

Supporting Information

Polymer Incarcerated Gold-palladium Nanoclusters with Boron on Carbon: A Mild and Efficient Catalyst for the Sequential Aerobic Oxidation-Michael Addition of 1,3-Dicarbonyl Compounds to Allylic Alcohols

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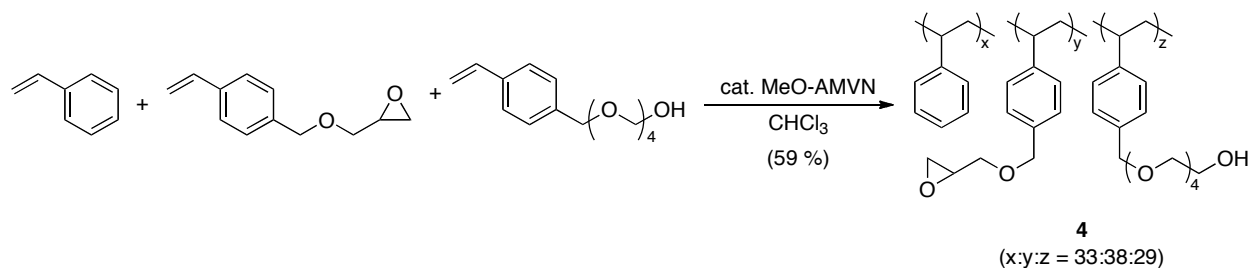
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General Information: Standard column chromatography was performed on 70-230 mesh silica gel (obtained from Merck) using flash column chromatography techniques.¹ IR spectra were measured on a JASCO FT/IR-610 spectrometer. Melting points were obtained using a Büchi Melting Point B-545 and were not corrected. ¹H and ¹³C NMR spectra were recorded on a JEOL ECX-500 in CDCl₃. Chemical shifts were reported in parts per million (ppm) from tetramethylsilane using the solvent resonance as the internal standard (chloroform: δ 7.26 ppm) for ¹H NMR and (deuteriochloroform: δ 77.0 ppm) for ¹³C NMR. ¹¹B CP-MAS NMR spectrum was recorded on a JEOL/JNM ECA-500 spectrometer with a 4mm MAS probe at spectral frequency 160.47 MHz. The boron spectrum was referenced to H₃BO₃ as a standard. High resolution mass spectrometry was carried out using a JEOL JMS-T100TD (DART). The content of all metals in the final material was determined by ICP analysis using a Shimadzu ICPS-7510 equipment: 10-20 mg of the final material was heated in a mixture of sulfuric acid and nitric acid at 200 °C for 2 hours. After cooling at room temperature, *aqua regia* (HCl:HNO₃ = 1:3 v/v) was added and the samples were diluted to 50 mL with distilled water. STEM images and EDS analyses were obtained using a JEOL JEM-2100F instrument operated at 200 kV. All TEM specimens were prepared by placing a drop of a suspension of the sample in methanol on carbon-coated copper grids and allowed to dry in air without staining. SEM images were obtained using a JEOL JSM-6700F instrument operated at 5.0 KV. Nitrogen adsorption measurements at 77 K were performed on a ASAP 2010 volumetric adsorption analyzer surface area and porosity analyzer equipped with pressure transducers covering the 1, 10, and 1000 Torr ranges (Micromeritics Instrument Corp.). The instrument was equipped with a turbo vacuum pump capable of reducing the pressure to below 10⁻⁷ Torr. The initial pressure at the start of any run was below 10⁻⁵ Torr.

Reagents: Unless stated otherwise, commercial reagents were used as received with the exception of NaBH₄, which was recrystallized from diglyme according to the literature.² The carbon black source used in this study was ketjen black EC 300J purchased from Lion Corporation. Allylic alcohols **2b**, **2d-i** were prepared according to the literature.³

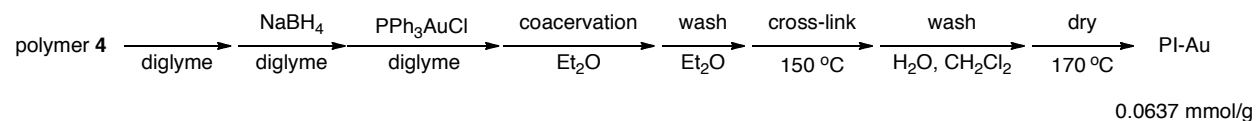
Part I: Preparation of PI-M (M = Au, Au-Sc, B) and PI/CB-B Catalysts

Preparation of Polymer 4



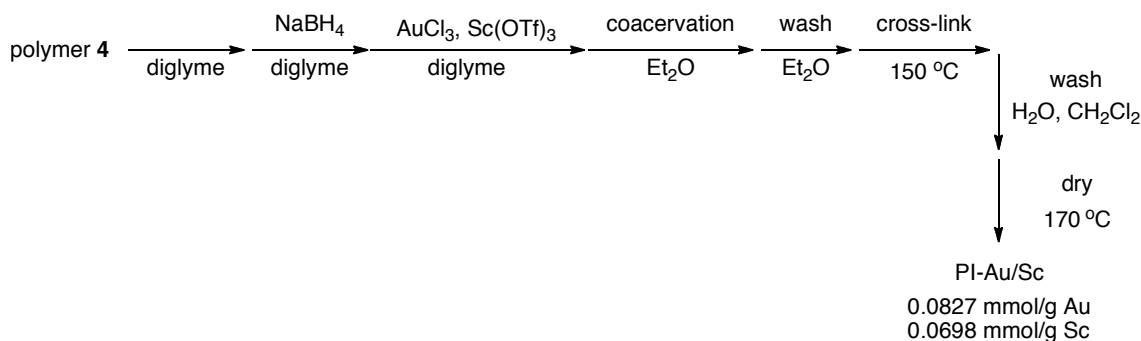
Polymer **4** was prepared according to the described procedure:⁴ styrene (3.21 g, 30.8 mmol), 4-vinylbenzyl glycidyl ether (5.88 g, 30.9 mmol), 2-(2-(2-(4-vinylbenzyloxy)ethoxy)ethoxy)ethanol (8.22 g, 30.9 mmol) and 2,2'-azobis(4-methoxy)-2,4-valeronitrile (0.2370 g, 0.769 mmol) were combined in chloroform (15.0 mL). The mixture was stirred for 48 h at room temperature. The resulting polymer was precipitated into diethyl ether, filtered, washed with diethyl ether and dried for 24 h *in vacuo* to afford the desired product **4** as white foam (10.25 g, 59%). The molar ratio of each functionalities was determined by ¹H NMR spectroscopy (x:y:z = 33:38:29). M_n and M_w were determined by GPC (M_n = 19,785, M_w = 23,188, M_w/M_n = 1.2).

Preparation of PI-Au



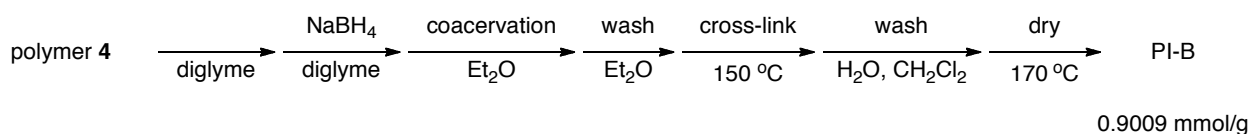
PI Au was prepared according to the described procedure:⁴ to a solution of polymer **4** (0.9028 g) in diglyme (13 mL) was added NaBH₄ (56.3 mg, 1.488 mmol). Then a solution of PPh₃AuCl (35.8 mg, 0.0722 mmol) in diglyme (4 mL) was added dropwise and the reaction was allowed to stir overnight at room temperature. Then diethyl ether (80 mL) was added dropwise. The coacervated Au polymer was washed several times with diethyl ether and was dried at room temperature. Next, the polymer was heated at 150 °C for 5 h without solvent. The cross-linked polymer was washed with DCM and H₂O, filtered, grounded with a motor and pestle, and heated at 170 °C for 5 h to provide a wine red solid (PI-Au, 0.9146 g, Au loading: 0.0637 mmol/g, B loading: 1.469 mmol/g).

Preparation of PI-Au/Sc



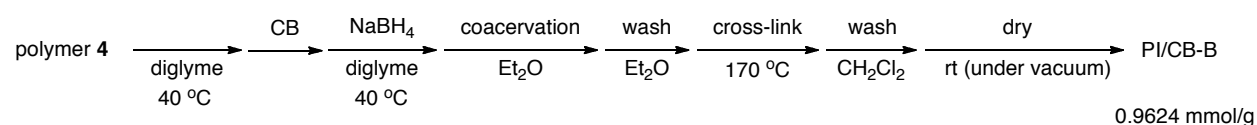
PI Au was prepared according to the described procedure: to a solution of polymer **4** (0.3768 g) in diglyme (7 mL) was added NaBH_4 (4.7 mg, 0.1242 mmol). Then a solution of AuCl_3 (9.5 mg, 0.0314 mmol) and $\text{Sc}(\text{OTf})_3$ (15.5 mg, 0.0315 mmol) in diglyme (2 mL) was added dropwise and the reaction was allowed to stir overnight at room temperature. Then diethyl ether (50 mL) was added dropwise. The coacervated Au polymer was washed several times with diethyl ether and was dried at room temperature. Next, the polymer was heated at 150°C for 5 h without solvent. The cross-linked polymer was washed with DCM and H_2O , filtered, grounded with a motor and pestle, and heated at 170°C for 5 h to provide a wine red solid (PI-Au/Sc, 0.2873 g, Au loading: 0.0827 mmol/g, Sc loading: 0.0698 mmol/g).

Preparation of PI-B



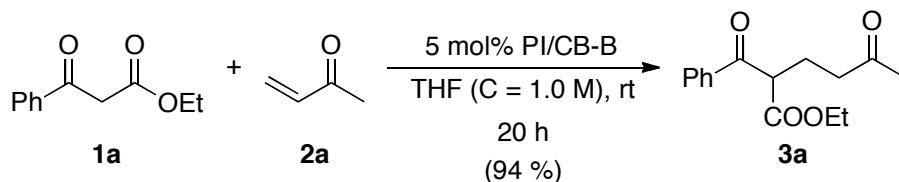
To a solution of polymer **4** (0.8695 g) in diglyme (12 mL) was added dropwise a solution of NaBH_4 (0.0570 g, 1.51 mmol) in diglyme (4 mL). After stirring for 4 h at room temperature, diethyl ether (40 mL) was added dropwise. The precipitated boron immobilized polymer was washed several times with diethyl ether and dried at room temperature. Next, the polymer was heated at 150°C for 8 h without solvent. The cross-linked polymer was washed with DCM and H_2O , filtered, grounded with a motor and pestle and heated at 170°C for 8 h to provide a pale yellow powder (PI-B, 0.7849 g, B loading: 0.9009 mmol/g).

Preparation of PI/CB-B



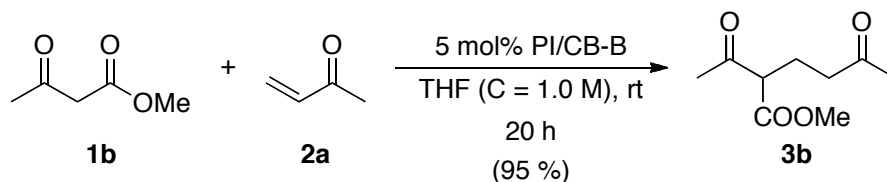
To a solution of polymer 4 (0.1563 g) in diglyme (3.9 mL) at 40 °C was added ketjen black (0.1573 g). After stirring the heterogeneous solution for 15 minutes, a solution of NaBH₄ (17.7 mg, 0.469 mmol) in diglyme (1.6 mL) was added dropwise. After stirring for 4 h at 40 °C, diethyl ether (40 mL) was added dropwise. The boron containing polymer-coated carbon black was washed several times with diethyl ether and dried at room temperature. Next, the recovered solid powder was heated at 170°C for 4 h without solvent. After cooling to room temperature, the cross-linked polymer coated carbon black was washed several times with DCM, filtered, grounded with a motor and pestle and dried under vacuum to provide the desired black powder (PI/CB-B, 0.3312 g, B loading: 0.9624 mmol/g).

Part II: Scope of the Michael Addition of 1,3-Dicarbonyl Compounds to Electron-deficient Olefins



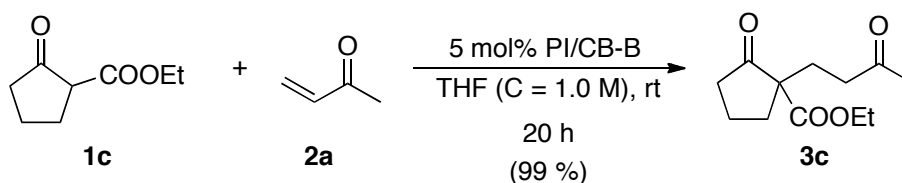
General Procedure for the PI/CB-B-catalyzed Michael Addition of 1,3-Dicarbonyl Compounds to Electron-deficient Olefins. In a screw-cap glass vial was added PI/CB-B (0.0261 g, 0.0251 mmol, 5 mol %), β -ketoester **1a** (87.0 μ L, 0.502 mmol), and vinyl ketone **2a** (61.0 μ L, 0.754 mmol) and dissolved in THF (0.5 mL). The reaction mixture was allowed to stir for 20 h at room temperature. Then the reaction mixture was filtered, washed with DCM, and the resulting filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3a** (0.1239 g, 0.472 mmol, 94%) as a clear colourless oil.

Ethyl 2-benzoyl-5-oxohexanoate (3a) (Table 2, entry 1). R_f 0.22 (EtOAc:hexane = 1:4); ^1H NMR (CDCl_3 , 500 MHz) δ 8.00-7.91 (m, 2H), 7.53-7.50 (m, 1H), 7.43-7.40 (m, 2H), 4.37 (dd, J = 6.3, 6.2 Hz, 1H), 4.10-4.04 (m, 2H), 2.58-2.47 (m, 2H), 2.21-2.10 (m, 2H), 2.06 (s, 3H), 1.09 (t, J = 7.4 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 207.8, 195.2, 169.7, 135.8, 133.6, 128.7, 128.6, 61.3, 52.5, 40.4, 29.9, 22.6, 13.9. This is a known compound and the spectral data are identical to those reported in literature.⁵

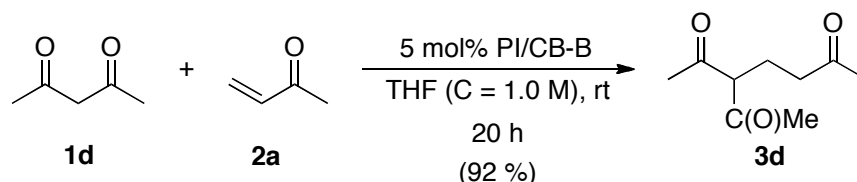


Methyl 2-acetyl-5-oxohexanoate (3b) (Table 2, entry 2). Following the above general procedure with β -ketoester **1b** (54.0 μ L, 0.500 mmol) and vinyl ketone **2a** (61.0 μ L, 0.754 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3b** (0.0886 g, 0.476 mmol, 95%) as a clear colourless oil. R_f 0.30 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 3.67 (s, 3H), 3.46 (t, J = 7.4 Hz, 1H), 2.46-2.42 (m, 2H), 2.18 (s, 3H), 2.07 (s, 3H), 2.05-2.00 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 207.4, 202.8, 169.9,

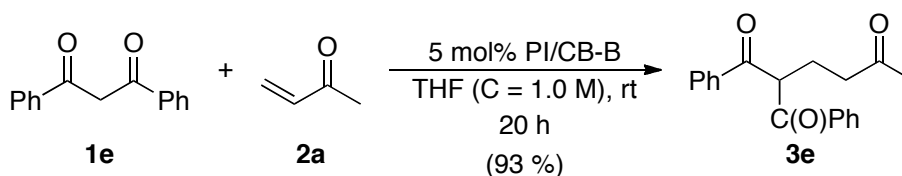
57.9, 52.4, 40.3, 29.9, 29.0, 21.6. This is a known compound and the spectral data are identical to those reported in literature.⁶



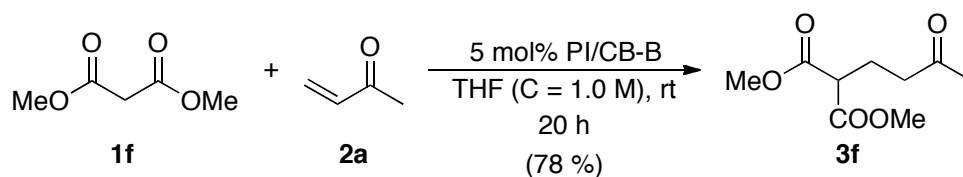
Ethyl 2-oxo-1-(3-oxobutyl)cyclopentane carboxylate (3c) (Table 2, entry 3). Following the above general procedure with β -ketoester **1c** (74.0 μ L, 0.499 mmol) and vinyl ketone **2a** (61.0 μ L, 0.754 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3c** (0.1119 g, 0.495 mmol, 99%) as a clear colourless oil. R_f 0.43 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 4.10 (q, J = 6.8 Hz, 2H), 2.67-2.60 (m, 1H), 2.43-2.19 (m, 4H), 2.07-1.80 (m, 8H), 1.18 (t, J = 7.4 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 214.9, 207.7, 171.3, 61.3, 58.8, 38.7, 37.9, 34.2, 29.8, 26.8, 19.5, 13.9. This is a known compound and the spectral data are identical to those reported in literature.⁷



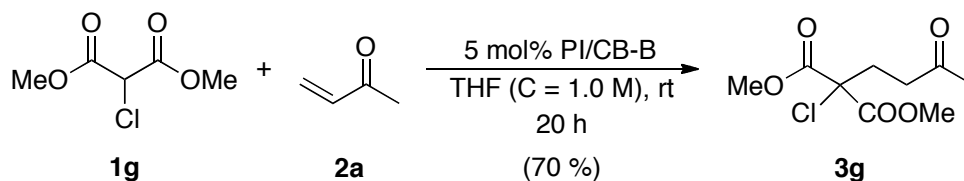
3-Acetylheptane-2,6-dione (3d) (Table 2, entry 4). Following the above general procedure with 1,3-diketone **1d** (51.0 μ L, 0.497 mmol) and vinyl ketone **2a** (60.0 μ L, 0.740 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3d** (0.0781 g, 0.459 mmol, 92%) as a clear colourless oil. R_f 0.33 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 3.62 (t, J = 6.8 Hz, 1H), 2.38 (t, J = 6.8 Hz, 2H), 2.13 (s, 6H), 2.07 (s, 3H), 2.01 (q, J = 6.8 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 207.4, 204.1 (x2), 66.7, 40.4, 30.0, 29.9 (x2), 21.3. This is a known compound and the spectral data are identical to those reported in literature.⁷



2-Benzoyl-1-phenylhexane-1,5-dione (3e) (Table 2, entry 5). Following the above general procedure with 1,3-diketone **1e** (0.1123 g, 0.501 mmol) and vinyl ketone **2a** (61.0 μ L, 0.754 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3e** (0.1365 g, 0.464 mmol, 93%) as a white solid. R_f 0.2 (EtOAc:hexane = 1:4); ^1H NMR (CDCl_3 , 500 MHz) δ 8.04 (d, J = 7.9 Hz, 4H), 7.57 (t, J = 7.4 Hz, 2H), 7.45 (t, J = 7.9 Hz, 4H), 5.49 (t, J = 6.8 Hz, 1H), 2.70 (t, J = 6.8 Hz, 2H), 2.32 (dd, J = 13.0, 6.2 Hz, 2H), 2.12 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 208.7, 196.3, 135.7, 133.6, 128.9, 128.6, 54.6, 40.7, 30.0, 23.1. This is a known compound and the spectral data are identical to those reported in literature.⁸

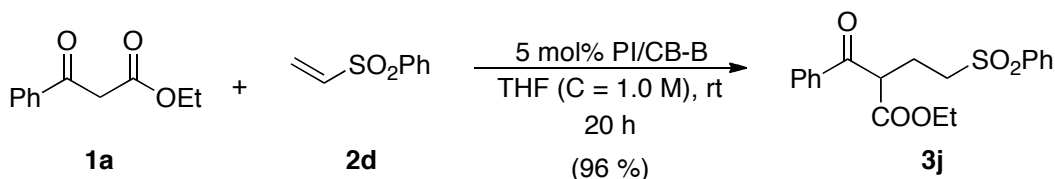


Dimethyl 2-(3-oxobutyl)malonate (3f) (Table 2, entry 6). Following the above general procedure with malonate **1f** (57.0 μ L, 0.499 mmol) and vinyl ketone **2a** (61.0 μ L, 0.754 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3f** (0.0787 g, 0.389 mmol, 78%) as a clear colourless oil. R_f 0.42 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 3.67 (s, 6H), 3.38 (t, J = 7.4 Hz, 1H), 2.48 (t, J = 7.4 Hz, 1H), 2.09 (d, J = 7.4 Hz, 2H), 2.08 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 207.2, 169.5, 52.5, 50.2, 40.3, 29.9, 22.4. This is a known compound and the spectral data are identical to those reported in literature.⁹



Dimethyl 2-chloro-2-(3-oxobutyl)malonate (3g) (Table 2, entry 7). Following the above general procedure with malonate **1g** (68.0 μ L, 0.501 mmol) and vinyl ketone **2a** (61.0 μ L, 0.754 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3g** (0.0829 g, 0.350 mmol, 70%) as a clear colourless oil. R_f 0.22 (EtOAc:hexane

42.4, 32.7, 23.2, 13.9. This is a known compound and the spectral data are identical to those reported in literature.¹⁰



Ethyl 2-benzoyl-4-(phenylsulfonyl)butanoate (3j) (Table 2, entry 10). Following the above general procedure with β -ketoester **1a** (87.0 μL , 0.502 mmol) and vinyl sulfone **2d** (0.0930 g, 0.553 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3j** (0.1743 g, 0.484 mmol, 96%) as a clear colourless oil. R_f 0.38 (EtOAc:hexane = 2:3); IR (neat, NaCl): 3068 (m), 2988 (m), 2939 (m), 2329 (w), 1971 (w), 1739 (s), 1685 (s), 1600 (m), 1446 (s), 1248 (s), 1154 (s), 1085 (s) cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 7.98-7.96 (m, 2H), 7.90-7.88 (m, 2H), 7.66-7.54 (m, 4H), 7.47-7.46 (m, 2H), 4.66 (t, J = 6.8 Hz, 1H), 4.10 (q, J = 7.4 Hz, 2H), 3.27-3.19 (m, 2H), 2.41-2.35 (m, 2H), 1.12 (t, J = 7.4 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 194.1, 168.8, 138.6, 135.5, 133.89, 133.87, 129.3, 128.8, 128.7, 127.9, 61.8, 53.3, 51.5, 21.9, 13.8; ESI-HRMS (m/z) calcd. for $\text{C}_{19}\text{H}_{21}\text{O}_5\text{S}_2$ [(M+H) $^+$]: 361.11097, found: 361.11270.

