Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Ring-Opening/Annulation Reaction of Cyclopropyl Ethanols: Concise Access to Thiophene Aldehydes via C–S bond formation

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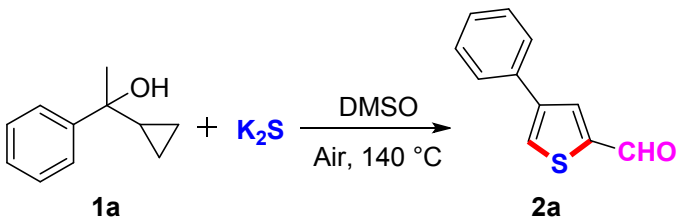
General remark

^{1}H NMR and ^{13}C NMR spectra of materials and products were respectively recorded on 300 MHz and 75 MHz (VARIAN 300 M), 400 MHz and 100 MHz (BRUKER 400 M or JNM-ECS 400 M) in $CDCl_3$. All chemical shifts were given as δ value (ppm) with reference to tetramethylsilane (TMS) as an internal standard. All compounds were further characterized by HRMS; copies of ^{1}H NMR and ^{13}C NMR spectra were provided. Products were purified by flash chromatography on 200-300 mesh silica gels. All melting points were determined without correction. All reactions were carried out under air in oven-dried glassware, unless otherwise noted. All reagents were purchased commercially and used as received, unless otherwise noted.

Screening Reaction Conditions

Initially, investigation commenced our study by examining the reaction of 1-cyclopropyl-1-phenylethanol (**1a**) and K_2S in DMSO at 120 °C for 8 h under Air atmosphere. To our delight, the desired product 5-phenylthiophene-2-carbaldehyde (**2a**) was observed in 41% yield (table S1, entry 1). The structure of **2a** was confirmed by single-crystal XRD analysis. Based on the result, several sulfur sources, such as elemental sulfur, $Na_2S{\cdot}9H_2O$, $Na_2S_2O_3{\cdot}5H_2O$, $K_2S_2O_3$, NH_4HS, and NaHS were screened for this reaction. The results demonstrated that potassium sulfide was the best sulfur source (entries 2-7). At this stage, to observe the effect of solvent on the yield of **2a,** DMSO, *N*,*N*-dimethylethylenediamine (DMF), toluene, acetonitrile, ethyl alcohol and dichloromethane (DCM) were tested for the reaction(entries 8-13). Interestingly, the **2a** was obtained in moderate yields when the reaction was carried out in DMSO, and no product of **2a** was detected in other solvents. Therefore, we conjectured daringly that DMSO could play a vital role in the sulfuration/annulation process. It is noteworthy that reaction temperatures are essential to the transformation, the yields were decreased drastically at lower temperature (entries 1 and 14). On the basis of all of the screening results, the optimal reaction conditions were **1a** (0.3 mmol) and K_2S (0.9 mmol) in DMSO (2 mL) at 140 °C for 8 h under air.

Table S1. Optimization of the Reaction Conditions [a]



1a **2a**

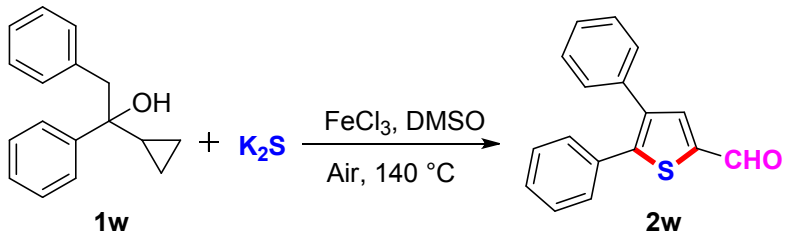
Entry	S reagent	Solvent	Temp. (°C)	Yield[b] (%)

1	K_2S	DMSO	120	41
2	S	DMSO	140	37
3	$Na_2S·9H_2O$	DMSO	140	27
4	$Na_2S_2O_3$□5H$_2$O	DMSO	140	32
5	$K_2S_2O_3$	DMSO	140	n.d.[c]
6	NH_4HS	DMSO	140	n.d.
7	NaHS	DMSO	140	trace
8	K_2S	DMF	140	n.d.
9	K_2S	THF	140	n.d.
10	K_2S	toluene	140	n.d.
11	K_2S	CH_3CN	140	n.d.
12	K_2S	EtOH	140	n.d.
13	K_2S	DCM	140	n.d.
14	K_2S	DMSO	100	n.d.
15	K_2S	DMSO	140	71
16[d]	K_2S	DMSO	140	n.d.
17[e]	K_2S	DMSO	140	67

[a]Reaction conditions: **1a** (0.3 mmol), K_2S (0.9 mmol) and DMSO (2 mL) under air at 140 °C for 8h. [b]Isolated yield. [c]n.d. = not detected. [d]Under Ar. [e]Under O_2.

Initially, investigation commenced our study by examining the reaction of 1-cyclopropyl-1,2-diphenylethanol (**1w**) and K_2S in DMSO at 140 °C for 10 h under Air atmosphere. To our delight, the desired product 4,5-diphenylthiophene-2-carbaldehyde (**2w**) was observed in 40% yield (table S2, entry 10). Based on the result, several sulfur sources, such as elemental sulfur, $Na_2S·9H_2O$, $Na_2S_2O_3 \cdot 5H_2O$, $K_2S_2O_3$, NH_4HS, and NaHS were screened for this reaction. The results demonstrated that potassium sulfide was the best sulfur source (entries 2-7). At this stage, to observe the effect of additive on the yield of **2w**, $FeCl_3$, $CuCl_2$, $ZnCl_2$, $FeCl_2$, and $Fe(acac)_3$ were tested for the reaction (entries 8-11). Interestingly, the **2w** was obtained in higher yields when the $FeCl_3$ was used as additive. Subsequently, we further screened the amount of $FeCl_3$. As a result, 1.0 equiv of $FeCl_3$ showed the highest catalytic activity, and the desired product **2w** was obtained in 68% yield (entries 1 and 12-15). On the basis of all of the screening results, the optimal reaction conditions were 1a (0.3 mmol), K_2S (0.9 mmol), and $FeCl_3$ (0.3 mmol) in DMSO (2 mL) at 140 °C for 10 h under air.

Table S2. Optimization of the Reaction Conditions [a]



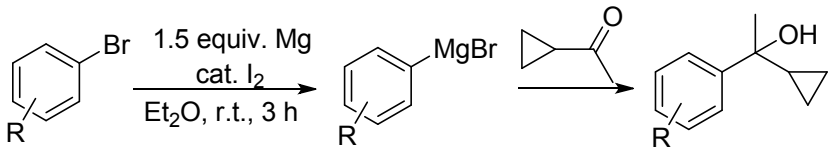
1w 2w

Entry	S reagent	Additive	Equiv ($FeCl_3$)	Yield[b] (%)
1	K_2S	$FeCl_3$	1.0	68
2	S	$FeCl_3$	1.0	40
3	$Na_2S·9H_2O$	$FeCl_3$	1.0	35
4	$Na_2S_2O_3$□$5H_2O$	$FeCl_3$	1.0	30
5	$K_2S_2O_3$	$FeCl_3$	1.0	n.d.[c]
6	NH_4HS	$FeCl_3$	1.0	n.d.
7	NaHS	$FeCl_3$	1.0	trace
8	K_2S	$CuCl_2$	1.0	60
9	K_2S	$ZnCl_2$	1.0	31
10	K_2S	$FeCl_2$	1.0	42
11	K_2S	$Fe(acac)_3$	1.0	49
12	K_2S	$FeCl_3$	0	40
13	K_2S	$FeCl_3$	0.1	55
14	K_2S	$FeCl_3$	0.5	60
15	K_2S	$FeCl_3$	2.0	66

[a]Reaction conditions: **1a** (0.3 mmol), K_2S (0.9 mmol), $FeCl_3$ (0.3 mmol), DMSO (2 mL) under air at 140 °C for 10 h. [b]Isolated yield. [c]n.d. = not detected.

Experimental Procedures

General procedure for the synthesis of cyclopropyl ethanols 1a-1v:[1]

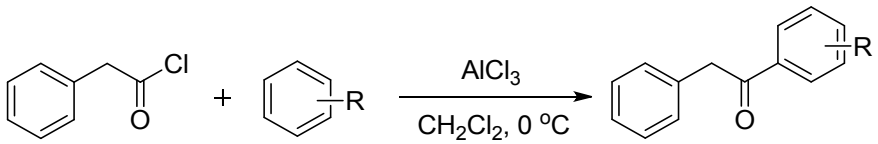


Grignard reactions were performed with thoroughly dried glass apparatus. To a magnetically stirred mixture of Mg turnings (7.5 mmol, 180 mg), I_2 (10 mg) was added and a solution of the bromobenzene derivative (6 mmol) in dry Et_2O (10 mL) under N_2 atmosphere dropwise during 10 min and allowed to stir at room temperature for 3 h. Then temperature was decreased to 0 °C and cyclopropyl methyl ketone (5 mmol, 0.465 mL) was added and allowed to stir at room temperature for 2 h. After completion of the reaction, the mixture was treated with saturated NH_4Cl aq. (5 mL) and the reaction mixture was extracted with ethyl acetate (3 x 5 mL). The organic layer was dried

over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography using hexane and ethyl acetate (40:1) as eluent.

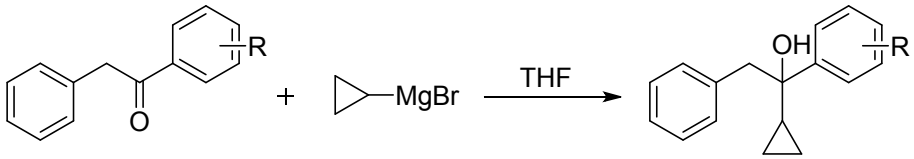
General procedure for the synthesis of substituted 1-cyclopropyl-1,2-diphenyle -thanols 1w-1z, 1aa,1ab:[2,3]

Step 1:



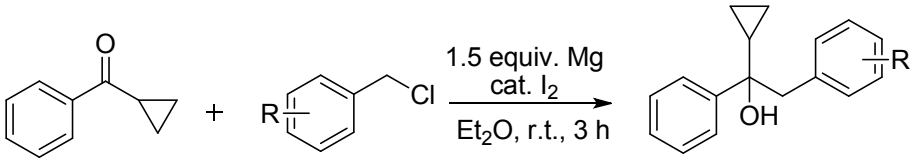
Arylacetyl chloride (10 mmol, 1.54 g) was stirred with the appropriate substituted aromatic compound (13 mmol) in methylene chloride (20 mL) at 0 °C. Anhydrous $AlCl_3$ (13 mmol, 1.73 g) was added slowly portion wise, and the reaction mixture was stirred at room temperature until arylacetyl chlorid was completely consumed (monitored by TLC). The reaction mixture was then poured onto ice-water mixture. The organic layer was separated, washed with brine, and dried over anhydrous Na_2SO_4, filtered, and concentrated in vacuo. The crude product was purified by silica gel column chromatography to give the deoxybenzoins.

Step 2:



To a solution of cyclopropylmagnesium bromide (1.0 M THF solution; 6 mL, 6 mmol) in THF (10 mL) at 0 °C was added dropwise a solution of deoxybenzoins (5 mmol) in THF (5 mL). The resulting mixture was stirred at room temperature for 15 h. The mixture was treated with saturated NH_4Cl aq (10 mL). The organic layer was extracted with Et_2O. The combined organic layers were dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude product was purified by column chromatography using hexane and ethyl acetate (40:1) as eluent.

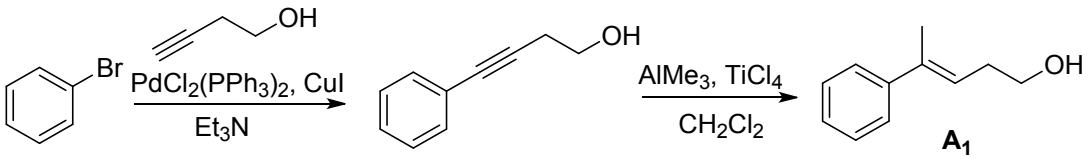
General procedure for the synthesis of substituted 1-cyclopropyl-1,2-diphenyle-thanols 1ac-1ak:[1]



Grignard reactions were performed with thoroughly dried glass apparatus. To a magnetically stirred mixture of Mg turnings (7.5 mmol, 180 mg), I_2 (10 mg) was added and a solution of the benzyl chloride derivative (6 mmol) in dry Et_2O (10 mL) under N_2 atmosphere dropwise during 10

min and allowed to stir at room temperature for 3 h. Then temperature was decreased to 0 °C and cyclopropyl phenyl ketone (5 mmol, 0.73 g) was added and allowed to stir at room temperature for 2 h. After completion of the reaction, the mixture was treated with saturated NH_4Cl aq. (5 mL) and the reaction mixture was extracted with ethyl acetate (3 x 5 mL). The organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by column chromatography using hexane and ethyl acetate (40:1) as eluent.

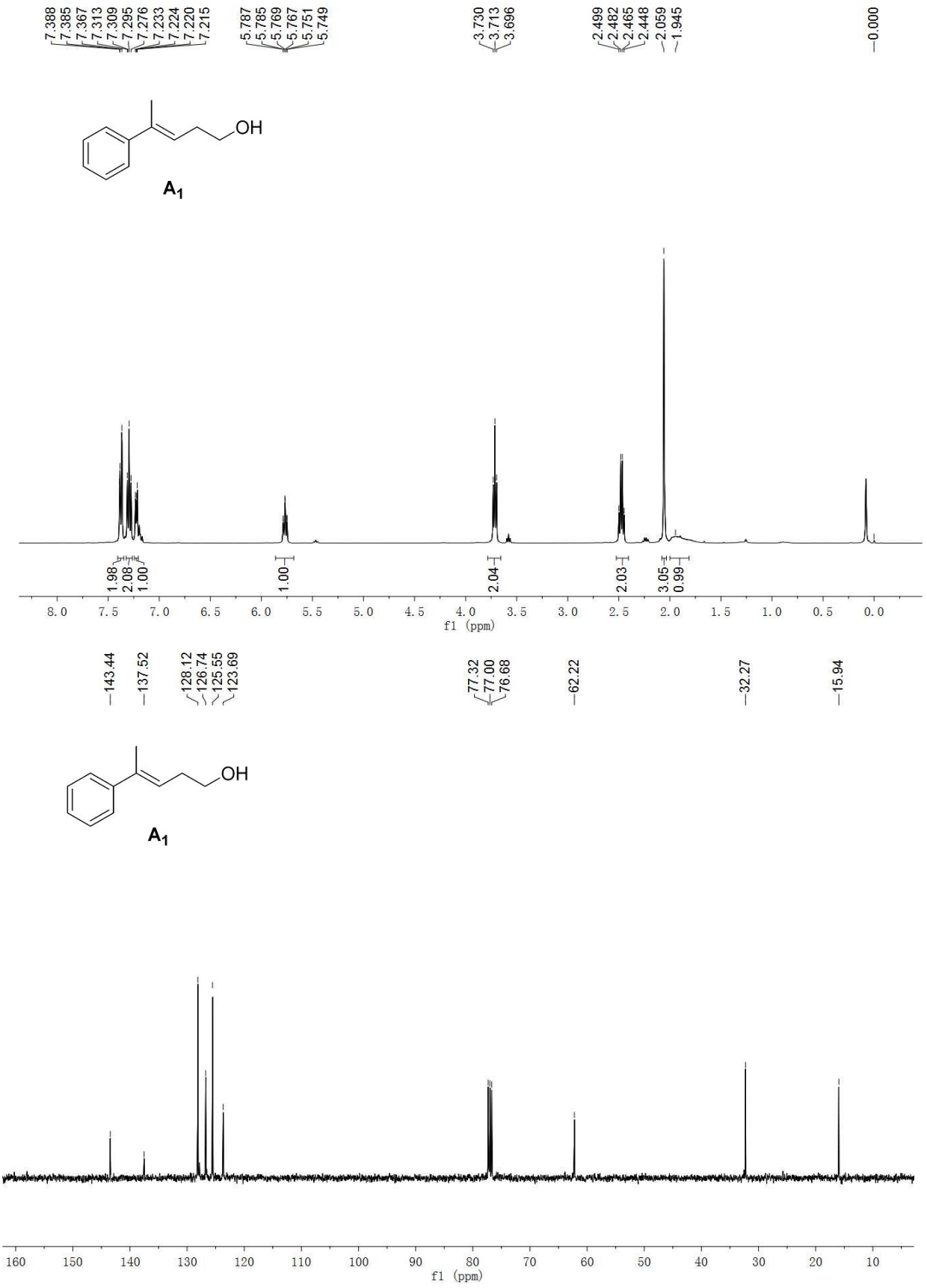
General procedure for the synthesis of (*E*)-homoallylic alcohol:[4]



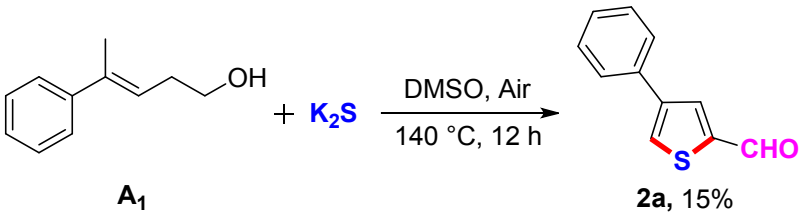
To a solution of aryl bromide (10 mmol) in Et_3N (40 mL) were added $PdCl_2(PPh_3)_2$ (2 mol%, 0.140 g, 0.2 mmol), and CuI (1 mol%, 0.019 g, 0.1 mmol) under N_2 atmosphere. After the reaction mixture was stirred for 5 min. at room temperature, but-3-yn-1-ol (1.2 equiv, 0.91 mL, 12 mmol) was added by a syringe. The reaction mixture was then heated to 60℃ and stirred overnight. The resulting mixture was filtered through a short column of silica gel and concentrated in vacuo. The obtained crude product was purified by column chromatography on silica (Hexanes/EtOAc = 5:1) to give the corresponding 4-arylbut-3-yn-1-ol.

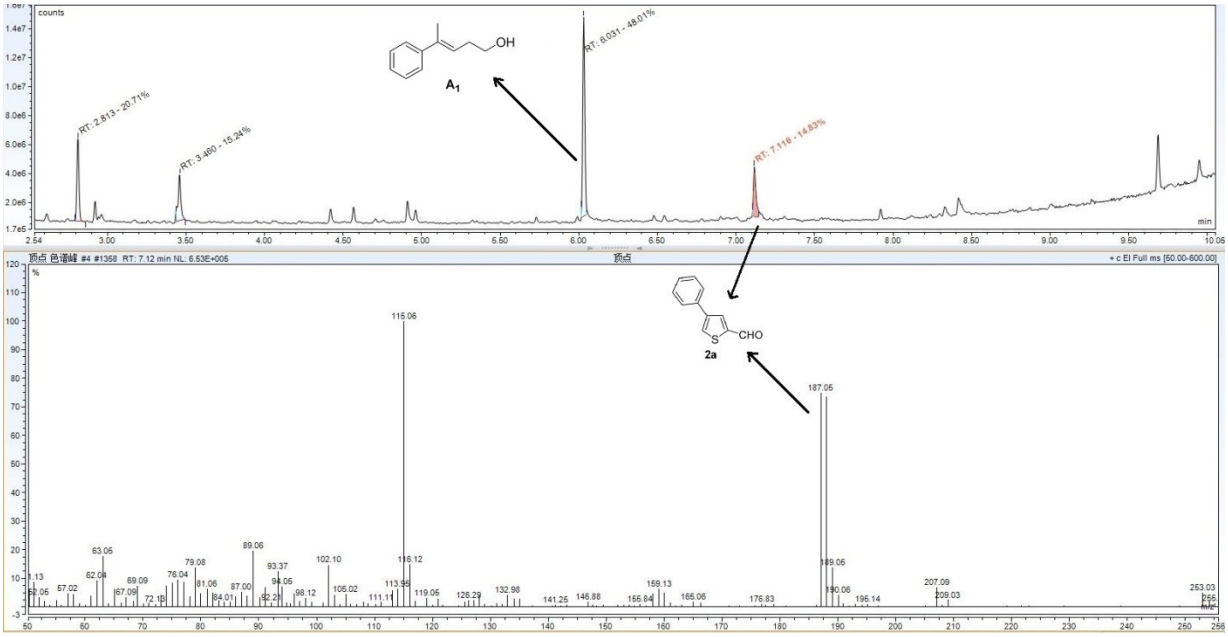
To a cooled (0 ℃) solution of $AlMe_3$ (2 equiv, 5.0 mL, 2.0 M solution in toluene, 10 mmol) in DCM (10 mL) was added 4-arylbut-3-yn-1-ol (5.0 mmol) dropwise at 0°C and the mixture was stirred at the same temperature for 20 min. The reaction mixture was then cooled to –45 °C followed by the slow addition of a cooled (0 ℃) solution of $TiCl_4$ (1 equiv, 0.55 mL, 5.0 mmol) in DCM (15 mL). The resulting mixture was allowed to stir for 4 h at －45 ℃ and then quenched by the slow addition of cooled (0 ℃) MeOH (4.0 mL). 3 M HCl aq. saturated with NaCl (10 mL) was then added and the mixture was stirred at room temperature for 30 min. The resulting solution was extracted with Et_2O (3 × 25 mL), dried over Na_2SO_4 and concentrated in vacuo. The obtained crude product was purified by column chromatography on silica gel (hexanes/EtOAc = 10/1-5:1) to give the desired (Z)-homoallylic alcohol.

Colourless oil (0.68 g, 84% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.39-7.37 (m, 2 H), 7.31-7.28 (m, 2 H), 7.23-7.22 (m, 1 H), 5.79-5.75 (m, 1 H), 3.73-3.70 (m, 2 H), 2.50-2.45 (m, 2 H), 2.06 (s, 3 H), 1.95 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 143.4, 137.5, 128.1, 126.7, 125.6, 123.7, 62.2, 32.3, 15.9.



Reaction of (*E*)-homoallylic alcohol (A_1) and 4-phenylthiophene-2-carbaldehyde (2a) detected by GC-MS analysis.





X-Ray Crystal Data of Compound 2p:

Crystal **2p** Growth with the Volatilization Method: An amount of 20 mg **2p** was dissolved in acetone/PE (1:1) on the brown small reagent bottle (5 mL), which acted as good solvent, and a layer of ether was injected on the surface of acetone, and the cap is covered with a thin film, white crystals will be presented after seven days.

Single crystals of $C_{11}H_9OS$ (**2p**) was determinate. A suitable crystal was selected and determinate on a SuperNova, Dual, Cu at zero, Eos diffractometer. The crystal was kept at 294.51 (13) K during data collection. Using Olex2,[5] the structure was solved with the ShelXS[6] structure solution program using Direct Methods and refined with the ShelXL[7] refinement package using Least Squares minimisation.

All hydrogen atoms were placed by geometrical considerations and were added to the structure factor calculations. The crystal structure (excluding structure factor) has been deposited to Cambridge Crystallographic Data Centre and allocated deposition number: **2p**: CCDC 1907301.

X-Ray Data of Compound 2p. The ellipsoids are shown at 30% probability

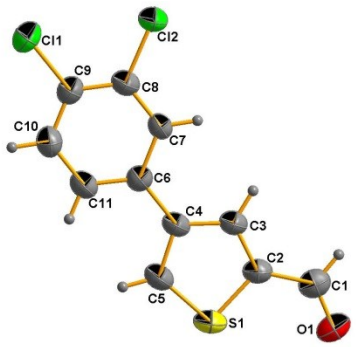


Figure S1. X-Ray Crystal structure of 2p.

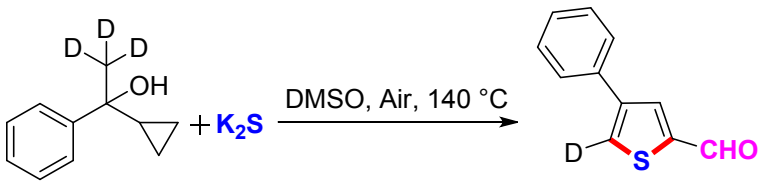
Table S3. **Crystal data and structure refinement for 2p.**

Identification code	**2p**
Empirical formula	$C_{11}H_6Cl_2OS$
Formula weight	257.12
Temperature/K	293(2)
Crystal system	orthorhombic
Space group	Pnma
a/Å	11.6812(11)
b/Å	6.6839(10)
c/Å	13.6312(10)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	1064.3(2)
Z	4
ρ_{calc}g/cm^3	1.605

μ/mm^{-1}	0.771
F(000)	520.0
Crystal size/mm^3	0.17 × 0.14 × 0.12
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	6.78 to 52.04
Index ranges	-11 ≤ h ≤ 14, -8 ≤ k ≤ 4, -9 ≤ l ≤ 16
Reflections collected	2354
Independent reflections	1140 [R_{int} = 0.0308, R_{sigma} = 0.0509]
Data/restraints/parameters	1140/0/91
Goodness-of-fit on F^2	1.053
Final R indexes [I>=2σ (I)]	R_1 = 0.0459, wR_2 = 0.1039
Final R indexes [all data]	R_1 = 0.0773, wR_2 = 0.1271
Largest diff. peak/hole / e Å^{-3}	0.38/-0.26

Mechanistic Studies

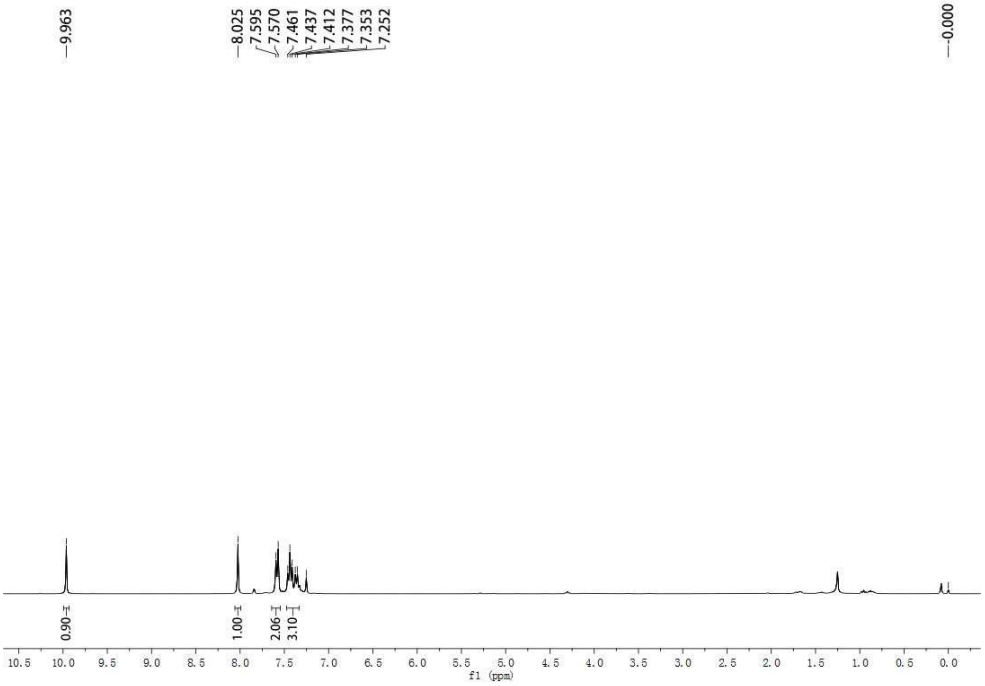
(a) The Isotope Experiment 1:



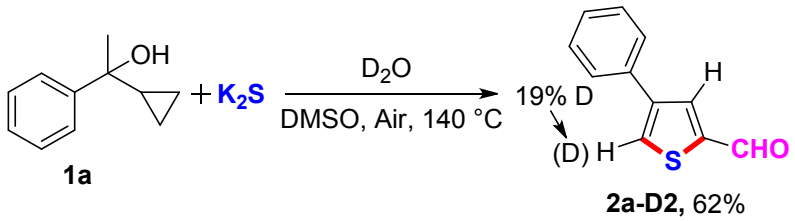
1a-D1 **2a-D1,** 68%

To a schlenk tube were added 1-cyclopropyl-1-phenylethanol **1a-D1** (0.3 mmol), K_2S (3.0 equiv), and DMSO (2 mL). Then the mixture was stirred at 140 °C (oil bath temperature) for 8 h under air. After the reaction was finished, the product **2a-D1** was isolated by silica gel column chromatography (petroleum ether), and ^{1}H NMR spectra were recorded in $CDCl_3$.

^{1}H NMR ($CDCl_3$, 400 MHz) δ = 9.96 (s, 0.90 H), 8.03 (s, 1 H), 7.59-7.57 (d, *J* = 8 Hz, 2 H), 7.46-7.35 (m, 3 H).

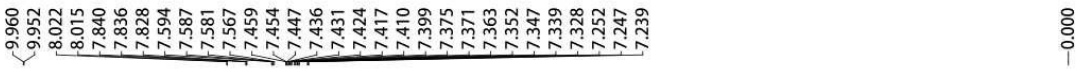


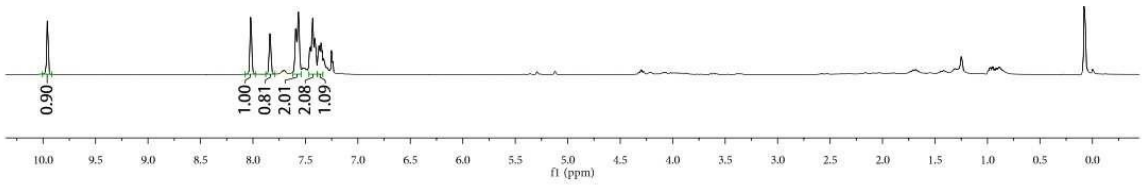
(b) The Isotope Experiment 2:



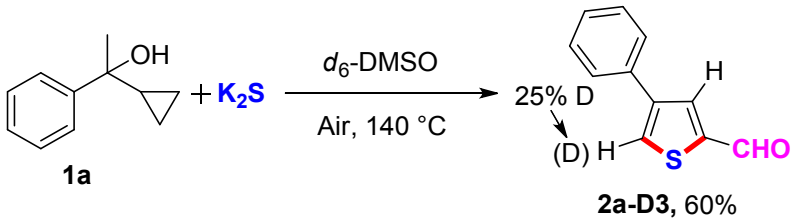
To a schlenk tube were added 1-cyclopropyl-1-phenylethanol **1a** (0.3 mmol), K_2S (3.0 equiv), D_2O (3.0 equiv), and DMSO (2 mL). Then the mixture was stirred at 140 °C (oil bath temperature) for 8 h under air. After the reaction was finished, the product **2a-D2** was isolated by silica gel column chromatography (petroleum ether), and 1H NMR spectra were recorded in $CDCl_3$.

^{1}H NMR ($CDCl_3$, 400 MHz) δ = 9.96-9.95 (d, *J* = 4 Hz, 0.9 H), 8.02-8.01 (d, *J* = 4 Hz, 1 H), 7.84-7.83 (d, *J* = 4 Hz, 0.81 H), 7.59-7.57 (m, 2 H), 7.46-7.40 (m, 2 H), 7.38-7.33 (s, 1 H).



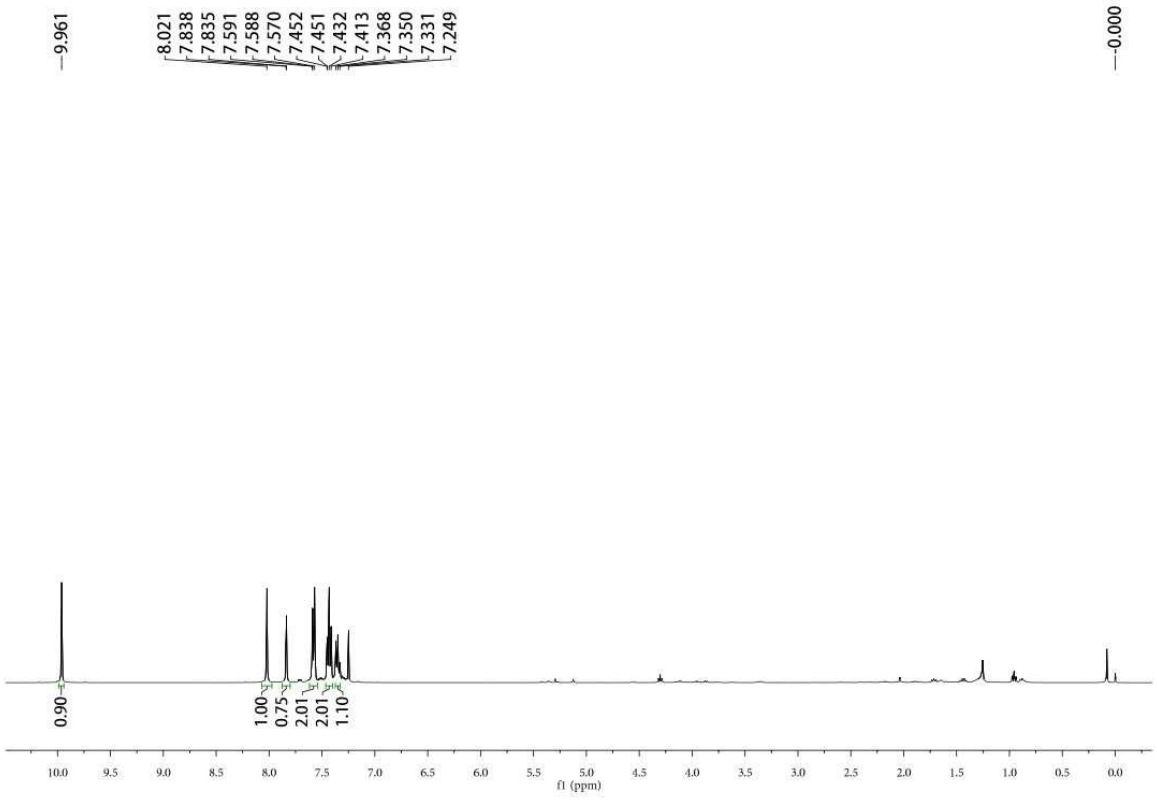


(c) The Isotope Experiment 3:

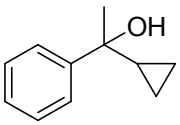


To a schlenk tube were added 1-cyclopropyl-1-phenylethanol **1a** (0.3 mmol), K_2S (3.0 equiv), and d_6-DMSO (2 mL). Then the mixture was stirred at 140 °C (oil bath temperature) for 8 h under air. After the reaction was finished, the product **2a-D3** was isolated by silica gel column chromatography (petroleum ether), and 1H NMR spectra were recorded in $CDCl_3$.

