Supporting Informations

Copper catalyzed C-O bond formation via oxidative cross-coupling reaction of aldehydes and ethers

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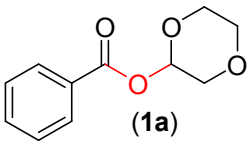
General information:

All manipulations were carried out under open air. All the reagents were commercial grade and purified according to the established procedures. Column chromatography was generally performed on silica gel (300-400 mesh) and reactions were monitored by thin layer chromatography (TLC) using UV light to visualize the course of the reactions. The ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) data were recorded on Varian spectrometers using $CDCl_3$ as solvent at room temperature. ^{1}H NMR spectra was recorded with tetramethylsilane as the internal reference; ^{13}C NMR spectra was recorded with $CDCl_3$ as the internal reference. Multiplicities are indicated as the following: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. ESI-MS were obtained using Agilent 1260-6120 instrument. ESI-HRMS were done on Varian 7.0 T FTICR-mass spectrometer. Elemental analysis was performed with a Perkin Elmer 2400 elemental analyzer. IR spectra were recorded in KBr or neat on a Nicolet Impact 410 spectrophotometer.

A typical procedure for copper catalyzed synthesis of α-acyloxy ethers *via* oxidative cross-coupling: benzaldehyde (1.0 mmol), $Cu(OAc)_2.H_2O$ (4 mg, 2 mol %), 1,4-dioxane (40 mmol), and TBHP (4.0 equiv, 70% aqueous solution) were added to a tube under open air. The reaction mixture was heated in an oil bath at 80℃. During this period complete disappearance of benzaldehyde was observed as judged from TLC. After cooling to room temperature, the organic solvent was removed under vacuum. The crude product was purified over a column of silica gel and eluted with (20: 1, hexane / ethyl acetate) to give **1a** (189 mg, 91% yield).

Spectral data:

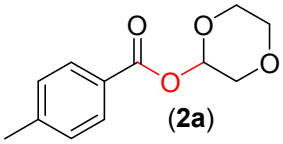
1,4-dioxan-2-yl benzoate



(**1a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66-3.70 (m, 1H), 3.82-3.84 (m, 2H), 3.89-3.90 (m, 2H), 4.19-4.24 (m, 1H), 6.10 (t, J = 2.0 Hz, 1H), 7.44-7.46 (m, 2H), 7.57-7.61 (m, 1H), 8.12-8.14(m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 61.8, 66.1, 67.8, 89.8, 128.4, 129.7, 129.9, 133.4, 165.2; ESI-MS ($C_{11}H_{12}O_4Na$): 231; IR (KBr, cm^{-1}): 1725.

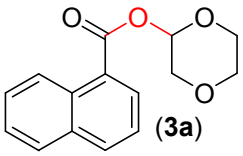
1,4-dioxan-2-yl 4-methylbenzoate



(**2a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.42 (s, 3H), 3.66-3.69 (m, 1H), 3.81-3.83 (m, 2H), 3.88-3.89 (m, 2H), 4.18-4.23 (m, 1H), 6.08 (t, J = 2.0 Hz, 1H), 7.25 (d, J = 8.4 Hz, 2H), 8.01 (d, J = 8.4 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 21.6, 61.8, 66.1, 67.9, 89.6, 127.0, 129.1, 129.9, 144.1, 165.2; ESI-MS ($C_{12}H_{14}O_4Na$): 245; IR (KBr, cm^{-1}): 1731. Anal. Calcd. for $C_{12}H_{14}O_4$: C, 64.85%; H, 6.35%. Found: C, 65.01%; H, 6.42%.

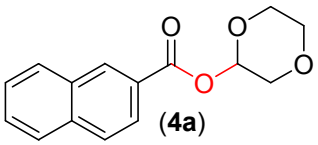
1,4-dioxan-2-yl 1-naphthoate



(**3a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.70-3.74 (m, 1H), 3.84-3.87 (m, 2H), 3.95-3.96 (m, 2H), 4.24-4.28 (m, 1H), 6.21 (t, J = 2.0 Hz, 1H), 7.50-7.54 (m, 2H), 7.61-7.65(m, 1H), 7.88-7.90 (m, 1H), 8.04-8.06(m, 1H), 8.34-8.36 (m, 1H), 9.00-9.02(m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 61.9, 66.2, 67.9, 89.8, 124.5, 125.7, 126.1, 126.3, 128.0, 128.6, 130.9, 131.5, 133.8, 134.0, 165.9; ESI-MS ($C_{15}H_{14}O_4Na$): 281; IR (KBr, cm^{-1}): 1732. Anal. Calcd. for $C_{15}H_{14}O_4$: C, 69.76%; H, 5.46%. Found: C, 69.52%; H, 5.40%.

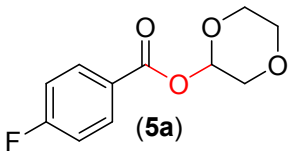
1,4-dioxan-2-yl 2-naphthoate



(**4a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.69-3.73 (m, 1H), 3.84-3.87 (m, 2H), 3.94-3.95 (m, 2H), 4.25-4.30 (m, 1H), 6.16 (t, J = 2.0 Hz, 1H), 7.53-7.62 (m, 2H), 7.87-7.89(m, 2H), 7.96-7.98 (m, 1H), 8.11-8.14(m, 1H), 8.69 (s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 61.8, 66.2, 67.9, 89.9, 125.3, 126.7, 126.9, 127.8, 128.3, 128.5, 129.4, 131.5, 132.4, 135.7, 165.4; ESI-MS ($C_{15}H_{14}O_4Na$): 281; IR (KBr, cm^{-1}): 1732. Anal. Calcd. for $C_{15}H_{14}O_4$: C, 69.76%; H, 5.46%. Found: C, 69.59%; H, 5.52%.

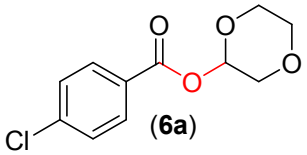
1,4-dioxan-2-yl 4-fluorobenzoate



(**5a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66-3.71 (m, 1H), 3.82-3.85 (m, 2H), 3.89-3.90 (m, 2H), 4.18-4.24 (m, 1H), 6.09 (t, J = 2.0 Hz, 1H), 7.11-7.16 (m, 2H), 8.13-8.16 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 61.8, 66.1, 67.8, 89.9, 115.5, 115.7, 125.9, 132.4, 132.5, 164.2, 164.7, 167.3; ESI-MS ($C_{11}H_{11}NaFO_4$): 249; IR (KBr, cm^{-1}): 1729.

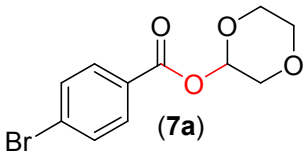
1,4-dioxan-2-yl 4-chlorobenzoate



(**6a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66-3.70 (m, 1H), 3.82-3.84 (m, 2H), 3.89-3.90 (m, 2H), 4.17-4.24 (m, 1H), 6.09 (t, J = 2.0 Hz, 1H), 7.44 (d, J = 8.8 Hz, 2H), 8.06 (d, J = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): 61.8, 66.1, 67.7, 90.0, 128.1, 128.8, 131.1, 131.3, 139.9, 164.4; ESI-MS ($C_{11}H_{11}ClNaO_4$): 265; IR (KBr, cm^{-1}):1722.

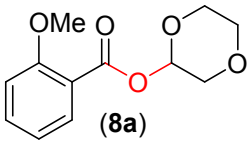
1,4-dioxan-2-yl 4-bromobenzoate



(**7a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66-3.70 (m, 1H), 3.82-3.84 (m, 2H), 3.89-3.90 (m, 2H), 4.17- 4.23 (m, 1H), 6.08 (t, J = 2.0 Hz, 1H), 7.60 (d, J = 8.0 Hz, 2H), 7.98 (d, J = 8.0 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.8, 66.1, 67.7, 90.0, 128.6, 131.4, 131.8, 164.5; ESI-MS ($C_{11}H_{11}{}^{79}BrNaO_4$): 309; ESI-MS ($C_{11}H_{11}{}^{81}BrNaO_4$): 311; IR (KBr, cm^{-1}): 1717.

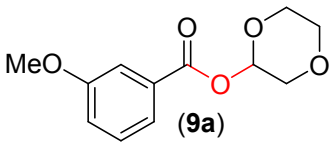
1,4-dioxan-2-yl 2-methoxybenzoate



(**8a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.64-3.68 (m, 1H), 3.79-3.81 (m, 2H), 3.86-3.87(m, 2H), 3.91 (s, 3H), 4.21-4.27 (m, 1H), 6.08 (t, J = 2.0 Hz, 1H), 6.97-6.99(m, 2H), 7.46-7.49 (m, 1H), 7.88-7.90(m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 56.0, 61.8, 66.1, 67.8, 89.6, 112.1, 119.4, 120.1, 131.9, 134.0, 159.6, 164.5; ESI-MS ($C_{12}H_{14}NaO_5$): 261; IR (KBr, cm^{-1}): 1721. Anal. Calcd. for $C_{12}H_{14}O_5$: C, 60.50%; H, 5.92%. Found: C, 60.36%; H, 5.90%.

1,4-dioxan-2-yl 3-methoxybenzoate

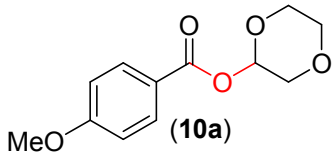


(**9a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.65-3.70 (m, 1H), 3.81-3.83 (m, 2H), 3.85(s, 3H), 3.89-3.90(m, 2H), 4.18-4.25 (m, 1H), 6.09 (t, J = 2.0 Hz, 1H), 7.11-7.14(m, 1H), 7.34-7.38(m, 1H), 7.62-7.63(m, 1H),

7.71-7.74(m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): 55.4, 61.8, 66.1, 67.8, 89.9, 114.4, 119.8, 122.3, 129.4, 131.0, 159.6, 165.1; ESI-MS ($C_{12}H_{14}NaO_5$): 261; IR (KBr, cm^{-1}): 1723. Anal. Calcd. for $C_{12}H_{14}O_5$: C, 60.50%; H, 5.92%. Found: C, 60.41%; H, 5.95%.

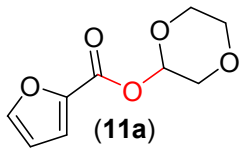
1,4-dioxan-2-yl 4-methoxybenzoate



(**10a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66-3.69 (m, 1H), 3.81-3.83 (m, 2H), 3.87 (s, 3H), 3.87-3.88 (m, 2H), 4.19-4.22 (m, 1H), 6.07 (t, J = 2.0Hz, 1H), 6.93 (d, J = 8.8 Hz, 2H), 8.08 (d, J = 8.8 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 55.4, 61.8, 66.1, 67.9, 89.5, 113.7, 122.0, 132.0, 163.7, 164.9; ESI-MS ($C_{12}H_{14}NaO_5$): 261; IR (KBr, cm^{-1}): 1721.

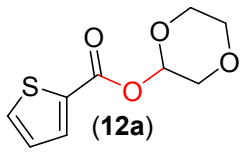
1,4-dioxan-2-yl furan-2-carboxylate



(**11a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.65-3.69 (m, 1H), 3.80-3.87 (m, 4H), 4.17-4.23 (m, 1H), 6.07(t, J = 2.0Hz, 1H), 6.53-6.54 (m, 1H), 7.29-7.30 (m, 1H), 7.62-7.63 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.7, 66.0, 67.7, 89.7, 111.9, 118.9, 144.0, 146.9, 157.2; ESI-MS ($C_9H_{10}NaO_5$): 221; IR (KBr, cm^{-1}): 1730.

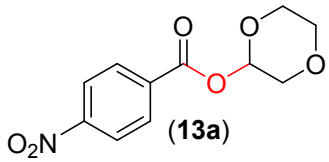
1,4-dioxan-2-yl thiophene-2-carboxylate



(**12a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.65-3.69 (m, 1H), 3.80-3.87 (m, 4H), 4.18-4.22 (m, 1H), 6.05(s, 1H), 7.11-7.13 (m, 1H), 7.60-7.62 (m, 1H), 7.89-7.91 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.8, 66.0, 67.7, 89.9, 127.9, 133.2, 134.2, 160.8; ESI-MS ($C_9H_{10}O_4NaS$): 237; IR (KBr, cm^{-1}): 1718.

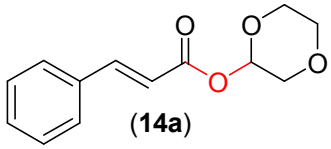
1,4-dioxan-2-yl 4-nitrobenzoate



(**13a**)

^{1}H NMR ($CDCl_3$, 400 MHz): 3.69-3.73 (m, 1H), 3.85-3.88 (m, 2H), 3.93-3.94 (m, 2H), 4.20-4.24 (m, 1H), 6.13 (t, J = 2.0Hz, 1H), 8.31-8.32 (m, 4H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.7, 66.1, 67.6, 90.7, 123.6, 131.0, 135.1, 150.8, 163.4,; ESI-MS ($C_{12}H_{11}NNaO_4$):276. IR (KBr, cm^{-1}): 1731.

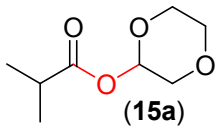
(E)-1,4-dioxan-2-yl cinnamate



(**14a**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.64-3.69 (m, 1H), 3.80-3.85 (m, 4H), 4.16-4.22 (m, 1H), 5.99 (t, J = 2.0 Hz, 1H), 6.52 (d, J = 16.0 Hz, 1H), 7.39-7.40 (m, 3H), 7.53-7.56 (m, 2H), 7.79 (d, J = 16.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 61.7, 66.1, 67.9, 89.3, 117.4, 128.2, 128.9, 130.5, 134.2, 146.1, 165.5; ESI-MS ($C_{13}H_{14}NaO_4$): 257; IR (KBr, cm^{-1}): 1699.

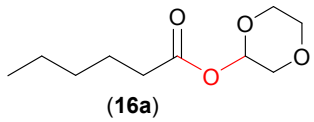
1,4-dioxan-2-yl isobutyrate



(15a)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.20-1.25 (m, 6H), 2.61-2.68(m, 1H), 3.61-3.64(m, 1H), 3.70-3.76(m, 4H) , 4.08-4.13(m, 1H), 5.84(t, J = 2.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 18.7, 18.8, 34.0, 61.7, 66.0, 67.7, 89.0, 175.7; ESI-MS ($C_8H_{14}NaO_4$): 197; IR (KBr, cm^{-1}):1743. Anal. Calcd. for $C_8H_{14}O_4$: C, 55.16%; H, 8.10%. Found: 55.20%; H, 8.08%.

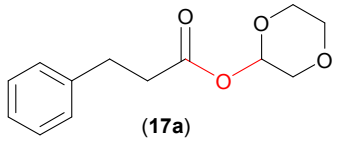
1,4-dioxan-2-yl hexanoate



(16a)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.90 (t, J = 6.8 Hz, 3H), 1.31-1.34(m, 4H), 1.65-1.71(m, 2H), 2.37-2.41(m, 2H) , 3.61-3.65 (m, 1H), 3.69-3.76 (m, 4H), 4.08-4.14 (m, 1H), 5.86 (t, J = 2.0 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.8, 22.2, 24.4, 31.2, 34.3, 61.7, 66.0, 67.8, 89.0, 172.6; ESI-MS ($C_{10}H_{18}NaO_4$): 225; IR (KBr, cm^{-1}):1744. Anal. Calcd. for $C_{10}H_{18}O_4$: C, 59.39%; H, 8.97%. Found: 59.42%; H, 8.94%.

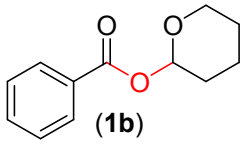
1,4-dioxan-2-yl 3-phenylpropanoate



(17a)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.73 (t, J = 7.6 Hz, 2H), 2.99 (t, J = 7.6 Hz, 2H), 3.56-3.60 (m, 1H), 3.65-3.77 (m, 4H), 3.99-4.05 (m, 1H), 5.84 (t, J = 2.0 Hz, 1H), 7.17-7.22 (m, 3H), 7.27-7.31 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 30.7, 35.8, 61.6, 66.0, 67.7, 89.3, 126.3, 128.3, 128.5, 140.2, 171.7; ESI-MS ($C_{13}H_{16}NaO_4$): 259; IR (KBr, cm^{-1}): 1701.

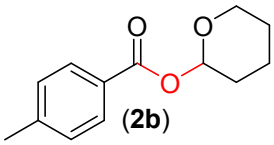
tetrahydro-2H-pyran-2-yl benzoate



(1b)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.64-1.97 (m, 6H), 3.73-3.78 (m, 1H), 3.96-4.02 (m, 1H), 6.25 (t, J = 3.2 Hz, 1H), 7.43-7.47 (m, 2H), 7.57-7.58 (m, 1H), 8.08-8.10 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 18.6, 25.0, 29.2, 63.1, 93.1, 128.4, 129.7, 130.3, 133.1, 165.1; ESI-MS ($C_{12}H_{14}NaO_3$): 229; IR (KBr, cm^{-1}):1725.

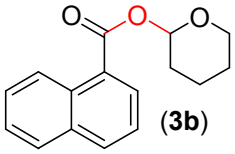
tetrahydro-2H-pyran-2-yl 4-methylbenzoate



(**2b**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.60-1.74 (m, 3H), 1.84-1.94 (m, 3H), 2.40(s, 3H), 3.72-3.76 (m, 1H), 3.96-4.02 (m, 1H), 6.23 (t, J = 3.6 Hz, 1H), 7.24 (d, J = 8.4Hz, 2H), 7.98 (d, J = 8.4Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 18.6, 21.6, 25.0, 29.3, 63.2, 92.9, 127.6, 129.1, 129.7, 143.8, 165.2; ESI-MS ($C_{13}H_{16}NaO_3$): 243; IR (KBr, cm^{-1}):1725. Anal. Calcd. for $C_{13}H_{16}O_3$: C, 70.89%; H, 7.32%. Found: 70.20%; H, 7.28%.

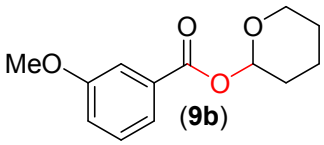
tetrahydro-2H-pyran-2-yl 1-naphthoate



(**3b**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.66-1.97 (m, 6H), 3.77-3.82 (m, 1H), 4.00-4.06 (m, 1H), 6.36(s, 1H), 7.48-7.54 (m, 2H), 7.59-7.63 (m, 1H), 7.86-7.88 (m,1H), 8.01-8.03 (m,1H), 8.25-8.27(m, 1H), 8.98-9.00(m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 18.7, 25.0, 29.3, 63.3, 93.2, 124.4, 125.9, 126.2, 127.0, 127.8, 128.5, 130.4, 131.5, 133.6, 133.8, 166.0; ESI-MS ($C_{16}H_{16}NaO_3$): 279; IR (KBr, cm^{-1}):1725. Anal. Calcd. for $C_{16}H_{16}O_3$: C, 74.98%; H, 6.29%. Found: 74.35%; H, 6.28%.

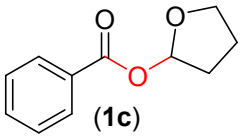
tetrahydro-2H-pyran-2-yl 3-methoxybenzoate



(**9b**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.61-1.76 (m, 3H), 1.85-1.97 (m, 3H), 3.73-3.78 (m, 1H), 3.85(s, 3H), 3.96-3.99 (m, 1H), 6.24 (t, J = 3.2 Hz, 1H), 7.10-7.12 (m, 1H), 7.35-7.37 (m, 1H), 7.60-7.61 (m, 1H),7.67-7.70 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 18.6, 25.0, 29.2, 55.4, 63.2, 93.2, 114.4, 119.3, 122.1, 129.4, 131.6, 159.6, 165.0; ESI-MS ($C_{13}H_{16}NaO_4$): 259; IR (KBr, cm^{-1}): 1726. Anal. Calcd. for $C_{16}H_{16}O_3$: C, 66.09%; H, 6.83%. Found: 66.41%; H, 6.77%.

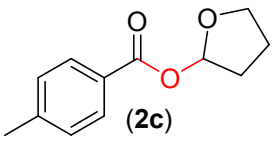
tetrahydrofuran-2-yl benzoate



(**1c**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.96-2.02 (m, 1H), 2.09-2.16 (m, 3H), 3.96-4.01 (m, 1H), 4.10-4.16 (m, 1H), 6.53-6.54 (m, 1H), 7.40-7.44 (m, 2H), 7.52-7.54 (m, 1H), 8.01-8.03 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 22.9, 32.2, 32.3, 69.0, 99.6, 128.3, 129.6, 130.3, 133.0, 165.9; ESI-MS ($C_{11}H_{12}NaO_3$): 215; IR (KBr, cm^{-1}):1721.

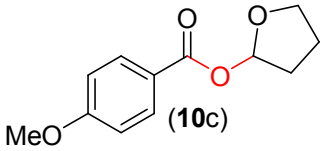
tetrahydrofuran-2-yl 4-methylbenzoate



(**2c**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.97-2.01 (m, 1H), 2.11-2.16 (m, 3H), 2.39 (s, 3H), 3.98-4.02 (m, 1H), 4.12-4.16 (m, 1H), 6.52-6.54 (m, 1H), 7.22 (d, J = 8.4Hz, 2H), 7.91 (d, J = 8.4Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.6, 22.9, 32.2, 68.9, 99.5, 129.0, 129.7, 130.2, 143.7, 166.0; ESI-HRMS: calcd. for $C_{12}H_{14}NaO_3$, 229.0841; found, 229.0842; IR (KBr, cm^{-1}):1721.

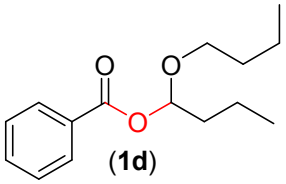
tetrahydrofuran-2-yl 4-methoxybenzoate



(**10c**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 1.98-2.03 (m, 1H), 2.09-2.17 (m, 3H), 3.85 (s, 3H), 3.98-4.01 (m, 1H), 4.10-4.16 (m, 1H), 6.51-6.52 (m, 1H), 6.90 (d, J = 9.2Hz, 2H), 7.98 (d, J = 9.2Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 23.0, 32.2, 55.4, 68.9, 99.3, 113.5, 131.7, 163.4, 165.6; ESI-HRMS: calcd. for $C_{12}H_{14}NaO_4$, 245.0790; found, 245.0794; IR (KBr, cm^{-1}):1722.

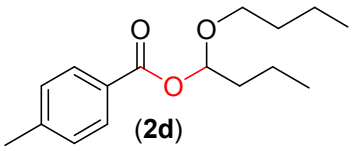
1-butoxybutyl benzoate



(**1d**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.90 (t, J = 7.2 Hz, 3H), 0.96 (t, J = 7.2 Hz, 3H), 1.35-1.40 (m,2H), 1.44-1.51(m, 2H), 1.55-1.59(m, 2H),1.78-1.84 (m, 2H), 3.53-3.58 (m, 1H), 3.70-3.76 (m, 1H), 6.09 (t, J = 5.2 Hz, 1H), 7.42-7.45 (m, 2H), 7.55-7.58 (m, 1H), 8.07-8.09 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.7, 13.8, 17.4, 19.1, 26.5, 31.6, 36.7, 69.3, 99.5, 128.3, 129.7, 130.2, 133.0, 166.3; ESI-MS ($C_{15}H_{22}NaO_3$): 273; IR (KBr, cm^{-1}):1720.

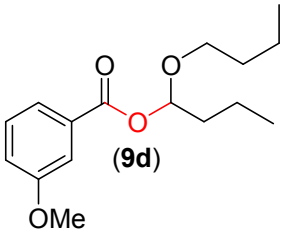
1-butoxybutyl 4-methylbenzoate



(**2d**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.90 (t, J = 7.2 Hz, 3H), 0.95 (t, J = 7.2 Hz, 3H), 1.38-1.53 (m,6H), 1.77-1.81 (m, 2H), 2.41 (s, 3H), 3.52-3.57 (m, 1H), 3.69-3.75 (m, 1H), 6.08 (t, J = 5.6 Hz, 1H), 7.24 (d, J =8.0Hz, 2H), 7.97 (d, J = 8.0Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.7, 17.4, 19.1, 21.6, 31.6, 36.7, 69.2, 99.2, 127.4, 129.0, 129.7, 143.7, 166.4; ESI-HRMS: calcd. for $C_{16}H_{24}NaO_3$, 287.1623; found, 287.1630; IR (KBr, cm^{-1}):1722.

1-butoxybutyl 3-methoxybenzoate

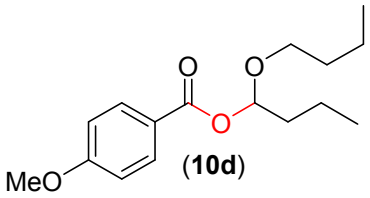


(**9d**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.90 (t, J = 7.6 Hz, 3H), 0.96 (t, J = 7.6 Hz, 3H), 1.39-1.59 (m,6H), 1.78-1.84 (m,2H), 3.53-3.58 (m, 1H), 3.70-3.76 (m, 1H), 3.85 (s, 3H), 6.09 (t, J = 5.6 Hz, 1H), 7.09-7.12

(m, 1H), 7.33-7.37 (m, 1H), 7.59-7.60 (m, 1H), 7.33-7.37 (m, 1H), 7.67-7.69 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.7, 17.4, 19.1, 31.6, 36.7, 55.3, 69.3, 99.5, 114.1, 119.6, 122.1, 129.3, 131.5, 159.5, 166.2; ESI-HRMS: calcd. for $C_{16}H_{24}NaO_4$, 303.1573; found, 303.1570; IR (KBr, cm^{-1}):1721.

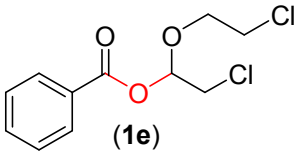
1-butoxybutyl 4-methoxybenzoate



(**10d**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 0.90 (t, J = 7.2 Hz, 3H), 0.95 (t, J = 7.2 Hz, 3H), 1.34-1.40 (m,2H), 1.43-1.50 (m, 2H), 1.54-1.58 (m, 2H),1.77-1.82 (m, 2H), 3.52-3.57 (m, 1H), 3.69-3.75 (m, 1H), 3.85 (s, 3H), 6.07 (t, J = 5.6 Hz, 1H), 6.92 (d, J =8.8Hz, 2H), 8.03 (d, J = 8.8Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 13.7, 13.8, 17.4, 19.1, 31.6, 36.7, 55.3, 69.1, 99.0, 113.6, 122.5, 131.7, 163.5, 166.0; ESI-HRMS: calcd. for $C_{16}H_{24}NaO_4$, 303.1573; found, 303.1576; IR (KBr, cm^{-1}):1721.

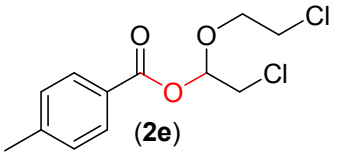
1-(2-chloroethoxy)-2-chloroethyl benzoate



(**1e**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.65 (t, J = 5.6 Hz, 2H), 3.75 (t, J = 5.6 Hz, 2H), 3.97-4.02 (m, 1H), 4.06-4.08 (m, 1H), 6.20 (t, J = 5.2 Hz, 1H), 7.45-7.49 (m, 2H), 7.60-7.62 (m, 1H), 8.07-8.09 (m,2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 42.3, 43.7, 70.4, 96.7, 128.5, 129.0, 129.9, 133.7, 165.8; ESI-MS ($C_{11}H_{16}{}^{35}Cl_2NO_3$, M + NH_4): 280; ESI-MS ($C_{11}H_{16}{}^{37}Cl_2NO_3$, M + NH_4): 282; IR (KBr, cm^{-1}):1652.

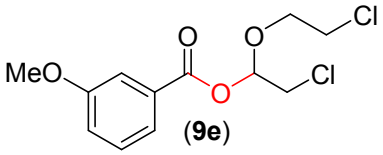
1-(2-chloroethoxy)-2-chloroethyl 4-methylbenzoate



(**2e**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 2.42 (s, 3H), 3.65 (t, J = 5.2 Hz, 2H), 3.74 (t, J = 5.2 Hz, 2H), 3.96-4.02 (m, 1H), 4.05-4.08 (m, 1H), 6.18 (t, J = 5.2 Hz, 1H), 7.26 (d, J = 8.4Hz, 2H), 7.96 (d, J = 8.4Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 21.7, 29.7, 42.3, 43.8, 70.4, 96.5, 126.2, 129.2, 130.0, 144.6, 165.9; ESI-HRMS ($C_{12}H_{18}Cl_2NO_3$, M + NH_4): calcd. 294.0664 (^{35}Cl), 296.0634 (^{37}Cl); found, 294.0651 (35Cl), 296.0642 (^{37}Cl); IR (KBr, cm^{-1}):1653.

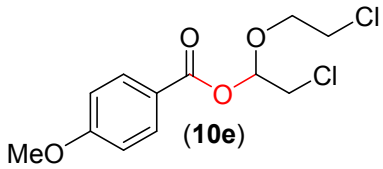
1-(2-chloroethoxy)-2-chloroethyl 3-methoxybenzoate



(**9e**)

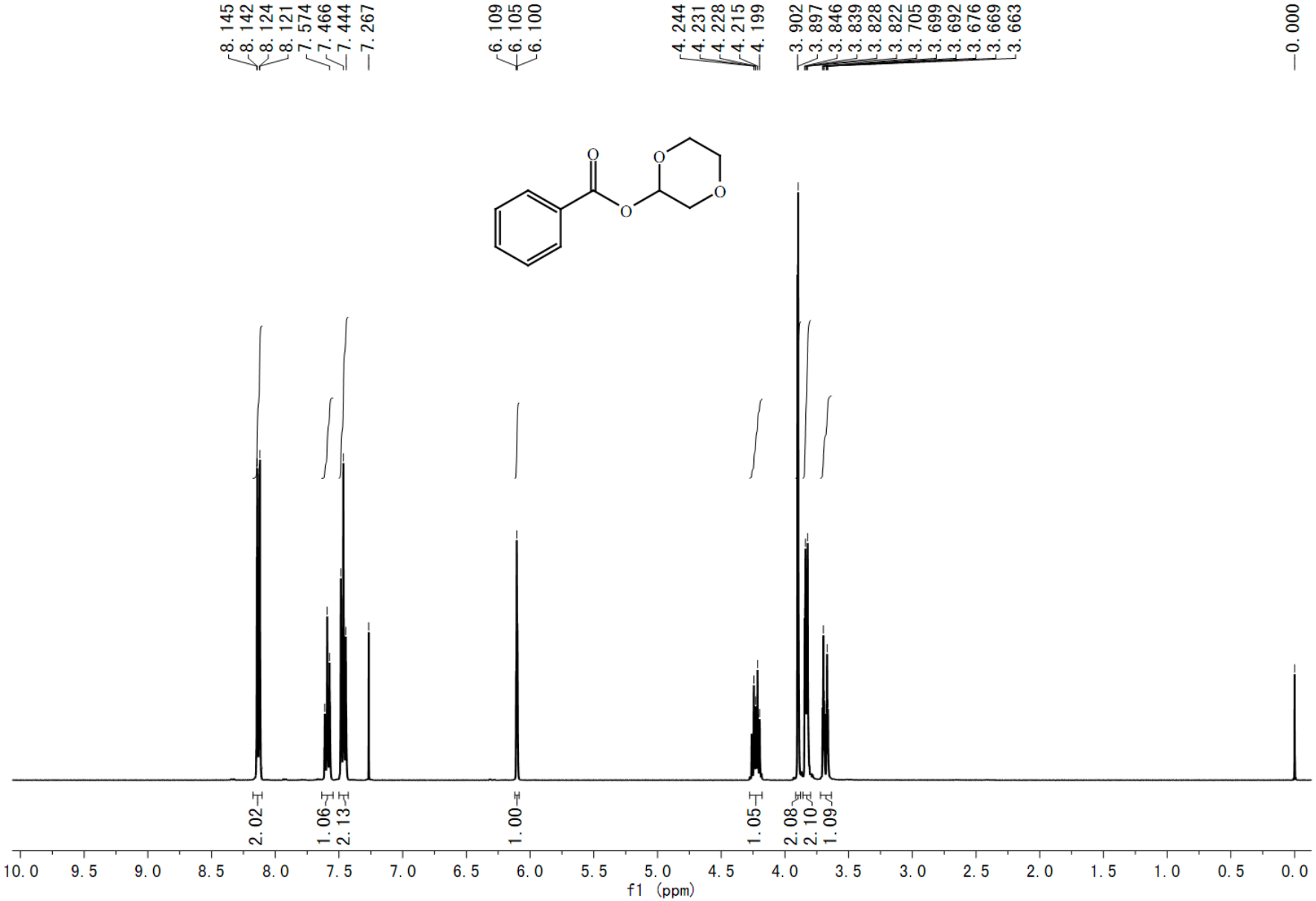
^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.66 (t, J = 5.2 Hz, 2H), 3.76 (t, J = 5.2 Hz, 2H), 3.86 (s, 3H), 3.97-4.03 (m, 1H), 4.06-4.11 (m, 1H), 6.19 (t, J = 5.2 Hz, 1H), 7.13-7.15 (m, 1H), 7.37-7.39 (m, 1H), 7.58-7.59 (m, 1H), 7.66-7.69 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 42.4, 43.7, 55.5, 70.4, 96.8, 114.4, 120.2, 122.3, 129.6, 159.6, 165.7; ESI-HRMS ($C_{12}H_{18}Cl_2NO_4$, M + NH_4): calcd. 310.0613 (^{35}Cl), 312.0584 (^{37}Cl); found, 310.0604 (^{35}Cl), 312.0592 (^{37}Cl); IR (KBr, cm^{-1}):1652.