Part III: Recover and Reuse of PI/CB-B Catalyst for the Michael Reaction

General Recover and Reuse Procedure for the PI-B/C-catalyzed Michael Addition of Ethyl Benzoylacetate (1a) to Methyl Vinyl Ketone (2a). In a screw-cap glass vial was added PI/CB-B (0.0261g, 0.0251 mmol, 5 mol %), β -ketoester **1a** (87.0 μL , 0.502 mmol), and vinyl ketone **2a** (61.0 μL , 0.754 mmol) that was purified *via* filtration through a plug of basic alumina or by distillation and dissolved in THF (0.5 mL). The reaction mixture was allowed to stir for 20 h at room temperature. Then the reaction mixture was filtered, washed with DCM, and the resulting filtrate was concentrated under reduced pressure. The yield of Michael adduct **3a** was determined by ^1H NMR of the crude reaction mixture using mesitylene as an internal standard. The leaching of boron was checked by ICP analysis of the crude reaction mixture. The recovered PI/CB-B catalyst (approx. 1 mg was lost during filtration) was dried under vacuum for 2 h at room temperature and reused for the subsequent Michael reaction.

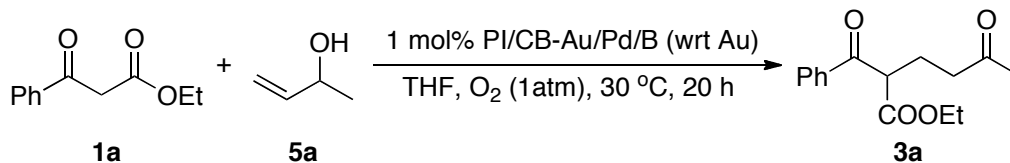
Part IV: Preparation of Bimetallic PI/CB Catalysts

Table S-1. General Preparation Method for Bimetallic PI/CB Catalysts

Polymer 4	→ diglyme	→ CB	→ NaBH ₄ diglyme (0 °C) (15 min)	→ PPh ₃ AuCl, [M] diglyme (0 °C to rt) (4 h)	→ Et ₂ O filter + wash (Et ₂ O)	↓ 170 °C (4 h)	↓ filter + wash (DCM)	← dry	PI/CB Au/ M /B
<div style="border: 1px solid black; padding: 5px; display: inline-block;"> Target Loading: Au = 0.28 mmol/g M = 0.28 mmol/g B = 4.2 mmol/g </div>									
entry	M	Au (mmol/g)	M (mmol/g)	B (mmol/g)					
1	Na ₂ PtCl ₆	0.1092	0.1105	2.656					
2	Pd(OAc) ₂	0.2007	0.2183	2.456					
3	Cu(OAc) ₂	0.2096	0.1545	2.335					
4	(PPh ₃) ₂ NiCl ₂	0.1526	0.2241	2.306					
5	CoCl ₂	0.1437	0.1538	2.524					
6	[RhCl(COD)] ₂	0.1605	0.1749	2.447					
7	FeCl ₂	0.1784	0.1687	2.564					
8	[RuCl ₂ (p-cymene)] ₂	0.1655	0.1527	2.383					

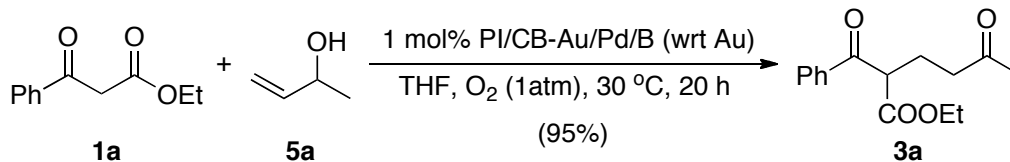
Preparation of PI/CB-Au/Pd/B: To a solution of **4** (0.1448 g) in diglyme (9.6 mL) was added ketjen black (0.1458 g). The resulting suspension was cooled to 0 °C and then a solution of NaBH₄ (46.0 mg, 1.21 mmol) in diglyme (2.2 mL) was added dropwise. The reaction mixture was allowed to stir for approximately 15 min, then a solution of PPh₃AuCl (40.2 mg, 0.0811 mmol) and Pd(OAc)₂ (18.2 mg, 0.0811 mmol) in diglyme (5.8 mL) was added dropwise. The reaction mixture was allowed to warm to room temperature and was stir for 4 h. Diethyl ether (80 mL) was added dropwise and then the suspension was filtered and washed with excess diethyl ether. The isolated polymer-coated carbon black was allowed to dry, gently grounded with motor and pestle, and then was heated at 170 °C for 4 h. The polymer-incarcerated catalyst was then re-suspended in DCM, filtered, washed with excess DCM, and allowed to dry to provide PI/CB-Au/Pd/B (0.4035 g, Au loading: 0.2007 mmol/g, Pd loading: 0.2183 mmol/g, B loading: 2.456 mmol/g).

Part V: Recover and Reuse of the PI/CB-Au/Pd/B Catalyst for the Sequential Aerobic Oxidation-Michael Reaction



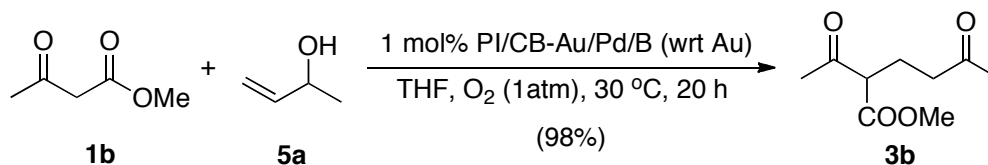
General Recover and Reuse Procedure for the PI/CB-Au/Pd/B-catalyzed Aerobic Oxidation-Michael Addition of Ethyl Benzoylacetate (1a**) to 1-Buten-3-ol (**5a**).** In a screw-cap test tube was added PI/CB-Au/Pd/B (0.0250 g, 5.02 μ mol, 1 mol % with respect to Au), β -ketoester **1a** (87.0 μ L, 0.502 mmol), and allylic alcohol **5a** (65.0 μ L, 0.750 mmol) and was dissolved in THF (0.5 mL). The reaction mixture was allowed to stir for 20 h at 30 °C with a balloon of oxygen gas. Then the reaction mixture was filtered, washed with DCM, and the resulting filtrate was concentrated under reduced pressure. The yield of **3a** was determined by ¹H NMR of the crude reaction mixture using mesitylene as an internal standard. The leaching of the metals was determined by ICP analysis of the crude reaction mixture. The recovered PI/CB-Au/Pd/B catalyst (approx. 1-4 mg was lost during filtration) was dried under vacuum for 2 h at room temperature and reused for the subsequent sequential reaction.

Part VI: Scope of the PI/CB-Au/Pd/B-catalyzed Sequential Reaction



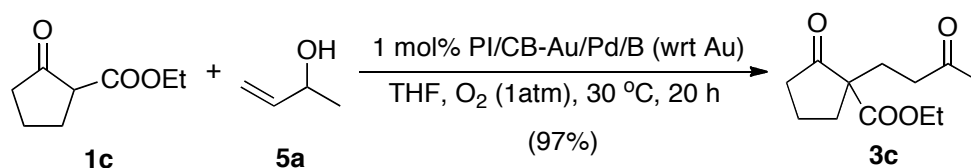
General Procedure for the PI/CB-Au/Pd/B-catalyzed Sequential Aerobic Oxidation-Michael Addition of 1,3-Dicarbonyl Compounds to Allylic Alcohols. In a screw-cap test tube was added PI/CB-Au/Pd/B (0.0250 g, 5.02 μ mol, 1 mol % with respect to Au), β -ketoester **1a** (87.0 μ L, 0.502 mmol), and allylic alcohol **5a** (65.0 μ L, 0.750 mmol) and was dissolved in THF (0.5 mL). The reaction mixture was allowed to stir for 20 h at 30 °C (or 60 °C in certain cases) under a balloon of oxygen gas. Then the reaction mixture was filtered, washed with DCM, and the resulting filtrate was concentrated under reduced pressure. The crude product was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3a** (0.1253 g, 0.478 mmol, 95%) as a clear colourless oil.

Ethyl 2-benzoyl-5-oxohexanoate (3a) (Table 6, entry 1). R_f 0.22 (EtOAc:hexane = 1:4); ¹H NMR (CDCl₃, 500 MHz) δ 8.00-7.91 (m, 2H), 7.53-7.50 (m, 1H), 7.43-7.40 (m, 2H), 4.37 (dd, J = 6.3, 6.2 Hz, 1H), 4.10-4.04 (m, 2H), 2.58-2.47 (m, 2H), 2.21-2.10 (m, 2H), 2.06 (s, 3H), 1.09 (t, J = 7.4 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 207.8, 195.2, 169.7, 135.8, 133.6, 128.7, 128.6, 61.3, 52.5, 40.4, 29.9, 22.6, 13.9. This is a known compound and the spectral data are identical to those reported in literature.⁵

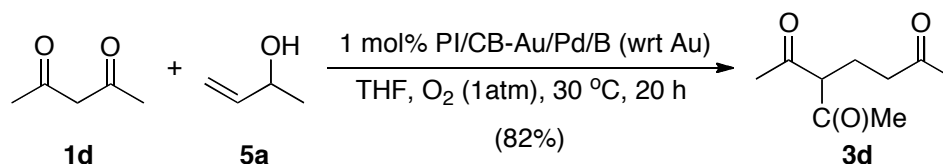


Methyl 2-acetyl-5-oxohexanoate (3b) (Table 6, entry 2). Following the above general procedure with β -ketoester **1b** (54.0 μ L, 0.500 mmol) and allylic alcohol **5a** (65.0 μ L, 0.750 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3b** (0.0912 g, 0.490 mmol, 98%) as a clear colourless oil. R_f 0.30 (EtOAc:hexane = 2:3); ¹H NMR (CDCl₃, 500 MHz) δ 3.67 (s, 3H), 3.46 (t, J = 7.4 Hz, 1H), 2.46-2.42 (m, 2H), 2.18 (s, 3H), 2.07 (s, 3H), 2.05-2.00 (m, 2H); ¹³C NMR (CDCl₃, 125 MHz) δ 207.4, 202.8, 169.9,

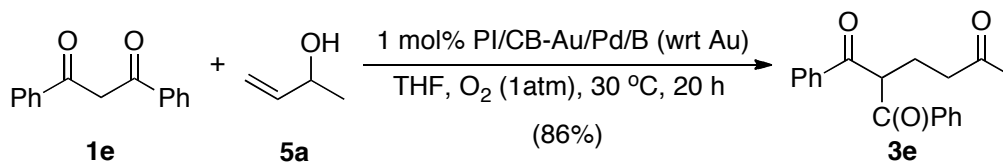
57.9, 52.4, 40.3, 29.9, 29.0, 21.6. This is a known compound and the spectral data are identical to those reported in literature.⁶



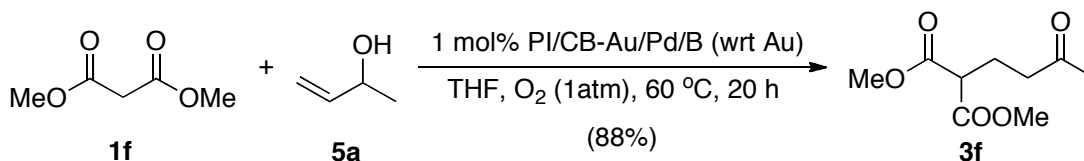
Ethyl 2-oxo-1-(3-oxobutyl)cyclopentane carboxylate (3c) (Table 6, entry 3). Following the above general procedure with β -ketoester **1c** (74.0 μ L, 0.499 mmol) and allylic alcohol **5a** (65.0 μ L, 0.750 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3c** (0.1092 g, 0.483 mmol, 97%) as a clear colourless oil. R_f 0.43 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 4.10 (q, J = 6.8 Hz, 2H), 2.67-2.60 (m, 1H), 2.43-2.19 (m, 4H), 2.07-1.80 (m, 8H), 1.18 (t, J = 7.4 Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 214.9, 207.7, 171.3, 61.3, 58.8, 38.7, 37.9, 34.2, 29.8, 26.8, 19.5, 13.9. This is a known compound and the spectral data are identical to those reported in literature.⁷



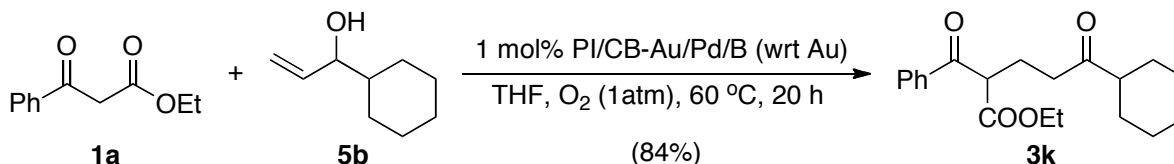
3-Acetylheptane-2,6-dione (3d) (Table 6, entry 4). Following the above general procedure with 1,3-diketone **1d** (50.0 μ L, 0.487 mmol) and allylic alcohol **5a** (65.0 μ L, 0.750 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3d** (0.0680 g, 0.400 mmol, 82%) as a clear colourless oil. R_f 0.33 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 3.62 (t, J = 6.8 Hz, 1H), 2.38 (t, J = 6.8 Hz, 2H), 2.13 (s, 6H), 2.07 (s, 3H), 2.01 (q, J = 6.8 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 207.4, 204.1 (x2), 66.7, 40.4, 30.0, 29.9, 21.3. This is a known compound and the spectral data are identical to those reported in literature.⁷



2-Benzoyl-1-phenylhexane-1,5-dione (3e) (Table 6, entry 5). Following the above general procedure with 1,3-diketone **1e** (0.1121 g, 0.500 mmol) and allylic alcohol **5a** (65.0 μ L, 0.750 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3e** (0.1258 g, 0.427 mmol, 86%) as a white solid. R_f 0.2 (EtOAc:hexane = 1:4); ^1H NMR (CDCl_3 , 500 MHz) δ 8.04 (d, J = 7.9 Hz, 4H), 7.57 (t, J = 7.4 Hz, 2H), 7.45 (t, J = 7.9 Hz, 4H), 5.49 (t, J = 6.8 Hz, 1H), 2.70 (t, J = 6.8 Hz, 2H), 2.32 (dd, J = 13.0, 6.2 Hz, 2H), 2.12 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 208.7, 196.3, 135.7, 133.6, 128.9, 128.6, 54.6, 40.7, 30.0, 23.1. This is a known compound and the spectral data are identical to those reported in literature.⁸

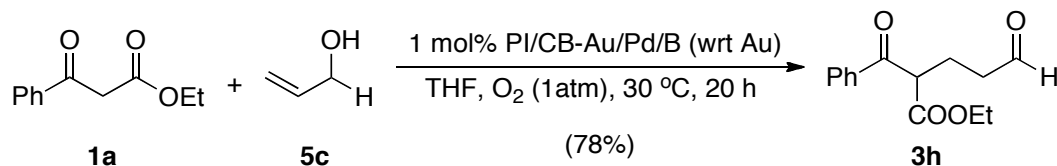


Dimethyl 2-(3-oxobutyl)malonate (3f) (Table 6, entry 6). Following the above general procedure with malonate **1f** (57.0 μ L, 0.499 mmol) and allylic alcohol **5a** (86.0 μ L, 0.992 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3f** (0.0889 g, 0.440 mmol, 88%) as a clear colourless oil. R_f 0.42 (EtOAc:hexane = 2:3); ^1H NMR (CDCl_3 , 500 MHz) δ 3.67 (s, 6H), 3.38 (t, J = 7.4 Hz, 1H), 2.48 (t, J = 7.4 Hz, 1H), 2.09 (d, J = 7.4 Hz, 2H), 2.08 (s, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 207.2, 169.5, 52.5, 50.2, 40.3, 29.9, 22.4. This is a known compound and the spectral data are identical to those reported in literature.⁹

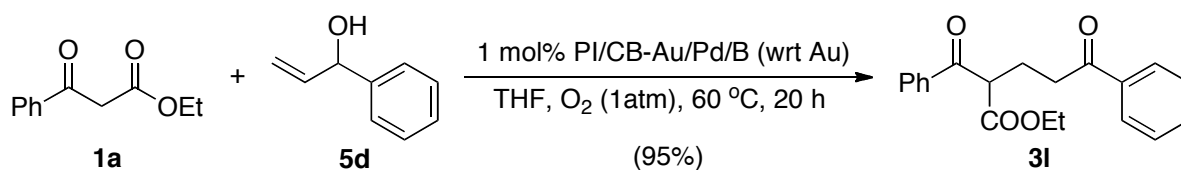


Ethyl 2-benzoyl-5-cyclohexyl-5-oxopentanoate (3k) (Table 6, entry 7). Following the above general procedure with β -ketoester **1a** (87.0 μ L, 0.502 mmol), and allylic alcohol **5b** (77.5 mg, 0.553 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3k** (0.1295 g, 0.422 mmol, 84%) as a clear colourless oil. R_f 0.43 (EtOAc:hexane = 1:4); IR (neat, NaCl): 3062 (m), 2934 (s), 2854 (s), 2666 (w), 1975 (w),

1909 (w), 1739 (s), 1693 (s), 1599 (m), 1448 (s), 1371 (m), 1247 (s), 1032 (m) cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 8.00 (d, $J = 7.3$ Hz, 2H), 7.57-7.54 (m, 1H), 7.45 (t, $J = 7.9$ Hz, 2H), 4.42 (dd, $J = 7.9, 6.2$ Hz, 1H), 4.16-4.07 (m, 2H), 2.64-2.49 (m, 2H), 2.28-2.16 (m, 3H), 1.81-1.62 (m, 4H), 1.30-1.18 (m, 6H), 1.14 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 213.1, 195.3, 169.8, 136.0, 133.5, 128.7, 128.6, 61.2, 52.7, 50.7, 37.3, 28.3, 25.7, 25.5, 22.7, 13.9; ESI-HRMS (m/z) calcd. for $\text{C}_{20}\text{H}_{36}\text{O}_4$ $[(\text{M}+\text{H})^+]$: 331.19093, found: 331.19196.

