

Organophosphorus-Mediated cross-Rauhut– Currier/Wittig Domino Reaction for The Efficient Synthesis of Trisubstituted Cyclopentenes

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408100, P.R. China

Supporting Information

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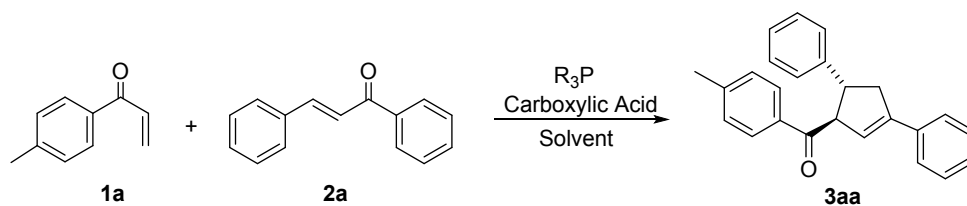
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1. General Information

Unless otherwise indicated, all reagents were purchased from commercial distributors and used without further purification. ^1H NMR and ^{13}C NMR were recorded at 400 MHz and 100 MHz respectively, using tetramethylsilane as an internal reference. Mass spectroscopy data were collected on HRMS-EI, EI-MS instrument. Melting points were detected by a digital melting point apparatus and were uncorrected. Column chromatography was performed over silica gel 200-300.

2. Optimization of the Reaction Conditions

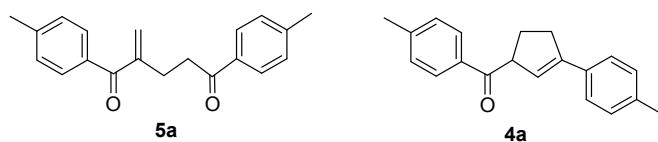
Table S1. Screening of Reaction Conditions.^[a]



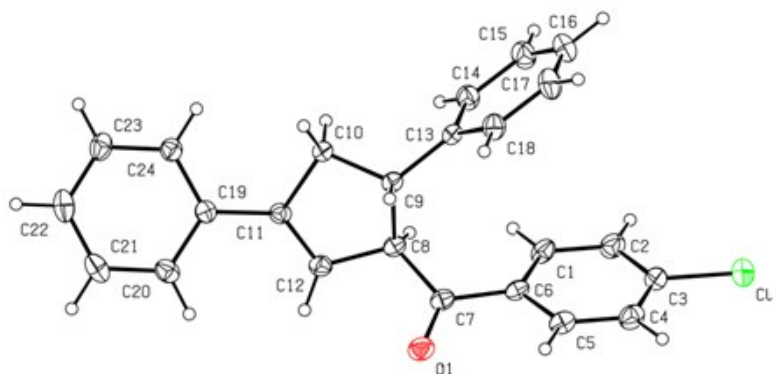
Entry	R_3P	Carboxylic Acid	Solvent	T ($^\circ\text{C}$)	Yield (%) ^[b]
1	$(n\text{-Bu})_3\text{P}$	PivOH	dioxane	100	56
2	Me_3P	PivOH	dioxane	100	29
3	$(t\text{-Bu})_3\text{P}$	PivOH	dioxane	100	0
4	Cy_3P	PivOH	dioxane	100	Trace
5	Ph_3P	PivOH	dioxane	100	Trace
6	$(n\text{-Bu})_3\text{P}$	BzOH	dioxane	100	40
7	$(n\text{-Bu})_3\text{P}$	HOAc	dioxane	100	83
8	$(n\text{-Bu})_3\text{P}$	<i>p</i> - NO_2BzOH	dioxane	100	0
9 ^[c]	$(n\text{-Bu})_3\text{P}$	$(\text{CF}_3)_2\text{CHOH}$	dioxane	100	68
10	$(n\text{-Bu})_3\text{P}$	$(\text{CO}_2\text{H})_2$	dioxane	100	0
11 ^[d]	$(n\text{-Bu})_3\text{P}$	HOAc	dioxane	100	61

12 ^[e]	(<i>n</i> -Bu) ₃ P	HOAc	dioxane	100	50
13 ^[f]	(<i>n</i> -Bu) ₃ P	-	dioxane	100	0
14	(<i>n</i> -Bu) ₃ P	HOAc	toluene	100	76
15	(<i>n</i> -Bu) ₃ P	HOAc	DME	100	0
16	(<i>n</i> -Bu) ₃ P	HOAc	CH ₃ CN	100	0
17	(<i>n</i> -Bu) ₃ P	HOAc	DMF	100	0
18	(<i>n</i> -Bu) ₃ P	HOAc	dioxane	90	47
19	(<i>n</i> -Bu) ₃ P	HOAc	dioxane	110	66

[a] The mixture of **1a** (0.1 mmol), **2a** (0.2 mmol), phosphine (100 mol %), and acid (2.0 equiv.) was stirred in solvent (2 mL) at 100 °C for 48h under N₂ (1 atm). [b] Isolated yield. [c] Dioxane/(CF₃)₂CHOH (35/1, 1.5 mL) [d] 1.0 equiv. HOAc. [e] 3.0 equiv. HOAc. [f] Major product is **5a** and minor product is **4a**.



3. X-ray Crystallographic Data of Trisubstituted Cyclopentene **3da**



Datablock: 200715_3da_0m

Bond precision: C-C = 0.0019 A Wavelength=0.71073

Cell: a=7.972(3) b=9.389(3) c=13.279(5)
 alpha=85.406(17) beta=84.165(19) gamma=67.620(18)

Temperature: 170 K

	Calculated	Reported
Volume	913.4(6)	913.3(6)
Space group	P -1	P -1
Hall group	-P 1	-P 1
Moiety formula	C24 H19 Cl O	C24 H19 Cl O
Sum formula	C24 H19 Cl O	C24 H19 Cl O
Mr	358.84	358.84
Dx, g cm-3	1.305	1.305
Z	2	2
Mu (mm-1)	0.219	0.219
F000	376.0	376.0
F000'	376.44	
h,k,lmax	10,12,17	10,12,17
Nref	4031	4019
Tmin,Tmax	0.927,0.951	0.717,0.746
Tmin'	0.904	

Correction method= # Reported T Limits: Tmin=0.717 Tmax=0.746
AbsCorr = MULTI-SCAN

Data completeness= 0.997 Theta(max)= 27.113

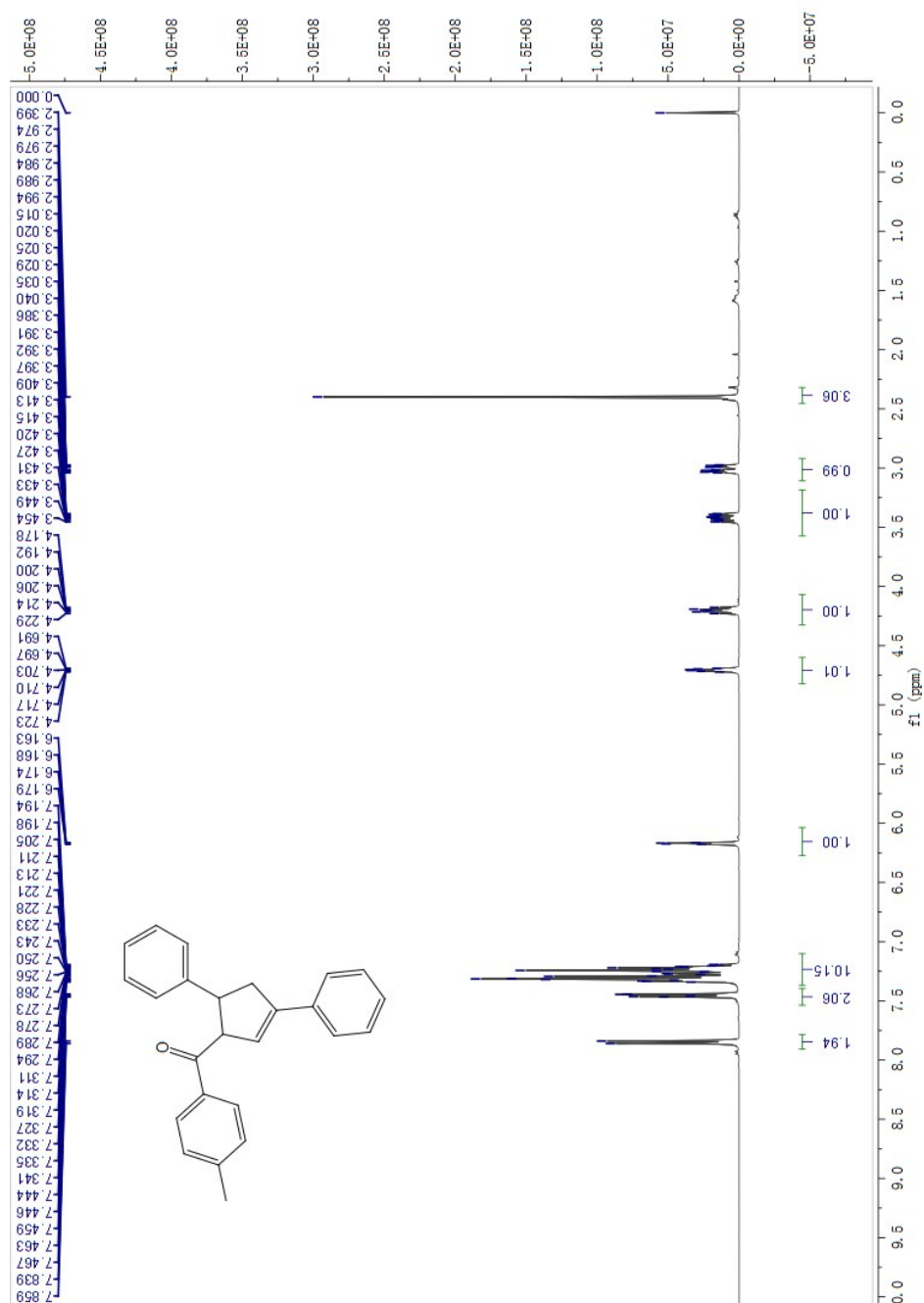
R(reflections)= 0.0372(3609) wR2(reflections)= 0.0972(4019)

S = 1.047 Npar= 235

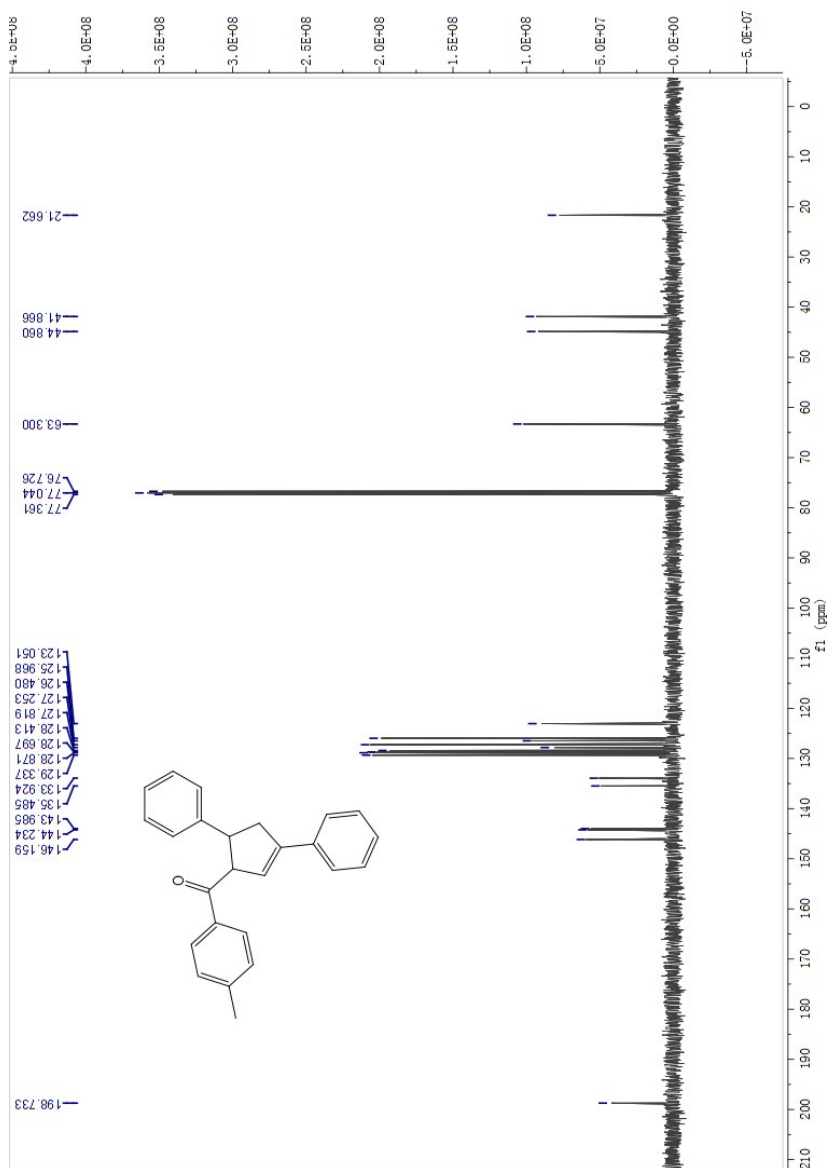
CCDC 2022239 contains the supplementary crystallographic data for **3da**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via [www.ccdc.cam.ac.uk/ data_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif)

4. ¹H NMR, ¹³C NMR, HR-MS Spectra of New Products

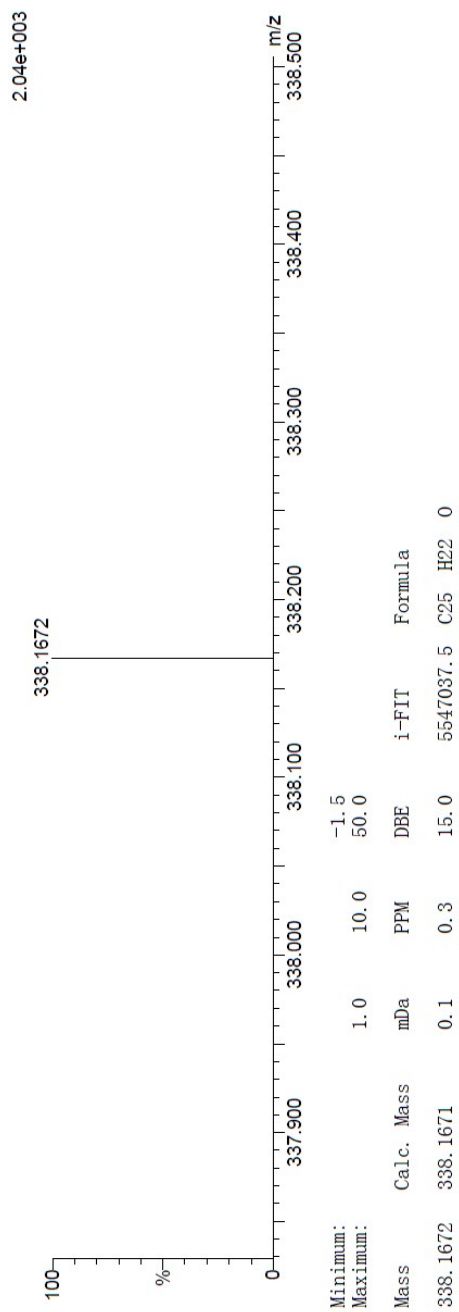
¹H NMR Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-tolyl) methanone **3aa**



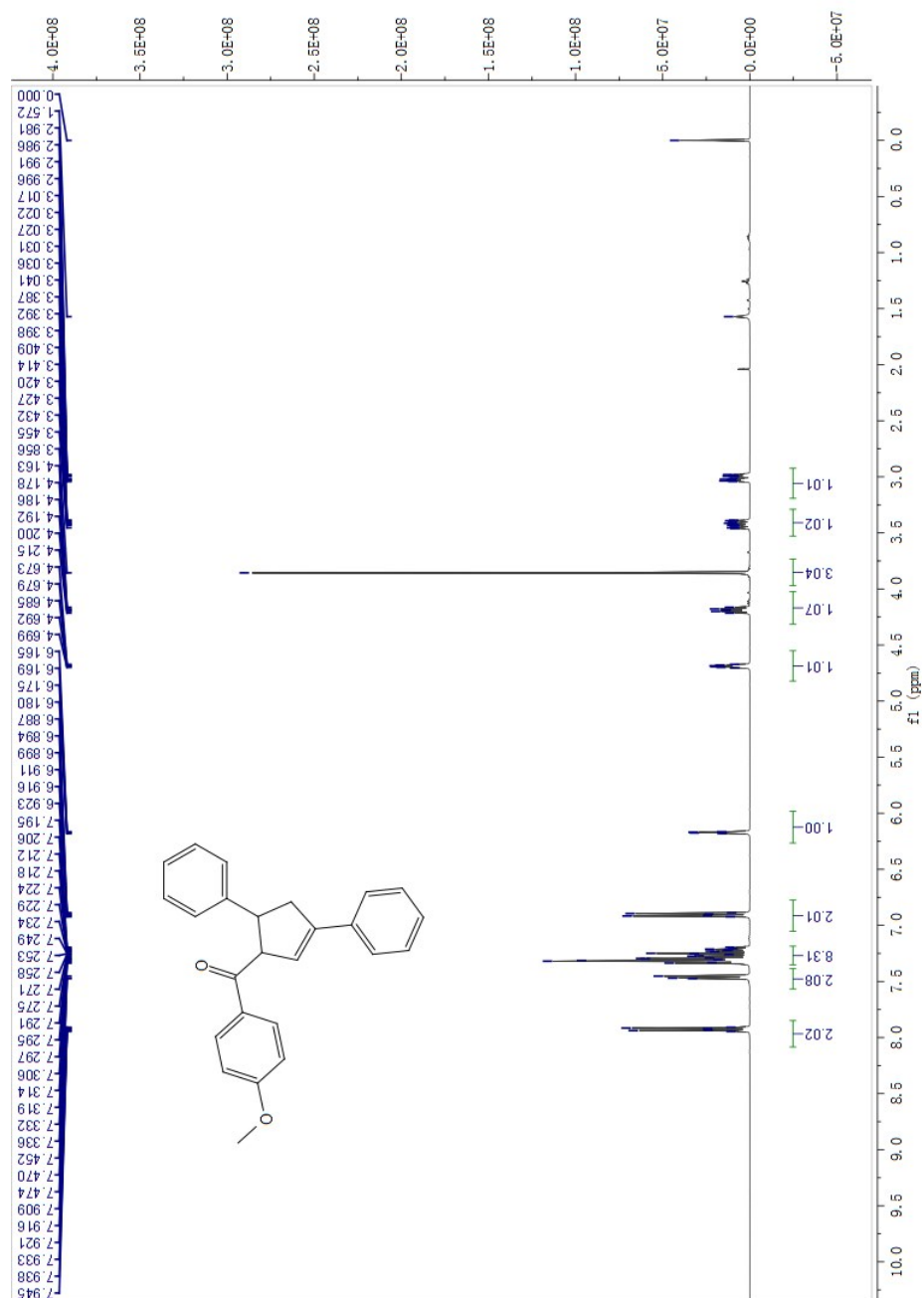
^{13}C NMR Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-tolyl) methanone **3aa**



