

## Supporting Information

# A Dual Role for Acetohydrazide in Pd-Catalyzed Controlled C(sp<sup>3</sup>)-H Acetoxylation of Aldehydes

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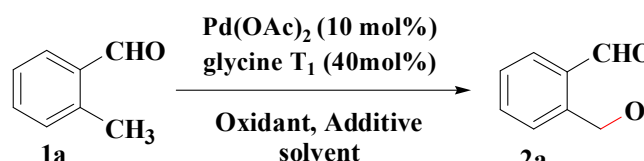
## 1. General Information

All the reagents were purchased from Aladdin and Alfa without further purification. The MCM-48 molecular sieve was purchased from NanJing Ji Cang Nano Technology Ltd. in China. Thin layer chromatography (TLC) was performed on pre-coated silica gel GF254 plates. The  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were measured on a 600 MHz Bruker Avance III nuclear magnetic resonance spectrometer, using  $\text{CDCl}_3$  as the solvent with tetramethylsilane (TMS) as the internal standard. Chemical shifts ( $\delta$ ) are expressed in ppm. The structures of known compounds were further corroborated by comparing their  $^1\text{H}$  NMR data with those of literature.

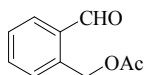
## 2. Screening reaction conditions

A mixture of *o*-methylbenzaldehyde **1a** (0.20 mmol),  $\text{Pd}(\text{OAc})_2$  (4.5 mg, 10 mol %), glycine (40 mol %), oxidant (0.40 mmol) and additive (0.40 mmol) in solvent (2.0 mL) were charged in a 25 mL oven dried reaction tube. The reaction mixture was heated and refluxed for 48 h at 110  $^\circ\text{C}$ . After cooling to room temperature, the mixture was filtered, and the filtrate was evaporated in vacuo. The residue was purified by flash column chromatography (silica gel, ethyl acetate/petroleum ether = 1:5 to 1:10 as an eluent) to afford the desired product **3a**.

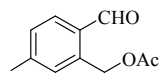
**Table S1. Screening reaction conditions**

|  |                                  |                       |                                            |          |
|------------------------------------------------------------------------------------|----------------------------------|-----------------------|--------------------------------------------|----------|
| Entry                                                                              | Oxidant                          | Additive              | Solvent                                    | Yield(%) |
| 1                                                                                  | $\text{PhI}(\text{OAc})_2$       |                       | $\text{CH}_3\text{CN}$                     | NP       |
| 2                                                                                  | $\text{PhI}(\text{OAc})_2$       |                       | $\text{AcOH}-\text{Ac}_2\text{O}$          | NP       |
| 3                                                                                  | $\text{PhI}(\text{OAc})_2$       |                       | $\text{AcOH}$                              | NP       |
| 4                                                                                  | $\text{PhI}(\text{OAc})_2$       | $\text{LiOAc}$        | $\text{DCE}$                               | NP       |
|                                                                                    | $\text{PhI}(\text{OAc})_2$       | $\text{Ac}_2\text{O}$ | toluene                                    | NP       |
| 5                                                                                  | $\text{PhI}(\text{OAc})_2$       |                       | $\text{C}_6\text{H}_6-\text{Ac}_2\text{O}$ | NP       |
| 6                                                                                  | Oxone                            |                       | $\text{AcOH}$                              | NP       |
| 7                                                                                  | Oxone                            |                       | $\text{AcOH}-\text{Ac}_2\text{O}$          | NP       |
| 8                                                                                  | $\text{MeCOOO}^t\text{Bu}$       |                       | $\text{Ac}_2\text{O}$                      | NP       |
| 9                                                                                  | $\text{MeCOOO}^t\text{Bu}$       |                       | $\text{Ac}_2\text{O}-\text{HFIP}$          | NP       |
| 10                                                                                 | BQ                               |                       | $\text{AcOH}$                              | NP       |
| 11                                                                                 | $\text{CH}_3\text{CO}_3\text{H}$ |                       | $\text{Ac}_2\text{O}$                      | NP       |
| 12                                                                                 | $\text{K}_2\text{S}_2\text{O}_8$ |                       | $\text{AcOH}$                              | 35       |
| 13                                                                                 | $\text{Ag}_2\text{O}$            |                       | $\text{AcOH}$                              | NP       |
| 14                                                                                 | $\text{Ag}_2\text{CO}_3$         |                       | $\text{AcOH}$                              | NP       |
| 15                                                                                 | $\text{AgOAc}$                   |                       | $\text{AcOH}$                              | NP       |
| 16                                                                                 | $\text{Cu}(\text{OAc})_2$        |                       | $\text{AcOH}$                              | NP       |

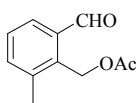
### 3. Characterization Data for the Products



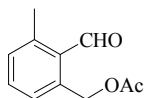
2-formylbenzyl acetate **2a**.<sup>1</sup> Yield: 73% (25.9 mg); <sup>1</sup>H NMR (600 MHz, Chloroform-*d*) δ 10.20 (s, 1H), 7.93-7.85 (m, 1H), 7.66-7.59 (m, 1H), 7.58-7.47 (m, 2H), 5.56 (s, 2H), 2.15 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 192.4, 170.6, 137.9, 133.9, 133.6, 133.3, 128.6, 128.4, 63.5, 20.9; MS (EI) *m/z* (%) 178.4(M<sup>+</sup>, 2), 135.0(88), 118.0(100), 90.0(97), 65.0(28).



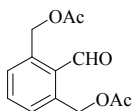
2-formyl-5-methylbenzyl acetate **2b**. Yield: 67% (25.7 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.13 (s, 1H), 7.77 (d, *J* = 8.2 Hz, 1H), 7.38 – 7.30 (m, 2H), 5.52 (s, 2H), 2.45 (s, 3H), 2.15 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 192.0, 170.6, 145.1, 137.8, 133.6, 131.4, 129.5, 129.1, 63.6, 21.9, 21.0.



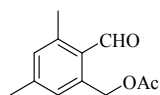
2-formyl-6-methylbenzyl acetate. **2c**. Yield: 63% (24.2 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.31 (s, 1H), 7.74 (d, *J* = 7.3 Hz, 1H), 7.54 – 7.36 (m, 2H), 5.55 (s, 2H), 2.47 (s, 3H), 2.06 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 192.20, 170.77, 139.45, 136.03, 135.31, 134.85, 129.55, 129.06, 58.73, 20.81, 19.20.



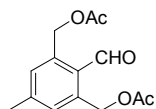
2-formyl-3-methylbenzyl acetate. **2d**. Yield: 37% (14.2 mg); <sup>1</sup>H NMR (600 MHz, Chloroform-*d*) δ 10.59 (s, 1H), 7.51-7.41 (m, 1H), 7.36 (s, 1H), 7.25-7.17 (m, 1H), 5.47 (s, 2H), 2.67 (s, 3H), 2.13 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 192.8, 170.6, 141.8, 138.4, 133.3, 131.8, 126.7, 64.3, 29.7, 21.0, 20.1.



(2-formyl-1,3-phenylene)bis(methylene) diacetate **2d'**. Yield: 24% (12.0 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.53 (s, 1H), 7.61 – 7.56 (m, 1H), 7.54 – 7.48 (m, 2H), 5.48 (s, 4H), 2.13 (s, 6H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 191.9, 170.5, 138.9, 133.4, 131.7, 129.3, 63.8, 20.9.

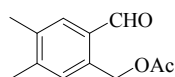


2-formyl-3,5-dimethylbenzyl acetate **2e**. Yield: 37% (15.2 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.53 (s, 1H), 7.14 (s, 1H), 7.05 (s, 1H), 5.45 (s, 2H), 2.63 (s, 3H), 2.38 (s, 3H), 2.13 (s, 3H). <sup>13</sup>C NMR (150 MHz, CDCl<sub>3</sub>) δ 192.2, 170.6, 144.3, 142.1, 138.5, 132.5, 129.3, 127.6, 64.4, 21.7, 21.0, 20.1.

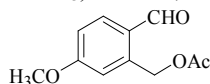


(2-formyl-5-methyl-1,3-phenylene)bis(methylene) diacetate **2e'**. Yield: 21% (11.1 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.47 (s, 1H), 7.30 (s, 2H), 5.46 (s, 4H), 2.44 (s, 3H), 2.14 (s, 6H); <sup>13</sup>C NMR (151

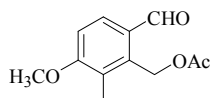
MHz, CDCl<sub>3</sub>)  $\delta$  191.4, 170.5(2C), 144.6, 139.1(2C), 130.1(2C), 129.2, 63.9(2C), 21.9, 21.0(2C).



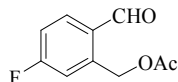
2-formyl-4,5-dimethylbenzyl acetate **2f**. Yield: 64% (26.4 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$  10.13 (s, 1H), 7.63 (s, 1H), 5.48 (s, 2H), 2.35 (s, 3H), 2.34 (s, 3H), 2.12 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  192.1, 170.7, 143.7, 137.2, 135.1, 134.3, 131.7, 130.7, 63.3, 21.0, 20.2, 19.9.



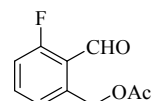
2-formyl-5-methoxybenzyl acetate **2g**.<sup>2</sup> Yield: 49% (20.4 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$  10.02 (s, 1H), 7.83 (d, *J* = 8.5 Hz, 1H), 7.03 (d, *J* = 2.2 Hz, 1H), 6.97 (dd, *J* = 8.5, 2.5 Hz, 1H), 5.55 (s, 2H), 3.91 (s, 3H), 2.17 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  191.0, 170.5, 164.0, 140.5, 136.4, 127.0, 114.5, 112.4, 63.5, 55.6, 20.9.



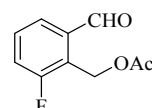
6-formyl-3-methoxy-2-methylbenzyl acetate **2h**. Yield: 52% (23.0 mg); <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  10.13 (s, 1H), 7.76 (d, *J* = 8.6 Hz, 1H), 6.98 (d, *J* = 8.6 Hz, 1H), 5.58 (s, 2H), 3.92 (s, 3H), 2.28 (s, 3H), 2.06 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  191.1, 170.8, 162.1, 136.3, 132.6, 128.5, 128.3, 109.9, 77.3, 77.0, 76.82, 58.8, 55.8, 20.8, 11.2.



5-fluoro-2-formylbenzyl acetate **2i**.<sup>1</sup> Yield: 67% (26.3 mg); <sup>1</sup>H NMR (600 MHz, Chloroform-*d*)  $\delta$  10.11 (s, 1H), 7.95-7.84 (m, 1H), 7.27-7.23 (m, 1H), 7.21-7.17 (m, 1H), 5.57 (s, 2H), 2.19 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  190.9, 170.3, 166.0(d, *J* = 257.3 Hz, 1C), 141.82 (d, *J* = 9.0 Hz, 1H), 136.47 (d, *J* = 10.0 Hz, 3H), 129.84 (d, *J* = 2.8 Hz, 1H), 115.4 (d, *J* = 23.8 Hz, 1C), 115.1 (d, *J* = 21.9 Hz, 1C), 63.0, 20.9; MS (EI) *m/z* (%) 153.0(M-Ac, 55), 135.9(100), 108.0(91), 77.0(31); Anal. Calcd. For C<sub>10</sub>H<sub>9</sub>FO<sub>3</sub>: C, 61.22; H, 4.62; F, 9.68%. Found: C, 61.24; H, 4.60; F, 9.61%.

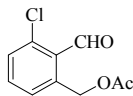


3-fluoro-2-formylbenzyl acetate **2j**.<sup>3</sup> Yield: 66% (26.0 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$  10.52 (s, 1H), 7.63-7.55 (m, 1H), 7.32 (d, *J* = 7.8 Hz, 1H), 7.21-7.07 (m, 1H), 5.52 (s, 2H), 2.17 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  188.53 (d, *J* = 11.0 Hz, 1C), 170.45, 166.17 (d, *J* = 258.3 Hz, 1C), 140.08, 135.69 (d, *J* = 10.4 Hz, 1C), 123.19 (d, *J* = 3.4 Hz, 1C), 121.43 (d, *J* = 6.4 Hz, 1C), 115.74 (d, *J* = 21.6 Hz, 1C), 63.71, 63.69, 20.89; MS (EI) *m/z* (%) 153.0(M-Ac, 86), 136.0(100), 108.0(93), 95.0(45), 75.0(55); Anal. Calcd. For C<sub>10</sub>H<sub>9</sub>FO<sub>3</sub>: C, 61.22; H, 4.62; F, 9.68%. Found: C, 61.21; H, 4.64; F, 9.67%.

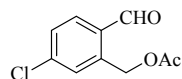


2-fluoro-6-formylbenzyl acetate **2k**. Yield: 51% (20.0 mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>)  $\delta$  10.27 (s, 1H), 7.72 (d, *J* = 7.6 Hz, 1H), 7.56 – 7.51 (m, 1H), 7.39 – 7.32 (m, 1H), 5.54 (d, *J* = 1.1 Hz, 2H), 2.07 (s, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>)  $\delta$  190.64 (d, *J* = 2.4 Hz, 1C), 170.5, 161.57 (d, *J* = 251.4 Hz, 1C), 136.38 (d, *J* = 2.4 Hz, 1C), 130.73 (d, *J* = 8.8 Hz, 1C), 127.31 (d, *J* = 3.2 Hz, 1C), 123.80 (d, *J* = 14.8

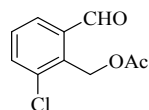
Hz, 1C), 121.14 (d,  $J = 23.4$  Hz, 1C), 55.07 (d,  $J = 6.1$  Hz, 1C), 20.8; MS (EI)  $m/z$  (%) 153.0 (M-Ac, 53), 136.0 (100), 108.0 (88), 83.0 (31), 71.0 (36); Anal. Calcd. For  $C_{10}H_9FO_3$ : C, 61.22; H, 4.62; F, 9.68%. Found: C, 61.20; H, 4.65; F, 9.70%.



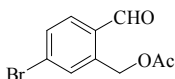
3-chloro-2-formylbenzyl acetate **2l**. Yield: 53% (22.5 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.64 (s, 1H), 7.59-7.49 (m, 1H), 7.48-7.38 (m, 2H), 5.49 (s, 2H), 2.16 (s, 3H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  191.9, 170.4, 140.3, 139.2, 134.2, 130.1, 129.8, 126.4, 63.7, 20.8.



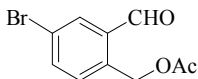
5-chloro-2-formylbenzyl acetate **2m**.<sup>4</sup> Yield: 56% (23.7 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.14 (s, 1H), 7.81 (d,  $J = 8.2$  Hz, 1H), 7.55-7.52 (m, 1H), 7.50 (dd,  $J = 8.2, 1.8$  Hz, 1H), 5.54 (s, 2H), 2.18 (s, 3H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  191.2, 170.3, 140.6, 139.9, 134.7, 131.7, 128.5, 128.4, 62.8, 20.9.



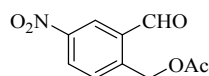
2-chloro-6-formylbenzyl acetate **2n**. Yield: 34% (14.4 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.28 (s, 1H), 7.83 (d,  $J = 7.6$  Hz, 1H), 7.67 (d,  $J = 7.9$  Hz, 1H), 7.50 (t,  $J = 7.9$  Hz, 1H), 5.65 (s, 2H), 2.08 (s, 3H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  190.6, 170.5, 137.0, 136.7, 134.9, 134.3, 130.2, 129.8, 58.6, 20.7.



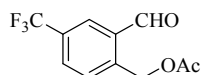
5-bromo-2-formylbenzyl acetate **2o**. Yield: 47% (24.1 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.13 (s, 1H), 7.73 (d,  $J = 8.1$  Hz, 1H), 7.70 (s, 1H), 7.67 (dd,  $J = 8.1, 1.5$  Hz, 1H), 5.53 (s, 2H), 2.17 (s, 3H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  191.4, 170.4, 139.8, 134.6, 132.1, 131.6, 131.4, 129.4, 62.7, 20.9.



4-bromo-2-formylbenzyl acetate **2p**. Yield: 37% (19 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.15 (s, 1H), 7.99 (d,  $J = 2.0$  Hz, 1H), 7.72 (dd,  $J = 8.2, 2.0$  Hz, 1H), 7.41 (d,  $J = 8.2$  Hz, 1H), 5.48 (s, 2H), 2.12 (s, 3H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  190.6, 170.3, 136.8, 135.4, 135.3, 135.2, 130.7, 122.6, 62.8, 20.81.

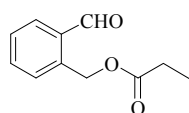


2-formyl-4-nitrobenzyl acetate **2q**. Yield: 43% (19.1 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.26 (s, 1H), 8.73 (d,  $J = 2.2$  Hz, 1H), 8.45 (dd,  $J = 8.5, 2.2$  Hz, 1H), 7.78 (d,  $J = 8.5$  Hz, 1H), 5.64 (s, 2H), 2.20 (s, 3H), -0.00 (s, 1H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  190.2, 170.2, 147.8, 144.8, 134.1, 129.2, 128.0, 127.8, 62.9, 20.8.

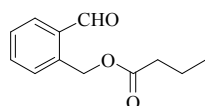


2-formyl-4-(trifluoromethyl)benzyl acetate **2r**. Yield: 31% (15.0 mg);  $^1H$  NMR (600 MHz,  $CDCl_3$ )  $\delta$  10.34 (s, 1H), 8.13 (d,  $J = 7.7$  Hz, 1H), 7.96 (d,  $J = 7.8$  Hz, 1H), 7.68 (t,  $J = 7.8$  Hz, 1H), 5.59 (s, 2H), 2.08 (s, 3H);  $^{13}C$  NMR (151 MHz,  $CDCl_3$ )  $\delta$  190.06, 170.12, 136.59, 135.48, 133.91, 131.21, 130.93 (q,  $J = 5.6$  Hz, 1C), 129.48, 123.54 (q,  $J = 274.5$  Hz, 1C), 57.99 (q,  $J = 2.5$  Hz, 1C), 20.63; MS (EI)  $m/z$  (%) 203.0 (M-Ac, 58), 186.0 (100), 158.0 (93), 138.0 (43), 109.0 (37), 89.0 (17), 75.0 (22); Anal. Calcd. For

C<sub>11</sub>H<sub>9</sub>F<sub>3</sub>O<sub>3</sub>: C, 53.67; H, 3.68; F, 23.15%. Found: C, 53.59; H, 3.60; F, 23.21%.



propionic acid 2-formyl-benzyl ester **2s**. Yield:25% (9.7mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.21 (s, 1H), 7.92-7.84 (m, 1H), 7.67-7.56 (m, 1H), 7.56-7.46 (m, 2H), 5.56 (s, 2H), 2.43 (q, *J* = 7.6 Hz, 2H), 1.19 (t, *J* = 7.6 Hz, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 192.4, 173.9, 138.1, 133.9, 133.6, 133.1, 128.6, 128.4, 63.4, 27.6, 9.1.



