Carbocation Catalyzed Ring Closing Aldehyde-Olefin Metathesis

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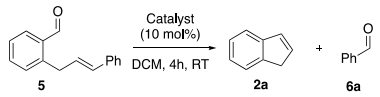
Supporting Information

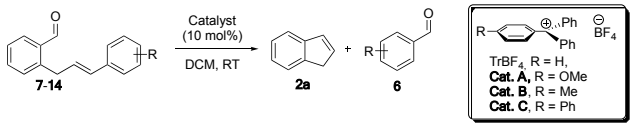
Table S1. Optimization of Lewis Acid Catalysed Aldehyde-olefin Ring Closing Metathesis.[a]



Entry	Catalyst	Solvent	Yield (%)[b]		
			5	**2a**	**6a**
1	$TrBF_4$	DCM	0	71	86
2	$FeCl_3$	DCM	0	0	74
3	$InCl_3$	DCM	0	14	78
4	$BF_3{\cdot}Et_2O$	DCM	0	20	100
5	$HBF_4{\cdot}Et_2O$	DCM	0	0	83
6	$AlCl_3$	DCM	64	3	17
8	$TrBF_4$	DCE	0	18	26
9	$TrBF_4$	CH_3CN	91	0	0
10	$TrBF_4$	1,4-dioxane	98	0	0
11	$TrBF_4$	toluene	89	10	11

[a] Reaction conditions: $TrBF_4$ was added to **5a** in DCM (0.01 M) for 4 hours at room temperature. [b] Yield was determined by ^1H NMR spectroscopy.

Table S2. Evaluation of Olefin Substitution and Carbocation Lewis Acidity on Aldehyde-olefin Metathesis.[a]



Entry	S.M.	R	Catalyst	t (h)	Yield[b]		
					S.M.	**2a**	**6**
1	**7**	p-NO_2	$TrBF_4$	2.5	100	0	0
2	**8**	p-MeO	$TrBF_4$	5 min	0	22	26
3	**9a**	o-Me	$TrBF_4$	40 min	0	75	84
4	**10**	p-Me	$TrBF_4$	15 min	0	60	74
5	**S1**	p-F	$TrBF_4$	1.5	0	71	86
6	**S2**	o-Ph	$TrBF_4$	40 min	0	71	80
7	**S3**	2,4,6-trimethyl	$TrBF_4$	3.5	0	65	79
8	**S4**	1-naphthyl	$TrBF_4$	2	0	58	78
9	**9a**	o-Me	**Cat A**	29	8	17	78
10	**9a**	o-Me	**Cat B**	4	0	76	85
11	**9a**	o-Me	**Cat C**	3	0	80	86
12[c]	**9a**	o-Me	**Cat C**	8	0	80	86

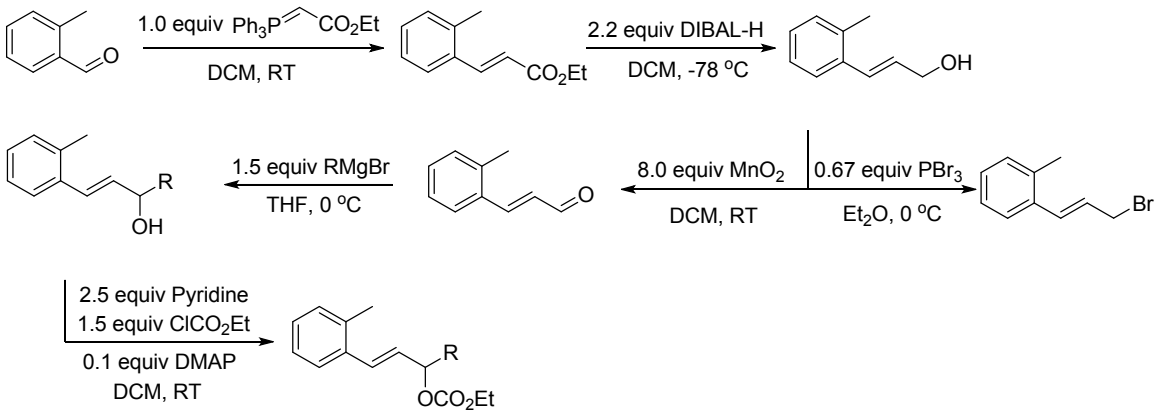
[a] Reaction conditions: $TrBF_4$ (10 mol%) was added to **7-14** in DCM (0.01 M) for the indicated time at room temperature. [b] Yields were determined by ^1H NMR spectroscopy. [c] 5 mol% of **Cat C** was used.

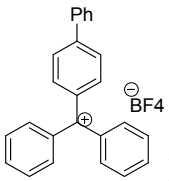
General information

General Experimental Procedure: All reactions are performed in pre-dried solvents under nitrogen atmosphere unless otherwise noticed. All carbocations were dissolved in DCM (0.0625 M) and used as a stock solution. All chemicals and solvents were purchased from Acros, Aldrich, Fluka, Chemtronica or TCI and were used without further purification unless otherwise mentioned. All reactions are performed at room temperature in pre-dried solvents under nitrogen atmosphere unless otherwise noticed. The ^{1}H NMR and ^{13}C NMR spectra were recorded at 500 or 400 MHz and 125 MHz or 100 MHz, respectively. The chemical shifts are reported in ppm relative to $CDCl_3$ (δ = 7.26) and CD_2Cl_2 (δ = 5.30) for ^{1}H NMR and relative to the central $CDCl_3$ resonance (δ = 77.0) and CD_2Cl_2 resonance (δ = 54.0) for ^{13}C NMR. HRMS (FTMS + p NSI) data were recorded by using Thermo Scientific LTQ Orbitrap XL, and samples were infused directly. Flash chromatography was carried out using Merck silica gel 60 (230-400 mesh).

Experiment section

o-Methylcinnamyl bromide and esters were prepared from commercially available chemicals following the similar route as reported.[1,2]





Procedure for the preparation of *p*-PhTrBF$_4$:[3] The triarylmethanol (0.1 mmol) was dissolved in diethyl ether (0.5 mL) and cooled to 0 °C. Under vigorous stirring a solution of tetrafluoroboric acid in diethyl ether (50 wt%, 0.2 mmol) was added dropwise over 5 min. With each drop a deeply colored precipitate formed. After the solvent had been removed, the precipitate was washed with diethyl ether and dried in vacuum affording the corresponding product as a red solid (20 mg, 50%).

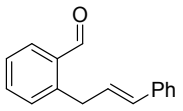
^{1}H-NMR (500 MHz, $CDCl_3$): 8.22 (t, J = 7.5 Hz, 2 H, Ar-H), 8.18 (d, J = 8.5 Hz, 2 H, Ar-H), 7.94-7.92 (m, 2 H, Ar-H), 7.87 (t, J = 7.8 Hz, 4 H, Ar-H), 7.81 (d, J = 8.0 Hz, 2 H, Ar-H), 7.68 (d, J = 7.5 Hz, 4 H, Ar-H), 7.62-7.60 (m, 3 H, Ar-H). ^{13}C-NMR (125 MHz, $CDCl_3$): 207.2, 157.3, 144.6, 142.7, 142.1, 140.4, 139.4, 137.6, 132.5, 130.9, 130.3, 129.6, 129.1.

General procedure A for the preparation of substrates 5, 7-9, 10a, 11-14:[4] To a 5-mL Schlenck flask equipped with a stir bar, was added (2-formylphenyl)boronic acid (30.0 mg, 0.2 mmol), 7 mol% of $Pd(dba)_2$ (8.1 mg, 0.014 mmol), K_2CO_3 (248.8 mg, 1.8 mmol), toluene (1 mL) and *o*-methylcinnamyl bromide (0.24 mmol). The reaction was stirred under reflux until no starting material left. When the mixture was cooled down, Et_2O (5 mL) was added and washed with water, brine and dried over anhydrous Na_2SO_4. Concentration and purification by chromatography provided the desired products.

General procedure B for the preparation of substrates 10b-10j:[5] To a solution of n-butylmagnesium bromide (0.25 mL, 1.0 M solution in THF, 0.25 mmol) in THF (0.5 mL) was added n-butyllithium (0.25 mL, 2.0 M soution in cyclohexane, 0.50 mol) at 0 °C, and the mixture was stirred for 10 min. The resulting mixture was cooled to -40 °C and a solution of corresponding substrate (0.5 mmol) in THF (0.5 mL) was added dropwise. After stirring for 0.5 h at -40 °C, CuCN•2LiCl (0.15 mL, 1.0 M solution in THF, 0.15 mmol) and *o*-methylcinnamyl bromide (0.37 mL, 3.6 mmol) was added sequentially. After stirring for 0.5 h at -40 °C, the reaction was quenched with saturated aqueous NH_4Cl. The mixture was extracted with diethyl ether, and the combined organic layers were dried over anhydrous Na_2SO_4. Concentration and purification by chromatography provided the desired products.

General procedure C for the preparation of substrates 15:[6] To 10-mL Schlenck flask equipped with a stir bar, was added 2 mol% of $Pd(OAc)_2$ (0.90 mg, 0.004 mmol), potassium phosphate (424 mg, 2.0 mmol), (2-formylphenyl)boronic acid (90.0 mg, 0.6 mmol), ester (0.20 mmol) and H_2O (0.2 mL). The mixture was stirred at room temperature until starting material disappeared. Then the mixture was extracted with Et_2O (2.0 mL×4). The organic layer was dried over Na_2SO_4, filtered, and the filtrate was evaporated under reduced pressure. The residue was purified by flash column chromatography to afford the desired products.

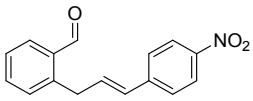
General procedure D for the aldehyde-olefin metathesis: To 50-mL Schlenck flask equipped with a stir bar, was added substrates (0.2 mmol), DCM (20 mL) and 5 mol% of *p*-$PhTrBF_4$ (3.2 μL, $[p\text{-}PhTrBF_4]_{DCM}$ = 0.0625 M). The mixture was stirred at room temperature until starting material disappeared. Then one drop of MeOH was added to quench the reaction. Solvent was removed under vacuum and the residue was purified by flash column chromatography to afford the desired products.



2-Cinnamylbenzaldehyde 5[7] was prepared according to *general procedure A* as a clear oil (35.5 mg, 80%):

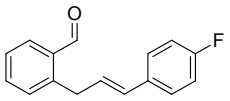
^{1}H-NMR (400 MHz, $CDCl_3$): 10.30 (s, 1 H, CHO), 7.86 (dd, J_1 = 7.6 Hz, J_2 = 1.2 Hz, 1 H, Ar-H), 7.55 (td, J_1 = 7.6 Hz, J_2 = 1.6 Hz, 1 H, Ar-H), 7.42 (t, J = 7.4 Hz, 1 H, Ar-H), 7.37 (d,

J = 7.6 Hz, 1 H, Ar-H), 7.33 (d, *J* = 6.8 Hz, 2 H, Ar-H), 7.29 (d, *J* = 7.2 Hz, 2 H, Ar-H), 7.20 (t, *J* = 7.2 Hz, 1 H, Ar-H), 6.40-6.39 (m, 2 H, CH = CH), 3.99 (d, *J* = 4.4 Hz, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 142.4, 137.2, 134.0, 133.8, 132.1, 131.6, 131.1, 128.6, 128.5, 127.2, 127.0, 126.1, 35.8.



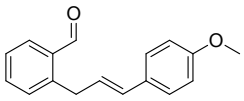
(E)-2-(3-(4-nitrophenyl)allyl)benzaldehyde 7 was prepared according to *general procedure A* as a light yellow solid (28.9 mg, 54%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.23 (s, 1 H, CHO), 8.13 (d, *J* = 9.0 Hz, 2 H, Ar-H), 7.86 (d, *J* = 7.5 Hz, 1 H, Ar-H), 7.58 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H, Ar-H), 7.47 (t, *J* = 7.5 Hz, 1 H, Ar-H), 7.43 (d, *J* = 7.5 Hz, 2 H, Ar-H), 7.36 (d, *J* = 8.0 Hz, 1 H, Ar-H), 6.61 6.25 (dt, J_1 = 15.0 Hz, J_2 = 6.5 Hz, 1 H, CH=), 6.44 (d, *J* = 16.0 Hz, 1 H, =CH), 4.04 (d, *J* = 6.0 Hz, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.6, 146.7, 143.8, 141.1, 134.1, 133.92, 133.85, 133.5, 131.3, 129.5, 127.3, 126.6, 123.9, 36.2. HRMS (FTMS + p NSI): m/z calcd for $[C_{16}H_{13}NO_3+Na]^+$ 290.0788, found 290.0787.



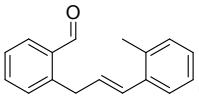
(E)-2-(3-(4-fluorophenyl)allyl)benzaldehyde 8 was prepared according to *general procedure A* as a white solid (30.0 mg, 62%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.28 (s, 1 H, CHO), 7.86 (dd, J_1 = 7.8 Hz, J_2 = 0.8 Hz, 1 H, Ar-H), 7.55 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H, Ar-H), 7.43 (t, *J* = 7.5 Hz, 1 H, Ar-H), 7.36 (d, *J* = 7.5 Hz, 1 H, Ar-H), 7.29-7.27 (m, 2 H, Ar-H), 6.98-6.95 (m, 2 H, Ar-H), 6.37-6.28 (m, 2 H, CH=CH), 3.97 (d, *J* = 5.5 Hz, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 162.1 (d, *J* = 245.0 Hz), 142.3, 134.0, 133.8, 133.4 (d, *J* = 3.8 Hz), 132.4, 131.1, 130.3, 128.4 (d, *J* = 3.8 Hz), 127.6 (d, *J* = 7.5 Hz), 127.0, 115.3 (d, *J* = 21.3 Hz), 35.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{16}H_{13}FO+Na]^+$ 263.0843, found 263.0842.



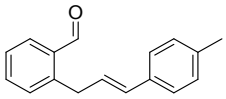
(E)-2-(3-(4-methoxyphenyl)allyl)benzaldehyde 9 was prepared according to *general procedure A* as a light yellow solid (35.3 mg, 46%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.31 (s, 1 H, CHO), 7.86 (d, *J* = 7.0 Hz, 1 H, Ar-H), 7.54 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H, Ar-H), 7.41 (t, *J* = 7.5 Hz, 1 H, Ar-H), 7.36 (d, *J* = 7.5 Hz, 1 H, Ar-H), 7.26 (d, *J* = 8.5 Hz, 2 H, Ar-H), 6.82 (d, *J* = 9.0 Hz, 2 H, Ar-H), 6.34 (d, *J* = 16.0 Hz, 1 H, =CH), 6.25 (dt, J_1 = 15.5 Hz, J_2 = 6.5 Hz, 1 H, CH=), 3.96 (d, *J* = 6.5 Hz, 2 H, CH_2), 3.79 (s, 3 H, MeO). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 159.0, 142.8, 134.0, 133.9, 131.9, 131.1, 131.0, 130.0, 127.3, 126.9, 126.4, 113.9, 55.3, 35.7. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{16}O_2+Na]^+$ 275.1043, found 275.1041.



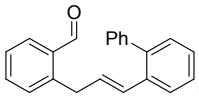
(E)-2-(3-(o-tolyl)allyl)benzaldehyde 10a was prepared according to *general procedure A* as a clear oil (35.2 mg, 75%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.32 (s, 1 H, CHO), 7.88 (d, J = 7.5 Hz, 1 H, Ar-H), 7.56 (t, J = 7.5 Hz, 1 H, Ar-H), 7.43 (t, J = 7.8 Hz, 1 H, Ar-H), 7.41-7.38 (m, 2 H, Ar-H), 7.14-7.13 (m, 3 H, Ar-H), 6.63 (d, J = 16.0 Hz, 1 H, =CH), 6.27 (dt, J_1 = 15.5 Hz, J_2 = 6.8 Hz, 1 H, CH=), 4.02 (d, J = 6.5 Hz, 2 H, CH_2), 2.30 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 142.7, 136.4, 135.2, 134.0, 133.9, 132.1, 131.0, 130.2, 130.0, 129.7, 127.2, 127.0, 126.0, 125.6, 36.1, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{16}O+Na]^+$ 259.1093, found 259.1092.



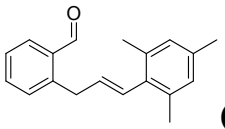
(E)-2-(3-(p-tolyl)allyl)benzaldehyde 11 was prepared according to *general procedure A* as a clear oil (35.0 mg, 74%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.31 (s, 1 H, CHO), 7.87 (d, J = 7.5 Hz, 1 H, Ar-H), 7.55 (t, J = 7.5 Hz, 1 H, Ar-H), 7.42 (t, J = 7.5 Hz, 1 H, Ar-H), 7.37 (d, J = 7.5 Hz, 1 H, Ar-H), 7.23 (d, J = 8.0 Hz, 2 H, Ar-H), 7.10 (d, J = 8.0 Hz, 2 H, Ar-H), 6.40-6.32 (m, 2 H, CH=CH), 3.98 (d, J = 5.0 Hz, 2 H, CH_2), 2.33 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 142.6, 137.0, 134.4, 134.0, 133.8, 131.9, 131.4, 131.0, 129.2, 127.5, 126.9, 126.0, 35.7, 21.1. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{16}O+Na]^+$ 259.1093, found 259.1092.



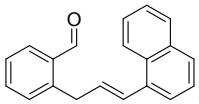
(E)-2-(3-([1,1'-biphenyl]-2-yl)allyl)benzaldehyde 12 was prepared according to *general procedure A* as a clear oil (47.0 mg, 79%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.27 (s, 1 H, CHO), 7.83 (d, J = 7.5 Hz, 1 H, Ar-H), 7.56-7.55 (m, 1 H, Ar-H), 7.51 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H, Ar-H), 7.40-7.27 (m, 10 H, Ar-H), 6.38-6.28 (m, 2 H, CH=CH), 3.90 (d, J = 5.5 Hz, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.3, 142.6, 140.8, 140.5, 135.3, 133.9, 133.8, 131.8, 131.0, 130.8, 130.0, 129.7, 129.4, 128.0, 127.4, 127.2, 126.90, 126.87, 126.0, 35.9. HRMS (FTMS + p NSI): m/z calcd for $[C_{22}H_{18}O+Na]^+$ 321.1250, found 321.1246.



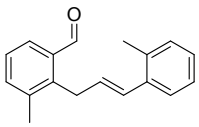
(E)-2-(3-mesitylallyl)benzaldehyde 13 was prepared according to *general procedure A* as a clear oil (36.0 mg, 68%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.36 (s, 1 H, CHO), 7.89 (d, J = 7.5 Hz, 1 H, Ar-H), 7.57 (td, J_1 = 7.5 Hz, J_2 = 1.0 Hz, 1 H, Ar-H), 7.45-7.40 (m, 2 H, Ar-H), 6.85 (s, 2 H, Ar-H), 6.33 (d, J = 16.0 Hz, 1 H, =CH), 6.27 (dt, J_1 = 16.0 Hz, J_2 = 6.5 Hz, 1 H, CH=), 4.03 (d, J = 6.0 Hz, 2 H, CH_2), 2.27 (s, 3 H, CH_3), 2.23 (s, 6 H, 2×CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.3, 142.8, 135.9, 135.7, 134.0, 133.94, 133.87, 133.2, 131.8, 130.9, 129.4, 128.4, 126.9, 36.1, 20.9, 20.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{19}H_{20}O+Na]^+$ 287.1406, found 287.1406.



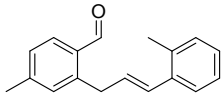
(E)-2-(3-(naphthalen-1-yl)allyl)benzaldehyde 14 was prepared according to *general procedure A* as a light yellow solid (44.0 mg, 81%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.36 (s, 1 H, CHO), 8.03 (d, J = 8.0 Hz, 1 H, Ar-H), 7.90 (d, J = 8.0 Hz, 1 H, Ar-H), 7.40 (d, J = 7.0 Hz, 1 H, Ar-H), 7.75 (d, J = 8.0 Hz, 1 H, Ar-H), 7.58 (t, J = 7.5 Hz, 1 H, Ar-H), 7.55 (d, J = 7.0 Hz, 1 H, Ar-H), 7.52-7.40 (m, 5 H, Ar-H), 7.16 (d, J = 15.5 Hz, 1 H, =CH), 6.42 (dt, J_1 = 15.5 Hz, J_2 = 6.5 Hz, 1 H, CH=), 4.12 (d, J = 6.0 Hz, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 142.4, 135.1, 134.0, 133.9, 133.6, 132.4, 131.8, 131.1, 129.0, 128.4, 127.6, 127.0, 125.9, 125.7, 125.6, 123.8, 123.7, 36.2. HRMS (FTMS + p NSI): m/z calcd for $[C_{20}H_{16}O+Na]^+$ 295.1093, found 295.1093.



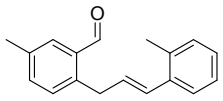
(E)-3-methyl-2-(3-(o-tolyl)allyl)benzaldehyde 10b was prepared according to *general procedure B* as a clear oil (76.0 mg, 61%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.32 (s, 1 H, CHO), 7.44 (d, J = 7.5 Hz, 1 H, Ar-H), 7.36-7.31 (m, 2 H, Ar-H), 7.12-7.10 (m, 3 H, Ar-H), 6.47 (d, J = 16.0 Hz, 1 H, =CH), 6.18 (dt, J_1 = 15.5 Hz, J_2 = 6.5 Hz, 1 H, CH=), 4.02 (d, J = 6.0 Hz, 2 H, CH_2), 2.44 (s, 3 H, CH_3), 2.22 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.8, 140.3, 138.3, 136.5, 136.0, 135.0, 134.3, 130.1, 129.8, 129.2, 129.1, 127.1, 126.7, 126.0, 125.5, 31.4, 19.7, 19.3. HRMS (FTMS + p NSI): m/z calcd for $[C_{18}H_{18}O+Na]^+$ 273.1250, found 273.1254.



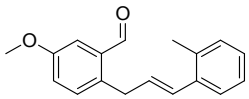
(E)-4-methyl-2-(3-(o-tolyl)allyl)benzaldehyde 10c was prepared according to *general procedure B* as a clear oil (84.0 mg, 67%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.25 (s, 1 H, CHO), 7.76 (d, J = 8.0 Hz, 1 H, Ar-H), 7.40-7.39 (m, 1 H, Ar-H), 7.22 (d, J = 8.0 Hz, 1 H, Ar-H), 7.17 (s, 1 H, Ar-H), 7.14-7.12 (m, 3 H, Ar-H), 6.62 (d, J = 16.0 Hz, 1 H, =CH), 6.25 (dt, J_1 = 15.5 Hz, J_2 = 6.8 Hz, 1 H, CH=), 3.98 (d, J = 7.0 Hz, 2 H, CH_2), 2.41 (s, 3 H, CH_3), 2.30 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.1, 145.0, 142.6, 136.4, 135.1, 132.5, 131.7, 131.6, 130.2, 130.0, 129.5, 127.7, 127.2, 126.0, 125.6, 36.1, 21.8, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{18}H_{18}O+H]^+$ 251.1430, found 251.1429.



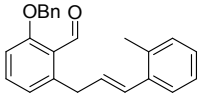
(E)-5-methyl-2-(3-(o-tolyl)allyl)benzaldehyde 10d was prepared according to *general procedure B* as a clear oil (62.6 mg, 50%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.29 (s, 1 H, CHO), 7.67 (s, 1 H, Ar-H), 7.39-7.35 (m, 2 H, Ar-H), 7.26 (d, J = 7.5 Hz, 1 H, Ar-H), 7.13-7.11 (m, 3 H, Ar-H), 6.60 (d, J = 15.5 Hz, 1 H, =CH), 6.24 (dt, J_1 = 16.0 Hz, J_2 = 6.5 Hz, 1 H, CH=), 3.96 (d, J = 6.0 Hz, 2 H, CH_2), 2.41 (s, 3 H, CH_3), 2.29 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 140.0, 136.7, 136.5, 135.1, 134.8, 133.7, 132.3, 131.0, 130.3, 130.1, 129.4, 127.1, 126.0, 125.6, 35.7, 20.8, 19.7. HRMS (FTMS + p NSI): m/z calcd for $[C_{18}H_{18}O+Na]^+$ 273.1250, found 273.1252.



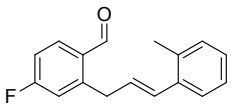
(E)-5-methoxy-2-(3-(o-tolyl)allyl)benzaldehyde 10e was prepared according to *general procedure B* as a yellow oil (60.0 mg, 45%):

Yellow oil: ^{1}H-NMR (500 MHz, $CDCl_3$): 10.32 (s, 1 H, CHO), 7.40 (d, J = 3.0 Hz, 1 H, Ar-H), 7.38-7.37 (m, 1 H, Ar-H), 7.28 (d, J = 8.5 Hz, 1 H, Ar-H), 7.14-7.11 (m, 4 H, Ar-H), 6.57 (d, J = 16.0 Hz, 1 H, =CH), 6.24 (dt, J_1 = 15.5 Hz, J_2 = 6.5 Hz, 1 H, CH=), 3.93 (d, J = 6.5 Hz, 2 H, CH_2), 3.87 (s, 3 H, OCH_3), 2.28 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 191.7, 158.5, 136.3, 135.1, 135.0, 134.6, 132.2, 130.5, 130.1, 129.4, 127.2, 126.0, 125.5, 121.0, 114.1, 55.5, 35.0, 19.7. HRMS (FTMS + p NSI): m/z calcd for $[C_{18}H_{18}O_2+Na]^+$ 289.1199, found 289.1197.



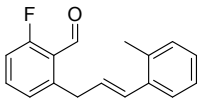
(E)-2-(benzyloxy)-6-(3-(o-tolyl)allyl)benzaldehyde 10f was prepared according to *general procedure B* as a light yellow solid (80.0 mg, 47%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.75 (s, 1 H, CHO), 7.45-7.34 (m, 8 H, Ar-H), 7.13-7.11 (m, 3 H, Ar-H), 6.95 (dd, J_1 = 8.0 Hz, J_2 = 4.0 Hz, 1 H, Ar-H), 6.66 (d, J = 15.5 Hz, 1 H, =CH), 6.18 (dt, J_1 = 15.5 Hz, J_2 = 7.0 Hz, 1 H, CH=), 5.18 (s, 2 H, CH_2), 3.95 (d, J = 7.0 Hz, 2 H, CH_2), 2.32 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.2, 162.4, 144.1, 136.7, 136.2, 135.1, 134.7, 130.09, 130.06, 129.1, 128.7, 128.2, 127.3, 126.9, 125.9, 125.6, 123.32, 123.26, 110.9, 70.7, 37.2, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{24}H_{22}O_2+Na]^+$ 365.1512, found 365.1514.



(E)-4-fluoro-2-(3-(o-tolyl)allyl)benzaldehyde 10g was prepared according to *general procedure B* as a clear oil (76.3, 60%):

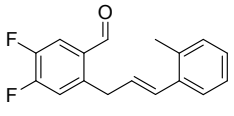
^{1}H-NMR (500 MHz, $CDCl_3$): 10.24 (s, 1 H, CHO), 7.89 (dd, J_1 = 8.8 Hz, J_2 = 6.0 Hz, 1 H, Ar-H), 7.40 (t, J = 4.3 Hz, 1 H, Ar-H), 7.16-7.14 (m, 3 H, Ar-H), 7.11-7.08 (m, 2 H, Ar-H), 6.65 (d, J = 16.0 Hz, 1 H, =CH), 6.22 (dt, J_1 = 15.5 Hz, J_2 = 6.5 Hz, 1 H, CH=), 4.01 (d, J = 6.5 Hz, 2 H, CH_2), 2.31 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 190.7, 165.9 (d, J = 255.0 Hz), 146.2 (d, J = 8.8 Hz), 136.1, 135.2, 135.0 (d, J = 10.0 Hz), 130.44 (d, J = 2.5 Hz), 130.35, 130.2, 128.7, 127.4, 126.1, 125.6, 117.7 (d, J = 22.5 Hz), 114.1(d, J = 21.3 Hz), 35.9 (d, J = 1.3 Hz), 19.7. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{15}FO+Na]^+$ 277.0999, found 277.0998.



(E)-2-fluoro-6-(3-(o-tolyl)allyl)benzaldehyde 10h was prepared according to *general procedure B* as a white solid (100.0 mg, 79%):

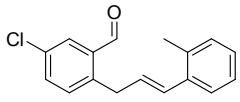
^{1}H-NMR (500 MHz, $CDCl_3$): 10.57 (s, 1 H, CHO), 7.53-7.48 (m, 1 H, Ar-H), 7.40 (t, J = 4.0 Hz, 1 H, Ar-H), 7.17 (d, J = 7.5 Hz, 1 H, Ar-H), 7.14-7.12 (m, 3 H, Ar-H), 7.08-7.05 (m, 1 H, Ar-H), 6.67 (d, J = 15.5 Hz, 1 H, =CH), 6.18 (dt, J_1 = 15.5 Hz, J_2 = 7.0 Hz, 1 H, CH=), 3.98

(d, J = 7.0 Hz, 2 H, CH_2), 2.32 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 189.0 (d, J = 11.3 Hz), 166.3 (d, J = 256.3 Hz), 144.6, 136.5, 135.3 (d, J =11.3 Hz), 135.1, 130.2, 129.7, 129.2, 127.2, 126.6 (d, J = 3.8 Hz), 126.0, 125.6, 122.1 (d, J = 6.3 Hz), 114.3 (d, J = 21.3 Hz), 36.8 (d, J = 1.3 Hz), 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{15}FO+Na]^+$ 277.0999, found 277.0999.



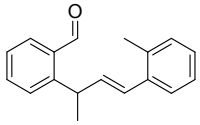
(E)-4,5-difluoro-2-(3-(o-tolyl)allyl)benzaldehyde 10i was prepared according to *general procedure B* as a light yellow solid (78.0 mg, 57%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.24 (s, 1 H, CHO), 7.71 (dd, J_1 = 10.3 Hz, J_2 = 8.0 Hz, 1 H, Ar-H), 7.39 (t, J = 4.5 Hz, 1 H, Ar-H), 7.20 (dd, J_1 = 10.8 Hz, J_2 = 7.3 Hz, 1 H, Ar-H), 7.17-7.15 (m, 3 H, Ar-H), 6.63 (d, J = 16.0 Hz, 1 H, =CH), 6.18 (dt, J_1 = 16.0 Hz, J_2 = 6.5 Hz, 1 H, CH=), 3.96 (d, J = 6.5 Hz, 2 H, CH_2), 2.30 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 189.2, 153.7 (dd, J_1 = 256.9 Hz, J_2 = 13.1 Hz), 149.2 (dd, J_1 = 123.8 Hz, J_2 = 12.5 Hz), 140.6 (dd, J_1 = 6.3 Hz, J_2 = 3.8 Hz), 135.9, 135.2, 128.6, 127.5, 126.1, 125.5, 119.7 (d, J = 17.5 Hz), 119.6 (dd, J_1 = 17.5 Hz, J_2 = 2.5 Hz), 115.5 (d, J = 18.8 Hz), 35.0, 19.7. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{14}FO_2+Na]^+$ 295.0905, found 295.0904.



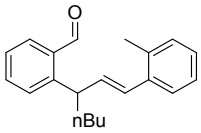
(E)-5-chloro-2-(3-(o-tolyl)allyl)benzaldehyde 10j was prepared according to *general procedure B* as a yellow oil (60.0 mg, 45%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.27 (s, 1 H, CHO), 7.84 (d, J = 2.5 Hz, 1 H, Ar-H), 7.52 (dd, J_1 = 8.0 Hz, J_2 = 2.0 Hz, 1 H, Ar-H), 7.37 (t, J = 4.3 Hz, 1 H, Ar-H), 7.33 (d, J = 8.0 Hz, 1 H, Ar-H), 7.15-7.11 (m, 3 H, Ar-H), 6.61 (d, J = 16.0 Hz, 1 H, =CH), 6.21 (dt, J_1 = 16.0 Hz, J_2 = 6.5 Hz, 1 H, CH=), 3.97 (d, J = 6.5 Hz, 2 H, CH_2), 2.29 (s, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 190.8, 140.9, 136.1, 135.2, 135.0, 133.9, 133.2, 132.5, 131.1, 130.2, 130.1, 129.3, 127.4, 126.1, 125.5, 35.4, 19.7. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{15}ClO+Na]^+$ 293.0704, found 293.0704.



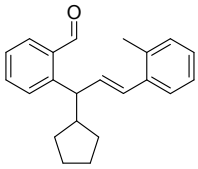
(E)-2-(4-(o-tolyl)but-3-en-2-yl)benzaldehyde 15a was prepared according to *general procedure C* as a clear oil (32.1 mg, 64%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.39 (s, 1 H, CHO), 7.85 (d, J = 7.5 Hz, 1 H, Ar-H), 7.57 (t, J = 7.5 Hz, 1 H, Ar-H), 7.48 (d, J = 7.5 Hz, 1 H, Ar-H), 7.42-7.39 (m, 2 H, Ar-H), 7.17-7.13 (m, 3 H, Ar-H), 6.64 (d, J = 15.5 Hz, 1 H, =CH), 6.18 (dd, J_1 = 16.0 Hz, J_2 = 6.3 Hz, 1 H, CH=), 4.80-4.75 (m, 1 H, CH), 2.31 (s, 3 H, CH_3), 1.53 (d, J = 7.0 Hz, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 148.3, 136.5, 135.8, 135.2, 134.0, 133.3, 132.1, 130.2, 127.9, 127.4, 127.2, 126.6, 126.0, 125.5, 36.6, 21.4, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{18}H_{18}O+Na]^+$ 273.1250, found 273.1252.