1-(2-chloroethoxy)-2-chloroethyl 4-methoxybenzoate



(**10e**)

^{1}H NMR ($CDCl_3$, 400 MHz): δ 3.65 (t, J = 5.2 Hz, 2H), 3.74 (t, J = 5.2 Hz, 2H), 3.87 (s, 3H), 3.96-4.00(m, 1H), 4.05-4.09 (m, 1H), 6.17 (t, J = 5.2 Hz, 1H), 6.94 (d, J =8.8Hz, 2H), 8.03 (d, J = 8.8Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz): δ 42.4, 43.8, 55.5, 70.3, 96.4, 113.8, 121.2, 132.1, 164.0, 165.5; ESI-HRMS ($C_{12}H_{18}Cl_2NO_4$, M + NH_4): calcd. 310.0613 (^{35}Cl), 312.0584 (^{37}Cl); found, 310.0608 (^{35}Cl), 312.0577 (^{37}Cl); IR (KBr, cm^{-1}):1653.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7.466
7.444
7.267
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6.105
6.100
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4.231
4.228
4.215
4.199
3.902
3.897
3.846
3.839
3.828
3.822
3.705
3.699
3.692
3.676
3.669
3.663
0.000
O
O
O
O
2.02
1.06
2.13
1.00
1.05
2.08
2.10
1.09
10.0
9.5
9.0
8.5
8.0
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6.0
5.5
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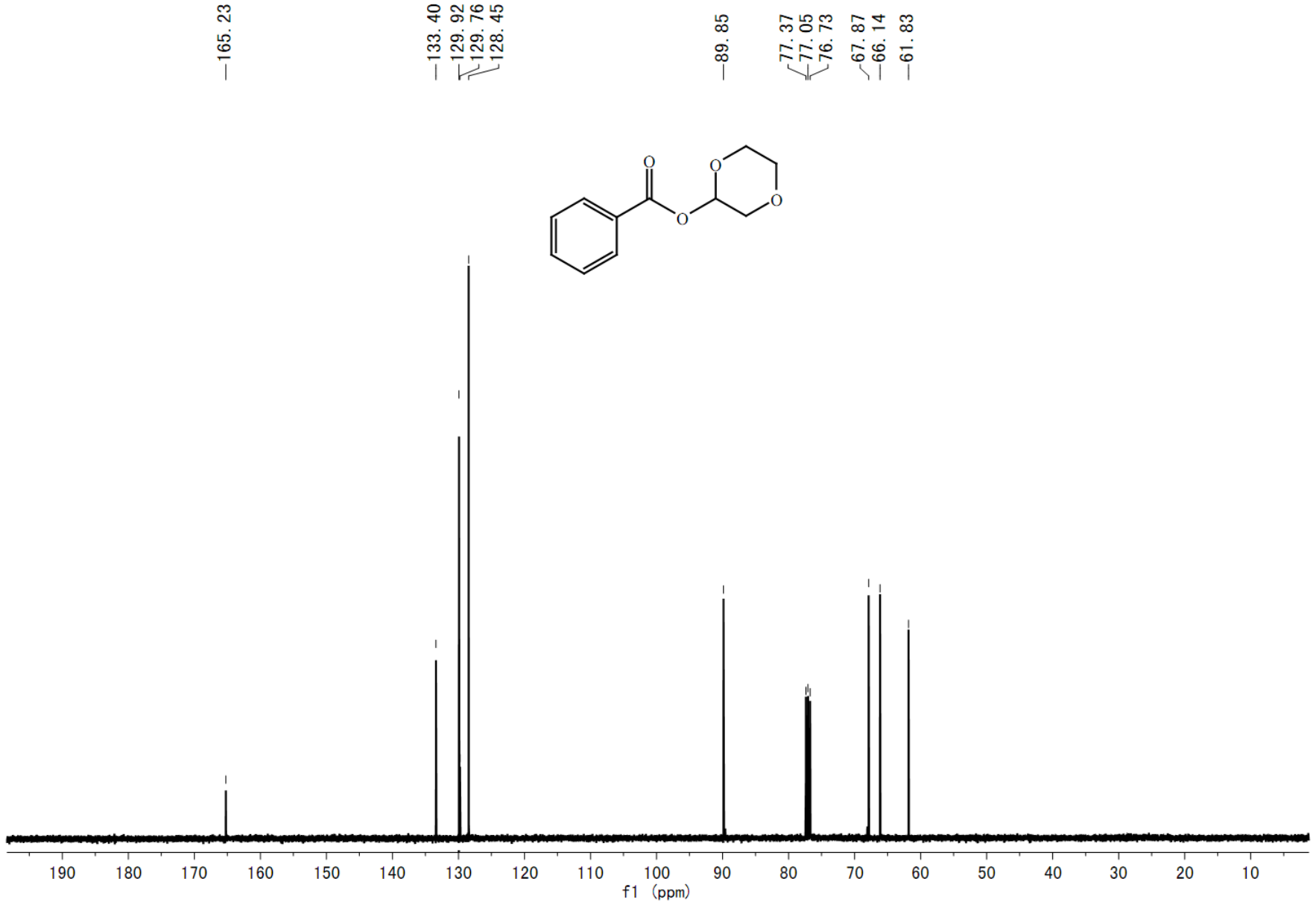
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129.92
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77.37
77.05
76.73
67.87
66.14
61.83
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O
O
O
190
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120
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60
50
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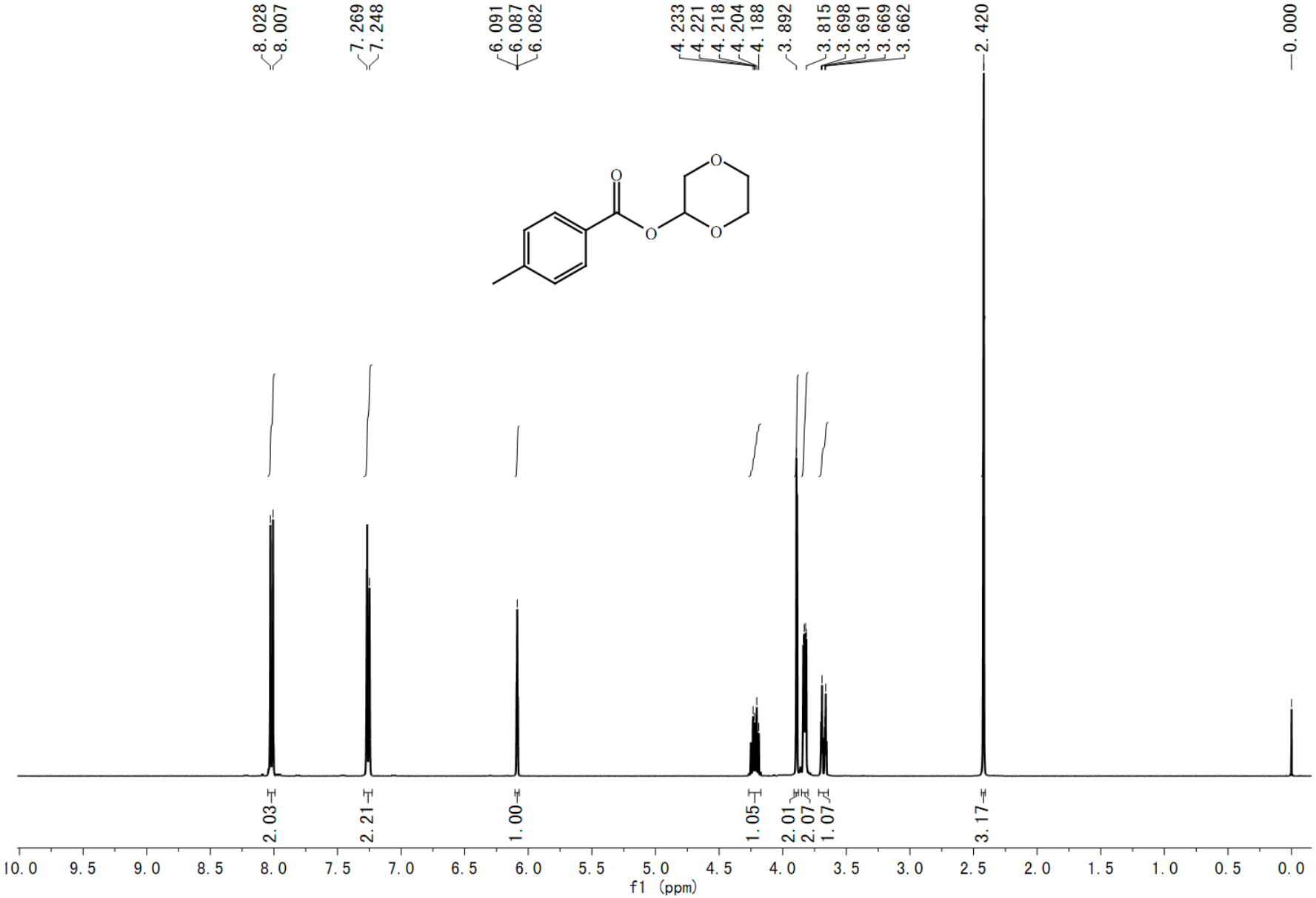
30
20
10
f1 (ppm)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4.204
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3.691
3.669
3.662
2.420
0.000
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O
O
O
2.03
2.21
1.00
1.05
2.01
2.07
1.07
3.17
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
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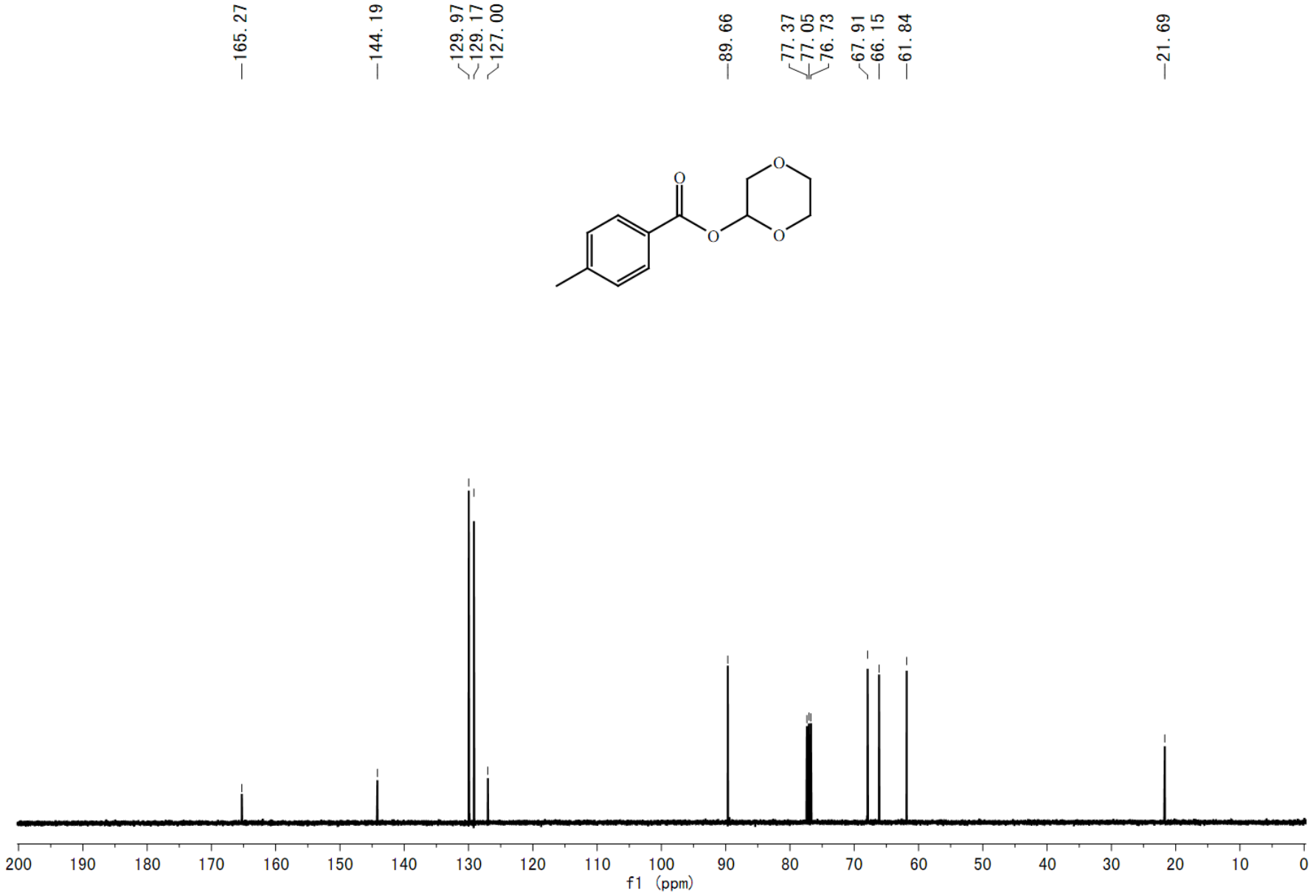
1.0
0.5
0.0
f1 (ppm)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127.00
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67.91
66.15
61.84
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190
180
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130
120
110
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90
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50
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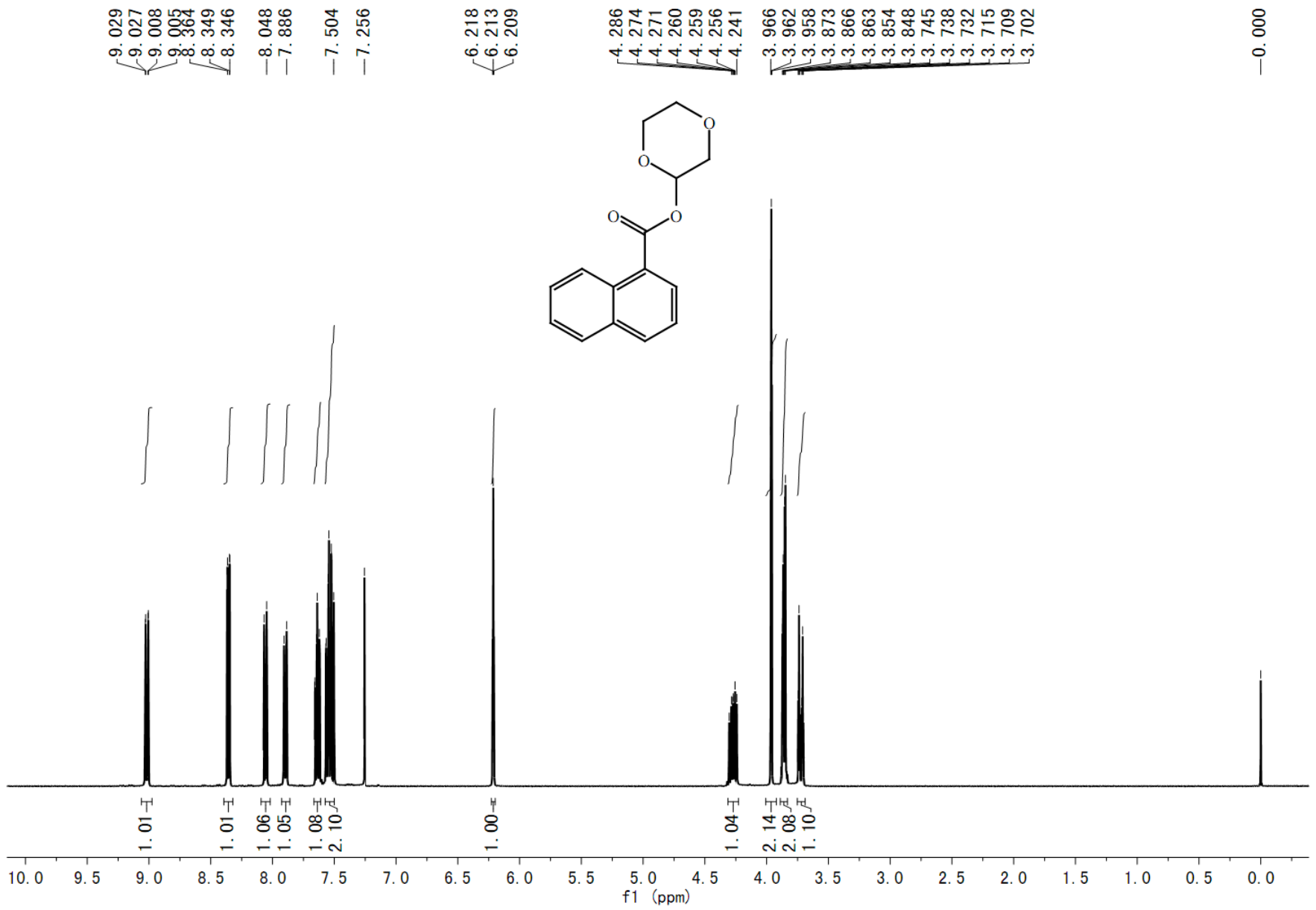
10
0
f1 (ppm)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6.209
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3.962
3.958
3.873
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3.863
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3.848
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3.732
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3.709
3.702
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O
O
O
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1.01
1.06
1.05
1.08
2.10
1.00
1.04
2.14
2.08
1.10
10.0
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7.5
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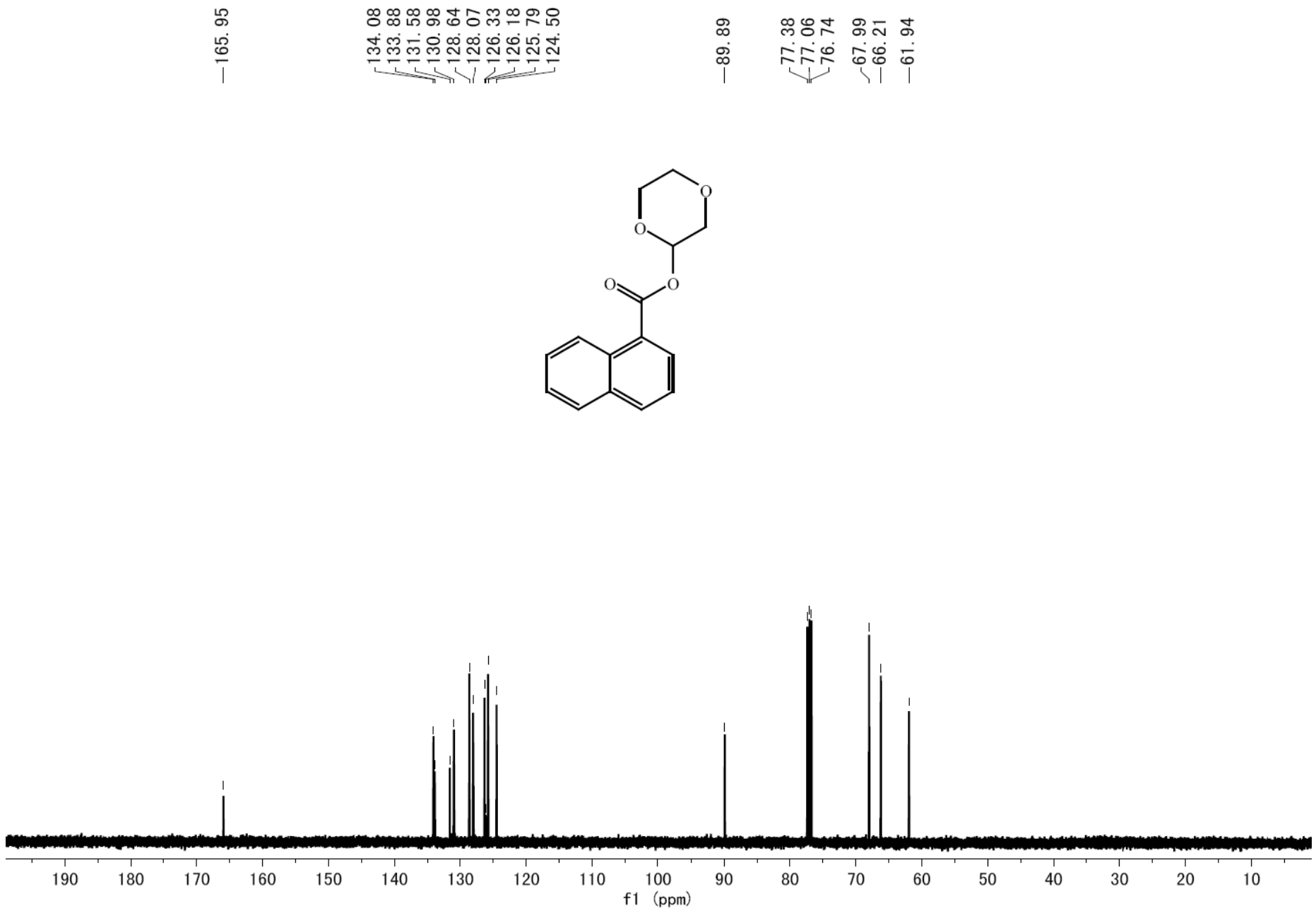
0.0
f1 (ppm)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77.06
76.74
67.99
66.21
61.94
O
O
O
O
190
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120
110
100
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60
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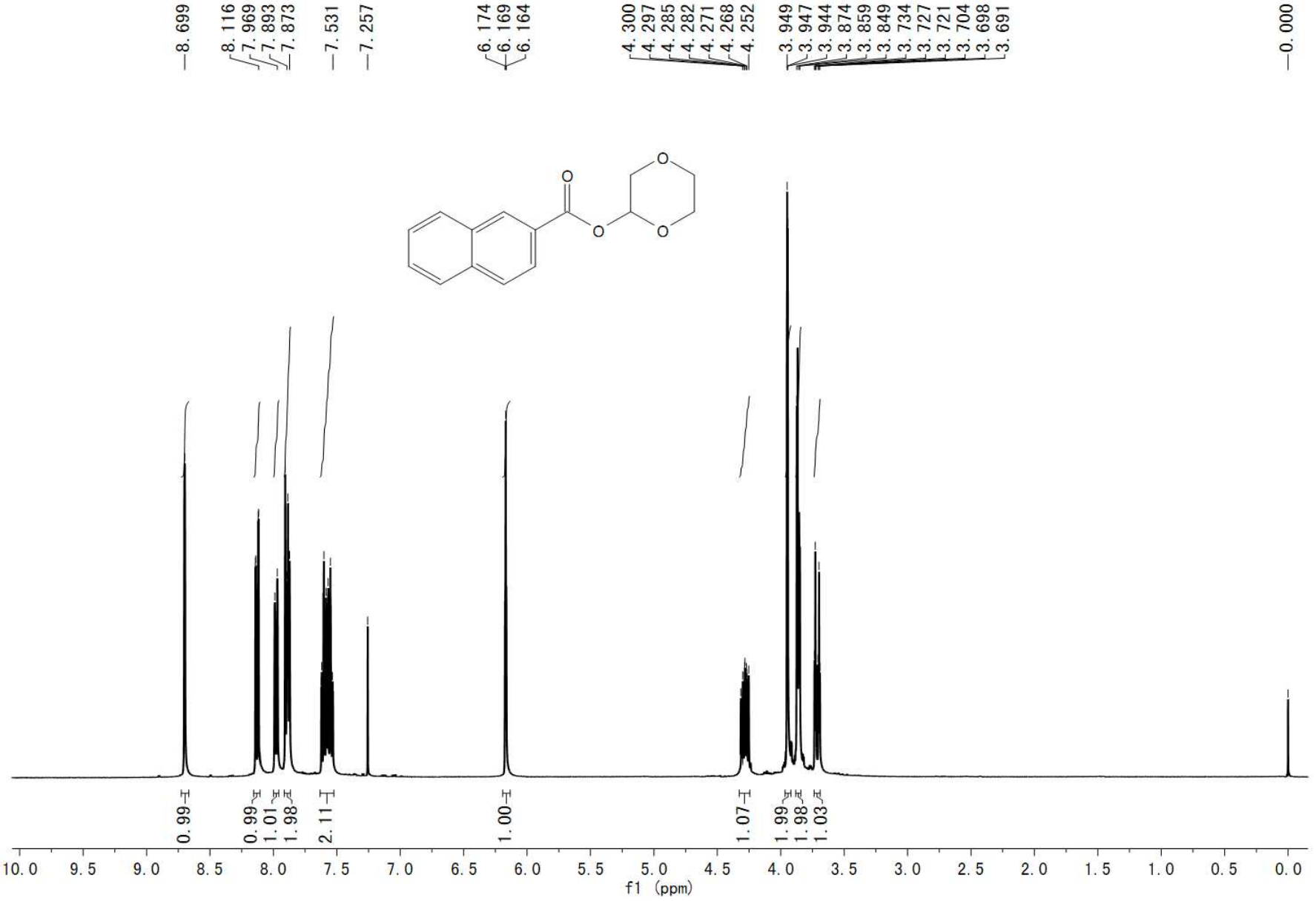
20
10
f1 (ppm)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6.164
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4.268
4.252
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3.947
3.944
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3.859
3.849
3.734
3.727
3.721
3.704
3.698
3.691
0.000
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O
O
O
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0.99
1.01
1.98
2.11
1.00
1.07
1.99
1.98
1.03
10.0
9.5
9.0
8.5
8.0
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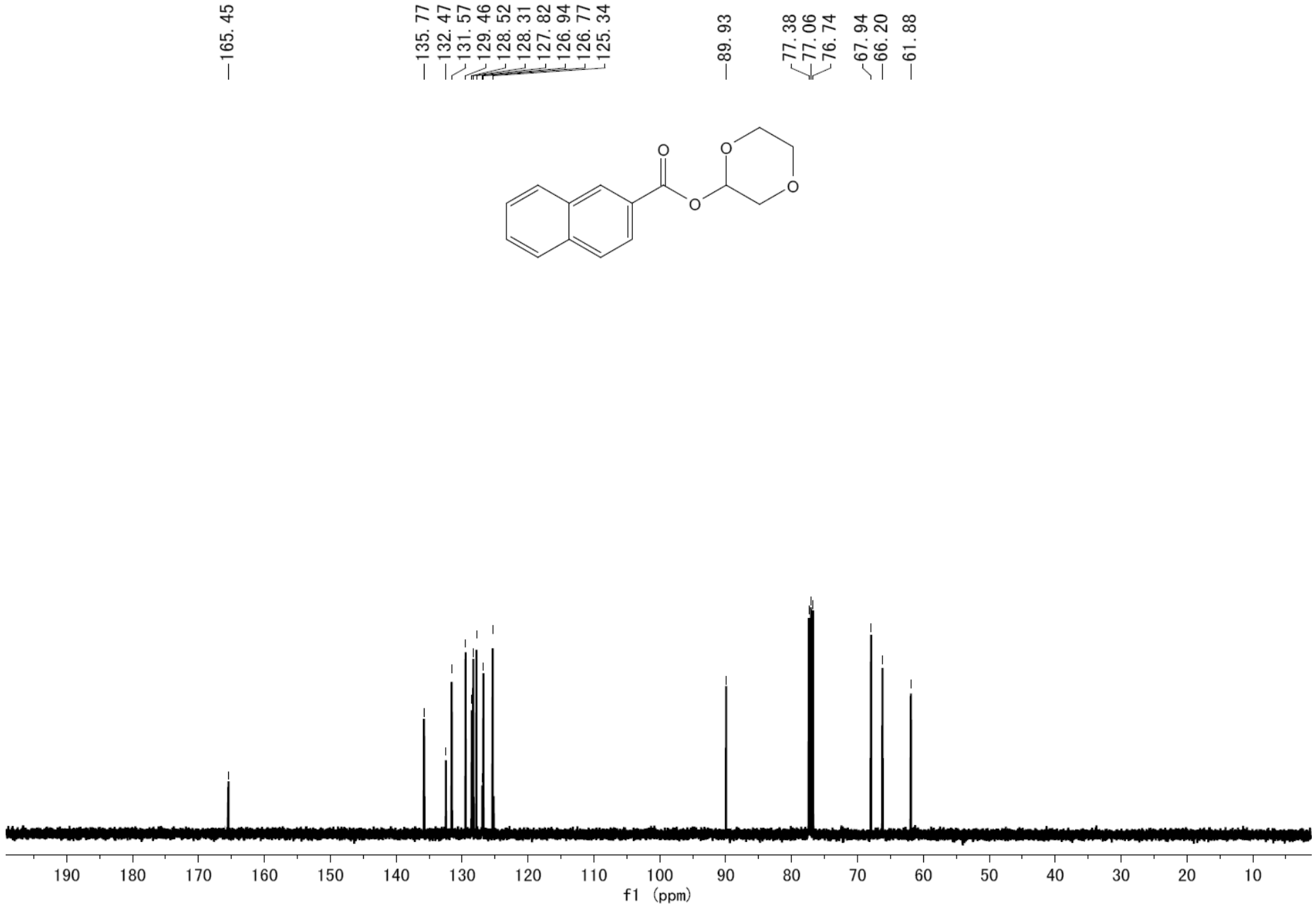
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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128.31
127.82
126.94
126.77
125.34
89.93
77.38
77.06
76.74
67.94
66.20
61.88
O
O
O
O
190
180
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160
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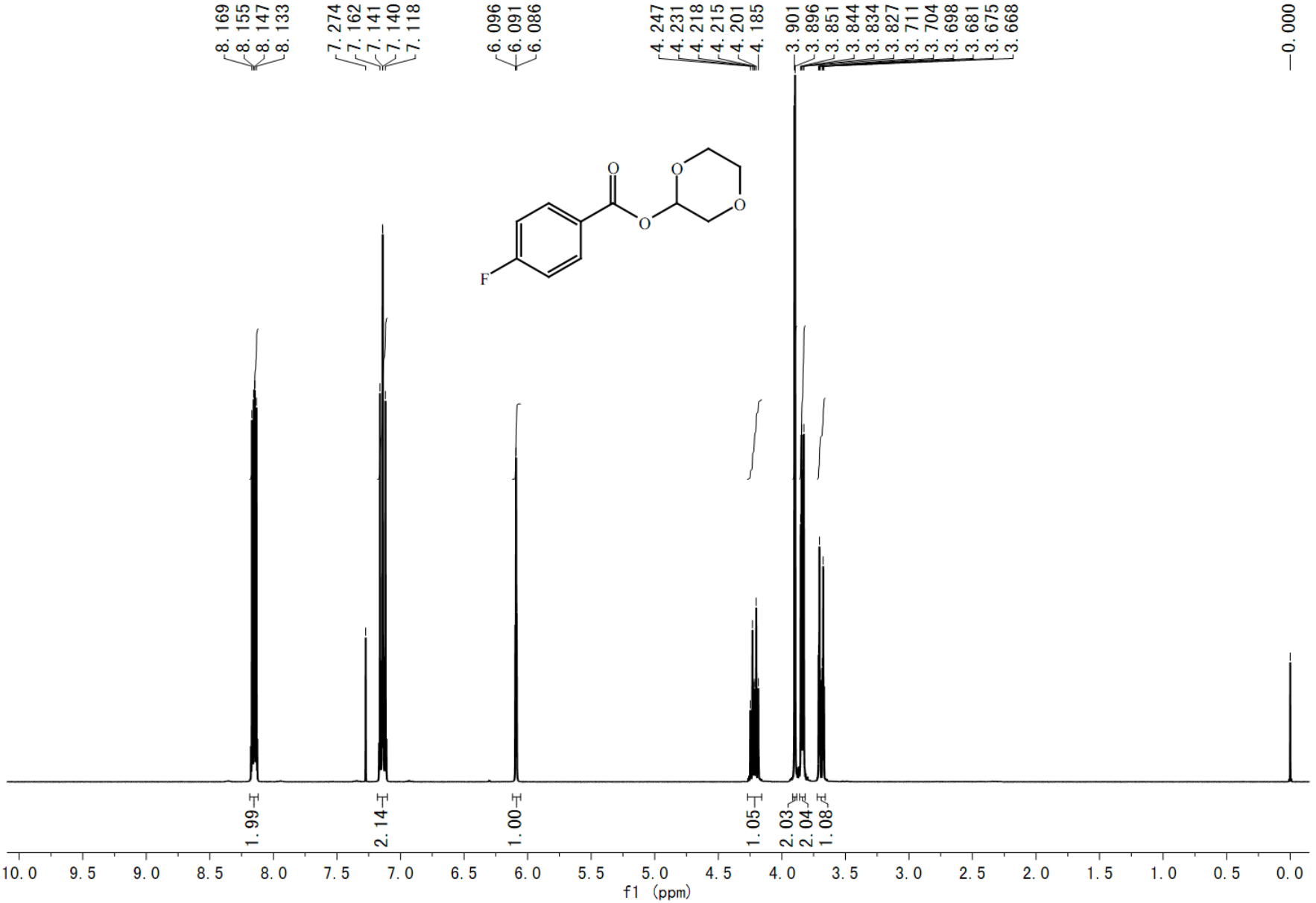
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.140
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6.091
6.086
4.247
4.231
4.218
4.215
4.201
4.185
3.901
3.896
3.851
3.844
3.834
3.827
3.711
3.704
3.698
3.681
3.675
3.668
0.000
O
O
O
O
F
1.99
2.14
1.00
1.05
2.03
2.04
1.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
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3.0
2.5
2.0
1.5
1.0
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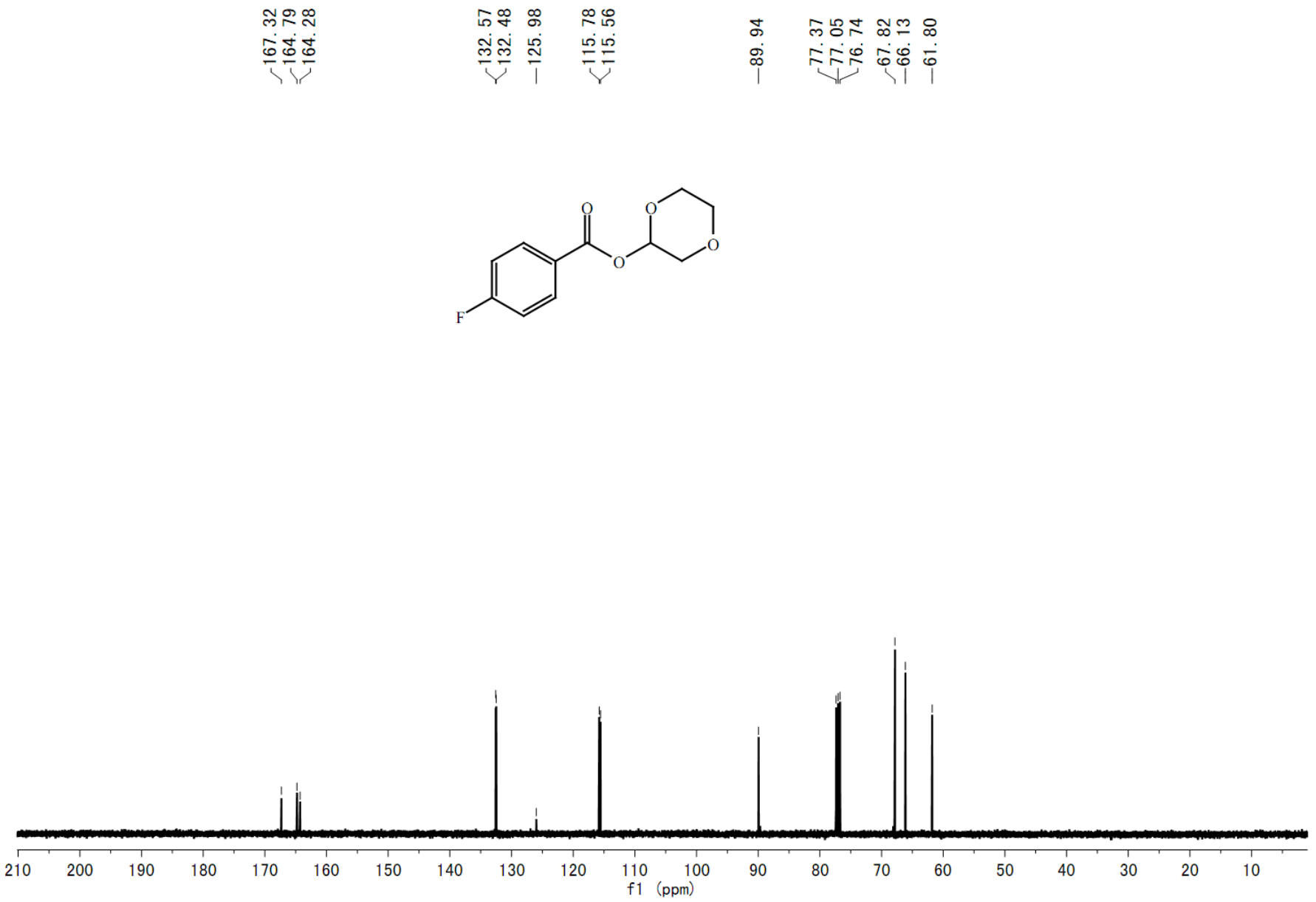
0.0
f1 (ppm)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115.56
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77.37
77.05
76.74
67.82
66.13
61.80
O
O
O
O
F
210
200
190
180
170
160
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140
130
120
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100
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80
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60
50
40
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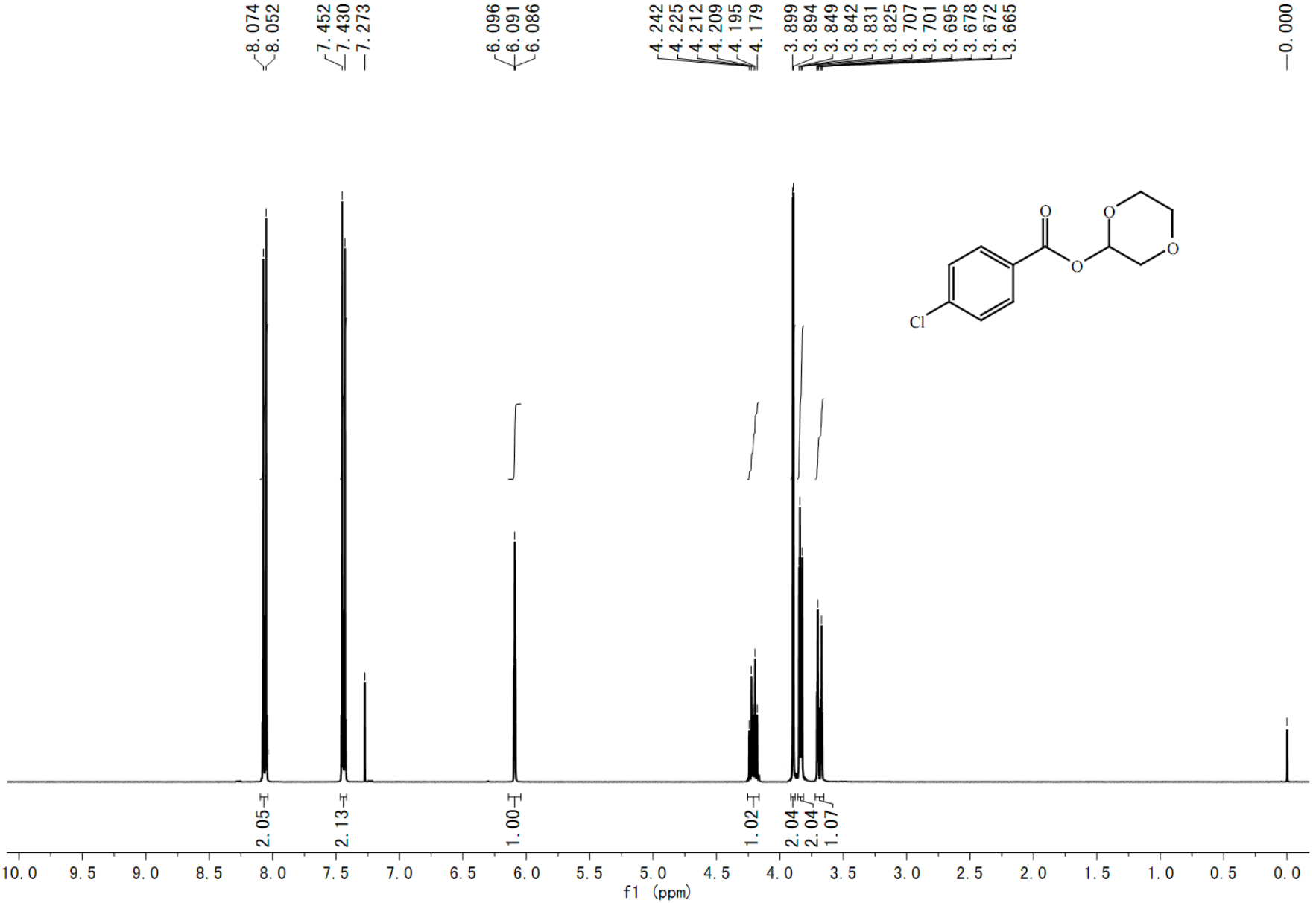
20
10
f1 (ppm)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6.096
6.091
6.086
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4.225
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4.209
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4.179
3.899
3.894
3.849
3.842
3.831
3.825
3.707
3.701
3.695
3.678
3.672
3.665
0.000
O
O
O
O
Cl
2.05
2.13
1.00
1.02
2.04
2.04
1.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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1.0
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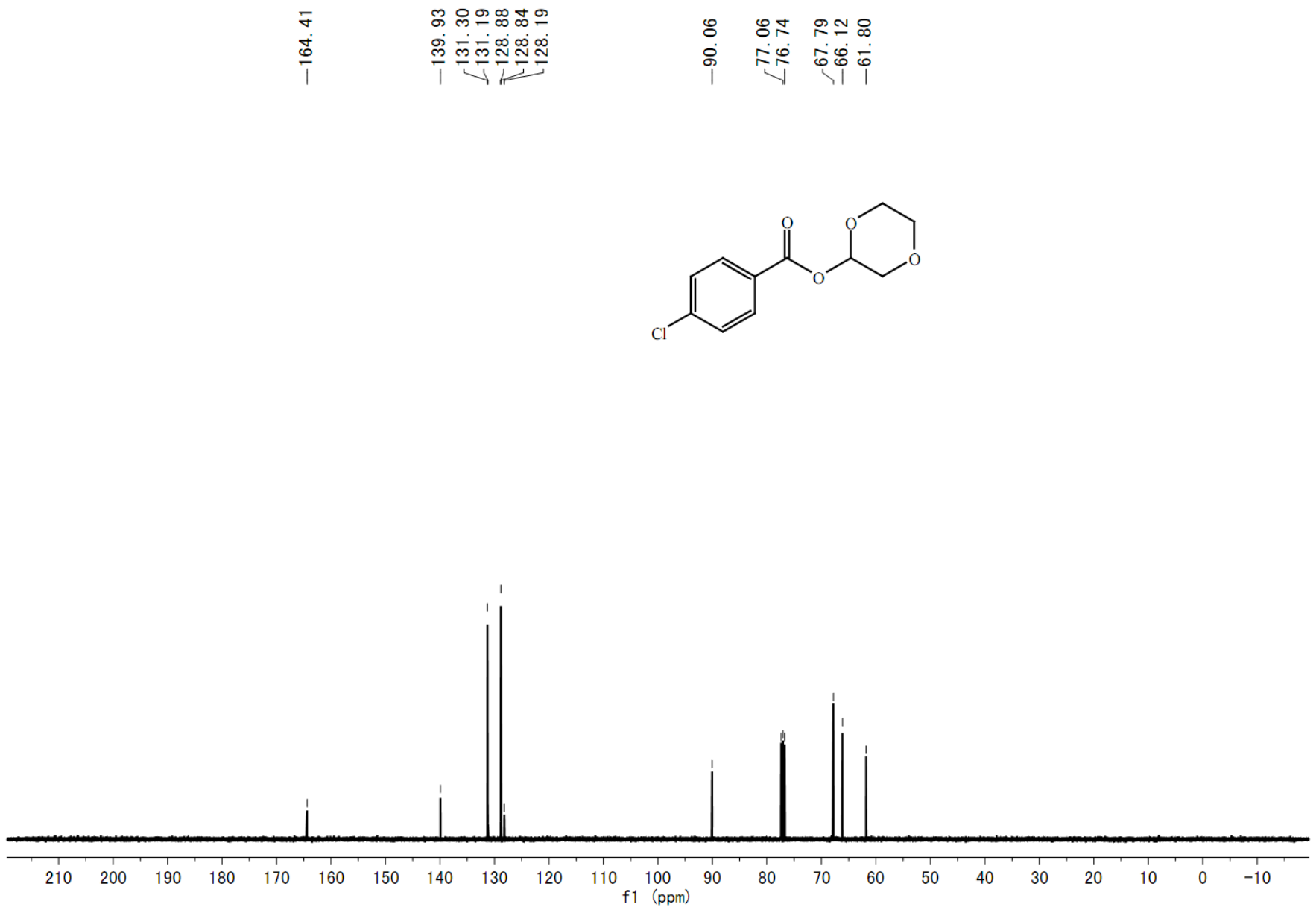
f1 (ppm)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139.93
131.30
131.19
128.88
128.84
128.19
90.06
77.06
76.74
67.79
66.12
61.80
O
O
O
O
Cl
210
200
190
180
170
160
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120
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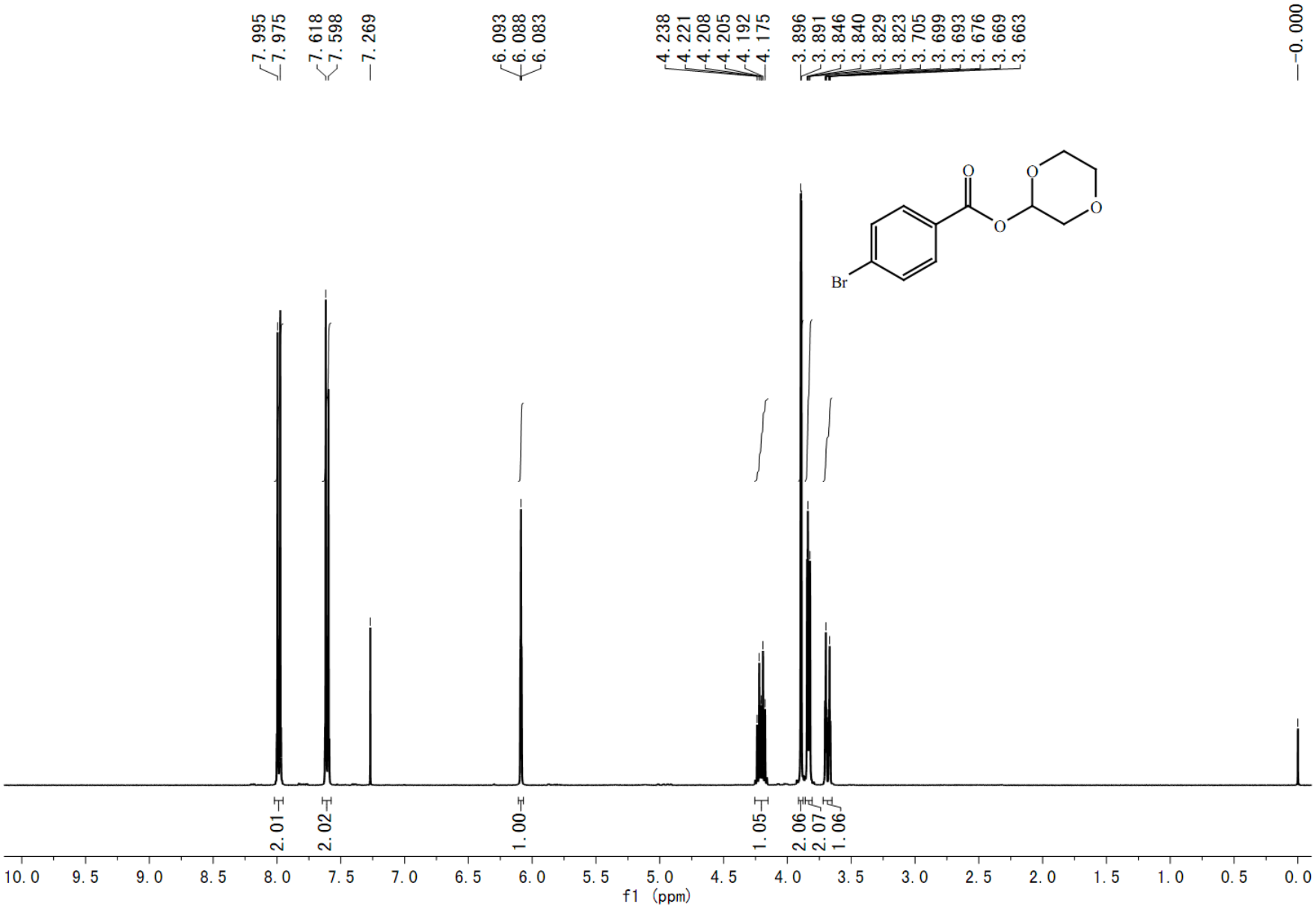
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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6.083
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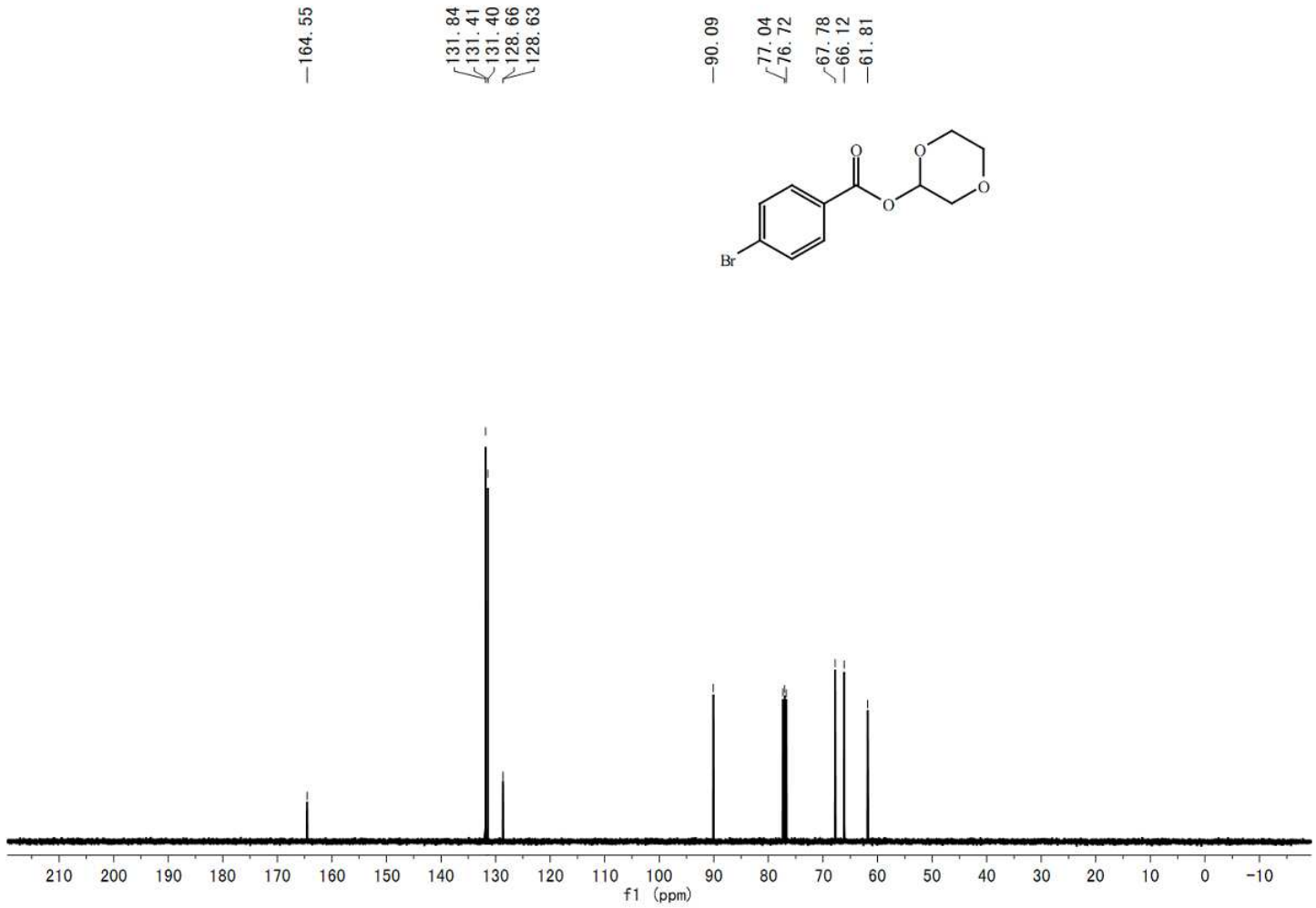
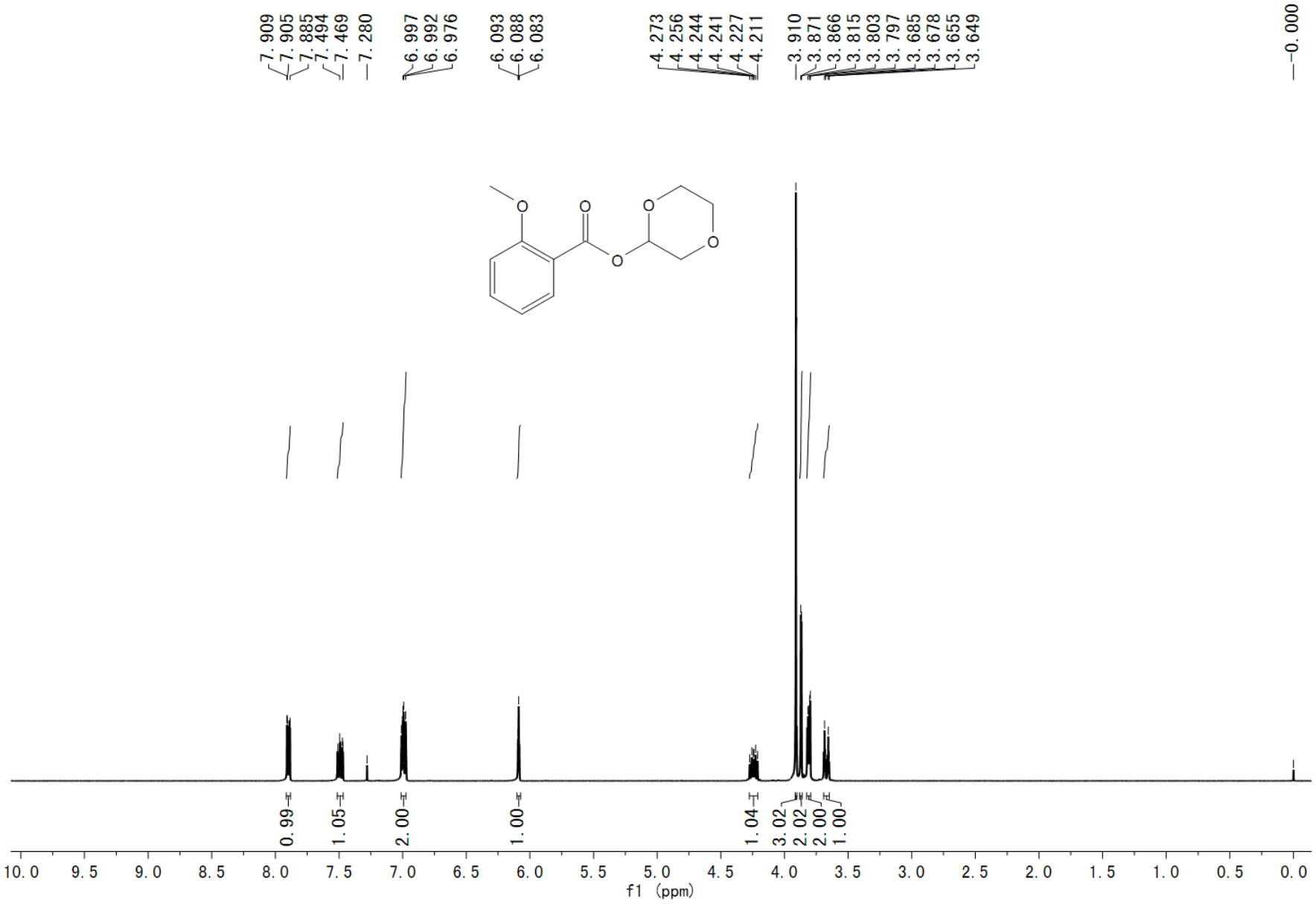
4.208
4.205
4.192
4.175
3.896
3.891
3.846
3.840
3.829
3.823
3.705
3.699
3.693
3.676
3.669
3.663
0.000
O
O
O
O
Br
2.01
2.02
1.00
1.05
2.06
2.07
1.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


