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Supporting Information for

α-Arylsulfonyloxyacrylates: attractive *O*-centered electrophiles for synthesis of α-substituted acrylates via Pd-catalysed Suzuki reactions

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1 General Information

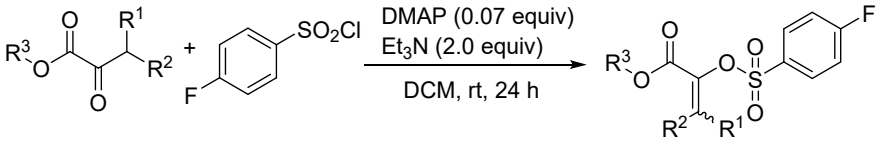
1.1 Solvents, Reagents, and Starting Materials

All reactions were carried out under an atmosphere of nitrogen in oven-dried glassware. The α-ketoesters are commercially available or prepared following literature known methods.[1] The potassium (hetero)aryltrifluoroborates were reported by our previous work or prepared according to the literature procedures.[2] The employed phosphorus ligands are commercially available. The dried solvents were obtained from commercial sources and used without further purification unless otherwise noted.

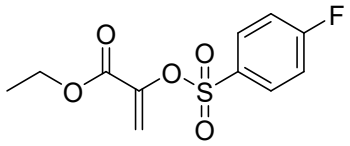
1.2 Instruments

NMR spectra were recorded on a BrukerAvance 500 spectrometer (500 MHz) (500 MHz for ^{1}H NMR, 126 MHz for ^{13}C NMR, and 471 MHz for ^{19}F NMR). Chemical shifts were reported in ppm downfield from tetramethylsilane and calibrated using residue undeuterated solvent (Chloroform-*d* at 7.26 ppm ^{1}H NMR; 77.0 ppm ^{13}C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad). Coupling constants are reported in Hertz where available. High resolution mass spectra (HRMS) were recorded on Waters Premier GC-TOF MS, Waters G2-Xs QTOF MS, and JEOL-AccuTOF-GCv4G-GCT MS. Analytical thin layer chromatography was performed on Polygram SIL G/UV254 plates. Visualization was accomplished with short wave UV light, or $KMnO_4$ staining solutions. Flash column chromatography was performed using silica gel (300-400 mesh) with solvents to use.

2 General Procedure of Synthesis of α-Arylsulfonyloxyacrylates

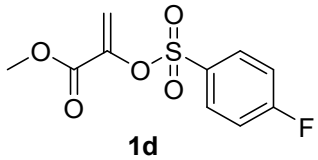


A mixture of α-ketoester (15 mmol, 1.0 equiv), *p*-fluorosulfonyl chloride (3.5 g, 18 mmol), DMAP (134.5 mg, 1.1 mmol) was dissolved in dichloromethane (30 mL). Triethylamine (4.1 mL, 29.5 mmol) was added dropwise and reacted for 24 h. At the end of the reaction, the mixture was then quenched with H_2O (20 mL). The organic phase was separated and the aqueous phase extracted with ethyl acetate (3 x 15 mL). The combined organic phase was dried by $MgSO_4$ and the solvent removed *in vacuo*. Crude products were purified by silica gel column chromatography.



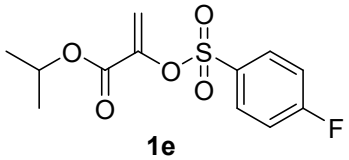
1a

Ethyl 2-(((4-fluorophenyl)sulfonyl)oxy)acrylate (1a). Flash column chromatography to afford product **1a** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 – 7.86 (m, 2H), 7.18 (t, *J* = 8.6 Hz, 2H), 6.08 (d, *J* = 2.5 Hz, 1H), 5.57 (d, *J* = 2.5 Hz, 1H), 4.06 (q, *J* = 7.2 Hz, 2H), 1.12 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.7 (d, *J* = 258.3 Hz), 160.3, 142.8, 131.3 (d, *J* = 2.5 Hz), 131.2 (d, *J* = 10.1 Hz), 116.9, 116.2 (d, *J* = 22.7 Hz), 61.7, 13.5. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.16. HRMS (ESI) $[M+Na]^+$: calculated for$C_{11}H_{11}FO_5SNa$: 297.0209, found 297.0210.



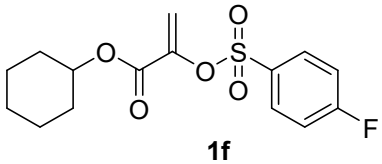
1d

Methyl 2-(((4-fluorophenyl)sulfonyl)oxy)acrylate (1d). Flash column chromatography to afford product **1d** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.91 (m, 2H), 7.22 (t, *J* = 8.5 Hz, 2H), 6.13 (d, *J* = 2.5 Hz, 1H), 5.64 (d, *J* = 2.6 Hz, 1H), 3.67 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.0 (d, *J* = 258.3 Hz), 161.1, 142.7, 131.4 (d, *J* = 2.5 Hz), 131.3 (d, *J* = 10.1 Hz), 117.40, 116.4 (d, *J* = 25.2 Hz), 52.6. ^{19}F NMR (471 MHz, $CDCl_3$) δ -101.82. HRMS (ESI) $[M+Na]^+$: calculated for $C_{10}H_9FO_5SNa$: 283.0052, found 283.0059.



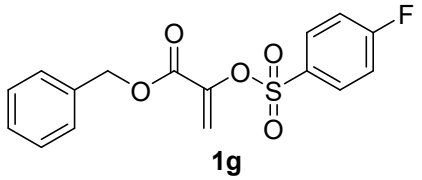
1e

Isopropyl 2-(((4-fluorophenyl)sulfonyl)oxy)acrylate (1e). Flash column chromatography to afford product **1e** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 – 7.85 (m, 2H), 7.38 – 7.09 (m, 2H), 6.15 (s, 1H), 5.68 (d, *J* = 2.3 Hz, 1H), 4.99 (p, *J* = 6.3 Hz, 1H), 1.21 (d, *J* = 6.4 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.1 (d, *J* = 258.3 Hz), 160.2, 143.4, 131.8 (d, *J* = 2.5 Hz), 131.5 (d, *J* = 10.1 Hz), 117.0, 116.5 (d, *J* = 23.9 Hz), 70.2, 21.5. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.0. HRMS (ESI) $[M+Na]^+$: calculated for $C_{12}H_{13}FO_5SNa$: 311.0362, found 311.0364.



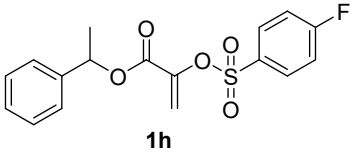
1f

Cyclohexyl2-(((4-fluorophenyl)sulfonyl)oxy)acrylate (1f). Flash column chromatography to afford product **1f** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 – 7.97 (m, 2H), 7.26 – 7.21 (m, 2H), 6.15 (d, *J* = 2.3 Hz, 1H), 5.66 (d, *J* = 2.3 Hz, 1H), 4.80 – 4.74 (m, 1H), 1.80 – 1.67 (m, 4H), 1.54 – 1.49 (m, 1H), 1.43 – 1.24 (m, 5H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.1 (d, *J* = 257.0 Hz), 160.1, 143.4, 131.7 (d, *J* = 2.5 Hz), 131.4 (d, *J* = 10.1 Hz), 116.9, 116.5 (d, *J* = 25.2 Hz), 74.8, 31.2, 25.2, 23.4. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.02. HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{17}FO_5S$ Na: 351.0678, found 351.0685.



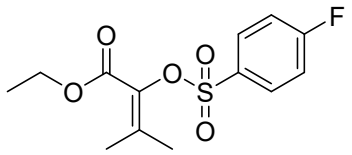
1g

Benzyl 2-(((4-fluorophenyl)sulfonyl)oxy)acrylate (1g). Flash column chromatography to afford product **1g** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 – 7.90 (m, 2H), 7.38 – 7.35 (m, 3H), 7.30 – 7.26 (m, 2H), 7.10 (t, *J* = 8.5 Hz, 2H), 6.23 (d, *J* = 2.3 Hz, 1H), 5.76 (d, *J* = 2.4 Hz, 1H), 5.13 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.9 (d, *J* = 258.3 Hz), 160.6, 142.8, 134.7, 131.4 (d, *J* = 8.8 Hz), 131.3 (d, *J* = 3.8 Hz), 128.6, 128.5, 128.4, 117.9, 116.4 (d, *J* = 25.2 Hz), 67.7. ^{19}F NMR (471 MHz, $CDCl_3$) δ -101.64. HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{13}FO_5S$ Na: 359.0365, found 359.0368.



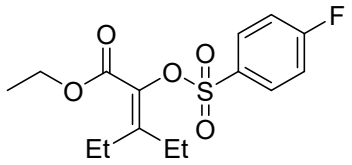
1h

1-Phenylethyl 2-(((4-fluorophenyl)sulfonyl)oxy)acrylate (1h). Flash column chromatography to afford product **1h** as a colorless liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 – 7.85 (m, 2H), 7.41 – 7.26 (m, 5H), 7.19 – 7.06 (m, 2H), 6.22 (d, *J* = 2.4 Hz, 1H), 5.85 (q, *J* = 6.6 Hz, 1H), 5.74 (d, *J* = 2.4 Hz, 1H), 1.53 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.9 (d, *J* = 258.3 Hz), 159.9, 143.1, 140.4, 131.4 (d, *J* = 3.8 Hz), 131.3 (d, *J* = 10.1 Hz), 128.5, 128.2, 126.1, 117.5, 116.4 (d, *J* = 22.7 Hz), 74.6, 21.8. ^{19}F NMR (471 MHz, $CDCl_3$) δ -101.73. HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{15}FO_5SNa$: 373.0522, found 373.0526.



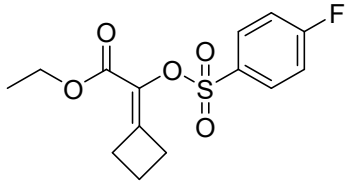
4a

Ethyl 2-(((4-fluorophenyl)sulfonyl)oxy)-3-methylbut-2-enoate (4a). Flash column chromatography to afford product **4a** as a colorless liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.99 (m, 2H), 7.24 (t, *J* = 8.5 Hz, 2H), 4.10 (q, *J* = 7.1 Hz, 2H), 2.18 (s, 3H), 1.82 (s, 3H), 1.20 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.8 (d, *J* = 257.0 Hz), 162.0, 144.5, 132.9, 132.8(d, *J* = 13.8 Hz), 131.1(d, *J* = 8.8 Hz), 116.3 (d, *J* = 25.2 Hz), 61.1, 21.3, 20.4, 13.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.73. HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{15}FO_5SNa$: 325.0522, found 325.0524.



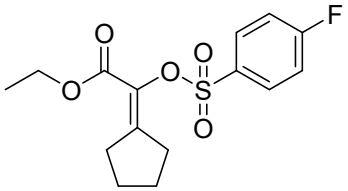
4b

Ethyl 3-ethyl-2-(((4-fluorophenyl)sulfonyl)oxy)pent-2-enoate (4b). Flash column chromatography to afford product **4b** as a colorless liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.97 (m, 2H), 7.25 – 7.20 (m, 2H), 4.11 (q, *J* = 7.1 Hz, 2H), 2.50 (q, *J* = 7.5 Hz, 2H), 2.15 (q, *J* = 7.6 Hz, 2H), 1.21 (t, *J* = 7.1 Hz, 3H), 1.09 (t, *J* = 7.5 Hz, 3H), 0.97 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.8 (d, *J* = 258.3 Hz), 162.1, 153.8, 132.7 (d, *J* = 3.8 Hz), 132.4, 131.1 (d, *J* = 8.8 Hz), 116.3 (d, *J* = 25.2 Hz), 61.2, 24.6, 23.9, 13.9, 12.9, 11.6. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.76. HRMS (ESI) $[M+H]^+$: calculated for $C_{15}H_{19}FO_5S$: 331.1015, found 331.1002.



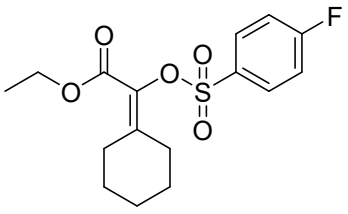
4c

Ethyl 2-cyclobutylidene-2-(((4-fluorophenyl)sulfonyl)oxy)acetate (4c). Flash column chromatography to afford product **4c** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.96 – 7.93 (m, 2H), 7.26 – 7.14 (m, 2H), 4.01 (q, *J* = 7.1 Hz, 2H), 3.07 – 3.03 (m, 2H), 2.78 – 2.74 (m, 2H), 1.98 (p, *J* = 7.9 Hz, 2H), 1.11 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.6 (d, *J* = 257.0 Hz), 161.2, 156.4, 132.4 (d, *J* = 2.5 Hz), 131.1 (d, *J* = 10.1 Hz), 129.5, 116.0 (d, *J* = 22.7 Hz), 60.8, 31.4, 29.7, 16.8, 13.8. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.88.HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{15}FO_5SNa$: 337.0522, found 337.0523.



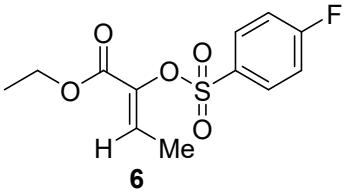
4d

Ethyl 2-cyclopentylidene-2-(((4-fluorophenyl)sulfonyl)oxy)acetate (4d). Flash column chromatography to afford product **4d** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.98 (m, 2H), 7.23 – 7.18 (m, 2H), 4.05 (t, *J* = 7.1 Hz, 2H), 2.81 – 2.76 (m, 2H), 2.57 – 2.53 (m, 2H), 1.75 (p, *J* = 6.9 Hz, 2H), 1.64 (q, *J* = 7.1 Hz, 2H), 1.15 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.7 (d, *J* = 257.0 Hz), 161.8, 158.2, 133.1 (d, *J* = 3.8 Hz), 133.1 (d, *J* = 10.1 Hz), 130.2, 116.2 (d, *J* = 22.7 Hz), 60.9, 33.4, 32.7, 26.6, 25.4, 13.9. ^{19}F NMR (471 MHz, $CDCl_3$) δ -103.03. HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{17}FO_5SNa$: 351.0678, found 351.0679.



4e

Ethyl 2-cyclohexylidene-2-(((4-fluorophenyl)sulfonyl)oxy)acetate (4e). Flash column chromatography to afford product **4e** as a white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 – 7.92 (m, 2H), 7.24 – 7.18 (m, 2H), 4.07 (q, *J* = 7.1 Hz, 2H), 2.72 – 2.66 (m, 2H), 2.17 (m, *J* = 6.8, 5.1 Hz, 2H), 1.60 (p, *J* = 5.7 Hz, 2H), 1.55 – 1.44 (m, 4H), 1.18 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.8 (d, *J* = 258.3 Hz), 162.2, 150.1, 132.5 (d, *J* = 3.8 Hz), 131.1 (d, *J* = 8.8 Hz), 130.0, 116.3 (d, *J* = 25.2 Hz), 61.1, 30.0, 29.5, 27.5, 27.1, 25.7, 13.8. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.71. HRMS (ESI) $[M+Na]^+$: calculated for $C_{16}H_{19}FO_5SNa$: 365.0835, found 365.0833.



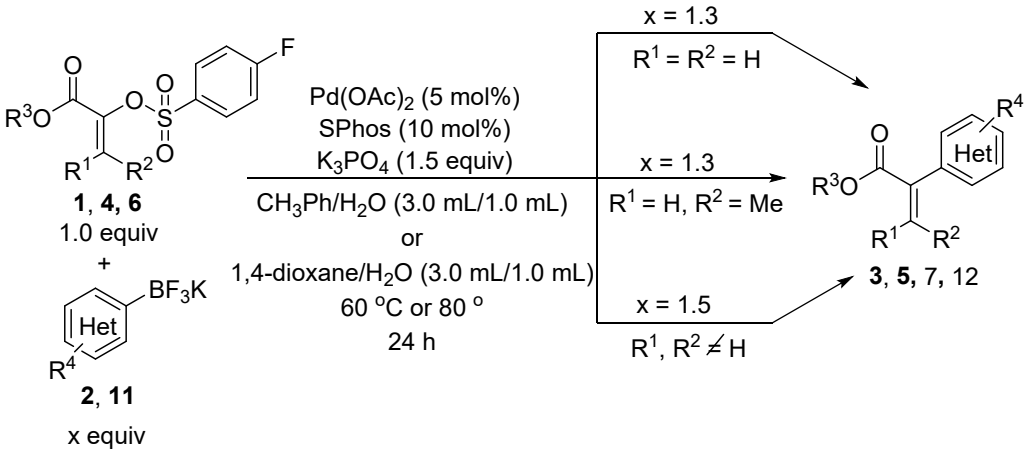
6

Ethyl (*Z*)-2-(((4-fluorophenyl)sulfonyl)oxy)but-2-enoate (6). Flash column chromatography to afford product **6** as a yellow liquid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.00 – 7.97 (m, 2H), 7.22 – 7.17 (m, 2H), 6.76 – 6.72 (m, 1H), 4.08 (q, *J* = 7.1 Hz, 2H), 1.75 (d, *J* = 7.3 Hz, 3H), 1.15 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 165.8 (d, *J* = 258.3 Hz), 161.2, 138.0,

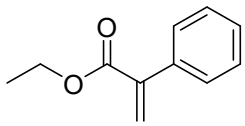
132.4 (d, J = 2.5 Hz), 131.6, 131.1 (d, J = 10.1 Hz), 116.2 (d, J = 25.2 Hz), 61.4, 13.8, 12.2. ^{19}F NMR (471 MHz, $CDCl_3$) δ -102.56. HRMS (ESI) $[M+Na]^+$: calculated for $C_{12}H_{13}FO_5SNa$: 311.0365, found 311.0372.

3 General Procedure of Suzuki Reaction

3.1 General Procedure of the (Hetero)arylation Reactions of α-Arylsulfonyloxyacrylates

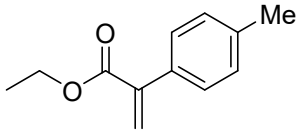


To a reaction tube equipped with a magnetic bar were added α-arylsulfonyloxyacrylate **1, 4** or **6** (0.2 mmol), potassium (hetero)aryltrifluoroborate **2** or **11** (0.26 mmol or 0.3 mmol), SPhos (8.2 mg, 0.02 mmol), K_3PO_4 (63.6 mg, 0.3 mmol), $Pd(OAc)_2$ (2.3 mg, 0.01 mmol). The toluene or 1,4-dioxane (3.0 mL) and H_2O (1.0 mL) were added subsequently. The reaction mixture was stirred at 60 °C or 80 °C for 24 h. The reaction was allowed to cool to room temperature, diluted with water (10 mL) and extracted with ethyl acetate (3 × 10 mL). The organic phase was dried with $MgSO_4$ and concentrated *in vacuo*. The crude product was purified by silica gel column chromatography to afford the product.



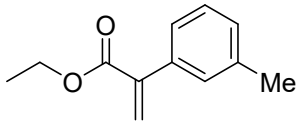
3a

Ethyl 2-phenylacrylate (3a). Flash column chromatography to afford product **3a** as a yellow liquid (25.7 mg, 73% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.42 (m, 2H), 7.37-7.34 (m, 3H), 6.35 (d, J = 1.3 Hz, 1H), 5.89 (d, J = 1.2 Hz, 1H), 4.30 (q, J = 7.1 Hz, 2H), 1.34 (t, J = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 141.6, 136.8, 128.3, 128.0(9), 128.0(5), 126.4, 61.1, 14.2. These data are consistent with the published literature.[3]



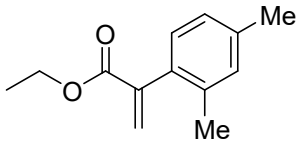
3b

Ethyl 2-(*p*-tolyl)acrylate (3b). Flash column chromatography to afford product **3b** as a colorless liquid (28.2 mg, 74% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.32-7.31 (m, 2H), 7.17 (d, *J* = 7.8 Hz, 2H), 6.30 (d, *J* = 1.3 Hz, 1H), 5.86 (d, *J* = 1.3 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 2.37 (s, 3H), 1.34 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 141.4, 138.0, 133.9, 128.8, 128.1, 125.7, 61.0, 21.2, 14.2. These data are consistent with the published literature.[4]



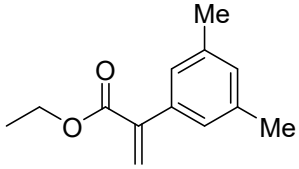
3c

Ethyl 2-(*m*-tolyl)acrylate (3c). Flash column chromatography to afford product **3c** as a colorless liquid (20.9 mg, 55% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.26 – 7.20 (m, 3H), 7.16 – 7.13 (m, 1H), 6.31 (d, *J* = 1.3 Hz, 1H), 5.86 (d, *J* = 1.3 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 2.37 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 141.7, 137.7, 136.7, 128.9, 128.7, 127.9, 126.2, 125.4, 61.1, 21.4, 14.2.These data are consistent with the published literature.[4]



3d

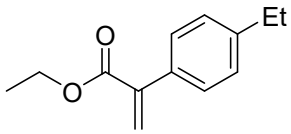
Ethyl 2-(2,4-dimethylphenyl)acrylate (3d). Flash column chromatography to afford product **3d** as a colorless liquid (28.6 mg, 70% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.04 – 6.99 (m, 3H), 6.47 (d, *J* = 1.8 Hz, 1H), 5.67 (d, *J* = 1.8 Hz, 1H), 4.24 (q, *J* = 7.1 Hz, 2H), 2.33 (s, 3H), 2.17 (s, 3H), 1.28 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 142.0, 137.8, 135.9, 134.5, 130.7, 129.4, 128.0, 126.3, 60.9, 21.1, 19.8, 14.2. HRMS (ESI) $[M+Na]^+$: calculated for $C_{13}H_{16}O_2Na$: 227.1048, found 227.1049.



3e

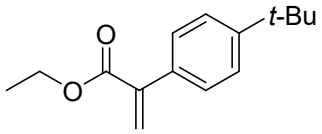
Ethyl 2-(3,5-dimethylphenyl)acrylate (3e). Flash column chromatography to afford product **3e** as a yellow liquid (32.7 mg, 80% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.01 (d, *J* = 25.1 Hz, 3H), 6.29 (d, *J* = 1.4 Hz, 1H), 5.85 (d, *J* = 1.3 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 2.33 (s, 6H), 1.34 (t, *J*

= 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 141.8, 137.6, 136.7, 129.8, 126.1, 125.9, 61.0, 21.3, 14.2. This compound was reported in the published literature.[1b]



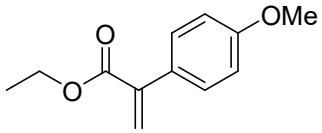
3f

Ethyl 2-(4-ethylphenyl)acrylate (3f). Flash column chromatography to afford product **3f** as a yellow liquid (23.7 mg, 58% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.34 (m, 2H), 7.20 (d, *J* = 8.0 Hz, 2H), 6.30 (d, *J* = 1.3 Hz, 1H), 5.87 (d, *J* = 1.3 Hz, 1H), 4.30 (q, *J* = 7.1 Hz, 2H), 2.67 (q, *J* = 7.7 Hz, 2H), 1.34 (t, *J* = 7.2 Hz, 3H), 1.25 (t, *J* = 7.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 144.3, 141.4, 134.1, 128.2, 127.6, 125.7, 61.0, 28.6, 15.4, 14.2. This compound was reported in the published literature.[5]



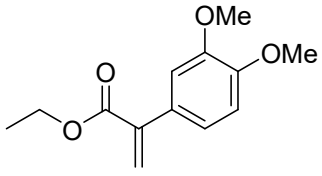
3g

Ethyl 2-(4-(*tert*-butyl)phenyl)acrylate (3g). Flash column chromatography to afford product **3g** as a colorless liquid (38.6 mg, 83% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 (s, 4H), 6.31 (d, *J* = 1.4 Hz, 1H), 5.89 (d, *J* = 1.3 Hz, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 1.35 (t, *J* = 7.0 Hz, 3H).1.34 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 151.1, 141.3, 133.8, 128.0, 125.7, 125.0, 61.0, 34.6, 31.3, 14.2. This compound was reported in the published literature.[1b]



3h

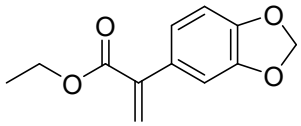
Ethyl 2-(4-methoxyphenyl)acrylate (3h). Flash column chromatography to afford product **3h** as a colorless liquid (24.7 mg, 60% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.32 (m, 2H), 6.90 – 6.87 (m, 2H), 6.25 (d, *J* = 1.2 Hz, 1H), 5.82 (d, *J* = 1.2 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.82 (s, 3H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 159.6, 140.9, 129.5, 129.2, 124.9, 113.5, 61.0, 55.3, 14.2. These data are consistent with the published literature.[6]



3i

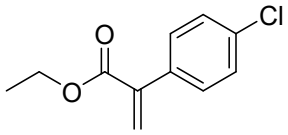
Ethyl 2-(3,4-dimethoxyphenyl)acrylate (3i). Flash column chromatography to afford product **3i** as a yellow liquid (30.2 mg, 64% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.01-6.99 (m, 2H), 6.85 (d,

J = 6.7 Hz, 1H), 6.26 (d, *J* = 1.2 Hz, 1H), 5.84 (d, *J* = 1.3 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 3.89 (s, 6H), 1.34 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.0, 149.1, 148.4, 141.0, 129.5, 125.2, 120.9, 111.6, 110.7, 61.0, 55.9, 55.8, 14.2. This compound was reported in the published literature.[7]



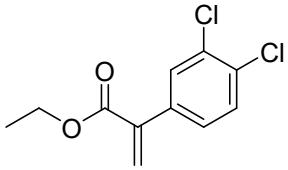
3j

Ethyl 2-(benzo[d][1,3]dioxol-5-yl)acrylate (3j). Flash column chromatography to afford product **3j** as a yellow liquid (33.0 mg, 75% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 6.95 – 6.87 (m, 2H), 6.79 (d, *J* = 8.0 Hz, 1H), 6.25 (s, 1H), 5.97 (s, 2H), 5.81 (s, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 147.6, 147.4, 141.0, 130.8, 125.5, 122.1, 108.9, 107.9, 101.1, 61.1, 14.2. These data are consistent with the published literature.[8]



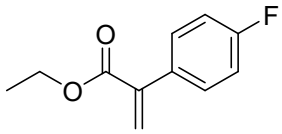
3k

Ethyl 2-(4-chlorophenyl)acrylate (3k). Flash column chromatography to afford product **3k** as a yellow liquid (22.7 mg, 54% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.30 (m, 4H), 6.37 (d, *J* = 1.1 Hz, 1H), 5.89 (d, *J* = 1.1 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.4, 140.4, 135.2, 134.2, 129.7, 128.3, 126.9, 61.2, 14.2. These data are consistent with the published literature.[4]



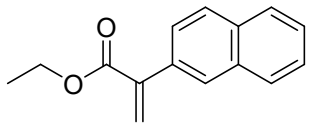
3l

Ethyl 2-(3,4-dichlorophenyl)acrylate (3l). Flash column chromatography to afford product **3l** as a yellow liquid (15.2 mg, 31% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = 2.1 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.28 – 7.26 (m, 1H), 6.41 (d, *J* = 0.9 Hz, 1H), 5.92 (d, *J* = 1.0 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.33 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.0, 139.4, 136.7, 132.3, 132.2, 130.3, 130.0, 128.0 127.7, 61.4, 14.2. These data are consistent with the published literature.[9]



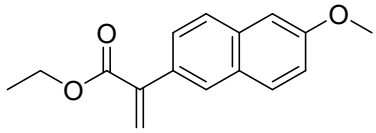
3m

Ethyl 2-(4-fluorophenyl)acrylate (3m). Flash column chromatography to afford product **3m** as a yellow liquid (15.1 mg, 39% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.41 – 7.37 (m, 2H), 7.06 – 7.02 (m, 2H), 6.35 (d, *J* = 1.2 Hz, 1H), 5.86 (d, *J* = 1.2 Hz, 1H), 4.29 (q, *J* = 7.1 Hz, 2H), 1.34 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.6, 162.7 (d, *J* = 252 Hz), 140.5, 132.8 (d, *J* = 2.5 Hz), 130.1 (d, *J* = 8.8 Hz), 126.5, 115.1 (d, *J* = 21.4 Hz), 61.2, 14.2. ^{19}F NMR (471 MHz, $CDCl_3$) δ -113.95. These data are consistent with the published literature.[6]



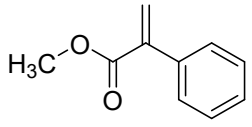
3n

Ethyl 2-(naphthalen-2-yl)acrylate (3n). Flash column chromatography to afford product **3n** as a yellow liquid (23.5 mg, 52% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.93 (d, *J* = 1.8 Hz, 1H), 7.88 – 7.80 (m, 3H), 7.59-7.54 (m, 1H), 7.50-7.48 (m, 2H), 6.44 (d, *J* = 1.2 Hz, 1H), 6.02 (d, *J* = 1.2 Hz, 1H), 4.34 (q, *J* = 7.1 Hz, 2H), 1.39 – 1.34 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.9, 141.6, 134.2, 133.1, 132.9, 128.3, 127.6, 127.5, 127.4, 126.7, 126.3, 126.2, 126.1, 61.2, 14.2. These data are consistent with the published literature.[10]



3o

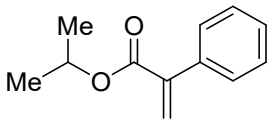
Ethyl 2-(6-methoxynaphthalen-2-yl)acrylate (3o). Flash column chromatography to afford product **3o** as a white solid (31.3 mg, 61% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, *J* = 1.8 Hz, 1H), 7.75 – 7.69 (m, 2H), 7.53 – 7.50 (m, 1H), 7.17 – 7.10 (m, 2H), 6.38 (s, 1H), 5.98 (s, 1H), 4.33 (q, *J* = 7.2 Hz, 2H), 3.93 (s, 3H), 1.35 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 158.0, 141.5, 134.2, 131.9, 129.8, 128.5, 127.3, 126.6, 126.4, 125.9, 119.0, 105.5, 61.1, 55.3, 14.2. These data are consistent with the published literature.[4]



3p

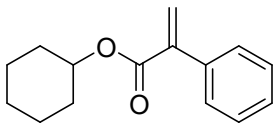
Methyl 2-phenylacrylate (3p). Flash column chromatography to afford product **3p** as a colorless liquid (27.2 mg, 84% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.40 (m, 2H), 7.39 – 7.34 (m,

3H), 6.37 (s, 1H), 5.90 (s, 1H), 3.83 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.3, 141.3, 136.7, 128.3, 128.2, 128.1, 127.0, 52.2. These data are consistent with the published literature.[3]



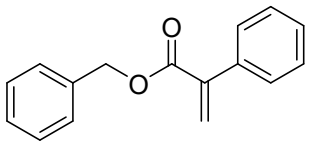
3q

***Iso*-propyl 2-phenylacrylate (3q).** Flash column chromatography to afford product **3q** as a yellow liquid (20.2 mg, 53% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 – 7.42 (m, 2H), 7.41 – 7.32 (m, 3H), 6.33 (s, 1H), 5.89 (s, 1H), 5.23 – 5.13 (m, 1H), 1.34 (d, J = 6.3 Hz, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.4, 141.9, 136.8, 128.3, 128.2, 126.0, 68.6, 21.8. These data are consistent with the published literature.[3]



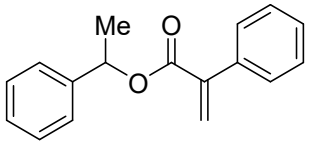
3r

Cyclohexyl 2-phenylacrylate (3r). Flash column chromatography to afford product **3r** as a colorless liquid (30.4 mg, 66% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 – 7.40 (m, 2H), 7.39 – 7.30 (m, 3H), 6.33 (d, J = 1.2 Hz, 1H), 5.87 (t, J = 1.0 Hz, 1H), 4.98 – 4.92 (m, 1H), 1.94 – 1.87 (m, 2H), 1.77 – 1.69 (m, 2H), 1.55 – 1.51 (m, 2H), 1.45 – 1.37 (m, 2H), 1.36 – 1.23 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.2, 141.9, 136.9, 128.3, 128.0, 127.9, 126.0, 73.29, 31.5, 25.4, 23.6. These data are consistent with the published literature.[15]



3s

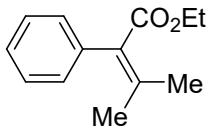
Benzyl 2-phenylacrylate (3s). Flash column chromatography to afford product **3s** as a yellow liquid (33.4 mg, 70% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.42 (m, 2H), 7.39 – 7.33 (m, 8H), 6.40 (s, 1H), 5.92 (s, 1H), 5.28 (s, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.6, 141.2, 136.6, 135.9, 128.7, 128.5, 128.4, 128.3, 128.2, 127.1, 66.8. These data are consistent with the published literature.[3]



3t

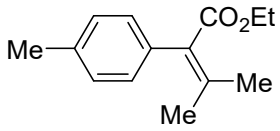
1-Phenylethyl 2-phenylacrylate (3t). Flash column chromatography to afford product **3t** as a colorless liquid (31.7 mg, 63% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.44 – 7.31 (m, 10H), 6.41 (s, 1H), 6.06 (q, J = 6.6 Hz, 1H), 5.92 (s, 1H), 1.63 (d, J = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$)

δ 165.9, 141.6, 141.5, 136.7, 128.5, 128.4, 128.3, 128.1, 128.0, 127.9, 126.6, 126.0, 22.3. HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{16}O_2Na$: 275.1048, found 275.1054.



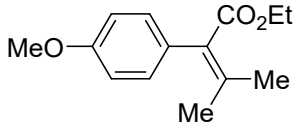
5a

Ethyl 3-methyl-2-phenylbut-2-enoate (5a). Flash column chromatography to afford product **5a** as a yellow liquid (25.3 mg, 62% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.33 (m, 2H), 7.29 – 7.27 (m, 1H), 7.21 – 7.19 (m, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.12 (s, 3H), 1.70 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.8, 143.9, 138.1, 130.3, 129.4, 128.0, 126.9, 60.4, 23.1, 22.5, 14.2. These data are consistent with the published literature.[12]



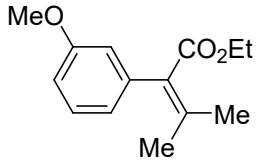
5b

Ethyl 3-methyl-2-(*p*-tolyl)but-2-enoate (5b). Flash column chromatography to afford product **5b** as a colorless liquid (32.3 mg, 74% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16 – 7.11 (m, 2H), 7.09 – 7.06 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.35 (s, 3H), 2.09 (s, 3H), 1.70 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.0, 143.2, 136.5, 135.0, 130.2, 129.3, 128.8, 60.4, 22.9, 22.5, 21.2, 14.2. These data are consistent with the published literature.[4]



5c

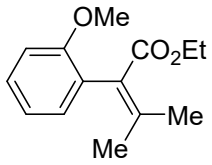
Ethyl 2-(4-methoxyphenyl)-3-methylbut-2-enoate (5c). Flash column chromatography to afford product **5c** as a colorless liquid (29.9 mg, 64% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.12 – 7.09 (m, 2H), 6.88 – 6.85 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.81 (s, 3H), 2.07 (s, 3H), 1.70 (s, 3H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.1, 158.5, 143.1, 130.5, 130.3, 129.9, 113.5, 60.4, 55.2, 22.9, 22.5, 14.2. HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{18}O_3Na$: 257.1154, found 257.1142.



