Electronic Supplementary Material (ESI) for Green Chemistry.
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Supplementary information for

Visible-light induced metal-free cascade Wittig/hydroalkylation reactions

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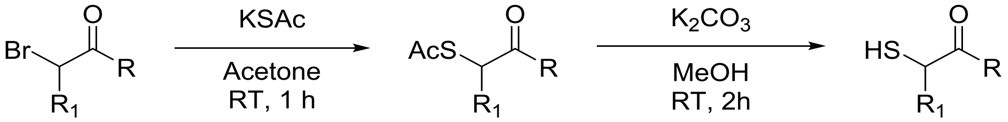
1. General information

All chemical reagents were purchased from commercial suppliers and used without further purification. The solvents used in this method, methylene chloride and dimethyl sulfoxide, are ultra-dry solvents containing molecular sieves ($H_2O \leq 50$ ppm). Two-neck bottles used in this experiment were baked at 125 °C for at least two hours before use. All the reactions were run under nitrogen atmosphere unless otherwise stated.

The products were purified by column chromatography using force-flow chromatography on silica gel (200-300 mesh). Thin-layer chromatography (TLC) was visualized by fluorescence quenching or iodine stain. All compounds were characterized by ^{1}H NMR, ^{13}C NMR, FT-IR and HRMS. All NMR spectra were recorded on an Agilent NMR Systems 400 MHz Spectrometer The ^{1}H NMR spectra of the compounds were measured at 400 MHz and are internally referenced to residual protic $CDCl_3$ (δ 7.26 ppm). ^{13}C NMR spectra were measured at 101 MHz and data are reported referenced to $CDCl_3$ (δ 77.0 ppm, the middle peak). ^{19}F NMR spectra were measured at 376 MHz. Infrared spectra (FT-IR) were recorded by Thermo Scientific™ Nicolet iS5 Fourier transform Spectrometer equipped with ATR. HRMS: electrospray ionization mass (ESI-MS) was performed on Agilent 6210 Series TOF MS. The 55W household CFL lamp was purchased online from JD.COM directly. The manufacture is Foshan Lighting. The brand name is FSL. The type is 4U-55W-E27.

2. The synthesis of substrates

General protocol A for the synthesis of thiols



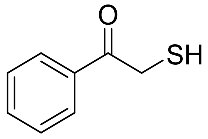
Supplementary Figure S1. synthesis of thiols from commercial compounds in two steps.

General procedure for synthesis of thioesters:

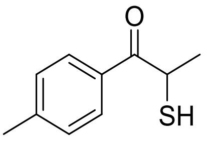
To a starting bromide (10 mmol) in acetone (100 mL) was added S-potassium thioacetate (1.2 equiv.) at room temperature. The reaction mixture was filtered after 1h and the solvent was removed *in vacuo*. The resulting residue was distributed between water (100 mL) and DCM (300 mL), and the combined organic layer was washed with brine, dried over Na_2SO_4, and concentrated in vacuo to give the thioacetate.

General procedure for synthesis of thiols:

To the round bottomed flask with K_2CO_3 (8 mmol) in MeOH (60 mL) under N_2 was added thioesters (6 mmol) by syringe. The reaction mixture was stirred 1 hour before it was quenched with HCl (25 mL, 1 M). The mixture was extracted with CH_2Cl_2 (150 mL), and the combined organic layer was washed with brine, dried over Na_2SO_4 and concentrated in vacuo. The desired thiols were obtained by flash chromatography.

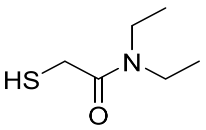


2-mercapto-1-phenylethan-1-one (a) was prepared from 2-bromo-1-phenylethan-1-one according to the general procedures as yellow oil (0.547 g, 60%), which was purified by flash chromatography (petroleum ether: ethyl acetate = 40: 1). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 – 7.93 (m, 2H), 7.62 – 7.54 (m, 1H), 7.53 – 7.46 (m, 2H), 3.96 (d, J = 7.3 Hz, 2H), 2.13 (t, J = 7.3 Hz, 1H). HRMS (ESI) calcd for C_8H_9OS $[M + H]^+$ m/z = 153.0369, found: 153.0367. Data were in agreement with a known literature[1].

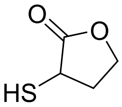


2-mercapto-1-(p-tolyl) propan-1-one (b) was prepared from 2-bromo-1-(p-tolyl) propan-1-one using the general procedures as yellow oil (0.81 g, 75%) without further purification. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.84 (d, J = 8.2 Hz, 2H), 7.23 (d, J = 8.3 Hz, 2H), 4.34 (dq, J = 9.5, 6.8 Hz, 1H), 2.37 (s, 3H), 1.99 (d, J = 9.5 Hz, 1H), 1.56 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$)

δ 197.4, 144.2, 132.2, 129.5, 128.8, 36.6, 21.7, 21.0. IR ν_{max}/cm^{-1} (film): 2926, 1674, 1606, 1370, 1336, 1236, 1182, 946, 830, 751. HRMS (ESI) calcd for $C_{10}H_{13}OS$ [M + H] $^+$ m/z = 181.0682, found: 181.0682.

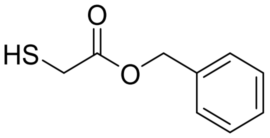


***N, N*-diethyl-2-mercaptoacetamide (c)** was prepared from 2-chloro-*N, N*-diethylacetamide according to the general procedures as colorless oil (0.57 g, 65% yield), which was purified by flash chromatography (petroleum ether: ethyl acetate = 2: 1). ^{1}H NMR (400 MHz, cdcl$_3$) δ 3.40 – 3.25 (m, 6H), 2.13 (t, *J* = 7.5 Hz, 1H), 1.19 (t, *J* = 7.2 Hz, 3H), 1.10 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 169.1, 42.7, 40.8, 26.3, 14.5, 12.9. HRMS (ESI) calcd for $C_6H_{14}NOS$ [M + H] $^+$ m/z = 148.0791, found: 148.0795. Data were in agreement with a known literature[2].



3-mercaptodihydrofuran-2(3*H*)-one (d) was prepared from 3-bromodihydrofuran-2(3*H*)-one using the general procedures as colorless oil (0.25 g, 35%), which was purified by flash chromatography (petroleum ether: ethyl acetate = 5: 1). ^{1}H NMR (400 MHz, CDCl$_3$) δ 4.41 (ddd, *J* = 9.1, 7.6, 5.9 Hz, 1H), 4.32 – 4.23 (m, 1H), 3.69 (ddd, *J* = 8.4, 6.9, 5.1 Hz, 1H), 2.73 – 2.61 (m, 1H), 2.30 (d, *J* = 5.1 Hz, 1H), 2.14 (m, 1H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 176.6, 66.5, 34.7, 31.9. HRMS (ESI) calcd for $C_4H_7O_2S$ [M + H] $^+$ m/z = 119.0161, found: 119.0161. Data were in agreement with a known literature[3].

Procedure for synthesis of benzyl 2-mercaptoacetate (e):

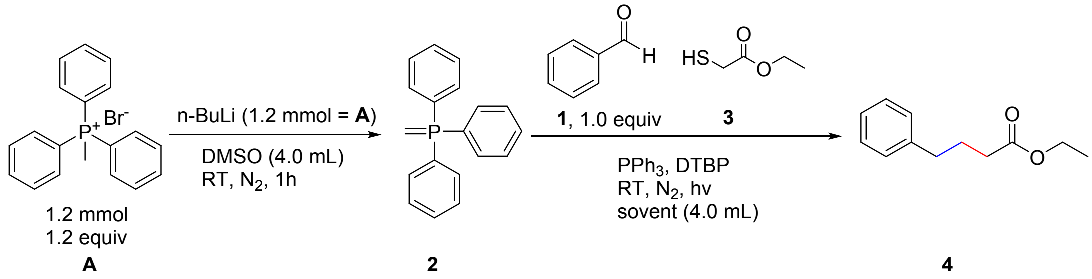


To a round bottomed flask with BnOH (2.06 mL, 20mmol) and 2-mercaptoacetic acid (1.66 mL, 24 mmol) in DCM (120 mL) was added *p*-Toluenesulfonic acid (0.3 equiv, 1.03 g, 6 mmol). The reaction mixture was stirred for 5 hours. The mixture was washed with saturated sodium bicarbonate solution (150 mL), brine (200 mL), dried over Na_2SO_4, concentrated and purified by flash chromatography (petroleum ether: ethyl acetate = 20: 1) to give the benzyl 2-mercaptoacetate as colorless oil (2.29 g, 63%).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.45 – 7.30 (m, 5H), 5.18 (s, 2H), 3.30 (d, *J* = 8.3 Hz, 2H), 2.03 (t, *J* = 8.3 Hz, 1H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 170.7, 135.4, 128.7, 128.5, 128.4, 67.4, 26.6. HRMS (ESI) calcd for $C_9H_{11}O_2S$ [M + H] $^+$ m/z =183.0474, found:183.0477. Data were in agreement with a known literature[2].

3. Optimization of the reaction condition

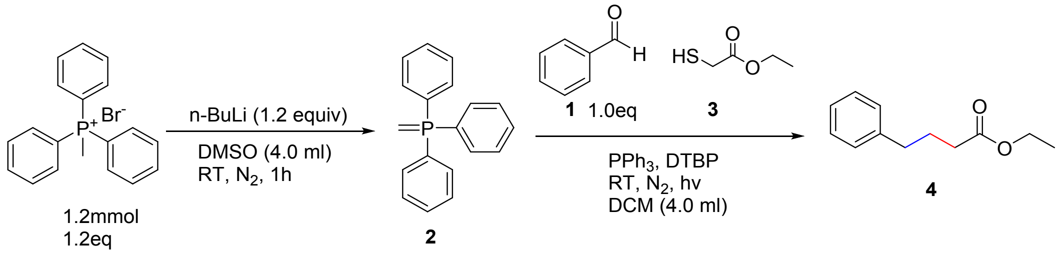
Table 1 | Optimization of reaction conditions [a].



entry	A	3	DTBP	PPh3	solvent	yield[b]
1	1.1 equiv	3.0 equiv	6.0 equiv	4.5 equiv	DMSO	trace
2	1.2 equiv	3.0 equiv	6.0 equiv	4.5 equiv	DMSO	32%
3	1.2 equiv	3.0 equiv	6.0 equiv	4.5 equiv	DCM	93%
4	1.2 equiv	2.0 equiv	4.0 equiv	2.5 equiv	DCM	93%
5	1.2 equiv	1.5 eqiuiv	3.0 equiv	1.9 equiv	DCM	88%
6[c]	1.2 equiv	2.0 equiv	4.0equiv	2.5 equiv	DCM	trace
7[d]	1.2 equiv	2.0 equiv	4.0equiv	2.5 equiv	DCM	N.D.

[a]Reaction conditions: 1 (1.2 mmol), n-BuLi (1.2 mmol), 4.0 ml ultra-dry DMSO, N_2 protection, room temperature, 1 h; then 2 (1.0 mmol), 3 (2.0 mmol), PPh_3 (2.5 mmol), DTBP (4.0 mmol), 4 mL ultra-dry DCM as another solvent, room temperature, 40 W household CFL bulb irradiation on two sides, 12 h. [b]Yields from ^{1}H-NMR, 1,3,5-trimethoxybenzene as the internal standard. N.D. = not detected. DTBP = di-tert-butyl peroxide. [c]Without DTBP. [d]Without light.

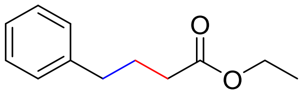
4. General procedure for the reaction



To a 25 mL oven-dried two necked bottle was added methyltriphenylphosphonium bromide (0.44 g, 1.2 mmol). The flask was degassed three times and protected with N_2 before ultra-dry dimethyl sulfoxide (4 mL) was added. n-Butyllithium (0.75 mL, 1.2 mmol, 1.6 M solution in Hexanes) was added and the mixture was stirred at room temperature for 1 hour. Triphenylphosphine (0.65 g, 2.5 mmol) was dissolved in ultra-dry dichloromethane (4 mL). Then aldehyde/ketone (1.0 mmol), thiol (2.0 mmol), triphenylphosphine solution and DTBP (0.73 mL, 4 mmol) were added into the reaction mixture in order by micro-syringe. The reaction was stirred and irradiated using two 55 W household CFL bulbs (6 cm away, to keep the reaction at room temperature) for 12 hours. When the reaction was complete, the mixture was extracted with EtOAc (3×15 mL), washed with water (2×5 mL) and brine (5 mL). The organic layer was dried with sodium sulfate, filtered and concentrated. The residue was purified by column chromatography on silica to give the product.

5. Characterization data for all key compounds

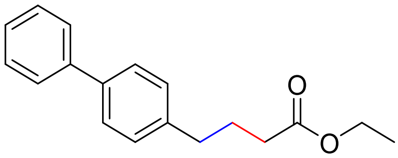
ethyl 4-phenylbutanoate **(4)**



Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73mL) in the reaction mixture. The desired product was obtained as colorless oil (93% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.25 (m, 2H), 7.22 – 7.17 (m, 3H), 4.14 (q, J = 7.1 Hz, 2H), 2.67 (t, J = 7.6 Hz, 2H), 2.33 (t, J = 7.5 Hz, 2H), 2.04 – 1.90 (m, 2H), 1.26 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.5, 141.5, 128.6, 128.5, 126.0, 60.3, 35.3, 33.8, 26.7, 14.3. HRMS (ESI) calcd for $C_{12}H_{16}NaO_2$ $[M + Na]^+$ m/z = 215.1043, found: 215.1042. Data were in agreement with a known literature[4].

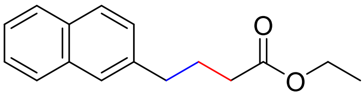
ethyl 4-([1,1'-biphenyl]-4-yl) butanoate **(5)**



Prepared following the general procedure employing 4-Biphenylcarboxaldehyde (182 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (95% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.63 – 7.58 (m, 2H), 7.55 (d, J = 8.1 Hz, 2H), 7.45 (t, J = 7.6 Hz, 2H), 7.35 (t, J = 7.3 Hz, 1H), 7.31 – 7.25 (m, 2H), 4.16 (q, J = 7.1 Hz, 2H), 2.72 (t, J = 7.6 Hz, 2H), 2.38 (t, J = 7.5 Hz, 2H), 2.08 – 1.97 (m, 2H), 1.29 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.6, 141.2, 140.7, 139.1, 129.0, 128.8, 127.2, 127.1, 127.1, 60.4, 34.9, 33.8, 26.6, 14.4. IR v_{max}/cm^{-1} (film): 2978, 2930, 1732, 1486, 1372, 1178, 1145, 1025, 761, 697. HRMS (ESI) calcd for $C_{18}H_{20}NaO_2$ $[M + Na]^+$ m/z = 291.1356, found: 291.1352.

ethyl 4-(naphthalen-2-yl) butanoate **(6)**



Prepared following the general procedure employing 3-Iodobenzaldehyde (140 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (90% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 100: 1).

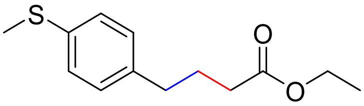
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.81 (t, J = 9.7 Hz, 3H), 7.64 (s, 1H), 7.46 (pd, J = 6.8, 1.3 Hz, 2H), 7.36 (dd, J = 8.4, 1.3 Hz, 1H), 4.15 (q, J = 7.1 Hz, 2H), 2.84 (t, J = 7.6 Hz, 2H), 2.38 (t, J = 7.5 Hz, 2H), 2.12 – 2.04 (m, 2H), 1.28 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.6, 139.0, 133.7, 132.2, 128.1, 127.7, 127.5, 127.3, 126.7, 126.0, 125.3, 60.4, 35.4, 33.8, 26.5, 14.4. HRMS (ESI) calcd for $C_{16}H_{18}NaO_2$ [M + Na]$^+$ m/z = 265.1199, found: 265.1199. Data were in agreement with a known literature[4].

ethyl 4-(p-tolyl) butanoate **(7)**

Prepared following the general procedure employing p-Tolualdehyde (120 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (95% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.16 – 7.01 (m, 4H), 4.12 (q, J = 7.1 Hz, 2H), 2.65 – 2.57 (m, 2H), 2.35 – 2.27 (m, 5H), 1.99 – 1.88 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.61 138.5, 135.5, 129.2, 128.5, 60.3, 34.8, 33.8, 26.8, 21.1, 14.4. HRMS (ESI) calcd for $C_{13}H_{19}O_2$ [M + H]$^+$ m/z = 207.1380, found: 207.1380. Data were in agreement with a known literature[4].

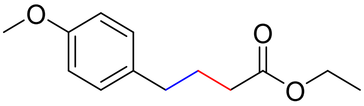
ethyl 4-(4-(methylthio) phenyl) butanoate **(8)**



Prepared following the general procedure employing 4-(methylthio) benzaldehyde (152 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (93% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.22 – 7.17 (m, 2H), 7.13 – 7.07 (m, 2H), 4.12 (q, J = 7.1 Hz, 2H), 2.61 (t, J = 7.6 Hz, 2H), 2.46 (s, 3H), 2.30 (t, J = 7.5 Hz, 2H), 1.97 – 1.86 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.5, 138.7, 135.6, 129.1, 127.3, 60.4, 34.7, 33.7, 26.6, 16.4, 14.4. IR ν_{max}/cm^{-1} (film): 3307, 2979, 2922, 2852, 1731, 1493, 1199, 1144, 1025, 798. HRMS (ESI) calcd for $C_{13}H_{19}O_2S$ [M + H]$^+$ m/z = 239.1100, found: 239.1102.

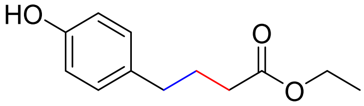
ethyl 4-(4-methoxyphenyl) butanoate **(9)**



Prepared following the general procedure employing p-Anisaldehyde (136 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73mL) in the reaction mixture. The desired product was obtained as colorless oil (95% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.12 – 7.07 (m, 2H), 6.86 – 6.80 (m, 2H), 4.12 (q, J = 7.1 Hz, 2H), 3.77 (s, 3H), 2.59 (t, J = 7.6 Hz, 2H), 2.30 (t, J = 7.5 Hz, 2H), 1.97 – 1.87 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.5, 158.0, 133.5, 129.4, 113.8, 60.2, 55.2, 34.3, 33.6, 26.8, 14.3. HRMS (ESI) calcd for $C_{13}H_{18}NaO_3$ [M + Na]$^+$ m/z =245.1148, found: 245.1155. Data were in agreement with a known literature[4].

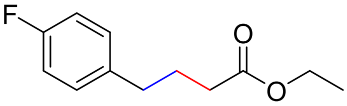
ethyl 4-(4-hydroxyphenyl) butanoate **(10)**



Prepared following the general procedure employing p-Hydroxybenzaldehyde (122 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (94 mg, 45%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 5: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.06 – 6.98 (m, 2H), 6.79 – 6.72 (m, 2H), 5.52 (br, 1H), 4.13 (q, J = 7.1 Hz, 2H), 2.57 (t, J = 7.6 Hz, 2H), 2.31 (t, J = 7.5 Hz, 2H), 1.96 – 1.86 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 174.1, 154.1, 133.5, 129.7, 115.4, 60.6, 34.4, 33.8, 26.9, 14.4. HRMS (ESI) calcd for $C_{12}H_{16}NaO_3$ [M + Na]$^+$ m/z = 231.0992, found: 231.0992. Data were in agreement with a known literature[5].

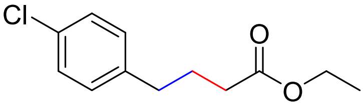
ethyl 4-(4-fluorophenyl) butanoate **(11)**



Prepared following the general procedure employing 4-Fluorobenzaldehyde (124 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73mL) in the reaction mixture. The desired product was obtained as colorless oil (87% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.16 – 7.09 (m, 2H), 7.00 – 6.90 (m, 2H), 4.12 (q, J = 7.1 Hz, 2H), 2.64 – 2.58 (m, J = 7.2 Hz, 2H), 2.30 (t, J = 7.4 Hz, 2H), 1.97 – 1.87 (m, 2H), 1.24 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.4, 161.5 (d, J = 243.6 Hz), 137.1 (d, J = 3.1 Hz), 129.9 (d, J = 7.8 Hz), 115.2 (d, J = 21.1 Hz), 77.5, 77.2, 76.8, 60.4, 34.4, 33.6, 26.7, 14.3. ^{19}F NMR (376 MHz, CDCl$_3$) δ -111.43 – -125.11 (m). Data were in agreement with a known literature[4].

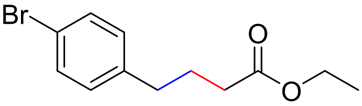
ethyl 4-(4-chlorophenyl) butanoate **(12)**



Prepared following the general procedure employing 4-Chlorobenzaldehyde (140 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (158 mg, 70%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.22 (m, 2H), 7.12 – 7.08 (m, 2H), 4.12 (q, J = 7.1 Hz, 2H), 2.61 (t, J = 7.6 Hz, 2H), 2.30 (t, J = 7.4 Hz, 2H), 1.97 – 1.87 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.4, 140.0, 131.8, 129.9, 128.6, 60.4, 34.6, 33.6, 26.5, 14.4. For other data, please refer to literature[6].

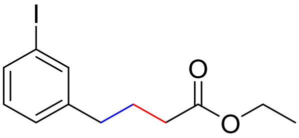
ethyl 4-(4-bromophenyl) butanoate **(13)**



Prepared following the general procedure employing 4-Chlorobenzaldehyde (140 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (211 mg, 78%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 (d, J = 8.2 Hz, 2H), 7.04 (d, J = 8.1 Hz, 2H), 4.11 (q, J = 7.1 Hz, 2H), 2.59 (t, J = 7.6 Hz, 2H), 2.29 (t, J = 7.4 Hz, 2H), 1.91 (p, J = 7.5 Hz, 2H), 1.24 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.3, 140.5, 131.5, 130.3, 119.8, 60.4, 34.6, 33.6, 26.4, 14.3. HRMS (ESI) calcd for $C_{12}H_{15}BrNaO_2$ [M + Na]$^+$ m/z = 293.0148, found: 293.0150. Data were in agreement with a known literature[4].

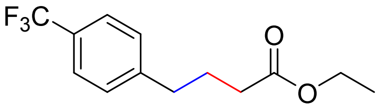
ethyl 4-(3-iodophenyl) butanoate **(14)**



Prepared following the general procedure employing 3-Iodobenzaldehyde (140 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (241 mg, 76%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.48 (m, 2H), 7.13 (d, J = 7.6 Hz, 1H), 7.00 (t, J = 7.7 Hz, 1H), 4.12 (q, J = 7.1 Hz, 2H), 2.58 (t, J = 7.6 Hz, 2H), 2.30 (t, J = 7.4 Hz, 2H), 1.97 – 1.87 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.3, 144.0, 137.6, 135.2, 130.2, 127.9, 94.6, 60.4, 34.8, 33.6, 26.4, 14.4. IR ν_{max}/cm^{-1} (film): 2957, 2928, 1738, 1158, 968, 747, 697. HRMS (ESI) calcd for $C_{12}H_{15}INaO_2$ [M + Na]$^+$ m/z = 341.0009, found: 341.0013.

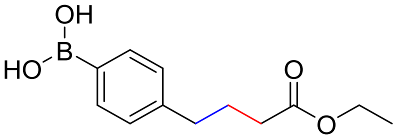
ethyl 4-(4-(trifluoromethyl) phenyl) butanoate **(15)**



Prepared following the general procedure employing 4-(Trifluoromethyl) benzaldehyde (174 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (89% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.53 (d, J = 8.1 Hz, 2H), 7.29 (d, J = 8.0 Hz, 2H), 4.13 (q, J = 7.1 Hz, 2H), 2.71 (t, J = 7.7 Hz, 2H), 2.32 (t, J = 7.4 Hz, 2H), 2.02 – 1.91 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{19}F NMR (376 MHz, CDCl$_3$) δ -62.43 (s). For other data, please refer to literature[7].

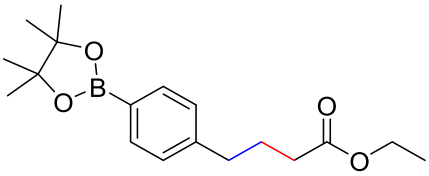
(4-(4-ethoxy-4-oxobutyl) phenyl) boronic acid **(16)**



Prepared following the general procedure employing (4-formylphenyl) boronic acid (150 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (160 mg, 68%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 2: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.15 (d, J = 7.8 Hz, 2H), 7.33 (d, J = 7.8 Hz, 2H), 4.15 (q, J = 7.1 Hz, 2H), 2.74 (t, J = 7.6 Hz, 2H), 2.36 (t, J = 7.4 Hz, 2H), 2.07 – 1.97 (m, 2H), 1.27 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.6, 146.5, 136.0, 134.0, 128.4, 60.5, 35.6, 33.8, 26.5, 14.4. IR ν_{max}/cm^{-1} (film): 3734, 2927, 1731, 1609, 1408, 1339, 1305, 1180, 1019, 690. HRMS (ESI) calcd for $C_{12}H_{17}BNaO_4$ [M + Na]$^+$ m/z = 259.1112, found: 259.1110.

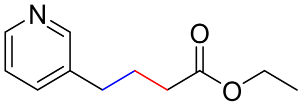
ethyl 4-(4-(4,4,5,5-tetramethyl-1,3,2-dioxaborolan-2-yl) phenyl) butanoate **(17)**



Prepared following the general procedure employing 4-Formylphenylboronic acid pinacol cyclic ester (232 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (267 mg, 84%) after purification by flash column chromatography on silica gel (petroleum ether: DCM = 1: 2).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.74 (d, J = 7.8 Hz, 2H), 7.19 (d, J = 7.8 Hz, 2H), 4.12 (q, J = 7.1 Hz, 2H), 2.66 (t, J = 7.6 Hz, 2H), 2.30 (t, J = 7.5 Hz, 2H), 2.00 – 1.90 (m, 2H), 1.34 (s, 12H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.6, 145.0, 135.1, 131.2, 128.1, 83.8, 60.4, 35.4, 33.8, 26.5, 25.0, 14.4. HRMS (ESI) calcd for $C_{18}H_{27}BNaO_4$ [M + Na]$^+$ m/z = 341.1895, found: 341.1895. Data were in agreement with a known literature[4].

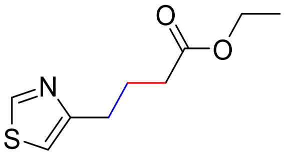
ethyl 4-(pyridin-3-yl) butanoate **(18)**



Prepared following the general procedure employing 3-Pyridinecarboxaldehyde (107 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (70% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (DCM: MeOH = 100: 2).

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.48 – 8.39 (m, 2H), 7.56 – 7.45 (m, 1H), 7.21 (dd, J = 7.8, 4.9 Hz, 1H), 4.12 (q, J = 7.1 Hz, 2H), 2.65 (t, J = 7.6 Hz, 2H), 2.32 (t, J = 7.4 Hz, 2H), 1.99 – 1.88 (m, 2H), 1.24 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.2, 150.1, 147.7, 136.8, 136.0, 123.5, 60.5, 33.6, 32.4, 26.3, 14.4. HRMS (ESI) calcd for $C_{11}H_{16}NO_2$ [M + H]$^+$ m/z = 194.1176, found: 194.1178. Data were in agreement with a known literature[8].

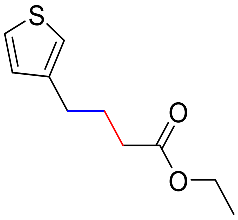
ethyl 4-(thiazol-4-yl) butanoate **(19)**



Prepared following the general procedure employing thiazole-4-carbaldehyde (199 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (85% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 8: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 8.70 (d, J = 1.9 Hz, 1H), 6.96 – 6.89 (m, 1H), 4.07 (q, J = 7.1 Hz, 2H), 2.83 (t, J = 7.5 Hz, 2H), 2.30 (t, J = 7.4 Hz, 2H), 2.07 – 1.95 (m, 2H), 1.19 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.3, 157.2, 152.4, 113.2, 60.3, 33.6, 30.5, 24.5, 14.3. HRMS (ESI) calcd for $C_9H_{14}NO_2S$ [M + H]$^+$ m/z = 200.0740, found: 200.0742. Data were in agreement with a known literature[9].

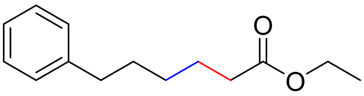
ethyl 4-(thiophen-3-yl) butanoate **(20)**



Prepared following the general procedure employing thiophene-3-carbaldehyde (112 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (91% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 8: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.26 – 7.23 (m, 1H), 6.96 – 6.93 (m, 2H), 4.13 (q, J = 7.1 Hz, 2H), 2.68 (t, J = 7.5 Hz, 2H), 2.32 (t, J = 7.5 Hz, 2H), 2.02 – 1.90 (m, 2H), 1.25 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.5, 141.8, 128.2, 125.5, 120.6, 60.4, 33.8, 29.6, 25.9, 14.4. IR ν_{max}/cm^{-1} (film): 3357, 2920, 2850, 1658, 1632, 1470, 722. HRMS (ESI) calcd for $C_{10}H_{14}NaO_2S$ [M + Na]$^+$ m/z = 221.0607, found: 221.0606.

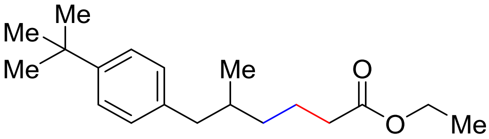
ethyl 6-phenylhexanoate **(21)**



Prepared following the general procedure employing Phenylpropyl aldehyde (134 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (69% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 50: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.31 – 7.25 (m, 2H), 7.21 – 7.16 (m, 3H), 4.13 (q, J = 7.1 Hz, 2H), 2.65 – 2.59 (m, 2H), 2.30 (t, J = 7.5 Hz, 2H), 1.72 – 1.61 (m, 4H), 1.43 – 1.34 (m, 2H), 1.26 (t, J = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.8, 142.6, 128.5, 128.4, 125.7, 60.3, 35.8, 34.4, 31.2, 28.9, 25.0, 14.4. HRMS (ESI) calcd for $C_{14}H_{20}N_aO_2$ [M + Na] $^+$ m/z = 243.1356, found: 243.1358. Data were in agreement with a known literature[10].

ethyl 6-(4-(tert-butyl) phenyl)-5-methylhexanoate **(22)**



Prepared following the general procedure employing Phenylpropyl aldehyde (204 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (141 mg, 40%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

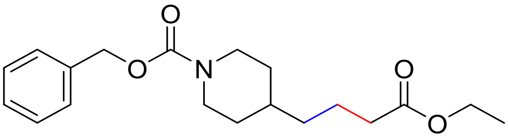
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.29 (d, J = 8.2 Hz, 2H), 7.07 (d, J = 8.2 Hz, 2H), 4.12 (q, J = 7.1 Hz, 2H), 2.59 (dd, J = 13.4, 6.2 Hz, 1H), 2.35 (dd, J = 13.4, 8.0 Hz, 1H), 2.28 (t, J = 7.3Hz, 2H), 1.79 – 1.67 (m, 2H), 1.65 – 1.57 (m, 1H), 1.43 – 1.35 (m, 1H), 1.31 (s, 9H), 1.24 (t, J = 7.1 Hz, 3H), 1.21 – 1.09 (m, 1H), 0.87 (d, J = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.9, 148.5, 138.3, 128.9, 125.1, 60.3, 43.2, 36.2, 34.9, 34.8, 34.5, 31.6, 22.8, 19.5, 14.4. IR ν_{max}/cm^{-1} (film): 2960, 2922, 1735, 1465, 1364, 1260, 1180, 1098, 663, 603, 543. HRMS (ESI) calcd for $C_{19}H_{30}NaO_2$ [M + Na]$^+$ m/z = 313.2138, found: 313.2136.

ethyl 6-(5-methylfuran-2-yl) hexanoate **(23)**

Prepared following the general procedure employing 3-(5-methylfuran-2-yl) propanal (138 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (110 mg, 49%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 50: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 5.83 (s, 2H), 4.12 (q, J = 7.1 Hz, 2H), 2.56 (t, J = 7.6 Hz, 2H), 2.29 (t, J = 7.5 Hz, 2H), 2.24 (s, 3H), 1.69 – 1.60 (m, 4H), 1.42 – 1.33 (m, 2H), 1.24 (t, J = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.9, 154.5, 150.3, 105.9, 105.4, 60.3, 34.4, 28.8, 28.0, 28.0, 24.9, 14.4, 13.6. IR ν_{max}/cm^{-1} (film): 2920, 2850, 1734, 1658, 1631, 1469, 1365, 1172, 1019, 778. HRMS (ESI) calcd for $C_{13}H_{20}NaO_3$ $[M + Na]^+$ m/z = 247.1305, found: 247.1306.

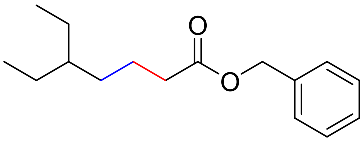
benzyl 4-(4-ethoxy-4-oxobutyl) piperidine-1-carboxylate **(24)**



Prepared following the general procedure employing benzyl 4-formylpiperidine-1-carboxylate (247 mg, 1.0 mmol), ethyl 2-mercaptoacetate (240 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (150 mg, 45%) after purification by flash column chromatography on silica gel (DCM: CH3OH=100: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.26 (m, 5H), 5.11 (s, 2H), 4.24 – 4.04 (m, 4H), 2.75 (br, 2H), 2.28 (t, J = 7.5 Hz, 2H), 1.72 – 1.56 (m, 4H), 1.46 – 1.35 (m, 1H), 1.32 – 1.21 (m, 5H), 1.18 – 1.02 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.7, 155.4, 137.1, 128.6, 128.0, 127.9, 67.0, 60.4, 44.3, 36.0, 35.8, 34.5, 32.1, 22.2, 14.4. HRMS (ESI) calcd for $C_{19}H_{27}NNaO_4$ $[M + Na]^+$ m/z = 356.1832, found: 356.1830. Data were in agreement with a known literature[11].

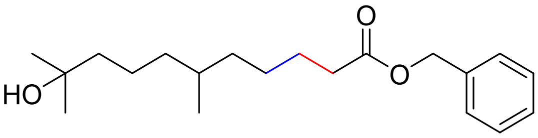
benzyl 5-ethylheptanoate **(25)**



Prepared following the general procedure employing 2-ethylbutanal (100 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (112 mg, 45%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 5H), 5.13 (s, 2H), 2.35 (t, J = 7.5 Hz, 2H), 1.68 – 1.59 (m, 2H), 1.33 – 1.23 (m, 6H), 1.23 – 1.14 (m, 1H), 0.83 (t, J = 7.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.7, 136.3, 128.6, 128.3, 128.3, 66.2, 40.3, 34.9, 32.4, 25.5, 22.4, 11.0. IR ν_{max}/cm^{-1} (film): 2957, 2920, 2951, 1735, 1654, 1631, 1468, 1161, 695. HRMS (ESI) calcd for $C_{16}H_{24}NaO_2$ $[M + Na]^+$ m/z = 271.1669, found: 271.1669.

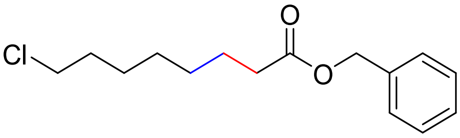
benzyl 10-hydroxy-6,10-dimethylundecanoate **(26)**



Prepared following the general procedure employing 7-hydroxy-3,7-dimethyloctanal (172 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (60% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 3: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.38 – 7.26 (m, 5H), 5.10 (s, 2H), 2.34 (t, J = 7.5 Hz, 2H), 1.83 (br, 1H), 1.68 – 1.55 (m, 2H), 1.43 – 1.23 (m, 9H), 1.19 (s, 6H), 1.16 – 1.01 (m, 2H), 0.83 (d, J = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.6, 136.2, 128.5, 128.1, 70.9, 66.0, 44.3, 37.5, 36.6, 34.3, 32.6, 29.3, 29.2, 26.6, 25.3, 21.8, 19.6. IR ν_{max}/cm^{-1} (film): 2934, 2865, 1738, 1456, 1378, 1214, 1164, 748, 697. HRMS (ESI) calcd for $C_{20}H_{32}NaO_3$ [M + Na] $^+$ m/z = 343.2244, found: 343.3343.

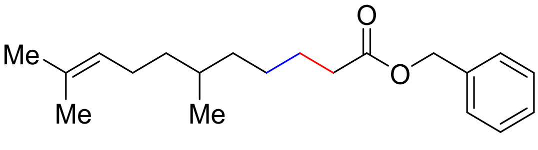
benzyl 8-chlorooctanoate **(27)**



Prepared following the general procedure employing 5-chloropentanal (120 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (76% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 80: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.37 – 7.27 (m, 5H), 5.10 (s, 2H), 3.50 (t, J = 6.7 Hz, 2H), 2.34 (t, J = 7.5 Hz, 2H), 1.78 – 1.69 (m, 2H), 1.67 – 1.61 (m, 2H), 1.45 – 1.35 (m, 2H), 1.35 – 1.28 (m, 4H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.6, 136.3, 128.7, 128.5, 128.3, 66.2, 45.2, 34.4, 32.7, 29.0, 28.6, 26.8, 25.0. IR ν_{max}/cm^{-1} (film): 2932, 2856, 1734, 1456, 1260, 1166, 734, 696. HRMS (ESI) calcd for $C_{15}H_{21}ClNaO_2$ [M + Na]$^+$ m/z = 291.1122, found: 291.1123.

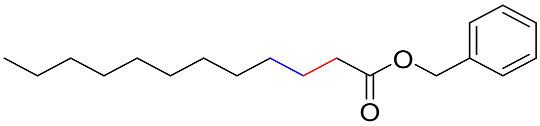
benzyl 6,10-dimethylundec-9-enoate **(28)**



Prepared following the general procedure employing 3,7-dimethyloct-6-enal (154 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (70% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 100: 1).

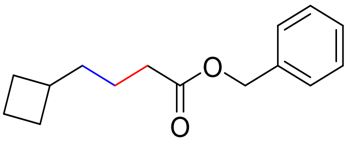
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.43 – 7.27 (m, 5H), 5.17 – 5.04 (m, 3H), 2.36 (t, J = 7.5 Hz, 2H), 2.04 – 1.88 (m, 2H), 1.68 (s, 3H), 1.66 – 1.58 (m, 4H), 1.42 – 1.22 (m, 6H), 1.18 – 1.05 (m, 2H), 0.84 (d, $J = 6.5$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.8, 136.3, 131.2, 128.7, 128.3, 128.3, 125.1, 66.2, 37.2, 36.7, 34.5, 32.4, 26.7, 25.9, 25.7, 25.4, 19.7, 17.8. IR ν_{max}/cm^{-1} (film): 2960, 2926, 2856, 1739, 1455, 1377, 1260, 1161, 1099, 800, 697. HRMS (ESI) calcd for $C_{20}H_{30}NaO_2$ [M + Na]$^+$ m/z = 325.2138, found: 325.2138.

benzyl dodecanoate **(29)**



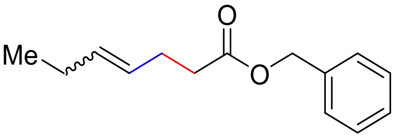
Prepared following the general procedure employing nonanal (142 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (60% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 100: 1).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.41 – 7.29 (m, 5H), 5.13 (s, 2H), 2.36 (t, $J = 7.5$ Hz, 2H), 1.71 – 1.61 (m, 2H), 1.34 – 1.25 (m, 16H), 0.90 (t, $J = 6.9$ Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.7, 136.3, 128.6, 128.3, 128.2, 66.2, 34.5, 32.0, 29.7, 29.7, 29.6, 29.5, 29.3, 29.3, 25.1, 22.8, 14.2. HRMS (ESI) calcd for $C_{19}H_{30}NaO_2$ [M + Na]$^+$ m/z = 313.2138, found: 313.2140. Data were in agreement with a known literature[12].

benzyl 4-cyclobutylbutanoate **(30)**



Prepared following the general procedure employing cyclobutene carbaldehyde (84 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (139 mg, 60%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 30: 1).
^{1}H NMR (400 MHz, CDCl$_3$) δ 7.42 – 7.28 (m, 5H), 5.13 (s, 2H), 2.34 (t, $J = 7.5$ Hz, 2H), 2.31 – 2.19 (m, 1H), 2.09 – 1.98 (m, 2H), 1.91 – 1.71 (m, 2H), 1.66 – 1.52 (m, 4H), 1.46 – 1.37 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.7, 136.3, 128.6, 128.3, 128.2, 66.1, 36.5, 35.8, 34.4, 28.3, 22.8, 18.5. IR ν_{max}/cm^{-1} (film): 2932, 2859, 1738, 1455, 1213, 1162, 748, 670. HRMS (ESI) calcd for $C_{15}H_{20}NaO_2$ [M + Na]$^+$ m/z = 255.1356, found: 255.1356.

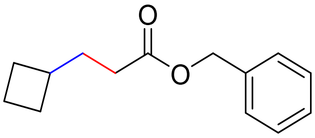
benzyl hept-4-enoate **(31)**



Prepared following the general procedure employing cyclopropane carbaldehyde (70 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (128 mg, 59%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 150: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.45 – 7.28 (m, 5H), 5.57 – 5.28 (m, 2H), 5.13 (s, 2H), 2.47 – 2.29 (m, 4H), 2.12 – 1.93 (m, 2H), 0.96 (td, J = 7.5, 3.7 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$, double equivalent carbon) δ 173.1, 173.1, 136.2, 136.2, 133.5, 133.3, 128.6, 128.6, 128.5, 128.3, 128.3, 128.2, 127.0, 126.8, 66.3, 66.2, 34.5, 34.5, 28.0, 25.6, 22.8, 20.6, 14.3, 13.8. IR ν_{max}/cm^{-1} (film): 2962, 2931, 1738, 1455, 1160, 967, 735, 697. HRMS (ESI) calcd for $C_{14}H_{18}NaO_2$ [M + Na]$^+$ m/z = 241.1199, found: 241.1199.

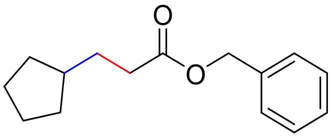
benzyl 3-cyclobutylpropanoate **(32)**



Prepared following the general procedure employing cyclobutanone (70 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (163 mg, 75%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.41 – 7.26 (m, 5H), 5.09 (s, 2H), 2.25 (t, J = 7.6 Hz, 2H), 2.07 – 1.97 (m, 2H), 1.89 – 1.68 (m, 4H), 1.65 – 1.51 (m, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.8, 136.3, 128.7, 128.5, 128.3, 66.2, 35.6, 32.3, 32.1, 28.1, 22.4, 18.4. IR ν_{max}/cm^{-1} (film): 2920, 2850, 1658, 1632, 1470, 852, 720, 634. HRMS (ESI) calcd for $C_{14}H_{18}NaO_2$ [M + Na]$^+$ m/z = 241.1199, found: 241.1199.

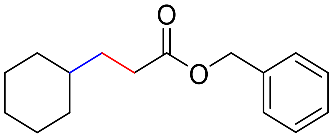
benzyl 3-cyclopentylpropanoate **(33)**



Prepared following the general procedure employing cyclopentanone (84 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (106 mg, 46%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.42 – 7.29 (m, 5H), 5.12 (s, 2H), 2.38 (t, J = 7.6 Hz, 2H), 1.81 – 1.70 (m, 3H), 1.71 – 1.45 (m, 6H), 1.15 – 1.03 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.9, 136.3, 128.7, 128.3, 128.3, 66.2, 39.8, 33.8, 32.5, 31.3, 25.3. IR ν_{max}/cm^{-1} (film): 2949, 2866, 1738, 1454, 1257, 1166, 1001, 734, 696. HRMS (ESI) calcd for $C_{15}H_{20}NaO_2$ [M + Na]$^+$ m/z = 255.1356, found: 255.1356.

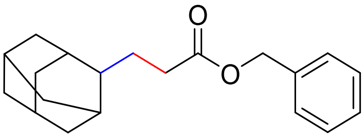
benzyl 3-cyclohexylpropanoate **(34)**



Prepared following the general procedure employing cyclohexanone (98 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (189 mg, 77%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.39 – 7.27 (m, 5H), 5.11 (s, 2H), 2.40 – 2.31 (m, 2H), 1.73 – 1.60 (m, 5H), 1.55 (dd, J = 15.4, 7.1 Hz, 2H), 1.27 – 1.08 (m, 4H), 0.94 – 0.81 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 174.0, 136.3, 128.6, 128.2, 128.2, 66.1, 37.3, 33.0, 32.4, 32.0, 26.6, 26.3. IR ν_{max}/cm^{-1} (film): 2922, 2851, 1738, 1448, 1260, 1159, 1080, 695. HRMS (ESI) calcd for $C_{16}H_{22}NaO_2$ [M + Na]$^+$ m/z = 269.1512, found: 269.1512.

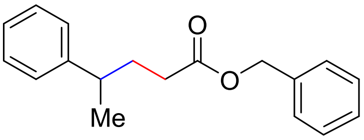
benzyl 3-(adamantan-2-yl) propanoate **(35)**



Prepared following the general procedure employing adamantan-2-one (150 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (244 mg, 82%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.42 – 7.28 (m, 5H), 5.13 (s, 2H), 2.40 – 2.32 (m, 2H), 1.92 – 1.76 (m, 8H), 1.71 (d, J = 13.4 Hz, 6H), 1.62 (t, J = 7.4 Hz, 1H), 1.51 (d, J = 12.4 Hz, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 174.0, 136.3, 128.6, 128.3, 128.2, 66.2, 44.1, 39.2, 38.4, 32.7, 31.7, 31.6, 28.3, 28.1, 27.9. IR ν_{max}/cm^{-1} (film): 2904, 2850, 1737, 1454, 1238, 1151, 747, 696. HRMS (ESI) calcd for $C_{20}H_{26}NaO_2$ [M + Na]$^+$ m/z = 321.1825, found: 321.1825.

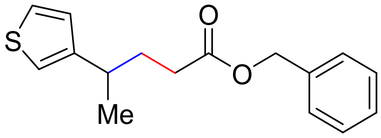
benzyl 4-phenylpentanoate **(36)**



Prepared following the general procedure employing acetophenone (120 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (159 mg, 60%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.41 – 7.27 (m, 7H), 7.24 – 7.15 (m, 3H), 5.14 – 5.05 (m, 2H), 2.79 – 2.67 (m, 1H), 2.34 – 2.17 (m, 2H), 2.04 – 1.86 (m, 2H), 1.29 (d, J = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.6, 146.3, 136.2, 128.6, 128.6, 128.3, 128.3, 127.1, 126.3, 66.2, 39.52, 33.3, 32.7, 22.3. IR ν_{max}/cm^{-1} (film): 2959, 2928, 1734, 1453, 1206, 1154, 762, 698. HRMS (ESI) calcd for $C_{18}H_{20}NaO_2$ [M + Na]$^+$ m/z = 291.1356, found: 291.1357.

