Supporting Information

Catalytic Enantioselective Desymmetrization of meso-Glutaric Anhydrides Using a Stable Ni_2-Schiff Base Catalyst

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1. General

The reactions were performed in an oven-dried 10 mL test tube with a Teflon-coated magnetic stirring bar unless otherwise noted. All work-up and purification procedures were carried out with reagent-grade solvents under ambient atmosphere.

2. Instrumentation

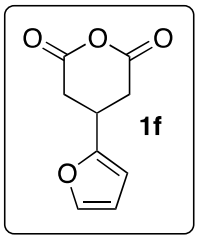
Infrared (IR) spectra were recorded on a JASCO FT/IR 4100 Fourier transform infrared spectrophotometer. NMR (^{1}H NMR: 400 MHz, ^{13}C NMR: 100 MHz) was recorded on JEOL ECS-400 spectrometers. Chemical shifts for proton are reported in parts per million downfield from tetramethylsilane and are referenced to residual protium in the NMR solvent ($CDCl_3$: δ 7.26 ppm). For ^{13}C NMR, chemical shifts were reported in the scale relative to NMR solvent ($CDCl_3$: δ 77.0 ppm) as an internal reference. NMR data are reported as follows: chemical shifts, multiplicity (s: singlet, d: doublet, dd: doublet of doublets, t: triplet, q: quartet, m: multiplet, br: broad signal), coupling constant (Hz), and integration. Optical rotation was measured using a 2 mL cell with a 1.0 dm path length on a JASCO polarimeter P-1030. High-resolution mass spectra (ESI-Orbitrap) were measured on ThermoFisher Scientific LTQ Orbitrap XL. HPLC analysis was conducted on a JASCO HPLC system equipped with Daicel chiral-stationary-phase columns (ϕ 0.46 cm × 25 cm).

3. Materials

Unless otherwise noted, materials were purchased from commercial suppliers and were used without purification. THF, diethyl ether, toluene and CH_2Cl_2 were purified by passing through a solvent purification system (Glass Contour). Dry $CHCl_3$ and Ni_2-(Schiff base) catalyst were purchased from Wako Pure Chemical Co. Ltd. (Osaka, Japan) and used as received. 3-Methyl glutaric anhydride was purchased form TCI Chemical Industries Co. Ltd (Tokyo, Japan). 3-Phenyl glutaric acid was purchased from Aldrich and used for the synthesis of anhydride **1c** as reported.[1] Anhydrides **1b**,[2] **1d**,[3] **1e**,[4] and **1g**[5] were synthesized using literature procedure. Anhydride **1f** is not reported and was synthesized using the same procedure as of compound **1e**.

(1) Reed, P. E.; Katzenellenbogen, J. A. *J. Med. Chem.* **1991**, *34*, 1162.
(2) Poldy, J.; Peakall, R.; Barrow, R. A. *Tetrahedron Lett.* **2008**, *49*, 2446.
(3) Yamamoto, Y.; Iwasa, M.; Sawada, S.; Oda, J.; *Agric. Biol. Chem*. **1990**, *54*, 3269.
(4) Fryszkowska, A.; Komar, M.; Koszelewski, D.; Ostaszewski, R. *Tetrahedron. Assym*. **2005**, *16*, 2475.
(5) Watanabe, Y.; Yamazaki, T.; Kubota, T.; *Org. Lett*. **2010**, *12*, 268.

4-(furan-2-yl)dihydro-2*H*-pyran-2,6(3*H*)-dione (1f)



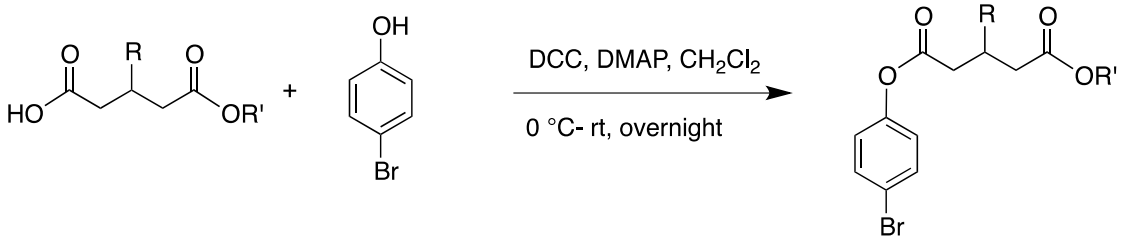
Light yellow solid; mp: 84-86 °C; IR (neat): ν 1815, 1752, 1508, 1268 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.31 (d, *J* = 1.6 Hz, 1H), 6.27 (dd, *J* = 2.0 Hz, 3.2 Hz, 1H), 6.10 (d, J= 3.2 Hz, 1H), 3.51-3.45 (m, 1H), 3.04-2.90 (m, 4H); ^{13}C NMR ($CDCl_3$): δ 165.4, 152.1, 142.8, 110.5, 106.2, 34.5, 28.1; HRMS (ESI- Orbitrap) calcd. for $C_9H_9O_4$ *m/z* 181.0495 $[M+H]^+$, found 181.0493.

General Procedure for the desymmetrization reaction

To a oven-dried glass test tube equipped with a magnetic stirring bar was charged the corresponding anhydride (0.2 mmol), Ni_2-(Schiff base) ((*R*)-isomer unless otherwise noted, 0.01 mmol, 5 mol%) and $CHCl_3$ (0.4 mL. 0.5 M).[a] The reaction mixture was then cooled to the given temperature, followed by the addition of the corresponding alcohol (2 mmol, 10 equiv). The reaction mixture was then stirred for the time as indicated in the table. Dichloromethane (2 mL) was added to the reaction mixture and then extracted with sat. $NaHCO_3$ (2 × 2 mL). The aquoeus layer collected was then washed with dichloromethane (2 × 2 mL) and then acidified to pH = 1.0 using 1N HCl. The compound was then extracted with ethyl acetate (3 × 10 mL), dried over Na_2SO_4 and evaporated under reduced presuure to furnish the compound in very good purity.

[a] In case of anhydride **1g**, 4 mL (0.05 M) of $CHCl_3$ was used.

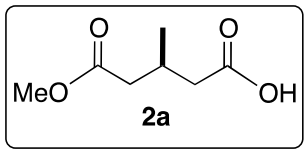
The ee value was determined using chiral HPLC analysis by converting the carboxylic acid to *p*-bromo phenol ester using literature procedure.[6]



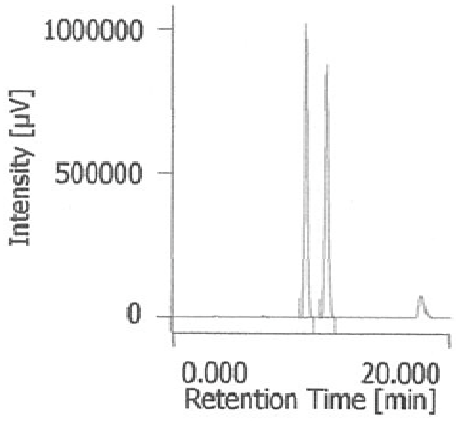
Absolute configuration of **2a**, **2c**, and **2e** was determined by comparing signs of optical rotation (and chiral HPLC profiles for **2a**) with those reported on literature. For the others, it was correlated by analogy.

(6) Manzano, R.; Andrés, J. M.; Muruzábal, M.-D.; Pedrosa, R. *J. Org. Chem.* **2010**, *75*, 5417.

Methyl (*S*)-3-methylglutarate (2a) is reported.[6] Yield = 91%; ee = 94%. $[\alpha]_D^{23}$ -0.7 (*c* 0.20, $CHCl_3$, 94% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 9.5 min (major), t_R = 11.3 min (minor), (lit.[6] t_R = 9.5 min ((*S*)-isomer), t_R = 11.3 min ((*R*)-isomer) under the same conditions for HPLC).



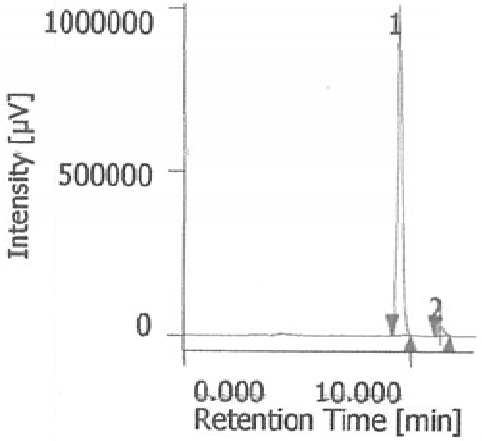
Racemic sample



ピーク情報

ピーク名	tR [min]	area%
Unknown	9.533	50.003
Unknown	11.008	49.997

Chiral sample "*R*-cat"

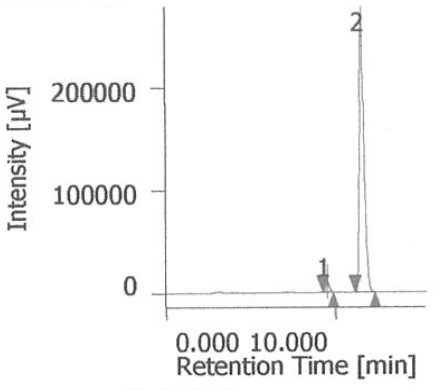


ピーク情報

ピーク名	tR [min]	面積%
Unknown	9.442	96.931
Unknown	11.300	3.069

Methyl (*R*)-3-methylglutarate (*ent*-2a) (product of (*S*)-Ni_2-(Schiff base)-catalyzed reaction) Yield = 88%; ee = 94%. $[\alpha]_D^{23}$ +0.9 (*c* 0.17, $CHCl_3$, 94% ee,), (lit.[6] $[\alpha]_D^{25}$ +0.6 (*c* 1.0, $CHCl_3$, 84% ee)); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 9.5 min (minor), t_R = 11.5 min (major).

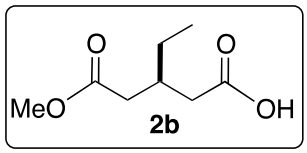
Chiral sample "*S*-cat"



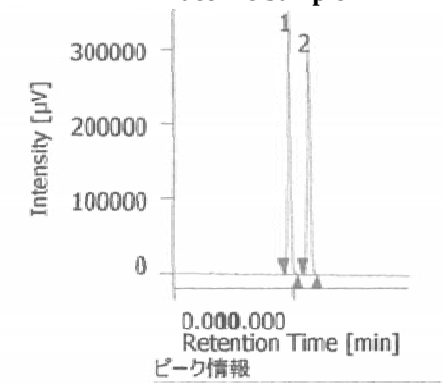
ピーク情報

ピーク名	tR [min]	面積%
Unknown	9.500	3.005
Unknown	11.542	96.995

Methyl 3-ethylglutarate (2b) is reported.[7] Yield = 99%; ee = 90%. HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 9.6 min (major), t_R = 11.2 min (minor).



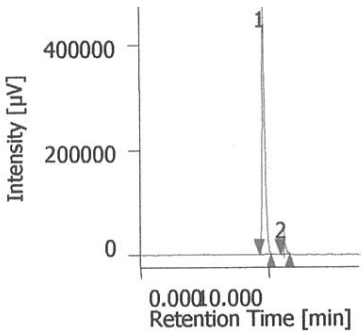
Racemic Sample



ピーク情報

ピーク名	tR [min]	面積%
Unknown	9.567	50.112
Unknown	11.150	49.888

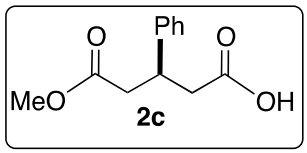
Chiral Sample



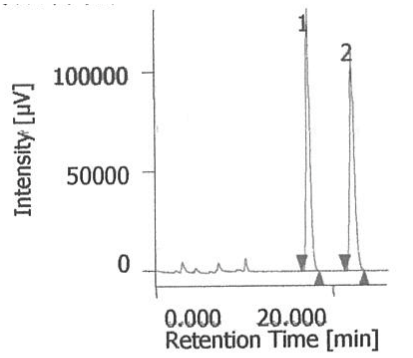
ピーク情報

ピーク名	tR [min]	面積%
Unknown	9.558	95.013
Unknown	11.150	4.987

Methyl (*S*)-3-phenylglutarate (2c) is reported.[8] Yield = 93%; ee = 80%. $[\alpha]_D^{23}$ -2.8 (*c* 0.83, $CHCl_3$, 80% ee,), (lit.[4] $[\alpha]_D^{20}$ -3.6 (*c* 1.1, $CHCl_3$)); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 17.1 min (major), t_R = 22.0 min (minor).



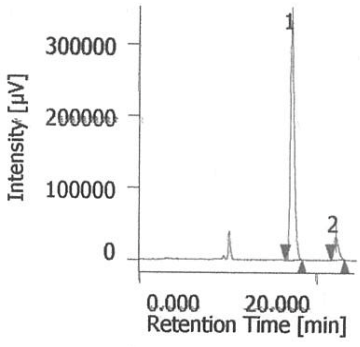
Racemic Sample



ピーク情報

ピーク名	tR [min]	面積%
Unknown	17.142	49.997
Unknown	22.067	50.003

Chiral Sample

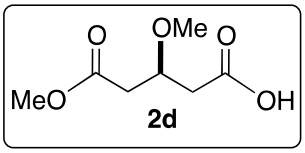


ピーク情報

ピーク名	tR [min]	面積%
Unknown	17.058	89.825
Unknown	22.050	10.175

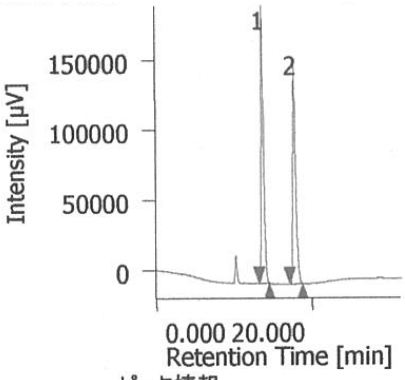
(7) Lam, L. K. P.; Hui, A. H. F.; Jones, J. B. *J. Org. Chem*. **1986**, *51*, 2047.
(8) Park, S. E.; Nam, E. H.; Jang, H. B.; Oh, J. S.; Some, S.; Lee, Y. S.; Song, C. E. *Adv. Synth. Catal*. **2010**, *352*, 2211.

Methyl 3-methoxyglutarate (2d)



Colorless oil. Yield = 83%. IR (neat): ν 3224, 1736, 1613, 1438 cm^{-1}; ^1H NMR ($CDCl_3$): δ 4.03-3.97 (m, 1H), 3.64 (s, 3H), 3.34 (s, 3H), 2.63-2.50 (m, 4H); ^{13}C NMR ($CDCl_3$): δ 176.4, 171.3, 74.0, 57.5, 51.8, 38.7, 38.6; HRMS (ESI- Orbitrap) calcd. for $C_7H_{11}O_5$ *m/z* 175.0601 $[M-H]^-$, found 175.0611. $[\alpha]_D^{23}$ -2.4 (*c* 0.20, $CHCl_3$, 83% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 13.6 min (major), t_R = 17.7 min (minor).

