Supporting Information

Access to Cyclobutadienes via an Organocatalytic Dienamine-iminium-allenamine Cascade Approach

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A: General Information and Starting Materials

General Information. Proton nuclear magnetic resonance (^{1}H NMR) spectra and carbon nuclear magnetic resonance (^{13}C NMR) spectra were recorded on a Bruker ACF300 spectrometer (500 MHz and 125 MHz). Chemical shifts for protons are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CDCl_3$: δ 7.26). Chemical shifts for carbon are reported in parts per million downfield from tetramethylsilane and are referenced to the carbon resonances of the solvent ($CDCl_3$: δ 77.16). Data are represented as follows: chemical shift, integration, multiplicity (br = broad, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants in Hertz (Hz). All high resolution mass spectra were obtained on a Finnigan/MAT 95XL-T mass spectrometer. For thin layer chromatography (TLC), Merck pre-coated TLC plates (Merck 60 F254) were used, and compounds were visualized with a UV light at 254 nm. Flash chromatography separations were performed on Merck 60 (0.040-0.063 mm) mesh silica gel.

Starting Materials. All solvents and inorganic reagents were from commercial sources and used without purification unless otherwise noted. Alkyne aldehydes were prepared following the literature procedures.[1]

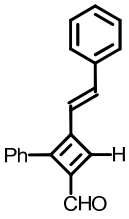
B: General Procedure for Cascade Reactions

To a solution of toluene (0.3 mL) was added α,β-unsaturated aldehydes **1** (0.15 mmol), alkyne aldehydes **2** (0.10 mmol), catalyst (0.02 mmol) and K_2CO_3 (0.02 mmol). The reaction mixture was stirred at room temperature for 72 h and then the solvent was removed under vacuum. The residue was purified by silica gel chromatography (hexane/ethyl acetate = 30:1 to 20:1) to yield the desired product.

The procedure for the synthesis of **3aa** in 1.0 mmol scale: To a solution of toluene (3.0 mL) was added **1a** (1.5 mmol), **2a** (1.0 mmol), catalyst **V** (0.2 mmol) and K_2CO_3 (0.2 mmol). The reaction mixture was stirred at room temperature for 72 h and then the solvent was removed under vacuum. The residue was purified by silica gel chromatography (hexane/ethyl acetate = 30:1 to 20:1) to yield the desired product **3aa** (214 mg, 83%) as yellow oil.

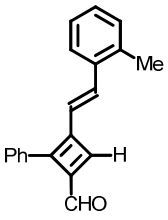
C: Characterization Data of Cascade Product

(*E*)-2-phenyl-3-styrylcyclobuta-1,3-dienecarbaldehyde (3aa)



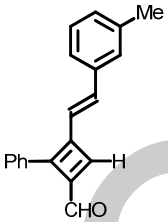
Yellow oil, 22.4 mg, 87% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.66 (s, 1H), 8.01-7.92 (m, 2H), 7.61-7.55 (m, 3H), 7.43-7.42 (m, 1H), 7.40-7.37 (m, 1H), 7.33-7.30 (m, 1H), 7.25-7.24 (m, 2H), 7.16-7.15 (m, 2H), 6.52 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 143.3, 137.2, 136.4, 131.9, 129.8, 128.8, 128.7, 127.2, 127.0, 122.3, 119.5, 109.6, 86.9. HRMS (EI): exact mass calculated for M ($C_{19}H_{14}O$) requires m/z 258.1045, found m/z 258.1039.

(*E*)-3-(2-methylstyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3ba)



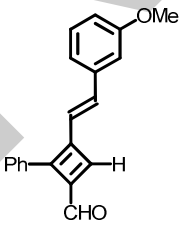
Yellow oil, 22.6 mg, 83% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.69 (s, 1H), 8.27 (d, *J* = 16.5 HMz, 1H), 7.67-7.65 (m, 1H), 7.57-7.56 (m, 2H), 7.43-7.42 (m, 3H), 7.26-7.21 (m, 3H), 7.19-7.16 (m, 1H), 6.56 (s, 1H), 2.44 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 143.5, 136.8, 136.3, 134.2, 131.9, 130.5, 129.7, 128.6, 128.5, 127.1, 126.2, 125.3, 122.2, 120.5, 109.5, 86.9, 19.9. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O$) requires m/z 272.1201, found m/z 272.1195.

(*E*)-3-(3-methylstyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3ca)



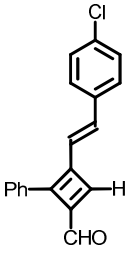
Yellow oil, 22.8 mg, 84% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.67 (s, 1H), 7.99 (d, *J* = 16.5 HMz, 1H), 7.60-7.59 (m, 2H), 7.45-7.42 (m, 3H), 7.39-7.38 (m, 2H), 7.31-7.29 (m, 1H), 7.28-7.24 (m, 1H), 7.16-7.15 (m, 1H), 6.53 (s, 1H), 2.41 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 143.4, 138.3, 137.1, 136.5, 131.9, 129.8, 129.6, 128.7, 128.6, 127.8, 126.9, 124.1, 122.3, 119.3, 109.5, 87.0, 21.4. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O$) requires m/z 272.1201, found m/z 272.1196.

(*E*)-3-(3-methoxystyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3da)



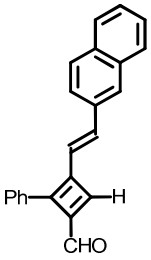
Yellow oil, 26.5 mg, 92% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.68 (s, 1H), 7.99 (d, *J* = 16.5 HMz, 1H), 7.60-7.58 (m, 2H), 7.45-7.43 (m, 3H), 7.34-7.30 (m, 1H), 7.28-7.24 (m, 1H), 7.19-7.18 (m, 1H), 7.11 (s, 1H), 6.91-6.89 (m, 1H), 6.55 (s, 1H), 3.87 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 159.9, 143.2, 138.6, 136.2, 131.9, 129.8, 129.7, 128.7, 127.4, 122.3, 119.7, 119.6, 114.6, 112.1, 109.7, 86.9, 55.2. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O_2$) requires m/z 288.1150, found m/z 288.1142.

(*E*)-3-(4-chlorostyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3ea)



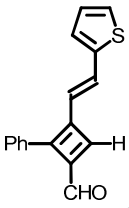
Yellow oil, 24.8 mg, 85% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.67 (s, 1H), 7.97 (d, *J* = 16.5 HMz, 1H), 7.59-7.57 (m, 2H), 7.50-7.49 (m, 2H), 7.46-7.42 (m, 2H), 7.38-7.36 (m, 2H), 7.24-7.21 (m, 2H), 6.56 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.2, 143.0, 135.7, 135.0, 134.4, 131.9, 129.9, 129.0, 128.7, 128.1, 127.8, 122.2, 120.0, 109.9, 86.7. HRMS (EI): exact mass calculated for M ($C_{19}H_{13}OCl$) requires m/z 292.0655, found m/z 292.0652.

(*E*)-3-(2-(naphthalen-2-yl)vinyl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3fa)



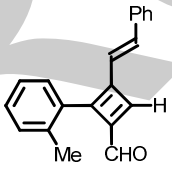
Yellow oil, 27.4 mg, 89% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.71 (s, 1H), 8.20 (d, *J* = 16.5 HMz, 1H), 7.94 (s, 1H), 7.88-7.84 (m, 3H), 7.80-7.78 (m, 1H), 7.64-7.62 (m, 2H), 7.51-7.46 (m, 5H), 7.41-7.38 (m, 1H), 6.57 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 143.3, 136.4, 134.6, 133.6, 131.9, 129.8, 128.7, 128.5, 128.3, 128.1, 127.7, 126.4, 123.2, 122.3, 119.8, 109.7, 87.0. HRMS (EI): exact mass calculated for M ($C_{23}H_{16}O$) requires m/z 308.1201, found m/z 308.1203.

(*E*)-2-phenyl-3-(2-(thiophen-2-yl)vinyl)cyclobuta-1,3-dienecarbaldehyde (3ga)



Yellow oil, 23.0 mg, 87% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.64 (s, 1H), 8.16 (d, *J* = 16.5 HMz, 1H), 7.61-7.60 (m, 2H), 7.45-7.44 (m, 3H), 7.32-7.30 (m, 1H), 7.19-7.18 (m, 1H), 7.13-7.10 (m, 1H), 7.07-7.05 (m, 1H), 6.48 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.2, 143.1, 143.0, 131.9, 129.8, 129.4, 128.7, 128.2, 127.9, 127.0, 126.1, 122.3, 119.1, 110.0, 86.9. HRMS (EI): exact mass calculated for M ($C_{17}H_{12}OS$) requires m/z 264.0609, found m/z 264.0603.

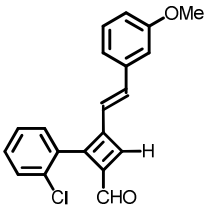
(*E*)-3-styryl-2-o-tolylcyclobuta-1,3-dienecarbaldehyde (3ab)



Yellow oil, 23.7 mg, 87% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.70 (s, 1H), 8.02 (d, *J* = 16.5 HMz, 1H), 7.57-7.56 (m, 3H), 7.41-7.38 (m, 2H), 7.35-7.29 (m, 3H), 7.27-7.24 (m, 2H), 6.60 (s, 1H), 2.58 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.2, 143.0, 140.7, 137.2, 136.2, 132.5, 129.9, 129.8, 128.7, 128.6, 127.7, 127.1, 125.9, 122.2, 119.7, 108.8, 90.6, 20.9. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O$) requires m/z 272.1201, found m/z 272.1196.