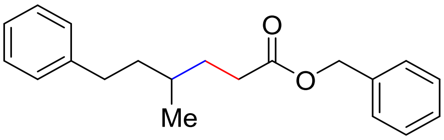
benzyl 4-(thiophen-3-yl) pentanoate **(37)**



Prepared following the general procedure employing 1-(thiophen-3-yl) ethan-1-one (126 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (175 mg, 64%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.42 – 7.31 (m, 5H), 7.29 – 7.24 (m, 1H), 6.98 – 6.91 (m, 2H), 5.10 (d, J = 2.2 Hz, 2H), 2.94 – 2.83 (m, 1H), 2.30 (t, J = 7.7 Hz, 2H), 2.01 – 1.83 (m, 2H), 1.28 (d, J = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.5, 147.2, 136.2, 128.7, 128.3, 126.7, 125.6, 119.6, 66.3, 34.7, 33.1, 32.4, 21.8. IR ν_{max}/cm^{-1} (film): 2955, 2867, 1737, 1455, 1257, 1165, 748, 778, 696. HRMS (ESI) calcd for $C_{16}H_{18}NaO_2S$ [M + Na]$^+$ m/z = 297.0920, found: 297.0921.

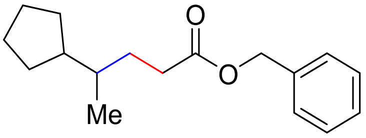
benzyl 4-methyl-6-phenylhexanoate **(38)**



Prepared following the general procedure employing 4-phenylbutan-2-one (148 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (178 mg, 60%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.45 – 7.34 (m, 5H), 7.34 – 7.26 (m, 2H), 7.25 – 7.18 (m, 3H), 5.16 (s, 2H), 2.76 – 2.57 (m, 2H), 2.51 – 2.31 (m, 2H), 1.86 – 1.75 (m, 1H), 1.74 – 1.63 (m, 1H), 1.62 – 1.43 (m, 3H), 0.99 (d, J = 6.0 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.8, 142.8, 136.2, 128.6, 128.4, 128.4, 128.3, 128.3, 125.7, 66.2, 38.6, 33.4, 32.2, 32.1, 31.9, 19.3. IR ν_{max}/cm^{-1} (film): 3027, 2927, 2857, 1735, 1496, 1454, 1255, 1158, 745, 699. HRMS (ESI) calcd for $C_{20}H_{24}NaO_2$ [M + Na]$^+$ m/z = 319.1669, found: 319.1669.

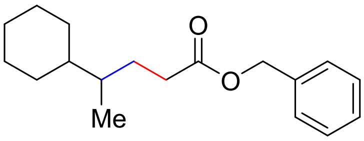
benzyl 4-cyclopentylpentanoate **(39)**



Prepared following the general procedure employing 1-cyclopentylethan-1-one (112 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (210 mg, 80%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.40 – 7.29 (m, 4H), 5.12 (s, 2H), 2.37 (dddd, J = 22.1, 15.7, 9.7, 6.1 Hz, 2H), 1.91 – 1.80 (m, 1H), 1.80 – 1.67 (m, 2H), 1.64 – 1.35 (m, 6H), 1.33 – 1.25 (m, 1H), 1.19 – 1.06 (m, 2H), 0.88 (d, J = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.1, 136.3, 128.6, 128.3, 128.3, 66.2, 46.2, 37.9, 32.2, 30.9, 30.8, 30.3, 25.6, 25.5, 17.5. IR ν_{max}/cm^{-1} (film): 2951, 1867, 1738, 1455, 1258, 1160, 749, 696. HRMS (ESI) calcd for $C_{17}H_{24}NaO_2$ [M + Na]$^+$ m/z = 283.1669, found: 283.1670.

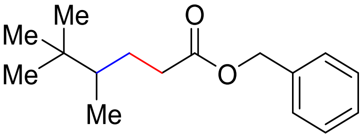
benzyl 4-cyclohexylpentanoate **(40)**



Prepared following the general procedure employing 1-cyclohexylethan-1-one (126 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (250 mg, 90%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.28 (m, 5H), 5.12 (s, 2H), 2.36 (dddd, J = 39.4, 15.7, 9.6, 6.1 Hz, 2H), 1.81 – 1.72 (m, 2H), 1.64 (ddd, J = 23.7, 14.7, 7.1 Hz, 3H), 1.51 – 1.40 (m, 1H), 1.35 – 0.93 (m, 8H), 0.83 (d, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.0, 136.3, 128.6, 128.3, 128.3, 66.2, 42.6, 37.8, 32.7, 30.7, 29.3, 28.8, 27.0, 26.9, 26.9, 15.9. IR ν_{max}/cm^{-1} (film): 2924, 2852, 1737, 1449, 1259, 1164, 697. HRMS (ESI) calcd for $C_{18}H_{26}NaO_2$ [M + Na]$^+$ m/z = 297.1825, found: 297.1825.

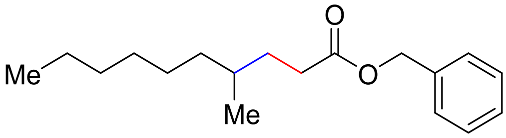
benzyl 4,5,5-trimethylhexanoate **(41)**



Prepared following the general procedure employing 3,3-dimethylbutan-2-one (100 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (212 mg, 82%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.28 (m, 5H), 5.12 (s, 2H), 2.53 – 2.41 (m, 1H), 2.33 – 2.22 (m, 1H), 2.02 – 1.88 (m, 1H), 1.27 – 1.07 (m, 2H), 0.94 – 0.78 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.0, 136.3, 128.7, 128.4, 128.3, 66.2, 42.7, 33.6, 33.2, 27.4, 27.2, 14.0. IR ν_{max}/cm^{-1} (film): 2958, 2921, 2851, 1739, 1468, 1158, 694. HRMS (ESI) calcd for $C_{16}H_{24}NaO_2$ [M + Na]$^+$ m/z = 271.1669, found: 271.1668.

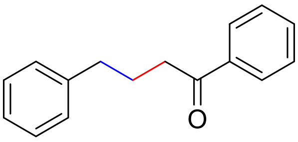
benzyl 4-methyldecanoate **(42)**



Prepared following the general procedure employing octan-2-one (128 mg, 1.0 mmol), benzyl 2-mercaptoacetate (364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (250 mg, 90%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 400: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.43 – 7.29 (m, 5H), 5.13 (s, 2H), 2.48 – 2.27 (m, 2H), 1.78 – 1.63 (m, 1H), 1.57 – 1.41 (m, 2H), 1.38 – 1.22 (m, 9H), 1.21 – 1.08 (m, 1H), 0.95 – 0.85 (m, 6H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 174.0, 136.3, 128.6, 128.3, 128.2, 66.2, 36.8, 32.5, 32.2, 32.0, 29.7, 27.0, 22.8, 19.4, 14.2. ^{13}C NMR (101 MHz, CDCl$_3$) δ 174.0, 136.3, 128.6, 128.3, 128.2, 66.2, 36.8, 32.5, 32.2, 32.0, 32.0, 29.7, 27.0, 22.8, 19.4, 14.2. IR ν_{max}/cm^{-1} (film): 2956, 2926, 2856, 1738, 1456, 1257, 1164, 749, 696. HRMS (ESI) calcd for $C_{18}H_{28}O_2$ [M + Na]$^+$ m/z = 299.1982, found: 299.1981.

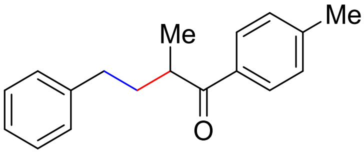
1,4-diphenylbutan-1-one **(43)**



Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), 2-mercapto-1-phenylethan-1-one (**a**, 304 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (134 mg, 60%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 150: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.94 (dt, J = 8.5, 1.7 Hz, 2H), 7.58 – 7.52 (m, 1H), 7.48 – 7.42 (m, 2H), 7.33 – 7.27 (m, 2H), 7.21 (dd, J = 10.3, 6.2 Hz, 3H), 2.99 (t, J = 7.3 Hz, 2H), 2.74 (t, J = 7.6 Hz, 2H), 2.10 (dt, J = 18.7, 7.4 Hz, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 200.2, 141.8, 137.2, 133.0, 128.7, 128.6, 128.5, 128.1, 126.1, 37.8, 35.3, 25.8. HRMS (ESI) calcd for $C_{16}H_{16}NaO$ [M + Na]$^+$ m/z = 247.1093, found: 247.1093. Data were in agreement with a known literature[13].

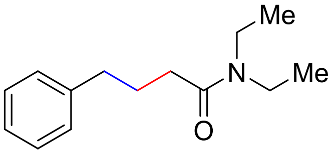
2-methyl-4-phenyl-1-(p-tolyl) butan-1-one **(44)**



Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), 2-mercapto-1-(p-tolyl) propan-1-one (**b**, 360 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (209 mg, 83%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.78 (d, J = 8.2 Hz, 2H), 7.33 – 7.10 (m, 7H), 3.51 – 3.39 (m, 1H), 2.70 – 2.57 (m, 2H), 2.41 (s, 3H), 2.17 (ddd, *J* = 15.5, 13.8, 7.1 Hz, 1H), 1.74 (ddt, *J* = 13.5, 8.4, 6.7 Hz, 1H), 1.23 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 203.8, 143.8, 142.0, 129.4, 128.6, 128.6, 128.5, 126.0, 39.8, 35.4, 33.7, 21.7, 17.5. IR ν_{max}/cm^{-1} (film): 2928, 1679, 1607, 1453, 1231, 1176, 973, 746, 699. HRMS (ESI) calcd for $C_{18}H_{21}O$ [M + H]$^+$ m/z = 253.1587, found: 253.1587.

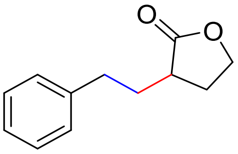
N, N-diethyl-4-phenylbutanamide **(45)**



Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), N, N-diethyl-2-mercaptoacetamide (**c**, 294 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (109 mg, 50%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 5: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 – 7.25 (m, 2H), 7.22 – 7.14 (m, 3H), 3.37 (q, *J* = 7.1 Hz, 2H), 3.23 (q, *J* = 7.2 Hz, 2H), 2.68 (t, *J* = 7.6 Hz, 2H), 2.30 (t, *J* = 7.2 Hz, 2H), 2.04 – 1.94 (m, 2H), 1.10 (td, *J* = 7.1, 1.4 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.9, 142.0, 128.6, 128.4, 126.0, 42.0, 40.2, 35.5, 32.3, 27.0, 14.5, 13.3. HRMS (ESI) calcd for $C_{14}H_{21}NNaO$ [M + Na]$^+$ m/z = 242.1515, found: 242.1515. Data were in agreement with a known literature[14].

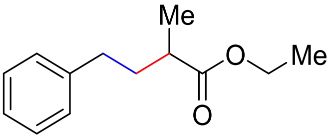
3-phenethyldihydrofuran-2(3H)-one **(46)**



Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), 3-mercaptodihydrofuran-2(3H)-one (**d**, 236 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (80% yield from ^{1}H-NMR) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 5: 1).

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.18 (m, 5H), 4.33 (dd, *J* = 8.8, 2.8 Hz, 1H), 4.17 (m, 1H), 2.85 – 2.68 (m, 2H), 2.54 – 2.44 (m, 1H), 2.43 – 2.32 (m, 1H), 2.29 – 2.17 (m, 1H), 2.03 – 1.88 (m, 1H), 1.83 – 1.70 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 179.4, 140.9, 128.6, 128.5, 126.3, 66.5, 38.5, 33.4, 32.1, 28.9. IR ν_{max}/cm^{-1} (film): 2923, 2853, 1764, 1686, 1377, 1143, 1022, 701. HRMS (ESI) calcd for $C_{12}H_{14}NaO_2$ [M + Na]$^+$ m/z = 213.0886, found: 213.0886.

ethyl 2-methyl-4-phenylbutanoate **(47)**

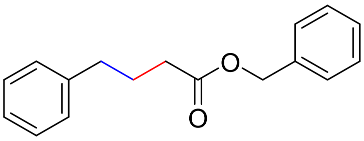


Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), ethyl 2-

mercaptopropanoate (268 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (129 mg, 63%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.33 – 7.27 (m, 2H), 7.23 – 7.17 (m, 3H), 4.16 (q, J = 7.1 Hz, 2H), 2.64 (t, J = 7.6 Hz 2H), 2.53 – 2.43 (m, 1H), 2.10 – 1.96 (m, 1H), 1.80 – 1.68 (m, 1H), 1.29 (t, J = 7.0 Hz 3H), 1.21 (d, J = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 176.5, 141.8, 128.5, 128.4, 125.9, 60.2, 39.1, 35.5, 33.6, 17.2, 14.3. HRMS (ESI) calcd for $C_{13}H_{18}NaO_2$ [M + Na] $^+$ m/z = 229.1199, found: 229.1199. Data were in agreement with a known literature[15].

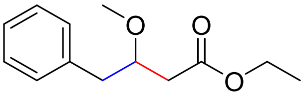
benzyl 4-phenylbutanoate **(48)**



Prepared following the general procedure employing benzaldehyde (106 mg, 1.0 mmol), benzyl 2-mercaptoacetate (**e**, 364 mg, 2.0 mmol), triphenylphosphine (650 mg, 2.5 mmol), di-tert-butyl peroxide (584 mg, 4.0 mmol, 0.73 mL) in the reaction mixture. The desired product was obtained as colorless oil (215 mg, 85%) after purification by flash column chromatography on silica gel (petroleum ether: ethyl acetate = 200: 1).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.43 – 7.27 (m, 7H), 7.25 – 7.16 (m, 3H), 5.14 (s, 2H), 2.68 (t, J = 7.6 Hz, 2H), 2.41 (t, J = 7.5 Hz, 2H), 2.08 – 1.95 (m, 2H). ^{13}C NMR (101 MHz, CDCl$_3$) δ 173.4, 141.5, 136.2, 128.7, 128.6, 128.5, 128.3, 128.3, 126.1, 66.3, 35.2, 33.8, 26.6. HRMS (ESI) calcd for $C_{17}H_{18}NaO_2$ [M + Na] $^+$ m/z = 277.1199, found: 277.1191. Data were in agreement with a known literature[16].

ethyl 3-methoxy-4-phenylbutanoate **(50)**

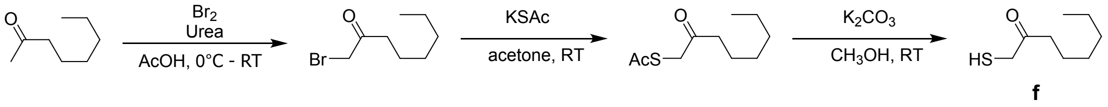


To a 25 mL oven-dried two necked bottle were added (methoxymethyl)triphenylphosphonium chloride (0.42 g, 1.2 mmol). The flask was degassed three times and protected with N_2 before ultra-dry dimethyl sulfoxide (DMSO, 4 mL) was added. n-Butyllithium (0.75 mL, 1.2 mmol, 1.6 M solution in Hexanes) was added and the mixture was stirred at room temperature for 1 hour. Triphenylphosphine (0.65 g, 2.5 mmol) was dissolved in ultra-dry dichloromethane. Then benzaldehyde (1.0 mmol), ethyl 2-mercaptoacetate (2.0 mmol), triphenylphosphine solution and DTBP (0.73 mL, 4 mmol) were added into the reaction mixture in order by micro-syringe. The reaction was stirred and irradiated using two 55 W household CFL bulbs (6 cm away, to keep the reaction at room temperature) for 12 hours. When the reaction was complete, the mixture was washed with water (2×5 mL) and brine (5 mL). The organic layer was dried with sodium sulfate, filtered and concentrated. The residue was purified by column chromatography on silica (petroleum ether: dichloromethane = 1:2) to give the product as colorless oil (50% yield from ^{1}H-NMR).

^{1}H NMR (400 MHz, CDCl$_3$) δ 7.32 – 7.26 (m, 2H), 7.25 – 7.18 (m, 3H), 4.12 (q, J = 7.1 Hz, 2H), 3.87 (dt, *J* = 12.7, 6.3 Hz, 1H), 3.36 (s, 3H), 2.93 (dd, *J* = 13.7, 6.0 Hz, 1H), 2.76 (dd, *J* = 13.7, 6.6 Hz, 1H), 2.43 (qd, *J* = 15.4, 6.3 Hz, 2H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.7, 138.1, 129.7, 128.5, 126.5, 79.1, 60.6, 57.6, 40.2, 39.4, 14.4. IR ν_{max}/cm^{-1} (film): 2930, 1734, 1454, 1260, 1102, 1031, 700. HRMS (ESI) calcd for $C_{13}H_{18}NaO_3$[M + Na]$^+$ m/z = 245.1148, found: 245.1148.

6. Synthesis of indolizidine 209D.

Synthesis of 1-mercaptooctan-2-one (f)

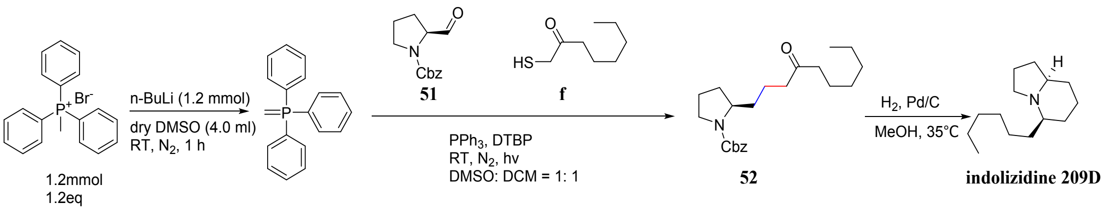


Supplementary Figure S2. synthesis of **f** from commercial compounds in three steps.

To a round bottomed flask with 2-Octanone (7.68 g, 60 mmol) and urea (6.0 g, 99.6 mmol) dissolved in 40ml acetic acid was added dropwise Br_2 at 0 °C. Then the resulting mixture was stirred at room temperature overnight. The reaction mixture was distributed between water (150 mL) and DCM (300 mL). The organic layer was washed with 10% Sodium carbonate solution (200 mL), brine (200 mL), dried with Na_2SO_4, concentrated and purified by flash chromatography (petroleum ether: ethyl acetate = 50: 1) to give 1-bromooctan-2-one (8.5 g, 69%) as colorless oil. Data were in agreement with a known literature[17].

1-mercaptooctan-2-one (f) was prepared from 1-bromooctan-2-one according to the general procedures (Supplementary Figure S1) as a yellow oil (2.5 g, 35%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.30 (t, J = 6.2 Hz, 2H), 2.52 (t, J = 7.4 Hz, 2H), 1.90 (t, J = 7.6 Hz, 1H), 1.61 – 1.49 (m, 2H), 1.27 – 1.22 (m, 6H), 0.83 (t, J = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 205.6, 41.0, 34.3, 31.6, 28.8, 24.0, 22.5, 14.0. IR ν_{max}/cm^{-1} (film): 2955, 2929, 2857, 1710, 1458, 1403, 1059. HRMS (ESI) calcd for $C_8H_{17}OS$ $[M + H]^+$ m/z = 161.0995, found: 161.0993.

Synthesis of indolizidine 209D



Supplementary Figure S4. synthesis of **indolizidine 209D** in two steps.

Synthesis of 52

To a 25 ml oven-dried two necked bottle was added methyltriphenylphosphonium bromide (0.44 g, 1.2 mmol). The flask was degassed three times and protected with N_2 before ultra-dry dimethyl sulfoxide (4 mL) was added. n-Butyllithium (0.75 ml, 1.2 mmol, 1.6 M solution in Hexanes) was added and the mixture was stirred at room temperature for 1 hour. Triphenylphosphine (0.47 g, 1.8 mmol) was dissolved in ultra-dry dichloromethane (2 mL). Commercially available aldehyde **51** (benzyl (*S*)-2-formylpyrrolidine-1-carboxylate, 1.0 mmol, 0.23 g), **f** (1.5 mmol, 0.24 g), triphenylphosphine solution and DTBP (0.55 mL, 3 mmol) were added into the reaction mixture in order by micro-syringe. The reaction was stirred and irradiated using two 40 W household CFL bulbs (6 cm away, to keep the reaction at room temperature) for 12 hours. Then **f** (1.5 mmol, 0.24 g), triphenylphosphine solution (0.47 g in 2 mL ultra-dry dichloromethane) and DTBP (0.55 mL, 3 mmol) were added into the reaction mixture in order by micro-syringe again and the reaction was stirred and irradiated for another 12 hours. The reaction mixture was extracted with EtOAc (3×15 mL), washed with water (2×5 mL) and brine (5 mL). The organic layer was dried with sodium sulfate, filtered and concentrated. The residue was purified by column chromatography on silica (petroleum ether: ethyl acetate = 3: 1) to give the product **52** (0.14 g, 40%) as pale yellow oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 – 7.19 (m, 5H), 5.19 – 5.00 (m, 2H), 3.80 (s, 1H), 3.53 – 3.28 (m, 2H), 2.52 – 2.16 (m, 4H), 1.79 (m, 5H), 1.51 (s, 4H), 1.37 – 1.16 (m, 7H), 0.86 (t, J = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$, double equivalent carbon) δ 211.3, 211.0, 155.0, 137.2, 128.5, 127.9, 66.8, 66.5, 57.8, 57.1, 46.7, 46.4, 42.9, 42.5, 34.3, 33.5, 31.7, 30.7, 29.9, 29.0, 23.9, 23.1, 22.6, 20.4, 14.1. HRMS (ESI) calcd for $C_{22}H_{33}NNaO_3$ [M + Na]$^+$ m/z = 382.2353, found: 382.2352. Data were in agreement with a known literature[18].

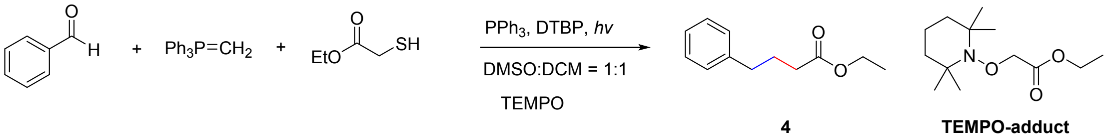
Synthesis of indolizidine 209D

A solution of **52** (100 mg, 0.28 mmol) in MeOH (5 mL) containing Pd on carbon (20%, 20 mg) was stirred under H_2 at 35 ℃ for 12 h. After filtration through Celite, the mixture was concentrated and purified by column chromatography on silica (100% EA) to give the product **indolizidine 209D** (50 mg, 86%) as colorless oil.

^{1}H NMR (400 MHz, $CDCl_3$) δ 3.26 (td, J = 8,8, 2.0 Hz, 1H), 1.97 (q, J = 9.0 Hz, 1H), 1.92 – 1.58 (m, 8H), 1.50 – 0.98 (m, 14H), 0.87 (t, J = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 65.2, 64.1, 51.7, 34.8, 32.0, 31.1, 31.0, 30.7, 29.9, 26.0, 24.9, 22.8, 20.6, 14.2. HRMS (ESI) calcd for $C_{14}H_{27}NNa$ [M + Na]$^+$ m/z = 232.2036, found: 232.2036. Data were in agreement with a known literature[19].

7. Mechanistic studies

Radical trapping experiment

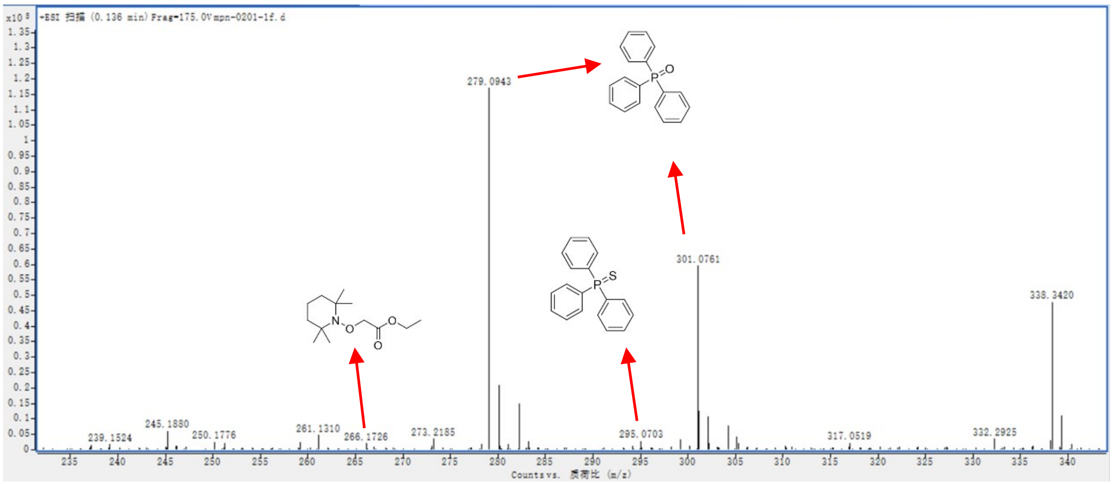


To elucidate the reaction mechanism, the radical-inhibitor experiments was performed by addition of 2 equivalent radical-trapping reagent 2,2,6,6-tetramethyl-1-piperidyloxy (TEMPO) to the reaction. Eventually, the reaction was completely inhibited. TEMPO-adduct was observed in the LC-MS while there was no compound **4**, thus suggesting a radical process.

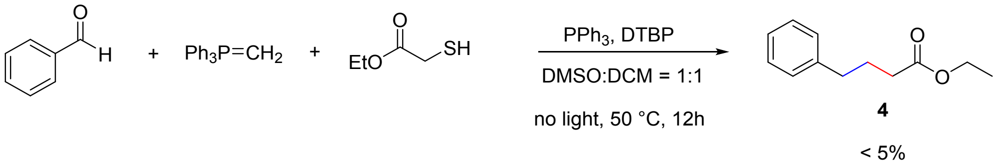
TEMPO-adduct can be detected in mass spectrometry. HRMS (ESI) calcd for TEMPO-adduct $C_{13}H_{25}NNaO_3[M + Na]^+$ m/z = 266.1727, found: 266.1726.

We could also see $Ph_3P{=}O$. HRMS (ESI) calcd $C_{18}H_{16}OP$ $[M + H]^+$ m/z = 279.0933, found: 279.0943. HRMS (ESI) calcd for $C_{18}H_{15}NaOP$ $[M + Na]^+$ m/z = 301.0753, found: 301.0761.

$Ph_3P{=}S$ could also be observed. HRMS (ESI) calcd for $C_{18}H_{16}PS[M + H]^+$ m/z = 295.0705, found: 295.0703.

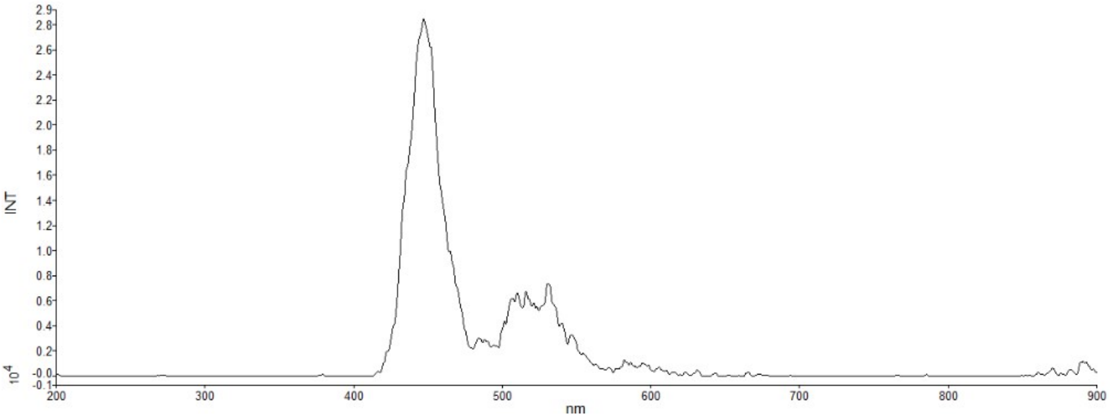


Control experiment (heat to 50 ℃ without light)



When the reaction was heat up to 50°C without light, we could not observe the desired product from ^{1}HNMR.

UV-Vis spectra of the CFL lamp



The CFL lamp was purchased online from JD.COM directly. The manufacture is Foshan Lighting. The brand name is FSL. The type is 4U-55W-E27.

8. References

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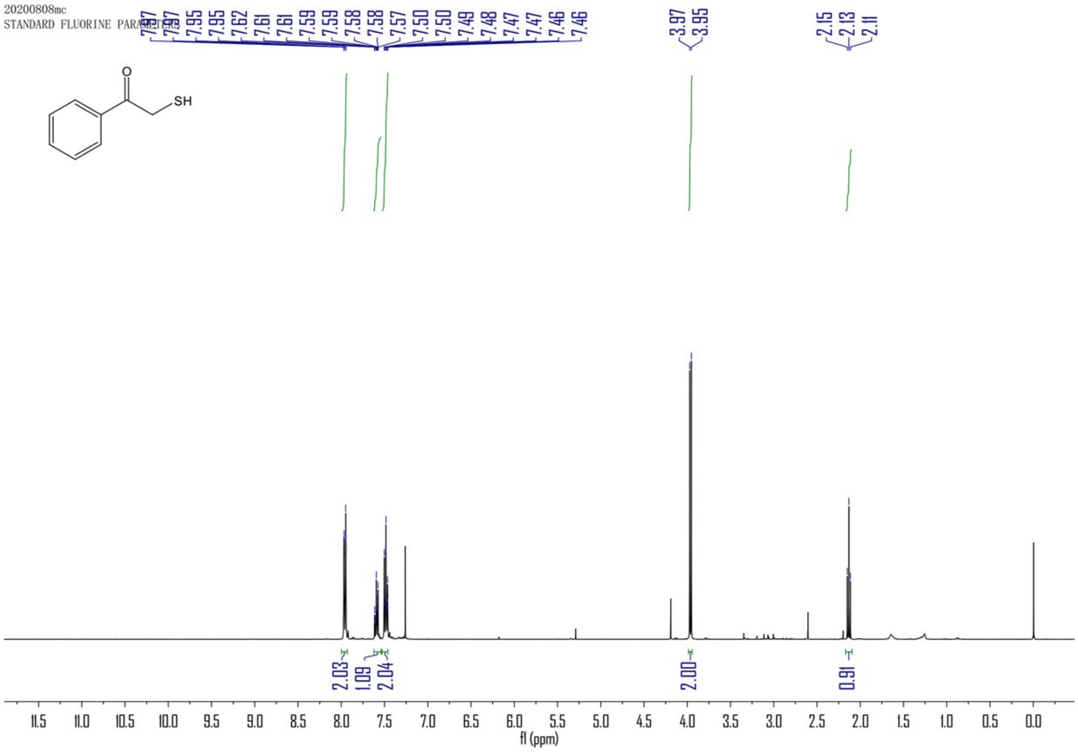
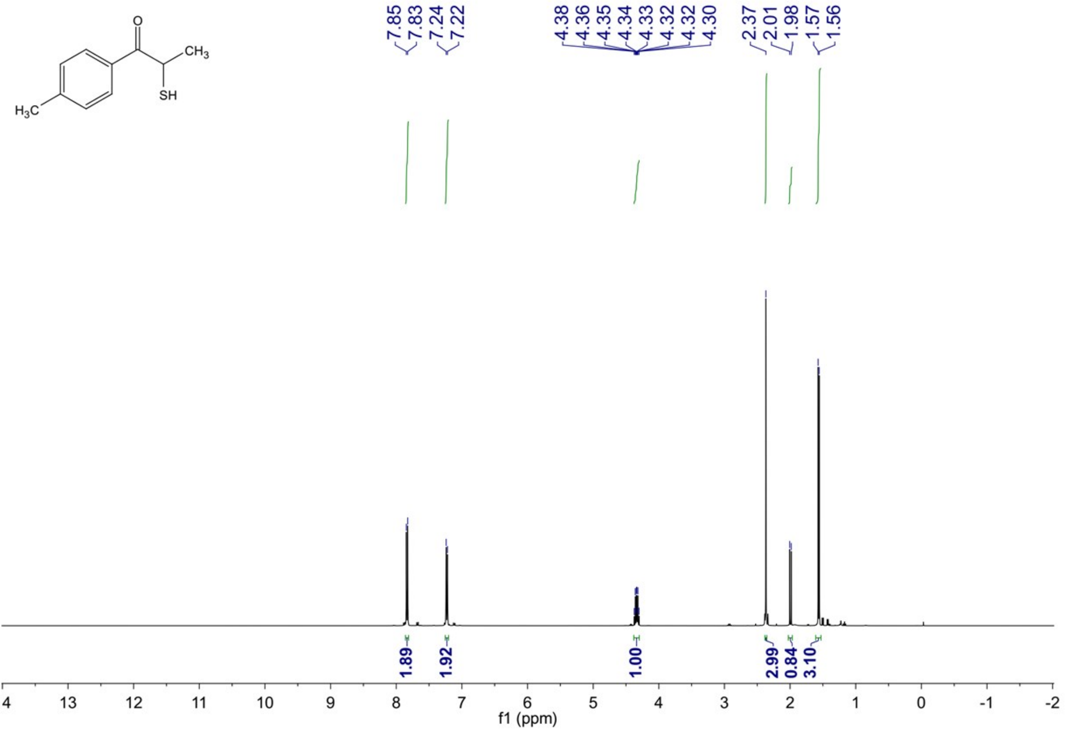
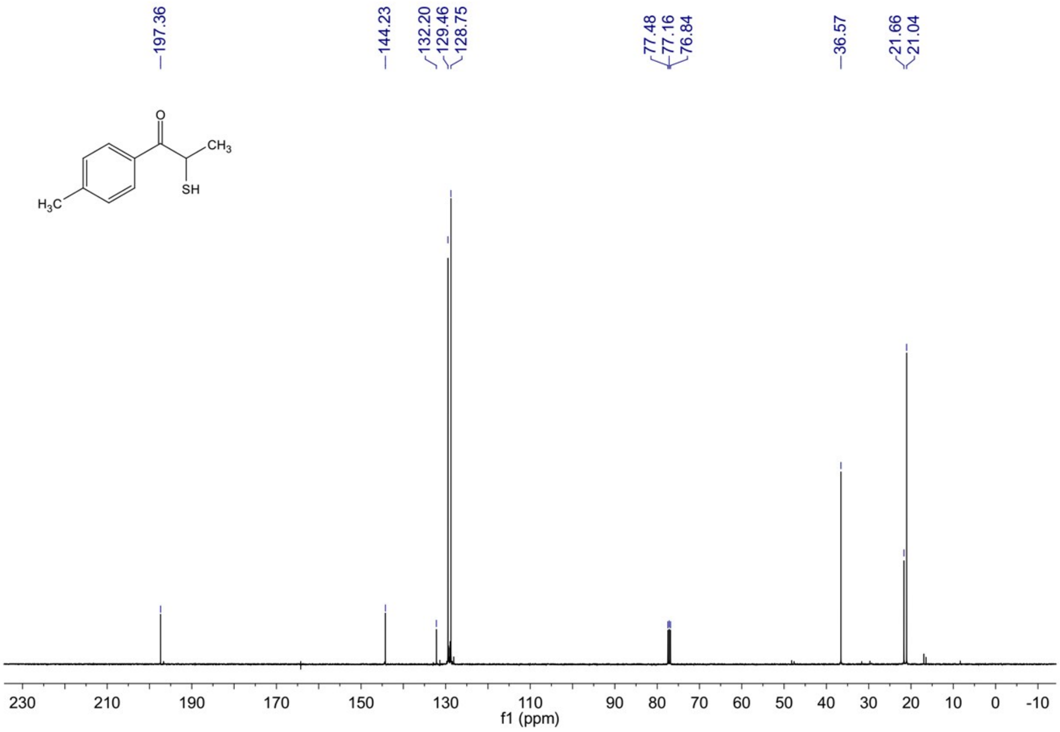
9. Spectral data

a



b

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21.04
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SH
H3C
230
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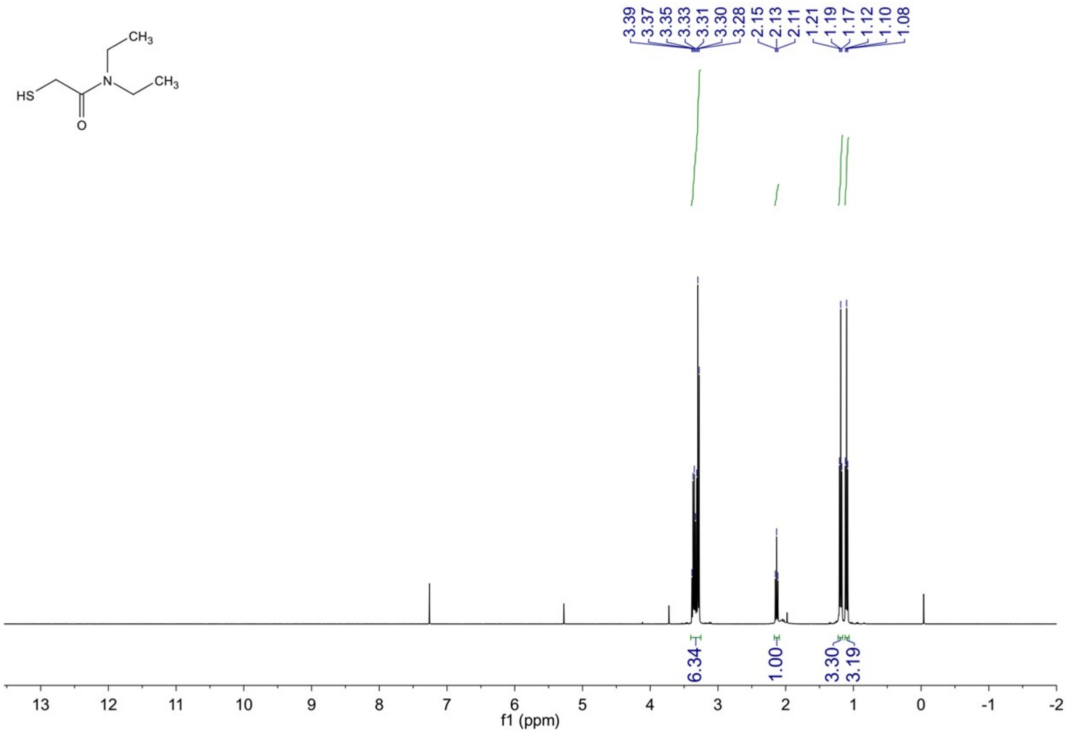
f1 (ppm)

c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HS
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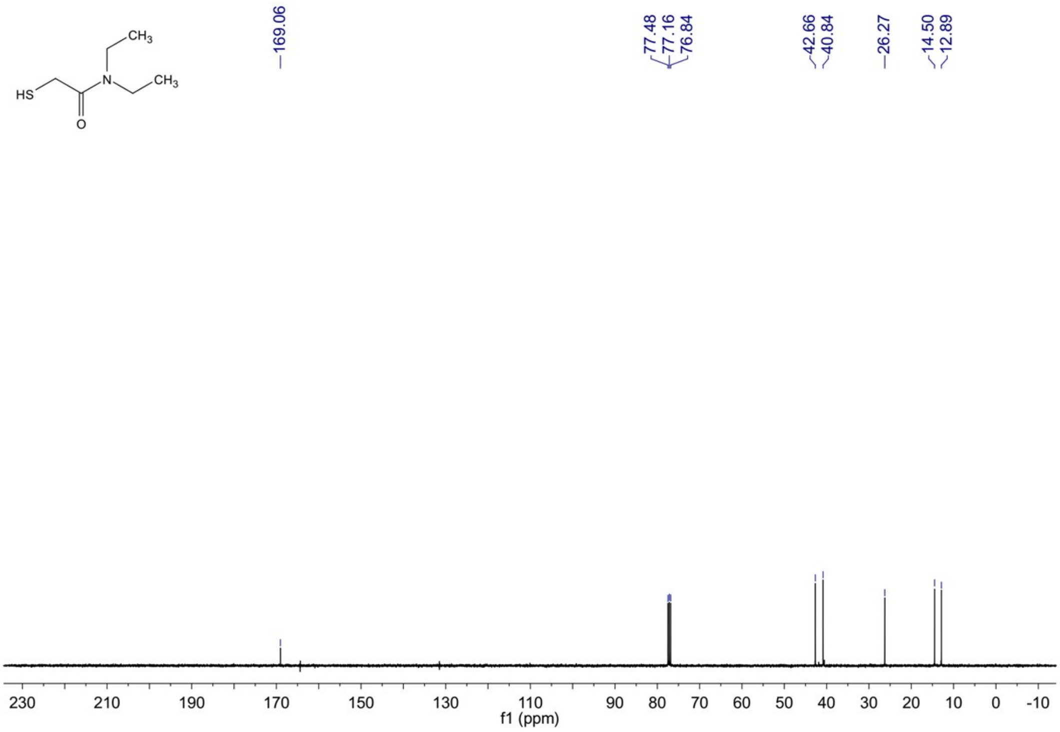
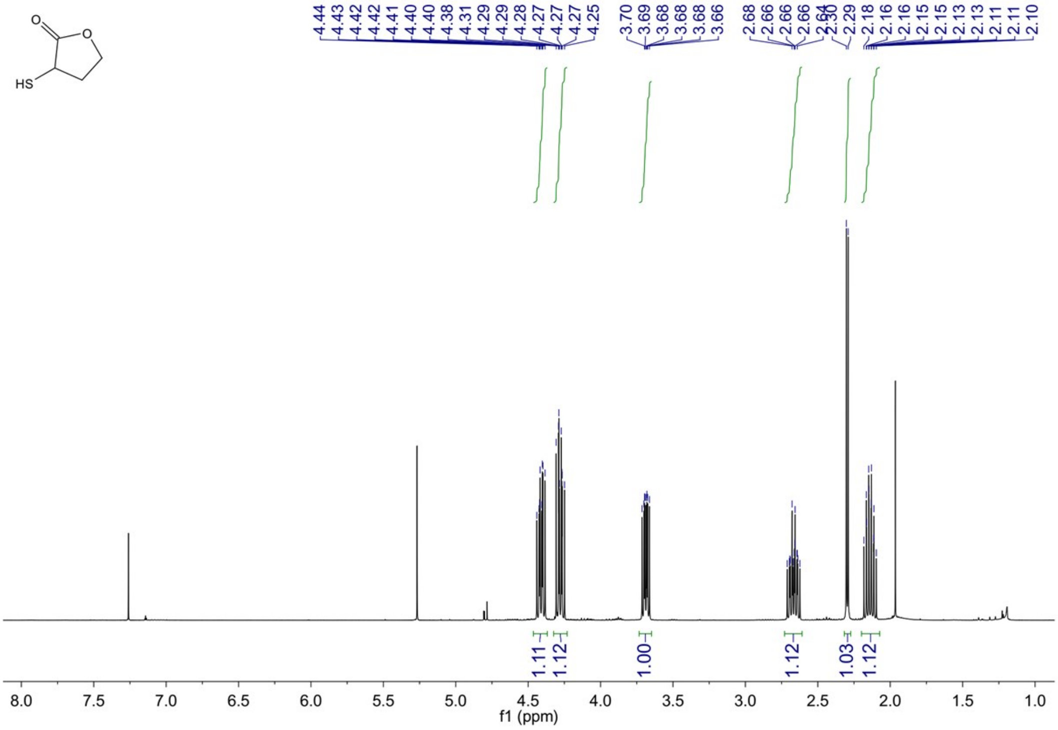
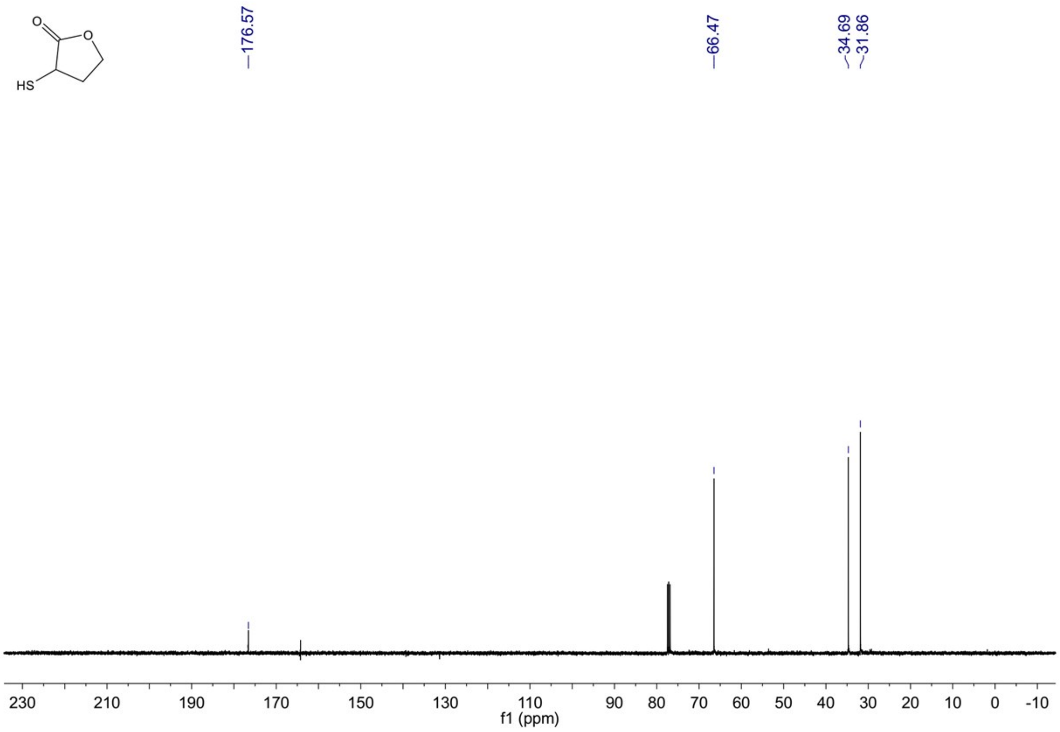
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

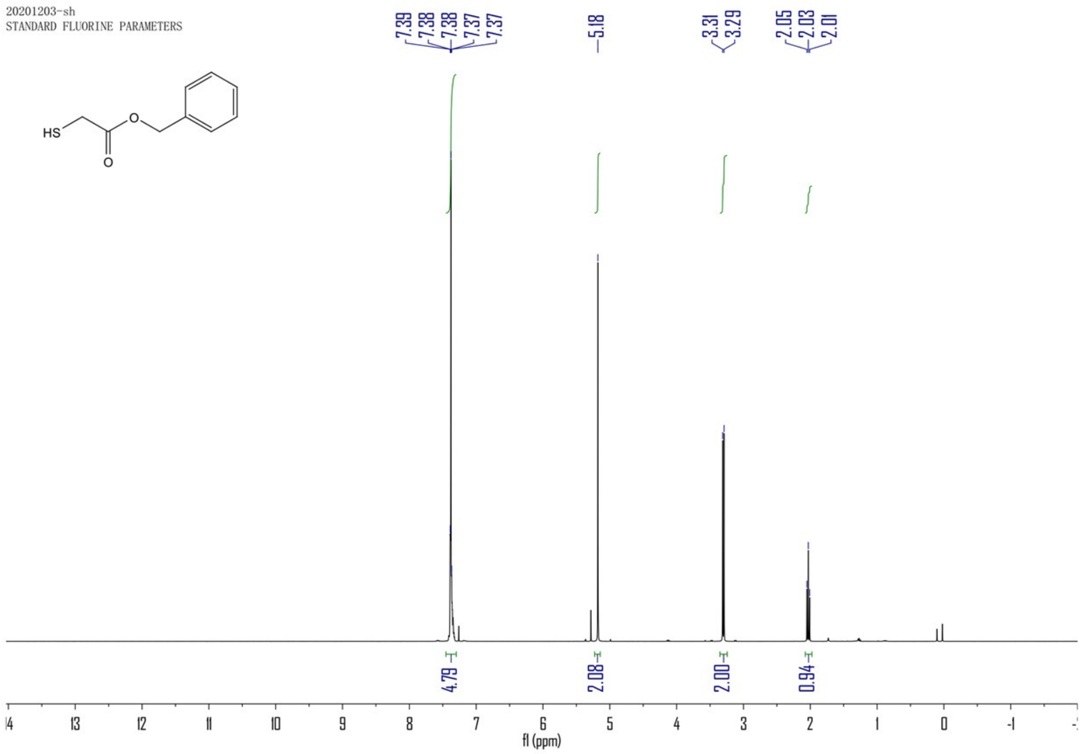

CH3
HS
N
O
CH3
169.06
77.48
77.16
76.84
42.66
40.84
26.27
14.50
12.89
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