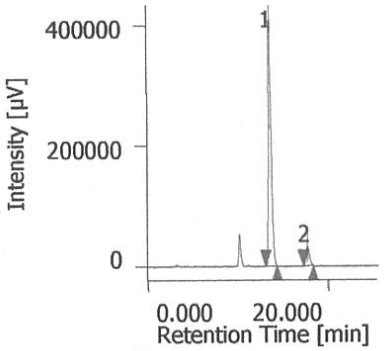
Racemic Sample



ピーク情報

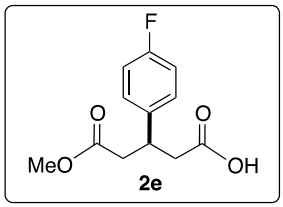
ピーク名	tR [min]	面積%
Unknown	13.633	50.099
Unknown	17.667	49.901

Chiral Sample



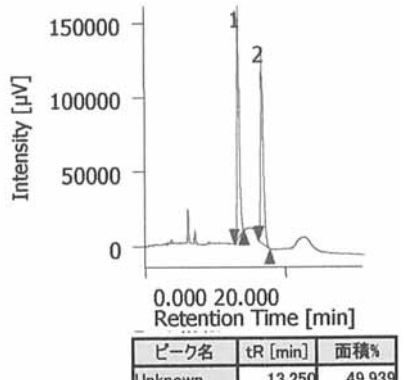
ピーク情報

ピーク名	tR [min]	面積%
Unknown	13.508	91.468
Unknown	17.617	8.532



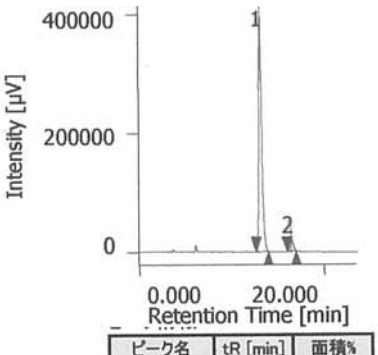
Methyl (*S*)-3-(4-fluorophenyl)glutarate (2e) is reported.[7]
Yield = 92%; ee = 88%. $[\alpha]_D^{23}$ -2.0 (*c* 0.49, $CHCl_3$, 88% ee,), (lit.[4] $[\alpha]_D^{20}$ -4.3 (*c* 1.0, $CHCl_3$)); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 9/1, flow rate = 1.0 mL/min) t_R = 13.3 min (major), t_R = 16.6 min (minor)

Racemic Sample



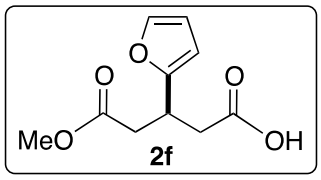
ピーク名	tR [min]	面積%
Unknown	13.250	49.939
Unknown	16.592	50.061

Chiral sample



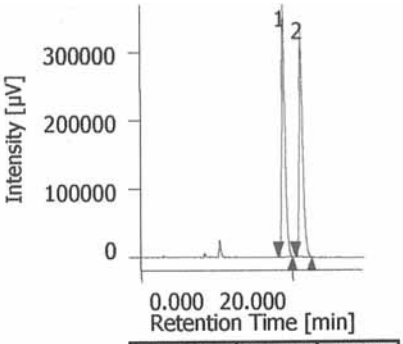
ピーク名	tR [min]	面積%
Unknown	13.083	93.907
Unknown	16.383	6.093

Methyl 3-(furan-2-yl)glutarate (2f)



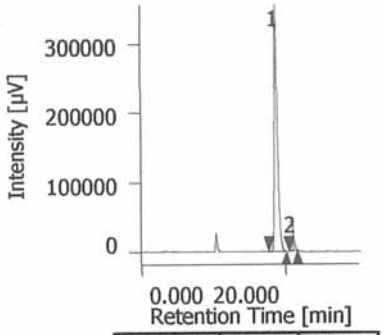
Colorless oil. Yield = 85%. IR (neat): ν 3122, 1737, 1712, 1437 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.29 (d, J = 1.4 Hz, 1H), 6.25 (dd, *J* = 1.8 Hz, 3.2 Hz, 1H), 6.07 (d, *J* = 3.2 Hz, 1H), 3.76-3.71 (m, 1H), 3.64 (s, 3H), 2.76-2.69 (m, 4H); ^{13}C NMR ($CDCl_3$): δ 177.3, 171.9, 155.0, 141.6, 110.2, 105.5, 51.8, 37.7, 37.5, 31.6; HRMS (ESI-Orbitrap) calcd. for $C_{10}H_{11}O_5$ *m/z* 211.0601 $[M-H]^-$, found 211.0609; $[\alpha]_D^{23}$ +1.7 (*c* 0.73, $CHCl_3$, 89% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 18.7 min (major), t_R = 21.0 min (minor).

Racemic Sample



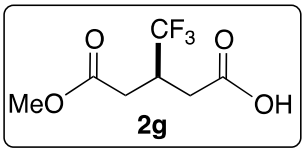
ピーク名	tR [min]	面積%
Unknown	18.733	49.996
Unknown	21.033	50.004

Chiral Sample



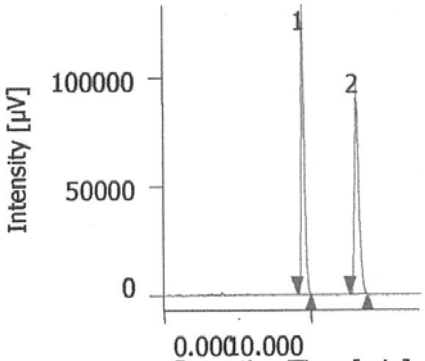
ピーク名	tR [min]	面積%
Unknown	18.683	94.665
Unknown	20.975	5.335

Methyl 3-trifluoromethylglutarate (2g)



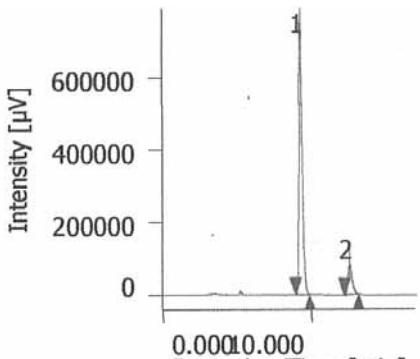
Colorless oil. Yield = 86%. IR (neat): ν 3203, 1743, 1720, 1440 cm^{-1}; ^1H NMR ($CDCl_3$): δ 3.72 (s, 3H), 3.33-3.25 (m, 1H), 2.78-2.69 (m, 2H), 2.63-2.51 (m, 2H); ^{13}C NMR ($CDCl_3$): δ 176.1, 170.7, 126.7 (d, *J* = 279.4 Hz), 52.2, 36.7 (q, *J* = 28.8 Hz), 32.6 (m); HRMS (ESI-Orbitrap) calcd. for $C_7H_8O_4F_3$ *m/z* 213.0369 $[M-H]^-$, found 213.0371; $[\alpha]_D^{23}$ -2.0 (*c* 0.27, $CHCl_3$, 75% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 9.3 min (major), t_R = 12.6 min (minor).

Racemic Sample



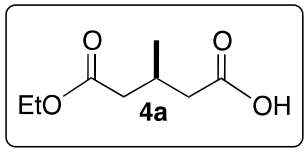
ピーク名	tR [min]	面積%
Unknown	9.433	49.958
Unknown	13.075	50.042

Chiral Sample



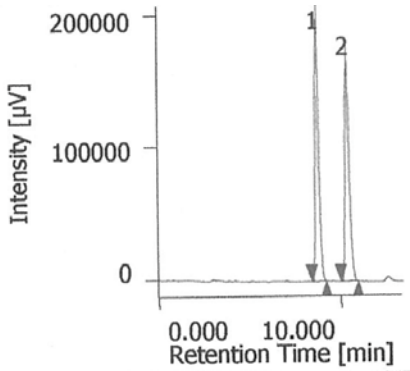
ピーク名	tR [min]	面積%
Unknown	9.258	87.357
Unknown	12.558	12.643

Ethyl 3-methylglutarate (4a)



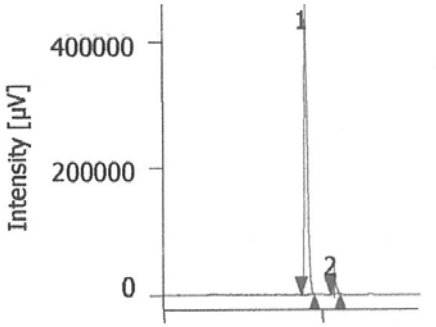
Colorless oil. Yield = 88%. IR (neat): ν 3239, 1733, 1712, 1460 cm^{-1}; ^1H NMR ($CDCl_3$): δ 4.14 (q, J = 7.2 Hz, 2H), 2.52-2.23 (m, 4H), 1.26 (t, J = 7.2 Hz, 3H), 1.06 (d, J = 6.8 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 178.1, 172.3, 60.4, 40.8, 40.5, 27.2, 19.8, 14.2; HRMS (ESI-Orbitrap) calcd. for $C_8H_{13}O_4$ m/z 173.0808 $[M-H]^-$, found 173.0822; $[\alpha]_D^{23}$ -1.2 (c 0.49, $CHCl_3$, 90% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, n-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 8.9 min (major), t_R = 10.7 min (minor).

Racemic Sample



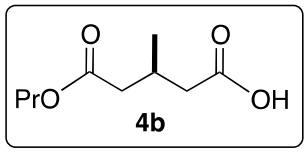
ピーク名	tR [min]	面積%
Unknown	8.683	49.991
Unknown	10.325	50.009

Chiral Sample



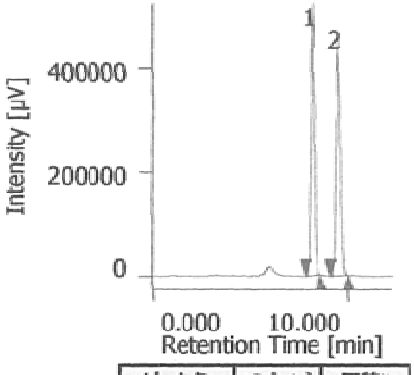
ピーク名	tR [min]	面積%
Unknown	8.925	95.020
Unknown	10.717	4.980

Propyl 3-methylglutarate (4b) is reported.[9]



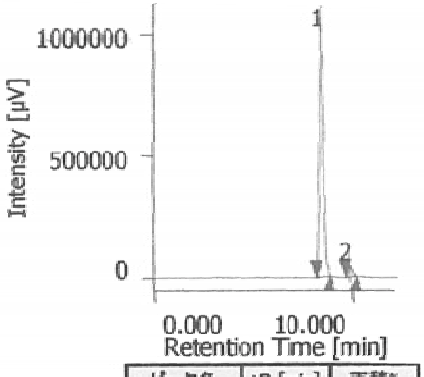
Yield = 90%; ee = 92%. HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, n-hexane / ^{i}PrOH = 9/1, flow rate = 1.0 mL/min) t_R = 8.4 min (major), t_R = 9.8 min (minor).

Racemic Sample



ピーク名	tR [min]	面積%
Unknown	8.150	49.988
Unknown	9.475	50.012

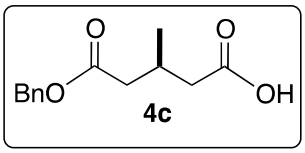
Chiral Sample



ピーク名	tR [min]	面積%
Unknown	8.350	95.770
Unknown	9.800	4.230

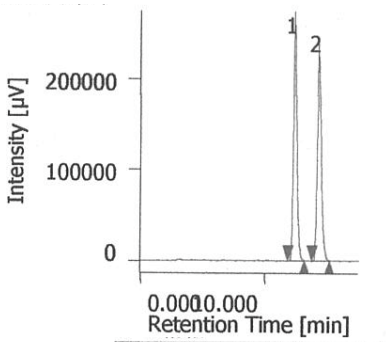
(9) Ito, H.; Inoue, T.; Iguchi, K. *Org. Lett*. **2008**, *10*, 3873.

Benzyl 3-methylglutarate (4c)



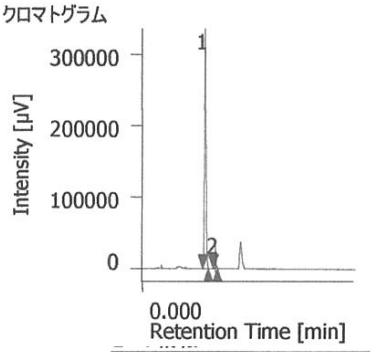
Colorless oil. Yield = 80%. IR (neat): ν 3034, 1734, 1708, 1455 cm^{-1}; ^1H NMR ($CDCl_3$): δ 7.39-7.29 (m, 5H), 5.12 (s, 2H), 2.56-2.41 (m, 2H), 2.38-2.25 (m, 2H), 1.04 (d, J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 178.3, 172.1, 135.8, 128.5, 128.2, 66.3, 40.7, 40.4, 27.2, 19.8; HRMS (ESI-Orbitrap) calcd. for $C_{13}H_{15}O_4$ *m/z* 235.0965. $[M-H]^-$, found 235.0972; $[\alpha]_D^{23}$ +1.8 (*c* 0.40, $CHCl_3$, 91% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 12.6 min (major), t_R = 14.7 min (minor).

Racemic Sample



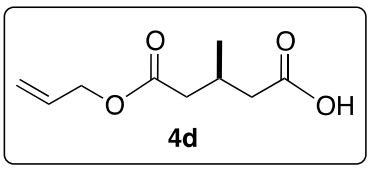
ピーク名	tR [min]	面積%
Unknown	12.342	50.027
Unknown	14.275	49.973

Chiral sample



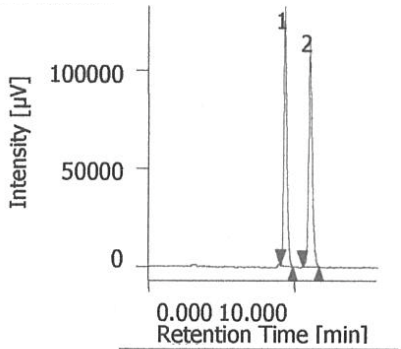
ピーク名	tR [min]	面積%
Unknown	12.583	95.406
Unknown	14.650	4.594

Allyl 3-methylglutarate (4d)



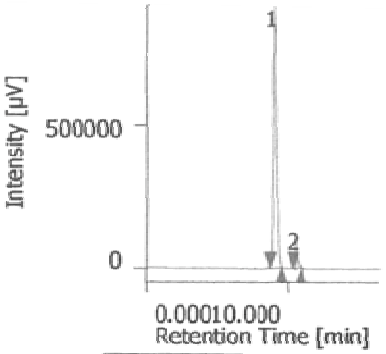
Colorless oil. Yield = 88%. IR (neat): ν 3091, 1759, 1733, 1649, 1484 cm^{-1}; ^1H NMR ($CDCl_3$): δ 11.41 (br, 1H), 5.97-5.87 (m, 1H), 5.35-5.22 (m, 2H), 4.60-4.58 (m, 2H), 2.52-2.44 (m, 3H), 2.35-2.26 (m, 2H), 1.06 (d, J = 6.4 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 178.7, 172.0, 132.0, 118.4, 65.1, 40.6, 27.1, 19.8; HRMS (ESI-Orbitrap) calcd. for $C_9H_{13}O_4$ *m/z* 185.0808 $[M-H]^-$, found 185.0813; $[\alpha]_D^{23}$ +1.1 (*c* 0.85, $CHCl_3$, 90% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 9.0 min (major), t_R = 10.5 min (minor).

Racemic Sample



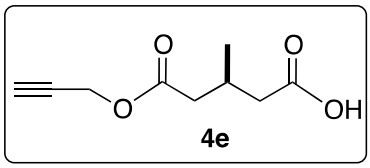
ピーク名	tR [min]	面積%
Unknown	9.300	49.201
Unknown	11.008	50.799

Chiral Sample



ピーク名	tR [min]	面積%
Unknown	8.975	94.795
Unknown	10.483	5.205

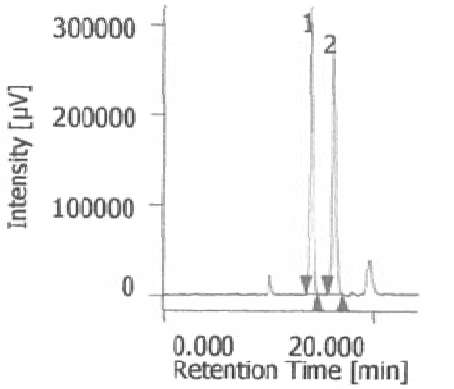
Propargyl 3-methylglutarate (4e)



4e

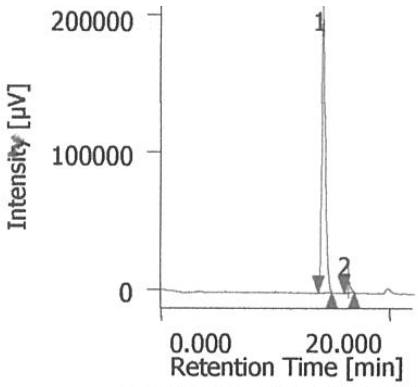
Colorless oil. Yield = 98%. IR (neat): ν 3292, 2967, 1739, 1709 cm^{-1}; ^1H NMR ($CDCl_3$): δ 4.66-4.62 (m, 2H), 2.49-2.41 (m, 4H), 2.32-2.27 (m, 2H), 1.04 (d, J = 3.6 Hz, 3H); ^{13}C NMR ($CDCl_3$): δ 178.4, 171.4, 77.5, 74.9, 51.9, 40.32, 40.28, 27.1, 19.8; HRMS (ESI-Orbitrap) calcd. for $C_9H_{11}O_4$ m/z 183.0652 $[M-H]^-$, found 183.0656; $[\alpha]_D^{23}$ +2.4 (*c* 0.69, $CHCl_3$, 91% ee); HPLC (Daicel CHIRALPAK AD-H, ϕ 0.46 cm × 25 cm, detection 220 nm, *n*-hexane / ^{i}PrOH = 19/1, flow rate = 1.0 mL/min) t_R = 14.1 min (major), t_R = 16.3 min (minor).

