Supporting Information

Visible-Light-Catalyzed Decarboxylation of Aryl acetic acids for the Construction of Aromatic Aldehydes

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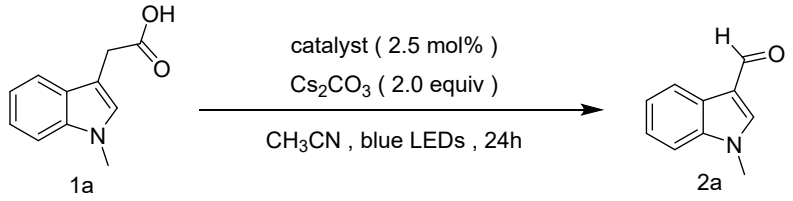
I. General Experimental Information

All reactions were carried out in flame-dried sealed tubes with magnetic stirring. Unless otherwise noted, all experiments were performed under argon atmosphere. Reagents were purchased from Accela, Acros, Aladdin, Adamas, Energy Chemical or TCI. Solvents were treated with 4 Å molecular sieves or sodium and distilled prior to use. Purifications of reaction products were carried out by flash chromatography using Qingdao Haiyang Chemical Co. Ltd silica gel (400-630 mesh). ^{1}H NMR and ^{13}C NMR spectra were recorded with tetramethylsilane (TMS) as internal standard at ambient temperature on a Bruker Avance III 400 MHz or 500 MHz for ^{1}H NMR Chemical shifts are reported in parts per million (ppm) and coupling constants are reported as Hertz (Hz). Splitting patterns are designated as singlet (s), doublet (d), triplet (t), doublet of doublet (dd), quartet (q). Splitting patterns that could not be interpreted or easily visualized are designated as multiple (m).

II. Optimization of Reaction Conditions

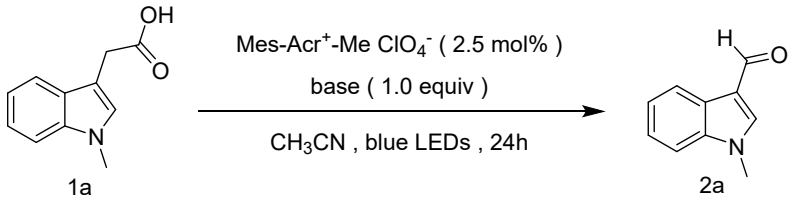
Table S1. Catalyst screening[a]



entry	catalyst	yield (%)[b]
1	$Ir(ppy)_3$	0
2	$Ru(bpy)_3Cl_2$	0
3	$Ir(dF(CF_3)ppy_2)(dtbbpy)^+$	37
4	Eosin Y	23
5	4CzIPN	10
6	Mes-Acr^+-$MeClO_4^-$	52

[a]All the reactions were performed using 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.1 mmol), Cs_2CO_3 (0.2 mmol), catalysts (2.5 mol %) and dry CH_3CN (2 mL) was added under 50 W blue LEDs and air atmosphere for 24 h, followed by flash chromatography on SiO_2. [b]Isolated yield.

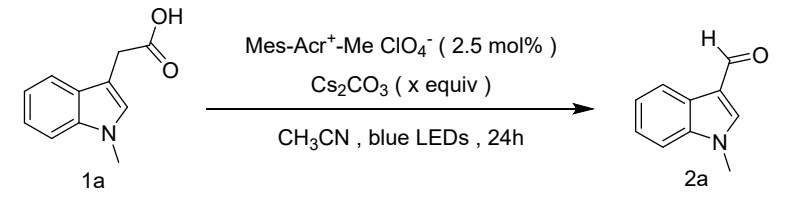
Table S2. Base Screening [a]



entry	base	yield (%)[b]
1	Cs_2CO_3	38
2	CH_3COONa	16
3	Na_2CO_3	33
4	K_2CO_3	13
5	K_3PO_4	21
6	Et_3N	20
7	DIPEA	20

[a]All the reactions were performed using 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.1 mmol), base (0.2 mmol) Mes-Acr^+-$MeClO_4^-$ (2.5 mol %) and dry CH_3CN (2 mL) was added under 50 W blue LEDs and air atmosphere for 24 h, followed by flash chromatography on SiO_2. [b]Isolated yield.

Table S3. The dosage of Cs_2CO_3 screening [a]

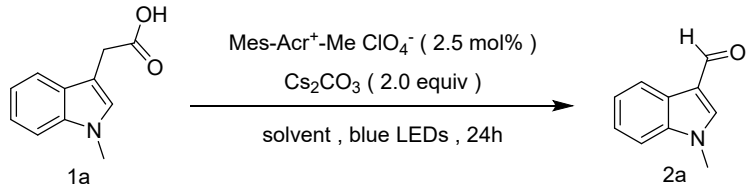


entry	Cs_2CO_3(x equiv)	Yield (%)[b]

1	1	38
2	1.5	50
3	2	52
4	3	10
5	3.5	10

[a]All the reactions were performed using 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.1 mmol), Cs_2CO_3 (x mmol), Mes-Acr^+-$MeClO_4^-$ (2.5 mol %) and dry CH_3CN (2 mL) was added under 50 W blue LEDs and air atmosphere for 24 h, followed by flash chromatography on SiO_2. [b]Isolated yield.

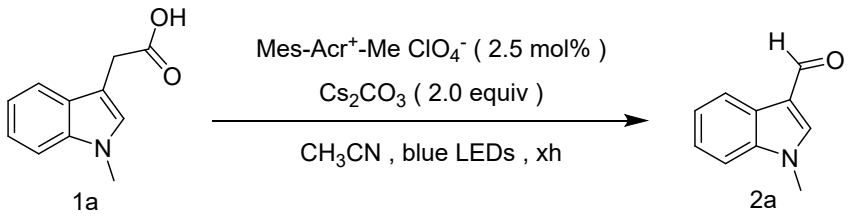
Table S4. solvent screening [a]



entry	solvent	yield (%)[b]
1	THF	11
2	CH_3CN	52
3	1,4-dioxane	0
4	MeOH	13
5	DMF	20
6	DCE	49
7	Toluene	50
8	DMSO	10
9	EtOH	8
10	DCM	25

[a]All the reactions were performed using 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.1 mmol), Cs_2CO_3 (0.2 mmol), Mes-Acr^+-$MeClO_4^-$ (2.5 mol %) and dry solvents (2 mL) was added under 50 W blue LEDs and air atmosphere for 24 h, followed by flash chromatography on SiO_2. [b]Isolated yield.

Table S5. Time screening [a]



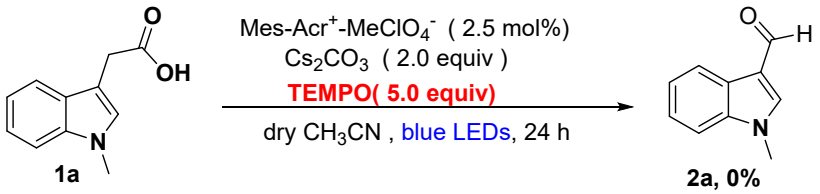
entry	time	yield (%)[b]
1	12	33
2	24	52
3	48	50

[a]All the reactions were performed using 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.1 mmol), Cs_2CO_3 (0.2 mmol), Mes-Acr^+-$MeClO_4^-$ (2.5 mol %) and dry CH_3CN (2 mL) was added under 50 W blue LEDs and air atmosphere for

x h, followed by flash chromatography on SiO_2. [b]Isolated yield.

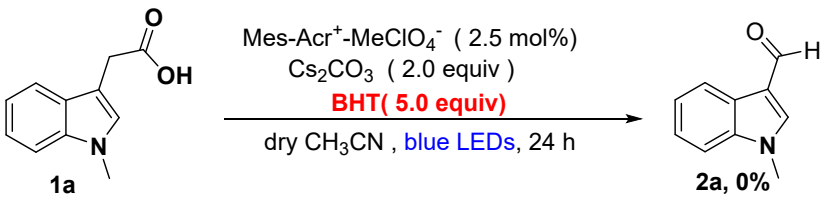
III. Control Experiments for the Mechanism Studies

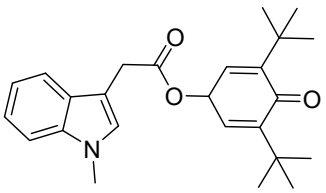
a) TEMPO was added to the reaction of 2-(1-methyl-1H-indol-3-yl) acetic acid 1a

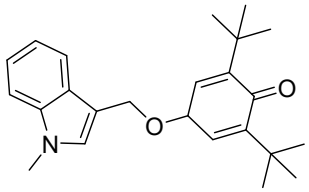


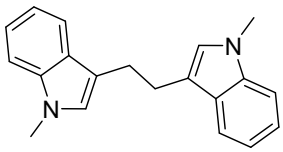
The reactions were carried out employing 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.10 mmol), Cs_2CO_3 (0.20 mmol), TEMPO (5.0 equiv.) with Mes-Acr^+-$MeClO_4^-$ (2.5 mol %) in dry CH_3CN (2 mL) at rt for 24 h under 50 W blue LEDs and Air in a sealed tube, no product was formed.

b) BHT was added to the reaction of 2-(1-methyl-1H-indol-3-yl) acetic acid 1a




BHT-2a detected by HRMS


BHT-2a' detected by HRMS


BHT-2a'' detected by HRMS

The reactions were carried out employing 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (0.10 mmol), Cs_2CO_3 (0.20 mmol), BHT (5.0 equiv.) with Mes-Acr^+-Me ClO_4^- (2.5 mol %) in dry CH_3CN (2 mL) at rt for 24 h under 50 W blue LEDs and Air in a sealed tube, no product was formed.

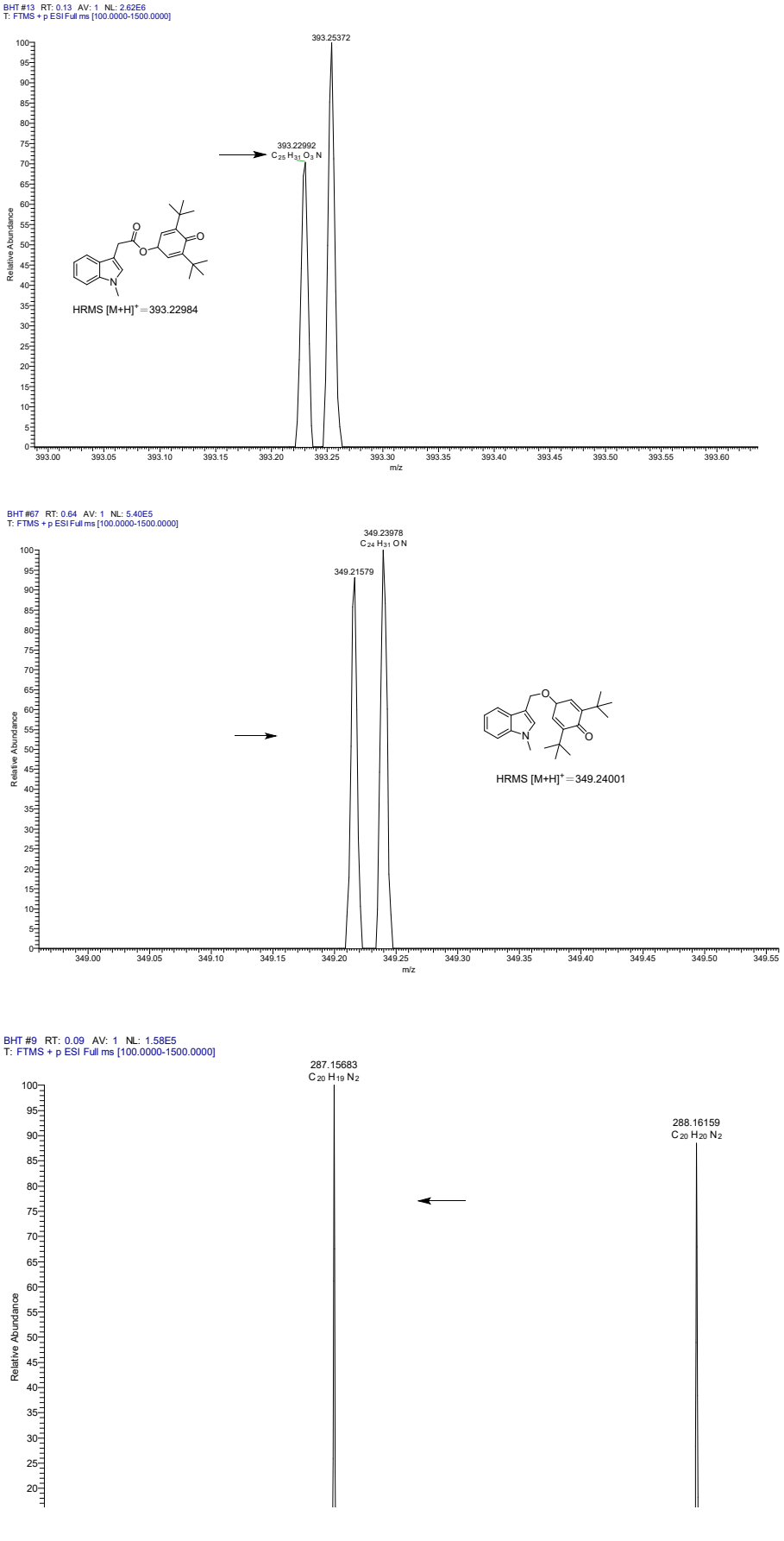
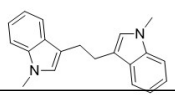


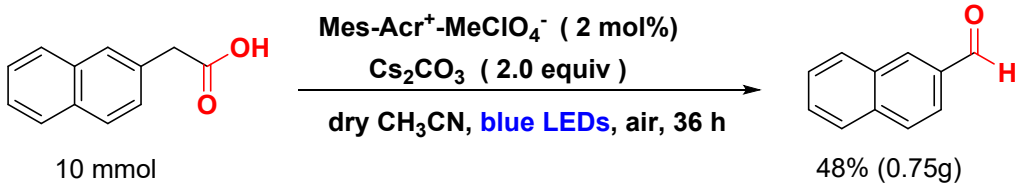
Figure S1. HRMS spectra of reaction mixture in radical trapping experiment



Chemical Formula: $C_{20}H_{20}N_2$
Exact Mass: 288.16

IV. Gram-scale reaction and synthetic applications

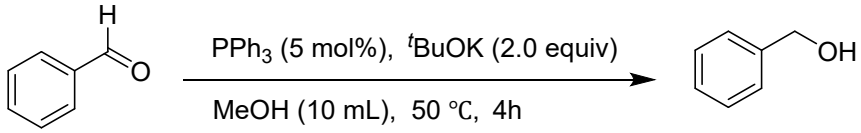
a) Gram-scale reaction of 2-Naphthylacetic acid 1r



10 mmol 48% (0.75g)

The reactions were carried out employing 2-Naphthylacetic acid **1r** (10 mmol), Cs_2CO_3 (20 mmol), Mes-Acr^+-$MeClO_4^-$ (2.5 mol %) in dry CH_3CN (20 mL) at rt for 36 h under 50 W blue LEDs and Air in a sealed tube, giving the corresponding product **2r** in 48% yield.