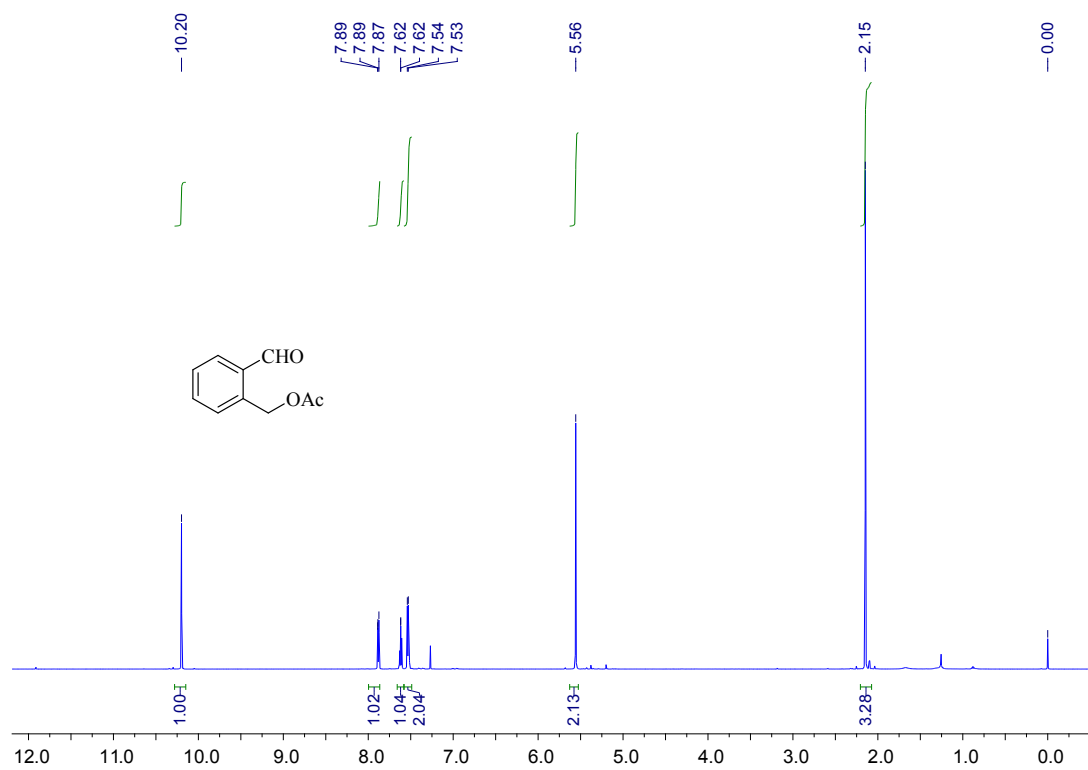
butyric acid 2-formyl-benzyl ester **2t**. Yield:21% (8.5mg); <sup>1</sup>H NMR (600 MHz, CDCl<sub>3</sub>) δ 10.21 (s, 1H), 7.91-7.82 (m, 1H), 7.66-7.56 (m, 1H), 7.56-7.43 (m, 2H), 5.56 (s, 2H), 2.38 (t, *J* = 7.4 Hz, 2H), 1.75-1.65 (m, 2H), 0.96 (t, *J* = 7.4 Hz, 3H); <sup>13</sup>C NMR (151 MHz, CDCl<sub>3</sub>) δ 192.4, 173.1, 138.1, 133.9, 133.6, 133.1, 128.6, 128.4, 63.3, 36.2, 18.5, 13.7.

#### References:

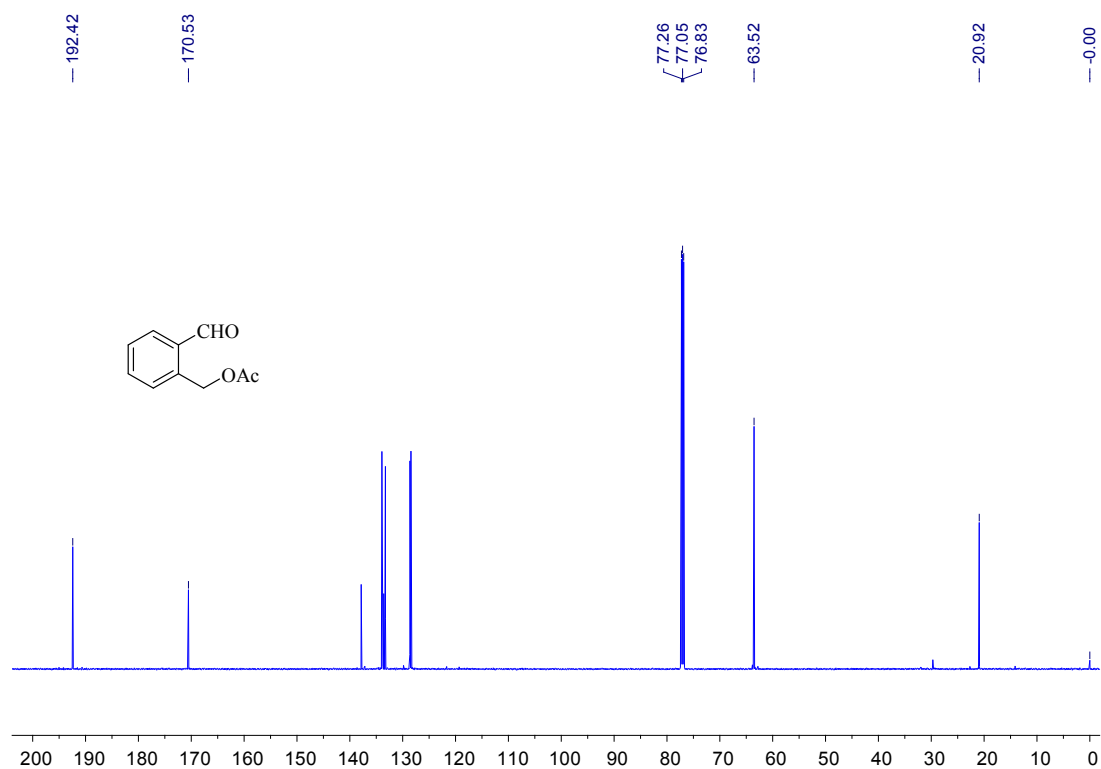
- [1] J. W. Clark-Lewis, M. M. Mahandru *Australian Journal of Chemistry* 1974, 27, 2689-2692.
- [2] CAS Registry Number: 904983-36-4.
- [3] CAS Registry Number: 1824455-82-4.
- [4] CAS Registry Number: 1824624-43-2.

#### 4. $^1\text{H}$ NMR, $^{13}\text{C}$ NMR and MS Spectra of the Products

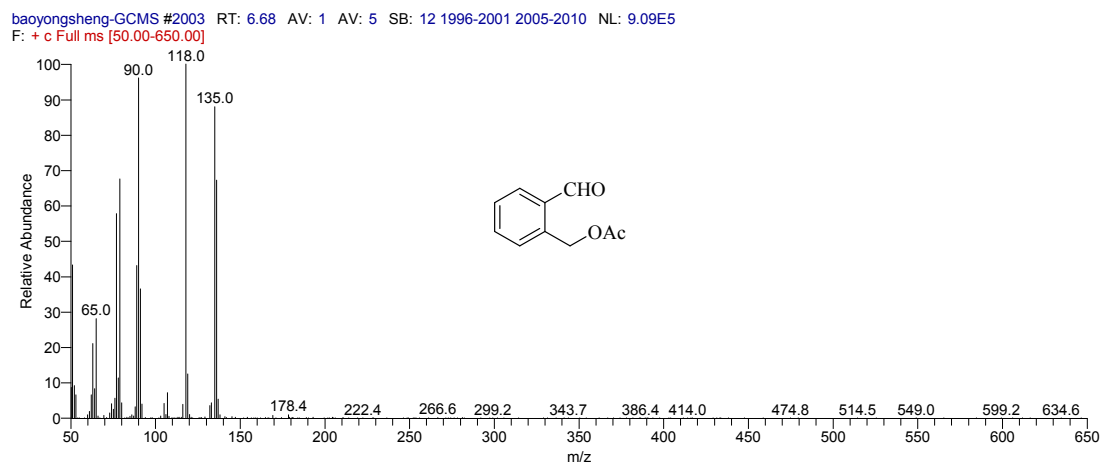
$^1\text{H}$  NMR of 2-formylbenzyl acetate **2a**



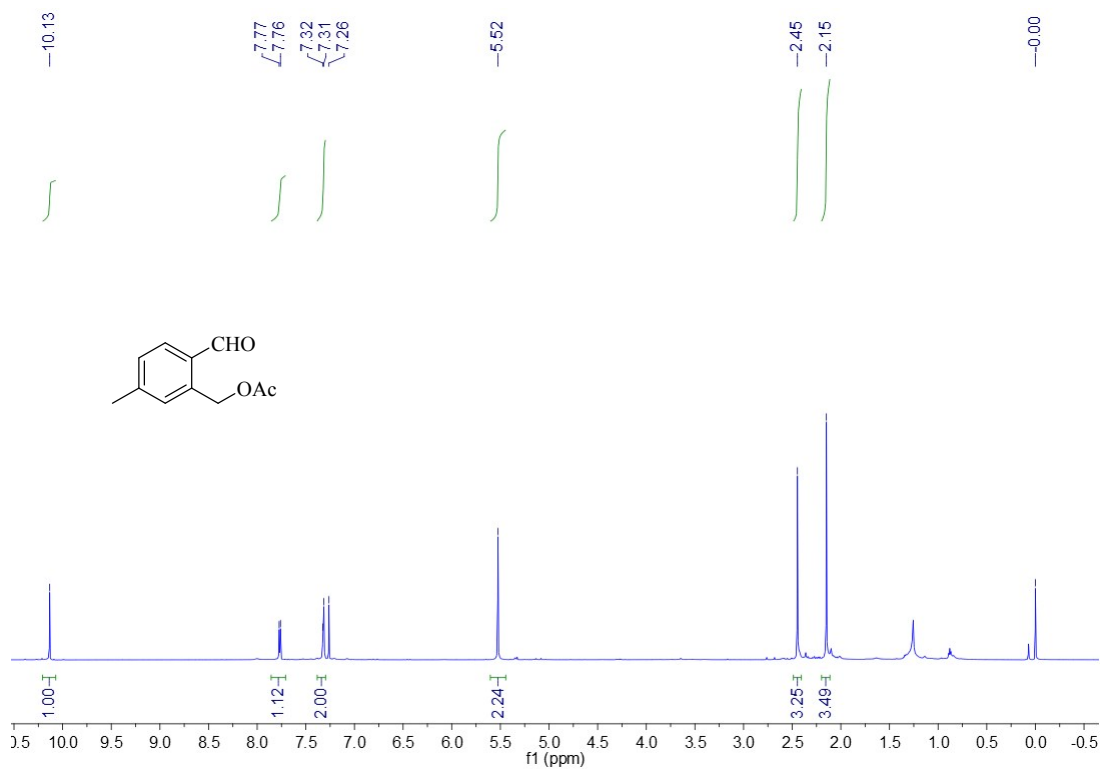
$^{13}\text{C}$  NMR of 2-formylbenzyl acetate **2a**



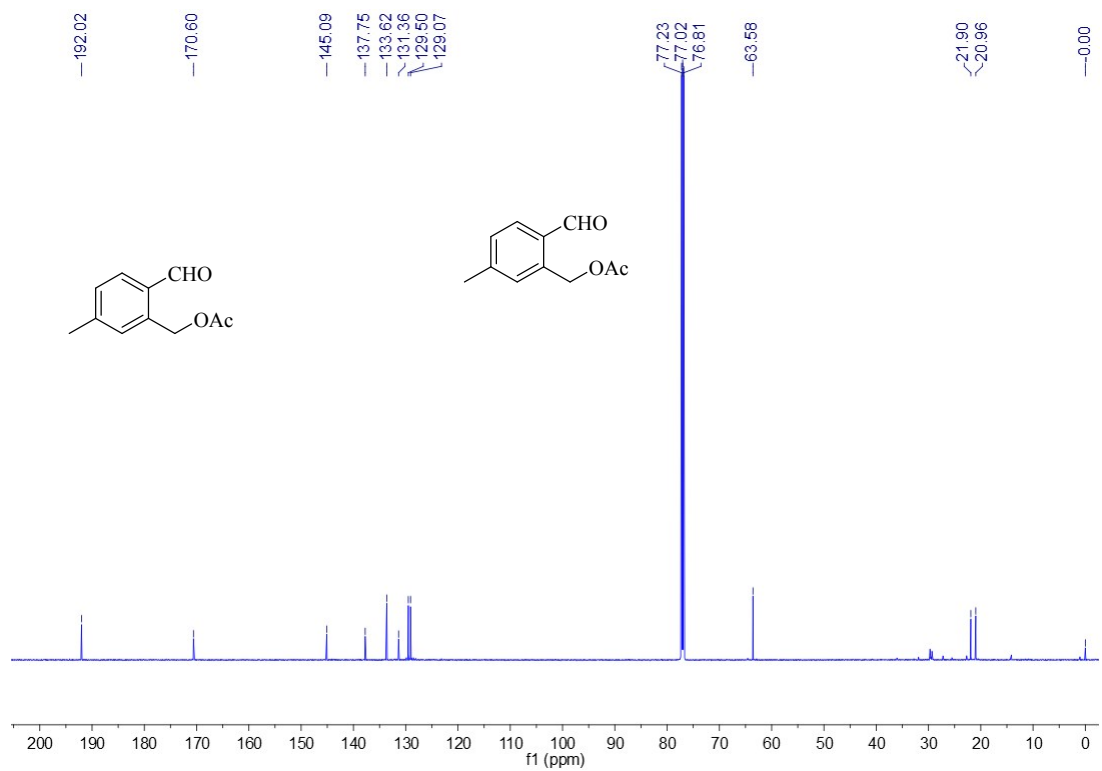
MS(EI) of 2-formylbenzyl acetate **2a**



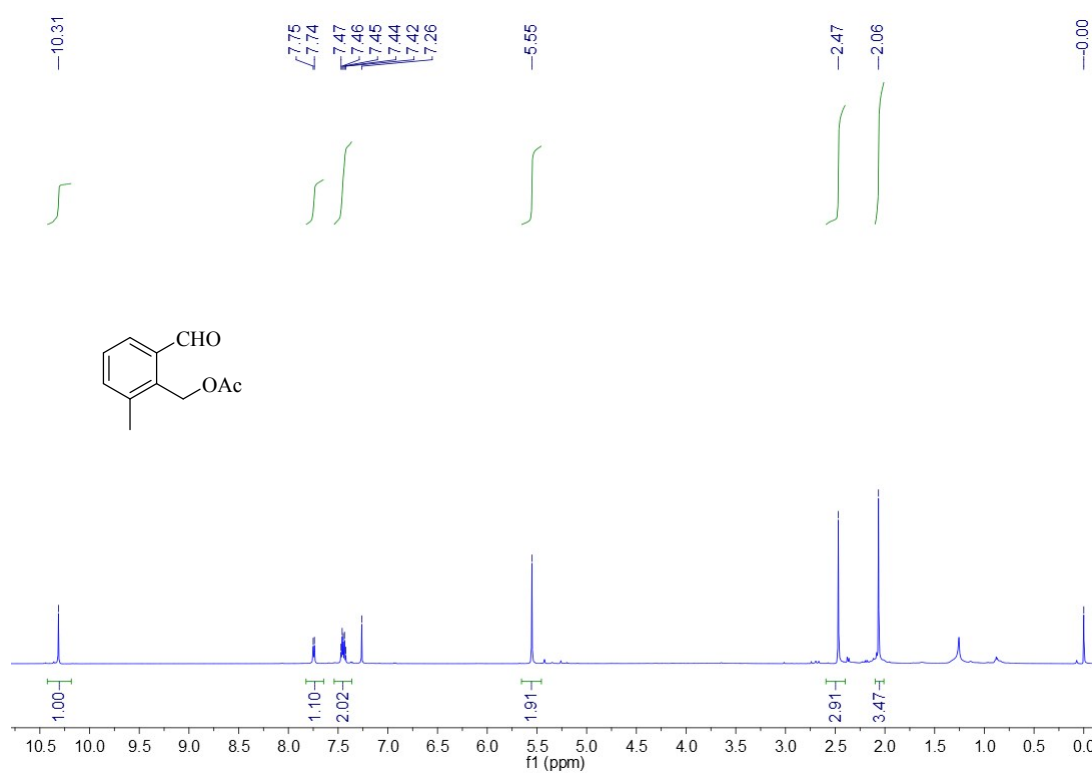
<sup>1</sup>H NMR of 2-formyl-5-methylbenzyl acetate **2b**



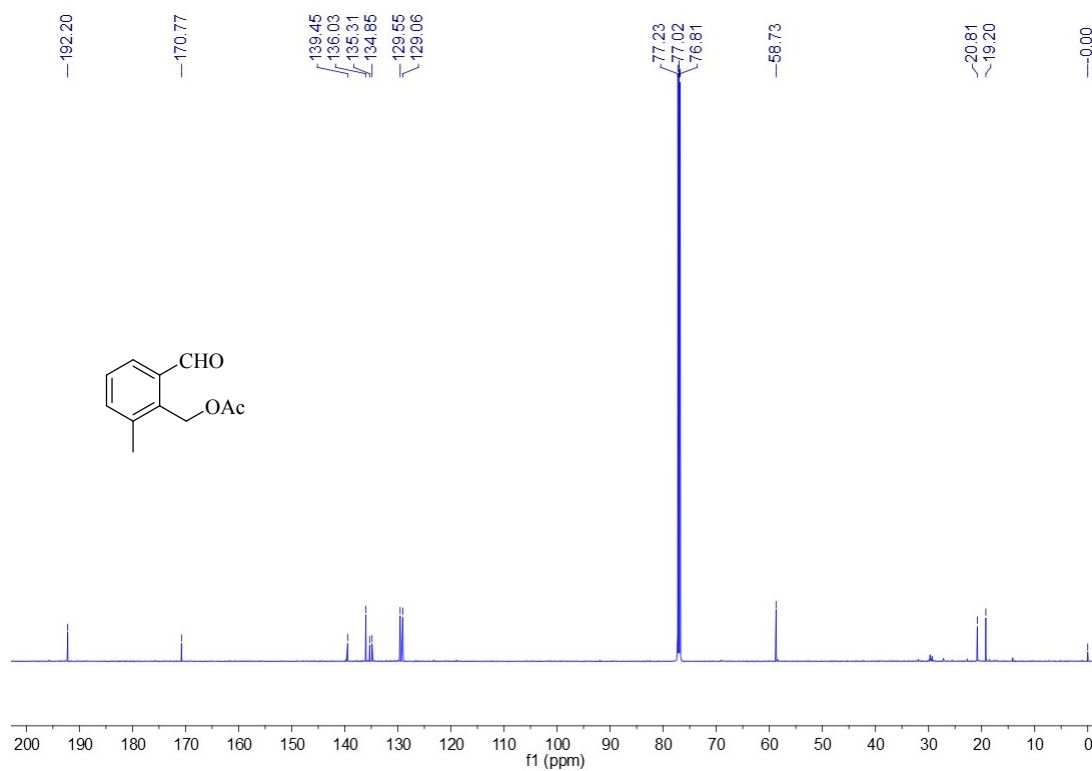
**<sup>13</sup>C NMR of 2-formyl-5-methylbenzyl acetate **2b****