Racemic Sample



ピーク名	tR [min]	面積%
Unknown	13.983	50.275
Unknown	16.142	49.725

Chiral Sample



ピーク名	tR [min]	面積%
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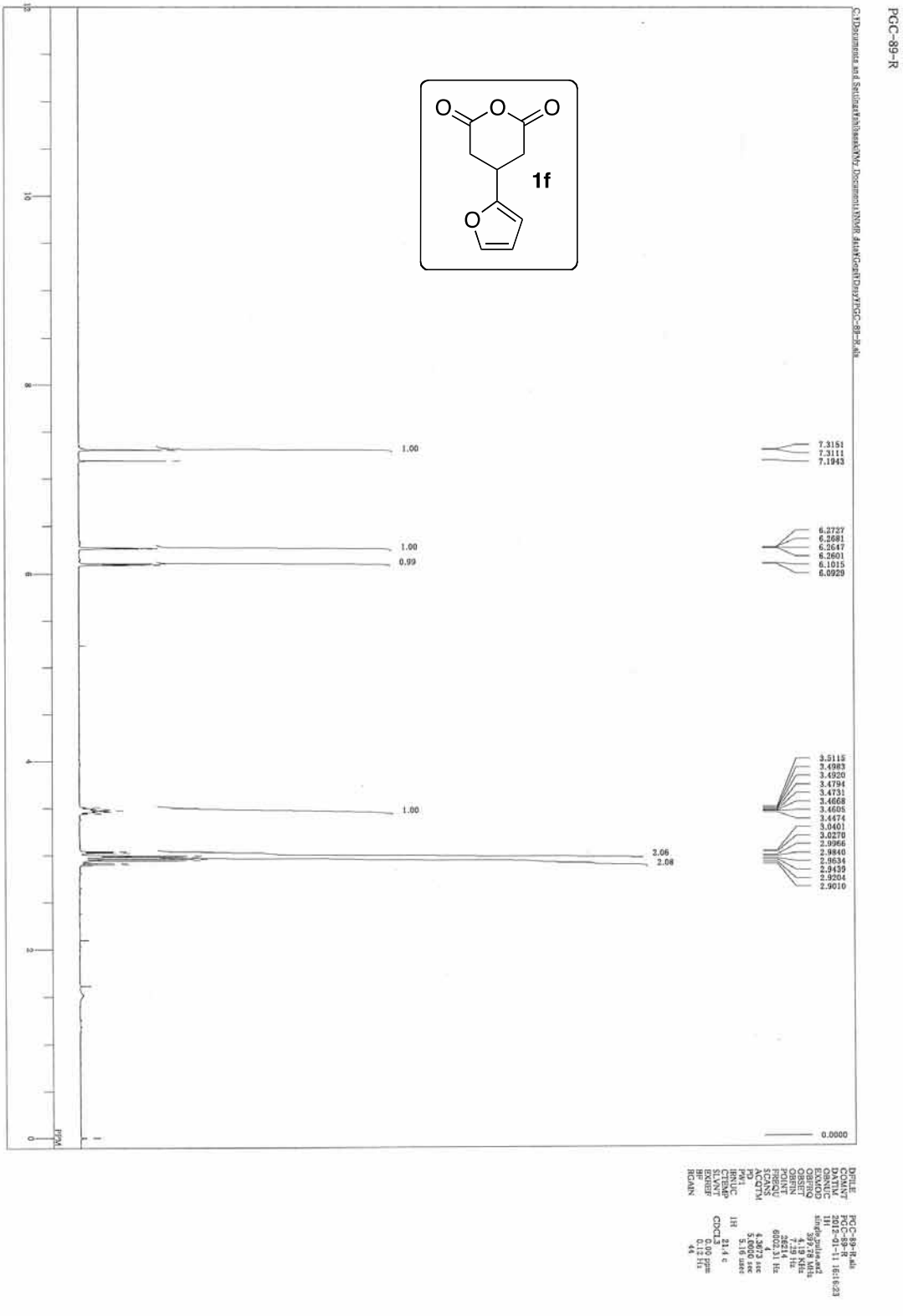
Unknown	16.333	4.426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7.3111
7.1943
6.2727
6.2681
6.2647
6.2601
6.1015
6.0929
3.5115
3.4983
3.4920
3.4794
3.4731
3.4668
3.4605
3.4474
3.0401
3.0270
2.9966
2.9840
2.9634
2.9439
2.9204
2.9010
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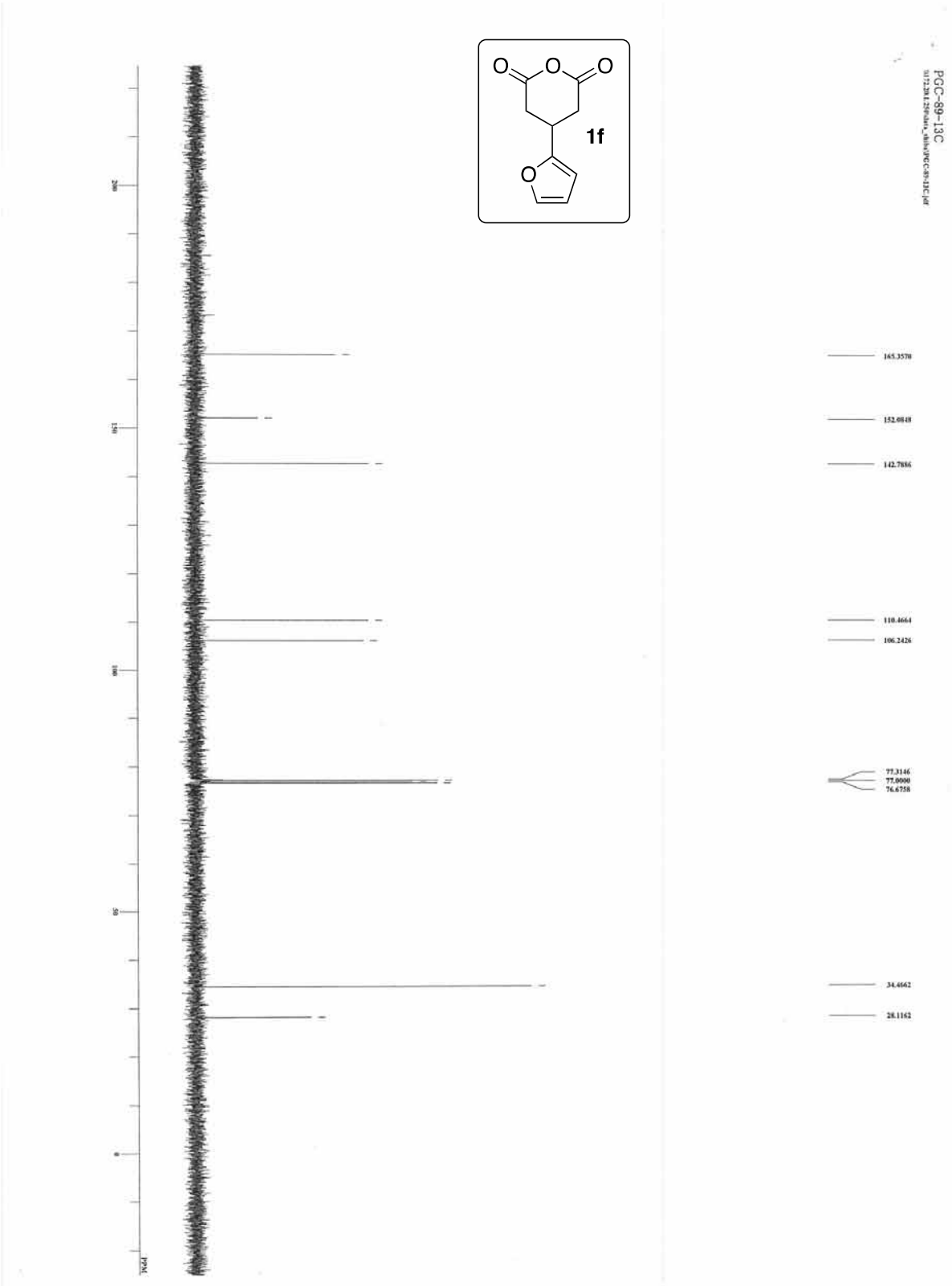
1.00
0.99
1.00
2.06
2.08
0.0000
PPM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O
O
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142.7886
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106.2426
77.3146
77.0000
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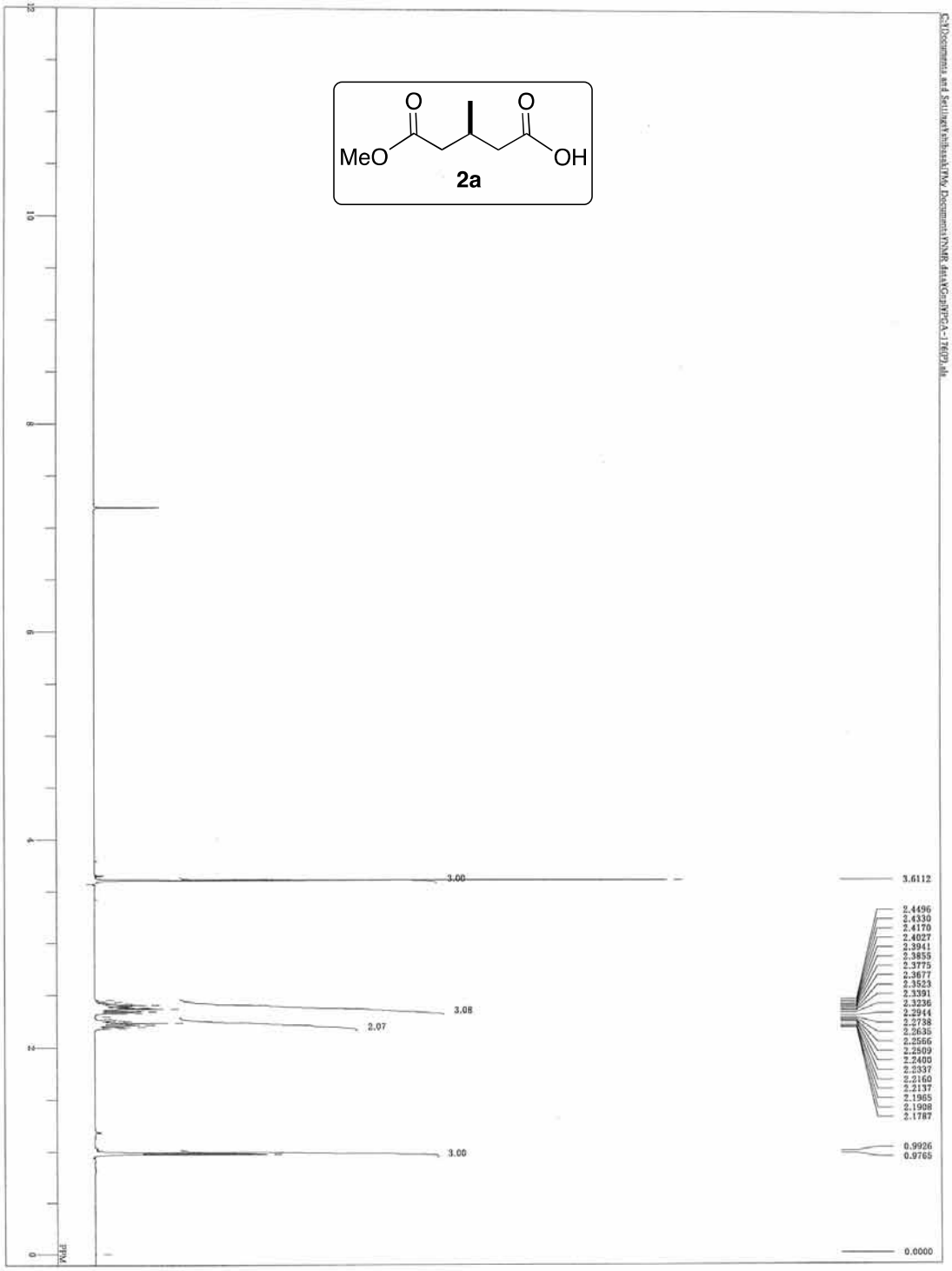
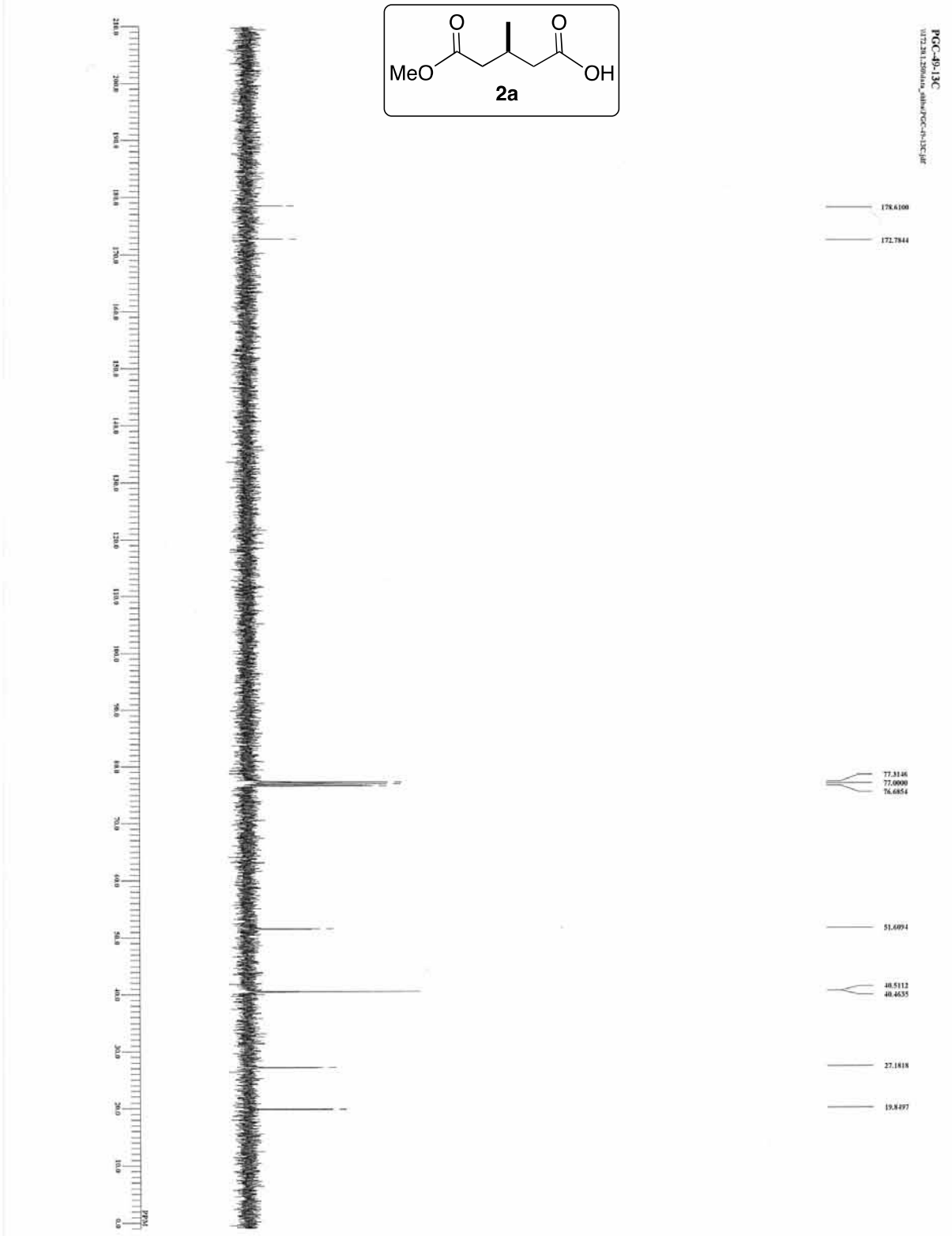
34.4662
28.1162
200
150
100
50
0
PPM

PGA-176(P)