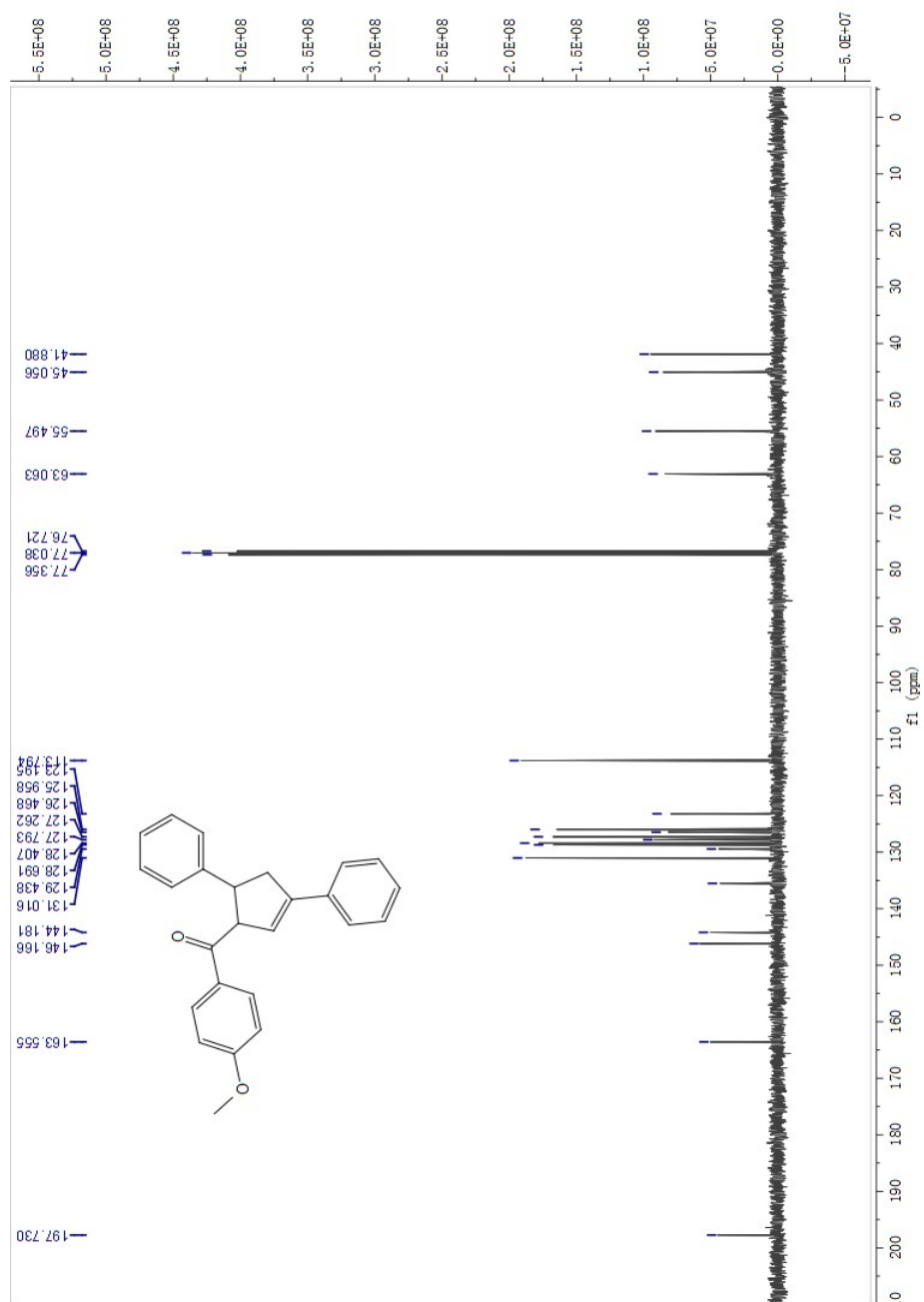
HR-MS Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-tolyl) methanone **3aa**



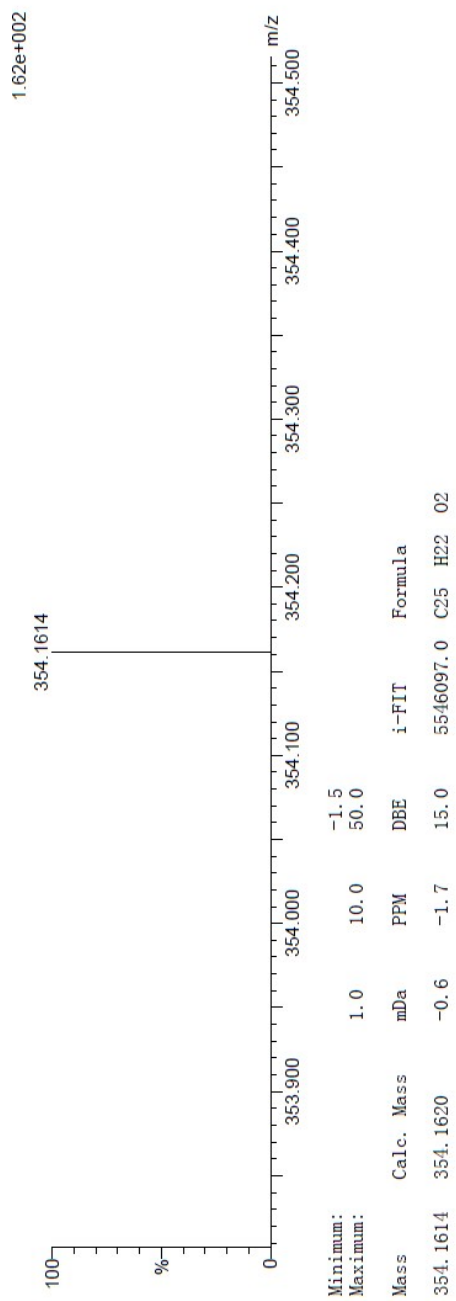
^1H NMR Spectrum of (3,5-diphenylcyclopent-2-en-1-yl) (4-methoxyphenyl) methanone **3ba**



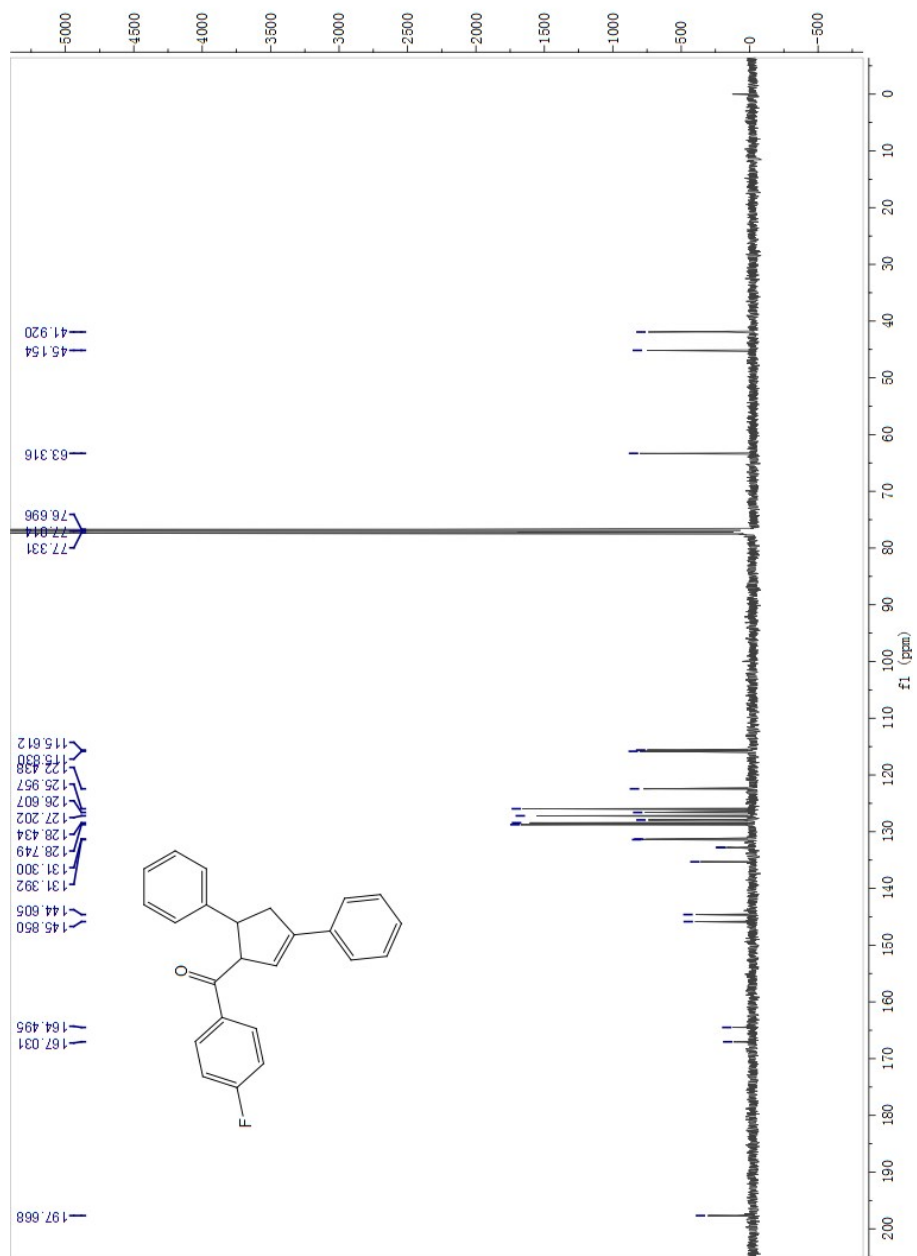
^{13}C NMR Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-methoxyphenyl) methanone **3ba**



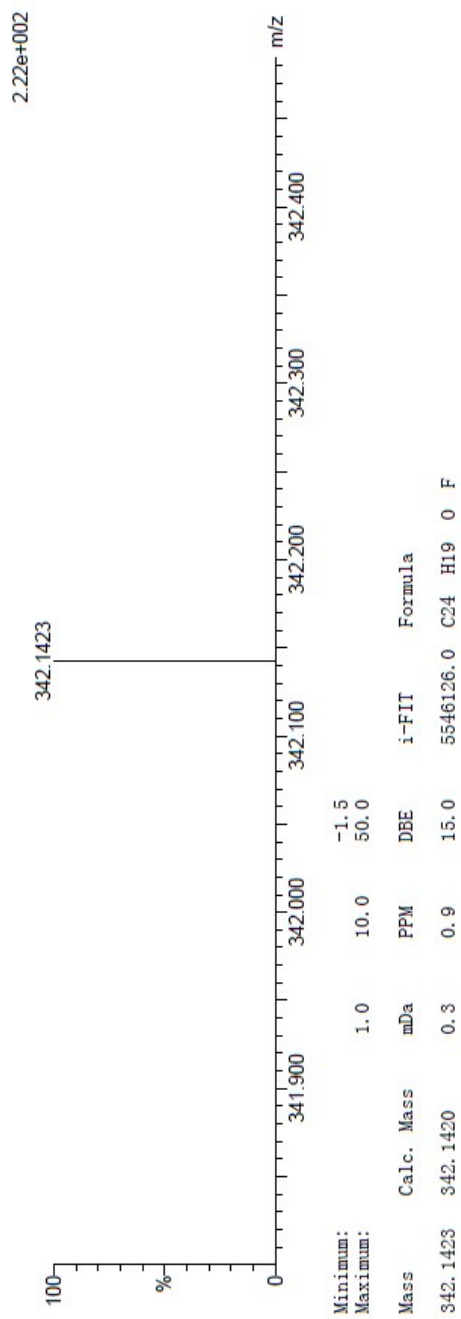
HR-MS Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-methoxyphenyl) methanone **3ba**



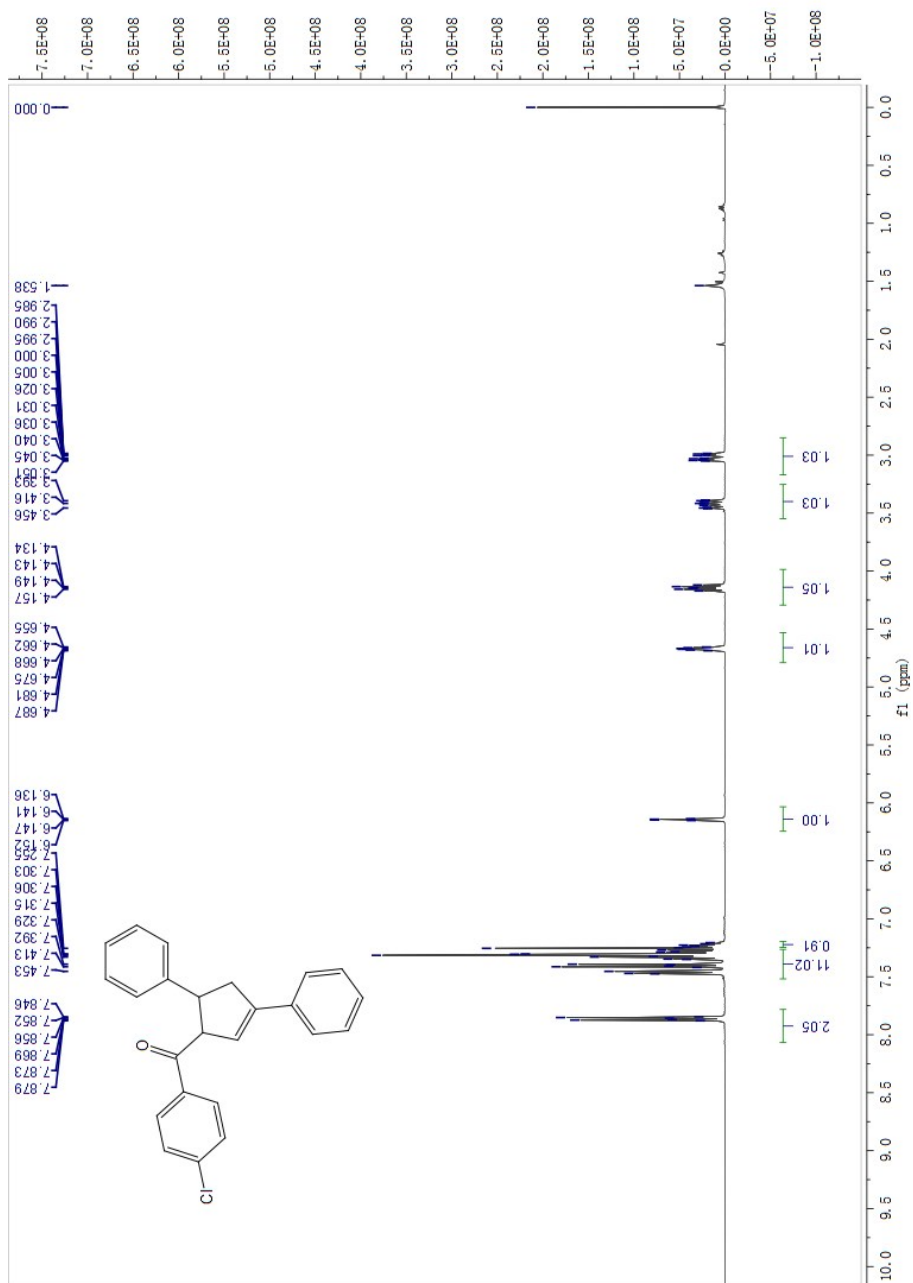
¹³C NMR Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-fluorophenyl) methanone **3ca**



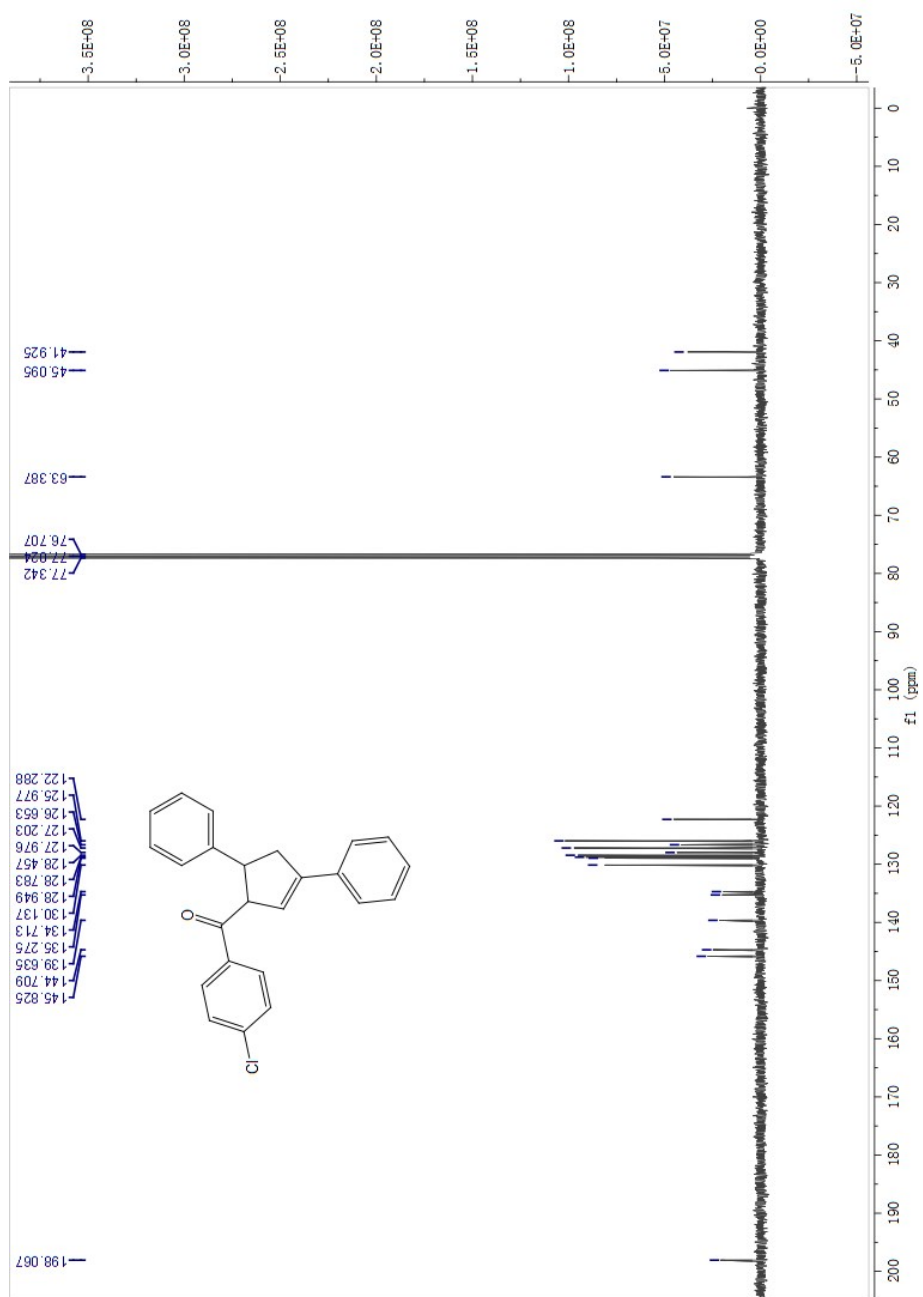
HR-MS Spectrum of (3,5-diphenylcyclopent-2-en-1-yl)(4-fluorophenyl) methanone **3ca**