5d

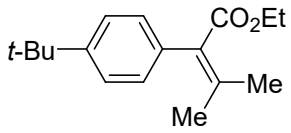
Ethyl 2-(3-methoxyphenyl)-3-methylbut-2-enoate (5d). Flash column chromatography to afford product **5d** as a colorless liquid (30.5 mg, 65% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.27

(m, 1H), 6.87 – 6.74 (m, 3H), 4.20 (q, *J* = 7.1 Hz, 2H), 3.83 (s, 3H), 2.13 (s, 3H), 1.73 (s, 3H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.7, 159.3, 143.9, 139.4, 130.2, 129.0, 121.9, 114.9, 112.5, 60.4, 55.2, 23.1, 22.5, 14.1. These data are consistent with the published literature.[4]



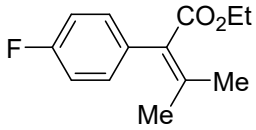
5e

Ethyl 2-(2-methoxyphenyl)-3-methylbut-2-enoate (5e). Flash column chromatography to afford product **5e** as a colorless liquid (27.6 mg, 59% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 – 7.27 (m, 1H), 7.12 – 7.10 (m, 1H), 6.97 – 6.93 (m, 1H), 6.88 (d, *J* = 1.1 Hz, 1H), 4.18 – 4.13 (q, *J* = 7.5 Hz, 2H), 3.78 (s, 3H), 2.20 (s, 3H), 1.74 (s, 3H), 1.18 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.4, 157.1, 146.8, 131.6, 128.5, 127.6, 126.5, 120.2, 110.5, 59.9, 55.4, 23.9, 22.5, 14.2. These data are consistent with the published literature.[4]



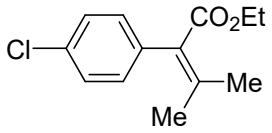
5f

Ethyl 2-(4-(*tert*-butyl)phenyl)-3-methylbut-2-enoate (5f). Flash column chromatography to afford product **5f** as a yellow liquid (48.4 mg, 93% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 2H), 7.12 (d, *J* = 8.3 Hz, 2H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.07 (s, 3H), 1.71 (s, 3H), 1.32 (s, 9H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.2, 149.7, 142.5, 134.8, 130.3, 128.9, 124.9, 60.4, 34.5, 31.3, 22.9, 22.6, 14.2. HRMS (ESI) $[M+H]^+$: calculated for $C_{17}H_{24}O_2$: 261.1852, found 261.1855.



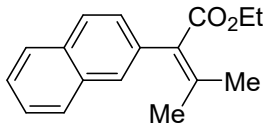
5g

Ethyl 2-(4-fluorophenyl)-3-methylbut-2-enoate (5g). Flash column chromatography to afford product **5g** as a yellow liquid (27.1 mg, 61% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.16 – 7.11 (m, 2H), 7.03 – 6.99 (m, 2H), 4.15 (q, *J* = 7.1 Hz, 2H), 2.11 (s, 3H), 1.67 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.5, 161.8 (d, *J* = 245.7 Hz), 144.9, 134.1 (d, *J* = 2.5 Hz), 131.1 (d, *J* = 7.6 Hz), 129.3, 115.1 (d, *J* = 21.5 Hz), 60.4, 23.2, 22.5, 14.2. ^{19}F NMR (471 MHz, CDCl3) δ -115.58. HRMS (ESI) $[M+H]^+$: calculated for $C_{13}H_{15}FO_2$: 223.1134, found 223.1137.



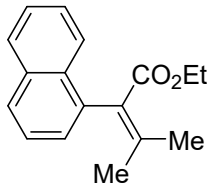
5h

Ethyl 2-(4-chlorophenyl)-3-methylbut-2-enoate (5h). Flash column chromatography to afford product **5h** as a colorless liquid (24.3 mg, 51% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.29 (m, 2H), 7.14 – 7.10 (m, 2H), 4.16 (q, *J* = 7.1 Hz, 2H), 2.13 (s, 3H), 1.70 (s, 3H), 1.21 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.2, 145.5, 132.6, 130.8, 129.1, 128.2, 60.5, 23.3, 22.6, 14.2.These data are consistent with the published literature.[4]



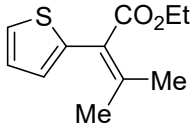
5i

Ethyl 3-methyl-2-(naphthalen-2-yl)but-2-enoate (5i). Flash column chromatography to afford product **5i** as a yellow liquid (30.9 mg, 61% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.84 – 7.75 (m, 3H), 7.65 (d, *J* = 1.7 Hz, 1H), 7.51 – 7.42 (m, 2H), 7.32 (d, *J* = 1.7 Hz, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 2.17 (s, 3H), 1.74 (s, 3H), 1.20 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.7, 144.7, 135.6, 133.2, 132.3, 130.2, 128.2, 127.9, 127.8, 127.6, 127.5, 125.9, 125.8, 60.4, 23.3, 22.6, 14.2. These data are consistent with the published literature.[13]



5j

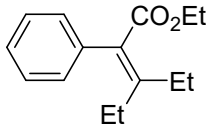
Ethyl 3-methyl-2-(naphthalen-1-yl)but-2-enoate (5j). Flash column chromatography to afford product **5j** as a colorless liquid (31.0 mg, 61% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 – 7.79 (m, 3H), 7.48 – 7.45 (m, 3H), 7.31 – 7.29 (m, 1H), 4.10 – 4.05 (m, 2H), 2.30 (s, 3H), 1.54 (s, 3H), 1.07 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.3, 147.9, 136.2, 133.6, 132.2, 128.2, 127.9, 127.5, 127.3, 125.9, 125.6, 125.5, 125.4, 60.2, 23.8, 22.4, 14.1. HRMS (ESI) $[M+Na]^+$: calculated for $C_{17}H_{18}O_2Na$: 277.1024, found 277.1203.



5k

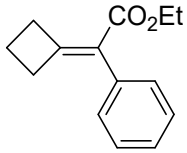
Ethyl 3-methyl-2-(thiophen-2-yl)but-2-enoate (5k). Flash column chromatography to afford product **5k** as a yellow liquid (19.3 mg, 46% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.29 – 7.27 (m, 1H), 7.00 – 6.98 (m, 1H), 6.87 – 6.83 (m, 1H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.09 (s, 3H), 1.86 (s, 3H),

1.26 (t, J = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.2, 146.0, 138.5, 127.2, 126.5, 125.5, 123.6, 60.7, 23.3, 22.9, 14.1. HRMS (ESI) $[M+Na]^+$: calculated for $C_{11}H_{14}O_2SNa$: 233.0612, found 233.0613.



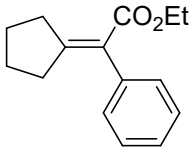
5l

Ethyl 3-ethyl-2-phenylpent-2-enoate (5l). Flash column chromatography to afford product **5l** as a colorless liquid (32.5 mg, 70% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.32 (m, 2H), 7.31 – 7.26 (m, 1H), 7.23 – 7.19 (m, 2H), 4.15 (q, J = 7.1 Hz, 2H), 2.45 (q, J = 7.5 Hz, 2H), 2.00 (q, J = 7.5 Hz, 2H), 1.22 (t, J = 7.1 Hz, 3H), 1.16 (t, J = 7.5 Hz, 3H), 0.95 (t, J = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.8, 153.5, 137.9, 129.9, 129.3, 128.0, 126.9, 60.3, 25.6, 14.1, 13.3, 12.7. HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{20}O_2Na$: 255.1361, found 255.1263.



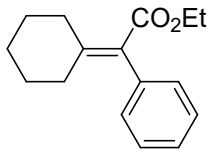
5m

Ethyl 2-cyclobutylidene-2-phenylacetate (5m). Flash column chromatography to afford product **5m** as a yellow liquid (21.6 mg, 50% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.32 (m, 2H), 7.28-7.23 (m, 3H), 4.22 (q, J = 7.1 Hz, 2H), 3.29 – 3.24 (m, 2H), 2.82 – 2.78 (m, 2H), 2.10-2.04 (m, 2H), 1.59 (s, 3H), 1.29 (t, J = 7.1 Hz, 4H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.8, 162.4, 135.7, 129.2, 127.9, 126.9, 125.9, 60.2, 34.4, 32.5, 17.2, 14.4. HRMS (ESI) $[M+Na]^+$: calculated for $C_{14}H_{16}O_2Na$: 239.1048, found 239.1044.



5n

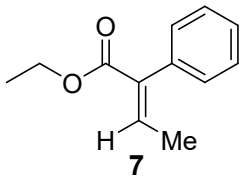
Ethyl 2-cyclopentylidene-2-phenylacetate (5n). Flash column chromatography to afford product **5n** as a yellow liquid (25.3 mg, 55% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.34 (m, 2H), 7.29 – 7.27 (m, 1H), 7.19 – 7.17 (m, 2H), 4.20 – 4.16 (q, J = 7.0 Hz , 2H), 2.91 – 2.88 (m, 2H), 2.24 – 2.21 (m, 2H), 1.82-1.76 (m, 2H), 1.63-1.58 (m, 3H), 1.24 (t, J = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.5, 162.8, 139.1, 129.2, 127.9, 126.7, 125.8, 60.1, 35.2, 34.0, 26.8, 25.8, 14.3. HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{18}O_2Na$: 253.1204, found 253.1205.



5o

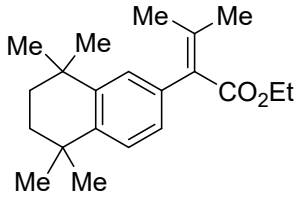
Ethyl 2-cyclohexylidene-2-phenylacetate (5o). Flash column chromatography to afford product **5o** as a light yellow liquid (24.4 mg, 50% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.33 (m,

2H), 7.29 – 7.27 (m, 1H), 7.24 – 7.20 (m, 2H), 4.20 – 4.16 (q, *J* = 7.0 Hz, 2H), 2.55 – 2.53 (m, 2H), 2.12 – 2.09 (m, 2H), 1.74 – 1.70 (m, 2H), 1.64 – 1.55 (m, 4H), 1.24 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.2, 148.9, 137.4, 129.4, 128.1, 127.7, 126.9, 60.4, 32.6, 32.0, 28.2(5), 28.2(2), 26.3, 14.1. These data are consistent with the published literature.[14]



7

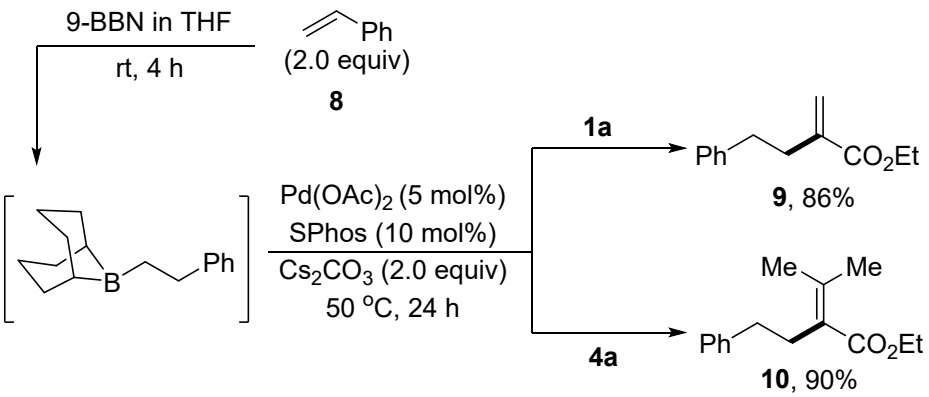
Ethyl (*E*)-2-phenylbut-2-enoate (7). Flash column chromatography to afford product **7** as a light yellow liquid (30.8 mg, 81% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.35 (m, 2H), 7.33 – 7.29 (m, 1H), 7.20 – 7.14 (m, 3H), 4.21 (q, *J* = 7.1 Hz, 2H), 1.75 (d, *J* = 7.3 Hz, 3H), 1.26 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 139.6, 135.1, 134.9, 129.8, 127.9, 127.3, 60.7, 15.4, 14.2. These data are consistent with the published literature.[15]



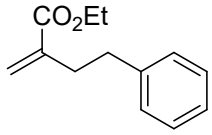
12

Ethyl 3-methyl-2-(5,5,8,8-tetramethyl-5,6,7,8-tetrahydronaphthalen-2-yl)but-2-enoate (12). Flash column chromatography to afford product **12** as a colorless liquid (45.9 mg, 73% yield). ^{1}H NMR (500 MHz,$CDCl_3$) δ 7.23 (d, *J* = 8.1 Hz, 1H), 7.12 (d, *J* = 1.9 Hz, 1H), 6.98 – 6.94 (m, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.04 (s, 3H), 1.72 (s, 3H), 1.68 (s, 4H), 1.28 (s, 6H), 1.26 (s, 6H), 1.25 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.4, 144.2, 143.3, 141.4, 134.4, 130.8, 127.6, 126.4, 125.9, 60.4, 35.1(2), 35.1(0), 34.2, 34.0, 31.8, 22.7, 22.6, 14.2. These data are consistent with the published literature.[16]

3.2 General Procedure of the Alkylation Reactions of α-Arylsulfonyloxyacrylates

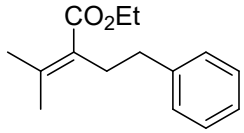


One Schlenk tube equipped with a magnetic stir bar was charged with styrene **8** (208 mg, 2 mmol, 1.0 equiv) and a solution of 9-BBN in THF (4 mL, 0.5 M, 1.0 equiv), stirred for 4 h at room temperature to afford a solution of *B*-phenylethyl-9-BBN in THF. Another Schlenk tube equipped with a magnetic stir bar was charged with **1a** (109.6 mg, 0.4 mmol, 1.0 equiv) or **4a** (120.8 mg, 0.4 mmol, 1.0 equiv), $Pd(OAc)_2$ (4.5 mg, 0.02 mmol), SPhos (16.4 mg, 0.04 mmol), Cs_2CO_3 (260.8 mg, 0.8 mmol, 2.0 equiv) and the solution of *B*-phenylethyl-9-BBN in THF (1.6 mL, prepared above). The mixture was stirred at 50 °C for 24 h. After completed, the reaction mixture was cooled down to room temperature, diluted with ethyl acetate (2 mL), and pushed through a plug of silica gel with ethyl acetate. The filtrate was concentrated under reduced pressure. The residue was chromatographed on silica gel column to give the target product **9** (70.2 mg, 86% yield) or **10** (83.6 mg, 90% yield).



9

Ethyl 2-methylene-4-phenylbutanoate (9). Flash column chromatography to afford product **9** as a yellow liquid (35.1 mg, 86% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34 – 7.30 (m, 2H), 7.25 – 7.21 (m, 3H), 6.19 (d, *J* = 1.4 Hz, 1H), 5.53 (q, *J* = 1.3 Hz, 1H), 4.26 (q, *J* = 7.2 Hz, 2H), 2.83 (d, *J* = 8.4 Hz, 2H), 2.70 – 2.64 (m, 2H), 1.35 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 167.1, 141.4, 140.1, 128.5, 128.3, 125.9, 125.1, 60.6, 34.9, 33.9, 14.2. These data are consistent with the published literature.[17]



10

Ethyl 3-methyl-2-phenethylbut-2-enoate (10). Flash column chromatography to afford product **10** as a colorless liquid (41.8 mg, 90% yield). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.27 (m, 2H), 7.22 – 7.19 (m, 3H), 4.21 (q, *J* = 7.1 Hz, 2H), 2.73 – 2.69 (m, 2H), 2.63 – 2.57 (m, 2H), 2.00 (s, 3H), 1.73 (s, 3H), 1.32 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 169.5, 143.1, 141.8,

128.5, 128.2, 127.0, 125.8, 59.9, 35.3, 32.2, 22.9, 21.7, 14.3. HRMS (ESI) $[M+Na]^+$: calculated for $C_{15}H_{20}O_2Na$: 255.1361, found 255.1360.

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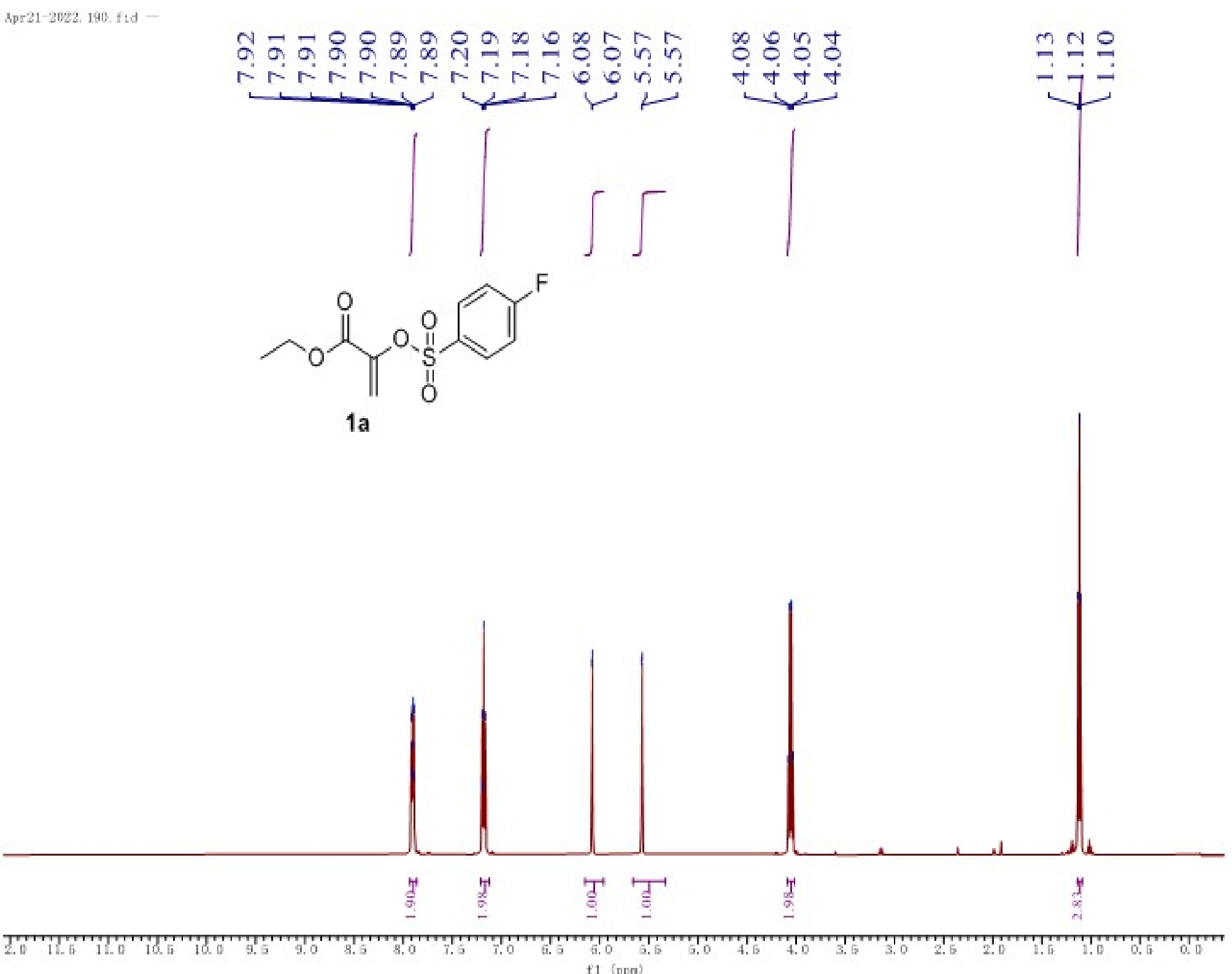
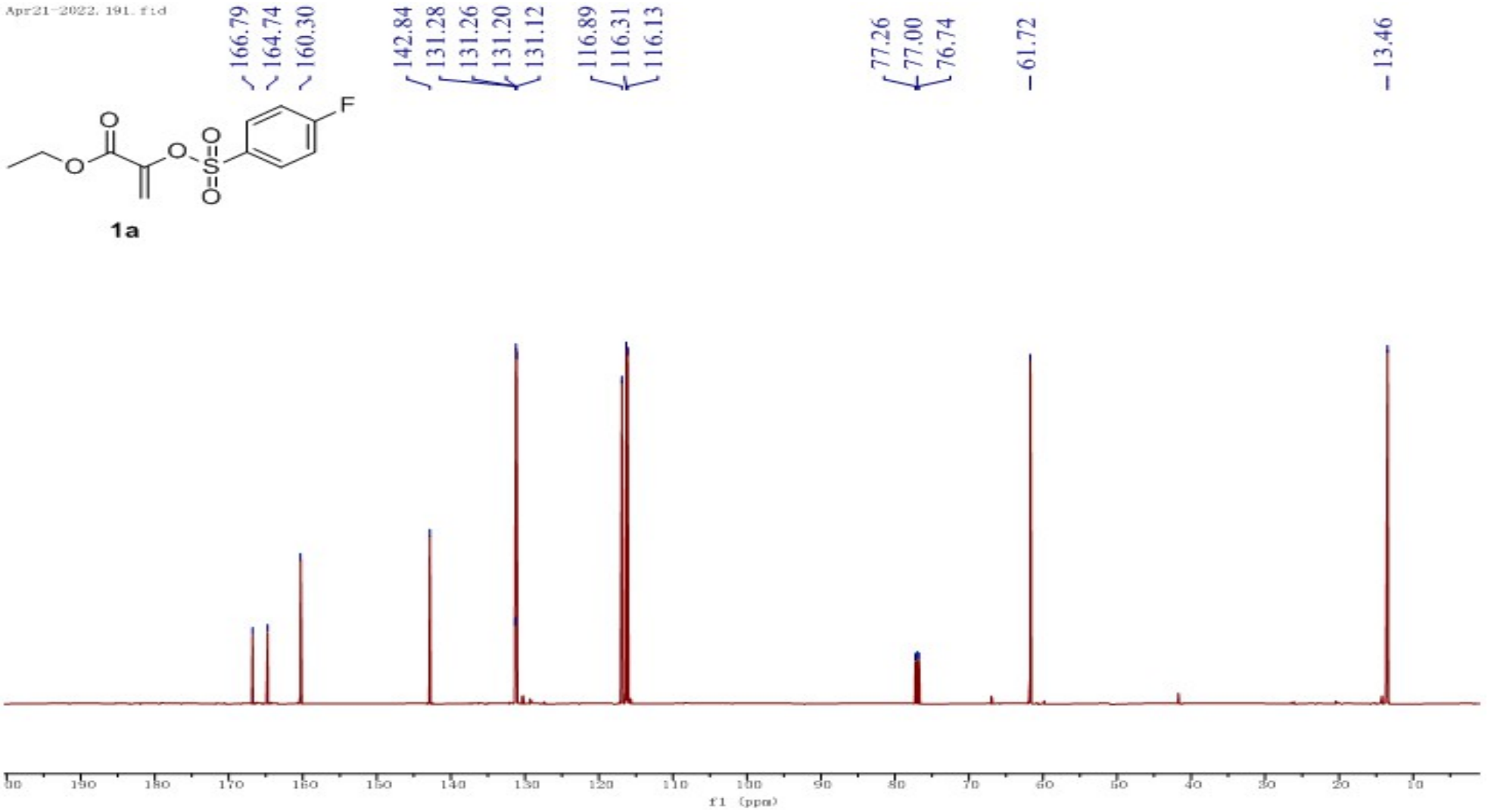
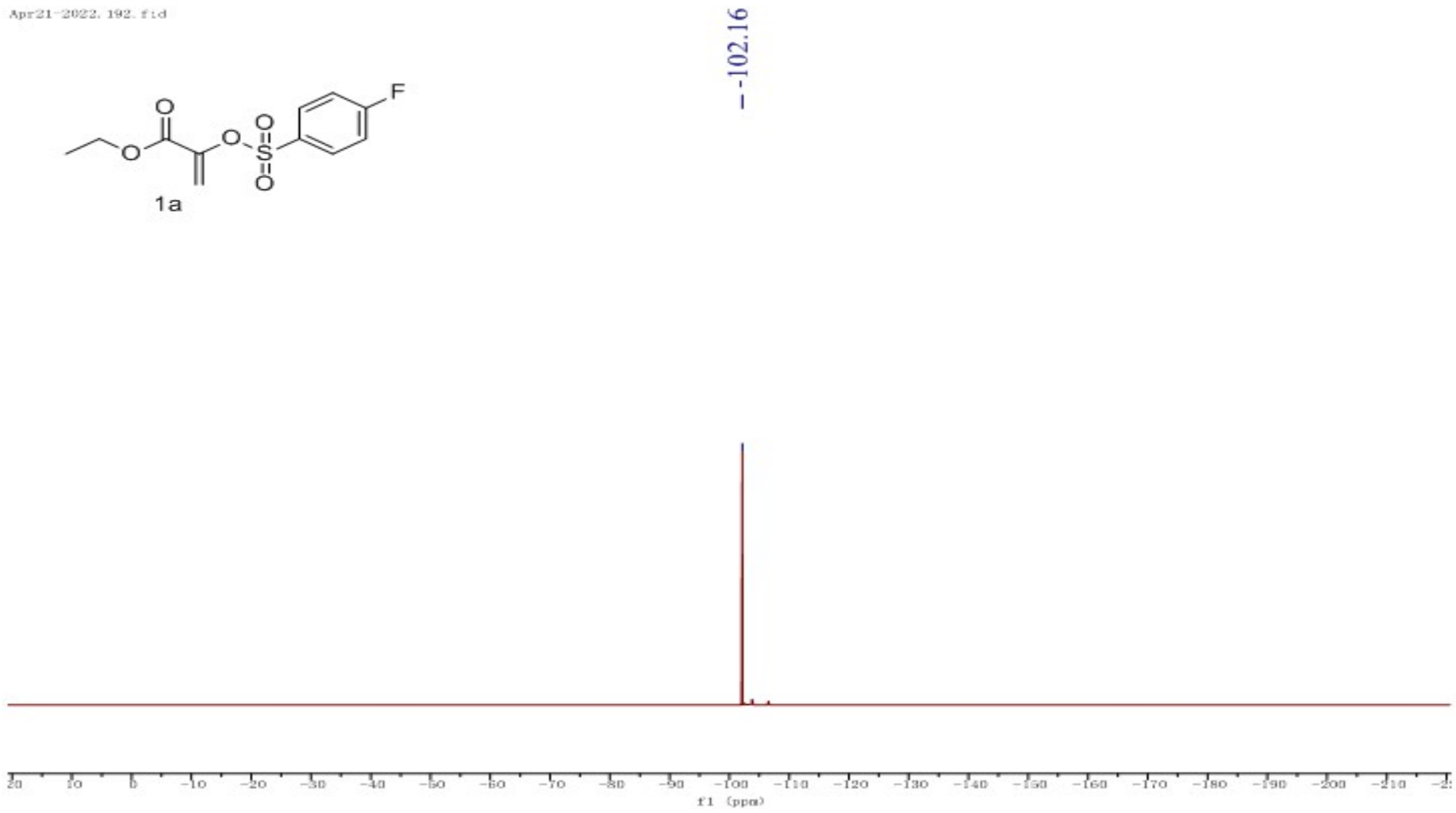
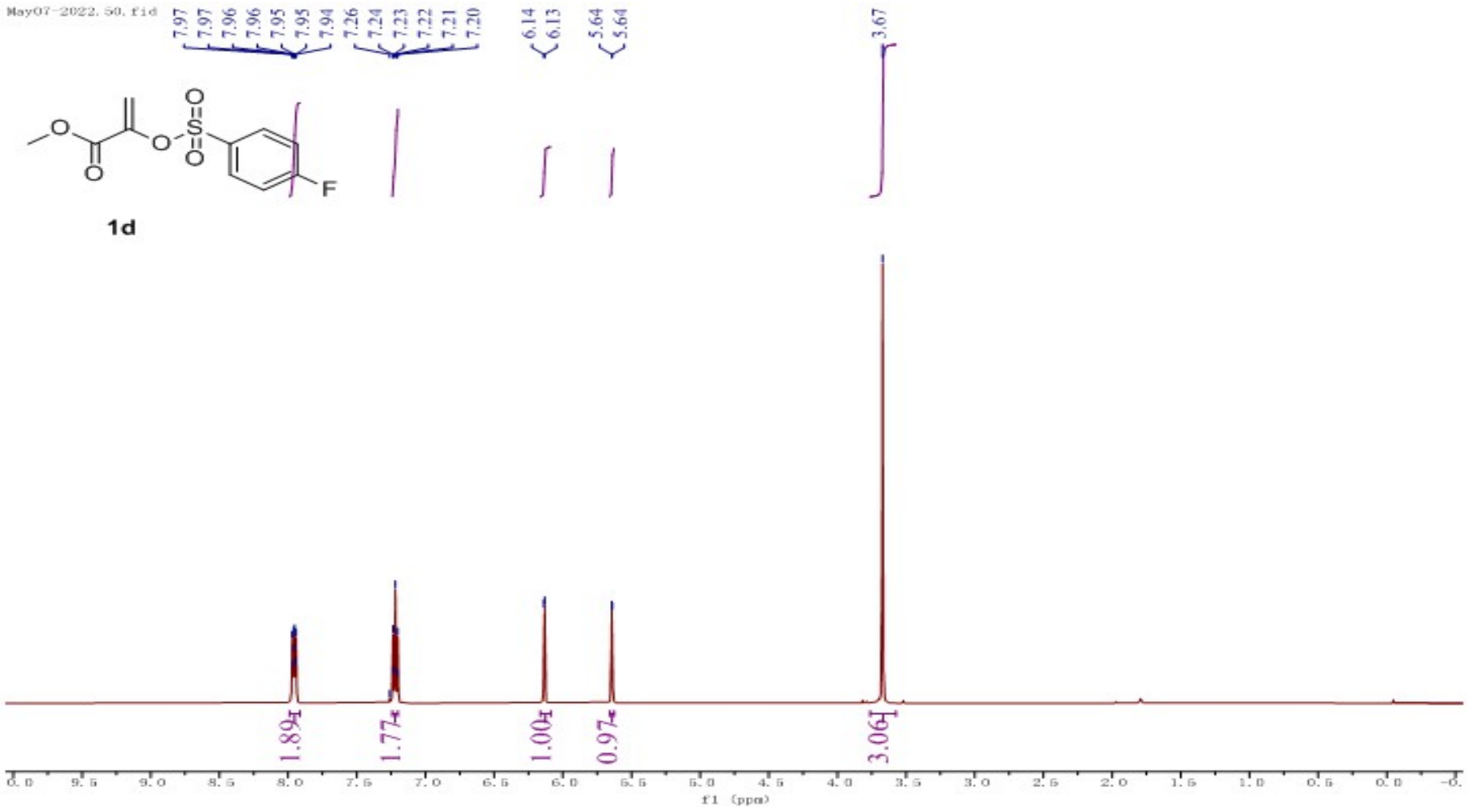
5 NMR Spectra of New Compounds


Apr21-2022.190.fid
7.92
7.91
7.91
7.90
7.90
7.89
7.89
7.20
7.19
7.18
7.16
6.08
6.07
5.57
5.57
4.08
4.06
4.05
4.04
1.13
1.12
1.10
F
O
O
O
O
O
S
O
1a
1.90
1.98
1.00
1.01
1.98
2.83
f1 (ppm)


Apr21-2022.191.fid
166.79
164.74
160.30
142.84
131.28
131.26
131.20
131.12
116.89
116.31
116.13
77.26
77.00
76.74
61.72
13.46
F
O
O
O
O
S
O
1a
f1 (ppm)


Apr21-2022.192.fid
-102.16
F
O
O
O
O
S
O
1a
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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7.97
7.96
7.96
7.95
7.95
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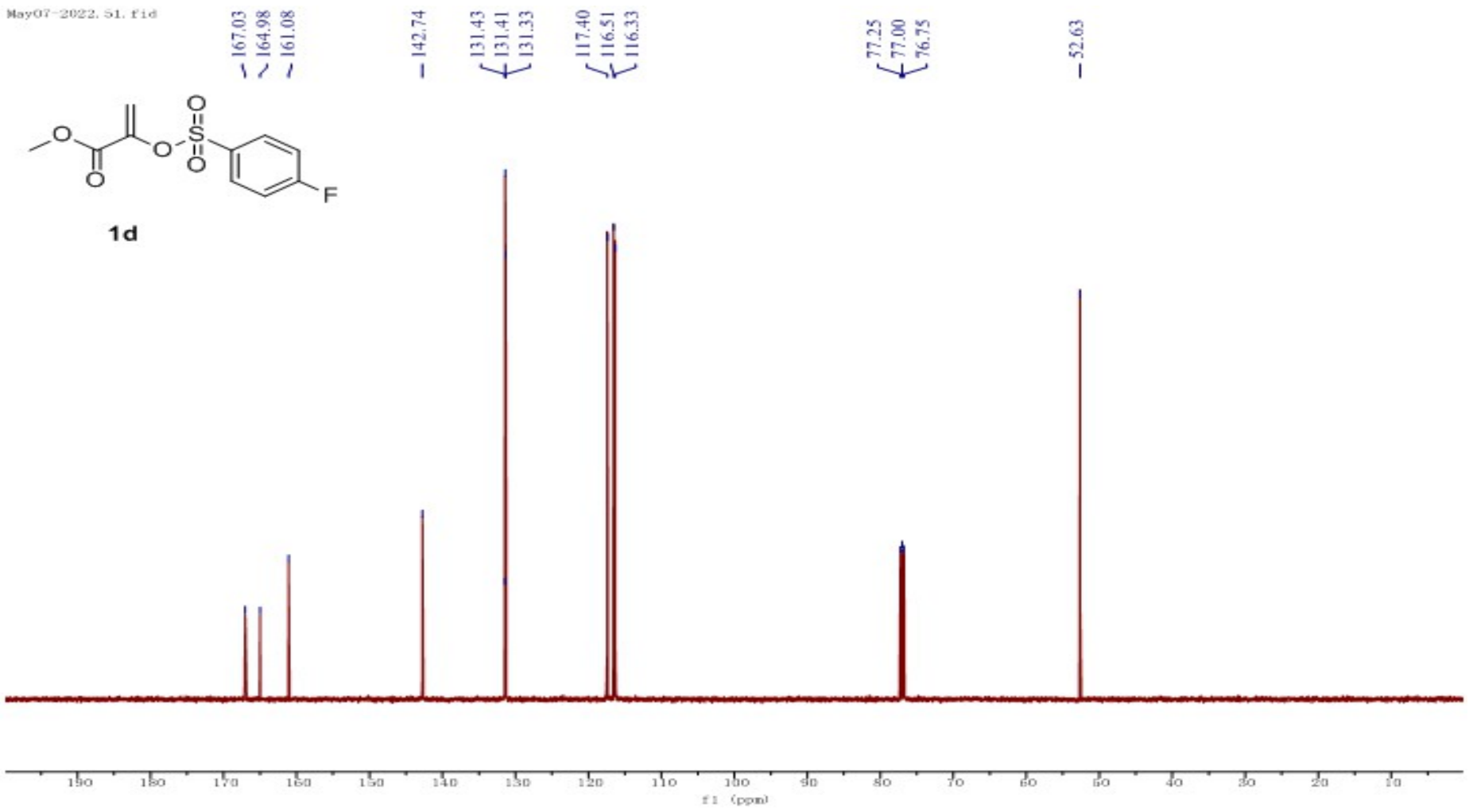
7.26
7.24
7.23
7.22
7.21
7.20
6.14
6.13
5.64
5.64
3.67
O
O
O
O
S
O
F
1d
1.89
1.77
1.00
0.97
3.06
f1 (ppm)