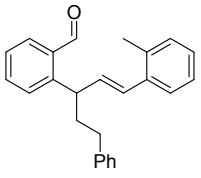
(E)-2-(1-(o-tolyl)hept-1-en-3-yl)benzaldehyde 15b was prepared according to *general procedure C* as a clear oil (40.0 mg, 68%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.40 (s, 1 H, CHO), 7.85 (d, J = 7.5 Hz, 1 H, Ar-H), 7.57 (t, J = 7.8 Hz, 1 H, Ar-H), 7.48 (d, J = 8.0 Hz, 1 H, Ar-H), 7.40-7.37 (m, 2 H, Ar-H), 7.15-7.11 (m, 3 H, Ar-H), 6.62 (d, J = 16.0 Hz, 1 H, =CH), 6.23 (dt, J_1 = 15.8 Hz, J_2 = 7.8 Hz, 1 H, CH=), 4.60 (q, J = 7.5 Hz, 1 H, CH_2), 2.29 (s, 3 H, CH_3), 1.91-1.79 (m, 2 H, CH_2), 1.43-1.26 (m, 4 H, 2×CH_2), 0.88 (t, J = 7.0 Hz, 3 H, CH_3). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 147.7, 136.5, 135.2, 134.9, 134.0, 133.6, 131.9, 130.2, 128.4, 128.1, 127.2, 126.5, 126.0, 125.5, 42.5, 36.1, 29.9, 22.7, 19.8, 14.0. HRMS (FTMS + p NSI): m/z calcd for $[C_{21}H_{24}O+Na]^+$ 315.1719, found 315.1720.



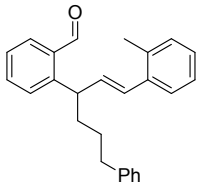
(E)-2-(1-cyclopentyl-3-(o-tolyl)allyl)benzaldehyde 15c was prepared according to *general procedure C* as a clear oil (51.2 mg, 84%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.42 (s, 1 H, CHO), 7.83 (d, J = 8.0 Hz, 1 H, Ar-H), 7.57 (t, J = 7.5 Hz, 1 H, Ar-H), 7.53 (d, J = 7.5 Hz, 1 H, Ar-H), 7.38-7.36 (m, 2 H, Ar-H), 7.13-7.10 (m, 3 H, Ar-H), 6.61 (d, J = 15.5 Hz, 1 H, =CH), 6.24 (dd, J_1 = 15.5 Hz, J_2 = 8.0 Hz, 1 H, CH=), 4.42 (t, J = 9.0 Hz, 1 H, CH), 2.42-2.34 (m, 1 H, one proton of CH_2), 2.28 (s, 3 H, CH_3), 1.96-1.90 (m, 1 H, one proton of CH_2), 1.73-1.68 (m, 1 H, one proton of CH_2), 1.65-1.59 (m, 2 H, CH_2), 1.53-1.41 (m, 3 H, CH_2 and one proton of CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 147.5, 136.6, 135.2, 134.7, 133.9, 133.7, 131.7, 130.2, 128.60, 128.57, 127.1, 126.3, 125.9, 125.5, 48.4, 45.4, 31.6, 31.4, 25.2, 25.1, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{22}H_{24}O+Na]^+$ 327.1719, found 327.1715.



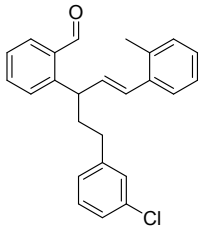
(E)-2-(5-phenyl-1-(o-tolyl)pent-1-en-3-yl)benzaldehyde 15d was prepared according to *general procedure C* as a clear oil (51.1 mg, 75%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.29 (s, 1 H, CHO), 7.86 (d, J = 8.0 Hz, 1 H, Ar-H), 7.59 (t, J = 7.5 Hz, 1 H, Ar-H), 7.52 (d, J = 7.5 Hz, 1 H, Ar-H), 7.42-7.39 (m, 2 H, Ar-H), 7.29 (t, J = 7.5 Hz, 2 H, Ar-H), 7.21-7.14 (m, 6 H, Ar-H), 6.67 (d, J = 16.0 Hz, 1 H, =CH), 6.26 (dd, J_1 = 15.8 Hz, J_2 = 7.8 Hz, 1 H, CH=), 4.66 (q, J = 7.5 Hz, 1 H, CH), 2.77-2.72 (m, 1 H, one proton of CH_2), 2.69-2.63 (m, 1 H, one proton of CH_2), 1.96-1.84 (m, 2 H, CH_2), 2.26-2.13 (m, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 147.0, 141.8, 136.4, 135.3, 134.3, 134.0, 133.6, 132.1, 130.2, 128.9, 128.42, 128.38, 128.0, 127.3, 126.7, 126.02, 125.95, 125.5, 42.0, 37.9, 33.9, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{25}H_{24}O+Na]^+$ 363.1719, found 363.1719.



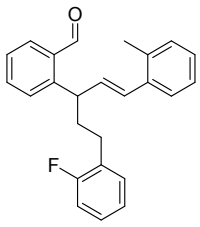
(E)-2-(6-phenyl-1-(o-tolyl)hex-1-en-3-yl)benzaldehyde 15e was prepared according to *general procedure C* as a clear oil (55.3 mg, 78%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.36 (s, 1 H, CHO), 7.83 (d, J = 7.5 Hz, 1 H, Ar-H), 7.56 (t, J = 7.5 Hz, 1 H, Ar-H), 7.45 (d, J = 8.0 Hz, 1 H, Ar-H), 7.40-7.37 (m, 2 H, Ar-H), 7.28-7.25 (m, 2 H, Ar-H), 7.19-7.12 (m, 6 H, Ar-H), 6.62 (d, J = 16.0 Hz, 1 H, =CH), 6.21 (dd, J_1 = 15.5 Hz, J_2 = 8.0 Hz, 1 H, CH=), 4.67 (q, J = 7.5 Hz, 1 H, CH), 2.66 (t, J = 7.3 Hz, 2 H, CH_2), 2.28 (s, 3 H, CH_3), 1.96-1.84 (m, 2 H, CH_2), 1.80-1.72 (m, 1 H, one proton of CH_2), 1.71-1.62 (m, 1 H, one proton of CH_2). ^{13}C-NMR (100 MHz, $CDCl_3$): 192.5, 147.3, 142.2, 136.4, 135.2, 134.6, 134.0, 133.6, 132.4, 130.2, 128.5, 128.4, 128.3, 128.1, 127.2, 126.5, 126.0, 125.7, 125.5, 42.3, 35.80, 35.77, 29.3, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{26}H_{26}O+Na]^+$ 377.1876, found 377.1874.



(E)-2-(5-(3-chlorophenyl)-1-(o-tolyl)pent-1-en-3-yl)benzaldehyde 15f was prepared according to *general procedure C* as a clear oil (51.0 mg, 68%):

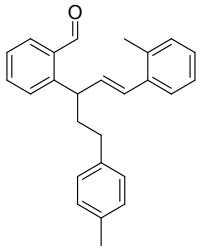
^{1}H-NMR (500 MHz, $CDCl_3$): 10.28 (s, 1 H, CHO), 7.84 (d, J = 7.5 Hz, 1 H, Ar-H), 7.59 (t, J = 7.5 Hz, 1 H, Ar-H), 7.51 (d, J = 7.5 Hz, 1 H, Ar-H), 7.43-7.39 (m, 2 H, Ar-H), 7.22-7.13 (m, 6 H, Ar-H), 7.03 (d, J = 7.0 Hz, 1 H, Ar-H), 6.67 (d, J = 15.5 Hz, 1 H, =CH), 6.25 (dd, J_1 = 15.5 Hz, J_2 = 8.0 Hz, 1 H, CH=), 4.69 (q, J = 7.5 Hz, 1 H, CH), 2.75-2.69 (m, 1 H, one proton of CH_2), 2.66-2.60 (m, 1 H, one proton of CH_2), 2.31 (s, 3 H, CH_3), 2.21-2.10 (m, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 146.8, 143.9, 136.3, 135.3, 134.2, 134.04, 134.00, 133.6, 132.8, 130.2, 129.7, 129.1, 128.5, 127.9, 127.4, 126.8, 126.6, 126.1, 126.0, 125.5, 42.1, 37.7, 33.6, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{25}H_{23}ClO+Na]^+$ 397.1330, found 397.1327.



(E)-2-(5-(2-fluorophenyl)-1-(o-tolyl)pent-1-en-3-yl)benzaldehyde 15g was prepared according to *general procedure C* as a clear oil (56.6 mg, 79%):

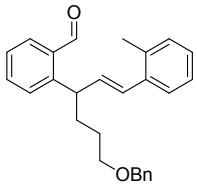
^{1}H-NMR (500 MHz, $CDCl_3$): 10.31 (s, 1 H, CHO), 7.83 (d, J = 7.5 Hz, 1 H, Ar-H), 7.59 (t, J = 7.5 Hz, 1 H, Ar-H), 7.52 (d, J = 8.0 Hz, 1 H, Ar-H), 7.42-7.39 (m, 2 H, Ar-H), 7.16-7.13 (m, 5 H, Ar-H), 7.05 (t, J = 7.3 Hz, 1 H, Ar-H), 7.01 (t, J = 9.3 Hz, 1 H, Ar-H), 6.70 (d, J = 1.5 Hz, 1 H, =CH), 6.26 (dd, J_1 = 15.8 Hz, J_2 = 7.8 Hz, 1 H, CH=), 4.70 (q, J = 7.5 Hz, 1 H, CH),

2.82-2.76 (m, 1 H, one proton of CH_2), 2.72-2.66 (m, 1 H, one proton of CH_2), 2.31 (s, 3 H, CH_3), 2.24-2.12 (m, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 161.1 (d, J = 243.8 Hz), 146.8, 136.4, 135.3, 134.1, 134.0, 133.6, 132.4, 130.6 (d, J = 5.0 Hz), 130.2, 129.0, 128.7 (d, J = 15.0 Hz), 128.0, 127.7 (d, J = 8.8 Hz), 127.3, 126.7, 126.0, 125.6, 124.0 (d, J = 2.5 Hz), 115.3 (d, J = 22.5 Hz), 42.2, 36.3, 27.4 (d, J = 2.5 Hz), 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{25}H_{23}FO+Na]^+$ 381.1625, found 381.1625.



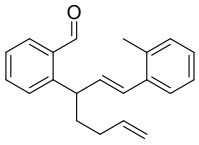
(E)-2-(1-(o-tolyl)-5-(p-tolyl)pent-1-en-3-yl)benzaldehyde 15h was prepared according to *general procedure C* as a clear oil (62.4 mg, 88%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.30 (s, 1 H, CHO), 7.86 (d, J = 7.5 Hz, 1 H, Ar-H), 7.59 (t, J = 7.5 Hz, 1 H, Ar-H), 7.51 (d, J = 7.5 Hz, 1 H, Ar-H), 7.42-7.39 (m, 2 H, Ar-H), 7.18-7.14 (m, 3 H, Ar-H), 7.10 (d, J = 8.0 Hz, 2 H, Ar-H), 7.06 (d, J = 8.0 Hz, 2 H, Ar-H), 6.67 (d, J = 15.5 Hz, 1 H, =CH), 6.26 (dd, J_1 = 15.8 Hz, J_2 = 7.8 Hz, 1 H, CH=), 4.64 (q, J = 7.5 Hz, 1 H, CH), 2.73-2.67 (m, 1 H, one proton of CH_2), 2.65-2.59 (m, 1 H, one proton of CH_2), 2.32 (s, 3 H, CH_3), 2.30 (s, 3 H, CH_3), 2.24-2.11 (m, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.3, 147.1, 138.6, 136.4, 135.4, 135.3, 134.4, 134.0, 133.6, 131.9, 130.2, 129.1, 128.8, 128.3, 128.0, 127.3, 126.6, 126.0, 125.5, 42.0, 38.0, 33.4, 21.0, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{26}H_{26}O+Na]^+$ 377.1876, found 377.1874.



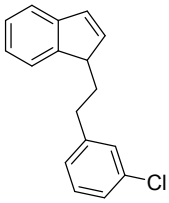
(E)-2-(6-(benzyloxy)-1-(o-tolyl)hex-1-en-3-yl)benzaldehyde 15i was prepared according to *general procedure C* as a clear oil (50.0 mg, 65%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.36 (s, 1 H, CHO), 7.83 (d, J = 7.5 Hz, 1 H, Ar-H), 7.57 (t, J = 7.5 Hz, 1 H, Ar-H), 7.49 (d, J = 7.5 Hz, 1 H, Ar-H), 7.40-7.37 (m, 2 H, Ar-H), 7.35-7.32 (m, 4 H, Ar-H), 7.29-7.27 (m, 1 H, Ar-H), 7.14-7.12 (m, 3 H, Ar-H), 6.63 (d, J = 15.5 Hz, 1 H, =CH), 6.22 (dd, J_1 = 15.8 Hz, J_2 = 7.8 Hz, 1 H, CH=), 4.65 (q, J = 7.5 Hz, 1 H, CH), 4.49 (s, 2 H, CH_2), 3.50 (t, J = 6.0 Hz, 2 H, CH_2), 2.28 (s, 3 H, CH_3), 2.03-1.89 (m, 2 H, CH_2), 1.78-1.70 (m, 1 H, one proton of CH_2), 1.68-1.61 (m, 1 H, one proton of CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.5, 147.2, 138.5, 136.4, 135.2, 134.5, 134.0, 133.6, 132.3, 130.2, 128.6, 128.3, 128.1, 127.6, 127.5, 127.2, 126.6, 126.0, 125.5, 72.9, 70.1, 42.2, 32.8, 27.8, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{27}H_{28}O_2+Na]^+$ 407.1982, found 407.1977.



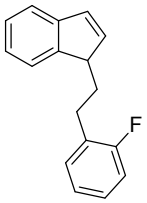
(E)-2-(1-(o-tolyl)hepta-1,6-dien-3-yl)benzaldehyde 15j was prepared according to *general procedure C* as a clear oil (8.7 mg, 15%):

^{1}H-NMR (500 MHz, $CDCl_3$): 10.39 (s, 1 H, CHO), 7.85 (d, J = 7.5 Hz, 1 H, Ar-H), 7.58 (t, J = 8.0 Hz, 1 H, Ar-H), 7.49 (d, J = 8.0 Hz, 1 H, Ar-H), 7.41-7.38 (m, 2 H, Ar-H), 7.14-7.12 (m, 3 H, Ar-H), 6.64 (d, J = 16.0 Hz, 1 H, =CH), 6.22 (dd, J_1 = 16.0 Hz, J_2 = 7.8 Hz, 1 H, CH=), 5.88-5.80 (m, 1 H, CH=), 5.02-4.98 (m, 2 H, =CH_2), 4.65 (q, J = 7.5 Hz, 1 H, CH), 2.29 (s, 3 H, CH_3), 2.18-2.07 (m, 2 H, CH_2), 2.02-1.89 (m, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 192.4, 147.2, 138.1, 136.4, 135.3, 134.4, 134.0, 133.6, 132.0, 130.2, 128.8, 128.0, 127.3, 126.6, 126.0, 125.5, 115.2, 41.8, 35.3, 31.7, 19.8. HRMS (FTMS + p NSI): m/z calcd for $[C_{21}H_{22}O+Na]^+$ 313.1563, found 313.1563.



1-(2-fluorophenethyl)-1*H*-indene 16f was prepared according to *general procedure D* as a clear oil (58.9 mg, 83%):

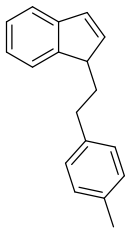
^{1}H-NMR (500 MHz, $CDCl_3$): 7.43 (d, J = 7.0 Hz, 1 H, Ar-H), 7.38 (d, J = 7.5 Hz, 1 H, Ar-H), 7.28 (t, J = 7.0 Hz, 1 H, Ar-H), 7.23-7.16 (m, 4 H, Ar-H), 7.06 (d, J = 7.5 Hz, 1 H, Ar-H), 6.86 (dd, J_1 = 5.5 Hz, J_2 = 1.0 Hz, 1 H, CH=), 6.56 (dd, J_1 = 5.5 Hz, J_2 = 1.5 Hz, 1 H, CH=), 3.54 (t, J = 6.5 Hz, 1 H, CH), 2.70-2.64 (m, 1 H, one proton of CH_2), 2.62-2.56 (m, 1 H, one proton of CH_2), 2.29-2.20 (m, 1 H, one proton of CH_2), 1.91-1.84 (m, 1 H, one proton of CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 147.2, 144.3, 138.5, 134.1, 131.4, 129.6, 128.5, 126.61, 126.55, 126.0, 124.8, 122.8, 121.1, 49.8, 33.0, 32.9. HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{15}Cl+H]^+$ 255.0935, found 255.0933.



1-(3-chlorophenethyl)-1*H*-indene 16g was prepared according to *general procedure D* as a clear oil (38.6 mg, 81%):

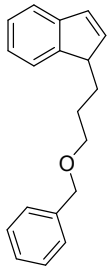
^{1}H-NMR (500 MHz, $CDCl_3$): 7.44 (d, J = 7.0 Hz, 1 H, Ar-H), 7.37 (d, J = 7.5 Hz, 1 H, Ar-H), 7.27 (t, J = 7.5 Hz, 1 H, Ar-H), 7.22-7.15 (m, 3 H, Ar-H), 7.05 (t, J = 7.8 Hz, 1 H, Ar-H), 7.01 (t, J = 9.0 Hz, 1 H, Ar-H), 6.86 (dd, J_1 = 5.5 Hz, J_2 = 1.0 Hz, 1 H, CH=), 6.61 (dd, J_1 = 5.5 Hz, J_2 = 1.5 Hz, 1 H, CH=), 3.53 (t, J = 6.8 Hz, 1 H, CH), 2.79-2.73 (m, 1 H, one proton of CH_2), 2.69-2.63 (m, 1 H, one proton of CH_2), 2.28-2.21 (m, 1 H, one proton of CH_2), 1.89-1.82 (m, 1 H, one proton of CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 161.1 (d, J = 242.5 Hz), 147.3, 144.3, 138.7, 131.3, 130.5 (d, J = 2.0 Hz), 129.0 (d, J = 16.3 Hz), 127.6 (d, J = 8.8 Hz), 126.5, 124.8,

123.9 (d, J = 3.8 Hz), 122.8, 121.1, 115.2 (d, J = 22.5 Hz), 50.0, 31.8, 26.8 (d, J = 2.5 Hz). HRMS (FTMS + p NSI): m/z calcd for $[C_{17}H_{15}F+H]^+$ 239.1231, found 239.1231.



1-(4-methylphenethyl)-1*H*-indene 16h was prepared according to *general procedure D* as a clear oil (38.0 mg, 81%):

^{1}H-NMR (500 MHz, $CDCl_3$): 7.44 (d, J = 7.0 Hz, 1 H, Ar-H), 7.37 (d, J = 7.5 Hz, 1 H, Ar-H), 7.27 (t, J = 7.3 Hz, 1 H, Ar-H), 7.21 (t, J = 7.3 Hz, 1 H, Ar-H), 7.10 (s, 4 H, Ar-H), 6.85 (dd, J_1 = 5.5 Hz, J_2 = 1.0 Hz, 1 H, CH=), 6.60 (dd, J_1 = 5.5 Hz, J_2 = 1.5 Hz, 1 H, CH=), 3.53 (t, J = 6.8 Hz, 1 H, CH), 2.72-2.60 (m, 2 H, CH_2), 2.33 (s, 3 H, CH_3), 2.26-2.19 (m, 1 H, one proton of CH_2), 1.87-1.80 (m, 1 H, one proton of CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 147.6, 144.3, 139.2, 138.9, 135.3, 131.1, 129.1, 128.2, 126.5, 124.7, 122.8, 121.0, 50.0, 33.4, 33.2, 21.0. HRMS (FTMS + p NSI): m/z calcd for $[C_{18}H_{18}+H]^+$ 235.1481, found 235.1481.



1-(3-(benzyloxy)propyl)-1*H*-indene 16i was prepared according to *general procedure D* as a clear oil (42.3 mg, 80%):

^{1}H-NMR (500 MHz, $CDCl_3$): 7.42 (d, J = 7.5 Hz, 1 H, Ar-H), 7.36-7.33 (m, 5 H, Ar-H), 7.30-7.25 (m, 2 H, Ar-H), 7.19 (t, J = 7.5 Hz, 1 H, Ar-H), 6.81 (d, J = 5.5 Hz, 1 H, CH=), 6.54 (d, J = 5.5 Hz, 1 H, CH=), 4.49 (s, 2 H, OCH_2), 3.51-3.46 (m, 3 H, CH_2 and one proton of CH_2), 1.78-1.70 (m, 1 H, one proton of CH_2), 1.69-1.59 (m, 2 H, CH_2). ^{13}C-NMR (125 MHz, $CDCl_3$): 147.6, 144.3, 139.1, 138.6, 131.0, 128.4, 127.6, 127.5, 126.4, 124.7, 122.9, 121.0, 72.9, 70.4, 50.1, 28.0, 27.5. HRMS (FTMS + p NSI): m/z calcd for $[C_{19}H_{20}O+H]^+$ 287.1406, found 287.1407.