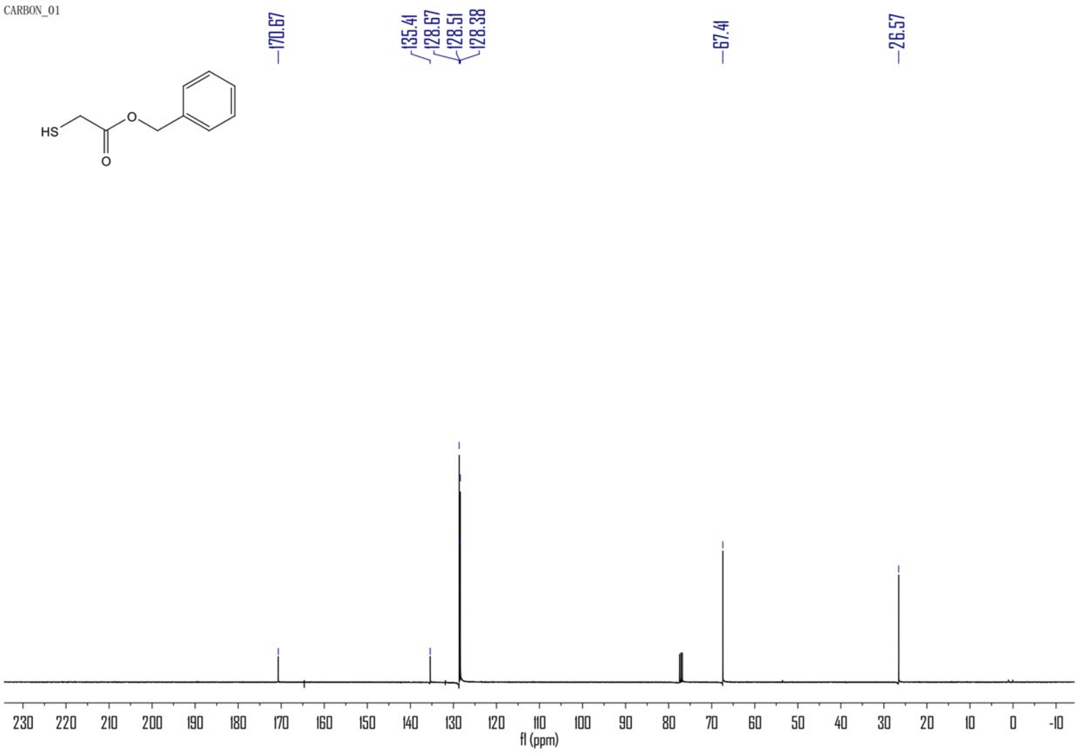
d


O
O
HS
4.44
4.43
4.42
4.42
4.41
4.40
4.40
4.38
4.31
4.29
4.29
4.28
4.27
4.27
4.27
4.25
3.70
3.69
3.68
3.68
3.68
3.66
2.68
2.66
2.66
2.66
2.64
2.30
2.29
2.18
2.16
2.16
2.15
2.15
2.13
2.13
2.11
2.11
2.10
1.11
1.12
1.00
1.12
1.03
1.12
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)

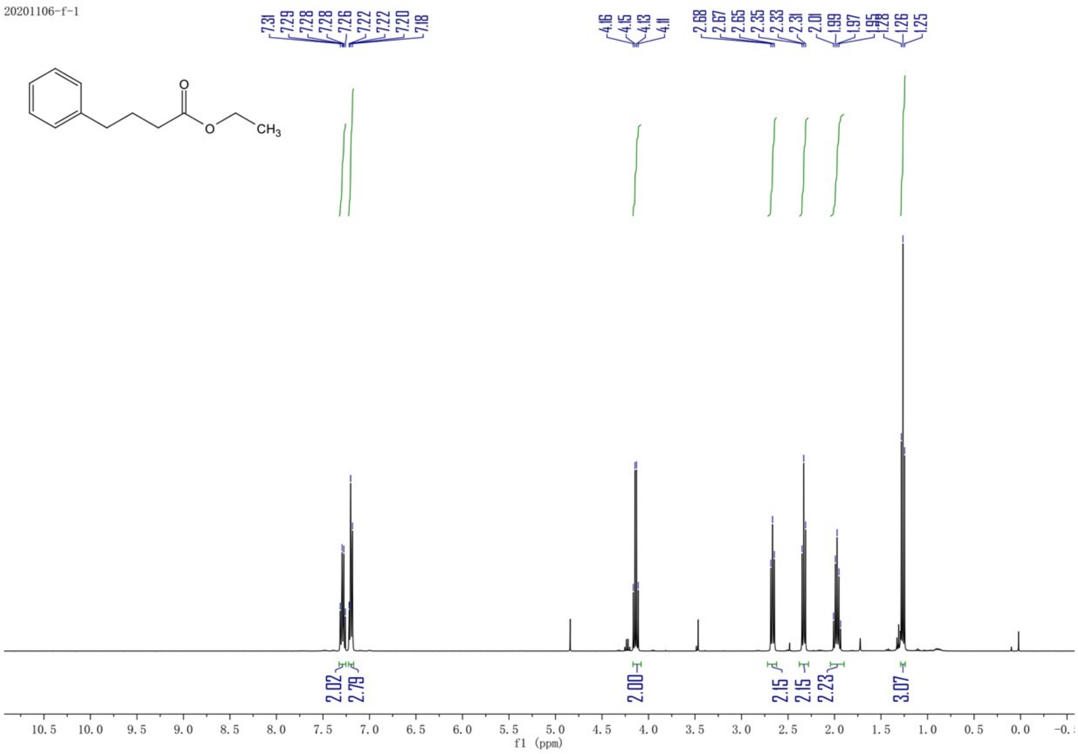

O
O
HS
176.57
66.47
34.69
31.86
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

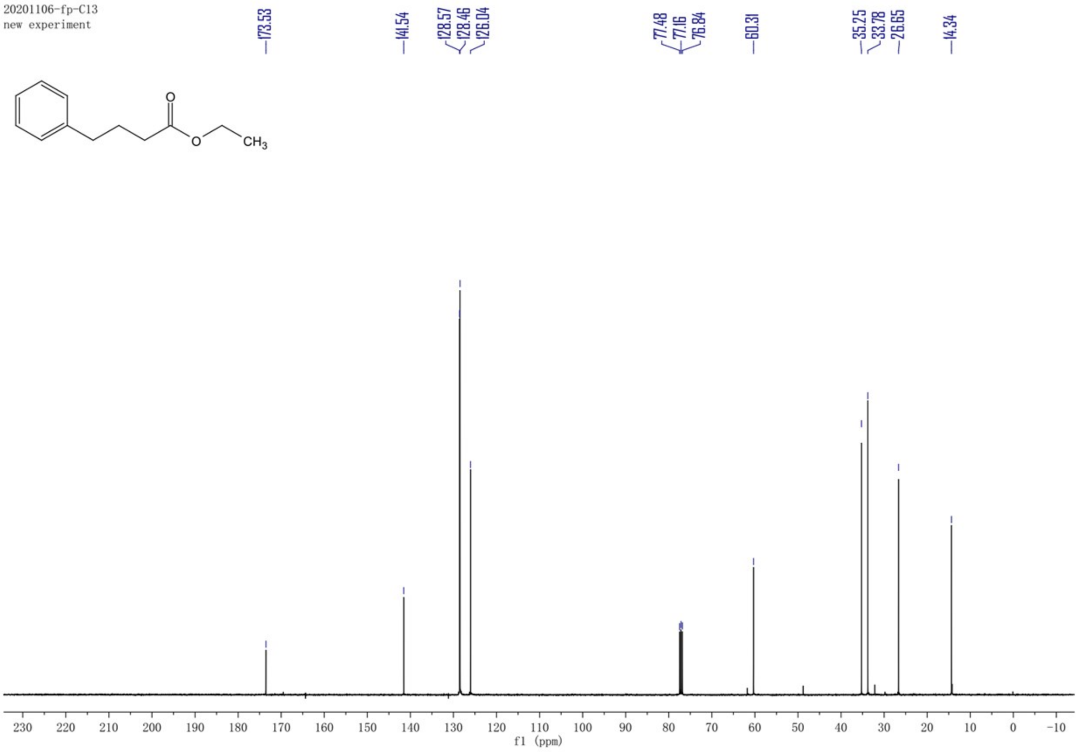
e


20201203-sh
STANDARD FLUORINE PARAMETERS
HS
O
O
7.39
7.38
7.38
7.37
7.37
5.18
3.31
3.29
2.05
2.03
2.01
4.79
2.08
2.00
0.94
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
f1 (ppm)

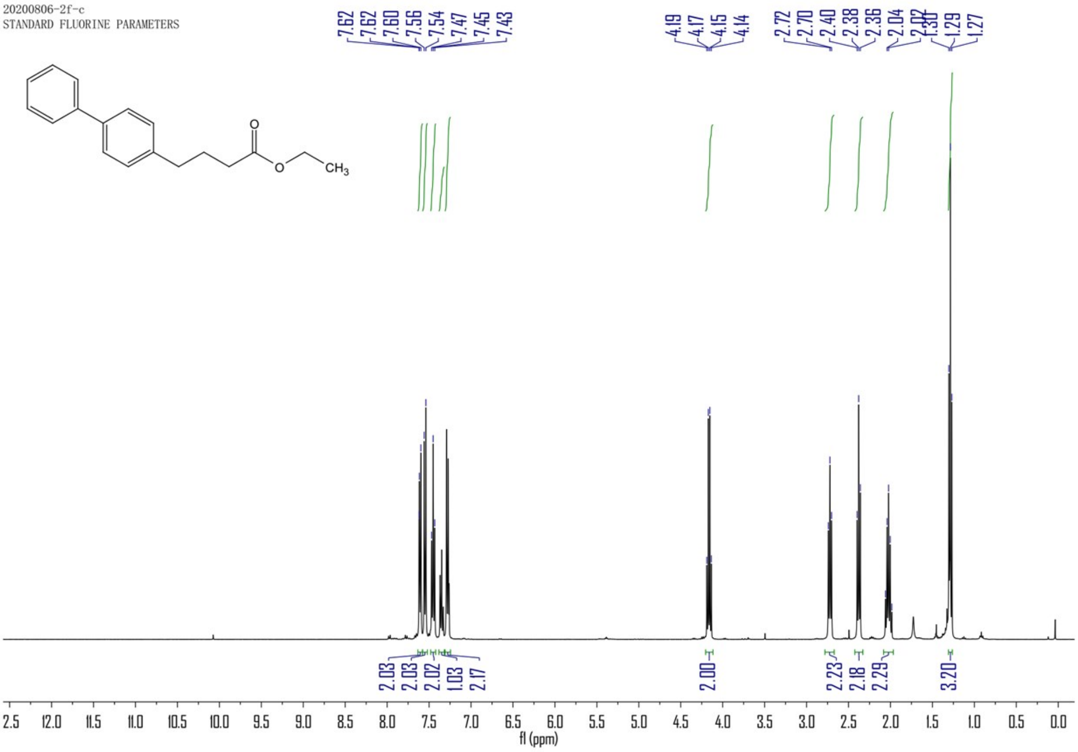

CARBON_01
170.67
135.41
128.67
128.51
128.38
67.41
26.57
HS
O
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

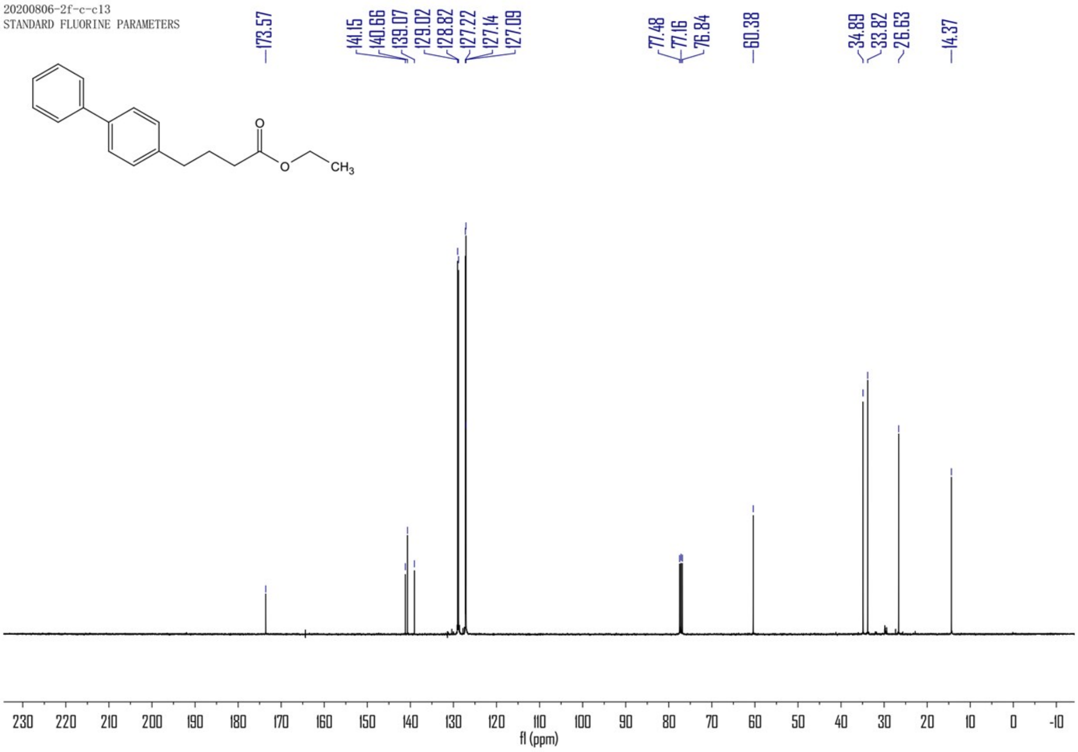
4


20201106-f-1
7.31
7.29
7.28
7.28
7.26
7.22
7.22
7.20
7.18
4.16
4.15
4.13
4.11
2.68
2.67
2.65
2.35
2.33
2.31
2.01
1.99
1.97
1.95
1.28
1.26
1.25
O
O
CH3
2.02
2.79
2.00
2.15
2.15
2.23
3.07
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

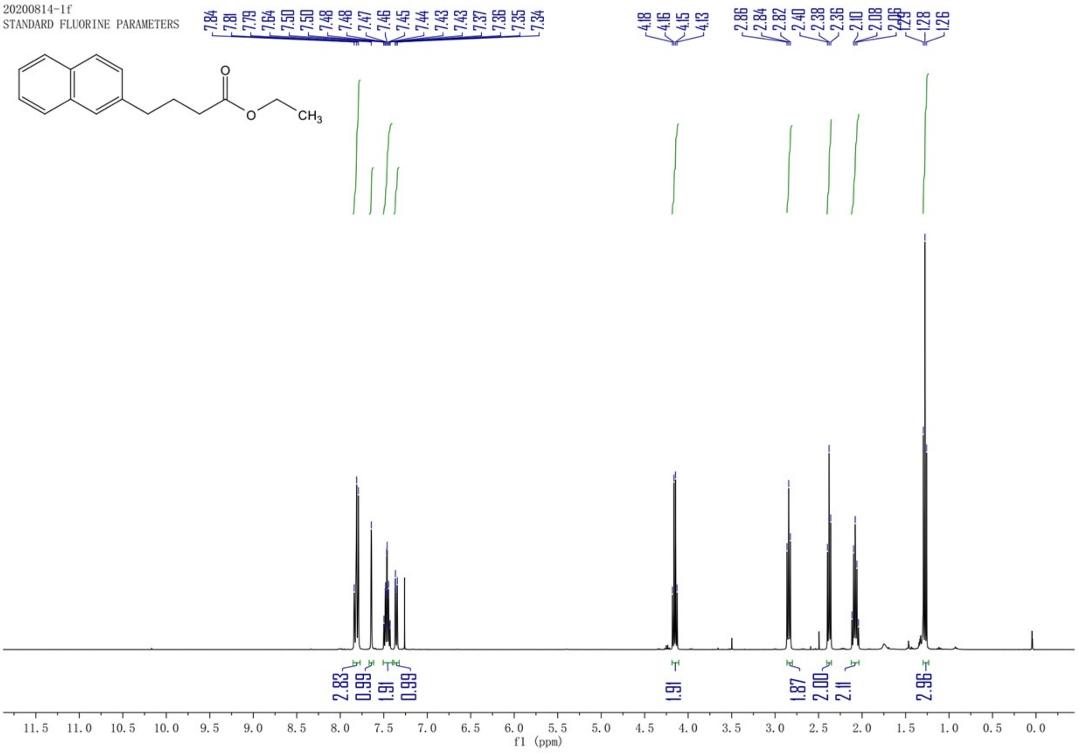

20201106-fp-C13
new experiment
173.53
141.54
128.57
128.46
126.04
77.48
77.16
76.84
60.31
35.25
33.78
26.65
14.34
O
O
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

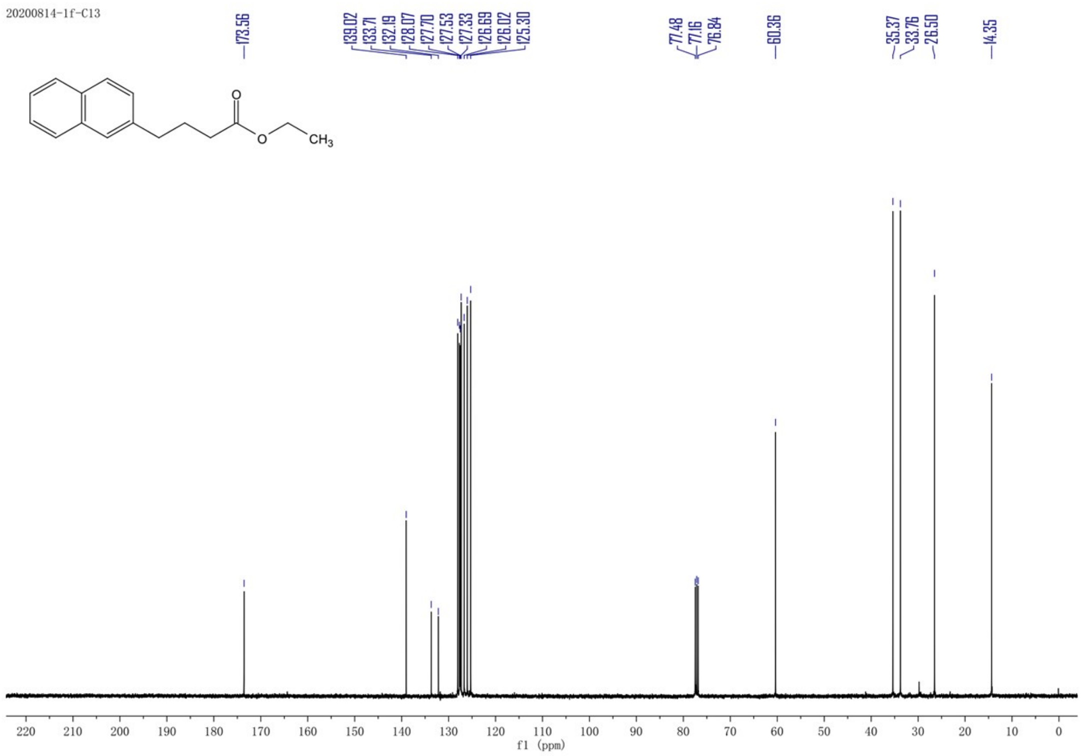
5


20200806-2f-c
STANDARD FLUORINE PARAMETERS
7.62
7.62
7.60
7.56
7.54
7.47
7.45
7.43
4.19
4.17
4.15
4.14
2.72
2.70
2.40
2.38
2.36
2.04
2.02
1.30
1.29
1.27
O
O
CH3
2.03
2.03
2.02
1.03
2.17
2.00
2.23
2.18
2.29
3.20
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

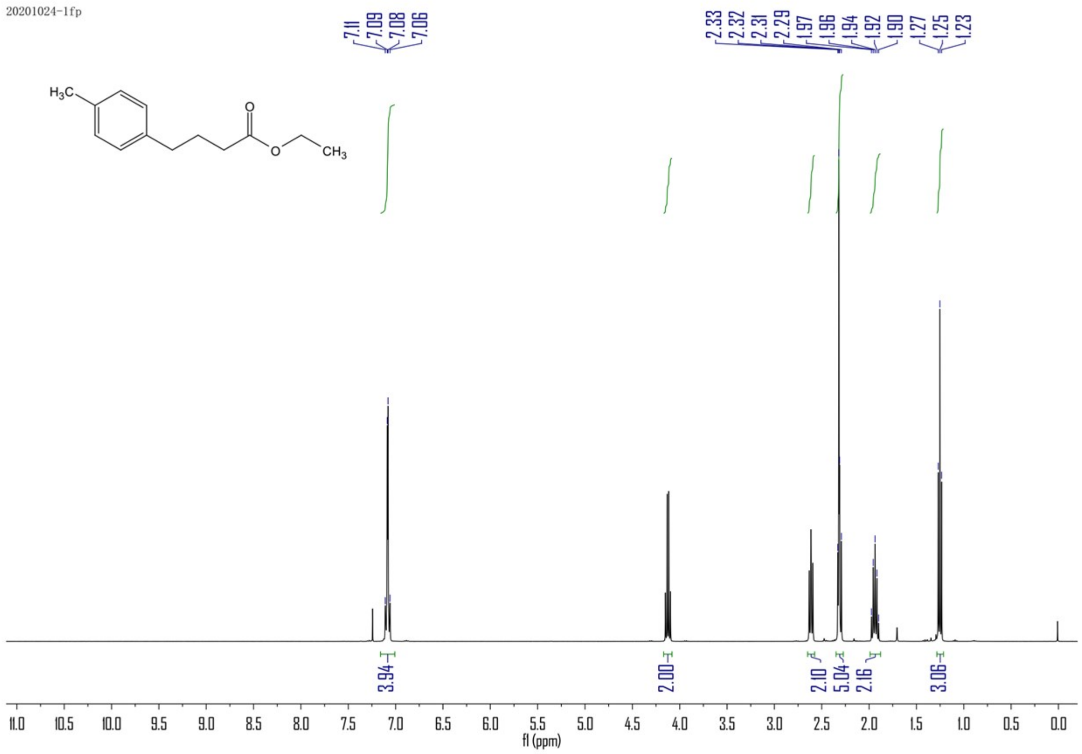

20200806-2f-c-c13
STANDARD FLUORINE PARAMETERS
173.57
141.15
140.66
139.07
129.02
128.82
127.22
127.14
127.09
77.48
77.16
76.84
60.38
34.89
33.82
26.63
14.37
O
O
CH3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

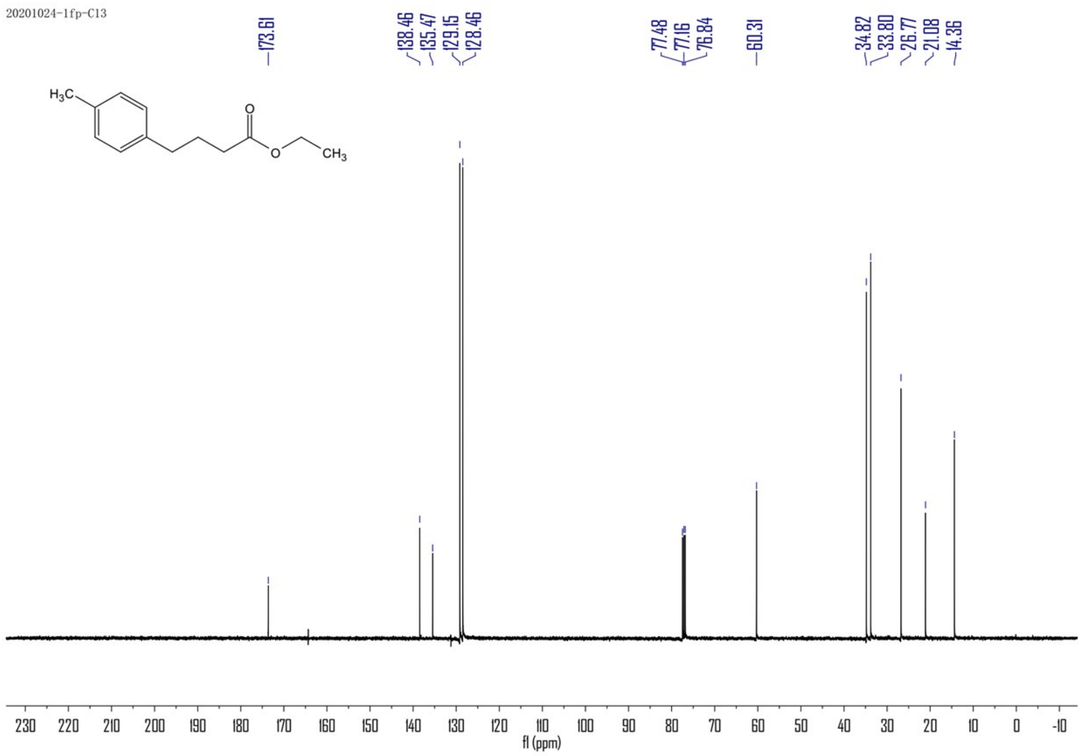
6


20200814-1f
STANDARD FLUORINE PARAMETERS
7.84
7.81
7.79
7.64
7.50
7.48
7.48
7.47
7.46
7.45
7.44
7.43
7.43
7.37
7.36
7.35
7.34
4.18
4.16
4.15
4.13
2.86
2.84
2.82
2.40
2.38
2.36
2.10
2.08
2.06
1.29
1.28
1.26
O
O
CH3
2.83
0.99
1.91
0.99
1.91
1.87
2.00
2.11
2.96
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

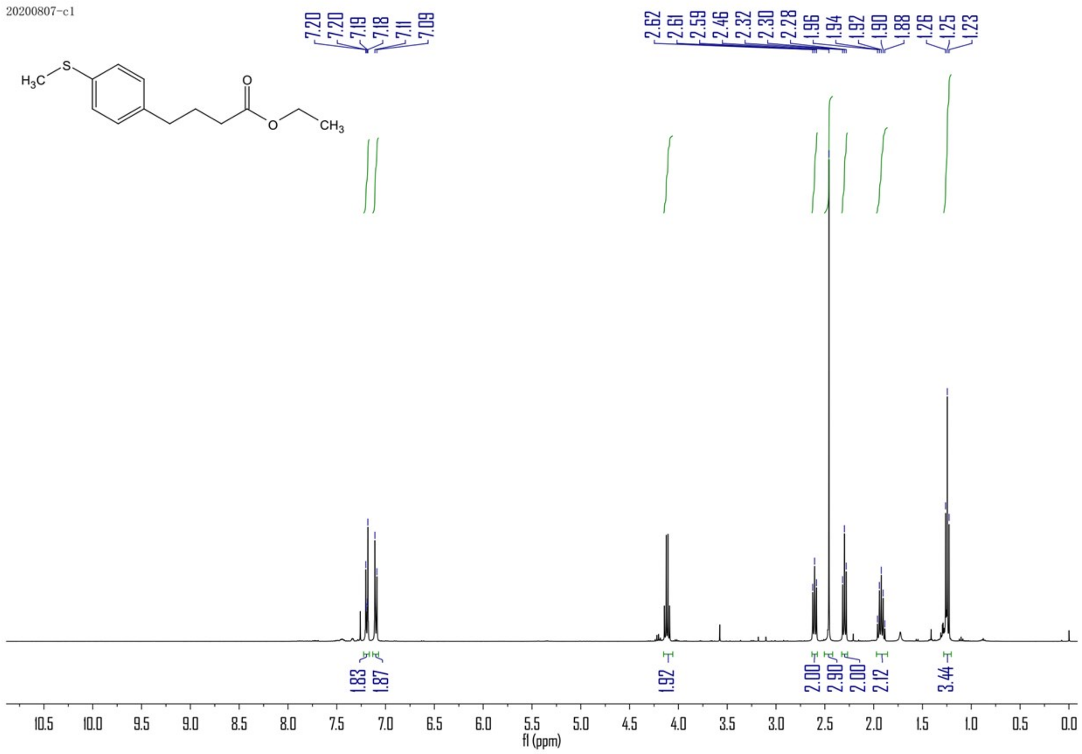

20200814-1f-C13
173.56
139.02
133.71
132.19
128.07
127.70
127.53
127.33
126.69
126.02
125.30
77.48
77.16
76.84
60.36
35.37
33.76
26.50
14.35
O
O
CH3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

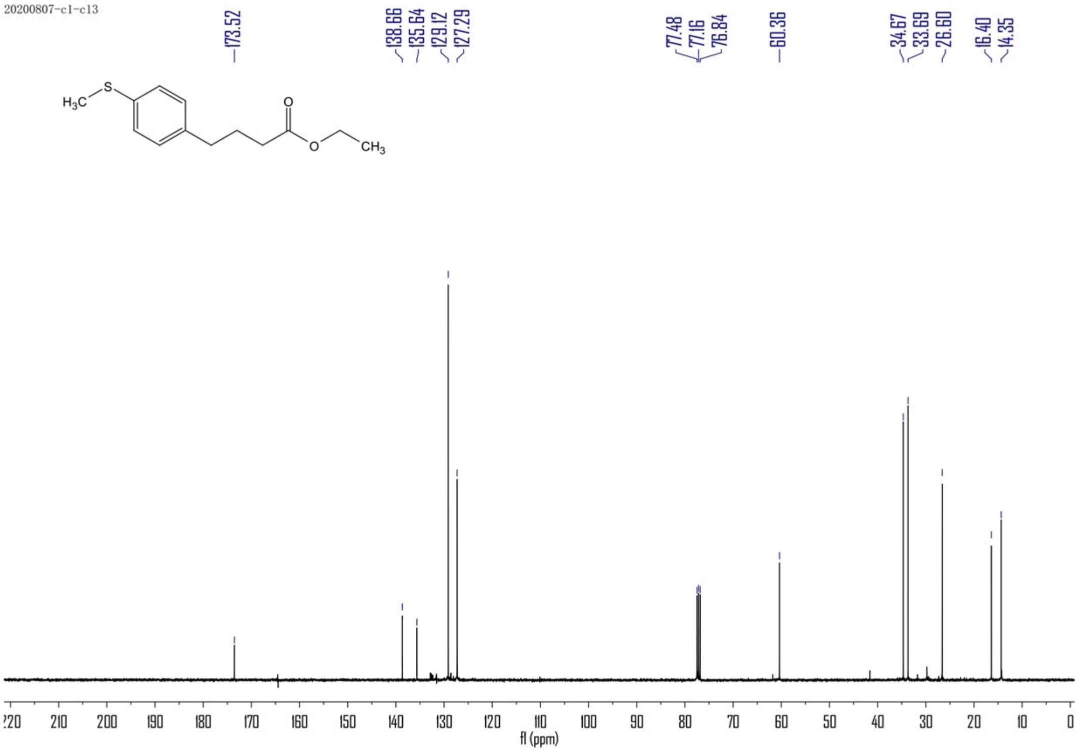
7


20201024-1fp
7.11
7.09
7.08
7.06
2.33
2.32
2.31
2.29
1.97
1.96
1.94
1.92
1.90
1.27
1.25
1.23
H3C
O
O
CH3
3.94
2.00
2.10
5.04
2.16
3.06
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

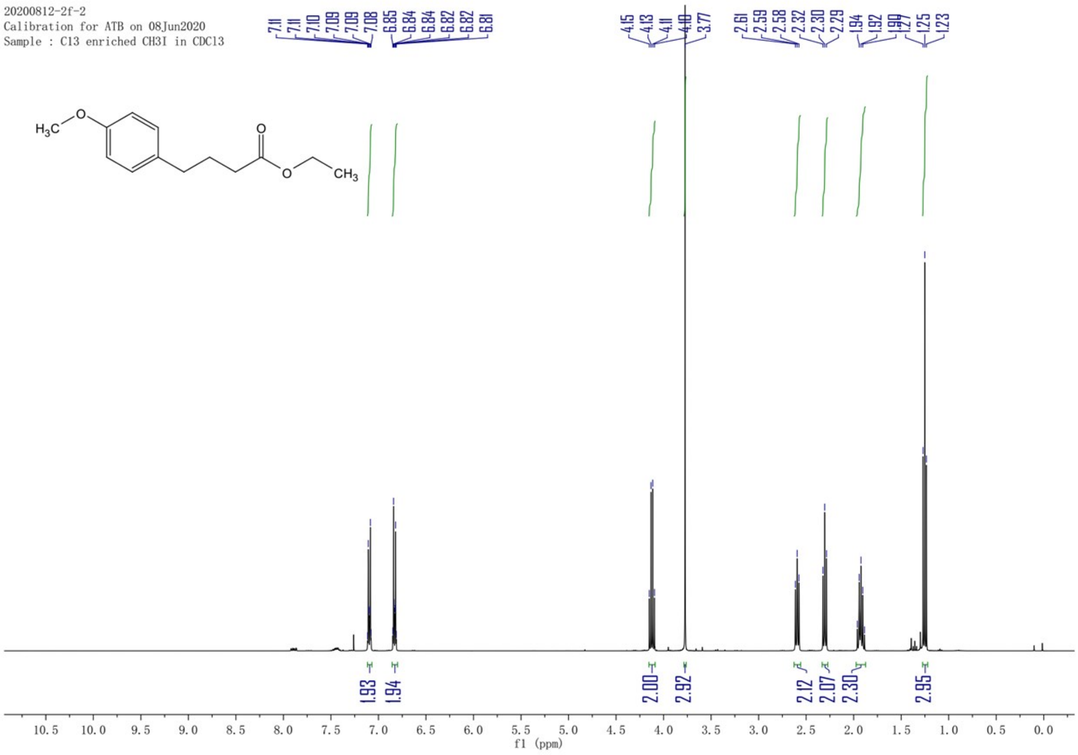

20201024-1fp-C13
173.61
138.46
135.47
129.15
128.46
77.48
77.16
76.84
60.31
34.82
33.80
26.77
21.08
14.36
H3C
O
O
CH3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
fl (ppm)

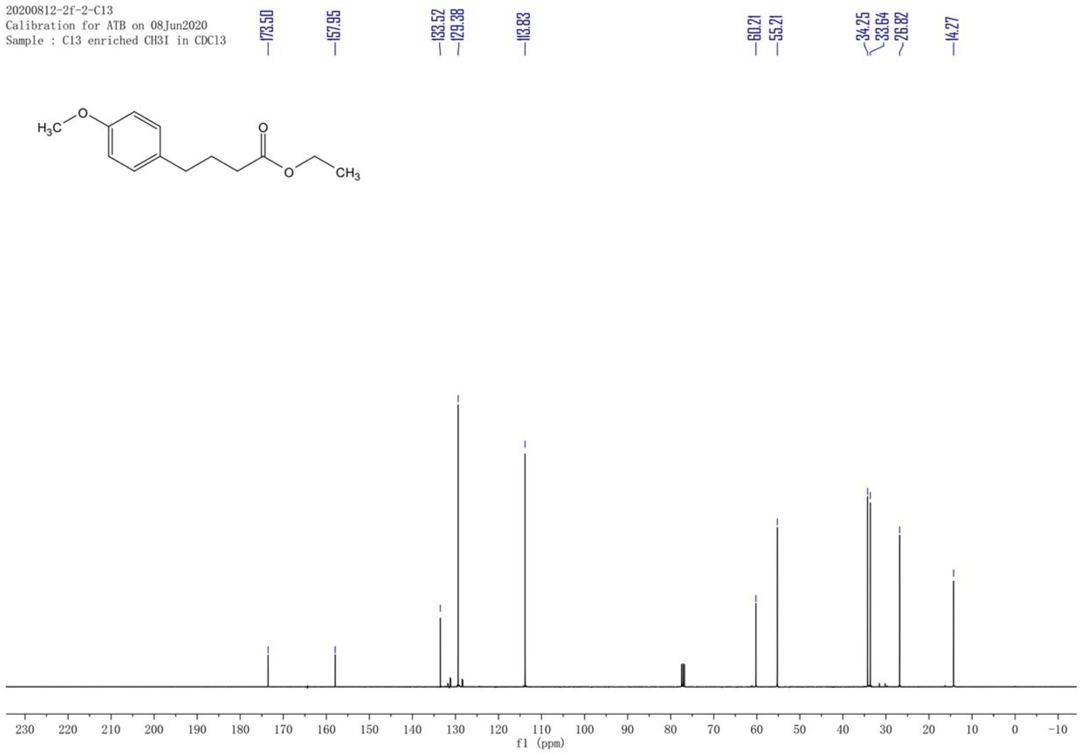
8


20200807-c1
7.20
7.20
7.19
7.18
7.11
7.09
2.62
2.61
2.59
2.46
2.32
2.30
2.28
1.96
1.94
1.92
1.90
1.88
1.26
1.25
1.23
H3C
S
O
O
CH3
1.83
1.87
1.92
2.00
2.90
2.00
2.12
3.44
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
fl (ppm)

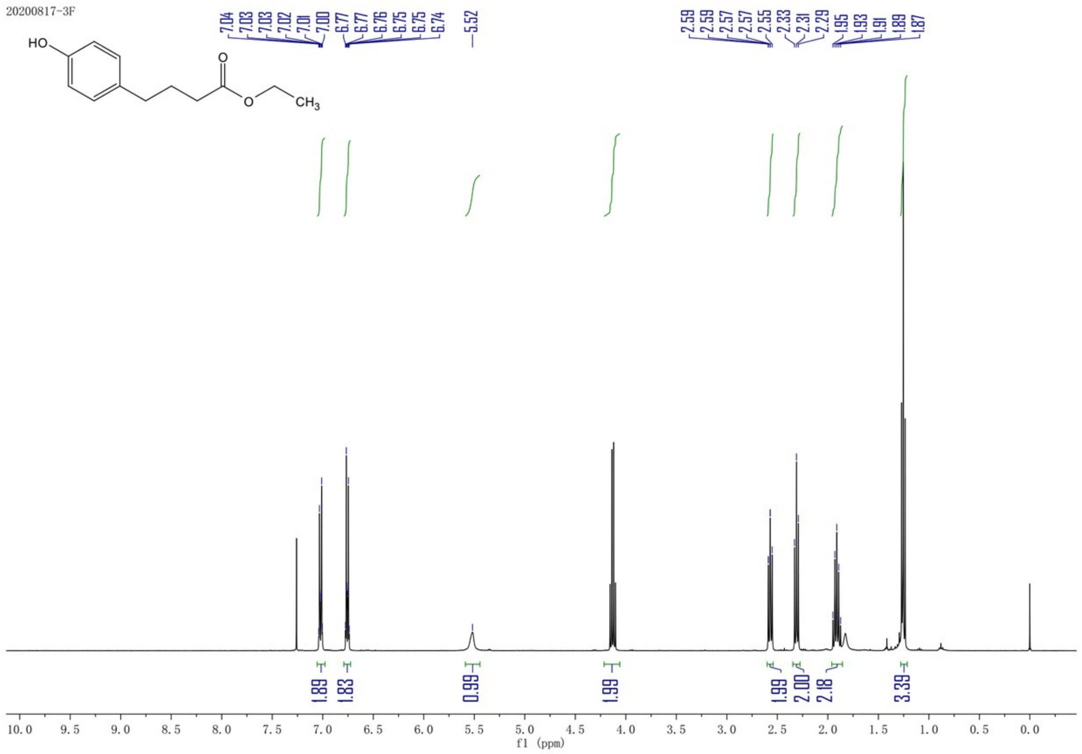

20200807-c1-c13
173.52
138.66
135.64
129.12
127.29
77.48
77.16
76.84
60.36
34.67
33.69
26.60
16.40
14.35
H3C
S
O
O
CH3
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

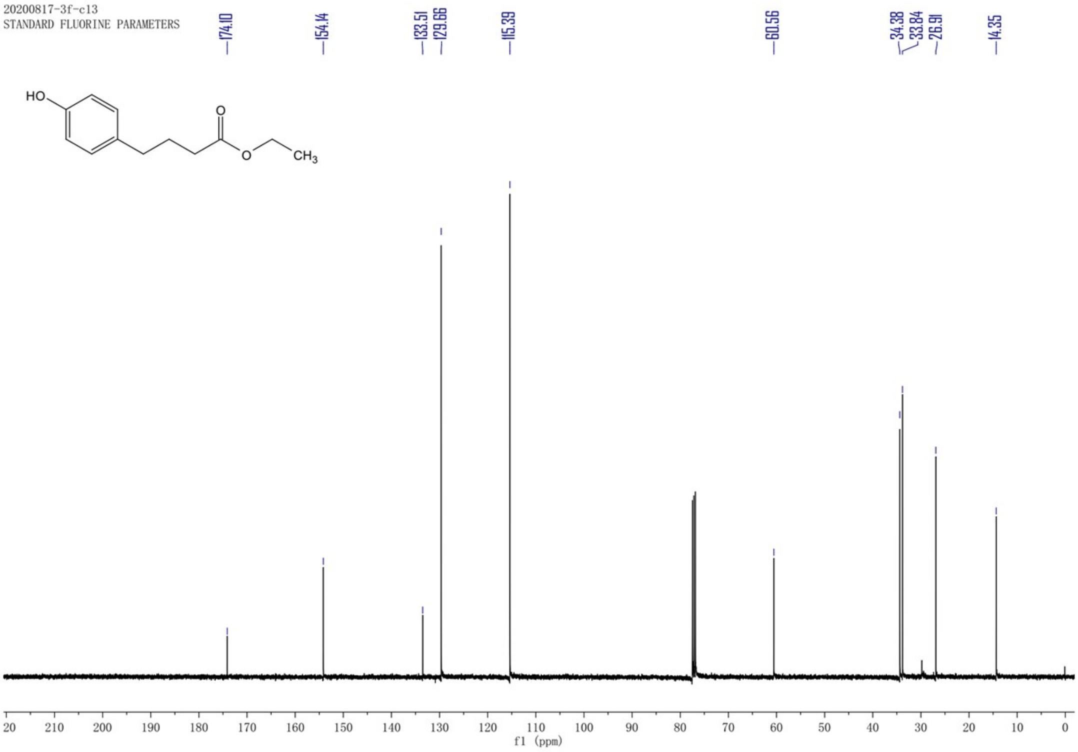
9


20200812-2f-2
Calibration for ATB on 08Jun2020
Sample : C13 enriched CH3I in CDCl3
7.11
7.11
7.10
7.09
7.09
7.08
6.85
6.84
6.84
6.82
6.82
6.81
4.15
4.13
4.11
4.10
3.77
2.61
2.59
2.58
2.32
2.30
2.29
1.94
1.92
1.90
1.27
1.25
1.23
H3C
O
O
O
CH3
1.93
1.94
2.00
2.92
2.12
2.07
2.30
2.95
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

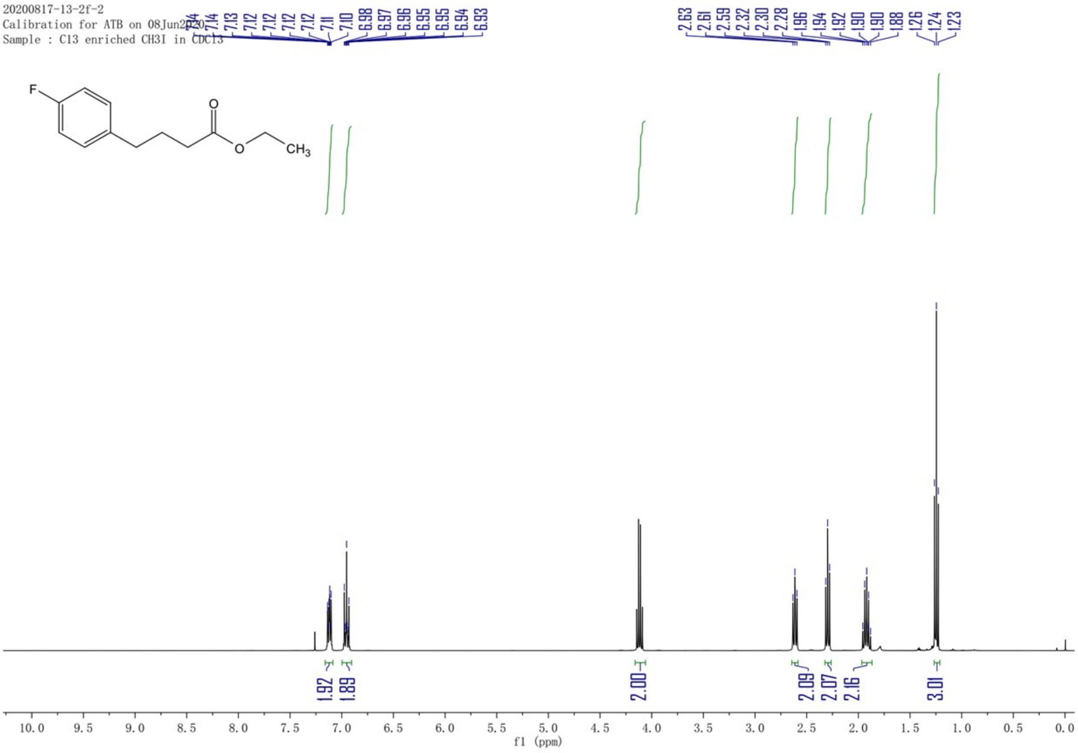

20200812-2f-2-C13
Calibration for ATB on 08Jun2020
Sample : C13 enriched CH3I in CDCl3
173.50
157.95
133.52
129.38
113.83
60.21
55.21
34.25
33.64
26.82
14.27
H_3C
O
O
O
CH_3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

