

Electronic Supplementary Information

Gallium-Catalyzed Reductive Lactonization of Keto Acids using a Hydrosilane

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General

All reactions were carried out under a N₂ atmosphere, unless otherwise noted. Benzene and toluene were distilled from a Na/benzophenone ketyl. All gallium compounds and other metal compounds were commercially available and were used without further purification. Hydrosilanes were used without further purification. 3-Benzoylpropionic acid (**1a**) is commercial available and purified by recrystallization prior to use. Reactions were monitored by TLC analysis of reaction aliquots. Column chromatography was performed using a silica gel. ¹H NMR spectra were measured at 500 (or 300) MHz using tetramethylsilane as an internal standard (0.00 ppm). ¹³C NMR spectra were measured at 125 (or 75) MHz using the center peak of chloroform (77.0 ppm). High-resolution mass spectra (HRMS) were measured using NBA (3-nitrobenzylalcohol) as a matrix.

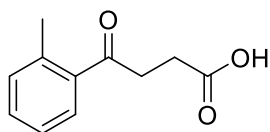
Synthesis of keto acid derivatives

Keto acid derivatives were synthesized by either Method A or Method B described in the corresponding literatures, except for **1a**.

Method A: To a solution of succinic anhydride (10 mmol) and AlCl₃ (22 mmol, 2.9 g) in CH₂Cl₂ (20 mL) was added aromatic hydrocarbon derivatives (11 mmol). The reaction mixture was stirred at room temperature over 6 h. After the reaction, the mixture was quenched with an aqueous solution of 1N HCl (10 mL). The aqueous layer was extracted with CH₂Cl₂ (15 mL x 3) and the organic phase was combined, dried with anhydrous Na₂SO₄ and evaporated under reduced pressure. The crude product was purified by recrystallization (hexane/CH₂Cl₂ or toluene) to give the corresponding keto acid.

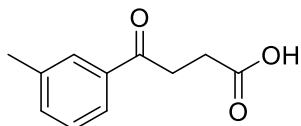
Method B: To a solution of Mg (13.0 mmol, 316 mg) in THF (5 mL) was added dropwise a solution of aryl bromide derivatives (12 mmol) and THF (15 mL) for 24 h. The mixture was then added to a solution of succinic anhydride (10 mmol) in THF (10 mL) at -90 °C. The mixture was stirred at room temperature over 6 h. After the reaction, the mixture was quenched with an aqueous solution of 1N HCl (10 mL). The aqueous layer was extracted with Et₂O (15 mL x 3) and the organic phase was combined, dried with anhydrous Na₂SO₄ and evaporated under reduced pressure. The crude product was purified by recrystallization (hexane/CH₂Cl₂ or toluene) to give the corresponding keto acids.

3-(2-Methylbenzoyl)propionic acid¹ (**1b**)



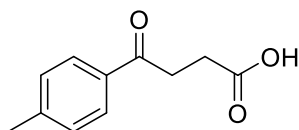
The preparation is reported in our previous report, and the spectra data are in agreement with the literature¹.

3-(3-Methylbenzoyl)propionic acid¹ (1c)



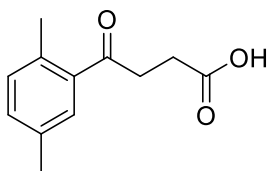
Method B; a white solid: ¹H NMR (CDCl₃, 500 MHz) δ 2.41 (s, 3 H, CH₃), 2.81 (t, J = 6.5 Hz, 2 H, CH₂), 3.30 (t, J = 6.5 Hz, 2 H, CH₂), 7.34-7.40 (m, 2 H, ArH), 7.77-7.79 (m, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 21.3, 28.0, 33.2, 125.2, 128.5, 128.6, 134.1, 136.4, 138.4, 178.8, 198.1; MS (EI) m/z (%) 192 (M⁺, 22), 119 (100).

3-(4-Methylbenzoyl)propionic acid¹ (1d)



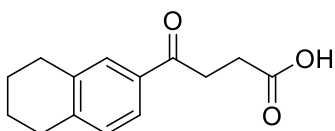
Method A; a white solid: ¹H NMR (CDCl₃, 300 MHz) δ 2.41 (s, 3 H, CH₃), 2.80 (t, J = 6.5 Hz, 2 H, CH₂), 3.29 (t, J = 6.5 Hz, 2 H, CH₂), 7.26 (d, J = 7.4 Hz, 2 H, ArH), 7.87 (d, J = 7.4 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 75 MHz) δ 21.6, 28.0, 33.0, 128.1, 129.3, 133.9, 144.2, 178.6, 197.5; MS (EI) m/z (%) 192 (M⁺, 17), 119 (100).

3-(2, 5-Dimethylbenzoyl)propionic acid¹ (1e)



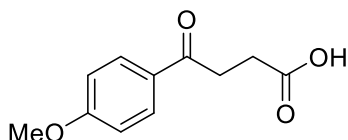
Method A; a white solid: ¹H NMR (CDCl₃, 500 MHz) δ 2.37 (s, 3 H, CH₃), 2.45 (s, 3 H, CH₃), 2.79 (t, J = 6.5 Hz, 2 H, CH₂), 3.22 (t, J = 6.5 Hz, 2 H, CH₂), 7.13 (d, J = 7.5 Hz, 1 H, ArH), 7.20 (d, J = 7.5 Hz, 1 H, ArH), 7.50 (s, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 20.9, 20.9, 28.2, 35.8, 129.2, 131.9, 132.3, 135.2, 137.1, 178.5, 201.7; MS (EI) m/z (%) 206 (M⁺, 50), 133 (100).

3-(5,6,7,8-Tetrahydro-2-naphthoyl)propionic acid¹ (1f)



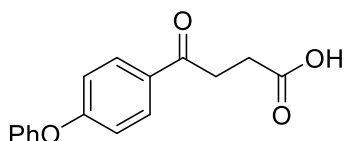
The preparation is reported in our previous report, and the spectra data are in agreement with the literature.¹

3-(4-Methoxybenzoyl)propionic acid¹ (1g)¹



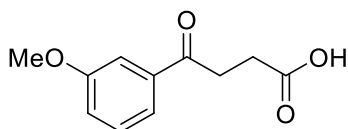
Method A; a white solid: ¹H NMR (CDCl₃, 500 MHz) δ 2.80 (t, J = 6.5 Hz, 2 H, CH₂), 3.28 (t, J = 6.5 Hz, 2 H, CH₂), 3.88 (s, 3 H, CH₃), 6.94 (d, J = 9.0 Hz, 2 H, ArH), 7.97 (d, J = 9.0 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 28.1, 32.8, 55.5, 113.8, 129.5, 130.3, 163.7, 178.2, 196.4; MS (EI) m/z (%) 208 (M⁺, 9), 135 (100).

3-(4-Phenoxybenzoyl)propionic acid¹ (1h)



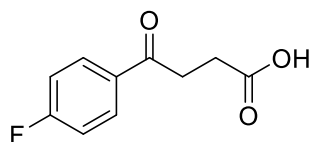
The preparation is reported in our previous report, and spectra data are in agreement with the literature.¹

3-(3-Methoxybenzoyl)propionic acid² (1i)



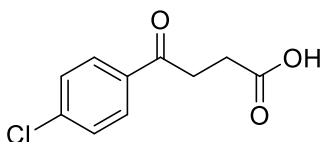
Method B; a white solid: mp 108–109 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.81 (t, J = 6.5 Hz, 2 H, CH₂), 3.30 (t, J = 6.5 Hz, 2 H, CH₂), 3.85 (s, 3 H, CH₃), 7.12 (d, J = 8.0 Hz, 1 H, ArH), 7.38 (dd, J = 8.0 Hz, 7.5 Hz, 1 H, ArH), 7.50 (s, 1 H, ArH), 7.56 (d, J = 7.5 Hz, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 28.0, 33.2, 55.4, 112.2, 119.9, 120.7, 129.6, 137.7, 159.8, 178.8, 197.6; IR (ATR, cm⁻¹) 1686 s; MS (EI) m/z (%) 208 (M⁺, 21), 135 (100); HRMS (FAB-Magnetic Sector): calcd. for C₁₁H₁₃O₄ [M+H]⁺: 209.080, found: 209.0818.

3-(4-Fluorobenzoyl)propionic acid¹ (1j)



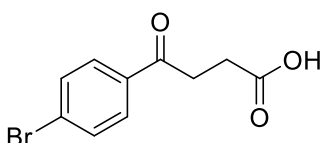
Method A; a white solid: ¹H NMR (CDCl₃, 300 MHz) δ 2.82 (t, J = 6.5 Hz, 2 H, CH₂), 3.29 (t, J = 6.5 Hz, 2 H, CH₂), 7.14 (dd, J = 8.3 Hz, 8.3 Hz, 2 H, ArH), 8.01 (dd, J = 8.3 Hz, 5.7 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 75 MHz) δ 28.0, 33.0, 115.8 (J_{C-F} = 22.6 Hz), 130.7 (J_{C-F} = 8.8 Hz), 132.8 (J_{C-F} = 2.5 Hz), 165.9 (J_{C-F} = 255.3 Hz), 178.7, 196.2; MS (EI) m/z (%) 196 (M⁺, 9), 123 (100).

3-(4-Chlorobenzoyl)propionic acid¹ (1k)



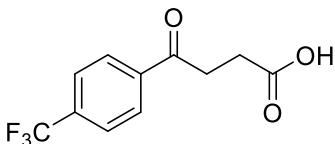
Method A; a white solid: ¹H NMR (CDCl₃, 500 MHz) δ 2.81 (t, *J* = 6.5 Hz, 2 H, CH₂), 3.28 (t, *J* = 6.5 Hz, 2 H, CH₂), 7.45 (d, *J* = 8.0 Hz, 2 H, ArH), 7.92 (d, *J* = 8.0 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 27.9, 33.1, 129.0, 129.5, 134.7, 139.8, 178.5, 196.6; MS (EI) *m/z* (%) 214 (M⁺+2, 2), 212 (M⁺, 7), 139 (100).

3-(4-Bromobenzoyl)propionic acid¹ (1l)



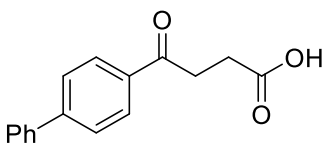
Method A; a white solid: ¹H NMR (CDCl₃, 300 MHz) δ 2.81 (t, *J* = 6.5 Hz, 2 H, CH₂), 3.27 (t, *J* = 6.5 Hz, 2 H, CH₂), 7.62 (d, *J* = 8.3 Hz, 2 H, ArH), 7.84 (d, *J* = 8.3 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 75 MHz) δ 27.9, 33.1, 128.5, 129.5, 132.0, 135.1, 178.5, 196.8; MS (EI) *m/z* (%) 258 (M⁺+2, 8), 256 (M⁺, 8), 183 (100).

3-(4-Trifluoromethylbenzoyl)propionic acid³ (1m)



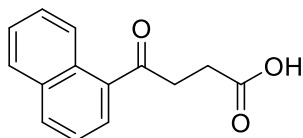
Method B; a white solid: mp 129–130 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.85 (t, *J* = 6.5 Hz, 2 H, CH₂), 3.33 (t, *J* = 6.5 Hz, 2 H, CH₂), 7.75 (d, *J* = 8.0 Hz, 2 H, ArH), 8.09 (d, *J* = 8.0 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 27.8, 33.4, 123.5 (*J*_{C-F} = 272.9 Hz), 125.7 (*J*_{C-F} = 3.8 Hz), 128.4, 134.6 (*J*_{C-F} = 32.7 Hz), 139.0 (*J*_{C-F} = 1.3 Hz), 178.5, 196.9; IR (ATR, cm⁻¹) 1715 m, 1688 s; MS (FAB) *m/z* (%) 247 (M+H⁺); HRMS (FAB-Magnetic Sector): calcd. for C₁₁H₁₀F₃O₃ [M+H]⁺: 247.0577, found: 247.0592.

4-([1,1'-Biphenyl]-4-yl)-4-oxobutanoic acid¹ (1n)



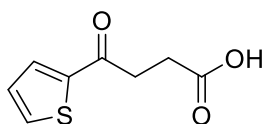
The preparation is reported in our previous report, and spectra data are in agreement with the literature.¹

4-(1-Naphthyl)-4-oxobutanoic acid¹ (1o)



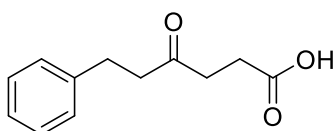
Method B; a white solid: ¹H NMR (CDCl₃, 300 MHz) δ 2.90 (t, *J* = 6.5 Hz, 2 H, CH₂), 3.40 (t, *J* = 6.5 Hz, 2 H, CH₂), 7.50-7.60 (m, 3 H, ArH), 7.88 (d, *J* = 8.5 Hz, 1 H, ArH), 7.95 (d, *J* = 8.5 Hz, 1 H, ArH), 8.00 (d, *J* = 8.5 Hz, 1 H, ArH), 8.62 (d, *J* = 8.5 Hz, 1 H, ArH); ¹³C NMR (CDCl₃, 75 MHz) δ 28.4, 36.3, 124.3, 125.8, 126.5, 127.7, 128.0, 128.4, 130.1, 132.9, 133.9, 135.2, 178.9, 201.8; MS (EI) *m/z* (%) 228 (M⁺, 74), 155 (100).

4-(2-Thienyl)-4-oxobutanoic acid⁴ (1p)



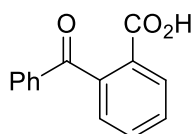
Method A; a green solid: ¹H NMR (CDCl₃, 500 MHz) δ 2.81 (t, *J* = 6.5 Hz, 2 H, CH₂), 3.26 (t, *J* = 6.5 Hz, 2 H, CH₂), 7.14 (t, *J* = 4.5 Hz, 1 H, ArH), 7.65 (d, *J* = 4.5 Hz, 1 H, ArH), 7.77 (d, *J* = 4.5 Hz, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 28.0, 33.6, 128.1, 132.1, 133.8, 143.4, 178.5, 190.7; MS (EI) *m/z* (%) 184 (M⁺, 14), 110 (100); HRMS (EI-Quadrupole): calcd. for C₈H₈O₃S [M]⁺: 184.0194, found: 184.0177.

γ-Oxo-benzenehexanoic acid⁵ (3)



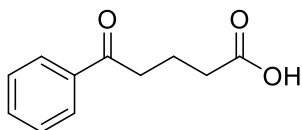
Method B; a white solid: mp 90–91 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.63 (t, *J* = 6.5 Hz, 2 H, CH₂), 2.70 (t, *J* = 6.5 Hz, 2 H, CH₂), 2.79 (t, *J* = 7.5 Hz, 2 H, CH₂), 2.92 (t, *J* = 7.5 Hz, 2 H, CH₂), 7.17-7.21 (m, 3 H, ArH), 7.26-7.29 (m, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 27.7, 29.6, 36.9, 44.1, 126.1, 128.3, 128.5, 140.8, 178.5, 207.8; IR (ATR, cm⁻¹) 1703 w; MS (EI) *m/z* (%) 206 (M⁺, 30), 91 (100); HRMS (EI-Quadrupole): calcd. for C₁₂H₁₄O₃ [M]⁺: 206.0943, found: 206.0948.

2-Benzoylbenzoic acid¹ (5)



The preparation is reported in our previous report, and the spectra data are in agreement with the literature.¹

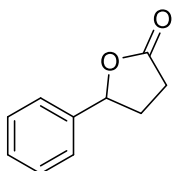
5-Oxo-5-phenylpentanoic acid⁶ (7)



Method B; a white solid: ¹H NMR (CDCl₃, 500 MHz) δ 2.09 (quint, $J = 7.0$ Hz, 2 H, CH₂), 2.51 (t, $J = 7.0$ Hz, 2 H, CH₂), 3.08 (t, $J = 7.0$ Hz, 2 H, CH₂), 7.46 (t, $J = 7.0$ Hz, 2 H, ArH), 7.56 (d, $J = 7.0$ Hz, 1 H, ArH), 7.96 (d, $J = 7.0$ Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 18.9, 33.0, 37.3, 128.0, 128.6, 133.1, 136.7, 179.4, 199.3; MS (EI) m/z (%) 192 (M⁺, 7), 105 (100).

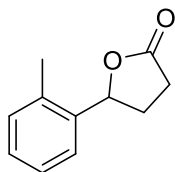
General procedure for the synthesis of the γ -lactone derivatives: To a screw-capped tube, GaCl₃ (0.010 mmol, 8.8 mg), benzene (2 mL), a keto acid (1.0 mmol), and PhSiH₃ (1.00 mmol, 108 mg) were successively added. After the tube was sealed with a cap that contained a PTFE septum, the mixture was heated at 60 °C for the reaction time shown in Table 2. After the reaction, H₂O (2 mL) was added to the reaction mixture, which of the organic layer was extracted with EtOAc (5 mL x 3). The combined organic phases were evaporated under reduced pressure. To remove the siloxane residue, MeOH (15 mL) was rinsed to the crude material. The formed precipitate was filtered, and the filtrate was concentrated under reduced pressure. The crude product was purified by a silica gel column chromatography (hexane/EtOAc) to afford the lactone derivative.

5-Phenyl-dihydro-furan-2-one⁷ (2a)



General procedure was followed with 3-benzoylpropionic acid (**1a**, 178.4 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2a** as a colorless oil (152.8 mg, 94%): ¹H NMR (CDCl₃, 300 MHz) δ 2.12-2.28 (m, 1 H, CH₂), 2.62-2.73 (m, 3 H, CH₂, CH₂), 5.49-5.54 (m, 1 H, CH), 7.32-7.42 (m, 5 H, ArH); ¹³C NMR (CDCl₃, 75 MHz) δ 28.9, 30.9, 81.2, 125.2, 128.4, 128.7, 139.4, 176.9; IR (ATR, cm⁻¹) 1772 s; MS (EI) m/z (%) 162 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₀H₁₀O₂ [M]⁺: 162.0681, found: 162.0685.

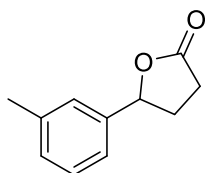
5-*o*-Tolyl-dihydro-furan-2-one⁷ (2b)



General procedure was followed with 3-(2-methylbenzoyl)propionic acid (**1b**, 192.6 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2b** as a colorless oil (151.8 mg, 86%): ¹H

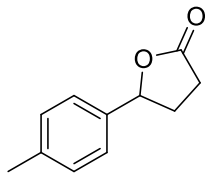
NMR (CDCl₃, 500 MHz) δ 2.06-2.14 (m, 1 H, CH₂), 2.34 (s, 3 H, CH₃), 2.64-2.71 (m, 3 H, CH₂, CH₂), 5.69-5.72 (m, 1 H, CH), 7.18 (dd, J = 4.5 Hz, 4.5 Hz, 1 H, ArH), 7.23-7.25 (m, 2 H, ArH), 7.35 (dd, J = 5.0 Hz, 4.5 Hz, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 18.9, 28.6, 29.5, 78.8, 124.1, 126.4, 128.1, 130.7, 134.1, 137.5, 177.1; IR (ATR, cm⁻¹) 1774 s; MS (EI) m/z (%) 176 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₁H₁₂O₂ [M]⁺: 176.0837, found: 176.0844.

5-*m*-Tolyl-dihydro-furan-2-one⁷ (2c)



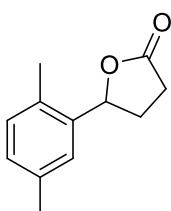
General procedure was followed with 3-(3-methylbenzoyl)propionic acid (**1c**, 192.4 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2c** as a colorless oil (166.7 mg, 95%): ¹H NMR (CDCl₃, 500 MHz) δ 2.13-2.23 (m, 1 H, CH₂), 2.36 (s, 3 H, CH₃), 2.60-2.67 (m, 3 H, CH₂, CH₂), 5.46-5.49 (m, 1 H, CH), 7.11-7.15 (m, 3 H, ArH), 7.25-7.29 (m, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 21.3, 28.9, 30.9, 81.2, 122.3, 125.8, 128.6, 129.1, 138.5, 139.3, 176.9; IR (ATR, cm⁻¹) 1774 s; MS (EI) m/z (%) 176 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₁H₁₂O₂ [M]⁺: 176.0837, found: 176.0839.

5-*p*-Tolyl-dihydro-furan-2-one⁷ (2d)



General procedure was followed with 3-(4-methylbenzoyl)propionic acid (**1d**, 192.0 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2d** as a white solid (157.7 mg, 90%): mp 70–71 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.15-2.21 (m, 1 H, CH₂), 2.36 (s, 3 H, CH₃), 2.61-2.66 (m, 3 H, CH₂, CH₂), 5.47-5.50 (m, 1 H, CH), 7.19-7.24 (m, 4 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 21.1, 29.0, 30.9, 81.3, 125.3, 129.3, 136.2, 138.3, 177.0; IR (ATR, cm⁻¹) 1764 s; MS (EI) m/z (%) 176 (M⁺, 87), 121 (100); HRMS (EI-Quadrupole): calcd. for C₁₁H₁₂O₂ [M]⁺: 176.0837, found: 176.0839.

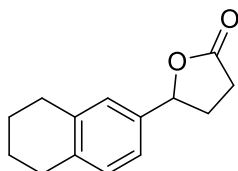
5-(2, 5-Dimethylphenyl)-dihydro-furan-2-one⁸ (2e)



General procedure was followed with 3-(2,5-dimethylbenzoyl)propionic acid (**1e**, 206.7 mg) for 24

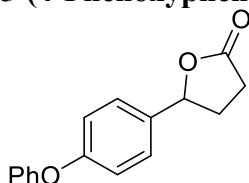
h. Column chromatography (7/3 = hexane/EtOAc) afforded **2e** as a pale yellow solid (147.4 mg, 78%): mp 76–77 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.07-2.12 (m, 1 H, CH₂), 2.28 (s, 3 H, CH₃), 2.32 (s, 3 H, CH₃), 2.64-2.66 (m, 3 H, CH₂, CH₂), 5.67-5.69 (m, 1 H, CH), 7.03-7.08 (m, 2 H, ArH), 7.16 (s, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 18.4, 21.0, 28.6, 29.6, 78.9, 124.7, 128.7, 130.6, 130.8, 135.9, 137.3, 177.1; IR (ATR, cm⁻¹) 1768 s; MS (EI) *m/z* (%) 190 (M⁺,100); HRMS (EI-Quadrupole): calcd. for C₁₂H₁₄O₂ [M]⁺: 190.0994, found: 199.0990.

5-(5,6,7,8-Tetrahydronaphthalen-2-yl)dihydrofuran-2(3H)-one (**2f**)



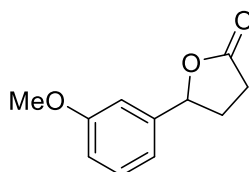
General procedure was followed with 4-oxo-4-(5,6,7,8-tetrahydronaphthalen-2-yl)butanoic acid (**1f**, 232.3 mg) for 24 h. Column chromatography (8/2 = hexane/EtOAc) afforded **2f** as a pale yellow oil (210.4 mg, 97%): ¹H NMR (CDCl₃, 500 MHz) δ 1.78-1.81 (m, 4 H, CH₂), 2.15-2.22 (m, 1 H, CH₂), 2.58-2.66 (m, 3 H, CH₂), 2.76 (m, 4 H, CH₂), 5.43-5.46 (m, 1 H, CH), 7.03-7.04 (m, 2 H, ArH), 7.07-7.09 (m, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 23.0, 29.0, 29.1, 29.4, 30.9, 81.4, 122.4, 126.0, 129.5, 136.3, 137.6, 177.1; IR (ATR, cm⁻¹) 1774 s; MS (EI) *m/z* (%) 216 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₄H₁₆O₂ [M]⁺: 216.1150, found: 216.1137.

5-(4-Phenoxyphenyl)-dihydro-furan-2-one (**2h**)



General procedure was followed with 3-(4-phenoxybenzoyl)propionic acid (**1h**, 270.1 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2h** as a white solid (182.0 mg, 72%): mp 60–62 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.16-2.25 (m, 1 H, CH₂), 2.60-2.68 (m, 3 H, CH₂, CH₂), 5.46-5.49 (m, 1 H, CH), 7.00-7.02 (m, 4 H, ArH), 7.12 (t, *J* = 7.5 Hz, 1 H, ArH), 7.29-7.36 (m, 4 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 29.1, 30.9, 81.0, 118.7, 119.1, 123.6, 127.0, 129.8, 133.7, 156.7, 157.5, 176.7; IR (ATR, cm⁻¹) 1753 s; MS (EI) *m/z* (%) 254 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₆H₁₄O₃ [M]⁺: 254.0943, found: 254.0949.

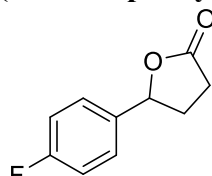
5-(3-Methoxyphenyl)-dihydro-furan-2-one (**2i**)



General procedure was followed with 3-(3-methoxybenzoyl)propionic acid (**1i**, 110.6 mg) for 24 h.

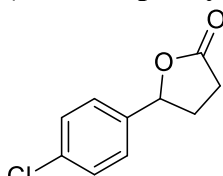
Column chromatography (8/2 = hexane/EtOAc) afforded **2i** as a colorless oil (160.5 mg, 84%): ^1H NMR (CDCl_3 , 500 MHz) δ 2.14-2.22 (m, 1 H, CH_2), 2.62-2.68 (m, 3 H, CH_2 , CH_2), 3.81 (s, 3 H, OCH_3), 5.47-5.50 (m, 1 H, CH), 6.86-6.91 (m, 3 H, ArH), 7.27-7.32 (m, 1 H, ArH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 28.8, 30.8, 55.2, 80.9, 110.7, 113.7, 117.3, 129.8, 141.0, 159.8, 176.8; IR (ATR, cm^{-1}) 1774 s; MS (EI) m/z (%) 192 (M^+ , 100); HRMS (FAB-Magnetic Sector): calcd. for $\text{C}_{11}\text{H}_{13}\text{O}_3$ [$\text{M}+\text{H}$] $^+$: 193.0859, found: 193.0864.

5-(4-Fluorophenyl)-dihydro-furan-2-one⁷ (**2j**)



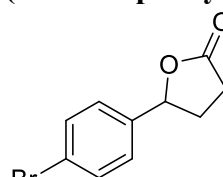
General procedure was followed with 3-(4-fluorobenzoyl)propionic acid (**1j**, 192.6 mg) for 7 days. Column chromatography (7/3 hexane/EtOAc) afforded **2j** as a pale yellow oil (136.2 mg, 76%): ^1H NMR (CDCl_3 , 500 MHz) δ 2.14-2.22 (m, 1 H, CH_2), 2.62-2.68 (m, 3 H, CH_2 , CH_2), 5.47-5.50 (m, 1 H, CH), 7.08 (t, $J = 8.5$ Hz, 2 H, ArH), 7.32 (dd, $J = 8.5$ Hz, 5.5 Hz, 2 H, ArH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 29.0, 31.0, 80.6, 115.7 ($J_{\text{C-F}} = 21.1$ Hz), 127.2 ($J_{\text{C-F}} = 8.6$ Hz), 135.0 ($J_{\text{C-F}} = 3.8$ Hz), 162.6 ($J_{\text{C-F}} = 246.6$ Hz), 176.6; IR (ATR, cm^{-1}) 1773 s; MS (EI) m/z (%) 180 (M^+ , 95), 125 (100); HRMS (EI-Quadrupole): calcd. for $\text{C}_{10}\text{H}_9\text{O}_2\text{F}$ [M] $^+$: 180.0587, found: 180.0587.

5-(4-Chlorophenyl)-dihydro-furan-2-one⁷ (**2k**)



General procedure was followed with 3-(4-chlorobenzoyl)propionic acid (**1k**, 212.8 mg) for 7 days. Column chromatography (7/3 = hexane/EtOAc) afforded **2k** as a colorless oil (123.2 mg, 63%): ^1H NMR (CDCl_3 , 500 MHz) δ 2.13-2.18 (m, 1 H, CH_2), 2.64-2.69 (m, 3 H, CH_2 , CH_2), 5.46-5.49 (m, 1 H, CH), 7.28 (d, $J = 9.0$ Hz, 2 H, ArH), 7.36 (d, $J = 9.0$ Hz, 2 H, ArH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 28.8, 30.9, 80.4, 126.6, 128.9, 134.2, 137.8, 176.5; IR (ATR, cm^{-1}) 1776 s; MS (EI) m/z (%) 198 (M^++2 , 17), 196 (M^+ , 56), 58 (100); HRMS (EI-Quadrupole): calcd. for $\text{C}_{10}\text{H}_9\text{O}_2\text{Cl}$ [M] $^+$: 196.0291, found: 196.0295.

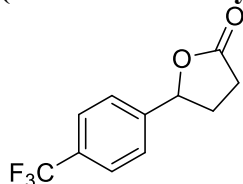
5-(4-Bromophenyl)-dihydro-furan-2-one⁷ (**2l**)



General procedure was followed with 3-(4-bromobenzoyl)propionic acid (**1l**, 161.4 mg) for 7 days.

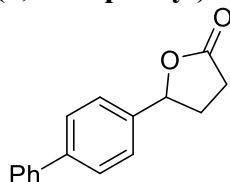
Column chromatography (7/3 = hexane/EtOAc) afforded **2l** as a white solid (161.4 mg, 67%): mp 81–82 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.13-2.18 (m, 1 H, CH₂), 2.64-2.69 (m, 3 H, CH₂, CH₂), 5.45-5.48 (m, 1 H, CH), 7.22 (d, *J* = 9.0 Hz, 2 H, ArH), 7.52 (d, *J* = 9.0 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 28.8, 30.9, 80.4, 122.4, 126.9, 131.9, 138.4, 176.5; IR (ATR, cm⁻¹) 1760 s; MS (EI) *m/z* (%) 242 (M⁺+2, 32), 240 (M⁺, 33) 58 (100); HRMS (EI-Quadrupole): calcd. for C₁₀H₉O₂⁸¹Br [M+2]⁺: 241.9765, found: 241.9757.

5-(4-Trifluoromethylphenyl)-dihydro-furan-2-one⁹ (**2m**)



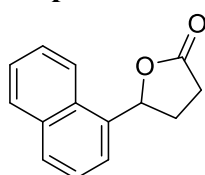
General procedure was followed with 3-(4-trifluoromethylbenzoyl)propionic acid (**1m**, 246.2 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2m** as a pale orange oil (144.6 mg, 63%): ¹H NMR (CDCl₃, 500 MHz) δ 2.13-2.21 (m, 1 H, CH₂), 2.62-2.77 (m, 3 H, CH₂, CH₂), 5.55-5.58 (m, 1 H, CH), 7.47 (d, *J* = 8.0 Hz, 2 H, ArH), 7.66 (d, *J* = 8.0 Hz, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 28.6, 30.9, 80.1, 123.8 (*J*_{C-F} = 272.0 Hz), 125.4, 125.7 (*J*_{C-F} = 3.9 Hz), 130.6 (*J*_{C-F} = 32.6 Hz), 143.4, 176.4; IR (ATR) 1782 s; MS (EI) *m/z* (%) 230 (M⁺, 52), 58 (100); HRMS (FAB-Magnetic Sector): calcd. for C₁₁H₁₀O₂F₃ [M+H]⁺: 231.0627, found: 231.0633.

5-(1,1'-Biphenyl)-4-yl-dihydro-furan-2-one¹⁰ (**2n**)



General procedure was followed with 4-([1,1'-biphenyl]-4-yl)-4-oxobutanoic acid (**1n**, 254.6 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2n** as a pale yellow solid (171.0 mg, 72%): mp 103–105 °C; ¹H NMR (CDCl₃, 500 MHz) δ 2.19-2.29 (m, 1 H, CH₂), 2.66-2.72 (m, 3 H, CH₂, CH₂), 5.54-5.57 (m, 1 H, CH), 7.35-7.46 (m, 5 H, ArH), 7.58-7.62 (m, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 29.0, 30.9, 81.0, 125.8, 127.1, 127.5, 127.5, 128.8, 138.3, 140.4, 141.4, 176.8; IR (ATR, cm⁻¹) 1766 s; MS (EI) *m/z* (%) 238 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₆H₁₄O₂ [M]⁺: 238.0994, found: 238.1000.

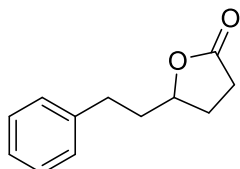
5-Naphthalen-1-yl-dihydro-furan-2-one (**2o**)



General procedure was followed with 4-(1-naphthyl)-4-oxobutanoic acid (**1o**, 228.3 mg) for 24 h.

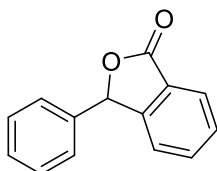
Column chromatography (7/3 = hexane/EtOAc) afforded **2o** as a colorless oil (165.4 mg, 78%): ¹H NMR (CDCl₃, 500 MHz) δ 2.23-2.31 (m, 1 H, CH₂), 2.60-2.73 (m, 2 H, CH₂), 2.83-2.91 (m, 1 H, CH₂), 6.23-6.26 (m, 1 H, CH), 7.45-7.56 (m, 4 H, ArH), 7.81-7.90 (m, 3 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 28.2, 29.9, 78.6, 121.5, 122.4, 125.3, 125.9, 126.5, 128.7, 129.1, 129.4, 133.7, 135.0, 177.1; IR (ATR, cm⁻¹) 1774 s; MS (EI) *m/z* (%) 212 (M⁺, 100); HRMS (EI-Quadrupole): calcd. for C₁₄H₁₂O₂ [M]⁺: 212.0837, found: 212.0835.

5-Phenethyldihydrofuran-2(3H)-one¹¹ (**4**)



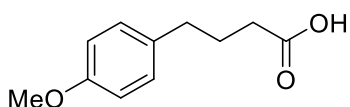
General procedure was followed with γ -oxo-benzenehexanoic acid (**3**, 206.6 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **4** as a colorless oil (181.8 mg, 96%): ¹H NMR (CDCl₃, 500 MHz) δ 1.83-1.95 (m, 2 H, CH₂), 2.01-2.09 (m, 1 H, CH₂), 2.27-2.34 (m, 1 H, CH₂), 2.51-2.55 (m, 2 H, CH₂), 2.70-2.76 (m, 1 H, CH₂), 2.80-2.86 (m, 1 H, CH₂), 4.44-4.50 (m, 1 H, CH), 7.19-7.22 (m, 3 H, ArH), 7.28-7.31 (m, 2 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 27.9, 28.8, 31.6, 37.3, 79.8, 126.1, 128.4, 128.5, 140.7, 177.1; IR (ATR, cm⁻¹) 1770 s; MS (EI) *m/z* (%) 190 (M⁺, 51), 130 (100); HRMS (EI-Quadrupole): calcd. for C₁₂H₁₄O₂ [M]⁺: 190.0994, found: 190.0994.

3-Phenylisobenzofuran-1-one¹² (**6**)



General procedure was followed with 2-benzoylbenzoic acid (**5**, 226.1 mg) for 24 h. Column chromatography (9/1 = hexane/EtOAc) afforded **6** as a white solid (103.9 mg, 49%): mp 116–117 °C; ¹H NMR (CDCl₃, 500 MHz) δ 6.41 (s, 1 H, CH), 7.27-7.39 (m, 6 H, ArH), 7.56 (t, *J* = 7.5 Hz, 1 H, ArH), 7.63 (t, *J* = 7.5 Hz, 1 H, ArH), 7.96 (d, *J* = 7.5 Hz, 1 H, ArH); ¹³C NMR (CDCl₃, 125 MHz) δ 82.7, 122.8, 125.6, 125.6, 126.9, 128.9, 128.9, 129.3, 129.3, 134.3, 136.4, 149.6, 170.5; IR (ATR, cm⁻¹) 1746 s; MS (EI) *m/z* (%) 210 (M⁺, 85) 105 (100); HRMS (EI-Quadrupole): calcd. for C₁₄H₁₀O₂ [M]⁺: 210.0681, found: 210.0689.

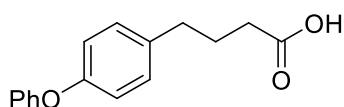
4-(4-Methoxyphenyl)butanoic acid¹³ (**2g'**)



General procedure was followed with 3-(4-methoxybenzoyl)propionic acid (**1g**, 208.2 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2g'** as a white solid (41.0 mg, 21%): mp

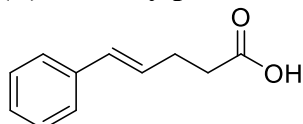
57–59 °C ^1H NMR (CDCl_3 , 500 MHz) δ 1.93 (quint, $J = 7.5$ Hz, 2 H, CH_2), 2.36 (t, $J = 7.5$ Hz, 2 H, CH_2), 2.61 (t, $J = 7.5$ Hz, 2 H, CH_2), 6.83 (d, $J = 8.5$ Hz, 2 H, ArH), 7.10 (d, $J = 8.5$ Hz, 2 H, ArH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 26.4, 33.2, 34.0, 55.2, 113.8, 129.4, 133.2, 157.9, 179.7; IR (ATR, cm^{-1}) 1694 s; MS (EI) m/z (%) 194 (M^+ , 33), 121 (100); HRMS (EI-Quadrupole): calcd. for $\text{C}_{11}\text{H}_{14}\text{O}_3$ [M] $^+$: 190.0943, found: 190.0947.

4-(4-Phenoxyphenyl)butanoic acid¹⁴ (**2h'**)



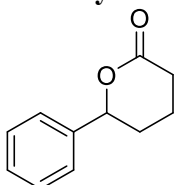
General procedure was followed with 3-(4-phenoxybenzoyl)propionic acid (**1h**, 270.1 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **2h'** as a white solid (11.3 mg, 4%): mp 41–43 °C; ^1H NMR (CDCl_3 , 500 MHz) δ 1.96 (quint, $J = 7.5$ Hz, 2 H, CH_2), 2.39 (t, $J = 7.5$ Hz, 2 H, CH_2), 2.61 (t, $J = 7.5$ Hz, 2 H, CH_2), 6.94 (d, $J = 8.0$ Hz, 2 H, ArH), 6.99 (dd, $J = 9.0$ Hz, 1.0 Hz 2 H, ArH), 7.08 (t, $J = 8.0$ Hz, 1 H, ArH), 7.14 (d, $J = 9.0$ Hz, 2 H, ArH), 7.32 (dd, $J = 8.0$ Hz, 1.0 Hz 2 H, ArH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 26.3, 33.2, 34.2, 118.6, 119.0, 123.0, 129.7, 136.1, 155.4, 157.5, 179.4; IR (ATR, cm^{-1}) 1690 s; MS (EI) m/z (%) 256 (M^+ , 41), 183 (100); HRMS (EI-Quadrupole): calcd. for $\text{C}_{16}\text{H}_{16}\text{O}_3$ [M] $^+$: 256.10995, found: 256.10940.

