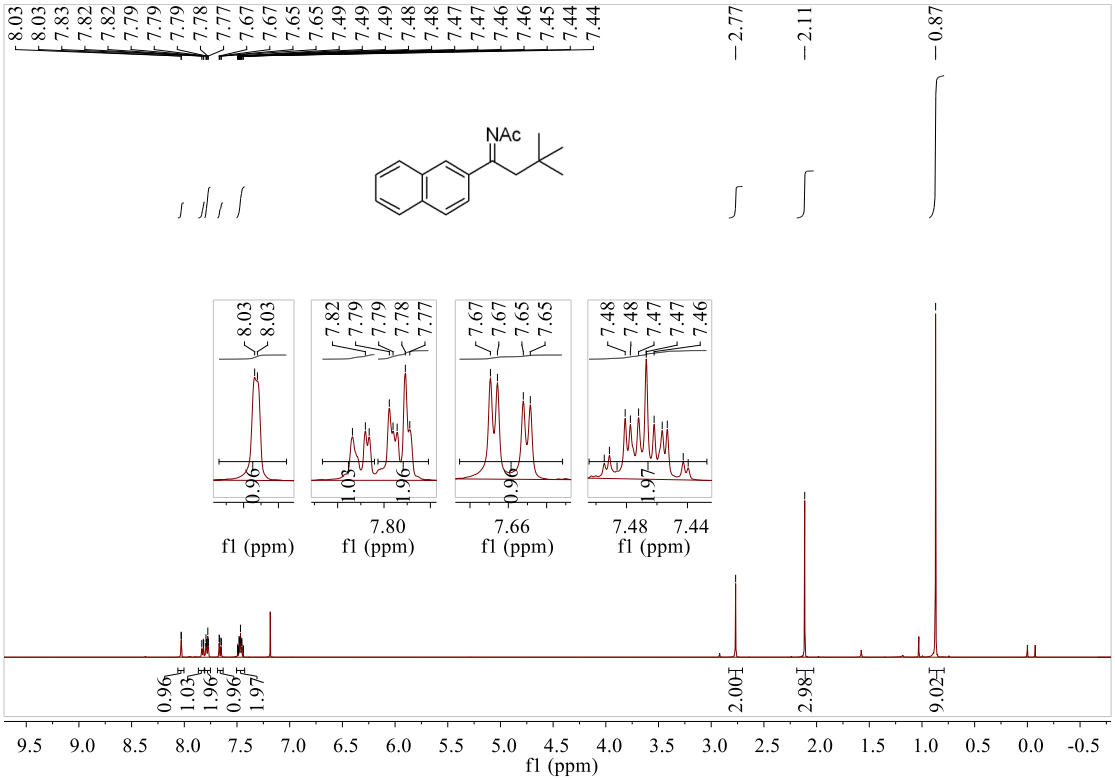


^{13}C NMR spectrum of

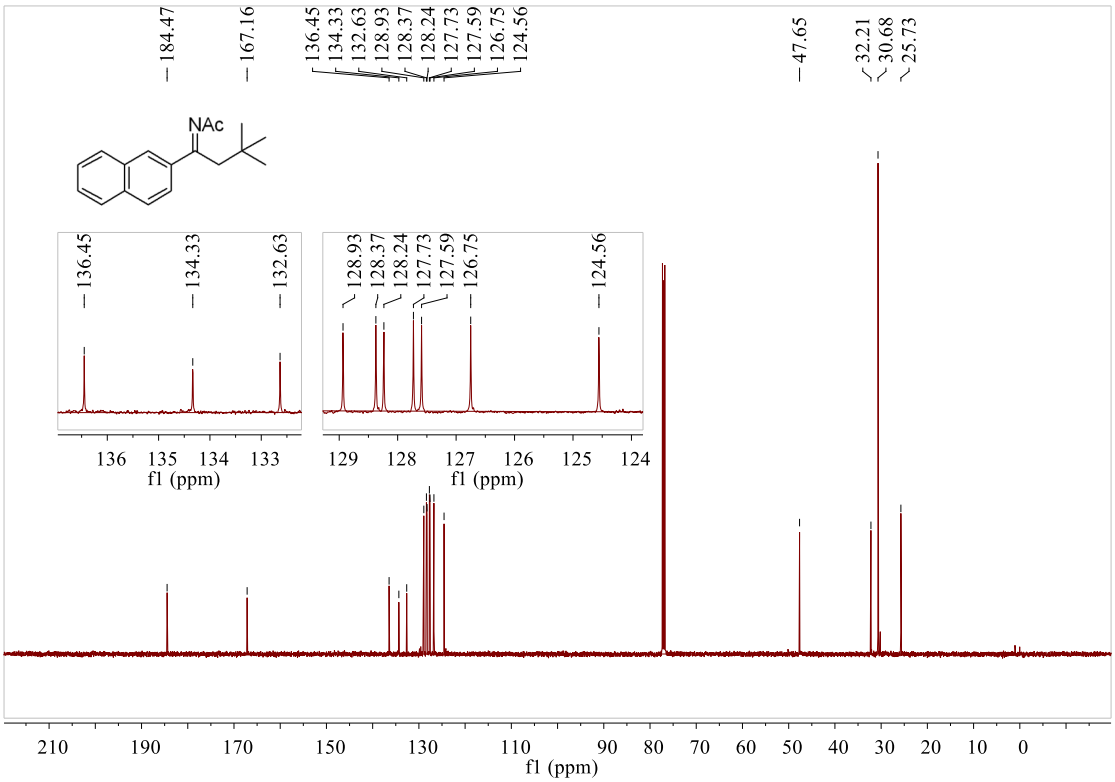
N-(1-(benzo[b]thiophen-2-yl)-3,3-dimethylbutylidene)acetamide (16)



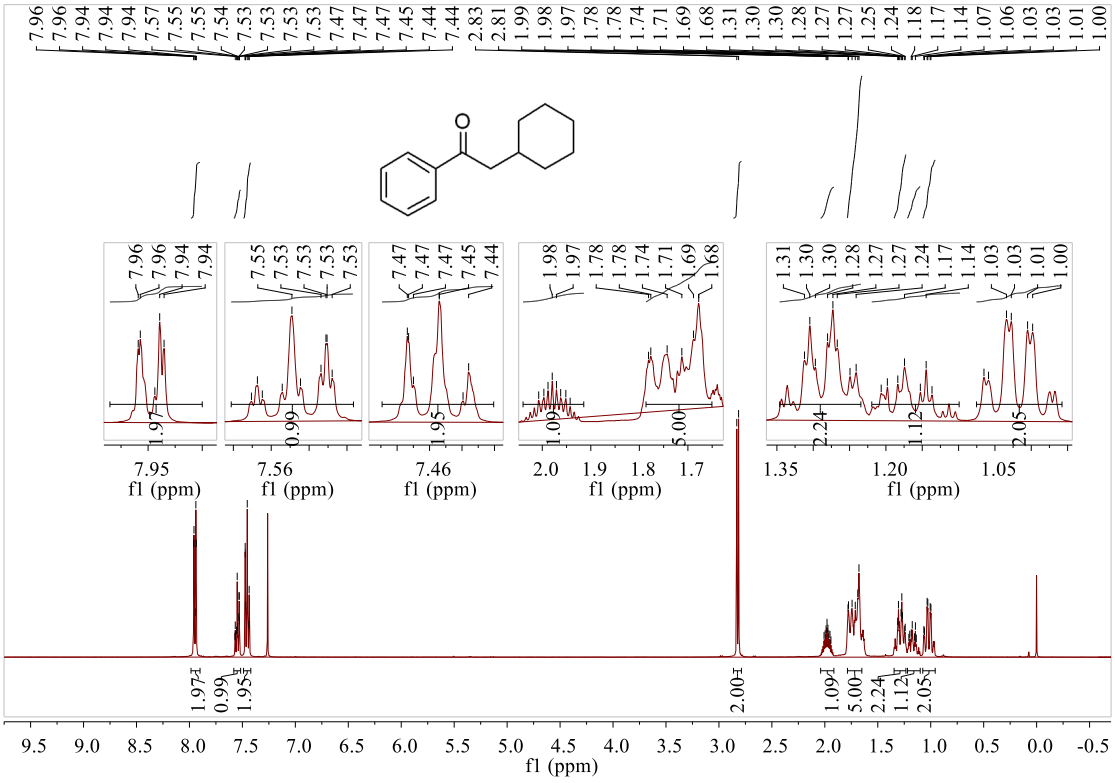
^{1}H NMR spectrum of **N-(3,3-dimethyl-1-(naphthalen-2-yl)butylidene)acetamide (17)**



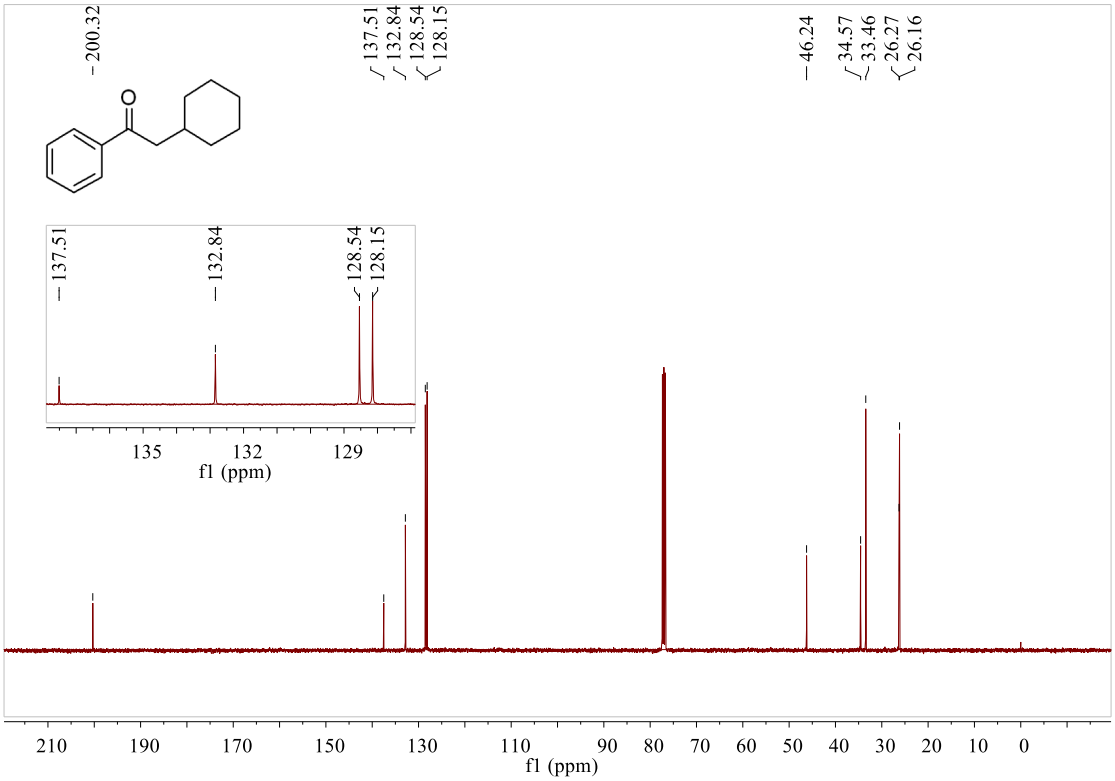
^{13}C NMR spectrum of **N-(3,3-dimethyl-1-(naphthalen-2-yl)butylidene)acetamide (17)**