May07-2022.51.fid
167.03
164.98
161.08
142.74
131.43
131.41
131.33
117.40
116.51
116.33
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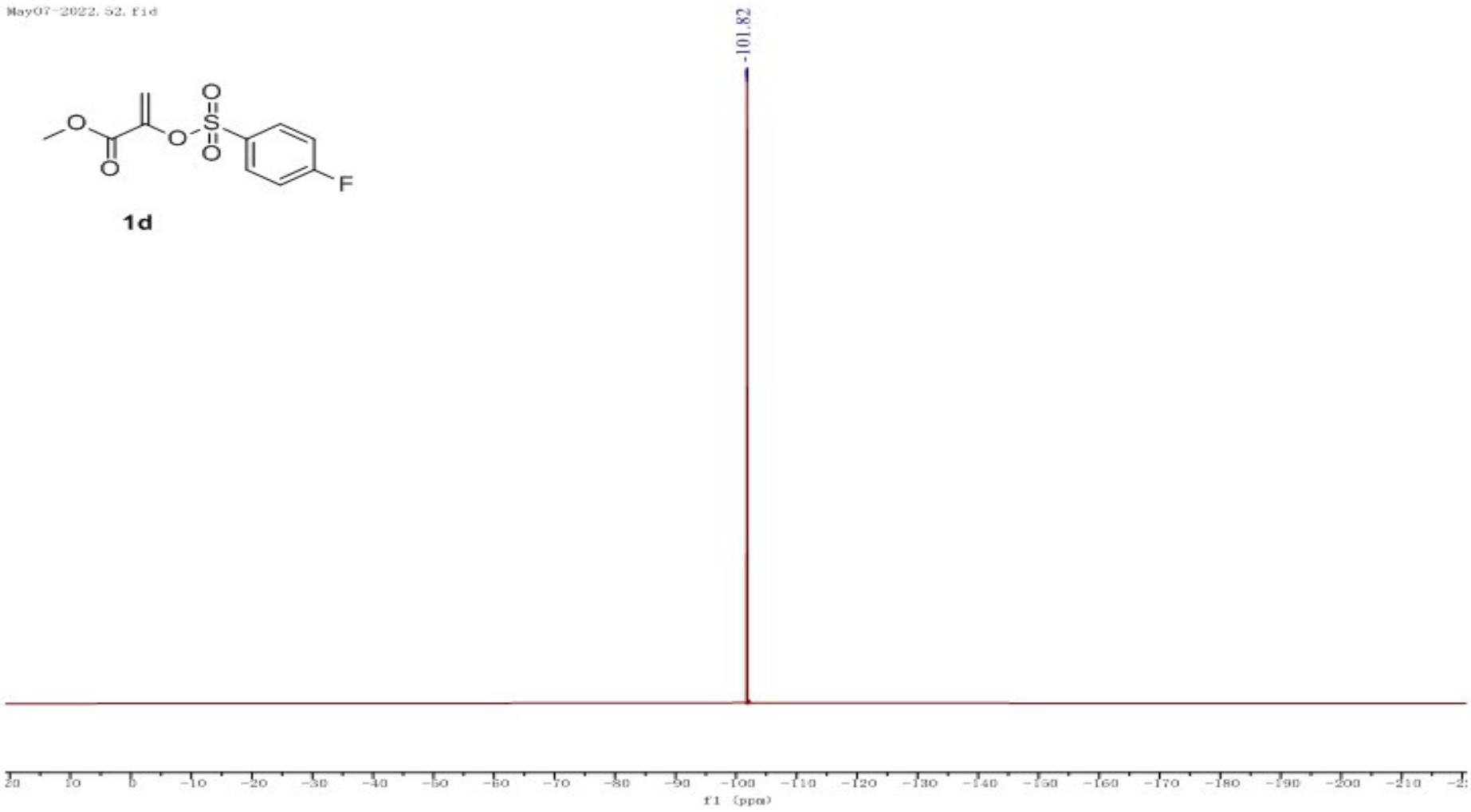
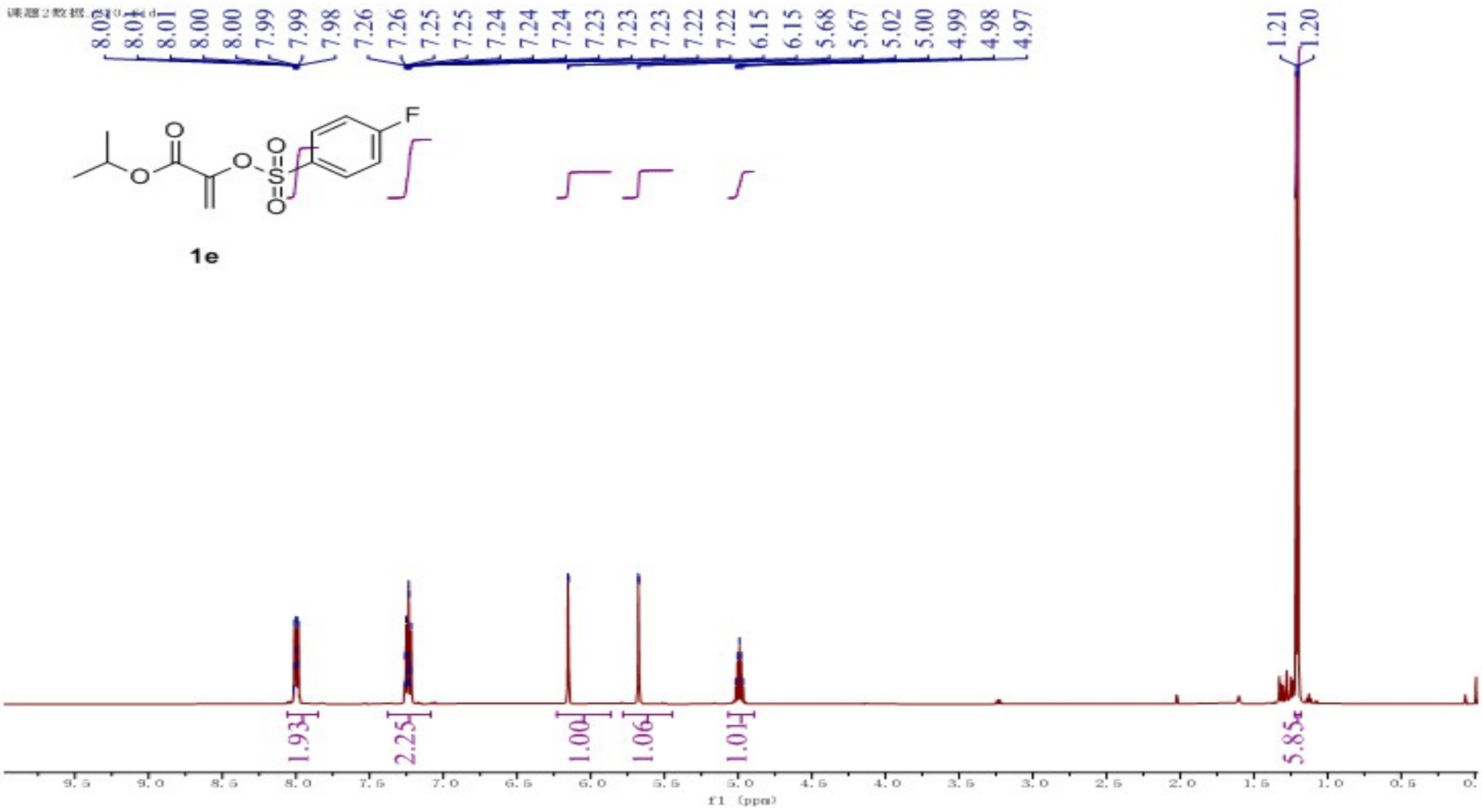
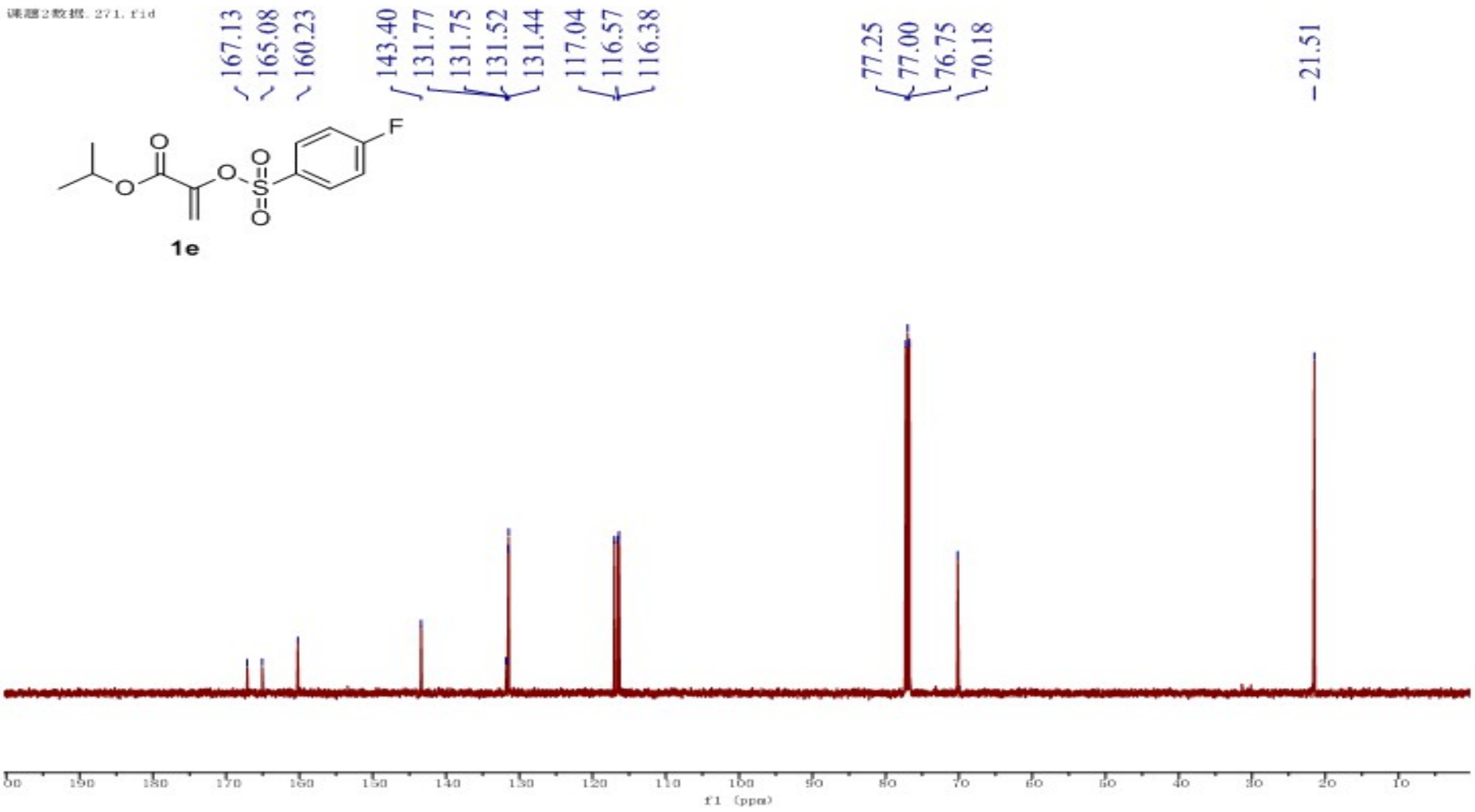
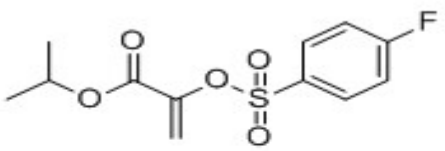
77.00
76.75
52.63
1d
f1 (ppm)


May07-2022.52.fid
-101.82
1d
f1 (ppm)

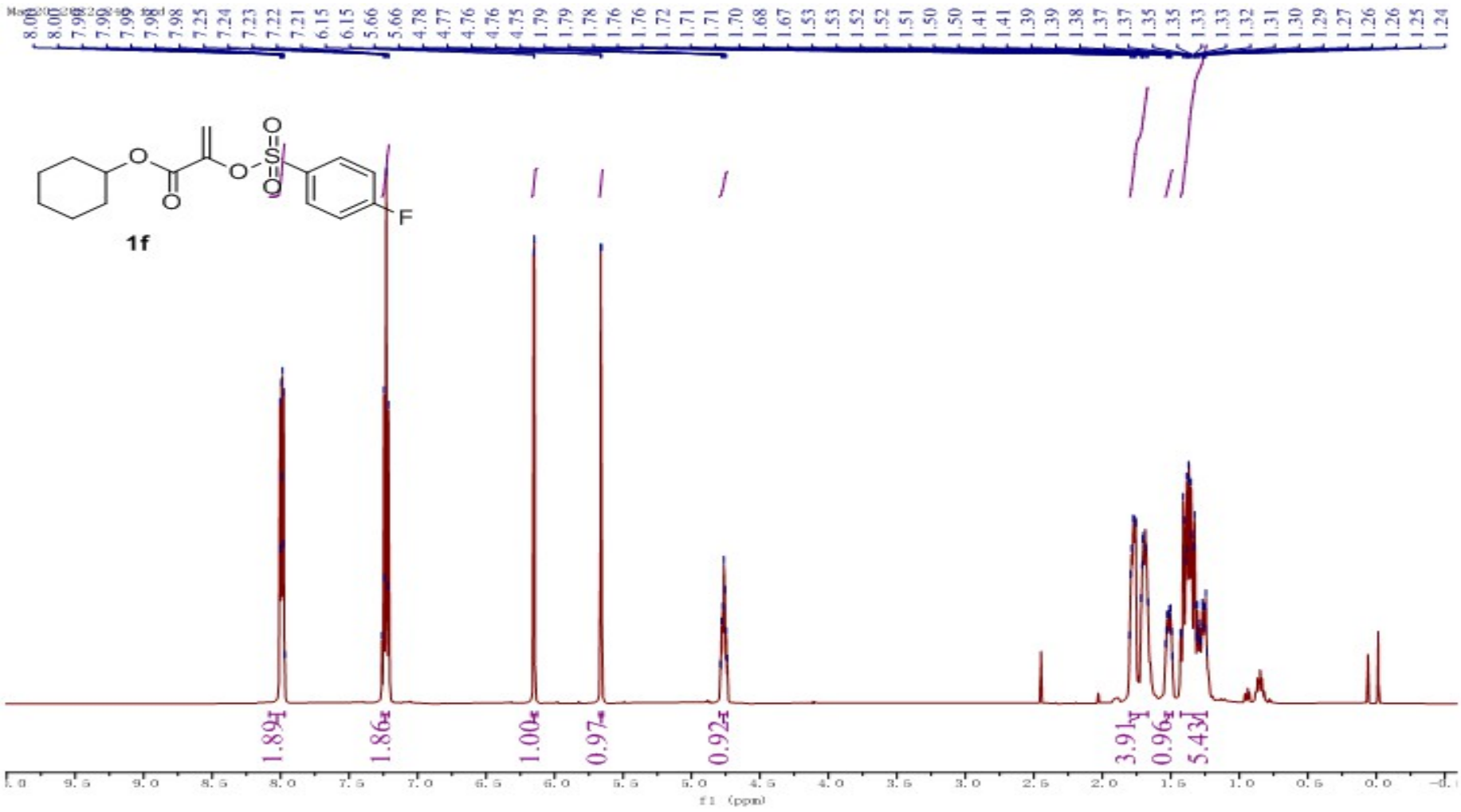

8.02
8.01
8.01
8.00
8.00
7.99
7.99
7.98
7.26
7.26
7.25
7.25
7.24
7.24
7.24
7.23
7.23
7.23
7.22
7.22
6.15
6.15
5.68
5.67
5.02
5.00
4.99
4.98
4.97
1.21
1.20
F
O
O
O
S
O
O
1e
1.93
2.25
1.00
1.06
1.01
5.85
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.
f1 (ppm)

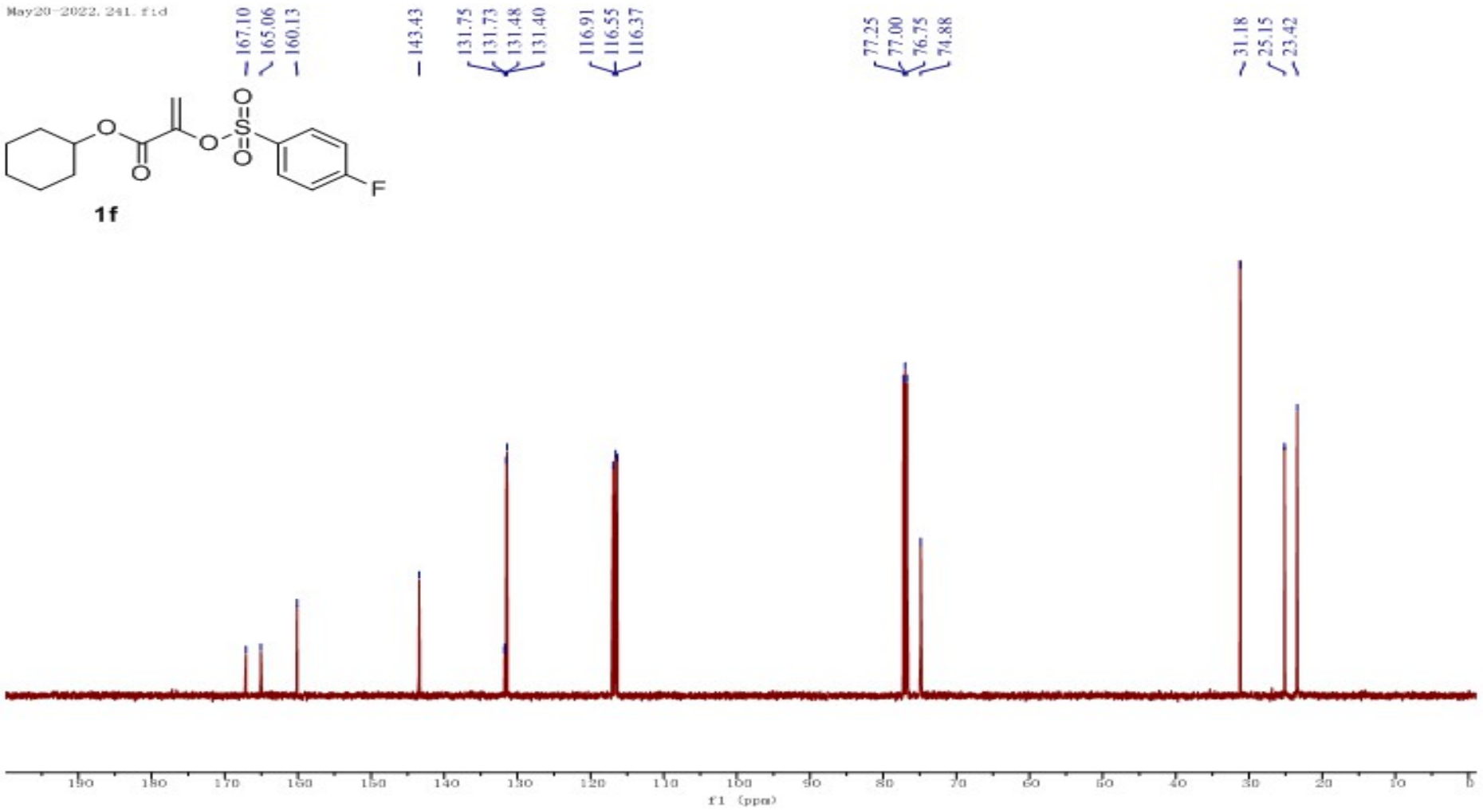

课题2数据. 271. fid
167.13
165.08
160.23
143.40
131.77
131.75
131.52
131.44
117.04
116.57
116.38
77.25
77.00
76.75
70.18
21.51
F
O
O
O
O
O=S=O
1e
f1 (ppm)

课题2数据. 272. fid



1e


1f
F
1.89
1.86
1.00
0.97
0.92
3.91
0.96
5.43
f1 (ppm)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165.06
160.13
143.43
131.75
131.73
131.48
131.40
116.91
116.55
116.37
77.25
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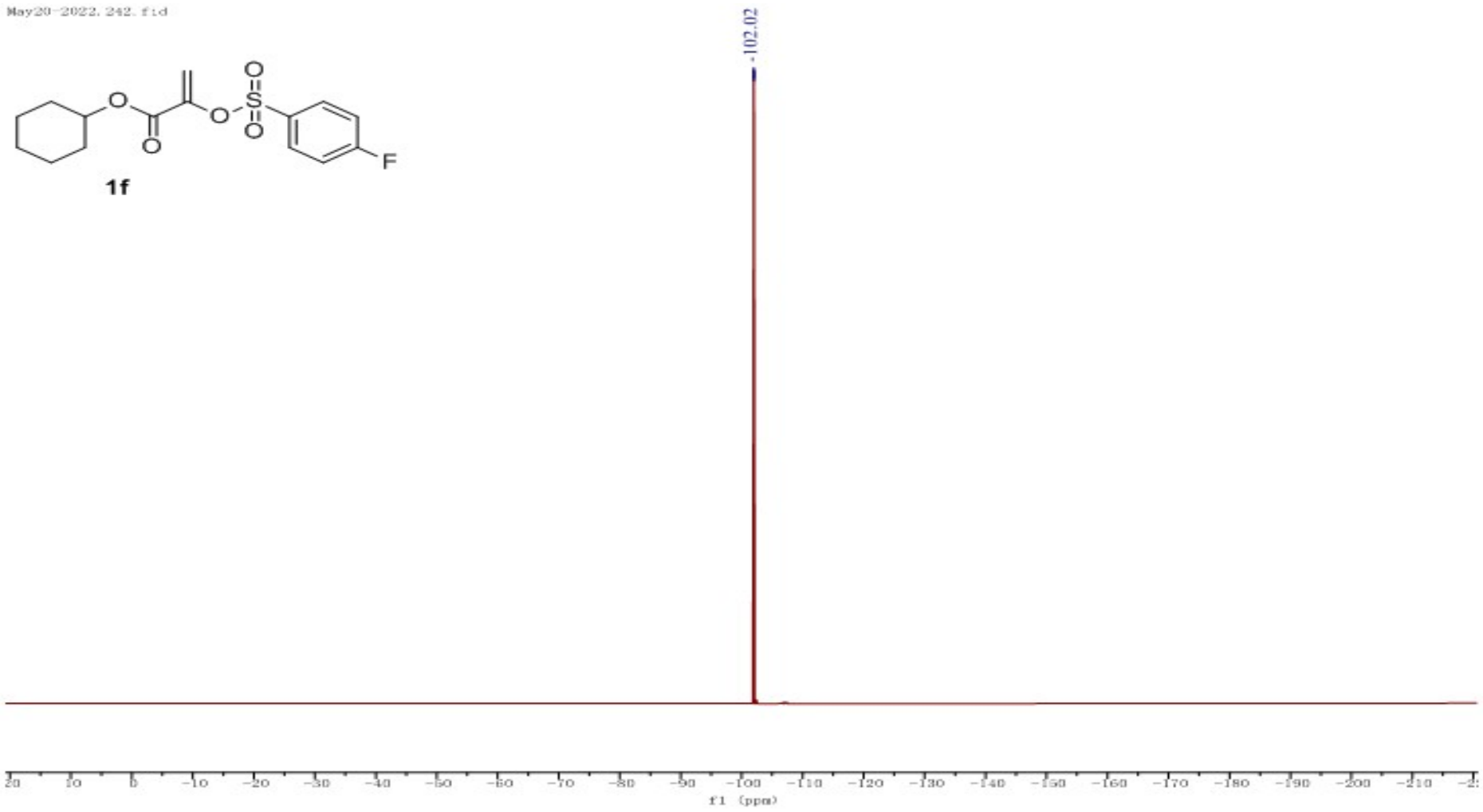
O
O
O
O
S
O
F
1f
f1 (ppm)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-102.02
O
O
O
O
S
O
F
1f
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10
0
-10
-20
-30
-40
-50
-60
-70
-80
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-120
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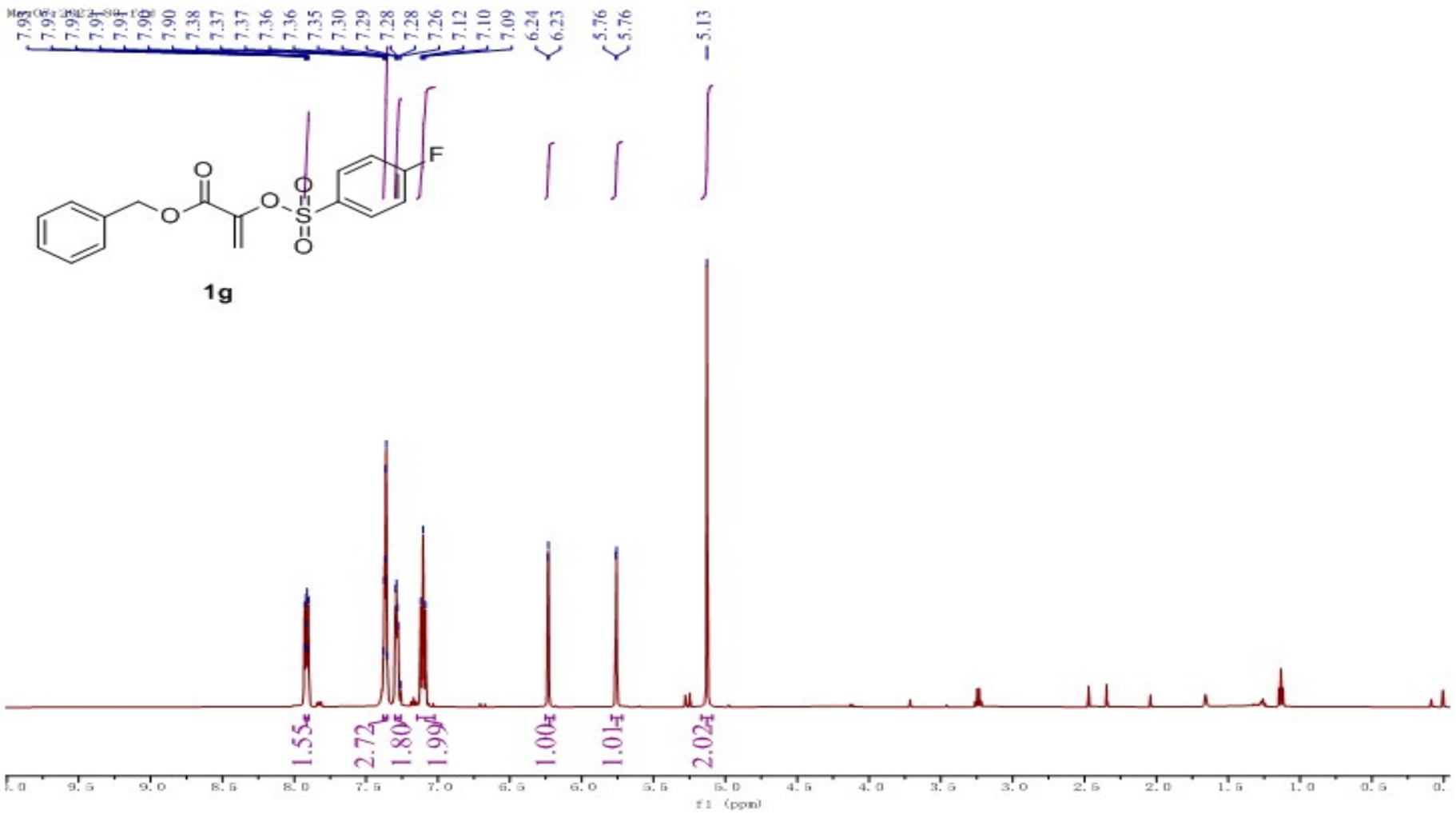
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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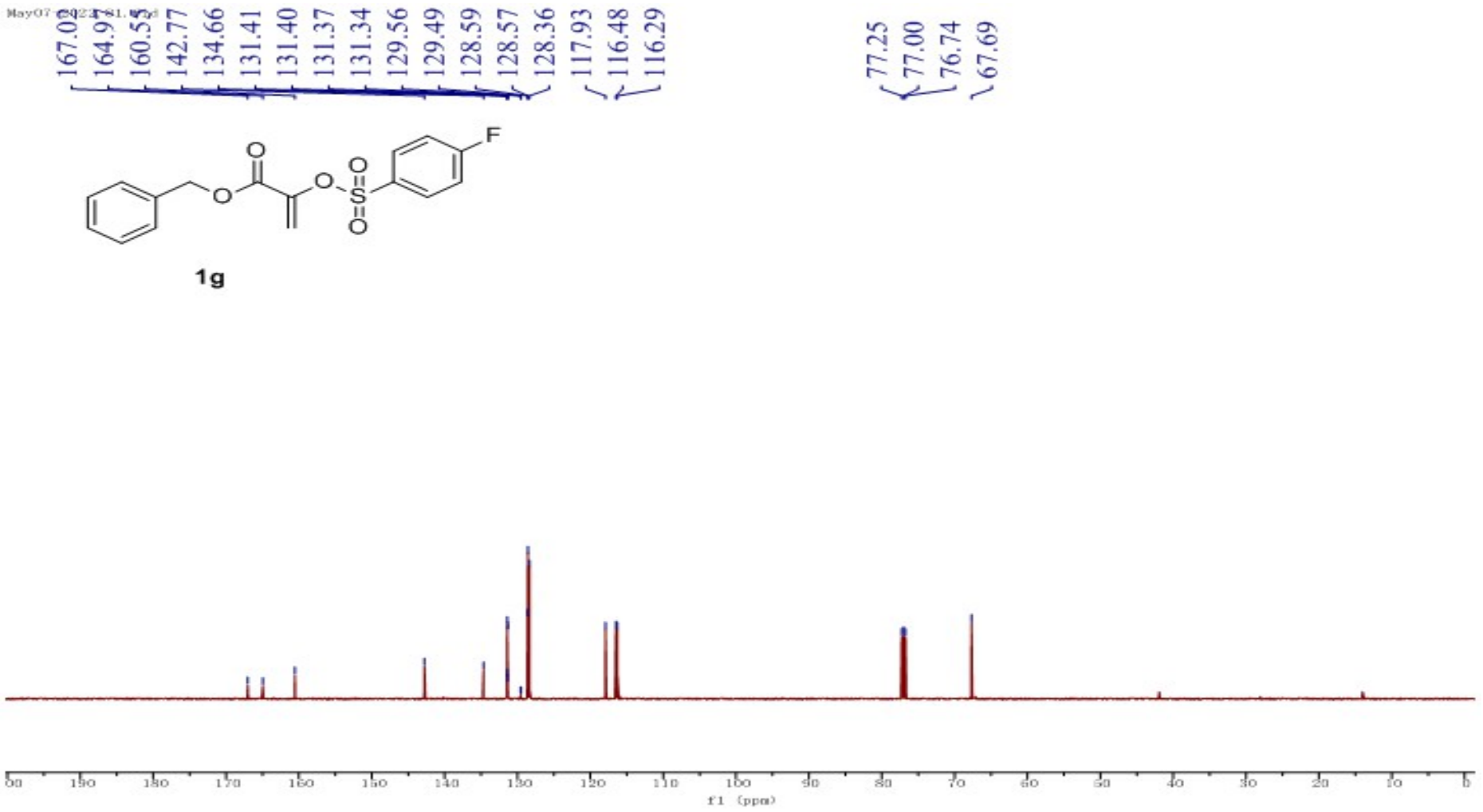
7.36
7.35
7.30
7.29
7.28
7.28
7.26
7.12
7.10
7.09
6.24
6.23
5.76
5.76
5.13
F
O
O
O
O
S
O
O
1g
1.55
2.72
1.80
1.99
1.00
1.01
2.02
f1 (ppm)