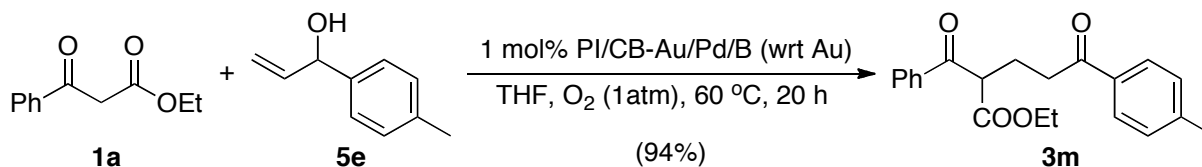


Ethyl 2-benzoyl-5-oxopentanoate (3h) (Table 6, entry 8). Following the above general procedure with β -ketoester **1a** (87.0 μL , 0.502 mmol) and allylic alcohol **5c** (32.0 μL , 0.765 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3h** (0.1029 g, 0.414 mmol, 83%) as a clear colourless oil. R_f 0.22 (EtOAc:hexane = 1:4); ^1H NMR (CDCl_3 , 500 MHz) δ 9.77 (s, 1H), 8.01-8.00 (m, 2H), 7.60-7.57 (m, 1H), 7.48 (t, $J = 7.9$ Hz, 2H), 4.44-4.41 (m, 1H), 4.16-4.10 (m, 2H), 2.68-2.55 (m, 2H), 2.35-2.22 (m, 2H), 1.15 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 201.1, 194.8, 169.5, 135.8, 133.7, 128.8, 128.6, 61.5, 52.6, 41.1, 21.1, 13.9. This is a known compound and the spectral data are identical to those reported in literature.¹⁰

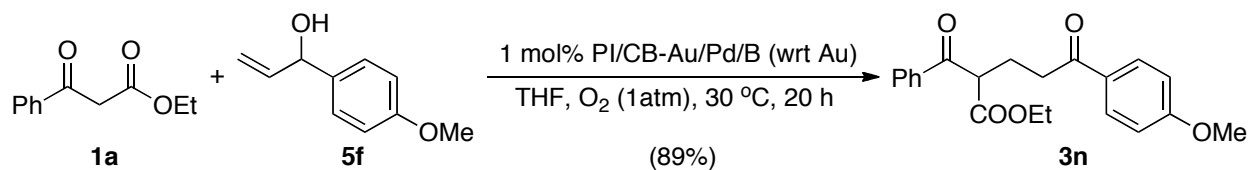


Ethyl 2-benzoyl-5-oxo-5-phenylpentanoate (3l) (Table 6, entry 9). Following the above general procedure with β -ketoester **1a** (87.0 μL , 0.502 mmol), and allylic alcohol **5d** (73.0 μL , 0.555 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3l** (0.1544 g, 0.476 mmol, 95%) as a clear colourless oil. R_f 0.36 (EtOAc:hexane = 1:4); IR (neat, NaCl): 3064 (m), 2981 (s), 2937 (s), 1974 (w), 1913 (w), 1734 (s), 1686 (s), 1596 (m), 1448 (s), 1221 (s), 974 (s) cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 8.07 (d, $J = 7.9$ Hz, 2H), 7.95 (d, $J = 7.4$ Hz, 2H), 7.59-7.53 (m, 2H), 7.50-7.43 (m, 4H), 4.57 (dd, $J =$

7.9, 6.2 Hz, 1H), 4.19-4.10 (m, 2H), 3.22-3.16 (m, 1H), 3.12-3.06 (m, 1H), 2.49-2.36 (m, 2H), 1.15 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 199.2, 195.3, 169.8, 136.6, 135.9, 133.6, 133.1, 128.71, 128.70, 128.6, 128.0, 61.4, 52.8, 35.6, 23.2, 13.9; ESI-HRMS (m/z) calcd. for $\text{C}_{20}\text{H}_{20}\text{O}_4$ $[(\text{M}+\text{H})^+]$: 325.14398, found: 325.14334.

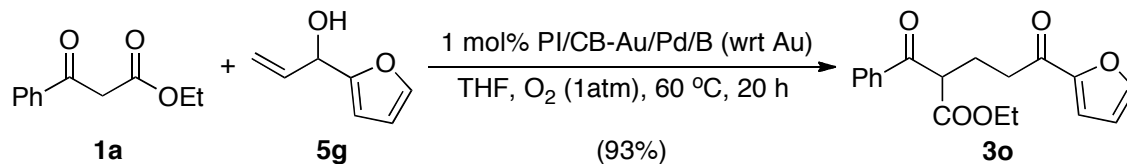


Ethyl 2-benzoyl-5-oxo-5-(*p*-tolyl)pentanoate (3m) (Table 6, entry 10). Following the above general procedure with β -ketoester **1a** (87.0 μL , 0.502 mmol), and allylic alcohol **5e** (81.9 mg, 0.553 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3m** (0.1604 g, 0.474 mmol, 94%) as a clear colourless oil. R_f 0.31 (EtOAc:hexane = 1:4); IR (neat, NaCl): 3061 (m), 3026 (m), 2981 (s), 2934 (s), 1977 (w), 1921 (w), 1737 (s), 1681 (s), 1606 (s), 1449 (m), 1369 (m), 1226 (s), 977 (m) cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 8.06 (d, $J = 7.4$ Hz, 2H), 7.84 (d, $J = 7.9$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.47 (t, $J = 7.9$ Hz, 2H), 7.23 (d, $J = 7.9$ Hz, 2H), 4.56 (dd, $J = 7.4, 6.2$ Hz, 1H), 4.19-4.09 (m, 2H), 3.18-3.12 (m, 1H), 3.08-3.02 (m, 1H), 2.46-2.34 (m, 5H), 1.14 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 198.8, 195.3, 169.8, 143.9, 135.9, 134.1, 133.5, 129.2, 128.7, 128.0, 61.3, 52.8, 35.5, 23.3, 21.5, 13.9; ESI-HRMS (m/z) calcd. for $\text{C}_{21}\text{H}_{22}\text{O}_4$ $[(\text{M}+\text{H})^+]$: 339.15963, found: 339.16042.

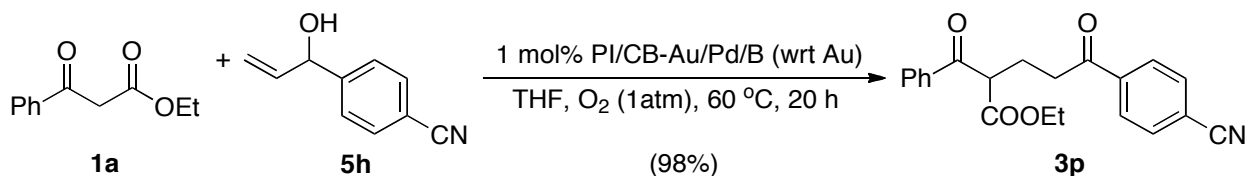


Ethyl 2-benzoyl-5-(4-Methoxyphenyl)-5-oxopentanoate (3n) (Table 6, entry 11). Following the above general procedure with β -ketoester **1a** (87.0 μL , 0.502 mmol), and allylic alcohol **5f** (90.7 mg, 0.552 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3n** (0.1597 g, 0.448 mmol, 89%) as a clear colourless oil. R_f 0.24 (EtOAc:hexane = 1:4); IR (neat, NaCl): 3061 (m), 2979 (s), 2940 (s), 2841 (m), 2057 (w), 1974 (w), 1910 (w), 1737 (s), 1681 (s), 1604 (s), 1513 (s), 1255 (s), 1030 (s), 977 (s) cm^{-1} ; ^1H

NMR (CDCl₃, 500 MHz) δ 8.06 (d, J = 8.5 Hz, 2H), 7.92 (d, J = 9.1 Hz, 2H), 7.58-7.55 (m, 1H), 7.46 (t, J = 7.4 Hz, 2H), 6.90 (d, J = 8.5 Hz, 2H), 4.56 (dd, J = 7.9, 6.8 Hz, 1H), 4.18-4.08 (m, 2H), 3.83 (s, 3H), 3.15-3.08 (m, 1H), 3.05-2.97 (m, 1H), 2.46-2.3 (m, 2H), 1.14 (s, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 197.7, 195.4, 169.8, 163.5, 135.9, 133.5, 130.2, 129.7, 128.7, 113.6, 61.3, 55.4, 52.9, 35.2, 23.4, 13.9; ESI-HRMS (m/z) calcd. for C₂₁H₂₂O₅ [(M+H)⁺]: 355.15455, found: 355.15293.

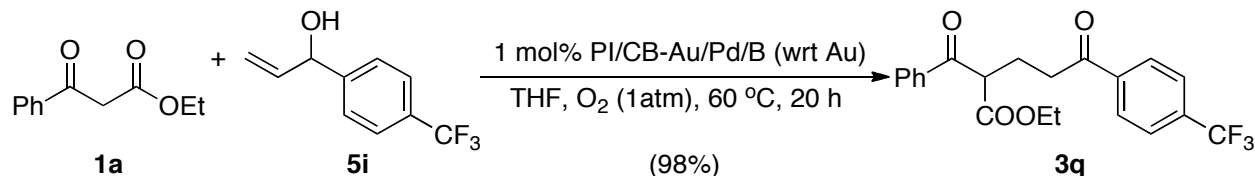


Ethyl 2-benzoyl-5-(furan-2-yl)-5-oxopentanoate (3o) (Table 6, entry 12). Following the above general procedure with β -ketoester **1a** (87.0 μ L, 0.502 mmol), and allylic alcohol **5g** (68.6 mg, 0.553 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3o** (0.1467 g, 0.467 mmol, 93%) as a clear colourless oil. R_f 0.25 (EtOAc:hexane = 1:4); IR (neat, NaCl): 3139 (s), 3067 (s), 2981 (s), 2937 (s), 2546 (w), 2393 (w), 1974 (w), 1913 (w), 1734 (s), 1678 (s), 1571 (s), 1449 (s), 1366 (s), 1239 (s), 764 (m) cm⁻¹; ¹H NMR (CDCl₃, 500 MHz) δ 7.97 (d, J = 7.4 Hz, 2H), 7.51-7.47 (m, 2H), 7.39 (t, J = 7.4 Hz, 2H), 7.11 (d, J = 3.4 Hz, 1H), 6.44-6.43 (m, 1H), 4.46 (dd, J = 7.9, 6.2 Hz, 1H), 4.09-4.03 (m, 2H), 2.98-2.83 (m, 2H), 2.38-2.26 (m, 2H), 1.07 (t, J = 6.2 Hz, 3H); ¹³C NMR (CDCl₃, 125 MHz) δ 195.1, 188.2, 169.6, 152.3, 146.4, 135.8, 133.5, 128.64, 128.62, 117.1, 112.1, 61.3, 52.7, 35.4, 23.0, 13.8; ESI-HRMS (m/z) calcd. for C₁₈H₁₈O₅ [(M+H)⁺]: 315.12325, found: 315.12383.



Ethyl 2-benzoyl-5-(4-cyanophenyl)-5-oxopentanoate (3p) (Table 6, entry 13). Following the above general procedure with β -ketoester **1a** (87.0 μ L, 0.502 mmol), and allylic alcohol **5h** (88.0 mg, 0.553 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 2:3) to provide **3p** (0.1727 g, 0.494 mmol, 98%) as a pale yellow solid (mp. 83-84 °C). R_f 0.20 (EtOAc:hexane = 1:4); IR (KBr): 3070 (m), 2989 (m), 2931 (m), 2907 (m),

2227 (s), 1932 (w), 1744 (s), 1689 (s), 1670 (s), 1596 (m), 1449 (m), 1292 (s), 1198 (s), 1054 (s) cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 8.02-8.00 (m, 4H), 7.72 (d, $J = 8.5$ Hz, 2H), 7.56 (t, $J = 7.4$ Hz, 1H), 7.46 (t, $J = 7.9$ Hz, 2H), 4.51 (t, $J = 8.4$ Hz, 1H), 4.16-4.06 (m, 2H), 3.21-3.06 (m, 2H), 2.47-2.34 (m, 2H), 1.12 (t, $J = 7.4$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 197.8, 194.9, 169.6, 139.4, 135.8, 133.6, 132.4, 128.7, 128.5, 128.3, 117.8, 116.3, 61.4, 52.5, 35.9, 22.9, 13.8; ESI-HRMS (m/z) calcd. for $\text{C}_{21}\text{H}_{19}\text{O}_4\text{N}$ $[(\text{M}+\text{H})^+]$: 350.13923, found: 350.14089.



Ethyl 2-benzoyl-5-oxo-5-(4-(trifluoromethyl)phenyl)pentanoate (3q**) (Table 6, entry 14).**

Following the above general procedure with β -ketoester **1a** (87.0 μL , 0.502 mmol), and allylic alcohol **5i** (111.7 mg, 0.553 mmol). The crude reaction mixture was purified by column chromatography (EtOAc:hexane = 1:4) to provide **3q** (0.1925 g, 0.491 mmol, 98%) as a pale yellow solid (mp. 55-56 °C). R_f 0.49 (EtOAc:hexane = 1:4); IR (KBr): 3067 (w), 2984 (m), 2968 (m), 2907 (m), 1982 (w), 1921 (w), 1725 (s), 1692 (s), 1600 (m), 1452 (m), 1328 (s), 1159 (s), 991 (s) cm^{-1} ; ^1H NMR (CDCl_3 , 500 MHz) δ 8.05-8.03 (m, 4H), 7.70 (d, $J = 8.5$ Hz, 2H), 7.57 (t, $J = 7.4$ Hz, 1H), 7.47 (t, $J = 7.4$ Hz, 2H), 4.54 (t, $J = 7.4$ Hz, 1H), 4.18-4.09 (m, 2H), 3.23-3.08 (m, 2H), 2.49-2.36 (m, 2H), 1.14 (t, $J = 6.8$ Hz, 3H); ^{13}C NMR (CDCl_3 , 125 MHz) δ 198.2, 195.1, 169.7, 139.2, 135.9, 134.4 (q, $^2J_{\text{CF}} = 32$ Hz), 133.6, 128.7, 128.6, 128.3, 125.6 (q, $^3J_{\text{CF}} = 4$ Hz), 123.5 (q, $^1J_{\text{CF}} = 273$ Hz), 61.4, 52.7, 36.0, 23.0, 13.8; ESI-HRMS (m/z) calcd. for $\text{C}_{21}\text{H}_{19}\text{O}_4\text{F}_3$ $[(\text{M}+\text{H})^+]$: 393.13137, found: 393.13140.

Part VII: Control Experiments

The PI/CB catalysts used in the control experiments were prepared following the general procedure outlined in Part IV. For the direct comparison between PI/CB-Au/Pd/B (0.1791 mmol/g Au, 0.2054 mmol/g Pd, 2.898 mmol/g B) and PI/CB-Au/Pd (0.2534 mmol/g Au, 0.2860 mmol/g Pd), these catalysts were prepared by initially following the general procedure for PI/CB-Au/Pd/B, except approximately half of the composite material was removed after the cross-linking step (heated at 170 °C) and stirred overnight in a THF:H₂O (1:1, 10 mL) to completely remove any boron. The reactions were monitored by taking a small aliquot of the reaction mixture, diluting with CDCl₃, and then obtaining ¹H NMR spectra.