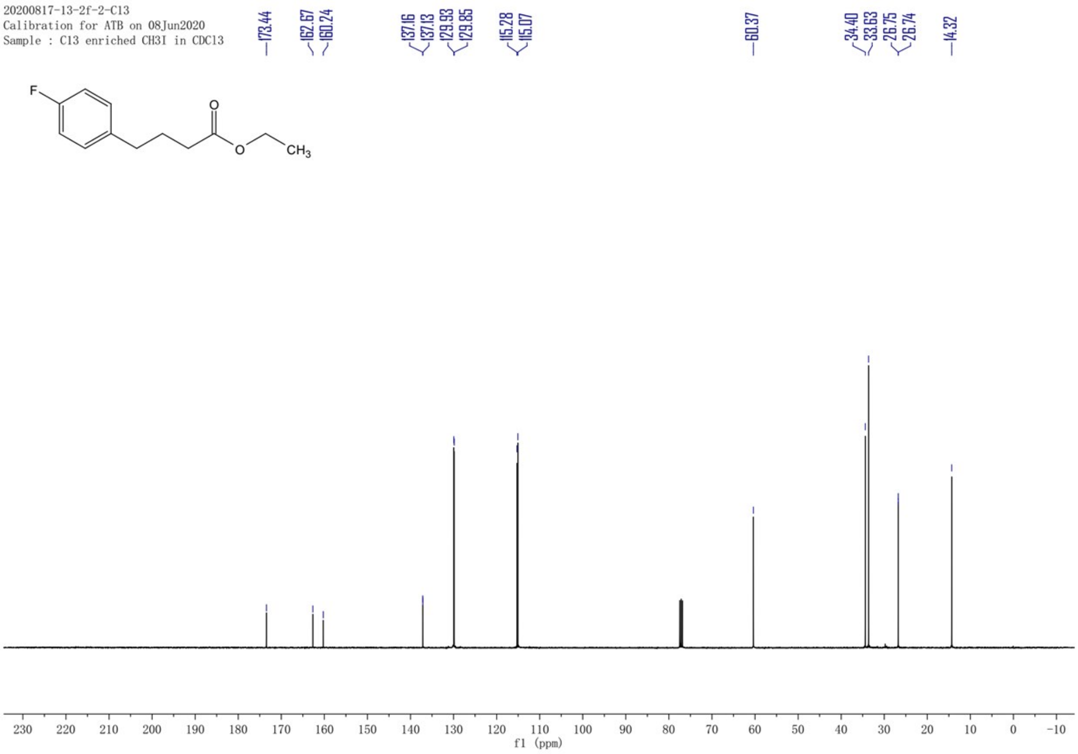
10

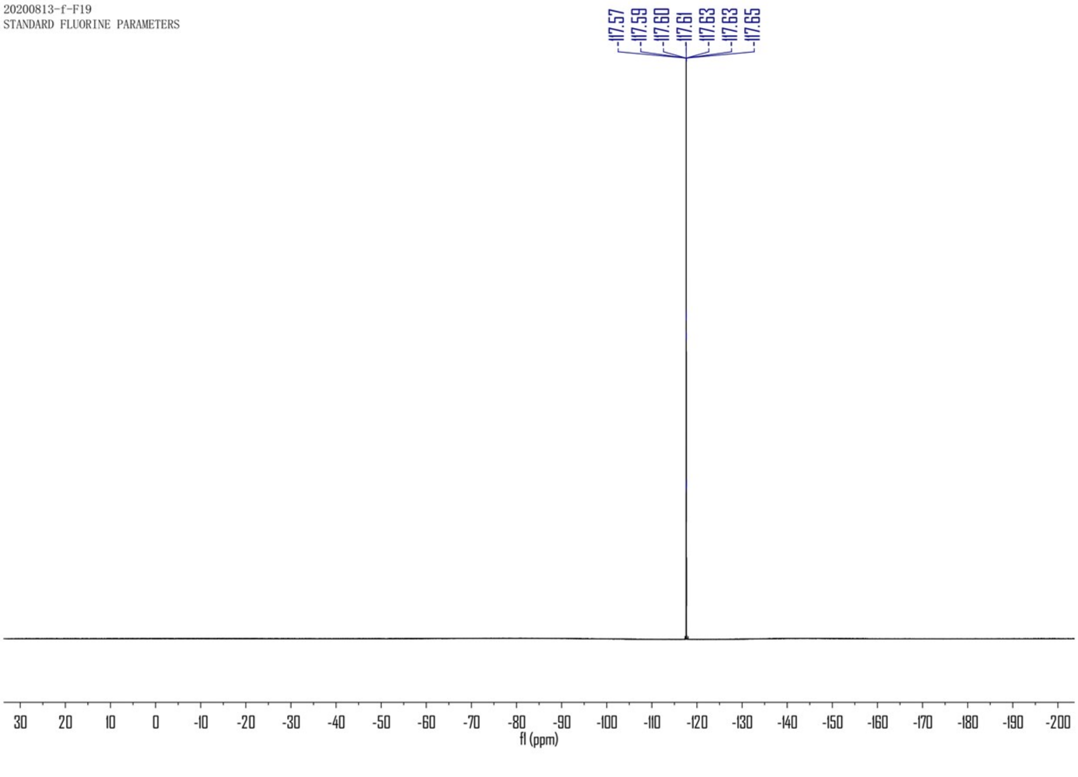

20200817-3F
HO
O
O
CH_3
7.04
7.03
7.03
7.02
7.01
7.00
6.77
6.77
6.76
6.75
6.75
6.74
5.52
2.59
2.59
2.57
2.57
2.55
2.33
2.31
2.29
1.95
1.93
1.91
1.89
1.87
1.89
1.83
0.99
1.99
1.99
2.00
2.18
3.39
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)


20200817-3f-c13
STANDARD FLUORINE PARAMETERS
174.10
154.14
133.51
129.66
115.39
60.56
34.38
33.84
26.91
14.35
HO
O
O
CH3
20
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

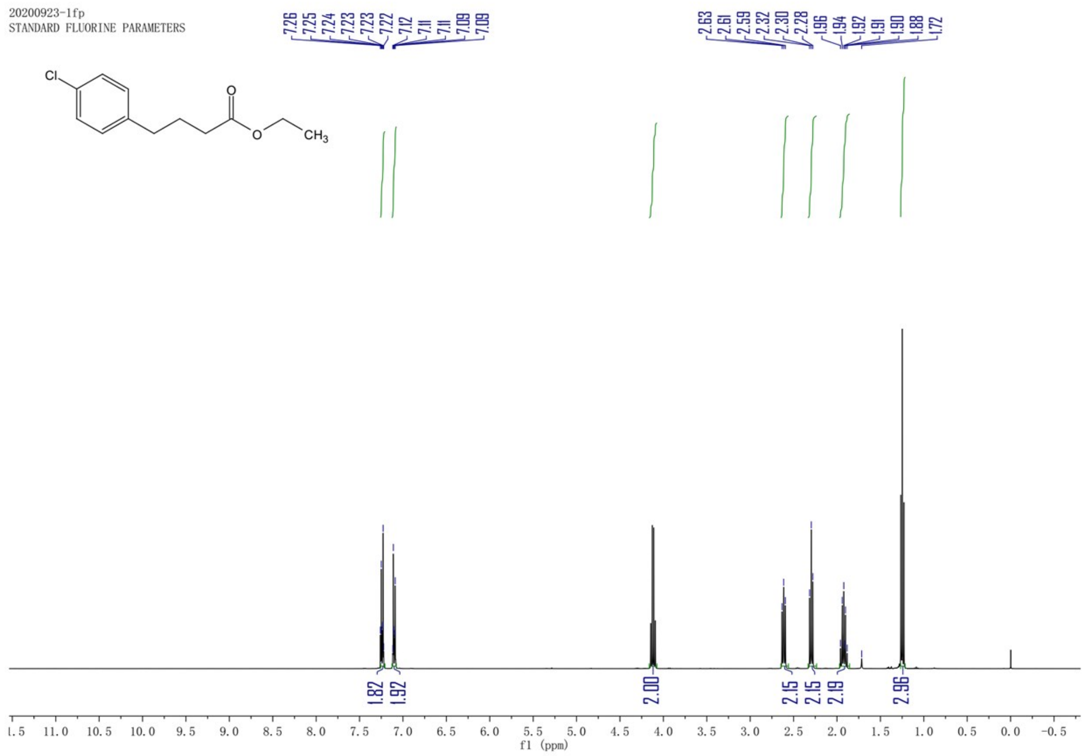
11

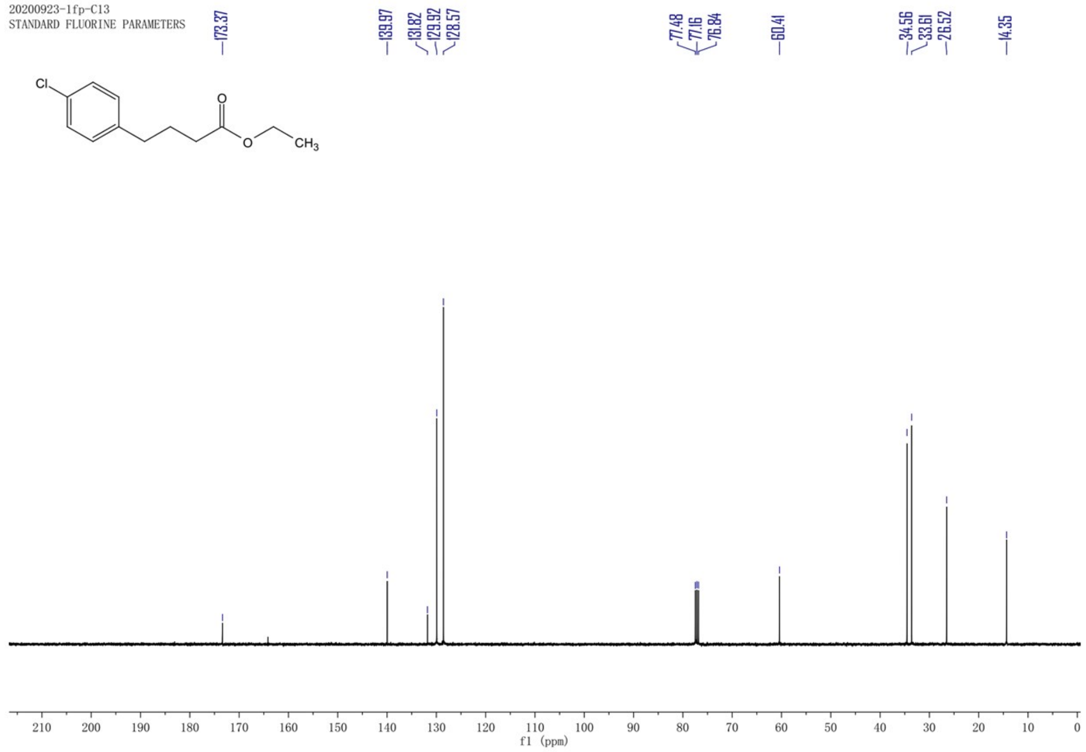

20200817-13-2f-2
Calibration for ATB on 08Jun2020
Sample : C13 enriched CH3I in CDCl3
7.14
7.14
7.13
7.12
7.12
7.12
7.12
7.11
7.10
6.98
6.97
6.96
6.95
6.95
6.94
6.93
2.63
2.61
2.59
2.32
2.30
2.28
1.96
1.94
1.92
1.90
1.90
1.88
1.26
1.24
1.23
F
O
O
CH3
1.92
1.89
2.00
2.09
2.07
2.16
3.01
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


20200817-13-2f-2-C13
Calibration for ATB on 08Jun2020
Sample : C13 enriched CH3I in CDCl3
173.44
162.67
160.24
137.16
137.13
129.93
129.85
115.28
115.07
60.37
34.40
33.63
26.75
26.74
14.32
F
O
O
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

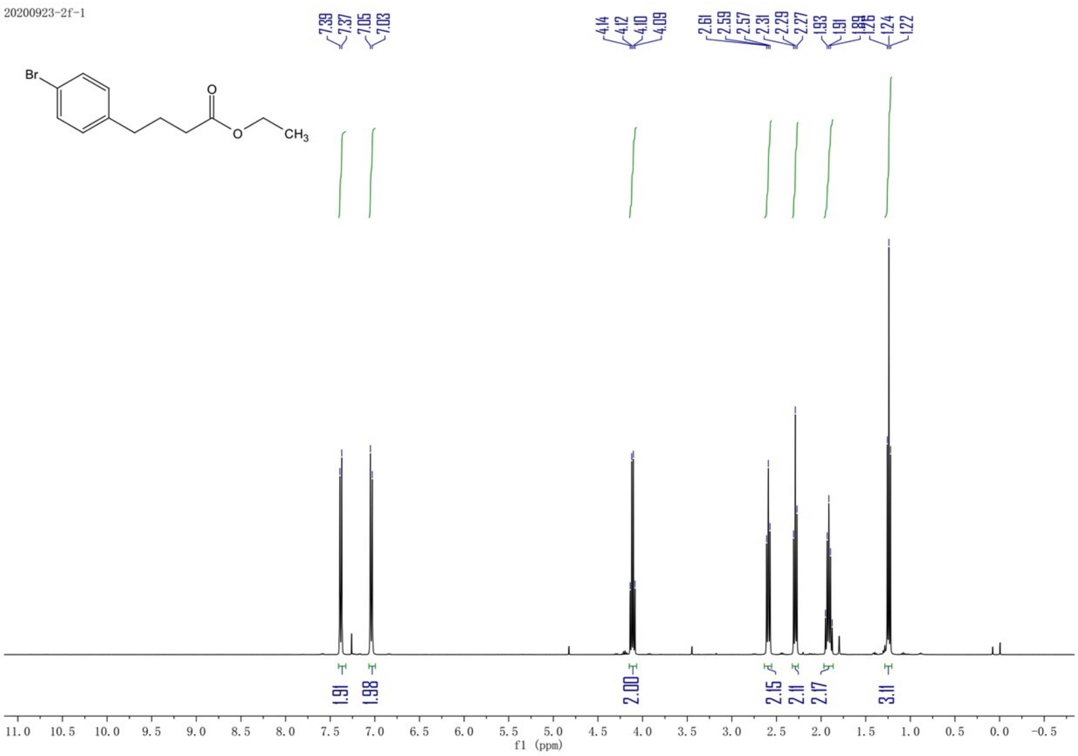

20200813-f-F19
STANDARD FLUORINE PARAMETERS
-117.57
-117.59
-117.60
-117.61
-117.63
-117.63
-117.65
30
20
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
f1 (ppm)

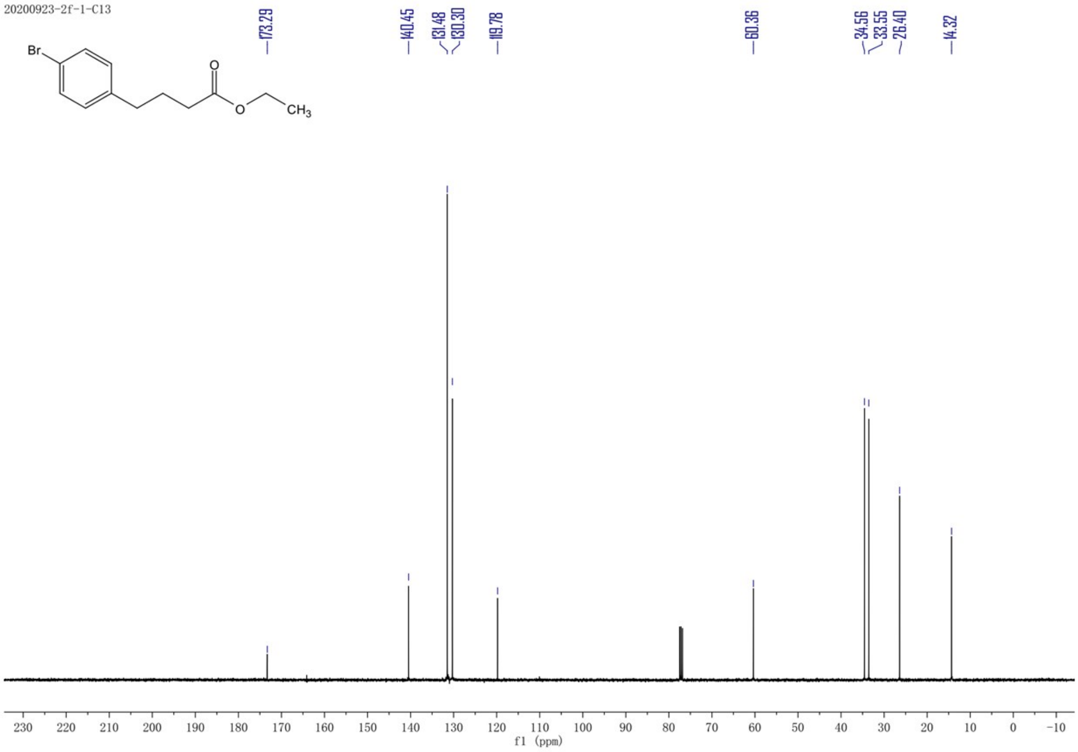
12


20200923-1fp
STANDARD FLUORINE PARAMETERS
7.26 7.25 7.24 7.23 7.23 7.22 7.12 7.11 7.11 7.09 7.09
2.63 2.61 2.59 2.32 2.30 2.28 1.96 1.94 1.92 1.91 1.90 1.88 1.72
Cl
O
O
CH3
1.82 1.92 2.00 2.15 2.15 2.19 2.96
1.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

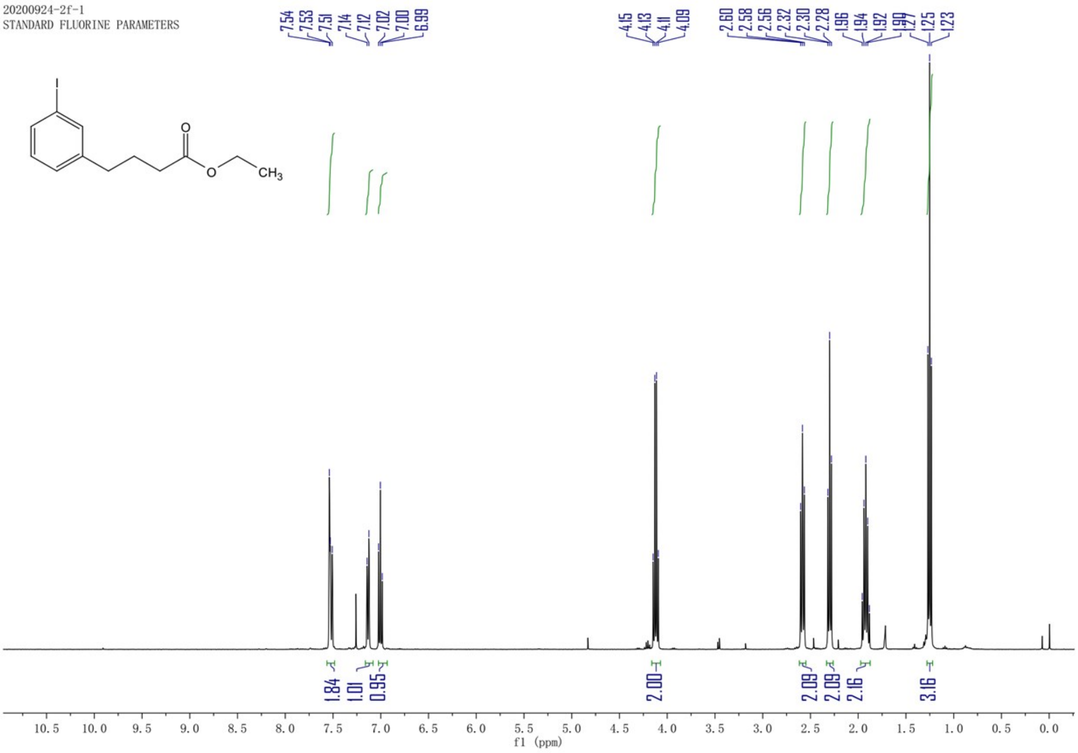

20200923-1fp-C13
STANDARD FLUORINE PARAMETERS
173.37
139.97 131.82 129.92 128.57
77.48 77.16 76.84
60.41
34.56 33.61 26.52
14.35
Cl
O
O
CH3
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

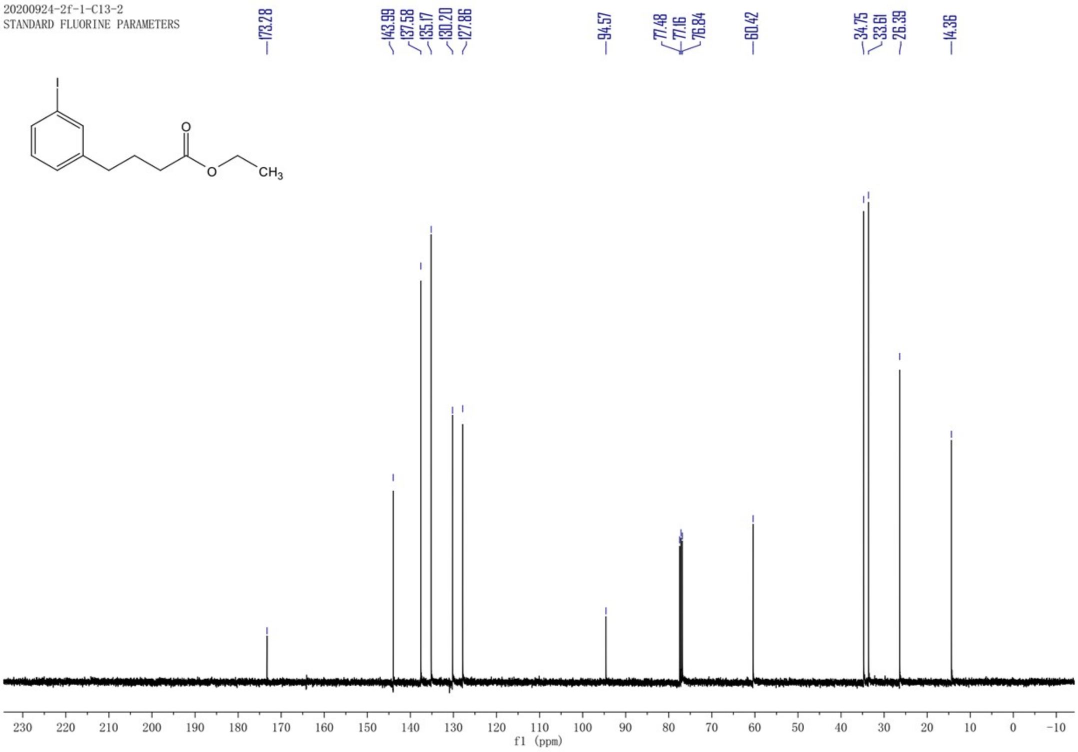
13


20200923-2f-1
Br
O
O
CH3
7.39
7.37
7.05
7.03
4.14
4.12
4.10
4.09
2.61
2.59
2.57
2.31
2.29
2.27
1.93
1.91
1.89
1.26
1.24
1.22
1.91
1.98
2.00
2.15
2.11
2.17
3.11
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)

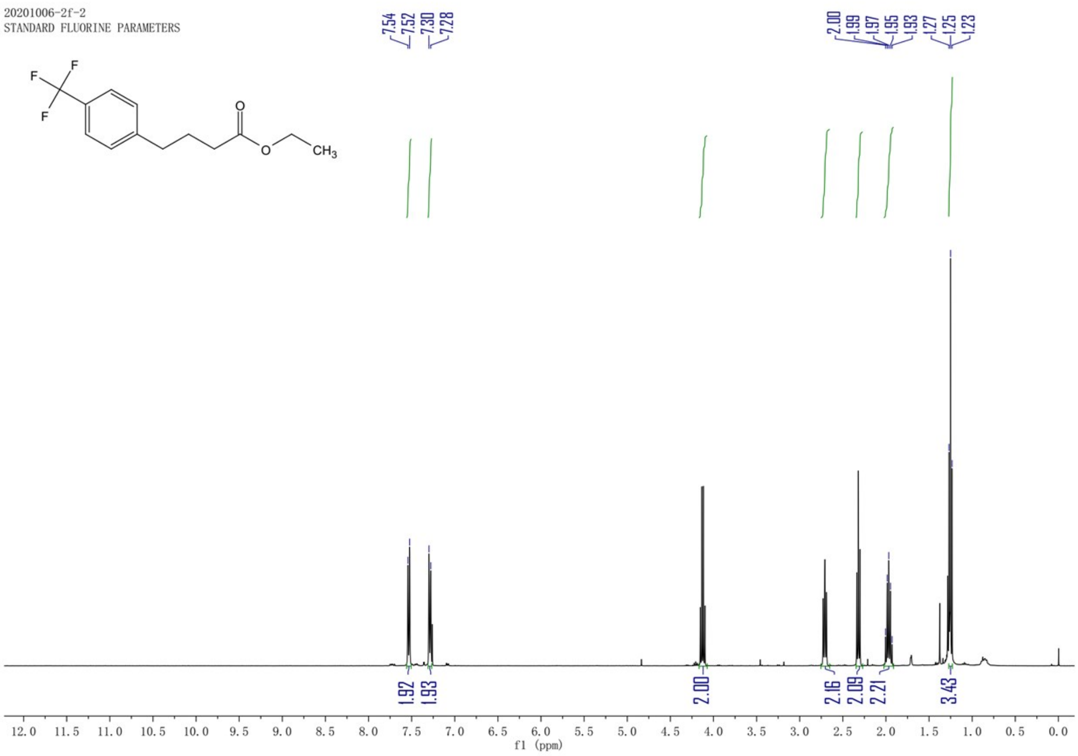

20200923-2f-1-C13
Br
O
O
CH3
173.29
140.45
131.48
130.30
119.78
60.36
34.56
33.55
26.40
14.32
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

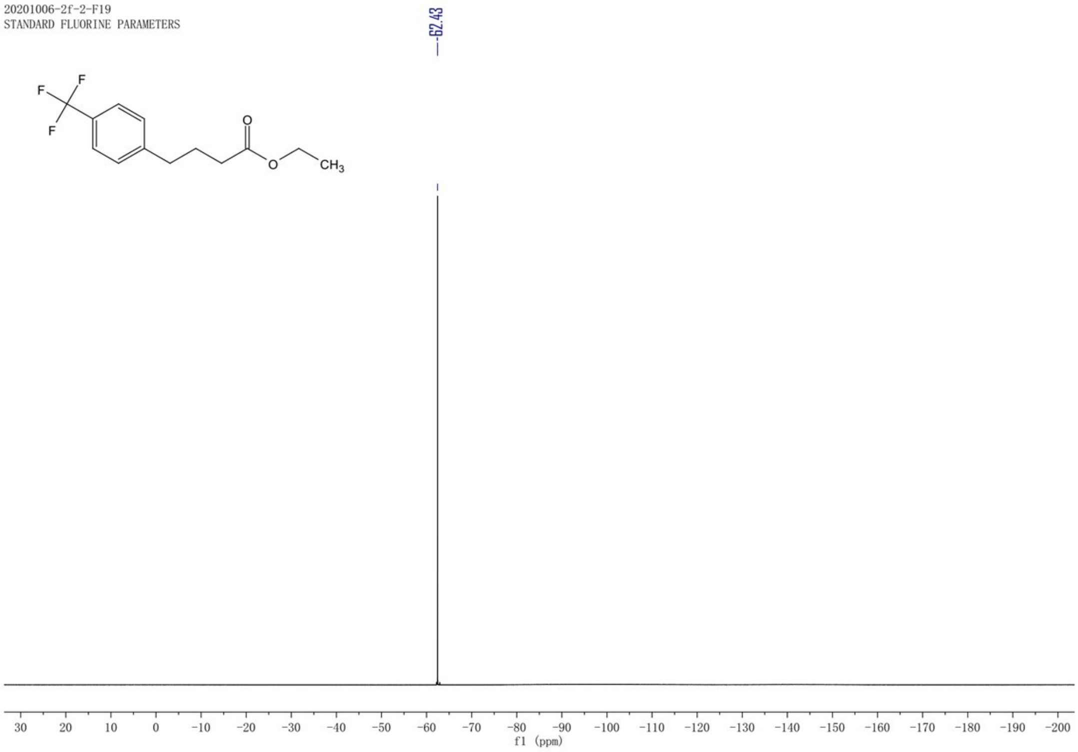
14


20200924-2f-1
STANDARD FLUORINE PARAMETERS
I
O
O
CH3
7.54
7.53
7.51
7.14
7.12
7.02
7.00
6.99
4.15
4.13
4.11
4.09
2.60
2.58
2.56
2.32
2.30
2.28
1.96
1.94
1.92
1.90
1.27
1.25
1.23
1.84
1.01
0.95
2.00
2.09
2.09
2.16
3.16
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

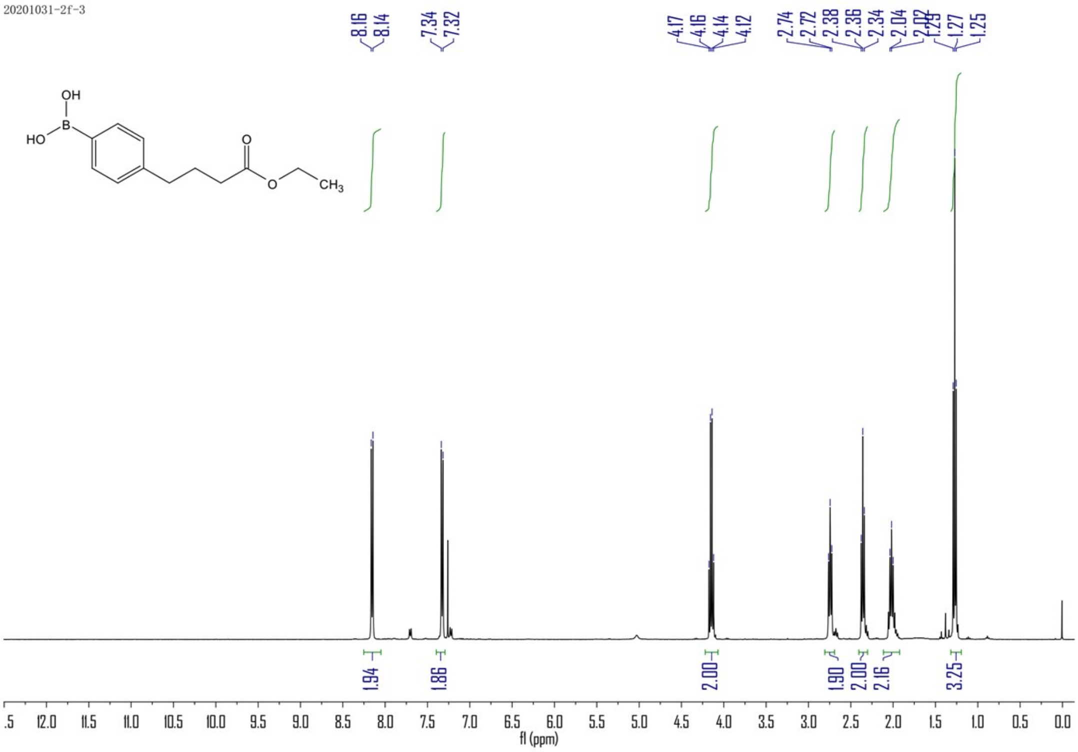

20200924-2f-1-C13-2
STANDARD FLUORINE PARAMETERS
I
O
O
CH3
173.28
143.99
137.58
135.17
130.20
127.86
94.57
77.48
77.16
76.84
60.42
34.75
33.61
26.39
14.36
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

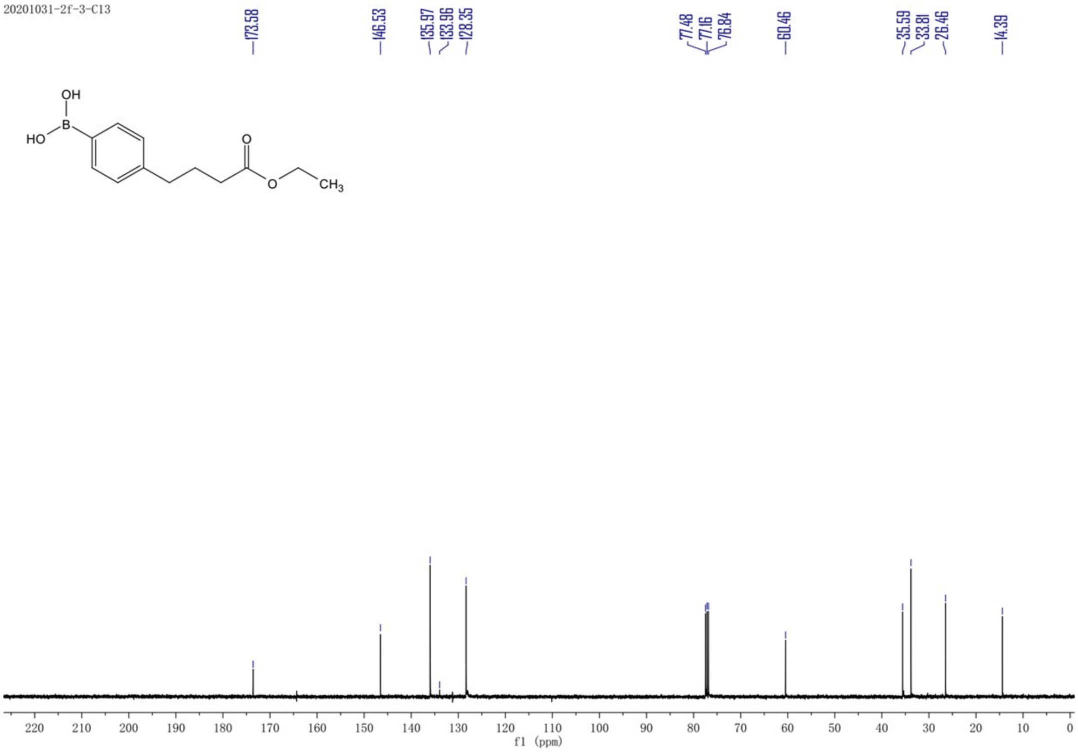
15


20201006-2f-2
STANDARD FLUORINE PARAMETERS
F
F
F
O
O
CH3
7.54
7.52
7.30
7.28
2.00
1.99
1.97
1.95
1.93
1.27
1.25
1.23
1.92
1.93
2.00
2.16
2.09
2.21
3.43
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

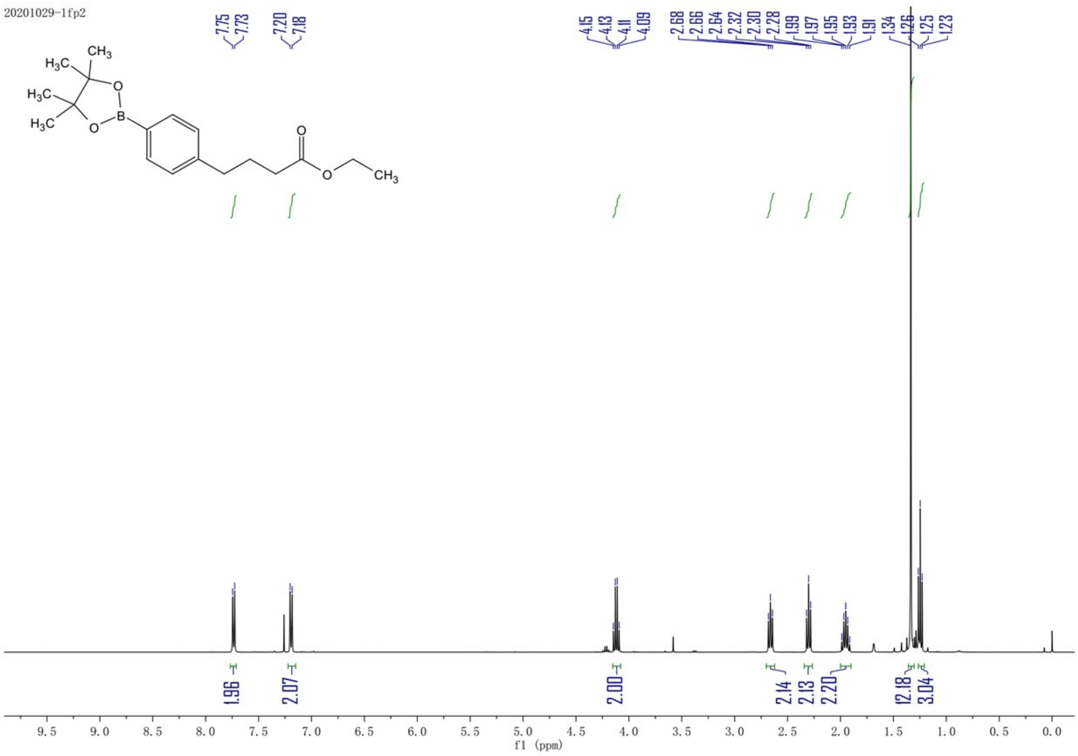

20201006-2f-2-F19
STANDARD FLUORINE PARAMETERS
-62.43
F
F
F
O
O
CH3
30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200
f1 (ppm)

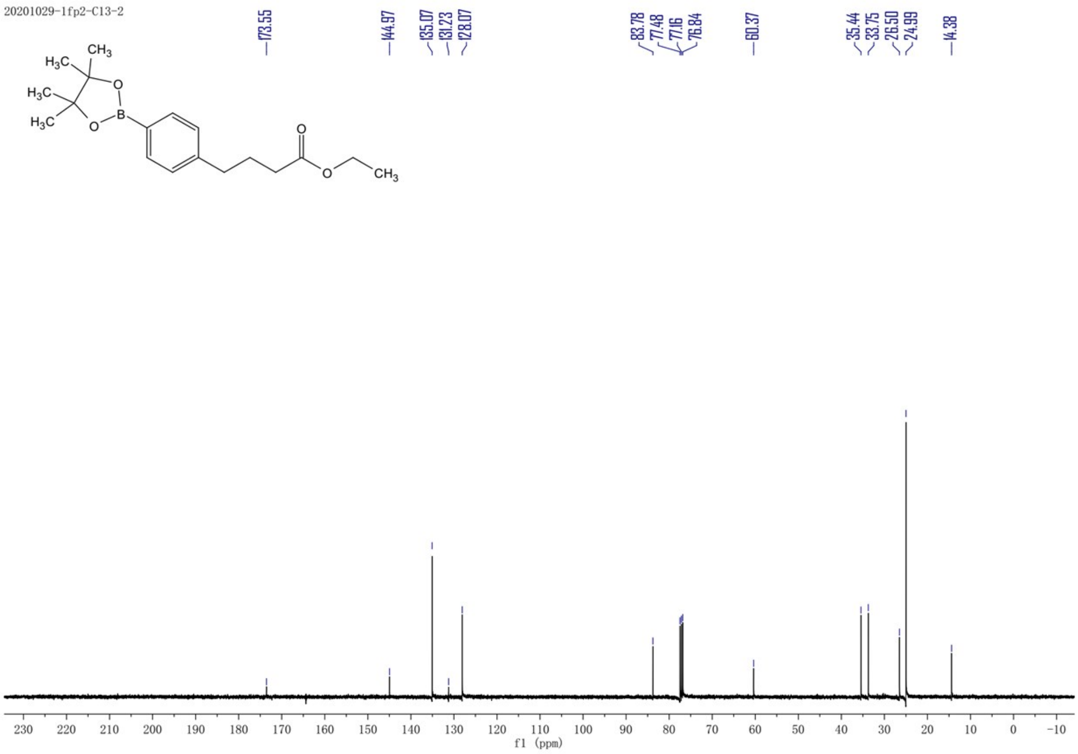
16


20201031-2f-3
8.16
8.14
7.34
7.32
4.17
4.16
4.14
4.12
2.74
2.72
2.38
2.36
2.34
2.04
2.02
1.29
1.27
1.25
OH
B
HO
O
O
CH3
1.94
1.86
2.00
1.90
2.00
2.16
3.25
.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

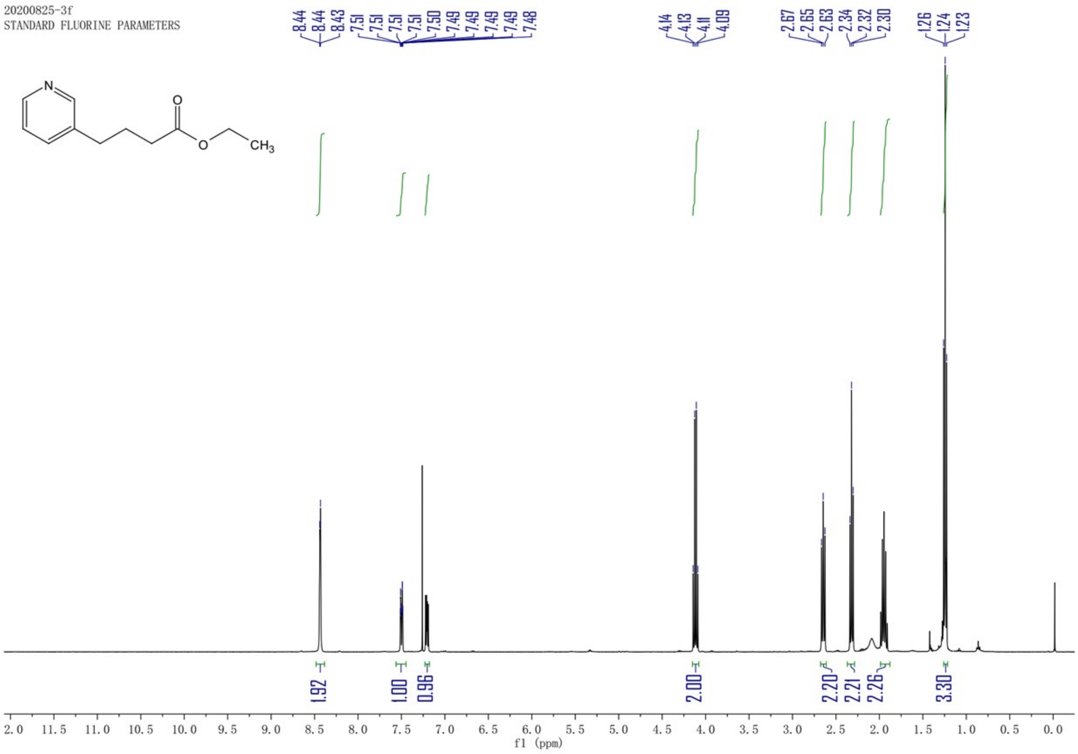

20201031-2f-3-C13
173.58
146.53
135.97
133.96
128.35
77.48
77.16
76.84
60.46
35.59
33.81
26.46
14.39
OH
B
HO
O
O
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

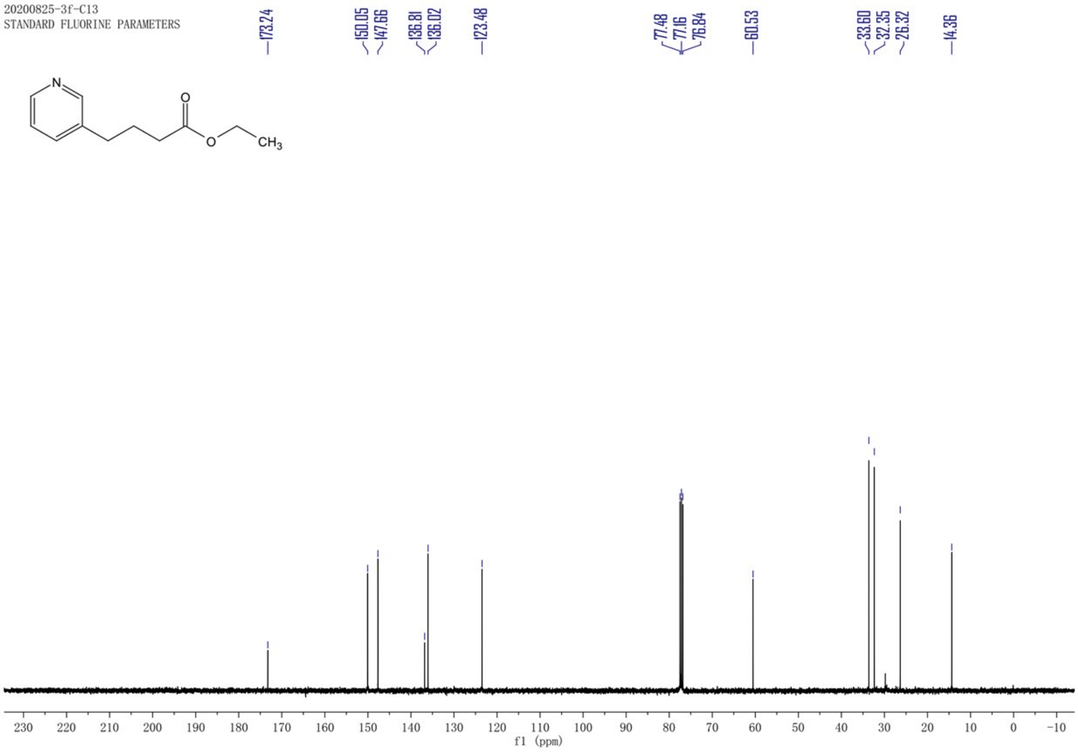
17


20201029-1fp2
H3C
CH3
H3C
H3C
O
B
O
O
O
CH3
7.75
7.73
7.20
7.18
4.15
4.13
4.11
4.09
2.68
2.66
2.64
2.32
2.30
2.28
1.99
1.97
1.95
1.93
1.91
1.34
1.26
1.25
1.23
1.96
2.07
2.00
2.14
2.13
2.20
12.18
3.04
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

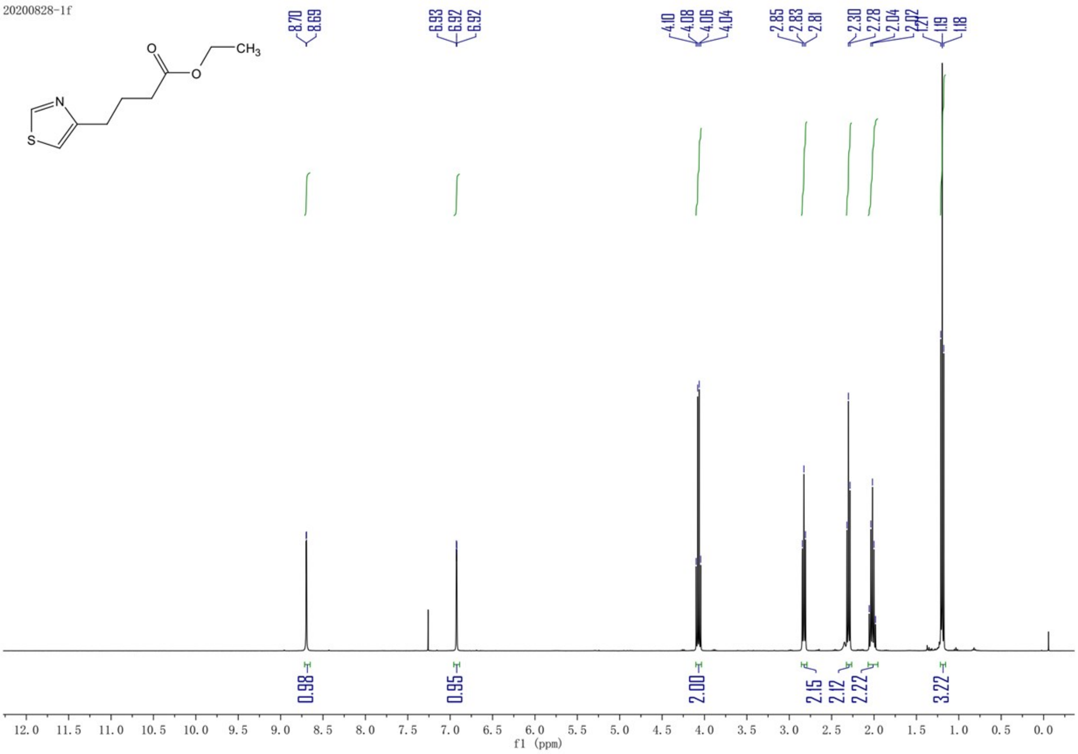

20201029-1fp2-C13-2
H3C
CH3
H3C
H3C
O
B
O
O
O
CH3
173.55
144.97
135.07
131.23
128.07
83.78
77.48
77.16
76.84
60.37
35.44
33.75
26.50
24.99
14.38
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