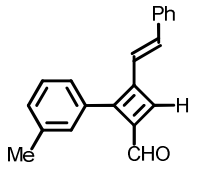
(*E*)-3-(3-methoxystyryl)-2-(2-chlorophenyl)cyclobuta-1,3-dienecarbaldehyde

(3ac)



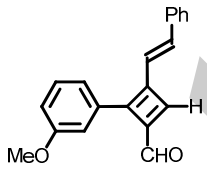
Yellow oil, 26.4 mg, 82% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.69 (s, 1H), 8.00-7.97 (m, 1H), 7.60-7.58 (m, 1H), 7.49-7.48 (m, 1H), 7.37-7.27 (m, 4H), 7.17-7.16 (m, 1H), 7.09 (s, 1H), 6.88-6.86 (m, 1H), 6.54 (s, 1H), 3.85 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.2, 159.9, 143.9, 138.6, 136.7, 136.4, 133.7, 130.7, 129.7, 129.6, 126.8, 126.6, 120.1, 119.9, 114.9, 111.9, 91.4, 55.3. HRMS (EI): exact mass calculated for M ($C_{20}H_{15}O_2Cl$) requires m/z 322.0761, found m/z 322.0756.

(*E*)-3-styryl-2-m-tolylcyclobuta-1,3-dienecarbaldehyde (3ad)



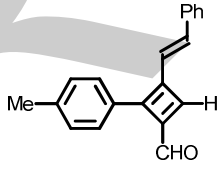
Yellow oil, 22.8 mg, 84% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.67 (s, 1H), 8.02 (d, *J* = 16.5 HMz, 1H), 7.59-7.58 (m, 2H), 7.41-7.39 (m, 4H), 7.34-7.31 (m, 2H), 7.28-7.26 (m, 2H), 6.54 (s, 1H), 2.41 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 143.2, 138.5, 137.2, 136.2, 132.4, 130.7, 129.0, 128.7, 128.6, 128.5, 127.3, 127.0, 122.1, 119.5, 110.0, 86.6, 21.2. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O$) requires m/z 272.1201, found m/z 272.1194.

(*E*)-2-(3-methoxyphenyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ae)



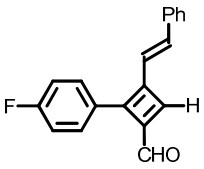
Yellow oil, 26.2 mg, 91% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.68 (s, 1H), 8.02 (d, *J* = 16.5 HMz, 1H), 7.59-7.57 (m, 2H), 7.41-7.38 (m, 2H), 7.35-7.33 (m, 2H), 7.28-7.25 (m, 1H), 7.20-7.19 (m, 1H), 7.10 (s, 1H), 7.02-7.00 (m, 1H), 6.53 (s, 1H), 3.86 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 159.5, 143.4, 137.2, 136.4, 129.7, 128.8, 128.7, 127.0, 124.4, 123.2, 119.5, 116.6, 116.4, 109.5, 86.7, 55.3. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O_2$) requires m/z 288.1150, found m/z 288.1144.

(*E*)-3-styryl-2-p-tolylcyclobuta-1,3-dienecarbaldehyde (3af)



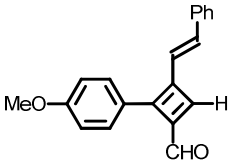
Yellow oil, 23.9 mg, 88% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.67 (s, 1H), 8.02 (d, *J* = 16.5 HMz, 1H), 7.59-7.57 (m, 2H), 7.50-7.48 (m, 2H), 7.42-7.39 (m, 2H), 7.35-7.32 (m, 1H), 7.29-7.28 (m, 1H), 7.26-7.24 (m, 2H), 6.54 (s, 1H), 2.43 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 142.9, 140.4, 137.3, 136.1, 131.8, 129.5, 128.7, 128.6, 127.5, 127.0, 119.5, 119.3, 110.3, 86.6, 21.7. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O$) requires m/z 272.1201, found m/z 272.1193.

(*E*)-2-(4-fluorophenyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ag)



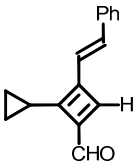
Yellow oil, 24.0 mg, 87% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.68 (s, 1H), 8.00 (d, *J* = 16.5 HMz, 1H), 7.60-7.56 (m, 4H), 7.43-7.39 (m, 2H), 7.36-7.34 (m, 1H), 7.27-7.22 (m, 1H), 7.16-7.12 (m, 2H), 6.52 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.2, 163.4 (d, J = 252.7 Hz), 143.3, 137.1, 136.4, 134.0 (d, J = 8.7 Hz), 128.8, 128.7, 127.0, 126.9, 119.4, 118.4 (d, J = 3.6 Hz), 116.2 (d, J = 22.3 Hz), 108.4, 86.7. HRMS (EI): exact mass calculated for M ($C_{19}H_{13}OF$) requires m/z 276.0950, found m/z 276.0948.

(*E*)-2-(4-methoxyphenyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ah)



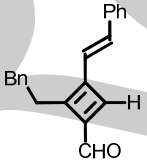
Yellow oil, 26.8 mg, 93% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.64 (s, 1H), 8.03 (d, *J* = 16.5 HMz, 1H), 7.60-7.53 (m, 4H), 7.43-7.39 (m, 2H), 7.35-7.33 (m, 1H), 7.30-7.25 (m, 1H), 6.97-6.94 (m, 2H), 6.53 (s, 1H), 3.87 (s, 3H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.4, 161.0, 142.3, 137.3, 135.8, 133.7, 128.8, 128.6, 127.9, 127.0, 119.6, 114.5, 114.3, 110.8, 86.6, 55.4. HRMS (EI): exact mass calculated for M ($C_{20}H_{16}O_2$) requires m/z 288.1150, found m/z 288.1148.

(*E*)-2-cyclopropyl-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ai)



Yellow oil, 18.4 mg, 83% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.56 (s, 1H), 7.91 (d, *J* = 16.5 HMz, 1H), 7.53-7.52 (m, 2H), 7.40-7.37 (m, 2H), 7.32-7.31 (m, 1H), 7.13-7.10 (m, 1H), 6.29 (s, 1H), 1.65-1.61 (m, 1H), 1.09-1.07 (m, 2H), 0.98-0.96 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.7, 142.8, 137.3, 135.3, 129.2, 128.7, 128.5, 126.9, 119.5, 116.8, 74.2, 10.1, 1.5. HRMS (EI): exact mass calculated for M ($C_{16}H_{14}O$) requires m/z 222.1045, found m/z 222.1046.

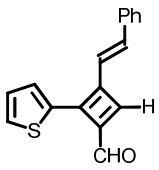
(*E*)-2-phenethyl-3-styrylcyclobuta-1,3-dienecarbaldehyde (3aj)



Yellow oil, 25.7 mg, 90% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.60 (s, 1H), 7.90 (d, *J* = 16.5 HMz, 1H), 7.48-7.47 (m, 2H), 7.39-7.26 (m, 8H), 7.09-7.06 (m, 1H), 6.31 (s, 1H), 3.02-2.99 (m, 2H), 2.93-2.90 (m, 2H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.7, 143.3, 140.0, 137.2, 135.8, 129.0, 128.7, 128.6, 128.5, 128.4, 127.1, 126.6, 119.5, 111.6, 79.0, 34.7, 22.6. HRMS (EI): exact mass calculated for M ($C_{21}H_{18}O$) requires m/z 286.1358, found m/z 286.1347.

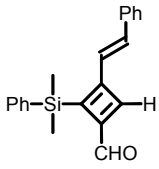
(*E*)-3-styryl-2-(thiophen-2-yl)cyclobuta-1,3-dienecarbaldehyde (3ak)

Yellow oil, 23.2 mg, 88% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.66 (s, 1H),



8.01 (d, *J* = 16.5 HMz, 1H), 7.69-7.88 (m, 1H), 7.59-7.56 (m, 2H), 7.42-7.39 (m, 3H), 7.35-7.33 (m, 1H), 7.25-7.22 (m, 2H), 6.52 (s, 1H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.3, 143.0, 137.2, 136.2, 130.1, 129.6, 128.8, 128.7, 127.2, 127.0, 126.2, 121.5, 119.5, 104.9, 86.9. HRMS (EI): exact mass calculated for M ($C_{17}H_{12}OS$) requires m/z 264.0609, found m/z 264.0605.

