

Supporting Information

Synthesis of Aromatic β -Keto Esters via a Carbonylative Suzuki-Miyaura Coupling
Reaction of α -Iodo Esters with Arylboronic Acids

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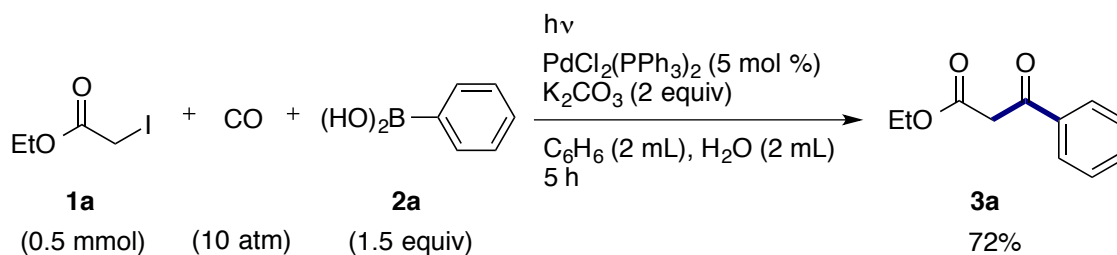
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General Information

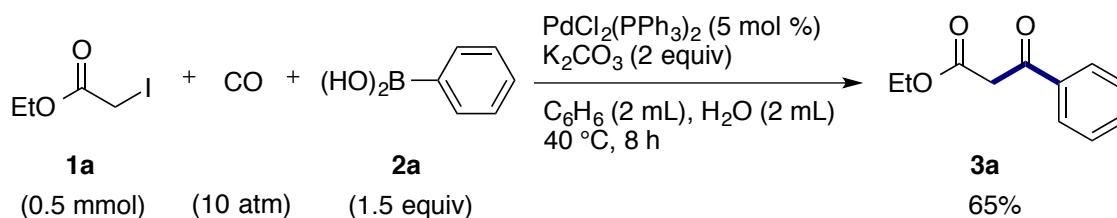
Thin layer chromatography (TLC) was performed on Merck precoated plates (silica gel 60 F254, Art 5715, 0.25 mm) and were visualized by fluorescence quenching under UV light or by staining with *p*-anisaldehyde/AcOH/H₂SO₄/EtOH, or 12MoO₃.H₃PO₄/EtOH. The products were purified by flash chromatography on silica gel (Kanto Chem. Co. Silica Gel 60N (spherical, neutral, 40-50 mm)) and, if necessary, were further purified by recycling preparative HPLC (Japan Analytical Industry Co. Ltd., LC-918) equipped with GPC columns (JAIGEL-1H + JAIGEL-2H columns) using CHCl₃ as eluent. ¹H NMR spectra were recorded with a JEOL JMN-ECS400 (400 MHz) spectrometer and referenced to the solvent peak at 7.26 ppm. ¹³C NMR spectra were recorded with a JEOL JMN-ECS400 (100 MHz) spectrometer and referenced to the solvent peak at 77.16 ppm. ¹⁹F NMR spectra were recorded with a JEOL JMN-ECS400 (376 MHz) spectrometer. All the fluorine NMR spectra were calibrated using CF₃COOH (-76.55 ppm). Splitting patterns are indicated as follows: br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Infrared spectra were recorded on a JASCO FT/IR-4100 spectrometer and are reported as wavenumber (cm⁻¹). High-resolution mass spectra were recorded with a JEOL MS700 spectrometer. Benzene was not distilled for this reaction.

Typical Procedure for the Synthesis of **3a** in Light Conditions



A magnetic stirring bar, **1a** (121.9 mg, 0.5 mmol), **2a** (91.7 mg, 0.75 mmol), PdCl₂(PPh₃)₂ (17.4 mg, 0.025 mmol), K₂CO₃ (139.1 mg, 1.0 mmol), C₆H₆ (2 mL) and H₂O (2 mL) were placed in a stainless steel autoclave for photoreaction equipped with an inserted Pyrex glass liner. The autoclave was closed, purged three times with carbon monoxide, pressurized with 10 atm of CO and then irradiated by black light (15 W × 2) with stirring for 5 h. Excess CO was discharged at room temperature after the reaction. The reaction mixture was added to water (20 mL) and extracted with ether (20 mL × 3). The organic layer was washed with brine, and dried over MgSO₄, then filtered and concentrated *in vacuo* to give a residue, which was subjected to silica gel column chromatography using hexane/EtOAc = 30/1 as eluent affording **3a** (69.2 mg, 72%).

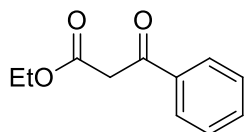
Typical Procedure for the Synthesis of **3a** in Thermal Conditions



A magnetic stirring bar, **1a** (120.5 mg, 0.5 mmol), **2a** (91.1 mg, 0.75 mmol), PdCl₂(PPh₃)₂ (17.8 mg, 0.025 mmol), K₂CO₃ (138.1 mg, 1.0 mmol), C₆H₆ (2 mL) and H₂O (2 mL) were placed in a stainless steel autoclave. The autoclave was closed, purged three times with CO, pressurized with 10 atm of CO and then heated in 40 °C for 8 h. Excess CO was discharged at room temperature after the reaction. The reaction mixture was added to water (20 mL) and extracted with ether (20 mL × 3). The organic layer was washed with brine, and dried over MgSO₄, then filtered and concentrated *in vacuo* to give a residue, which was subjected to silica gel column chromatography using hexane/EtOAc = 30/1 as eluent affording **3a** (62.3 mg, 65%).

Spectral Data

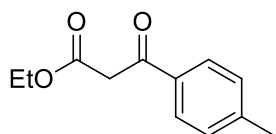
Ethyl 3-oxo-3-phenylpropanoate (3a)



This product is commercially available.

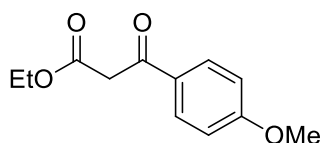
Light brown oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 7.96-7.94 (m, 2H), 7.62-7.59 (m, 1H), 7.51-7.47 (m, 2H), 4.22 (q, $J = 7.2$ Hz, 2H), 4.00 (s, 2H), 1.26 (t, $J = 7.2$ Hz, 3H), Enol Form 12.58 (s, 1H), 7.79-7.77 (m, 2H), 7.47-7.41 (m, 3H), 5.67 (s, 1H), 4.27 (q, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ Keto Form 192.67, 167.65, 136.11, 133.85, 128.89, 128.61, 61.59, 46.11, 14.18, Enol Form 173.32, 171.54, 133.54, 131.35, 128.61, 126.15, 87.49, 60.44, 14.41.

Ethyl 3-oxo-3-(*p*-tolyl)propanoate (3b)



Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 7.86-7.84 (m, 2H), 7.29-7.27 (m, 2H), 4.21 (q, $J = 7.6$ Hz, 2H), 3.97 (s, 2H), 2.42 (s, 3H), 1.26 (t, $J = 7.6$ Hz, 3H), Enol Form 12.58 (s, 1H), 7.69-7.67 (m, 2H), 7.24-7.21 (m, 2H), 5.64 (s, 1H), 4.26 (q, $J = 6.8$ Hz, 2H), 2.40 (s, 3H), 1.33 (t, $J = 6.8$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ Keto Form 192.21, 167.71, 144.74, 133.61, 129.50, 128.85, 61.45, 45.98, 21.73, 14.12, Enol Form 173.34, 171.65, 141.77, 130.65, 129.30, 126.03, 86.66, 60.27, 21.51, 14.36; IR (neat) 2984 cm^{-1} , 1746 cm^{-1} , 1682 cm^{-1} ; MS (relative intensity) m/z 206 (M^+ , 77), 119 (100), 91 (70); HRMS (EI) m/z calcd for $\text{C}_{12}\text{H}_{14}\text{O}_3$ 206.0943, found 206.0948.

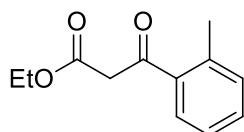
Ethyl 3-(4-methoxyphenyl)-3-oxopropanoate (3c)



Light brown oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 7.95-7.92 (m, 2H), 6.97-6.94 (m, 2H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.95 (s, 2H), 3.88 (s, 3H), 1.26 (t, $J = 7.2$ Hz, 3H), Enol Form 12.64 (s, 1H), 7.75-7.73 (m, 2H), 6.94-6.91 (m, 2H), 5.58 (s, 1H), 4.26 (q, $J = 7.2$ Hz, 2H),

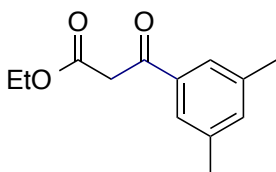
3.85 (s, 3H), 1.33 (t, $J = 7.2$ Hz, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ Keto Form 191.10, 167.87, 164.09, 131.00, 129.20, 114.03, 61.51, 55.63, 45.93, 14.20, Enol Form 173.47, 171.49, 162.21, 132.32, 125.88, 113.55, 85.80, 60.26, 55.50, 14.44; IR (neat) 2984 cm^{-1} , 1747 cm^{-1} , 1513 cm^{-1} , 1025 cm^{-1} ; MS (relative intensity) m/z 222 (M^+ , 25), 135 (100); HRMS (EI) m/z calcd for $\text{C}_{12}\text{H}_{14}\text{O}_4$ 222.0892, found 222.0891.