(*E*)-5-Phenylpent-4-enoic acid¹⁵ (**8**)



General procedure was followed with 5-oxo-5-phenylpentanoic acid (**7**, 192.3 mg) for 24 h. Column chromatography (7/3 = hexane/EtOAc) afforded **8'** as a white solid (12.0 mg, 8%): mp 88–90 °C; ^1H NMR (CDCl_3 , 500 MHz) δ 2.55 (m, 4 H, CH_2), 6.18–6.24 (m, 1 H, CH), 6.45 (d, $J = 16.0$ Hz, 1 H, CH), 7.21 (t, $J = 7.5$ Hz, 1 H, ArH), 7.29 (t, $J = 7.5$ Hz, 2 H, ArH), 7.34 (d, $J = 7.5$ Hz, 2 H, ArH); ^{13}C NMR (CDCl_3 , 125 MHz) δ 27.9, 33.7, 126.1, 127.2, 128.0, 128.5, 131.2, 137.2, 178.9; IR (ATR, cm^{-1}) 1694 s; MS (EI) m/z (%) 176 (M^+ , 39), 117 (100); HRMS (EI-Quadrupole): calcd. for $\text{C}_{11}\text{H}_{12}\text{O}_2$ [M] $^+$: 176.0837, found: 176.0838.

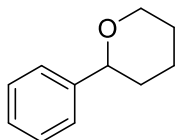
6-Phenyltetrahydro-2H-pyran-2-one¹⁰ (**9**)



General procedure was followed with 5-oxo-5-phenylpentanoic acid (**7**, 192.6 mg) and triethylsilane for 4 h. Column chromatography (7/3 = hexane/EtOAc) afforded **8** as a colorless oil (14.5 mg, 8%): ^1H NMR (CDCl_3 , 500 MHz) δ 1.83–1.90 (zm, 1 H, CH_2), 1.97–2.02 (m, 2 H, CH_2),

2.15-2.19 (m, 1 H, CH_2), 2.55-2.61 (m, 1 H, CH_2), 2.69-2.74 (m, 1 H, CH_2), 2.51-2.55 (m, 2 H, CH_2), 2.70-2.76 (m, 1 H, CH_2), 2.80-2.86 (m, 1 H, CH_2), 5.36 (dd, 1 H, $J = 10.5, 3.5$ Hz, CH), 7.31-7.40 (m, 5 H, ArH); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 18.6, 29.5, 30.5, 81.6, 125.7, 128.2, 128.6, 139.7, 171.3; IR (ATR, cm^{-1}) 1732 s; MS (EI) m/z (%) 176 (M^+ , 51), 104 (100); HRMS (EI-Quadrupole): calcd. for $C_{11}H_{12}O_2 [M]^+$: 176.0837, found: 176.0834.

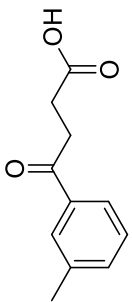
2-Phenyltetrahydropyran¹⁶ (9')



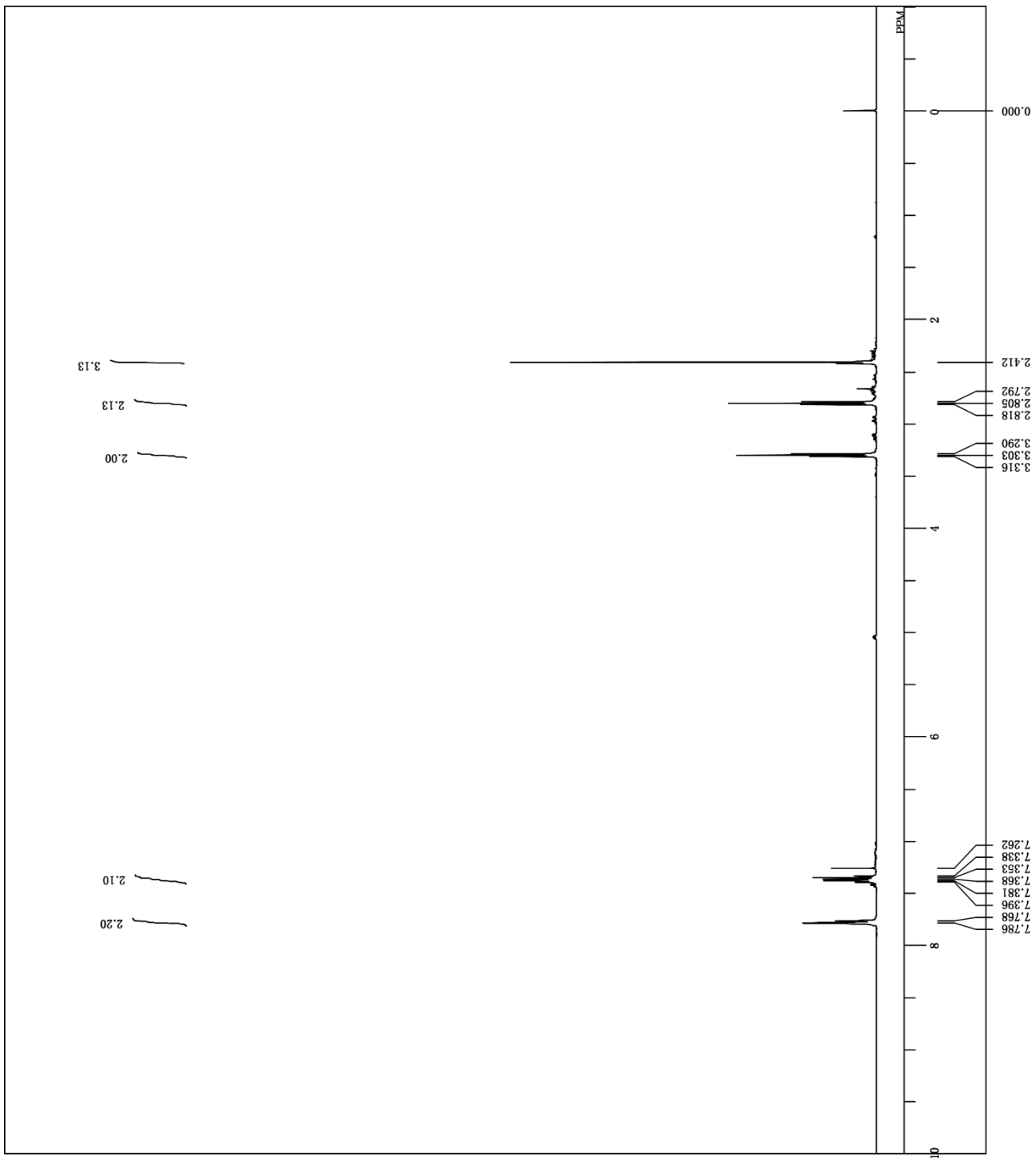
General procedure was followed with 5-oxo-5-phenylpentanoic acid (7, 192.6 mg) and triethylsilane for 4 h. Gel permeation chromatography (chloroform) afforded **9'** as a colorless oil (34.6 mg, 21%): 1H NMR ($CDCl_3$, 500 MHz) δ 1.57-1.72 (m, 5 H, CH_2), 1.82-1.84 (m, 1 H, CH_2), 1.93-1.95 (m, 1 H, CH_2), 3.59-3.64 (m, 1 H, CH_2), 4.12-4.15 (m, 1 H, CH_2), 4.32 (dd, $J = 11.0$ Hz, 2.0 Hz, 1 H, CH_2), 7.23-7.25 (m, 1 H, ArH), 7.31-7.36 (m, 4 H, ArH); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 24.0, 25.9, 34.0, 69.0, 80.1, 125.8, 127.2, 128.2, 143.3; MS (EI) m/z (%) 162 (M^+ , 93), 105 (100); HRMS (EI-Quadrupole): calcd. for $C_{11}H_{14}O [M]^+$: 162.1045, found: 162.1042.

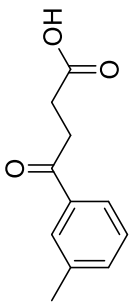
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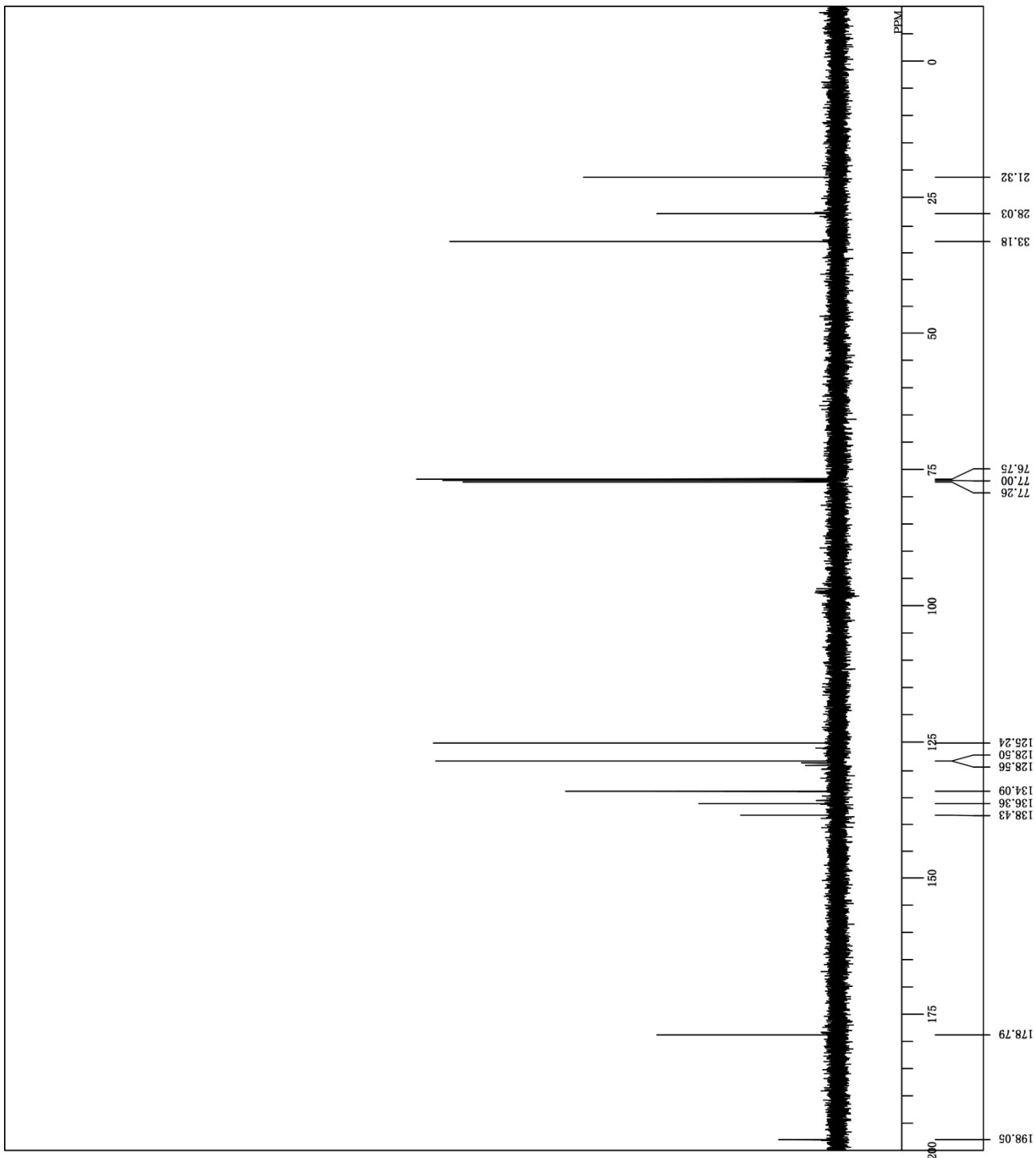


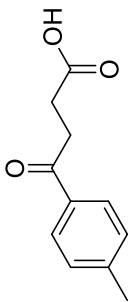
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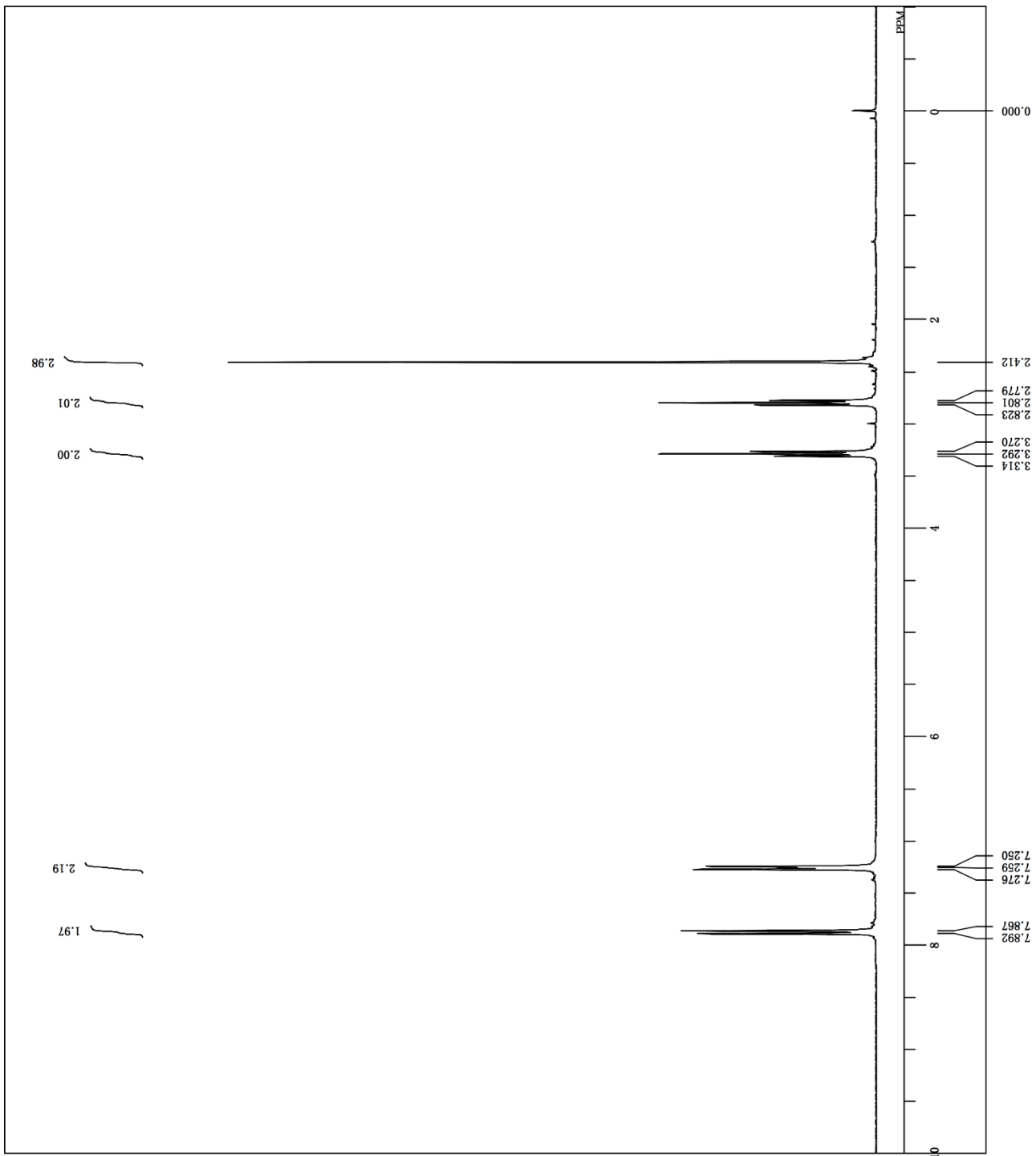


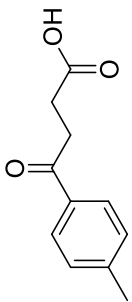
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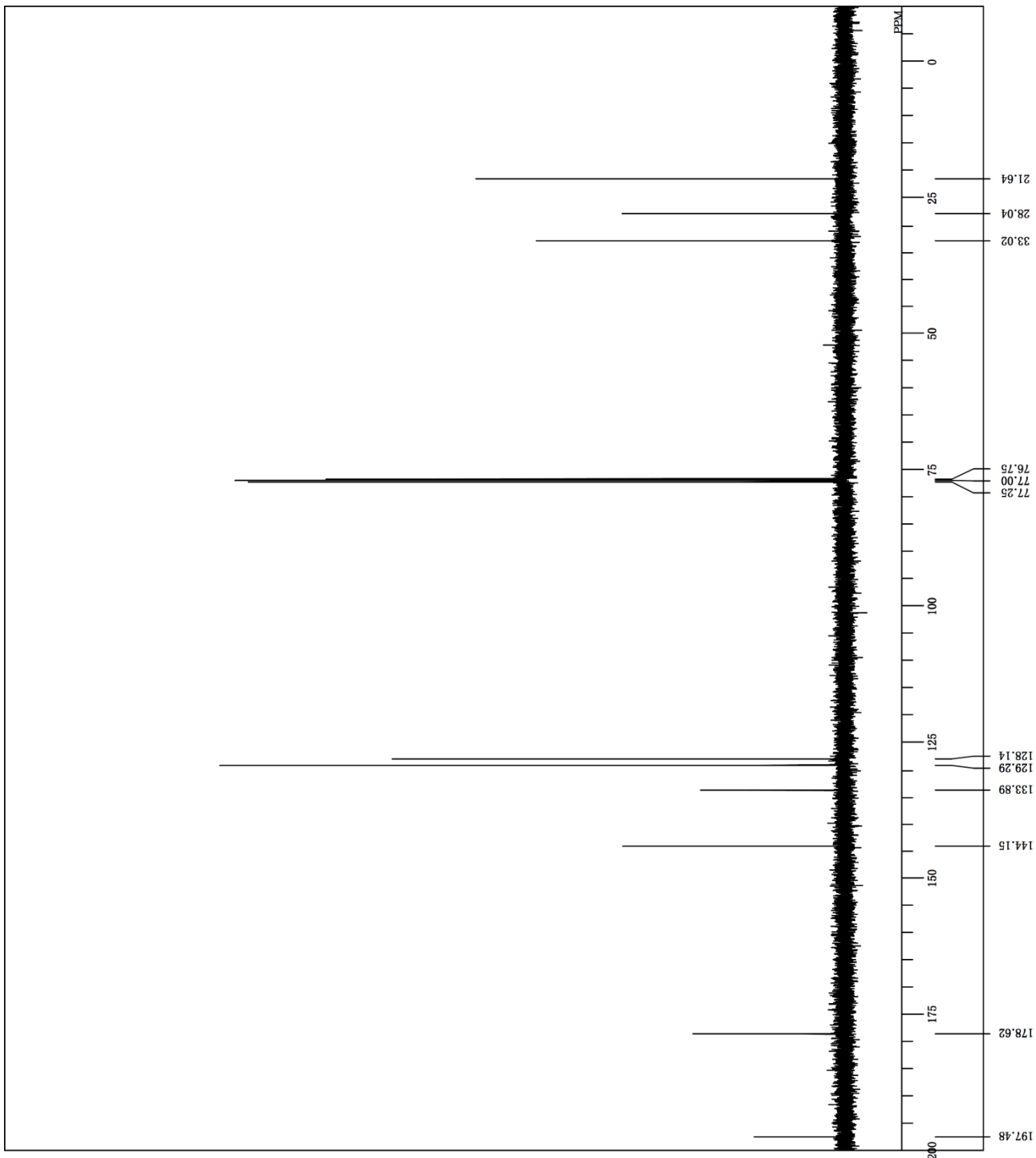


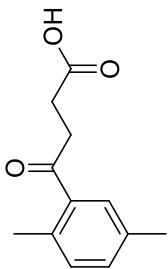
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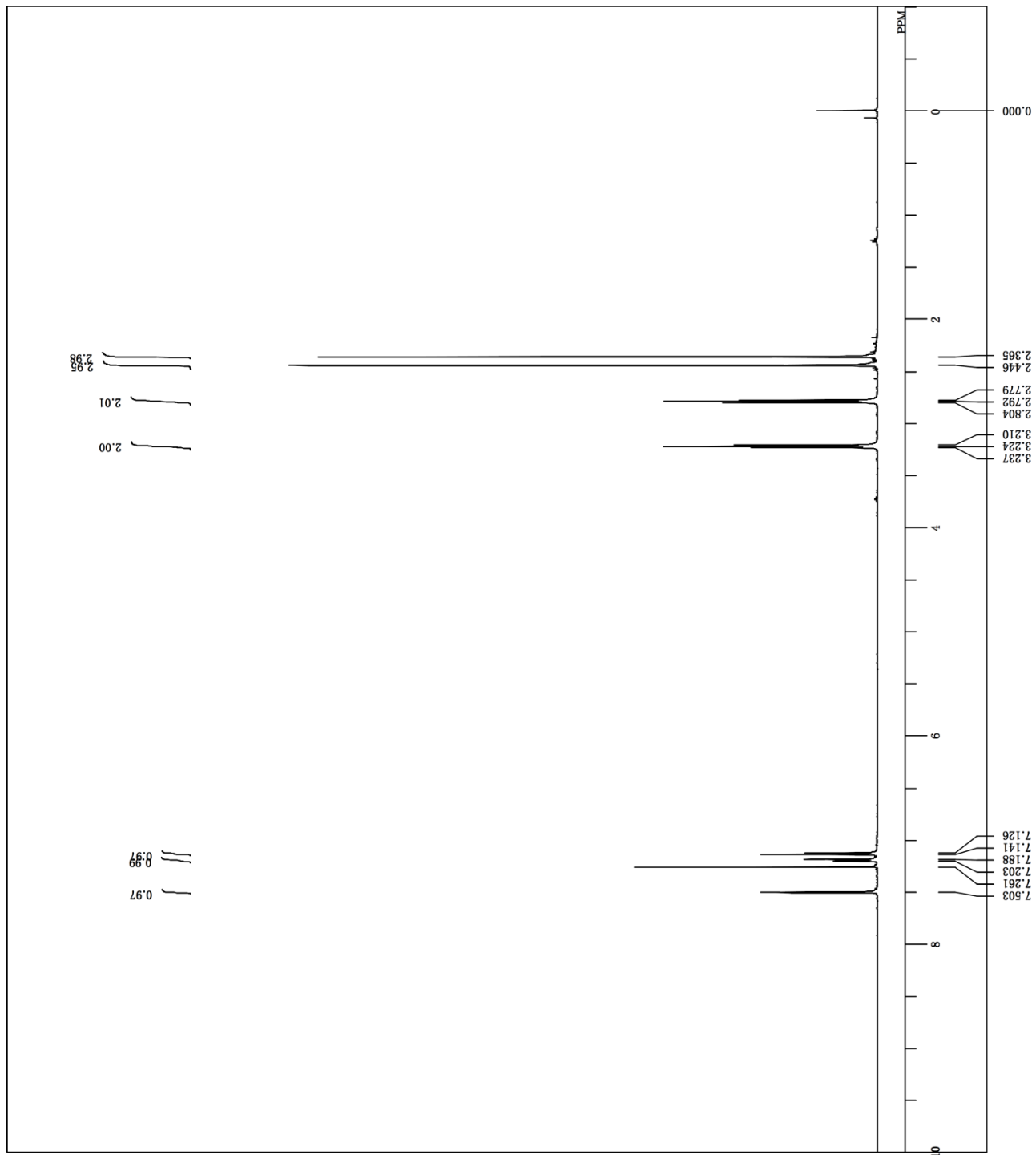


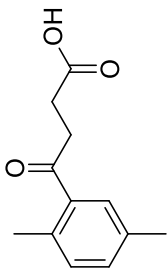
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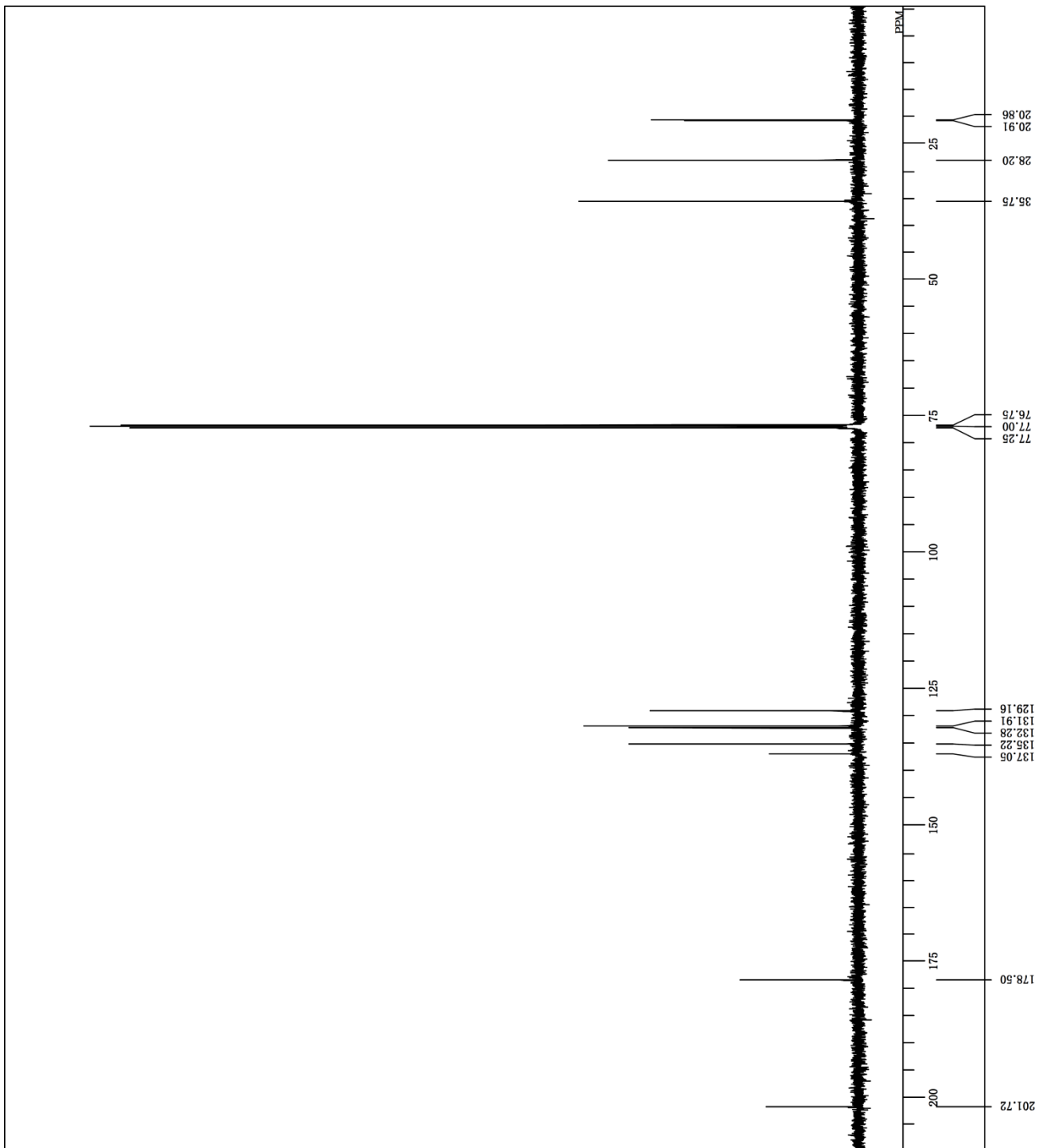


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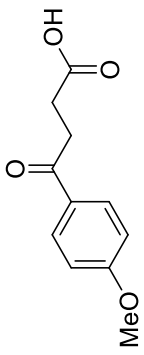




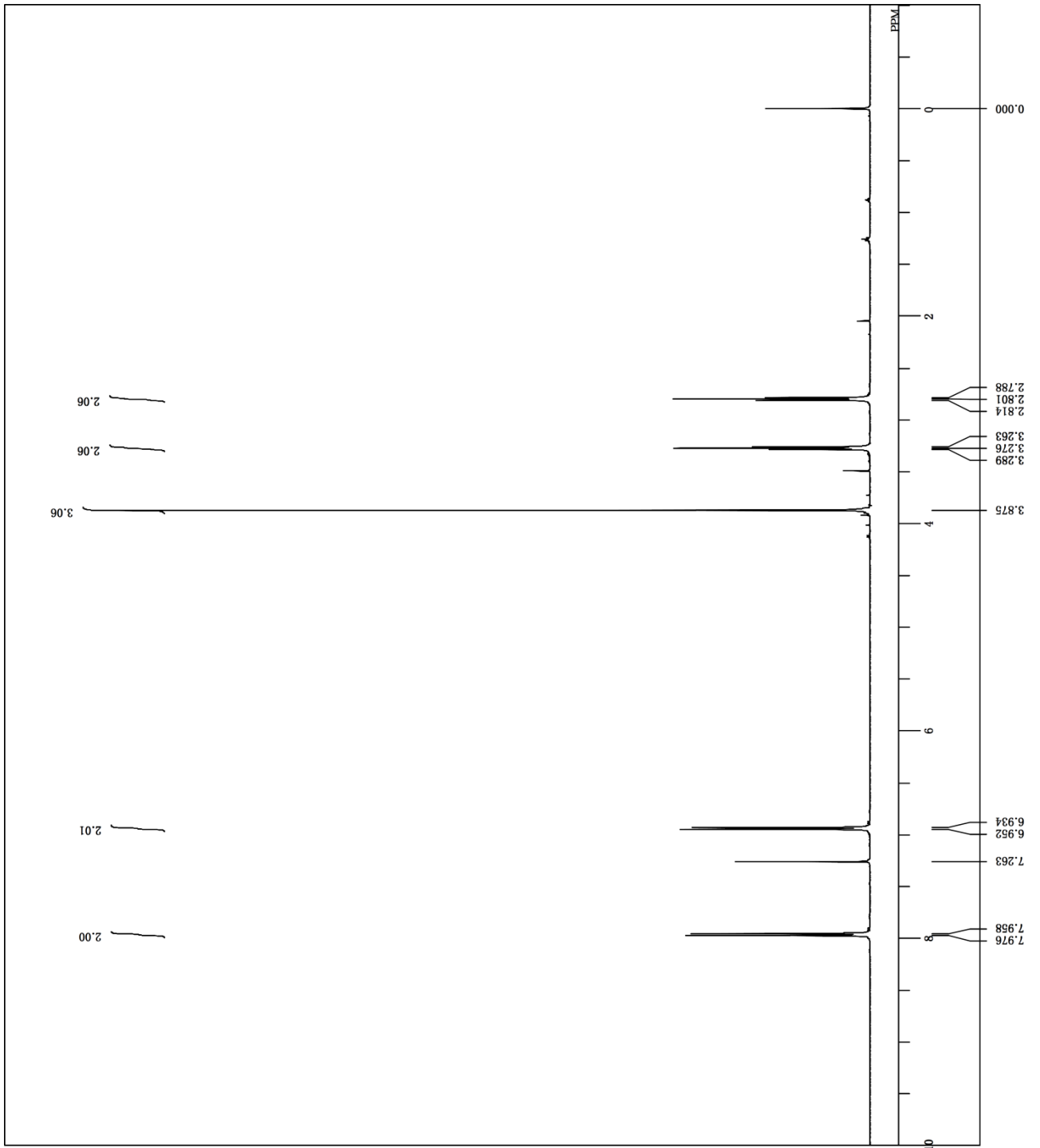
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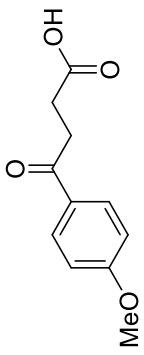


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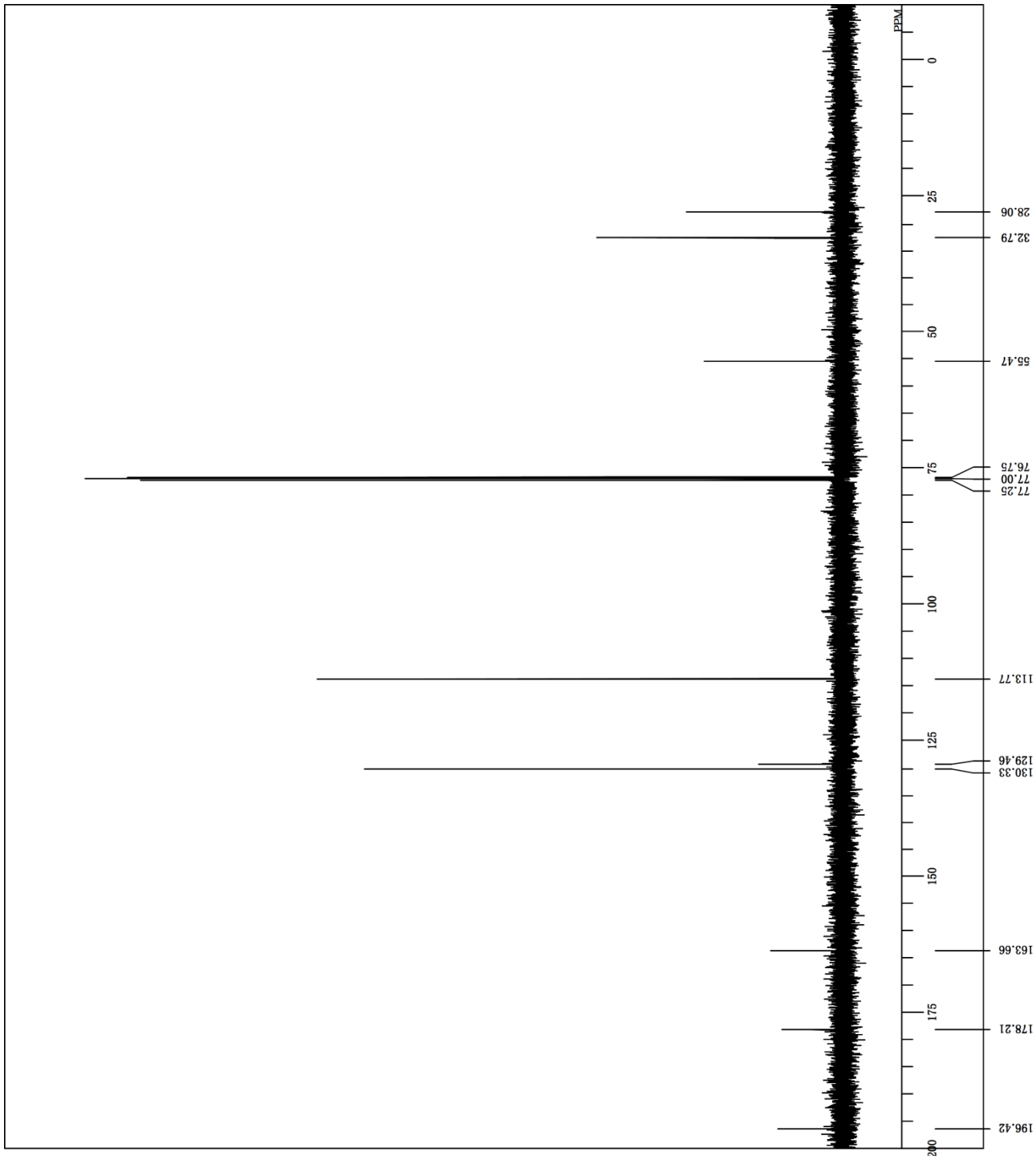


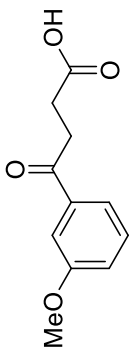
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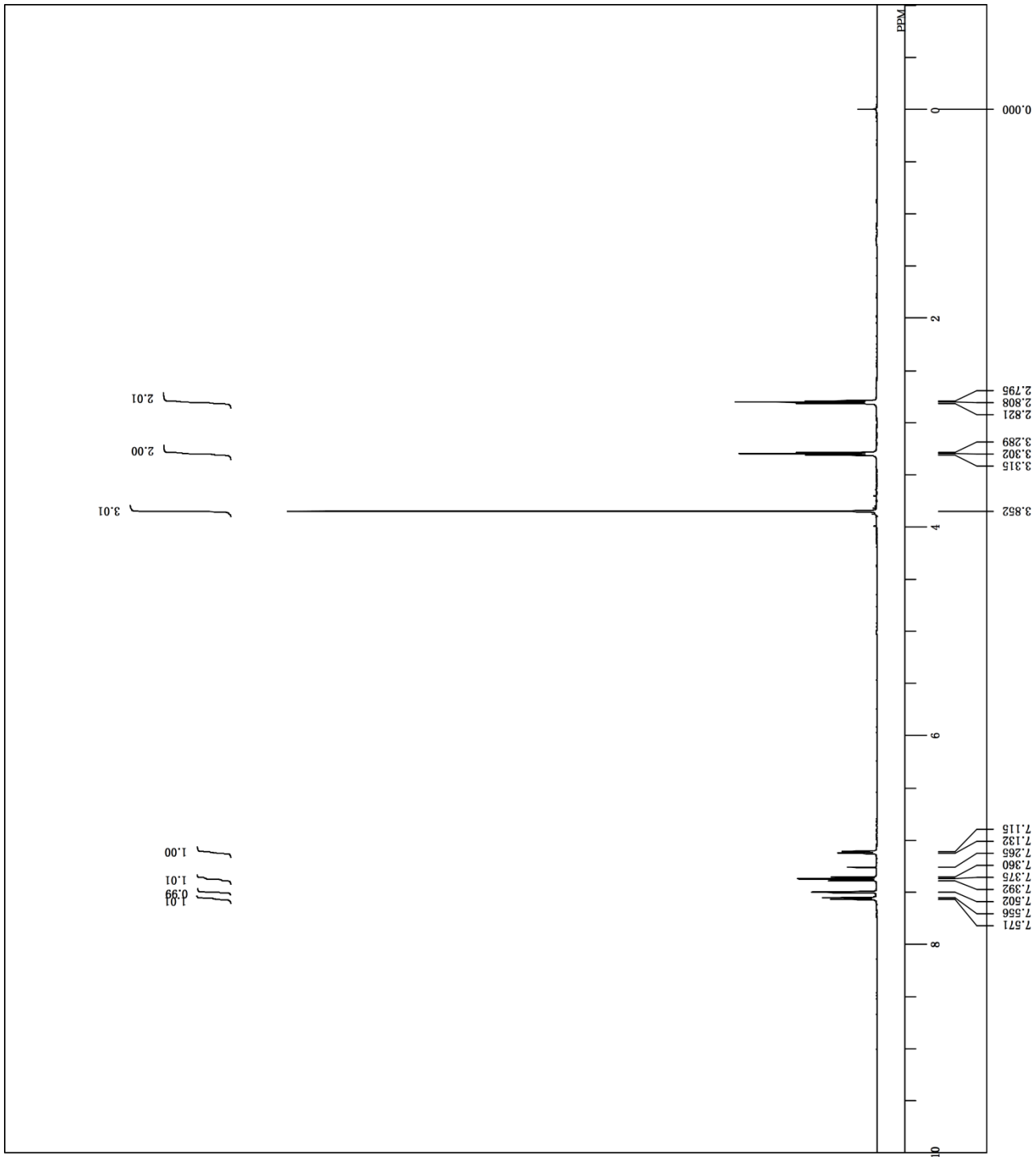