167.02
164.97
160.55
142.77
134.66
131.41
131.40
131.37
131.34
129.56
129.49
128.59
128.57
128.36
117.93
116.48
116.29
77.25
77.00
76.74
67.69
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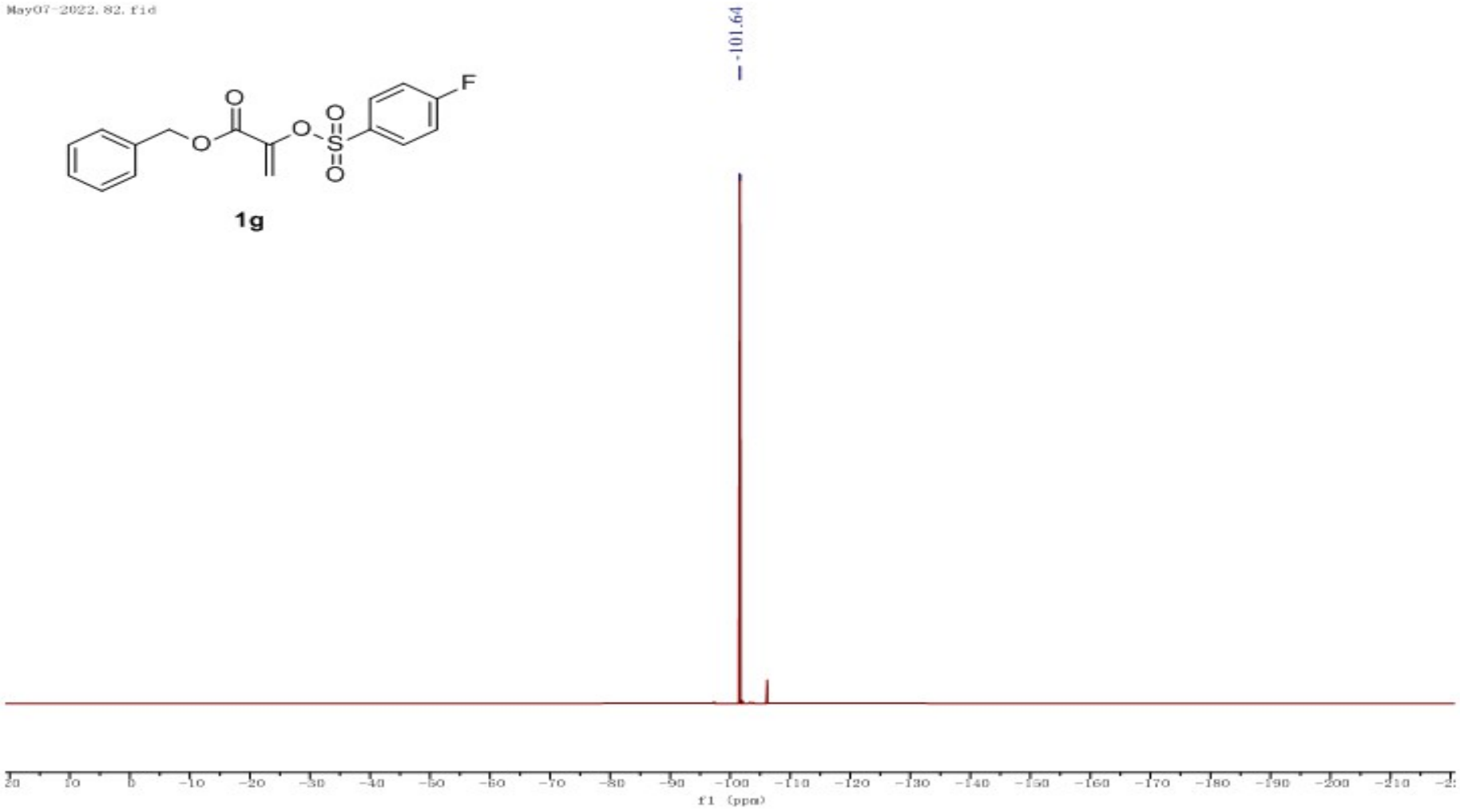
O
O
O
S
O
O
1g
f1 (ppm)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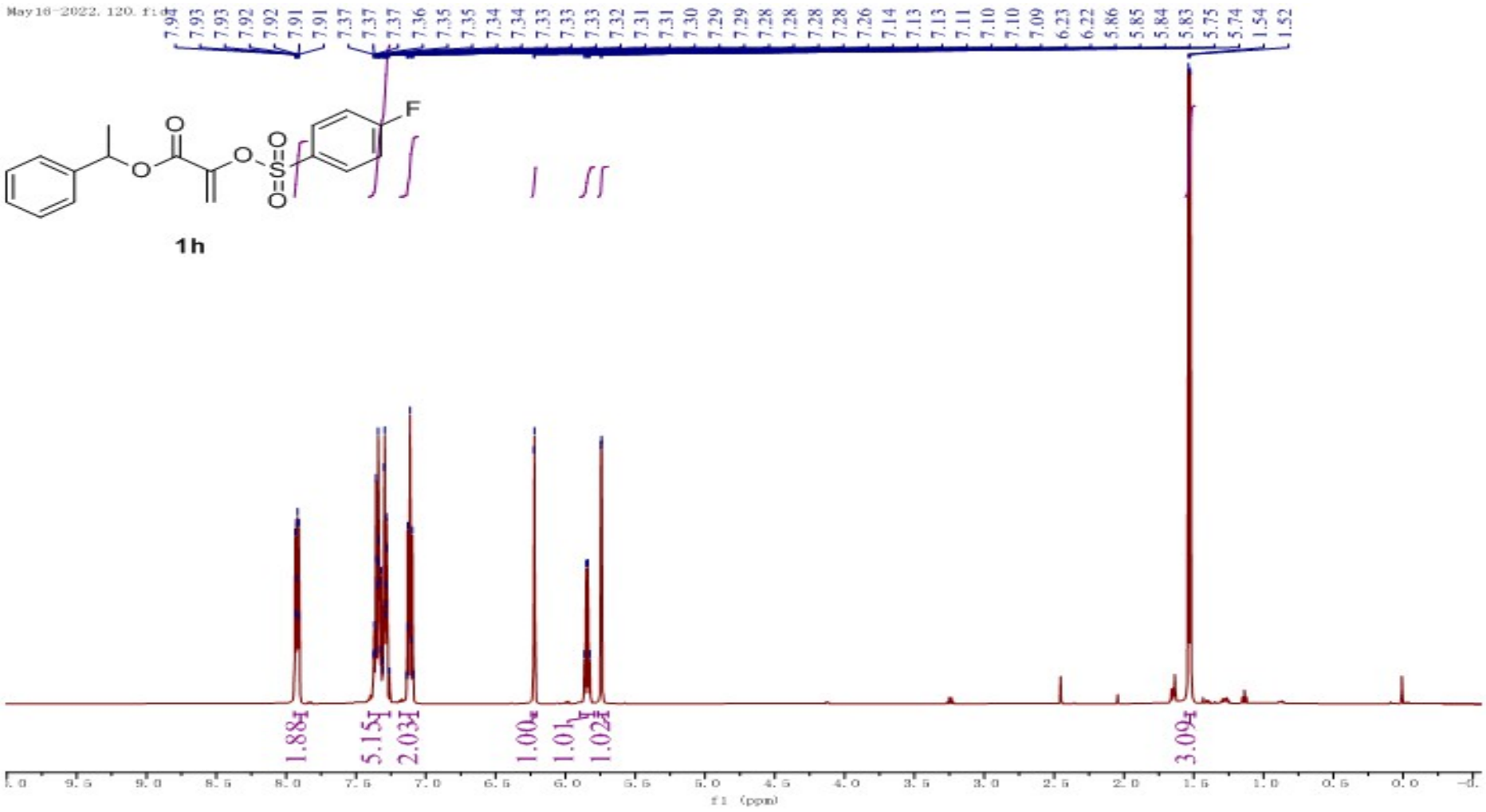
-101.64
F
O
O
O
O
O
S
1g
f1 (ppm)


May16-2022. 120. fid
7.94
7.93
7.93
7.92
7.92
7.91
7.91
7.37
7.37
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.33
7.32
7.31
7.31
7.30
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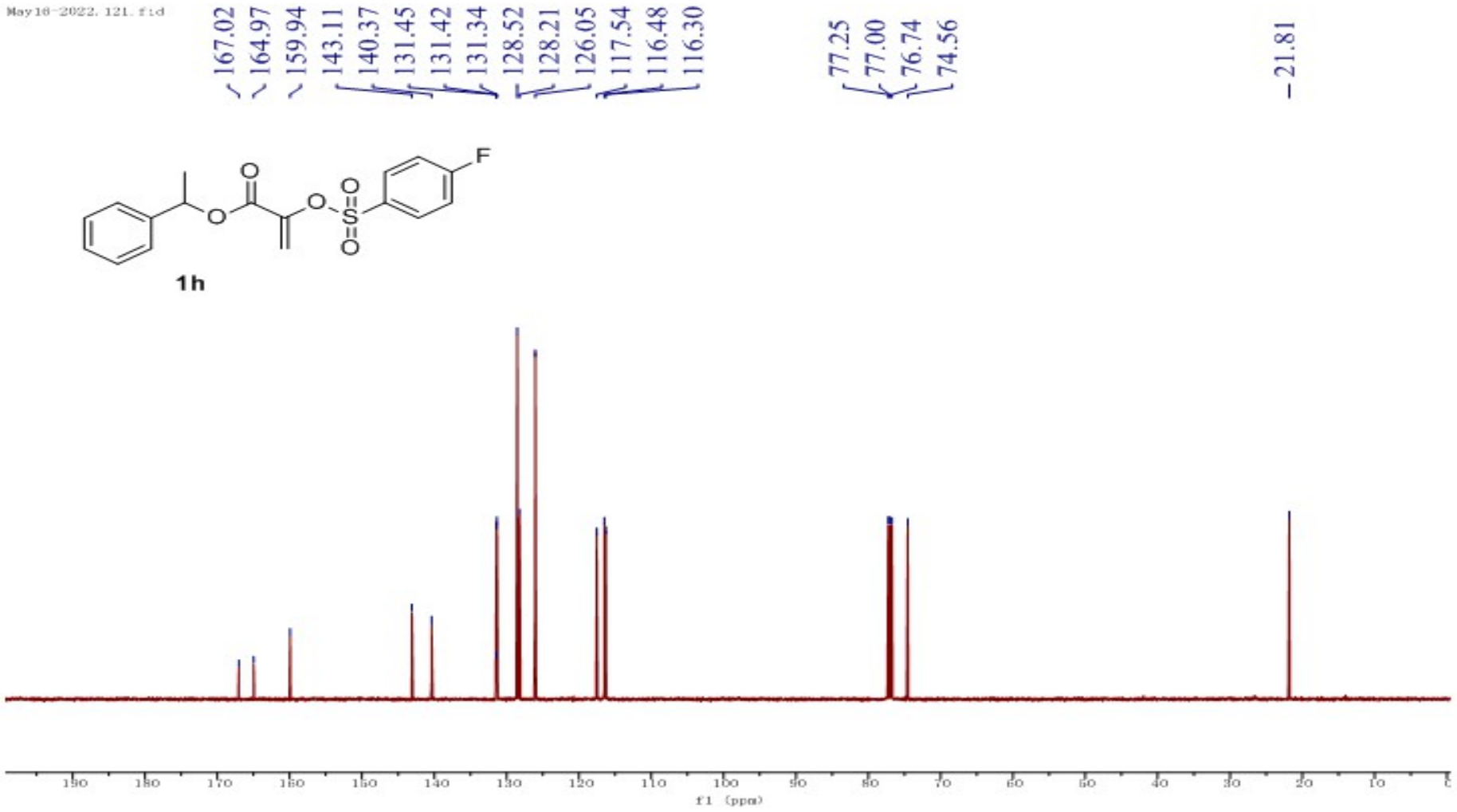
7.29
7.28
7.28
7.28
7.28
7.26
7.14
7.13
7.13
7.11
7.10
7.10
7.09
6.23
6.22
5.86
5.85
5.84
5.83
5.75
5.74
1.54
1.52
F
O
O
O
O=S=O
1h
1.88
5.15
2.03
1.00
1.01
1.02
3.09
f1 (ppm)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167.02
164.97
159.94
143.11
140.37
131.45
131.42
131.34
128.52
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126.05
117.54
116.48
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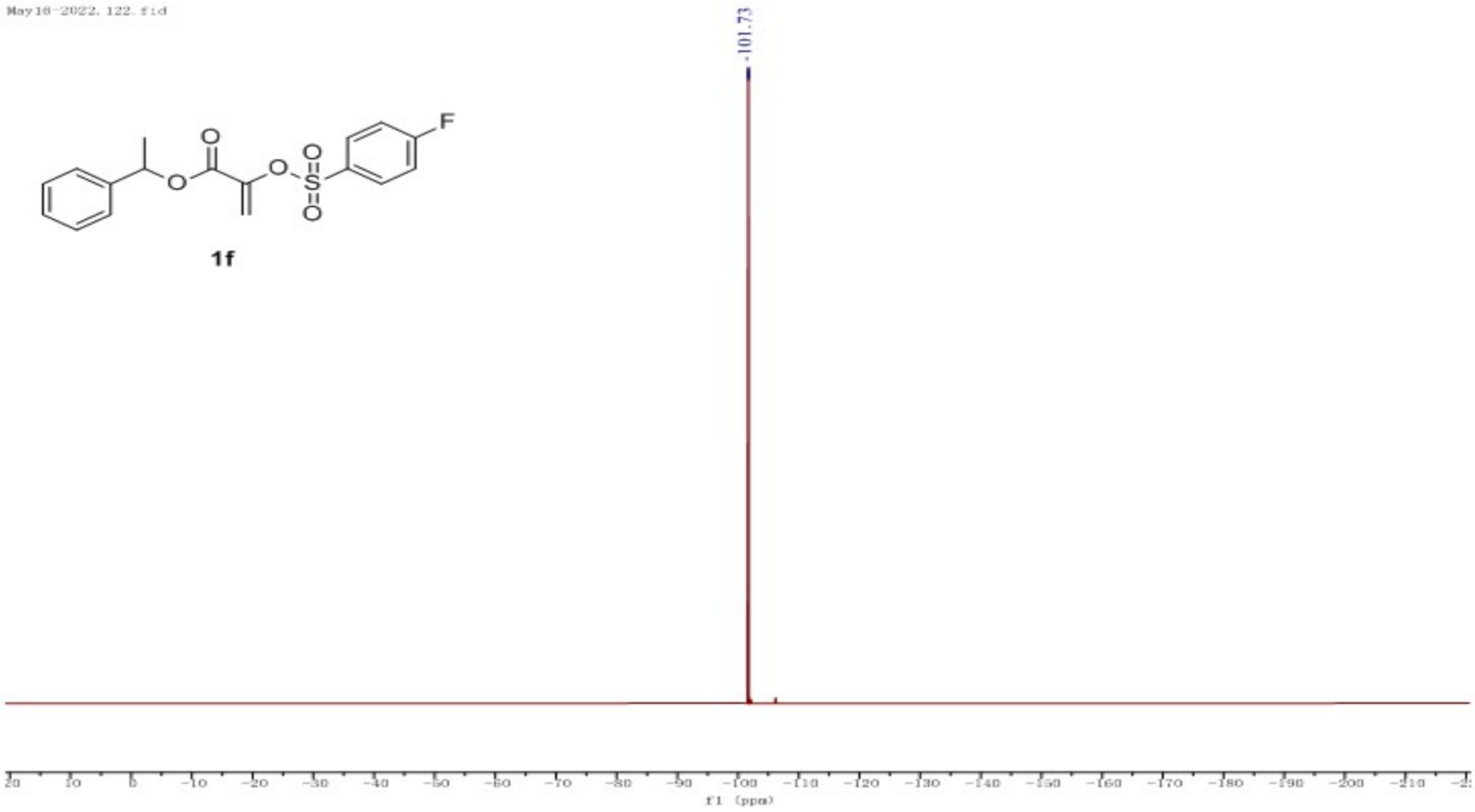
O
O
O
O
S
O
1h
f1 (ppm)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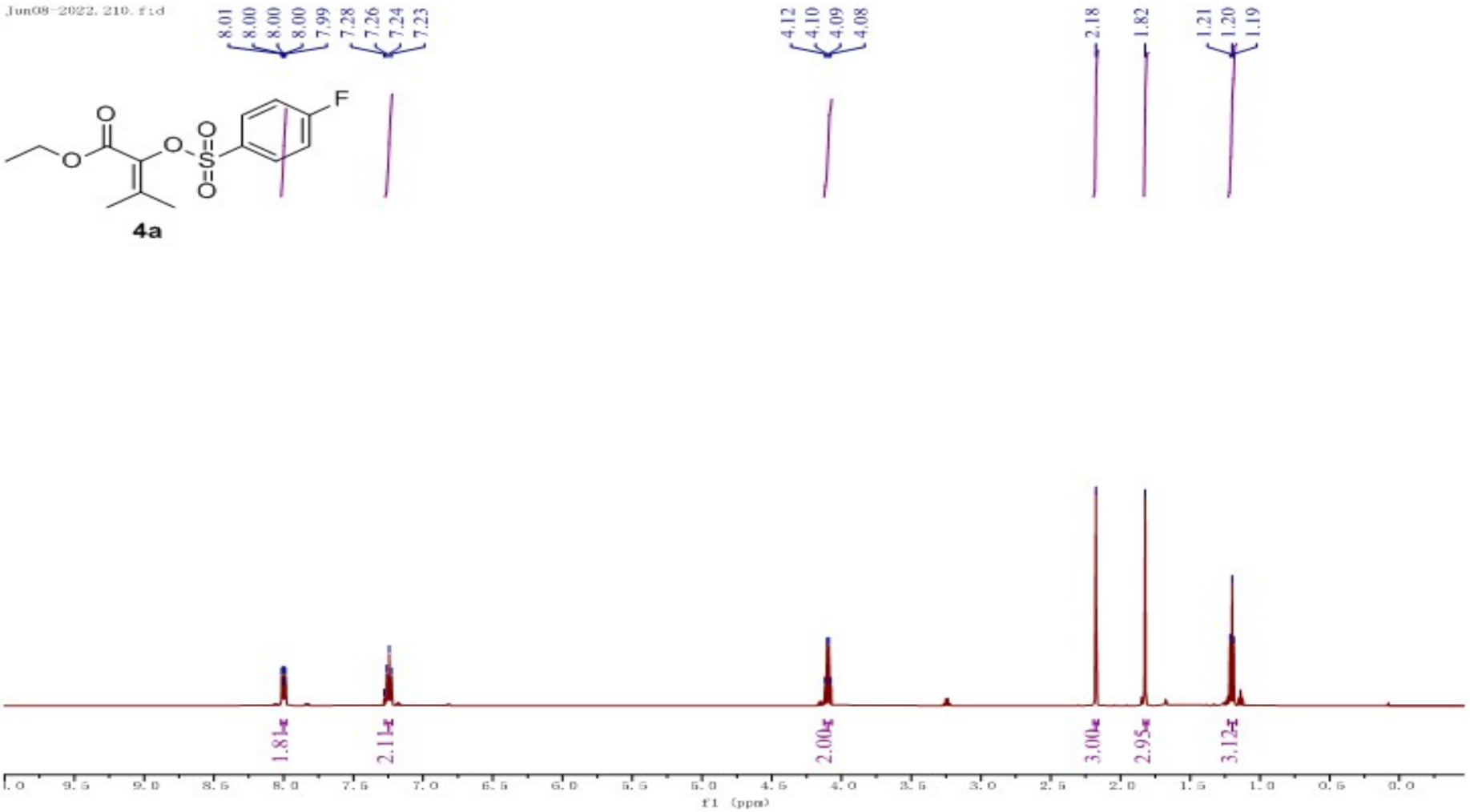
-101.73
F
O
O
O
O
S
O
1f
f1 (ppm)


Jun08-2022.210.fid
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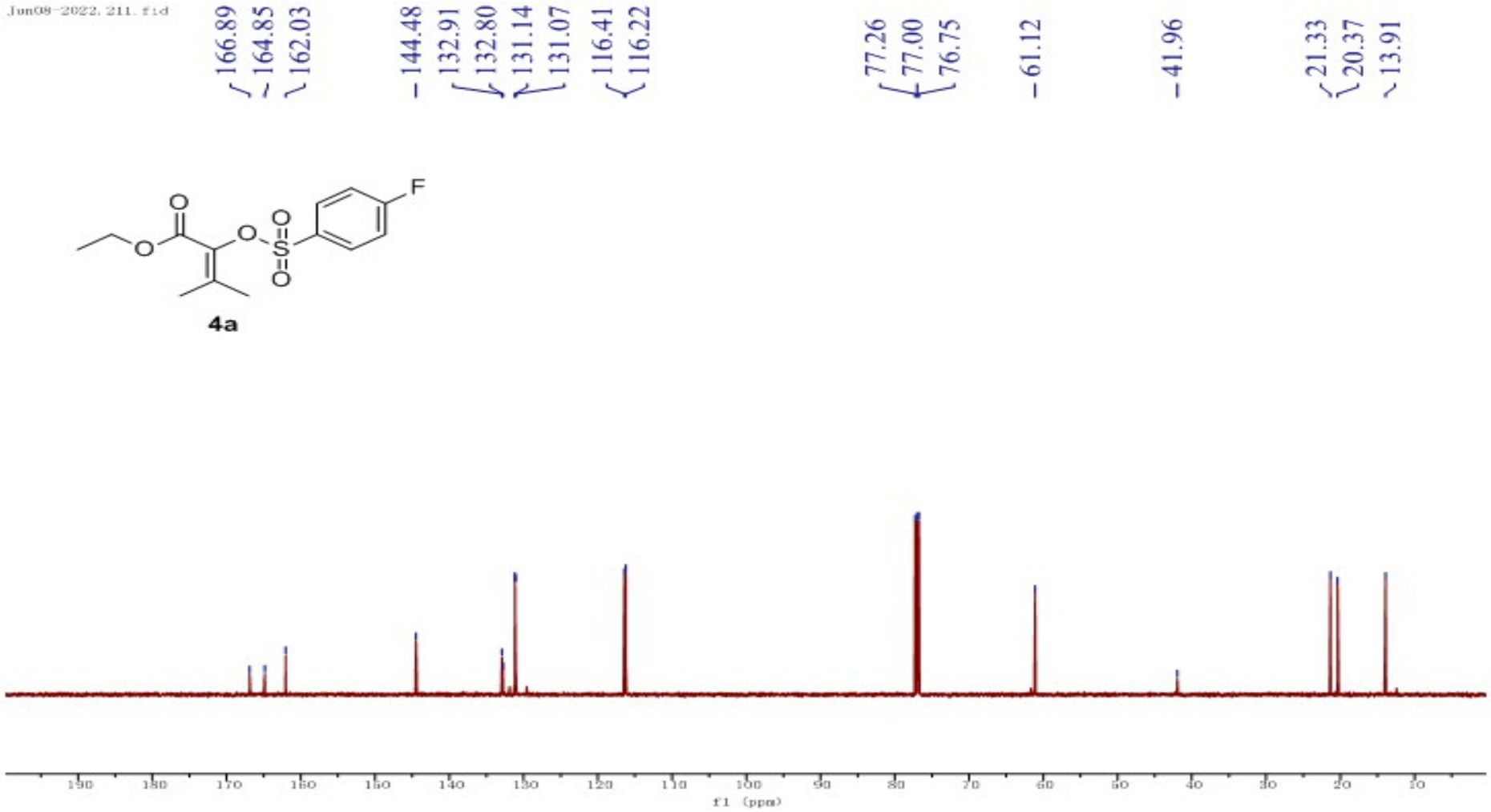
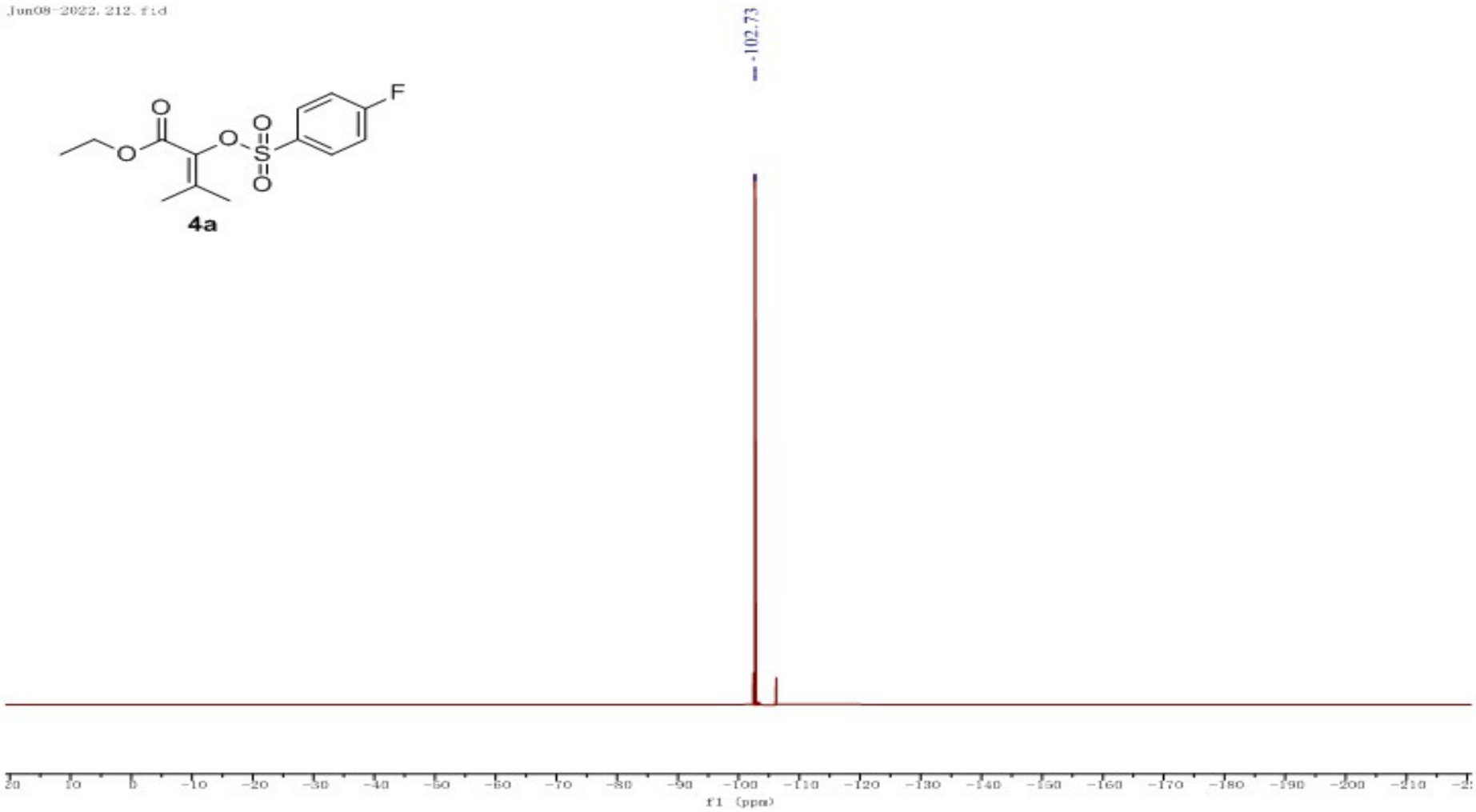
8.00
8.00
8.00
7.99
7.28
7.26
7.24
7.23
4.12
4.10
4.09
4.08
2.18
1.82
1.21
1.20
1.19
F
4a
1.81
2.11
2.00
3.00
2.95
3.12
f1 (ppm)


Jun08-2022.211.fid
166.89
164.85
162.03
144.48
132.91
132.80
131.14
131.07
116.41
116.22
77.26
77.00
76.75
61.12
41.96
21.33
20.37
13.91
F
O
O
O
S
O
O
4a
f1 (ppm)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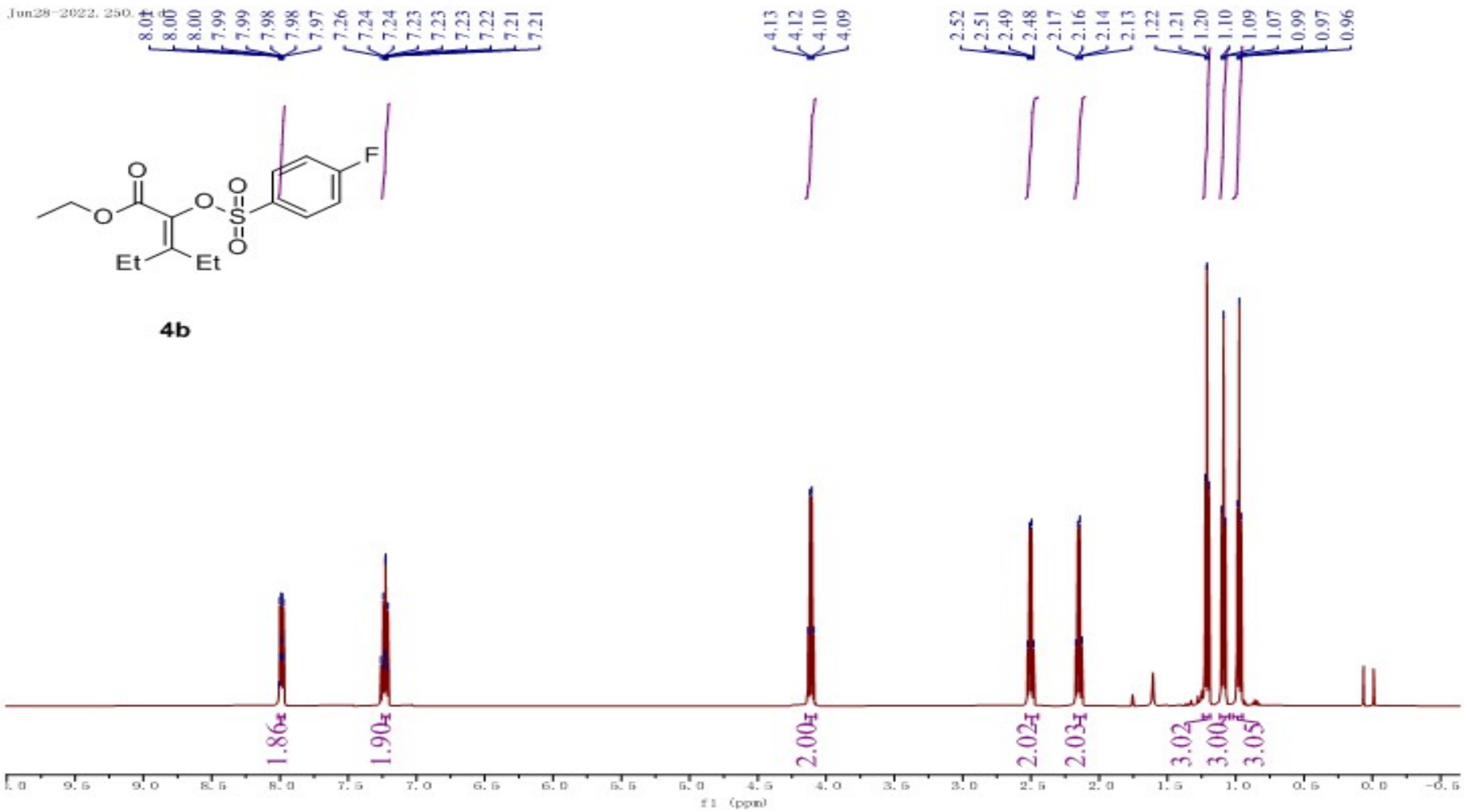
-102.73
F
O
O
O
O
O
O
4a
f1 (ppm)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8.00
8.00
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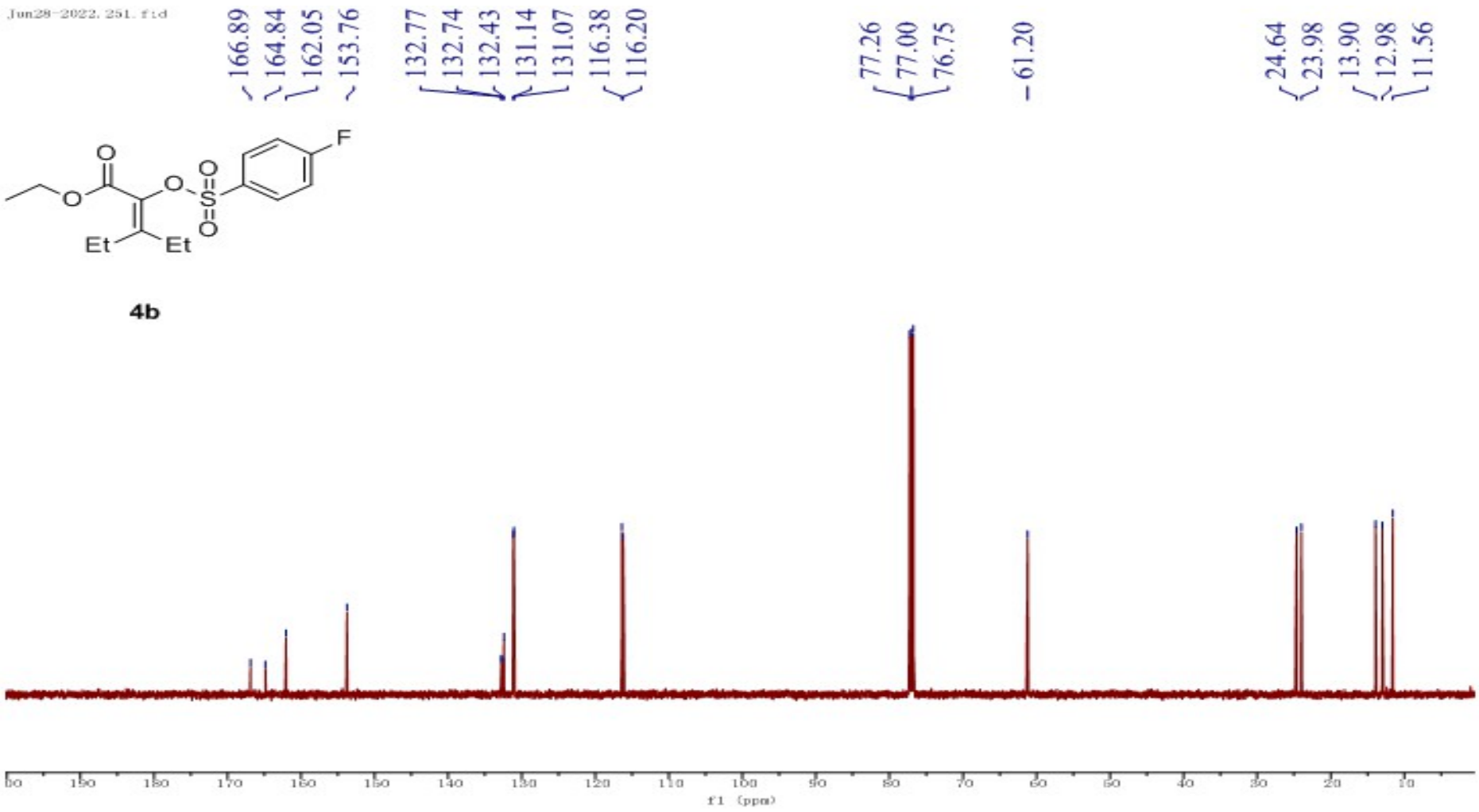
7.99
7.98
7.98
7.97
7.26
7.24
7.24
7.23
7.23
7.23
7.22
7.21
7.21
4.13
4.12
4.10
4.09
2.52
2.51
2.49
2.48
2.17
2.16
2.14
2.13
1.22
1.21
1.20
1.10
1.09
1.07
0.99
0.97
0.96
F
O
O
O
O
S
O
Et
Et
4b
1.86
1.90
2.00
2.02
2.03
3.02
3.00
3.05
f1 (ppm)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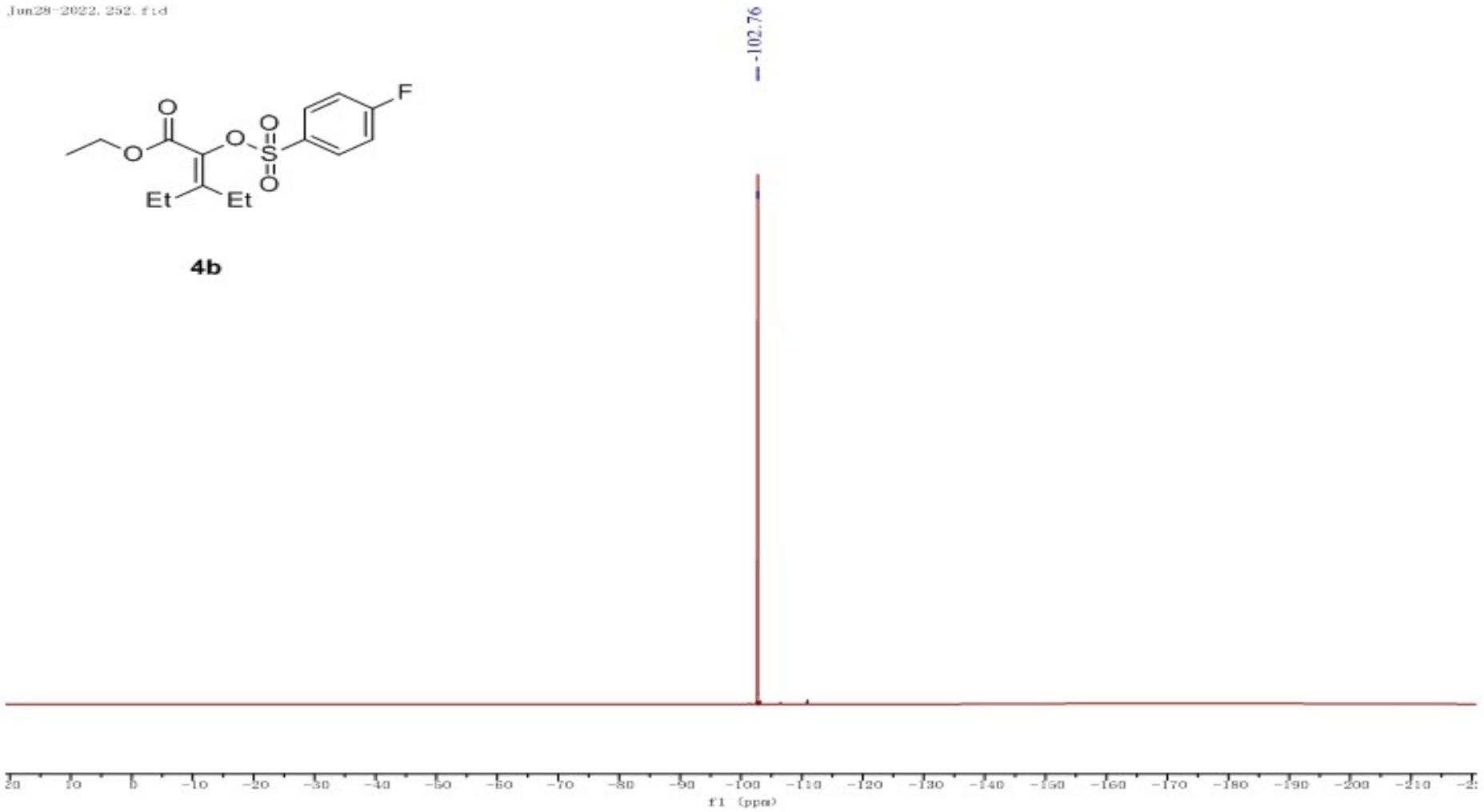
131.14
131.07
116.38
116.20
77.26
77.00
76.75
61.20
24.64
23.98
13.90
12.98
11.56
F
O
O
O
O
S
O
Et
Et
4b
f1 (ppm)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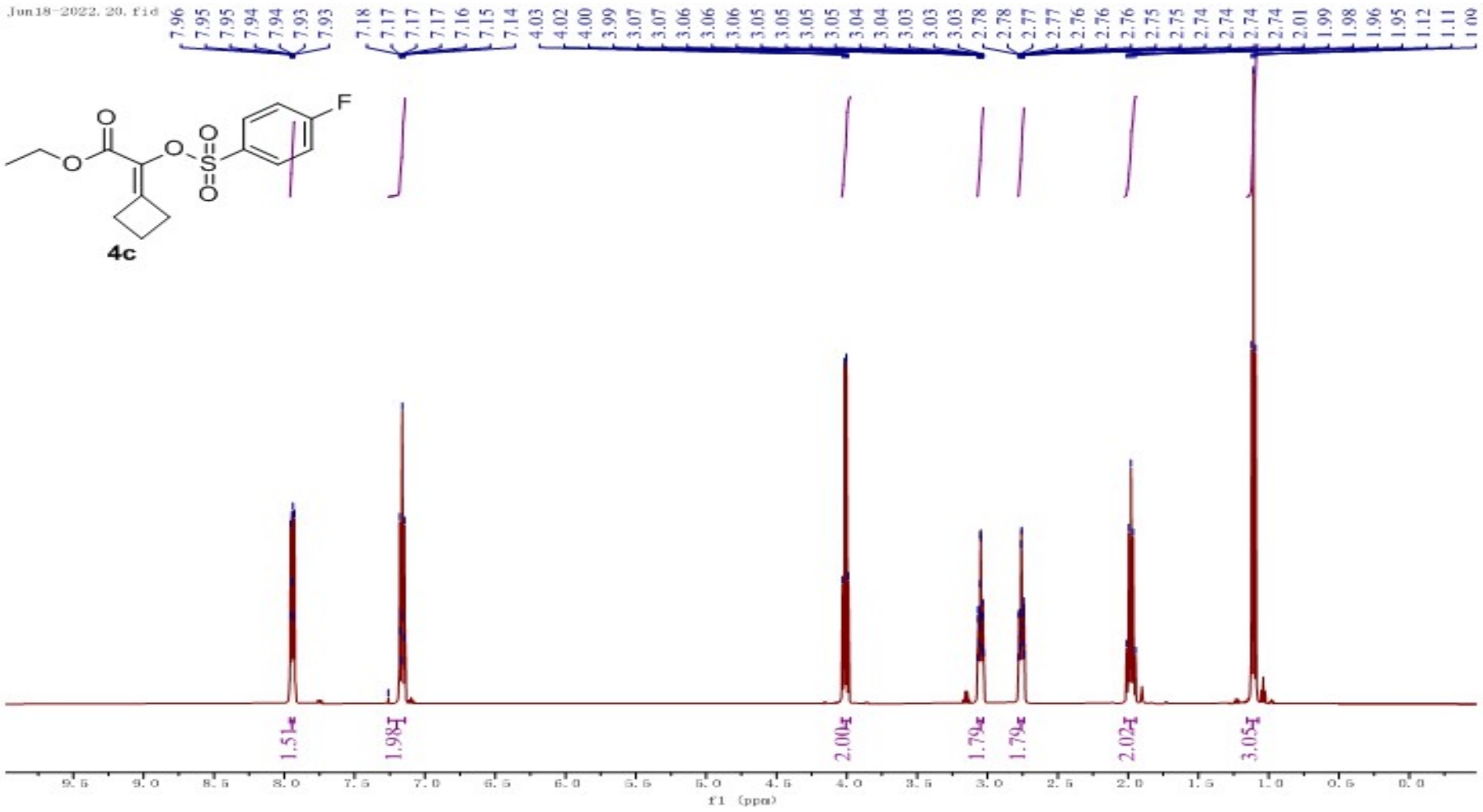
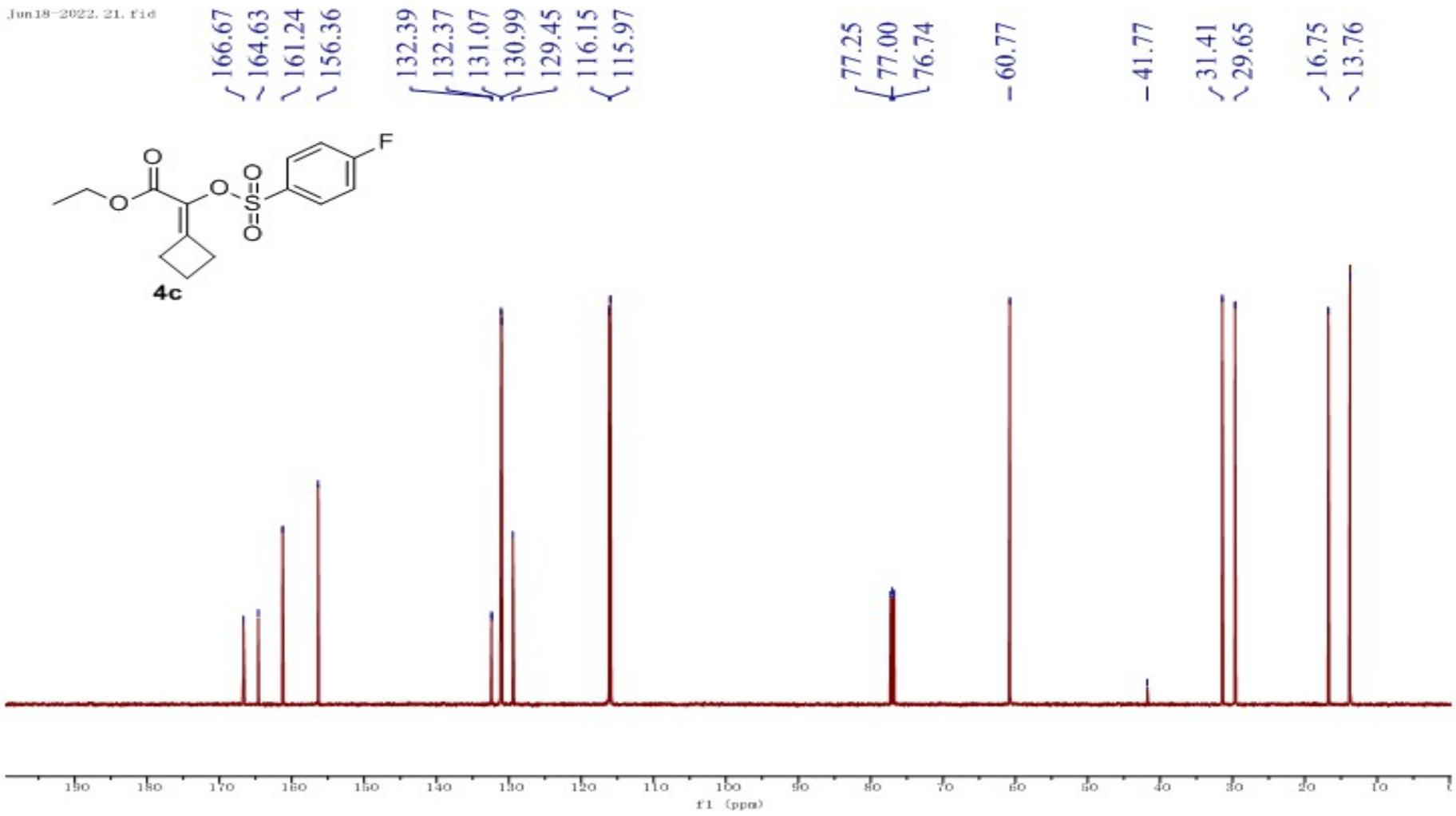
-102.76
F
O
O
O
O
S
O
Et
Et
4b
f1 (ppm)