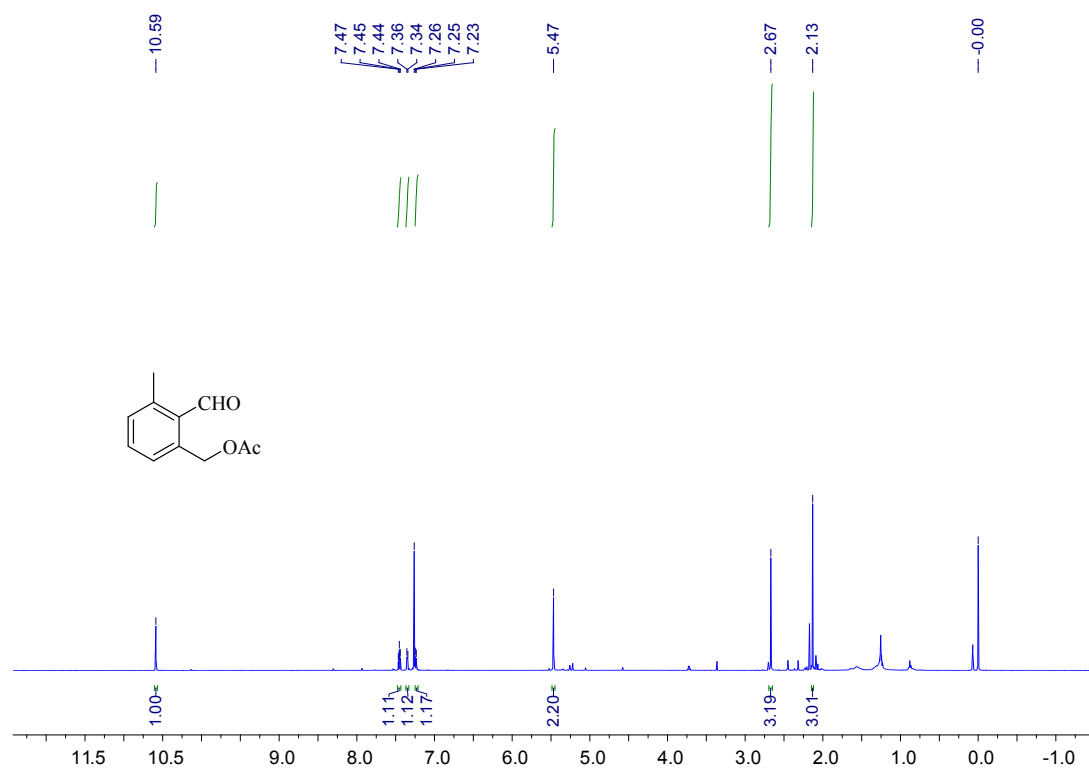
**<sup>1</sup>H NMR of 2-formyl-6-methylbenzyl acetate **2c****



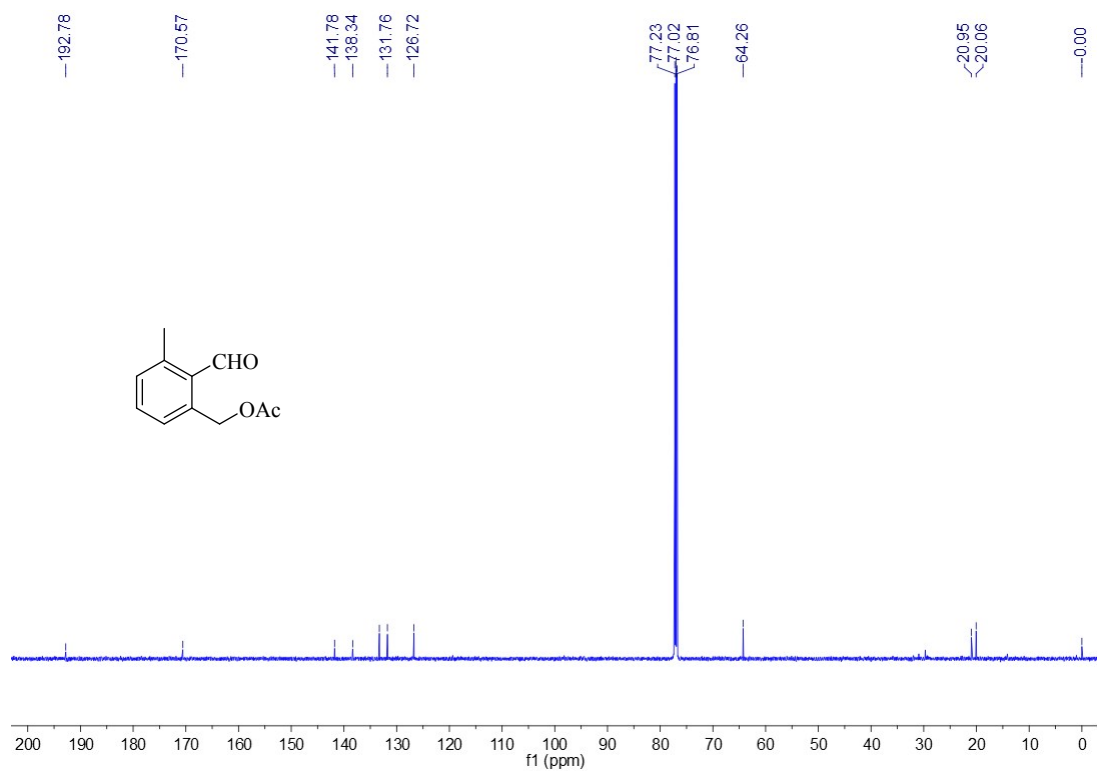
**<sup>13</sup>C NMR of 2-formyl-6-methylbenzyl acetate **2c****



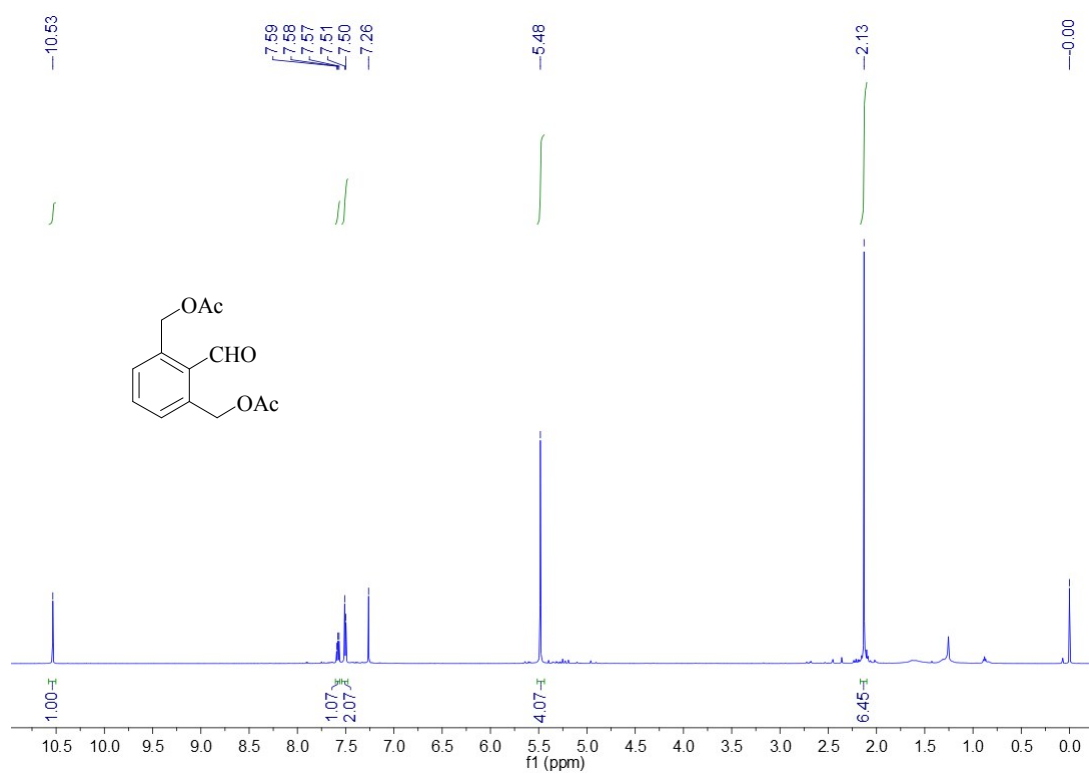
**<sup>1</sup>H NMR of 2-formyl-3-methylbenzyl acetate **2d****



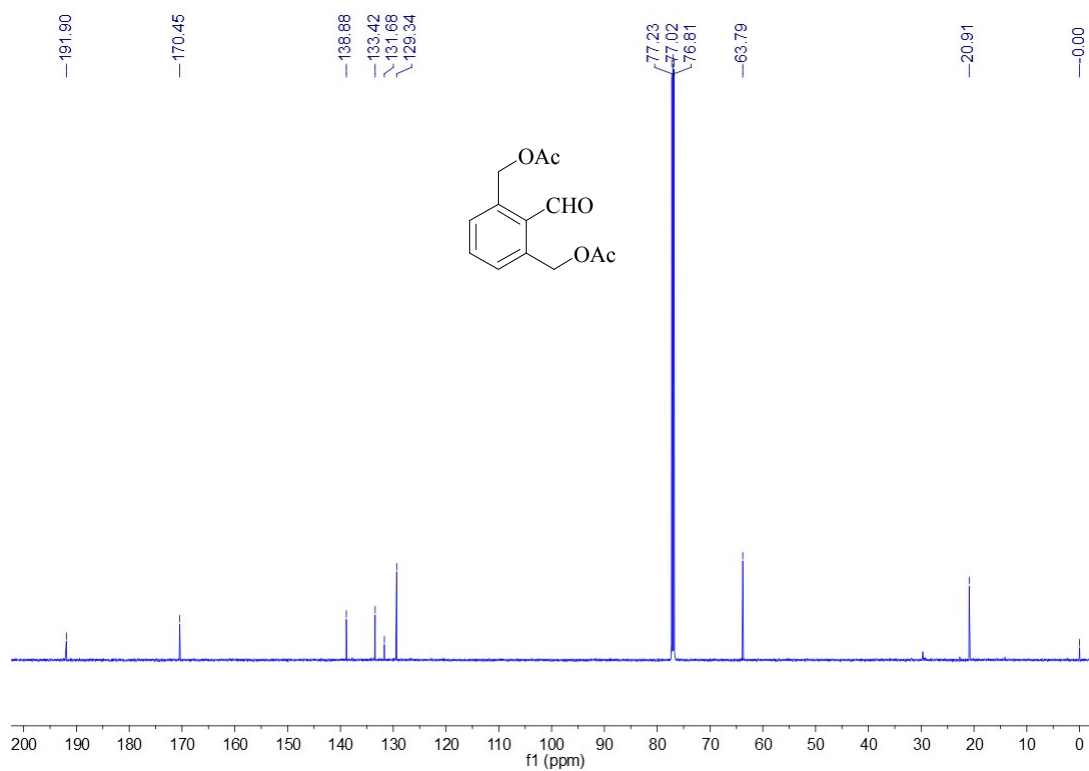
**<sup>13</sup>C NMR of 2-formyl-3-methylbenzyl acetate **2d****



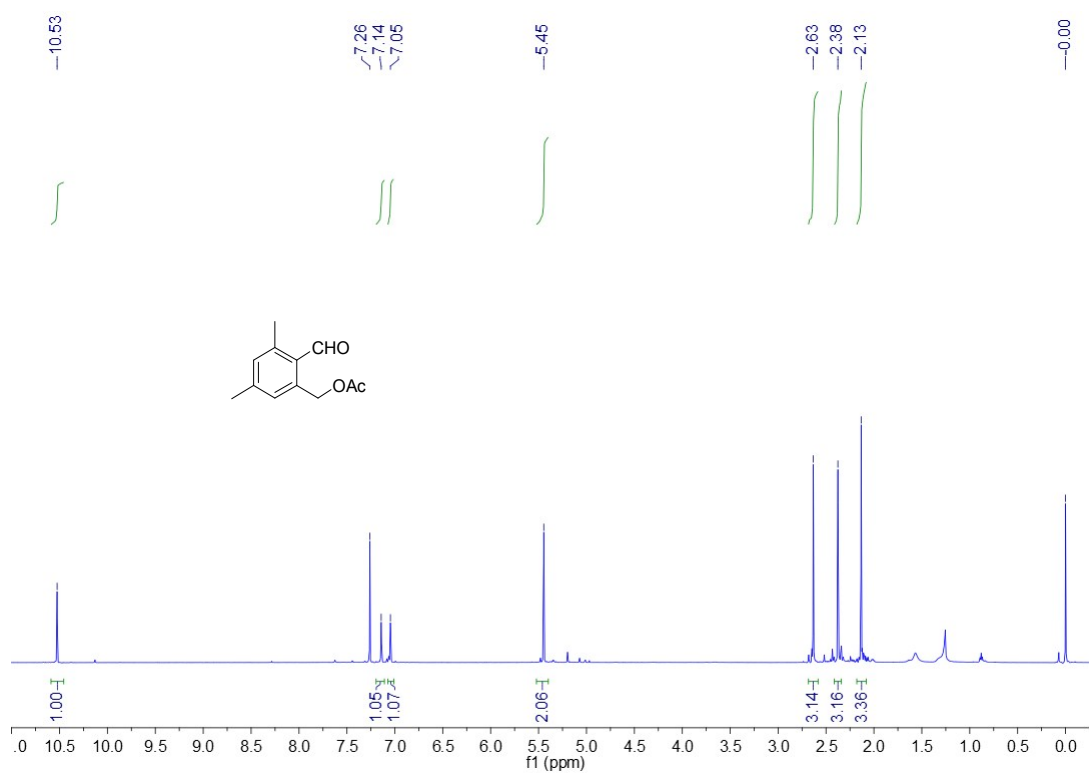
**<sup>1</sup>H NMR of (2-formyl-1,3-phenylene)bis(methylene) diacetate **2d'****



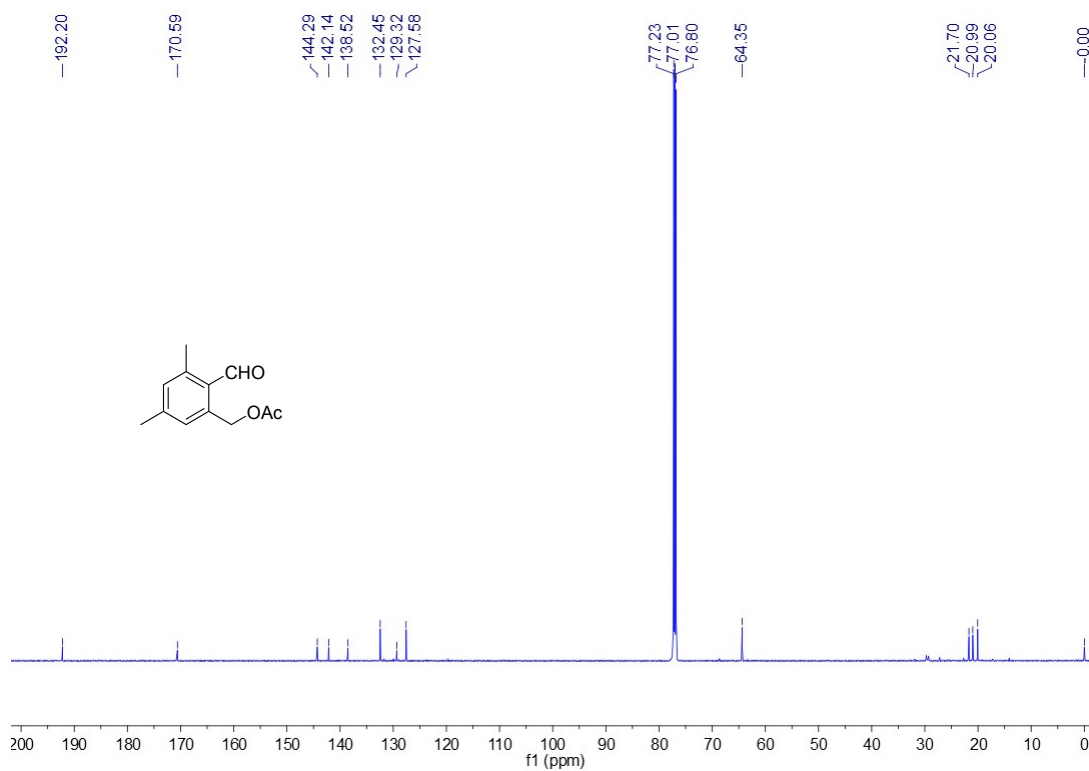
<sup>13</sup>C NMR of (2-formyl-1,3-phenylene)bis(methylene) diacetate **2d'**



