Supporting Information

Deuteration of Formyl Groups via a Catalytic Radical H/D Exchange Approach

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Table of Contents

1. General information

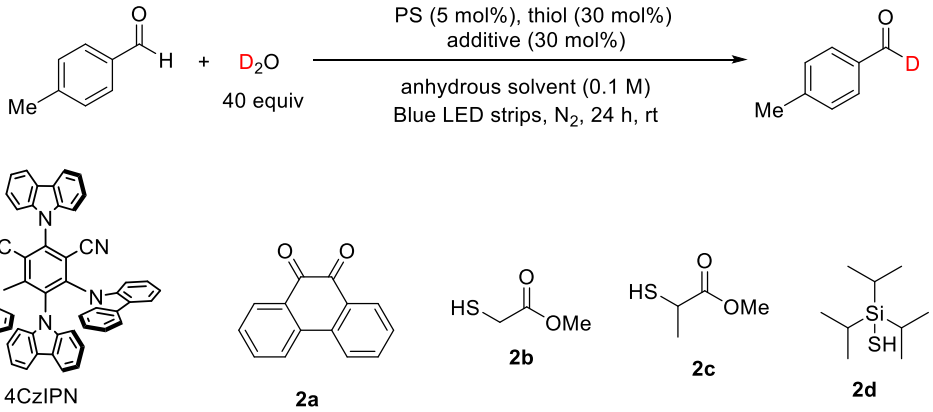
Commercially available reagents were purchased from Sigma Aldrich, Matrix Chemical, AKSci, Alfa Aesar, Acros, AmBeed or TCI, and used as received unless otherwise noted. Merck 60 silica gel was used for chromatography, and Whatman silica gel plates with a fluorescence F254 indicator were used for thin-layer chromatography (TLC) analysis. ^{1}H and ^{13}C NMR spectra were recorded on Bruker Avance 500 MHz or Varian 400 MHz. Chemical shifts in ^{1}H NMR spectra are reported in parts permillion (ppm) relative to residual chloroform (7.26 ppm) or dimethyl sulfoxide (2.50 ppm) as internal standards. 1H NMR data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, quint = =quintet, sext = sextet, m = multiplet, br = broad), coupling constant in Hertz (Hz) and hydrogen numbers based on integration intensities. ^{13}C NMR chemical shifts are reported in ppm relative to the central peak of $CDCl_3$ (77.16 ppm) or $(CD_3)_2SO$ (39.52 ppm) as internal standards. Low-resolution mass spectrometry was performed in Analytical and Biological Mass Spectrometry Center. Both blue LED strip and 34 W Kessil Blue LEDs were purchased from Amazon.

2. General procedure and optimization of reaction conditions

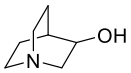
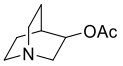
Photocatalyst 4CzIPN was synthesized according to reported procedure.[1]

General procedure: To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added aldehyde (0.2 mmol, 1.0 equiv), photocatalyst 4CzIPN (8 mg, 5 mol%), indicated reagents and 1 mL anhydrous solvents. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for indicated time. After completion of the reaction, the mixture was concentrated. The yield and deuteration ratio were determined by ^{1}H NMR.

Table S1. Optimization of reaction conditions



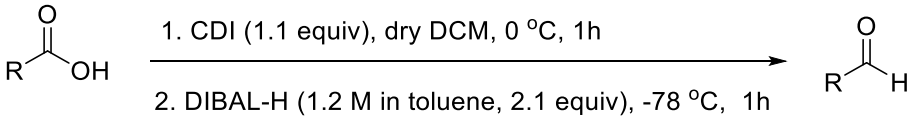
entry	PS	thiol (30 mol%)	addictive (30 mol%)	solvent	y (%)	D (%)
1	**2a**	BnSH	-	MeCN	94	<5
2	4CzIPN	BnSH	-	MeCN	92	30
3	4CzIPN	Benzenethiol	-	MeCN	95	<5
4	4CzIPN	4-Nitrobenzenethiol	-	MeCN	94	<5
5	4CzIPN	BnSSBn	-	MeCN	93	25
6	4CzIPN	BnSH	Na_2CO_3	MeCN	90	<5
7	4CzIPN	**2b**	-	MeCN	90	40
8	4CzIPN	**2c**	-	MeCN	90	33
9	Eosin Y	**2b**	-	MeCN	93	<5
10	Rhodamine B	**2b**	-	MeCN	95	<5
11	Phenylglyoxylic acid	**2b**	-	MeCN	92	<5

12	9,10-Phenanthrenequinone	**2b**		MeCN	94	<5
13	Methylene Blue	**2b**	-	MeCN	93	30
14	4CzIPN	**2b**	2,6-Lutidine	MeCN	89	33
15	4CzIPN	**2b**		MeCN	92	36
16	4CzIPN	**2b**		MeCN	91	19
17	4CzIPN	**2b**		MeCN	92	15
18	4CzIPN	**2b**	Pyridine	MeCN	90	26
19	4CzIPN	**2b**	DMAP	MeCN	91	12
20	4CzIPN	**2b**	Quinoline	MeCN	88	14
21	4CzIPN	**2b**	Sodium dodecyl sulfate	MeCN	88	40
22	4CzIPN	**2b**	$PhCO_2Na$	MeCN	93	53
23	4CzIPN	**2b**	$PhCO_2Na$	DMF	93	<5
24	4CzIPN	**2b**	$PhCO_2Na$	$PhCF_3$	96	<5
25	4CzIPN	**2b**	$PhCO_2Na$	Acetone	97	40
26	4CzIPN	**2b**	$PhCO_2Na$	THF	94	<5
27	4CzIPN	**2b**	$PhCO_2Na$	EtOAc	94	65
28[a]	4CzIPN	**2b**	$PhCO_2Na$	EtOAc	92	79
29[b]	4CzIPN	**2b**	$PhCO_2Na$	EtOAc	96	85
30[c]	4CzIPN	**2b**	$PhCO_2Na$	EtOAc	95	95
31	4CzIPN	**2d**	$PhCO_2Na$	EtOAc	95 (91[d])	97
31[e]	4CzIPN	**2d**	$PhCO_2Na$	EtOAc	94	72
31[f]	4CzIPN	**2d**	$PhCO_2Na$	EtOAc	95	93

[a]Reaction time: 40h; [b]Reaction time: 48h; [c]Reaction time 72h; [d]Isolated yield; [e] Reaction time 10h; [f] Reaction time 20h. Y: yield; D: deuteration ratio.

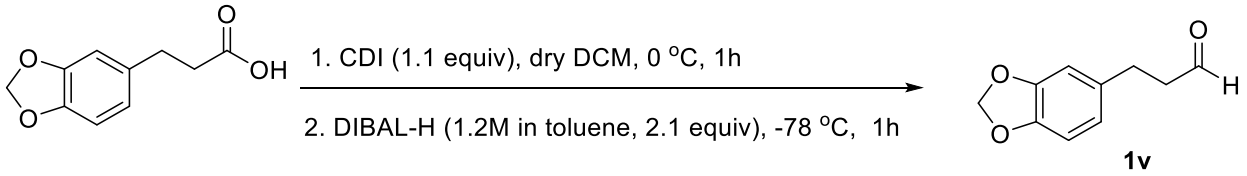
3. Preparation of aldehyde substrates 1.

3.1 General procedure for the synthesis of compound (1v, 1y, 1ab)



General procedure: To a dried 10 mL Schlenk tube was added acid (1 mmol, 1.0 equiv) and capped with rubber stopper. The tube is purged with N_2 and protected by N_2 in balloon. 5 mL anhydrous DCM was added under N_2. The solution was cooled to 0 °C (ice bath) and 178mg (1,1 mmol, 1.1 eq) 1,1'-carbonyldiimidazole (CDI) were added. After stirring for 60 min, the colorless reaction solution was cooled to -78 °C (dry ice/acetone bath) for 15 min. Subsequently, 1.75 mL (2.1 mmol, 2.1 eq) DIBAL-H solution (1.2 M in toluene) were added dropwise with a syringe throughout 10 min. The reaction mixture was stirred at -78 °C for 1h. The reaction mixture was quenched by the addition of 2mL EtOAc followed by 5 mL tartaric acid solution (25 % in H_2O) under vigorous stirring. The mixture was warmed up to RT and stirred vigorously for 15 min. The mixture was extracted with EtOAc (3 x 10 mL) and the combined organic extracts were washed with 1 M HCl (1 x 10 mL), saturated $NaHCO_3$ (1 x 10 mL) and brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product purified by flash chromatography on silica gel.

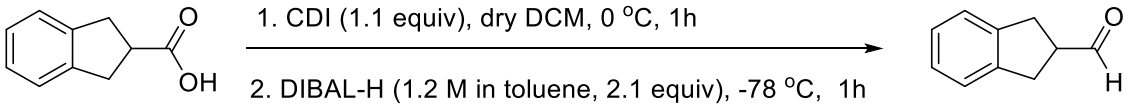
Preparation of 3-(benzo[*d*][1,3]dioxol-5-yl)propanal (1v)[2]



1v

This substrate was synthesized following the *general procedure*. The crude product was purified by flash chromatography on silica gel (acetone/hexane = 1/9) to afford product (139 mg, 78% yield) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (1H, t, *J* = 1.2 Hz), 6.72 (1H, d, *J* = 7.8 Hz), 6.67 (d, *J* = 1.5 Hz, 1H), 6.63 (dd, *J* = 7.9, 1.4 Hz, 1H), 5.91 (s, 2H), 2.87 (2H, t, *J* = 7.4 Hz), 2.72 (t, *J* = 7.3 Hz, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.32, 147.88, 146.04, 134.15, 121.15, 108.83, 108.36, 101.10, 45.55, 28.01.

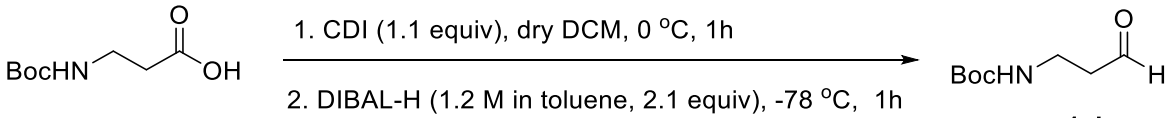
Preparation of 2,3-dihydro-1*H*-indene-2-carbaldehyde (1y) [3]



1y

This substrate was synthesized following the *general procedure*. The crude product was purified by flash chromatography on silica gel (Et_2O/hexane = 1/19) to afford product (112 mg, 77% yield) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.78 (d, *J* = 1.3 Hz, 1H), 7.25 (dd, *J* = 5.3, 3.5 Hz, 2H), 7.18 (dd, *J* = 5.4, 3.3 Hz, 2H), 3.35–3.25 (m, 3H), 3.23-3.16 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.84, 141.17, 126.86, 124.68, 50.70, 32.99.

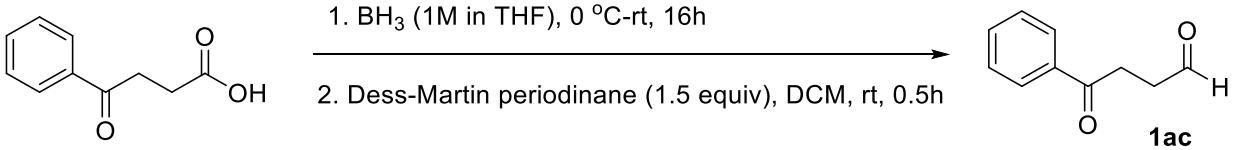
Preparation of *tert*-butyl (3-oxopropyl)carbamate (1ab) [4]



1ab

This substrate was synthesized following the *general procedure*. The crude product was purified by flash chromatography on silica gel (EtOAc/hexane = 1/3) to afford product (151 mg, 87% yield) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.70 (s, 1H), 5.05 (br, 1H), 3.32 (q, *J* = 6.0 Hz, 2H), 2.60 (t, *J* = 5.9 Hz, 2H), 1.32 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.43, 155.83, 79.25, 44.22, 34.02, 28.29.

3.2 Preparation of 4-oxo-4-phenylbutanal (1ac) [5]

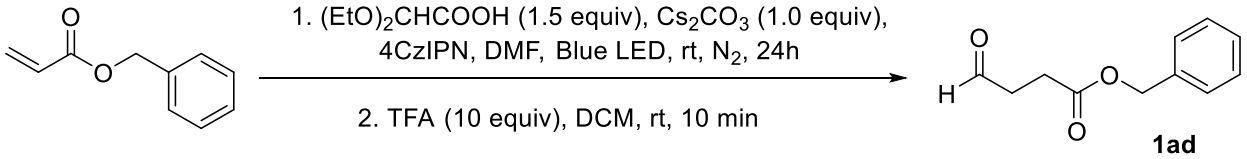


1ac

At 0 °C, to the solution of 4-oxo-4-phenylbutanoic acid (356 mg, 2 mmol, 1.0 equiv) in anhydrous THF 5 mL was added BH_3 (1M in THF, 6 mL, 6 mmol, 3.0 equiv) dropwise for 10 min. The reaction was warmed up to room temperature and stirred for 16h. After the completion of reaction, the solvent was removed under vacuum. To the crude mixture was added DCM 5 mL followed by 1.3g (3 mmol, 1.5 equiv) Dess-Martin periodinane, the reaction was stirred at room temperature for 0.5 h indicated by TLC. 5 mL Saturated sodium thiosulfate solution was added to flask to quench reaction and followed by 5 mL saturated $NaHCO_3$ solution. The mixture was stirred for 15 min till it turns to be clear. The reaction was extracted with DCM (3 x 10 mL) and the combined organic extracts were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel (EtOAc/hexane = 1/2) to afford product 194 mg (60% yield for two steps) as colorless oil. ^{1}H NMR

(400 MHz, $CDCl_3$) δ 9.91 (s, 1H), 7.99 (d, *J* = 7.4 Hz, 2H), 7.58 (t, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 3.33 (t, *J* = 6.3 Hz, 2H), 2.94 (t, *J* = 6.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.75, 197.94, 136.56, 133.47, 128.79, 128.21, 37.76, 31.16.

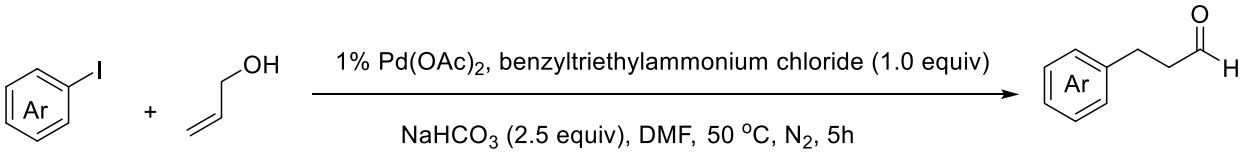
3.3 Synthesis of benzyl 4-oxobutanoate (1ad)



1ad

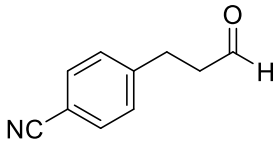
To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added benzyl acrylate (162mg, 1.0 mmol, 1.0 equiv), 2,2-diethoxyacetic acid (222mg, 1.5 mmol, 1.5 equiv), photocatalyst 4CzIPN (40mg, 5 mol%), Cs_2CO_3 (325mg, 1 mmol, 1.0 equiv), DMF (5 mL, 0.2M). The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 24h. After completion of the reaction, the mixture was diluted by adding 10 ml EtOAc and 30 ml water. The reaction was extracted with EtOA (3 x 10 mL) and the combined organic extracts were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. 5 ml DCM was added to crude residue followed by 0.75 mL TFA (10 equiv). The reaction mixture was stirred at room temperature for 10 min. 10 mL saturated $NaHCO_3$ solution was added slowly to quench the reaction. The reaction mixture was extracted with DCM (3 x 10 mL) and the combined organic extracts were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel (acetone/hexane = 1/4) to afford product 163 mg (85% yield for two steps) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 1H), 7.34 (s, 5H), 5.13 (s, 2H), 2.79 (t, *J* = 6.1 Hz, 2H), 2.67 (t, *J* = 6.5 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.95, 172.11, 135.77, 128.60, 128.60, 128.32, 128.23, 66.63, 38.50, 26.61.

3.4 General procedure for the synthesis of substrates (1ae, 1aj, 1ak)



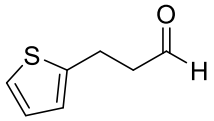
To a 25 mL Schlenk tube equipped with a stir bar was added with substituted iodobenzene (2.0 mmol, 1.0 equiv), $Pd(OAc)_2$ (4.5 mg, 0.02 mmol, 1 mol%), benzyltriethylammonium chloride (454mg, 2 mmol, 1.0 equiv), $NaHCO_3$ (420 mg, 5 mmol, 2.5 equiv), allyl alcohol (174mg, 1.5 equiv) and DMF (6.0 mL). The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The reaction mixture was stirred at 50 °C for 5 h. Upon cooling to room temperature, the reaction mixture was filtrated through a pad of celite, washed with 50 mL of ethyl acetate and washed with brine (2*10.0 mL). The organic layer was separated, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography on silica gel to provide the corresponding products.

Synthesis of 4-(3-oxopropyl)benzonitrile (1ae)[6]



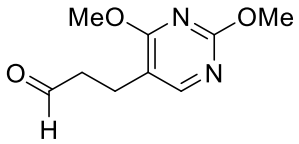
This substrate was synthesized following the *general procedure*. The crude product was purified by flash chromatography on silica gel (EtOAc/hexane = 1/3) to afford product (277 mg, 87% yield) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 7.52 (d, *J* = 7.5 Hz, 2H), 7.27 (d, *J* = 7.5 Hz, 2H), 2.96 (t, *J* = 7.0 Hz, 2H), 2.78 (t, *J* = 7.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.36, 146.16, 132.34, 129.23, 118.87, 110.16, 44.47, 28.02.

Synthesis of 3-(thiophen-2-yl)propanal (1aj)[7]



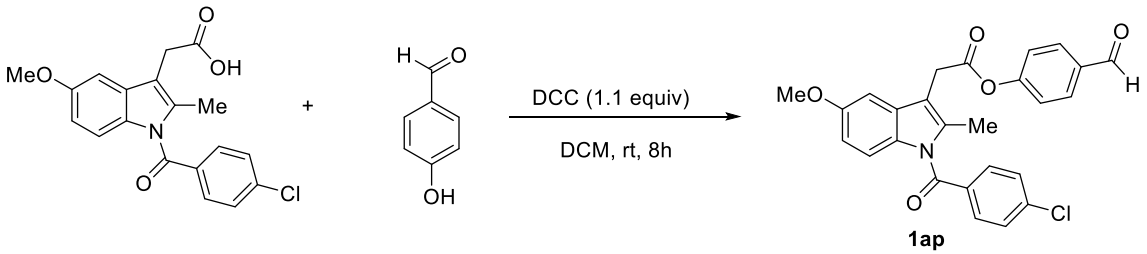
This substrate was synthesized following the *general procedure*. The crude product was purified by flash chromatography on silica gel (EtOAc/hexane = 1/9) to afford product (255 mg, 91% yield) as orange oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 1H), 7.13 (d, *J* = 4.9 Hz, 1H), 6.92 (s, 1H), 6.82 (s, 1H), 3.18 (t, *J* = 7.3 Hz, 2H), 2.83 (t, *J* = 7.2 Hz, 2H).

Synthesis of 3-(2,4-dimethoxypyrimidin-5-yl)propanal(1ak)



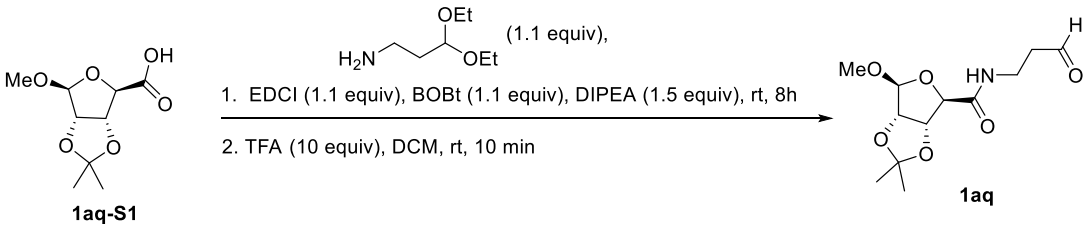
This substrate was synthesized following the *general procedure*. The crude product purified by flash chromatography on silica gel (EtOAc/hexane = 1/1) to afford product (353 mg, 90% yield) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.77 (s, 1H), 8.01 (s, 1H), 3.96 (s, 3H), 3.93 (s, 3H), 2.76 (t, *J* = 6.4 Hz, 2H), 2.68 (t, *J* = 6.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.19, 169.35, 164.46, 157.33, 113.51, 54.77, 54.75, 53.99, 53.97, 42.98, 19.59.

3.5 Synthesis of 4-formylphenyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate (1ap)



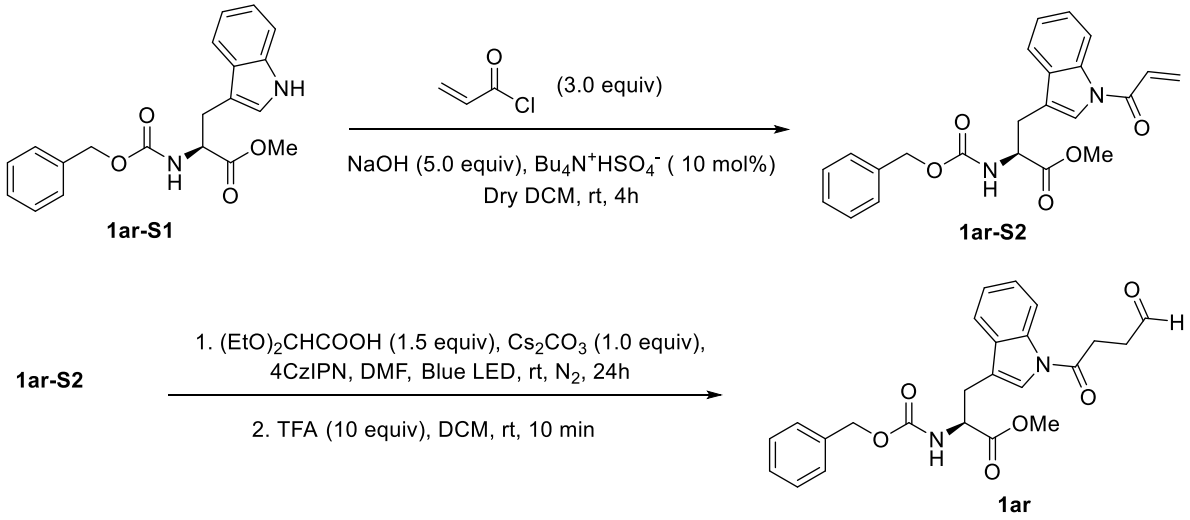
At 0 °C, to the solution of indomethacin (357 mg, 1 mmol, 1.0 equiv) in 5 ml DCM was added 4-hydroxybenzaldehyde (122mg, 1 mmol, 1.0 equiv) and *N*,*N*'-dicyclohexylcarbodiimide (206mg, 1 mmol, 1.0 equiv). The reaction mixture was warmed up to room temperature and stirred for 8 hours. After the completion of the reaction, the mixture was filtrated through a pad of celite, washed with 20 mL DCM. The filtra was concentrated and purified by flash chromatography on silica gel (EtOAc/hexane = 1/3) to afford product (322 mg, 70% yield) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.90 (d, *J* = 8.5 Hz, 2H), 7.68 (d, *J* = 8.3 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.4 Hz, 2H), 7.04 (d, *J* = 2.5 Hz, 1H), 6.88 (d, *J* = 9.0 Hz, 1H), 6.70 (dd, *J* = 9.0, 2.5 Hz, 1H), 3.94 (s, 2H), 3.84 (s, 3H), 2.47 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.65, 168.74, 168.43, 156.30, 155.46, 139.61, 136.56, 134.16, 133.85, 131.36, 131.34, 131.00, 130.50, 129.33, 122.36, 115.22, 111.91, 111.59, 101.35, 55.90, 30.74, 13.56.

3.6 Synthesis of (3aR,4R,6S,6aS)-6-methoxy-2,2-dimethyl-*N*-(3-oxopropyl-3-*d*)tetrahydrofuro[3,4-*d*][1,3]dioxole-4-carboxamide (1aq)



At 0 °C, to a solution of (3*aR*,4*R*,6*S*,6*aS*)-6-methoxy-2,2-dimethyl-*N*-(3-oxopropyl-3-d)tetrahydrofuro[3,4-*d*][1,3]dioxole-4-carboxamide (**1aq-S1**, prepared from reported procedure[8], 218mg, 1mmol, 1.0 equiv) in 10 mL DCM was added EDCI (170 mg, 1.1 mmol, 1.1 equiv), HOBt (149 mg, 1.1 mmol, 1.1 equiv), DIPEA (193 mg, 1.5 mmol, 1.5 equiv) and 3,3-diethoxypropan-1-amine (162 mg, 1.1 mmol). The reaction mixture was warmed up to room temperature and stirred for 8 hours. After the completion of the reaction, the mixture was diluted with 20 mL DCM. The solution was washed by 1M HCl (aq) (10 ml), saturated $NaHCO_3$ (10 ml) and brine (10 ml). The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography (EtOAc/hexane = 1/1) on silica gel to provide the corresponding products 249 mg (91% yield for two steps) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 1H), 6.98 (br, 1H), 5.01 (d, *J* = 10.3 Hz, 2H), 4.52-4.47 (m, 2H), 3.52 (dd, *J* = 11.6, 5.7 Hz, 2H), 3.41 (s, 3H), 2.81 (s, 1H), 2.69 (t, *J* = 5.5 Hz, 2H), 1.43 (s, 3H), 1.27 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.02, 170.35, 112.78, 111.50, 111.46, 86.49, 84.46, 82.61, 82.60, 56.54, 55.52, 43.72, 32.50, 26.50, 24.98.

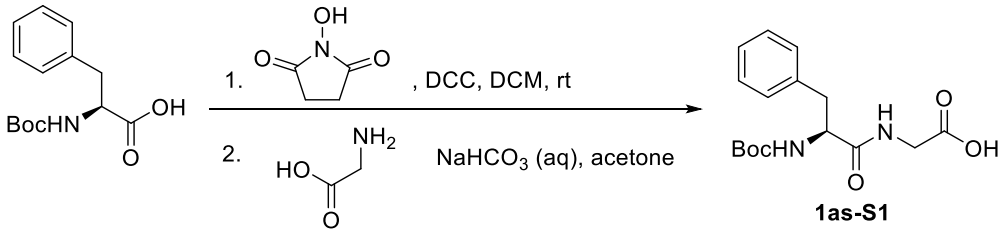
3.7 Synthesis of methyl *N*^{a}-((benzyloxy)carbonyl)-1-(4-oxobutanoyl)-*L*-tryptophanate (1ar)

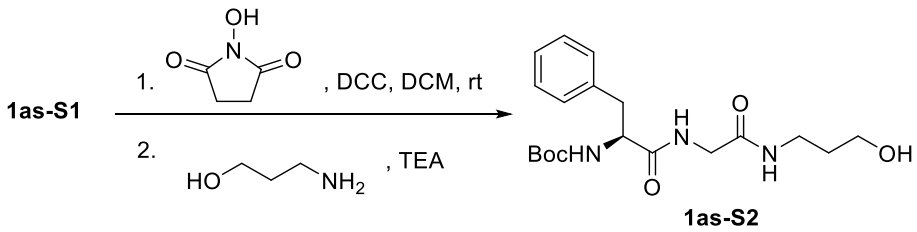


To a solution of Boc-L-Trp-OMe (**1ar-S1**, prepared according to reported procedure[9], 2.4 mmol, 1.0 equiv) in dry 10 mL DCM was added tetrabutylammonium hydrogen sulfate (81mg, 0.24 mmol, 10 mol%), powdered NaOH (480 mg, 12.0 mmol, 5.0 equiv). After stirring of reaction mixture for 15 min at room temperature, acryloyl chloride (648 mg, 7.2 mmol, 3.0 equiv) was added to flask. The reaction mixture was stirred at room temperature for 4h. After the completion of the reaction, the mixture was diluted with 20 mL DCM. The solution was washed by water (10 ml), saturated brine (10 ml). The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by flash chromatography (EtOAc/hexane = 2/3) on silica gel to provide the corresponding products **1ar-S2** (780 mg, 80% yield) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (d, *J* = 8.2 Hz, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.39-7.26 (m, 8H), 6.86 (dd, *J* = 16.7, 10.4 Hz, 1H), 6.64 (d, *J* = 16.7 Hz, 1H), 6.00 (d, *J* = 10.3 Hz, 1H), 5.44 (s, 1H), 5.12 (dd, *J* = 32.0, 12.1 Hz, 2H), 4.77 (d, *J* = 6.3 Hz, 1H), 3.69 (s, 3H), 3.26 (dt, *J* = 14.4, 9.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.05, 163.69, 155.82, 136.27, 136.09, 132.19, 130.69, 128.67, 128.67, 128.39, 128.22, 127.97, 125.62, 124.05, 122.88, 118.84, 117.20, 117.08, 67.18, 53.93, 53.89, 52.93, 52.70, 52.65, 28.14.

To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added **1ar-S2** (406 mg, 1mmol, 1.0 equiv), 2,2-diethoxyacetic acid (222mg, 1.5 mmol, 1.5 equiv), photocatalyst 4CzIPN (40mg, 5 mol%), Cs_2CO_3 (325mg, 1 mmol, 1.0 equiv) and DMF (5 mL). The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 24h. After completion of the reaction, the mixture was diluted by adding 10 ml EA and 30 ml water. The reaction was extracted with EtOAc (3 x 10 mL) and the combined organic extracts were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. 5 ml DCM was added to flask followed by 0.75 mL TFA (10 equiv). The reaction mixture was stirred at room temperature for 10 min. 10 mL saturated $NaHCO_3$ solution was added slowing to quench the reaction. The reaction was extracted with DCM (3 x 10 mL) and the combined organic extracts were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product purified by flash chromatography on silica gel (EtOAc/hexane/DCM = 1/2/2) to afford product 349 mg (80% yield for two steps) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ ^{1}H NMR (400 MHz, CDCl3) δ 9.86 (s, 1H), 8.35 (d, *J* = 8.0 Hz, 1H), 7.45 (d, *J* = 7.5 Hz, 1H), 7.33-7.21 (m, 8H), 5.48 (d, *J* = 7.0 Hz, 1H), 5.09 (dd, *J* = 30.5, 12.2 Hz, 2H), 4.75 (d, *J* = 6.3 Hz, 1H), 3.68 (s, 3H), 3.29-3.15 (m, 2H), 3.10 (t, *J* = 5.6 Hz, 2H), 2.93 (t, *J* = 5.7 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.97, 172.04, 169.42, 155.82, 136.29, 135.87, 130.38, 128.61, 128.30, 128.17, 125.65, 123.82, 122.67, 118.82, 117.36, 116.70, 67.11, 53.87, 52.68, 52.63, 37.85, 28.24, 28.08.

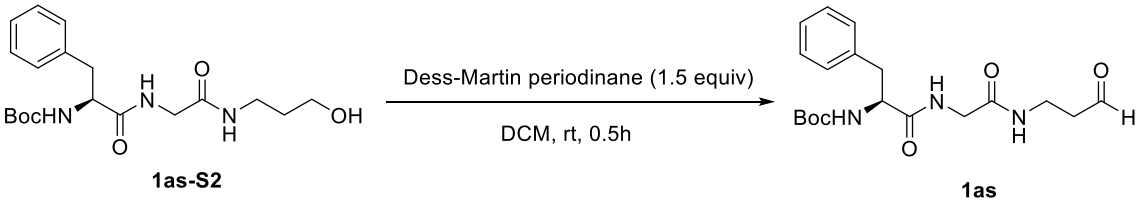
3.8 Synthesis of *tert*-butyl (*S*)-(1-oxo-1-((2-oxo-2-((3-oxopropyl)amino)ethyl)amino)-3-phenylpropan-2-yl)carbamate (1as)





To a solution of Boc-*L*-phenylalanine (796 mg, 3 mmol, 1.0 equiv) in 10 mL DCM was added *N*-hydroxysuccinimide (380 mg, 3.3 mmol, 1.1 equiv) and DCC (680 mg, 3.3 mmol, 1.1 equiv). The reaction mixture was stirred at RT for 8 h, after which the reaction mixture was filtrated through a pad of celite and washed by DCM (2 x 10 mL). The filtra was concentrated under vacuum. To the residue in flask was added acetone (10 mL), glycine (248 mg, 3.3 mmol, 1.1 equiv) and 5 mL saturated $NaHCO_3$. After the reaction mixture was stirred at room temperature for 5h, 20 mL water was added to flask. The reaction mixture was extracted by EtOAc (3 x 10 mL). To the aqueous solution was slowly added 2M HCl to pH = 3-4. The acidified solution was extracted by EtOAc (3 x 10 mL). The combined organic layer was dried over Na2SO4, filtrated and concentrated under vacuum to afford crude **1as-S1** (900mg). To a solution of **1as-S1** in 10 mL DCM was added *N*-hydroxysuccinimide (380 mg, 3.3 mmol) and DCC (680 mg, 3.3 mmol). The reaction mixture was stirred at RT for 8 h, after which the reaction mixture was filtrated through a pad of celite and washed by DCM (2 x 10 mL). To the filtra solution was added 3-aminopropan-1-ol (225 mg, 3 mmol) and TEA (61mg, 0.6 mmol), the reaction mixture was stirred at room temperature for 5h. After the reaction was completed, the reaction is concentrated and by flash chromatography on silica gel (EtOAc/MeOH = 9/1) to afford white foam solid (728 mg, 63% yield for 4 steps). ^{1}H NMR (400 MHz, CD_3OD) δ 7.77 (s, 1H), 7.36–7.21 (m, 5H), 6.82 (d, *J* = 6.3 Hz, 1H), 4.27 (d, *J* = 6.6 Hz, 1H), 3.91 (d, *J* = 16.8 Hz, 1H), 3.70 (d, *J* = 16.8

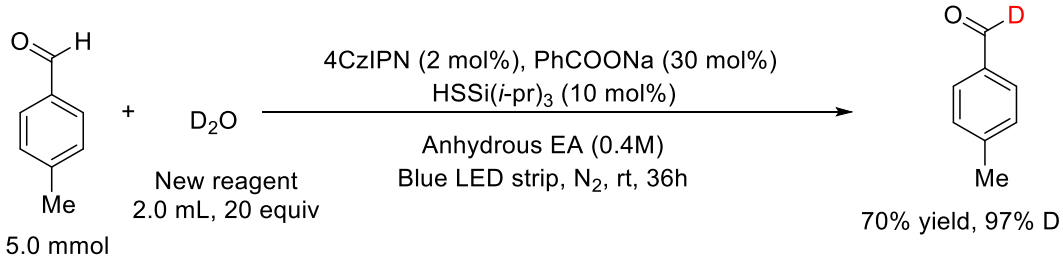
Hz, 1H), 3.61 (t, *J* = 6.1 Hz, 2H), 3.35-3.29 (m, 2H), 3.17-3.12 (m, 1H), 2.92 (dd, *J* = 13.3, 9.0 Hz, 1H), 1.75 (quint, *J* = 6.4 Hz, 2H), 1.41 (s, 9H). ^{13}C NMR (100 MHz, CD_3OD) δ 174.74, 174.69, 171.46, 171.38, 157.79, 138.18, 130.14, 129.32, 127.64, 80.76, 60.19, 57.84, 57.75, 43.46, 43.43, 38.48, 38.45, 37.44, 37.31, 32.92, 28.65.



1as-S2 **1as**

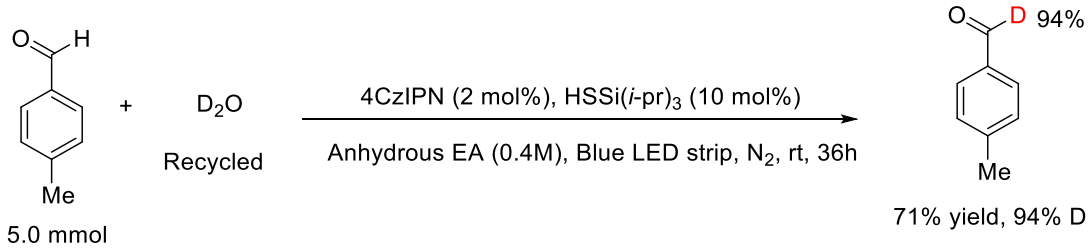
To a solution of **1as-S2** (200mg, 0.52 mmol, 1.0 equiv) in 3 mL DCM was added Dess-Martin periodinane (331mg, 0.78 mmol, 1.5 equiv), the reaction was stirred at room temperature for 0.5 h indicated by TLC. 5 mL Saturated sodium thiosulfate solution was added to flask to quench reaction and followed by 5 mL saturated $NaHCO_3$ solution. The mixture was stirred for 15 min till it turns to be clear. The reaction was extracted with DCM (3 x 10 mL) and the combined organic extracts were washed with brine (1 x 10 mL), dried over Na_2SO_4, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography on silica gel (EtOAc/MeOH = 9/1) to afford product (147mg, 75% yield) as white foam solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.67 (s, 1H), 7.38 (t, *J* = 5.6 Hz), 7.27–7.13 (m, 5H), 5.51 (d, *J* = 6.0 Hz, 1H), 4.34 (d, *J* = 6.7 Hz, 1H), 3.88 (dd, *J* = 16.7, 5.7 Hz, 1H), 3.75 (dd, *J* = 16.7, 5.7 Hz, 1H), 3.50-3.41 (m, 2H), 3.09 (dd, *J* = 13.7, 5.8 Hz, 1H), 2.91 (dd, *J* = 13.1, 8.2 Hz, 1H), 2.62 (t, *J* = 6.1 Hz, 2H), 1.32 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.19, 172.41, 169.28, 155.82, 136.61, 129.21, 128.54, 80.21, 56.09, 43.42, 43.00, 38.13, 33.08, 28.23.

4. Reused D_2O in deuteration reaction of 4-methylbenzaldehyde and gram scale synthesis
Procedure for deuteration in 1st batch



To an oven-dried 25 mL-Schlenk tube equipped with a stir bar, was added 4-methylbenzaldehyde (600 mg, 5.0 mmol, 1.0 equiv), photocatalyst 4CzIPN (80 mg, 2 mol%), PhCOONa (216 mg, 1.5 mmol 30 mol%) and the tube was evacuated and backfilled with N_2 (one time). 12.5 mL anhydrous EtOAc, thiol catalyst triisopropylsilanethiol (**2d**, 0.5 mL 1M, 10 mol%) in EtOAc solution, and D_2O (2.0mL, 20 equiv) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was dumped to 50 mL separatory funnel. Bottom D_2O layer was separated for the usage in next batch. Top organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/hexane = 1/97) on silica gel to provide the corresponding products 423 mg (70% yield, 97% D) as pale yellow liquid.

Procedure for deuteration in 2nd batch using D_2O from 1st batch

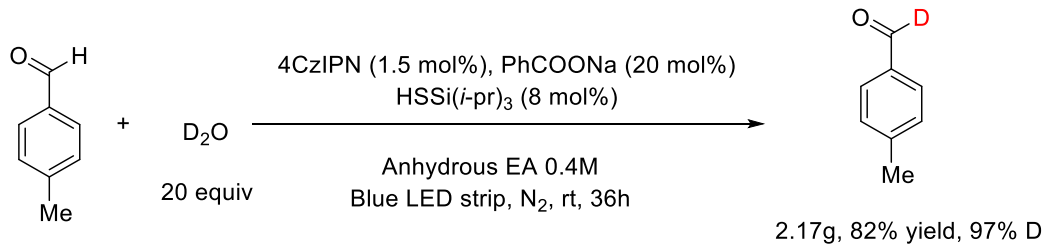


To an oven-dried 25 mL-Schlenk tube equipped with a stir bar, was added 4-methylbenzaldehyde (600 mg, 5.0 mmol, 1.0 equiv) and photocatalyst 4CzIPN (80 mg, 2 mol%), the tube was evacuated and backfilled with N_2 (one time). 12.5 mL anhydrous EtOAc, thiol catalyst triisopropylsilanethiol (**2d**, 0.5 mL 1M, 10 mol%) in EtOAc solution, and D_2O (from 1st batch together with 100 mg, 1.0 equiv to compensate the loss of D_2O) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was dumped to 50 mL separatory funnel. Bottom D_2O layer was separated for the usage in next batch. Top organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/hexane = 1/97) on silica gel to provide the corresponding products430 mg (71% yield, 94% D) as pale yellow liquid.

Procedure for feuteration of 3rd batch using D_2O from 2nd batch

To an oven-dried 25 mL-Schlenk tube equipped with a stir bar, was added 4-methylbenzaldehyde (600 mg, 5.0 mmol, 1.0 equiv) and photocatalyst 4CzIPN (80 mg, 2 mol%), the tube was evacuated and backfilled with N_2 (one time). 12.5 mL anhydrous EtOAc, thiol catalyst triisopropylsilanethiol (**2d**, 0.5 mL 1M, 10 mol%) in EA solution, and D_2O (from 2nd batch together with 100 mg, 1.0 equiv to compensate the loss of D_2O) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was dumped to 50 mL separatory funnel. Bottom D_2O layer was separated for the usage in next batch. Top organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/hexane = 1/97) on silica gel to provide the corresponding products 423 mg (70% yield, 91% D) as pale yellow liquid.

Gram scale synthesis of 4-methylbenzaldehyde-*formyl-d1*



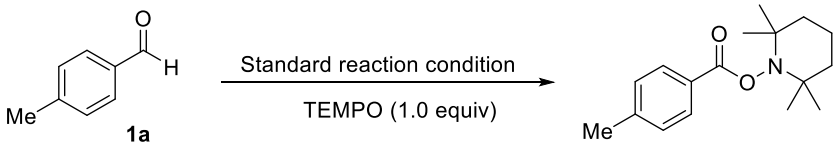
To an oven-dried 100 mL-Schlenk tube equipped with a stir bar, was added 4-methylbenzaldehyde (2.64g, 22.0 mmol, 1.0 equiv), photocatalyst 4CzIPN (264 mg, 1.5 mol%), PhCOONa (216 mg, 1.5 mmol 30 mol%) and the tube was evacuated and backfilled with N_2 (one time). 55 mL anhydrous EA, thiol catalyst triisopropylsilanethiol (**2d**, 1.76 mL 1M, 8 mol%) in EA solution, and D_2O (8.8 mL, 20 equiv) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was dumped to 125 mL separatory funnel. Top organic layer was dried over Na_2SO_4, filtered and concentrated under reduced pressure. The residue was purified by flash chromatography (acetone/hexane = 1/97) on silica gel to provide the corresponding products 2.17 g (82% yield, 97% D) as pale yellow liquid.

5. Table S2. Cost calculation for the synthesis of 1.0 g 4-Methylbenzaldehyde-*formyl-d1*

Item	Vendor	Price	MW	Mmol	Amount	Cost ($)	
Carbazole, 95%	Aksci	$250 for 1kg	167.21	10.00	1.67 g	0.42	
Tetrafluoroisophthalonitrile	Ambeed	$247 for 25g	200.10	2.00	0.4 g	3.95	
Dry THF	Sigma-aldrich	$92.92 for 2L	—	—	40 ml	1.86	
NaH (60%)	Fisher Scientific	$128 for 1 kg	24.00	15.00	0.60g	0.077	
						6.307	for 1.51g 4CzIPN
						4.18	for 1.0g 4CzIPN
Item	Vendor	Price	MW	Mmol	Amount	Cost ($)	
p-Tolualdehyde, 98% (GC)	Aksci	$180 for 1kg	120.15	22.00	2.64g	0.48	
4CzIPN	Self-made	$4.18 for 1g	799.00	0.83	0.66g	2.76	
PhCOONa 99%)	Fisher Scientific	$28.19 for 1kg	144.10	4.4	0.63g	0.18	
Triisopropylsilanethiol (97%)	Sigma-aldrich	$170 for 5g	190.42	1.76	0.335g	11.39	
Deuterium Oxide	Sigma-aldrich	$609 for 1kg	20.03	457.60	9.17g	5.58	
Anhydrous EtOAc	Sigma-aldrich	$62 for 2L	—	—	55 mL	1.7	
						22.09	for 2.17g product
						10.18	**for 1.0g product**

Through calculation, the cost of synthesizing 1.0 g 4-methylbenzaldehyde (97% D ratio) is only about $10.18. By comparison, Suzhou Liaojian Pharmaceutical Technology Co., Ltd. offers 2200 RMB ($314) to prepare 1.0 g same product. This protocol demonstrates its cost-effective advantage in synthesis of deuterated aldehydes.

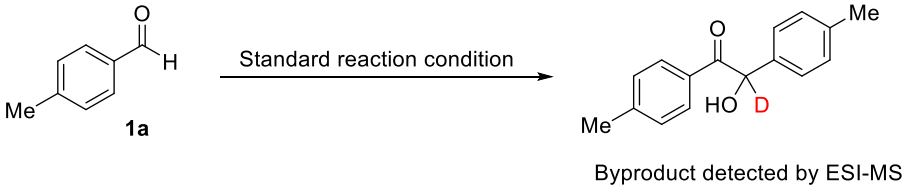
6. Acyl radical intermediate trapping experiment



Detected by ESI-MS

To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added 4-methylbenzaldehyde (0.2 mmol, 1.0 equiv), photocatalyst 4CzIPN (8 mg, 5 mol%), PhCOONa (8.6 mg, 30 mol%), TEMPO (31.2 mg, 0.2 mmol, 1.0 equiv) and the tube was evacuated and backfilled with N_2 (one time). 1 mL anhydrous EA, thiol catalyst triisopropylsilanethiol (**2d**, 60 uL, 30 mol%) in EA solution, and D_2O (160 uL, 40 equiv) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was diluted with Et_2O (20 mL). The solution was washed with brine 10 mL. The organic layer was dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The crude residue was analyzed by ESI-MS and found $[M+H]^+$ 276.1.

7. Observed side reaction



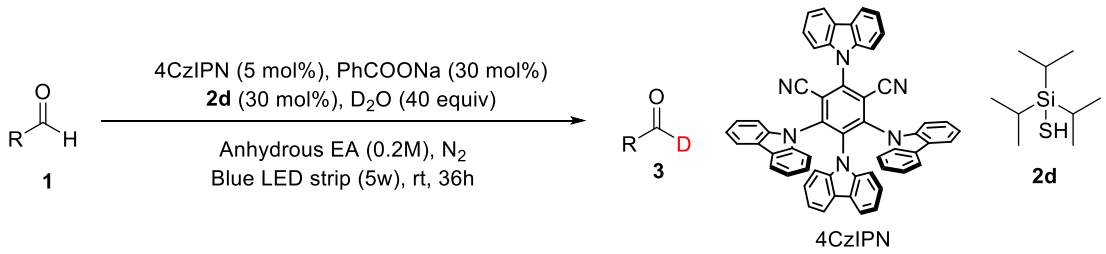
The crude residue was analyzed by ESI-MS and found $[M+Na]^+$ 263.98.

8. General procedure and characterization data for compounds 3

8.1 Preparation of hydrogen isotope exchanged thiol catalyst 2d in EtOAc solution

To an oven-dried 3 mL borosilicate glass vial equipped with a stir bar, was added **2d** in (1.0 mmol, 190 mg, 1.0 equiv), anhydrous EtOAc (1.0 mL) and D_2O (10.0 mmol, 200 μL, 10.0 equiv). The mixture was stirred at room temperature for 1 h. After the stirring was stopped, the top EA layer was used in reaction as the stock solution (1.0 M).

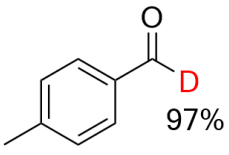
8.2 General procedure for the synthesis of deuterated aldehyde



General Procedure **A**: To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added aldehyde (0.2 mmol, 1.0 equiv), photocatalyst 4CzIPN (8 mg, 5 mol%), PhCOONa (8.6 mg, 30 mol%) and the tube was evacuated and backfilled with N_2 (one time). 1 mL anhydrous EtOAc, thiol catalyst triisopropylsilanethiol (**2d**, 60 uL, 30 mol%) in EtOAc solution, and D_2O (160 uL, 40 equiv) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was concentrated and purified by flash chromatography on silica gel.

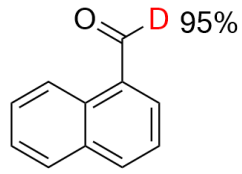
General Procedure **B:** To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added aldehyde (0.2 mmol, 1.0 equiv), photocatalyst 4CzIPN (8.0 mg, 5 mol%), PhCOONa (8.7 mg, 30 mol%), 1 mL anhydrous EtOAc and D_2O (40 ul, 10 equiv). The tube was stirred at room temperature for 10 min to exchange active protium with deuterium. The solvent was removed under reduced pressure and the tube was evacuated and backfilled with N_2 (one time). 1 mL anhydrous EA, thiol catalyst triisopropylsilanethiol (**2d**, 60 uL, 30 mol%) in EtOAc solution, and D_2O (160 uL, 40 equiv) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of a blue LED strip for 36h. After completion of the reaction, the mixture was concentrated and purified by flash chromatography on silica gel.

4-Methylbenzaldehyde-*formyl-d1* (3a)



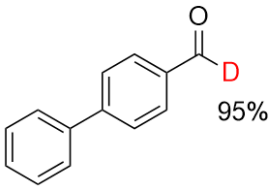
The reaction was carried out according to the general procedure **A**. 4-Methylbenzaldehyde (48.0 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (16.0 mg, 0.02 mmol, 5 mol%), PhCOONa (17.4 mg, 0.06 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (2 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/19) to afford product (44mg, 91% yield, 97% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.93 (s, 0.03H), 7.75 (d, *J* = 8.1 Hz, 2H), 7.30 (d, *J* = 7.8 Hz, 2H), 2.41 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.74 (t, *J* = 26.4 Hz), 145.63, 134.22 (t, *J* = 3.5 Hz), 129.92, 129.79, 21.91. **HRMS** (ESI) calcd for C_8H_8DO $[M + H]^+$ 122.0711, found 122.0704.