Ethyl 3-oxo-3-(*o*-tolyl)propanoate (3d)



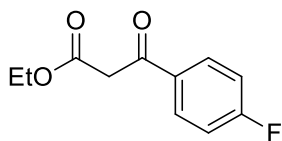
Light brown oil; ^1H -NMR (CDCl_3 , 400 MHz); δ Keto Form 7.67-7.65 (m, 1H), 7.43-7.39 (m, 1H), 7.34-7.19 (m, 2H), 4.20 (q, $J = 7.2$ Hz, 2H), 3.95 (s, 2H), 2.55 (s, 3H), 1.24 (t, $J = 7.2$ Hz, 3H), Enol Form 12.48 (s, 1H), 7.43-7.39 (m, 1H), 7.34-7.19 (m, 3H), 4.27 (q, $J = 6.8$ Hz, 2H), 2.47 (s, 3H), 1.34 (t, $J = 6.8$ Hz, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ Keto Form 195.65, 167.67, 139.44 136.22, 132.31, 132.30, 129.23, 125.86, 61.41, 48.31, 21.60, 14.11, Enol Form 174.98, 172.93, 136.60, 134.57, 131.07, 130.09, 128.47, 125.80, 91.70, 60.35, 20.59, 14.34; IR (neat) 2981 cm^{-1} , 2932 cm^{-1} , 1746 cm^{-1} , 1693 cm^{-1} , 1318 cm^{-1} , MS (relative intensity) m/z 206 (M^+ , 40), 119 (100), 91 (34); HRMS (EI) m/z calcd for $\text{C}_{12}\text{H}_{14}\text{O}_3$ 206.0943, found 206.0952.

Ethyl 3-(3,5-dimethylphenyl)-3-oxopropanoate (3e)



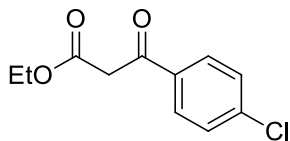
Color less oil; ^1H -NMR (CDCl_3 , 400 MHz); δ Keto Form 7.54 (s, 2H), 7.22 (s, 1H), 4.21 (q, $J = 6.8$ Hz, 2H), 3.96 (s, 2H), 2.37 (s, 6H), 1.26 (t, $J = 6.8$ Hz, 3H), Enol Form 12.54 (s, 1H), 7.39 (s, 2H), 7.09 (m, 1H), 5.64 (s, 1H), 4.26 (q, $J = 7.2$ Hz, 2H), 2.34 (s, 6H), 1.33 (t, $J = 7.2$ Hz, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ Keto Form 193.05, 167.82, 138.58, 136.26, 135.55, 126.41, 61.53, 46.18, 21.35, 14.21, Enol Form 173.39, 172.03, 138.24, 133.49, 133.09, 87.33, 60.37, 21.42, 14.44; IR (neat) 2982 cm^{-1} , 2921 cm^{-1} , 1742 cm^{-1} , 1685 cm^{-1} , 1605 cm^{-1} , 1329 cm^{-1} , 1146 cm^{-1} , 1035 cm^{-1} ; MS (relative intensity) m/z 220 (M^+ , 50), 175 (10), 133 (100), 105 (49); HRMS (EI) m/z calcd for $\text{C}_{13}\text{H}_{16}\text{O}_3$ 220.1099, found 220.1089.

Ethyl 3-(4-fluorophenyl)-3-oxopropanoate (3f)



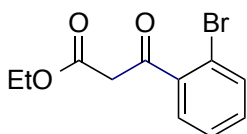
Light brown oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 8.00-7.96 (m, 2H), 7.18-7.14 (m, 2H), 4.21 (q, $J = 6.8$ Hz, 2 H), 3.97 (s, 2H), 1.26 (t, $J = 6.8$ Hz, 3H), Enol Form 12.61 (s, 1H) 7.79-7.76 (m, 2H), 7.14-7.09 (m, 2H), 5.59 (s, 1H), 4.27 (q, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ Keto Form 191.06, 167.45, 164.93, 132.59, 131.36 ($J_{\text{C-F}} = 35.2$ Hz), 116.08 ($J_{\text{C-F}} = 80.7$ Hz), 61.70, 46.08, 14.17 Enol Form 173.26, 170.47, 167.49, 131.33, 128.34 ($J_{\text{C-F}} = 31.6$ Hz), 115.77 ($J_{\text{C-F}} = 80.4$ Hz), 87.27, 60.52, 14.40; $^{19}\text{F-NMR}$ (CDCl_3 , 376 MHz) δ Keto Form -108.38, Enol Form -103.72; IR (neat) 2984 cm^{-1} , 1747 cm^{-1} , 848 cm^{-1} , MS (relative intensity) m/z 210 (M^+ , 52), 123 (100), 95 (29); HRMS (EI) m/z calcd for $\text{C}_{11}\text{H}_{11}\text{FO}_3$ 210.0692, found 210.0686.

Ethyl 3-(4-chlorophenyl)-3-oxopropanoate (3g)



Light brown oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 7.90-7.88 (m, 2H), 7.47-7.45 (m, 2H), 4.21 (q, $J = 7.2$ Hz, 2H), 3.96 (s, 2H), 1.26 (t, $J = 7.2$ Hz, 3H), Enol Form 12.58 (s, 1H), 7.72-7.70 (m, 2H), 7.41-7.38 (m, 2H), 5.64 (s, 1H), 4.27 (q, $J = 7.2$ Hz, 2H), 1.34 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ Keto Form 191.47, 167.36, 140.46, 134.47, 130.08, 129.28, 61.77, 46.10, 14.21, Enol Form 173.21, 170.29, 137.46, 132.06, 128.98, 127.50, 87.81, 60.63, 14.43; IR (neat) 2981 cm^{-1} , 1755 cm^{-1} , 1741 cm^{-1} , 703 cm^{-1} ; MS (relative intensity) m/z 228 (M^+ , 7), 226 (M^+ , 24), 139 (100), 111 (17); HRMS (EI) m/z calcd for $\text{C}_{11}\text{H}_{11}\text{ClO}_3$ 226.0397, found 226.0393.

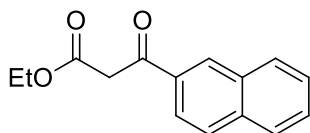
Ethyl 3-(2-bromophenyl)-3-oxopropanoate (3h)



Light brown oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 7.62-7.58 (m, 1H), 7.60-7.46 (m, 1H), 7.40-7.23 (m, 2H), 4.17 (q, $J = 7.2$ Hz, 2H), 4.01 (s, 2H), 1.23 (t, $J = 7.2$ Hz, 3H), Enol

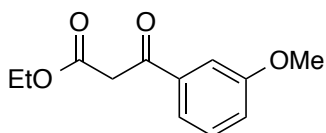
Form 12.42 (s, 1H), 7.62-7.58 (m, 1H), 7.60-7.46 (m, 1H), 7.40-7.23 (m, 2H), 5.44 (s, 1H), 4.26 (q, $J=7.2$ Hz, 2H), 1.32 (t, $J=7.2$ Hz, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ Keto Form 185.71, 166.87, 134.01, 132.45, 131.26, 130.32, 129.62, 127.61, 61.61, 48.89, 14.12, Enol Form 172.65, 172.00, 140.05, 135.85, 133.85, 127.45, 121.02, 119.29, 93.29, 60.65, 14.34; IR (neat) 2982 cm^{-1} , 2938 cm^{-1} , 1744 cm^{-1} , 1703 cm^{-1} , 1633 cm^{-1} , 1587 cm^{-1} , 1246 cm^{-1} , 1198 cm^{-1} , 1027 cm^{-1} , 761 cm^{-1} ; MS (relative intensity) m/z 272 (M^+ , 2), 270 (M^+ , 2), 227 (5), 225 (5), 191 (97), 185 (98), 183 (100), 157 (18), 155 (18); HRMS (EI) m/z calcd for $\text{C}_{11}\text{H}_{11}\text{BrO}_3$ 269.9892, found 269.9836.

Ethyl 3-(naphthalen-2-yl)-3-oxopropanoate (3i)



Light brown oil; ^1H -NMR (CDCl_3 , 400 MHz) δ Keto Form 8.46 (s, 1H), 8.03-7.97 (m, 2H), 7.93-7.84 (m, 2H), 7.66-7.54 (m, 2H), 4.26-4.21 (m, 2H), 4.13 (s, 2H), 1.27 (t, 6.4 Hz, 3H), Enol Form 12.68 (s, 1H), 8.03-7.97 (m, 2H), 7.93-7.84 (m, 2H), 7.66-7.54 (m, 2H), 5.82 (s, 1H), 4.33-4.27 (m, 2H), 1.36 (t, 7.2 Hz, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ Keto Form 192.56, 167.72, 135.91, 133.46, 132.50, 129.78, 128.98, 128.81, 127.91, 127.07, 126.79, 123.90, 61.63, 46.18, 14.43, Enol Form 173.34, 171.32, 134.76, 132.91, 130.70, 129.14, 128.35, 127.77, 127.65, 126.75, 126.77, 122.64, 87.95, 60.49, 14.43; IR (neat) 2980 cm^{-1} , 1747 cm^{-1} , 1731 cm^{-1} , 1031 cm^{-1} ; MS (relative intensity) m/z 242 (M^+ , 45), 155 (100), 127 (36); HRMS (EI) m/z calcd for $\text{C}_{15}\text{H}_{14}\text{O}_3$ 242.0943, found 242.0945.