Part VIII: STEM, EDS, and SEM Analyses

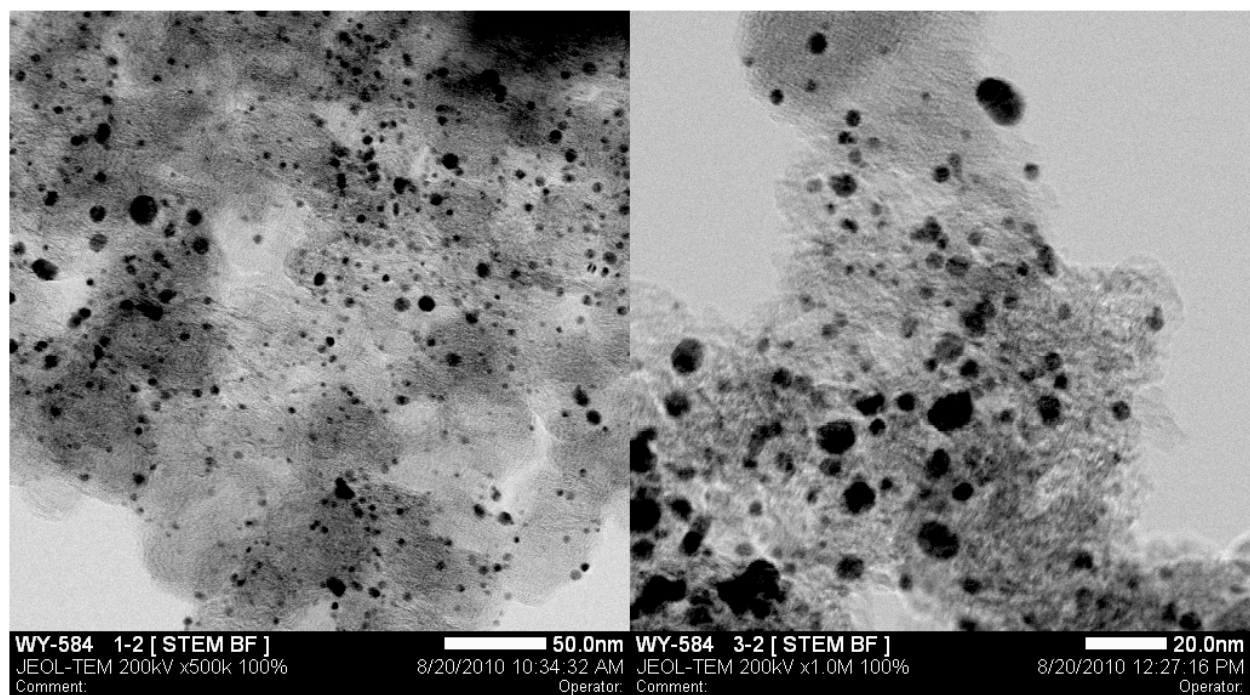


Figure S-1. Typical STEM Images of PI/CB-Au/Pd/B

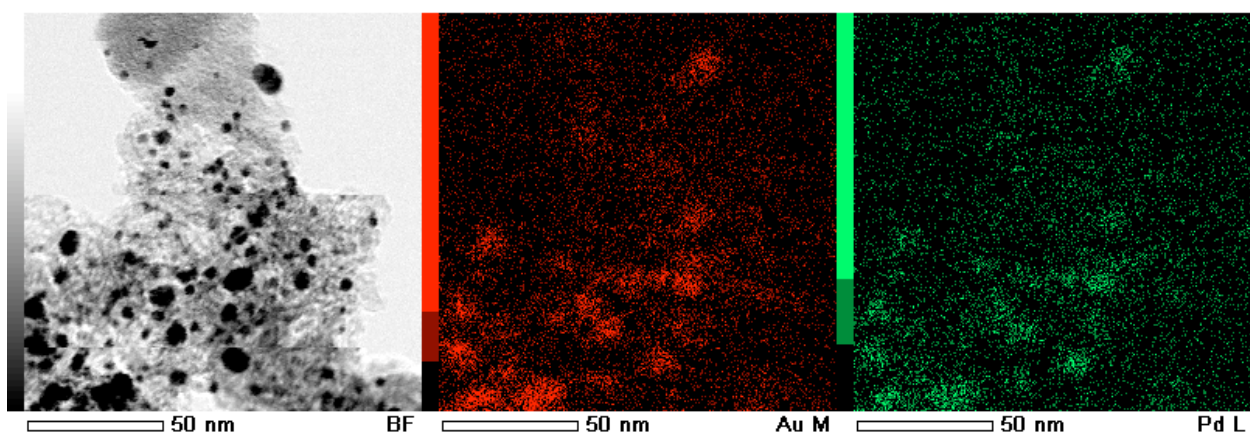


Figure S-2. EDS Mapping of PI/CB-Au/Pd/B

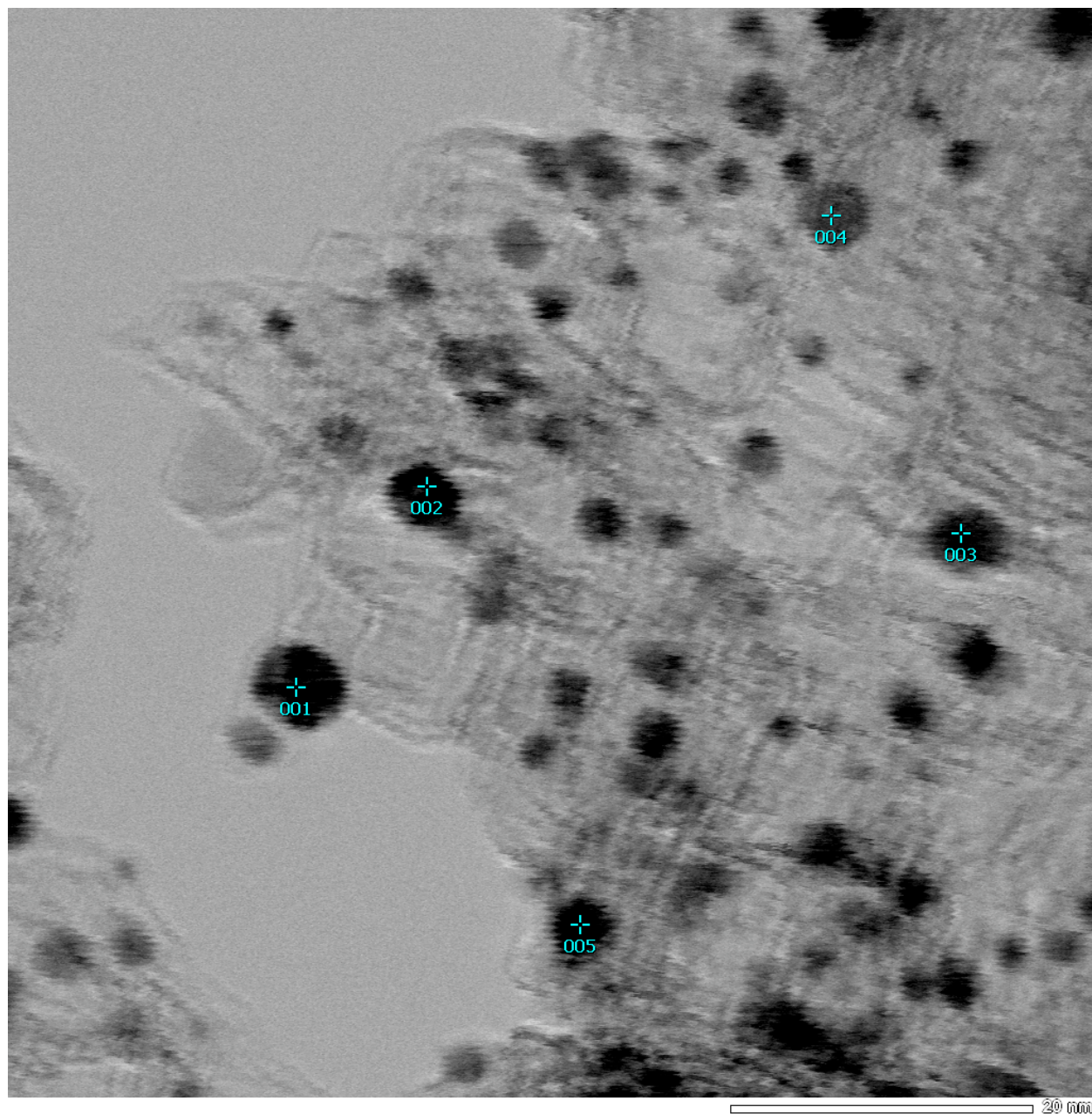


Figure S-3. EDS Spot Analysis of PI/CB-Au/Pd/B

Table S-2. Gold-Palladium Ratio Determined EDS Spot Analysis of PI/CB-Au/Pd/B

Spot	Au (%)	Pd (%)	Ratio Au:Pd
1	55.63	44.37	1.25
2	60.23	39.77	1.51
3	71.26	28.74	2.5
4	35.75	64.25	0.56
5	56.05	43.95	1.28

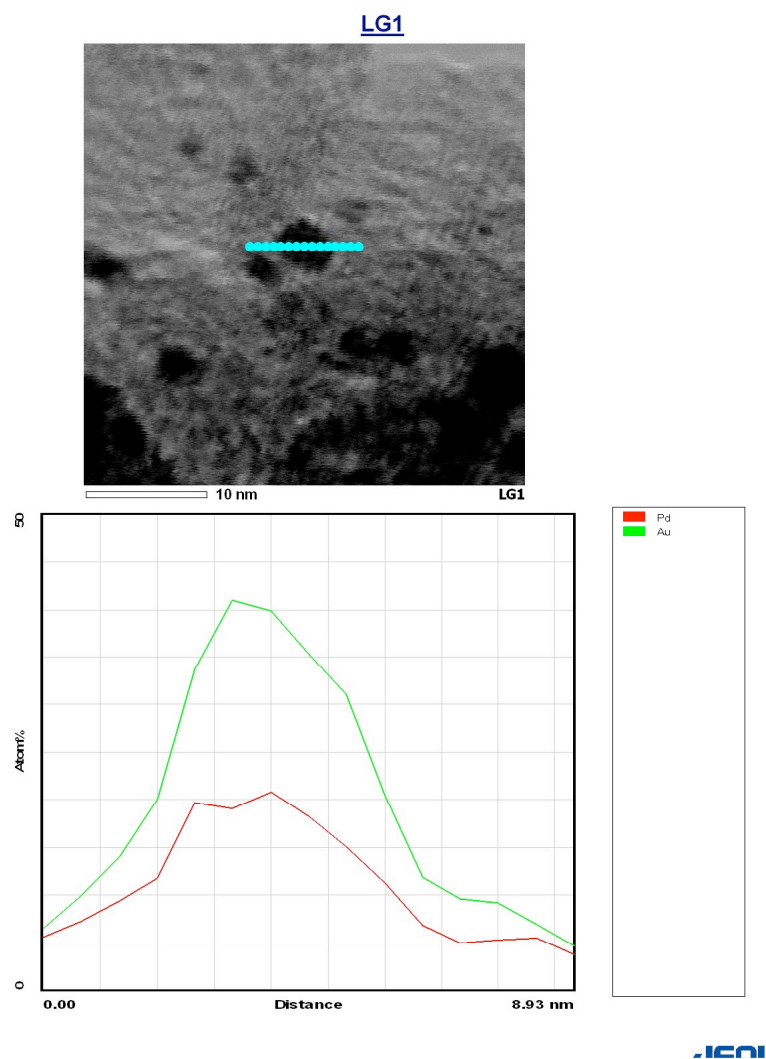


Figure S-4. EDS Line Analysis of PI/CB-Au/Pd/B

Although the gold and palladium metal loading was approximately 1:1 (determined by ICP analysis), the majority of the nanoclusters contained 1.25:1-2.5:1 ratio of gold to palladium. This suggests that palladium exists in both the nanoclusters and also along the polymer backbone. Line analysis of the nanoclusters suggests that the nanoclusters are gold/palladium alloys (green line = Au, red line = Pd).