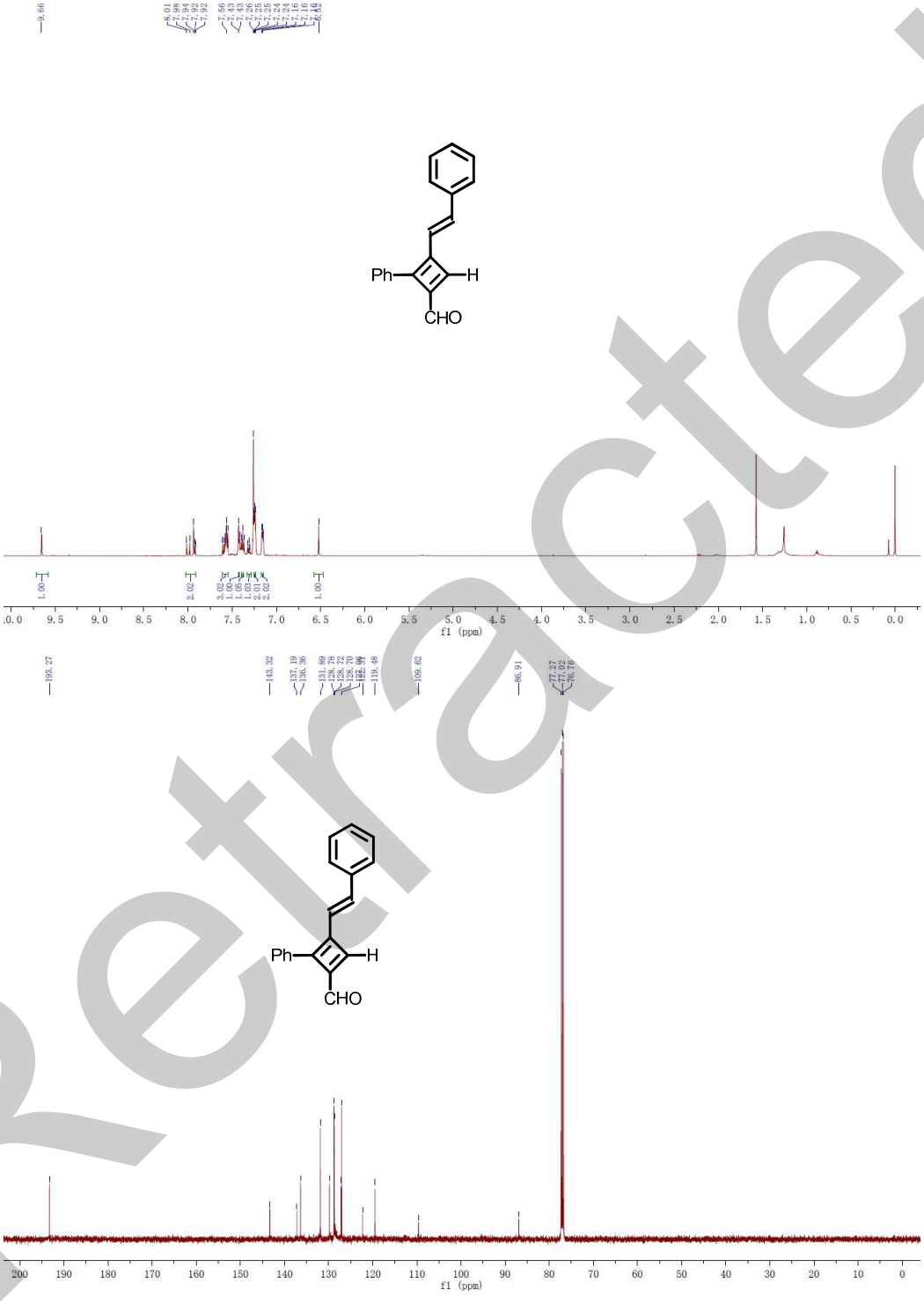
(*E*)-2-(dimethyl(phenyl)silyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3al)



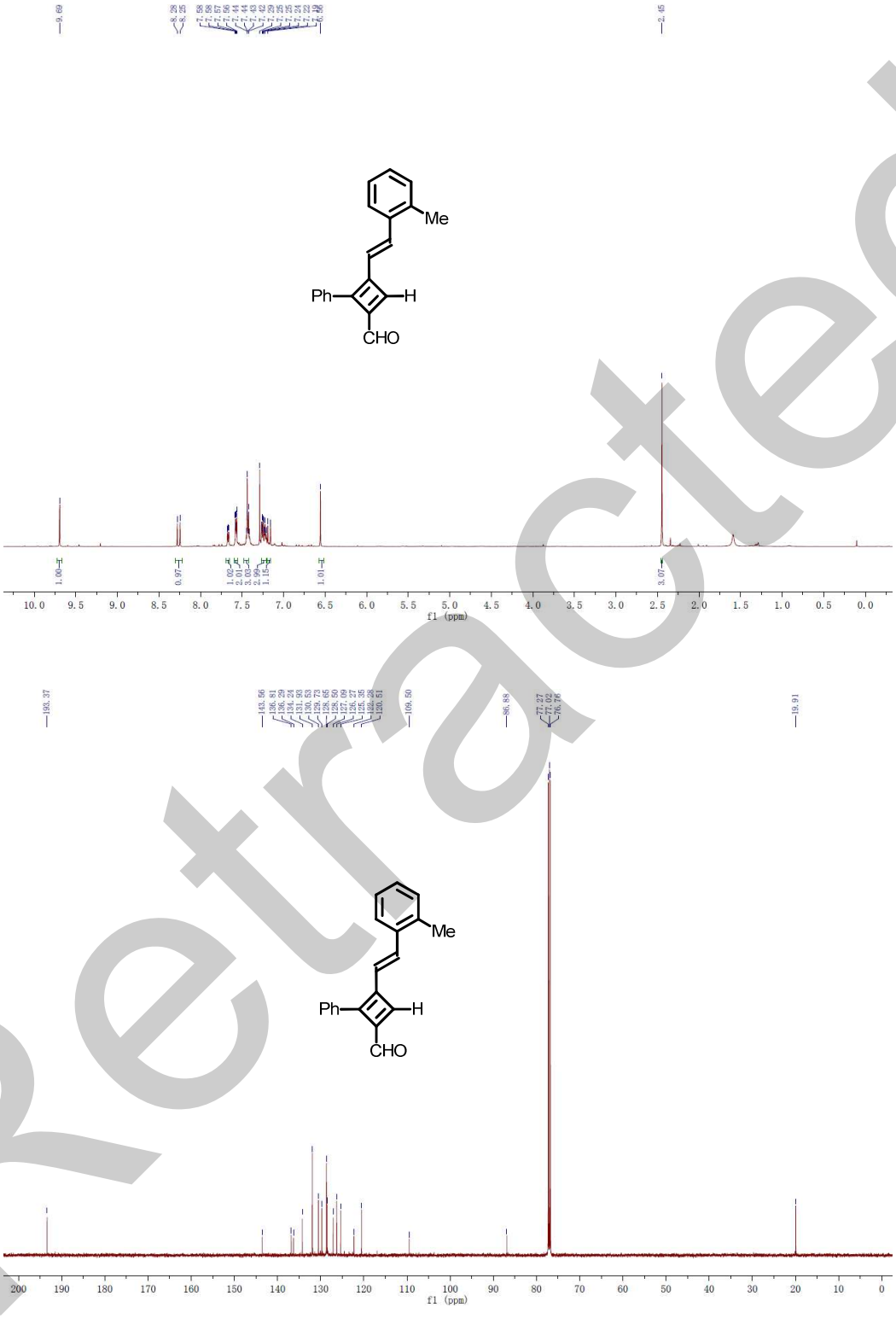
Yellow oil, 24.6 mg, 78% yield. ^{1}H NMR ($CDCl_3$, 500 MHz): δ (ppm) 9.64 (s, 1H), 8.00 (d, *J* = 16.5 HMz, 1H), 7.75-7.73 (m, 2H), 7.48-7.46 (m, 5H), 7.38-7.34 (m, 3H), 7.26-7.21 (m, 1H), 6.34 (s, 1H), 0.61 (s, 6H). ^{13}C NMR ($CDCl_3$, 125 MHz): δ (ppm) 193.4, 144.8, 137.0, 136.8, 135.8, 133.7, 129.9, 128.8, 128.7, 128.2, 127.1, 126.4, 119.3, 114.8, 102.8, 1.2. HRMS (EI): exact mass calculated for M ($C_{21}H_{20}OSi$) requires m/z 315.1205, found m/z 315.1198.

D: NMR Analysis of Cascade Product

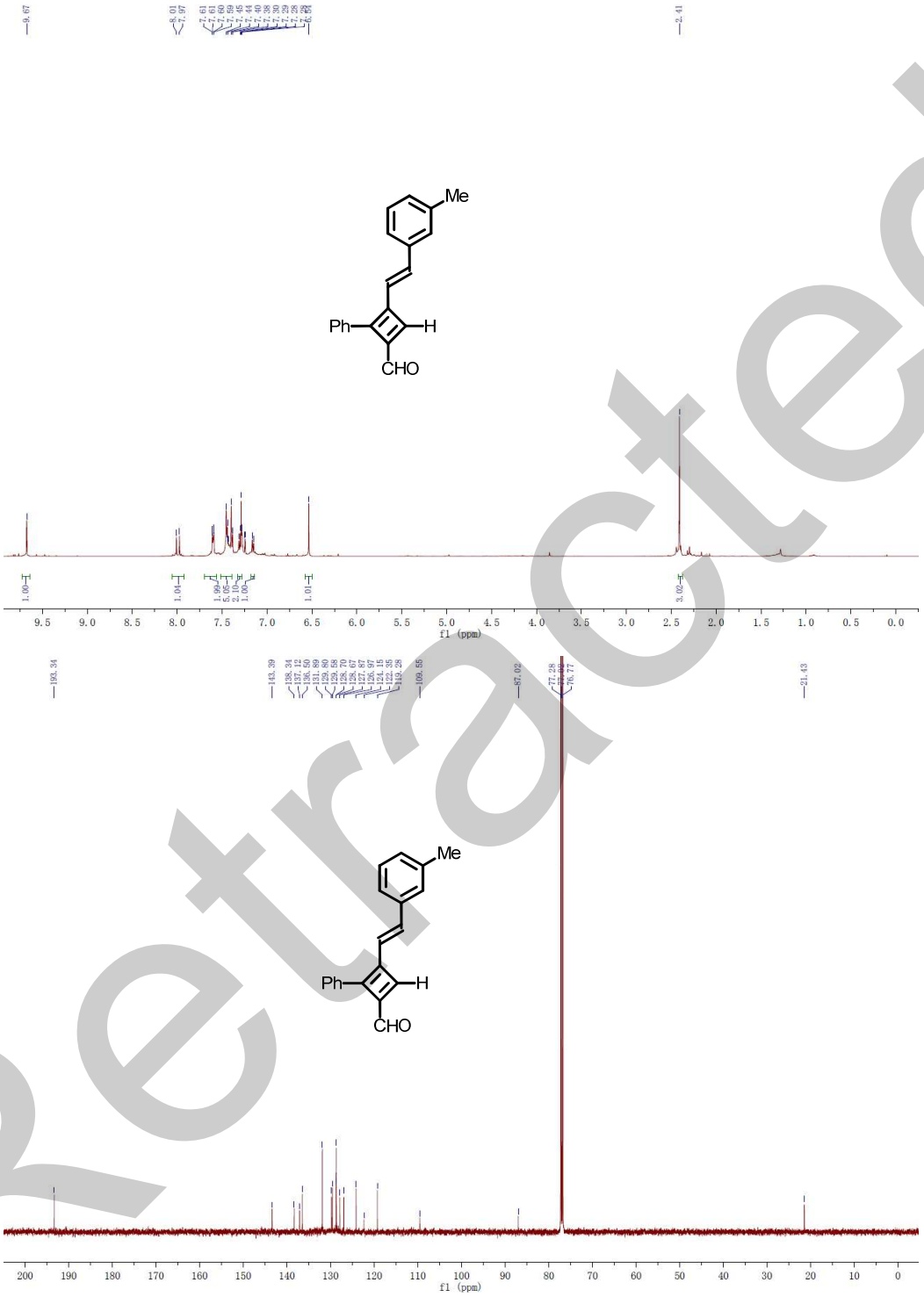
(*E*)-2-phenyl-3-styrylcyclobuta-1,3-dienecarbaldehyde (3aa)