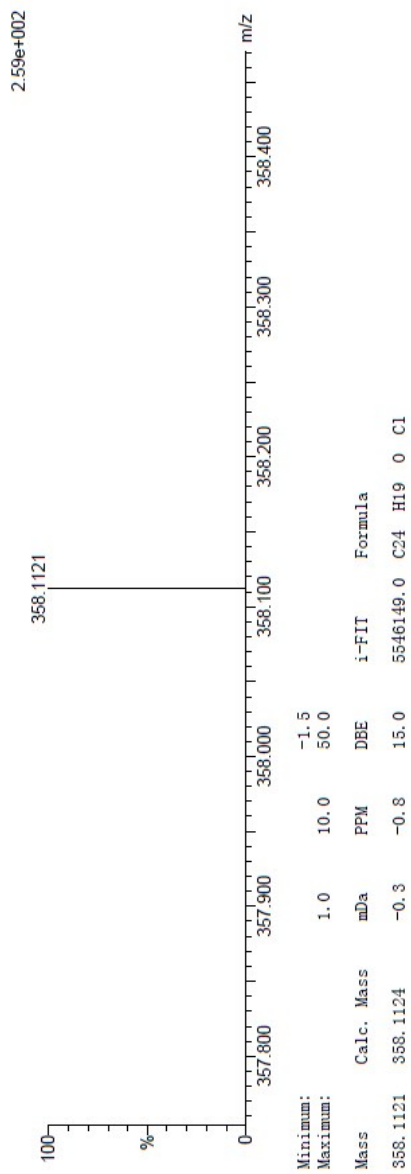
¹H NMR Spectrum of (4-chlorophenyl)(3,5-diphenylcyclopent-2-en-1-yl) methanone **3da**



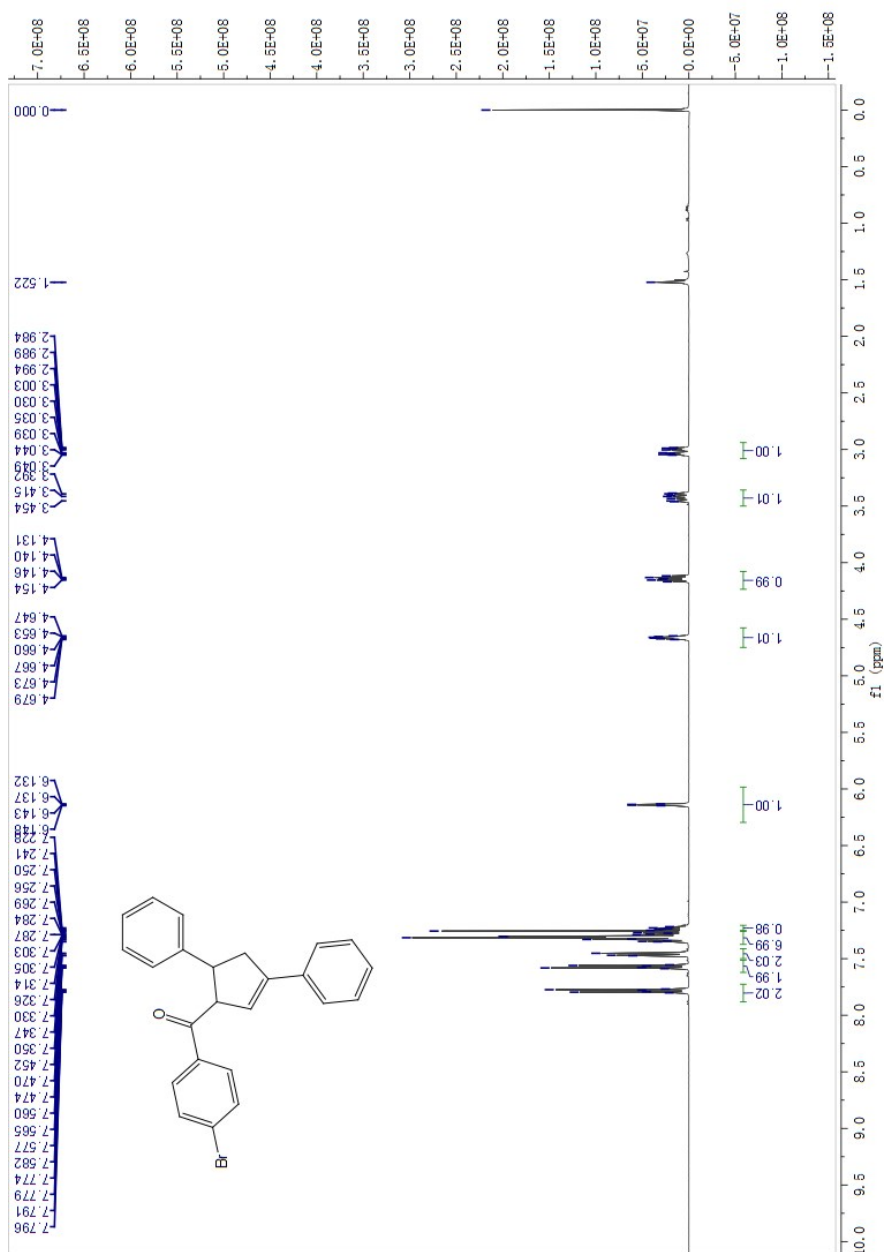
¹³C NMR Spectrum of (4-chlorophenyl)(3,5-diphenylcyclopent-2-en-1-yl) methanone **3da**



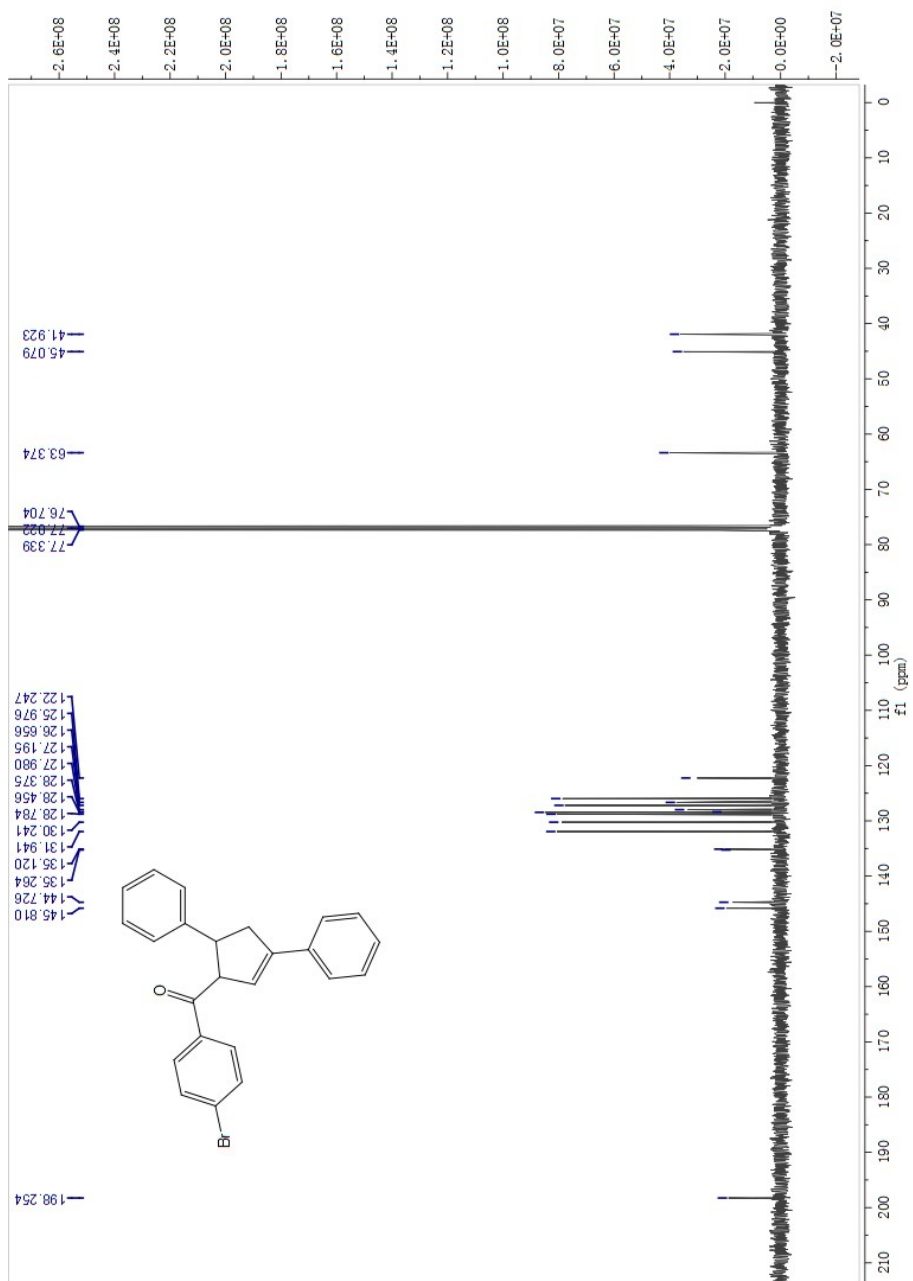
HR-MS Spectrum of (4-chlorophenyl)(3,5-diphenylcyclopent-2-en-1-yl) methanone **3da**



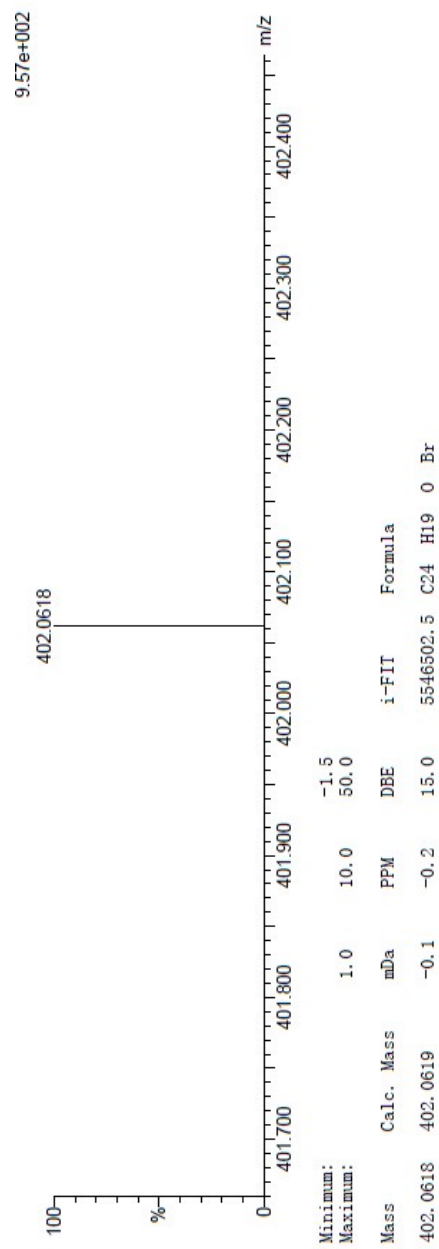
¹H NMR Spectrum of (4-bromophenyl)(3,5-diphenylcyclopent-2-en-1-yl) methanone **3ea**



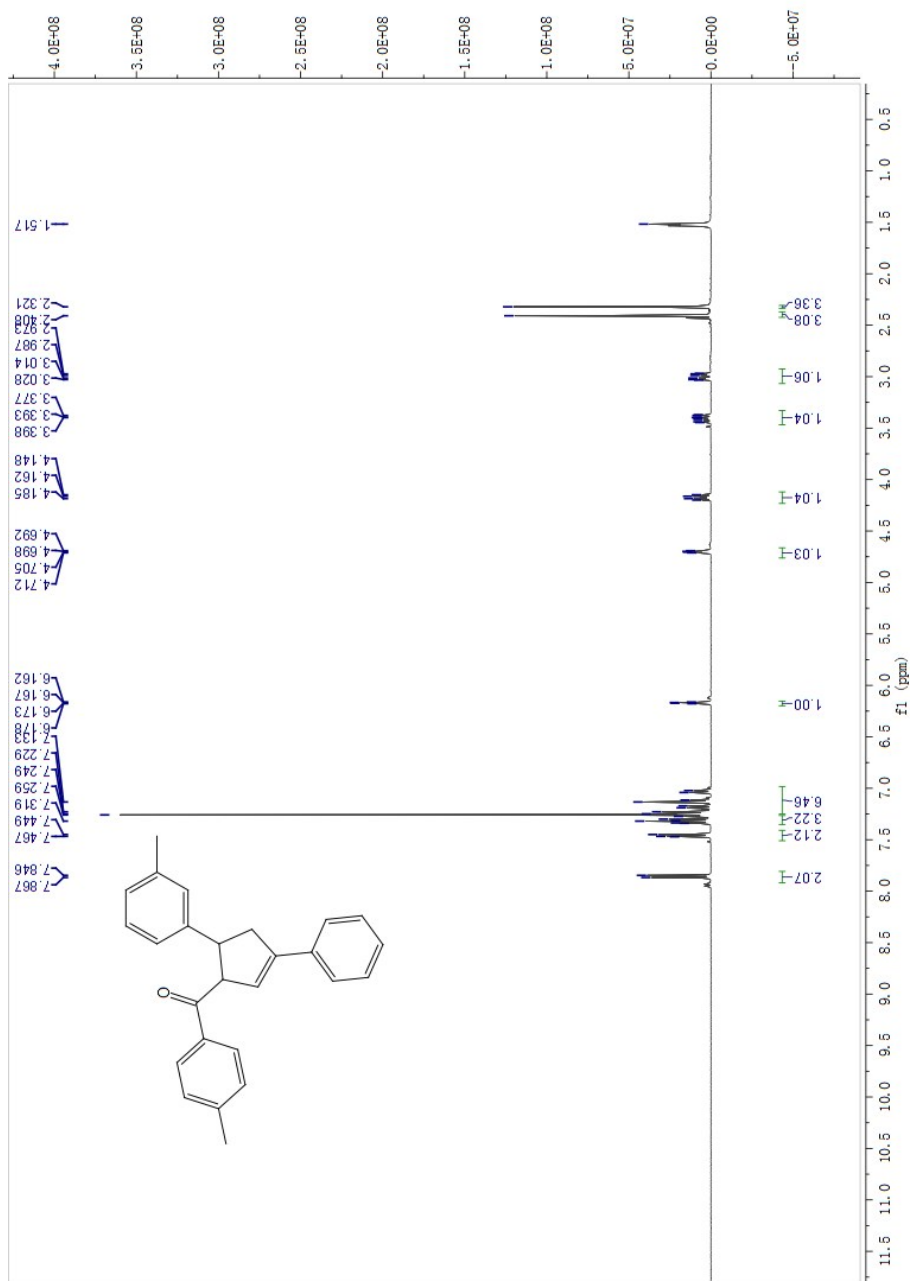
¹³C NMR Spectrum of (4-bromophenyl)(3,5-diphenylcyclopent-2-en-1-yl) methanone **3ea**



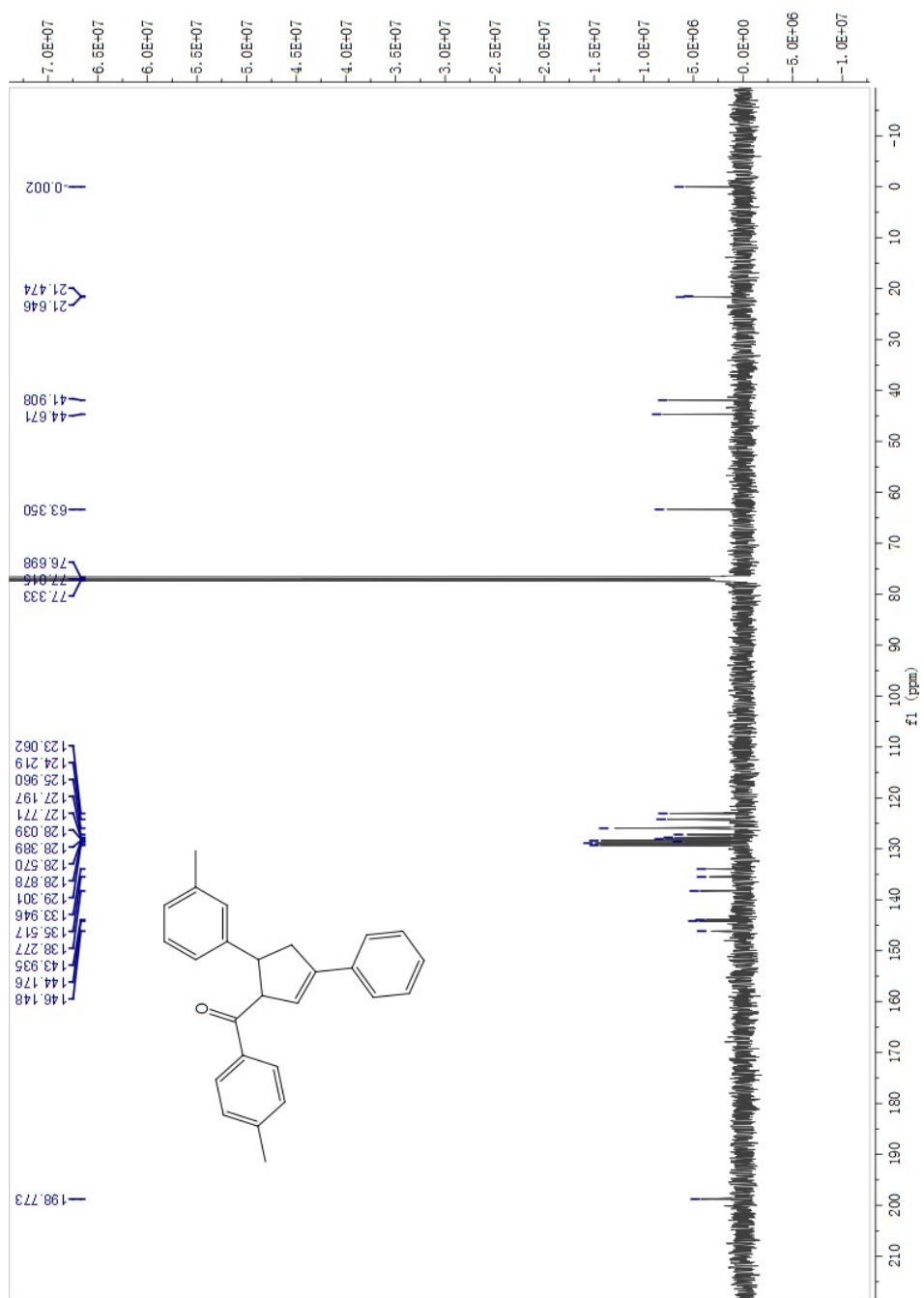
HR-MS Spectrum of (4-bromophenyl)(3,5-diphenylcyclopent-2-en-1-yl) methanone **3ea**



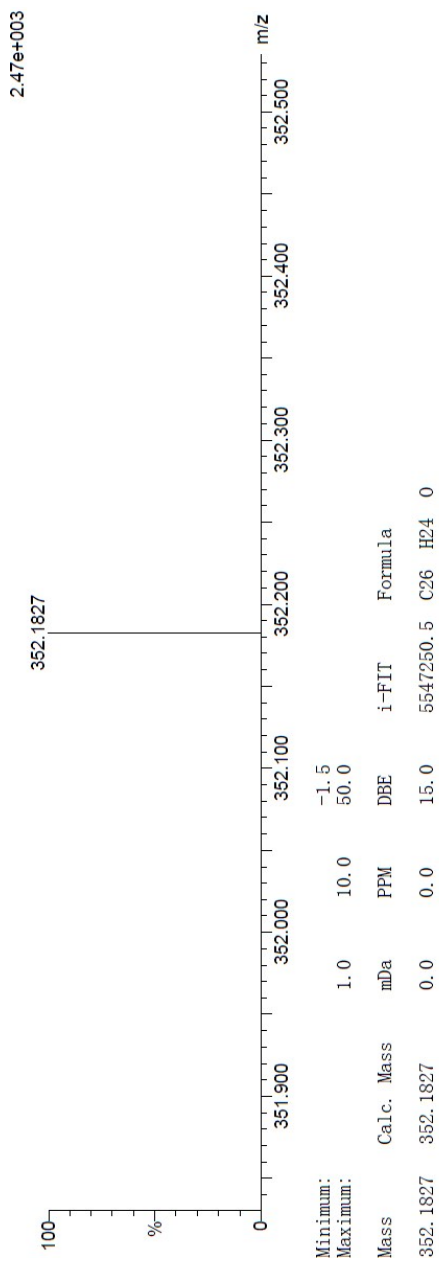
¹H NMR Spectrum of (3-phenyl-5-(3-tolyl)cyclopent-2-en-1-yl)(4-tolyl) methanone **3ab**