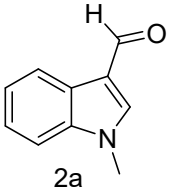
b) Synthetic applications of benzaldehyde 2g



2g 4b

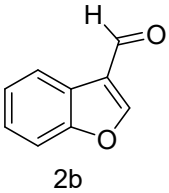
The reactions were carried out employing benzaldehyde **2g** (1.5 equiv.), PPh_3(5 mol%), ^{t}BuOK (2.0 equiv), MeOH(10 mL), at 50 °C under air for 4 h, the desired product **4b** was obtained in 70% yield.

V. Detail Characterization for the Compounds 2



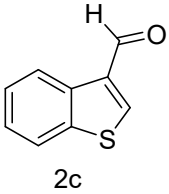
2a

1-Methyl-1H-indole-3-carbaldehyde (2a)[1]: The title compound was prepared from 2-(1-methyl-1H-indol-3-yl) acetic acid **1a** (34 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 15 mg, 52% yield; **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 9.97 (s, 1H), 8.32 (s, 1H), 7.64 (s, 1H), 7.37 (s, 3H), 3.85 (s, 3H).



2b

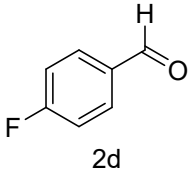
Benzofuran-3-carbaldehyde (2b)[2]: The title compound was prepared from 2-(benzofuran-3-yl) acetic acid **1b** (36 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 15 mg, 52% yield; **^{1}H NMR** (500 MHz, Chloroform-d) δ 10.17 (s, 1H), 8.26 (s, 1H), 8.19 – 8.17 (m, 1H), 7.55 (m, *J* = 7.2 Hz, 1H), 7.43 – 7.36 (m, 2H).



2c

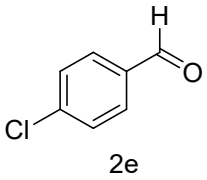
Benzo[b]thiophene-3-carbaldehyde (2c)[2]: The title compound was prepared from 2-(benzo[b]thiophen-3-yl) acetic acid **1c** (39 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether:

ethyl acetate); 21 mg, 64% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.13 (s, 1H), 8.68 (dt, *J* = 8.1, 1.0 Hz, 1H), 8.30 (s, 1H), 7.87 (dt, *J* = 8.0, 1.0 Hz, 1H), 7.53 – 7.49 (m, 1H), 7.47 – 7.43 (m, 1H).



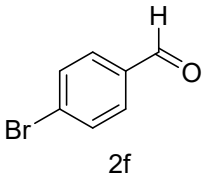
2d

4-Fluorobenzaldehyde (2d)[2]: The title compound was prepared from 2-(4-fluorophenyl) acetic acid **1d** (31 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 5 mg, 20% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.95 (s, 1H), 7.91 – 7.87 (m, 2H), 7.21 – 7.17 (m, 2H); **^{19}F NMR** (471 MHz, Chloroform-d) δ -102.26, -102.27, -104.39.



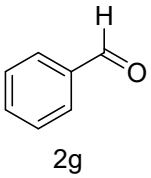
2e

4-Chlorobenzaldehyde (2e)[2]: The title compound was prepared from 2-(4-chlorophenyl) acetic acid **1e** (34 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 15 mg, 53% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.97 (s, 1H), 7.82 – 7.80 (m, 2H), 7.52 – 7.49 (m, 2H).



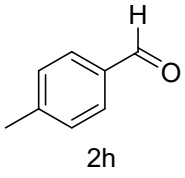
2f

4-Bromobenzaldehyde (2f)[2]: The title compound was prepared from 2-(4-bromophenyl) acetic acid **1f** (43 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 26 mg, 70% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.99 (s, 1H), 7.77 (d, *J* = 8.4 Hz, 2H), 7.70 (d, *J* = 8.5 Hz, 2H).



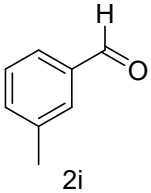
2g

Benzaldehyde (2g)[3] : The title compound was prepared from 2-phenylacetic acid **1g** (27 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 11 mg, 54% yield; **^{1}H NMR** (500 MHz, Chloroform-d) δ 10.02 (s, 1H), 7.88 (d, *J* = 7.7 Hz, 2H), 7.65 – 7.61 (m, 1H), 7.53 (t, *J* = 7.6 Hz, 2H).



2h

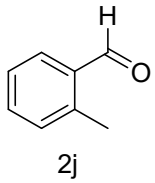
4-Methylbenzaldehyde (2h): The title compound was prepared from 2-(*p*-tolyl) acetic acid **1h** (30 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 10 mg, 42% yield; **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 9.88 (s, 1H), 7.70 (s, 2H), 7.25 (s, 2H), 2.36 (s, 3H).



2i

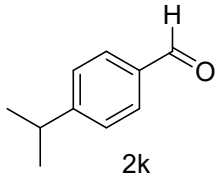
3-Methylbenzaldehyde (2i): The title compound was prepared from 2-(*m*-tolyl) acetic acid **1i** (30 mg, 0.20 mmol) and and was purified by column chromatography to give a

pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 9 mg, 38% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.94 (s, 1H), 7.77 – 7.74 (m, 2H), 7.31 (d, *J* = 7.9 Hz, 2H), 2.42 (s, 3H).



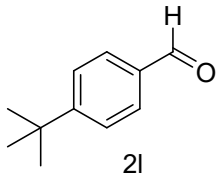
2j

2-Methylbenzaldehyde (2j): The title compound was prepared from 2-(*o*-tolyl) acetic acid **1j** (30 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 14 mg, 60% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.26 (s, 1H), 7.79 (dd, *J* = 7.6, 1.5 Hz, 1H), 7.46 (td, *J* = 7.5, 1.5 Hz, 1H), 7.35 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 7.6 Hz, 1H), 2.66 (s, 3H).



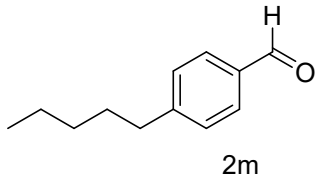
2k

4-Isopropylbenzaldehyde (2k): The title compound was prepared from 2-(4-isopropylphenyl) acetic acid **1k** (36 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 18 mg, 68% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.99 (s, 1H), 7.83 (d, *J* = 8.1 Hz, 2H), 7.40 (d, *J* = 8.1 Hz, 2H), 3.01 (p, *J* = 6.9 Hz, 1H), 1.30 (d, *J* = 6.9 Hz, 6H).



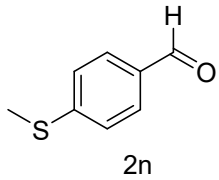
2l

4-(tert-butyl)Benzaldehyde (2l)[4]: The title compound was prepared from 2-(4-(*t*ert-butyl)phenyl) acetic acid **1l** (40 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 22 mg, 68% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.00 (s, 1H), 7.84 (d, *J* = 8.3 Hz, 2H), 7.58 (d, *J* = 8.4 Hz, 2H), 1.38 (s, 9H).



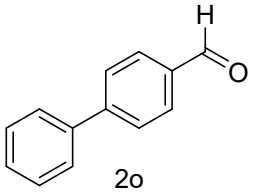
2m

4-Pentylbenzaldehyde (2m): The title compound was prepared from 2-(4-pentylphenyl) acetic acid **1m** (42 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 27 mg, 76% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.98 (s, 1H), 7.81 (d, *J* = 8.1 Hz, 2H), 7.35 (d, *J* = 7.9 Hz, 2H), 2.71 – 2.68 (m, 2H), 1.69 – 1.63 (m, 2H), 1.37 – 1.32 (m, 4H), 0.91 (t, *J* = 6.9 Hz, 3H).



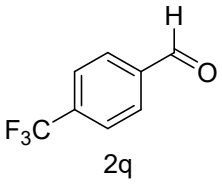
2n

4-(methylthio)Benzaldehyde (2n)[4]: The title compound was prepared from 2-(4-(methylthio)phenyl) acetic acid **1n** (36 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 18 mg, 60% yield; **^{1}H NM**R (500 MHz, Chloroform-*d*) δ 9.93 (s, 1H), 7.78 (d, *J* = 8.5 Hz, 2H), 7.33 (d, *J* = 8.4 Hz, 2H), 2.54 (s, 3H).



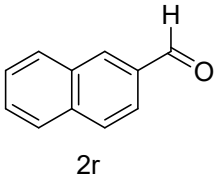
2o

[1,1'-biphenyl]-4-Carbaldehyde (2o)[2]: The title compound was prepared from 2-([1,1'-biphenyl]-4-yl) acetic acid **1o** (42 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 25 mg, 70% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.08 (s, 1H), 7.97 (d, *J* = 8.3 Hz, 2H), 7.77 (d, *J* = 8.3 Hz, 2H), 7.66 (d, *J* = 6.9 Hz, 2H), 7.51 (t, *J* = 7.4 Hz, 2H), 7.45 (t, *J* = 7.3 Hz, 1H).



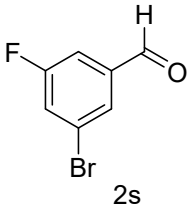
2q

4-(trifluoromethyl)Benzaldehyde (2q)[3]: The title compound was prepared from 2-(4-(trifluoromethyl)phenyl)acetic acid **1q** (41 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 8 mg, 23% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.09 (s, 1H), 8.01 – 7.98 (m, 2H), 7.80 (d, *J* = 8.0 Hz, 2H); **^{19}F NMR** (471 MHz, Chloroform-d) δ -63.11.



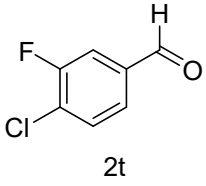
2r

2-Naphthaldehyde (2r)[2]: The title compound was prepared from 2-(naphthalen-2-yl)acetic acid **1r** (37 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 24 mg, 77% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.18 (s, 1H), 8.35 (s, 1H), 8.03 (d, *J* = 8.1 Hz, 1H), 8.00 – 7.95 (m, 2H), 7.93 (d, *J* = 8.1 Hz, 1H), 7.69 – 7.65 (m, 1H), 7.63 – 7.60 (m, 1H).



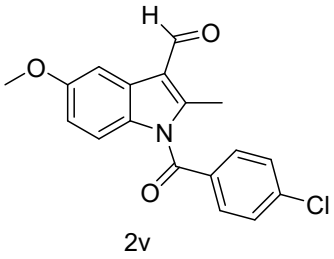
2s

3-Bromo-5-fluorobenzaldehyde (2s)[6]: The title compound was prepared from 2-(3-bromo-5-fluorophenyl)acetic acid **1s** (46 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 10 mg, 30% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 10.29 (d, *J* = 3.0 Hz, 1H), 7.64 – 7.59 (m, 2H), 7.21 – 7.17 (m, 1H); **^{19}F NMR** (471 MHz, Chloroform-d) δ -108.40, -108.41, -108.43.



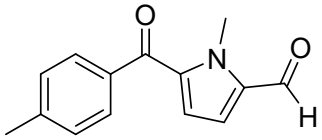
2t

4-Chloro-3-fluorobenzaldehyde (2t):[6] The title compound was prepared from 2-(4-chloro-3-fluorophenyl)acetic acid **1t** (38 mg, 0.20 mmol) and and was purified by column chromatography to give a pale white solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 6 mg, 16% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.92 (s, 1H), 7.95 (dd, *J* = 7.1, 2.1 Hz, 1H), 7.81 – 7.77 (m, 1H), 7.30 (t, *J* = 8.4 Hz, 1H); **^{19}F NMR** (471 MHz, Chloroform-d) δ -112.80, -112.82, -112.83.