1-Naphthaldehyde-*formyl-d1* (3b)



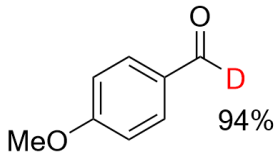
The reaction was carried out according to the general procedure **A**. 1-Naphthaldehyde (62.4 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.3 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (8 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/19) to afford product (32mg, 51% yield, 95% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 10.40 (s, 0.05H), 9.26 (d, *J* = 8.4 Hz, 1H), 8.09 (d, *J* = 8.2 Hz, 1H), 7.98 (d, *J* = 7.0 Hz, 1H), 7.92 (d, *J* = 8.2 Hz, 1H), 7.69 (t, *J* = 7.7 Hz, 1H), 7.63–7.57 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.32 (t, *J* = 26.6 Hz), 136.69, 135.41, 133.85, 131.463 (t, *J* = 3.4 Hz), 130.68, 129.13, 128.60, 127.12, 125.01. **HRMS** (ESI) calcd for $C_{11}H_8DO$ $[M + H]^+$ 158.0711, found 158.0712.

[1,1'-Biphenyl]-4-carbaldehyde-*formyl-d1* (3c)



The reaction was carried out according to the general procedure **A**. [1,1'-Biphenyl]-4-carbaldehyde (36.4 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.6 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc), D_2O (160 uL, 40 equiv) and anhydrous DCE/anhydrous EtOAc (0.5 mL/0.5 mL) were used. The reaction mixture was irritated by 34 W Blue LED strip for 5 h. The residue was purified by flash column chromatography (acetone/hexane = 1/9) to afford product (29mg, 79% yield, 95% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 0.09H), 7.96 (d, *J* = 8.1 Hz, 2H), 7.76 (d, *J* = 8.0 Hz, 2H), 7.66–7.63 (m, 2H), 7.51–7.47 (m, 2H), 7.44-7.40 (m, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.56 (t, *J* = 26.5 Hz), 147.12, 139.66, 135.12 (t, *J* = 3.5 Hz), 130.25, 129.02, 128.49, 127.65, 127.35. **HRMS** (ESI) calcd for $C_{13}H_9DO$ $[M + H]^+$ 184.0867, found 184.0858.

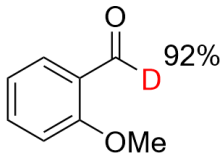
4-Methoxybenzaldehyde-*formyl-d1* (3d)



The reaction was carried out according to the general procedure **A**. 4-Methoxybenzaldehyde (54.8 mg, 0.4 mmol), 4CzIPN (16.0 mg, 0.02 mmol, 5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 20 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction

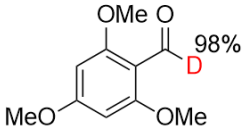
mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/9) to afford product (33mg, 92% yield, 91% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.85 (s, 0.06H), 7.80 (d, *J* = 8.8 Hz, 2H), 6.97 (d, *J* = 8.8 Hz, 2H), 3.85 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.48 (t, *J* = 26.2 Hz), 190.22, 164.65, 131.98, 129.91 (t, *J* = 3.5 Hz), 114.35. **HRMS** (ESI) calcd for C_8H_8DO $[M + H]^+$ 138.0600, found 138.0655.

2-Methoxybenzaldehyde-*formyl-d1* (3e)



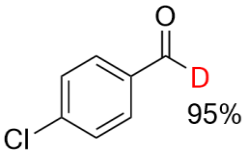
The reaction was carried out according to the general procedure **A**. 2-Methoxybenzaldehyde (54.8 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9) to afford product (54mg, 99% yield, 92% D ratio by ^{1}H NMR) as colorless oil. 1H NMR (400 MHz, $CDCl_3$) δ 10.44 (s, 0.08H), 7.80 (dd, *J* = 7.7, 1.8 Hz, 1H), 7.54–7.50 (m, 1H), 6.98 (dd, *J* = 15.9, 8.1 Hz, 2H), 3.89 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 189.49 (t, *J* = 26.2 Hz), 161.91, 136.02, 128.51, 124.78 (t, *J* = 3.3 Hz), 120.68, 111.69, 55.68. **HRMS** (ESI) calcd for C_8H_8DO $[M + H]^+$ 138.0600, found 138.0653.

2,4,6-Trimethoxybenzaldehyde-*formyl-d1* (3f)



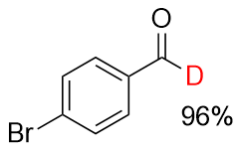
The reaction was carried out according to the general procedure **A**. 2,4,6-Trimethoxybenzaldehyde (39.2 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.6 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane = 2/1-3/1) to afford product (36mg, 99% yield, 98% D ratio by ^{1}H NMR) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.31 (s, 0.02H), 6.04 (s, 2H), 3.84 (s, s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 187.42 (t, *J* = 26.2 Hz), 166.31, 164.18, 108.71 (t, *J* = 3.4Hz). 90.29 (t, *J* = 2.4Hz), 56.03 (q, *J* = 4.8 Hz), 55.56 (q, *J* = 4.8 Hz). **HRMS** (ESI) calcd for $C_{10}H_{12}DO_4$ $[M + H]^+$ 198.0871, found 198.0862.

4-Chlorobenzaldehyde-*formyl-d1* (3g)



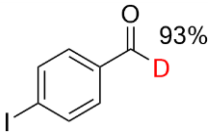
The reaction was carried out according to the general procedure **A**. 4-Chlorobenzaldehyde (56.0 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), NaOAc (9.8 mg, 0.12 mmol, 30 mol%) instead of PhCOONa, **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/19) to afford product (32mg, 57% yield, 95% D ratio by ^{1}H NMR) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 0.05H), 7.81 (d, *J* = 8.0 Hz, 2H), 7.49 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.59 (t, *J* = 26.7 Hz), 141.01, 134.72 (t, *J* = 3.6Hz), 130.96, 129.55. **HRMS** (ESI) calcd for C_7H_5DClO $[M + H]^+$ 142.0164, found 142.0160.

4-Bromobenzaldehyde-*formyl-d1* (3h)



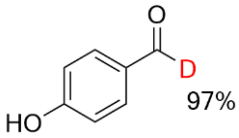
The reaction was carried out according to the general procedure **A**. 4-Bromobenzaldehyde (74.0 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (8.6 mg, 0.06 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc), D_2O (320 uL, 40 equiv) and anhydrous DCE/anhydrous EtOAc (1.0 mL/1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/19) to afford product (61mg, 82% yield, 96% D ratio by ^1H NMR) as white solid. ^1H NMR (400 MHz, $CDCl_3$) δ 9.96 (s, 0.05H), 7.73 (d, *J* = 8.8 Hz, 2H), 7.67 (d, *J* = 8.8 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.82 (t, *J* = 26.7 Hz), 135.09 (t, *J* = 3.6Hz), 132.52, 131.05, 129.86. **HRMS** (ESI) calcd for C_7H_5DBrO $[M + H]^+$ 185.9659 and 187.9639, found 186.9659 and 187.9639.

4-Iodobenzaldehyde-*formyl-d1* (3i)



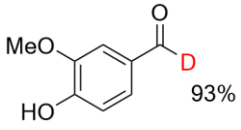
The reaction was carried out according to the general procedure **A**. 4-Iodobenzaldehyde (46.4 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.6 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/19) to afford product (23mg, 50% yield, 93% D ratio by ^1H NMR) as white solid. ^1H NMR (500 MHz, $CDCl_3$) δ 9.95 (s, 0.07H), 7.91 (d, *J* = 8.5 Hz, 2H), 7.59 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 191.42 (t, *J* = 26.3 Hz), 138.66, 135.72 (t, *J* = 3.7Hz), 131.02, 103.01. **HRMS** (ESI) calcd for C_7H_5DIIO $[M + H]^+$ 233.9521, found 233.9521.

4-Hydroxybenzaldehyde-*formyl-d1* (3j)



The reaction was carried out according to the general procedure **A**. 4-Hydroxybenzaldehyde (48.8 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (16.0 mg, 0.02 mmol, 5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (400 uL, 50 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (DCM/ EtOAc/hexane = 1/2/7) to afford product (48mg, 99% yield, 97% D ratio by ^1H NMR) as white solid. ^1H NMR (400 MHz, $CDCl_3$) δ 9.86 (s, 0.03H), 7.82 (d, *J* = 8.0 Hz, 2H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.21 (s, 1H). ^{13}C NMR (125 MHz, DMSO) δ 190.90 (t, *J* = 26.5 Hz), 163.56, 132.27, 128.55 (t, *J* = 3.0 Hz), 116.00. **HRMS** (ESI) calcd for $C_7H_6DO_2$ $[M + H]^+$ 124.0503, found 124.0497.

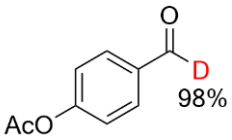
4-Hydroxy-3-methoxybenzaldehyde-*formyl-d1*(3k)



The reaction was carried out according to the general procedure **B**. To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added 4-hydroxy-3-methoxybenzaldehyde (30.4 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), NaOAc (4.9 mg, 0.06 mmol, 30 mol%), anhydrous Na_2CO_3 (21.2 mg, 0.2 mmol, 1.0 equiv), 1.0 mL anhydrous EtOAc and D_2O (40 ul, 10 equiv). The tube was stirred at room temperature for 10 min to exchange active protium with deuterium. The solvent was removed under reduced

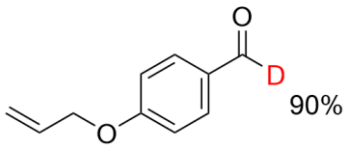
pressure and the tube was evacuated and backfilled with N_2 (one time). **2d** (60 uL, 1M in EA, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were added to tube under N_2 by syringe. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (DCM/ EtOAc /hexane = 1/1/3) to afford product (30mg, 99% yield, 97% D ratio by ^{1}H NMR) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 0.03H), 7.43-7.40 (m, 2H), 7.03 (d, *J* = 8.4 Hz, 1H), 6.43 (s, 1H), 3.94 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.77 (t, *J* = 26.5 Hz), 151.88 (s), 147.31 (s), 129.85 (t, *J* = 3.5 Hz), 127.63, 114.54, 108.94, 56.21. **HRMS** (ESI) calcd for $C_8H_8DO_3$ $[M + H]^+$ 154.0609, found 154.0603.

4-Formylphenyl acetate-*formyl-d1* (3l)



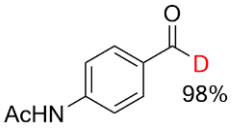
The reaction was carried out according to the general procedure **A**. 4-Formylphenyl acetate (32.8 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.6 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/5) to afford product (16mg, 50% yield, 98% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 0.02H), 7.91 (d, *J* = 8.4 Hz, 2H), 7.27 (d, *J* = 8.2 Hz, 2H), 2.33 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.90 (t, *J* = 26.5 Hz), 168.96, 155.60, 134.14 (t, *J* = 3.5 Hz), 131.40, 122.56, 21.17. **HRMS** (ESI) calcd for $C_9H_8DO_3$ $[M + H]^+$ 166.0609, found 166.0604.

4-(Allyloxy)benzaldehyde-*formyl-d1* (3m)



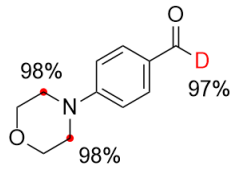
The reaction was carried out according to the general procedure **A**. To an oven-dried 10 mL-Schlenk tube equipped with a stir bar, was added 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), dry CsOAc (11.5 mg, 0.06 mmol, 30 mol%), Cs_2CO_3 (13.0 mg, 0.04 mmol, 20 mol%). The tube was evacuated and backfilled with N_2 (one time). 4-(Allyloxy)benzaldehyde (32.4 mg, 0.2 mmol, 1.0 equiv), 1 mL anhydrous EtOAc, thiol catalyst triisopropylsilanethiol (**2d**, 60 uL, 30 mol%) in EtOAc solution, and D_2O (160 uL, 40 equiv) were added by syringe under N_2. The mixture was degassed by freeze-pump-thaw method, then sealed with parafilm. The solution was then stirred at room temperature under the irradiation of two 34 W Kessil Blue LEDs instead of Blue LED strip. Electronic fan was used to cool the tube. After 1h, another potion of 4CzIPN (8.0 mg, 0.01 mmol 5 mol%) was added to reaction. The solution was then stirred at room temperature under the irradiation of two 34 W Kessil Blue LEDs for another 5h. Electronic fan was used to cool the tub. After completion of reaction, the mixture was concentrated and purified by flash column chromatography (acetone/hexane = 1/5) to afford product (25mg, 74% yield, 90% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.88 (s, 0.10H), 7.83 (d, *J* = 8.8 Hz, 2H), 7.01 (d, *J* = 8.8 Hz, 2H), 6.10-6.00 (m, 1H), 5.38 (ddd, *J* = 13.9, 11.8, 1.4 Hz, 2H), 4.62 (dt, *J* = 5.3, 1.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.61 (t, *J* = 26.2 Hz), 163.74, 132.41, 132.09, 130.10 (t, *J* = 3.5 Hz), 118.49, 115.14, 69.15. **HRMS** (ESI) calcd for $C_{10}H_{10}DO_3$ $[M + H]^+$ 164.0816, found 164.0809.

***N*-(4-Formylphenyl)acetamide-*formyl-d1* (3n)**



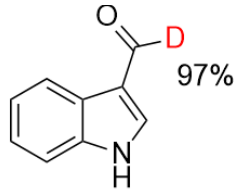
The reaction was carried out according to the general procedure **A**. *N*-(4-Formylphenyl)acetamide (65.2 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (400 uL, 50 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane = 2/1-3/1) to afford product (48mg, 85% yield, 97% D ratio by ^{1}H NMR) as light yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 0.02H), 7.85 (d, J = 8.4 Hz, 2H), 7.74 (br, 1H), 7.70 (d, J = 8.4 Hz, 2H), 2.23 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.91 (t, *J* = 26.4 Hz), 168.87, 143.69, 132.30 (t, *J* = 3.2 Hz), 131.30, 119.36, 24.96. **HRMS** (ESI) calcd for $C_9H_9DNO_2$ $[M + H]^+$ 165.0769, found 165.0763.

4-Morpholinobenzaldehyde-*formyl-d1* (3o)



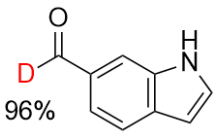
The reaction was carried out according to the general procedure **A**. 4-Morpholinobenzaldehyde (38.2 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), Na_2CO_3 (6.4 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (320 uL, 80 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane = 1/1) to afford product (32mg, 83% yield, 97% D ratio by ^{1}H NMR) as brown solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.79 (s, 0.03H), 7.76 (d, *J* = 9.0 Hz, 2H), 6.90 (d, *J* = 9.0 Hz, 2H), 3.84 (s, 4H), 3.32-3.30 (m, 0.1H, 98% D ratio). ^{13}C NMR (125 MHz, $CDCl_3$) δ 190.46 (t, *J* = 26.0 Hz), 155.38, 132.00, 127.71 (t, *J* = 3.3 Hz), 113.53, 66.44, 46.90, 46.74, 46.57, 46.41, 46.23 (quint, *J* = 20.9 Hz). **HRMS** (ESI) calcd for $C_{11}H_9D_5NO_2$ $[M + H]^+$ 197.1333, found 197.1324.

1*H*-indole-3-carbaldehyde-*formyl-d1* (3p)



The reaction was carried out according to the general procedure **A**. 1*H*-indole-3-carbaldehyde (58.4 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (400 uL, 50 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane/DCM = 1/1/1) to afford product (50mg, 87% yield, 97% D ratio by ^{1}H NMR) as light brown solid. ^{1}H NMR (400 MHz, DMSO) δ 12.13 (s, 1H), 9.93 (s, 0.03H), 8.28 (d, *J* = 2.0 Hz, 1H), 8.10 (s, 1H), 7.52-7.50 (m, 1H), 7.27-7.20 (m, 2H). ^{13}C NMR (100 MHz, DMSO) δ 184.70 (t, *J* = 26.0 Hz), 138.39, 137.03, 124.12, 123.43, 122.09, 120.82, 118.06 (t, *J* = 3.3 Hz), 112.39. **HRMS** (ESI) calcd for C_9H_7DNO $[M + H]^+$ 147.0663, found 147.0657.

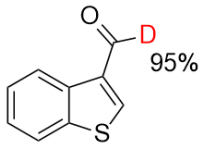
1*H*-indole-6-carbaldehyde-*formyl-d1* (3q)



The reaction was carried out according to the general procedure **A**. 1*H*-indole-3-carbaldehyde (58.4 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (400 uL, 50 equiv) and anhydrous EtOAc (2.0 mL) were used.

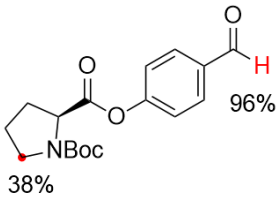
The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane/DCM = 1/1/1) to afford product (54mg, 93% yield, 96% D ratio by ^{1}H NMR) as light brown solid. ^{1}H NMR (400 MHz, DMSO) δ 11.69 (s, 1H), 10.01 (s, 0.04H), 8.01 (s, 1H), 7.71–7.67 (m, 2H), 7.55 (d, *J* = 8.2 Hz, 1H), 6.58 (s, 1H).^{13}C NMR (100 MHz, DMSO) δ 192.44 (t, *J* = 27.2 Hz), 135.27, 132.56, 130.37, 130.11 (t, *J* = 2.4 Hz), 120.43, 118.82, 115.51, 102.04. **HRMS** (ESI) calcd for C_9H_7DNO [M + H]$^+$ 147.0663, found 147.0657.

Benzo[*b*]thiophene-3-carbaldehyde-*formyl-d1* (2r)



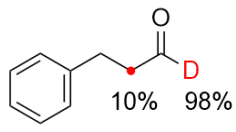
The reaction was carried out according to the general procedure **A**. Benzo[*b*]thiophene-3-carbaldehyde (64.8 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9) to afford product (41mg, 63% yield, 95% D ratio by ^{1}H NMR) as light brown solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.13 (s, 0.05H), 8.68 (d, *J* = 8.1 Hz, 1H), 8.29 (s, 1H), 7.87 (d, *J* = 7.9 Hz, 1H), 7.48 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 185.18 (t, *J* = 26.7 Hz), 143.26, 140.53, 136.47 (t, *J* = 3.7 Hz), 135.24, 126.24, 126.18, 124.90, 122.51, 77.48, 76.84. **HRMS** (ESI) calcd for C_9H_6DOS [M + H]$^+$ 164.0275, found 164.0271.

1-(*tert*-Butyl) 2-(4-formylphenyl) (*S*)-pyrrolidine-1,2-dicarboxylate-*formyl-d1* (3s)



The reaction was carried out according to the general procedure **A**. 1-(*tert*-Butyl) 2-(4-formylphenyl) (*S*)-pyrrolidine-1,2-dicarboxylate (prepared according to reported procedure [10],64.0 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc/anhydrous DCE (0.4mL/0.6mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane/DCM = 1/5/1) to afford product (42mg, 66% yield, 96% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, s, rotamer, 0.04H), 7.9-7.89 (d, d, *J* = 8.4 Hz, rotamer, 2H), 7.29 (t, *J* = 8.5 Hz, 2H), 4.50 (dd, dd, *J* =8.4, 4.0 Hz, 1H), 3.66-3.43 (m, 1.25H), 2.45-2.31 (m, 1H), 2.28-2.13 (m, 1H), 2.08-1.91(m, 2H), 1.47 and 1.46 (s, s, rotamer, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 191.07-190.32 (m, split by D, rotamer), 171.20, 171.12, 155.67, 155.37, 154.63, 153.75, 134.17-134.03 (m, rotamer), 131.40, 131.29, 122.41, 122.30, 80.53, 80.34, 59.34, 59.24, 46.78-46.10 (m, split by D), 31.15, 30.10, 24.61, 23.77. **HRMS** (ESI) calcd for $C_{17}H_{20}DNO_5Na$ [M + Na]$^+$ 343.1375, found 343.1365; calcd for $C_{17}H_{20}D_2NO_5$ [M + H]$^+$ 322.1618, found 322.1616.

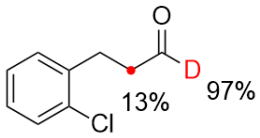
3-Phenylpropanal-*formyl-d1* (3t)



The reaction was carried out according to the general procedure **A**. 3-Phenylpropanal (53.6 mg, 0.4 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction

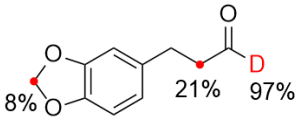
mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/9) to afford product (53mg, 99% yield, 98% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 (s, 0.02H), 7.31 (t, *J* = 7.0 Hz, 2H), 7.22 (t, *J* = 7.4 Hz, 3H), 2.97 (t, *J* = 7.3 Hz, 2H), 2.78 (t, *J* = 7.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.30 (t, *J* = 26.0 Hz), 140.43, 128.67, 128.36, 126.36, 45.15 (t, *J* = 3.7 Hz), 28.16. **HRMS** (ESI) calcd for C_9H_6DOS $[M + H]^+$ 164.0275, found 164.0271. **HRMS** (ESI) calcd for C_9H_6DONa $[M + Na]^+$ 158.0687, found 158.0682.

3-(2-Chlorophenyl)propanal-*formyl-d1* (3u)



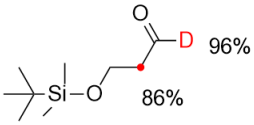
The reaction was carried out according to the general procedure **A**. 3-(2-Chlorophenyl)propanal (prepared according to reported procedure[1], 67.2 mg, 0.4 mmol), 4CzIPN (8.0 mg, 0.01 mmol, 2.5 mol%), PhCOONa (17.4 mg, 0.12 mmol, 30 mol%), **2d** (120 uL, 1M in EtOAc, 30 mol%), D_2O (320 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/9) to afford product (55mg, 82% yield, 98% D ratio by ^{1}H NMR) as pale-yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 9.83 (t, *J* = 1.3 Hz, 0.03H), 7.35 (dd, *J* = 7.6, 1.6 Hz, 1H), 7.25–7.23 (m, 1H), 7.21–7.14 (m, 2H), 3.06 (t, *J* = 7.6 Hz, 2H), 2.79 (t, *J* = 7.6 Hz, 1.74H). ^{13}C NMR (120 MHz, $CDCl_3$) δ 201.16 (t, *J* = 26.2 Hz), 138.21, 134.07, 130.73, 129.84, 128.10, 127.21, 43.40 (t, *J* = 3.9 Hz), 26.20. **HRMS** (ESI) calcd for $C_9H_8DClONa$ $[M + Na]^+$ 192.0297, found 192.0293.

3-(Benzo[*d*][1,3]dioxol-5-yl)propanal-*formyl-d1* (3v)



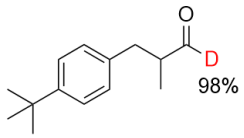
The reaction was carried out according to the general procedure **A**. 3-(Benzo[d][1,3]dioxol-5-yl)propanal (35.6 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/9) to afford product (30mg, 85% yield, 97% D ratio by ^{1}H NMR) as pale-yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (t, *J* = 1.2 Hz, 0.03H), 6.72 (d, *J* = 7.9 Hz, 1H), 6.67 (d, *J* = 1.5 Hz, 1H), 6.63 (dd, *J* = 7.9, 1.4 Hz, 1H), 5.91 and 5.89 (s, s, 1.84H, 21% D ratio), 2.87 (t, *J* = 7.4 Hz, 2H), 2.72 (t, *J* = 7.3 Hz, 1.58H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.35 (t, *J* = 26.3 Hz), 147.84, 146.08, 134.21, 121.16, 108.86, 108.41, 100.99, 45.46 (t, *J* = 3.7 Hz), 27.96. **HRMS** (ESI) calcd for $C_9H_9DO_3$ $[M]^+$ 180.0765, found 180.0759.

3-((*tert*-Butyldimethylsilyl)oxy)propanal-*formyl-d1* (3w)



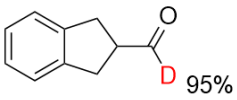
The reaction was carried out according to the general procedure **A**. 3-((*tert*-Butyldimethylsilyl)oxy)propanal (prepared according to reported procedure[11], 37.6 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/9) to afford product (33mg, 87% yield, 96% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 0.04H), 3.97 (s, 2H), 2.61–2.53 (m, 0.28H, 86% D ratio), 0.87 (s, 9H), 0.06 (s, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.00 (t, *J* = 26.9 Hz), 57.48, 46.68–45.63 (m), 25.95, 18.36, -5.31. **HRMS** (ESI) calcd for $C_9H_{19}D_2O_2Si$ $[M+H]^+$ 191.1431, found 191.1425; calcd for $C_9H_{18}D_3O_2Si$ $[M+H]^+$ 192.1494, found 192.1485.

3-(4-(*tert*-Butyl)phenyl)-2-methylpropanal-*formyl-d1* (3x)



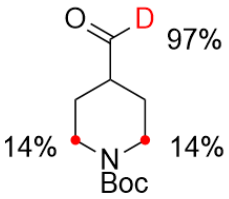
The reaction was carried out according to the general procedure **A**. 3-(4-(*tert*-Butyl)phenyl)-2-methylpropanal (40.8 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9) to afford product (25mg, 60% yield, 98% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.73 (d, *J* = 1.5 Hz, 0.02H), 7.32 (d, *J* = 8.3 Hz, 2H), 7.11 (d, *J* = 8.2 Hz, 2H), 3.06 (dd, *J* = 13.5, 5.8 Hz, 1H), 2.71–2.55 (m, 2H), 1.31 (s, 9H), 1.10 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.42 (t, *J* = 26.9 Hz), 149.39, 135.83, 128.80, 125.54, 48.00 (t, *J* = 3.5 Hz), 36.30, 34.54, 31.51, 13.43. **HRMS** (ESI) calcd for $C_{14}H_{19}DONa$ $[M+Na]^+$ 228.1462, found 228.1469.

2,3-Dihydro-1*H*-indene-2-carbaldehyde-*formyl-d1* (3y)



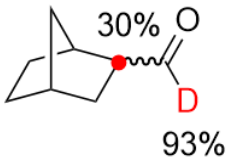
The reaction was carried out according to the general procedure **A**. 2,3-Dihydro-1H-indene-2-carbaldehyde (29.2 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9) to afford product (27mg, 91% yield, 95% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.78 (s, 0.05H), 7.26-7.23 (m, 2H), 7.18-7.16 (m, 2H), 3.32-3.25 (m, 3H), 3.32-3.16 (m, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.66 (t, *J* = 26.2 Hz), 141.22, 126.93, 124.75, 50.62–50.48 (t, *J* = 3.5 Hz), 33.04. **HRMS** (ESI) calcd for $C_{10}H_9DONa$ $[M+Na]^+$ 170.0687, found 170.0684.

***tert*-Butyl 4-formylpiperidine-1-carboxylate-*formyl-d1* (3z)**



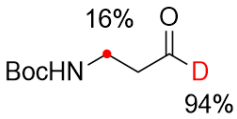
The reaction was carried out according to the general procedure **A**. *tert*-Butyl 4-formylpiperidine-1-carboxylate (29.2 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous DCE (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9) to afford product (27mg, 91% yield, 95% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.62 (s, 0.03H), 3.93 (br, 2H), 2.89 (t, *J* = 10.9 Hz, 1.73H), 2.40-2.35(m, 1H), 1.89-1.84 (m, 2H), 1.55-1.48 (m, 2H), 1.41 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.94 (t, *J* = 20.9 Hz), 154.85, 79.75, 47.87–47.75 (m), 42.80 (br), 28.39, 25.11. **HRMS** (ESI) calcd for $C_{11}H_{18}DNO3$ $[M+H]^+$ 215.1500, found 215.1500.

Bicyclo[2.2.1]hept-5-ene-2-carboxaldehyde-*formyl-d1* (3aa)



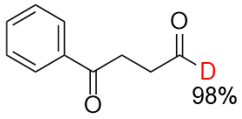
The reaction was carried out according to the general procedure **A**. Bicyclo[2.2.1]hept-5-ene-2-carboxaldehyde (36.6 mg, 0.3 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 3.7 mol%), PhCOONa (13.0 mg, 0.09 mmol, 30 mol%), Cs_2CO_3 (19.0 mg, 0.06 mmol, 20 mol%), **2d** (60 uL, 1M in EtOAc, 20 mol%), D_2O (240 uL, 40 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9) to afford product (19mg, 52% yield, 93% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.42 (d, *J* = 2.8 Hz, 1H), 6.21 (dd, *J* = 5.5, 3.0 Hz, 1H), 5.99 (dd, *J* = 5.7, 2.8 Hz, 1H), 3.24 (s, 1H), 2.98 (s, 1H), 2.90 (dt, *J* = 9.0, 3.8 Hz, 0.69H), 1.92–1.87 (m, 1H), 1.50–1.40 (m, 2H), 1.31 (dd, *J* = 8.3, 1.4 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 204.95 (t, *J* = 25.9 Hz), 138.28, 131.94, 52.22 (t, *J* = 3.5 Hz), 49.80, 45.18, 42.90, 27.73. **HRMS** (ESI) calcd for $C_8H_{10}DO$ $[M+H]^+$ 124.0867, found 124.0867.

***tert*-Butyl (3-oxopropyl)carbamate-*formyl-d1* (3ab)**



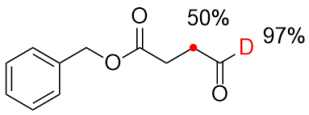
The reaction was carried out according to the general procedure A. *tert*-Butyl (3-oxopropyl)carbamate (34.6 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (40 uL, 1M in EtOAc, 20 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (2.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/3) to afford product (28mg, 81% yield, 94% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 0.06H), 4.89 (s, 1H), 3.46–3.36 (t, *J* = 5.6 Hz, 1.67H), 2.69 (t, *J* = 5.3 Hz, 2H), 1.42 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.24 (t, *J* = 26.0 Hz), 155.94, 79.61, 44.25 (t, *J* = 3.6 Hz), 34.13, 28.51. **HRMS** (ESI) calcd for $C_8H_{14}DO_3$ $[M]^+$ 197.1007, found 197.1000.

4-Oxo-4-phenylbutanal-*formyl-d1* (3ac)



The reaction was carried out according to the general procedure **A**. 4-Oxo-4-phenylbutanal (32.4 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/3) to afford product (27mg, 84% yield, 98% D ratio by ^1H NMR) as colorless oil. ^1H NMR (400 MHz, $CDCl_3$) δ 9.89 (s, 0.02H), 7.98 (d, *J* = 8.1 Hz, 2H), 7.57 (t, *J* = 6.8 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 2H), 3.32 (t, *J* = 6.2 Hz, 2H), 2.92 (t, *J* = 6.2 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.44 (t, *J* = 26.6 Hz), 197.91, 136.51, 133.41, 128.74, 128.15, 37.50 (t, *J* = 3.8 Hz), 31.08. **HRMS** (ESI) calcd for $C_8H_{10}DO_2$ $[M+H]^+$ 164.0816, found 164.0811.

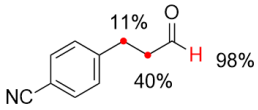
Benzyl 4-oxobutanoate-*formyl-d1* (3ad)



The reaction was carried out according to the general procedure **A**. Benzyl 4-oxobutanoate (38.4 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60

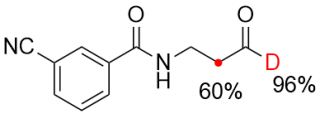
uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/3) to afford product (35mg, 84% yield, 97% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 0.03 H), 7.40-7.26 (m, 5H), 5.13 (s, 2H), 2.81 (t, *J* = 6.4 Hz, 0.95 H), 2.68 (t, *J* = 6.4 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.71 (t, *J* = 26.7 Hz), 172.21, 135.82, 128.69, 128.79, 128.42, 128.33 66.76, 38.43 (t, *J* = 3.8 Hz), 26.67. **HRMS** (ESI) calcd for $C_{11}H_{12}DO_3$ $[M+H]^+$ 194.0922, found 194.0921; calcd for $C_{11}H_{10}D_3O_3$ $[M+H]^+$ 196.1048, found 196.1047.

4-(3-Oxopropyl)benzonitrile-*formyl-d1* (3ae)



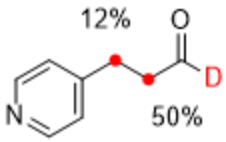
The reaction was carried out according to the general procedure **A**. 4-(3-Oxopropyl)benzonitrile (38.4 mg, 0.2 mmol), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (acetone/hexane = 1/3) to afford product (35mg, 84% yield, 97% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.81 (s, 0.02 H), 7.58 (d, *J* = 8.0 Hz, 2H), 7.30 (d, *J* = 7.9 Hz, 2H), 3.01 (t, *J* = 6.8 Hz, 1.77H), 2.82 (t, *J* = 7.1 Hz, 1.19H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.06 (t, *J* = 26.7 Hz), 146.20, 132.51, 129.32, 118.97, 110.41, 44.47 (t, *J* = 3.7 Hz), 28.06 (t, *J* = 6.1 Hz). **HRMS** (ESI) calcd for $C_{10}H_9DNO$ $[M+H]^+$ 161.0820, found 161.0813; calcd for $C_{10}H_8D_2NO$ $[M+H]^+$ 162.0882, found 162.0876.

3-Cyano-*N*-(3-oxopropyl)benzamide-*formyl-d1* (3af)



The reaction was carried out according to the general procedure **B**. 3-Cyano-*N*-(3-oxopropyl)benzamide (40.4 mg, 0.2 mmol), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by preparative TLC plate (EtOAc/hexane = 3/1) to afford product (32mg, 80% yield, 96% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.86 (s, 0.04 H), 8.05 (s, 1H), 7.96 (d, *J* = 7.7 Hz, 1H), 7.77 (d, *J* = 7.7 Hz, 1H), 7.56 (t, *J* = 7.8 Hz, 1H), 6.85 (br, 1H), 3.74 (d, *J* = 5.4 Hz, 2H), 2.90-2.86 (m, 0.79H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.90-201.24 (m), 165.39, 135.56, 134.88, 131.26, 130.96, 129.70, 118.09, 113.11, 42.39-42.86 (m), 33.74 (t, *J* = 5.0 Hz). **HRMS** (ESI) calcd for $C_{11}H_{10}DN_2O_2$ $[M+H]^+$ 204.0878, found 204.073; calcd for $C_{10}H_8D_2NO$ $[M+H]^+$ 205.0941, found 205.0931.

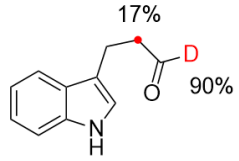
3-(Pyridin-4-yl)propanal-*formyl-d1* (3ag)



The reaction was carried out according to the general procedure **A**. 3-(Pyridin-4-yl)propanal (27.0 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous DCE (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by preparative TLC plate (EtOAc/hexane = 3/1) to afford product (21mg, 78% yield, 97% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.82 (t, *J* = 1.2 Hz, 1H), 8.52 (d, *J* = 5.7 Hz, 2H), 7.14 (d, *J* = 5.8 Hz, 2H), 2.95 (t, *J* = 7.2 Hz, 1.76H), 2.82 (t, *J* = 7.2 Hz, 1.05H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.35-199.73 (m), 149.77,

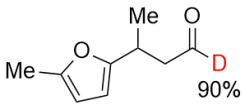
123.94, 43.84 (t, *J* = 3.7 Hz), 27.26 (t, *J* = 6.1 Hz). **HRMS** (ESI) calcd for C_8H_9DNO $[M+H]^+$ 137.0820, found 137.0814.

3-(1*H*-indol-3-yl)propanal-*formyl-d1* (3ah)



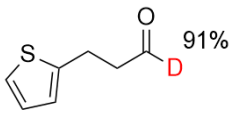
The reaction was carried out according to the general procedure **B**. 3-(1*H*-indol-3-yl)propanal-*formyl* (prepared according to reported procedure[12], 34.6 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), PhCOONa (8.7 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by Blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane/DCM = 3/1/1) to afford product (21mg, 60% yield, 90% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.85 (s, 0.1 H), 8.00 (br, 1H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 8.1 Hz, 1H), 7.22 (t, *J* = 7.1 Hz, 1H), 7.17–7.12 (m, 1H), 6.98 (s, 1H), 3.13 (t, *J* = 7.2 Hz, 2H), 2.85 (t, *J* = 7.3 Hz, 1.67H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.45 (t, *J* = 26.4 Hz), 136.45, 127.19, 122.31, 121.62, 119.54, 118.71, 111.34, 43.94 (t, *J* = 3.7 Hz), 17.94. **HRMS** (ESI) calcd for $C_{11}H_{11}DNO$ $[M+H]^+$ 175.0976, found 175.0974.

3-(5-Methylfuran-2-yl)butanal-*formyl-d1* (3ai)



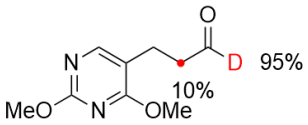
The reaction was carried out according to the general procedure **A**. 3-(5-Methylfuran-2-yl)butanal (30.4 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (16.0 mg, 0.02 mmol, 10 mol%), NaOAc (4.9 mg, 0.06 mmol, 30 mol%) instead of PhCOONa, Na_2CO_3 (6.4 mg, 0.06 mmol, 30 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by two 34 W Kessil Blue LEDs instead of LED strips for 6 h. The residue was purified by flash column chromatography (acetone/hexane = 1/19) to afford product (15mg, 50% yield, 90% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (s, 0.1H), 5.86 (d, *J* = 10.6 Hz, 2H), 3.37 (dd, *J* = 13.7, 6.9 Hz, 1H), 2.77 (dd, *J* = 16.7, 6.6 Hz, 1H), 2.55 (dd, *J* = 16.7, 7.2 Hz, 1H), 2.24 (s, 3H), 1.29 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.55 (t, *J* = 26.3 Hz), 156.56, 150.94, 105.95, 104.95, 49.25 (t, *J* = 3.6 Hz), 28.08, 19.23, 13.63. **HRMS** (ESI) calcd for $C_9H_{12}DNO_2$ $[M+H]^+$ 154.0973, found 154.0978.

3-(Thiophen-2-yl)propanal-*formyl-d1* (3aj)



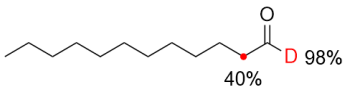
The reaction was carried out according to the general procedure **A**. 3-(Thiophen-2-yl)propanal (28.0 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane = 1/9-1/5) to afford product (27mg, 95% yield, 91% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.83 (s, 0.09H), 7.13 (d, *J* = 4.5 Hz, 1H), 6.91-6.91 (m, 1H), 6.82 (s, 1H), 3.18 (t, *J* = 7.2 Hz, 2H), 2.84 (t, *J* = 7.3 Hz, 2H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.73 (t, *J* = 26.3 Hz), 143.05, 127.06, 124.88, 123.74, 45.31 (t, *J* = 3.7 Hz), 22.50. **HRMS** (ESI) calcd for C_7H_7DOSNa $[M+Na]^+$ 164.0251, found 164.0250.

3-(2,4-Dimethoxypyrimidin-5-yl)propanal-*formyl-d1* (3ak)



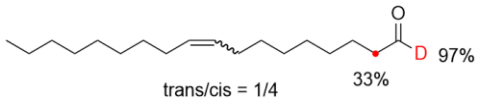
The reaction was carried out according to the general procedure **A**. 3-(2,4-Dimethoxypyrimidin-5-yl)propanal (39.2 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane/DCM = 5/3/2) to afford product (24mg, 62% yield, 95% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (t, *J* = 1.2 Hz, 0.05H), 8.03 (s, 1H), 3.97 (d, *J* = 1.2 Hz, 3H), 3.95 (d, *J* = 1.3 Hz, 3H), 2.81–2.75 (m, 2H), 2.73–2.64 (m, 1.8H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.95 (t, *J* = 26.4 Hz), 169.39, 164.51, 157.36, 113.56, 54.81 (q, *J* = 3.6 Hz), 54.02 (q, *J* = 3.5 Hz), 42.85 (t, *J* = 3.7 Hz), 19.60. **HRMS** (ESI) calcd for $C_9H_{11}DN_2O_3$ $[M+H]^+$ 198.0983, found 198.0978.

Dodecanal-*formyl-d1* (3al)



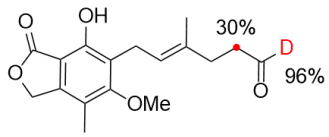
The reaction was carried out according to the general procedure **A**. Dodecanal (36.8 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane/ = 1/19) to afford product (24mg, 66% yield, 98% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (t, *J* = 1.2 Hz, 0.05H), 2.30 (t, *J* = 7.4 Hz, 1.29H), 1.57–1.47 (m, 2H), 1.15 (s, 16H), 0.77 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.78 (t, *J* = 26.4 Hz), 43.90 (t, *J* = 3.5 Hz), 32.05, 29.74, 29.73, 29.58, 29.51, 29.47, 29.32, 22.83, 22.21, 14.26. **HRMS** (ESI) calcd for $C_{22}H_{23}DONa$ $[M+Na]^+$ 208.1782, found 207.1785.

Olealdehyde-*formyl-d1* (3am)



The reaction was carried out according to the general procedure **A**. Olealdehyde (53.2 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (Et_2O/hexane/ = 1/19) to afford product (35mg, 65% yield, 97% D ratio by ^{1}H NMR, trans/cis = 1/4 by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.76 (t, *J* = 1.7 Hz, 0.03H), 5.43–5.35 (m, 1.37H, *cis*), 5.35–5.29 (m, 0.33H, *trans*), 2.41 (t, *J* = 7.4 Hz, 1.34H), 2.01-1.93 (m, 4H), 1.67–1.57 (m, 2H), 1.28 (dd, J = 17.4, 2.6 Hz, 20H), 0.88 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.75 (t, *J* = 25.9 Hz), 130.66 (*cis*), 130.30 (*cis*), 130.18 (*trans*), 129.83 (*trans*), 43.88 (t, *J* = 3.5 Hz), 32.75, 32.68, 32.05, 29.80, 29.68, 29.64, 29.47, 29.36, 29.33, 29.29, 29.05, 22.83, 22.20, 14.26. **HRMS** (ESI) calcd for $C_{18}H_{34}DO$ $[M+H]^+$ 268.2745, found 268.2746.

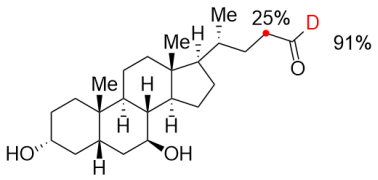
(*E*)-6-(4-hydroxy-6-methoxy-7-methyl-3-oxo-1,3-dihydroisobenzofuran-5-yl)-4-methylhex-4-enal-*formyl-d1* (3an)



The reaction was carried out according to the general procedure **B**. (*E*)-6-(4-Hydroxy-6-methoxy-7-methyl-3-oxo-1,3-dihydroisobenzofuran-5-yl)-4-methylhex-4-enal (prepared according to reported procedure[10], 30.4 mg, 0.1 mmol, 1.0 equiv), 4CzIPN (4.0 mg, 0.005 mmol, 5 mol%), **2d** (30 uL, 1M in EtOAc, 30 mol%),

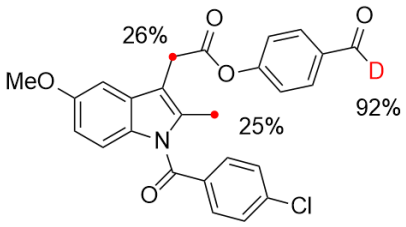
D_2O (80 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane = 3/7) to afford product (26mg, 85% yield, 96% D ratio by ^{1}H NMR) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.72 (s, 0.04H), 5.24 (t, *J* = 7.0 Hz, 1H), 5.19 (s, 2H), 3.75 (s, 3H), 3.38 (d, *J* = 6.9 Hz, 2H), 2.59–2.44 (m, 1.4H), 2.35–2.30 (m, 2H), 2.14 (s, 3H), 1.80 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 202.57-202.02 (m), 173.03, 163.78, 153.74, 144.19, 133.97, 123.05, 122.10, 116.86, 106.52, 70.18, 61.13, 42.21–41.43 (m), 31.83 (t, *J* = 5.6 Hz), 22.72, 16.41, 11.70. **HRMS** (ESI) calcd for $C_{17}H_{20}DO_5$ $[M+H]^+$ 306.1446, found 306.1439.

(*R*)-4-((3*R*,5*S*,7*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phenanthren-17-yl)pentanal-*formyl-d1* (3ao)



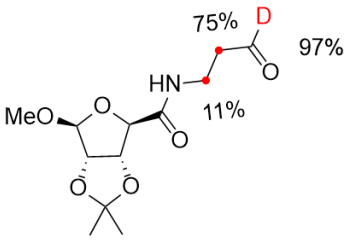
The reaction was carried out according to the general procedure **B**. (*R*)-4-((3*R*,5*S*,7*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phena -nthren-17-yl)pentanal (prepared according to reported procedure[10], 37.7 mg, 0.1 mmol, 1.0 equiv), 4CzIPN (4.0 mg, 0.005 mmol, 5 mol%), **2d** (30 uL, 1M in EtOAc, 30 mol%), D_2O (80 uL, 40 equiv) and anhydrous DCE (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane = 1/1-3/1) to afford product (23mg, 60% yield, 91% D ratio by ^{1}H NMR) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.79 (s, 0.09H), 3.66–3.52 (m, 2H), 2.49-2.41 (m, 0.75H), 2.38-2.30 (m,0.75H), 2.01-1.98 (m, 1H), 1.92-1,75 (m, 7H), 1.68-1.57(m, 4H), 1.53-1.40 (m, 6H), 1.36–1.22 (m, 6H), 1.17–1.01 (m, 3H), 0.94–0.89 (m, 6H), 0.67 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 203.00 (t, *J* = 25.9 Hz), 71.58, 71.50, 55.85, 55.08, 43.91, 42.57, 40.90 (t, *J* = 2.8 Hz), 40.26, 39.31, 37.43, 37.02, 35.38, 35.06, 34.22, 30.47, 28.81, 28.09, 27.03, 23.52, 21.31, 18.63, 12.28. **HRMS** (ESI) calcd for $C_{24}H_{39}DO_3Na$ $[M+Na]^+$ 400.2932, found 400.2918.

4-Formylphenyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1*H*-indol-3-yl)acetate-*formyl-d1* (3ap)



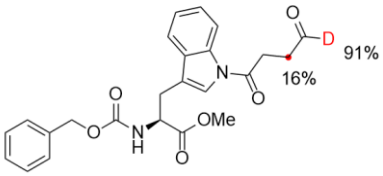
The reaction was carried out according to the general procedure **A**. (*R*)-4-((3*R*,5*S*,7*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[*a*]phena -nthren-17-yl)pentanal (46.1 mg, 0.1 mmol, 1.0 equiv), 4CzIPN (4.0 mg, 0.005 mmol, 5 mol%), **2d** (30 uL, 1M in EtOAc, 30 mol%), D_2O (80 uL, 40 equiv) and anhydrous DCE/anhydrous EtOAc (0.8 ml/0.2 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/hexane/DCM= 1/3/1) to afford product (23mg, 50% yield, 92% D ratio by ^{1}H NMR) as white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 0.08H), 7.90 (d, *J* = 8.5 Hz, 2H), 7.68 (d, *J* = 8.4 Hz, 2H), 7.48 (d, *J* = 8.3 Hz, 2H), 7.25 (d, *J* = 8.1 Hz, 2H), 7.04 (s, 1H), 6.88 (d, *J* = 9.0 Hz, 1H), 6.70 (dd, *J* = 9.0, 2.1 Hz, 1H), 3.94 (s, 1.49H), 3.84 (s, 3H), 2.47 (s, 2.25H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 190.65 (t, *J* = 26.4 Hz), 168.74, 168.43, 156.30, 155.46, 139.61, 136.56, 134.16 (t, *J* = 3.4 Hz), 133.85, 131.36, 131.34, 131.00, 130.50, 129.33, 122.36, 115.22, 111.91, 111.59, 101.35, 55.90, 30.74, 13.56. **HRMS** (ESI) calcd for $C_{26}H_{20}DClNO_5$ $[M+H]^+$ 463.1166, found 463.1151; calcd for $C_{26}H_{19}D_2ClNO_5$ $[M+Na]^+$ 464.1228, found 464.1207; calcd for $C_{26}H_{18}D_3ClNO_5$ $[M+Na]^+$ 465.1291, found 465.1260.

(3*aR*,4*R*,6*S*,6*aS*)-6-methoxy-2,2-dimethyl-*N*-(3-oxopropyl)tetrahydrofuro[3,4-*d*][1,3]dioxole-4-carboxamide-*formyl-d1* (3aq)



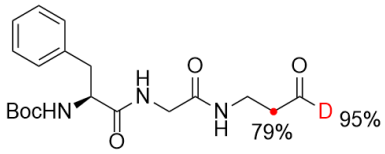
The reaction was carried out according to the general procedure **B**. (3*aR*,4*R*,6*S*,6*aS*)-6-methoxy-2,2-dimethyl-*N*-(3-oxopropyl)tetrahydrofuro[3,4-*d*][1,3]dioxole-4-carboxamide (54.6 mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc (1.0 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/MeOH= 19/1-9/1) to afford product (30mg, 55% yield, 97% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.80 (s, 0.03H), 6.98 (br, 1H), 5.06 (dd, *J* = 6.0, 1.3 Hz, 1H), 5.04 (s, 1H), 4.57 (s, 1H), 4.51 (d, *J* = 6.0 Hz, 1H), 3.55 (t, *J* = 5.5 Hz, 1.77H), 3.44 (s, 3H), 2.75-2.68 (m, 0.49H), 1.47 (s, 3H), 1.31 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 201.18-200.58 (m), 170.37, 112.88, 111.66, 86.63, 84.58, 82.75, 56.65, 43.71-43.10 (m), 32.52-32.42 (m), 26.60, 25.07. **HRMS** (ESI) calcd for $C_{12}H_{18}DNO_6Na$ $[M+Na]^+$ 297.1167, found 297.1151; calcd for $C_{12}H_{18}D_2NO_6$ $[M+H]^+$ 267.1411, found 276.1397.