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OH
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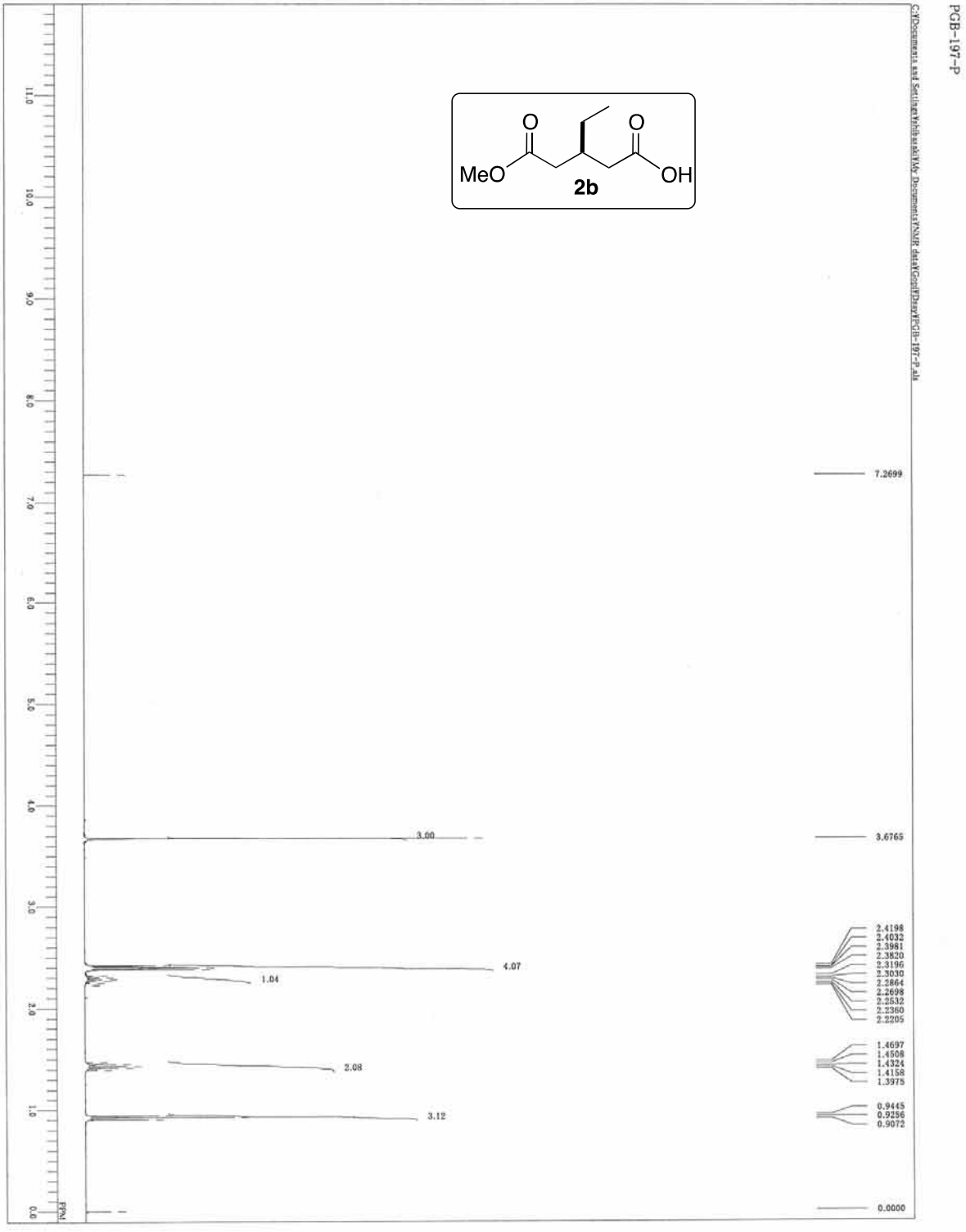
77.0000
76.6854
51.6094
40.5112
40.4635
27.1618
19.8497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O
OH
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2.4198
2.4032
2.3981
2.3820
2.3196
2.3030
2.2864
2.2698
2.2532
2.2360
2.2205
1.4697
1.4508
1.4324
1.4158
1.3975
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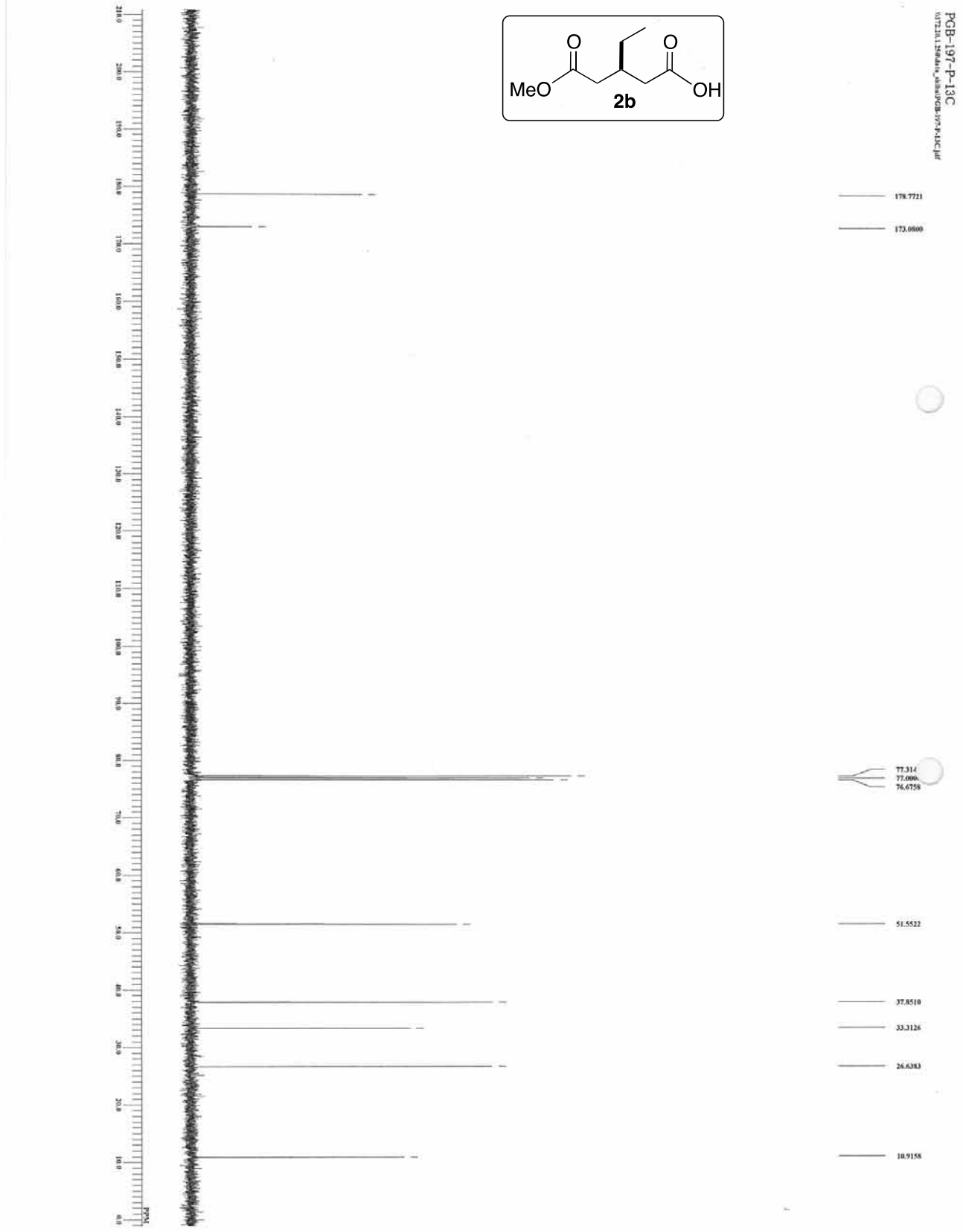
0.9256
0.9072
0.0000
3.00
4.07
1.04
2.08
3.12
PPM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O
OH
2b
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77.314
77.000
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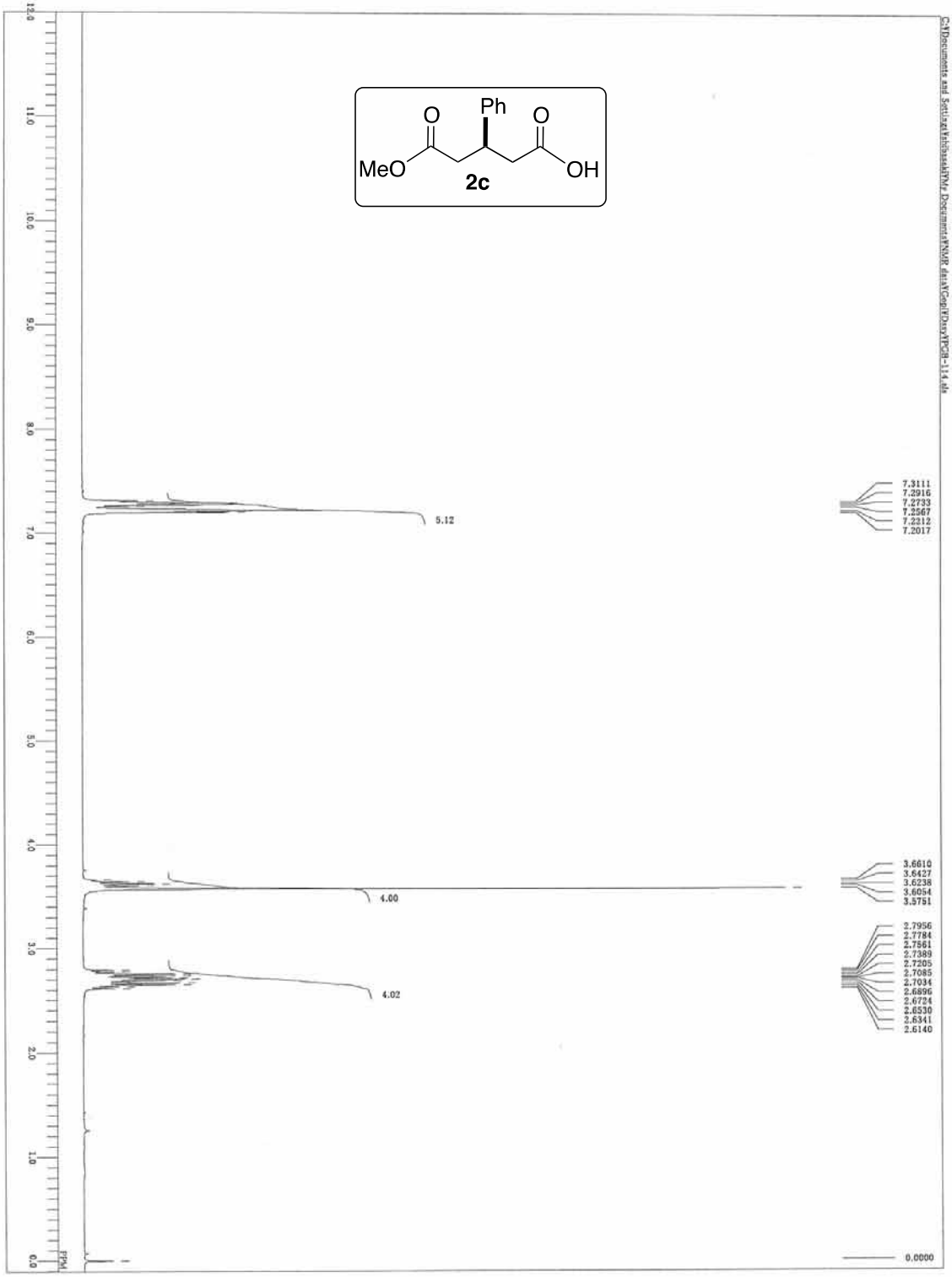
33.3126
26.6383
10.9158
PPM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OH
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4.02
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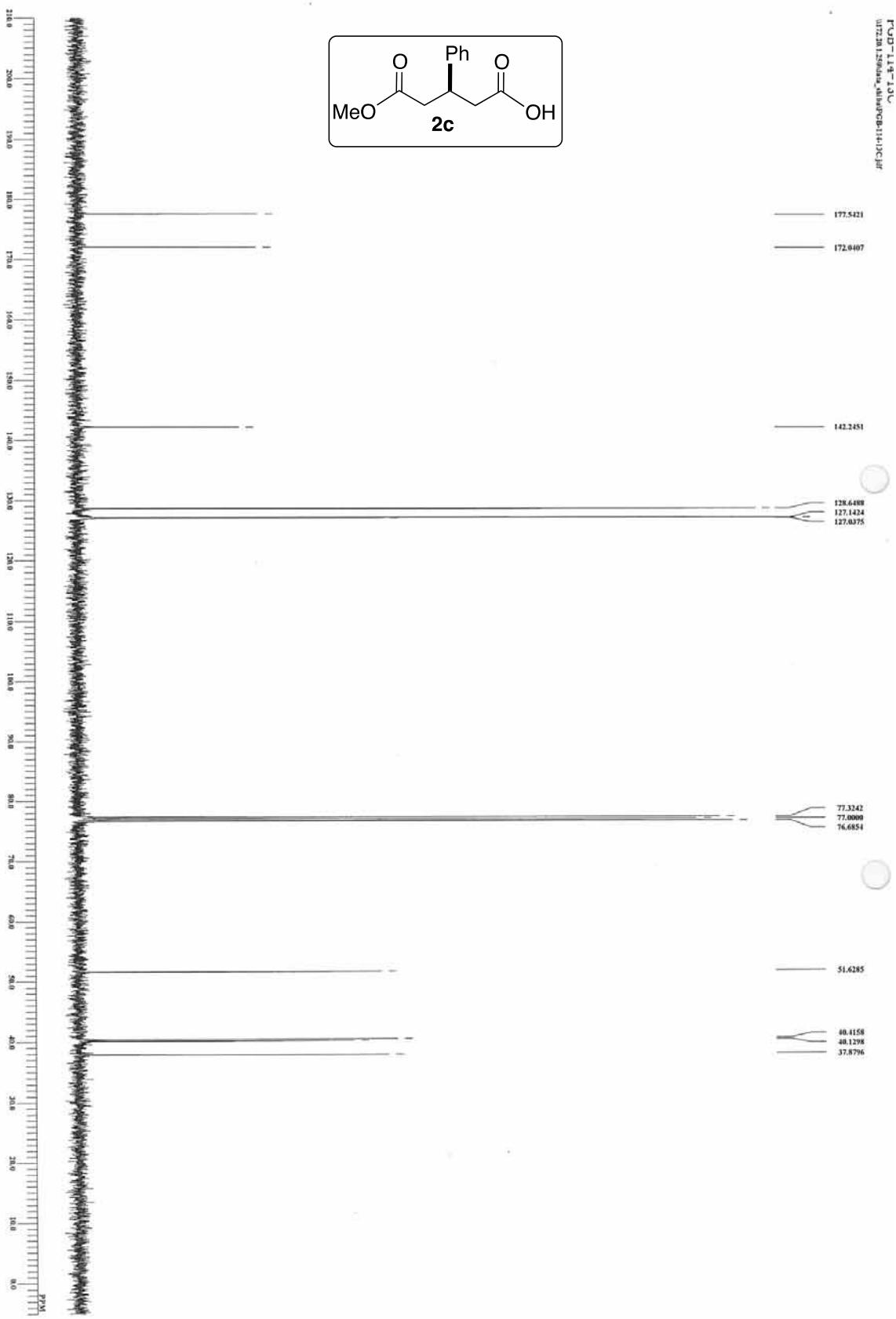
7.2916
7.2733
7.2567
7.2212
7.2017
3.6610
3.6427
3.6238
3.6054
3.5751
2.7956
2.7784
2.7561
2.7389
2.7205
2.7085
2.7034
2.6896
2.6724
2.6530
2.6341
2.6140
0.0000
PPM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MeO
OH
2c
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142.2451
128.6498
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127.0375
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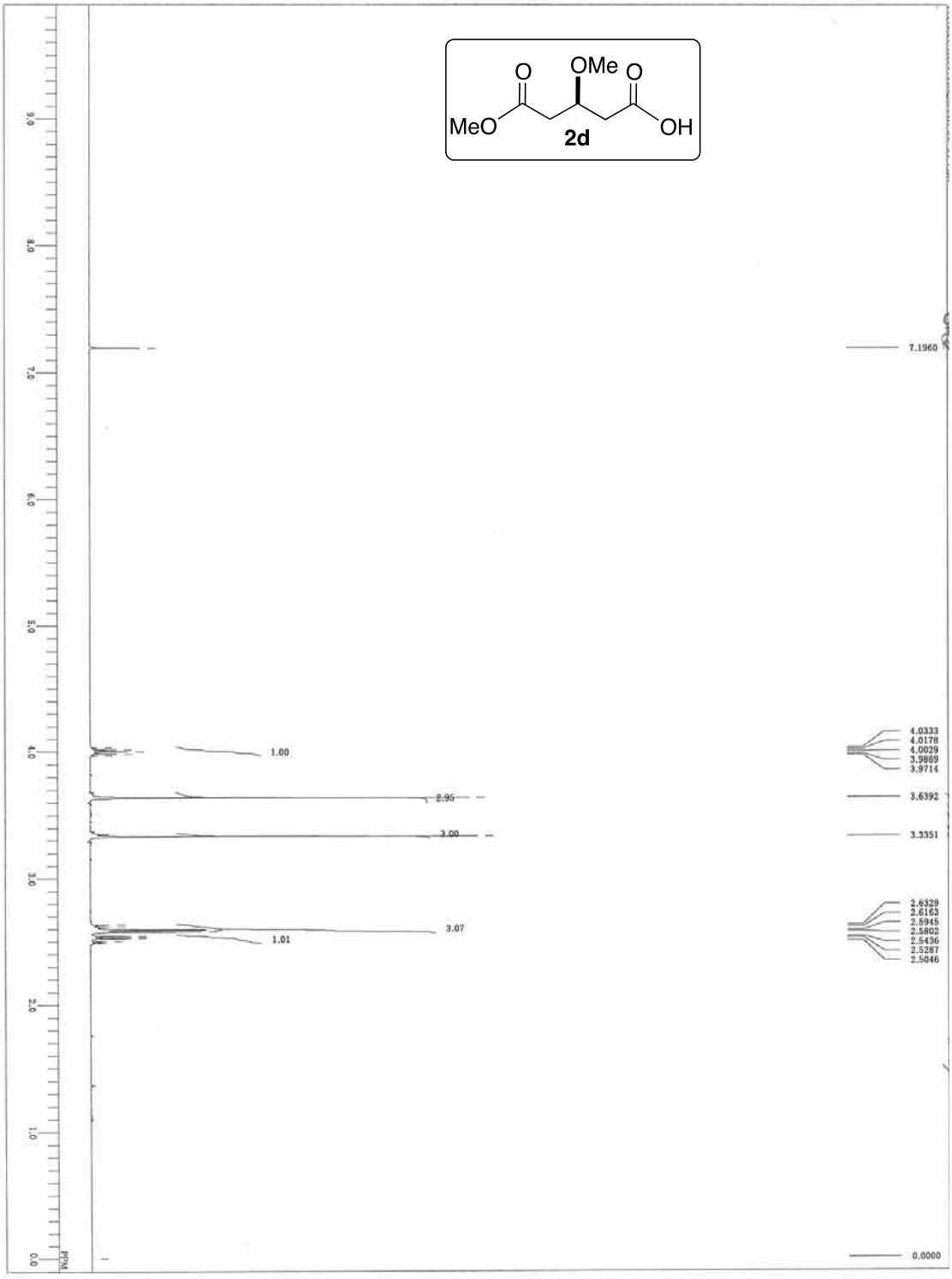
76.6854
51.6285
40.4158
40.1298
37.8796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O
MeO
OH
2d
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4.0029
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3.9714
3.6392
3.3351
2.6329
2.6163
2.5945
2.5802
2.5436
2.5287
2.5046
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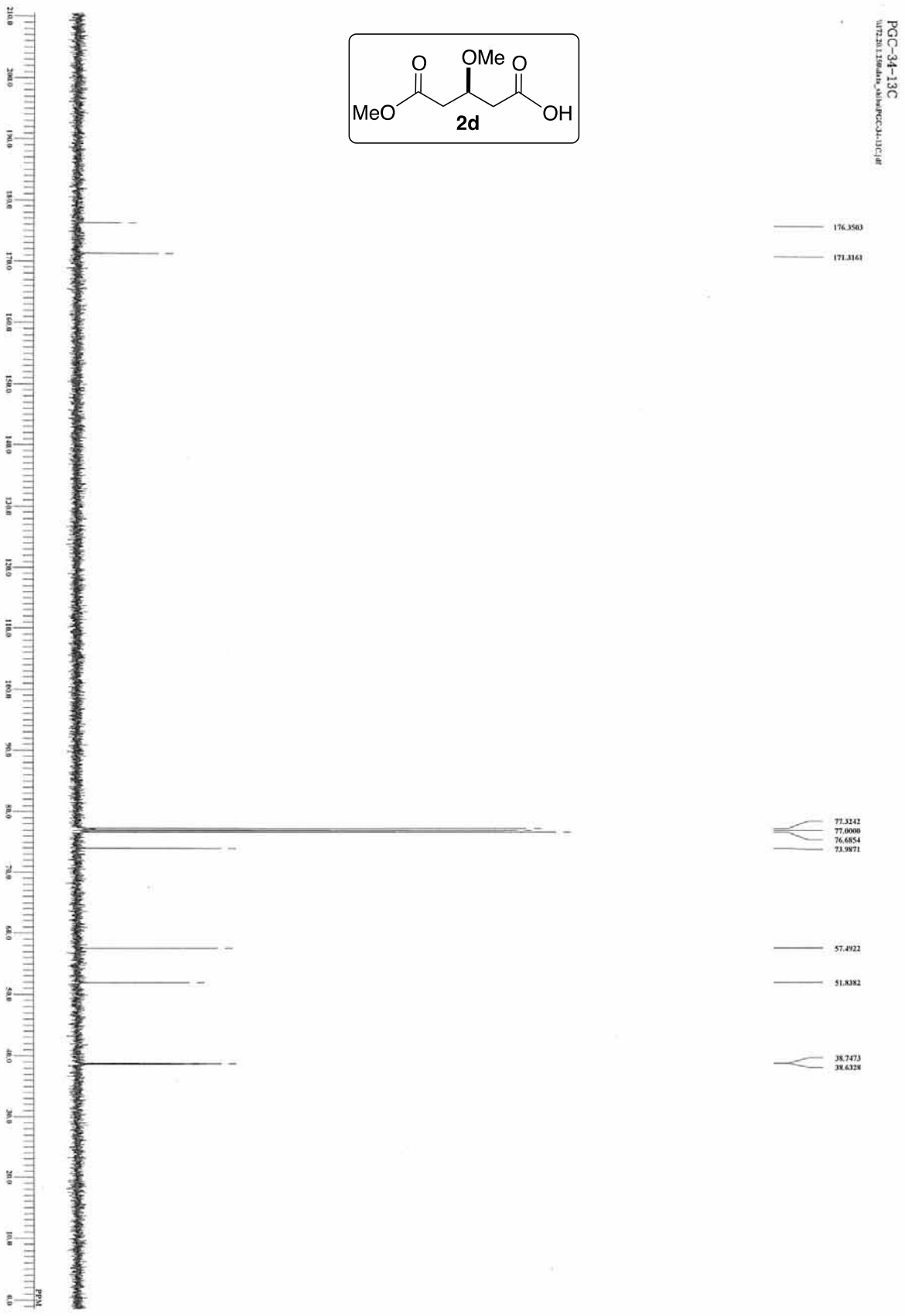
2.95
3.00
3.07
1.01
PPM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OMe
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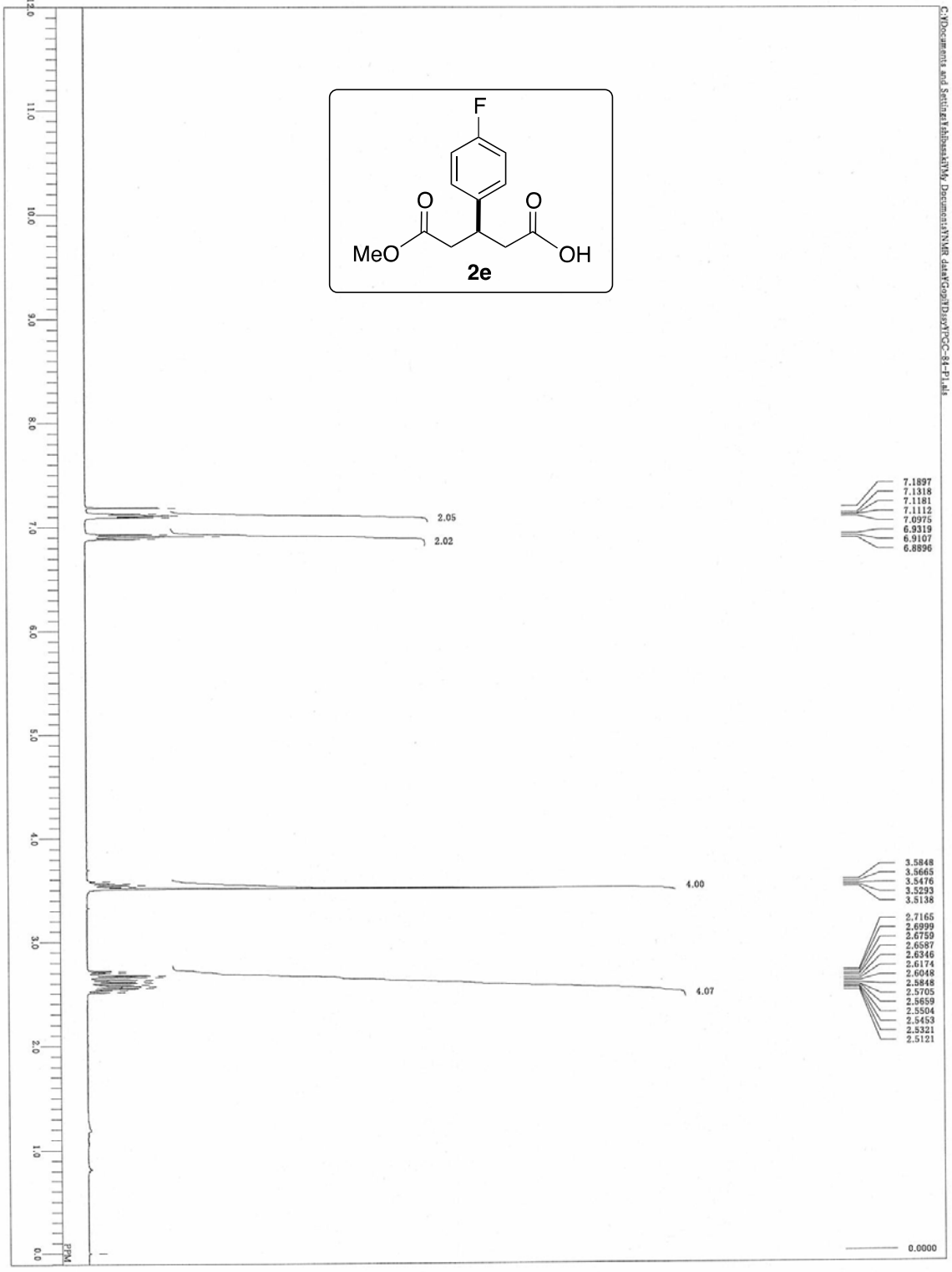
PPM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OH