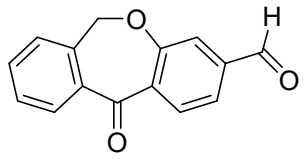
2v

1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indole-3-carbaldehyde (2v)[5]: The title compound was prepared from 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetic acid **1v** (71 mg, 0.20 mmol) and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 34 mg, 48% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 12.40 (s, 1H), 8.87 (d, *J* = 9.2 Hz, 1H), 7.99 – 7.96 (m, 2H), 7.48 – 7.46 (m, 2H), 7.43 (d, *J* = 3.0 Hz, 1H), 7.19 (dd, *J* = 9.2, 3.0 Hz, 1H), 3.86 (s, 3H), 2.69 (s, 3H).



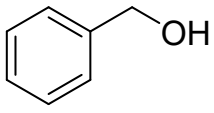
2w

1-(4-chlorobenzoyl)-6-Methoxy-2-methyl-1H-indole-3-carbaldehyde (2w)[6]: The title compound was prepared from 2-(1-(4-chlorobenzoyl)-6-methoxy-2-methyl-1H-indol-3-yl) acetic acid **1w** (71 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 28 mg, 62% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 12.40 (s, 1H), 8.87 (d, *J* = 9.2 Hz, 1H), 7.99 – 7.96 (m, 2H), 7.48 – 7.46 (m, 2H), 7.43 (d, *J* = 3.0 Hz, 1H), 7.19 (dd, *J* = 9.2, 3.0 Hz, 1H), 3.86 (s, 3H), 2.69 (s, 3H).



2x

11-Oxo-6,11-dihydrodibenzo[*b,e*]oxepine-2-carbaldehyde (2x)[5]: The title compound was prepared from 2-(11-oxo-6,11-dihydrodibenzo[*b,e*]oxepin-2-yl) acetic acid **1x** (54 mg, 0.20 mmol) and and was purified by column chromatography to give a pale yellow solid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 29 mg, 61% yield; **^{1}H NMR** (500 MHz, Chloroform-*d*) δ 9.99 (s, 1H), 8.73 (d, *J* = 2.2 Hz, 1H), 8.02 (dd, *J* = 8.5, 2.2 Hz, 1H), 7.88 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.62 – 7.58 (m, 1H), 7.51 (td, *J* = 7.6, 1.2 Hz, 1H), 7.42 – 7.39 (m, 1H), 7.17 (d, *J* = 8.6 Hz, 1H), 5.28 (s, 2H).



4b

Phenylmethanol (4b): The title compound was prepared from benzaldehyde **2b** (21.2 mg, 0.20 mmol) and and was purified by column chromatography to give Colourless and transparent liquid; R_f = 0.60 (20:1 petroleum ether: ethyl acetate); 15 mg, 70% yield; **^{1}H NMR** (400 MHz, Chloroform-*d*) δ 7.39 (d, *J* = 4.0 Hz, 4H), 7.35 – 7.30 (m, 1H), 4.70 (s, 2H), 2.04 (s, 1H).

IV. References

[1] Z. K. Lei, F. Xue, B. Wang, S. J. Wang, Y. H. Zhang, Yu Xia, W. W. Jin , C. J. Liu, A stable and recyclable Z-scheme g-C_3N_4/rGO/$BiVO_4$ heterojunction photocatalyst for site-selective C-3 formylation of indoles with methanol as a formyl source under visible light. *Green Chem.,* 2023, **25**, 348–356

[2] Y. Wang, B. Xu, X. Xiao, X. Jie, W. Su, Copper-catalyzed aerobic photodecarboxylation of carboxylic acids to aldehydes and ketones. doi.org/10.1016/j.gresc.2025.03.006.

[3] J. Cui, K.-K. Niu, R.-Z. Zhang, H. Liu, S. S. Yu. L.-B. Xing, Photocatalytic selective oxidation of toluene under encapsulated air conditions. *Chem. Commun*. 2024, **60**, 4310–4313;

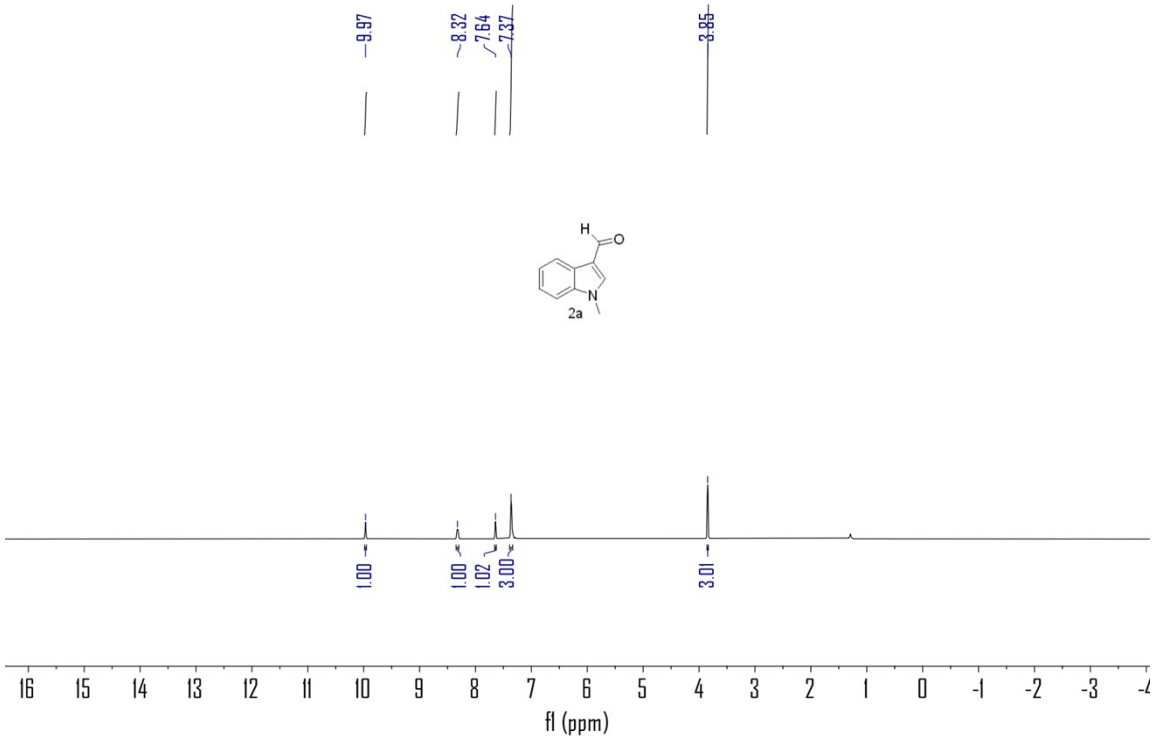
[4] M. Uygur, J. H. Kuhlmann, M. Ca. Pérez-Aguilar, D. G. Piekarski, O. G. Mancheño, Metal- and additive-free C–H oxygenation of alkylarenes by visible-light photoredox catalysis, *Green Chem*. 2021, **23**, 3392–3399;

[5] M Innocent, G Lalande, F Cam, T A, A Guérinot， Iron-Catalyzed, Light-Driven Decarboxylative Oxygenation. *Eur. J. Org. Chem.* 2023, **26**, e202300892;

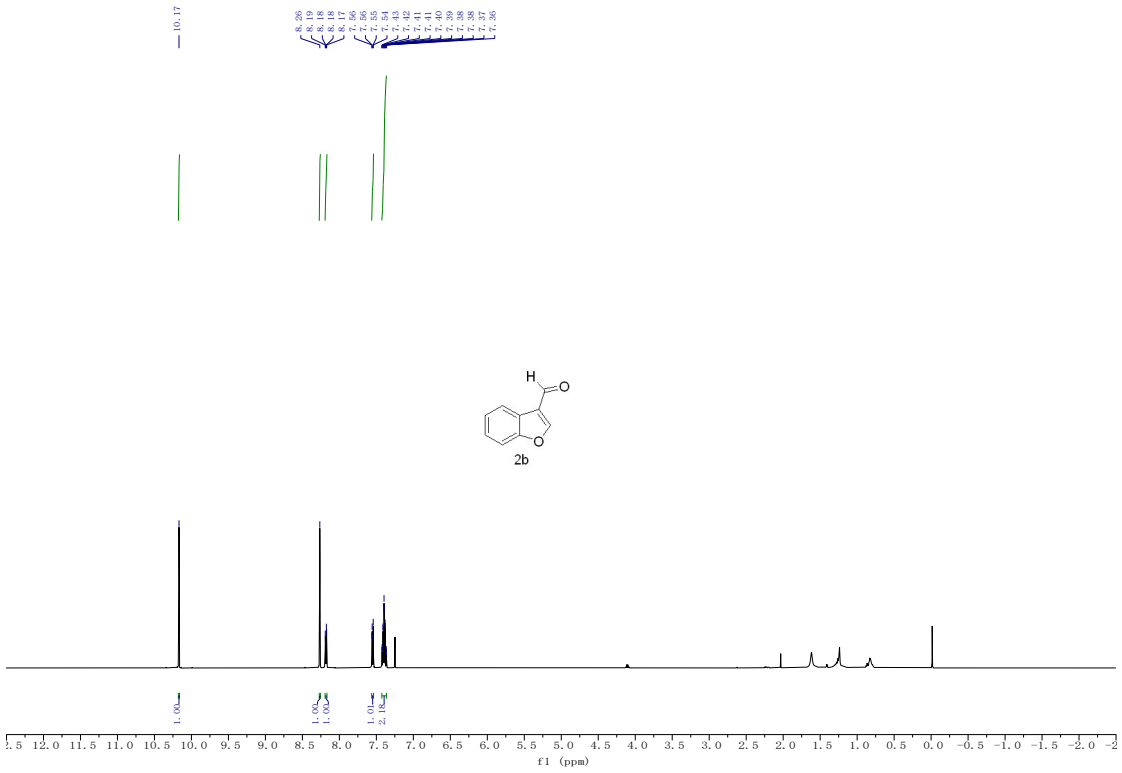
[6] Q. Z. Xu, W. Ou, H. Hou, Q. Y. Wang, L. Yu, C. L. Su, Photosynthesis of C-1-Deuterated Aldehydes via Chlorine RadicalMediated Selective Deuteration of the Formyl C−H Bond， *Org. Lett.* 2024, **26**, 4098−4103;

IX. ^{1}H NMR and ^{13}C NMR Spectrum of All Products.

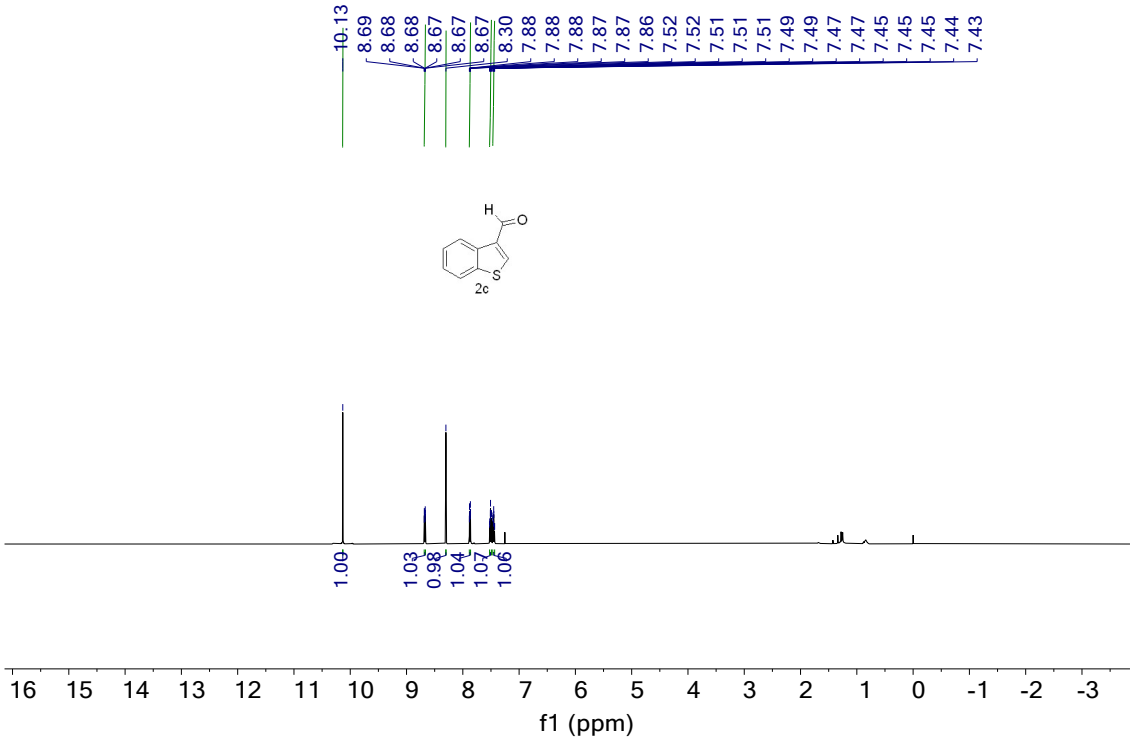
The ^{1}H NMR (400 MHz) spectrum for **2a** (using $CDCl_3$ as solvent)