Methyl *N*^{a}-((benzyloxy)carbonyl)-1-(4-oxobutanoyl-4-*d*)-*L*-tryptophanate-*formyl-d1* (3ar)



The reaction was carried out according to the general procedure **B**. Methyl *N*^{a}-((benzyloxy)carbonyl)-1-(4-oxobutanoyl-4-*d*)-*L*-tryptophanate (43.7 mg, 0.1 mmol, 1.0 equiv), 4CzIPN (4.0 mg, 0.005 mmol, 5 mol%), **2d** (30 uL, 1M in EtOAc, 30 mol%), D_2O (80 uL, 40 equiv) and anhydrous EtOAc/anhydrous DCE (0.5 mL/0.5 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by preparative TLC plate (EtOAc/hexane/DCM= 1/2/2) to afford product (29mg, 66% yield, 91% D ratio by ^{1}H NMR) as pale yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) 9.90 (s, 0.09H), 8.37 (d, *J* = 8.2 Hz, 1H), 7.47 (d, *J* = 7.8 Hz, 1H), 7.36–7.23 (m, 7H), 5.41 (d, *J* = 8.0 Hz, 1H), 5.12 (dd, *J* = 30.3, 12.2 Hz, 2H), 4.77 (dd, *J* = 7.6 Hz, 6.0 Hz, 1H), 3.70 (s, 3H), 3.36–3.09 (m, 4H), 2.97 (t, *J* = 6.3 Hz, 1.68H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 199.64 (t, *J* = 26.8 Hz), 172.05, 169.44, 155.82, 136.32, 135.94, 130.44, 128.68, 128.37, 128.25, 125.73, 123.89, 122.67, 118.87, 117.41, 116.77, 67.19, 53.91, 52.72 (q, *J* = 3.7 Hz), 37.93, 37.74 (t, *J* = 3.3 Hz), 28.29, 28.19. **HRMS** (ESI) calcd for $C_{24}H_{24}DN_2O_6$ $[M+H]^+$ 438.1770, found 438.1759.

***tert*-Butyl (*S*)-(1-oxo-1-((2-oxo-2-((3-oxopropyl)amino)ethyl)amino)-3-phenylpropan-2-yl)carbamate-*formyl-d1* (3as)**

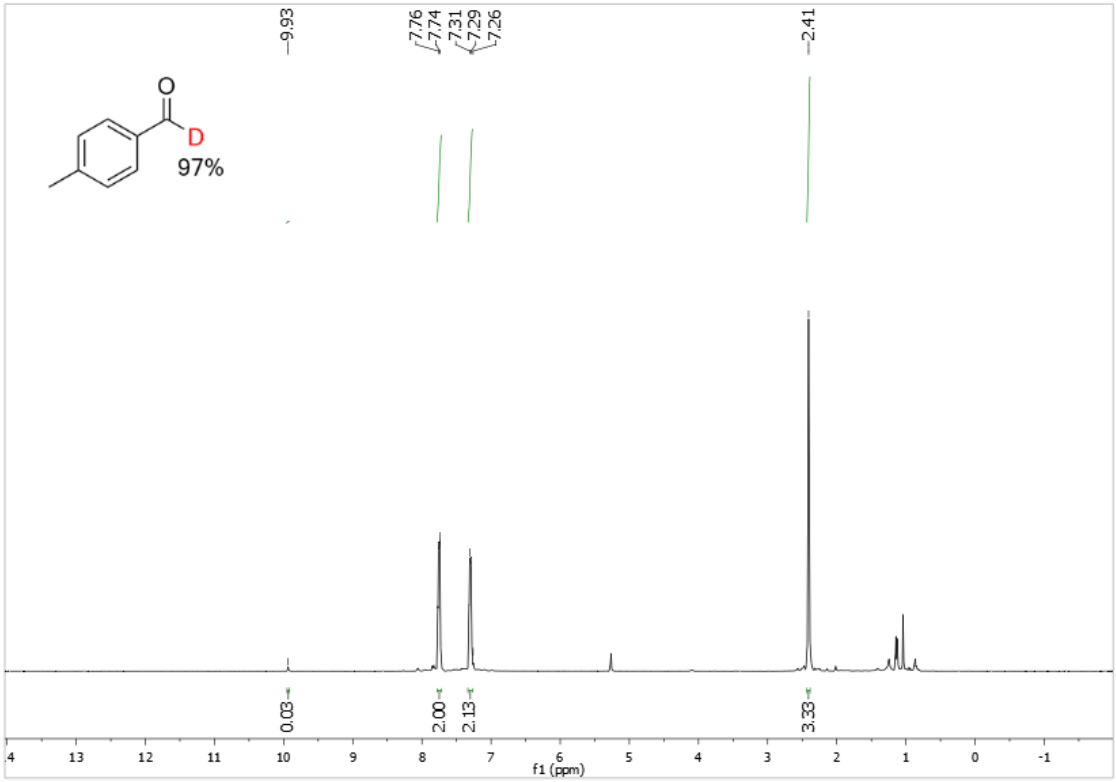


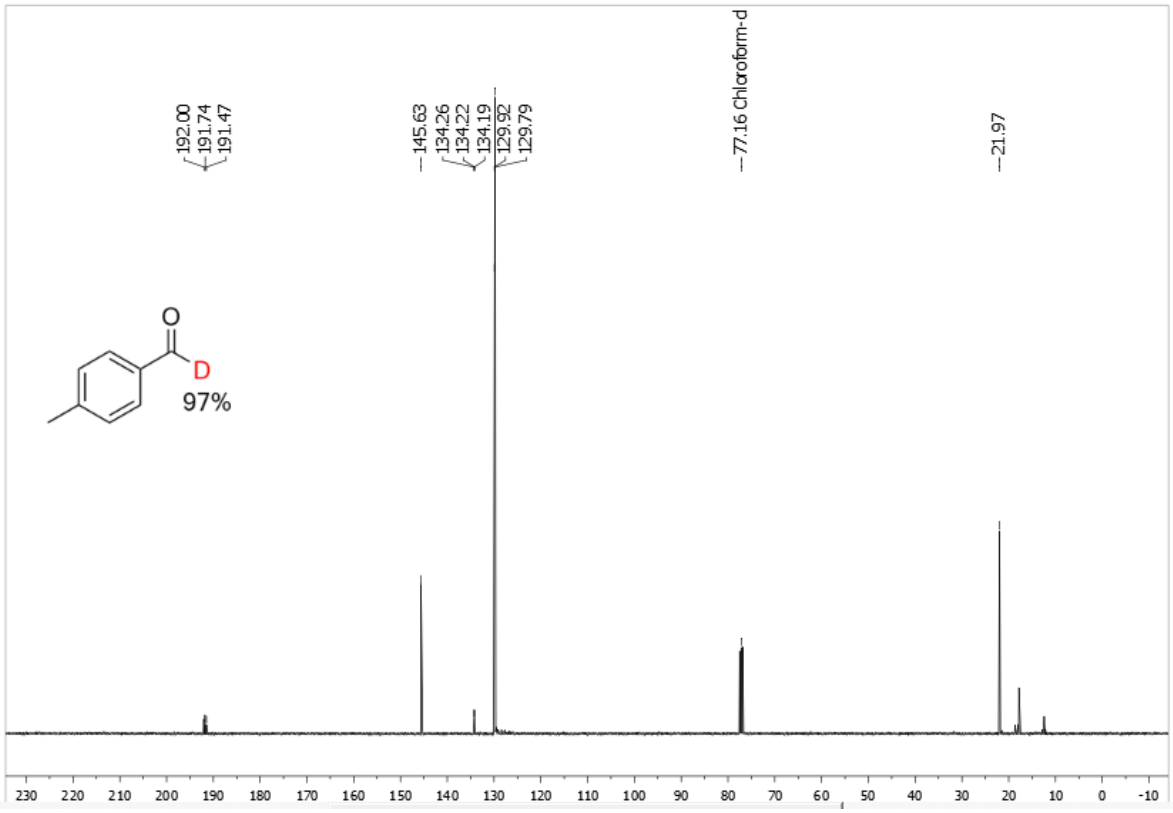
The reaction was carried out according to the general procedure **B**. *tert*-Butyl (*S*)-(1-oxo-1-((2-oxo-2-((3-oxopropyl)amino)ethyl)amino)-3-phenylpropan-2-yl)carbamate (75.4mg, 0.2 mmol, 1.0 equiv), 4CzIPN (8.0 mg, 0.01 mmol, 5 mol%), **2d** (60 uL, 1M in EtOAc, 30 mol%), D_2O (160 uL, 40 equiv) and anhydrous EtOAc anhydrous DCE (0.5 mL/0.5 mL) were used. The reaction mixture was irritated by blue LED strip for 36 h. The residue was purified by flash column chromatography (EtOAc/MeOH = 9/1) to afford product (66mg, 88% yield, 95% D ratio by ^{1}H NMR) as colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) 9.75 (s, 0.09H), 7.32-7.18

(m, 5H), 6.85 (s, 2H), 5.17 (s, 1H), 4.31 (dd, *J* = 14.1, 7.1 Hz, 1H), 3.90 (dd, *J* = 16.7, 6.0 Hz, 1H), 3.78 (dd, *J* = 16.7, 5.5 Hz, 1H), 3.51-3.48 (m, 2H), 3.12 (dd, *J* = 13.9, 6.3 Hz, 1H), 2.98 (dd, *J* = 13.6, 7.7 Hz, 1H), 2.68 (t, *J* = 6.2 Hz, 0.42H), 1.38 (s, 9H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 200.95 (t, *J* = 26.2 Hz), 172.14, 169.03, 155.94, 136.51, 129.31, 128.86, 127.21, 80.71, 56.41,43.21, 38.13, 33.09, 28.38. **HRMS** (ESI) calcd for $C_{19}H_{27}DN_3O_5$ $[M+H]^+$ 379.2086, found 379.2075; $C_{19}H_{26}D_2N_3O_5$ $[M+H]^+$ 380.2149, found 380.2136; $C_{19}H_{25}D_3N_3O_5$ $[M+H]^+$ 381.2212, found 381.2195.

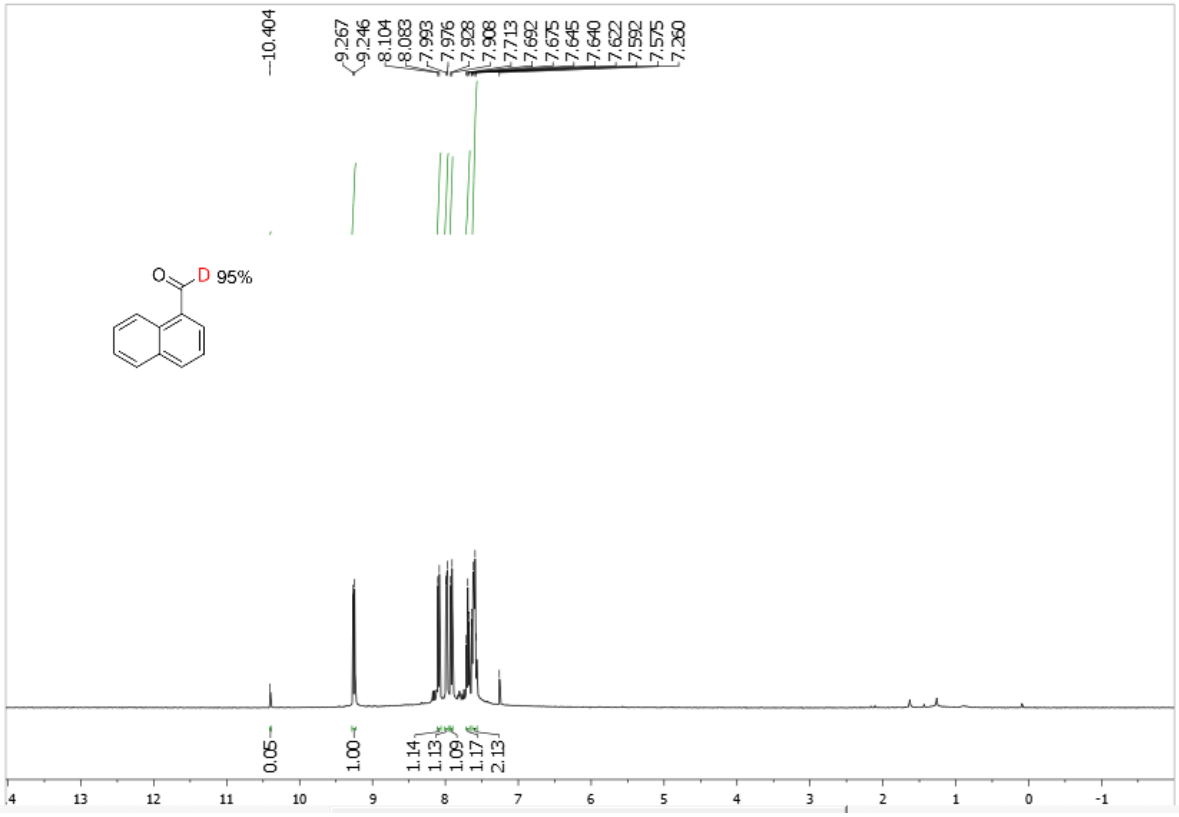
9. Spectra data

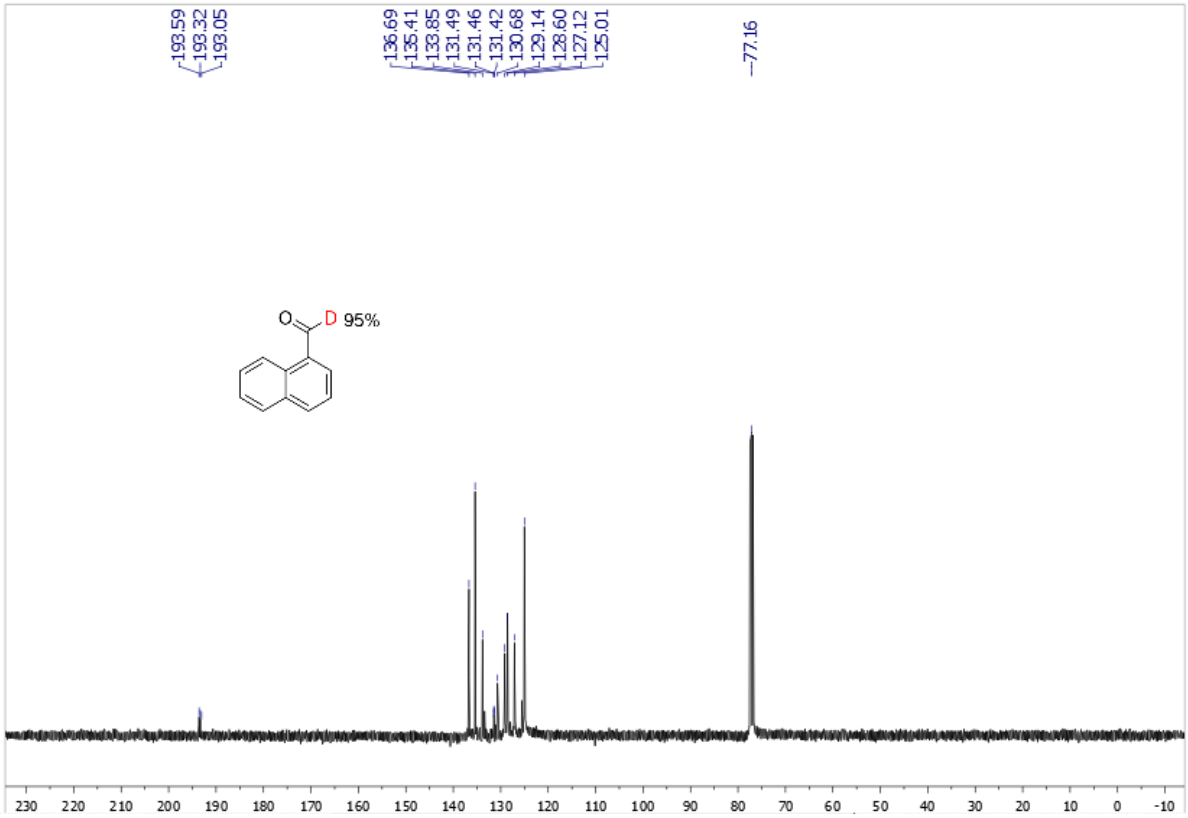
4-Methylbenzaldehyde-*formyl-d1 (3a)*



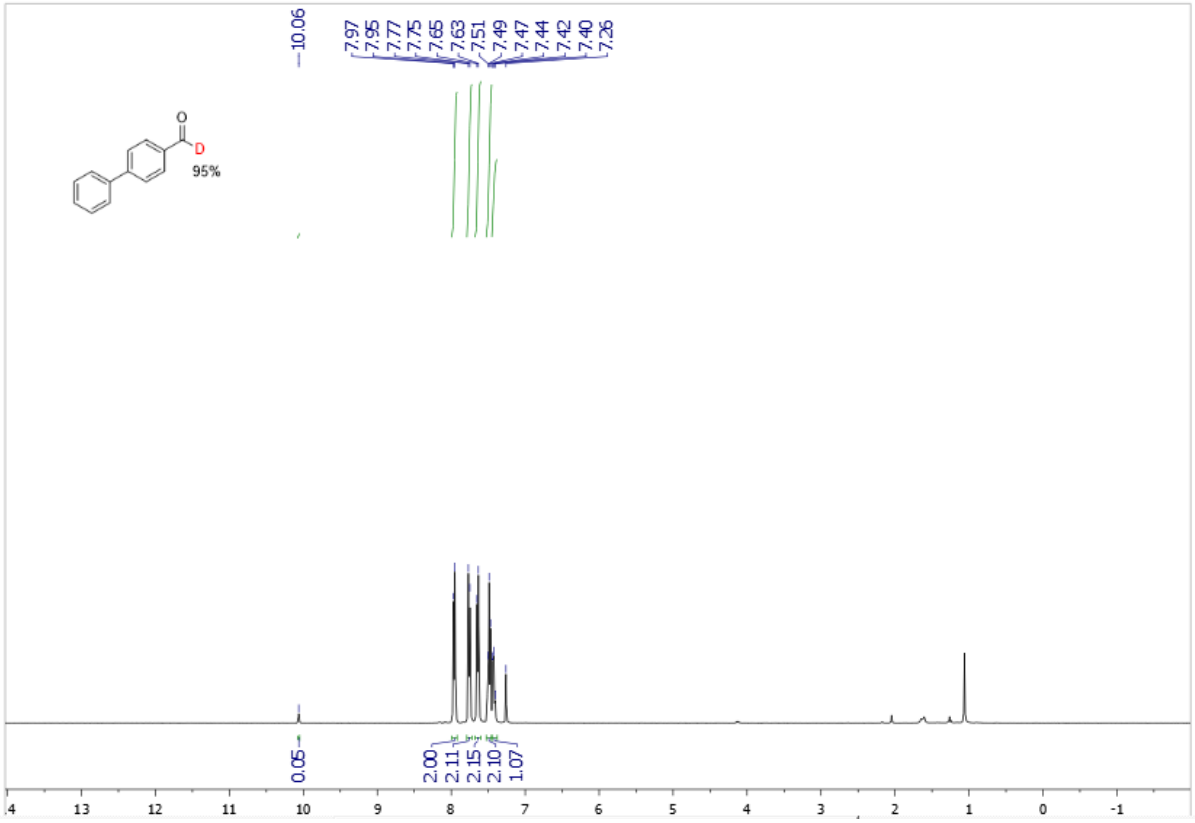


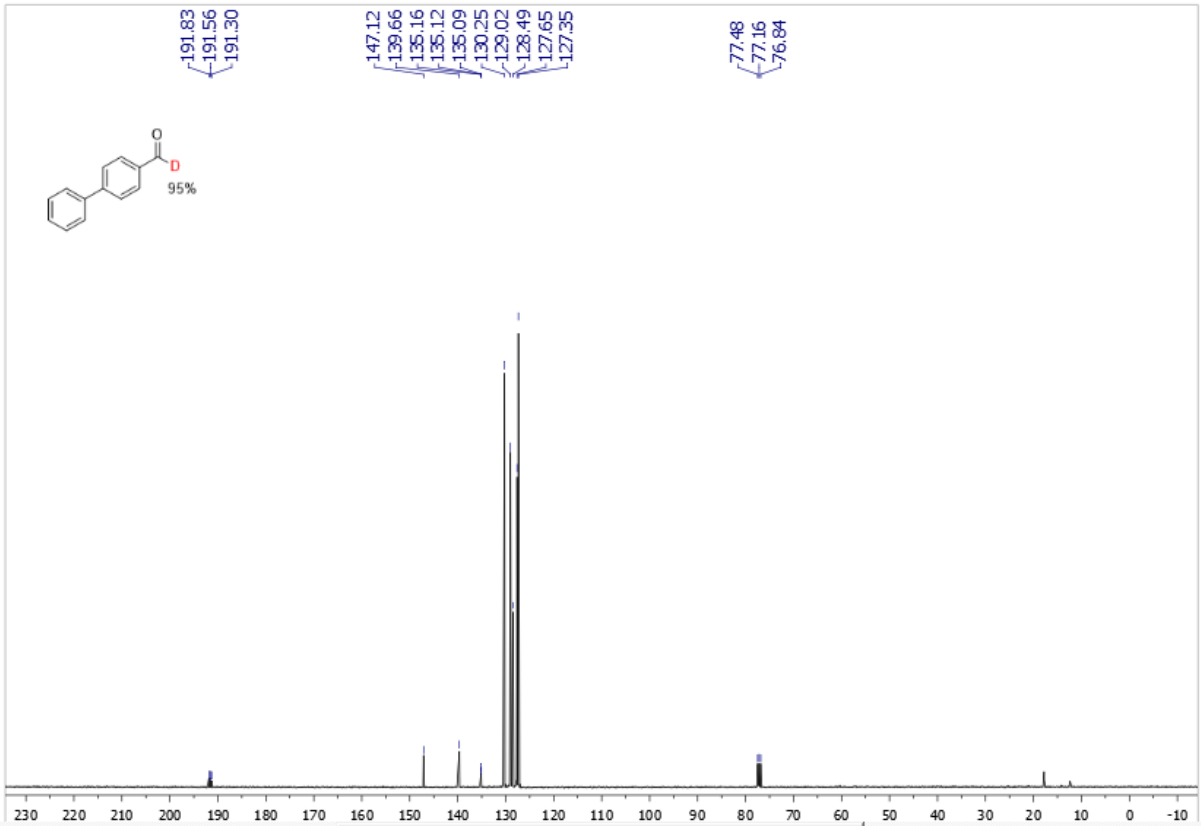
1-Naphthaldehyde-*formyl-d1 (3b)*



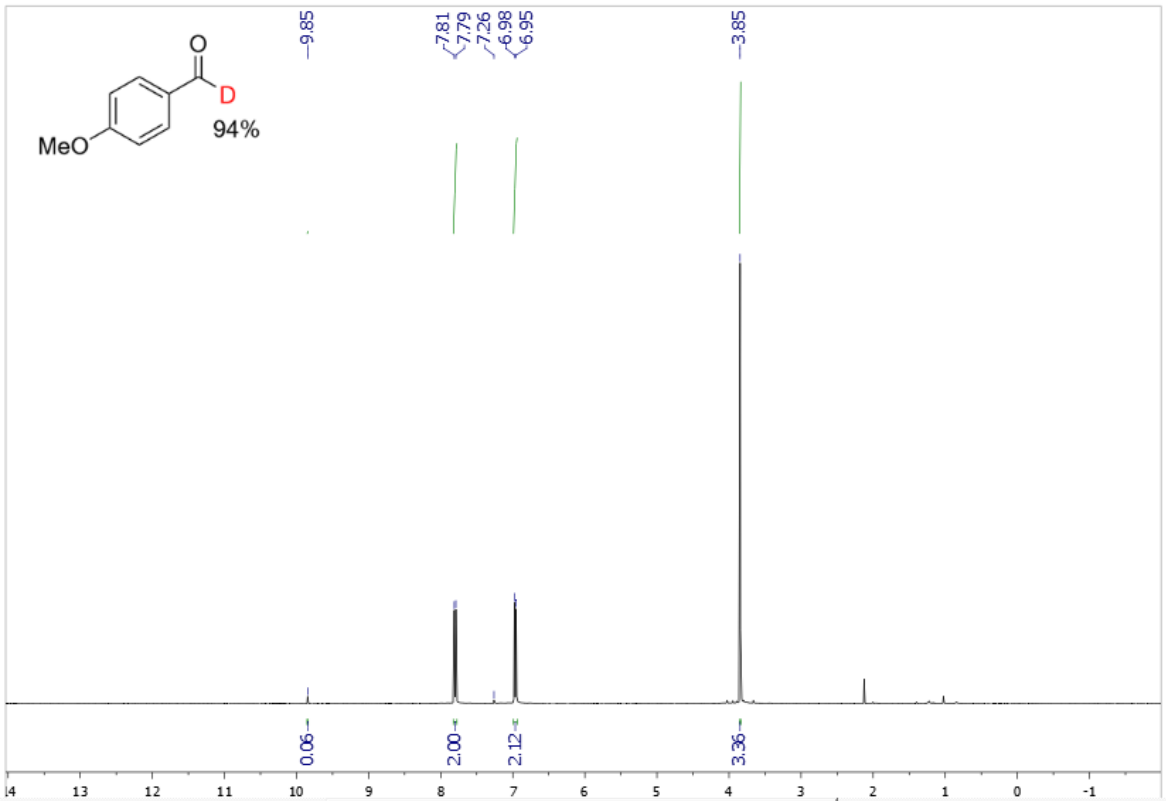


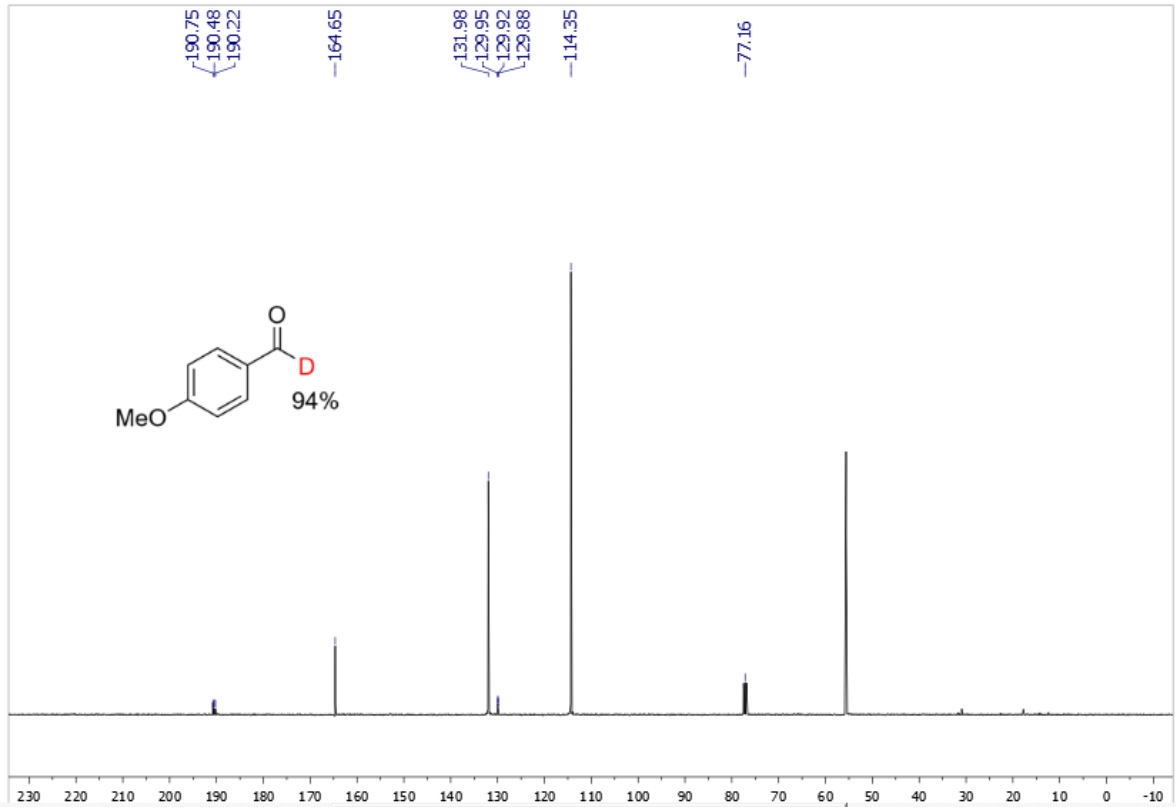
[1,1'-Biphenyl]-4-carbaldehyde-*formyl-d1 (3c)*



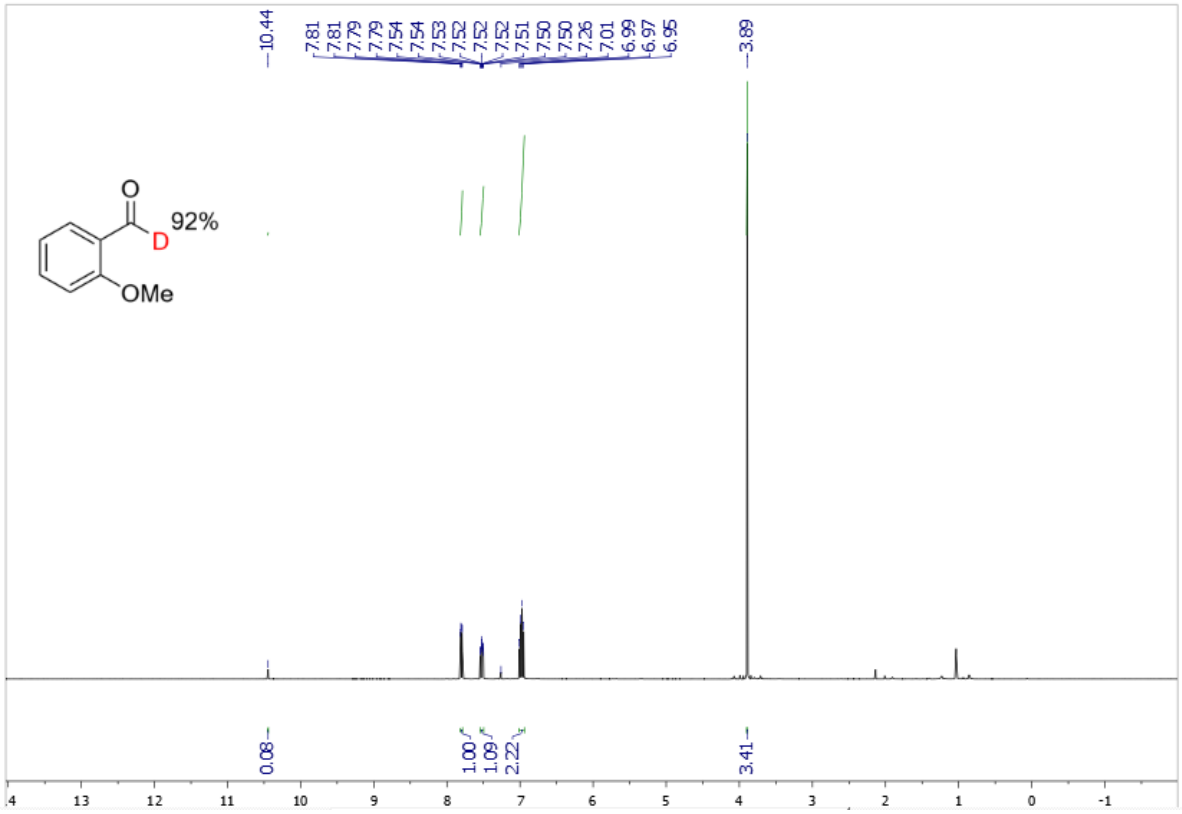


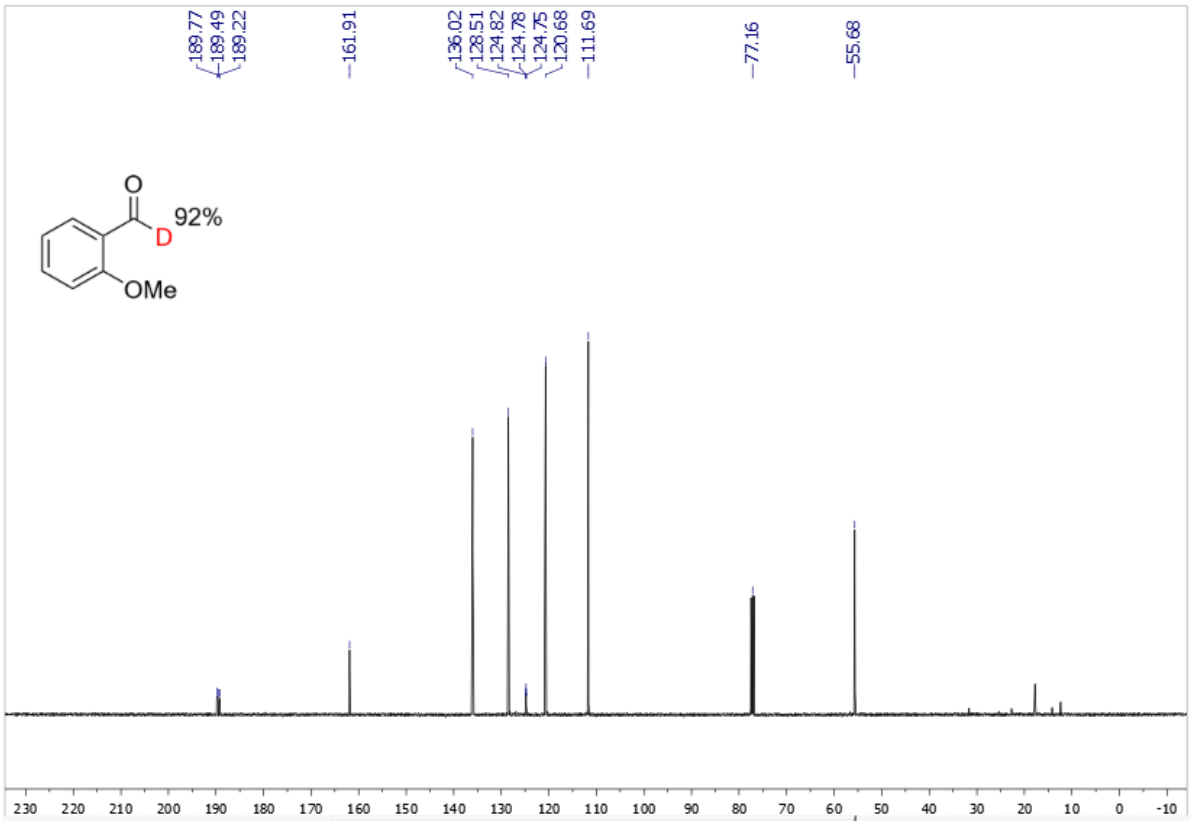
4-Methoxybenzaldehyde-*formyl-d1 (3d)*



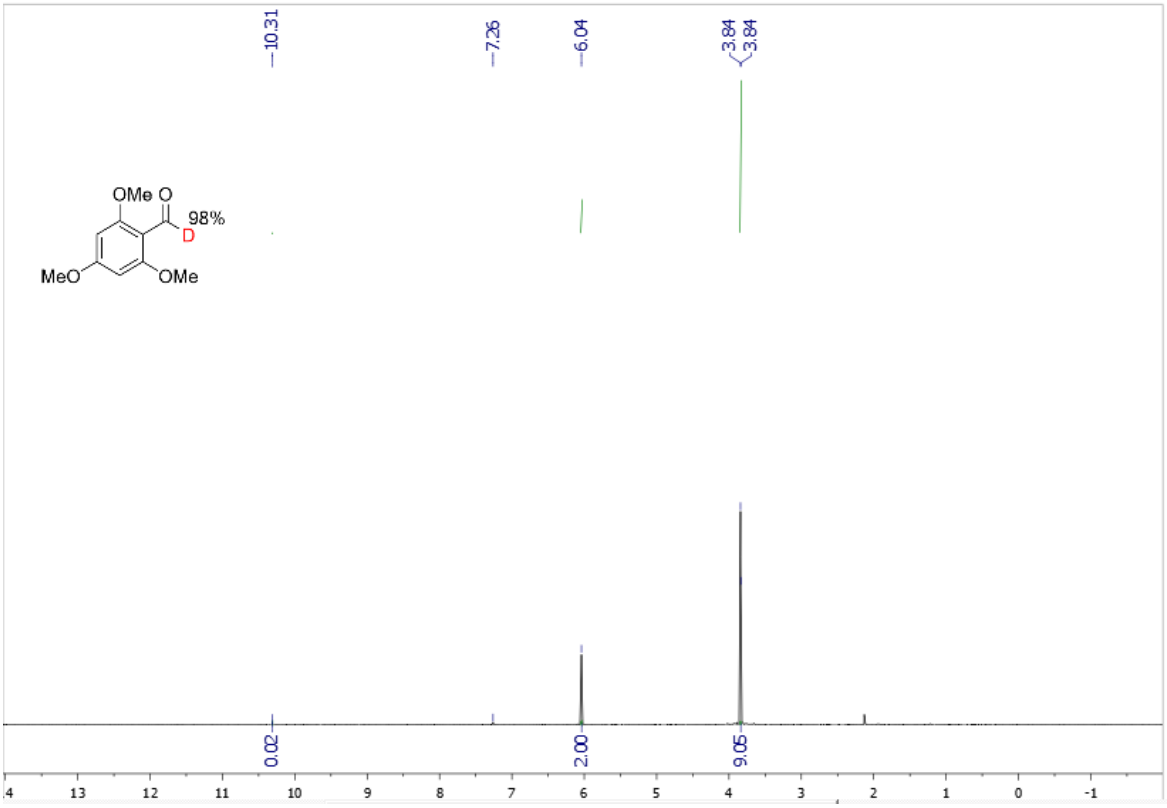


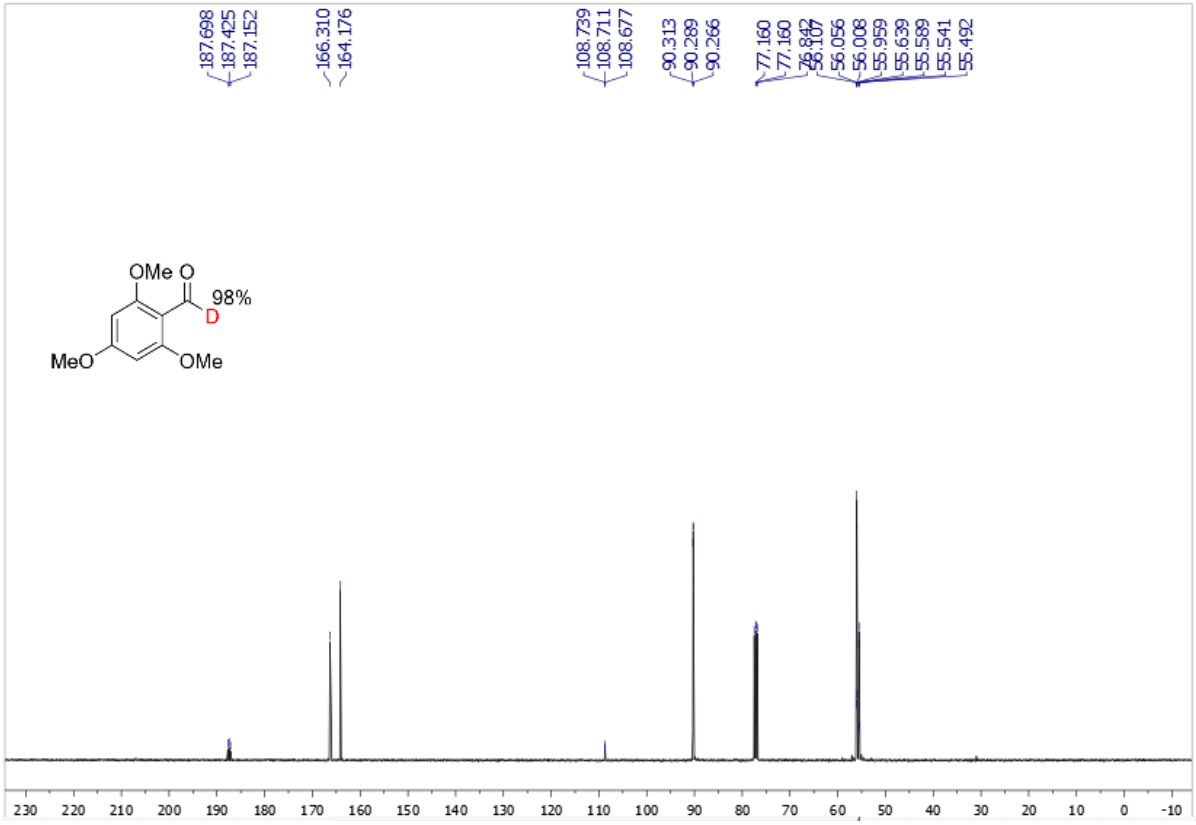
2-Methoxybenzaldehyde-*formyl-d1 (3e)*



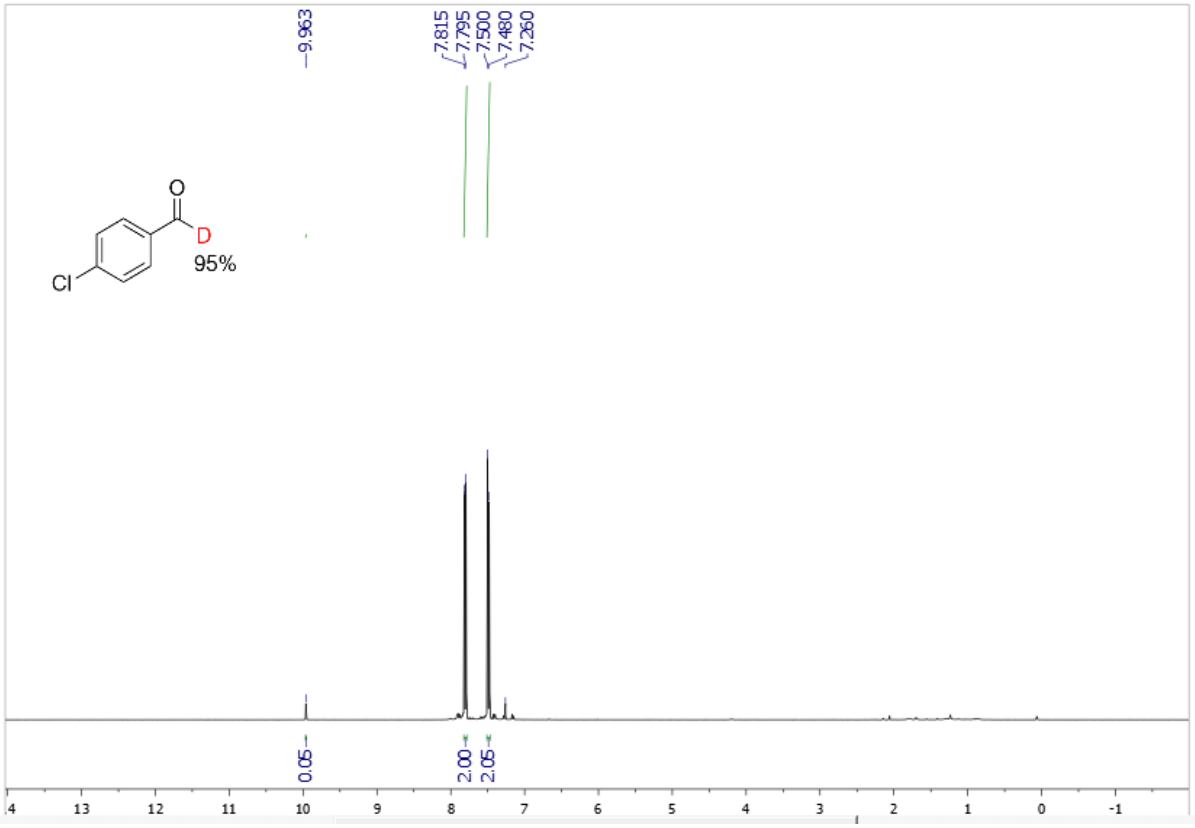


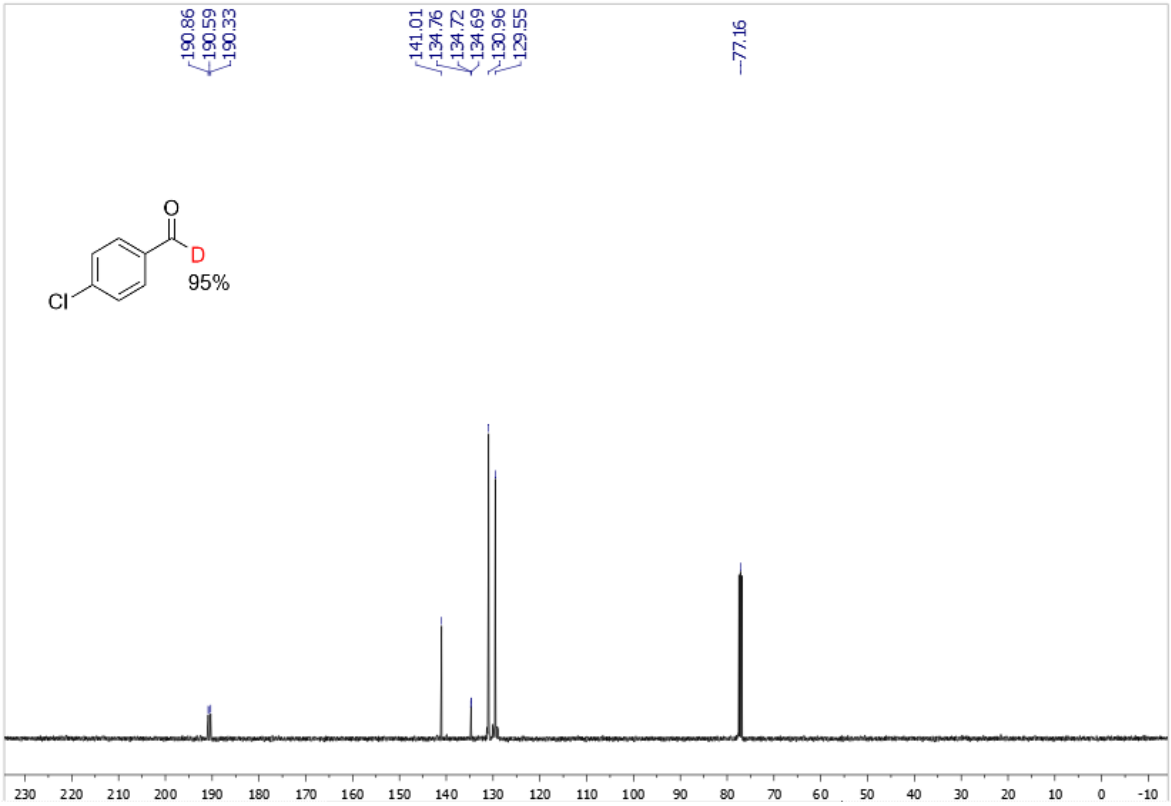
2,4,6-Trimethoxybenzaldehyde--*formyl-d1 (3f)*