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6.8896
2.05
2.02
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3.5665
3.5476
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3.5138
4.00
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2.6999
2.6759
2.6587
2.6346
2.6174
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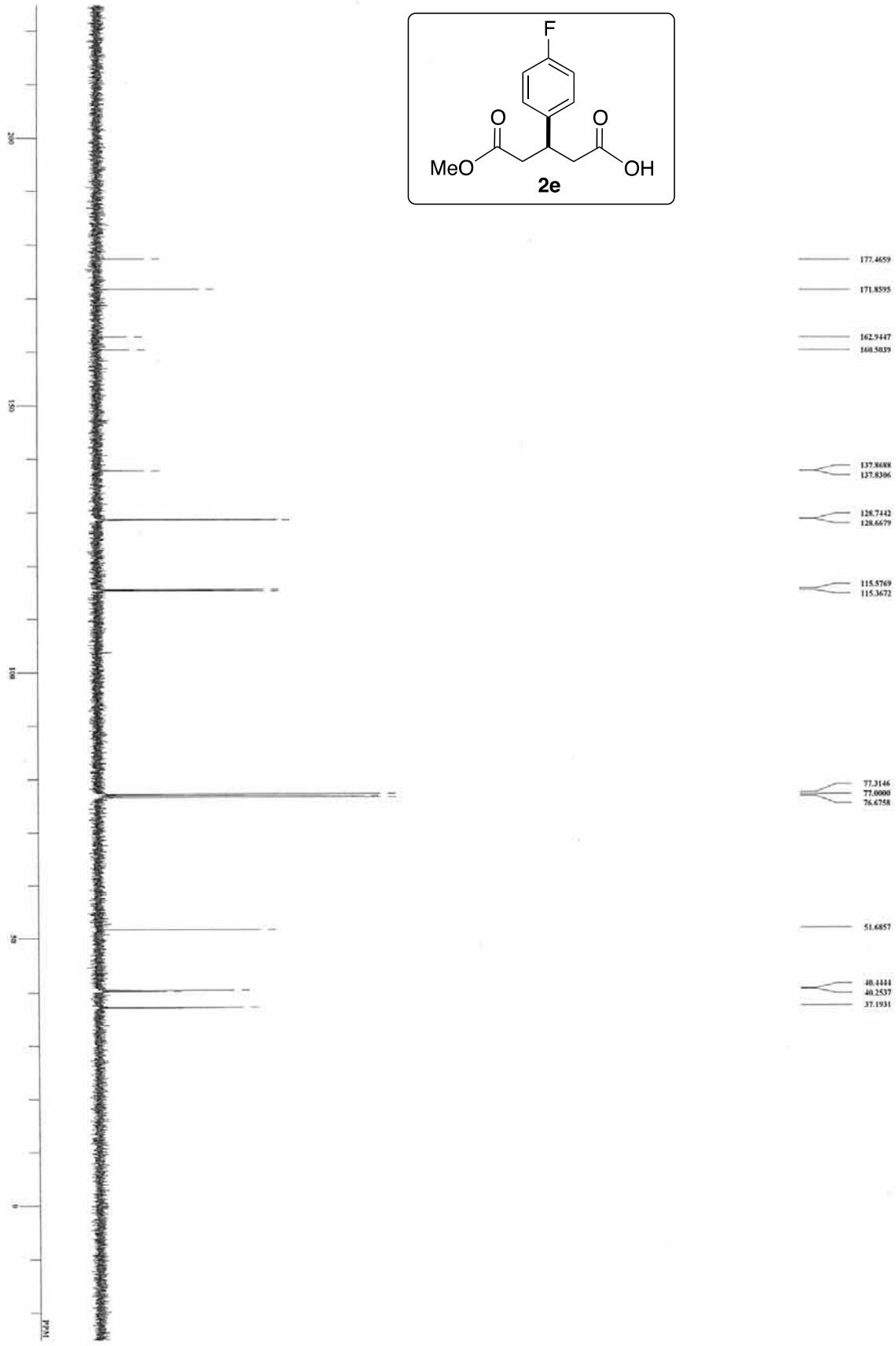
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115.5769
115.3672
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37.1931
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100
50
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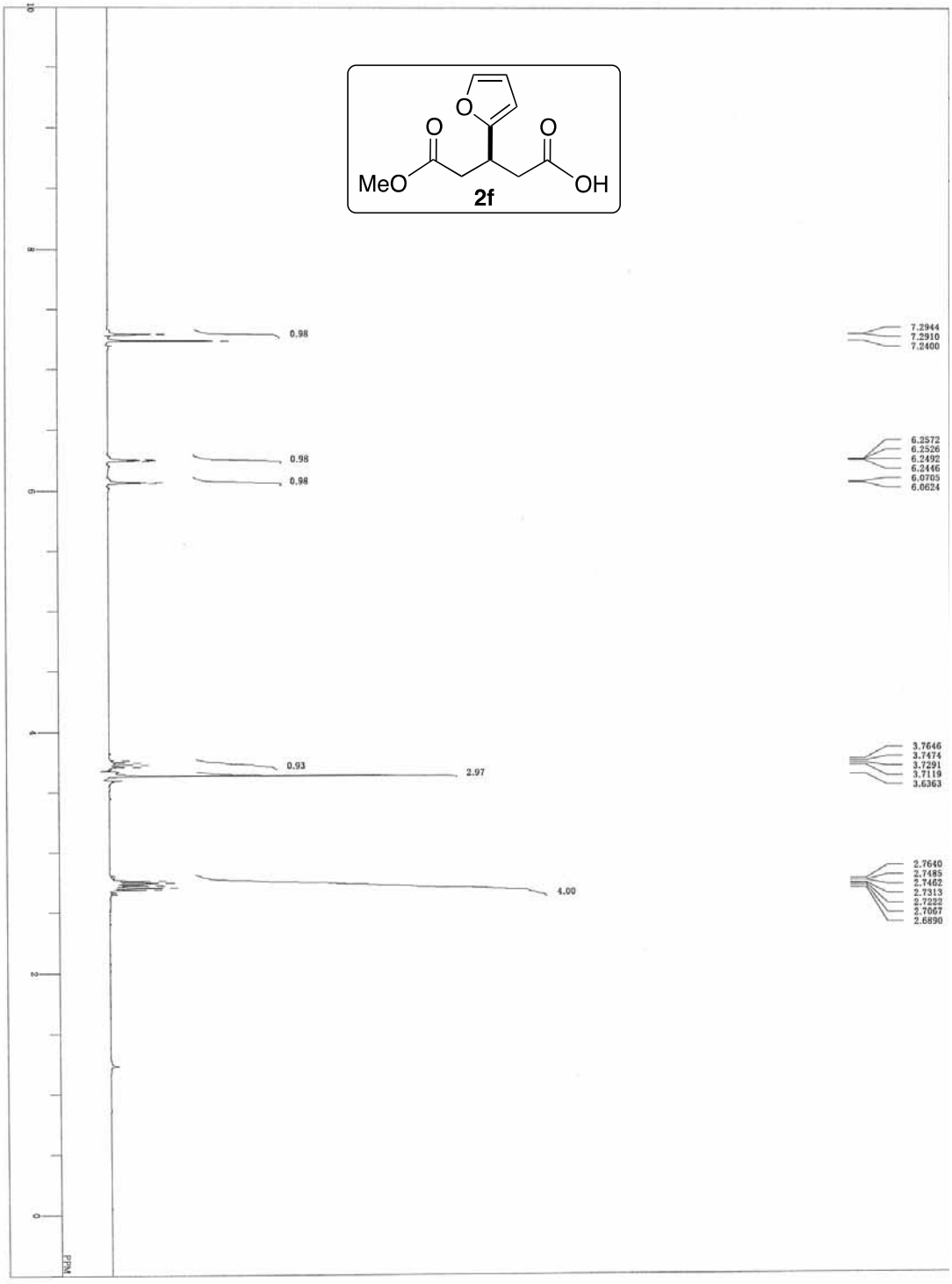
PPM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7.2910
7.2400
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6.2526
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6.2446
6.0705
6.0624
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3.7474
3.7291
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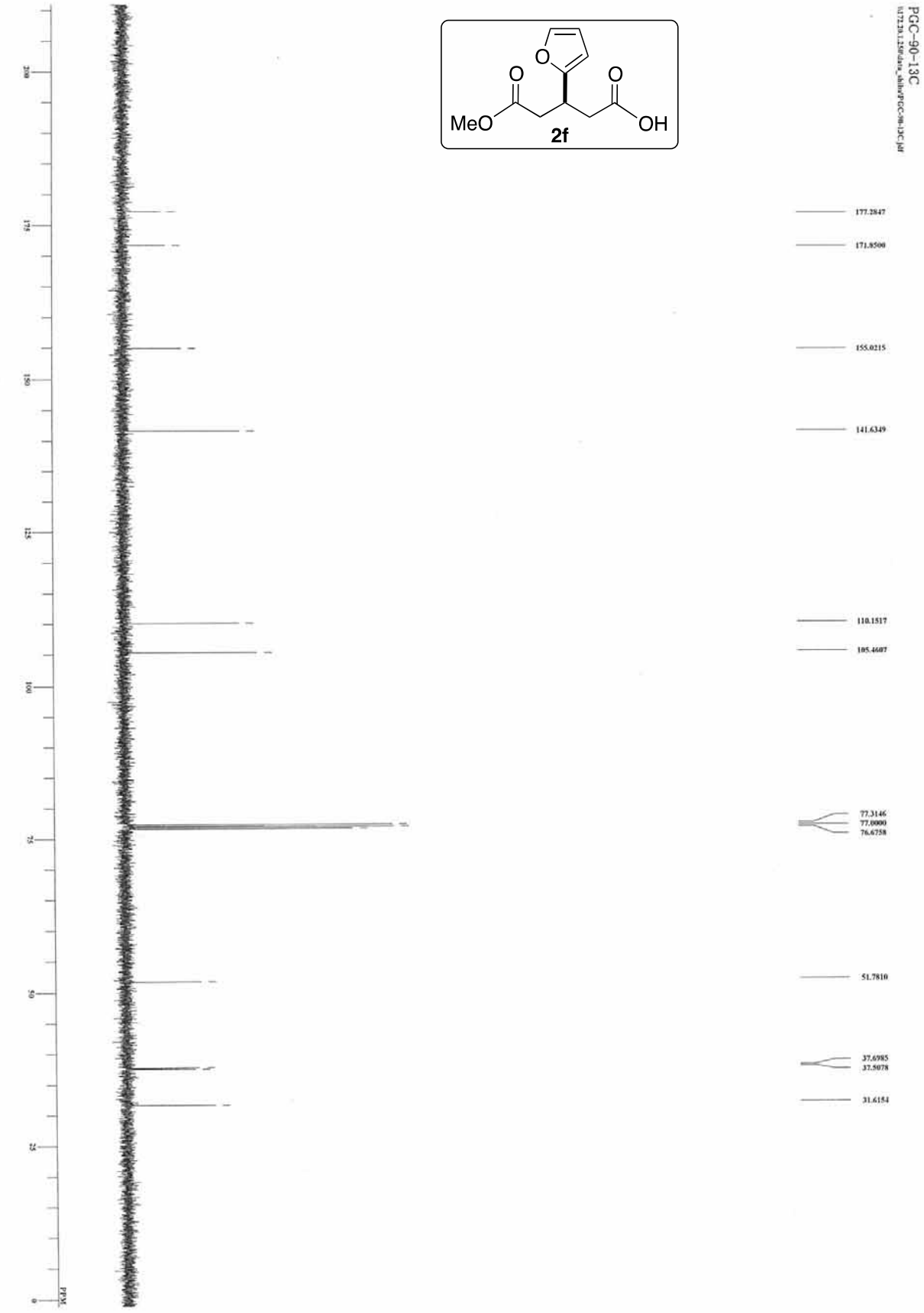
PPM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OH
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155.0215
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77.0000
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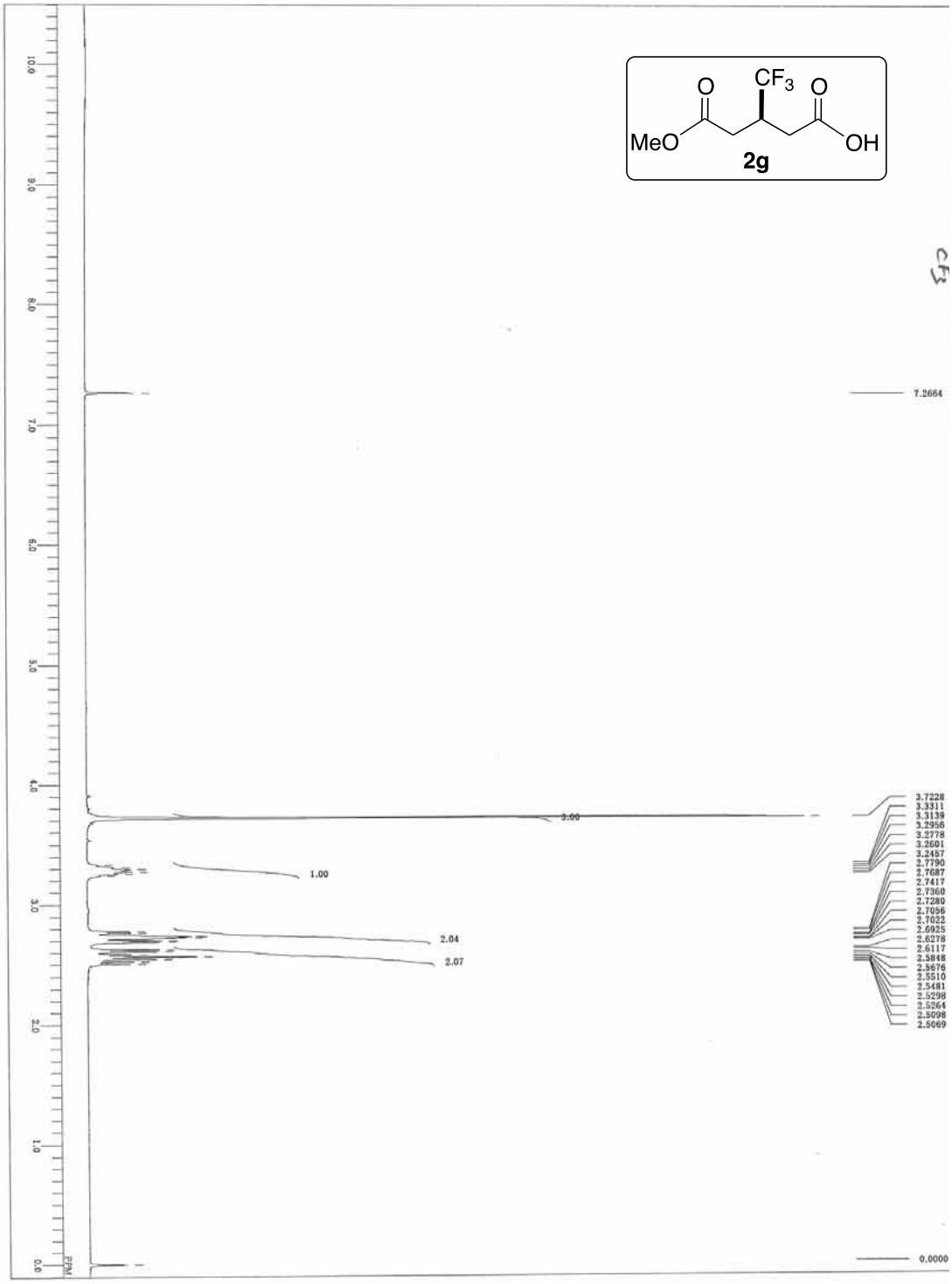
PPM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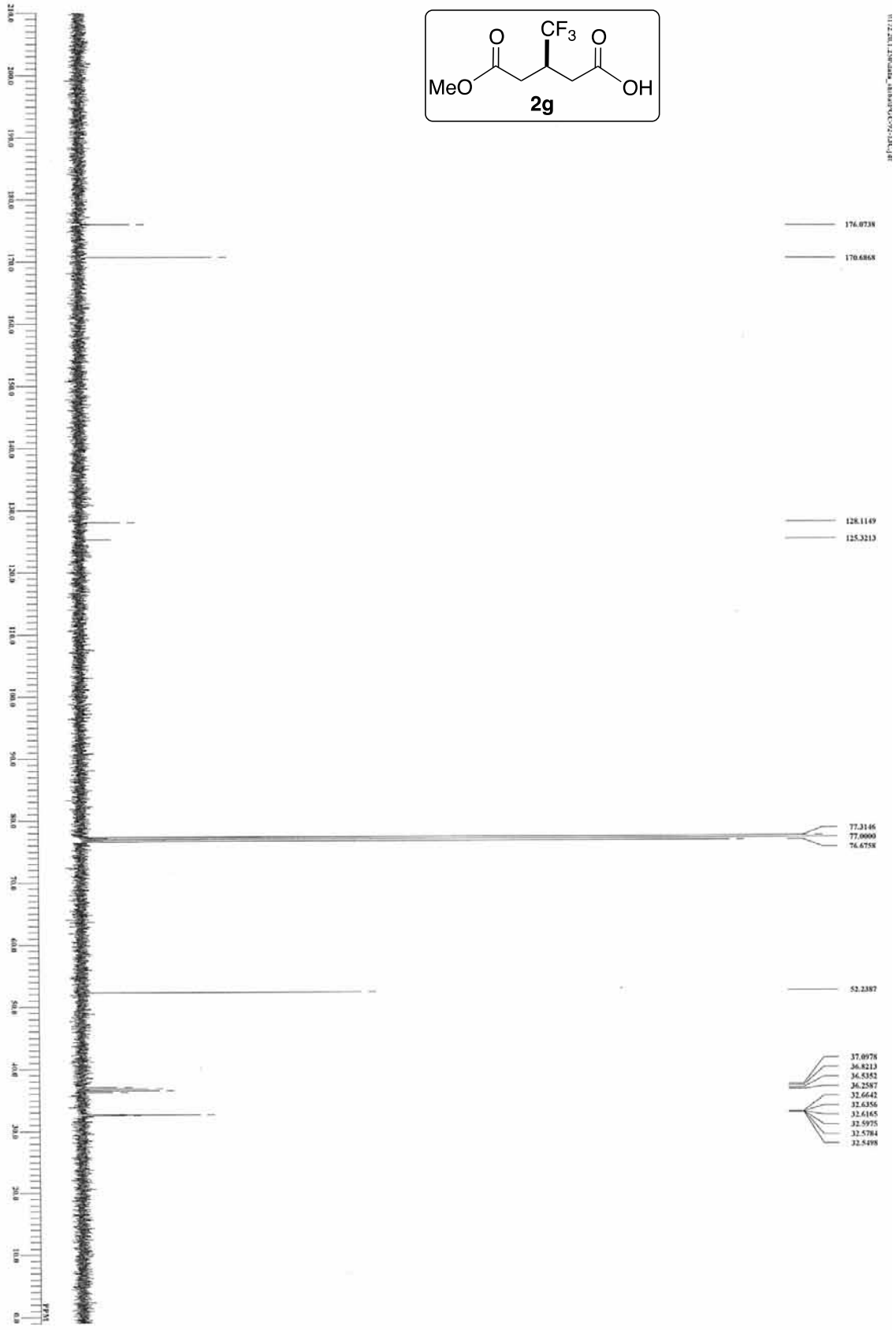
2.07
0.0000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CF3
O
OH
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76.6758
52.2387
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36.2587
32.6642
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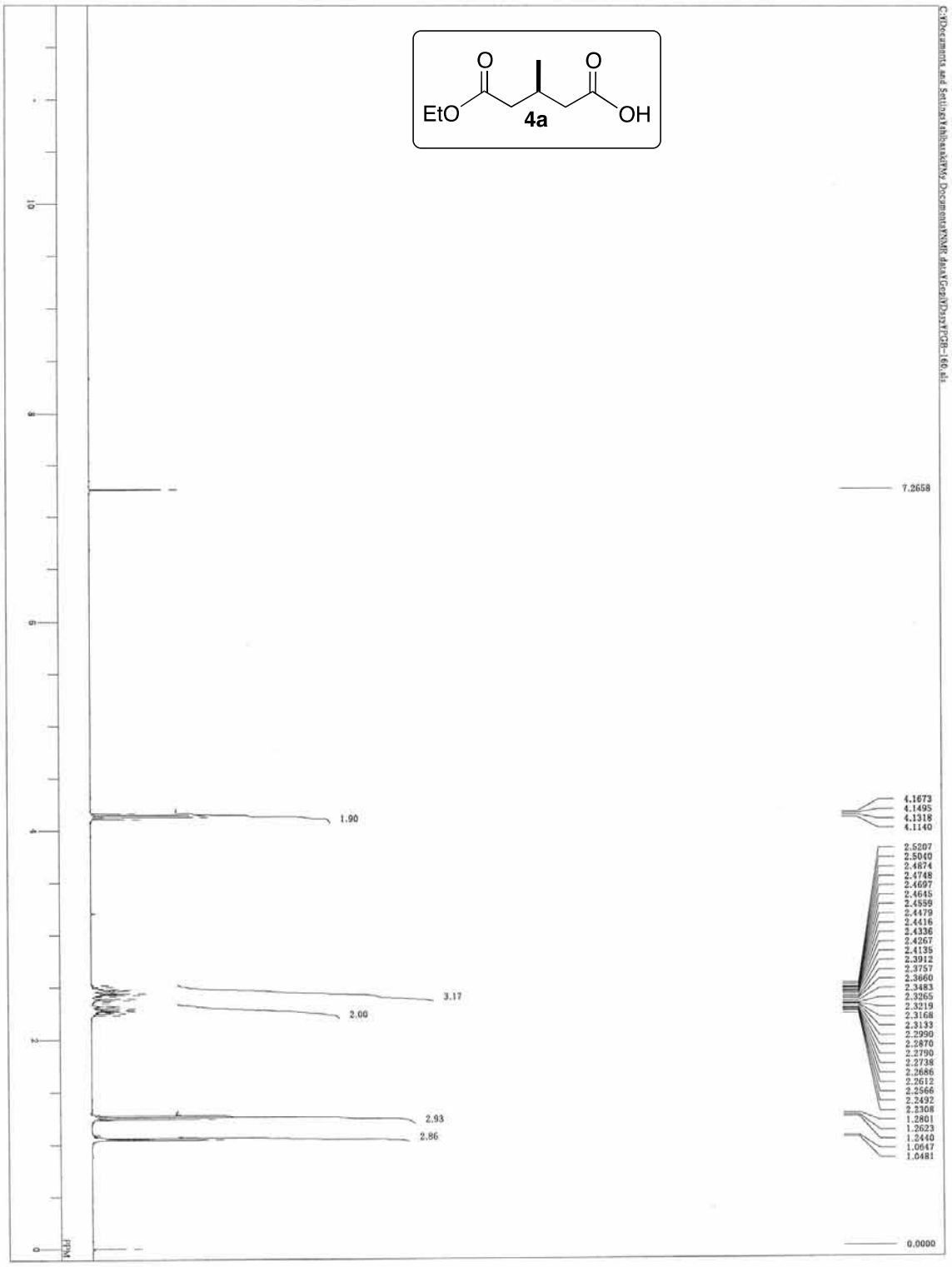
32.6165
32.5975
32.5784
32.5498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OH
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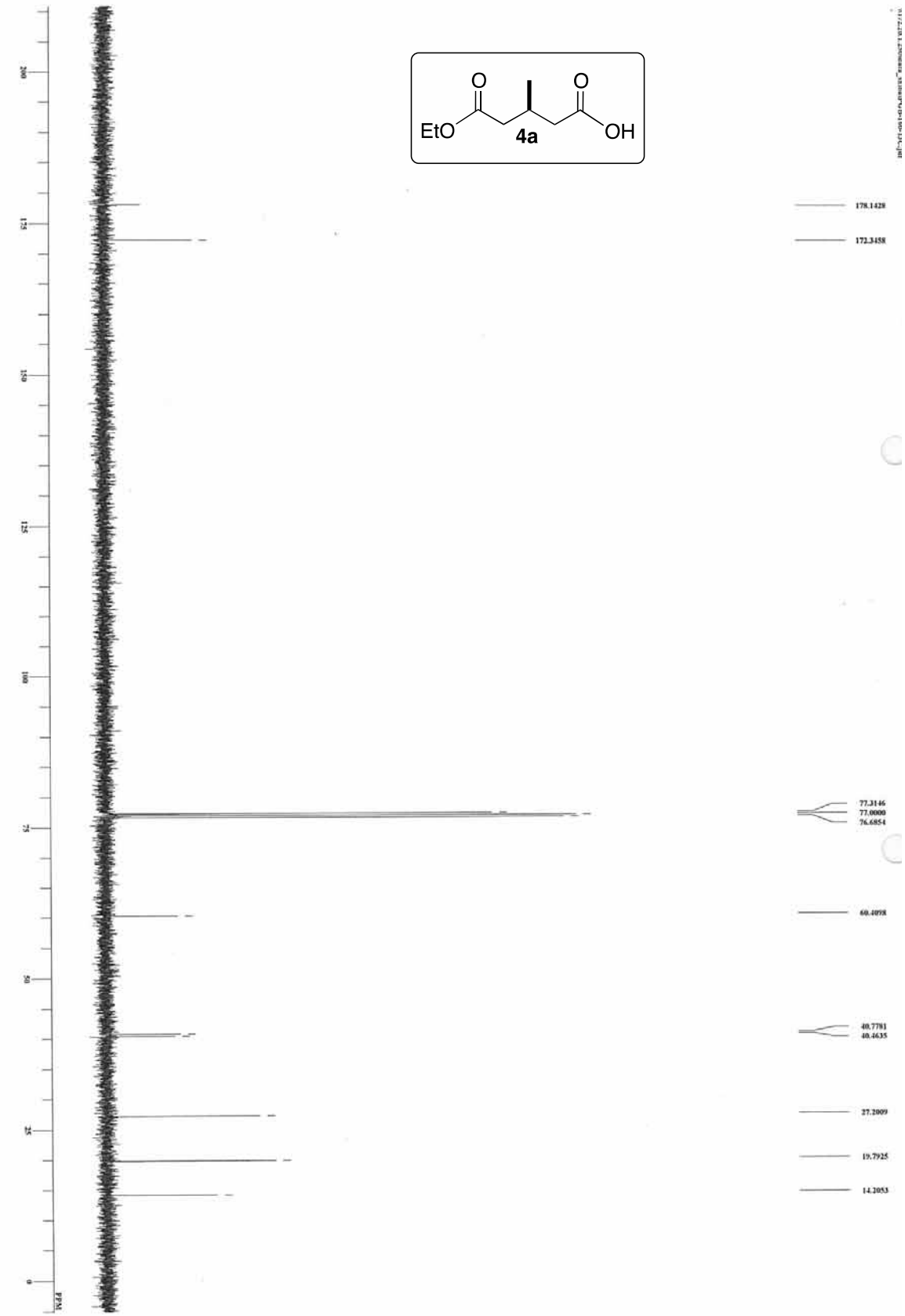
3.17
2.00
2.93
2.86
0.0000
PPM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O
OH
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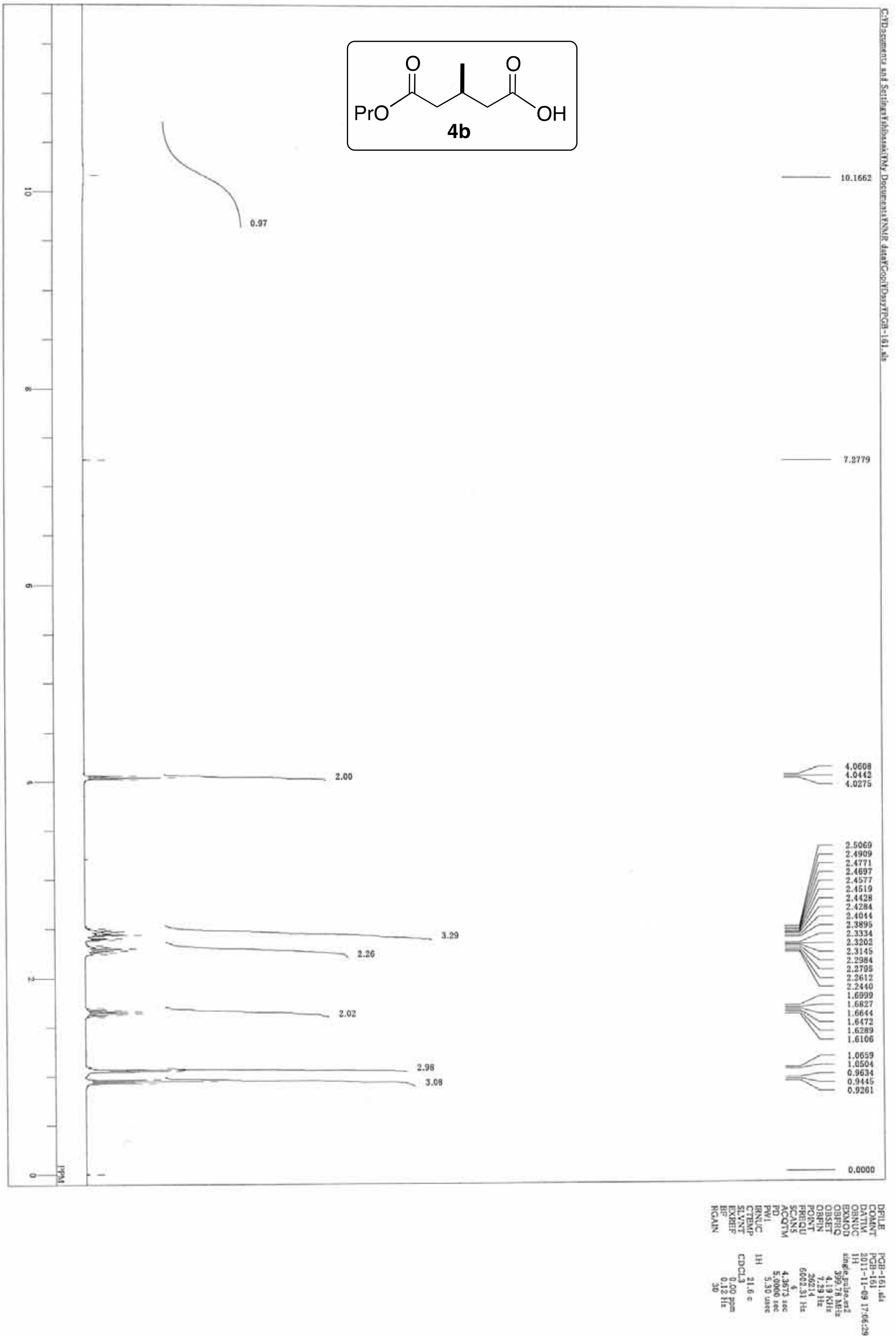
PPM