Ethyl 3-(3-methoxyphenyl)-3-oxopropanoate (3j)

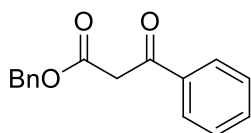


This compound was known. See: Baburajan, P.; Elango, K. P. *Tetrahedron Lett.* **2014**, *55*, 3525.

Colorless oil; ^1H -NMR (CDCl_3 , 400 MHz) δ Keto Form 7.50-7.47 (m, 2H), 7.39-7.30 (m, 1H), 7.14-7.12 (m, 1H), 4.21 (q, $J=7.2$ Hz, 2H), 3.97 (s, 2H), 3.84 (s, 3H), 1.26 (t, $J=7.2$ Hz, 3H), Enol Form 12.57 (s, 1H), 7.39-7.30 (m, 3H), 7.01-6.98 (m, 1H), 5.65 (s, 1H), 4.26 (q, $J=7.2$ Hz, 2H), 3.83 (s, 3H), 1.33 (t, $J=7.2$ Hz, 3H); ^{13}C -NMR (CDCl_3 , 100 MHz) δ

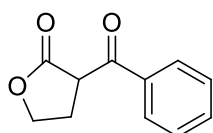
Keto Form 192.49, 167.61, 160.03, 137.44, 129.86, 121.30, 120.47, 112.59, 61.60, 55.57, 46.22, 14.41, Enol Form 173.28, 171.36, 159.81, 134.97, 129.66, 118.55, 117.37, 111.21, 87.74, 60.47, 55.46, 14.19.

Benzyl 3-oxo-3-phenylpropanoate (3k)



Light brown oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ Keto Form 7.94-7.92 (m, 2H), 7.62-7.58 (m, 1H), 7.49-7.31 (m, 7H) 5.20 (s, 2H), 4.05 (s, 2H), Enol Form 12.49 (s, 1H), 7.79-7.76 (m, 2H), 7.49-7.31 (m, 8H), 5.74 (s, 1H), 5.26 (s, 2H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ Keto Form 192.41, 167.45, 135.98, 135.41, 133.85, 131.44, 128.86, 128.63, 128.56, 128.42, 128.32, 126.16, 67.23, 46.00, Enol Form 172.98, 171.88, 135.86, 133.85, 133.35, 129.90, 128.86, 128.70, 128.63, 126.56, 128.41, 125.32, 87.27, 66.14; IR (neat) 3064 cm^{-1} , 2951 cm^{-1} , 1747 cm^{-1} , 1454 cm^{-1} ; MS (relative intensity) m/z 254 (M^+ , 18), 120 (96), 105 (65), 91 (100), 77 (38); HRMS (EI) m/z calcd for $\text{C}_{16}\text{H}_{14}\text{O}_3$ 254.0943, found 254.0944.

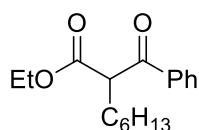
3-Benzoyldihydrofuran-2(3H)-one (3l)



This compound was known. See: Jiang, X.; Fu, D.; Zhang, G.; Cao, Y.; Liu, L.; Song, J.; Wang, R. *Chem. Commun.* **2010**, *46*, 4294.

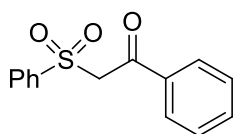
Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 8.11-8.09 (m, 2H), 7.64-7.50 (m, 3H), 4.63-4.52 (m, 2H), 4.48-4.42 (m, 1H), 2.92-2.85 (m, 1H), 2.57-2.48 (m, 1H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 193.11, 172.99 135.76, 132.82, 129.65, 128.92, 128.10, 68.00, 48.15, 26.13.

Ethyl 2-benzoylheptanoate (3m)



Colorless oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 8.00-7.98 (m, 2H), 7.60-7.56 (m, 1H), 7.50-7.46 (m, 2H), 4.30-4.26 (m, 1H), 4.14 (q, $J = 6.8$ Hz, 2H), 2.03-1.98 (m, 2H), 1.34-1.26 (m, 8H), 1.17 (t $J = 6.8$ Hz, 3H), 0.86 (t, $J = 7.2$ Hz, 3H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 195.48, 170.26, 136.47, 133.55, 128.85, 128.69, 61.44, 54.52, 31.66, 29.22, 29.13, 27.73, 22.67, 14.16; IR (neat) 2928 cm^{-1} , 1736 cm^{-1} , 1639 cm^{-1} , 1160 cm^{-1} ; MS (relative intensity) m/z 231 (M-OEt, 10), 192 (100), 105 (100), 77 (100); HRMS (EI) m/z calcd for $\text{C}_{17}\text{H}_{24}\text{O}_3$ 276.1725, found 276.1723.

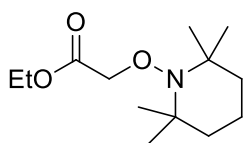
1-Phenyl-2-(phenylsulfonyl)ethanone (3n)



This compound was known. See: Xuan, J.; Feng, Z.-J.; Chen, J.-R.; Lu, L.-Q.; Xiao, W.-J. *Chem. – Eur. J.* **2014**, *20*, 3045.