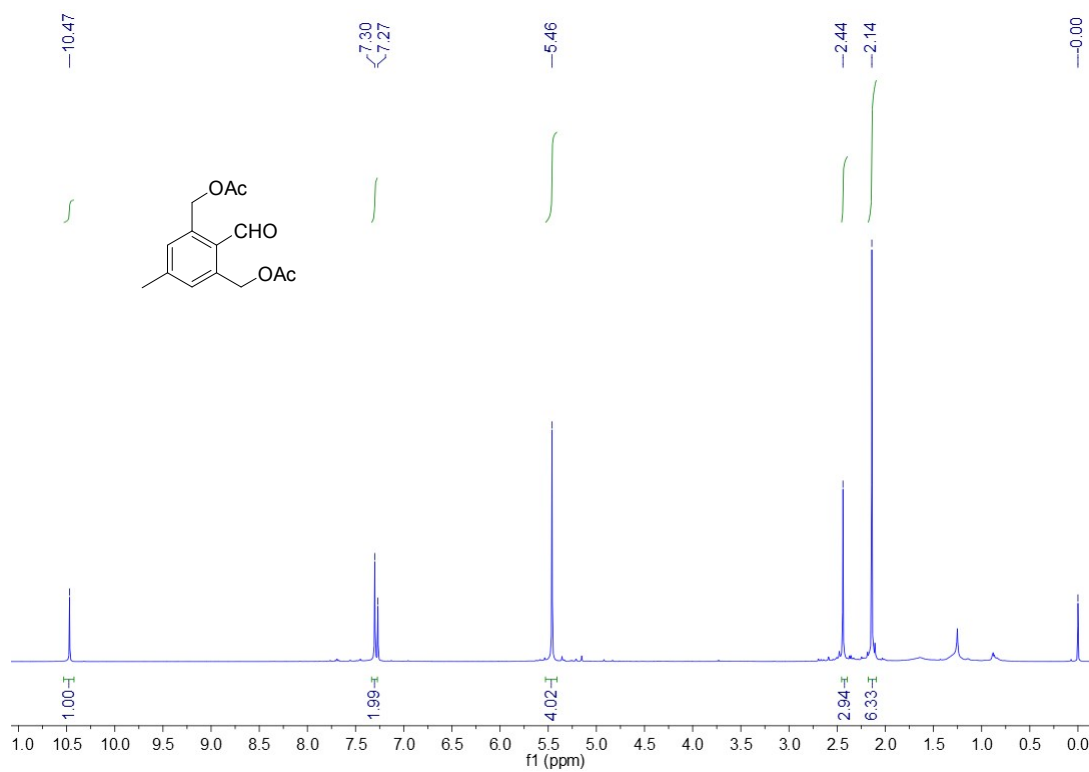
<sup>1</sup>H NMR of 2-formyl-3,5-dimethylbenzyl acetate **2e**



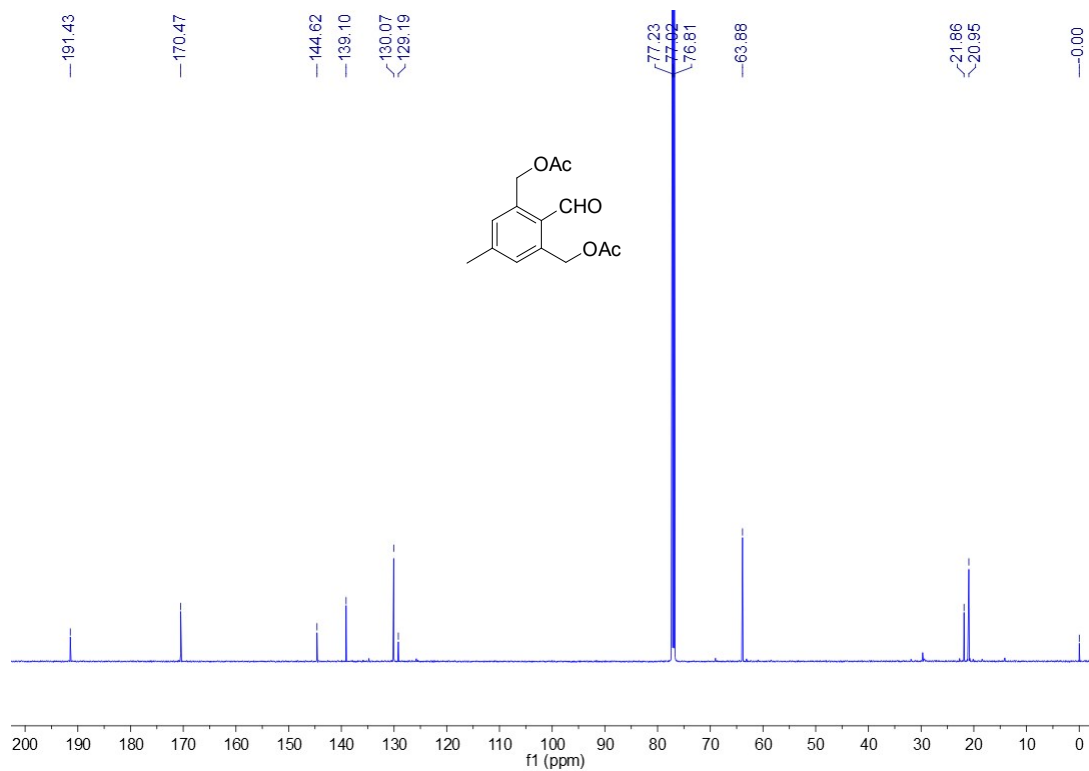
**<sup>13</sup>C NMR of 2-formyl-3,5-dimethylbenzyl acetate **2e****



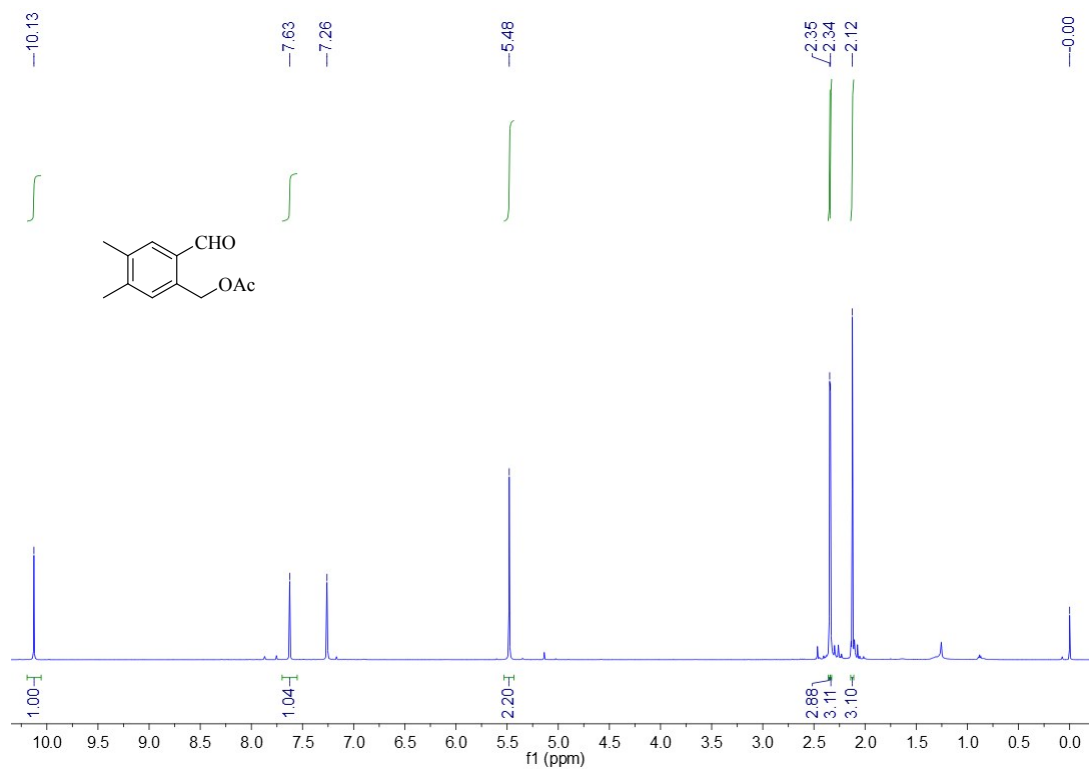
<sup>1</sup>H NMR of (2-formyl-5-methyl-1,3-phenylene)bis(methylene) diacetate **2e'**



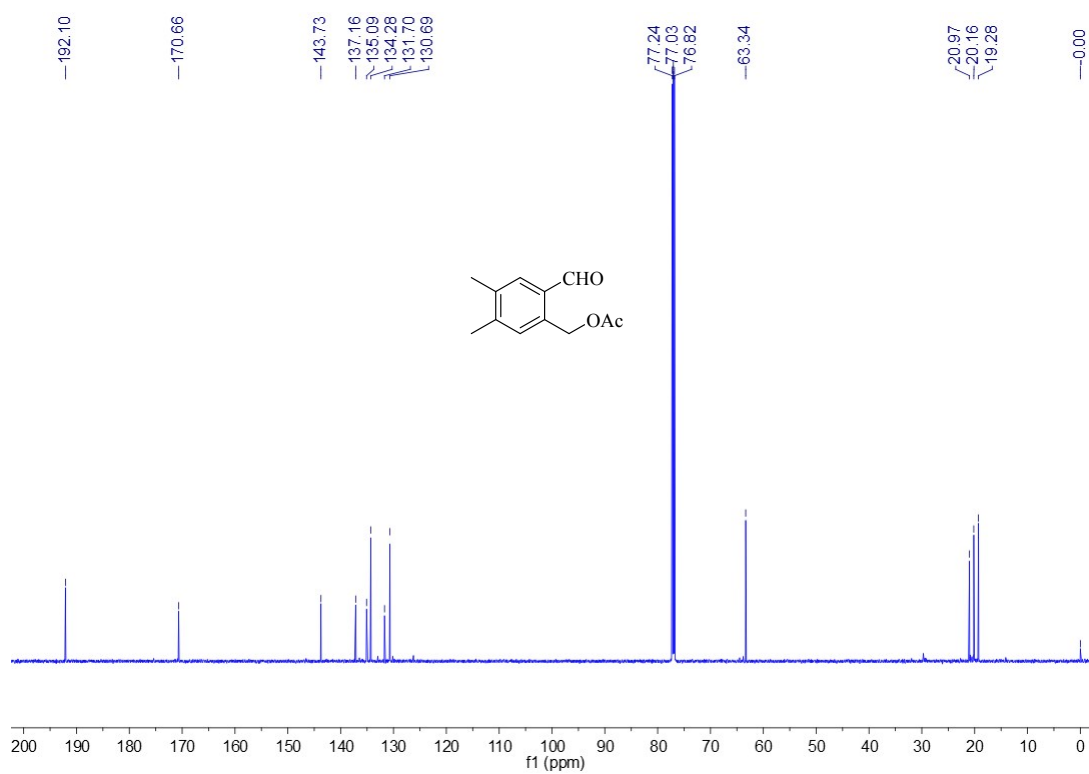
<sup>13</sup>C NMR of (2-formyl-5-methyl-1,3-phenylene)bis(methylene) diacetate **2e'**



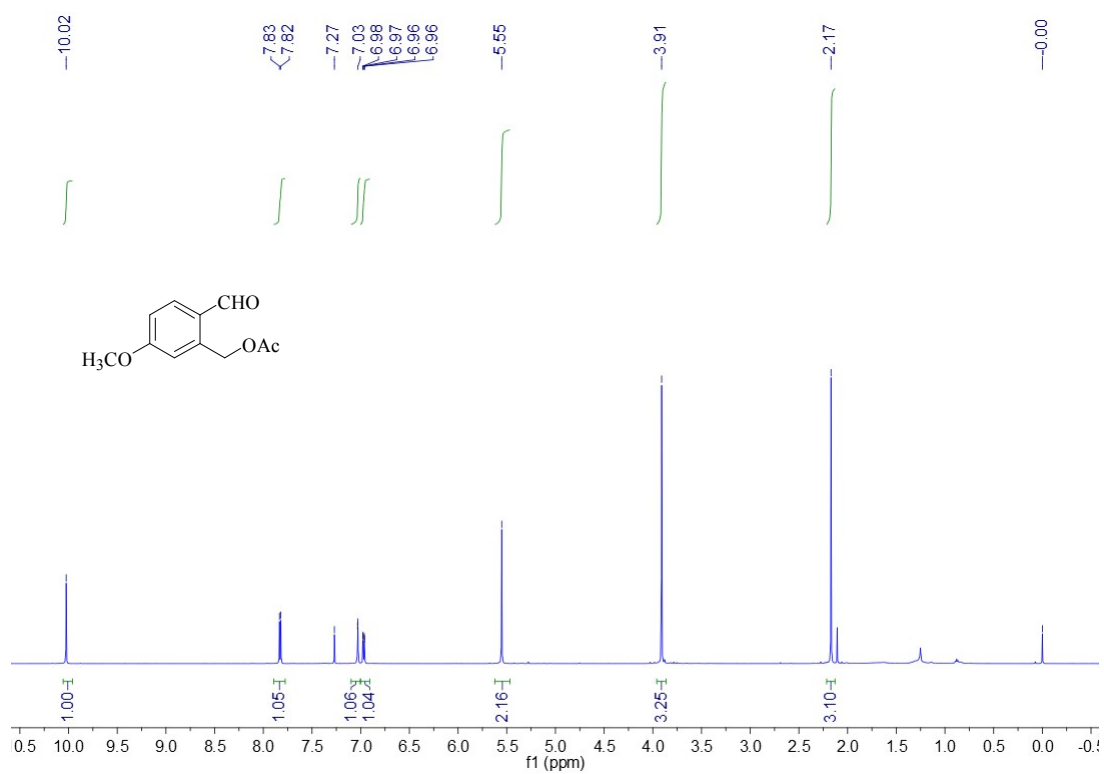
<sup>1</sup>H NMR of 2-formyl-4,5-dimethylbenzyl acetate **2f**



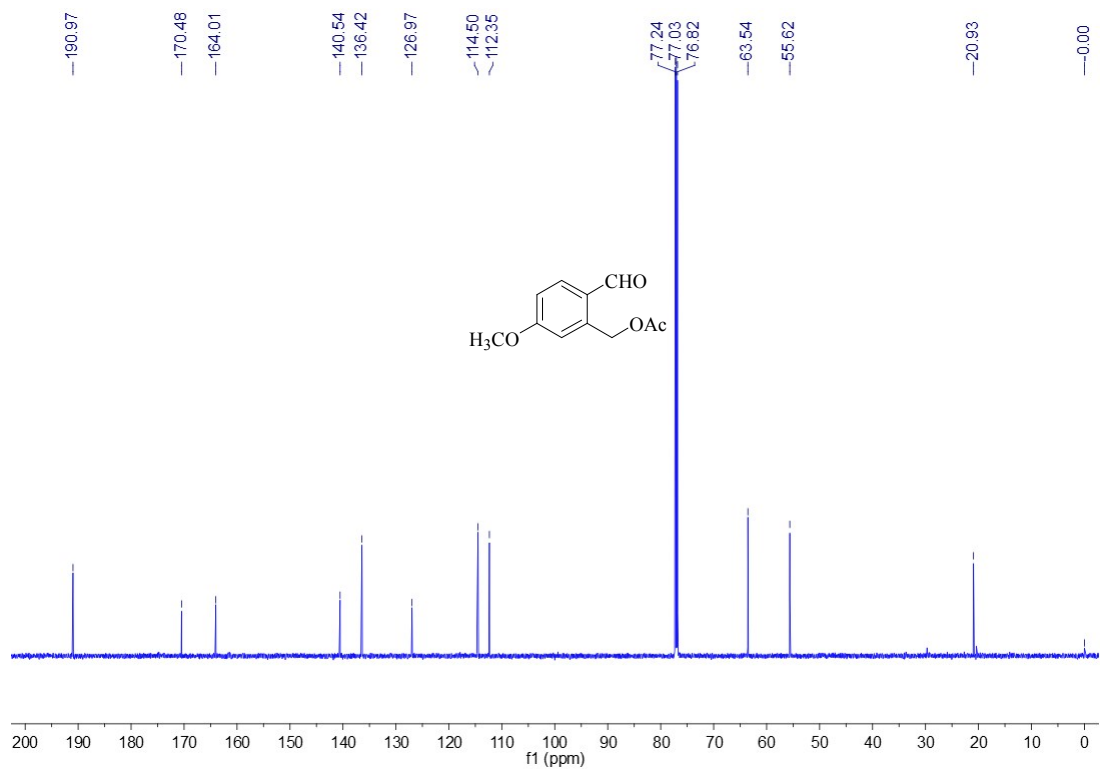
<sup>13</sup>C NMR of 2-formyl-4,5-dimethylbenzyl acetate **2f**



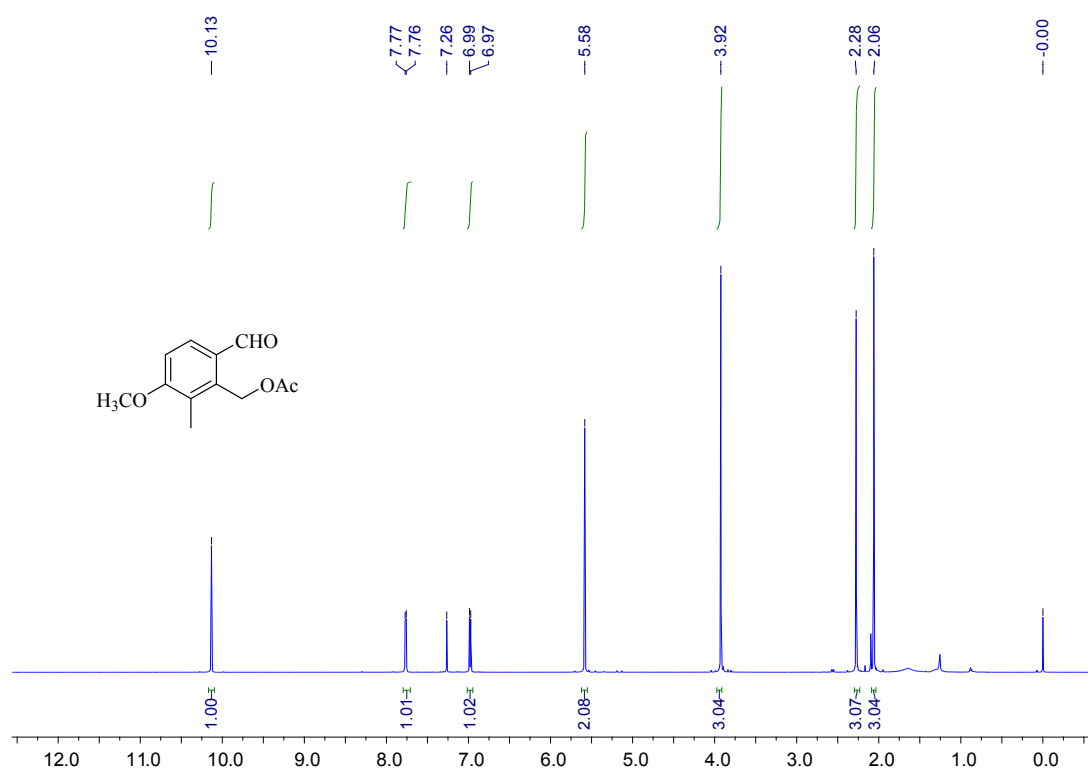
<sup>1</sup>H NMR of 2-formyl-5-methoxybenzyl acetate **2g**