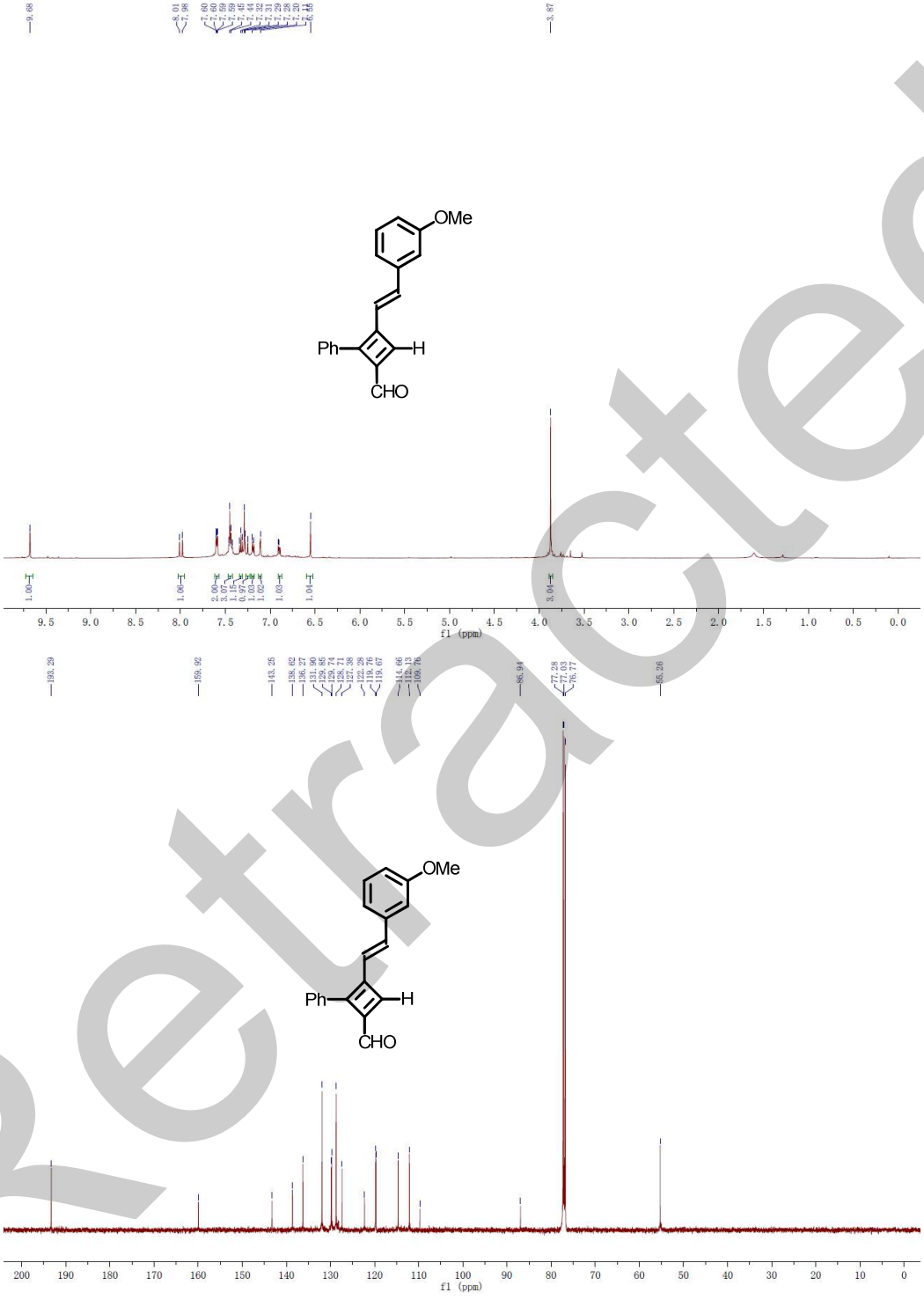
(*E*)-3-(2-methylstyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3ba)



(*E*)-3-(3-methylstyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3ca)

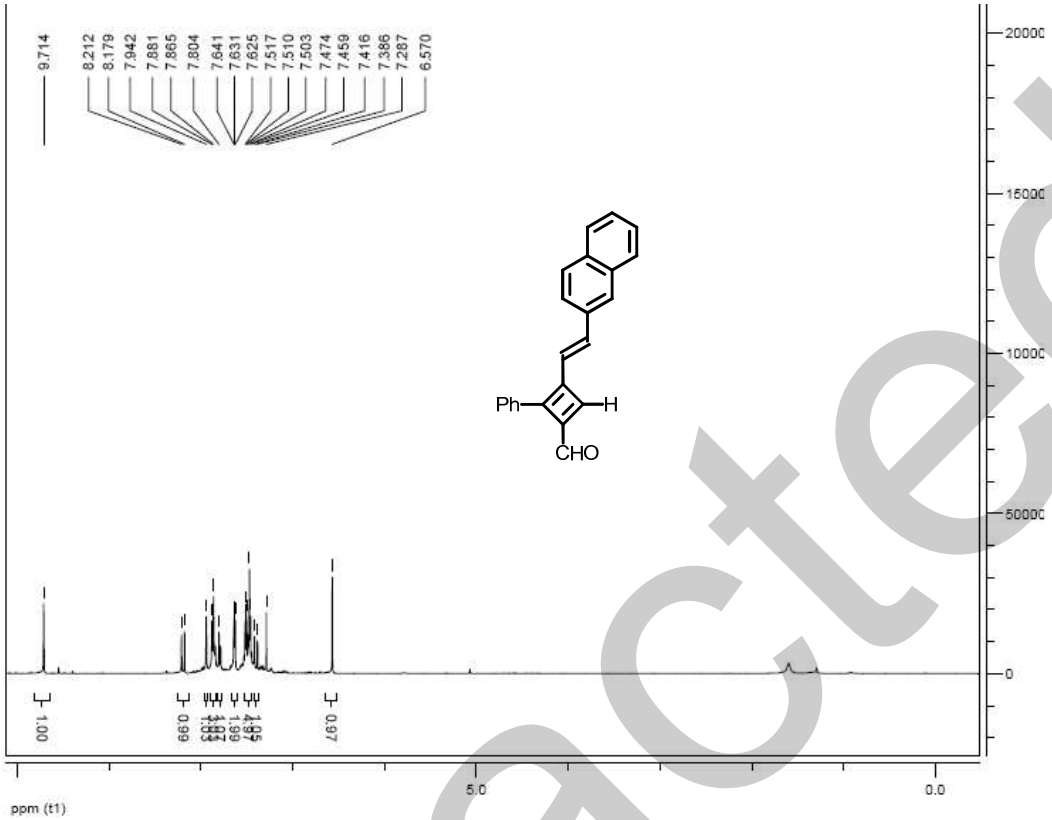


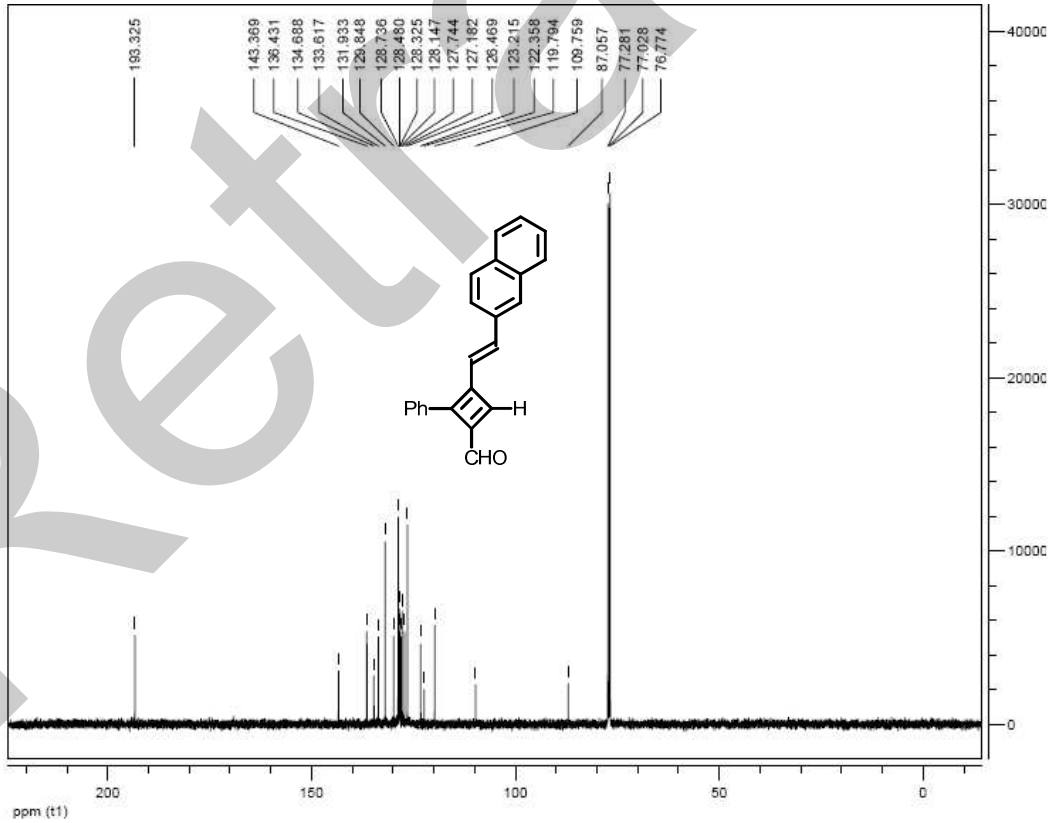
(*E*)-3-(3-methoxystyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3da)