164.55
131.84
131.41
131.40
128.66
128.63
90.09
77.04
76.72
67.78
66.12
61.81
O
O
O
O
Br
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.885
7.494
7.469
7.280
6.997
6.992
6.976
6.093
6.088
6.083
4.273
4.256
4.244
4.241
4.227
4.211
3.910
3.871
3.866
3.815
3.803
3.797
3.685
3.678
3.655
3.649
-0.000
O
O
O
O
O
0.99
1.05
2.00
1.00
1.04
3.02
2.02
2.00
1.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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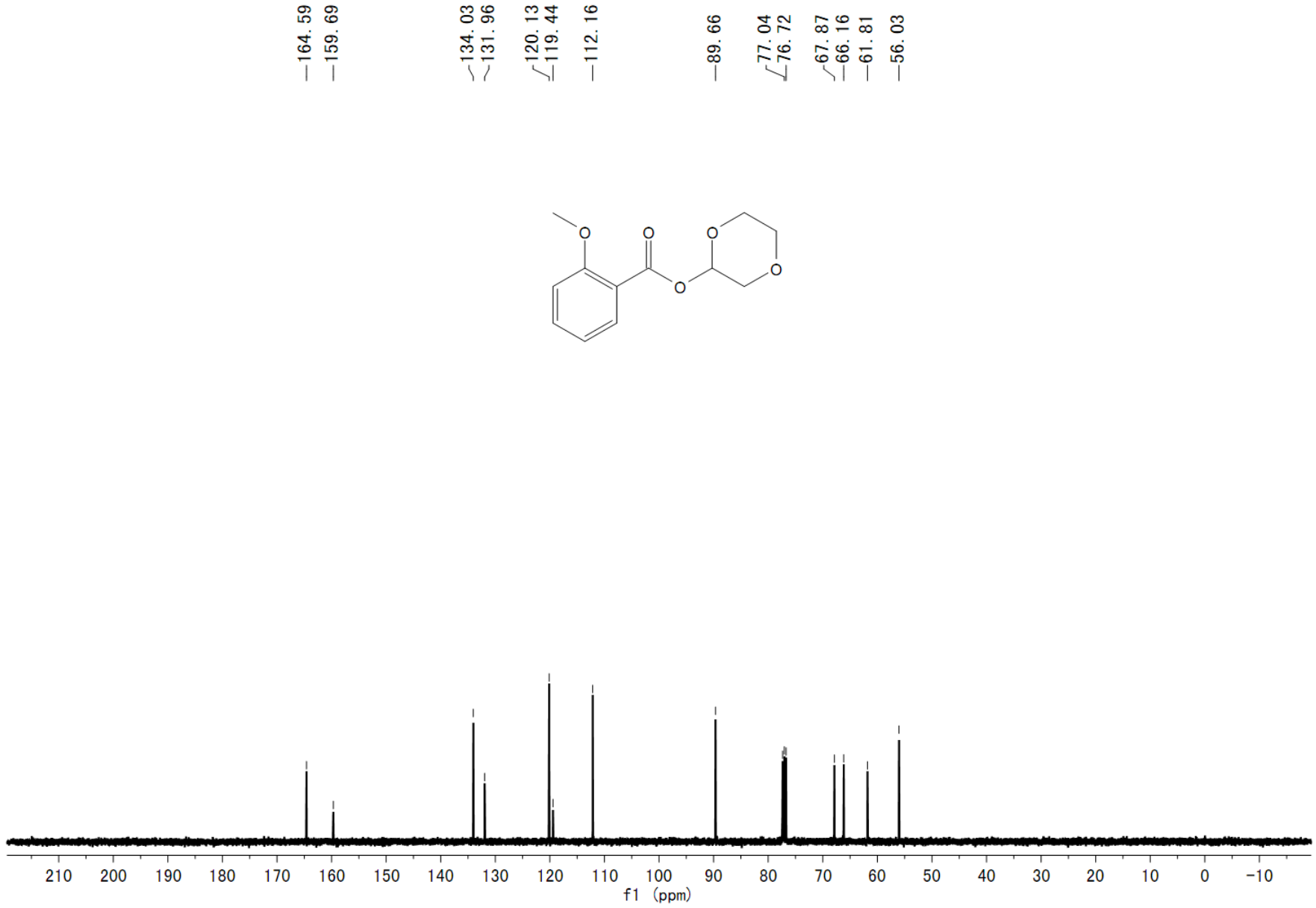
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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159.69
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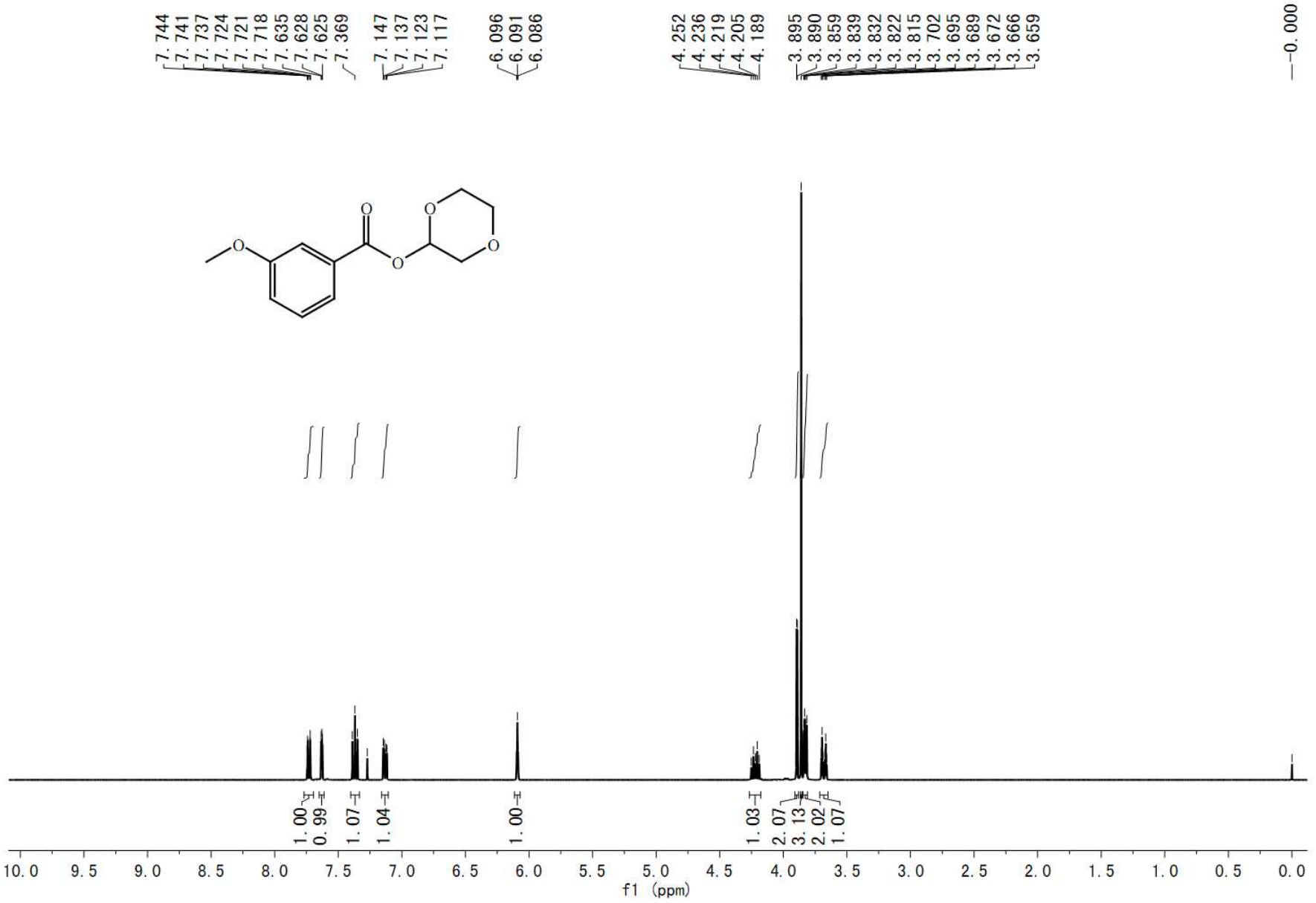
131.96
120.13
119.44
112.16
89.66
77.04
76.72
67.87
66.16
61.81
56.03
O
O
O
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.721
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7.628
7.625
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7.147
7.137
7.123
7.117
6.096
6.091
6.086
4.252
4.236
4.219
4.205
4.189
3.895
3.890
3.859
3.839
3.832
3.822
3.815
3.702
3.695
3.689
3.672
3.666
3.659
0.000
1.00
0.99
1.07
1.04
1.00
1.03
2.07
3.13
2.02
1.07
10.0
9.5
9.0
8.5
8.0
7.5
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6.5
6.0
5.5
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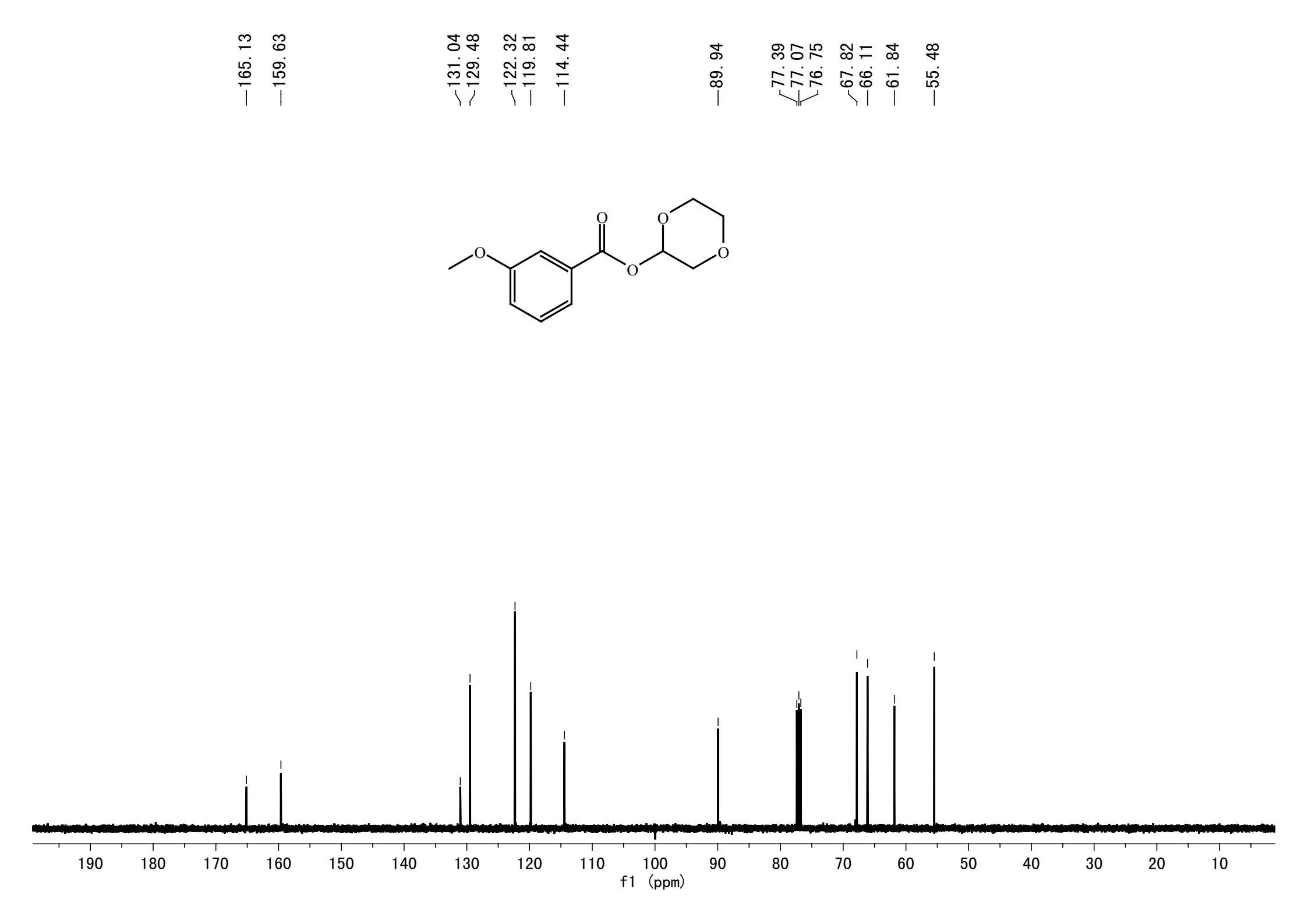
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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119.81
114.44
89.94
77.39
77.07
76.75
67.82
66.11
61.84
55.48
O
O
O
O
O
190
180
170
160
150
140
130
120
110
100
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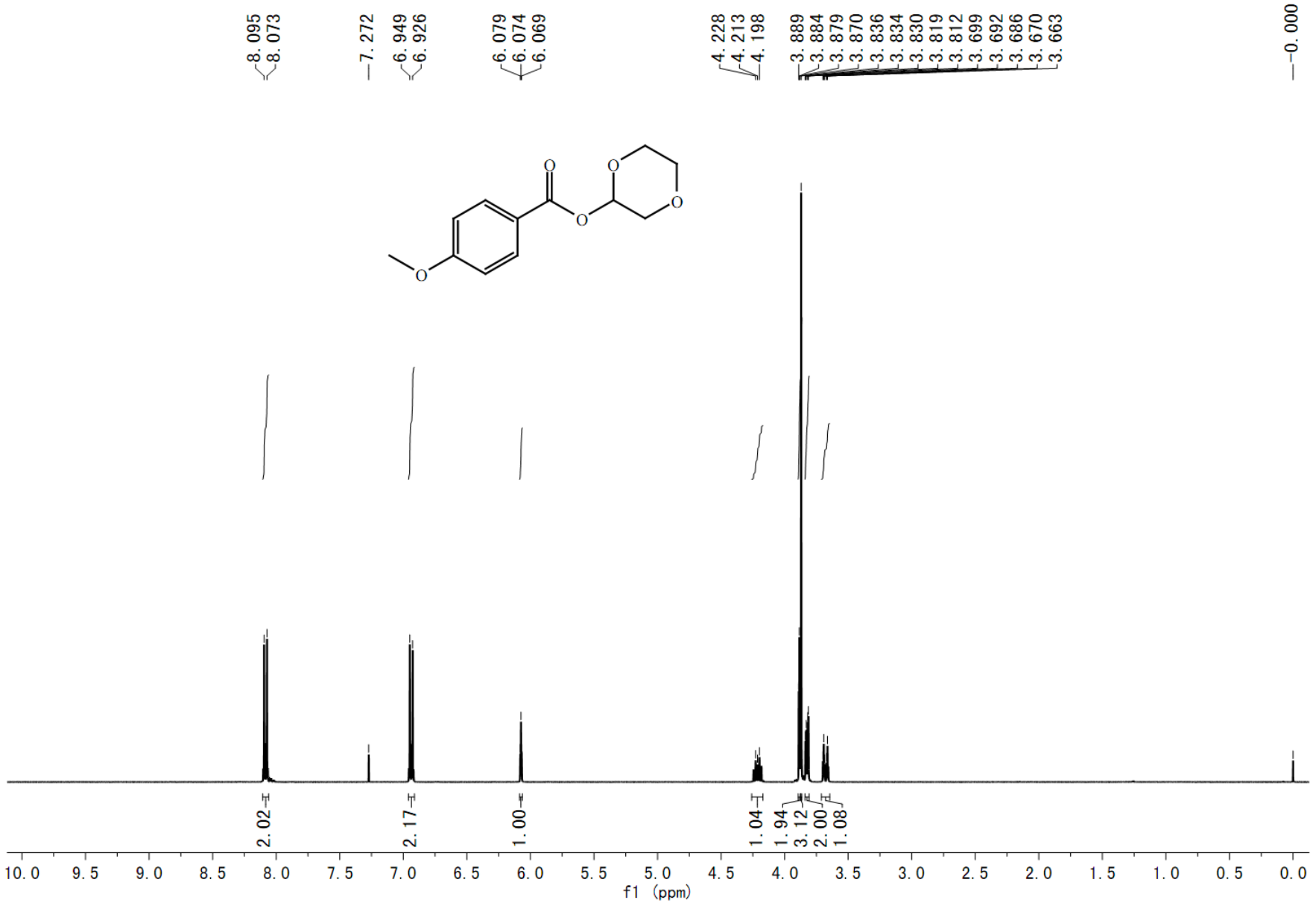
80
70
60
50
40
30
20
10
f1 (ppm)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7.272
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6.069
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4.213
4.198
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3.884
3.879
3.870
3.836
3.834
3.830
3.819
3.812
3.699
3.692
3.686
3.670
3.663
0.000
O
O
O
O
O
2.02
2.17
1.00
1.04
1.94
3.12
2.00
1.08
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
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1.5
1.0
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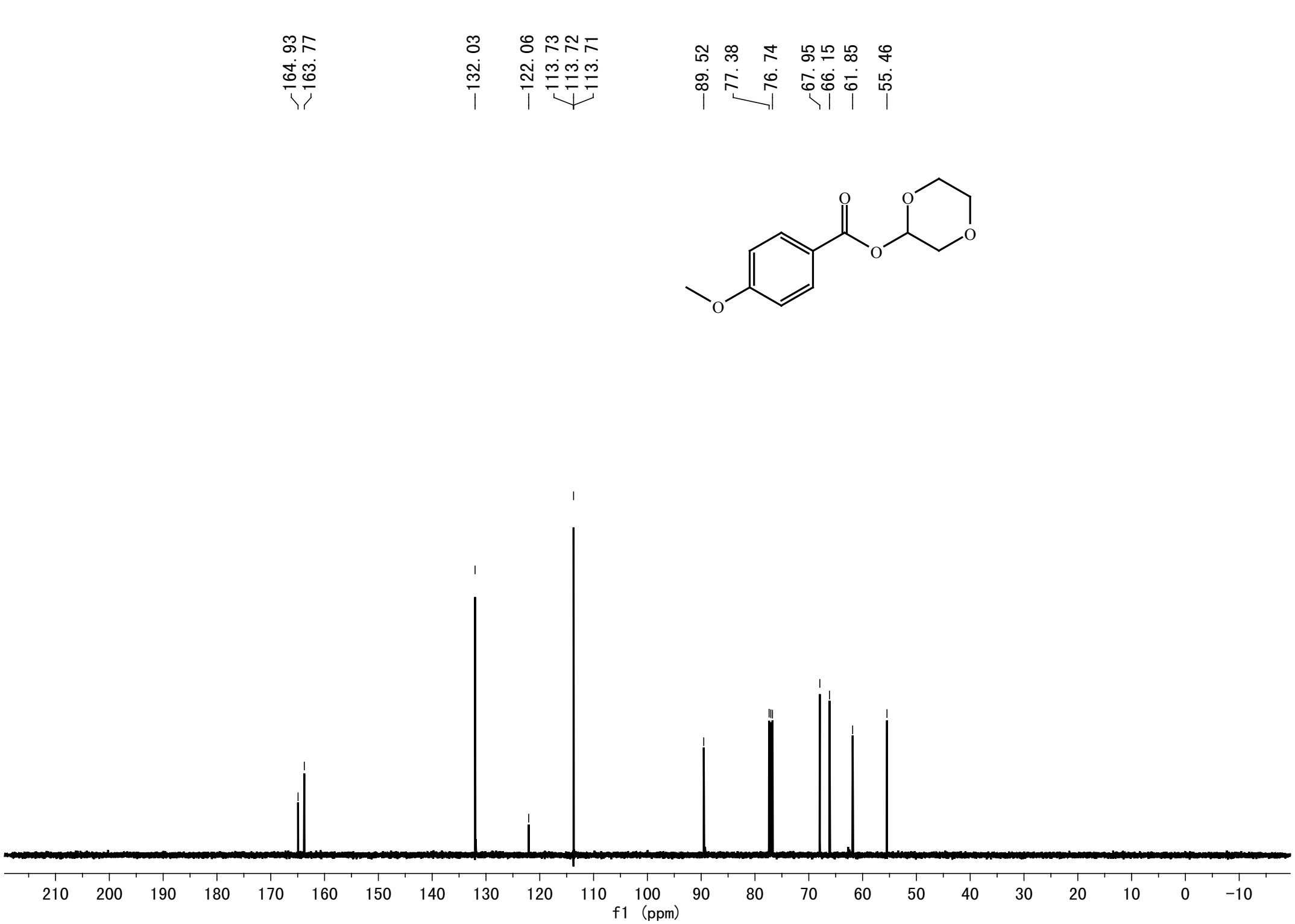
0.0
f1 (ppm)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122.06
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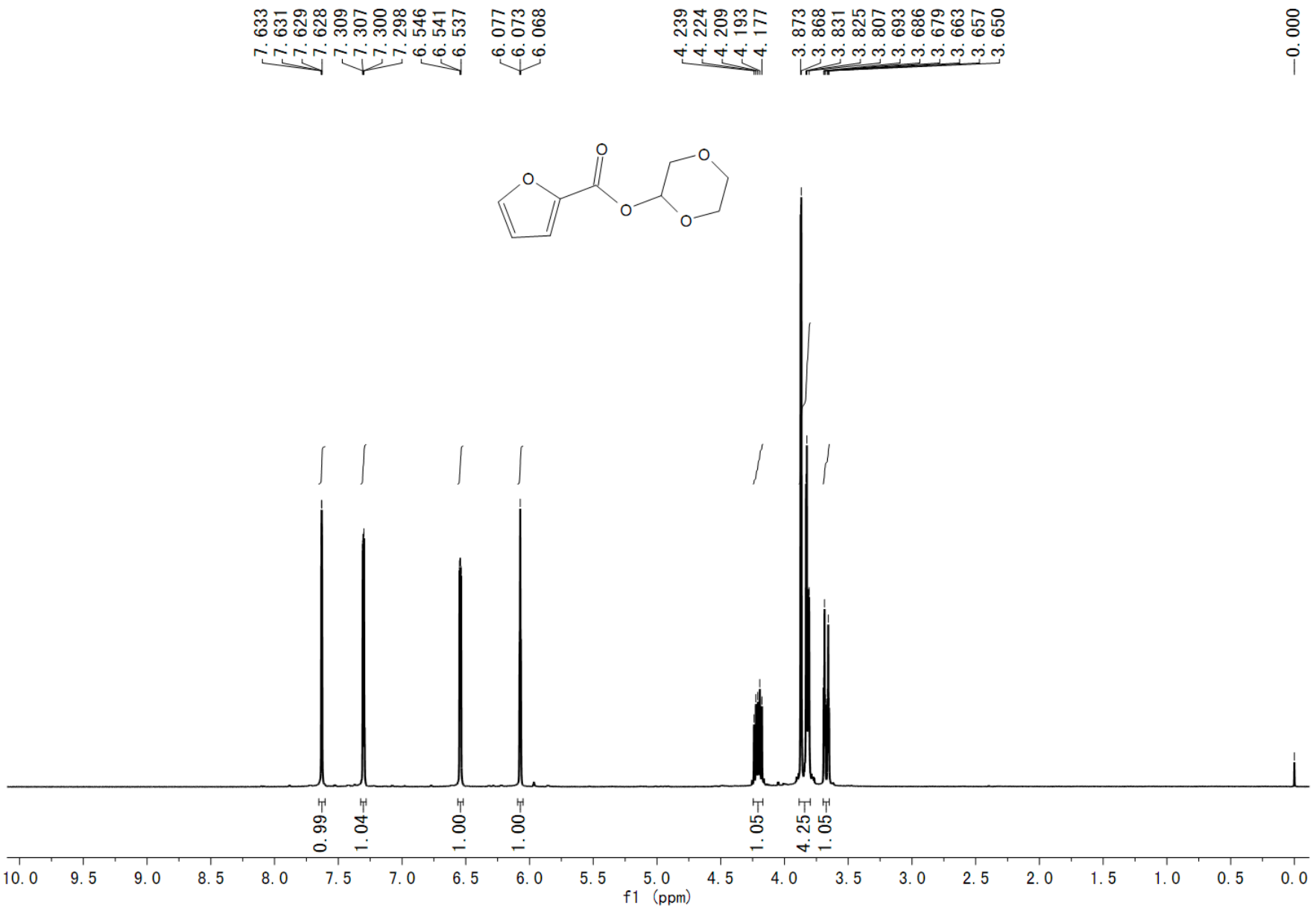
113.72
113.71
89.52
77.38
76.74
67.95
66.15
61.85
55.46
O
O
O
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)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6.537
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6.068
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4.224
4.209
4.193
4.177
3.873
3.868
3.831
3.825
3.807
3.693
3.686
3.679
3.663
3.657
3.650
0.000
0.99
1.04
1.00
1.00
1.05
4.25
1.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
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6.0
5.5
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3.0
2.5
2.0
1.5
1.0
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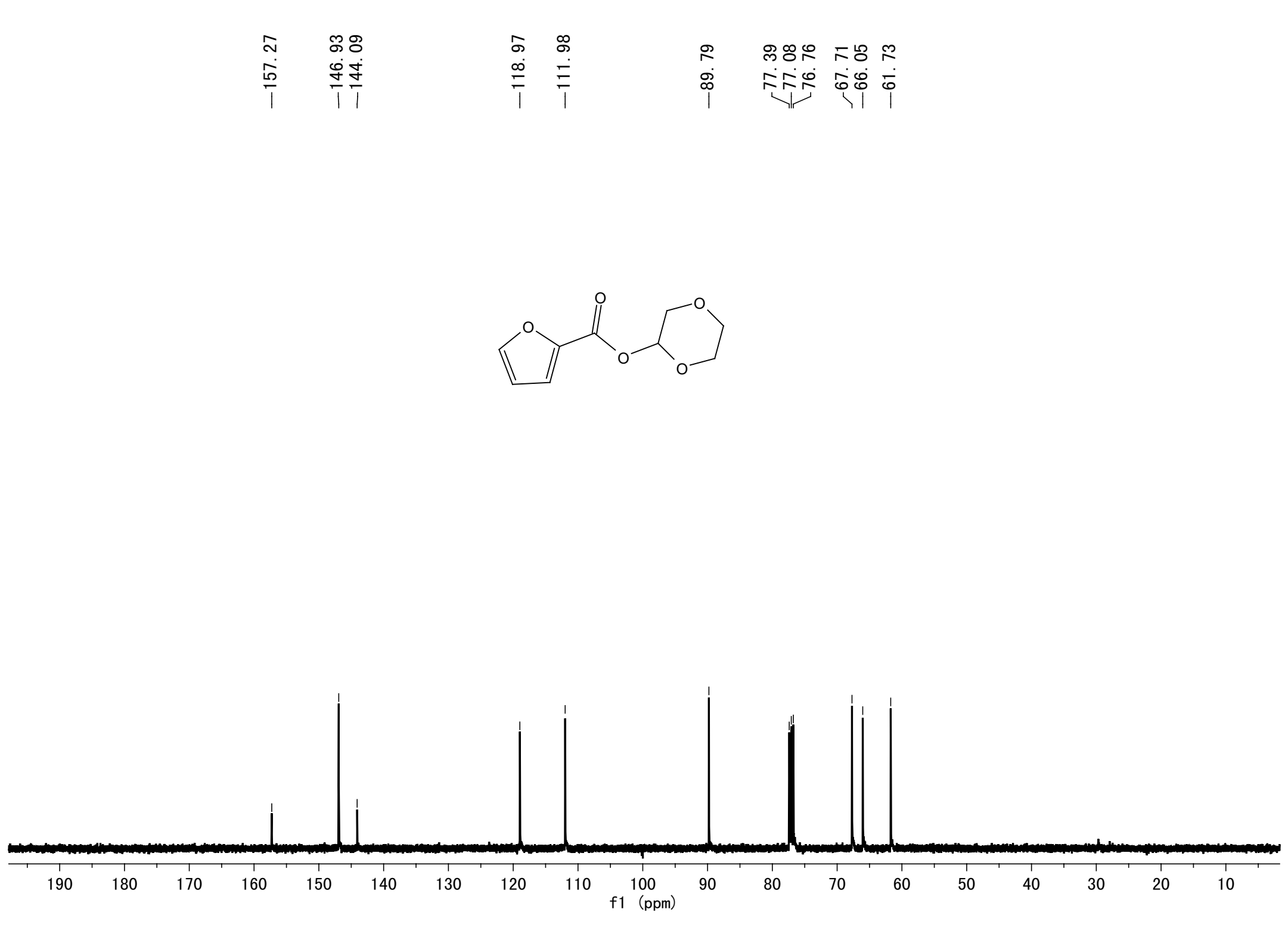
0.0
f1 (ppm)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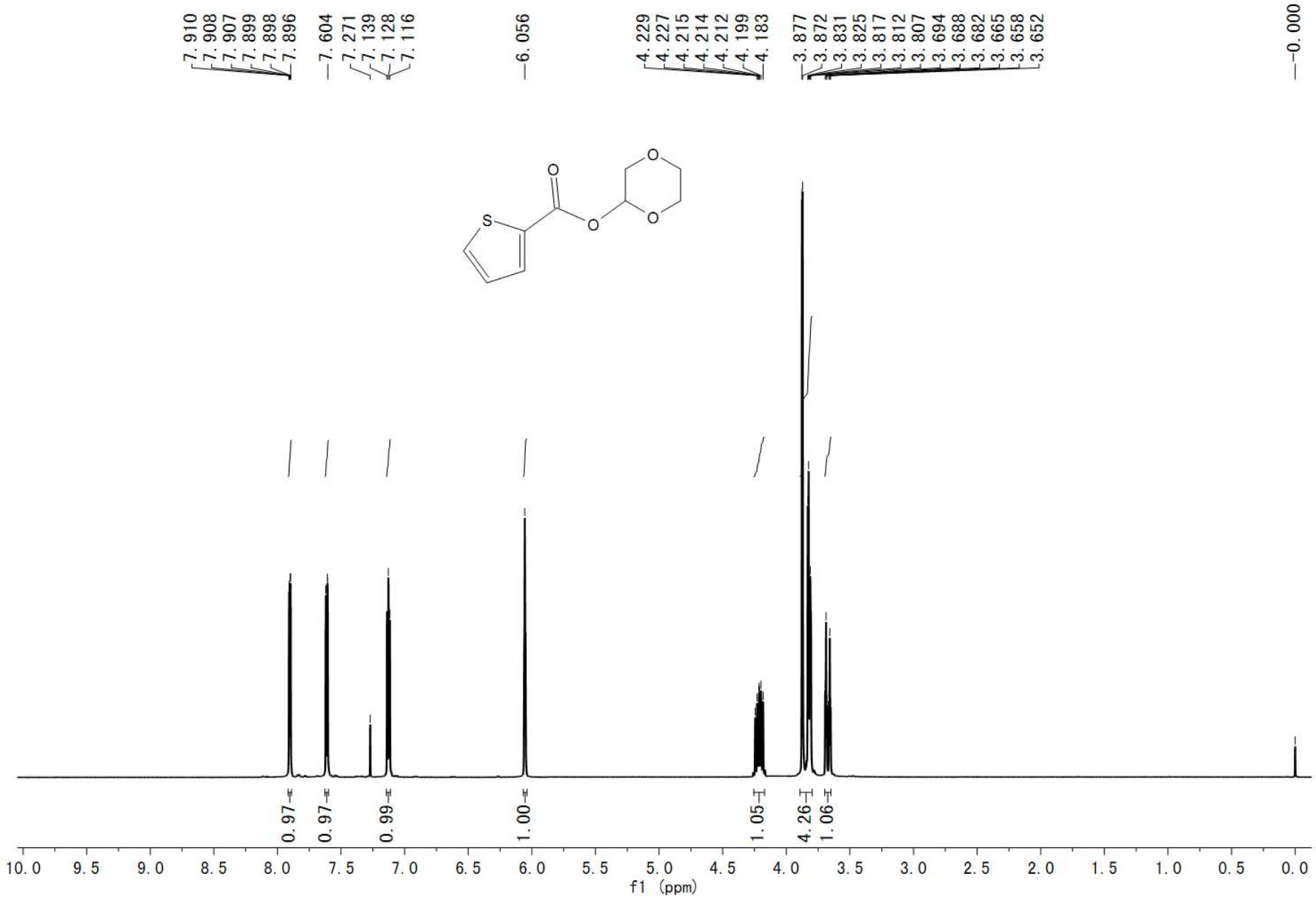
111.98
89.79
77.39
77.08
76.76
67.71
66.05
61.73
O
O
O
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.271
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4.227
4.215
4.214
4.212
4.199
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3.877
3.872
3.831
3.825
3.817
3.812
3.807
3.694
3.688
3.682
3.665
3.658
3.652
0.000
S
O
O
O
O
0.97
0.97
0.99
1.00
1.05
4.26
1.06
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9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.0
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1.0
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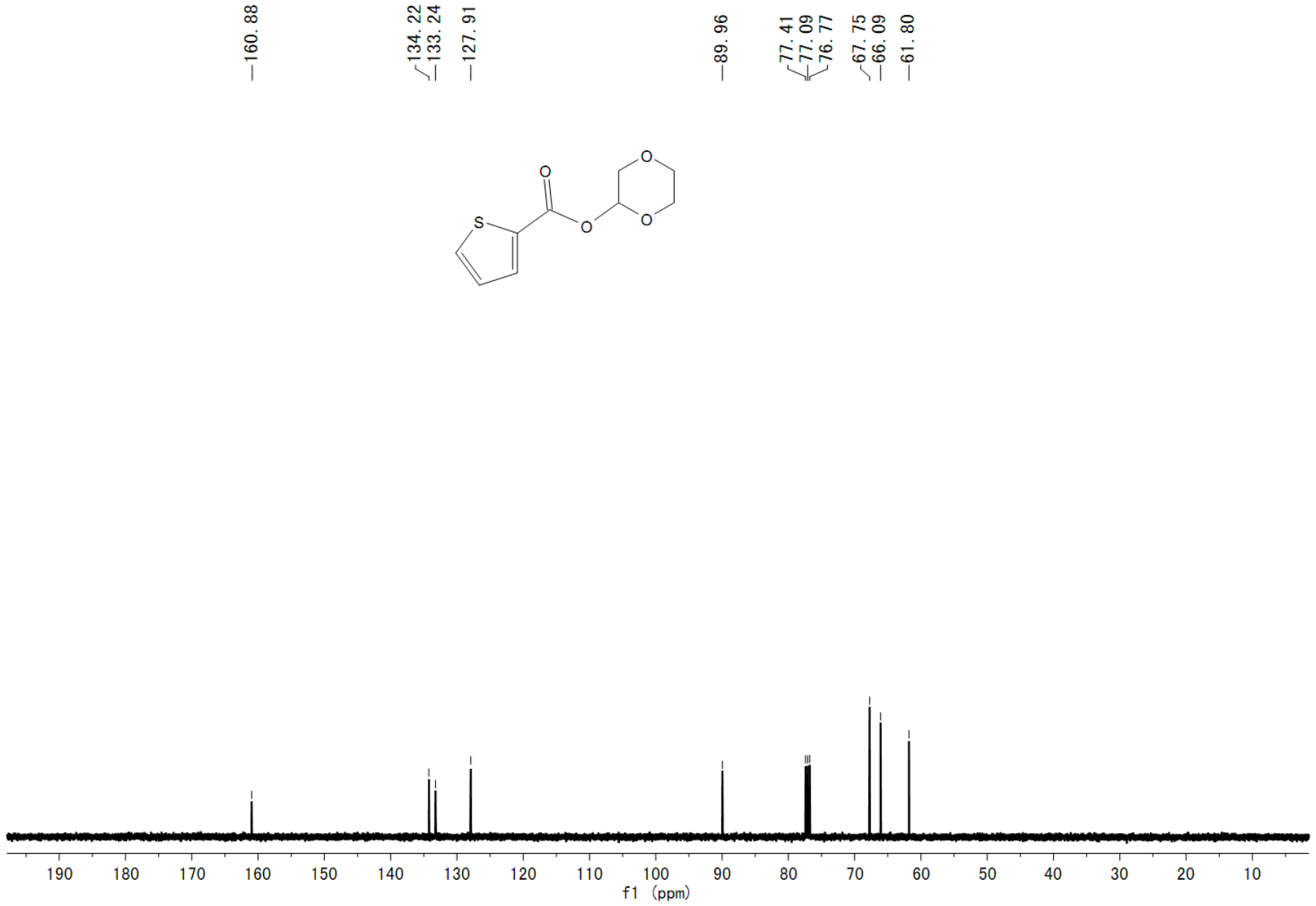
0.0
f1 (ppm)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76.77
67.75
66.09
61.80
O
S
O
O
O
190
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140
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120
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100