^{1}H NMR ($CDCl_3$, 400 MHz) δ = 9.96 (s, 0.9 H), 8.02 (s, 1 H), 7.84-7.83 (d, J = 4 Hz, 0.75 H), 7.59-7.57 (m, 2 H), 7.45-7.41 (m, 2 H), 7.37-7.33 (m, 1 H).

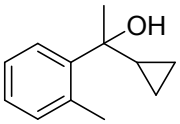


The data of products



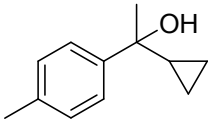
1-cyclopropyl-1-phenylethanol (1a)

Colorless oil (0.69 g, 85% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.50-7.48 (m, 2 H), 7.31-7.27 (m, 2 H), 7.22-7.18 (m, 1 H), 2.02 (s, 1 H), 1.44 (s, 3 H), 1.23-1.16 (m, 1 H), 0.50-0.34 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 148.0, 127.8, 126.5, 125.0, 73.0, 28.3, 22.6, 1.8, 1.0.



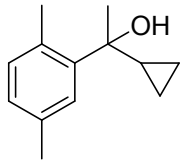
1-cyclopropyl-1-(*o*-tolyl)ethanol (1b)

Yellow oil (0.70 g, 80% yield). 1H NMR (400 MHz, CDCl3, ppm): δ = 7.60 (s, 1 H), 7.12 (s, 3 H), 2.58 (s, 3 H), 1.70 (s, 1 H), 1.45 (s, 3 H), 1.41-1.34 (m, 1 H), 0.60-0.46 (m, 3 H), 0.38- 0.34 (m, 1 H); 13C NMR (100 MHz, CDCl3, ppm): δ = 145.1, 136.1, 132.4, 126.8, 126.1, 125.3, 74.4, 26.9, 22.3, 3.1, 1.7.



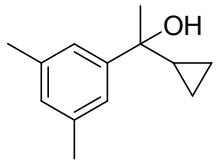
1-cyclopropyl-1-(*p*-tolyl)ethanol (1c)

Yellow oil (0.71 g, 81% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.42-7.40 (d, *J* = 8.0 Hz, 2 H), 7.15-7.13 (d, *J* = 8.0 Hz, 2 H), 2.34 (s, 3 H), 1.93 (s, 1 H), 1.47 (s, 3 H), 1.25-1.19 (m, 1 H), 0.44-0.38 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 145.0, 136.0, 128.5, 125.0, 72.8, 28.4, 22.6, 20.8, 1.7, 1.0.



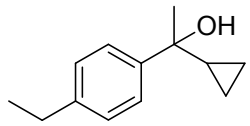
1-cyclopropyl-1-(2, 5-dimethylphenyl)ethanol (1d)

Yellow oil (0.71 g, 75% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.43 (s, 1 H), 7.05-7.02 (d, *J* = 9.0 Hz, 1 H), 6.97-6.95 (d, *J* = 6.0 Hz, 1 H), 2.54 (s, 3 H), 2.31 (s, 3 H), 1.60 (s, 1 H), 1.46 (s, 3 H), 1.44-1.35 (m, 1 H), 0.60-0.49 (m, 3 H), 0.42-0.35 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 145.0, 134.7, 132.8, 132.4, 127.5, 126.9, 74.4, 27.0, 22.3, 21.9, 21.2, 3.2, 1.8.



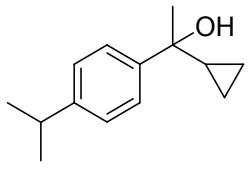
1-cyclopropyl-1-(3,5-dimethylphenyl)ethanol (1e)

Yellow oil (0.63 g, 66% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.11 (s, 2 H), 6.85 (s, 1 H), 2.54 (s, 3 H), 2.29 (s, 3 H), 1.90 (s, 1 H), 1.42 (s, 3 H), 1.20-1.17 (m, 1 H), 0.45-0.38 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 148.1, 137.1, 128.1, 122.8, 72.8, 28.4, 22.6, 21.3, 21.2, 1.7, 1.0.



1-cyclopropyl-1-(4-ethylphenyl)ethanol (1f)

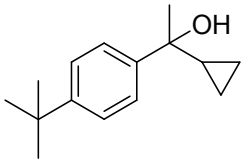
Yellow oil (0.82 g, 86% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.44-7.41 (d, *J* = 9.0 Hz, 2 H), 7.17-7.14 (d, *J* = 9.0 Hz, 2 H), 2.66-2.59 (m, 2 H), 1.76 (s, 1 H), 1.46 (s, 3 H), 1.27-1.17 (m, 4 H), 0.52-0.34 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 145.3, 142.5, 127.4, 125.1, 72.9, 28.5, 28.3, 22.6, 15.5, 1.7, 1.0.



1-cyclopropyl-1-(4-isopropylphenyl)ethanol (1g)

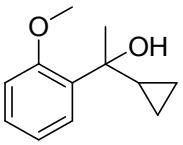
Colorless oil (0.71 g, 70% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.47-7.43 (m, 2 H), 7.22-7.18 (m, 2 H), 2.95-2.85 (m, 1 H), 1.59 (s, 1 H), 1.48 (s, 3 H), 1.27-1.22 (m, 7 H), 0.49-0.41 (m, 4

H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 147.2, 145.5, 126.0, 125.1, 72.9, 33.6, 28.5, 24.0, 22.7, 1.8, 1.1.



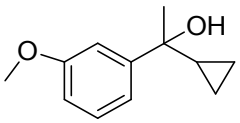
1-(4-(tert-butyl)phenyl)-1-cyclopropylethanol (1h)

Yellow oil (0.45 g, 41% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.46-7.44 (d, *J* = 8.0 Hz, 2 H), 7.36-7.34 (d, *J* = 8.0 Hz, 2 H), 1.71 (s, 1 H), 1.47 (s, 3 H), 1.31 (s, 9 H), 1.28-1.20 (m, 1 H), 0.53-0.38 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 149.4, 145.0, 124.9, 124.8, 72.9, 34.3, 31.3, 28.5, 22.6, 1.8, 1.1.



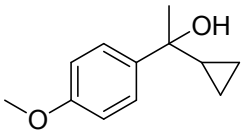
1-cyclopropyl-1-(2-methoxyphenyl)ethanol (1i)

Yellow oil (0.73 g, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.46-7.44 (d, *J* = 8.0 Hz, 1 H), 7.22-7.18 (m, 1 H), 6.95-6.87 (m, 2 H), 4.17 (s, 1 H), 3.81 (s, 3 H), 1.52 (s, 3 H), 1.36-1.30 (m, 1 H), 0.51-0.39 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 156.8, 135.1, 127.8, 126.6, 120.7, 111.1, 73.2, 55.1, 27.0, 21.1, 1.4, 1.2.



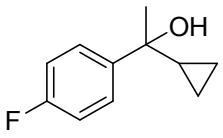
1-cyclopropyl-1-(3-methoxyphenyl)ethanol (1j)

Yellow oil (0.71 g, 74% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.24-7.21 (m, 1 H), 7.10-7.07 (m, 2 H), 6.79-6.75 (m, 1 H), 3.79 (s, 3 H), 1.91 (s, 1 H), 1.45 (s, 3 H), 1.25-1.18 (m, 1 H), 0.54-0.36 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 159.2, 149.9, 128.9, 117.5, 111.6, 111.2, 73.0, 55.0, 28.4, 22.6, 1.9, 1.0.



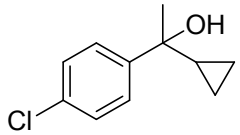
1-cyclopropyl-1-(4-methoxyphenyl)ethanol (1k)

Yellow oil (0.70 g, 73% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.45-7.42 (d, *J* = 9.0 Hz, 2 H), 6.86-6.84 (d, *J* = 6.0 Hz, 2 H), 3.78 (s, 3 H), 1.80 (s, 1 H), 1.46 (s, 3 H), 1.26-1.17 (m, 1 H), 0.52-0.33 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 158.2, 140.1, 126.3, 113.2, 72.7, 55.1, 28.6, 22.7, 1.7, 1.1.



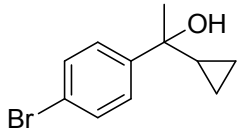
1-cyclopropyl-1-(4-fluorophenyl)ethanol (1l)

Yellow oil (0.69 g, 76% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.49-7.45 (m, 2 H), 7.00-6.96 (m, 2 H), 2.01 (s, 1 H), 1.44 (s, 3 H), 1.23-1.16 (m, 1 H), 0.52-0.47 (m, 1 H), 0.44-0.31 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 162.8, 160.4, 143.8, 126.8 (d, *J* = 7.0 Hz, 1 C), 114.4 (d, *J* = 20.0 Hz, 1 C), 72.8, 28.4, 22.7, 1.9, 1.0.



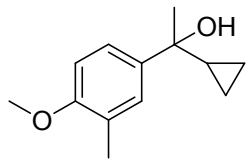
1-(4-chlorophenyl)-1-cyclopropylethanol (1m)

Yellow oil (0.66 g, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.45-7.42 (m, 2 H), 7.29-7.26 (m, 2 H), 1.80 (s, 1 H), 1.44 (s, 3 H), 1.24-1.17 (m, 1 H), 0.53-0.48 (m, 1 H), 0.46-0.34 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.6, 132.4, 128.0, 126.6, 72.9, 28.4, 22.7, 2.0, 1.0.



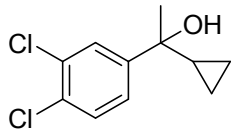
1-(4-bromophenyl)-1-cyclopropylethanol (1n)

Yellow oil (0.81 g, 67% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.41-7.39 (d, *J* = 8.0 Hz, 2 H), 7.36-7.34 (d, *J* = 8.0 Hz, 2 H), 2.21 (s, 1 H), 1.40 (s, 3 H), 1.19-1.12 (m, 1 H), 0.49-0.33 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.9, 130.8, 126.9, 120.4, 72.8, 28.1, 22.5, 1.9, 1.0.



1-cyclopropyl-1-(4-methoxy-3-methylphenyl)ethanol (1o)

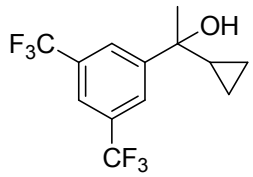
Yellow oil (0.65 g, 64% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.31-7.29 (m, 2 H), 6.78-6.75 (d, *J* = 9.0 Hz, 1 H), 3.80 (s, 3 H), 2.22 (s, 3 H), 1.72 (s, 1 H), 1.46 (s, 3 H), 1.26-1.18 (m, 1 H), 0.44-0.39 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 156.3, 139.6, 127.6, 125.7, 123.3, 109.0, 72.6, 55.1, 28.5, 22.6, 16.3, 1.6, 1.0.



1-cyclopropyl-1-(3,4-dichlorophenyl)ethanol (1p)

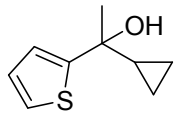
Yellow oil (0.59 g, 51% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.61 (s, 1 H), 7038-7.32 (m, 2 H), 1.95 (s, 1 H), 1.44-1.43 (d, *J* = 4.0 Hz, 3 H), 1.23-1.15 (m, 1 H), 0.56-0.37 (m, 4 H); ^{13}C

NMR (100 MHz, $CDCl_3$, ppm): δ = 148.6, 131.9, 130.4, 129.8, 127.3, 124.7, 72.6, 28.2, 22.7, 2.1, 1.0.



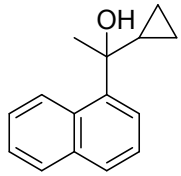
1-(3,5-bis(trifluoromethyl)phenyl)-1-cyclopropylethanol (1q)

Yellow oil (0.68 g, 46% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.01 (s, 2 H), 7.78 (s, 1 H), 2.03 (s, 1 H), 1.50 (s, 3 H), 1.50-1.23 (m, 1 H), 0.66-0.60 (m, 1 H), 0.56-0.43 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 151.1, 131.28 (q, *J* = 33.0 Hz, 1 C), 125.4 (d, *J* = 3.0 Hz, 1 C), 123.5 (q, *J* = 271.0 Hz, 1 C), 120.7 (d, *J* = 4.0 Hz, 1 C), 73.0, 28.2, 22.8, 2.4, 0.9.



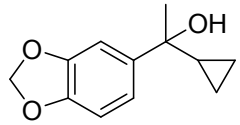
1-cyclopropyl-1-(thiophen-2-yl)ethanol (1r)

Yellow oil (0.60 g, 71% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.17-7.15 (m, 1 H), 6.98-6.97 (m, 1 H), 6.93-6.91 (m, 1 H), 2.13 (s, 1 H), 1.57 (s, 3 H), 1.30-1.24 (m, 1 H), 0.52-0.44 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 153.0, 126.3, 123.8, 122.5, 71.8, 29.4, 23.1, 1.5.



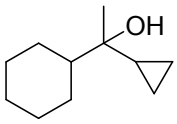
1-cyclopropyl-1-(naphthalen-1-yl)ethanol (1s)

Yellow oil (0.69 g, 74% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 8.86-8.84 (d, *J* = 8.0 Hz, 1 H), 7.85-7.81 (m, 2 H), 7.76-7.74 (d, *J* = 8.0 Hz, 1 H), 7.48-7.38 (m, 3 H), 1.84 (s, 1 H), 1.66 (s, 3 H), 1.62-1.57 (m, 1 H), 0.69-0.56 (m, 3 H), 0.50-0.44 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 143.2, 134.8, 131.1, 129.0, 128.5, 127.3, 125.1, 125.0, 124.8, 123.7, 74.9, 27.7, 22.9, 3.4, 2.0.



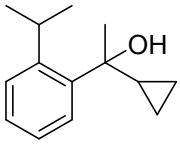
1-(benzo[d][1, 3] dioxol-5-yl)-1-cyclopropylethanol (1t)

Colorless oil (0.67 g, 65% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.04-7.00 (m, 2 H), 6.76-6.74 (d, *J* = 8.0 Hz, 1 H), 5.91 (s, 2 H), 1.94 (s, 1 H), 1.43 (s, 3 H), 1.22-1.18 (m, 1 H), 1.05-1.35 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 147.2, 146.0, 142.3, 118.1, 107.5, 106.3, 100.8, 72.9, 28.6, 22.7, 1.9, 1.1.



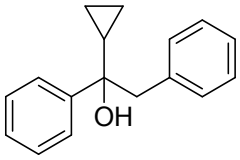
1-cyclohexyl-1-cyclopropylethanol (1u)

Colorless oil (0.72 g, 86% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 1.52-1.31 (m, 8 H), 1.08 (s, 3 H), 0.94-0.87 (m, 4 H), 0.36-0.29 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 70.9, 42.9, 26.1, 25.1, 23.2, 20.8, 14.0, 0.5, 0.2.



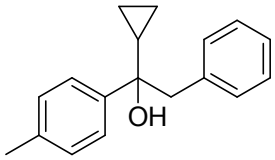
1-cyclopropyl-1-(4-isopropylphenyl)ethanol (1v)

Colorless oil (0.56 g, 55% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.65-7.63 (d, *J* = 6.0 Hz, 1 H), 7.38-7.36 (d, *J* = 6.0 Hz, 1 H), 7.26-7.20 (m, 1 H), 7.14-7.09 (m, 1 H), 3.99-3.90 (m, 1 H), 1.62 (s, 1 H), 1.48 (s, 3 H), 1.45-1.38 (m, 1 H), 1.27-1.23 (m, 6 H), 0.65-0.41 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 148.1, 144.1, 127.5, 127.3, 125.8, 125.1, 74.2, 29.2, 27.8, 24.9, 24.6, 22.9, 3.3, 1.9.



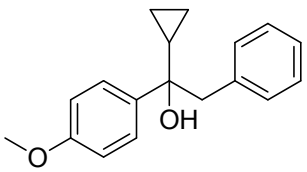
1-cyclopropyl-1,2-diphenylethanol (1w)

Yellow oil (0.96 g, 86% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.44-7.42 (d, *J* = 8.0 Hz, 2 H), 7.33-7.29 (m, 2 H), 7.25-7.16 (m, 4 H), 6.98 (s, 2 H), 3.23-3.15 (m, 2 H), 1.57 (s, 1 H), 1.36-1.31 (m, 1 H), 0.45-0.31 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.3, 136.4, 130.7, 127.9, 127.8, 126.7, 126.5, 125.7, 74.6, 49.2, 20.9, 1.6, 0.7.



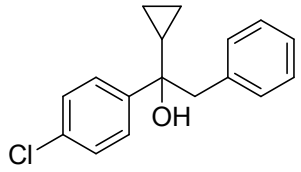
1-cyclopropyl-2-phenyl-1-(*p*-tolyl)ethanol (1x)

Yellow soild (0.89 g, 71% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.33-7.30 (m, 2 H), 7.19-7.10 (m, 5 H), 7.01-6.98 (m, 2 H), 3.22-3.12 (m, 2 H), 2.33 (s, 3 H), 1.54 (s, 1 H), 1.33-1.26 (m, 1 H), 0.42-0.31 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 143.2, 136.6, 136.7, 130.7, 128.5, 127.9, 126.4, 125.6, 74.5, 49.2, 21.0, 20.8, 1.5, 0.7.



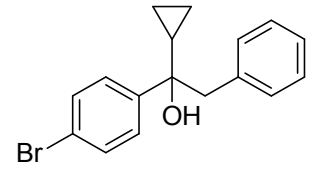
1-cyclopropyl-1-(4-methoxyphenyl)-2-phenylethanol (1y)

Yellow soild (0.87 g, 65% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.36-7.33 (m, 2 H), 7.18-7.15 (m, 3 H), 7.00-6.96 (m, 2 H), 6.85-6.82 (m, 2 H), 3.78 (s, 3 H), 3.16 (s, 2 H), 1.56 (s, 1 H), 1.32-1.27 (m, 1 H), 0.41-0.27 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 158.2, 138.2, 136.6, 130.7, 127.8, 127.0, 126.4, 113.0, 74.4, 55.1, 49.2, 20.7, 1.5, 0.7.



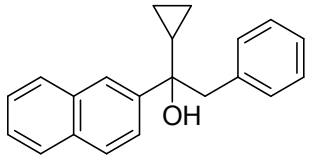
1-(4-chlorophenyl)-1-cyclopropyl-2-phenylethanol (1z)

Yellow oil (0.86 g, 63% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.35-7.32 (d, *J* = 9.0 Hz, 2 H), 7.26-7.24 (m, 2 H), 7.19-7.16 (m, 3 H), 7.00-6.95 (m, 2 H), 3.18-3.08 (m, 2 H), 1.61-1.60 (d, *J* = 3.0 Hz, 1 H), 1.32-1.26 (m, 1 H), 0.42-0.28 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 144.9, 136.0, 132.4, 130.6, 128.0, 127.9, 127.2, 126.6, 74.3, 49.0, 20.9, 1.7, 0.7.



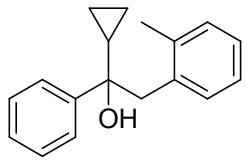
1-(4-bromophenyl)-1-cyclopropyl-2-phenylethanol (1aa)

Yellow soild (0.96 g, 61% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.42-7.40 (m, 2 H), 7.29-7.27 (m, 2 H), 7.19-7.17 (m, 3 H), 6.98-6.95 (m, 2 H), 3.17-3.10 (m, 2 H), 1.57 (s, 1 H), 1.31-1.24 (m, 1 H), 0.44-0.26 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 145.4, 135.9, 130.8, 130.6, 128.0, 127.6, 126.7, 120.6, 74.3, 49.0, 20.9, 1.7, 0.7.



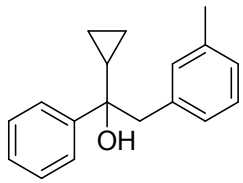
1-cyclopropyl-1-(naphthalen-2-yl)-2-phenylethanol (1ab)

Yellow soild (0.91 g, 63% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.83-7.77 (m, 4 H), 7.63-7.61 (d, *J* = 4.0 Hz, 1 H), 7.45-7.43 (m, 2 H), 7.20-7.13 (m, 3 H), 7.00-6.99 (m, 2 H), 3.34-3.24 (m, 2 H), 1.69 (s, 1 H), 1.47-1.40 (m, 1 H), 0.47-0.35 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 143.7, 136.3, 133.0, 132.3, 130.7, 128.2, 128.0, 127.4, 126.6, 125.9, 125.7, 124.4, 124.3, 74.8, 48.9, 21.1, 1.7, 0.9.



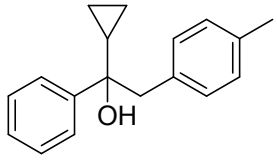
1-cyclopropyl-1-phenyl-2-(*o*-tolyl)ethanol (1ac)

Yellow oil (1.01 g, 80% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.41 (s, 2 H), 7.30-7.23 (m, 3 H), 7.09 (s, 2 H), 7.00-6.92 (m, 2 H), 3.28-3.13 (m, 2 H), 2.16-2.15 (d, *J* = 4.0 Hz, 3 H), 1.58-1.56 (m, 1 H), 1.40-1.39 (d, *J* = 4.0 Hz, 1 H), 0.43-0.30 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.8, 138.0, 134.8, 131.4, 130.3, 127.8, 126.7, 126.6, 125.6, 125.3, 74.9, 45.4, 20.8, 20.0, 1.8, 0.9.



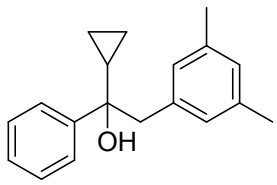
1-cyclopropyl-1-phenyl-2-(*m*-tolyl)ethanol (1ad)

Yellow oil (1.12 g, 89% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.40-7.37 (m, 2 H), 7.28-7.16 (m, 3 H), 7.05-6.89 (s, 4 H), 3.25-3.09 (m, 2 H), 2.13-2.12 (m, 3 H), 1.61-1.59 (m, 1 H), 1.40-1.31 (m, 1 H), 0.44-0.26 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.6, 137.9, 134.7, 131.4, 130.1, 127.7, 126.6, 126.5, 125.6, 125.2, 74.9, 45.4, 20.7, 19.9, 1.7, 0.8.



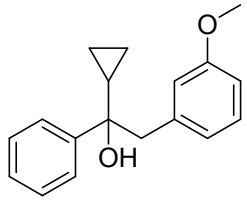
1-cyclopropyl-1-phenyl-2-(*p*-tolyl)ethanol (1ae)

Yellow oil (1.15 g, 91% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.44-7.43 (m, 2 H), 7.33-7.28 (m, 2 H), 7.25-7.23 (m, 1 H), 6.99-6.97 (d, *J* = 8.0 Hz, 2 H), 6.88-6.85 (m, 2 H), 3.20-3.11 (m, 2 H), 2.27-2.25 (d, *J* = 8.0 Hz, 3 H), 1.62-1.59 (m, 1 H), 1.35-1.30 (m, 1 H), 0.42-0.31 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.3, 136.0, 133.1, 130.5, 128.6, 127.7, 126.6, 125.7, 74.5, 48.6, 21.0, 20.9, 1.6, 0.7.



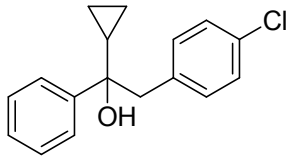
1-cyclopropyl-2-(3,5-dimethylphenyl)-1-phenylethanol (1af)

Yellow soild (1.19 g, 89% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 7.42-7.44 (d, *J* = 8.0 Hz, 2 H), 7.33-7.29 (m, 2 H), 7.24-7.21 (m, 1 H), 6.81 (s, 1 H), 6.59 (s, 2 H), 3.15-3.07 (m, 2 H), 2.19 (s, 6 H), 1.37 (s, 1 H), 1.35-1.30 (m, 1 H), 0.42-0.29 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 146.5, 137.3, 136.1, 128.5, 128.2, 127.7, 126.5, 125.7, 74.3, 49.1, 21.2, 20.9, 1.7, 0.6.



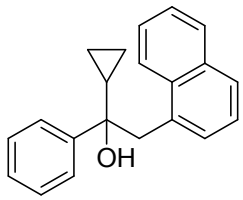
1-cyclopropyl-2-(3-methoxyphenyl)-1-phenylethanol (1ag)

Yellow soild (1.09 g, 81% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.42-7.40 (m, 2 H), 7.30-7.19 (m, 3 H), 7.07-6.94 (m, 2 H), 6.79-6.73 (m, 2 H), 3.17-3.07 (m, 2 H), 2.20 (s, 3 H), 1.64-1.63 (d, *J* = 3.0 Hz, 1 H), 1.33-1.26 (m, 1 H), 0.45-0.29 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 146.3, 137.3, 136.2, 131.5, 127.7, 127.7, 127.6, 127.1, 126.5, 125.7, 74.4, 49.1, 21.2, 20.8, 1.6, 0.6.



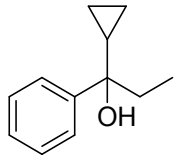
2-(4-chlorophenyl)-1-cyclopropyl-1-phenylethanol (1ah)

Yellow oil (1.18 g, 87% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.40-7.38 (d, *J* = 6.0 Hz, 2 H), 7.32-7.20 (m, 3 H), 7.13-7.10 (d, *J* = 9.0 Hz, 2 H), 6.88-6.86 (d, *J* = 6.0 Hz, 2 H), 3.16-3.07 (m, 2 H), 1.53 (s, 1 H), 1.35-1.26 (m, 1 H), 0.45-0.30 (m, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 145.8, 135.0, 132.3, 131.9, 127.9, 126.8, 125.7, 74.6, 48.4, 20.6, 1.5, 0.8.



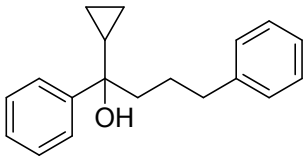
1-cyclopropyl-2-(naphthalen-1-yl)-1-phenylethanol (1ai)

Yellow oil (0.99 g, 69% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 8.12 (s, 1 H), 7.77-7.67 (m, 2 H), 7.44-7.40 (m, 4 H), 7.26-7.22 (m, 4 H), 7.07 (s, 1 H), 3.76-3.71 (m, 1 H), 3.59-3.53 (m, 1 H), 1.65-1.60 (d, *J* = 15.0 Hz, 1 H), 1.33-1.31 (d, *J* = 6.0 Hz, 1 H), 0.28 (s, 4 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 146.5, 133.7, 133.3, 132.8, 129.1, 128.4, 127.7, 127.2, 126.6, 125.7, 125.5, 125.2, 124.9, 124.7, 75.2, 45.0, 20.8, 1.8, 1.0.



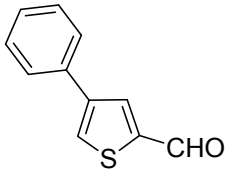
1-cyclopropyl-1-phenylpropan-1-ol (1aj)

Yellow oil (0.69 g, 78% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.45-7.43 (d, *J* = 6.0 Hz, 2 H), 7.33-7.28 (m, 2 H), 7.23-7.17 (m, 1 H), 1.97-1.76 (m, 2 H), 1.66-1.63 (m, 1 H), 1.31-1.22 (m, 1 H), 0.80-0.75 (m, 3 H), 0.49-0.23 (m, 4 H)； ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 146.0, 127.7, 126.4, 125.6, 75.1, 34.7, 21.3, 7.9, 1.2, 0.5.



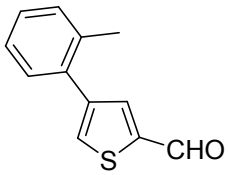
1-cyclopropyl-1,4-diphenylbutan-1-ol (1ak)

Yellow oil (0.64 g, 48% yield). ^{1}H NMR (300 MHz, $CDCl_3$, ppm): δ = 7.42-7.40 (d, *J* = 6.0 Hz, 2 H), 7.33-7.29 (m, 2 H), 7.25-7.20 (m, 3 H), 7.16-7.09 (m, 3 H), 2.62-2.50 (m, 2 H), 1.98-1.81 (m, 2 H), 1.71-1.60 (m, 1 H), 1.53-1.41 (m, 2 H), 1.30-1.24 (m, 1 H), 0.49-0.42 (m, 2 H), 0.38-0.31(m, 1 H), 0.29-0.23 (m, 1 H); ^{13}C NMR (75 MHz, $CDCl_3$, ppm): δ = 146.2, 142.2, 128.3, 128.2, 127.9, 126.5, 125.6, 125.5, 74.9, 41.9, 36.1, 25.4, 21.8, 1.4, 0.7.



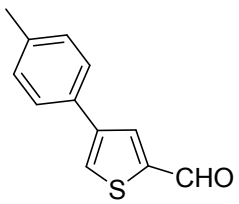
4-phenylthiophene-2-carbaldehyde (2a)

Colorless soild (40.6 mg, 71% yield), melting point: 44-46 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.98 (d, *J* = 1.2 Hz, 1 H), 8.04-8.03 (d, *J* = 4.0 Hz, 1 H), 7.86-7.85 (m, 1 H), 7.60-7.58 (m, 2 H), 7.46-7.42 (m, 2 H), 7.38-7.34 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 144.3, 143.6, 134.7, 134.3, 129.6, 129.0, 128.0, 126.3; HRMS calcd for $C_{11}H_9OS$ $[M+H]^+$ 189.0369; found: 189.0372.



4-(*o*-tolyl)thiophene-2-carbaldehyde (2b)

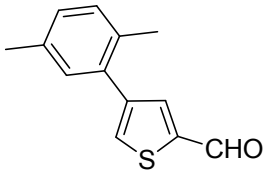
Yellow oil (36.9 mg, 61% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.96 (s, 1 H), 7.80(d, *J* = 1.2 Hz, 1 H), 7.63 (s, 1 H), 7.30-7.25 (m, 2 H), 2.34 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 144.2, 143.6, 137.9, 134.7, 131.5, 129.7, 129.0, 126.1, 21.1; HRMS calcd for $C_{12}H_{11}OS$ $[M+H]^+$ 203.0525; found: 203.0529.



4-(*p*-tolyl)thiophene-2-carbaldehyde (2c)

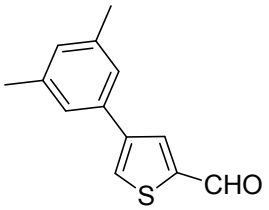
Yellow oil (40.6 mg, 65% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (s, 1 H), 8.00 (d, *J* = 1.2 Hz, 1 H), 7.79 (s, 1 H), 7.48-7.46 (d, *J* = 8.0 Hz, 2 H), 7.24-7.22 (d, *J* = 8.0 Hz, 2 H); ^{13}C

NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 144.2, 143.6, 137.9, 134.7, 131.5, 129.7, 129.0, 126.1, 21.1; HRMS calcd for $C_{12}H_{11}OS$ $[M+H]^+$ 203.0525; found: 203.0530.



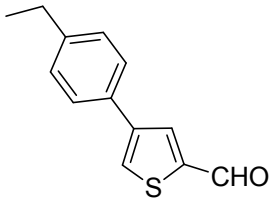
4-(2,5-dimethylphenyl)thiophene-2-carbaldehyde (2d)

Yellow oil (44.7 mg, 69% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (d, *J* = 1.2 Hz, 1 H), 7.79 (d, *J* = 1.2 Hz, 1 H), 7.61 (s, 1 H), 7.18-7.16 (d, *J* = 8.0 Hz, 1 H), 7.11-7.09 (d, *J* = 8.0 Hz, 2 H), 2.35 (s, 3 H), 2.30 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 143.7, 143.4, 137.5, 135.6, 134.6, 132.3, 132.2, 130.6, 130.2, 128.8, 20.8, 20.1; HRMS calcd for $C_{13}H_{13}OS$ $[M+H]^+$ 217.0682; found: 217.0686.