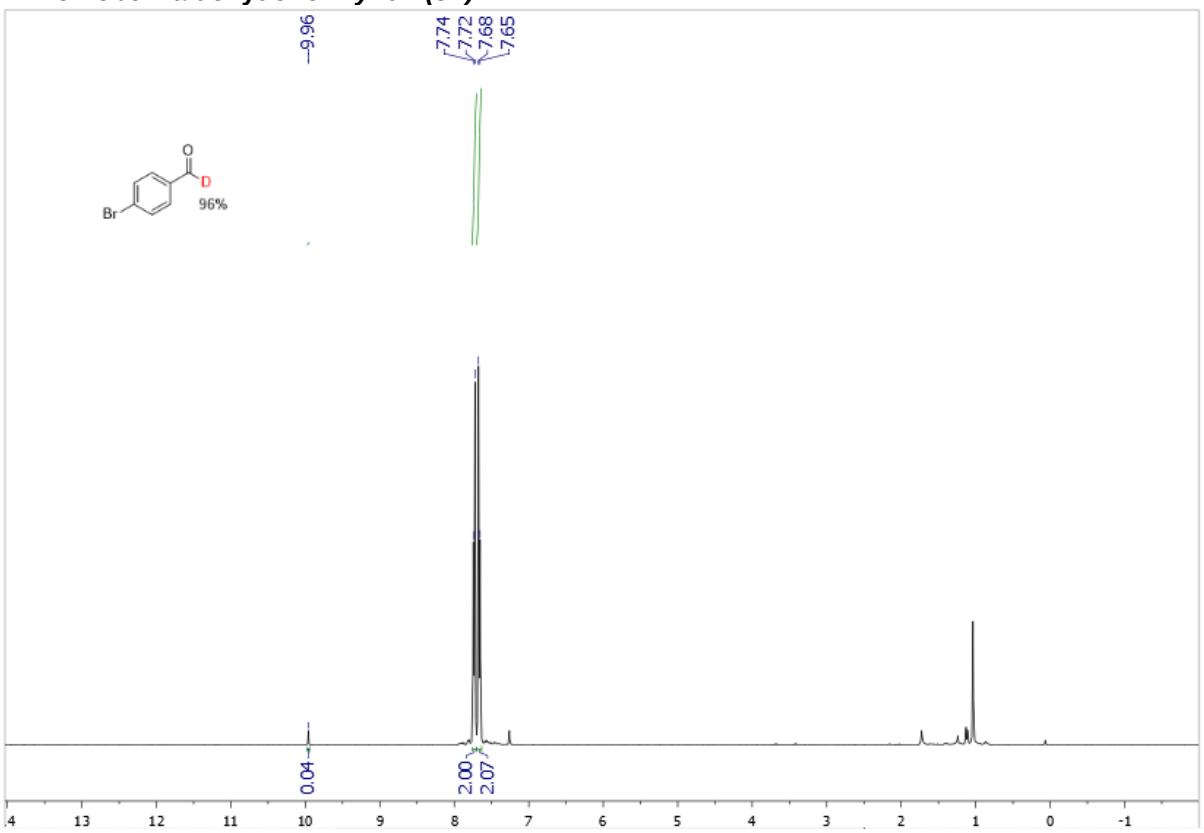


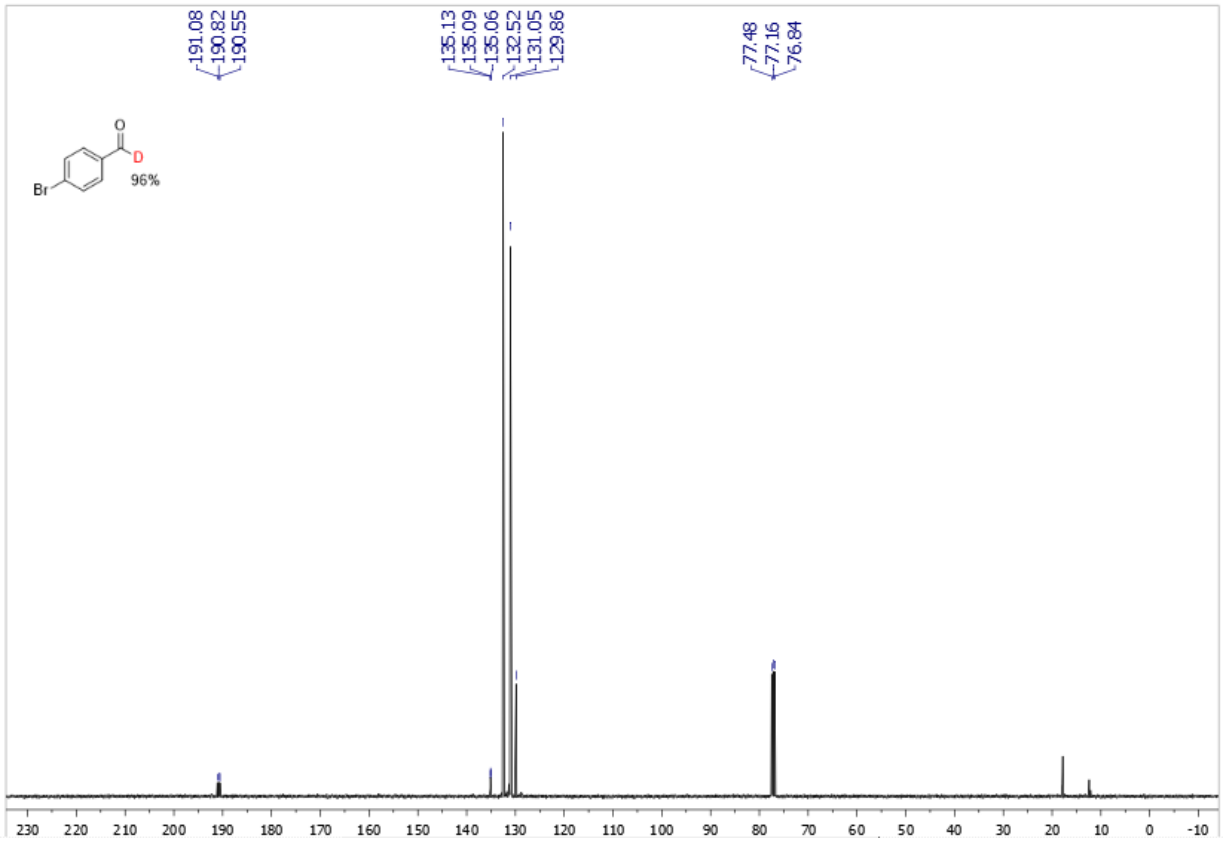
4-Chlorobenzaldehyde-*formyl-d1* (3g)



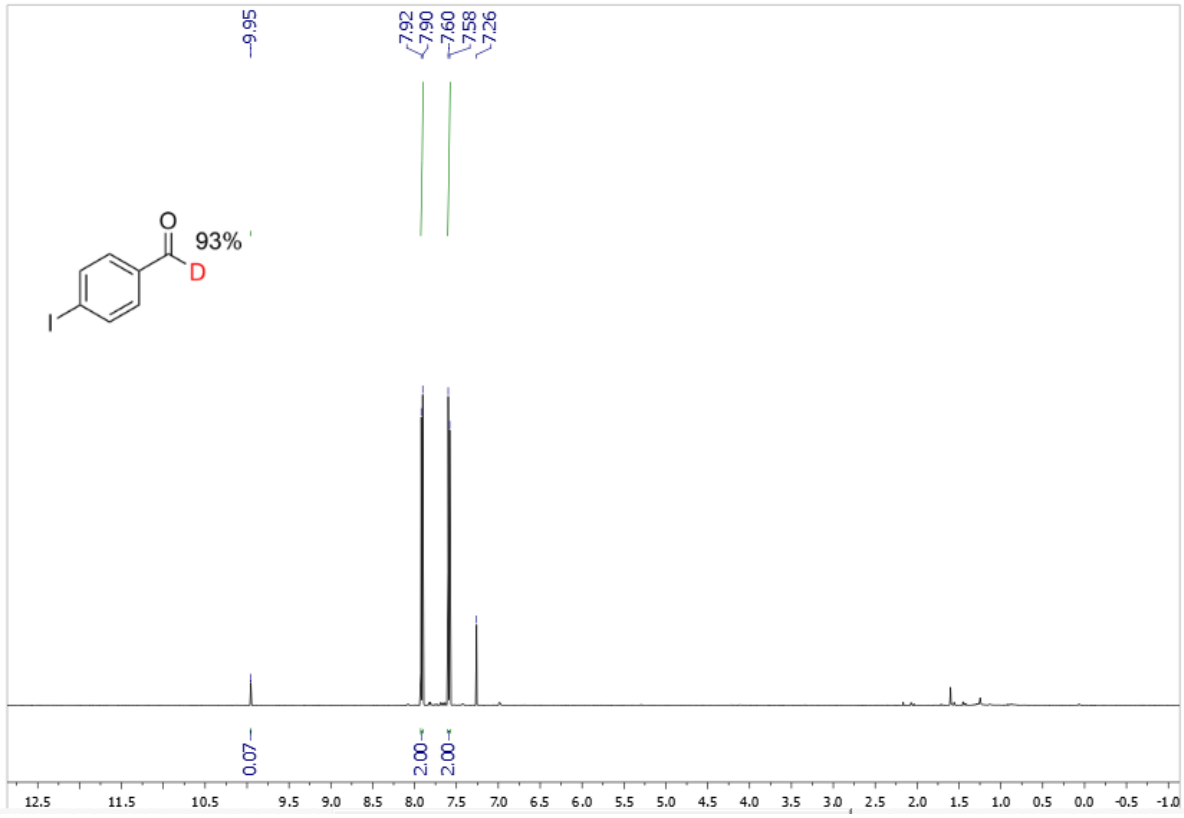


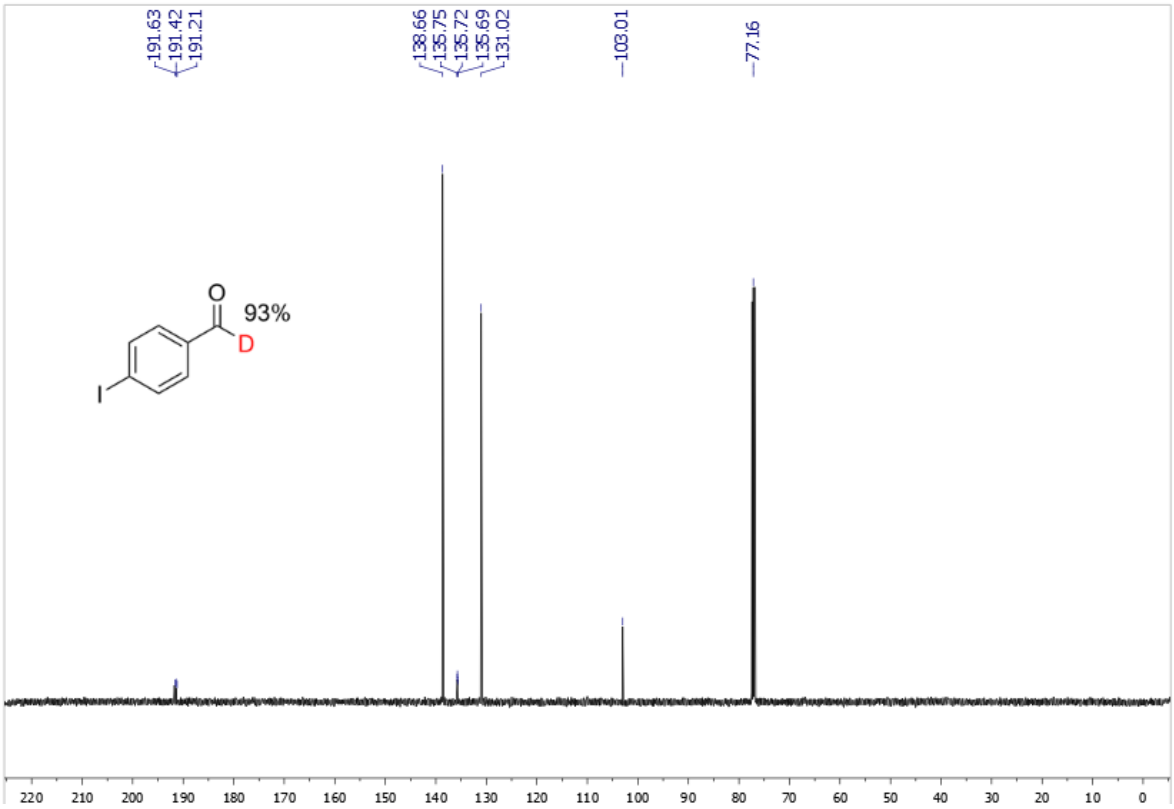
4-Bromobenzaldehyde-*formyl-d1 (3h)*



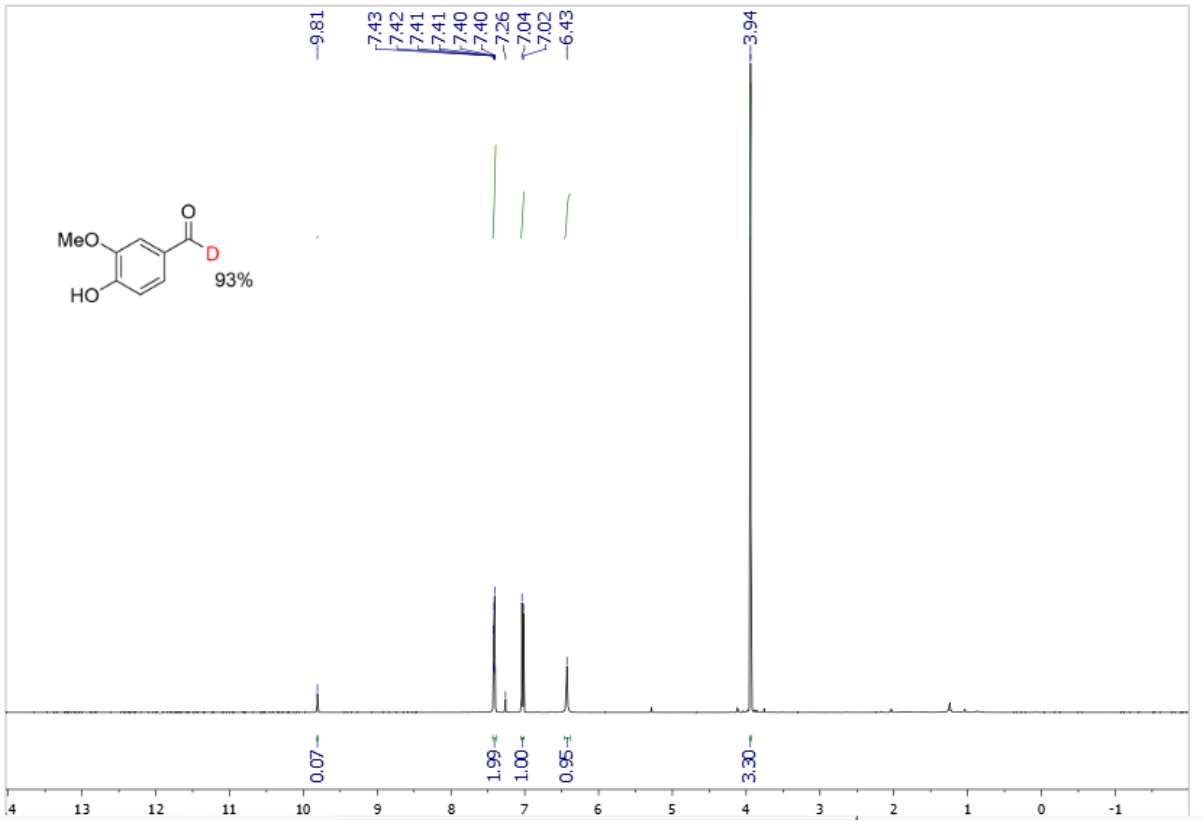


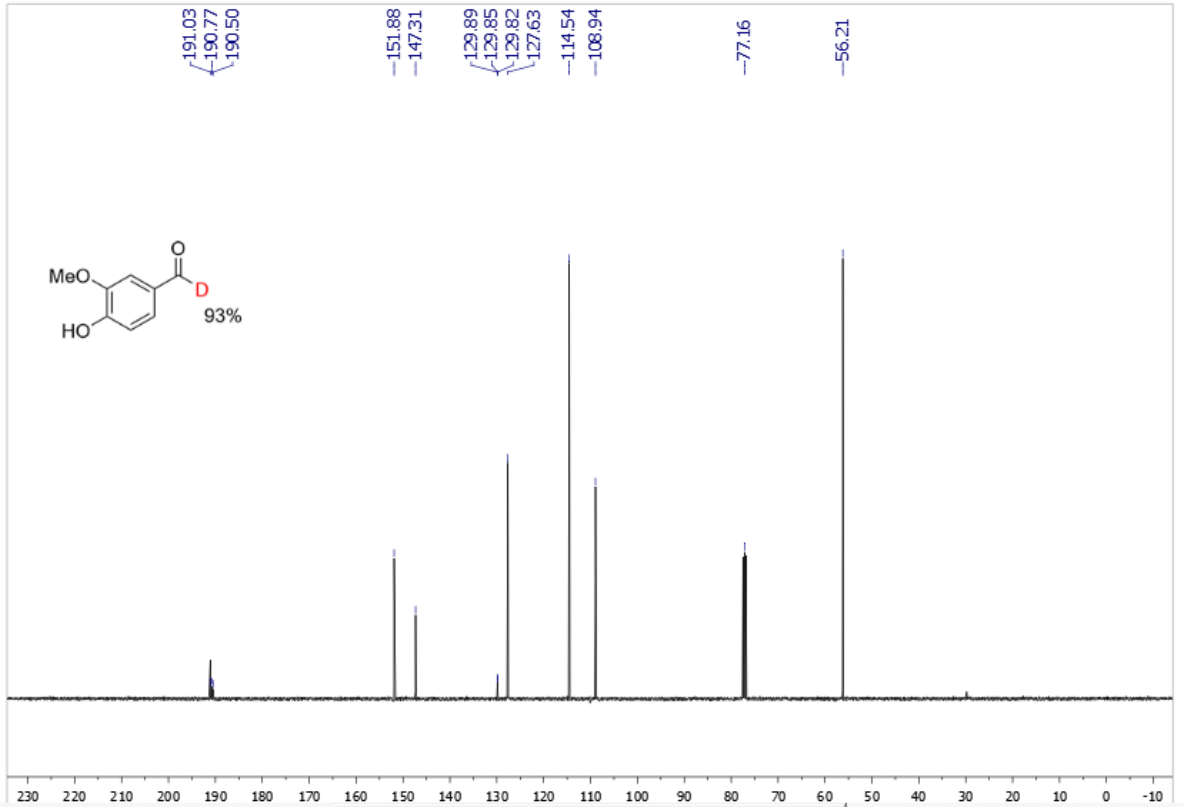
4-Iodobenzaldehyde-*formyl-d1 (3i)*



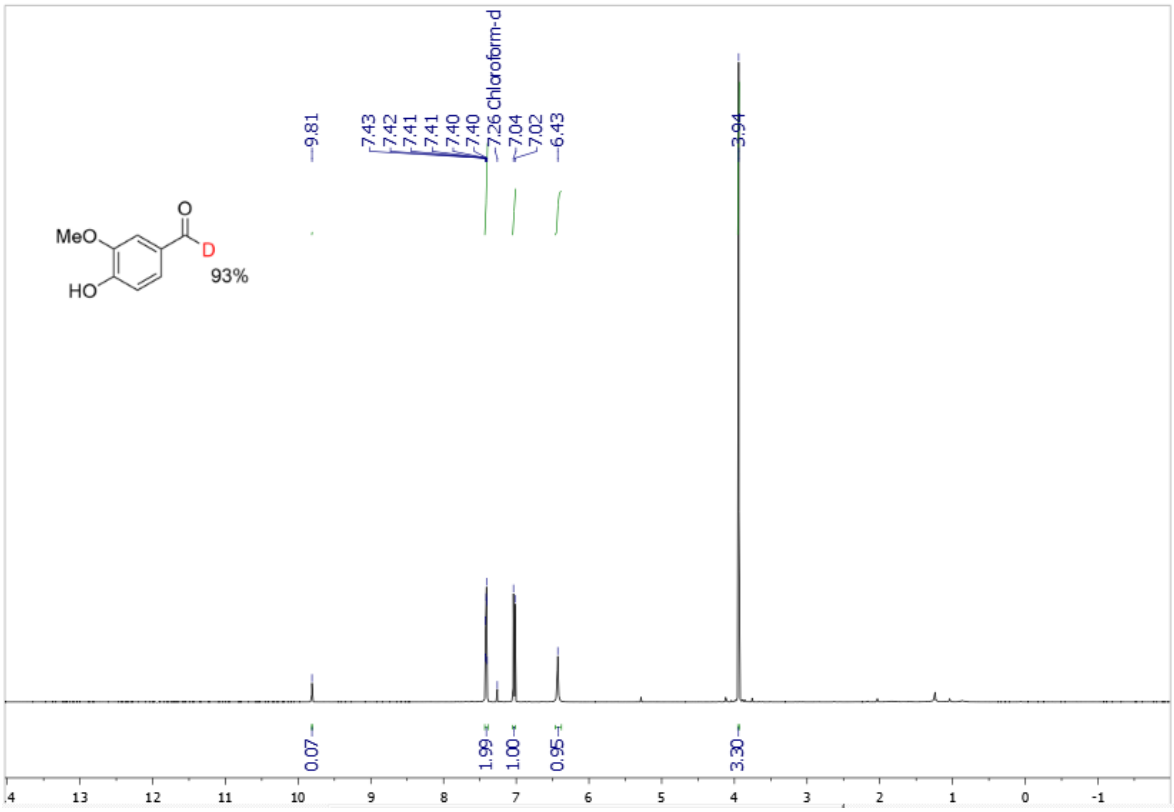


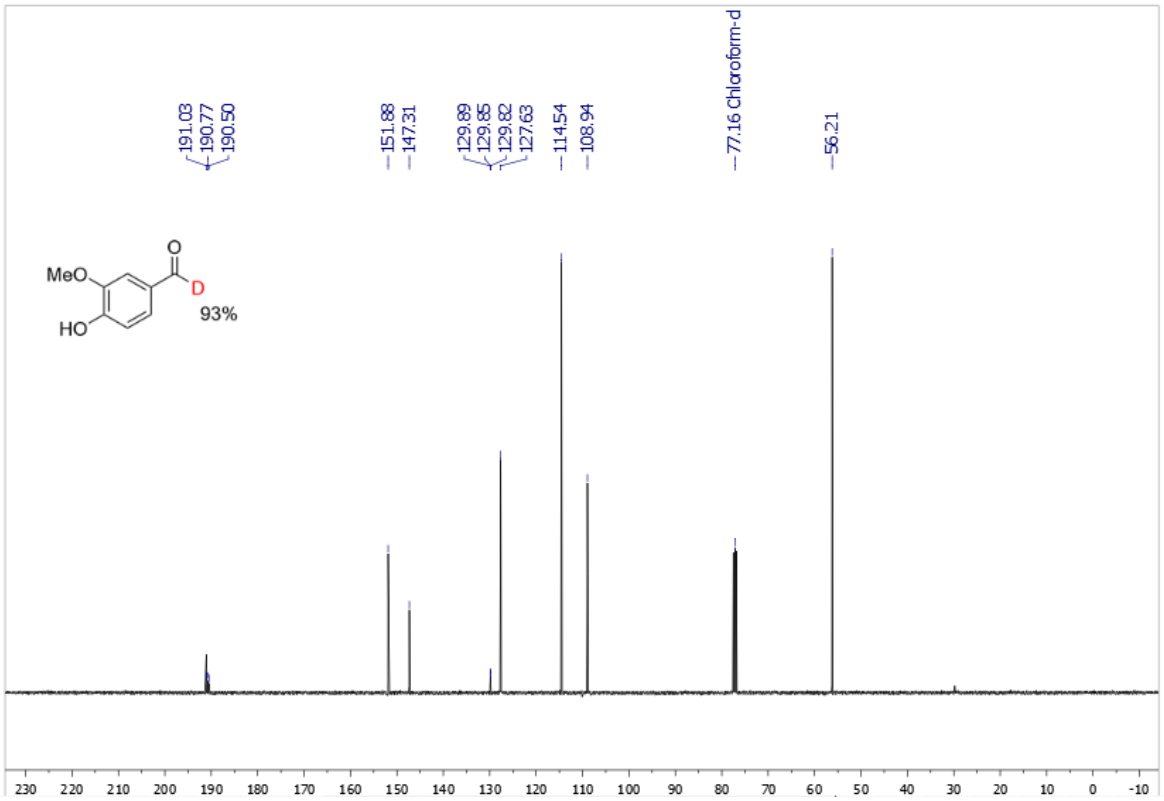
4-Hydroxybenzaldehyde-*formyl-d1 (3J)*



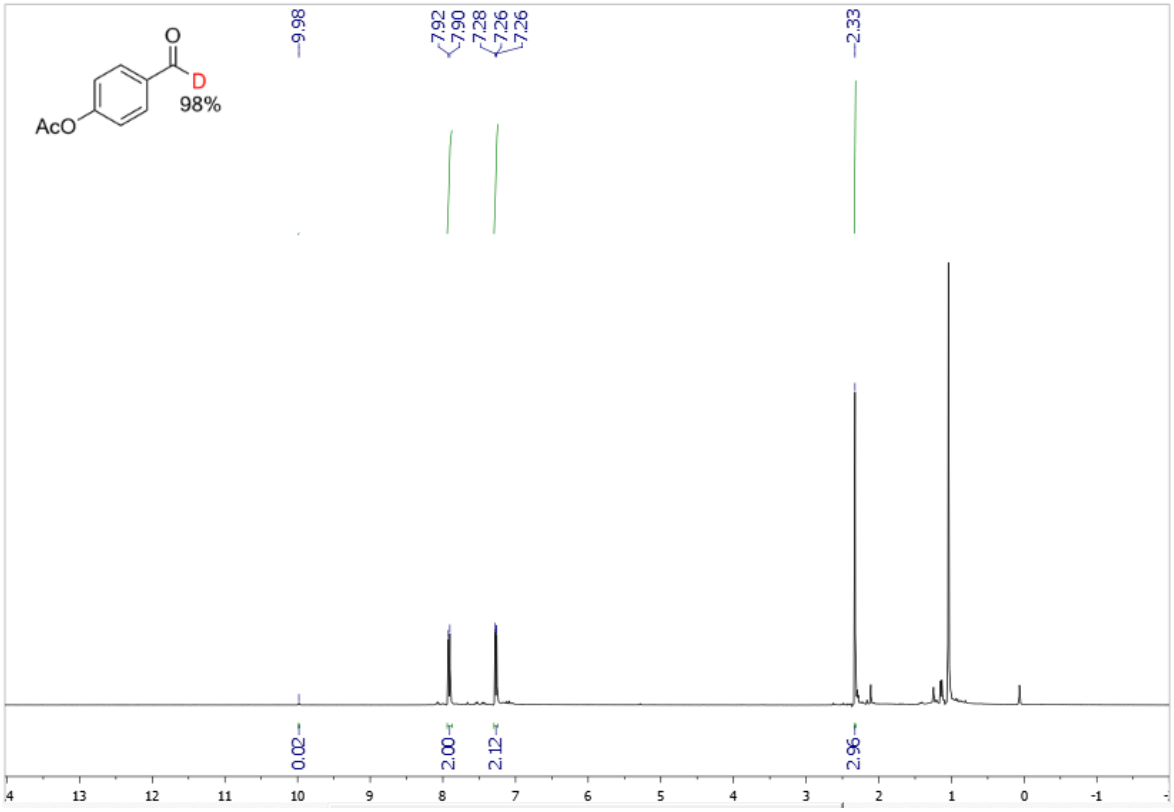


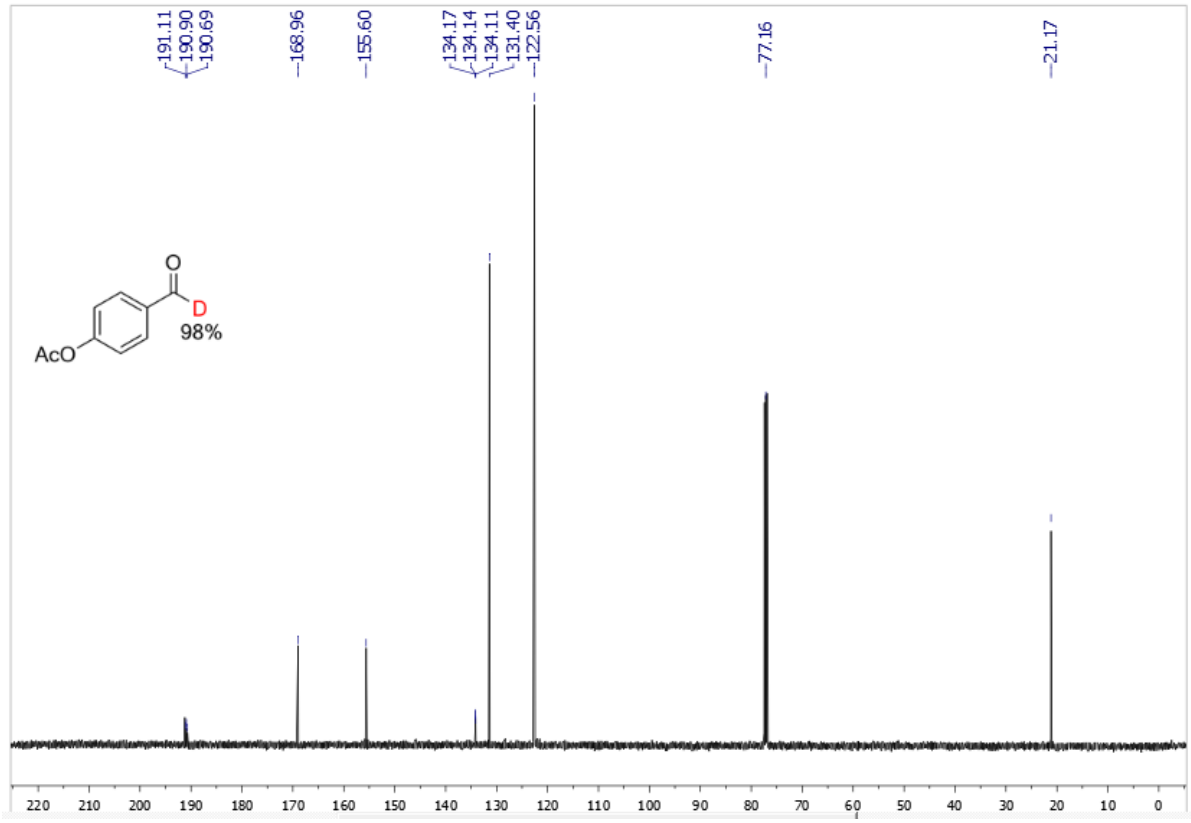
4-Hydroxy-3-methoxybenzaldehyde-*formyl-d1 (3k)*



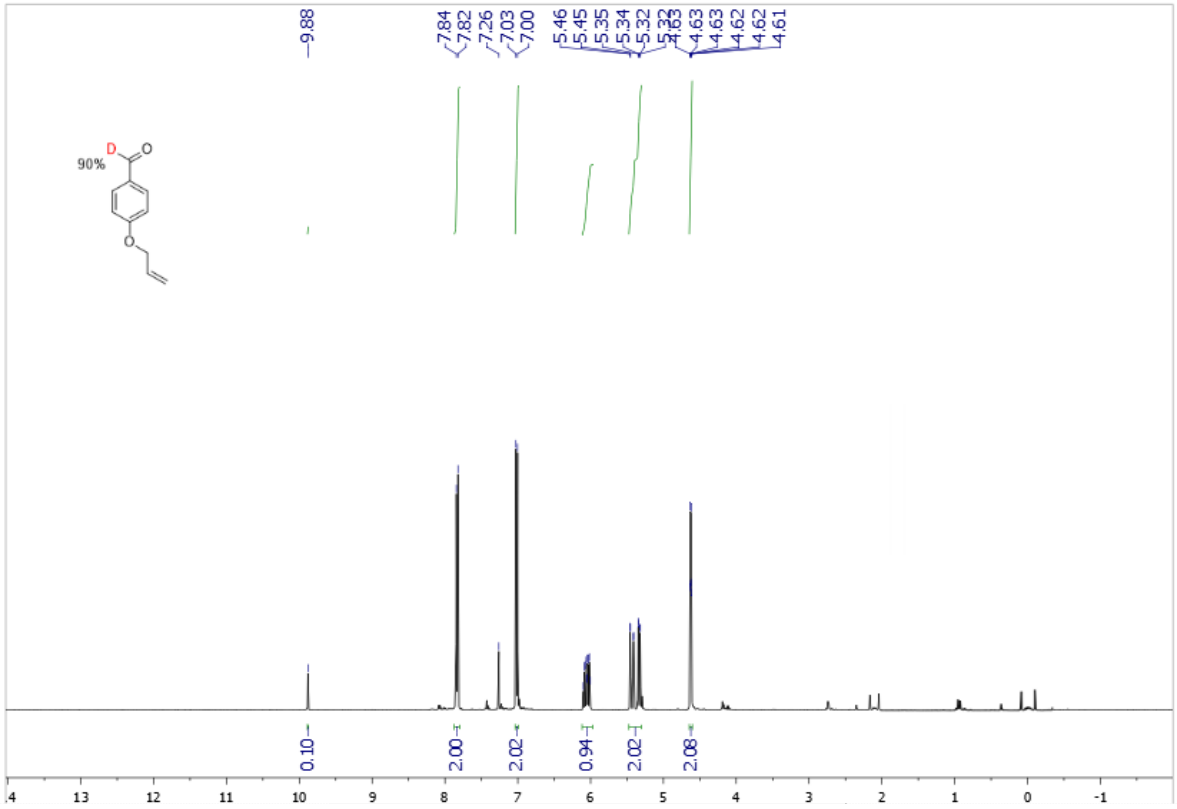


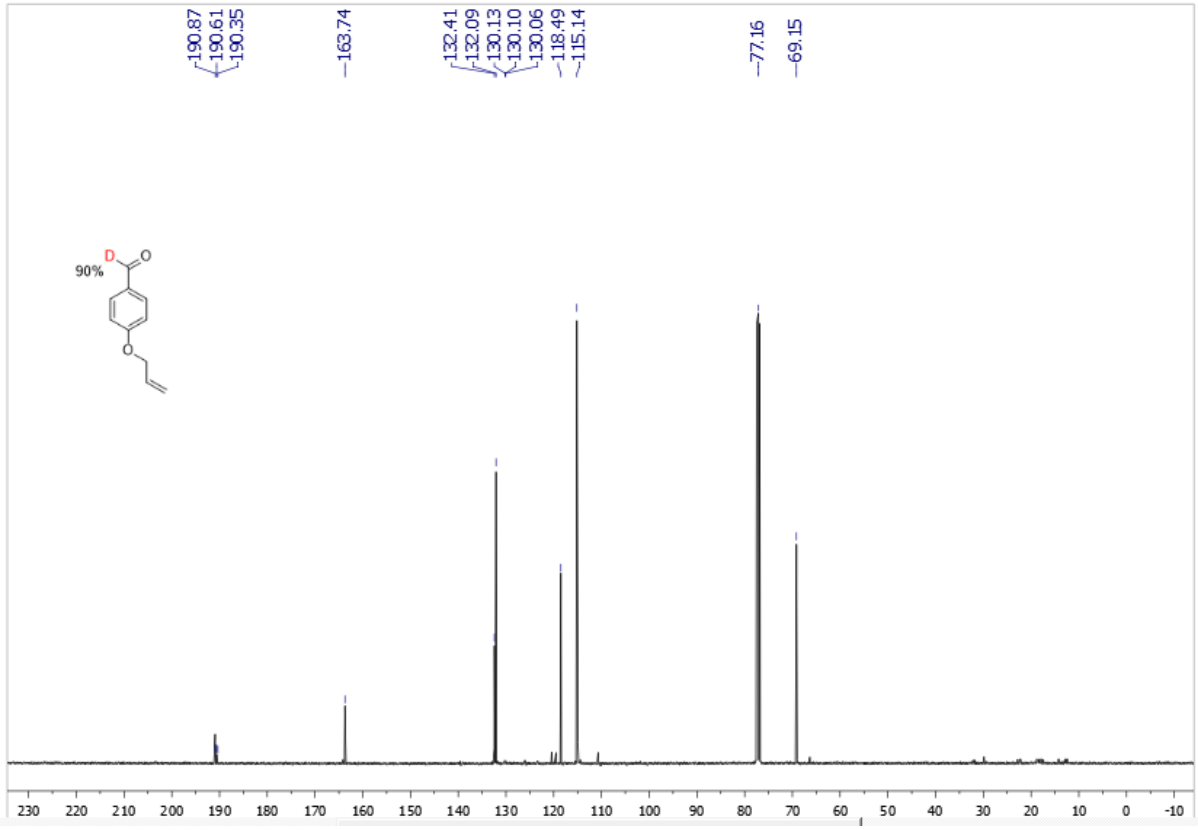
4-Formylphenyl acetate-*formyl-d1 (3l)*



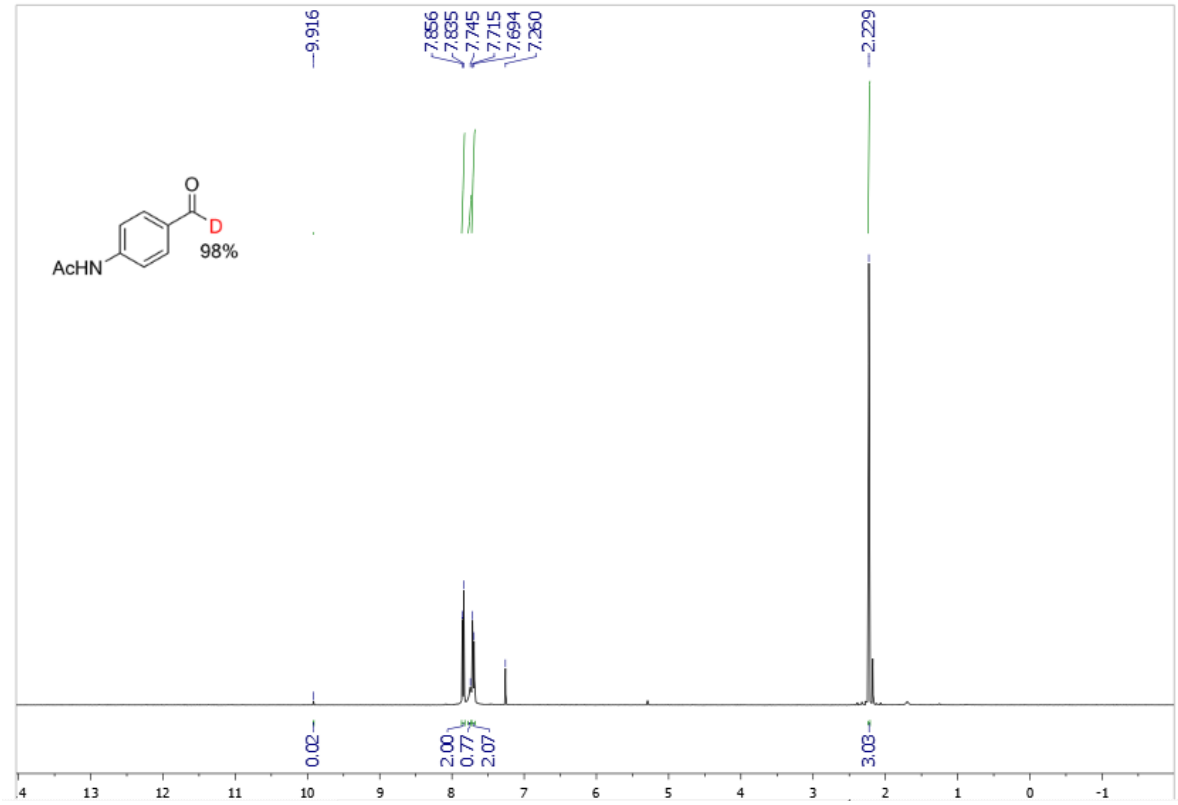


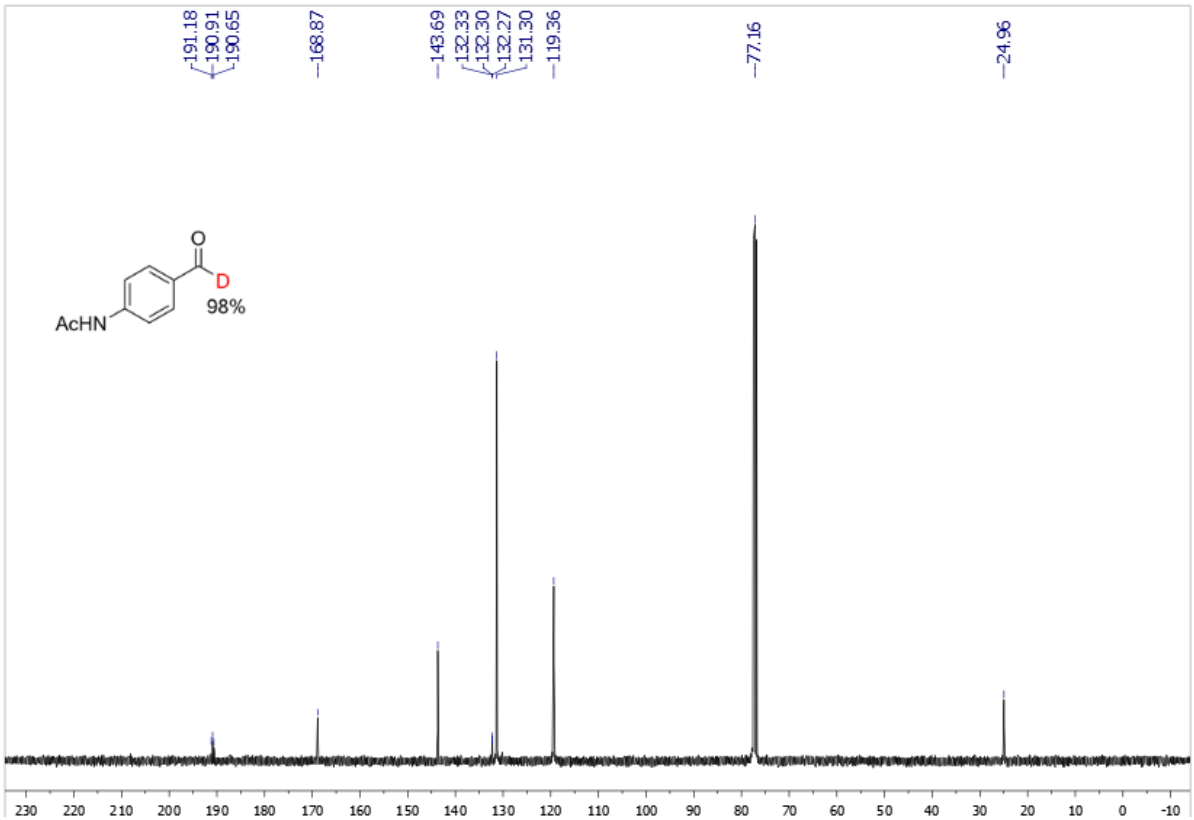
4-(Allyloxy)benzaldehyde-*formyl-d1 (3m)*



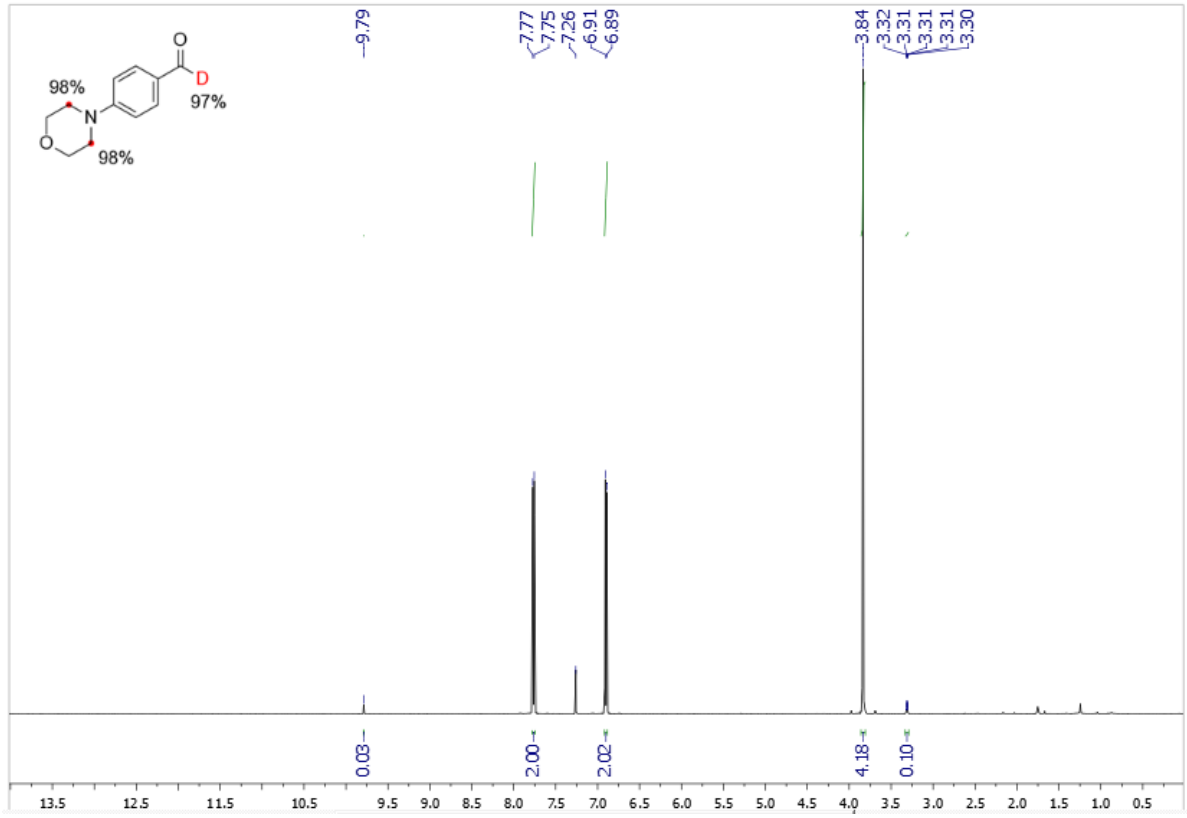


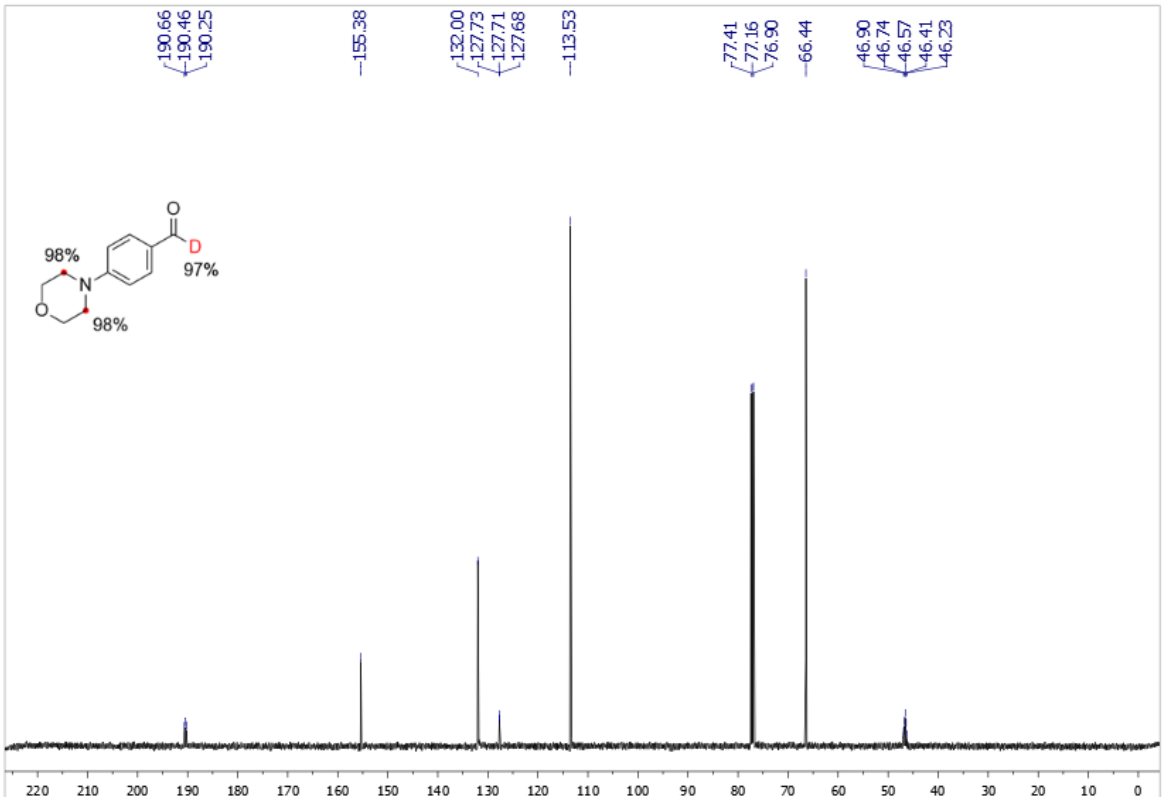
***N*-(4-Formylphenyl)acetamide-*formyl-d1* (3n)**