PrO
OH
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2.4577
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2.4428
2.4284
2.4044
2.3895
2.3334
2.3202
2.3145
2.2984
2.2795
2.2612
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1.6827
1.6644
1.6472
1.6289
1.6106
1.0659
1.0504
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0.97
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3.29
2.26
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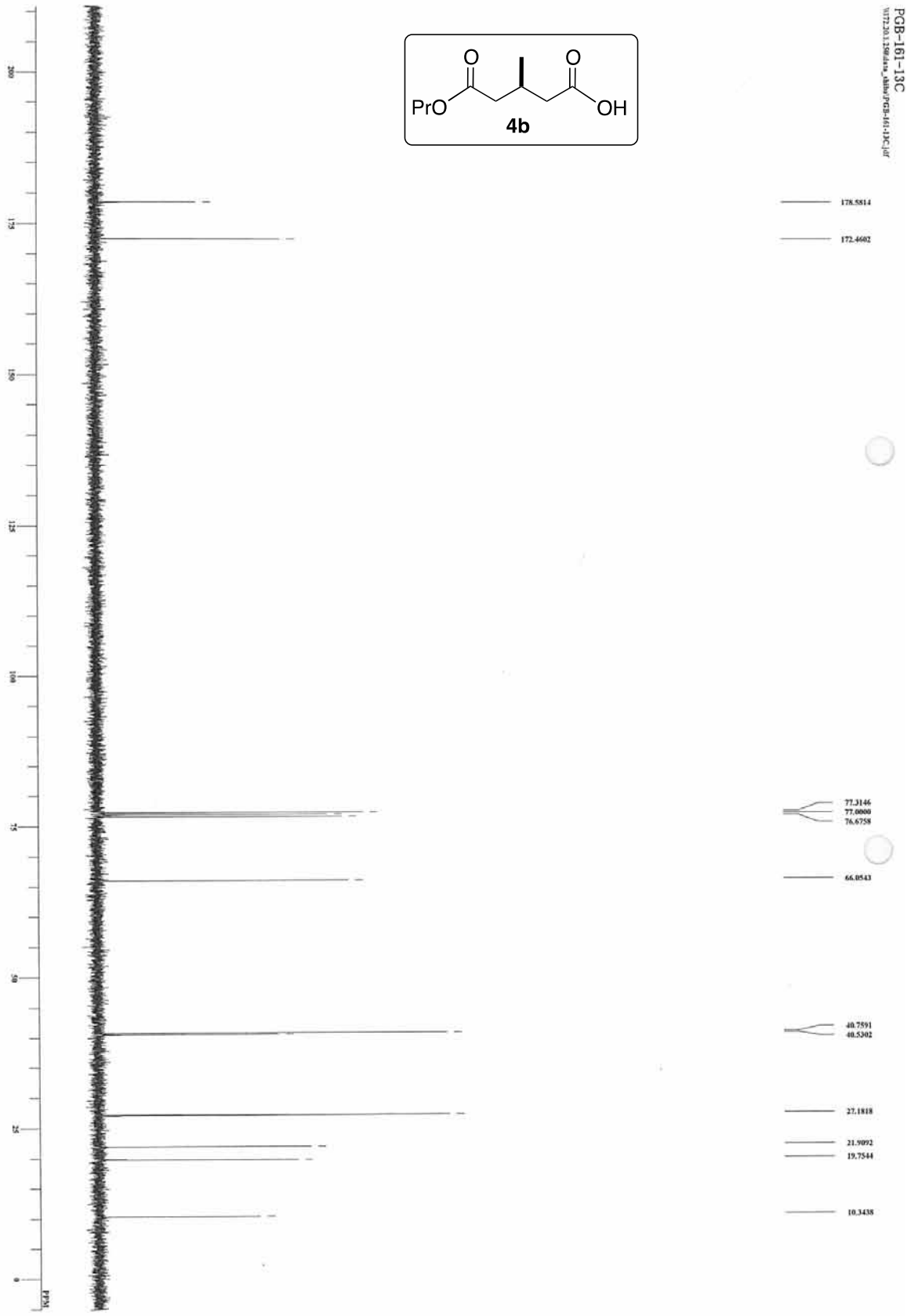
2.98
3.08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O
PrO
OH
4b
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77.0000
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27.1818
21.9092
19.7544
10.3438
200
150
100
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50
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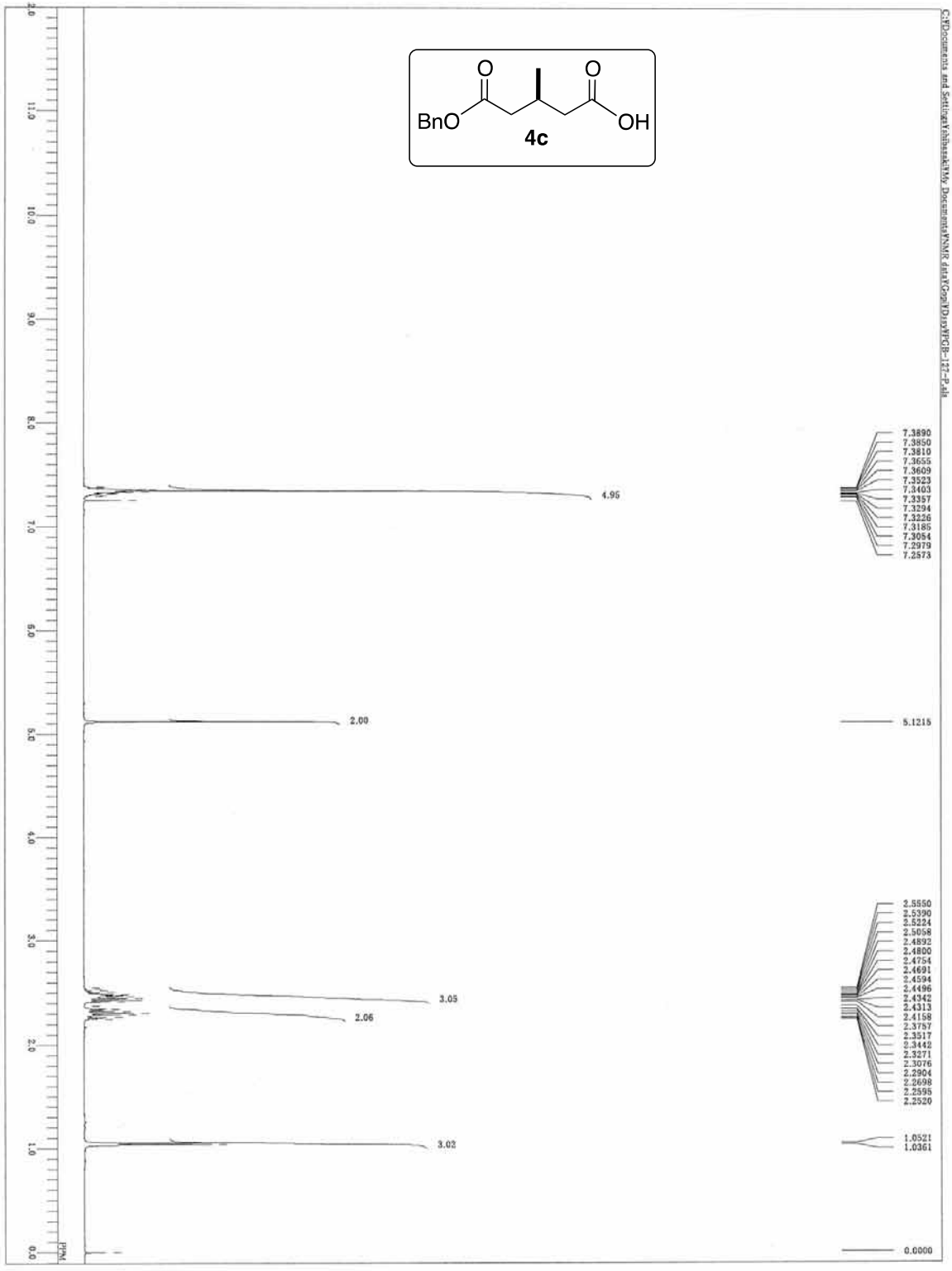
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7.3609
7.3523
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7.3294
7.3226
7.3185
7.3054
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7.2573
5.1215
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2.5390
2.5224
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2.4892
2.4800
2.4754
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2.4496
2.4342
2.4313
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2.3757
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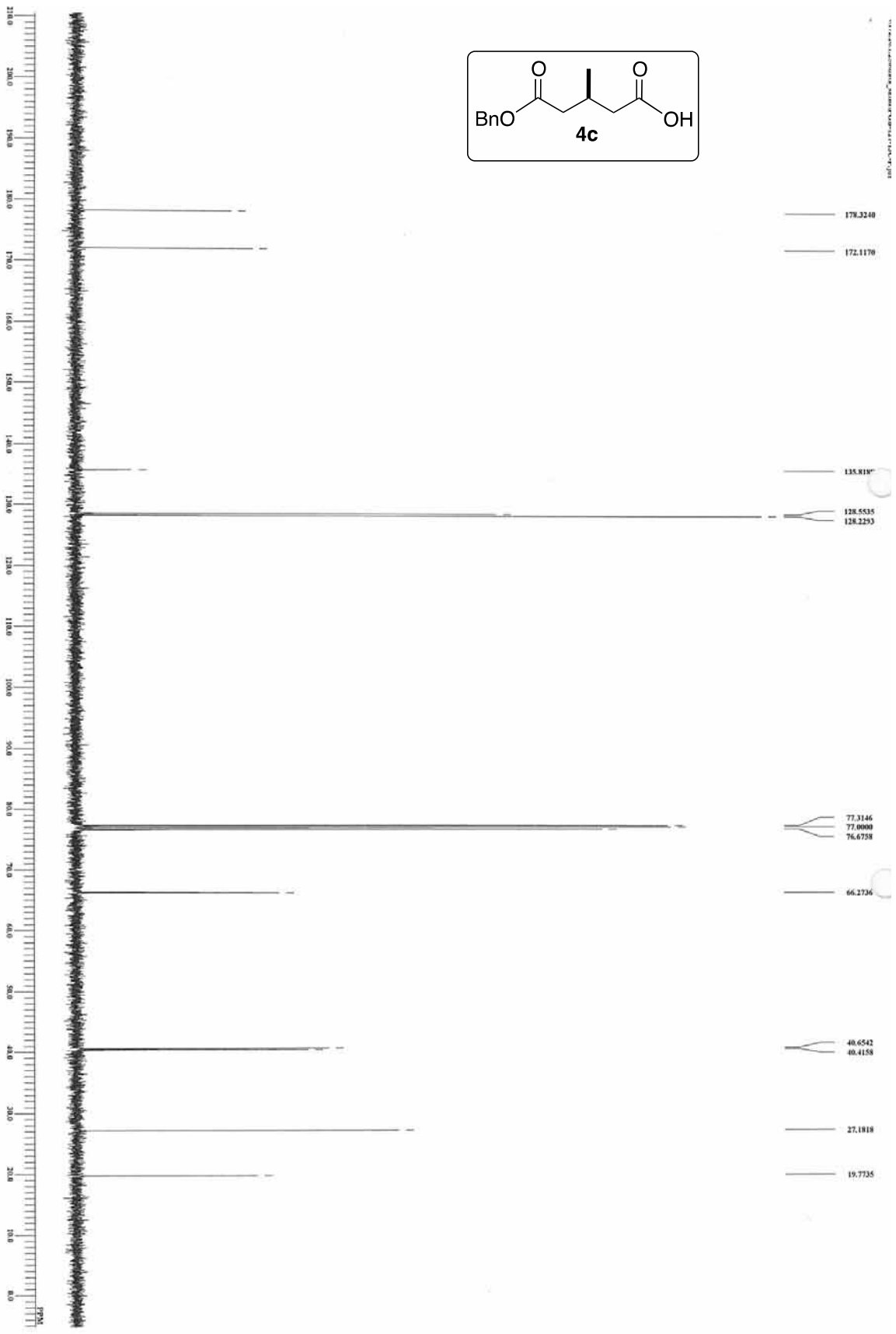
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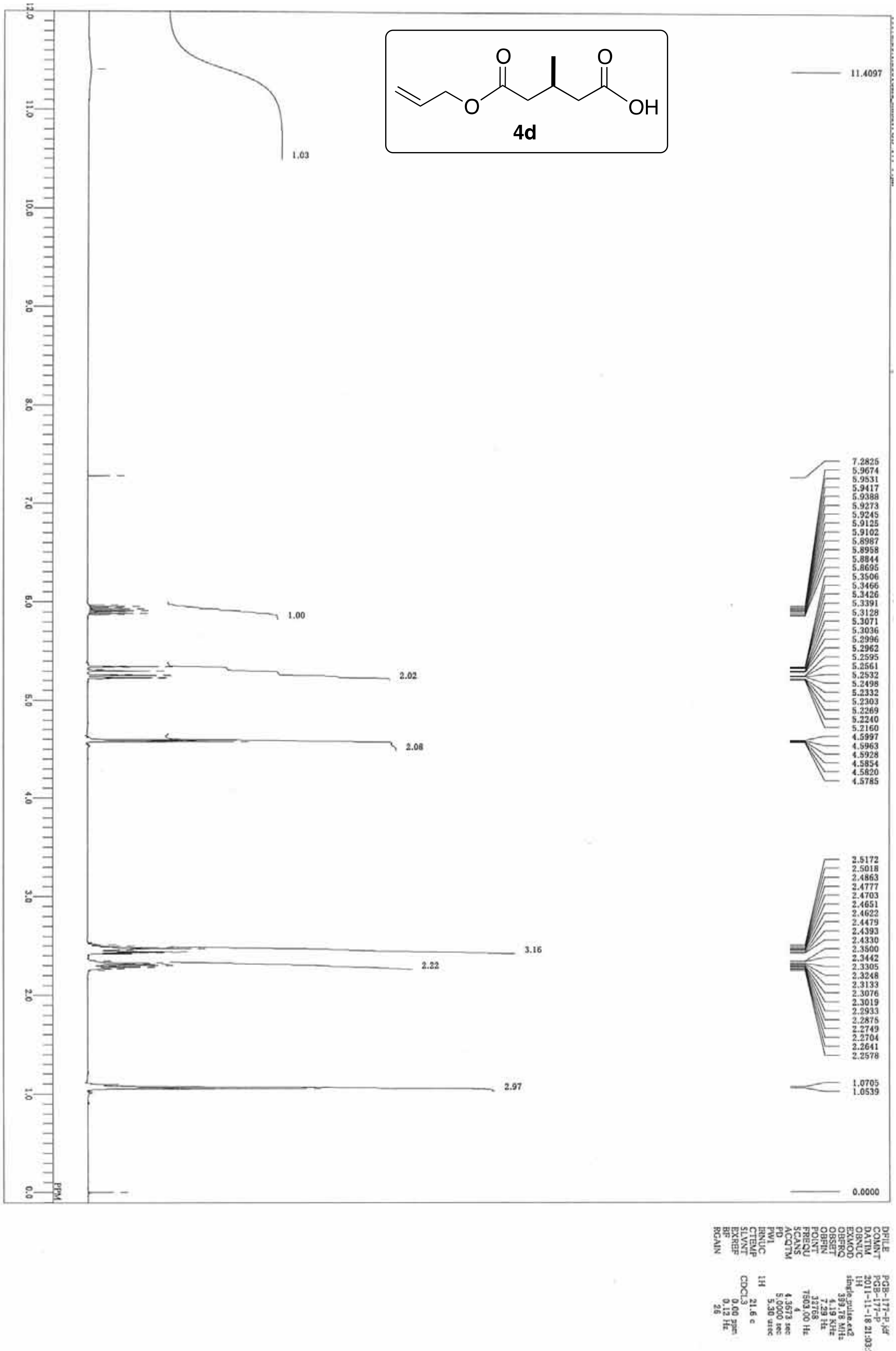