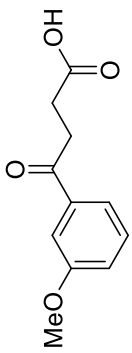
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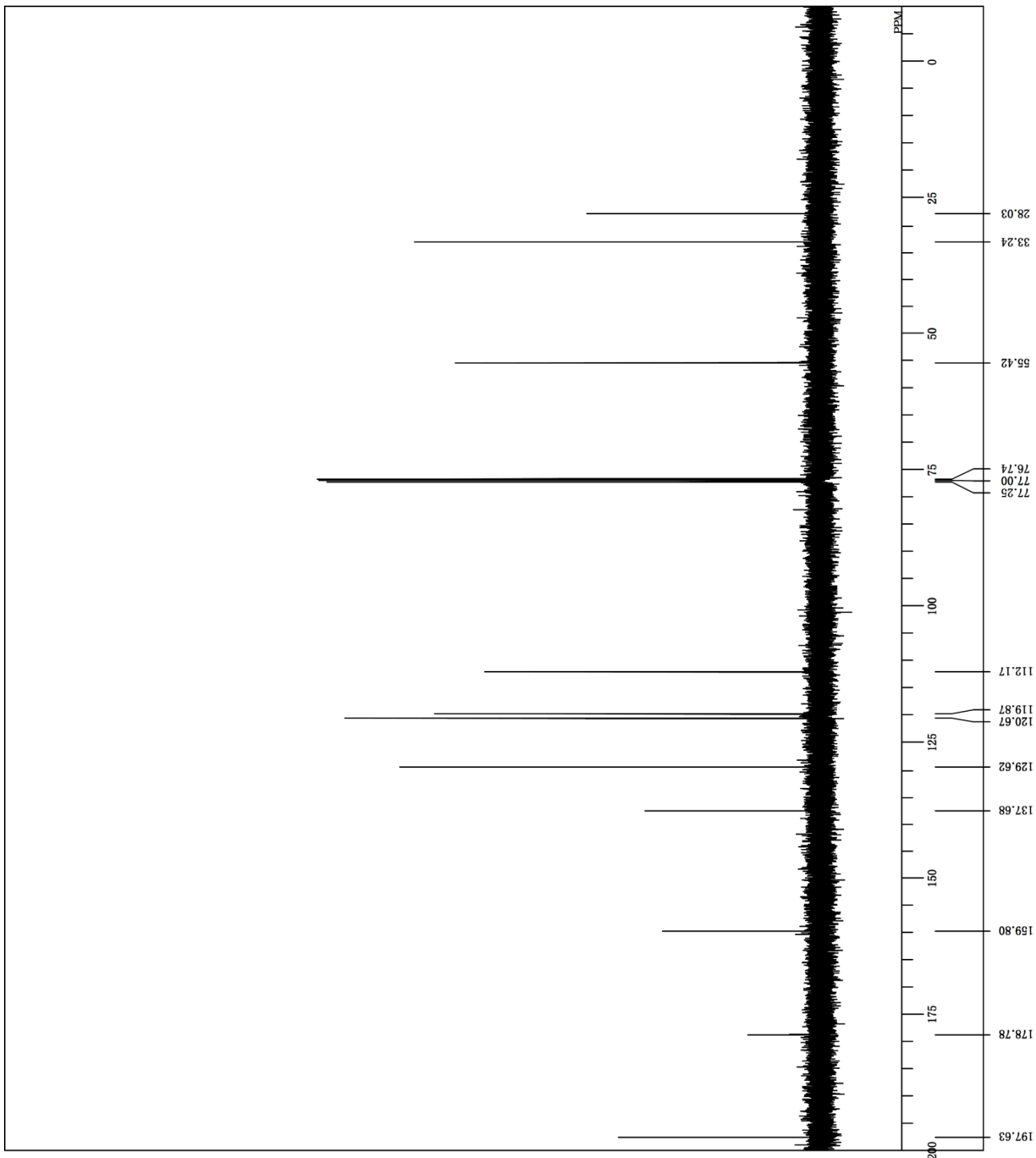


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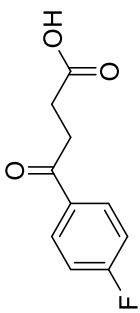




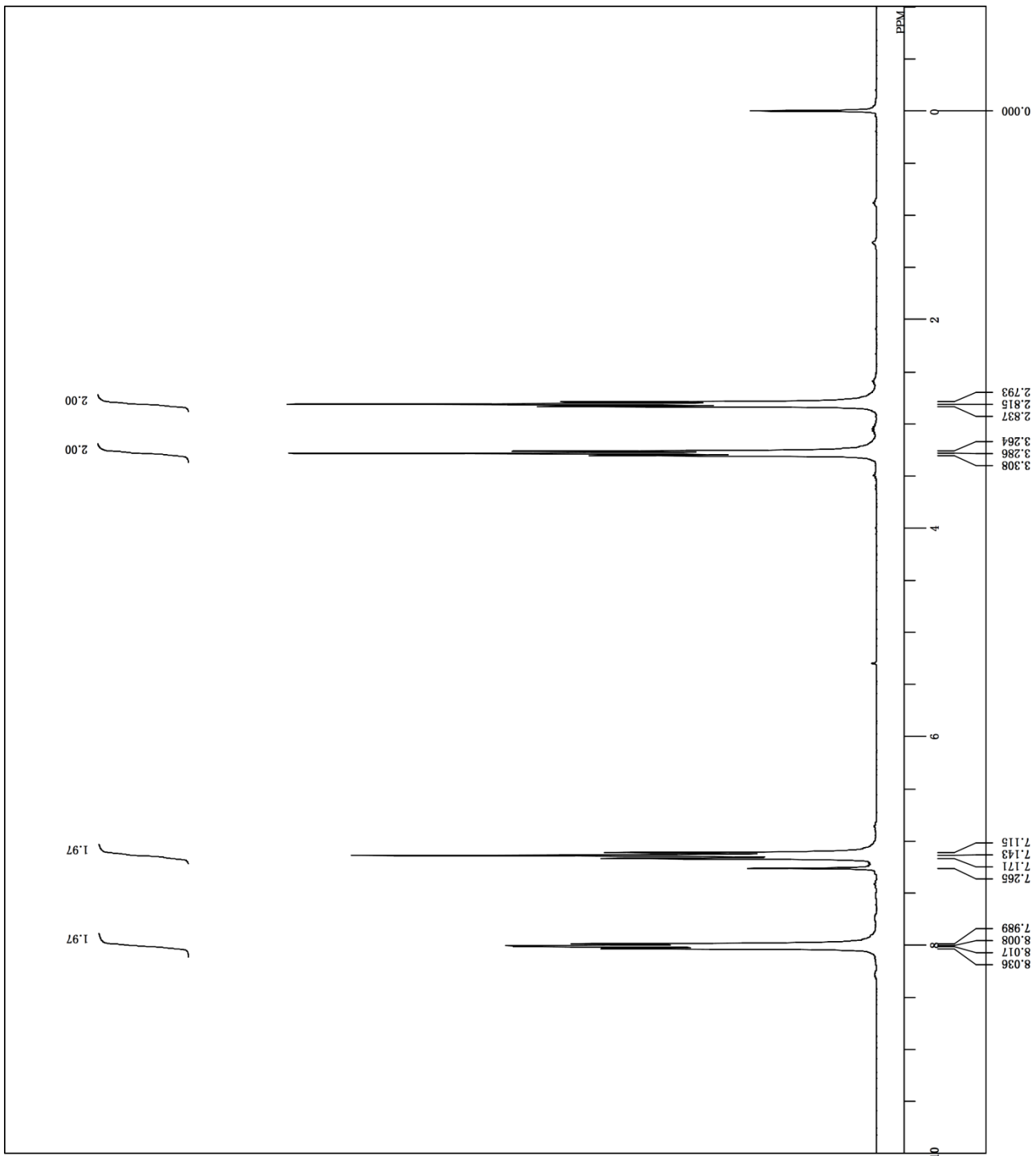
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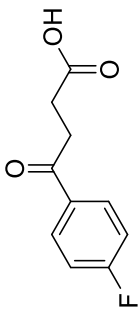


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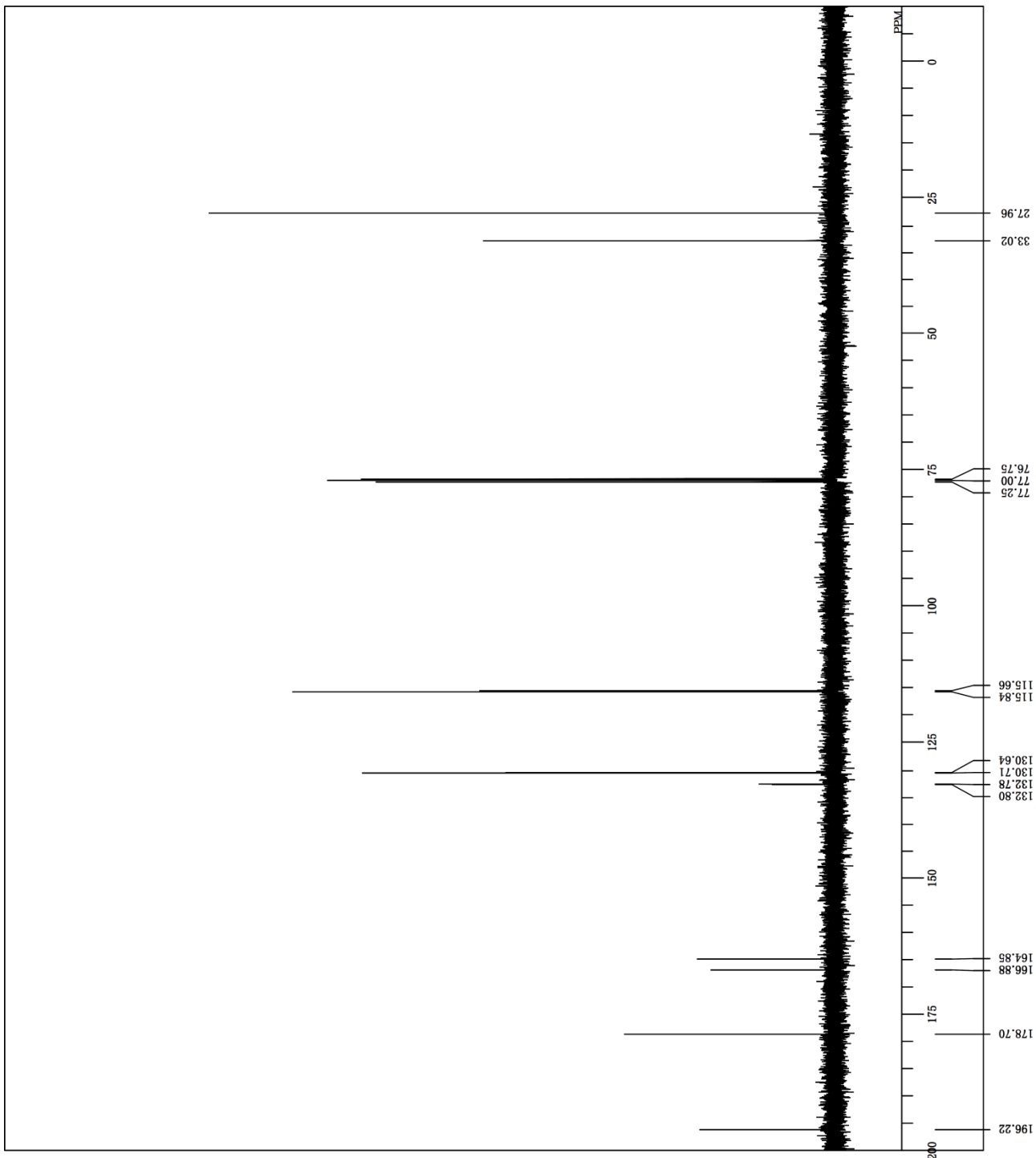


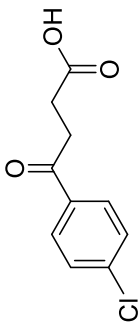
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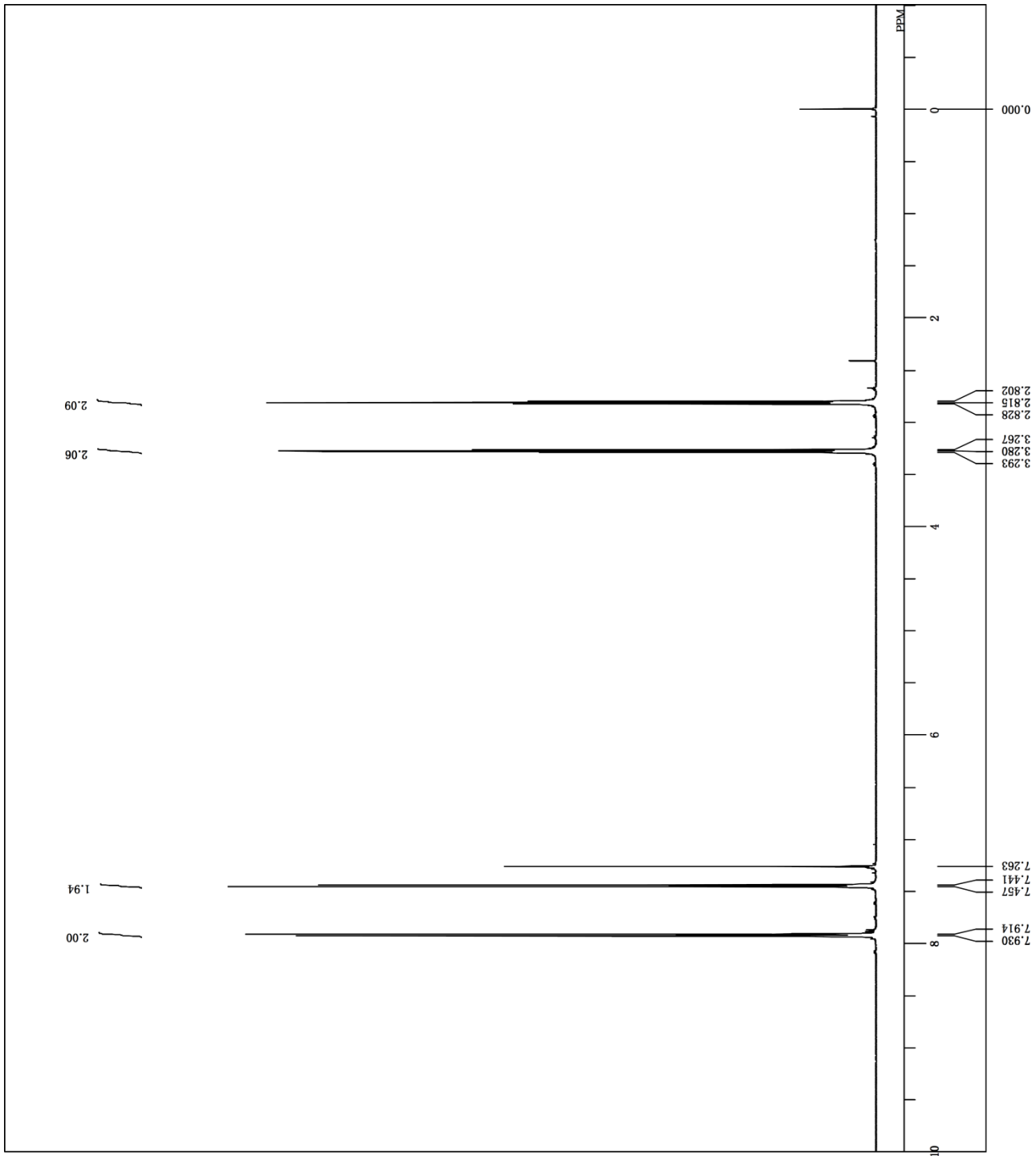


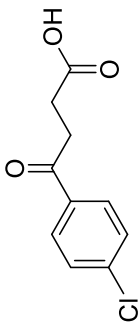
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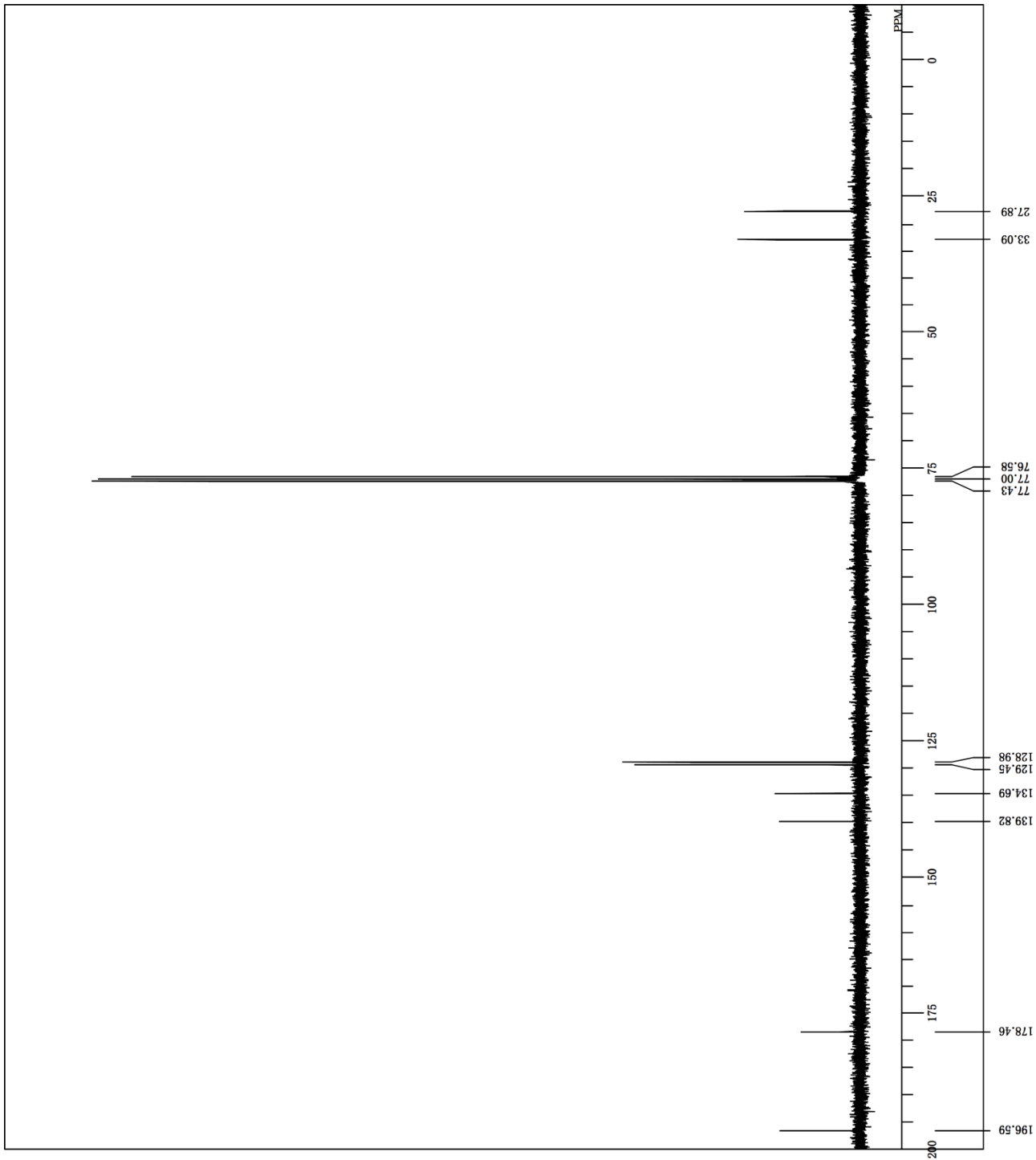


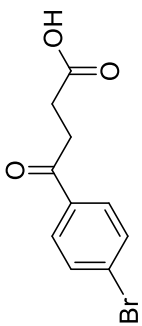
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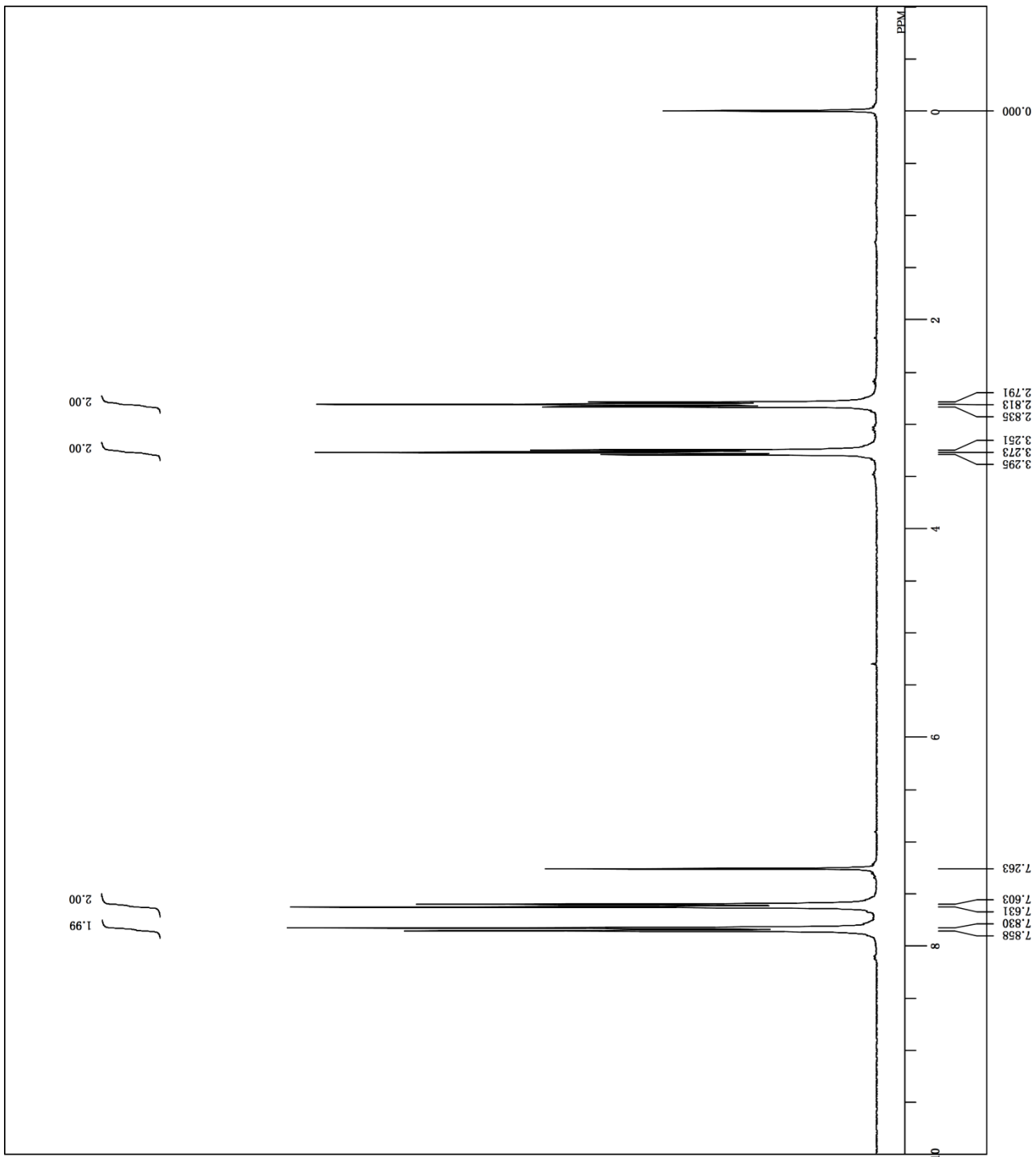


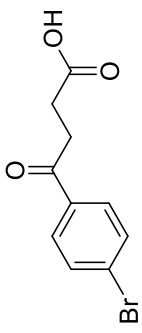
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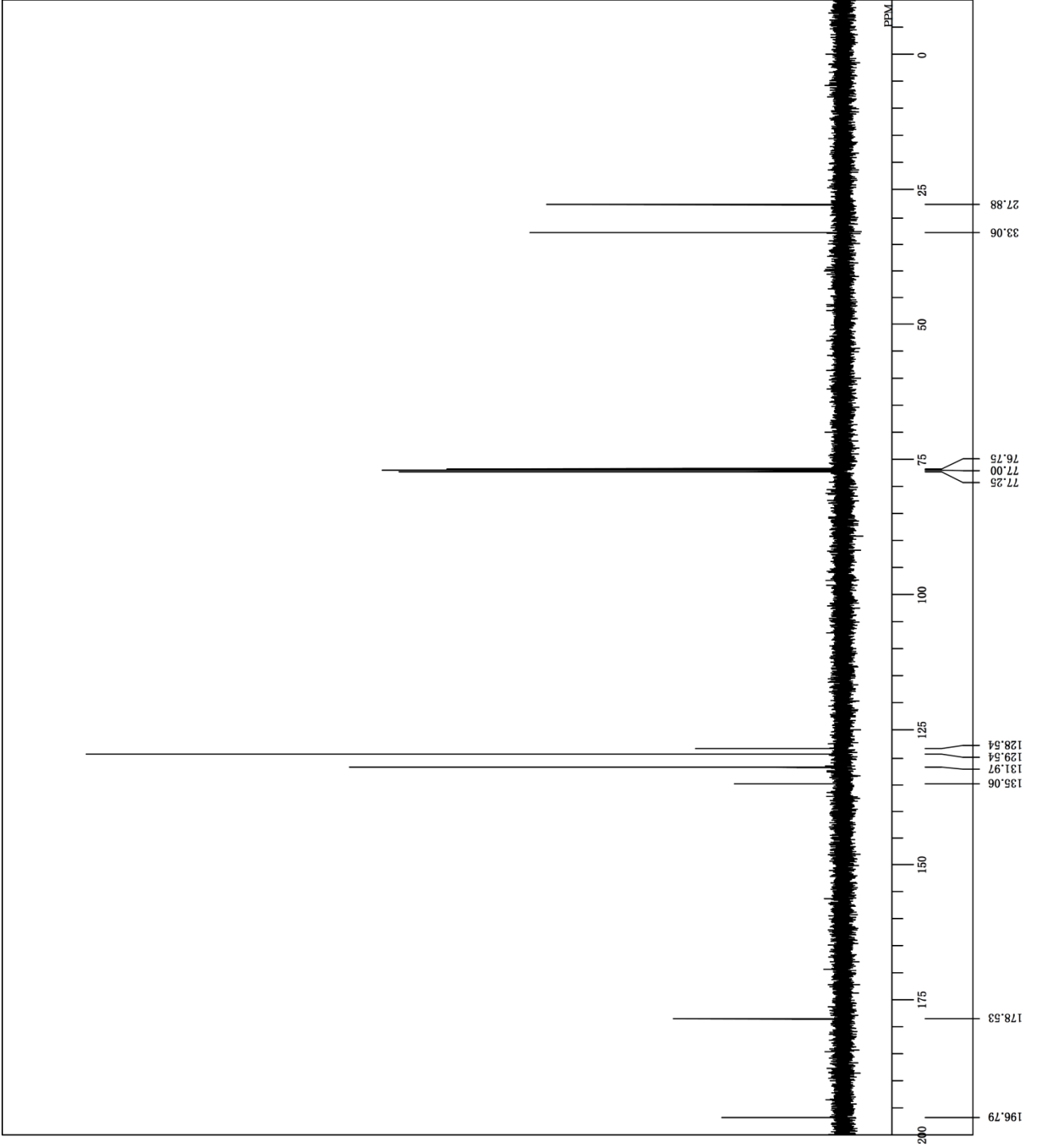


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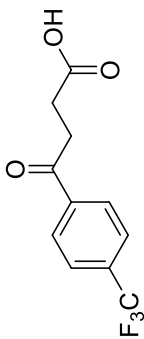




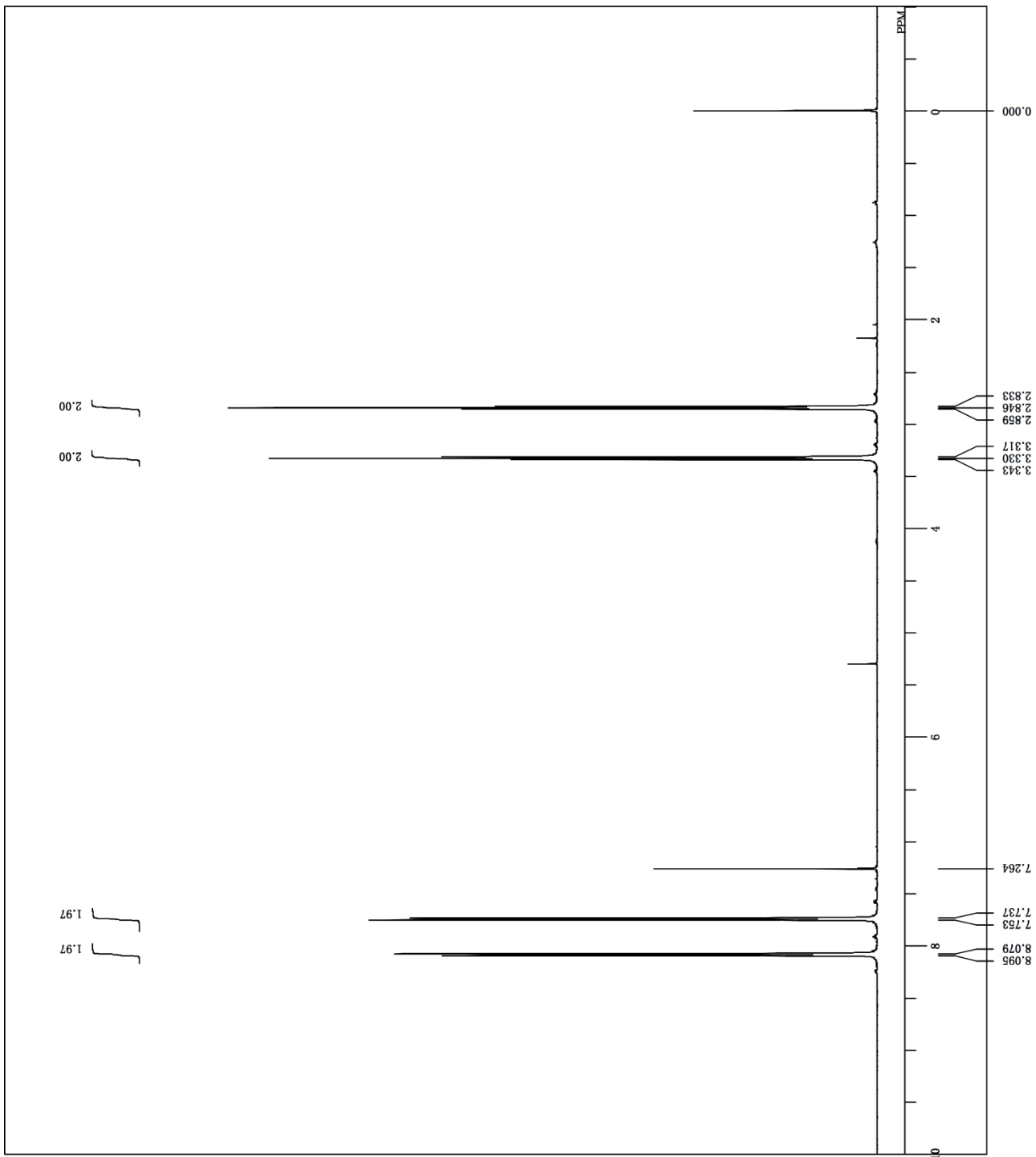
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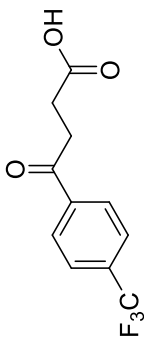


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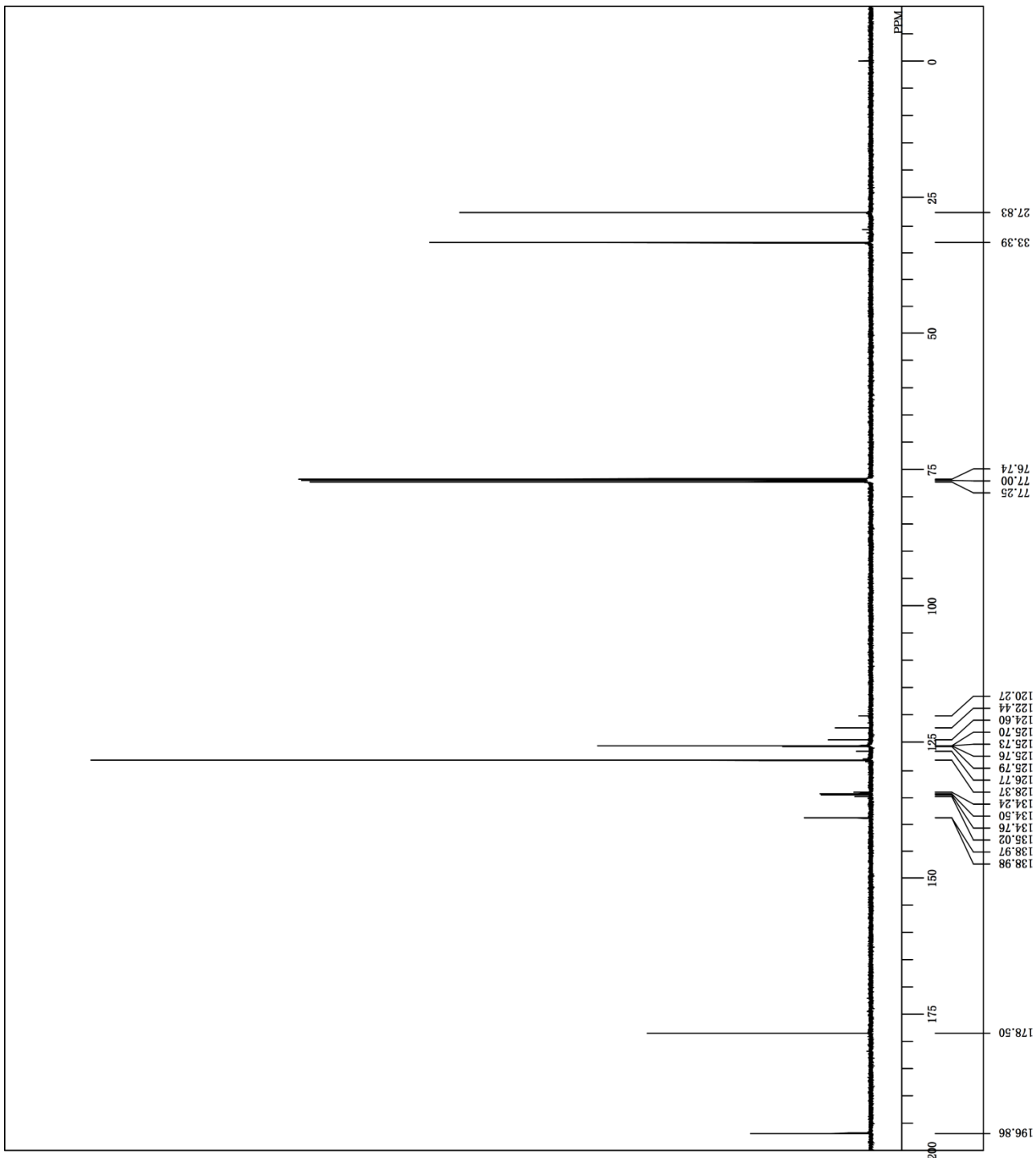


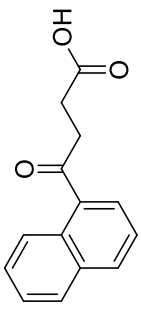
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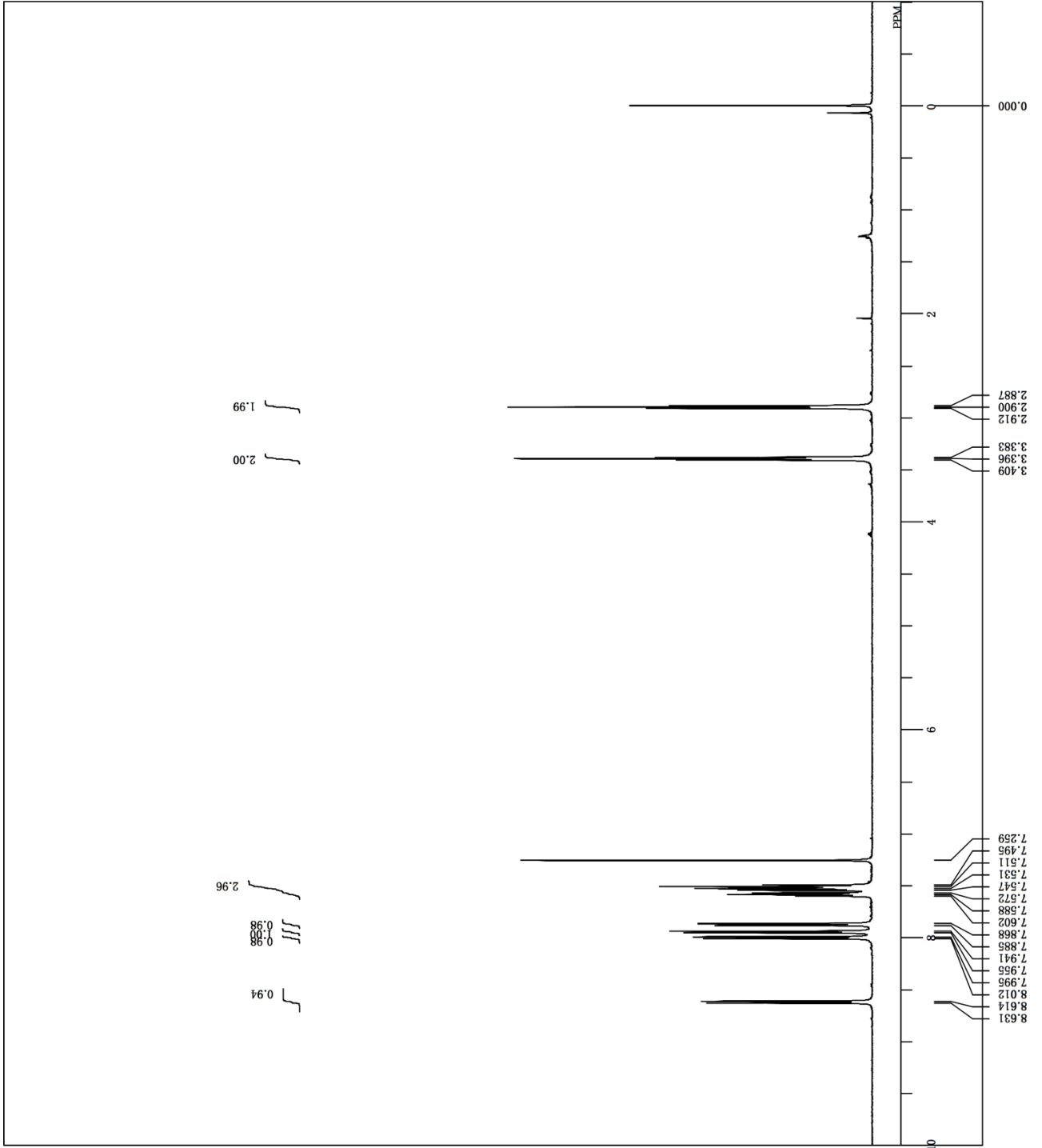