Jun18-2022.20.fid
4c
f1 (ppm)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132.37
131.07
130.99
129.45
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115.97
77.25
77.00
76.74
60.77
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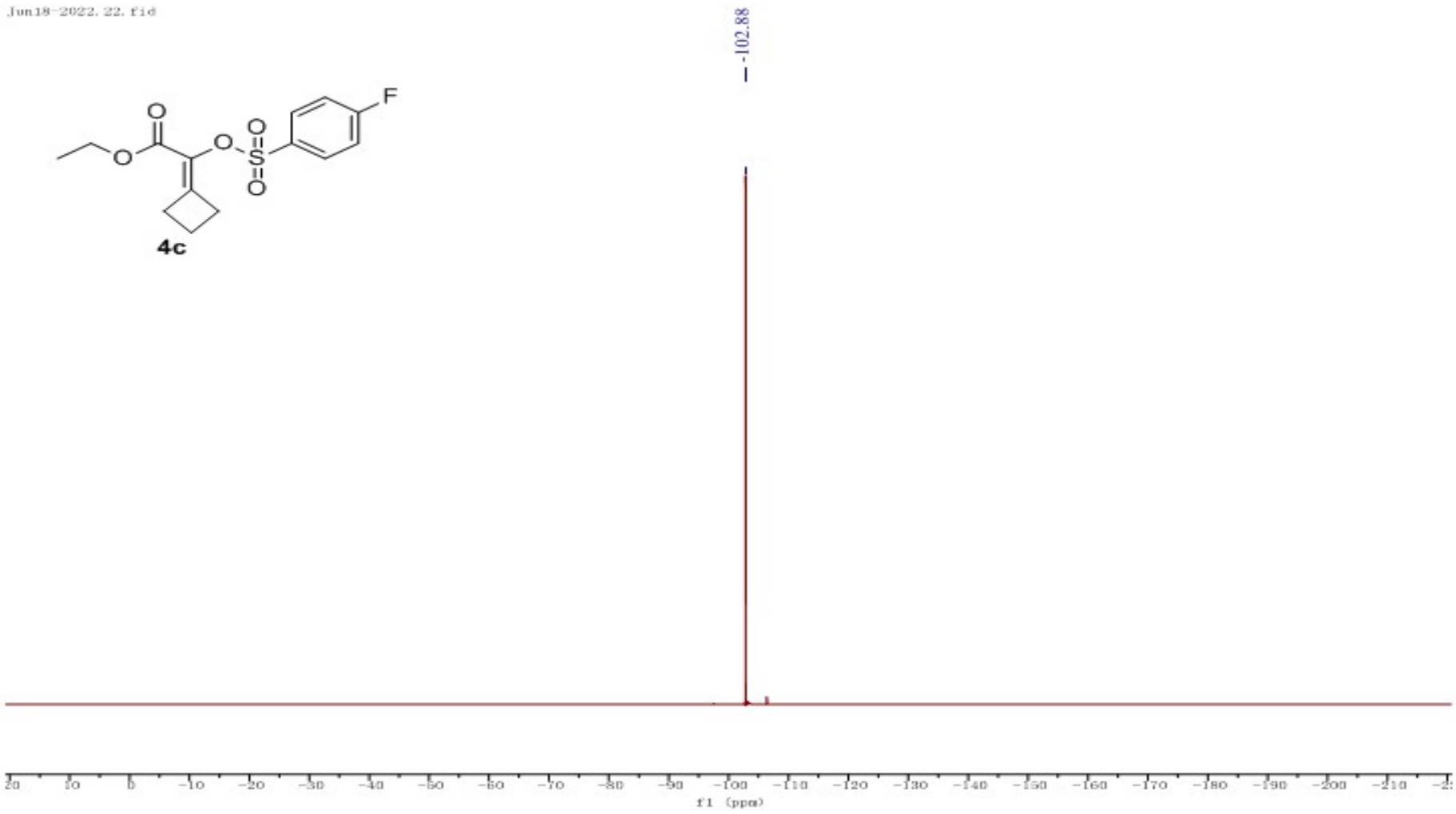
O
O
O
S
O
O
4c
f1 (ppm)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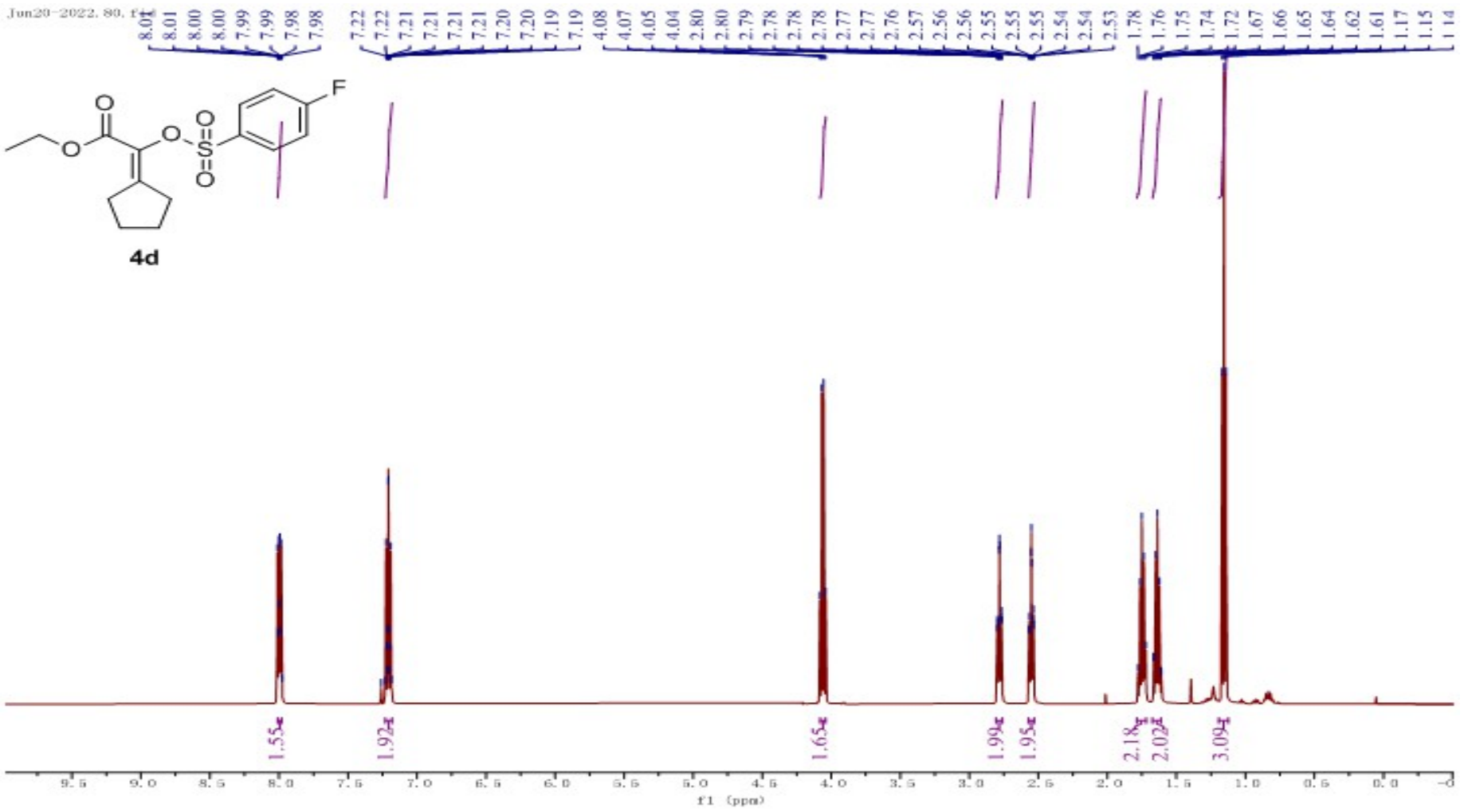
F
O
O
O
O
O
S
O
4c
f1 (ppm)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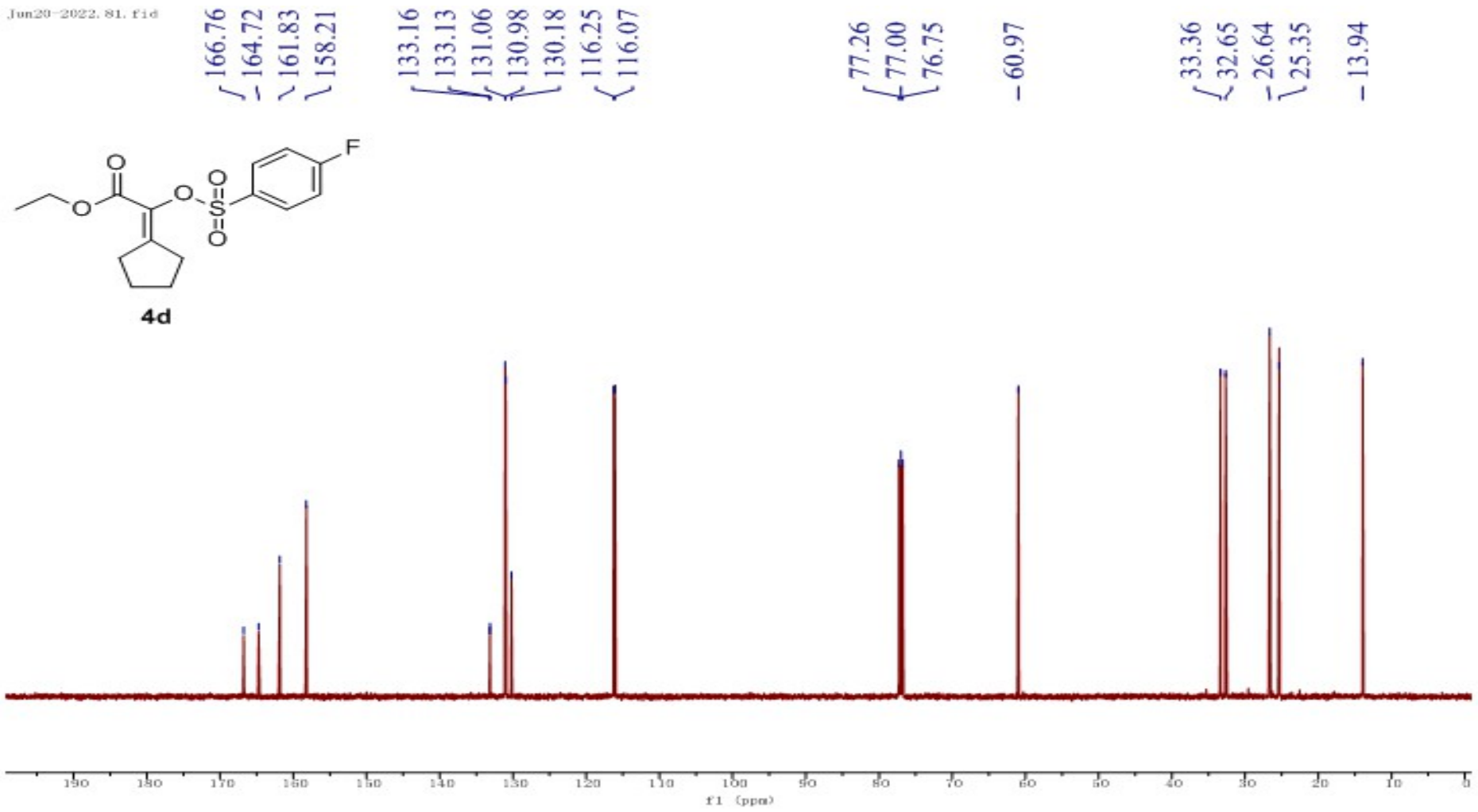
7.22
7.22
7.21
7.21
7.21
7.21
7.20
7.20
7.19
7.19
4.08
4.07
4.05
4.04
2.80
2.80
2.79
2.78
2.78
2.78
2.77
2.77
2.76
2.57
2.56
2.56
2.55
2.53
2.55
2.54
2.54
2.53
1.78
1.76
1.75
1.74
1.72
1.67
1.66
1.65
1.64
1.62
1.61
1.17
1.15
1.14
F
O
O
O
O
S
O
O
4d
1.55
1.92
1.65
1.99
1.95
2.18
2.02
3.09
f1 (ppm)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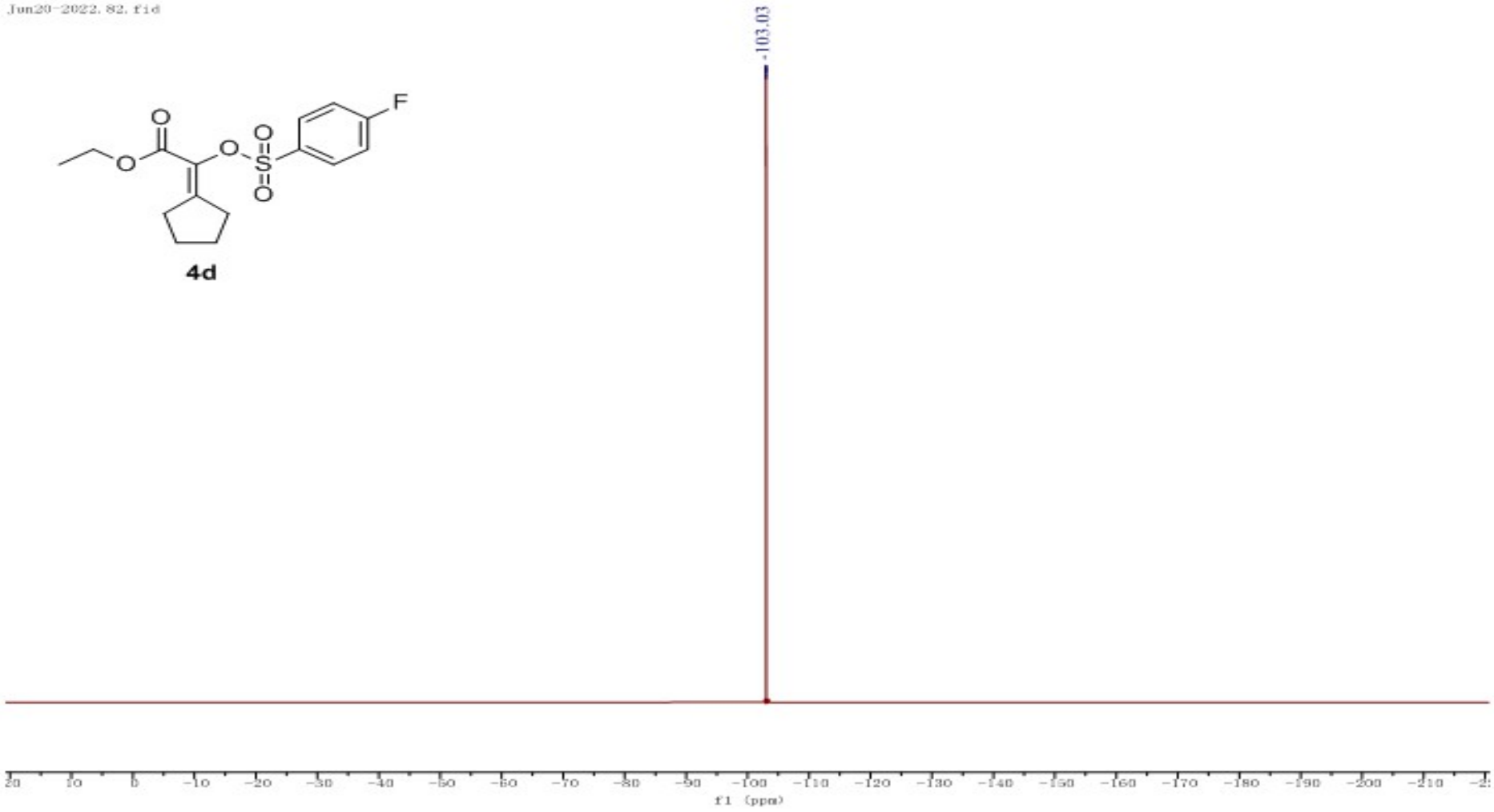
133.13
131.06
130.98
130.18
116.25
116.07
77.26
77.00
76.75
60.97
33.36
32.65
26.64
25.35
13.94
F
O
O
O
O
O=S=O
4d
f1 (ppm)


Jun20-2022.82.fid
-103.03
F
O
O
O
O
O
S
O
4d
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
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-130
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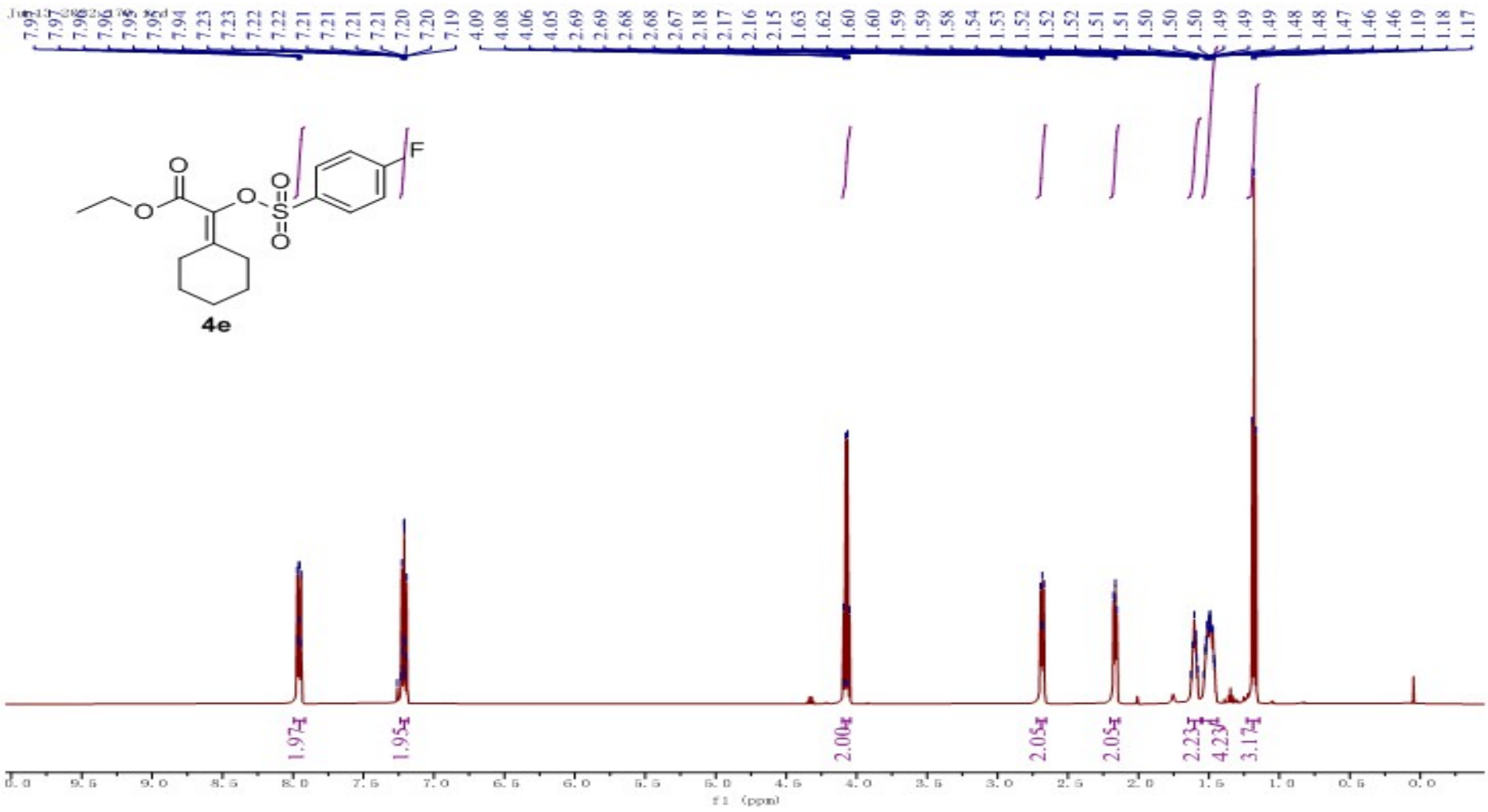
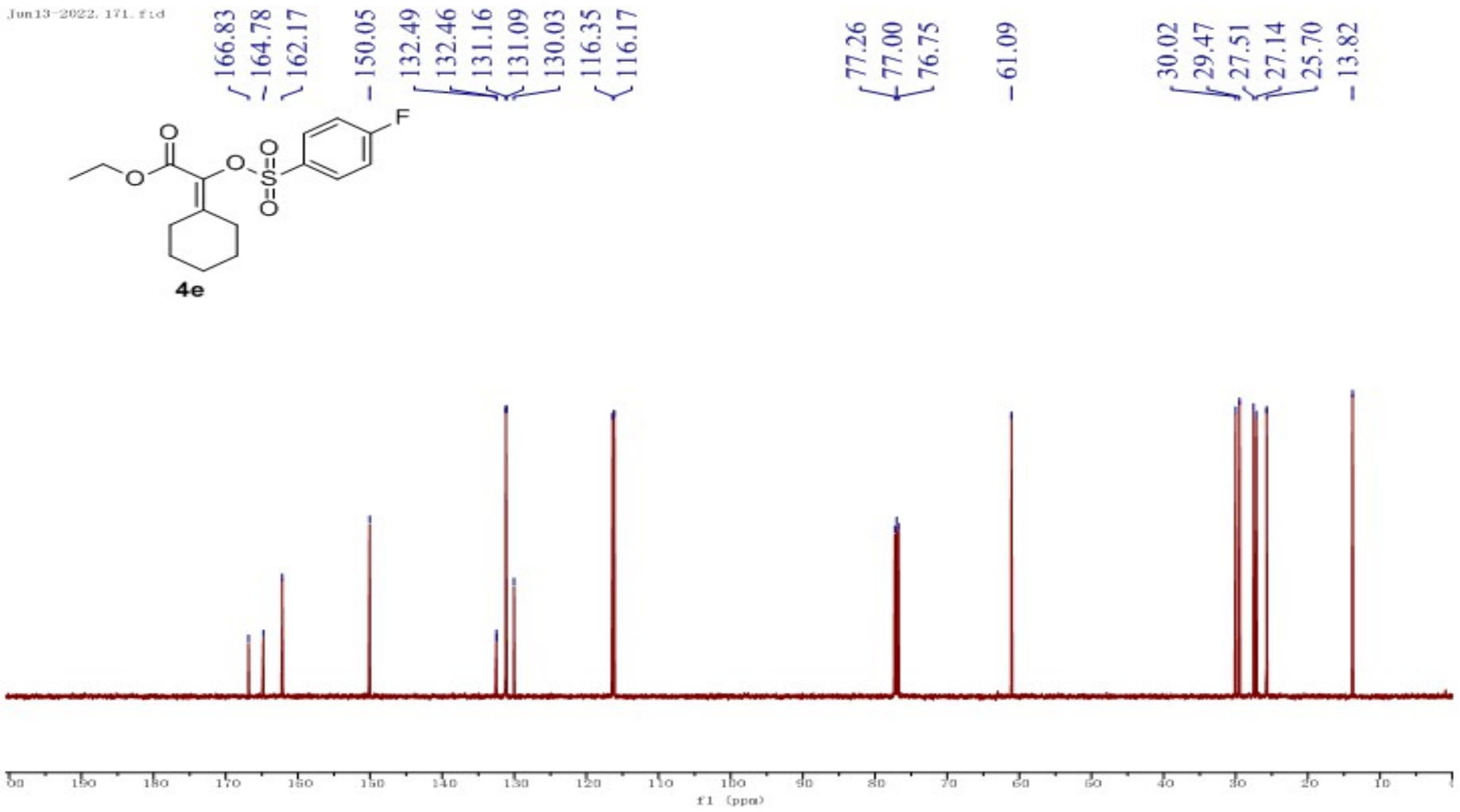
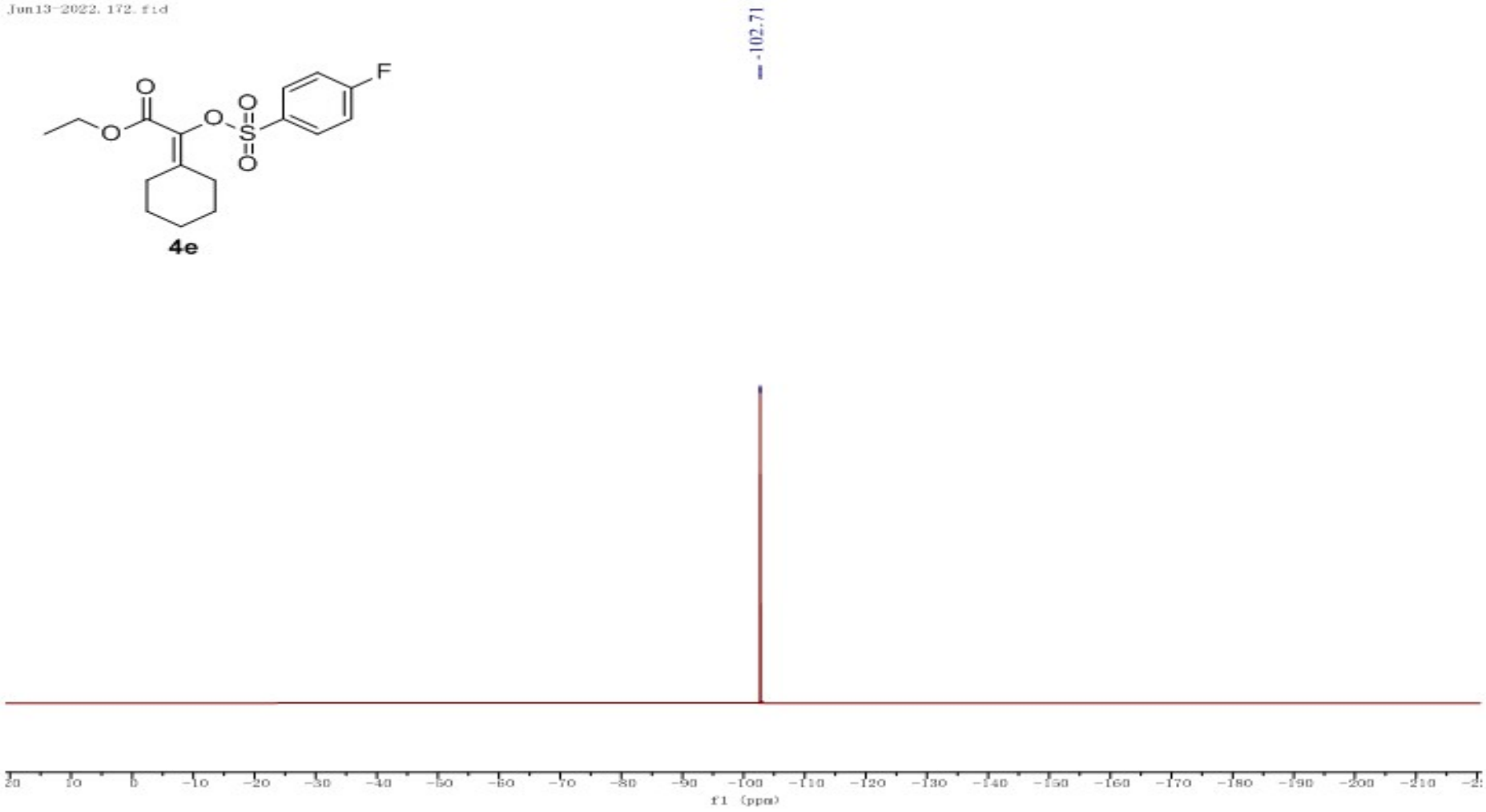
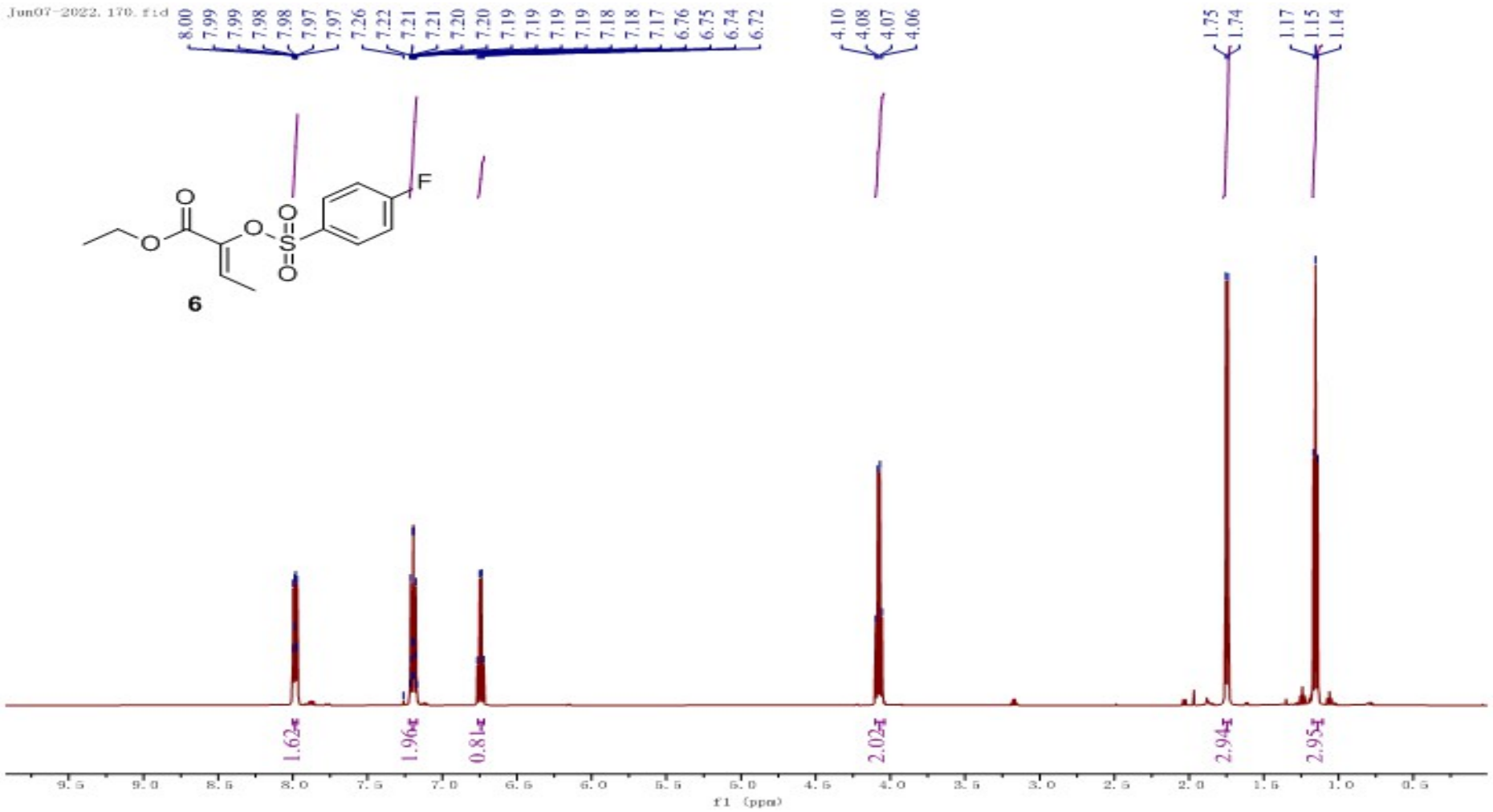
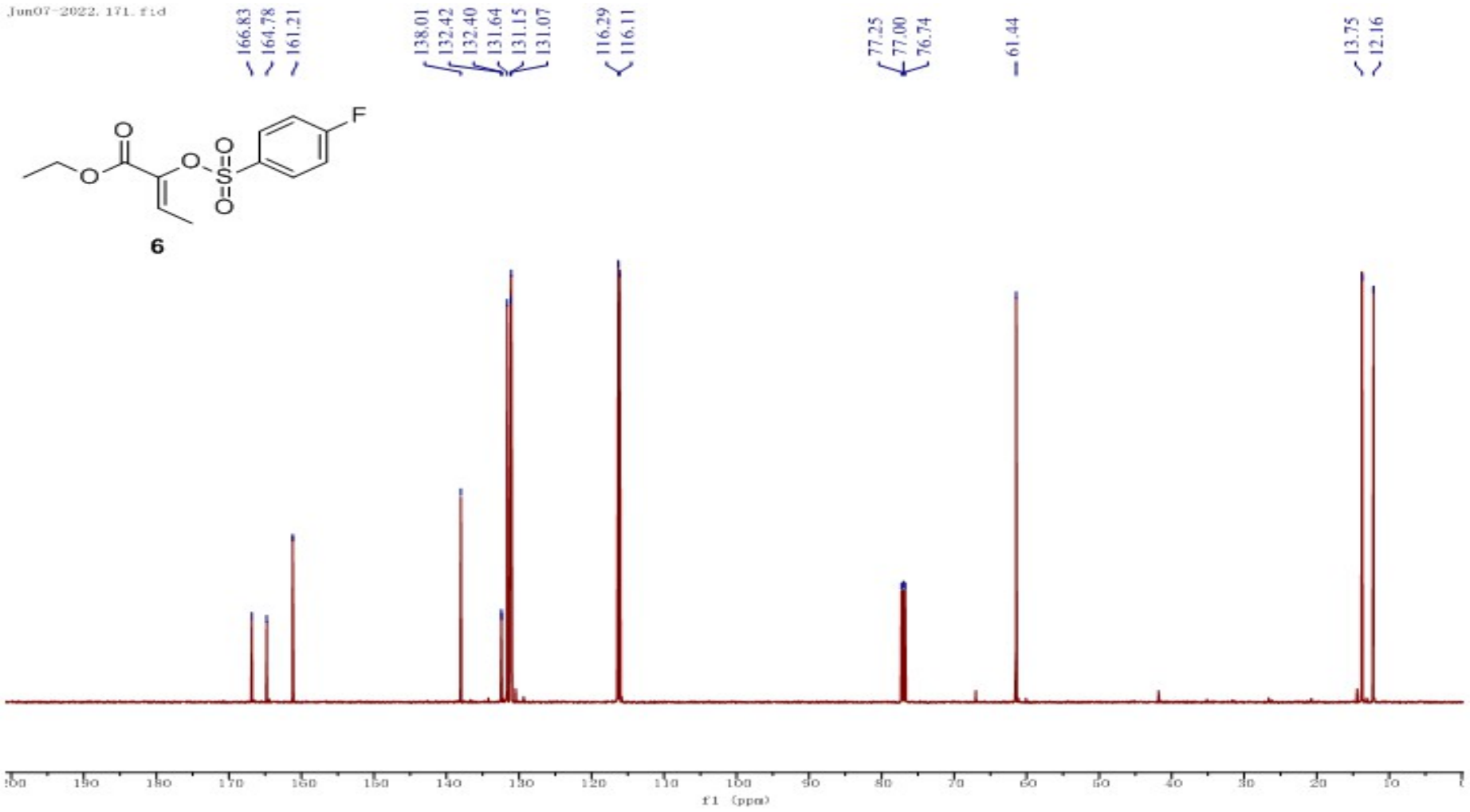
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