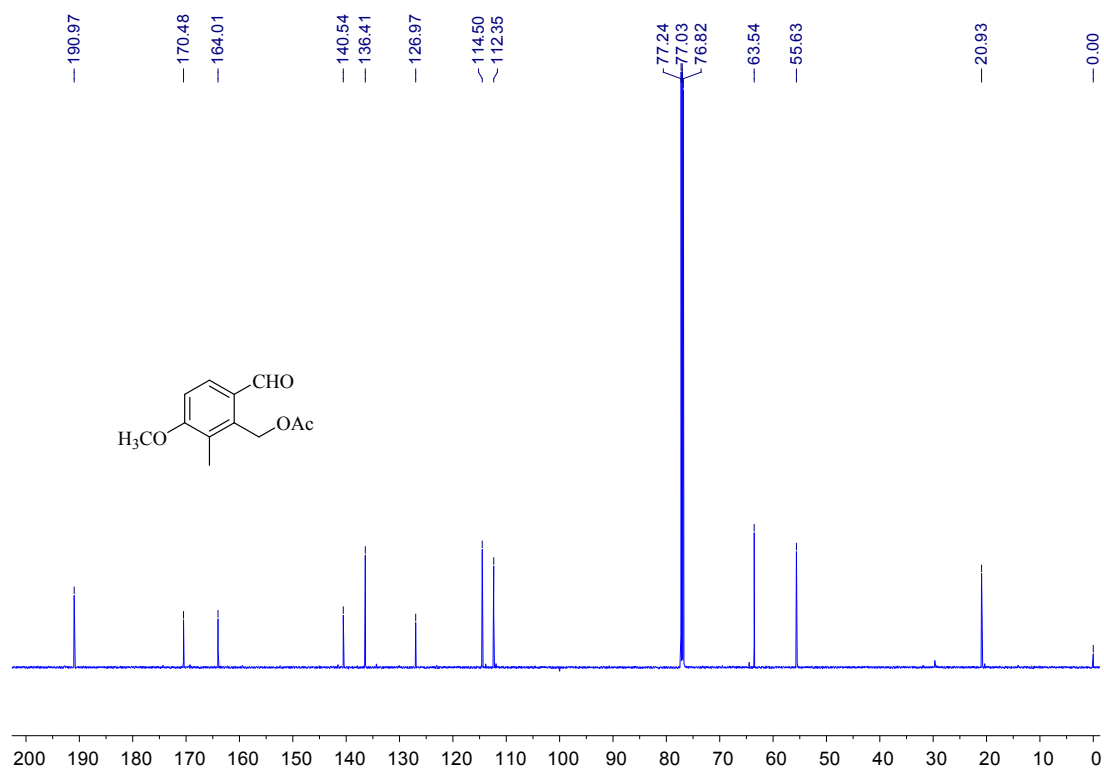
<sup>13</sup>C NMR of 2-formyl-5-methoxybenzyl acetate **2g**



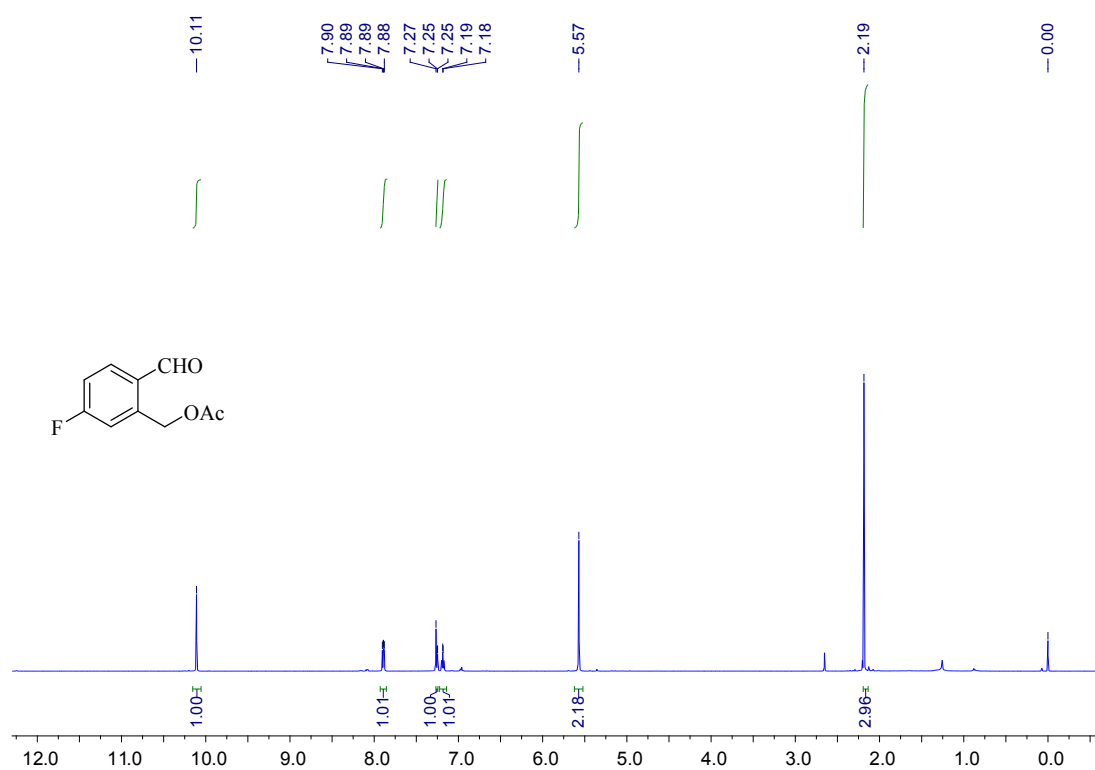
<sup>1</sup>H NMR of 6-formyl-3-methoxy-2-methylbenzyl acetate **2h**



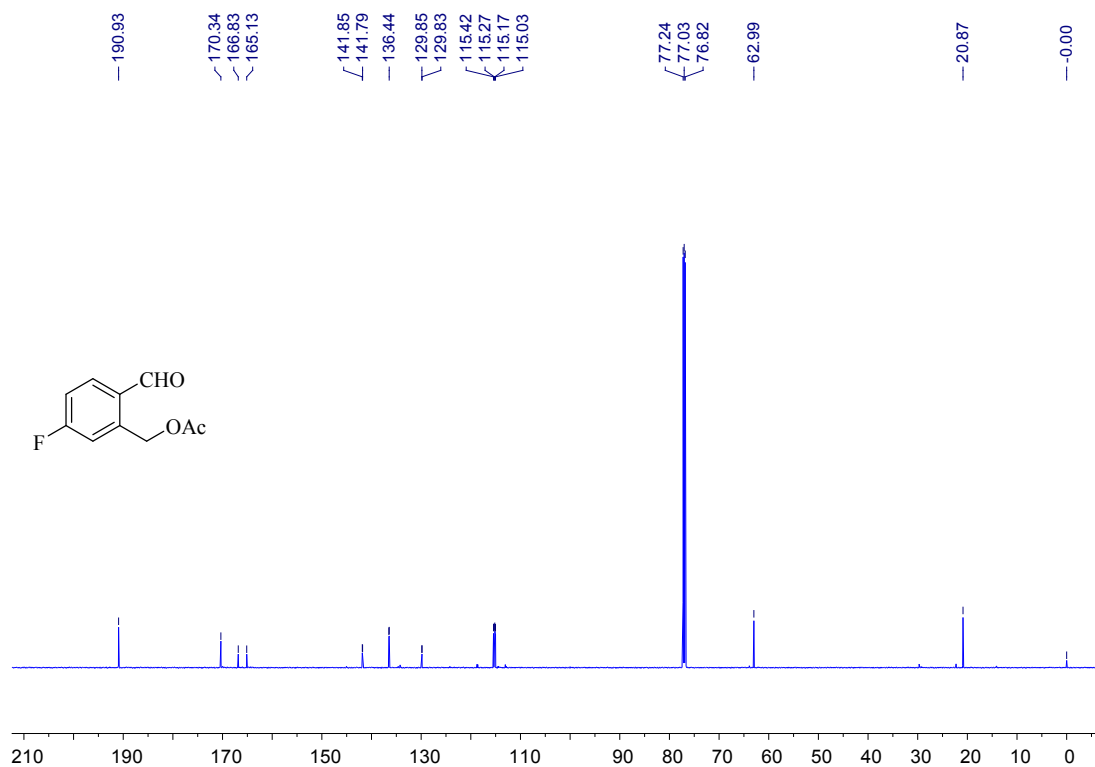
<sup>13</sup>C NMR of 6-formyl-3-methoxy-2-methylbenzyl acetate **2h**



<sup>1</sup>H NMR of 5-fluoro-2-formylbenzyl acetate **2i**



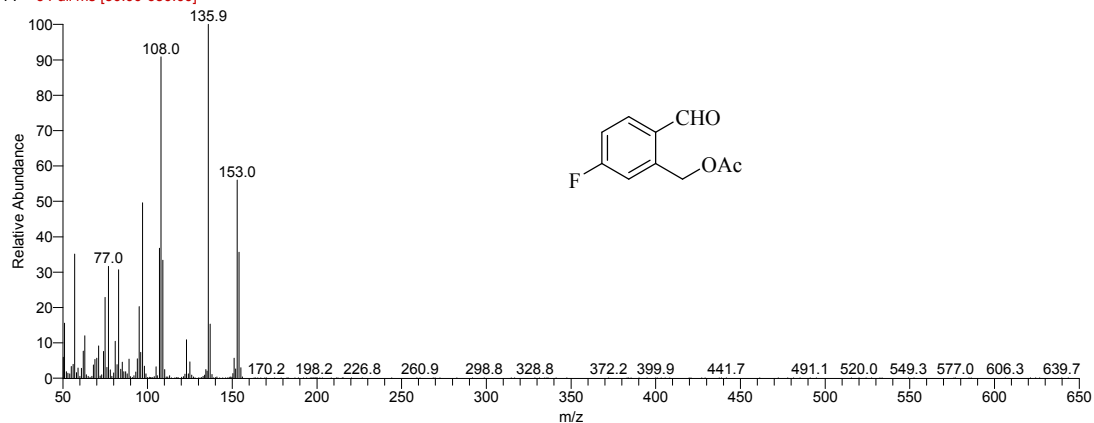
<sup>13</sup>C NMR of 5-fluoro-2-formylbenzyl acetate **2i**



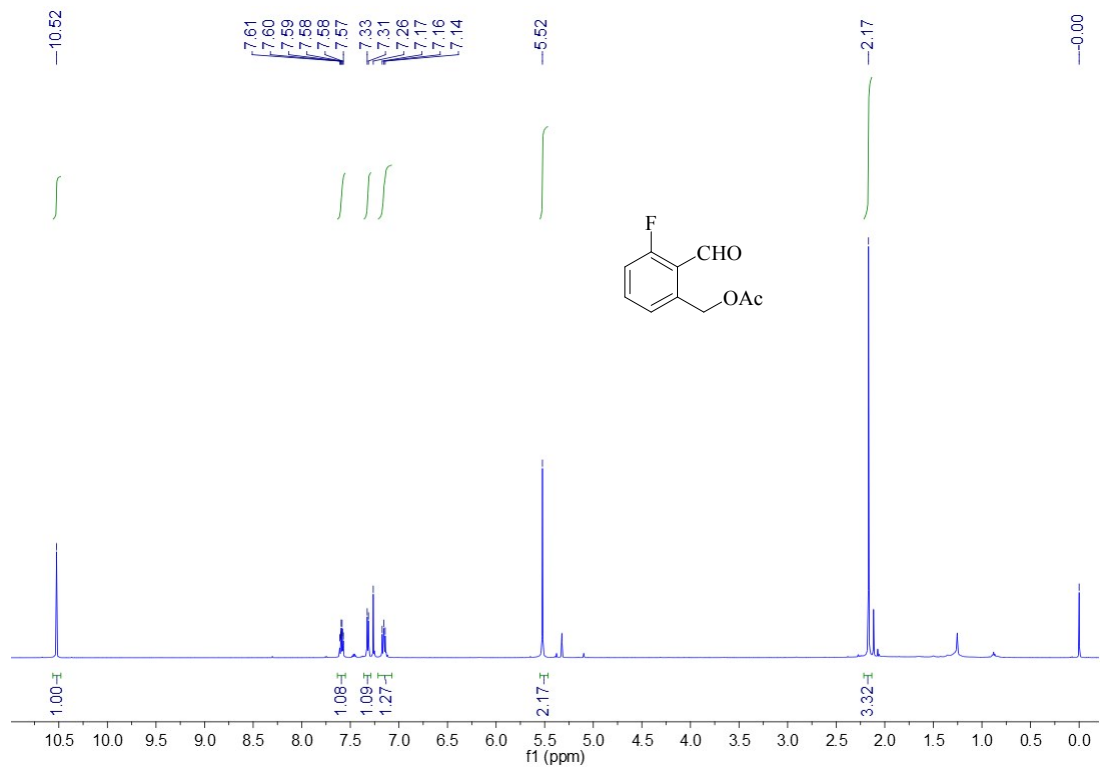
MS(EI) of 5-fluoro-2-formylbenzyl acetate **2i**

baoyongsheng126 #1958 RT: 6.54 AV: 1 AV: 5 SB: 12 1951-1956 1960-1965 NL: 8.10E6

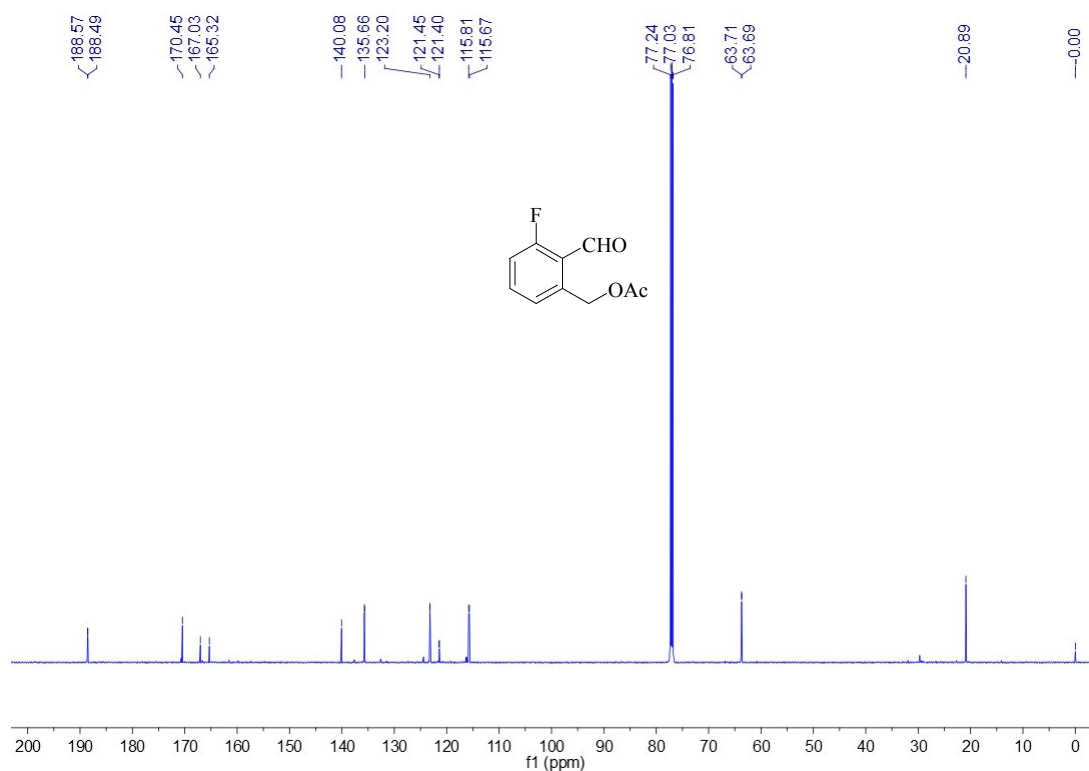
F: + c Full ms [50.00-650.00]



<sup>1</sup>H NMR of 3-fluoro-2-formylbenzyl acetate **2j**



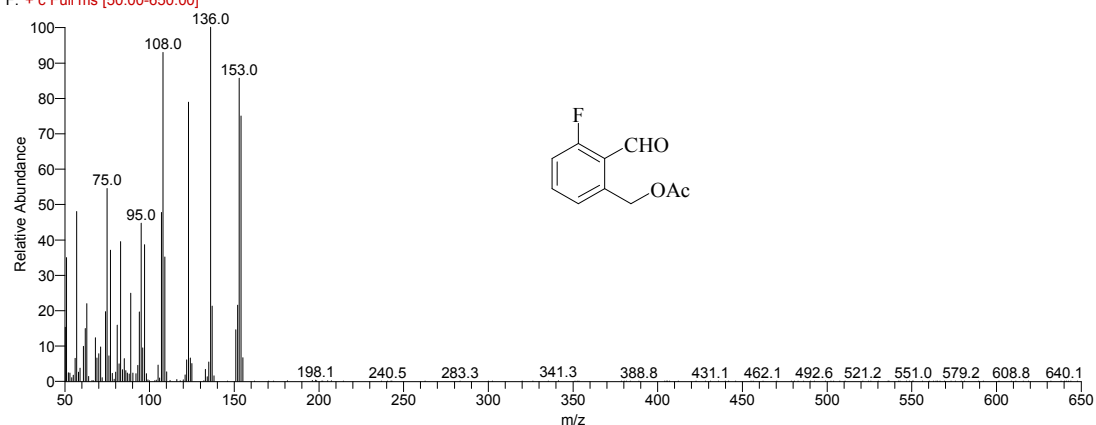
<sup>13</sup>C NMR of 3-fluoro-2-formylbenzyl acetate **2j**



