Supplementary Information
For

Electrochemical Esterification Reaction of Alkynes with Diols via Cleavage of Carbon-Carbon Triple Bonds without Catalyst and Oxidant

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Tabel of Contents

I. General Information S2

II. Experimental procedures and data S3

1.General procedure for the electrochemical esterification reaction of alkynes with diols S3

2.General procedure for cyclic voltammetry (CV) S3

3.Characterization data for the products S3

4. X-Ray crystal data of **3j** S13

III. References S24

NMR spectra of the products S25

I. General Information

NMR spectra were recorded on Bruker-600 (600 MHz for ^{1}H; 151 MHz for ^{13}C). ^{1}H NMR spectra were referenced relative to internal $Si(Me)_4$ (TMS) at δ 0.00 ppm or $CDCl_3$ at δ 7.26 ppm. ^{13}C NMR spectra were recorded at ambient temperature on Bruker-600 (151 MHz) spectrometers and are referenced relative to $CDCl_3$ at δ 77.16 ppm. Data for ^{1}H, ^{13}C NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet, q = quartet, quint = quintet, br = broad), integration, and coupling constant (Hz). High resolution mass spectra were recorded on P-SIMS-Gly of BrukerDaltonics Inc. using ESI-TOF (electrospray ionization-time of flight). *n*-Bu_4NPF_6 was purchased from Bidepharm, and MeCN was purchased from Shanghai Titan Scientific Co., Ltd. Diphenylacetylene, prop-1-yn-1-ylbenzene and all the terminal alkyns were purchased from Energy Chemical. 1-Ethyl-4-(p-tolylethynyl)benzene, but-1-yn-1-ylbenzene, pent-1-yn-1-ylbenzene were purchased from Alfa Aesar. Hex-1-yn-1-ylbenzene was purchased from Shanghai Macklin Biochemical Co., Ltd. Other alkynes were synthesized through the known methods.[1-12] Ethylene glycol was purchased from Energy Chemical. 1,3-Propanediol and 1,4-butanediol were purchased from Shanghai Aladin Biochemical Technology Co., Ltd. 2,2-Dimethyl-1,3-propanediol was purchased from Shanghai Macklin Biochemical Co., Ltd. Diethylene glycol and diethylene glycol monomethyl ether were purchased from Tianjin Heowns Biochemical Technology Co. Ltd.

IV. Experimental procedures and data

1. General procedure for the electrochemical esterification reaction of alkynes with diols

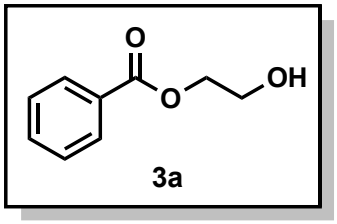
To an undivided cell (10 mL columnar round-bottom flask with a 24# mouth) fitted with a graphite rod anode (6 mm diameter, 120mm length) and a piece of Pt (1 cm × 1cm × 0.02 cm) cathode, the solid reagents alkyne (0.6 mmol) , *n*-Bu_4NPF_6 (0.6 mmol) were added. The vessel was evacuated and backfilled with N_2 (3 times). Then, the liquid reagents diol (27 mmol), MeCN (5 mL) were added in sequence via syringe. The electrolysis was carried out with constant current (18 mA) at 80 °C for 12 h. After that, the reaction mixture was cooled to room temperature and filtered through a plug of silica (eluted with 80 mL EtOAc). The filtrate was washed with water for two times (2 × 80 mL). Then the organic layer was washed with brine and dried over Na_2SO_4. The solvent was evaporated to dryness under reduced pressure and the residue was purified by column chromatography on silica gel to give product **3**.

2. General procedure for cyclic voltammetry (CV):

Cyclic voltammetry was performed in a three electrode cell connected to a schlenk line at room temperature. The working electrode was a glassy carbon electrode, and the counter electrode was a platinum electrode. The reference was an Ag wire. 10 mL of CH_3CN containing 0.1 M *n*-Bu_4NPF_6 were poured into the electrochemical cell in all experiments. The scan rate is 0.1 V/s, ranging from 0 V to 3 V. The test concentrations of **1a**, **2a** and **1j** are 1 mM.

3. Characterization data for the products

2-hydroxyethyl benzoate (**3a**)



3a

Following general procedure, alkyne **1a** and alcohol **2a** were used to afford the desired product (151.5 mg, 76% yield).

Alkyne **1k** and alcohol **2a** were used to afford the desired product (51.2 mg, 51% yield).

Alkyne **1l** and alcohol **2a** were used to afford the desired product (56.0 mg, 56% yield).

Alkyne **1m** and alcohol **2a** were used to afford the desired product (62.7 mg, 63% yield).

Alkyne **1n** and alcohol **2a** were used to afford the desired product (60.3 mg, 60% yield).

Alkyne **1o** and alcohol **2a** were used to afford the desired product (61.5 mg, 62% yield).

Alkyne **1p** and alcohol **2a** were used to afford the desired product (71.2 mg, 71% yield).

Alkyne **1q** and alcohol **2a** were used to afford the desired product (64.3 mg, 65% yield).

Alkyne **1r** and alcohol **2a** were used to afford the desired product (60.1 mg, 60% yield).

Alkyne **4a** and alcohol **2a** were used to afford the desired product (48.6 mg, 49% yield).

Alkyne **4i** and alcohol **2a** were used to afford the desired product (52mg, 52% yield).

Alkyne **4j** and alcohol **2a** were used to afford the desired product (58.5 mg, 59% yield).

Alkyne **4k** and alcohol **2a** were used to afford the desired product (47.7 mg, 48% yield).

Alkyne **5a** and alcohol **2a** were used to afford the desired product (29.2 mg, 29% yield).

2-Hydroxy-1,2-diphenylethanone **7** and alcohol **2a** were used to afford the desired product (86.3 mg, 43% yield).

Benzil **8** and alcohol **2a** were used to afford the desired product (84.3 mg, 43% yield).

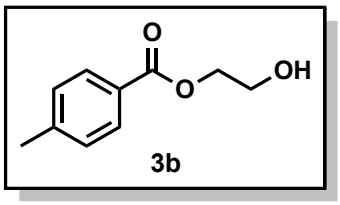
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06-8.05 (m, 2H), 7.58-7.55 (m, 1H), 7.45-7.43 (m, 2H), 4.47-4,45 (m, 2H), 3.96-3.95 (m, 2H), 2.05 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.1, 133.3, 130.0, 129.8, 128.6, 66.8, 61.5.

HRMS (ESI) calcd. for $C_9H_{10}NaO_3^+$ ($[M+Na]^+$): 189.0522, found: 189.0526.

2-hydroxyethyl 4-methylbenzoate (**3b**)



Following general procedure, alkyne **1b** and alcohol **2a** were used to afford the desired product (109.1 mg, 50% yield).

Alkyne **1k** and alcohol **2a** were used to afford the desired product (53.9 mg, 50% yield).

Alkyne **1s** and alcohol **2a** were used to afford the desired product (54.9 mg, 51% yield).

Alkyne **1t** and alcohol **2a** were used to afford the desired product (56.5 mg, 52% yield).

Alkyne **4b** and alcohol **2a** were used to afford the desired product (63 mg, 58% yield).

Alkyne **5b** and alcohol **2a** were used to afford the desired product (49.8 mg, 46% yield).

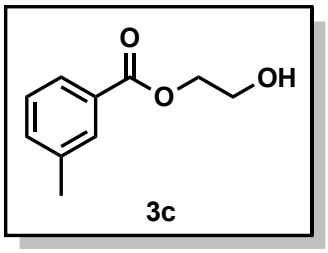
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95 (d, *J* = 8.2 Hz, 2H), 7.24 (d, *J* = 7.9 Hz, 2H), 4.46-4.44 (m, 2H), 3.96-3.95 (m, 2H), 2.41 (s, 3H), 2.10 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.2, 144.1, 129.9, 129.3, 127.2, 66.7, 61.7, 21.8.

HRMS (ESI) calcd. for $C_{10}H_{12}NaO_3^+$ ([M+Na]$^+$): 203.0679, found: 203.0683.

2-hydroxyethyl 3-methylbenzoate (**3c**)



Following general procedure, alkyne **1c** and alcohol **2a** were used to afford the desired product (132.0 mg, 61% yield).

Alkyne **1l** and alcohol **2a** were used to afford the desired product (60.7 mg, 56% yield).

Alkyne **1u** and alcohol **2a** were used to afford the desired product (42.2 mg, 39% yield).

Alkyne **4c** and alcohol **2a** were used to afford the desired product (54.2 mg, 50% yield).

Alkyne **5c** and alcohol **2a** were used to afford the desired product (33.8 mg, 31% yield).

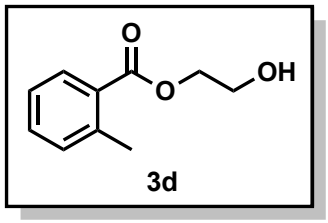
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87-7.85 (m, 2H), 7.38 (d, *J* = 7.6 Hz, 1H), 7.33 (t, *J* = 7.6 Hz, 1H), 4.47-4.45 (m, 2H), 3.97-3.95 (m, 2H), 2.40 (s, 3H), 2.14 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.3, 138.4, 134.1, 130.3, 129.9, 128.5, 127.0, 66.8, 61.6, 21.4.

HRMS (ESI) calcd. for $C_{10}H_{12}NaO_3^+$ ([M+Na]$^+$): 203.0679, found: 203.0682.

2-hydroxyethyl 2-methylbenzoate (**3d**)



Following general procedure, alkyne **1d** and alcohol **2a** were used to afford the desired product (122.4 mg, 57% yield).

Alkyne **1v** and alcohol **2a** were used to afford the desired product (54.9 mg, 51% yield).

Alkyne **4d** and alcohol **2a** were used to afford the desired product (56.6 mg, 52% yield).

Alkyne **5d** and alcohol **2a** were used to afford the desired product (45.6 mg, 42% yield).

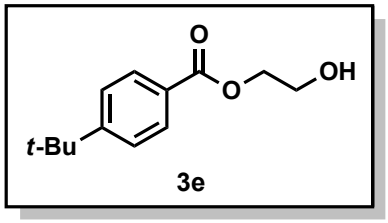
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.95-7.93 (m, 1H), 7.41 (t, *J* = 7.4 Hz, 1H), 7.27-7.24 (m, 2H), 4.44-4.43 (m, 2H), 3.96-3.95 (m, 2H), 2.61 (s, 3H), 2.25 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 168.0, 140.4, 132.3, 131.9, 130.7, 129.3, 125.9, 66.5, 61.5, 21.9.

HRMS (ESI) calcd. for $C_{10}H_{12}NaO_3^+$ ($[M+Na]^+$): 203.0679, found: 203.0677.

2-hydroxyethyl 4-(*tert*-butyl)benzoate (**3e**)



3e

Following general procedure, alkyne **1e** and alcohol **2a** were used to afford the desired product (169.7 mg, 64% yield).

Alkyne **1m** and alcohol **2a** were used to afford the desired product (94.8 mg, 71% yield).

Alkyne **4e** and alcohol **2a** were used to afford the desired product (83.6 mg, 63% yield).

Alkyne **5f** and alcohol **2a** were used to afford the desired product (83.4 mg, 63% yield).

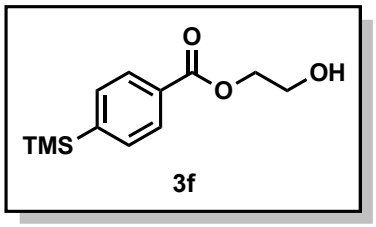
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.5 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 4.46-4.44 (m, 2H), 3.95-3.94 (m, 2H), 2.24 (brs, 1H), 1.33 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.2, 157.1, 129.7, 127.2, 125.5, 66.7, 61.6, 35.2, 31.2.

HRMS (ESI) calcd. for $C_{13}H_{19}O_3^+$ ($[M+H]^+$): 223.1329, found: 223.1329.

2-hydroxyethyl 4-(trimethylsilyl)benzoate (**3f**)



3f

Following general procedure, alkyne **1f** and alcohol **2a** were used to afford the desired product (212.3 mg, 74% yield).

Alkyne **1n** and alcohol **2a** were used to afford the desired product (114.7 mg, 80% yield).

Alkyne **5g** and alcohol **2a** were used to afford the desired product (50.4 mg, 35% yield).

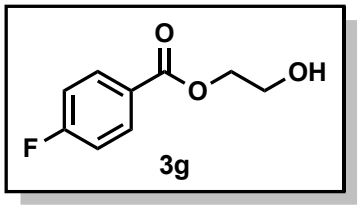
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.01 (d, *J* = 8.0 Hz, 2H), 7.60 (d, *J* = 8.0 Hz, 2H), 4.47-4.46 (m, 2H), 3.96-3.95 (m, 2H), 2.20 (brs, 1H), 0.29 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.3, 147.4, 133.5, 130.1, 128.7, 66.8, 61.6, -1.2.

HRMS (ESI) calcd. for $C_{12}H_{18}NaO_3Si^+$ ([M+Na]$^+$): 261.0917, found: 261.0914.

2-hydroxyethyl 4-fluorobenzoate (**3g**)



Following general procedure, alkyne **1g** and alcohol **2a** were used to afford the desired product (148.5 mg, 67% yield).

Alkyne **1o** and alcohol **2a** were used to afford the desired product (70.2 mg, 64% yield).

Alkyne **1w** and alcohol **2a** were used to afford the desired product (67.3 mg, 61% yield).

Alkyne **5h** and alcohol **2a** were used to afford the desired product (35.8 mg, 32% yield).

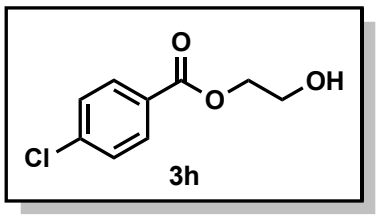
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 (dd, *J* = 8.1, 5.7 Hz, 2H), 7.10 (t, *J* = 8.3 Hz, 2H), 4.45-4.44 (m, 2H), 3.95-3.94 (m, 2H), 2.31 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.9, 166.1, 165.2, 132.4, 132.4, 126.3, 126.3, 115.8, 115.7, 66.9, 61.6.

HRMS (EI) calcd. for $C_9H_{10}FO_3^+$ ([M+H]$^+$): 185.0608, found:185.0609.

2-hydroxyethyl 4-chlorobenzoate (**3h**)



Following general procedure, alkyne **1h** and alcohol **2a** were used to afford the desired product (114.6 mg, 48% yield).

Alkyne **1p** and alcohol **2a** were used to afford the desired product (92.0 mg, 76% yield).

Alkyne **1x** and alcohol **2a** were used to afford the desired product (84.6 mg, 70% yield).

Alkyne **4f** and alcohol **2a** were used to afford the desired product (86.5 mg, 72% yield).

Alkyne **5i** and alcohol **2a** were used to afford the desired product (49.4 mg, 41% yield).

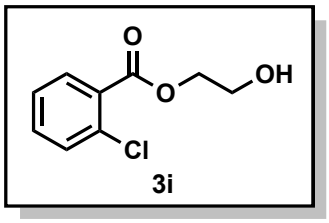
White solid, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 8.3 Hz, 2H), 7.41 (d, *J* = 8.3 Hz, 2H), 4.46-4.45 (m, 2H), 3.95 (s, 2H), 2.20 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.2, 139.8, 131.2, 128.9, 128.5, 67.0, 61.5.

HRMS (EI) calcd. for $C_9H_{10}ClO_3^+$ ([M+H]$^+$): 201.0313, found: 201.0316.

2-hydroxyethyl 2-chlorobenzoate (**3i**)



3i

Following general procedure, alkyne **1i** and alcohol **2a** were used to afford the desired product (126.9 mg, 53% yield).

Alkyne **1q** and alcohol **2a** were used to afford the desired product (80.9 mg, 67% yield).

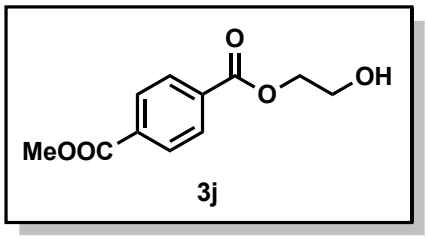
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.85 (dd, *J* = 7.8, 1.5 Hz, 1H), 7.47-7.42 (m, 2H), 7.33 (td, *J* = 7.8, 1.4 Hz, 1H), 4.48-4.46 (m, 2H), 3.96-3.95 (m, 2H), 2.09 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.1, 133.7, 132.9, 131.7, 131.2, 130.0, 126.8, 67.2, 61.2.

HRMS (ESI) calcd. for $C_9H_9ClNaO_3^+$ ([M+Na]$^+$): 223.0132, found: 223.0135.

2-hydroxyethyl methyl terephthalate (**3j**)



3j

Following general procedure, alkyne **1j** and alcohol **2a** were used to afford the desired product (126.8 mg, 47% yield).

Alkyne **4h** and alcohol **2a** were used to afford the desired product (54.1 mg, 40% yield).

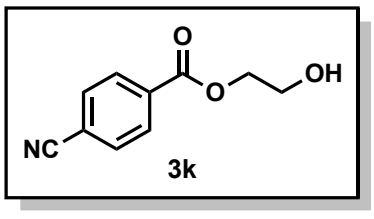
White solid, (eluent:petroleum ether/ethyl acetate = 1:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.12-8.08 (m, 4H), 4.49-4.48 (m, 2H), 3.98-3.97 (m, 2H), 3.94 (s, 3H), 2.19 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.4, 166.2, 134.2, 133.8, 129.8, 129.7, 67.1, 61.4, 52.6.

HRMS (ESI) calcd. for $C_{11}H_{12}NaO_5^+$ ([M+Na]$^+$): 247.0577, found: 247.0579.

2-hydroxyethyl 4-cyanobenzoate (**3k**)



3k

Following general procedure, alkyne **1r** and alcohol **2a** were used to afford the desired product (86.5 mg, 75% yield).

Alkyne **1t** and alcohol **2a** were used to afford the desired product (77.6 mg, 68% yield).

Alkyne **1u** and alcohol **2a** were used to afford the desired product (50.8 mg, 44% yield).

Alkyne **1v** and alcohol **2a** were used to afford the desired product (71.8 mg, 58% yield).

Alkyne **1w** and alcohol **2a** were used to afford the desired product (66.3 mg, 63% yield).

Alkyne **1x** and alcohol **2a** were used to afford the desired product (72.2 mg, 63% yield).

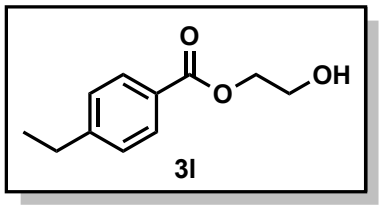
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.15 (d, *J* = 8.4 Hz, 2H), 7.74 (d, *J* = 8.5 Hz, 2H), 4.50-4.48 (m, 2H), 3.97 (s, 2H), 2.13 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 165.3, 133.9, 132.4, 130.3, 118.0, 116.7, 67.4, 61.2.

HRMS (ESI) calcd. for $C_{10}H_{10}NO_3^+$ ([M+H]$^+$): 192.0655, found: 192.0657.

2-hydroxyethyl 4-ethylbenzoate (**3l**)



3l

Following general procedure, alkyne **1s** and alcohol **2a** were used to afford the desired product (50.6 mg, 43% yield).

Alkyne **5e** and alcohol **2a** were used to afford the desired product (49.9 mg, 43% yield).

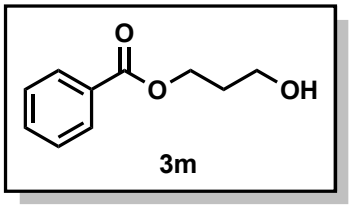
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.1 Hz, 2H), 7.27 (d, *J* = 6.8 Hz, 2H), 4.46-4.45 (m, 2H), 3.96-3.94 (m, 2H), 2.71 (q, *J* = 7.6 Hz, 2H), 2.17 (brs, 1H), 1.26 (t, *J* = 7.6 Hz, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.2, 150.3, 130.0, 128.1, 127.5, 66.7, 61.7, 29.1, 15.3.

HRMS (ESI) calcd. for $C_{11}H_{14}NaO_3^+$ ([M+H]$^+$): 217.0835, found: 217.0835.

3-hydroxypropyl benzoate (**3m**)



3m

Following general procedure, alkyne **1a** and alcohol **2b** were used to afford the desired product (139.4 mg, 64% yield).

Alkyne **1r** and alcohol **2b** were used to afford the desired product (54.1 mg, 50% yield).

Alkyne **4a** and alcohol **2b** were used to afford the desired product (58.4 mg, 54% yield).