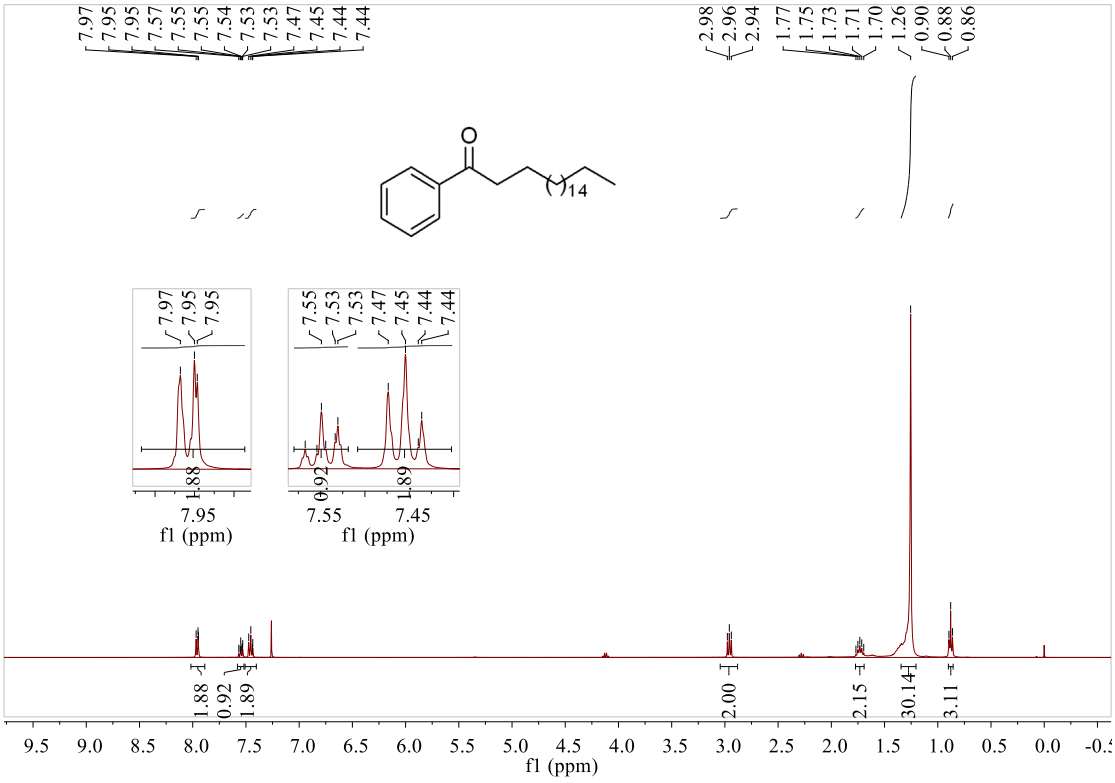
^{1}H NMR spectrum of **2-cyclohexyl-1-phenylethan-1-one (18)**



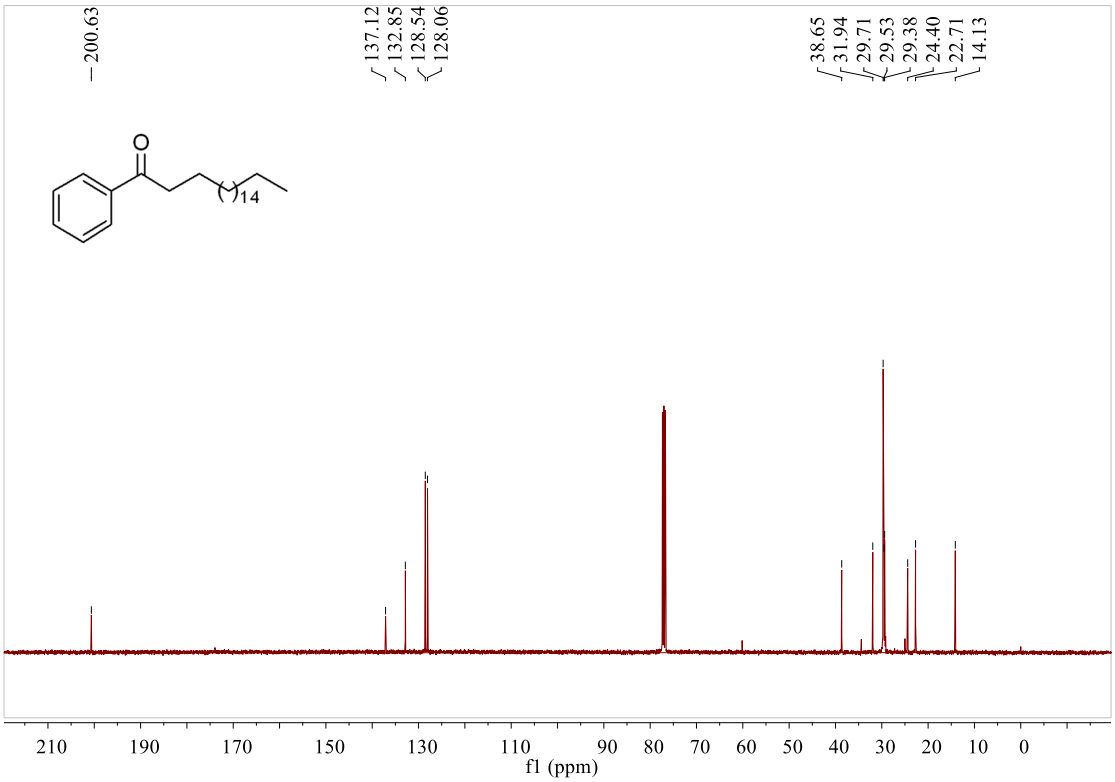
^{13}C NMR spectrum of **2-cyclohexyl-1-phenylethan-1-one (18)**



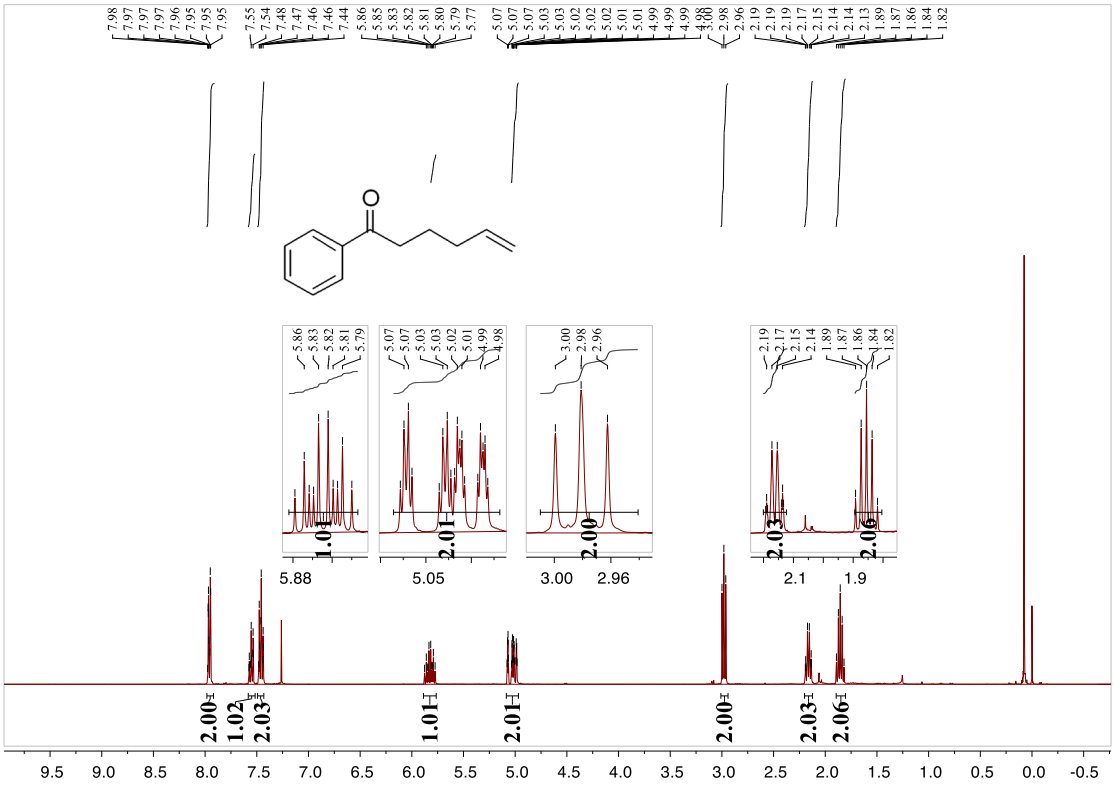
^{1}H NMR spectrum of **1-phenylhexan-1-one (19)**



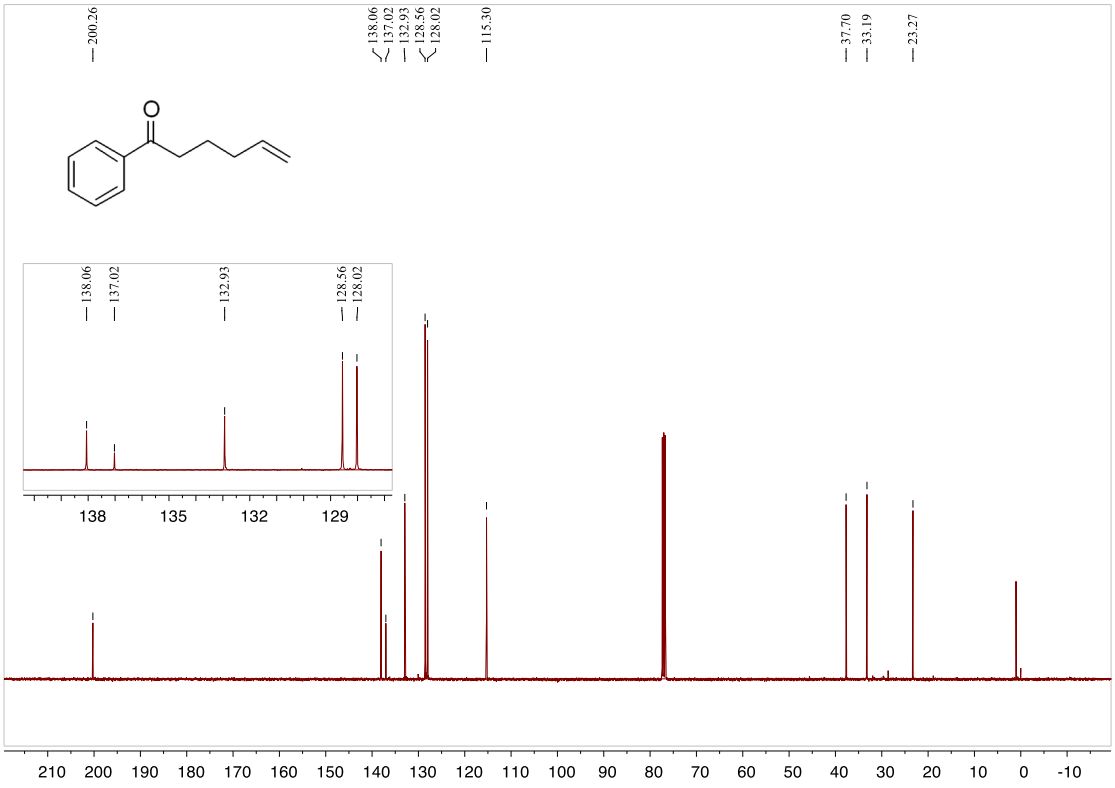
^{13}C NMR spectrum of **1-phenylhexan-1-one (19)**