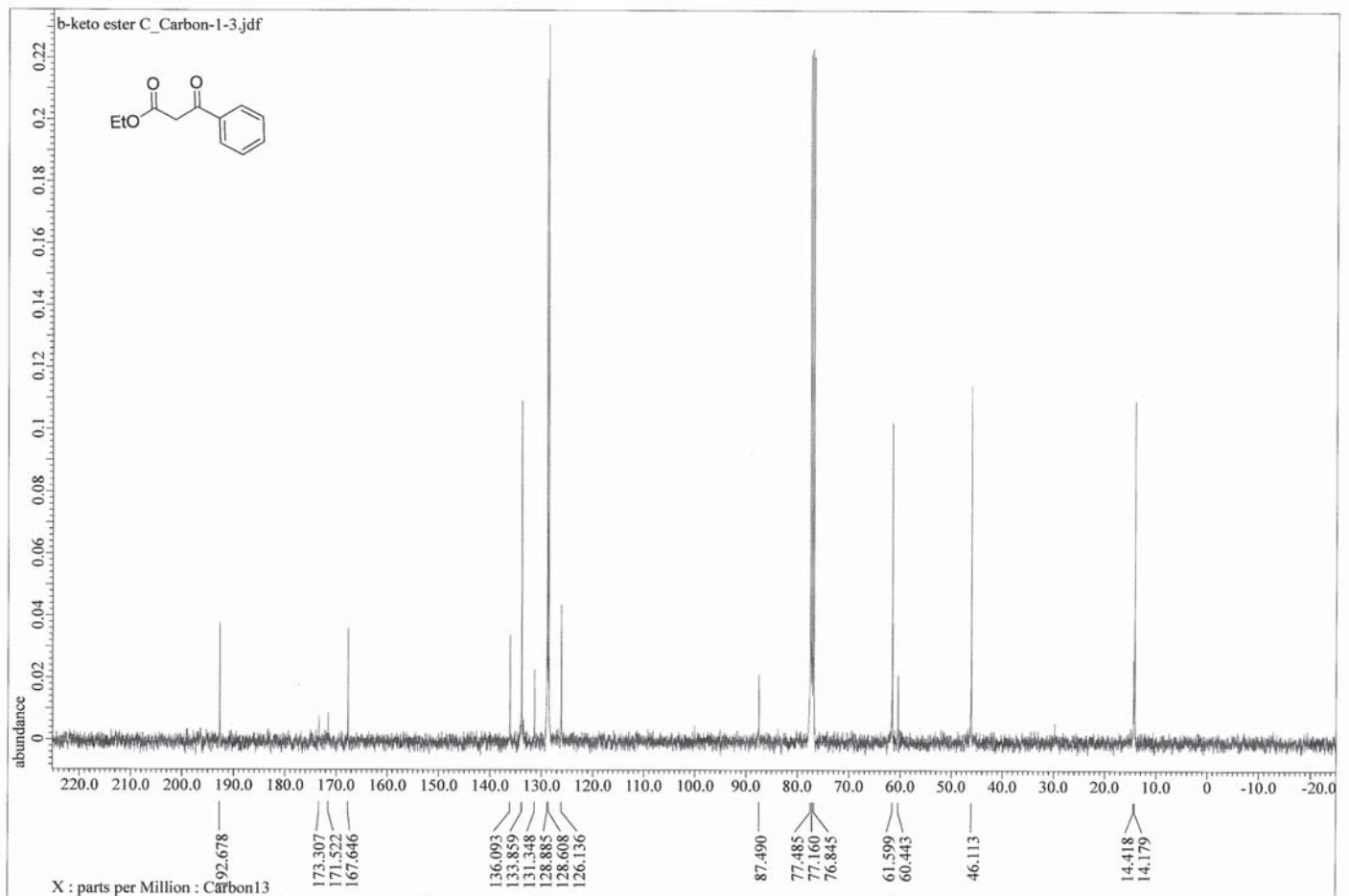
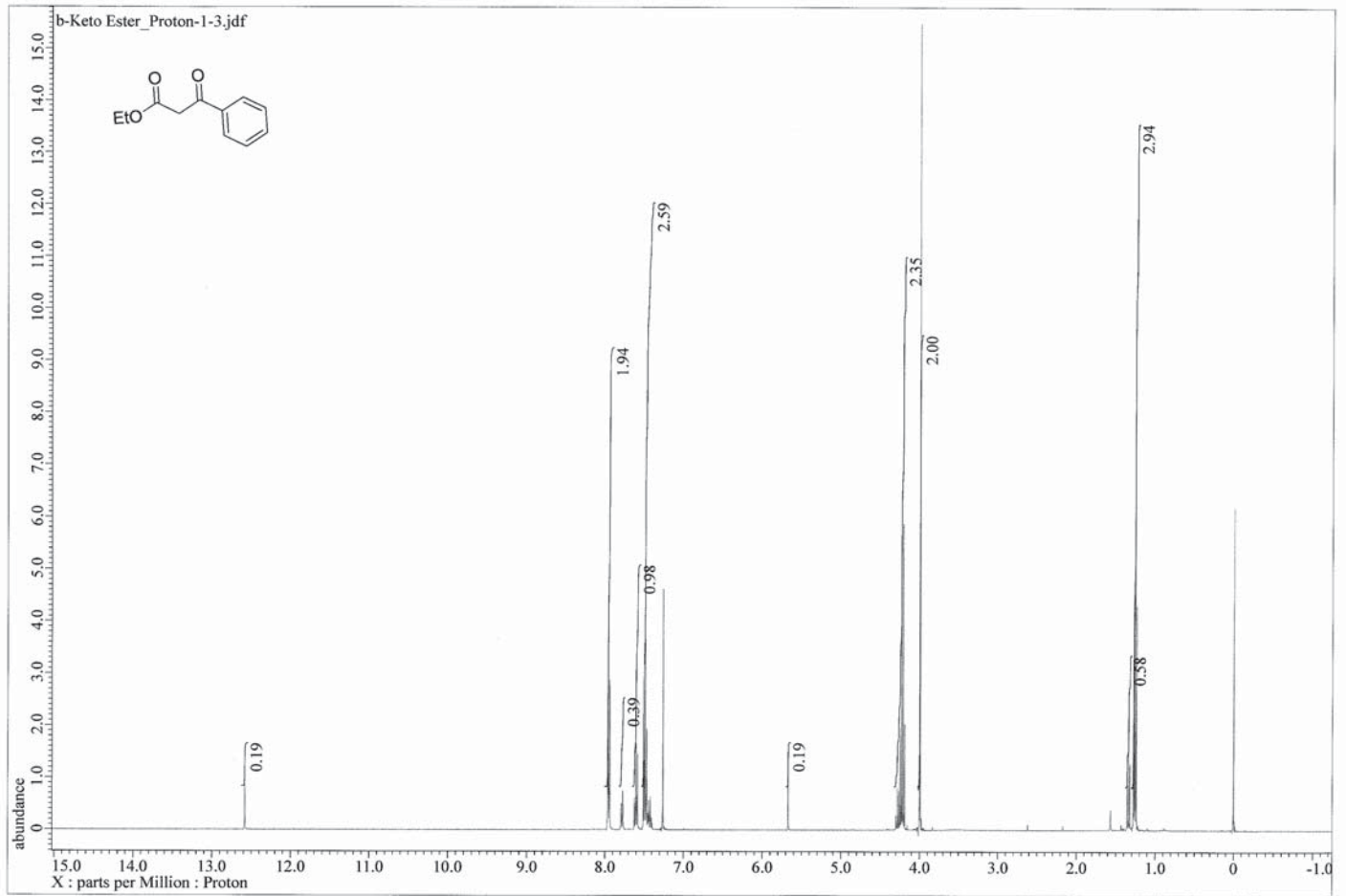
White solid (m.p. = 89-92 °C, Lit; m.p. = 90-92 °C); $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 7.96-7.89 (m 4H), 7.69-7.61 (m, 2H), 7.56 (t, $J = 7.6$ Hz, 2H), 7.49 (t, $J = 7.6$ Hz, 2H), 4.74 (s, 2H); $^{13}\text{C-NMR}$ (CDCl_3 , 400 MHz) δ 188.11, 138.80, 135.80, 134.51, 134.38, 129.39, 129.33, 128.98, 128.69, 63.52.

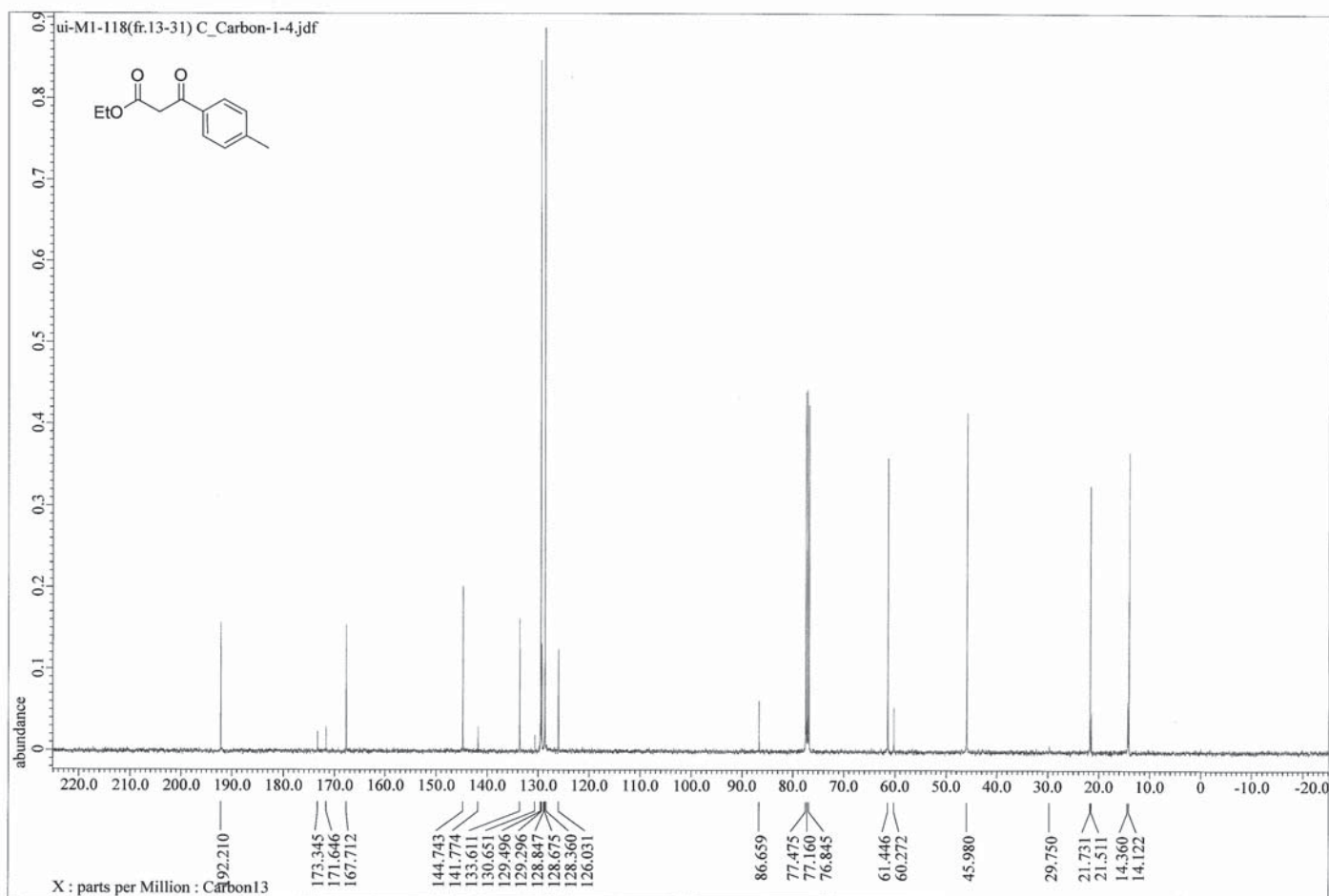
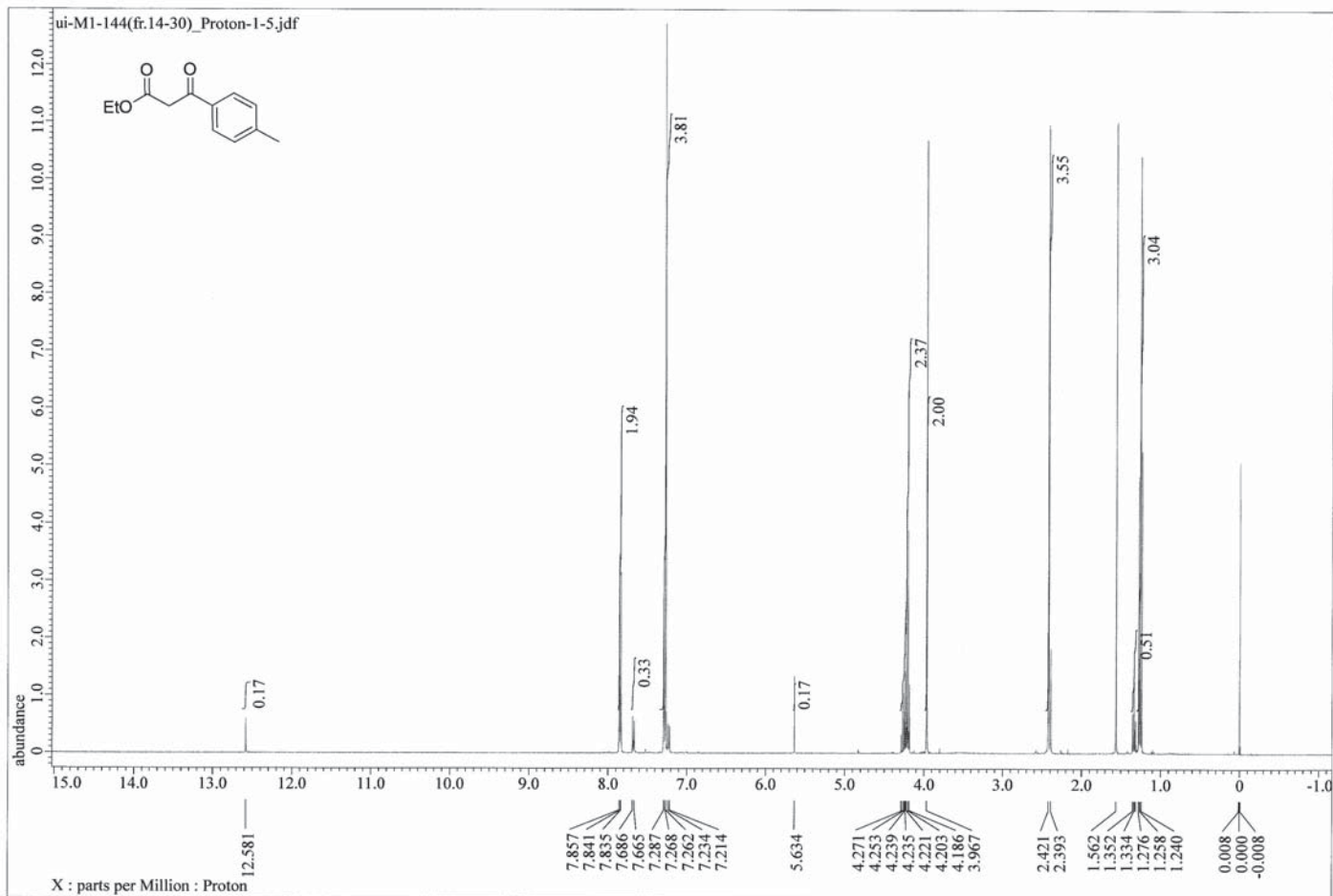
Ethyl 2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)acetate (4)