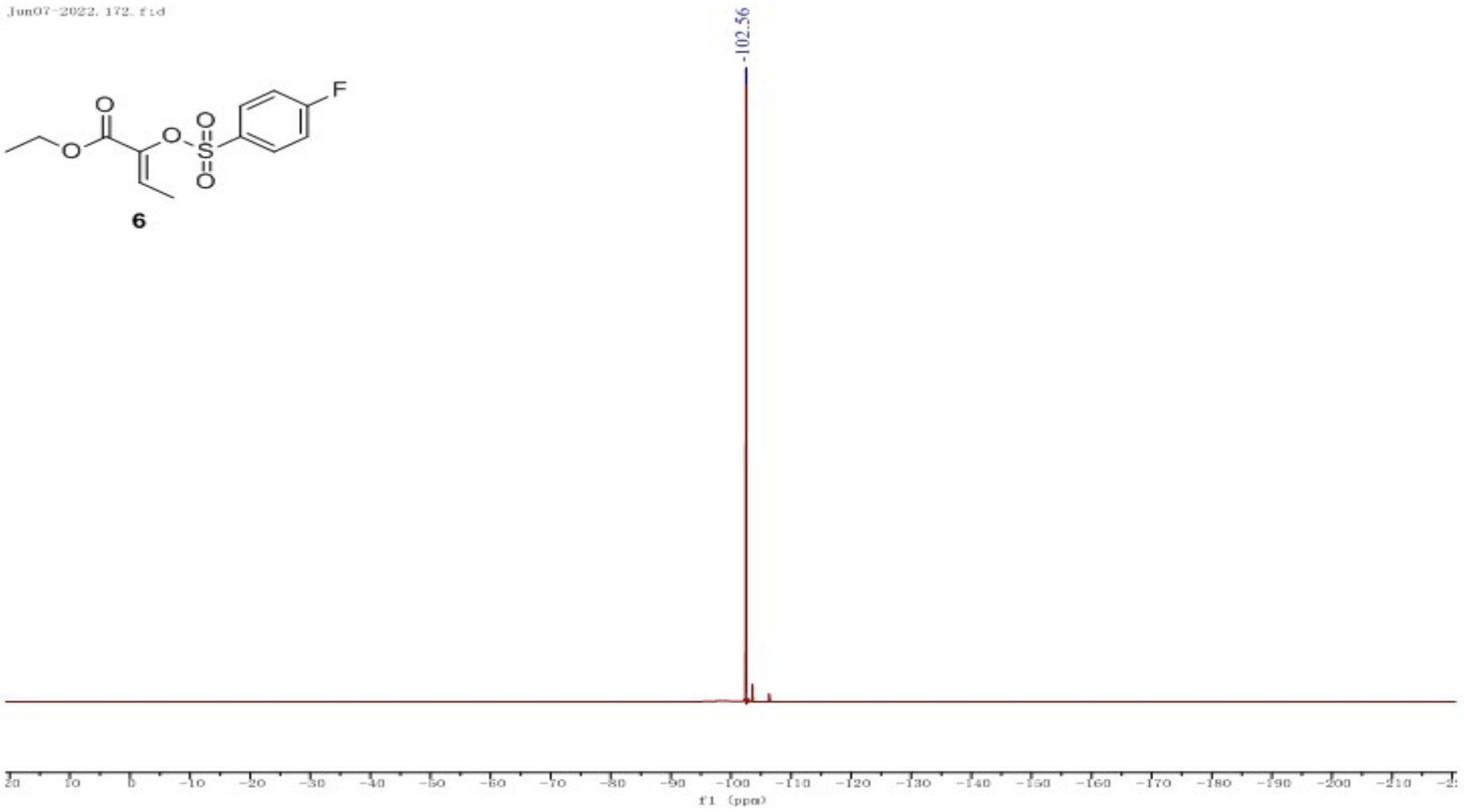

4e
1.97
1.95
2.00
2.05
2.05
2.23
4.23
3.17
f1 (ppm)

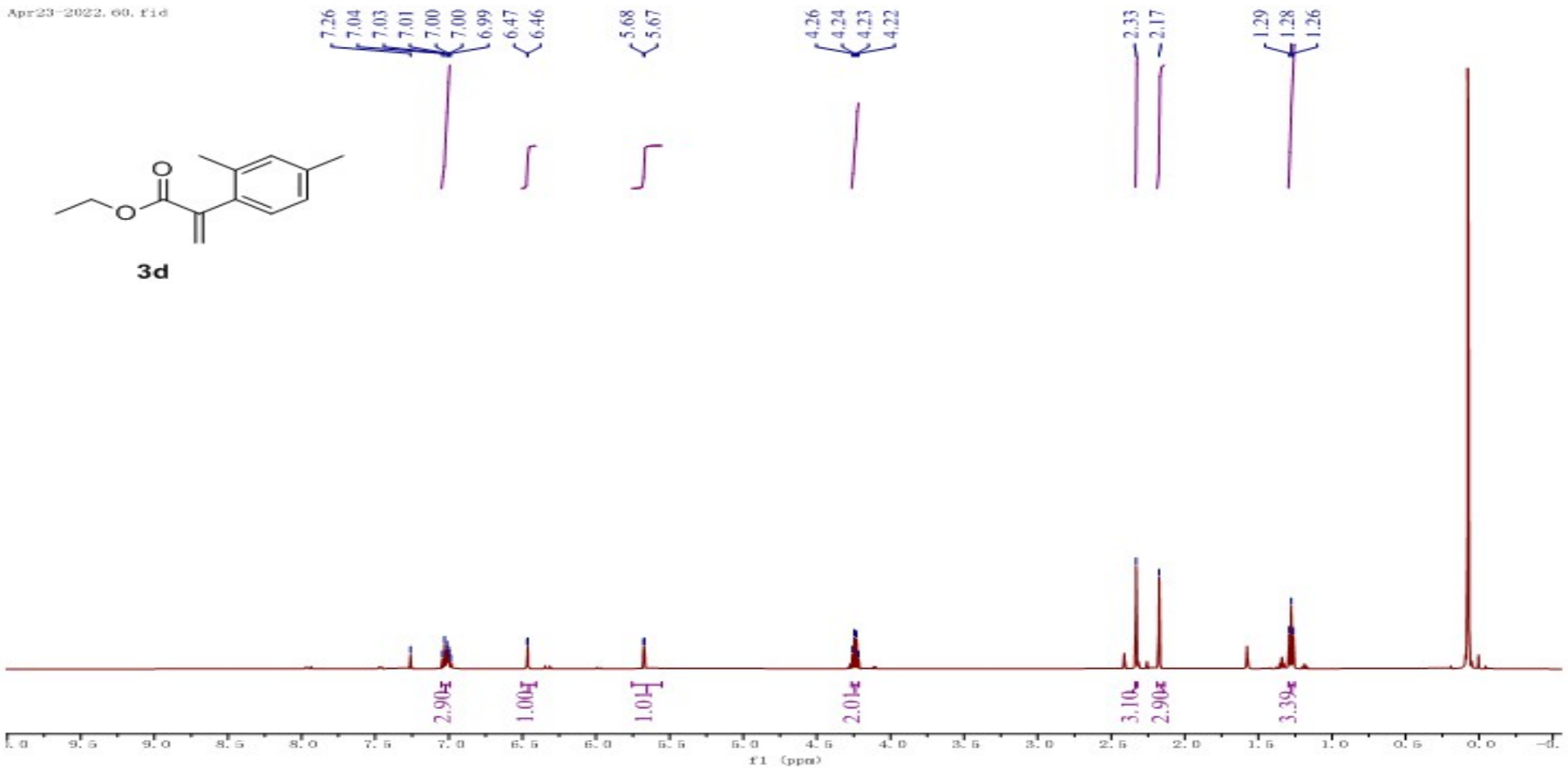

Jun13-2022.171.fid
166.83
164.78
162.17
150.05
132.49
132.46
131.16
131.09
130.03
116.35
116.17
77.26
77.00
76.75
61.09
30.02
29.47
27.51
27.14
25.70
13.82
F
O
O
O
O
S
O
4e
f1 (ppm)


Jun13-2022.172.fid
-102.71
F
O
O
O
O
S
O
4e
f1 (ppm)

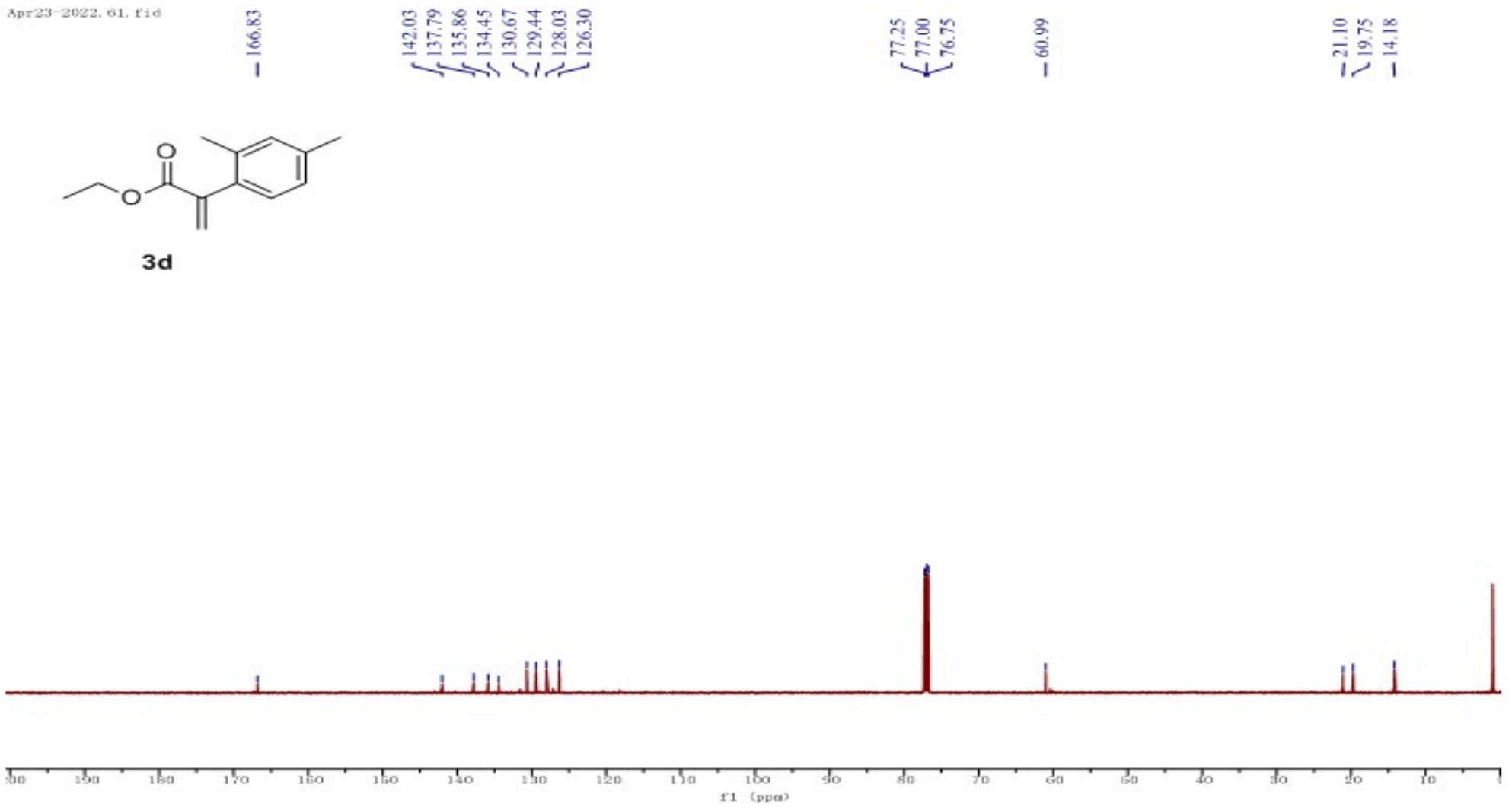

Jun07-2022.170.fid
8.00
7.99
7.99
7.98
7.98
7.97
7.97
7.26
7.22
7.21
7.21
7.20
7.20
7.19
7.19
7.19
7.19
7.18
7.18
7.17
6.76
6.75
6.74
6.72
4.10
4.08
4.07
4.06
1.75
1.74
1.17
1.15
1.14
F
O
O
O
O
S
O
O
6
1.62
1.96
0.81
2.02
2.94
2.95
f1 (ppm)


Jun07-2022. 171. fid
166.83
164.78
161.21
138.01
132.42
132.40
131.64
131.15
131.07
116.29
116.11
77.25
77.00
76.74
61.44
13.75
12.16
F
O
O
O
O
S
O
O
6
f1 (ppm)

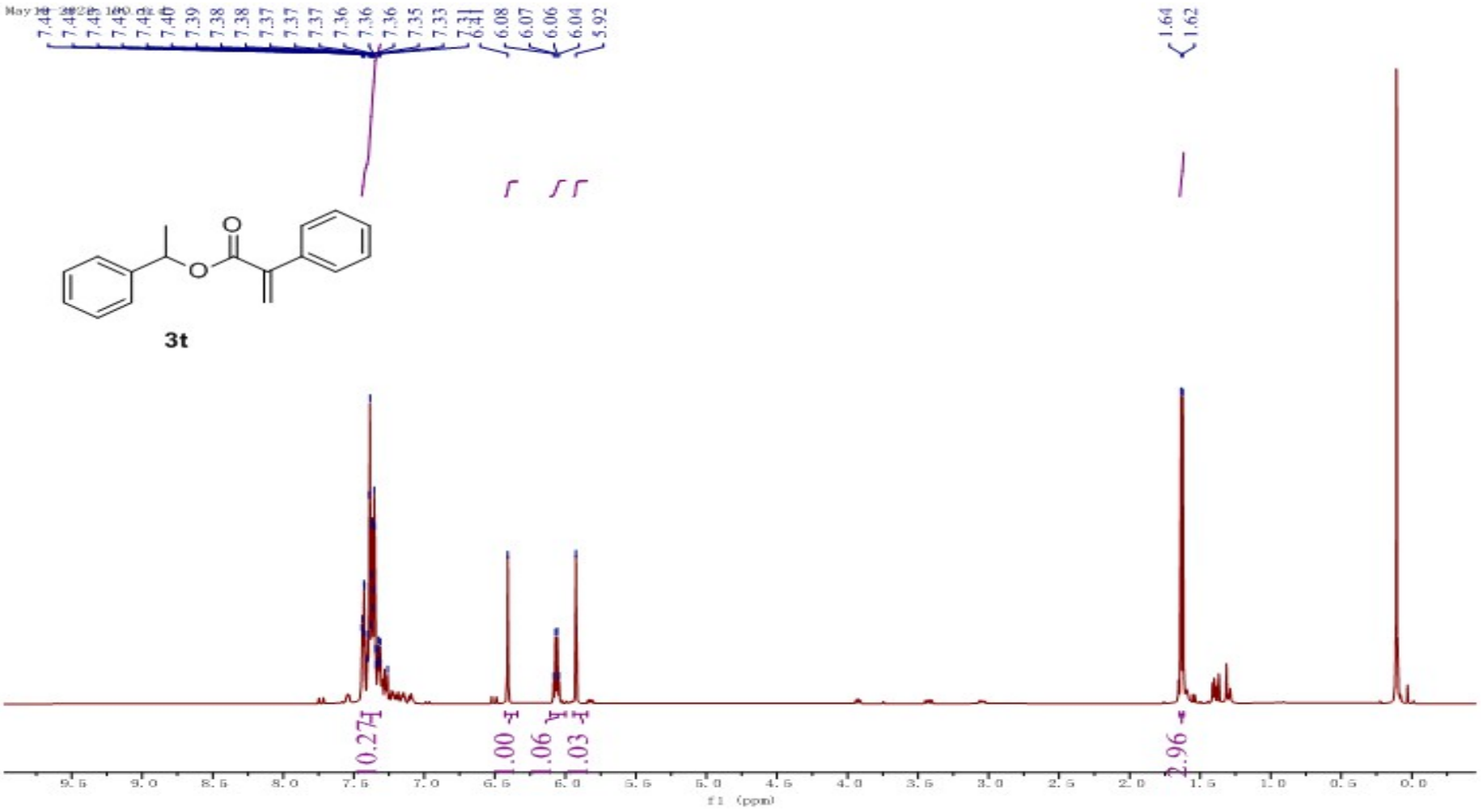

Jun07-2022.172.fid
-102.56
F
O
O
O
O
S
O
6
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)

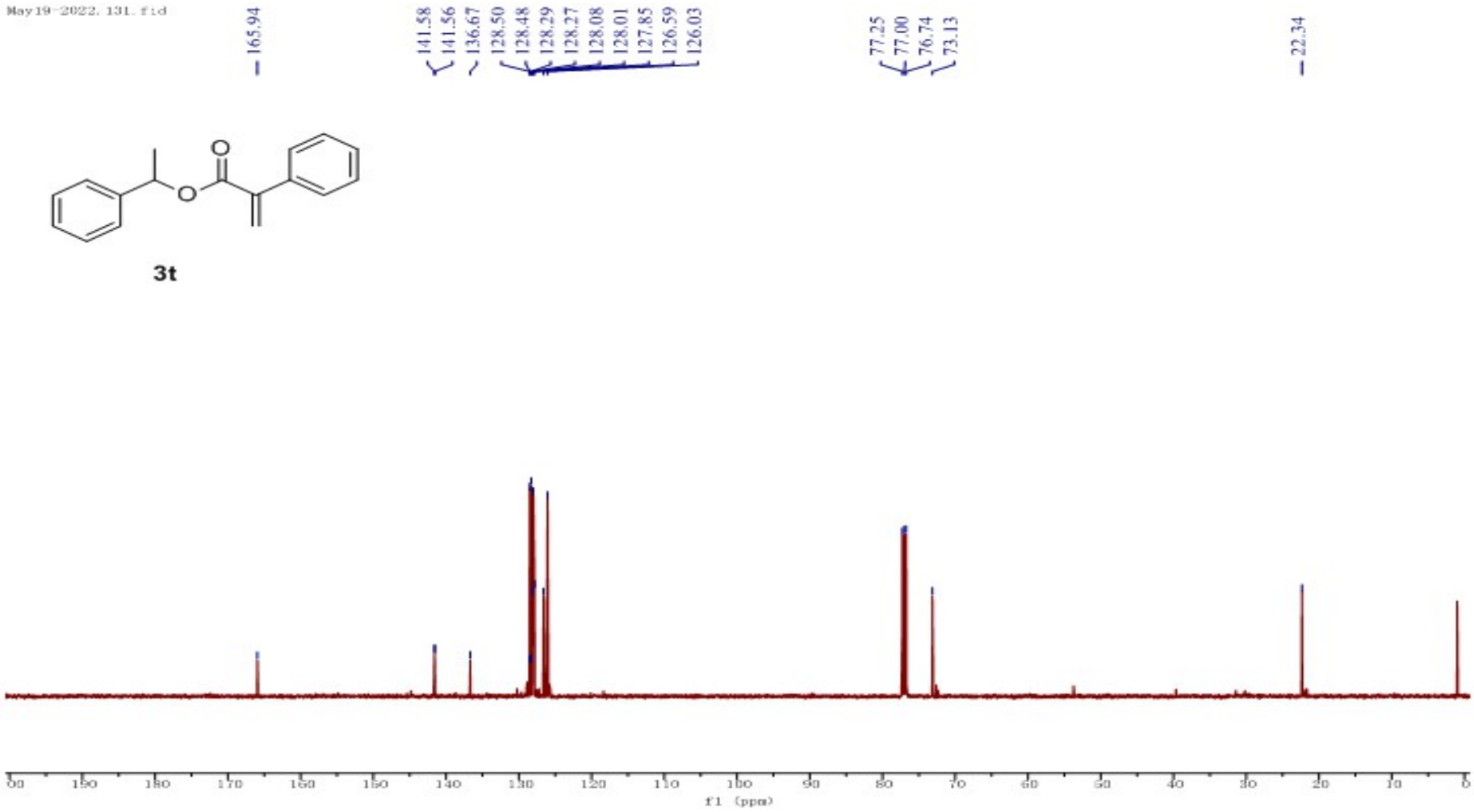

Apr23-2022.60.fid
7.26
7.04
7.03
7.01
7.00
7.00
6.99
6.47
6.46
5.68
5.67
4.26
4.24
4.23
4.22
2.33
2.17
1.29
1.28
1.26
2.90
1.00
1.01
2.01
3.10
2.90
3.39
f1 (ppm)

3d

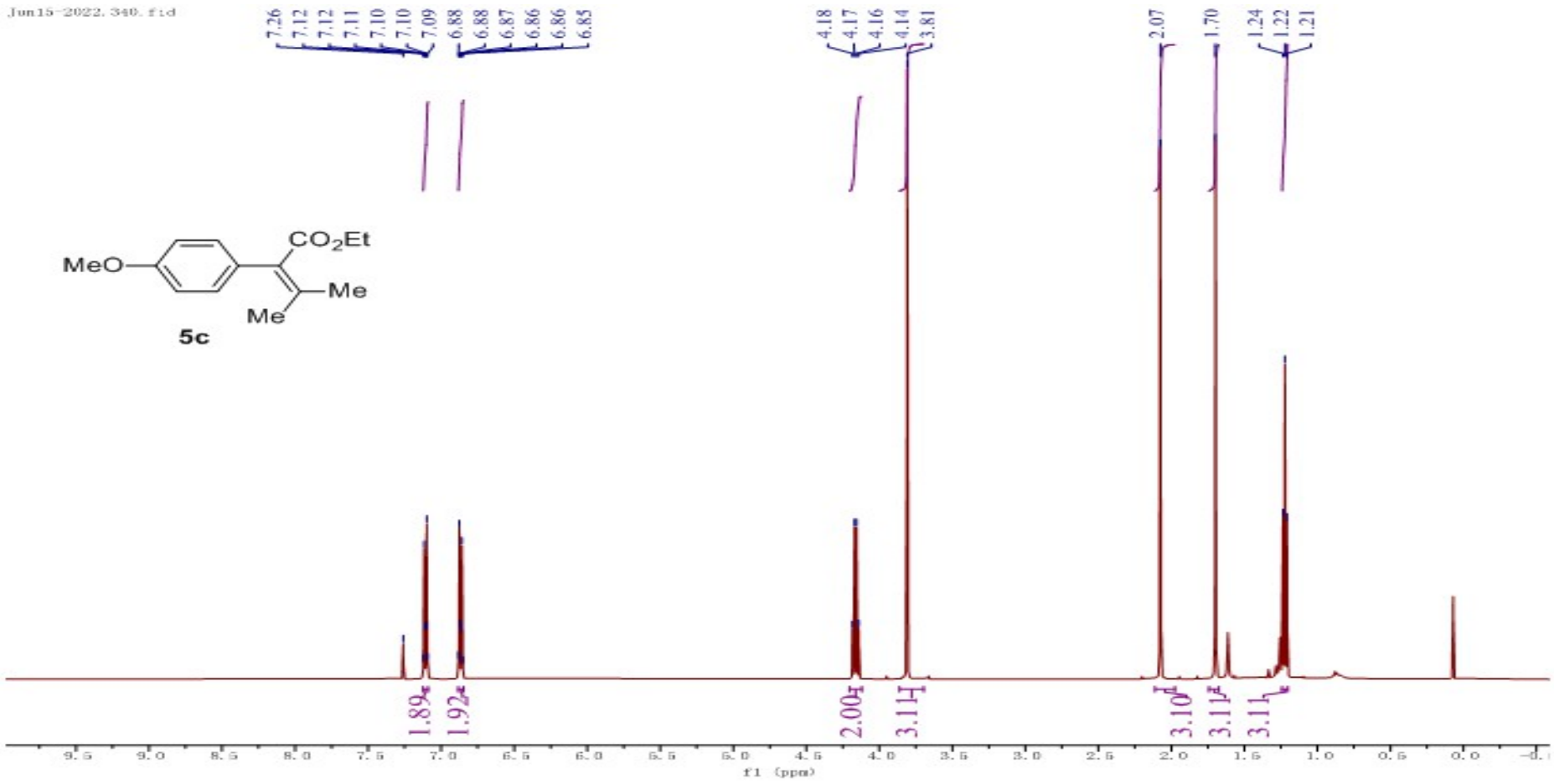

Apr23-2022.61.fid
166.83
142.03
137.79
135.86
134.45
130.67
129.44
128.03
126.30
77.25
77.00
76.75
60.99
21.10
19.75
14.18
f1 (ppm)