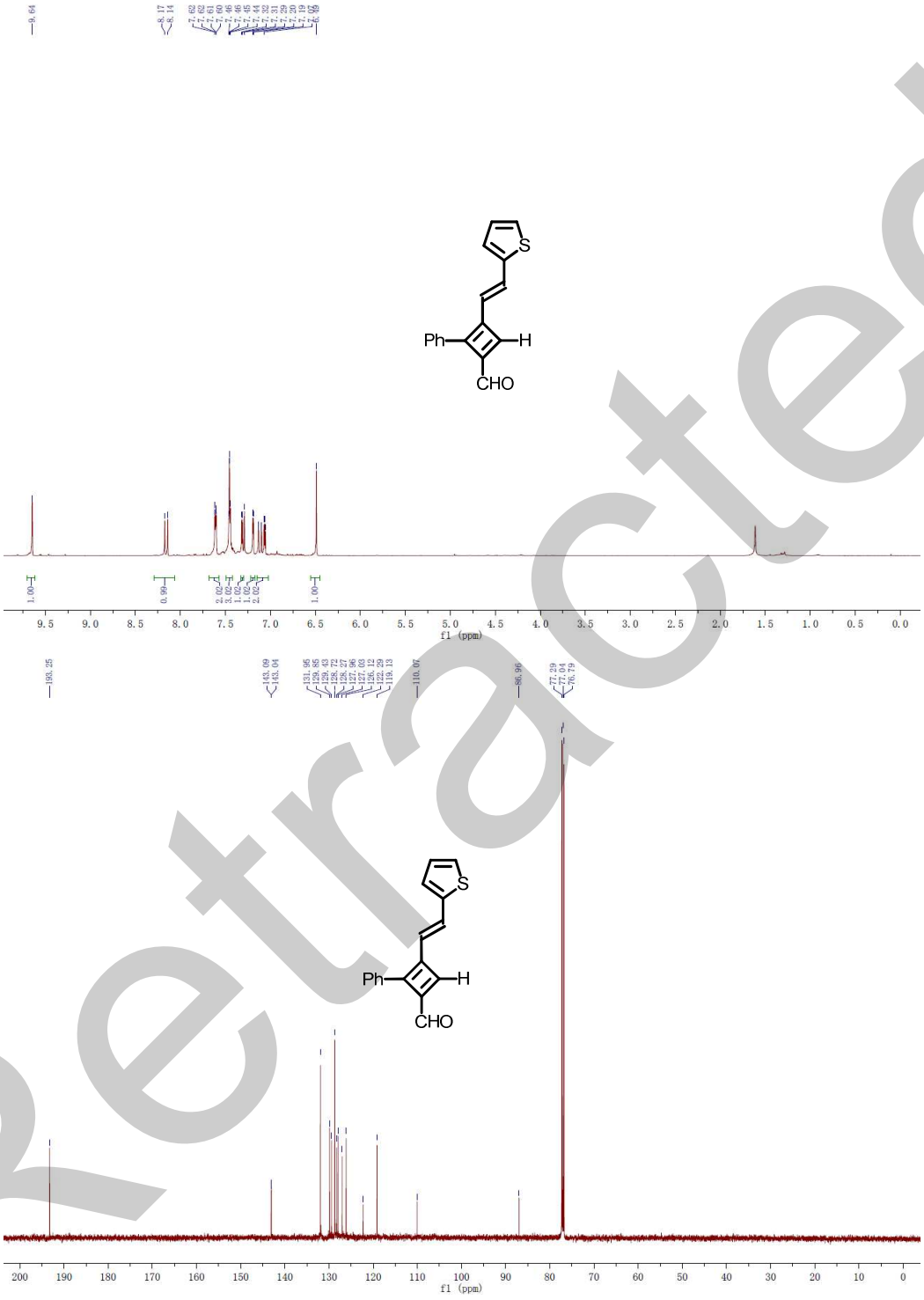
(*E*)-3-(4-chlorostyryl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3ea)

(*E*)-3-(2-(naphthalen-2-yl)vinyl)-2-phenylcyclobuta-1,3-dienecarbaldehyde (3fa)





(*E*)-2-phenyl-3-(2-(thiophen-2-yl)vinyl)cyclobuta-1,3-dienecarbaldehyde (3ga)



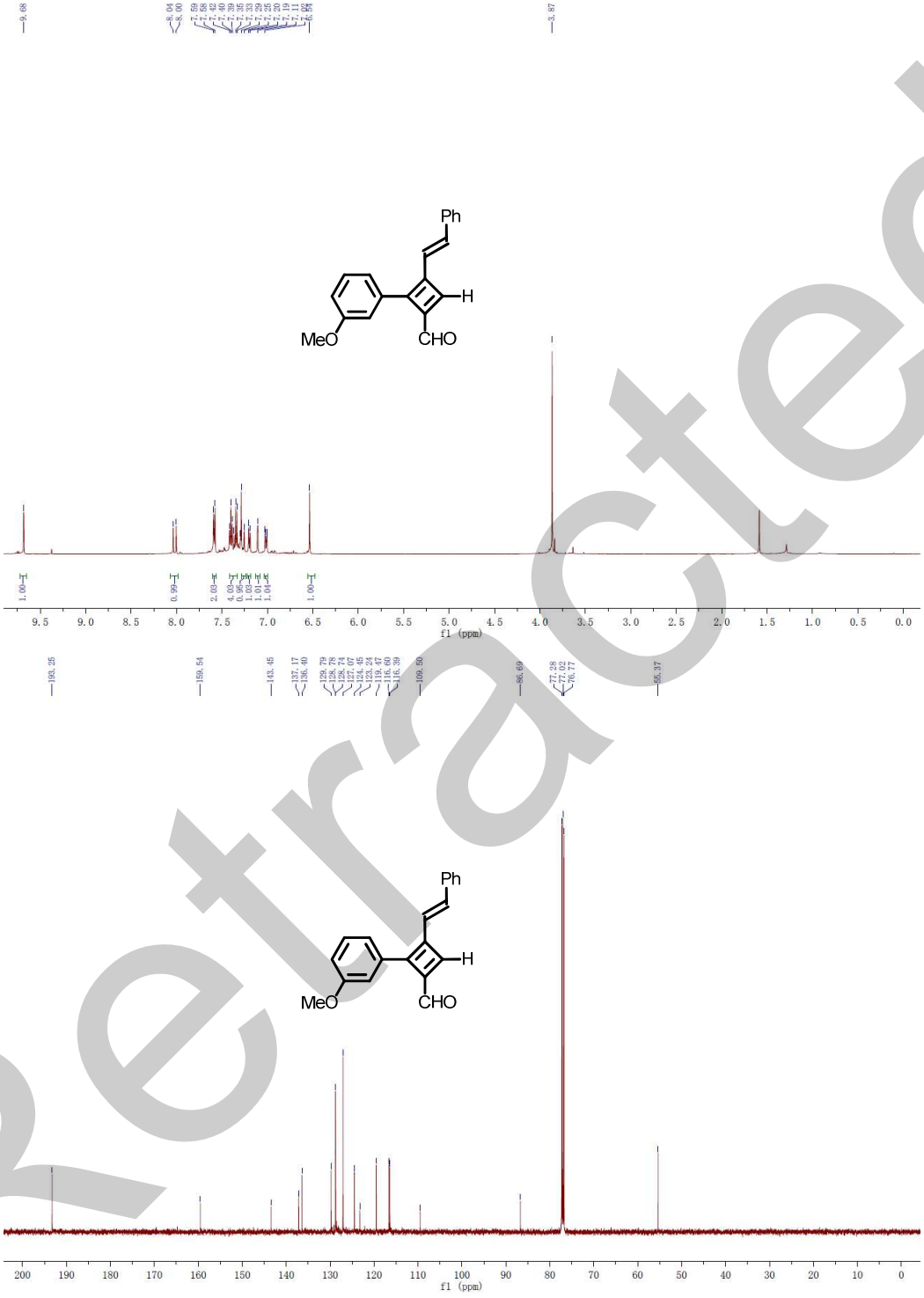
(*E*)-3-styryl-2-o-tolylcyclobuta-1,3-dienecarbaldehyde (3ab)

(*E*)-3-(3-methoxystyryl)-2-(2-chlorophenyl)cyclobuta-1,3-dienecarbaldehyde

(3ac)