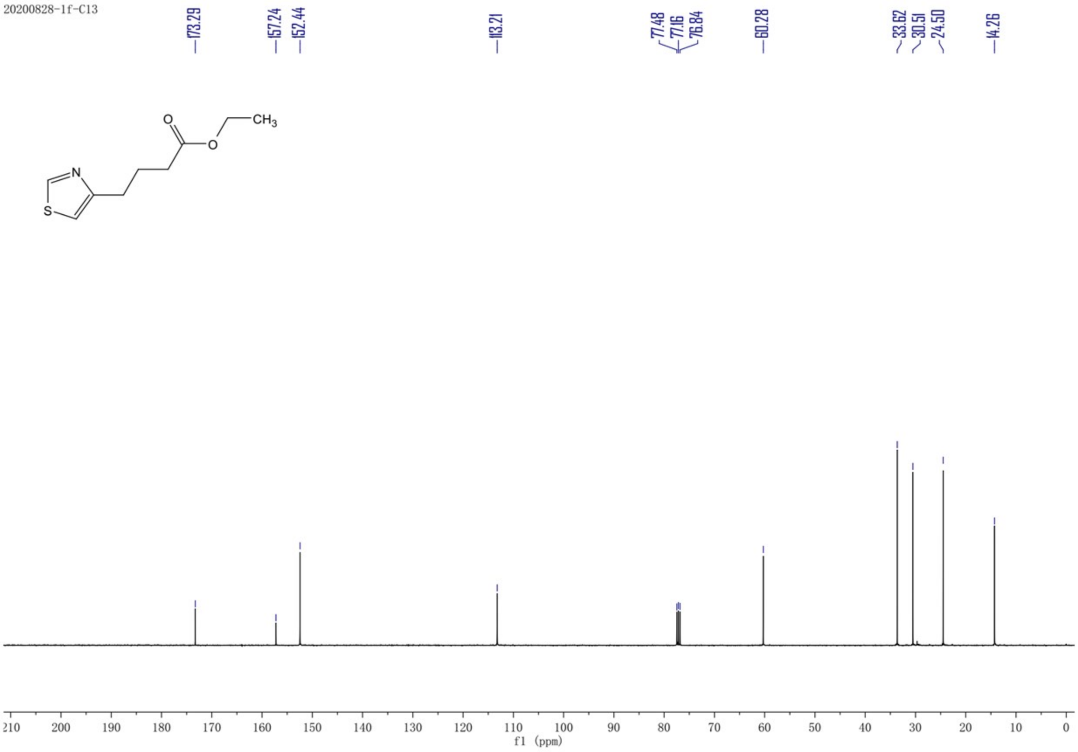
18


20200825-3f
STANDARD FLUORINE PARAMETERS
N
O
O
CH3
8.44
8.44
8.43
7.51
7.51
7.51
7.51
7.50
7.49
7.49
7.49
7.49
7.48
4.14
4.13
4.11
4.09
2.67
2.65
2.63
2.34
2.32
2.30
1.26
1.24
1.23
1.92
1.00
0.96
2.00
2.20
2.21
2.26
3.30
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

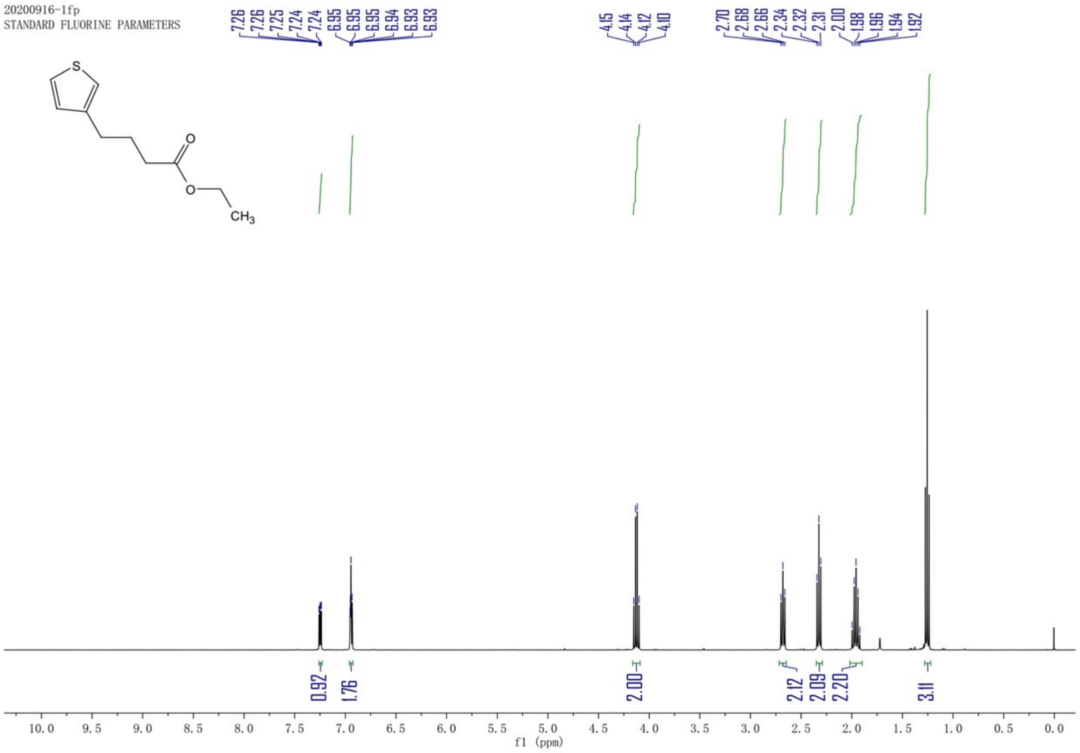

20200825-3f-C13
STANDARD FLUORINE PARAMETERS
N
O
O
CH3
173.24
150.05
147.66
136.81
136.02
123.48
77.48
77.16
76.84
60.53
33.60
32.35
26.32
14.36
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

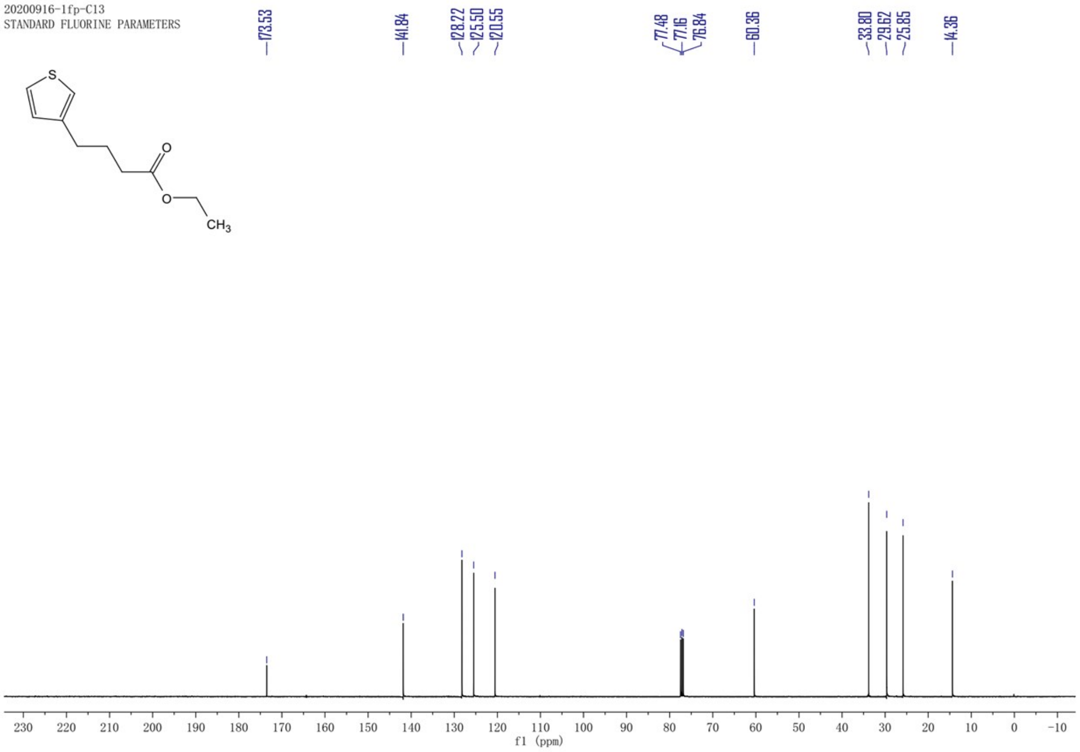
19


20200828-1f
O
O
CH3
N
S
8.70
8.69
6.93
6.92
6.92
4.10
4.08
4.06
4.04
2.85
2.83
2.81
2.30
2.28
2.04
2.02
1.21
1.19
1.18
0.98
0.95
2.00
2.15
2.12
2.22
3.22
12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

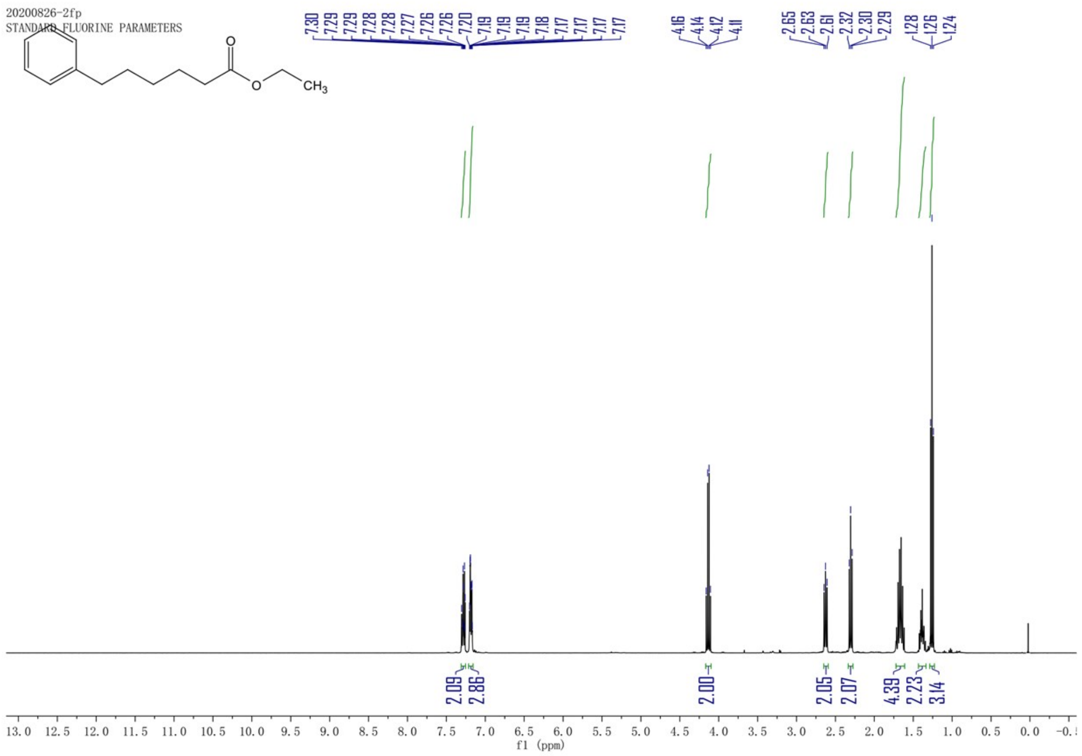

20200828-1f-C13
173.29
157.24
152.44
113.21
77.48
77.16
76.84
60.28
33.62
30.51
24.50
14.26
O
O
CH3
N
S
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

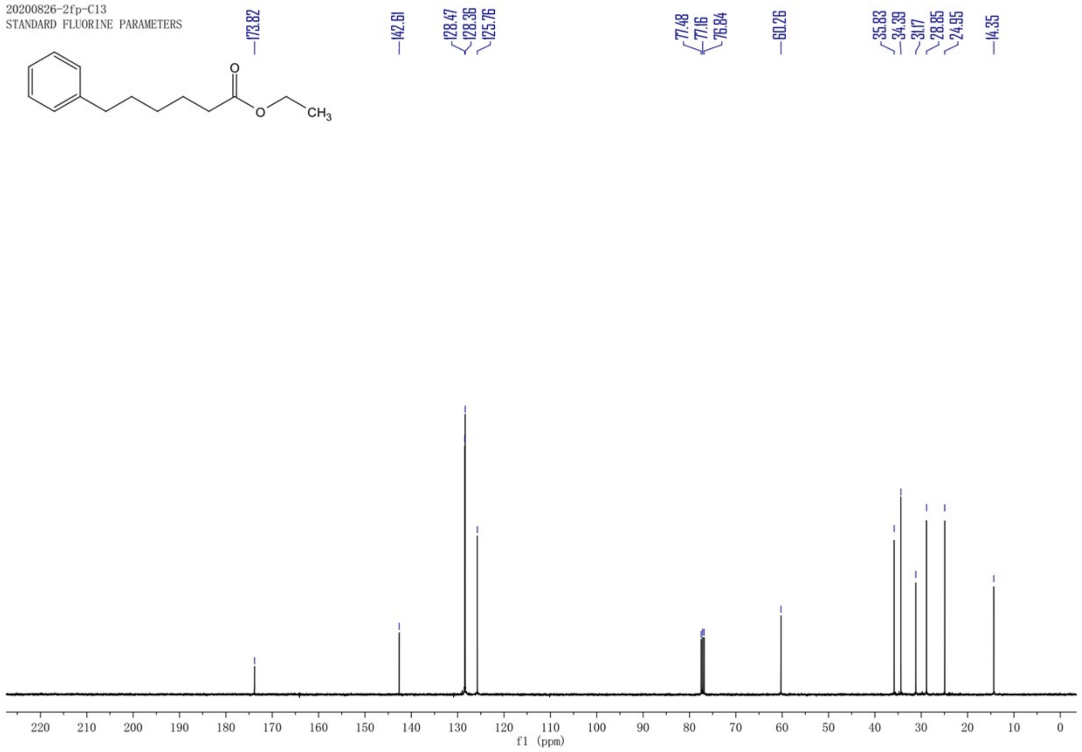
20


20200916-1fp
STANDARD FLUORINE PARAMETERS
7.26
7.26
7.25
7.24
7.24
6.95
6.95
6.95
6.94
6.93
6.93
4.15
4.14
4.12
4.10
2.70
2.68
2.66
2.34
2.32
2.31
2.00
1.98
1.96
1.94
1.92
S
O
O
CH3
0.92
1.76
2.00
2.12
2.09
2.20
3.11
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

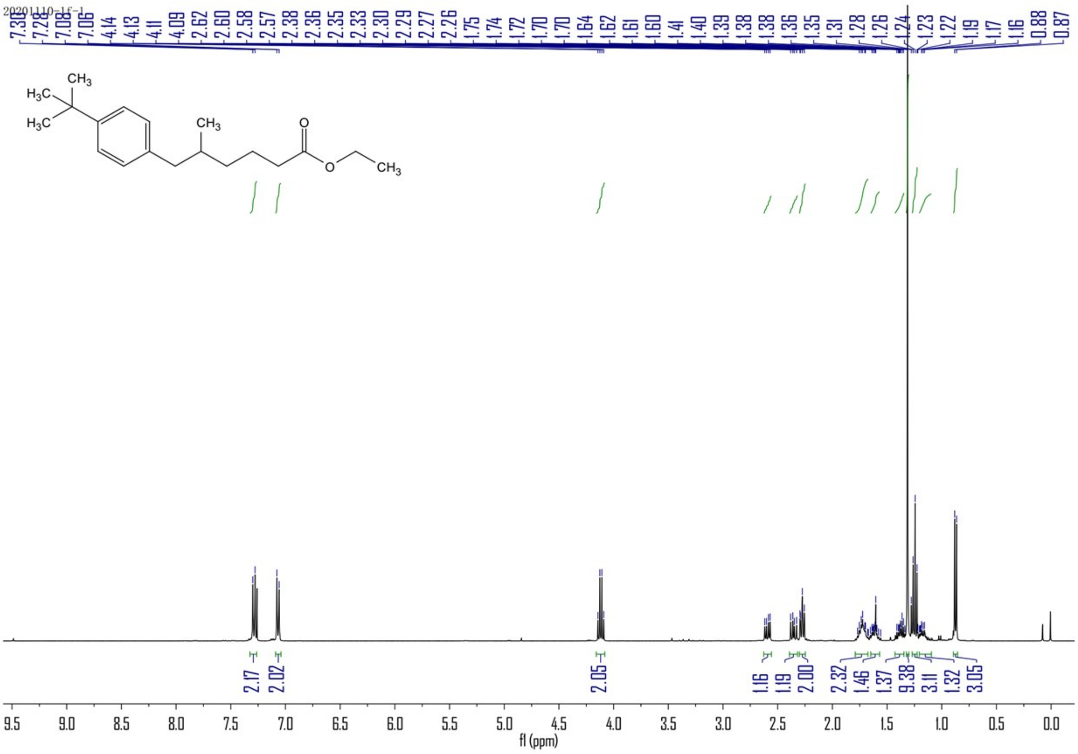

20200916-1fp-C13
STANDARD FLUORINE PARAMETERS
173.53
141.84
128.22
125.50
120.55
77.48
77.16
76.84
60.36
33.80
29.62
25.85
14.36
S
O
O
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

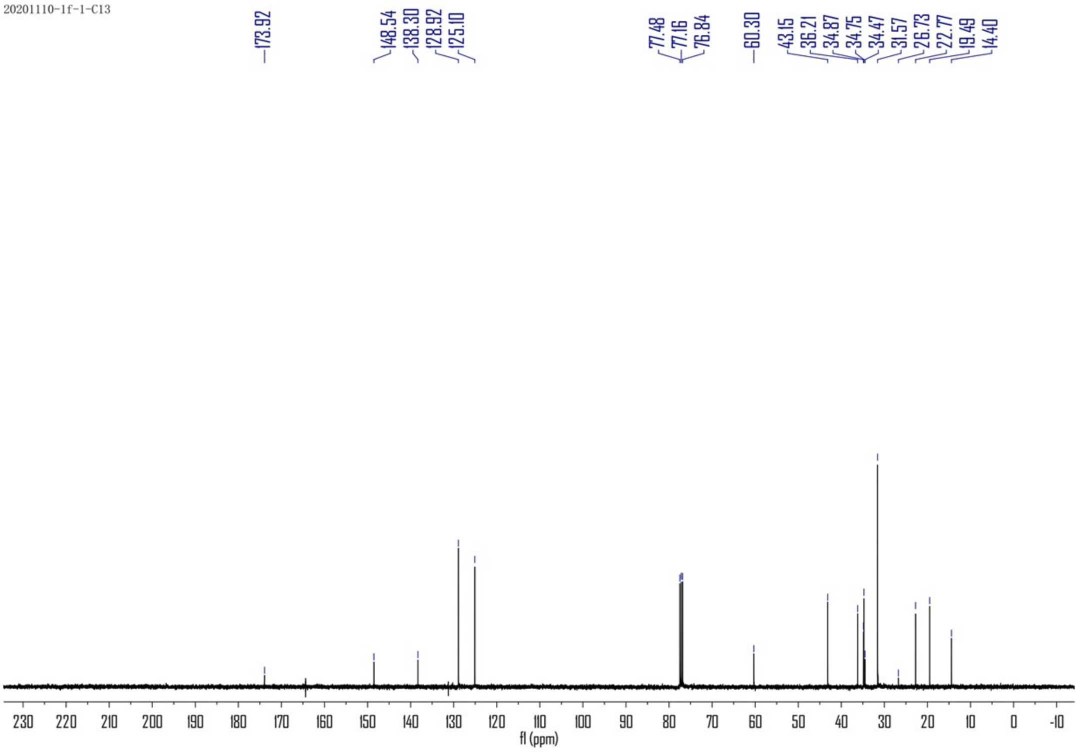
21


20200826-2fp
STANDARD FLUORINE PARAMETERS
O
O
CH3
7.30 7.29 7.29 7.28 7.28 7.27 7.26 7.26 7.20 7.19 7.19 7.19 7.18 7.17 7.17 7.17 7.17
4.16 4.14 4.12 4.11
2.65 2.63 2.61 2.32 2.30 2.29
1.28 1.26 1.24
2.09
2.86
2.00
2.05
2.07
4.39
2.23
3.14
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

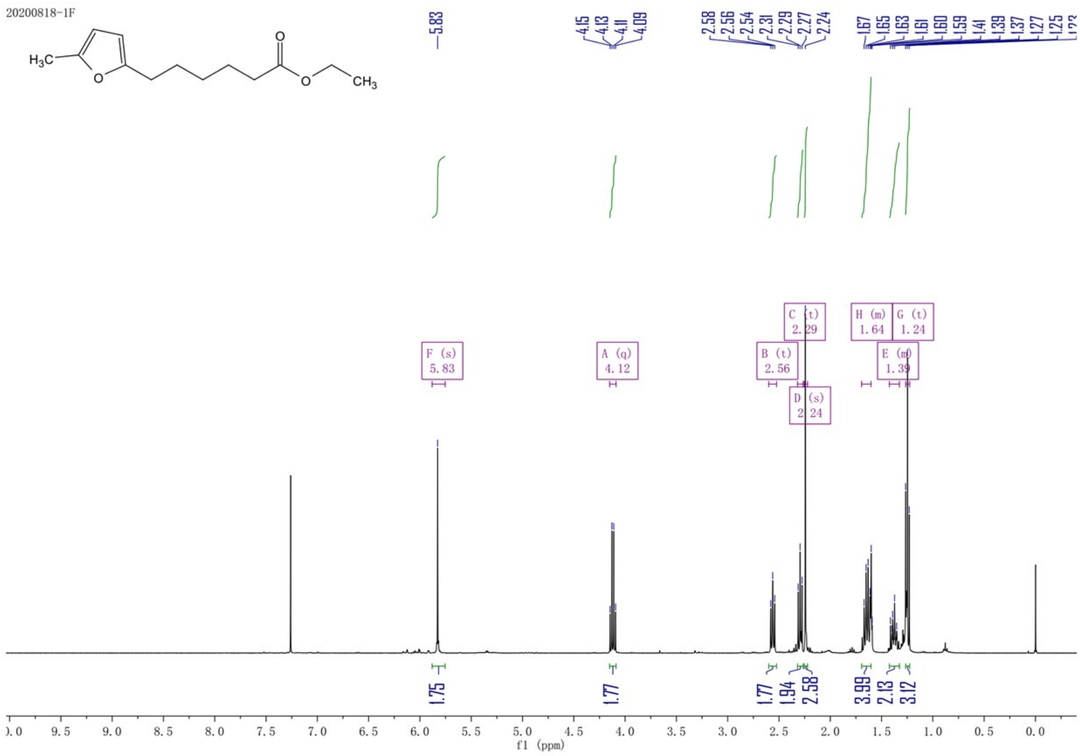

20200826-2fp-C13
STANDARD FLUORINE PARAMETERS
O
O
CH3
173.82
142.61
128.47 128.36 125.76
77.48 77.16 76.84
60.26
35.83 34.39 31.17 28.85 24.95
14.35
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

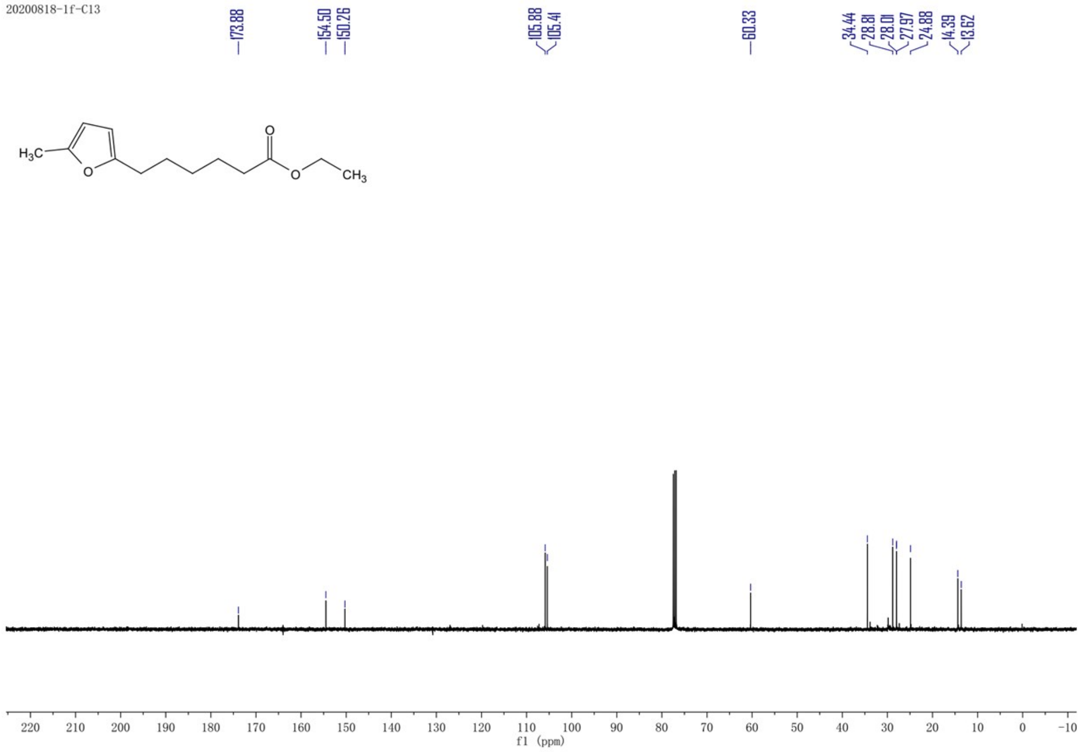
22


20201110-1f-1
7.30
7.28
7.08
7.06
4.14
4.13
4.11
4.09
2.62
2.60
2.58
2.57
2.38
2.36
2.35
2.33
2.30
2.29
2.27
2.26
1.75
1.74
1.72
1.70
1.70
1.64
1.62
1.61
1.60
1.41
1.40
1.39
1.38
1.38
1.36
1.35
1.31
1.28
1.26
1.24
1.23
1.22
1.19
1.17
1.16
0.88
0.87
CH3
H3C
H3C
CH3
O
O
CH3
2.17
2.02
2.05
1.16
1.19
2.00
2.32
1.46
1.37
9.38
3.11
1.32
3.05
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
fl (ppm)

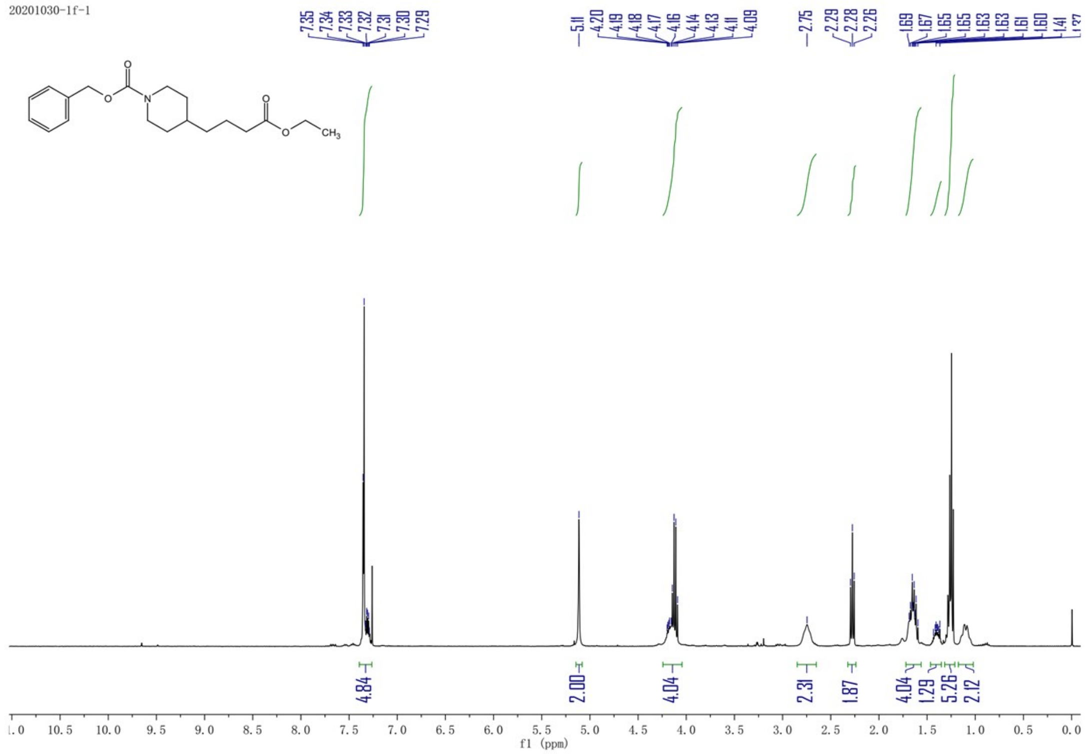

20201110-1f-1-C13
173.92
148.54
138.30
128.92
125.10
77.48
77.16
76.84
60.30
43.15
36.21
34.87
34.75
34.47
31.57
26.73
22.77
19.49
14.40
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
fl (ppm)

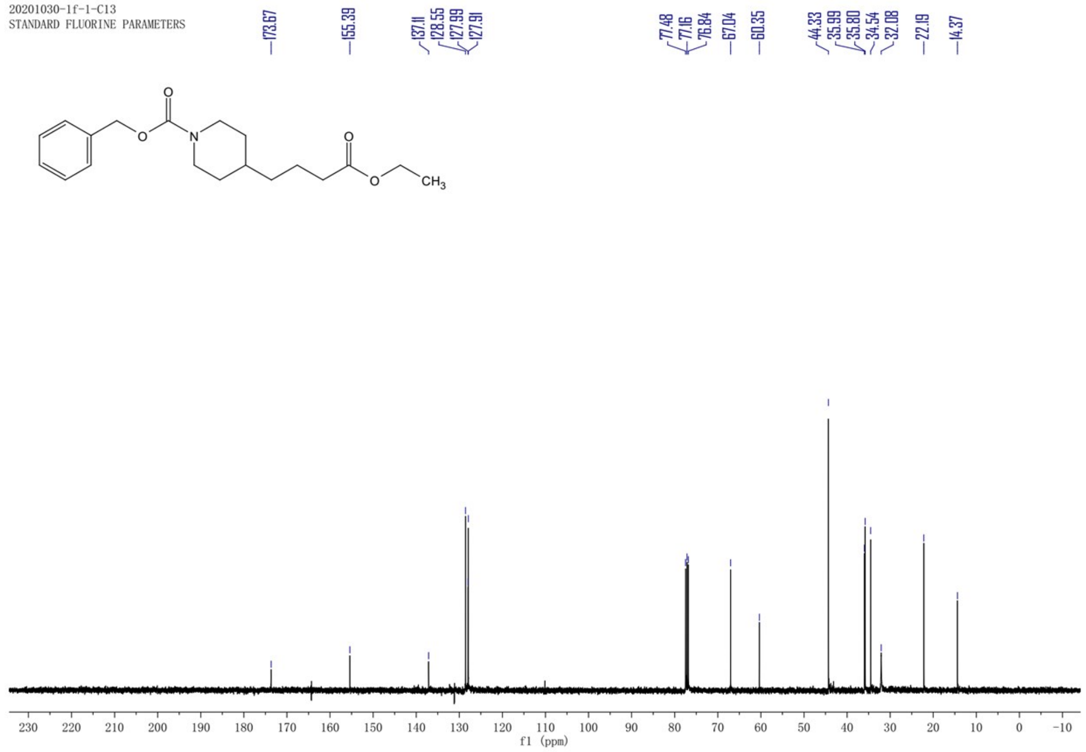
23


20200818-1F
H3C
O
O
O
CH3
5.83
4.15
4.13
4.11
4.09
2.58
2.56
2.54
2.31
2.29
2.27
2.24
1.67
1.65
1.63
1.61
1.60
1.59
1.41
1.39
1.37
1.27
1.25
F (s) 5.83
A (q) 4.12
B (t) 2.56
C (t) 2.29
D (s) 2.24
H (m) 1.64
E (m) 1.39
G (t) 1.24
1.75
1.77
1.77
1.94
2.58
3.99
2.13
3.12
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

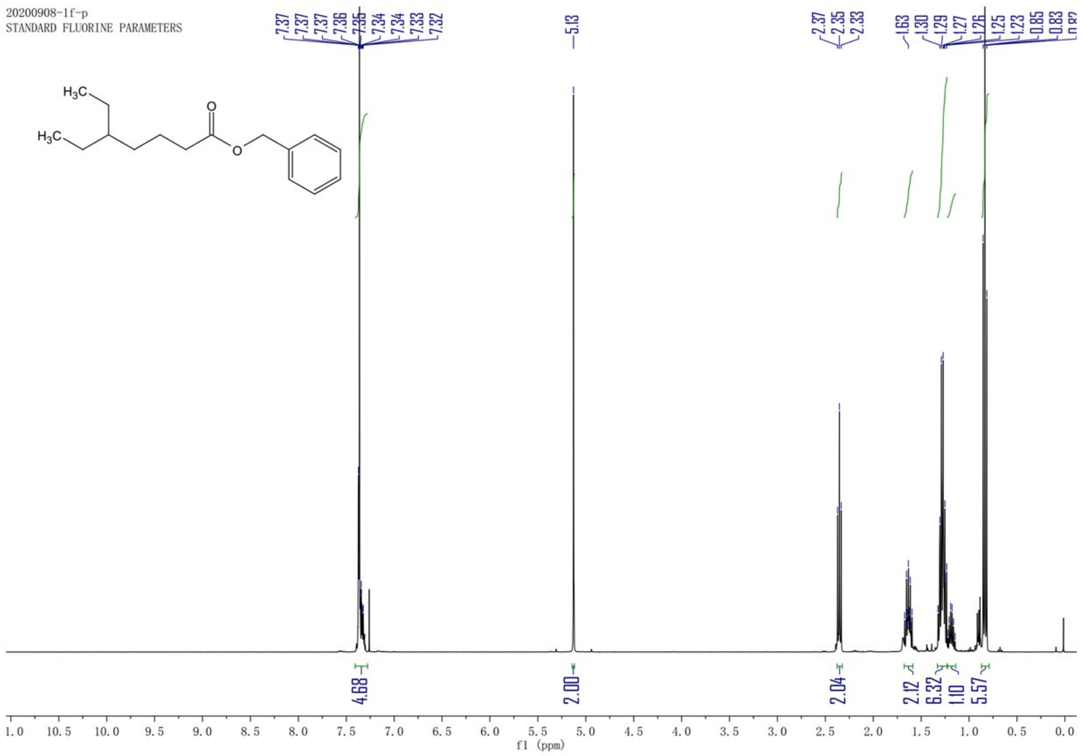

20200818-1f-C13
173.88
154.50
150.26
105.88
105.41
60.33
34.44
28.81
28.01
27.97
24.88
14.39
13.62
H3C
O
O
O
CH3
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

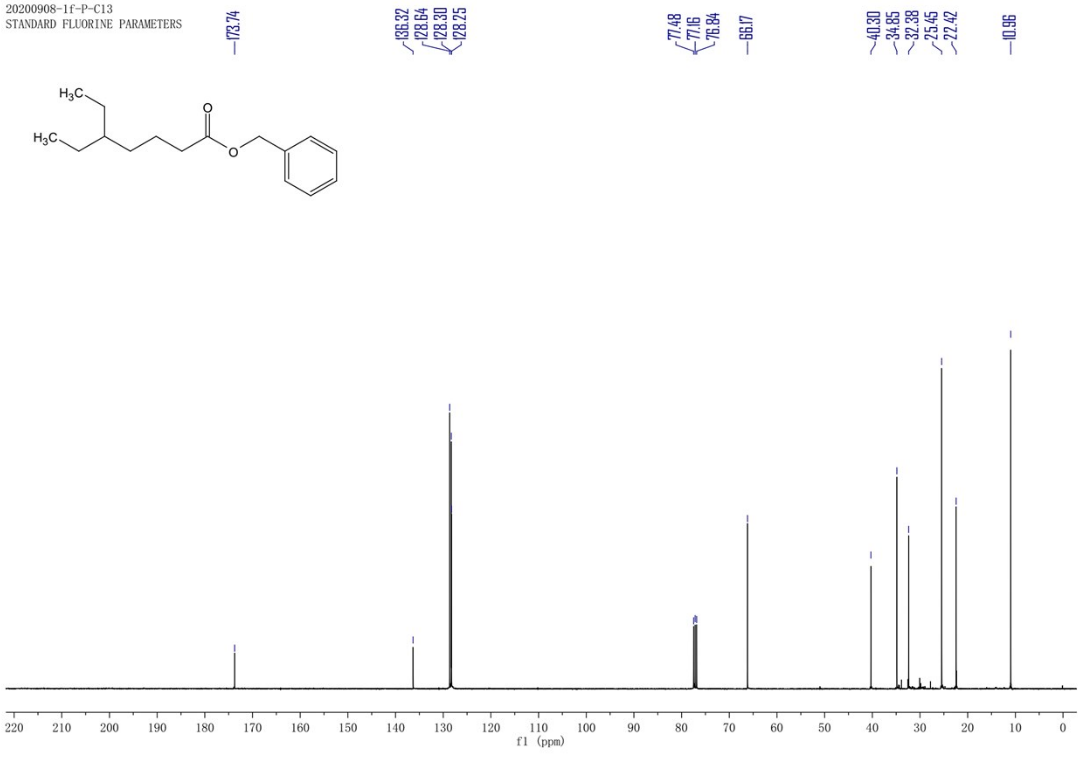
24


20201030-1f-1
7.35
7.34
7.33
7.32
7.31
7.30
7.29
5.11
4.20
4.19
4.18
4.17
4.16
4.14
4.13
4.11
4.09
2.75
2.29
2.28
2.26
1.69
1.67
1.65
1.65
1.63
1.61
1.60
1.41
O
O
N
O
O
CH3
4.84
2.00
4.04
2.31
1.87
4.04
1.29
5.26
2.12
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

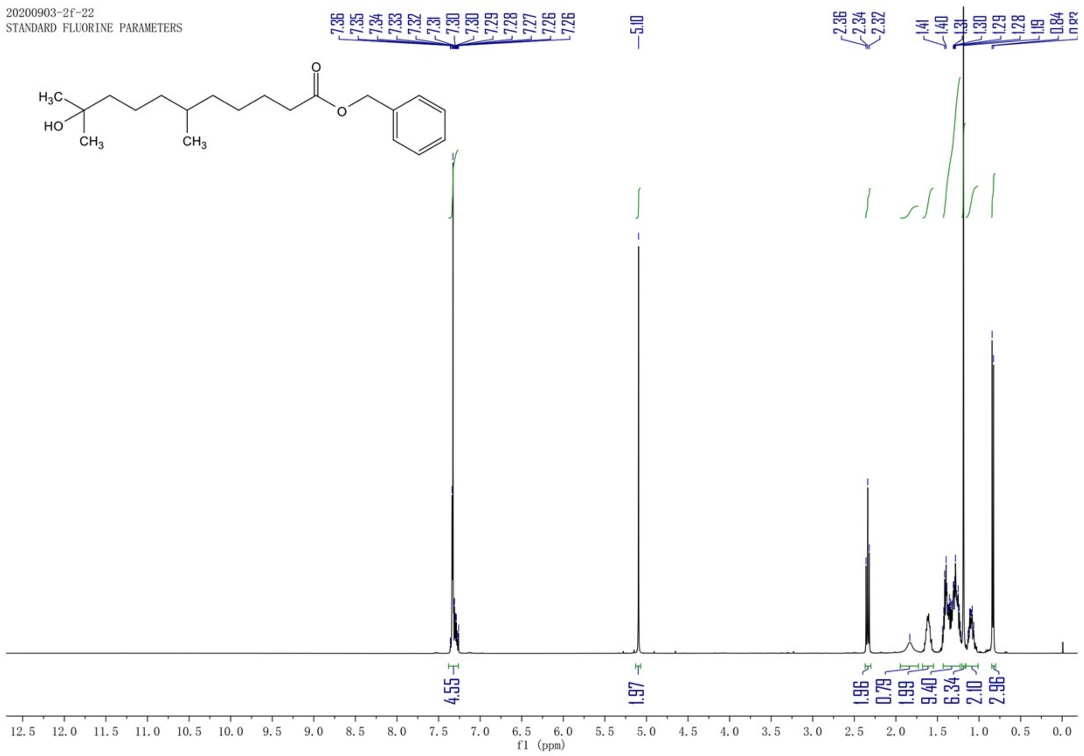

20201030-1f-1-C13
STANDARD FLUORINE PARAMETERS
173.67
155.39
137.11
128.55
127.99
127.91
77.48
77.16
76.84
67.04
60.35
44.33
35.99
35.80
34.54
32.08
22.19
14.37
O
O
N
O
O
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

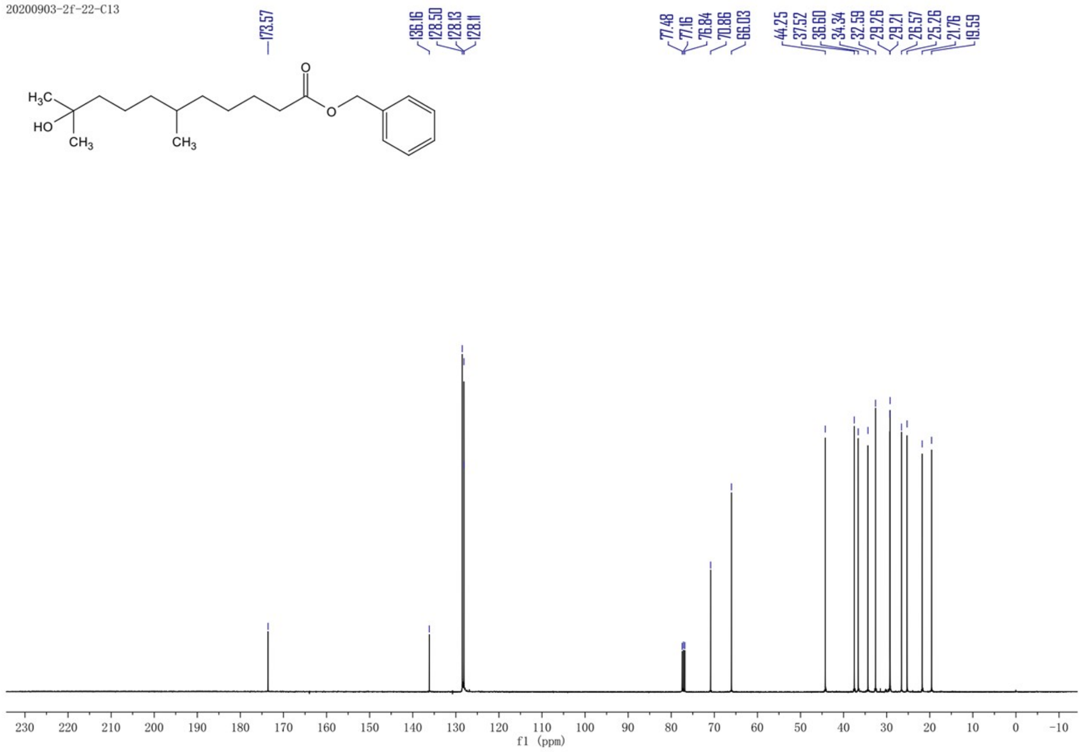
25


20200908-1f-p
STANDARD FLUORINE PARAMETERS
H_3C
H_3C
O
O
7.37
7.37
7.37
7.36
7.35
7.34
7.34
7.33
7.32
5.13
2.37
2.35
2.33
1.63
1.30
1.29
1.27
1.26
1.25
1.23
0.85
0.83
4.68
2.00
2.04
2.12
6.32
1.10
5.57
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

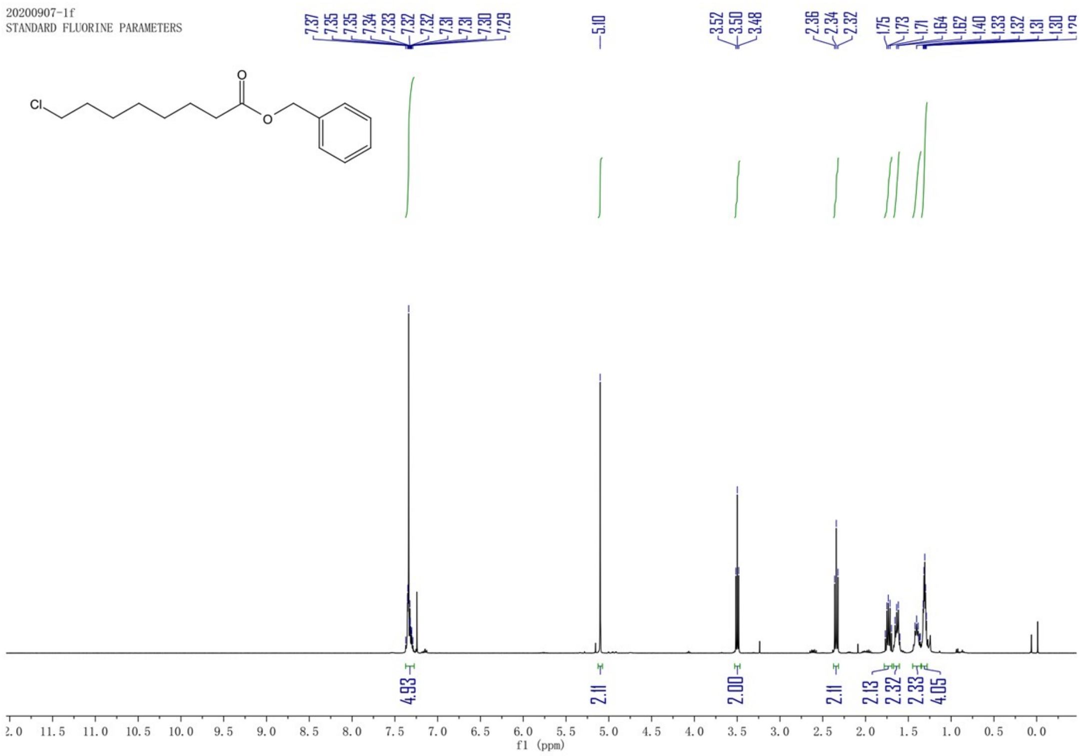

20200908-1f-P-C13
STANDARD FLUORINE PARAMETERS
H_3C
H_3C
O
O
173.74
136.32
128.64
128.30
128.25
77.48
77.16
76.84
66.17
40.30
34.85
32.38
25.45
22.42
10.96
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