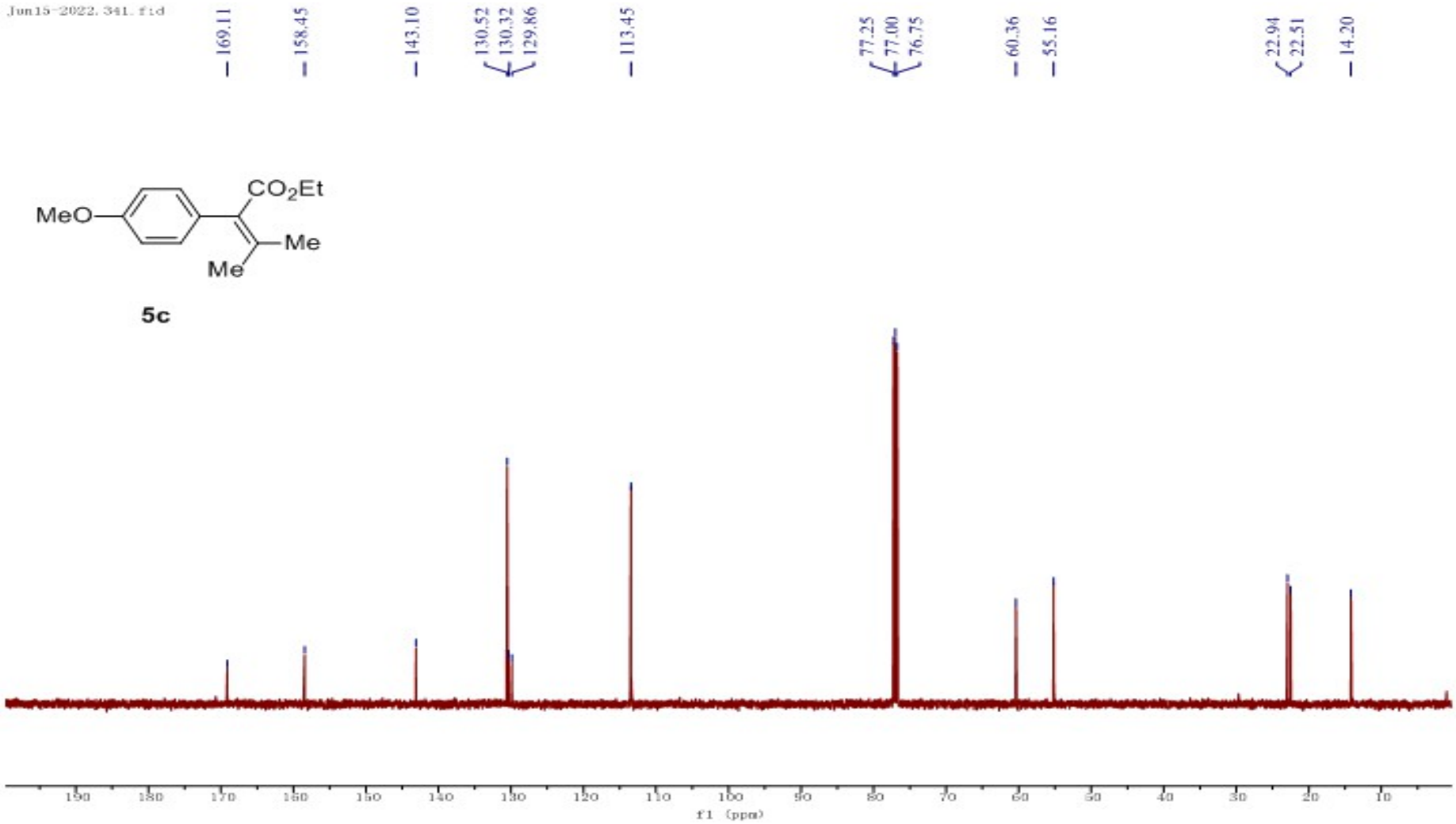
3d

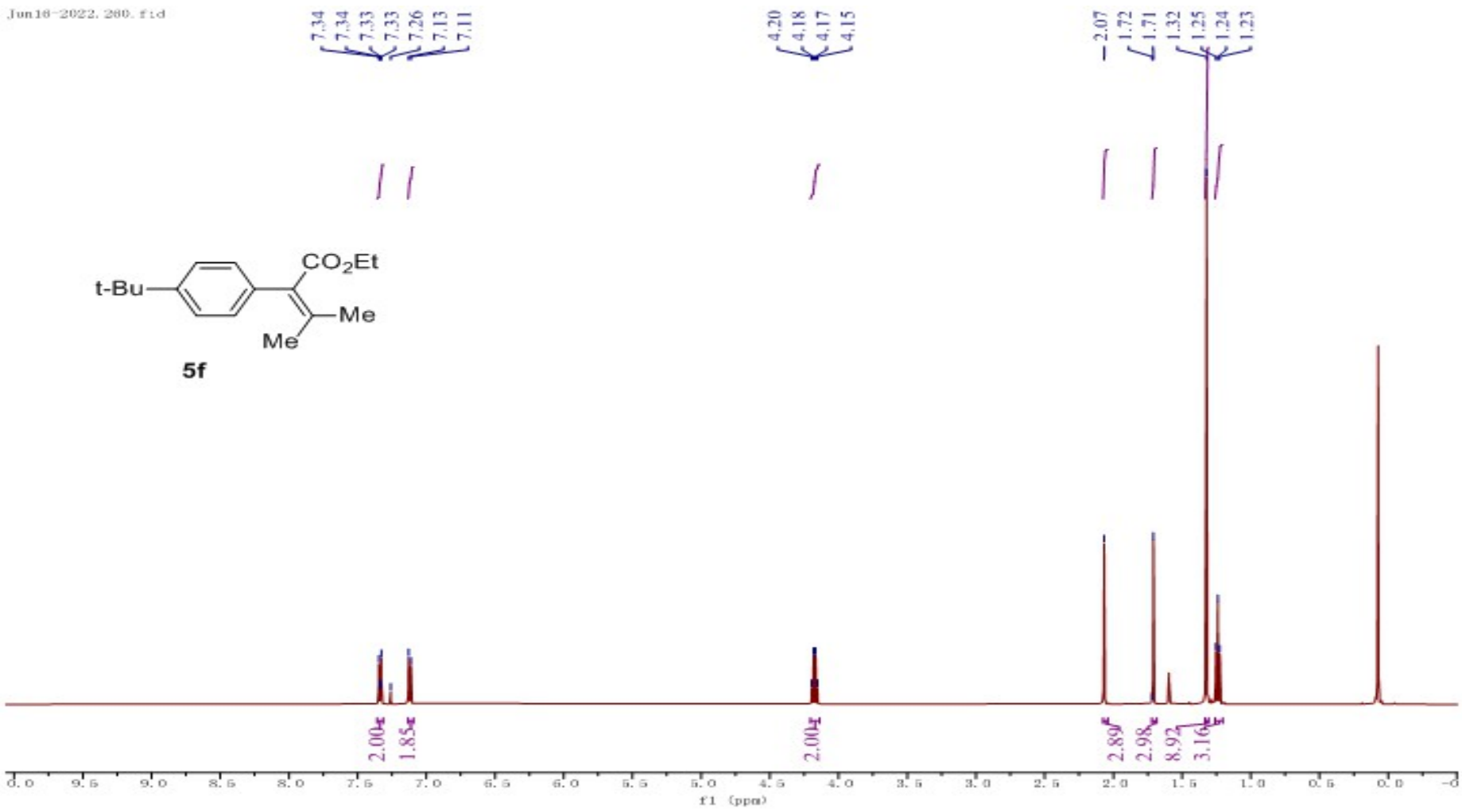

7.44
7.43
7.40
7.39
7.38
7.38
7.37
7.37
7.37
7.36
7.36
7.36
7.35
7.33
7.31
6.41
6.08
6.07
6.06
6.04
5.92
1.64
1.62
3t
0.27
1.00
1.06
1.03
2.96
f1 (ppm)

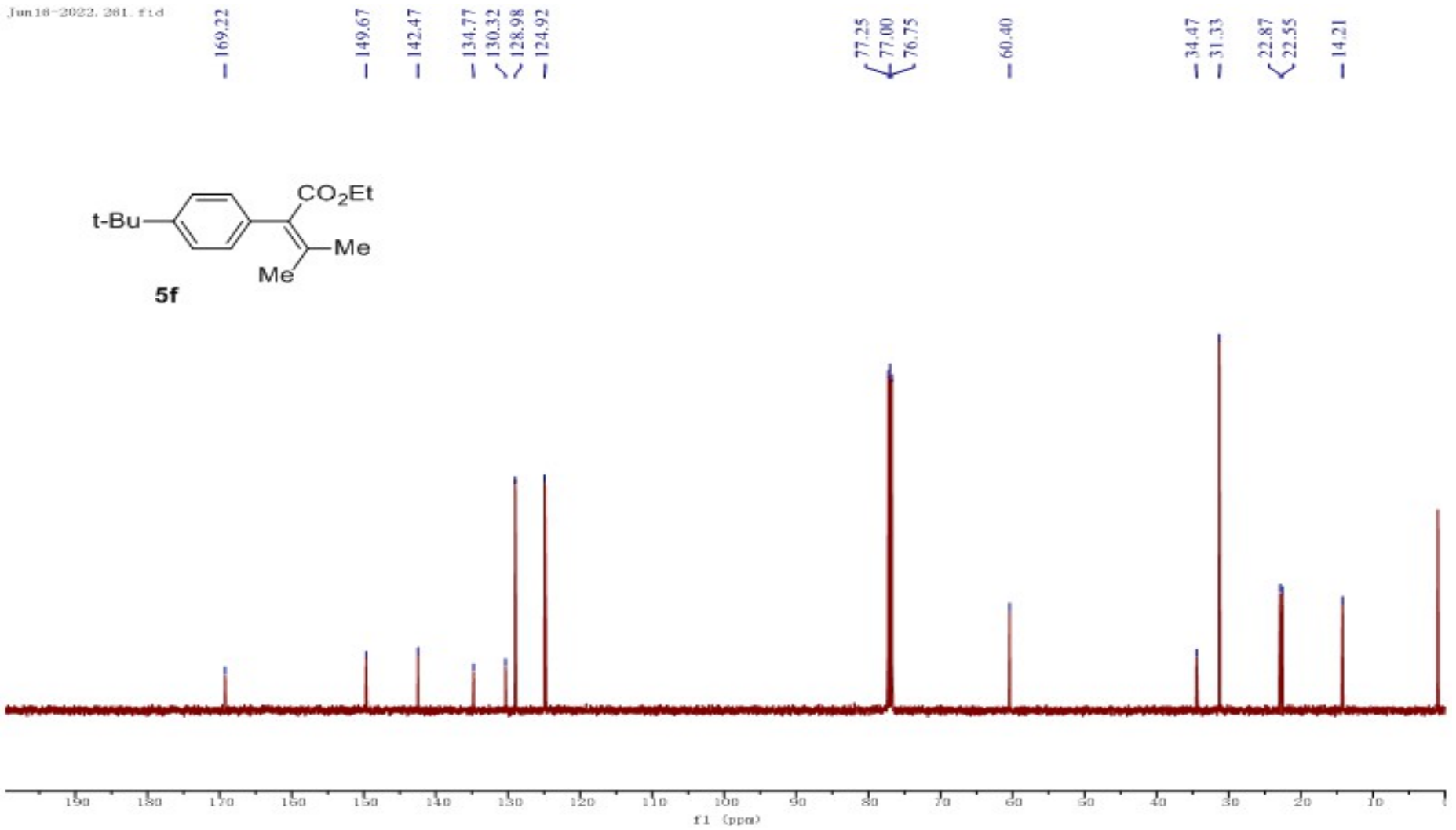

May19-2022.131.fid
165.94
141.58
141.56
136.67
128.50
128.48
128.29
128.27
128.08
128.01
127.85
126.59
126.03
77.25
77.00
76.74
73.13
22.34
f1 (ppm)

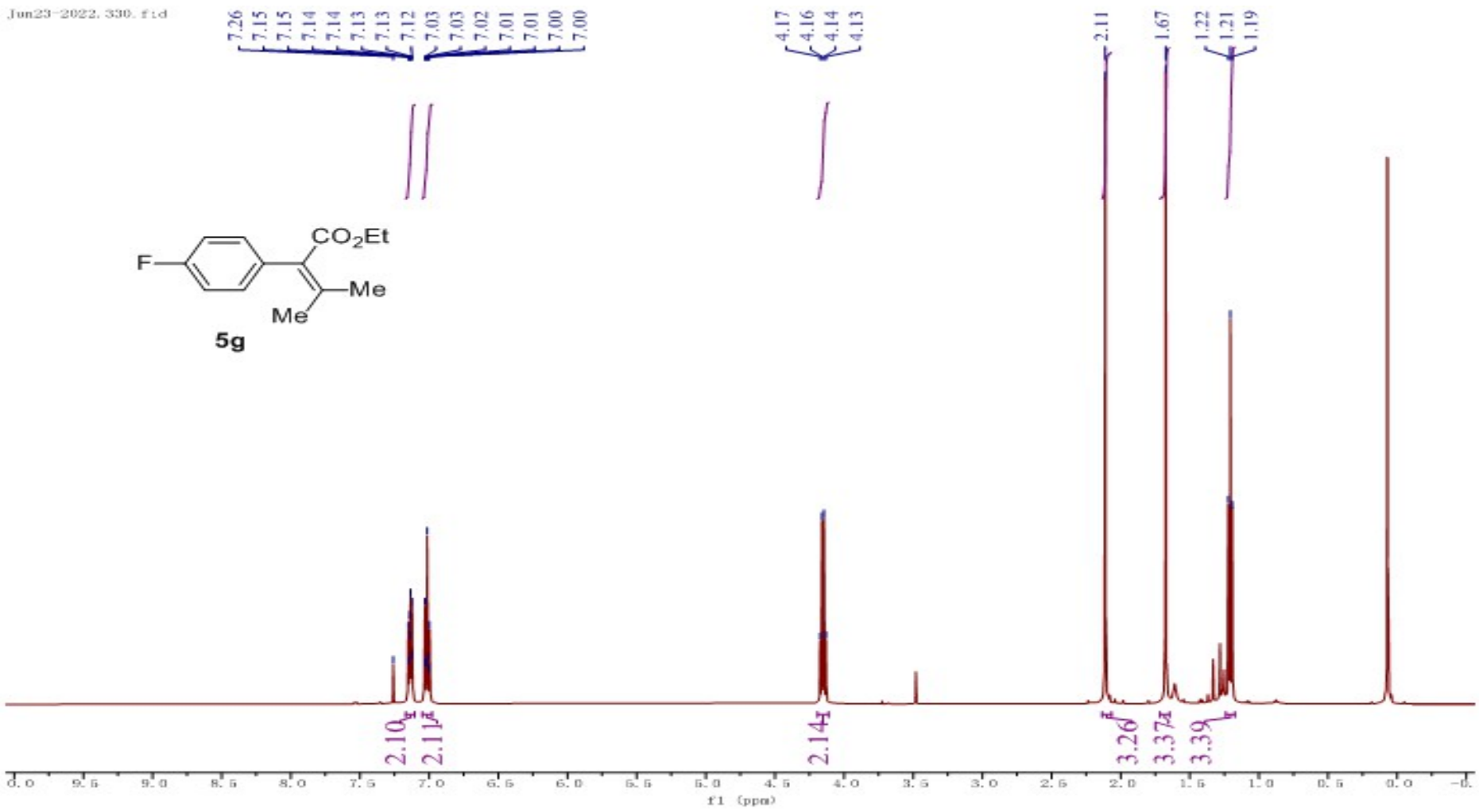
3t

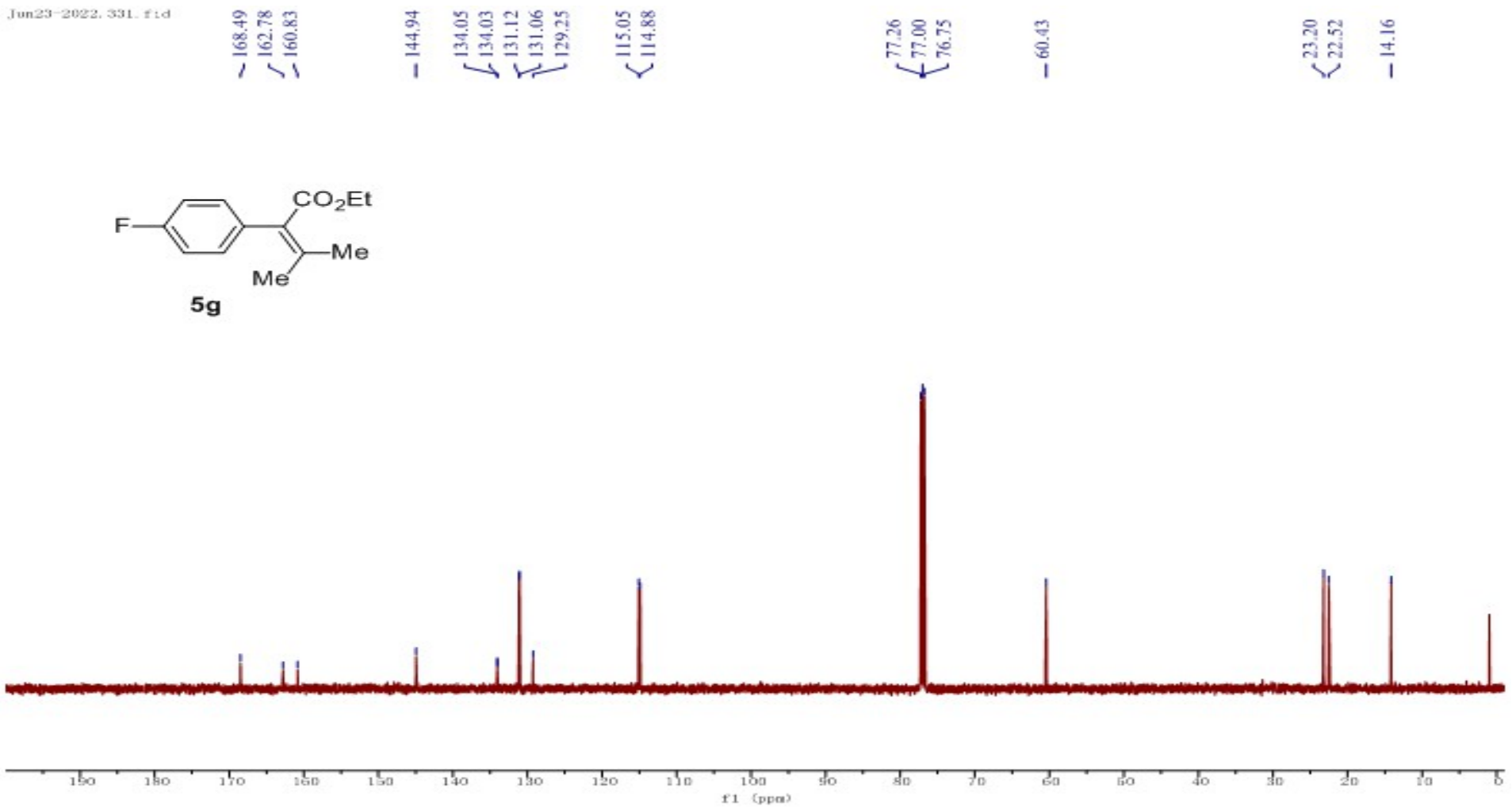

Jun15-2022.340.fid
7.26
7.12
7.12
7.11
7.10
7.10
7.09
6.88
6.88
6.87
6.86
6.86
6.85
4.18
4.17
4.16
4.14
3.81
2.07
1.70
1.24
1.22
1.21
MeO
CO2Et
Me
Me
5c
1.89
1.92
2.00
3.11
3.10
3.11
3.11
f1 (ppm)

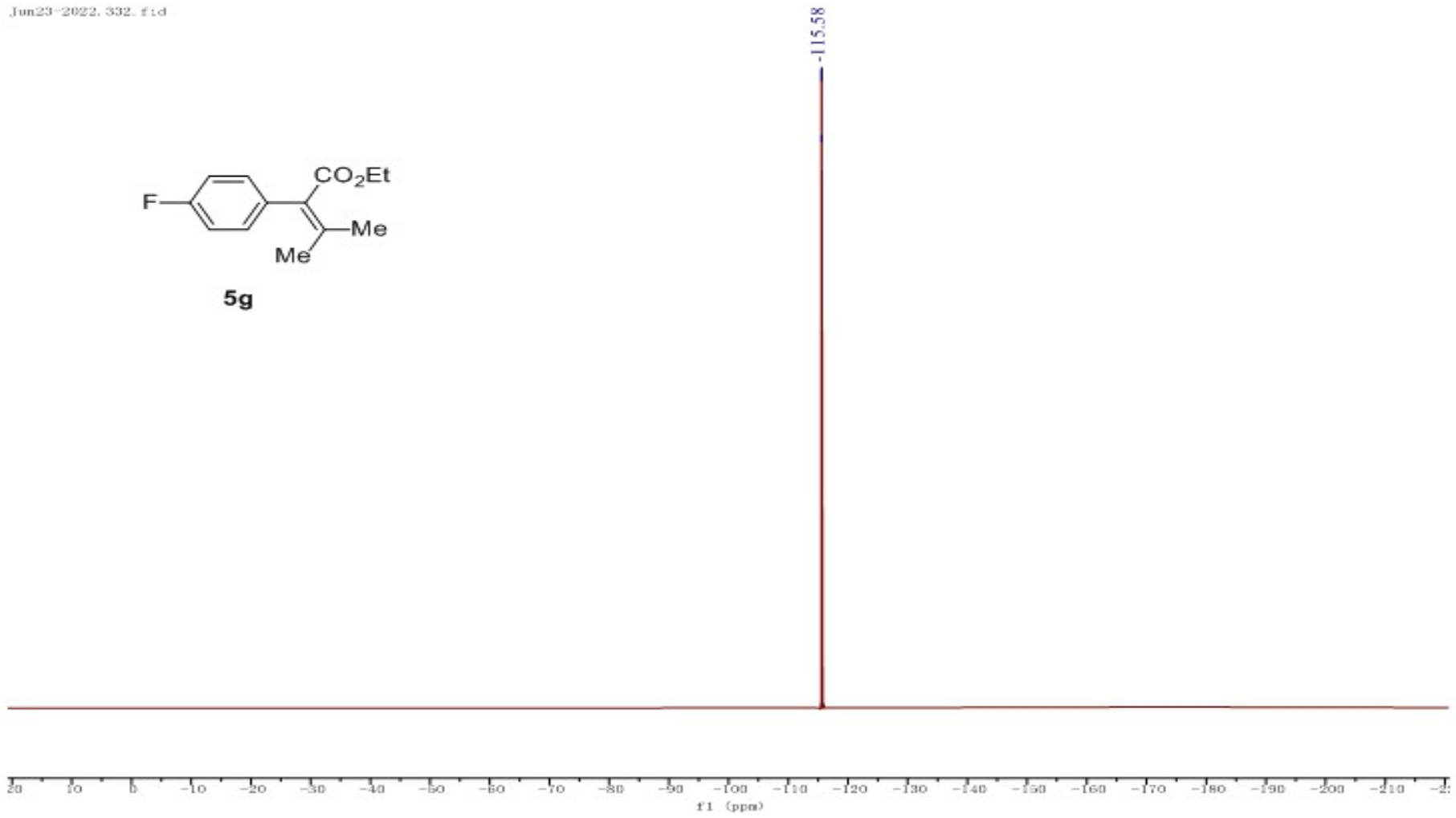

Jun15-2022.341.fid
169.11
158.45
143.10
130.52
130.32
129.86
113.45
77.25
77.00
76.75
60.36
55.16
22.94
22.51
14.20
MeO
CO2Et
Me
Me
5c
f1 (ppm)


Jun16-2022.280.fid
7.34
7.34
7.33
7.33
7.26
7.13
7.11
4.20
4.18
4.17
4.15
2.07
1.72
1.71
1.32
1.25
1.24
1.23
t-Bu
CO2Et
Me
Me
5f
2.00
1.85
2.00
2.89
2.98
8.92
3.16
f1 (ppm)

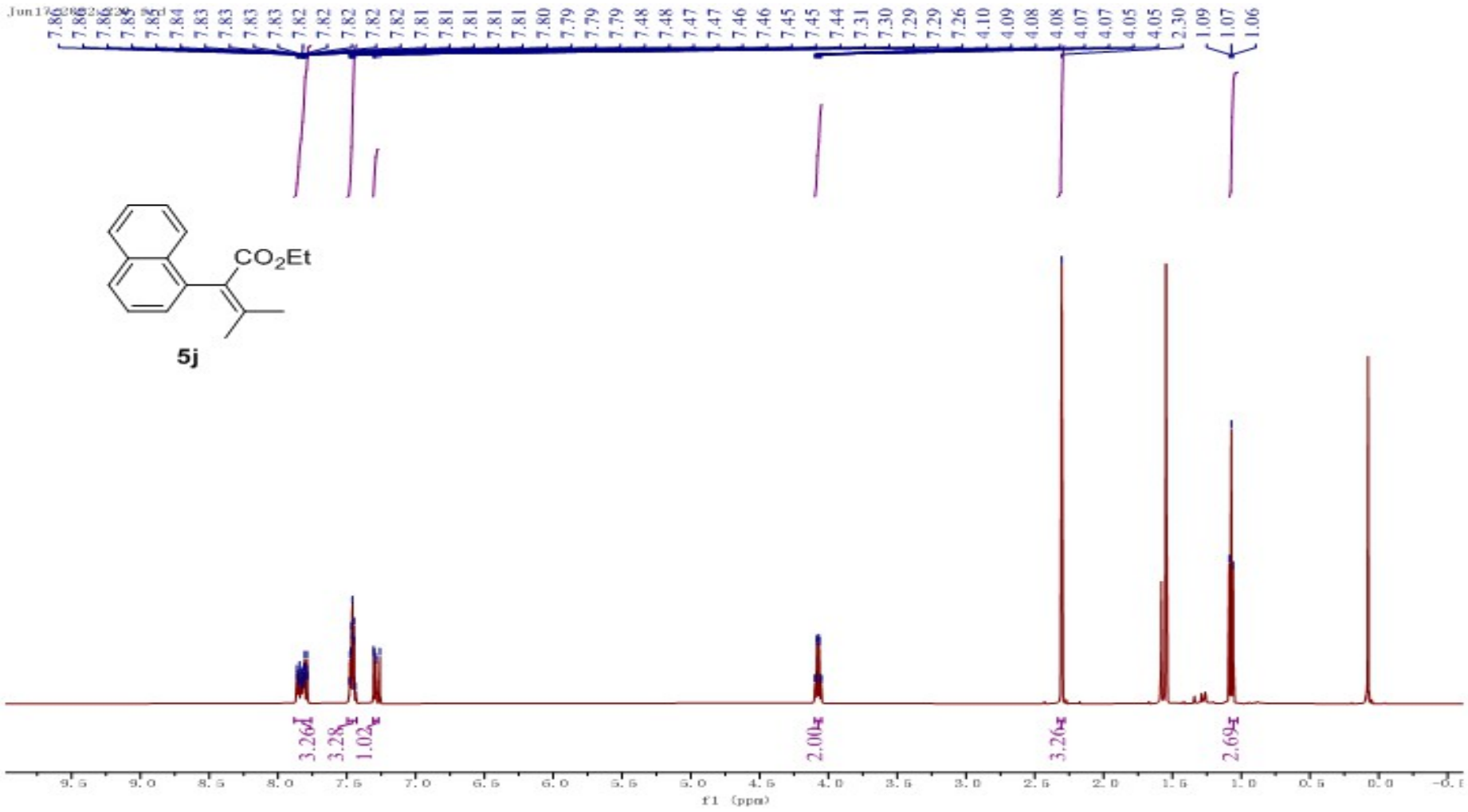

Jun16-2022.281.fid
169.22
149.67
142.47
134.77
130.32
128.98
124.92
77.25
77.00
76.75
60.40
34.47
31.33
22.87
22.55
14.21
t-Bu
CO2Et
Me
Me
5f
f1 (ppm)

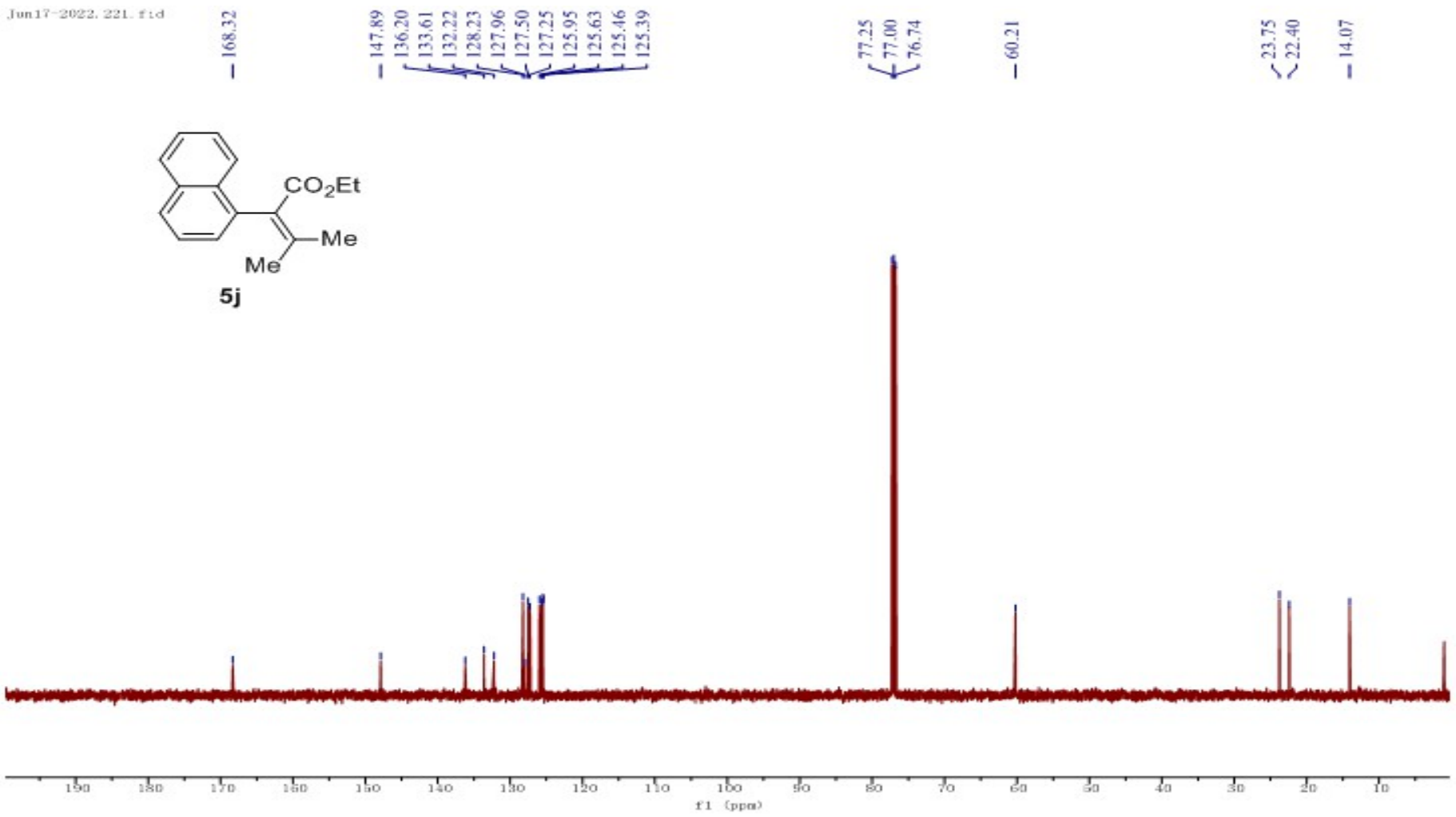

Jun23-2022.330.fid
7.26
7.15
7.15
7.14
7.14
7.13
7.13
7.12
7.03
7.03
7.02
7.01
7.01
7.00
7.00
4.17
4.16
4.14
4.13
2.11
1.67
1.22
1.21
1.19
CO2Et
F
Me
Me
5g
2.10
2.11
2.14
3.26
3.37
3.39
f1 (ppm)

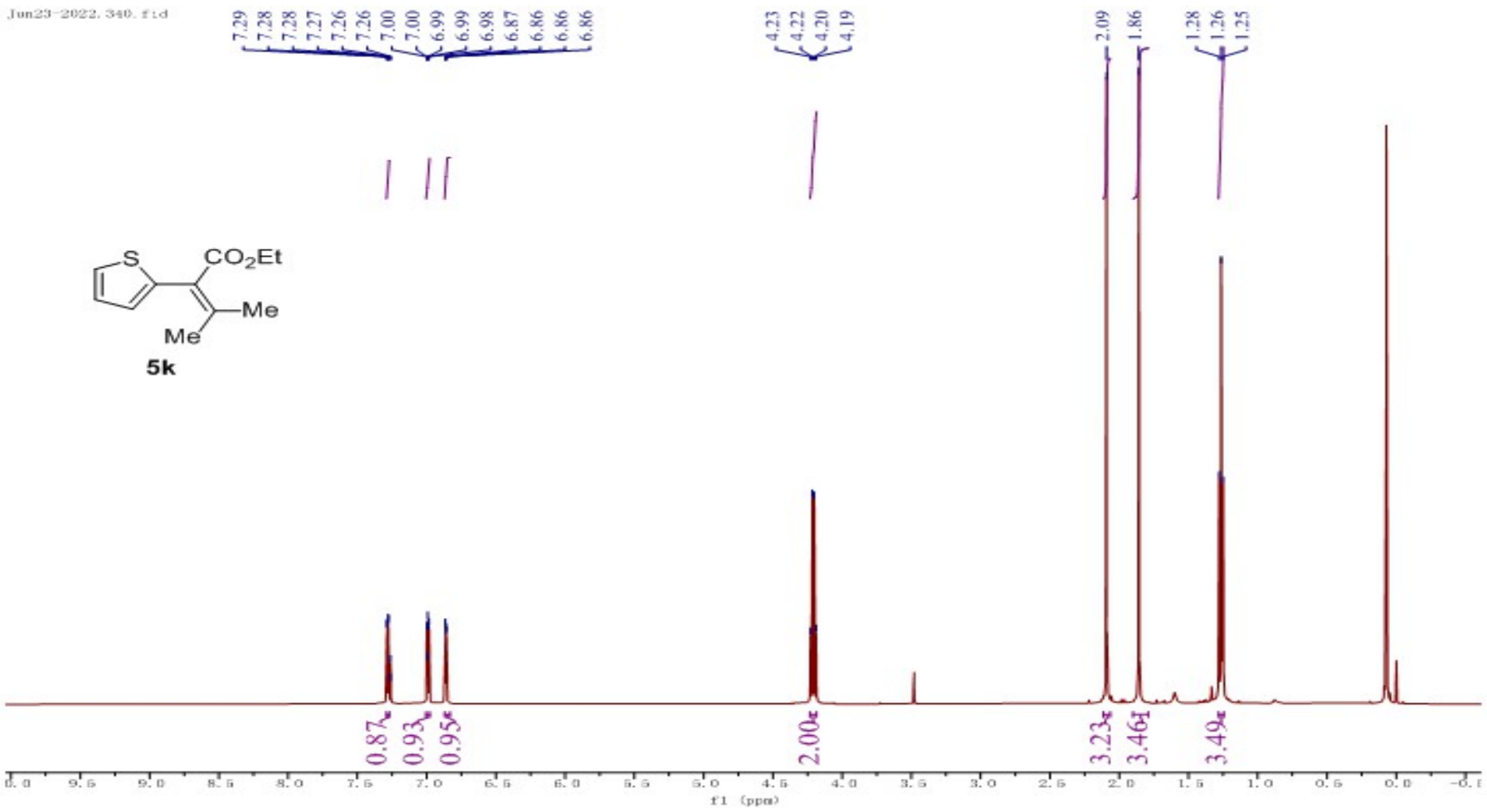

Jun23-2022.331.fid
168.49
162.78
160.83
144.94
134.05
134.03
131.12
131.06
129.25
115.05
114.88
77.26
77.00
76.75
60.43
23.20
22.52
14.16
F
CO2Et
Me
Me
5g
f1 (ppm)

