Supporting Information

Metal- and Additive-free C-H Oxygenation of Alkylarenes by Visible-light Photoredox Catalysis

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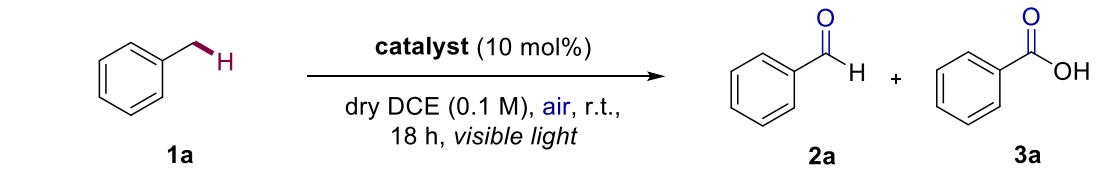
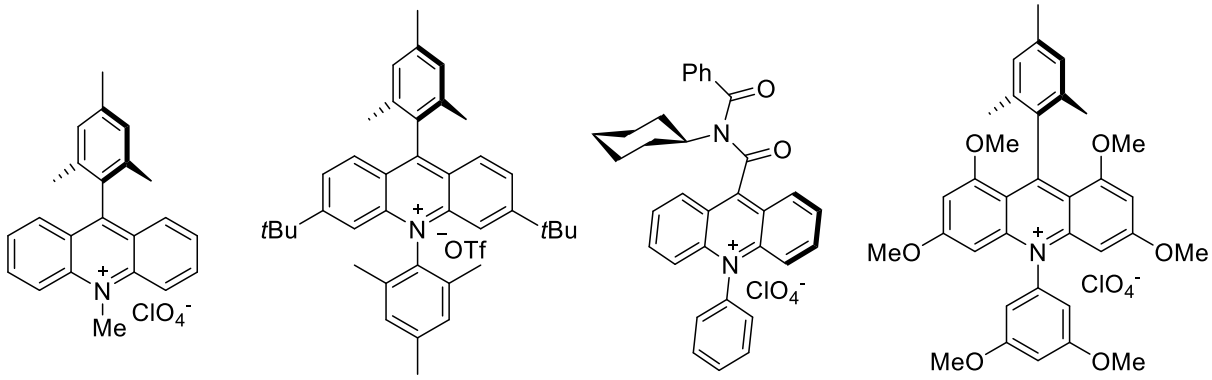
General Information

^{1}H and ^{13}C-NMR spectra were recorded in $CDCl_3$ (reference signals: ^{1}H = 7.26 ppm, ^{13}C = 77.2 ppm) on a Bruker Avance II 300 or Bruker Avance II 400. ^{1}H-NMR chemical shifts are given relative to TMS and are referenced to the solvent signal. Chemical shifts (δ) are given in ppm and spin-spin coupling constants (*J*) are given in Hz. Analytical thin layer chromatography was performed using silica gel 60 F254 and a solution of $KMnO_4$ served as staining agent. Column chromatography was performed on silica gel 60 (0.040-0.063 mm). ESI accurate masses were measured on a MicroTof (Bruker Daltronics, Bremen) with loop injection or on an LTQ Orbitrap LTQ XL (Thermo-Fisher Scientific, Bremen) with nano spray (alternatively HPLC, loop injection, syringe pump). Mass calibration on the MicroTof device was performed by using sodium formate cluster ions, immediately followed by the sample in a quasi-internal calibration. Steady-state fluorescence measurements were performed with a FP8500-Spectrofluorometer JASCO with the excitation of the chromophore at 420 nm. UV-Vis measurements were done in a JASCO V-750 Spectrophotometer at 25 °C. Photoreactions were performed in 10 mL headspace vials, ND20 from Carlo Erba. The vials were closed with aluminum headspace caps, ND20 equipped with a septum. The applied 5 W high power LEDs were supplied by Avonec with an emission maximum at 457 nm. The reactions were performed in a photoreactor consisting of a hollow metal block, which was cooled down by water. The scale ups were performed with two 30 W AC200-240V LEDs by Evoluchem with 450 nm wavelengths. The imide-acridinium salt **PC III** was prepared according to a recently published protocol by our group.[1] Other solvents and commercially available reagents were used without further purification.

Optimization of the photocatalyzed aldehyde synthesis

Table S1: Catalyst screening.

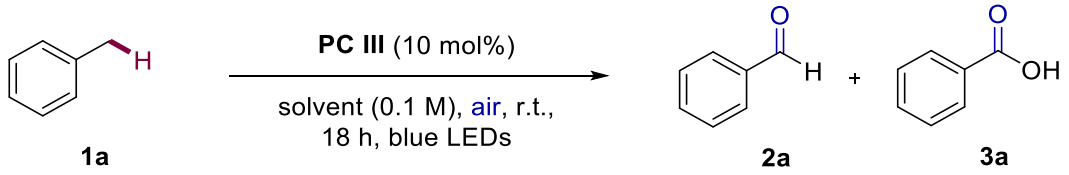




Entry	Catalyst	Yield **2a** (%)[a]	Yield **3a** (%)[a]
1	**PC I**	15	0
2	**PC II**	7	0
3	**PC III**	36	Traces
4	**PC IV**	1	0
5	**4-CzIPN**	0	0
6	**Na_2-Eosin Y**[b]	0	0
7	**H_2-Eosin Y**	20	0
8	**Rose bengal**[b]	0	0
9	**$Ru(bpy)_3(PF_6)_2$**	0	0
10	**$Ir[dF(CF_3)ppy]_2(dtbpy)PF_6$**	0	0
11	**F_2Irpic**	0	0
12[c]	-	0	0

Conditions: All reactions were performed in a 0.1 mmol scale in closed vials under air atmosphere. The vials were irradiated from the bottom plane with blue LEDs (457 nm) for 18 h. [a] Yields were determined by GC-FID with *n*-hexadecane as internal standard. [b] The reaction was irradiated with a single 525 nm LED. [c] The reaction was performed with and without irradiation.

Table S2: Solvent screening.

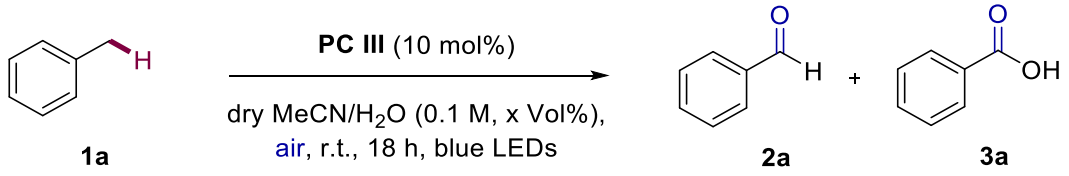


1a **2a** **3a**

Entry	Solvent	Yield **2a** (%)[a]	Yield **3a** (%)[a]
1	DCM	16	0
2	$CHCl_3$	34	Traces
3	TFE	25	0
4	HFIP	15	0
5	MeOH	0	0
6	DMF	0	0
7	DMSO	Traces	0
8	MeCN	51	5
9	THF	24	0
10[b]	MeCN	-	83

Conditions: All reactions were performed in 0.1 mmol scale with **PC III** (10 mol%) and the appropriate solvent (0.1 M) under air for 18 h. [a] Yields were determined by GC-FID with *n*-hexadecane as internal standard. [b] The reaction was performed open to air.

Table S3: Screening of the water amount.

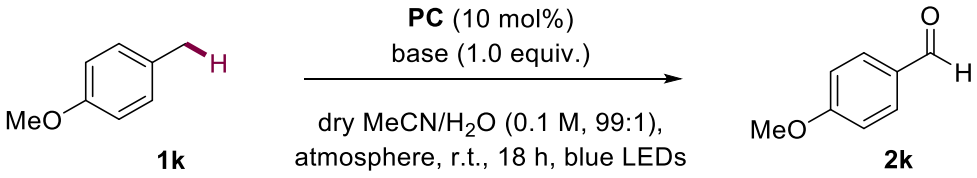


1a **2a** **3a**

Entry	H_2O (Vol%)	Yield **2a** (%)[a]	Yield **3a** (%)[a]
1	0.2	40	30
2	0.4	32	Traces
3	0.6	56	Traces
4	0.8	59	0
5	1.0	69	0
6	2.0	36	Traces
7	4.0	39	11
8	6.0	20	0
9	8.0	21	0
10	10	14	0

Conditions: All reactions were performed in 0.1 mmol scale with **PC III** (10 mol%) in MeCN/H_2O (0.1M) under air for 18 h. [a] Yields were calculated by calibration of GC-FID with *n*-hexadecane as internal standard.

Table S4: Screening of the water amount.



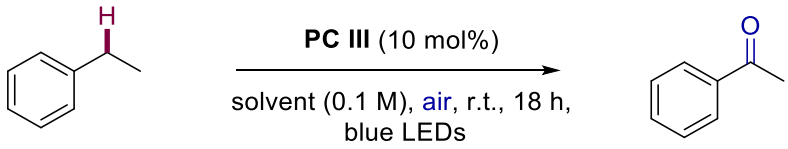
1k **2k**

Entry	Catalyst	Atmosphere	Base	Yield (%)[a]
1	**PC III**	Air	2,6-Di-*tert*-butyl-4-methylpyridine	0
2	**PC III**	Air	K_2HPO_4	Traces
3	**PC III**	Air	KH_2PO_4	0
4	**PC III**	Air	2,6-lutidine	Traces
5	**PC IV**	Air	K_2HPO_4	Traces
6	**PC IV**	Air	KH_2PO_4	0
7	**PC IV**	Air	2,6-lutidine	Traces
8	**PC IV**	Air	NaOAc	0
9	**PC IV**	Air	K_2CO_3	0
10	**PC IV**	Air	NaOH	0
11	**PC I**	O_2	2,6-lutidine	32%[b]
12	**PC III**	O_2	2,6-lutidine	Traces
13	**PC IV**	O_2	2,6-lutidine	56%[b]

Conditions: All reactions were performed in a 0.1 mmol scale. The vials were irradiated from the bottom plane with blue LEDs for 18 h. [a] Yields were determined by GC-FID with *n*-hexadecane as internal standard. [b] Isolated yields.

Optimization of the photocatalyzed ketone synthesis

Table S5: Solvent screening for the ketone generation.

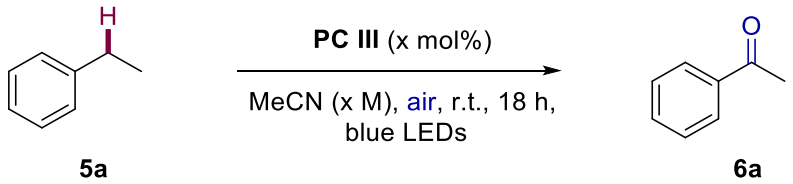


5a **6a**

Entry	Solvent	Yield (%)[a]
1	DCM	9
2	$CHCl_3$	45
3	DCE	54
4	TFE	39
5	MeCN	97
6	MeCN/H_2O (99:1)	74

Conditions: All reactions were performed in a 0.1 mmol scale. The vials were irradiated from the bottom plane with blue LEDs for 18 h. [a] Yields were determined by GC-FID with *n*-hexadecane as internal standard.

Table S6: Optimization of the concentration for the ketone generation.

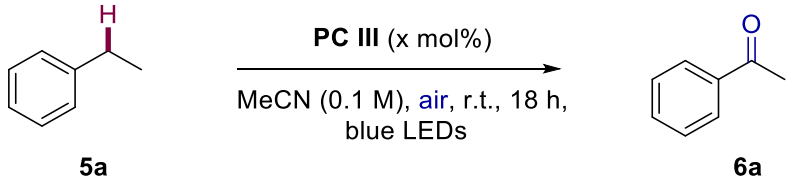


5a **6a**

Entry	Concentration (M)	Yield (%)[a]
1	0.400	57
2	0.200	62
3	0.130	53
4	0.067	58
5	0.050	30
6	0.040	34

Conditions: All reactions were performed in a 0.1 mmol scale. The vials were irradiated from the bottom plane with blue LEDs for 18 h. [a] Yields were determined by GC-FID with *n*-hexadecane as internal standard.

Table S7: Optimization of the catalyst loading.



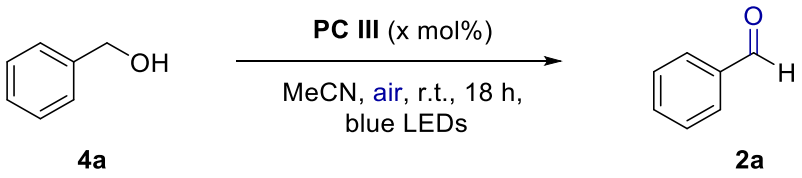
5a **6a**

Entry	Catalyst loading (mol%)	Yield (%)[a]
1	9	90
2	8	96
3	7	91
4	6	78
5	5	73

Conditions: All reactions were performed in a 0.1 mmol scale. The vials were irradiated from the bottom plane with blue LEDs for 18 h. [a] Yields were determined by GC-FID with *n*-hexadecane as internal standard.

Screening of the catalyst loading for the oxidation of benzylic alcohols

Table S8: Photocatalyzed oxidation of benzyl alcohol.



4a **2a**

Entry	Catalyst loading (mol%)	Yield (%)[a]
1	10	76
2	5.0	83
3	2.5	53
4	1.0	63
5	0.5	53

Conditions: All reactions were performed in 0.1 mmol scale under air in a closed vial. The reactions were irradiated from the bottom plane with a single 5 W blue LED at room temperature. [a] Yields were calculated by GC-FID with *n*-hexadecane as internal standard.

Calibration and representative analytical data

Table S9. Summary of the calibration data for the aldehydes.

Entry	Product	Slope	R^2
1	Benzaldehyde (**2a**)	3.002	0.9968
2	2-Methylbenzaldehyde (**2b**)	2.5275	0.9986
3	3-Methylbenzaldehyde (**2c**)	2.3229	0.9994
4	4-Methylbenzaldehyde (**2d**)	2.5445	0.9998
5	4-Fluorobenzaldehyde (**2n**)	3.755	0.9951

Representative calibration plot

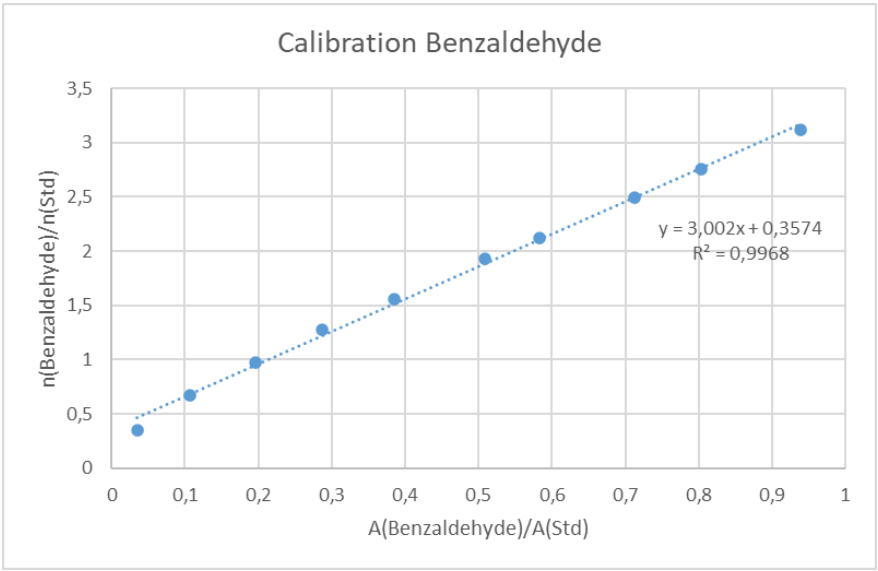


Table S10. Calibration of benzoic acid.

Entry	Product	Slope	R^2
1	Benzoic acid (**3a**)	2.9075	0.9958

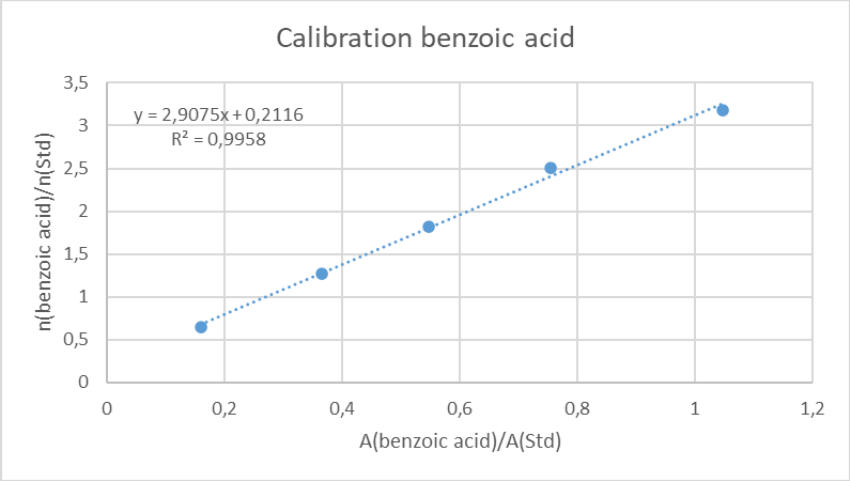
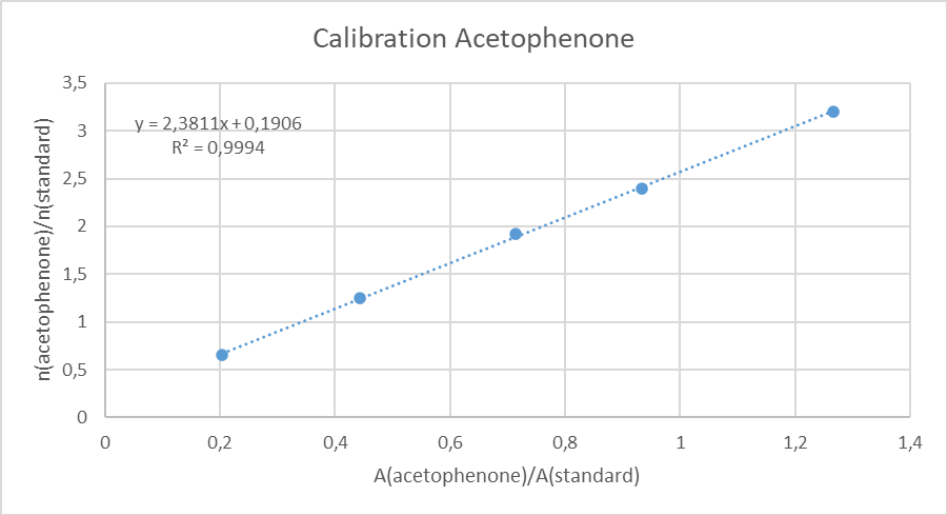


Table S11. Summary of the calibration data for the ketones.

Entry	Product	Slope	R^2
1	Acetophenone (**6a**)	2.3811	0.9994
2	Propiophenone (**6b**)	2.051	0.9996
3	Valerophenone (**6c**)	1.6116	0.9998
4	Hexanophenone (**6d**)	1.418	0.999
5	Heptanophenone (**6e**)	1.3302	0.9993
6	Octanophenone (**6f**)	1.1712	0.9998

Representative calibration plot



Photocatalyzed C─H oxygenation reaction of alkyl arenes

General procedure A for the C-H oxygenation of toluene derivatives

The corresponding toluene derivative (0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) were dissolved in a mixture of dry MeCN and water (0.1 M, 99:1) or dry DCE (0.1 M) under air. The closed vial was irradiated with a single 457 nm LED from the bottom plane for 18 h and the crude mixture was analyzed by GC-FID with *n*-hexadecane as internal standard or purified by flash column chromatography.

General procedure B for the C-H oxygenation of alkylarenes

The corresponding alkylarene (0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) were dissolved in MeCN (0.1 M) under air. The reaction was irradiated with a single 457 nm LED from the bottom plane for 18 h and the crude mixture was analyzed by GC-FID with *n*-hexadecane as internal standard or purified by flash column chromatography.

General procedure C for the C-H oxygenation of benzylic alcohols

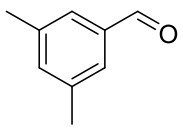
The corresponding benzylic alcohol (0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) were dissolved in MeCN (0.1 M) under air. The reaction was irradiated with a single 457 nm LED from the bottom plane for 18 h and the crude mixture was analyzed by GC-FID with *n*-hexadecane as internal standard or purified by flash column chromatography.

General procedure D for the C-H oxygenation of electron-rich substrates

The corresponding toluene derivative (0.1 mmol, 1.0 equiv.), the mesityl-acridinium salt **PC IV** (6.4 mg, 0.01 mmol, 10 mol%) and 2,6-lutidine (11.9 µL, 0.1 mmol, 1.0 equiv.) were dissolved in a mixture of dry MeCN and water (0.1 M, 99:1). In case of alkylarenes, the reaction mixture was dissolved in p.a. MeCN (0.1 M). Then, the reaction was bubbled with oxygen for 5 minutes. The closed vial was irradiated with a single 457 nm LED from the bottom plane for 18 h. After this time, the crude mixture was purified by flash column chromatography.

Analytical data of the isolated products

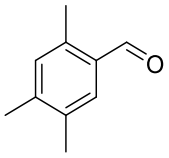
3,5-Dimethylbenzaldehyde (2e)



Following the general procedure **A**, the reaction between mesitylene (12.0 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (4.7 mg, 0.035 mmol, 35%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 7.49 (d, J = 1.8 Hz, 2H), 7.27 (s, 1H), 2.39 (d, J = 7.0 Hz, 9H). The spectrum was in accordance with the ones reported in the literature.[2]

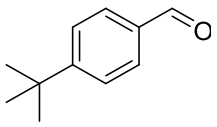
2,4,5-Trimethylbenzaldehyde (2f)



Following the general procedure **A**, the reaction between 1,2,4,5-tetramethylbenzene (13.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a white solid (2.8 mg, 0.019 mmol, 19%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.19 (s, 1H), 7.55 (s, 1H), 7.03 (s, 1H), 2.59 (s, 3H), 2.29 (s, 6H). The spectrum was in accordance with the ones reported in the literature.[3]

4-(*tert*-Butyl)benzaldehyde (2g)

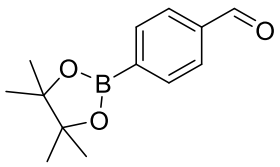


Following the general procedure **A**, the reaction between 4-(*tert*-butyl)benzaldehyde (14.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (10.7 mg, 0.065 mmol, 65%).

This product was also synthesized following the general procedure **C**, by the reaction of (4-(*tert*-butyl)phenyl)methanol (16.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) giving the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (12.3 mg, 0.076 mmol, 76%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.89 – 7.76 (m, 2H), 7.62 – 7.52 (m, 2H), 1.36 (s, 9H). The spectrum was in accordance with the ones reported in the literature.[4]

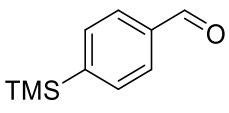
4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzaldehyde (2h)



Following the general procedure **A**, the reaction between 4,4,5,5-tetramethyl-2-(*p*-tolyl)-1,3,2-dioxaborolane (21.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a white solid (6.7 mg, 0.029 mmol, 29%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.05 (s, 1H), 7.96 (d, J = 7.9 Hz, 2H), 7.87 (d, J = 8.4 Hz, 2H), 1.36 (d, J = 1.1 Hz, 12H). The spectrum was in accordance with the ones reported in the literature.[5]

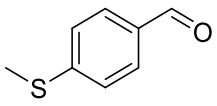
4-(Trimethylsilyl)benzaldehyde (2i)



Following the general procedure **A**, the reaction between trimethyl(*p*-tolyl)silane (16.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (10.7 mg, 0.060 mmol, 60%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.02 (s, 1H), 7.84 (d, *J* = 8.0 Hz, 2H), 7.69 (d, *J* = 8.0 Hz, 2H), 0.31 (s, 9H). The spectrum was in accordance with the ones reported in the literature.[6]

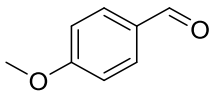
4-(Methylthio)benzaldehyde (2j)



Following the general procedure **A**, the reaction between methyl(*p*-tolyl)sulfane (13.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry DCE (0.1 M) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (7.4 mg, 0.049 mmol, 49%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.80 – 7.74 (m, 2H), 7.36 – 7.30 (m, 2H), 2.54 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[7]

4-Methoxybenzaldehyde (2k)

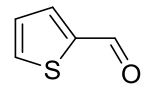


Following the general procedure **D**, the reaction between 1-methoxy-4-methylbenzene (12.2 mg, 0.1 mmol, 1.0 equiv.), 2,6-lutidine (10.3 mg, 0.1 mmol, 1.0 equiv.) and the acridinium salt **PC IV** (6.4 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a white solid (7.6 mg, 0.056 mmol, 56%).

This product was also synthesized following the general procedure **C**, by the reaction of (4-methoxyphenyl)methanol (13.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) in MeCN (0.1 M) giving the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a white solid (8.6 mg, 0.063 mmol, 63%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.89 (s, 1H), 7.87 – 7.80 (m, 2H), 7.05 – 6.92 (m, 2H), 3.89 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[8]

Thiophene-2-carbaldehyde (2l)

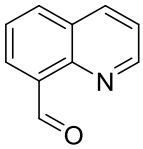


Following the general procedure **A**, the reaction between 2-methylthiophene (9.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (5.3 mg, 0.047 mmol, 47%).

This product was also synthesized following the general procedure **C**, by the reaction of thiophen-2-ylmethanol (11.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) in MeCN (0.1 M) giving the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (7.8 mg, 0.070 mmol, 70%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.95 (s, 1H), 7.81 – 7.75 (m, 2H), 7.25 – 7.20 (m, 1H). The spectrum was in accordance with the ones reported in the literature.[9]

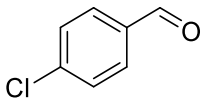
Quinoline-8-carbaldehyde (2m)



Following the general procedure **A**, the reaction between 8-methylquinoline (14.3 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a beige solid (7.8 mg, 0.050 mmol, 50%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 11.47 (s, 1H), 9.07 (dd, *J* = 4.2, 1.8 Hz, 1H), 8.35 (dd, *J* = 7.2, 1.5 Hz, 1H), 8.27 (dd, *J* = 8.4, 1.8 Hz, 1H), 8.11 (dd, *J* = 8.2, 1.6 Hz, 1H), 7.70 (t, *J* = 7.7 Hz, 1H), 7.54 (dd, *J* = 8.4, 4.2 Hz, 1H). The spectrum was in accordance with the ones reported in the literature.[10]

4-Chlorobenzaldehyde (2o)

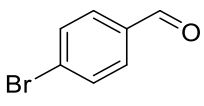


Following the general procedure **A**, the reaction between 1-chloro-4-methylbenzene (12.6 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (5.0 mg, 0.036 mmol, 36%).