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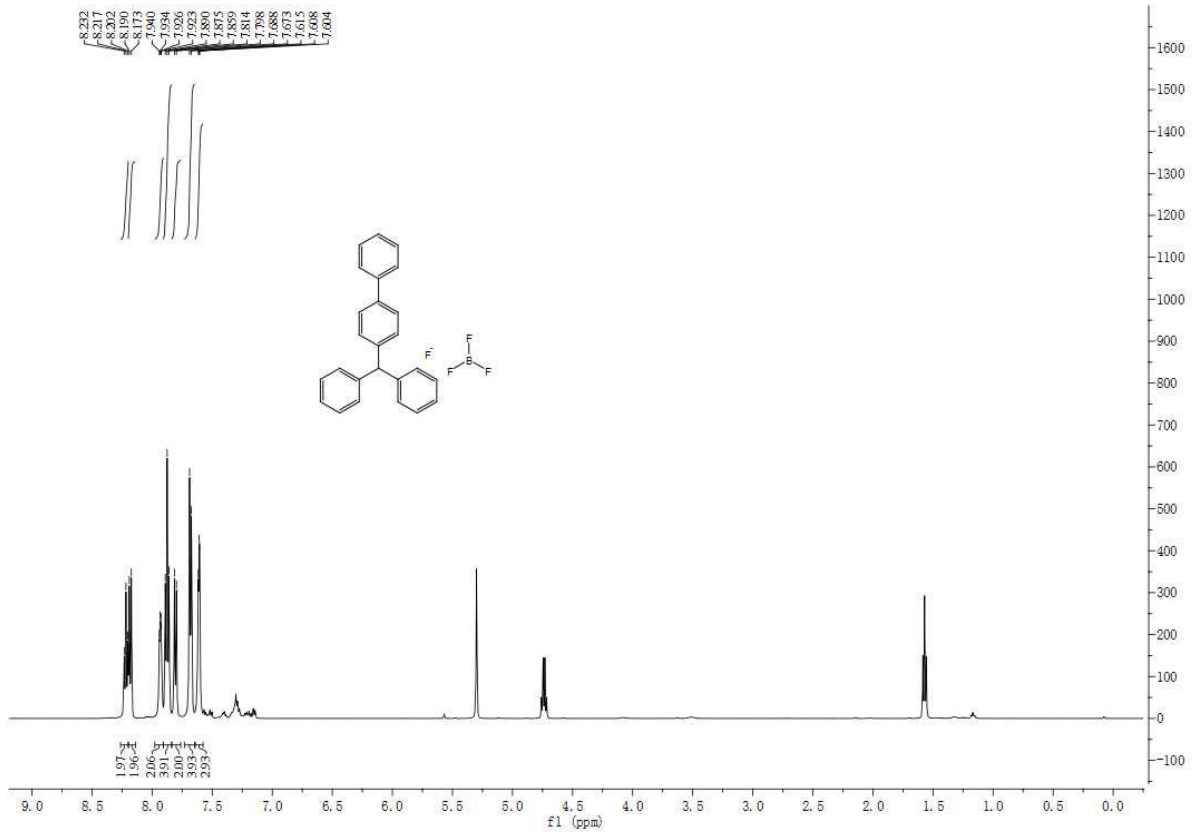
NMR-Spectra: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7.859
7.814
7.798
7.688
7.673
7.615
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7.604
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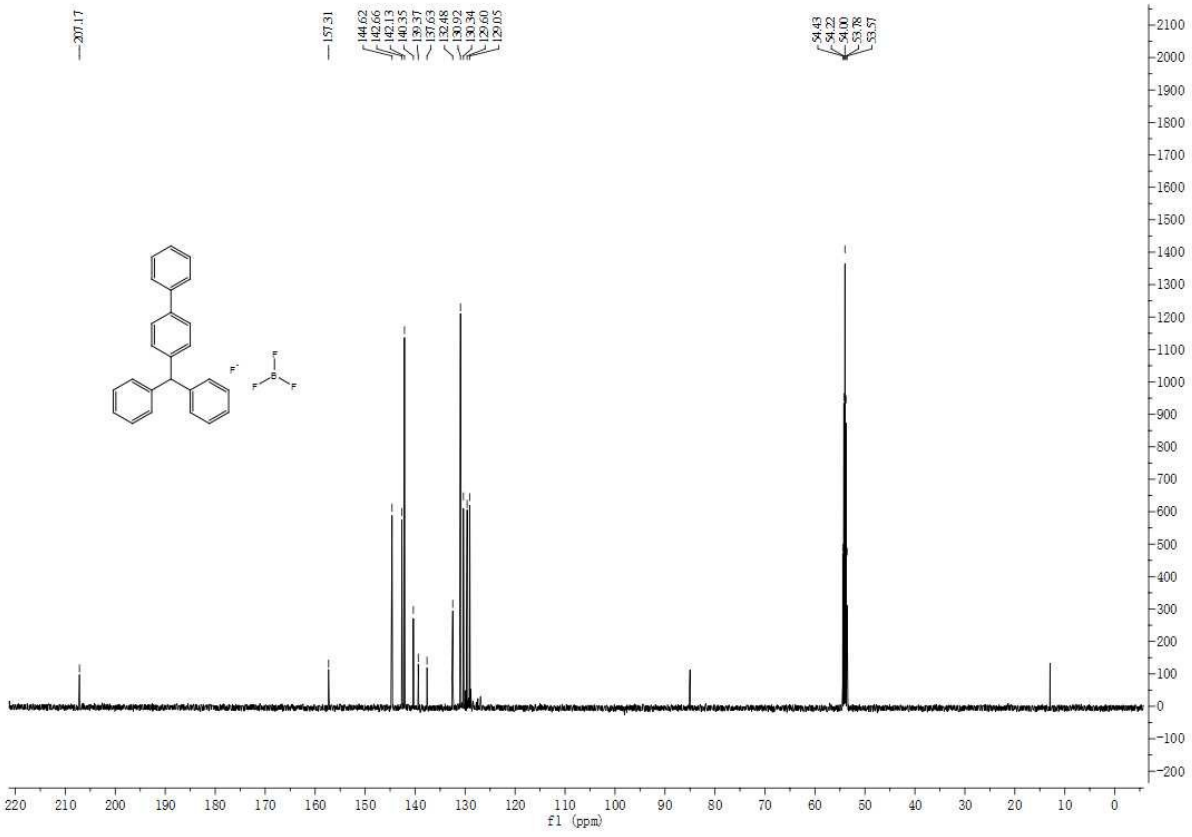
1.96
2.06
3.91
2.00
3.93
2.93
f1 (ppm)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54.00
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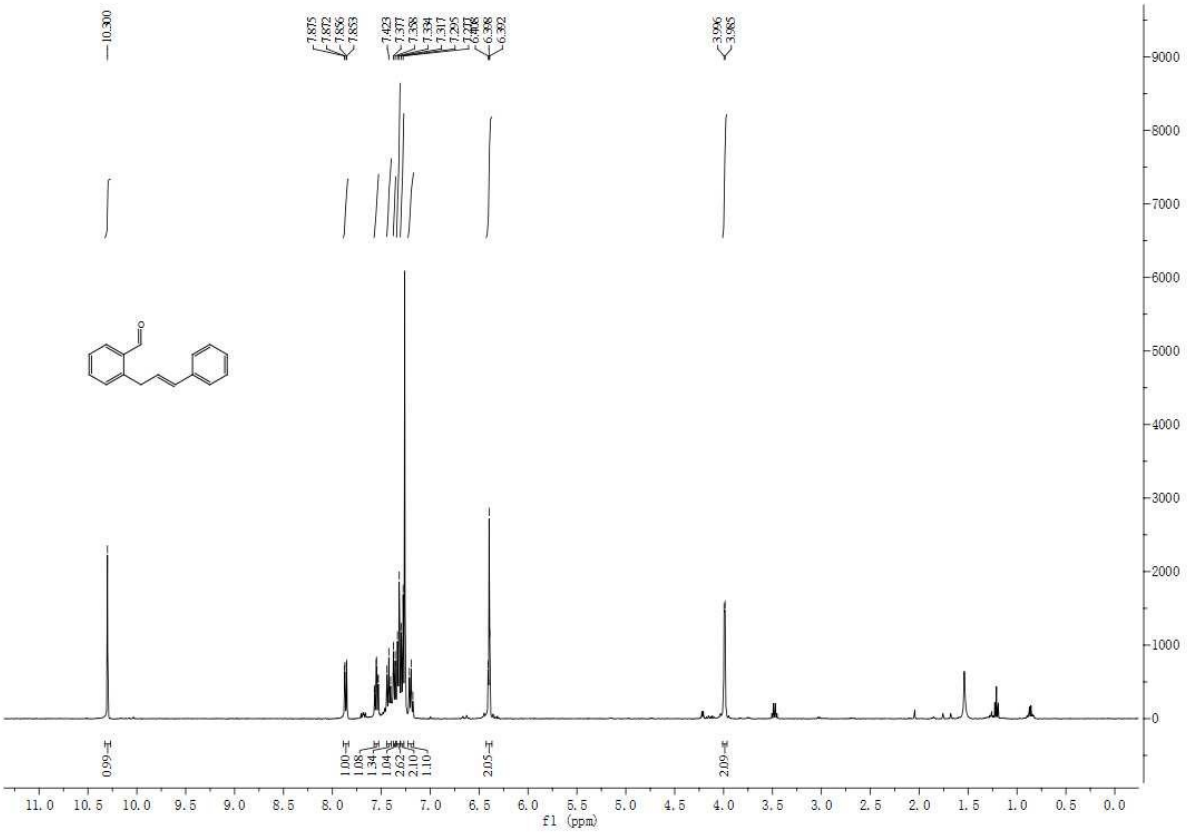
53.57
f1 (ppm)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3.996
3.985
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1.08
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1.04
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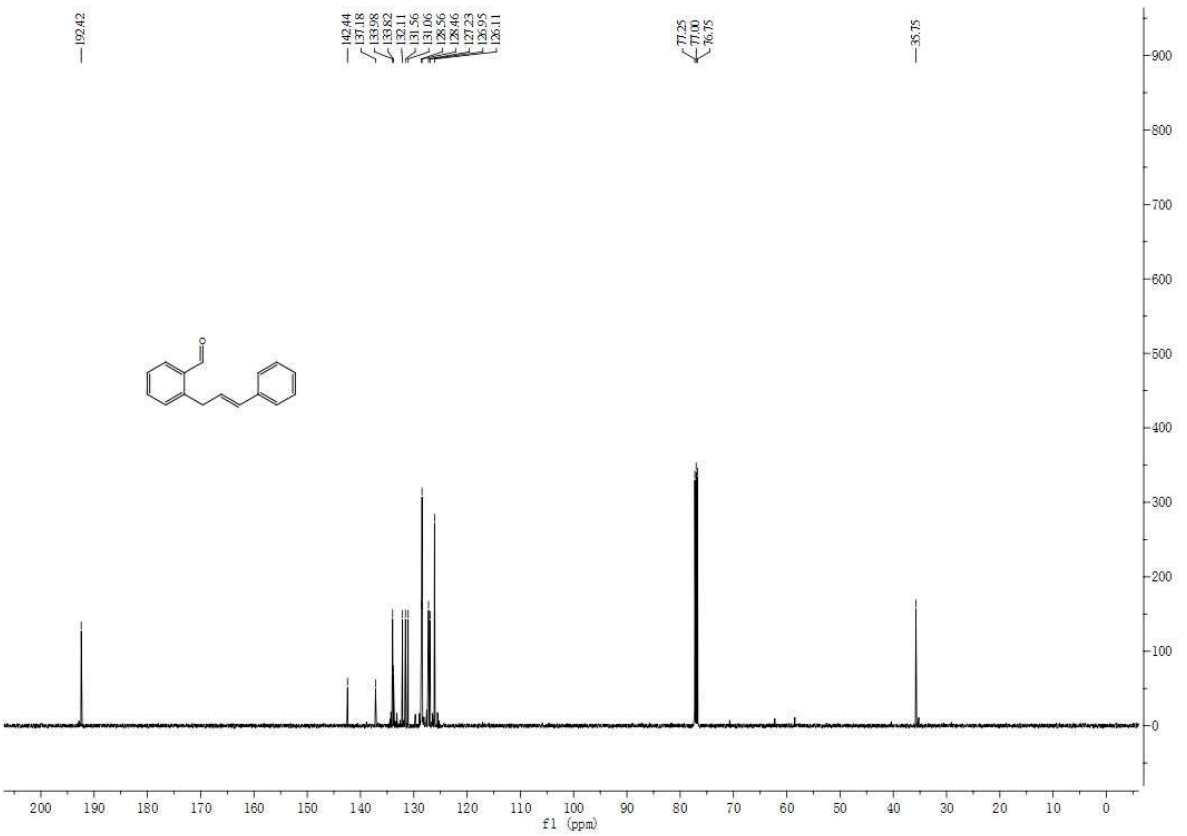
2.09
f1 (ppm)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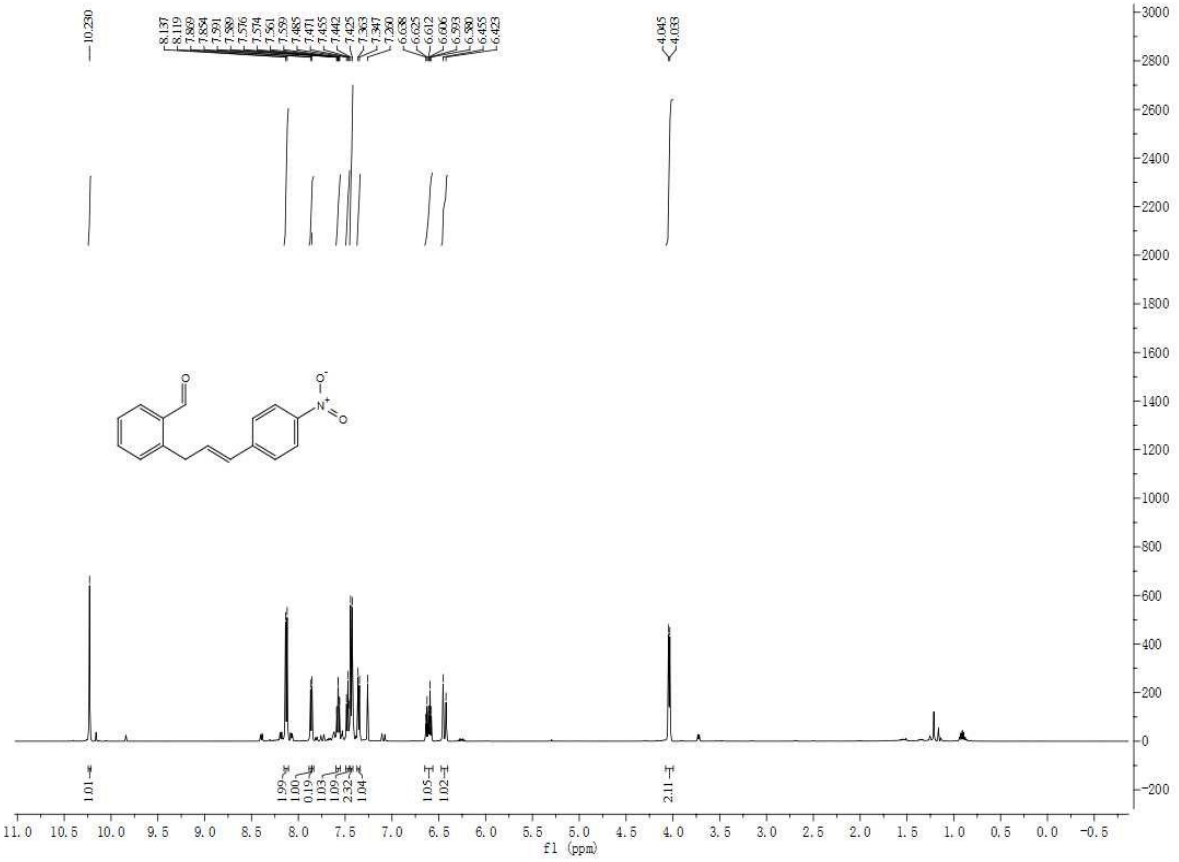
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6.625
6.612
6.606
6.593
6.580
6.455
6.423
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4.033
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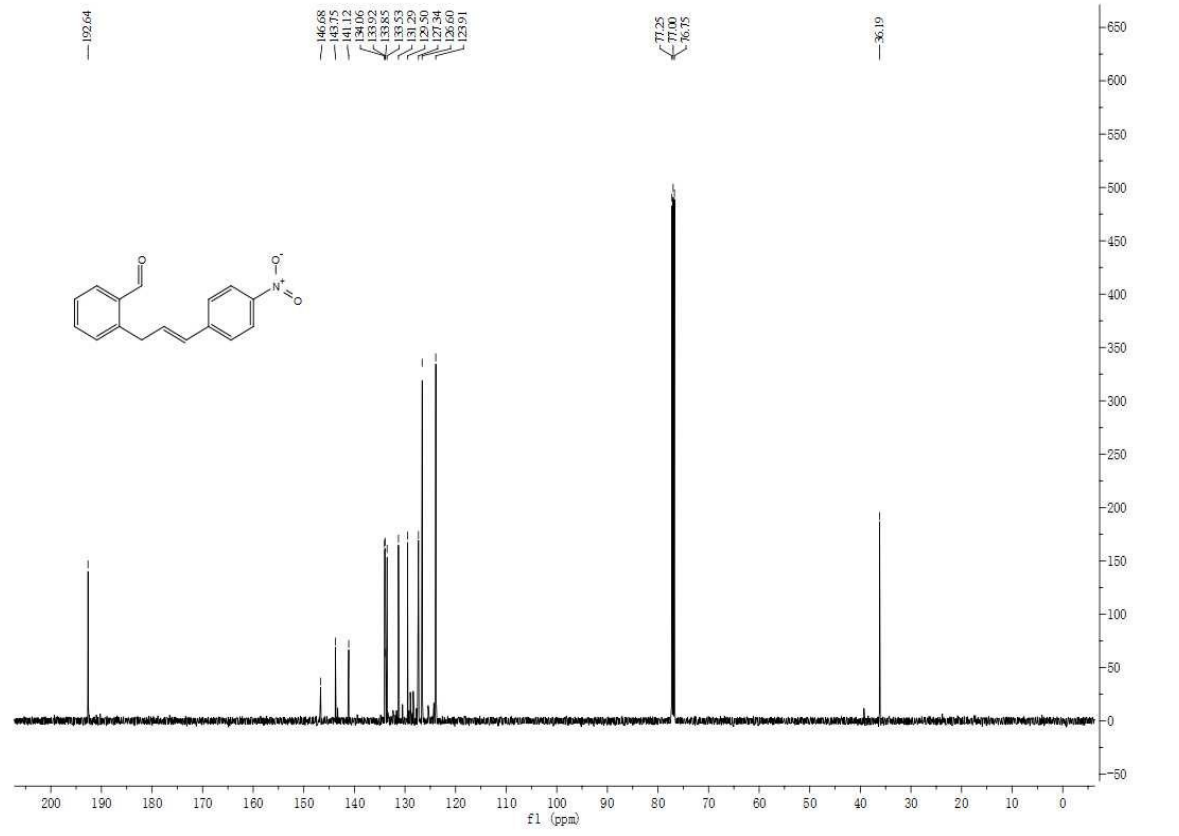
2.11
f1 (ppm)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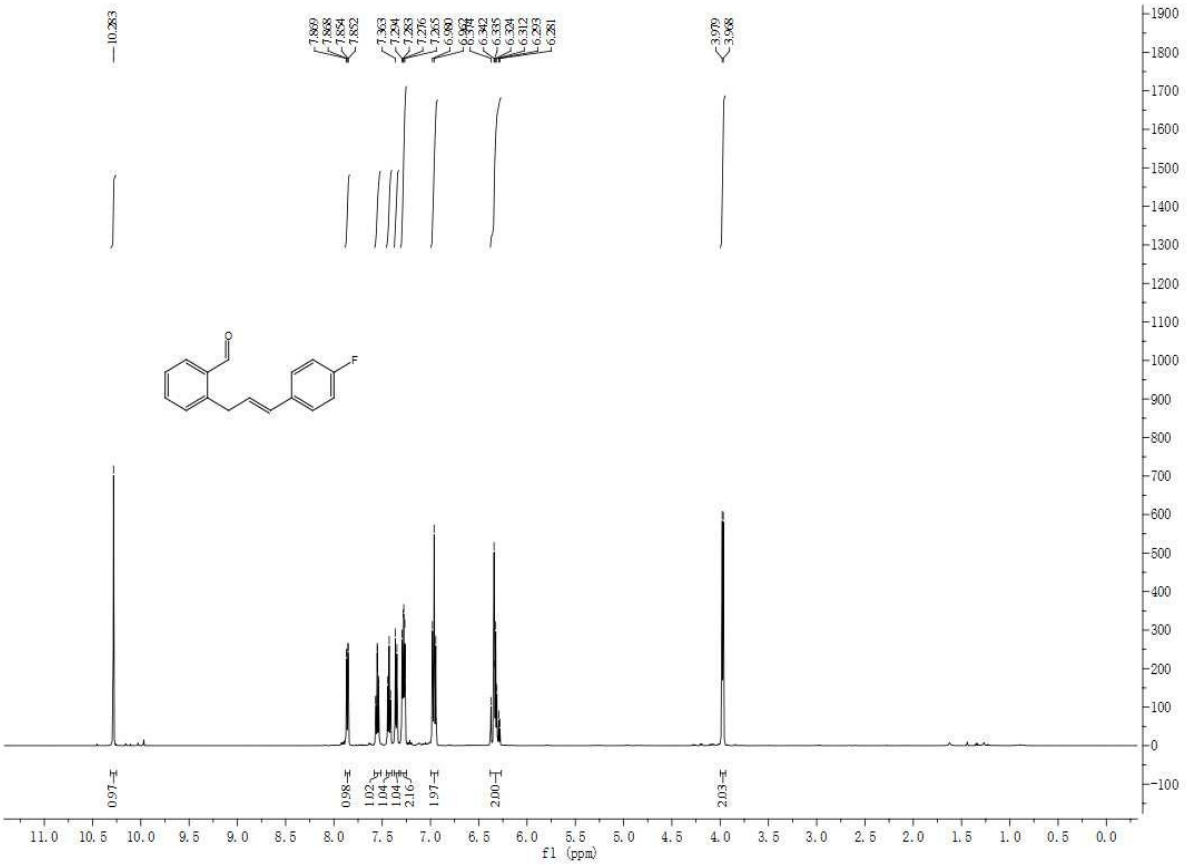
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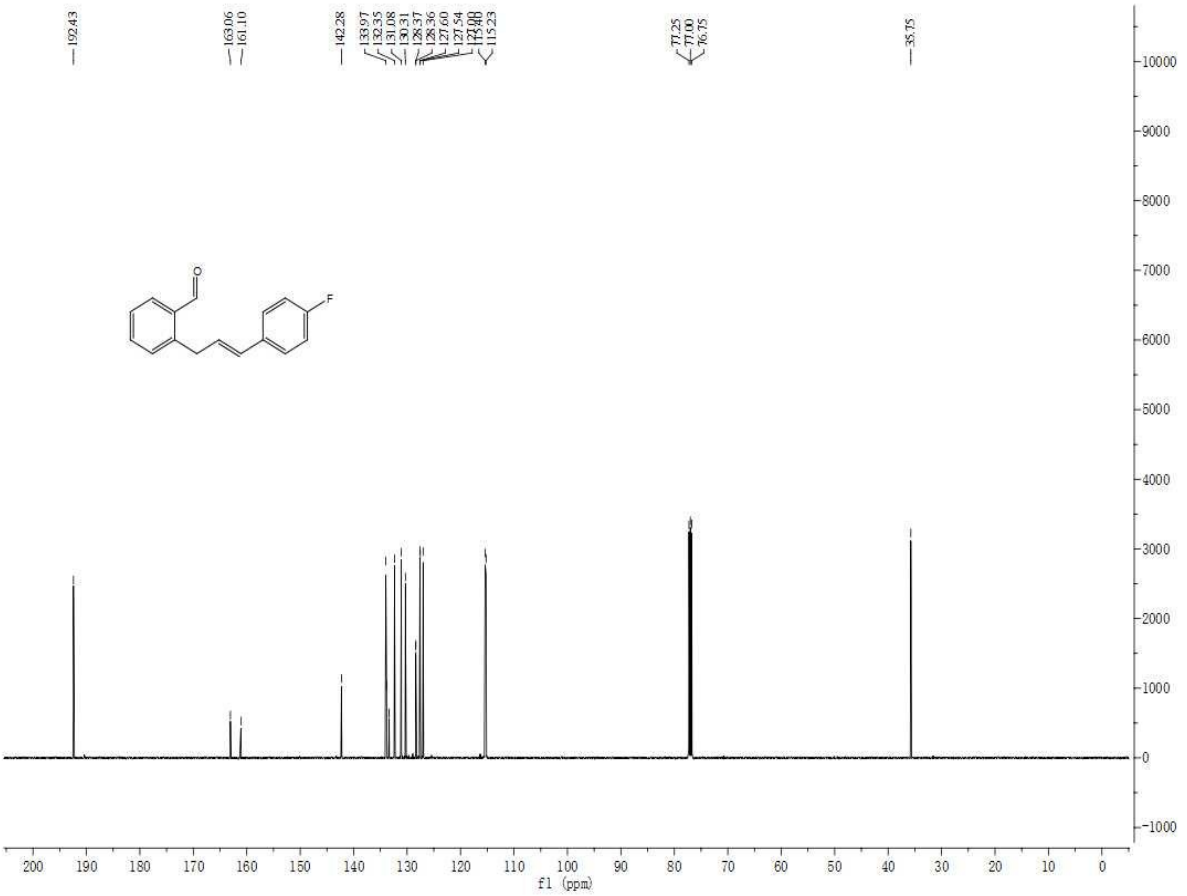
f1 (ppm)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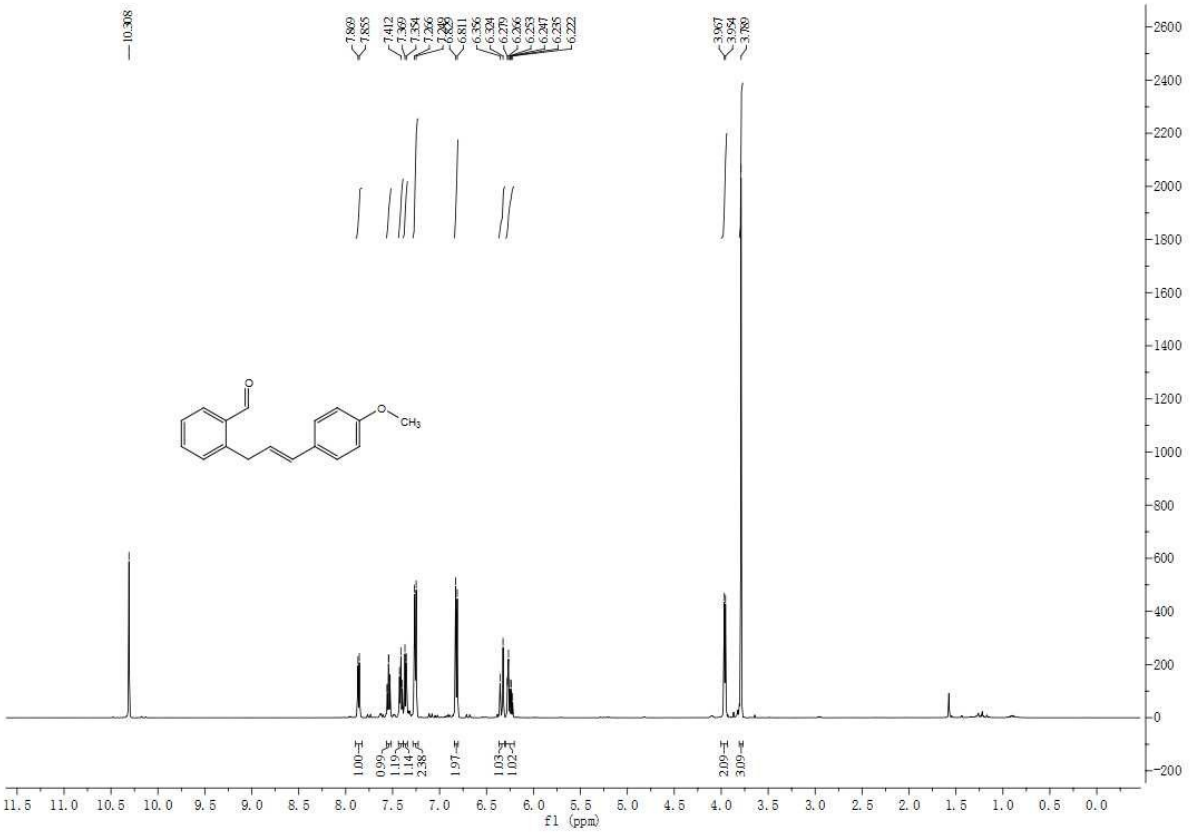
O
F
f1 (ppm)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6.247
6.235
6.222
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3.954
3.789
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O
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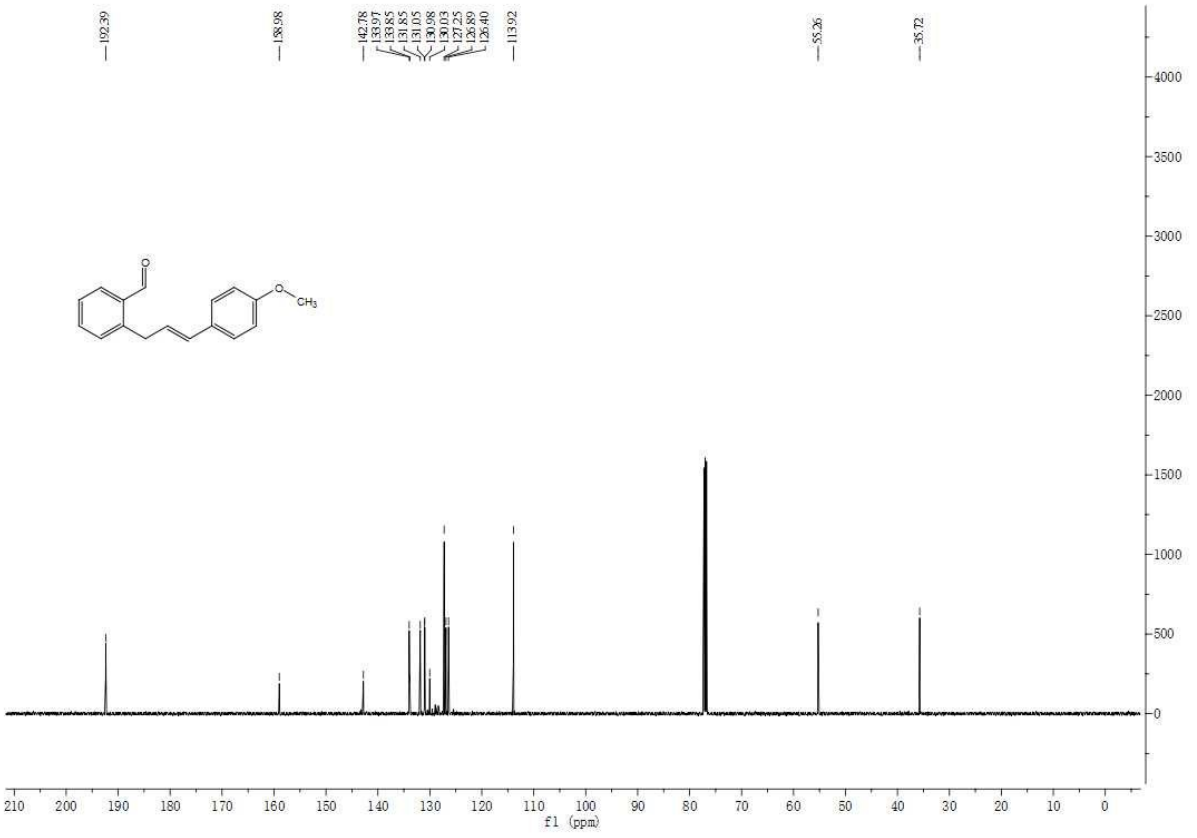
f1 (ppm)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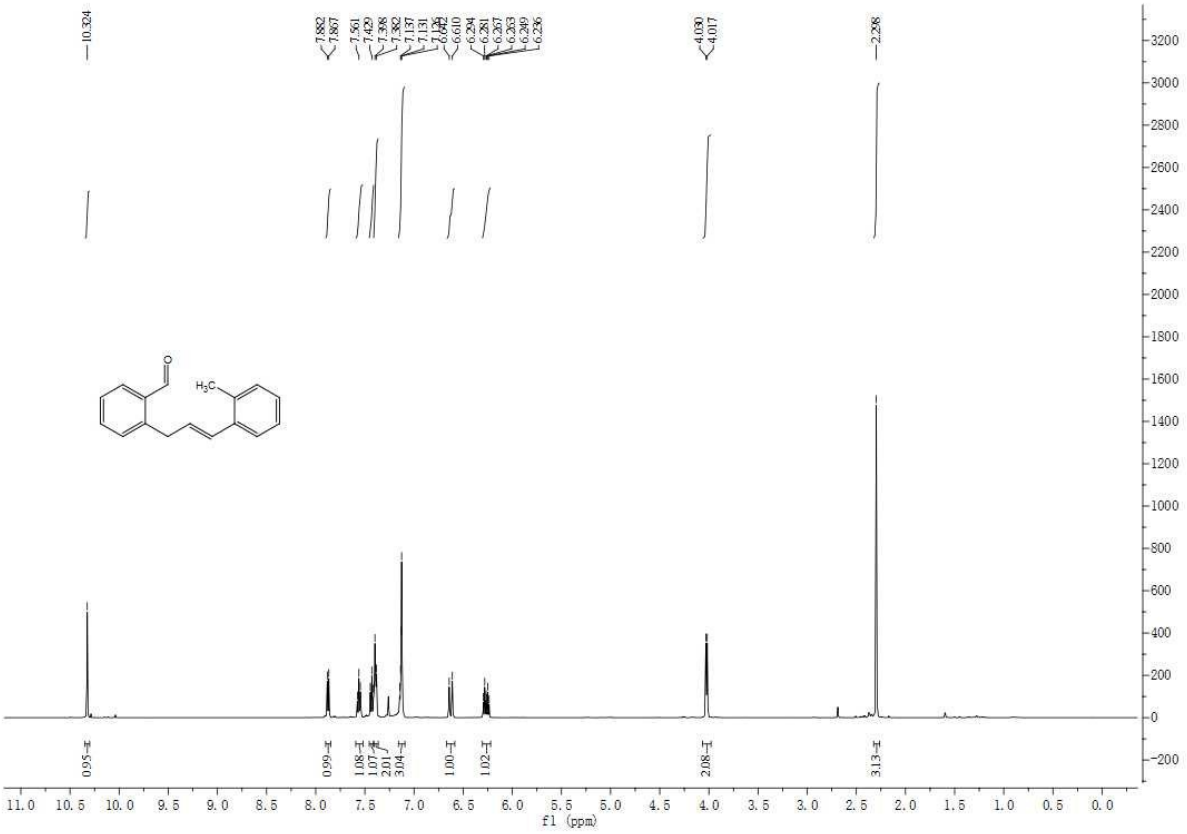
O
O
CH3
f1 (ppm)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6.249
6.236
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4.017
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O
H3C
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0.99
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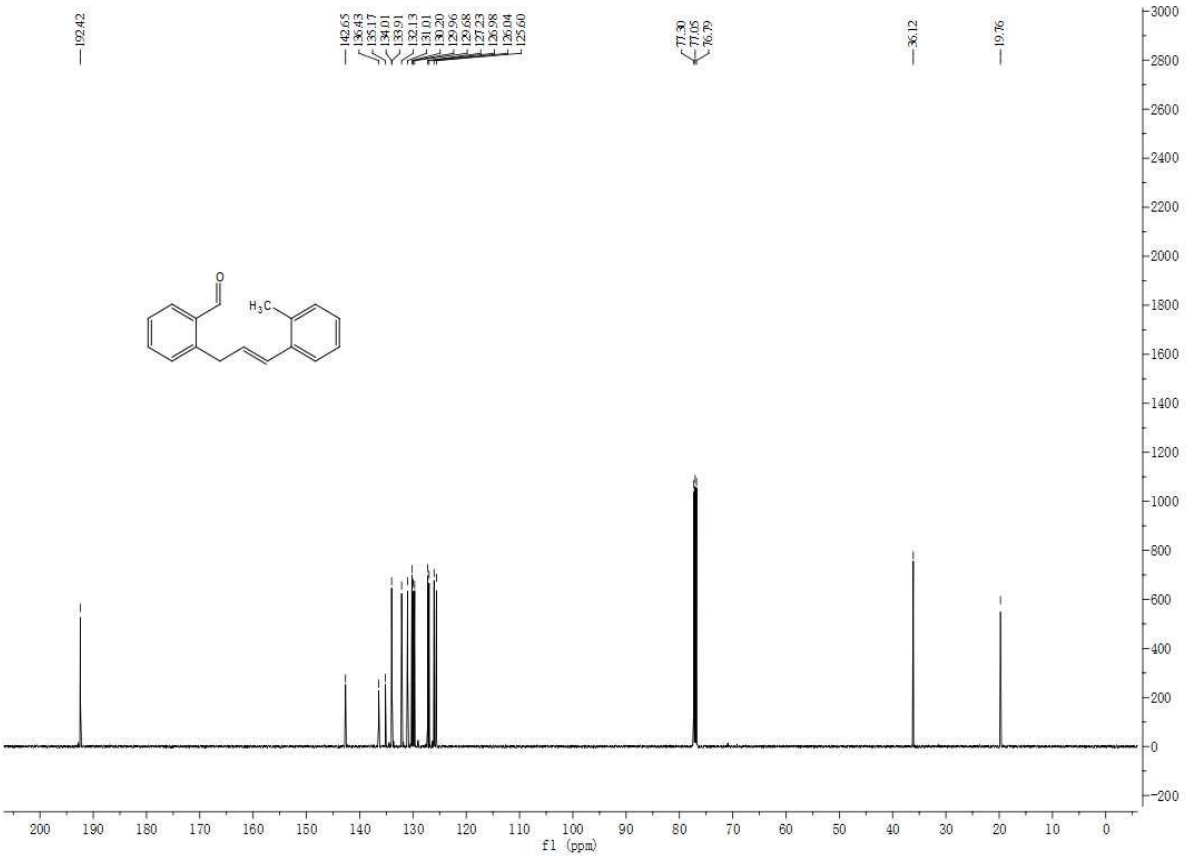
2.01
3.04
1.00
1.02
2.08
3.13
f1 (ppm)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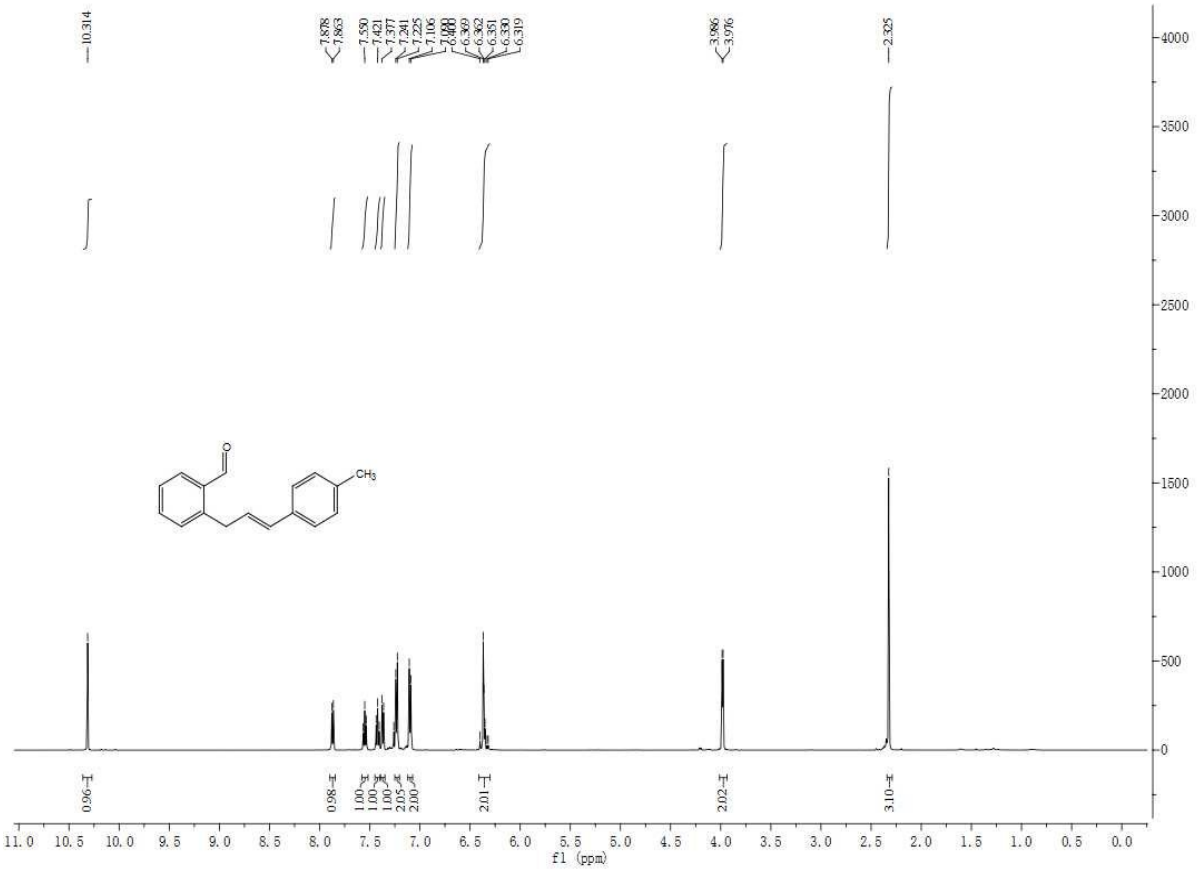
135.17
134.01
133.91
132.13
131.01
130.20
129.96
129.68
127.23
126.98
126.04
125.60
77.30
77.05
76.79
36.12
19.76
O
H3C
f1 (ppm)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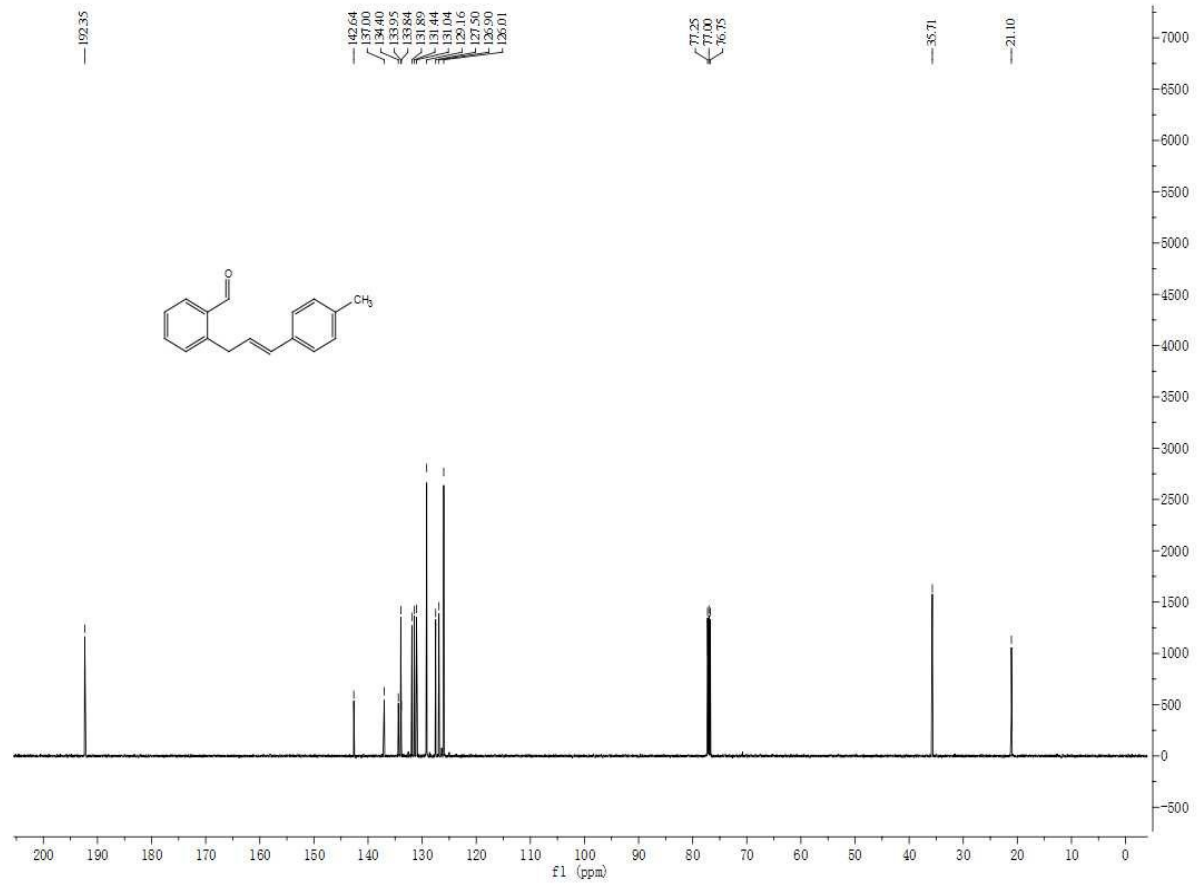
7.377
7.241
7.225
7.106
6.369
6.362
6.351
6.330
6.319
3.986
3.976
2.325
CH3
0.96
0.98
1.00
1.00
1.00
2.05
2.00
2.01
2.02
3.10
f1 (ppm)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133.84
131.89
131.44
131.04
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127.50
126.90
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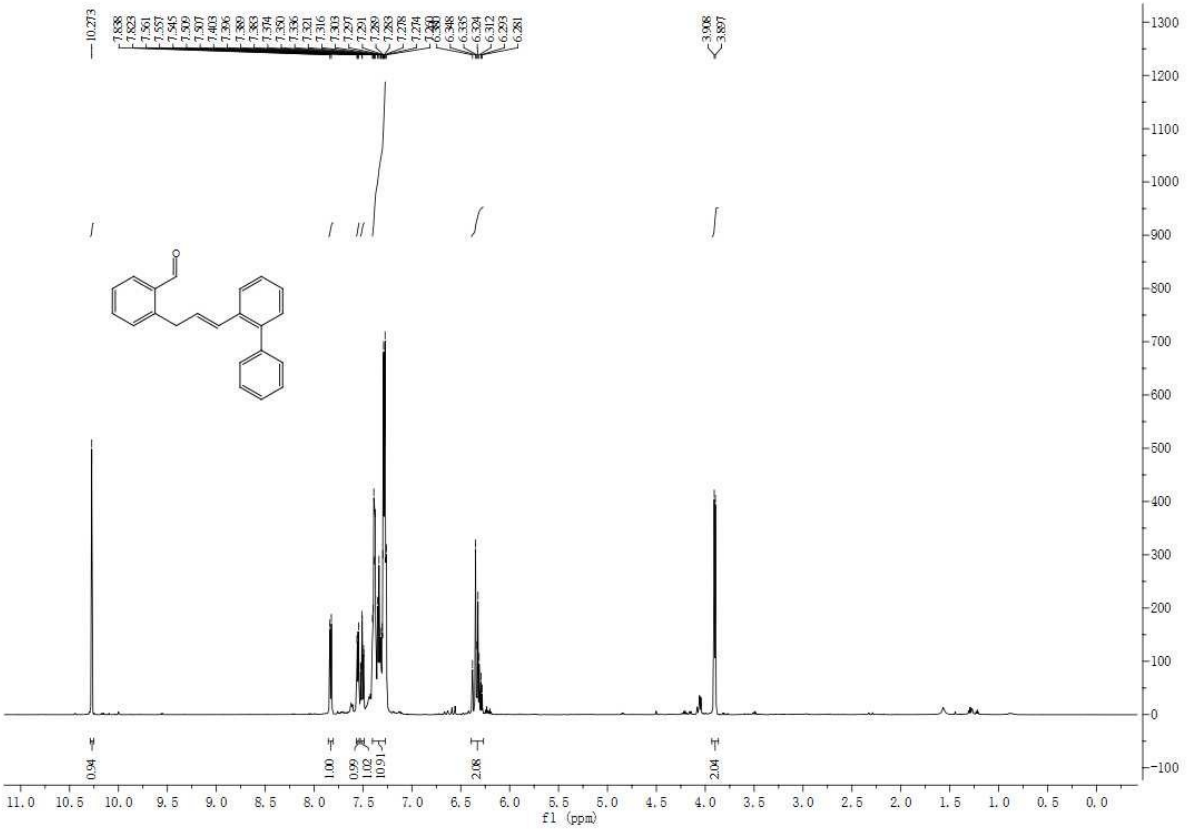
77.00
76.75
35.71
21.10
CH3
f1 (ppm)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7.383
7.374
7.369
7.336
7.321
7.316
7.305
7.297
7.291
7.289
7.283
7.278
7.274
6.398
6.355
6.334
6.312
6.293
6.281
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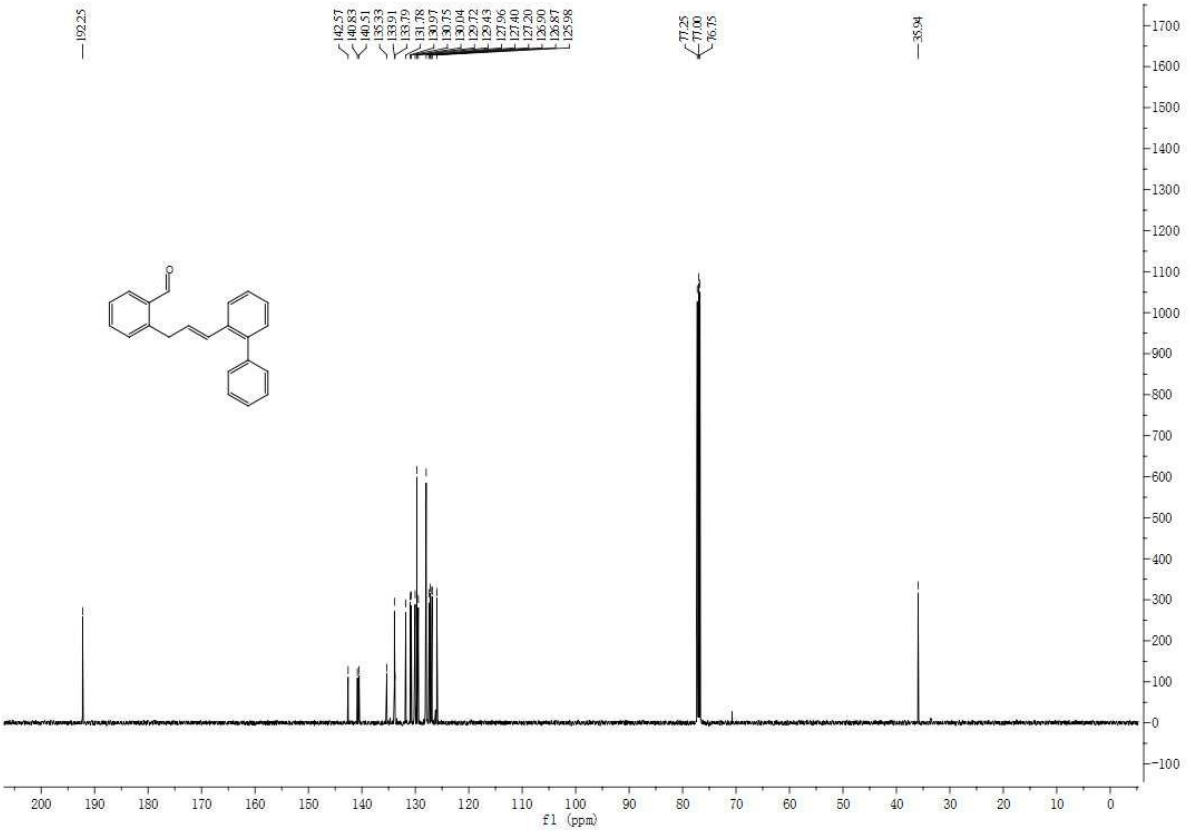
3.897
0.94
1.00
0.99
1.02
10.91
2.08
2.04
f1 (ppm)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131.78
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130.75
130.04
129.72
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127.20
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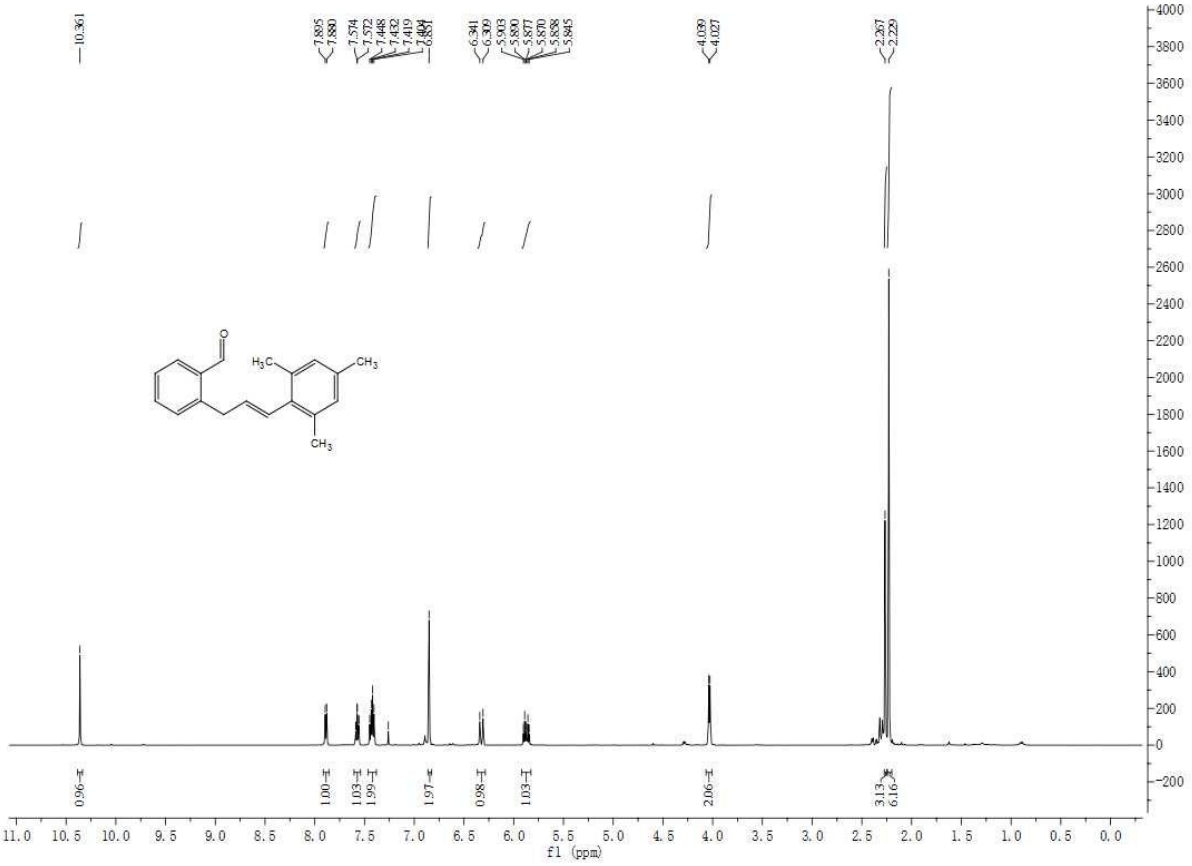
126.87
125.98
77.25
77.00
76.75
35.94
f1 (ppm)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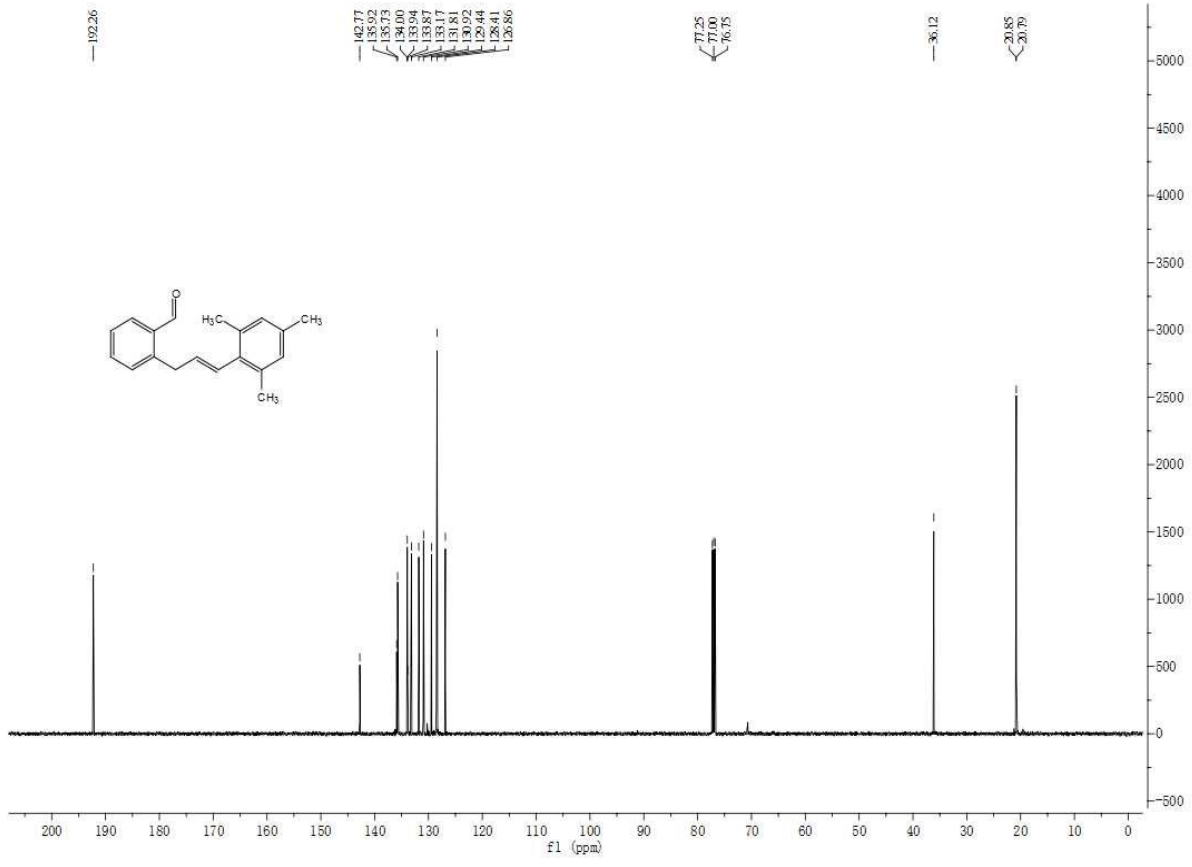
7.419
7.404
6.851
6.341
6.309
5.903
5.890
5.877
5.870
5.858
5.845
4.039
4.027
2.267
2.229
0.96
1.00
1.03
1.99
1.97
0.98
1.03
2.06
3.13
6.16
f1 (ppm)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77.00
76.75
36.12
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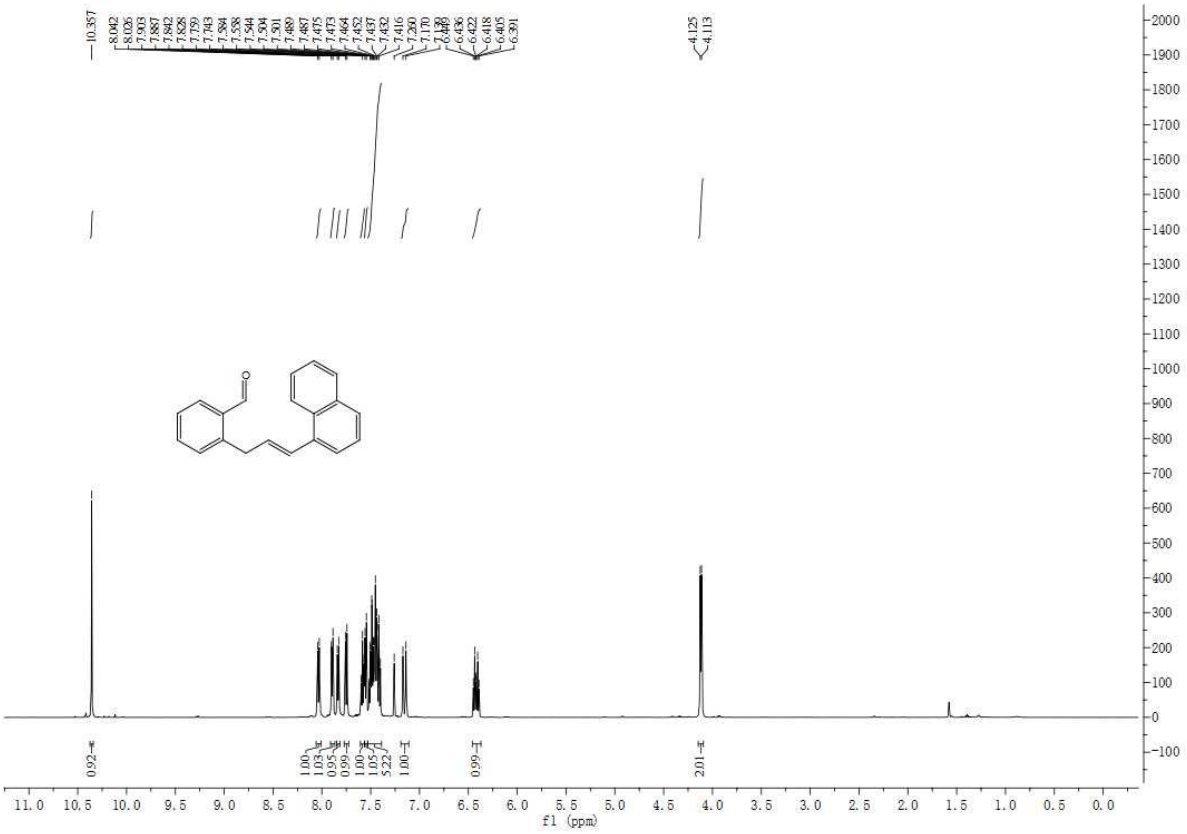
20.79
f1 (ppm)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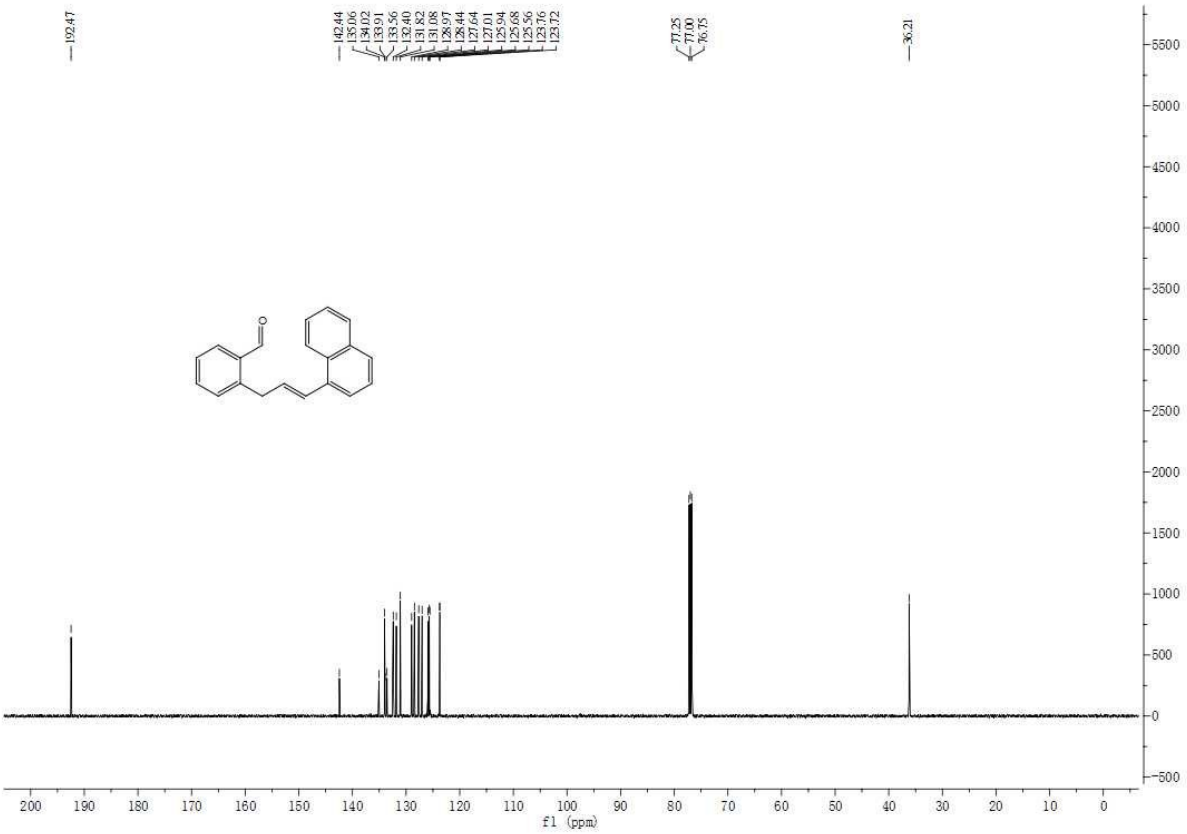
7.739
7.743
7.384
7.558
7.544
7.504
7.501
7.489
7.487
7.475
7.473
7.464
7.452
7.437
7.432
7.416
7.200
7.170
6.436
6.422
6.418
6.405
6.391
4.125
4.113
0.92
1.00
1.03
0.95
0.99
1.00
1.05
5.22
1.00
0.99
2.01
f1 (ppm)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135.06
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133.56
133.40
131.82
131.08
128.97
128.44
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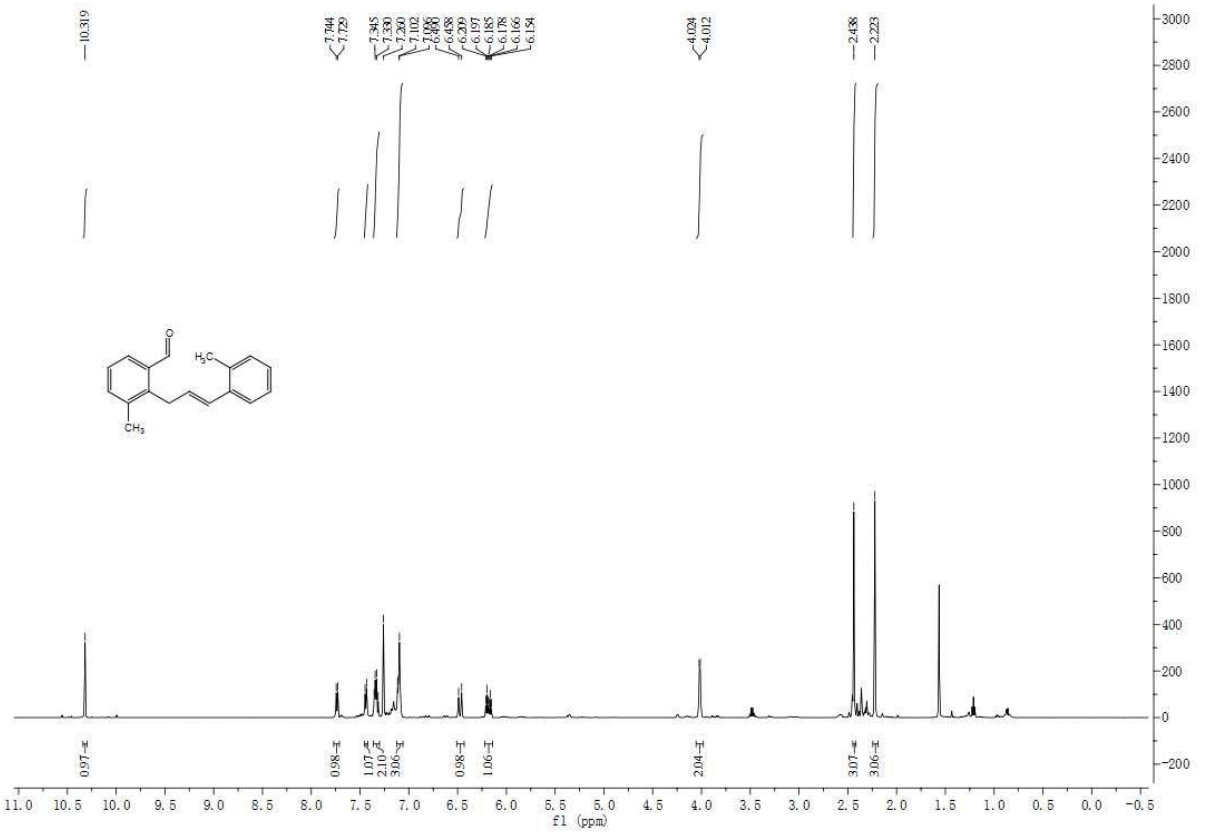
123.76
123.72
77.25
77.00
76.75
30.21