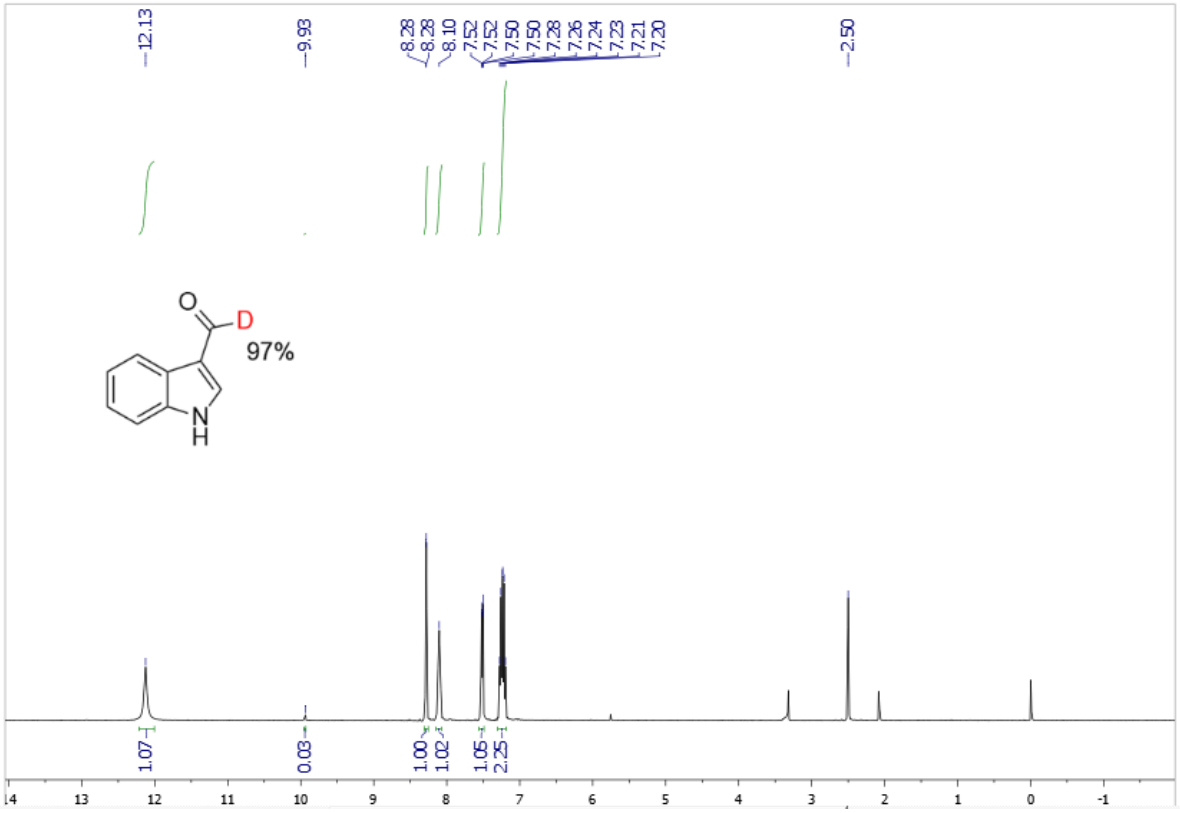


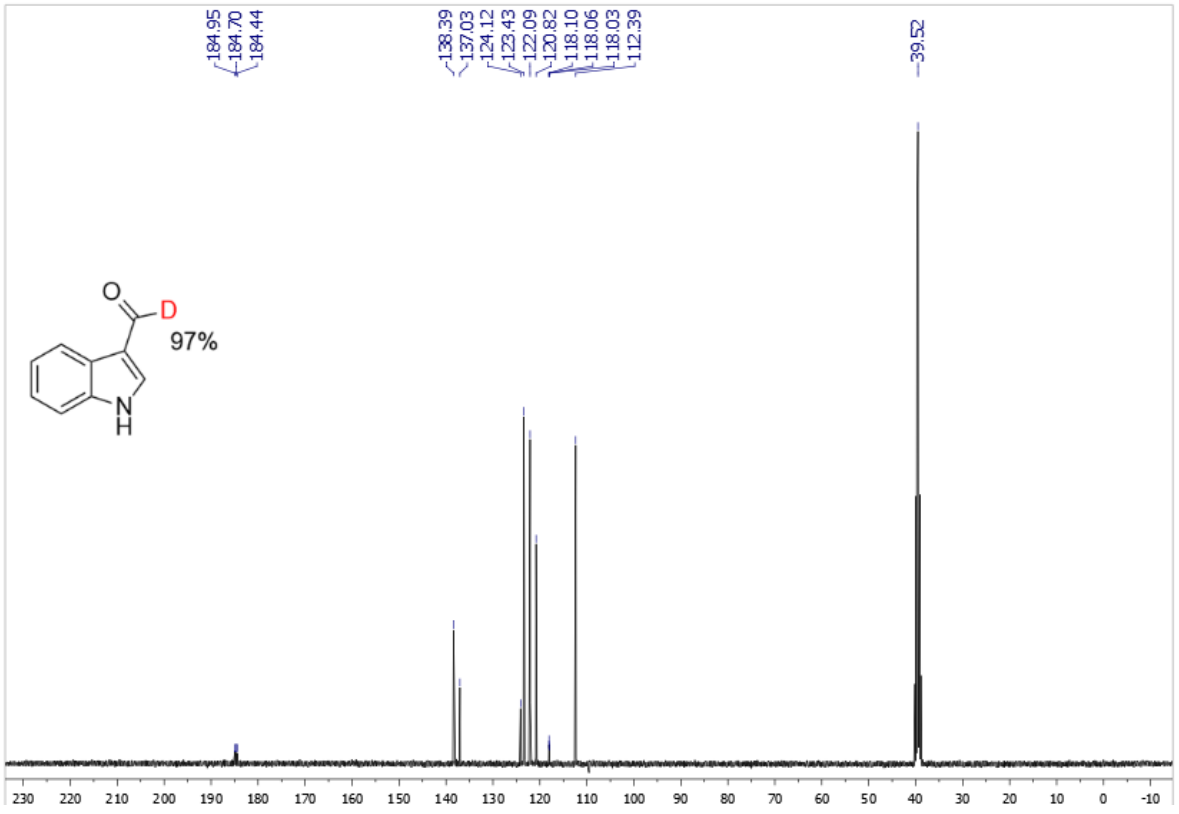
4-Morpholinobenzaldehyde-*formyl-d1 (3o)*



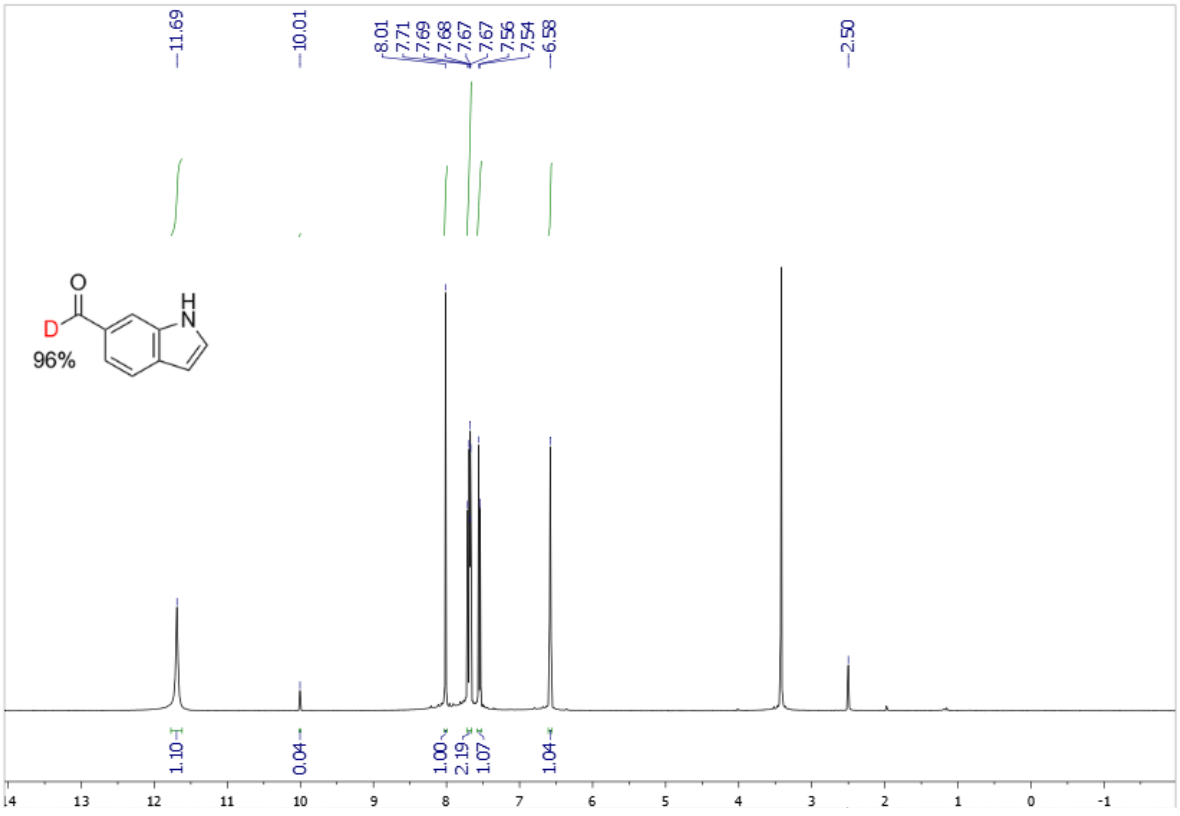


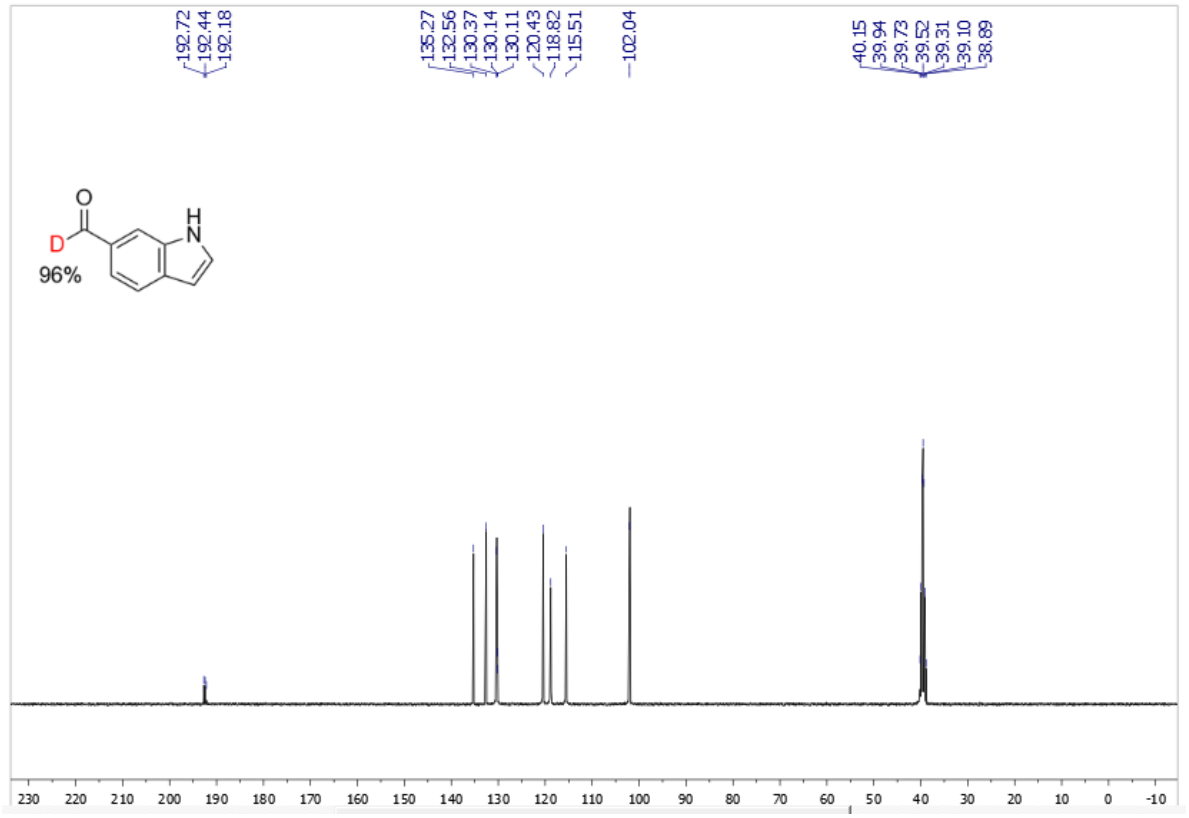
1*H*-indole-3-carbaldehyde-*formyl-d1 (3p)*



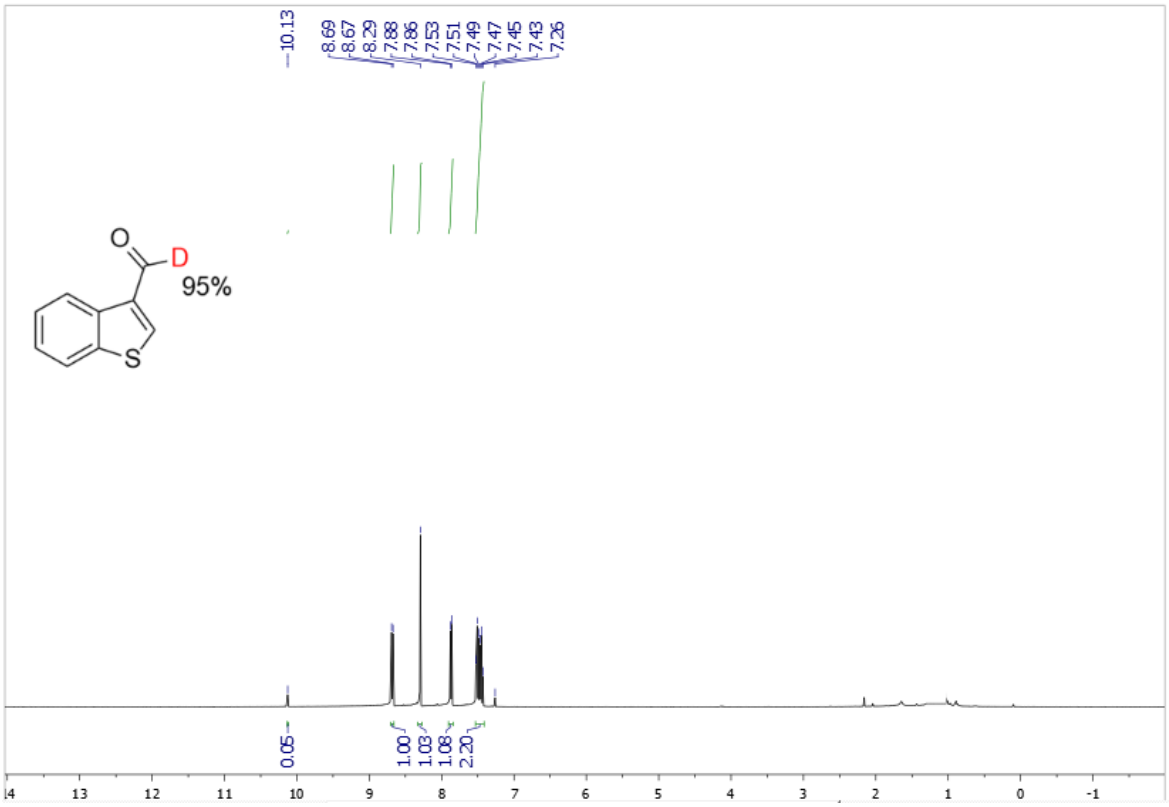


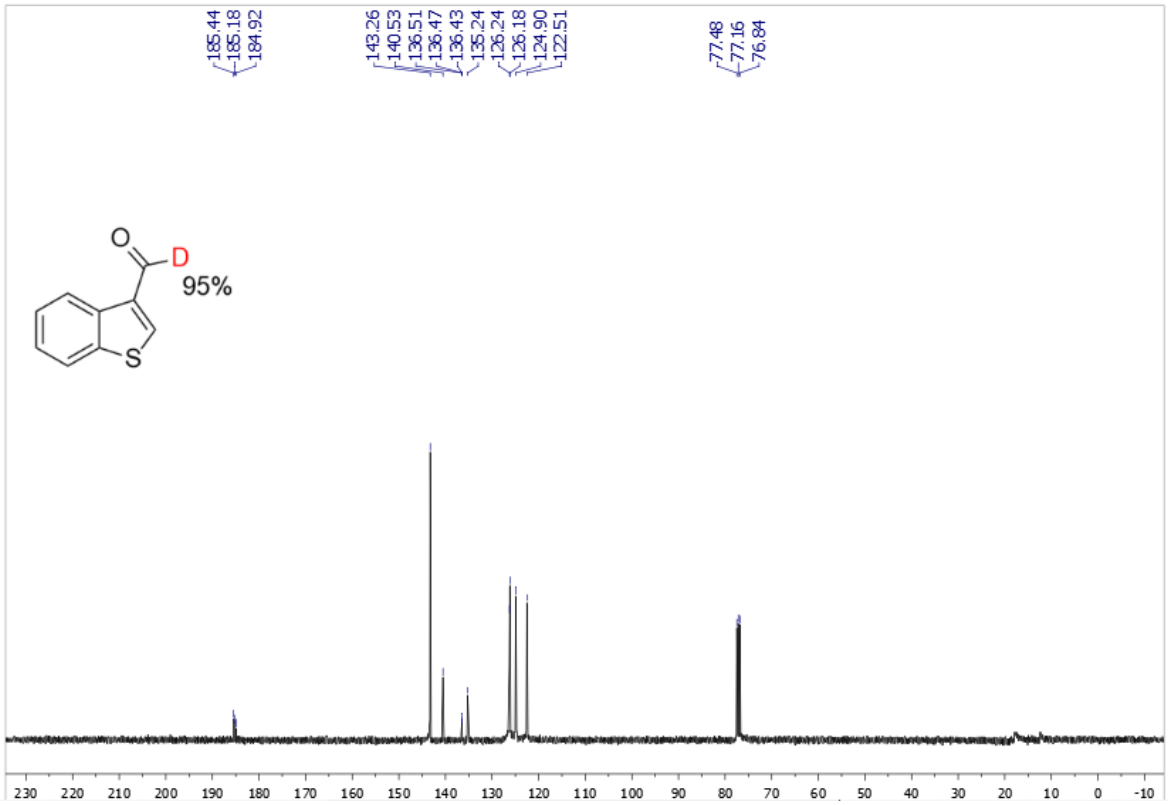
1*H*-indole-6-carbaldehyde-*formyl-d1 (3q)*



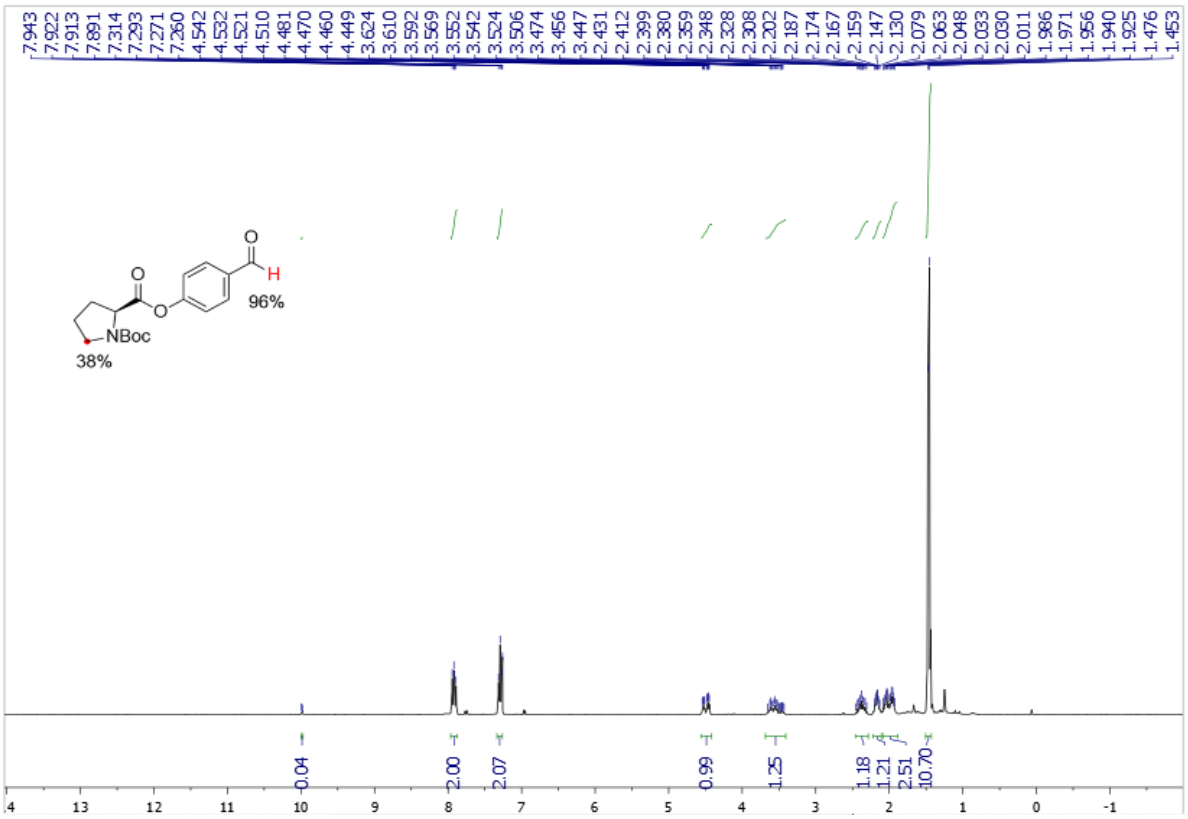


Benzo[b]thiophene-3-carbaldehyde-*formyl-d1 (3r)*





1-(*tert*-Butyl) 2-(4-formylphenyl) (*S*)-pyrrolidine-1,2-dicarboxylate-*formyl-d1 (3s)*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7.913
7.891
7.314
7.293
7.271
7.260
4.542
4.532
4.521
4.510
4.481
4.470
4.460
4.449
3.624
3.610
3.592
3.569
3.552
3.542
3.524
3.506
3.474
3.456
3.447
2.431
2.412
2.399
2.380
2.359
2.348
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2.308
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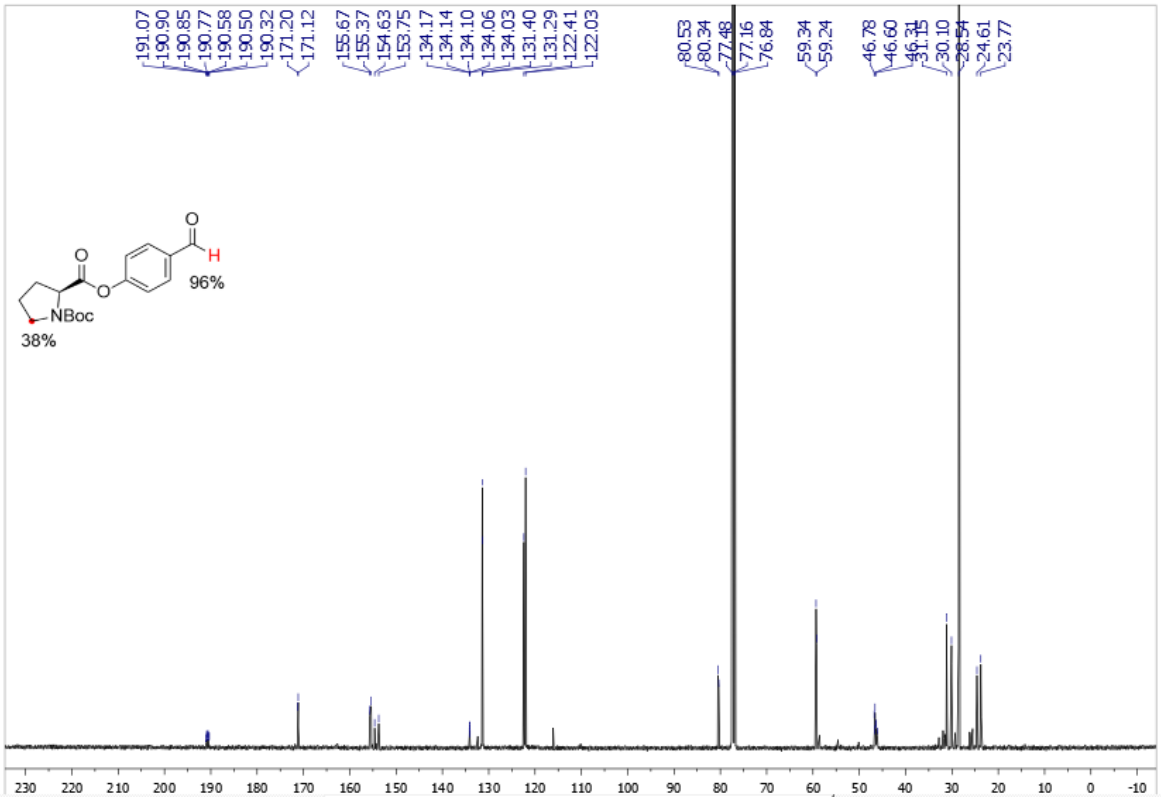
2.187
2.174
2.167
2.159
2.147
2.130
2.079
2.063
2.048
2.033
2.030
2.011
1.986
1.971
1.956
1.940
1.925
1.476
1.453
O
O
O
H
96%
NBoc
38%
0.04
2.00
2.07
0.99
1.25
1.18
1.21
2.51
10.70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171.12
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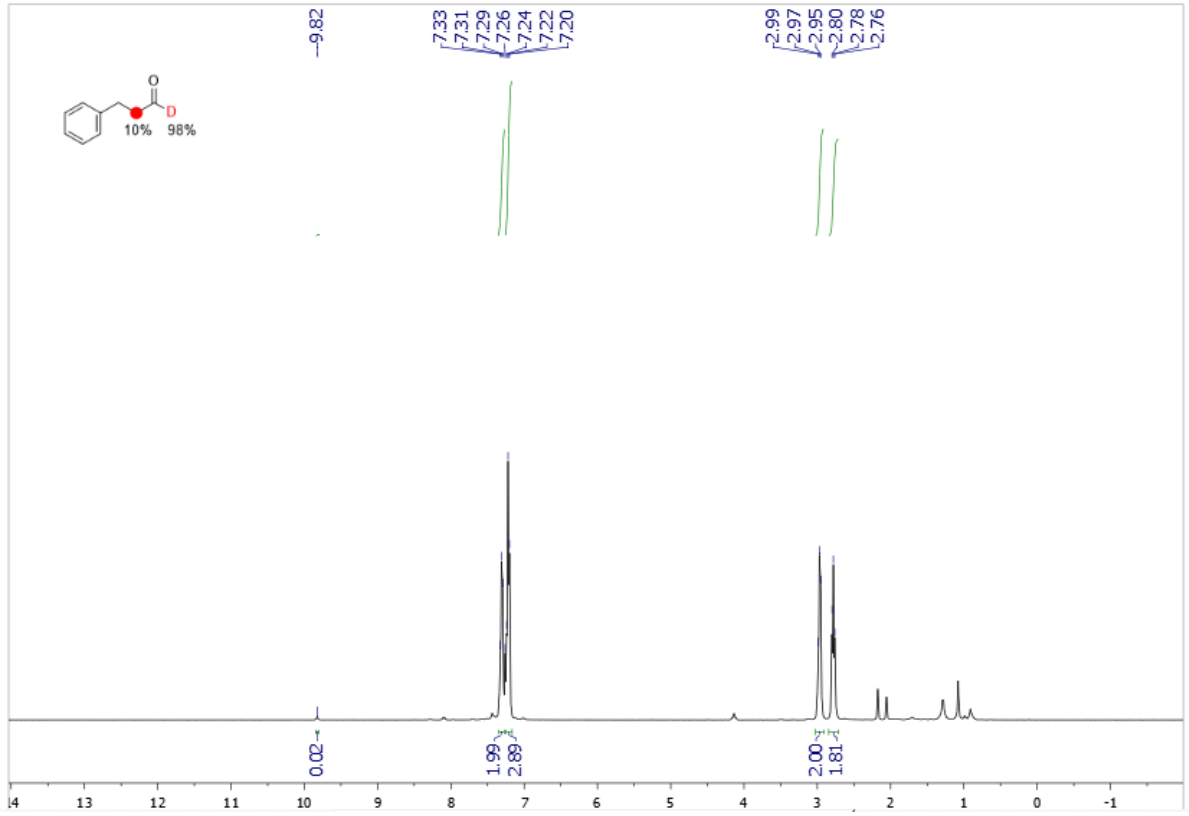
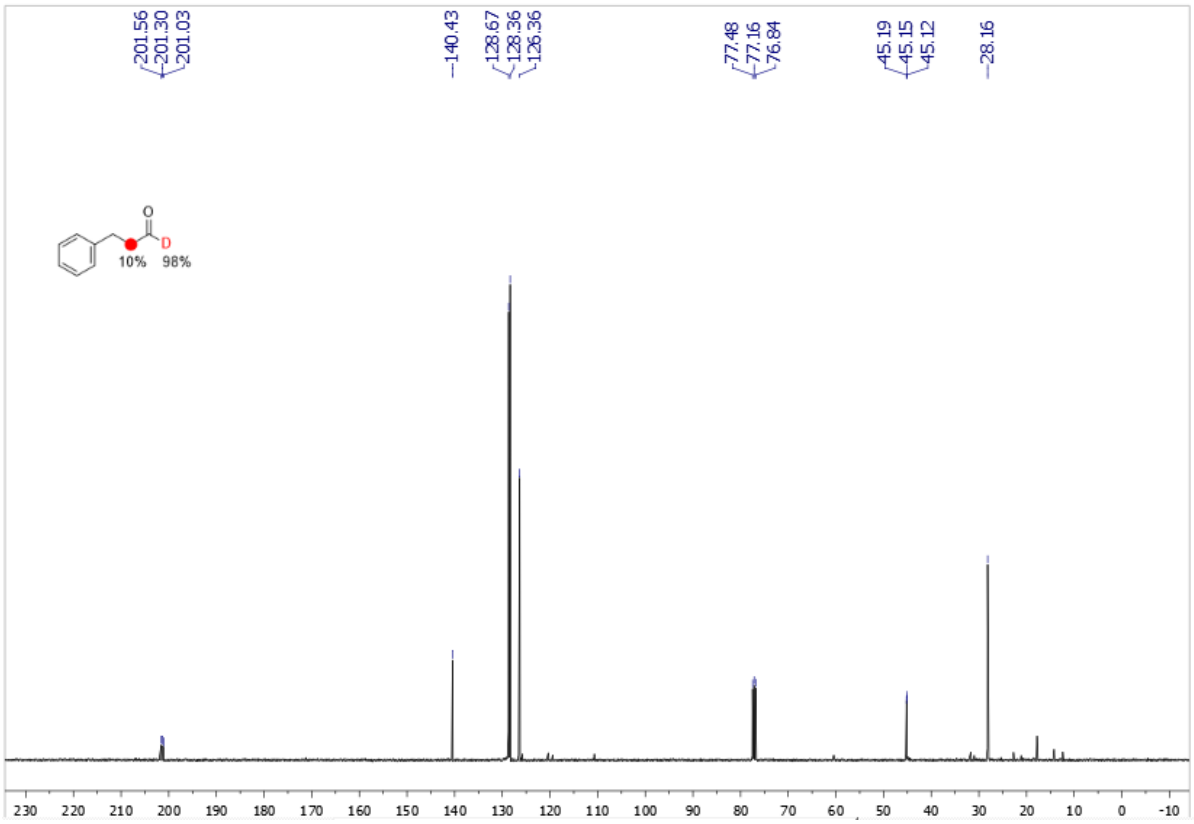
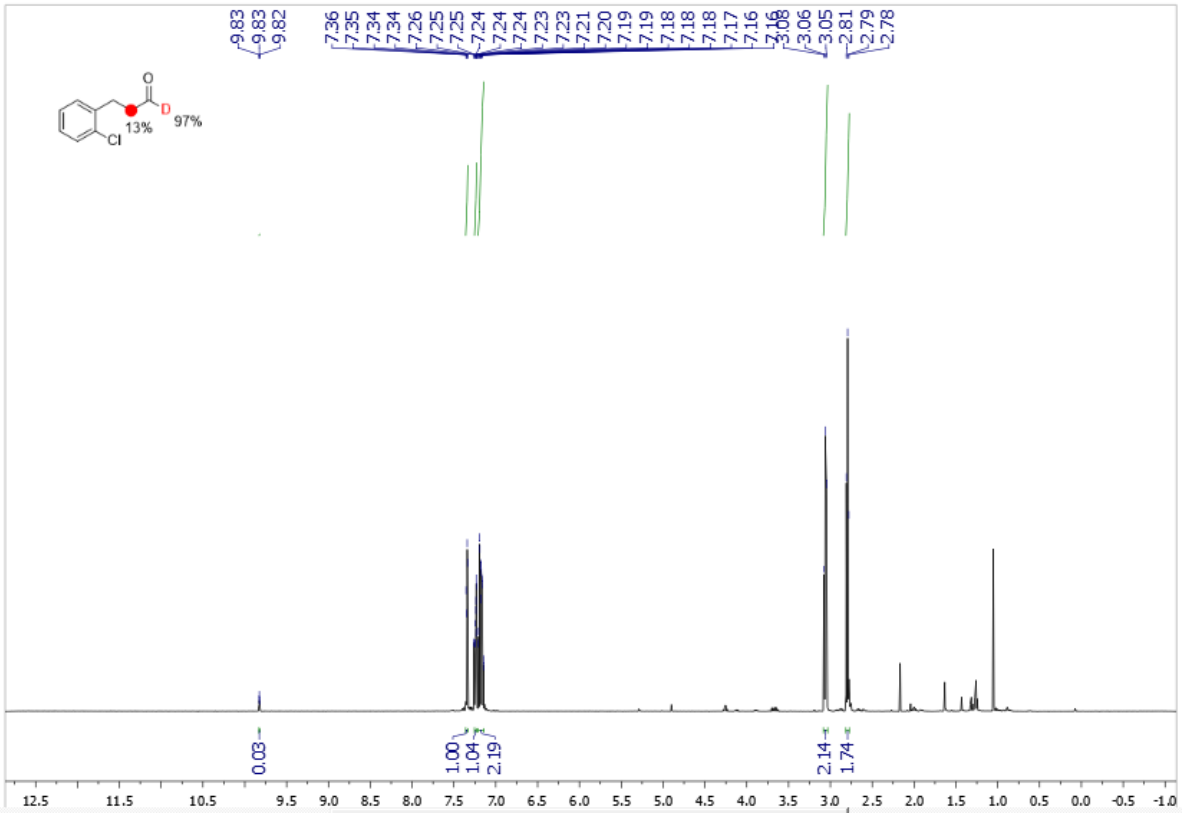
155.37
154.63
153.75
134.17
134.14
134.10
134.06
134.03
131.40
131.29
122.41
122.03
80.53
80.34
77.48
77.16
76.84
59.34
59.24
46.78
46.60
46.31
31.15
30.10
28.54
24.61
23.77
O
O
O
H
96%
NBoc
38%

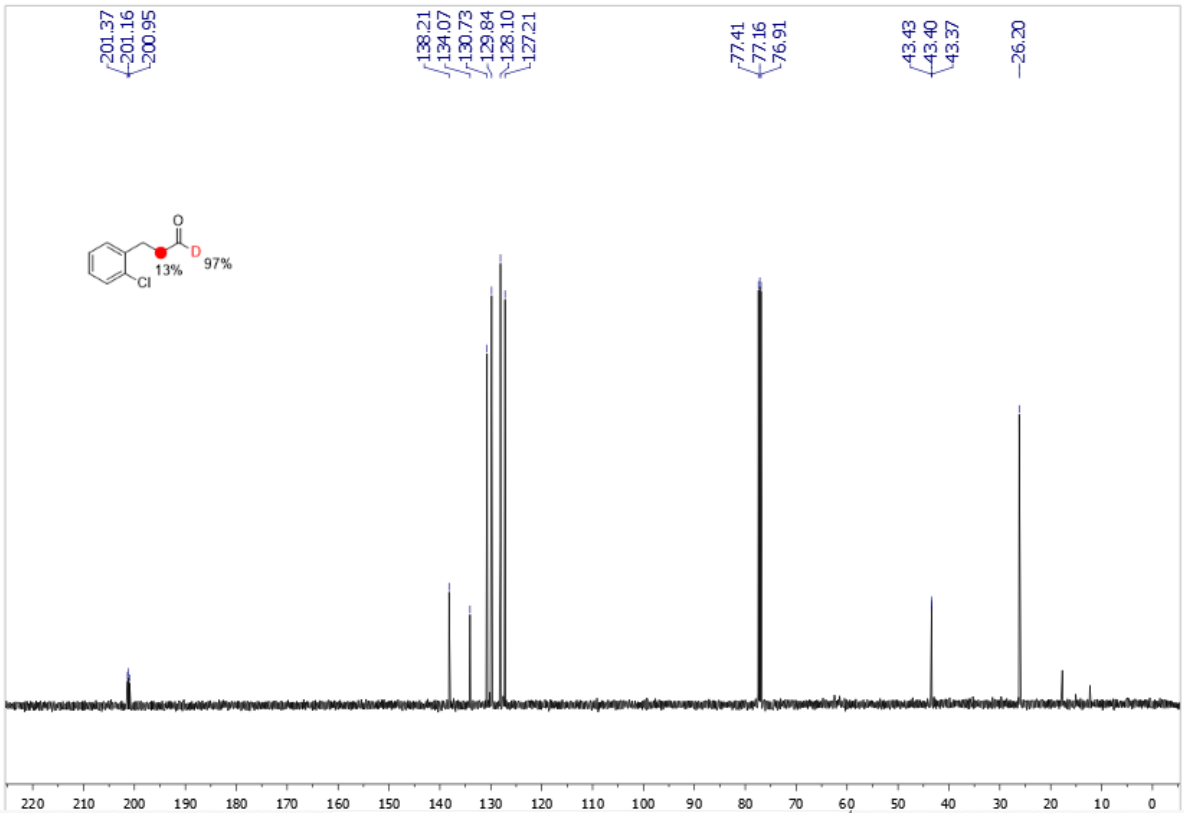
3-Phenylpropanal-*formyl-d1 (3t)*



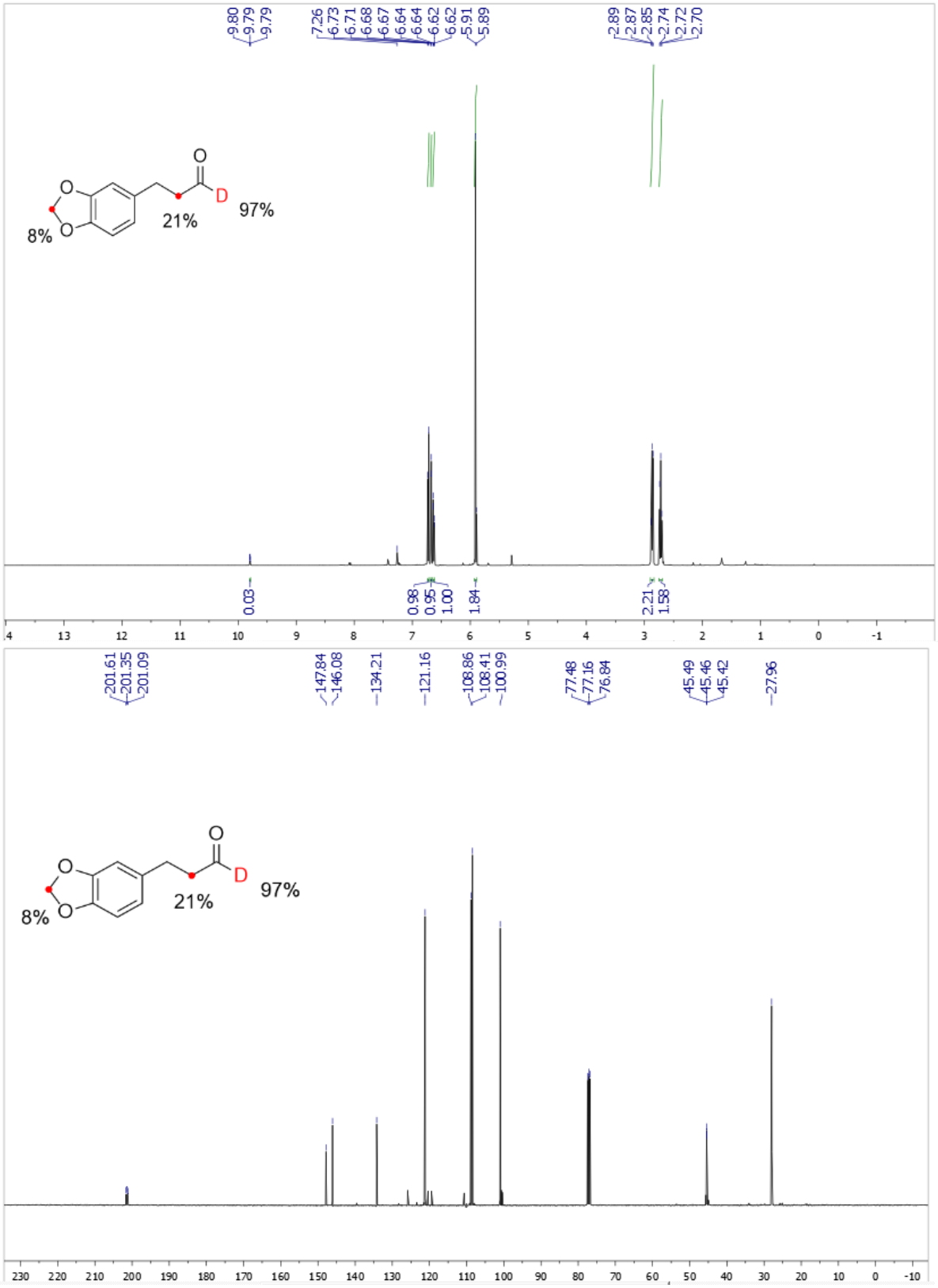


3-(2-Chlorophenyl)propanal-*formyl-d1 (3u)*

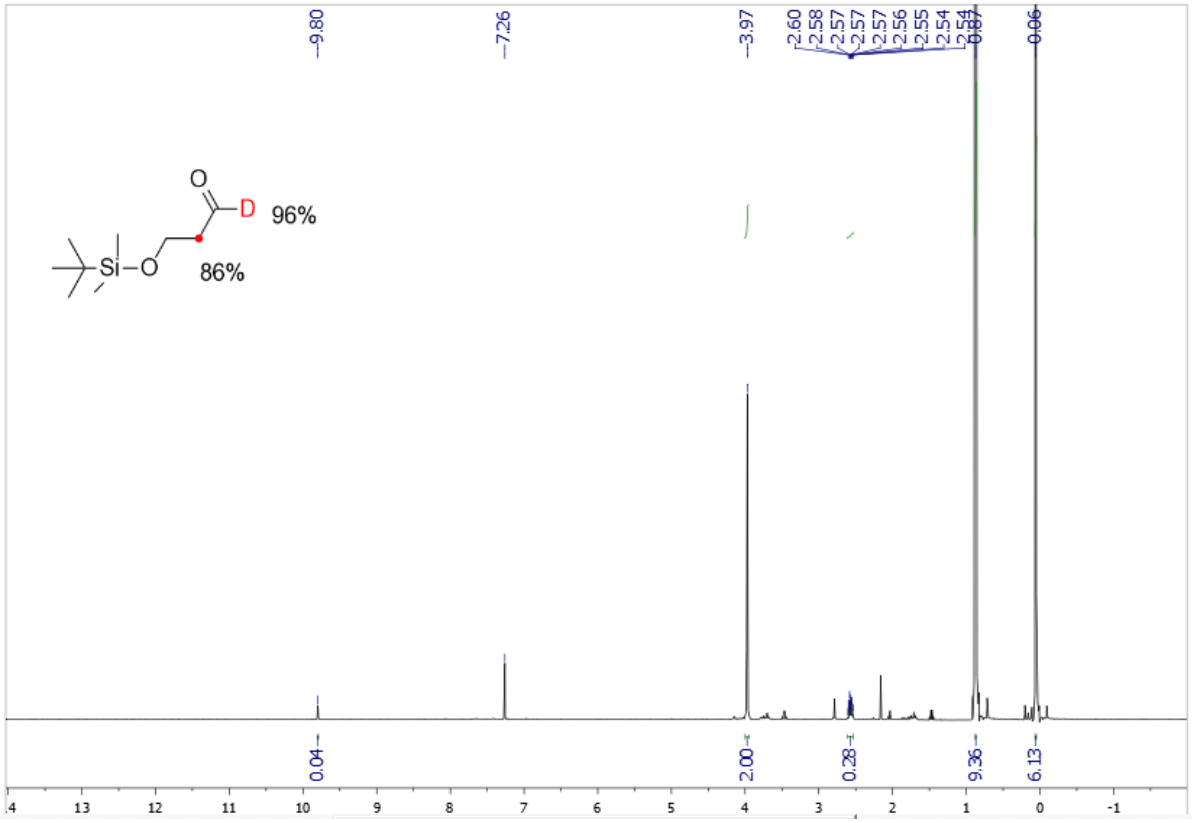


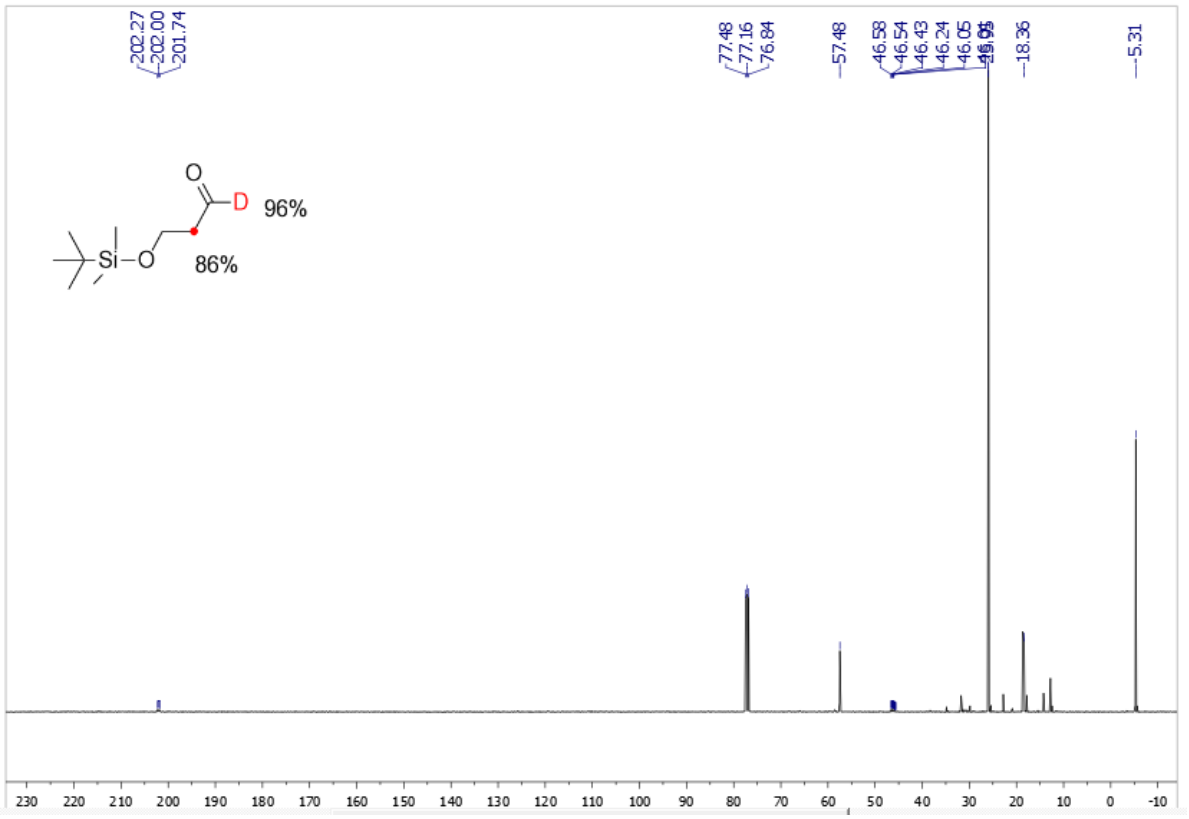


3-(Benzo[*d*][1,3]dioxol-5-yl)propanal-*formyl-d1 (3v)*

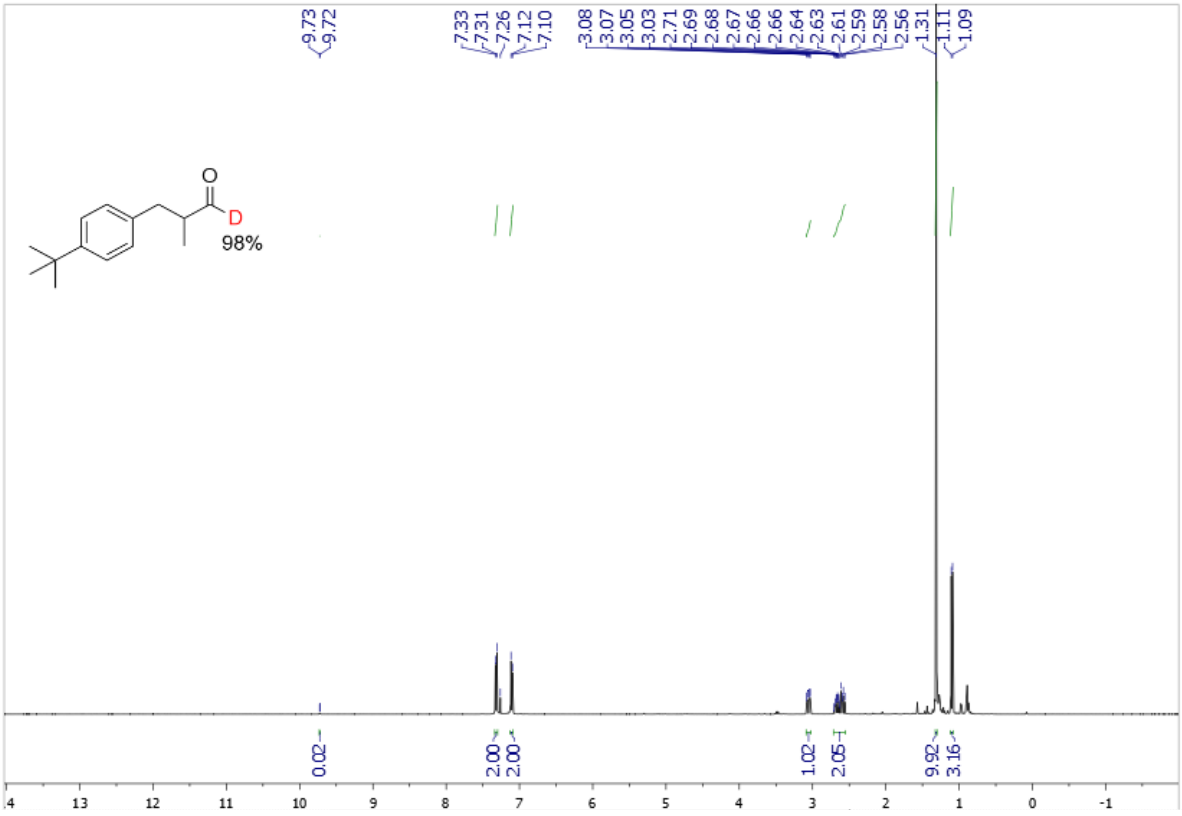


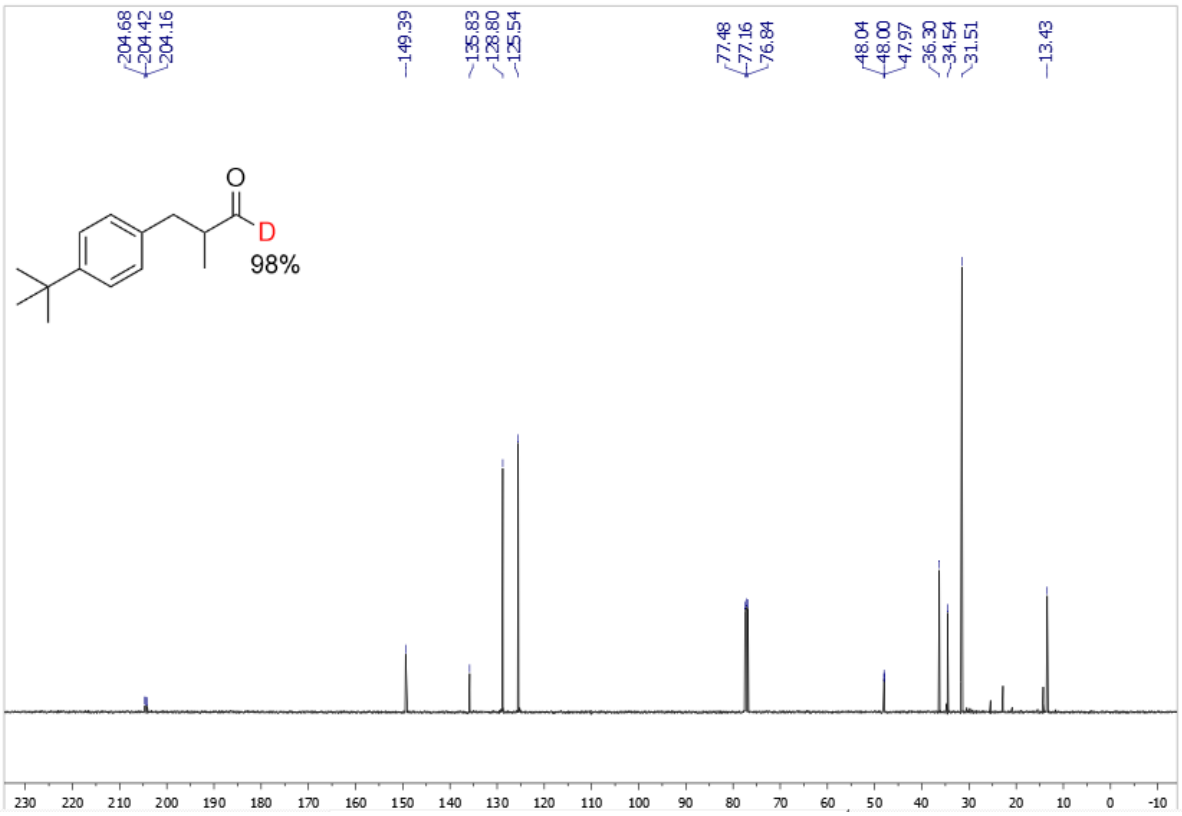
3-((*tert*-Butyldimethylsilyl)oxy)propanal-*formyl-d1 (3w)*