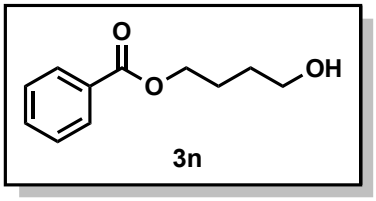
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.03 (d, *J* = 7.3 Hz, 2H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.44 (t, *J* = 7.8 Hz, 2H), 4.48 (t, *J* = 6.1 Hz, 2H), 3.77 (t, *J* = 6.1 Hz, 2H), 2.11-1.99 (m, 3H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.2, 133.2, 130.2, 129.7, 128.5, 61.9, 59.3, 32.0.

HRMS (ESI) calcd. for $C_{10}H_{12}NaO_3^+$ ([M+Na]$^+$): 203.0679, found: 203.0679.

4-hydroxybutyl benzoate (**3n**)



3n

Following general procedure, alkyne **1a** and alcohol **2c** were used to afford the desired product (118.2 mg, 51% yield).

Alkyne **1r** and alcohol **2c** were used to afford the desired product (34.9 mg, 30% yield).

Alkyne **4a** and alcohol **2c** were used to afford the desired product (50.8 mg, 44% yield).

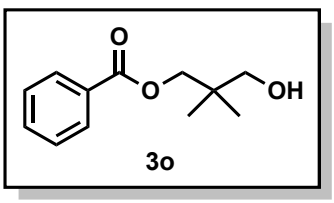
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.4 Hz, 2H), 7.55 (t, *J* = 7.4 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H), 4.36 (t, *J* = 6.5 Hz, 2H), 3.72 (t, *J* = 6.5 Hz, 2H), 1.89-1.85 (m, 2H), 1.75-1.71 (m, 2H), 1.65 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.8, 133.0, 130.5, 129.7, 128.5, 64.9, 62.5, 29.4, 25.4.

HRMS (ESI) calcd. for $C_{11}H_{14}NaO_3^+$ ([M+Na]$^+$): 217.0835, found: 217.0828.

3-hydroxy-2,2-dimethylpropyl benzoate (**3o**)



3o

Following general procedure, alkyne **1a** and alcohol **2d** were used to afford the desired product (201.1 mg, 80% yield).

Alkyne **1r** and alcohol **2d** were used to afford the desired product (29.5 mg, 25% yield).

Alkyne **4d** and alcohol **2a** were used to afford the desired product (87.9 mg, 70% yield).

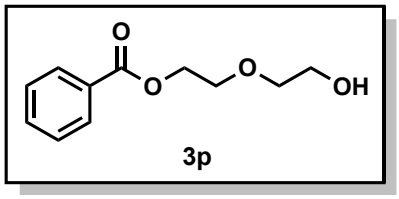
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.04 (d, *J* = 7.9 Hz, 2H), 7.57 (t, *J* = 7.3 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 2H), 4.18 (s, 2H), 3.39 (s, 2H), 2.41 (brs, 1H), 1.01 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.3, 133.3, 130.1, 129.8, 128.6, 69.9, 68.3, 36.9, 21.7.

HRMS (ESI) calcd. for $C_{12}H_{16}NaO_3^+$ ([M+Na]$^+$): 231.0992, found: 231.0992.

2-(2-hydroxyethoxy)ethyl benzoate (**3p**)



3p

Following general procedure, alkyne **1a** and alcohol **2c** were used to afford the desired product (78.2 mg, 31% yield).

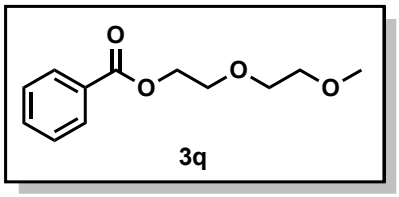
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.05 (dd, *J* = 8.2, 1.1 Hz, 2H), 7.57-7.54 (m, 1H), 7.44 (t, J = 7.8 Hz, 2H), 4.49 (t, *J* = 4.8 Hz, 2H), 3.84 (t, *J* = 4.8 Hz, 2H), 3.75 (t, *J* = 4.8 Hz, 2H), 3.65 (t, *J* = 4.8 Hz, 2H), 2.23 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7, 133.2, 130.1, 129.8, 128.5, 72.5, 69.4, 64.1, 61.9.

HRMS (ESI) calcd. for $C_{11}H_{14}NaO_4^+$ ([M+Na]$^+$): 233.0784, found: 233.0789.

2-(2-methoxyethoxy)ethyl benzoate (**3q**)



3q

Following general procedure, alkyne **1a** and alcohol **2d** were used to afford the desired product (107.0 mg, 40% yield).

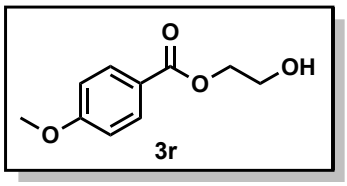
Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.06 (dd, J = 8.3, 1.2 Hz, 2H), 7.57-7.54 (m, 1H), 7.45-7.42 (m, 2H), 4.49-4.48 (m, 2H), 3.85-3.83 (m, 2H), 3.71-3.69 (m, 2H), 3.57-3.56 (m, 2H), 3.38 (s, 3H).

^{13}C NMR (151 MHz, CDCl3) δ 166.7, 133.1, 130.3, 129.8, 128.5, 72.1, 70.7, 69.4, 64.3, 59.2.

HRMS (ESI) calcd. for $C_{12}H_{16}NaO_4^+$ ([M+Na]$^+$): 247.0941, found: 247.0944.

2-hydroxyethyl 4-methoxybenzoate (**3r**)



Following general procedure, alkyne **4g** and alcohol **2a** were used to afford the desired product (49 mg, 42% yield).

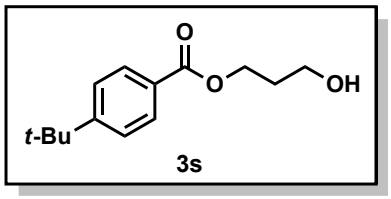
Colorless oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02-7.99 (m, 2H), 6.93-6.90 (m, 2H), 4.44-4.42 (m, 2H), 3.95-3.93 (m, 2H), 3.86 (s, 3H), 2.09 (brs, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.9, 163.7, 131.9, 122.4, 113.8, 66.6, 61.7, 55.6.

HRMS (ESI) calcd. for $C_{10}H_{12}NaO_4^+$ ([M+Na]$^+$): 219.0628, found: 219.0628.

3-hydroxypropyl 4-(*tert*-butyl)benzoate (**3s**)



Following general procedure, alkyne **5f** and alcohol **2b** were used to afford the desired product (73.8 mg, 52% yield).

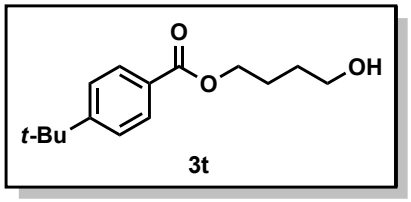
Yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, J = 8.5 Hz, 2H), 7.45 (d, J = 8.4 Hz, 2H), 4.48 (t, J = 6.1 Hz, 2H), 3.76 (t, J = 6.0 Hz, 2H), 2.08 (brs, 1H), 2.02-1.98 (m, 2H), 1.34 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.2, 156.9, 129.6, 127.4, 125.5, , 61.7, 59.3, 35.2, 32.1, 31.2.

HRMS (ESI) calcd. for $C_{14}H_{20}NaO_3^+$ ([M+Na]$^+$): 259.1305, found: 259.1303.

4-hydroxybutyl 4-(*tert*-butyl)benzoate (**3t**)



Following general procedure, alkyne **5f** and alcohol **2c** were used to afford the desired product (86.8 mg, 58% yield).

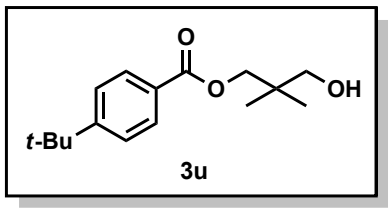
Yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.96 (d, *J* = 8.5 Hz, 2H), 7.45 (d, *J* = 8.5 Hz, 2H), 4.35 (t, *J* = 6.5 Hz, 2H), 3.72 (t, *J* = 6.5 Hz, 2H), 1.88-1.84 (m, 2H), 1.75-1.67 (m, 3H), 1.33 (s, 9H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 166.9, 156.7, 129.6, 127.7, 125.5, 64.7, 62.6, 35.2, 31.3, 29.4, 25.4.

HRMS (ESI) calcd. for $C_{15}H_{22}NaO_3^+$ ([M+Na]$^+$): 273.1461, found: 273.1462.

3-hydroxy-2,2-dimethylpropyl 4-(*tert*-butyl)benzoate (**3u**)



Following general procedure, alkyne **5f** and alcohol **2d** were used to afford the desired product (76.1 mg, 48% yield).

Pale yellow oil, (eluent:petroleum ether/ethyl acetate = 2:1).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.4 Hz, 2H), 7.46 (d, *J* = 8.4 Hz, 2H), 4.17 (s, 2H), 3.37 (s, 2H), 2.41 (brs, 1H), 1.34 (s, 9H), 1.00 (s, 6H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 167.3, 157.0, 129.7, 127.3, 125.6, 69.7, 68.4, 37.0, 35.3, 31.2, 21.7.

HRMS (ESI) calcd. for $C_{16}H_{25}O_3^+$ ([M+H]$^+$): 265.1798, found: 265.1795.

4. X-Ray crystal data of **3j** (CCDC 2031057)

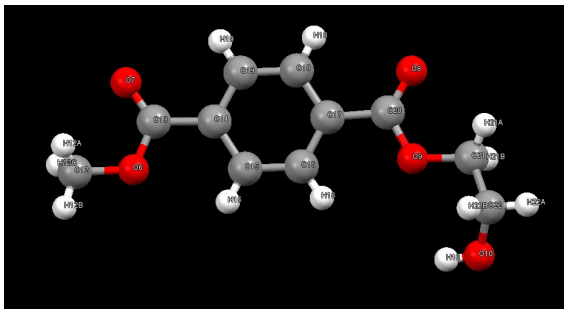


Table S1. Crystal data and structure refinement for 111.

Identification code	111
Empirical formula	C44 H48 O20
Formula weight	896.82

Temperature	293(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P 1 21 1
Unit cell dimensions	a = 8.3520(9) A alpha = 90 deg. b = 5.7066(6) A beta = 91.450(3) deg. c = 22.623(2) A gamma = 90 deg.
Volume	1077.90(19) A^3
Z, Calculated density	1, 1.382 Mg/m^3
Absorption coefficient	0.110 mm^-1
F(000)	472
Crystal size	0.25 x 0.21 x 0.14 mm
Theta range for data collection	0.900 to 27.550 deg.
Limiting indices	-10<=h<=10, -7<=k<=7, -29<=l<=29
Reflections collected / unique	16076 / 4909 [R(int) = 0.0296]
Completeness to theta = 25.242	99.2 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.6422
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	4909 / 1 / 293
Goodness-of-fit on F^2	0.961
Final R indices [I>2sigma(I)]	R1 = 0.0450, wR2 = 0.1350
R indices (all data)	R1 = 0.0594, wR2 = 0.1507
Absolute structure parameter	0.0(3)
Extinction coefficient	n/a
Largest diff. peak and hole	0.256 and -0.194 e.A^-3

Table S2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for 111.
U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(4)	-2031(2)	728(4)	-1384(1)	48(1)
O(1)	-6176(3)	3342(4)	-3939(1)	56(1)
O(5)	144(2)	4141(5)	-840(1)	65(1)
O(2)	-5774(4)	-221(5)	-4315(1)	72(1)
O(3)	-1632(3)	-3000(4)	-1657(1)	69(1)
C(1)	-6955(4)	4036(7)	-4491(1)	64(1)
C(3)	-4787(3)	618(5)	-3341(1)	39(1)
C(9)	-2195(3)	-1077(5)	-1751(1)	44(1)
C(2)	-5633(3)	1154(6)	-3916(1)	45(1)
C(4)	-4730(3)	2214(5)	-2878(1)	42(1)
C(6)	-3129(3)	-453(5)	-2297(1)	38(1)
C(7)	-3198(3)	-2071(5)	-2758(1)	49(1)
C(11)	-935(4)	2664(7)	-543(1)	57(1)
C(10)	-1133(3)	337(6)	-834(1)	53(1)
C(5)	-3900(3)	1698(5)	-2356(1)	43(1)
C(8)	-4033(3)	-1532(5)	-3277(1)	48(1)
O(9)	2996(3)	-151(5)	-1393(1)	58(1)
O(6)	-1144(3)	3321(4)	-3902(1)	62(1)
O(7)	-781(4)	-137(5)	-4332(1)	83(1)
O(10)	3043(3)	2645(7)	-323(1)	85(1)
C(19)	981(4)	-1738(6)	-3329(1)	55(1)
O(8)	3443(4)	-3707(5)	-1764(1)	86(1)
C(12)	-1938(5)	4133(8)	-4440(1)	70(1)
C(21)	3945(4)	-733(7)	-869(1)	63(1)
C(22)	4341(4)	1550(9)	-571(2)	74(1)
C(13)	-609(4)	1125(6)	-3911(1)	51(1)
C(14)	247(3)	431(5)	-3354(1)	45(1)
C(20)	2846(3)	-1790(6)	-1807(1)	51(1)
C(17)	1922(3)	-966(5)	-2336(1)	43(1)
C(16)	1177(3)	1209(5)	-2356(1)	46(1)
C(15)	339(3)	1907(5)	-2861(1)	46(1)
C(18)	1809(4)	-2441(5)	-2824(1)	54(1)

Table S3. Bond lengths [A] and angles [deg] for 111.

O(4)-C(9)	1.328(4)
O(4)-C(10)	1.454(3)
O(1)-C(1)	1.449(3)
O(1)-C(2)	1.329(4)
O(5)-H(5)	0.8200
O(5)-C(11)	1.415(4)
O(2)-C(2)	1.199(4)
O(3)-C(9)	1.210(4)
C(1)-H(1A)	0.9600
C(1)-H(1B)	0.9600
C(1)-H(1C)	0.9600
C(3)-C(2)	1.496(3)
C(3)-C(4)	1.390(4)
C(3)-C(8)	1.385(4)
C(9)-C(6)	1.487(4)
C(4)-H(4)	0.9300
C(4)-C(5)	1.385(3)
C(6)-C(7)	1.393(4)
C(6)-C(5)	1.391(4)
C(7)-H(7)	0.9300
C(7)-C(8)	1.385(4)
C(11)-H(11A)	0.9700
C(11)-H(11B)	0.9700
C(11)-C(10)	1.489(5)
C(10)-H(10A)	0.9700
C(10)-H(10B)	0.9700
C(5)-H(5A)	0.9300
C(8)-H(8)	0.9300
O(9)-C(21)	1.448(3)
O(9)-C(20)	1.326(4)
O(6)-C(12)	1.447(4)
O(6)-C(13)	1.331(4)
O(7)-C(13)	1.200(4)
O(10)-H(10)	0.8200
O(10)-C(22)	1.382(4)
C(19)-H(19)	0.9300
C(19)-C(14)	1.382(4)
C(19)-C(18)	1.380(4)
O(8)-C(20)	1.205(4)
C(12)-H(12A)	0.9600
C(12)-H(12B)	0.9600
C(12)-H(12C)	0.9600
C(21)-H(21A)	0.9700
C(21)-H(21B)	0.9700
C(21)-C(22)	1.500(6)

C(22)-H(22A)	0.9700
C(22)-H(22B)	0.9700
C(13)-C(14)	1.487(4)
C(14)-C(15)	1.398(4)
C(20)-C(17)	1.484(4)
C(17)-C(16)	1.388(4)
C(17)-C(18)	1.390(4)
C(16)-H(16)	0.9300
C(16)-C(15)	1.383(4)
C(15)-H(15)	0.9300
C(18)-H(18)	0.9300

C(9)-O(4)-C(10)	117.3(2)
C(2)-O(1)-C(1)	115.8(2)
C(11)-O(5)-H(5)	109.5
O(1)-C(1)-H(1A)	109.5
O(1)-C(1)-H(1B)	109.5
O(1)-C(1)-H(1C)	109.5
H(1A)-C(1)-H(1B)	109.5
H(1A)-C(1)-H(1C)	109.5
H(1B)-C(1)-H(1C)	109.5
C(4)-C(3)-C(2)	121.9(2)
C(8)-C(3)-C(2)	118.4(2)
C(8)-C(3)-C(4)	119.7(2)
O(4)-C(9)-C(6)	112.1(2)
O(3)-C(9)-O(4)	124.1(3)
O(3)-C(9)-C(6)	123.7(3)
O(1)-C(2)-C(3)	112.3(2)
O(2)-C(2)-O(1)	124.0(3)
O(2)-C(2)-C(3)	123.7(3)
C(3)-C(4)-H(4)	119.6
C(5)-C(4)-C(3)	120.7(3)
C(5)-C(4)-H(4)	119.6
C(7)-C(6)-C(9)	118.3(2)
C(5)-C(6)-C(9)	121.5(2)
C(5)-C(6)-C(7)	120.2(2)
C(6)-C(7)-H(7)	120.1
C(8)-C(7)-C(6)	119.9(3)
C(8)-C(7)-H(7)	120.1
O(5)-C(11)-H(11A)	109.0
O(5)-C(11)-H(11B)	109.0
O(5)-C(11)-C(10)	112.8(2)
H(11A)-C(11)-H(11B)	107.8
C(10)-C(11)-H(11A)	109.0
C(10)-C(11)-H(11B)	109.0
O(4)-C(10)-C(11)	106.9(2)
O(4)-C(10)-H(10A)	110.3
O(4)-C(10)-H(10B)	110.3
C(11)-C(10)-H(10A)	110.3

C(11)-C(10)-H(10B)	110.3
H(10A)-C(10)-H(10B)	108.6
C(4)-C(5)-C(6)	119.3(2)
C(4)-C(5)-H(5A)	120.3
C(6)-C(5)-H(5A)	120.3
C(3)-C(8)-C(7)	120.2(2)
C(3)-C(8)-H(8)	119.9
C(7)-C(8)-H(8)	119.9
C(20)-O(9)-C(21)	117.1(3)
C(13)-O(6)-C(12)	115.9(3)
C(22)-O(10)-H(10)	109.5
C(14)-C(19)-H(19)	119.8
C(18)-C(19)-H(19)	119.8
C(18)-C(19)-C(14)	120.4(3)
O(6)-C(12)-H(12A)	109.5
O(6)-C(12)-H(12B)	109.5
O(6)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
O(9)-C(21)-H(21A)	110.5
O(9)-C(21)-H(21B)	110.5
O(9)-C(21)-C(22)	106.2(3)
H(21A)-C(21)-H(21B)	108.7
C(22)-C(21)-H(21A)	110.5
C(22)-C(21)-H(21B)	110.5
O(10)-C(22)-C(21)	114.2(3)
O(10)-C(22)-H(22A)	108.7
O(10)-C(22)-H(22B)	108.7
C(21)-C(22)-H(22A)	108.7
C(21)-C(22)-H(22B)	108.7
H(22A)-C(22)-H(22B)	107.6
O(6)-C(13)-C(14)	113.1(2)
O(7)-C(13)-O(6)	122.9(3)
O(7)-C(13)-C(14)	124.0(3)
C(19)-C(14)-C(13)	118.4(2)
C(19)-C(14)-C(15)	119.5(3)
C(15)-C(14)-C(13)	122.1(3)
O(9)-C(20)-C(17)	112.5(3)
O(8)-C(20)-O(9)	123.5(3)
O(8)-C(20)-C(17)	124.0(3)
C(16)-C(17)-C(20)	122.2(2)
C(16)-C(17)-C(18)	119.6(3)
C(18)-C(17)-C(20)	118.2(3)
C(17)-C(16)-H(16)	119.9
C(15)-C(16)-C(17)	120.1(2)
C(15)-C(16)-H(16)	119.9
C(14)-C(15)-H(15)	120.0
C(16)-C(15)-C(14)	120.1(3)

C(16)-C(15)-H(15)	120.0
C(19)-C(18)-C(17)	120.3(3)
C(19)-C(18)-H(18)	119.9
C(17)-C(18)-H(18)	119.9

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for 111.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