This product was also synthesized following the general procedure **C**, by the reaction of (4-chlorophenyl)methanol (14.2 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) in MeCN (0.1 M) giving the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (13.0 mg, 0.093 mmol, 93%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.80 – 7.73 (m, 2H), 7.49 – 7.42 (m, 2H). The spectrum was in accordance with the ones reported in the literature.[9]

4-Bromobenzaldehyde (2p)

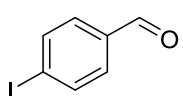


Following the general procedure **A**, the reaction between 1-bromo-4-methylbenzene (16.9 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry DCE (0.1 M) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (6.4 mg, 0.035 mmol, 35%).

This product was also synthesized following the general procedure **C**, by the reaction of (4-bromophenyl)methanol (18.5 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) in MeCN (0.1 M) giving the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (14.1 mg, 0.077 mmol, 77%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.98 (s, 1H), 7.77 – 7.73 (m, 2H), 7.71 – 7.67 (m, 2H). The spectrum was in accordance with the ones reported in the literature.[9]

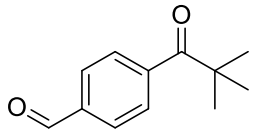
4-Iodobenzaldehyde (2q)



Following the general procedure **C**, the reaction between (4-iodophenyl)methanol (23.3 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (2.9 mg, 0.005 mmol, 5 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a white solid (13.4 mg, 0.058 mmol, 58%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.92 (s, 1H), 7.80 – 7.73 (m, 2H), 7.49 – 7.42 (m, 2H). The spectrum was in accordance with the ones reported in the literature.[11]

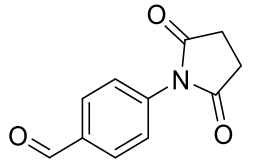
4-Pivaloylbenzaldehyde (2s)



Following the general procedure **A**, the reaction between methyl 2,2-dimethyl-1-(*p*-tolyl)propan-1-one (17.6 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a yellow oil (13.6 mg, 0.072 mmol, 72%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.92 (d, J = 8.3 Hz, 2H), 7.73 (d, J = 8.3 Hz, 2H), 1.34 (s, 9H). The spectrum was in accordance with the ones reported in the literature.[12]

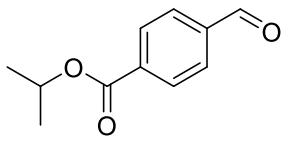
4-(2,5-Dioxopyrrolidin-1-yl)benzaldehyde (2t)



Following the general procedure **A**, the reaction between 1-(*p*-tolyl)pyrrolidine-2,5-dione (18.9 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (diethyl ether) as a white solid (13.6 mg, 0.067 mmol, 67%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.05 (s, 1H), 8.03 – 7.97 (m, 2H), 7.57 – 7.53 (m, 2H), 2.94 (s, 4H). **^{13}C-NMR** (101 MHz, $CDCl_3$) δ 191.3, 175.7, 137.2, 135.9, 130.5, 126.9, 28.6. **HRMS** (ESI-MS) mass calc. for $C_{12}H_{13}O_4NNa$ [M+MeOH+Na^+]: *m/z* = 258.0737, found: *m/z* = 258.0736.

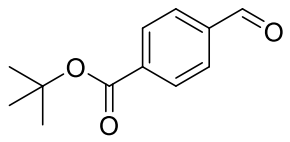
Isopropyl 4-formylbenzoat (2u)



Following the general procedure **A**, the reaction between isopropyl 4-methylbenzoate (17.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow solid (10.0 mg, 0.052 mmol, 52%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.19 (d, J = 8.3 Hz, 2H), 7.95 (d, J = 8.3 Hz, 2H), 5.28 (hept, J = 6.2 Hz, 1H), 1.39 (d, J = 6.3 Hz, 6H). The spectrum was in accordance with the ones reported in the literature.[13]

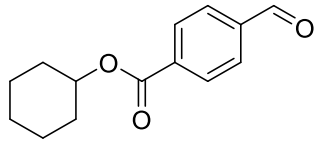
***tert*-Butyl 4-formylbenzoate (2v)**



Following the general procedure **A**, the reaction between methyl *tert*-butyl 4-methylbenzoate (19.2 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (14.1 mg, 0.069 mmol, 69%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.09 (s, 1H), 8.14 (d, *J* = 8.2 Hz, 2H), 7.93 (d, *J* = 8.7 Hz, 1H), 1.61 (s, 9H). The spectrum was in accordance with the ones reported in the literature.[14]

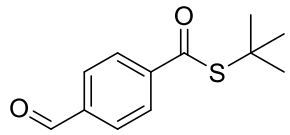
Cyclohexyl 4-formylbenzoate (2w)



Following the general procedure **A**, the reaction between methyl cyclohexyl 4-methylbenzoate (21.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a pale yellow oil (16.0 mg, 0.069 mmol, 69%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.20 (d, *J* = 8.3 Hz, 2H), 7.95 (d, *J* = 8.4 Hz, 2H), 5.10 – 5.01 (m, 1H), 2.02 – 1.91 (m, 2H), 1.86 – 1.75 (m, 2H), 1.68 – 1.54 (m, 4H), 1.52 – 1.28 (m, 2H). **^{13}C-NMR** (100 MHz, $CDCl_3$) δ 191.9, 165.1, 139.1, 136.2, 130.3, 129.6, 74.1, 31.7, 25.5, 23.8. **HRMS** (ESI-MS) mass calc. for $C_{14}H_{16}O_3Na$ [M+Na^+]: *m/z* = 255.1002, found: *m/z* = 255.0993.

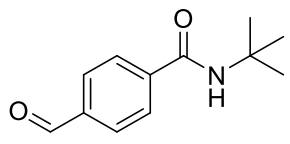
***S*-(*tert*-Butyl) 4-formylbenzothioate (2x)**



Following the general procedure **A**, the reaction between methyl *S*-(*tert*-butyl) 4-methylbenzothioate (20.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a yellow oil (19.7 mg, 0.089 mmol, 89%).

^{1}H-NMR (500 MHz, $CDCl_3$) δ 10.08 (s, 1H), 8.06 (d, *J* = 8.4 Hz, 2H), 7.93 (d, *J* = 8.2 Hz, 2H), 1.60 (s, 9H). **^{13}C-NMR** (125 MHz, $CDCl_3$) δ 192.2, 191.7, 142.8, 139.1, 129.9, 127.7, 49.1, 30.0. **HRMS** (EI-MS) mass calc. for $C_{12}H_{14}O_2S^+$ [M^+]: *m/z* = 222.0709, found: *m/z* = 222.0704.

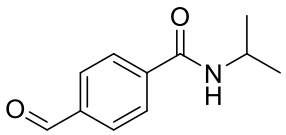
***N*-(*tert*-Butyl)-4-formylbenzamide (2y)**



Following the general procedure **A**, the reaction between *N*-(*tert*-butyl)-4-methylbenzamide (19.1 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane/ethyl acetate 9:1 to pentane/ethyl acetate 7:3) as a white solid (13.2 mg, 0.064 mmol, 64%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.06 (s, 1H), 7.93 (d, *J* = 8.5 Hz, 2H), 7.86 (d, *J* = 8.4 Hz, 2H), 5.99 (s, 1H), 1.49 (s, 9H). The spectrum was in accordance with the ones reported in the literature.[15]

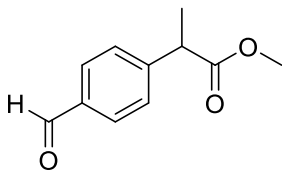
4-Formyl-*N*-isopropylbenzamide (2z)



Following the general procedure **A**, the reaction between *N*-isopropyl-4-methylbenzamide (21.9 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane/ethyl acetate 3:1) as a white solid (11.8 mg, 0.062 mmol, 62%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.07 (s, 1H), 7.94 (d, *J* = 8.6 Hz, 2H), 7.90 (d, *J* = 8.3 Hz, 2H), 6.00 (bs, 1H), 4.46 – 4.18 (m, 1H), 1.29 (d, *J* = 6.5 Hz, 6H). The spectrum was in accordance with the ones reported in the literature.[16]

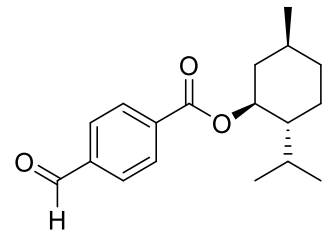
Methyl 2-(4-formylphenyl)propanoate (2aa)



Following the general procedure **B**, the reaction between methyl 2-(4-isobutyl-phenyl)propanoate (22.0 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 8:2) as a colorless oil (10.9 mg, 0.057 mmol, 57%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 9.99 (s, 1H), 7.85 (d, *J* = 8.3 Hz, 2H), 7.47 (d, *J* = 8.3 Hz, 2H), 3.81 (q, *J* = 7.2 Hz, 1H), 3.68 (s, 3H), 1.53 (d, *J* = 7.2 Hz, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$) δ 192.0, 174.2, 147.5, 135.6, 130.3, 128.4, 52.4, 45.8, 18.6. **HRMS** (ESI-MS) mass calc. for $C_{11}H_{12}O_3Na$ [M+Na^+]: *m/z* = 215.0677, found: *m/z* = 215.0679.

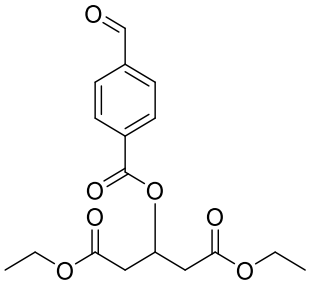
(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 4-formylbenzoate (2ab)



Following the general procedure **A**, the reaction between (1*S*,2*R*,5*S*)-2-isopropyl-5-methylcyclohexyl 4-methylbenzoate (16.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry DCE (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 10:1) as a colorless oil (28.8 mg, 0.067 mmol, 67%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.20 (d, *J* = 8.2 Hz, 2H), 7.95 (d, *J* = 8.4 Hz, 2H), 4.97 (td, *J* = 10.9, 4.4 Hz, 1H), 2.19 – 2.07 (m, 1H), 1.93 (qd, *J* = 6.9, 2.6 Hz, 1H), 1.77 – 1.72 (m, 2H), 1.67 – 1.47 (m, 2H), 1.20 – 1.04 (m, 2H), 0.95 – 0.92 (m, 7H), 0.80 (d, *J* = 6.9 Hz, 3H). The spectrum was in accordance with the ones reported in the literature.[17]

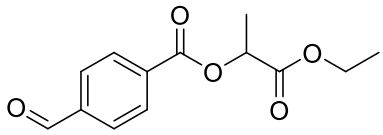
Diethyl 3-((4-formylbenzoyl)oxy)pentanedioate (2ac)



Following the general procedure **A**, the reaction between diethyl 3-((4-methylbenzoyl)oxy)pentanedioate (32.2 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a yellow oil (24.2 mg, 0.072 mmol, 72%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.24 – 8.06 (m, 2H), 8.05 – 7.85 (m, 2H), 5.80 (p, *J* = 6.3 Hz, 1H), 4.14 (qd, *J* = 7.1, 0.5 Hz, 4H), 3.10 – 2.67 (m, 4H), 1.21 (t, *J* = 7.1 Hz, 6H). **^{13}C-NMR** (100 MHz, $CDCl_3$) δ 191.7, 169.8, 164.6, 139.4, 135.0, 130.4, 129.7, 68.4, 61.1, 38.7, 14.3. **HRMS (**ESI-MS**)** mass calc. for $C_{17}H_{20}O_7Na$ [M+Na$^+$]: *m/z* = 359.1101, found: *m/z* = 359.1101.

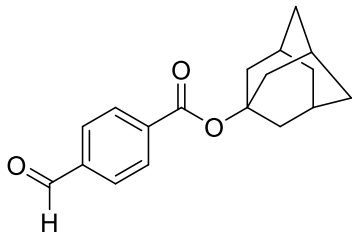
4-Ethoxy-4-oxobutan-2-yl 4-formylbenzoate (2ad)



Following the general procedure **A**, the reaction between 1-ethoxy-1-oxopropan-2-yl 4-methylbenzoate (23.6 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after preparative thin layer chromatography (pentane/diethyl ether 8:2) as a colorless oil (11.5 mg, 0.046 mmol, 46%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.11 (s, 1H), 8.25 (d, *J* = 8.2 Hz, 2H), 7.97 (d, *J* = 8.6 Hz, 2H), 5.34 (q, *J* = 7.1 Hz, 1H), 4.25 (q, *J* = 7.1 Hz, 2H), 1.65 (d, *J* = 7.1 Hz, 3H), 1.29 (t, *J* = 7.1 Hz, 3H). **^{13}C-NMR** (100 MHz, $CDCl_3$) δ 191.8, 170.6, 165.1, 139.5, 134.6, 130.6, 129.7, 69.9, 61.7, 17.2, 14.3. **HRMS (**ESI-MS**)** mass calc. for $C_{13}H_{14}O_5Na$ [M+Na$^+$]: *m/z* = 273.0733, found: *m/z* = 273.0735.

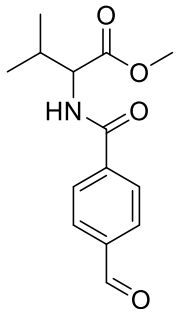
(1*S*,3*s*)-Adamantan-1-yl 4-formylbenzoate (2ae)



Following the general procedure **A**, the reaction between (1*S*,3*s*)-adamantan-1-yl 4-methylbenzoate (27.0 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 0.1 equiv.) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane/ethyl acetate 10:1) as a white solid (17.6 mg, 0.063 mmol, 63%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.09 (s, 1H), 8.13 (d, *J* = 8.3 Hz, 2H), 7.92 (d, *J* = 8.4 Hz, 2H), 2.27 – 2.23 (m, 9H), 1.77 – 1.68 (m, 6H). The spectrum was in accordance with the ones reported in the literature.[18]

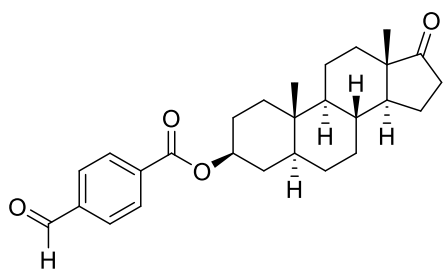
Methyl (4-formylbenzoyl)valinate (2af)



Following the general procedure **A**, the reaction between methyl (4-methylbenzoyl)valinate (24.9 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in a mixture of dry MeCN and water (0.1 M, 99:1) led to the desired product after flash column chromatography (pentane/ethyl acetate 2:1) as a white solid (16.3 mg, 0.062 mmol, 62%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.08 (s, 1H), 7.96 (s, 4H), 6.71 (d, *J* = 8.6 Hz, 1H), 4.79 (dd, *J* = 8.6, 4.8 Hz, 1H), 3.79 (s, 3H), 2.29 (hd, *J* = 6.9, 4.8 Hz, 1H), 1.01 (dd, *J* = 9.0, 6.9 Hz, 6H). The spectrum was in accordance with the ones reported in the literature.[19]

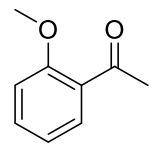
(3*R*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-Dimethyl-17-oxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-formylbenzoate (2ag)



Following the general procedure **A**, the reaction between (3*S*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-dimethyl-17-oxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-methylbenzoate (40.9 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry DCE (0.1 M) led to the desired product after flash column chromatography (pentane/ethyl acetate 5:1) as a light yellow solid (14.2 mg, 0.035 mmol, 35%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 10.10 (s, 1H), 8.19 (d, *J* = 8.1 Hz, 2H), 7.94 (d, *J* = 8.2 Hz, 2H), 4.98 (tt, *J* = 10.9, 4.9 Hz, 1H), 2.50 – 2.38 (m, 1H), 2.14 – 2.03 (m, 1H), 2.01 – 1.89 (m, 2H), 1.86 – 1.74 (m, 4H), 1.73 – 1.64 (m, 2H), 1.59 – 1.44 (m, 3H), 1.40 – 1.21 (m, 6H), 1.13 (td, *J* = 13.4, 3.7 Hz, 1H), 1.07 – 0.94 (m, 1H), 0.91 (s, 3H), 0.87 (s, 3H), 0.76 (td, *J* = 10.4, 3.9 Hz, 1H). The spectrum was in accordance with the ones reported in the literature.[20]

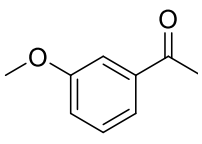
1-(2-Methoxyphenyl)ethan-1-one (6g)



Following the general procedure **D**, the reaction between 1-ethyl-2-methoxybenzene (13.6 mg, 0.1 mmol, 1.0 equiv.), the acridinium salt **PC IV** (5.1 mg, 0.008 mmol, 8 mol%) and 2,6-lutidine (10.3 mg, 0.1 mmol, 1.0 equiv.) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (5.8 mg, 0.039 mmol, 39%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 7.7, 1.9 Hz, 1H), 7.47 (ddd, *J* = 8.3, 7.3, 1.9 Hz, 1H), 7.03 – 6.95 (m, 2H), 3.92 (s, 3H), 2.62 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[21]

1-(3-Methoxyphenyl)ethan-1-one (6h)

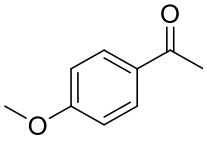


Following the general procedure **D**, the reaction between 1-ethyl-3-methoxybenzene (13.6 mg, 0.1 mmol, 1.0 equiv.), the acridinium salt **PC IV** (5.1 mg, 0.008 mmol, 8 mol%) and 2,6-lutidine (10.3 mg, 0.1 mmol, 1.0

equiv.) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (6.2 mg, 0.041 mmol, 41%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.56 – 7.52 (m, 1H), 7.50 – 7.48 (m, 1H), 7.37 (t, *J* = 7.9 Hz, 1H), 7.14 – 7.09 (m, 1H), 3.86 (s, 3H), 2.60 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[21]

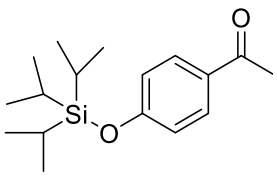
1-(4-Methoxyphenyl)ethan-1-one (6i)



Following the general procedure **D**, the reaction between 1-ethyl-4-methoxybenzene (13.6 mg, 0.1 mmol, 1.0 equiv.), the acridinium salt **PC IV** (5.1 mg, 0.008 mmol, 8 mol%) and 2,6-lutidine (10.3 mg, 0.1 mmol, 1.0 equiv.) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (8.6 mg, 0.057 mmol, 57%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.97 – 7.92 (m, 2H), 6.96 – 6.91 (m, 2H), 3.87 (s, 3H), 2.56 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[21]

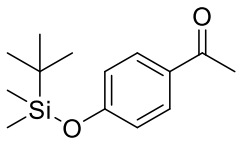
1-(4-((Triisopropylsilyl)oxy)phenyl)ethan-1-one (6j)



Following the general procedure **D**, the reaction between (4-ethylphenoxy)triisopropylsilane (27.8 mg, 0.1 mmol, 1.0 equiv.), the acridinium salt **PC IV** (5.1 mg, 0.008 mmol, 8 mol%) and 2,6-lutidine (10.3 mg, 0.1 mmol, 1.0 equiv.) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (11.0 mg, 0.038 mmol, 38%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.87 (d, *J* = 8.7 Hz, 2H), 6.90 (d, *J* = 8.7 Hz, 2H), 2.55 (s, 3H), 1.35 – 1.22 (m, 3H), 1.10 (d, *J* = 7.1 Hz, 18H). The spectrum was in accordance with the ones reported in the literature.[22]

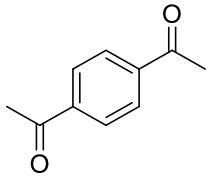
1-(4-((*tert*-Butyldimethylsilyl)oxy)phenyl)ethan-1-one (6k)



Following the general procedure **D**, the reaction between *tert*-butyl(4-ethylphenoxy)-dimethylsilane (23.6 mg, 0.1 mmol, 1.0 equiv.), the acridinium salt **PC IV** (5.1 mg, 0.008 mmol, 8 mol%) and 2,6-lutidine (10.3 mg, 0.1 mmol, 1.0 equiv.) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (15.0 mg, 0.060 mmol, 60%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.90 (d, *J* = 7.2 Hz, 2H), 6.88 (d, *J* = 7.2 Hz, 2H), 2.55 (s, 3H), 0.99 (s, 9H), 0.23 (s, 6H). The spectrum was in accordance with the ones reported in the literature.[23]

1,1'-(1,4-Phenylene)bis(ethan-1-one) (6l)

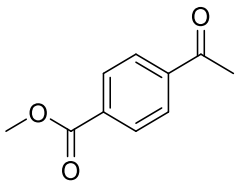


Following the general procedure **B**, the reaction between methyl 1-(4-ethylphenyl)ethan-1-one (14.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography

(pentane/diethyl ether 9:1) as a colorless solid (15.3 mg, 0.094 mmol, 94%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.03 (s, 4H), 2.65 (s, 6H). The spectrum was in accordance with the ones reported in the literature.[24]

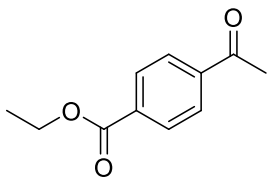
Methyl 4-acetylbenzoate (6m)



Following the general procedure **B**, the reaction between methyl 4-ethylbenzoate (16.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (8.1 mg, 0.057 mmol, 57%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.13 (d, J = 8.5 Hz, 2H), 8.01 (d, J = 8.5 Hz, 2H), 3.95 (s, 3H), 2.65 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[25]

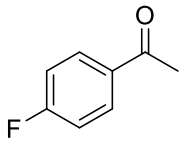
Ethyl 4-acetylbenzoate (6n)



Following the general procedure **B**, the reaction between methyl 1-ethoxy-4-ethylbenzene (17.8 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (11.9 mg, 0.062 mmol, 62%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.15 – 8.11 (m, 2H), 8.03 – 7.98 (m, 2H), 4.41 (q, J = 7.1 Hz, 2H), 2.65 (s, 3H), 1.42 (t, J = 7.1 Hz, 3H). The spectrum was in accordance with the ones reported in the literature.[26]

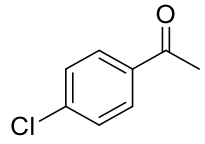
1-(4-Fluorophenyl)ethan-1-one (6o)



Following the general procedure **B**, the reaction between 1-ethyl-4-fluorobenzene (12.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (9.4 mg, 0.068 mmol, 68%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.03 – 7.93 (m, 2H), 7.17 – 7.09 (m, 2H), 2.59 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[21]

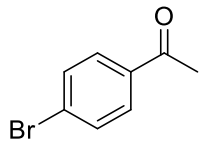
1-(4-Chlorophenyl)ethan-1-one (6p)



Following the general procedure **B**, the reaction between 1-chloro-4-ethylbenzene (14.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (12.8 mg, 0.083 mmol, 83%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.92 – 7.87 (m, 2H), 7.46 – 7.41 (m, 2H), 2.59 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[26]

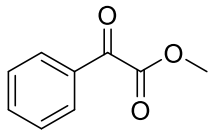
1-(4-Bromophenyl)ethan-1-one (6q)



Following the general procedure **B**, the reaction between methyl 1-bromo-4-ethylbenzene (18.5 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (18.2 mg, 0.091 mmol, 91%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.86 – 7.76 (m, 2H), 7.68 – 7.56 (m, 2H), 2.59 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[27]

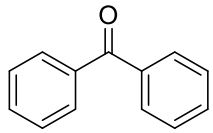
Methyl 2-oxo-2-phenylacetate (6r)



Following the general procedure **B**, the reaction between methyl 2-phenylacetate (15.0 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless oil (6.92 mg, 0.042 mmol, 42%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.13 – 7.92 (m, 2H), 7.73 – 7.63 (m, 1H), 7.52 (t, *J* = 7.8 Hz, 1H), 3.99 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[28]

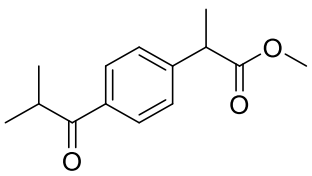
Benzophenone (6s)



Following the general procedure **B**, the reaction between diphenylmethane (16.4 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (4.7 mg, 0.008 mmol, 8 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane/diethyl ether 9:1) as a colorless solid (10.1 mg, 0.055 mmol, 55%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.75 (m, 4H), 7.63 – 7.56 (m, 2H), 7.52 – 7.45 (m, 4H). The spectrum was in accordance with the ones reported in the literature.[29]

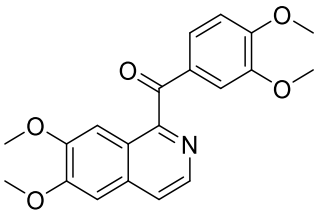
Methyl 2-(4-isobutyrylphenyl)propanoate (6t)



Following the general procedure **B**, the reaction between methyl 2-(4-isobutyl-phenyl)propanoate (22.0 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (pentane to pentane/diethyl ether 9:1) as a colorless oil (9.3 mg, 0.040 mmol, 40%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 7.92 (d, *J* = 8.4 Hz, 2H), 7.39 (d, *J* = 8.3 Hz, 2H), 3.79 (q, *J* = 7.2 Hz, 1H), 3.67 (s, 3H), 3.53 (p, *J* = 6.8 Hz, 1H), 1.52 (d, *J* = 7.2 Hz, 3H), 1.21 (d, *J* = 6.8 Hz, 6H). The spectrum was in accordance with the ones reported in the literature.[29]

(6,7-Dimethoxyisoquinolin-1-yl)(3,4-dimethoxyphenyl)methanone (6u)



Following the general procedure **D**, the reaction between 1-(3,4-dimethoxybenzyl)-6,7-dimethoxyisoquinoline (33.9 mg, 0.1 mmol, 1.0 equiv.) and the acridinium salt **PC IV** (6.4 mg, 0.01 mmol, 10 mol%) in MeCN (0.1 M) led to the desired product after flash column chromatography (ethyl acetate/pentane 10:1) as a colorless oil (10.7 mg, 0.030 mmol, 30%).