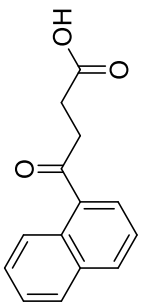
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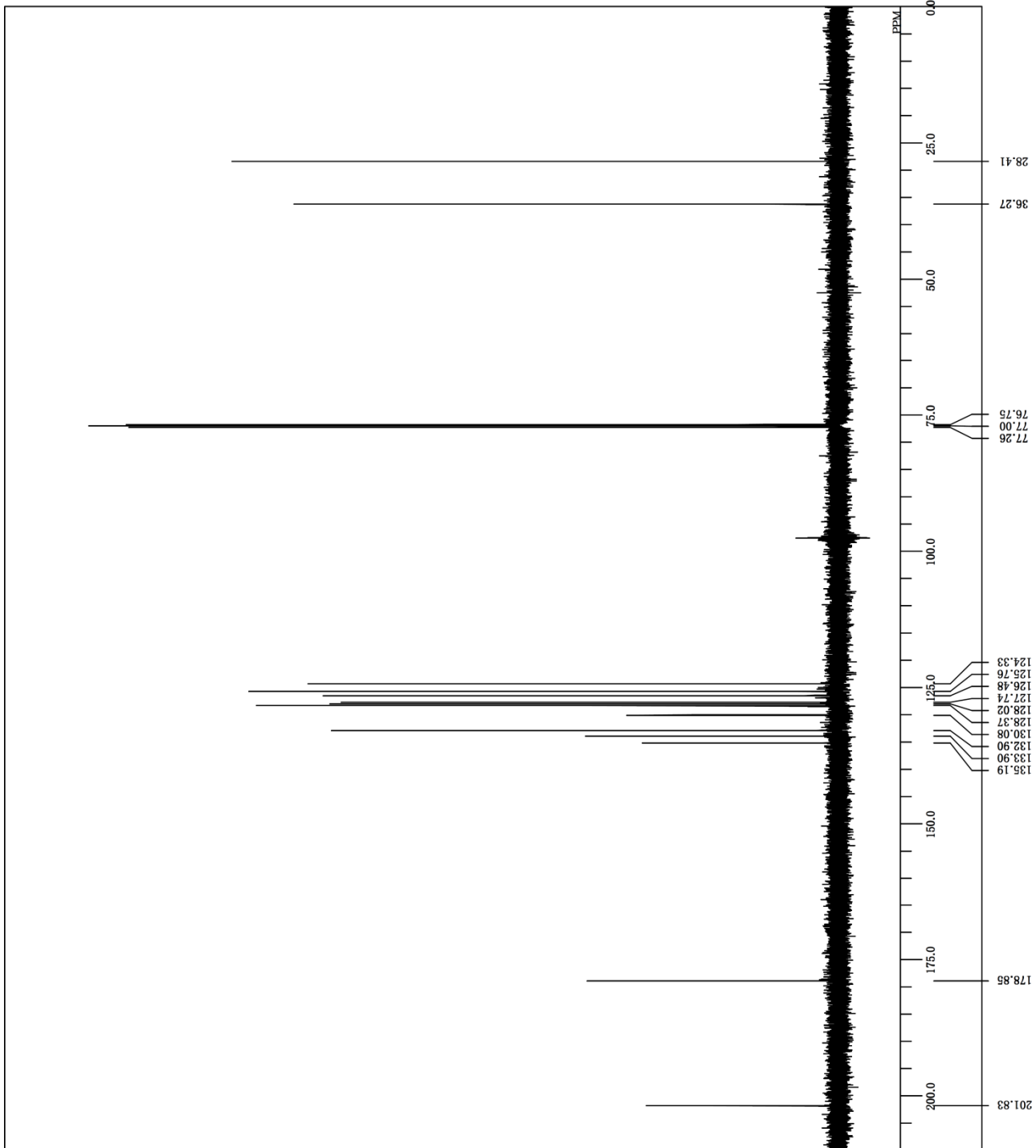


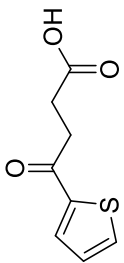
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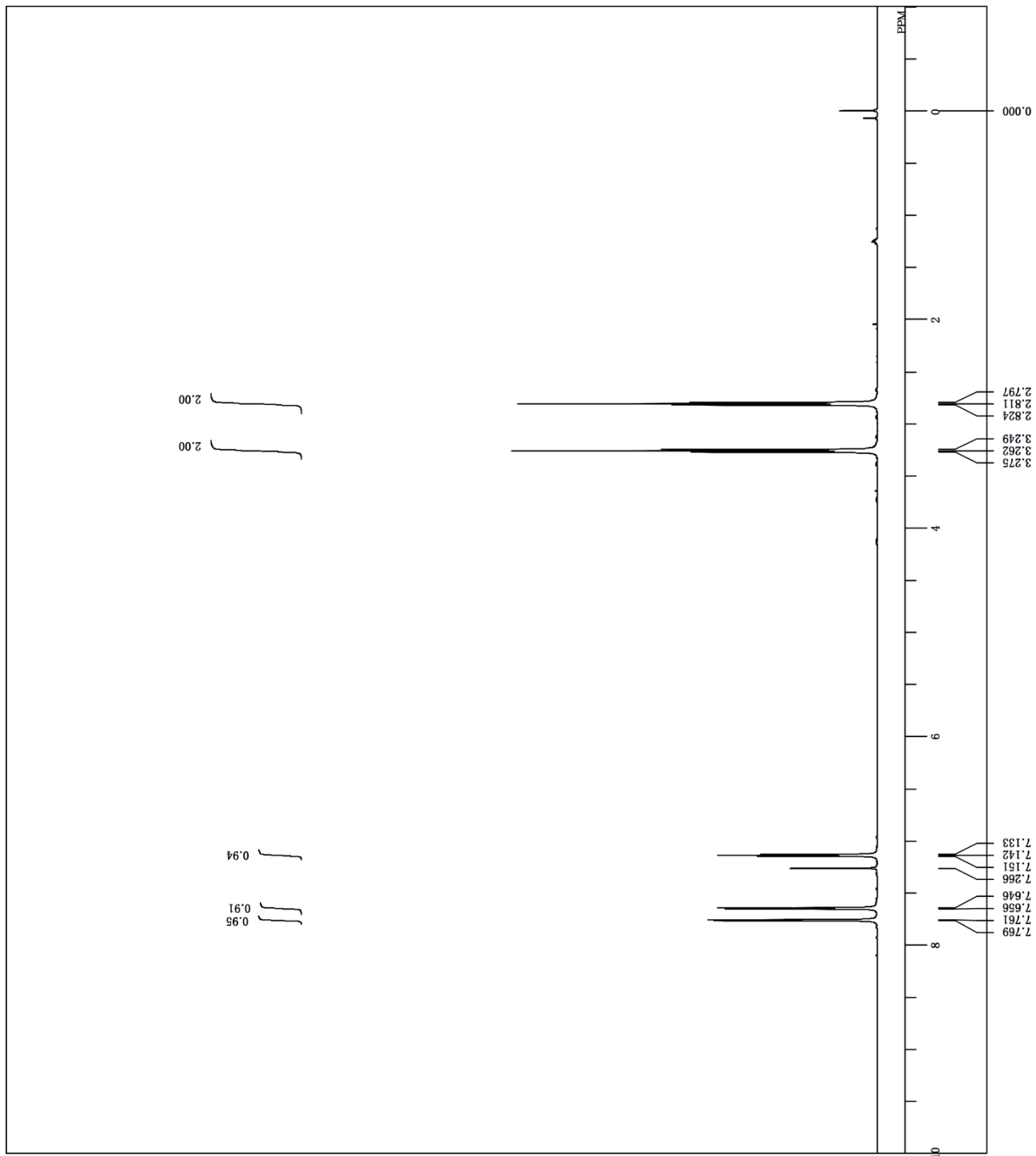


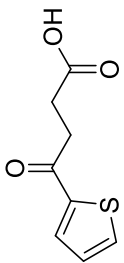
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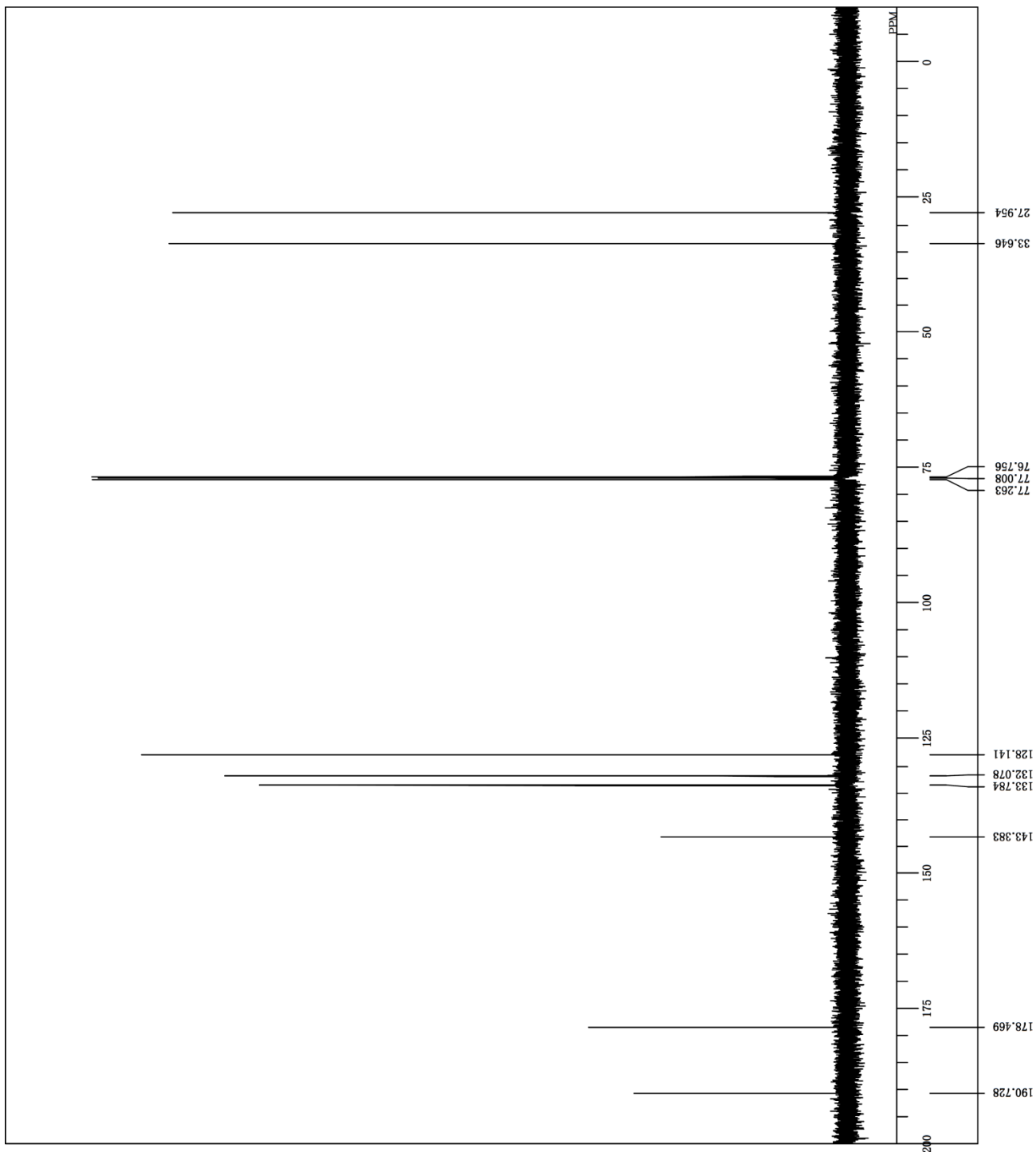


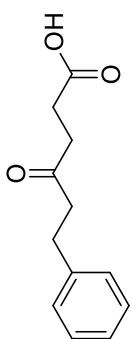
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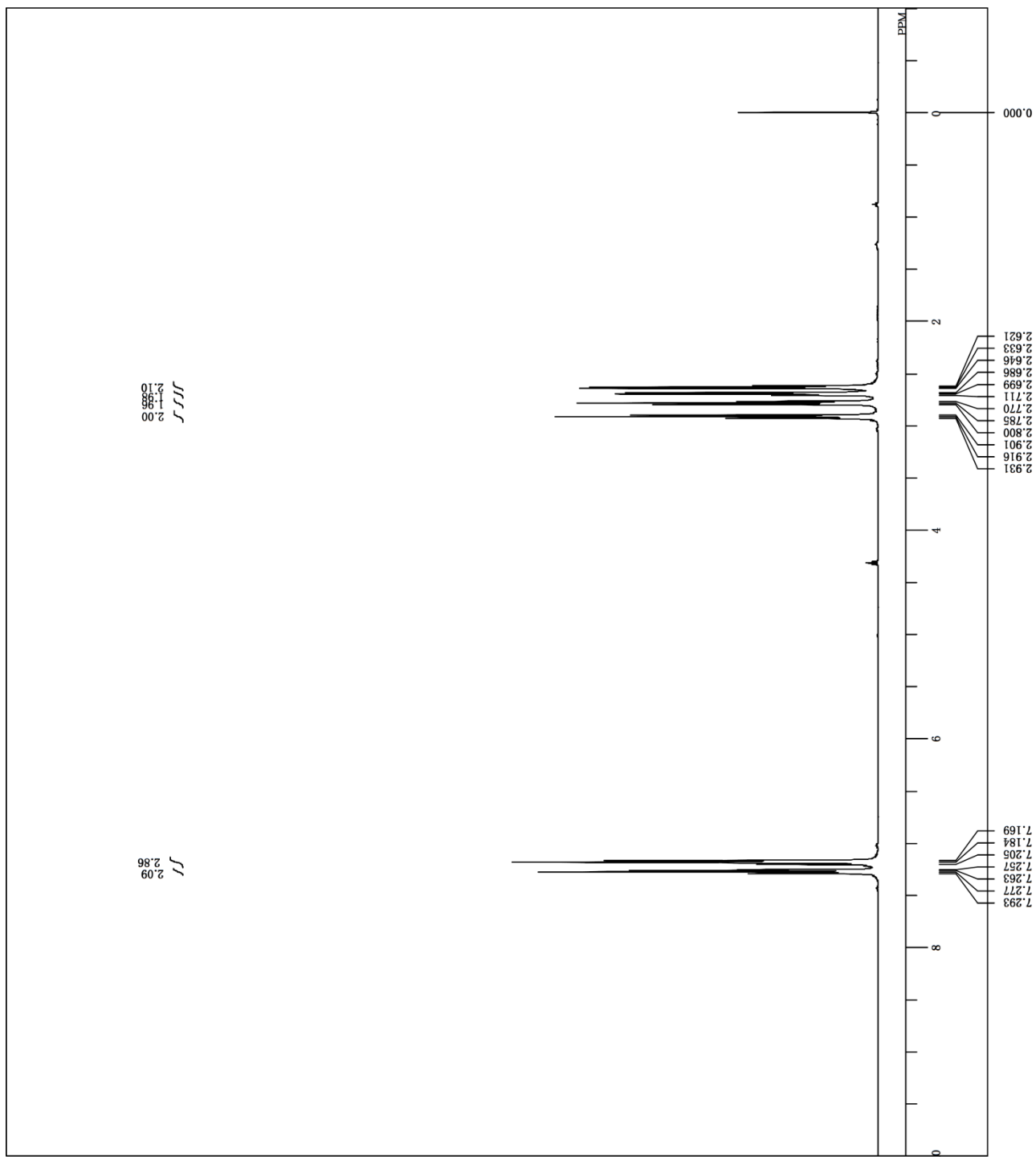


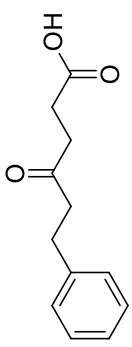
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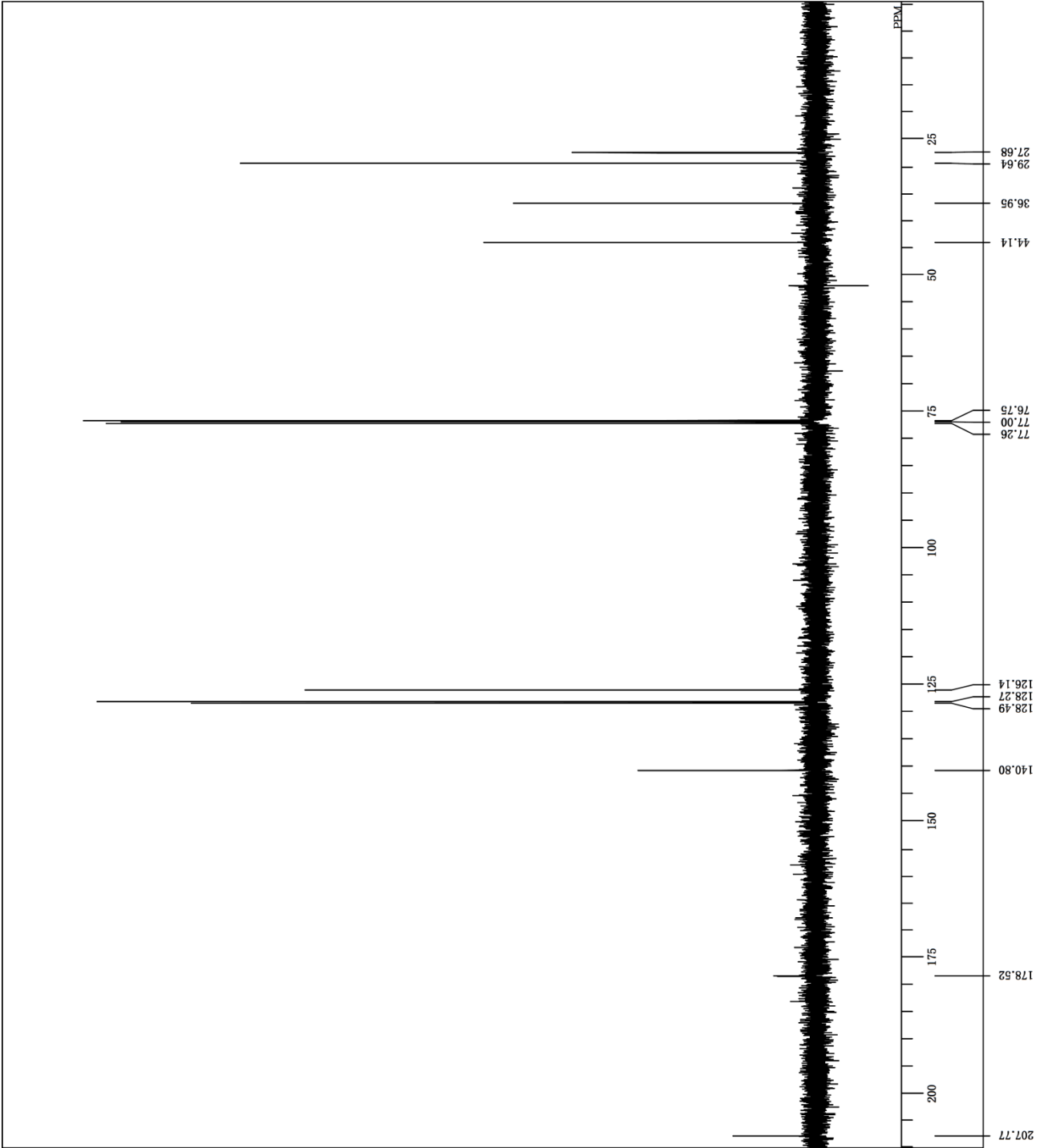


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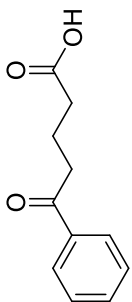




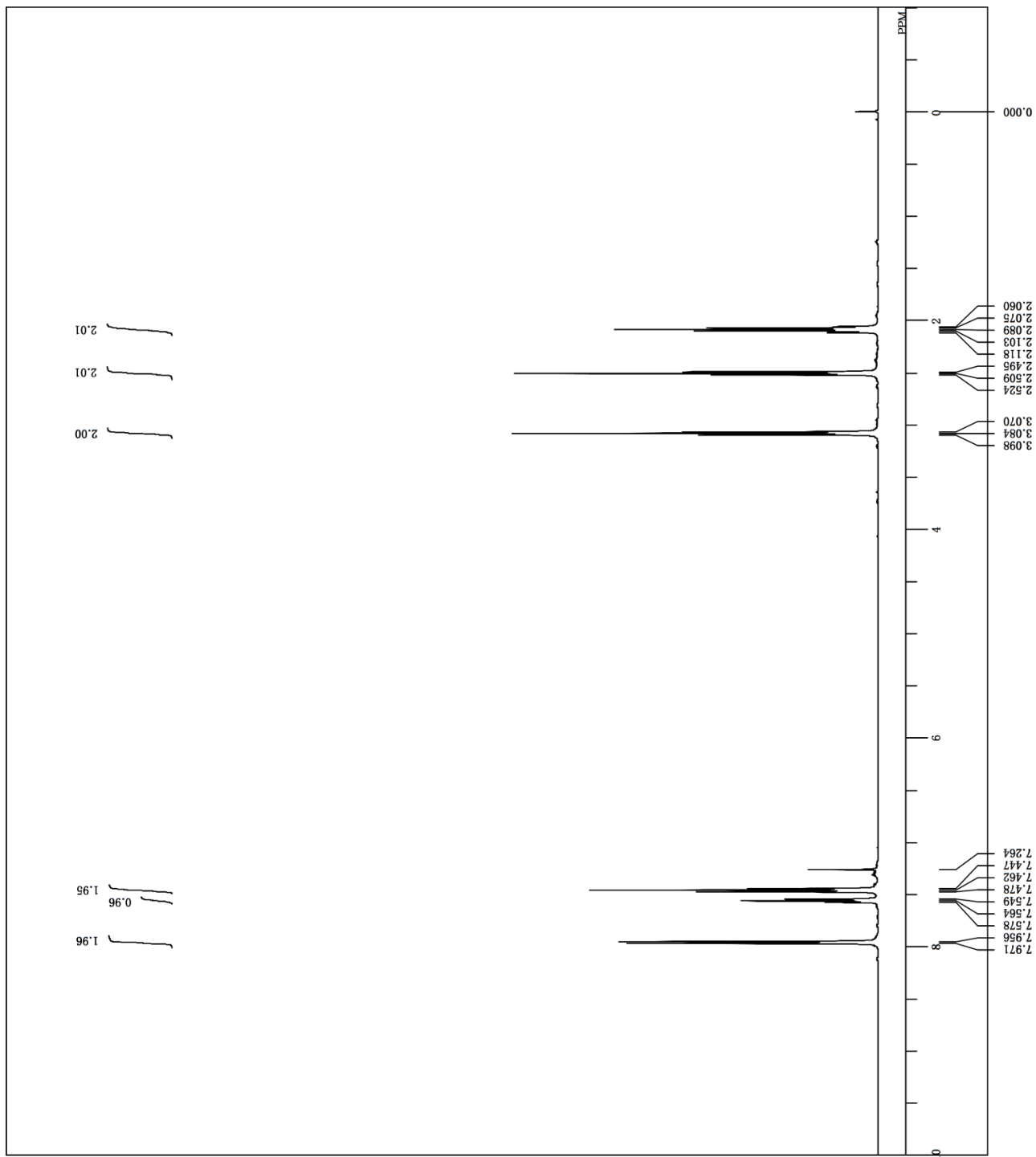
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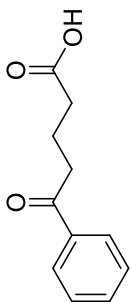


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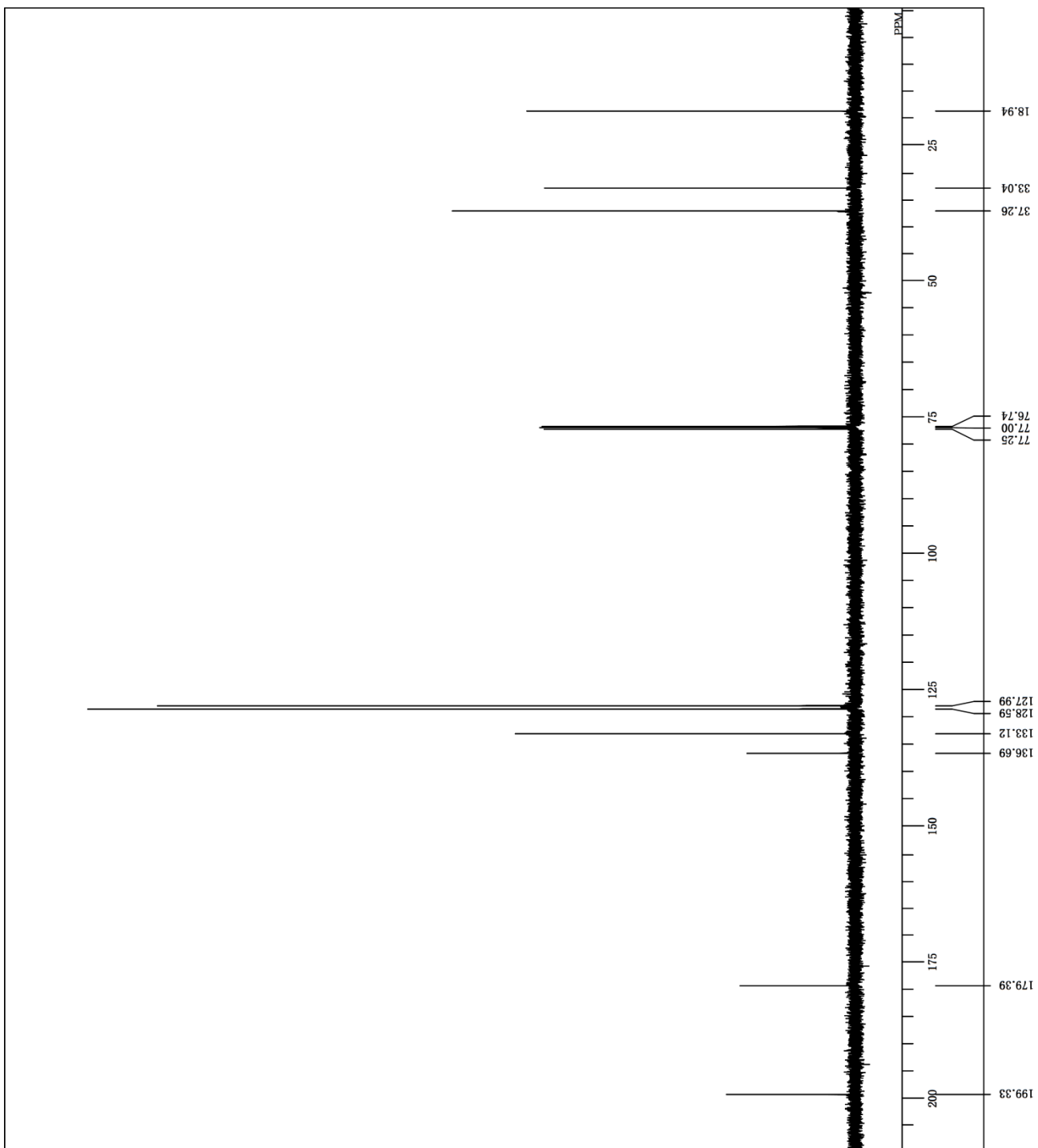


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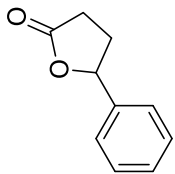




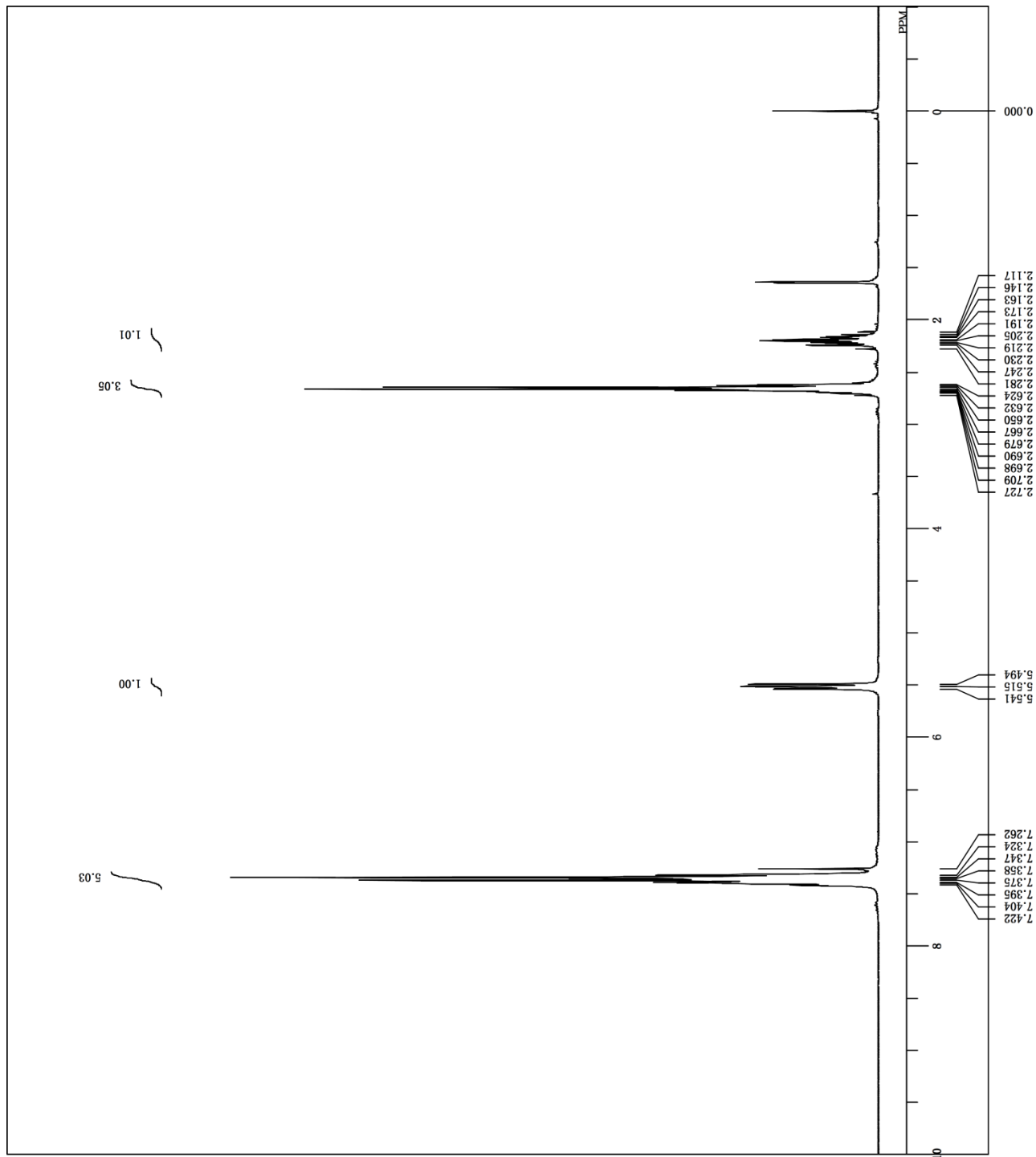
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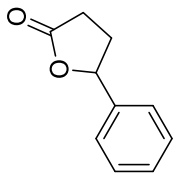


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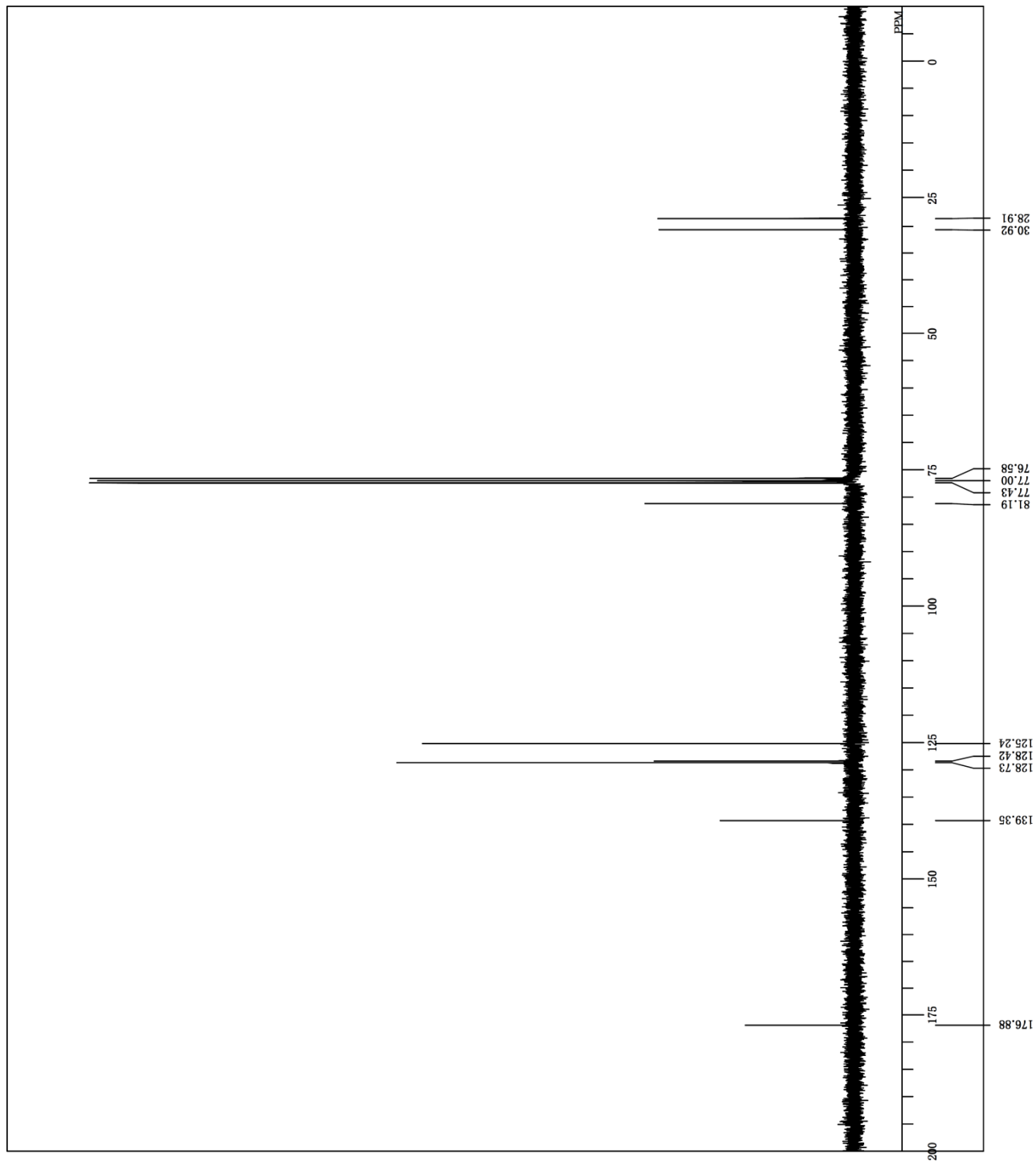


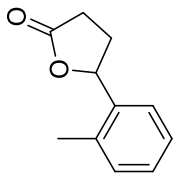
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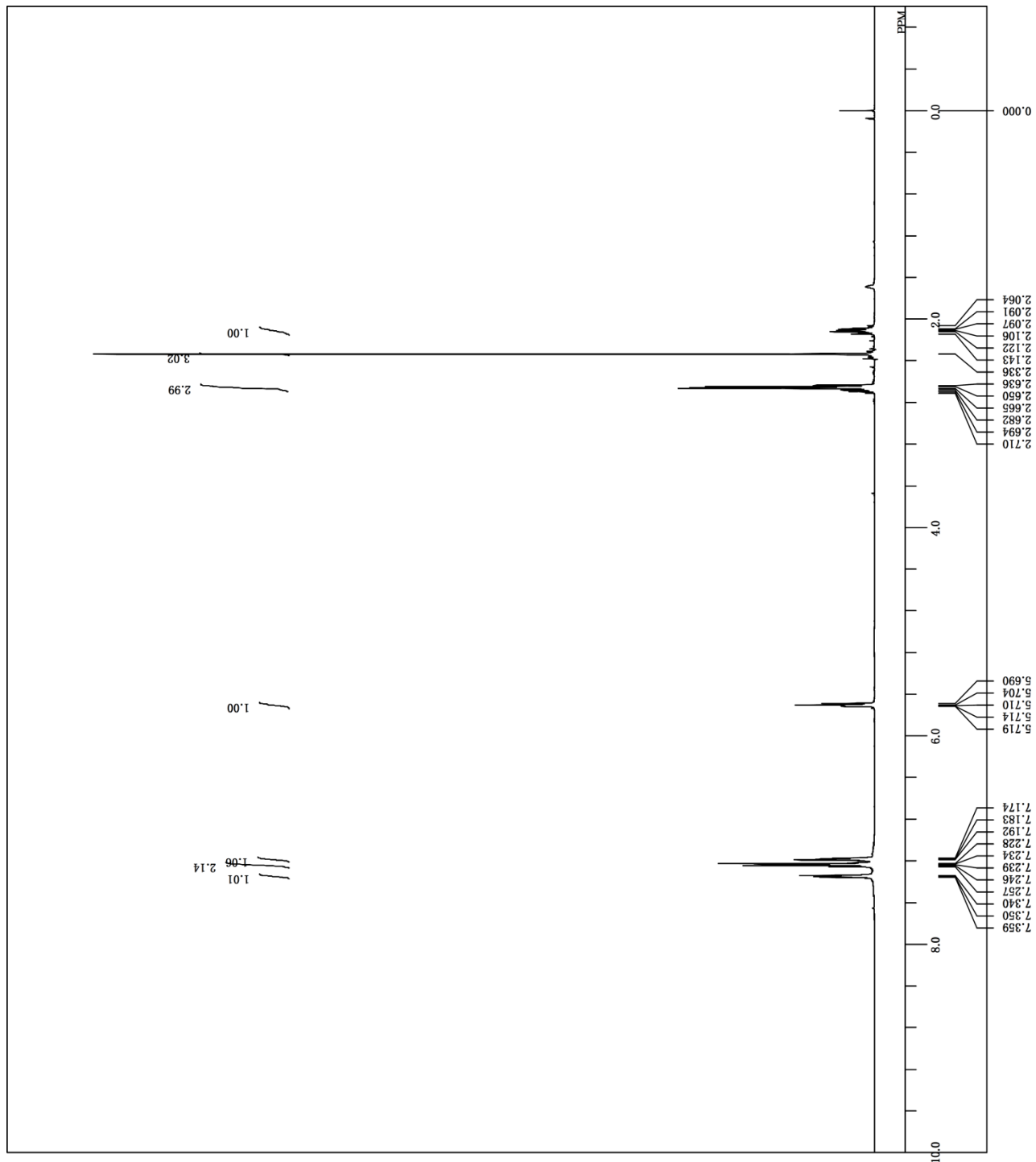