O
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4d
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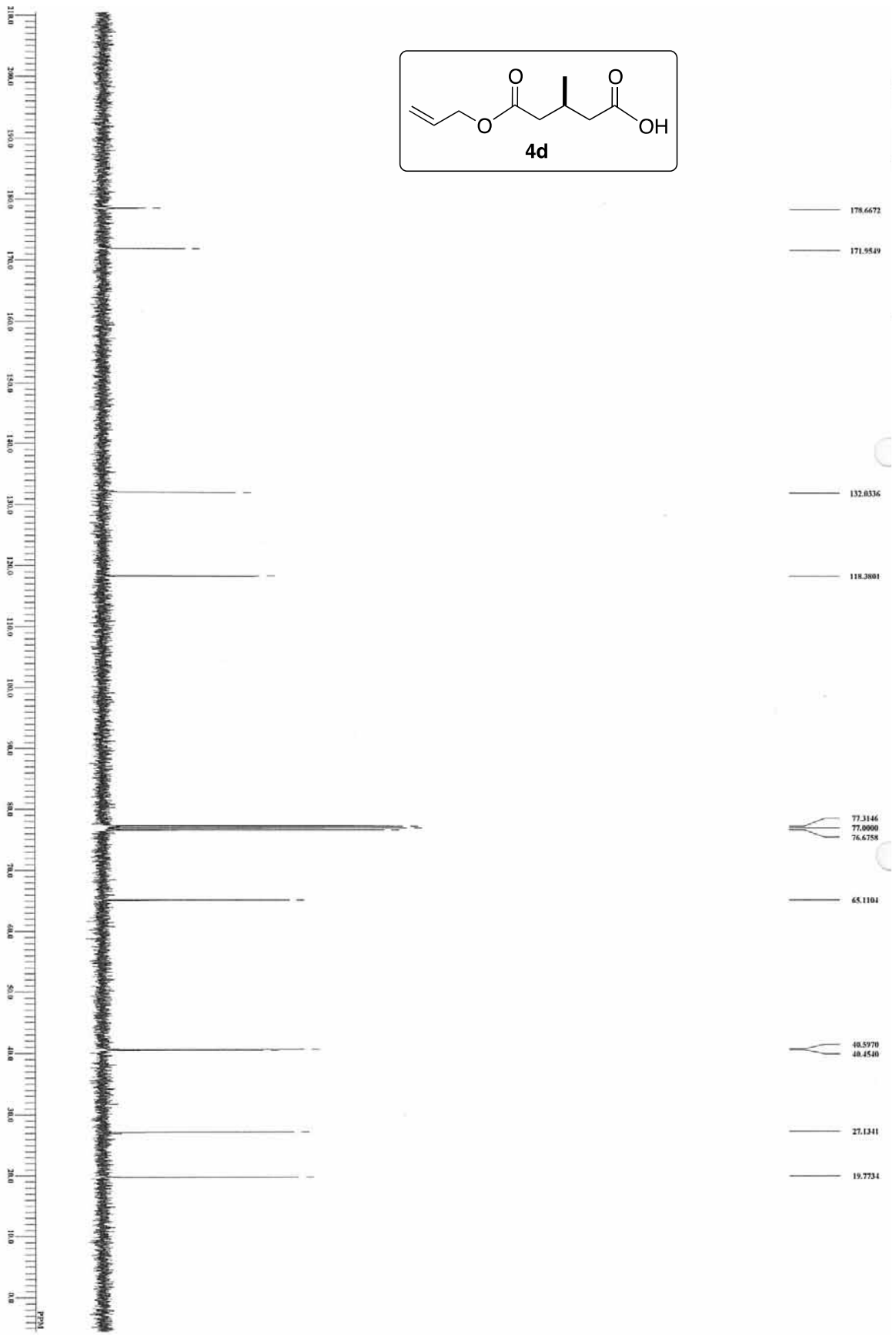
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O
OH
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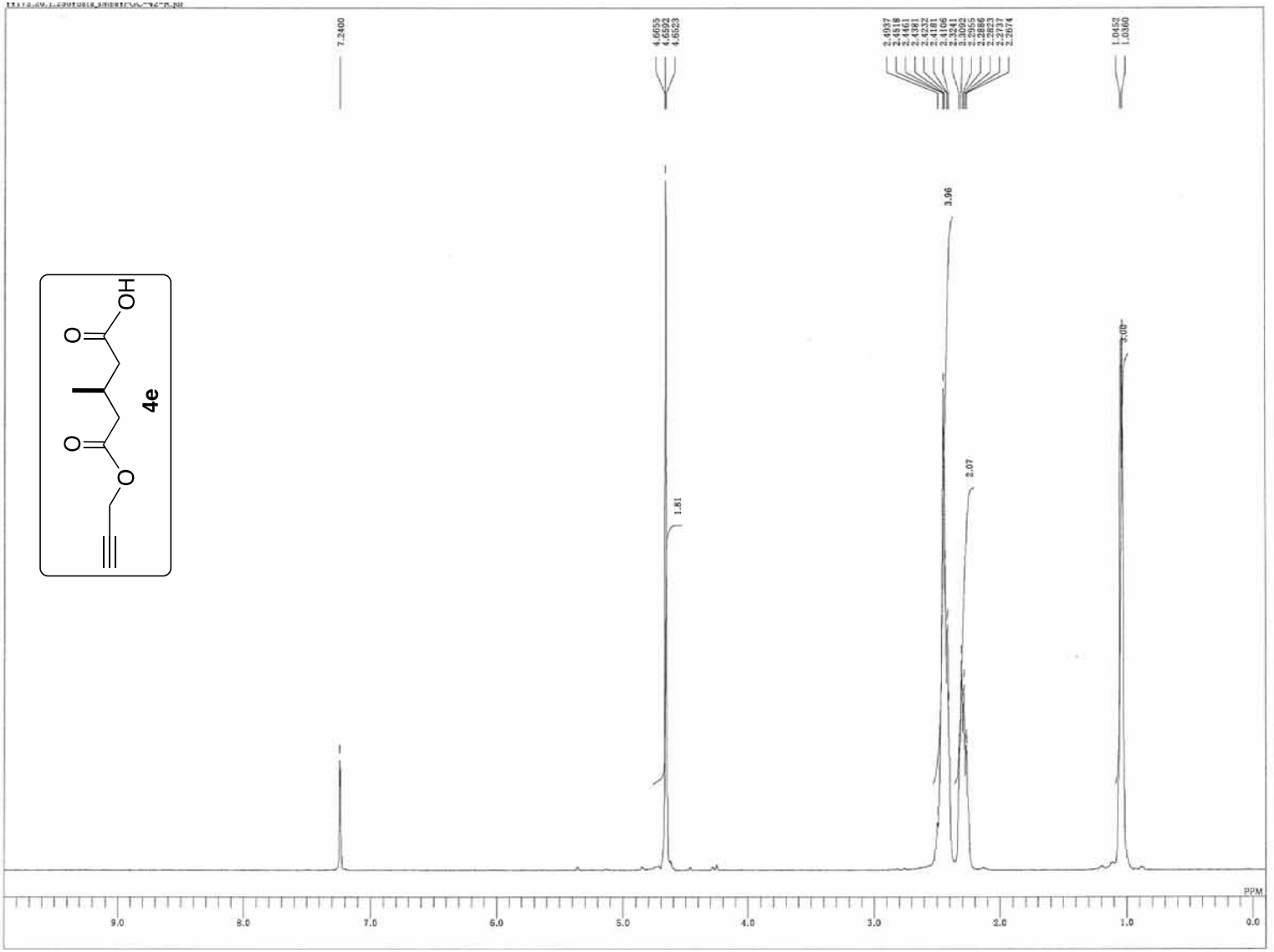
ppm