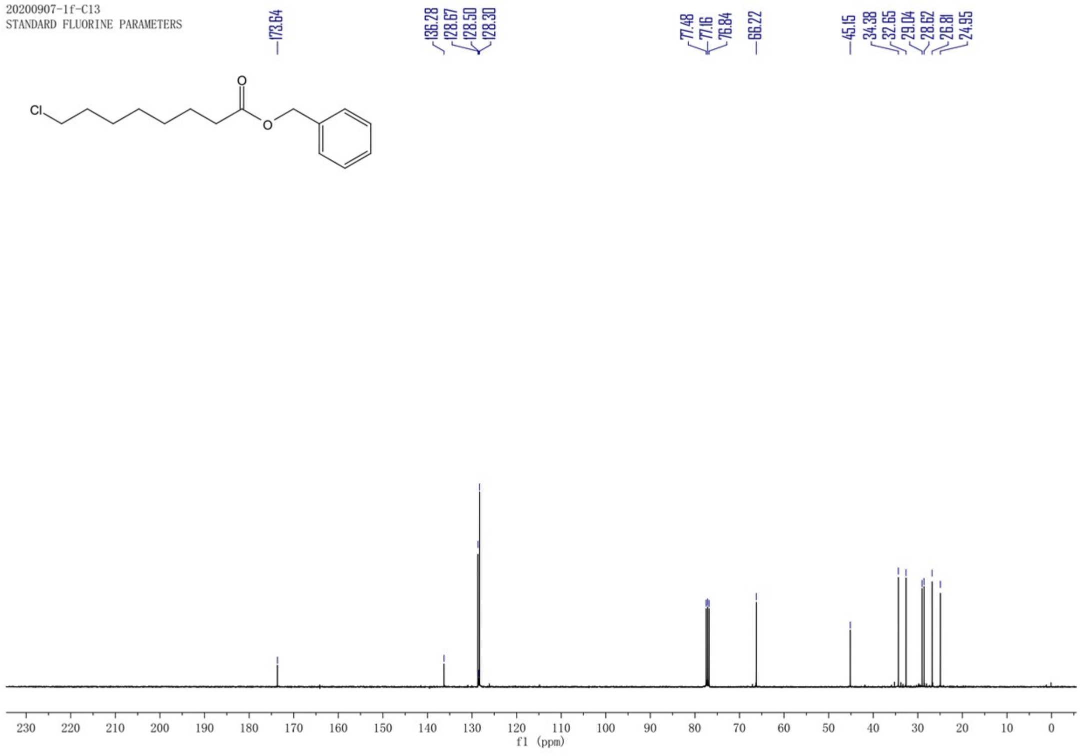
26


20200903-2f-22
STANDARD FLUORINE PARAMETERS
7.36
7.35
7.34
7.33
7.32
7.31
7.30
7.30
7.29
7.28
7.27
7.26
7.26
5.10
2.36
2.34
2.32
1.41
1.40
1.31
1.30
1.29
1.28
1.19
0.84
H$_3$C
HO
CH$_3$
CH$_3$
O
O
4.55
1.97
1.96
0.79
1.99
9.40
6.34
2.10
2.96
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

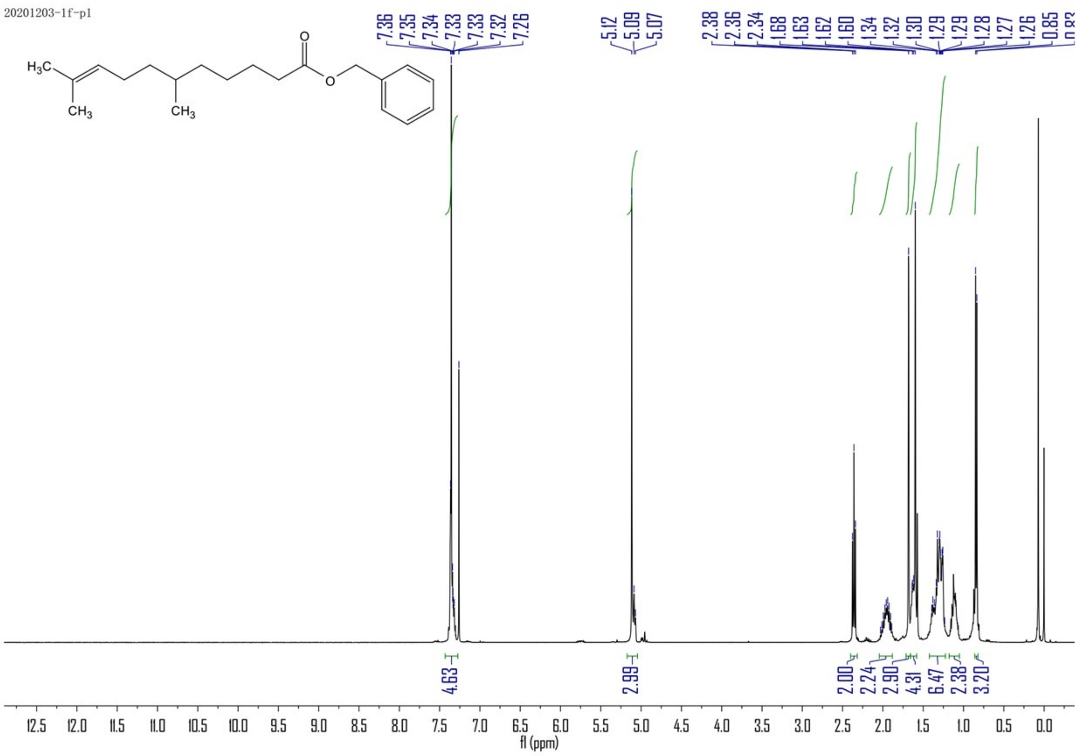

20200903-2f-22-C13
173.57
136.16
128.50
128.13
128.11
77.48
77.16
76.84
70.86
66.03
44.25
37.52
36.60
34.34
32.59
29.26
29.21
26.57
25.26
21.76
19.59
H$_3$C
HO
CH$_3$
CH$_3$
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

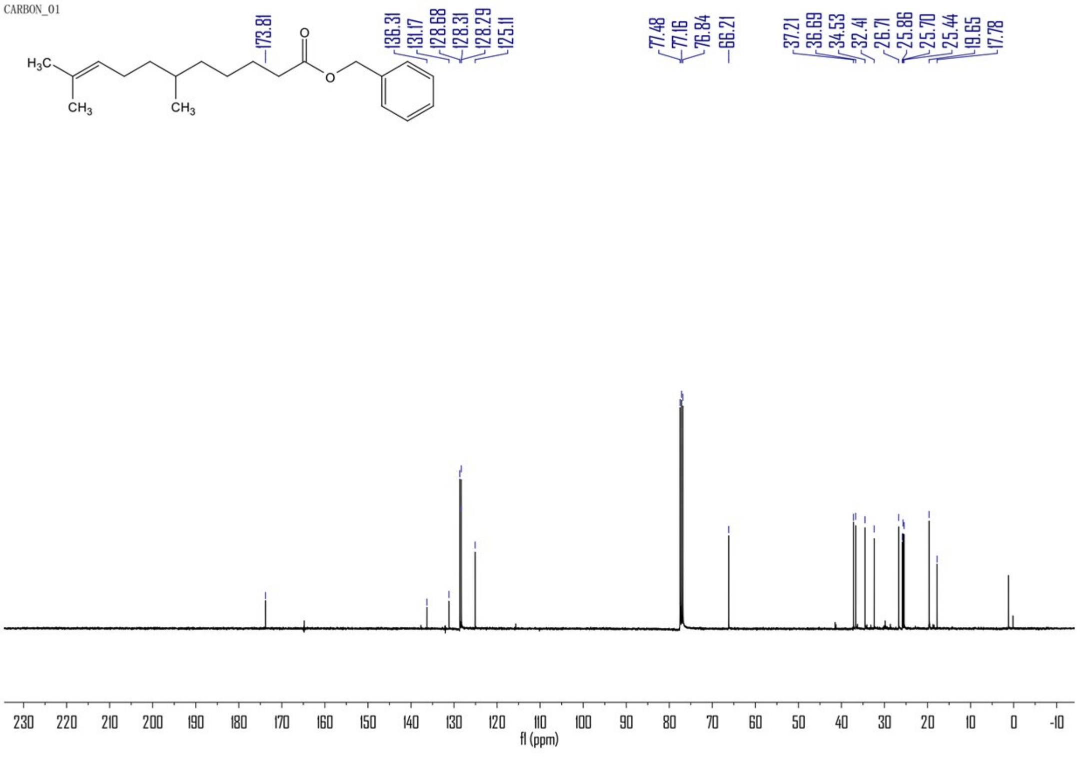
27


20200907-1f
STANDARD FLUORINE PARAMETERS
Cl
O
O
7.37
7.35
7.35
7.34
7.33
7.32
7.32
7.31
7.31
7.30
7.29
5.10
3.52
3.50
3.48
2.36
2.34
2.32
1.75
1.73
1.71
1.64
1.62
1.40
1.33
1.32
1.31
1.30
4.93
2.11
2.00
2.11
2.13
2.32
2.33
4.05
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

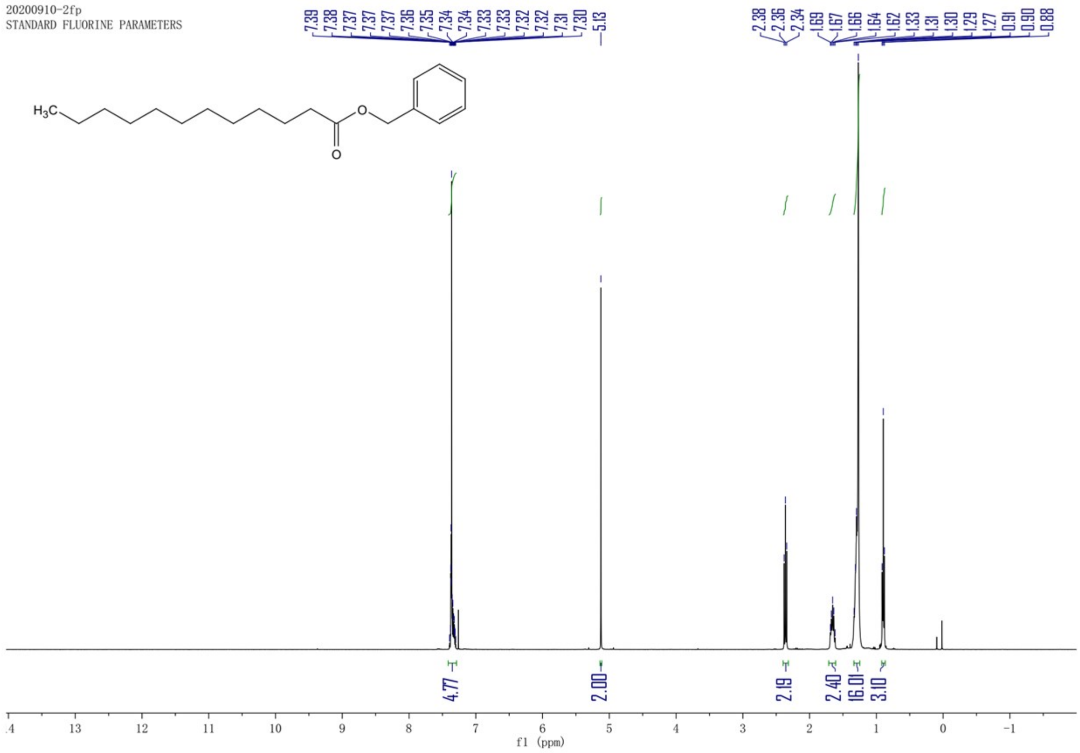

20200907-1f-C13
STANDARD FLUORINE PARAMETERS
Cl
O
O
173.64
136.28
128.67
128.50
128.30
77.48
77.16
76.84
66.22
45.15
34.38
32.65
29.04
28.62
26.81
24.95
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

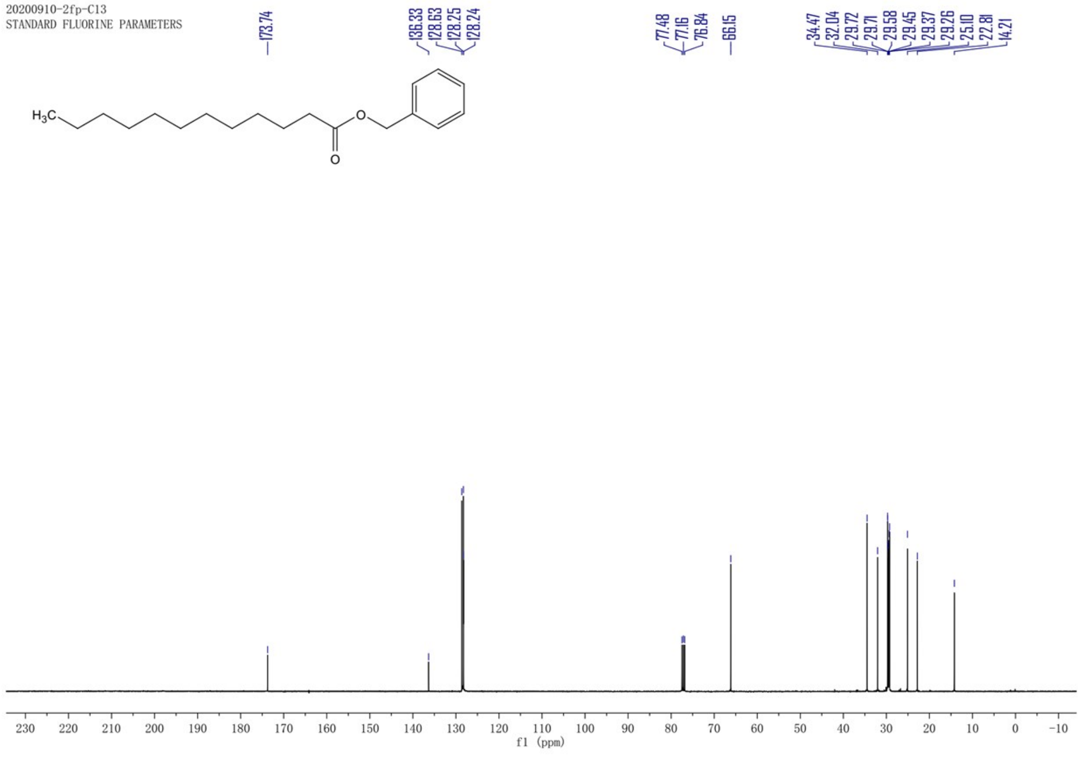
28


20201203-1f-p1
H3C
CH3
CH3
O
O
7.36
7.35
7.34
7.33
7.33
7.32
7.26
5.12
5.09
5.07
2.38
2.36
2.34
1.68
1.63
1.62
1.60
1.34
1.32
1.30
1.29
1.29
1.28
1.27
1.26
0.85
0.83
4.63
2.99
2.00
2.24
2.90
4.31
6.47
2.38
3.20
12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

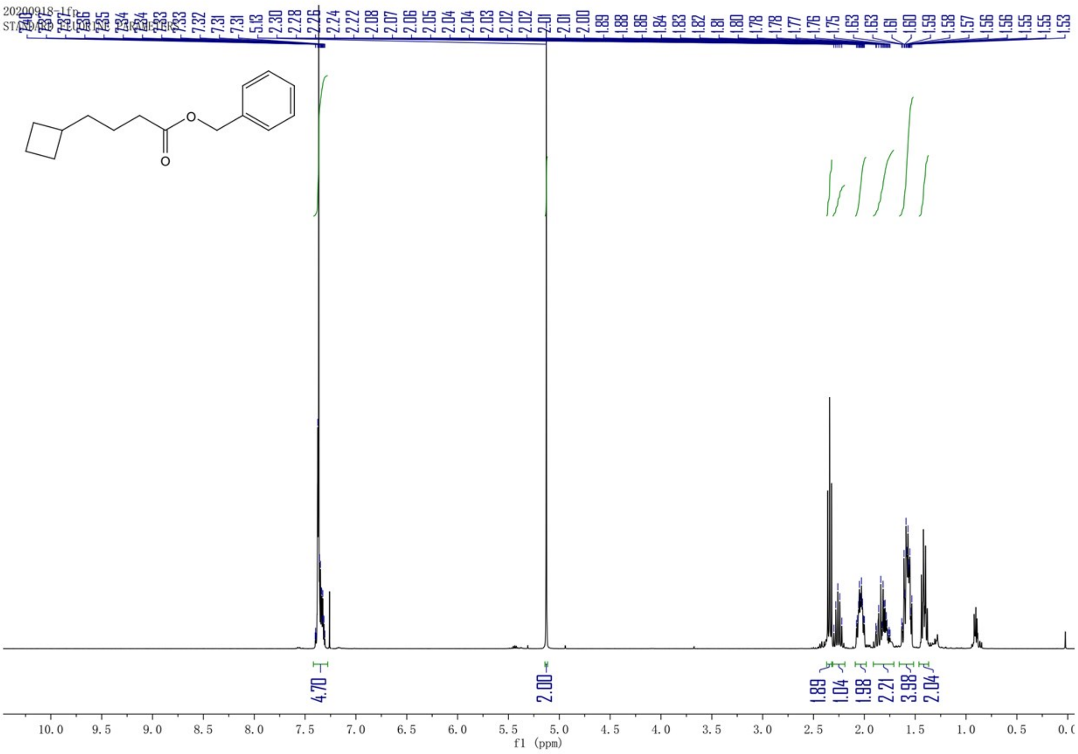

CARBON_01
H3C
CH3
CH3
O
O
173.81
136.31
131.17
128.68
128.31
128.29
125.11
77.48
77.16
76.84
66.21
37.21
36.69
34.53
32.41
26.71
25.86
25.70
25.44
19.65
17.78
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

29

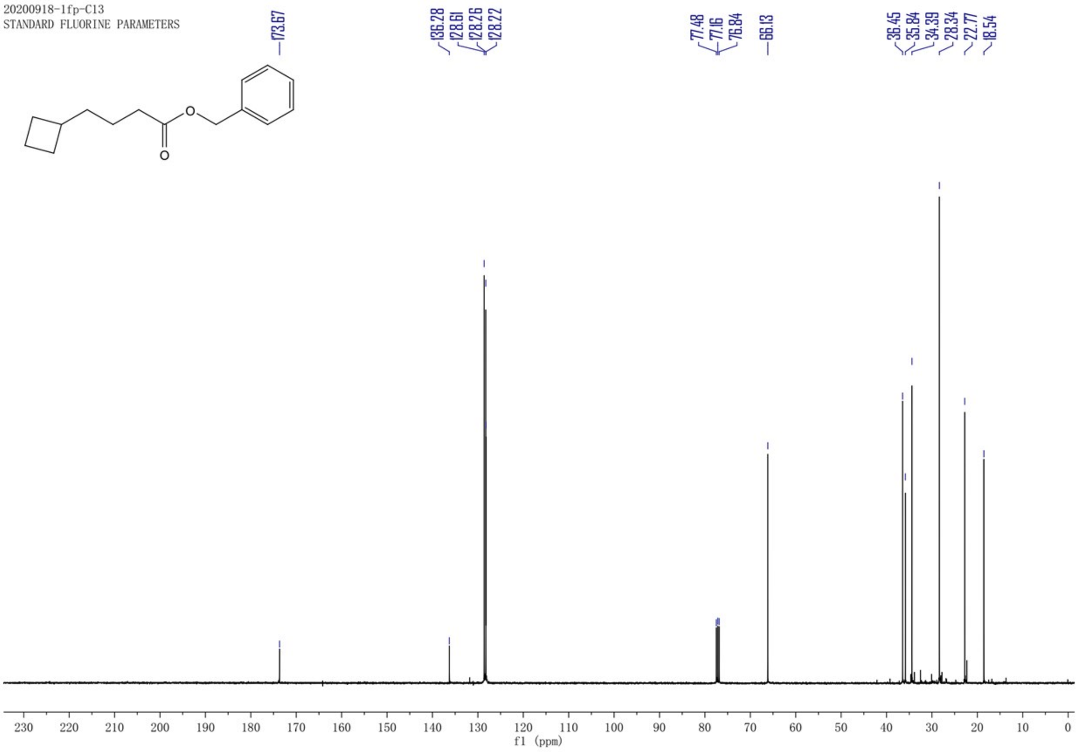

20200910-2fp
STANDARD FLUORINE PARAMETERS
7.39
7.38
7.37
7.37
7.37
7.36
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.31
7.30
5.13
2.38
2.36
2.34
1.69
1.67
1.66
1.64
1.62
1.33
1.31
1.30
1.29
1.27
0.91
0.90
0.88
H3C
O
O
4.77
2.00
2.19
2.40
16.01
3.10
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
f1 (ppm)


20200910-2fp-C13
STANDARD FLUORINE PARAMETERS
173.74
136.33
128.63
128.25
128.24
77.48
77.16
76.84
66.15
34.47
32.04
29.72
29.71
29.58
29.45
29.37
29.26
25.10
22.81
14.21
H3C
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

30

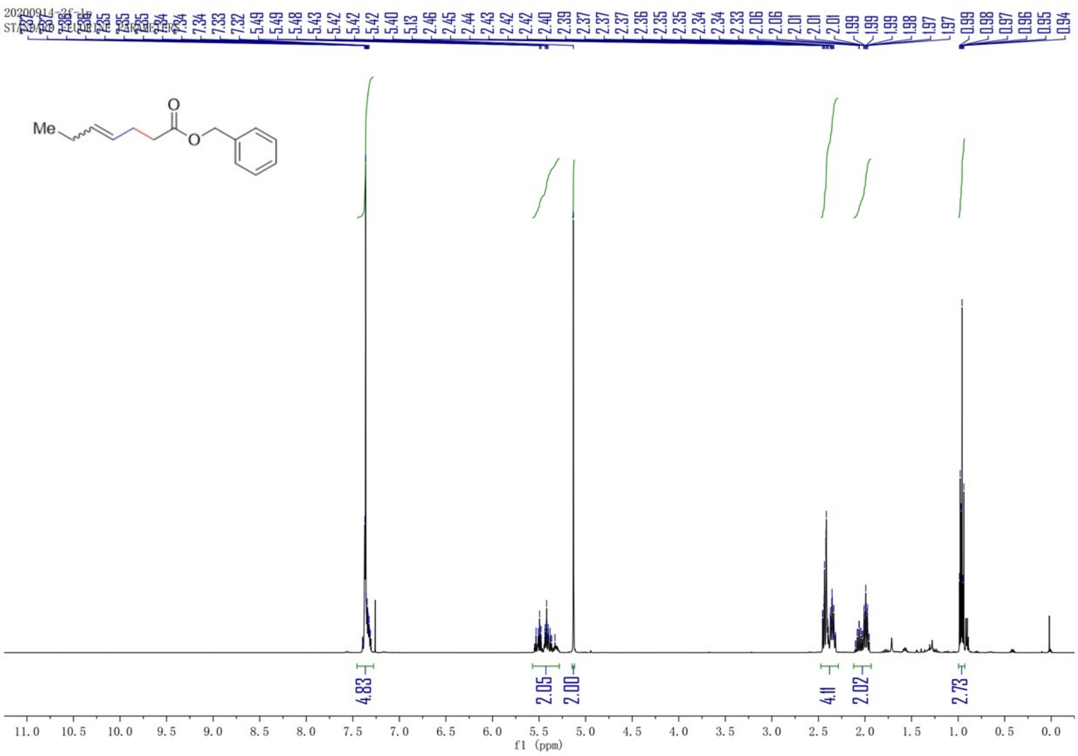


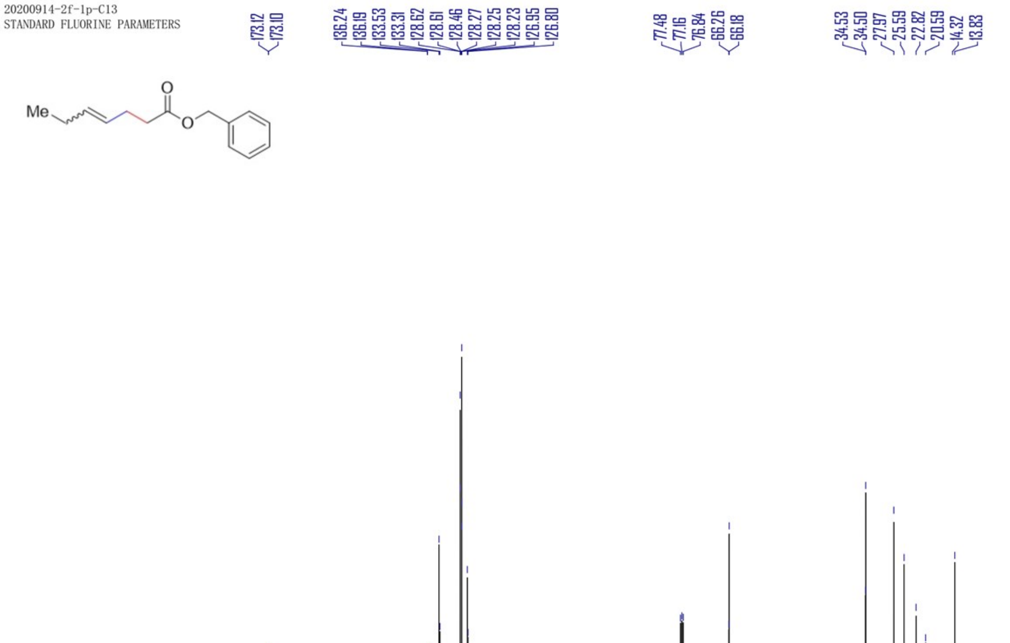
20200918-1fp
STANDARD FLUORINE PARAMETERS
7.40
7.37
7.37
7.36
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.31
7.31
5.13
2.30
2.28
2.26
2.24
2.22
2.08
2.07
2.06
2.05
2.04
2.04
2.03
2.02
2.02
2.01
2.01
2.00
1.89
1.88
1.86
1.84
1.83
1.82
1.81
1.80
1.78
1.78
1.77
1.76
1.75
1.63
1.63
1.61
1.60
1.59
1.58
1.57
1.56
1.56
1.55
1.55
1.53
O
O
4.70
2.00
1.89
1.04
1.98
2.21
3.98
2.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)



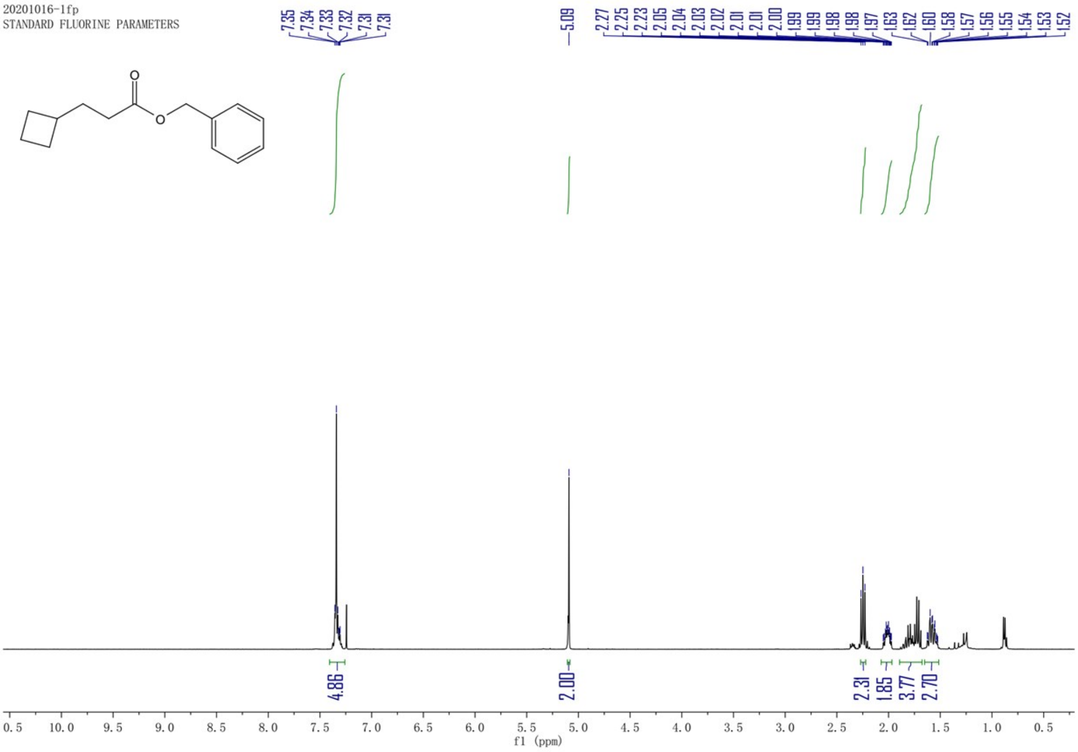
20200918-1fp-C13
STANDARD FLUORINE PARAMETERS
173.67
136.28
128.61
128.26
128.22
77.48
77.16
76.84
66.13
36.45
35.84
34.39
28.34
22.77
18.54
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

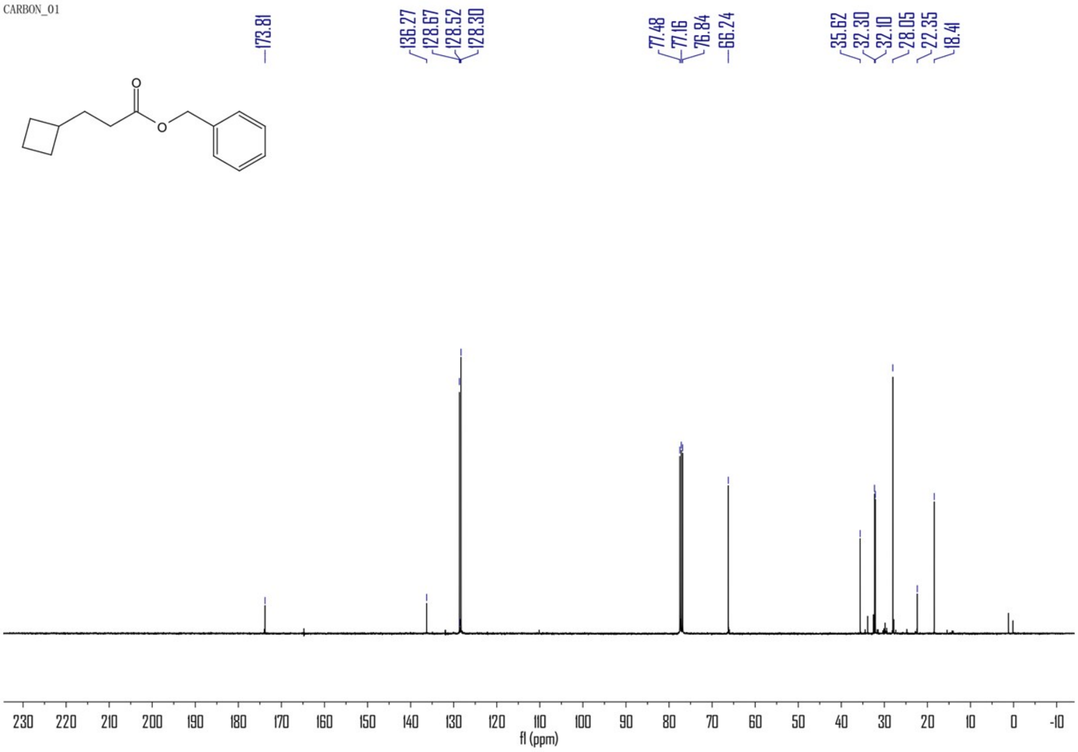
31


20200914-2f-1p
STANDARD FLUORINE PARAMETERS
Me
O
O
7.34
7.34
7.33
7.32
5.49
5.48
5.43
5.42
5.42
5.42
5.40
5.13
2.46
2.45
2.44
2.43
2.42
2.42
2.40
2.39
2.37
2.37
2.37
2.36
2.35
2.35
2.34
2.33
2.06
2.06
2.01
2.01
2.01
1.99
1.99
1.99
1.98
1.97
1.97
0.99
0.98
0.97
0.96
0.95
0.94
4.83
2.05
2.00
4.11
2.02
2.73
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

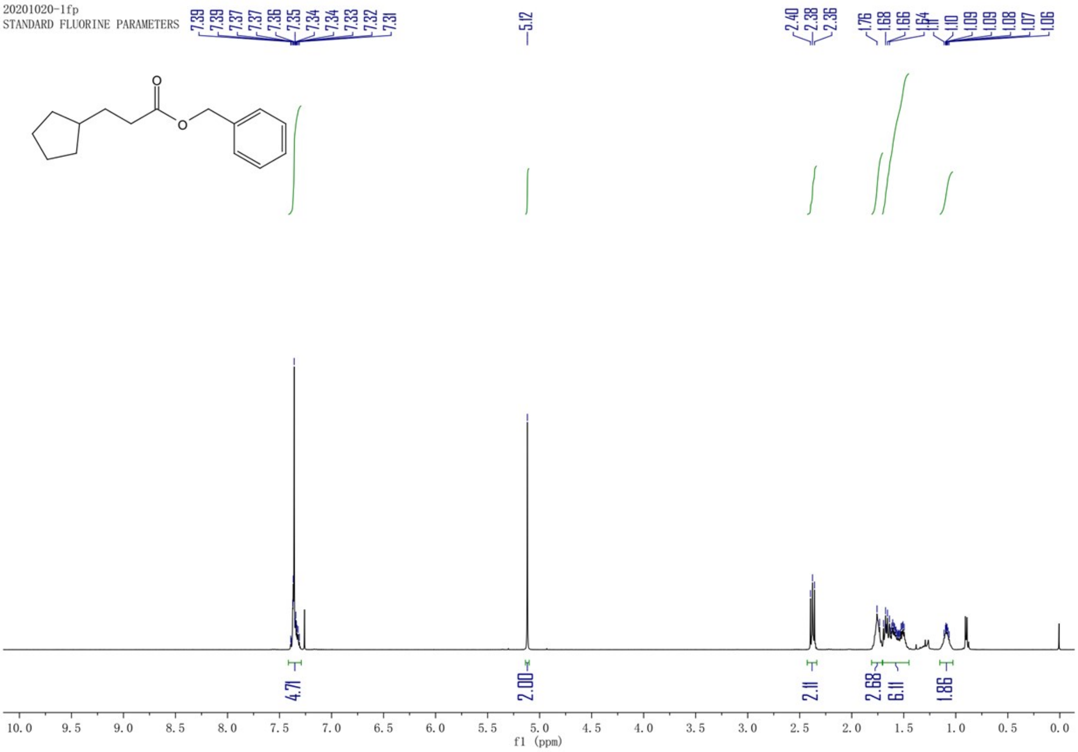

20200914-2f-1p-C13
STANDARD FLUORINE PARAMETERS
173.12
173.10
136.24
136.19
133.53
133.31
128.62
128.61
128.46
128.27
128.25
128.23
126.95
126.80
77.48
77.16
76.84
66.26
66.18
34.53
34.50
27.97
25.59
22.82
20.59
14.32
13.83
Me
O
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

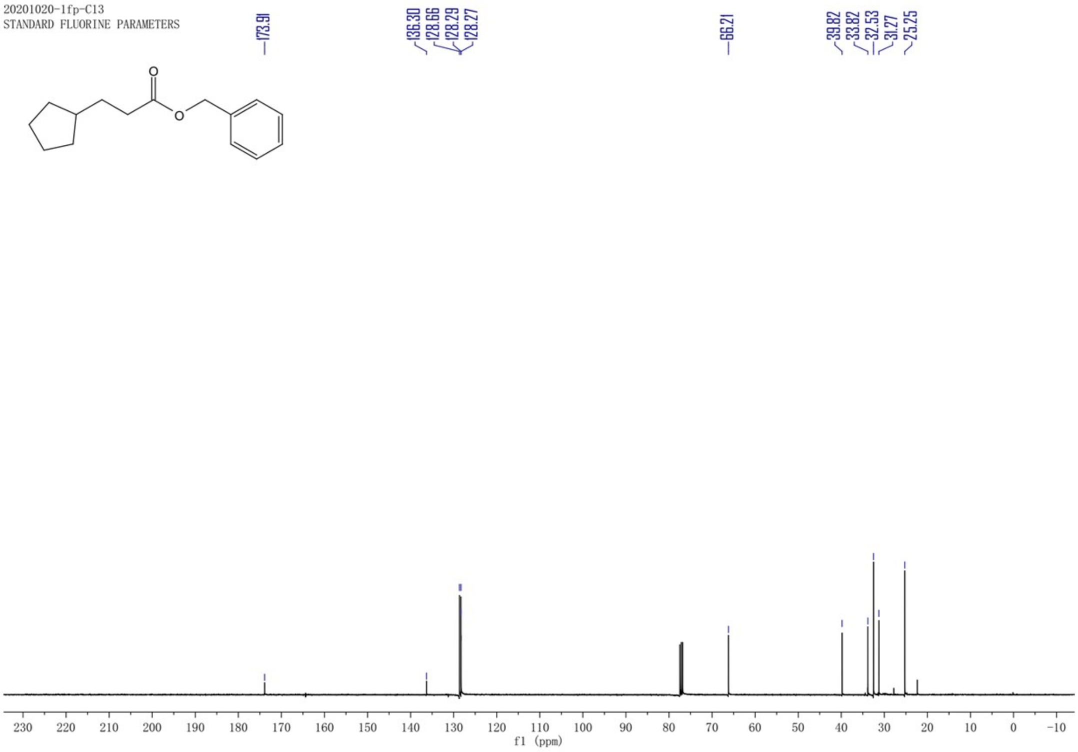
32


20201016-1fp
STANDARD FLUORINE PARAMETERS
7.35
7.34
7.33
7.32
7.31
7.31
5.09
2.27
2.25
2.23
2.05
2.04
2.03
2.02
2.01
2.01
2.00
1.99
1.99
1.98
1.98
1.97
1.63
1.62
1.60
1.58
1.57
1.56
1.55
1.54
1.53
1.52
O
O
4.86
2.00
2.31
1.85
3.77
2.70
0.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

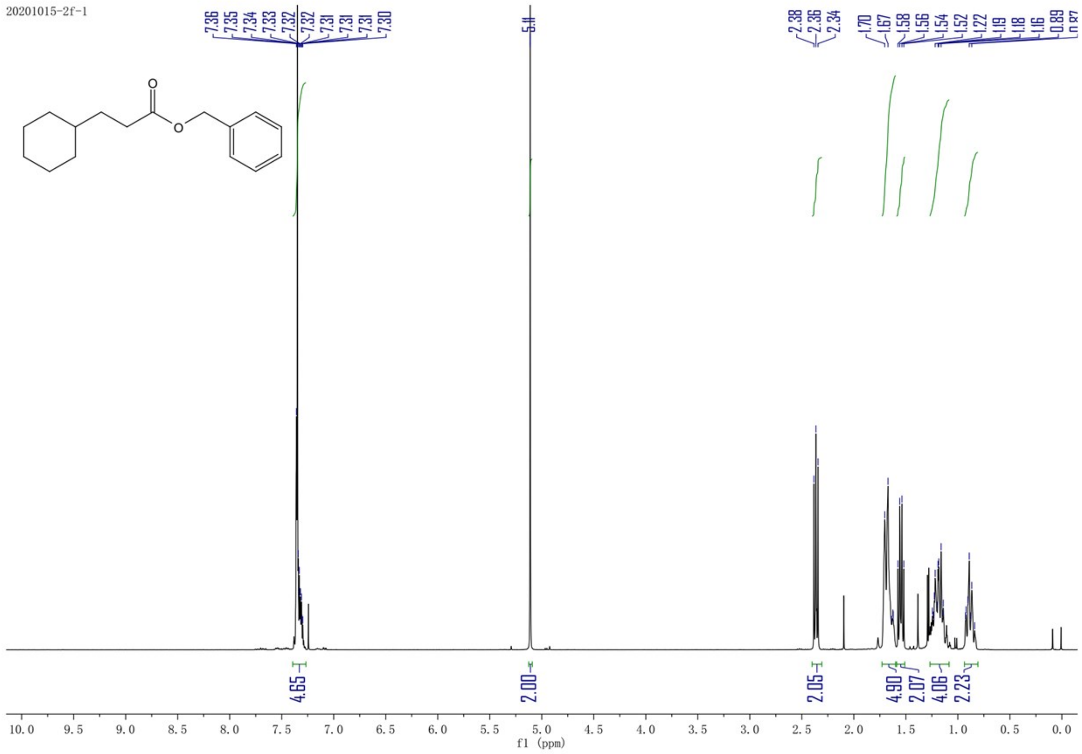

CARBON_01
173.81
136.27
128.67
128.52
128.30
77.48
77.16
76.84
66.24
35.62
32.30
32.10
28.05
22.35
18.41
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
fl (ppm)

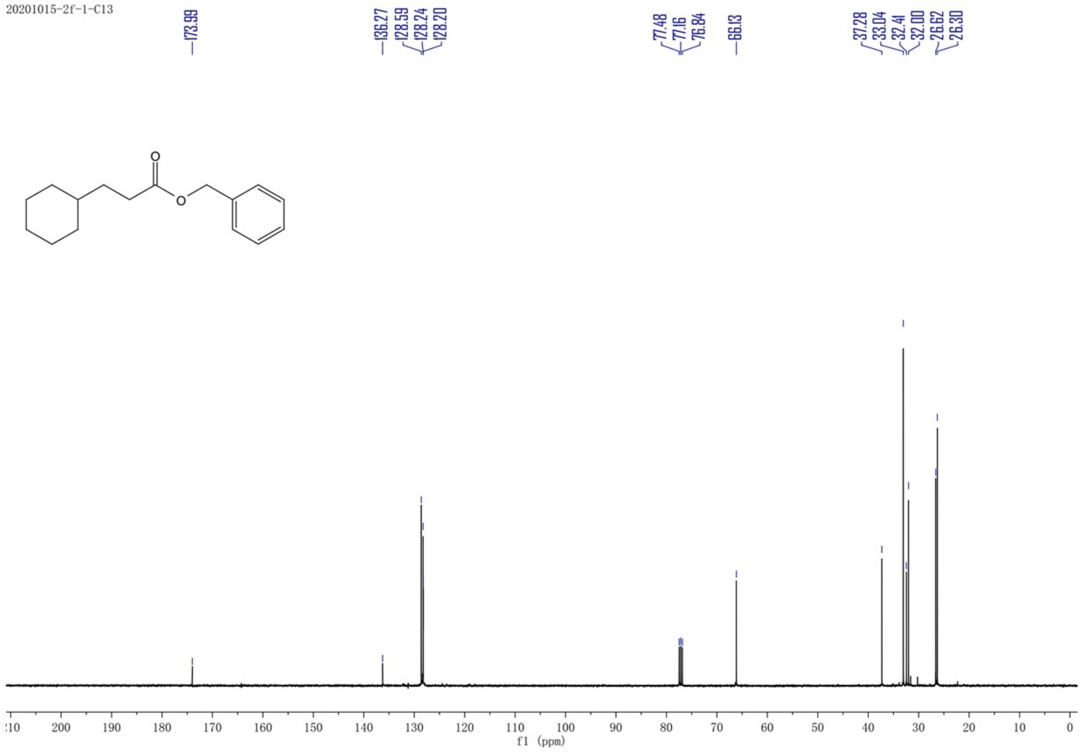
33


20201020-1fp
STANDARD FLUORINE PARAMETERS
7.39
7.39
7.37
7.37
7.36
7.35
7.34
7.34
7.33
7.32
7.31
5.12
2.40
2.38
2.36
1.76
1.68
1.66
1.64
1.11
1.10
1.09
1.09
1.08
1.07
1.06
O
O
4.71
2.00
2.11
2.68
6.11
1.86
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

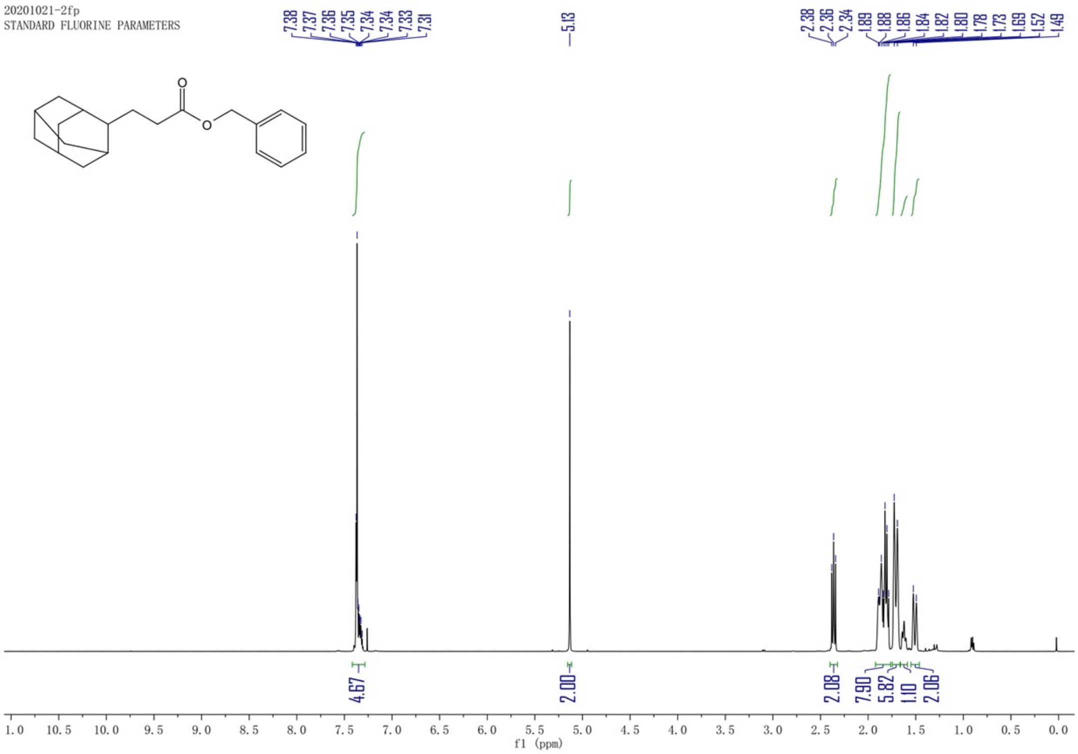

20201020-1fp-C13
STANDARD FLUORINE PARAMETERS
173.91
136.30
128.66
128.29
128.27
66.21
39.82
33.82
32.53
31.27
25.25
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

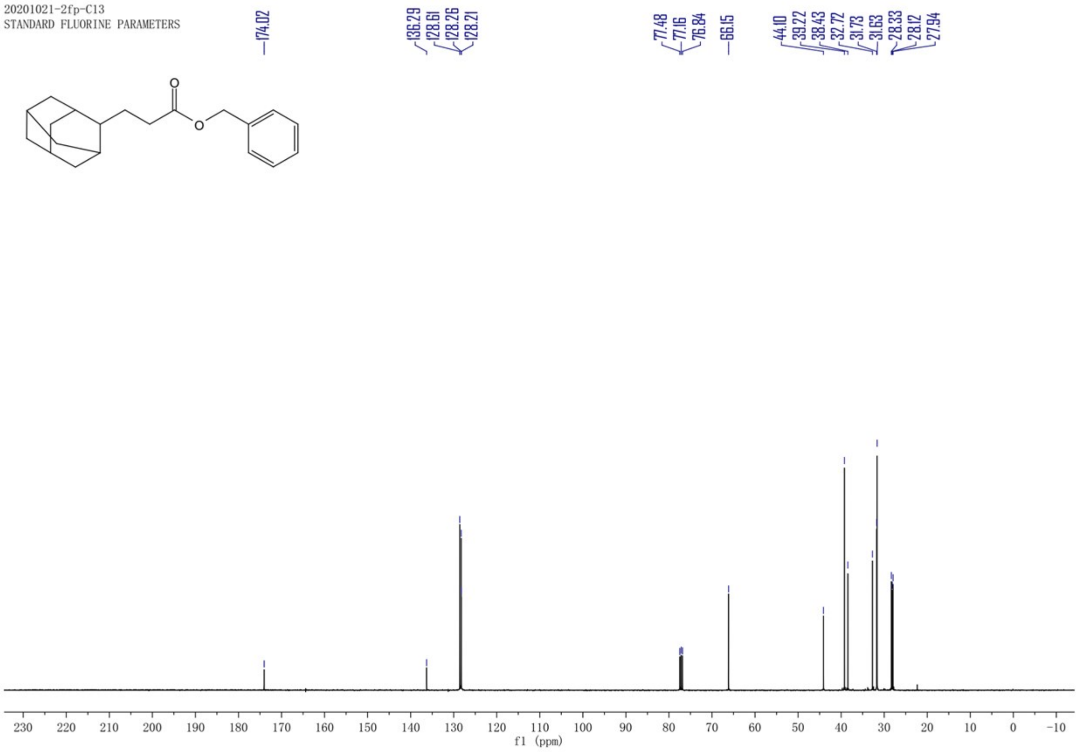
34


20201015-2f-1
O
O
7.36
7.35
7.34
7.33
7.32
7.32
7.31
7.31
7.31
7.30
5.11
2.38
2.36
2.34
1.70
1.67
1.58
1.56
1.54
1.52
1.22
1.19
1.18
1.16
0.89
0.87
4.65
2.00
2.05
4.90
2.07
4.06
2.23
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