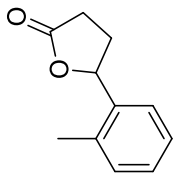
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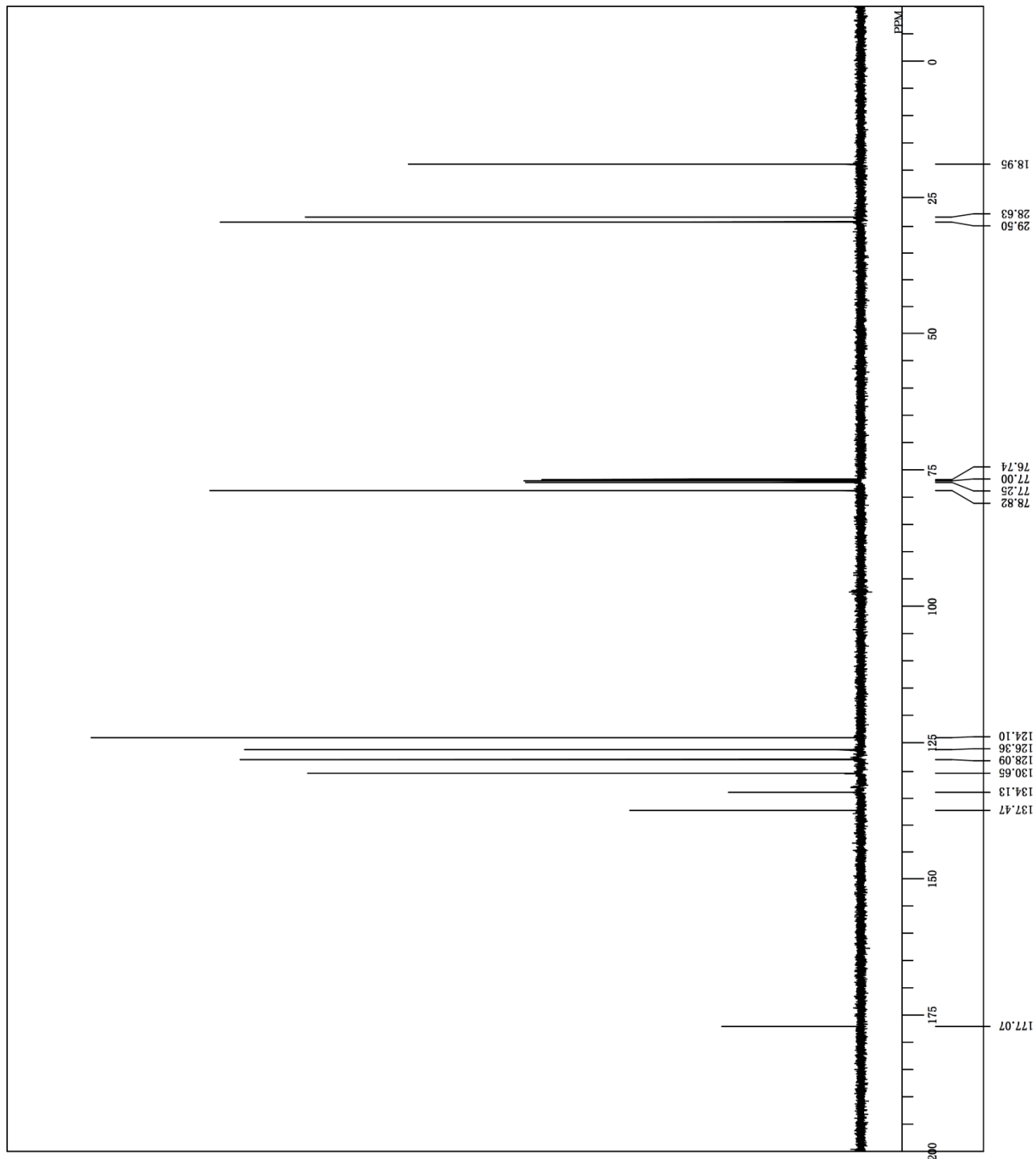


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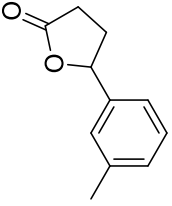




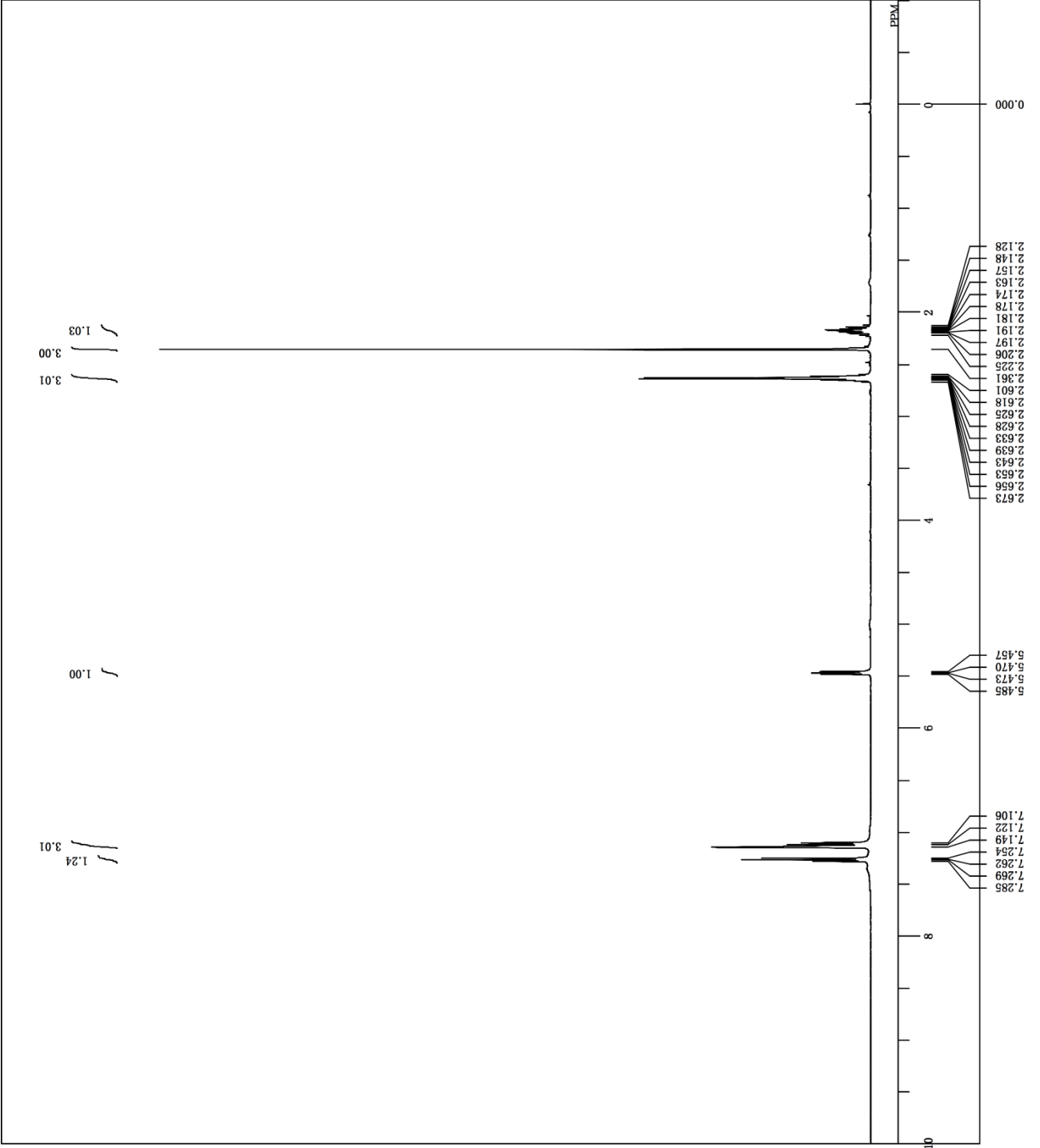
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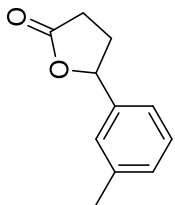


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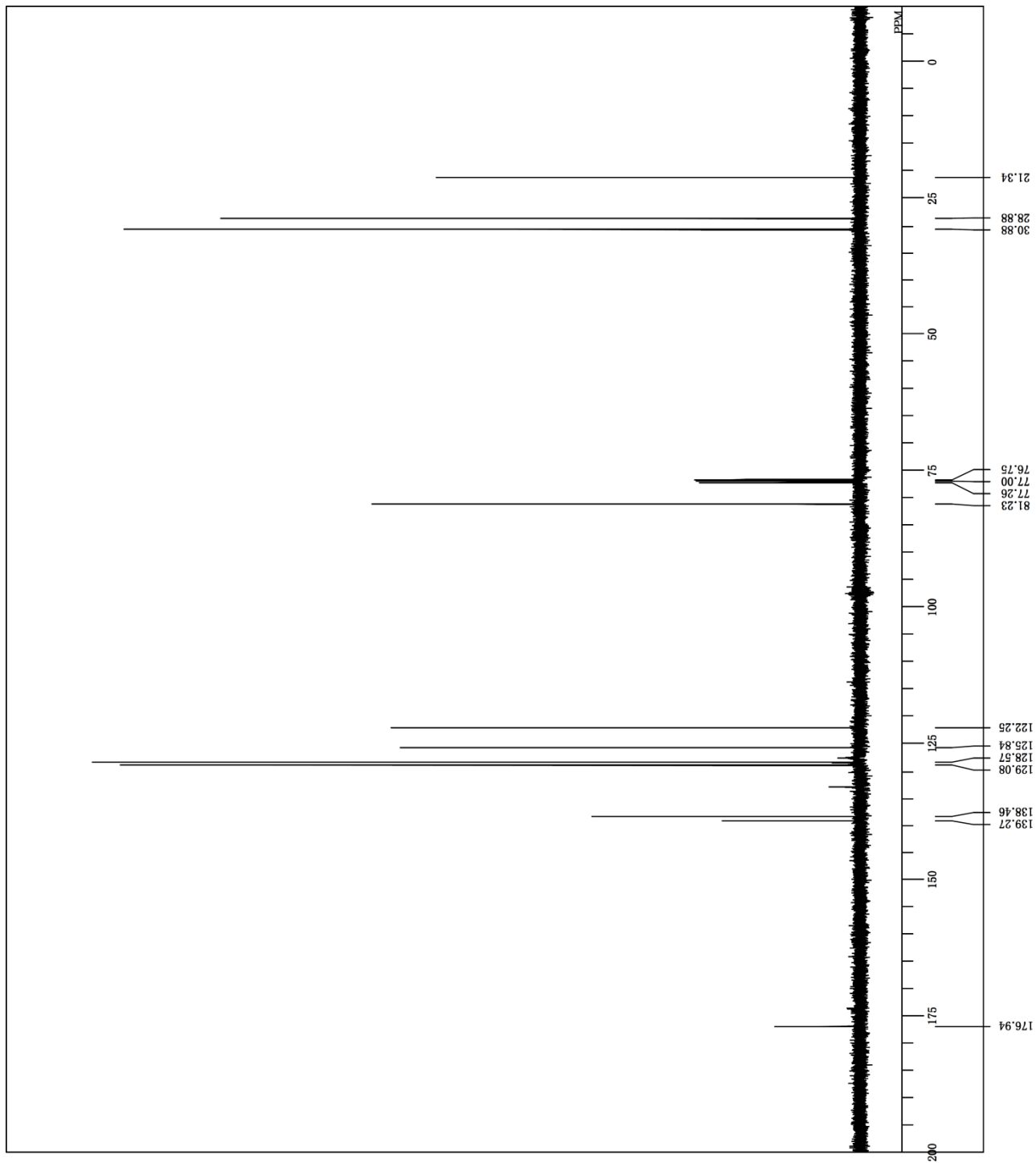


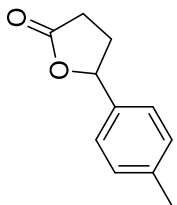
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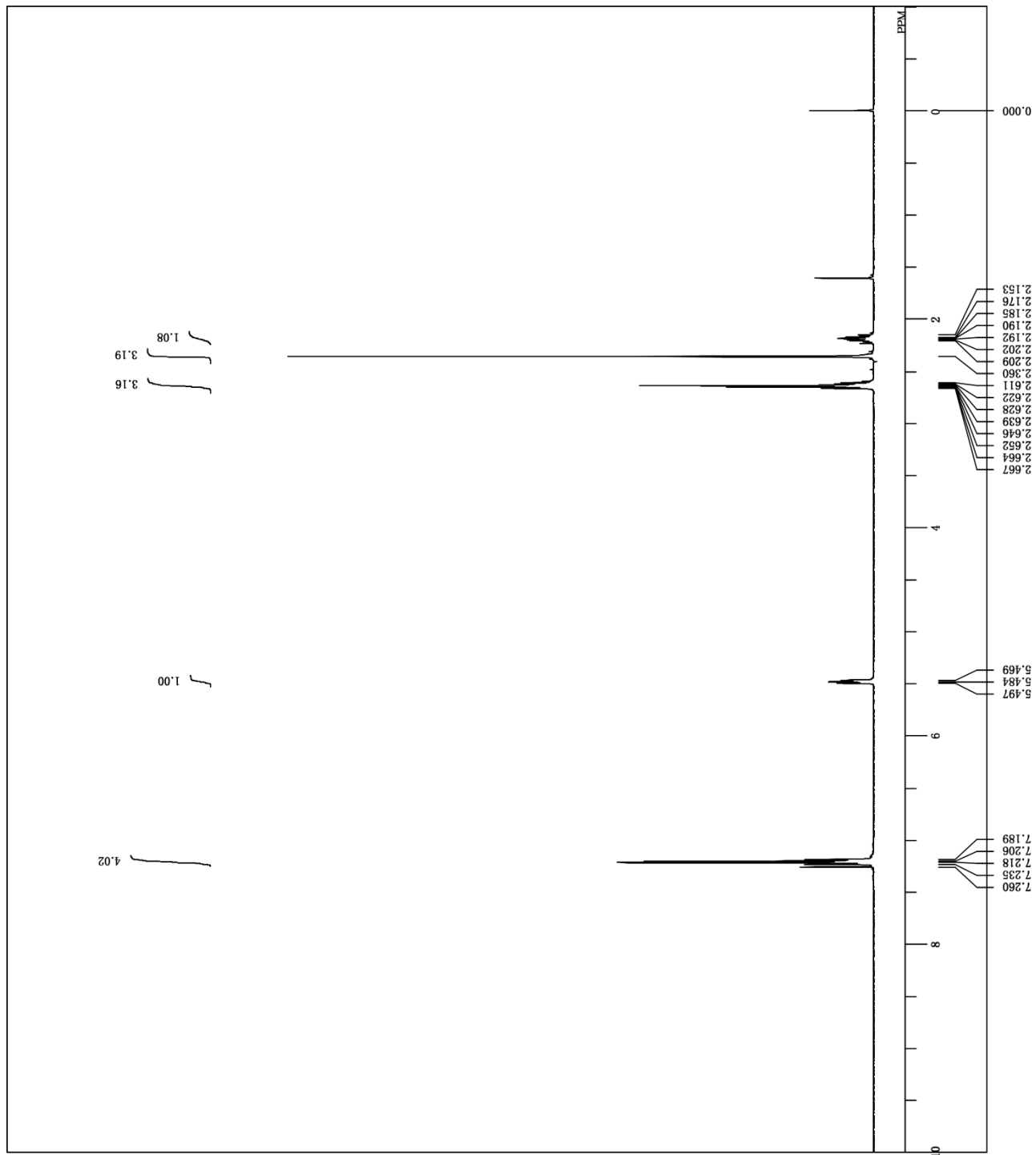


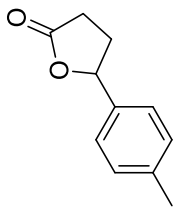
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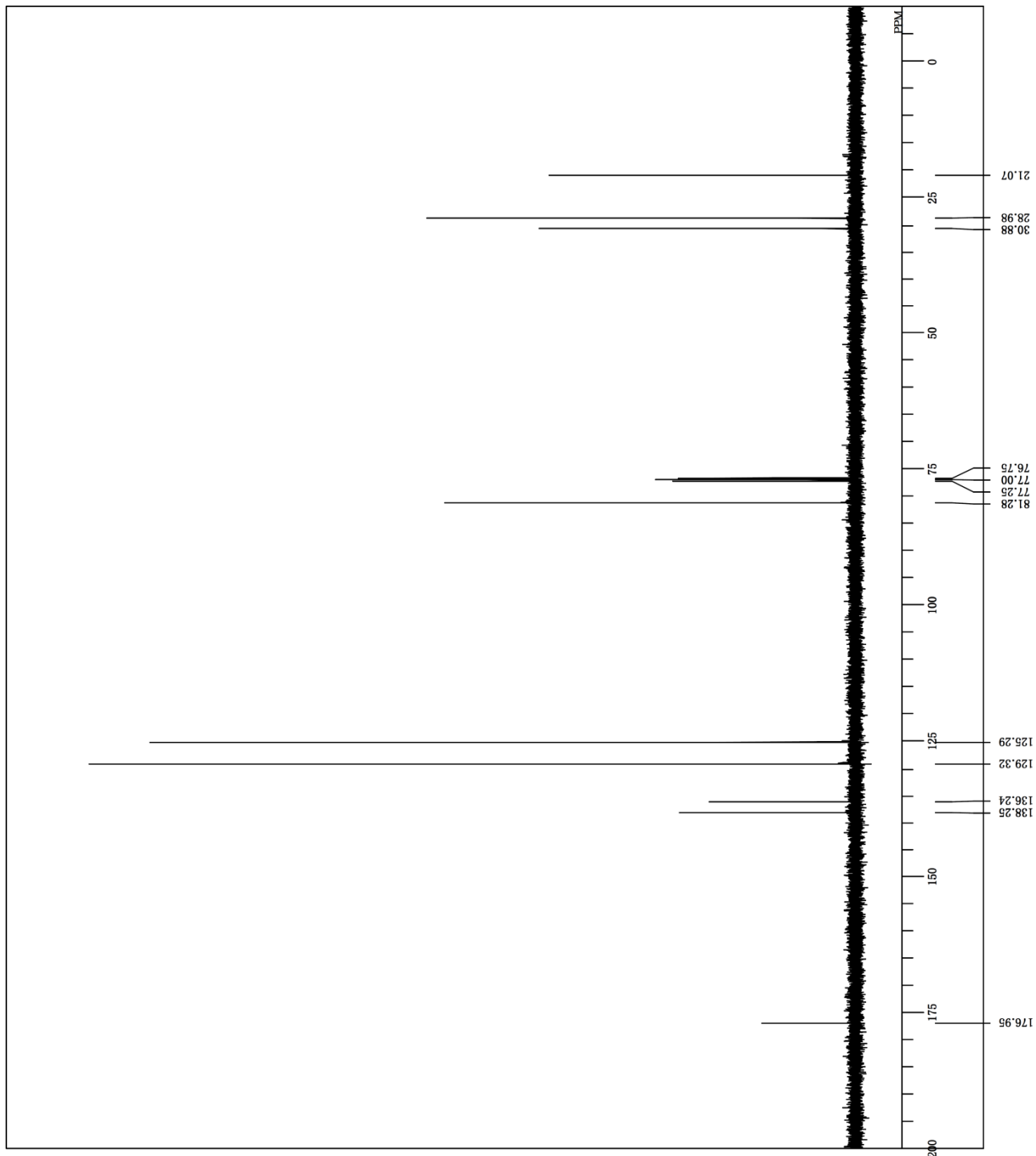


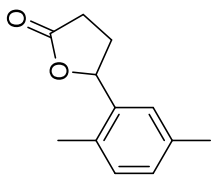
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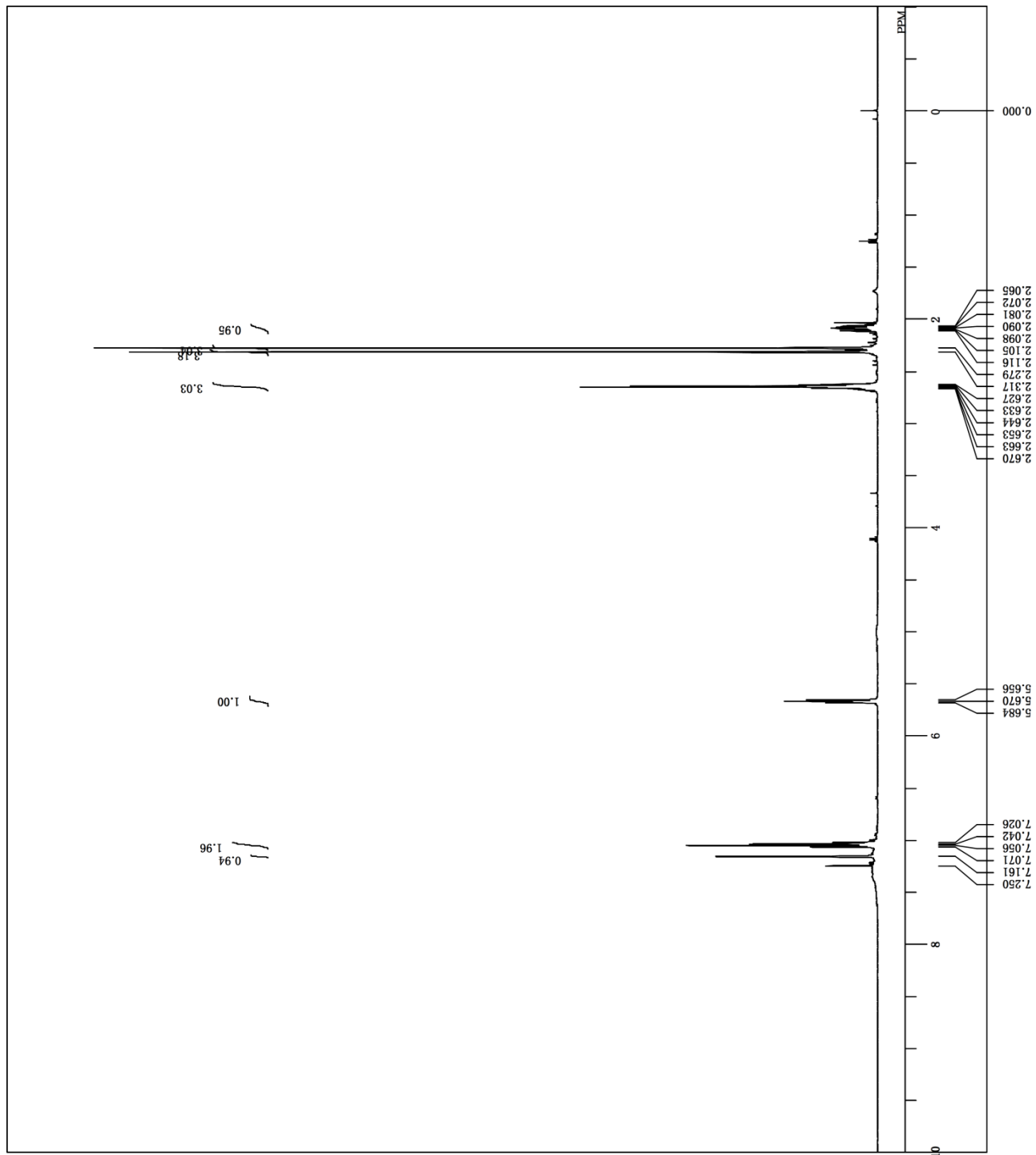


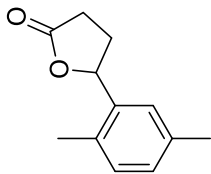
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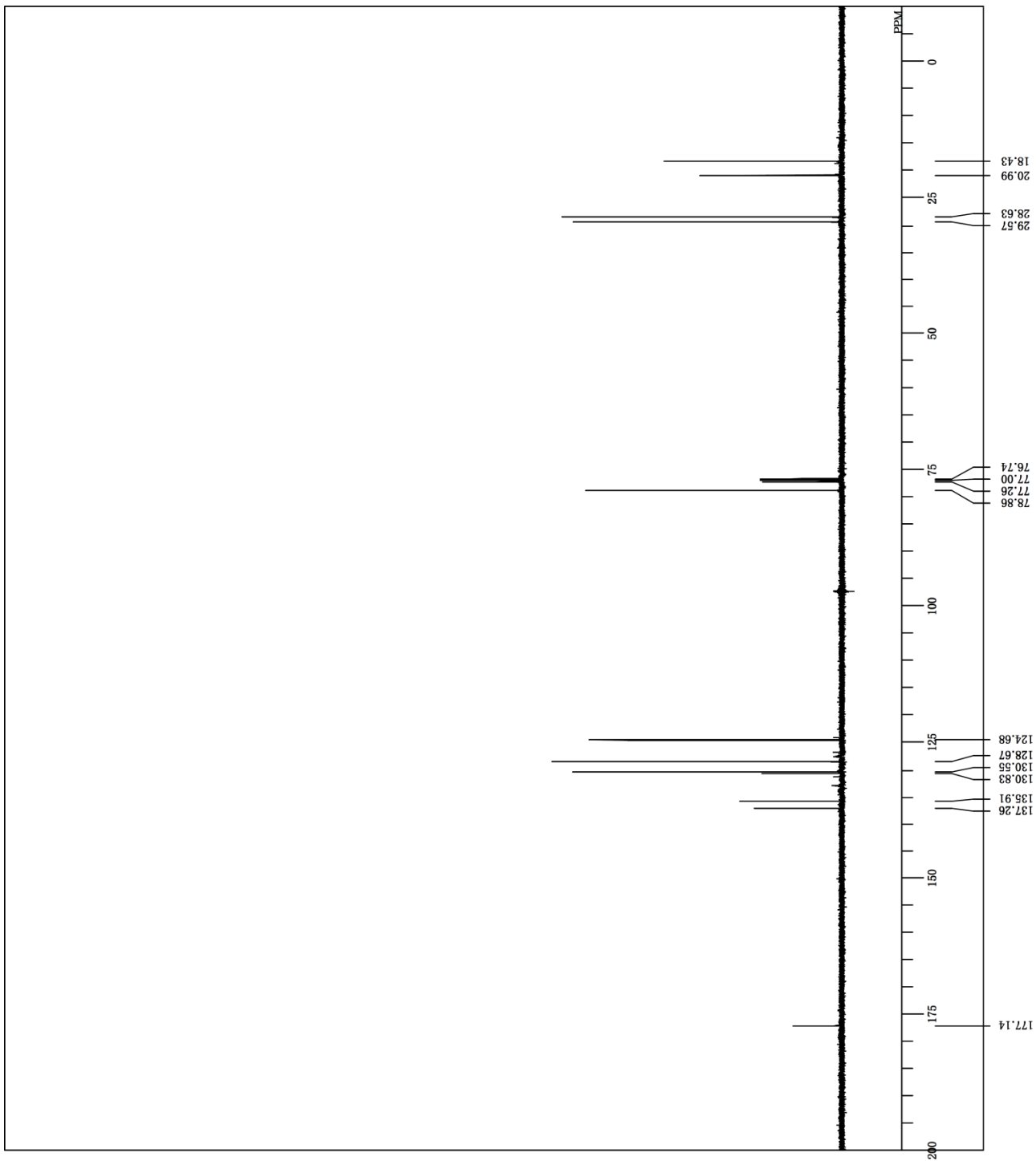


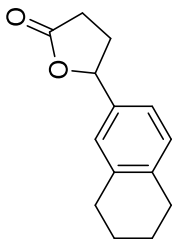
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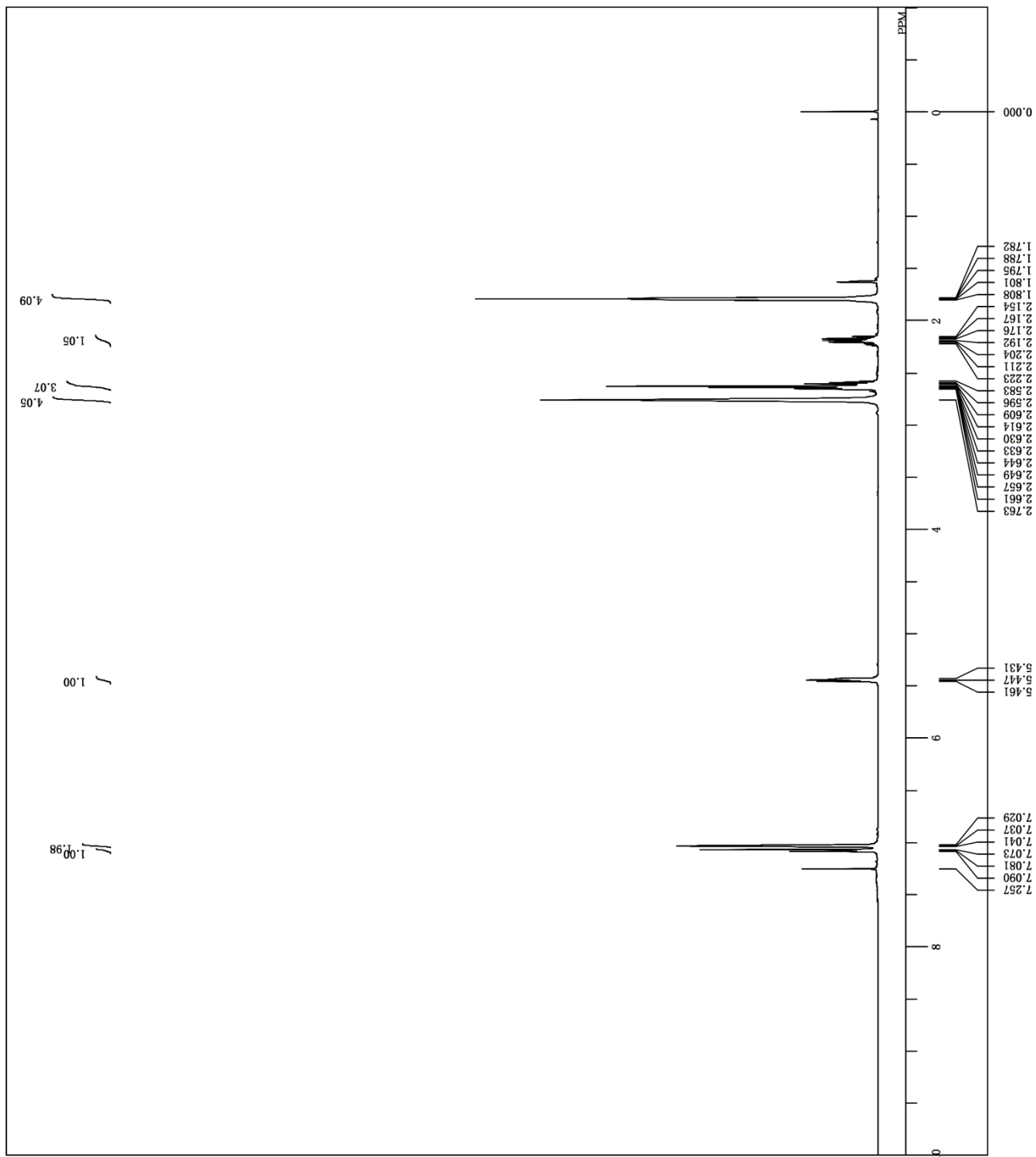


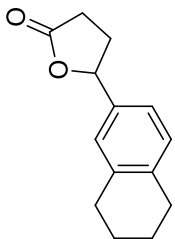
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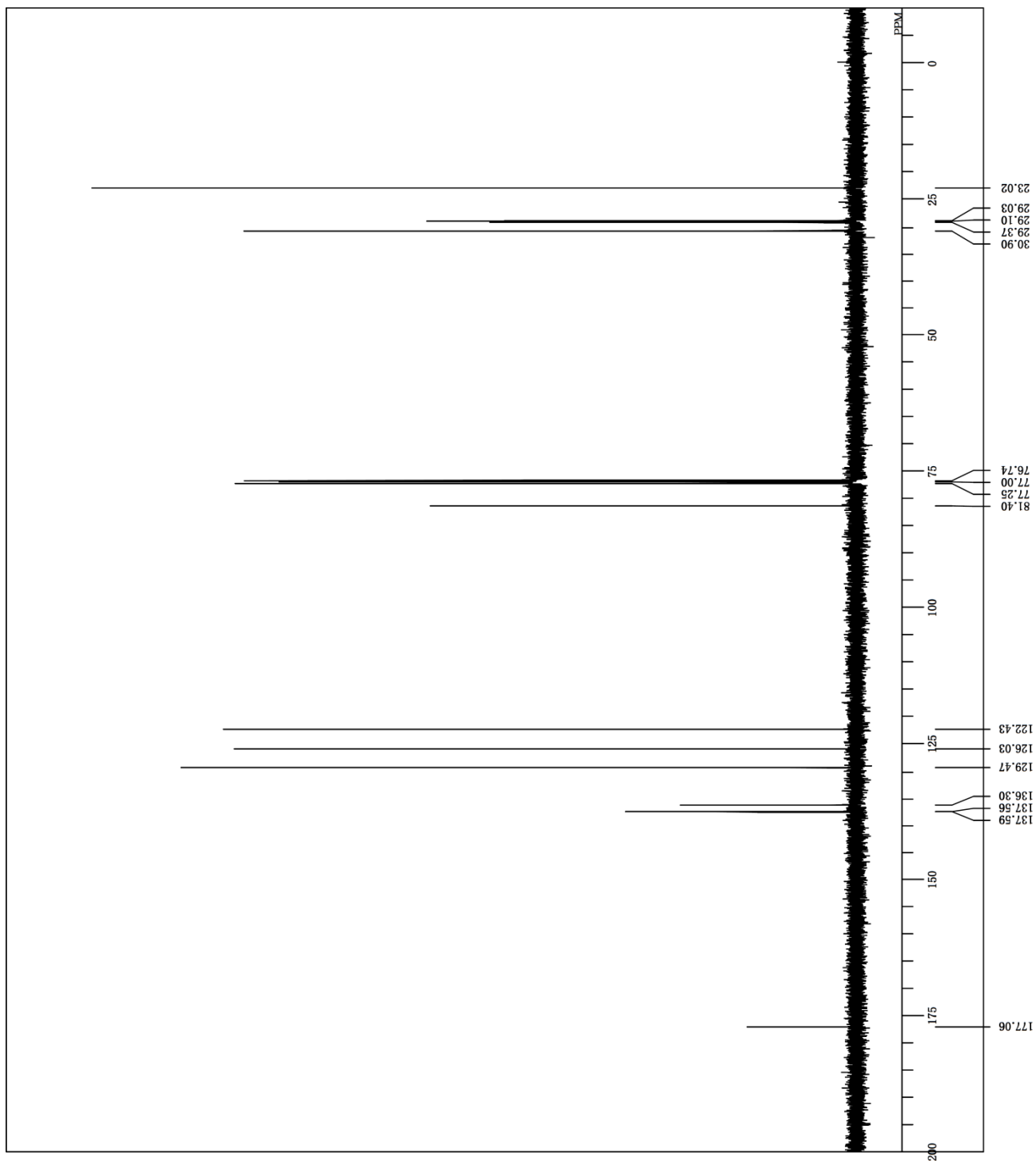