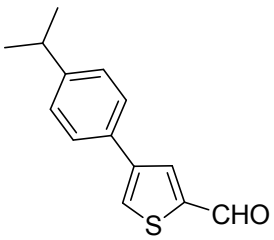
4-(3,5-dimethylphenyl)thiophene-2-carbaldehyde (2e)

Yellow oli (42.7 mg, 65% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.96-9.95 (d, *J* = 4.0 Hz, 1 H), 8.02-8.01 (d, *J* = 4.0 Hz, 1 H), 7.81 (s, 1 H), 7.20 (s, 2 H), 7.00 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 144.2, 143.8, 138.6, 135.0, 134.2, 129.7, 129.5, 124.2; HRMS calcd for $C_{13}H_{13}OS$ $[M+H]^+$ 217.0682; found: 217.0687.



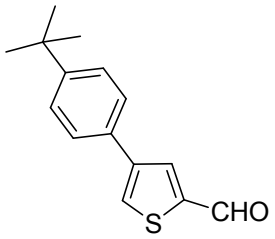
4-(4-ethylphenyl)thiophene-2-carbaldehyde (2f)

Yellow oli (40.2 mg, 62% yield). ^1H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95-9.94 (d, *J* = 4.0 Hz, 1 H), 8.00 (d, *J* = 1.6 Hz, 1 H), 7.79 (s, 1 H), 7.51-7.49 (d, *J* = 8.0 Hz, 2 H), 7.27-7.25 (d, *J* = 8.0 Hz, 2 H), 2.71-2.65 (m, 2 H), 1.28-1.24 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.9, 144.2, 143.6, 134.7, 131.7, 129.1, 128.5, 126.2, 28.5, 15.5; HRMS calcd for $C_{13}H_{13}OS$ $[M+H]^+$ 217.0682; found: 217.0688.



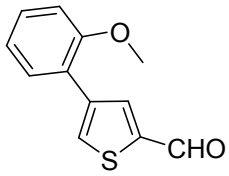
4-(4-isopropylphenyl)thiophene-2-carbaldehyde (2g)

Yellow solid (51.0 mg, 72% yield), melting point: 60-62 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.96 (d, *J* = 1.2 Hz, 1 H), 8.02-8.01 (d, *J* = 4.0 Hz, 1 H), 7.81-7.80 (m, 1 H), 7.53-7.50 (d, *J* = 12.0 Hz, 2 H), 7.31-7.29 (d, *J* = 8.0 Hz, 2 H), 3.02-2.88 (m, 1 H), 1.29 (s, 3 H), 1.27 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 148.9, 144.2, 143.6, 134.7, 131.9, 129.1, 127.1, 126.3, 33.8, 23.9; HRMS calcd for $C_{14}H_{15}OS$ $[M+H]^+$ 231.0838; found: 231.0843.



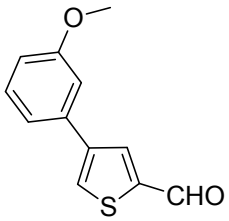
4-(4-(tert-butyl)phenyl)thiophene-2-carbaldehyde (2h)

Yellow solid (41.7 mg, 57% yield), melting point: 65-68 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.96-9.95 (d, *J* = 4.0 Hz, 1 H), 8.01 (d, *J* = 1.2 Hz, 1 H), 7.81-7.80 (d, *J* = 4.0 Hz, 1 H), 7.53-7.51 (d, *J* = 8.0 Hz, 2 H), 7.47-7.45 (d, *J* = 8.0 Hz, 2 H), 1.35 (s, 9 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 151.1, 144.2, 143.5, 134.7, 131.5, 129.2, 126.0, 34.6, 31.2; HRMS calcd for $C_{15}H_{17}OS$ $[M+H]^+$ 245.0995; found: 245.0999.



4-(2-methoxyphenyl)thiophene-2-carbaldehyde (2i)

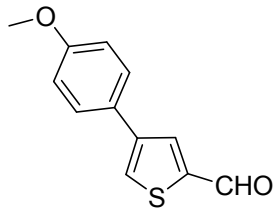
Yellow oil (39.3 mg, 60% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.97 (d, *J* = 0.8 Hz, 1 H), 8.11 (d, *J* = 1.2 Hz, 1 H), 8.00 (s, 1 H), 7.48-7.46 (m, 1 H), 7.36-7.32 (m, 1 H), 7.05-7.00 (m, 2 H), 3.89 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.2, 156.4, 142.9, 139.7, 137.7, 132.6, 129.4, 129.3, 123.5, 121.0, 111.4, 55.5; HRMS calcd for $C_{12}H_{11}O_2S$ $[M+H]^+$ 219.0475; found: 219.0482.



4-(3-methoxyphenyl)thiophene-2-carbaldehyde (2j)

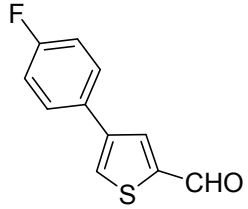
Yellow oil (35.2 mg, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.97 (d, *J* = 0.8 Hz, 1 H), 8.02 (d, *J* = 1.2 Hz, 1 H), 7.85 (s, 1 H), 7.38-7.34 (m, 1 H), 7.19-7.17 (d, *J* = 8.0 Hz, 1 H), 7.12-7.11 (m, 1 H), 6.92-6.89 (m, 1 H), 3.87 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0,

160.1, 144.3, 143.5, 135.7, 134.8, 130.1, 129.8, 118.8, 113.2, 11.2, 55.3; HRMS calcd for $C_{12}H_{11}O_2S$ $[M+H]^+$ 219.0475; found: 219.0481.



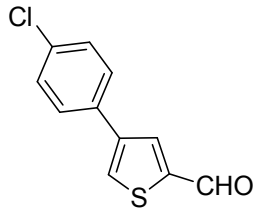
4-(4-methoxyphenyl)thiophene-2-carbaldehyde (2k)

Yellow oil (34.6 mg, 53% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (d, *J* = 1.2 Hz, 1 H), 7.98-7.97 (d, *J* = 4 Hz, 1 H), 7.74 (m, 1 H), 7.52-7.50 (m, 2 H), 6.97-6.95 (m, 2 H), 3.85 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 159.5, 144.2, 143.3, 134.6, 128.4, 127.4, 127.1, 114.4, 55.3; HRMS calcd for $C_{12}H_{11}O_2S$ $[M+H]^+$ 219.0475; found: 219.0480.



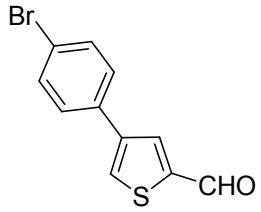
4-(4-fluorophenyl)thiophene-2-carbaldehyde (2l)

Yellow soild (47.0 mg, 76% yield), melting point: 58-61 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.96 (d, *J* = 0.8 Hz, 1 H), 7.98(d, *J* = 1.2 Hz, 1 H), 7.79 (s, 1 H), 7.57-7.53 (m, 2 H), 7.15-7.10 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.9, 162.5 (d, *J* = 247.0 Hz, 1 C), 144.5, 142.5, 134.5, 130.6, 129.3, 128.0 (d, *J* = 8.0 Hz, 1 C), 116.0 (d, *J* = 21.0 Hz, 1 C); HRMS calcd for $C_{11}H_8FOS$ $[M+H]^+$ 207.0275; found: 207.0279.



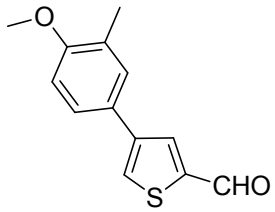
4-(4-chlorophenyl)thiophene-2-carbaldehyde (2m)

Yellow solid (40.1 mg, 60% yield), melting point: 56-59 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.97 (d, *J* = 0.8 Hz, 1 H), 8.00-7.99 (d, *J* = 4.0 Hz, 1 H), 7.83 (d, *J* = 0.8 Hz, 1 H), 7.53-7.51 (d, *J* = 8.0 Hz, 2 H), 7.42-7.40 (d, *J* = 8.0 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 144.6, 142.3, 134.3, 134.0, 132.8, 129.7, 129.2, 127.5; HRMS calcd for $C_{11}H_8ClOS$ $[M+H]^+$ 222.9979; found: 222.9985.



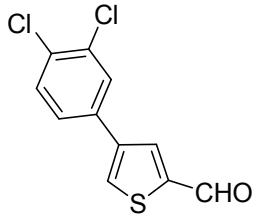
4-(4-bromophenyl)thiophene-2-carbaldehyde (2n)

Yellow solid (41.4 mg, 52% yield), melting point: 56-58 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.96 (d, *J* = 1.2 Hz, 1 H), 7.99 (d, *J* = 1.6 Hz, 1 H), 7.84 (d, *J* = 1.2 Hz, 1 H), 7.57-7.55 (m, 2 H), 7.46-7.44 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 144.6, 142.3, 134.3, 133.2, 132.1, 129.8, 127.8, 122.0; HRMS calcd for $C_{11}H_8BrOS$ $[M+H]^+$ 266.9474; found: 266.9482.



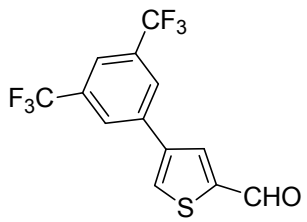
4-(4-methoxy-3-methylphenyl)thiophene-2-carbaldehyde (2o)

Yellow oil (34.8 mg, 49% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (d, *J* = 0.4 Hz, 1 H), 7.98(d, *J* = 0.8 Hz, 1 H), 7.73 (s, 1 H), 7.37 (s, 2 H), 6.88-6.86 (d, *J* = 8.0 Hz, 1 H), 3.87 (s, 3 H), 2.27 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.0, 157.7, 144.1, 143.5, 134.7, 128.7, 128.2, 127.2, 126.6, 124.7，110.2, 53.4, 16.3; HRMS calcd for $C_{13}H_{13}O_2S$ $[M+H]^+$ 233.0631; found: 233.0636.



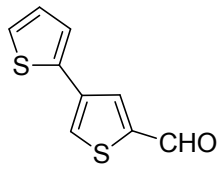
4-(3,4-dichlorophenyl)thiophene-2-carbaldehyde (2p)

Yellow solid (39.9 mg, 52% yield), melting point: 69-72 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.97 (d, *J* = 1.2 Hz, 1 H), 7.98 (d, *J* = 1.2 Hz, 1 H), 7.86-7.85 (m, 1 H), 7.68-7.67 (d, *J* = 4.0 Hz, 1 H), 7.52-7.50 (d, *J* = 8.0 Hz, 1 H), 7.43-7.40 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.7, 144.9, 141.1, 134.3, 134.0, 133.3, 132.1, 131.0, 130.3, 128.2, 125.5; HRMS calcd for $C_{11}H_7Cl_2OS$ $[M+H]^+$ 256.9589; found: 256.9597.



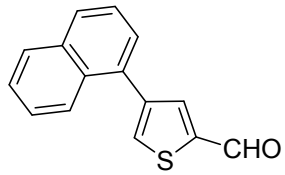
4-(3,5-bis(trifluoromethyl)phenyl)thiophene-2-carbaldehyde (2q)

Yellow solid (55.3 mg, 57% yield), melting point: 83-86 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 10.02 (d, *J* = 1.2 Hz, 1 H), 8.11-8.10 (d, *J* = 4.0 Hz, 1 H), 8.03 (d, *J* = 0.8 Hz, 3 H), 7.87 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.6, 145.3, 140.4, 136.4, 133.9, 132.5 (d, *J* = 34 Hz), 131.3, 126.3, 124.5, 121.5; HRMS calcd for $C_{13}H_7F_6OS$ $[M+H]^+$ 325.0117; found: 325.0124.



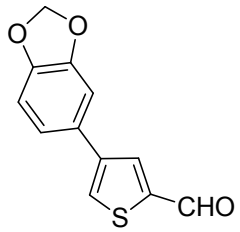
[2,3'-bithiophene]-5'-carbaldehyde (2r)

Yellow solid (30.3 mg, 52% yield), melting point: 80-82 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (d, *J* = 1.2 Hz, 1 H), 7.95 (d, *J* = 1.2 Hz, 1 H), 7.77-7.76 (m, 1 H), 7.29-7.25 (m, 2 H), 7.09-7.07 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 144.3, 137.3, 136.9, 134.2, 128.5, 128.0, 125.0, 124.1; HRMS calcd for $C_9H_7OS_2$ $[M+H]^+$ 194.9933; found: 194.9938.



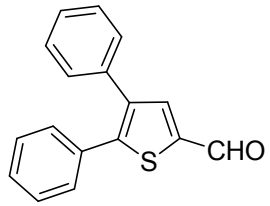
4-(naphthalen-1-yl)thiophene-2-carbaldehyde (2s)

Yellow oil (37.8 mg, 53% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 10.00 (d, *J* = 1.2 Hz, 1 H), 7.94-7.89 (m, 4 H), 7.80 (s, 1 H), 7.54-7.46 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 183.1, 143.7, 142.4, 138.1, 133.7, 133.1, 133.0, 131.3, 128.6, 128.5, 127.1, 126.6, 126.1, 125.3, 125.1; HRMS calcd for $C_{15}H_{11}OS$ $[M+H]^+$ 239.0525; found: 239.0530.



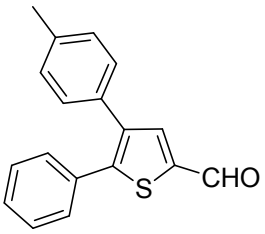
4-(benzo[d][1,3]dioxol-5-yl)thiophene-2-carbaldehyde (2t)

Yellow solid (35.5 mg, 51% yield), melting point: 97-100 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.94 (s, 1 H), 7.93 (d, *J* = 1.2 Hz, 1 H), 7.71 (s, 1 H), 7.07-7.04 (m, 2 H), 6.87-6.85 (d, *J* = 8 Hz, 1 H), 6.00 (s, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.9, 148.3, 147.5, 144.2, 143.3, 134.6, 128.7, 128.6, 119.9, 108.7, 106.8, 101.3; HRMS calcd for $C_{12}H_9O_3S$ $[M+H]^+$ 233.0267; found: 233.0273.



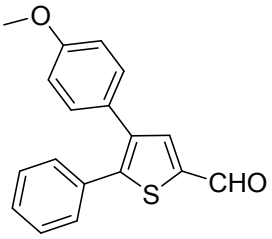
4,5-diphenylthiophene-2-carbaldehyde (2w)

Yellow oil (53.9 mg, 68% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.91 (s, 1 H), 7.79 (s, 1 H), 7.32-7.29 (m, 8 H), 7.28-7.24 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 148.7, 141.4, 139.5, 139.1, 135.1, 133.1, 129.2, 129.0, 128.9, 128.7, 128.6, 127.6; HRMS calcd for $C_{17}H_{13}OS$ $[M+H]^+$ 265.0682; found: 265.0687.



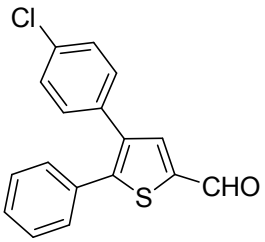
5-phenyl-4-(*p*-tolyl)thiophene-2-carbaldehyde (2x)

Yellow oil (43.4 mg, 52% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.90 (s, 1 H), 7.77 (s, 1 H), 7.34-7.28 (m, 5 H), 7.16-7.10 (m, 4 H), 2.35(s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 148.3, 141.2, 139.5, 139.2, 137.4, 133.2, 132.1, 129.3, 129.1, 128.8, 128.7, 21.2; HRMS calcd for $C_{18}H_{15}OS$ $[M+H]^+$ 279.0838; found: 279.0845.



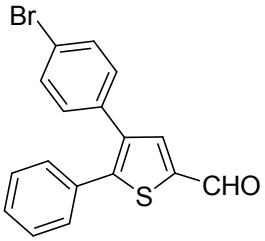
4-(4-methoxyphenyl)-5-phenylthiophene-2-carbaldehyde (2y)

Yellow oil (37.0 mg, 42% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.91 (s, 1 H), 7.77 (s, 1 H), 7.36-7.29 (m, 5 H), 7.20-7.16 (m, 2 H), 6.89-6.83 (m, 2 H), 3.82 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.7, 159.1, 148.0, 141.2, 139.2, 139.1, 133.3, 130.1, 130.1, 129.1, 128.8, 128.7, 127.5, 114.0, 55.2; HRMS calcd for $C_{18}H_{15}O_2S$ $[M+H]^+$ 295.0788; found: 295.0794.



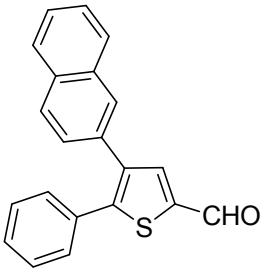
4-(4-chlorophenyl)-5-phenylthiophene-2-carbaldehyde (2z)

Yellow oil (41.2 mg, 46% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.91 (s, 1 H), 7.76 (s, 1 H), 7.35-7.25 (m, 7 H), 7.20-7.17 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.7, 148.9, 141.6, 138.6, 138.1, 133.5, 132.7, 130.6, 130.2, 129.1, 128.8, 127.9, 127.2; HRMS calcd for $C_{17}H_{12}ClOS$ $[M+H]^+$ 299.0292; found: 299.0298.



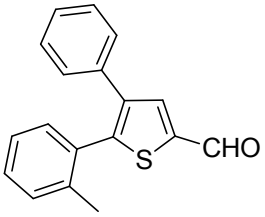
4-(4-bromophenyl)-5-phenylthiophene-2-carbaldehyde (2aa)

Yellow oil (22.7 mg, 39% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.91 (s, 1 H), 7.76 (s, 1 H), 7.45-7.43 (m, 2 H), 7.36-7.29 (m, 5 H), 7.14-7.12 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.7, 149.0, 141.6, 138.5, 138.1, 134.0, 132.7, 131.8, 130.5, 129.1, 129.1, 128.9, 121.9; HRMS calcd for $C_{17}H_{12}BrOS$ $[M+H]^+$ 342.9787; found: 342.9795.



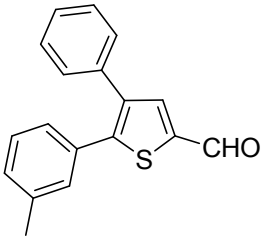
4-(naphthalen-2-yl)-5-phenylthiophene-2-carbaldehyde (2ab)

Yellow oil (49.9 mg, 53% yield), melting point: 106-108 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (s, 1 H), 7.90 (s, 1 H), 7.84-7.82 (m, 2 H), 7.79-7.73 (m, 2 H), 7.52-7.47 (m, 2 H), 7.36-7.31 (m, 3 H), 7.29-7.26 (m, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 148.8, 141.4, 139.4, 139.3, 133.4, 133.0, 132.6, 132.5, 129.1, 128.9, 128.7, 128.1, 128.0, 127.8, 127.7, 126.9, 126.4, 126.3; HRMS calcd for $C_{21}H_{15}OS$ $[M+H]^+$ 315.0838; found: 315.0843.



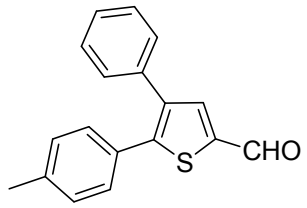
4-phenyl-5-(*o*-tolyl)thiophene-2-carbaldehyde (2ac)

Yellow oil (42.5 mg, 51% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (s, 1 H), 7.90 (s, 1 H), 7.34-7.28 (m, 2 H), 7.25-7.22 (m, 4 H), 7.17-7.15 (m, 3 H), 1.94(s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.9, 148.0, 142.0, 140.6, 137.6, 136.9, 135.0, 132.6, 130.9, 130.5, 129.2, 128.5, 127.8, 127.4, 126.0, 20.0; HRMS calcd for $C_{18}H_{15}OS$ $[M+H]^+$ 279.0838; found: 279.0843.



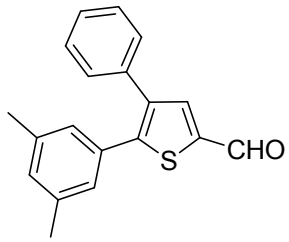
4-phenyl-5-(*m*-tolyl)thiophene-2-carbaldehyde (2ad)

Yellow oil (39.2 mg, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.95 (s, 1 H), 7.90 (s, 1 H), 7.34-7.28 (m, 2 H), 7.25-7.21 (m, 4 H), 7.17- 7.15 (m, 3 H), 1.94 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.9, 148.0, 142.0, 140.6, 137.6, 136.9, 135.0, 132.6, 130.9, 130.5, 129.2, 128.5, 127.8, 127.4, 126.0, 20.0; HRMS calcd for $C_{18}H_{15}OS$ $[M+H]^+$ 279.0838; found: 279.0843.



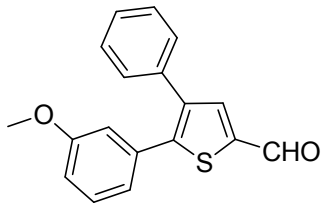
4-phenyl-5-(*p*-tolyl)thiophene-2-carbaldehyde (2ae)

Yellow oil (38.4 mg, 46% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.89 (s, 1 H), 7.76 (s, 1 H), 7.32-7.29 (m, 3 H), 7.27-7.24 (m, 2 H), 7.21 (s, 1 H), 7.19 (s, 1 H), 7.10-7.08 (d, *J* = 8 Hz, 2 H), 2.33 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 149.0, 141.0, 139.2, 139.1, 139.0, 135.3, 130.1, 129.4, 129.0, 128.9, 128.6, 127.5, 21.3; HRMS calcd for $C_{18}H_{15}OS$ $[M+H]^+$ 279.0838; found: 279.0844.



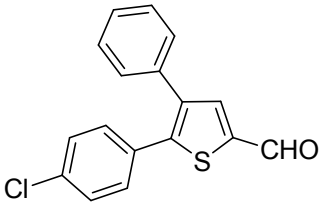
5-(3,5-dimethylphenyl)-4-phenylthiophene-2-carbaldehyde (2af)

Yellow oil (36.8 mg, 42% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.89 (s, 1 H), 7.77 (s, 1 H), 7.31-7.25 (m, 5 H), 6.96 (s, 1 H), 6.92 (s, 2 H), 2.22 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.8, 149.2, 141.0, 139.2, 139.0, 138.2, 135.2, 132.8, 130.6, 128.9, 128.5, 127.5, 126.9, 21.2; HRMS calcd for $C_{19}H_{17}OS$ $[M+H]^+$ 293.0995; found: 293.0990.



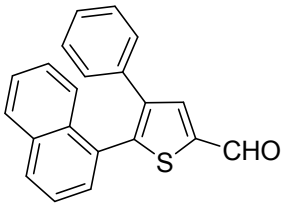
5-(3-methoxyphenyl)-4-phenylthiophene-2-carbaldehyde (2ag)

Yellow oil (47.6 mg, 54% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.89 (s, 1 H), 7.76 (s, 1 H), 7.32-7.24 (m, 5 H), 7.17-7.13 (m, 3 H), 7.08-7.05 (m, 1 H), 2.27 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.7, 148.9, 141.2, 139.3, 139.0, 138.4, 135.1, 132.9, 129.7, 129.6, 128.9, 128.5, 127.5, 126.2, 21.2; HRMS calcd for $C_{18}H_{15}O_2S$ $[M+H]^+$ 295.0788; found: 295.0792.



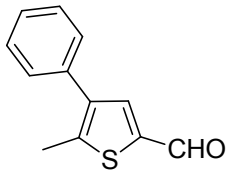
5-(4-chlorophenyl)-4-phenylthiophene-2-carbaldehyde (2ah)

Yellow oil (42.1 mg, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.91 (s, 1 H), 7.78 (s, 1 H), 7.34-7.32 (m, 3 H), 7.28-7.23 (m, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.7, 147.0, 141.6, 139.9, 139.0, 135.0, 134.8, 131.5, 130.4, 129.0, 128.8, 128.8, 127.8; HRMS calcd for $C_{17}H_{12}ClOS$ $[M+H]^+$ 299.0292; found: 299.0297.



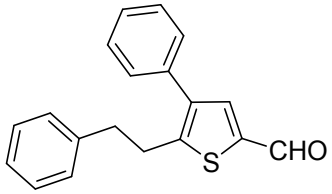
5-(naphthalen-1-yl)-4-phenylthiophene-2-carbaldehyde (2ai)

Yellow soild (49.0 mg, 52% yield), melting point: 119-121 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 10.00 (s, 1 H), 7.99 (s, 1 H), 7.90-7.84 (m, 2 H), 7.71-7.69 (d, *J* = 8 Hz, 1 H), 7.50-7.41 (m, 3 H), 7.35-7.33 (m, 1 H), 7.14-7.08 (m, 5 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.9, 146.5, 142.5, 141.5, 137.7, 134.8, 133.5, 131.5, 130.6, 129.9, 129.1, 128.4, 128.3, 127.9, 127.4, 126.7, 126.2, 125.5, 125.1; HRMS calcd for $C_{21}H_{15}OS$ $[M+H]^+$ 315.0838; found: 315.0844.



5-methyl-4-phenylthiophene-2-carbaldehyde (2aj)

Yellow soild (33.3 mg, 55% yield), melting point: 67-70 °C. ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.84 (s, 1 H), 7.70 (s, 1 H), 7.47-7.43 (m, 2 H), 7.38-7.35 (m, 3 H)，2.57 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.6, 146.0, 140.6, 139.7, 138.5, 135.1, 128.6, 128.5, 127.5, 15.3; HRMS calcd for $C_{12}H_{11}OS$ $[M+H]^+$ 203.0525; found: 203.0529.



5-phenethyl-4-phenylthiophene-2-carbaldehyde (2ak)

Yellow oil (41.2 mg, 47% yield). ^{1}H NMR (400 MHz, $CDCl_3$, ppm): δ = 9.85 (s, 1 H), 7.65 (s, 1 H), 7.43-7.35 (m, 3 H), 7.26-7.19 (m, 5 H)，7.09-7.08 (d, *J* = 4 Hz, 2 H), 3.22-3.18 (m, 2 H),

2.97-2.93 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$, ppm): δ = 182.6, 150.9, 140.9, 140.0, 138.4, 135.1, 128.6, 128.5, 128.3, 127.6, 126.4, 37.5, 31.1; HRMS calcd for $C_{19}H_{17}OS$ $[M+H]^+$ 292.0922; found: 292.0926.

VII. References

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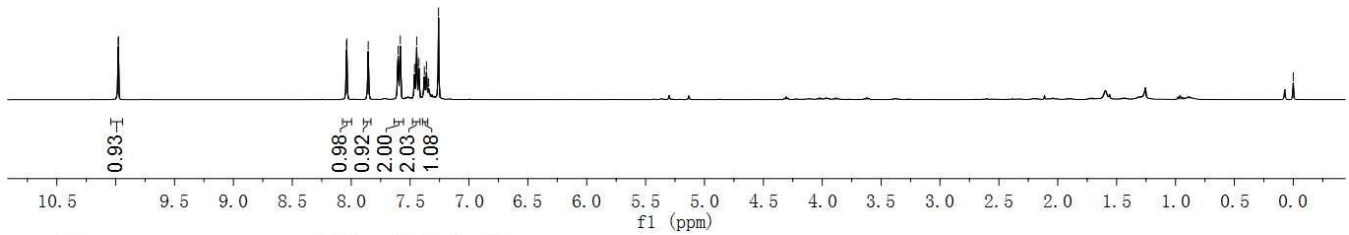
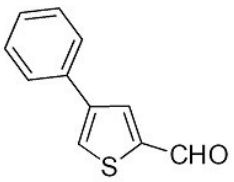
VII. NMR spectra

2a





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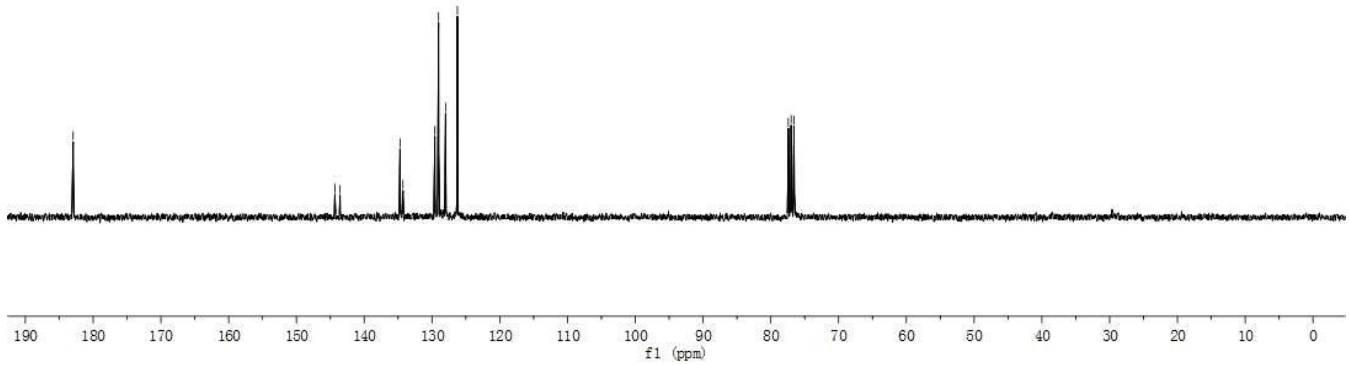

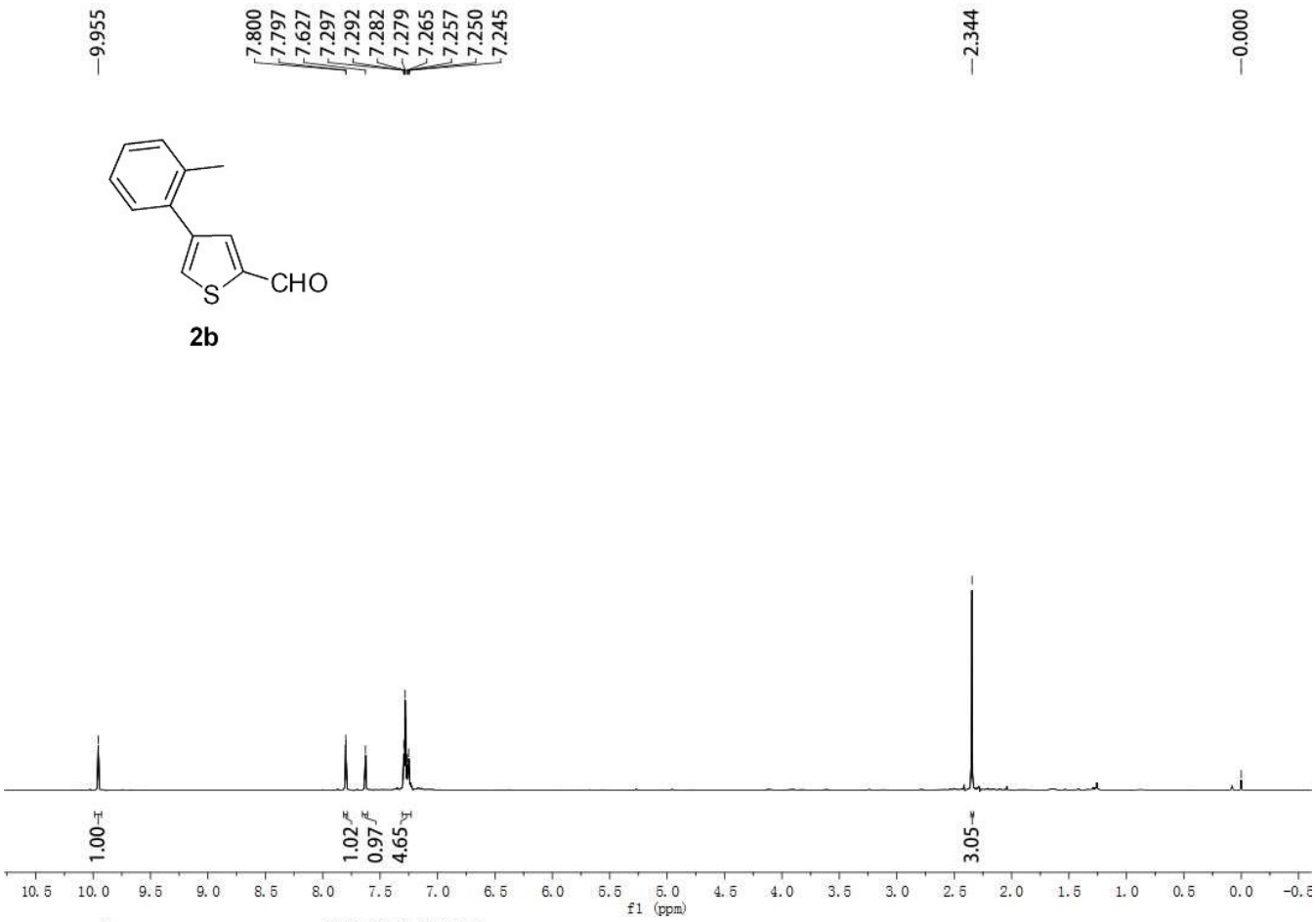
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0.000
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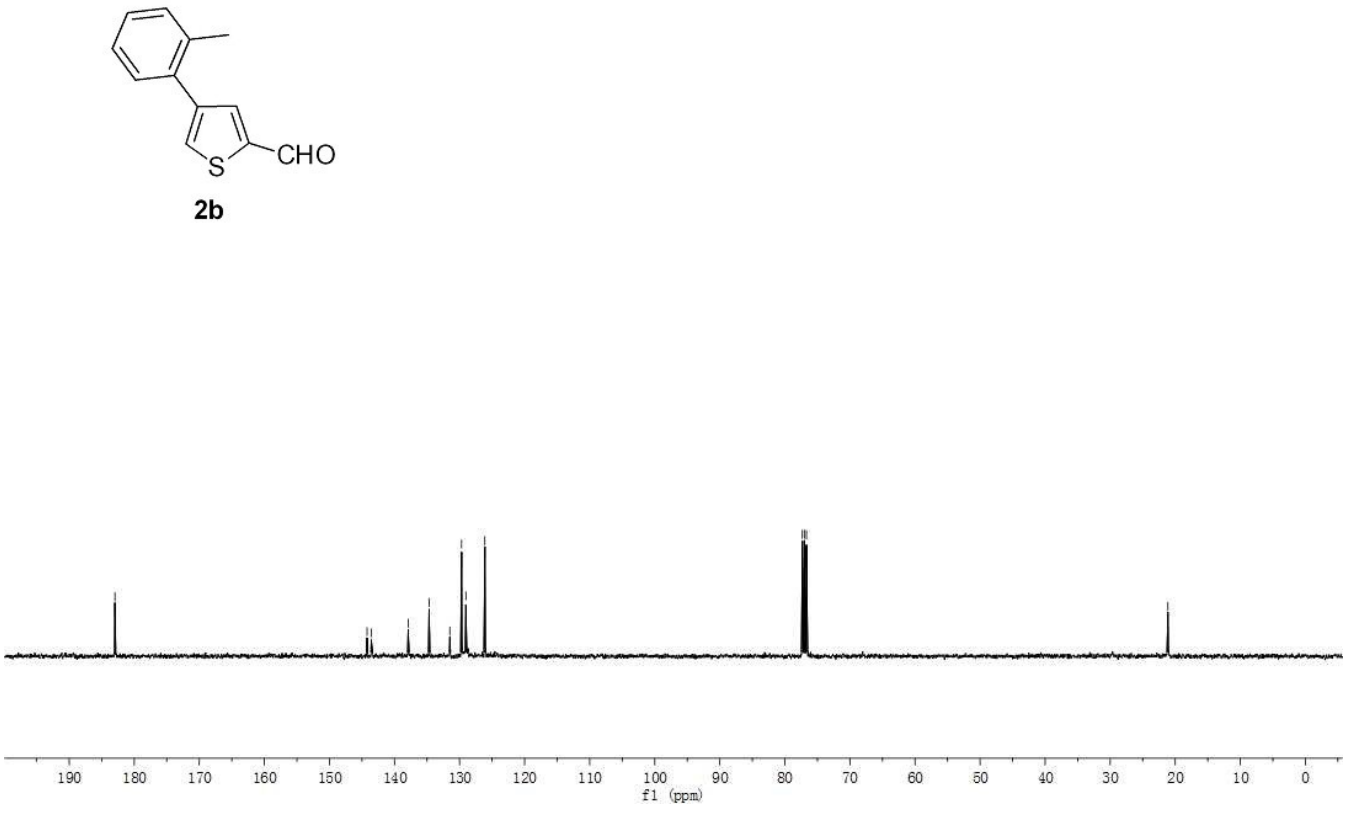
1.02
0.97
4.65
3.05
f1 (ppm)