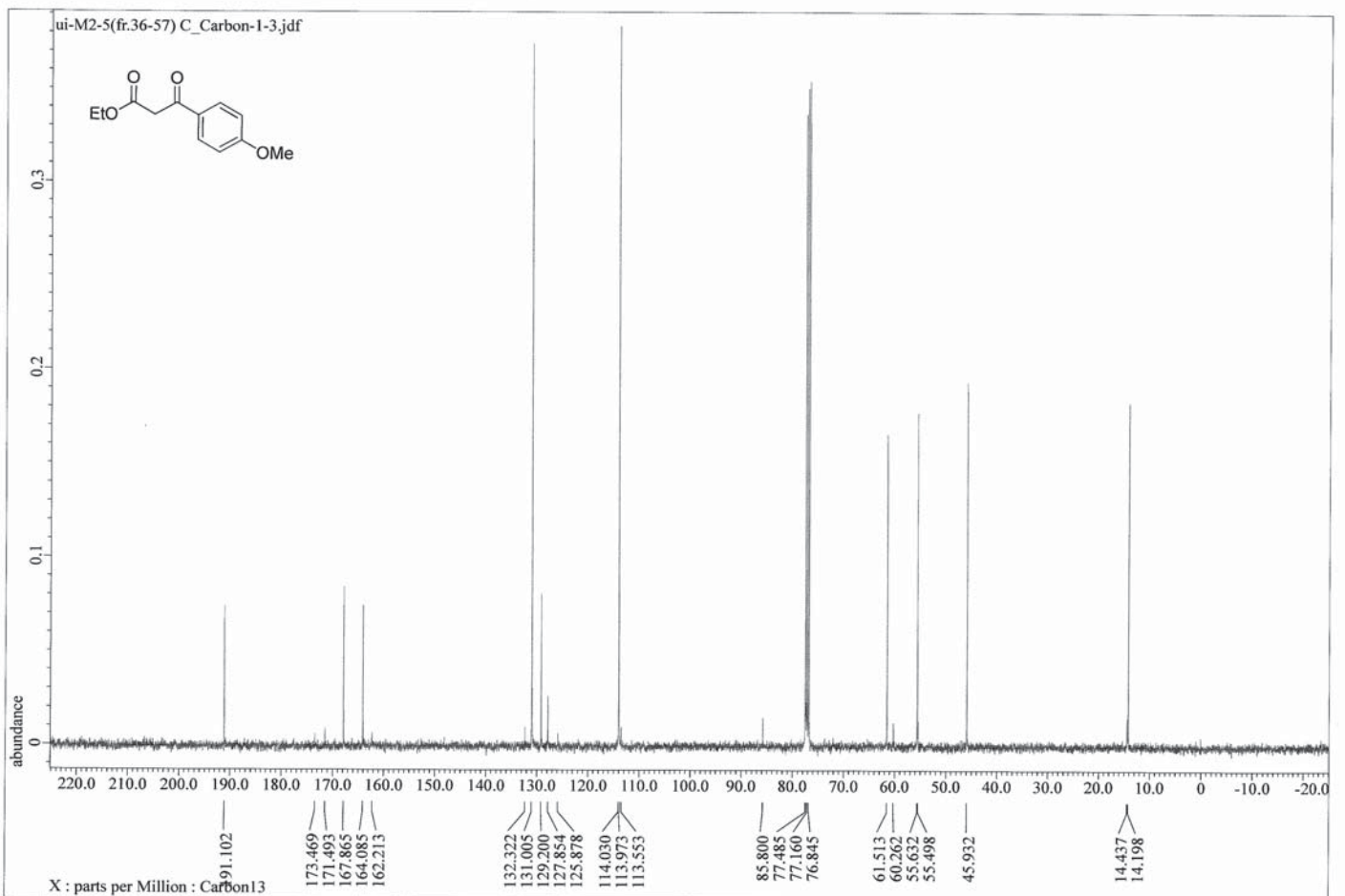
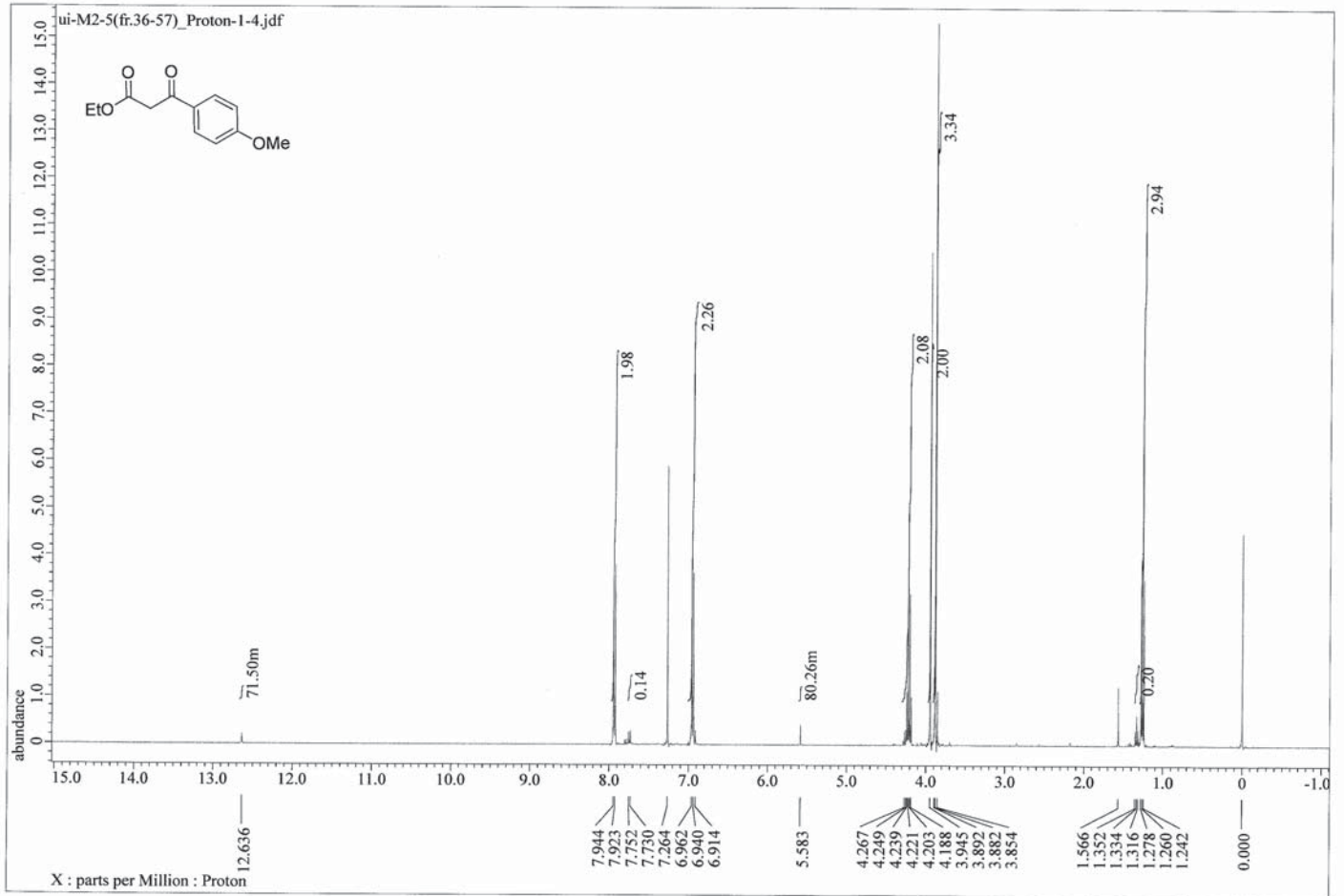