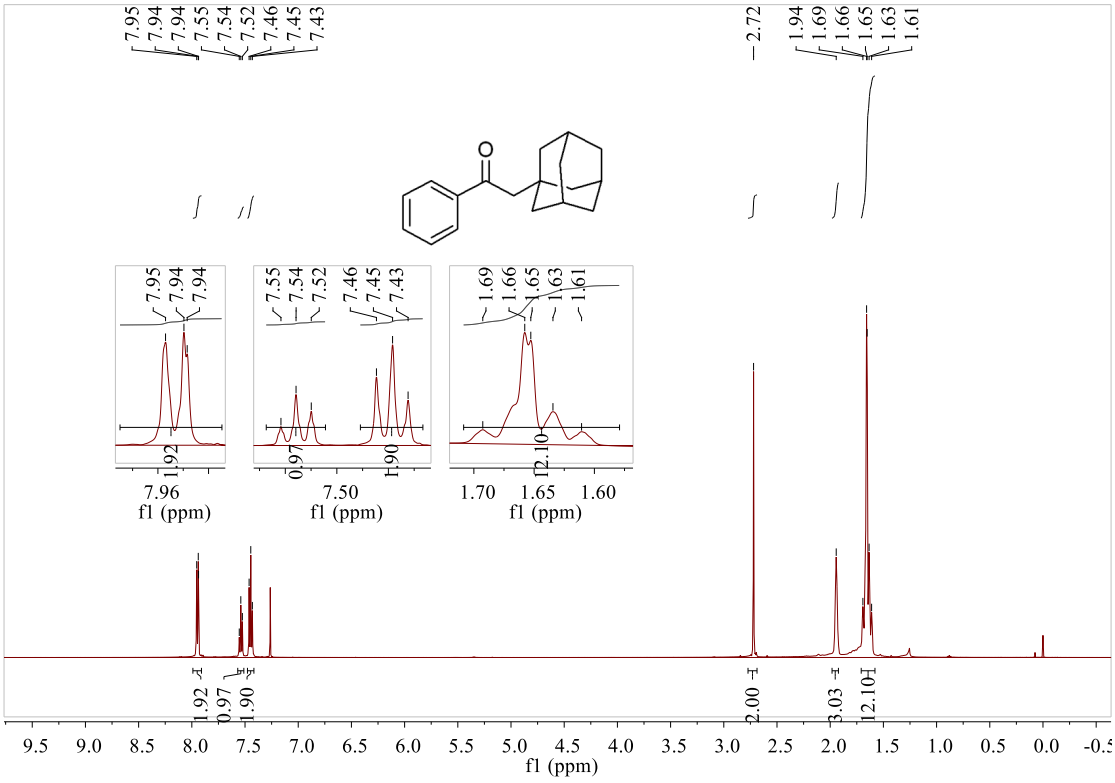
^{1}H NMR spectrum of **1-phenylhex-5-en-1-one (20)**



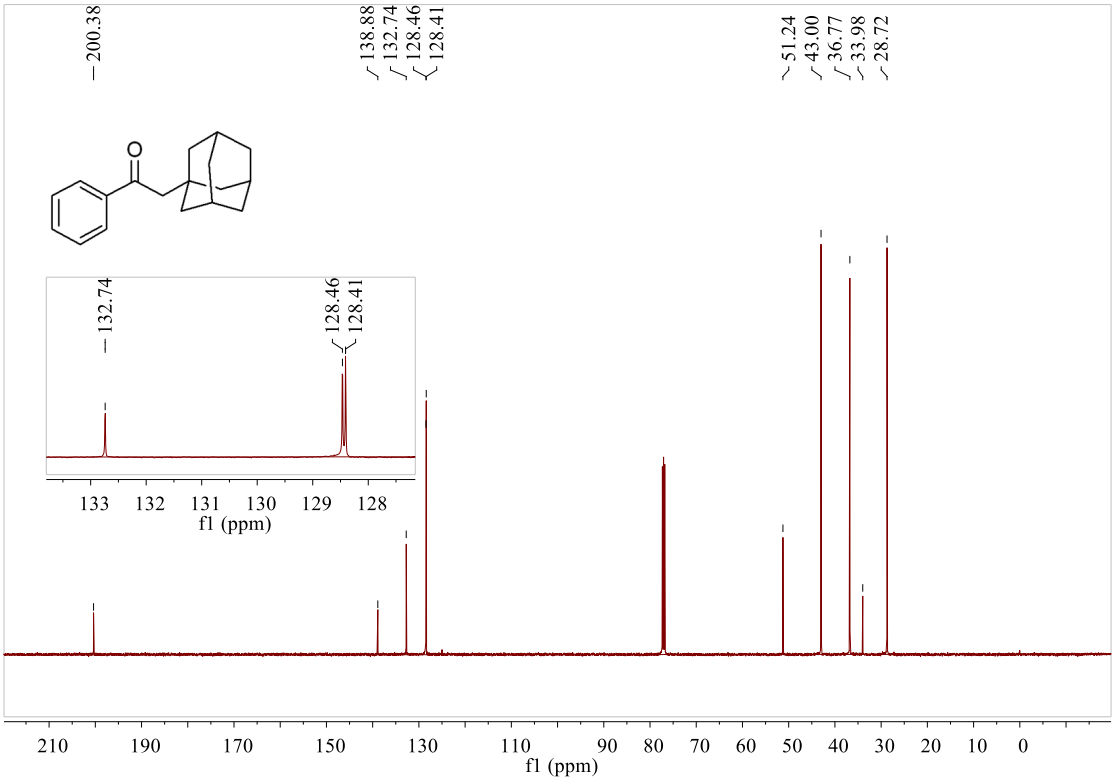
^{13}C NMR spectrum of **1-phenylhex-5-en-1-one (20)**



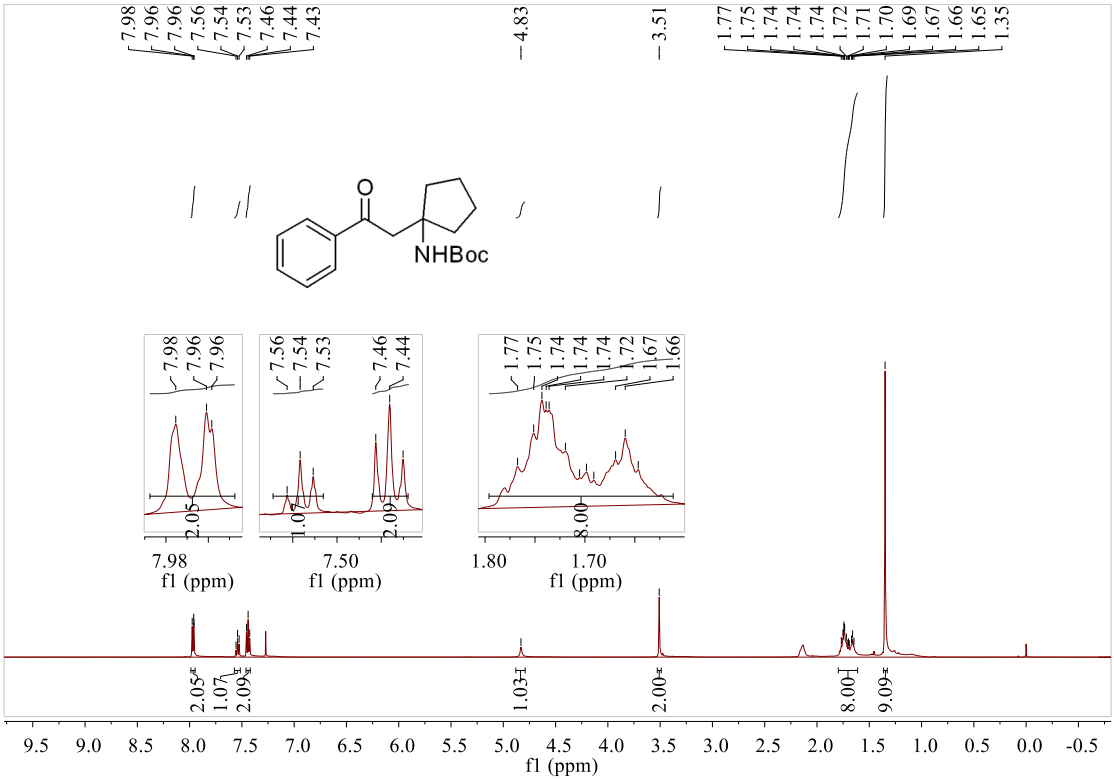
^{1}H NMR spectrum of **2-((3r,5r,7r)-adamantan-1-yl)-1-phenylethan-1-one (21)**



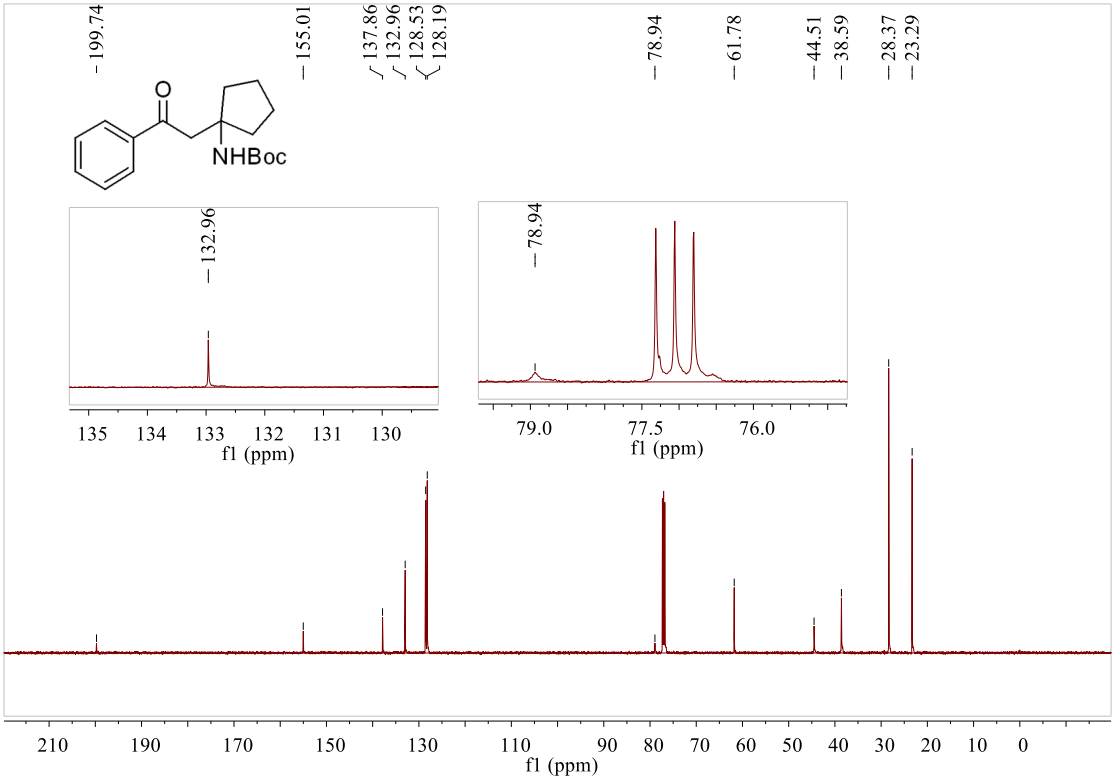
^{13}C NMR spectrum of **2-((3r,5r,7r)-adamantan-1-yl)-1-phenylethan-1-one (21)**