2b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129.69
129.03
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77.00
76.68
21.13
CHO
S
f1 (ppm)

2b


9.958
9.956
8.007
8.003
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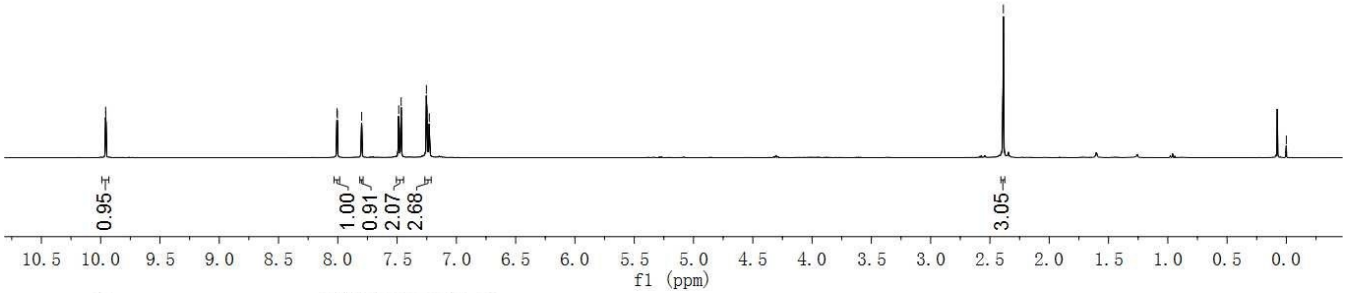
S

2c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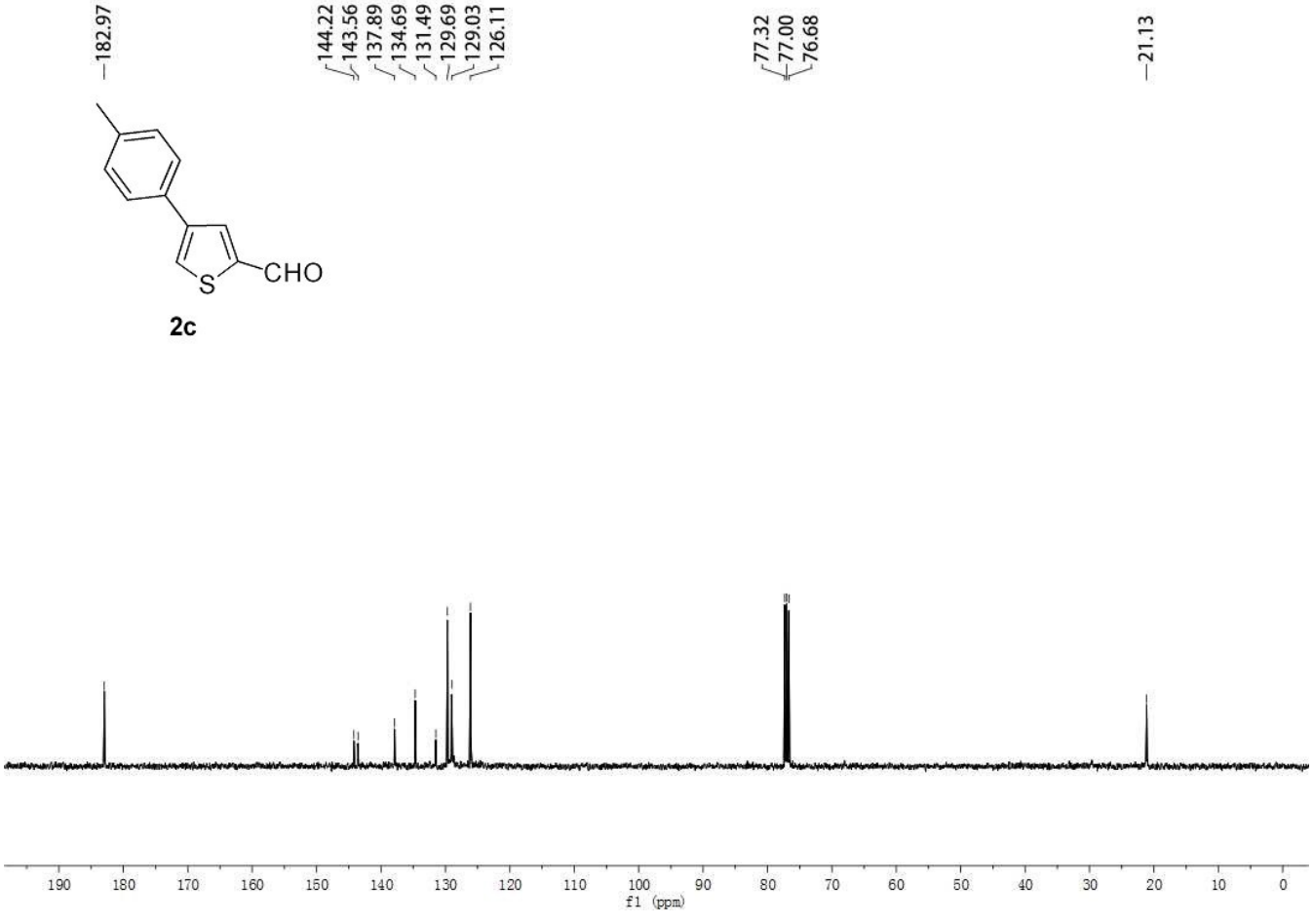
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143.56
137.89
134.69
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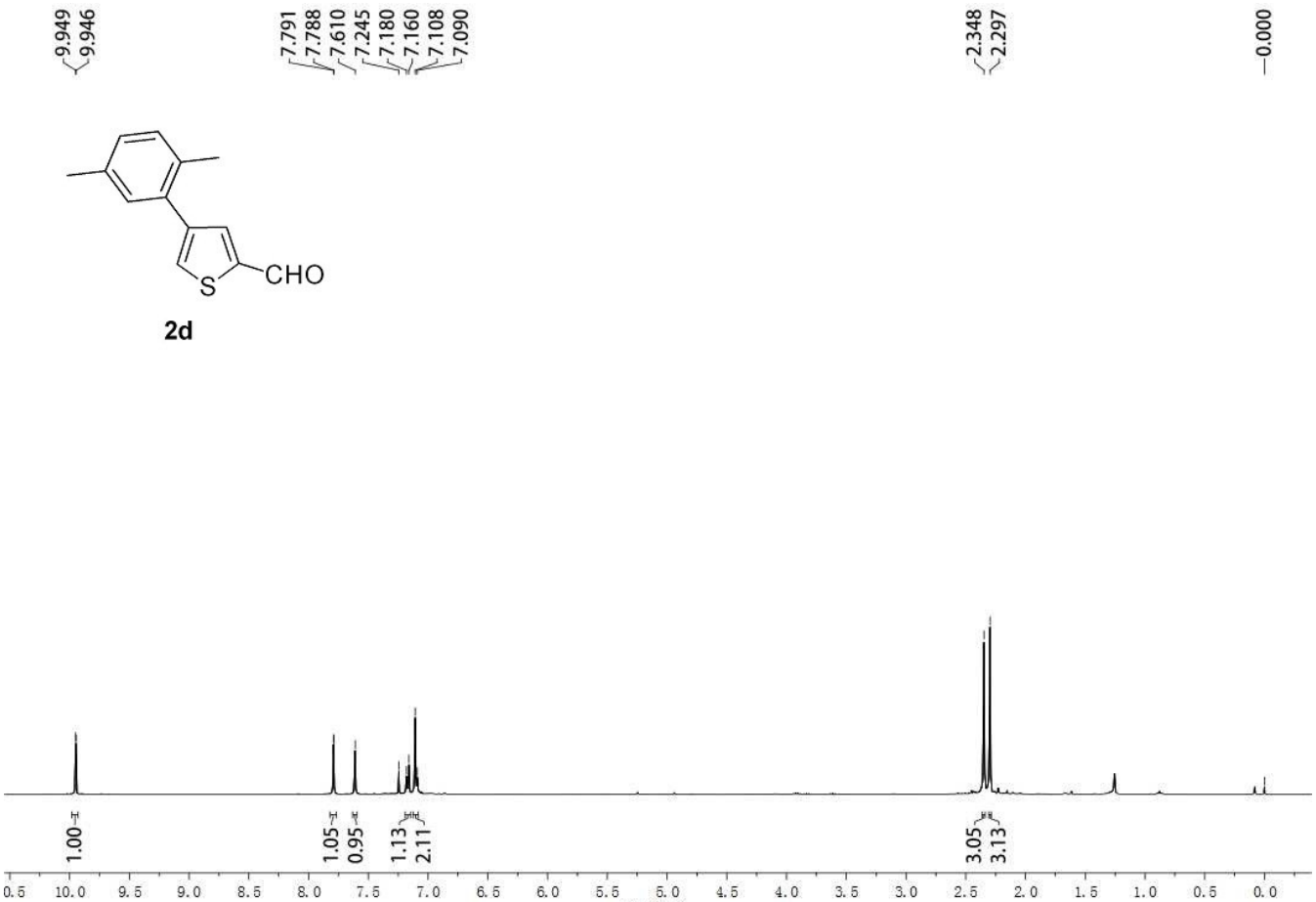
126.11
77.32
77.00
76.68
21.13
CHO
S
f1 (ppm)

2c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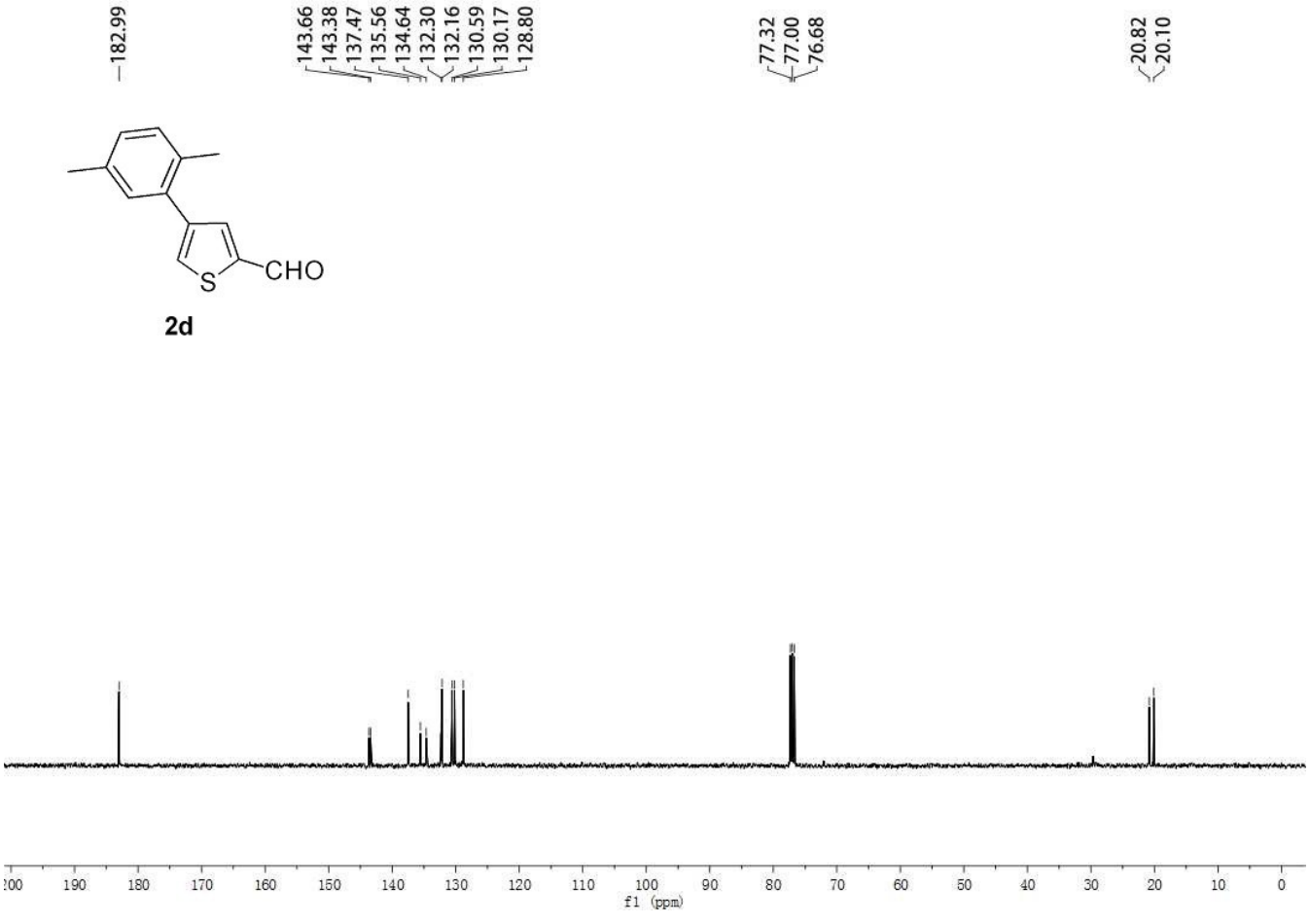
3.05
3.13
f1 (ppm)

2d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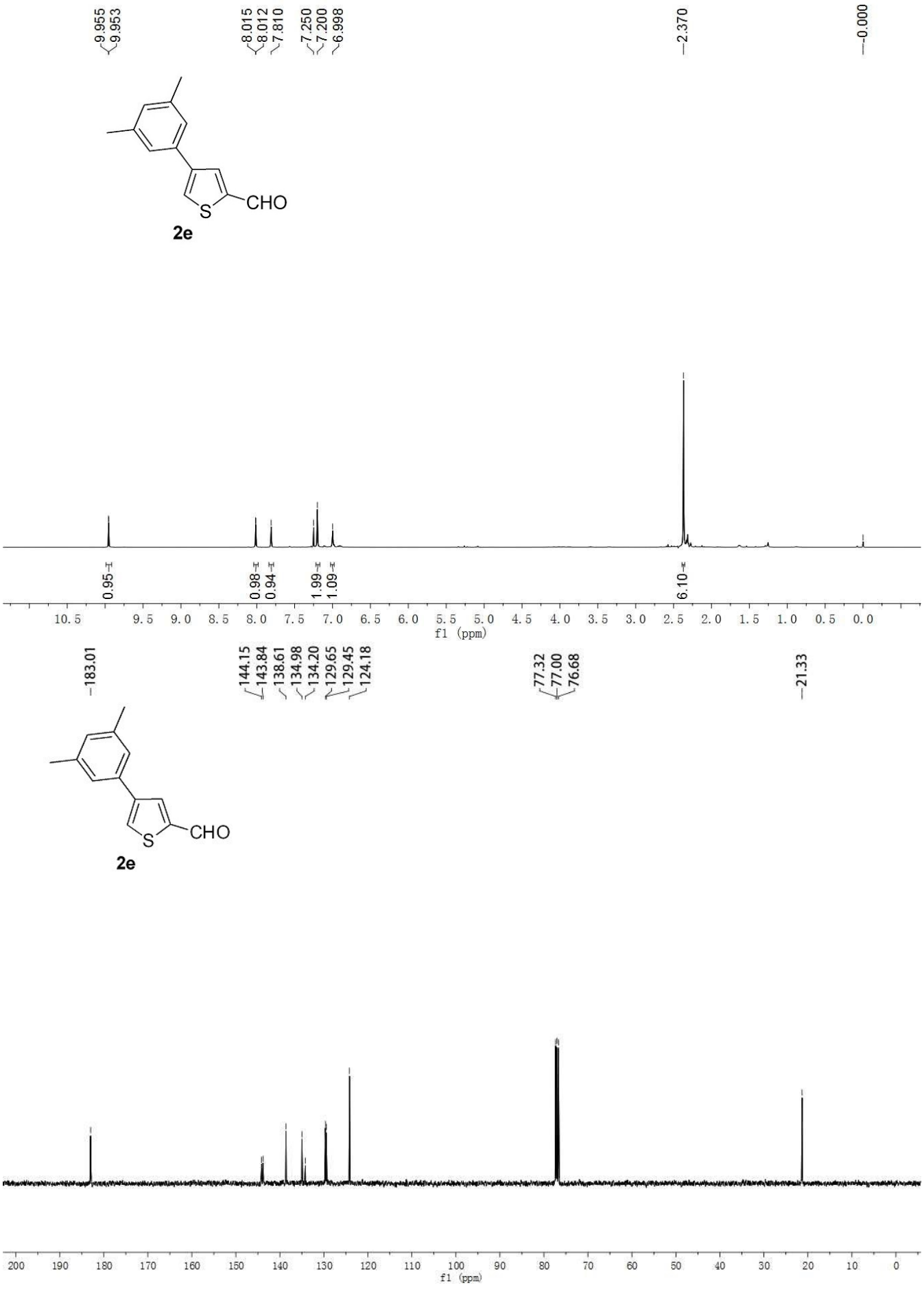
S
f1 (ppm)

2d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0.98
0.94
1.99
1.09
6.10
f1 (ppm)
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144.15
143.84
138.61
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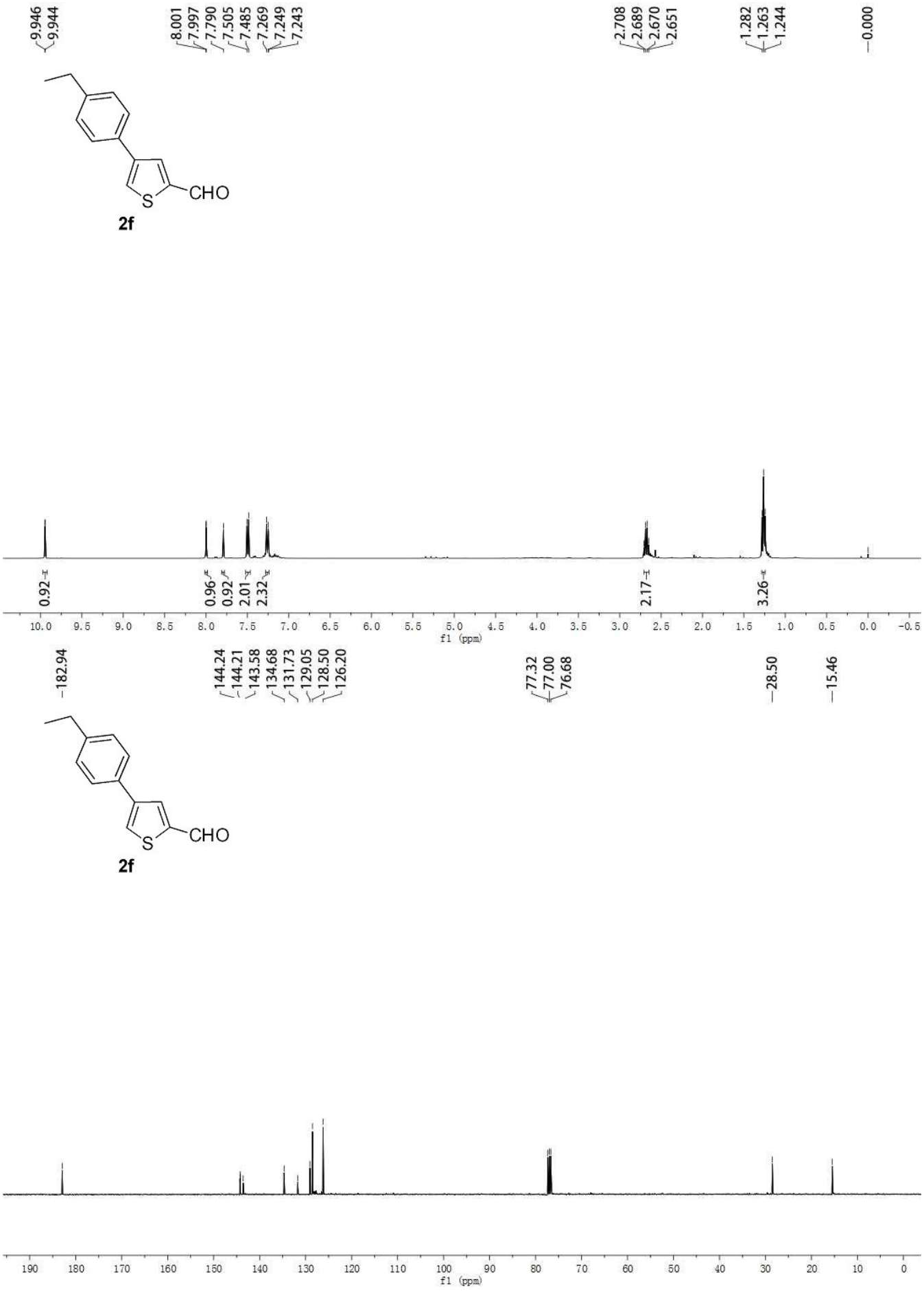
S
2e
f1 (ppm)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S
2f
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0.92
2.01
2.32
2.17
3.26
f1 (ppm)
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S
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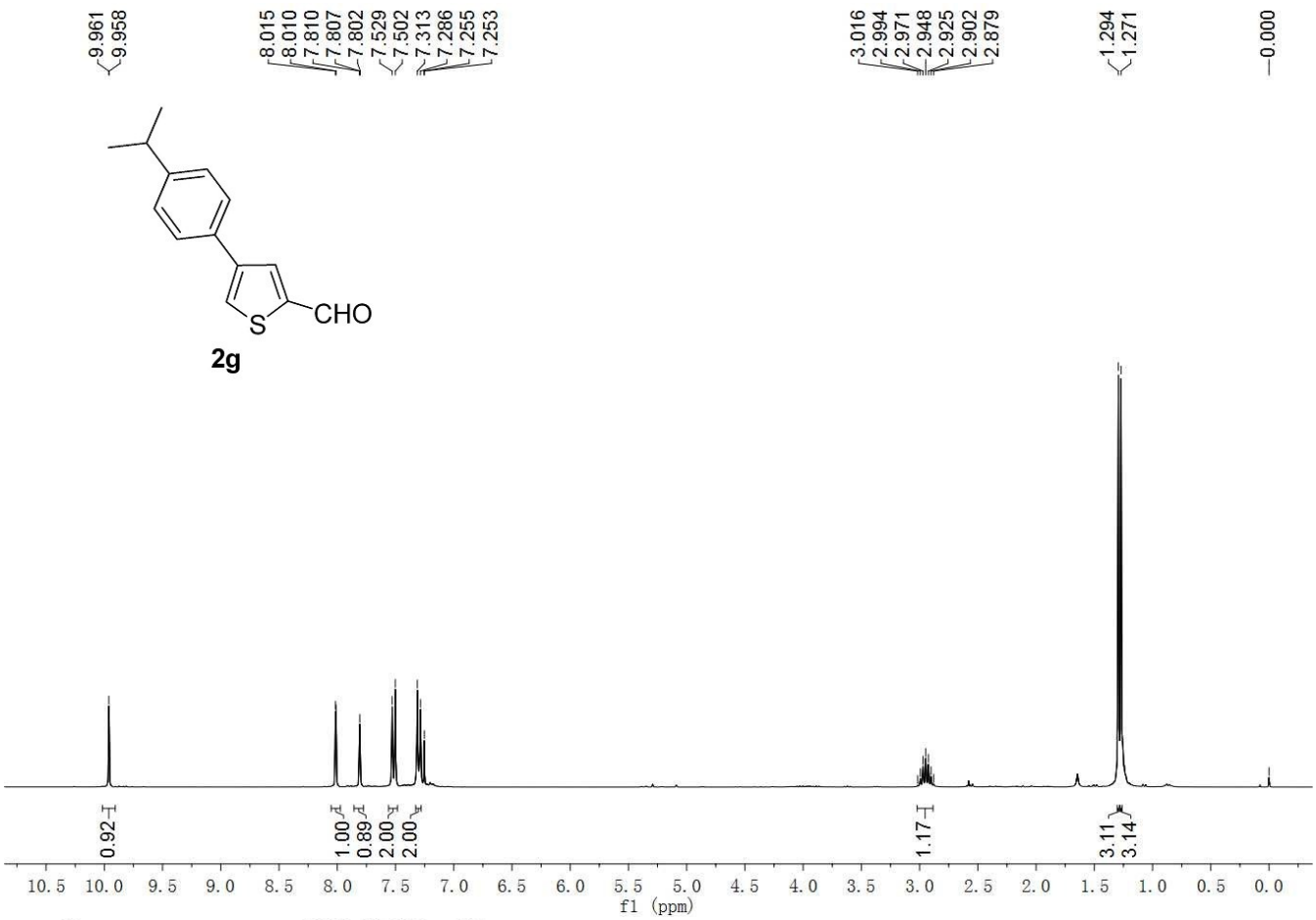
f1 (ppm)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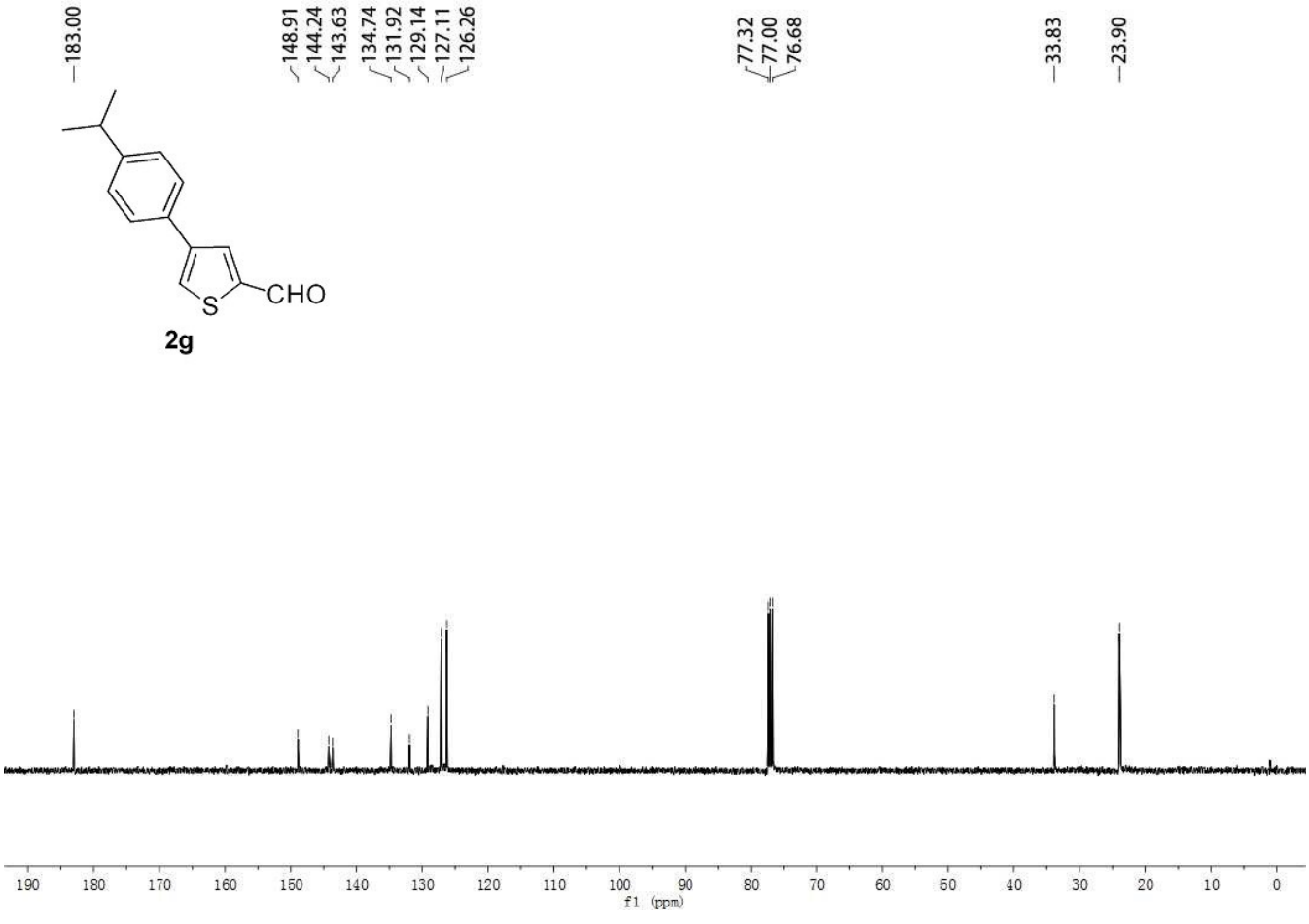
f1 (ppm)