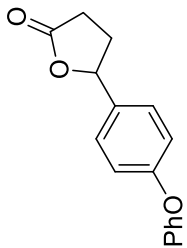
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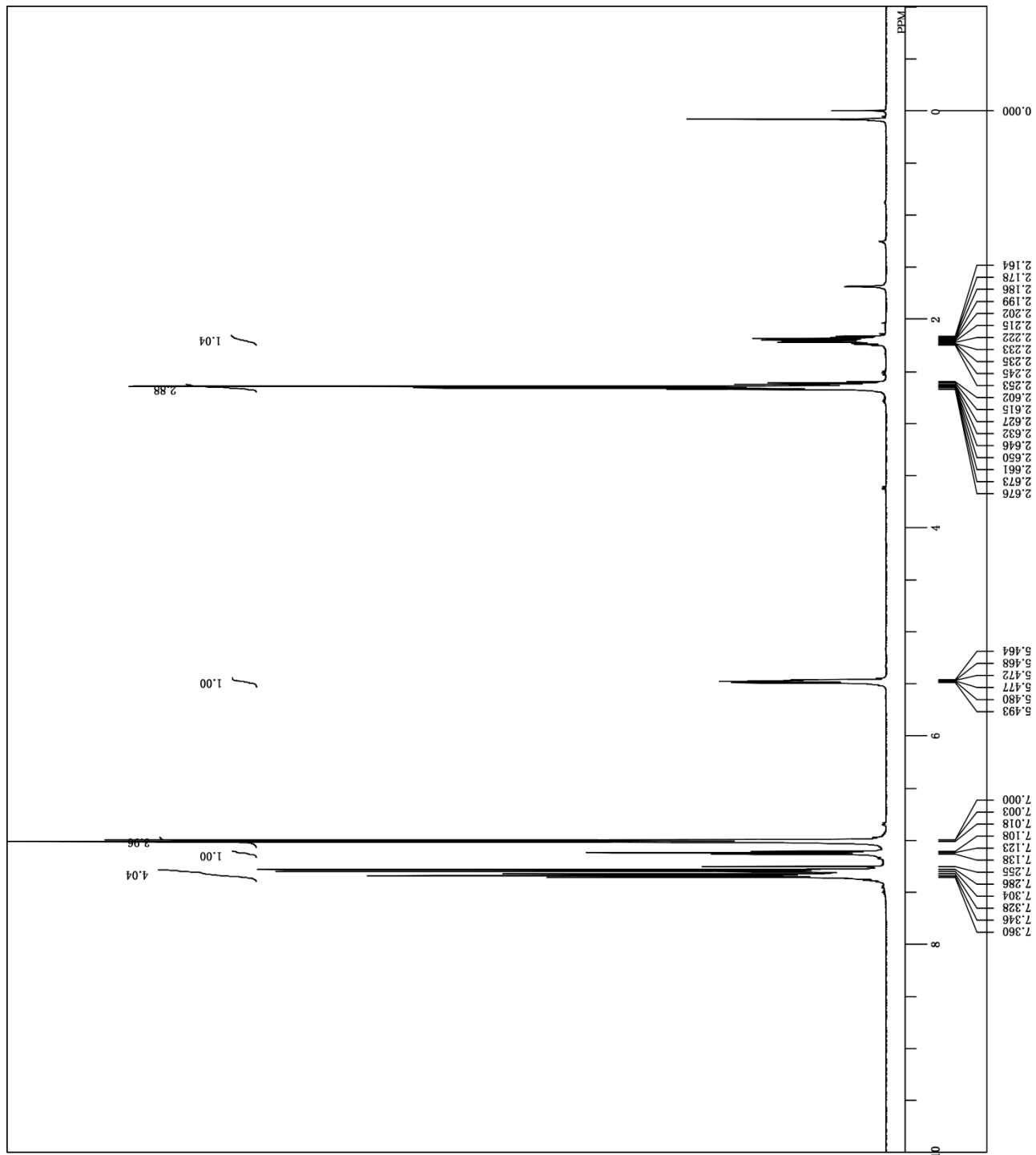


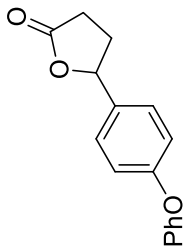
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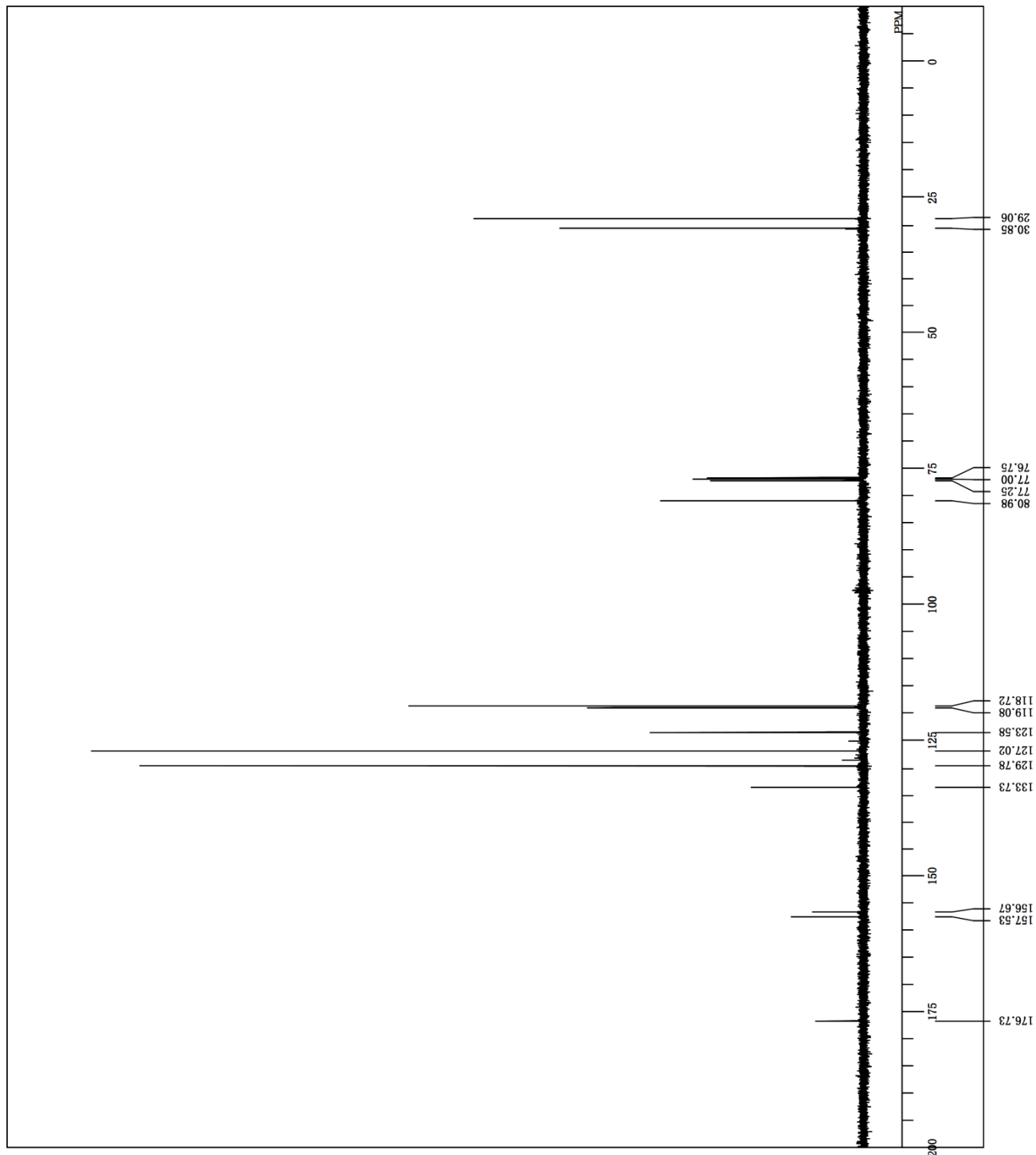


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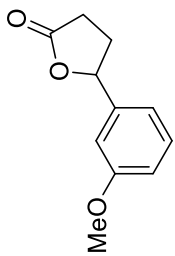




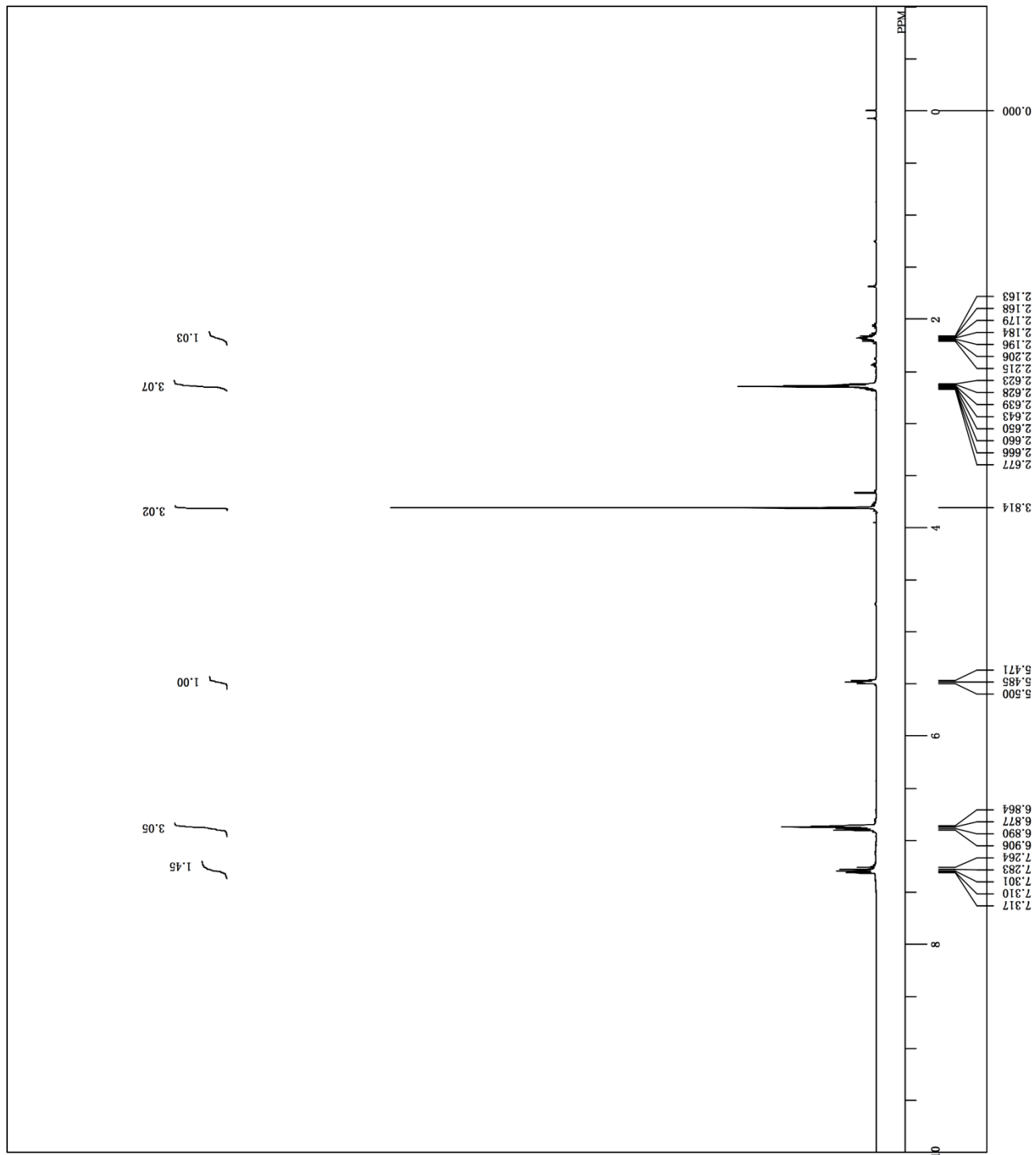
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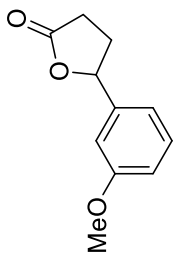


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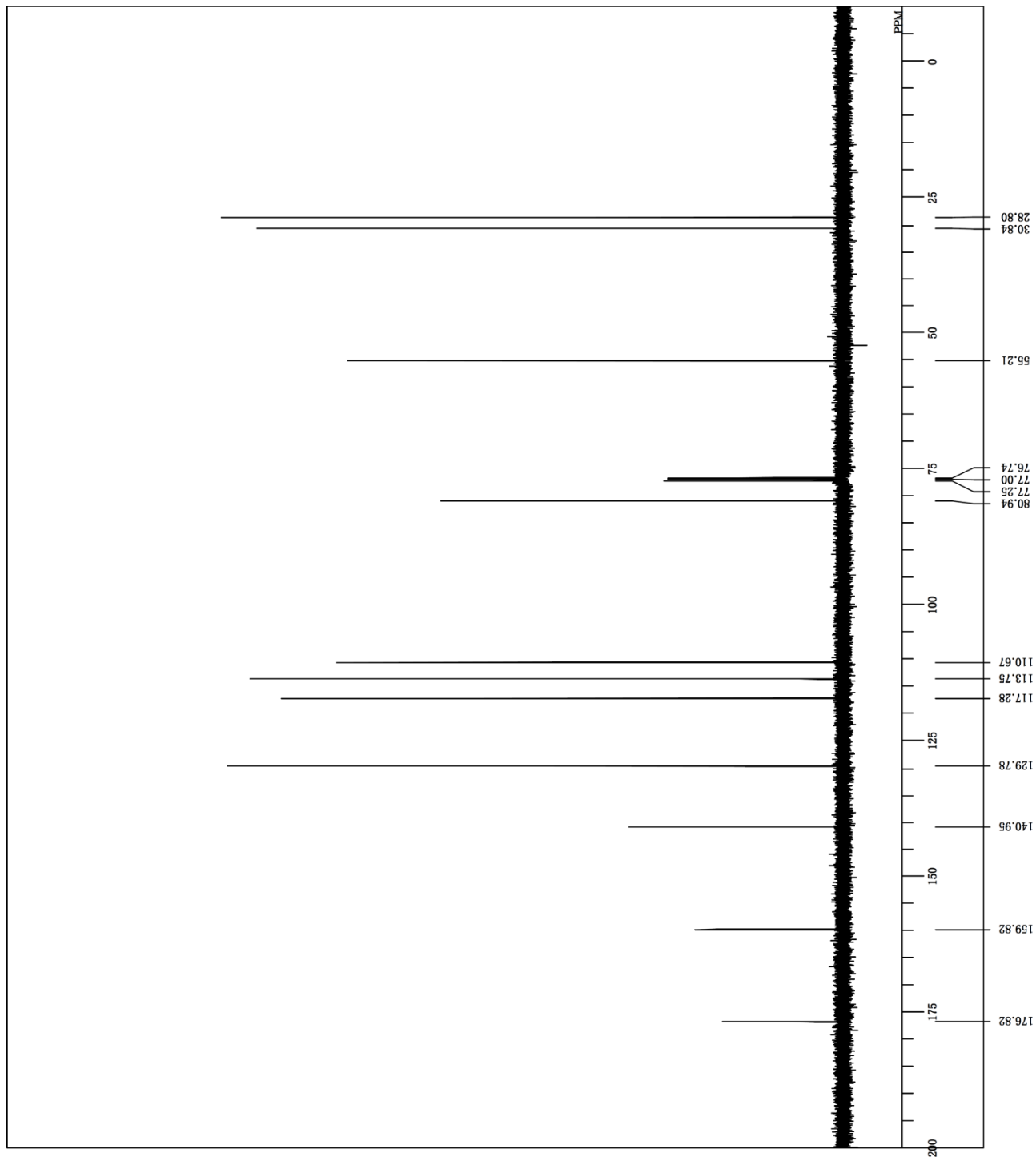


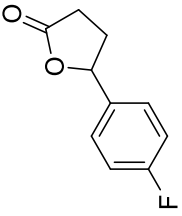
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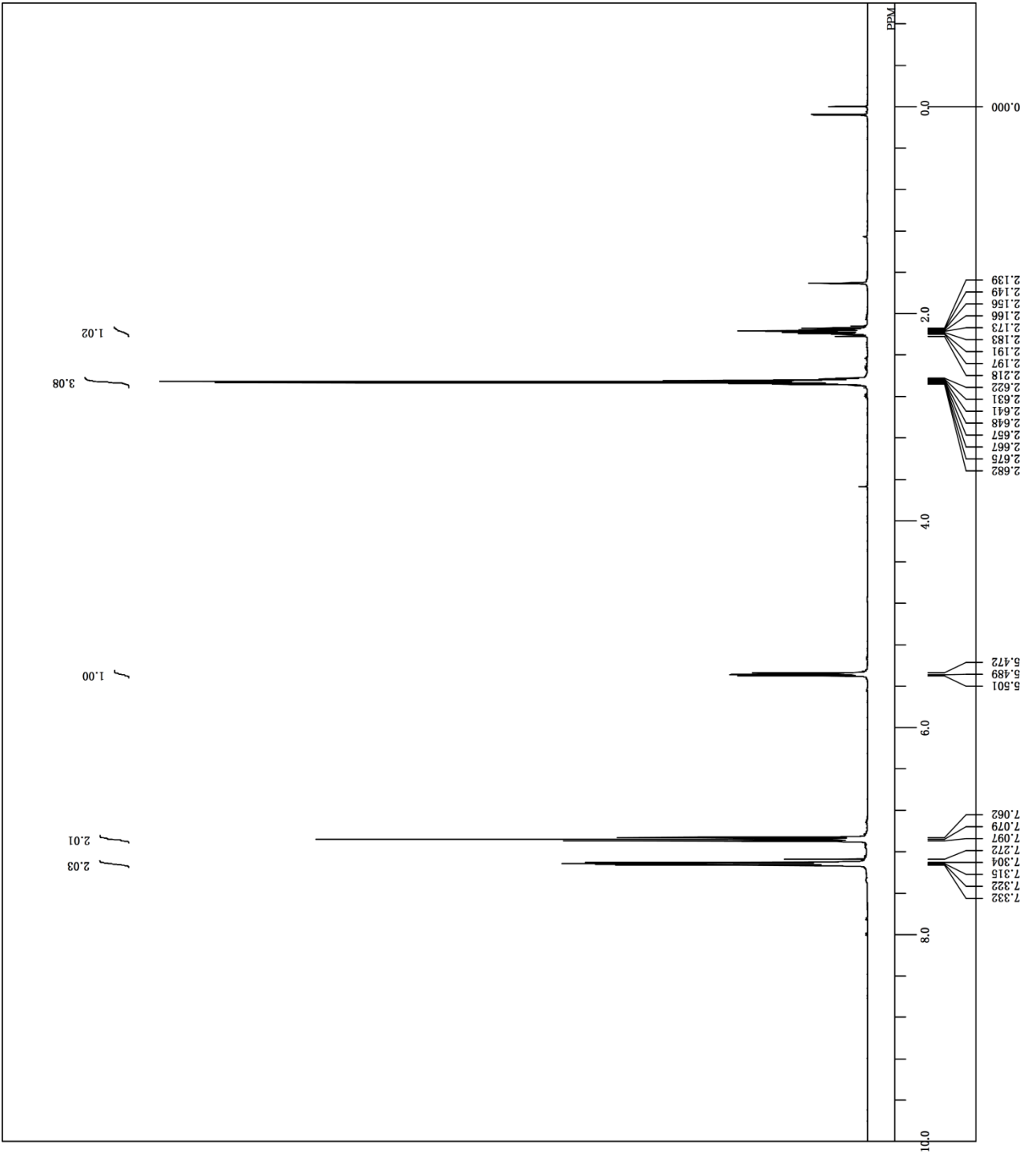


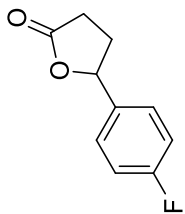
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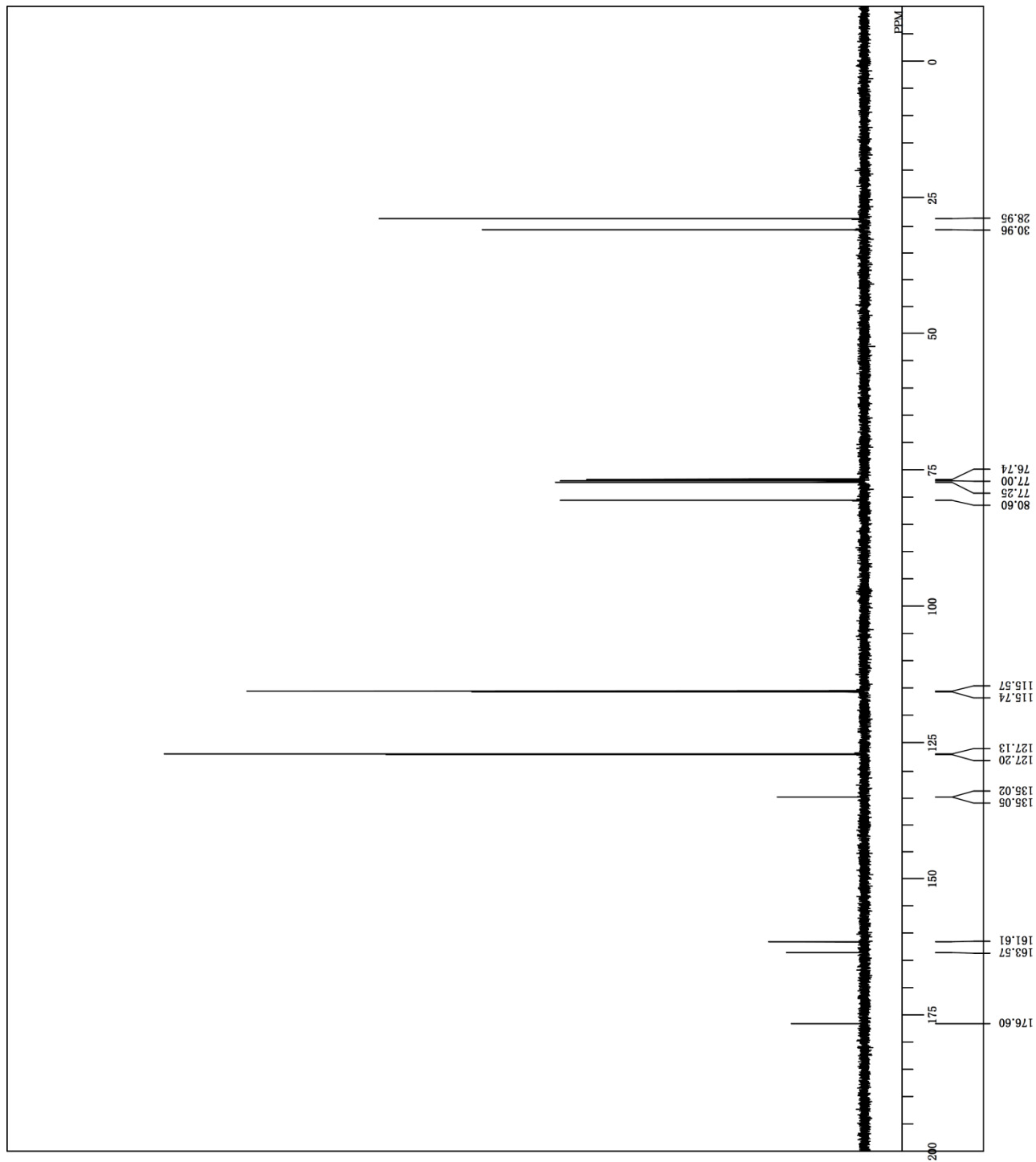


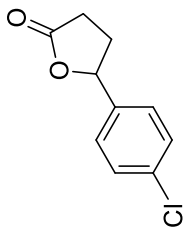
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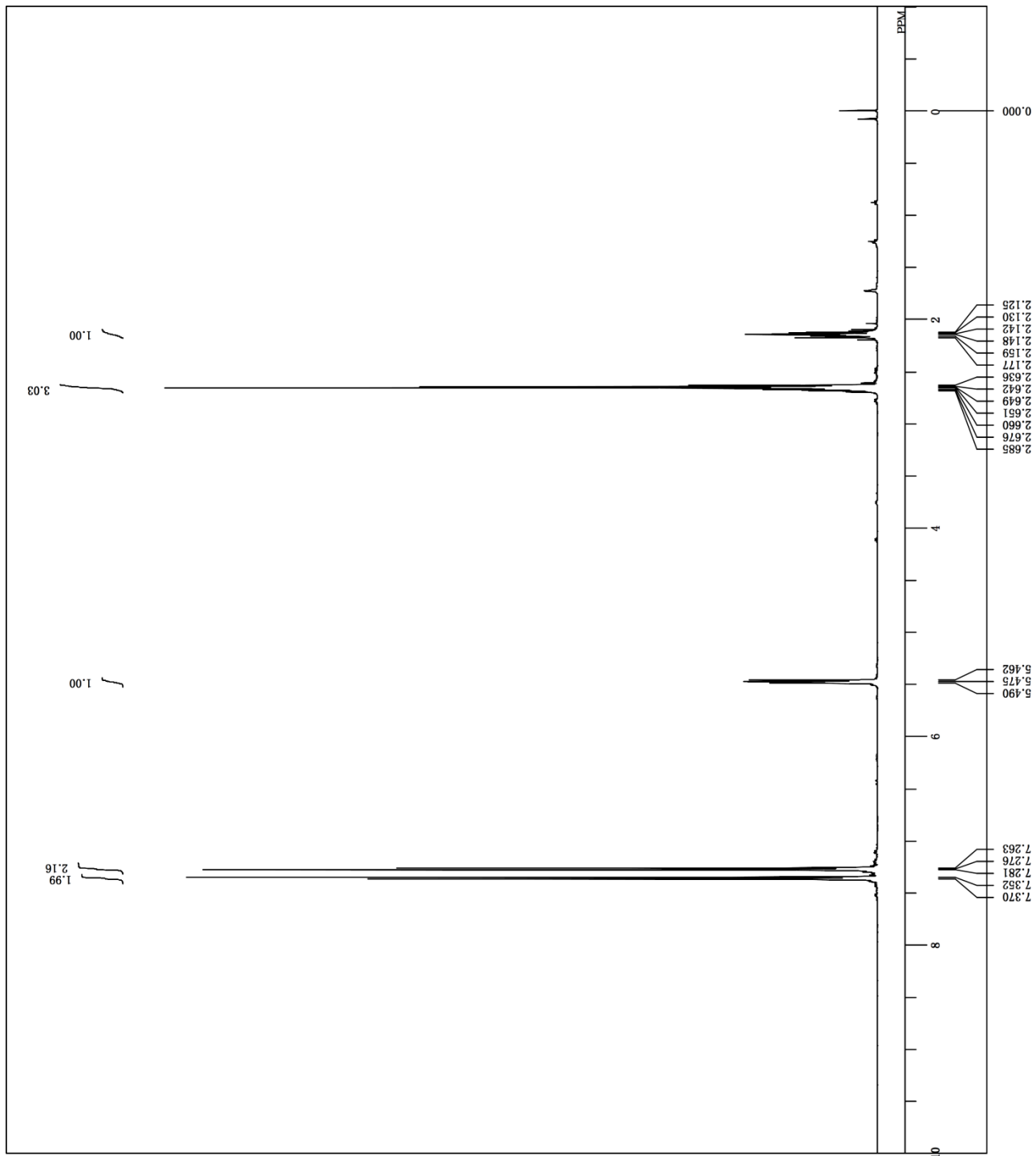


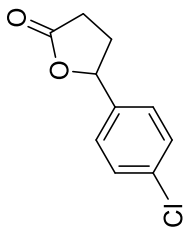
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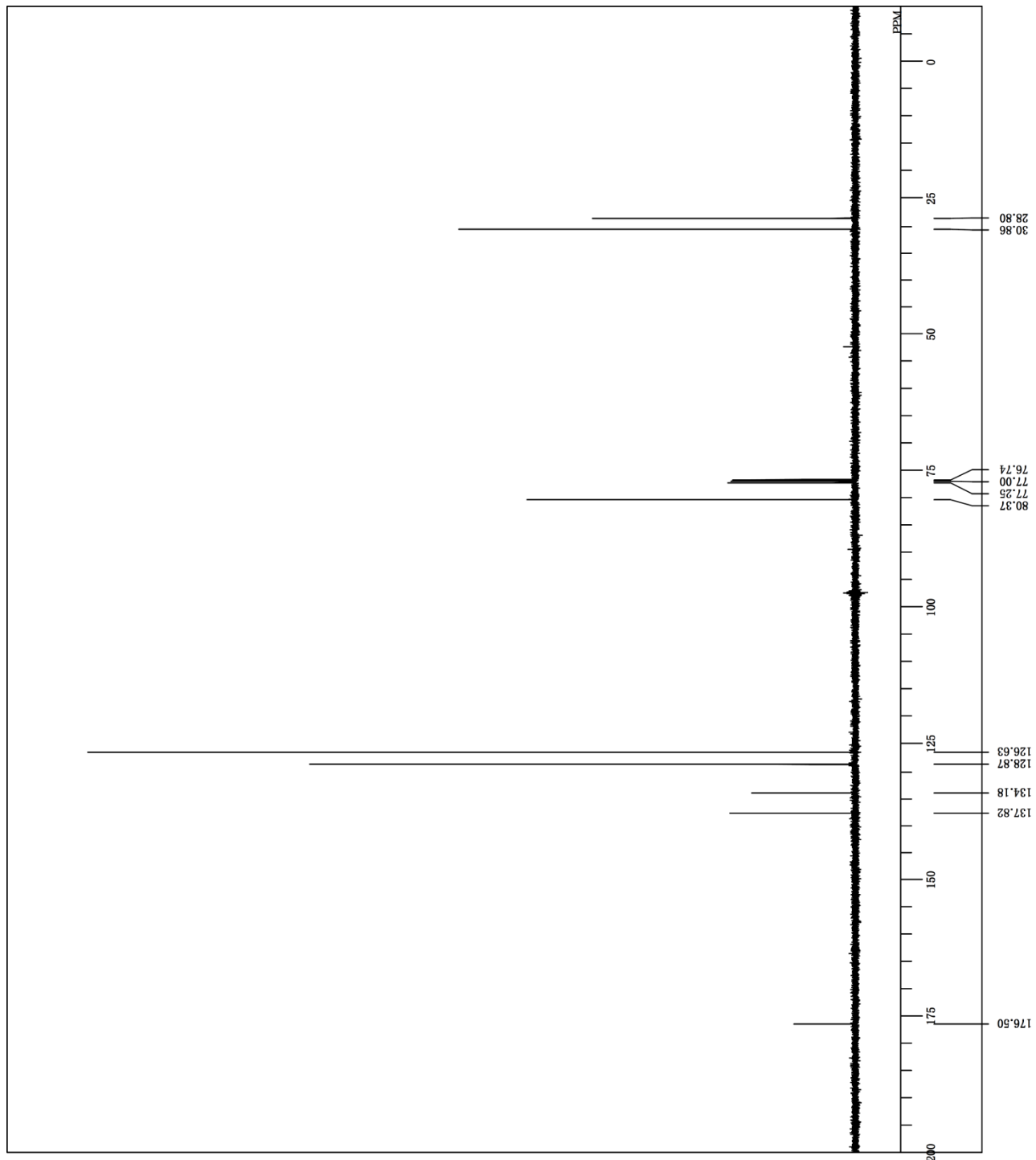


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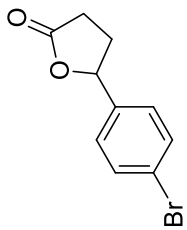




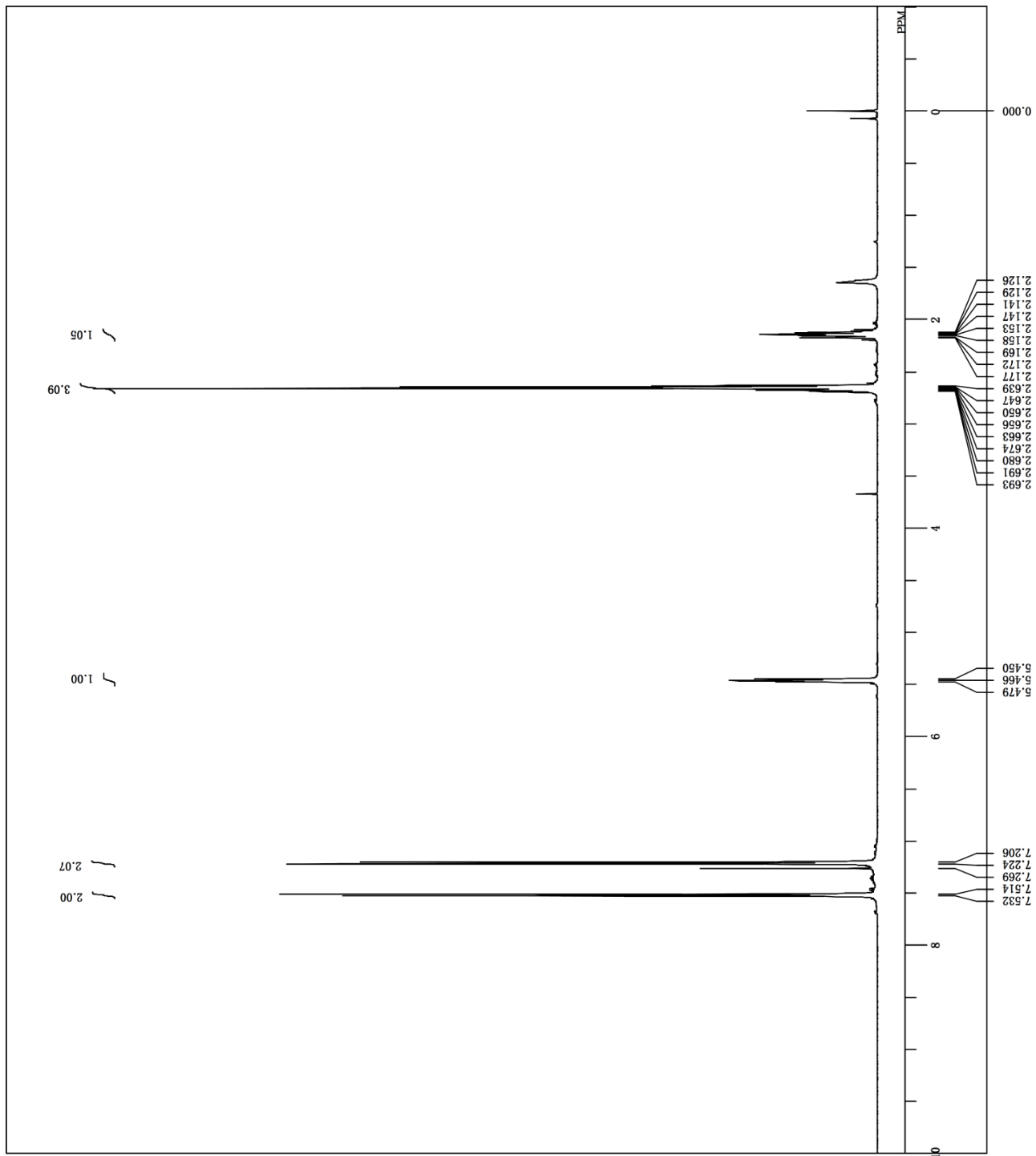
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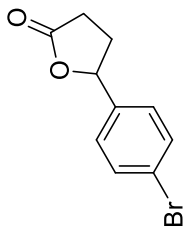


S61

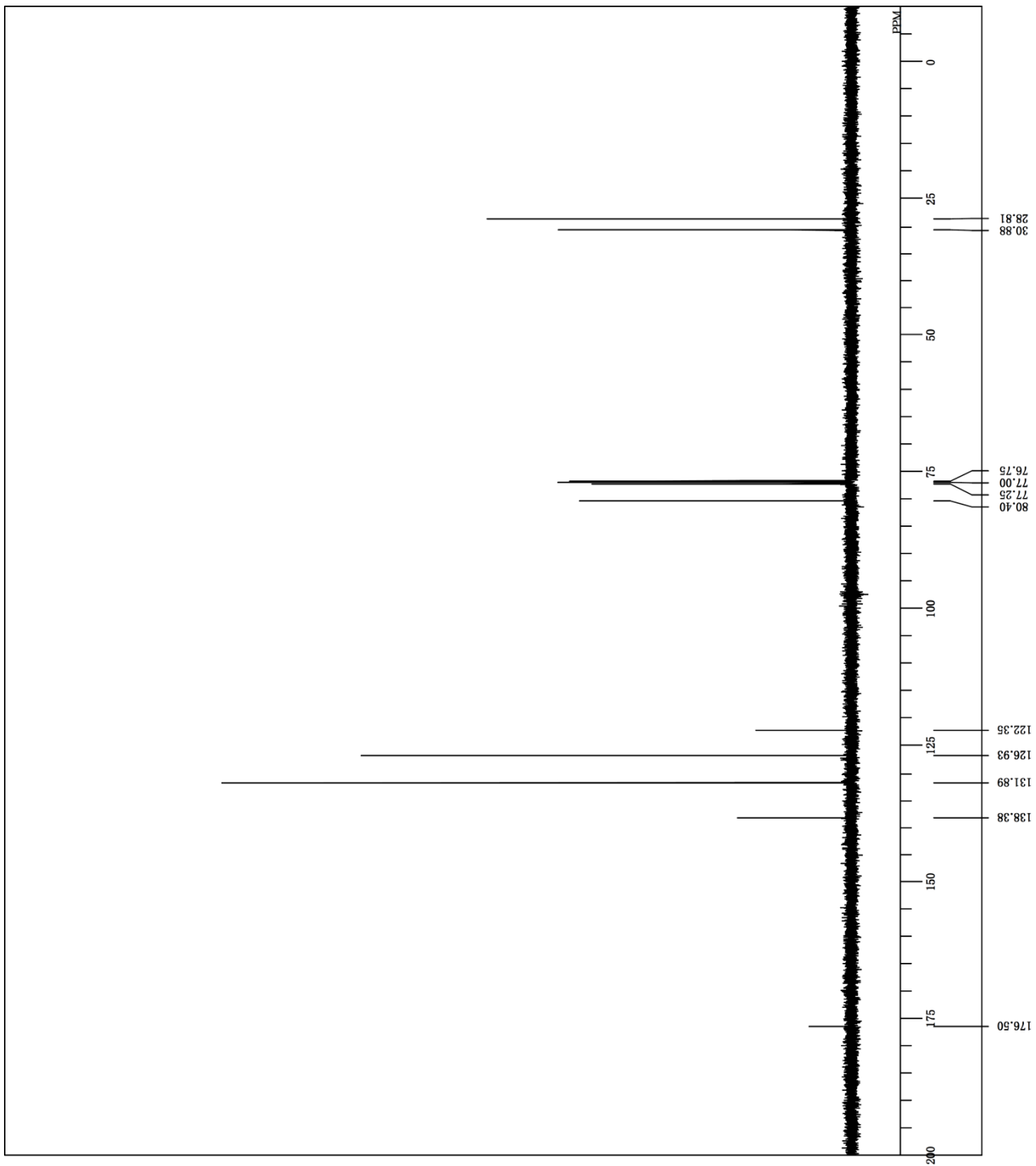


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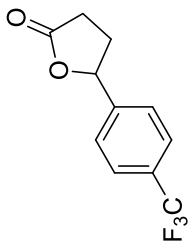