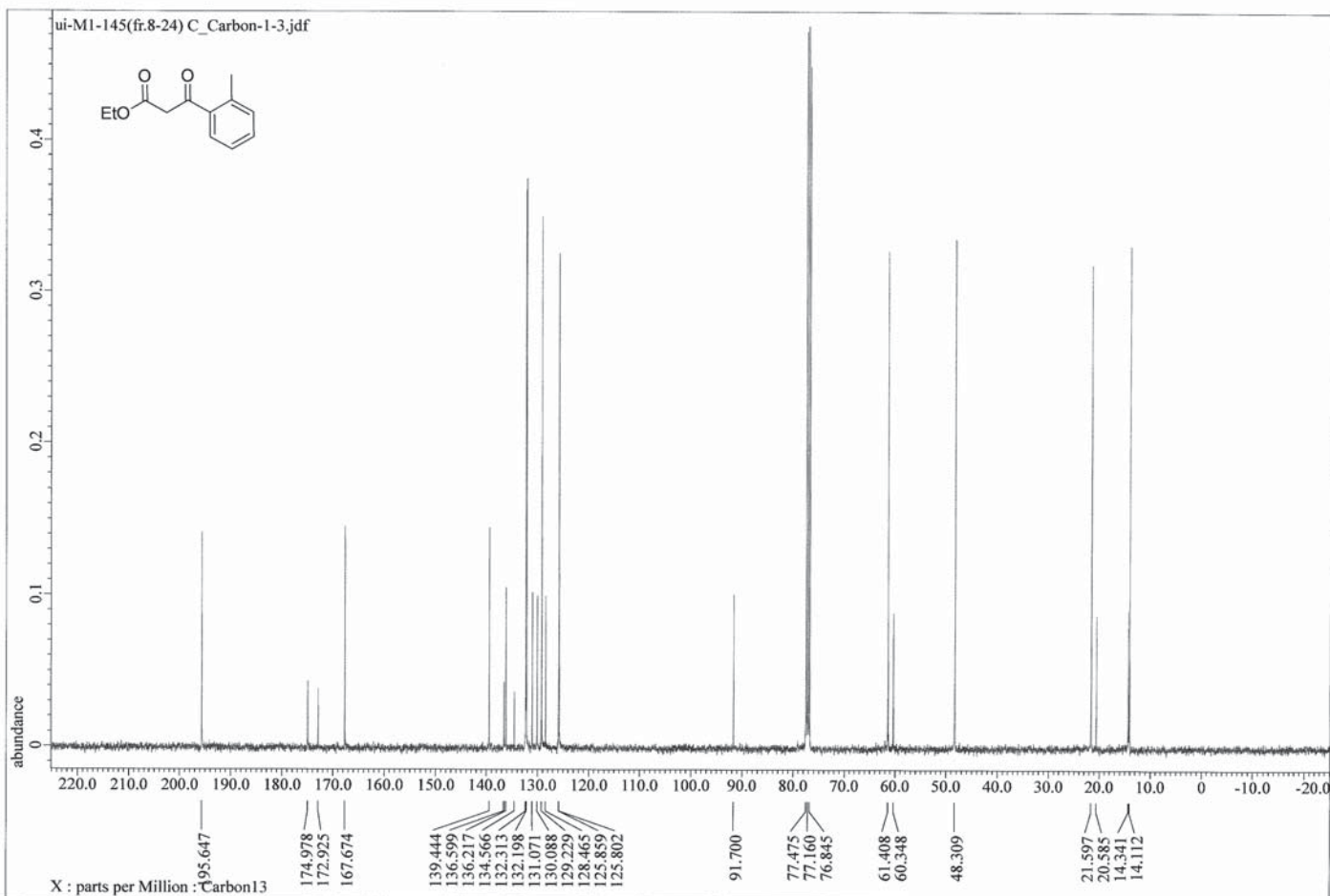
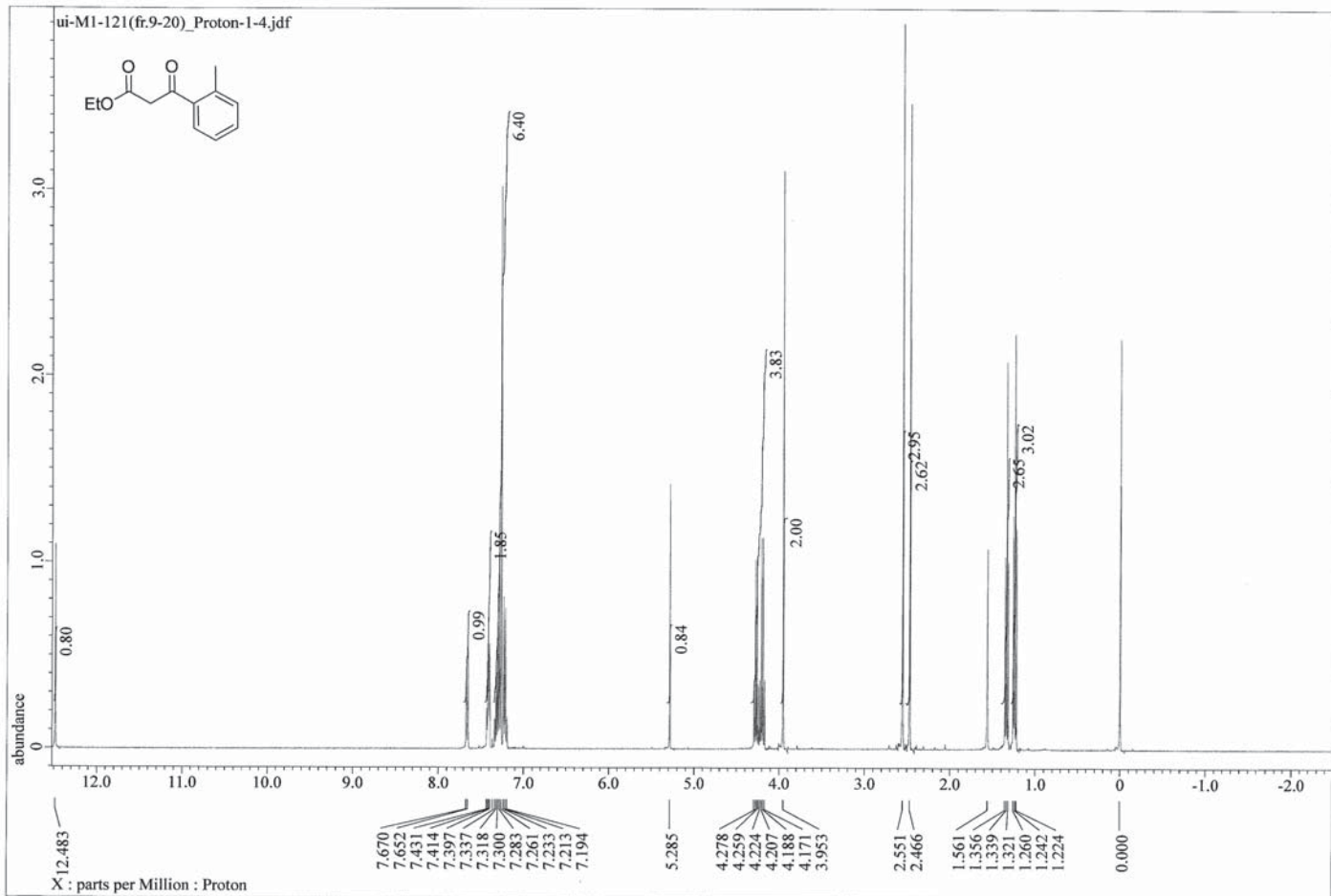
This compound was known, see; Sumino, S.; Fusano, A.; Ryu, I. *Org. Lett.* **2013**, *15*, 2826.