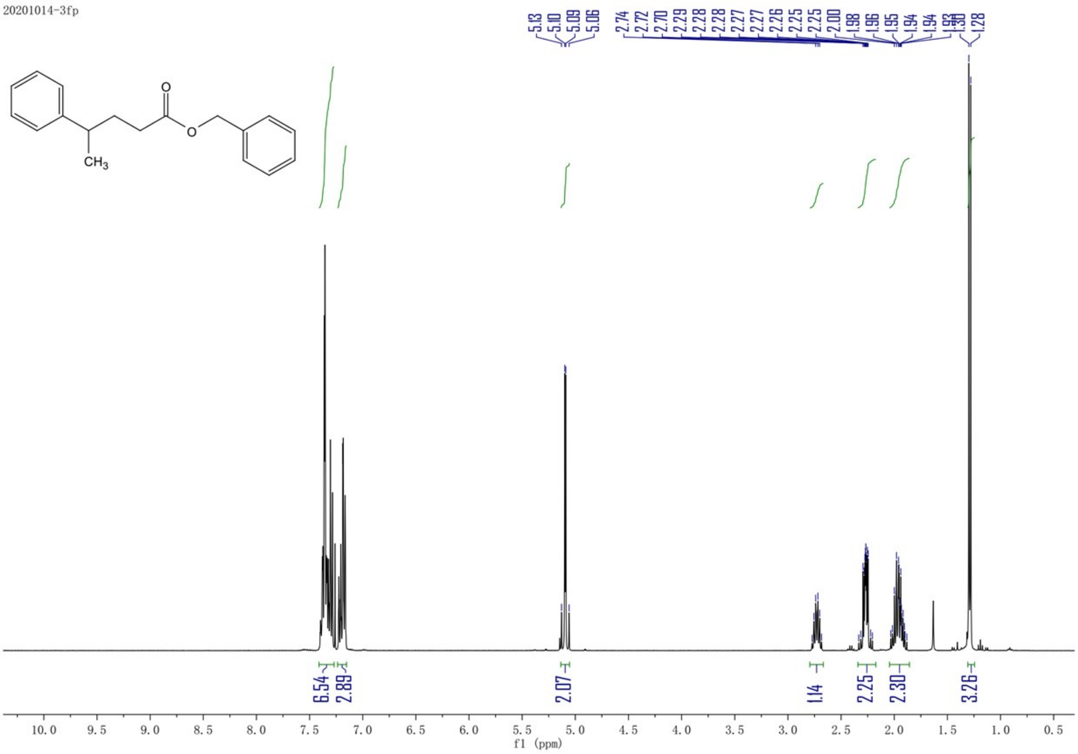

20201015-2f-1-C13
173.99
136.27
128.59
128.24
128.20
77.48
77.16
76.84
66.13
37.28
33.04
32.41
32.00
26.62
26.30
O
O
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

35

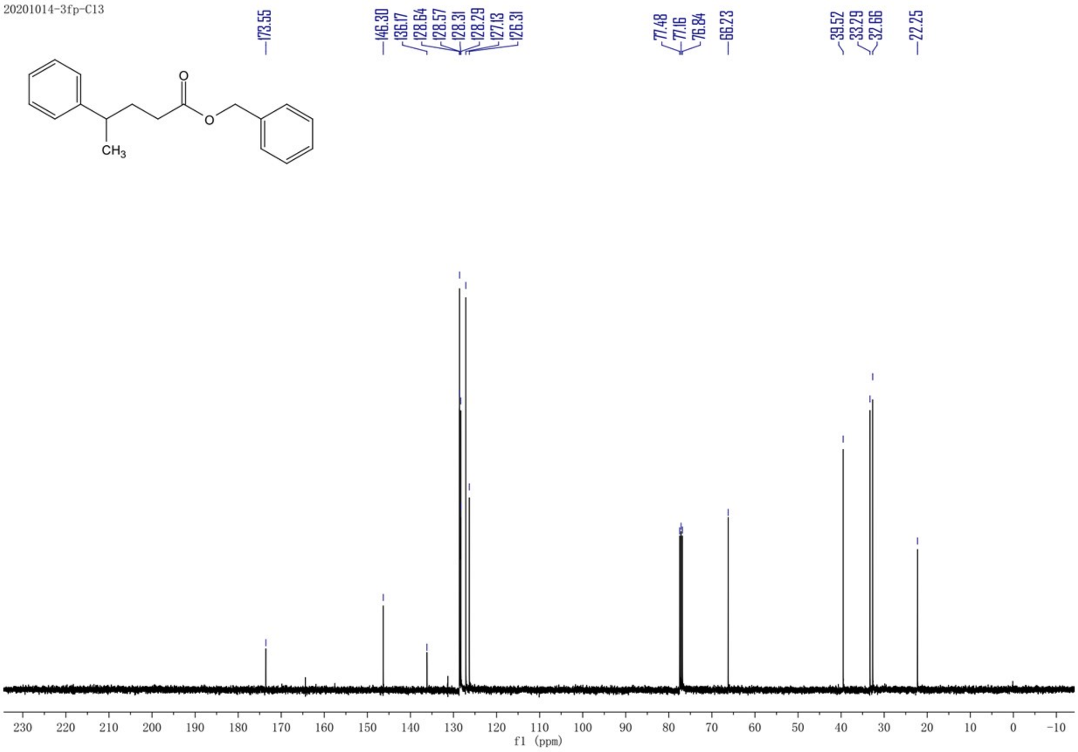

20201021-2fp
STANDARD FLUORINE PARAMETERS
7.38
7.37
7.36
7.35
7.34
7.34
7.33
7.31
5.13
2.38
2.36
2.34
1.89
1.88
1.86
1.84
1.82
1.80
1.78
1.73
1.69
1.52
1.49
O
O
4.67
2.00
2.08
7.90
5.82
1.10
2.06
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


20201021-2fp-C13
STANDARD FLUORINE PARAMETERS
174.02
136.29
128.61
128.26
128.21
77.48
77.16
76.84
66.15
44.10
39.22
38.43
32.72
31.73
31.63
28.33
28.12
27.94
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

36

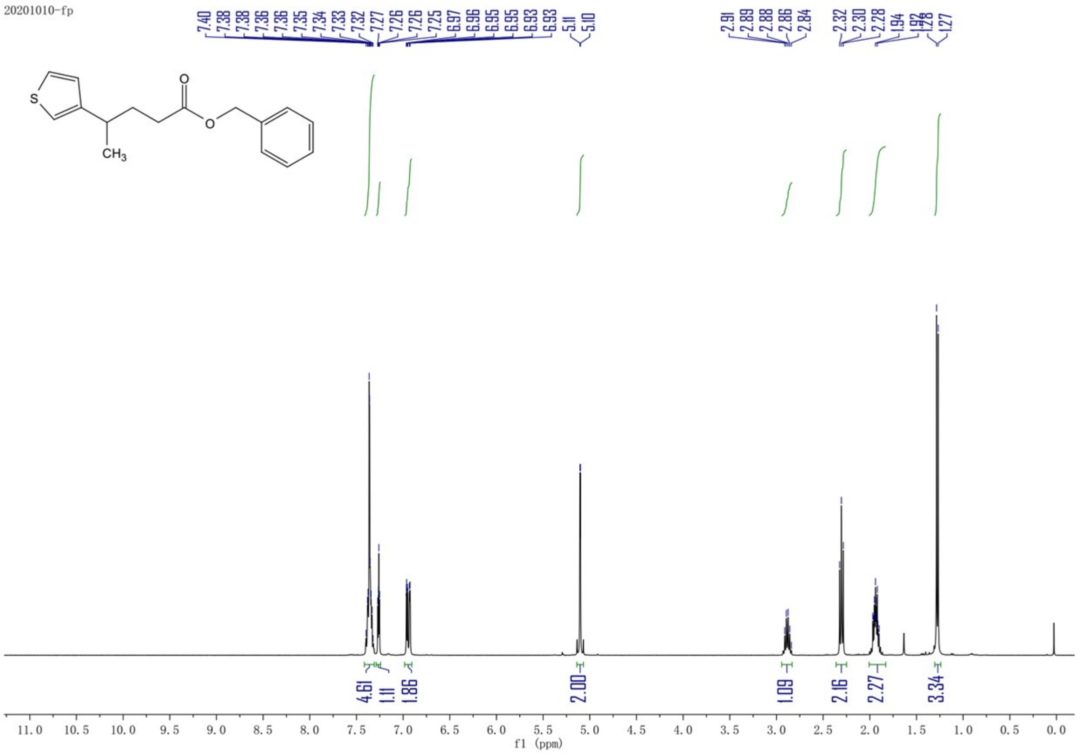


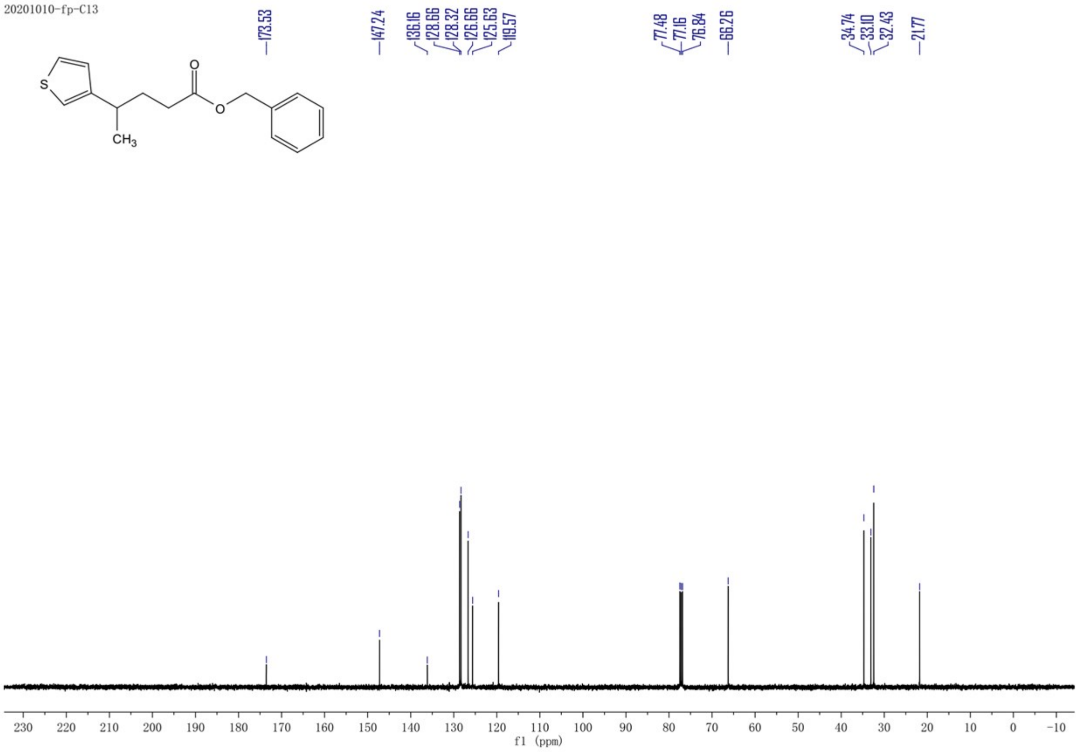
20201014-3fp
O
CH3
5.13
5.10
5.09
5.06
2.74
2.72
2.70
2.29
2.28
2.28
2.27
2.27
2.26
2.25
2.25
2.00
1.98
1.96
1.95
1.94
1.94
1.93
1.30
1.28
6.54
2.89
2.07
1.14
2.25
2.30
3.26
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)



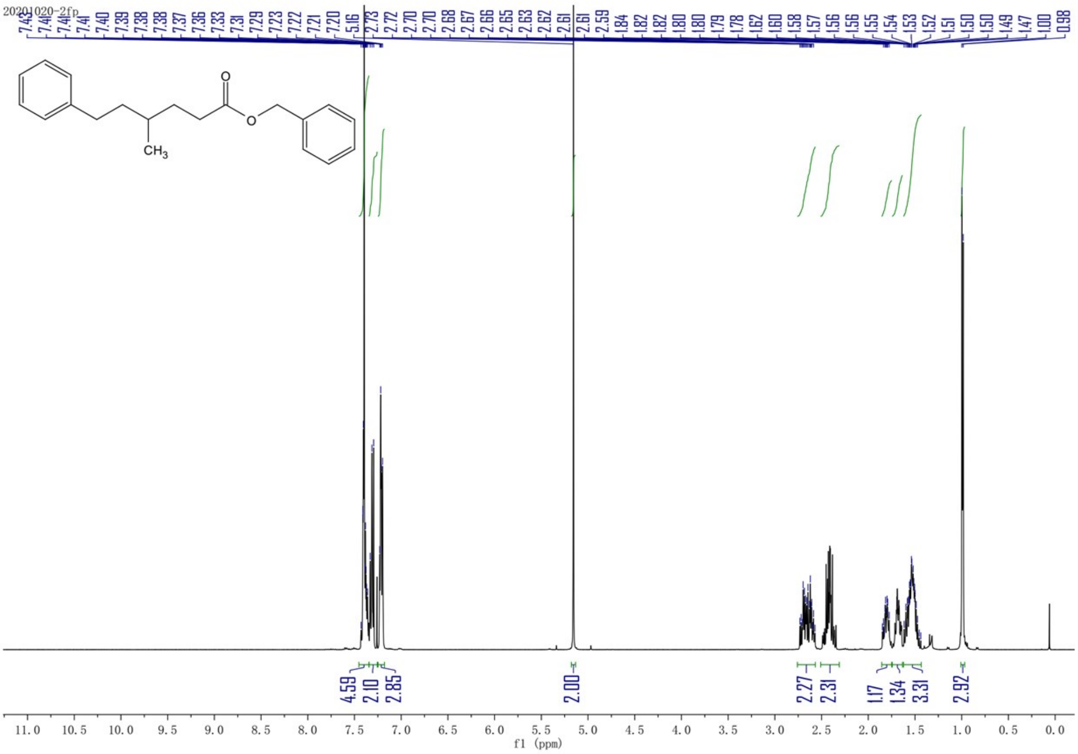
20201014-3fp-C13
O
CH3
173.55
146.30
136.17
128.64
128.57
128.31
128.29
127.13
126.31
77.48
77.16
76.84
66.23
39.52
33.29
32.66
22.25
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

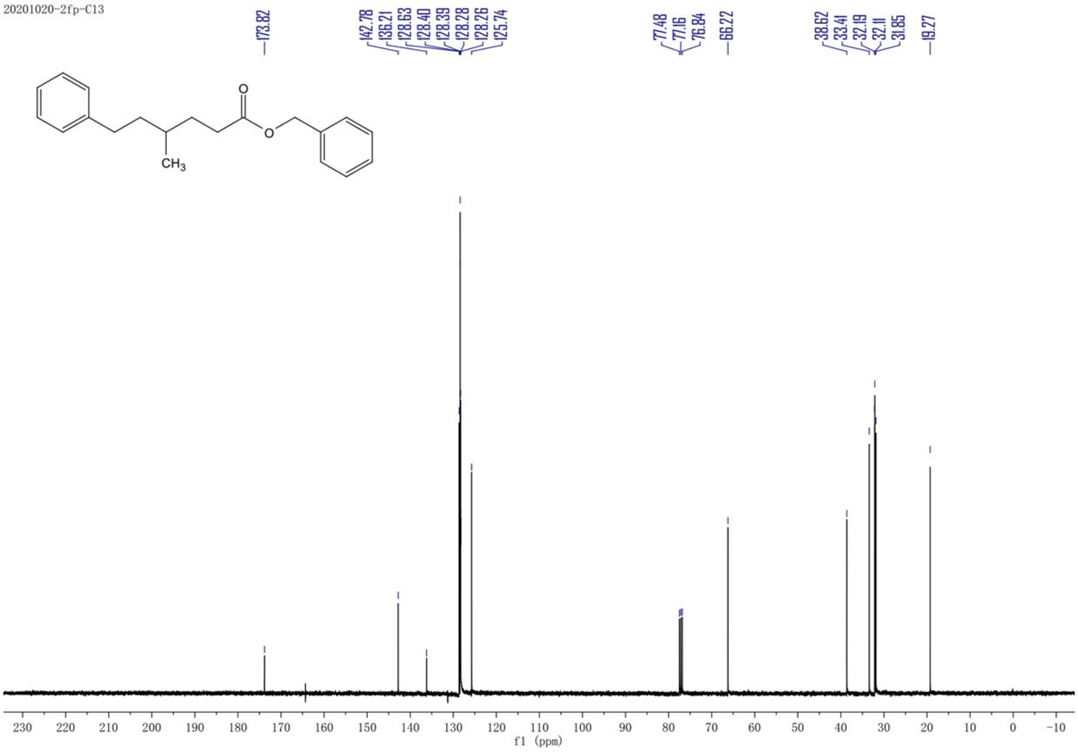
37


20201010-fp
S
O
O
CH3
7.40
7.38
7.38
7.36
7.36
7.35
7.34
7.33
7.32
7.27
7.26
7.26
7.25
6.97
6.96
6.95
6.95
6.93
6.93
5.11
5.10
2.91
2.89
2.88
2.86
2.84
2.32
2.30
2.28
1.94
1.92
1.28
1.27
4.61
1.11
1.86
2.00
1.09
2.16
2.27
3.34
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

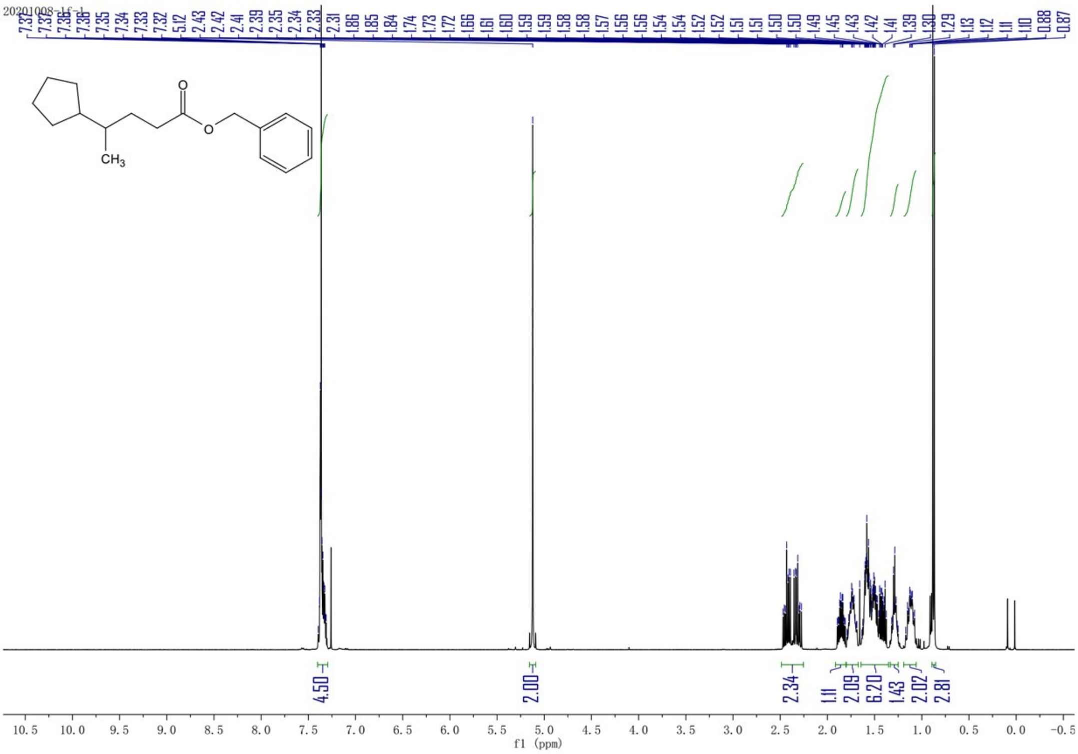

20201010-fp-C13
S
O
O
CH3
173.53
147.24
136.16
128.66
128.32
126.66
125.63
119.57
77.48
77.16
76.84
66.26
34.74
33.10
32.43
21.77
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

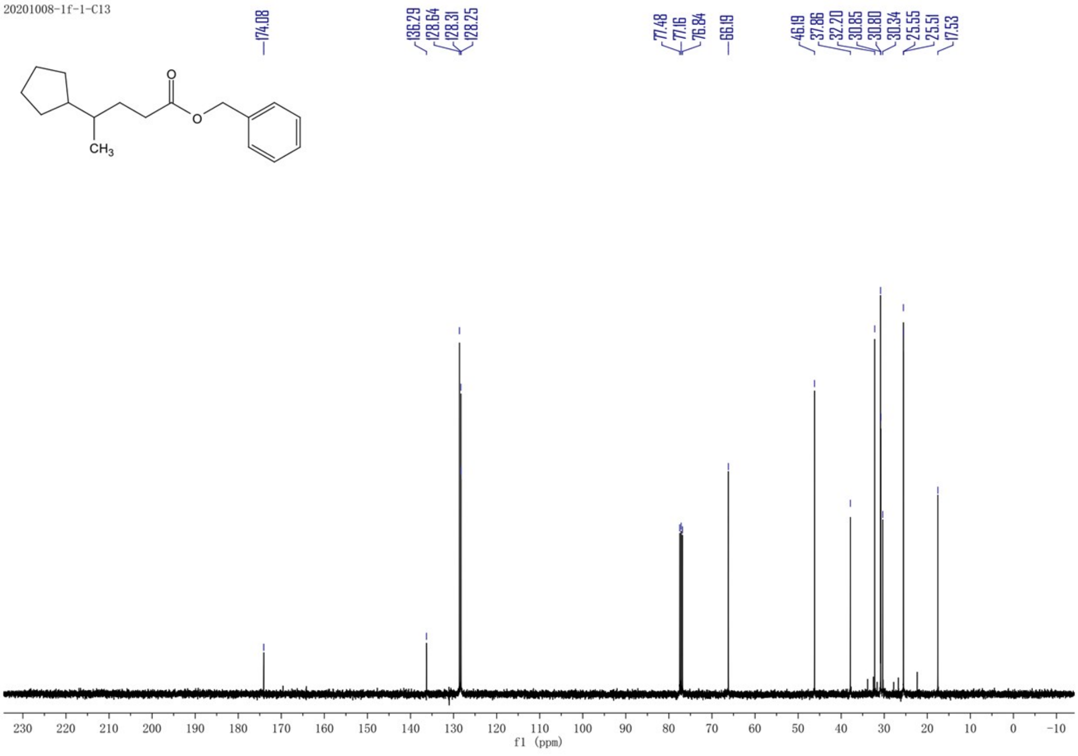
38


20201020-2fp
7.43
7.41
7.41
7.41
7.40
7.39
7.38
7.38
7.37
7.36
7.33
7.31
7.29
7.23
7.22
7.21
7.20
5.16
2.73
2.72
2.70
2.70
2.68
2.67
2.66
2.65
2.63
2.62
2.61
2.61
2.59
1.84
1.82
1.82
1.80
1.80
1.79
1.78
1.62
1.60
1.58
1.57
1.56
1.56
1.55
1.54
1.53
1.52
1.51
1.50
1.50
1.49
1.47
1.00
0.98
O
O
CH3
4.59
2.10
2.85
2.00
2.27
2.31
1.17
1.34
3.31
2.92
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

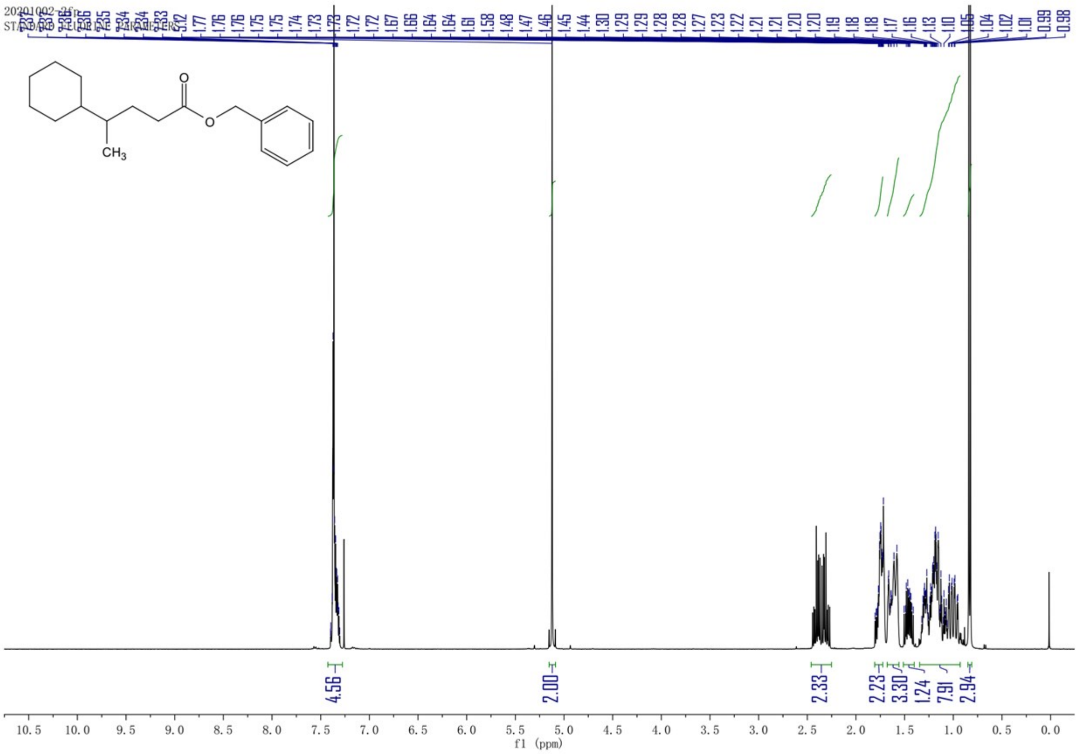

20201020-2fp-C13
173.82
142.78
136.21
128.63
128.40
128.39
128.28
128.26
125.74
77.48
77.16
76.84
66.22
38.62
33.41
32.19
32.11
31.85
19.27
O
O
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

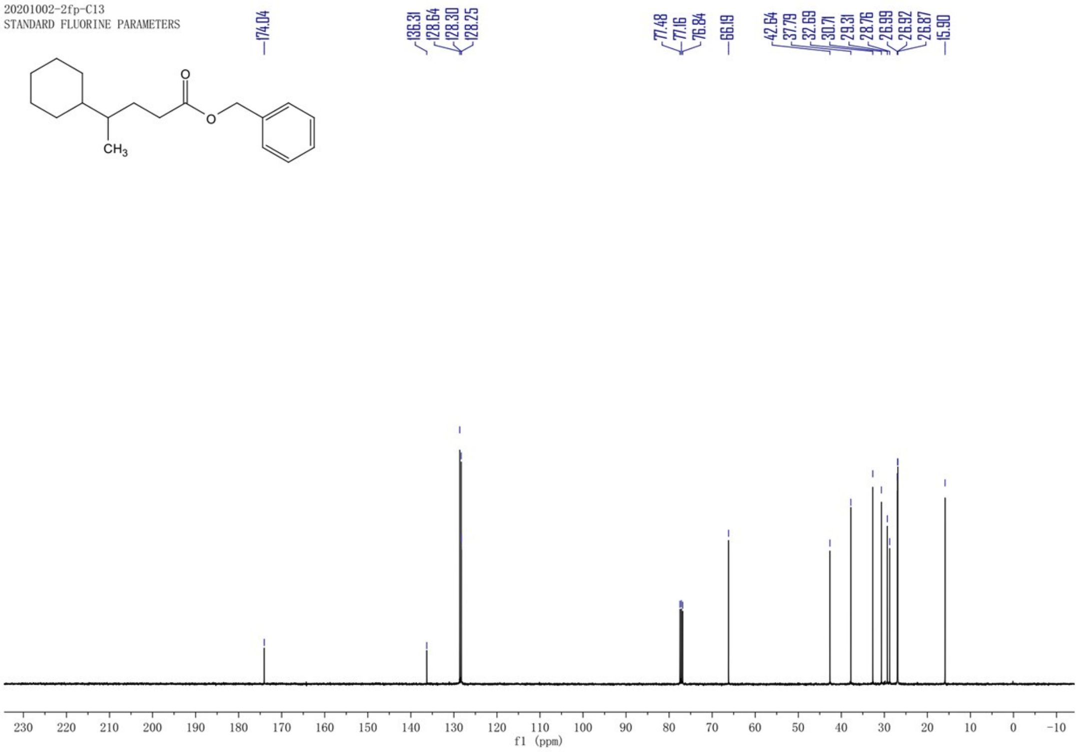
39


20201008-1f-1
7.37 7.37 7.36 7.36 7.35 7.34 7.33 7.32 5.12 2.43 2.42 2.41 2.39 2.35 2.34 2.33 2.31 1.86 1.85 1.84 1.74 1.73 1.72 1.66 1.61 1.60 1.59 1.59 1.58 1.58 1.57 1.56 1.56 1.54 1.54 1.52 1.52 1.51 1.51 1.50 1.50 1.49 1.45 1.43 1.42 1.41 1.39 1.30 1.29 1.13 1.12 1.11 1.10 0.88 0.87
O
O
CH3
4.50
2.00
2.34
1.11
2.09
6.20
1.43
2.02
2.81
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

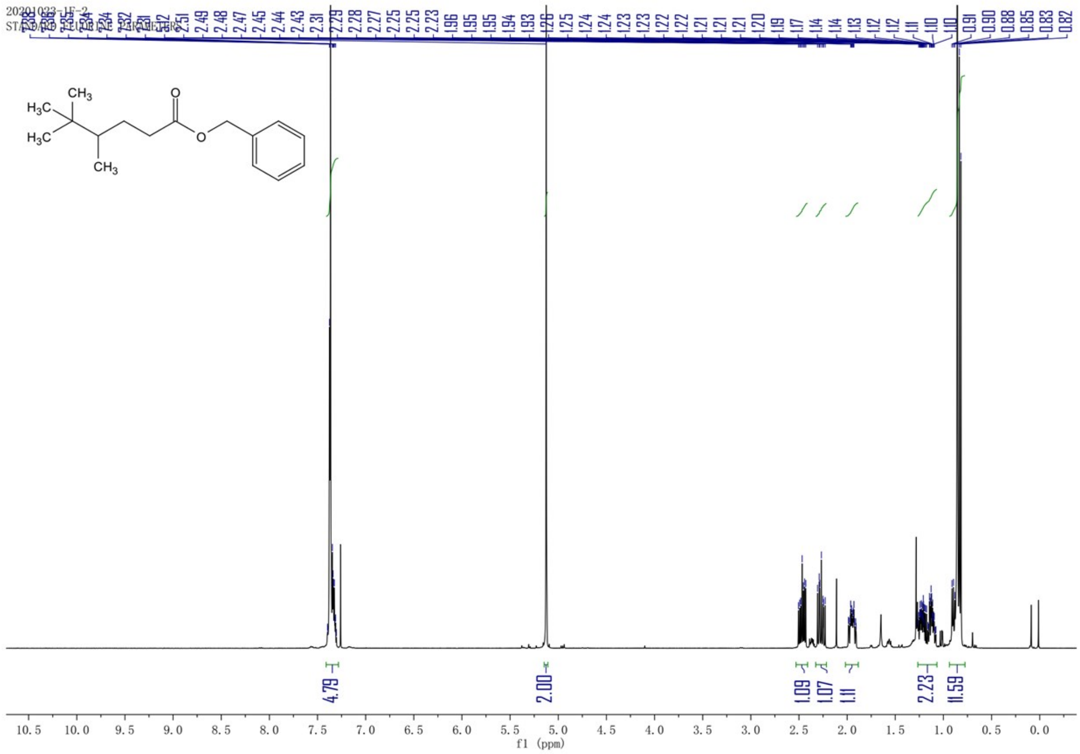

20201008-1f-1-C13
174.08
136.29
128.64
128.31
128.25
77.48
77.16
76.84
66.19
46.19
37.86
32.20
30.85
30.80
30.34
25.55
25.51
17.53
O
O
CH3
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

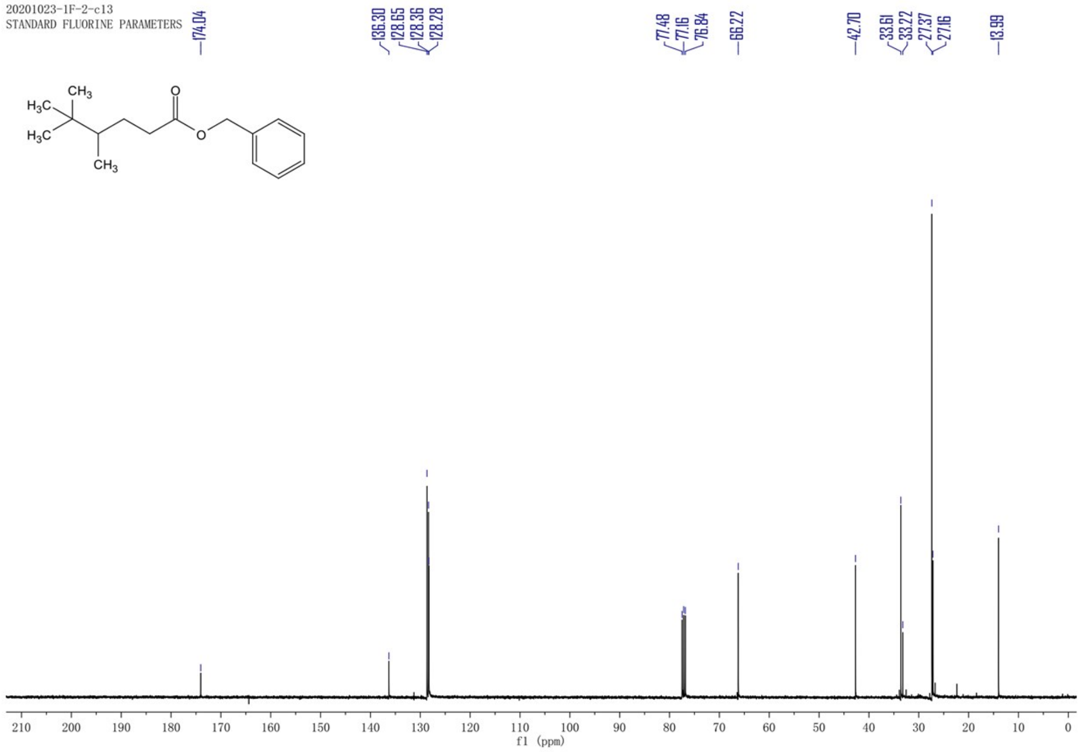
40


20201002-2fp
STANDARD FLUORINE PARAMETERS
7.37
7.37
7.36
7.36
7.35
7.34
7.34
7.33
5.12
1.77
1.76
1.76
1.75
1.75
1.74
1.73
1.73
1.72
1.72
1.67
1.66
1.64
1.64
1.61
1.58
1.48
1.47
1.46
1.45
1.44
1.30
1.29
1.29
1.28
1.28
1.27
1.23
1.22
1.21
1.21
1.20
1.20
1.19
1.18
1.18
1.17
1.16
1.13
1.10
1.05
1.04
1.02
1.01
0.99
0.98
CH3
O
O
4.56
2.00
2.33
2.23
3.30
1.24
7.91
2.94
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

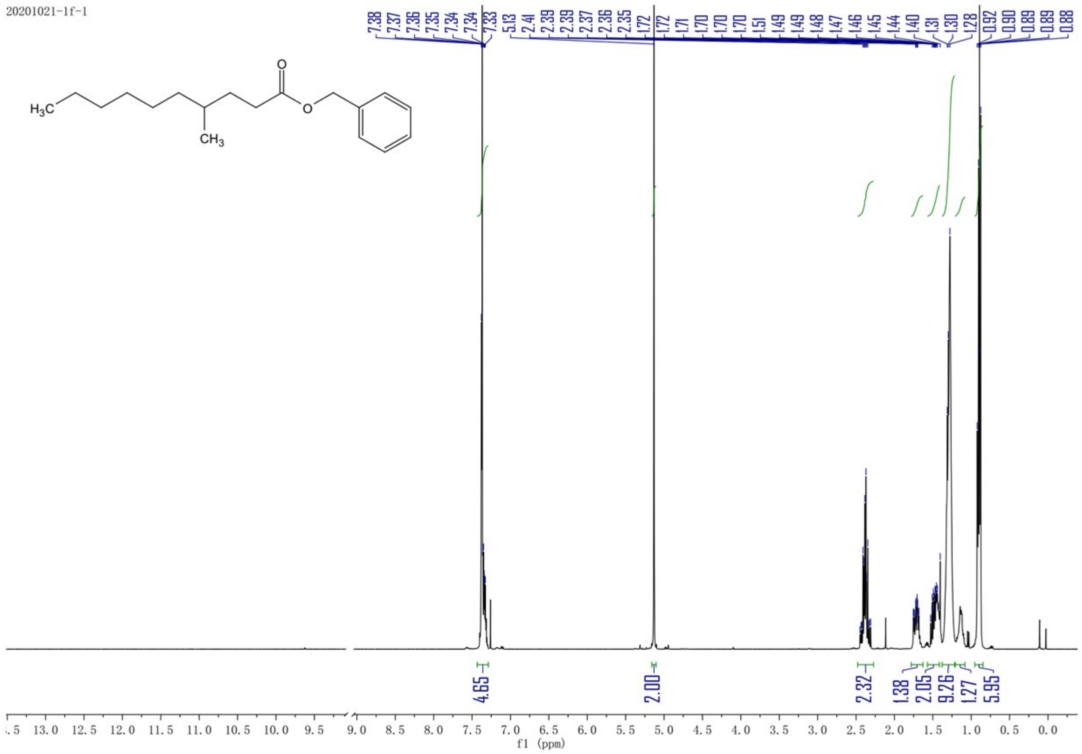

20201002-2fp-C13
STANDARD FLUORINE PARAMETERS
174.04
136.31
128.64
128.30
128.25
77.48
77.16
76.84
66.19
42.64
37.79
32.69
30.71
29.31
28.76
26.99
26.92
26.87
15.90
CH3
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

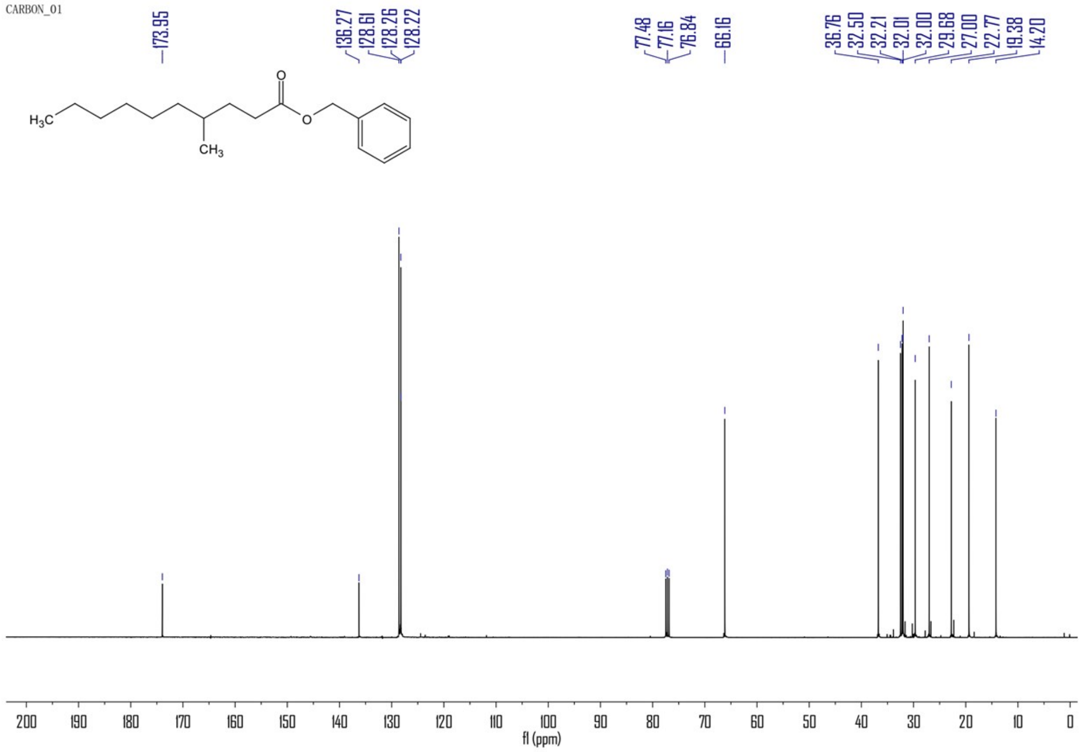
41


20201023-1F-2
STANDARD FLUORINE PARAMETERS
7.38 7.36 7.35 7.34 7.34 7.32 7.31 7.31 2.51 2.49 2.48 2.47 2.45 2.44 2.43 2.31 2.29 2.28 2.27 2.25 2.25 2.23 1.96 1.95 1.95 1.94 1.93 1.26 1.25 1.24 1.24 1.23 1.23 1.22 1.22 1.21 1.21 1.21 1.20 1.19 1.17 1.14 1.14 1.13 1.12 1.12 1.11 1.10 1.10 0.91 0.90 0.88 0.85 0.83 0.82
H_3C
CH_3
H_3C
CH_3
O
O
4.79
2.00
1.09
1.07
1.11
2.23
11.59
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

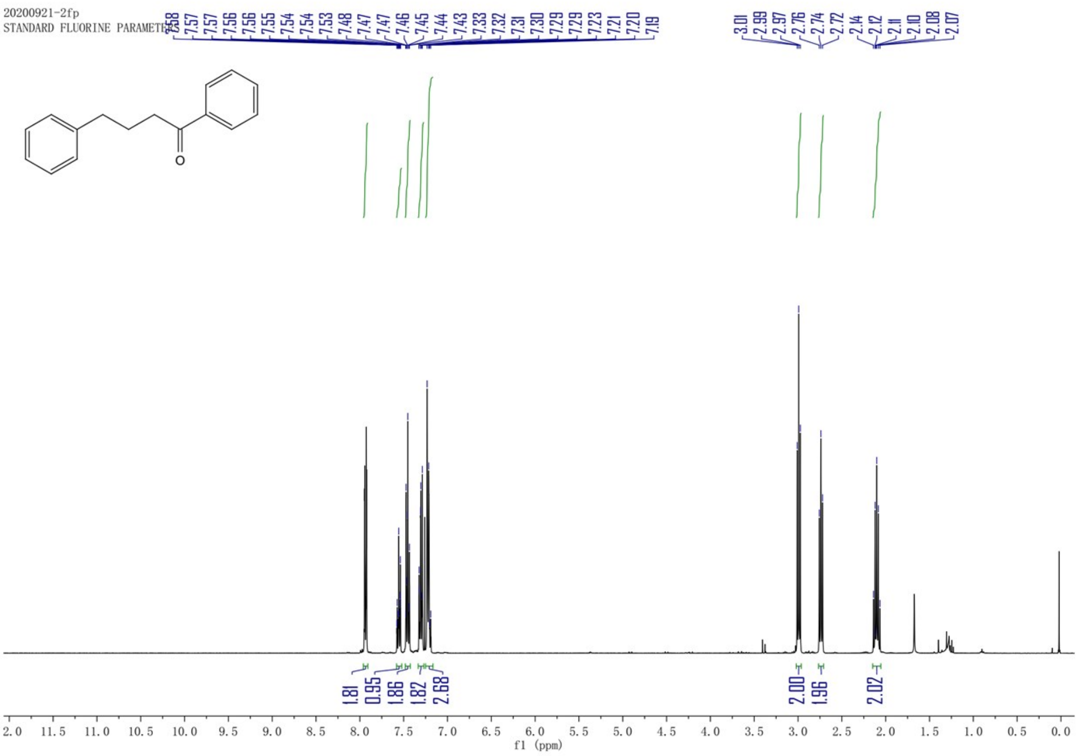

20201023-1F-2-c13
STANDARD FLUORINE PARAMETERS
174.04
136.30 128.65 128.36 128.28
77.48 77.16 76.84
66.22
42.70
33.61 33.22
27.37 27.16
13.99
H_3C
CH_3
H_3C
CH_3
O
O
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

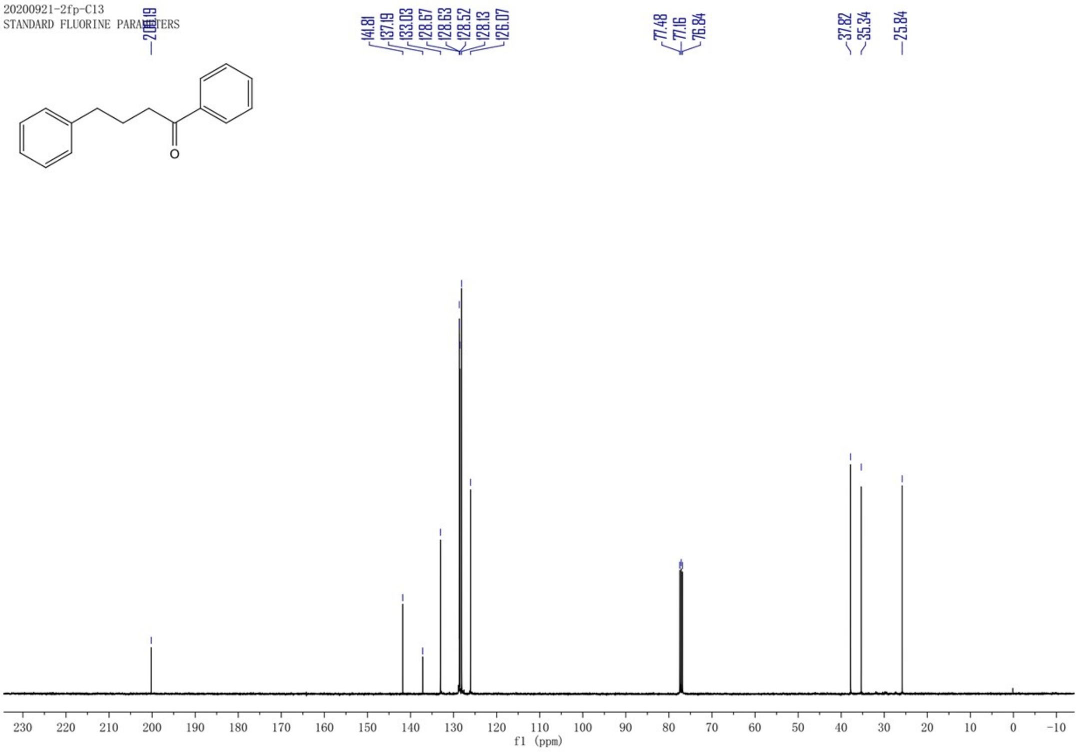
42


20201021-1f-1
H3C
CH3
O
O
7.38
7.37
7.36
7.35
7.34
7.34
7.33
5.13
2.41
2.39
2.39
2.37
2.36
2.35
1.72
1.72
1.71
1.70
1.70
1.70
1.51
1.49
1.49
1.48
1.47
1.46
1.45
1.44
1.40
1.31
1.30
1.28
0.92
0.90
0.89
0.89
0.88
4.65
2.00
2.32
1.38
2.05
9.26
1.27
5.95
f1 (ppm)