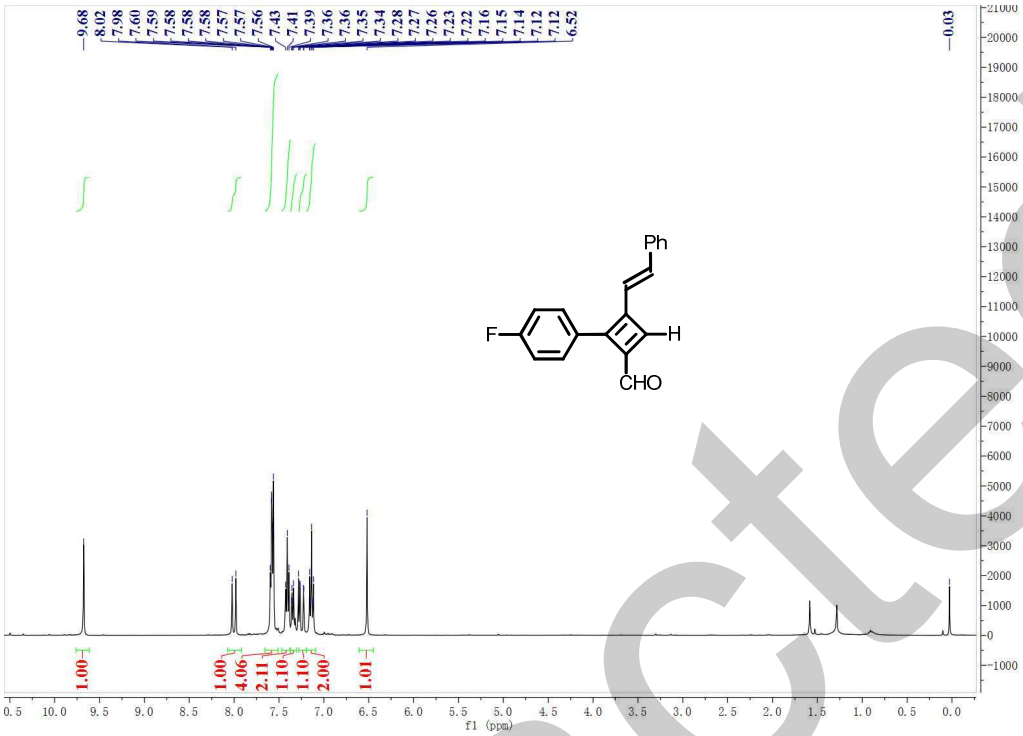
(*E*)-3-styryl-2-m-tolylcyclobuta-1,3-dienecarbaldehyde (3ad)

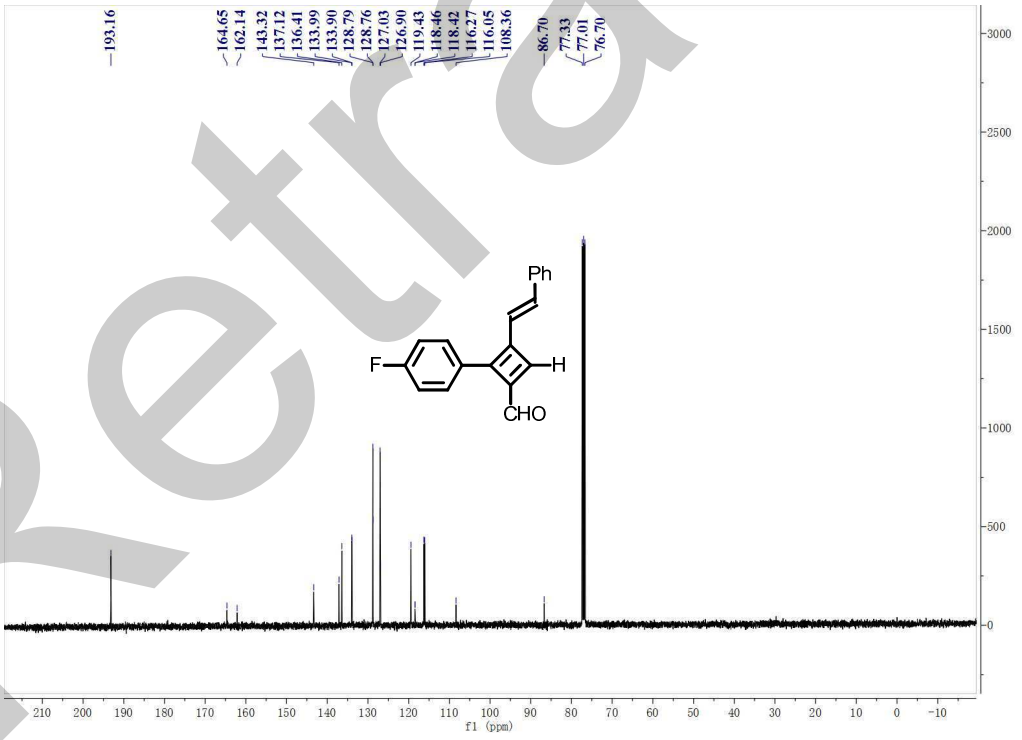
(*E*)-2-(3-methoxyphenyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ae)



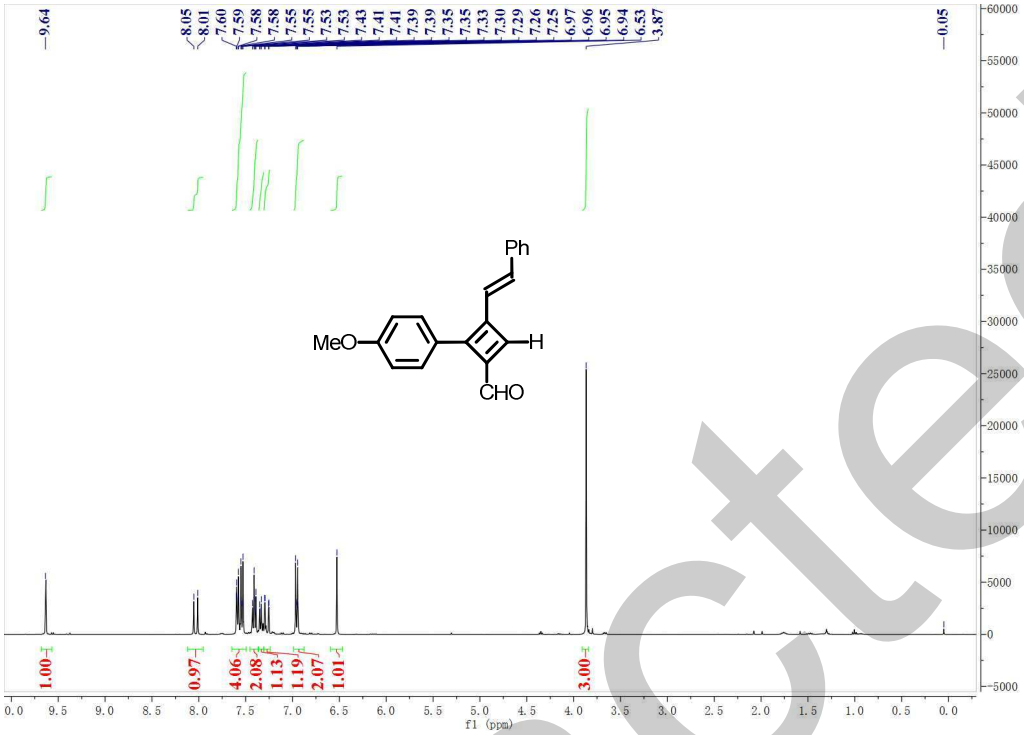
(*E*)-3-styryl-2-p-tolylcyclobuta-1,3-dienecarbaldehyde (3af)

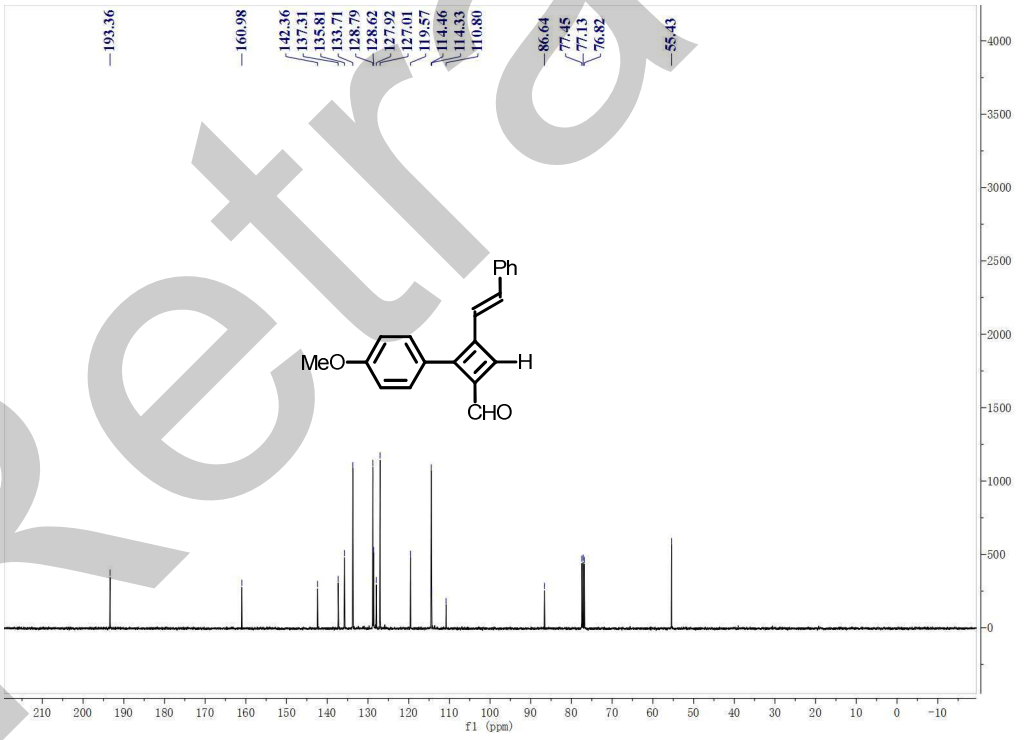
(*E*)-2-(4-fluorophenyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ag)