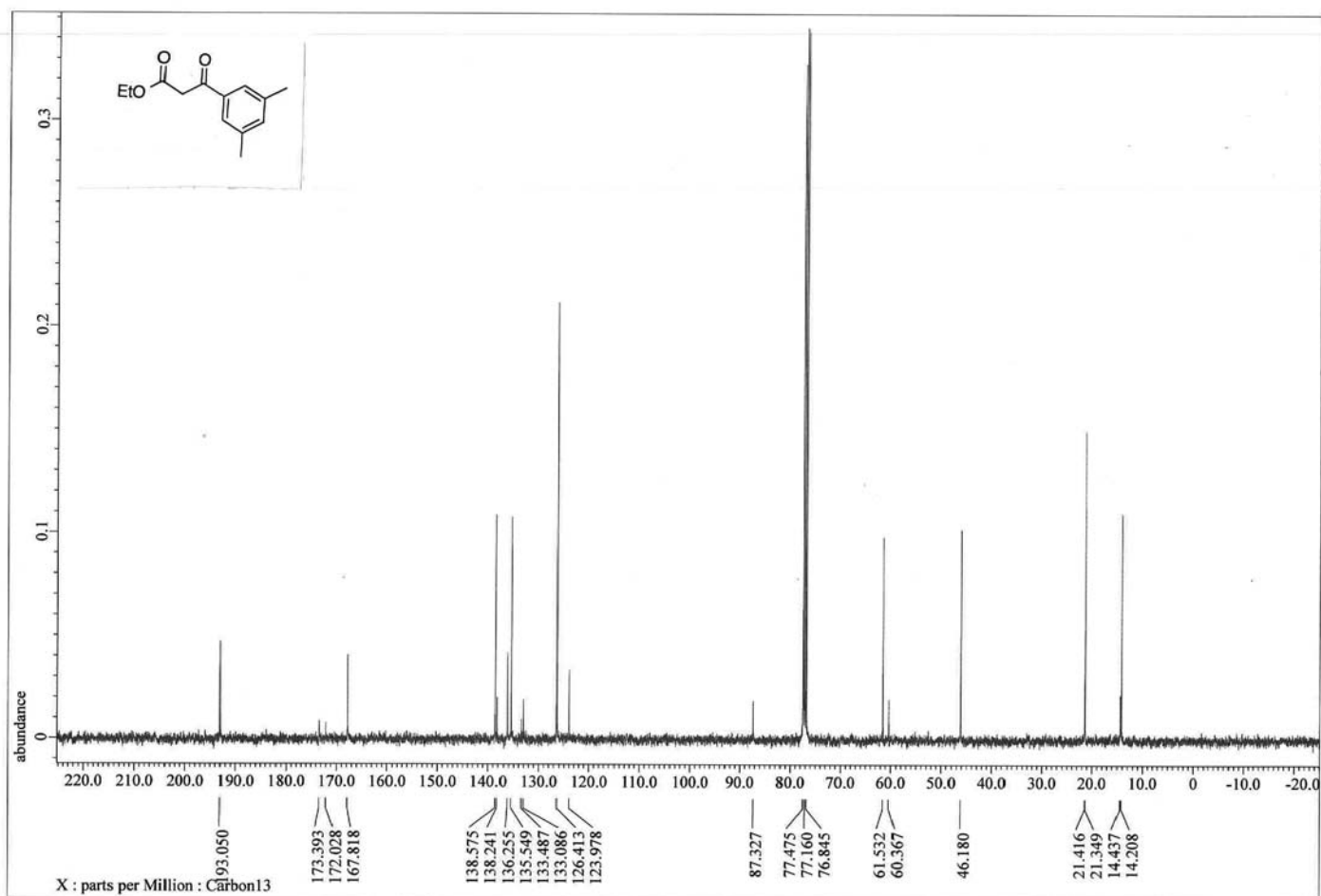
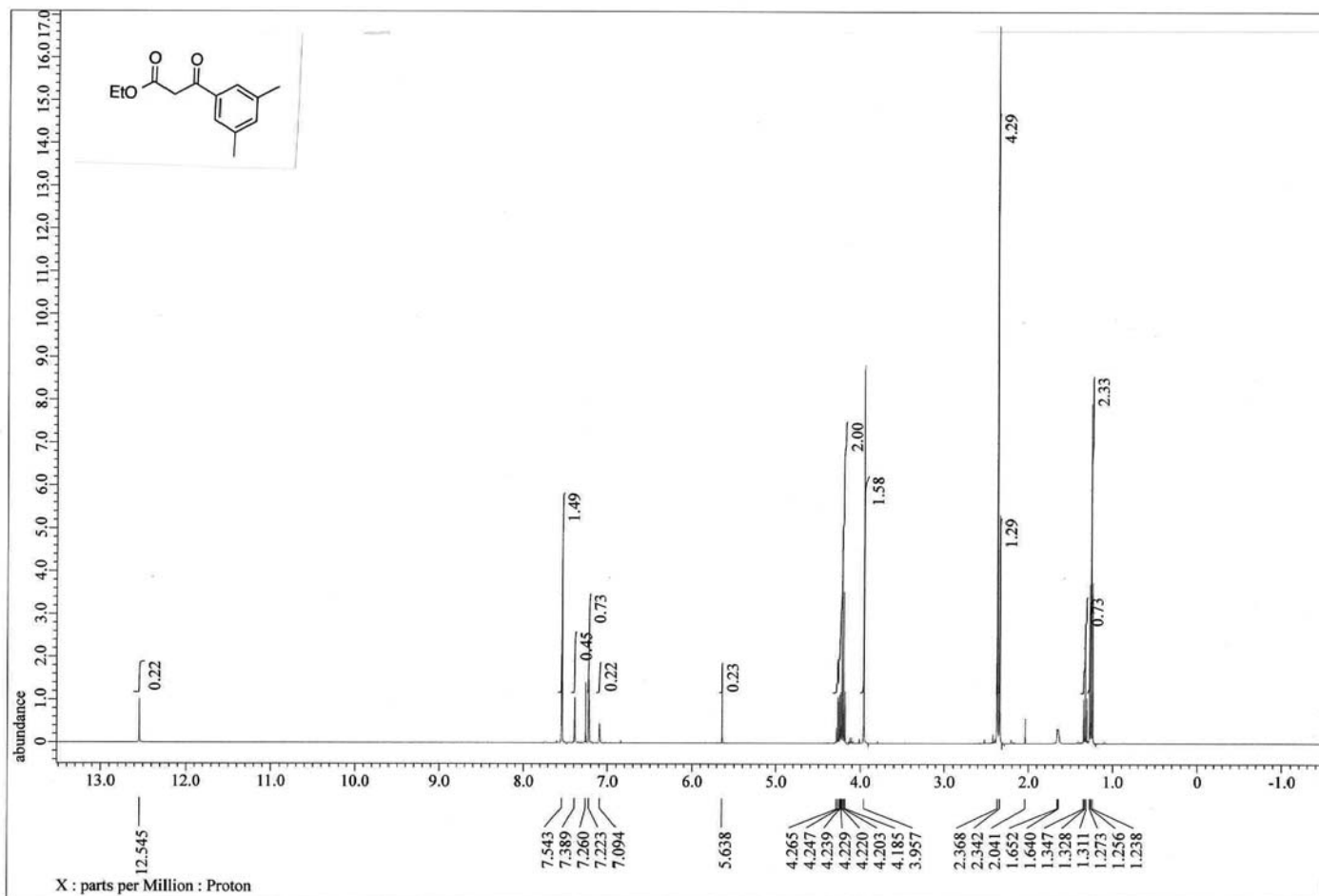
Yellow oil; $^1\text{H-NMR}$ (CDCl_3 , 400 MHz) δ 4.43 (s, 2H), 4.20 (q, $J = 6.8$ Hz, 2H), 1.45-1.42 (m, 4H), 1.31-1.25 (m, 2H), 1.28 (t, $J = 7.3$ Hz, 3H), 1.15 (s, 12 H); $^{13}\text{C-NMR}$ (CDCl_3 , 100 MHz) δ 169.96, 75.70, 60.59, 60.17, 39.82, 32.83, 20.19, 17.16, 14.32.