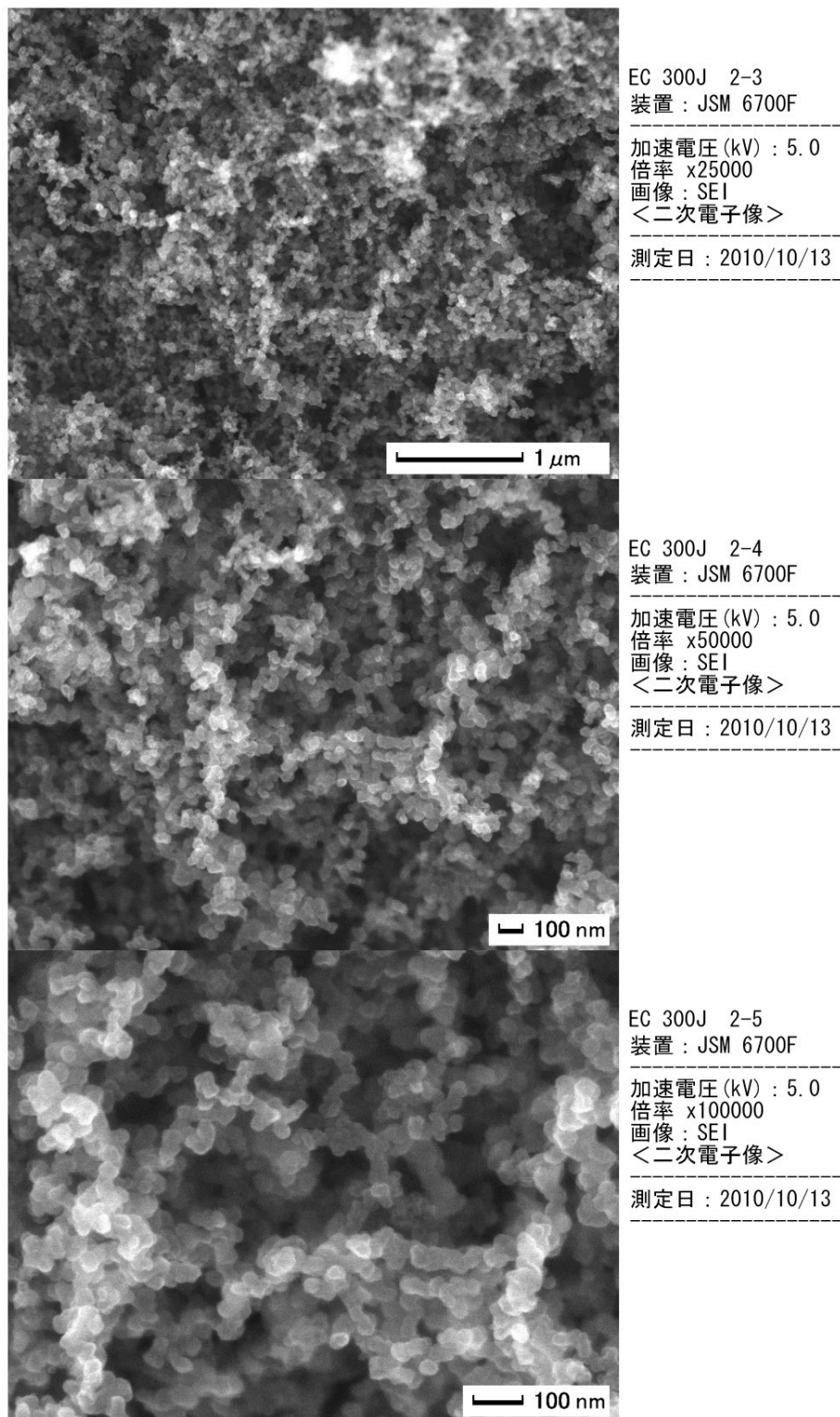


Figure S-5. Typical SEM Image of Ketjen Black EC 300J

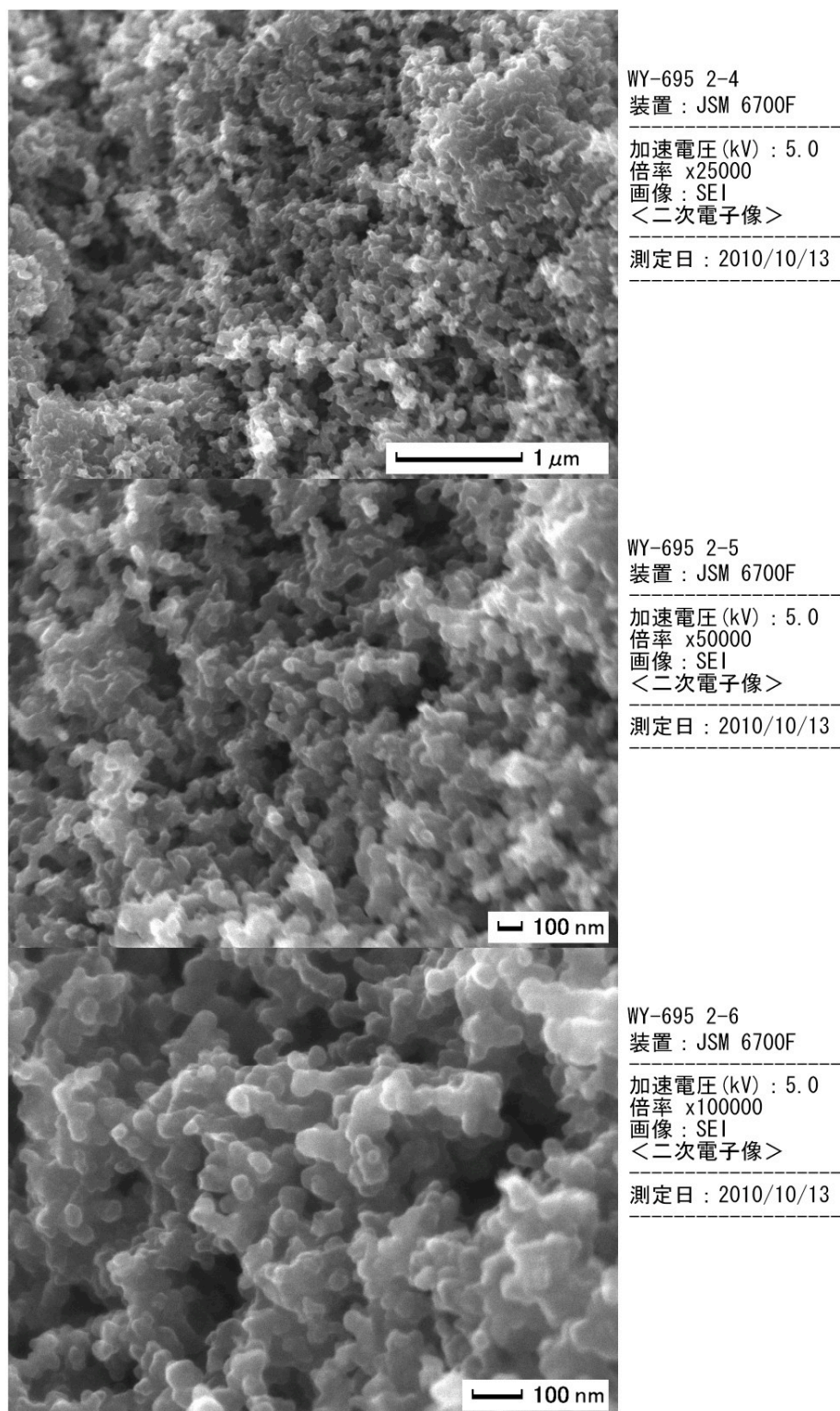


Figure S-6. Typical SEM Image of Ketjen Black EC 300J + Polymer 4

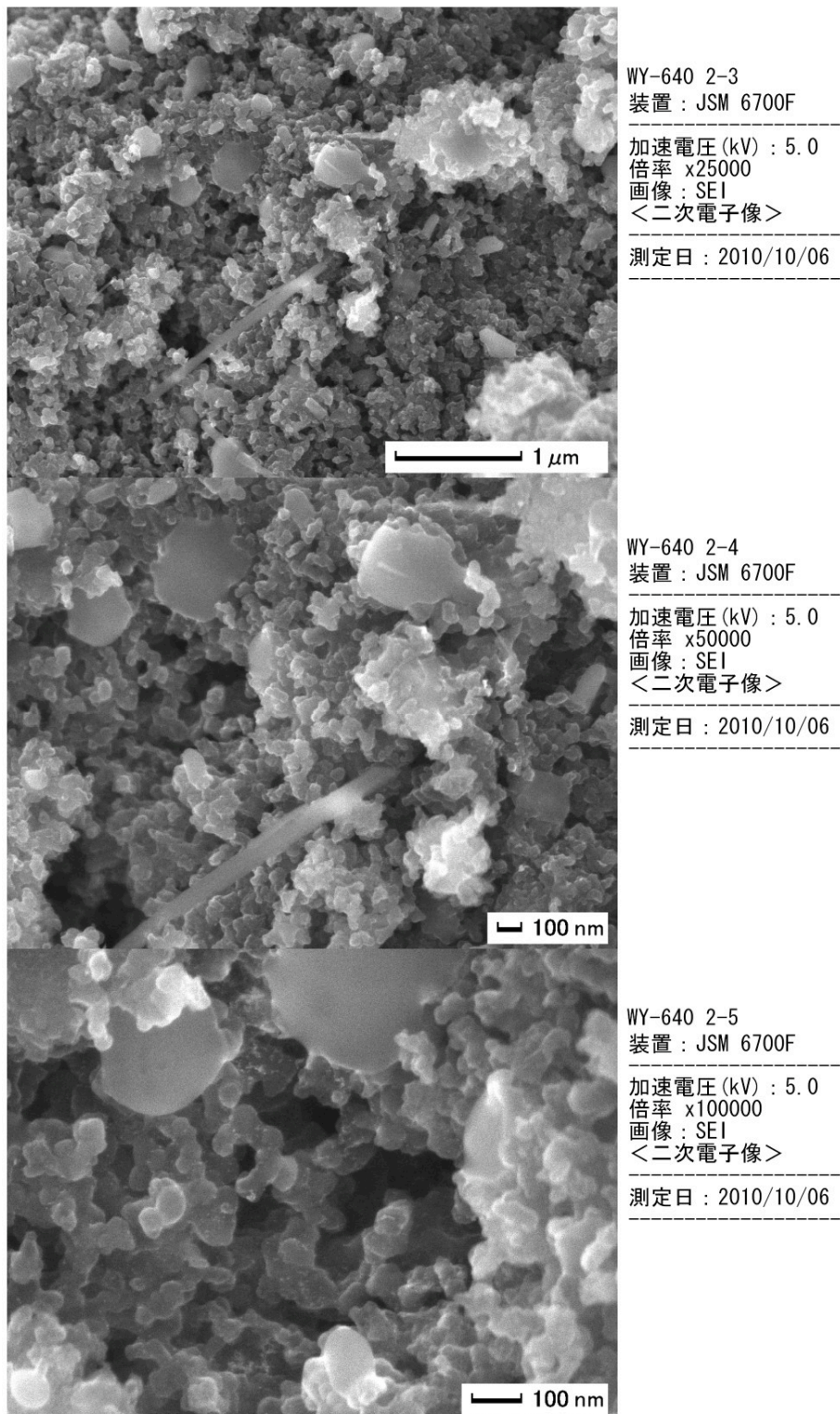


Figure S-7. Typical SEM Image of PI/CB-Au/Pd/B

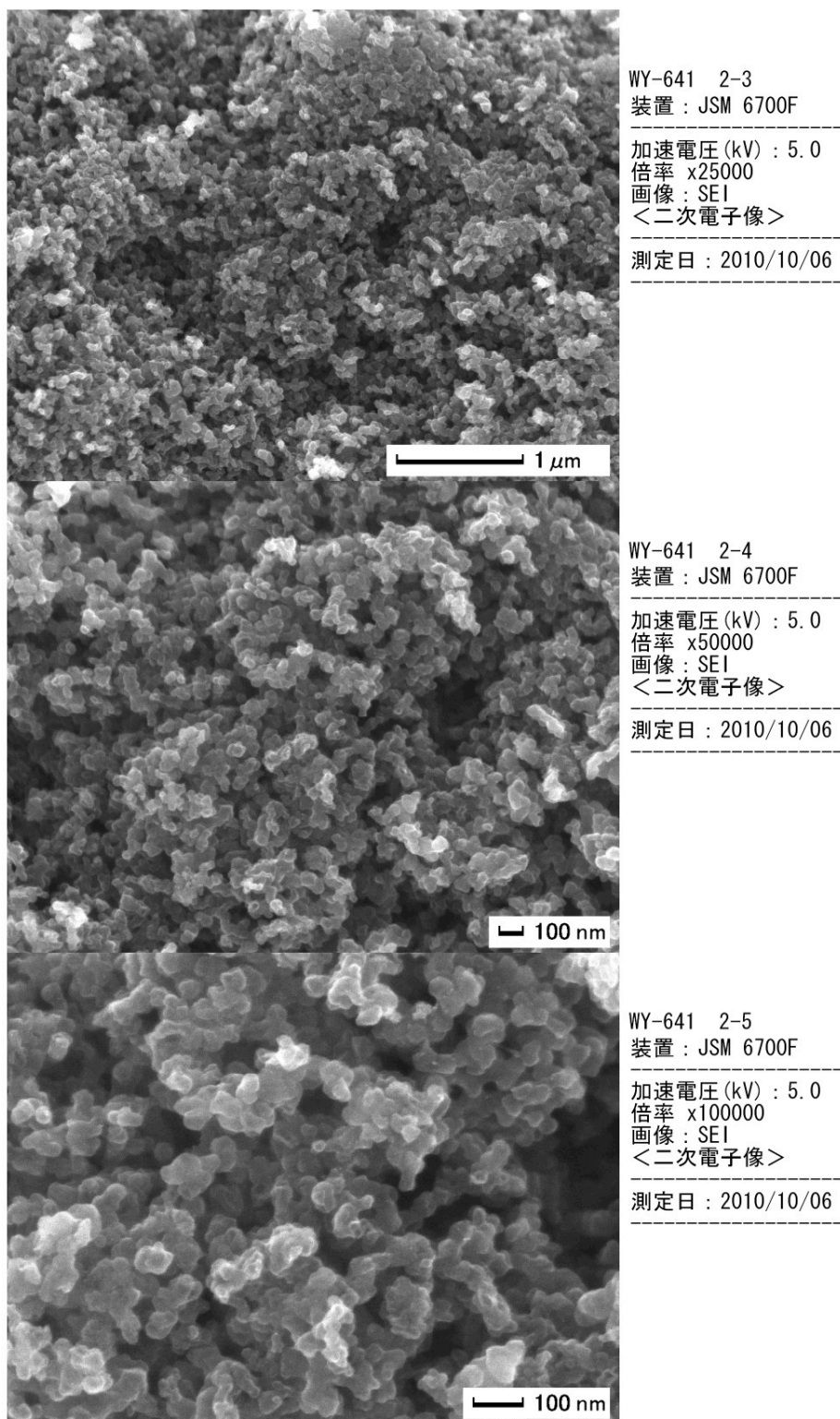
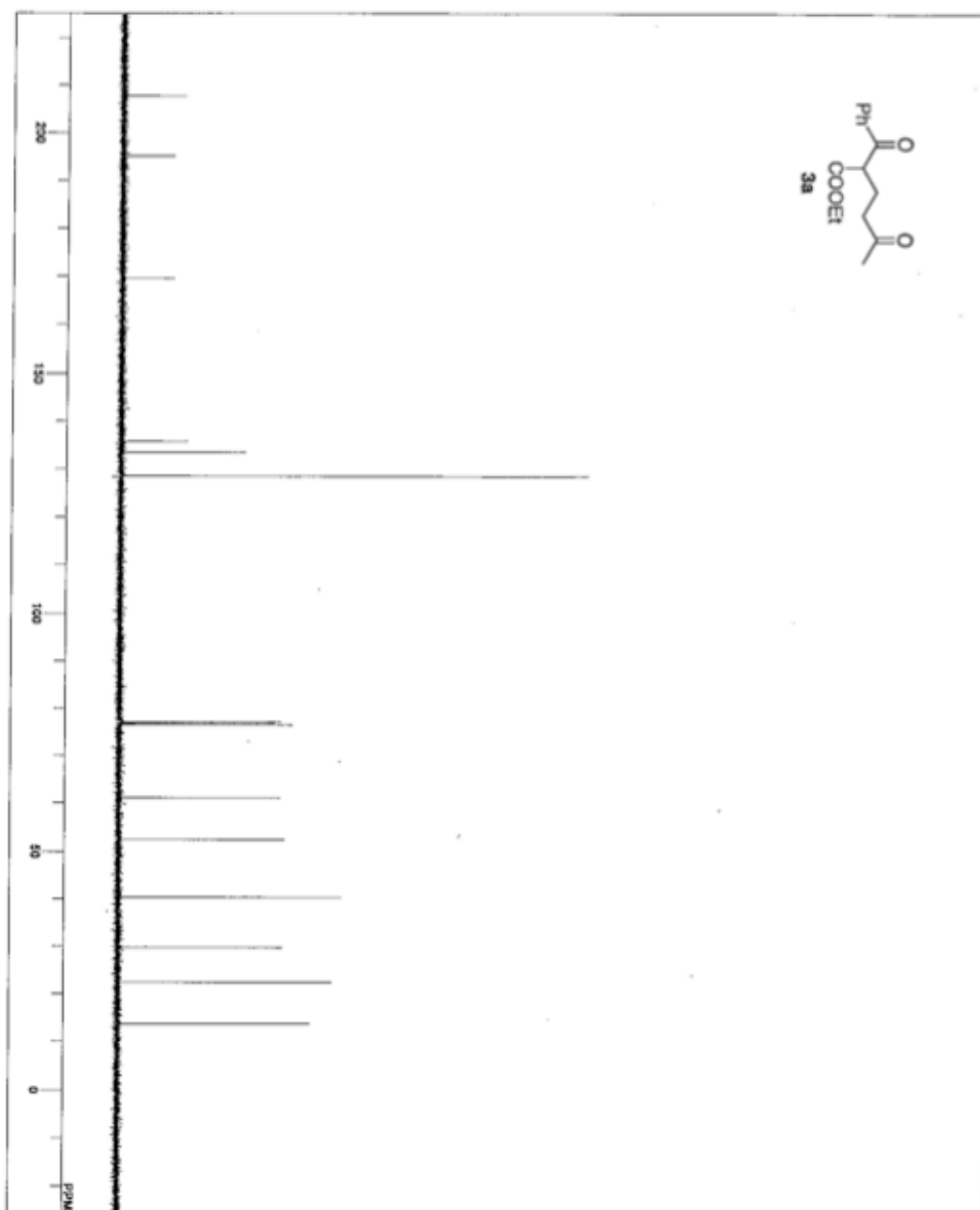
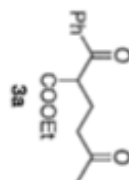
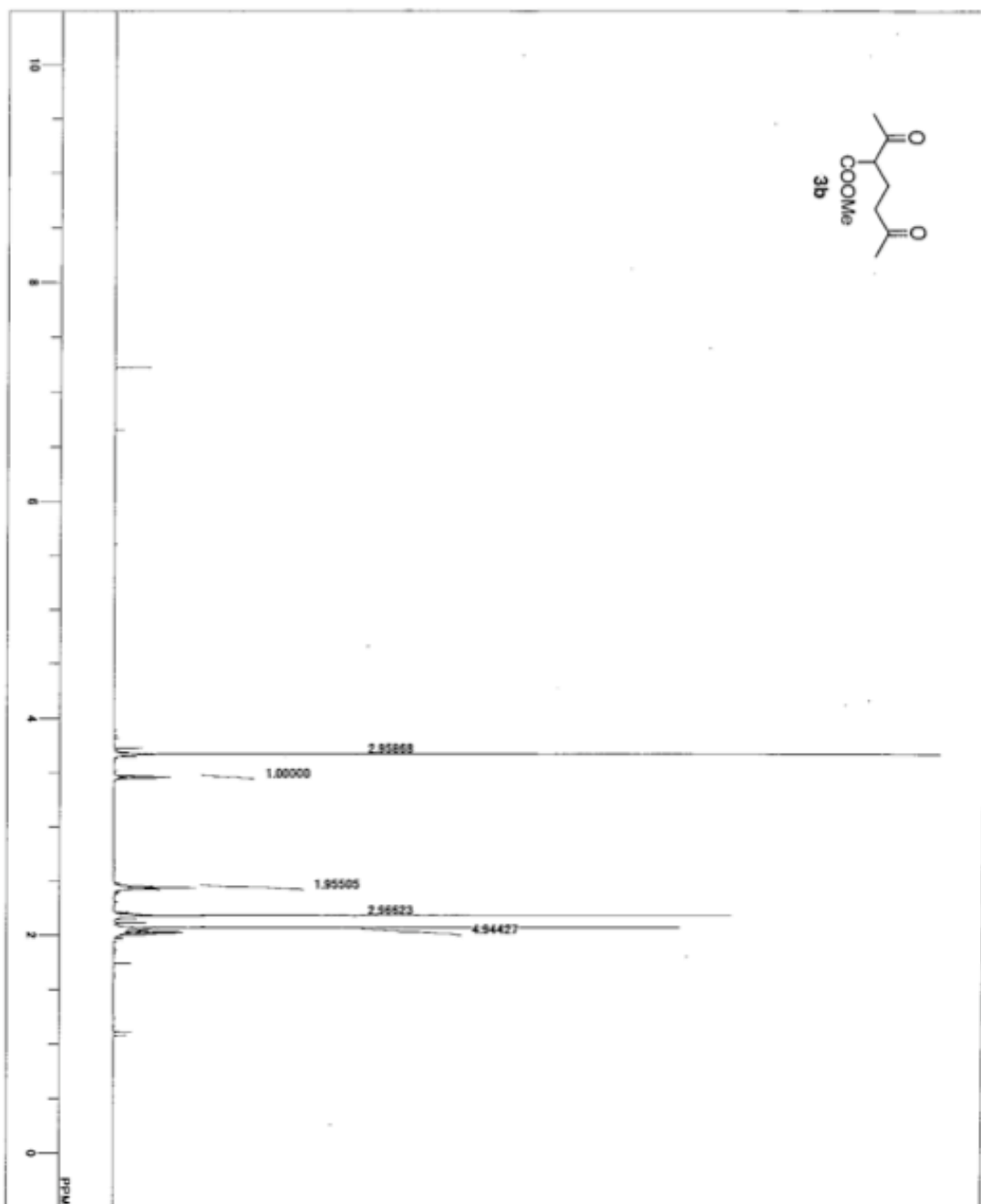
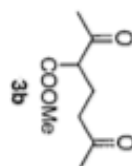


Figure S-8. Typical SEM Image of PI/CB-Au/Pd

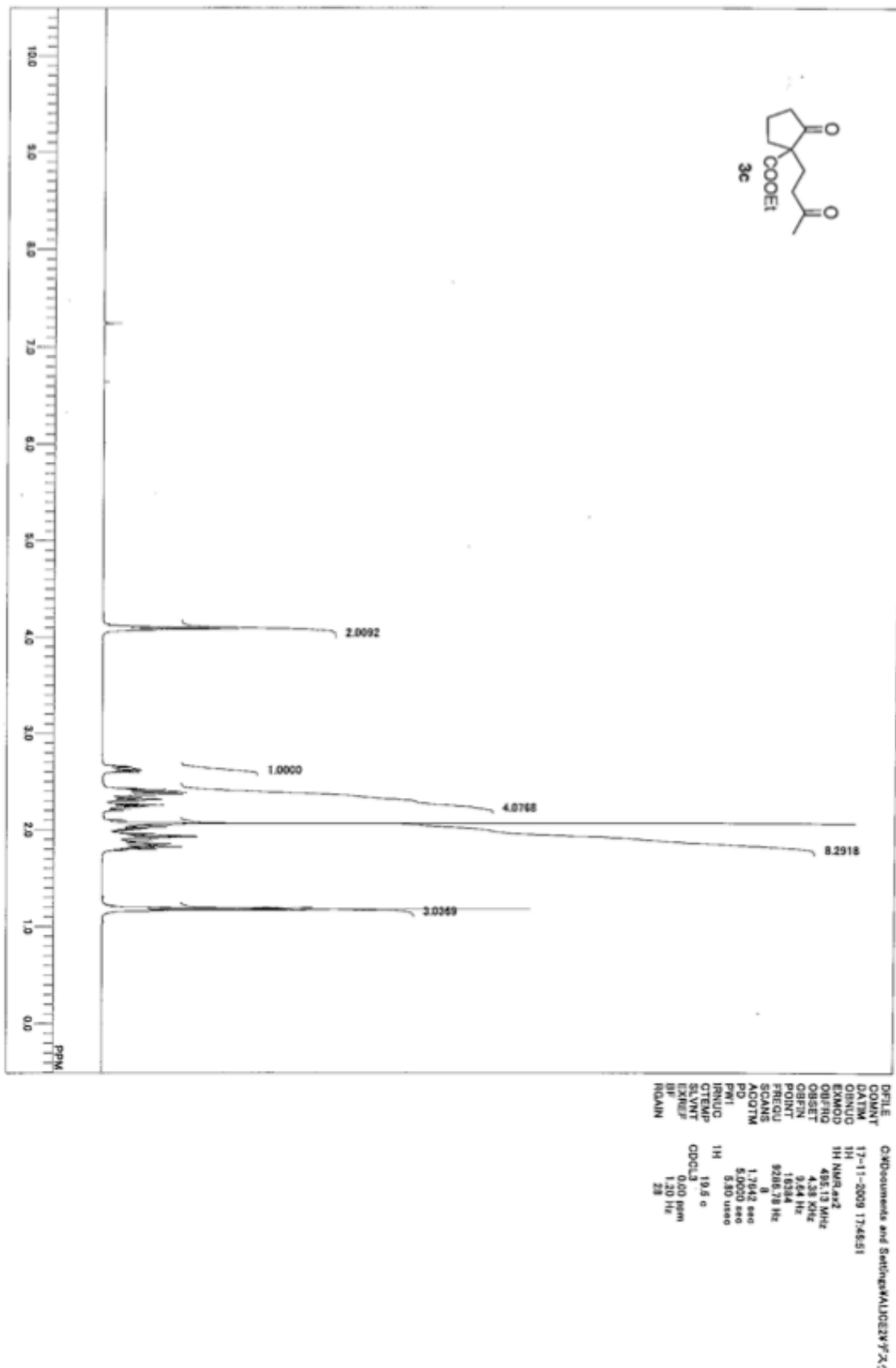
Part IX: ^1H and ^{13}C NMR Spectra of 3a-3q and ^{11}B CP-MAS of PI/CB-B

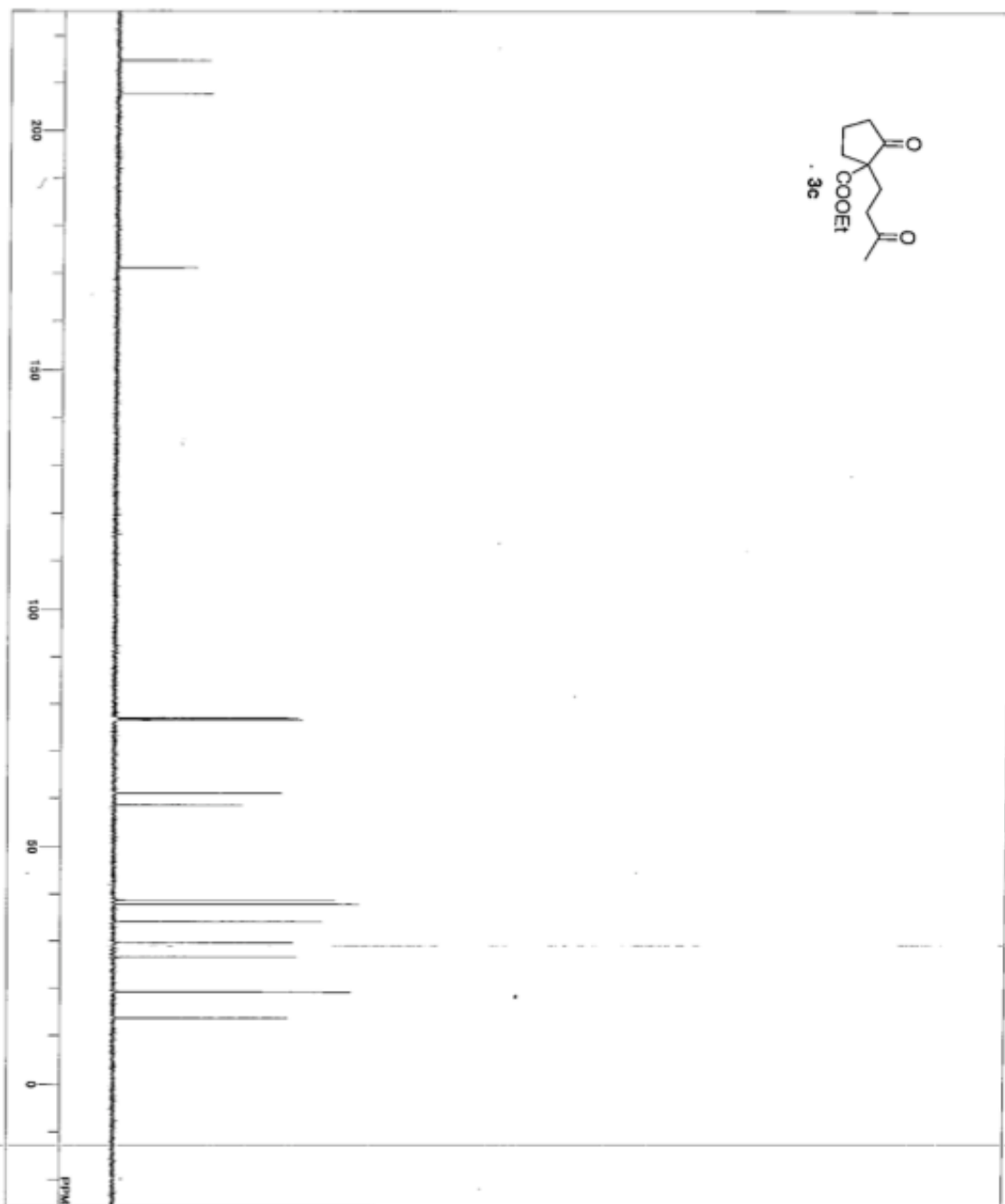
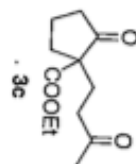


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 EXMUG 130 NMR/42
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 ORFRO 3.45 MHz
 ORFRO 3.45 MHz
 POINT 3000 Hz
 FREQU 3000.00 Hz
 SCANS 32
 ACQTM 0.8339 sec
 PD 2.0000 sec
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 TRNUC 1H
 GTEMP 18.5 o
 ELNPT 71.00 ppm
 ELNPT 71.12 Hz
 RF 48
 ROLAN

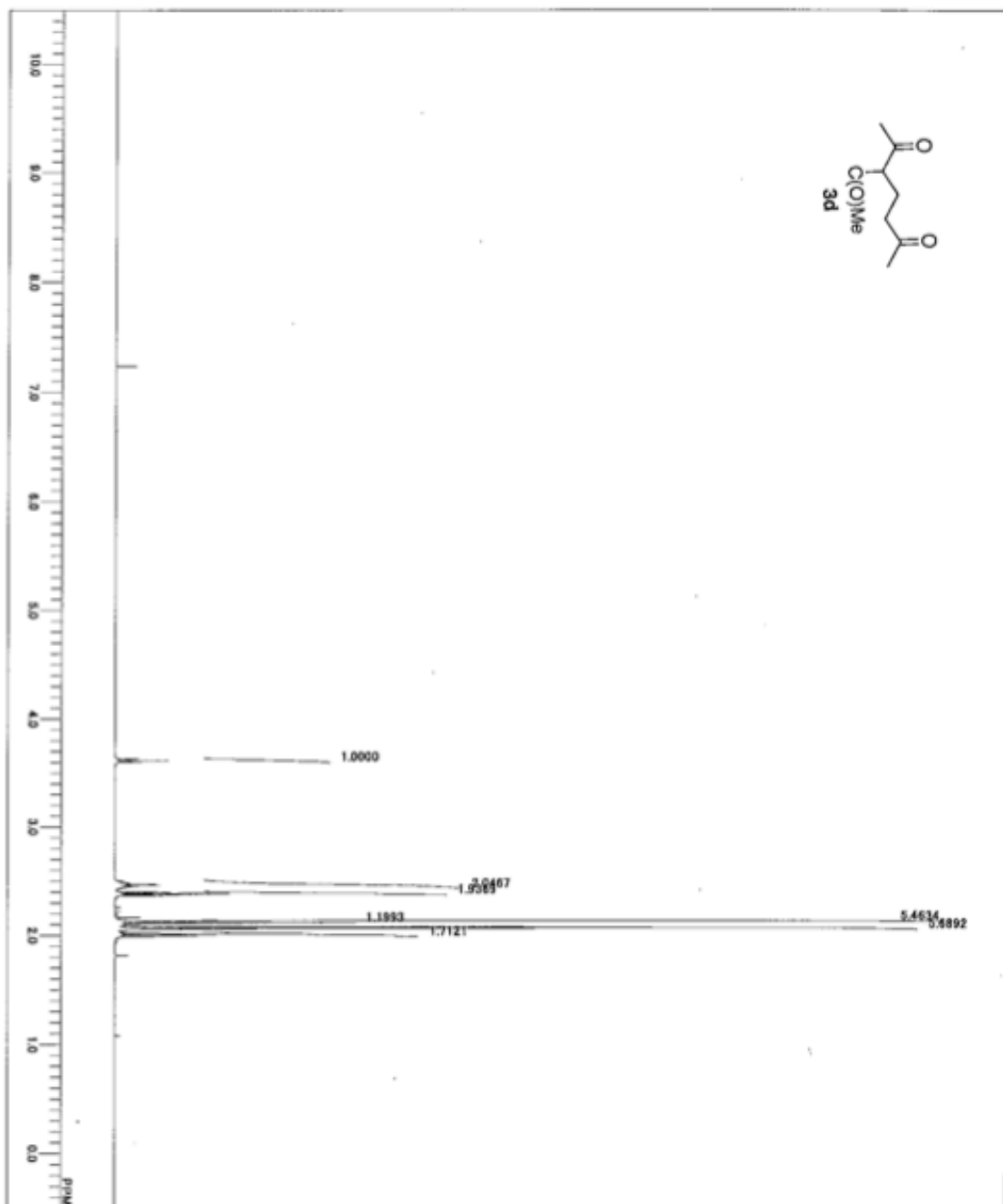
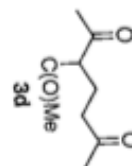