2g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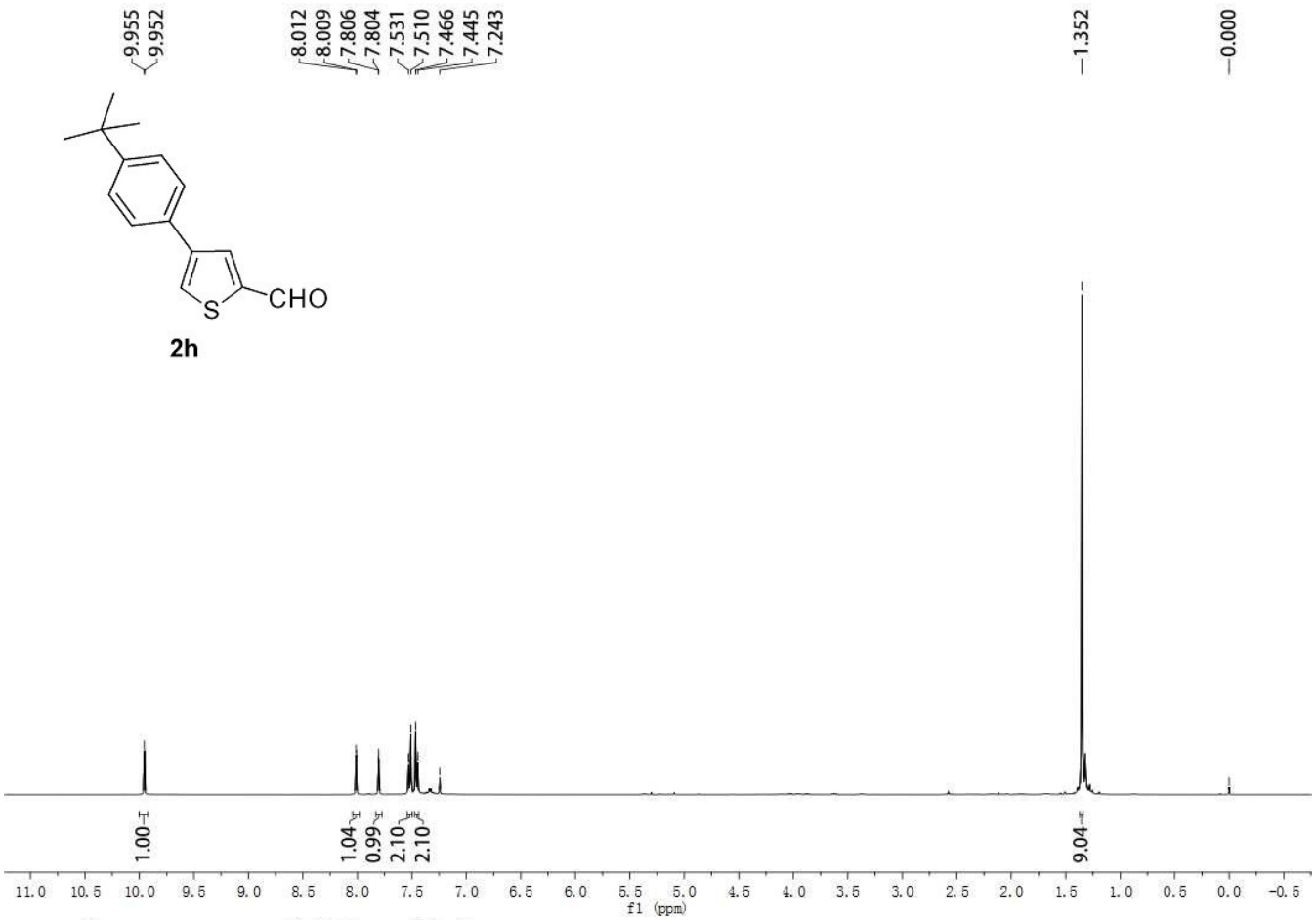
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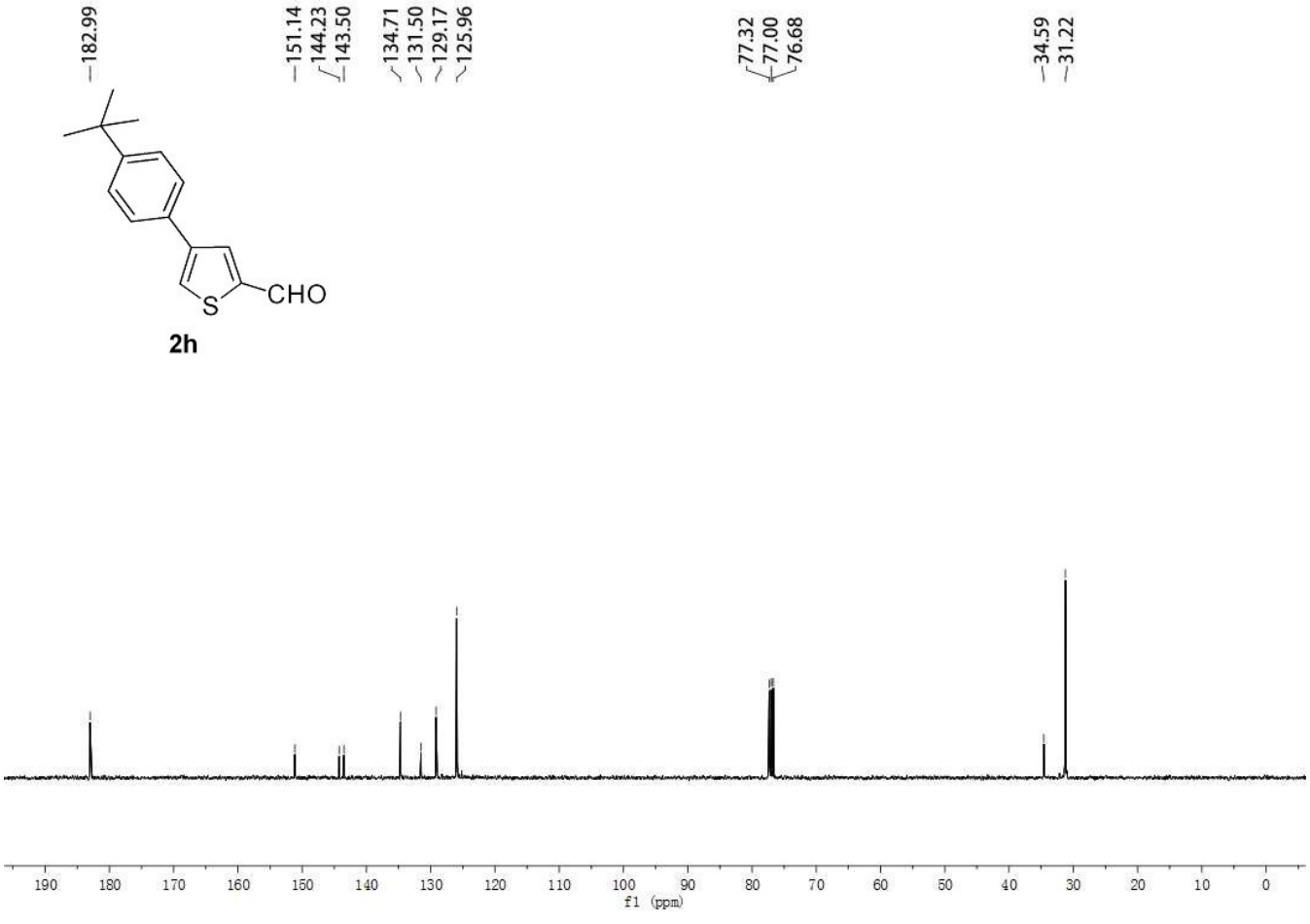
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S
2h
f1 (ppm)


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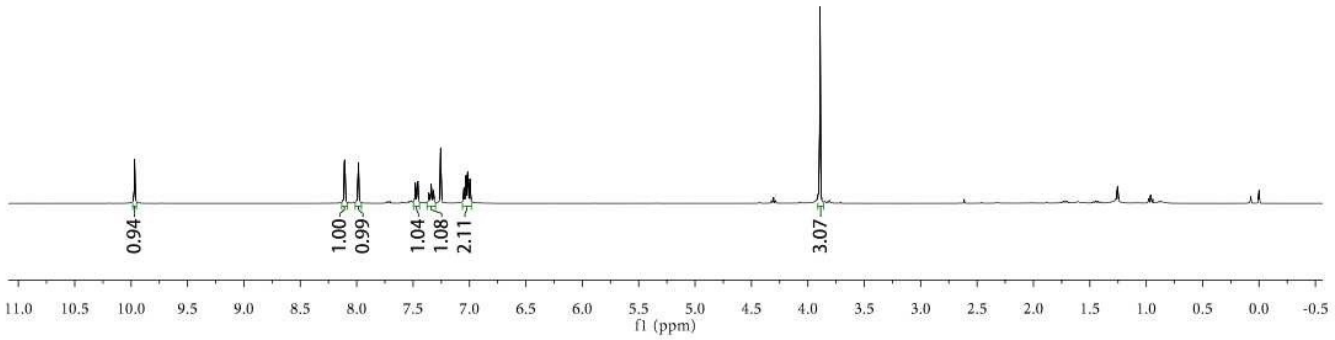
O
S
CHO

2i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f1 (ppm)


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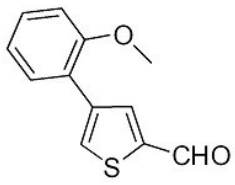
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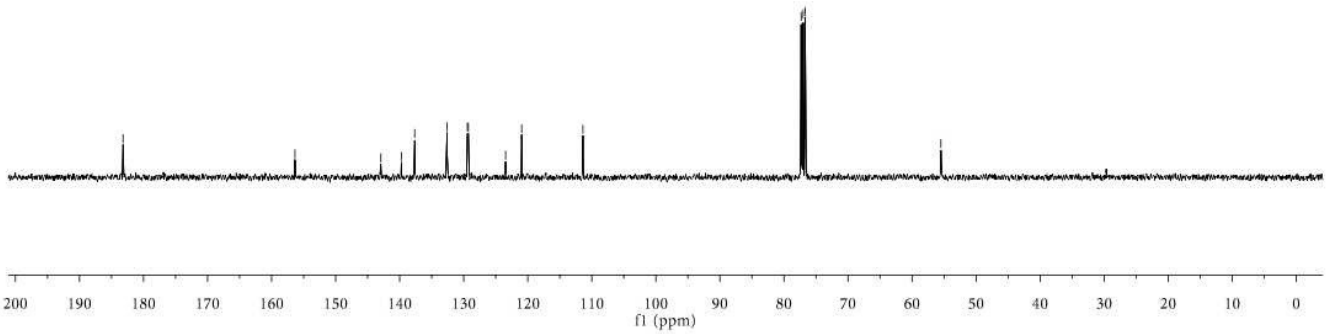
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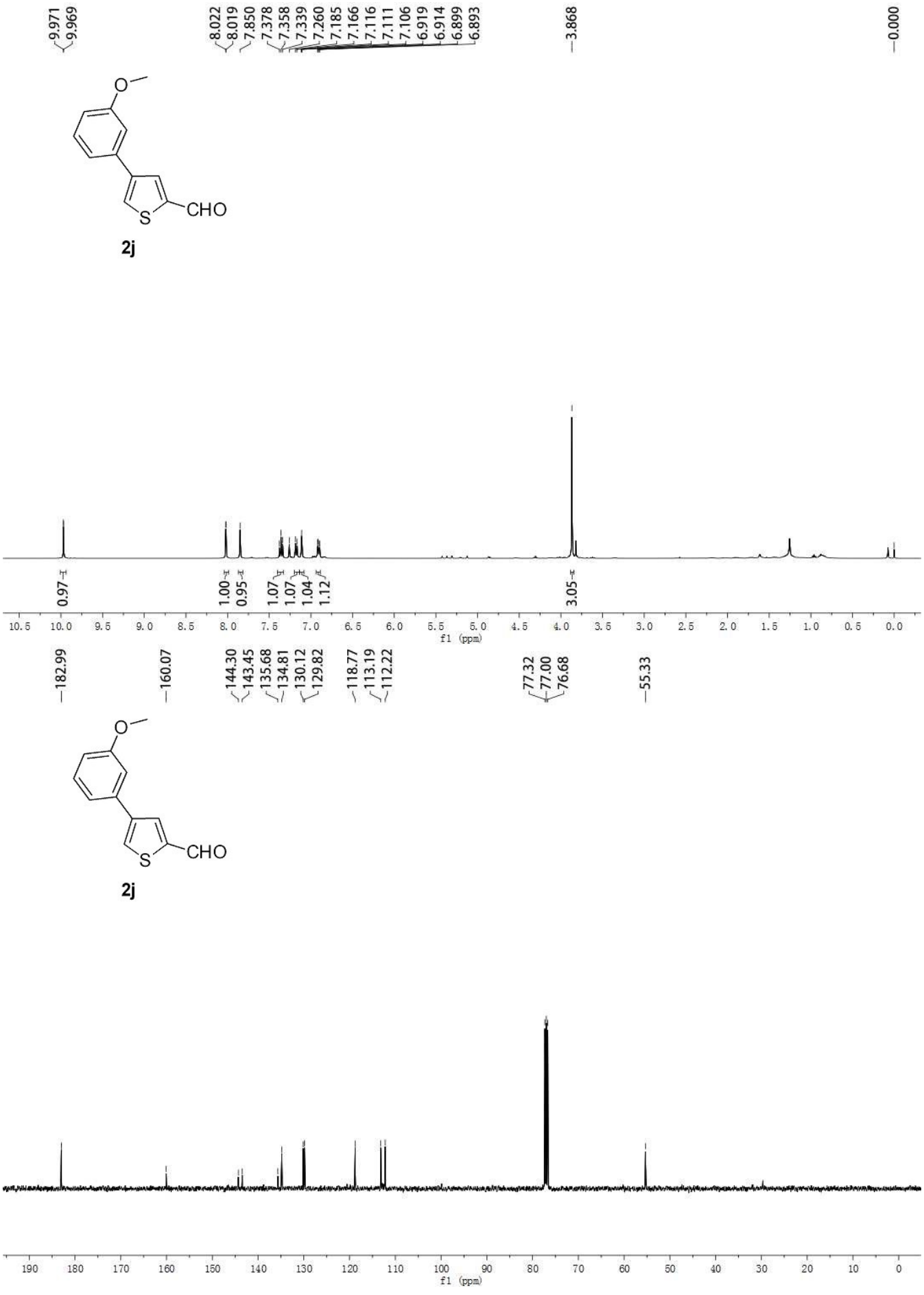
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O
CHO
S
2j
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1.07
1.07
1.04
1.12
3.05
f1 (ppm)
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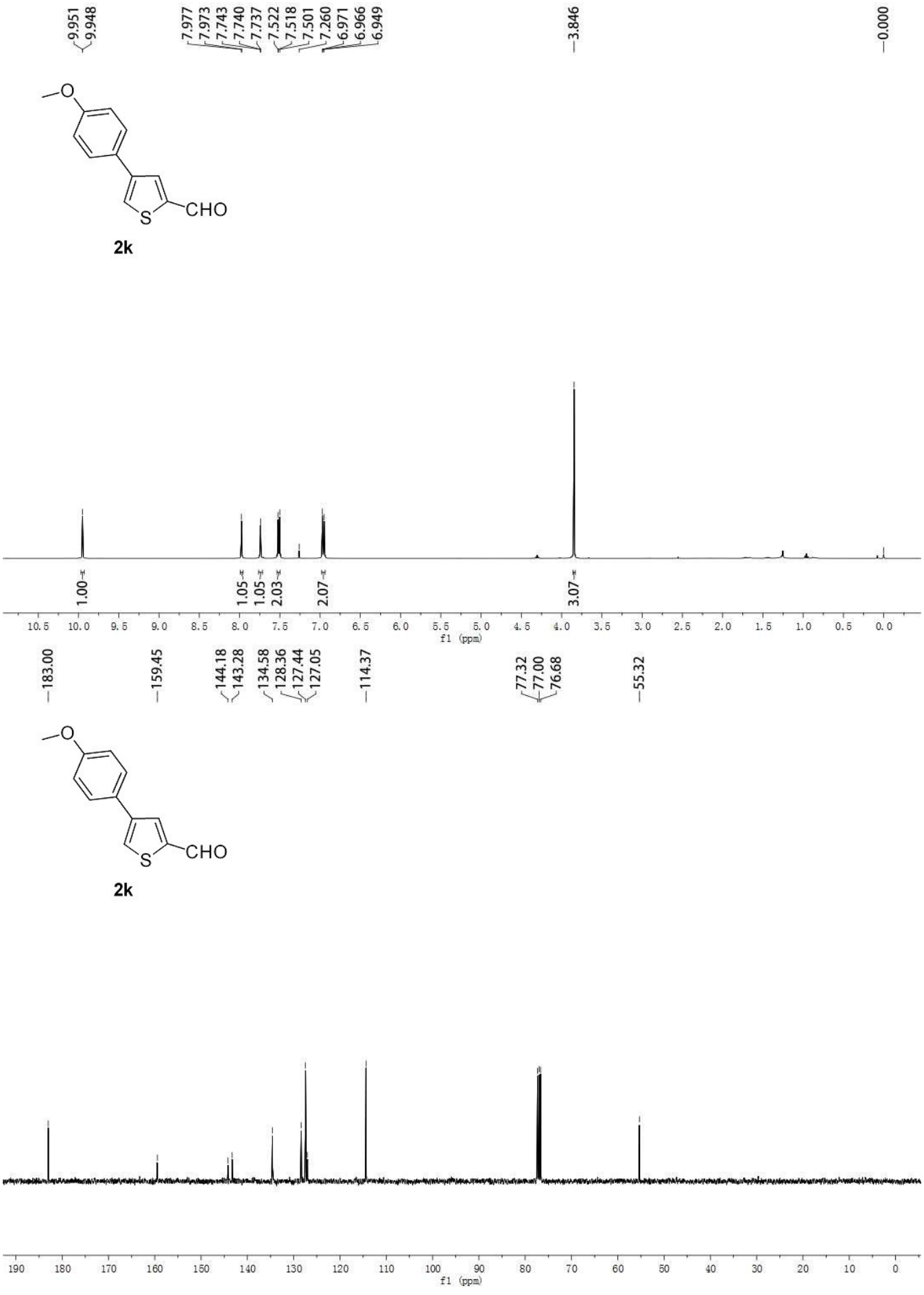
2j
f1 (ppm)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O
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1.05
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2.07
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55.32
O
S
CHO
2k
f1 (ppm)


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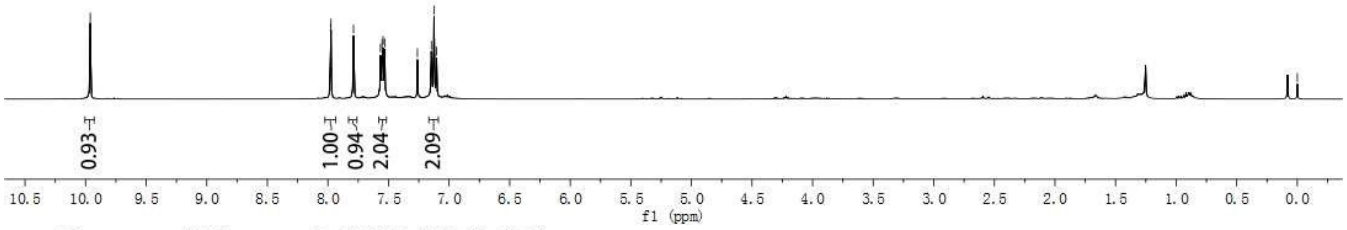
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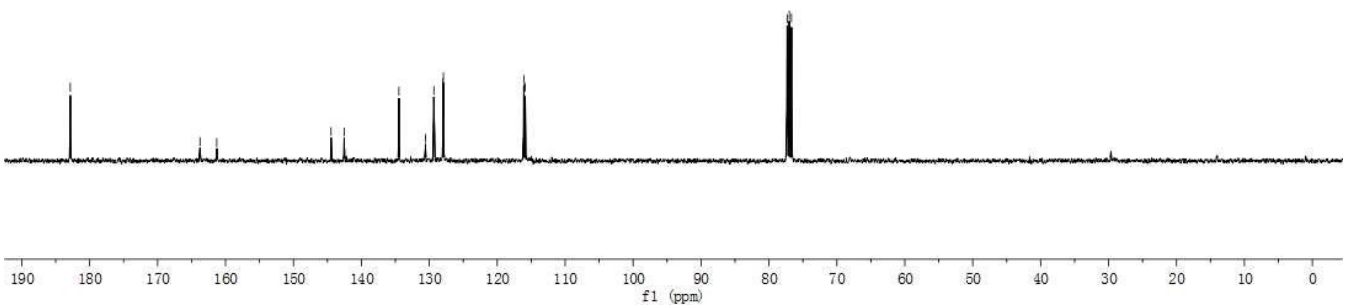
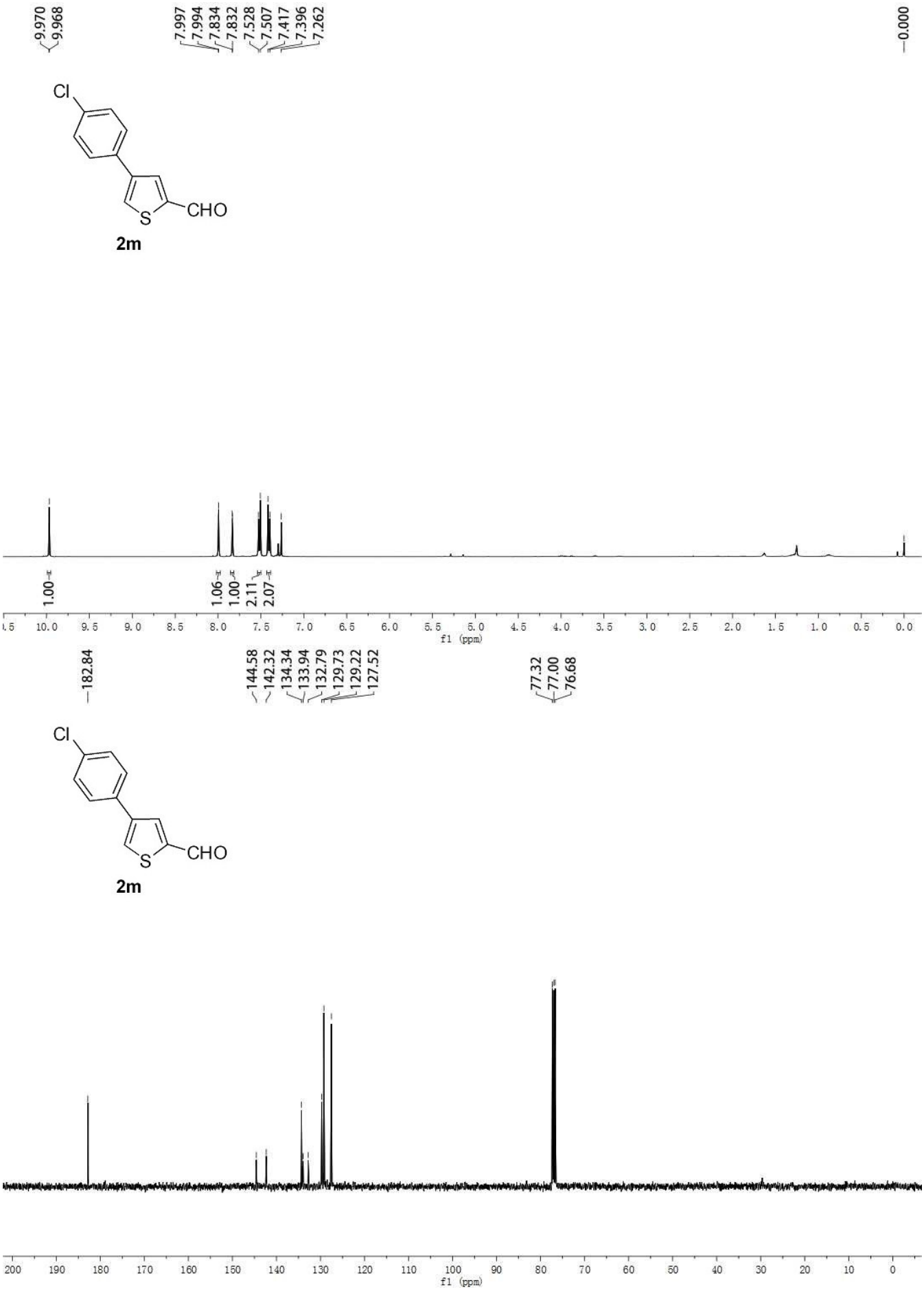
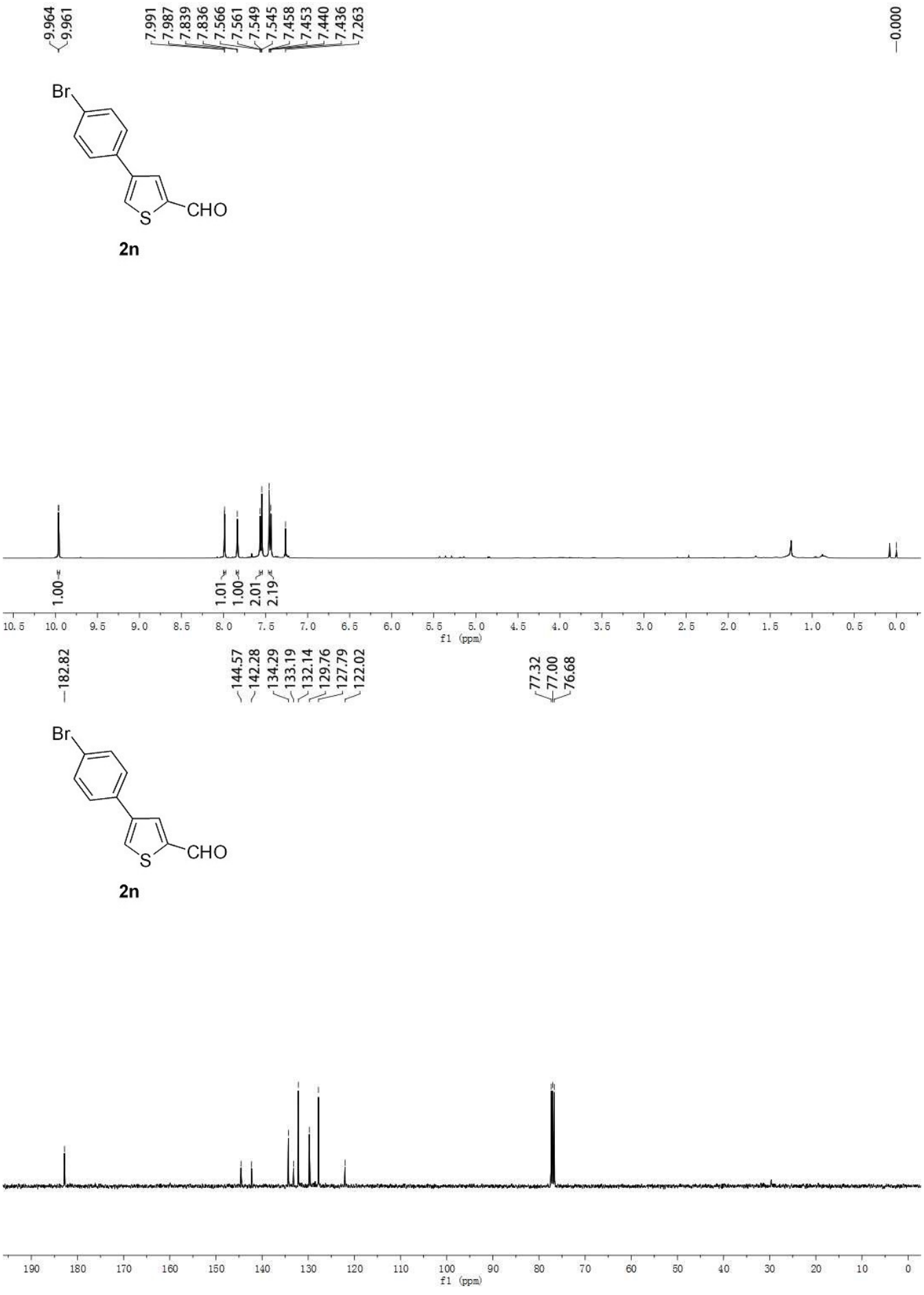
134.48
130.56
129.33
128.00
127.92
116.09
115.88
77.32
77.00
76.68


F
S
CHO

2l

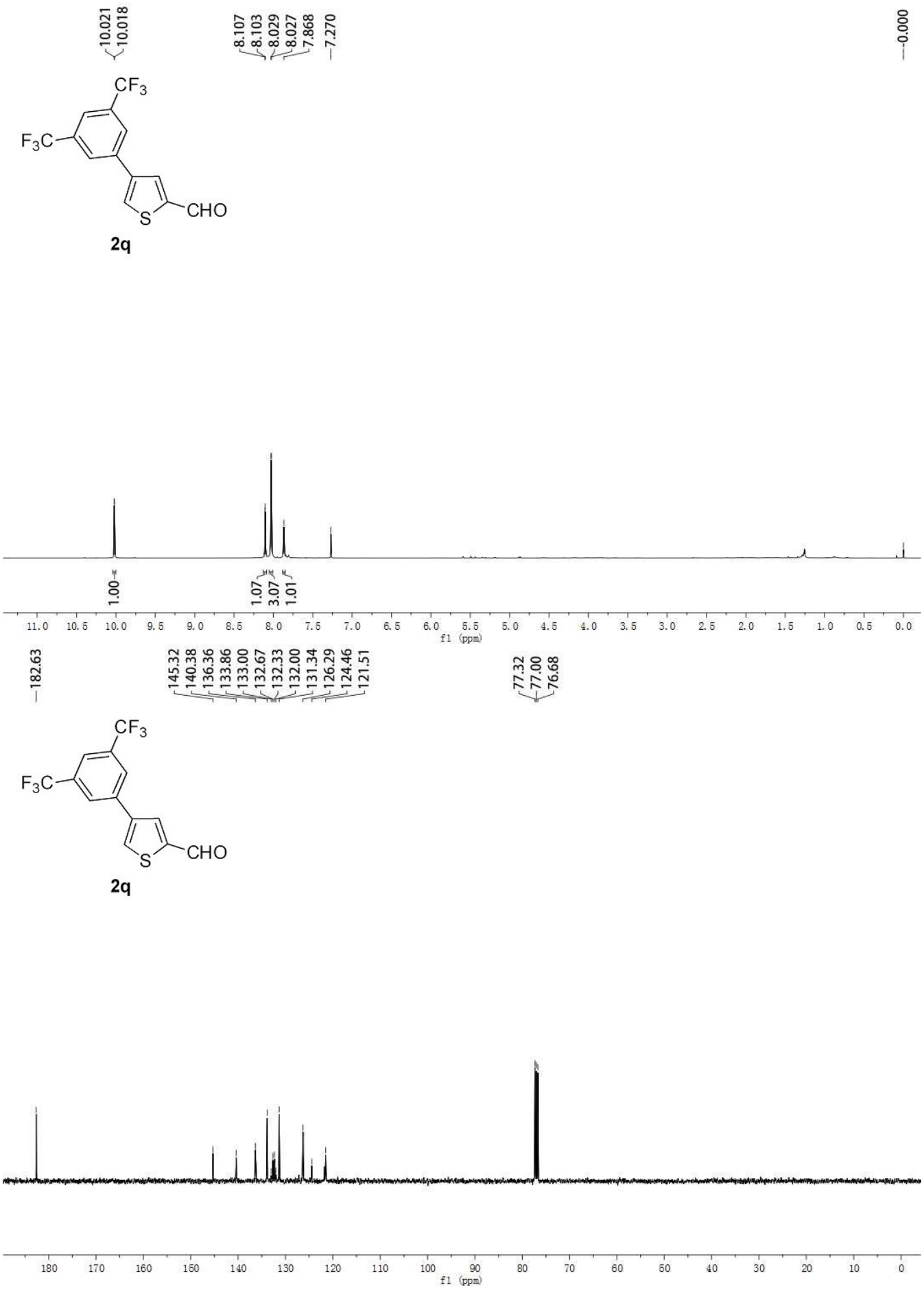

f1 (ppm)

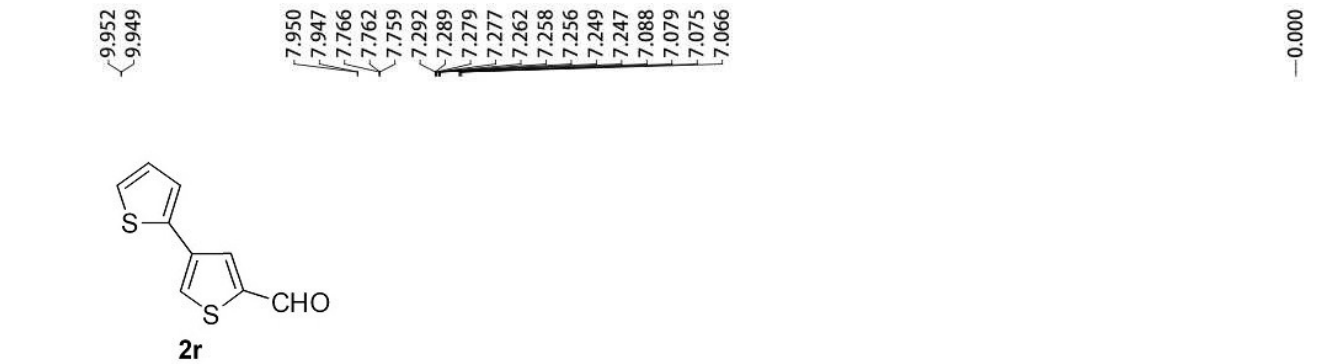

9.970
9.968
7.997
7.994
7.834
7.832
7.528
7.507
7.417
7.396
7.262
0.000
Cl
S
CHO
2m
1.00
1.06
1.00
2.11
2.07
f1 (ppm)
182.84
144.58
142.32
134.34
133.94
132.79
129.73
129.22
127.52
77.32
77.00
76.68
Cl
S
CHO
2m
f1 (ppm)


9.964
9.961
7.991
7.987
7.839
7.836
7.566
7.561
7.549
7.545
7.458
7.453
7.440
7.436
7.263
0.000
Br
S
CHO
2n
1.00
1.01
1.00
2.01
2.19
f1 (ppm)
182.82
144.57
142.28
134.29
133.19
132.14
129.76
127.79
122.02
77.32
77.00
76.68
Br
S
CHO
2n
f1 (ppm)