	U11	U22	U33	U23	U13	U12
O(4)	54(1)	48(1)	42(1)	5(1)	-11(1)	5(1)
O(1)	78(1)	51(1)	39(1)	2(1)	-14(1)	1(1)
O(5)	57(1)	60(1)	75(2)	11(1)	-18(1)	2(1)
O(2)	108(2)	60(2)	47(1)	-12(1)	-15(1)	0(1)
O(3)	87(2)	42(1)	76(1)	12(1)	-23(1)	9(1)
C(1)	82(2)	68(2)	41(2)	9(2)	-15(1)	0(2)
C(3)	43(1)	36(1)	37(1)	1(1)	2(1)	-8(1)
C(9)	42(1)	39(1)	50(1)	9(1)	1(1)	-3(1)
C(2)	52(1)	47(2)	37(1)	0(1)	1(1)	-11(1)
C(4)	50(1)	35(1)	41(1)	-1(1)	-4(1)	2(1)
C(6)	37(1)	36(1)	43(1)	5(1)	1(1)	-6(1)
C(7)	53(2)	34(1)	60(2)	0(1)	-1(1)	0(1)
C(11)	56(2)	70(2)	44(1)	1(1)	-9(1)	3(2)
C(10)	49(2)	63(2)	46(1)	13(1)	-12(1)	-3(1)
C(5)	50(1)	37(2)	40(1)	-2(1)	-4(1)	1(1)
C(8)	58(2)	38(2)	48(1)	-8(1)	0(1)	-4(1)
O(9)	60(1)	60(1)	53(1)	-4(1)	-7(1)	10(1)
O(6)	84(2)	58(2)	45(1)	-4(1)	-8(1)	-4(1)
O(7)	119(2)	76(2)	52(1)	-21(1)	-14(1)	-4(2)
O(10)	69(1)	125(3)	61(1)	-20(2)	-15(1)	20(2)
C(19)	62(2)	50(2)	52(2)	-20(1)	4(1)	-6(1)
O(8)	110(2)	48(2)	99(2)	-2(1)	-24(2)	16(2)
C(12)	89(2)	71(2)	50(2)	4(2)	-10(2)	0(2)
C(21)	55(2)	74(2)	60(2)	6(2)	-6(1)	11(2)
C(22)	55(2)	105(3)	60(2)	-11(2)	-14(1)	11(2)
C(13)	59(2)	55(2)	40(1)	-7(1)	5(1)	-16(1)
C(14)	47(1)	44(2)	44(1)	-6(1)	7(1)	-13(1)
C(20)	47(1)	44(2)	62(2)	2(1)	4(1)	-5(1)
C(17)	42(1)	38(1)	51(1)	-4(1)	9(1)	-7(1)
C(16)	54(2)	40(2)	44(1)	-10(1)	4(1)	-4(1)
C(15)	54(2)	38(2)	45(1)	-6(1)	2(1)	-4(1)
C(18)	54(2)	40(2)	69(2)	-13(1)	5(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for 111.

	x	y	z	U(eq)
H(5)	-301	4692	-1136	97
H(1A)	-6238	3786	-4810	96
H(1B)	-7236	5665	-4473	96
H(1C)	-7906	3117	-4556	96
H(4)	-5255	3645	-2918	51
H(7)	-2684	-3510	-2717	59
H(11A)	-1970	3429	-530	68
H(11B)	-549	2435	-140	68
H(10A)	-95	-342	-913	63
H(10B)	-1710	-724	-581	63
H(5A)	-3860	2778	-2048	51
H(8)	-4087	-2617	-3584	58
H(10)	2383	2980	-584	128
H(19)	917	-2730	-3655	66
H(12A)	-1228	3972	-4765	105
H(12B)	-2228	5751	-4396	105
H(12C)	-2885	3216	-4516	105
H(21A)	4917	-1544	-976	76
H(21B)	3342	-1733	-609	76
H(22A)	5148	1270	-263	88
H(22B)	4801	2599	-858	88
H(16)	1243	2198	-2029	55
H(15)	-164	3361	-2872	55
H(18)	2294	-3907	-2810	65

Table S6. Torsion angles [deg] for 111.

O(4)-C(9)-C(6)-C(7)	170.8(2)
O(4)-C(9)-C(6)-C(5)	-8.2(3)
O(5)-C(11)-C(10)-O(4)	-70.9(3)
O(3)-C(9)-C(6)-C(7)	-8.2(4)
O(3)-C(9)-C(6)-C(5)	172.8(3)
C(1)-O(1)-C(2)-O(2)	-1.0(4)
C(1)-O(1)-C(2)-C(3)	177.5(2)
C(3)-C(4)-C(5)-C(6)	-0.5(4)
C(9)-O(4)-C(10)-C(11)	172.6(2)
C(9)-C(6)-C(7)-C(8)	-178.8(2)
C(9)-C(6)-C(5)-C(4)	178.8(2)
C(2)-C(3)-C(4)-C(5)	-178.2(2)
C(2)-C(3)-C(8)-C(7)	178.2(2)
C(4)-C(3)-C(2)-O(1)	5.3(3)
C(4)-C(3)-C(2)-O(2)	-176.2(3)
C(4)-C(3)-C(8)-C(7)	-1.3(4)
C(6)-C(7)-C(8)-C(3)	0.6(4)
C(7)-C(6)-C(5)-C(4)	-0.2(4)
C(10)-O(4)-C(9)-O(3)	-0.8(4)
C(10)-O(4)-C(9)-C(6)	-179.8(2)
C(5)-C(6)-C(7)-C(8)	0.2(4)
C(8)-C(3)-C(2)-O(1)	-174.2(2)
C(8)-C(3)-C(2)-O(2)	4.3(4)
C(8)-C(3)-C(4)-C(5)	1.3(4)
O(9)-C(21)-C(22)-O(10)	69.7(4)
O(9)-C(20)-C(17)-C(16)	-6.0(4)
O(9)-C(20)-C(17)-C(18)	173.9(2)
O(6)-C(13)-C(14)-C(19)	-174.3(2)
O(6)-C(13)-C(14)-C(15)	5.1(4)
O(7)-C(13)-C(14)-C(19)	4.4(4)
O(7)-C(13)-C(14)-C(15)	-176.1(3)
C(19)-C(14)-C(15)-C(16)	0.8(4)
O(8)-C(20)-C(17)-C(16)	176.4(3)
O(8)-C(20)-C(17)-C(18)	-3.7(5)
C(12)-O(6)-C(13)-O(7)	-0.9(4)
C(12)-O(6)-C(13)-C(14)	177.9(3)
C(21)-O(9)-C(20)-O(8)	0.5(5)
C(21)-O(9)-C(20)-C(17)	-177.1(2)
C(13)-C(14)-C(15)-C(16)	-178.6(2)
C(14)-C(19)-C(18)-C(17)	-0.4(4)
C(20)-O(9)-C(21)-C(22)	163.2(3)
C(20)-C(17)-C(16)-C(15)	179.5(2)
C(20)-C(17)-C(18)-C(19)	-179.1(3)
C(17)-C(16)-C(15)-C(14)	-0.4(4)
C(16)-C(17)-C(18)-C(19)	0.9(4)

C(18)-C(19)-C(14)-C(13)	179.0(3)
C(18)-C(19)-C(14)-C(15)	-0.4(4)
C(18)-C(17)-C(16)-C(15)	-0.5(4)

Symmetry transformations used to generate equivalent atoms:

Table s7. Hydrogen bonds for 111 [A and deg.].

D-H...A				d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(5)	--H(5)	..O(3)	[1565.01]	0.82	2.07	2.8540(3)	159
O(10)	--H(10)	..O(5)	[1655.01]	0.82	2.05	2.7955(3)	150
C(4)	--H(4)	..O(1)	[ ]	0.93	2.42	2.7372(3)	100
C(10)	--H(10B)	..O(10)	[2645.02]	0.97	2.53	3.4596(4)	160

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NMR spectra of the products

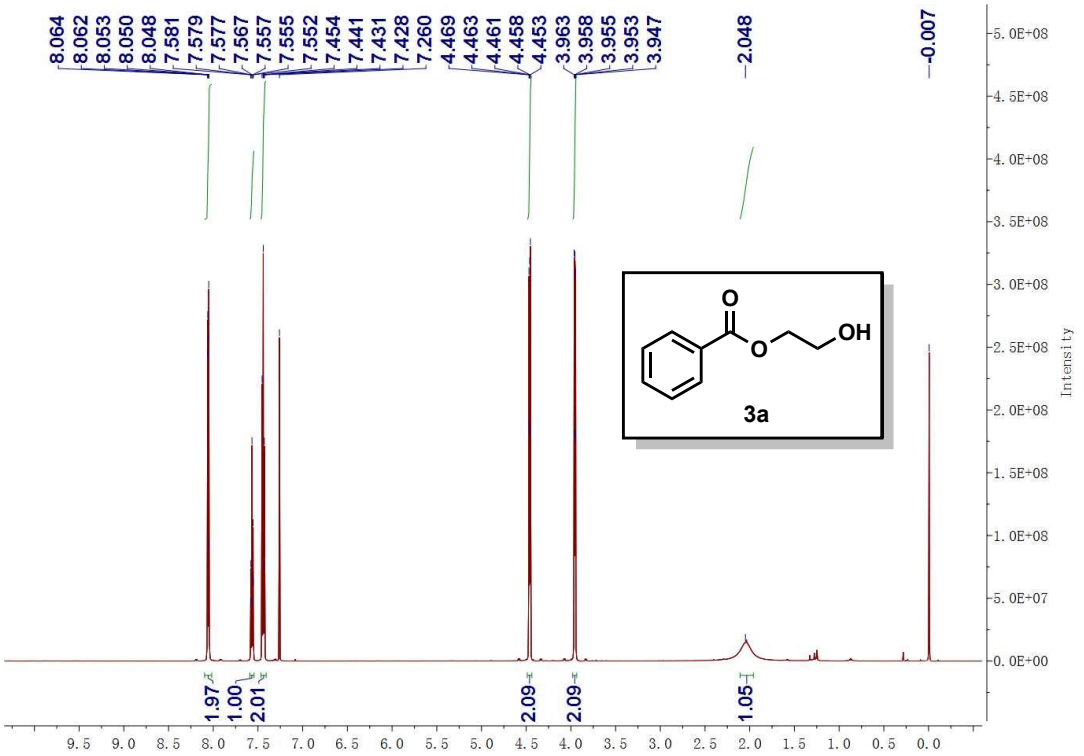

Figure S1. ^{1}H NMR of **3a**.

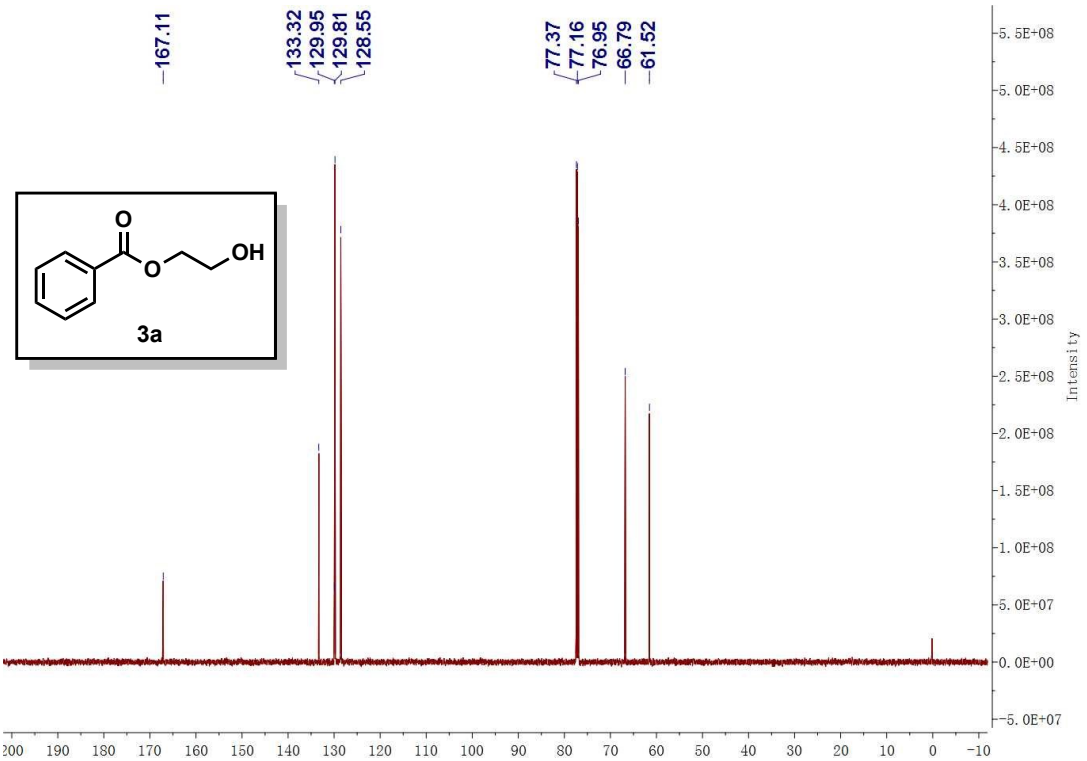

Figure S2. ^{13}C NMR of **3a**.

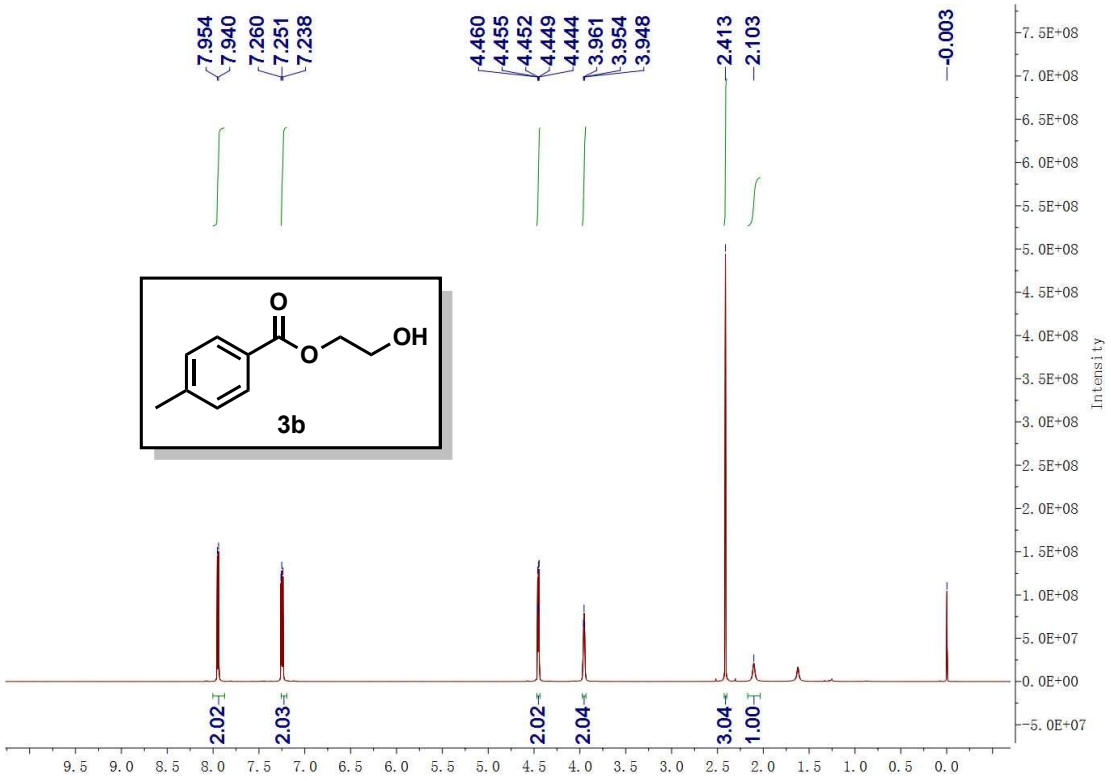

Figure S3. ^{1}H NMR of **3b**.

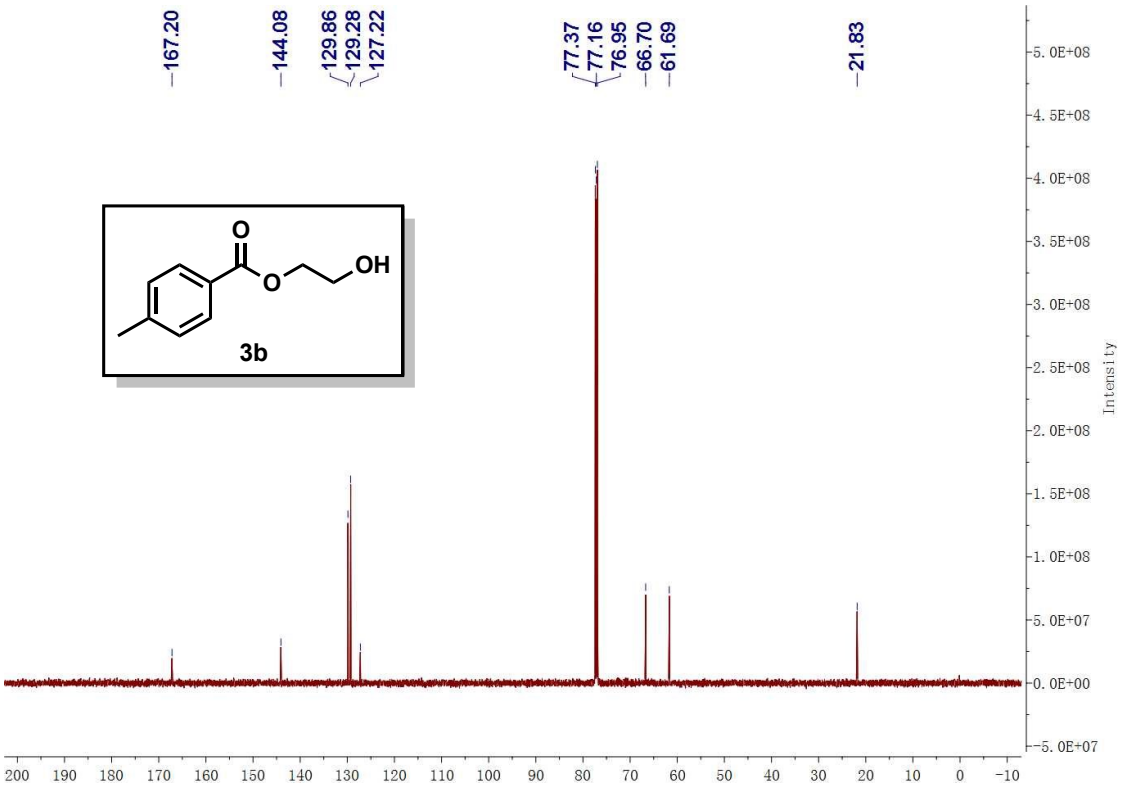

Figure S4. ^{13}C NMR of **3b**.

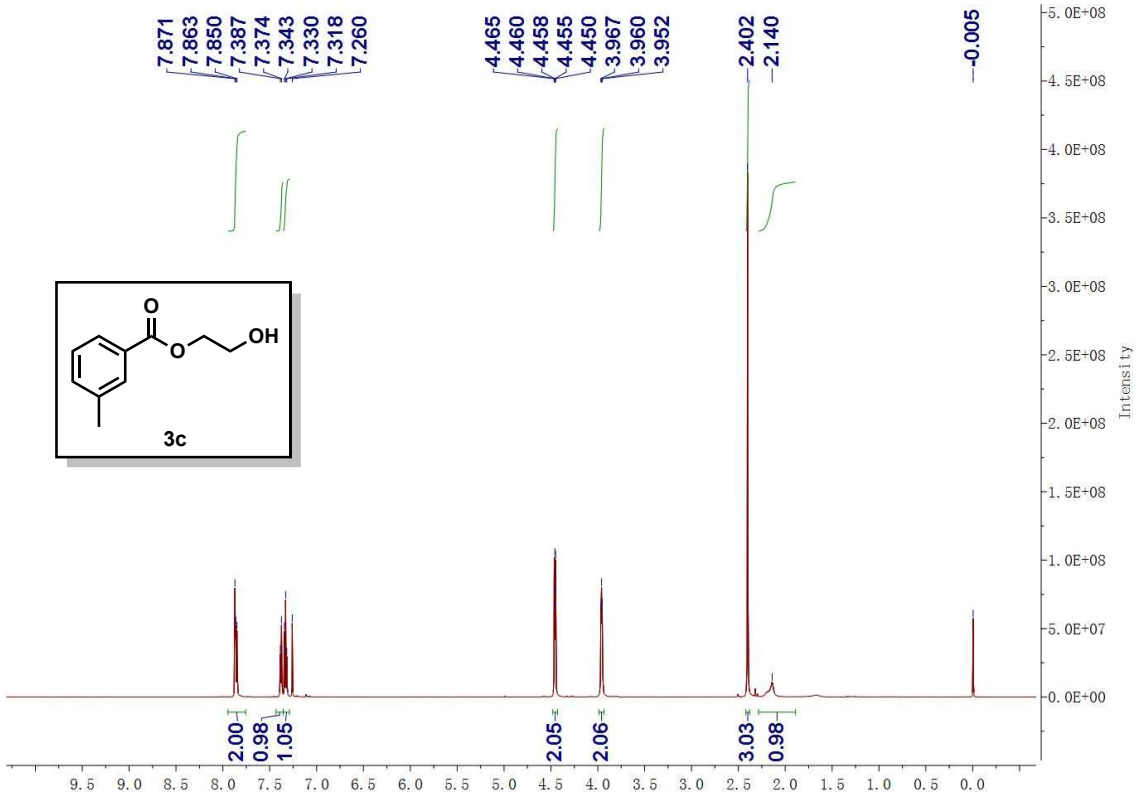

Figure S5. ^{1}H NMR of **3c**.