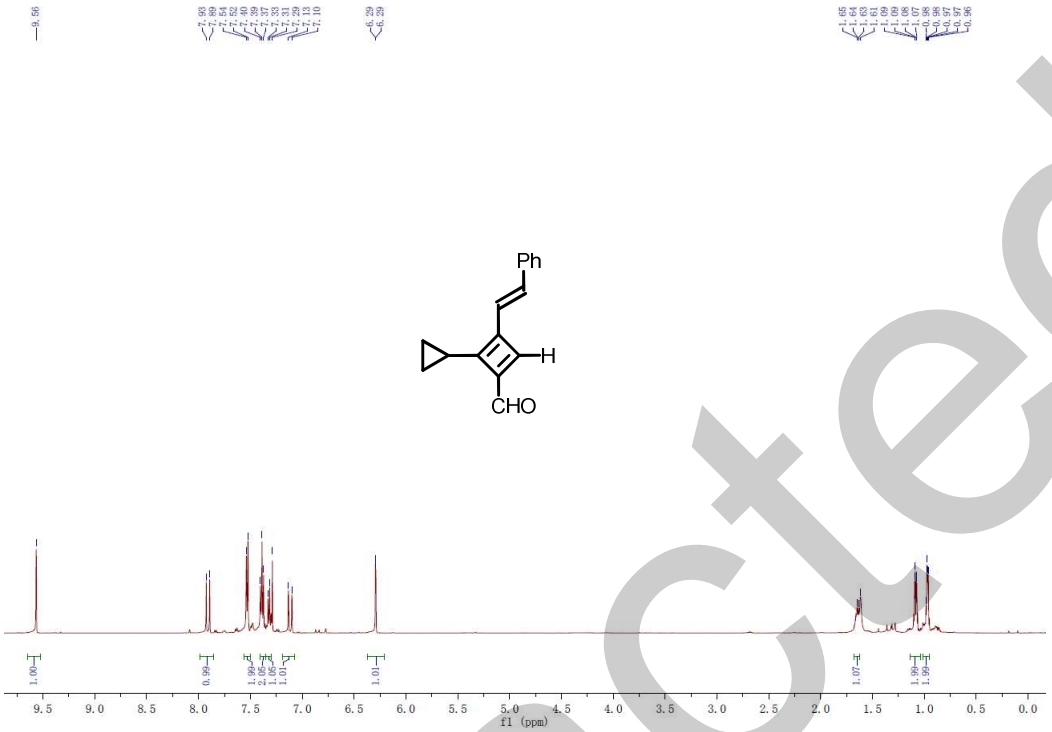


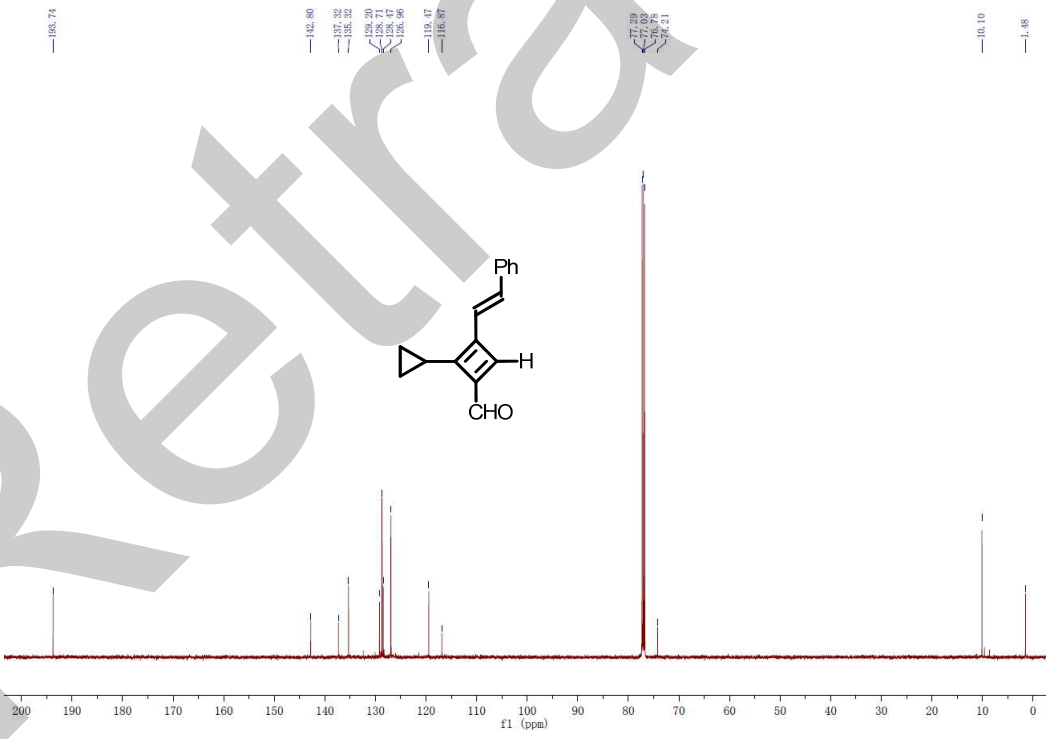
(*E*)-2-(4-methoxyphenyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ah)



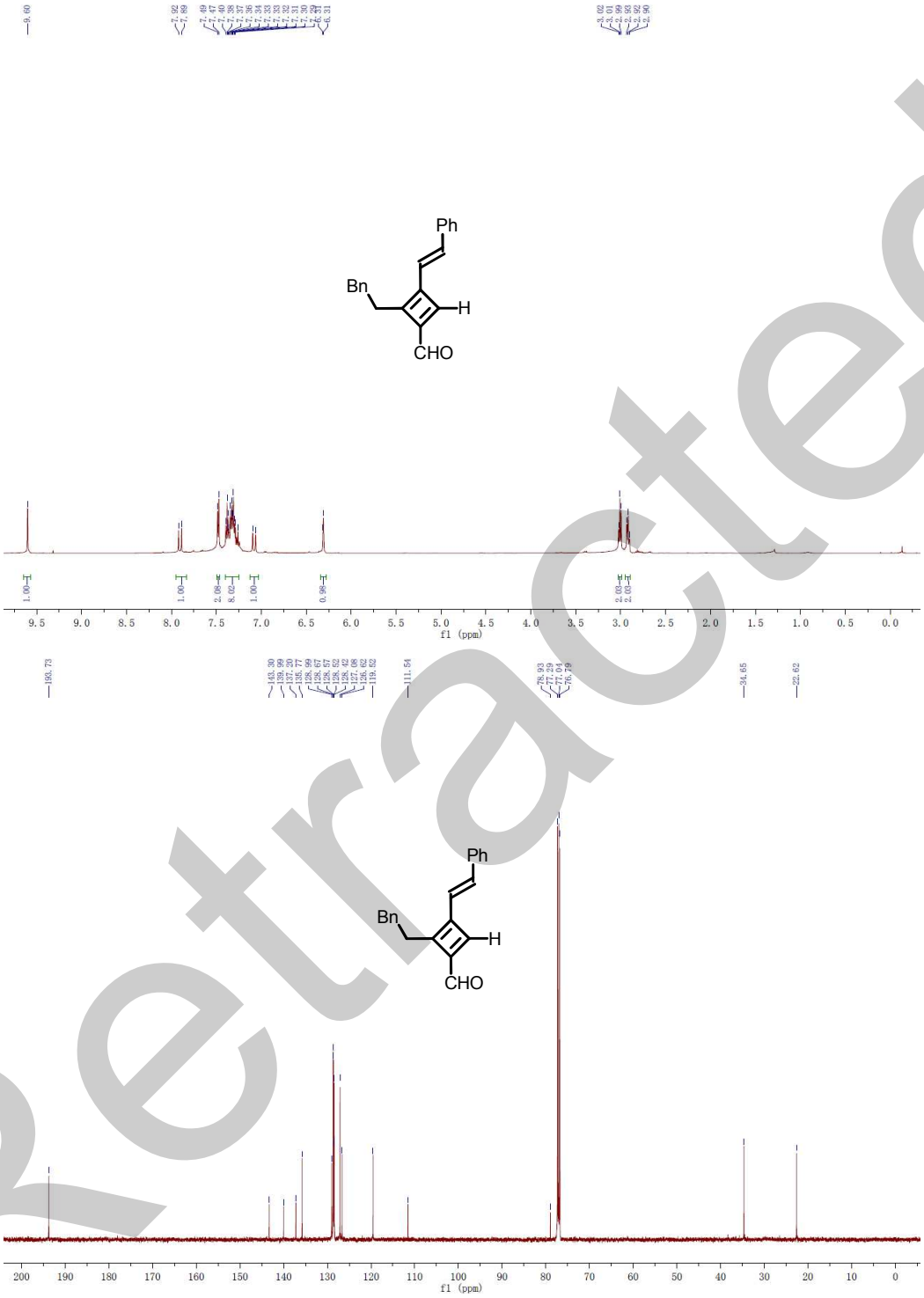


(*E*)-2-cyclopropyl-3-styrylcyclobuta-1,3-dienecarbaldehyde (3ai)