9.944
7.973
7.970
7.723
7.384
7.363
7.254
6.873
6.853
3.862
2.269
0.000
O
CHO
S
2o
0.88
0.95
1.02
1.92
1.00
3.03
3.06
f1 (ppm)
183.04
157.69
144.08
143.53
134.72
128.65
128.24
127.24
126.56
124.70
110.15
77.32
77.00
76.68
55.38
16.33
O
CHO
S
2o
f1 (ppm)

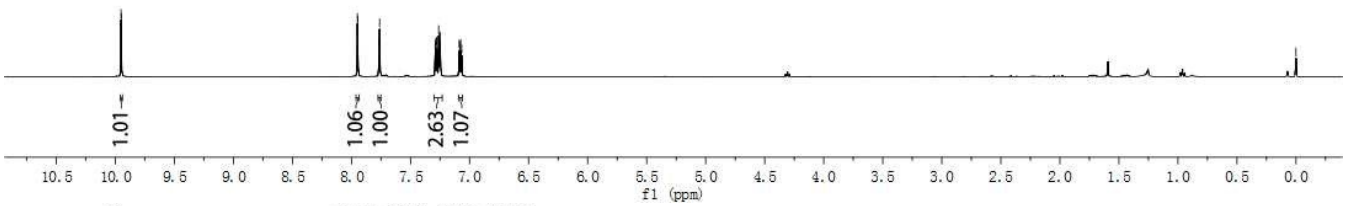

9.974
9.971
7.984
7.981
7.859
7.856
7.852
7.678
7.672
7.517
7.497
7.428
7.422
7.407
7.401
7.262
0.000
Cl
Cl
S
CHO
2p
1.00
1.06
1.00
1.07
1.15
1.19
f1 (ppm)
182.69
144.87
141.08
134.29
134.03
133.27
132.11
131.01
130.26
128.15
125.49
77.32
77.00
76.68
Cl
Cl
S
CHO
2p
f1 (ppm)


10.021
10.018
8.107
8.103
8.029
8.027
7.868
7.270
0.000
CF3
F3C
S
CHO
2q
1.00
1.07
3.07
1.01
f1 (ppm)
182.63
145.32
140.38
136.36
133.86
133.00
132.67
132.33
132.00
131.34
126.29
124.46
121.51
77.32
77.00
76.68
CF3
F3C
S
CHO
2q
f1 (ppm)

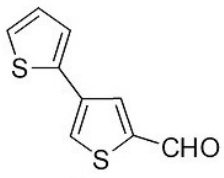

9.952
9.949
7.950
7.947
7.766
7.762
7.759
7.292
7.289
7.279
7.277
7.262
7.258
7.256
7.249
7.247
7.088
7.079
7.075
7.066
0.000

S
S
CHO

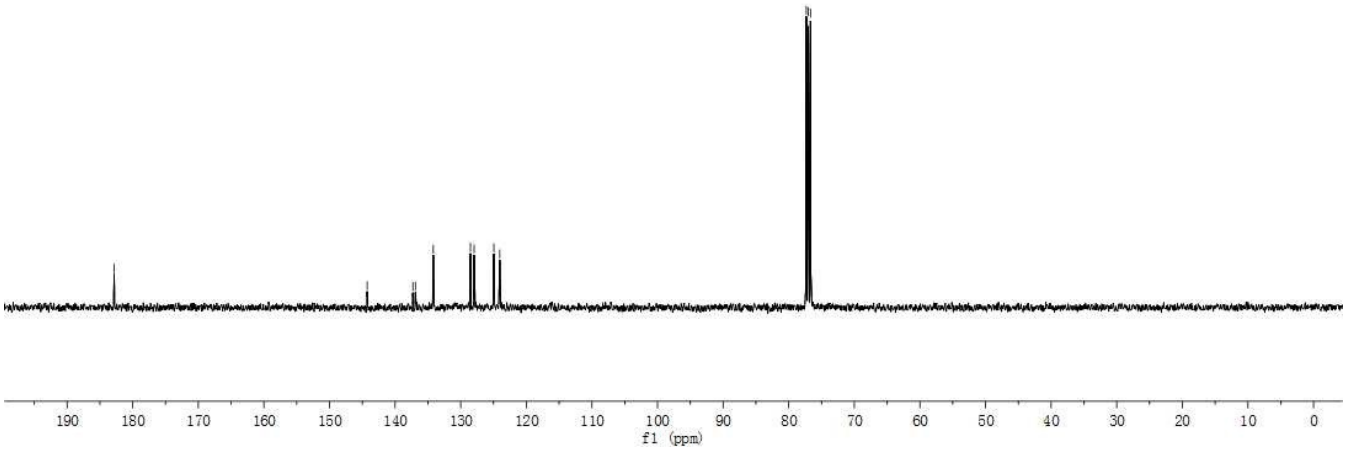
2r


1.01
1.06
1.00
2.63
1.07
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

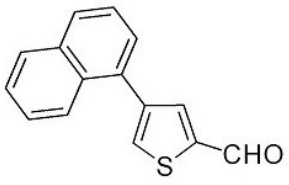

182.82
144.26
137.29
136.90
134.18
128.52
127.96
124.95
124.05
77.32
77.00
76.68


S
S
CHO

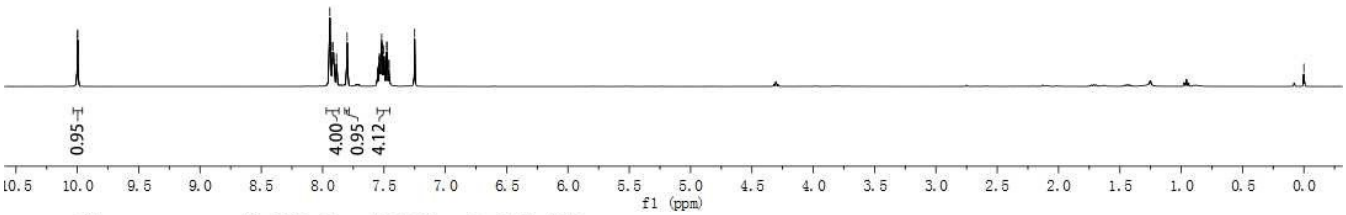
2r


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

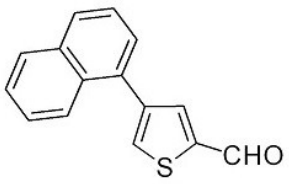

9.999
9.996
7.944
7.941
7.918
7.917
7.907
7.887
7.800
7.554
7.551
7.540
7.537
7.534
7.523
7.519
7.514
7.503
7.498
7.495
7.478
7.475
7.460
7.458
7.250
0.000


S
CHO

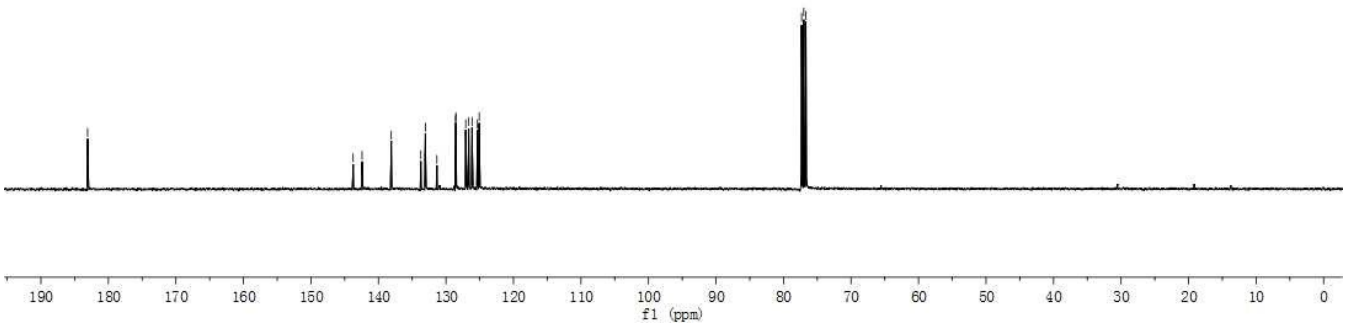
2s

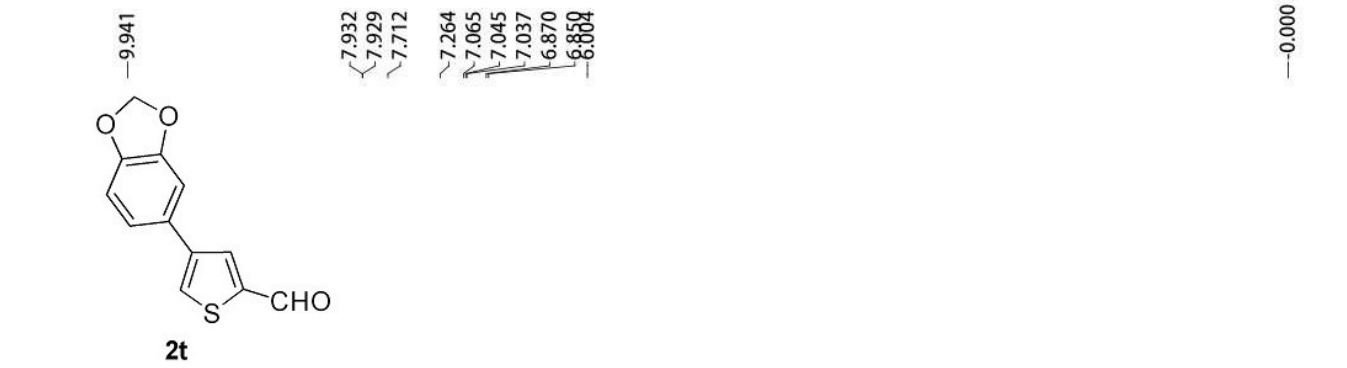

0.95
4.00
0.95
4.12
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


183.08
143.74
142.42
138.10
133.72
133.11
133.05
131.34
128.59
128.51
127.07
126.62
126.13
125.33
125.07
77.32
77.00
76.68

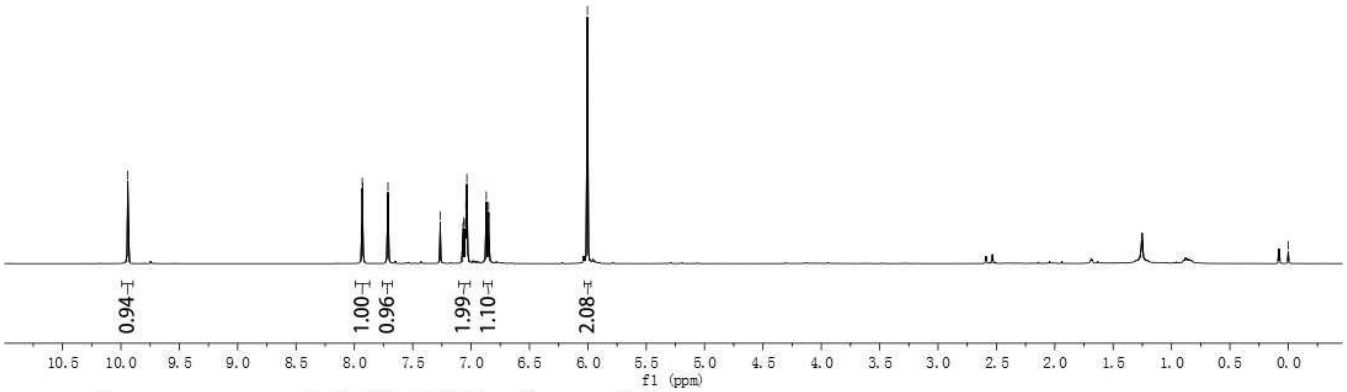

S
CHO

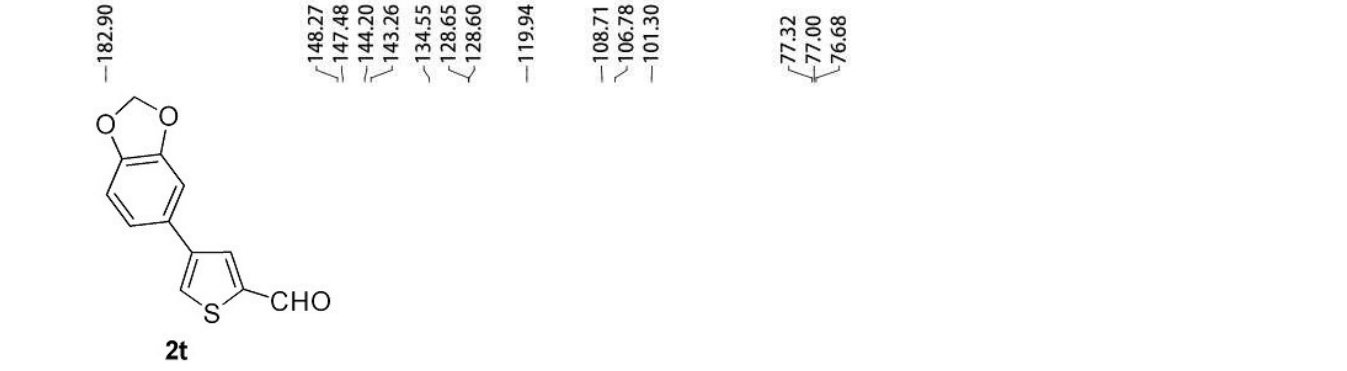
2s


190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

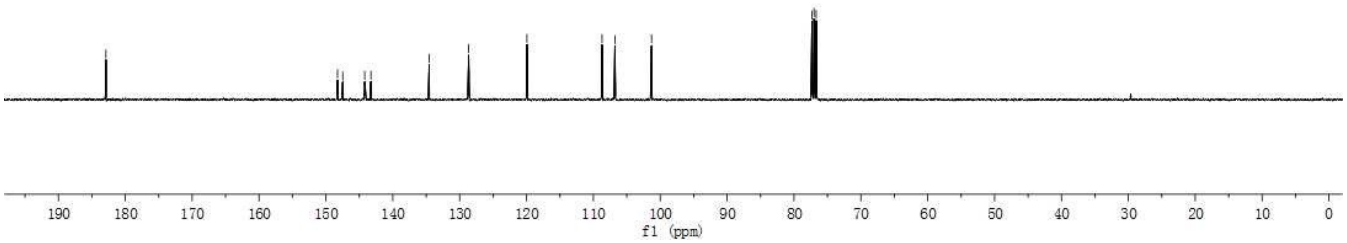

9.941
7.932
7.929
7.712
7.264
7.065
7.045
7.037
6.870
6.850
6.004
0.000
O
O
S
CHO

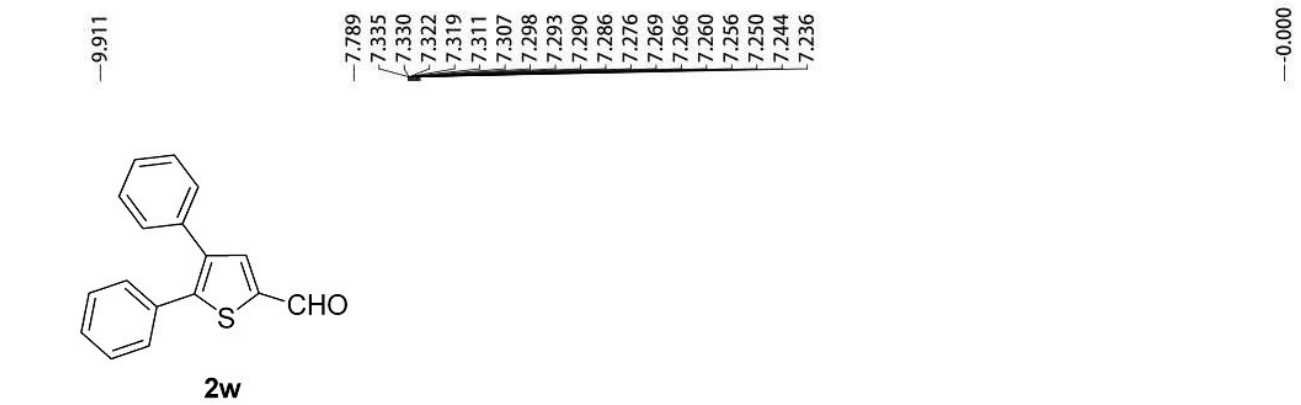
2t


0.94
1.00
0.96
1.99
1.10
2.08
f1 (ppm)

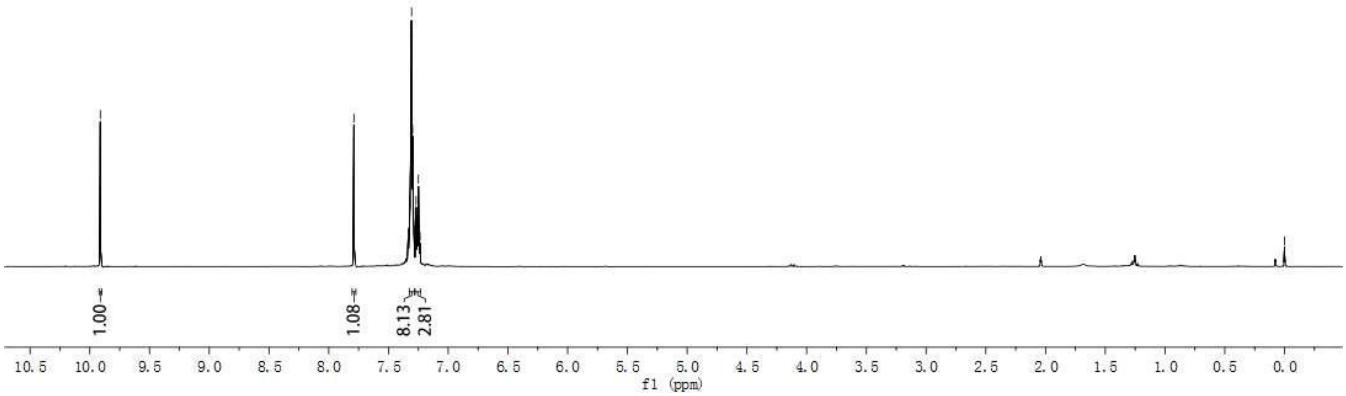

182.90
148.27
147.48
144.20
143.26
134.55
128.65
128.60
119.94
108.71
106.78
101.30
77.32
77.00
76.68
O
O
S
CHO

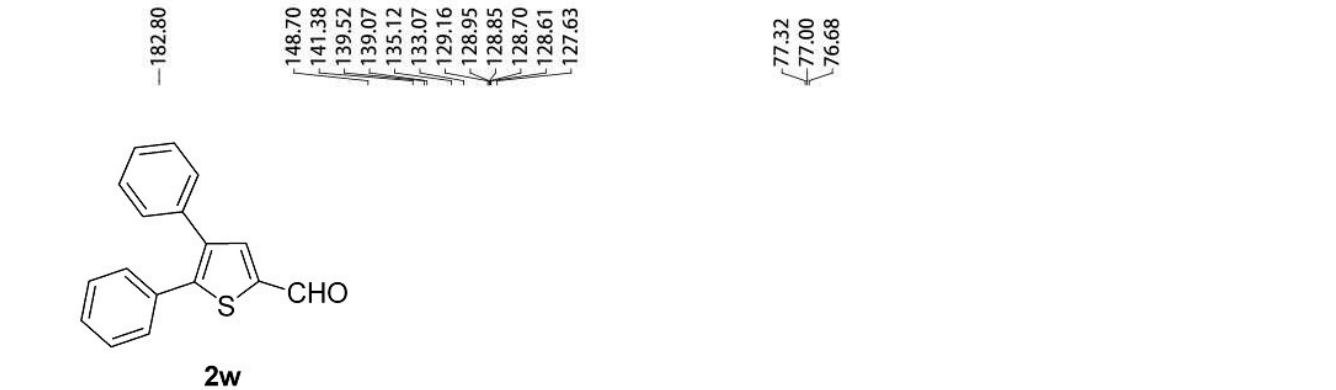
2t


f1 (ppm)

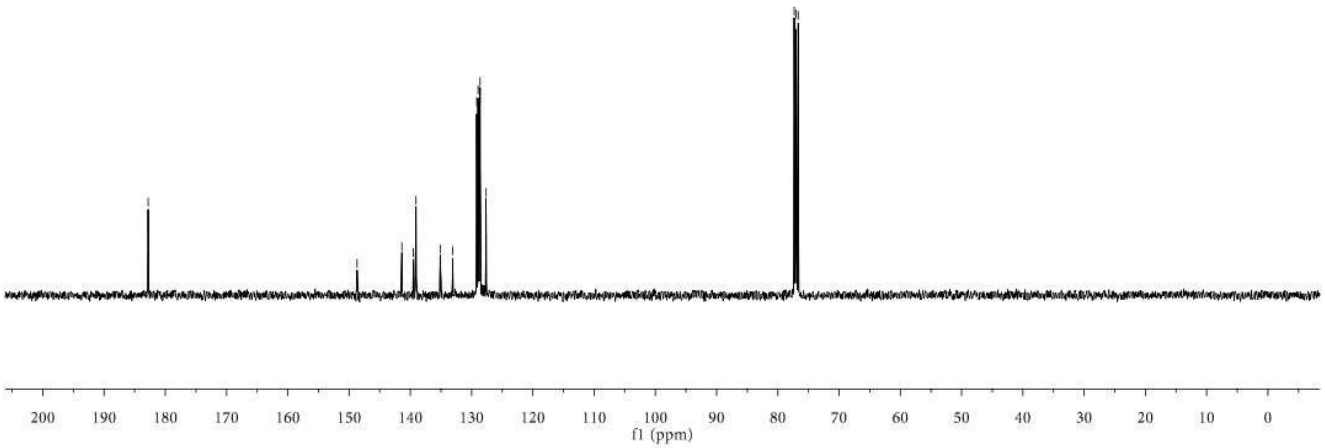

9.911
7.789
7.335
7.330
7.322
7.319
7.311
7.307
7.298
7.293
7.290
7.286
7.276
7.269
7.266
7.260
7.256
7.250
7.244
7.236
0.000
CHO
S

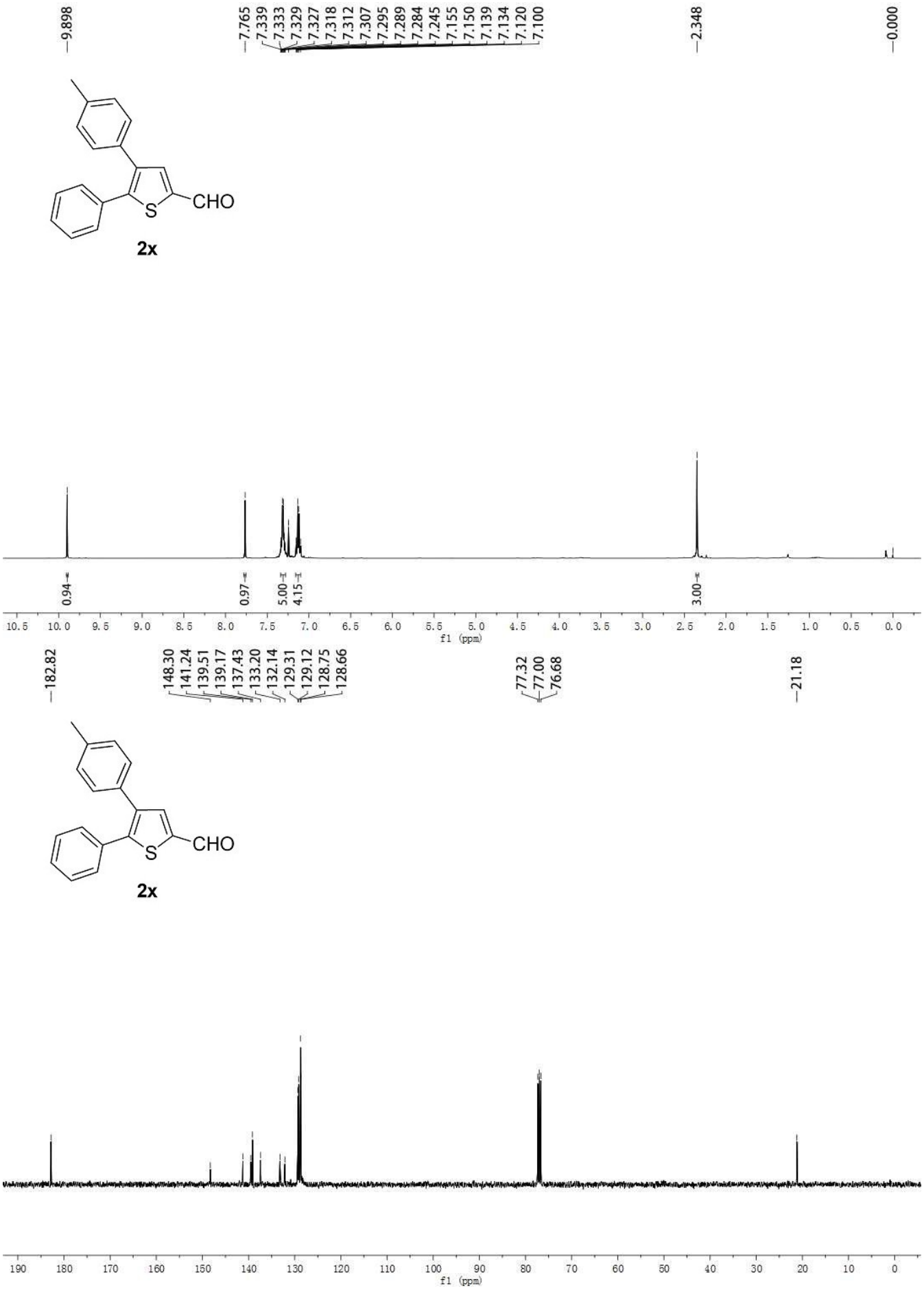
2w

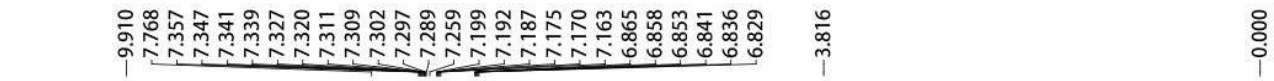

1.00
1.08
8.13
2.81
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

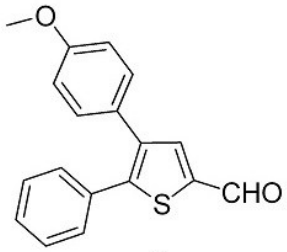

182.80
148.70
141.38
139.52
139.07
135.12
133.07
129.16
128.95
128.85
128.70
128.61
127.63
77.32
77.00
76.68
CHO
S

2w

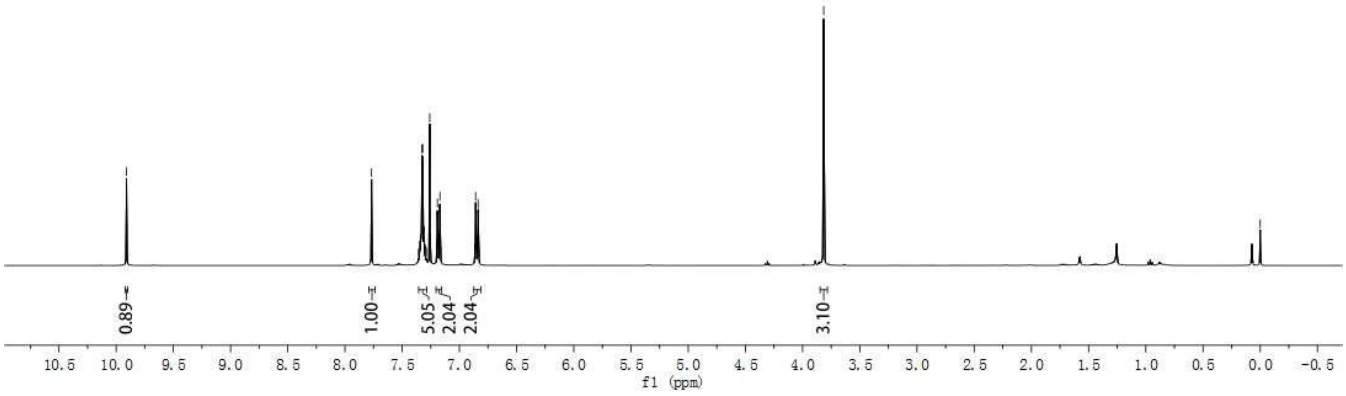

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)


9.898
7.765
7.339
7.333
7.329
7.327
7.318
7.312
7.307
7.295
7.289
7.284
7.245
7.155
7.150
7.139
7.134
7.120
7.100
2.348
0.000
CHO
S
2x
0.94
0.97
5.00
4.15
3.00
f1 (ppm)
182.82
148.30
141.24
139.51
139.17
137.43
133.20
132.14
129.31
129.12
128.75
128.66
77.32
77.00
76.68
21.18
CHO
S
2x
f1 (ppm)

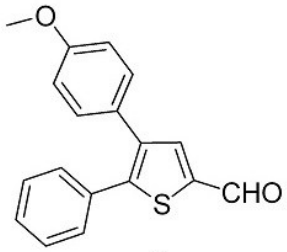

9.910
7.768
7.357
7.347
7.341
7.339
7.327
7.320
7.311
7.309
7.302
7.297
7.289
7.259
7.199
7.192
7.187
7.175
7.170
7.163
6.865
6.858
6.853
6.841
6.836
6.829
3.816
0.000


O
S
CHO

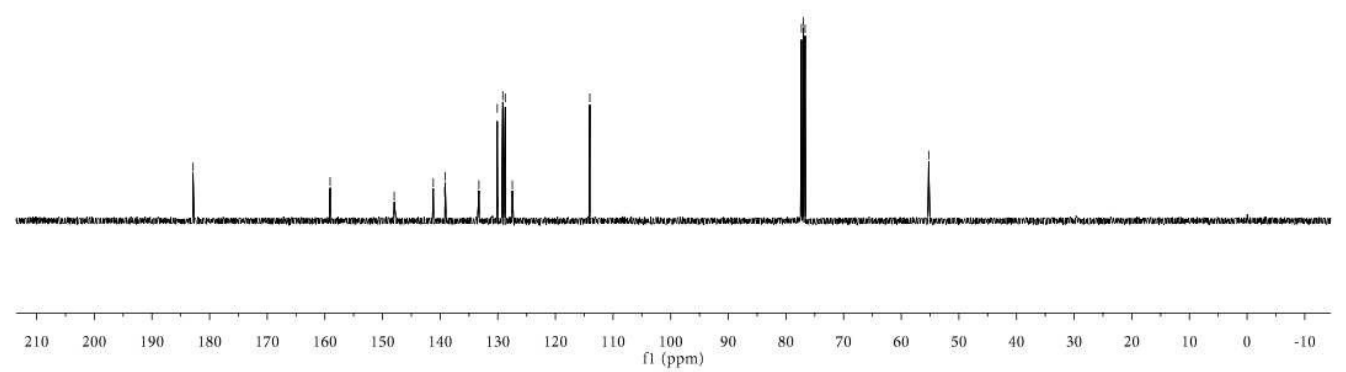
2y

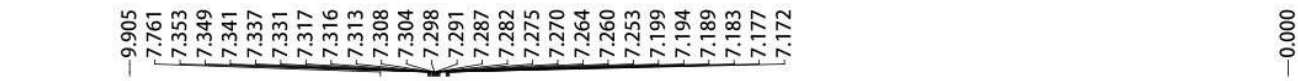

0.89
1.00
5.05
2.04
2.04
3.10
f1 (ppm)

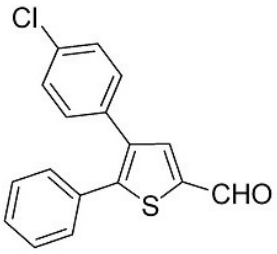

182.86
159.09
141.19
139.22
139.11
133.27
130.09
129.14
128.76
128.71
114.03
77.32
77.00
76.68
55.24


O
S
CHO

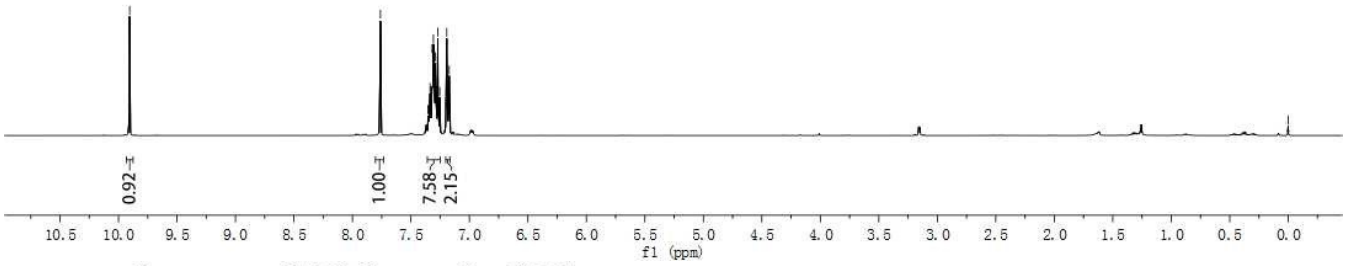
2y


f1 (ppm)

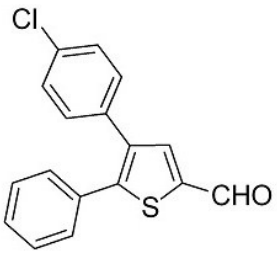

9.905
7.761
7.353
7.349
7.341
7.337
7.331
7.317
7.316
7.313
7.308
7.304
7.298
7.291
7.287
7.282
7.275
7.270
7.264
7.260
7.253
7.199
7.194
7.189
7.183
7.177
7.172
0.000