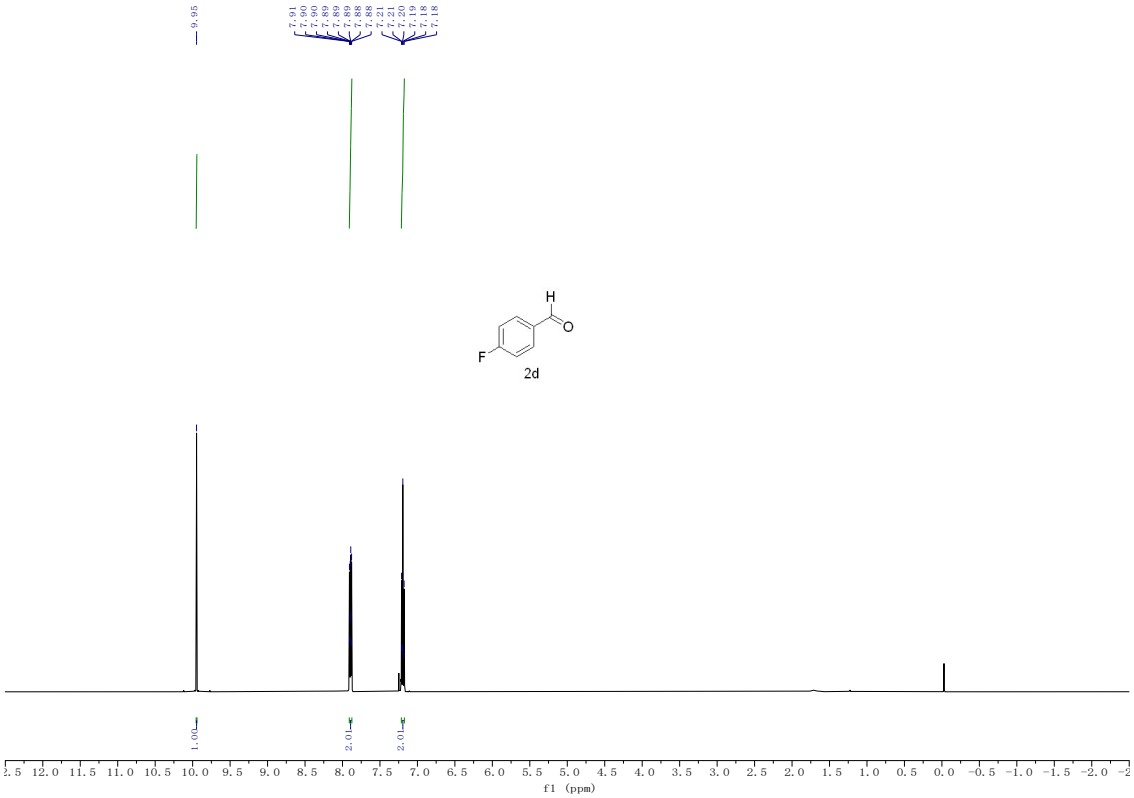
The ^{1}H NMR (500 MHz)) spectrum for **2b** (using $CDCl_3$ as solvent)

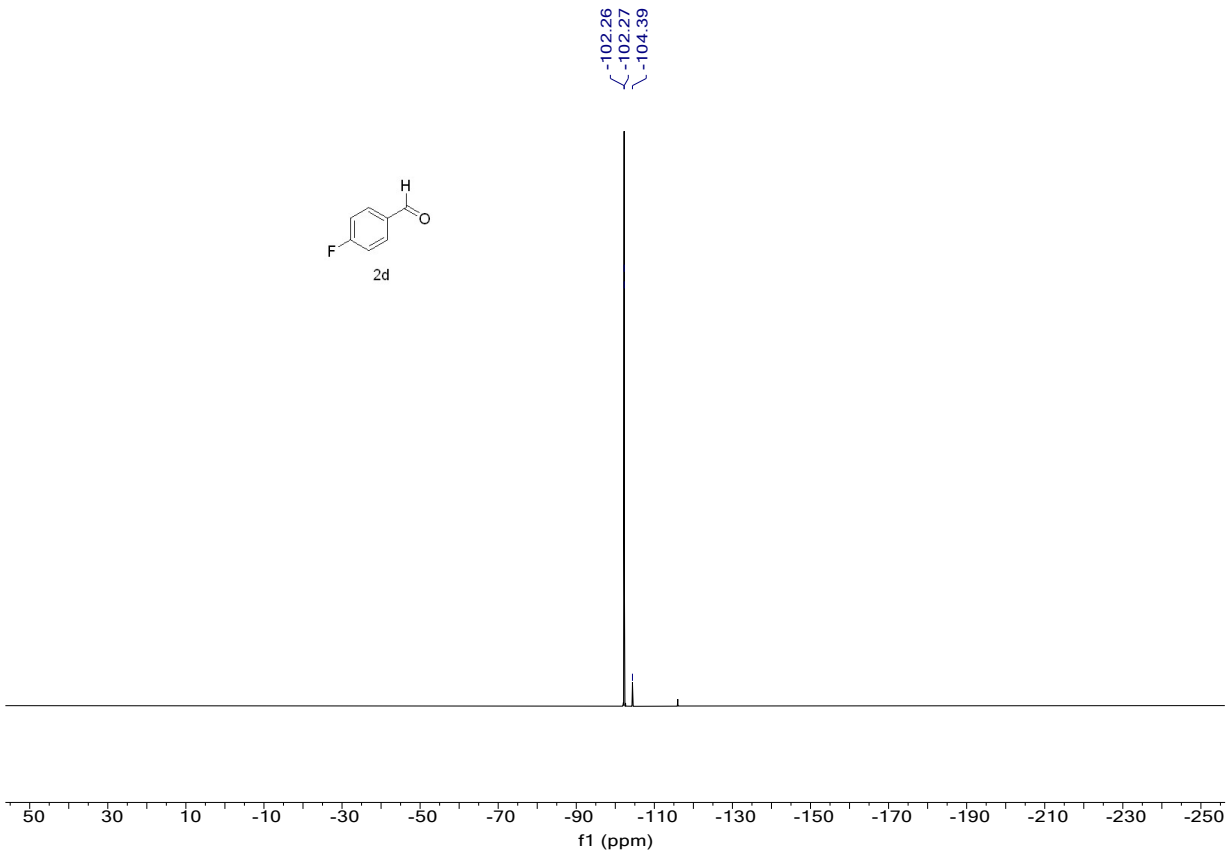


The ^{1}H NMR (400 MHz) spectrum for **2c** (using $CDCl_3$ as solvent)

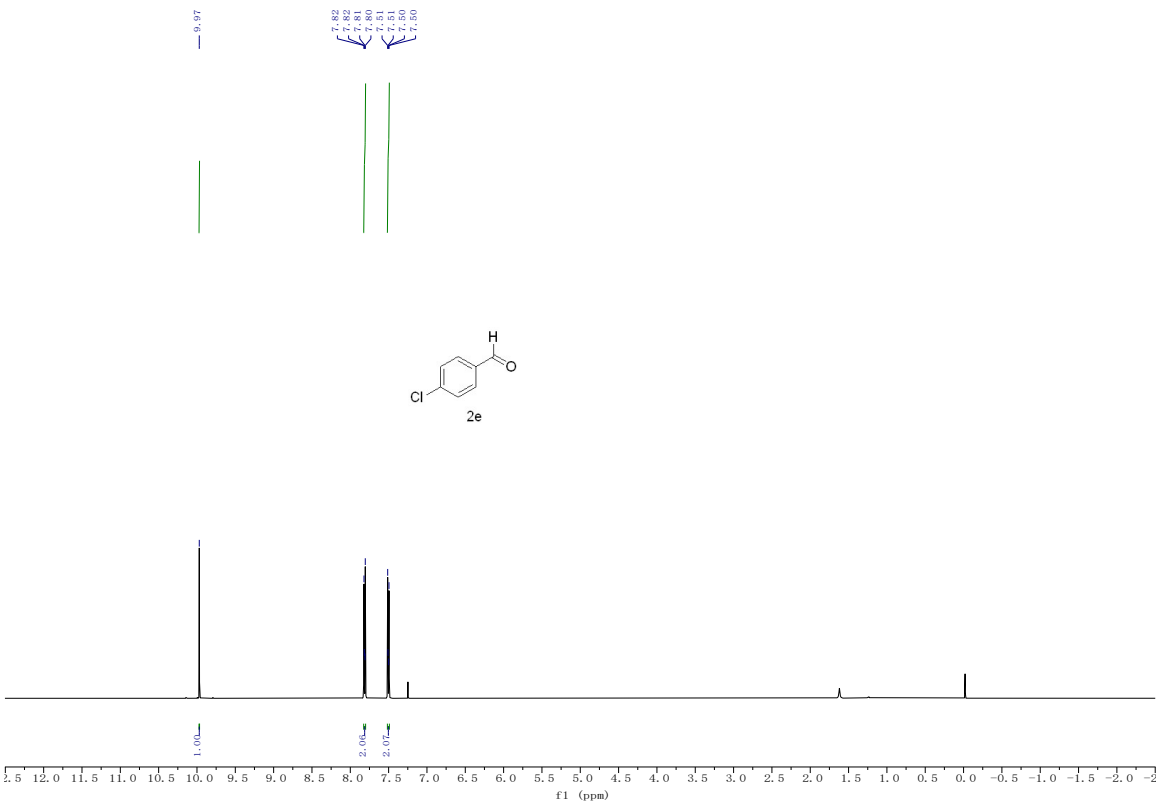


The ^{1}H NMR (400 MHz) and ^{19}F NMR(471 MHz)spectrum for **2d** (using $CDCl_3$ as solvent)

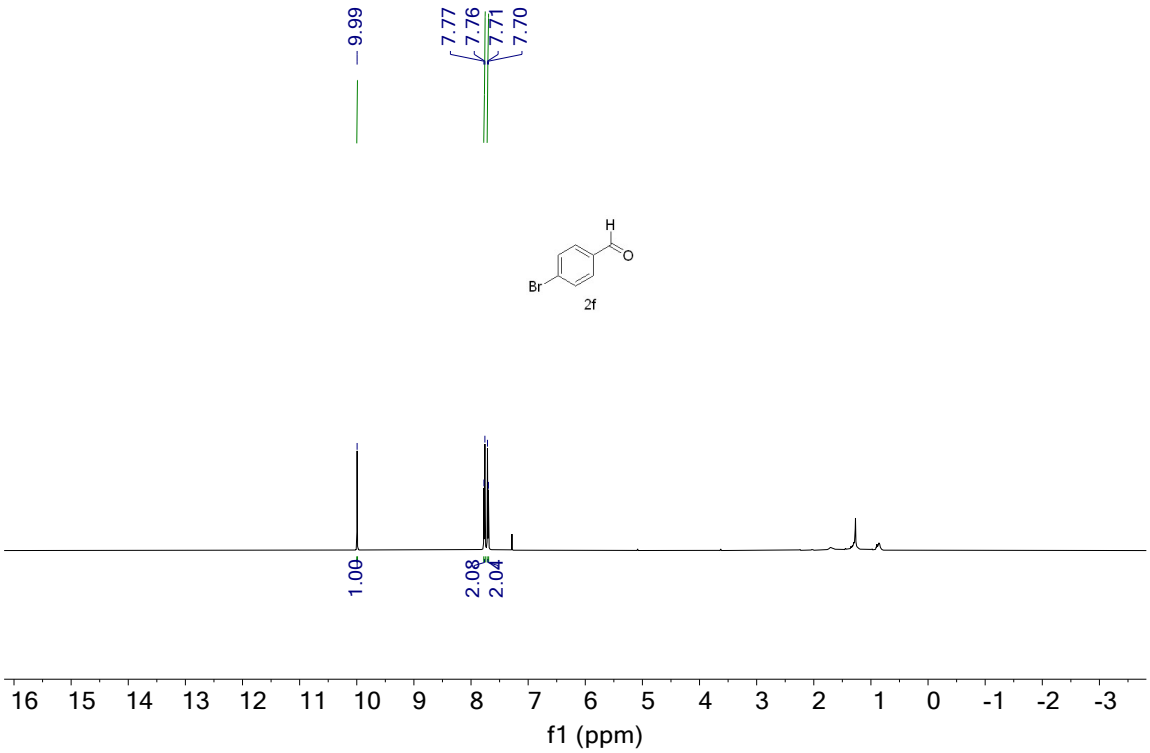




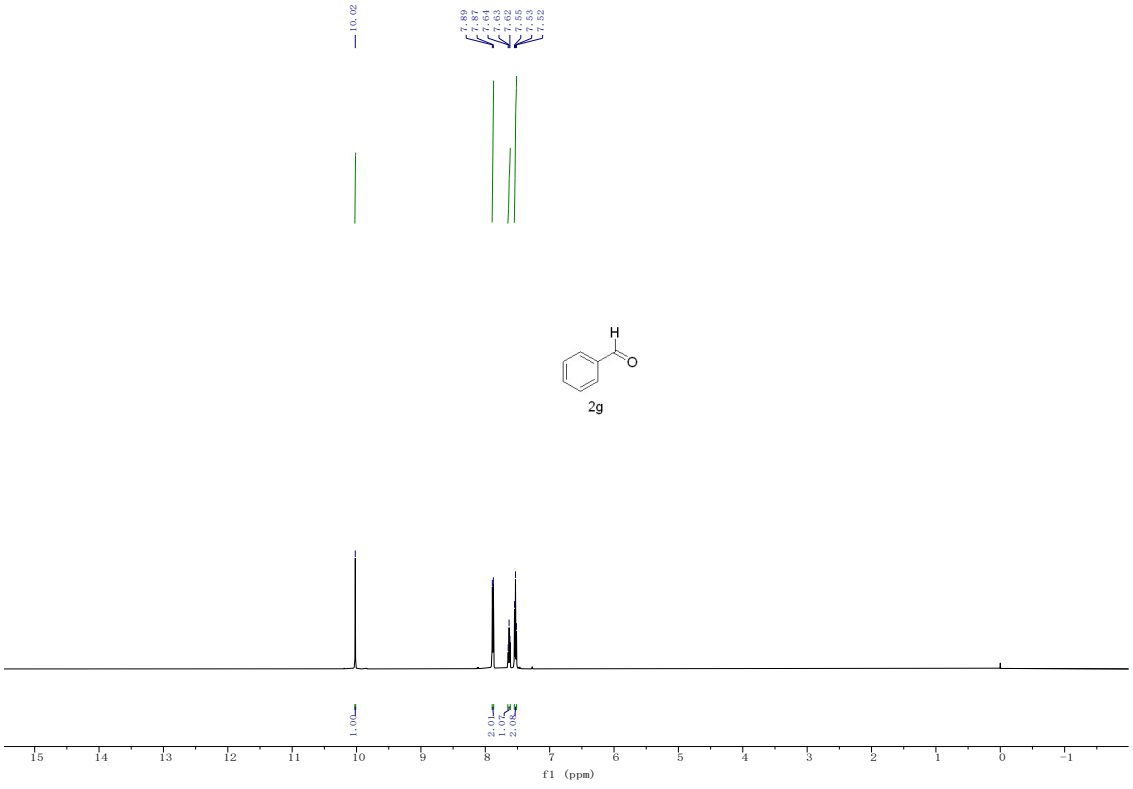
The ^{1}H NMR (500 MHz) spectrum for **2e** (using $CDCl_3$ as solvent)