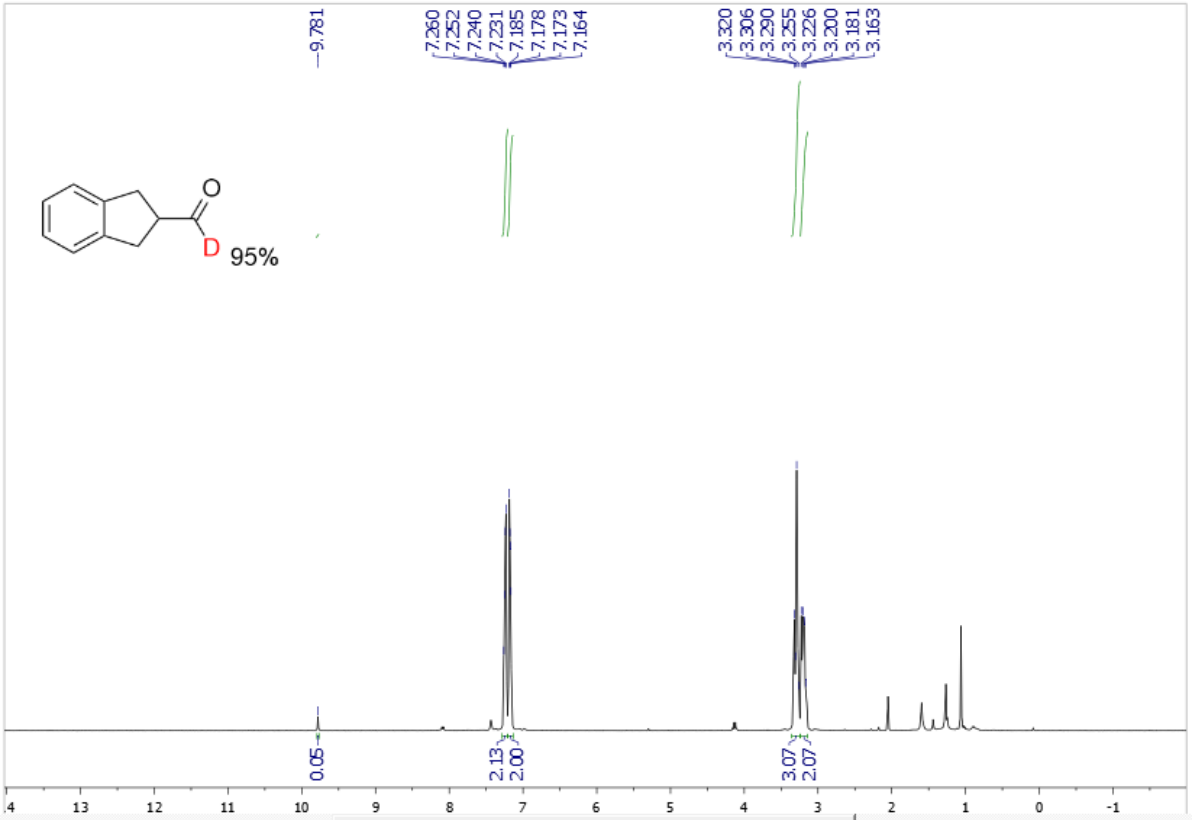


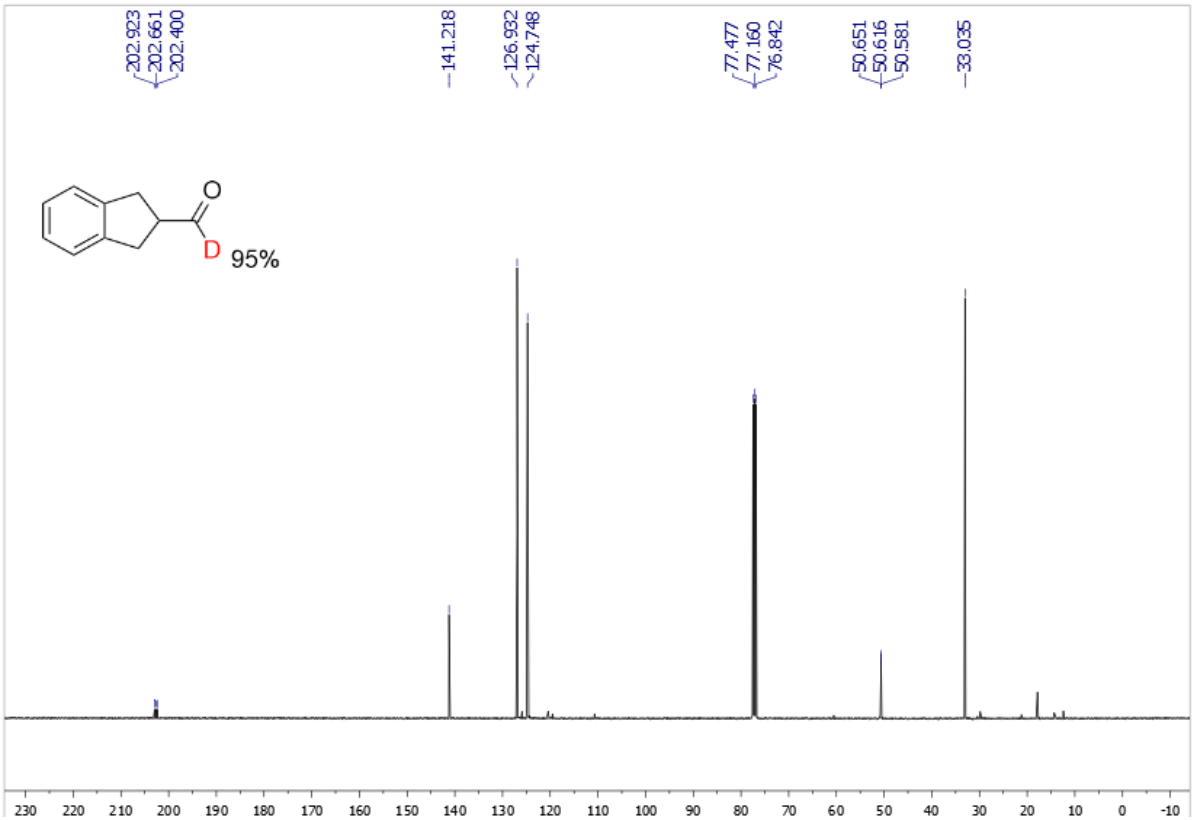
3-(4-(*tert*-Butyl)phenyl)-2-methylpropanal-*formyl-d1 (3x)*



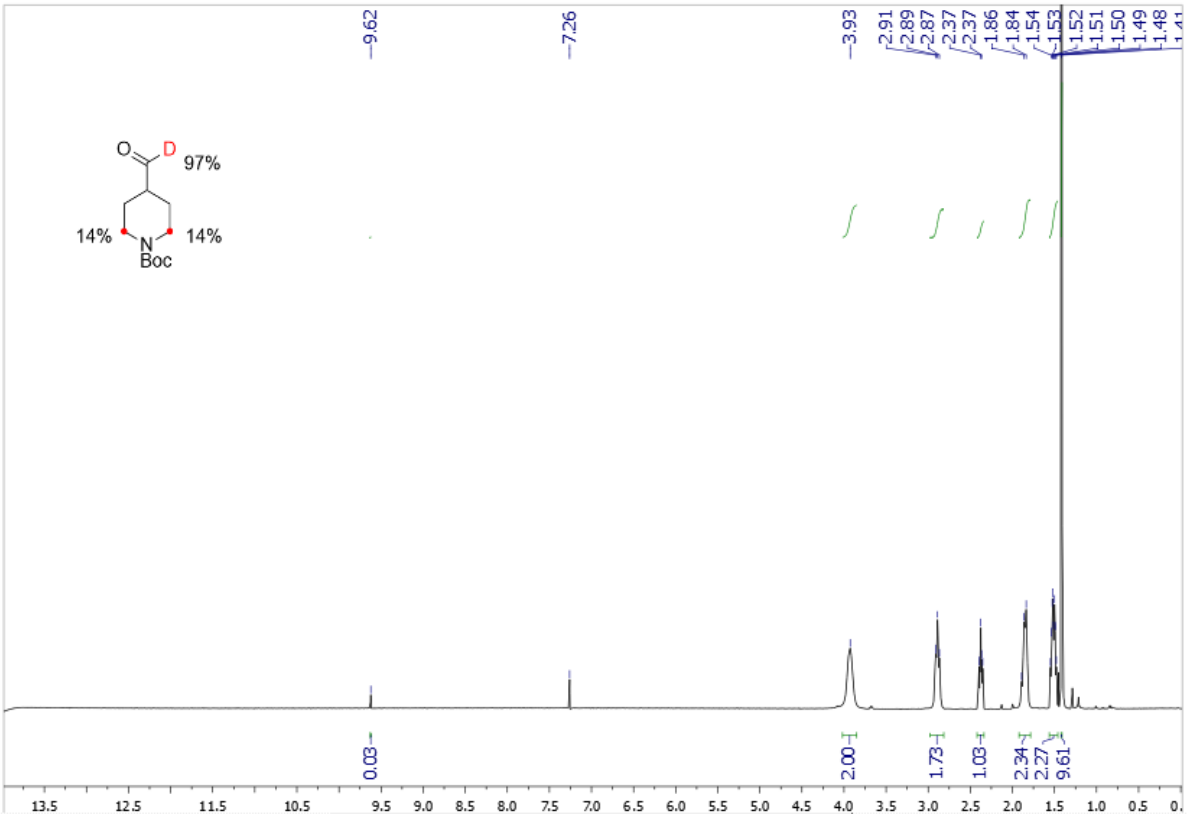


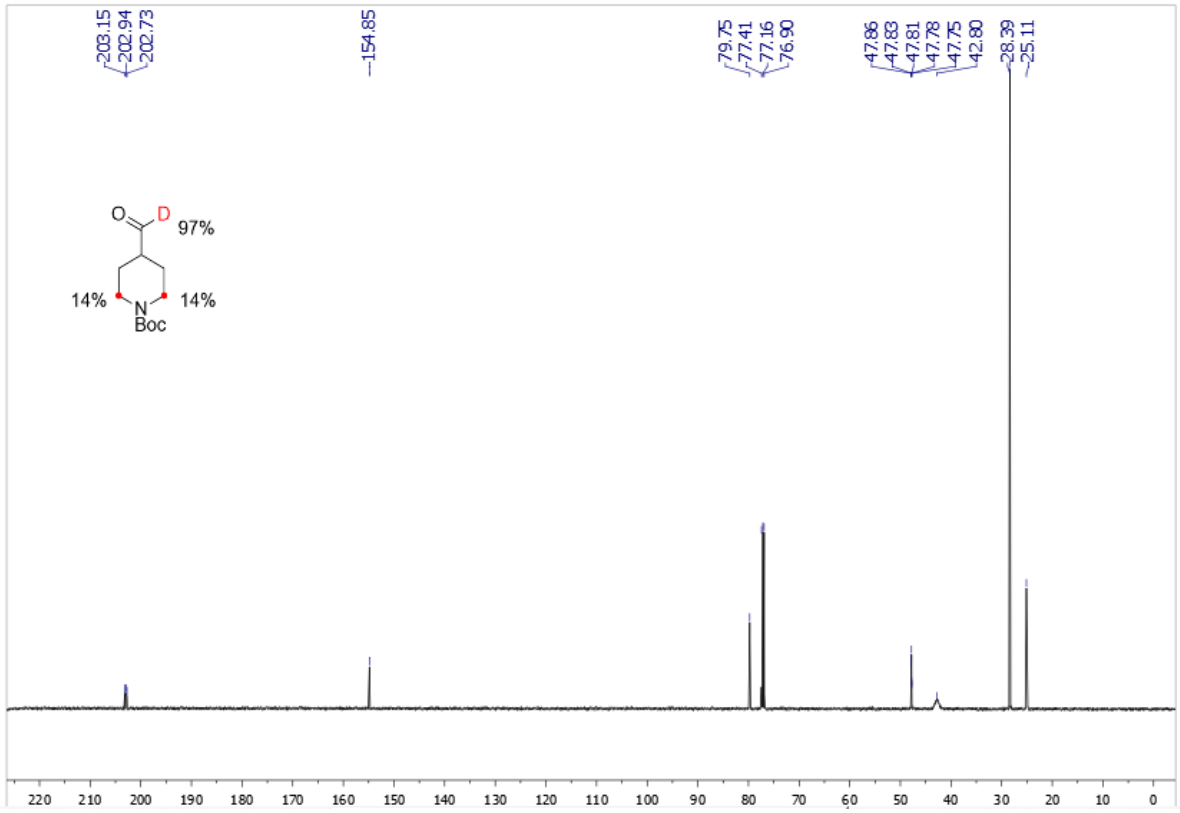
2,3-Dihydro-1*H*-indene-2-carbaldehyde-*formyl-d1* (*3y*)



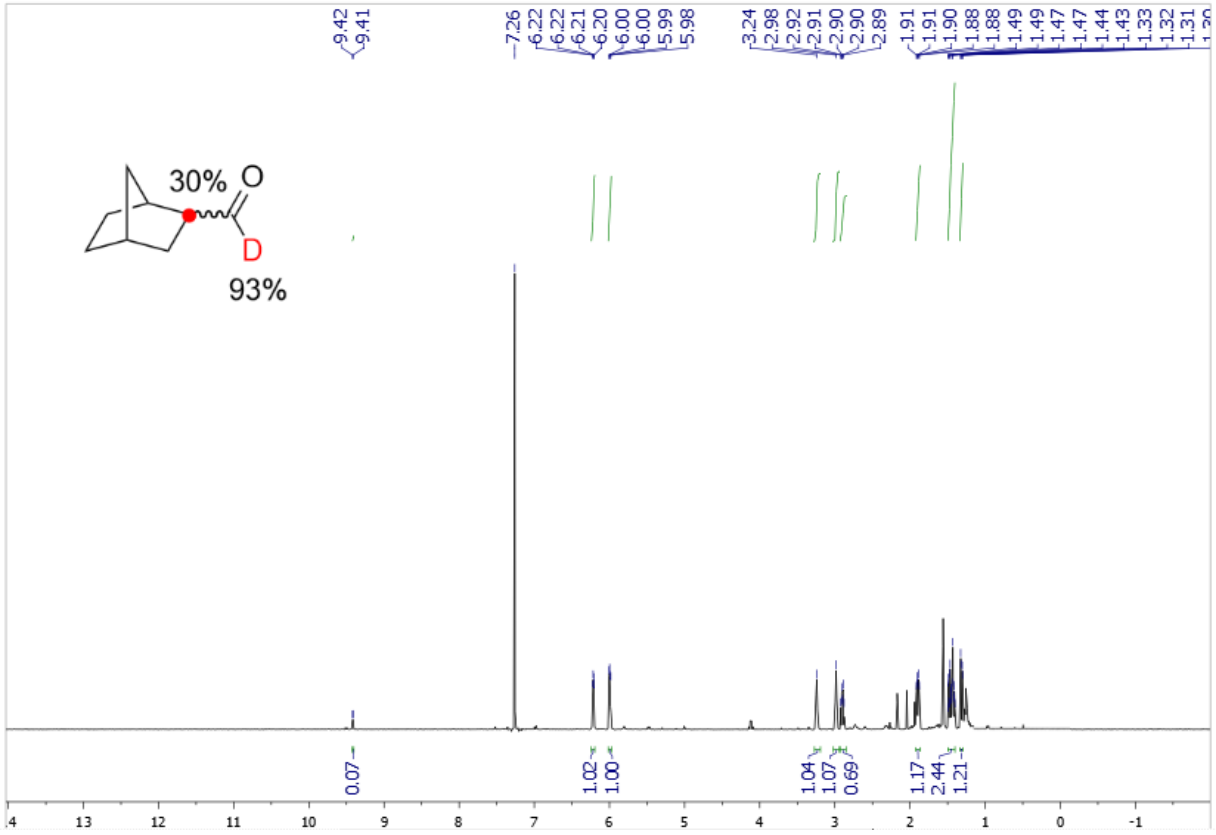


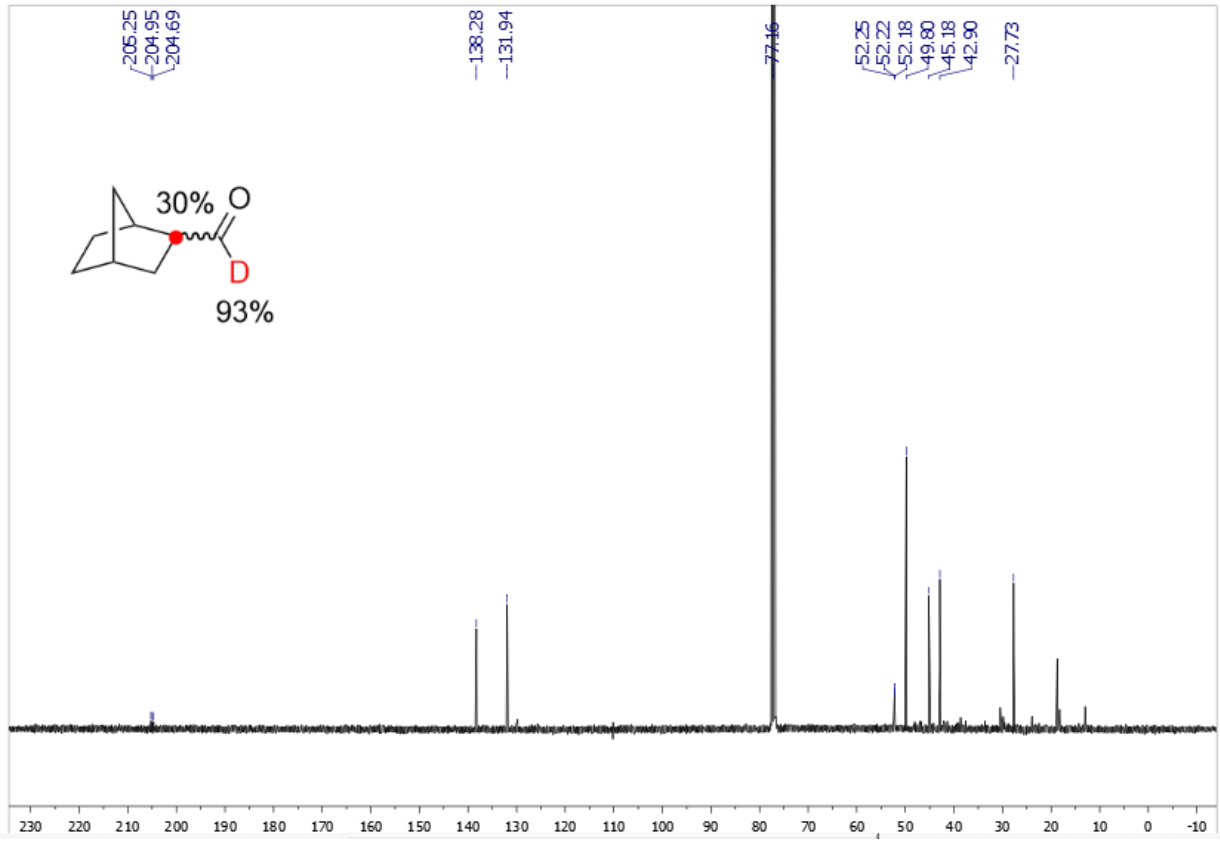
***tert*-Butyl 4-formylpiperidine-1-carboxylate-*formyl-d1* (3z)**



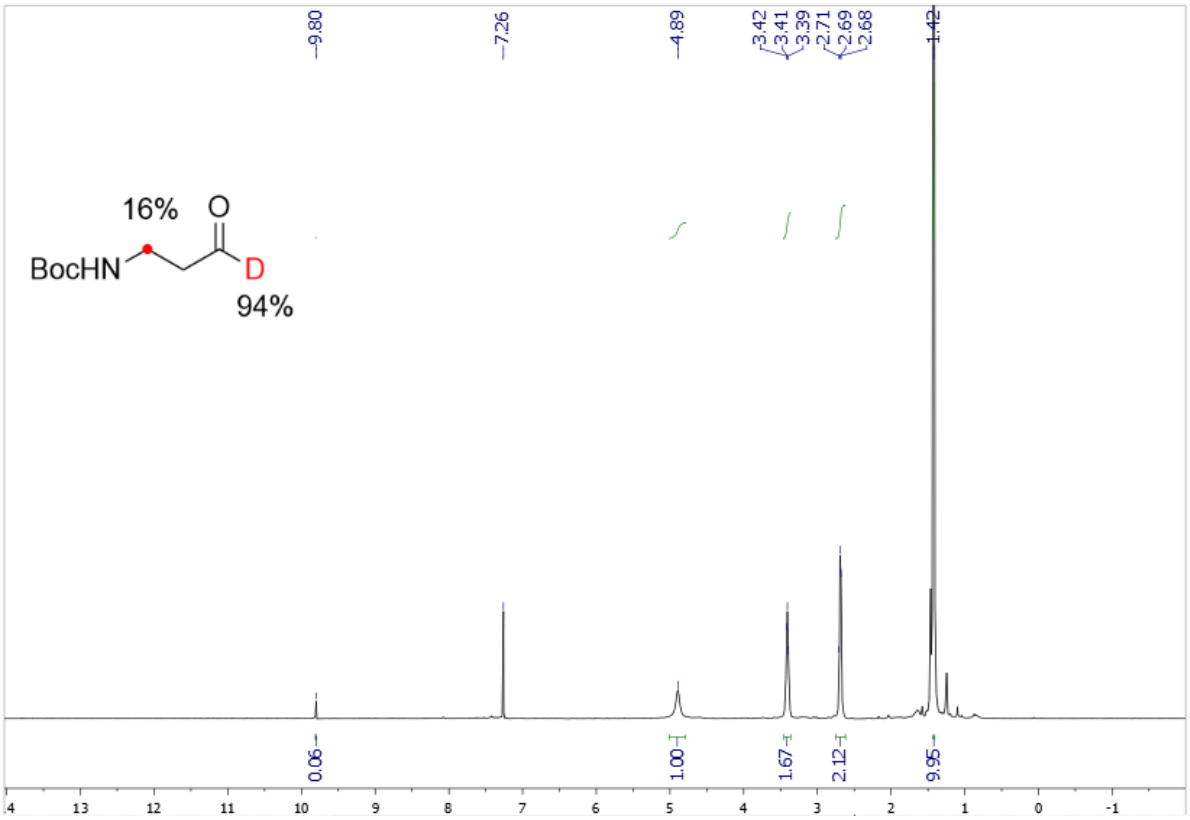


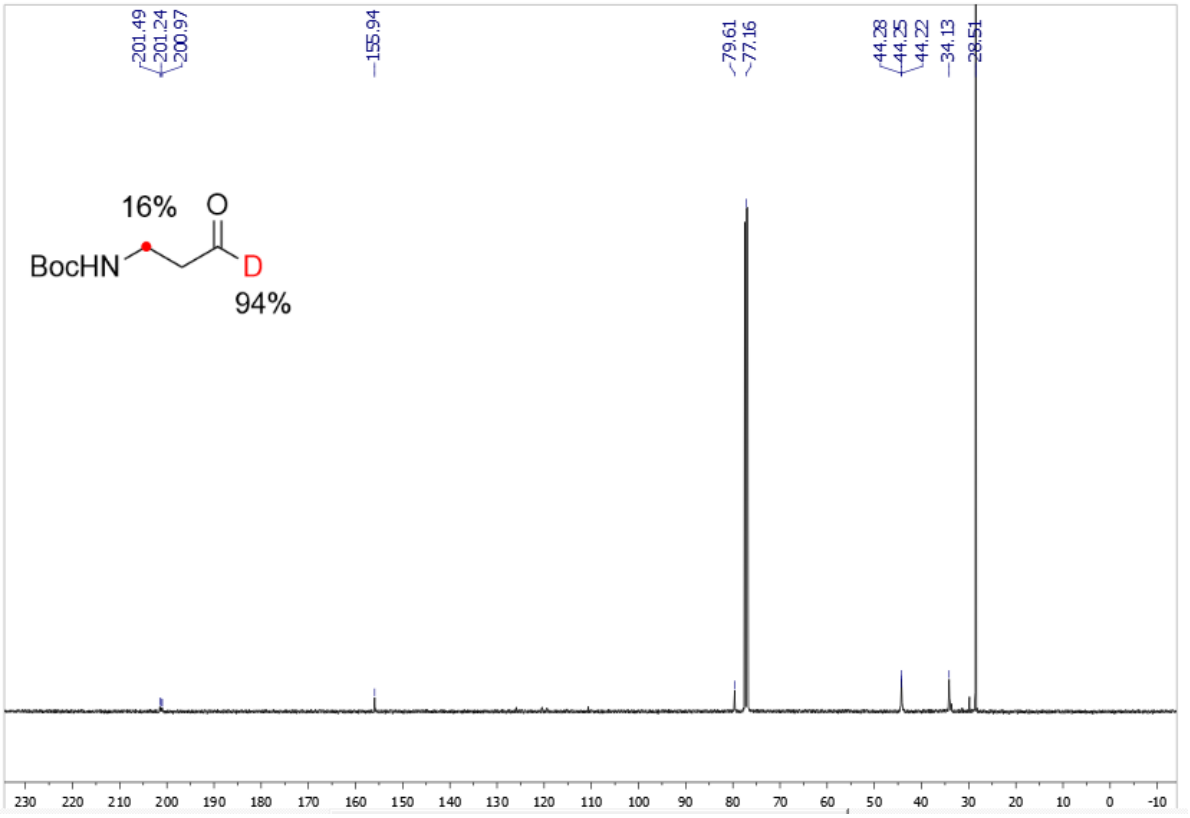
Bicyclo[2.2.1]hept-5-ene-2-carboxaldehyde-*formyl-d1 (3aa)*



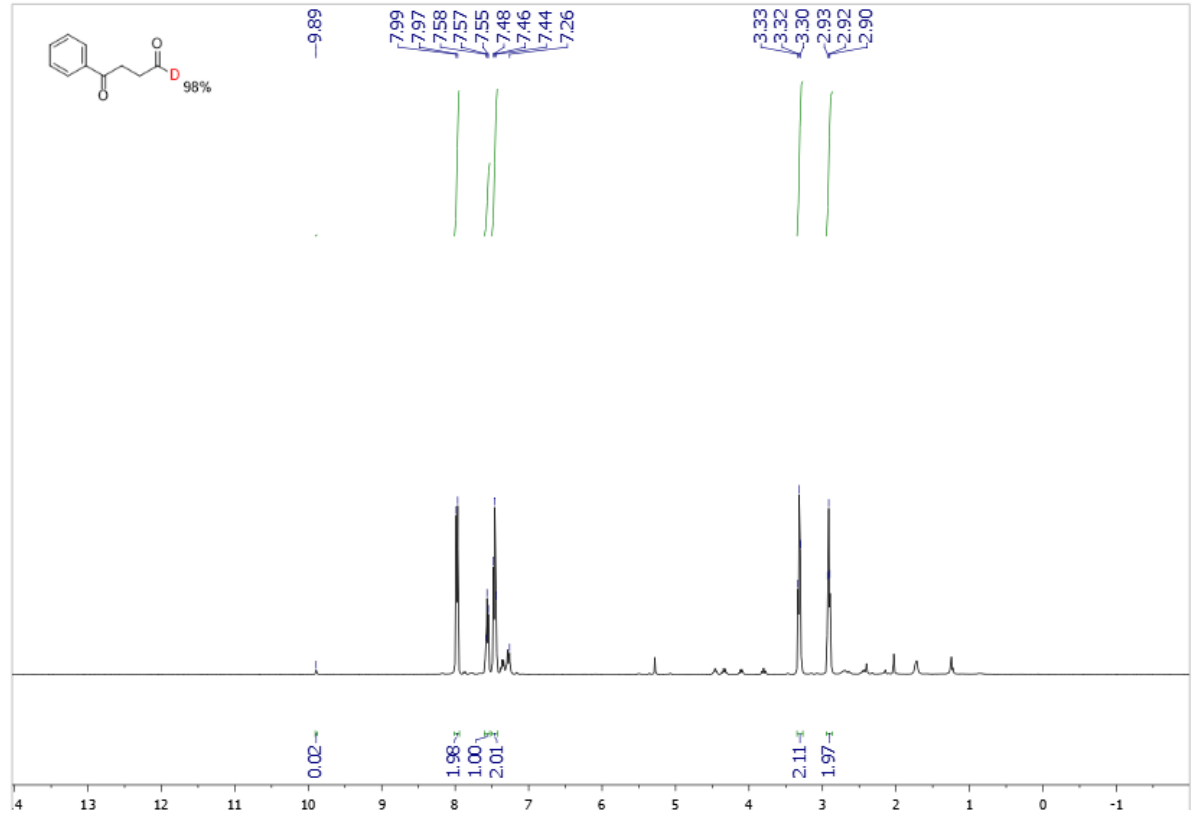


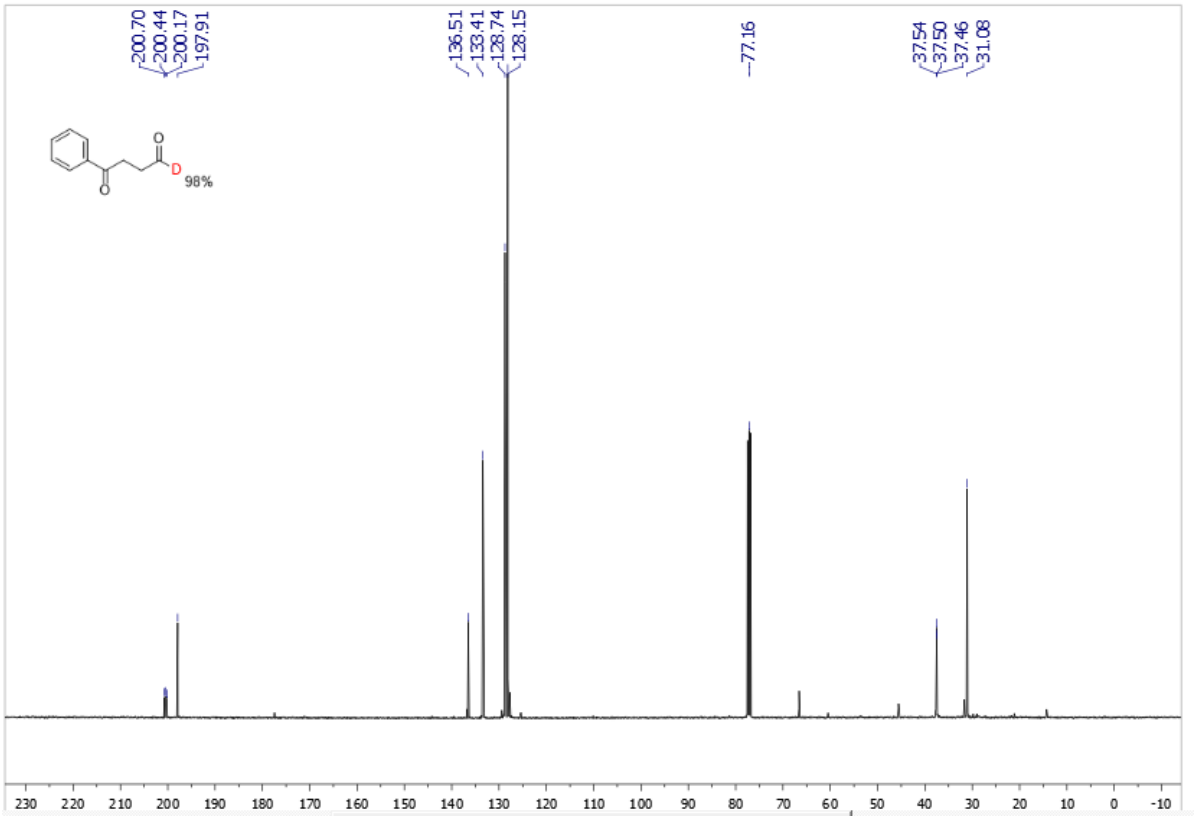
***tert*-Butyl (3-oxopropyl)carbamate-*formyl-d1* (3ab)**



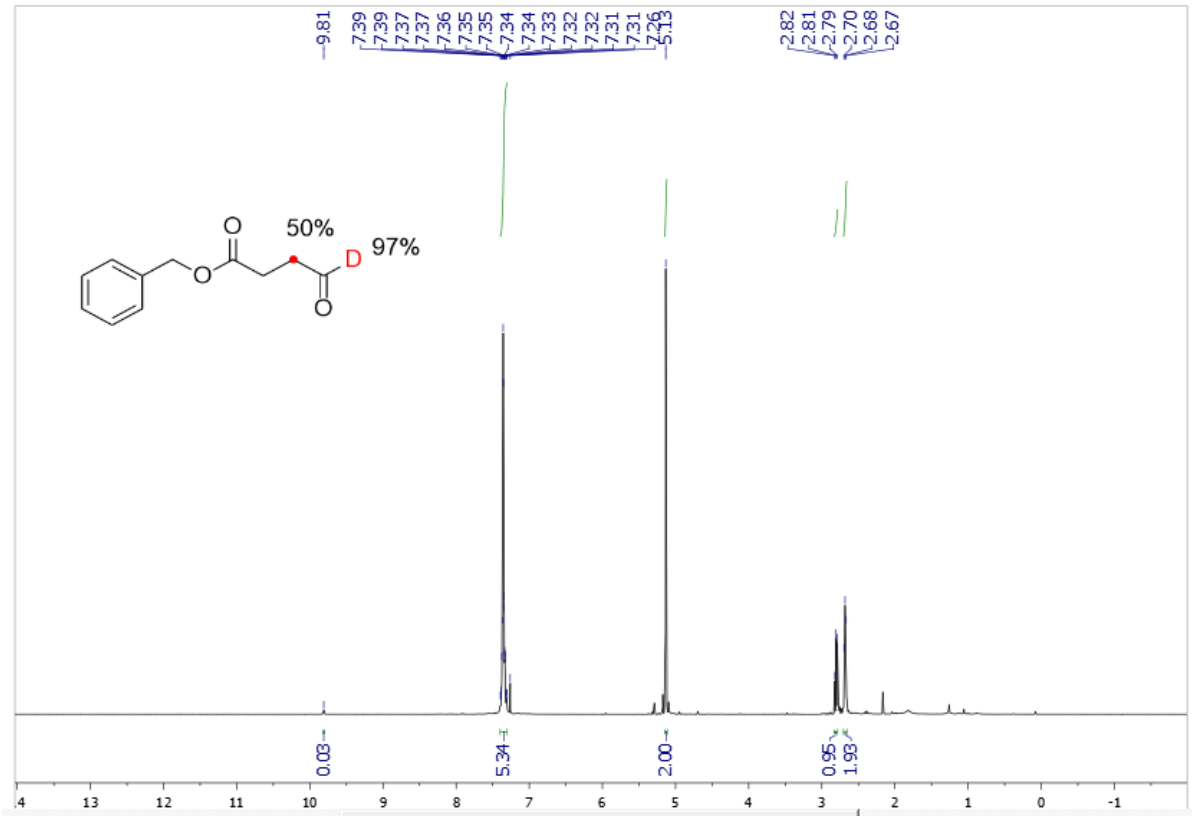


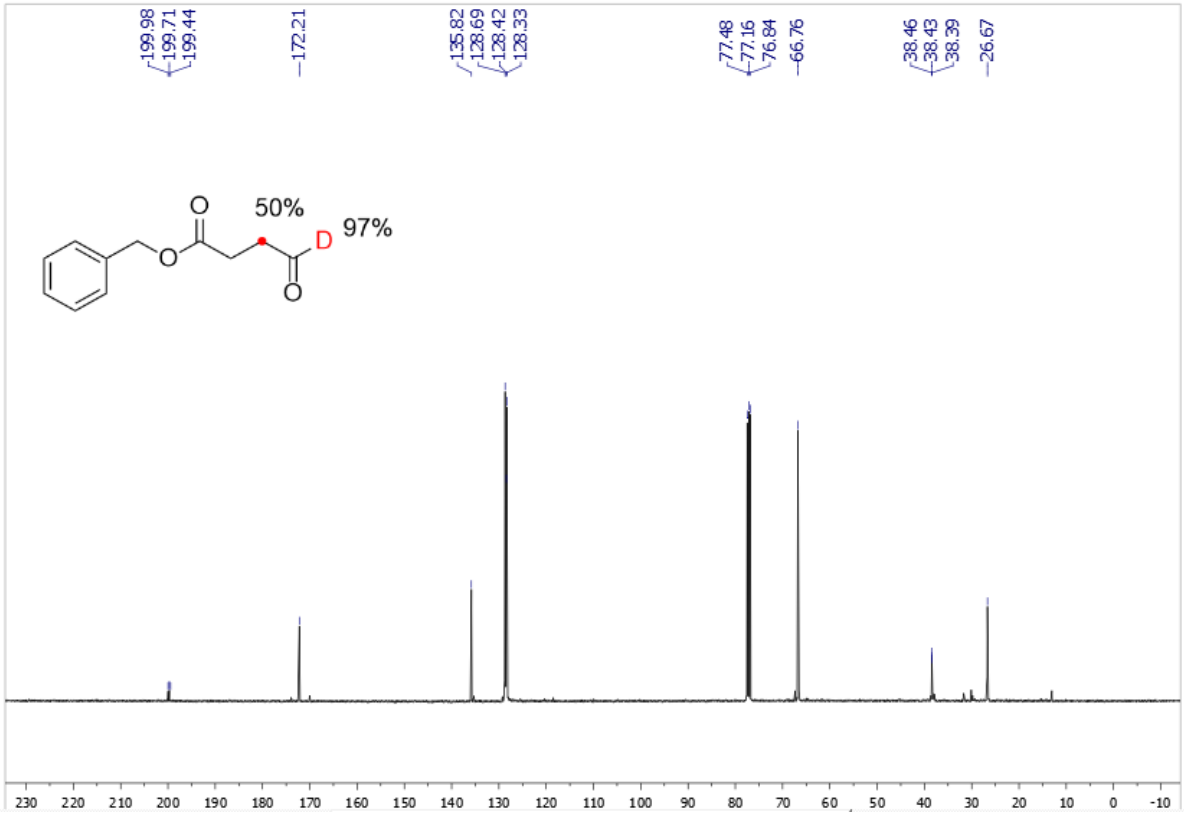
4-Oxo-4-phenylbutanal-*formyl-d1 (3ac)*



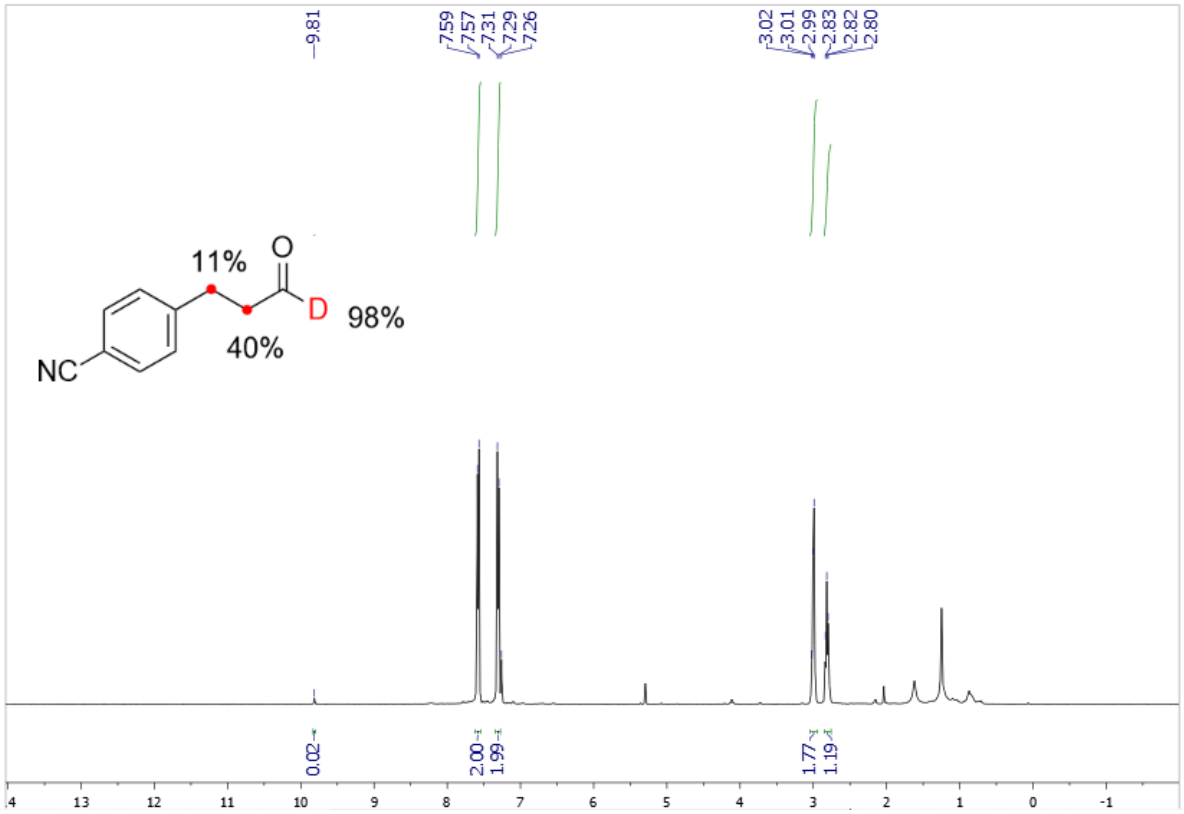


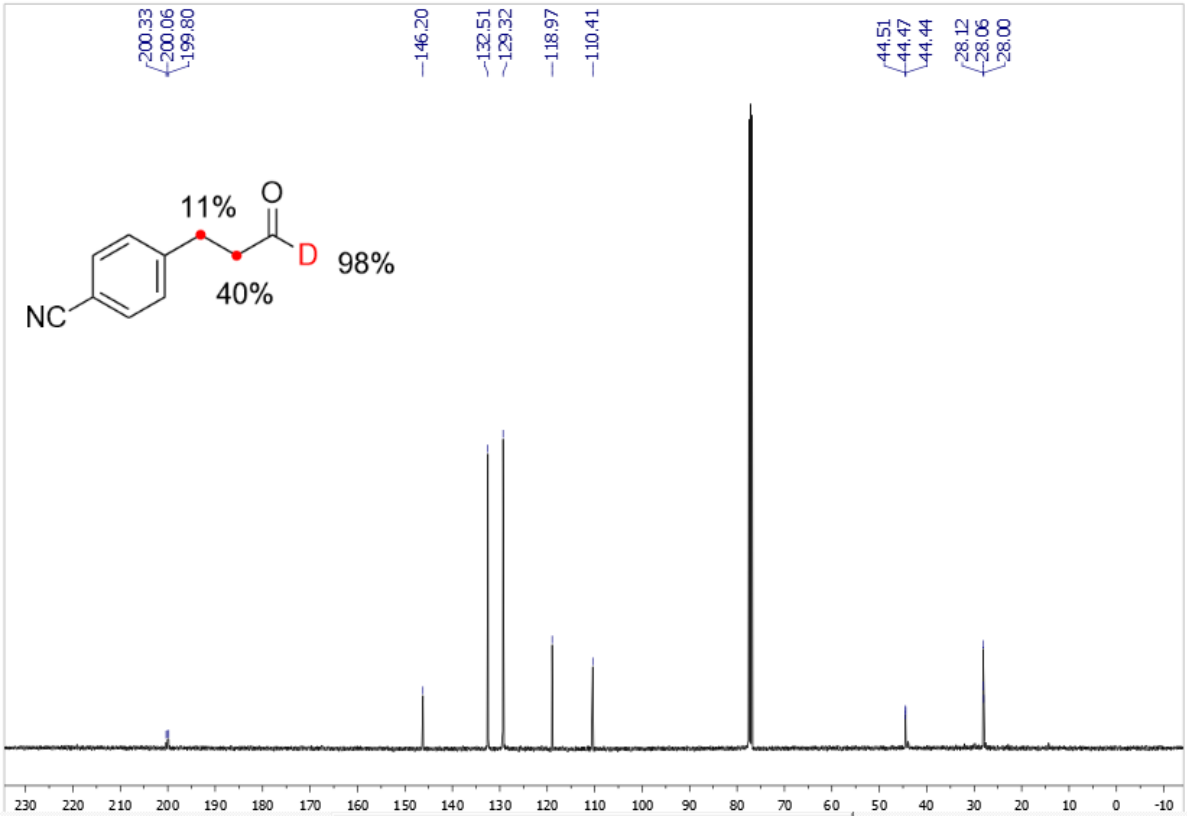
Benzyl 4-oxobutanoate-*formyl-d1* (3ad)



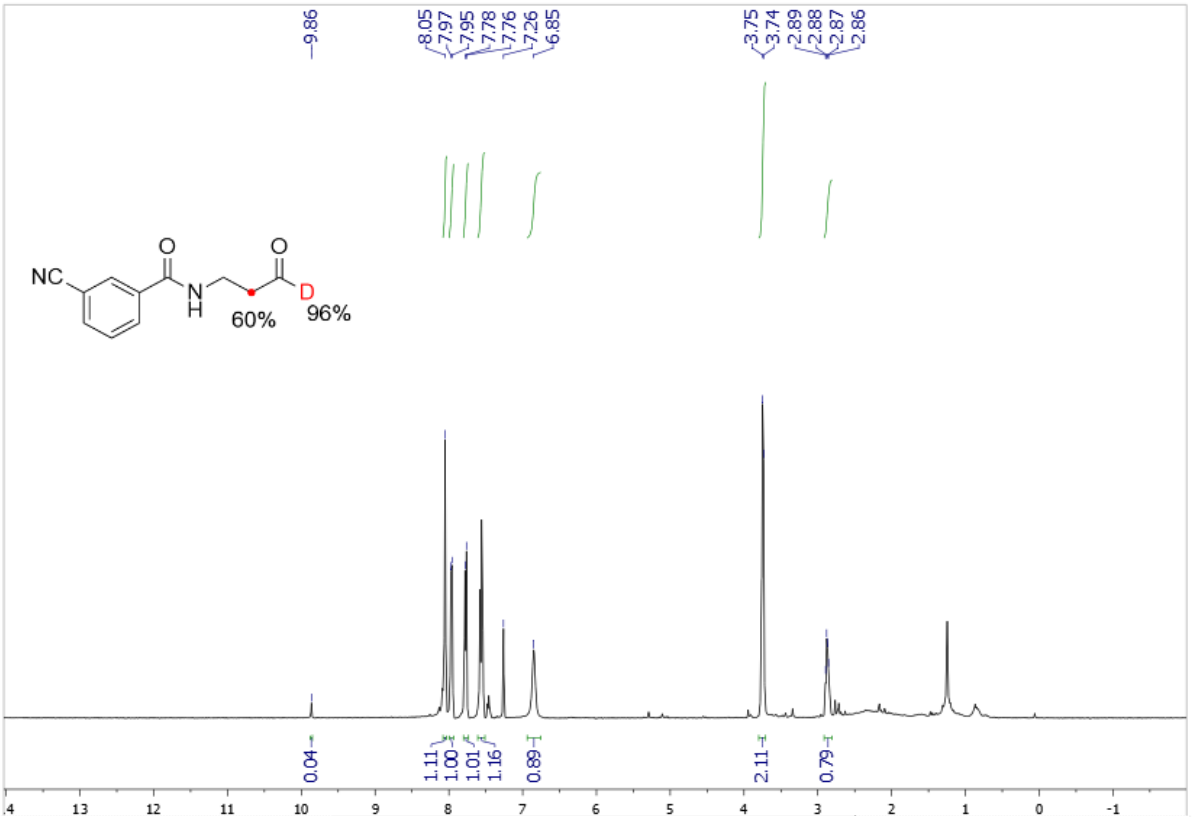


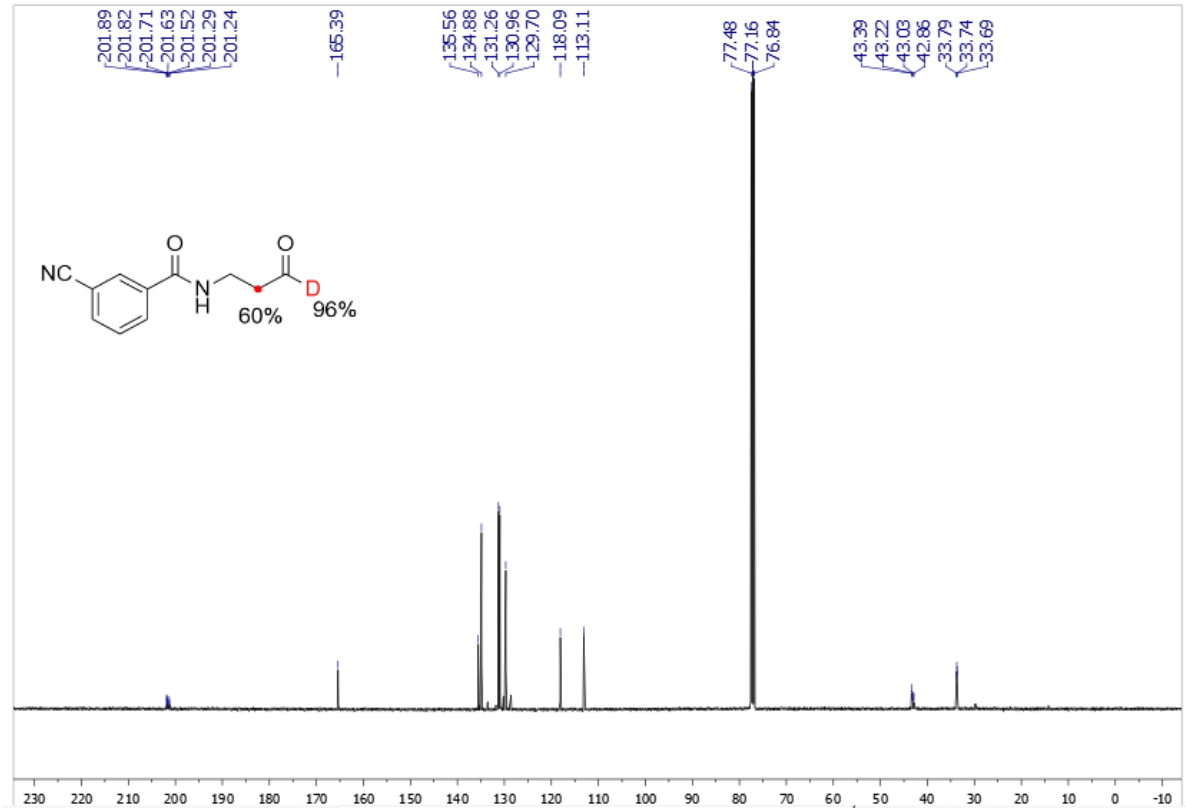
4-(3-Oxopropyl)benzonitrile-*formyl-d1* (3ae)