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80
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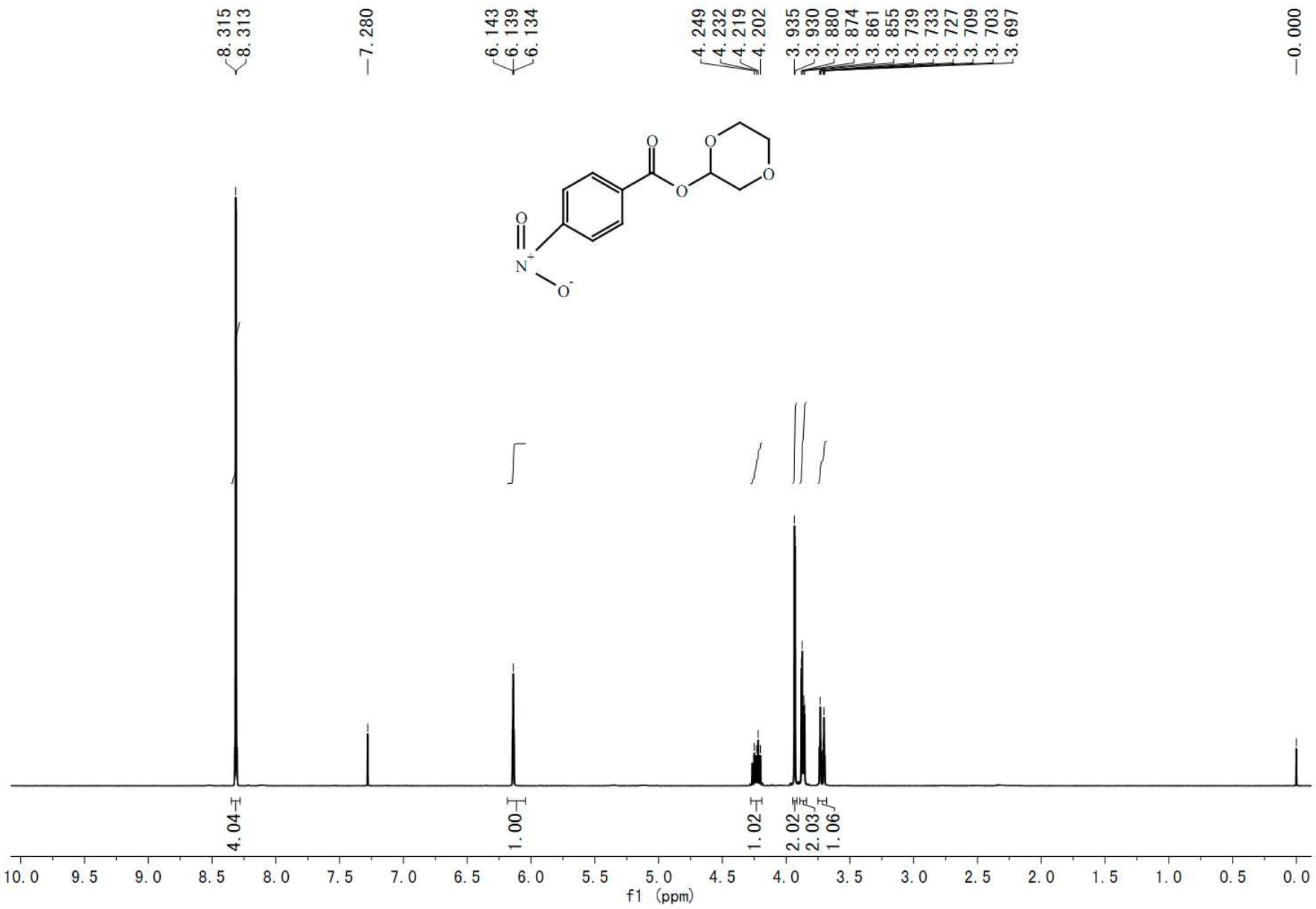
60
50
40
30
20
10
f1 (ppm)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3.930
3.880
3.874
3.861
3.855
3.739
3.733
3.727
3.709
3.703
3.697
0.000
4.04
1.00
1.02
2.02
2.03
1.06
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.5
2.0
1.5
1.0
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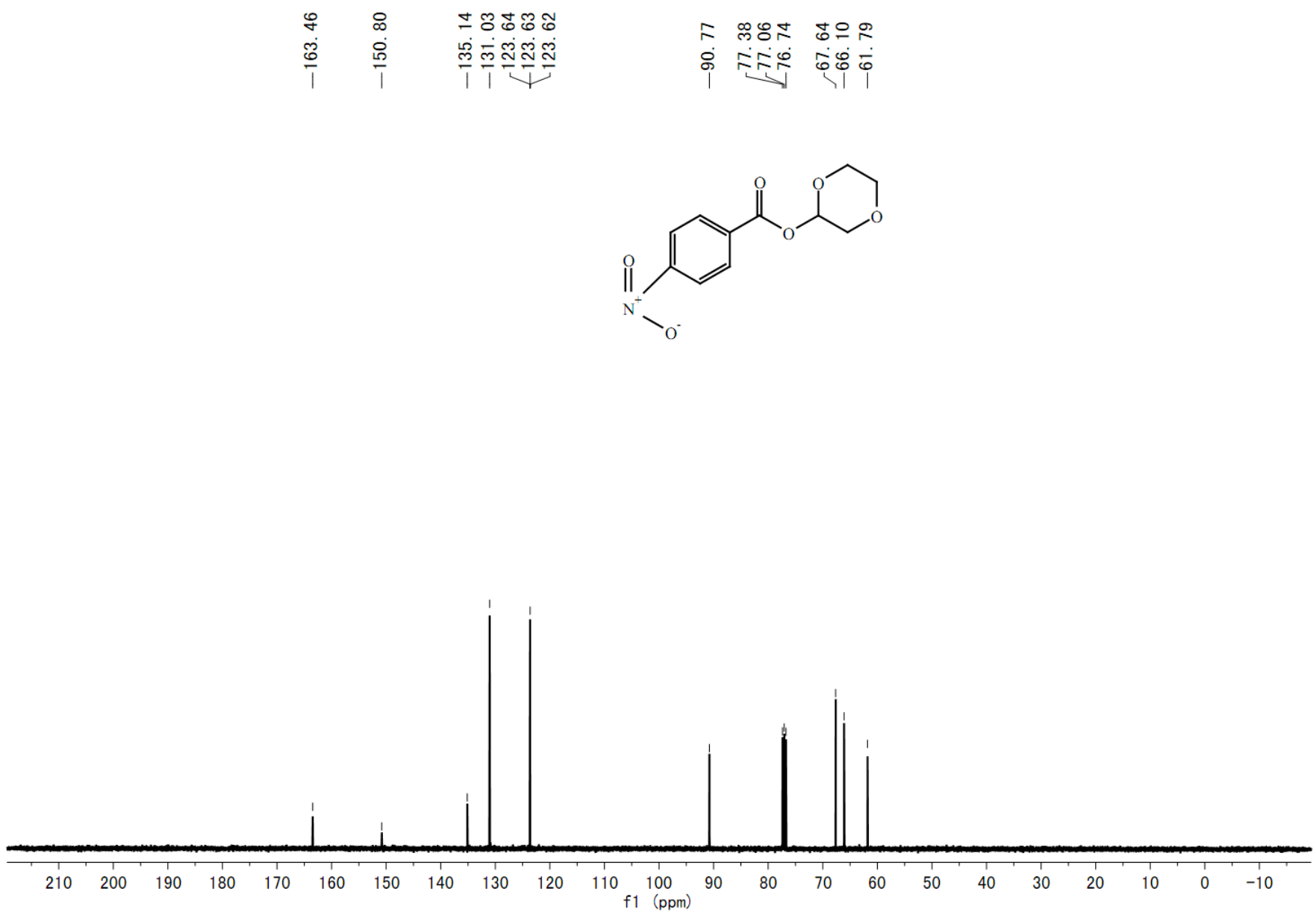
0.0
f1 (ppm)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77.06
76.74
67.64
66.10
61.79
O
O
O
O
O
N+
O-
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200
190
180
170
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120
110
100
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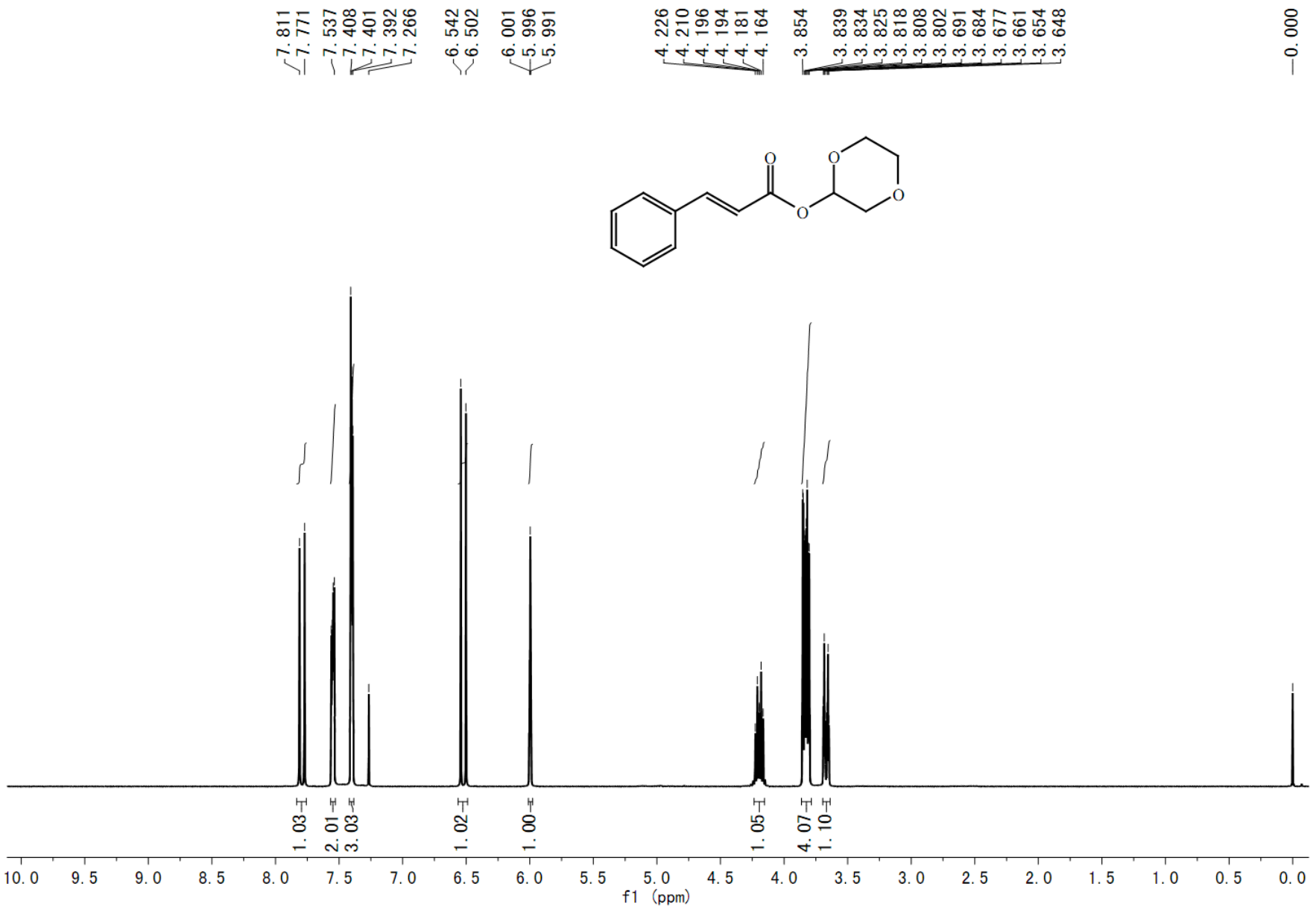
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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5.991
4.226
4.210
4.196
4.194
4.181
4.164
3.854
3.839
3.834
3.825
3.818
3.808
3.802
3.691
3.684
3.677
3.661
3.654
3.648
0.000
O
O
O
O
1.03
2.01
3.03
1.02
1.00
1.05
4.07
1.10
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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2.0
1.5
1.0
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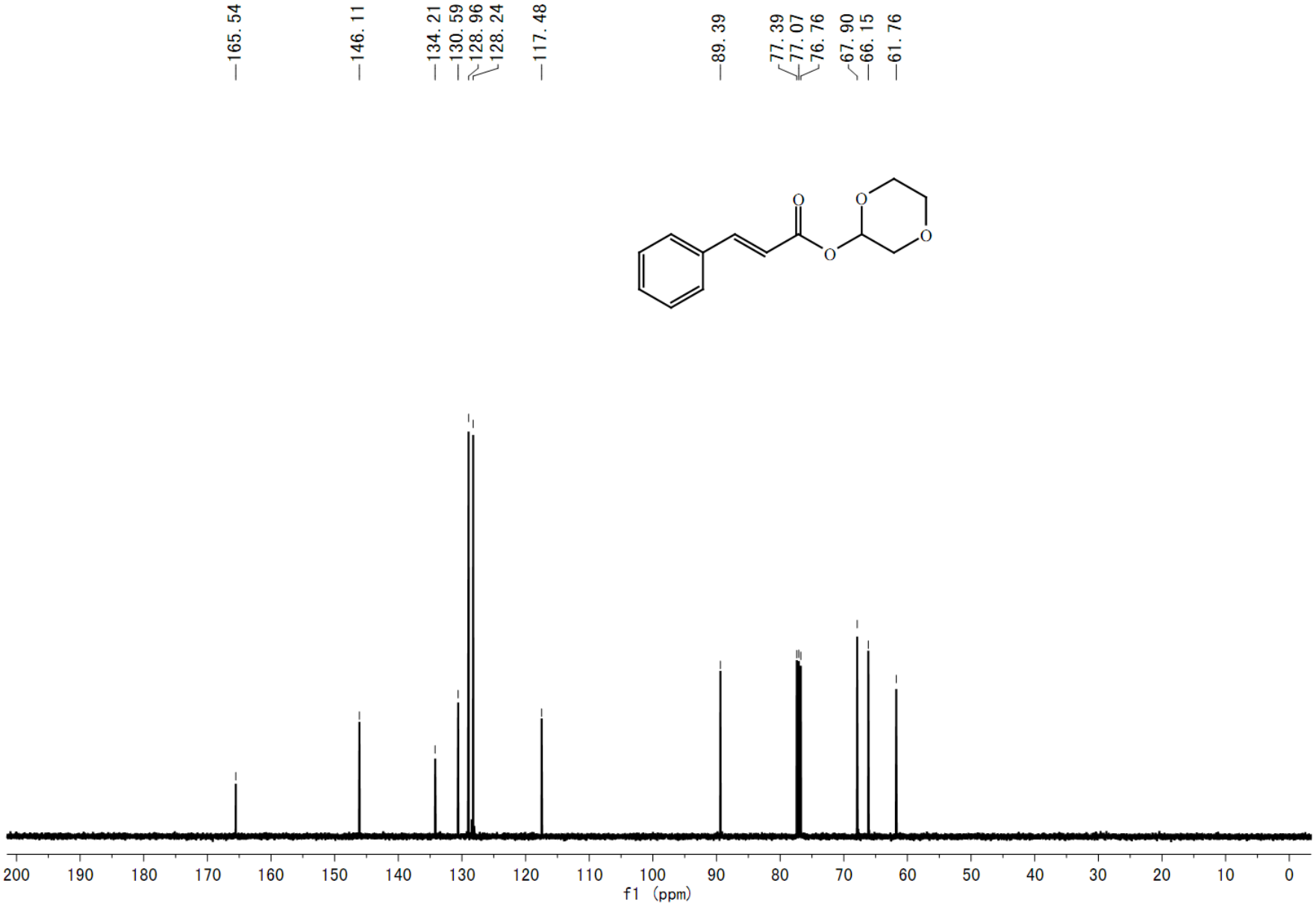
f1 (ppm)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128.24
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77.39
77.07
76.76
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66.15
61.76
O
O
O
O
200
190
180
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120
110
100
90
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70
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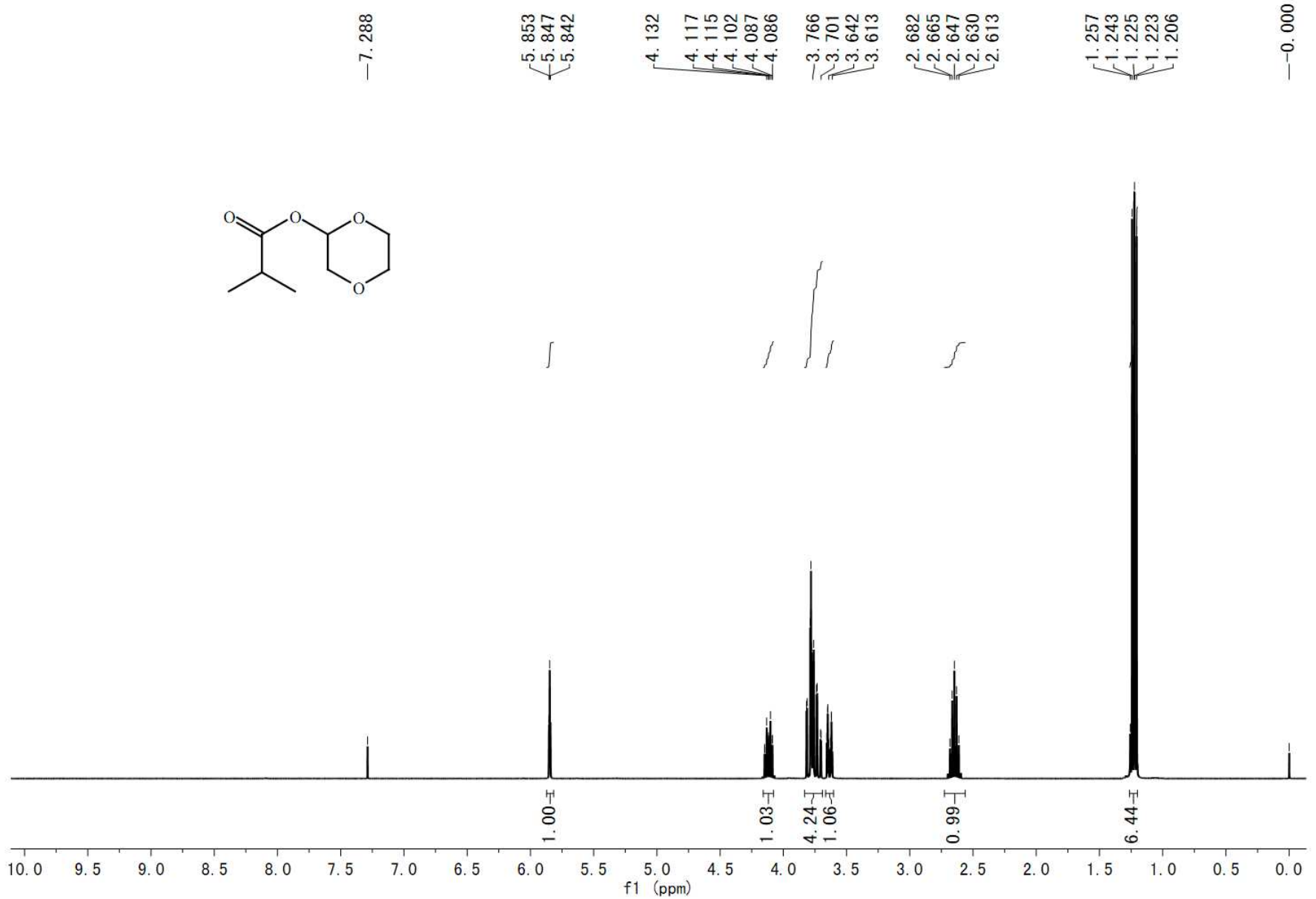
50
40
30
20
10
0
f1 (ppm)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4.086
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2.630
2.613
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1.243
1.225
1.223
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0.000
1.00
1.03
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1.06
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4.5
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3.0
2.5
2.0
1.5
1.0
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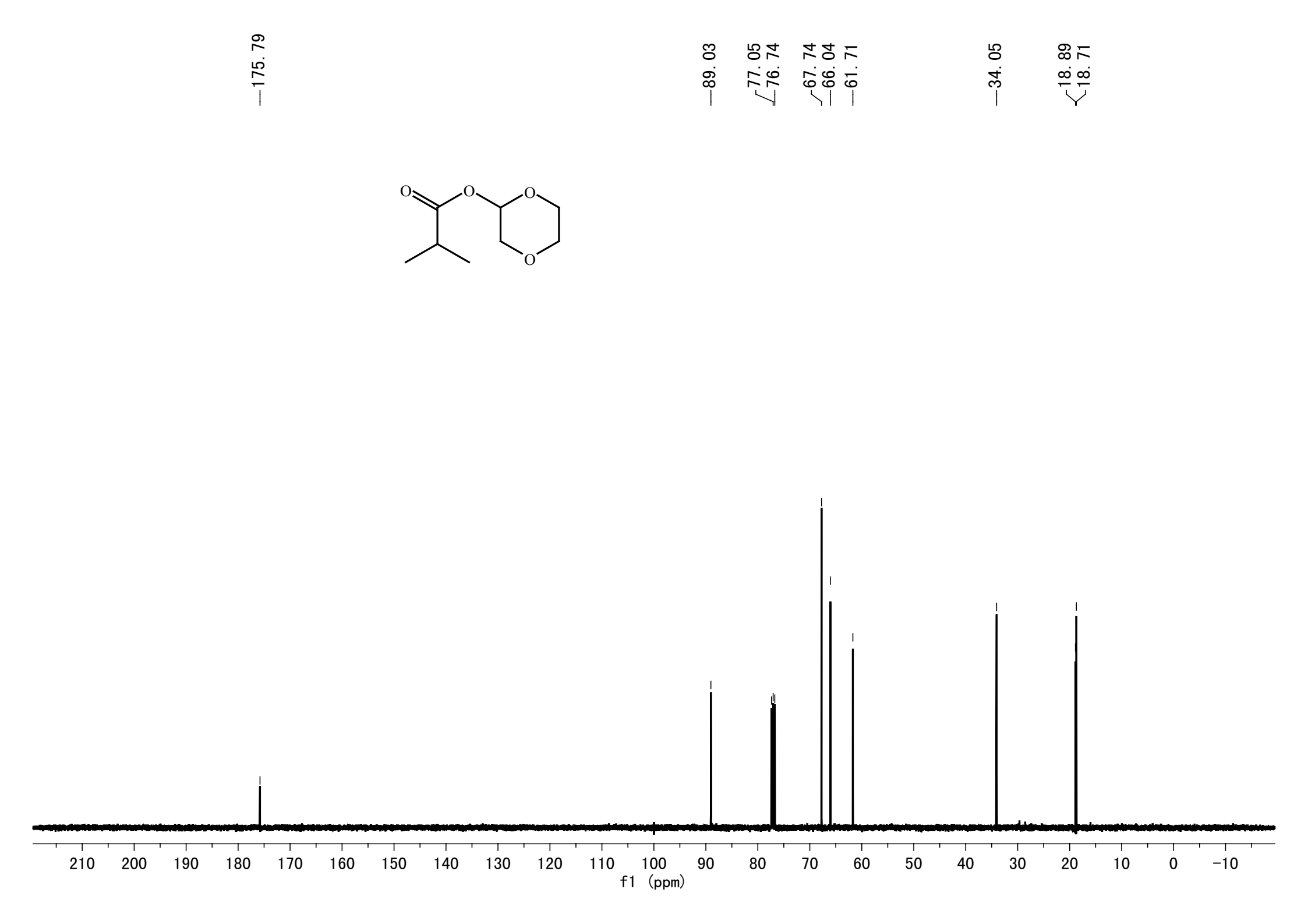
0.0
f1 (ppm)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34.05
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18.71
O
O
O
O
O
210
200
190
180
170
160
150
140
130
120
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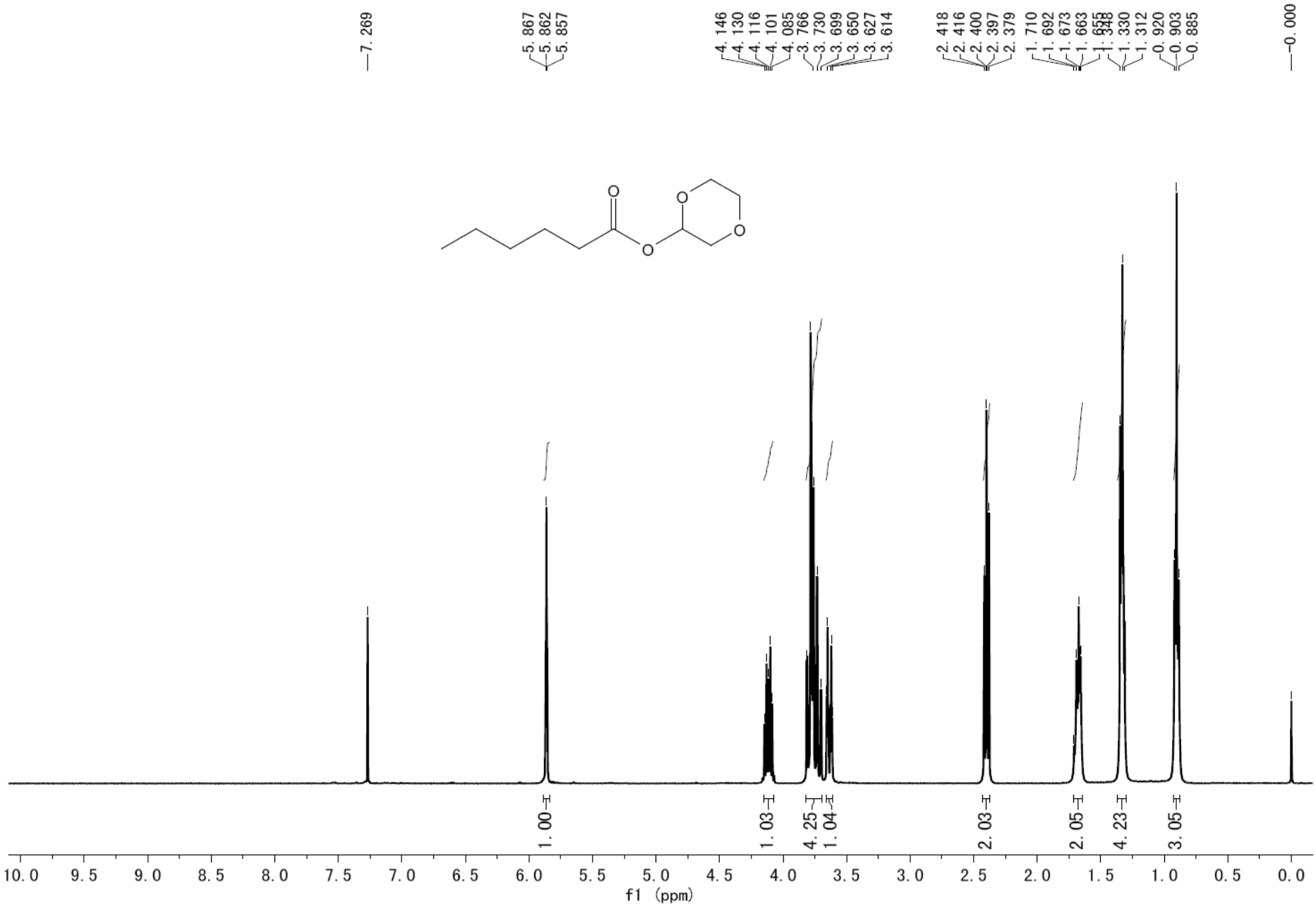
f1 (ppm)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3.614
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2.416
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2.379
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1.655
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0.000
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O
O
O
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1.03
4.25
1.04
2.03
2.05
4.23
3.05
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7.0
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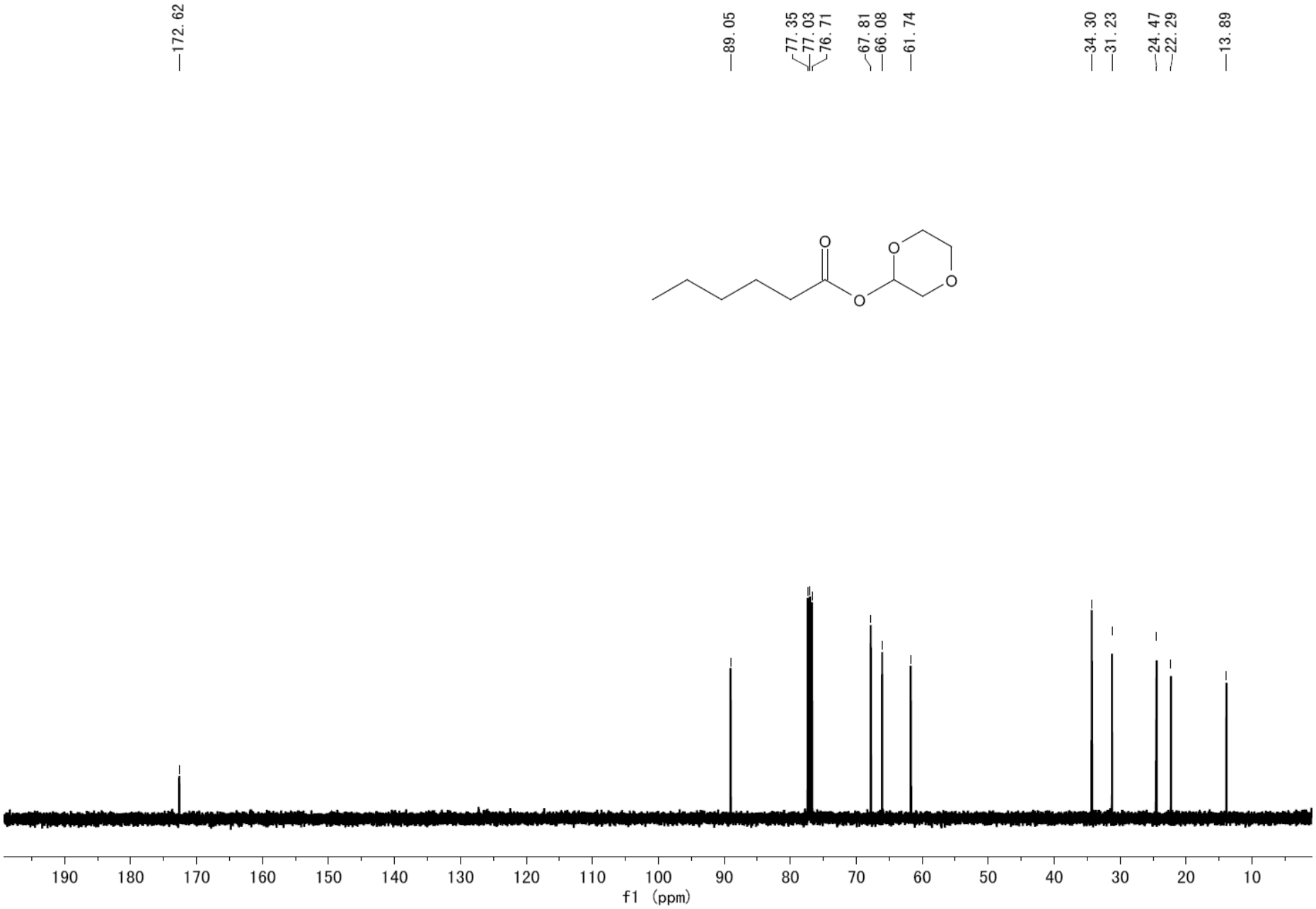
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61.74
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31.23
24.47
22.29
13.89
O
O
O
O
190
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100
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60
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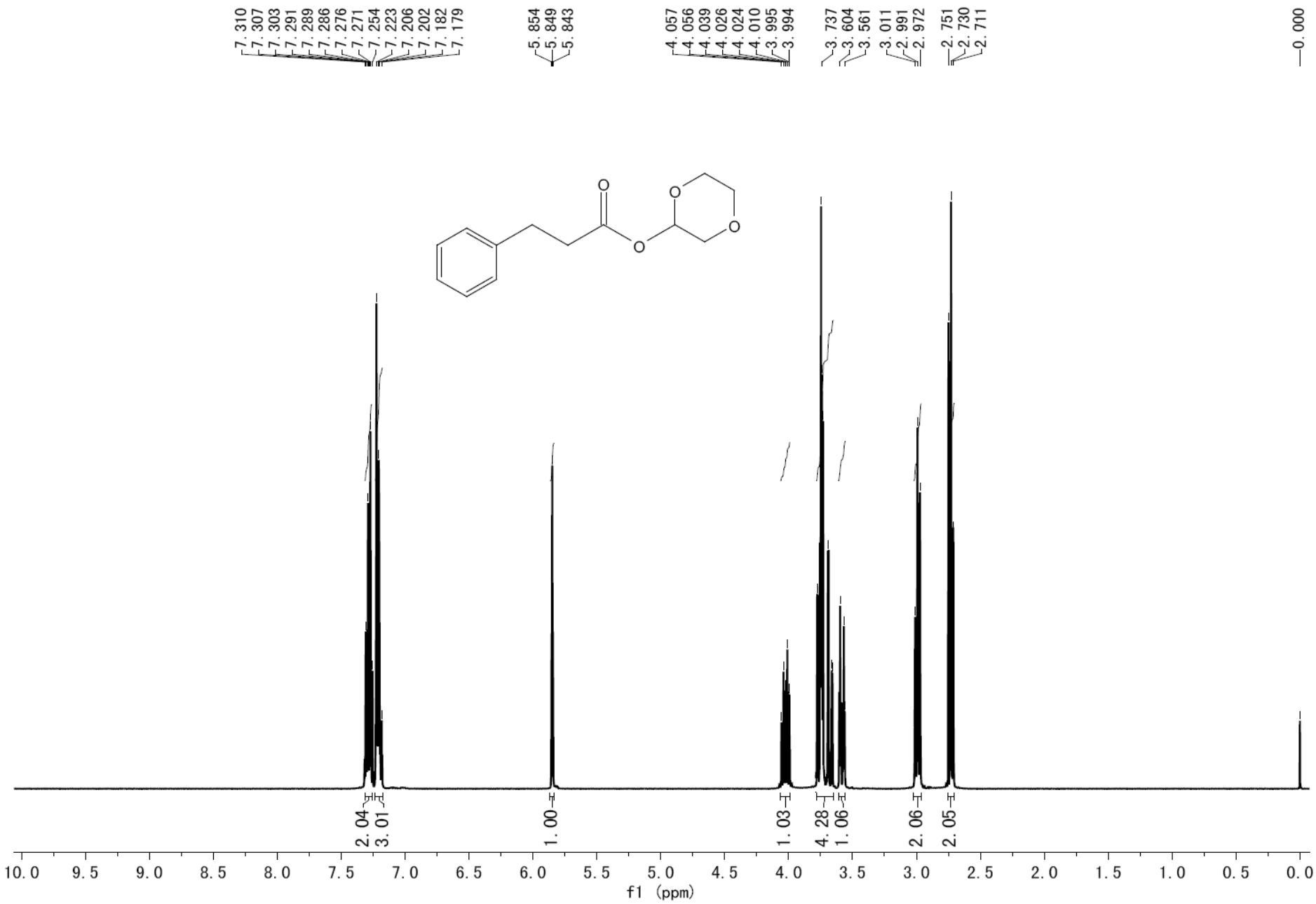
20
10
f1 (ppm)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7.179
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5.849
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4.056
4.039
4.026
4.024
4.010
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3.994
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3.604
3.561
3.011
2.991
2.972
2.751
2.730
2.711
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O
O
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3.01
1.00
1.03
4.28
1.06
2.06
2.05
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9.0