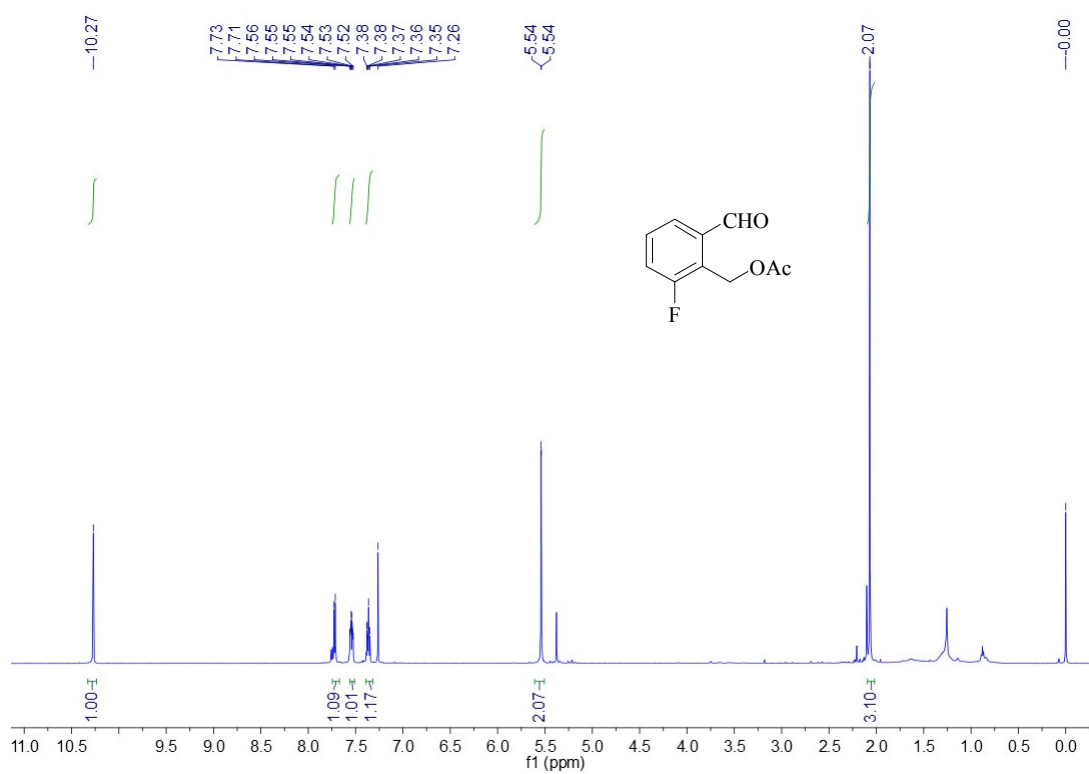
### MS(EI) of 3-fluoro-2-formylbenzyl acetate **2j**

baoyongsheng124\_191206112843 #2064 RT: 6.88 AV: 1 AV: 5 SB: 12 2057-2062 2066-2071 NL: 5.33E6

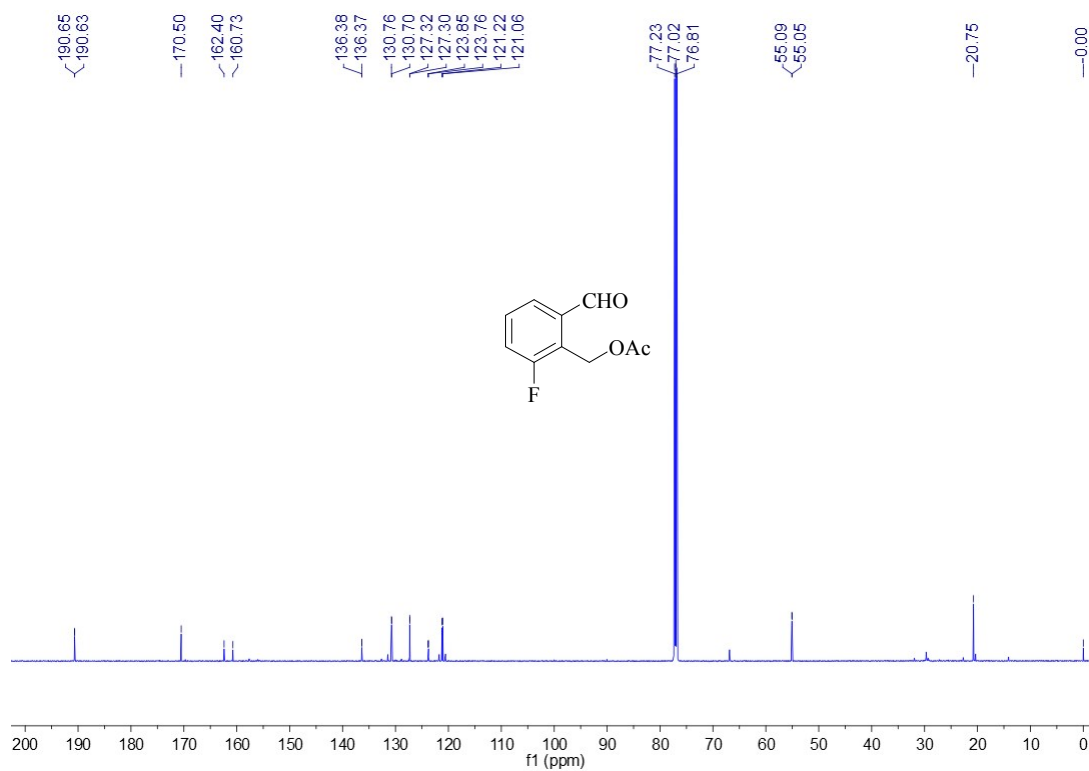
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### <sup>1</sup>H NMR of 2-fluoro-6-formylbenzyl acetate **2k**

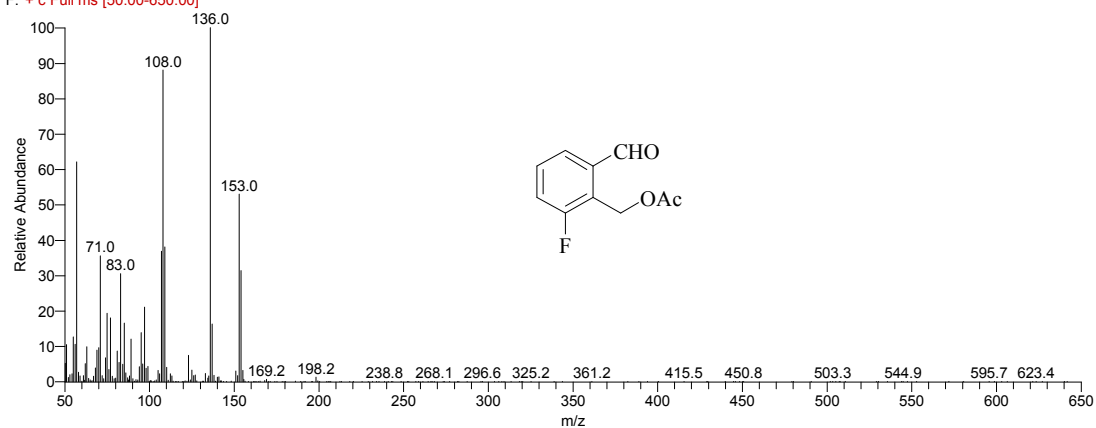


<sup>13</sup>C NMR of 2-fluoro-6-formylbenzyl acetate **2k**

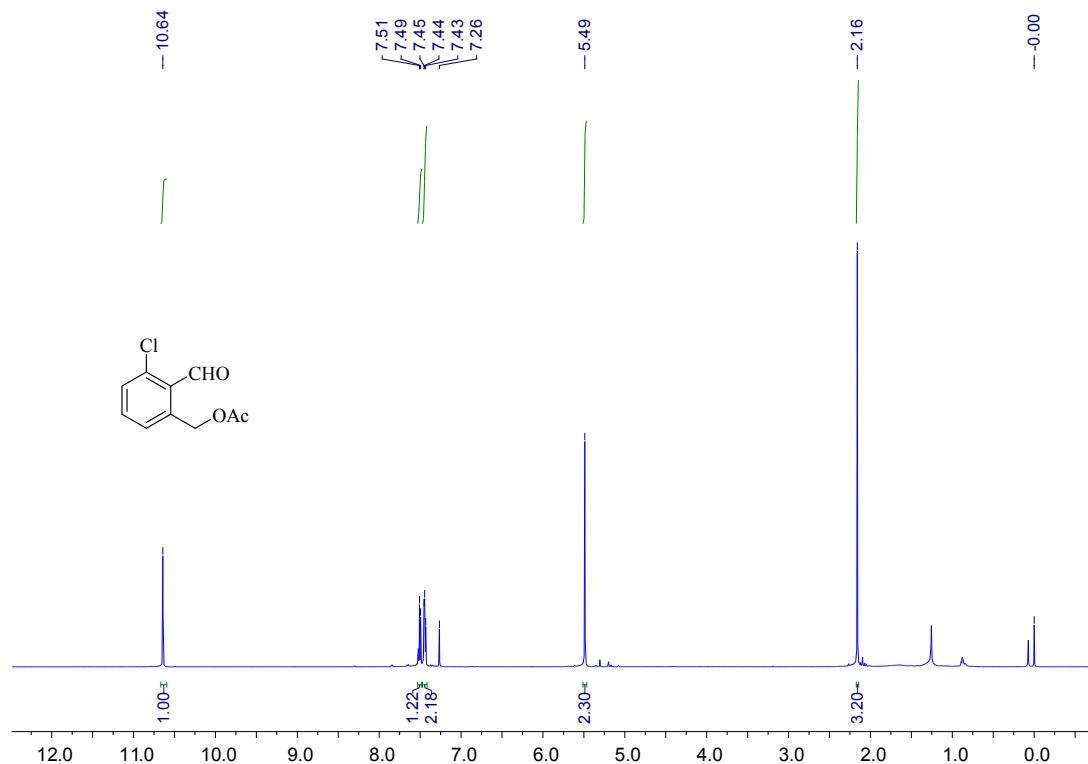


MS(EI) of 2-fluoro-6-formylbenzyl acetate **2k**

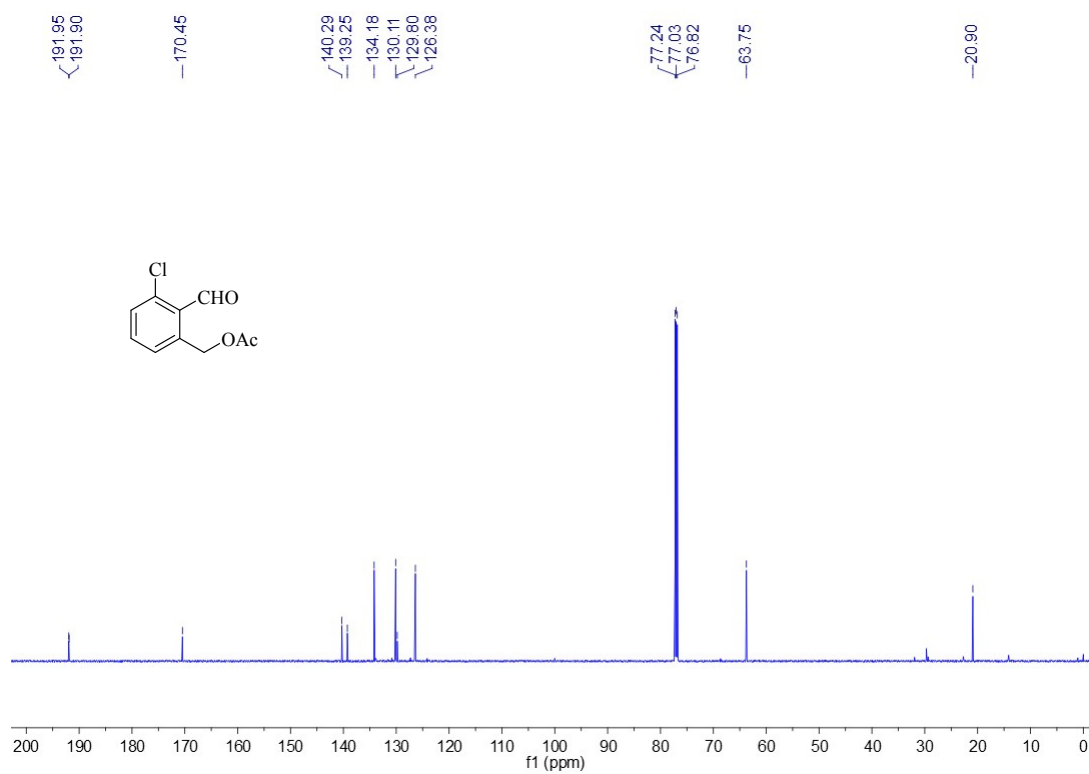
baoyongsheng127 #1949 RT: 6.51 AV: 1 AV: 5 SB: 12 1942-1947 1951-1956 NL: 2.28E6  
F: + c Full ms [50.00-650.00]



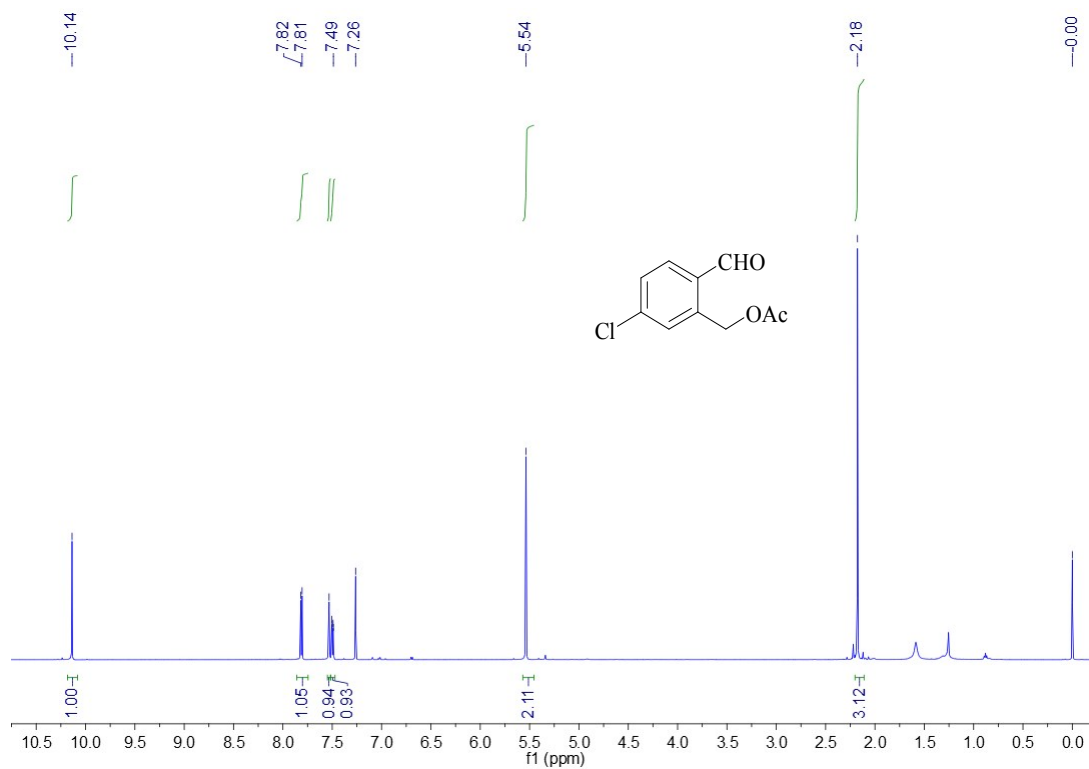
<sup>1</sup>H NMR of 3-chloro-2-formylbenzyl acetate **2l**



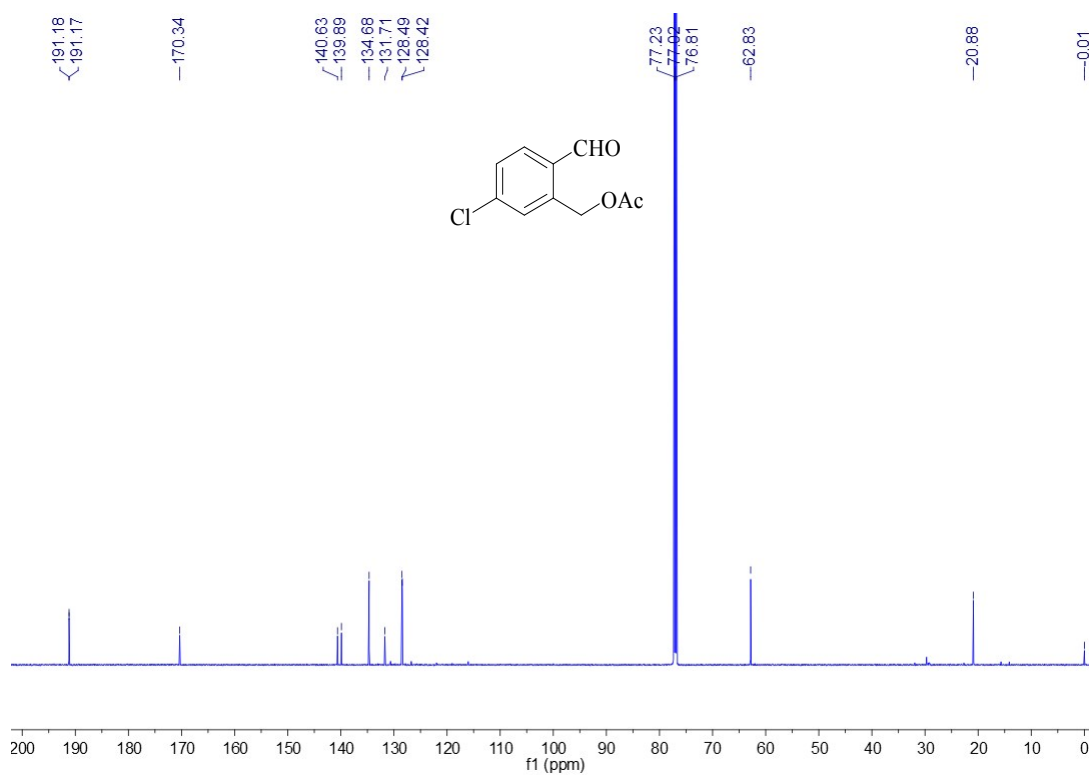
<sup>13</sup>C NMR of 3-chloro-2-formylbenzyl acetate **2l**