f1 (ppm)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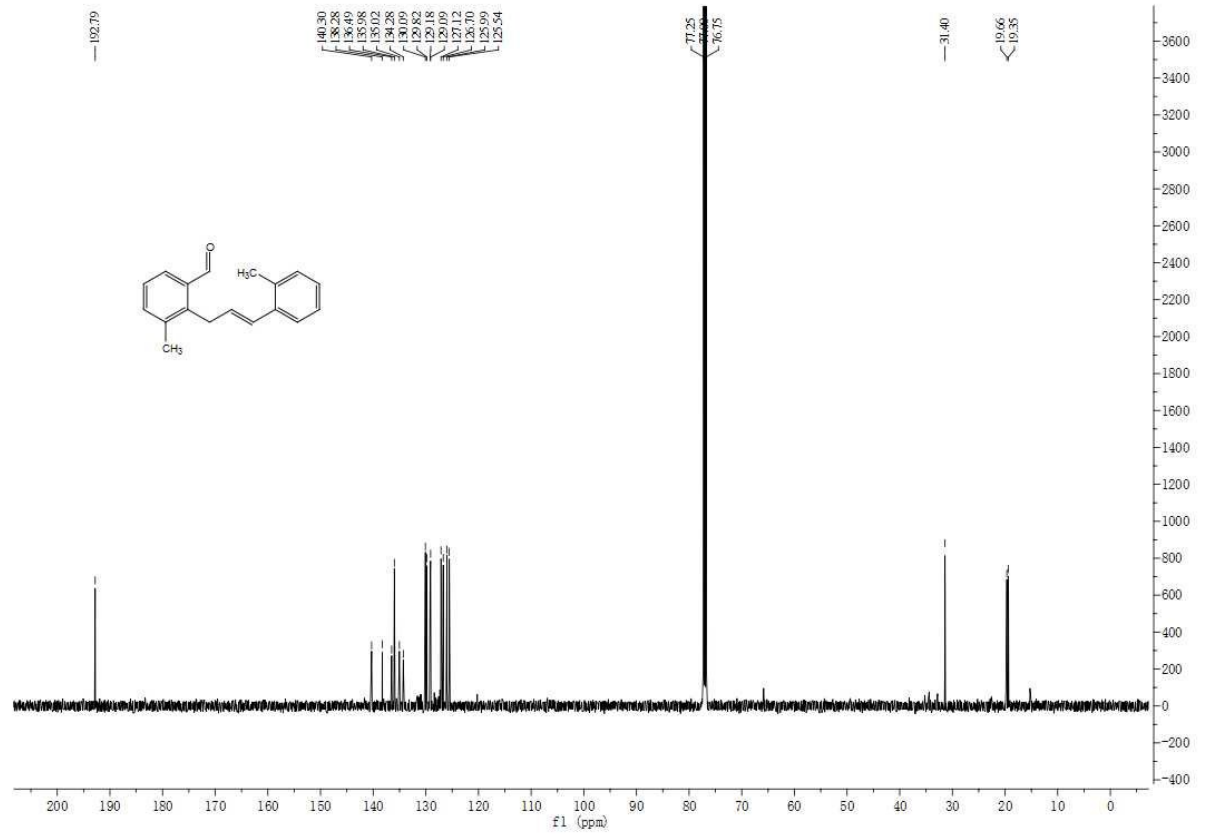
7.729
7.345
7.330
7.200
7.102
7.086
6.480
6.458
6.239
6.197
6.185
6.178
6.166
6.154
4.024
4.012
2.438
2.223
O
H3C
CH3
0.97
0.98
1.07
2.10
3.06
0.98
1.06
2.04
3.07
3.06
f1 (ppm)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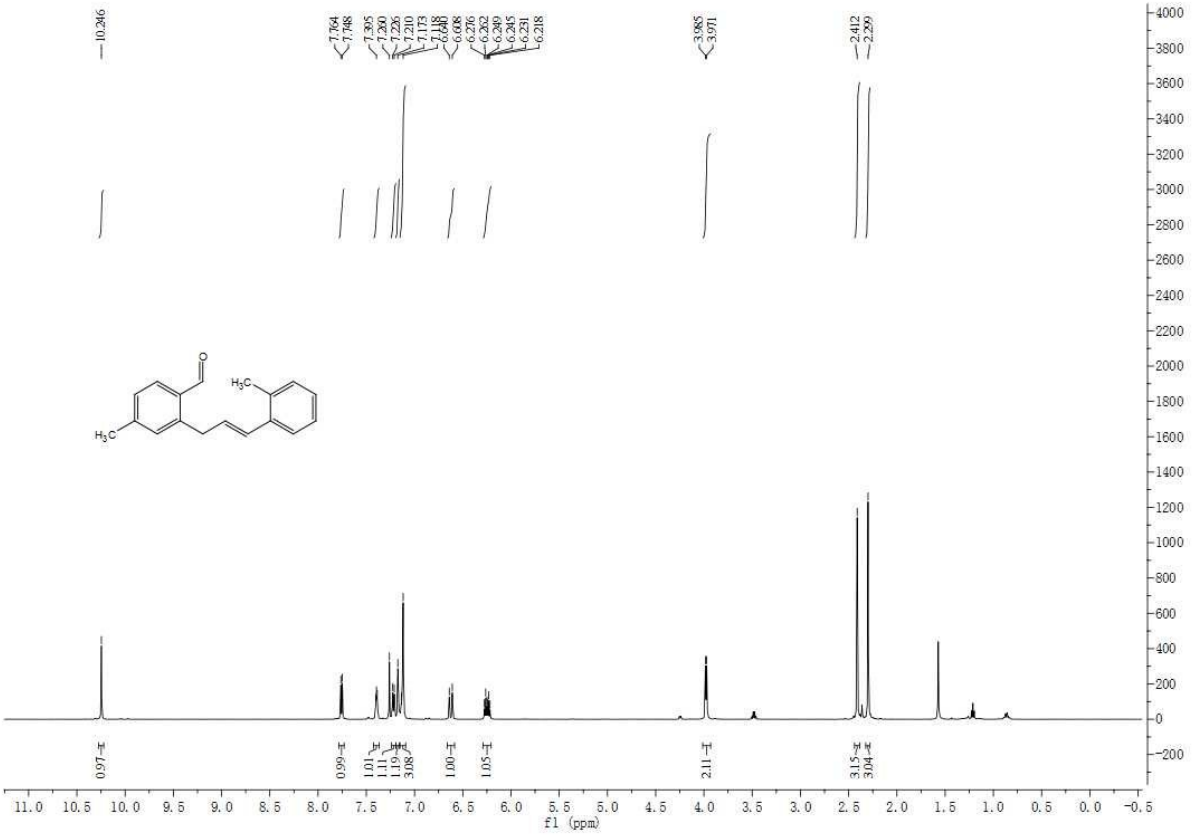
138.28
136.49
135.98
135.02
134.28
130.69
130.02
129.18
129.09
127.12
126.70
125.99
125.54
77.25
77.00
76.75
31.40
19.66
19.35
O
H3C
CH3
f1 (ppm)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7.390
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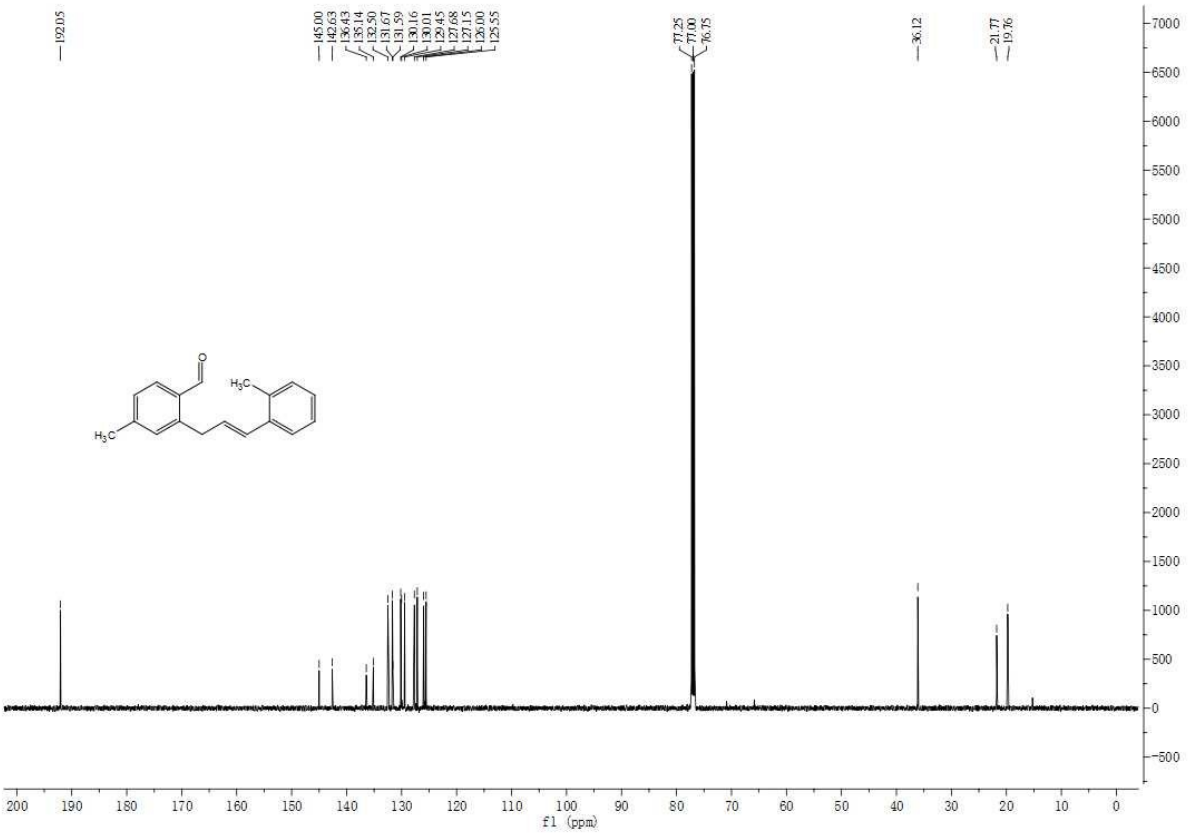
7.210
7.173
6.276
6.262
6.249
6.245
6.231
6.218
3.985
3.971
2.412
2.259
0.97
0.99
1.01
1.11
1.19
3.08
1.00
1.05
2.11
3.15
3.04
f1 (ppm)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131.67
131.59
130.16
130.01
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127.68
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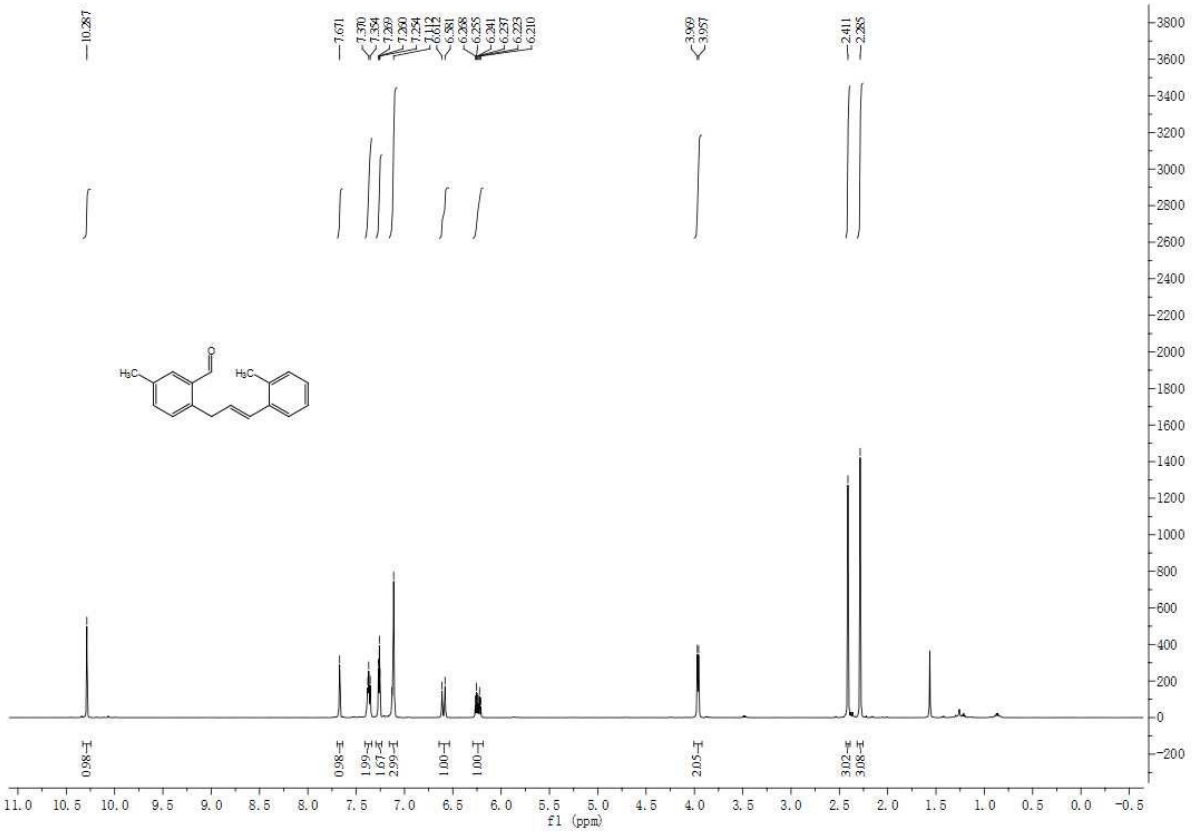
76.75
30.12
21.77
19.76
f1 (ppm)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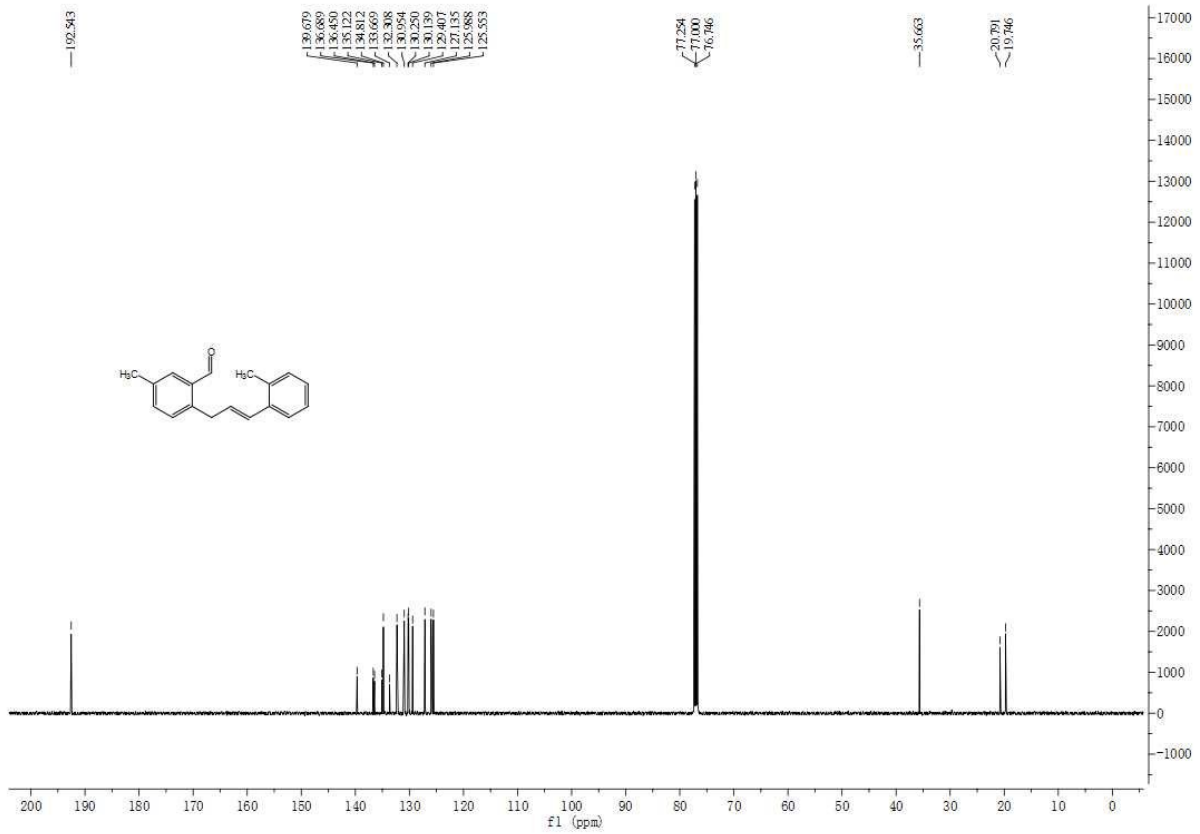
7.112
7.090
6.581
6.208
6.255
6.241
6.237
6.223
6.210
3.969
3.957
2.411
2.285
0.98
0.98
1.99
1.67
2.99
1.00
1.00
2.05
3.02
3.08
f1 (ppm)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135.122
134.812
133.669
132.308
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130.250
130.139
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125.553
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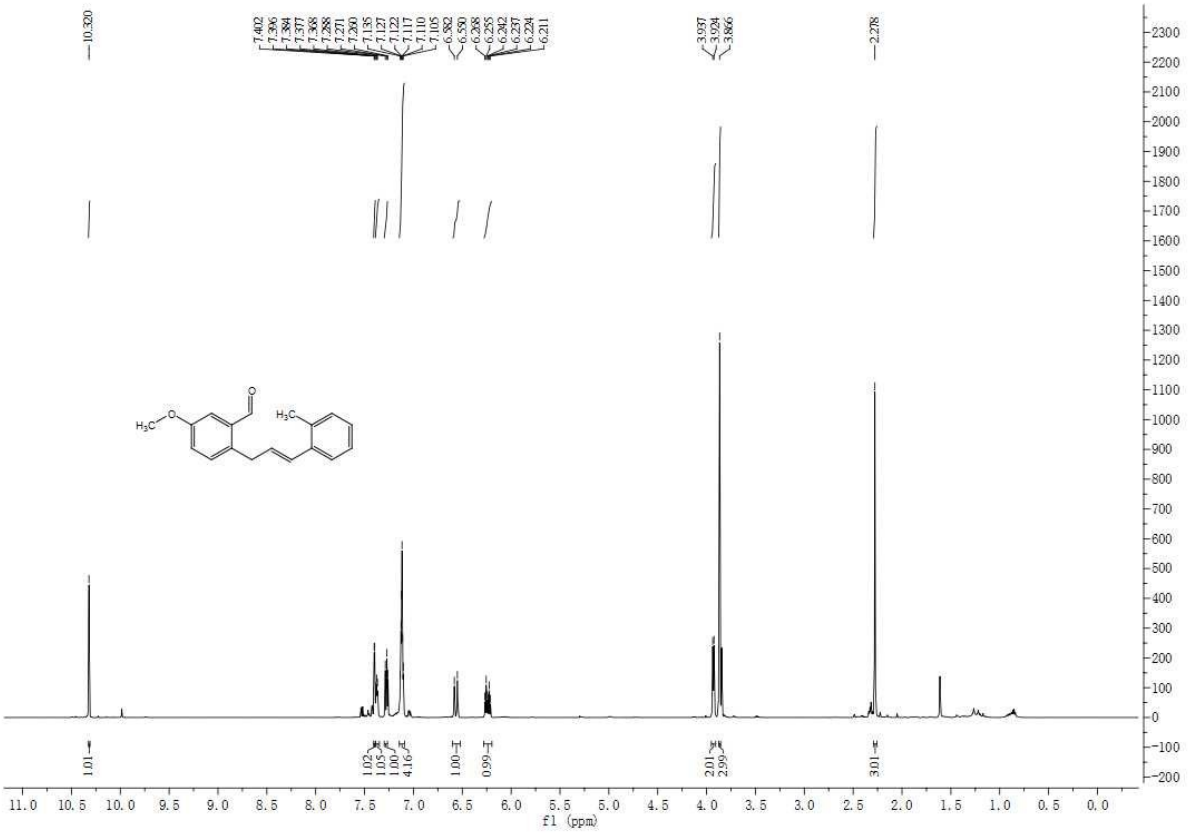
35.663
20.791
19.746
f1 (ppm)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7.127
7.122
7.117
7.110
7.105
6.882
6.590
6.288
6.255
6.242
6.237
6.224
6.211
3.937
3.924
3.866
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1.02
1.05
1.00
4.16
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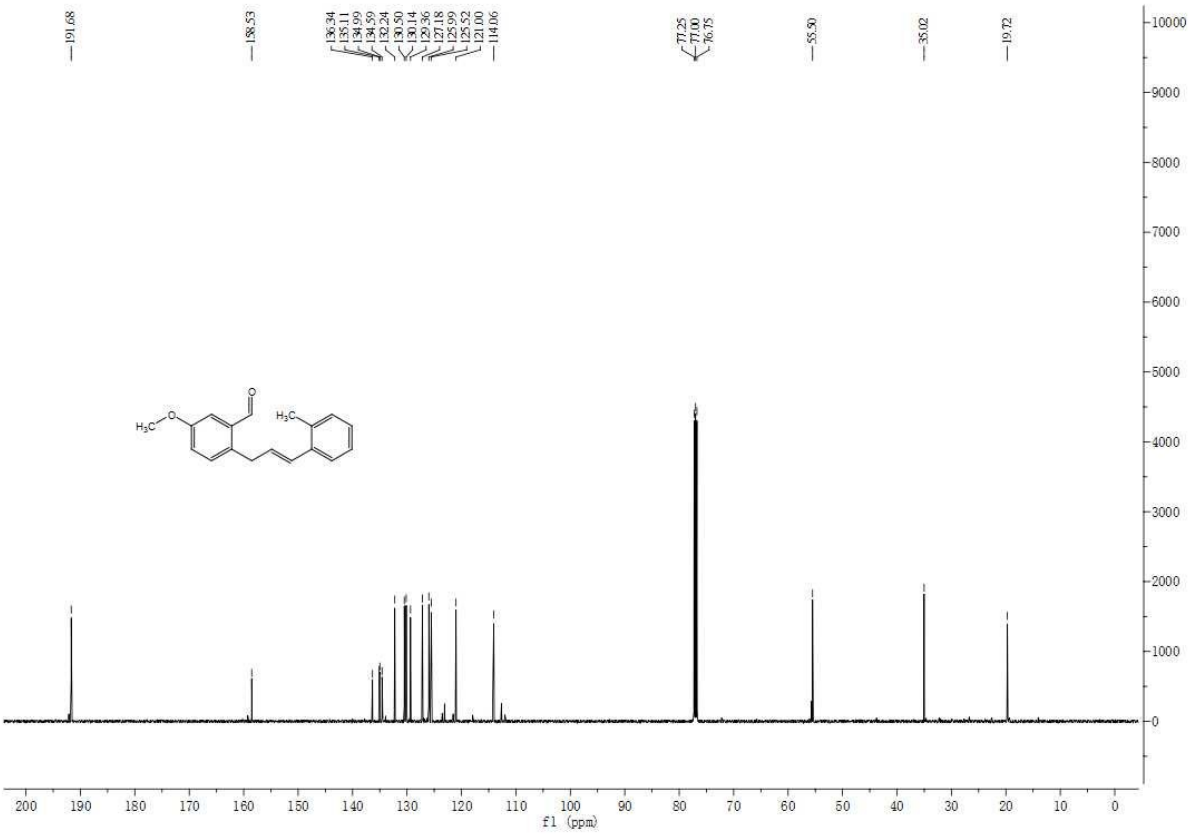
2.01
2.99
3.01
f1 (ppm)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135.11
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132.24
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130.14
129.36
127.18
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121.00
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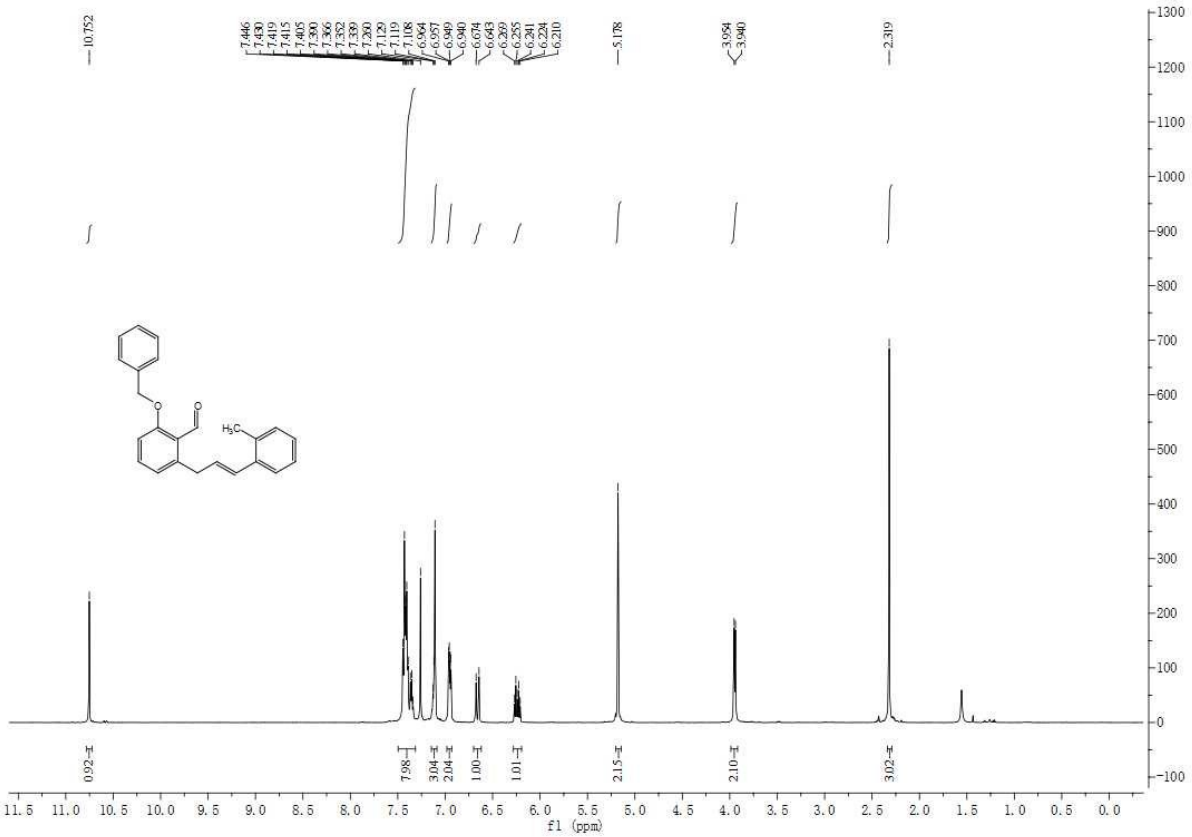
77.25
77.00
76.75
55.50
35.02
19.72
f1 (ppm)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7.108
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6.957
6.949
6.940
6.674
6.643
6.269
6.255
6.241
6.224
6.210
5.178
3.954
3.940
2.319
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1.01
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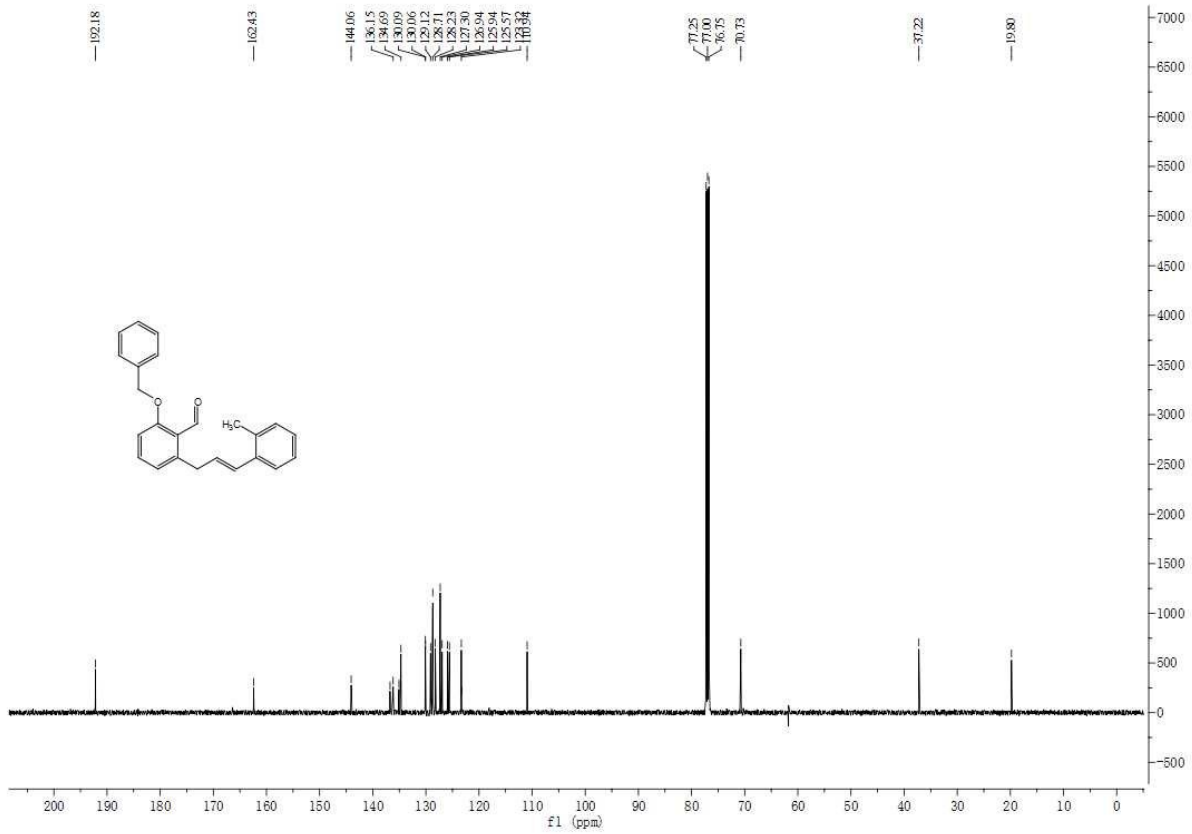
2.10
3.02
O
H3C
f1 (ppm)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129.12
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128.23
127.30
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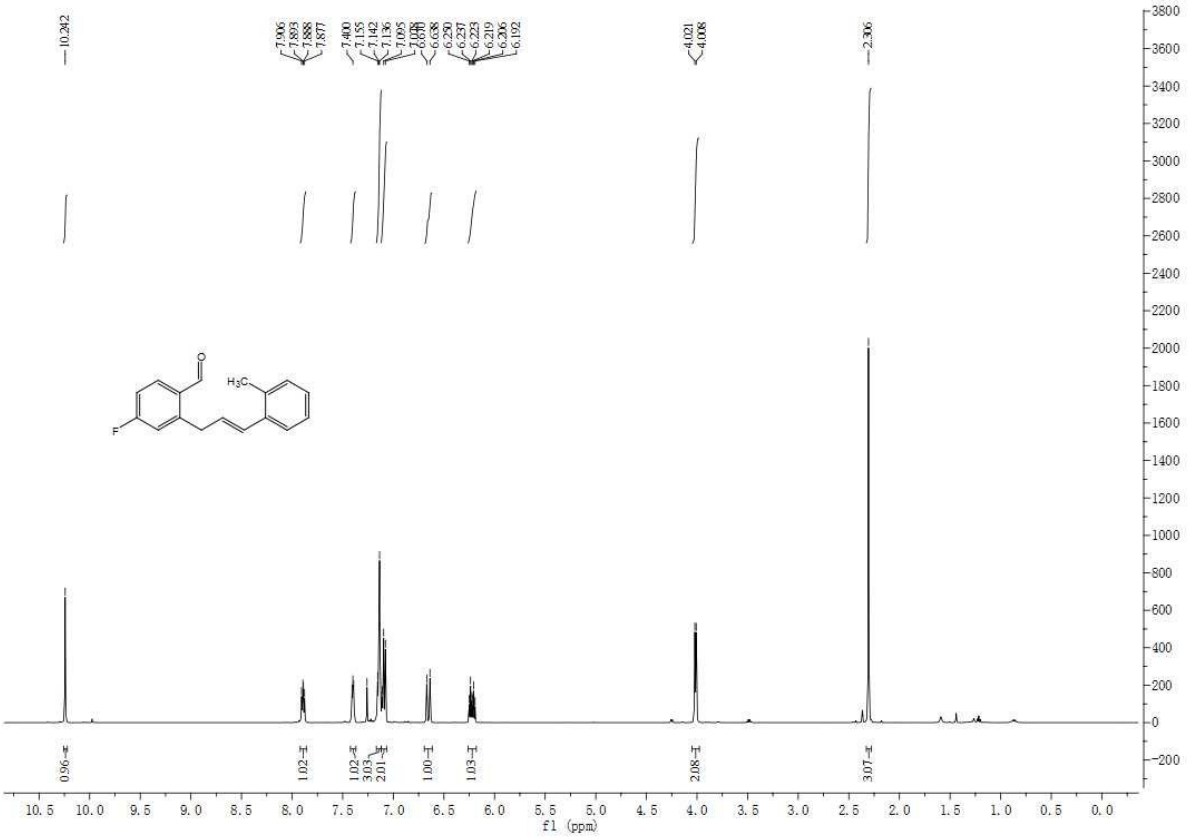
70.73
37.22
19.80
O
H3C
f1 (ppm)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7.095
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6.230
6.223
6.219
6.206
6.192
4.021
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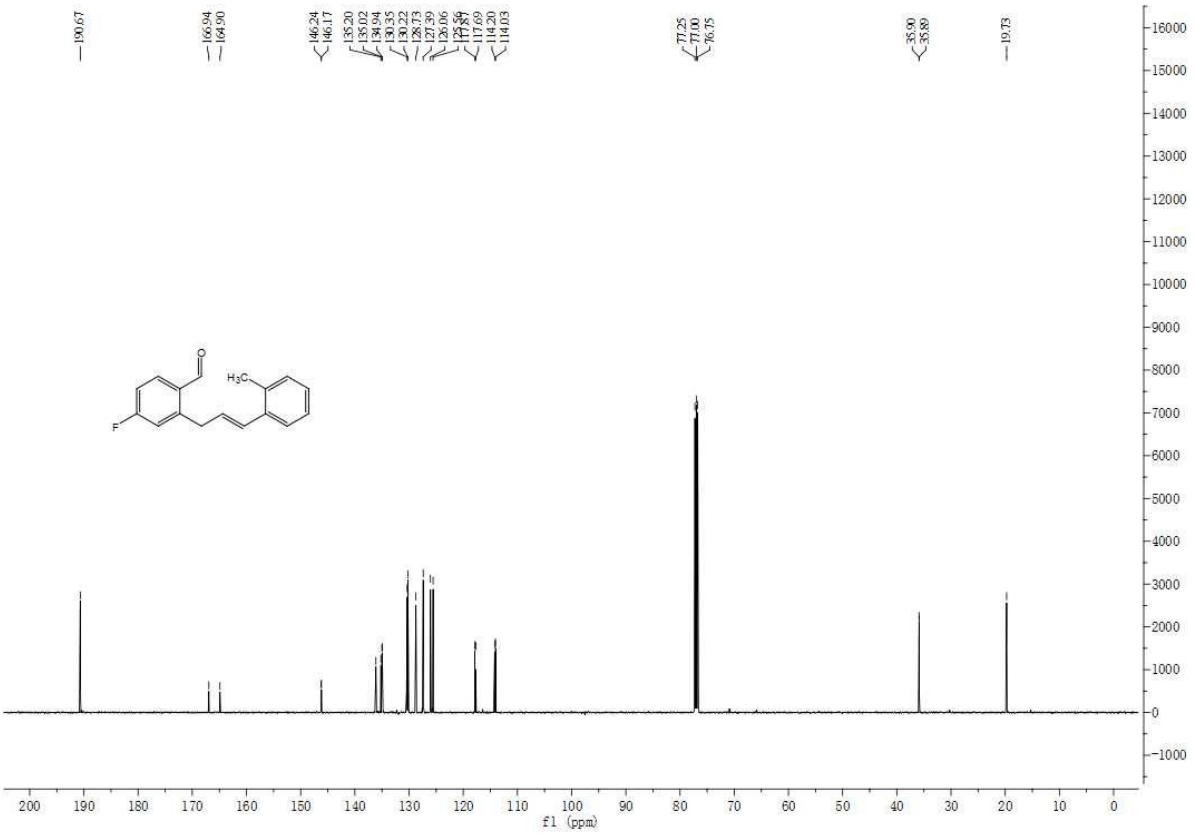
1.02
1.02
3.03
2.01
1.00
1.03
2.08
3.07
f1 (ppm)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134.94
130.35
130.22
128.73
127.39
126.06
125.56
125.51
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114.20
114.03
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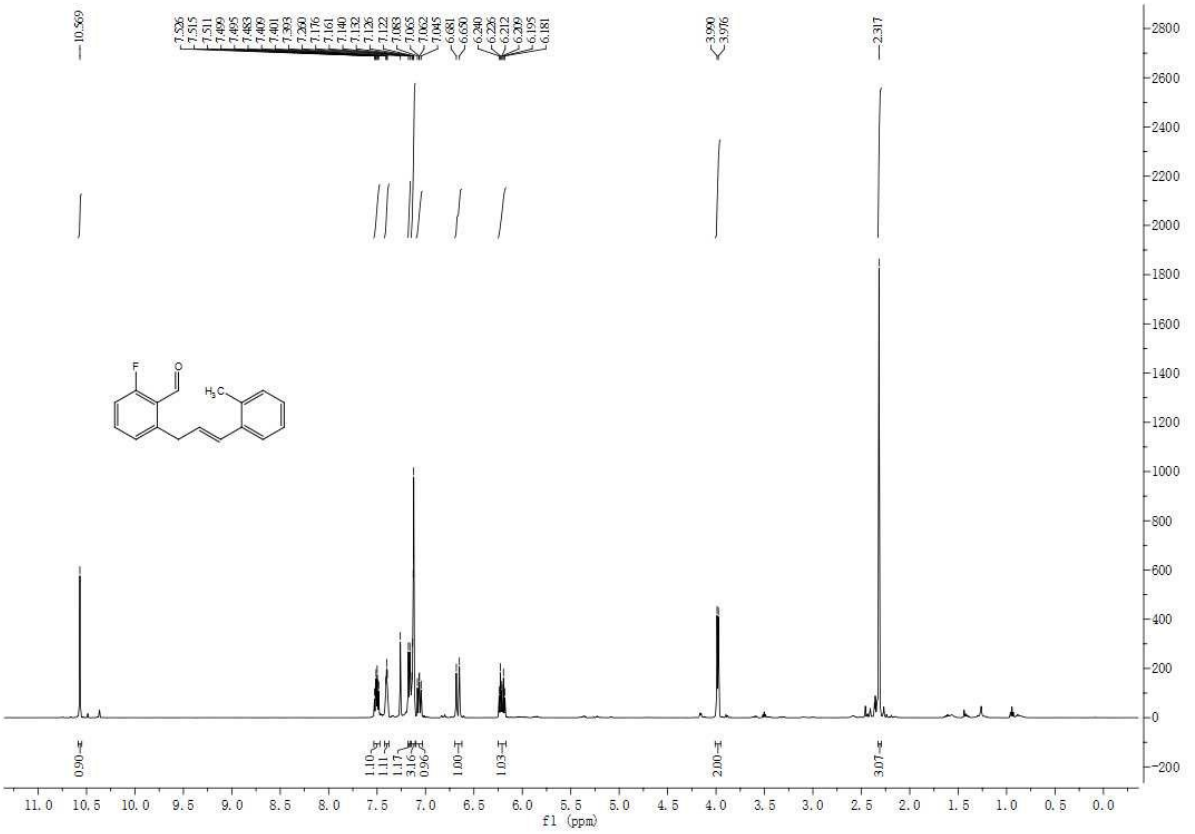
77.00
76.75
35.90
35.89
19.73
f1 (ppm)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7.200
7.176
7.161
7.140
7.132
7.126
7.122
7.083
7.065
7.062
7.045
6.681
6.659
6.240
6.226
6.212
6.210
6.195
6.181
3.990
3.976
2.317
F
O
H3C
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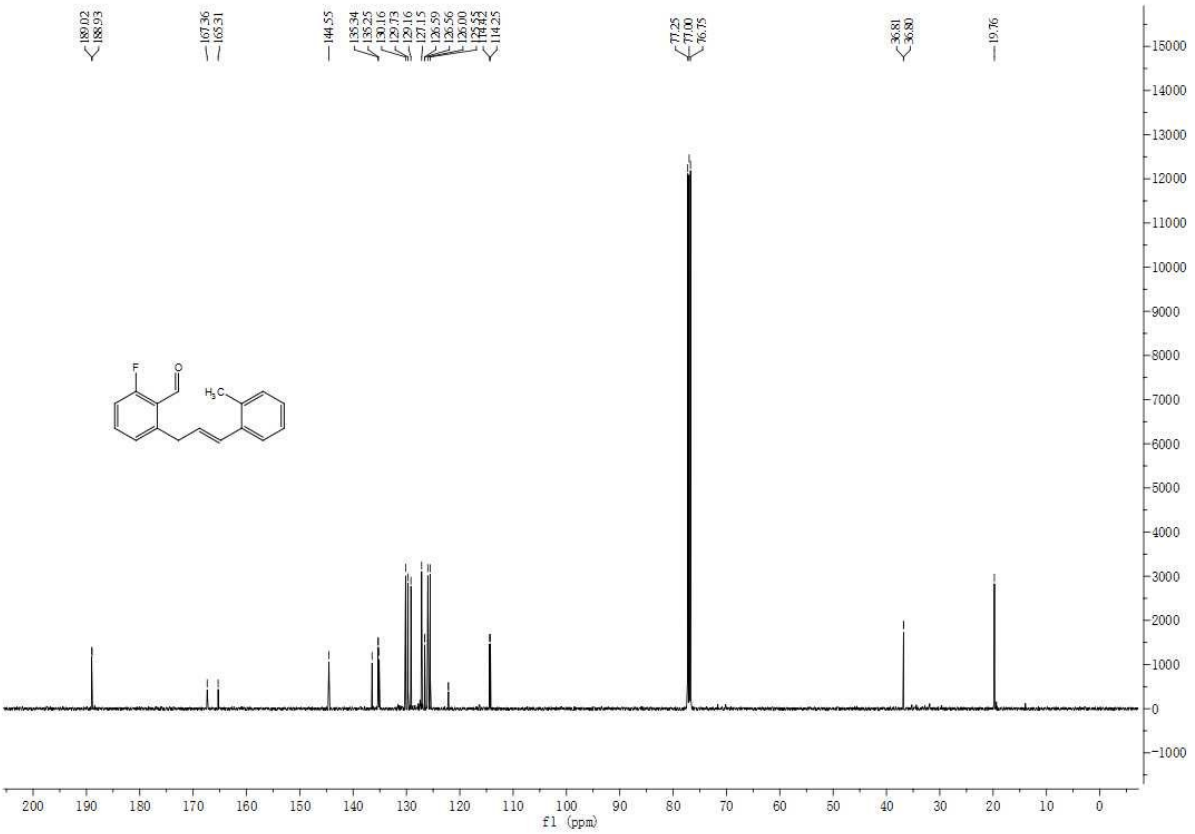
1.10
1.11
1.17
3.16
0.96
1.00
1.03
2.00
3.07
f1 (ppm)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127.15
126.56
126.50
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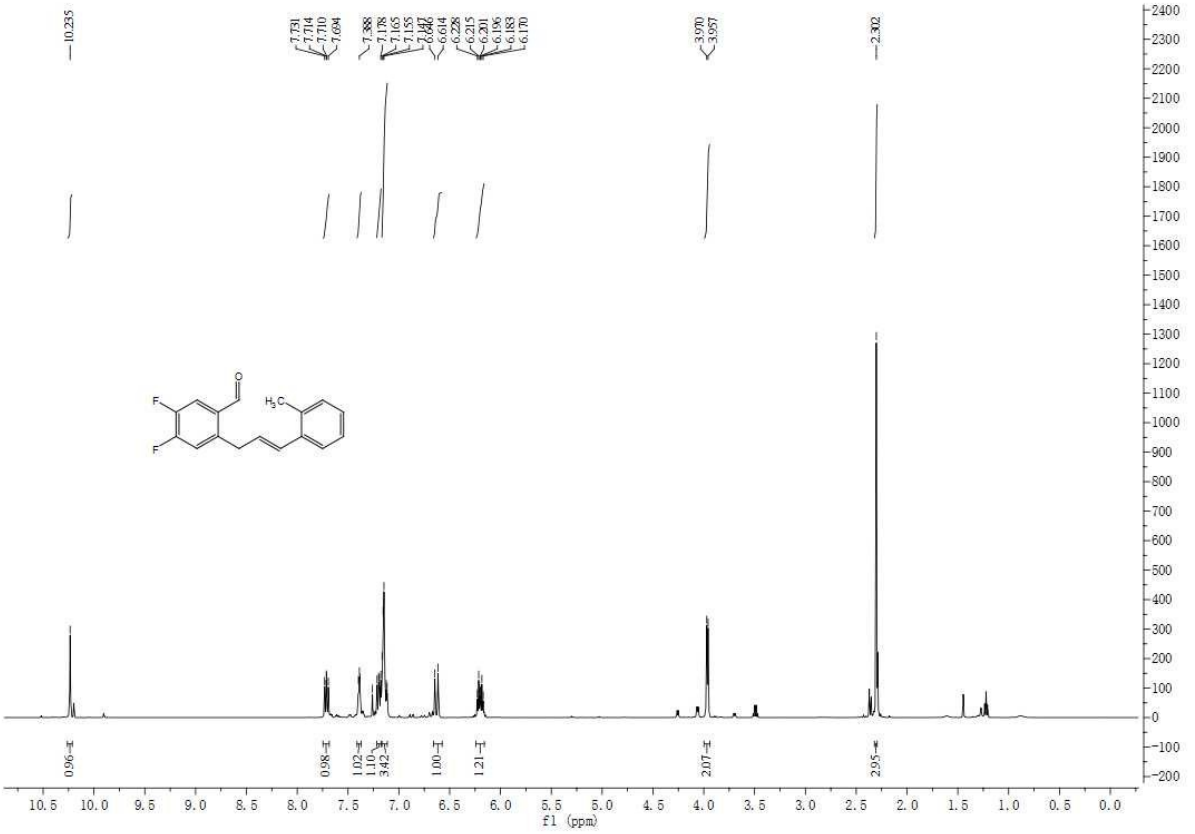
36.80
19.76
F
O
H3C
f1 (ppm)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6.614
6.228
6.215
6.201
6.196
6.183
6.170
3.970
3.957
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0.98
1.02
1.10
3.42
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1.21
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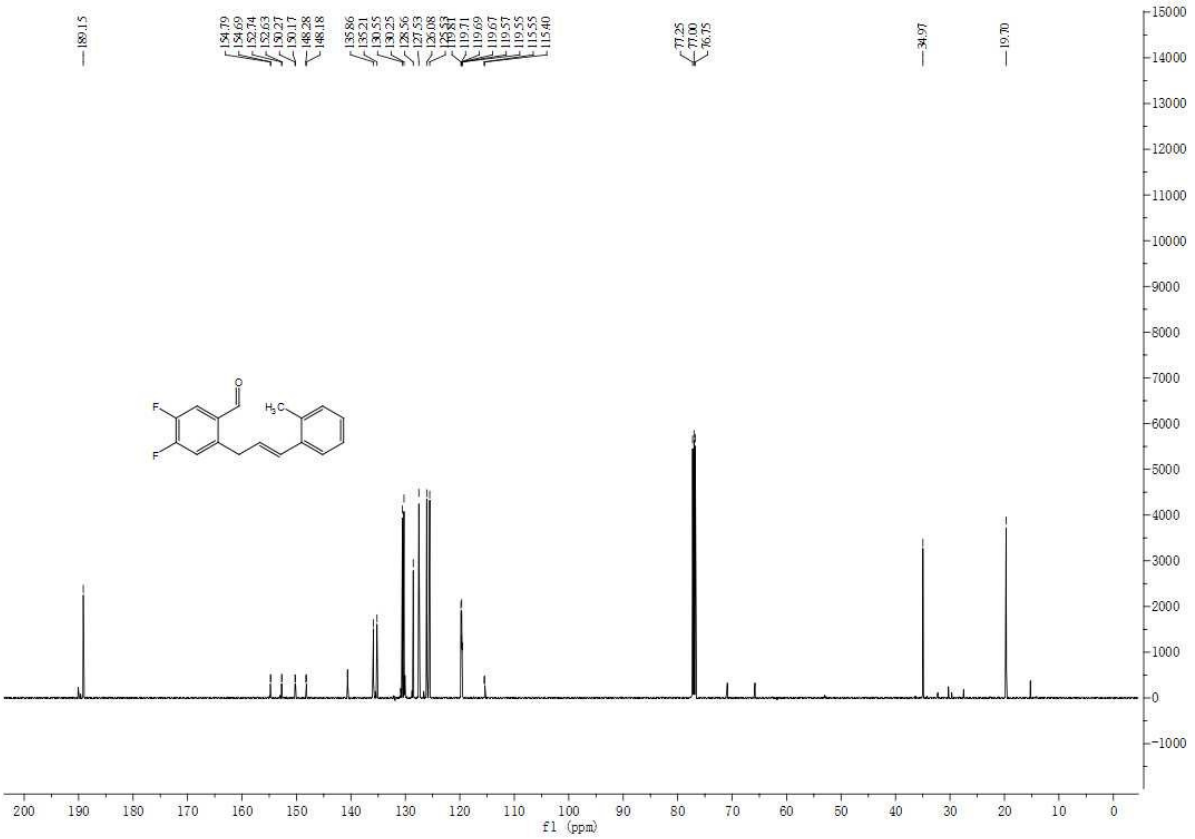
2.95
f1 (ppm)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128.56
127.53
126.08
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125.47
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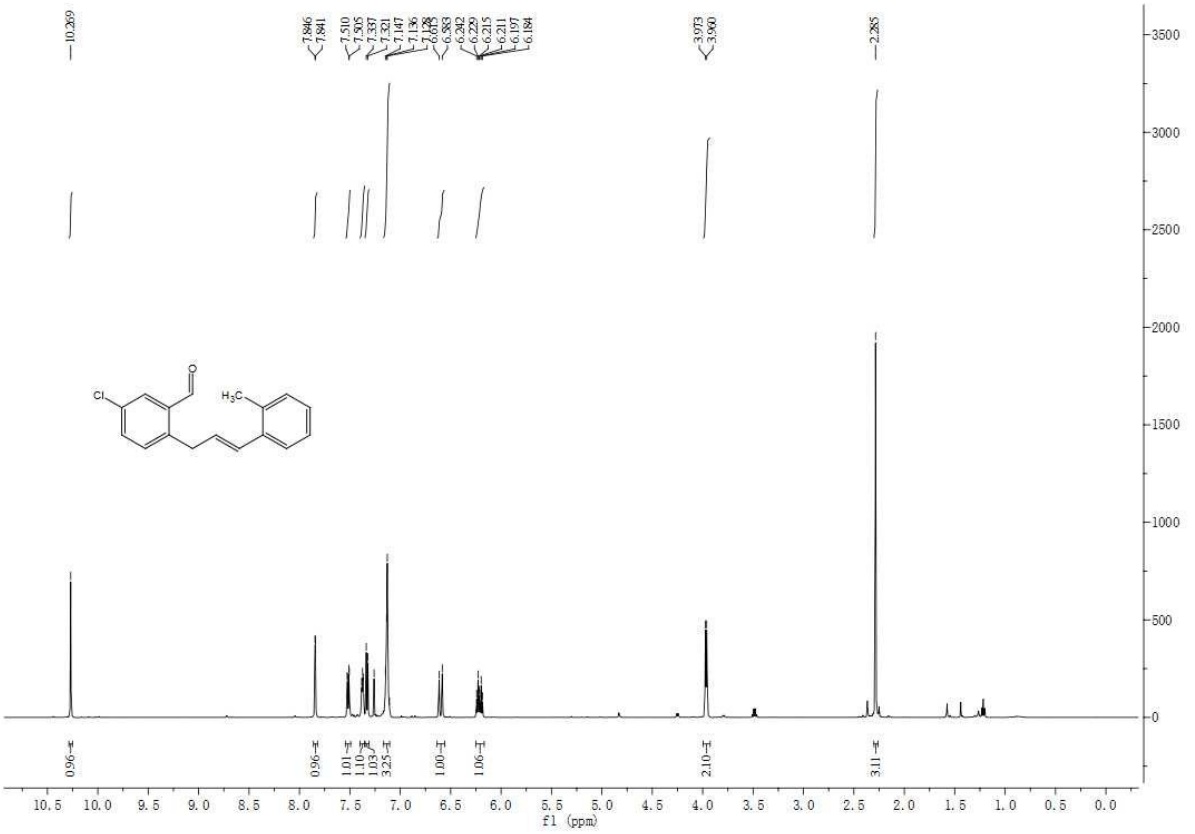
f1 (ppm)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6.242