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8.0
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6.5
6.0
5.5
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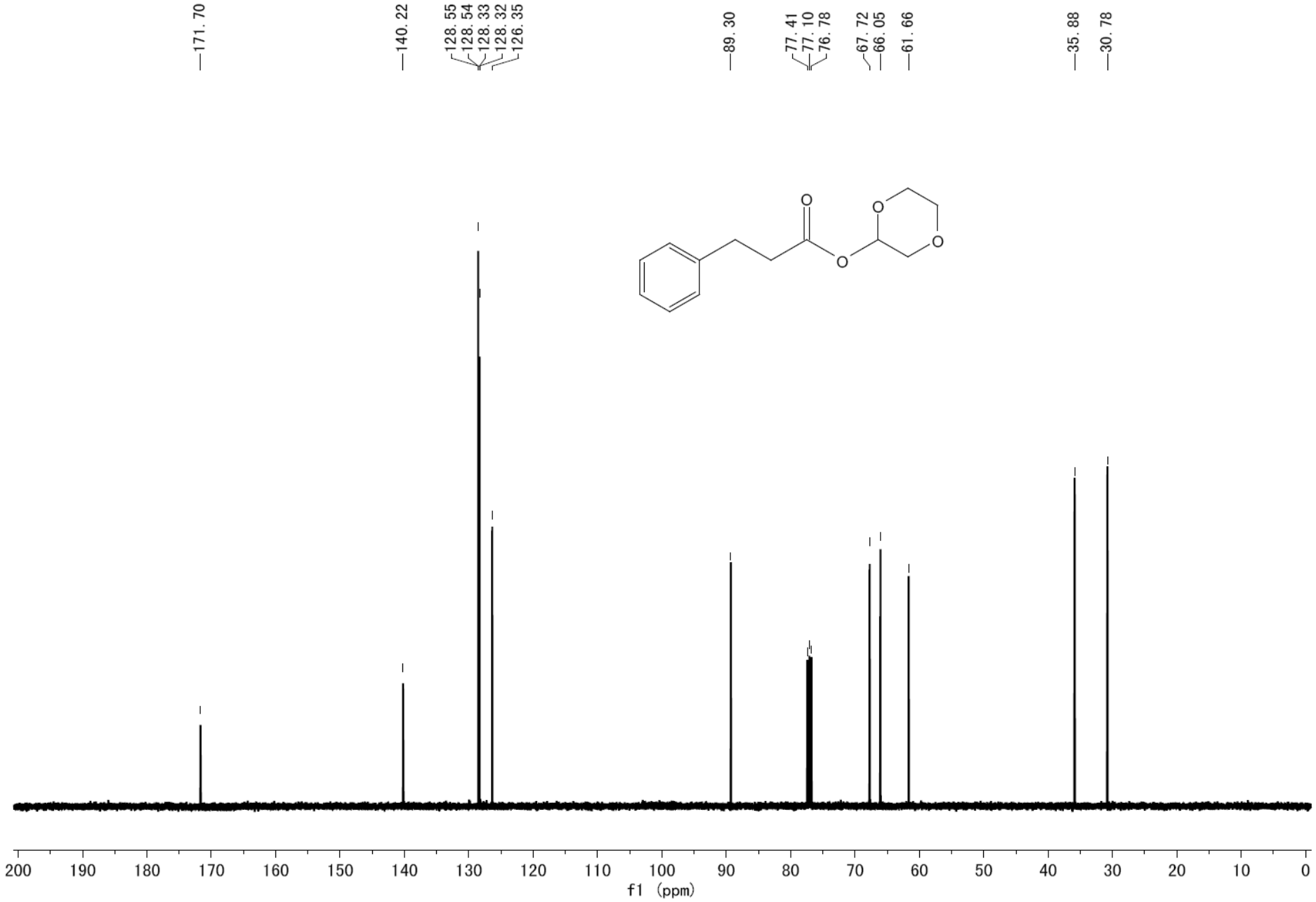
f1 (ppm)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89.30
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77.10
76.78
67.72
66.05
61.66
35.88
30.78
O
O
O
O
200
190
180
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160
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130
120
110
100
90
80
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50
40
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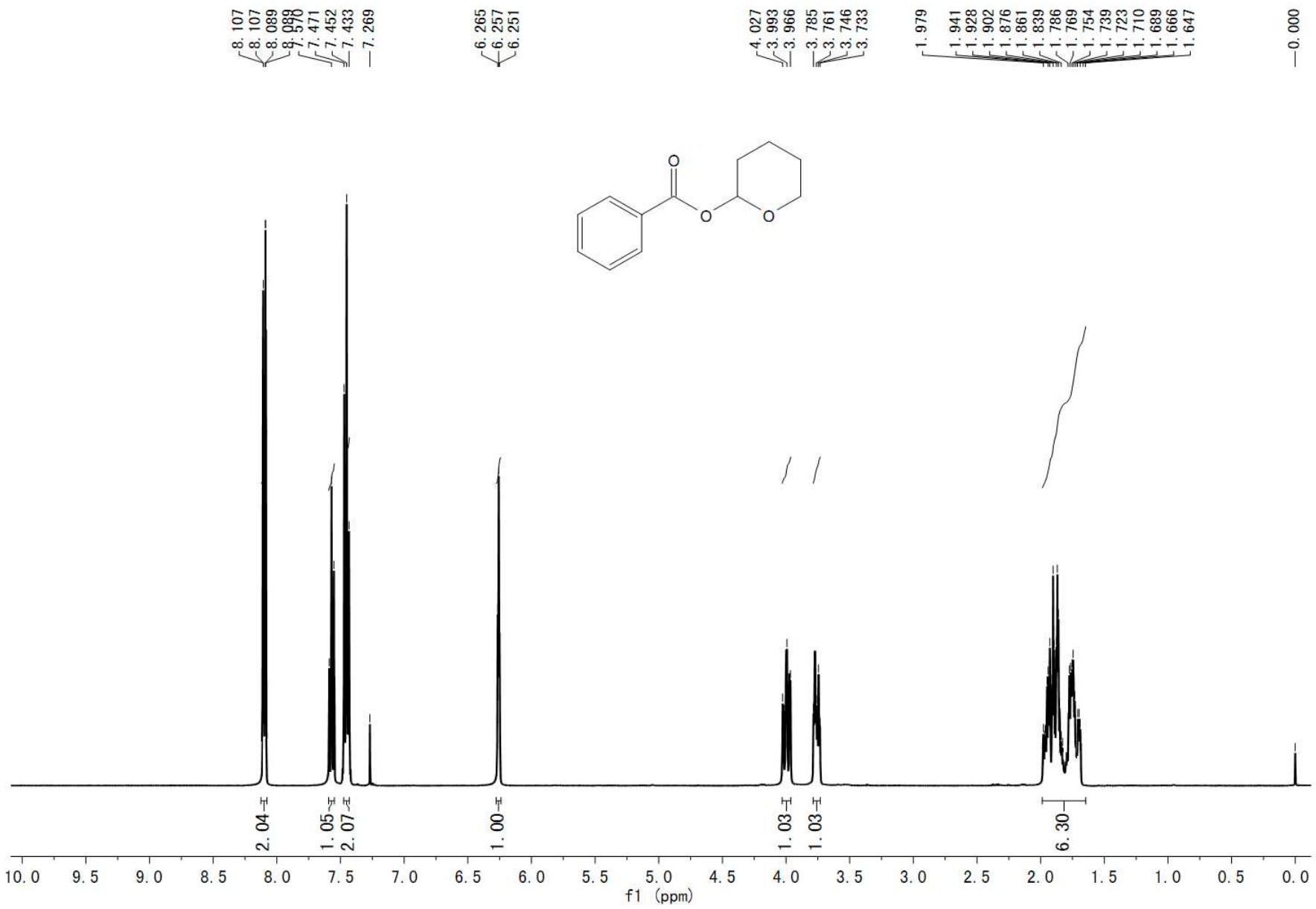
f1 (ppm)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3.761
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3.733
1.979
1.941
1.928
1.902
1.876
1.861
1.839
1.786
1.769
1.754
1.739
1.723
1.710
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1.666
1.647
0.000
O
O
O
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1.05
2.07
1.00
1.03
1.03
6.30
10.0
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9.0
8.5
8.0
7.5
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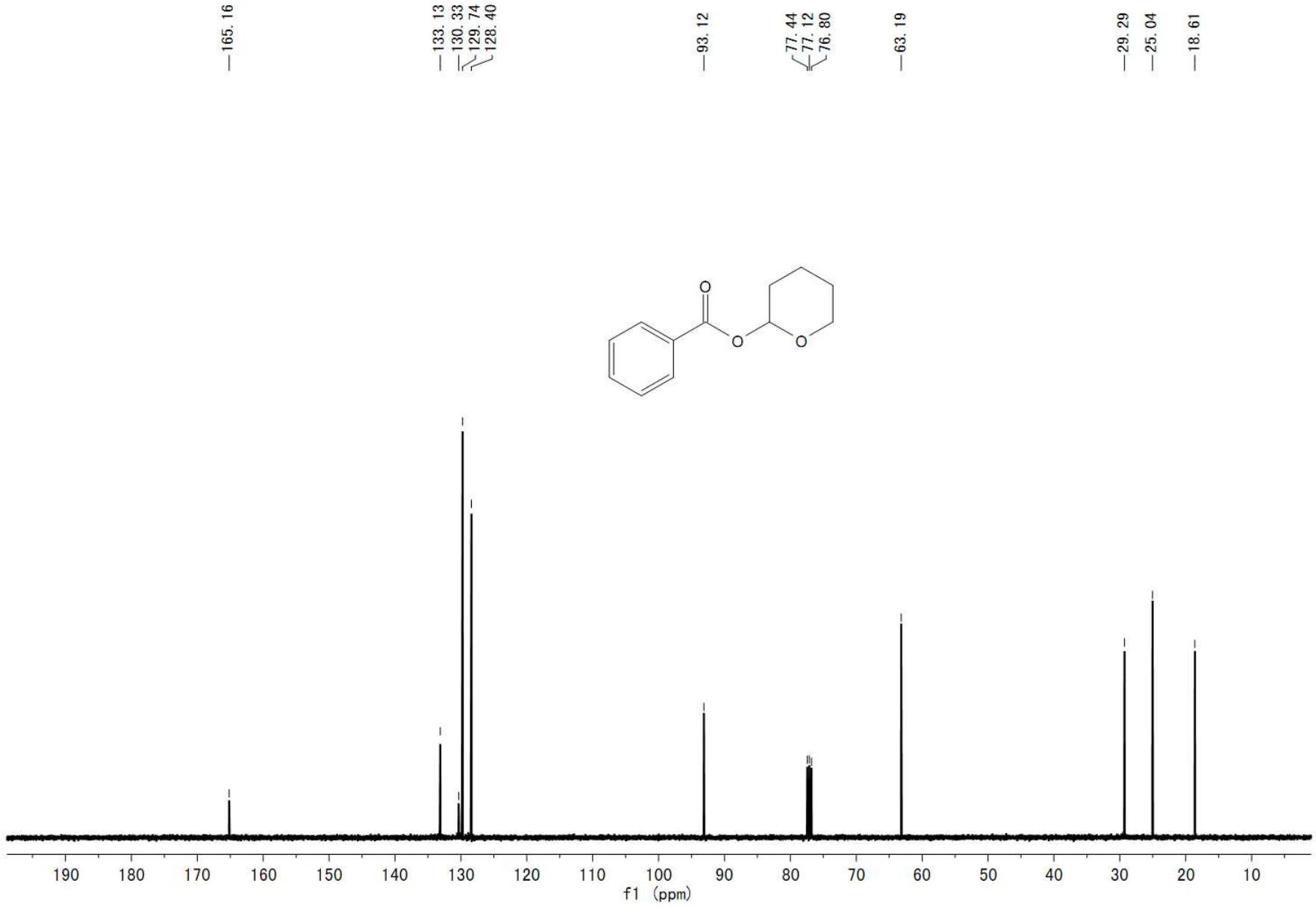
f1 (ppm)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77.44
77.12
76.80
63.19
29.29
25.04
18.61
O
O
O
190
180
170
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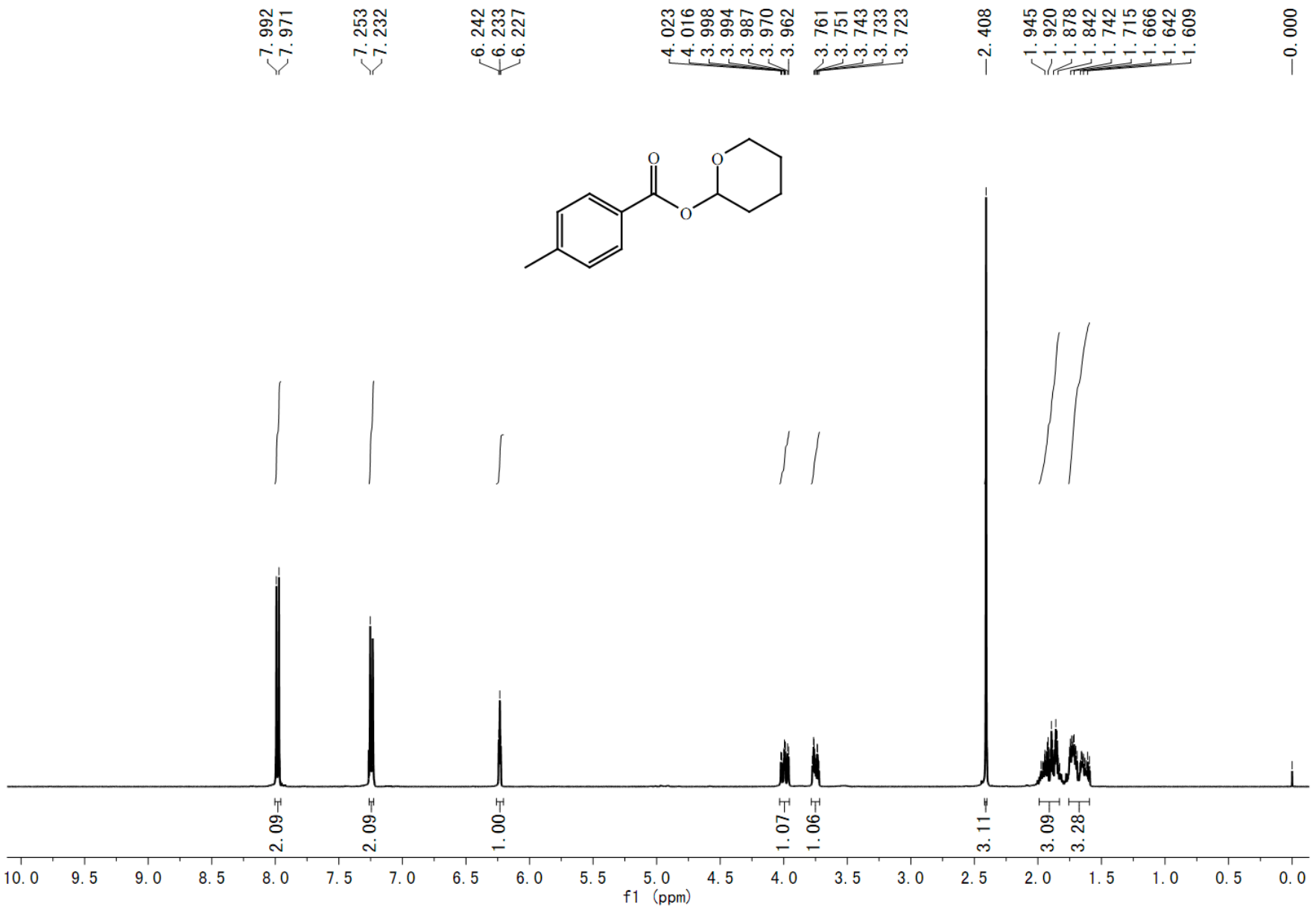
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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4.023
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3.994
3.987
3.970
3.962
3.761
3.751
3.743
3.733
3.723
2.408
1.945
1.920
1.878
1.842
1.742
1.715
1.666
1.642
1.609
0.000
O
O
O
2.09
2.09
1.00
1.07
1.06
3.11
3.09
3.28
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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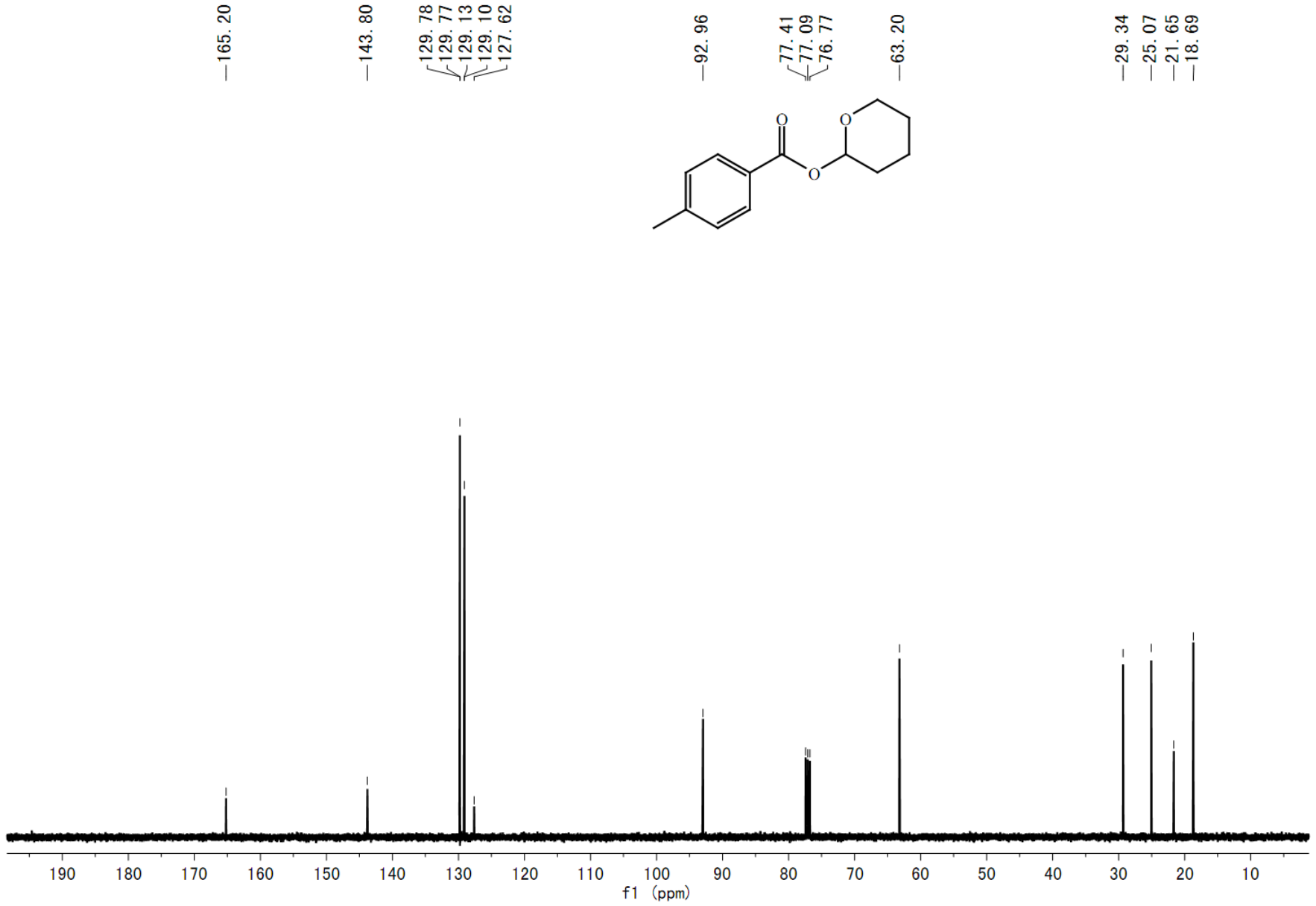
0.0
f1 (ppm)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77.09
76.77
63.20
29.34
25.07
21.65
18.69
O
O
O
190
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160
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140
130
120
110
100
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80
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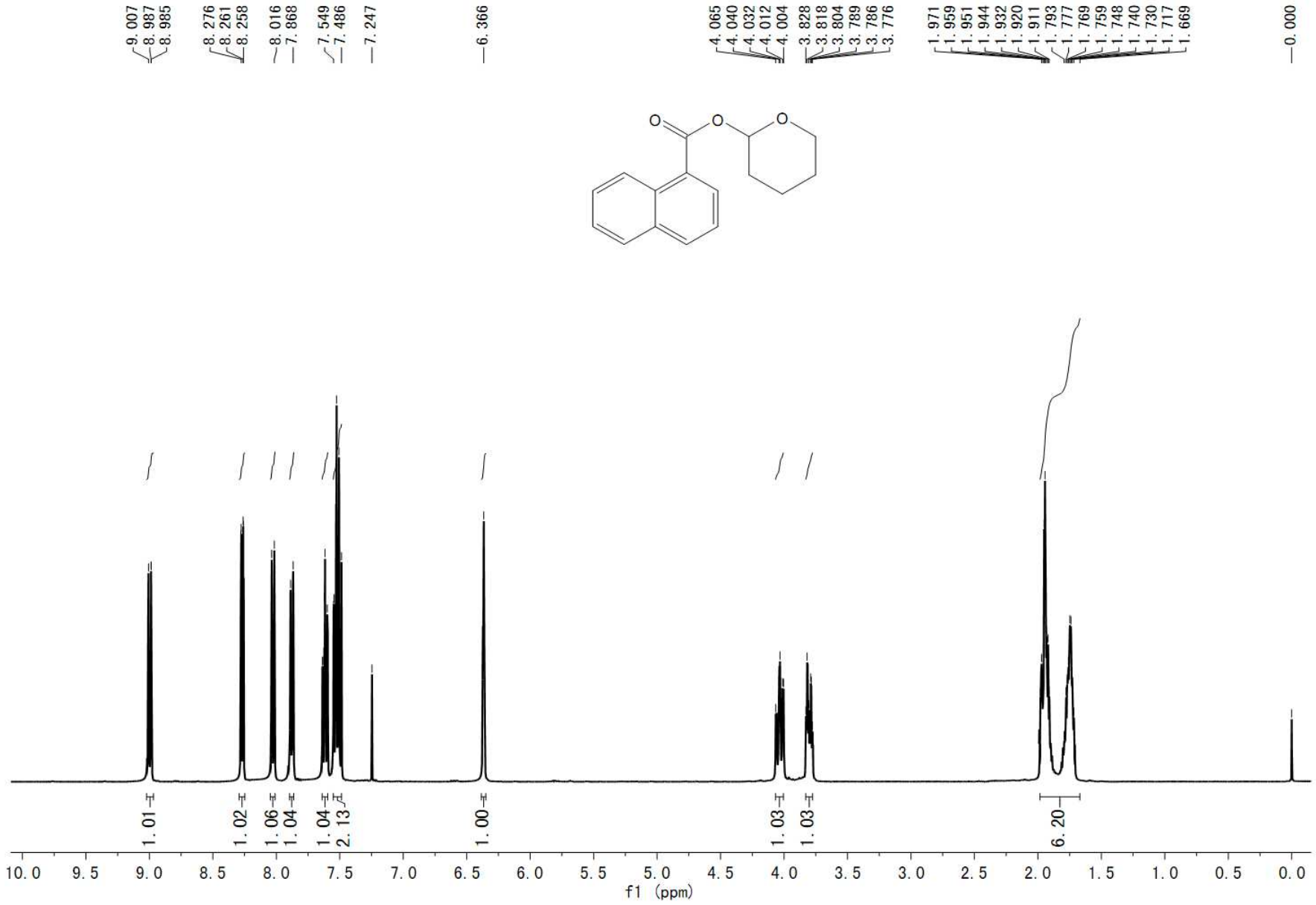
f1 (ppm)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8.016
7.868
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7.486
7.247
6.366
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4.040
4.032
4.012
4.004
3.828
3.818
3.804
3.789
3.786
3.776
1.971
1.959
1.951
1.944
1.932
1.920
1.911
1.793
1.777
1.769
1.759
1.748
1.740
1.730
1.717
1.669
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O
O
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1.02
1.06
1.04
1.04
2.13
1.00
1.03
1.03
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8.0
7.5
7.0
6.5
6.0
5.5
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2.0
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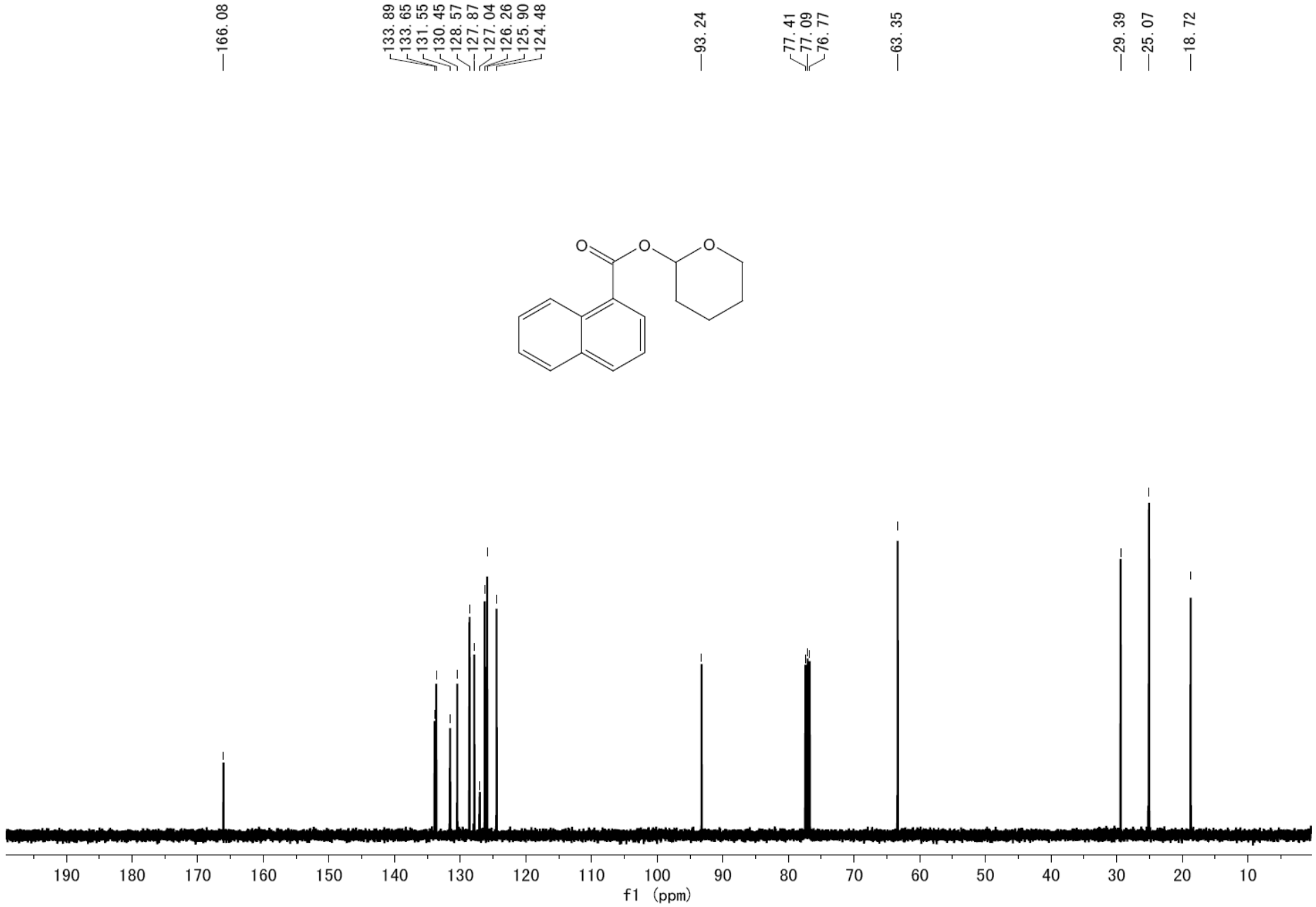
f1 (ppm)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77.41
77.09
76.77
63.35
29.39
25.07
18.72
O
O
O
190
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140
130
120
110
100
90
80
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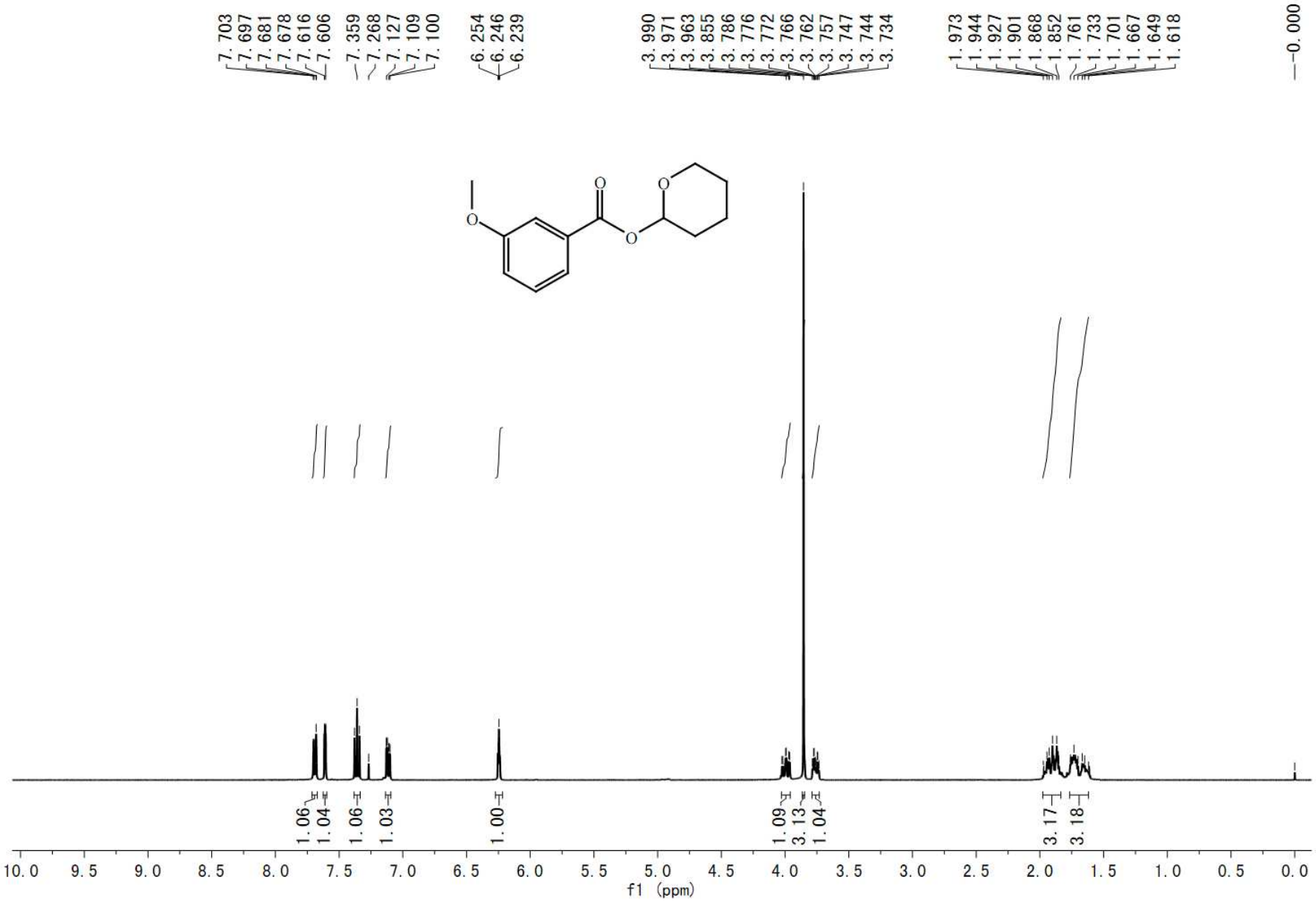
60
50
40
30
20
10
f1 (ppm)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7.606
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7.109
7.100
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6.246
6.239
3.990
3.971
3.963
3.855
3.786
3.776
3.772
3.766
3.762
3.757
3.747
3.744
3.734
1.973
1.944
1.927
1.901
1.868
1.852
1.761
1.733
1.701
1.667
1.649
1.618
0.000
O
O
O
O
1.06
1.04
1.06
1.03
1.00
1.09
3.13
1.04
3.17
3.18
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7.5
7.0
6.5
6.0
5.5
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2.0
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1.0
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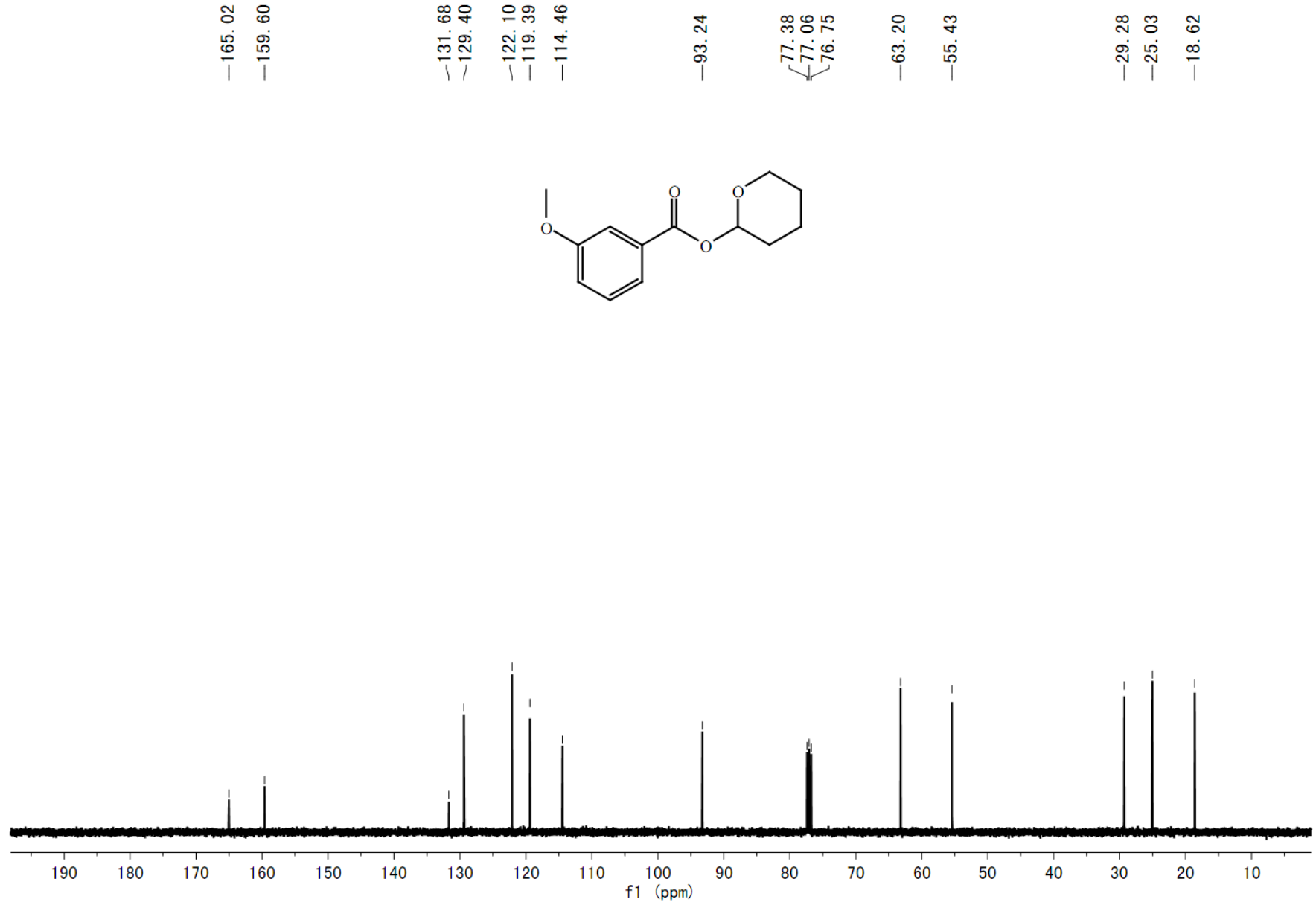
f1 (ppm)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93.24
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77.06