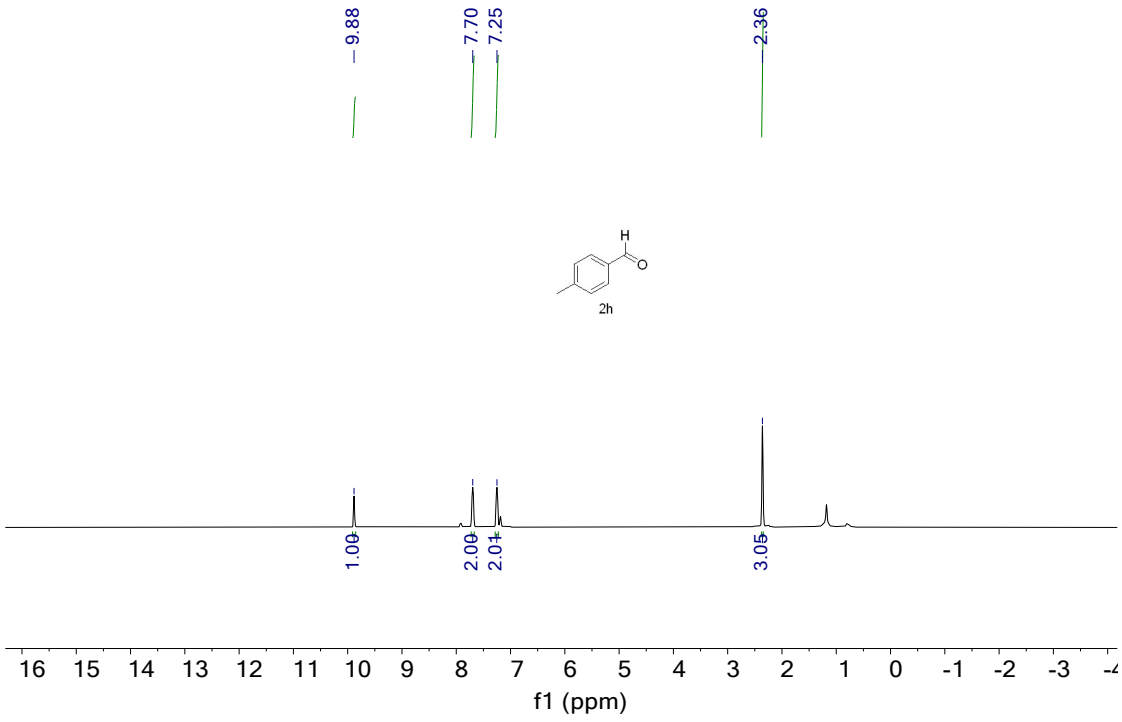
The ^{1}H NMR (500 MHz) spectrum for **2f** (using $CDCl_3$ as solvent)



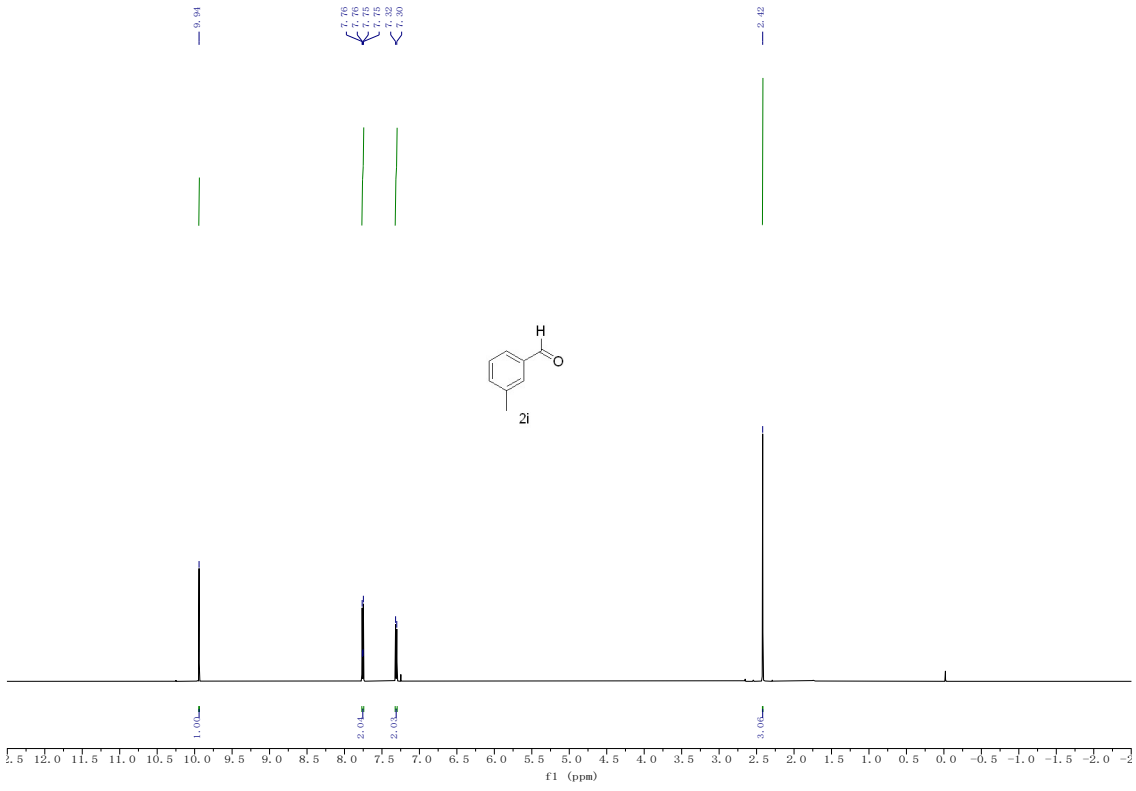
The ^{1}H NMR (500 MHz) spectrum for **2g**(using $CDCl_3$ as solvent)



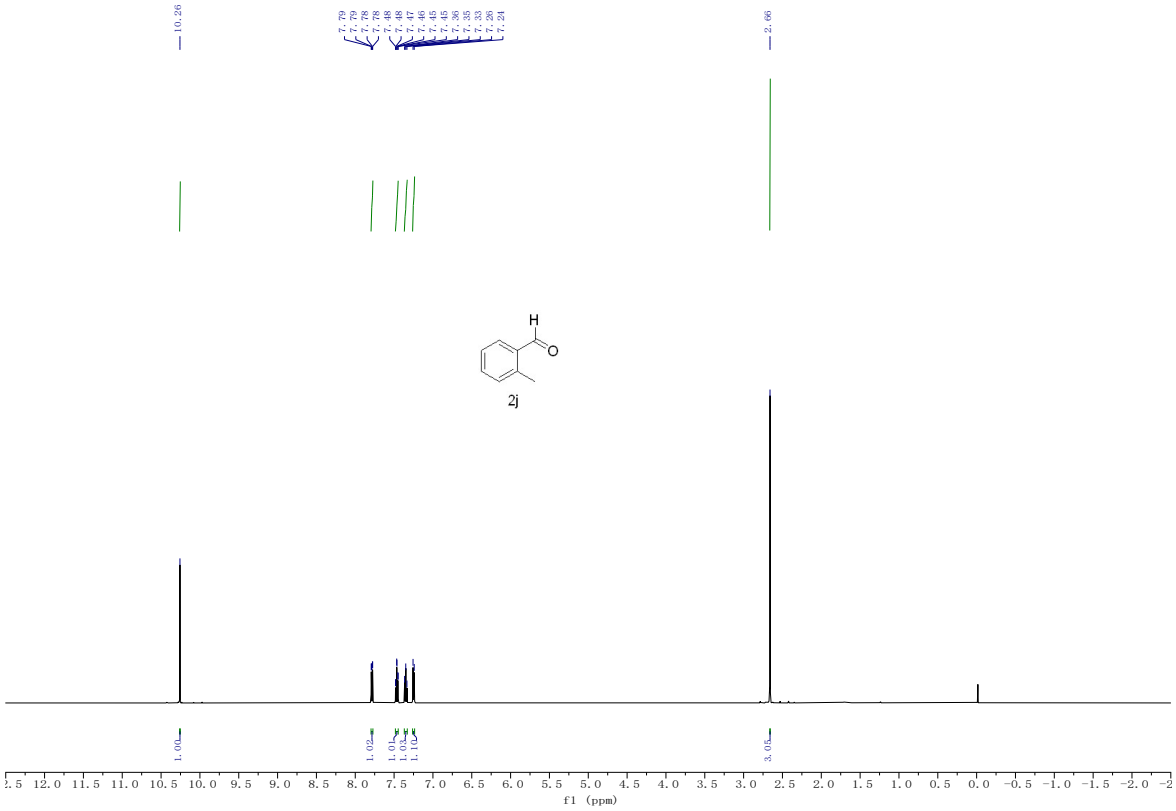
The ^{1}H NMR (500 MHz) spectrum for **2h** (using $CDCl_3$ as solvent)



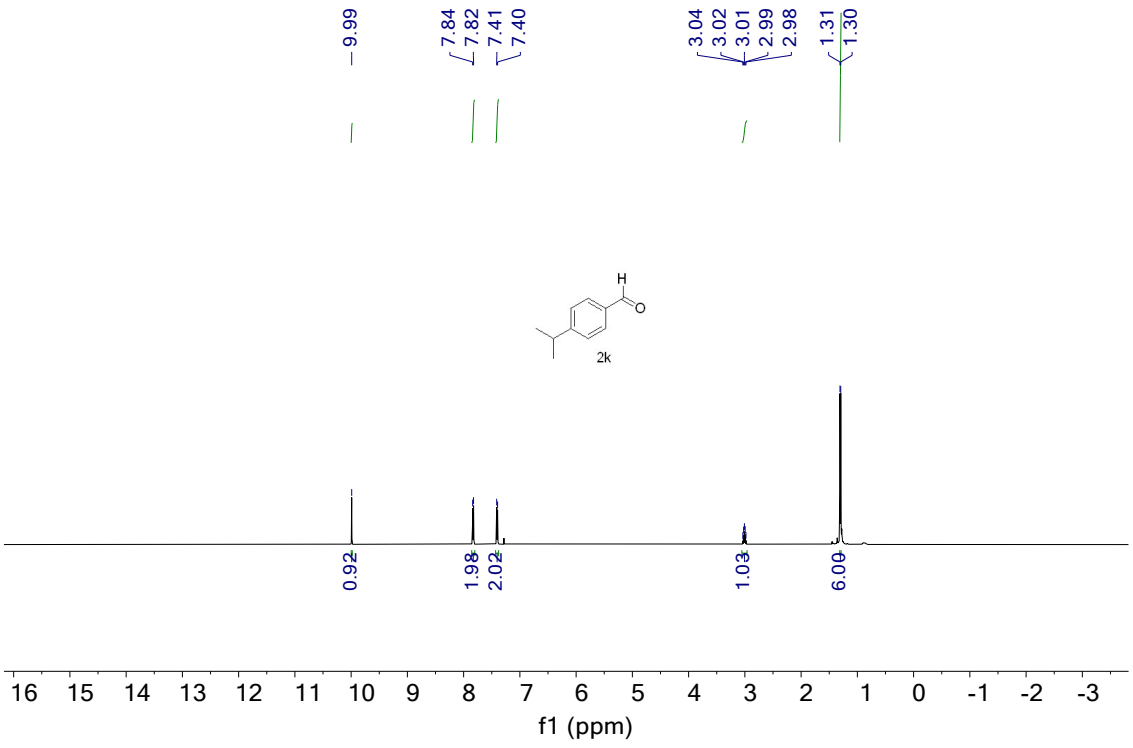
The ^{1}H NMR (500 MHz) spectrum for **2i** (using $CDCl_3$ as solvent)