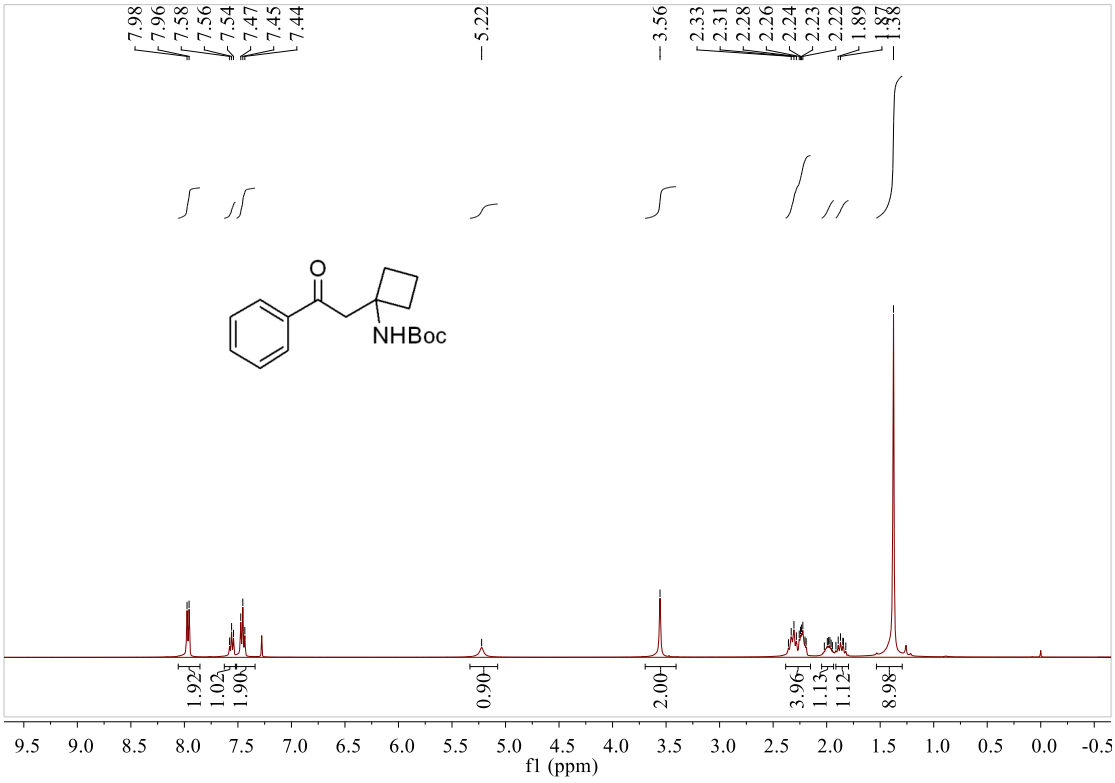
^{1}H NMR spectrum of ***tert*-butyl (1-(2-oxo-2-phenylethyl)cyclopentyl)carbamate (22)**



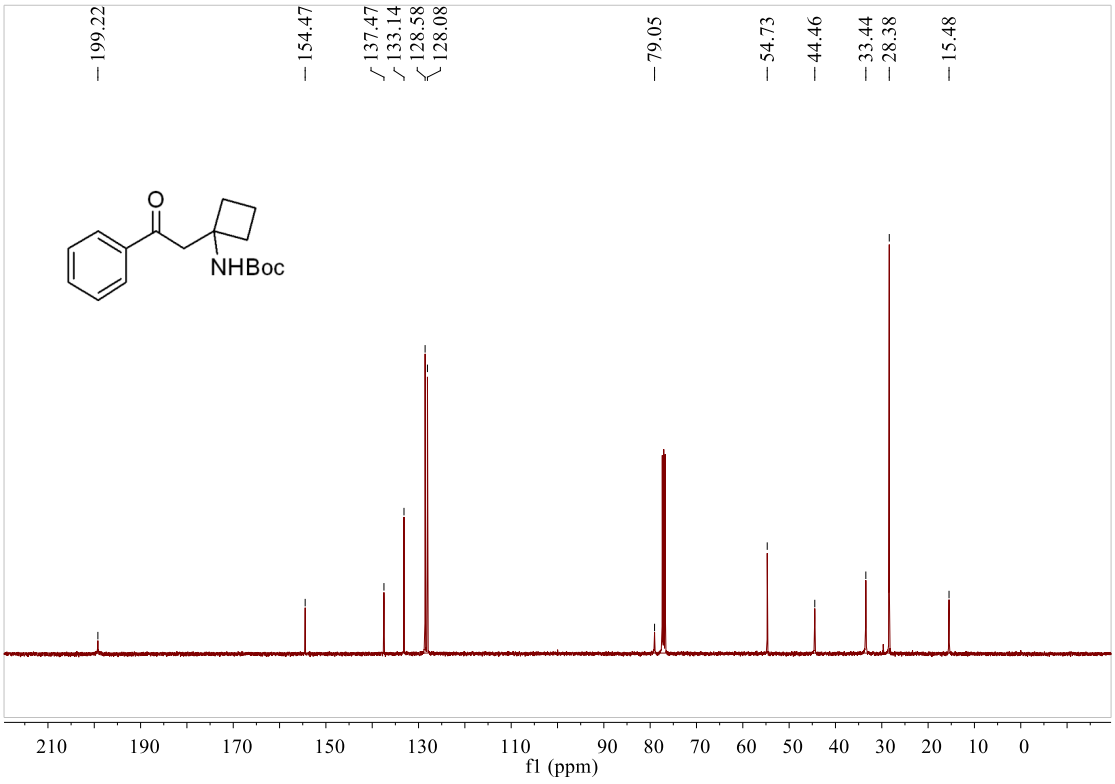
^{13}C NMR spectrum of ***tert*-butyl (1-(2-oxo-2-phenylethyl)cyclopentyl)carbamate (22)**



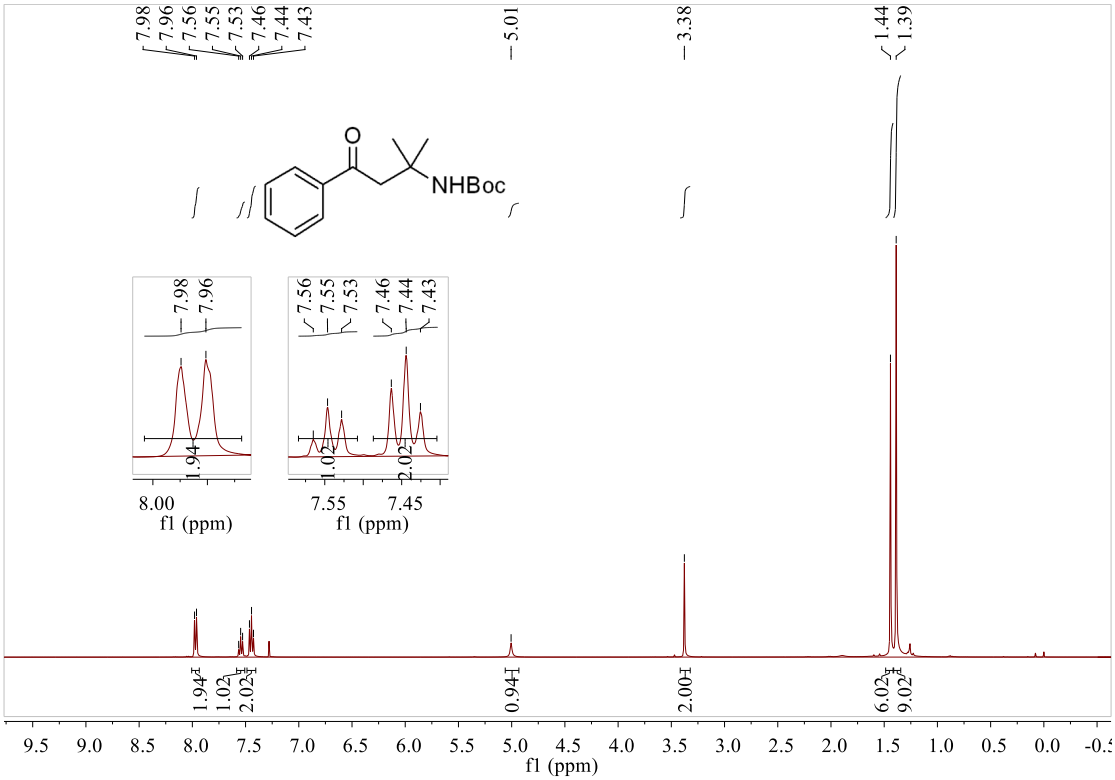
^{1}H NMR spectrum of ***tert*-butyl (1-(2-oxo-2-phenylethyl)cyclobutyl)carbamate (23)**



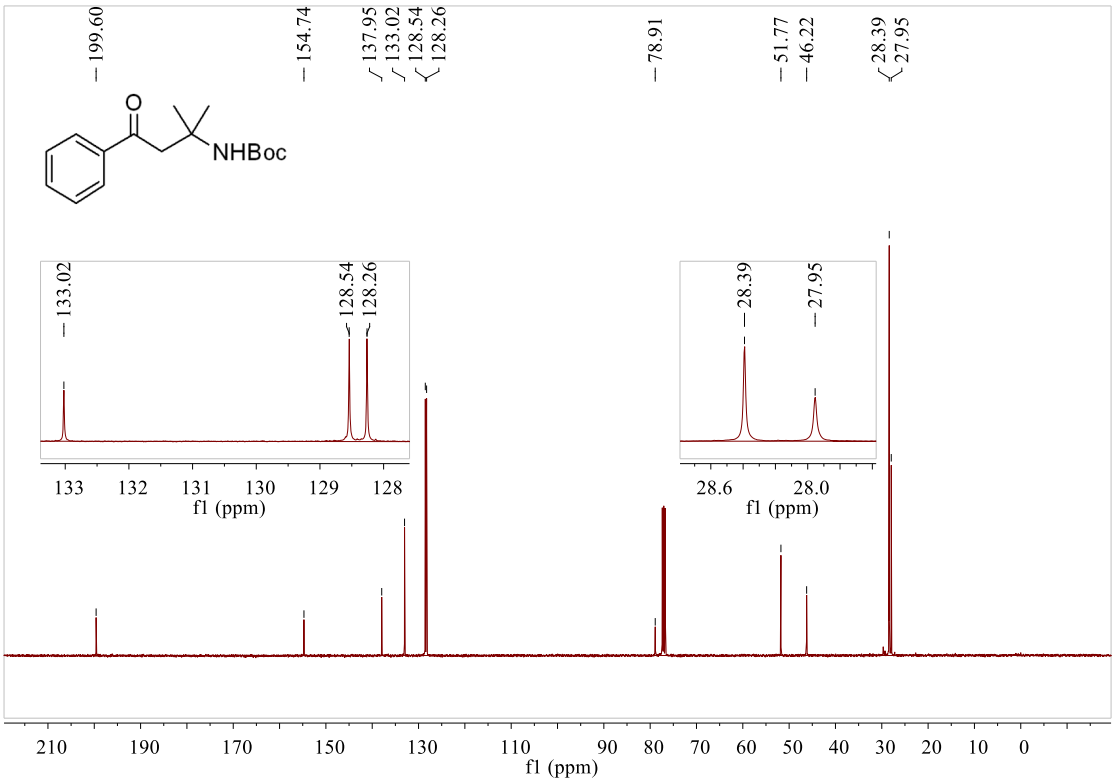
^{13}C NMR spectrum of ***tert*-butyl (1-(2-oxo-2-phenylethyl)cyclobutyl)carbamate (23)**



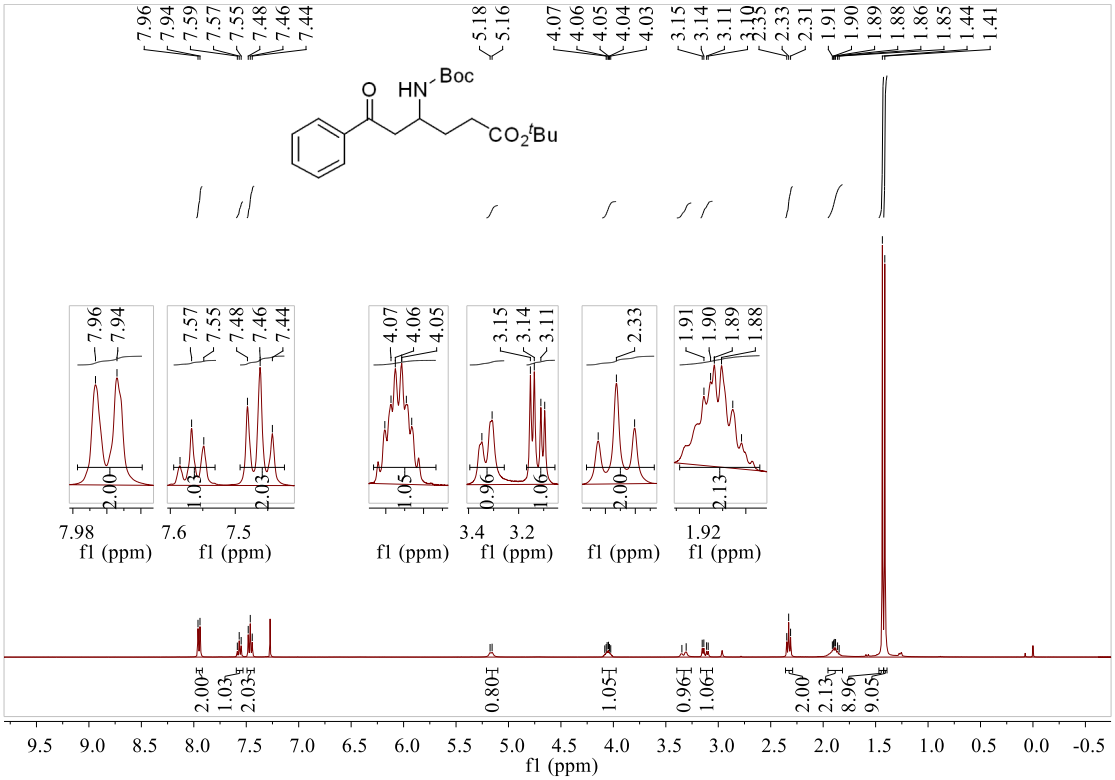
^{1}H NMR spectrum of ***tert*-butyl (2-methyl-4-oxo-4-phenylbutan-2-yl)carbamate (24)**