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 PCYCL 13105
 P1 1.7643 sec
 P2 0.0000 sec
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 P4 17.5 e
 INJUG 17.5 e
 CTENP 0.00 ppm
 SLVNT EXREF 0.12 Hz
 BF 34
 RGAIN 34

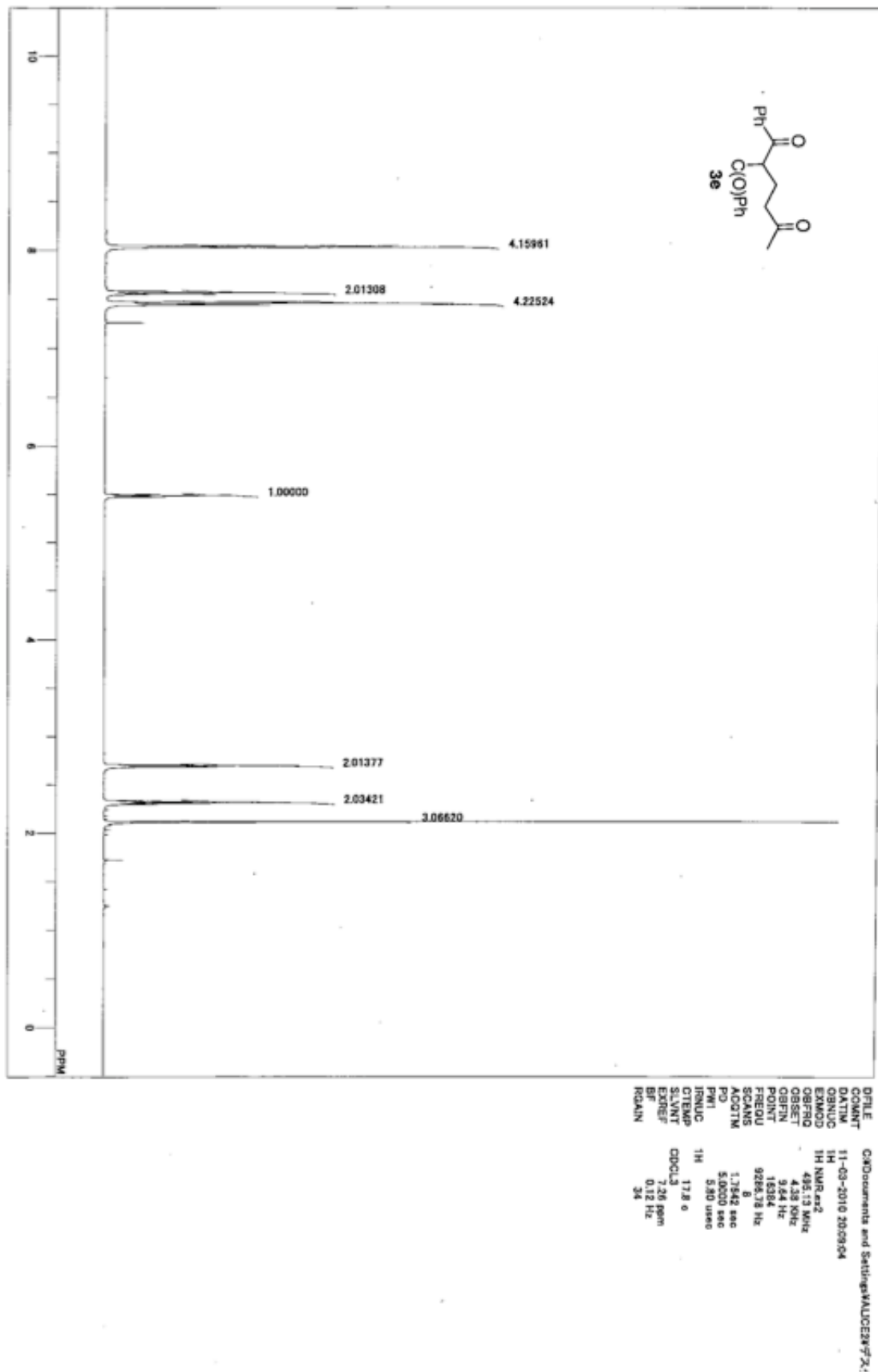


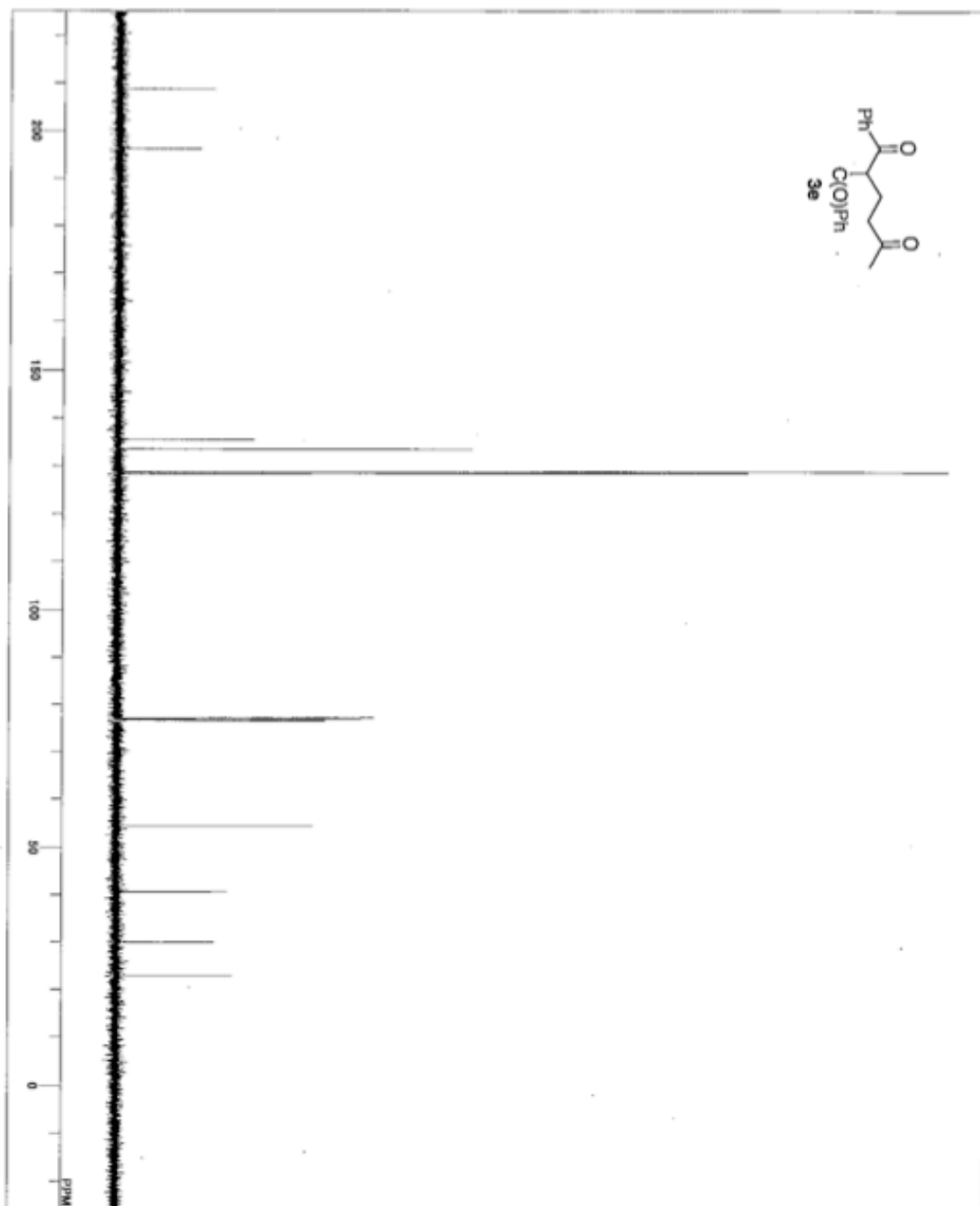
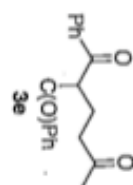


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 EXNO 130 NMR.a2
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 OBSER 164.5 MHz
 OBSER 164.5 MHz
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 POINT 32768
 POINT 32768
 FREQ 39052.50 Hz
 FREQ 39052.50 Hz
 FREQ 39052.50 Hz
 SCANS 98
 SCANS 98
 SCANS 98
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 ACQTM 0.8159 sec
 ACQTM 0.8159 sec
 PD 2.0000 sec
 PD 2.0000 sec
 PD 2.0000 sec
 PWT 3.57 usec
 PWT 3.57 usec
 PWT 3.57 usec
 IRNUC 1H
 IRNUC 1H
 IRNUC 1H
 QTEMP 20.6
 QTEMP 20.6
 QTEMP 20.6
 SOLVT CDCl3
 SOLVT CDCl3
 SOLVT CDCl3
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 EXREF 77.00 ppm
 EXREF 77.00 ppm
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 RNAME 158-130-1
 RNAME 158-130-1

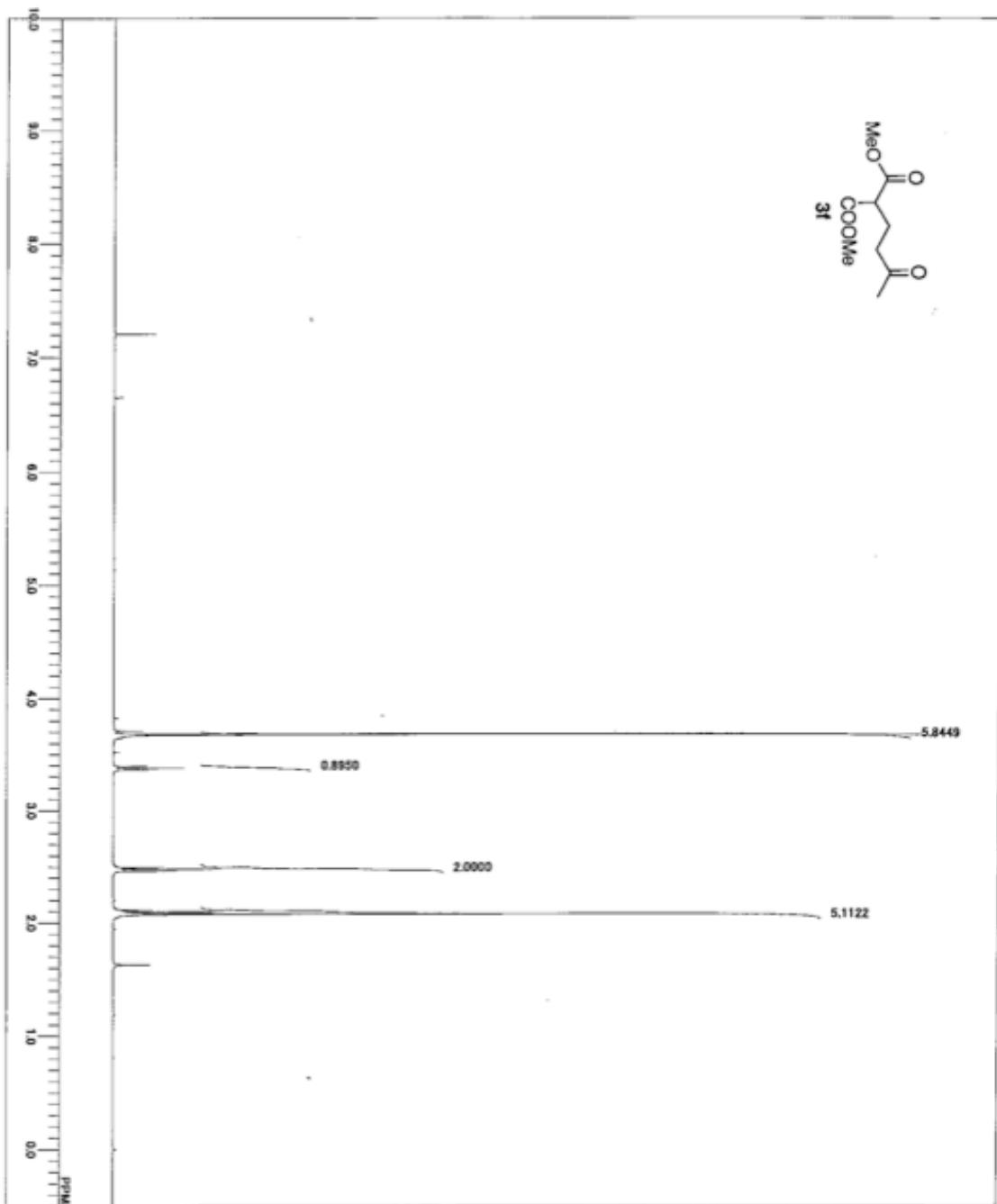


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 EXPNO 2
 PROCNO 2
 F2 - 400.13 MHz
 OBSERV 15384
 POINT 9286.78 Hz
 FREQ 9286.78 Hz
 SCANS 1
 AQC 1.7642 sec
 PD 5.0000 sec
 PWT 5.00 usec
 BRNUC 1H
 CTEMP 20.1 °C
 SOLVENT CDCl₃
 EXREF 0.00 ppm
 BP 0.12 Hz
 RGAIN 35

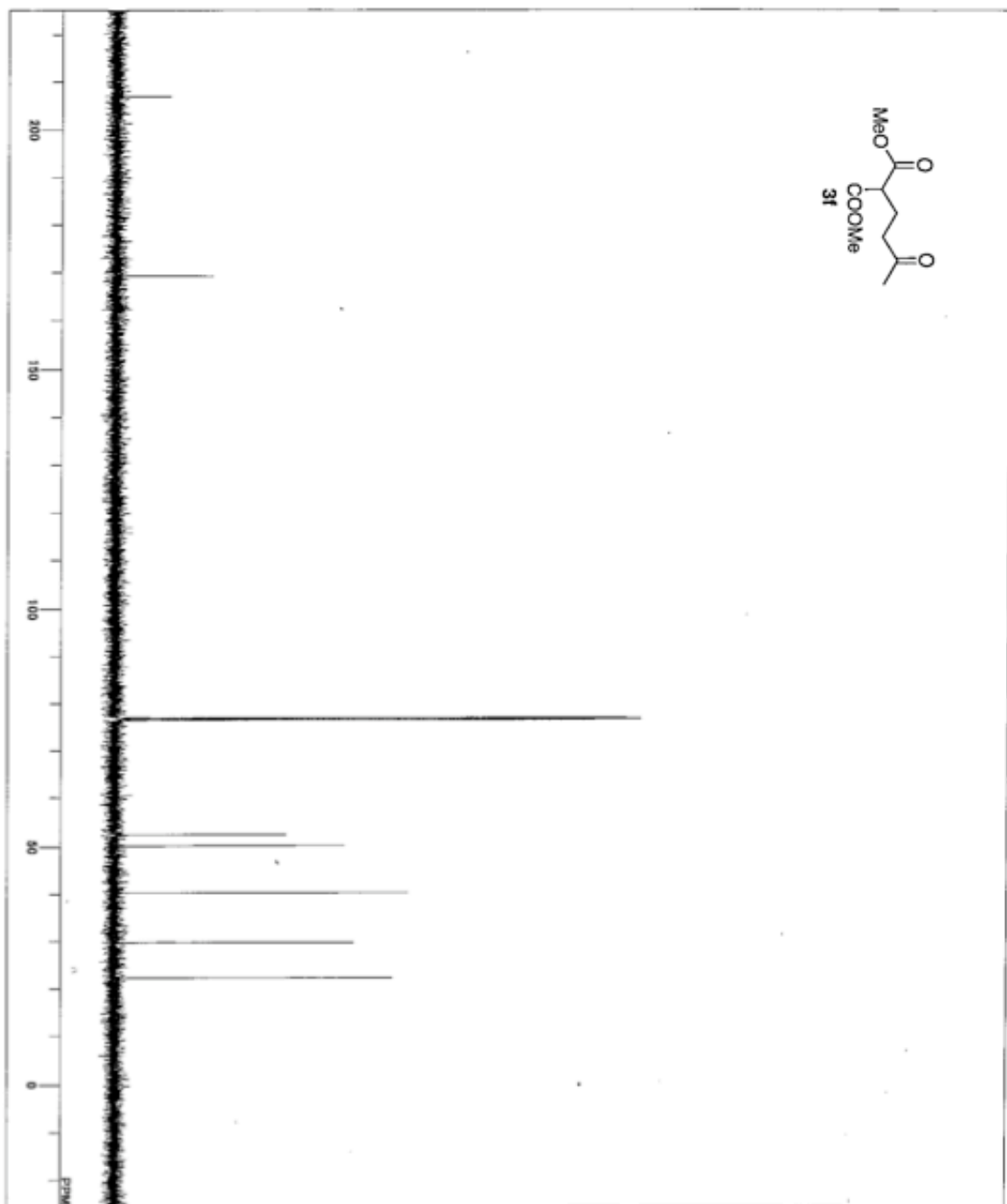
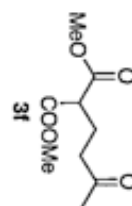