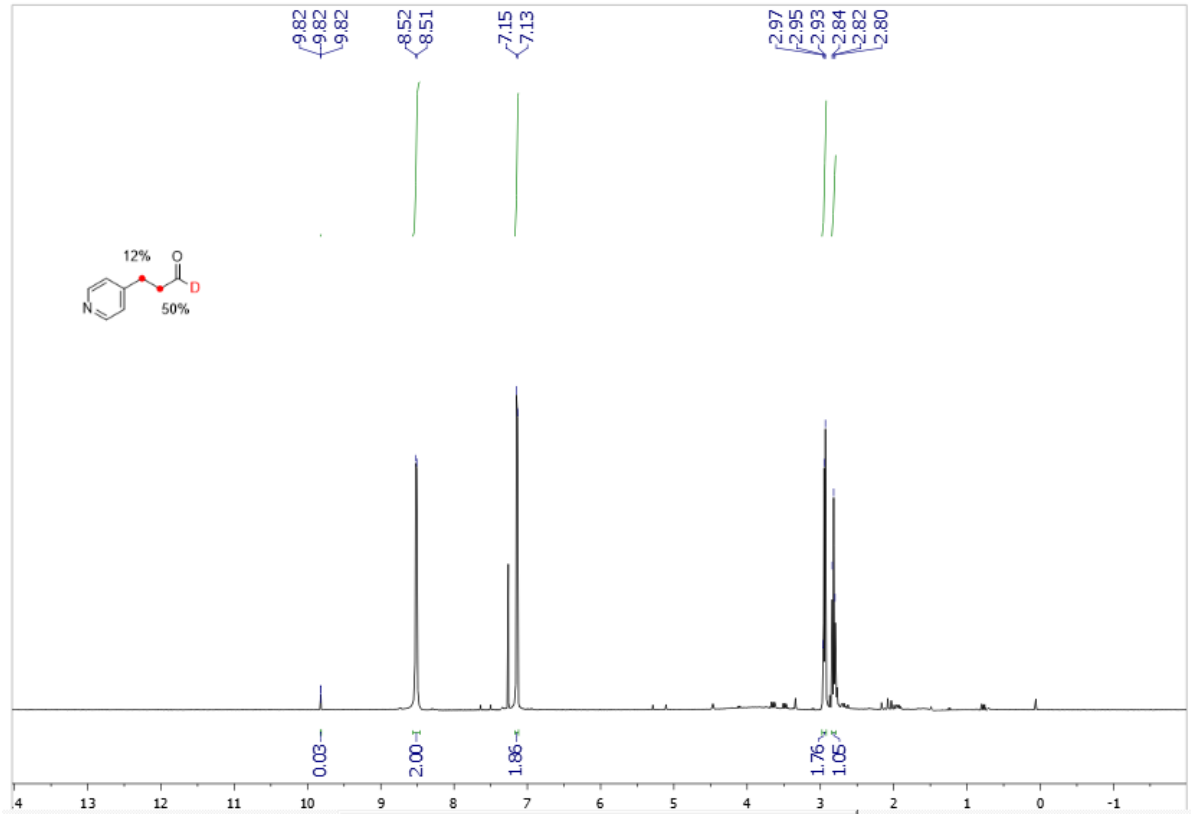


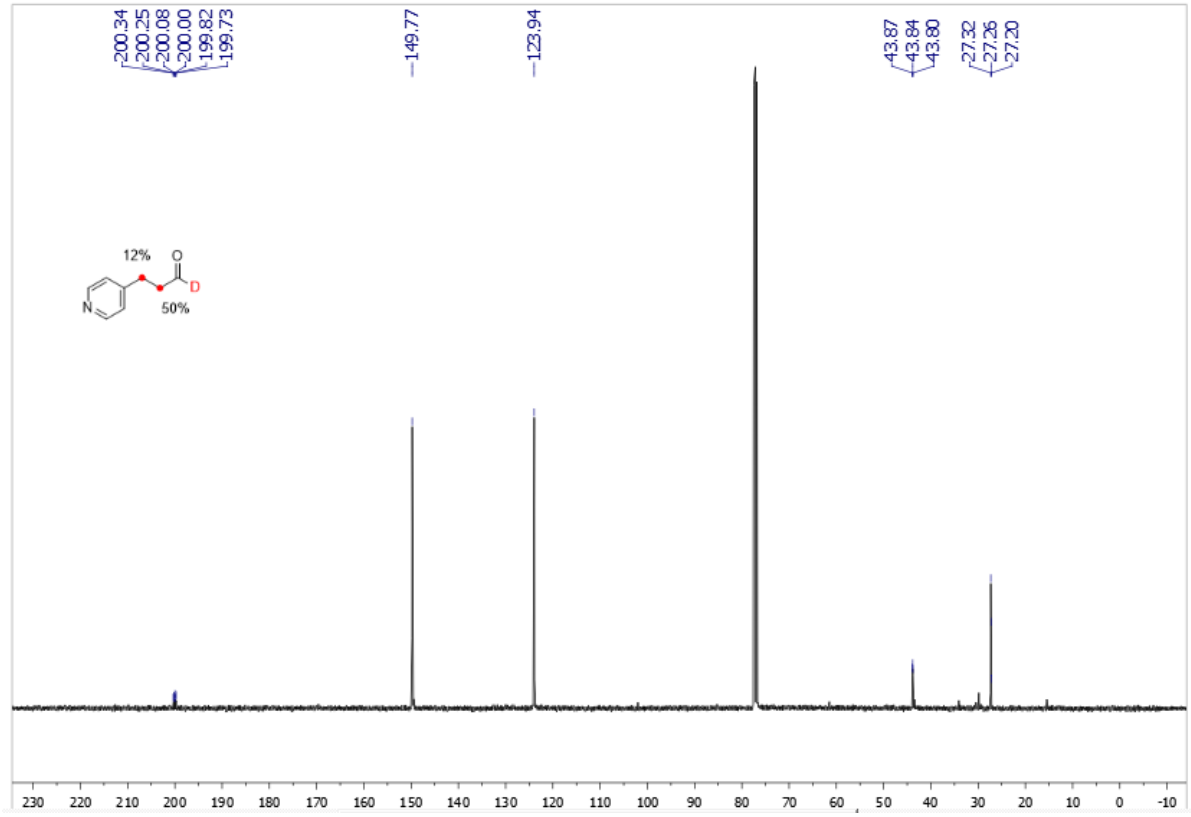
3-Cyano-*N*-(3-oxopropyl)benzamide-*formyl-d1* (3af)



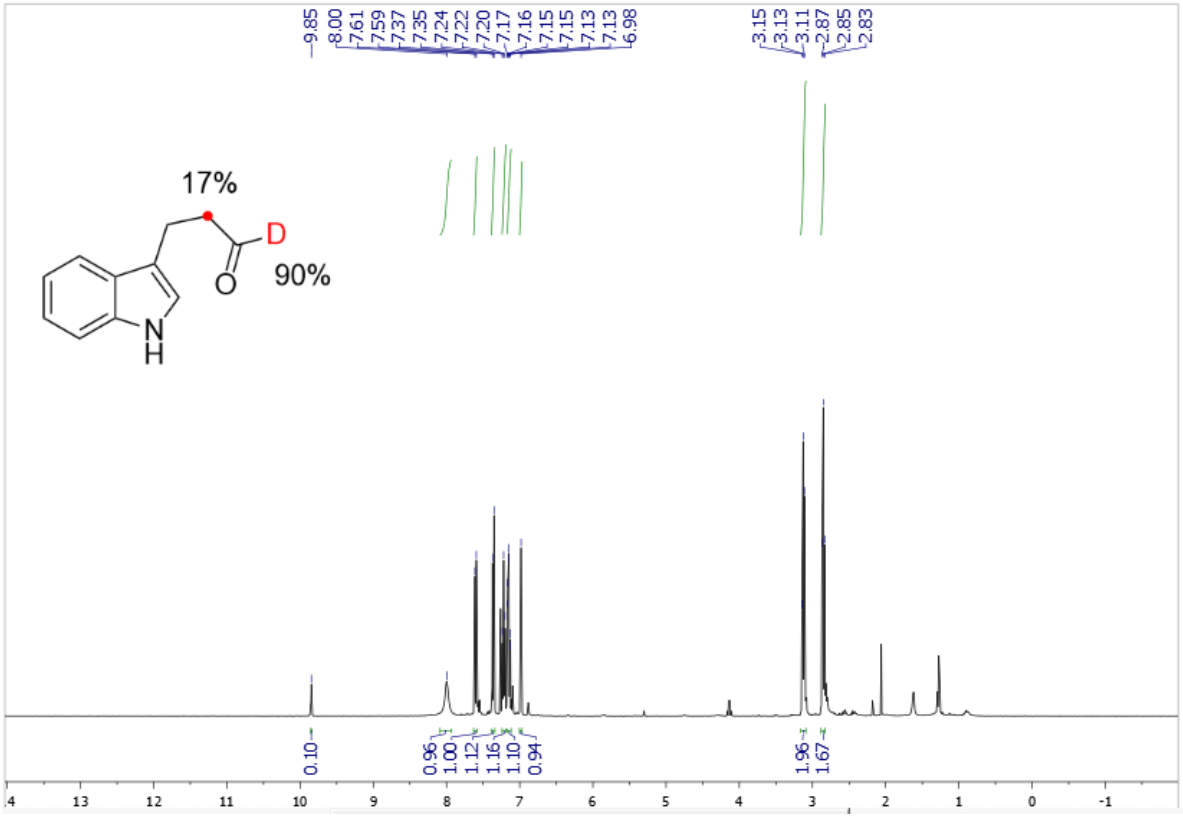


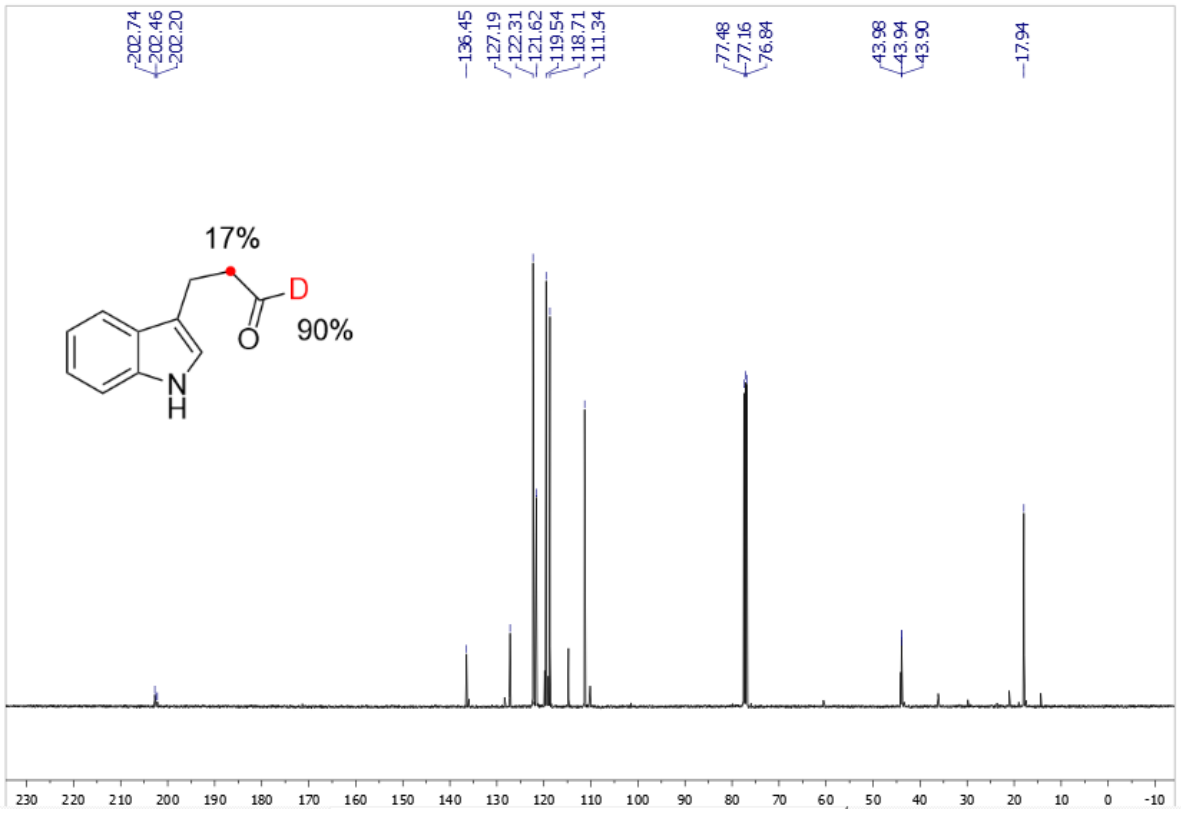
3-(Pyridin-4-yl)propanal-*formyl-d1 (3ag)*



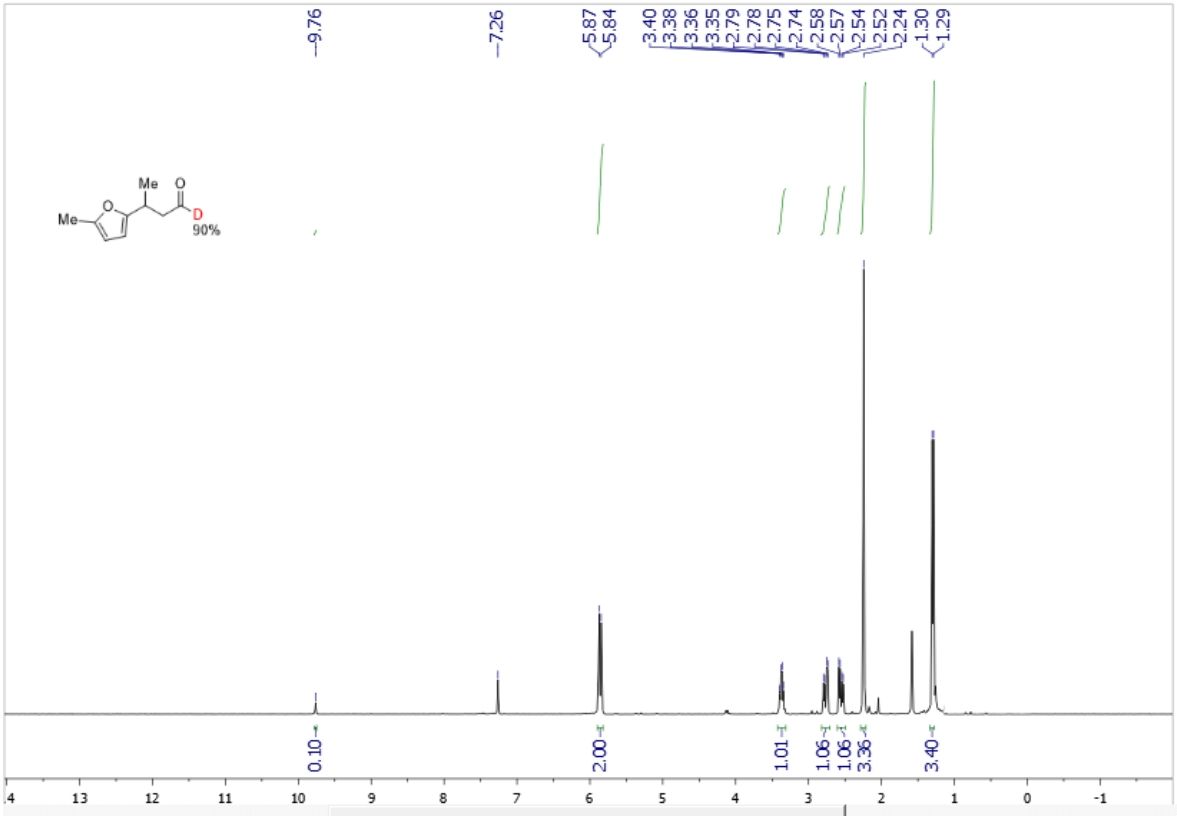


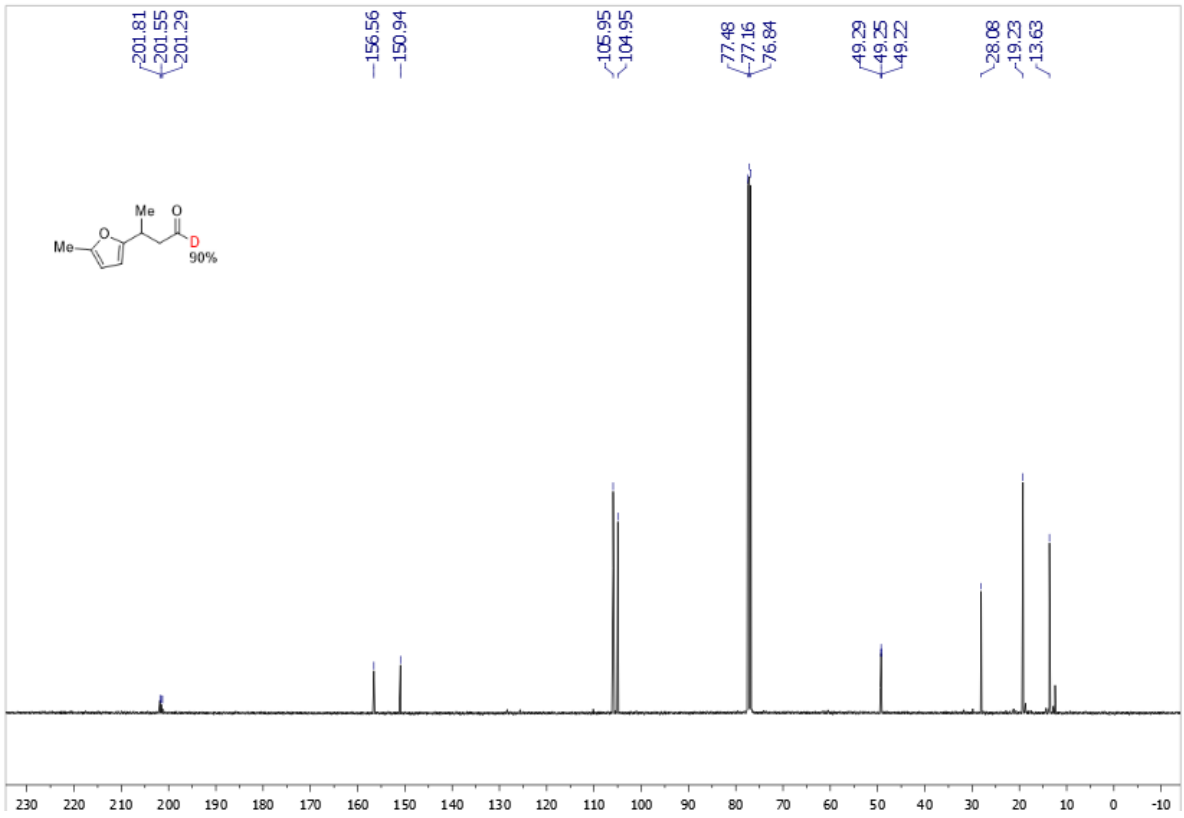
3-(1*H*-indol-3-yl)propanal-*formyl-d1* (3ah)



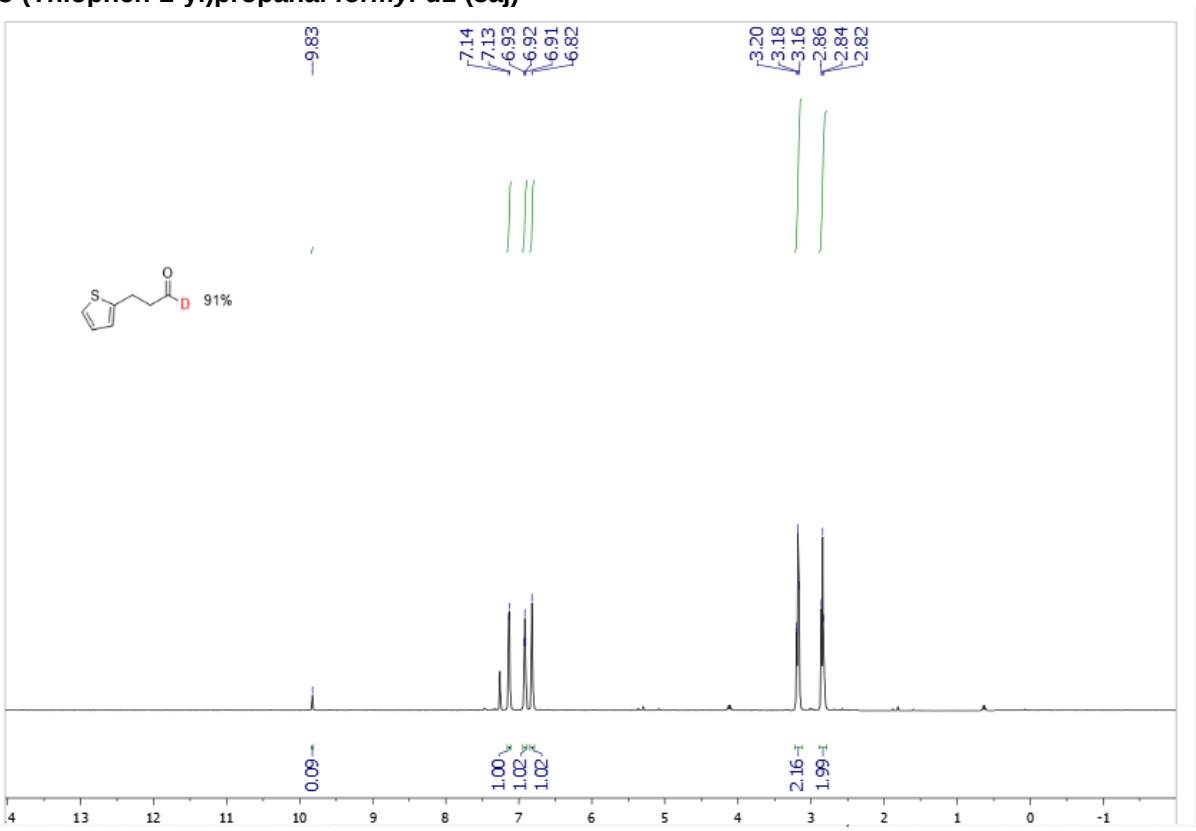


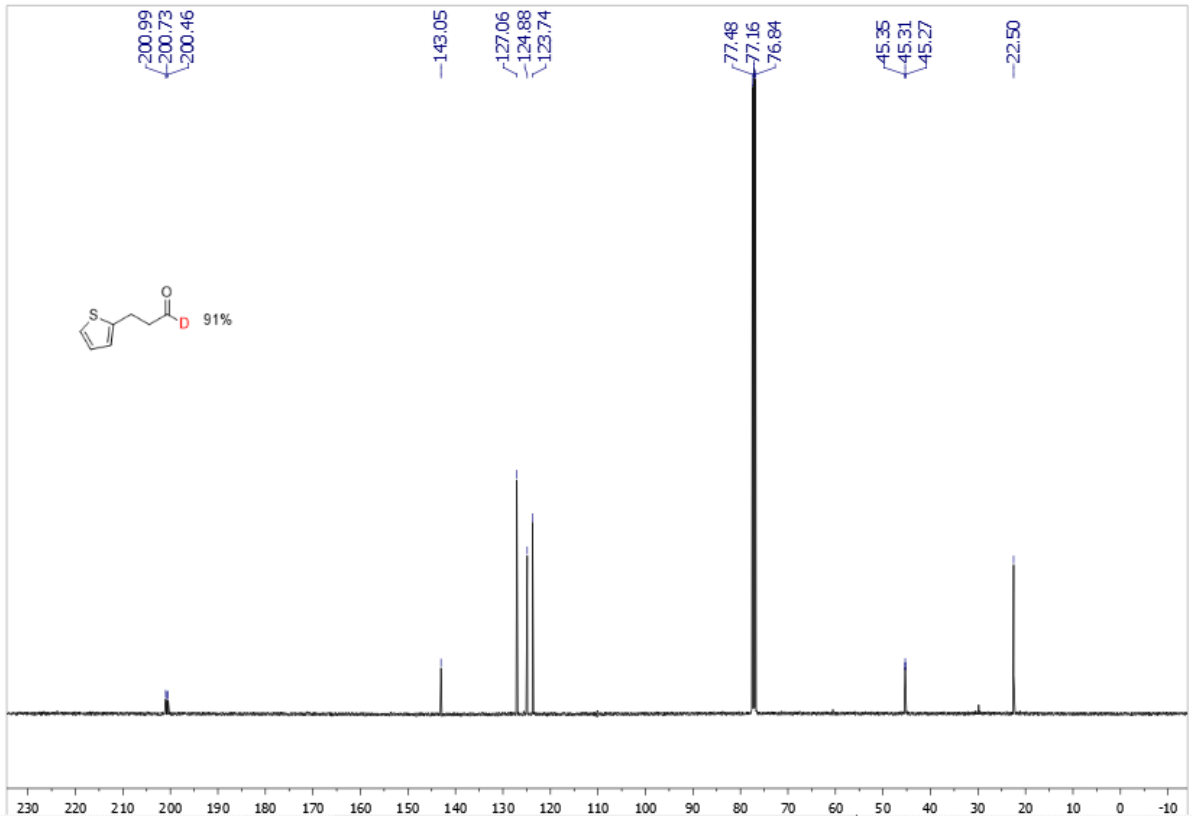
3-(5-Methylfuran-2-yl)butanal-*formyl-d1* (3ai)



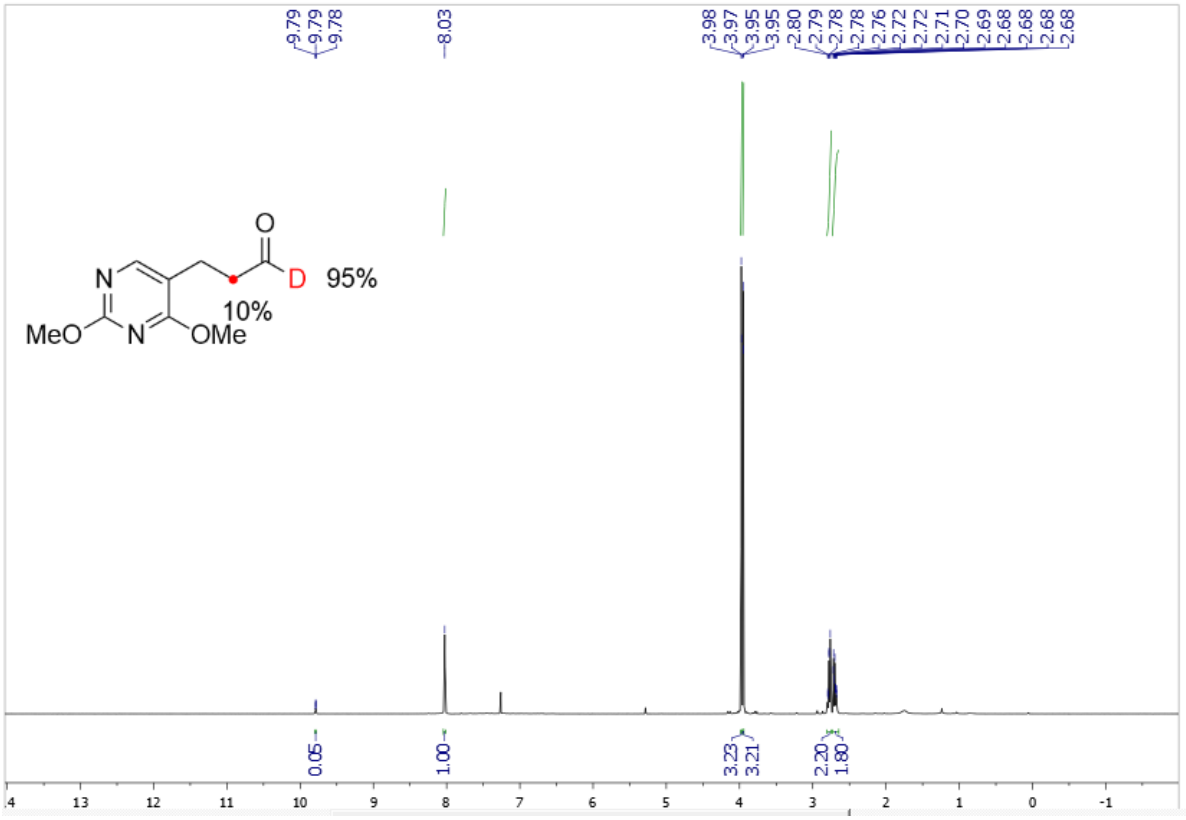


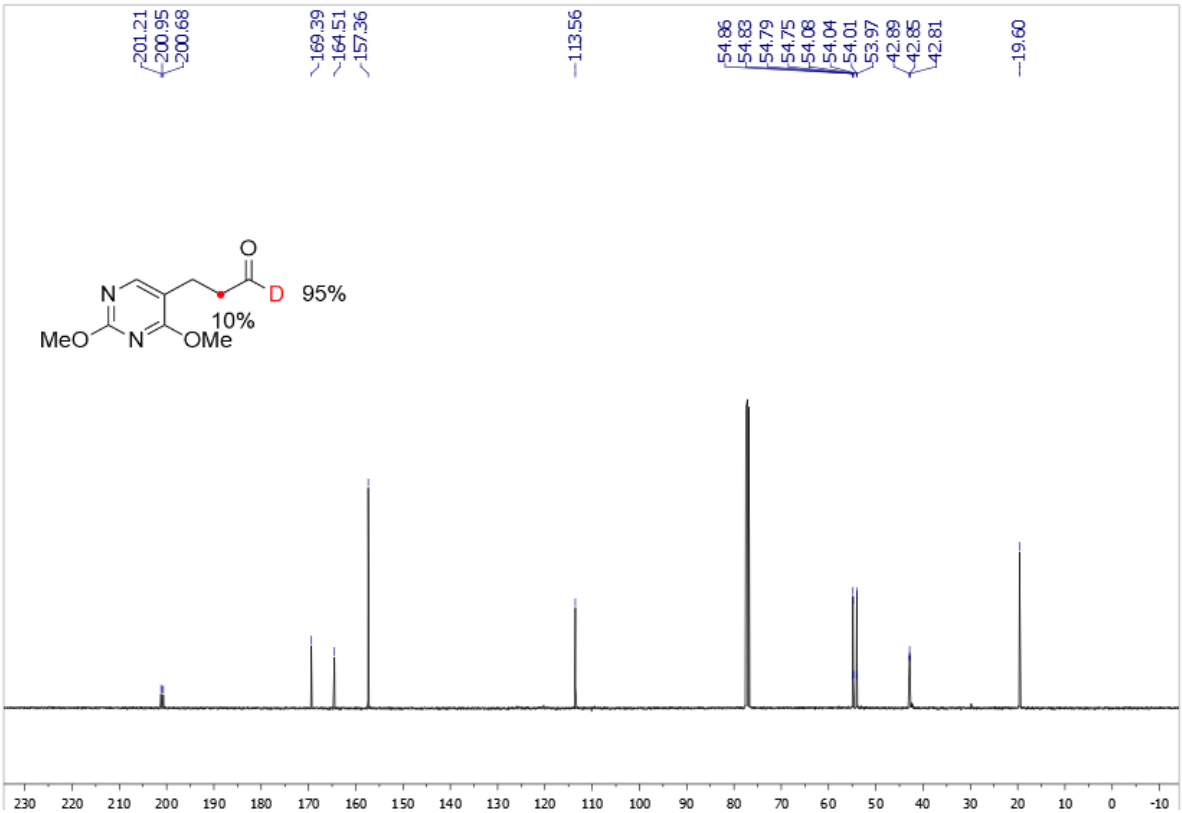
3-(Thiophen-2-yl)propanal-*formyl-d1* (3aj)



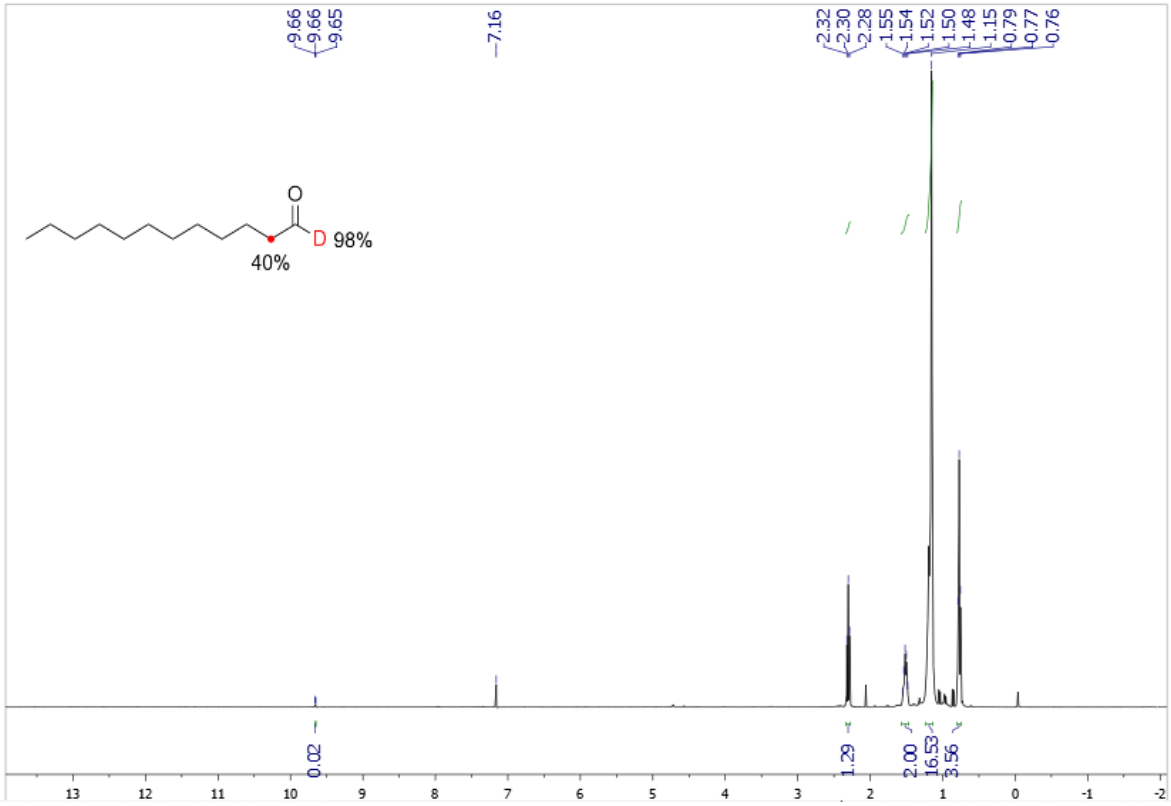


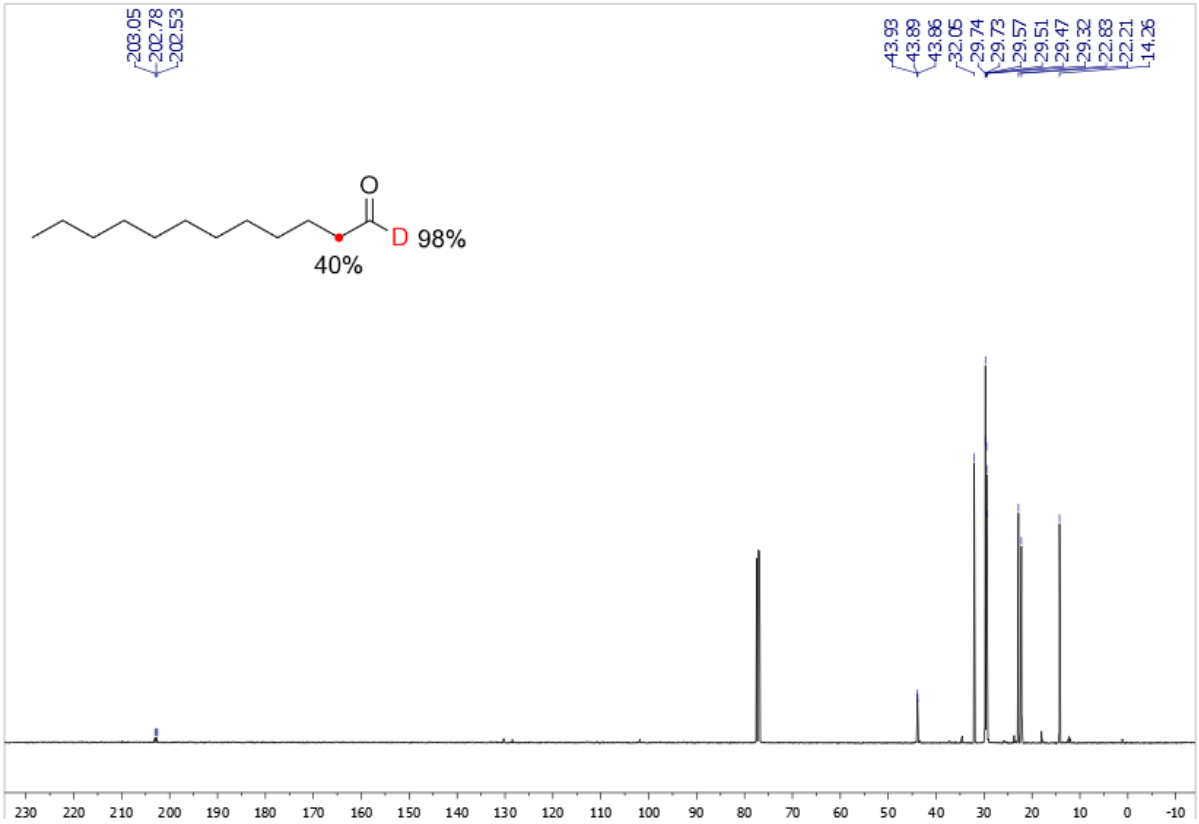
3-(2,4-Dimethoxypyrimidin-5-yl)propanal-*formyl-d1* (3ak)



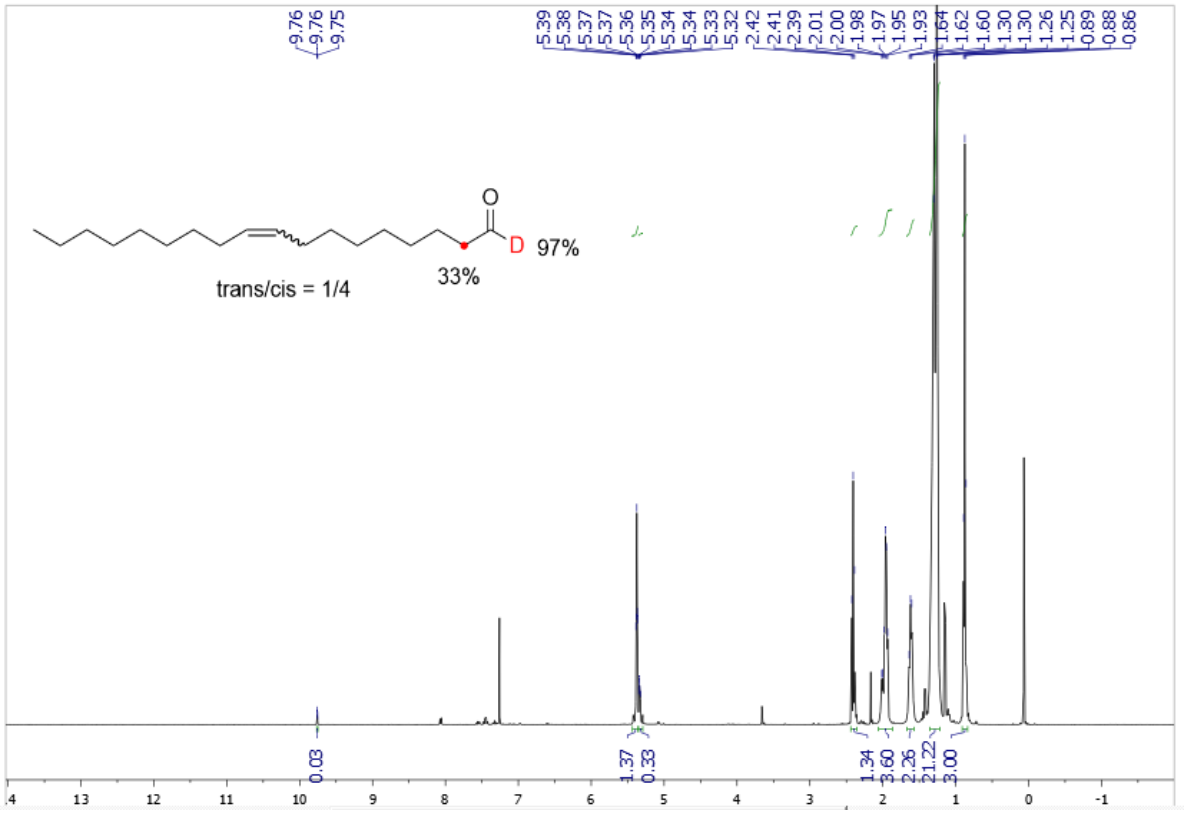


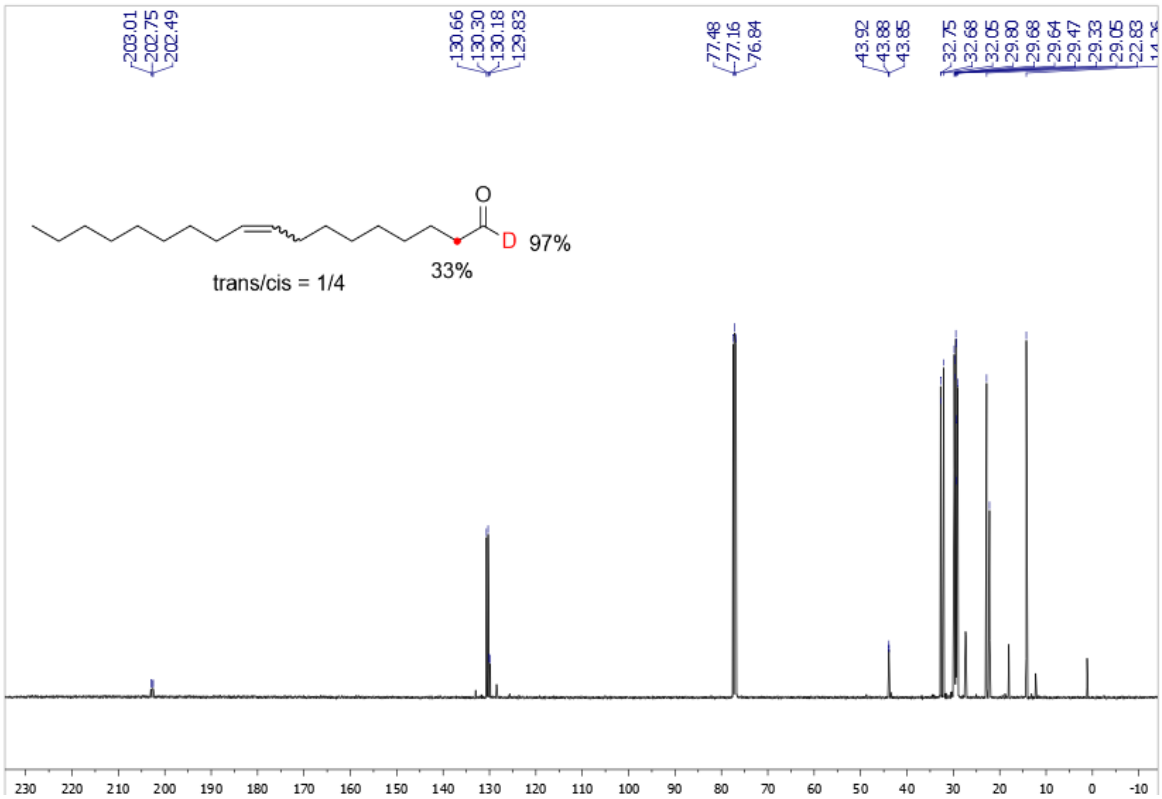
Dodecanal-*formyl-d1* (3al)



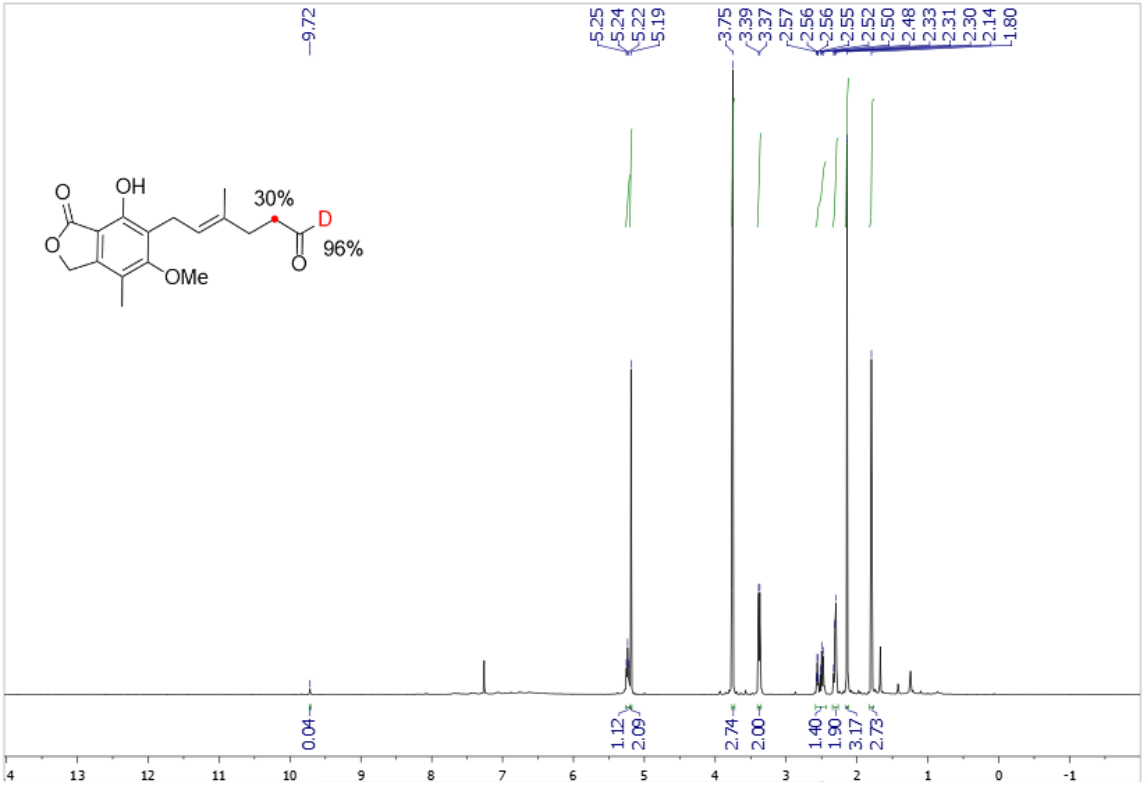


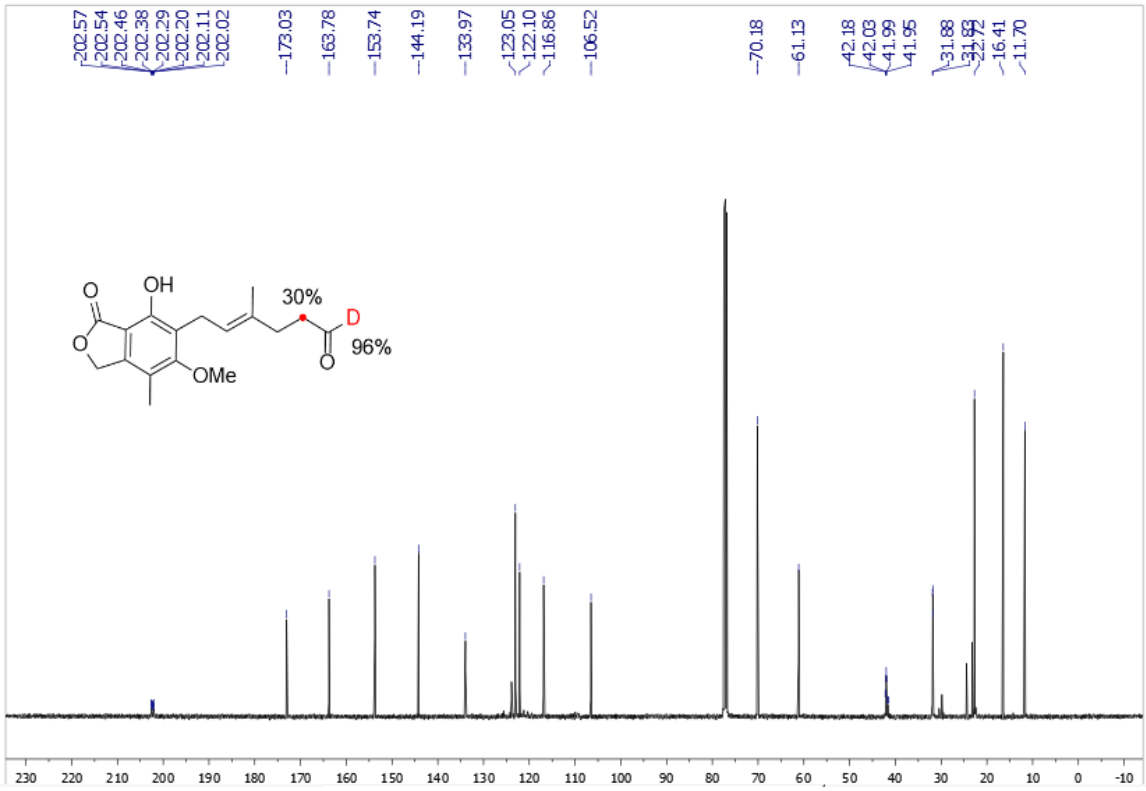
Olealdehyde-*formyl-d1* (3am)