Cl
S
CHO

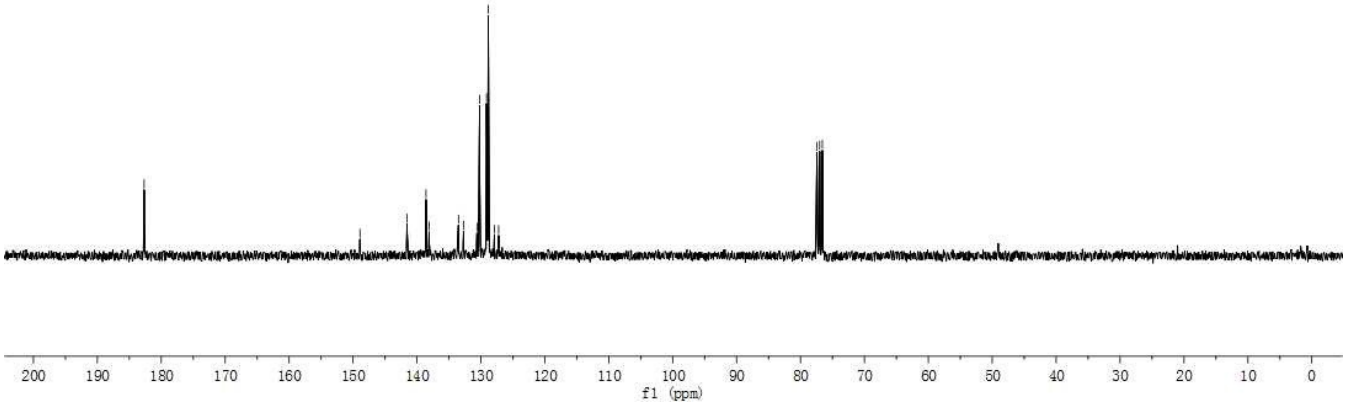
2z

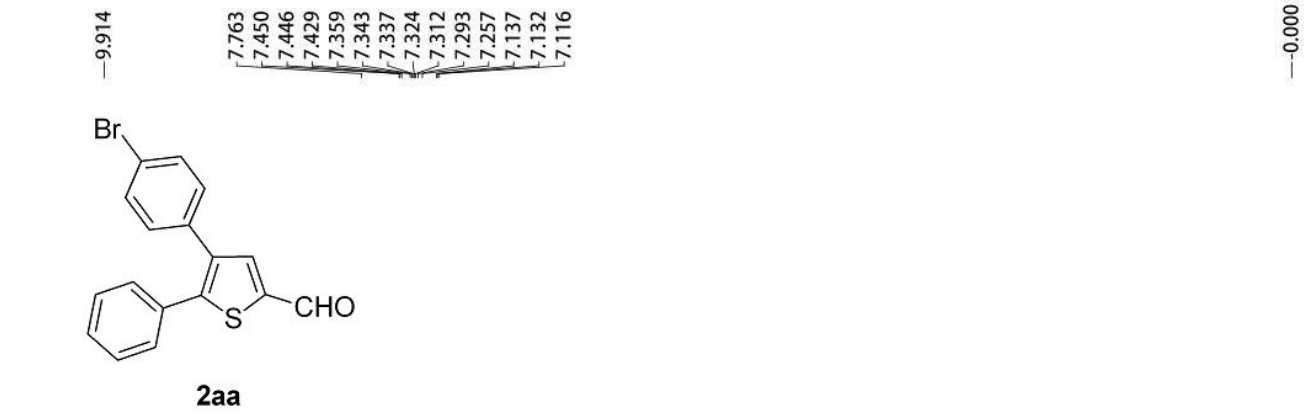

0.92
1.00
7.58
2.15
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


182.69
148.92
141.55
138.59
138.10
133.51
132.71
130.61
130.20
129.11
128.84
127.90
127.22
77.42
77.00
76.58

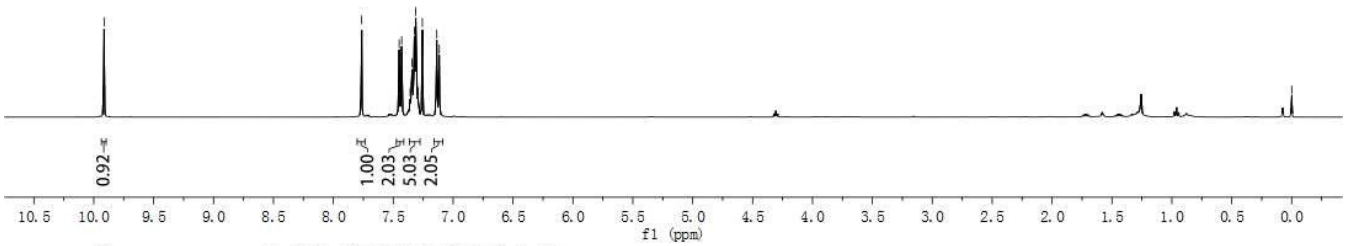

Cl
S
CHO

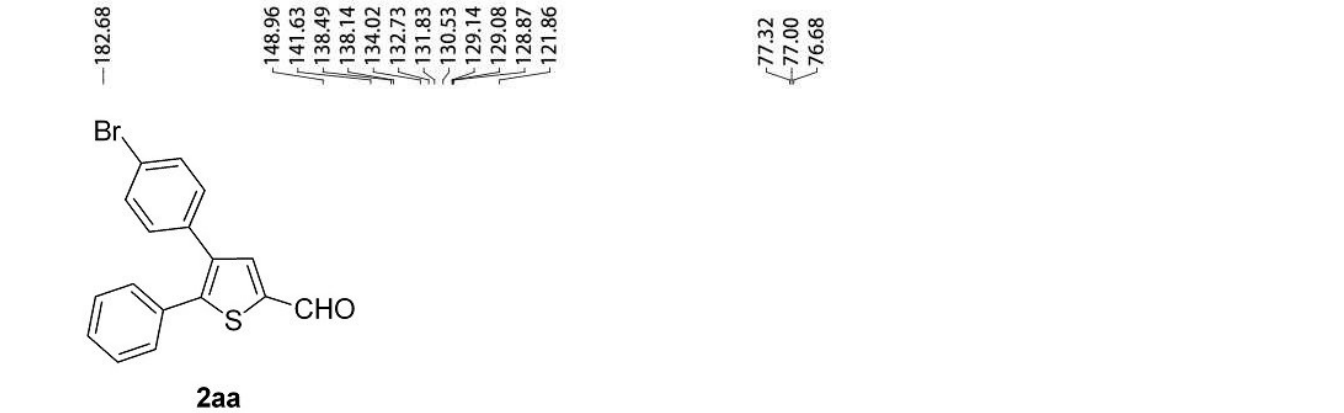
2z


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

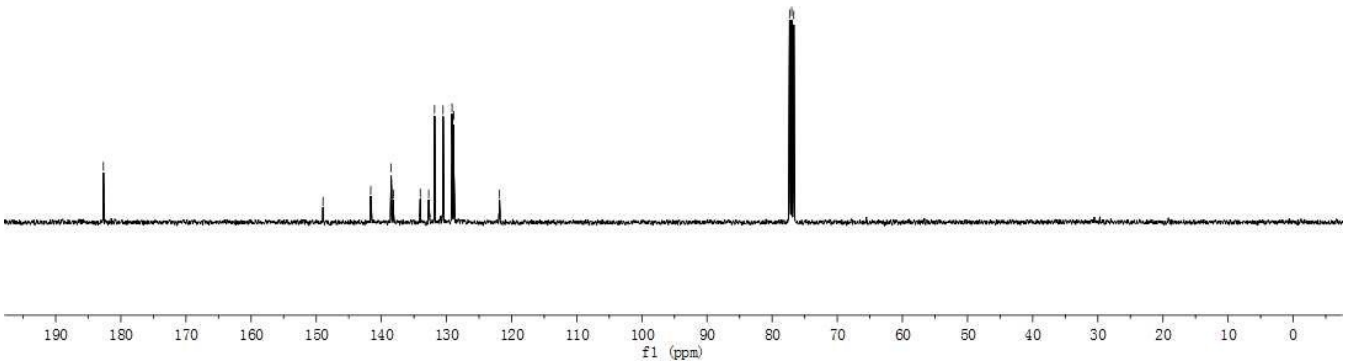

9.914
7.763
7.450
7.446
7.429
7.359
7.343
7.337
7.324
7.312
7.293
7.257
7.137
7.132
7.116
0.000
Br
S
CHO

2aa

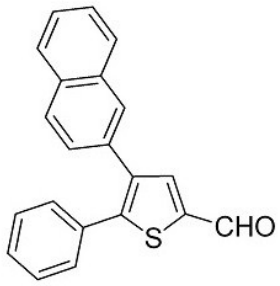

0.92
1.00
2.03
5.03
2.05
f1 (ppm)


182.68
148.96
141.63
138.49
138.14
134.02
132.73
131.83
130.53
129.14
129.08
128.87
121.86
77.32
77.00
76.68
Br
S
CHO

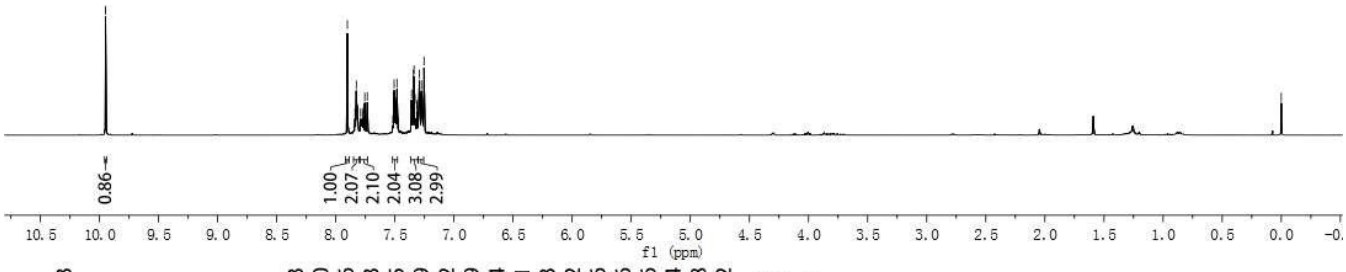
2aa


f1 (ppm)

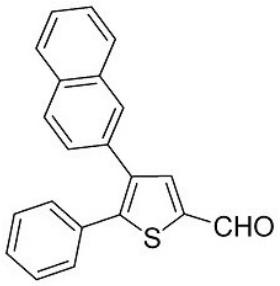

9.947
7.901
7.837
7.826
7.814
7.789
7.774
7.765
7.754
7.733
7.516
7.513
7.506
7.499
7.496
7.491
7.489
7.482
7.471
7.361
7.358
7.354
7.347
7.342
7.337
7.328
7.315
7.311
7.293
7.291
7.286
7.279
7.275
7.270
7.265
7.258
7.253
0.000


S
CHO

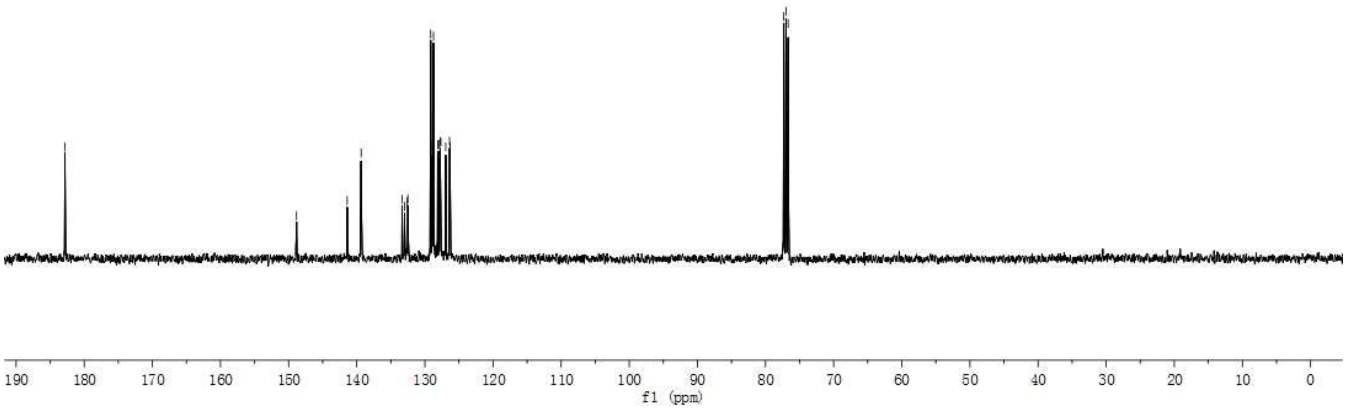
2ab

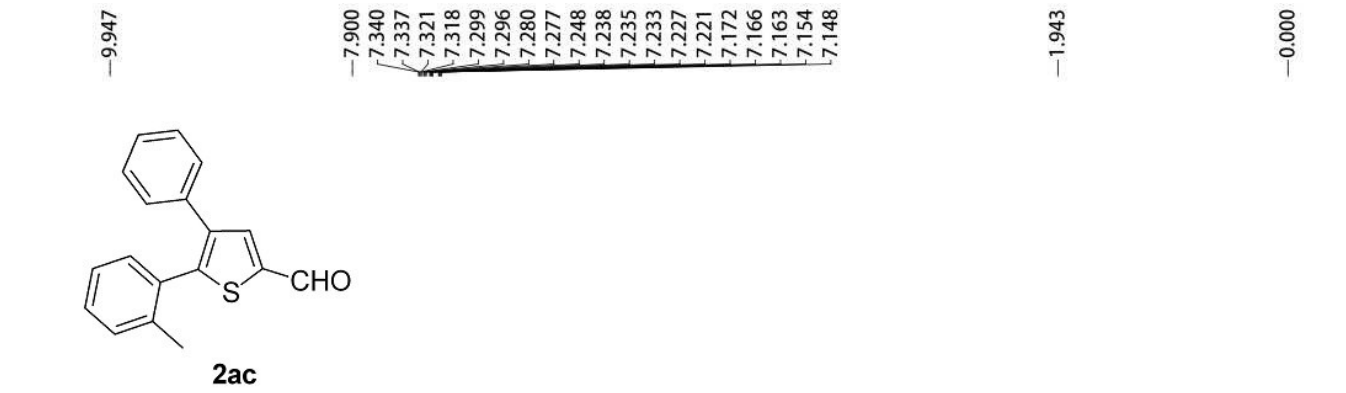

0.86
1.00
2.07
2.10
2.04
3.08
2.99
f1 (ppm)


182.83
148.83
141.40
139.36
139.33
133.35
132.99
132.62
132.49
129.14
128.91
128.73
128.12
127.96
127.76
127.66
126.94
126.38
126.32
77.32
77.00
76.68

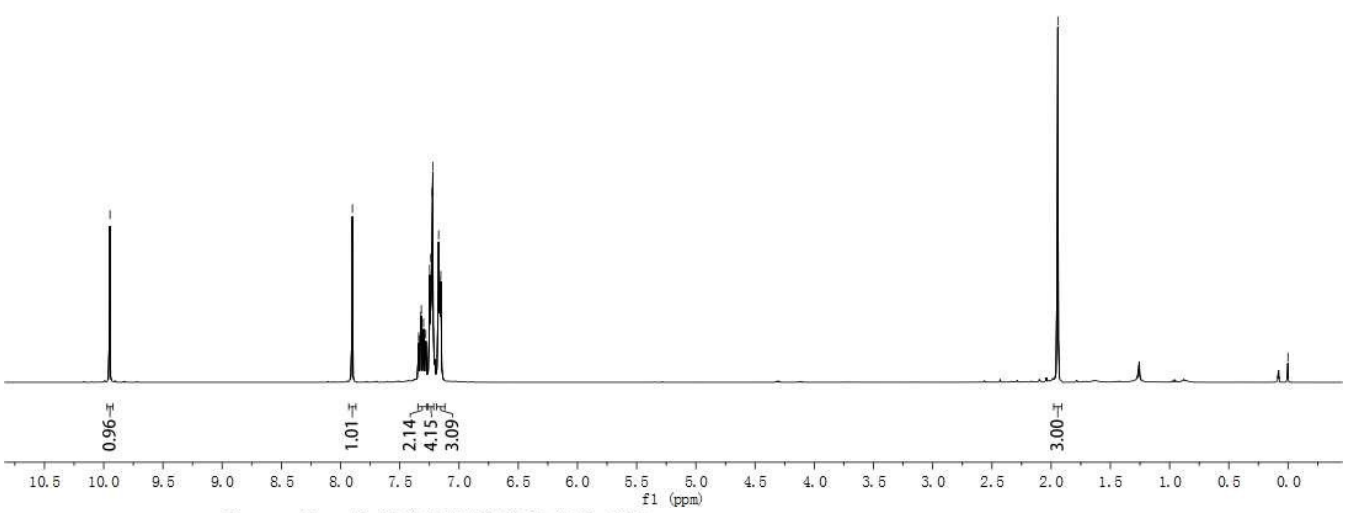

S
CHO

2ab


f1 (ppm)


9.947
7.900
7.340
7.337
7.321
7.318
7.299
7.296
7.280
7.277
7.248
7.238
7.235
7.233
7.227
7.221
7.172
7.166
7.163
7.154
7.148
1.943
0.000
CHO
S

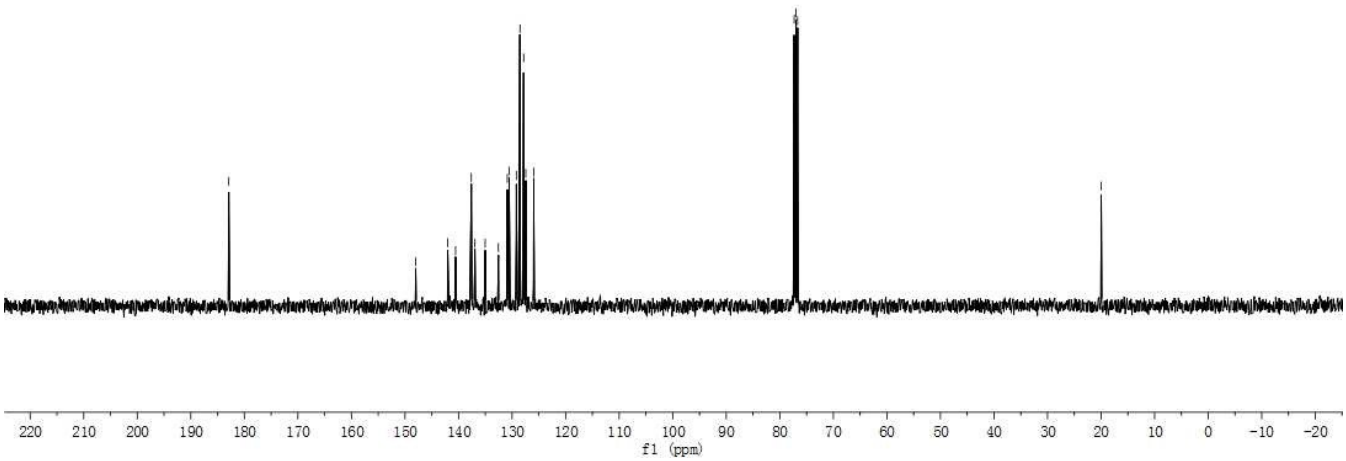
2ac

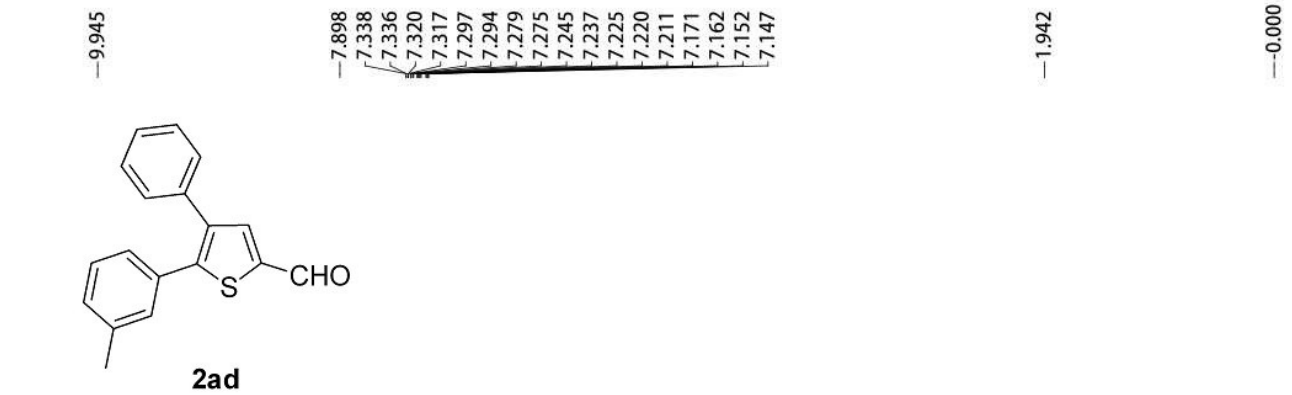

0.96
1.01
2.14
4.15
3.09
3.00
f1 (ppm)


182.90
147.99
142.01
140.55
137.62
136.94
135.03
132.56
130.89
130.54
129.20
128.53
127.83
127.44
125.96
77.32
77.00
76.68
19.96

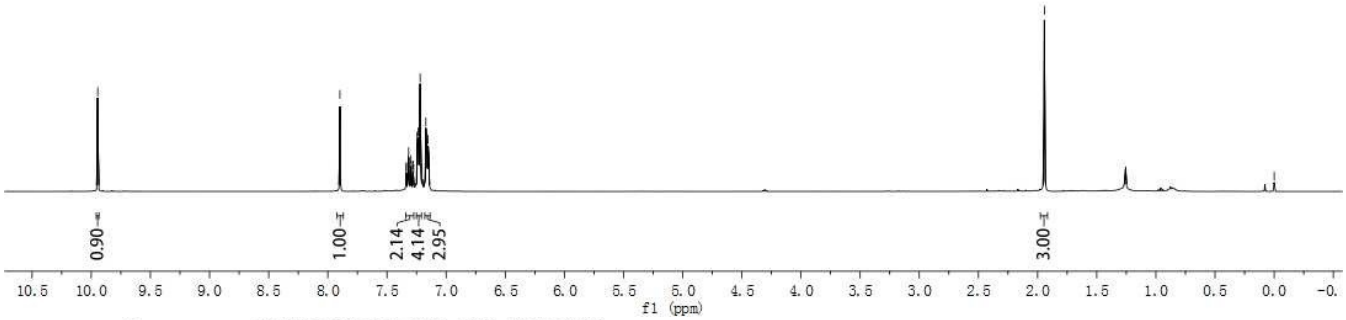

CHO
S

2ac

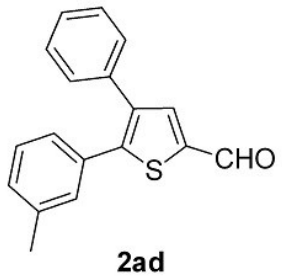

f1 (ppm)


9.945
7.898
7.338
7.336
7.320
7.317
7.297
7.294
7.279
7.275
7.245
7.237
7.225
7.220
7.211
7.171
7.162
7.152
7.147
1.942
0.000
CHO
S

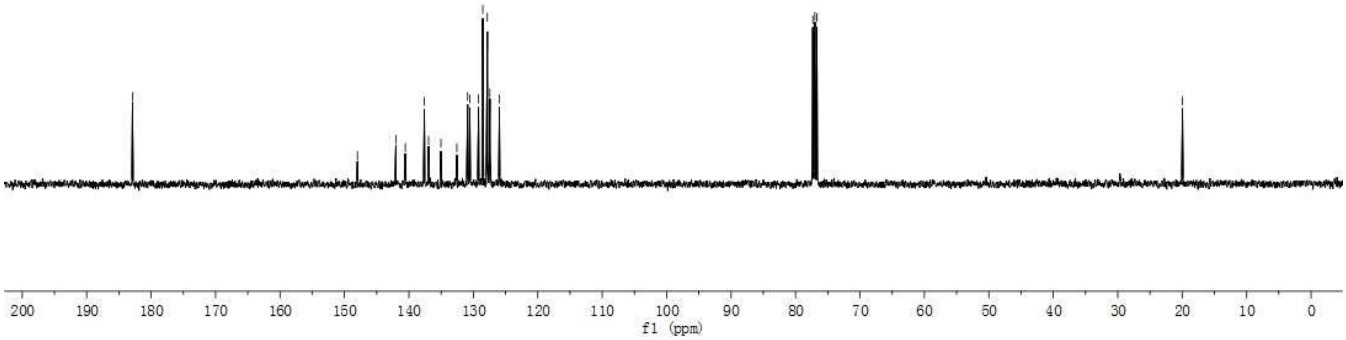
2ad

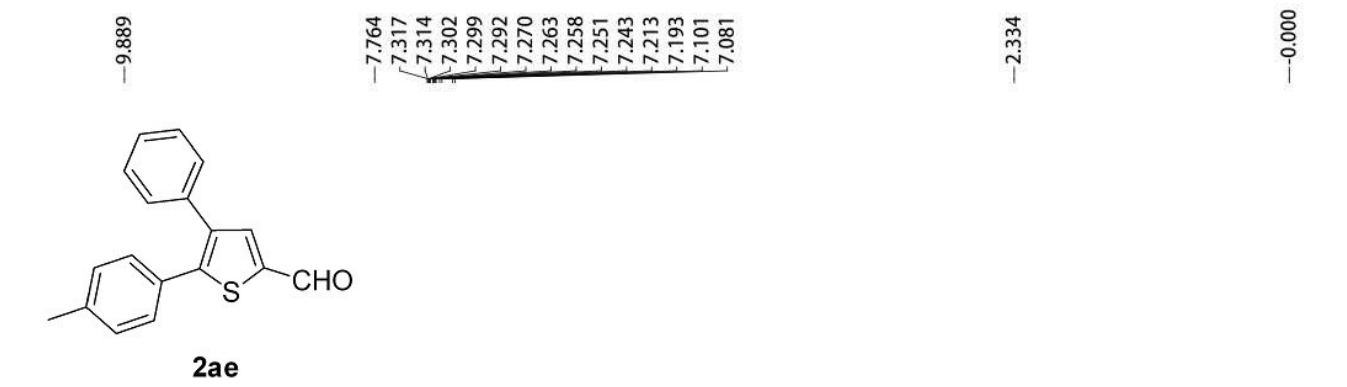

0.90
1.00
2.14
4.14
2.95
3.00
f1 (ppm)


182.89
147.98
142.02
140.56
137.60
136.94
135.03
132.56
130.89
130.54
129.19
128.53
127.83
127.44
125.96
77.32
77.00
76.68
19.96

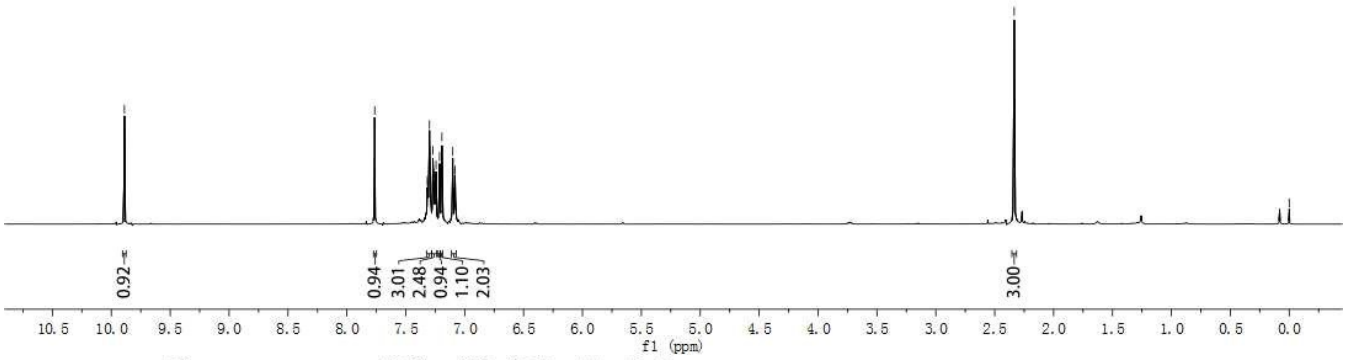

CHO
S

2ad


f1 (ppm)

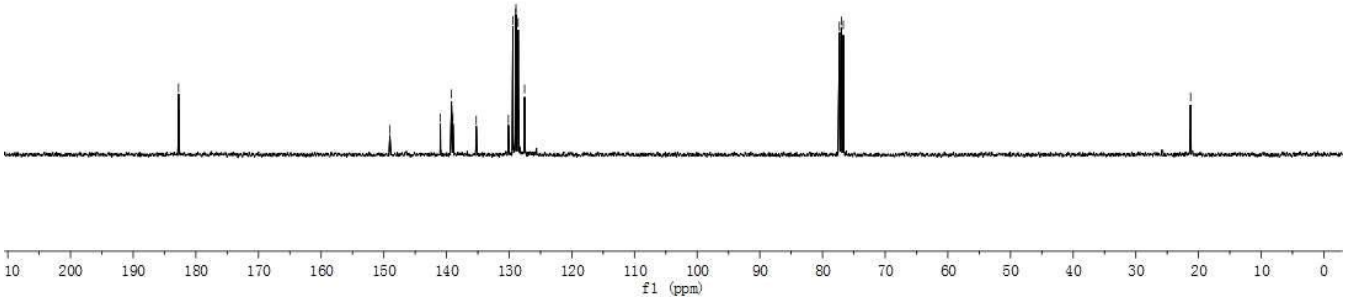

9.889
7.764
7.317
7.314
7.302
7.299
7.292
7.270
7.263
7.258
7.251
7.243
7.213
7.193
7.101
7.081
2.334
0.000
CHO
S

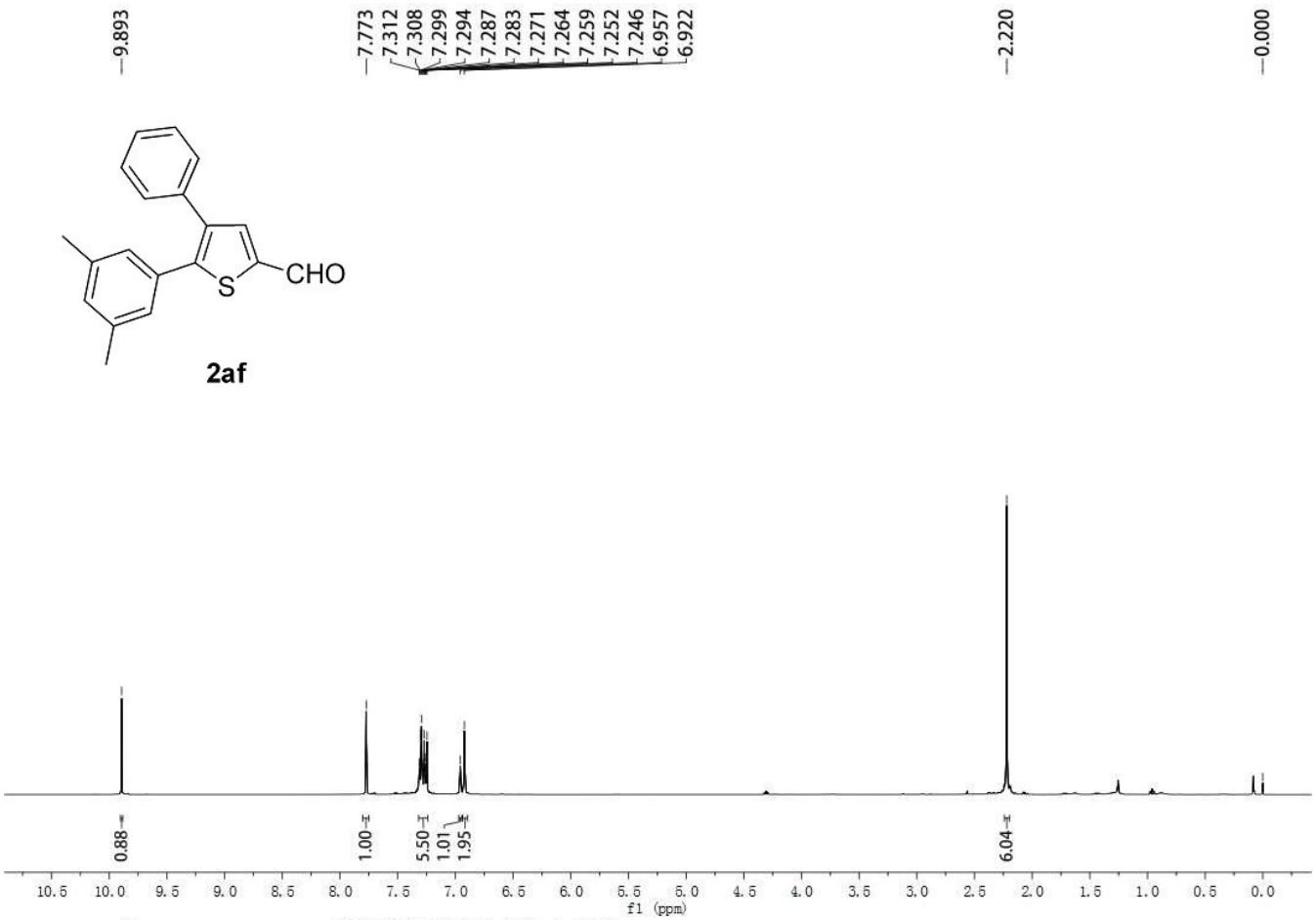
2ae


0.92
0.94
3.01
2.48
0.94
1.10
2.03
3.00
f1 (ppm)