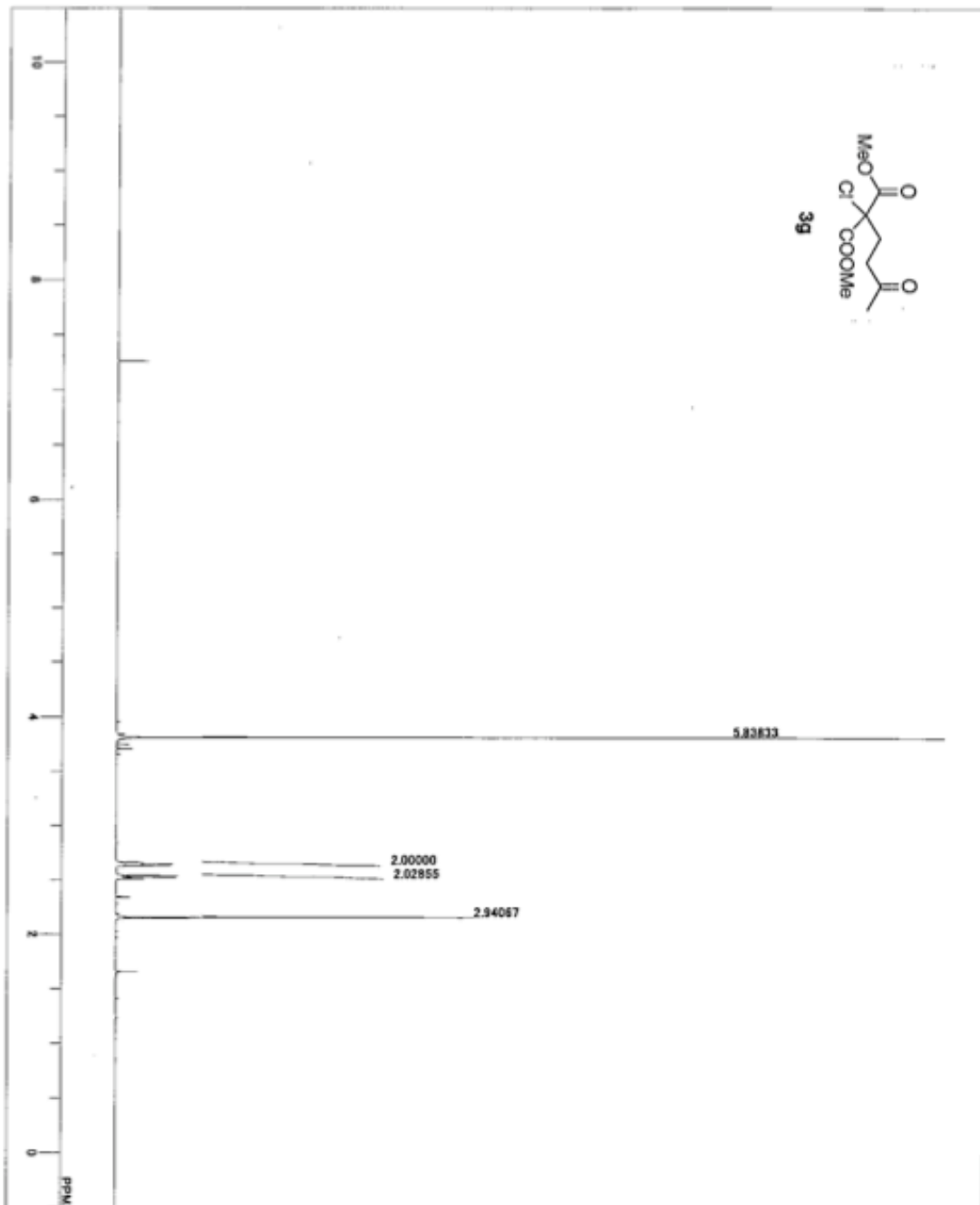
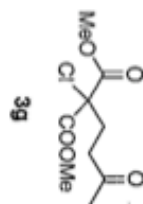
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 SCANS 0.3189 sec
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 CDOL3 77.00 ppm
 SLVNT EXREF 0.12 Hz
 RGAIN 50



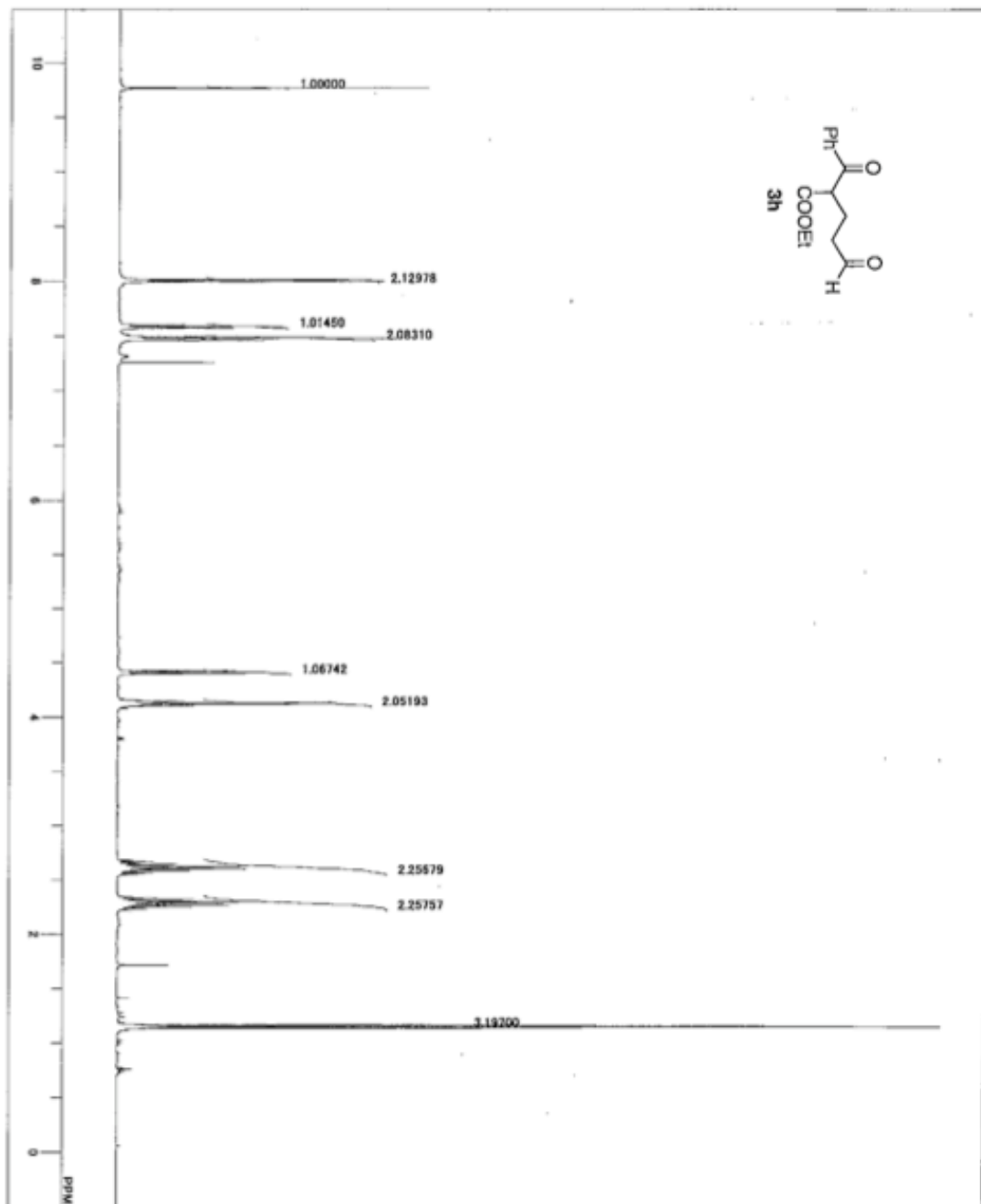
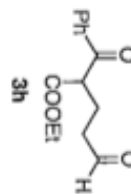
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 EXMOD 1H NMR-42
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 OBSF1N 5.54 Hz
 POINT 16394
 FREQ 9255.78 Hz
 SCANS 4
 ACQTM 1.745 sec
 PUL 3.000 sec
 PD 3.80 dB
 INJUD 1H 18.9 °
 OTEMP CDCL3
 SLVNT 0.00 ppm
 EXREF 0.12 Hz
 BF 40
 RGAIN



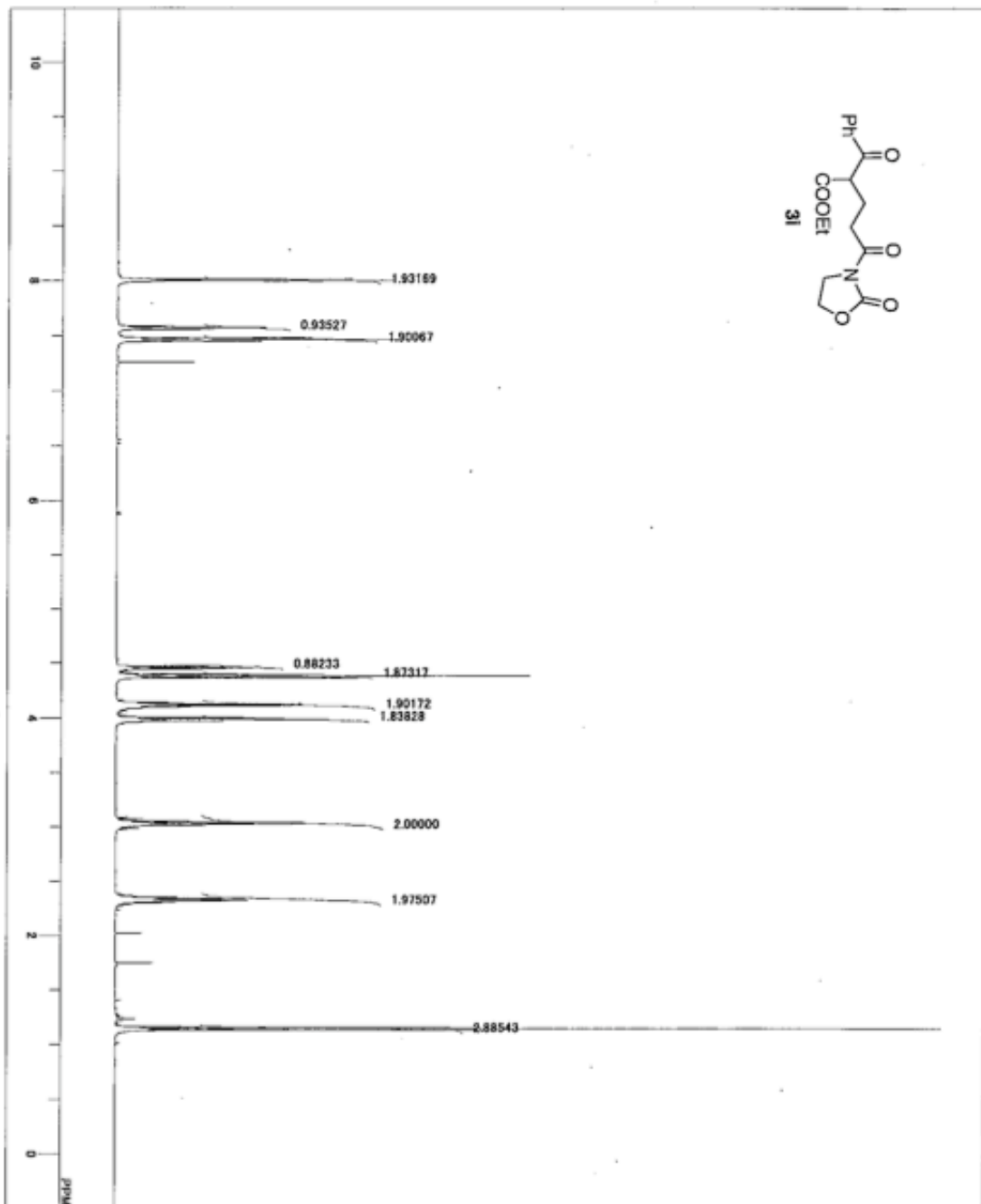
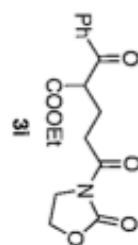
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 EXPOD 124.51 MHz
 OBSFQ 124.51 MHz
 OBSF 1.45 MHz
 QBPIN 8.00 Hz
 POINT 32768
 FREQ 38062.50 Hz
 SCANS 128
 ACQTM 0.8389 sec
 PD 2.0000 sec
 PW 3.67 sec
 IRNUC 1H
 CTMPP 20.6 s
 STVNT CDCL3
 EXREF 77.00 ppm
 BR 0.12 Hz
 RGAIN 50



D:\FILE C:\Documents and Settings\ALJOEY\FX 57-7
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 DATE 1H
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 EXMID 400.13 MHz
 OBSCT 16384 Hz
 OBSCT 16384 Hz
 POINT 9228.78 Hz
 POINT 9228.78 Hz
 SCANS 8
 ADOITM 1.7642 sec
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 IRNUC 1H
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 SLVNT CDCl3
 EXREF 7.26 ppm
 BP 0.12 Hz
 RGAIN 38

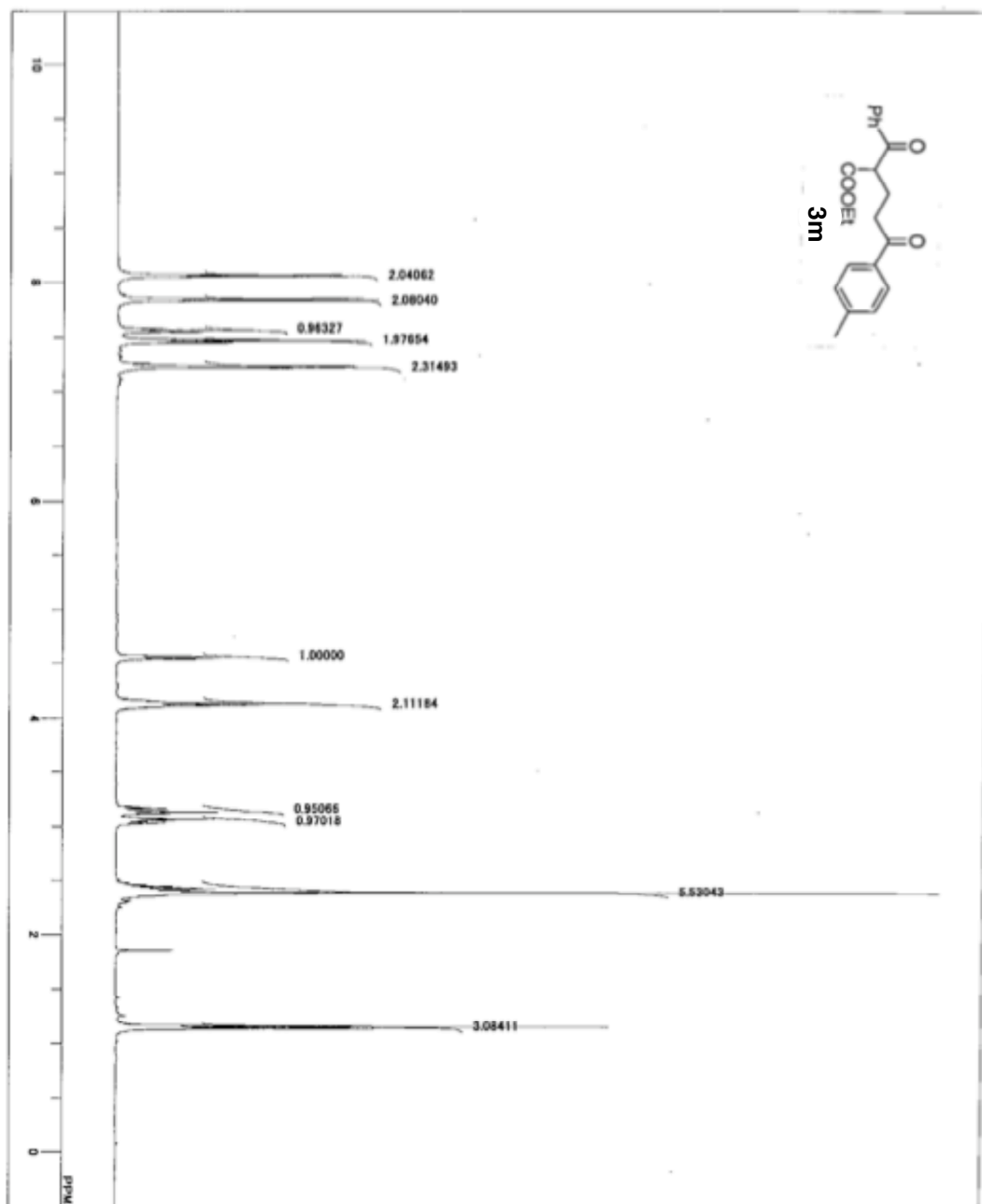


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 OBSER 13 C
 OBSER 13 C
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 PWT 5.30 usec
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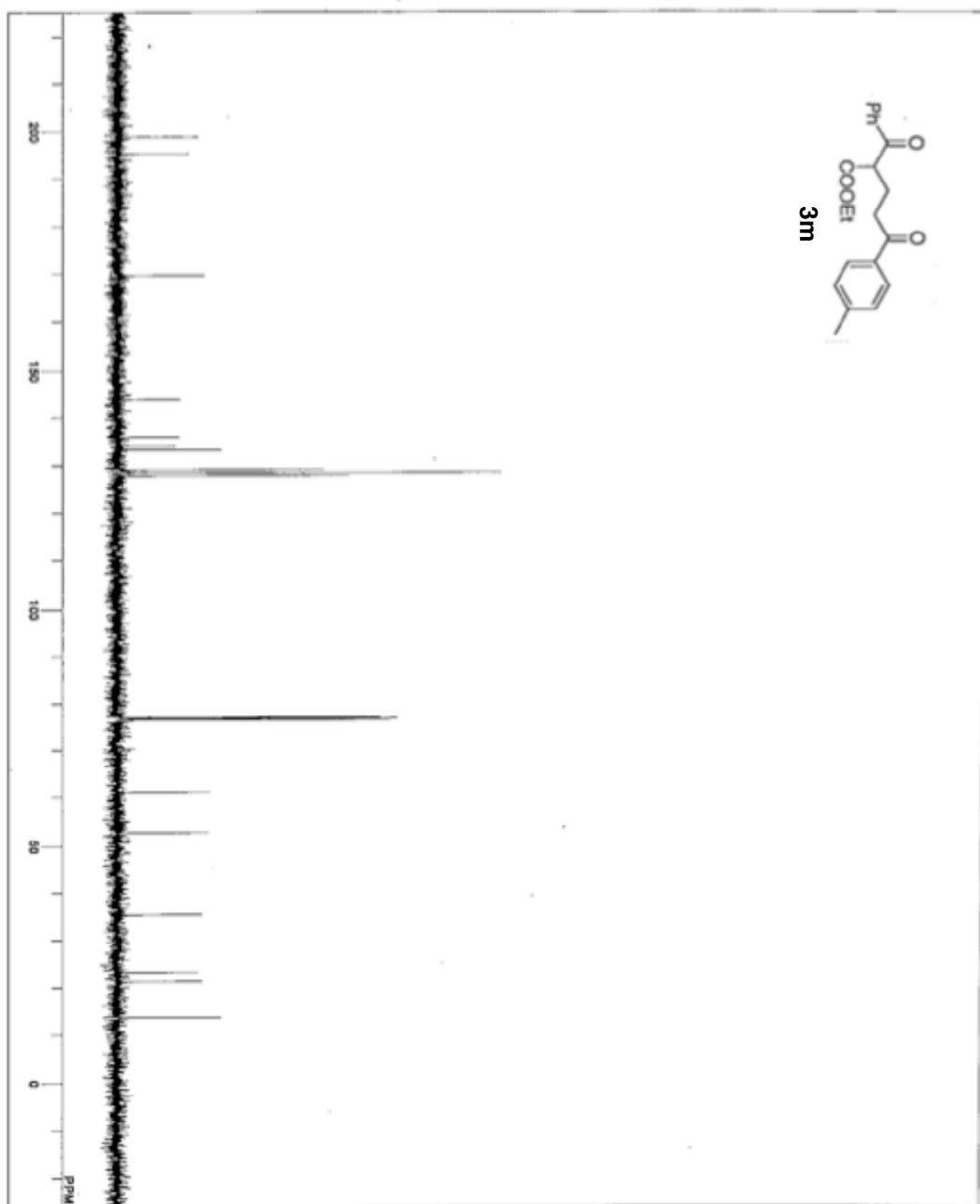
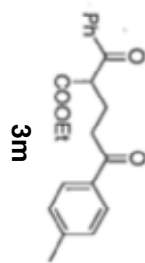


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 POFN 16384
 POINT 9286.78 Hz
 FREQU 9286.78 Hz
 SCANS 8
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 PD 5.0000 sec
 PWT 5.80 used
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 BP 34
 ROAIN