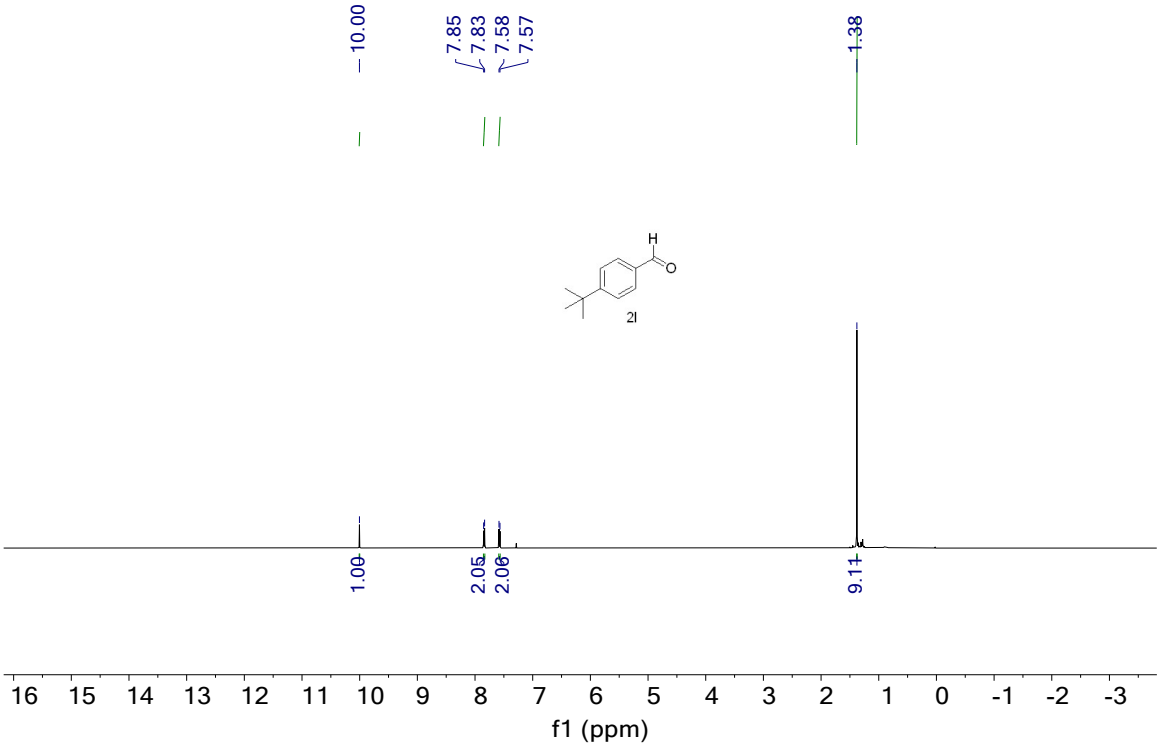
The ^{1}H NMR (400 MHz) spectrum for **2j** (using $CDCl_3$ as solvent)



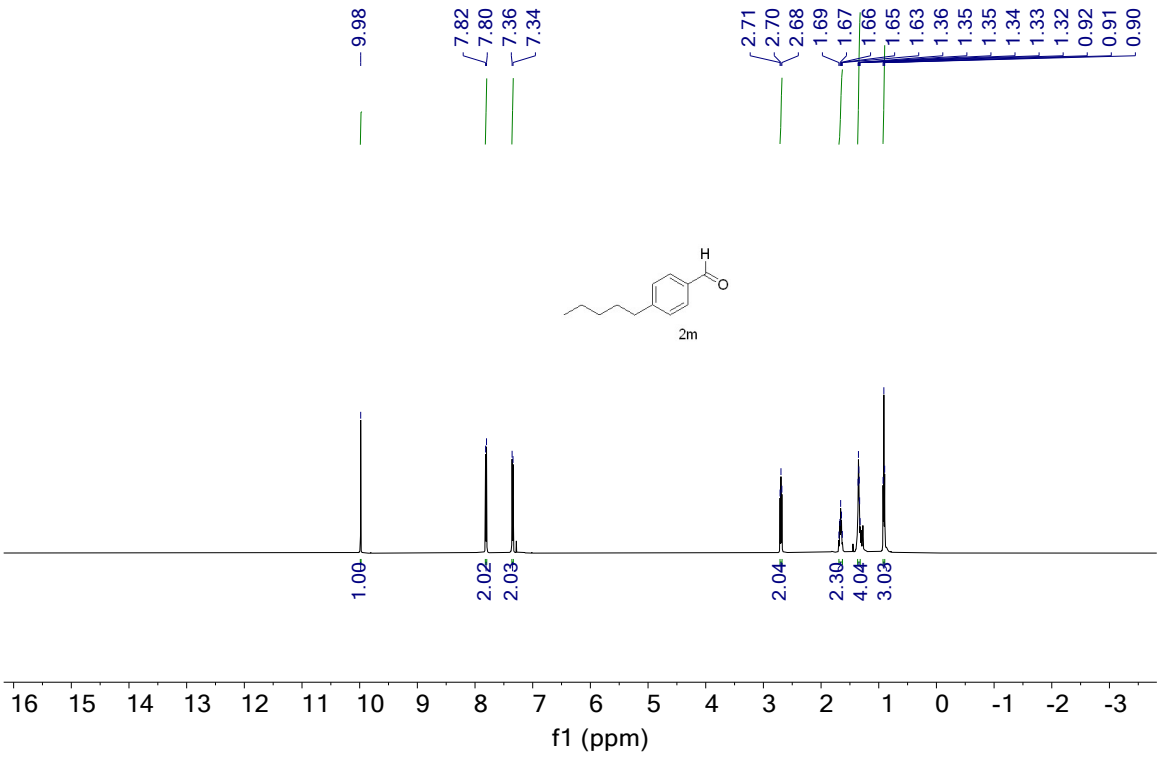
The ^{1}H NMR (500 MHz) spectrum for **2k** (using $CDCl_3$ as solvent)



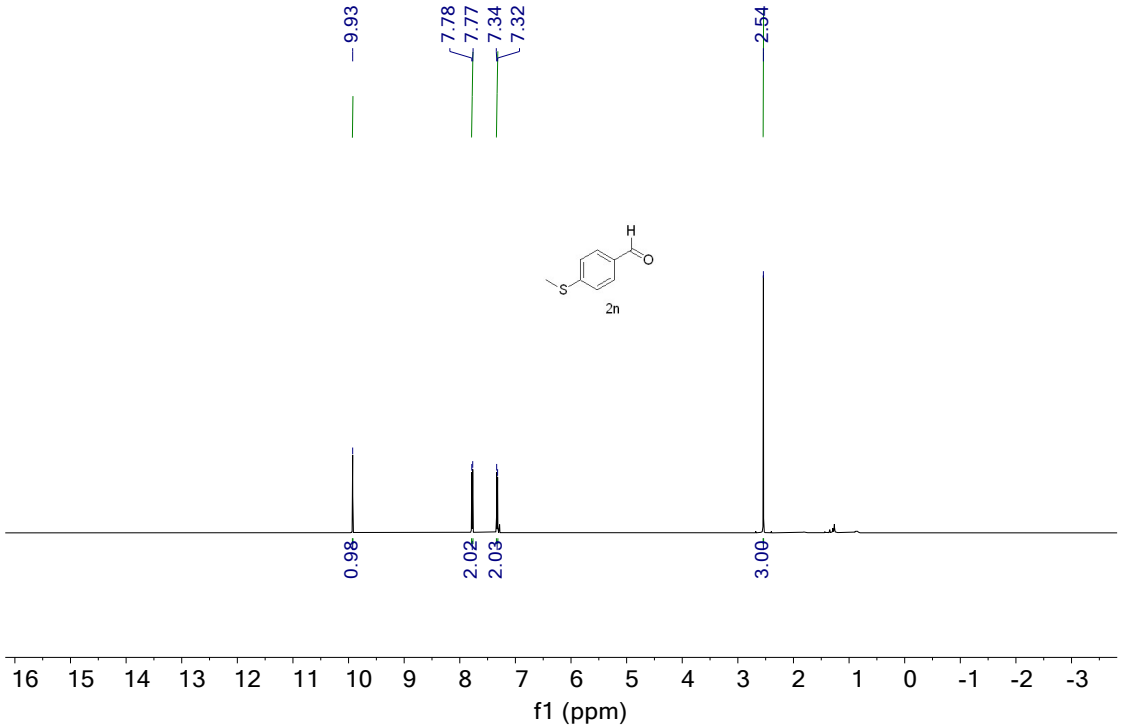
The ^{1}H NMR (500 MHz) spectrum for **2l** (using $CDCl_3$ as solvent)



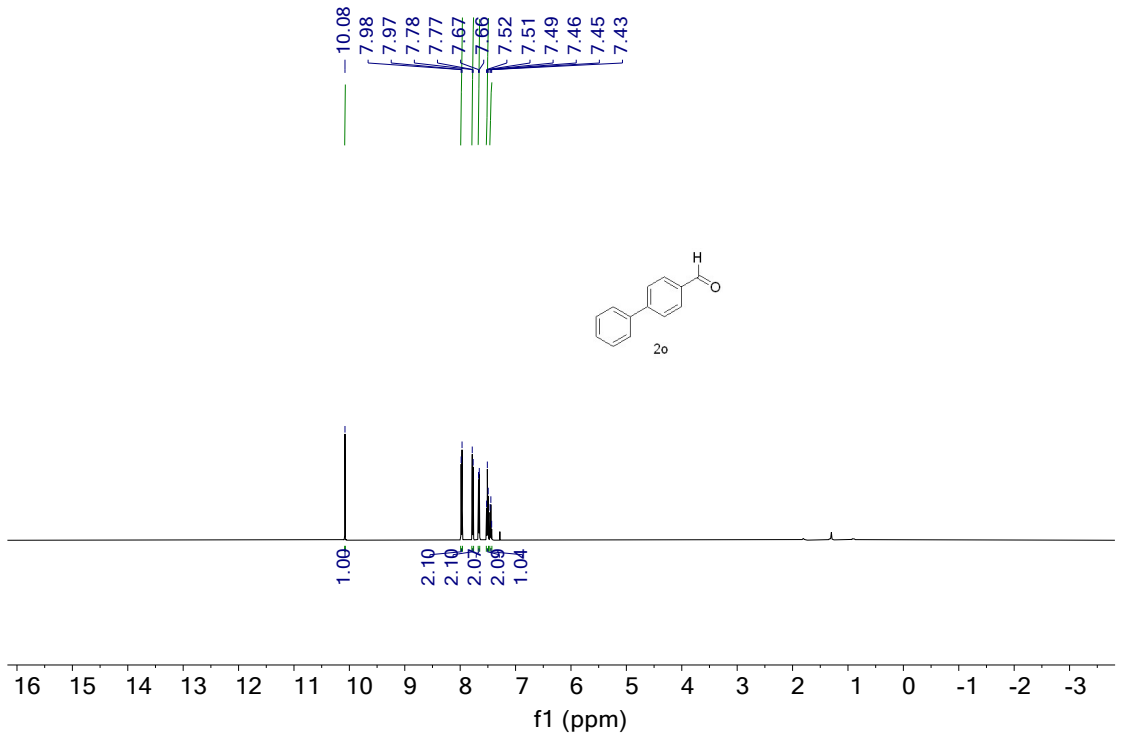
The ^{1}H NMR (400 MHz) spectrum for **2m** (using $CDCl_3$ as solvent)



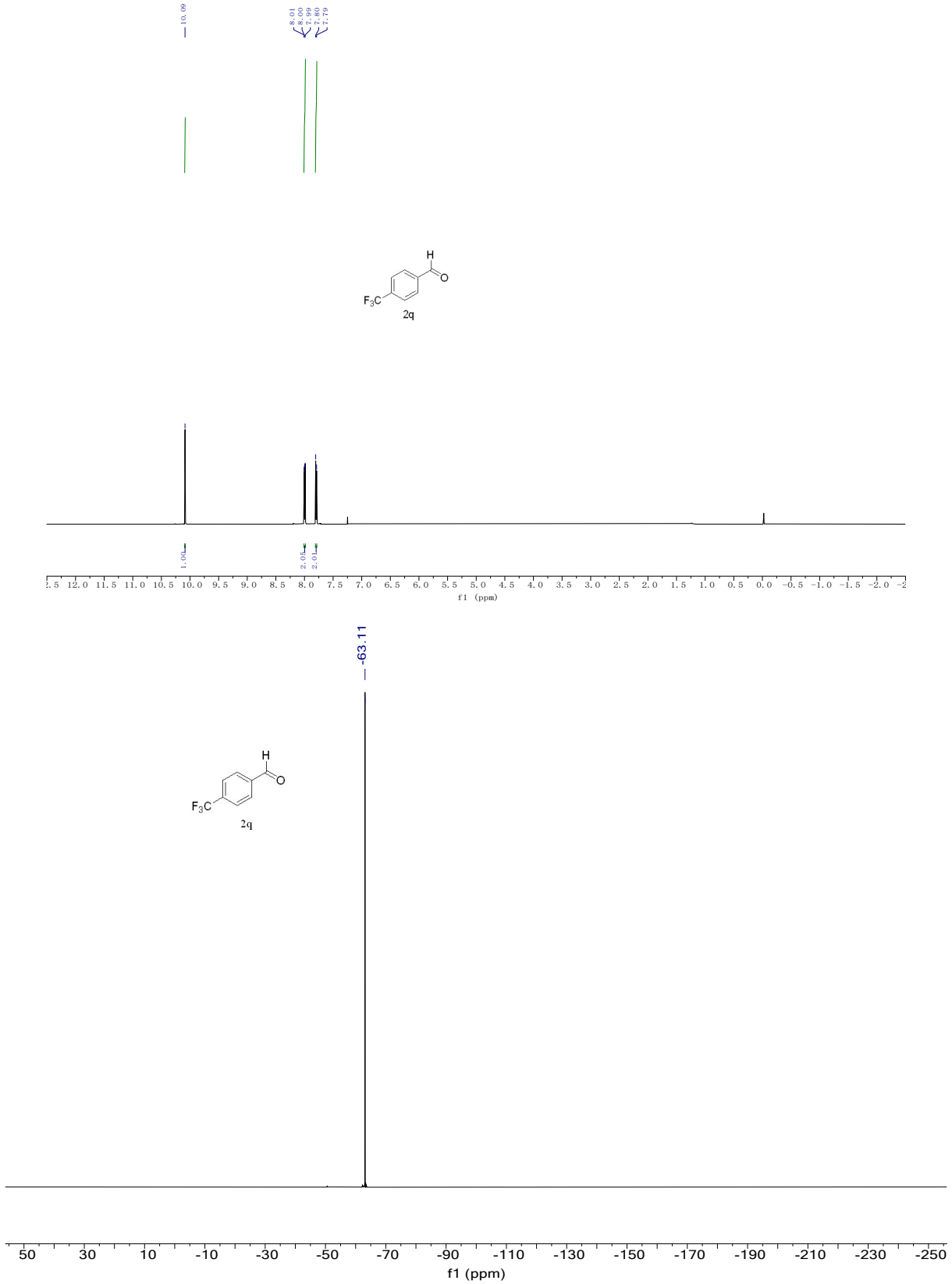
The ^{1}H NMR (500 MHz) spectrum for **2n** (using $CDCl_3$ as solvent)