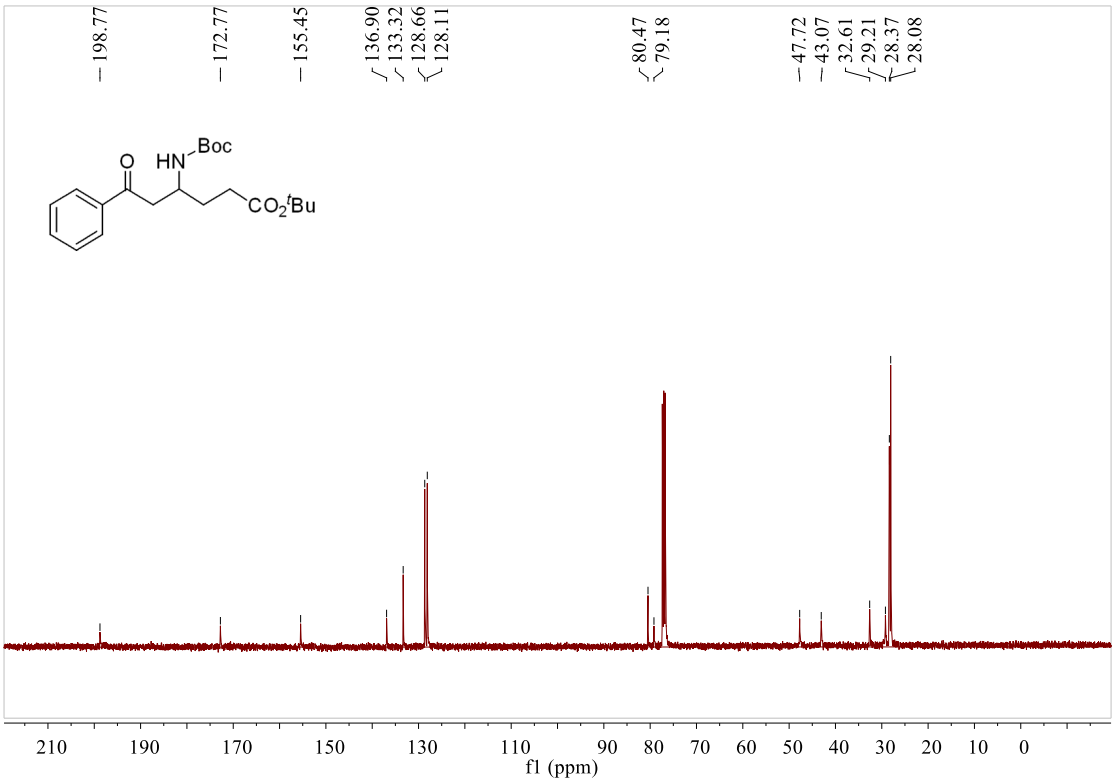
^{13}C NMR spectrum of ***tert*-butyl (2-methyl-4-oxo-4-phenylbutan-2-yl)carbamate (24)**



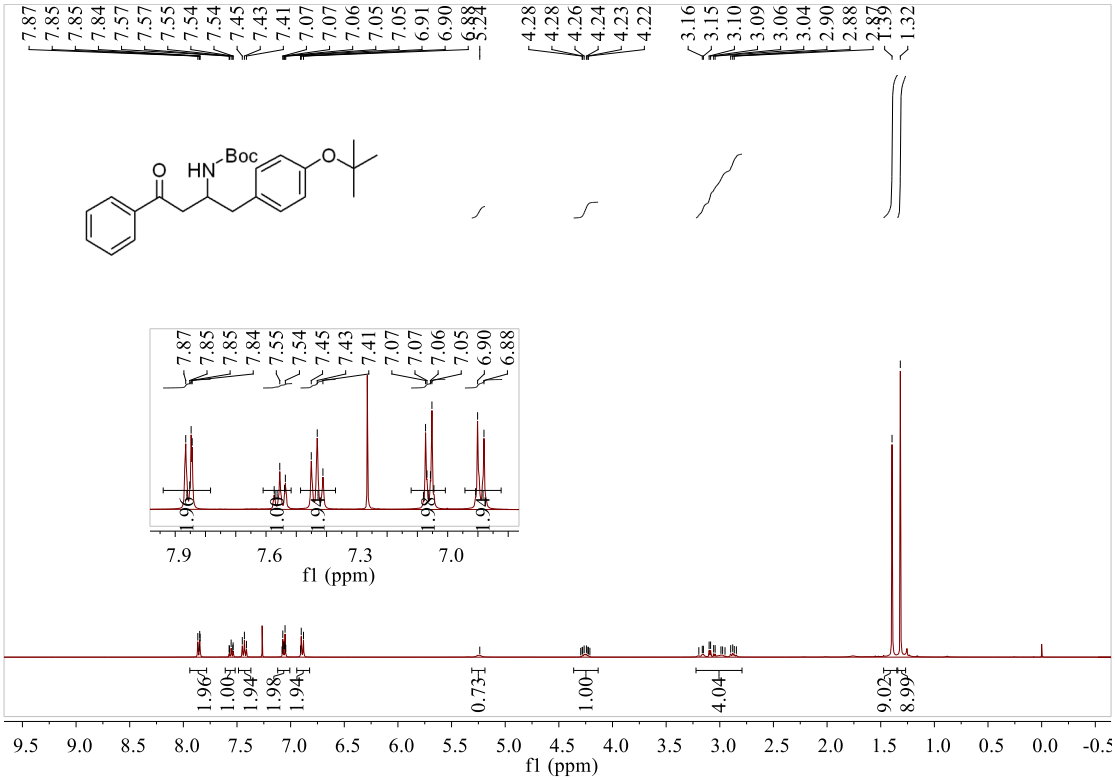
^{1}H NMR spectrum of ***tert*-butyl 4-((tert-butoxycarbonyl)amino)-6-oxo-6-phenylhexanoate (25)**



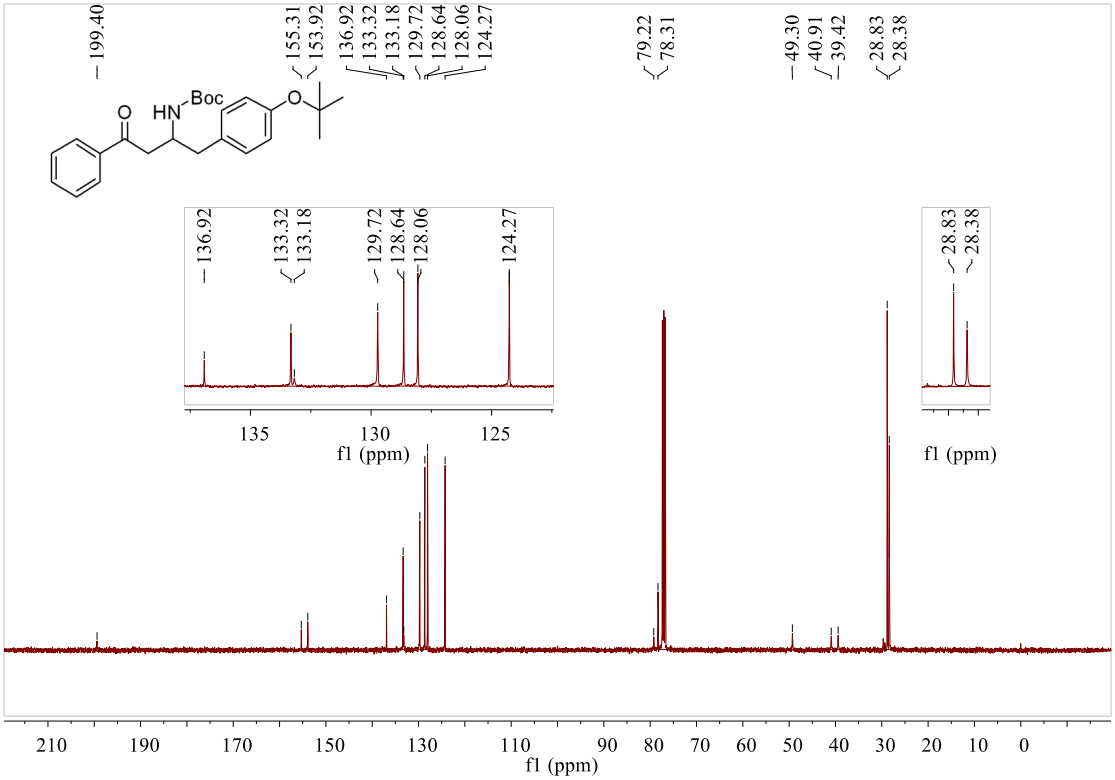
^{13}C NMR spectrum of ***tert*-butyl 4-((tert-butoxycarbonyl)amino)-6-oxo-6-phenylhexanoate (25)**



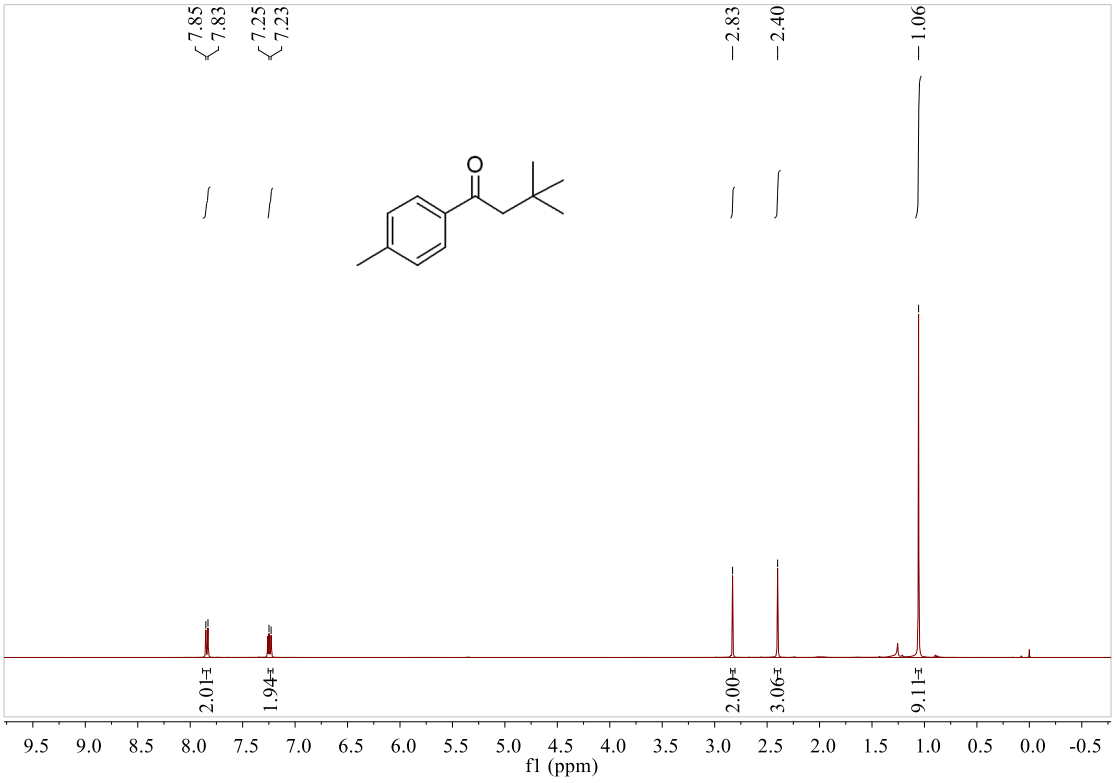
^{1}H NMR spectrum of ***tert*-butyl (1-(4-(tert-butoxy)phenyl)-4-oxo-4-phenylbutan-2-yl)carbamate (26)**