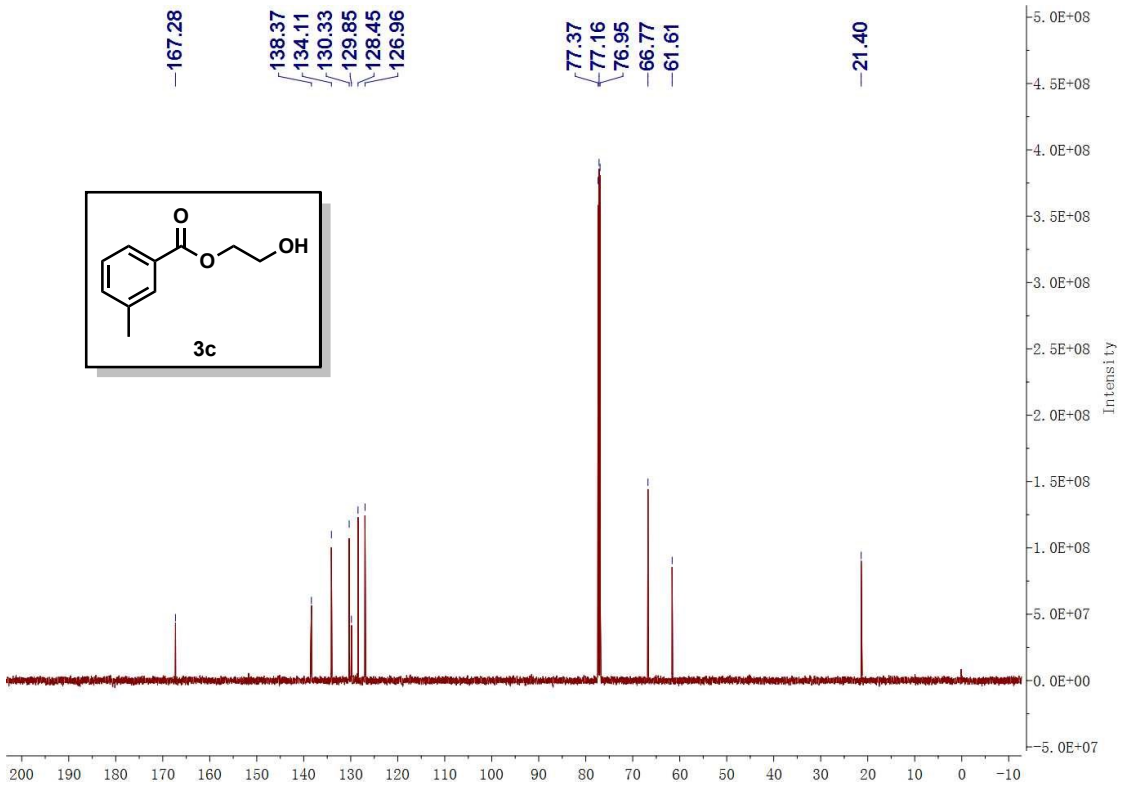

Figure S6. ^{13}C NMR of **3c**.

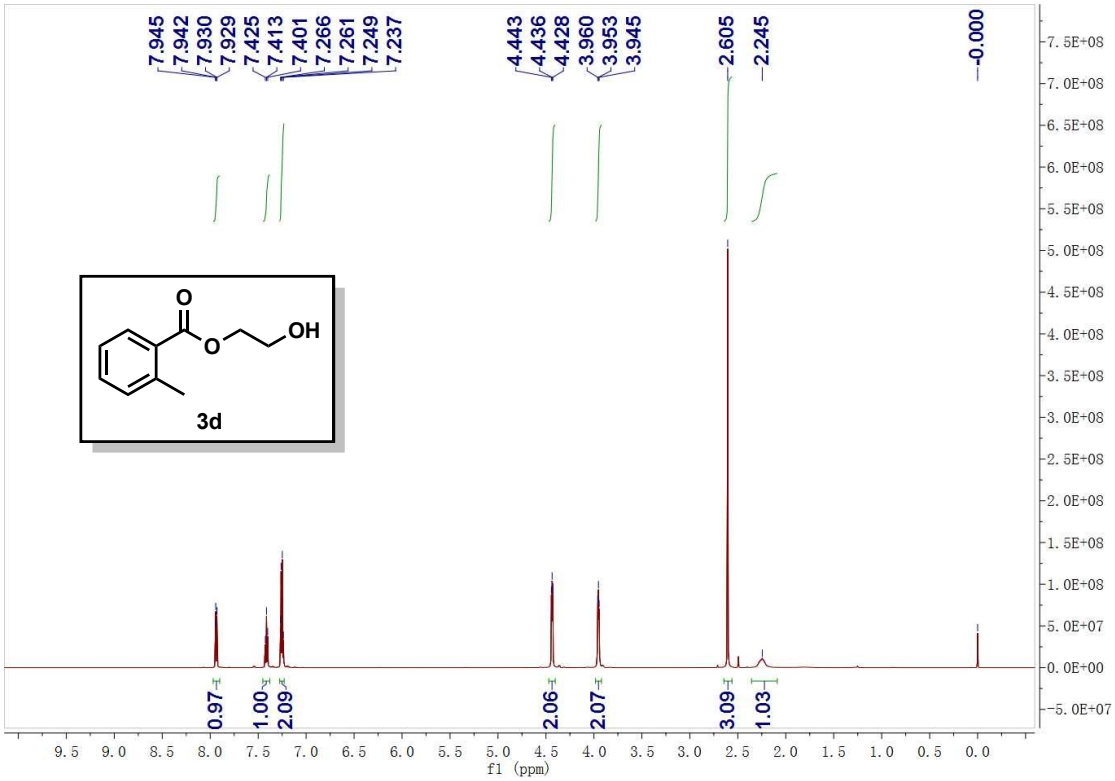

Figure S7. ^{1}H NMR of **3d**.

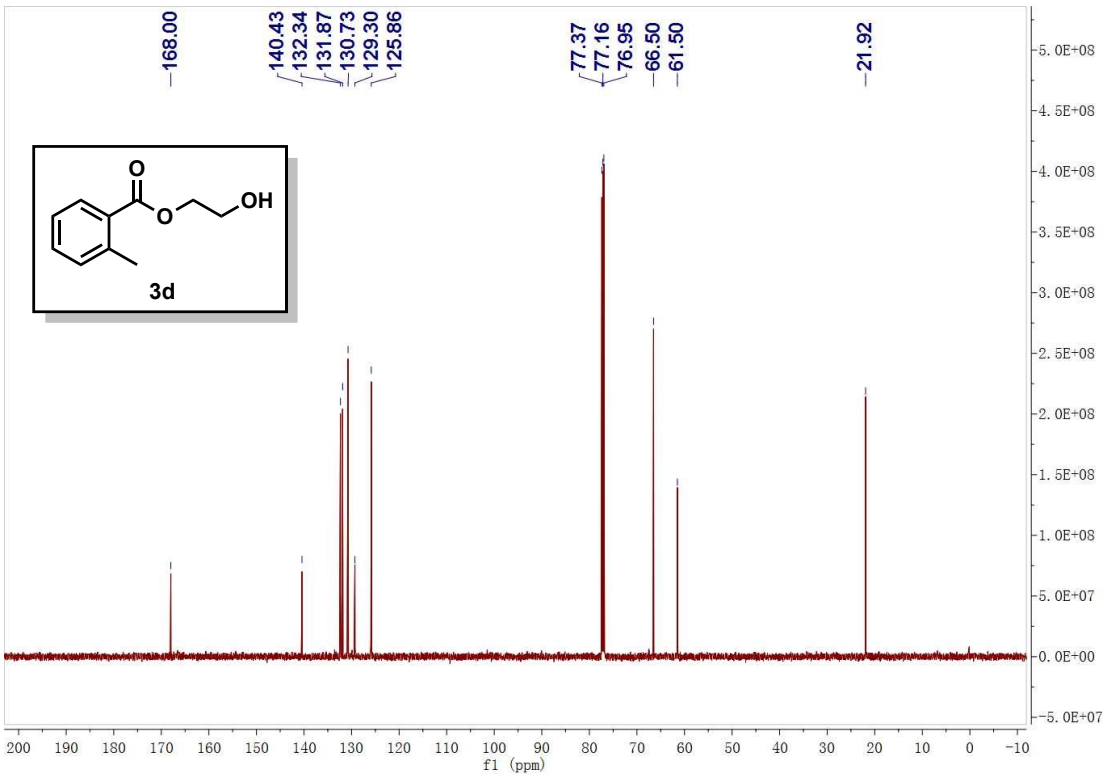

Figure S8. ^{13}C NMR of **3d**.

Figure S9. ^{1}H NMR of **3e**.

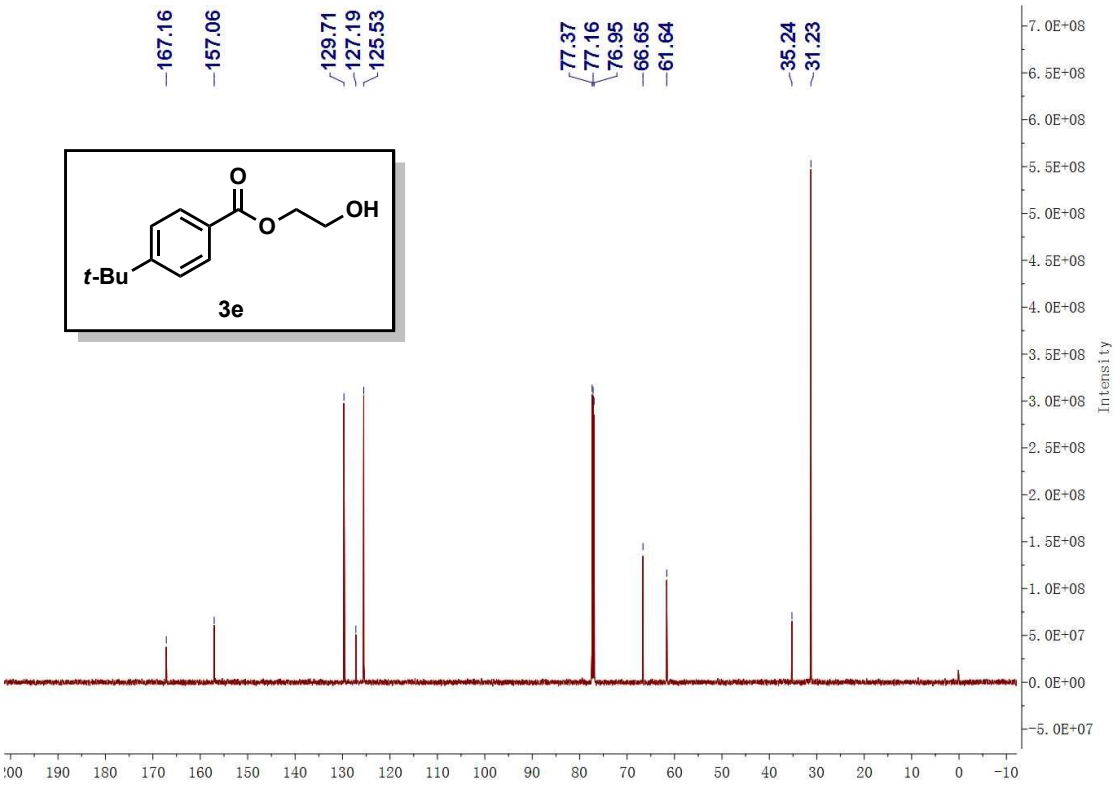

Figure S10. ^{13}C NMR of **3e**.

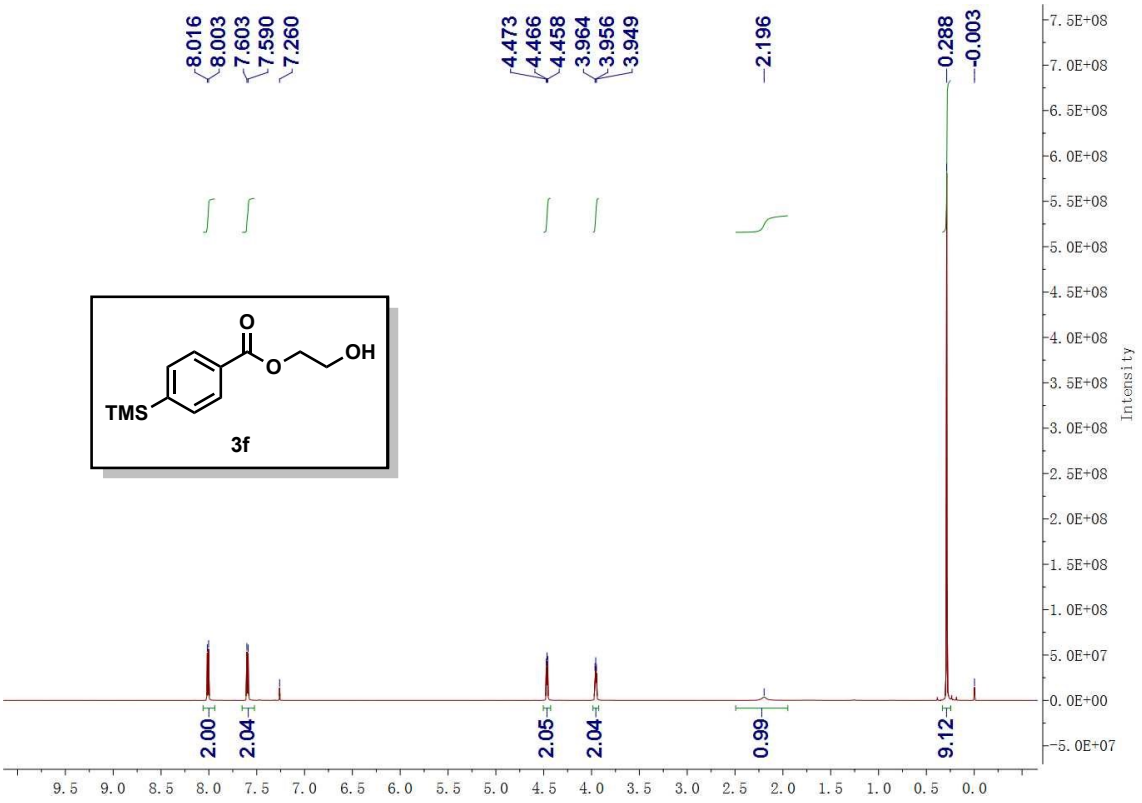

Figure S11. ^{1}H NMR of **3f**.

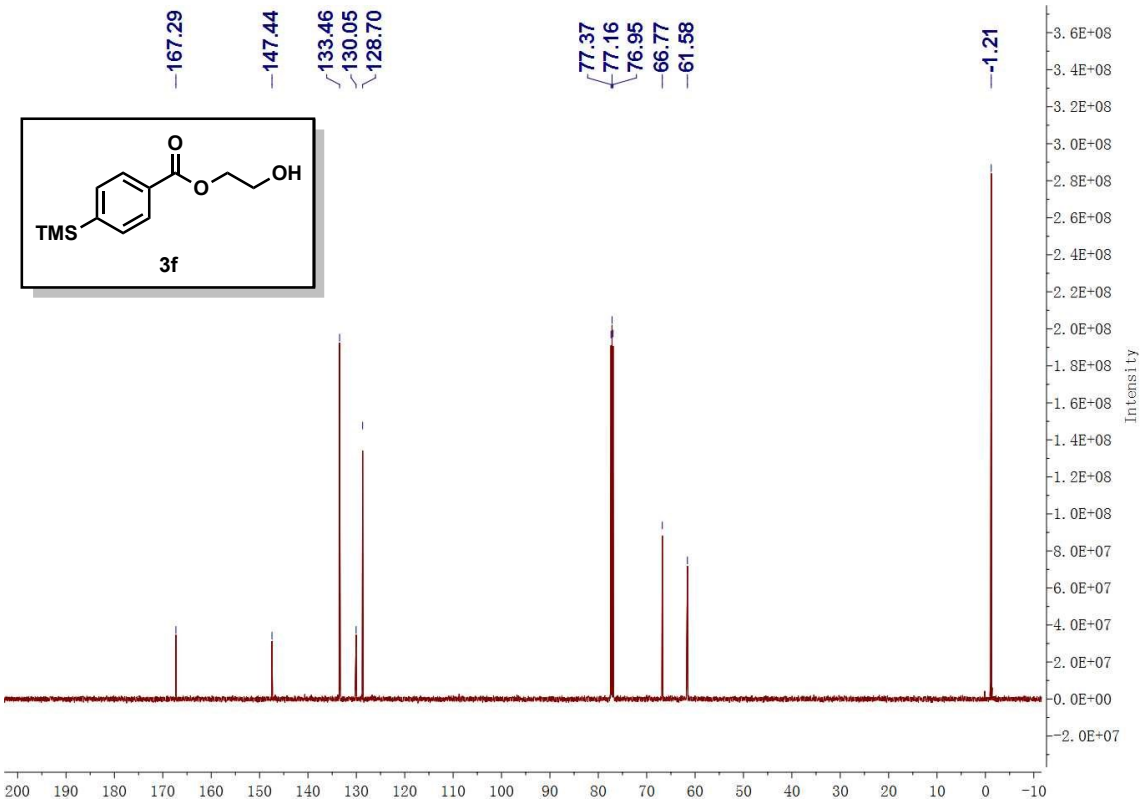

Figure S12. ^{13}C NMR of **3f**.