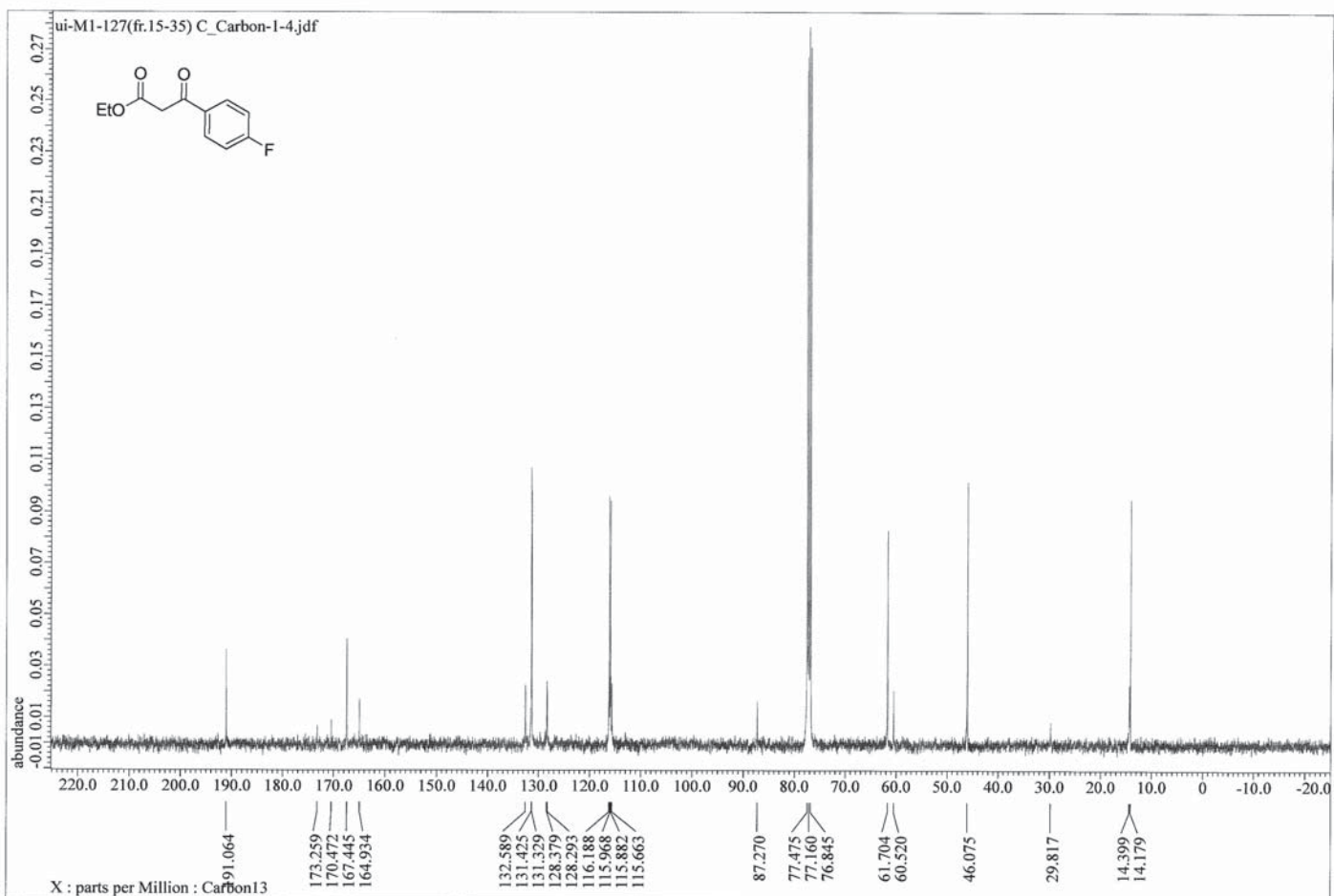
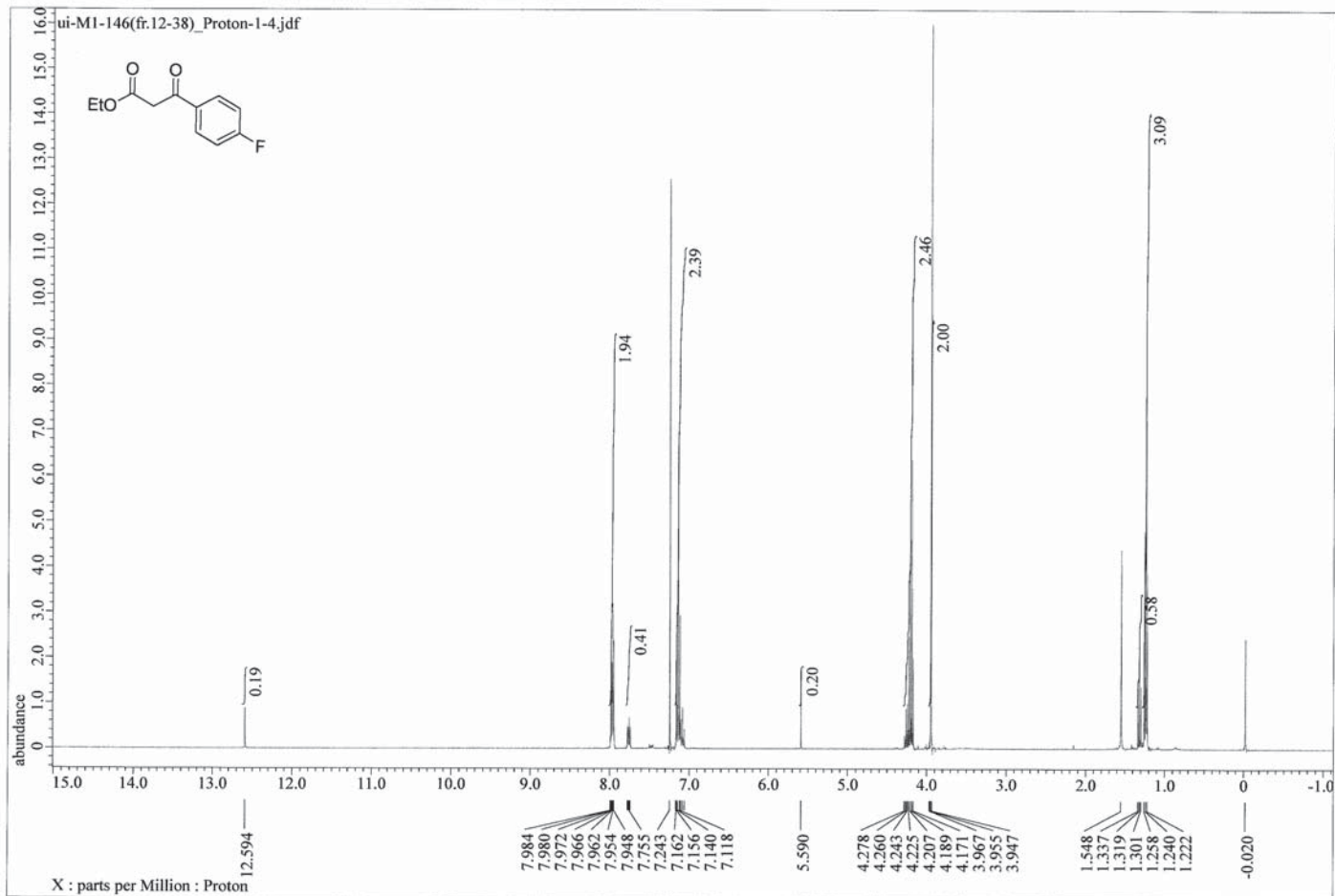




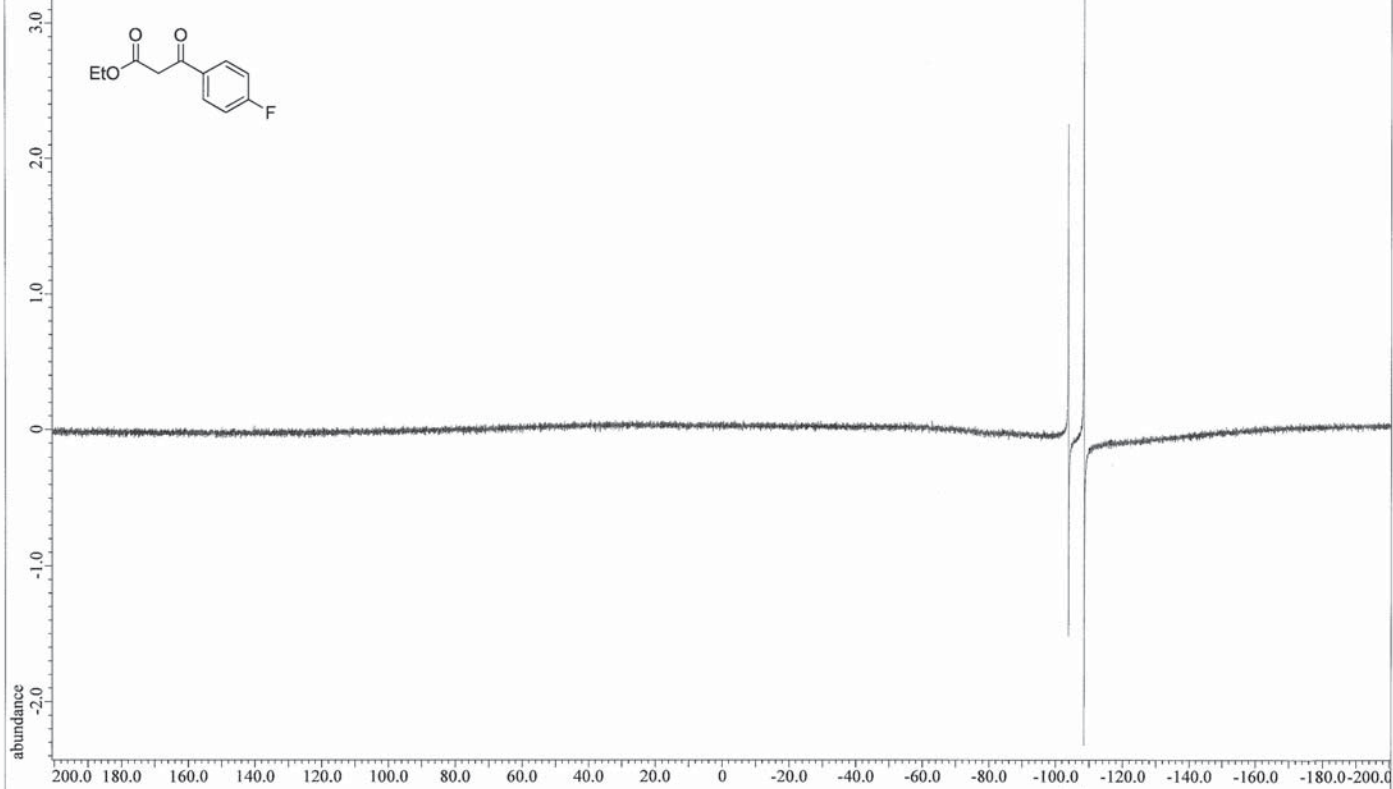
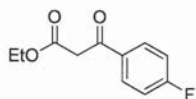








ui-M1-146 F_Proton-1-3.jdf



X : parts per Million : Fluorine19

-103.724
-103.785
-108.377
-108.438