76.75
63.20
55.43
29.28
25.03
18.62
O
O
O
O
190
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140
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120
110
100
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80
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60
50
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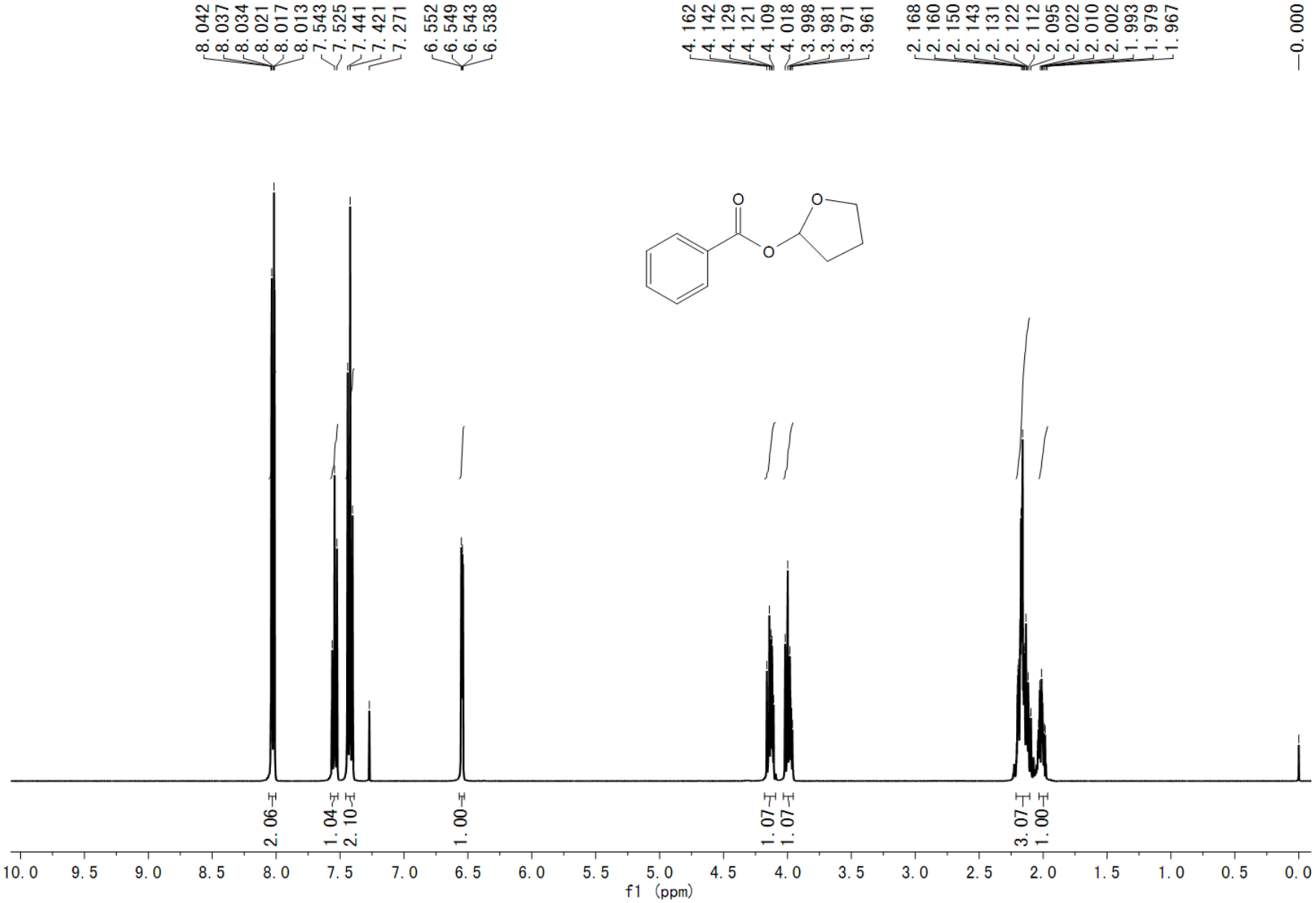
20
10
f1 (ppm)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6.549
6.543
6.538
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4.129
4.121
4.109
4.018
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3.971
3.961
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2.160
2.150
2.143
2.131
2.122
2.112
2.095
2.022
2.010
2.002
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0.000
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O
O
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2.10
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1.07
1.07
3.07
1.00
10.0
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9.0
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8.0
7.5
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6.0
5.5
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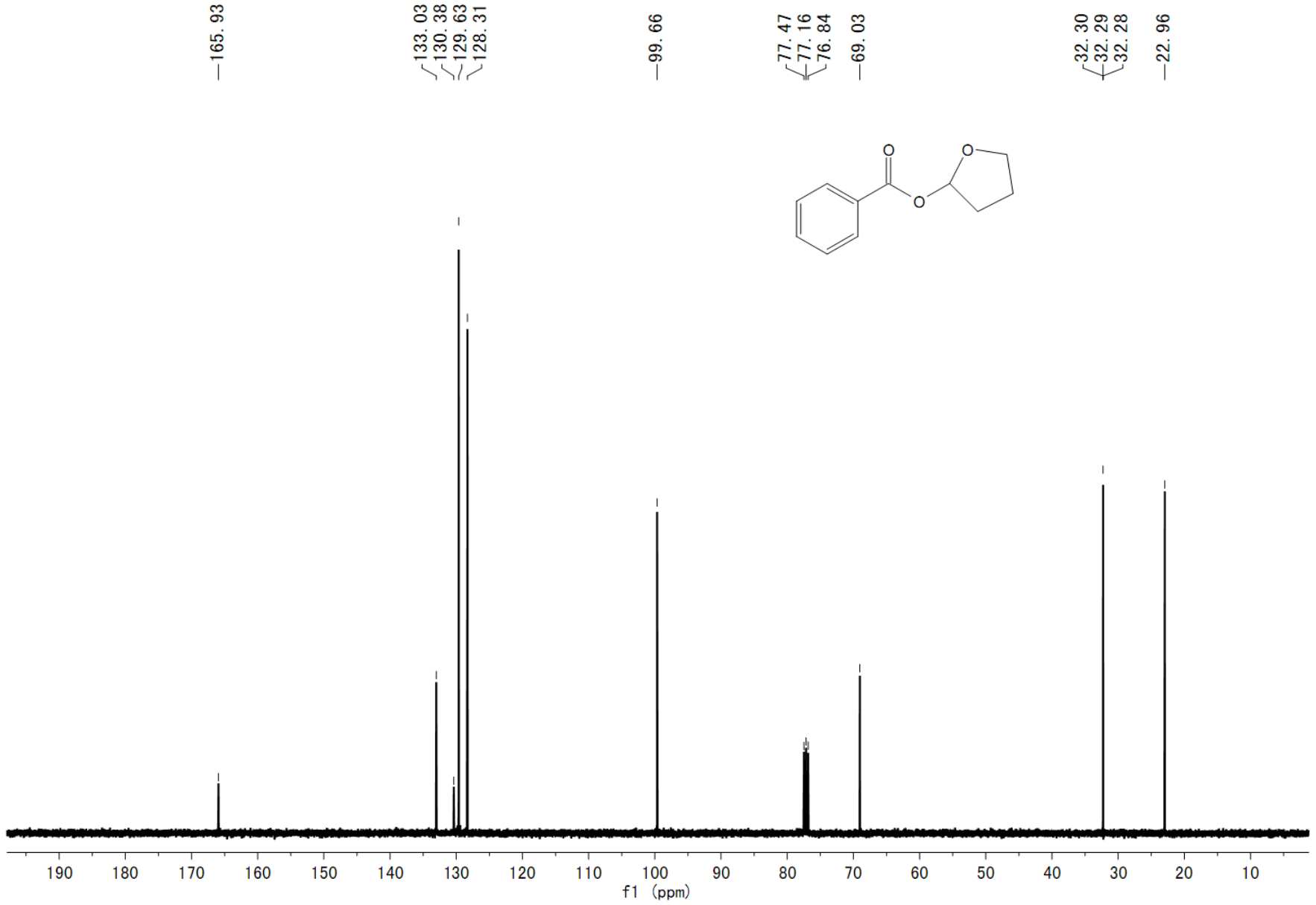
f1 (ppm)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69.03
32.30
32.29
32.28
22.96
O
O
O
190
180
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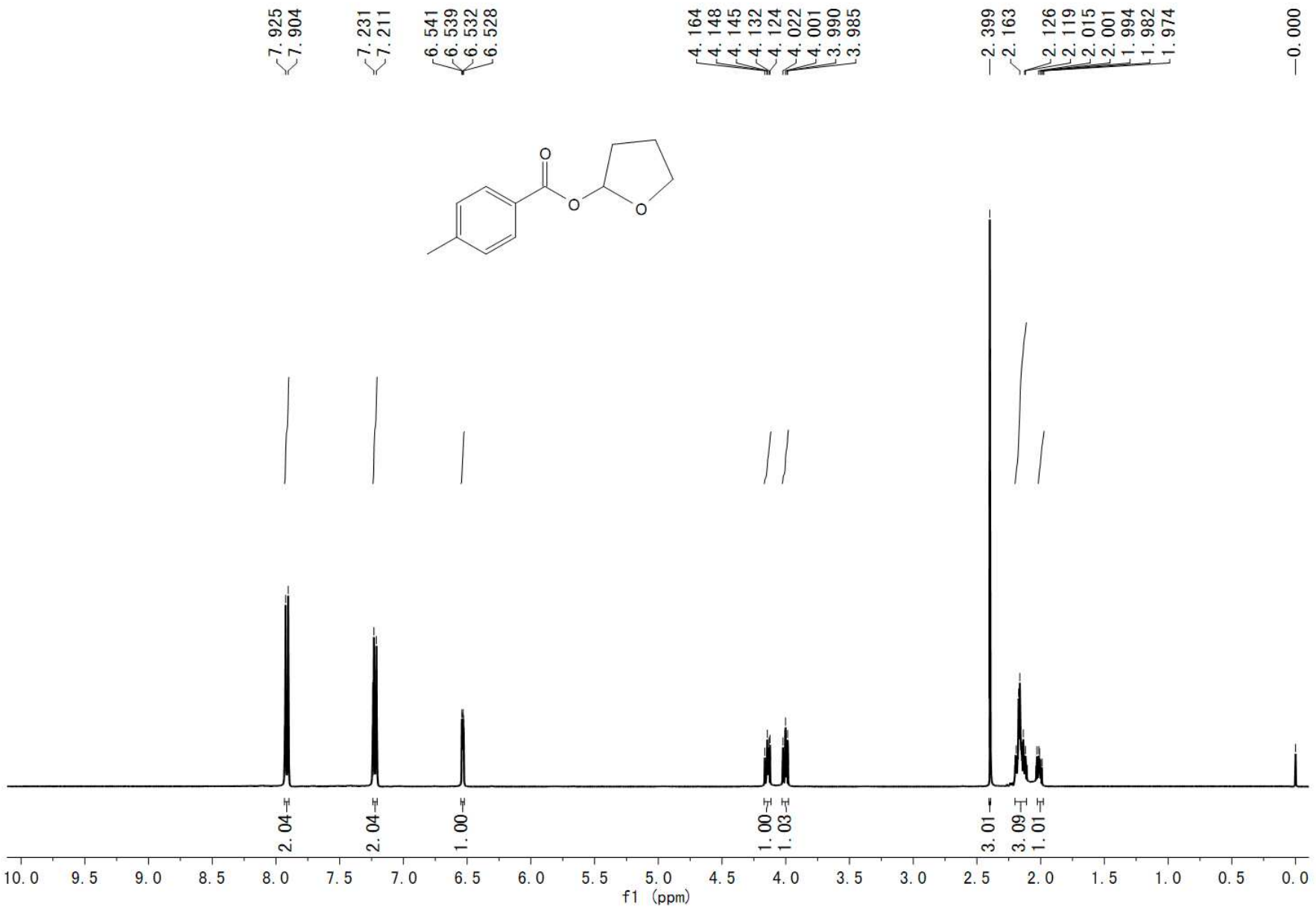
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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6.528
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4.145
4.132
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4.022
4.001
3.990
3.985
2.399
2.163
2.126
2.119
2.015
2.001
1.994
1.982
1.974
0.000
O
O
O
2.04
2.04
1.00
1.00
1.03
3.01
3.09
1.01
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9.0
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7.5
7.0
6.5
6.0
5.5
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3.0
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2.0
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1.0
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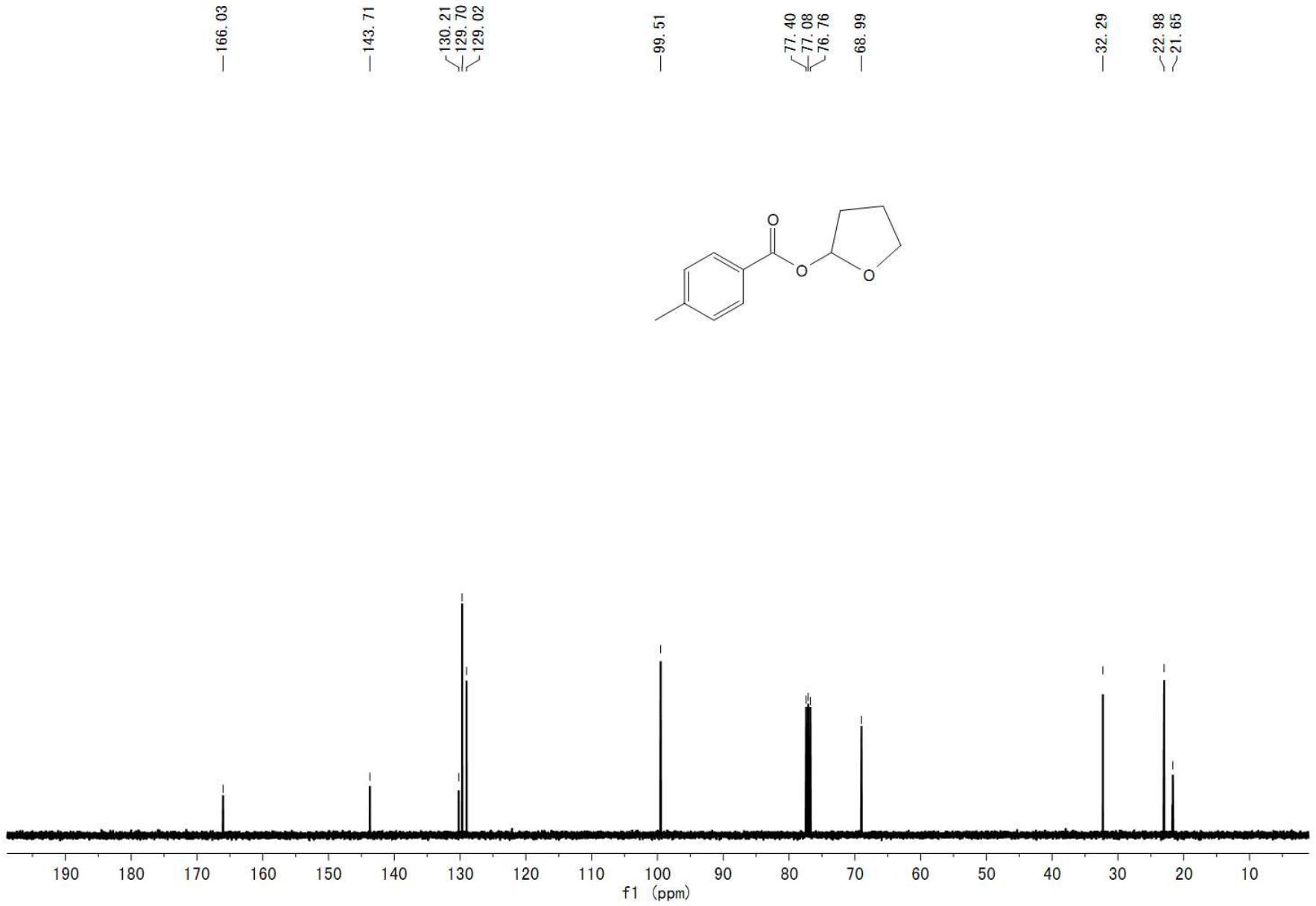
f1 (ppm)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32.29
22.98
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O
O
O
190
180
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140
130
120
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100
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80
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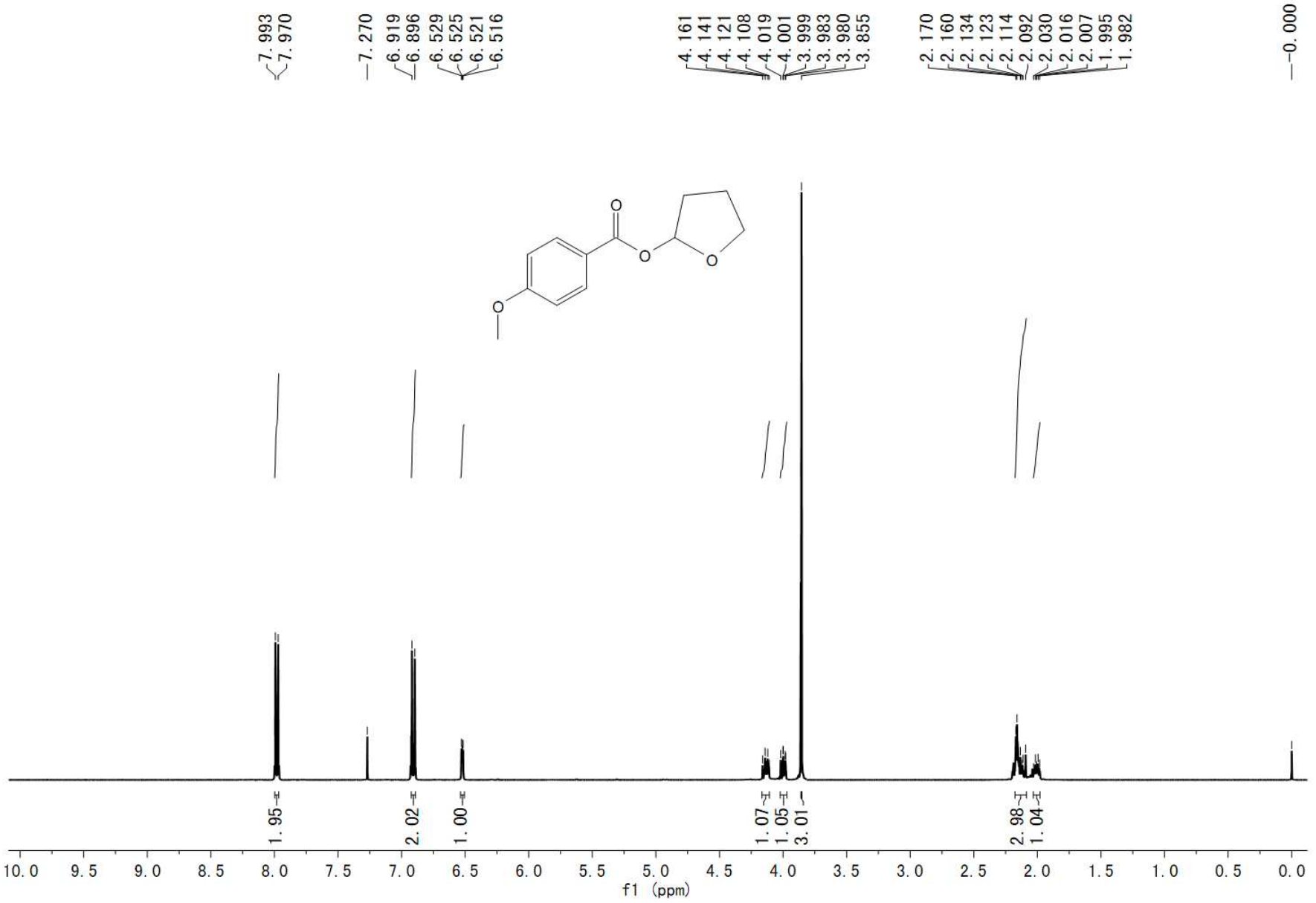
60
50
40
30
20
10
f1 (ppm)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6.521
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4.141
4.121
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1.982
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O
O
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2.02
1.00
1.07
1.05
3.01
2.98
1.04
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9.0
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8.0
7.5
7.0
6.5
6.0
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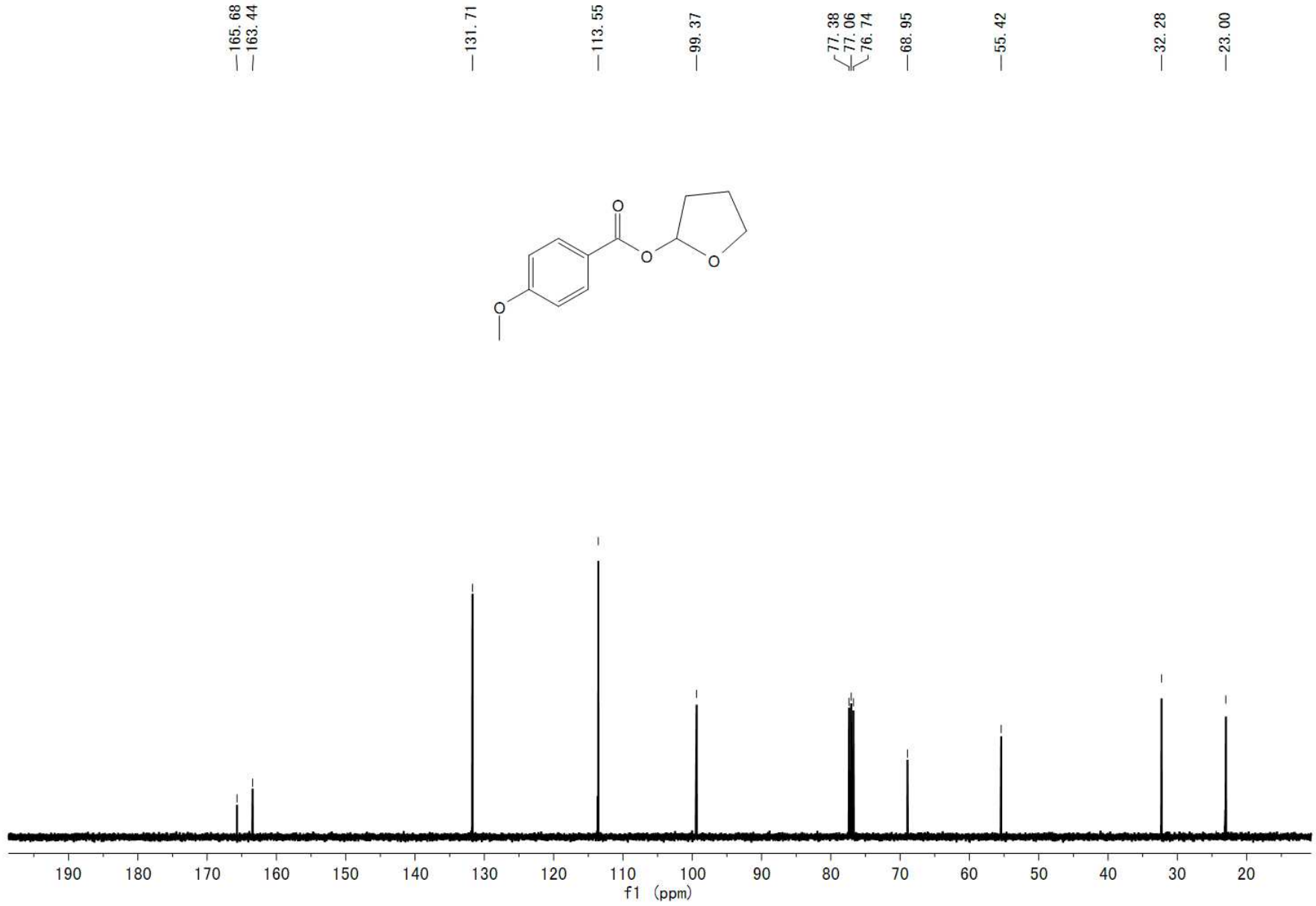
f1 (ppm)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23.00
O
O
O
O
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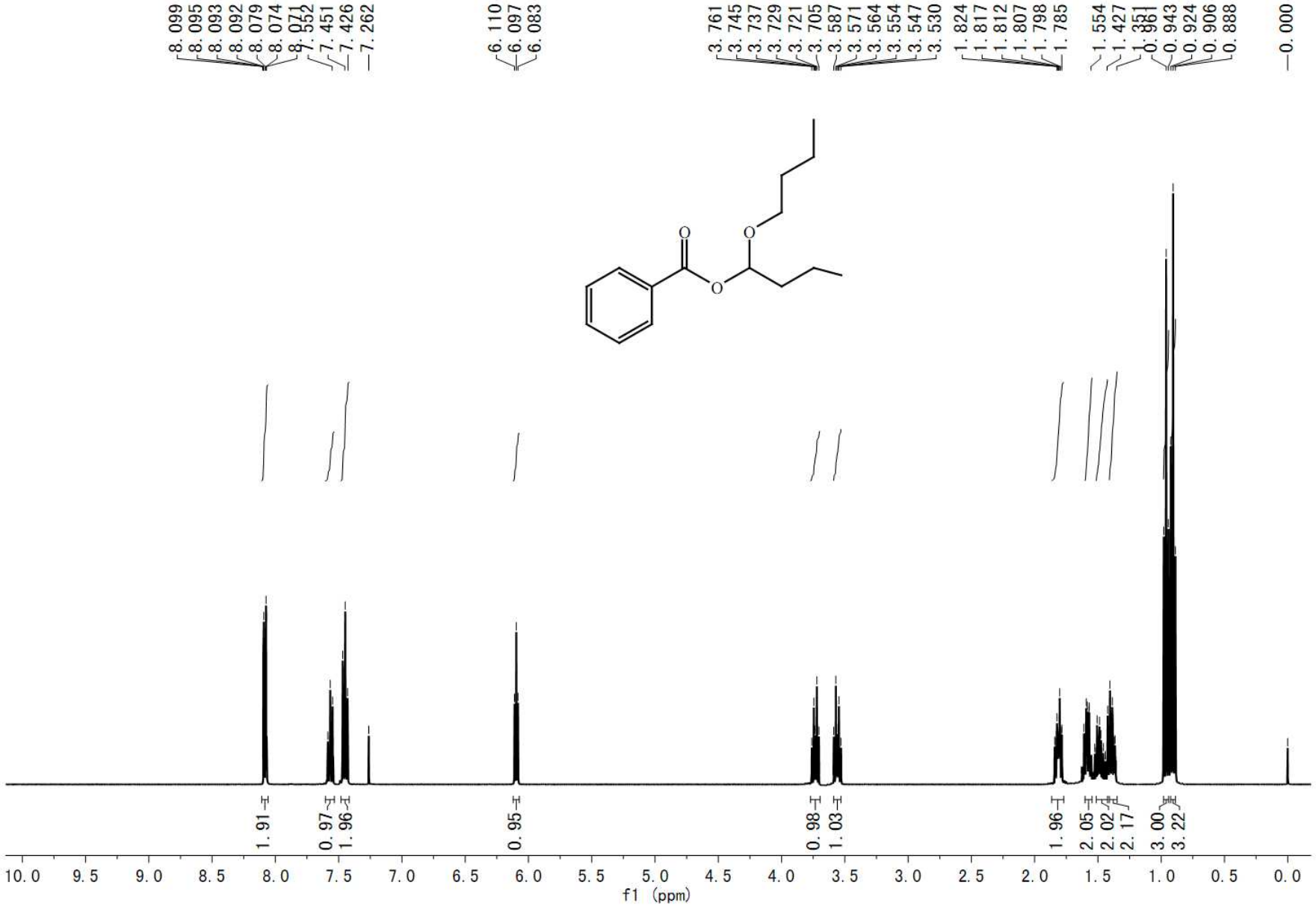
30
20
f1 (ppm)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0.000
O
O
O
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1.96
0.95
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1.03
1.96
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2.02
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3.22
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2.0
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1.0
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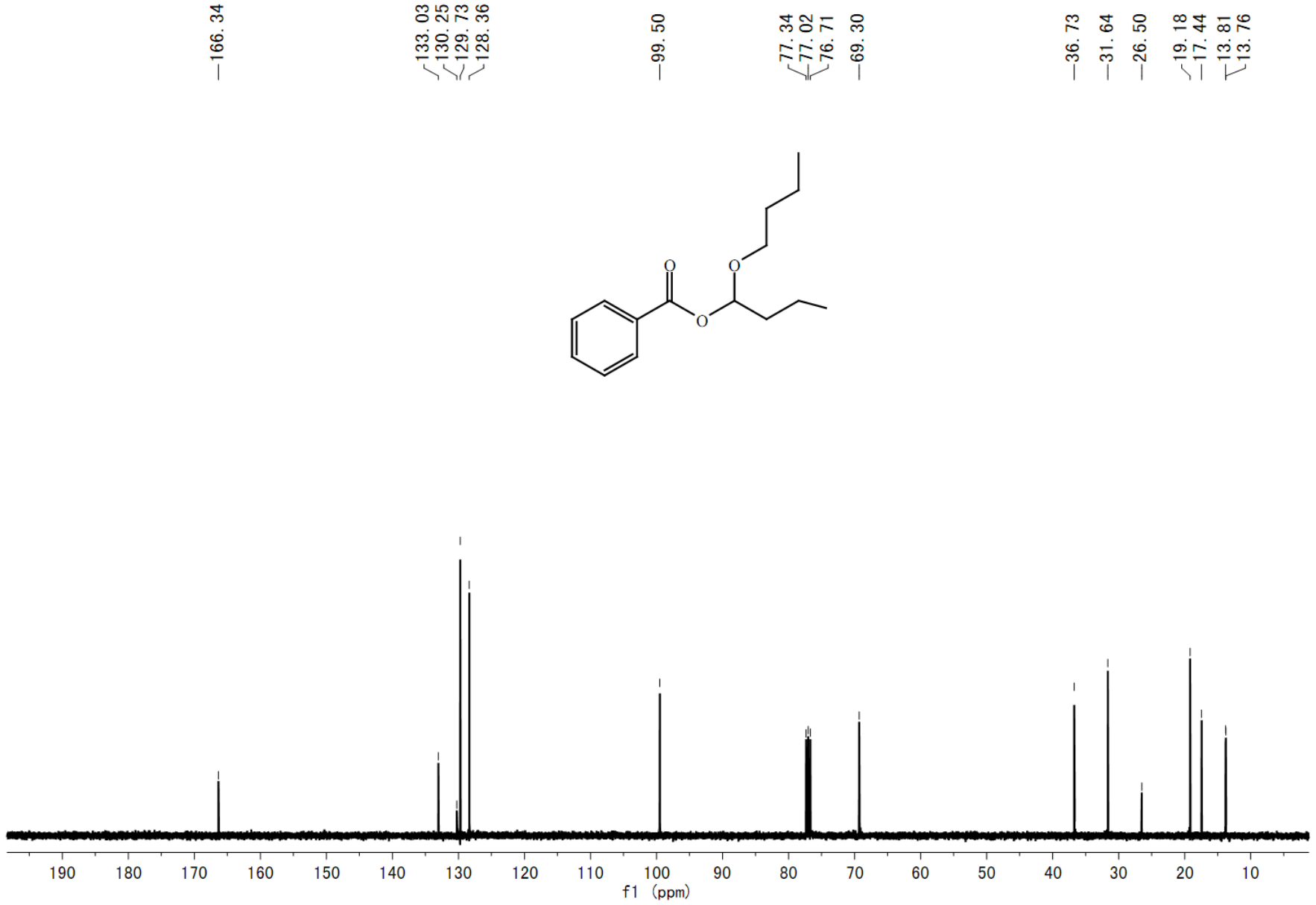
0.0
f1 (ppm)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26.50
19.18
17.44
13.81
13.76
O
O
O
190
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100
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80