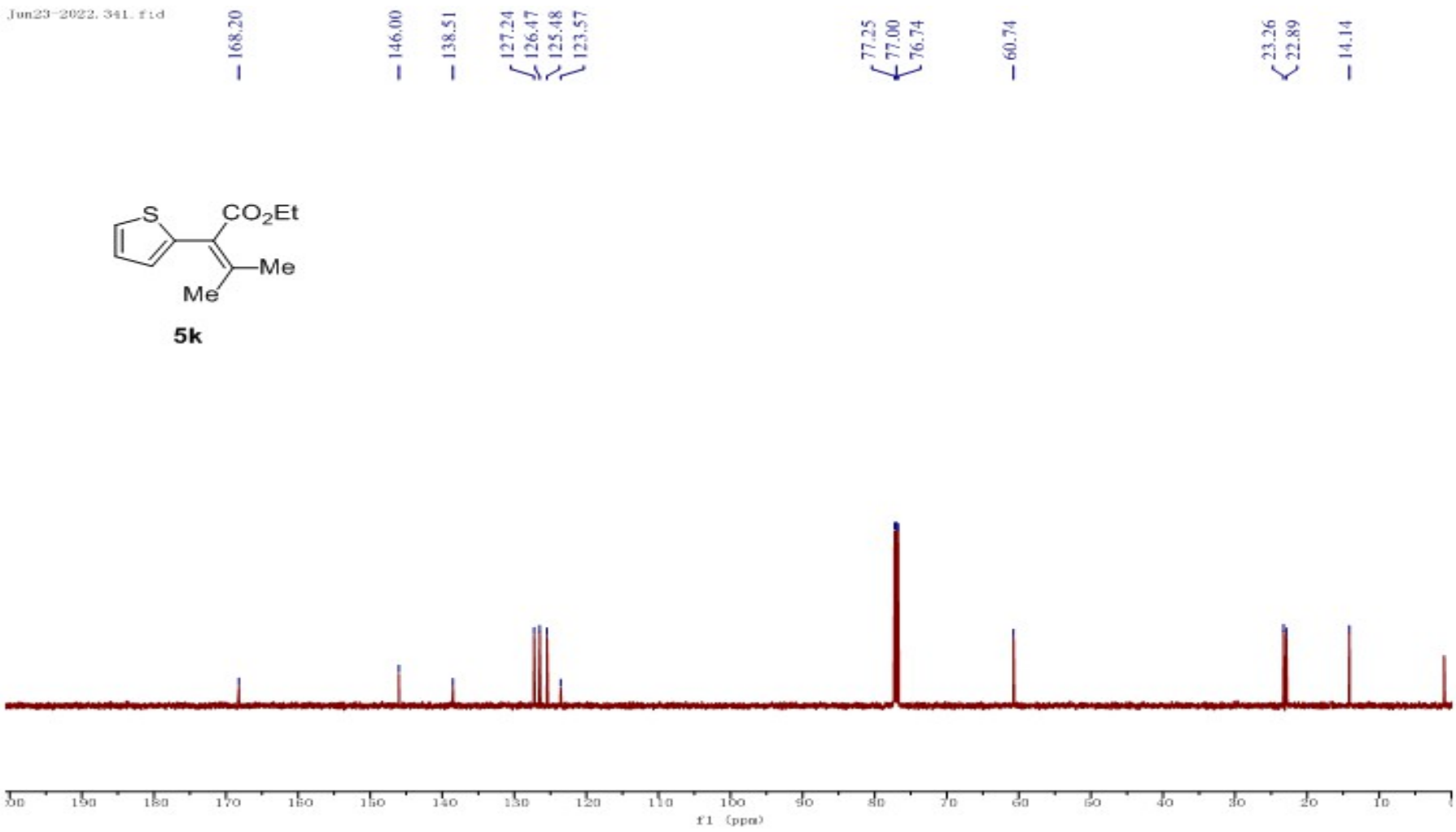


5g

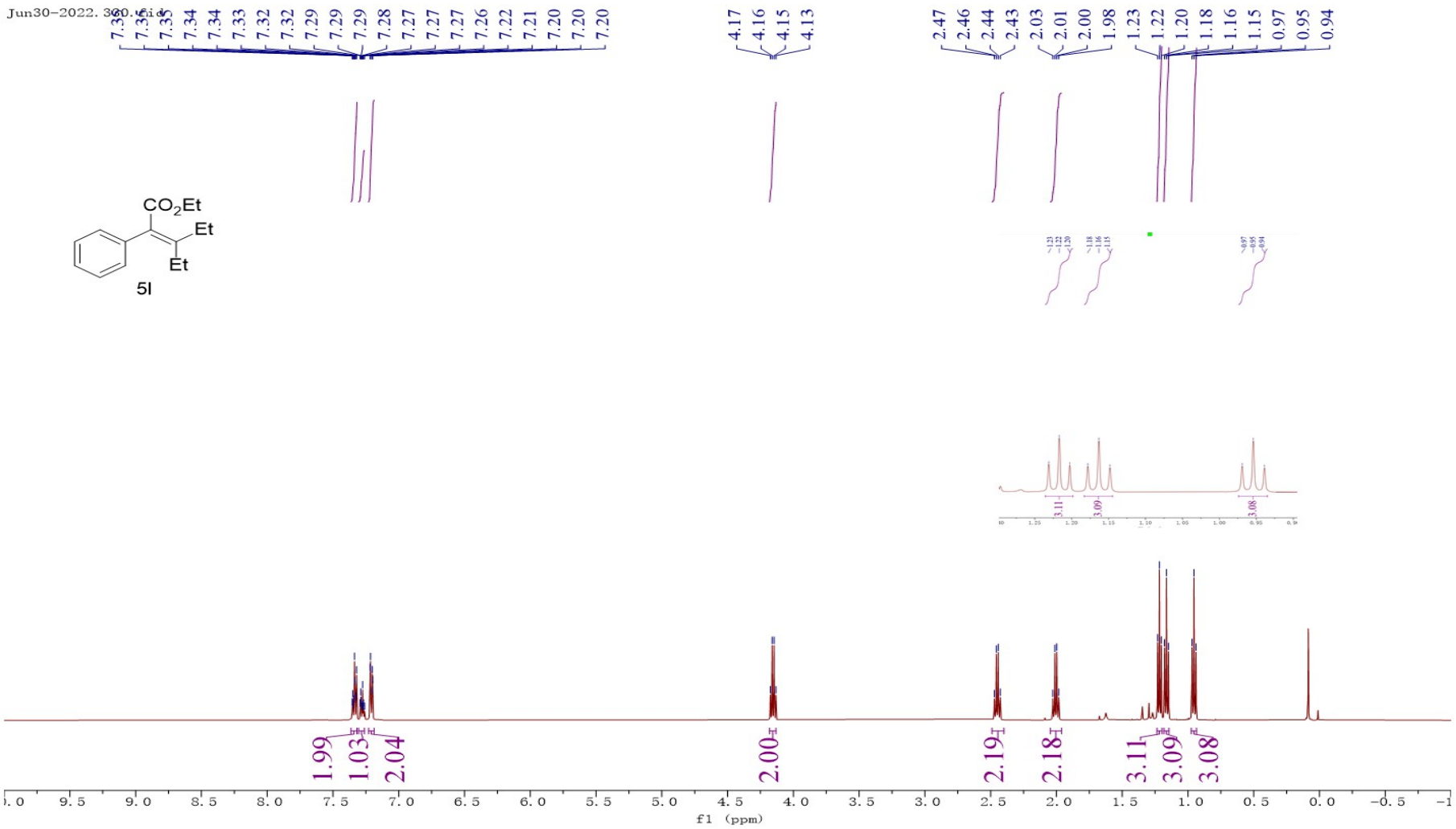

CO2Et
5j
3.26
3.28
1.02
2.00
3.26
2.69
f1 (ppm)

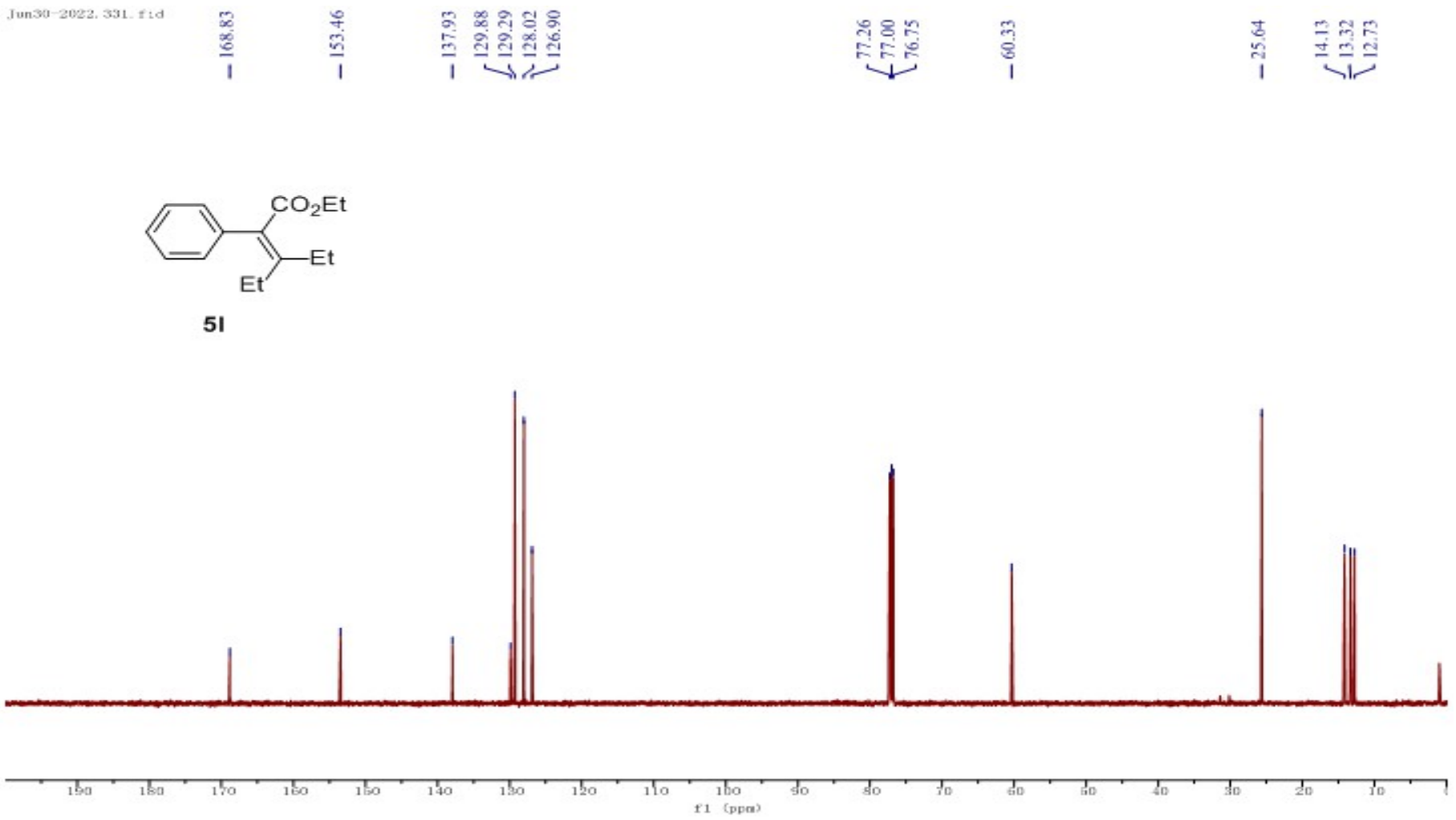

Jun17-2022.221.fid
168.32
147.89
136.20
133.61
132.22
128.23
127.96
127.50
127.25
125.95
125.63
125.46
125.39
77.25
77.00
76.74
60.21
23.75
22.40
14.07
CO2Et
Me
Me
5j
f1 (ppm)

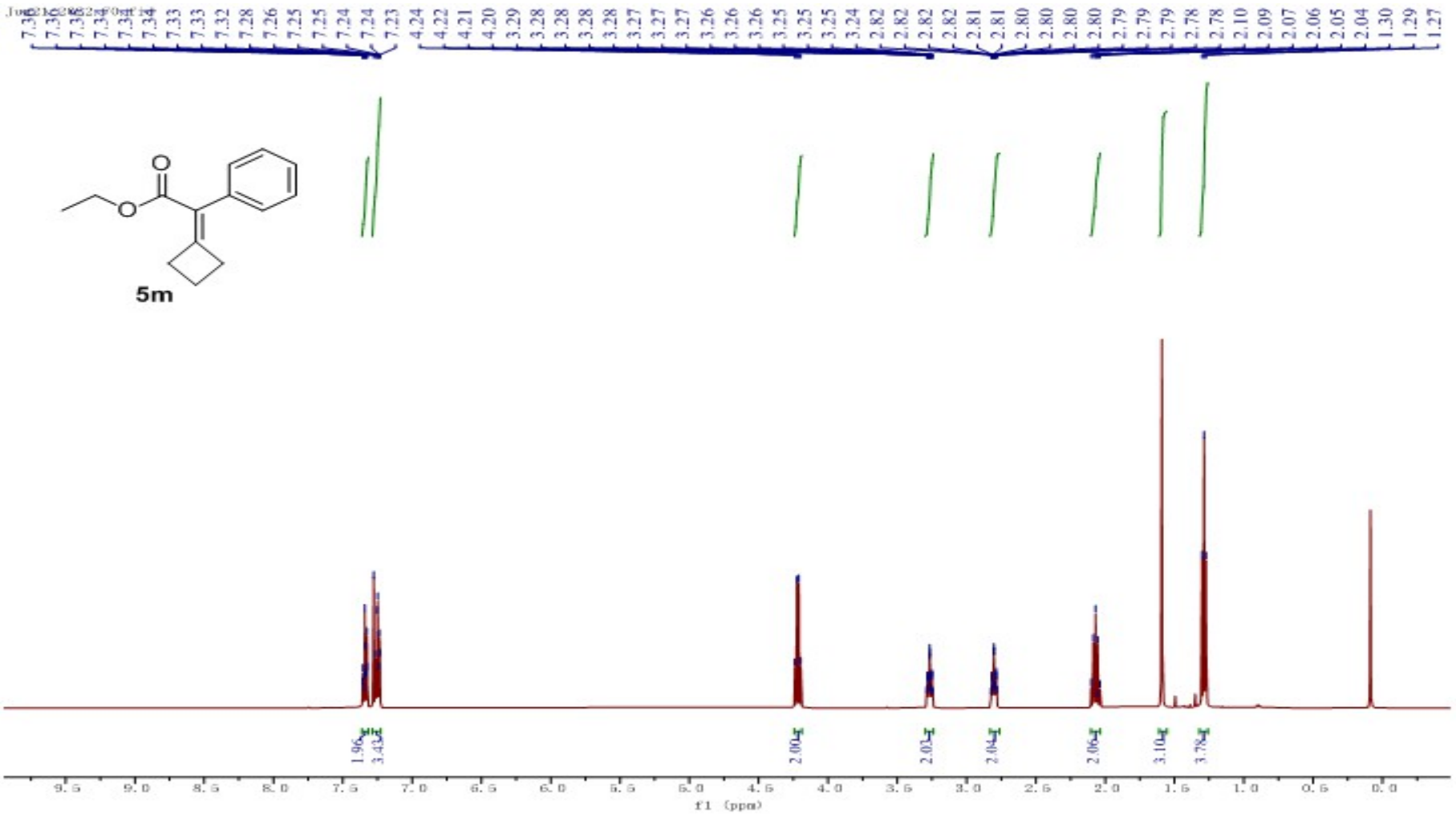

Jun23-2022.340.fid
7.29
7.28
7.28
7.27
7.26
7.26
7.00
7.00
6.99
6.99
6.98
6.87
6.86
6.86
6.86
4.23
4.22
4.20
4.19
2.09
1.86
1.28
1.26
1.25
S
CO2Et
Me
Me
5k
0.87
0.93
0.95
2.00
3.23
3.46
3.49
f1 (ppm)

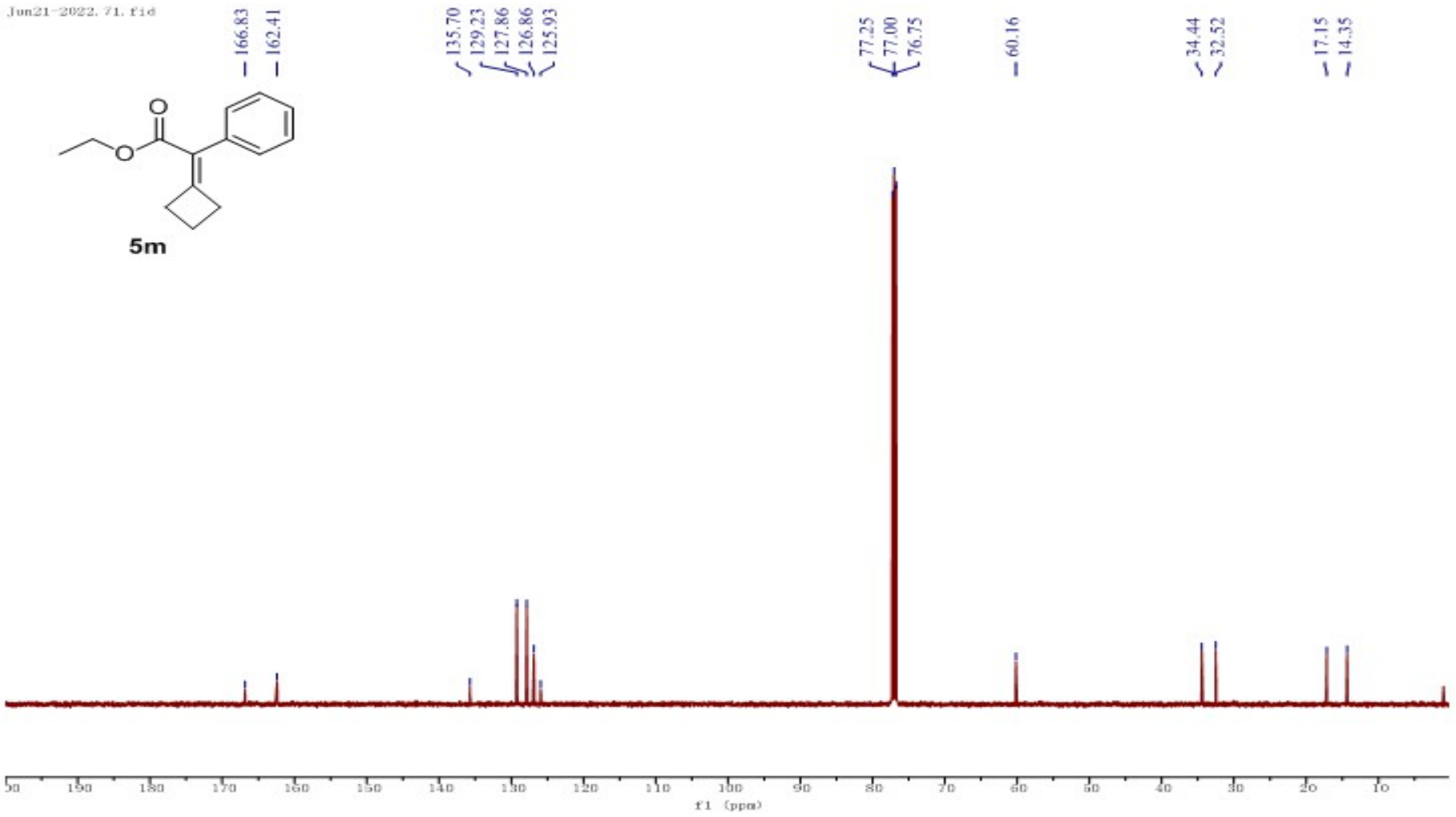

Jun23-2022.341.fid
168.20
146.00
138.51
127.24
126.47
125.48
123.57
77.25
77.00
76.74
60.74
23.26
22.89
14.14
S
CO2Et
Me
Me
f1 (ppm)

5k


Jun30-2022.300.fid
7.36 7.35 7.35 7.34 7.34 7.33 7.32 7.32 7.29 7.29 7.29 7.28 7.27 7.27 7.27 7.26 7.22 7.21 7.20 7.20 7.20
4.17 4.16 4.15 4.13
2.47 2.46 2.44 2.43 2.03 2.01 2.00 1.98 1.23 1.22 1.20 1.18 1.16 1.15 0.97 0.95 0.94
CO2Et
Et
Et
5l
1.99 1.03 2.04 2.00 2.19 2.18 3.11 3.09 3.08
f1 (ppm)


Jun30-2022.331.fid
168.83
153.46
137.93
129.88
129.29
128.02
126.90
77.26
77.00
76.75
60.33
25.64
14.13
13.32
12.73
CO2Et
Et
Et
5l
f1 (ppm)


5m
f1 (ppm)


Jun21-2022.71.fid
166.83
162.41
135.70
129.23
127.86
126.86
125.93
77.25
77.00
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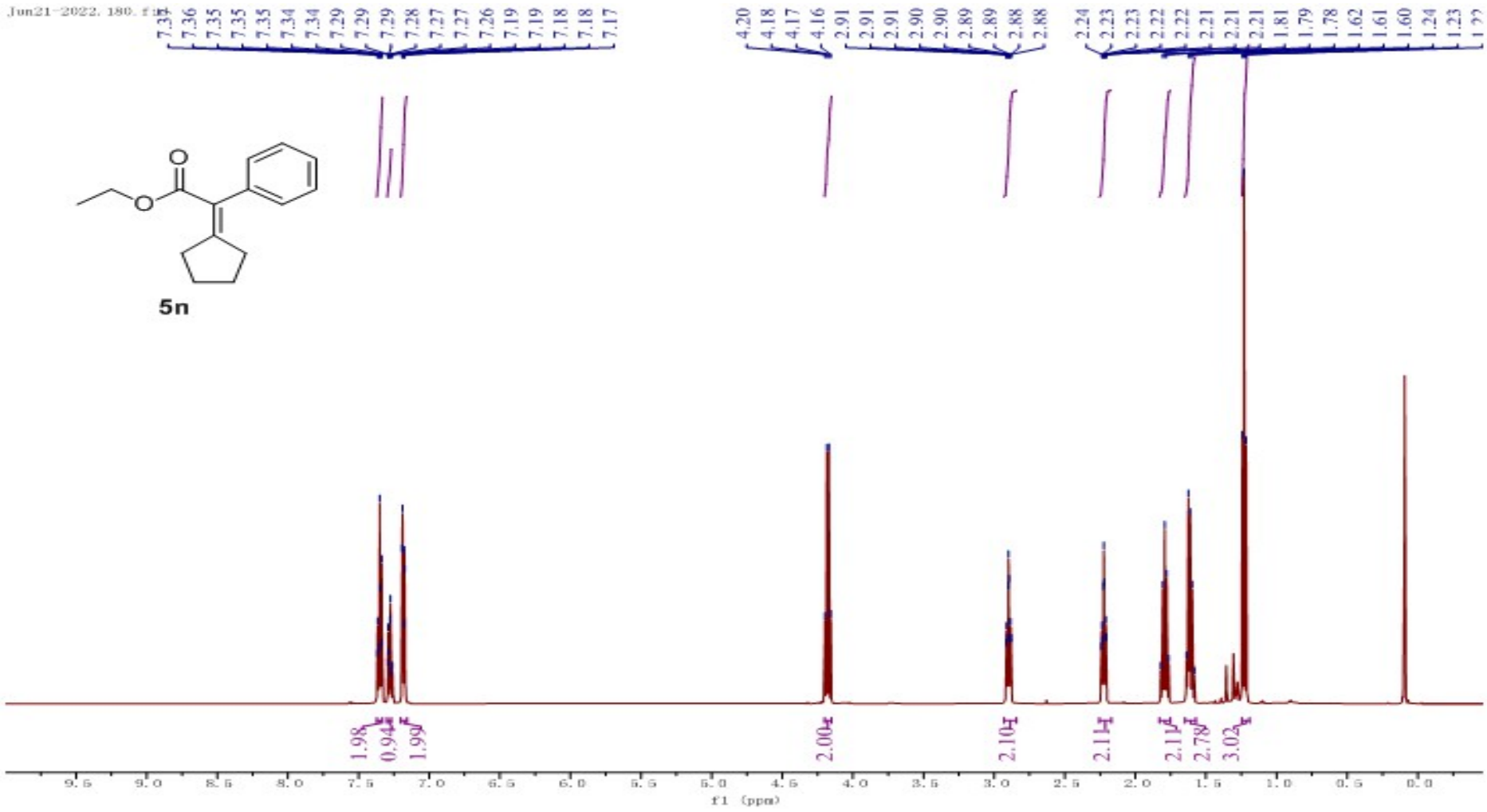
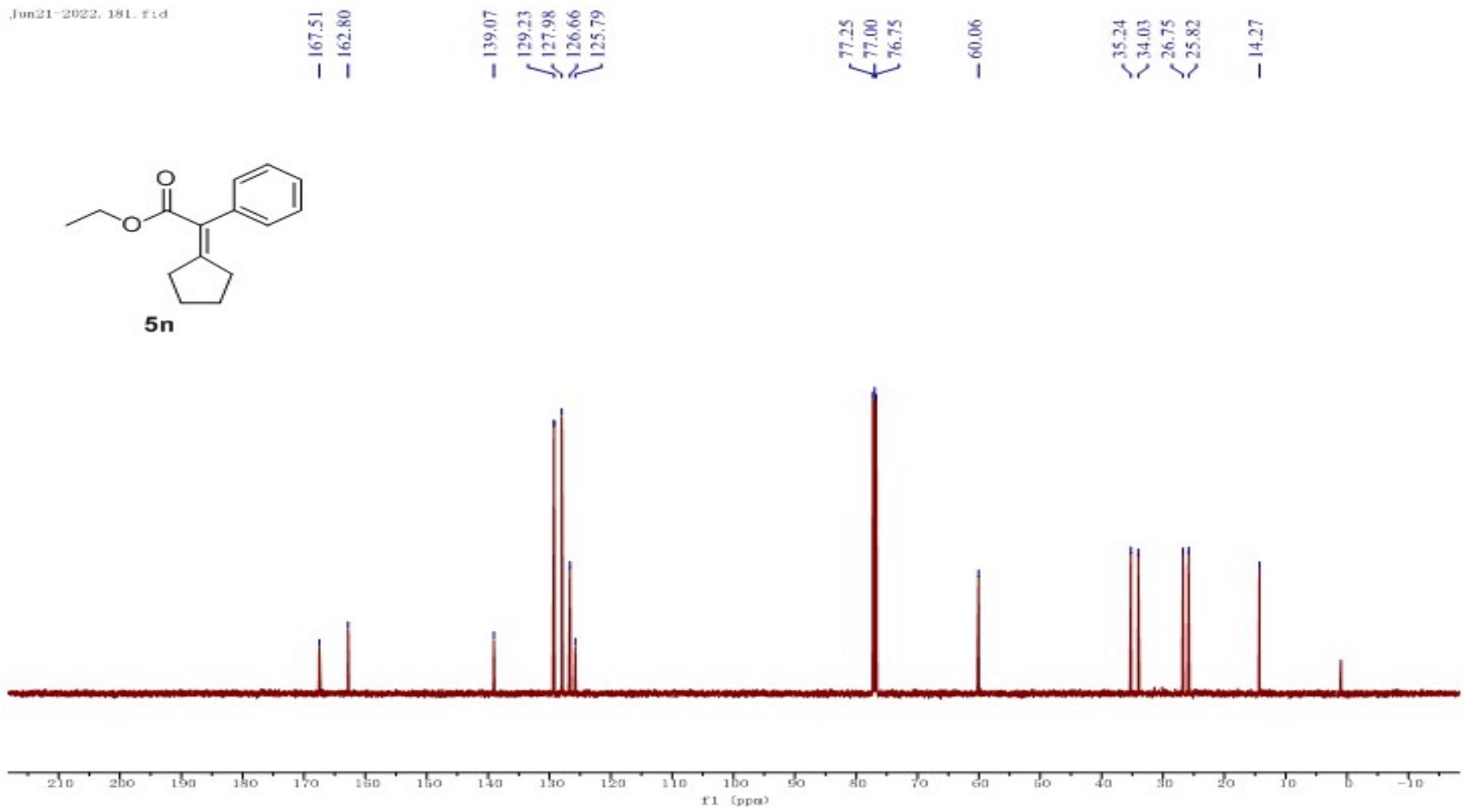
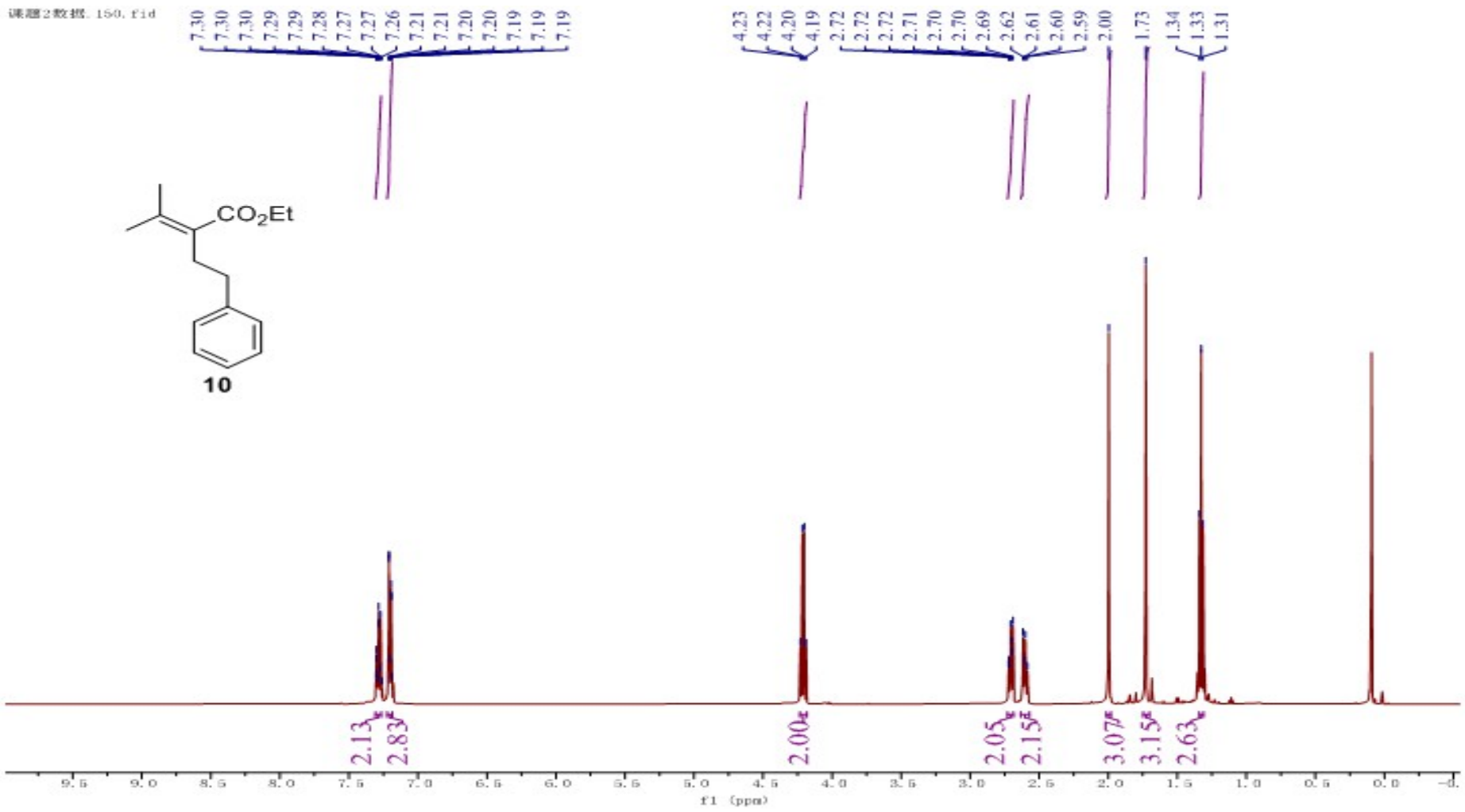
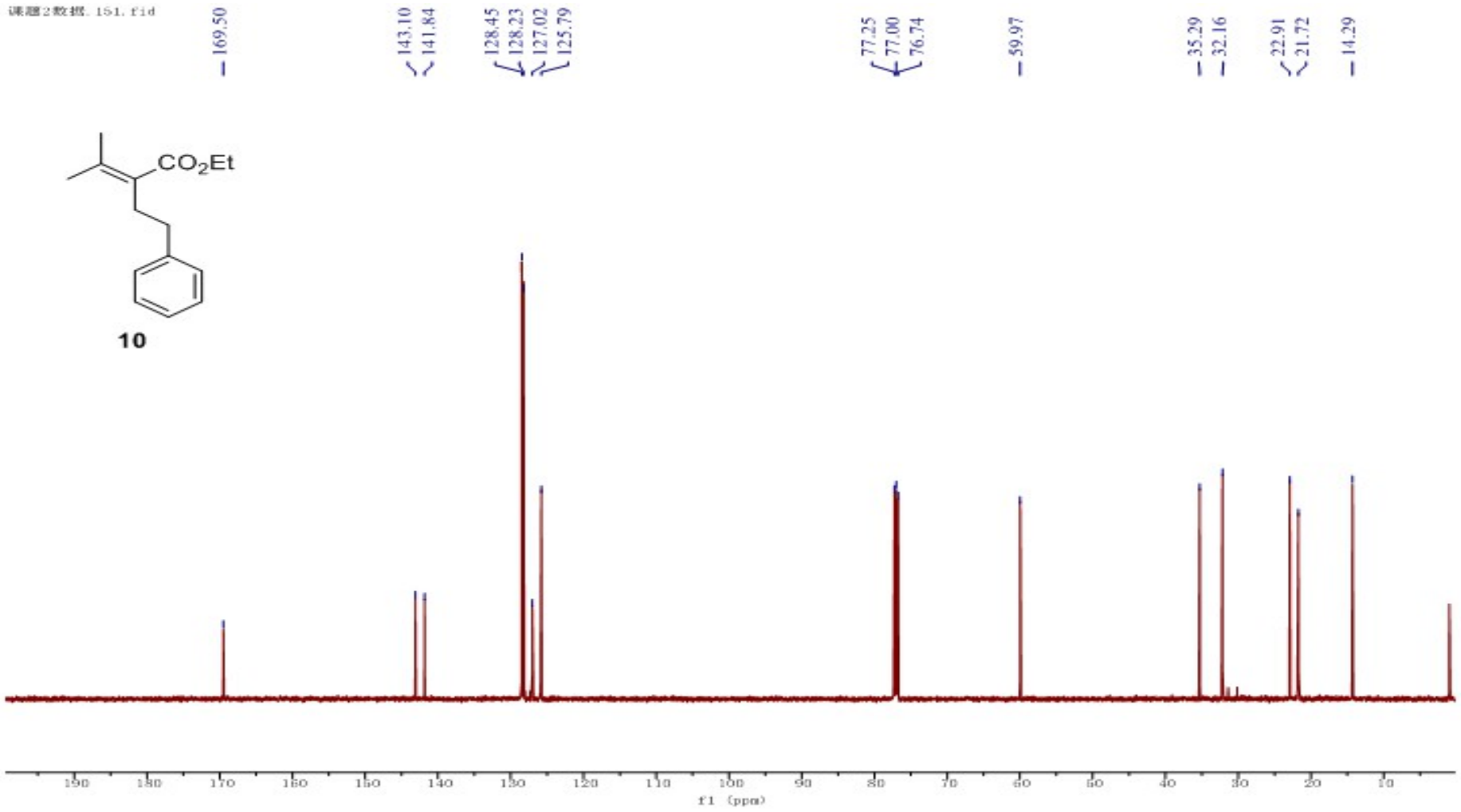
34.44
32.52
17.15
14.35
5m
f1 (ppm)


Jun21-2022. 180. fid
7.37
7.36
7.35
7.35
7.35
7.34
7.34
7.29
7.29
7.29
7.28
7.27
7.27
7.26
7.19
7.19
7.18
7.18
7.17
4.20
4.18
4.17
4.16
2.91
2.91
2.91
2.90
2.90
2.89
2.89
2.88
2.88
2.24
2.23
2.23
2.22
2.22
2.21
2.21
2.21
1.81
1.79
1.78
1.62
1.61
1.60
1.24
1.23
O
O
5n
1.98
0.94
1.99
2.00
2.10
2.11
2.11
2.78
3.02
f1 (ppm)


Jun21-2022.181.fid
167.51
162.80
139.07
129.23
127.98
126.66
125.79
77.25
77.00
76.75
60.06
35.24
34.03
26.75
25.82
14.27
5n
f1 (ppm)


课题2数据. 150. fid
7.30
7.30
7.30
7.29
7.29
7.28
7.27
7.27
7.26
7.21
7.21
7.20
7.20
7.19
7.19
7.19
4.23
4.22
4.20
4.19
2.72
2.72
2.72
2.71
2.70
2.70
2.69
2.62
2.61
2.60
2.59
2.00
1.73
1.34
1.33
1.31
CO2Et
10
2.13
2.83
2.00
2.05
2.15
3.07
3.15
2.63
f1 (ppm)


课题2数据. 151. fid
169.50
143.10
141.84
128.45
128.23
127.02
125.79
77.25
77.00
76.74
59.97
35.29
32.16
22.91
21.72
14.29
CO2Et
10
f1 (ppm)