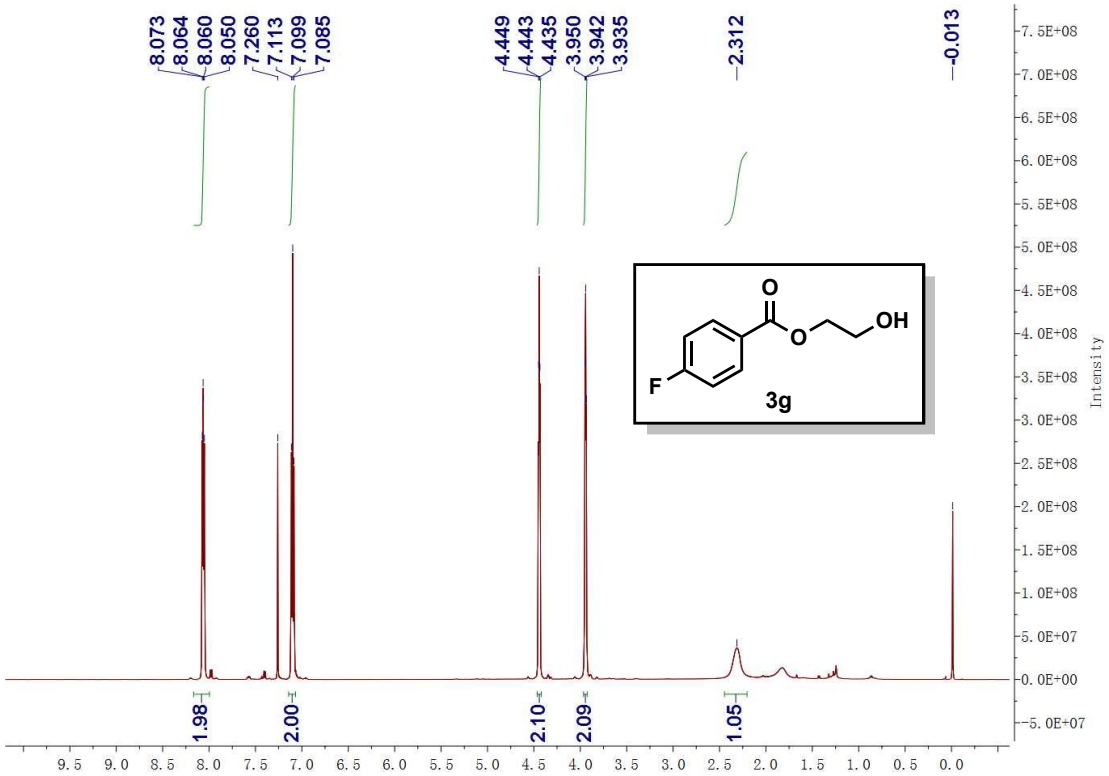

Figure S13. ^{1}H NMR of **3g**

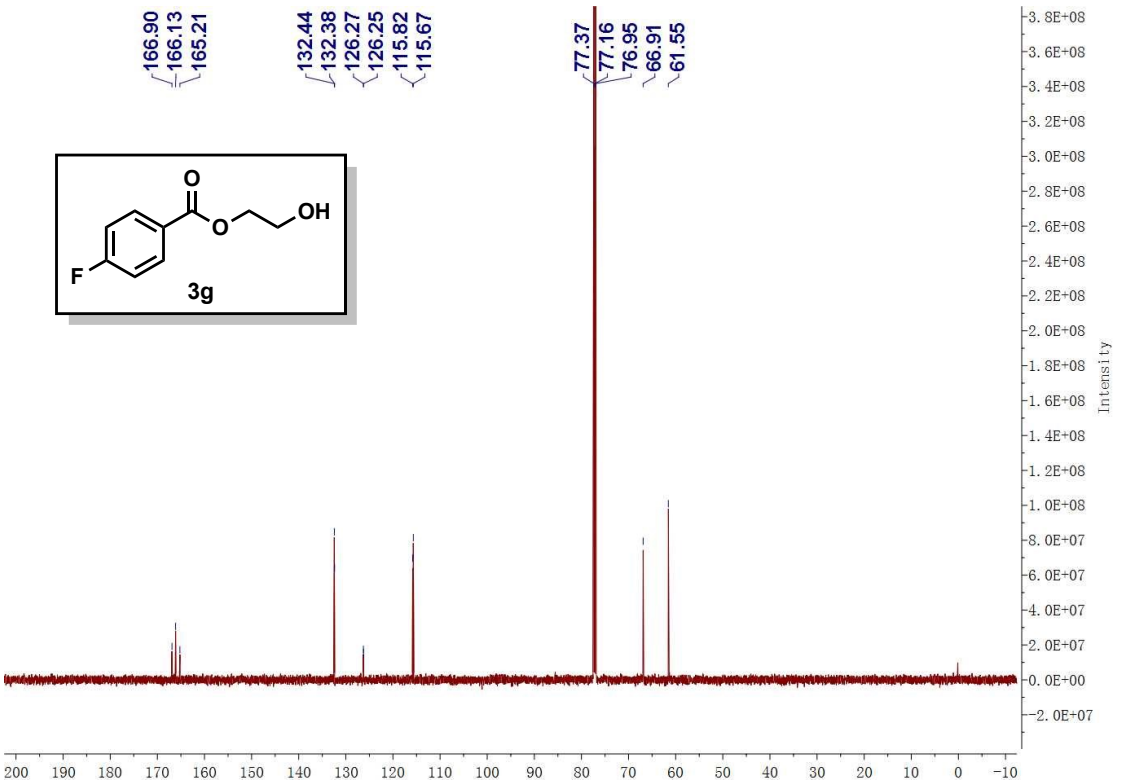

Figure S14. ^{13}C NMR of **3g**.

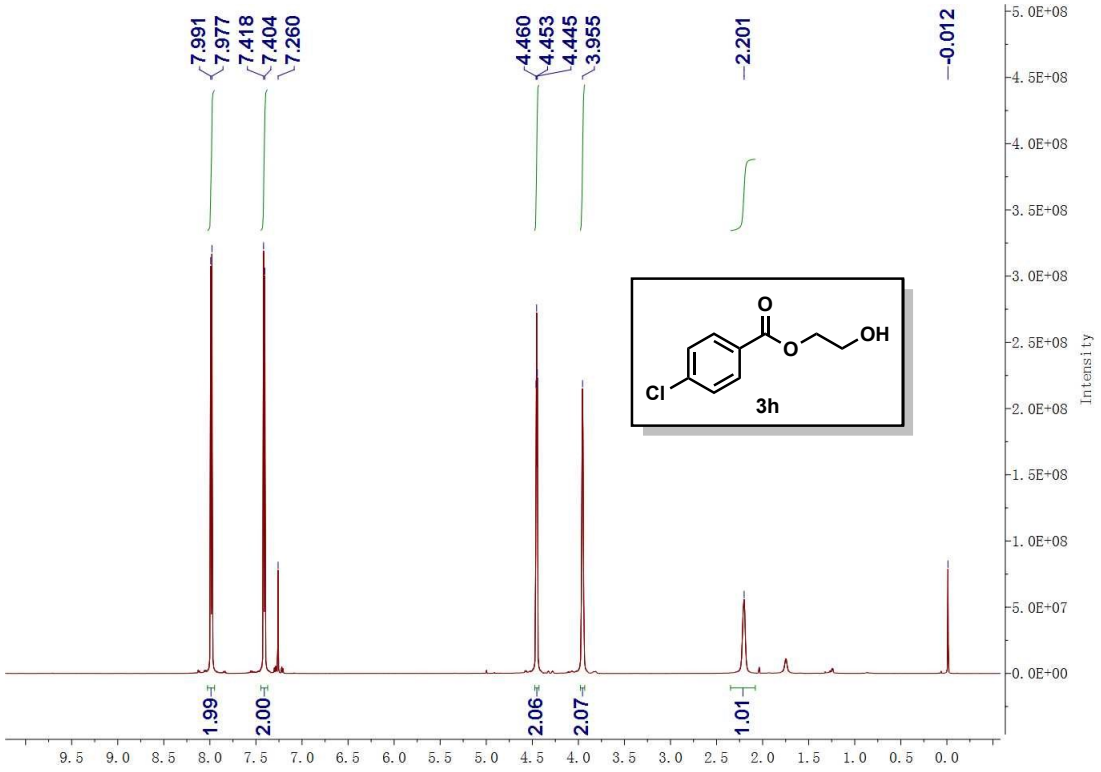

Figure S15. ^{1}H NMR of **3h**.

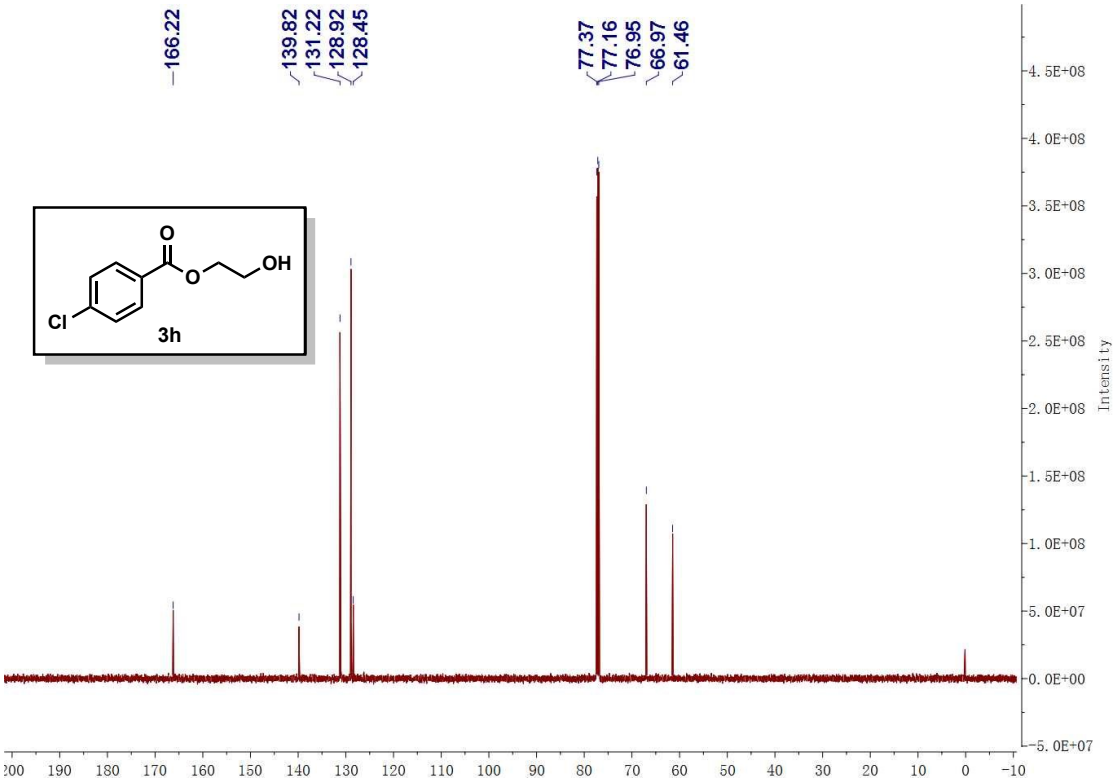

Figure S16. ^{13}C NMR of **3h**.

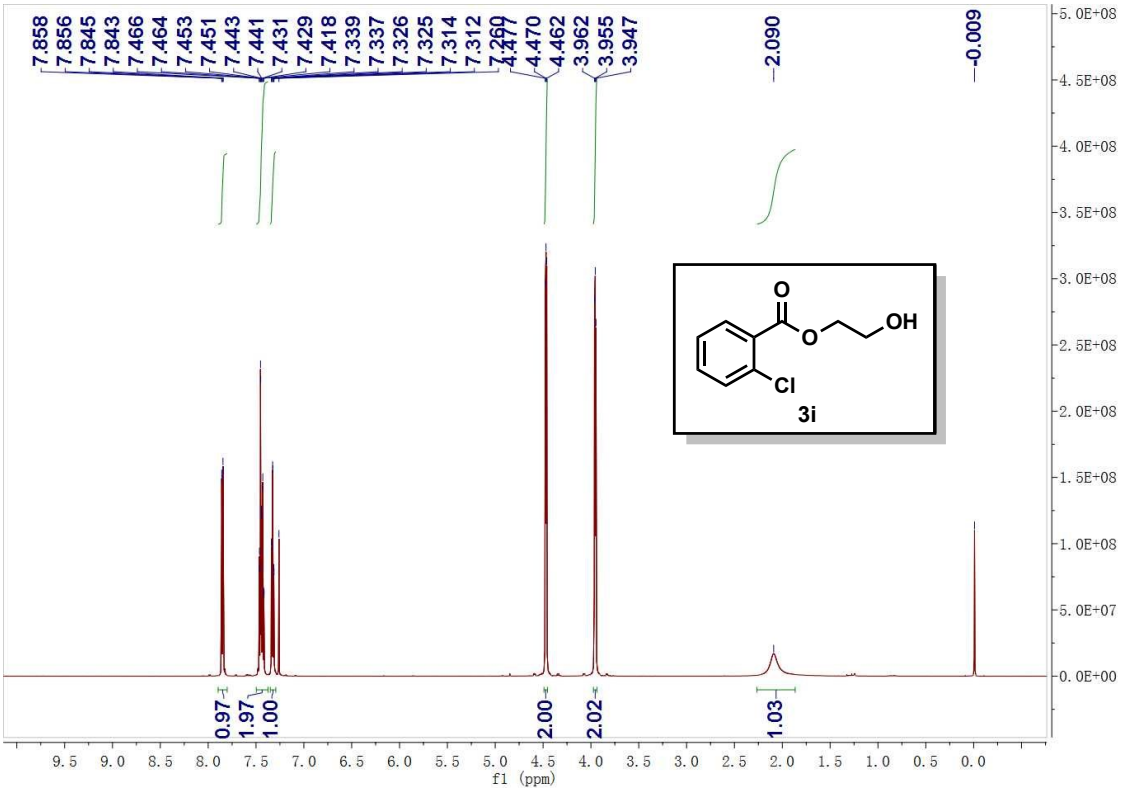

Figure S17. ^{1}H NMR of **3i**.

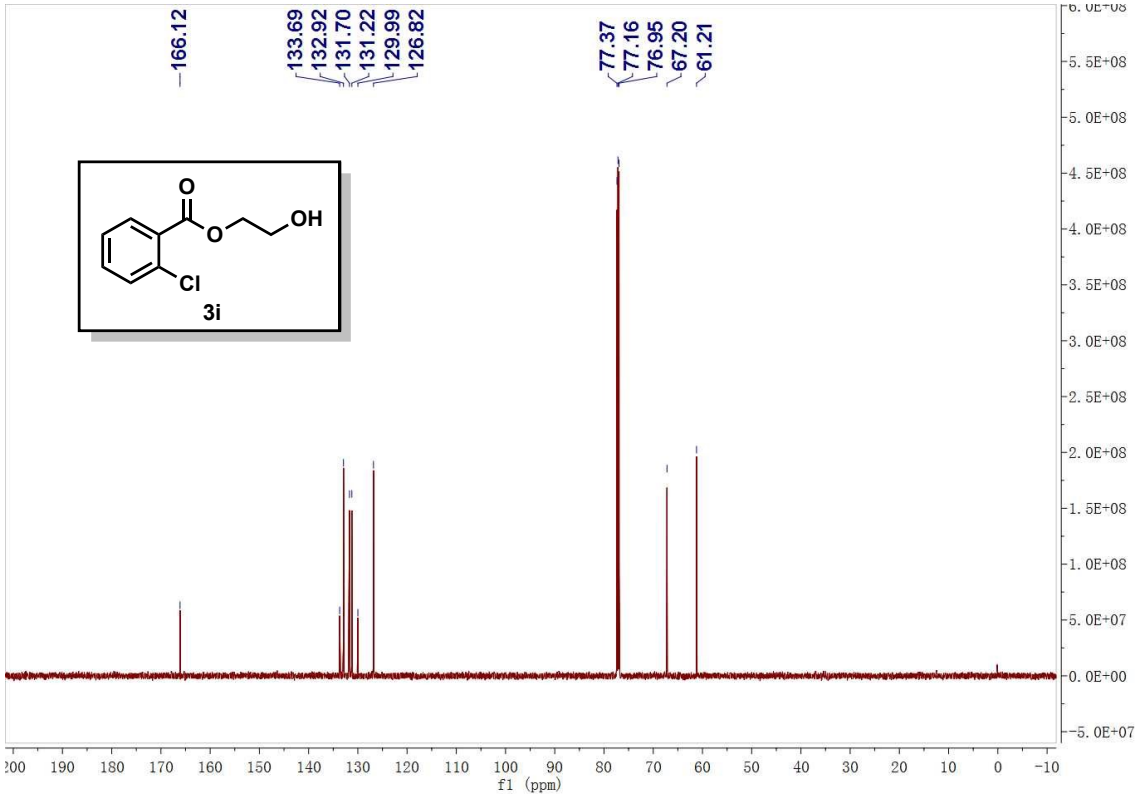

Figure S18. ^{13}C NMR of **3i**.

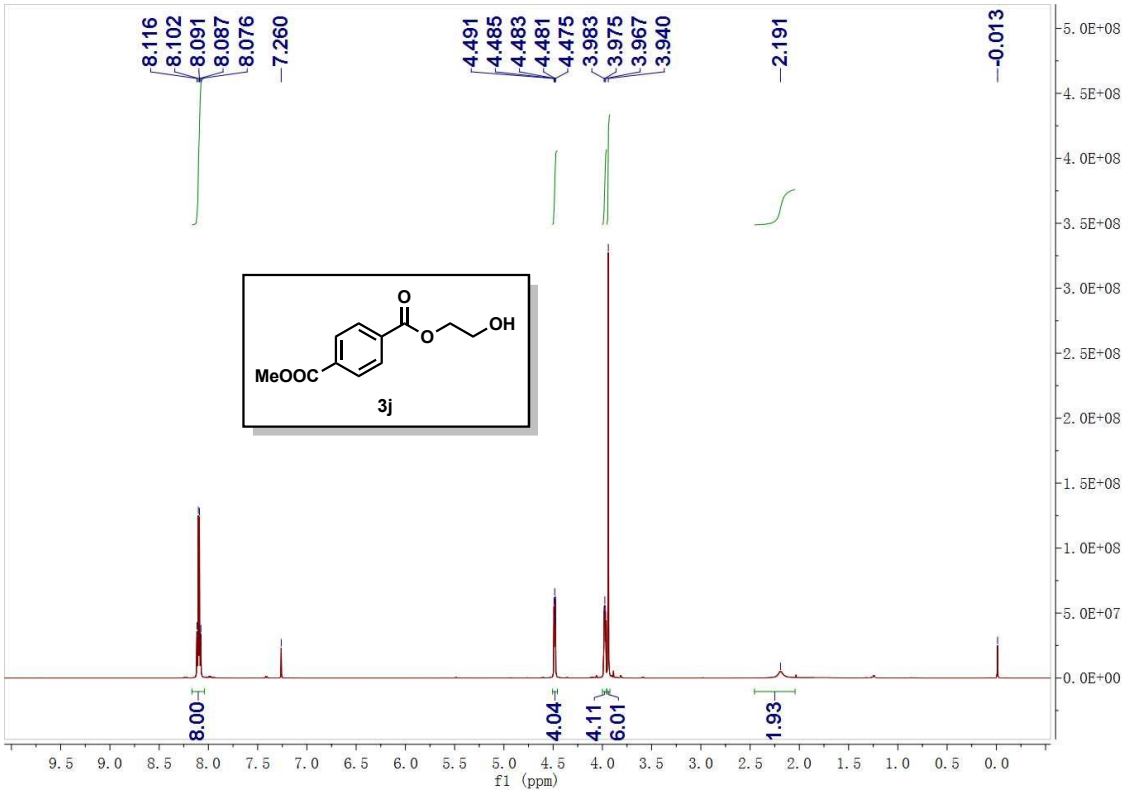

Figure S19. ^{1}H NMR of **3j**.

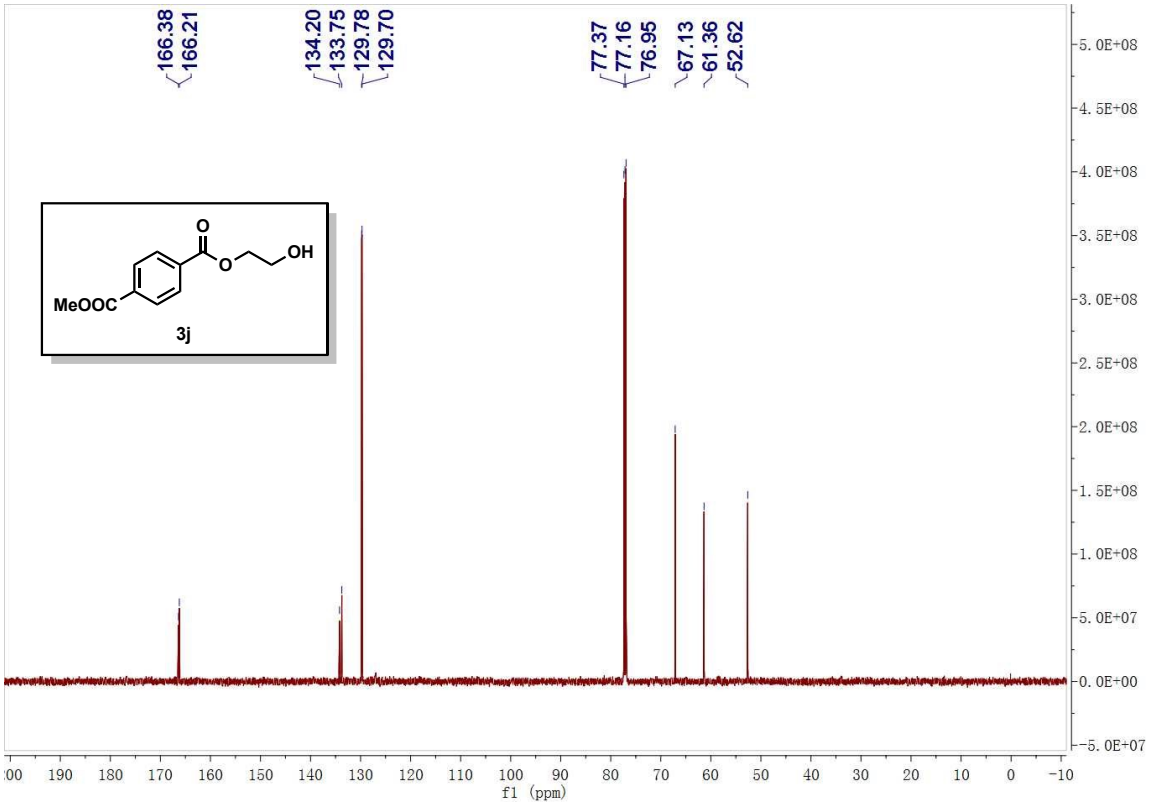

Figure S20. ^{13}C NMR of **3j**.