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60
50
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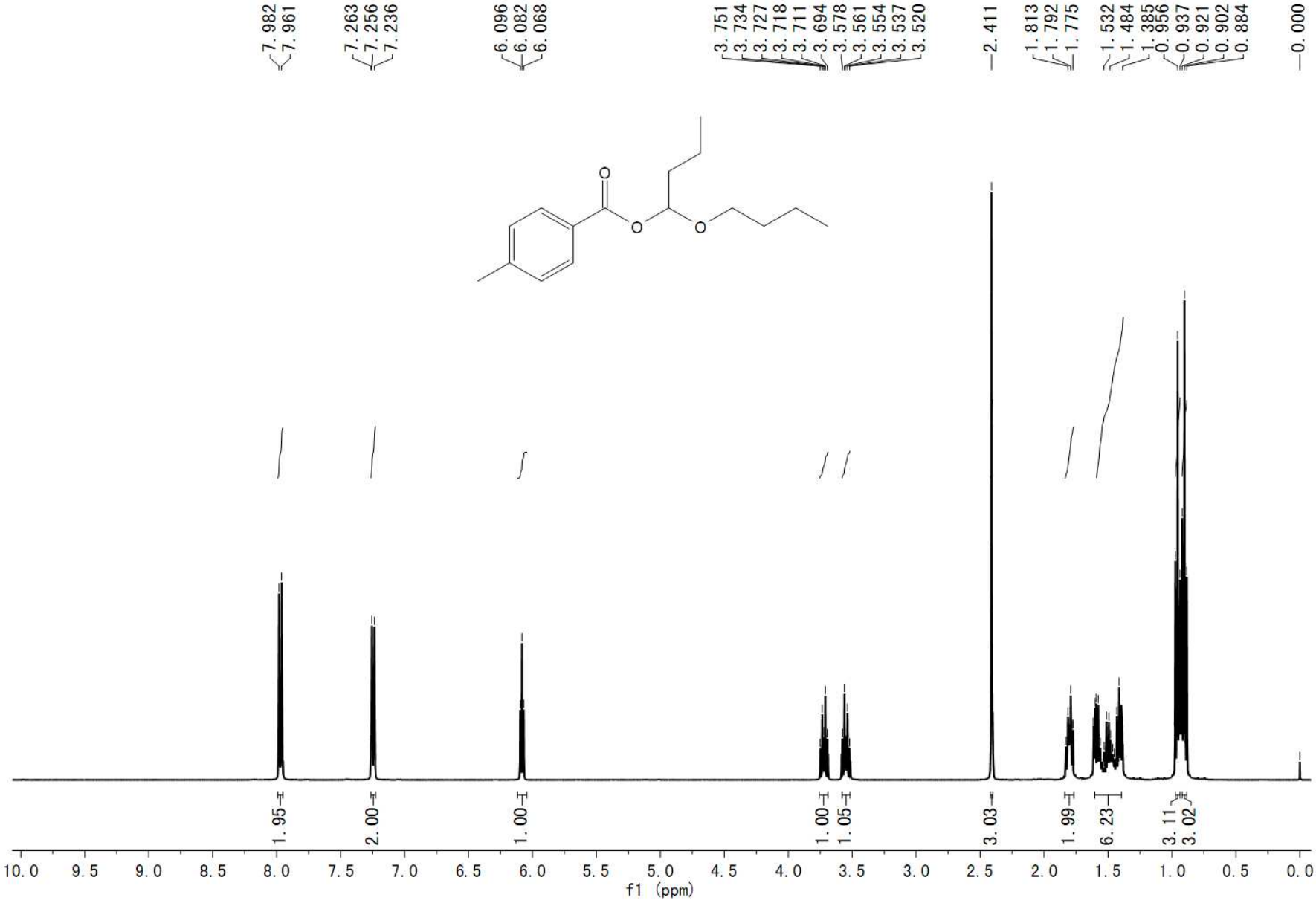
20
10
f1 (ppm)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1.775
1.532
1.484
1.385
0.956
0.937
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0.902
0.884
0.000
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O
O
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2.00
1.00
1.00
1.05
3.03
1.99
6.23
3.11
3.02
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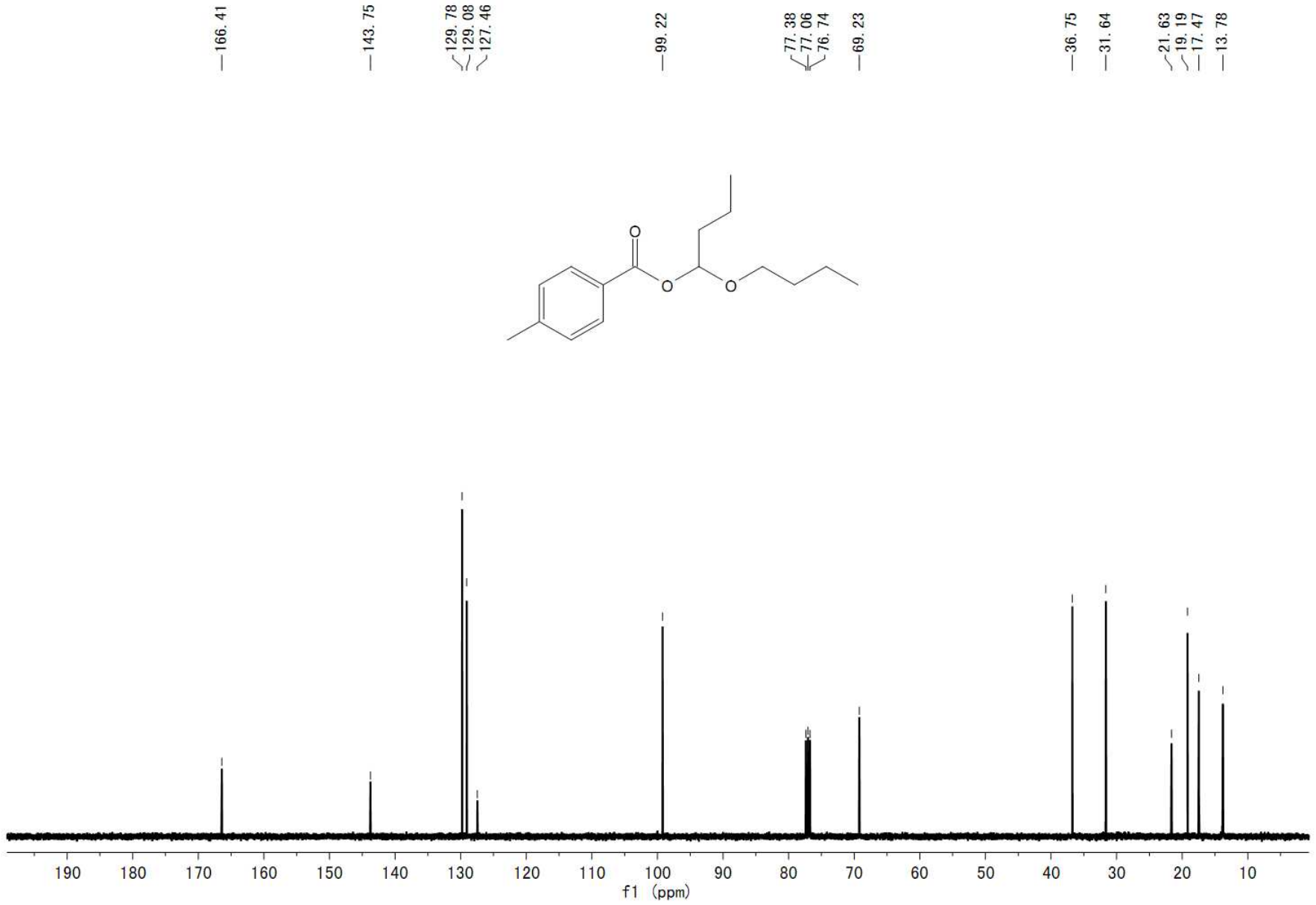
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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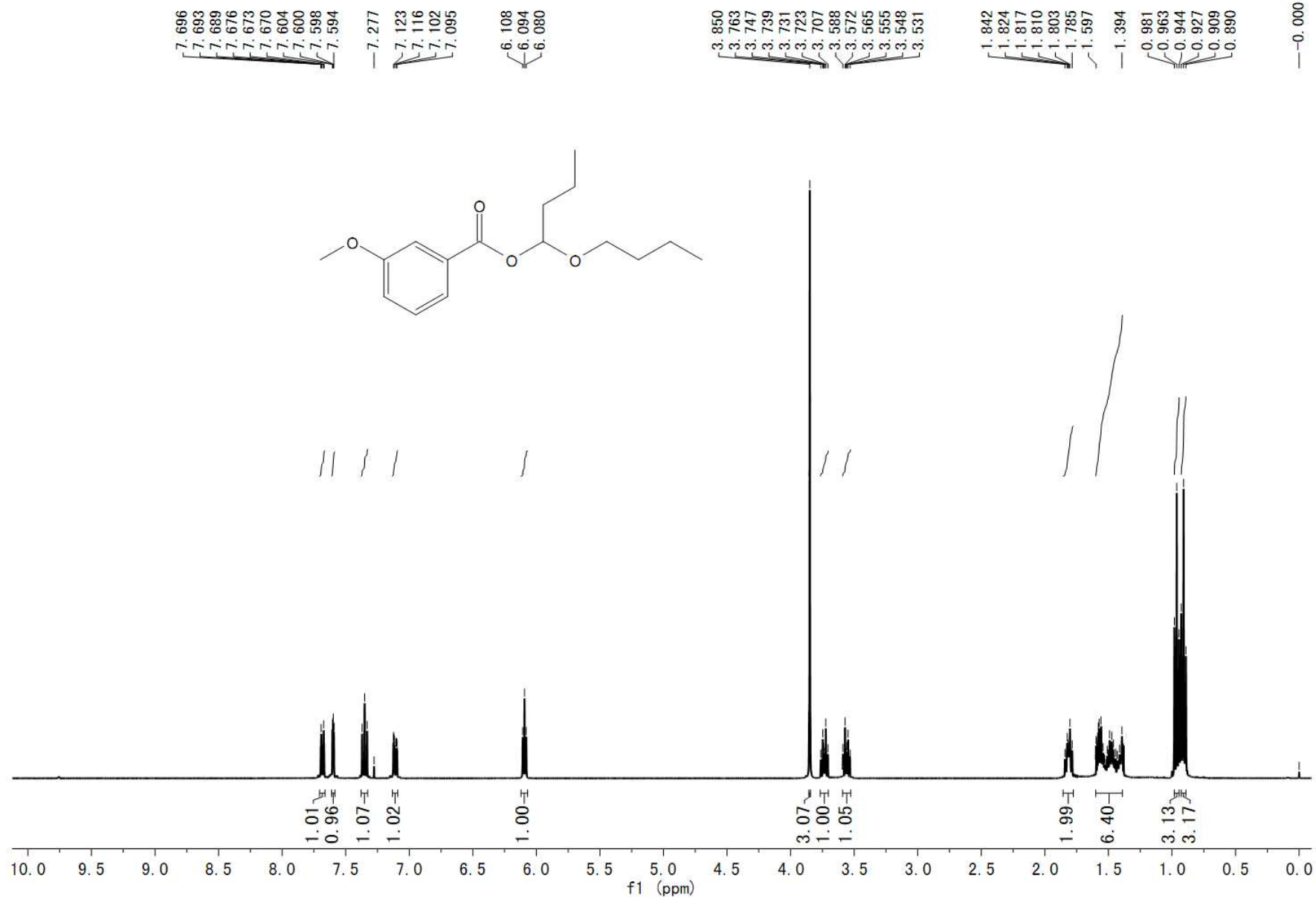
129.78
129.08
127.46
99.22
77.38
77.06
76.74
69.23
36.75
31.64
21.63
19.19
17.47
13.78
O
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.693
7.689
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7.670
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7.600
7.598
7.594
7.277
7.123
7.116
7.102
7.095
6.108
6.094
6.080
3.850
3.763
3.747
3.739
3.731
3.723
3.707
3.588
3.572
3.565
3.555
3.548
3.531
1.842
1.824
1.817
1.810
1.803
1.785
1.597
1.394
0.981
0.963
0.944
0.927
0.909
0.890
0.000
O
O
O
O
1.01
0.96
1.07
1.02
1.00
3.07
1.00
1.05
1.99
6.40
3.13
3.17
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
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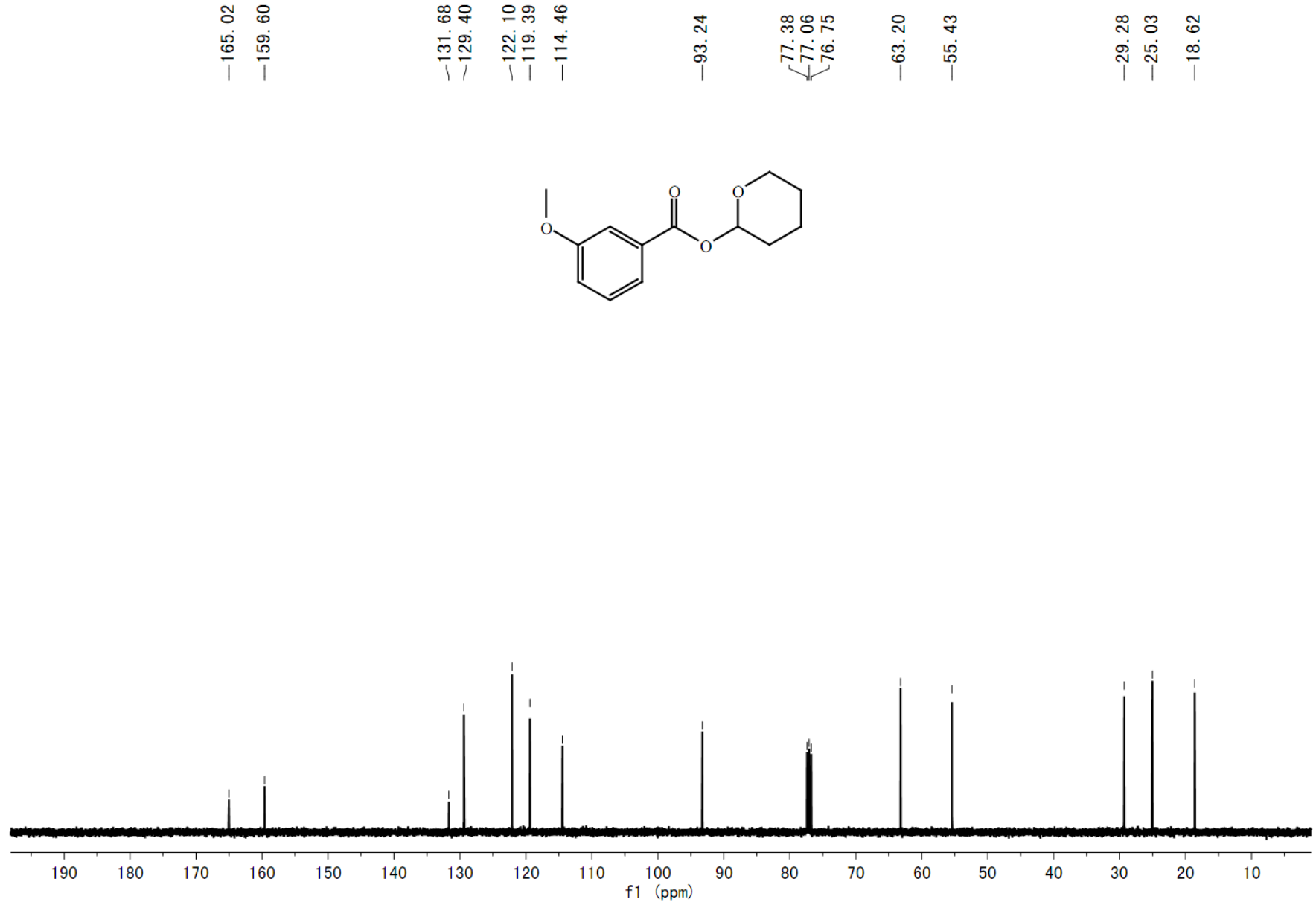
0.0
f1 (ppm)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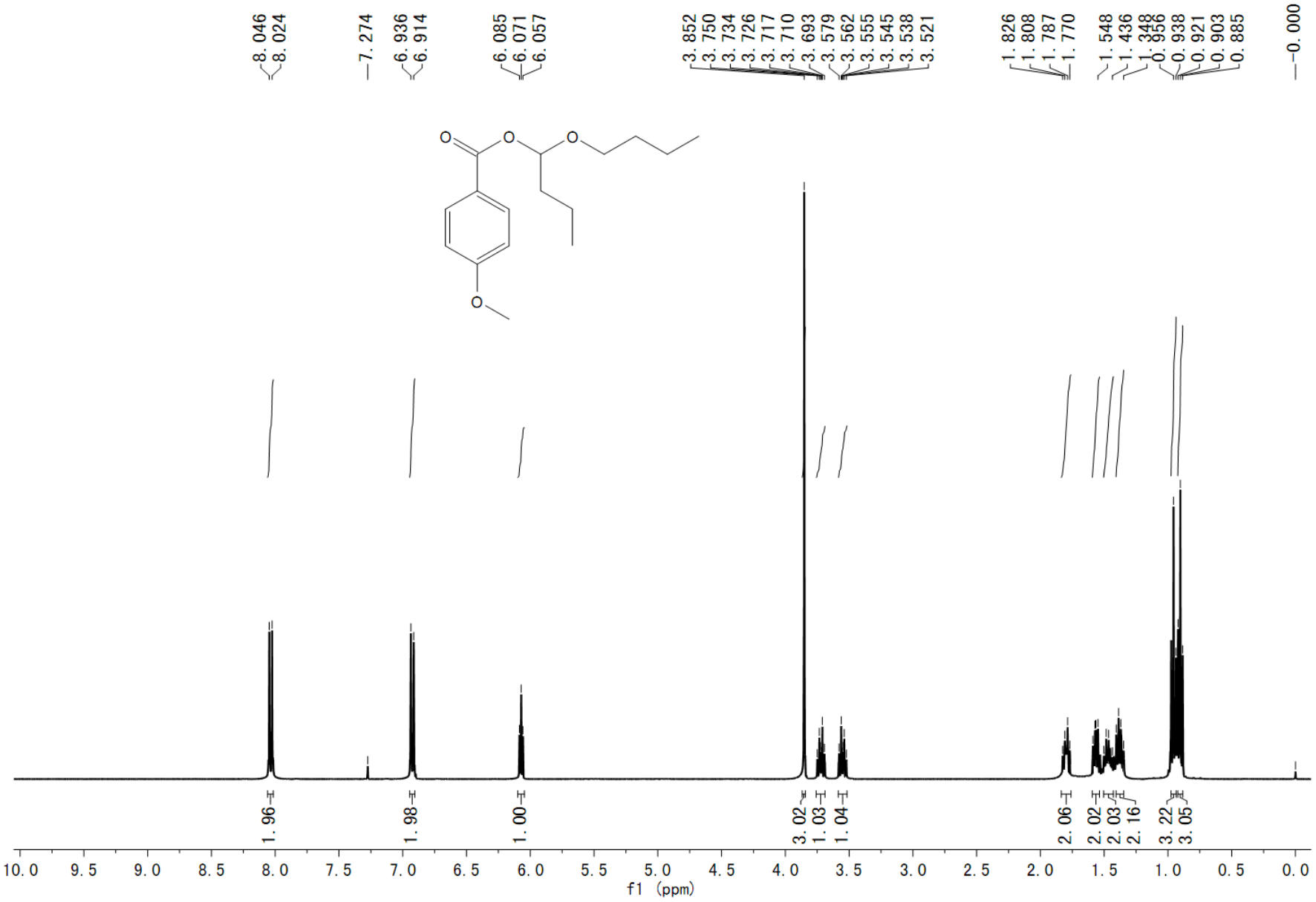
159.60
131.68
129.40
122.10
119.39
114.46
93.24
77.38
77.06
76.75
63.20
55.43
29.28
25.03
18.62
O
O
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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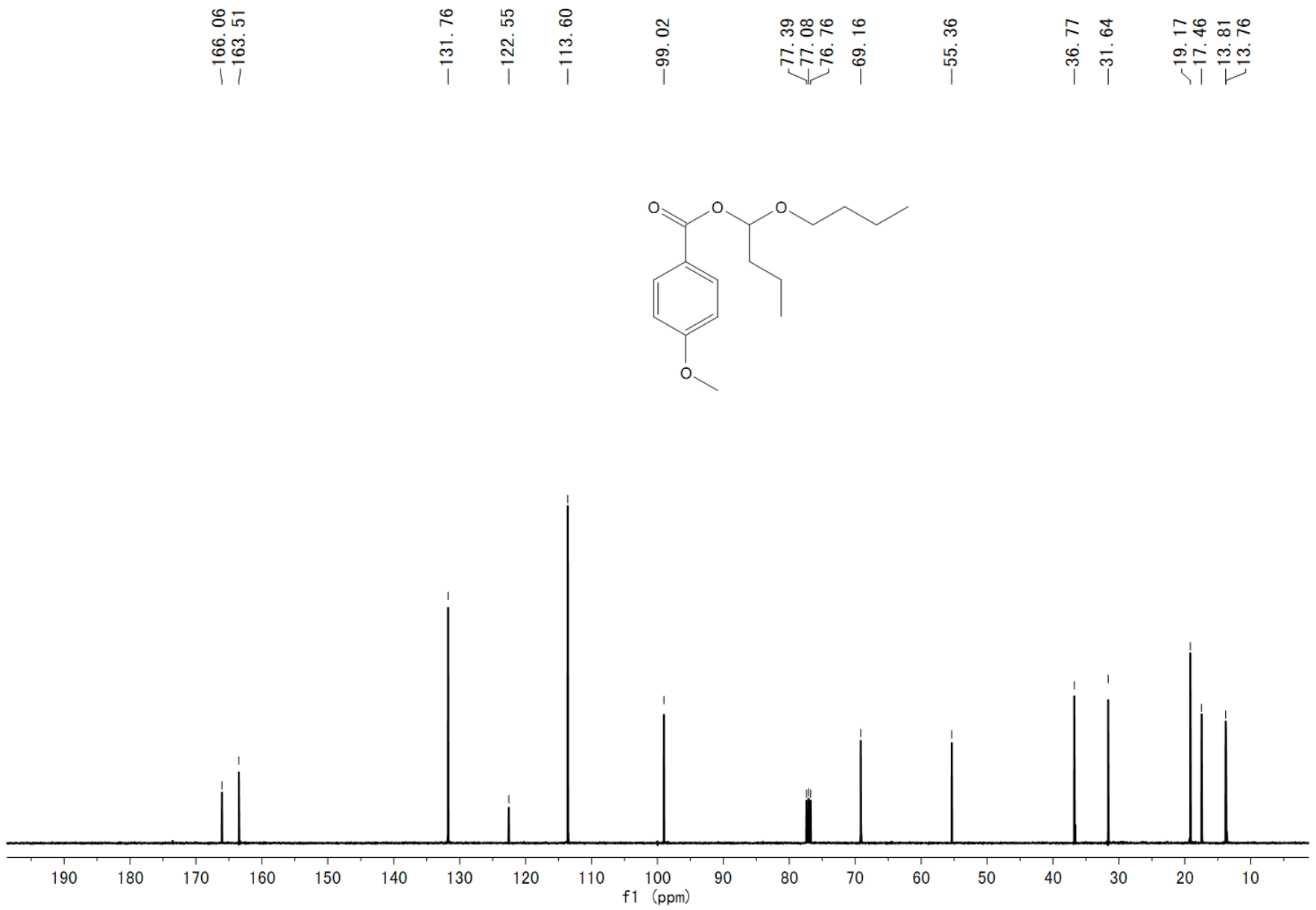
8.024
7.274
6.936
6.914
6.085
6.071
6.057
3.852
3.750
3.734
3.726
3.717
3.710
3.693
3.579
3.562
3.555
3.545
3.538
3.521
1.826
1.808
1.787
1.770
1.548
1.436
1.348
0.956
0.938
0.921
0.903
0.885
0.000
O
O
O
O
1.96
1.98
1.00
3.02
1.03
1.04
2.06
2.02
2.03
2.16
3.22
3.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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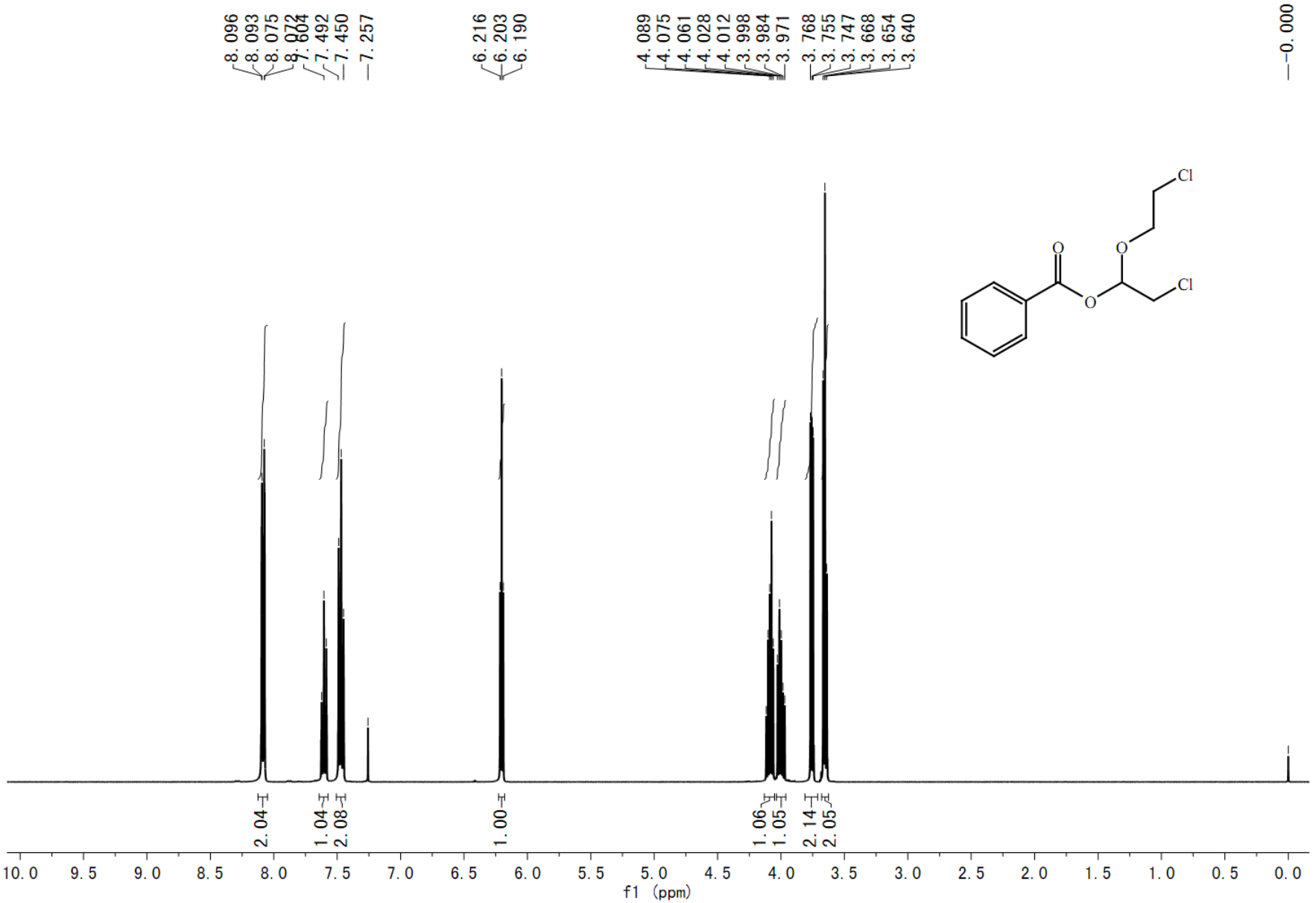
163.51
131.76
122.55
113.60
99.02
77.39
77.08
76.76
69.16
55.36
36.77
31.64
19.17
17.46
13.81
13.76
O
O
O
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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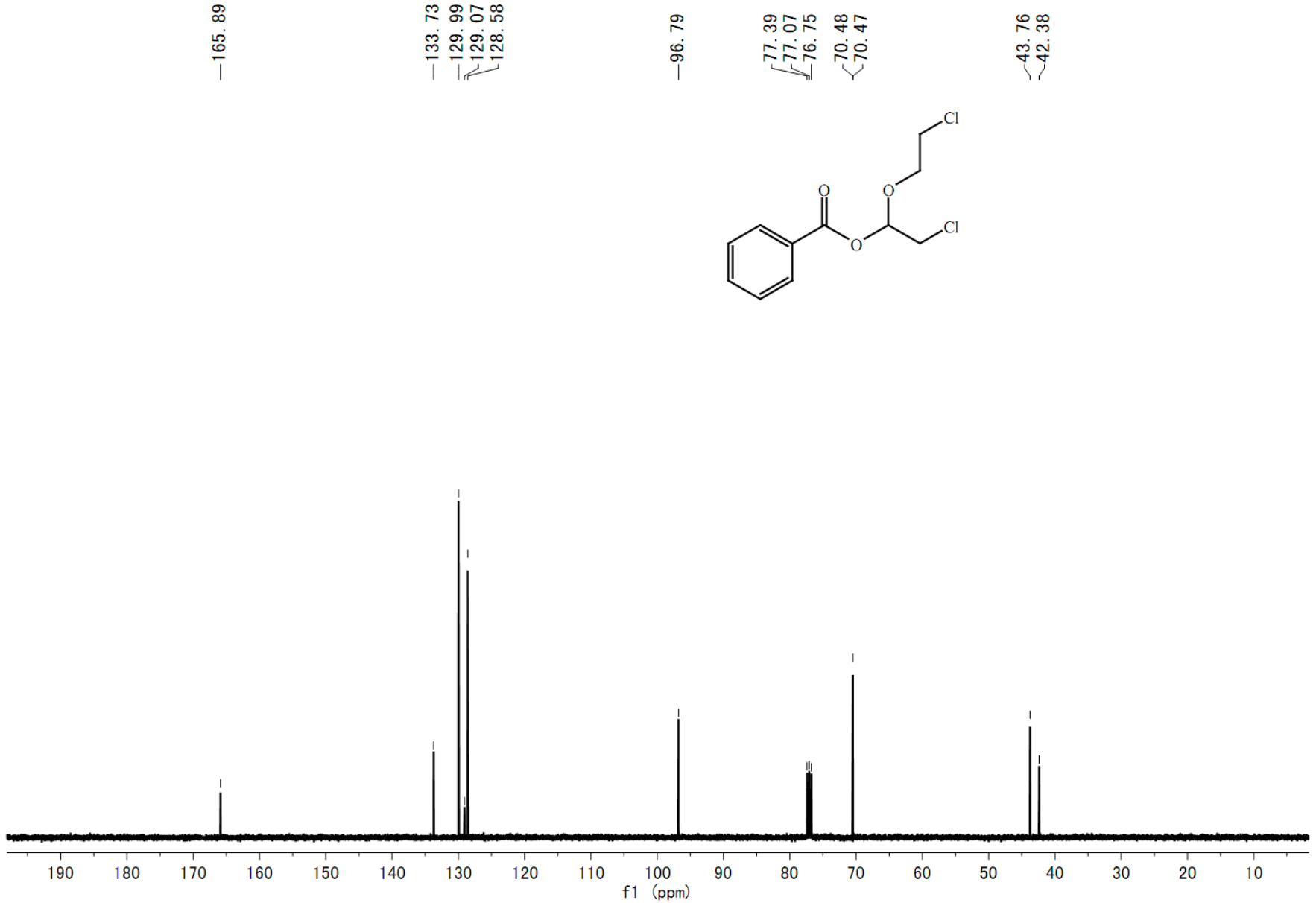
8.093
8.075
8.072
7.604
7.492
7.450
7.257
6.216
6.203
6.190
4.089
4.075
4.061
4.028
4.012
3.998
3.984
3.971
3.768
3.755
3.747
3.668
3.654
3.640
0.000
Cl
O
O
O
Cl
2.04
1.04
2.08
1.00
1.06
1.05
2.14
2.05
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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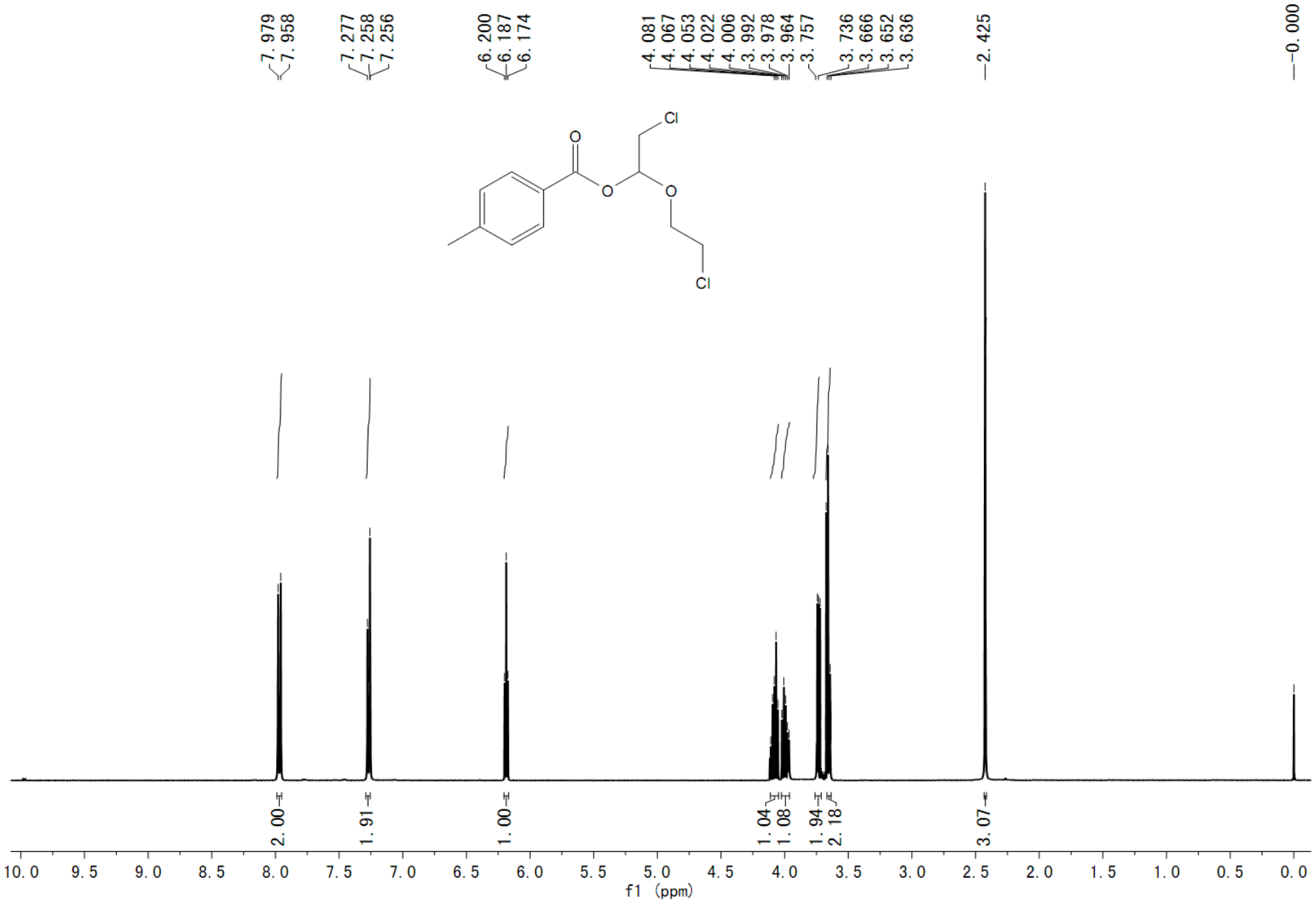
133.73
129.99
129.07
128.58
96.79
77.39
77.07
76.75
70.48
70.47
43.76
42.38
Cl
O
O
O
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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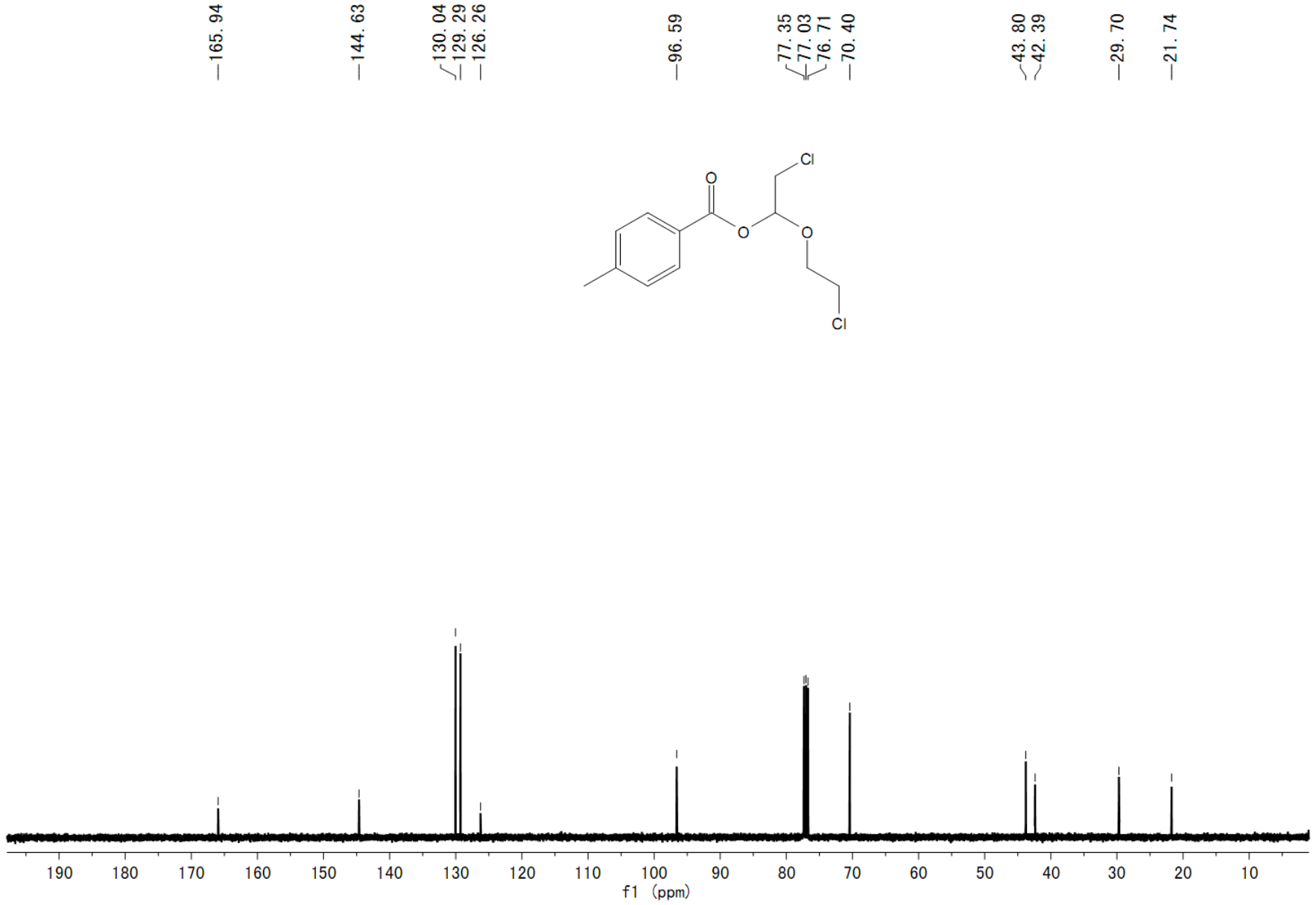
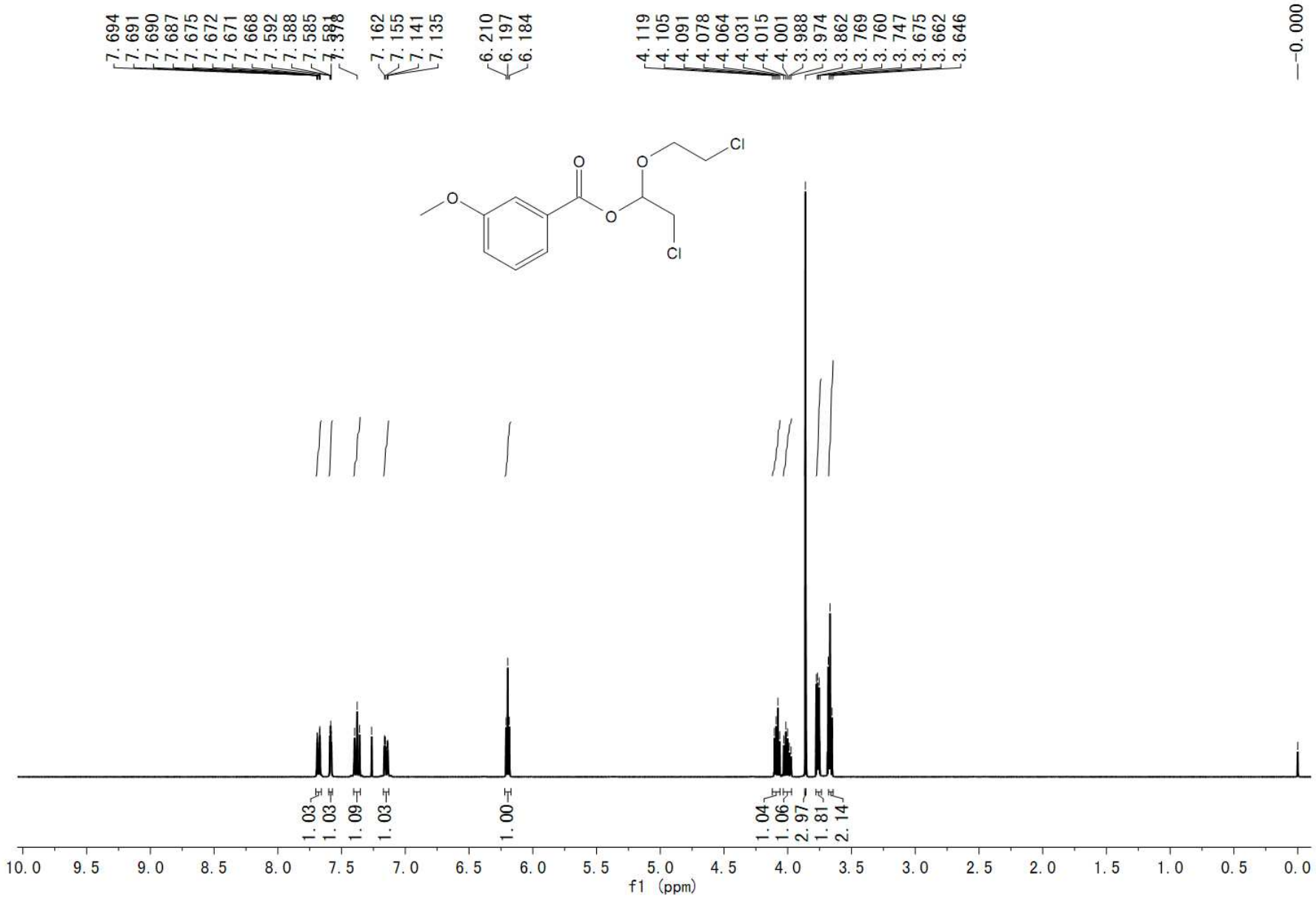
7.277
7.258
7.256
6.200
6.187
6.174
4.081
4.067
4.053
4.022
4.006
3.992
3.978
3.964
3.757
3.736
3.666
3.652
3.636
2.425
0.000
Cl
O
O
O
Cl
2.00
1.91
1.00
1.04
1.08
1.94
2.18
3.07
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.94
144.63
130.04
129.29
126.26
96.59
77.35
77.03
76.71
70.40
43.80
42.39
29.70
21.74
Cl
O
O
O
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.675
7.672
7.671
7.668
7.592
7.588
7.585
7.581
7.378
7.162
7.155
7.141
7.135
6.210
6.197
6.184
4.119
4.105
4.091
4.078
4.064
4.031
4.015
4.001
3.988
3.974
3.862
3.769
3.760
3.747
3.675
3.662
3.646
0.000
O
O
O
Cl
Cl
1.03
1.03
1.09
1.03
1.00
1.04
1.06
2.97
1.81
2.14
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
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1.0
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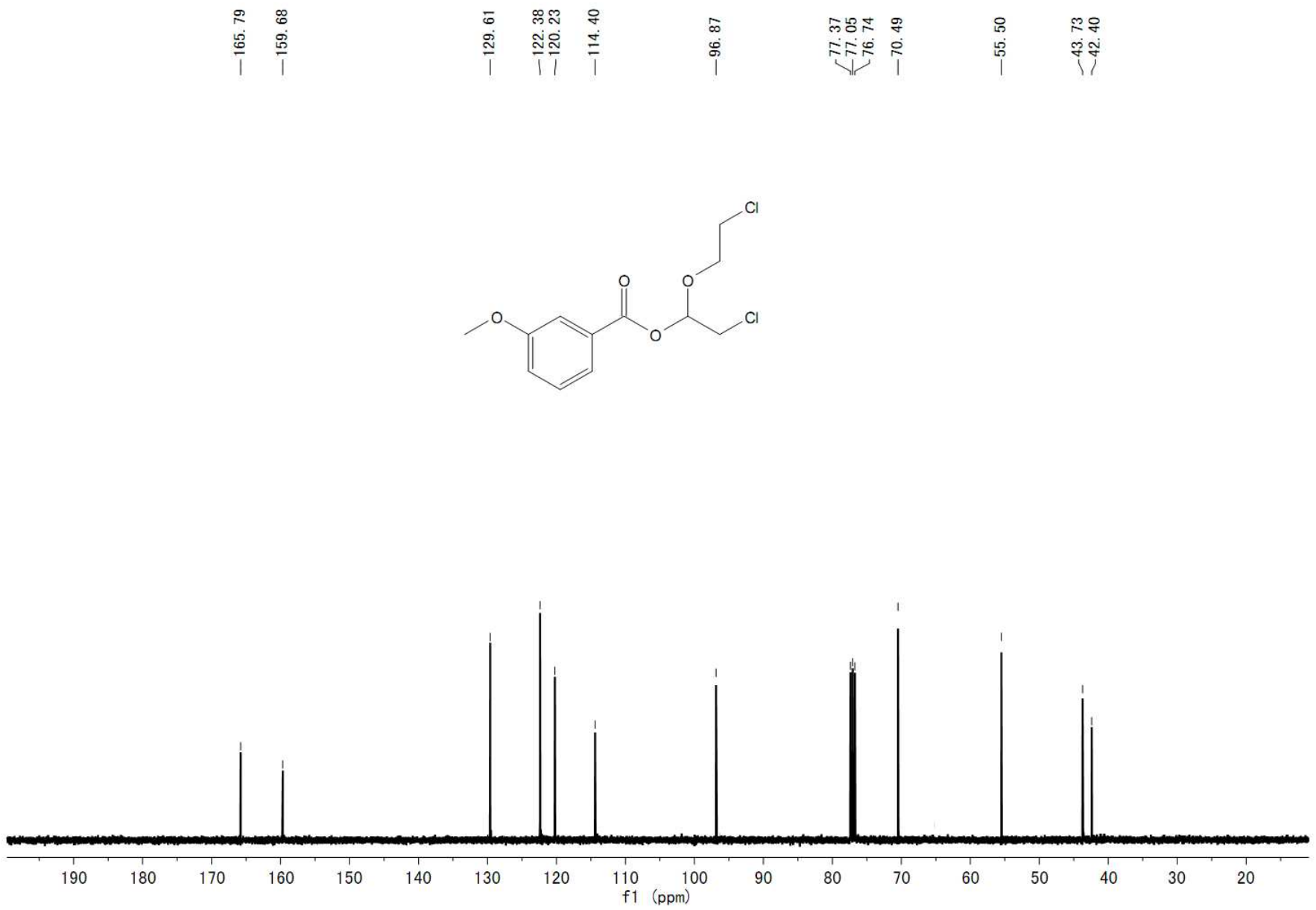
0.0
f1 (ppm)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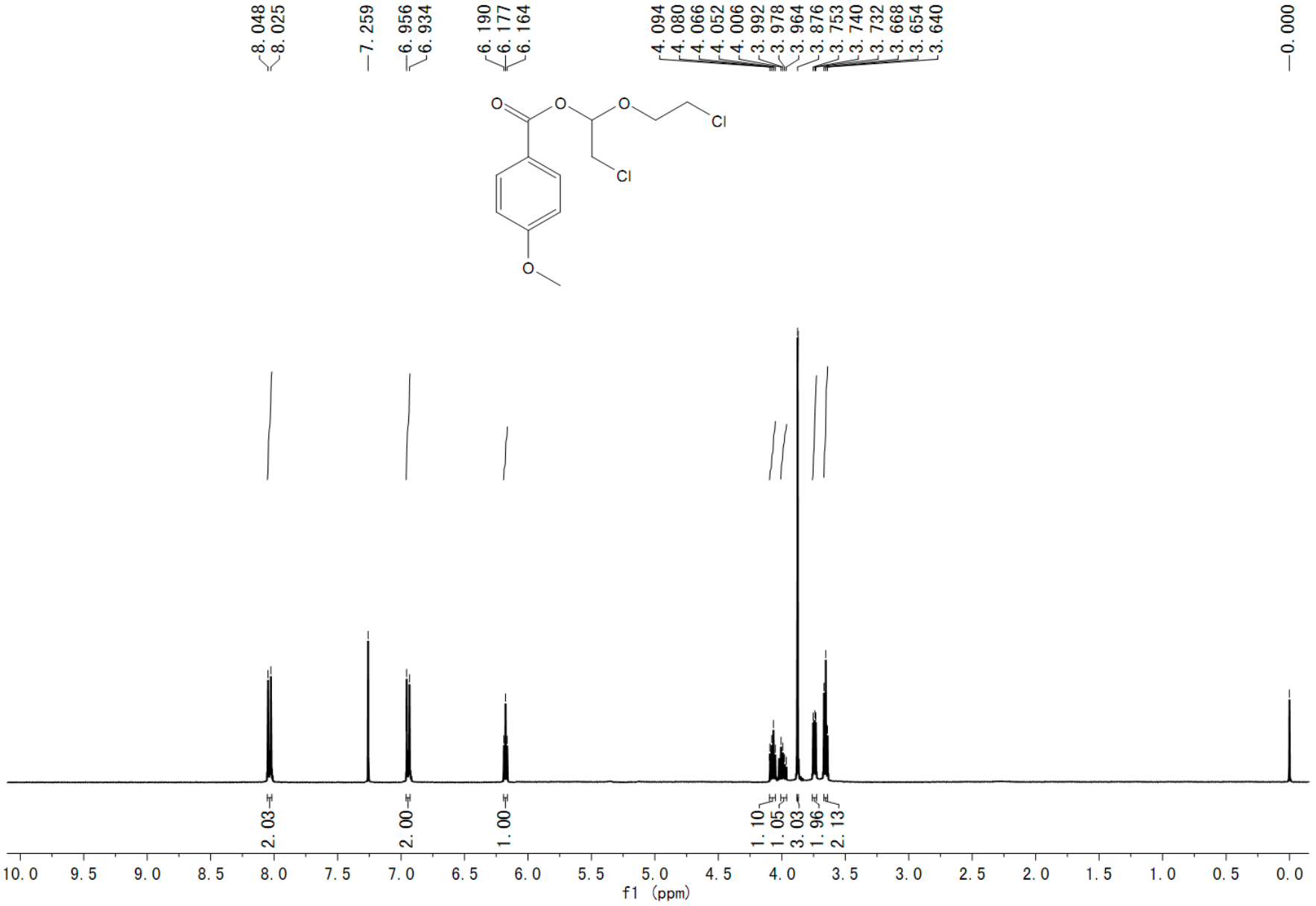
122.38
120.23
114.40
96.87
77.37
77.05
76.74
70.49
55.50
43.73
42.40
Cl
O
O
O
O
Cl
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