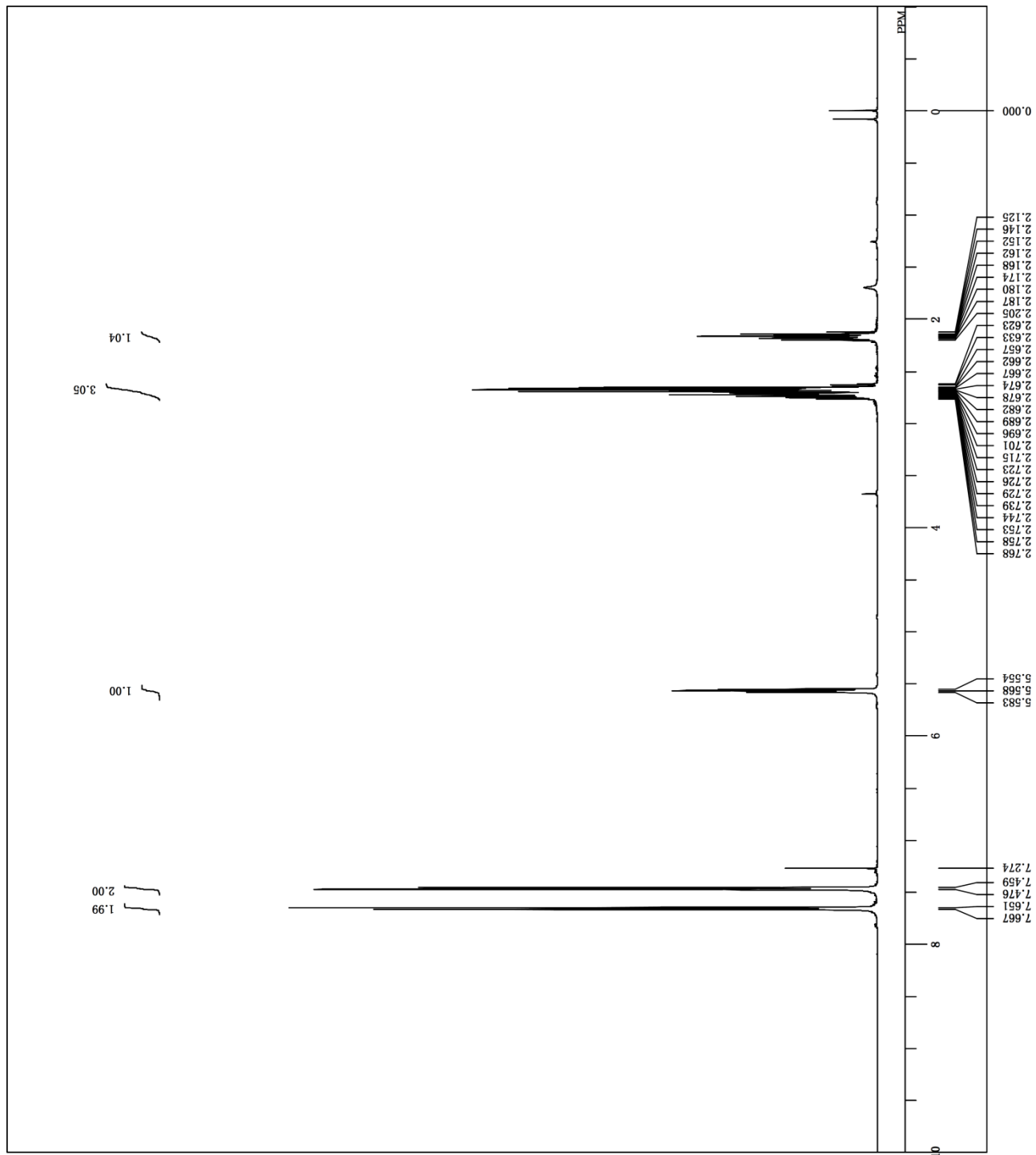
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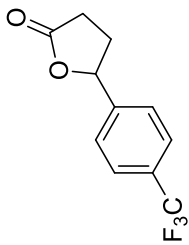


S63

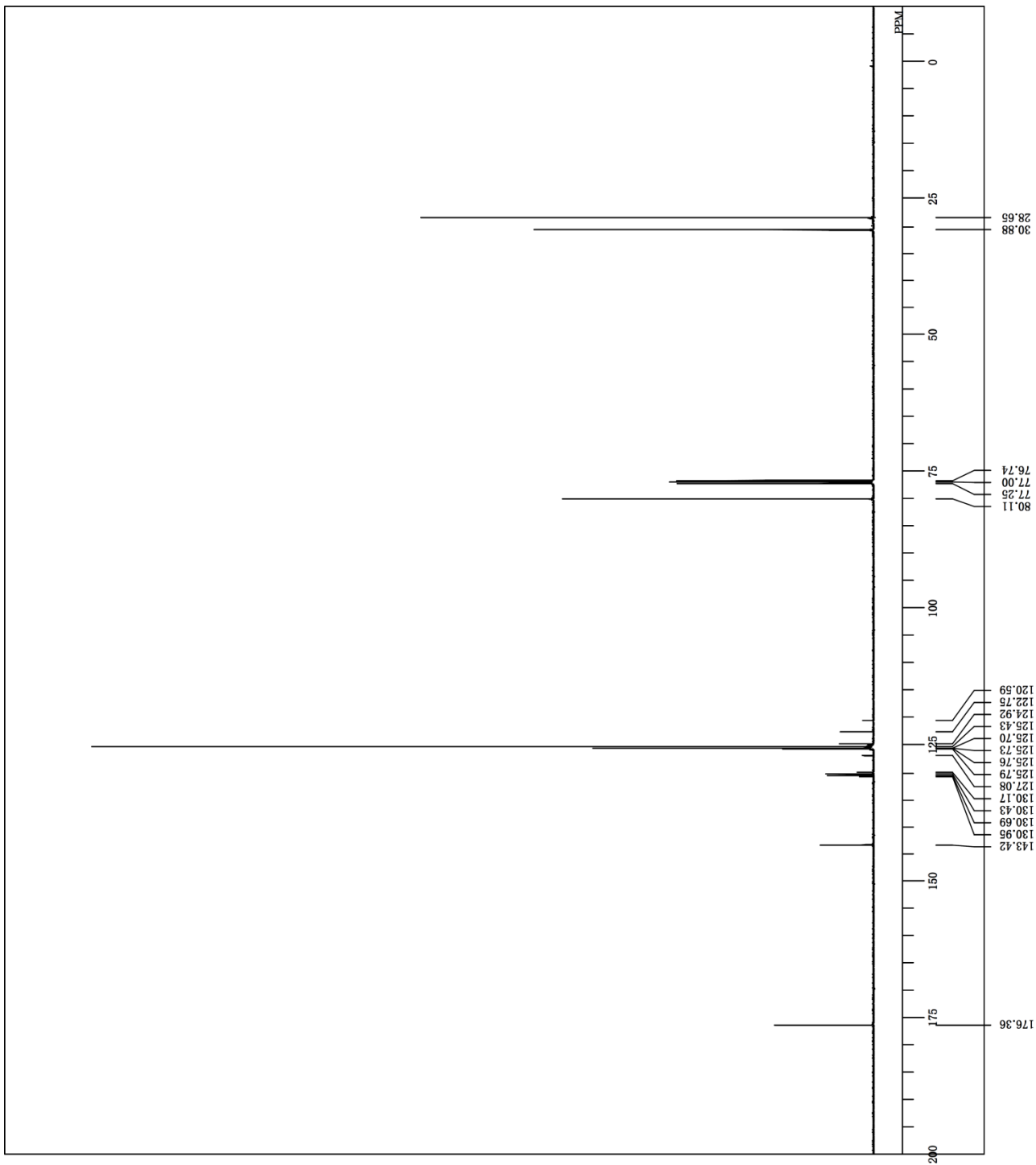


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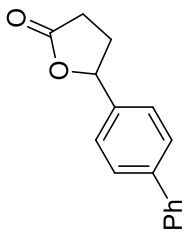




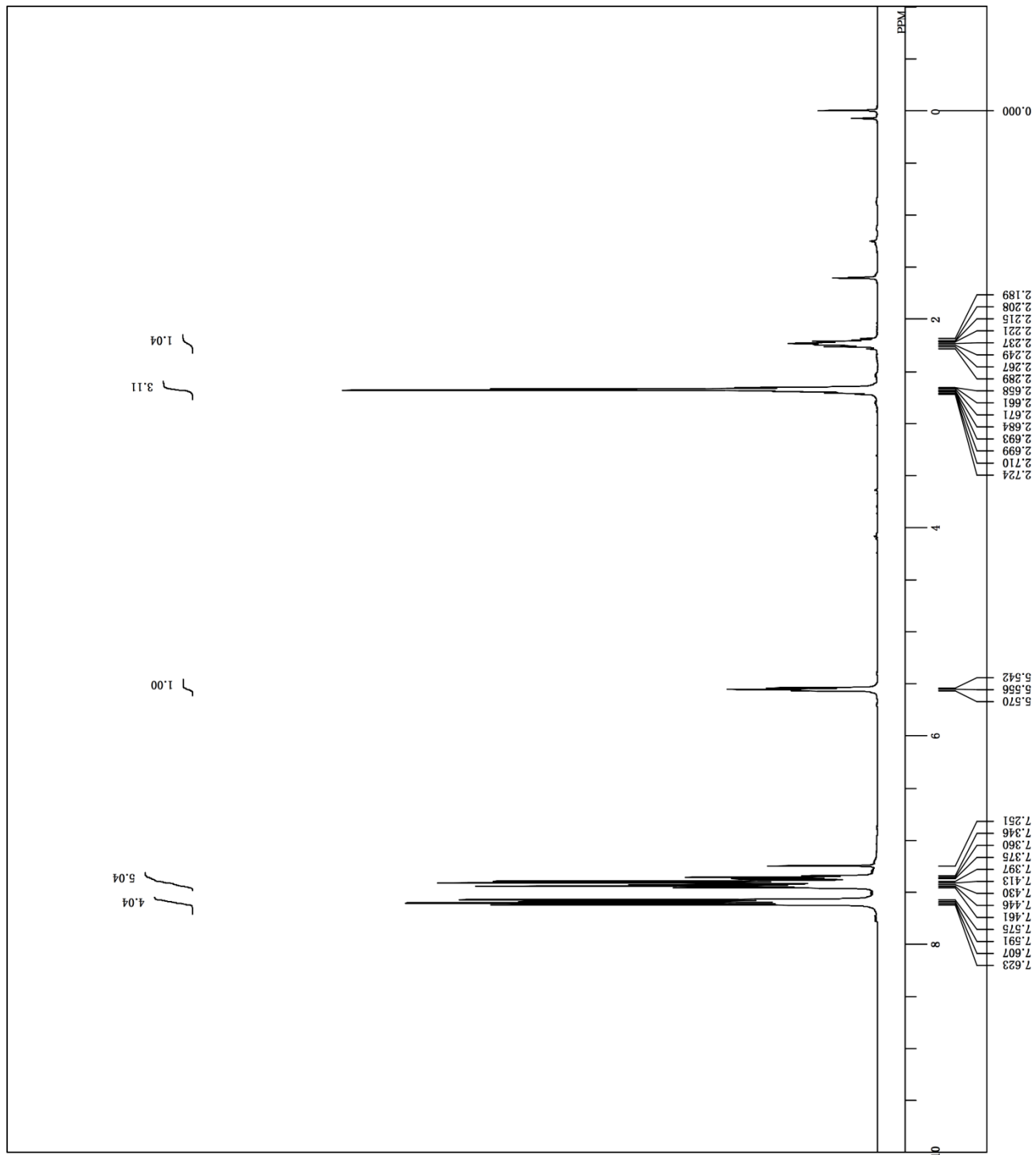
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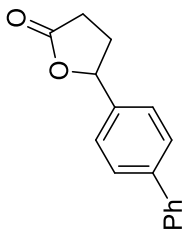


S65

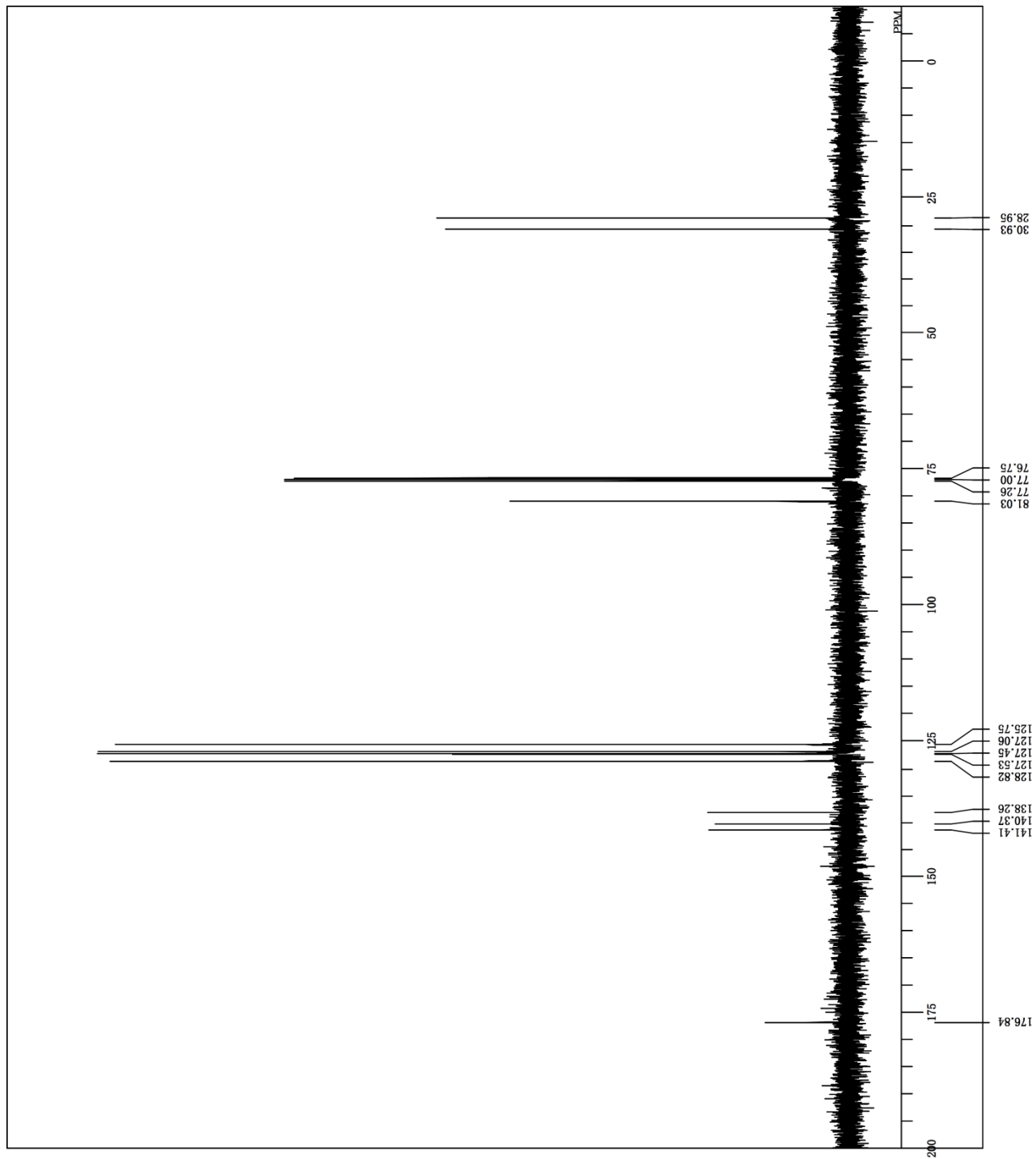


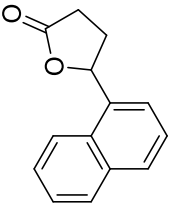
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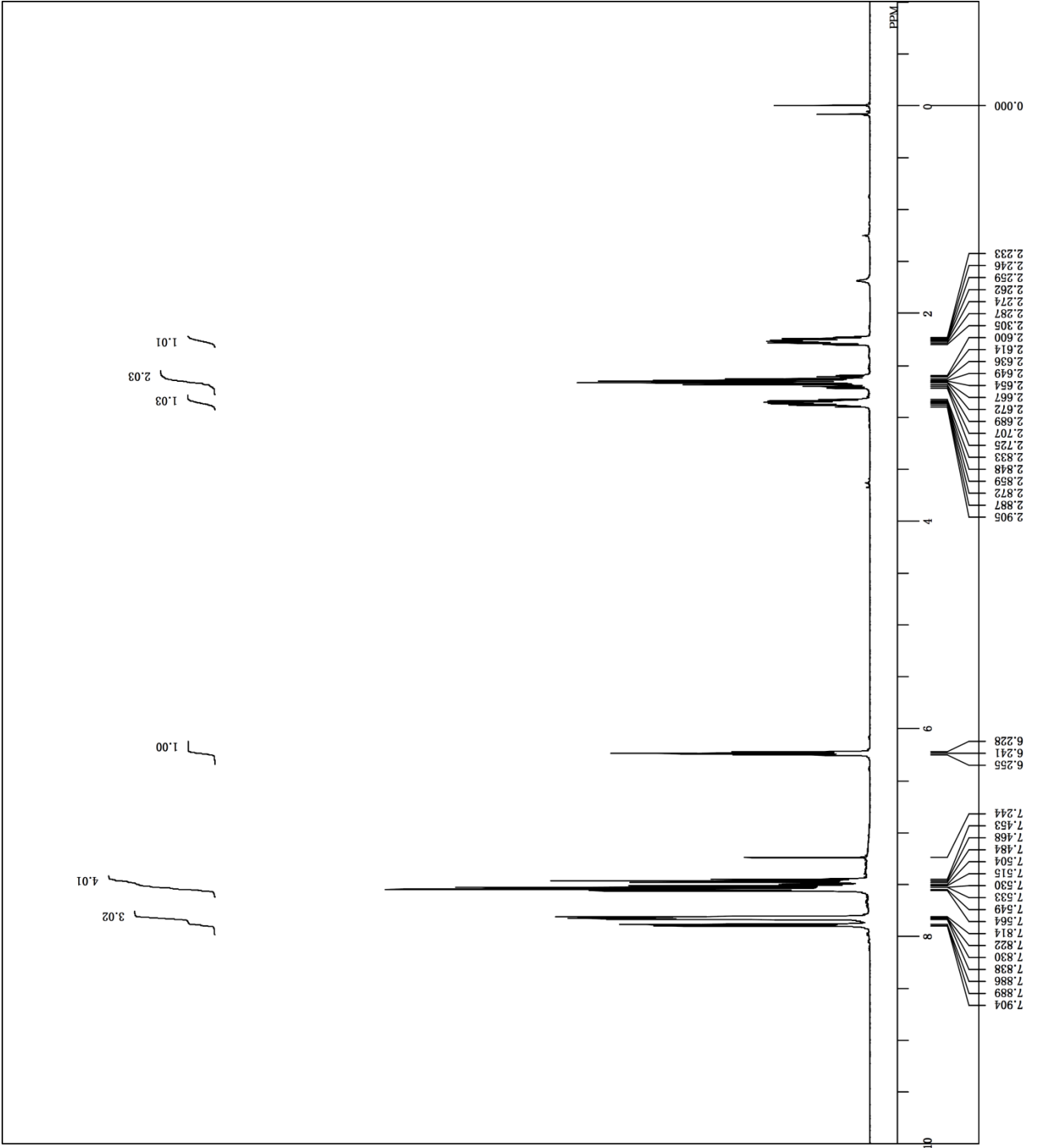


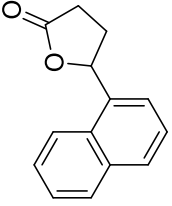
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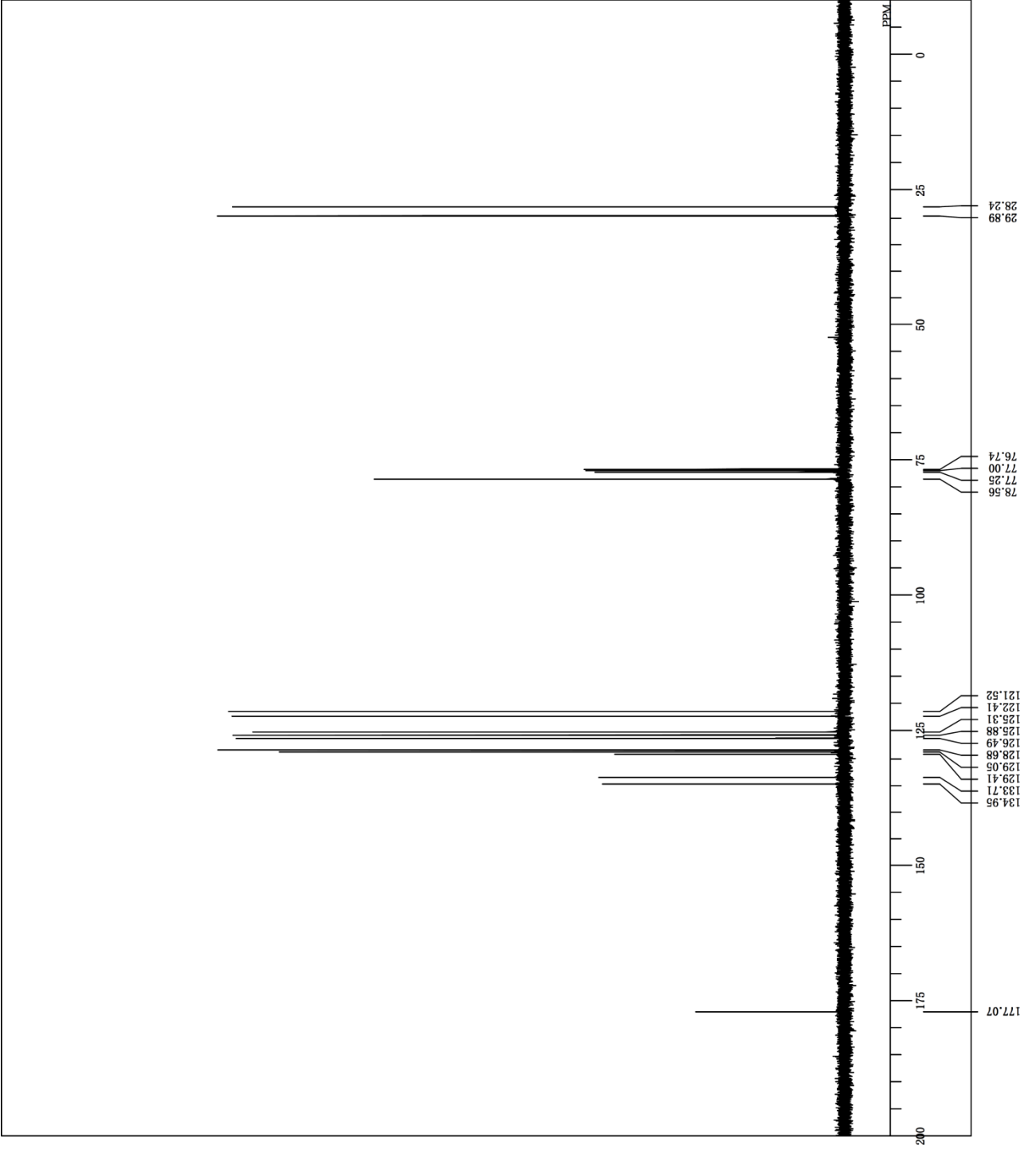


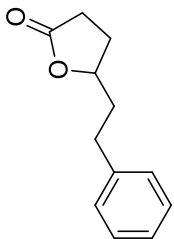
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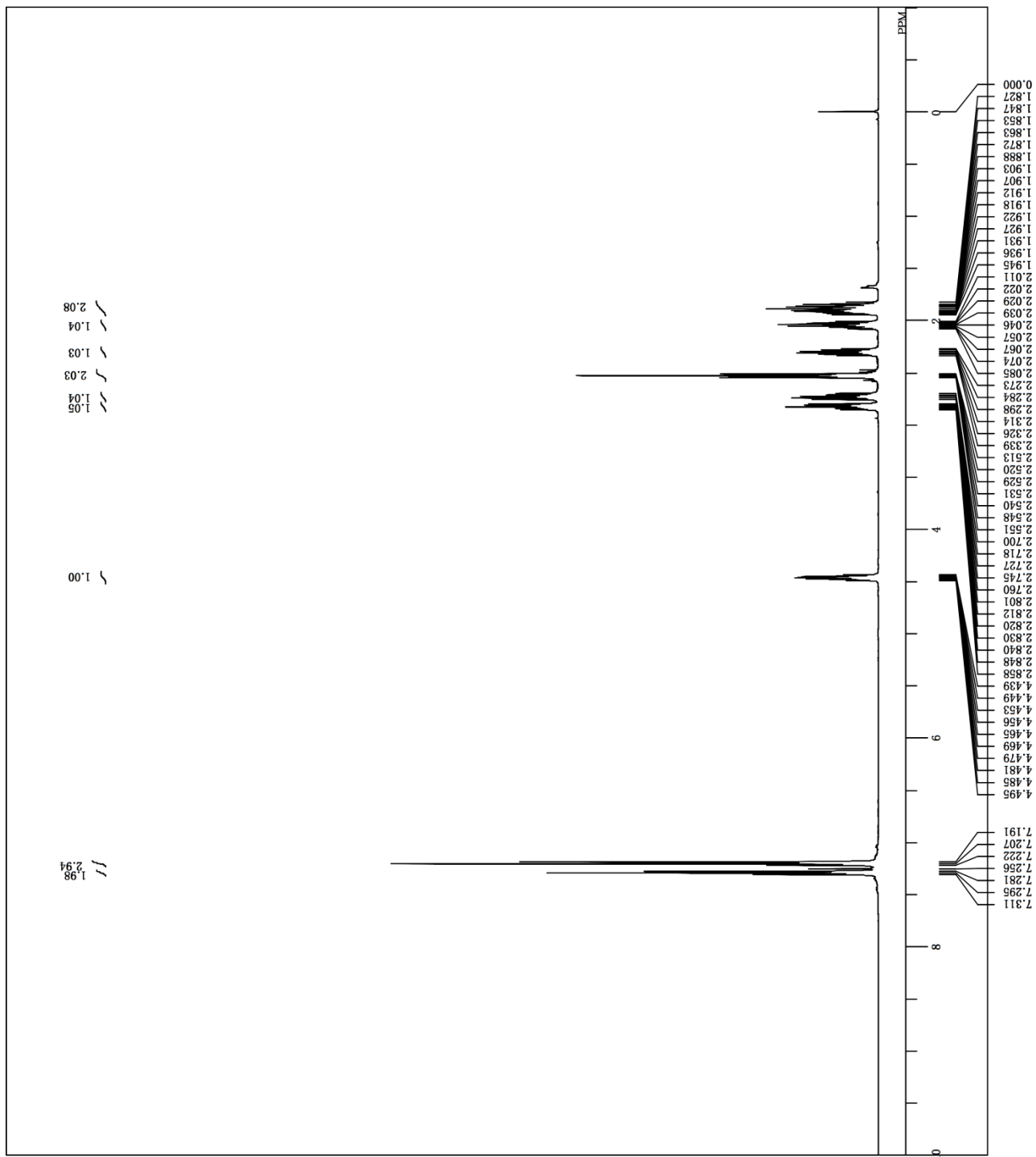


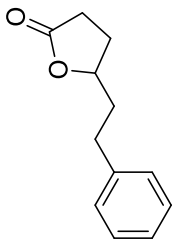
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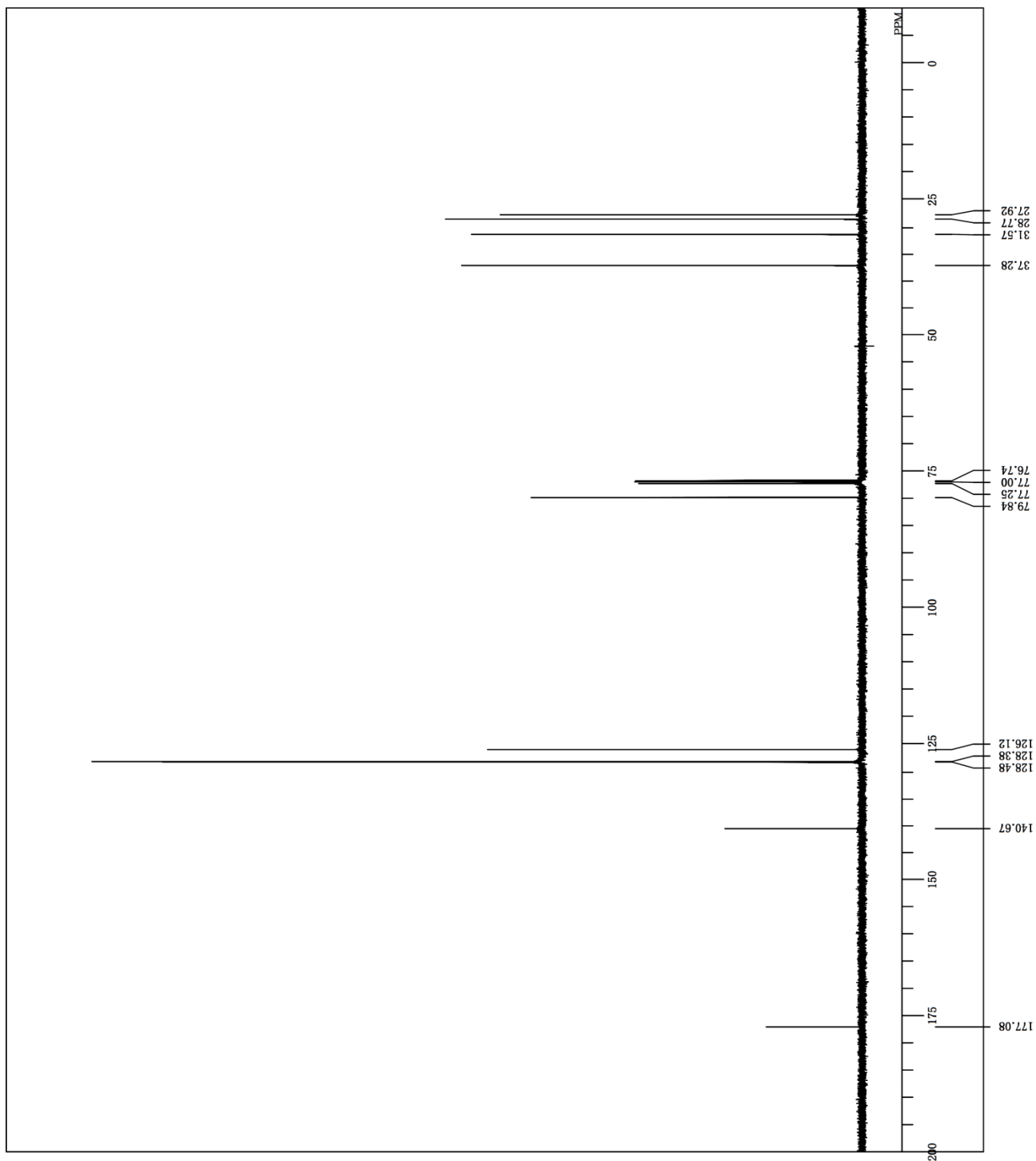


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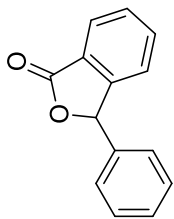




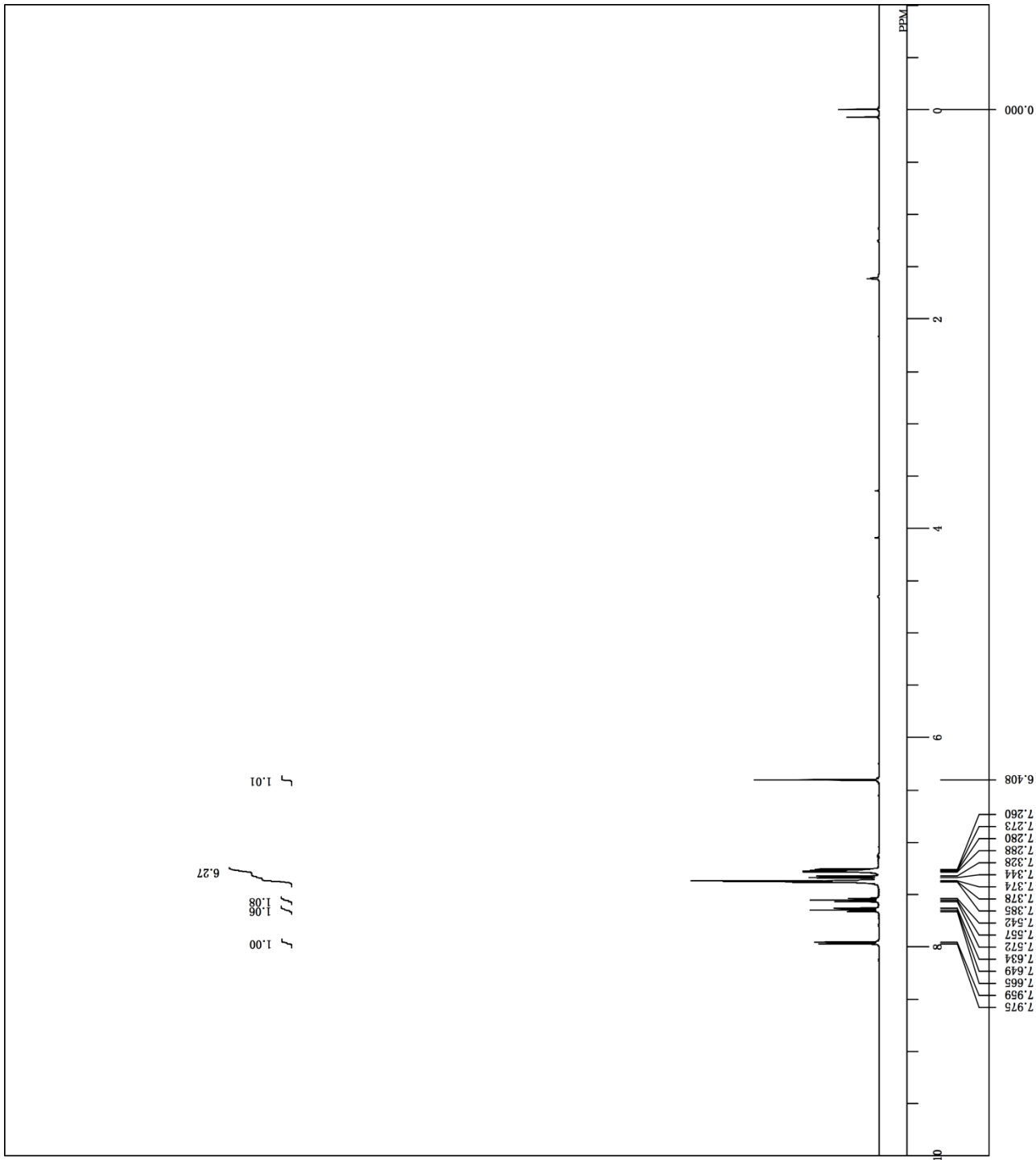
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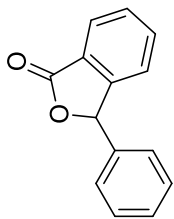


S71

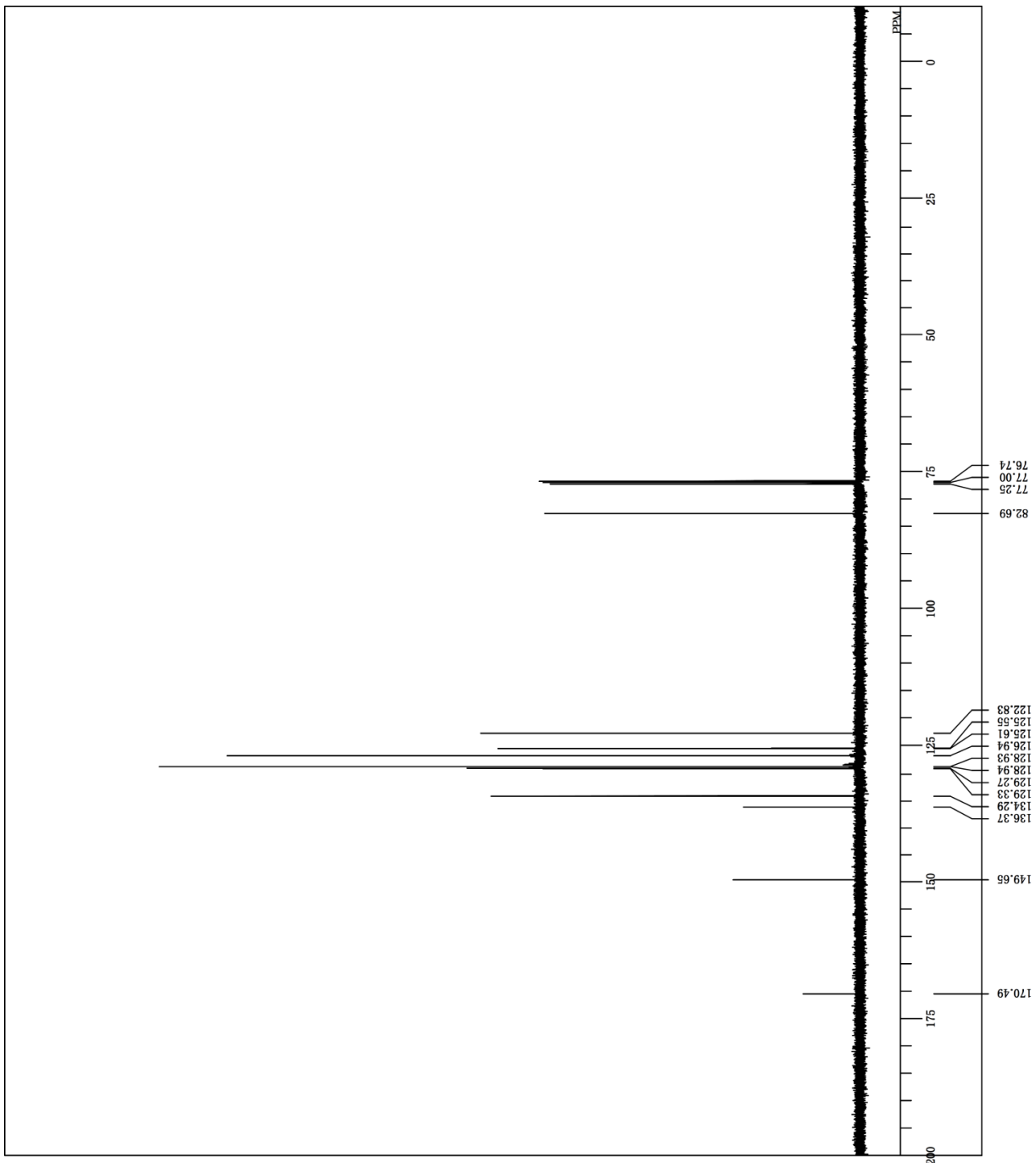


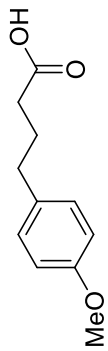
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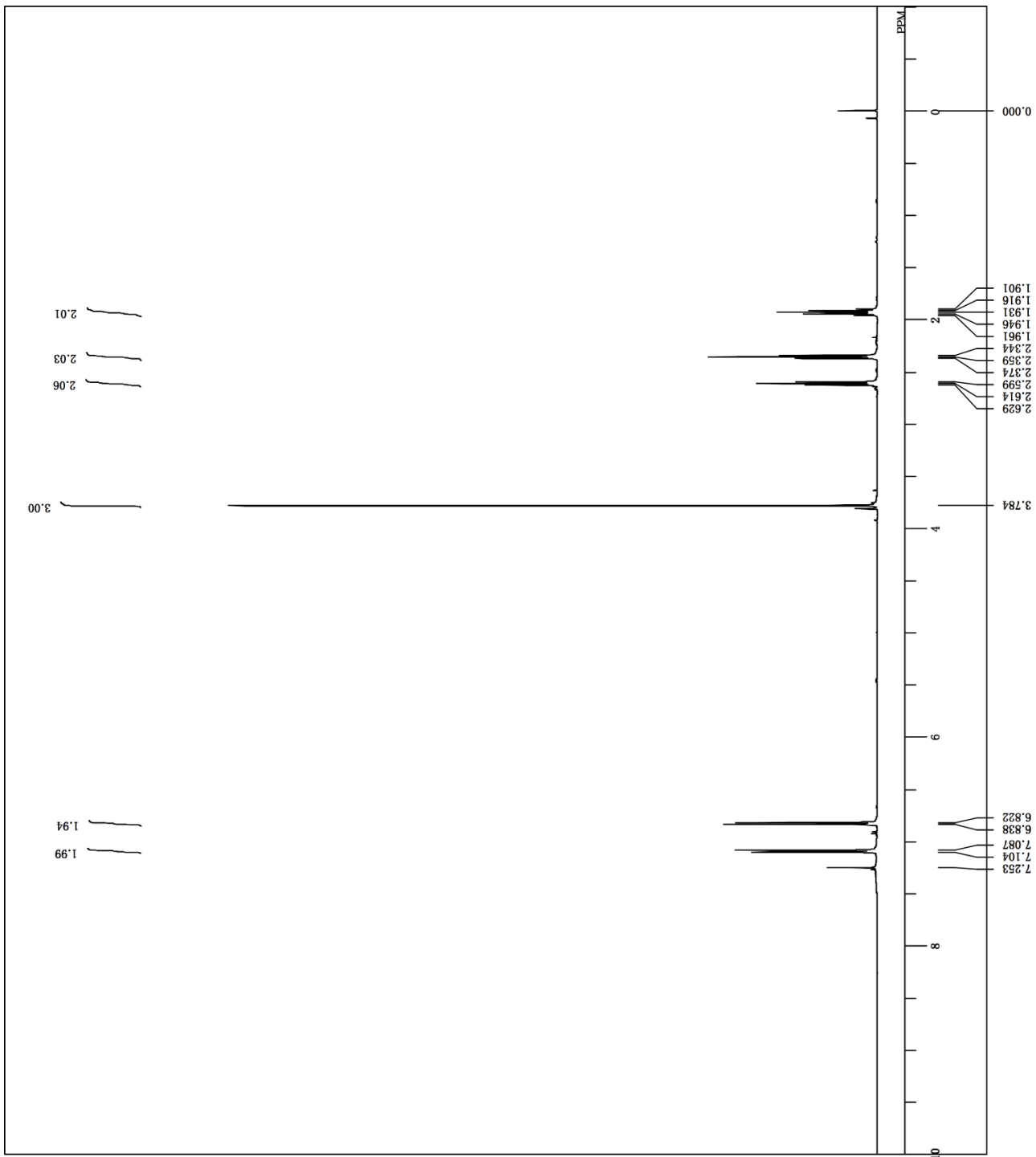