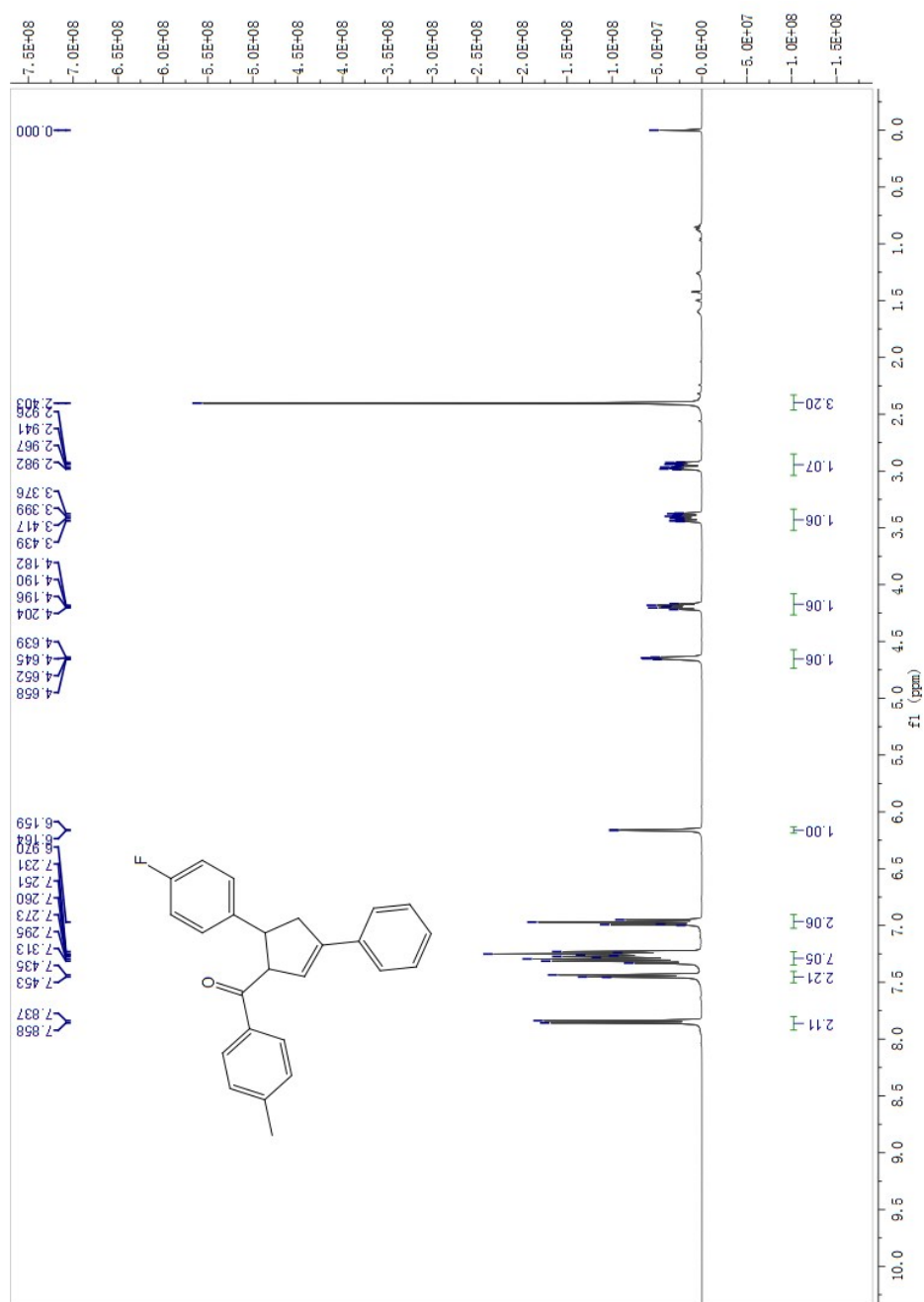
¹³C NMR Spectrum of (3-phenyl-5-(3-tolyl)cyclopent-2-en-1-yl)(4-tolyl) methanone **3ab**



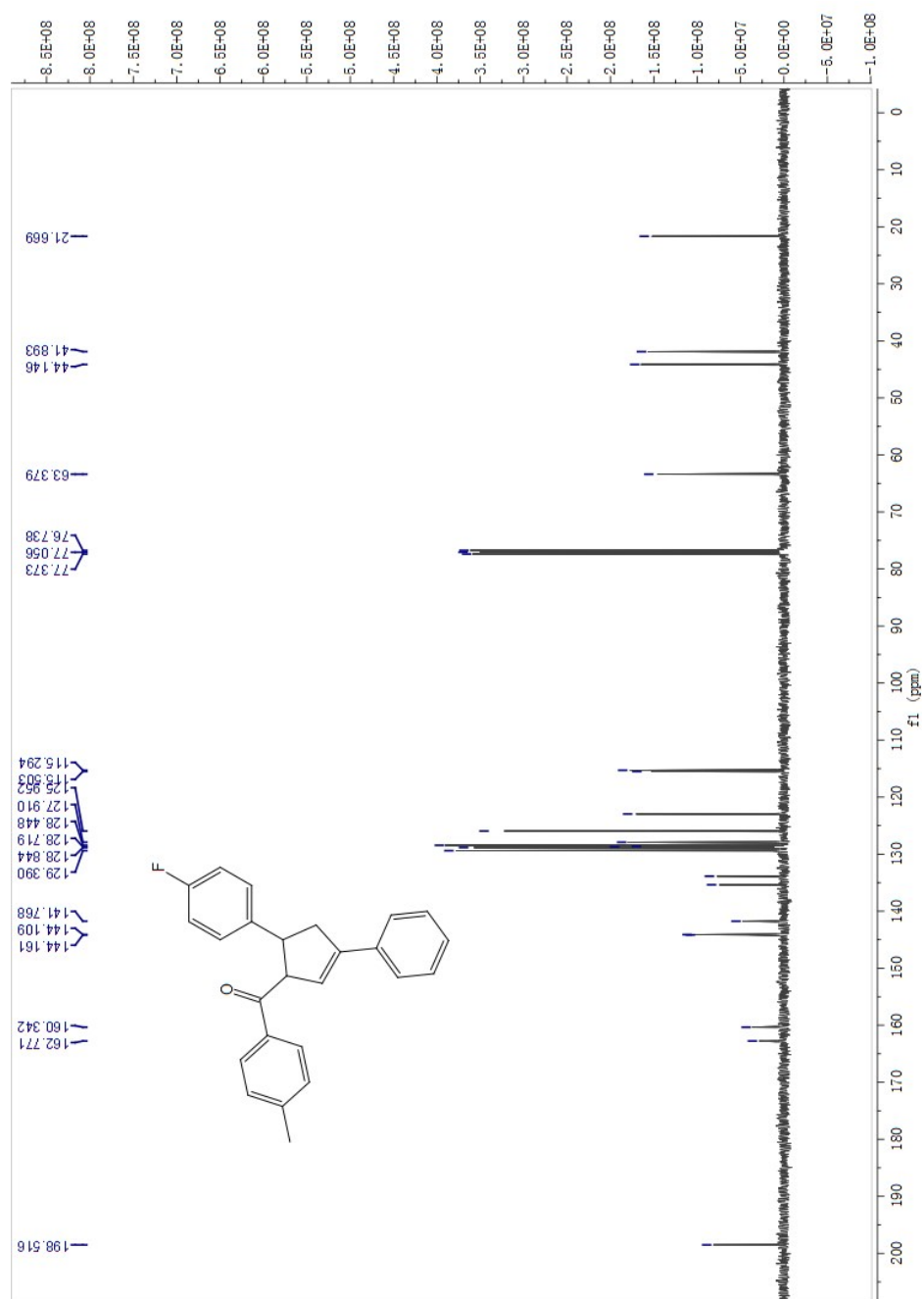
HR-MS Spectrum of (3-phenyl-5-(3-tolyl)cyclopent-2-en-1-yl)(4-tolyl) methanone **3ab**



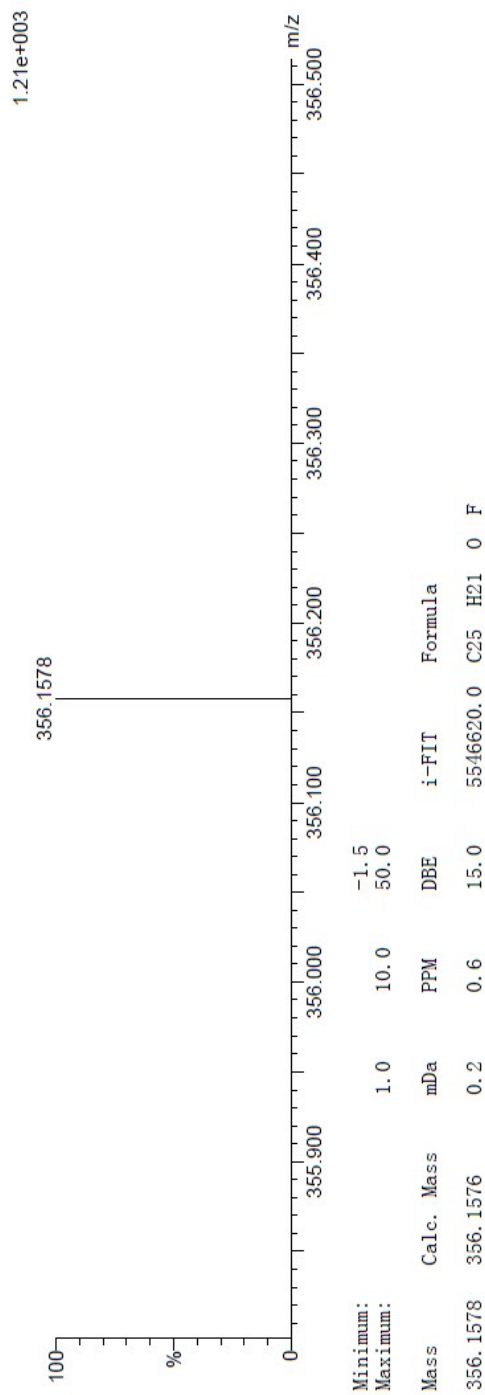
¹H NMR Spectrum of (5-(4-fluorophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ac**



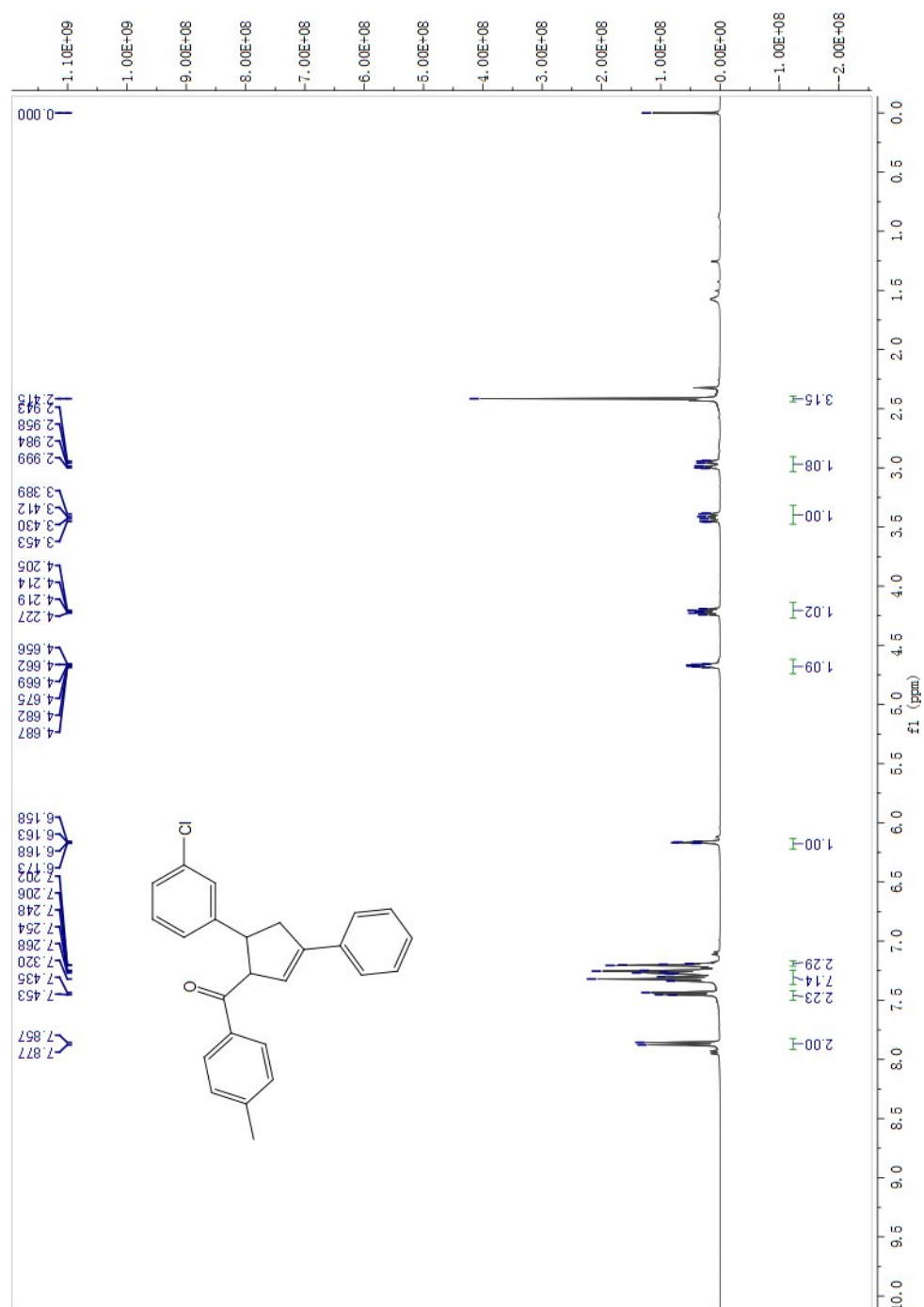
¹³C NMR Spectrum of (5-(4-fluorophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ac**



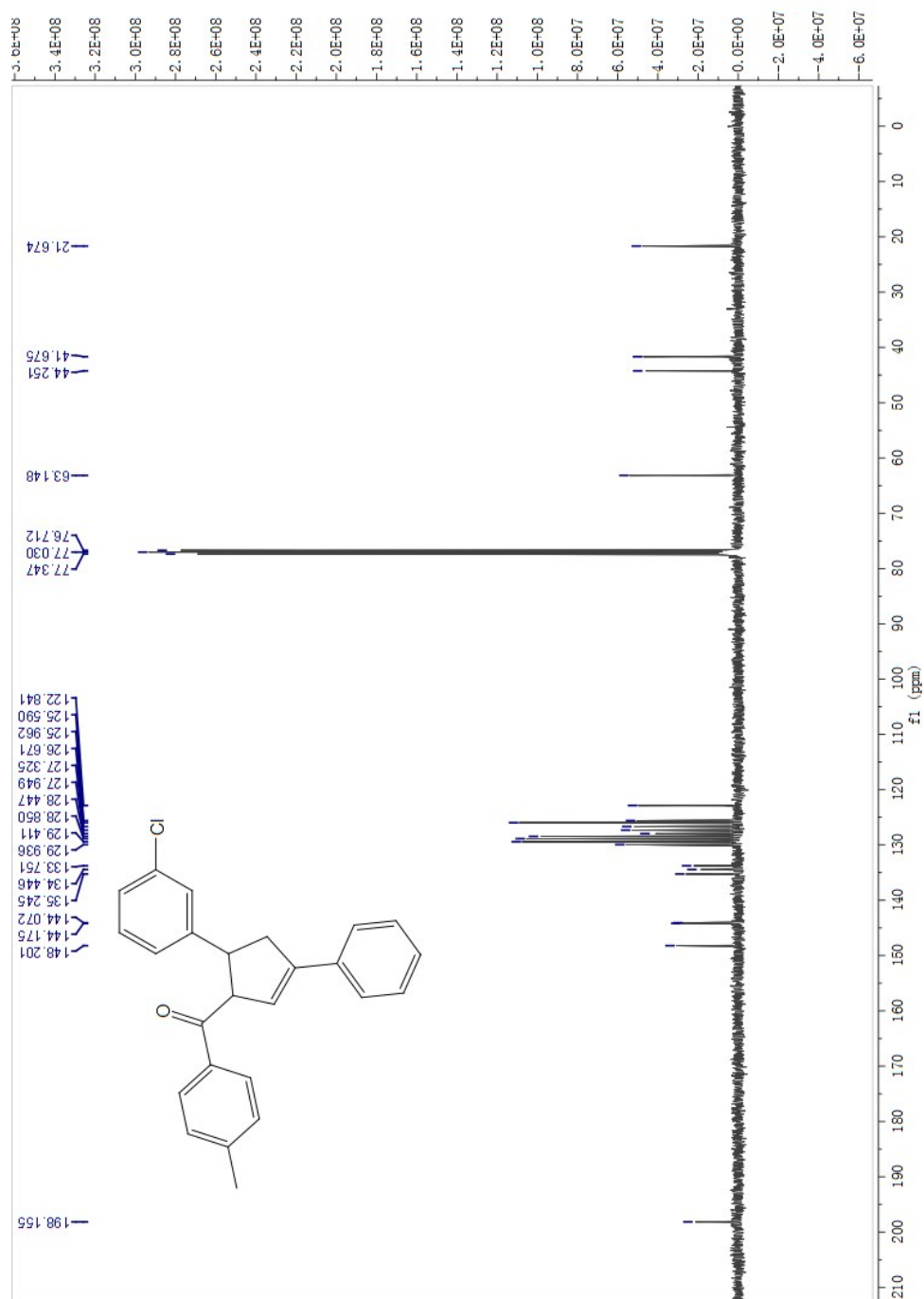
HR-MS Spectrum of (5-(4-fluorophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ac**



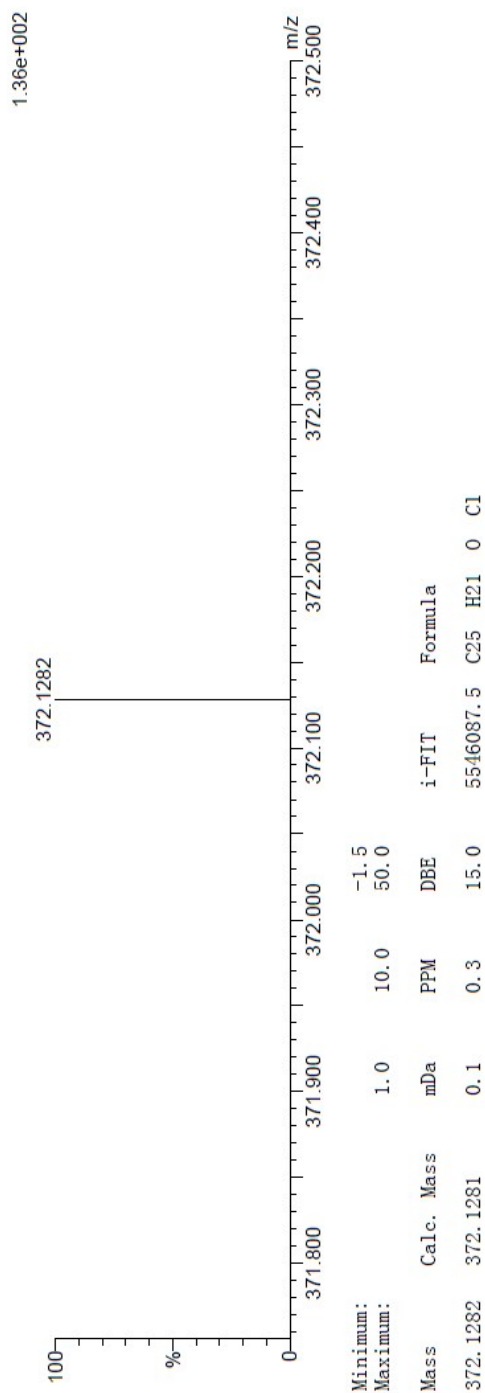
¹H NMR Spectrum of (5-(3-chlorophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ad**