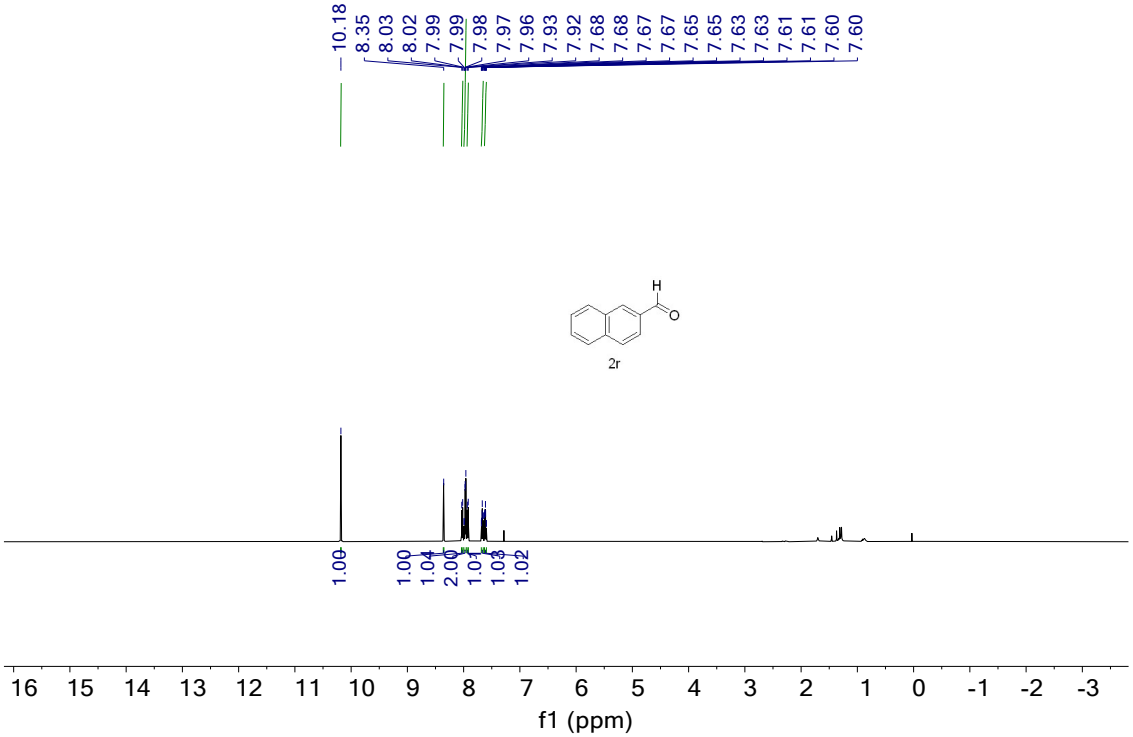
The ^{1}H NMR (500 MHz) spectrum for **2o** (using $CDCl_3$ as solvent)



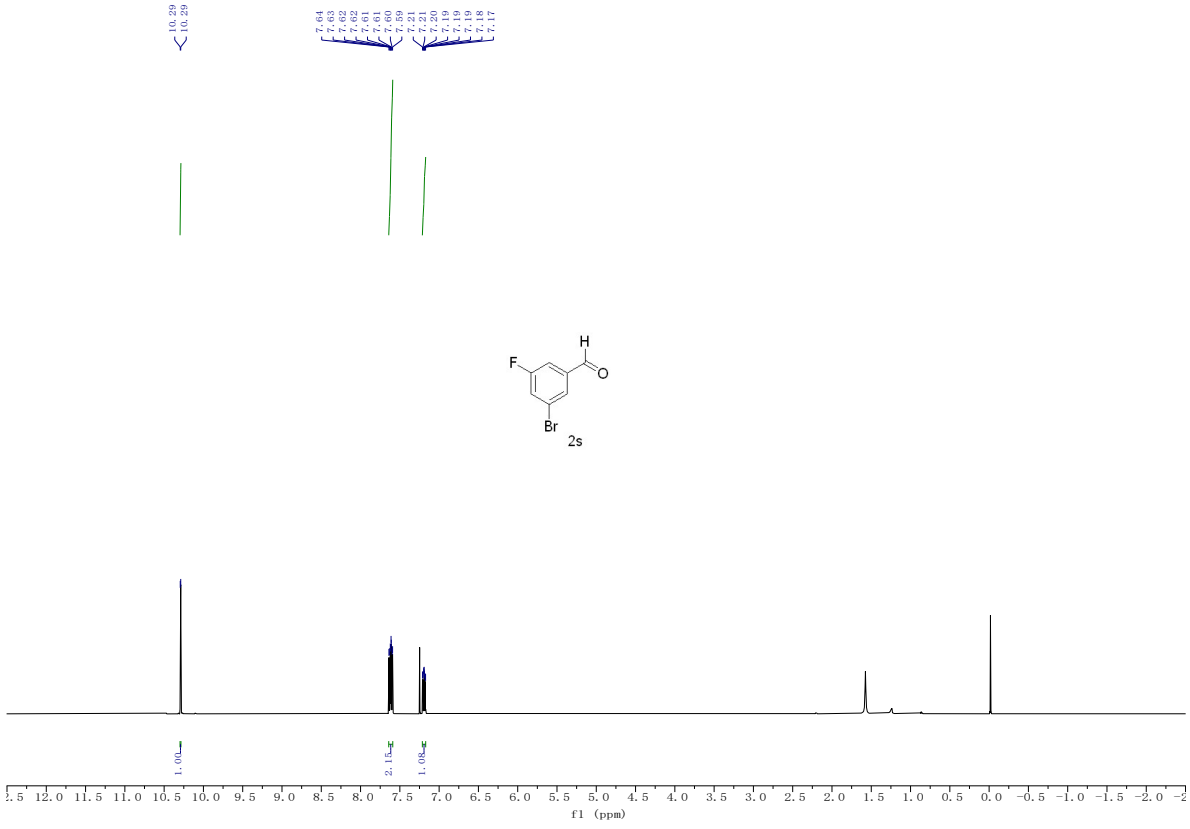
The ^{1}H NMR (500 MHz) and ^{19}F NMR(471 MHz) spectrum for **2q** (using $CDCl_3$ as solvent)

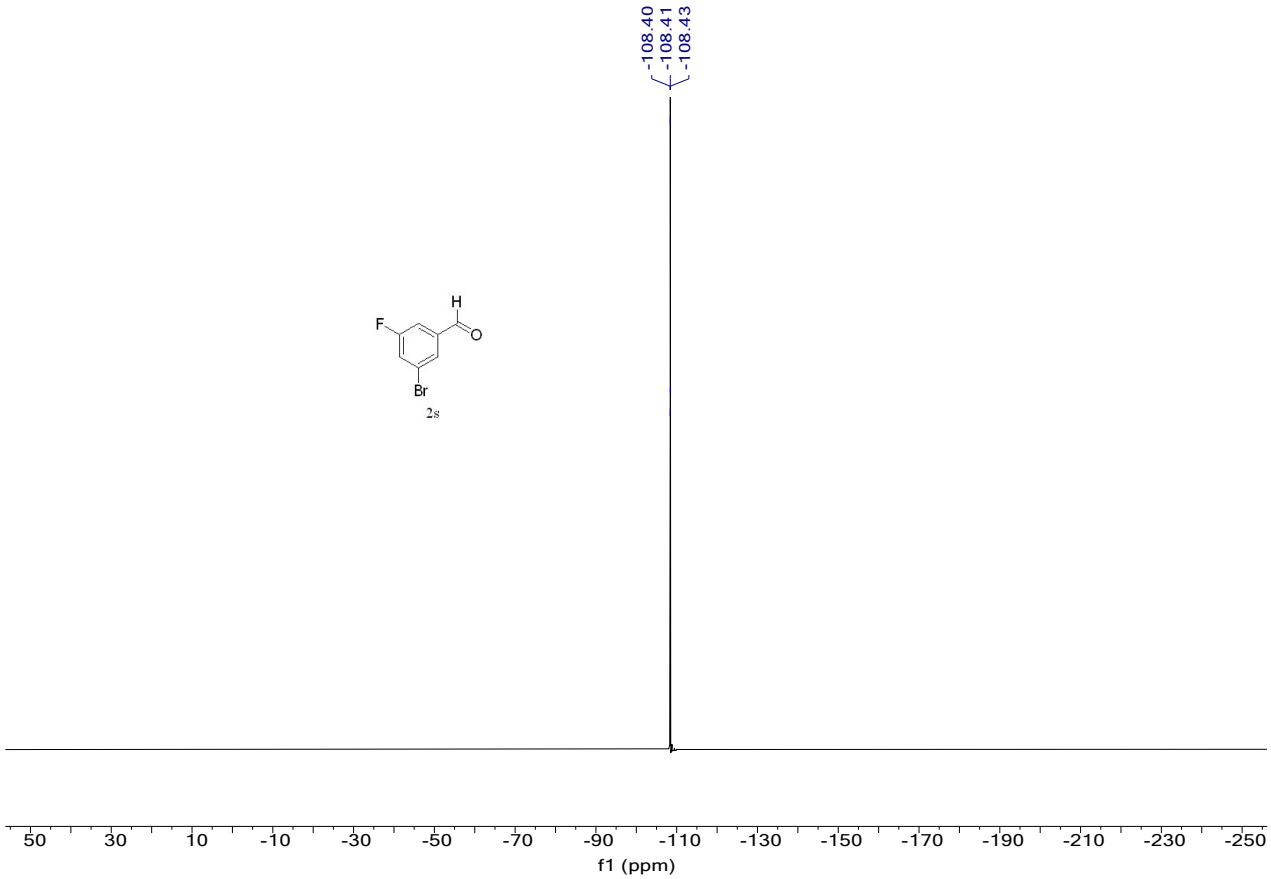


The ^{1}H NMR (500 MHz) spectrum for **2r** (using $CDCl_3$ as solvent)