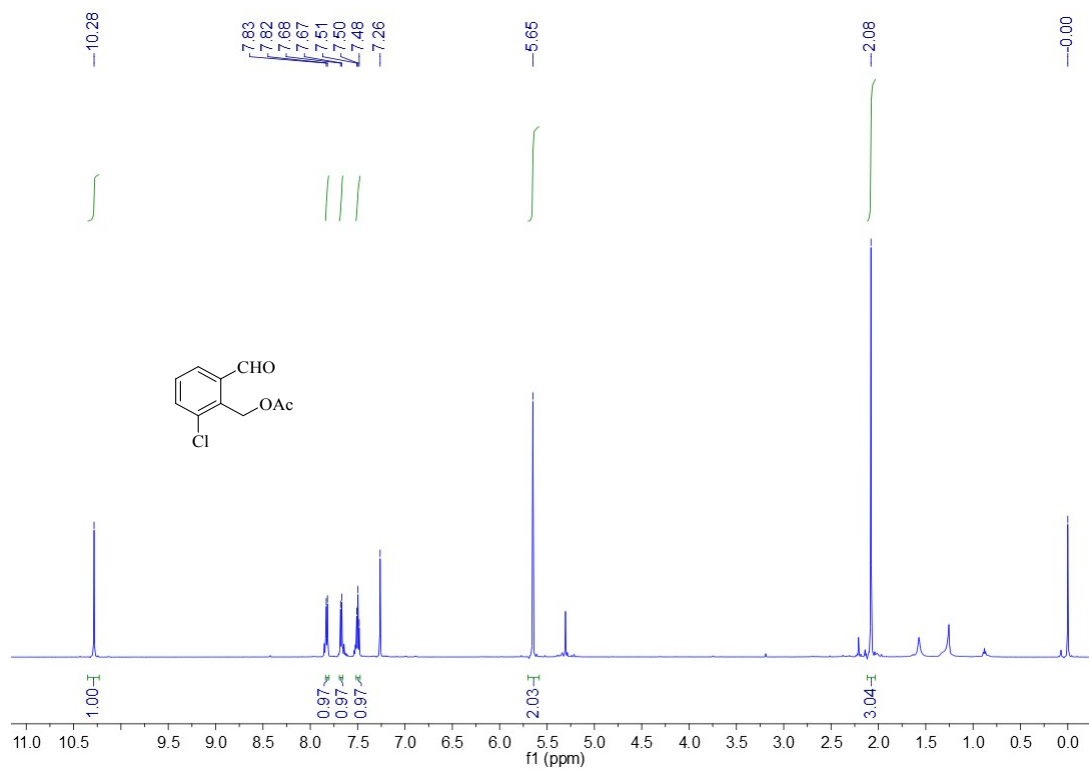
<sup>1</sup>H NMR of 5-chloro-2-formylbenzyl acetate **2m**



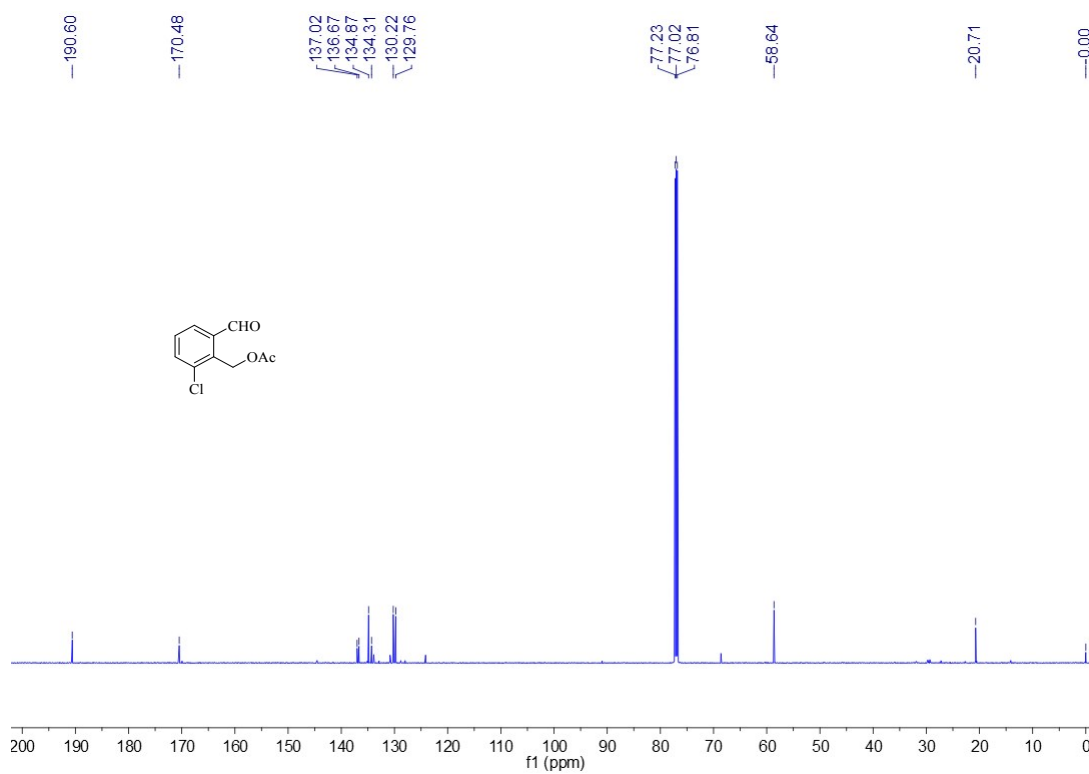
<sup>13</sup>C NMR of 5-chloro-2-formylbenzyl acetate **2m**.



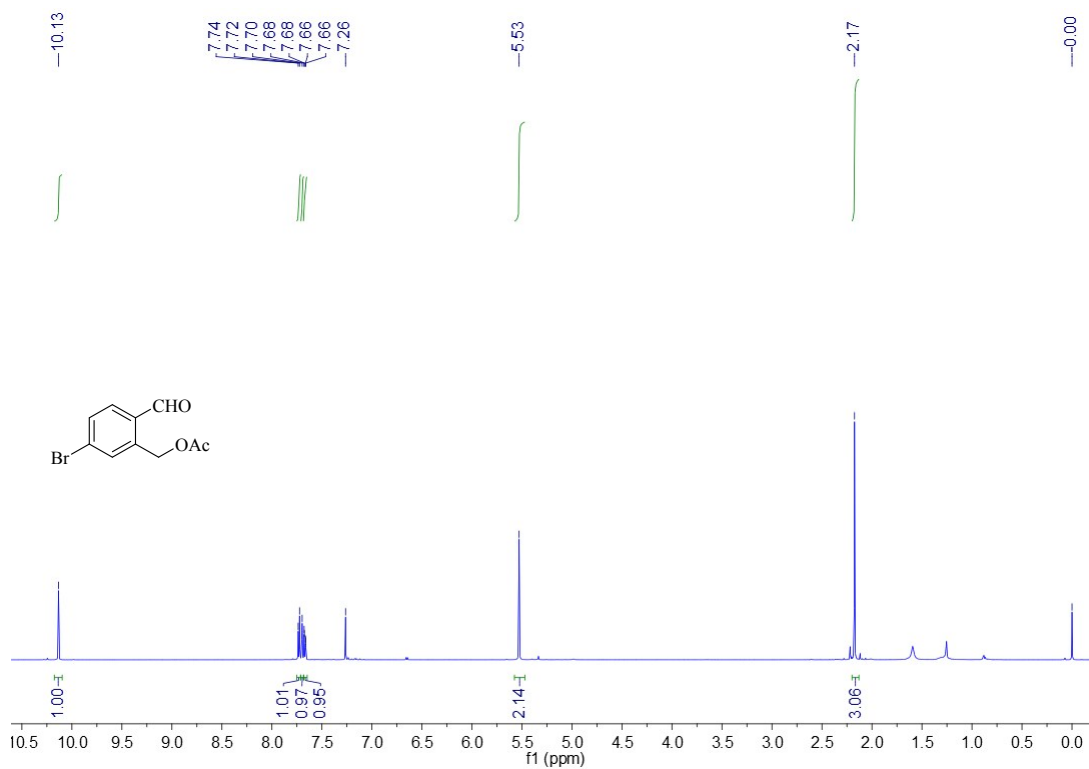
<sup>1</sup>H NMR of 2-chloro-6-formylbenzyl acetate **2n**



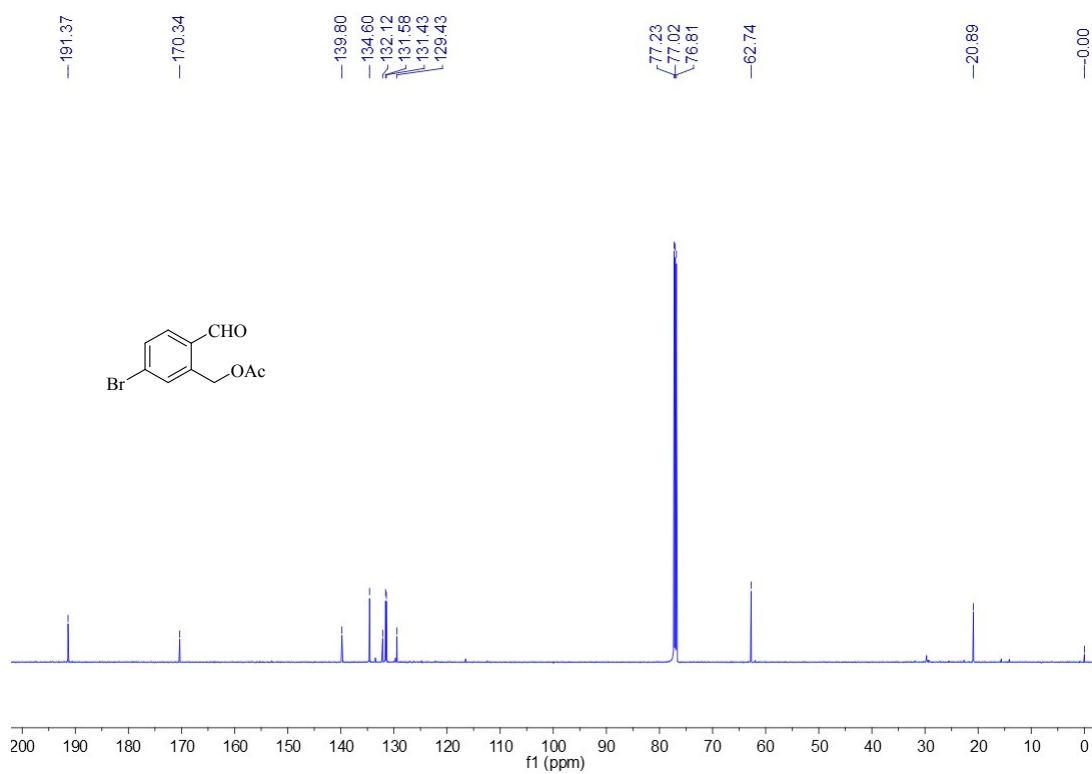
<sup>13</sup>C NMR of 2-chloro-6-formylbenzyl acetate **2n**



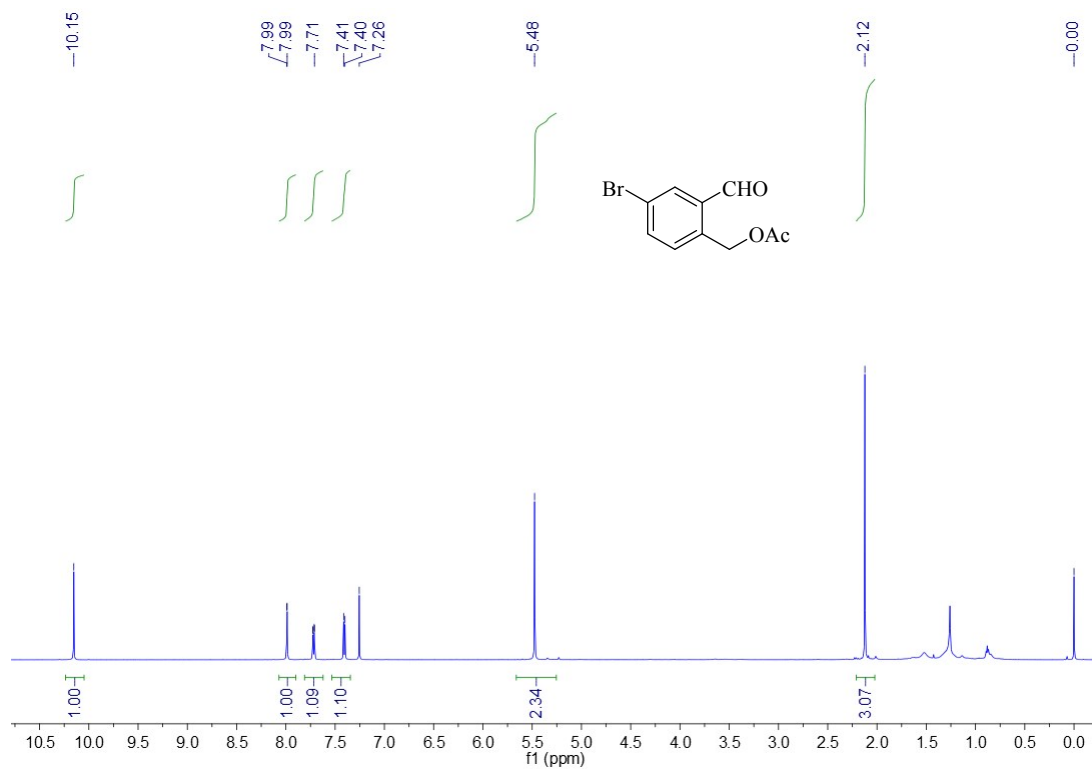
<sup>1</sup>H NMR of 5-bromo-2-formylbenzyl acetate **2o**



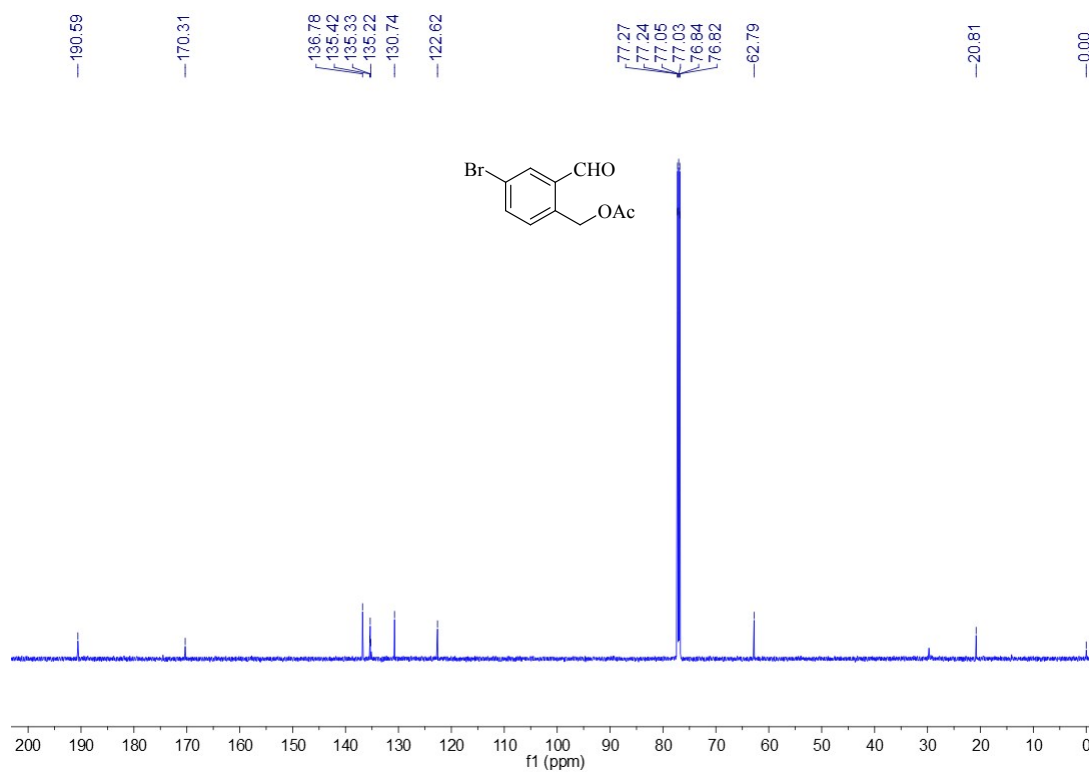
<sup>13</sup>C NMR of 5-bromo-2-formylbenzyl acetate **2o**



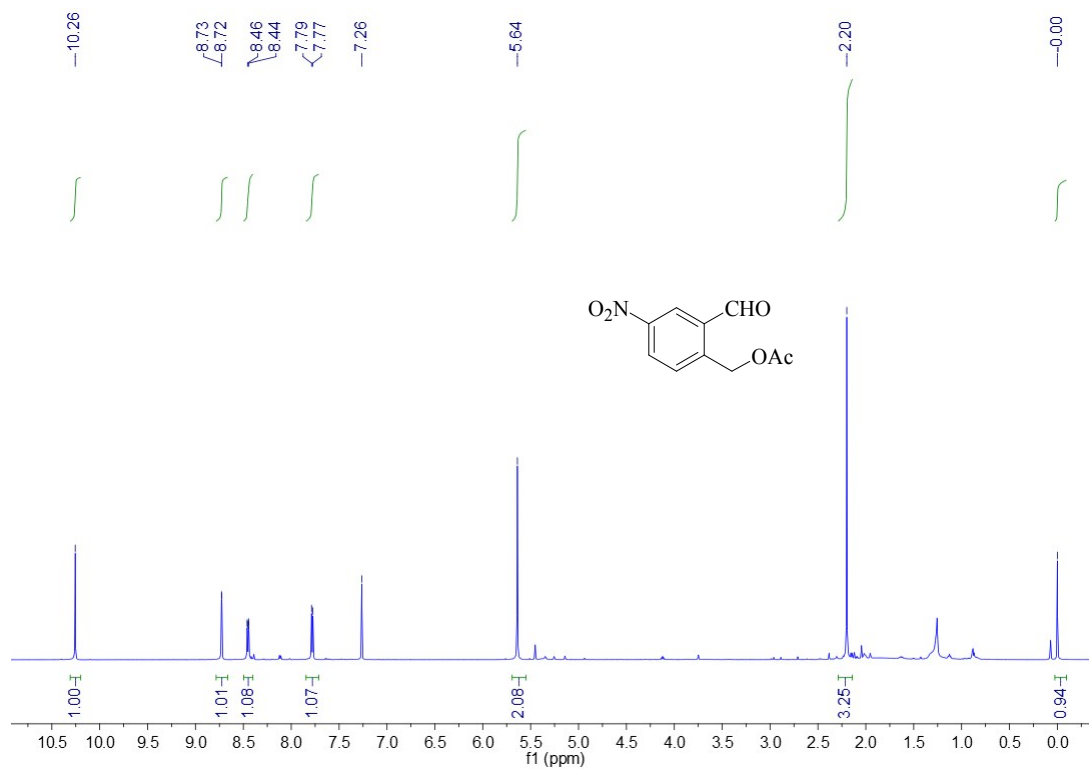
**<sup>1</sup>H NMR of 4-bromo-2-formylbenzyl acetate **2p****