6.229
6.215
6.211
6.197
6.184
3.973
3.960
2.285
Cl
O
H3C
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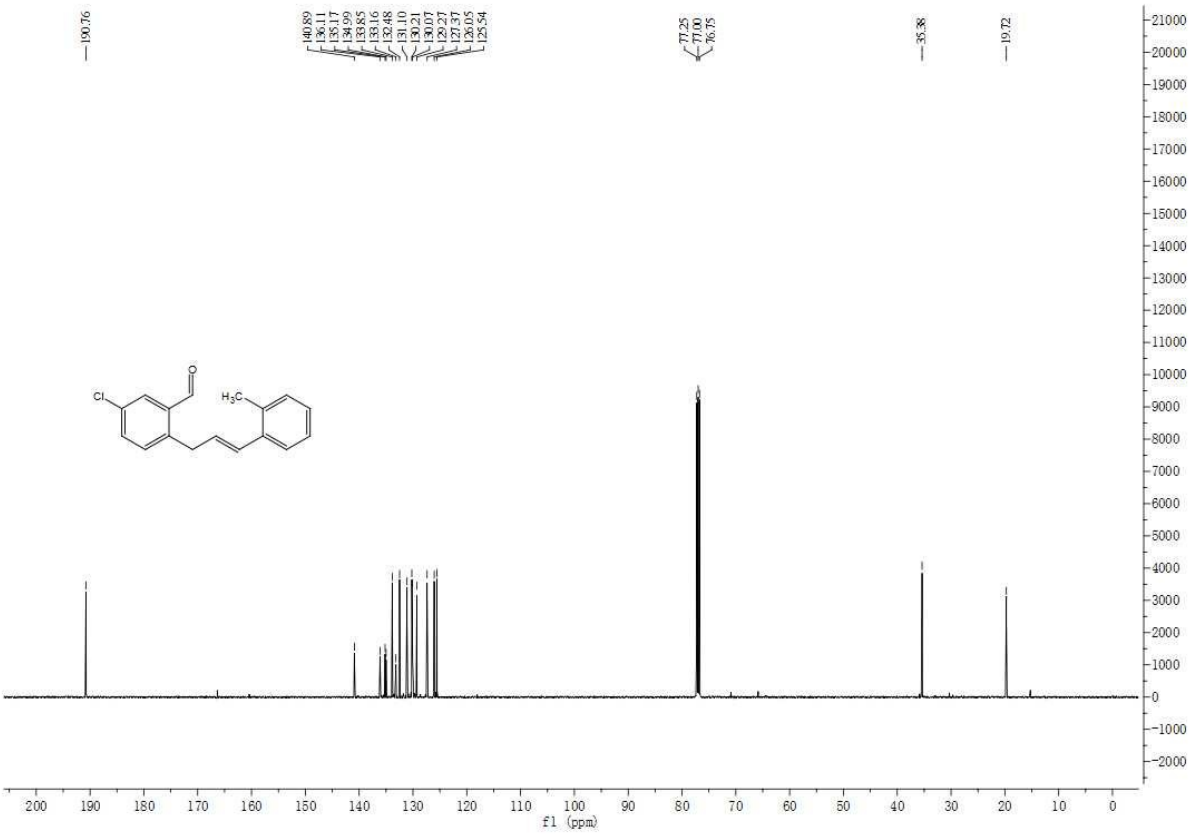
0.96
1.01
1.10
1.03
3.25
1.00
1.06
2.10
3.11
f1 (ppm)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130.07
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125.54
77.25
77.00
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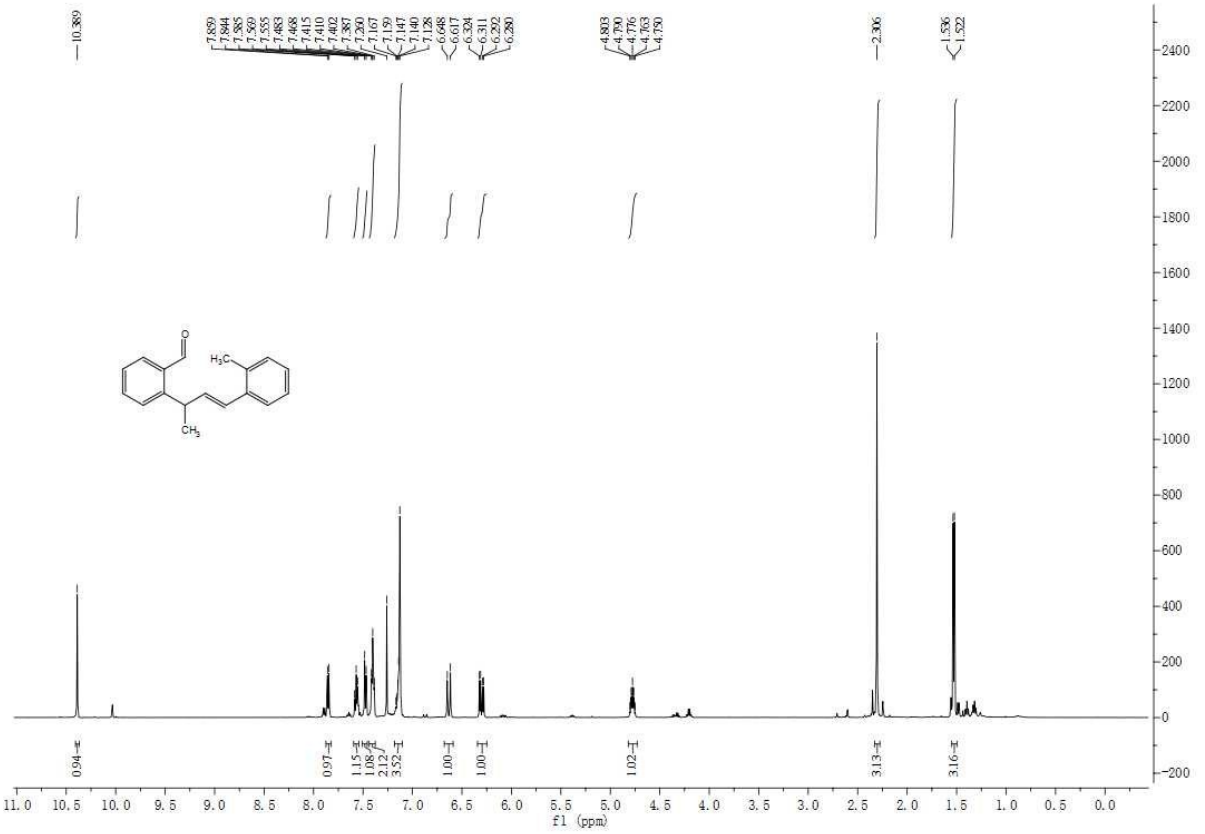
35.38
19.72
Cl
O
H3C
f1 (ppm)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7.167
7.159
7.147
7.140
7.128
6.648
6.617
6.324
6.311
6.292
6.280
4.803
4.790
4.776
4.763
4.750
2.306
1.536
1.522
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1.08
2.12
3.52
1.00
1.00
1.02
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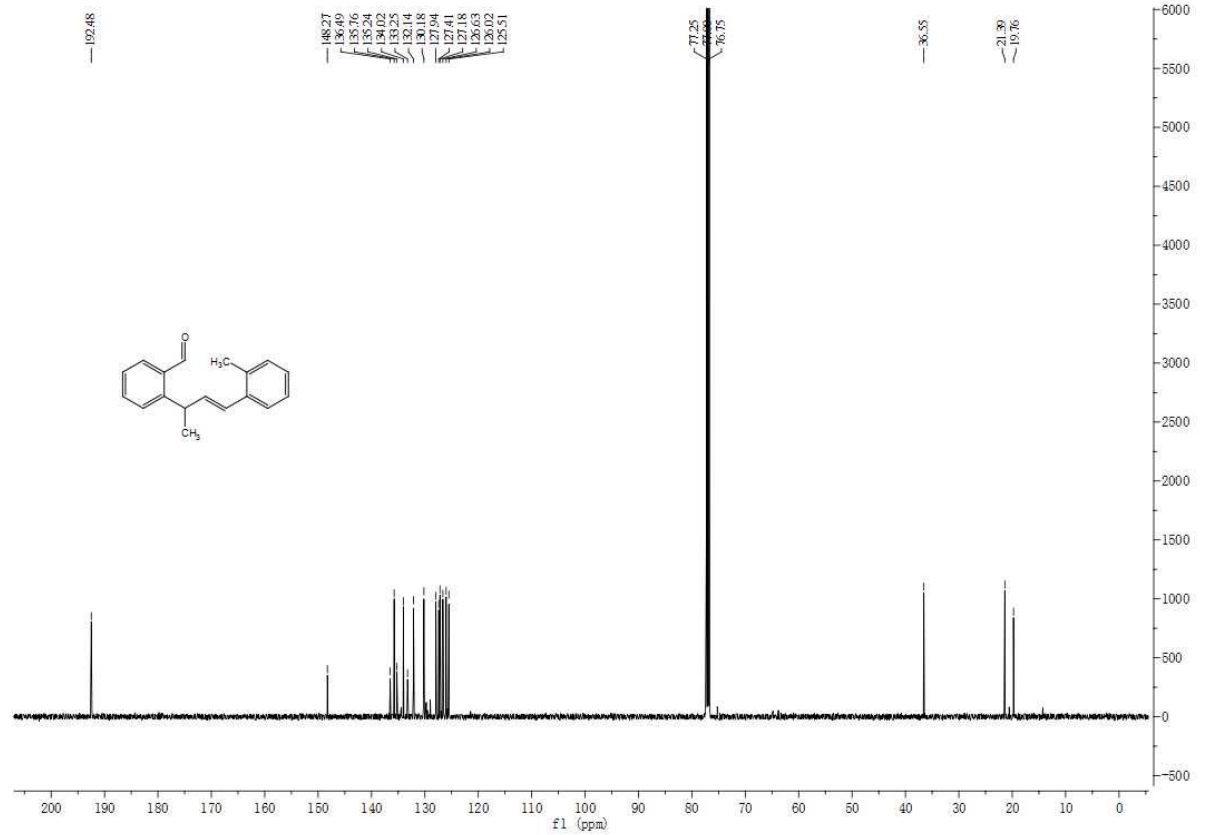
3.16
f1 (ppm)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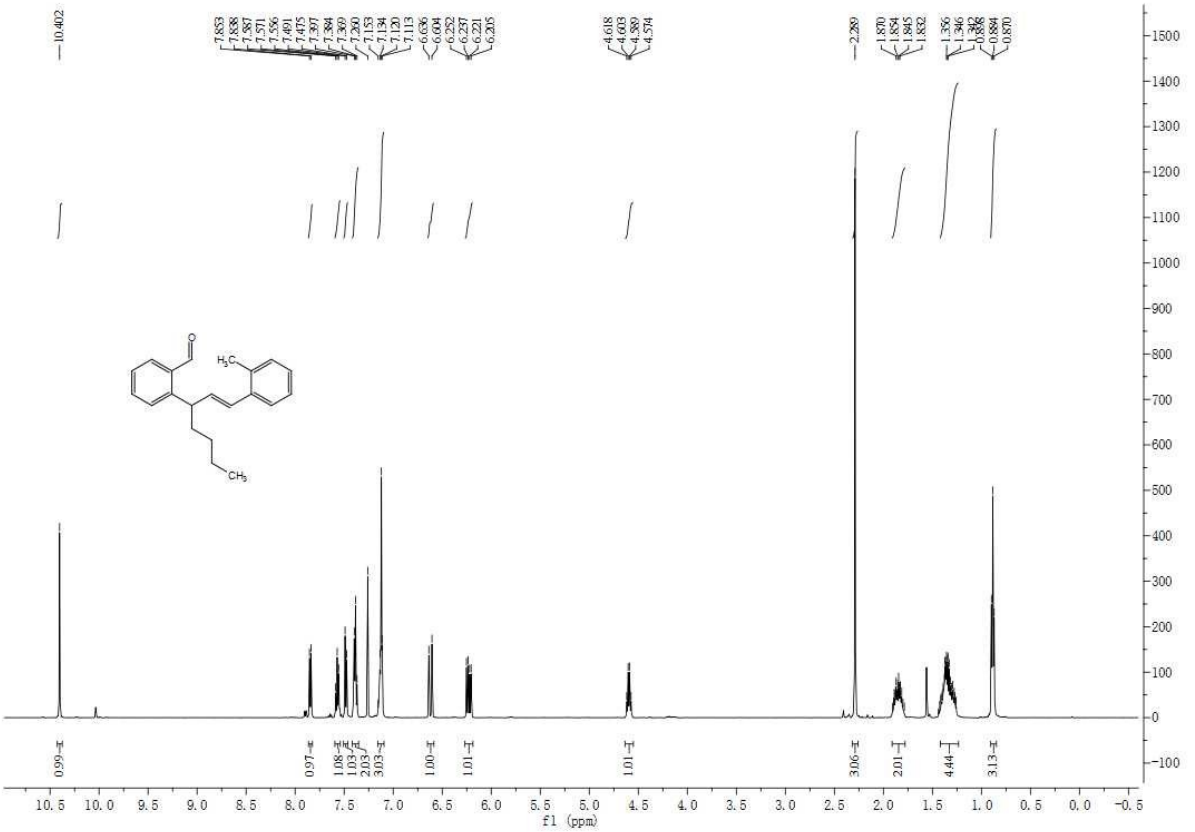
127.41
127.18
126.63
126.02
125.51
77.25
77.00
76.75
36.55
21.39
19.76
f1 (ppm)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7.153
7.134
7.120
7.113
6.636
6.604
6.252
6.237
6.221
6.205
4.618
4.603
4.589
4.574
2.289
1.870
1.854
1.845
1.832
1.366
1.346
1.328
1.308
0.884
0.870
0.99
0.97
1.08
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3.03
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1.01
1.01
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2.01
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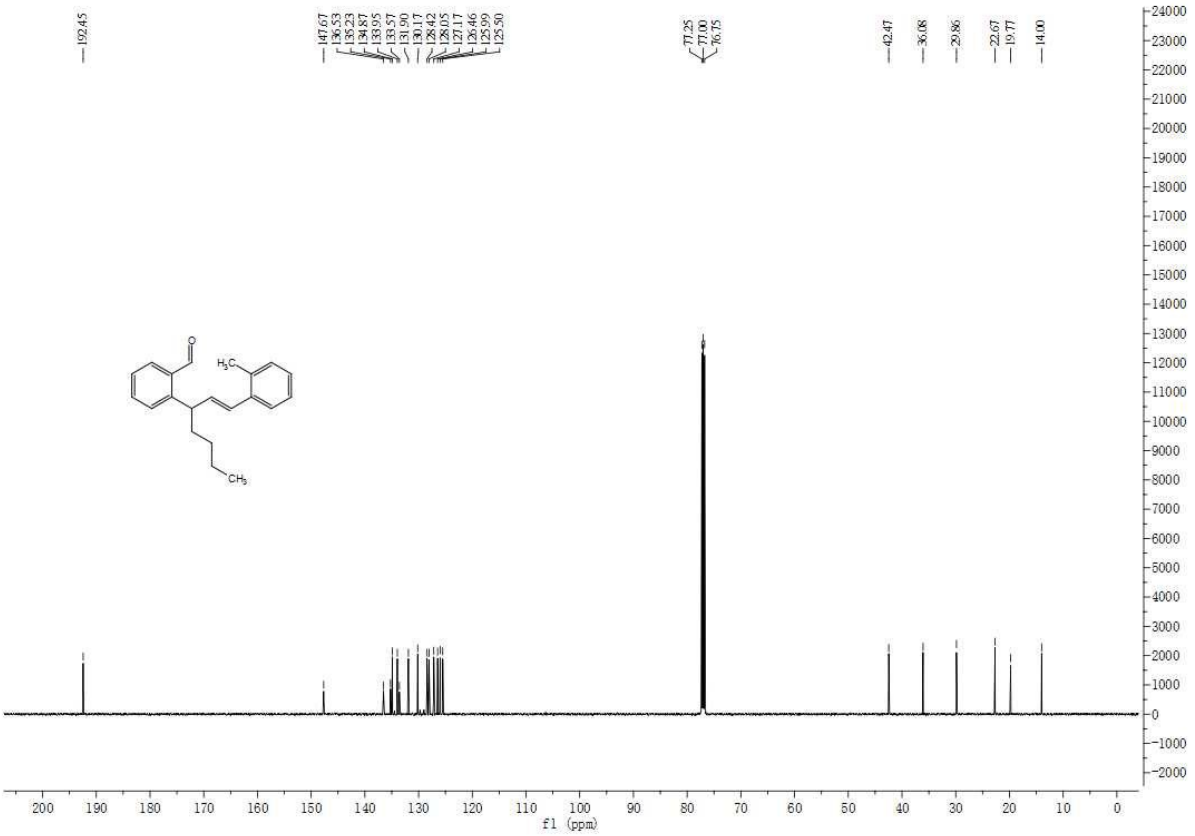
3.13
f1 (ppm)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127.17
126.46
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125.50
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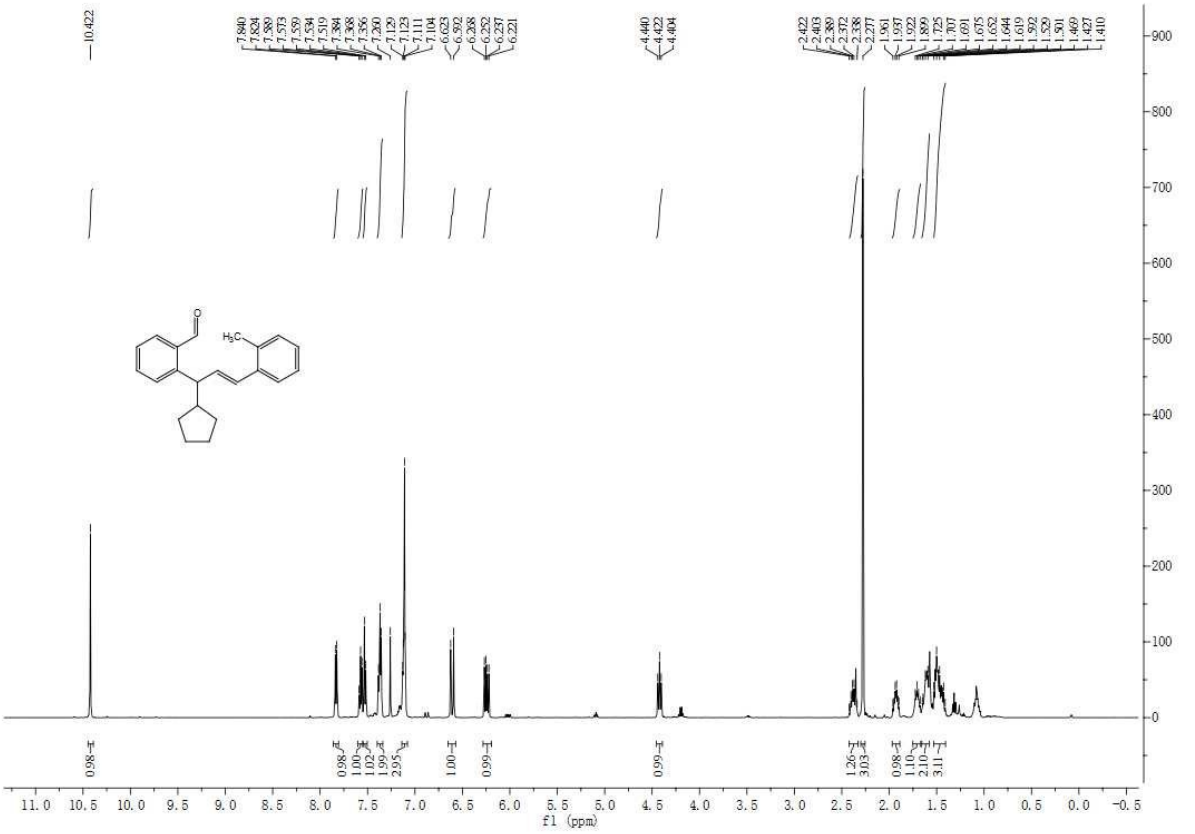
76.75
42.47
36.08
29.86
22.67
19.77
14.00
f1 (ppm)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7.129
7.123
7.111
7.104
6.623
6.992
6.388
6.252
6.237
6.221
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4.422
4.404
2.422
2.403
2.380
2.372
2.338
2.277
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1.937
1.922
1.899
1.725
1.707
1.691
1.675
1.652
1.644
1.619
1.592
1.529
1.501
1.469
1.427
1.410
O
H3C
0.98
0.98
1.00
1.02
1.99
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0.99
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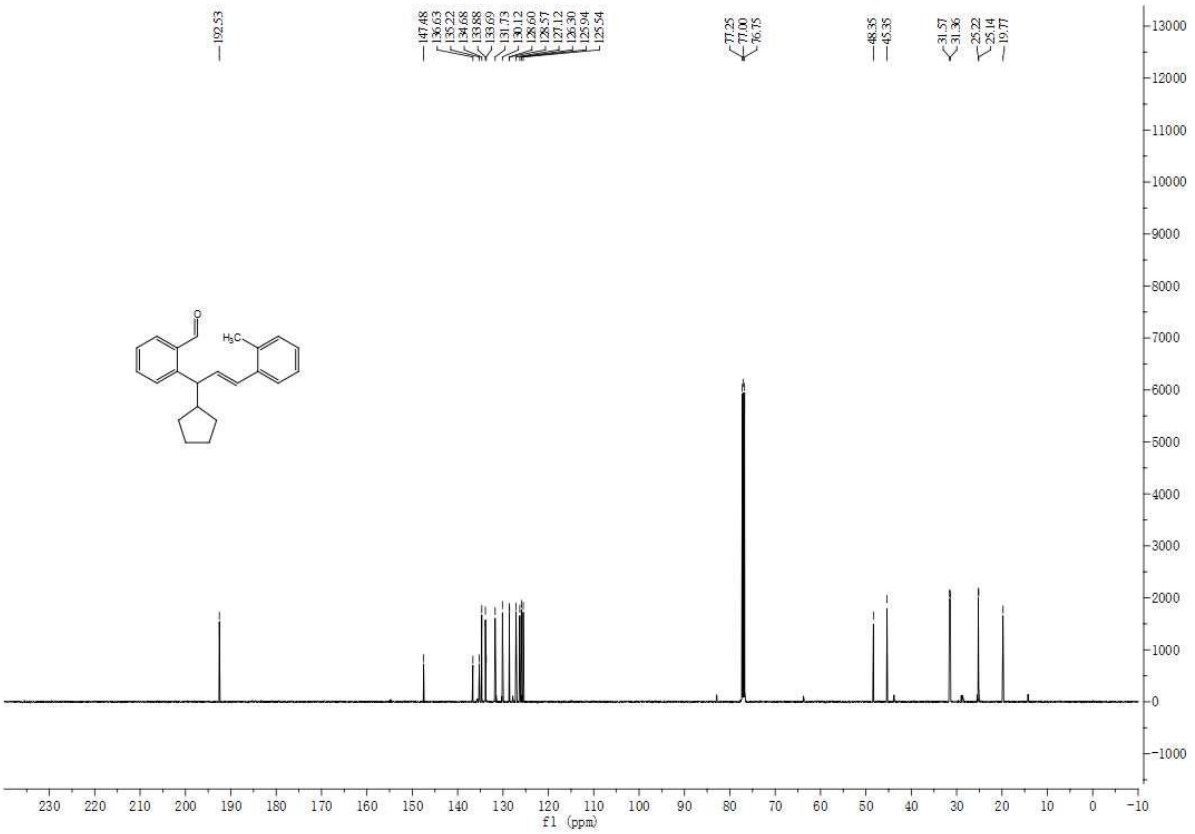
0.98
1.10
2.10
3.11
f1 (ppm)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127.12
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125.94
125.54
77.25
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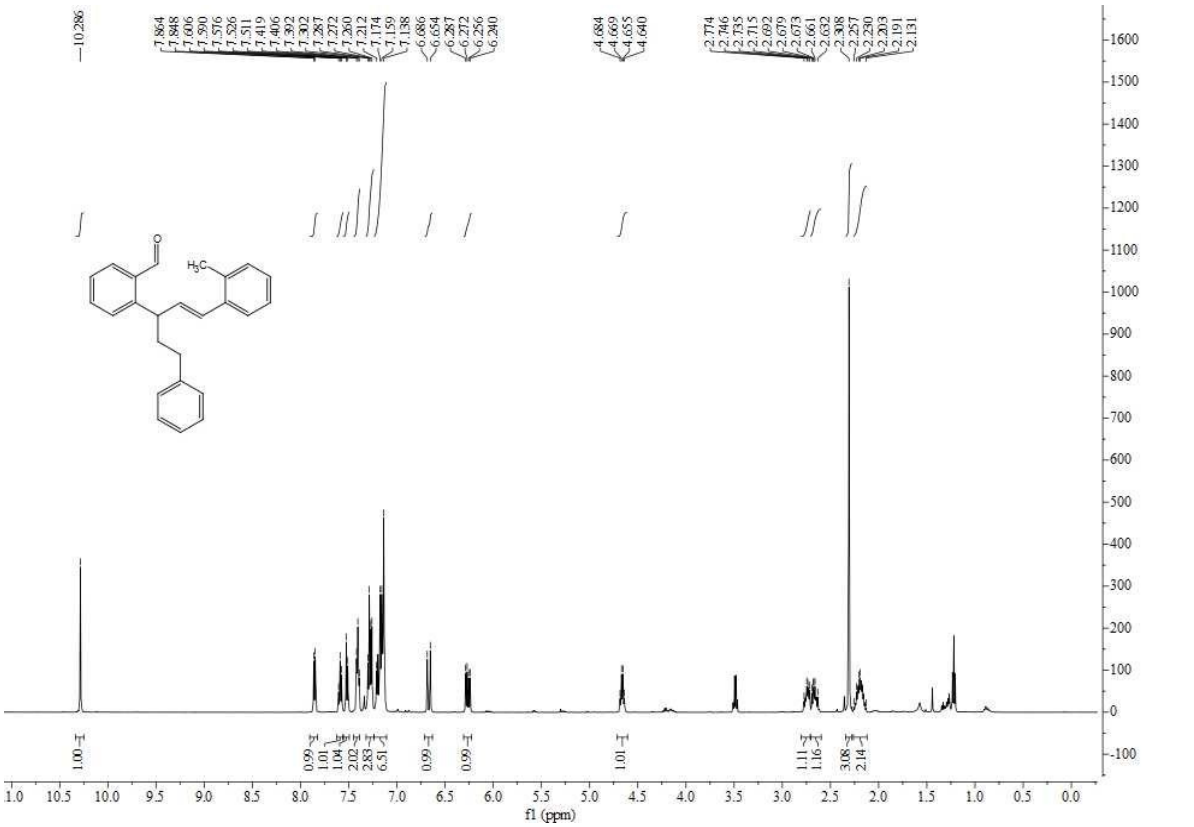
45.35
31.57
31.36
25.22
25.14
19.77
O
H3C
f1 (ppm)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7.212
7.174
7.159
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6.287
6.272
6.256
6.240
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4.655
4.640
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2.735
2.715
2.692
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2.673
2.661
2.632
2.308
2.257
2.230
2.203
2.191
2.131
O
H3C
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1.01
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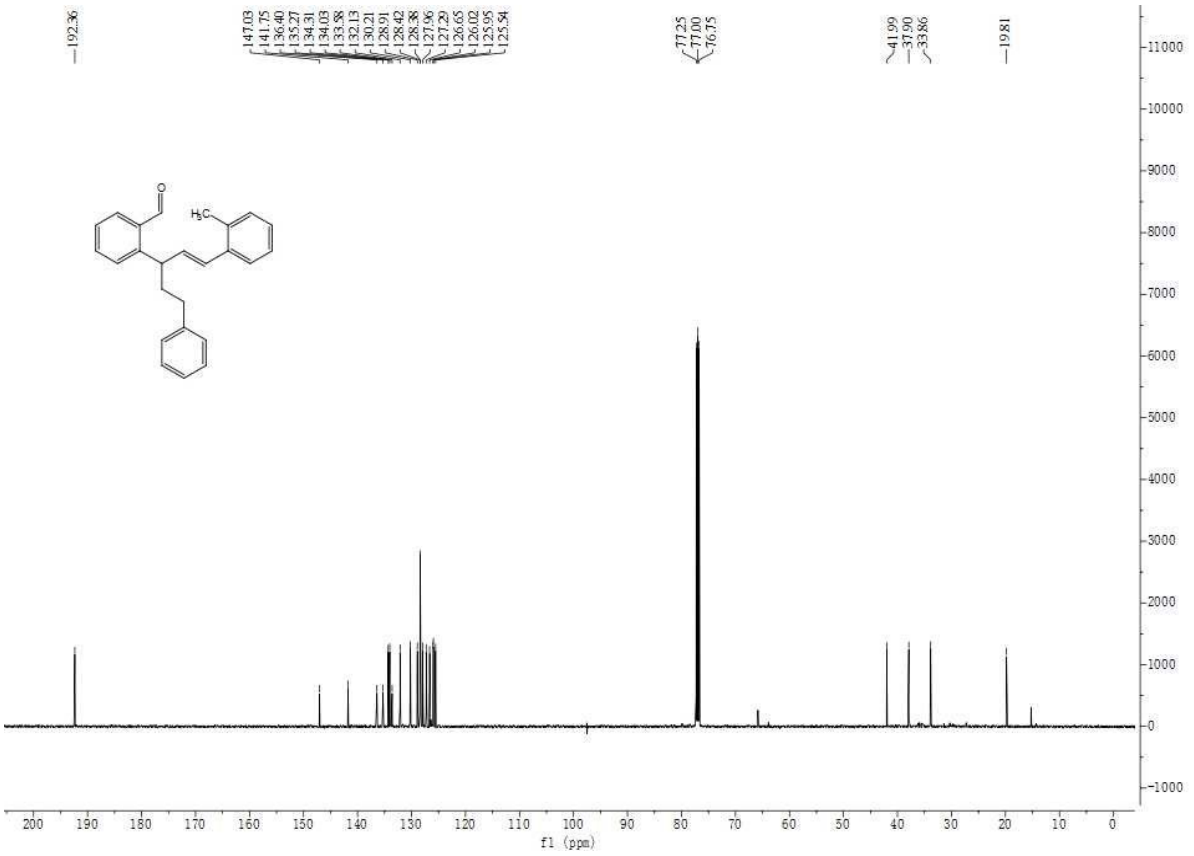
1.16
3.08
2.14
f1 (ppm)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128.42
128.38
127.96
127.29
126.65
126.02
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125.54
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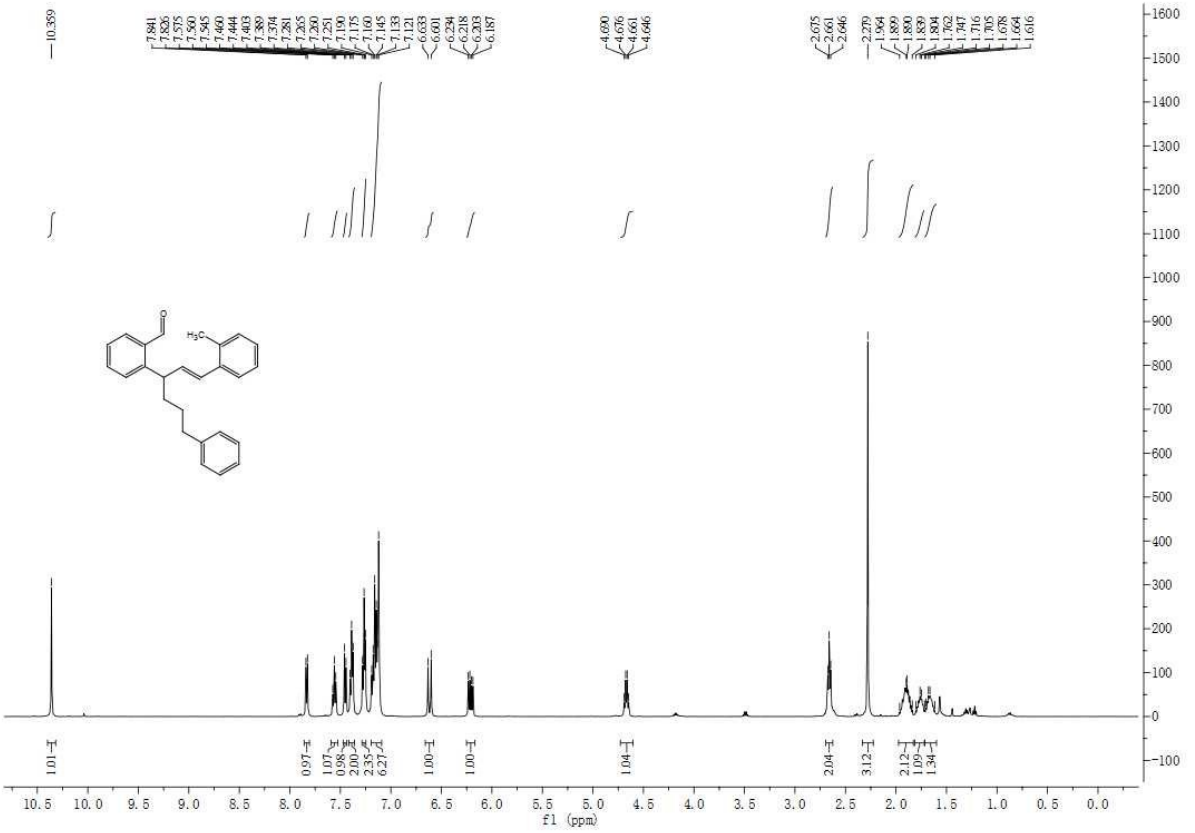
77.00
76.75
41.99
37.90
33.86
19.81
O
H3C
f1 (ppm)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7.265
7.250
7.251
7.190
7.175
7.160
7.145
7.133
7.121
6.633
6.601
6.234
6.218
6.203
6.187
4.690
4.676
4.661
4.646
2.675
2.661
2.646
2.279
1.964
1.899
1.880
1.839
1.804
1.762
1.747
1.716
1.705
1.678
1.664
1.616
O
H3C
1.01
0.97
1.07
0.98
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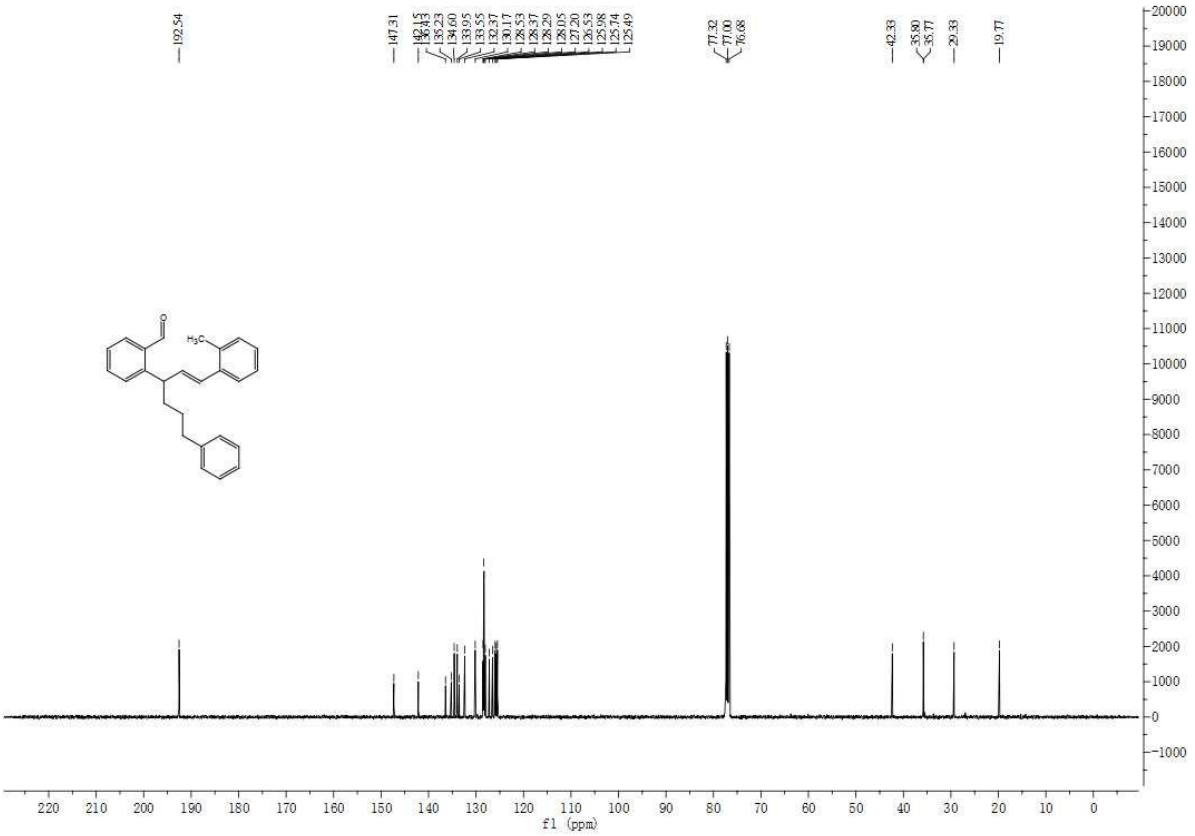
1.00
1.00
1.04
2.04
3.12
2.12
1.09
1.34
f1 (ppm)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130.17
128.53
128.37
128.29
126.95
126.70
125.53
125.08
125.74
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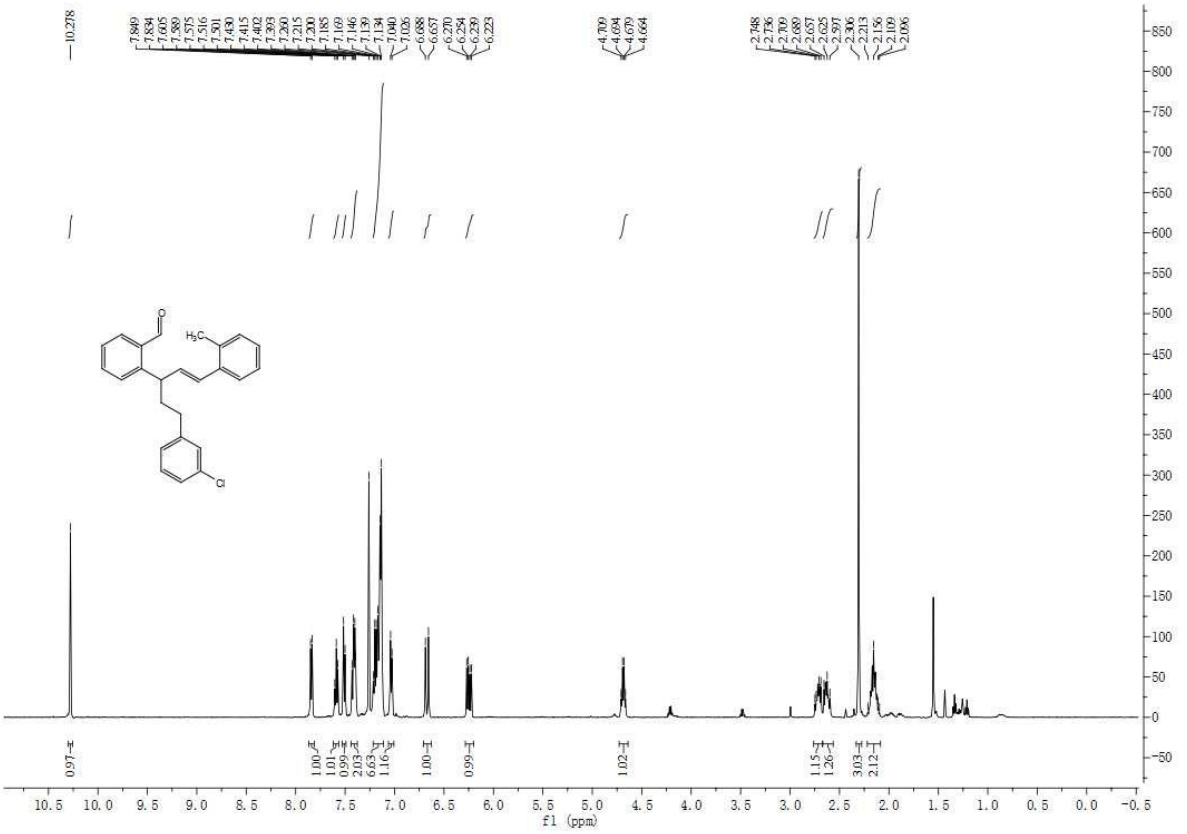
29.33
19.77
O
H3C
f1 (ppm)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7.215
7.200
7.185
7.169
7.146
7.139
7.134
7.040
7.026
6.688
6.657
6.270
6.254
6.239
6.223
4.709
4.694
4.679
4.664
2.748
2.736
2.709
2.689
2.657
2.625
2.997
2.306
2.213
2.156
2.109
2.066
O
H3C
Cl
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1.00
1.01
0.99
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1.00
0.99
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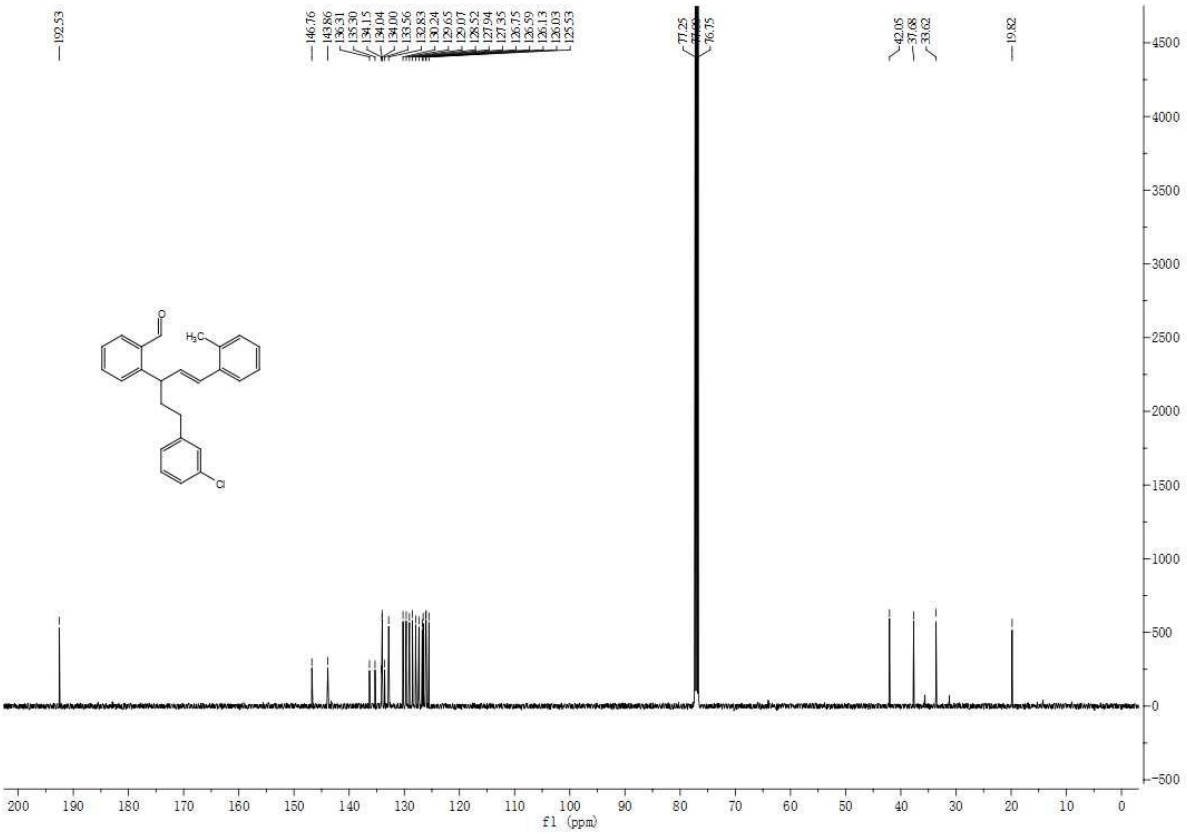
3.03
2.12
f1 (ppm)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129.65