^{1}H-NMR (400 MHz, $CDCl_3$) δ 8.47 (bs, 1H), 7.74 – 7.64 (m, 2H), 7.53 (s, 1H), 7.39 (d, *J* = 8.4 Hz, 1H), 7.16 (s, 1H), 6.86 (d, *J* = 8.3 Hz, 1H), 4.07 (s, 3H), 3.97 (s, 3H), 3.96 (s, 3H), 3.95 (s, 3H). The spectrum was in accordance with the ones reported in the literature.[30]

Determination of the quantum yield φ

Determination of the photon flux at 457 nm *via* ferrioxalate actinometry

According to the procedure of Yoon,[31] the photon flux of the LEDs (λ_{max} = 457 nm) was determined by standard ferrioxalate actinometry. A 0.15 M solution of ferrioxalate was prepared by dissolving potassium ferrioxalate trihydrate (0.737 g) in H_2SO_4 (10 mL of a 0.05 M solution). A buffered solution of 1,10-phenanthroline was prepared by dissolving 1,10-phenanthroline (25 mg) and sodium acetate (5.63 g) in H_2SO_4 (25 mL of a 0.5 M solution). Both solutions were stored in the dark. To determine the photon flux of the LED, the ferrioxalate solution (1.0 mL) was placed in a cuvette and irradiated for 90 seconds at λ_{max} = 457 nm. After irradiation, the phenanthroline solution (0.175 mL) was added to the cuvette and the mixture was allowed to stir in the dark for 1 h to allow the ferrous ions to completely coordinate to the phenanthroline. The absorbance of the solution was measured at 510 nm. A non-irradiated sample was also prepared and the absorbance at 510 nm was measured. Conversion was calculated according to the following formula:

$$mol\ Fe^{2+} = \frac{\mathrm{V} \bullet \Delta\mathrm{A}(510\ \mathrm{nm})}{l \bullet \varepsilon}$$

where V is the total volume (0.001175 L) of the solution after addition of phenanthroline, ΔA is the difference in absorbance at 510 nm between the irradiated and non-irradiated solutions, l is the path length (1.00 cm), and ε is the molar absorptivity of the ferrioxalate actinometer at 510 nm(11,100 $L \cdot mol^{-1} cm^{-1}$). The photon flux can be calculated with the following equation:

$$\text{Photon flux} = \frac{\text{mol } \mathrm{Fe}^{2+}}{\Phi \bullet \mathrm{t} \bullet \mathrm{f}}$$

where Φ is the quantum yield for the ferrioxalate actinometer (0.85 at λ_{ex} = 457 nm),[32] t is the irradiation time (90 s), and f is the fraction of light absorbed at λ_{ex} = 457 nm by the ferrioxalate actinometer. This value is calculated using the following equation, where A(457 nm) is the

absorbance of the ferrioxalate solution at 457 nm. An absorption spectrum gave an A(457 nm) value of about 1.2, indicating that the fraction of absorbed light (f) is around 0.94.

$$f = 1 - 10^{-A(457nm)}$$

The photon flux was thus calculated (average of three experiments) to be 3.96×10^{-9} einsteins s^{-1}.

Determination of the reaction quantum yield

Toluene (9.2 mg, 0.1 mmol) and the imide-acridinium photocatalyst **PC III** were dissolved in a mixture of dry MeCN and water (0.1 M, 99:1) and the mixture was irradiated with blue LEDs for 15 minutes in a closed vial. The yield was determined by GC-FID with *n*-hexadecane as internal standard (2.9 mg, 0.00278 mmol, 2.8%)

$$\Phi = \frac{\text{mol of formed product}}{\text{photon flux} \bullet \text{t} \bullet \text{f}}$$

with a photon flux of 3.96×10^{-9} einsteins s^{-1}, t = 900 s and f > 0.999.

The reaction quantum yield (Φ) was determined as 0.78.

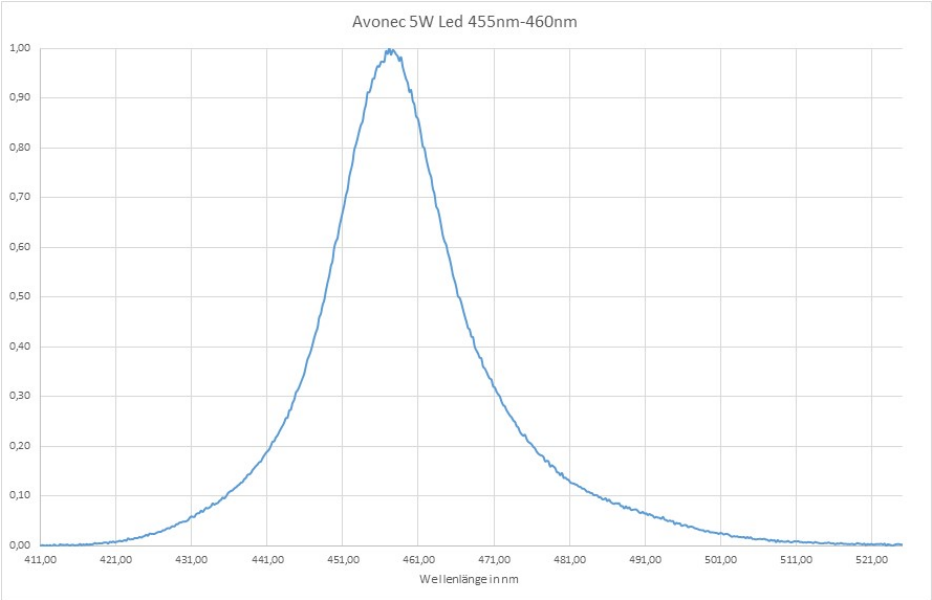


Figure S1: Emission spectrum of the applied LEDs provided by Avonec.

Quenching experiments

Emission intensities were recorded on a Jasco FP-8500 spectrofluorometer. Dry MeCN was degassed by argon bubbling for 30 minutes before using. The catalyst **PC III** was excited at 420 nm and the emission spectra were recorded between 430 and 800 nm. In a typical experiment, 0.1 mM solutions of the photocatalyst in mixture of dry MeCN and water (99:1) were prepared with the appropriate concentration of quencher in a 1.0 cm quartz cuvette and covered.

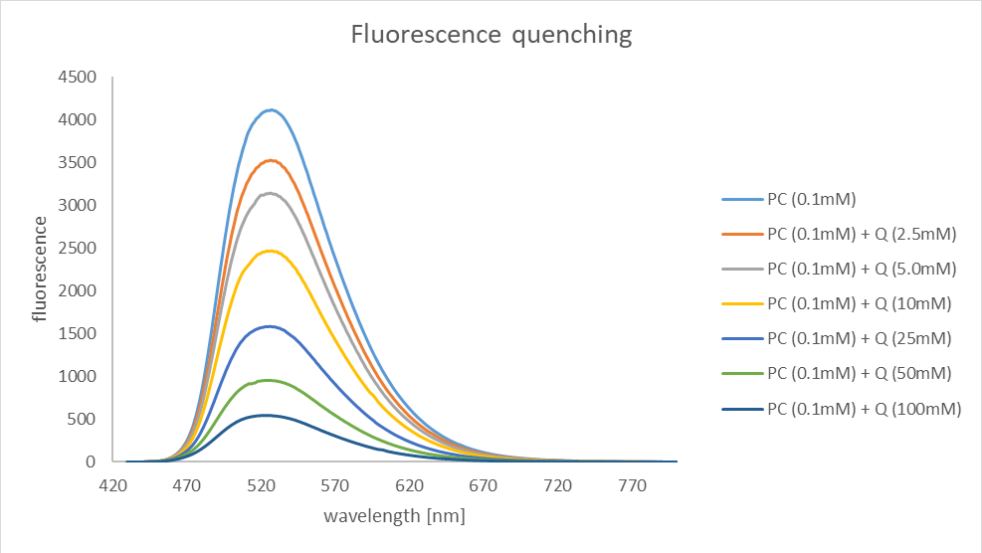
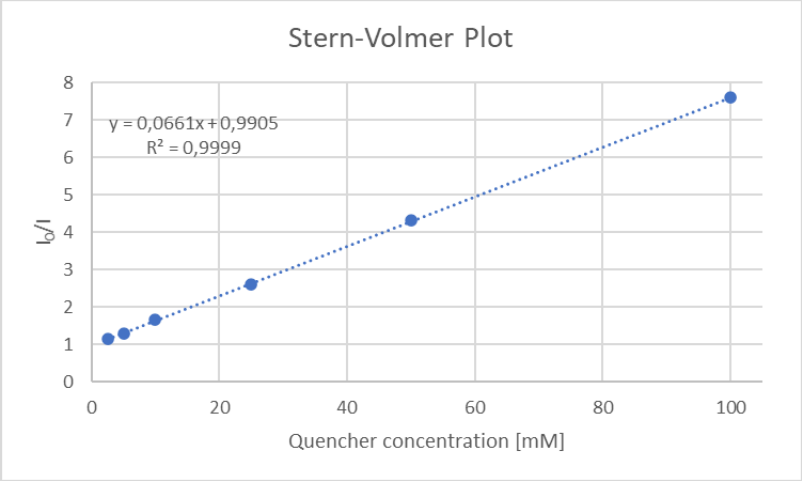


Figure S2: Fluorescence quenching of catalyst **PC III** by various amounts of toluene.

Stern-Volmer plot



Entry	Toluene **Q** (mM)	**I** @ λ_{max}	**I_0 / I**
1	-	4113.6	-
2	2.5	3520.9	1.168
3	5.0	3136.5	1.312
4	10	2467.6	1.667
5	25	1580.5	2.603
6	50	952.0	4.321
7	100	541.5	7.596

Figure S3: Stern-Volmer plot of the quenching experiments.

Detected intermediates

Bibenzyl as homocoupling product

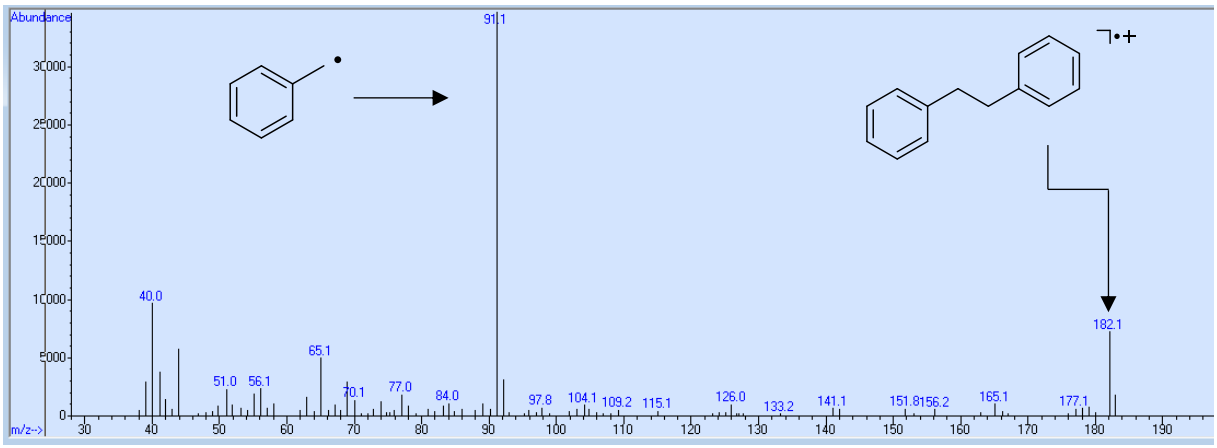


Figure S4: Observed intermediates by GC-MS.

Toluene-TEMPO adduct

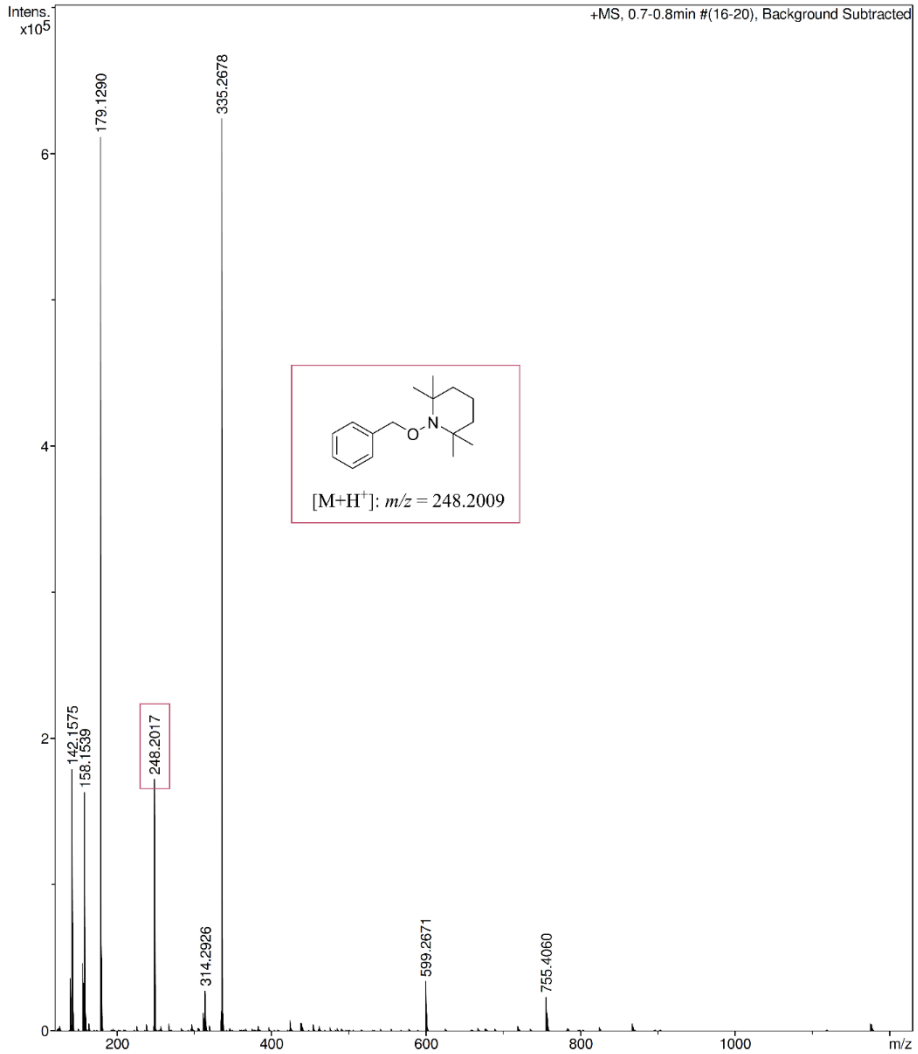


Figure S5: Observed intermediates by HRMS.

Benzyl hydroperoxide and benzyl alcohol intermediates

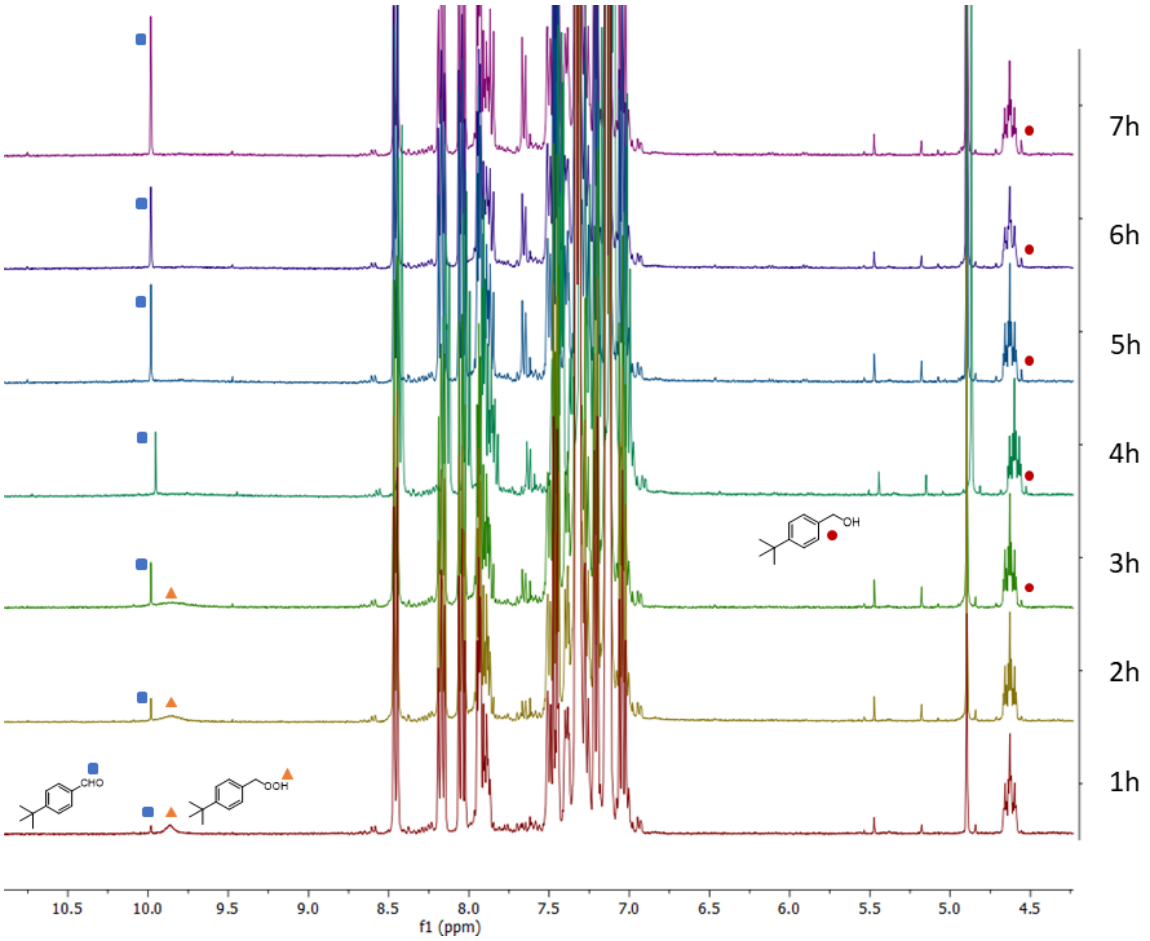


Figure S6: Obtained intermediates by reaction monitoring with ^{1}H-NMR.

Determination of the kinetic isotope effect (KIE)

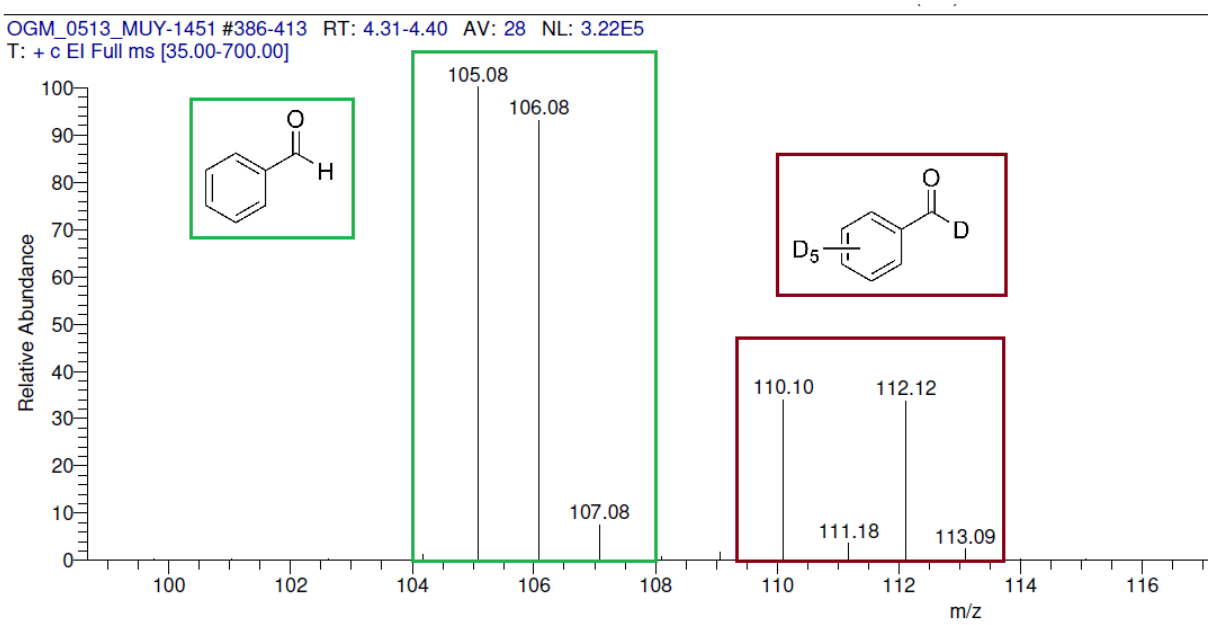


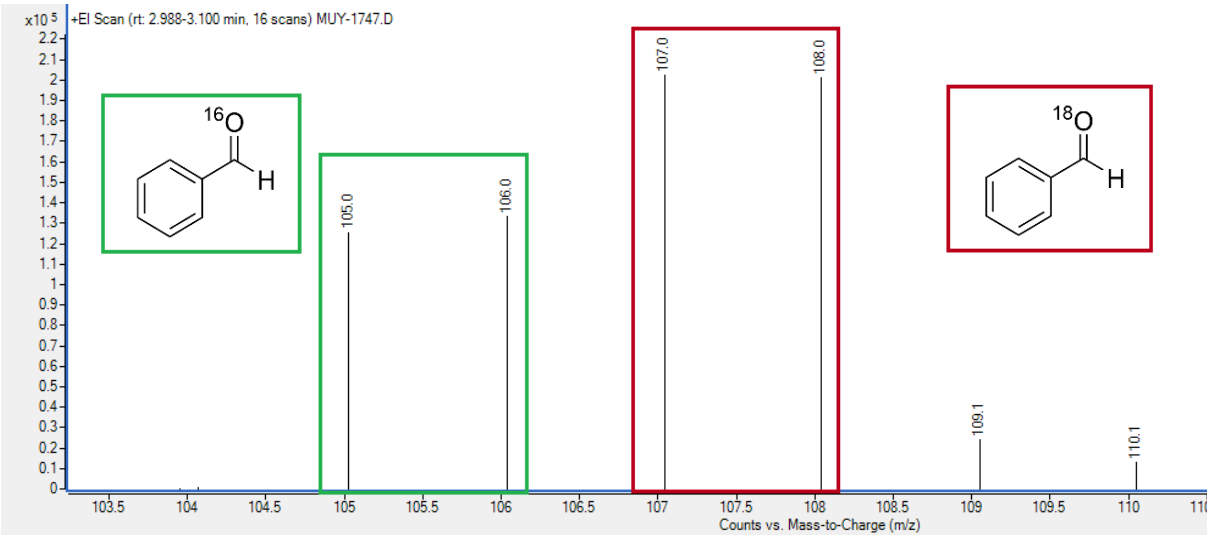
Figure S7: Estimation of the kinetic isotope effect by GC-MS

The kinetic isotope effect was estimated by comparing the sum of the isotope fragments of benzaldehyde-h_6 and benzaldehyde-d_6. Hereby, a KIE of ~2.7 could be determined.

Determination of the oxygen source

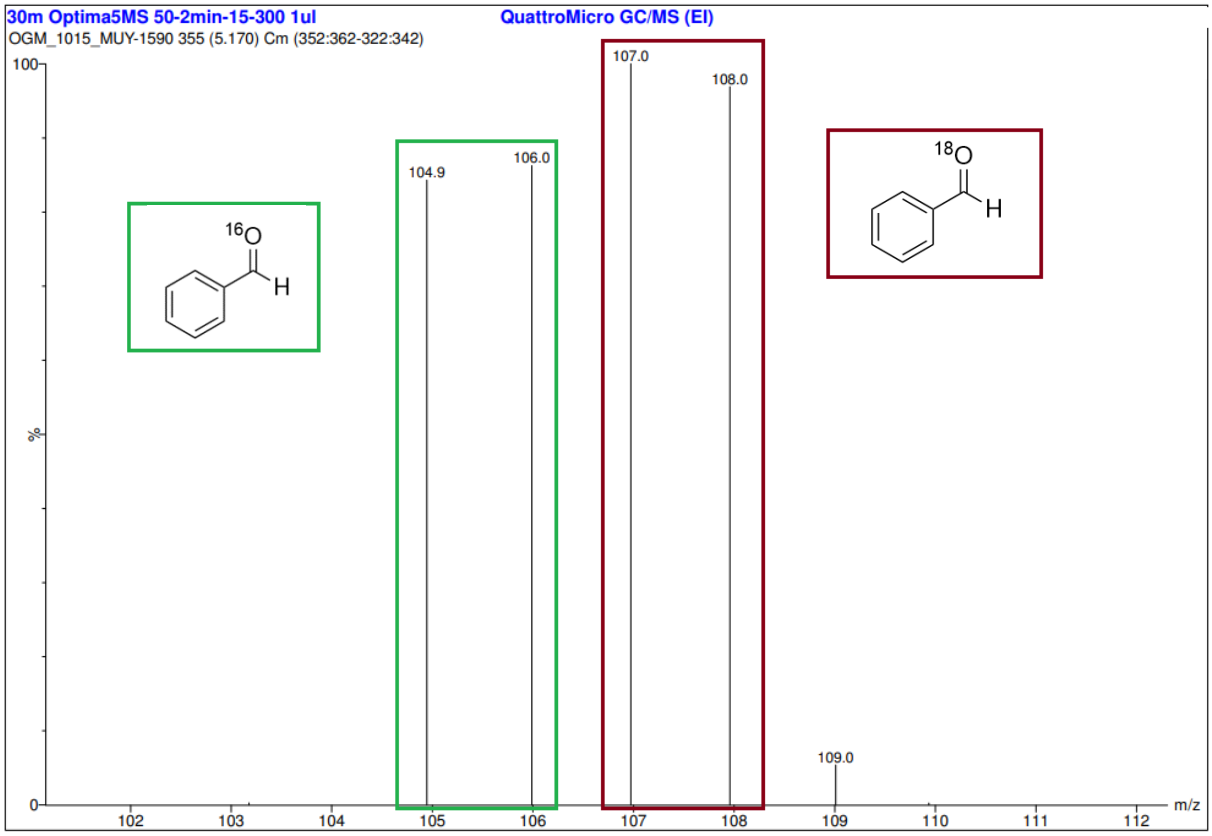
Reaction with $^{18}O_2$

The standard reaction was performed according to general procedure **A**, with toluene (9.2 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry MeCN (1 mL). The reaction was degassed by freeze pumping and the vial was backfilled with $^{18}O_2$. After two hours, the reaction was filtered over a short plug of silica and analyzed by GC-MS. The comparison of the relative amounts of benzaldehyde-^{16}O and benzaldehyde-^{18}O shows a ratio of approximately 1:1.6.