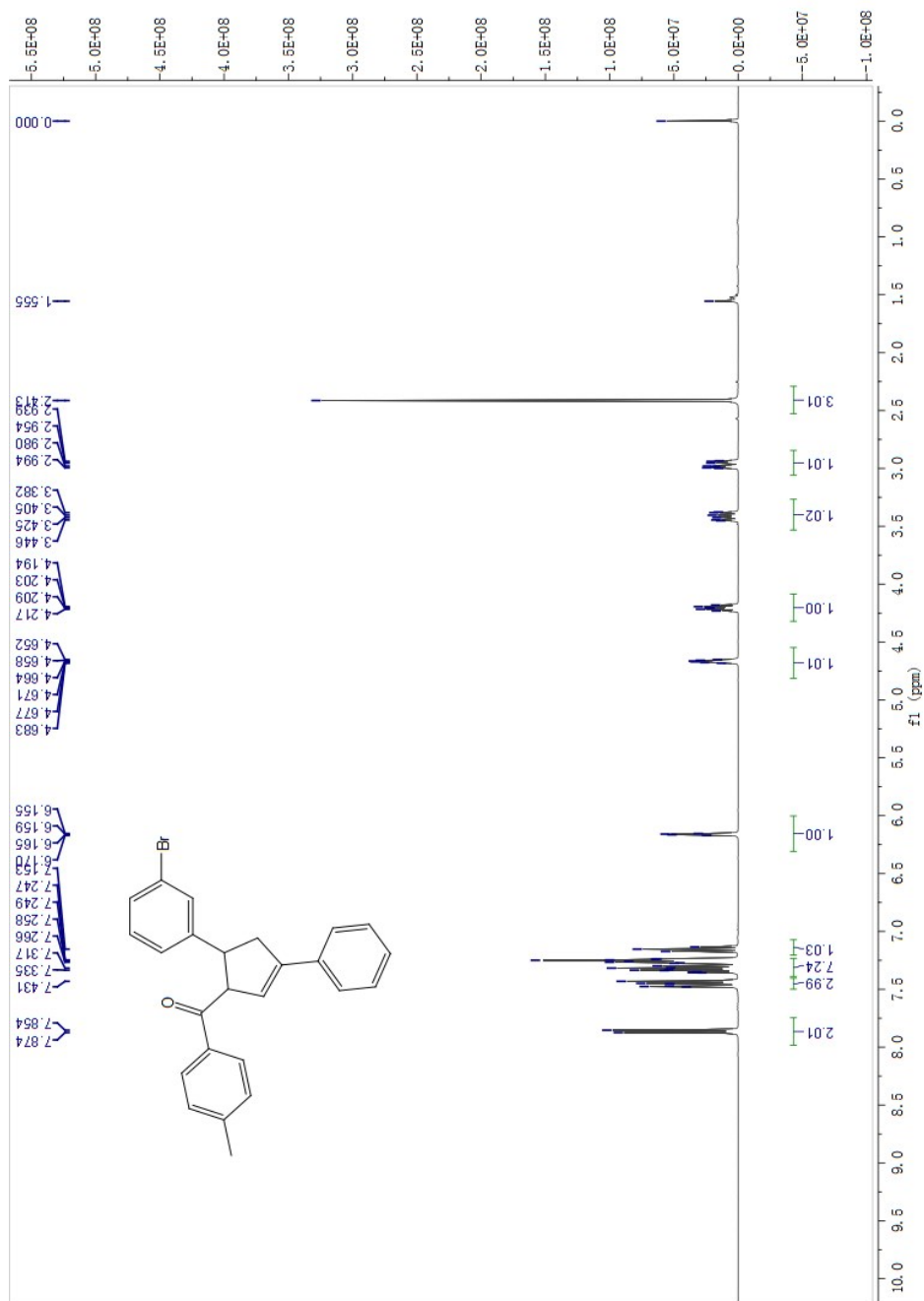
¹³C NMR Spectrum of (5-(3-chlorophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ad**



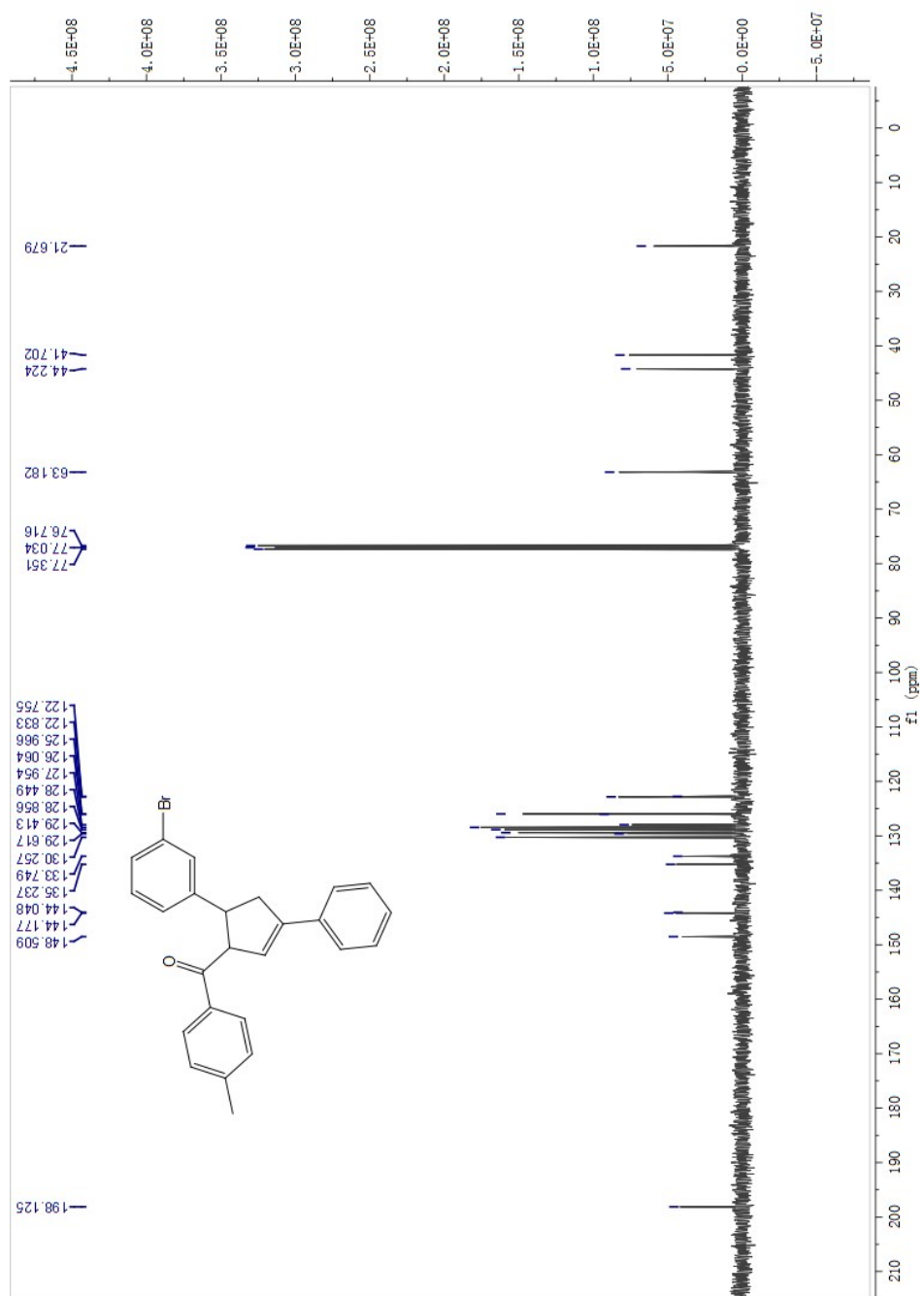
HR-MS Spectrum of (5-(3-chlorophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ad**



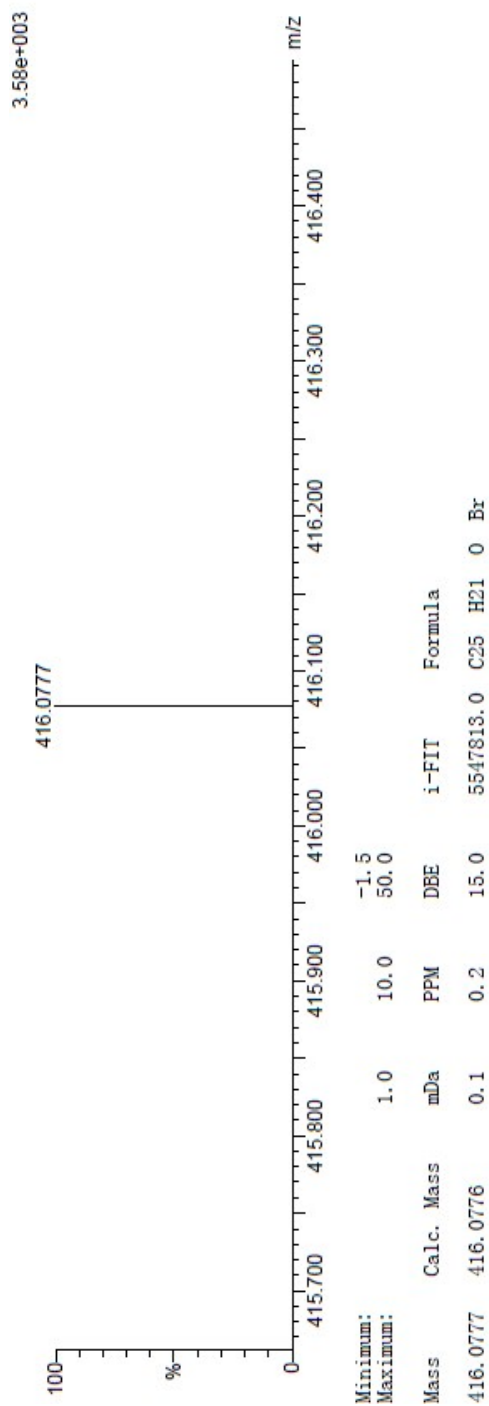
¹H NMR Spectrum of (5-(3-bromophenyl)-3-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ae**



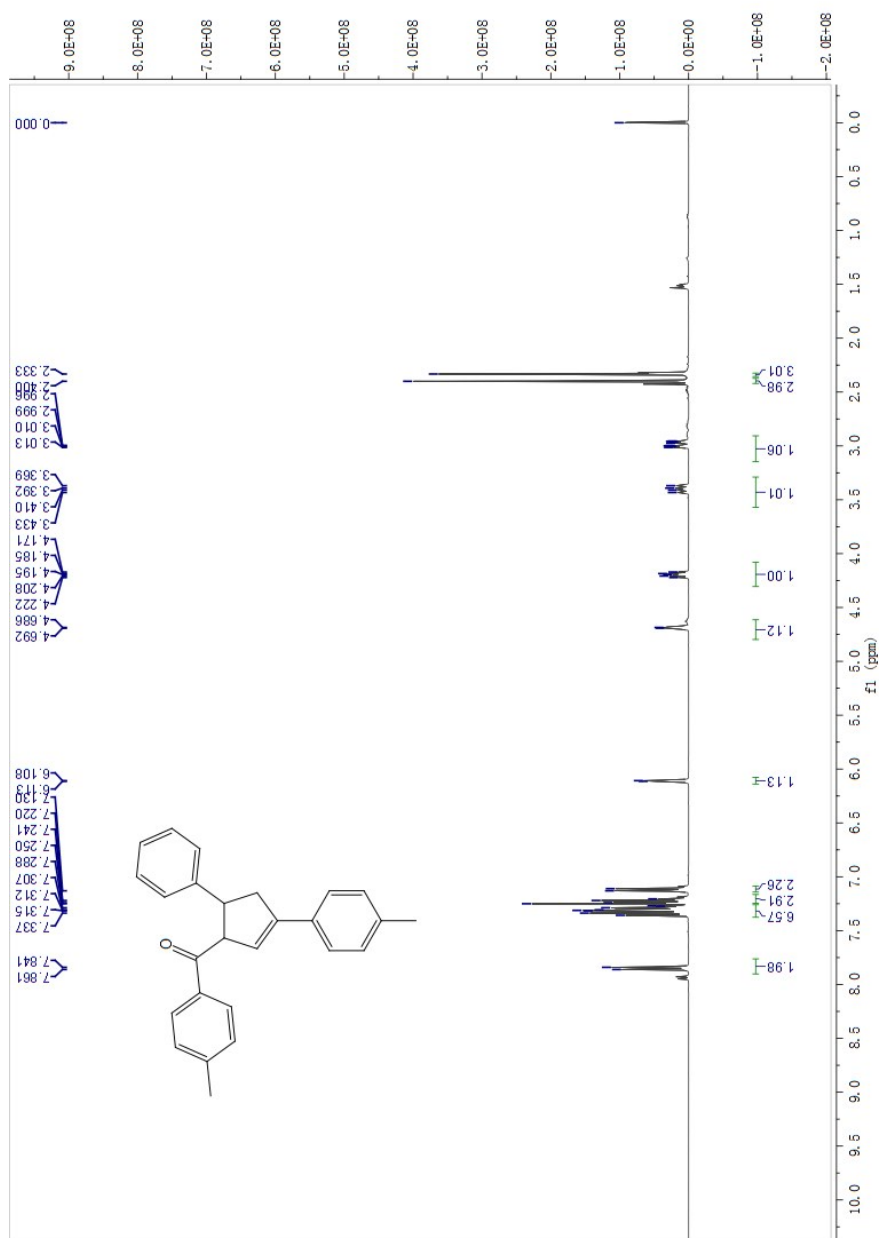
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(4-tolyl)methanone **3ae**



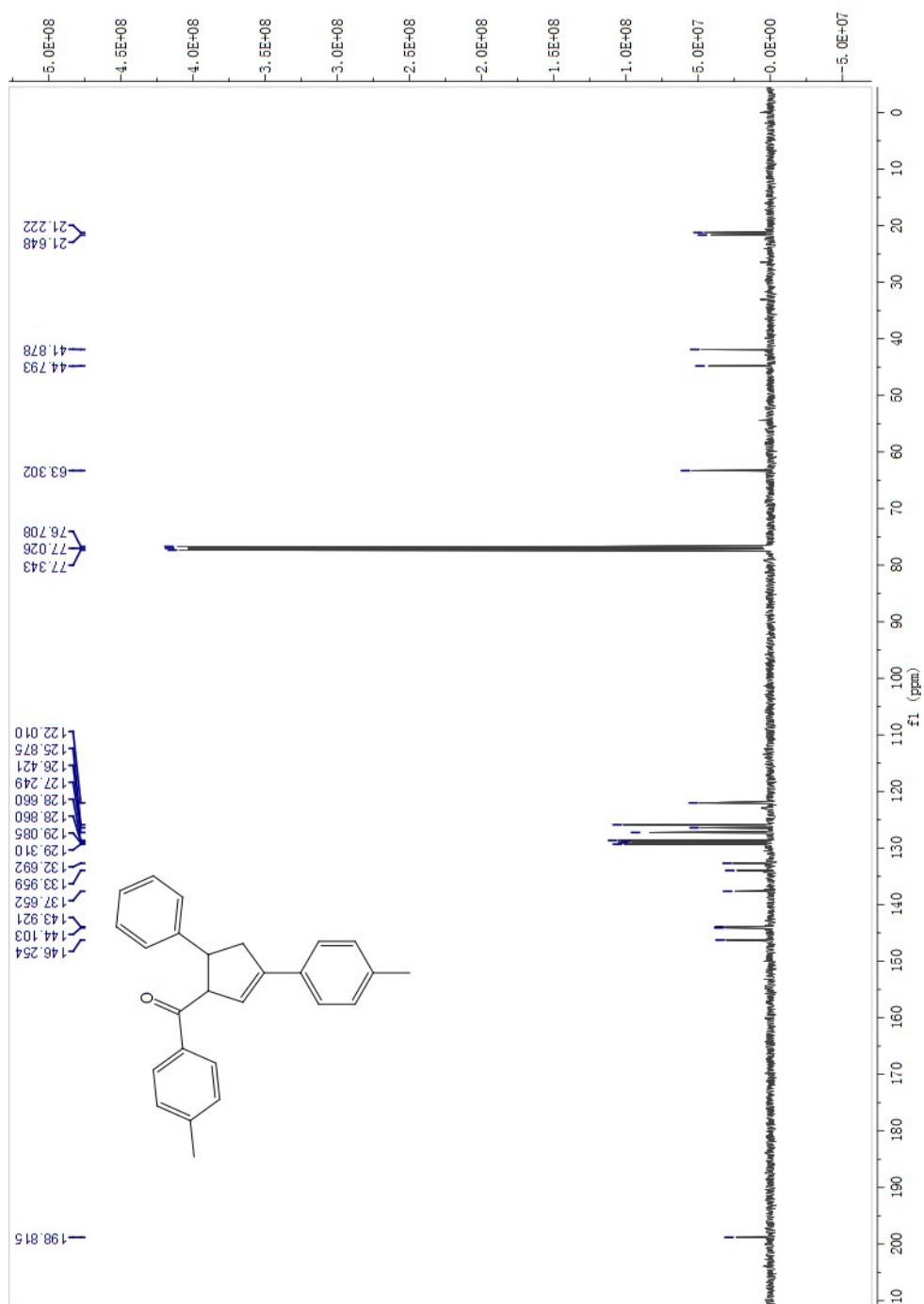
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(4-tolyl)methanone **3ae**



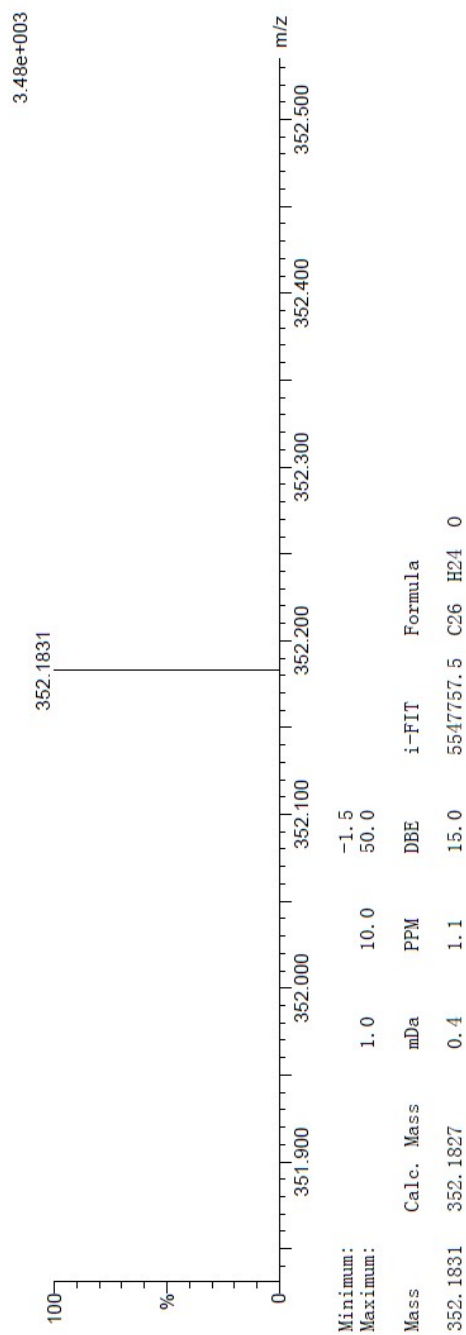
^1H NMR Spectrum of (5-phenyl-3-(4-tolyl)cyclopent-2-en-1-yl) (4-tolyl)methanone **3af**