129.07
128.52
127.94
127.35
126.75
126.59
126.13
126.03
125.53
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77.00
76.75
42.05
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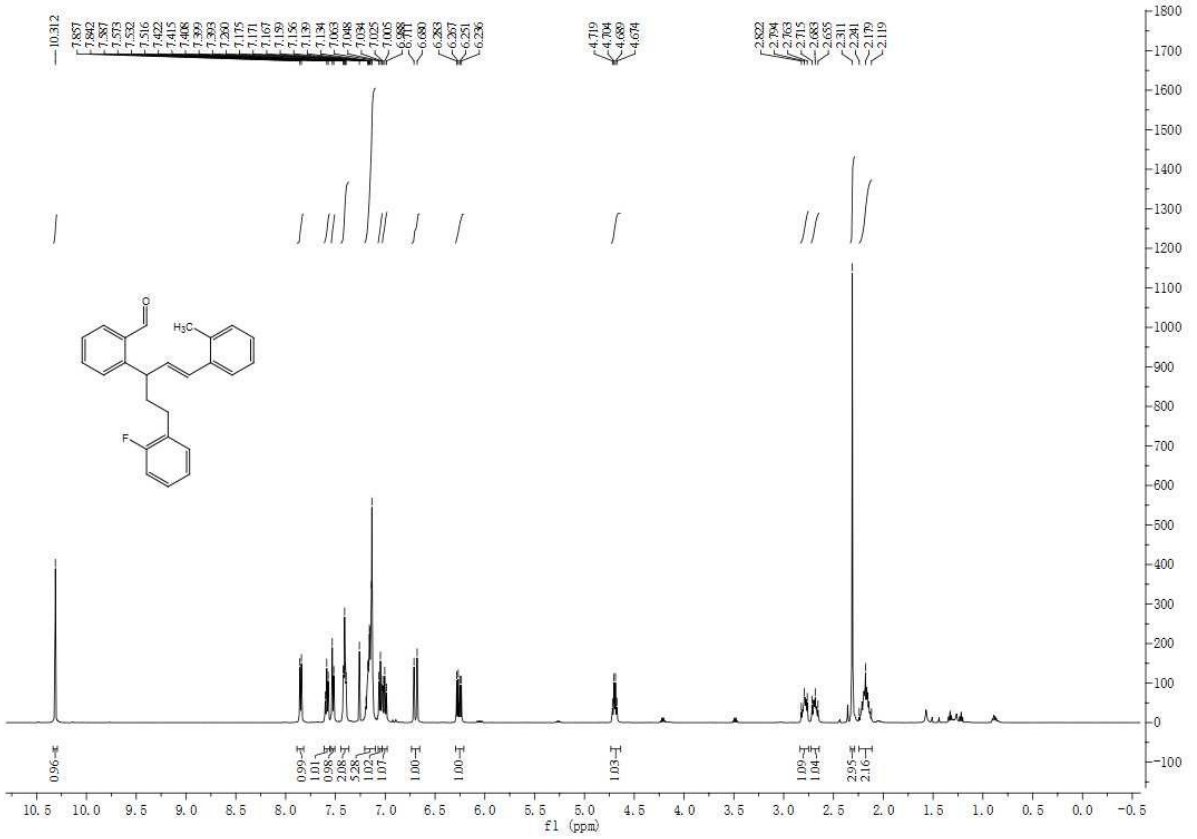
33.62
19.82
O
H3C
Cl
f1 (ppm)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7.171
7.167
7.159
7.156
7.139
7.134
7.063
7.048
7.034
7.025
7.005
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6.981
6.680
6.383
6.367
6.251
6.236
4.719
4.704
4.689
4.674
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2.794
2.763
2.715
2.683
2.655
2.311
2.241
2.179
2.119
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0.99
1.01
0.98
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1.04
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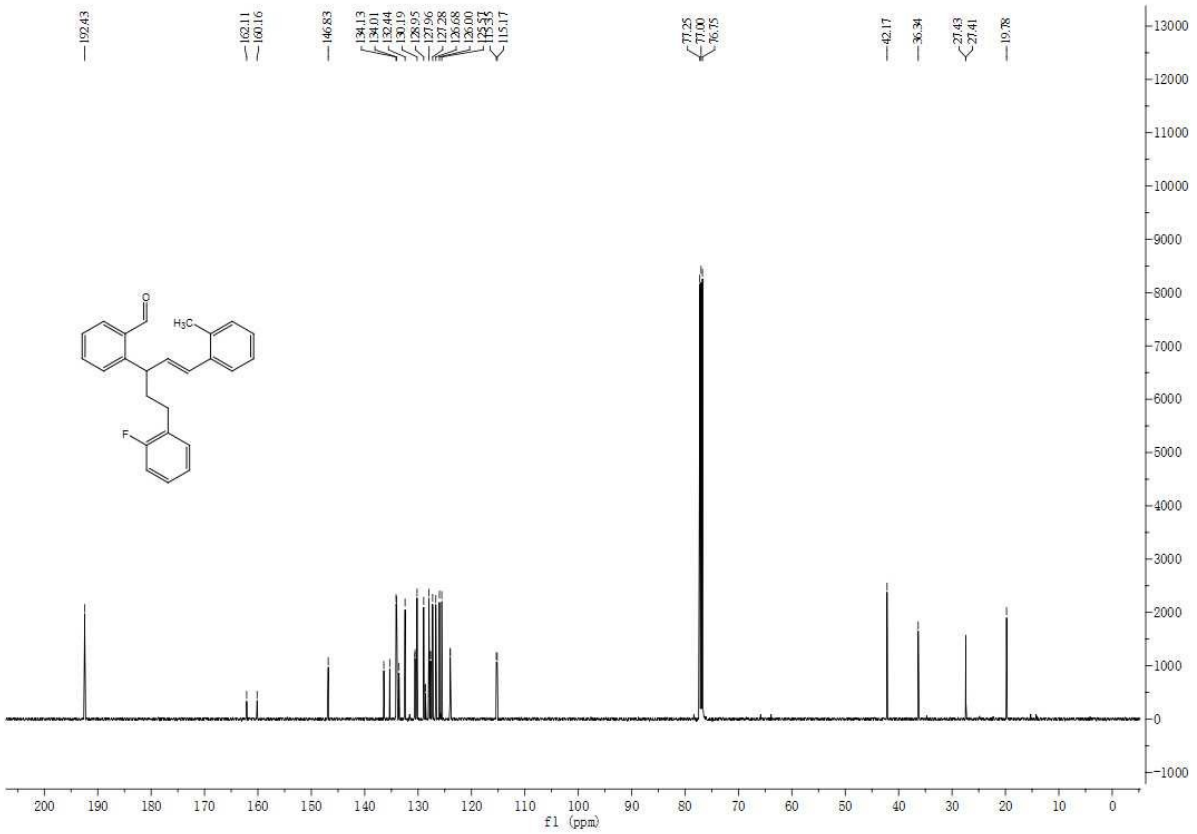
2.16
f1 (ppm)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127.28
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77.00
76.75
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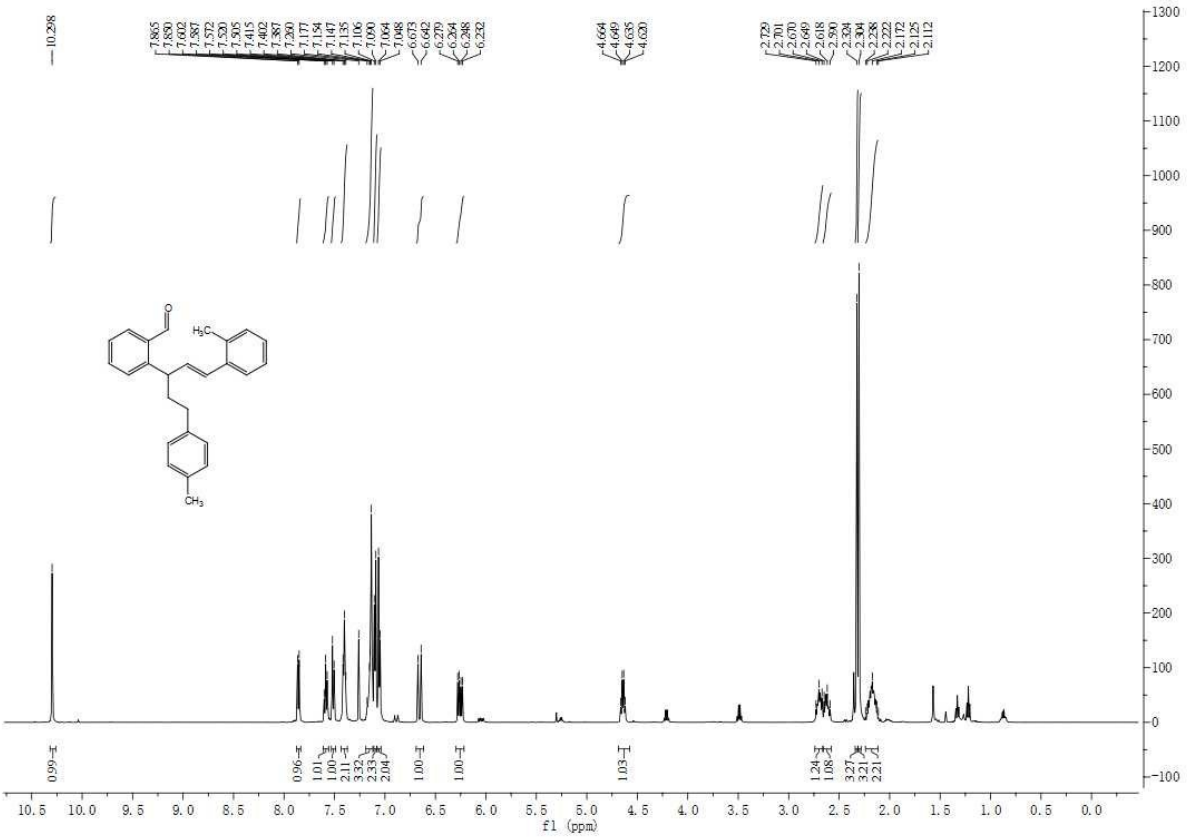
27.43
27.41
19.78
f1 (ppm)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7.048
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6.232
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4.649
4.635
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2.701
2.670
2.649
2.618
2.590
2.324
2.304
2.228
2.222
2.172
2.125
2.112
O
H3C
CH3
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0.96
1.01
1.00
2.11
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1.00
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3.27
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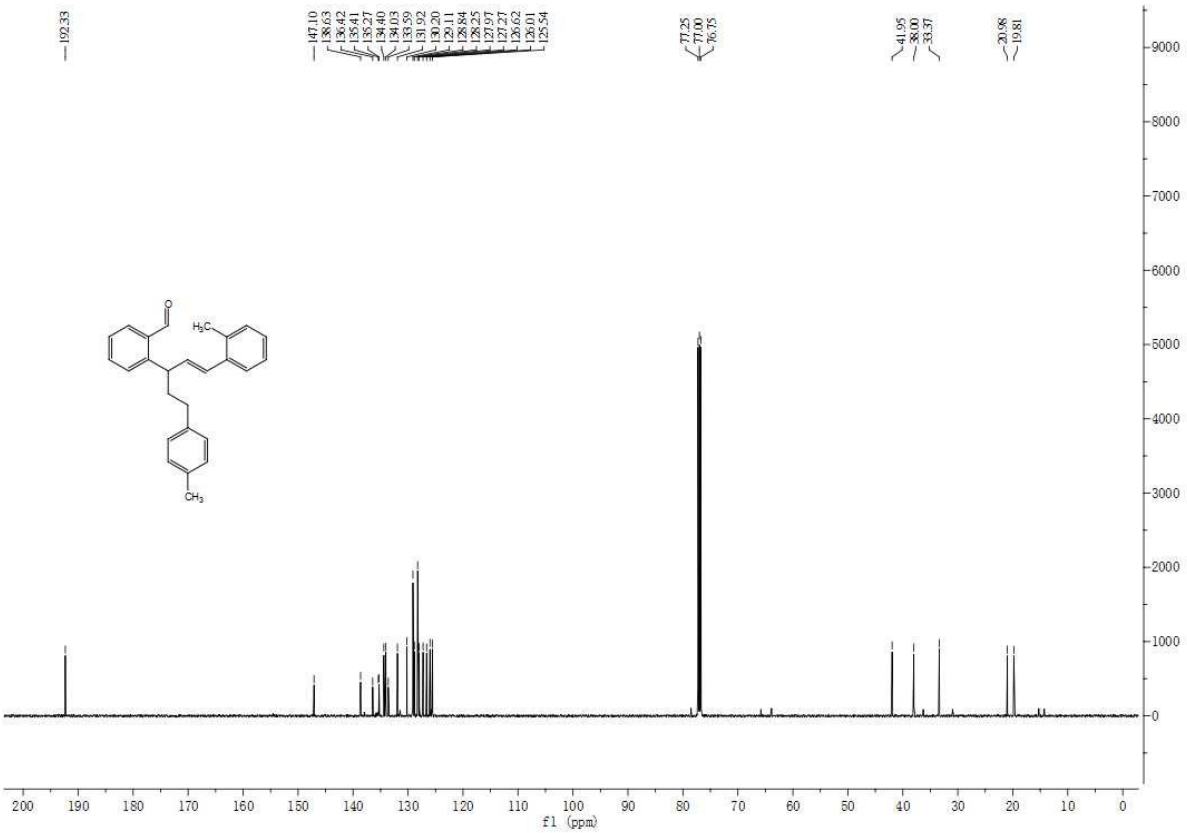
f1 (ppm)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127.27
126.62
126.01
125.54
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41.95
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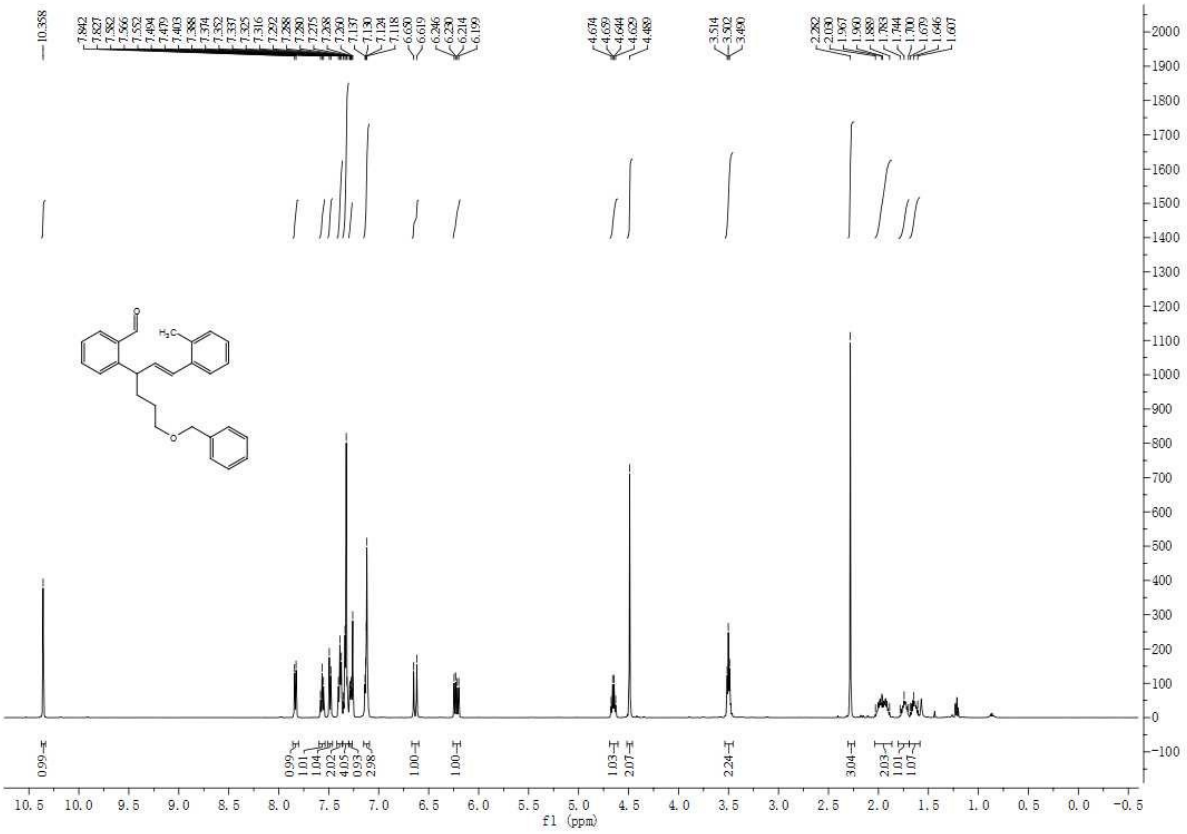
19.81
O
H3C
CH3
f1 (ppm)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6.601
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6.199
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4.659
4.644
4.629
4.489
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3.502
3.490
2.382
2.030
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1.900
1.889
1.783
1.764
1.740
1.670
1.646
1.607
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1.01
1.04
2.02
4.05
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1.00
1.03
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2.24
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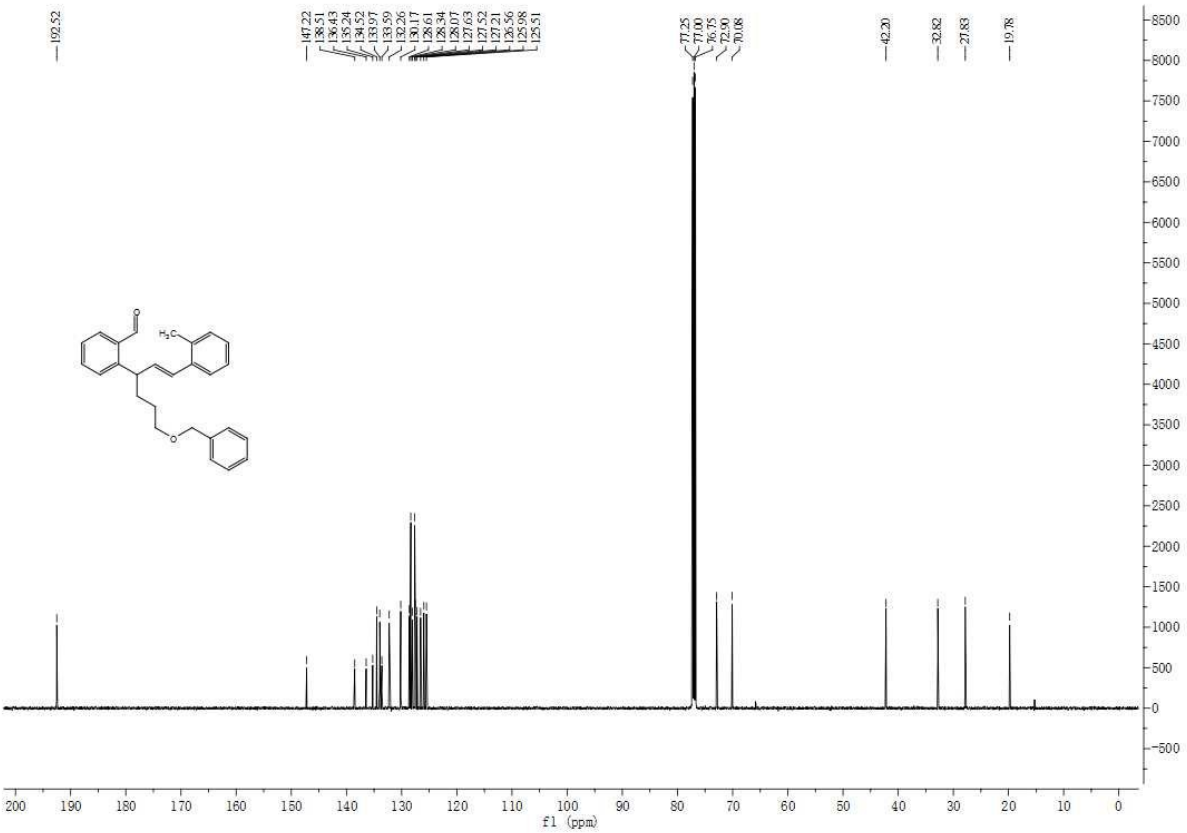
2.03
1.01
1.07
f1 (ppm)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128.07
127.63
127.52
127.21
126.56
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125.51
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77.00
76.75
72.90
70.08
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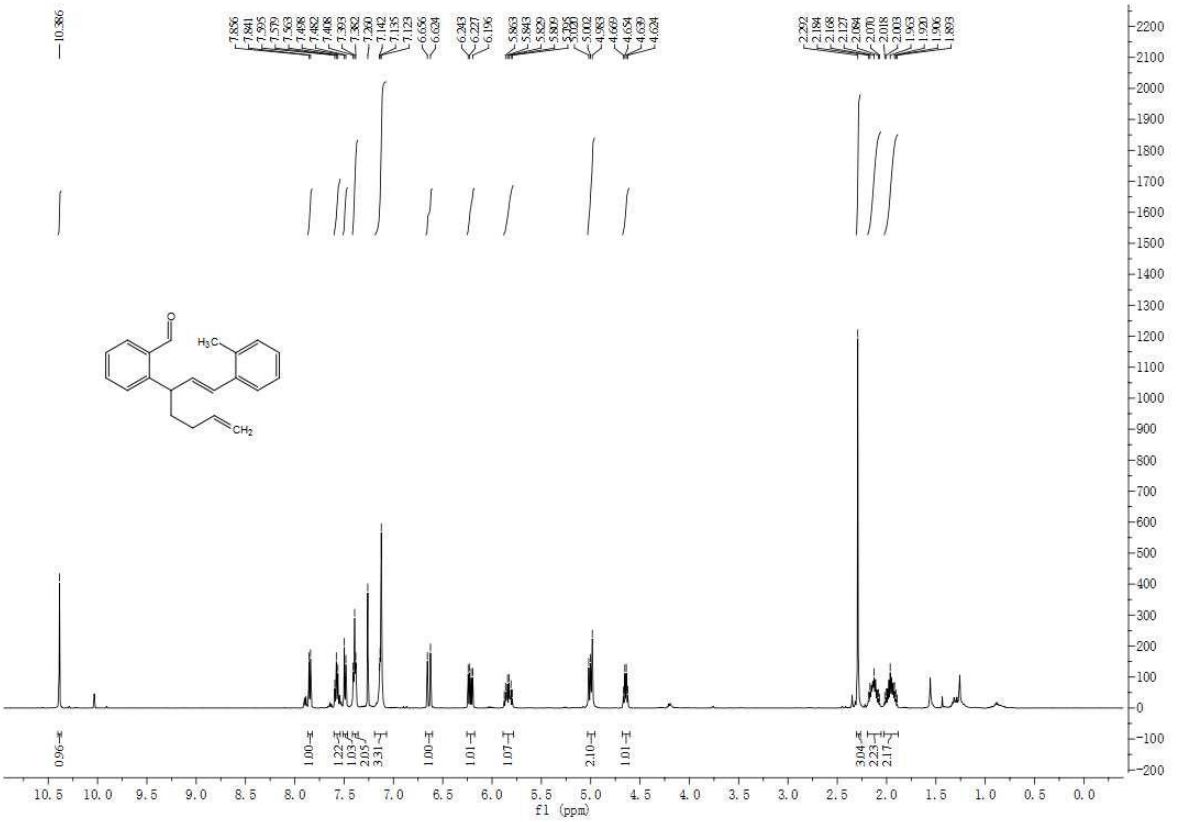
32.82
27.83
19.78
f1 (ppm)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5.002
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4.639
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1.906
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H3C
CH2
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3.04
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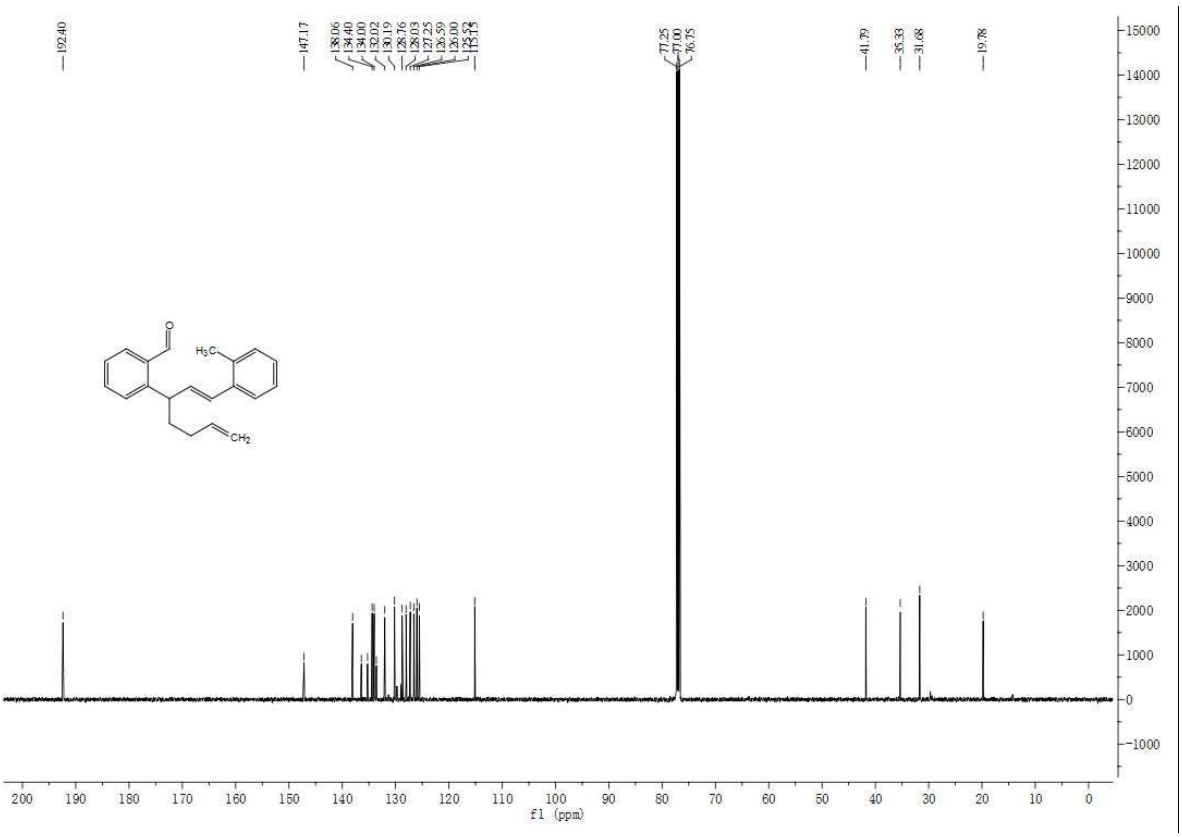
2.17
f1 (ppm)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126.59
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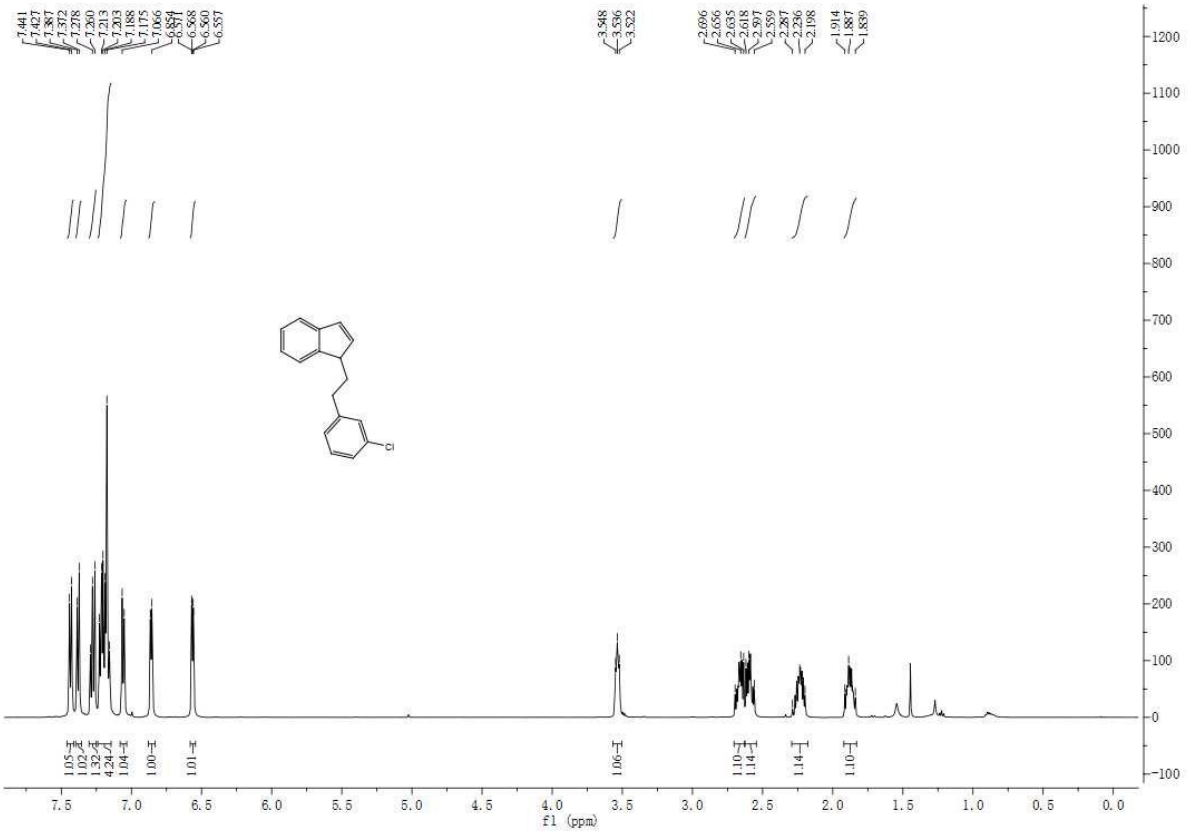
77.00
76.75
41.79
35.33
31.68
19.78
O
H3C
CH2
f1 (ppm)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2.599
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2.236
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1.887
1.839
Cl
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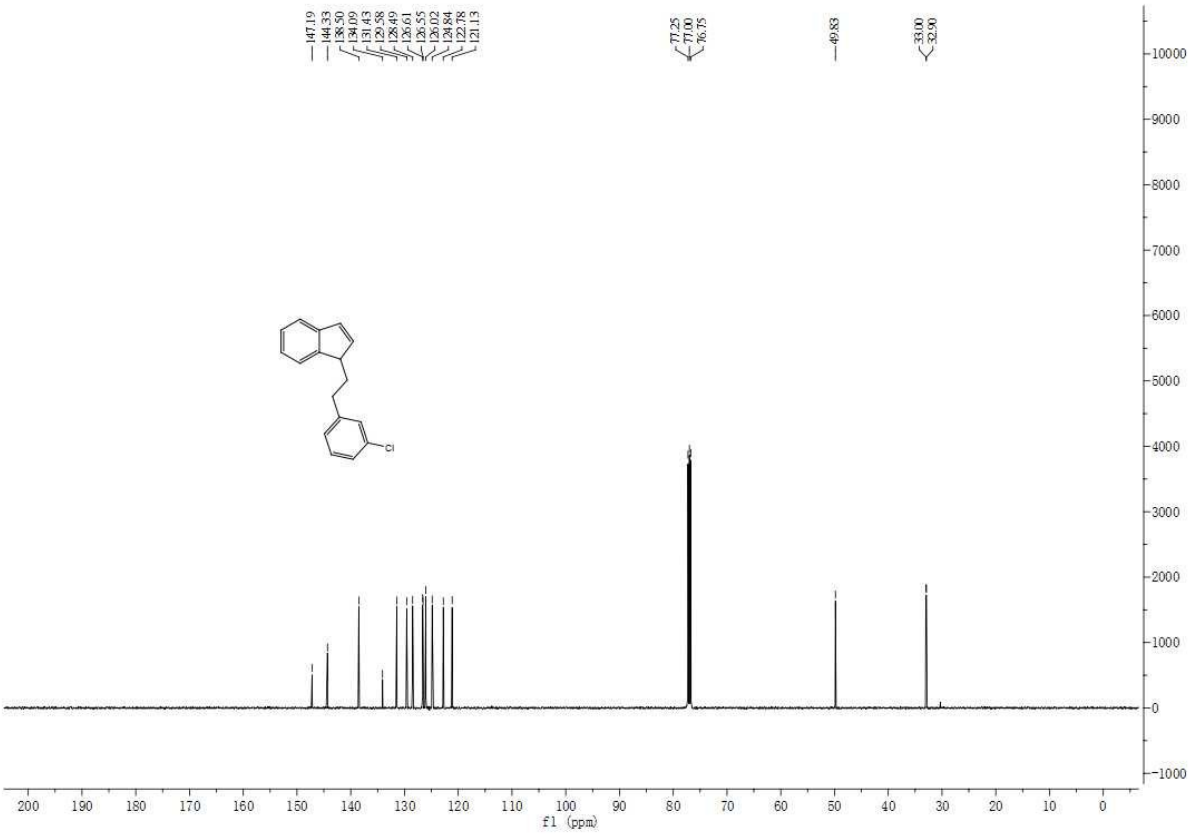
1.06
1.10
1.14
1.14
1.10
f1 (ppm)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121.13
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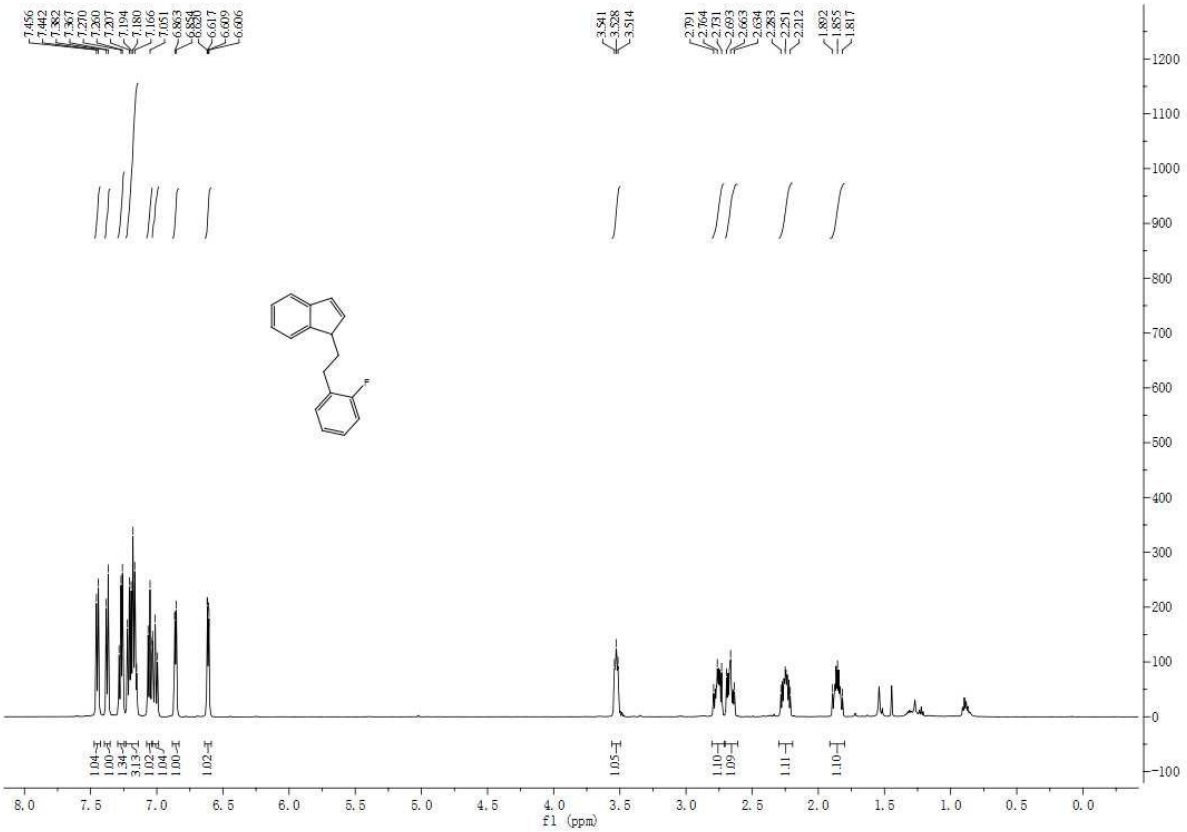
49.83
33.00
32.90
Cl
f1 (ppm)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6.617
6.609
6.606
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3.528
3.514
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2.731
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2.251
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1.10
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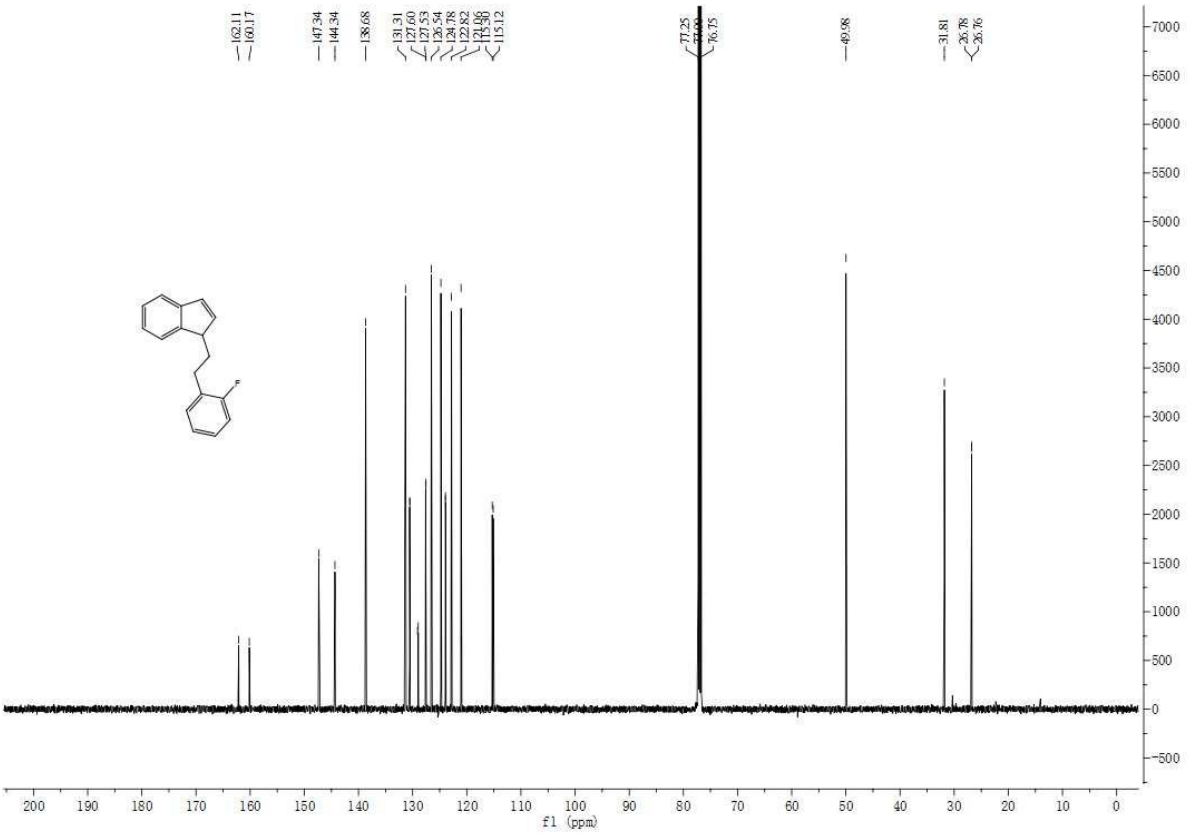
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124.78
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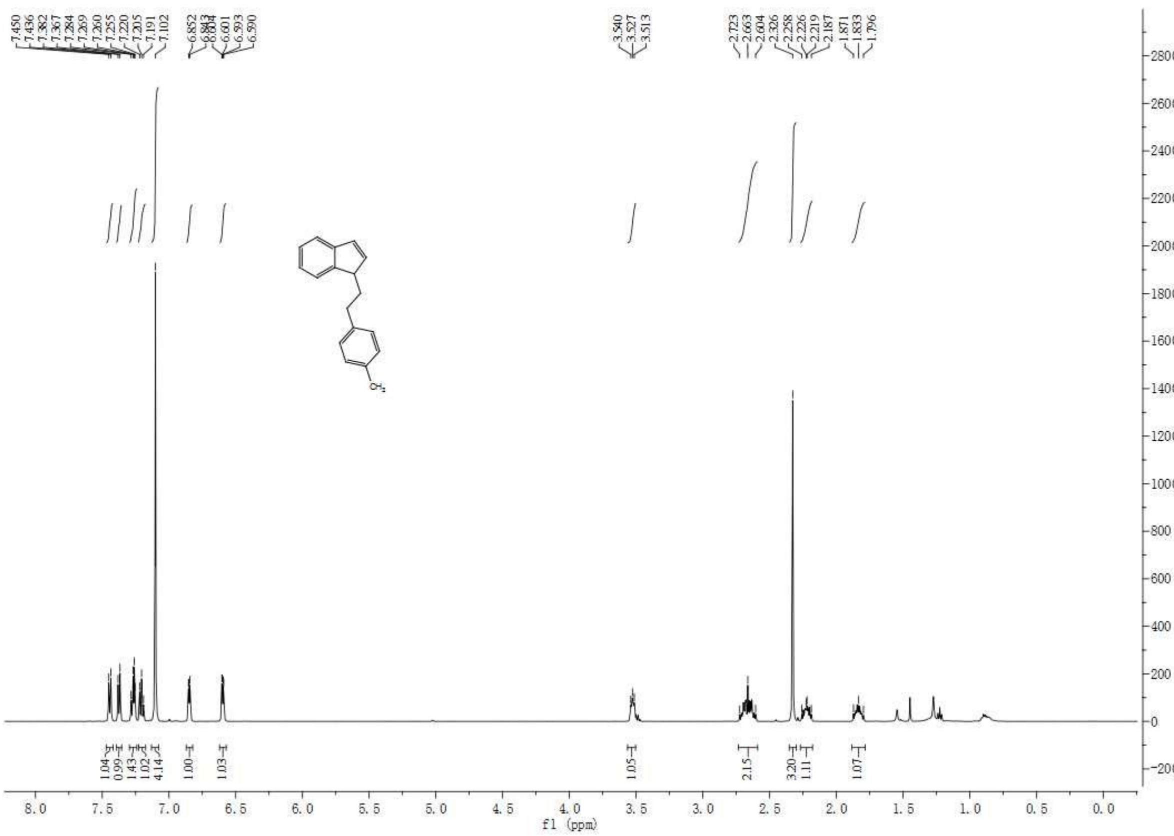
31.81
26.78
26.76
f1 (ppm)