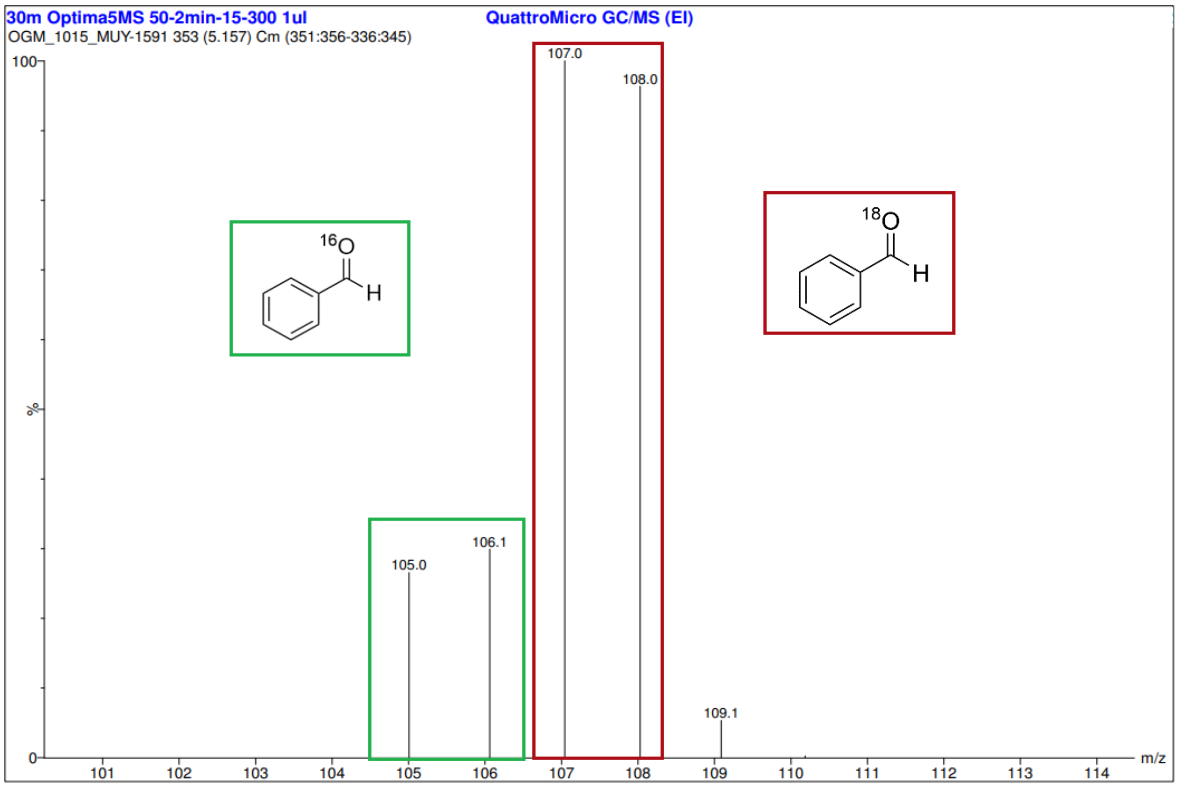
Reaction in $H_2{}^{18}O$

The standard reaction was performed according to general procedure **A**, with toluene (9.2 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry MeCN/$H_2{}^{18}O$ (1 mL, 99:1). After three hours, the reaction was filtered over a short plug of silica and analyzed by GC-MS. The comparison of the relative amounts of benzaldehyde-^{16}O and benzaldehyde-^{18}O shows a ratio of approximately 1:1.2.



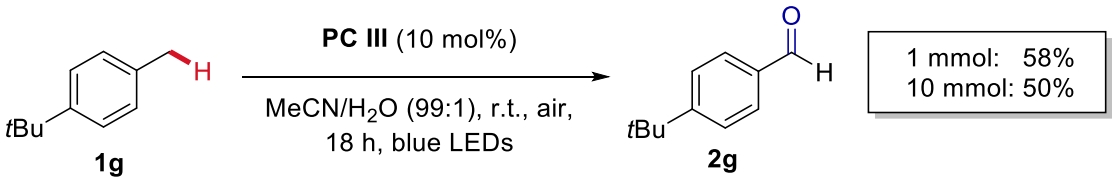
Control experiment

A control experiment was performed with benzaldehyde-^{16}O (10.6 mg, 0.1 mmol, 1.0 equiv.) and the imide-acridinium salt **PC III** (5.8 mg, 0.01 mmol, 10 mol%) in dry MeCN/$H_2{}^{18}O$ (0.1 M, 99:1). After three hours, the reaction was filtered over a short plug of silica and analyzed by GC-MS. The comparison of the relative amounts of benzaldehyde-^{16}O and benzaldehyde-^{18}O shows a ratio of approximately 1:3.5. A comparison of this result with the outcome of the standard reaction shows that the main oxygen source is molecular oxygen from air and not water.



Upscaling

The scale up was performed by irradiation of the reaction mixtures by two 30 W LEDs with additional fans to cool the reaction mixture.



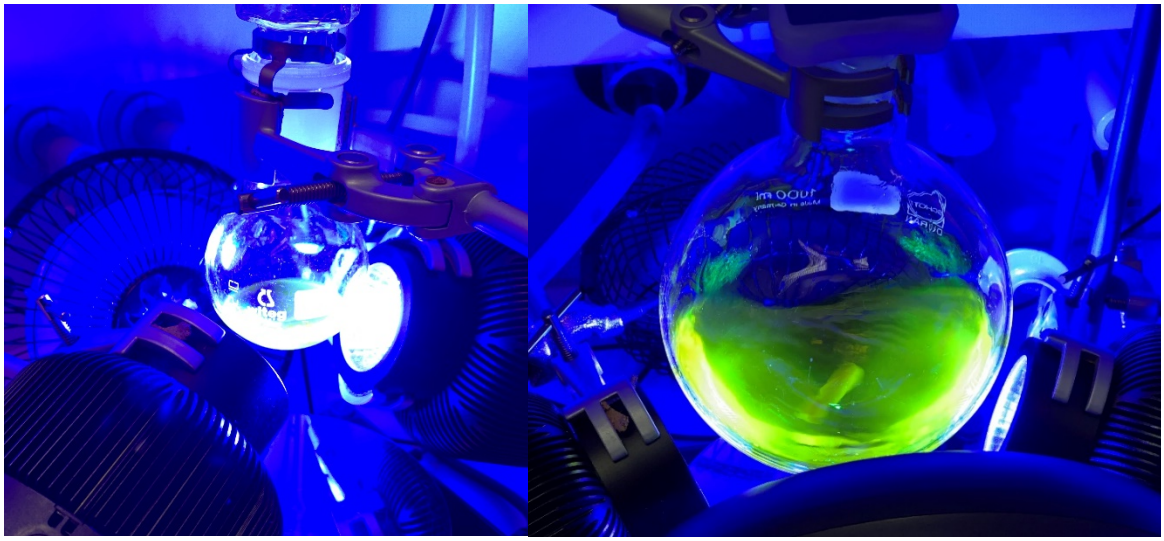
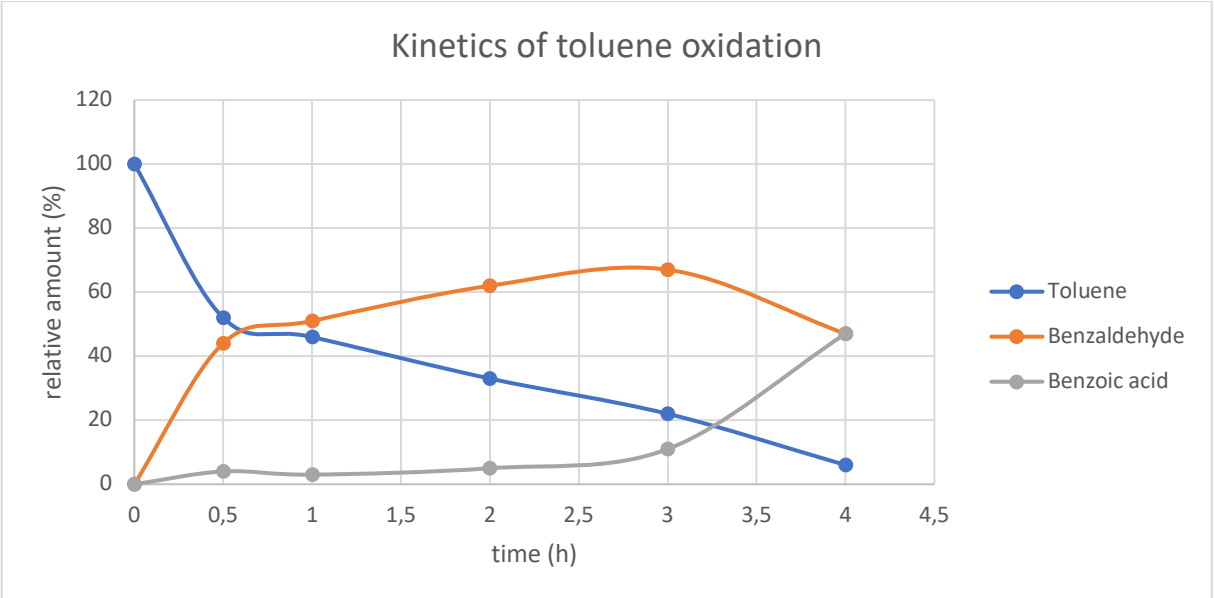


Figure S8: Scale-up to 1 mmol (left) and 10 mmol scale (right).

Reaction kinetics



To follow the reaction kinetics, the standard reaction was performed open to air in the presence of toluene (0.1 mmol, 9.2 mg, 1.0 equiv.), **PC III** (0.01 mmol, 5.8 mg, 10 mol%) in dry DCE (0.1 M). The reaction was monitored by GC-FID. It can be seen that toluene is subsequently transformed to benzaldehyde with only traces of benzoic acid until three hours. From this point, the obtained benzaldehyde is further oxidized to benzoic acid, which is most probably promoted

by reactive oxygen species in the reaction mixture or larger excess of oxygen in the reaction mixture, since the reaction is performed open to air.

Computational studies

Geometry optimizations were carried out with the Gaussian16 program[33] using B3LYP functional with def2tzvp basis set[34] taking into account the most stable ethylbenzene structure obtained in a previous study reported in the literature. [35] The electron density difference $\Delta\rho(r)$ between the optimized neutral structure and its radical cation (vertical ionized state) in the Franck-Condon type transition (plotted with isovalues of ± 0.008 e^{-}/au^{3}), as well as the rigid potential energy (PE) curve scan along the benzylic C-H bond for the deprotonation step, are calculated at DFT-b3lyp/def2tzvp level of theory (Figure S9). Additionally, for the toluene derivatives shown in Scheme 2 in the article, we used Grimme's dispersion with Becke-Johnson damping correction for all calculations. [36]

Four representative ethylbenzene-type substrates **5** with different oxidation potentials ($E_{p/2}$) were compared. Those include the two non-reactive extremes of 4-methoxyethylbenzene (**5i**), presenting a vast potential difference with the catalyst ($E_{p/2}$ = +1.52 V vs. SCE[37] vs. **PC III** with +2.40 V vs. SCE[1]), and 4-nitroethylbenzene (**5v**), the later not being able to be oxidized under the standard conditions due to its high oxidation potential. Figure S9 shows the a) oxidation step to the radical cation intermediate, for which the analysis of the electron density difference $\Delta\rho(r)$ indicates an electron density depletion in closer proximity to the benzylic position, which should facilitate the b) abstraction of H^{+} via C-H bond cleavage in the next step. It is clearly seen that, if the first single electron transfer (SET) to form the radical cation intermediate takes place, the C-H bond cleavage should involve a lower dissociation energy for electron deficient compounds (**5v**, 4-NO_2; ~ 4.0 eV) than for the electron rich, stabilized systems (**5i**, 4-OMe; ~ 4.5 eV), while presenting a similar dissociation energy of ~4.2 eV for toluene (**5a**) and **5l** (4-Ac).

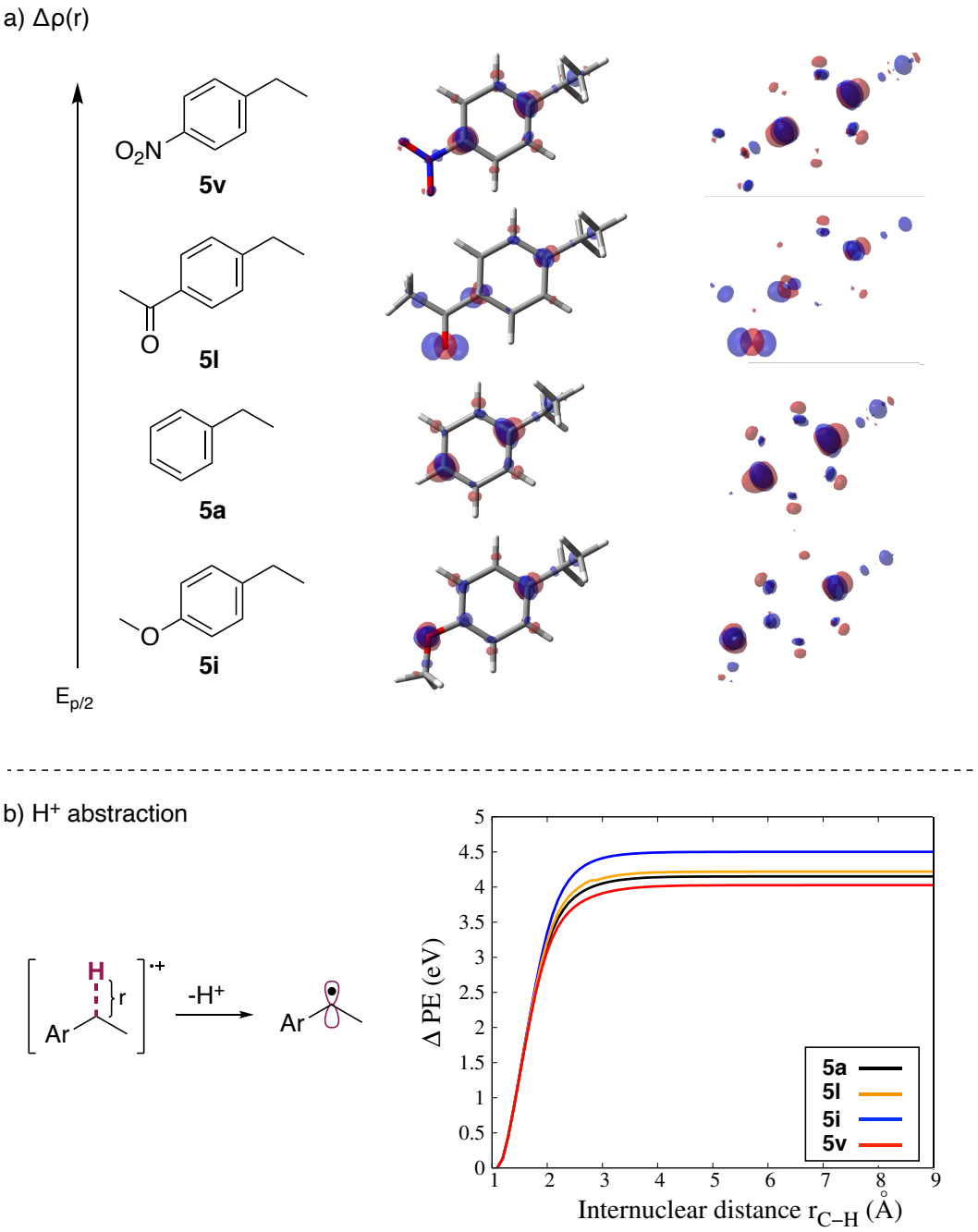


Figure S9: a) Electron density difference between neutral and oxidized ethylbenzene derivatives in a Franck–Condon type transition. Electron density depletion and accumulation of the electron density are shown in blue and red, respectively. The panels show the 3D density difference at isovalues of ±0.008 e^{-}/au^{3}. b) Rigid scan along the benzylic C-H bond for the deprotonation step from the radical cation.

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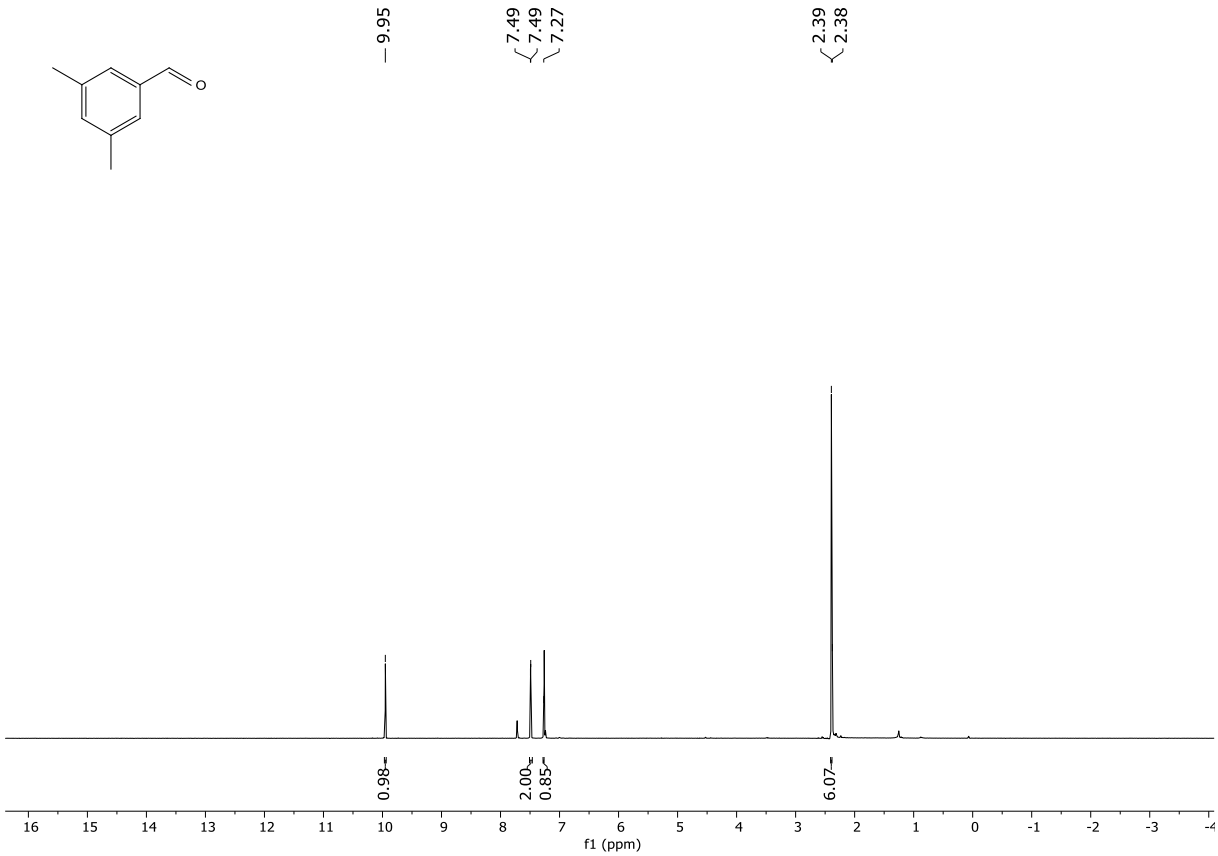
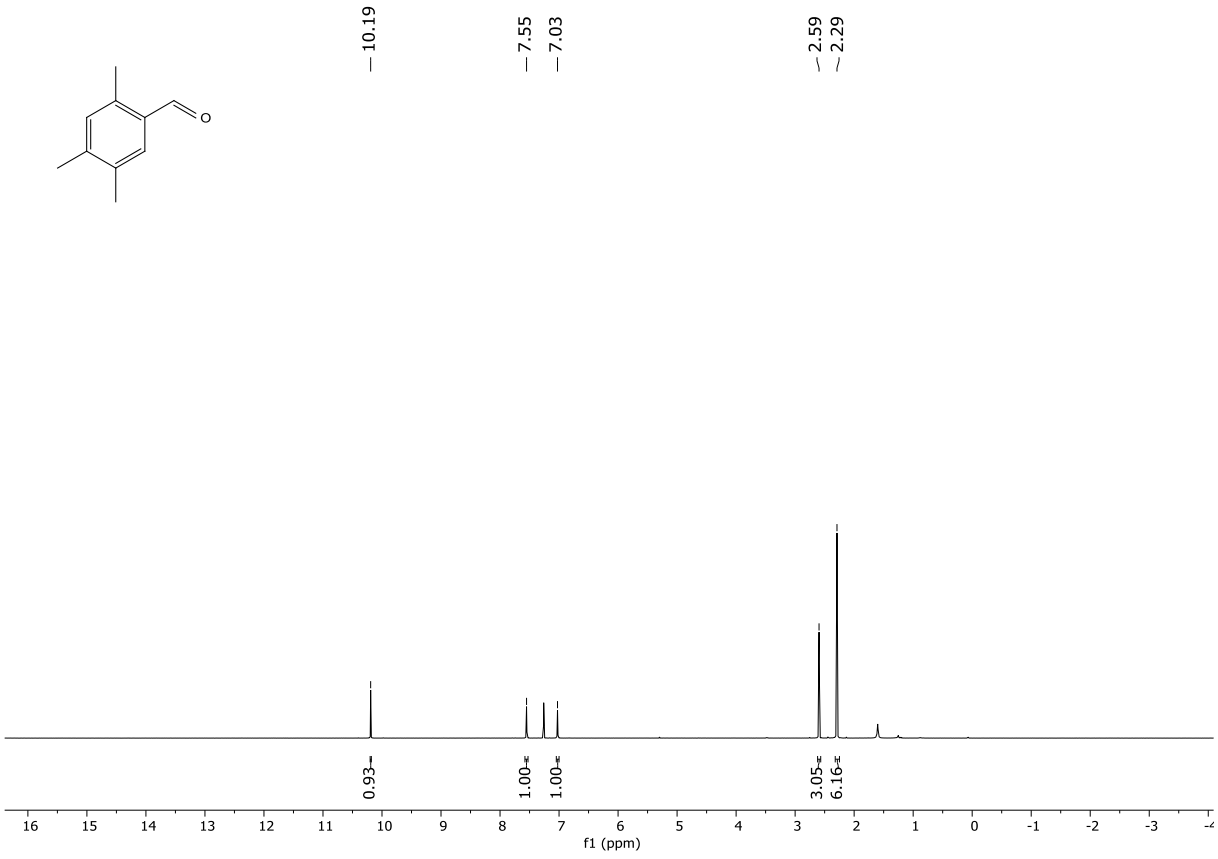
Appendix (NMR Collection)

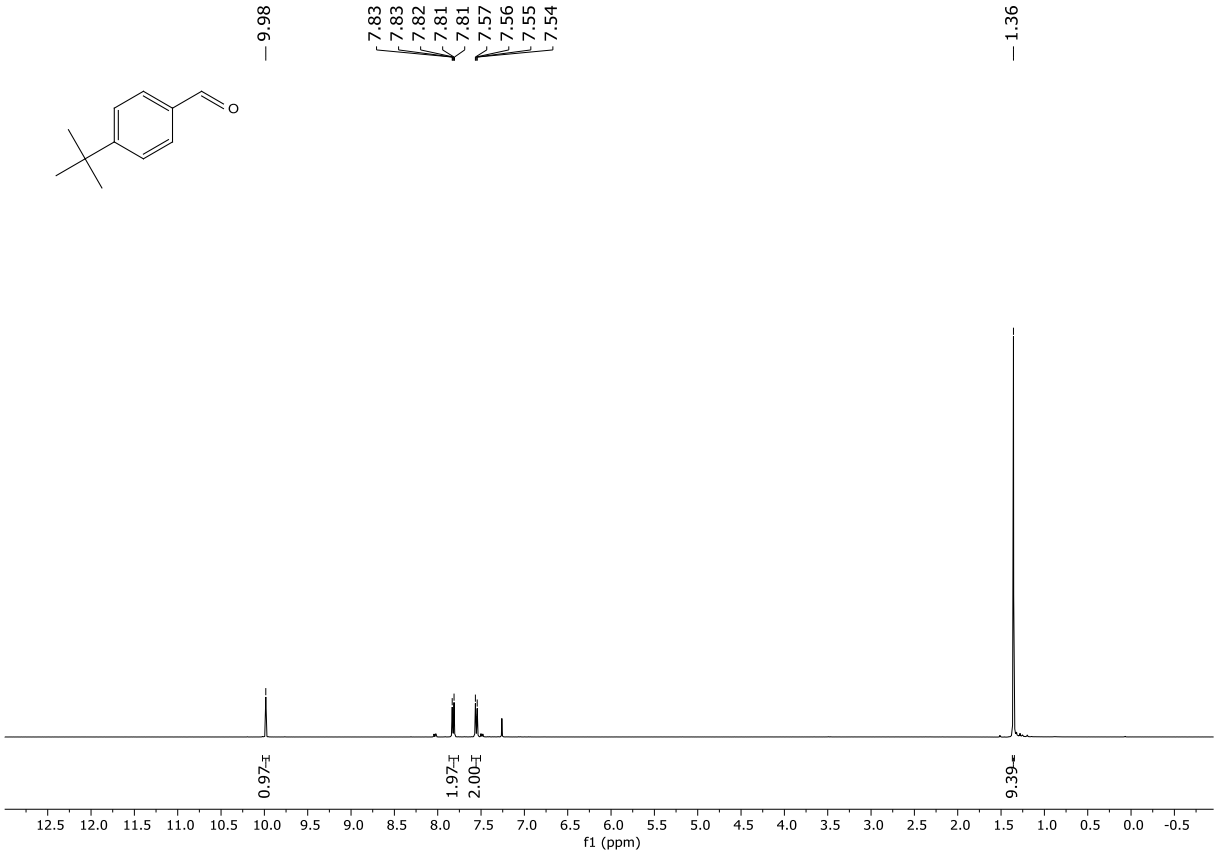
3,5-Dimethylbenzaldehyde (2e)



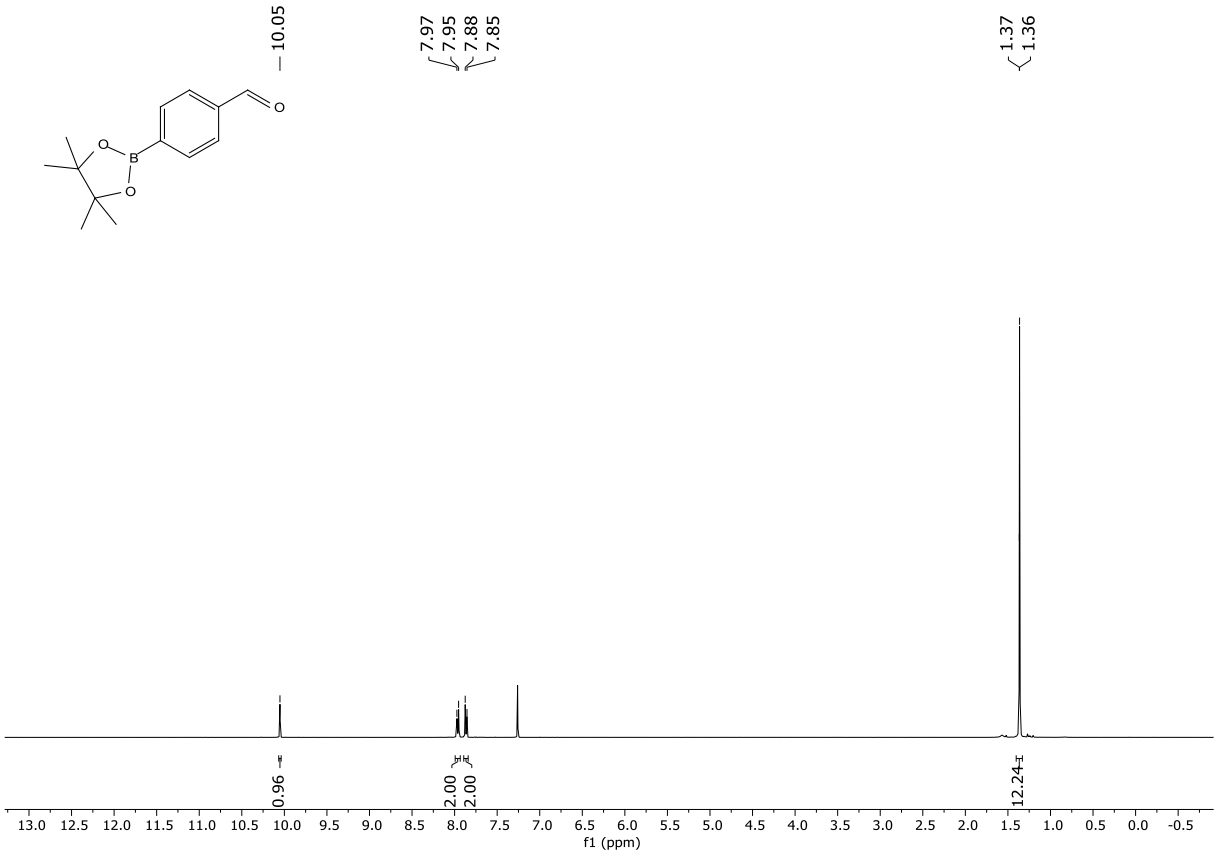
2,4,5-Trimethylbenzaldehyde (2f)