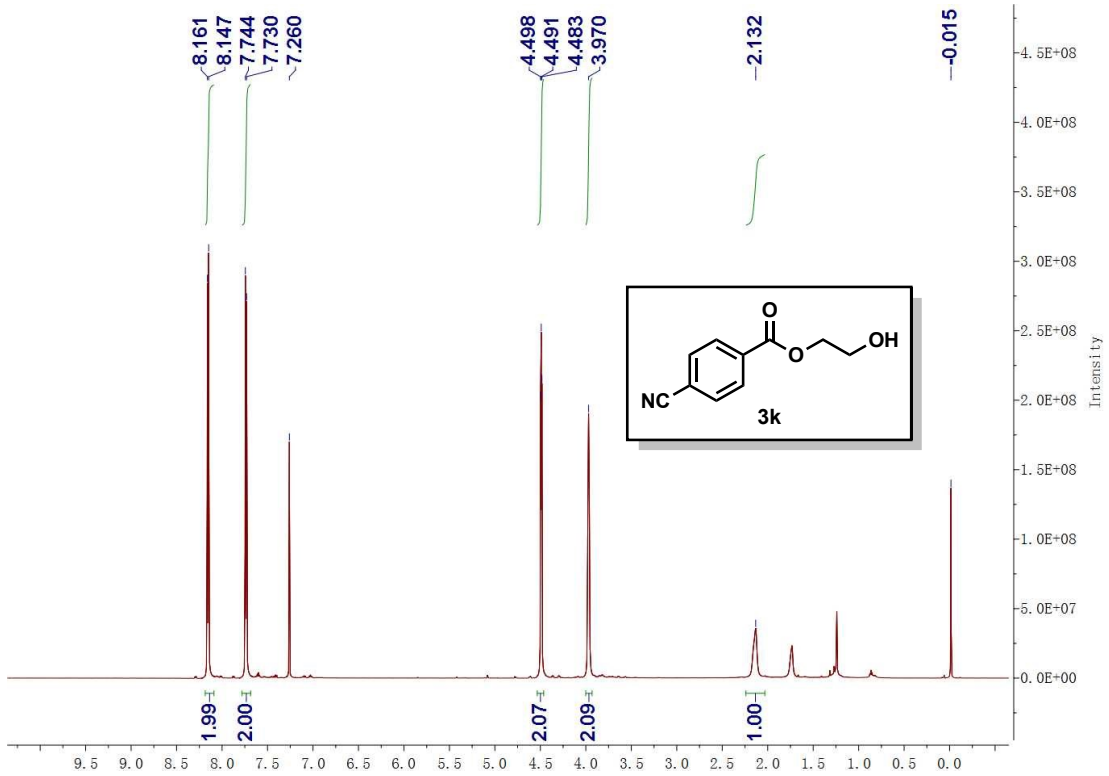

Figure S21. ^{1}H NMR of **3k**.

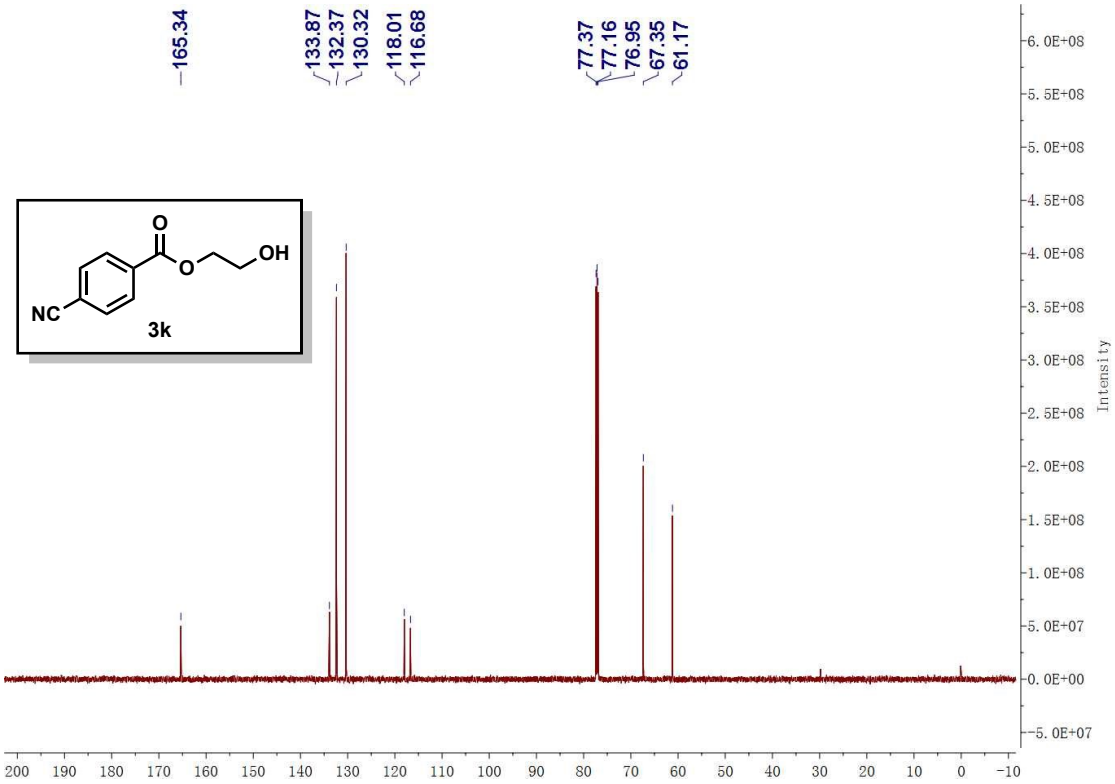

Figure S22. ^{13}C NMR of **3k**.

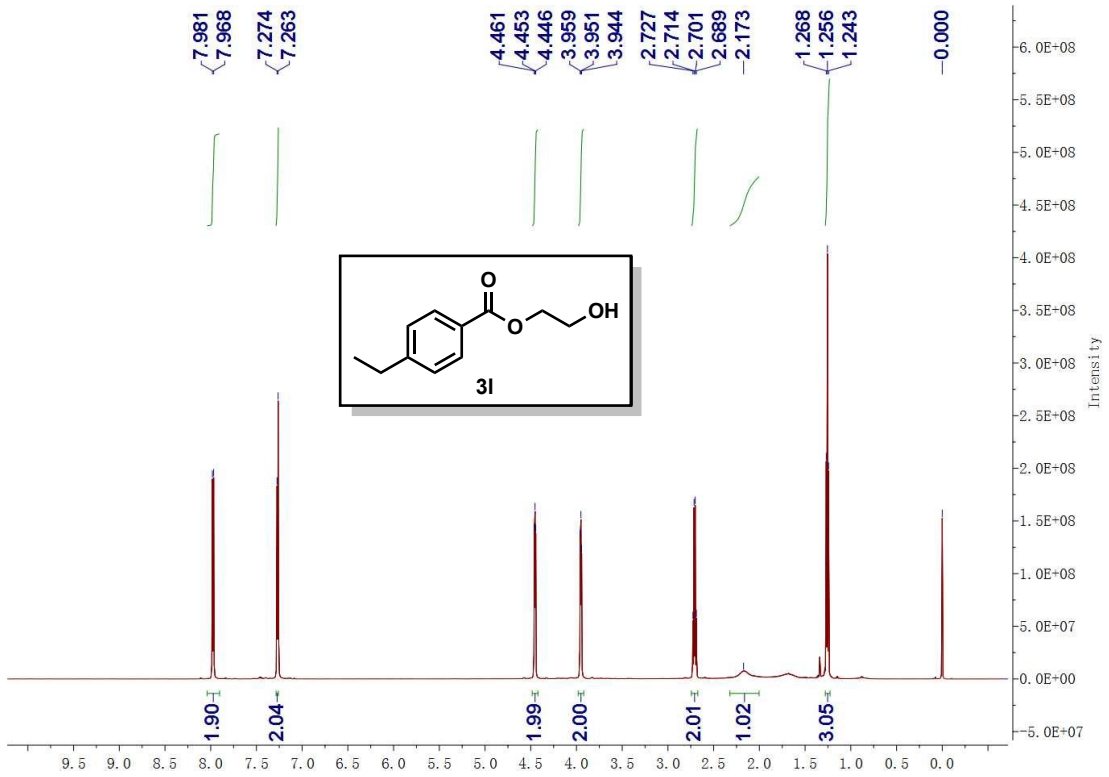

Figure S23. ^{1}H NMR of **3l.**

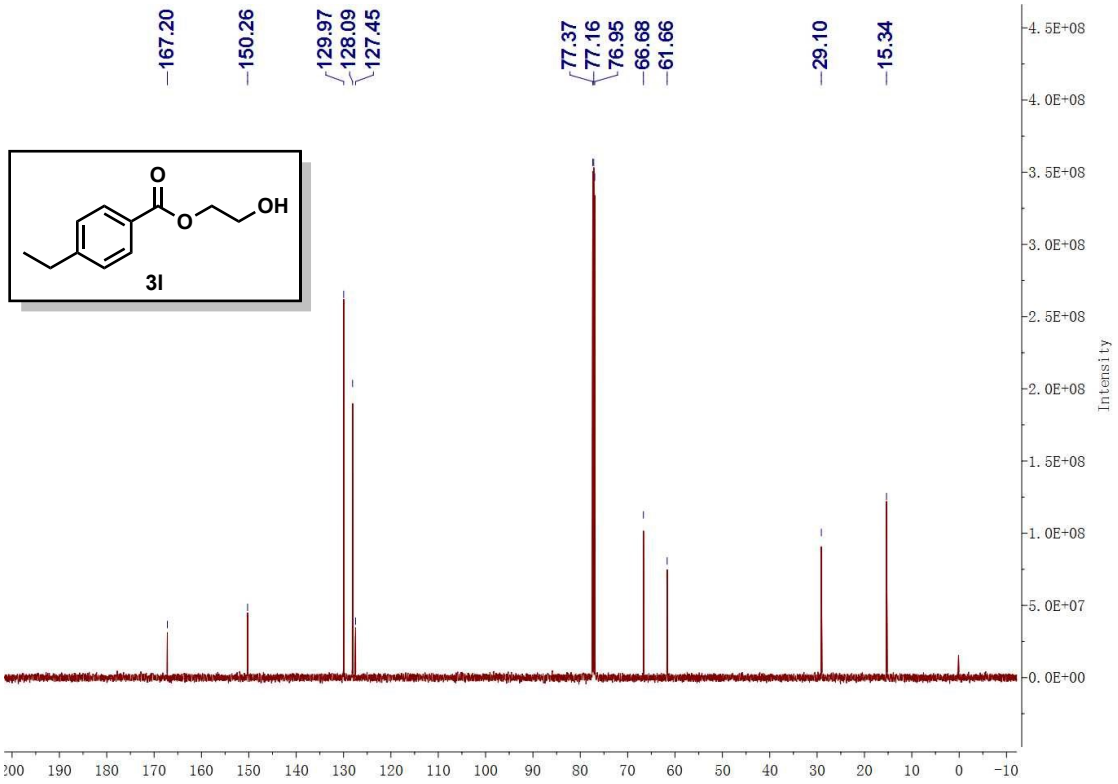

Figure S24. ^{13}C NMR of **3l.**

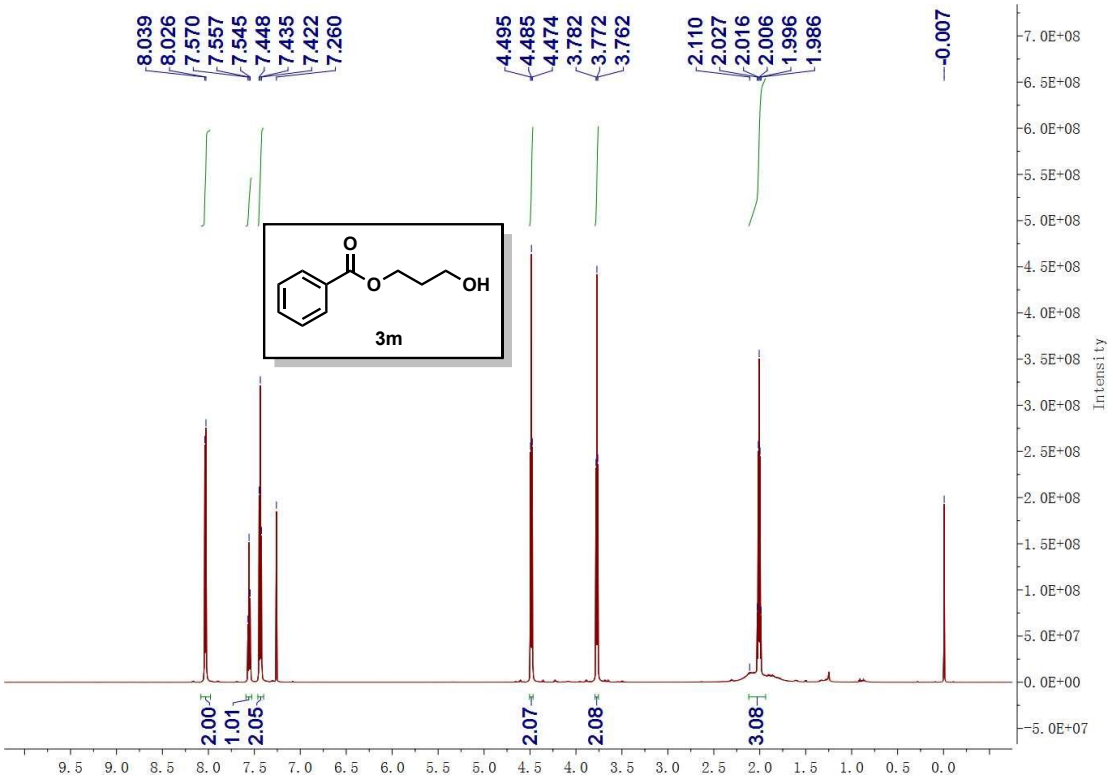

Figure S25. ^{1}H NMR of **3m**.

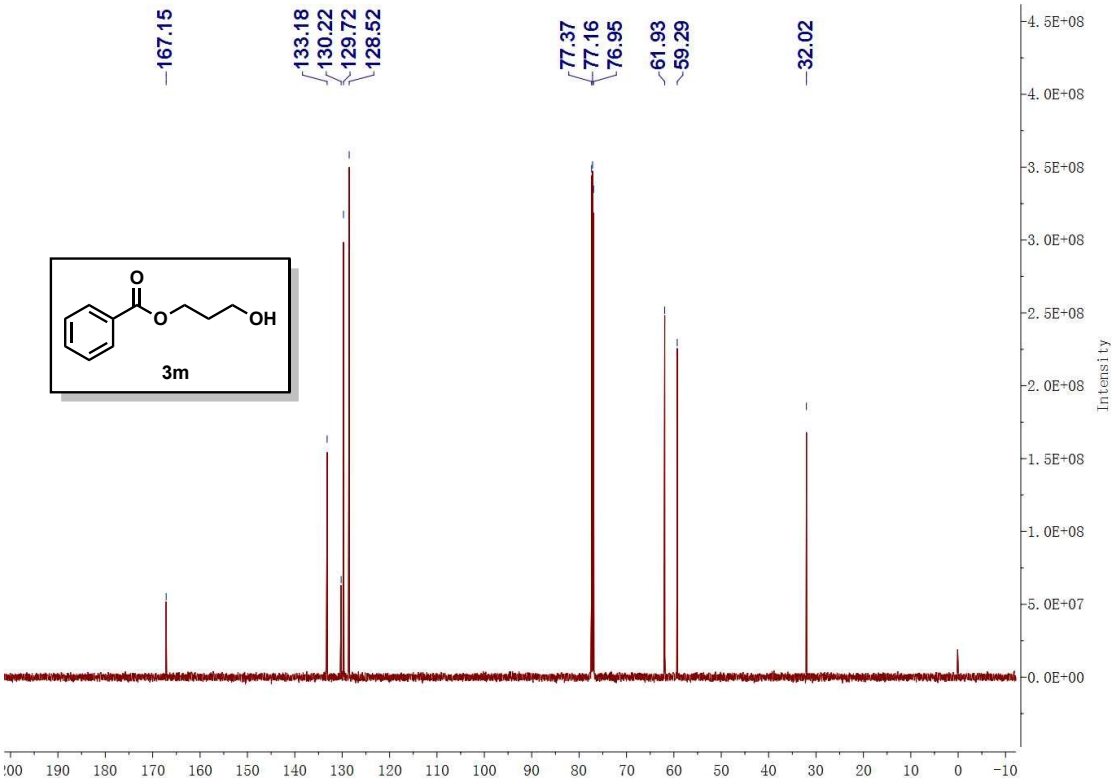

Figure S26. ^{13}C NMR of **3m**

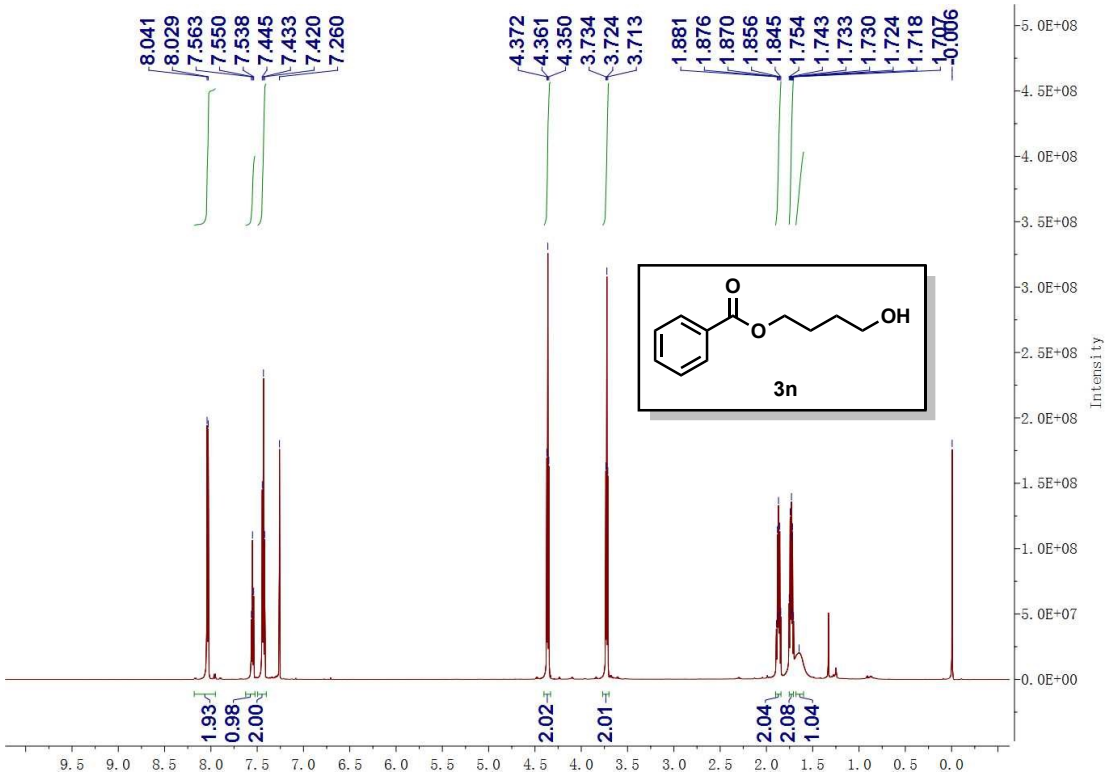

Figure S27. ^{1}H NMR of **3n**.

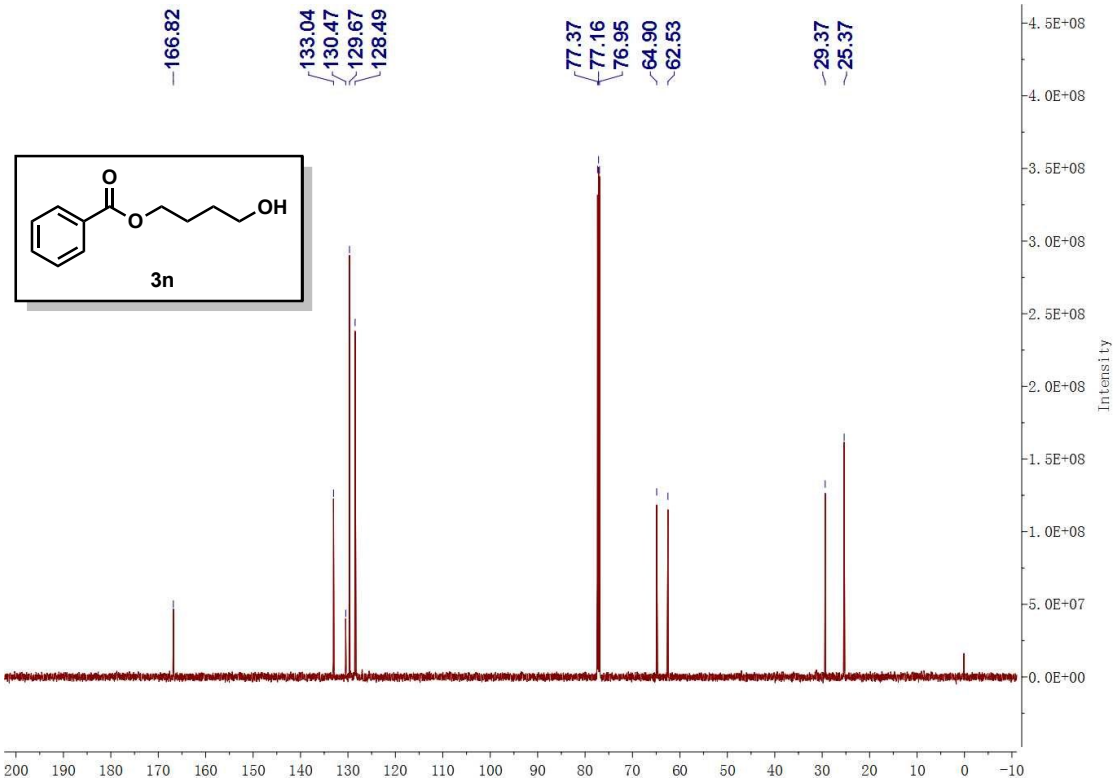

Figure S28. ^{13}C NMR of **3n**.