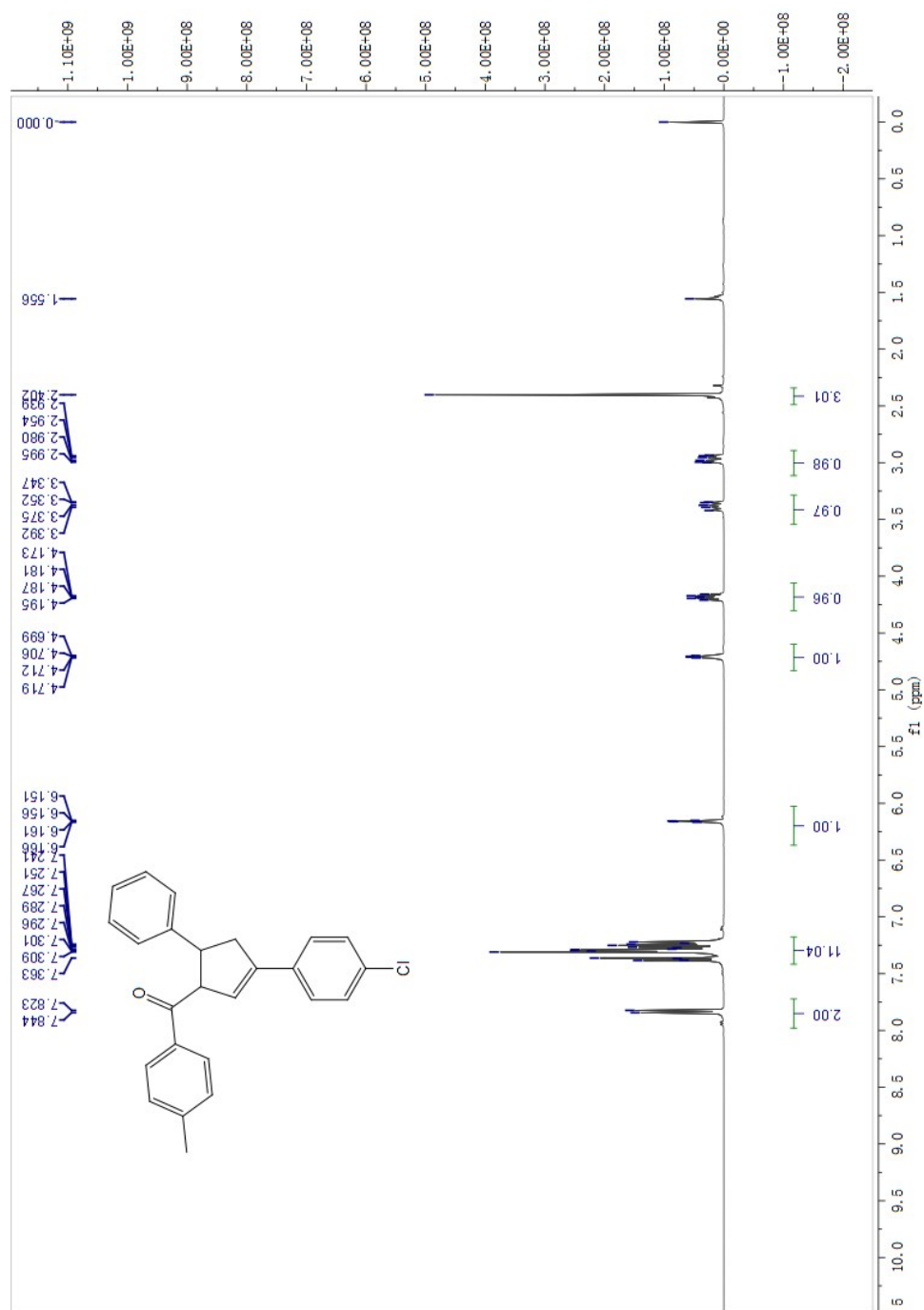
¹³C NMR Spectrum of (5-phenyl-3-(4-tolyl)cyclopent-2-en-1-yl) (4-tolyl)methanone **3af**



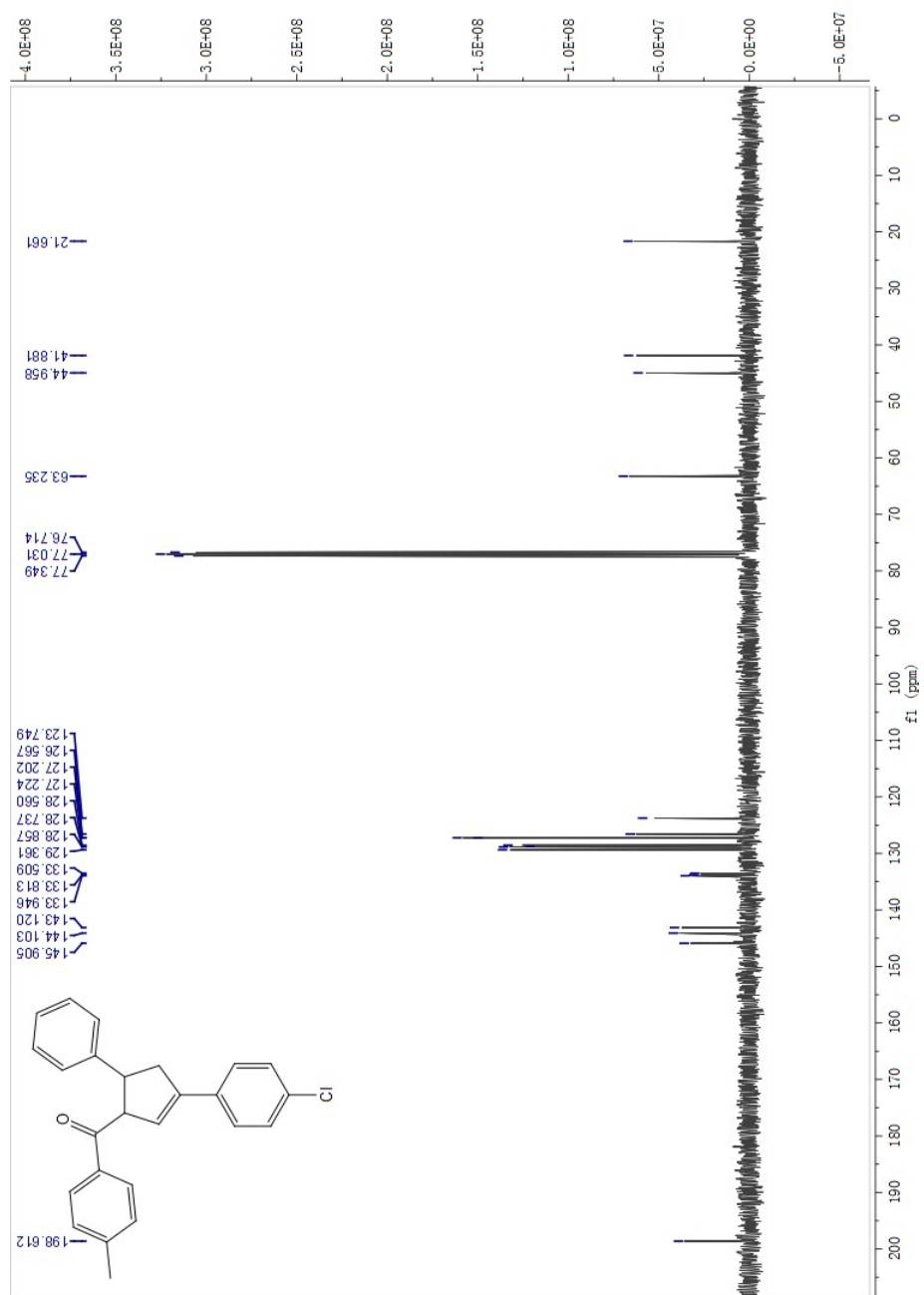
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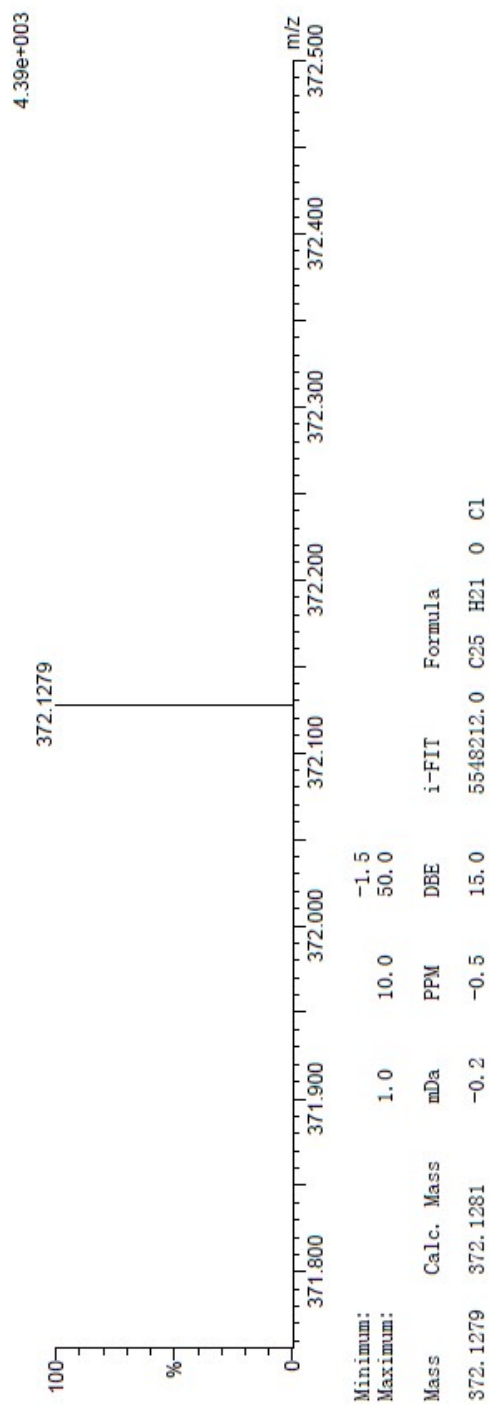
¹H NMR Spectrum of (3-(4-chlorophenyl)-5-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ag**



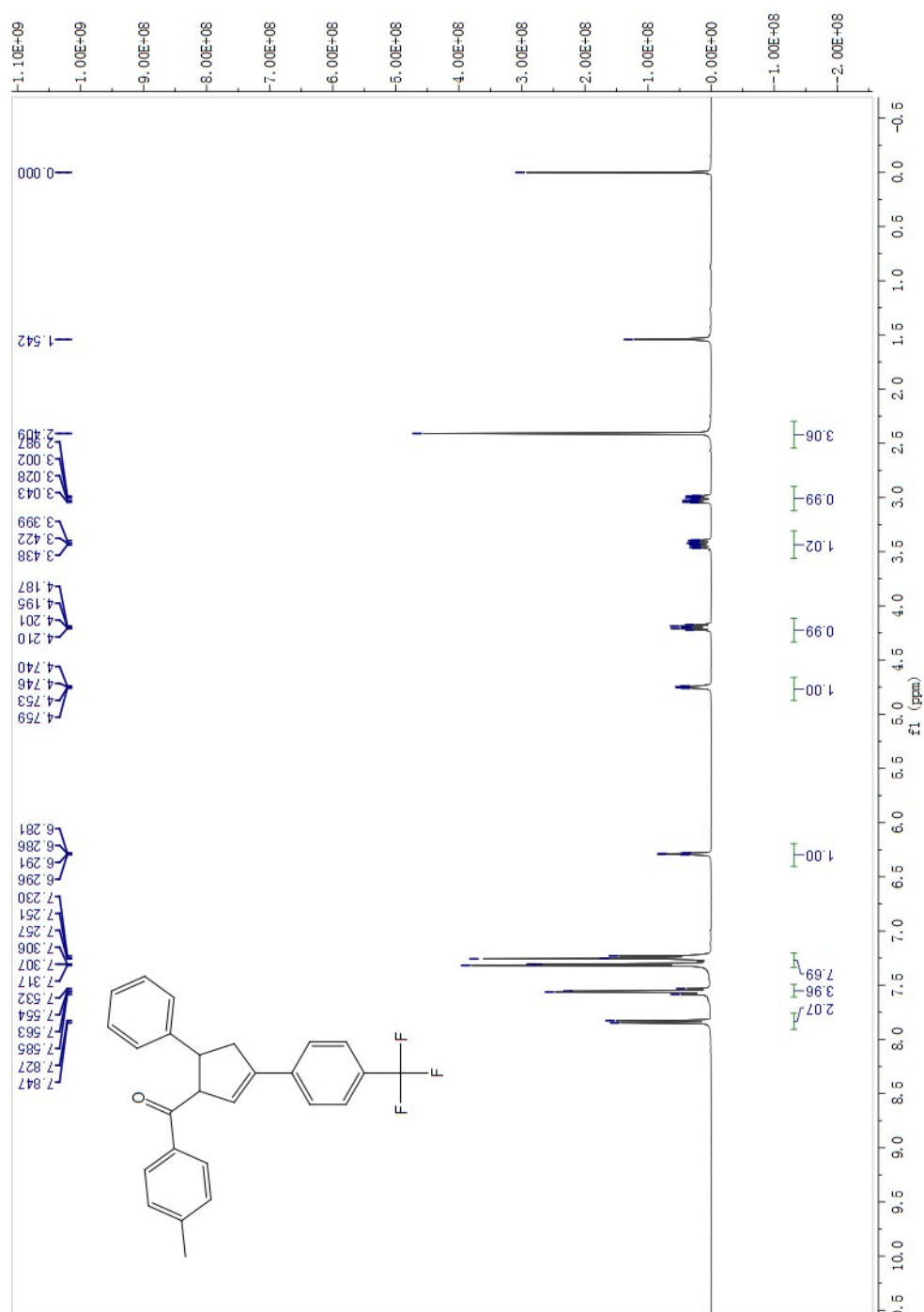
¹³C NMR Spectrum of (3-(4-chlorophenyl)-5-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ag**



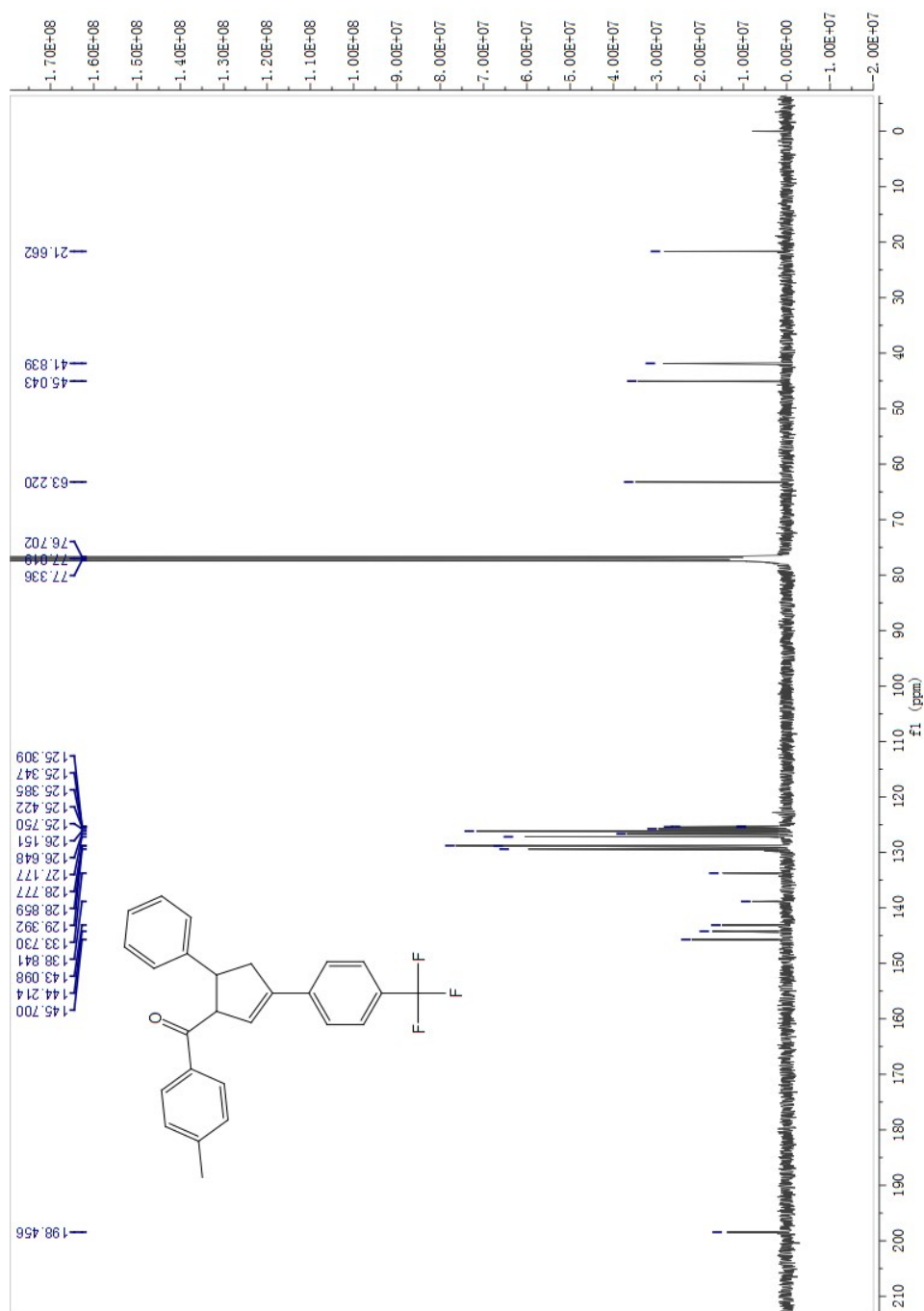
HR-MS Spectrum of (3-(4-chlorophenyl)-5-phenylcyclopent-2-en-1-yl)
(4-tolyl)methanone **3ag**