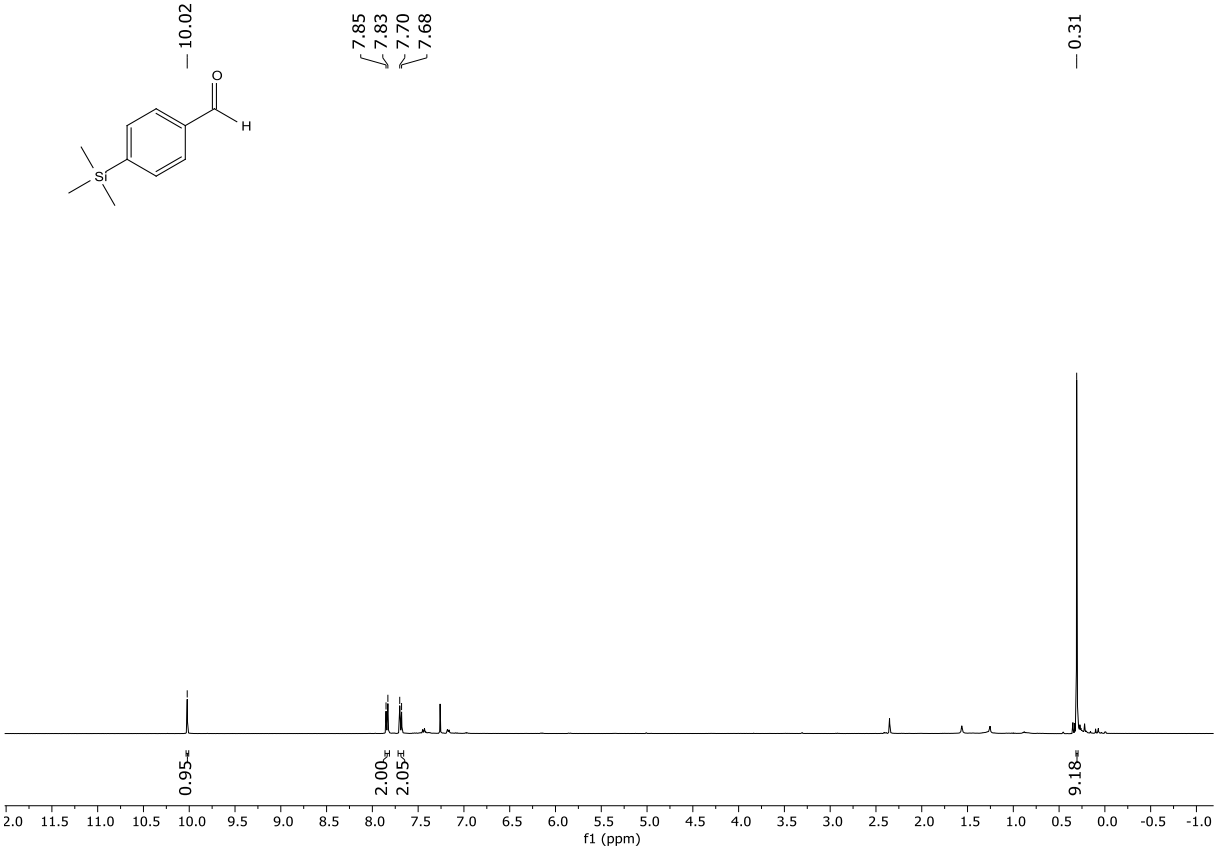
4-(*tert*-Butyl)benzaldehyde (2g)



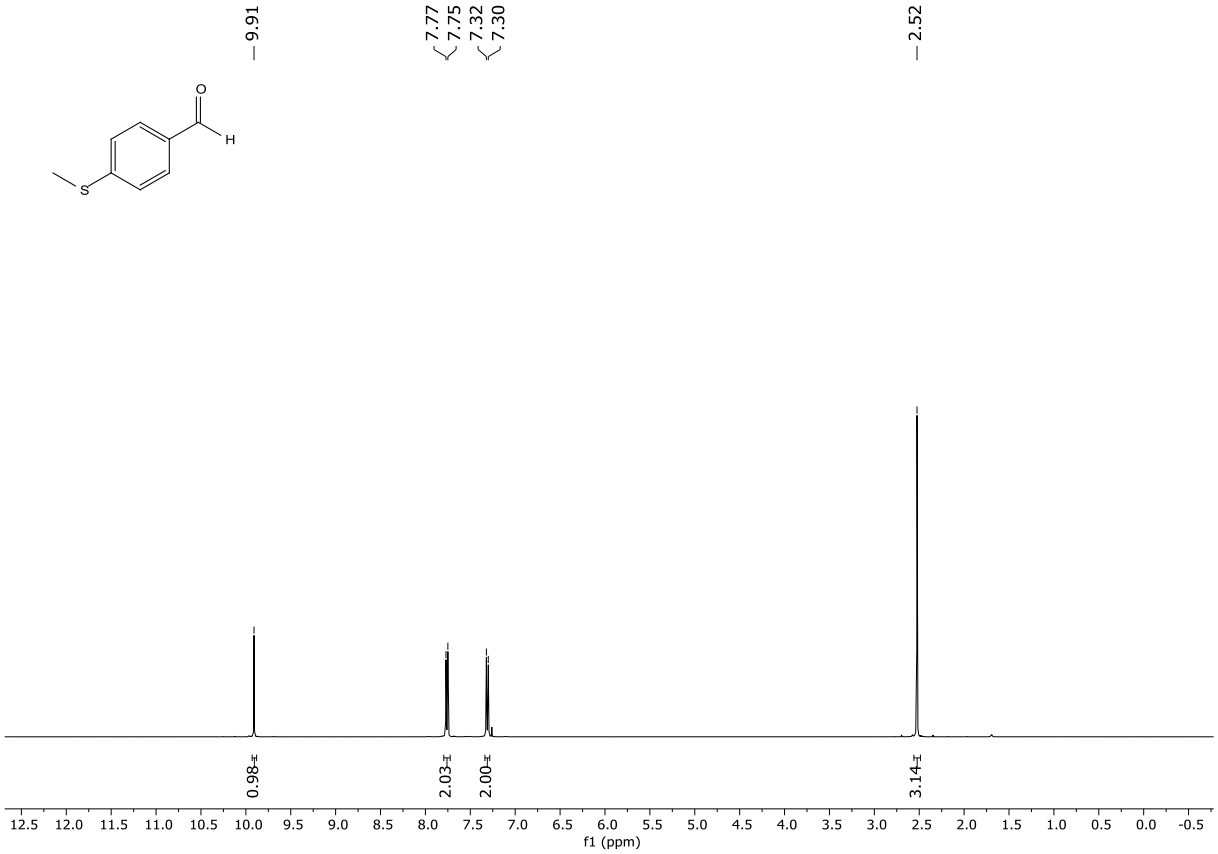
4-(4,4,5,5-Tetramethyl-1,3,2-dioxaborolan-2-yl)benzaldehyde (2h)



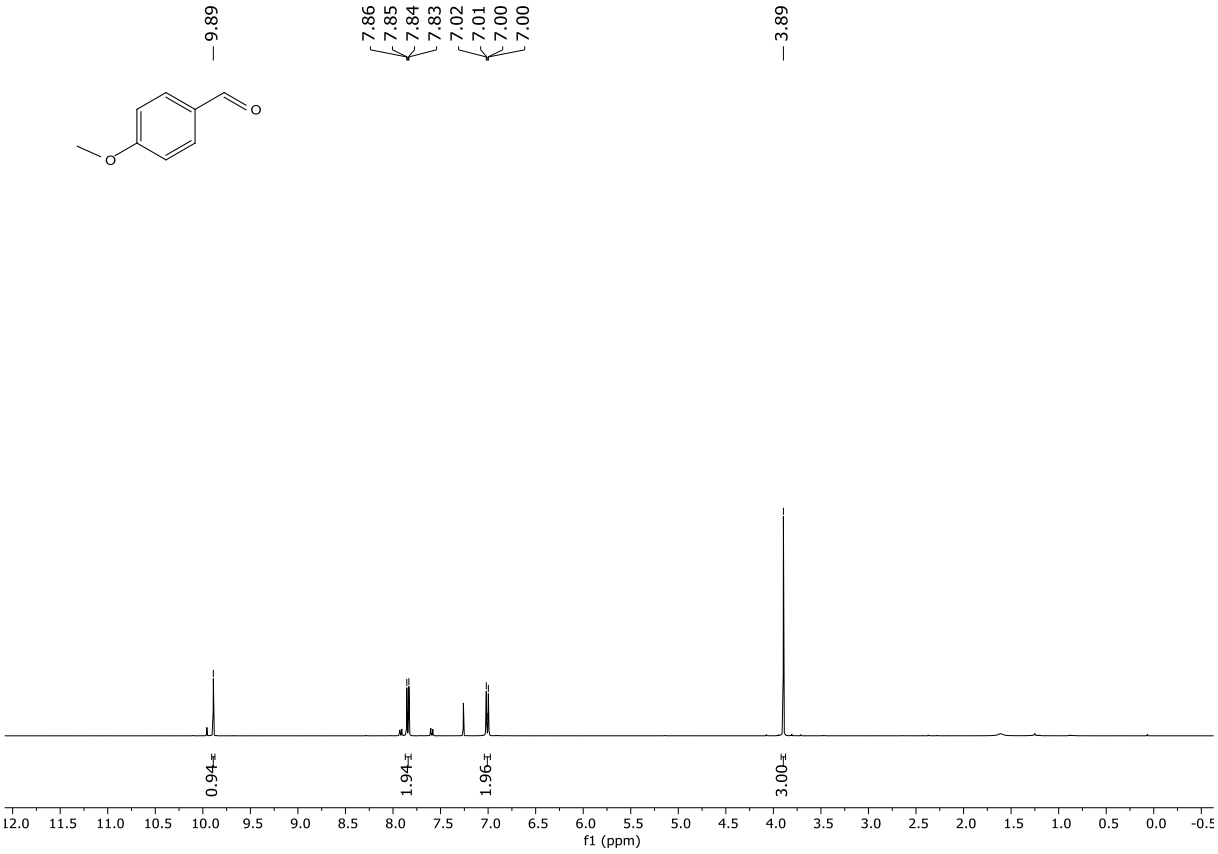
4-(Trimethylsilyl)benzaldehyde (2i)



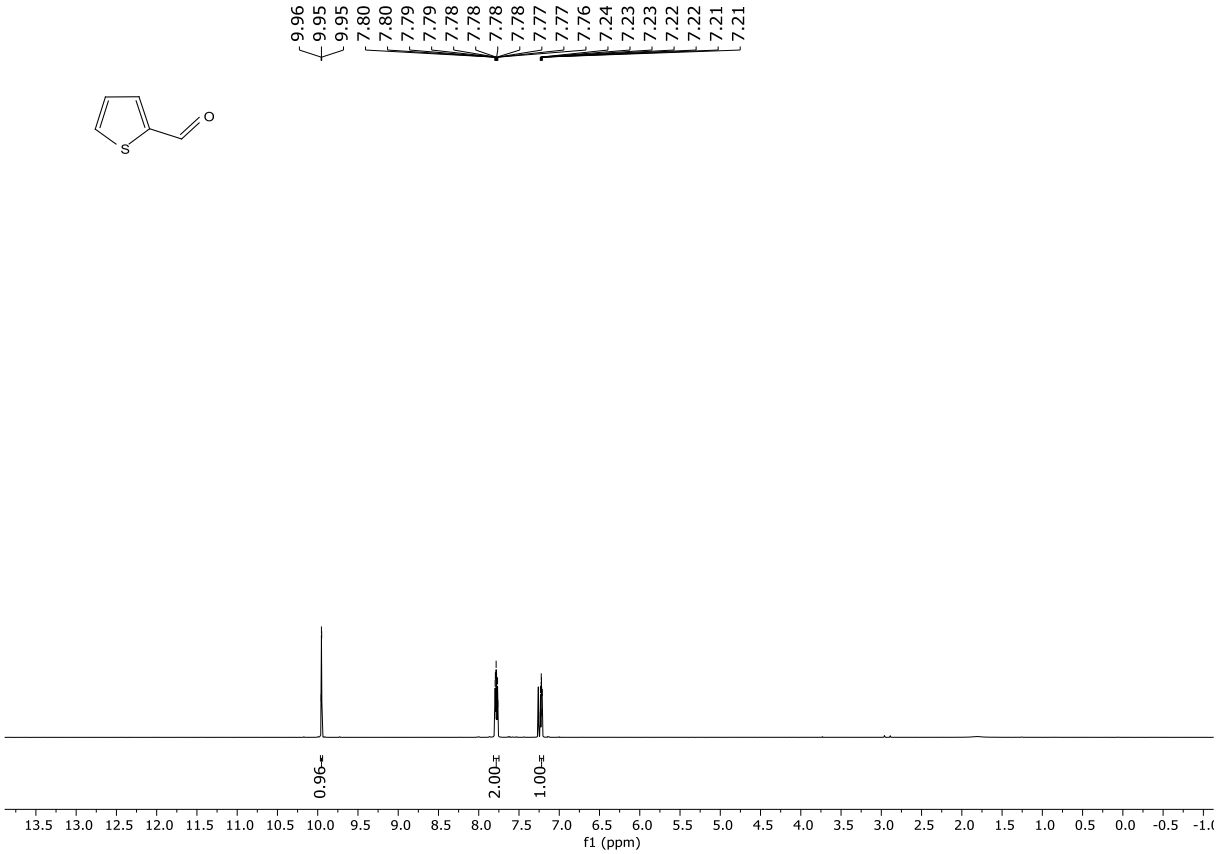
4-(Methylthio)benzaldehyde (2j)



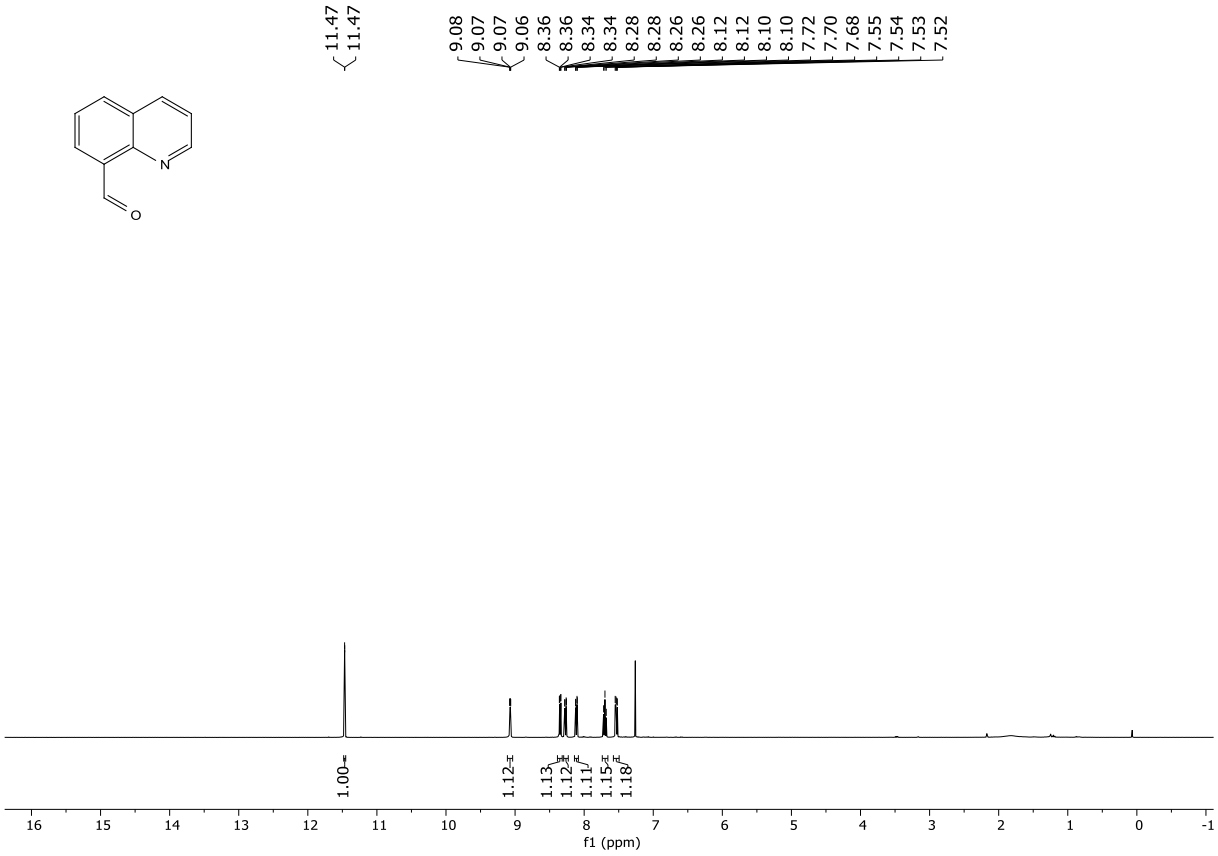
4-Methoxybenzaldehyde (2k)



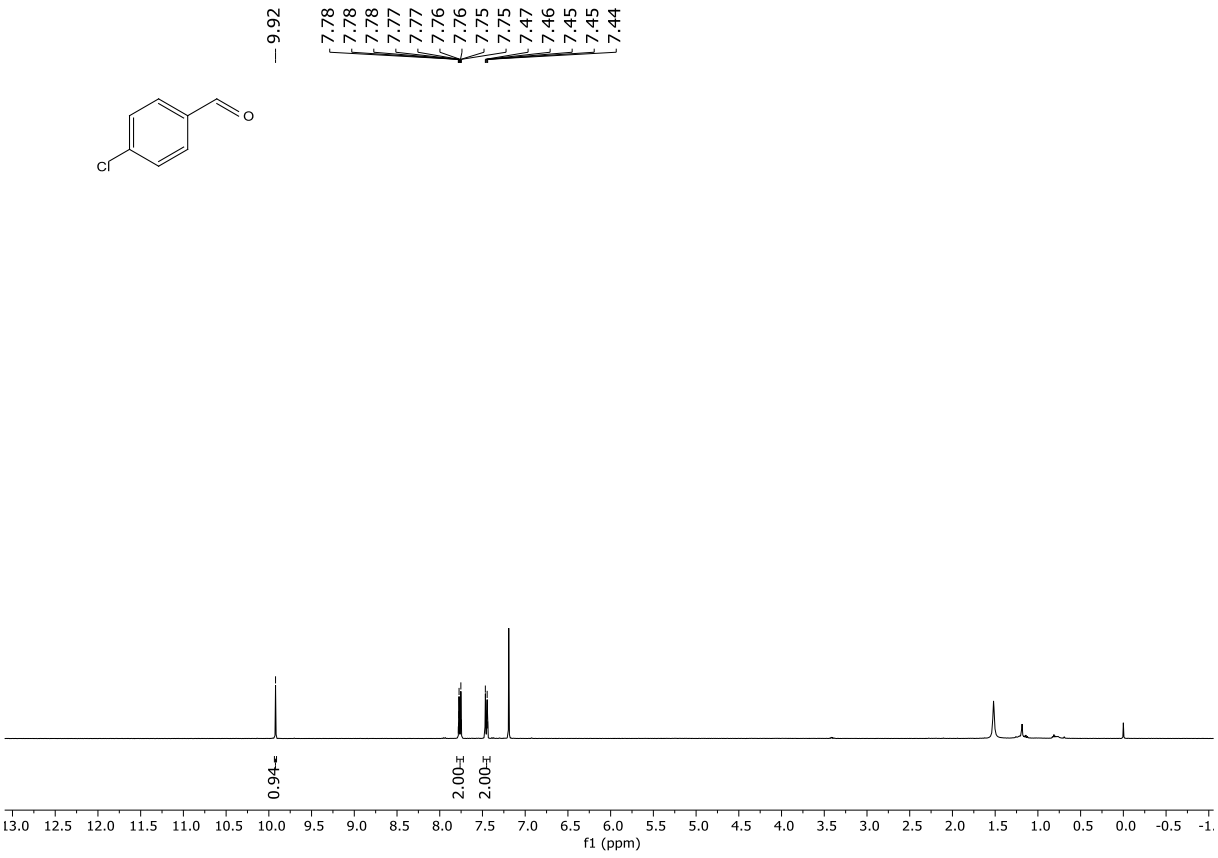
Thiophene-2-carbaldehyde (2l)



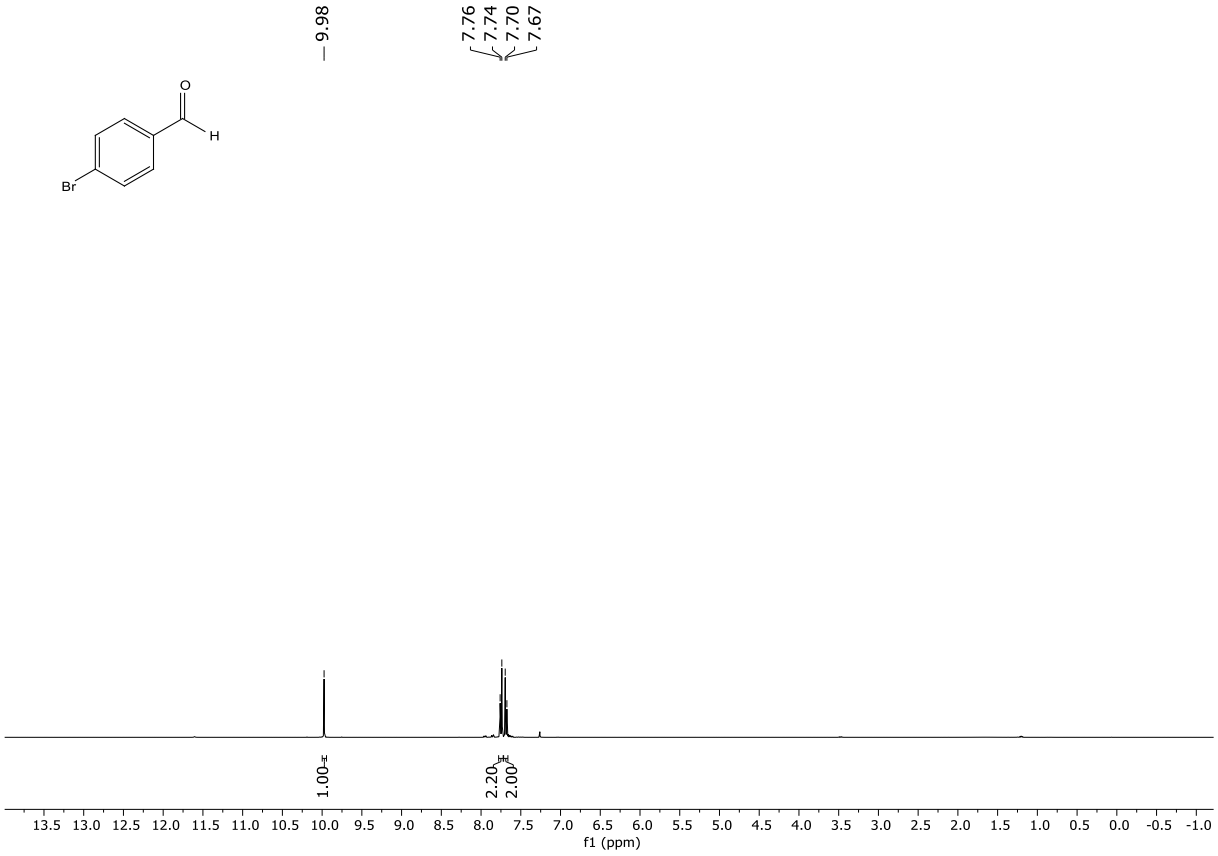
Quinoline-8-carbaldehyde (2m)



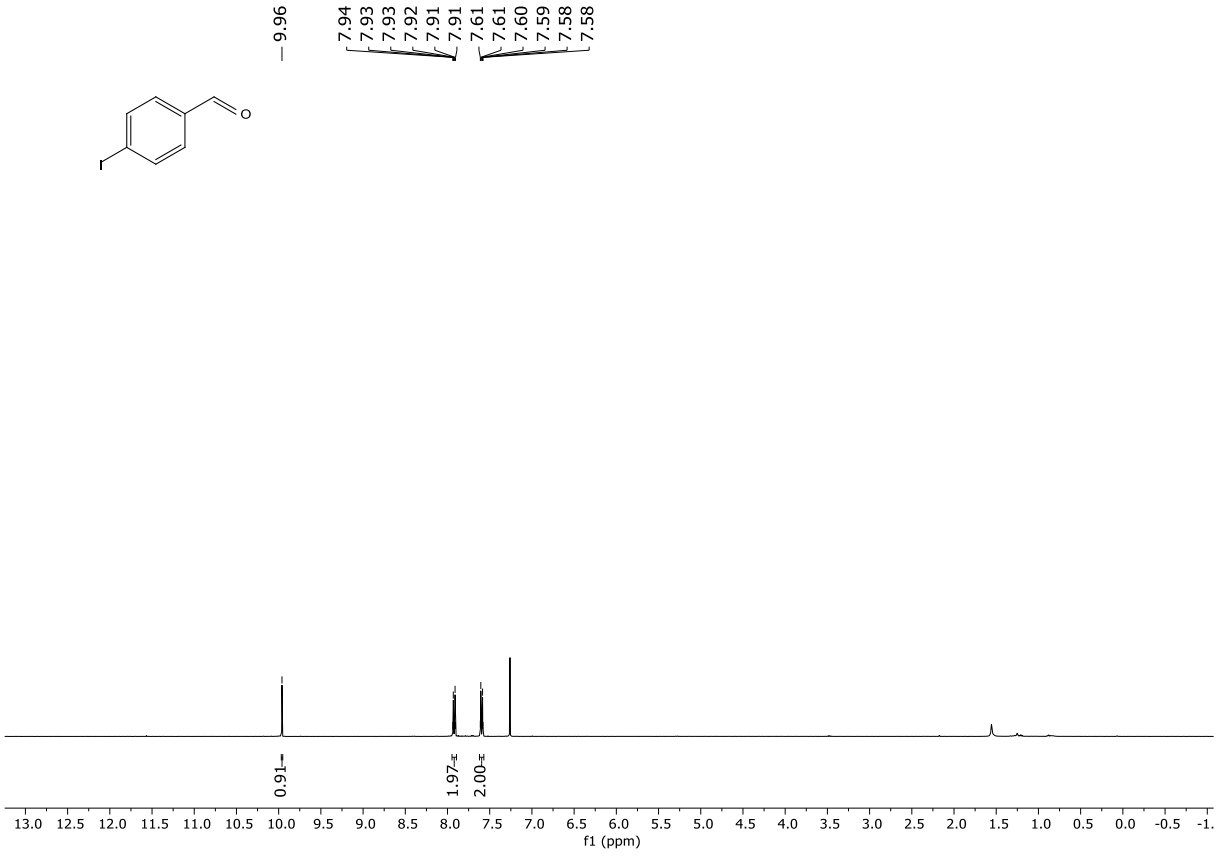
4-Chlorobenzaldehyde (2o)