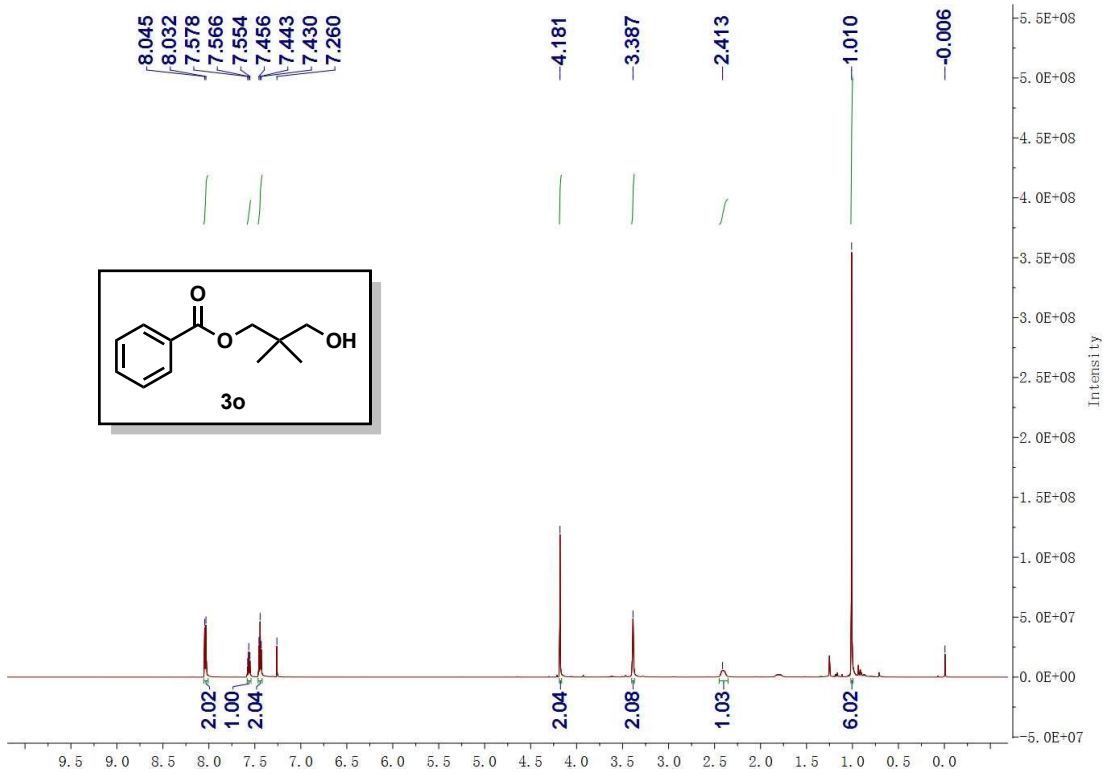

Figure S29. ^{1}H NMR of **3o**.

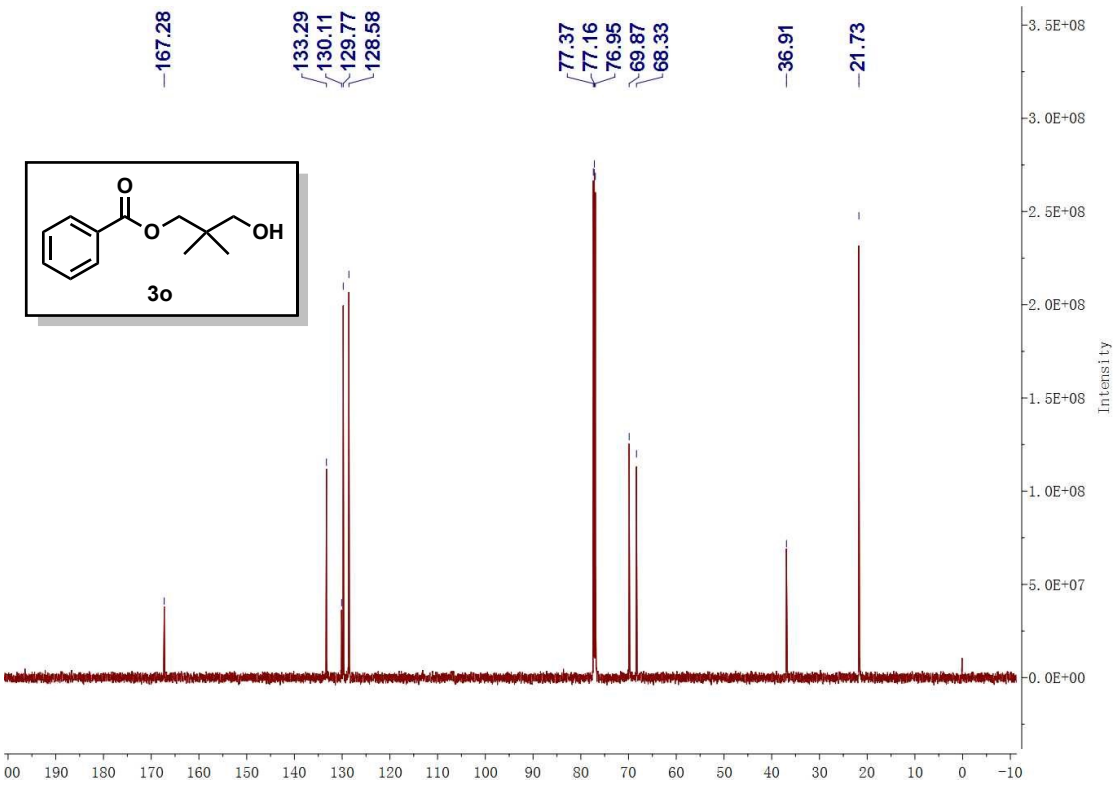

Figure S30. ^{13}C NMR of **3o**.

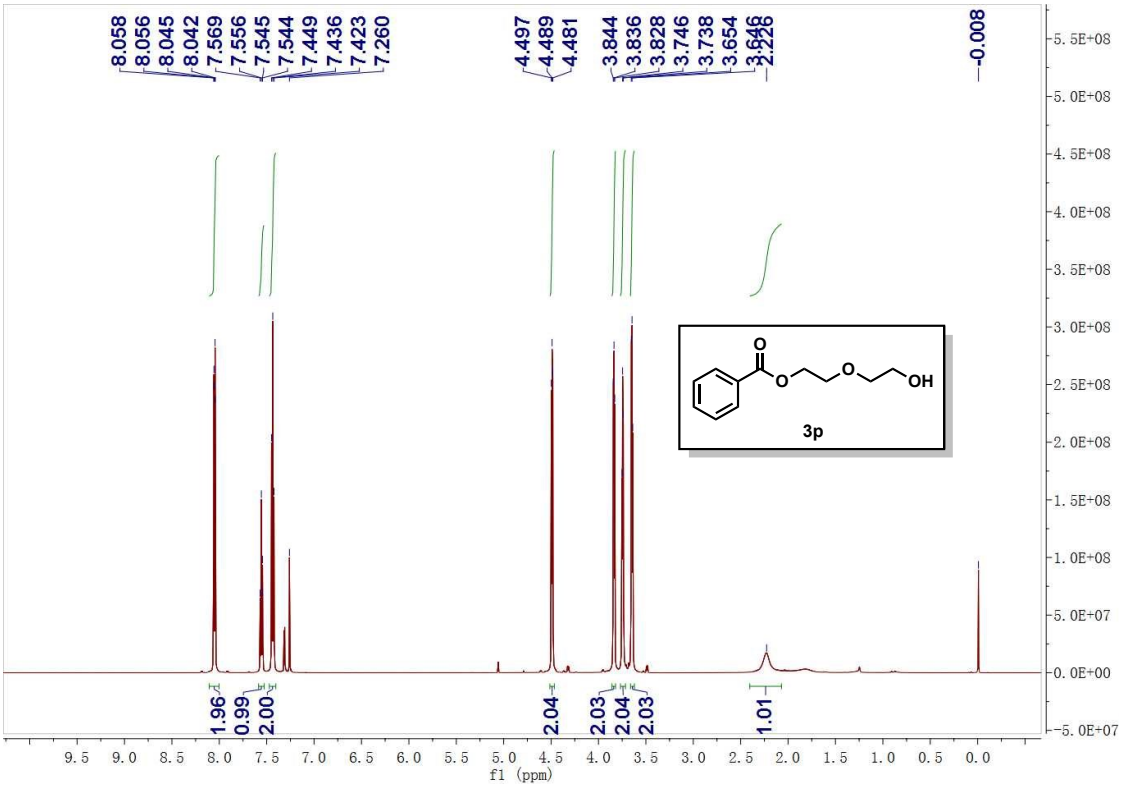

Figure S31. ^{1}H NMR of **3p**.

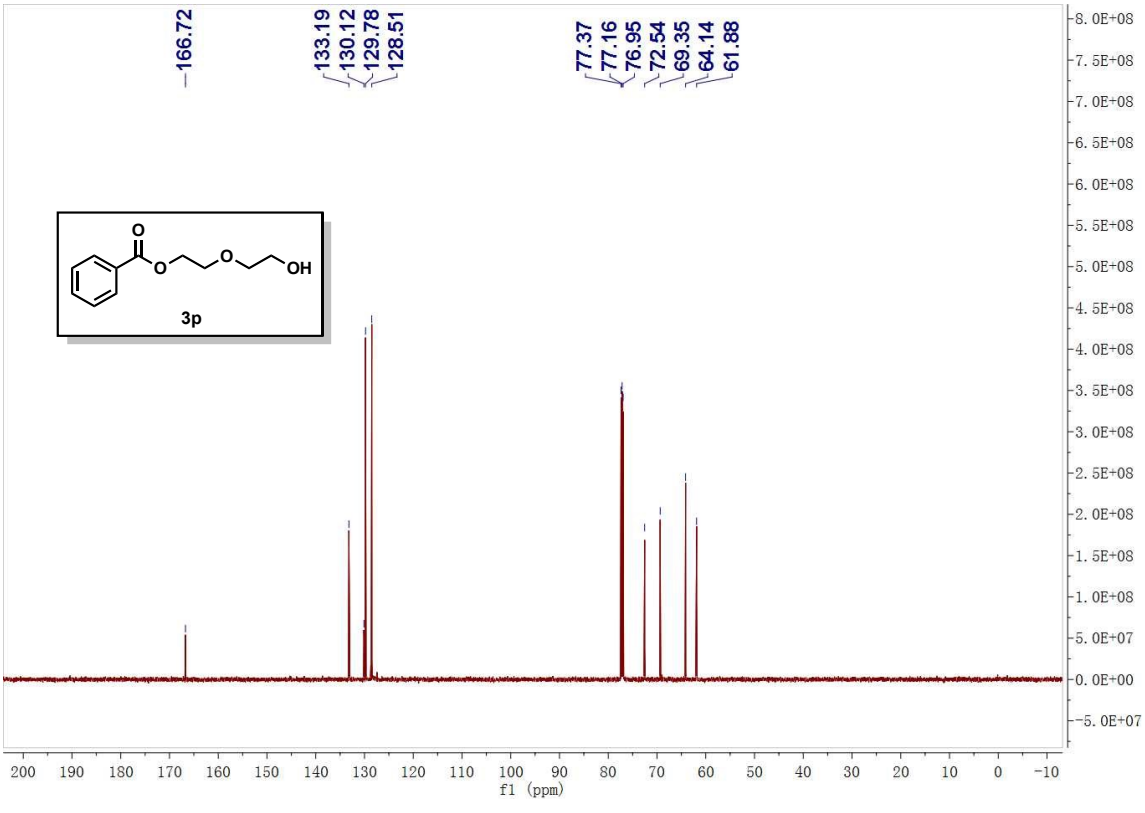

Figure S32. ^{13}C NMR of **3p**.

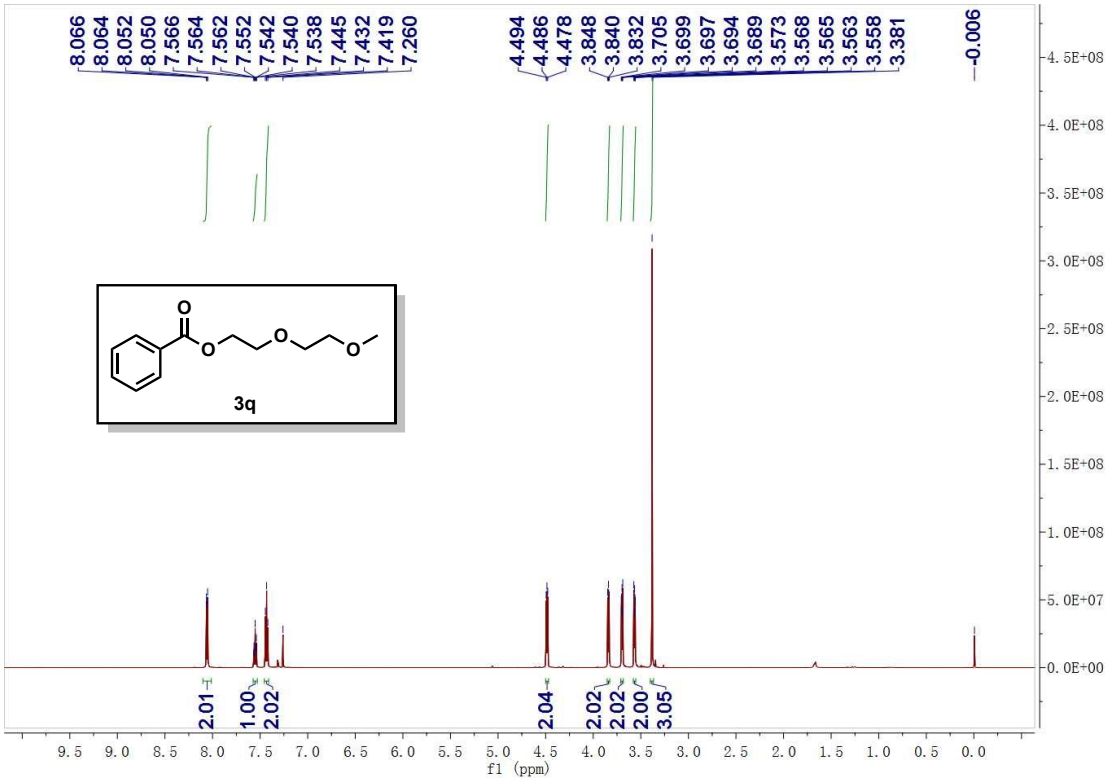

Figure S33. ^{1}H NMR of **3q**.

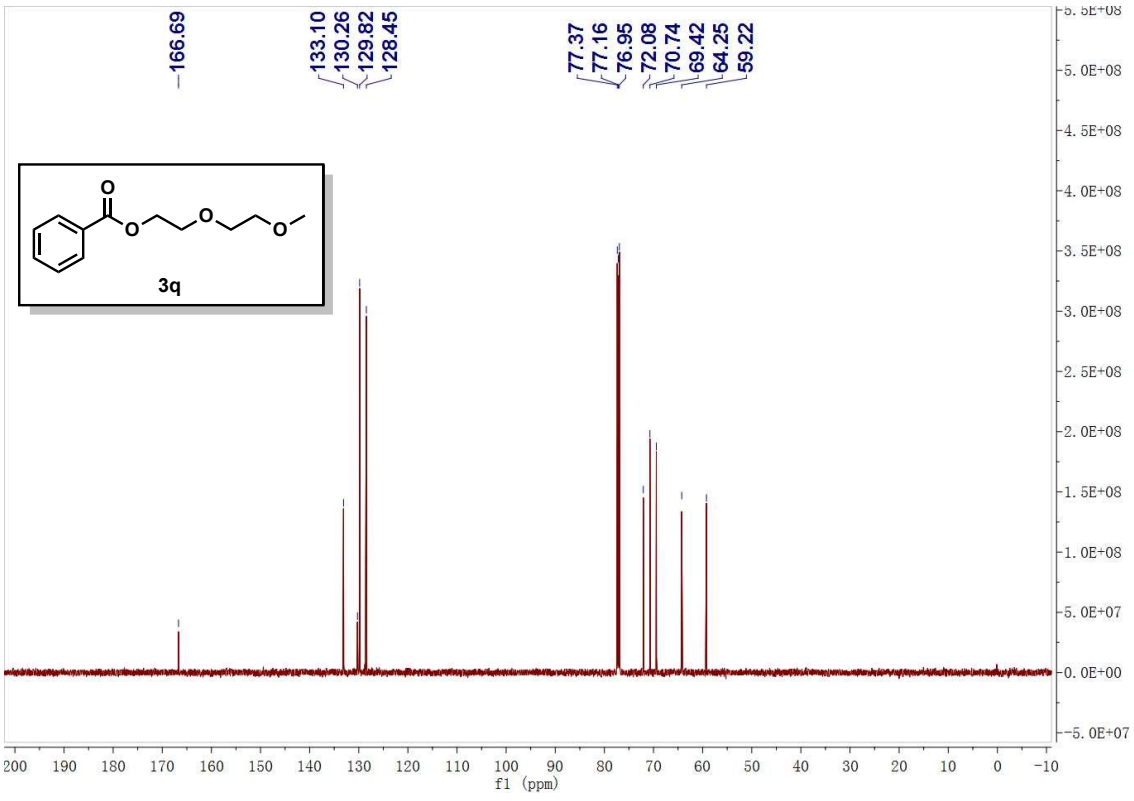

Figure S34. ^{13}C NMR of **3q**.

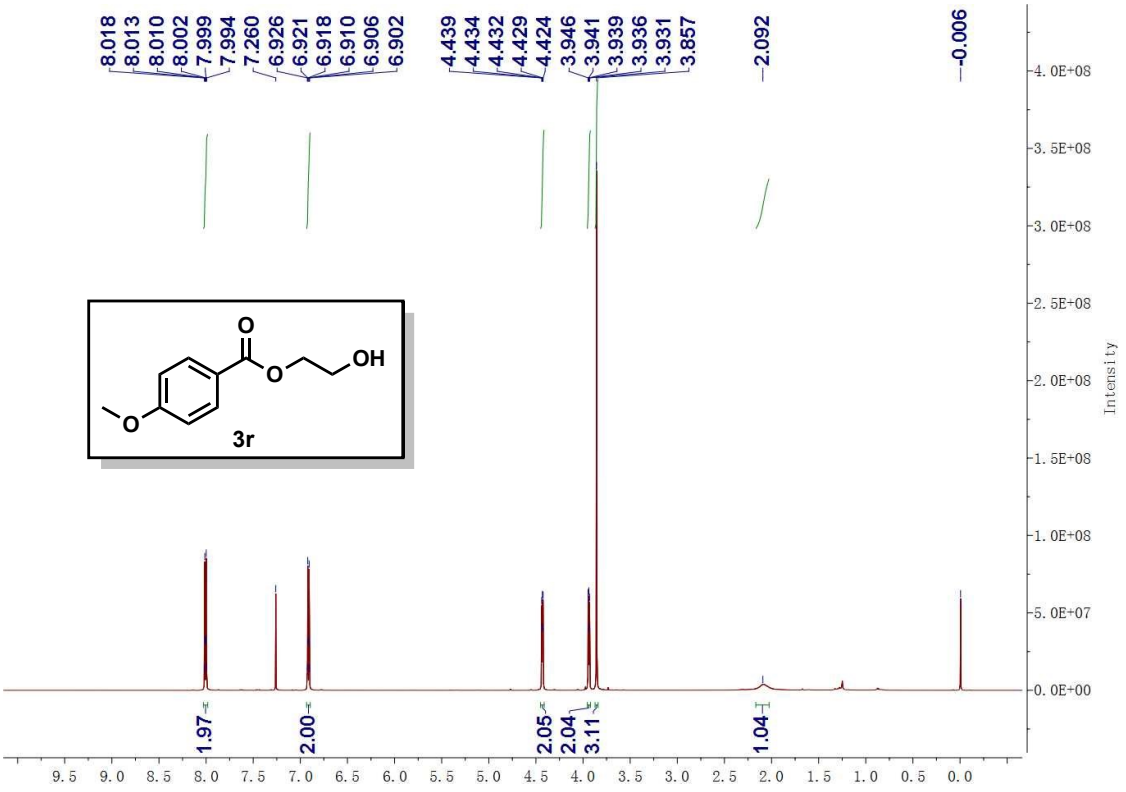

Figure S35. ^{1}H NMR of **3r**.