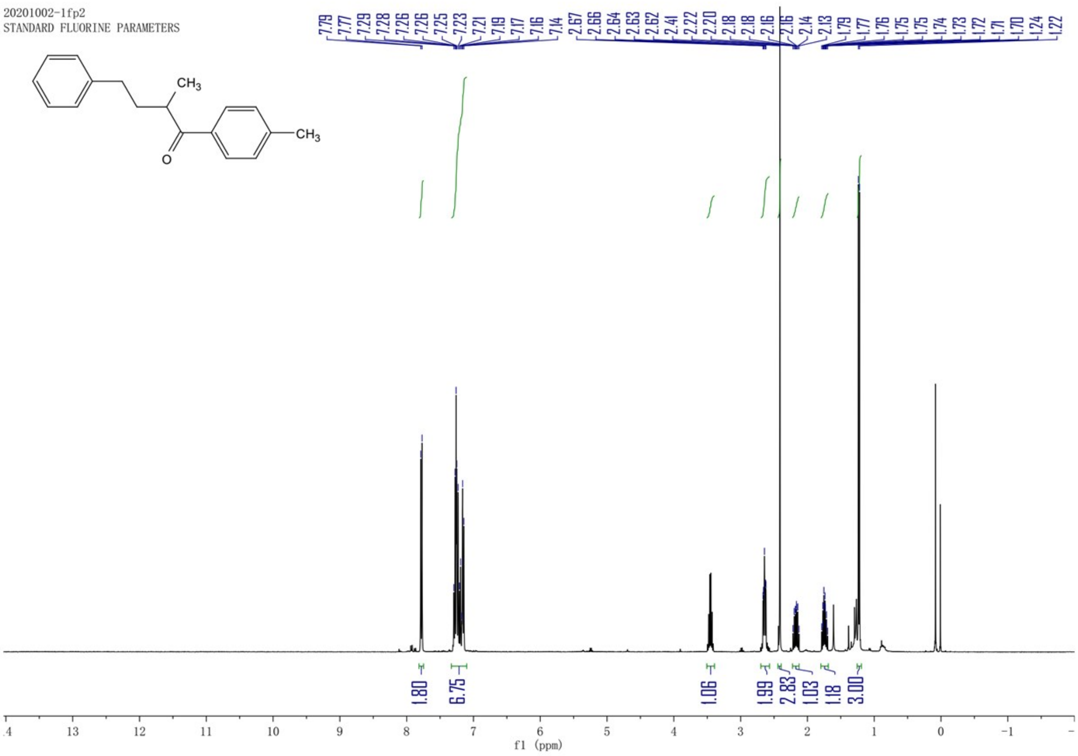

CARBON_01
H3C
CH3
O
O
173.95
136.27
128.61
128.26
128.22
77.48
77.16
76.84
66.16
36.76
32.50
32.21
32.01
32.00
29.68
27.00
22.77
19.38
14.20
f1 (ppm)

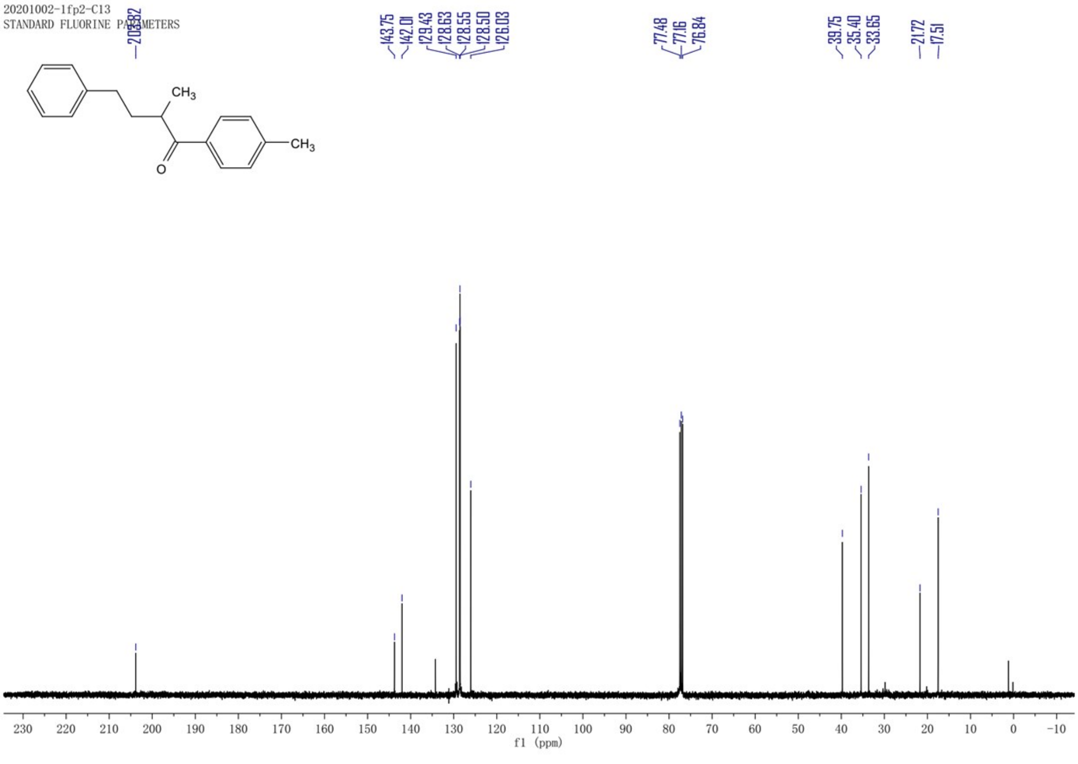
43


20200921-2fp
STANDARD FLUORINE PARAMETERS
7.58 7.57 7.57 7.56 7.56 7.55 7.54 7.54 7.53 7.48 7.47 7.47 7.46 7.45 7.44 7.43 7.33 7.32 7.31 7.30 7.29 7.29 7.23 7.21 7.20 7.19
3.01 2.99 2.97 2.76 2.74 2.72 2.14 2.12 2.11 2.10 2.08 2.07
O
1.81 0.95 1.86 1.82 2.68
2.00 1.96 2.02
2.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

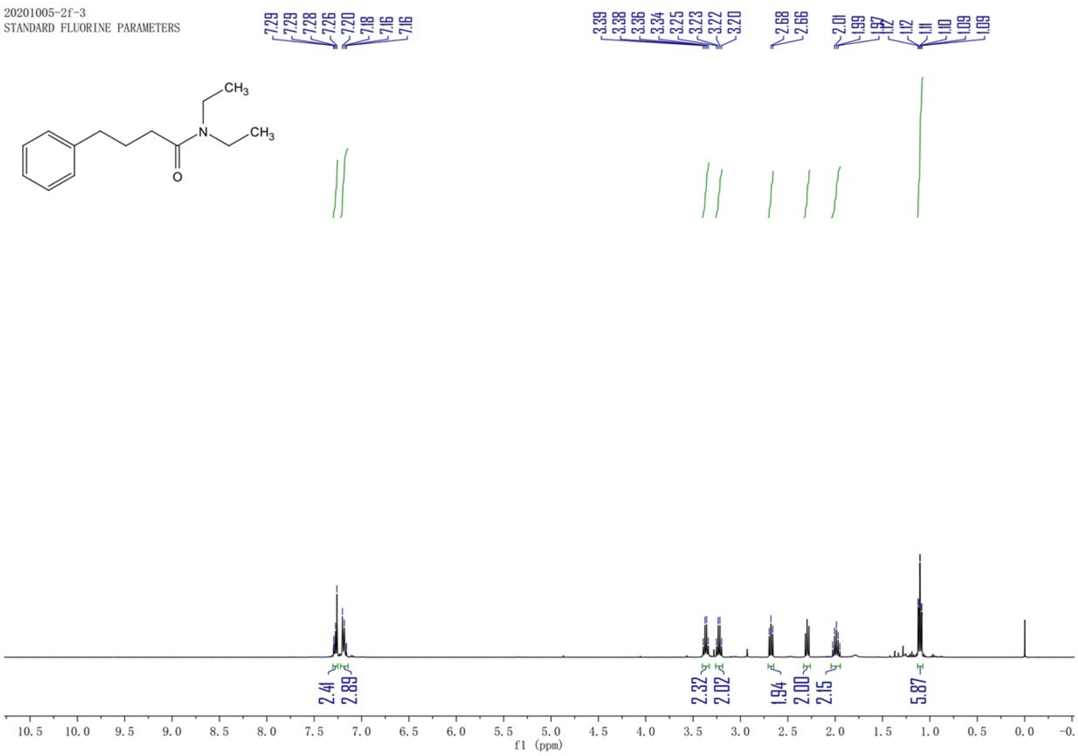

20200921-2fp-C13
STANDARD FLUORINE PARAMETERS
200.19
141.81 137.19 133.03 128.67 128.63 128.52 128.13 126.07
77.48 77.16 76.84
37.82 35.34
25.84
O
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

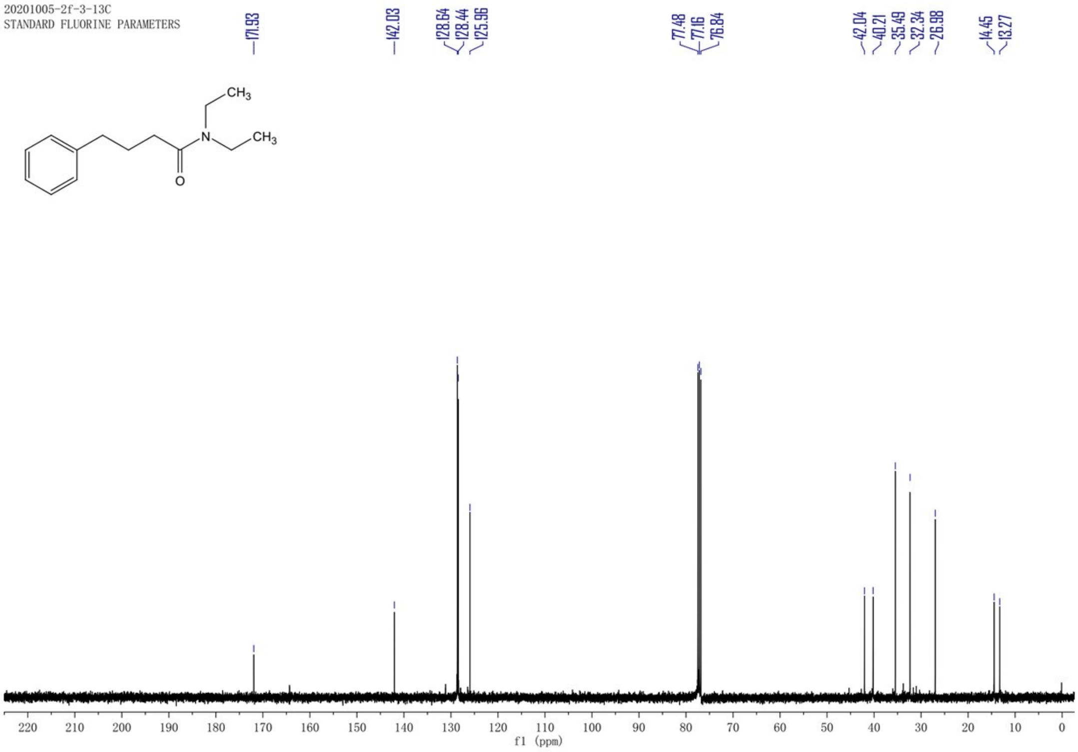
44


20201002-1fp2
STANDARD FLUORINE PARAMETERS
CH3
CH3
O
7.79
7.77
7.29
7.28
7.26
7.26
7.25
7.23
7.21
7.19
7.17
7.16
7.14
2.67
2.66
2.64
2.63
2.62
2.41
2.22
2.20
2.18
2.18
2.16
2.16
2.14
2.13
1.79
1.77
1.76
1.75
1.75
1.74
1.73
1.72
1.71
1.70
1.24
1.22
1.80
6.75
1.06
1.99
2.83
1.03
1.18
3.00
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
−1
f1 (ppm)

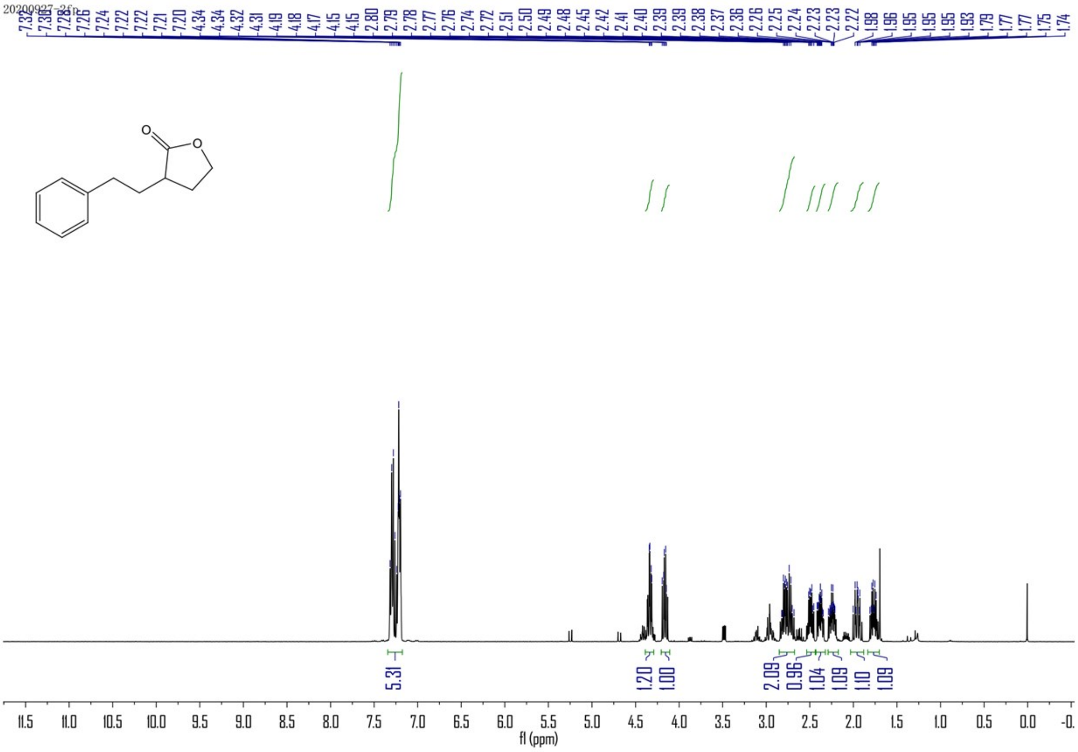

20201002-1fp2-C13
STANDARD FLUORINE PARAMETERS
—203.82
143.75
142.01
129.43
128.63
128.55
128.50
126.03
77.48
77.16
76.84
39.75
35.40
33.65
21.72
17.51
CH3
CH3
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
−10
f1 (ppm)

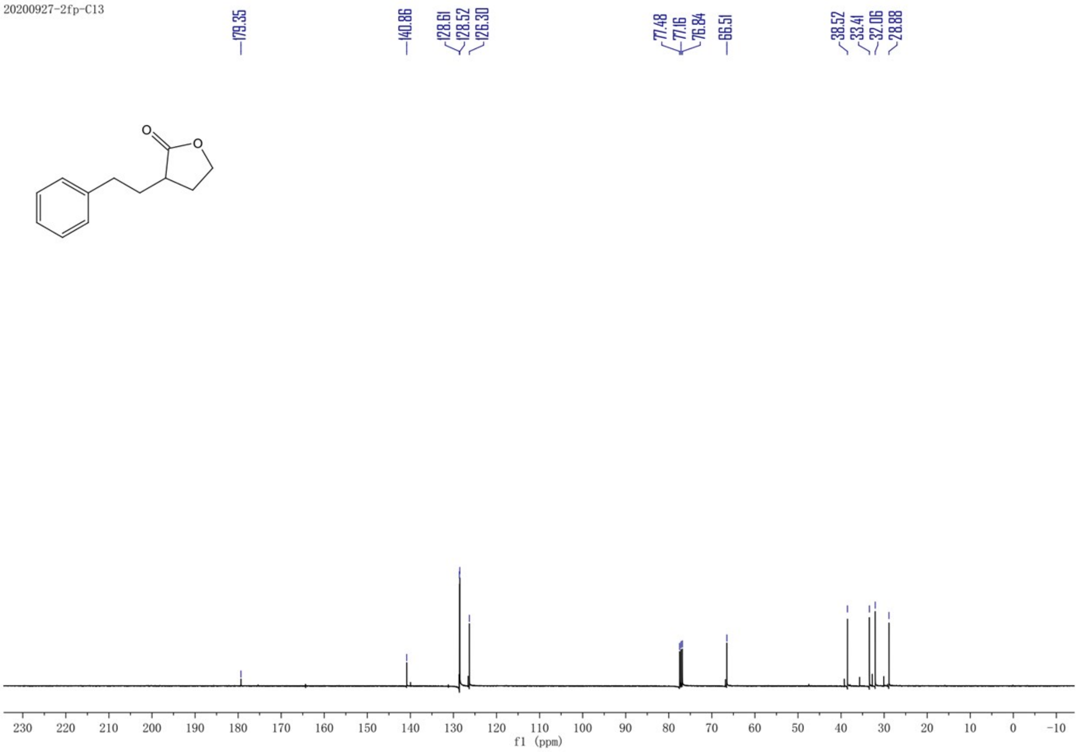
45


20201005-2f-3
STANDARD FLUORINE PARAMETERS
7.29
7.29
7.28
7.26
7.20
7.18
7.16
7.16
3.39
3.38
3.36
3.34
3.25
3.23
3.22
3.20
2.68
2.66
2.01
1.99
1.97
1.12
1.11
1.10
1.09
1.09
CH3
N
CH3
O
2.41
2.89
2.32
2.02
1.94
2.00
2.15
5.87
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)

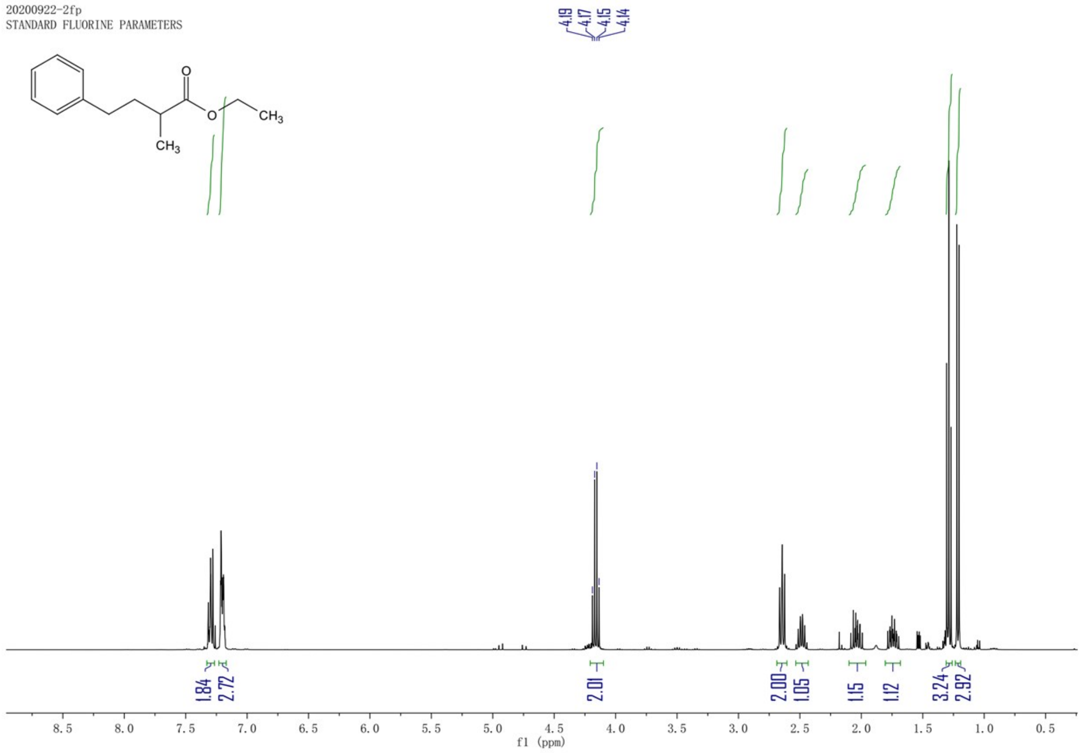

20201005-2f-3-13C
STANDARD FLUORINE PARAMETERS
171.93
142.03
128.64
128.44
125.96
77.48
77.16
76.84
42.04
40.21
35.49
32.34
26.98
14.45
13.27
CH3
N
CH3
O
220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

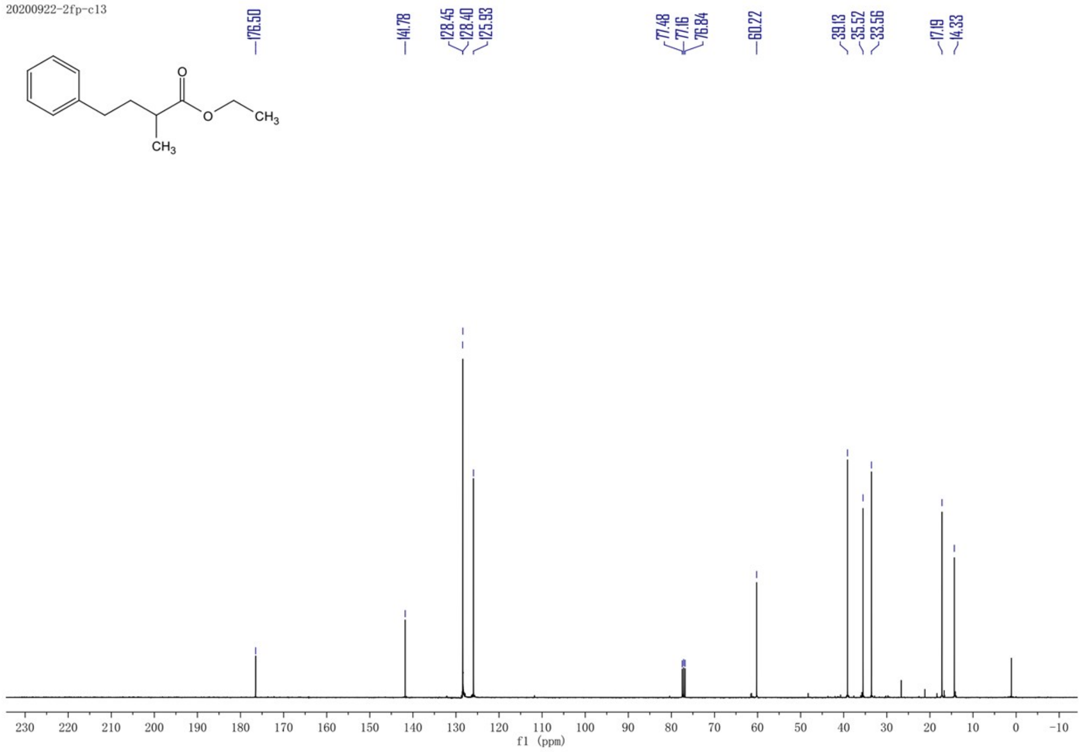
46


20200927-2fp
7.32
7.30
7.28
7.26
7.24
7.22
7.22
7.21
7.20
4.34
4.34
4.32
4.31
4.19
4.18
4.17
4.15
4.15
2.80
2.79
2.78
2.77
2.76
2.74
2.72
2.51
2.50
2.49
2.48
2.45
2.42
2.41
2.40
2.39
2.39
2.38
2.37
2.36
2.26
2.25
2.24
2.23
2.23
2.22
1.98
1.96
1.95
1.95
1.95
1.93
1.79
1.77
1.75
1.74
O
O
5.31
1.20
1.00
2.09
0.96
1.04
1.09
1.10
1.09
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.
f1 (ppm)

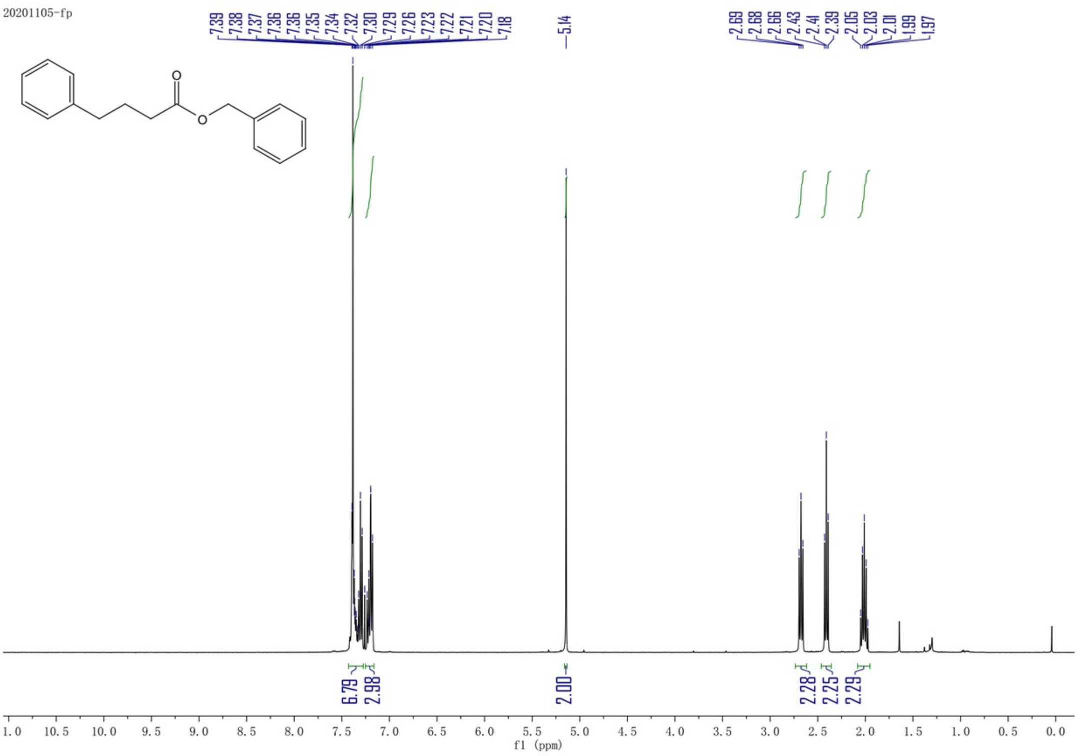

20200927-2fp-C13
179.35
140.86
128.61
128.52
126.30
77.48
77.16
76.84
66.51
38.52
33.41
32.06
28.88
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

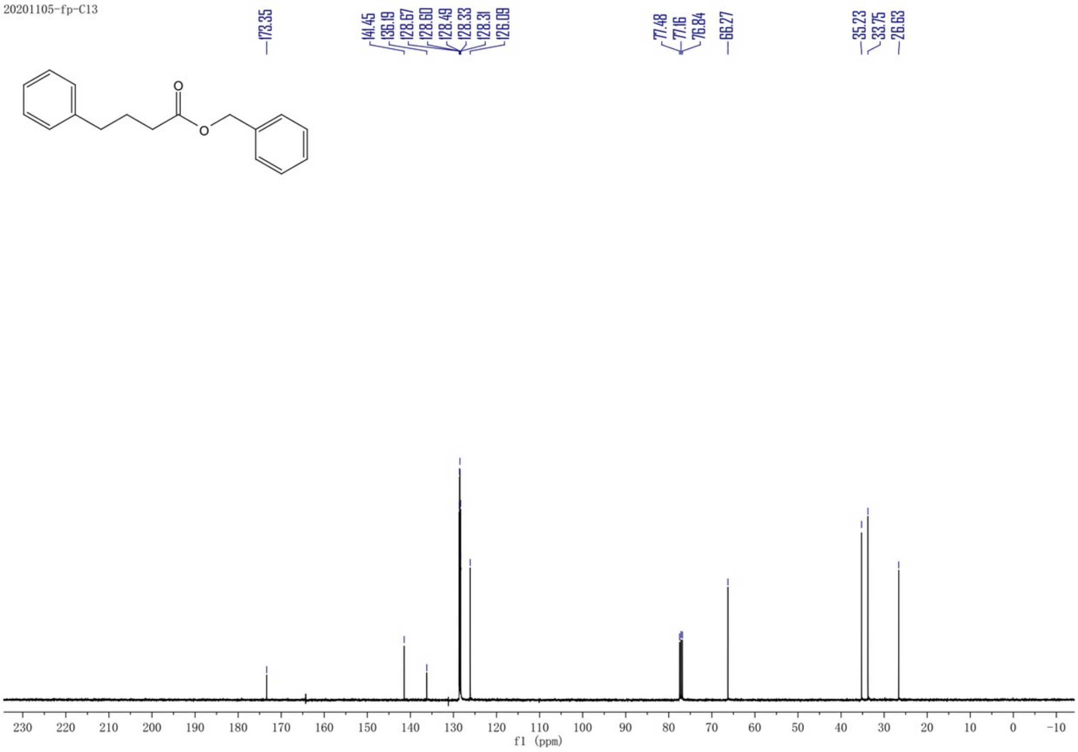
47


20200922-2fp
STANDARD FLUORINE PARAMETERS
O
O
CH3
CH3
4.19
4.17
4.15
4.14
1.84
2.72
2.01
2.00
1.05
1.15
1.12
3.24
2.92
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

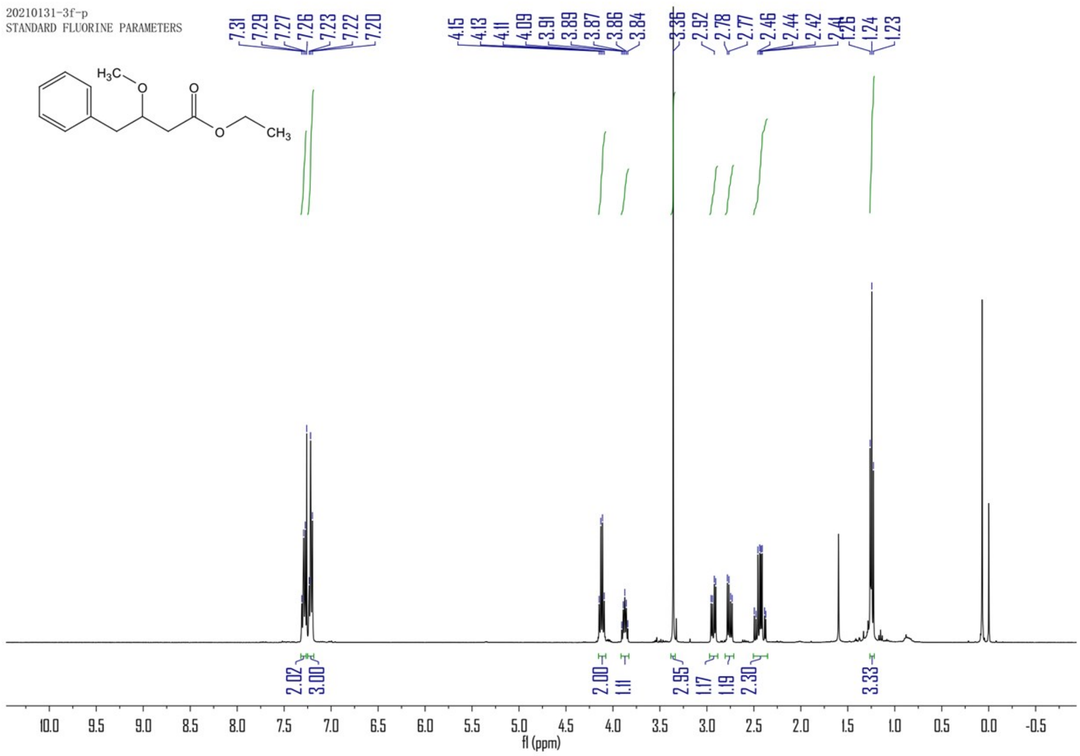

20200922-2fp-c13
176.50
141.78
128.45
128.40
125.93
77.48
77.16
76.84
60.22
39.13
35.52
33.56
17.19
14.33
O
O
CH3
CH3
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

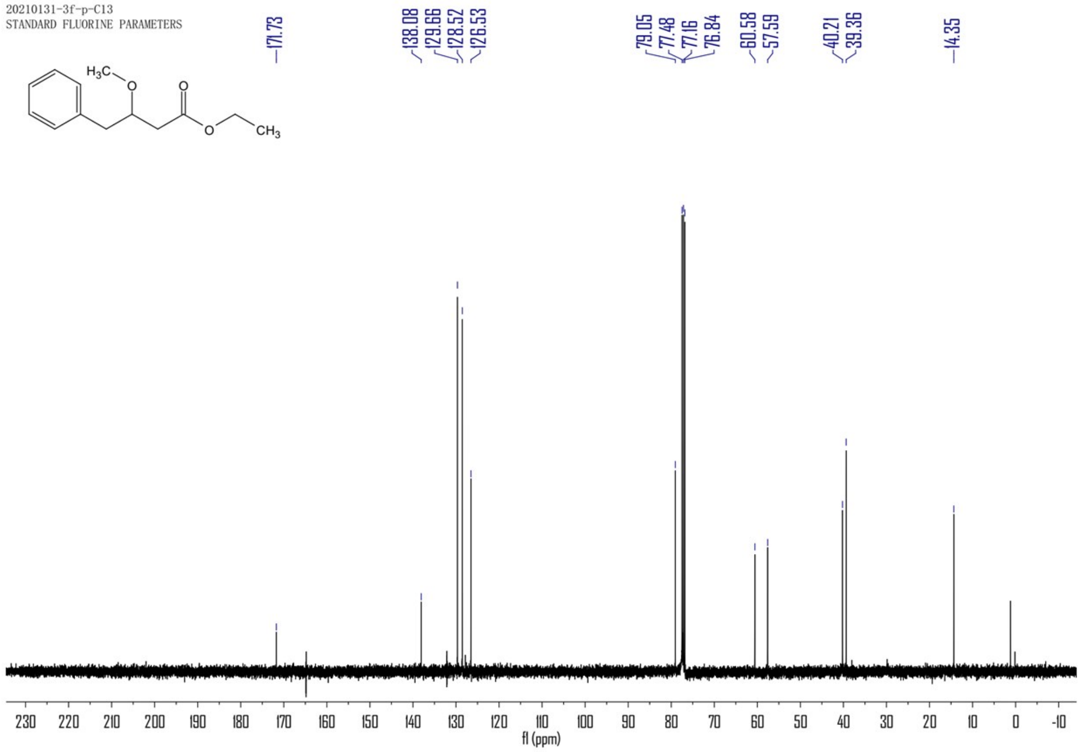
48


20201105-fp
O
O
7.39
7.38
7.37
7.36
7.36
7.35
7.34
7.32
7.30
7.29
7.26
7.23
7.22
7.21
7.20
7.18
5.14
2.69
2.68
2.66
2.43
2.41
2.39
2.05
2.03
2.01
1.99
1.97
6.79
2.98
2.00
2.28
2.25
2.29
1.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

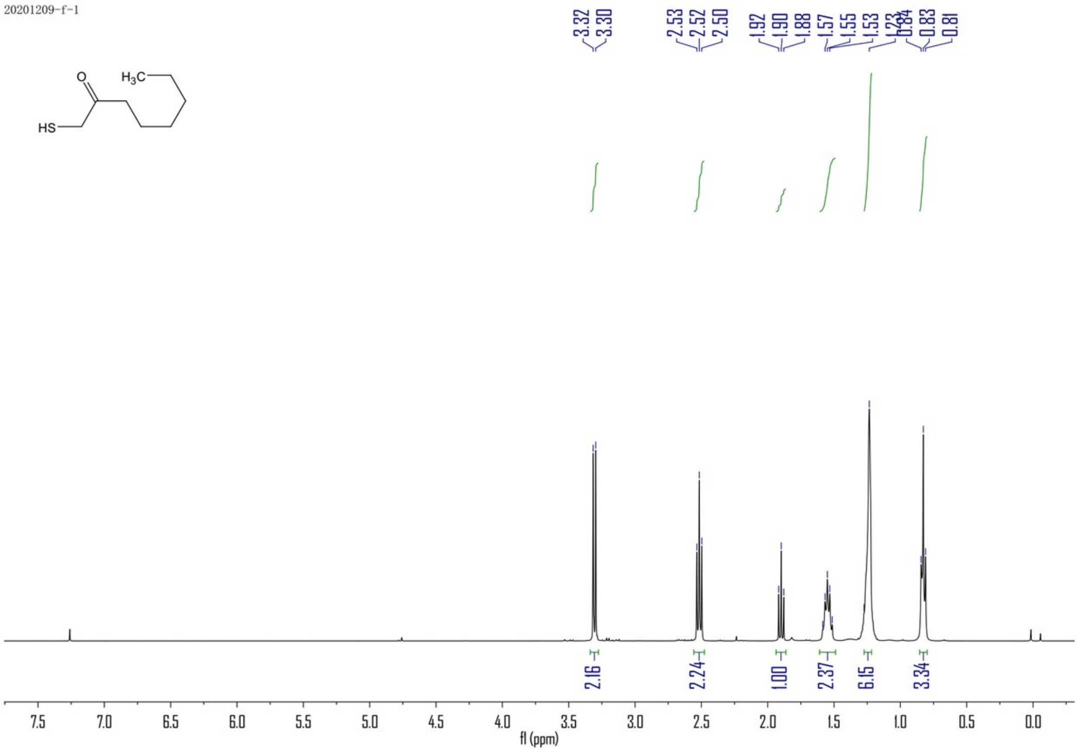

20201105-fp-C13
O
O
173.35
141.45
136.19
128.67
128.60
128.49
128.33
128.31
126.09
77.48
77.16
76.84
66.27
35.23
33.75
26.63
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

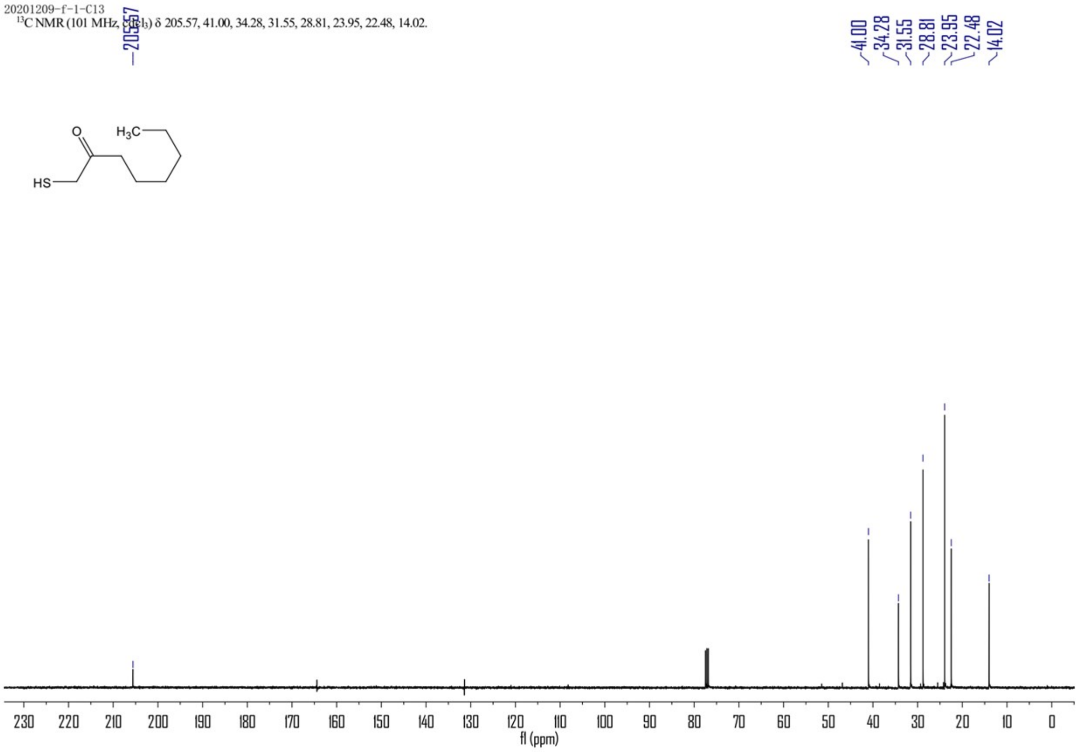
50


20210131-3f-p
STANDARD FLUORINE PARAMETERS
H3C
O
O
O
CH3
7.31
7.29
7.27
7.26
7.23
7.22
7.20
4.15
4.13
4.11
4.09
3.91
3.89
3.87
3.86
3.84
3.36
2.92
2.78
2.77
2.46
2.44
2.42
2.41
1.26
1.24
1.23
2.02
3.00
2.00
1.11
2.95
1.17
1.19
2.30
3.33
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
f1 (ppm)


20210131-3f-p-C13
STANDARD FLUORINE PARAMETERS
H3C
O
O
O
CH3
171.73
138.08
129.66
128.52
126.53
79.05
77.48
77.16
76.84
60.58
57.59
40.21
39.36
14.35
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

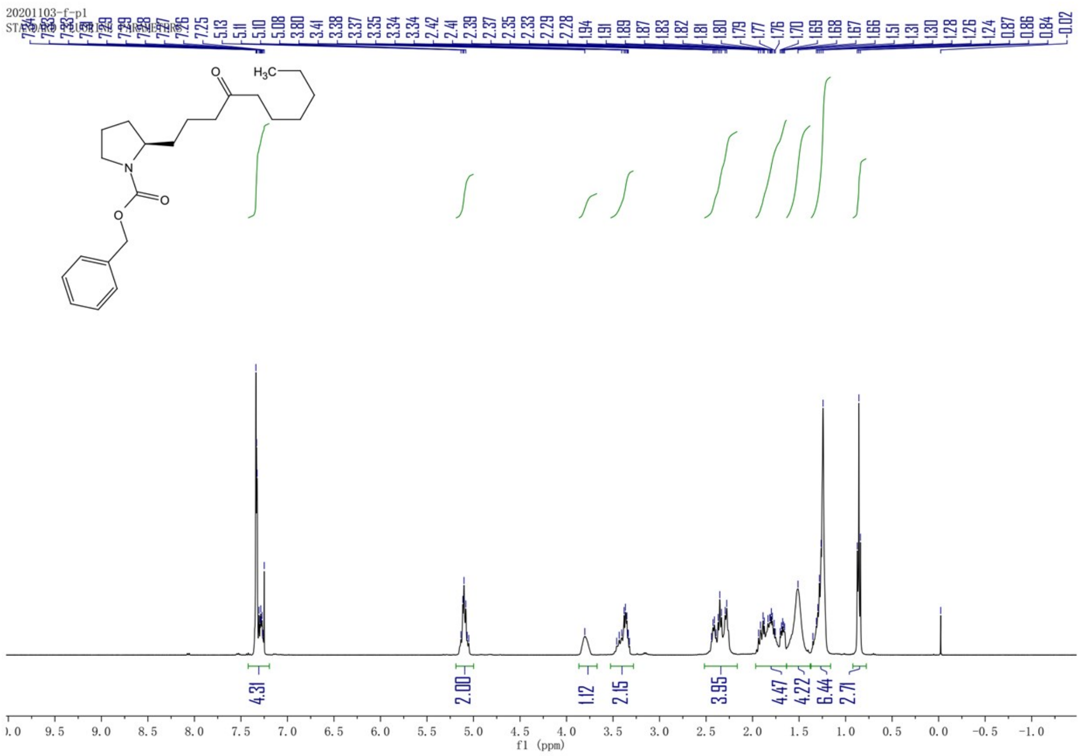
f

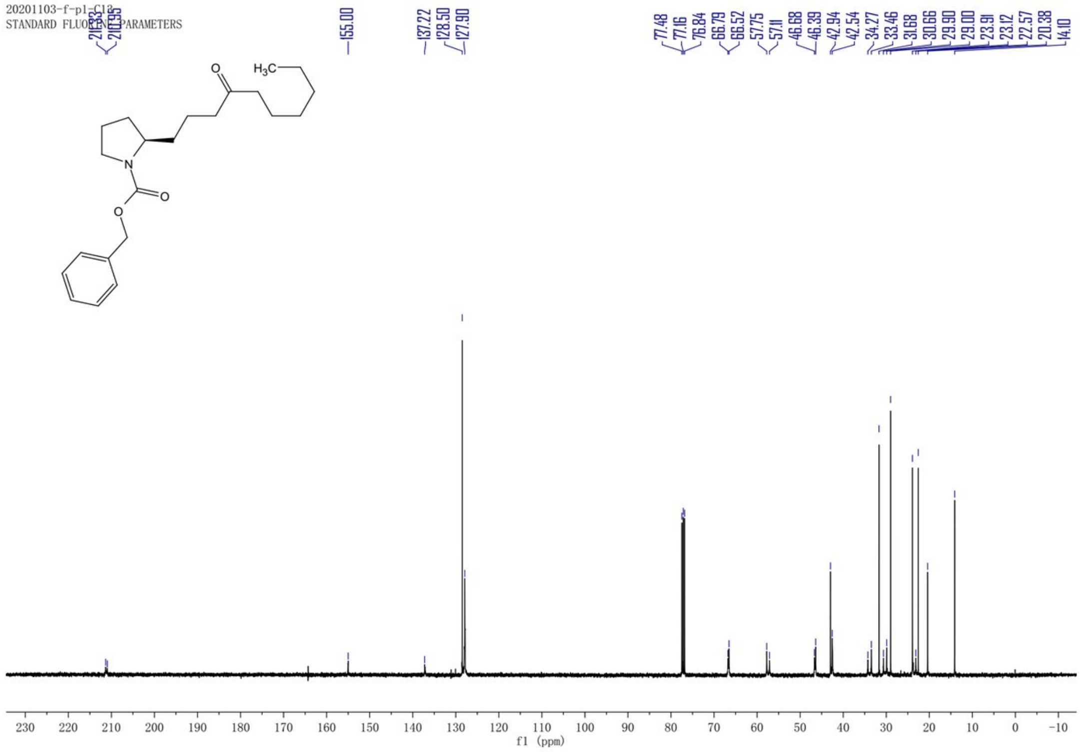




^{13}C NMR (101 MHz, CDCl$_3$) δ 205.57, 41.00, 34.28, 31.55, 28.81, 23.95, 22.48, 14.02.

52


20201103-f-p1
STANDARD FLUORINE PARAMETERS
7.34
7.33
7.33
7.31
7.29
7.29
7.28
7.27
7.26
7.25
5.13
5.11
5.10
5.08
3.80
3.41
3.38
3.37
3.35
3.34
3.34
2.42
2.41
2.39
2.37
2.35
2.33
2.29
2.28
1.94
1.91
1.89
1.87
1.83
1.82
1.81
1.80
1.79
1.77
1.76
1.70
1.69
1.68
1.67
1.66
1.51
1.31
1.30
1.28
1.26
1.24
0.87
0.86
0.84
-0.02
O
H3C
N
O
O
4.31
2.00
1.12
2.15
3.95
4.47
4.22
6.44
2.71
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)


20201103-f-p1-C13
STANDARD FLUORINE PARAMETERS
211.33
210.95
155.00
137.22
128.50
127.90
77.48
77.16
76.84
66.79
66.52
57.75
57.11
46.68
46.39
42.94
42.54
34.27
33.46
31.68
30.66
29.90
29.00
23.91
23.12
22.57
20.38
14.10
O
H3C
N
O
O
230
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

indolizidine 209D