8.048
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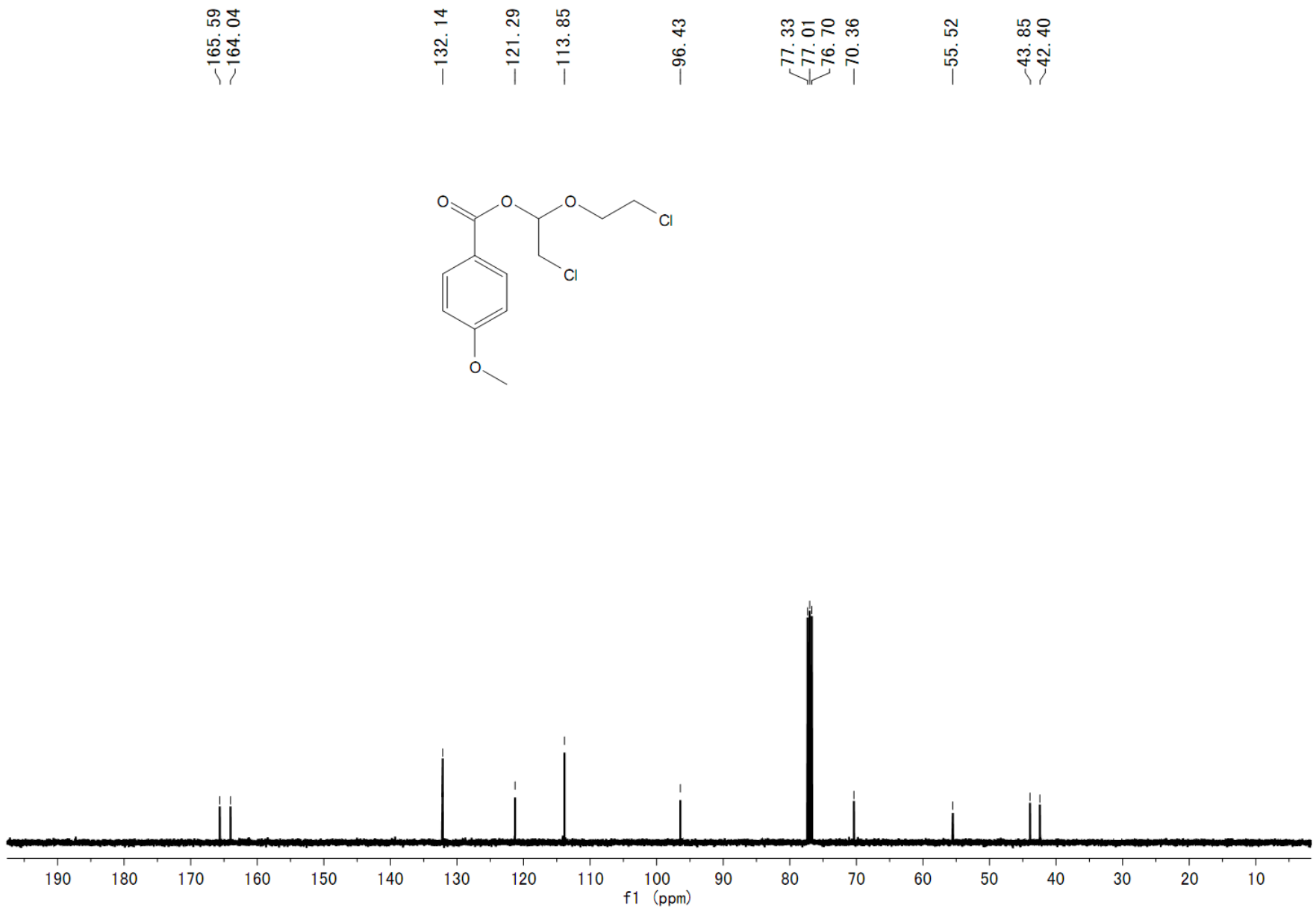
6.934
6.190
6.177
6.164
4.094
4.080
4.066
4.052
4.006
3.992
3.978
3.964
3.876
3.753
3.740
3.732
3.668
3.654
3.640
0.000
O
O
O
Cl
Cl
O
2.03
2.00
1.00
1.10
1.05
3.03
1.96
2.13
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


165.59
164.04
132.14
121.29
113.85
96.43
77.33
77.01
76.70
70.36
55.52
43.85
42.40
O
O
O
Cl
Cl
O
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)