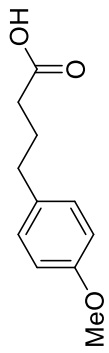
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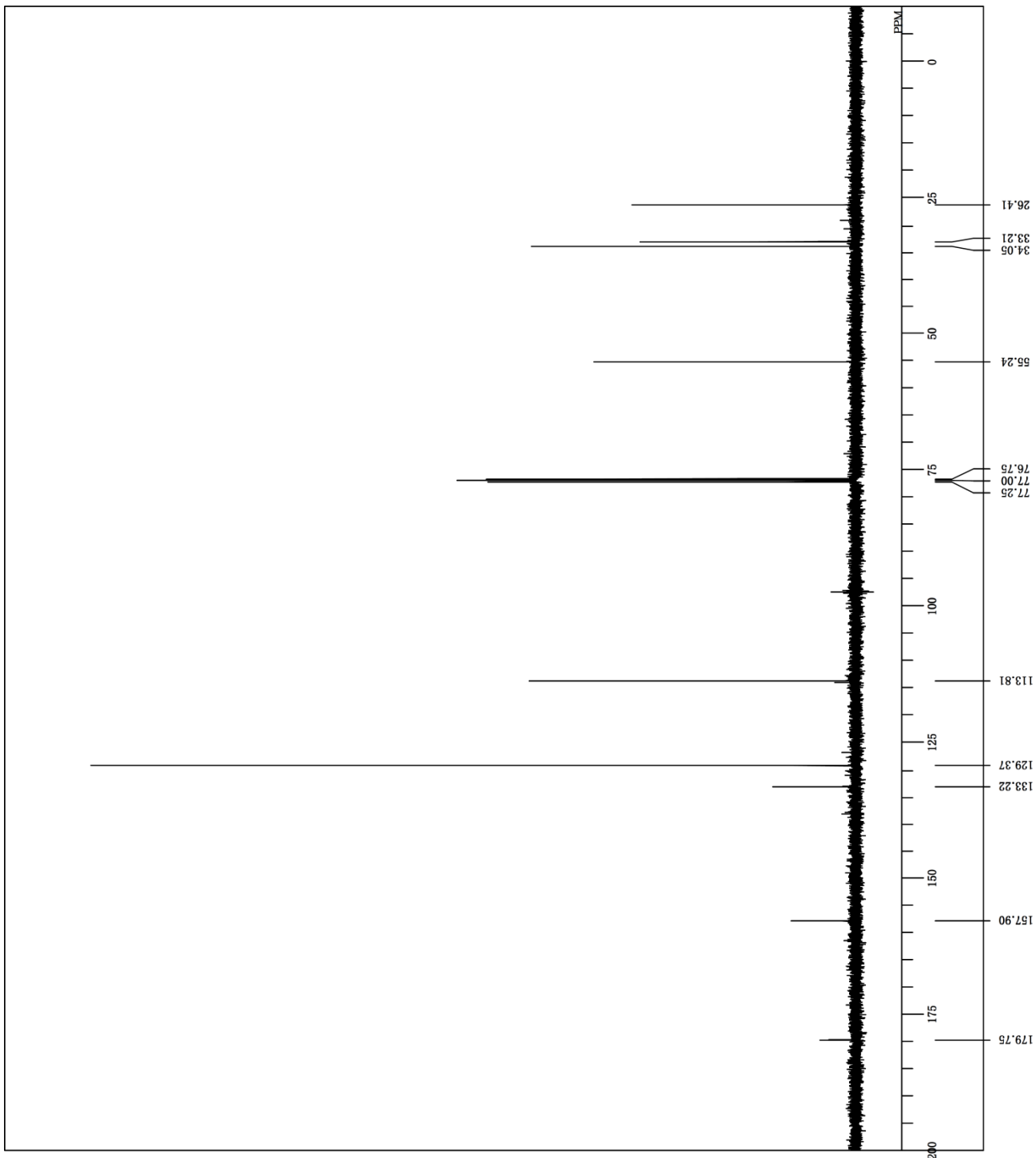


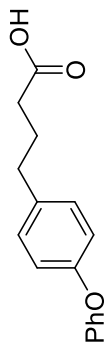
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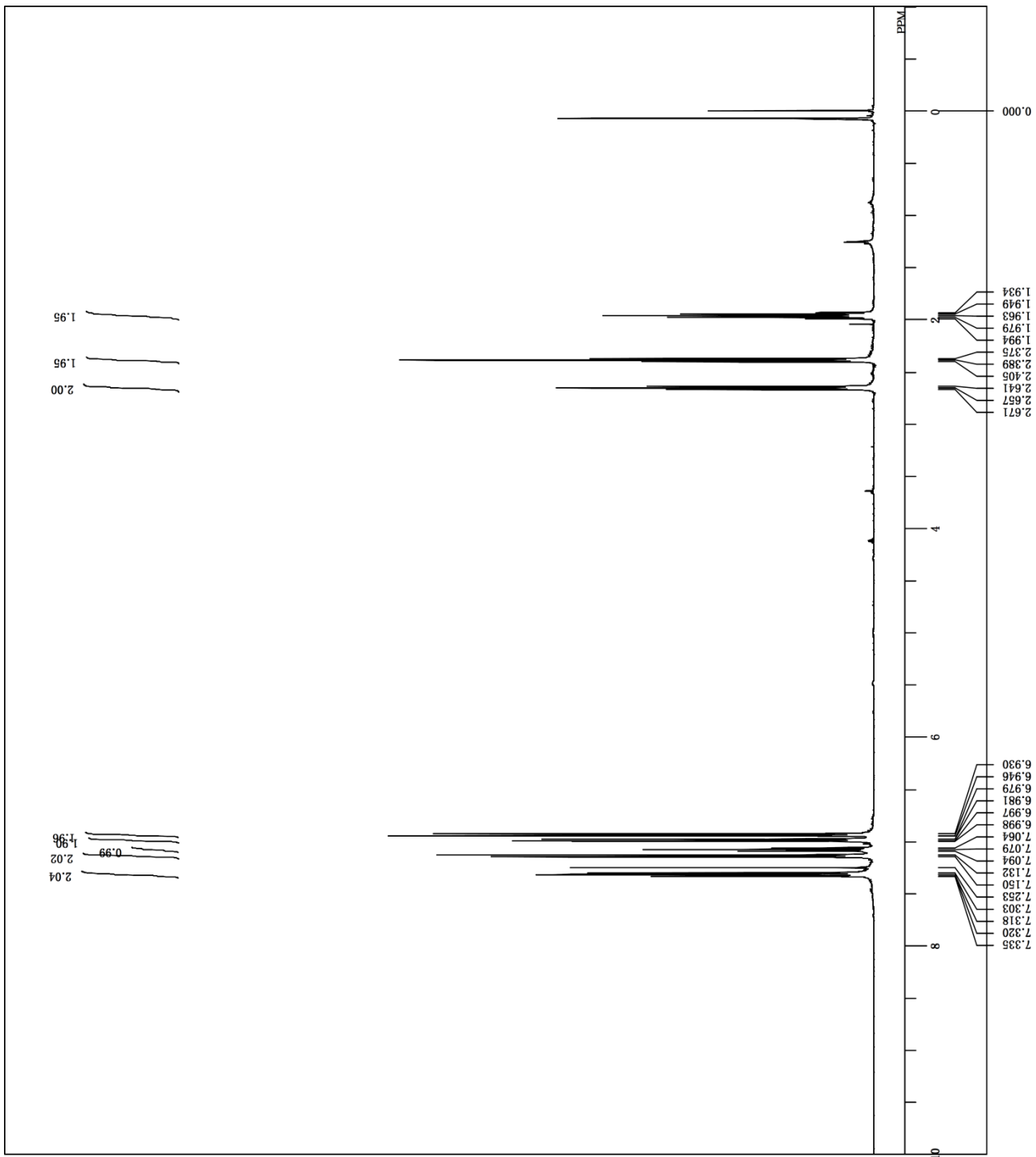


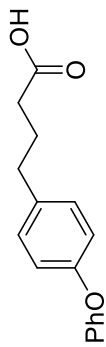
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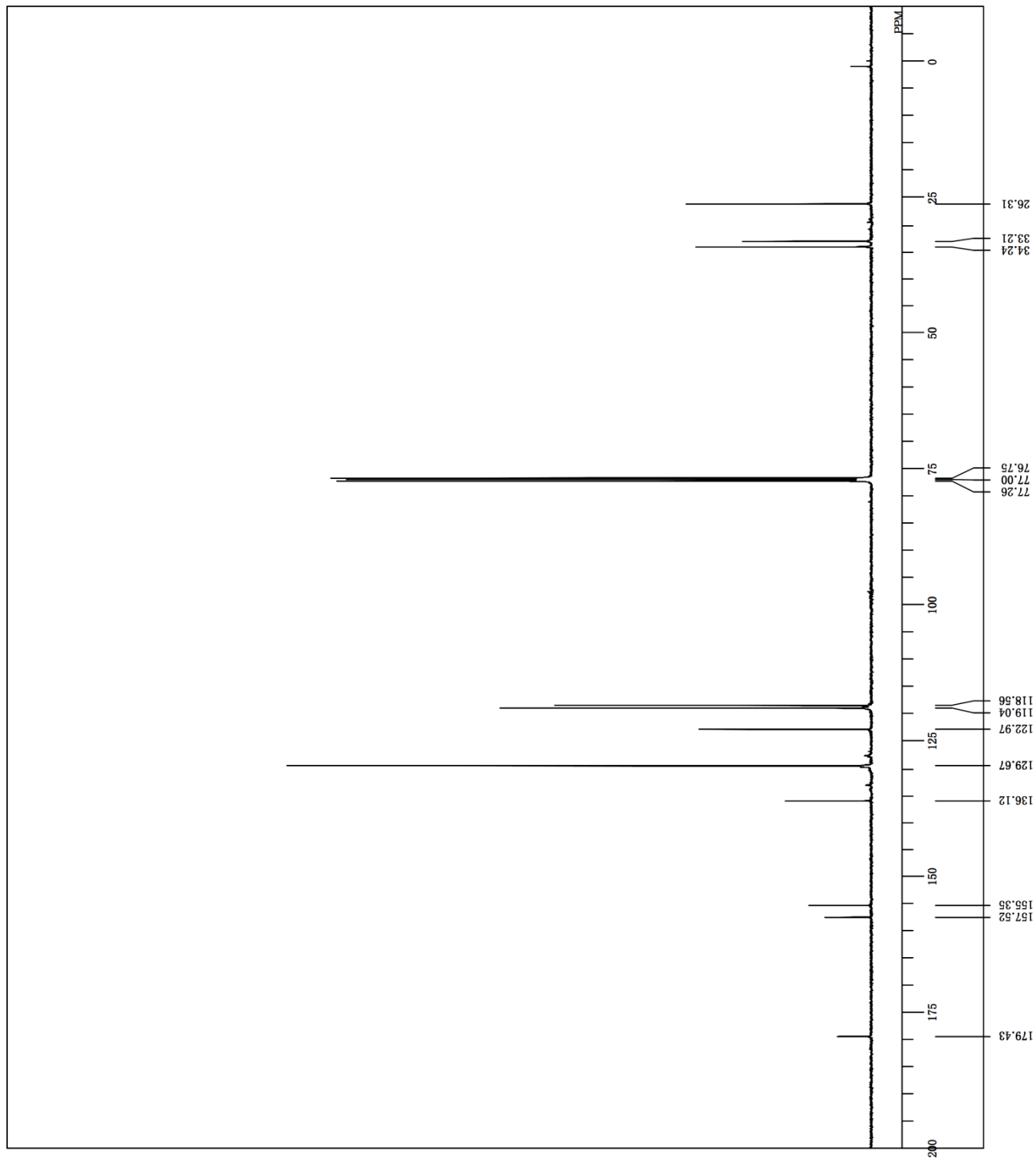


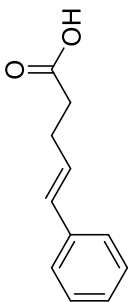
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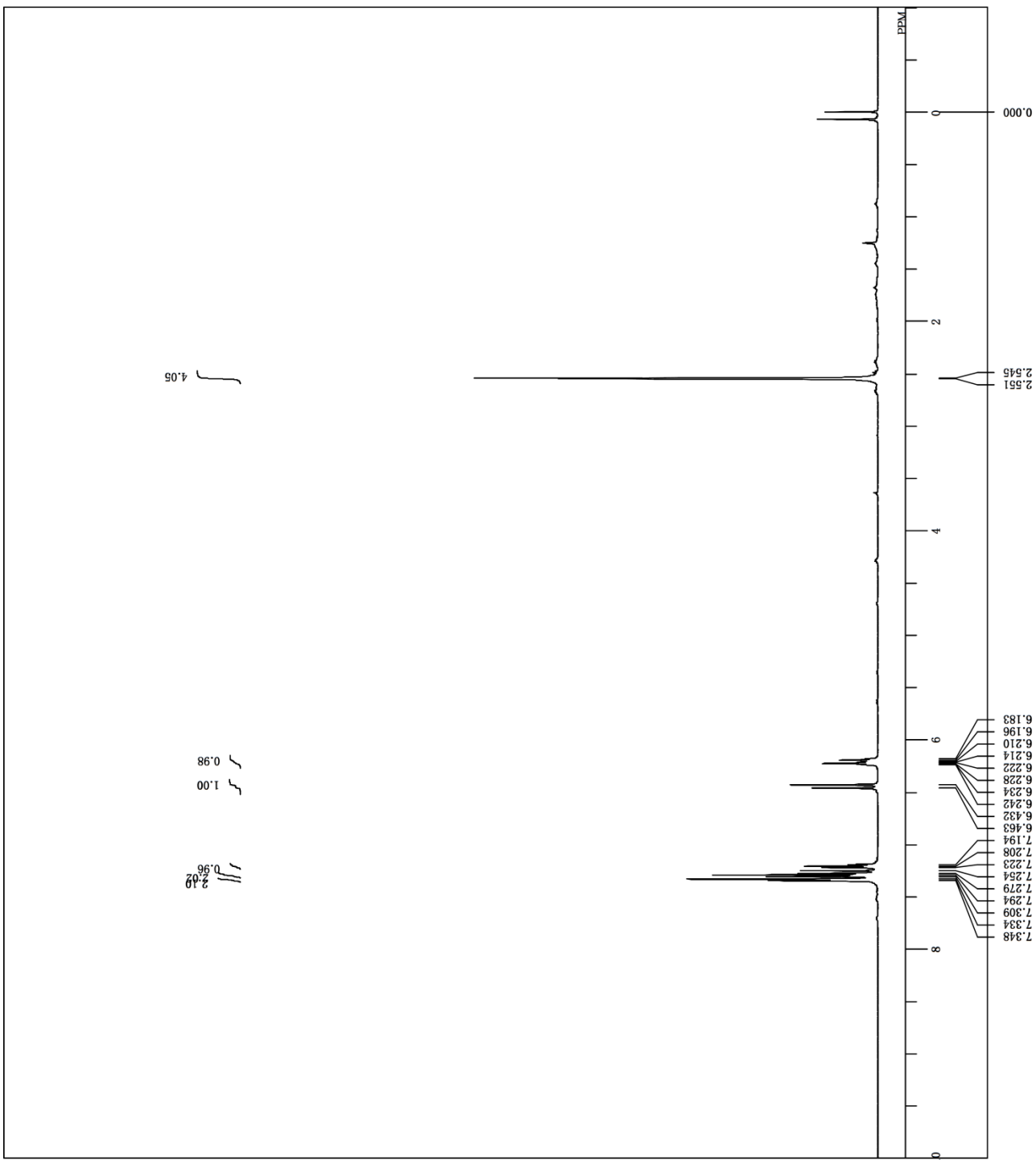


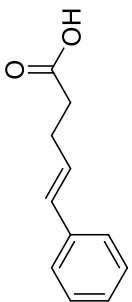
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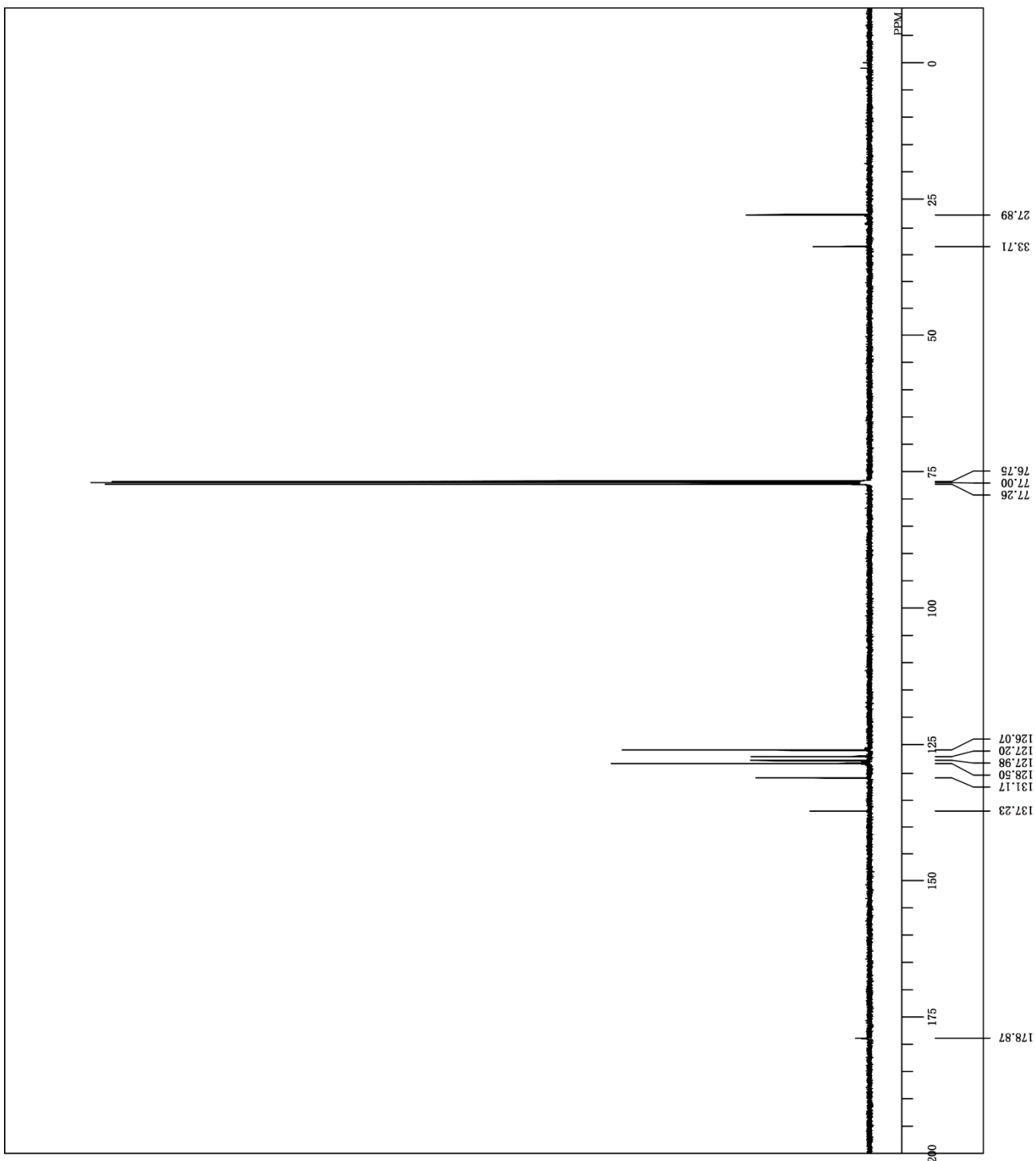


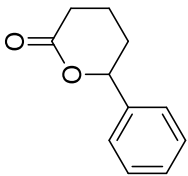
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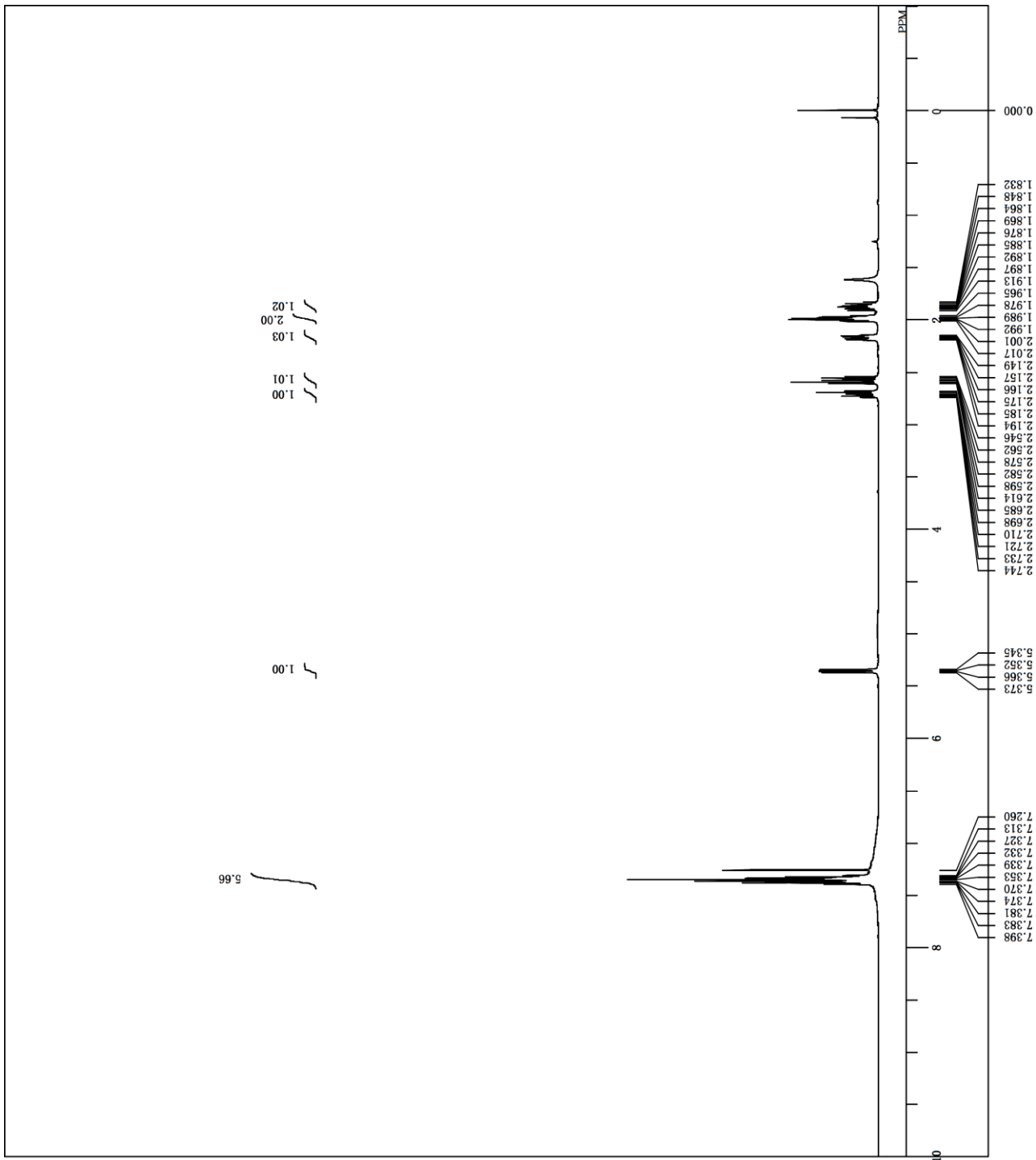


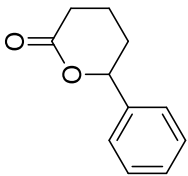
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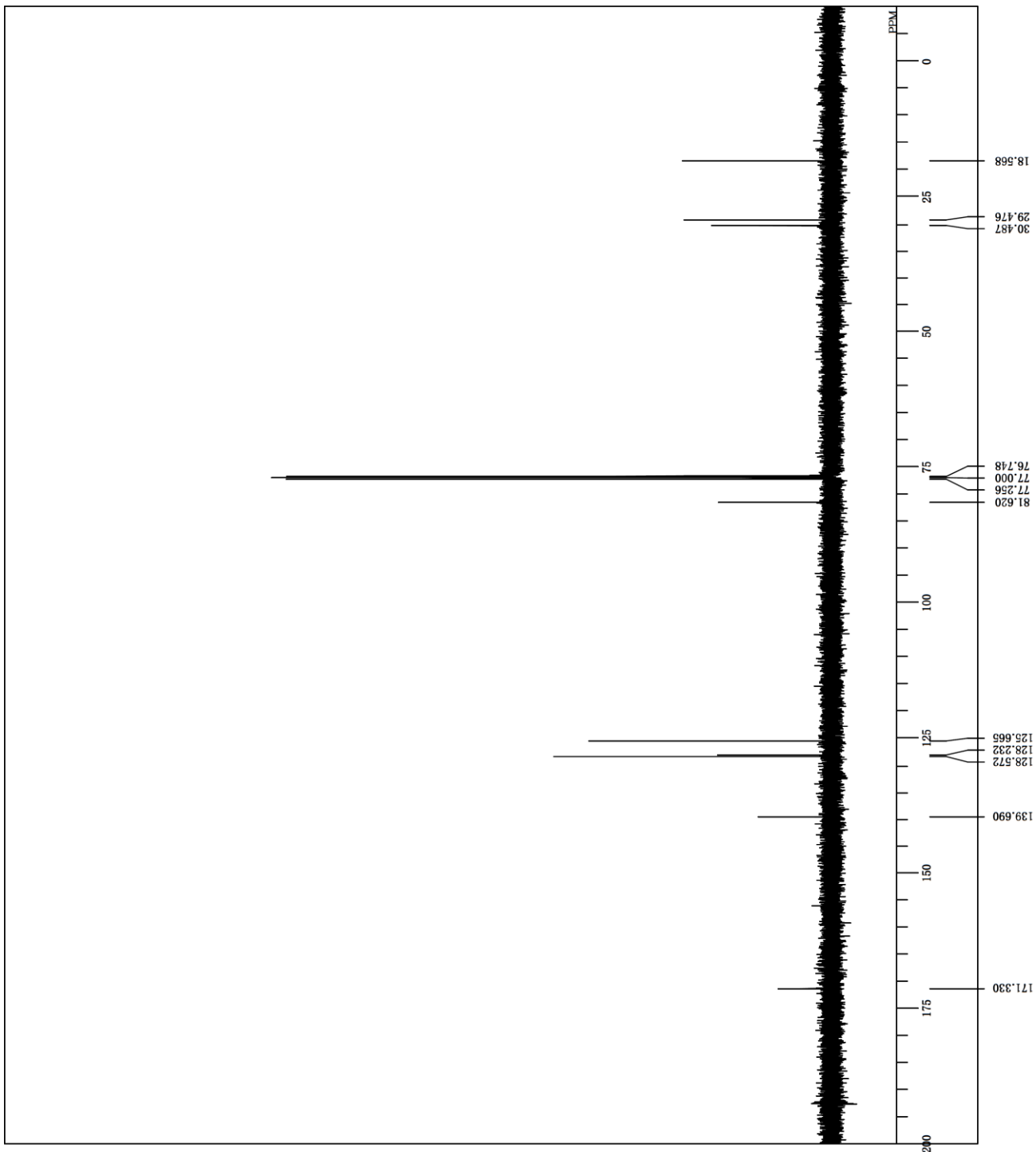


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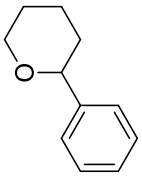




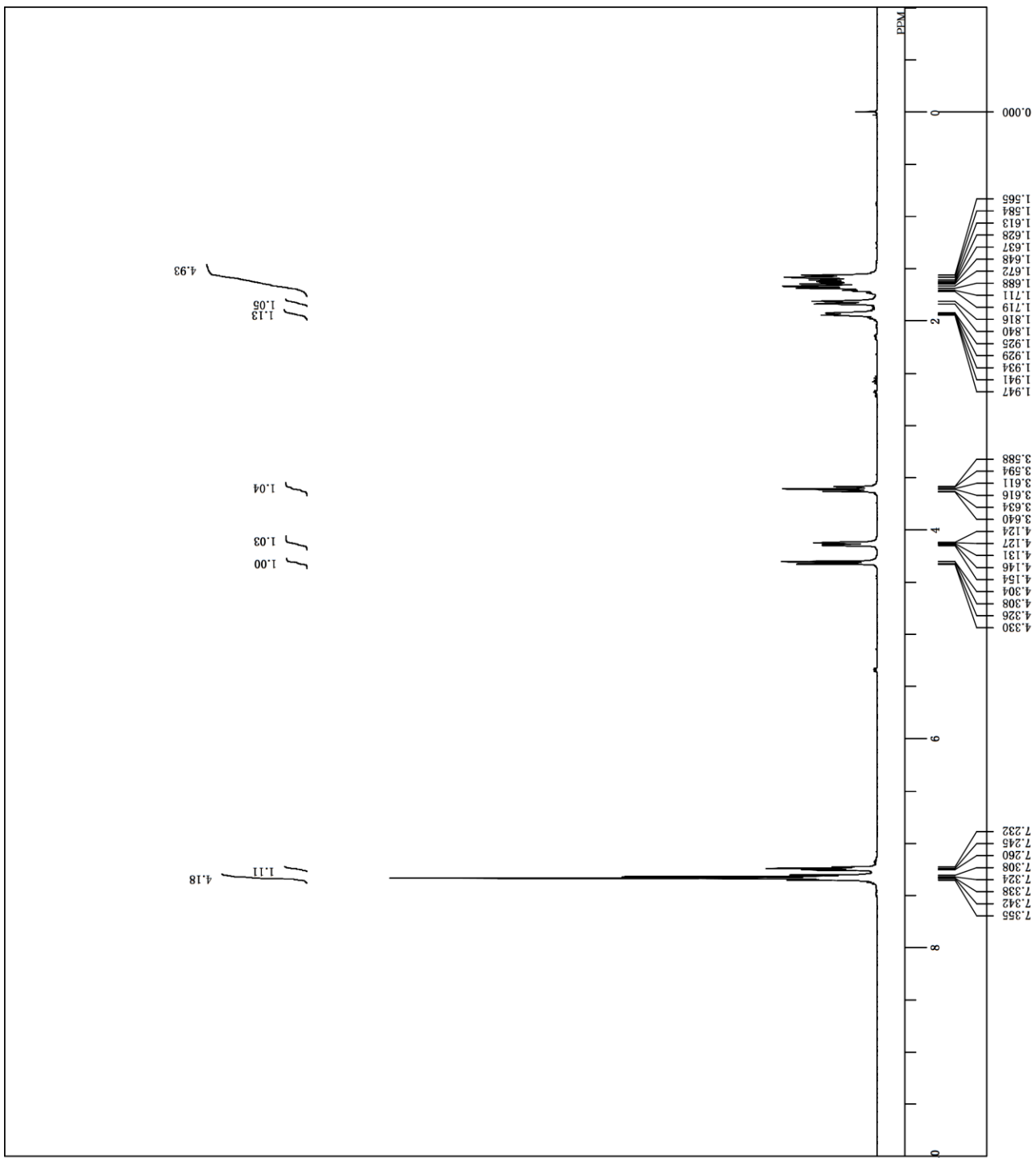
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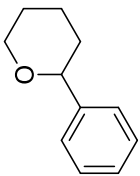


S81

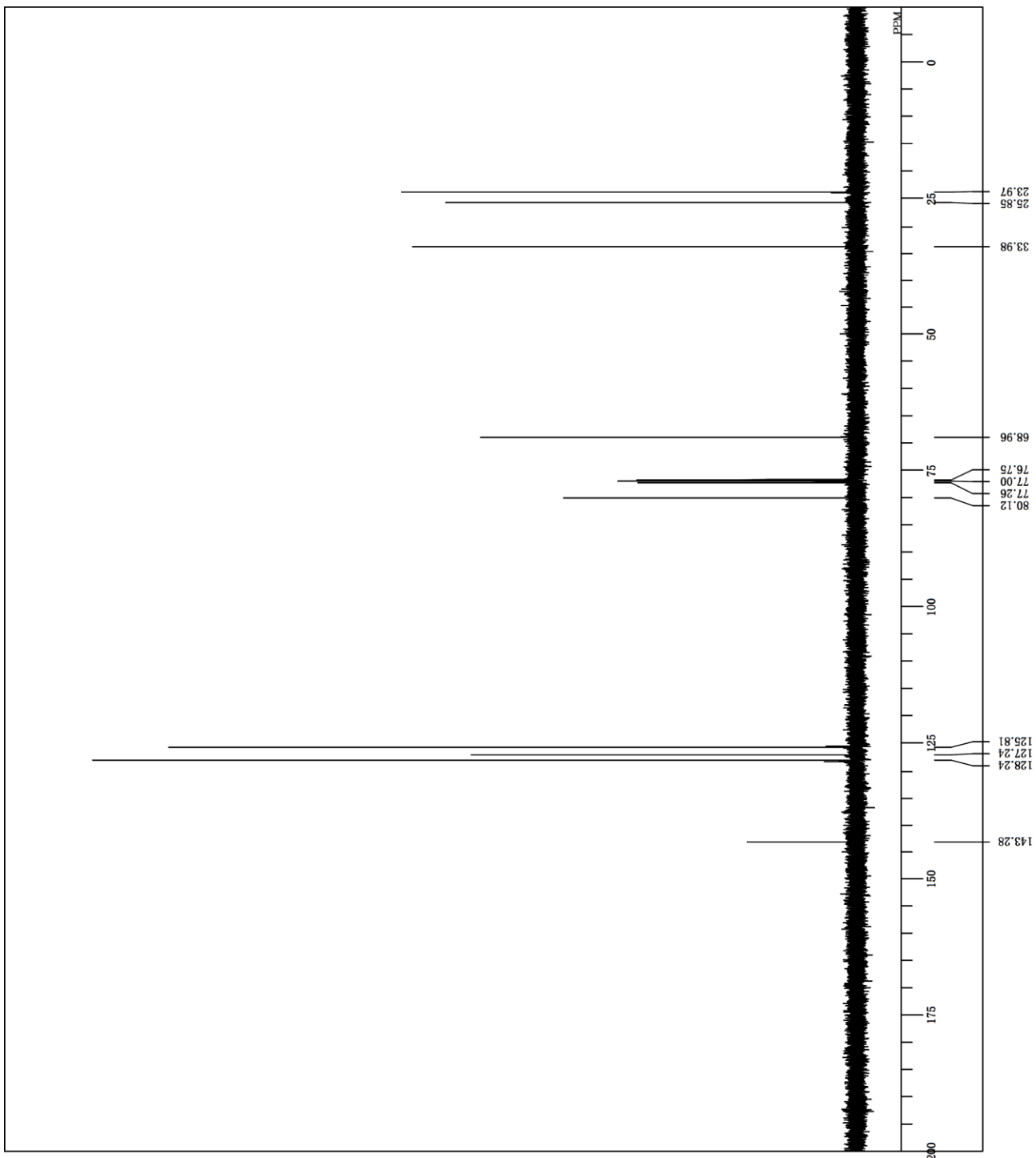


6





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S83