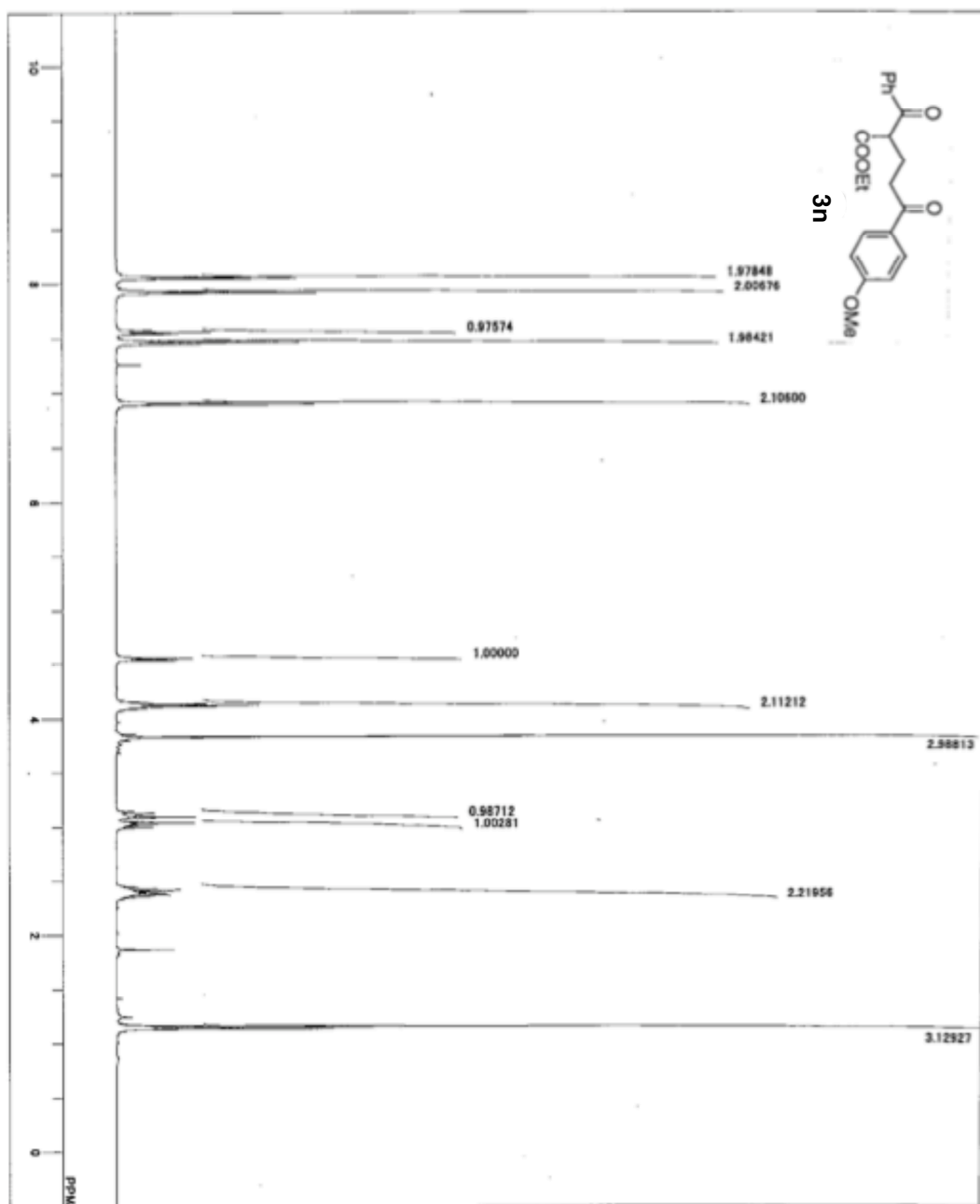


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 OFSET 18.64 Hz
 OFPIN 18.64 Hz
 OFSQU 9876.78 Hz
 SCANS 1
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 PD 5.0000 sec
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 RGAN 30

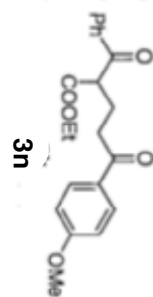
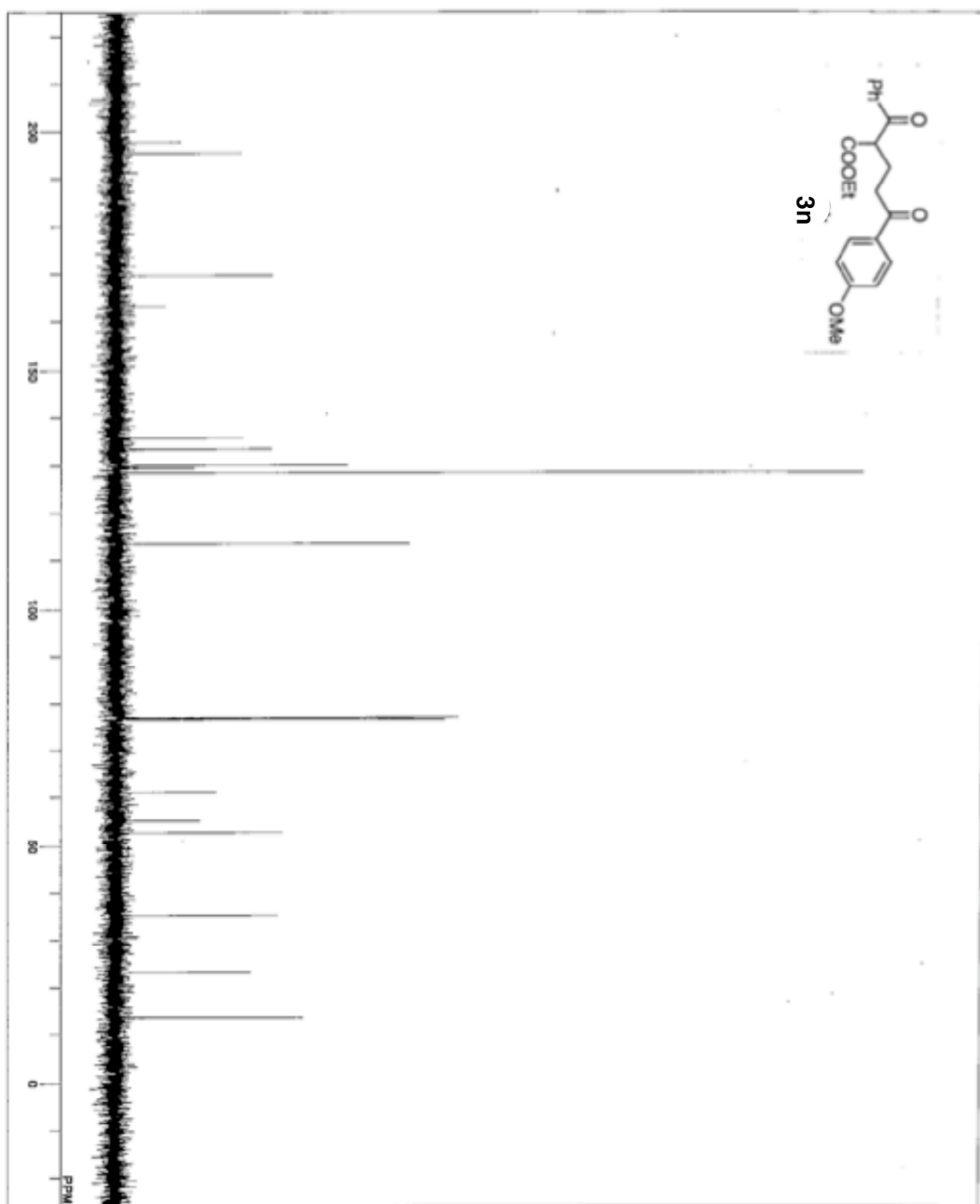


1H NMR (400 MHz, CDCl₃)
 7.3-7.1 (m, 5H, aromatic protons)
 6.8-6.5 (m, 5H, aromatic protons)
 6.2-6.0 (m, 5H, aromatic protons)
 5.8-5.5 (m, 5H, aromatic protons)
 5.2-5.0 (m, 5H, aromatic protons)
 4.8-4.5 (m, 5H, aromatic protons)
 4.2-4.0 (m, 5H, aromatic protons)
 3.8-3.5 (m, 5H, aromatic protons)
 3.2-3.0 (m, 5H, aromatic protons)
 2.8-2.5 (m, 5H, aromatic protons)
 2.2-2.0 (m, 5H, aromatic protons)
 1.8-1.5 (m, 5H, aromatic protons)
 1.2-0.8 (m, 5H, aromatic protons)
 0.5-0.2 (m, 5H, aromatic protons)

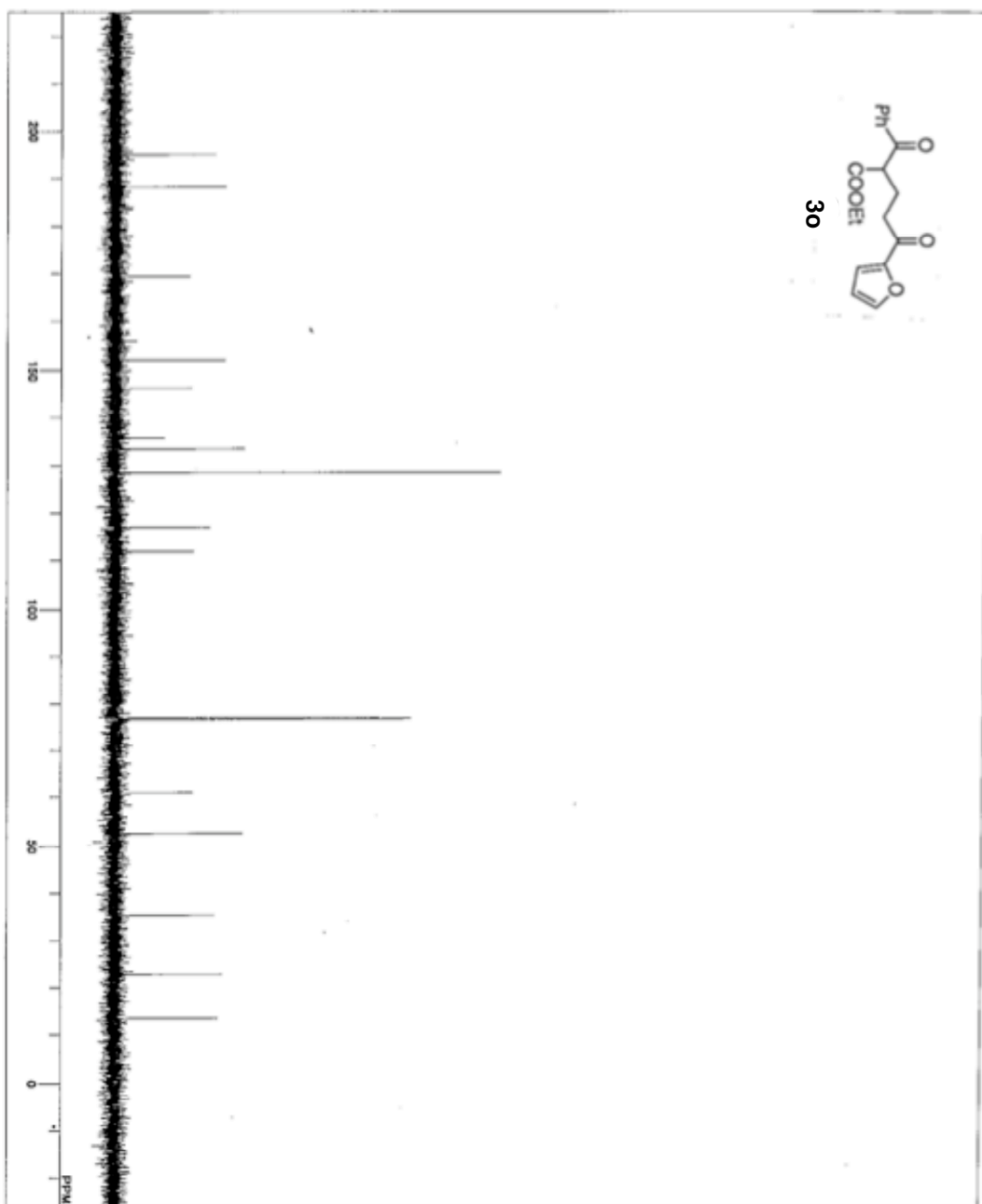
Chemicals and Biologicals/2010-10-10/10-10-10

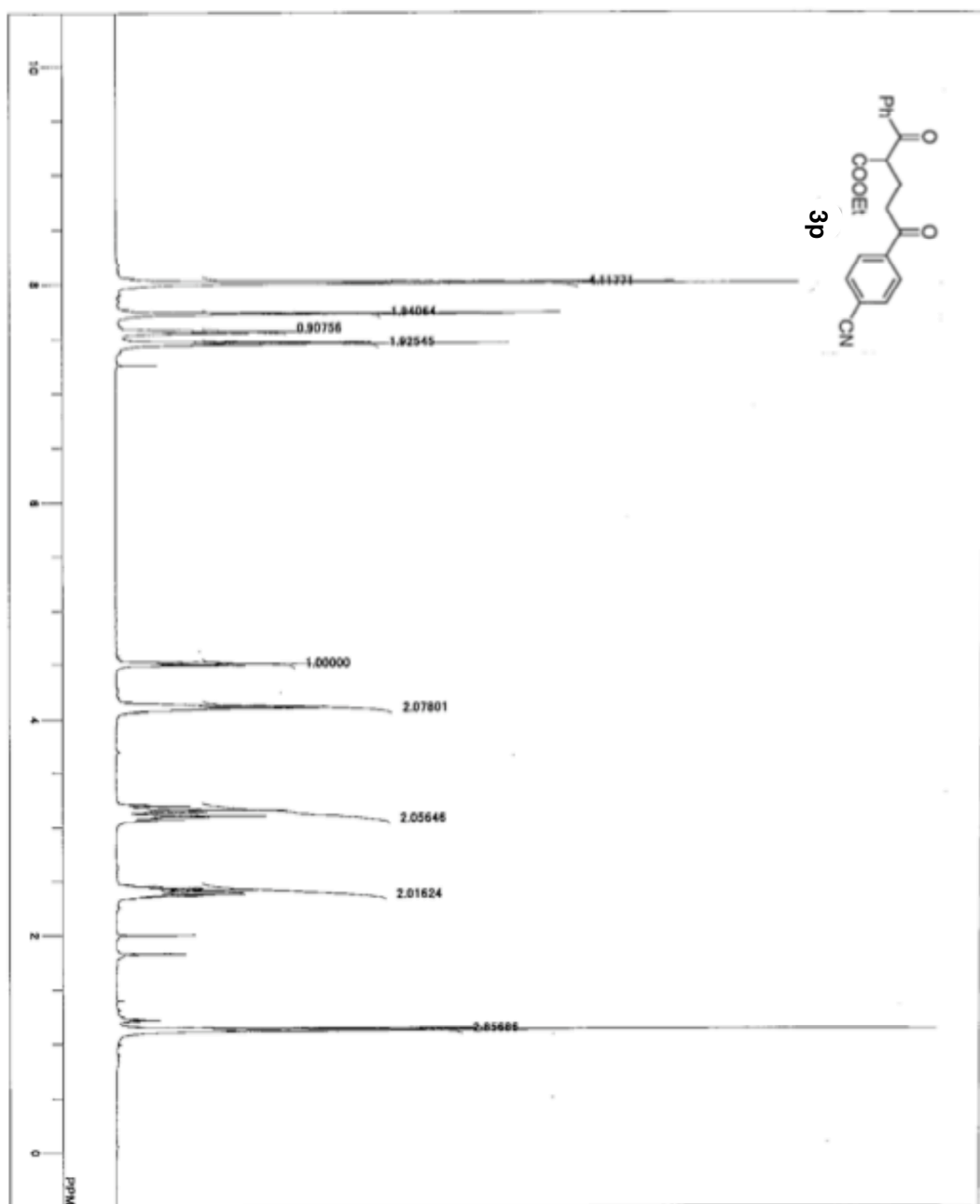


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 ACQTM: 1.00000
 SOLVT: CDCl3
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 RNAME: 3n

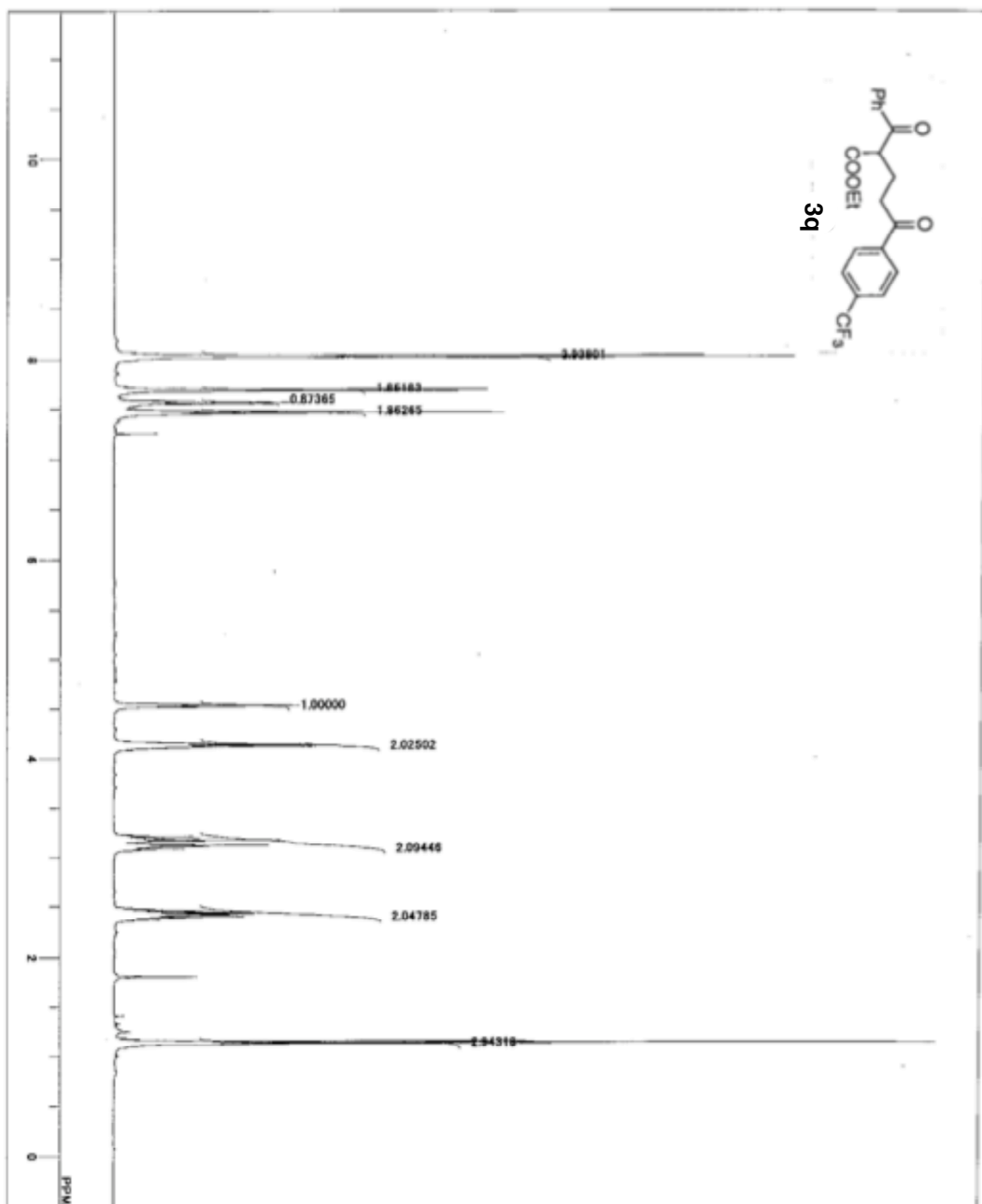


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 OFFSET: 3.45 MHz
 CHIN: 4.00 Hz
 POINT: 32768
 FREQ: 39942.56 Hz
 ACQ: 7.7 sec
 PD: 0.1332 sec
 PC: 2.0000 sec
 PWT: 3.67 sec
 INJ: 26.0 s
 QTEP: CDCL3
 SLVT: 71.00 ppm
 EXREF: 0.12 Hz
 BP: 60
 NUC1: 13C

[illegible]



NAME: C:\Documents and Settings\KALICEY\My Documents\11-1-11\11
 COUNT: 22-07-2010 18:45:52
 DATE: 22-07-2010 18:45:52
 INSTR: 400 MHz
 EXMOD: 400 MHz
 ORBIT: 4.38 Hz
 ORBIT: 9.64 Hz
 POINT: 10234
 POINT: 8281.78 Hz
 POINT: 1.7642 sec
 POINT: 5.0050 sec
 POINT: 5.00 sec
 NAME: 1H
 INSTR: 283.5 e
 EXMOD: 7.28 ppm
 ORBIT: 1.20 Hz
 POINT: 30



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 NAME 72MW-403-1H-1M1
 DATE 21-07-2010 18:43:37
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 PROCNO 1
 F2 400.13 MHz
 F1 100.62 MHz
 AQ 1.7642 sec
 ACQTM 5.0000 sec
 PD 5.80 used
 PWT 1H
 INJLOC 25.3 g
 OTEMP 128 rpm
 SLEW 120 Hz
 EXRF 30
 NSCAN 30

Part X: References

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