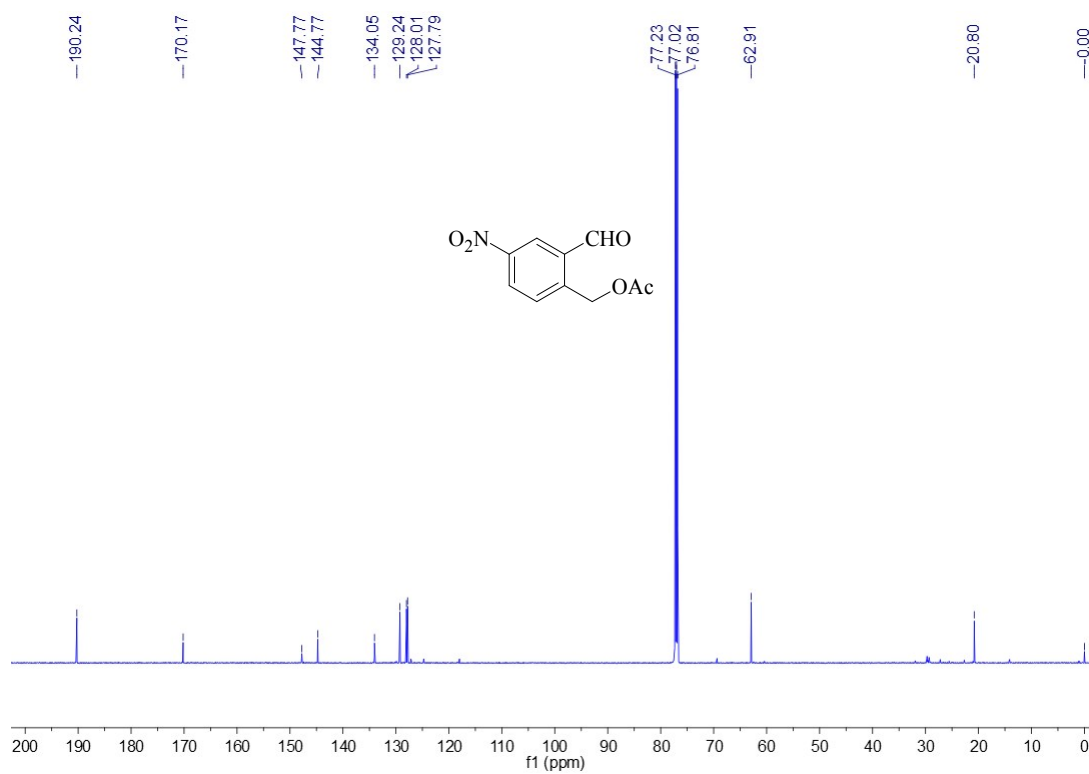
**<sup>13</sup>C NMR of 4-bromo-2-formylbenzyl acetate **2p****



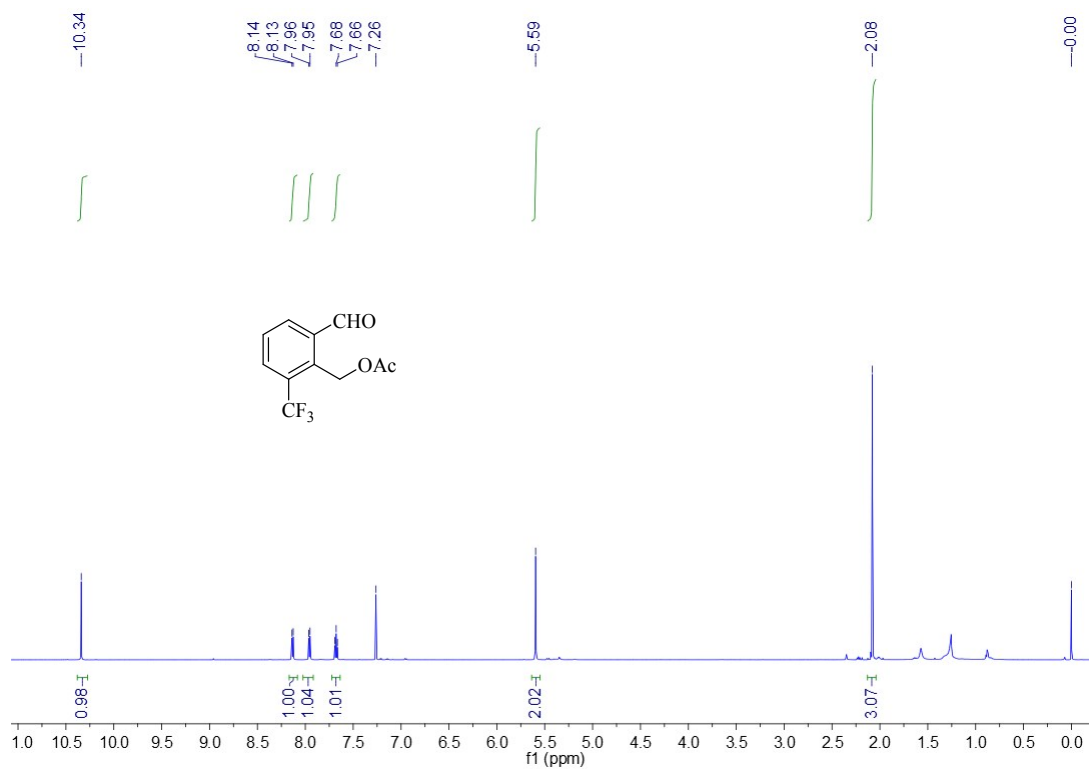
<sup>1</sup>H NMR of 2-formyl-4-nitrobenzyl acetate **2q**



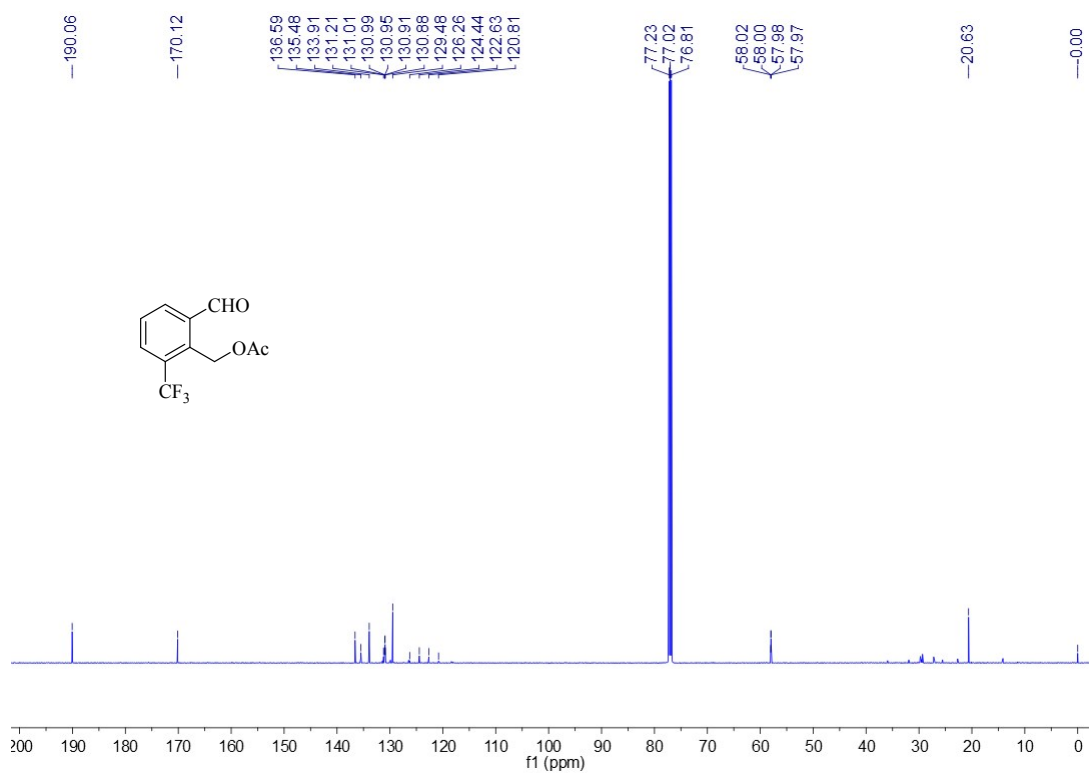
<sup>13</sup>C NMR of 2-formyl-4-nitrobenzyl acetate **2q**



<sup>1</sup>H NMR of 2-formyl-6-(trifluoromethyl)benzyl acetate **2r**

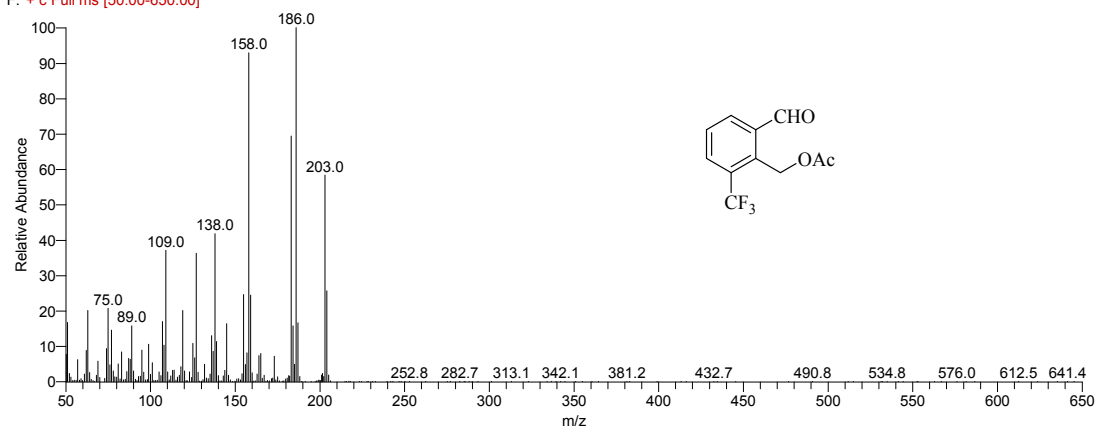


<sup>13</sup>C NMR of 2-formyl-6-(trifluoromethyl)benzyl acetate **2r**

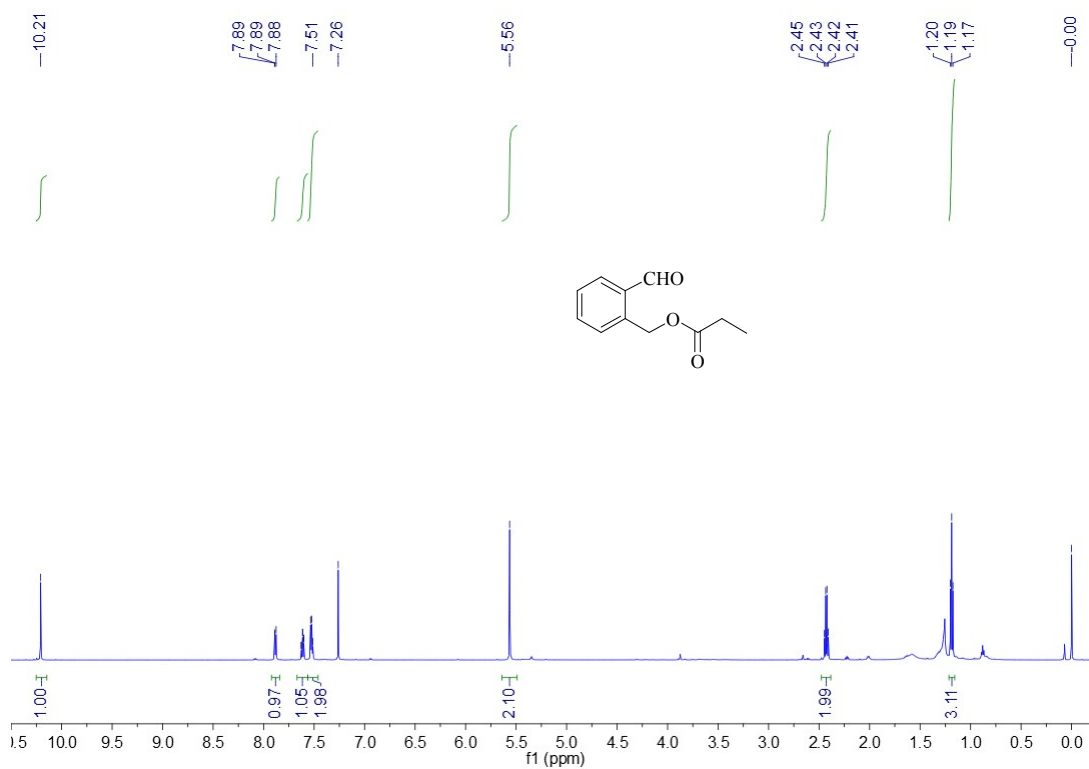


### MS(EI) of 2-formyl-4-(trifluoromethyl)benzyl acetate **2r**

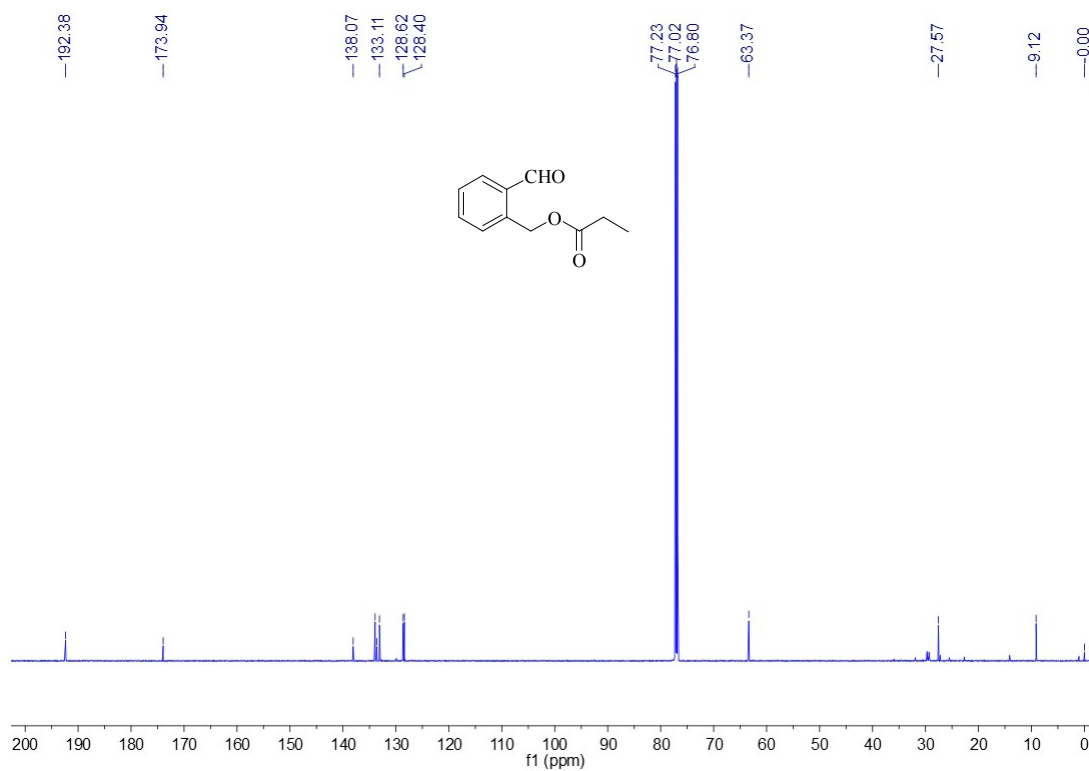
baoyongsheng133\_191206114818 #2012 RT: 6.71 AV: 1 AV: 5 SB: 12 2005-2010 2014-2019 NL: 4.18E6  
F: + c Full ms [50.00-650.00]



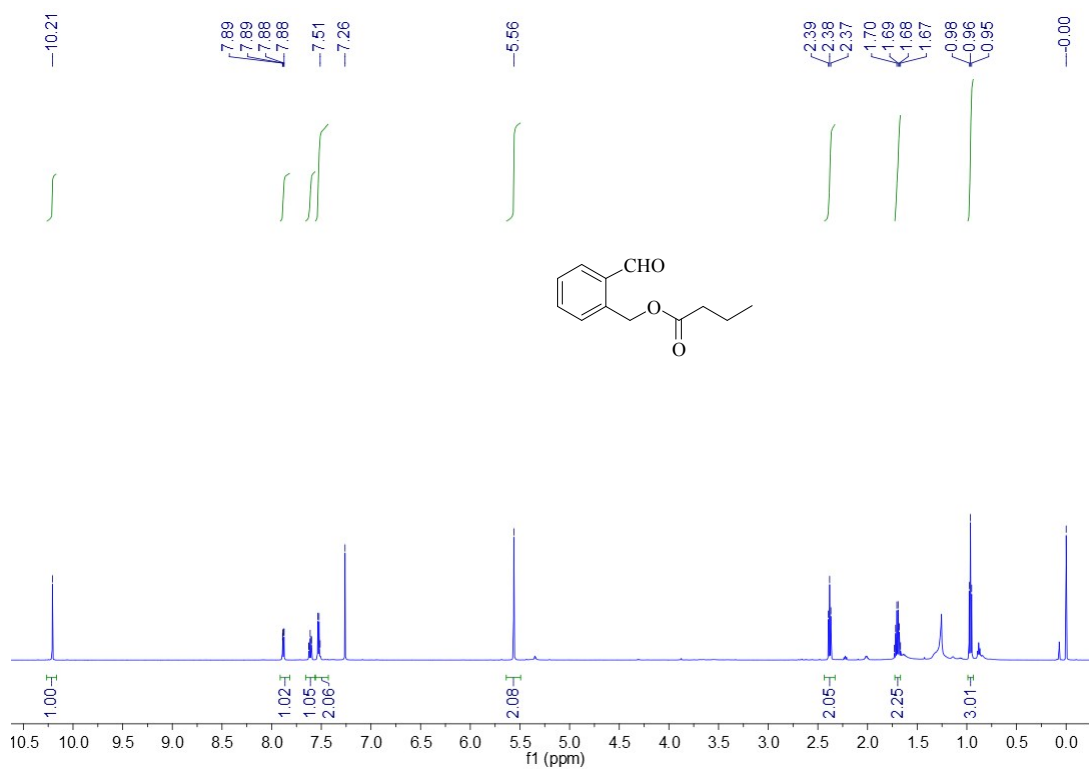
### <sup>1</sup>H NMR of propionic acid 2-formyl-benzyl ester **2s**



**<sup>13</sup>C NMR of propionic acid 2-formyl-benzyl ester **2s****



**<sup>1</sup>H NMR of butyric acid 2-formyl-benzyl ester **2t****



**<sup>13</sup>C NMR of butyric acid 2-formyl-benzyl ester **2t****