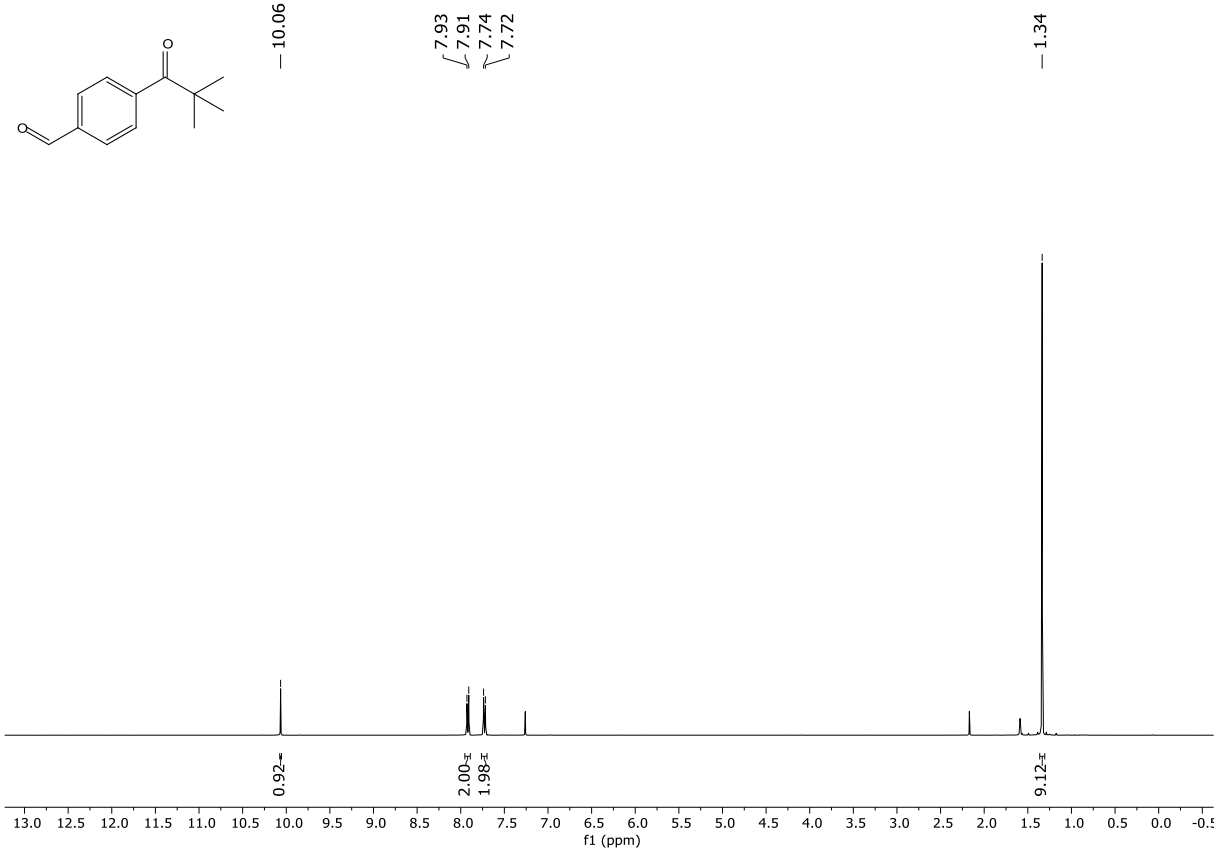
4-Bromobenzaldehyde (2p)



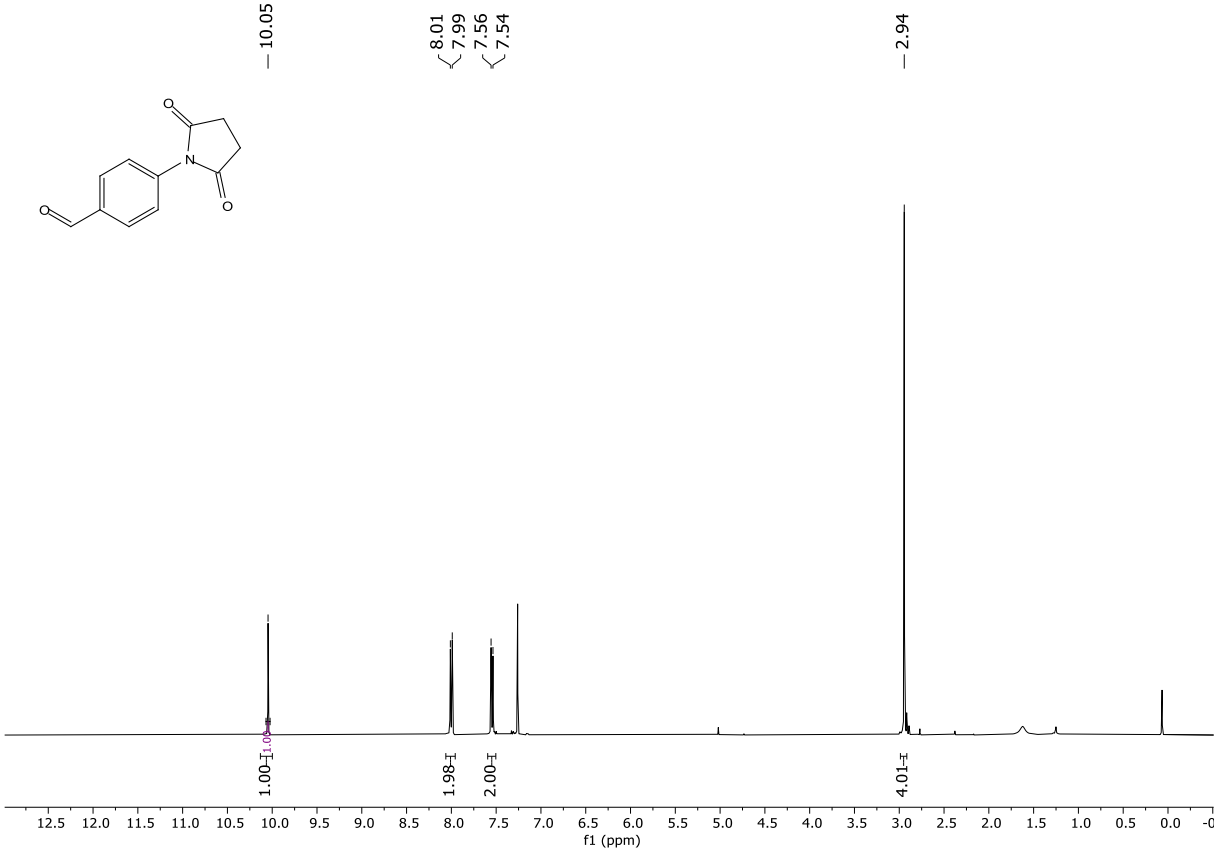
4-Iodobenzaldehyde (2q)

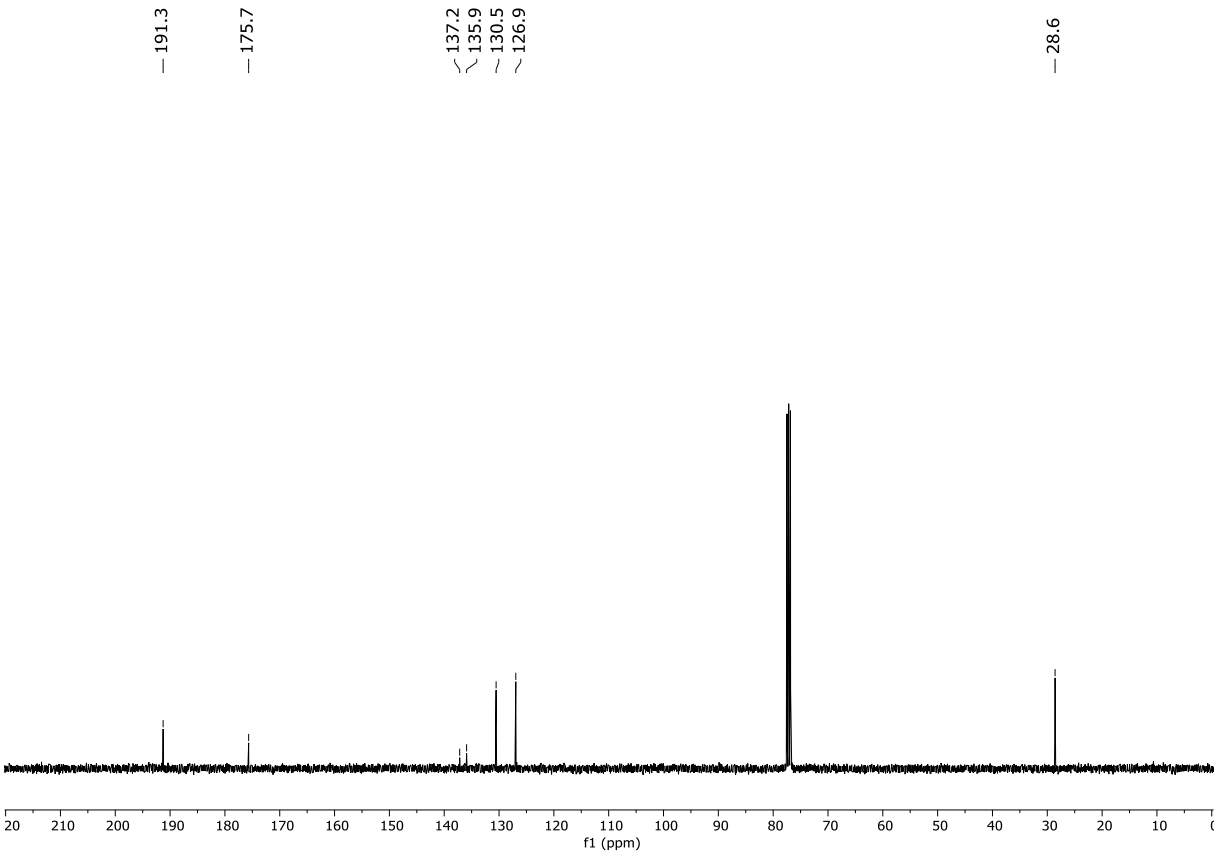


4-Pivaloylbenzaldehyde (2s)

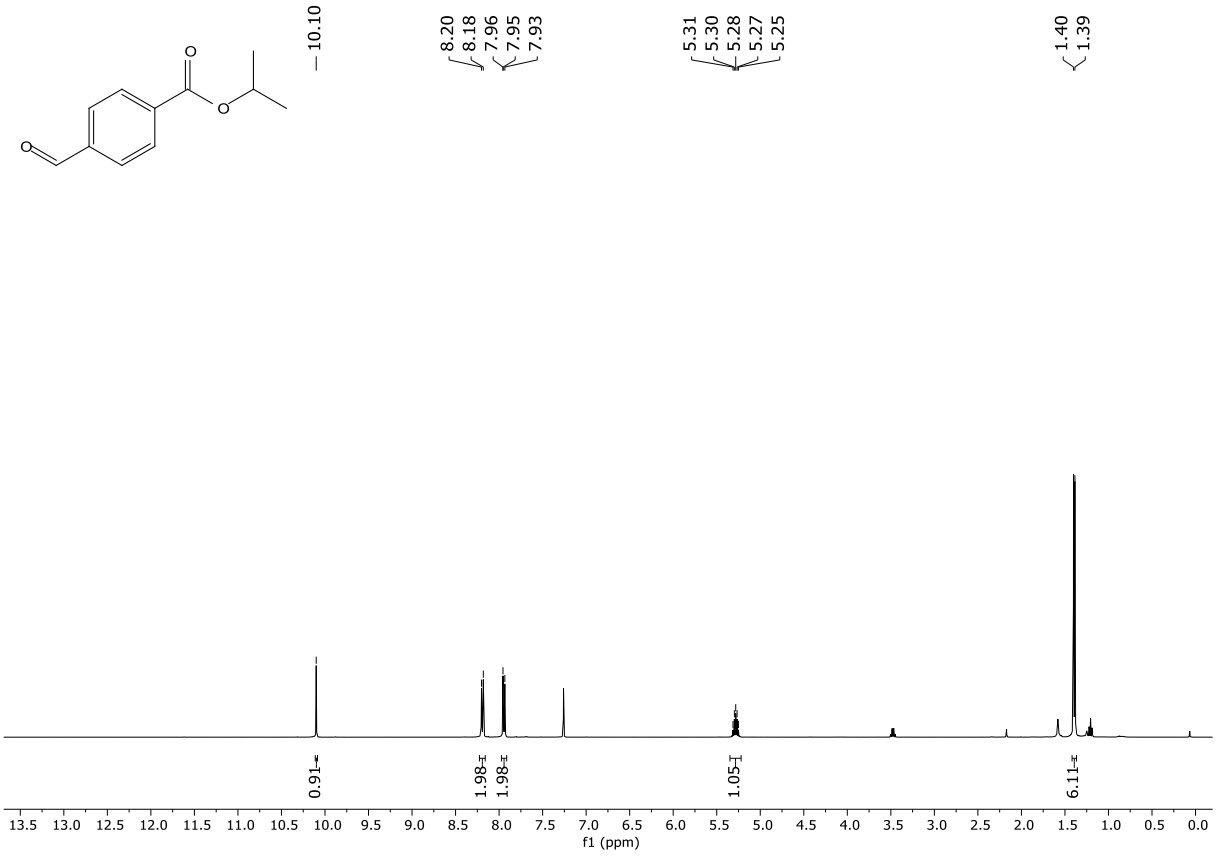


4-(2,5-Dioxopyrrolidin-1-yl)benzaldehyde (2t)

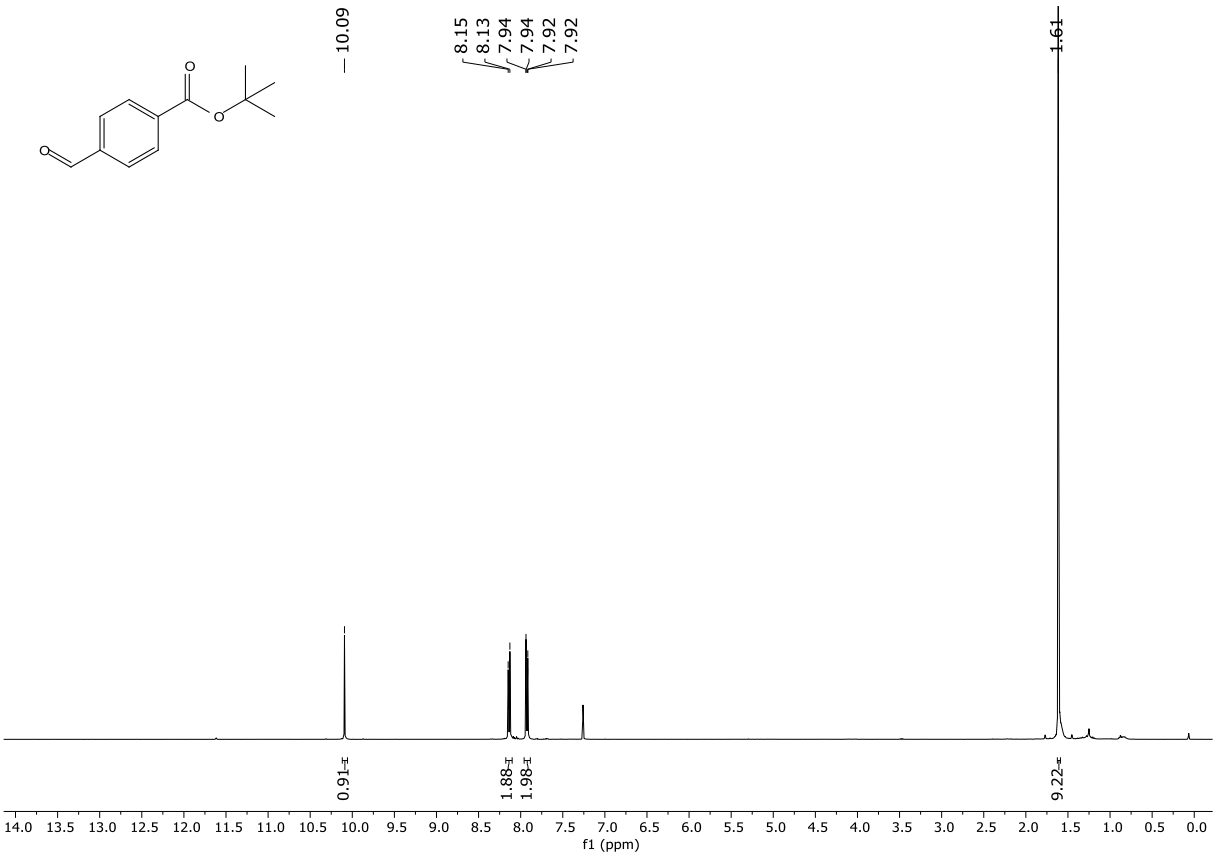




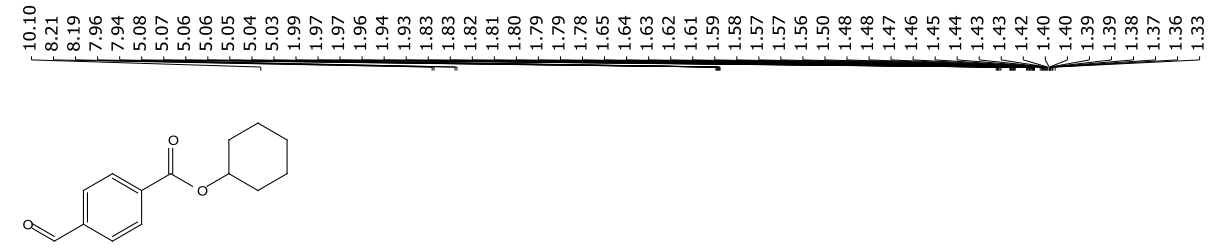
Isopropyl 4-formylbenzoat (2u)

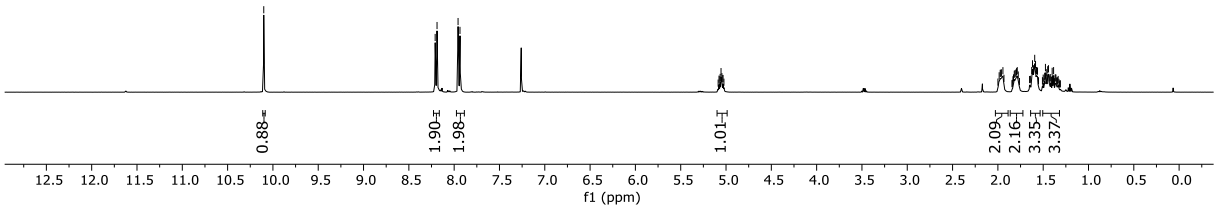


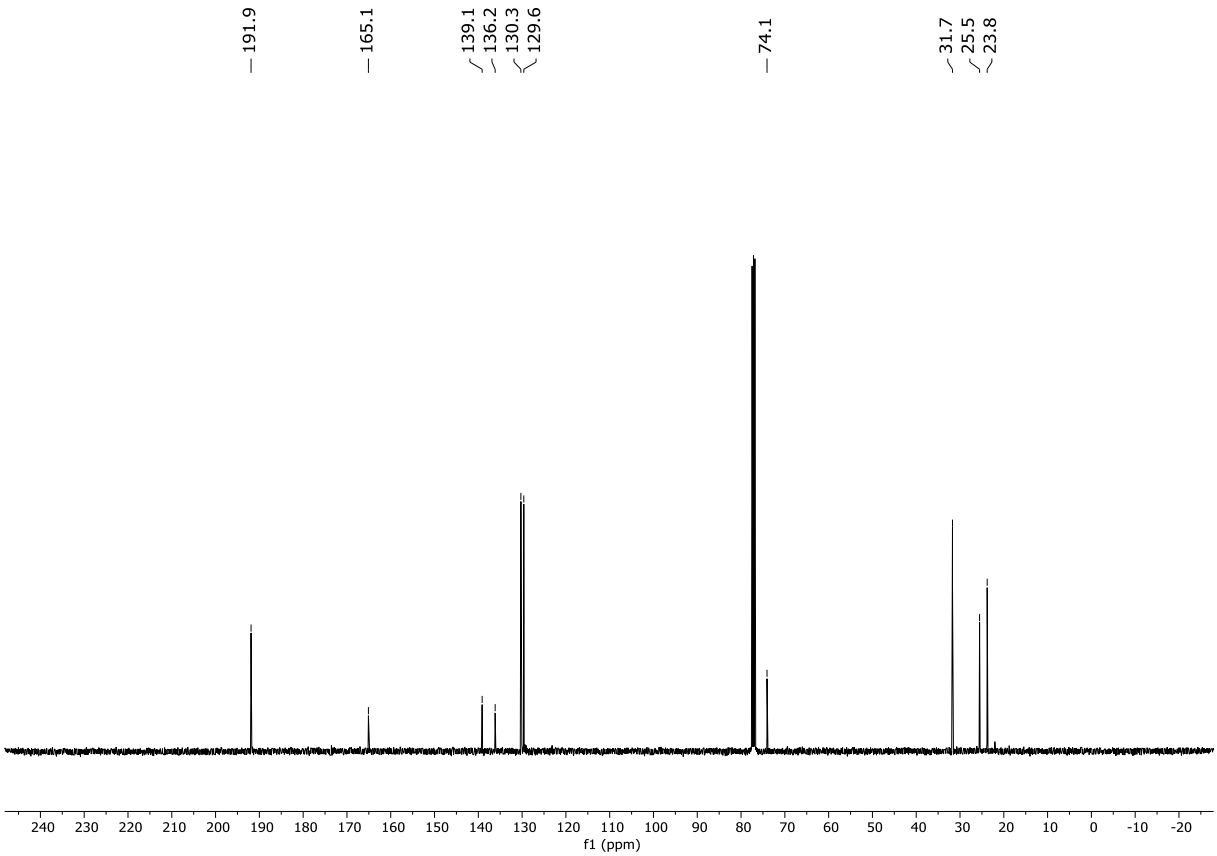
tert-Butyl 4-formylbenzoate (2v)



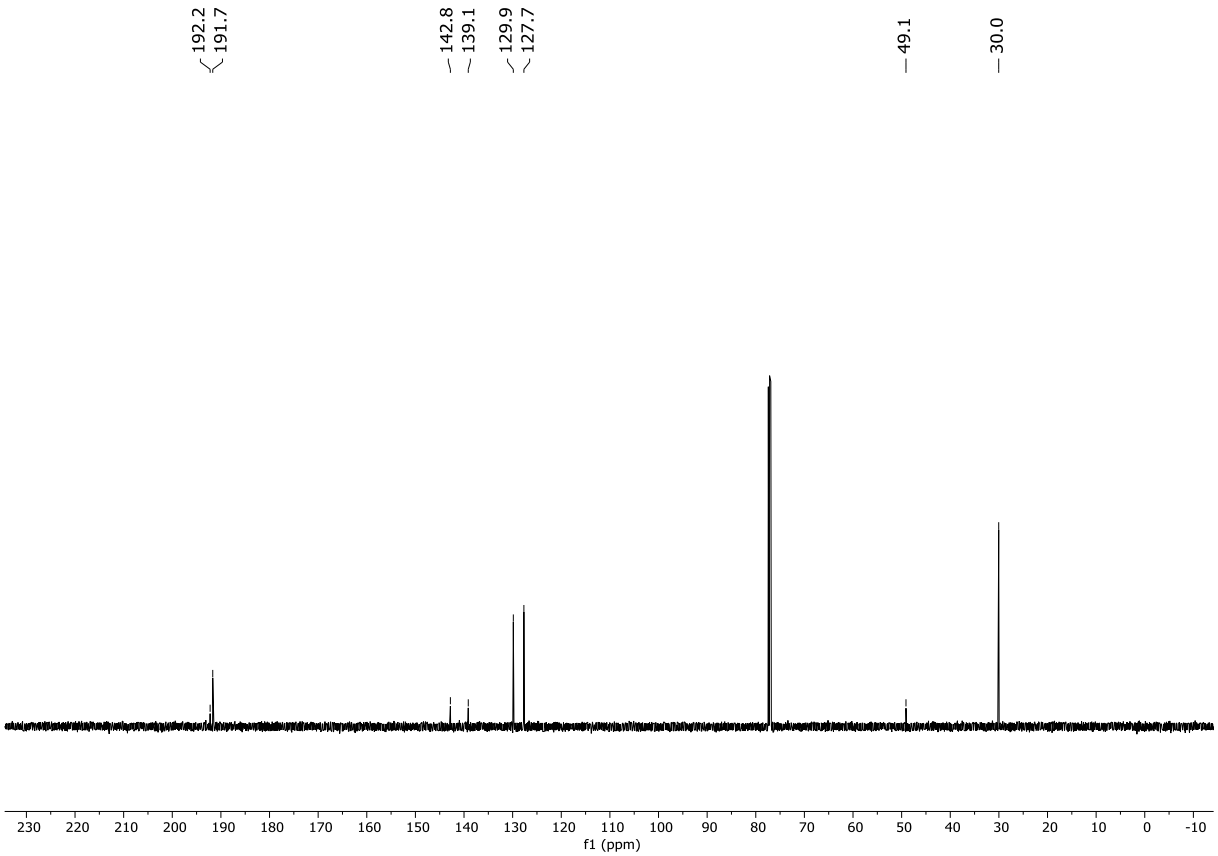
Cyclohexyl 4-formylbenzoate (2w)