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OBNUC     1H
EXMOD     single_pulse.ex2
OBFRQ     399.78 MHz
OBSET     4.19 KHz
OBFIN     7.29 Hz
POINT     32768
FREQU     7503.00 Hz
SCANS     4
ACQTM     4.3673 sec
PD        5.0000 sec
PW1       5.16 usec
IRNUC     1H
CTEMP     20.8 c
SLVNT     CDCL3
EXREF     7.24 ppm
BF        0.46 Hz
RGAIN     42
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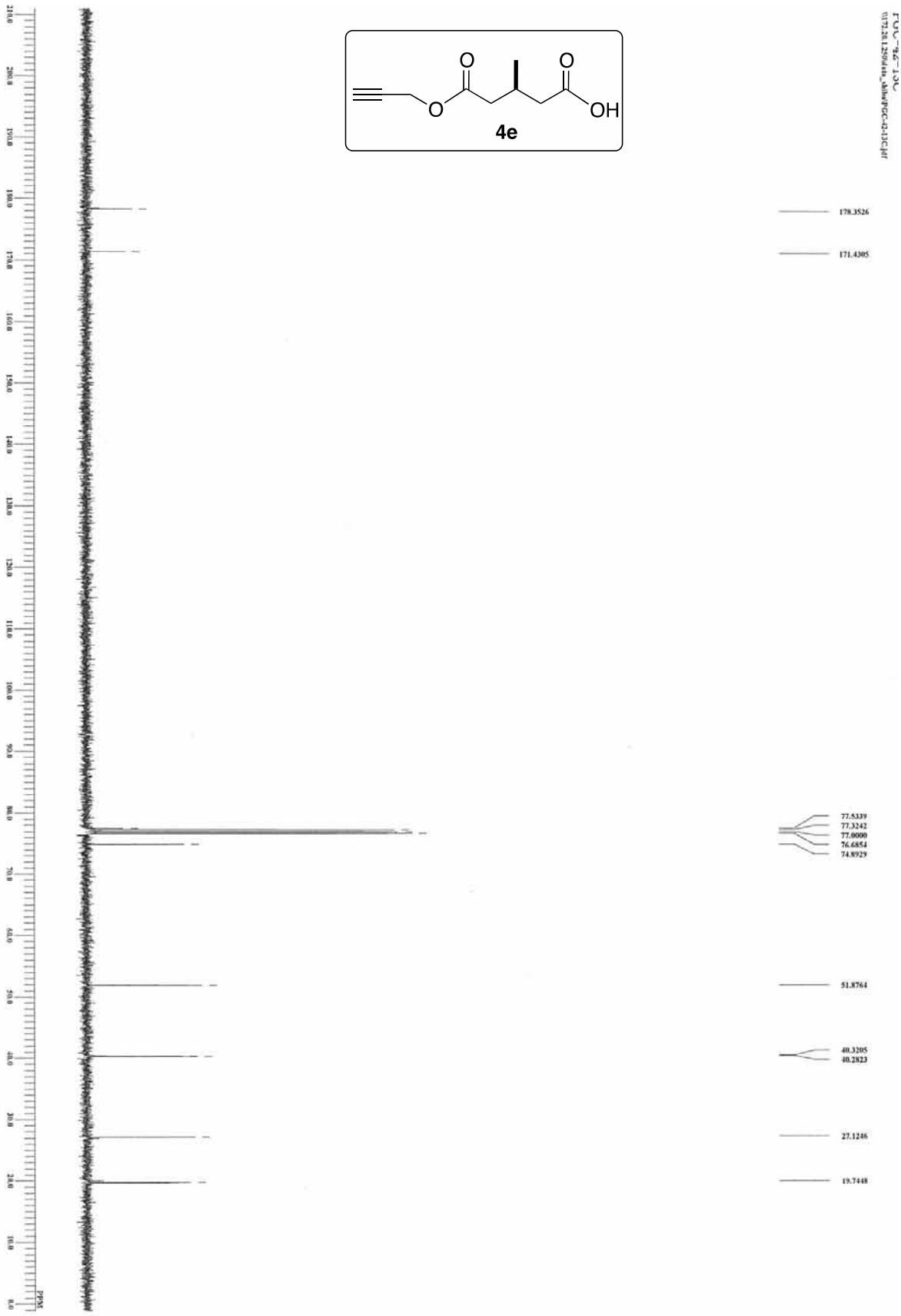
O
O
O
OH
4e
178.3526
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76.6854
74.8929
51.8764
40.3205
40.2823
27.1246
19.7448
PPM