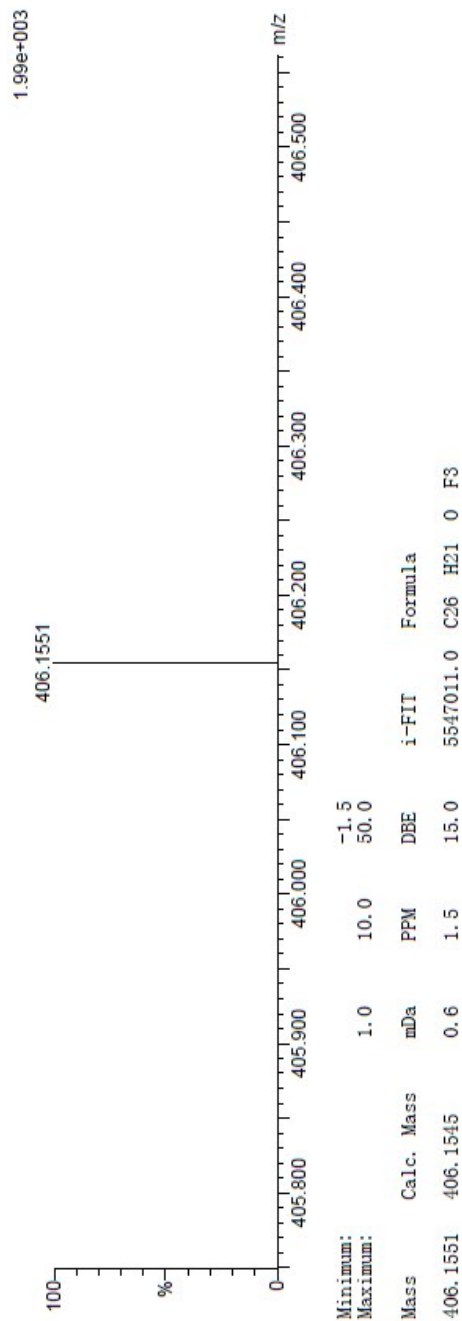
¹H NMR Spectrum of (5-phenyl-3-(4-(trifluoromethyl)phenyl) cyclopent-2-en-1-yl)(4-tolyl)methanone **3ah**



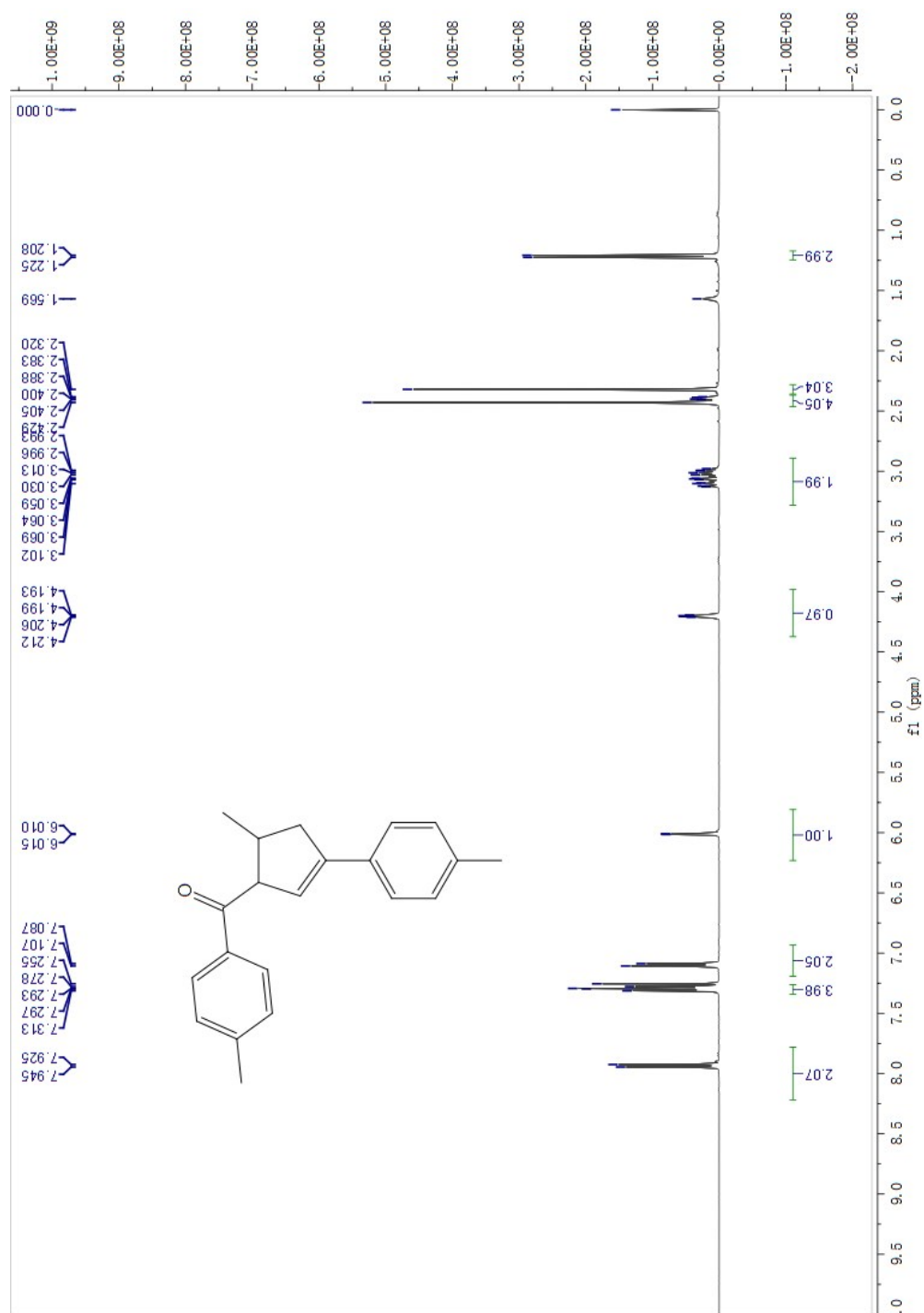
¹³C NMR Spectrum of (5-phenyl-3-(4-(trifluoromethyl)phenyl)cyclopent-2-en-1-yl)(4-tolyl)methanone **3ah**



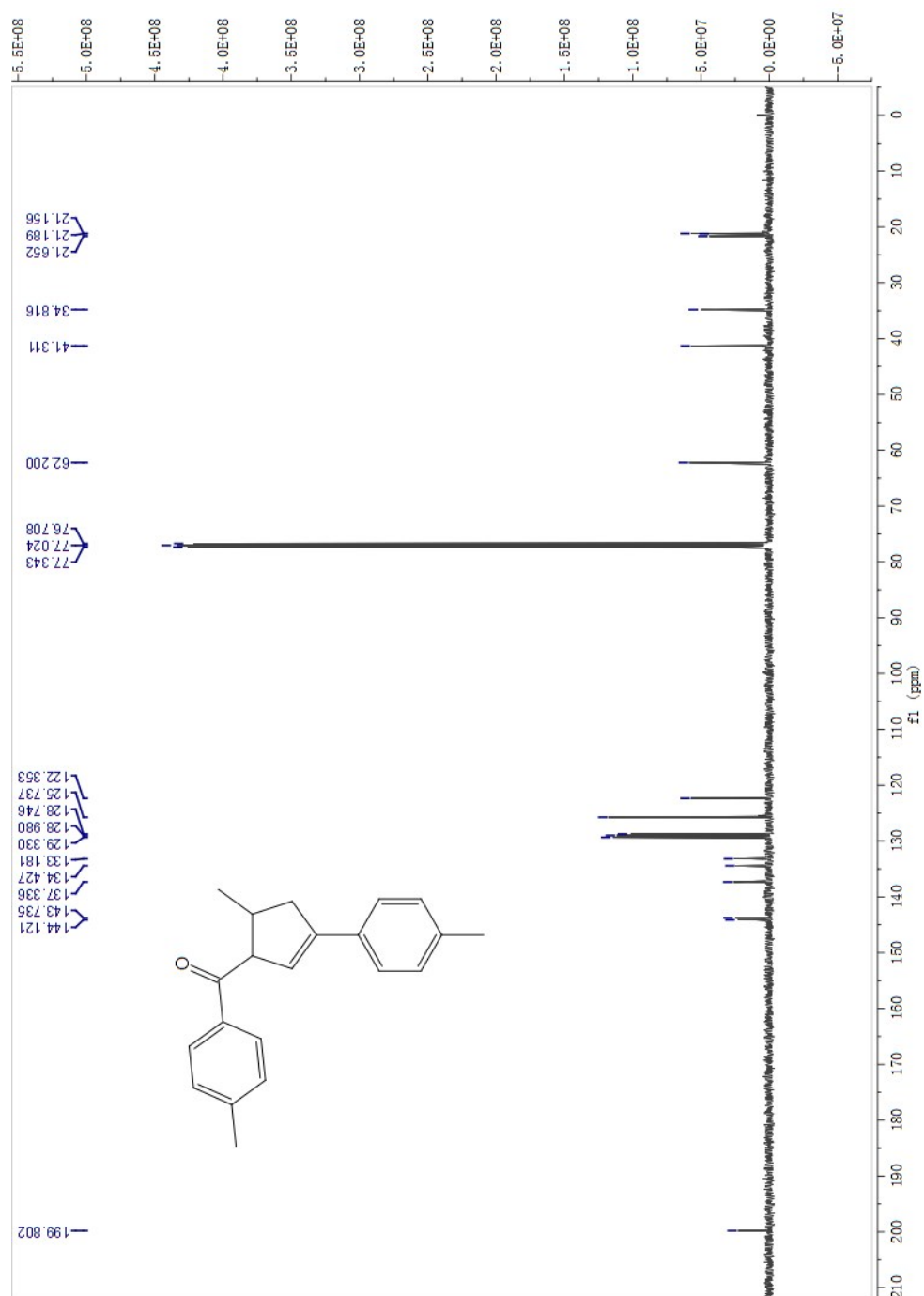
HR-MS Spectrum of (5-phenyl-3-(4-(trifluoromethyl)phenyl) cyclopent-2-en-1-yl)(4-tolyl)methanone **3ah**



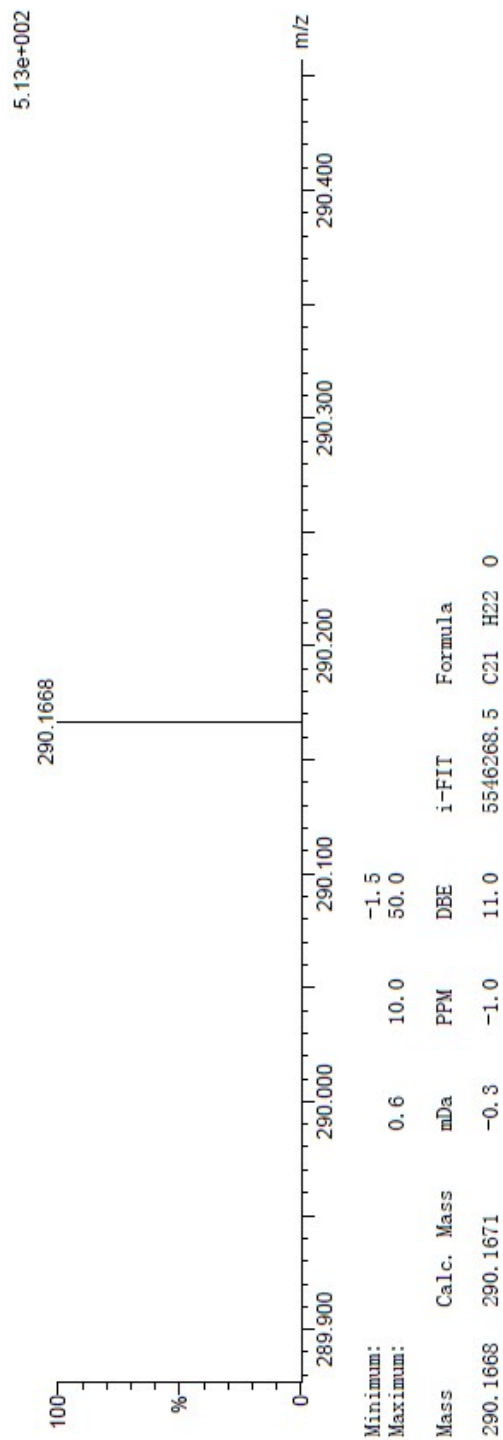
¹H NMR Spectrum of (5-methyl-3-(4-tolyl)cyclopent-2-en-1-yl) (4-tolyl)methanone **3ai**



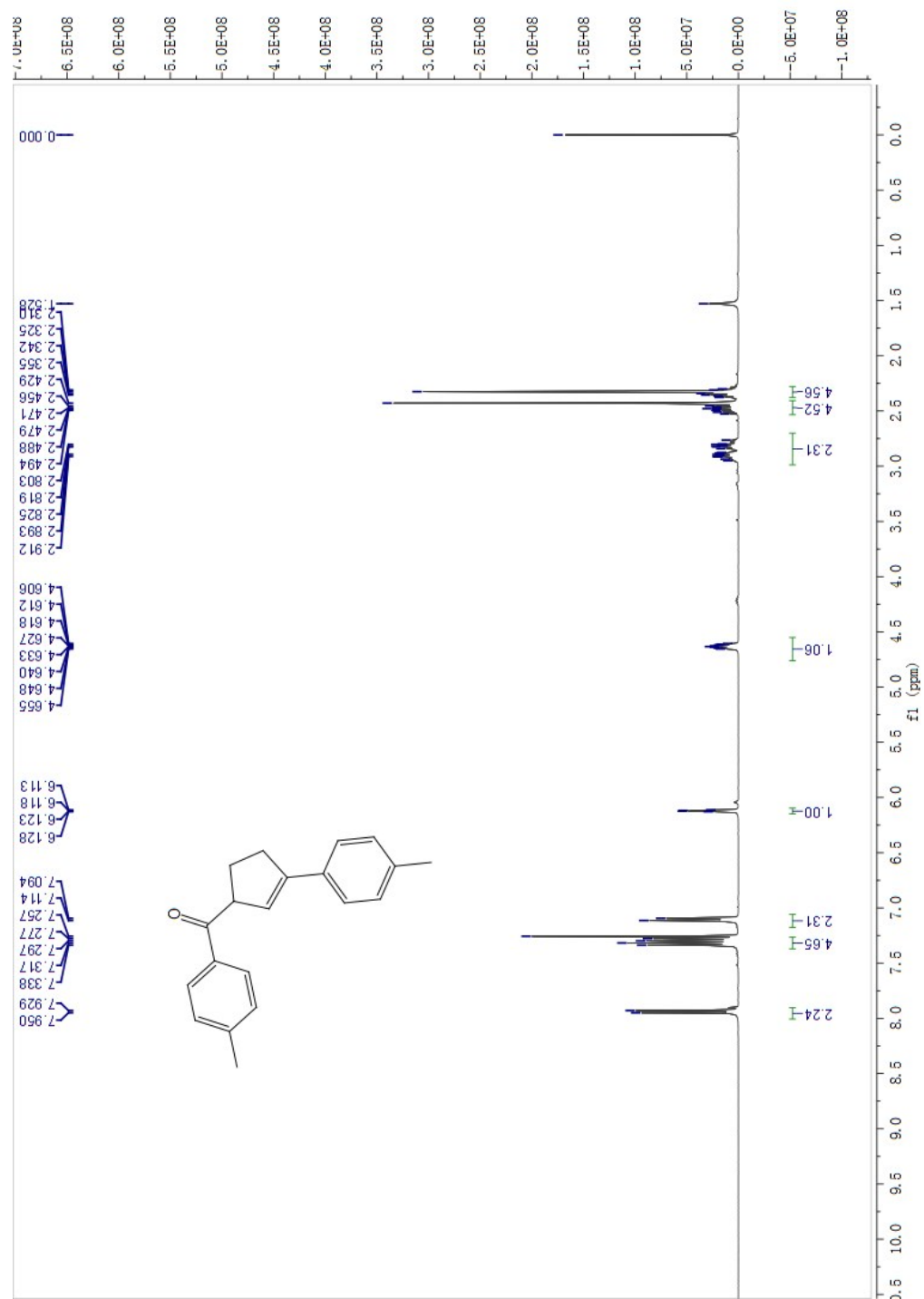
¹³C NMR Spectrum of (5-methyl-3-(4-tolyl)cyclopent-2-en-1-yl) (4-tolyl) methanone **3ai**



HR-MS Spectrum of (5-methyl-3-(4-tolyl)cyclopent-2-en-1-yl) (4-tolyl) methanone **3ai**