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2.226
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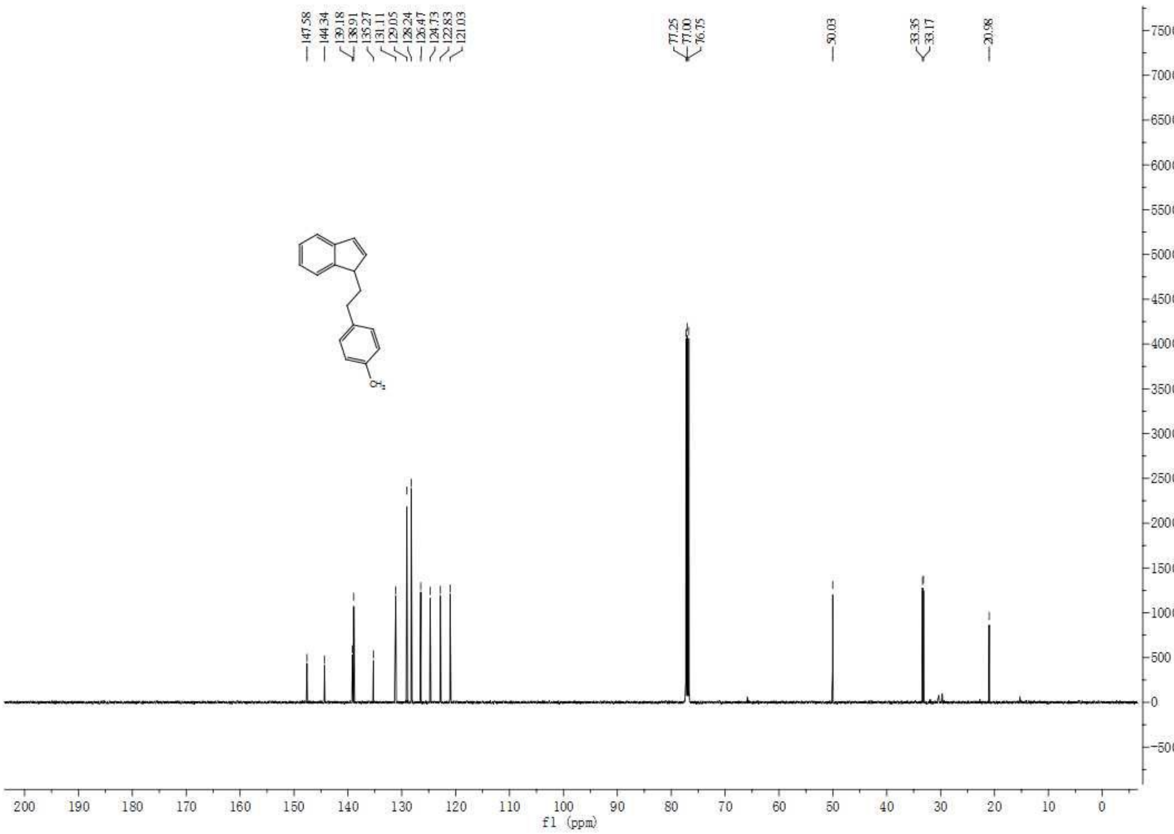
f1 (ppm)