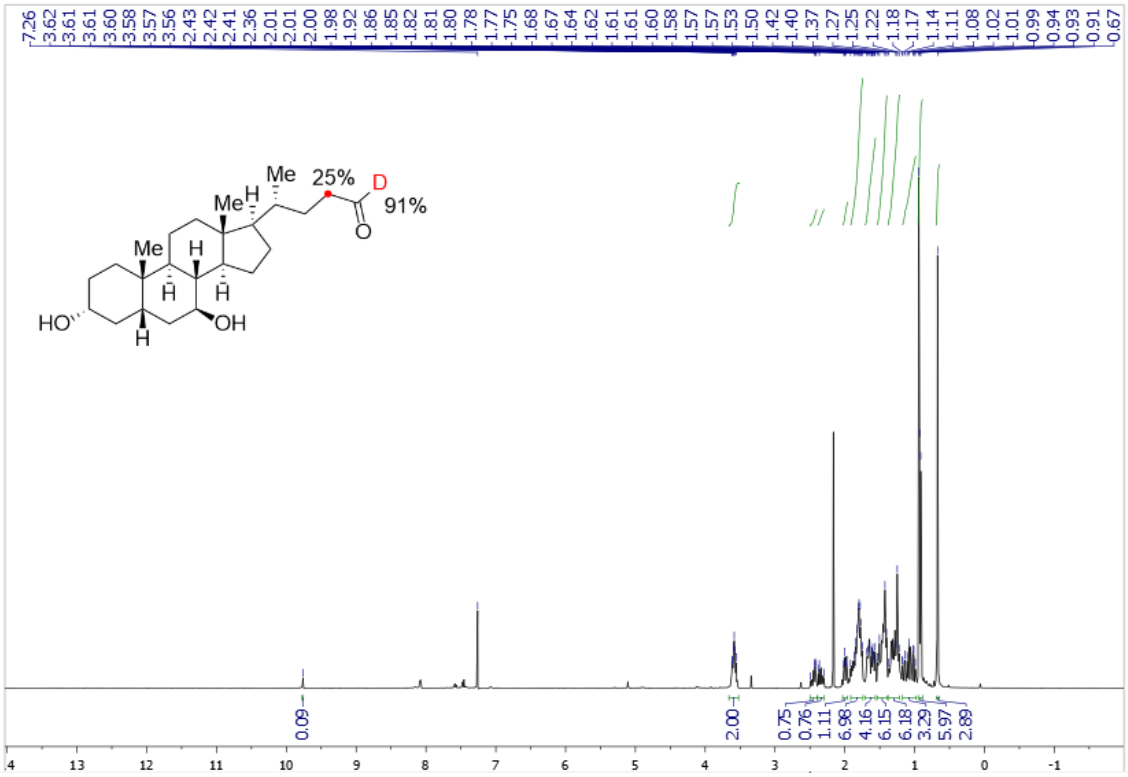


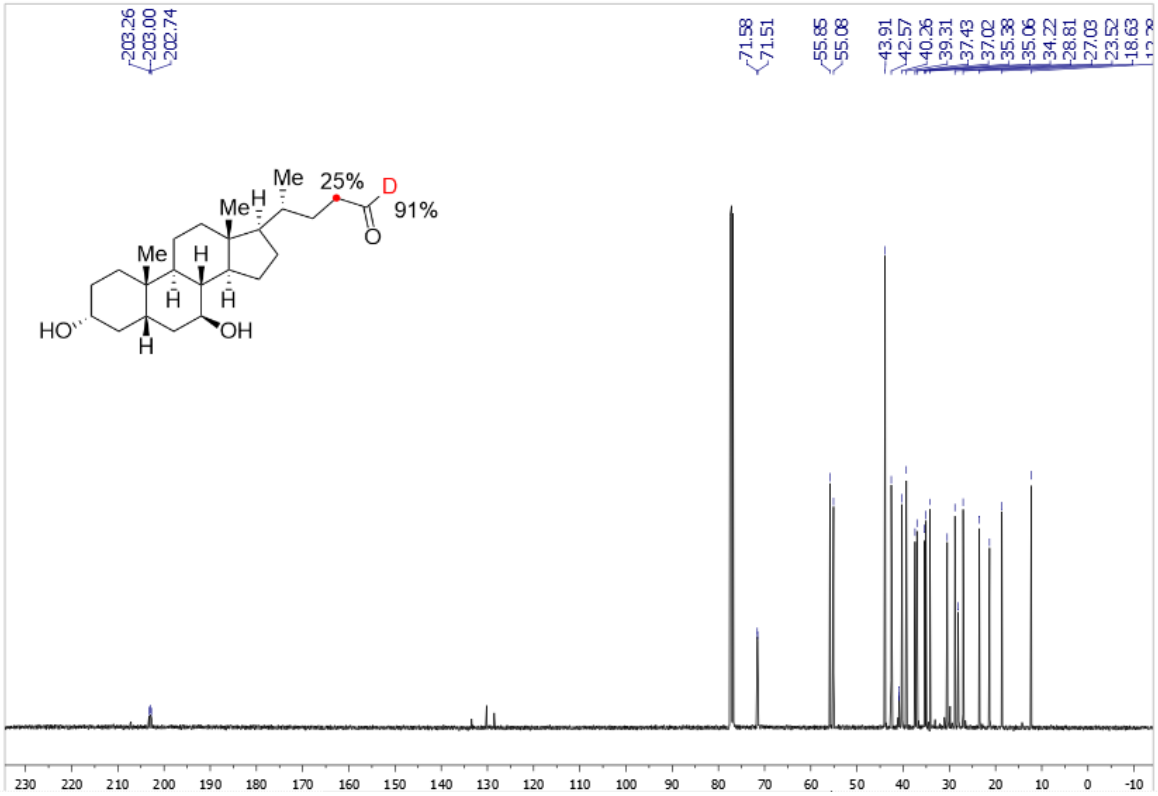
(*E*)-6-(4-hydroxy-6-methoxy-7-methyl-3-oxo-1,3-dihydroisobenzofuran-5-yl)-4-methylhex-4-enal-*formyl-d1* (3an)



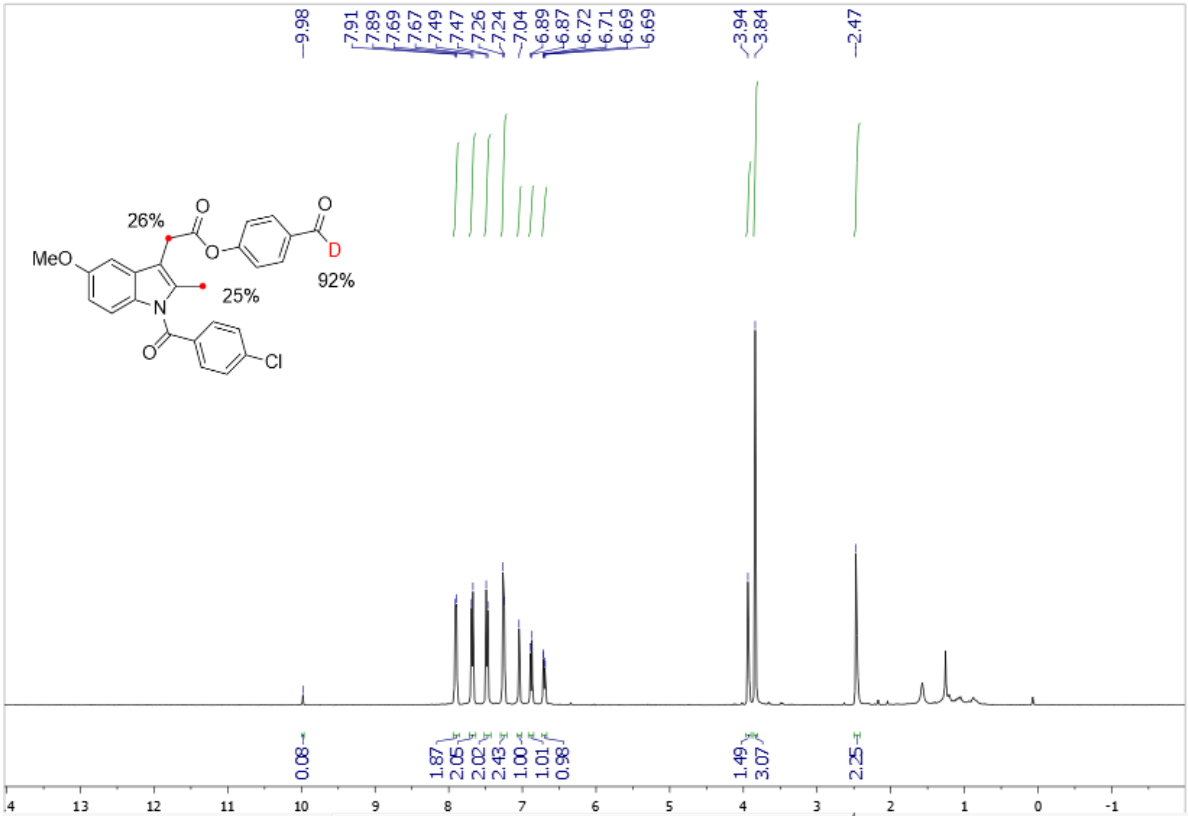


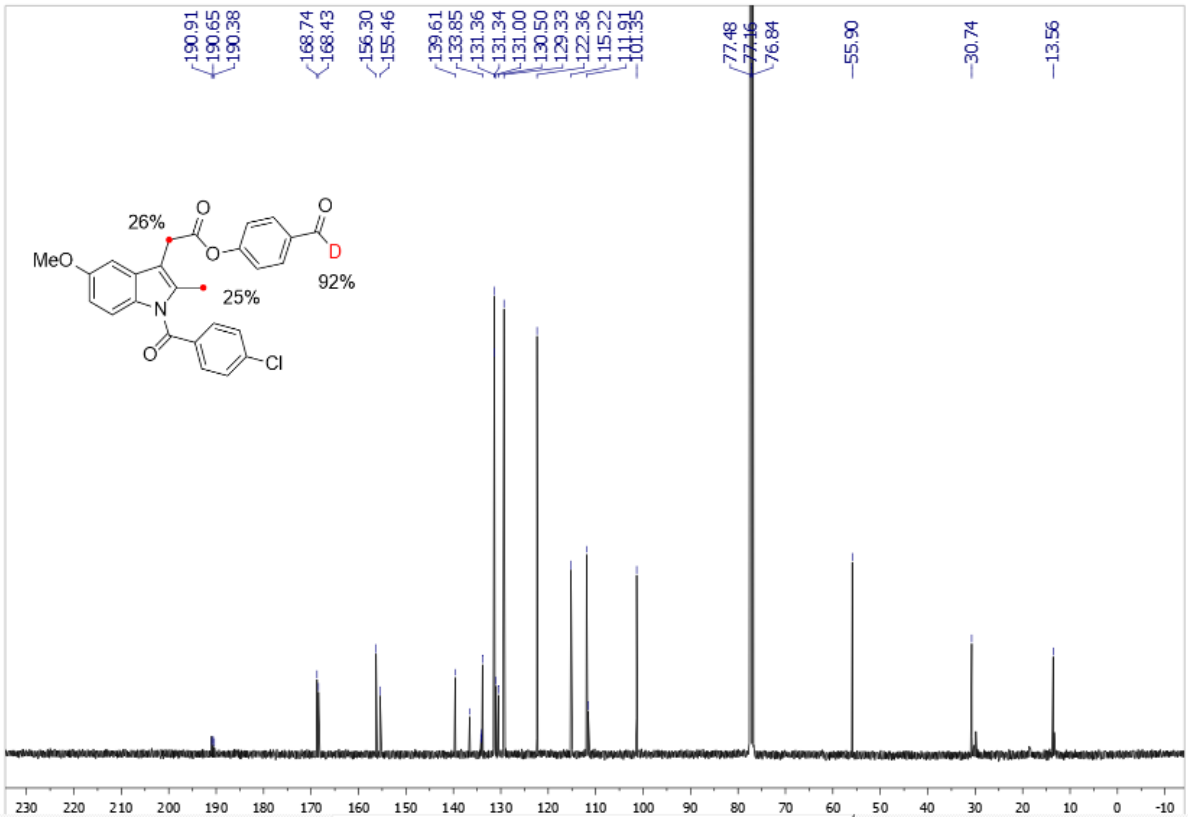
(*R*)-4-((3*R*,5*S*,7*S*,8*R*,9*S*,10*S*,13*R*,14*S*,17*R*)-3,7-dihydroxy-10,13-dimethylhexadecahydro-1*H*-cyclopenta[a]68henanthrene-17-yl)pentanal-*formyl-d1* (3ao)



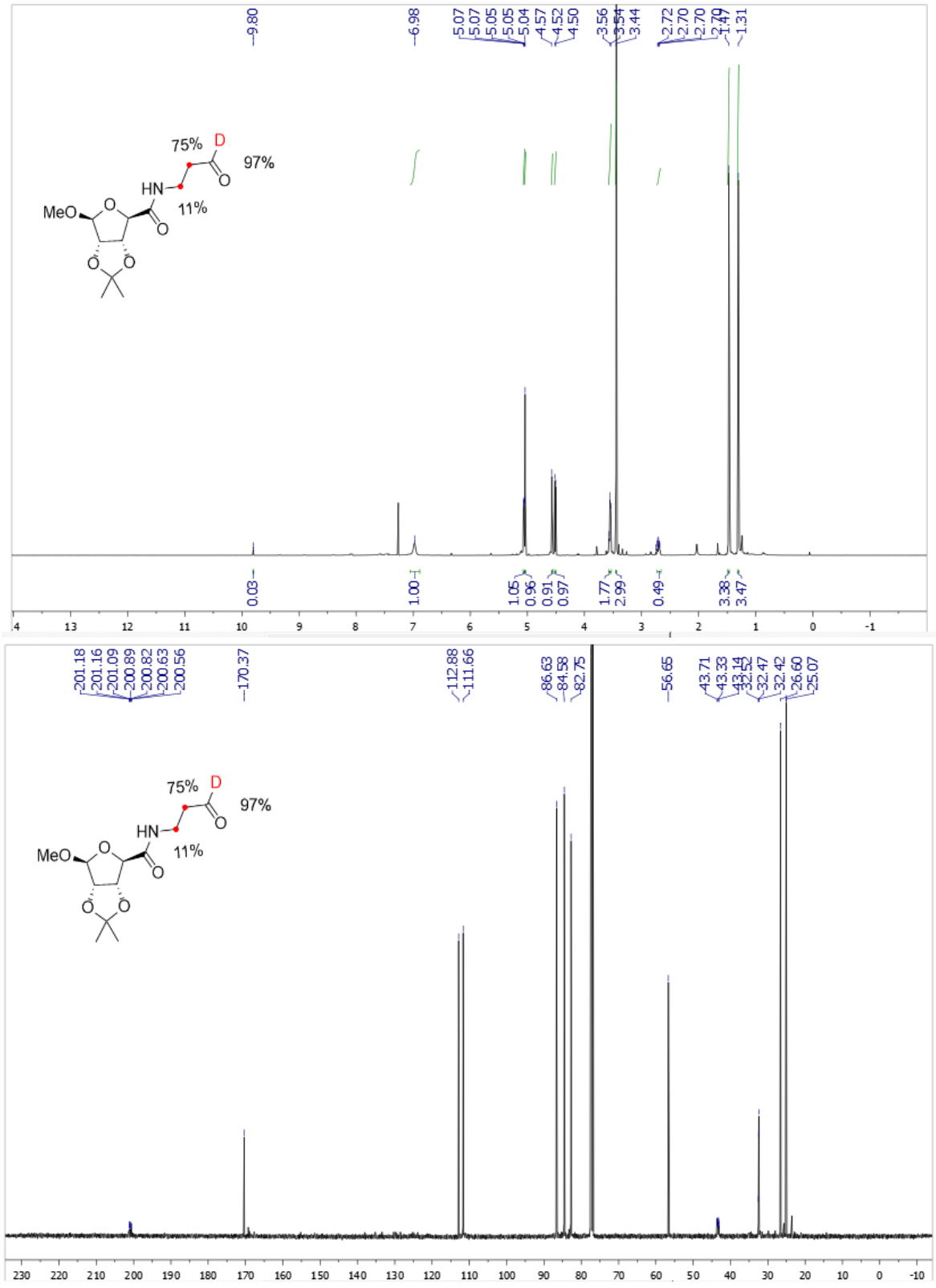


4-Formylphenyl 2-(1-(4-chlorobenzoyl)-5-methoxy-2-methyl-1H-indol-3-yl)acetate-*formyl-d1* (3ap)

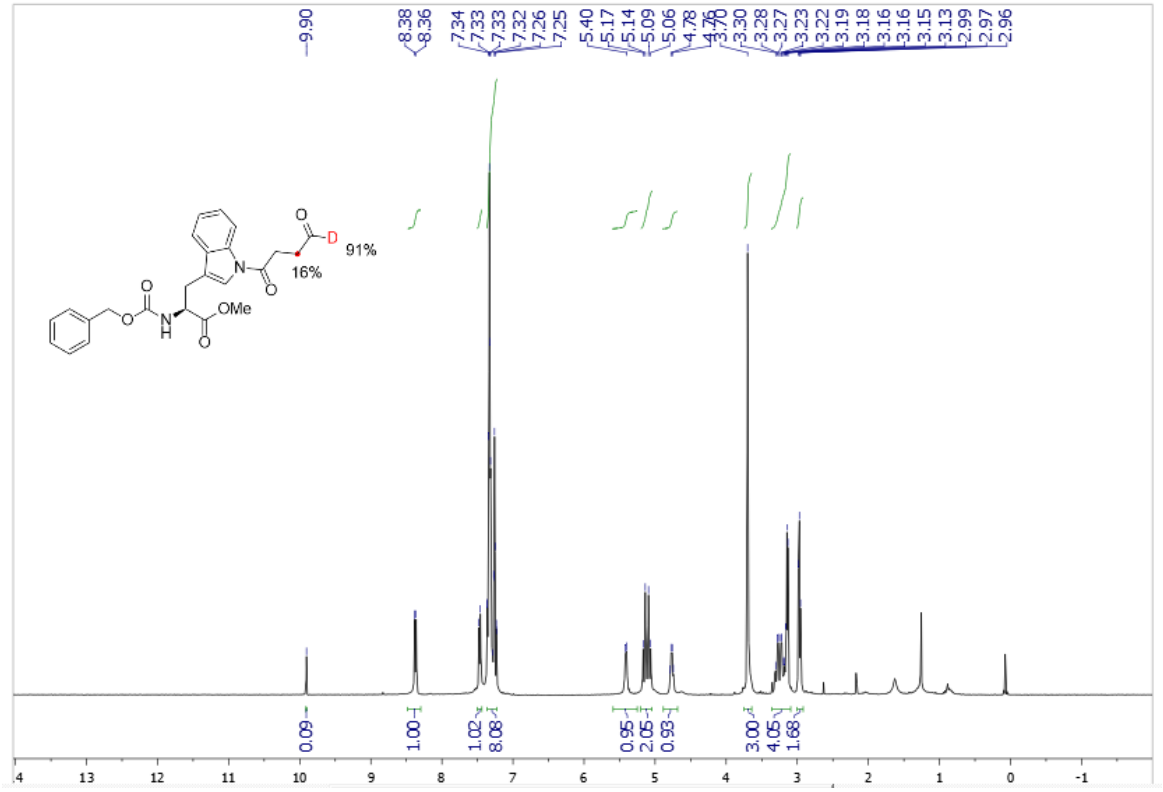


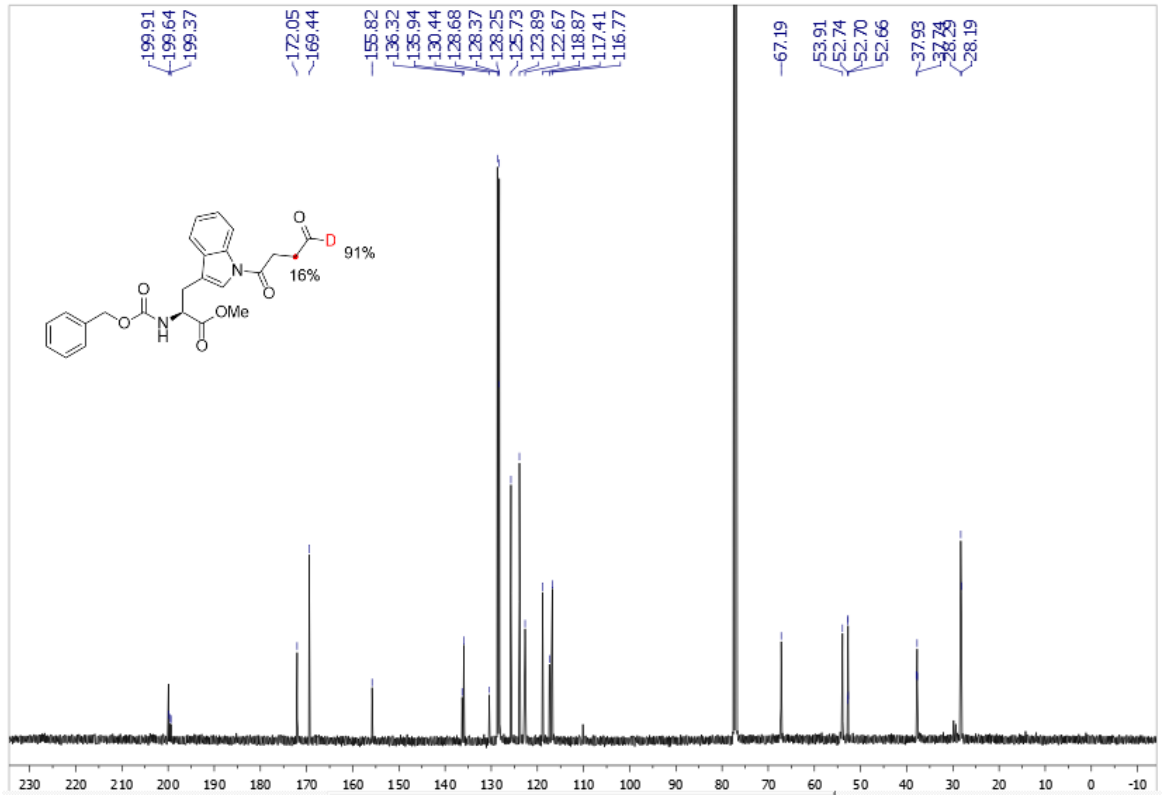


(3*aR*,4*R*,6*S*,6*aS*)-6-Methoxy-2,2-dimethyl-*N*-(3-oxopropyl)tetrahydrofuro[3,4-*d*][1,3]dioxole-4-carboxamide-*formyl-d1* (3aq)

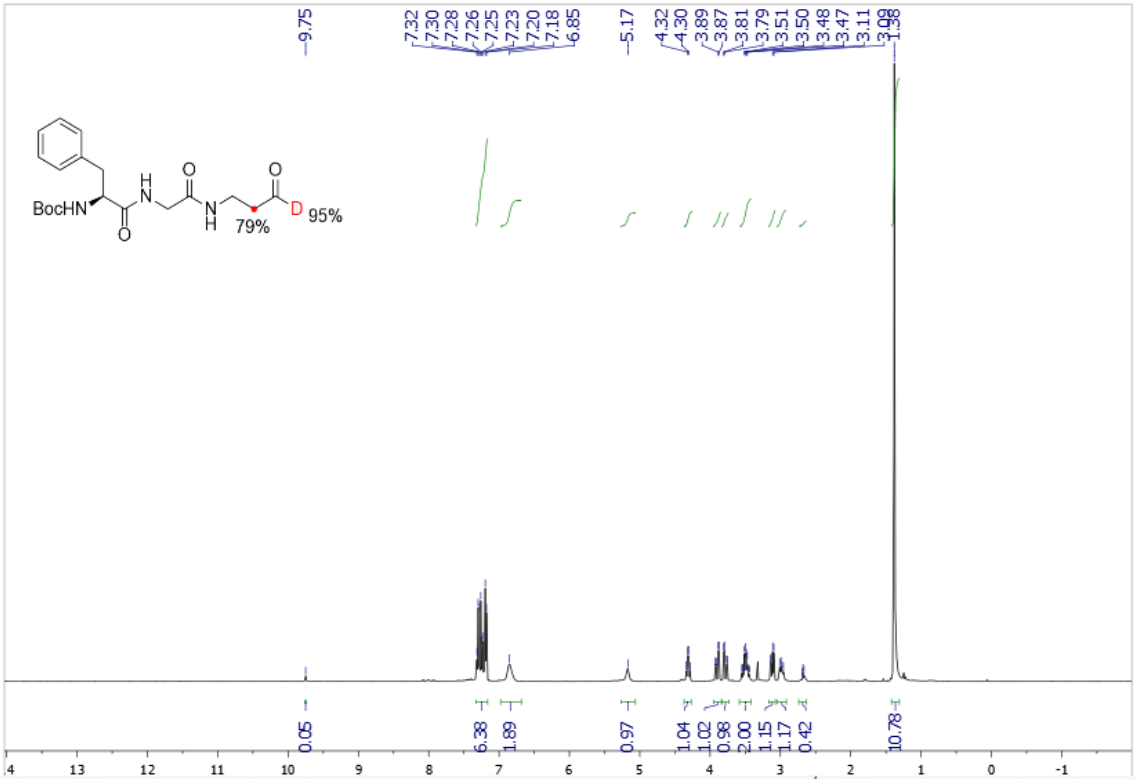


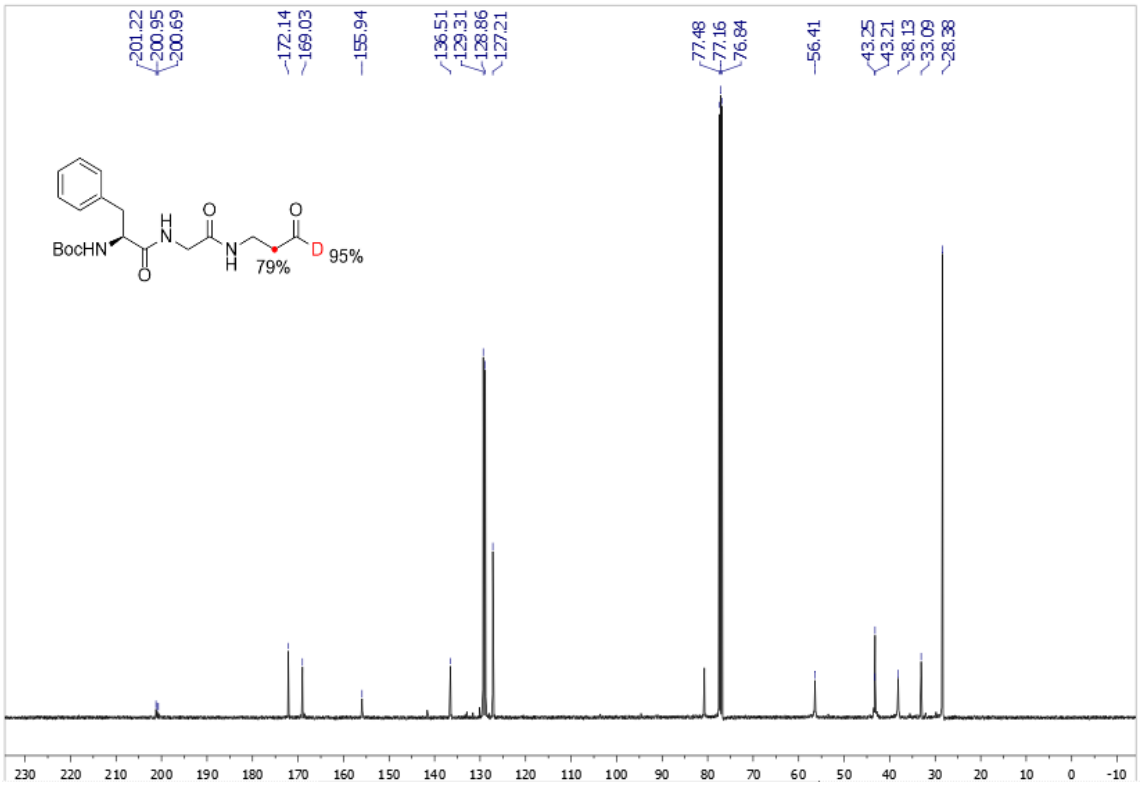
Methyl *N*^{a}-((benzyloxy)carbonyl)-1-(4-oxobutanoyl-4-*d*)-*L*-tryptophanate-*formyl-d1* (3ar)





***tert*-butyl (*S*)-(1-oxo-1-((2-oxo-2-((3-oxopropyl)amino)ethyl)amino)-3-phenylpropan-2-yl)carbamate (3as)**

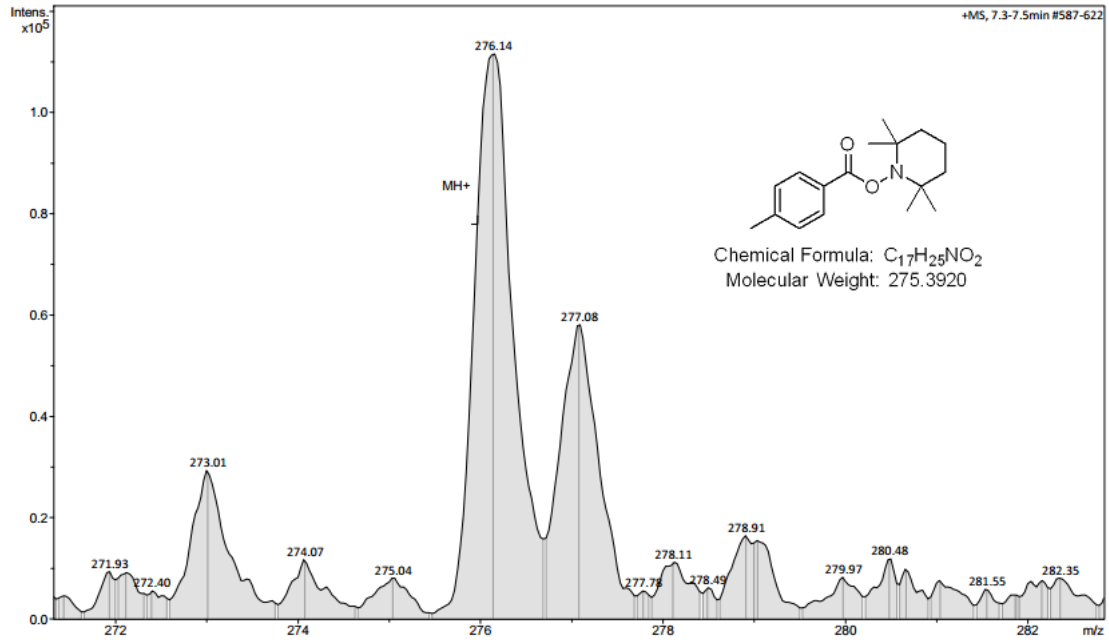

9.75
7.32
7.30
7.28
7.26
7.25
7.23
7.20
7.18
6.85
5.17
4.32
4.30
3.89
3.87
3.81
3.79
3.51
3.50
3.48
3.47
3.11
3.08
1.38
BocHN
79%
95%
0.05
6.38
1.89
0.97
1.04
1.02
0.98
2.00
1.15
1.17
0.42
10.78


201.22
200.95
200.69
172.14
169.03
155.94
136.51
129.31
128.86
127.21
77.48
77.16
76.84
56.41
43.25
43.21
38.13
33.09
28.38
BocHN
79%
95%

Generic Display Report

Analysis Info

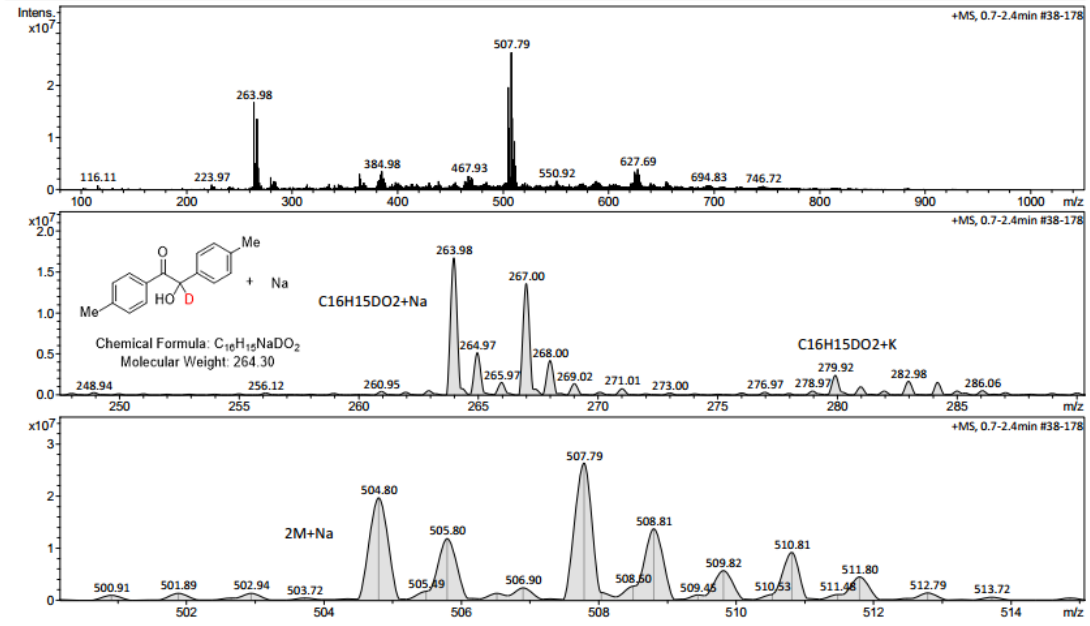
Analysis Name	D:\Data\2019_08\YZ-2758896\2758896_YZ-12-67B_00.d	Acquisition Date	8/14/2019 10:20:11 AM
Method	Tuning Mix.m	Operator	KK
Sample Name	2758896_YZ-12-67B_	Instrument	amaZon SL
Comment	dissolved in 400ul ACN, then diluted 1000x in ACN/H2O/0.1%FA		

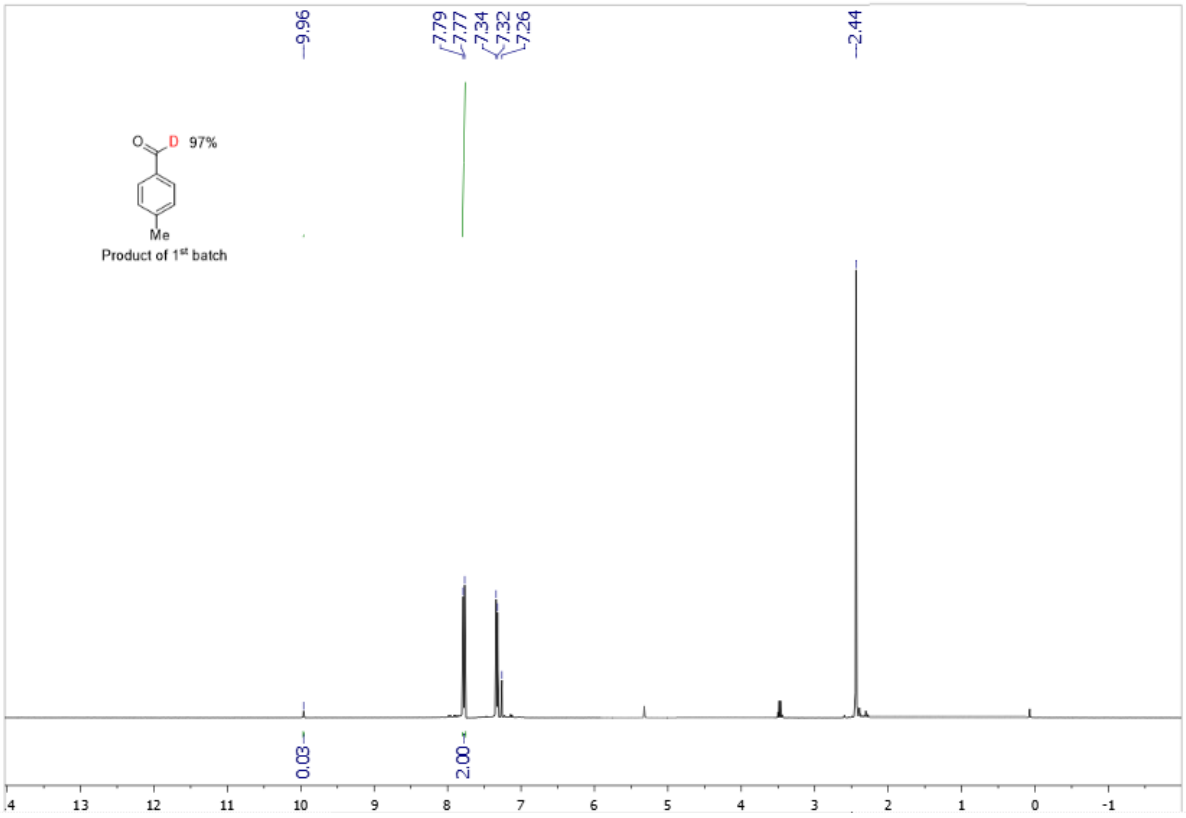


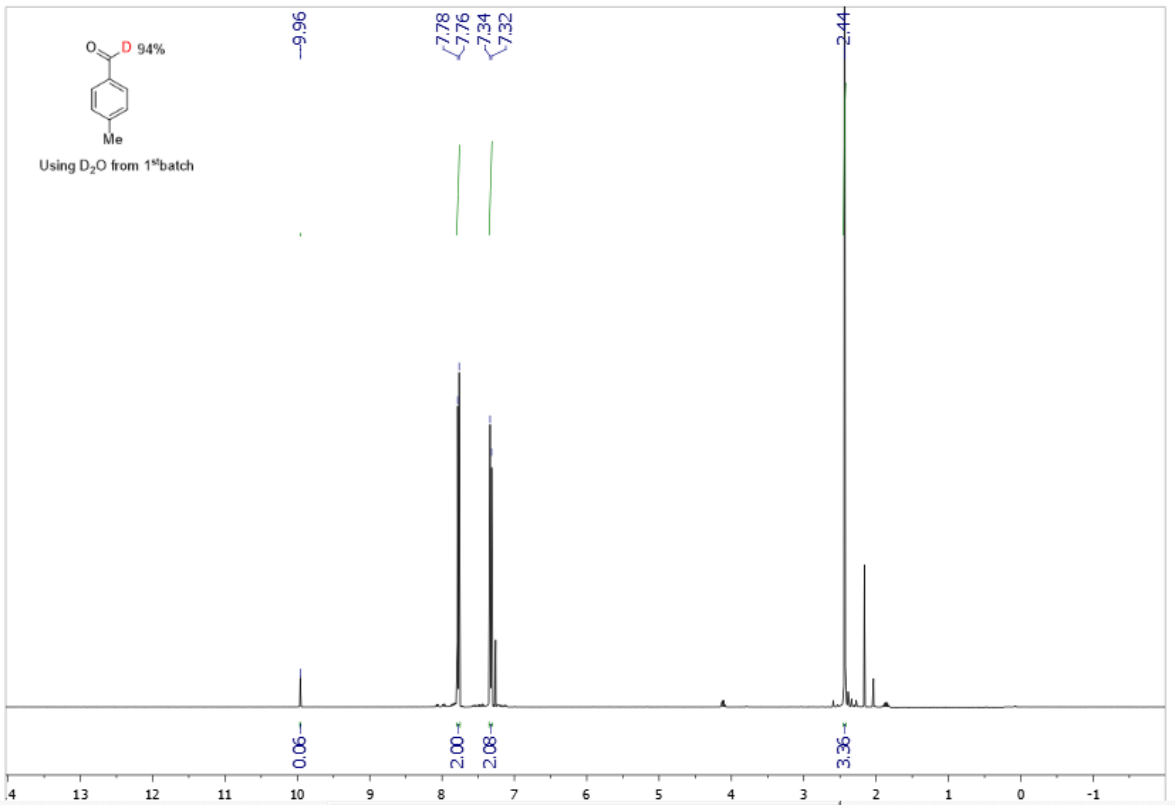
Generic Display Report

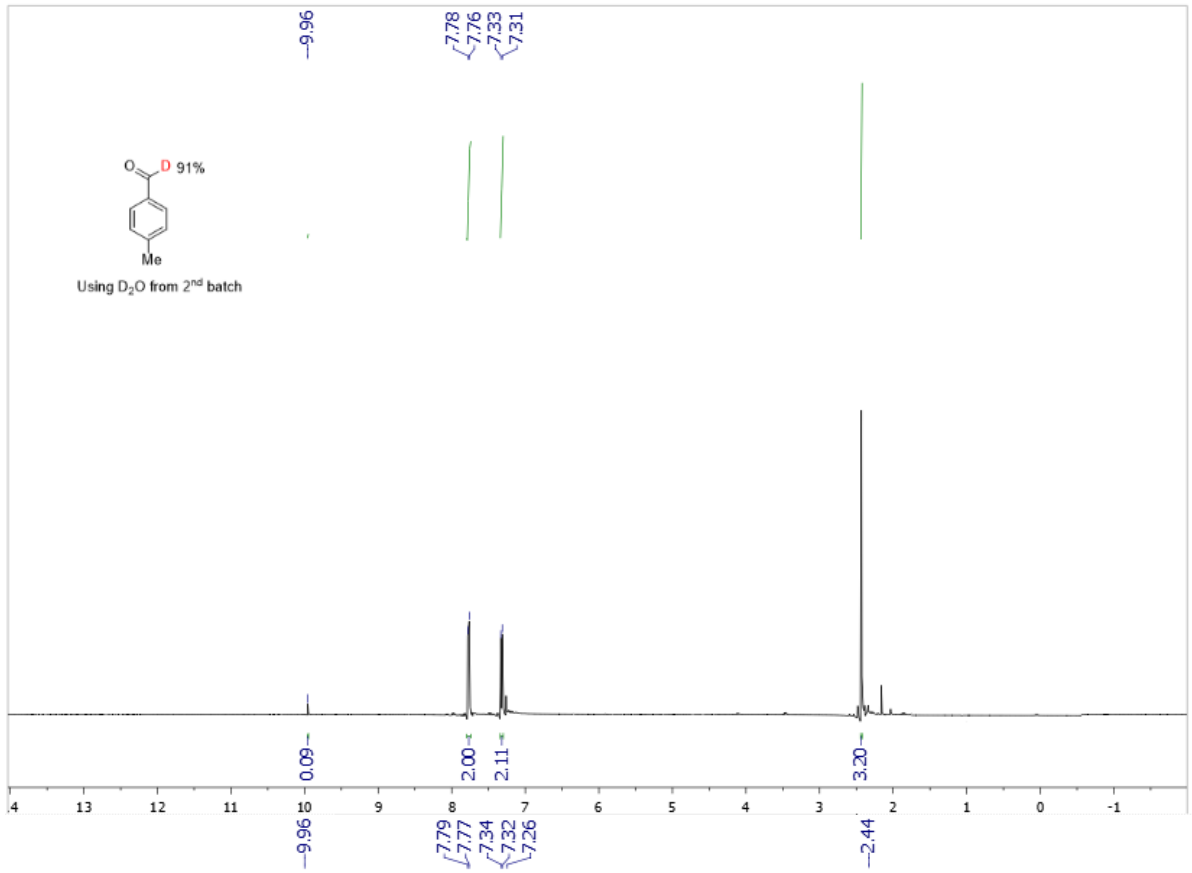
Analysis Info

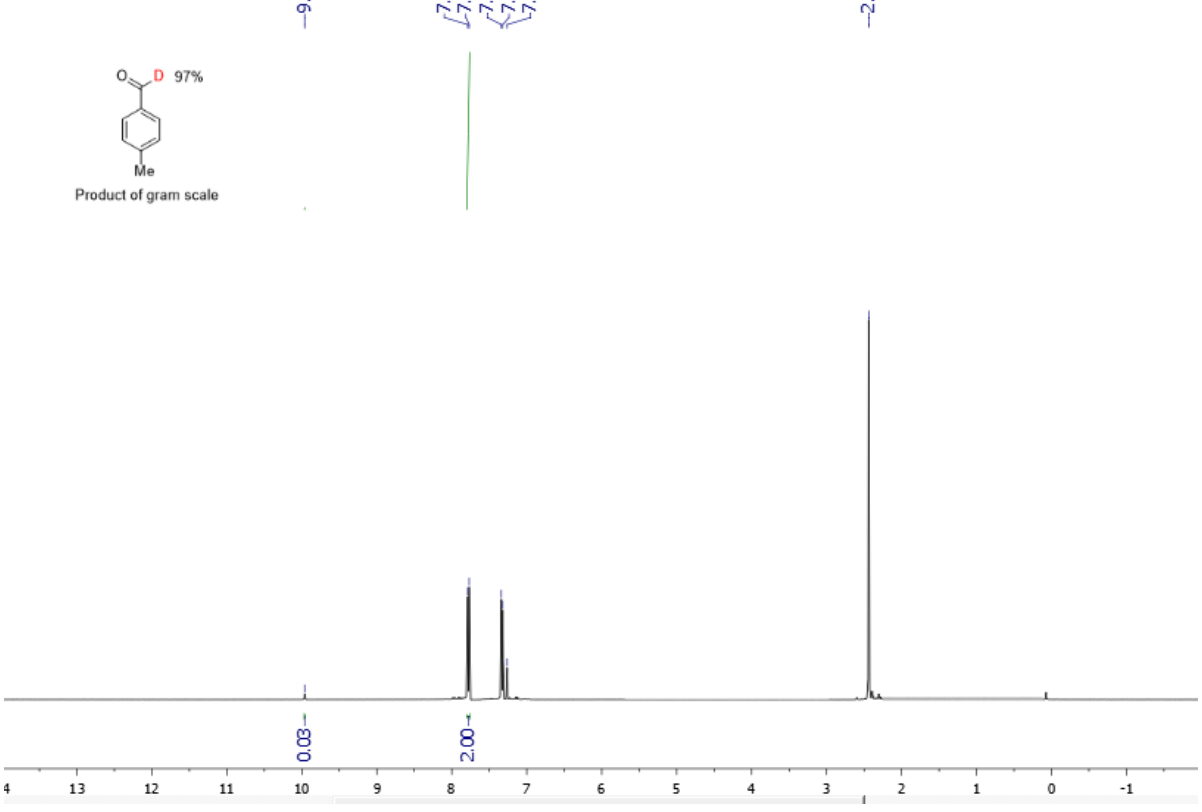
Analysis Name	D:\Data\2020_01\YZ-3080350\YZ-3080350_YZ-13-32P_inACN_only.d	Acquisition Date	1/8/2020 10:57:36 AM
Method	Tuning Mix.m	Operator	KK
Sample Name	YZ-13-32P	Instrument	amaZon SL
Comment	100x dil with ACN		




9.96
7.79
7.77
7.34
7.32
7.26
2.44
O
D 97%
Me
Product of 1st batch
0.03
2.00
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1


9.96
7.78
7.76
7.34
7.32
2.44
O
D 94%
Me
Using D2O from 1stbatch
0.06
2.00
2.08
3.36
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1


9.96
7.78
7.76
7.33
7.31
D 91%
Me
Using D2O from 2nd batch
0.09
2.00
2.11
3.20
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1


9.96
7.79
7.77
7.34
7.32
7.26
2.44
D 97%
Me
Product of gram scale
0.03
2.00
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1

9. Reference

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