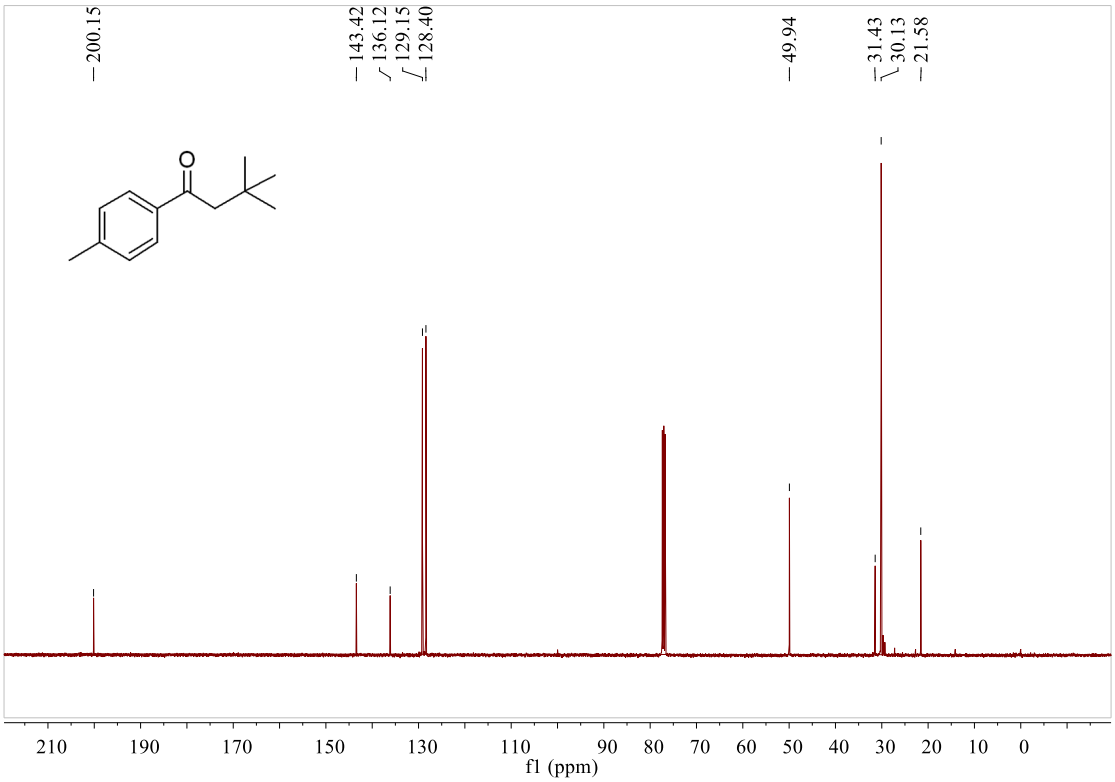
^{13}C NMR spectrum of ***tert*-butyl (1-(4-(tert-butoxy)phenyl)-4-oxo-4-phenylbutan-2-yl)carbamate (26)**



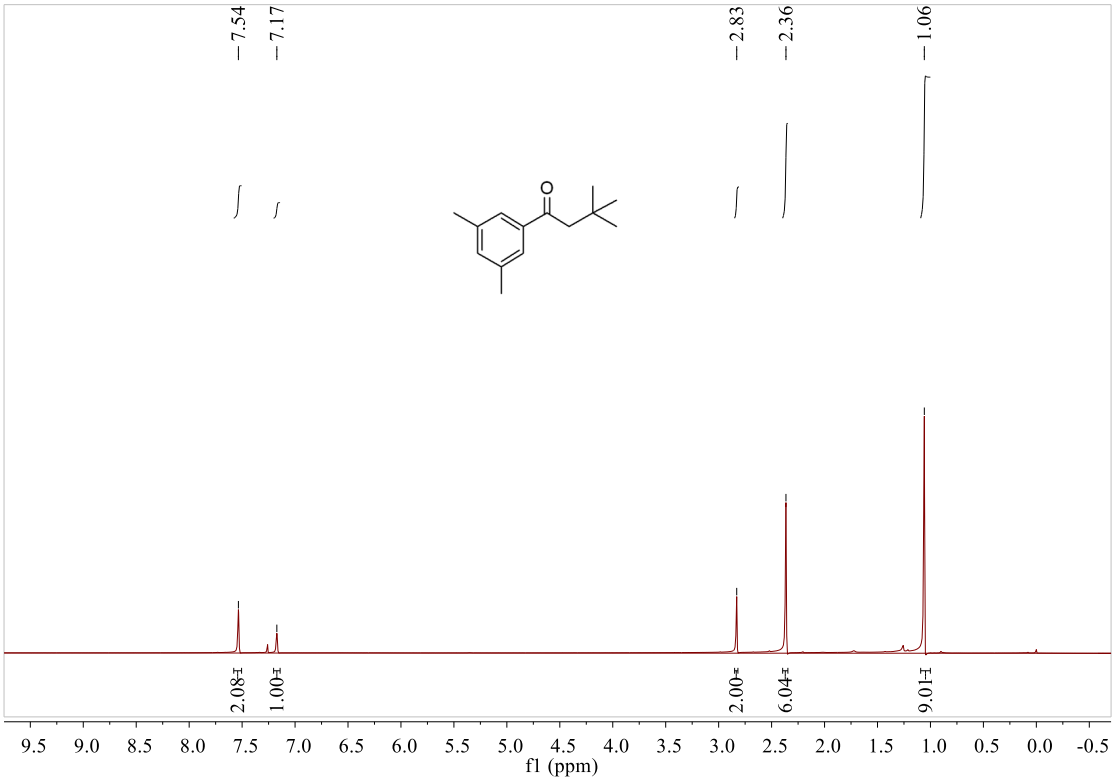
^{1}H NMR spectrum of **3,3-dimethyl-1-(p-tolyl)butan-1-one (27)**



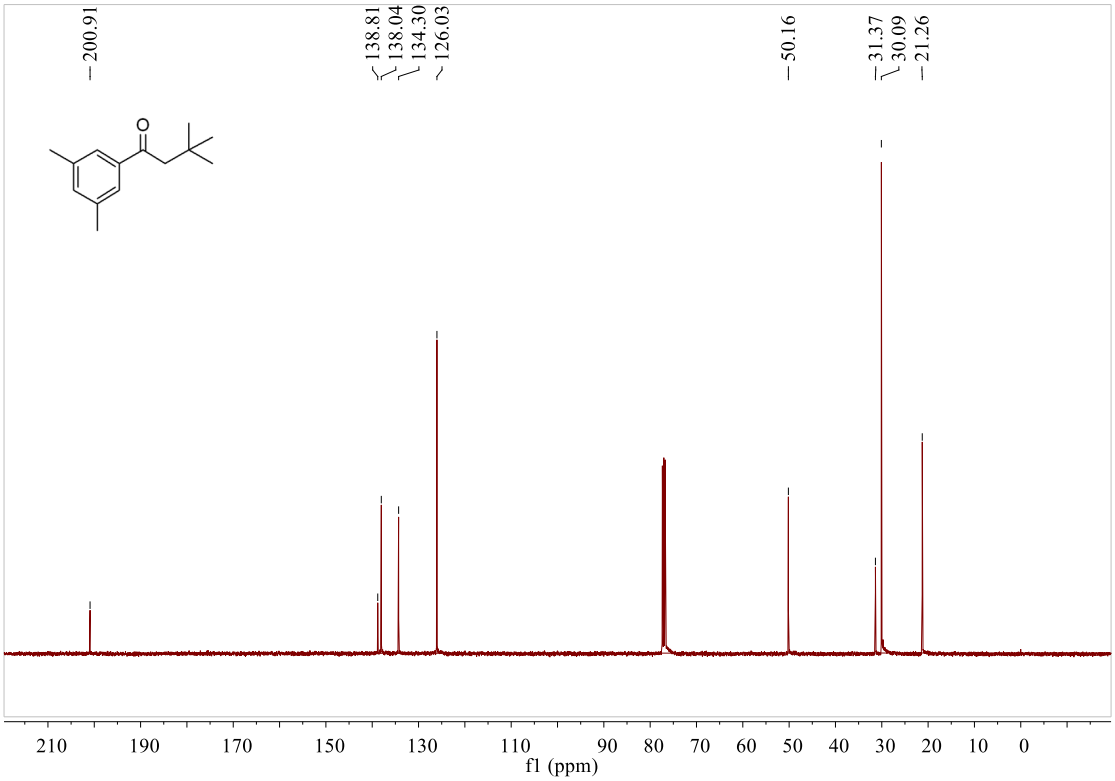
^{13}C NMR spectrum of **3,3-dimethyl-1-(p-tolyl)butan-1-one (27)**



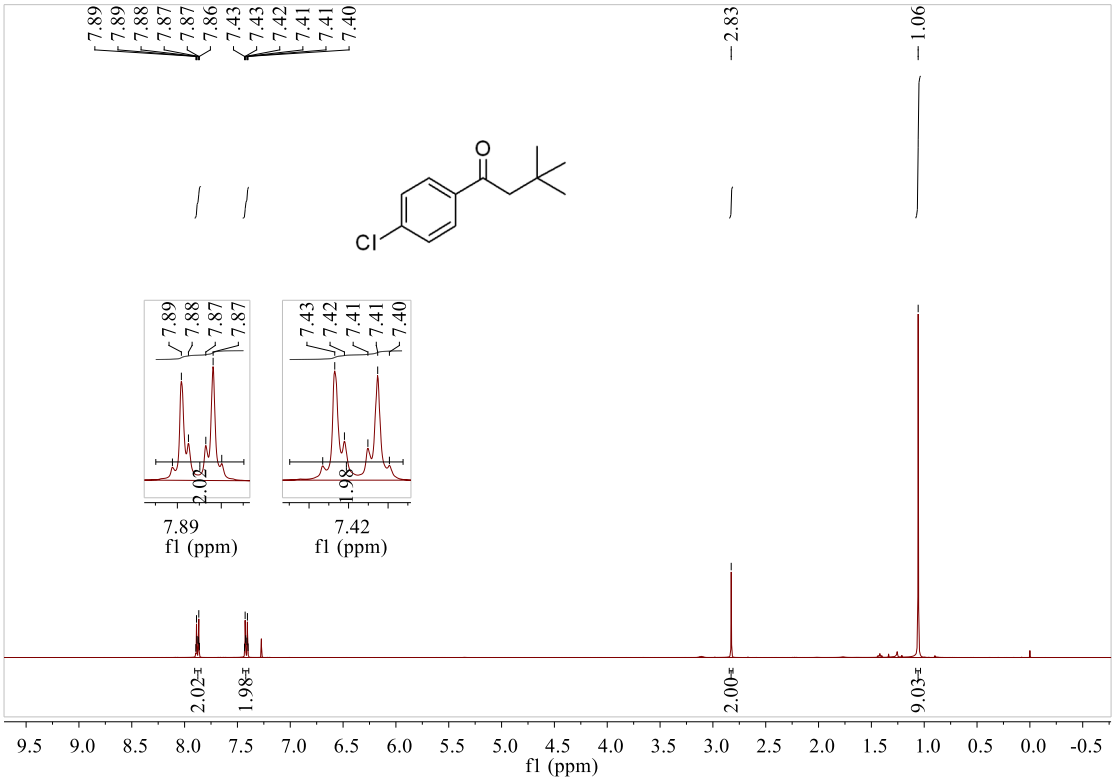
^{1}H NMR spectrum of **1-(3,5-dimethylphenyl)-3,3-dimethylbutan-1-one (28)**