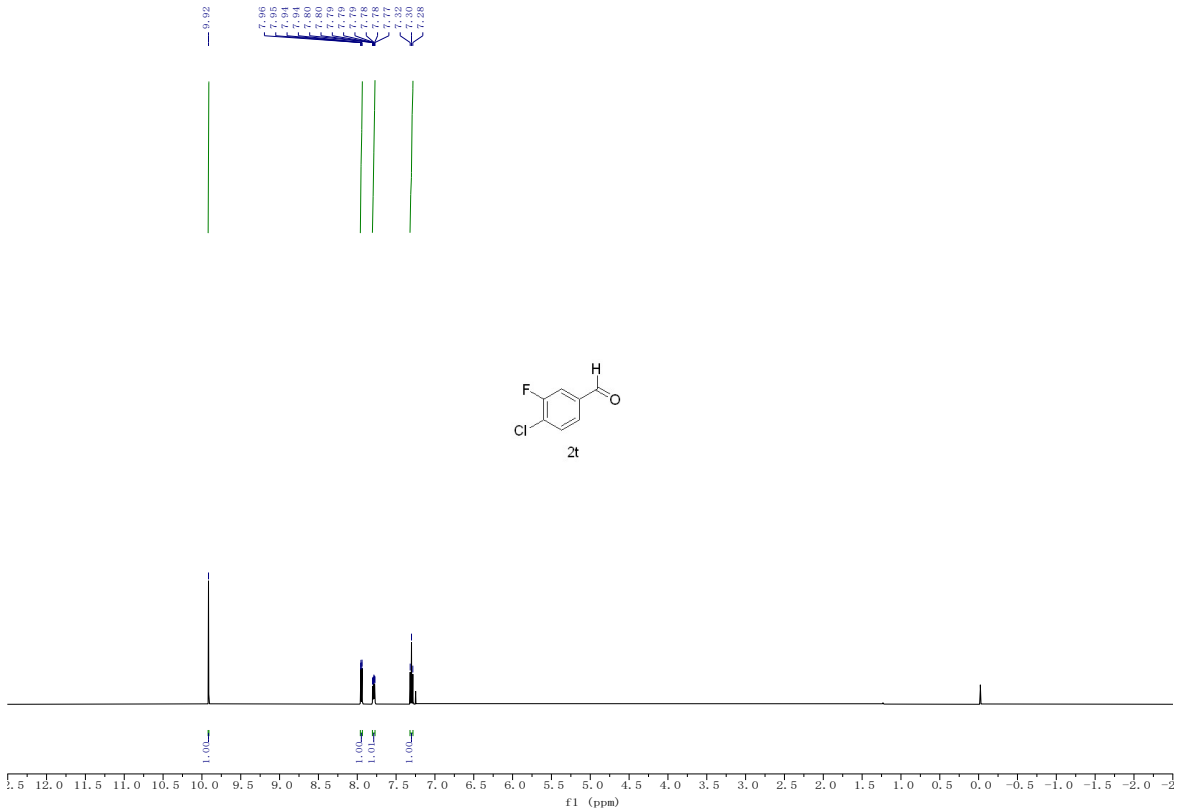


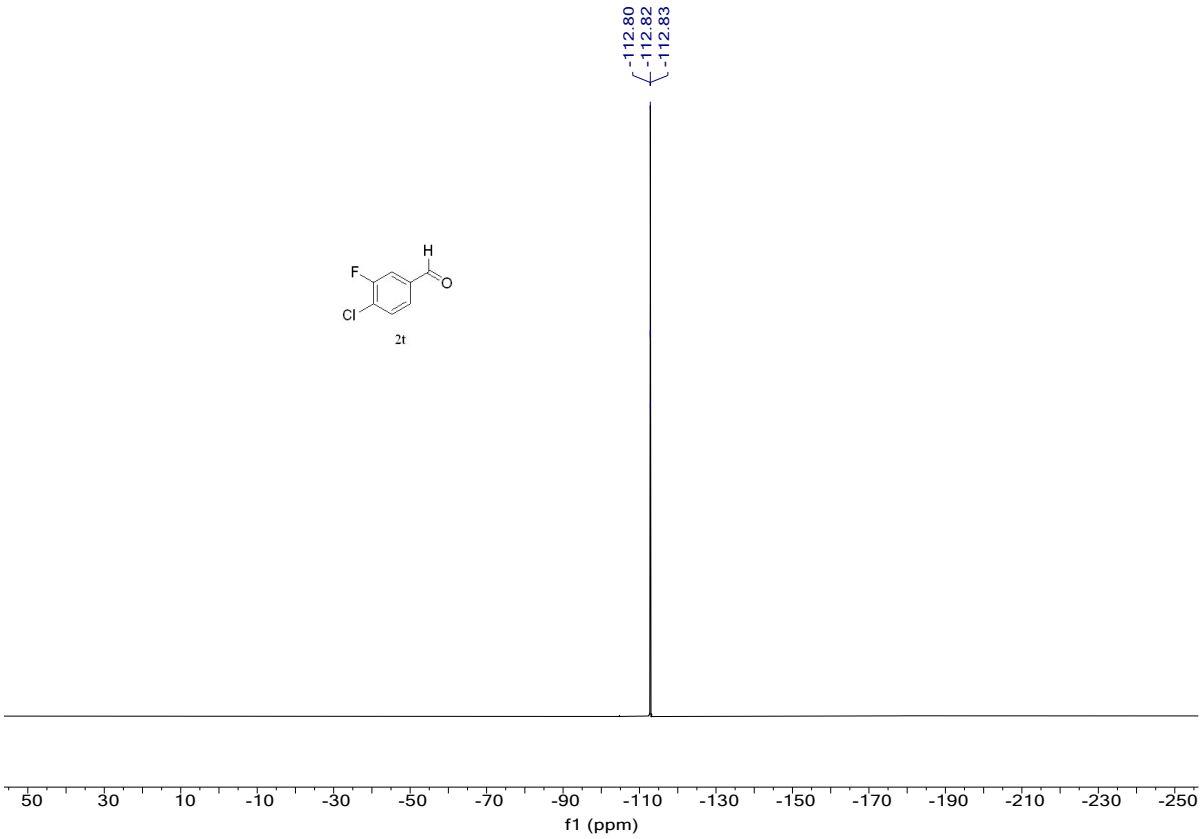
The ^{1}H NMR (400 MHz) and ^{19}F NMR(471 MHz) spectrum for **2s** (using $CDCl_3$ as solvent)



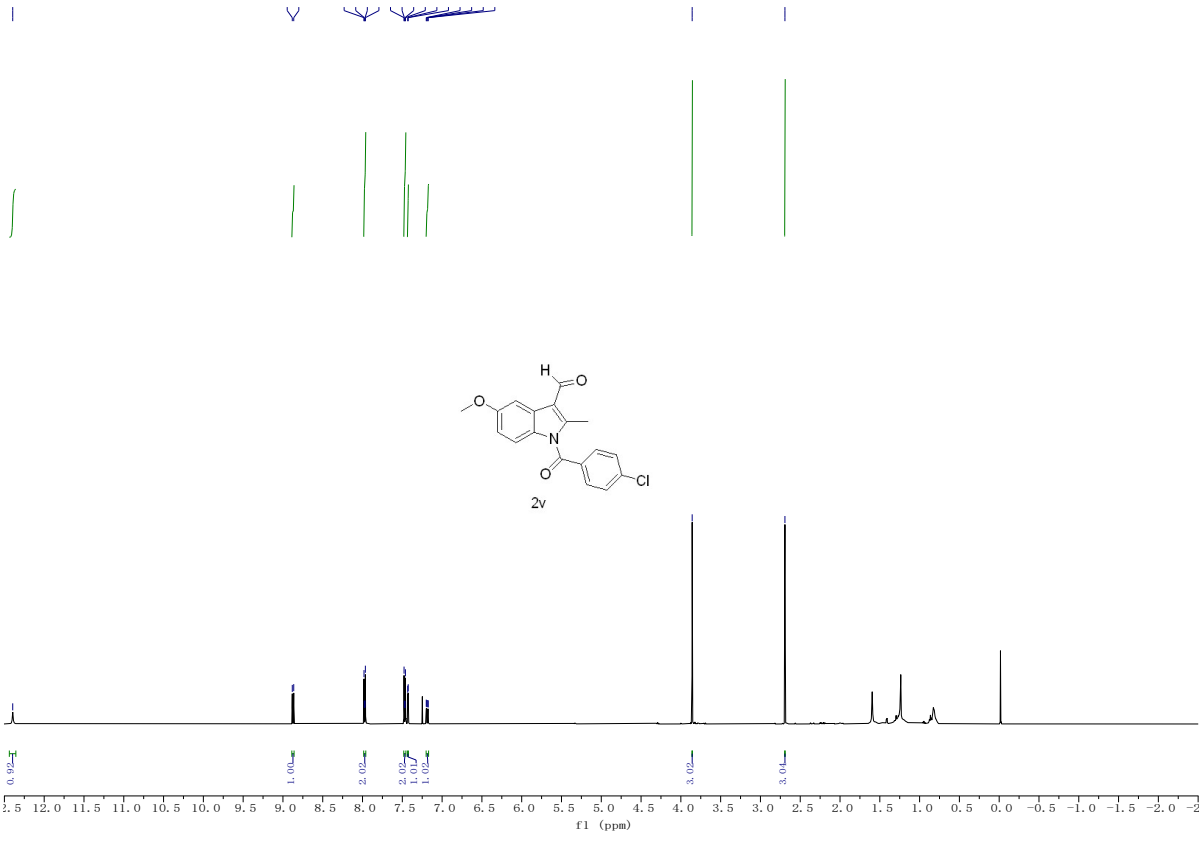


The ^{1}H NMR (400 MHz) and ^{19}F NMR(471 MHz) spectrum for **2t** (using $CDCl_3$ as solvent)

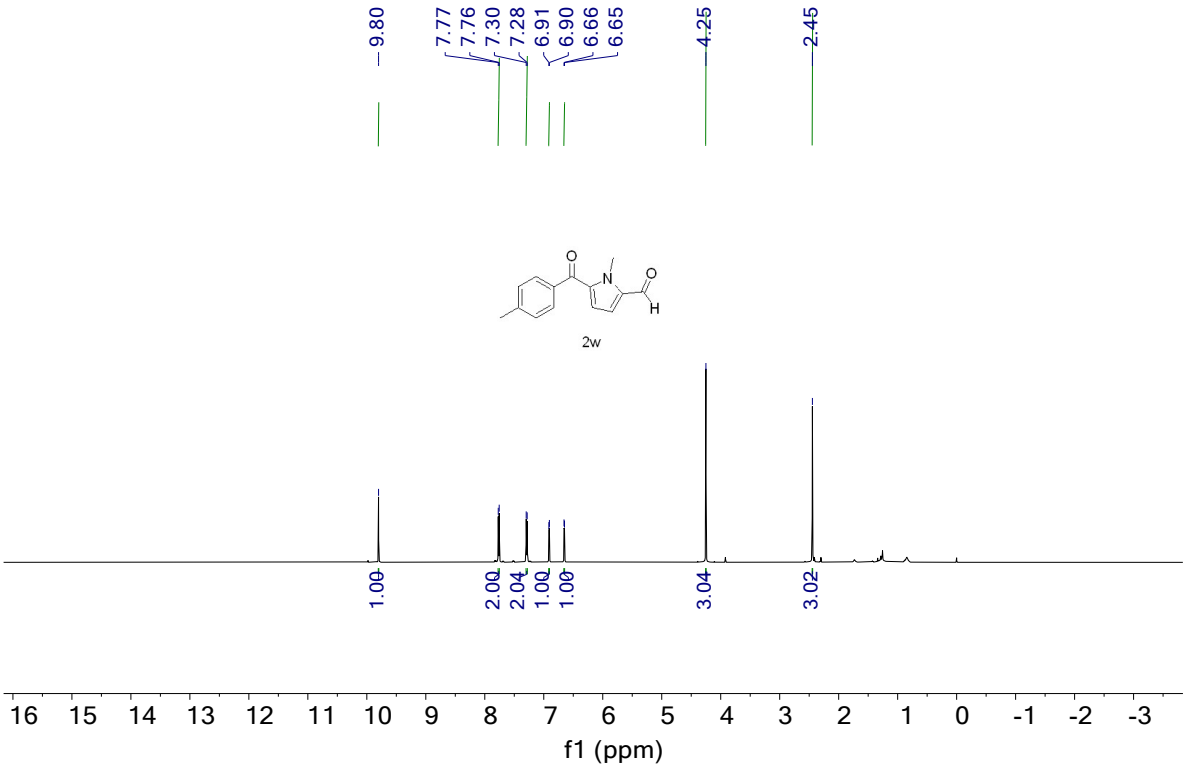




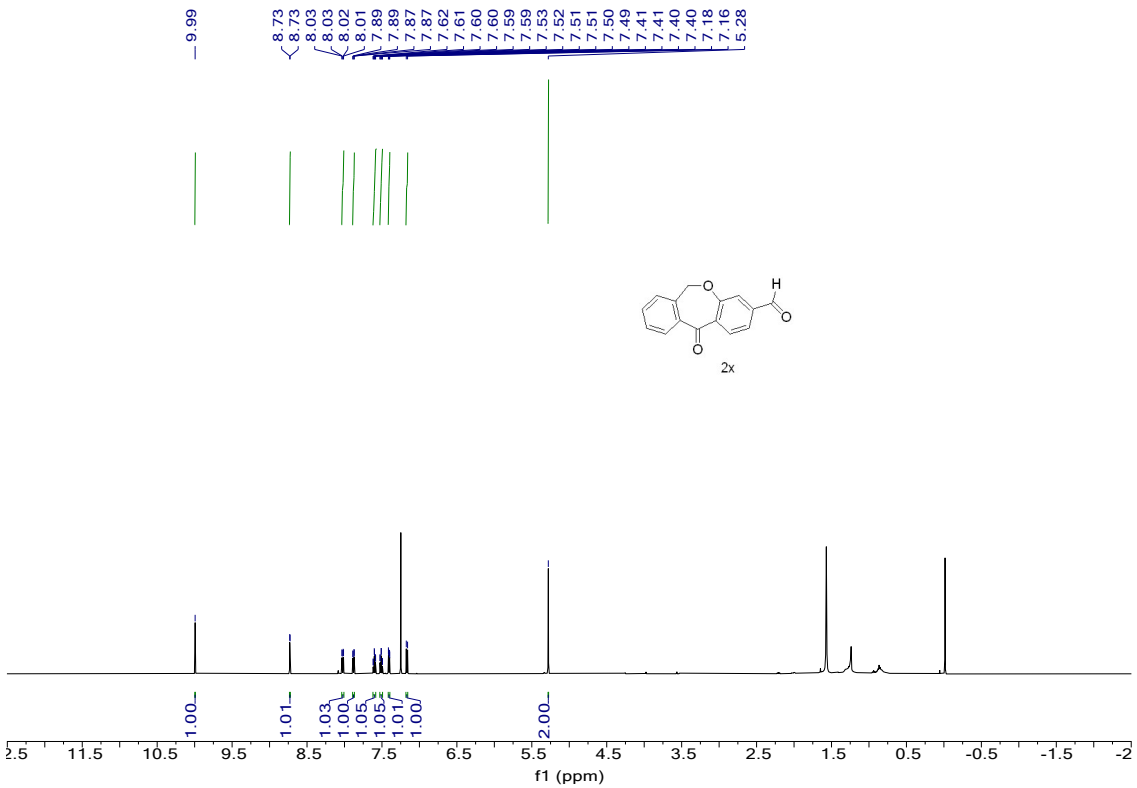
The ^{1}H NMR (500 MHz) spectrum for **2v** (using $CDCl_3$ as solvent)



The ^{1}H NMR (500 MHz) spectrum for **2w** (using $CDCl_3$ as solvent)



The ^{1}H NMR (500 MHz) spectrum for **2x** (using $CDCl_3$ as solvent)



The ^{1}H NMR (500 MHz) spectrum for **4b** (using $CDCl_3$ as solvent)