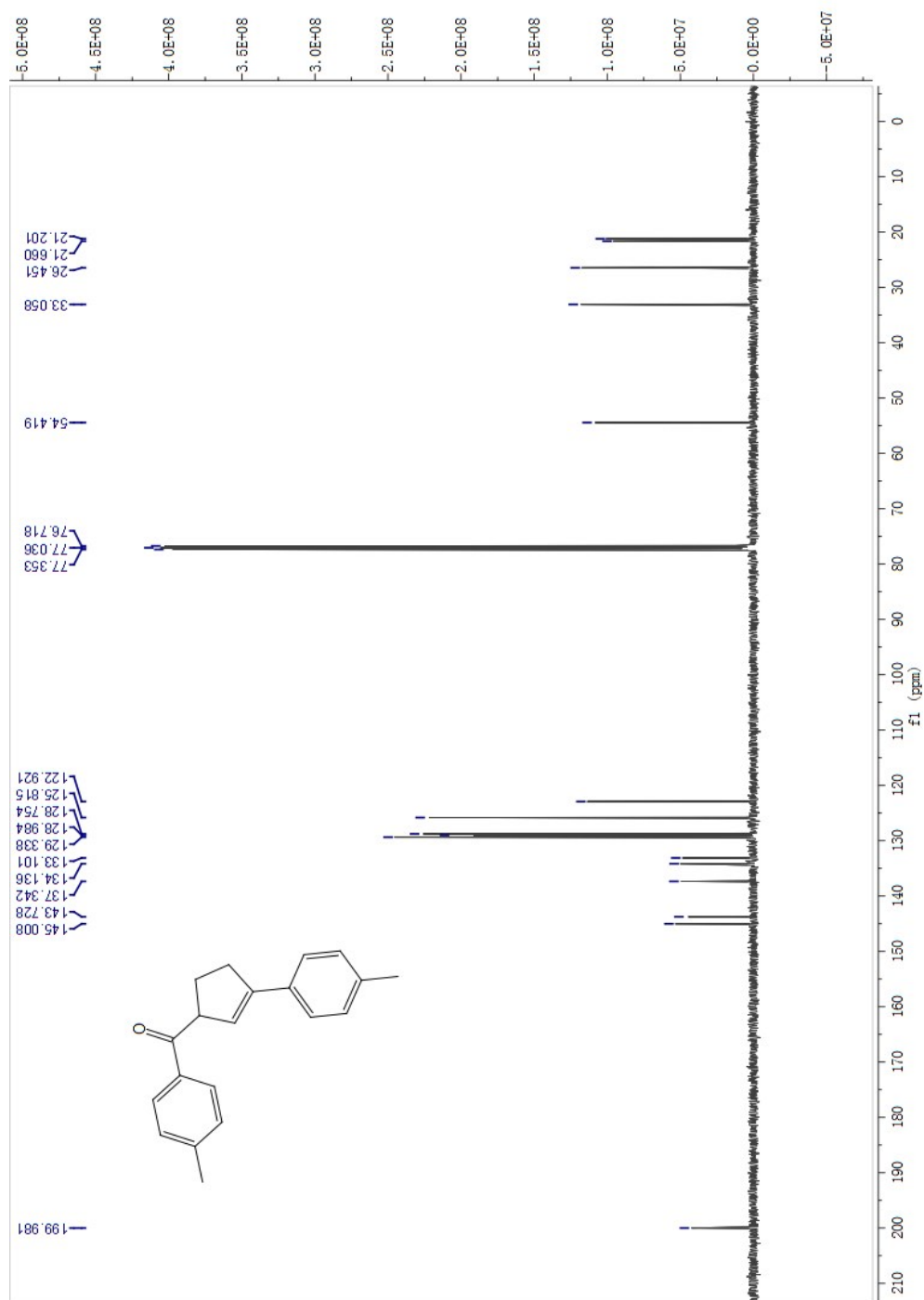


¹H NMR Spectrum of (4-tolyl(3-(4-tolyl)cyclopent-2-en-1-yl) methanone
4a

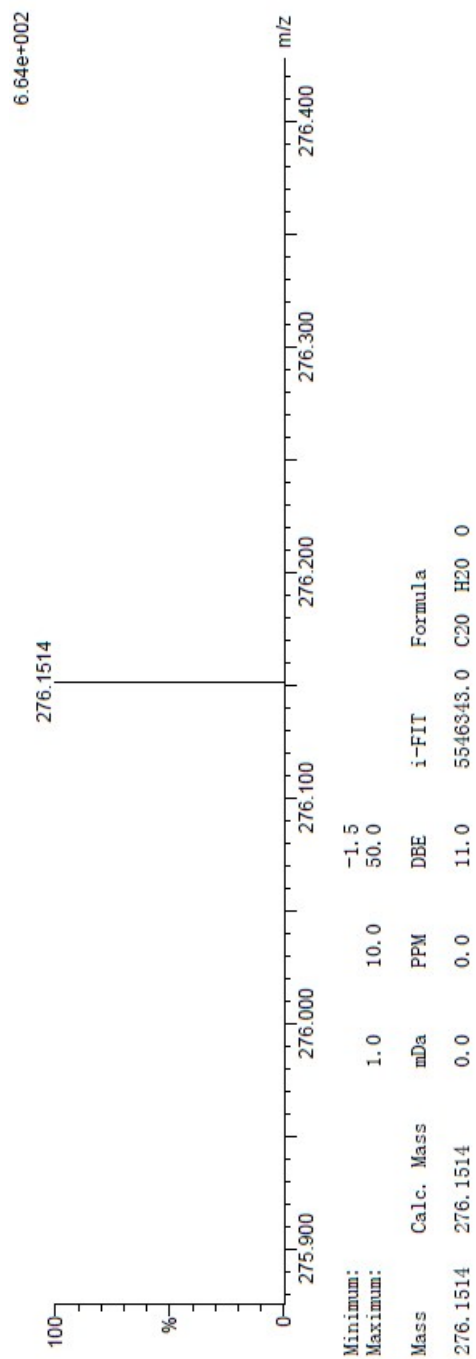


¹³C NMR Spectrum of (4-tolyl(3-(4-tolyl)cyclopent-2-en-1-yl)methanone

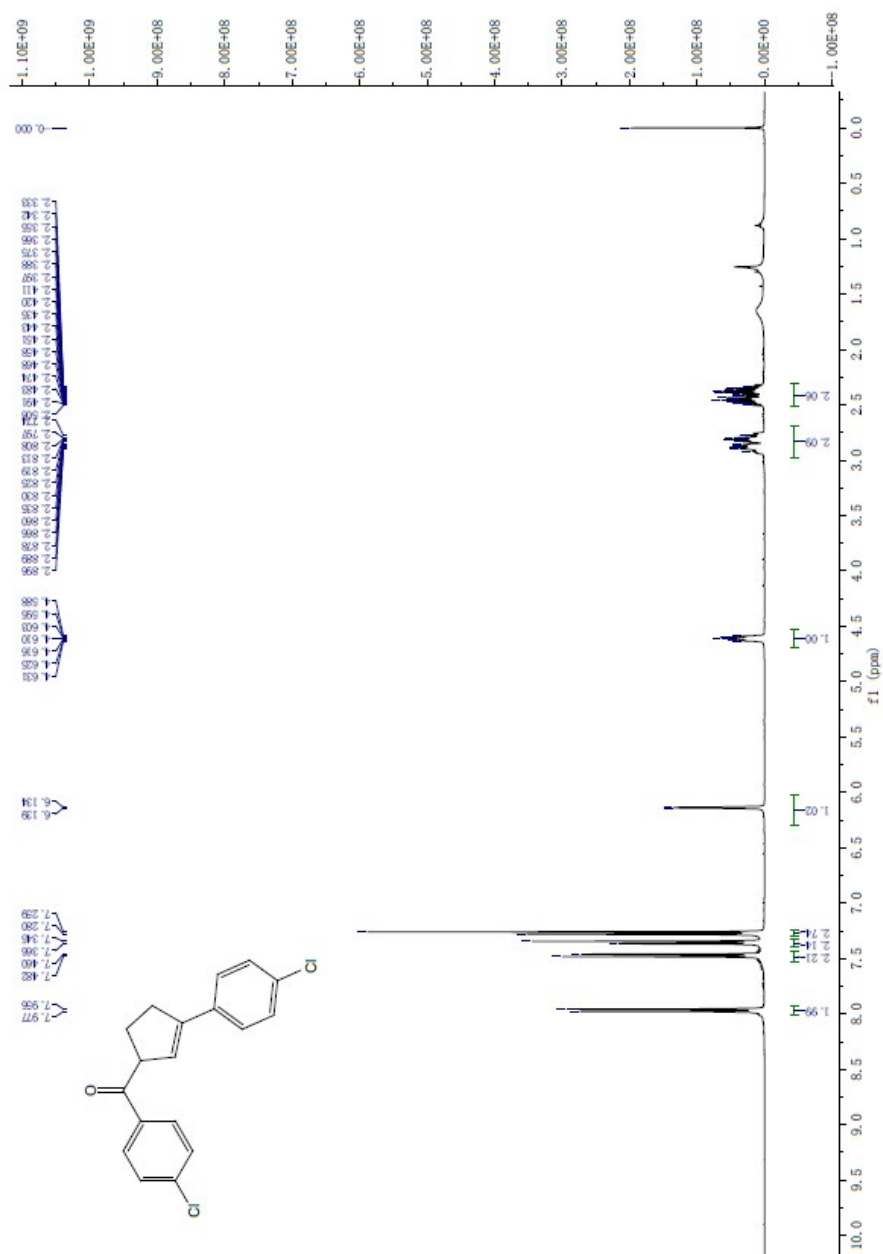
4a



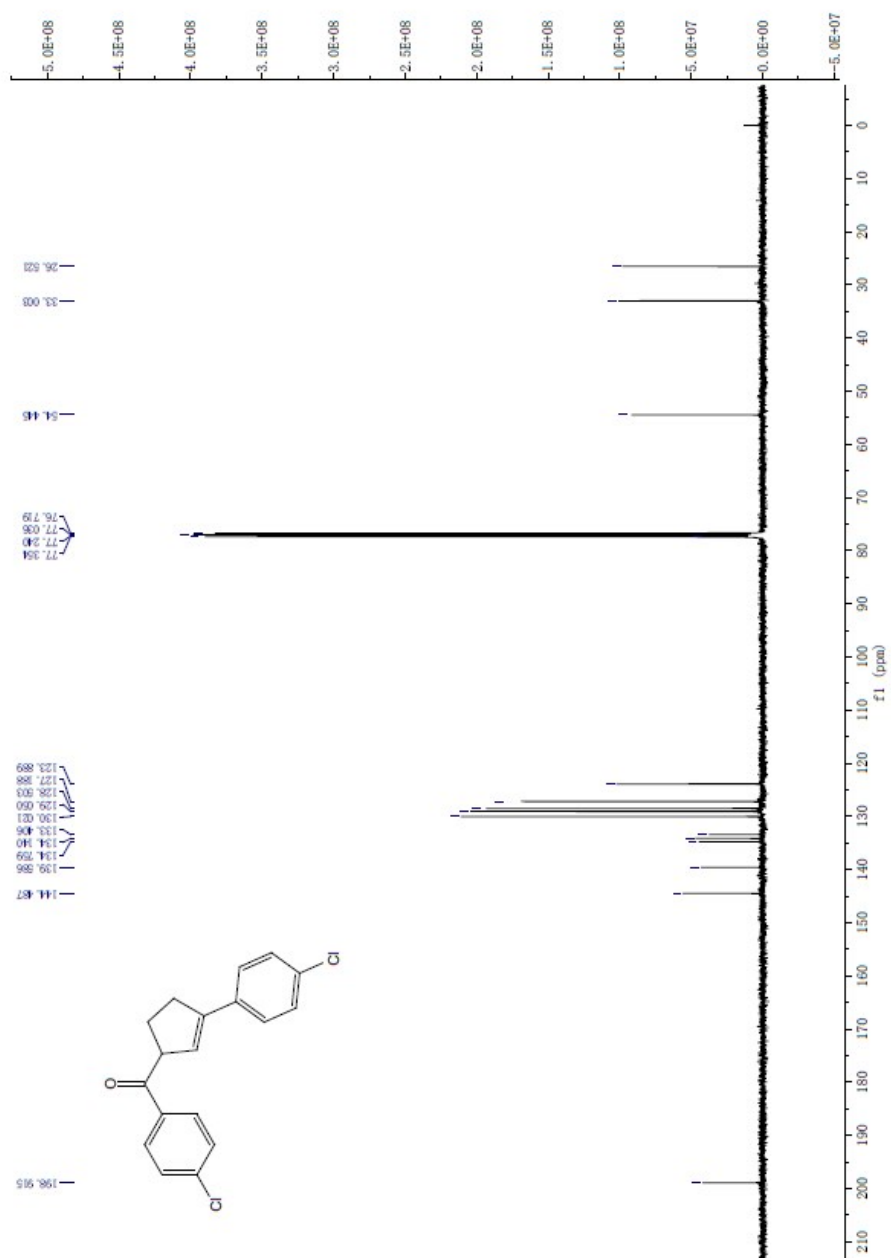
HR-MS Spectrum of (4-tolyl(3-(4-tolyl)cyclopent-2-en-1-yl)methanone
4a



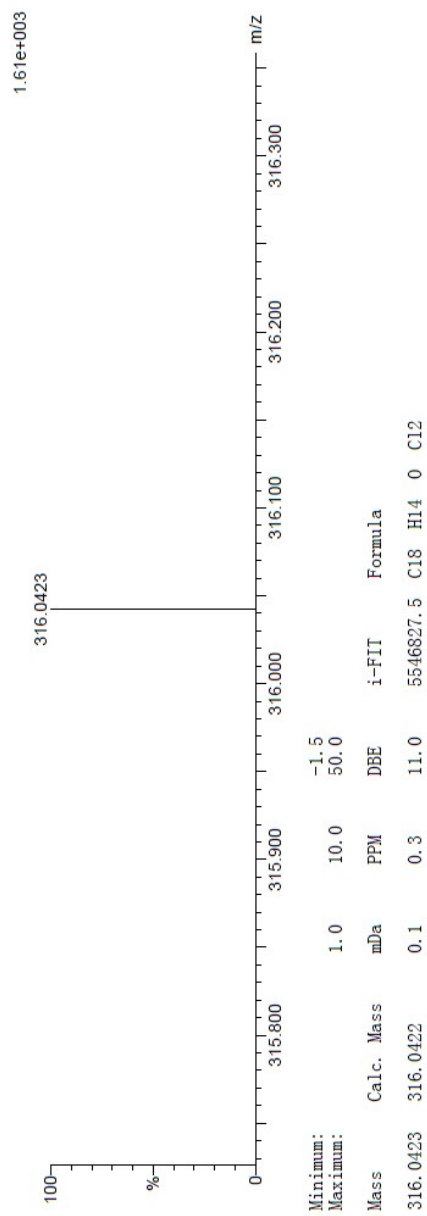
¹H NMR Spectrum of (4-chlorophenyl)(3-(4-chlorophenyl) cyclopent-2-en-1-yl)methanone **4d**



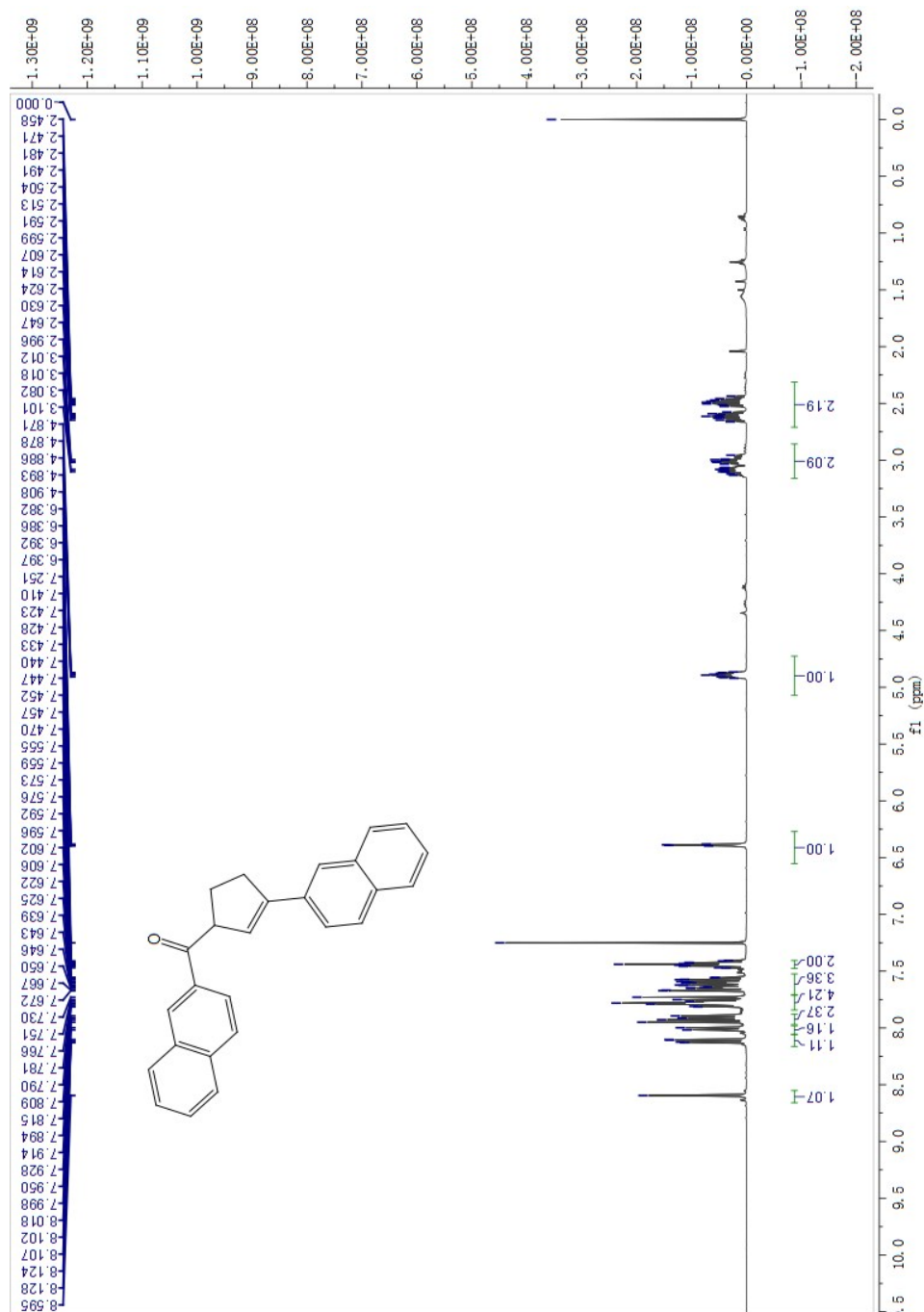
^{13}C NMR Spectrum of (4-chlorophenyl)(3-(4-chlorophenyl) cyclopent-2-en-1-yl)methanone **4d**



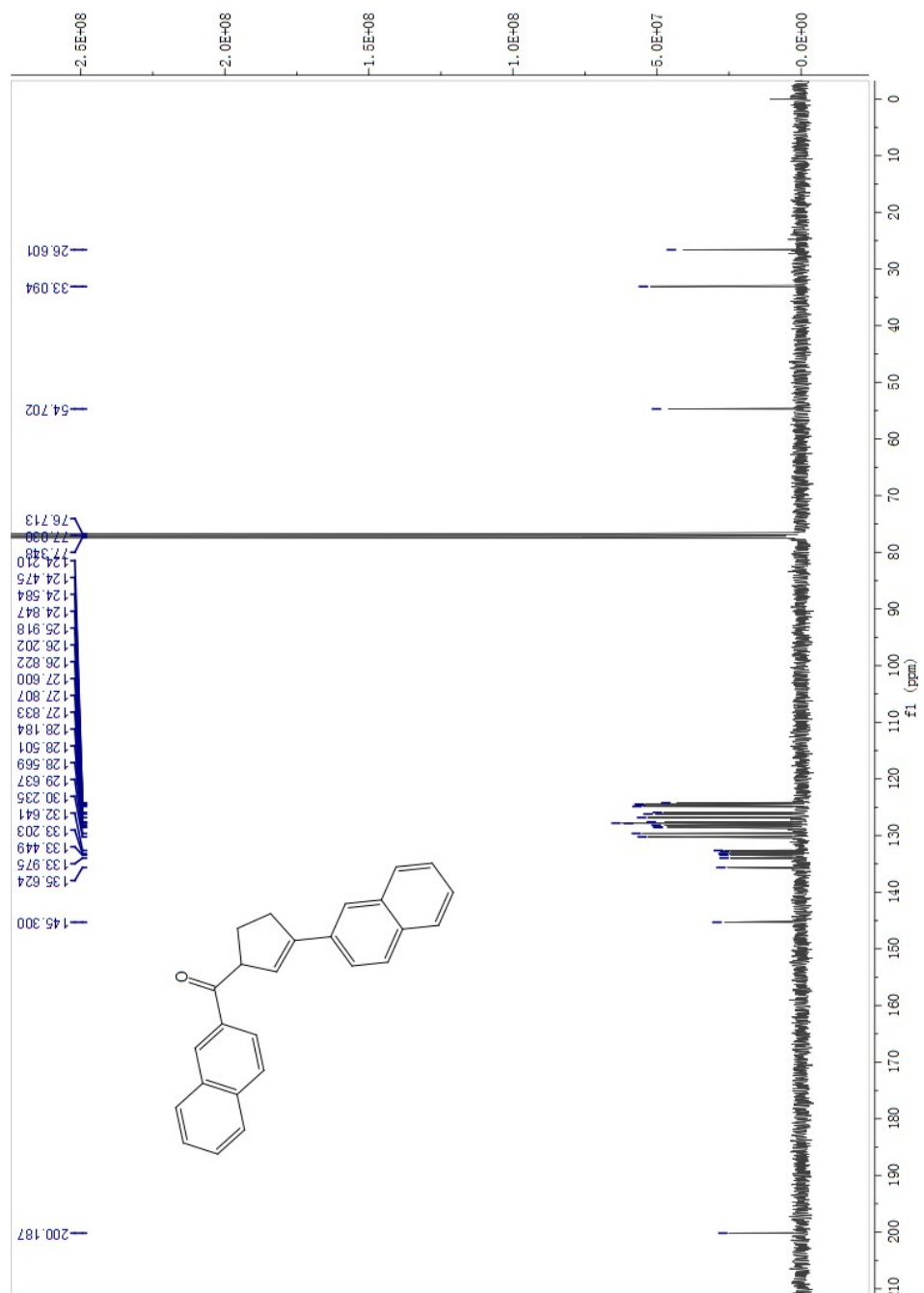
HR-MS Spectrum of (4-chlorophenyl)(3-(4-chlorophenyl) cyclopent-2-en-1-yl)methanone **4d**



¹H NMR Spectrum of naphthalen-2-yl(3-(naphthalen-2-yl) cyclopent-2-en-1-yl)methanone **4f**



¹³C NMR Spectrum of naphthalen-2-yl(3-(naphthalen-2-yl) cyclopent-2-en-1-yl)methanone **4f**



HR-MS Spectrum of naphthalen-2-yl(3-(naphthalen-2-yl) cyclopent-2-en-1-yl)methanone **4f**