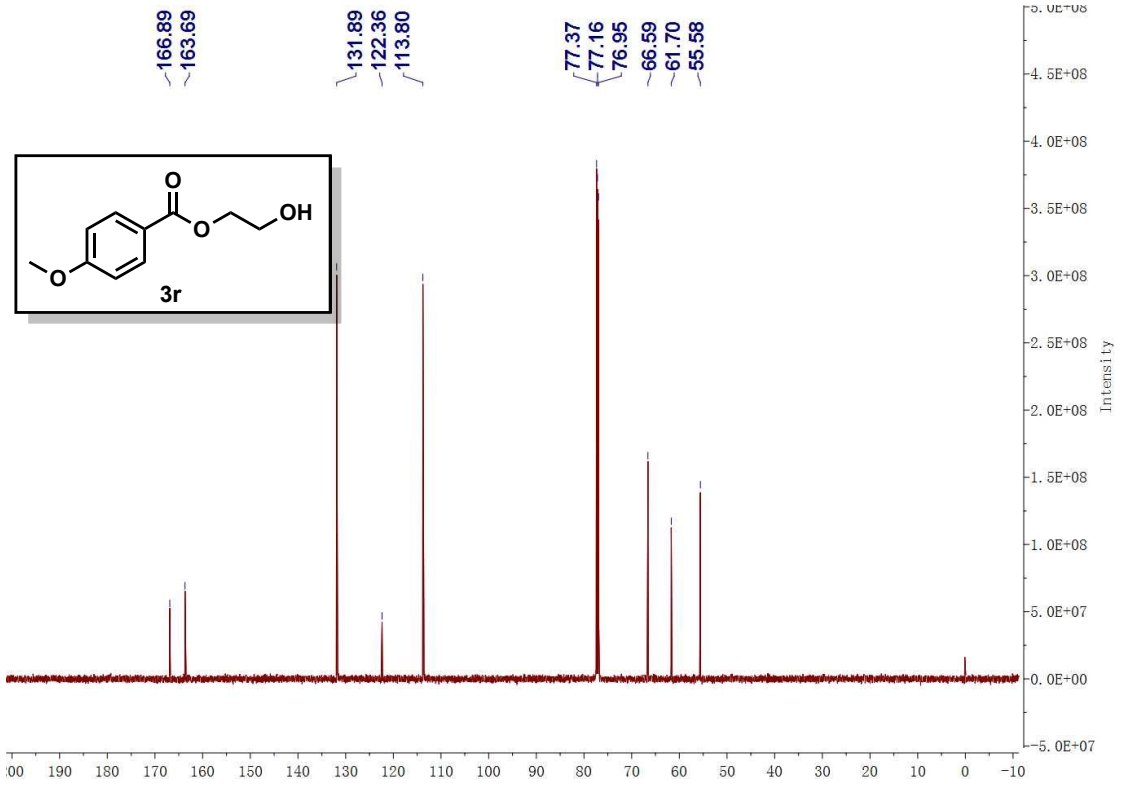

Figure S36. ^{13}C NMR of **3r**.

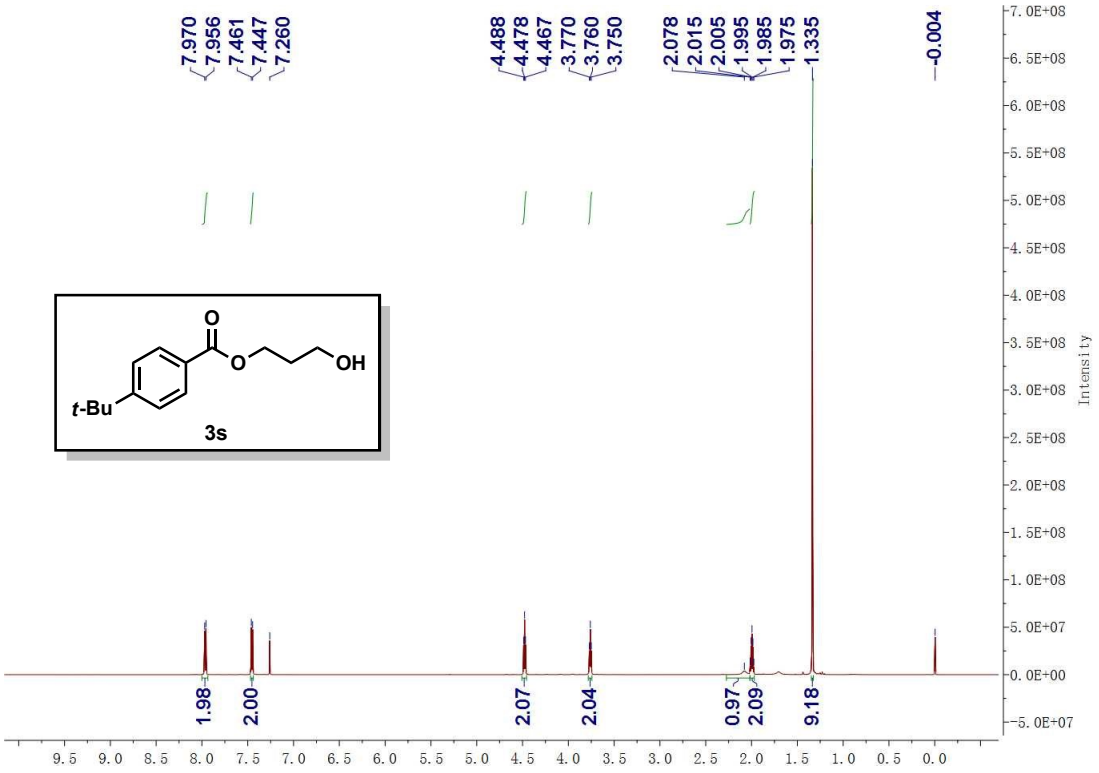

Figure S37. ^{1}H NMR of **3s**.

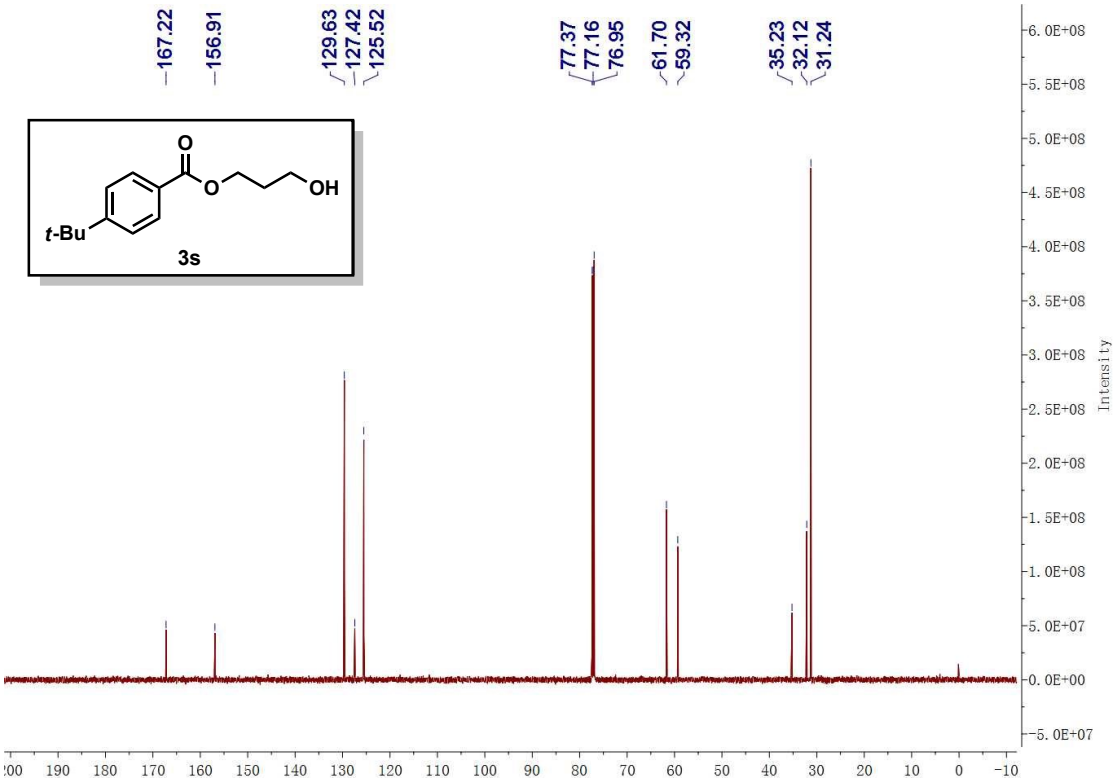

Figure S38. ^{13}C NMR of **3s**.

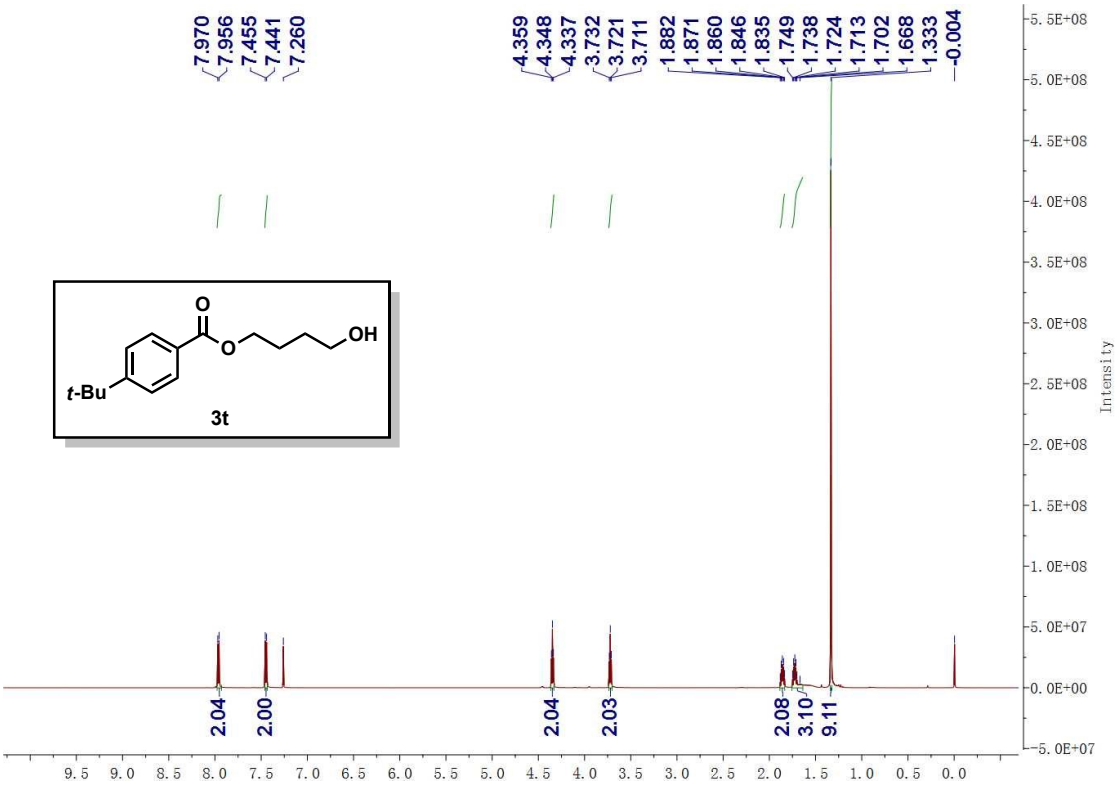

Figure S39. ^{1}H NMR of **3t**.

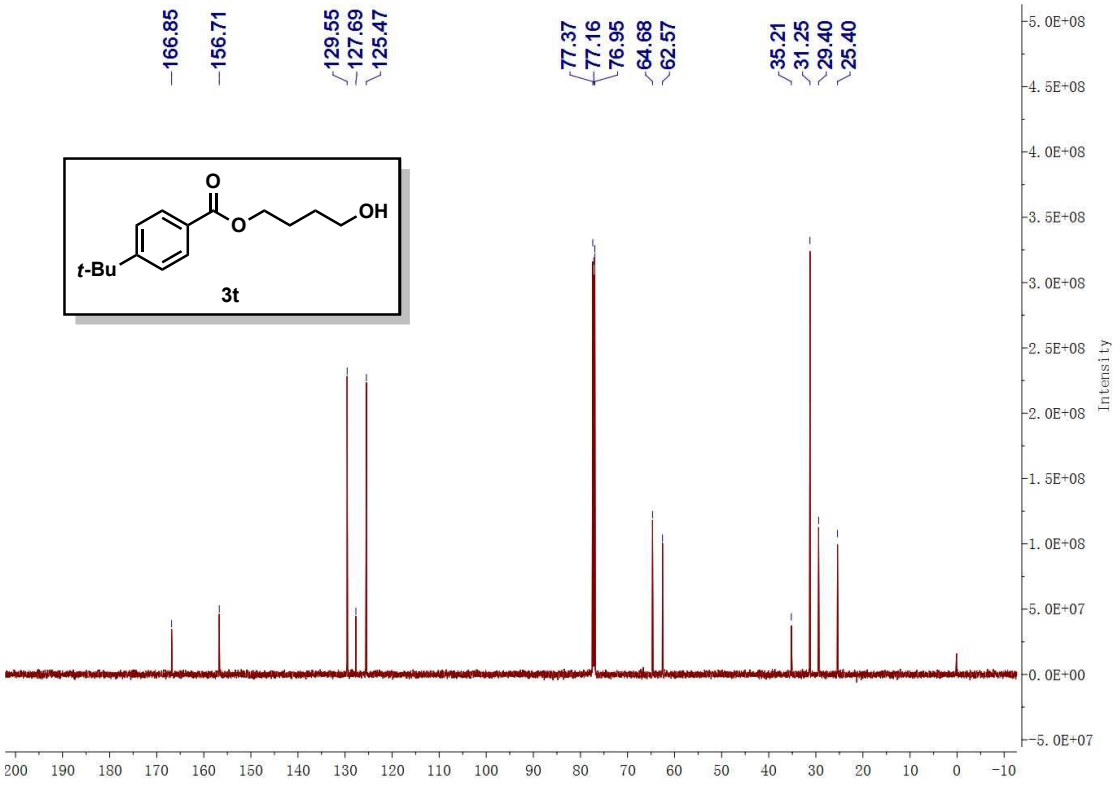

Figure S40. ^{13}C NMR of **3t**.

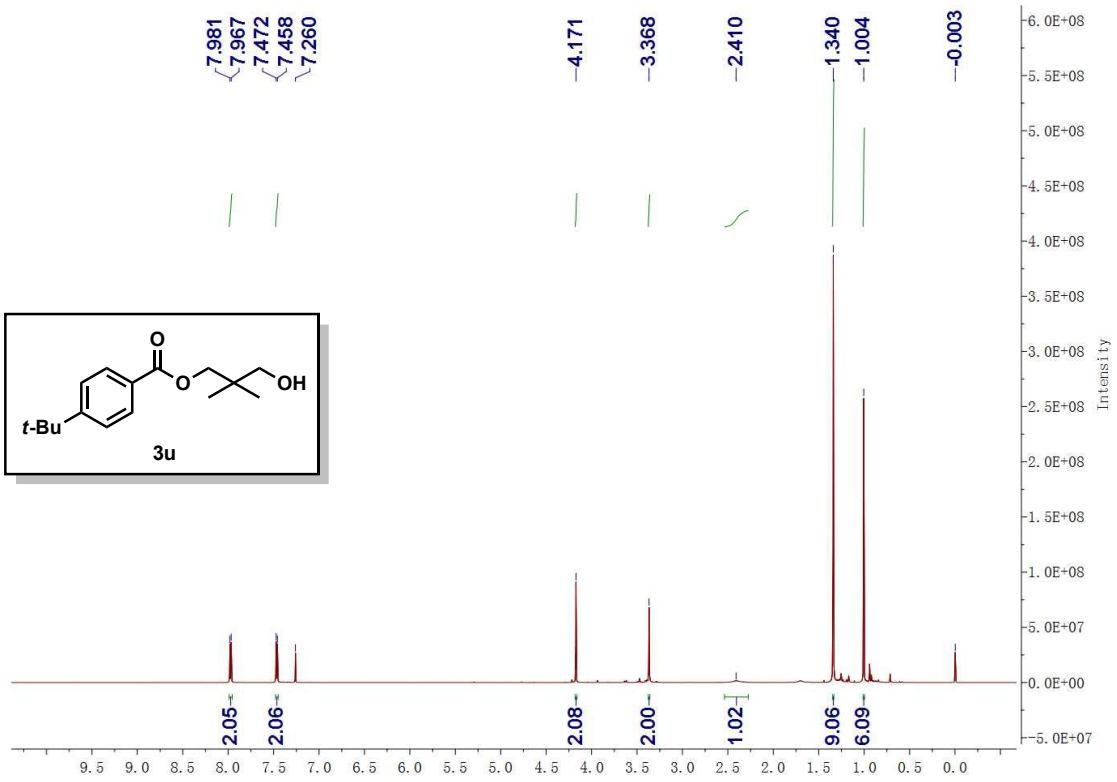

Figure S41. ^{1}H NMR of **3u**.

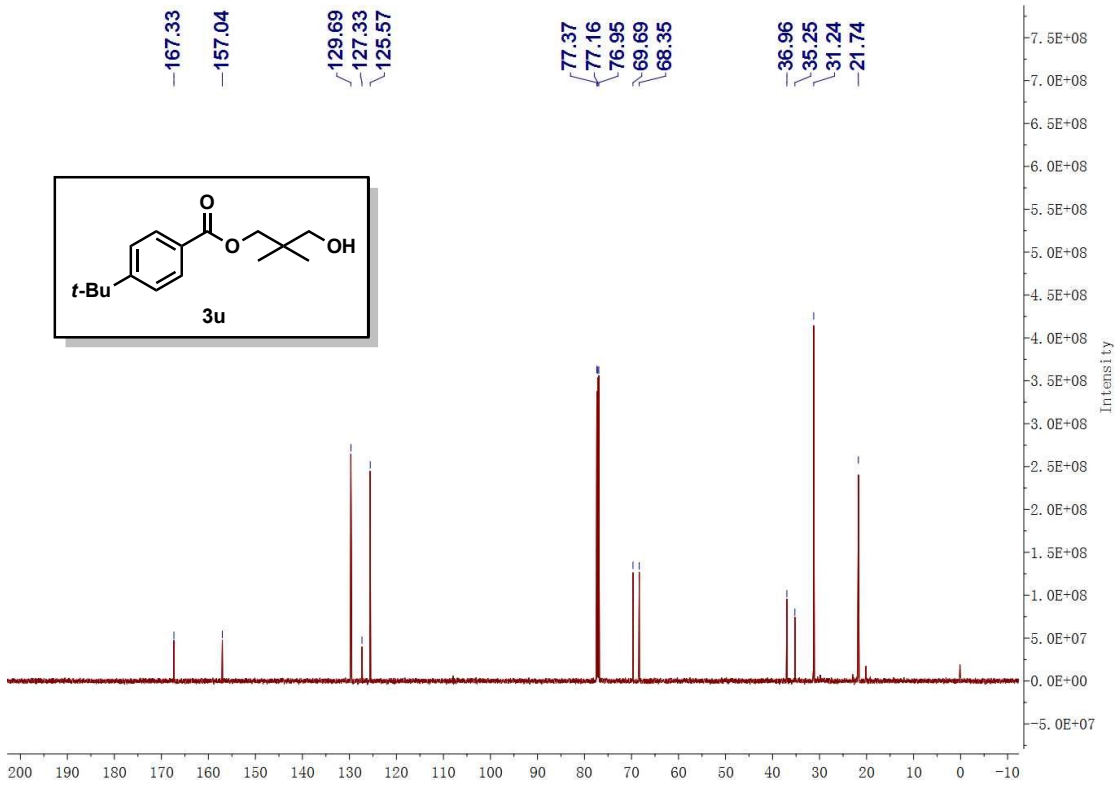

Figure S42. ^{13}C NMR of **3u**.