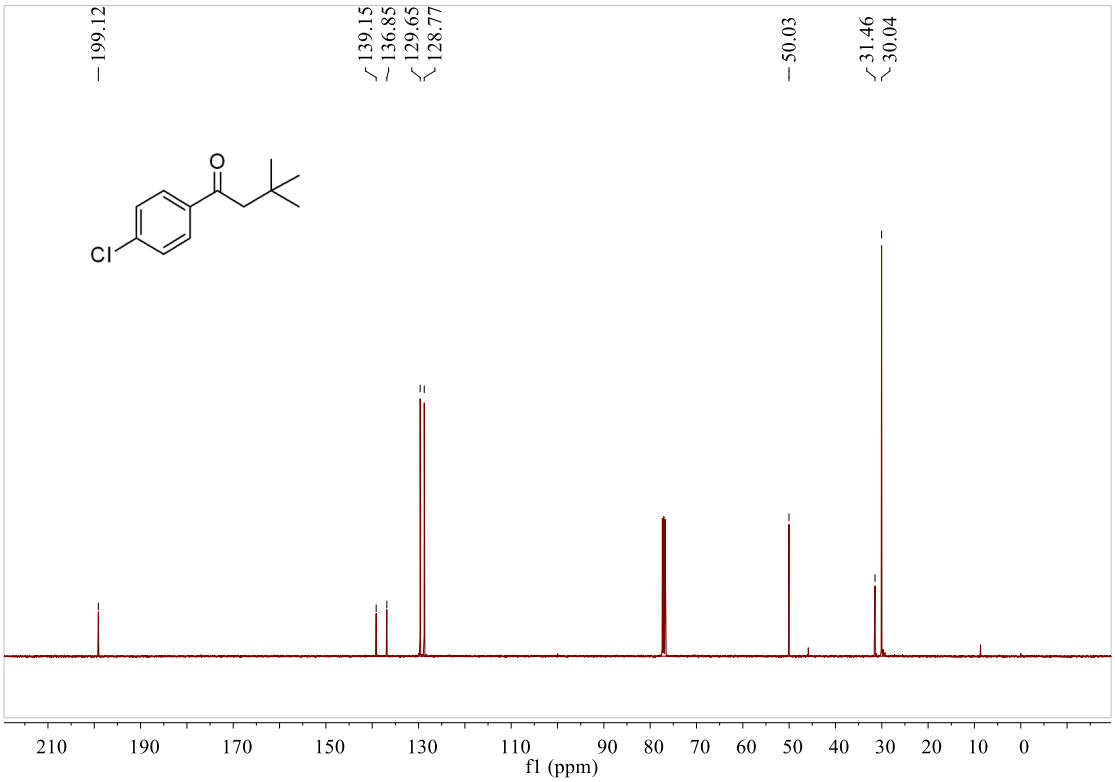
^{13}C NMR spectrum of **1-(3,5-dimethylphenyl)-3,3-dimethylbutan-1-one (28)**



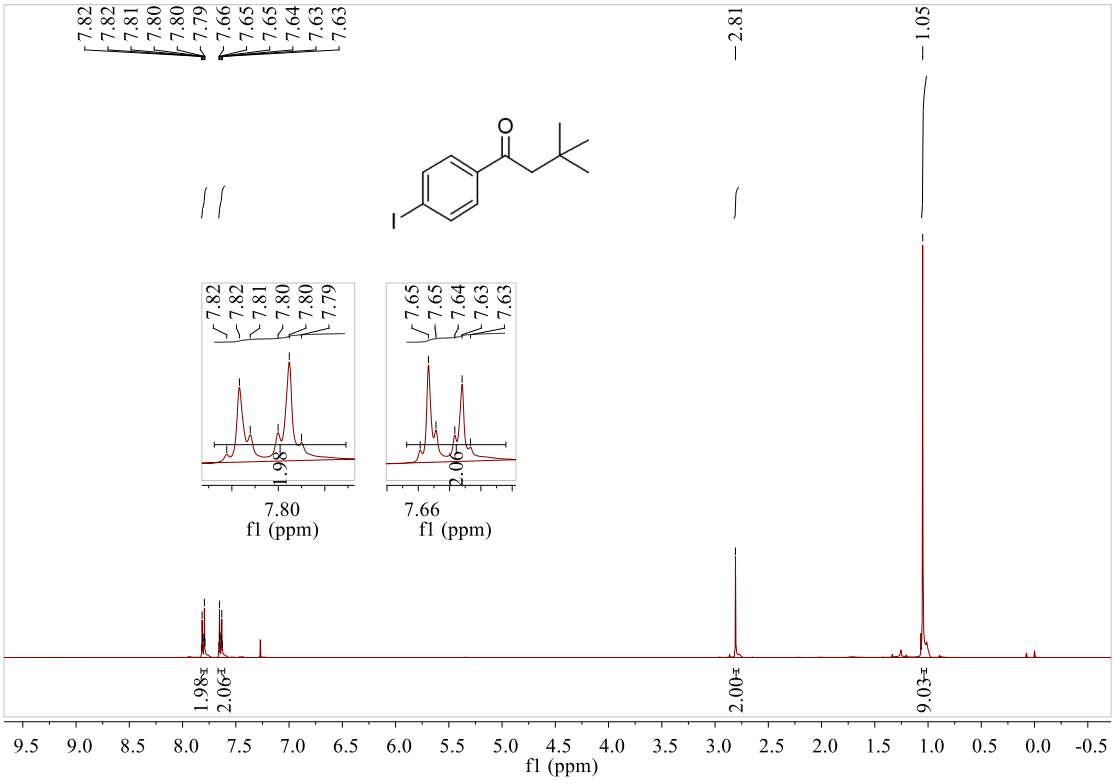
^{1}H NMR spectrum of **1-(4-chlorophenyl)-3,3-dimethylbutan-1-one (29)**



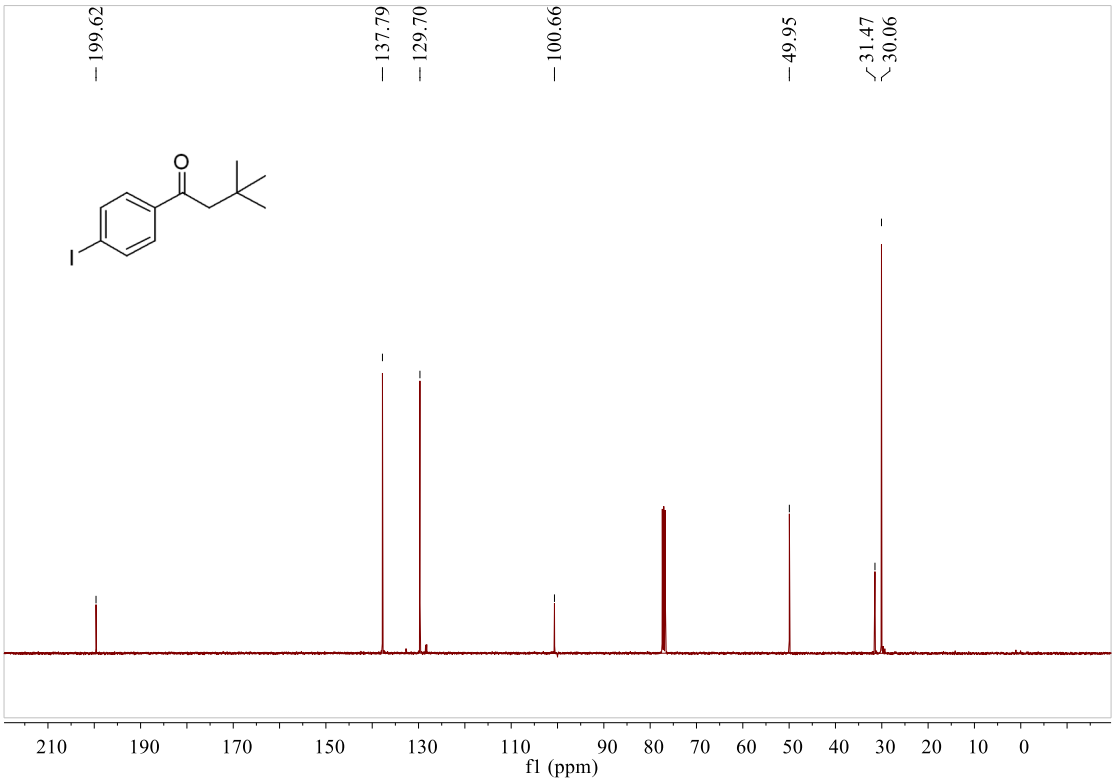
^{13}C NMR spectrum of **1-(4-chlorophenyl)-3,3-dimethylbutan-1-one (29)**



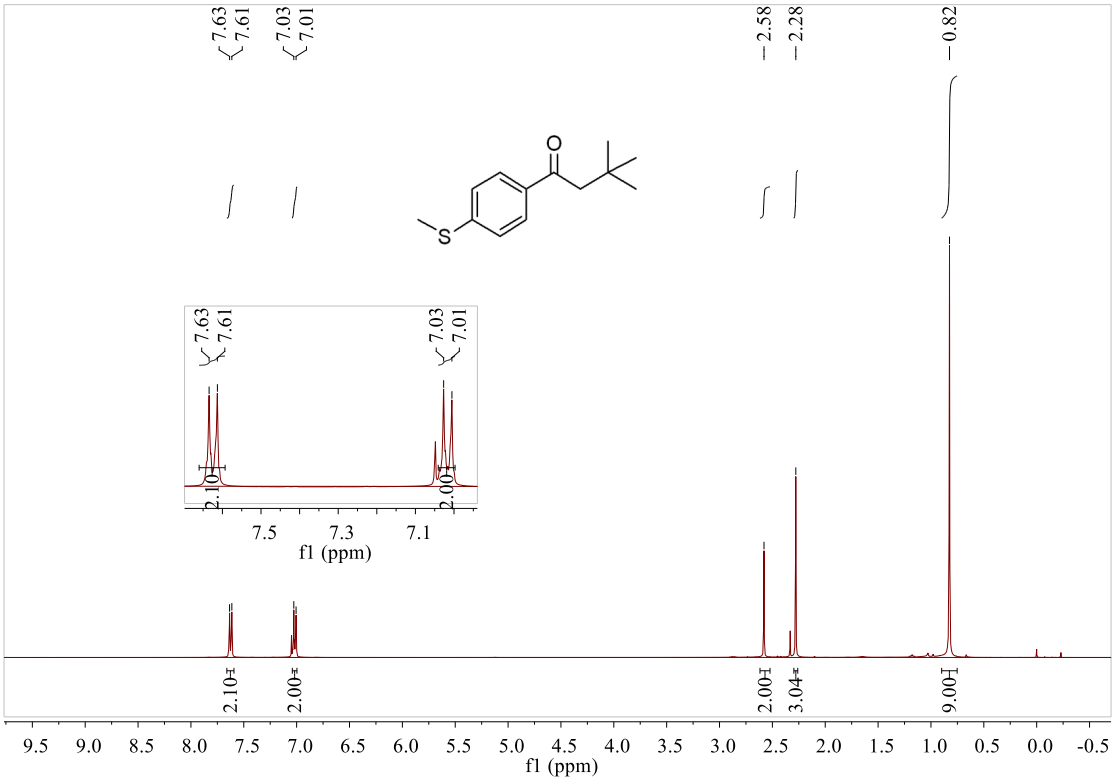
^{1}H NMR spectrum of **1-(4-iodophenyl)-3,3-dimethylbutan-1-one (30)**