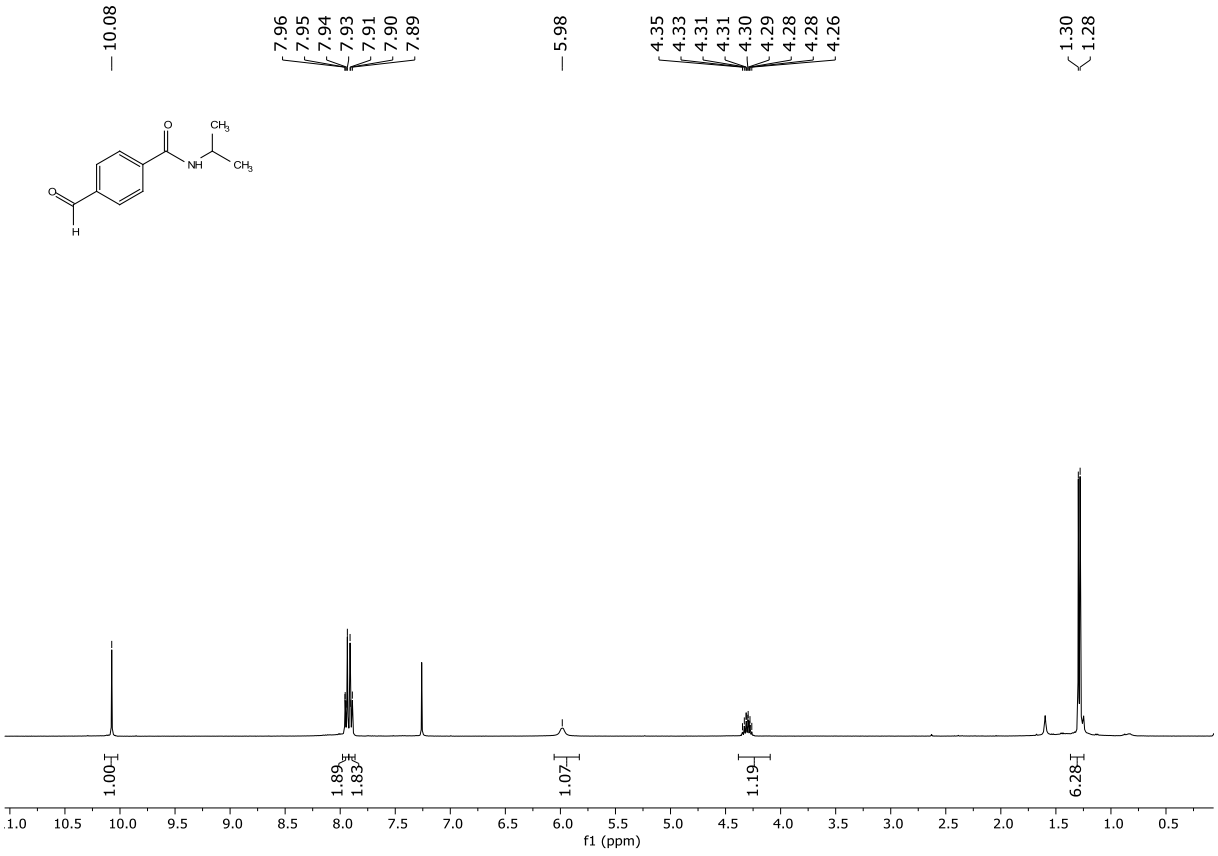


S-(*tert*-Butyl) 4-formylbenzothioate (2x)

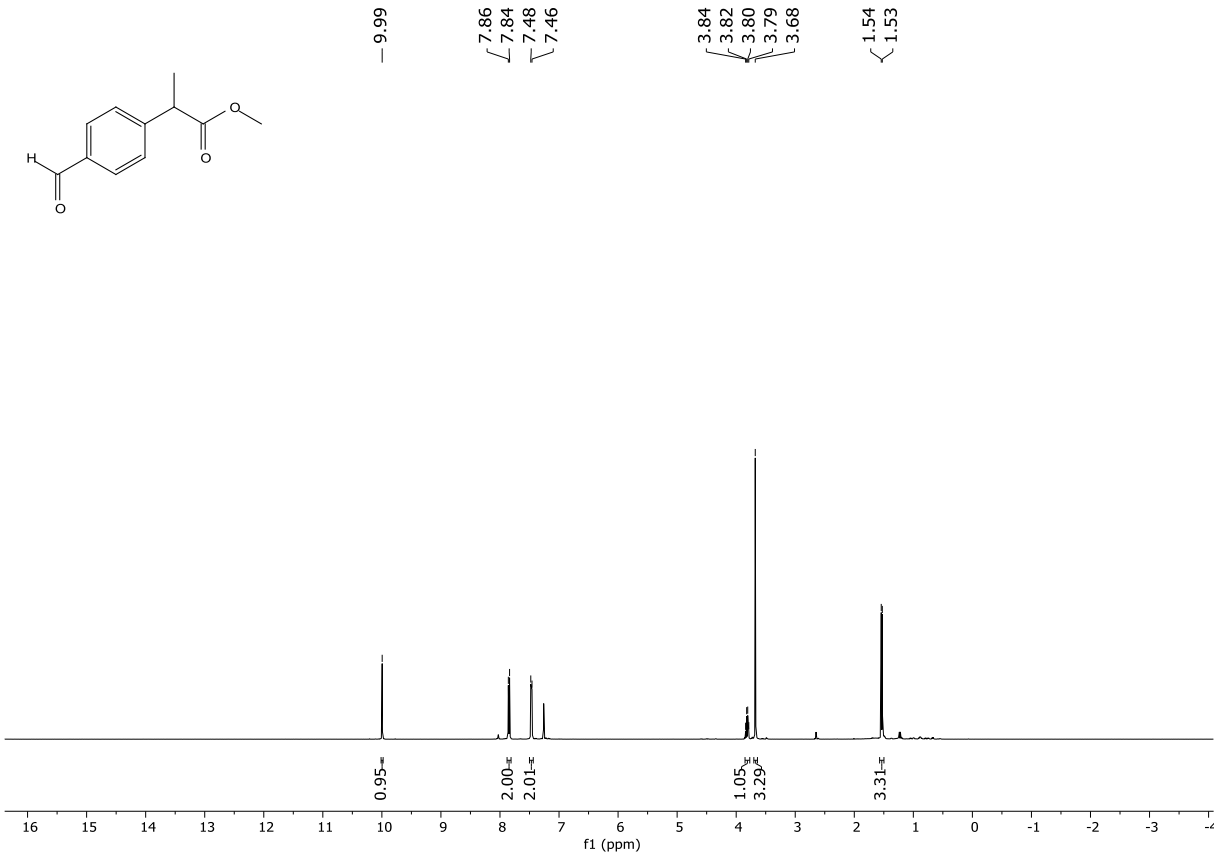


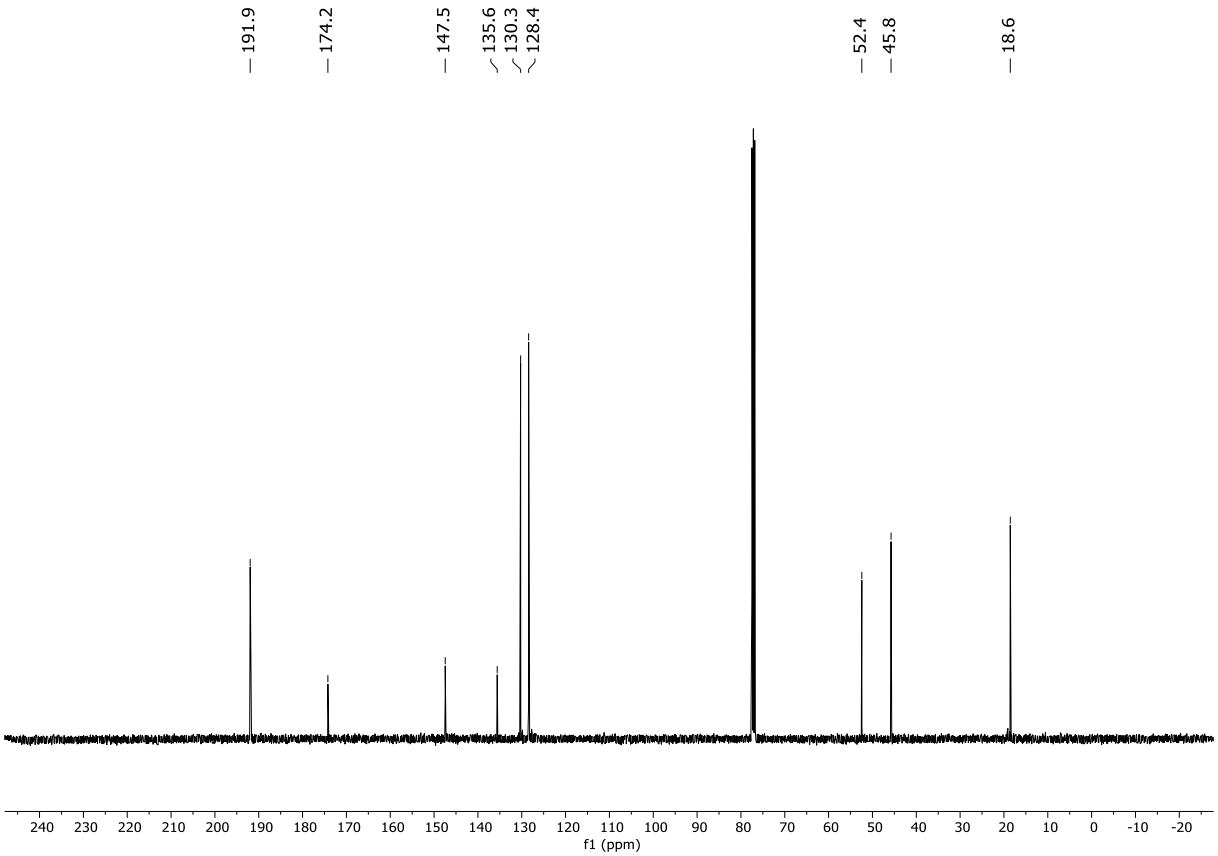
N-(*tert*-Butyl)-4-formylbenzamide (2y)

4-Formyl-*N*-isopropylbenzamide (2z)

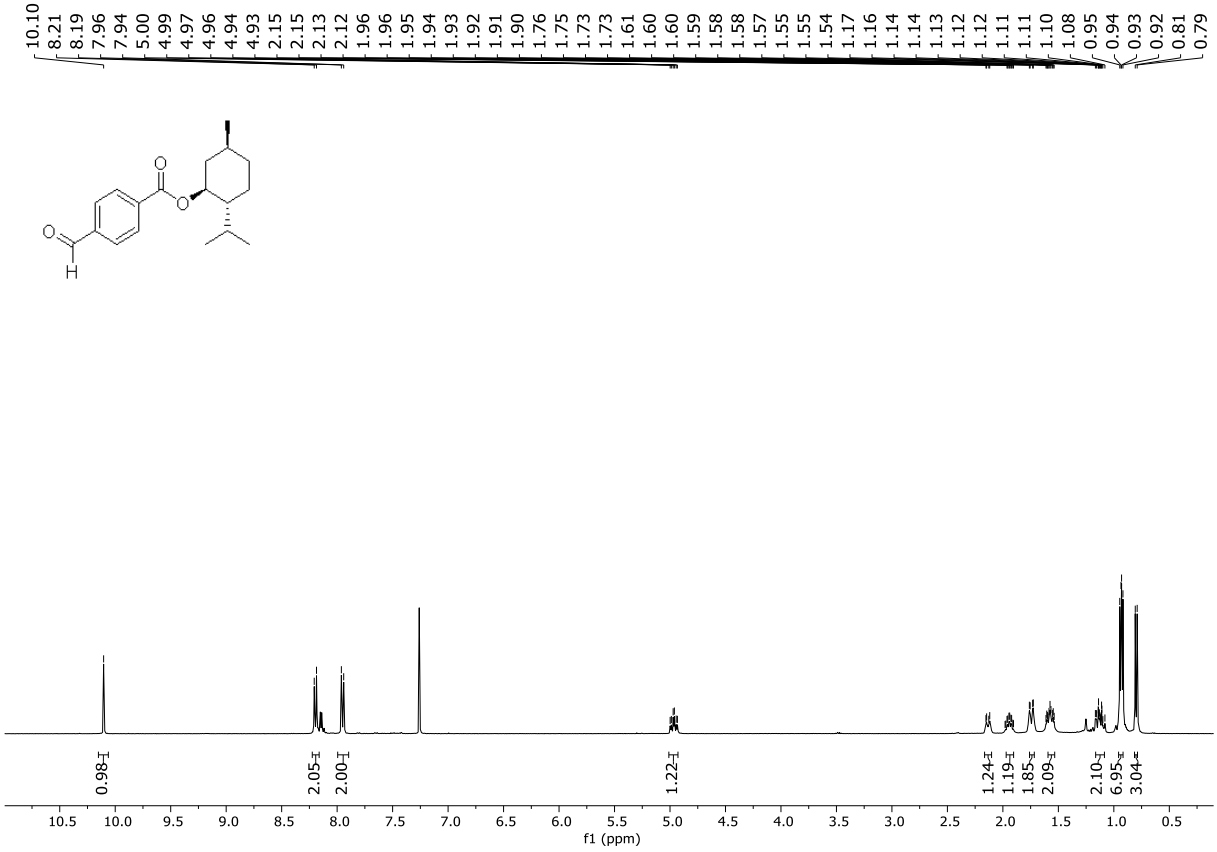


Methyl 2-(4-formylphenyl)propanoate (2aa)

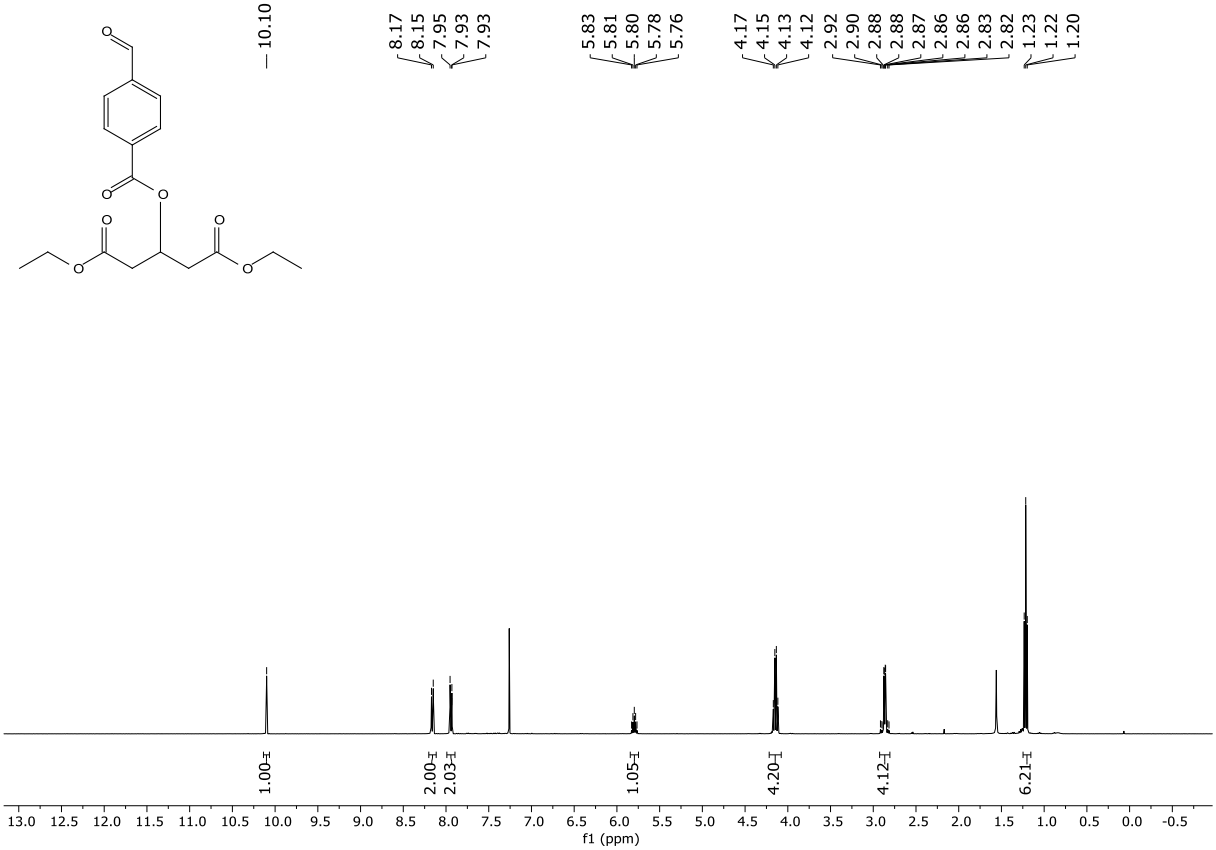


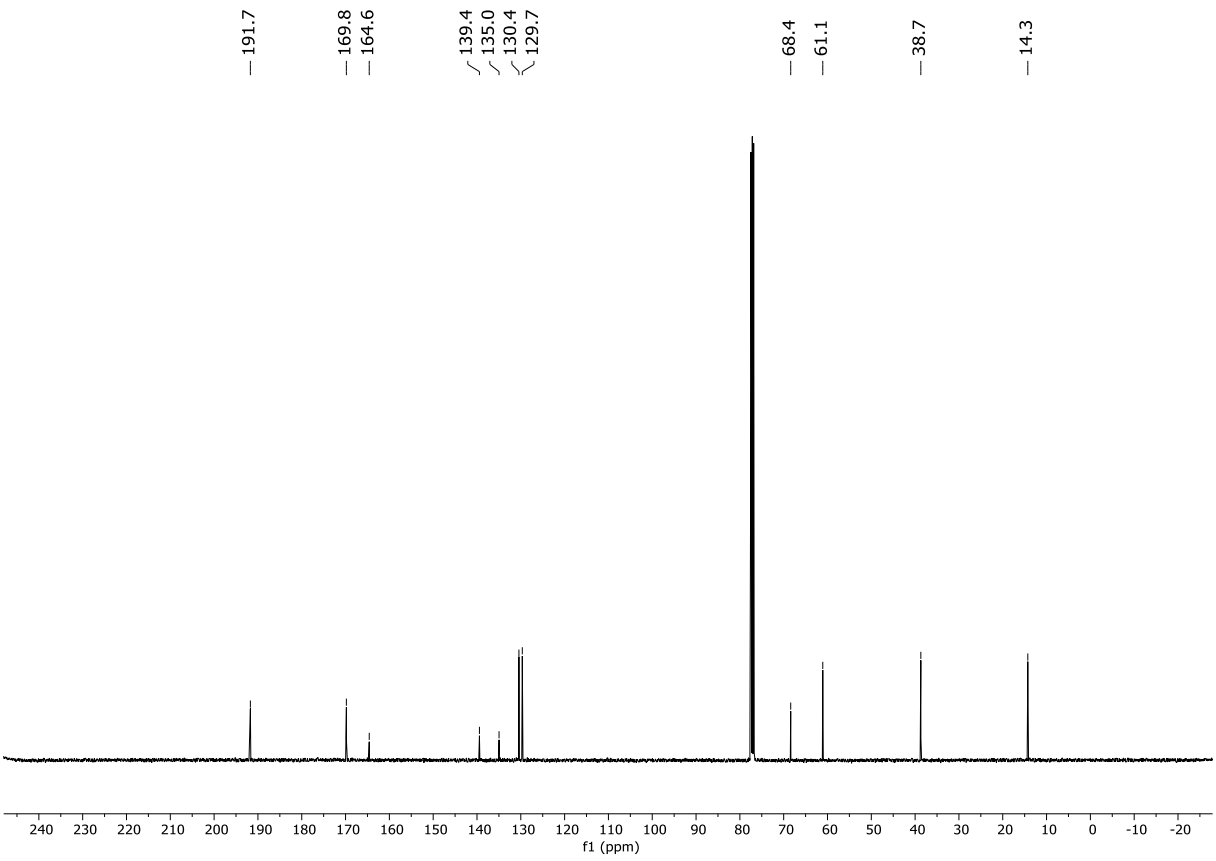


(1*S*,2*R*,5*S*)-2-Isopropyl-5-methylcyclohexyl 4-formylbenzoate (2ab)

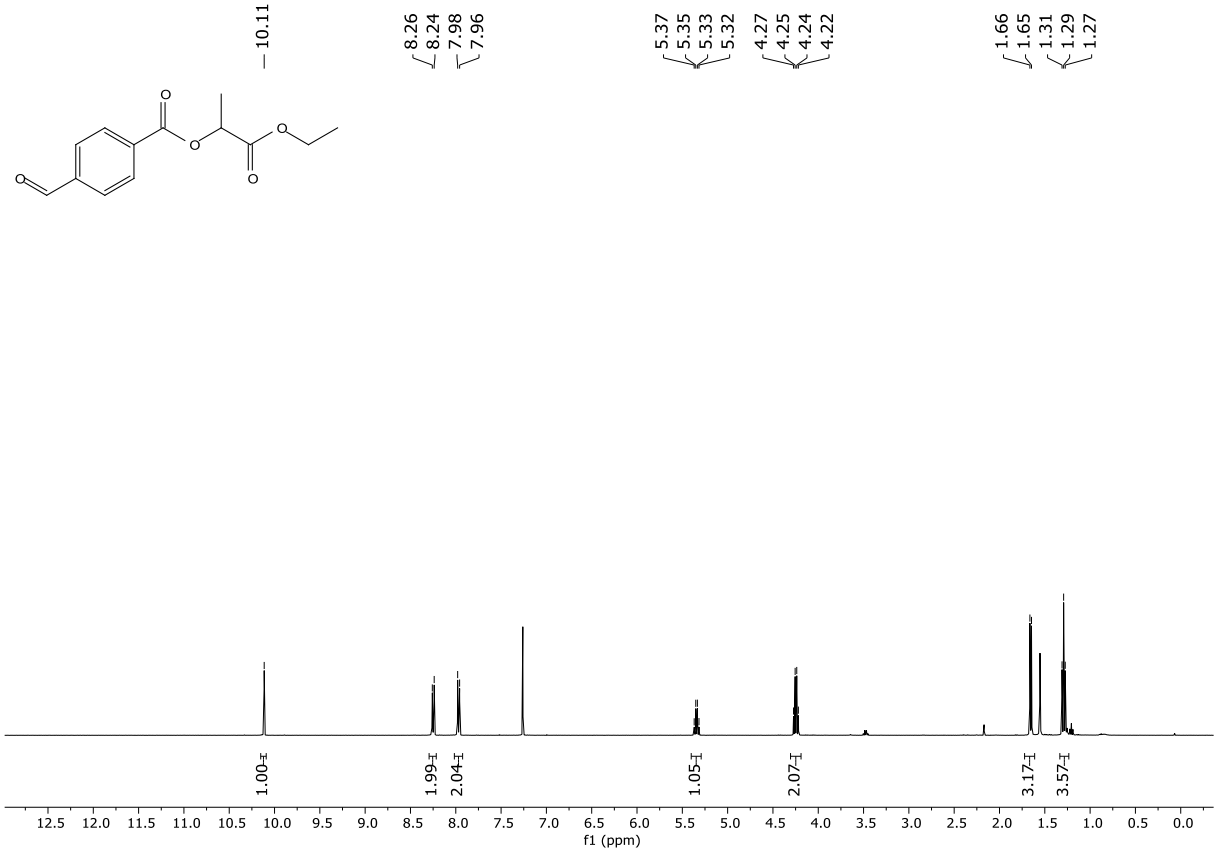


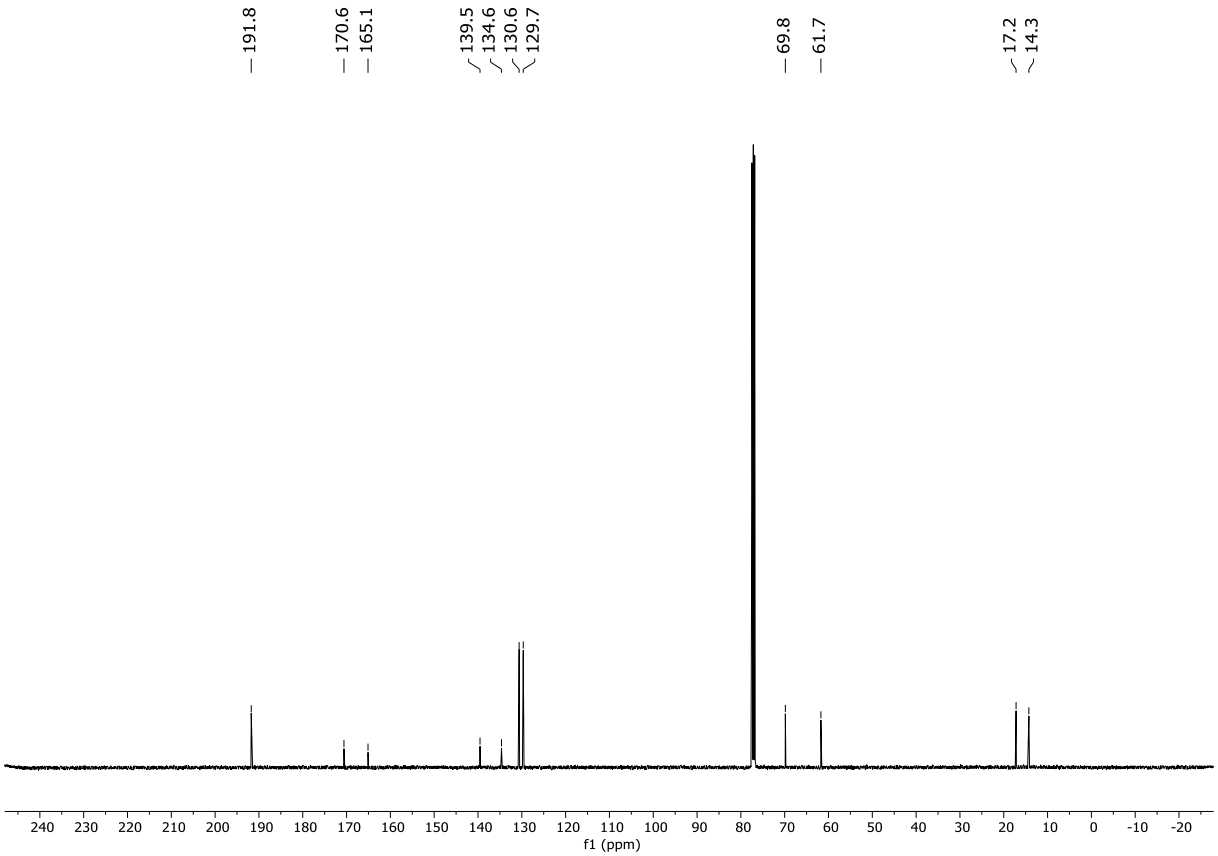
Diethyl 3-((4-formylbenzoyl)oxy)pentanedioate (2ac)