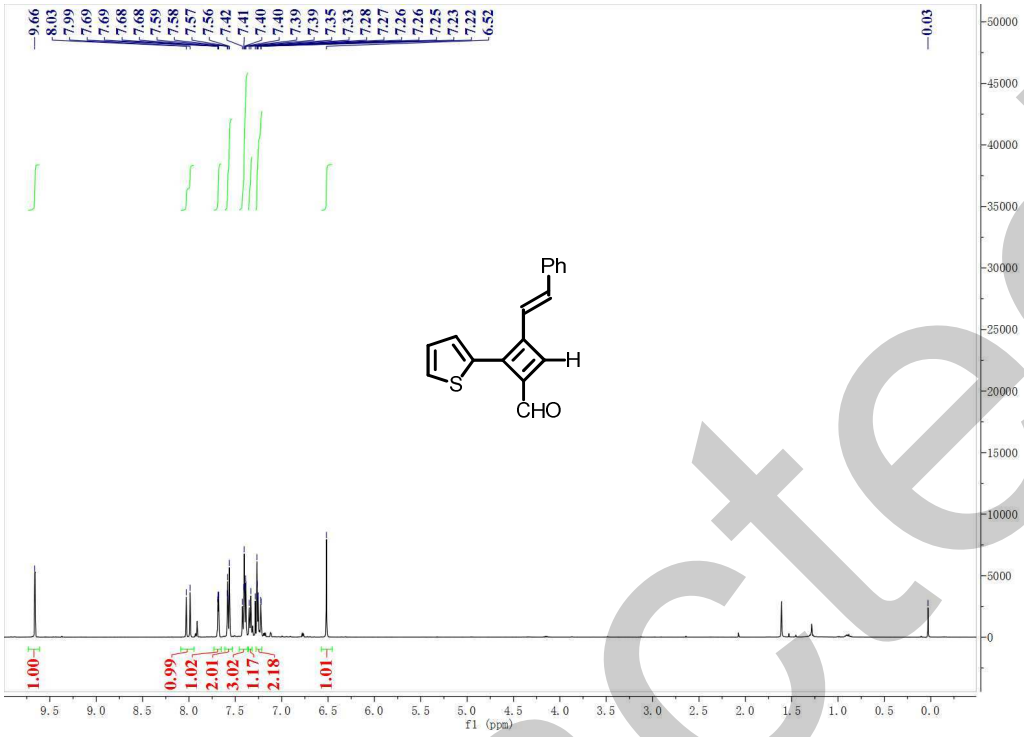


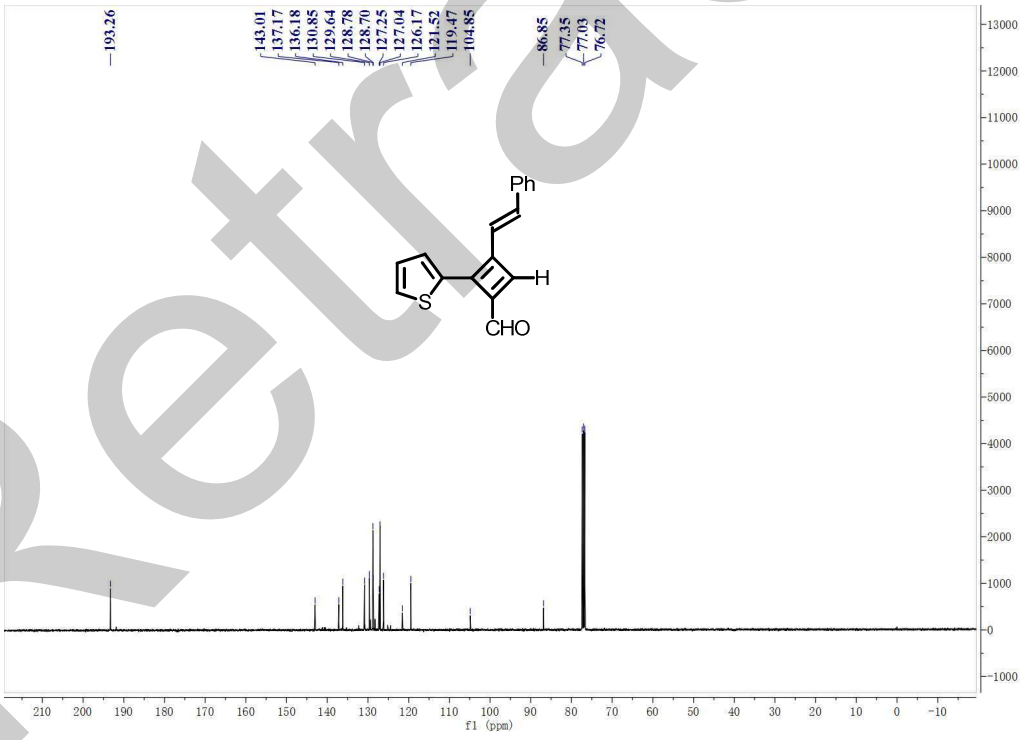


(*E*)-2-phenethyl-3-styrylcyclobuta-1,3-dienecarbaldehyde (3aj)

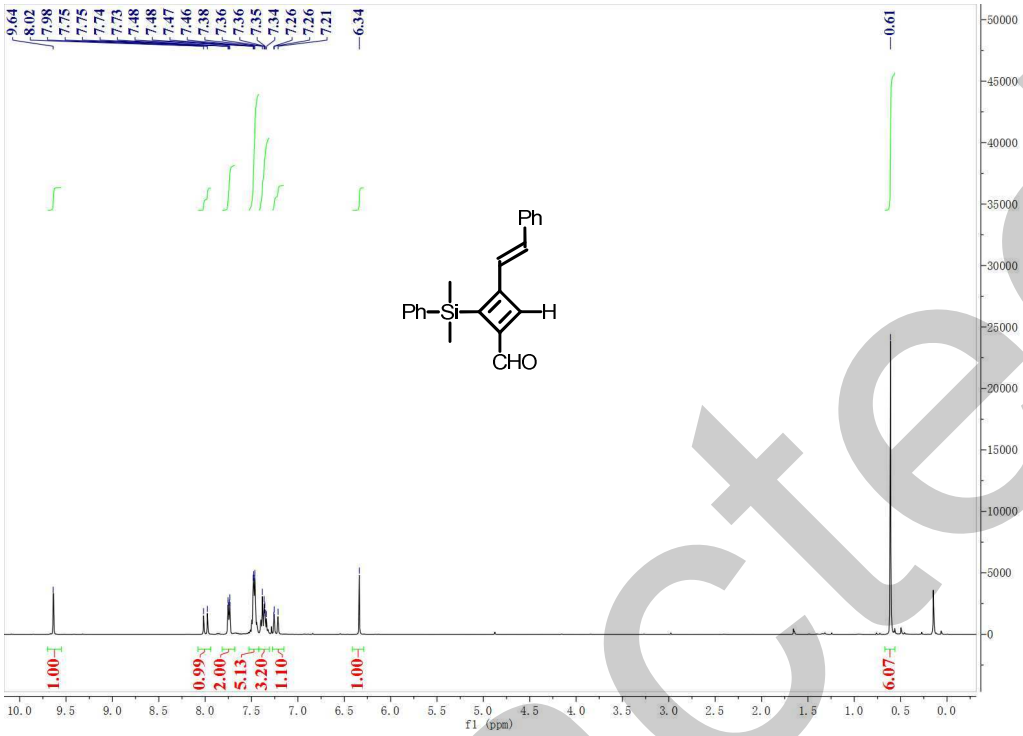


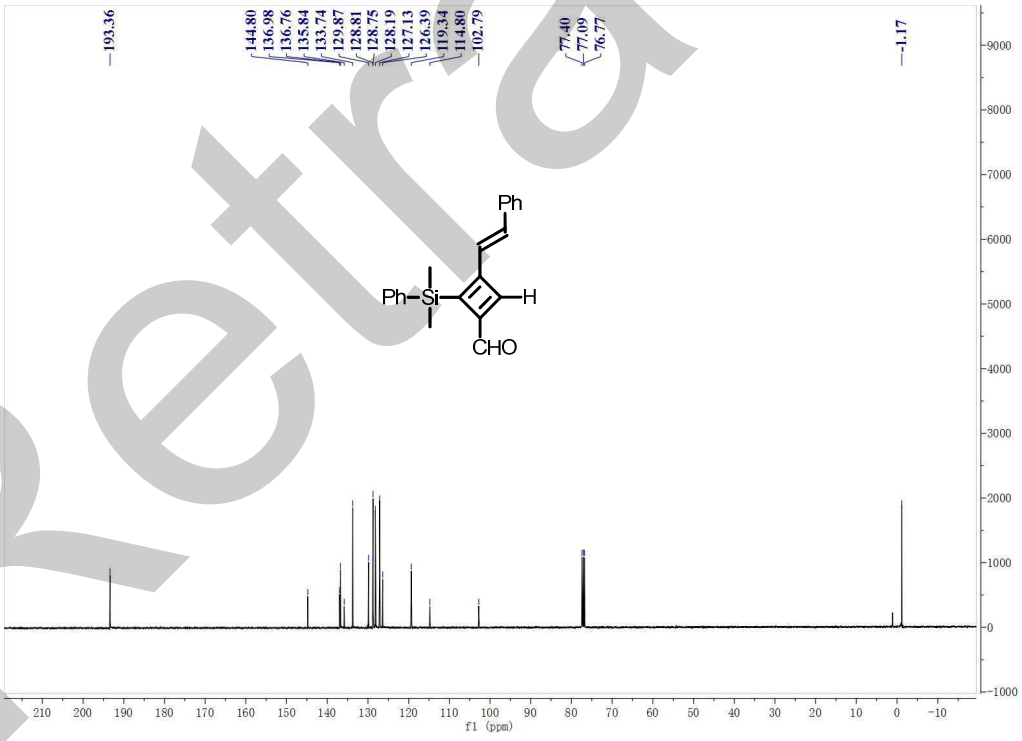
(*E*)-3-styryl-2-(thiophen-2-yl)cyclobuta-1,3-dienecarbaldehyde (3ak)





(*E*)-2-(dimethyl(phenyl)silyl)-3-styrylcyclobuta-1,3-dienecarbaldehyde (3al)





E: References

1. (a) H. Kuroda, E. Hanaki, H. Izawa, M. Kano, H. Itahashi, *Tetrahedron* 2004, **60**, 1913-1920;
(b) M. Journet, D. Cai, L. M. DiMichele, R. D. Larsen., *Tetrahedron Lett.* 1998, **39**, 6427-6428.