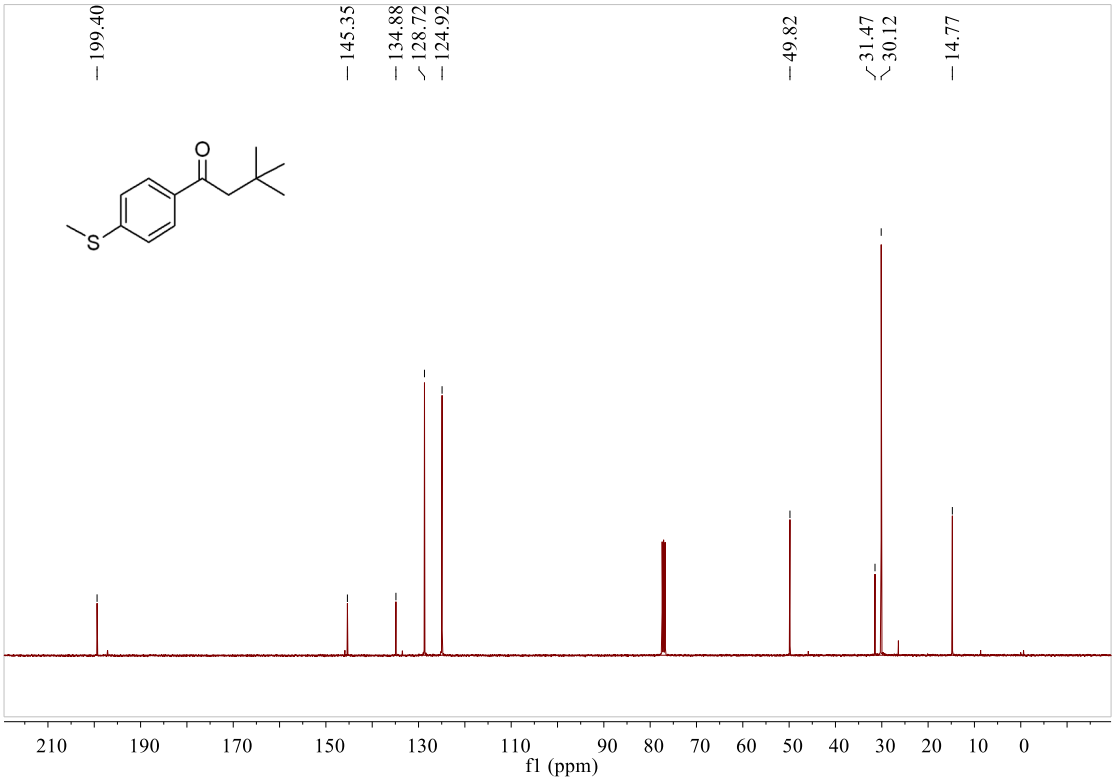
^{13}C NMR spectrum of **1-(4-iodophenyl)-3,3-dimethylbutan-1-one (30)**



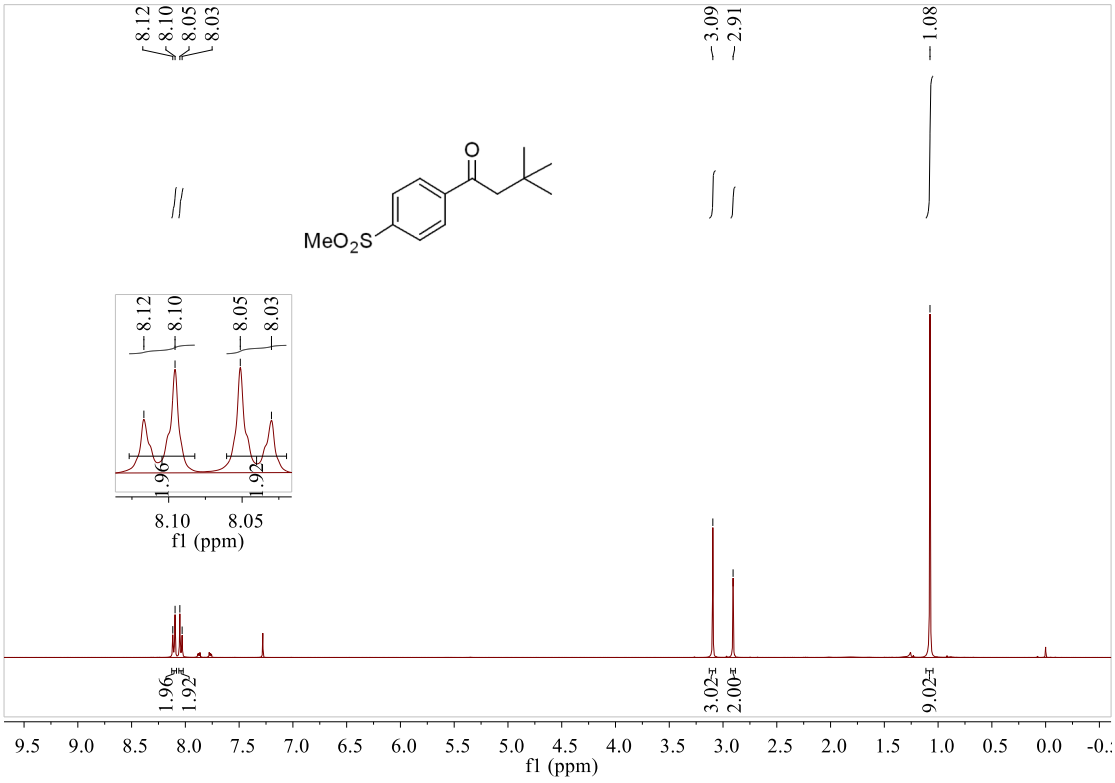
^{1}H NMR spectrum of **3,3-dimethyl-1-(4-(methylthio)phenyl)butan-1-one (31)**



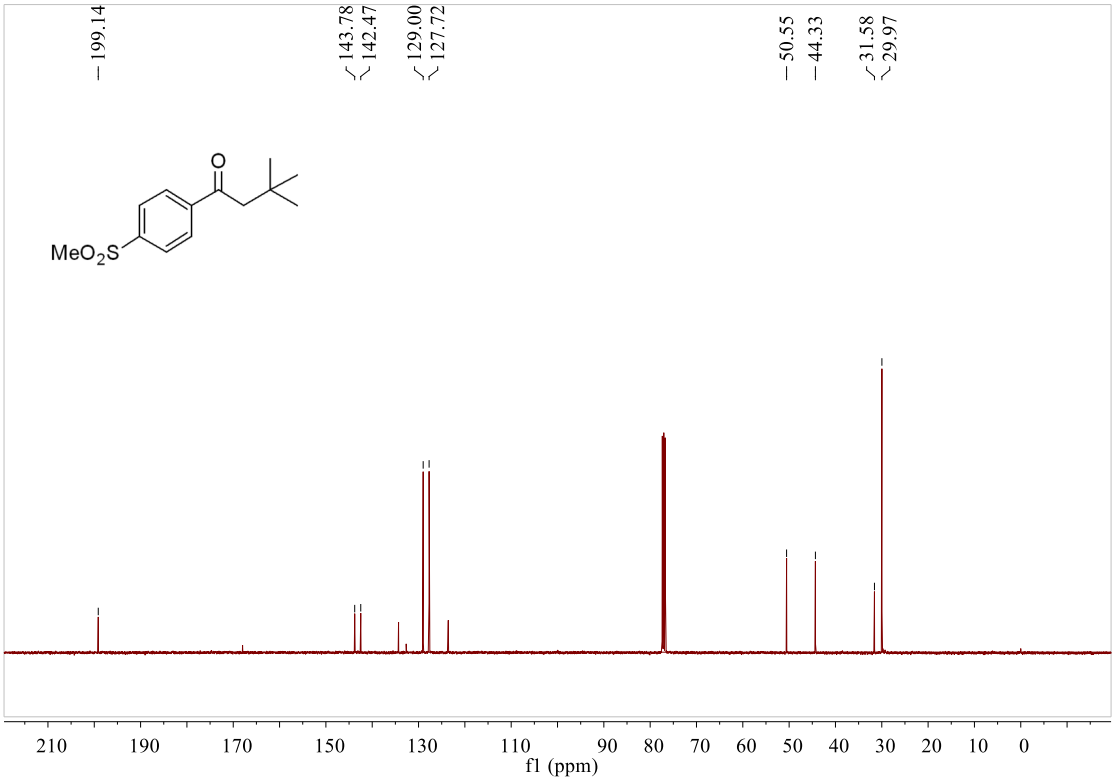
^{13}C NMR spectrum of **3,3-dimethyl-1-(4-(methylthio)phenyl)butan-1-one (31)**



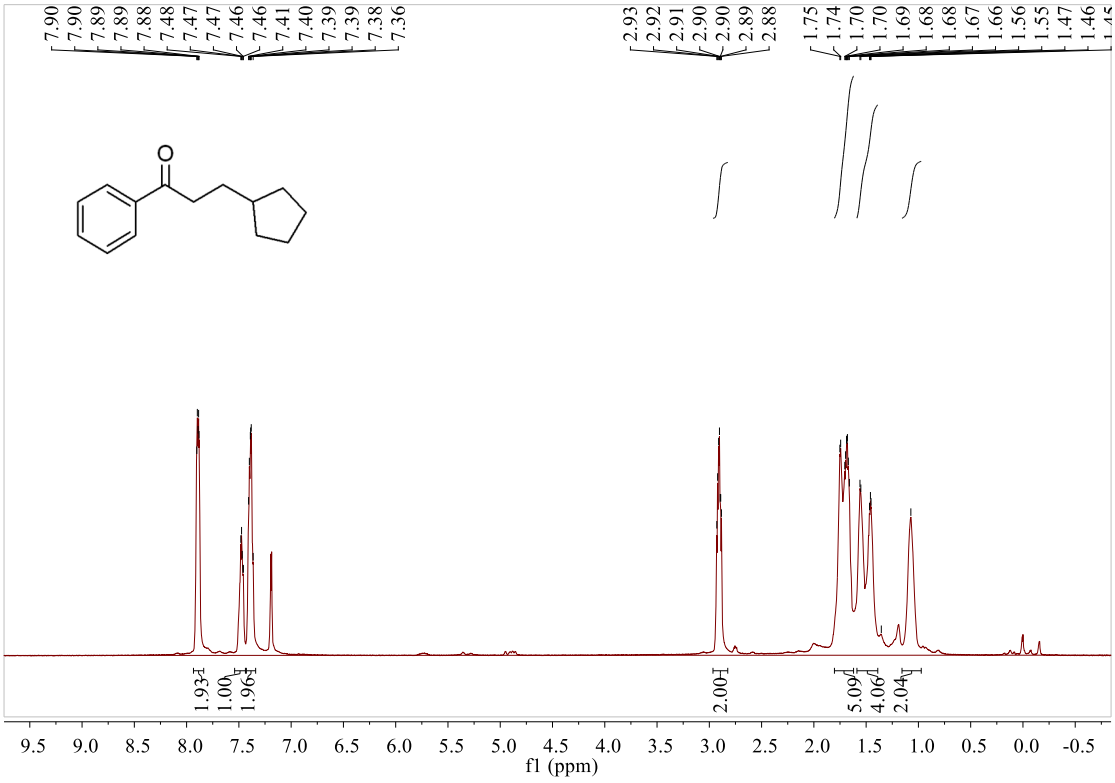
^{1}H NMR spectrum of **3,3-dimethyl-1-(4-(methylsulfonyl)phenyl)butan-1-one (32)**



^{13}C NMR spectrum of **3,3-dimethyl-1-(4-(methylsulfonyl)phenyl)butan-1-one (32)**



^{1}H NMR spectrum of **3-cyclopentyl-1-phenylpropan-1-one (33)**



^{13}C NMR spectrum of **3-cyclopentyl-1-phenylpropan-1-one (33)**