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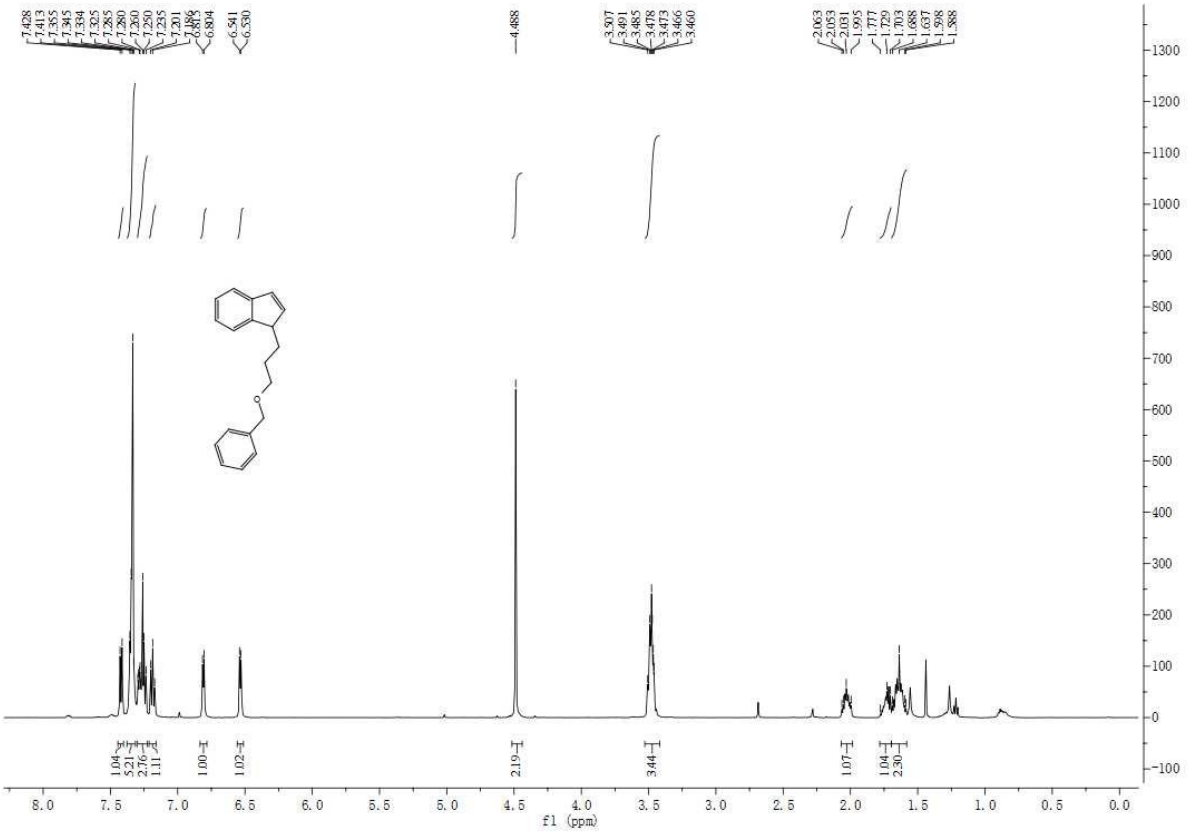
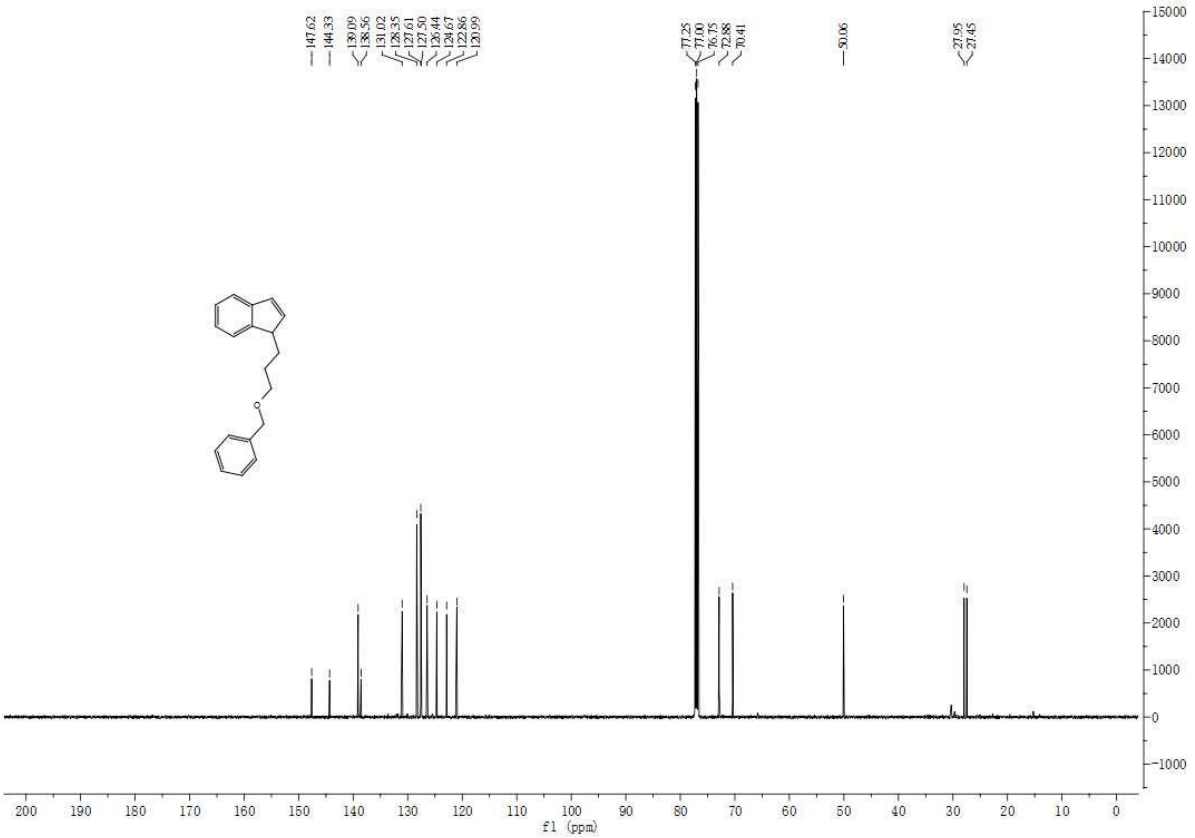
77.00
76.75
50.03
33.35
33.17
20.98
f1 (ppm)


7.428
7.413
7.355
7.345
7.334
7.325
7.285
7.280
7.260
7.250
7.235
7.201
6.804
6.541
6.530
4.488
3.507
3.491
3.485
3.478
3.473
3.466
3.460
2.063
2.053
2.031
1.995
1.777
1.729
1.703
1.688
1.637
1.598
1.368
1.04
5.21
2.76
1.11
1.00
1.02
2.19
3.44
1.07
1.04
2.30
f1 (ppm)


147.62
144.33
139.09
138.56
131.02
128.35
127.61
127.50
126.44
125.67
123.86
120.99
77.25
77.00
76.75
72.88
70.41
50.06
27.95
27.45
f1 (ppm)