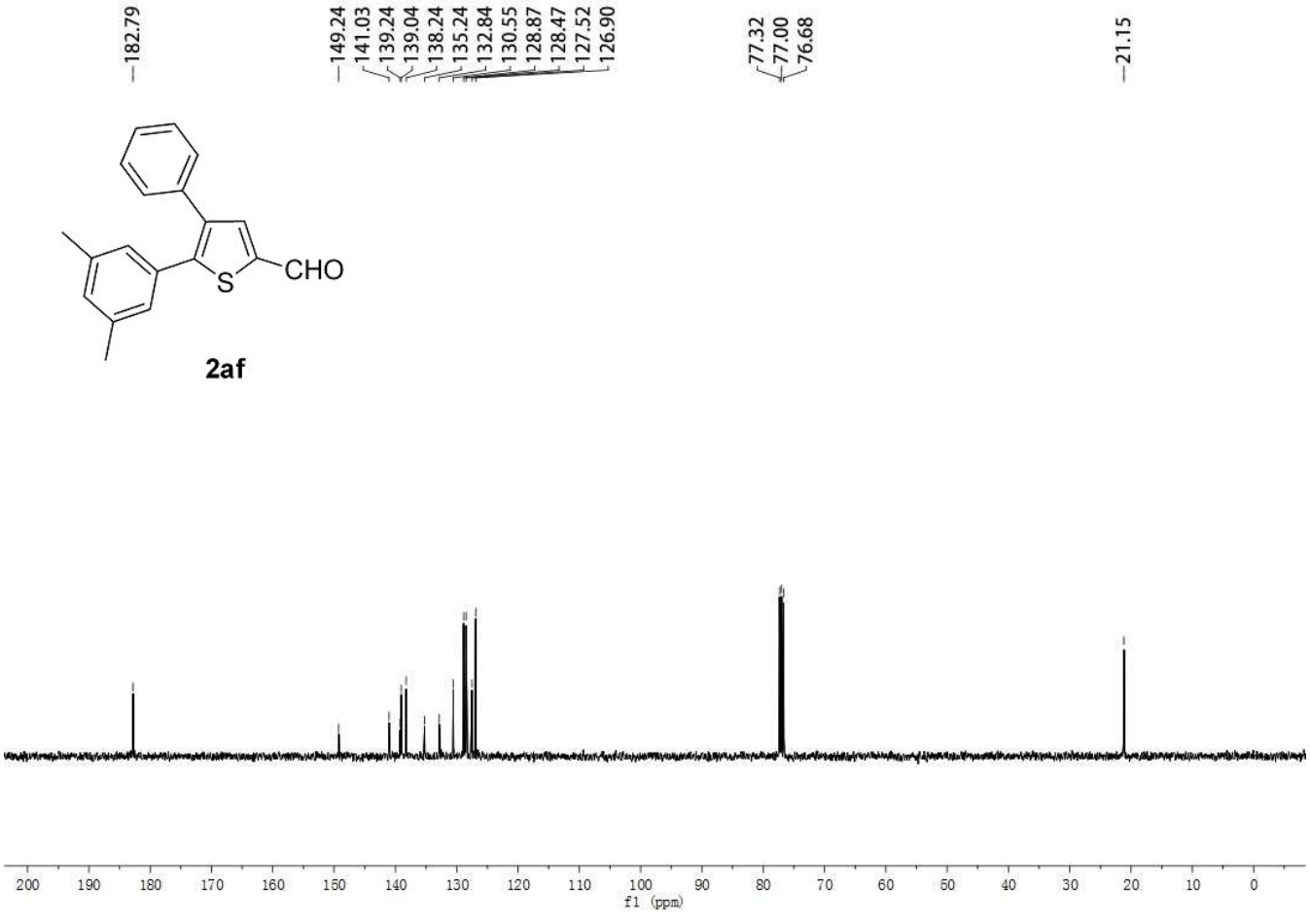

182.76
149.04
140.98
139.21
139.12
138.99
135.26
130.13
129.41
128.96
128.91
128.57
127.54
77.32
77.00
76.68
21.26
CHO
S

2ae

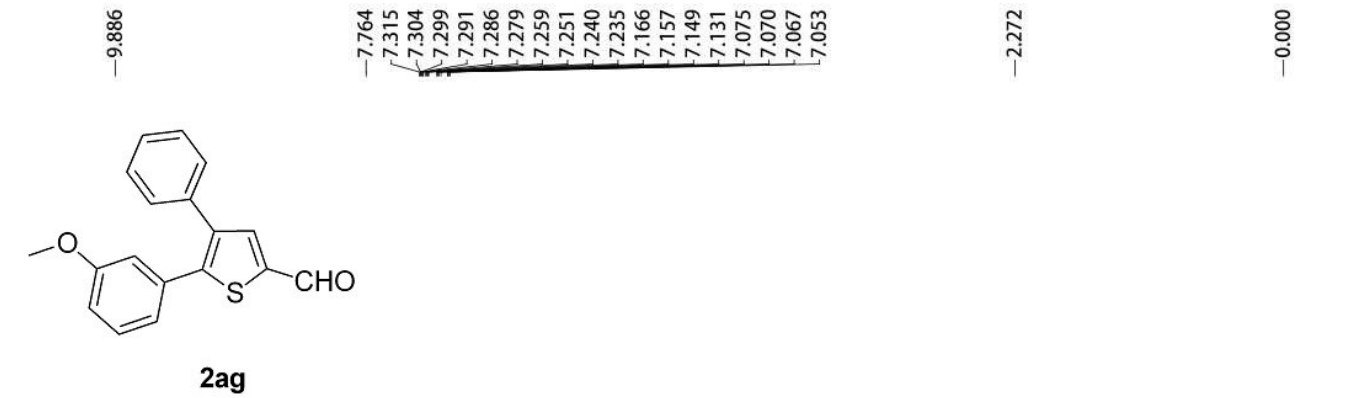

f1 (ppm)


9.893
7.773
7.312
7.308
7.299
7.294
7.287
7.283
7.271
7.264
7.259
7.252
7.246
6.957
6.922
2.220
0.000
CHO
S
0.88
1.00
5.50
1.01
1.95
6.04
f1 (ppm)

2af

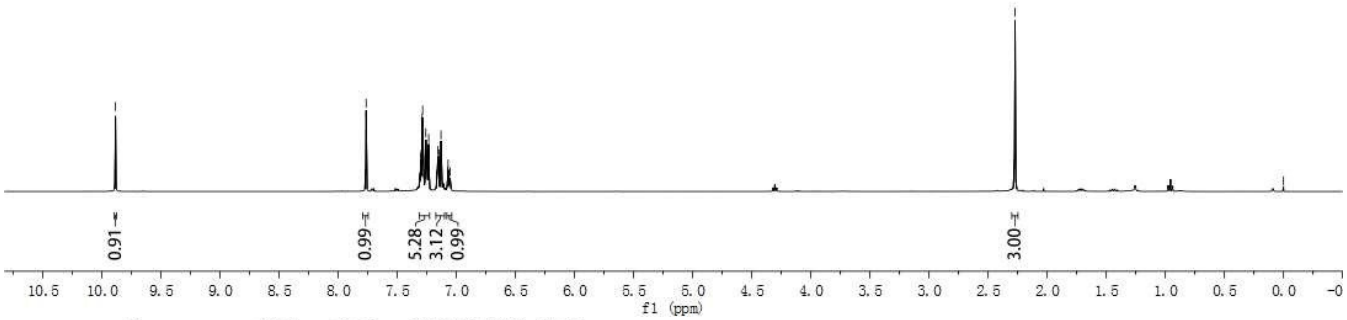

182.79
149.24
141.03
139.24
139.04
138.24
135.24
132.84
130.55
128.87
128.47
127.52
126.90
77.32
77.00
76.68
21.15
CHO
S
f1 (ppm)

2af

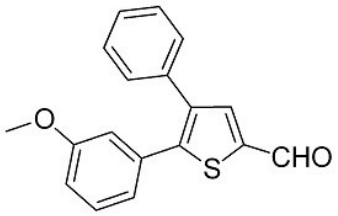

9.886
7.764
7.315
7.304
7.299
7.291
7.286
7.279
7.259
7.251
7.240
7.235
7.166
7.157
7.149
7.131
7.075
7.070
7.067
7.053
2.272
0.000

O
S
CHO

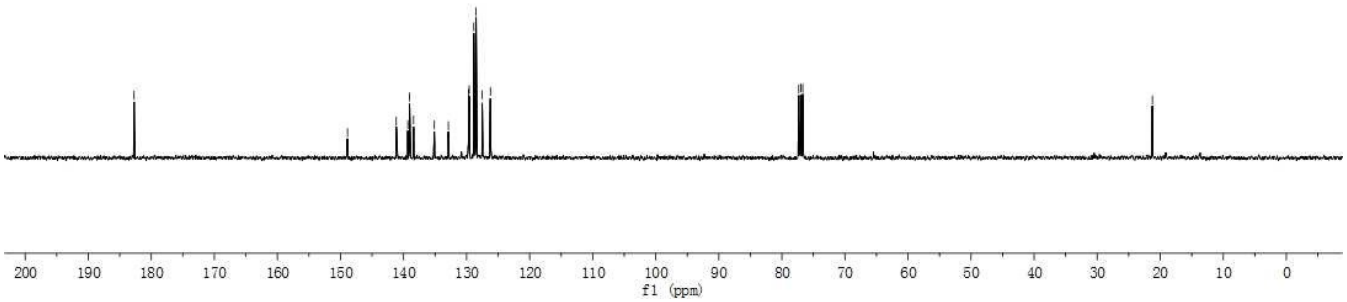
2ag

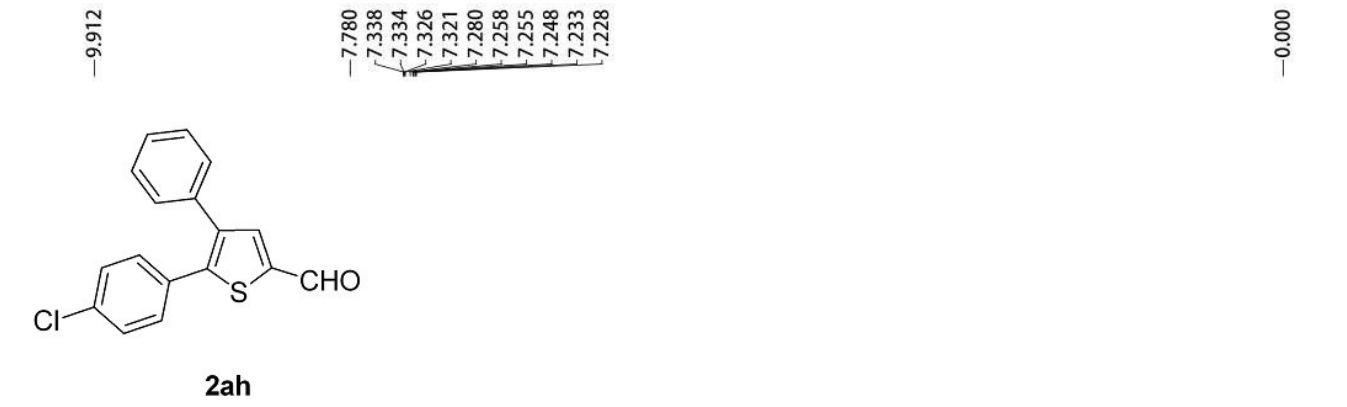

0.91
0.99
5.28
3.12
0.99
3.00
f1 (ppm)


182.73
148.88
141.15
139.31
139.03
138.40
135.11
132.89
129.69
129.58
128.85
128.49
127.52
126.22
77.32
77.00
76.68
21.24

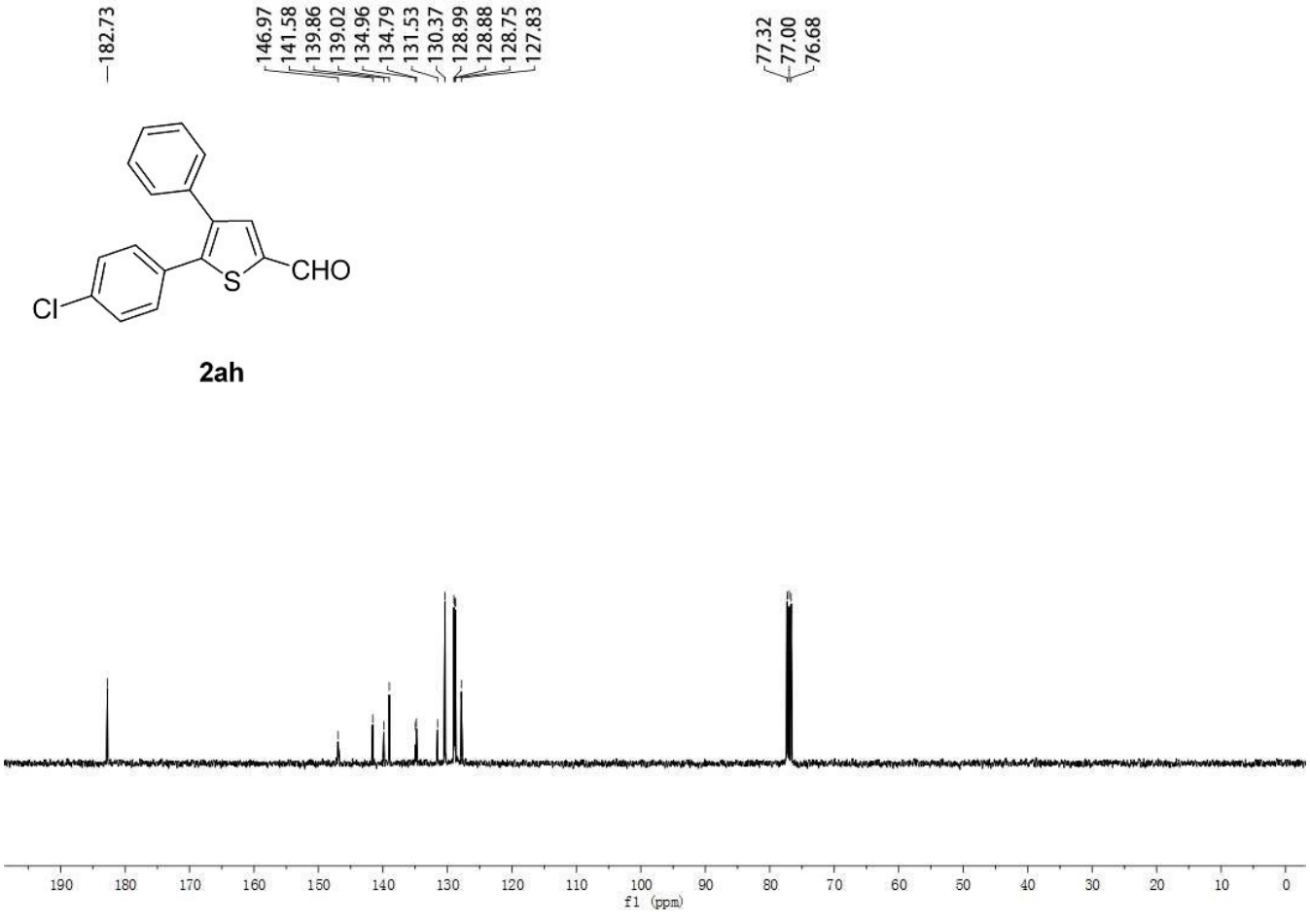

O
S
CHO

2ag

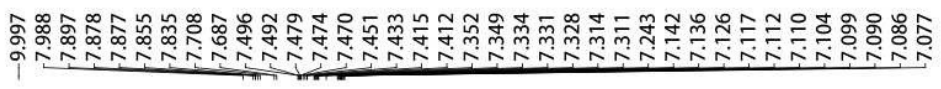

f1 (ppm)

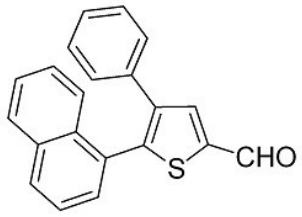

9.912
7.780
7.338
7.334
7.326
7.321
7.280
7.258
7.255
7.248
7.233
7.228
0.000
CHO
S
Cl
1.00
1.06
3.11
6.88
f1 (ppm)

2ah

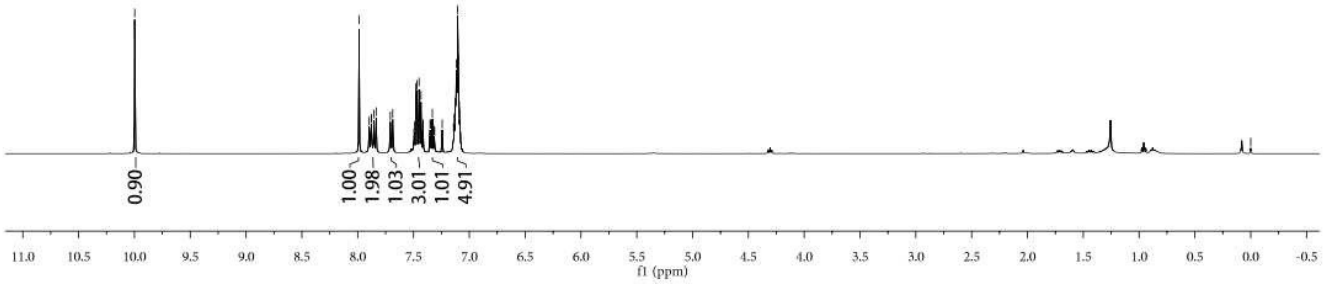

182.73
146.97
141.58
139.86
139.02
134.96
134.79
131.53
130.37
128.99
128.88
128.75
127.83
77.32
77.00
76.68
CHO
S
Cl
f1 (ppm)

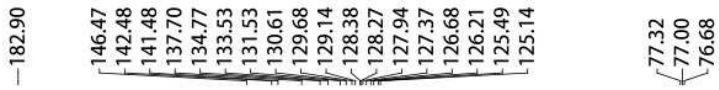
2ah

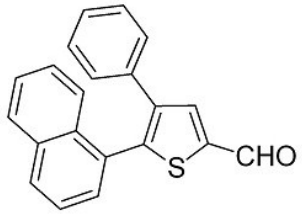

9.997
7.988
7.897
7.878
7.877
7.855
7.835
7.708
7.687
7.496
7.492
7.479
7.474
7.470
7.451
7.433
7.415
7.412
7.352
7.349
7.334
7.331
7.328
7.314
7.311
7.243
7.142
7.136
7.126
7.117
7.112
7.110
7.104
7.099
7.090
7.086
7.077
0.000


S
CHO

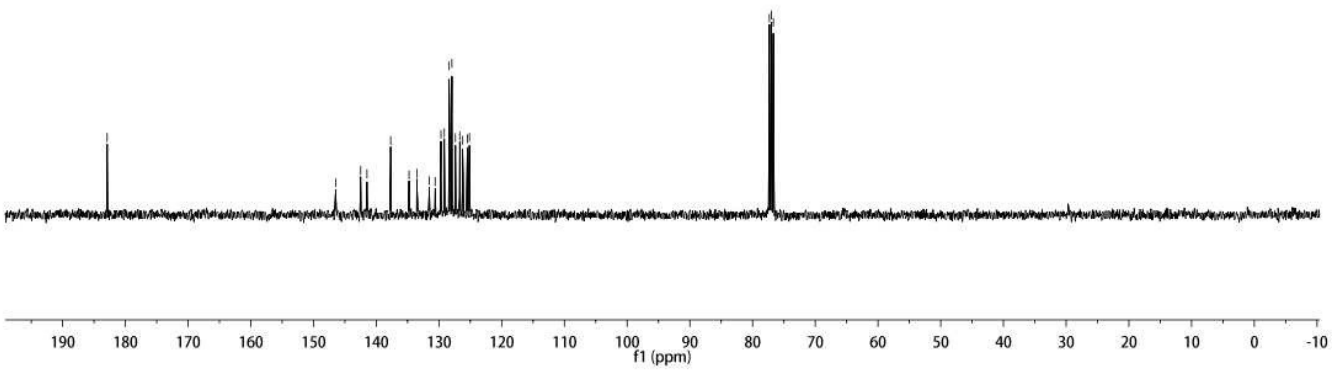
2ai


0.90
1.00
1.98
1.03
3.01
1.01
4.91
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


182.90
146.47
142.48
141.48
137.70
134.77
133.53
131.53
130.61
129.68
129.14
128.38
128.27
127.94
127.37
126.68
126.21
125.49
125.14
77.32
77.00
76.68


S
CHO

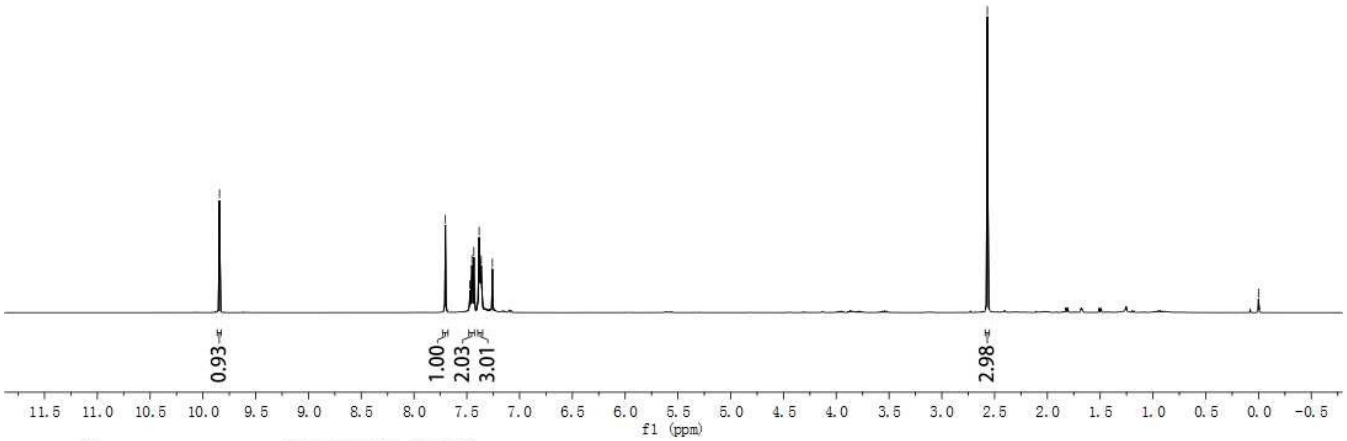
2ai


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

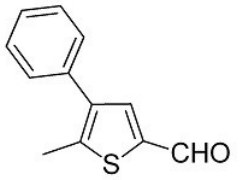

9.843
7.703
7.470
7.451
7.433
7.383
7.376
7.370
7.363
7.353
7.258
2.569
0.000

S
CHO

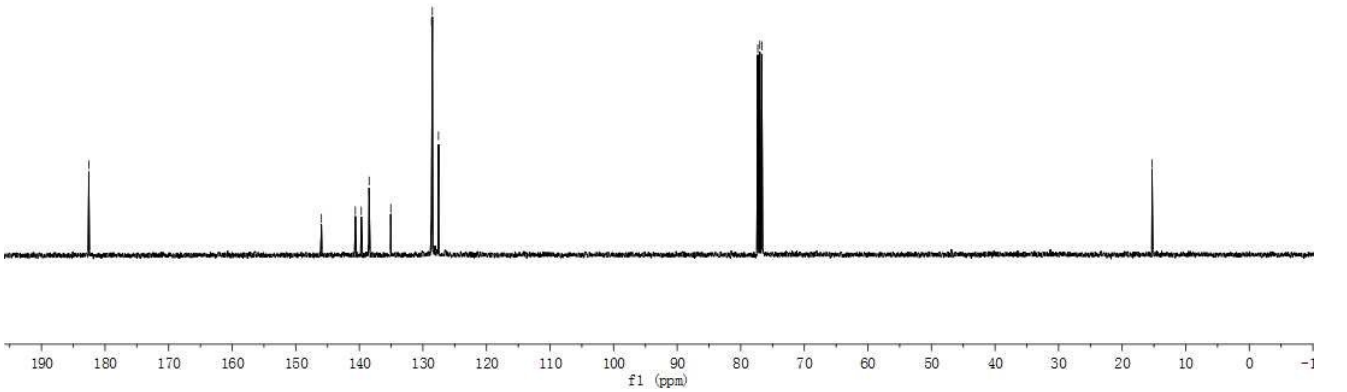
2aj

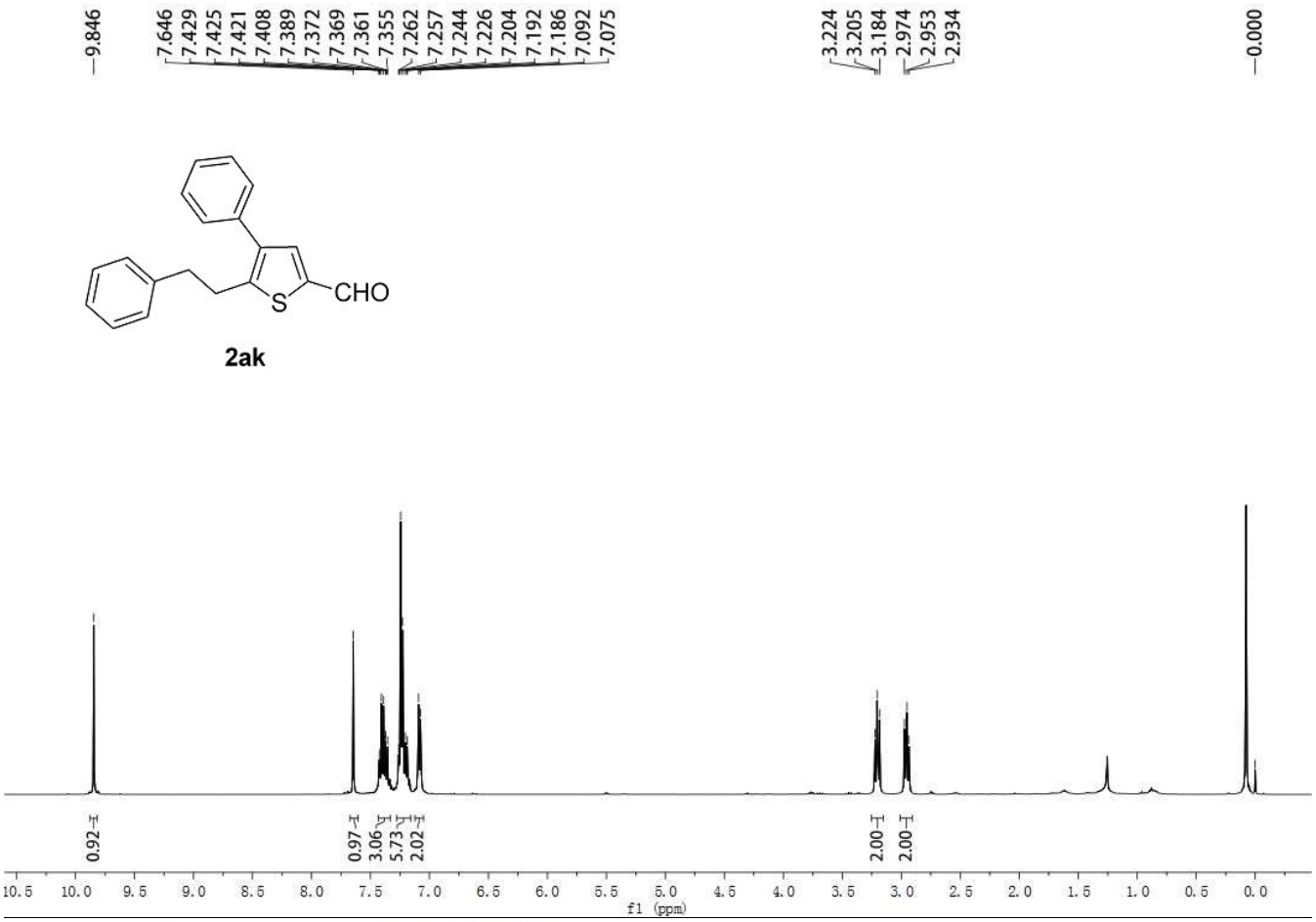

0.93
1.00
2.03
3.01
2.98
f1 (ppm)


182.55
145.96
140.63
139.69
138.46
135.05
128.63
128.52
127.54
77.32
77.00
76.68
15.29

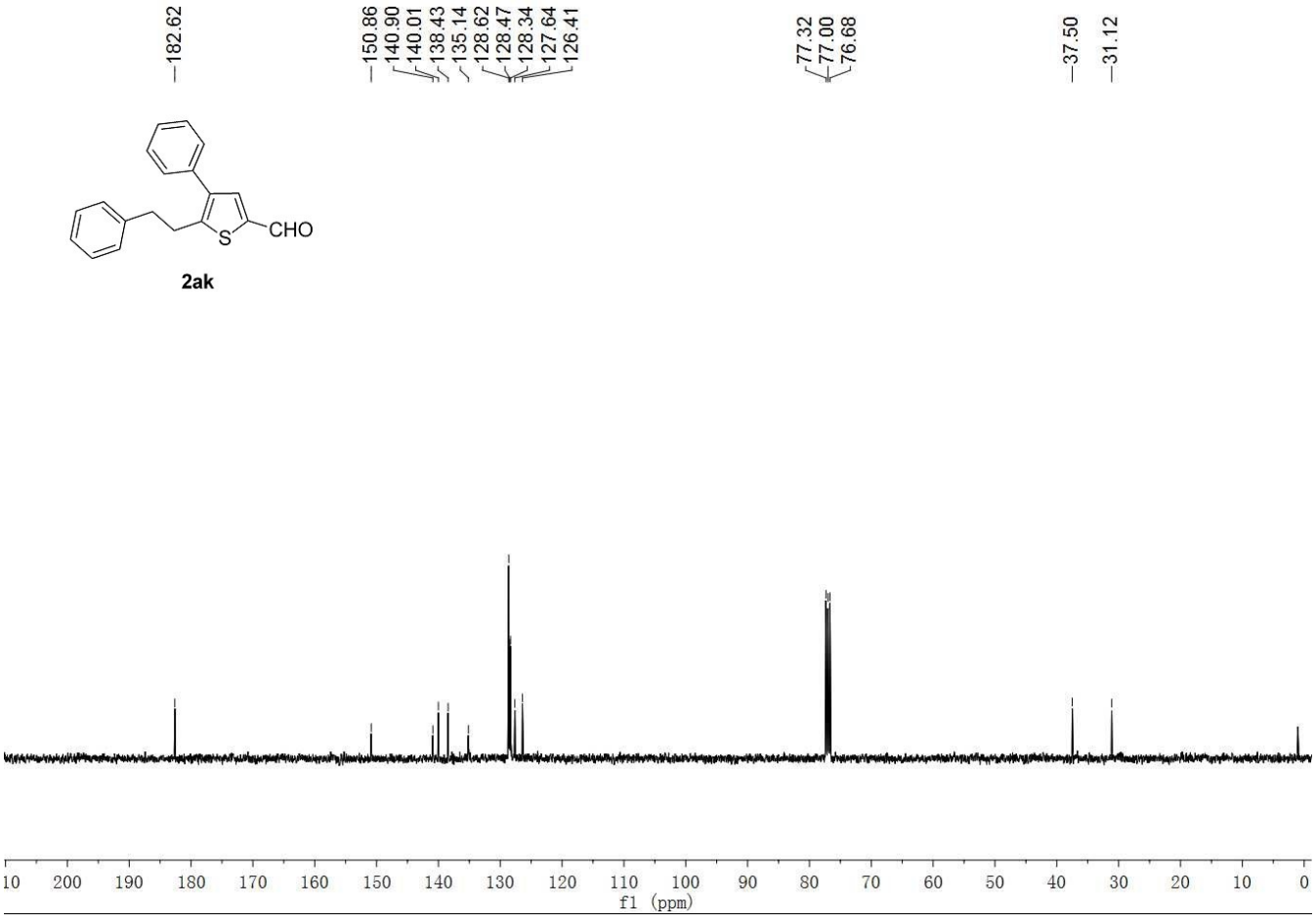

S
CHO

2aj


f1 (ppm)


9.846
7.646
7.429
7.425
7.421
7.408
7.389
7.372
7.369
7.361
7.355
7.262
7.257
7.244
7.226
7.204
7.192
7.186
7.092
7.075
3.224
3.205
3.184
2.974
2.953
2.934
0.000
CHO
S
0.92
0.97
3.06
5.73
2.02
2.00
2.00
f1 (ppm)

2ak


182.62
150.86
140.90
140.01
138.43
135.14
128.62
128.47
128.34
127.64
126.41
77.32
77.00
76.68
37.50
31.12
CHO
S
f1 (ppm)

2ak