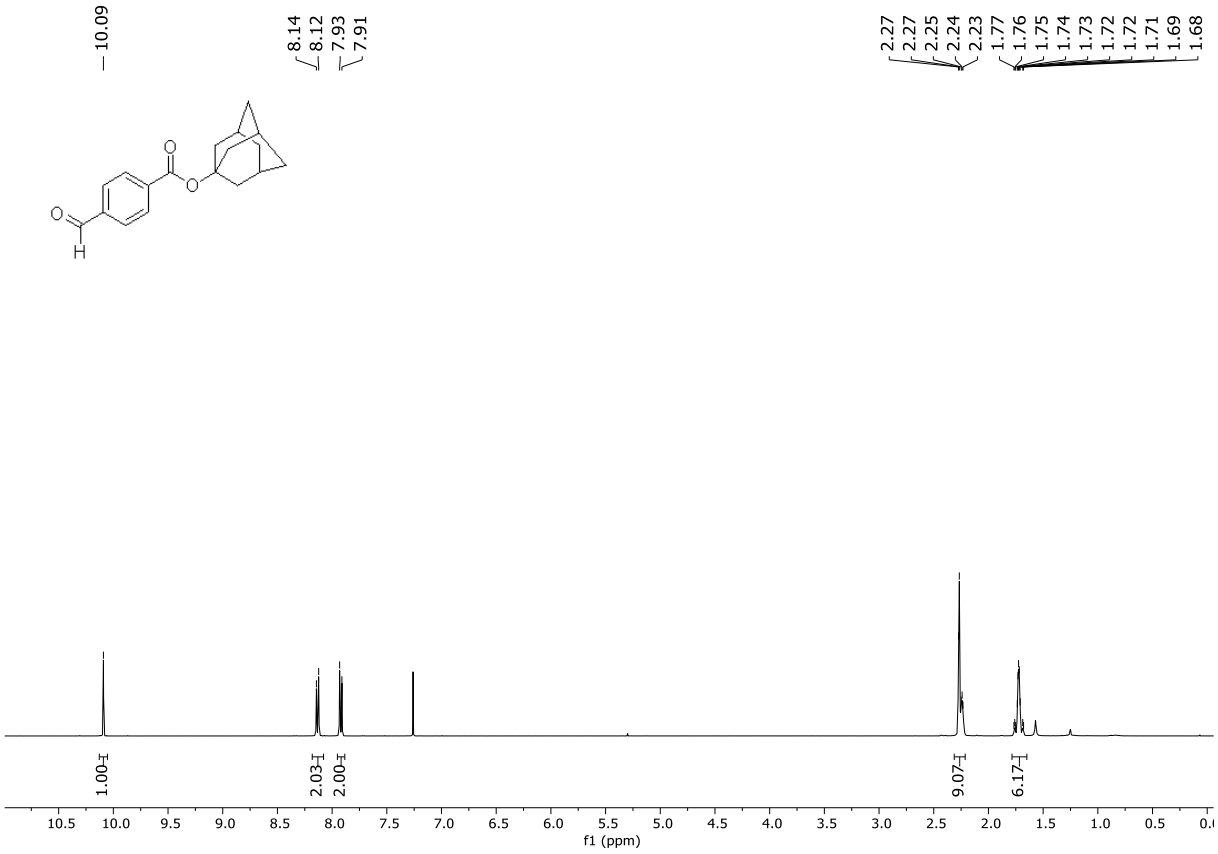


4-Ethoxy-4-oxobutan-2-yl 4-formylbenzoate (2ad)

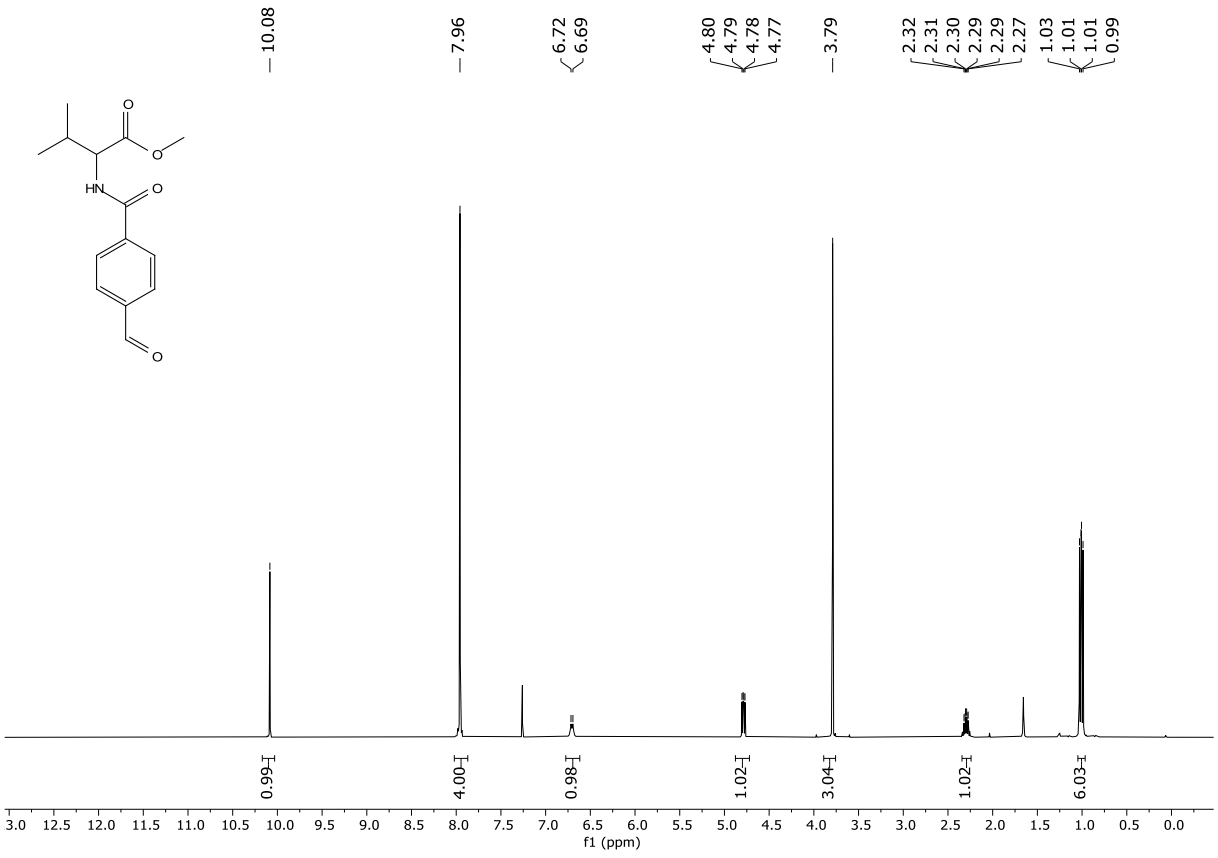




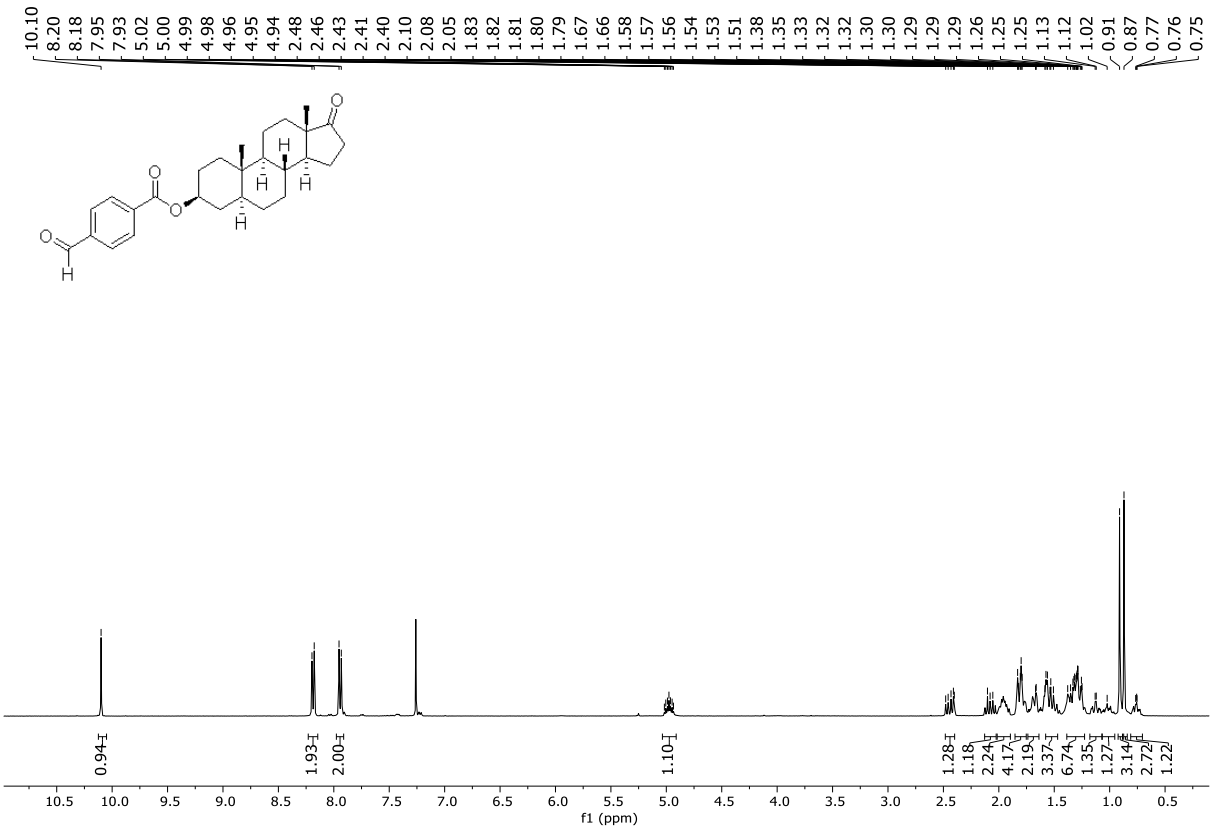
(1*S*,3*s*)-Adamantan-1-yl 4-formylbenzoate (2ae)



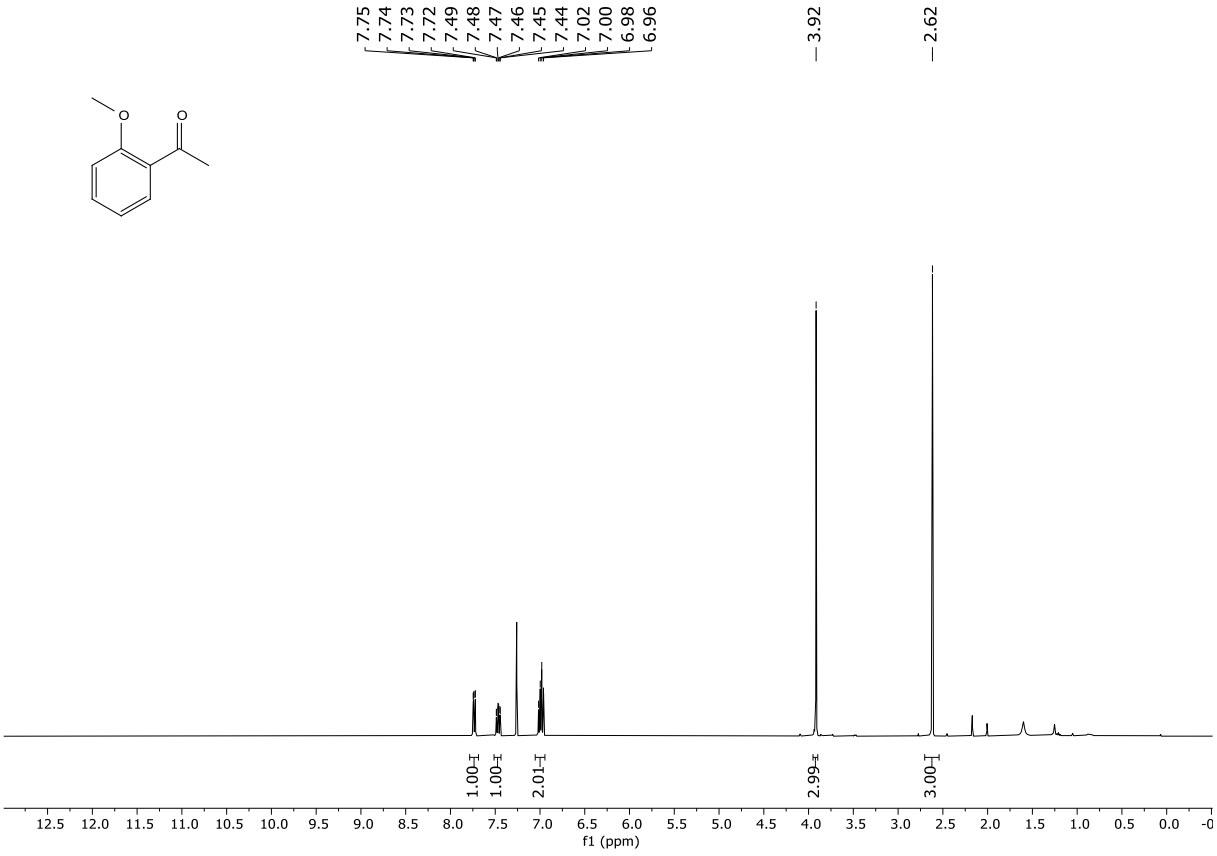
Methyl (4-formylbenzoyl)valinate (2af)



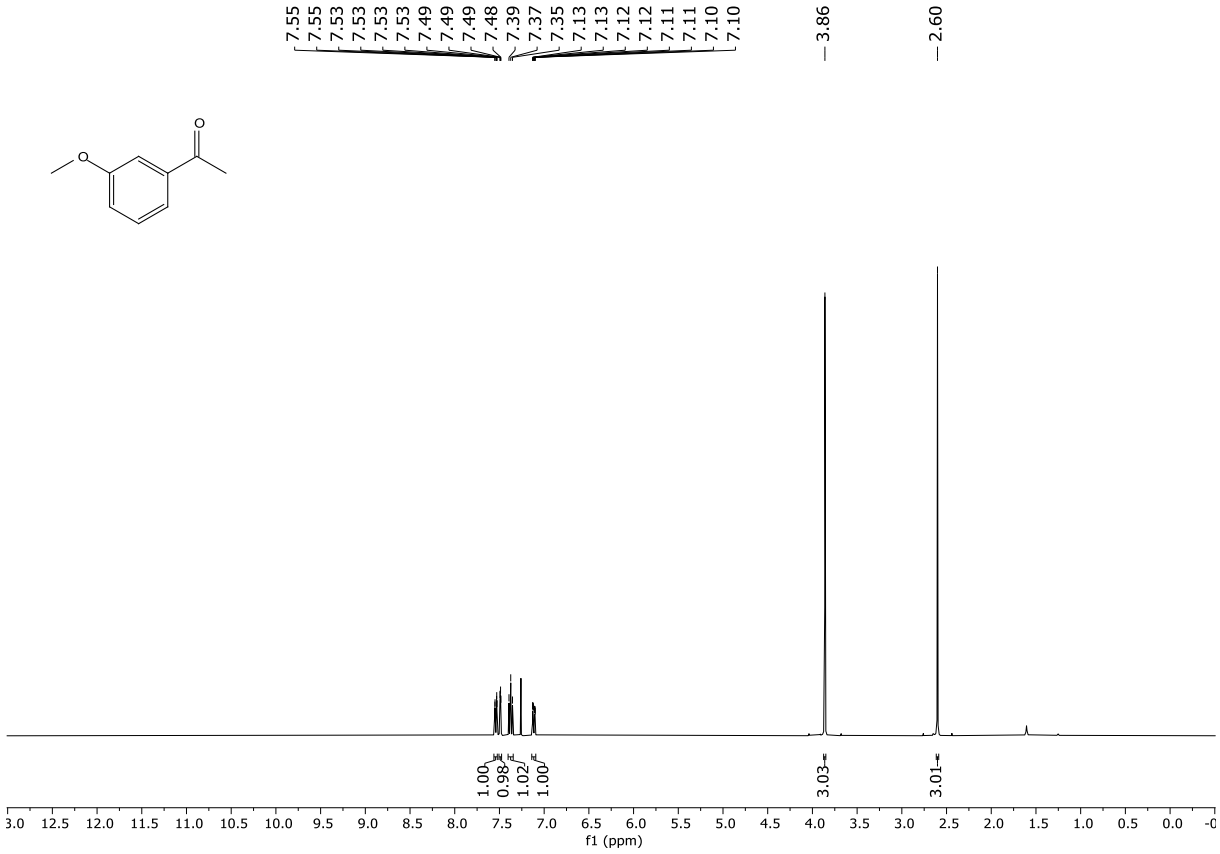
(3*R*,5*S*,8*R*,9*S*,10*S*,13*S*,14*S*)-10,13-Dimethyl-17-oxohexadecahydro-1*H*-cyclopenta[*a*]phenanthren-3-yl 4-formylbenzoate (2ag)



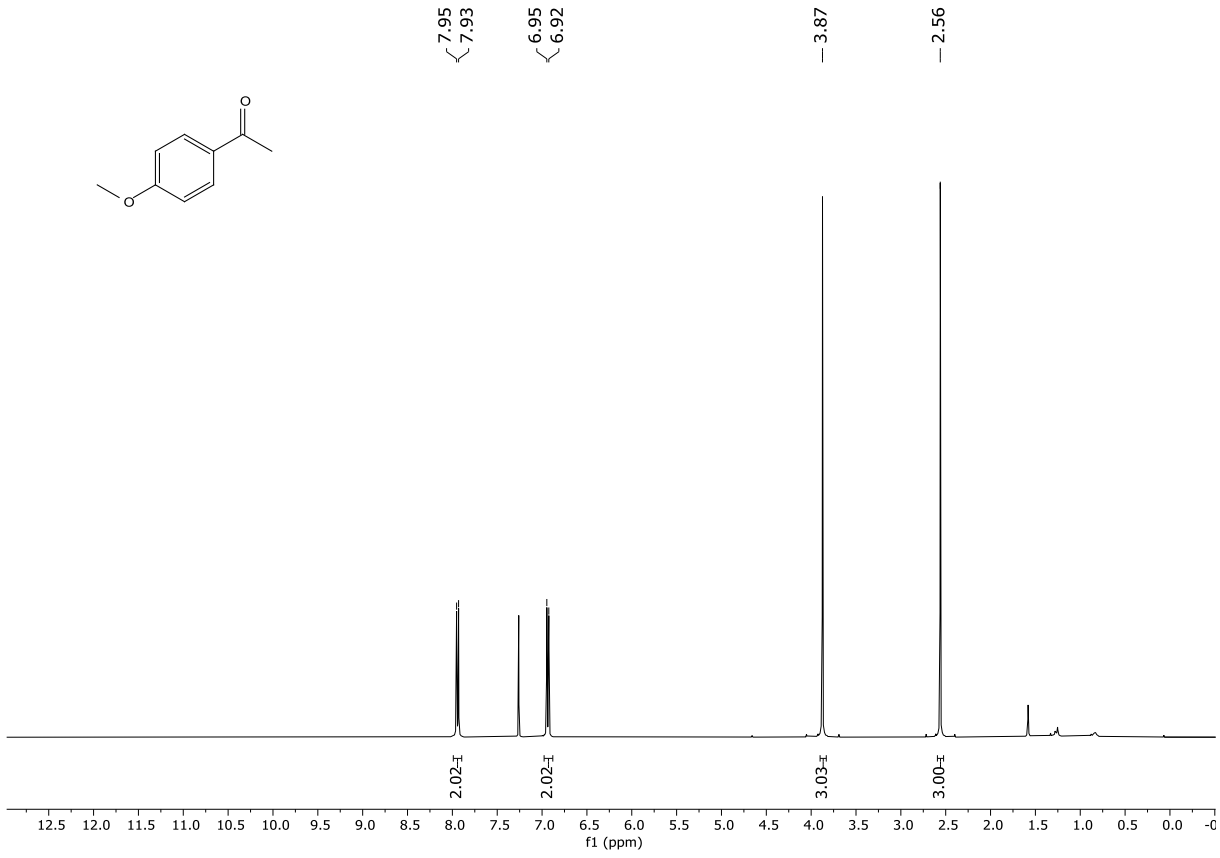
1-(2-Methoxyphenyl)ethan-1-one (6g)



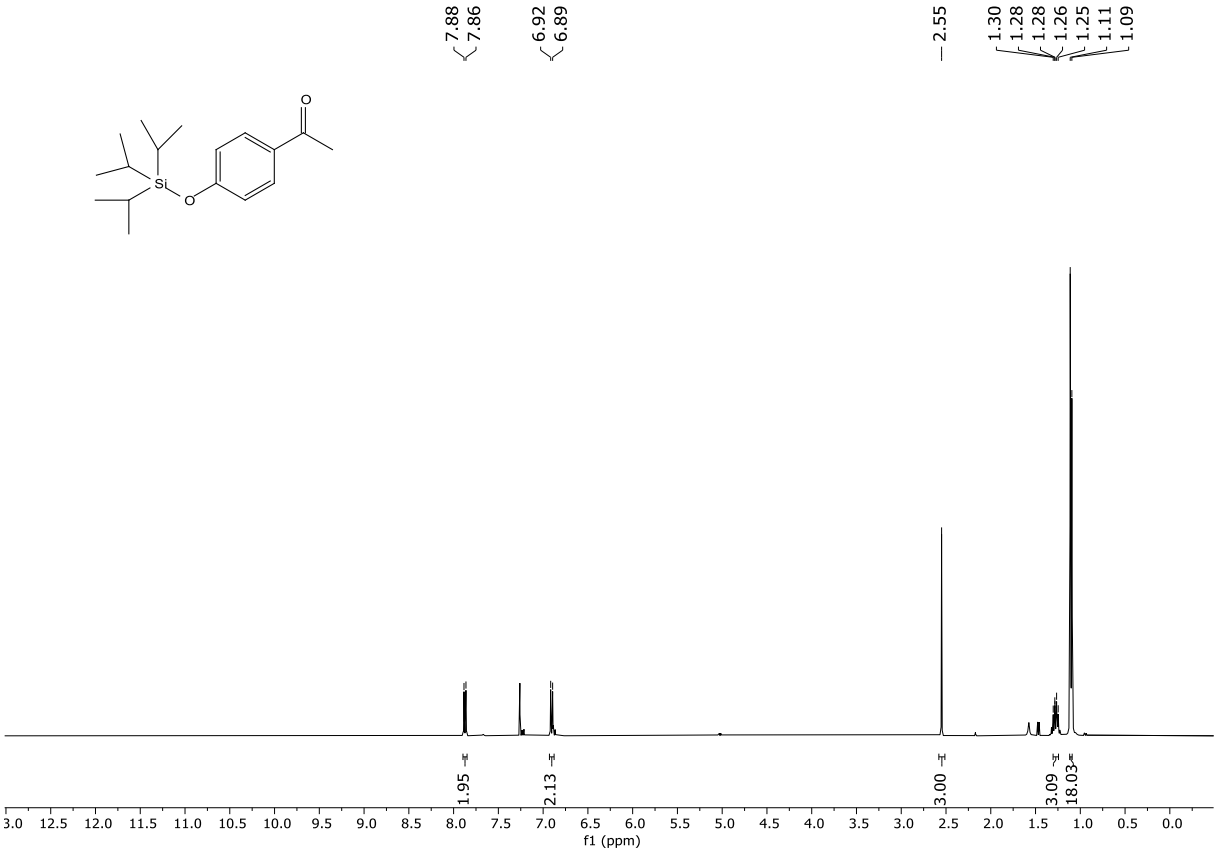
1-(3-Methoxyphenyl)ethan-1-one (6h)



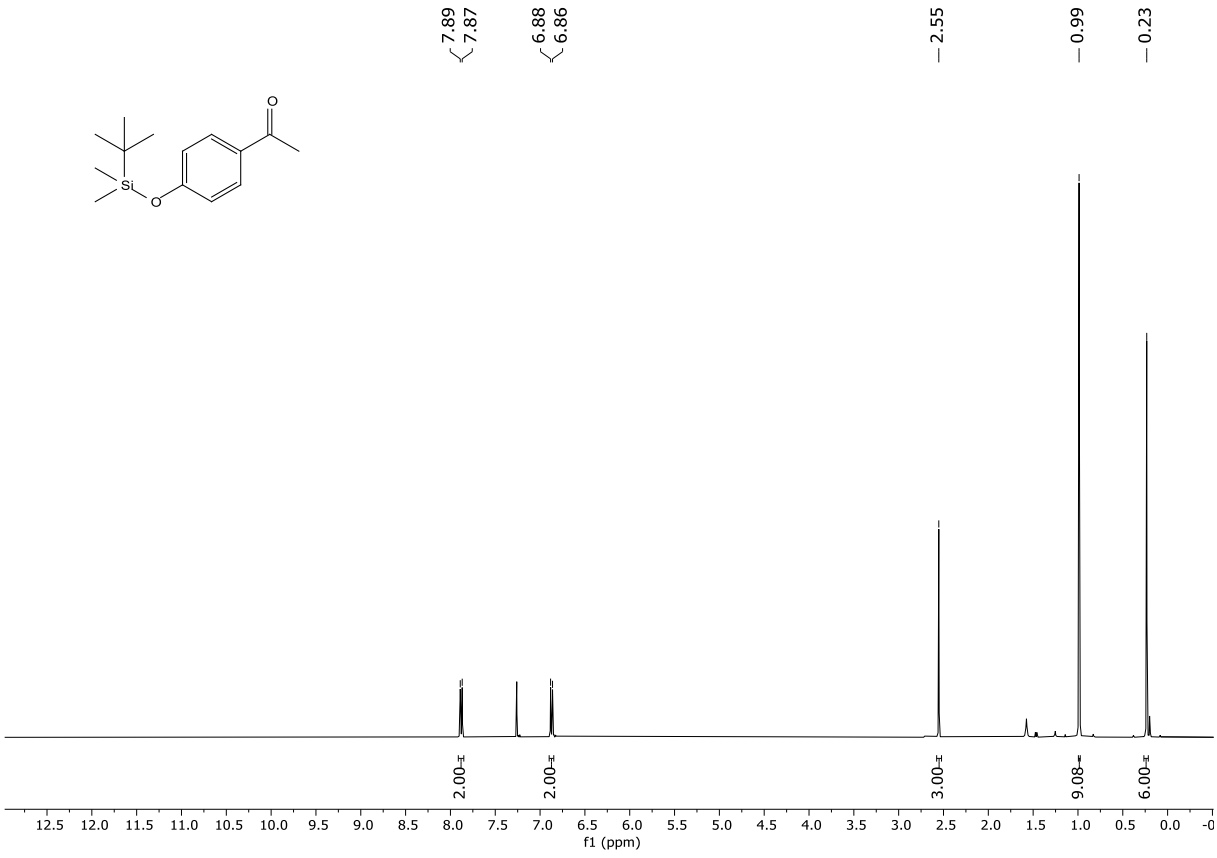
1-(4-Methoxyphenyl)ethan-1-one (6i)



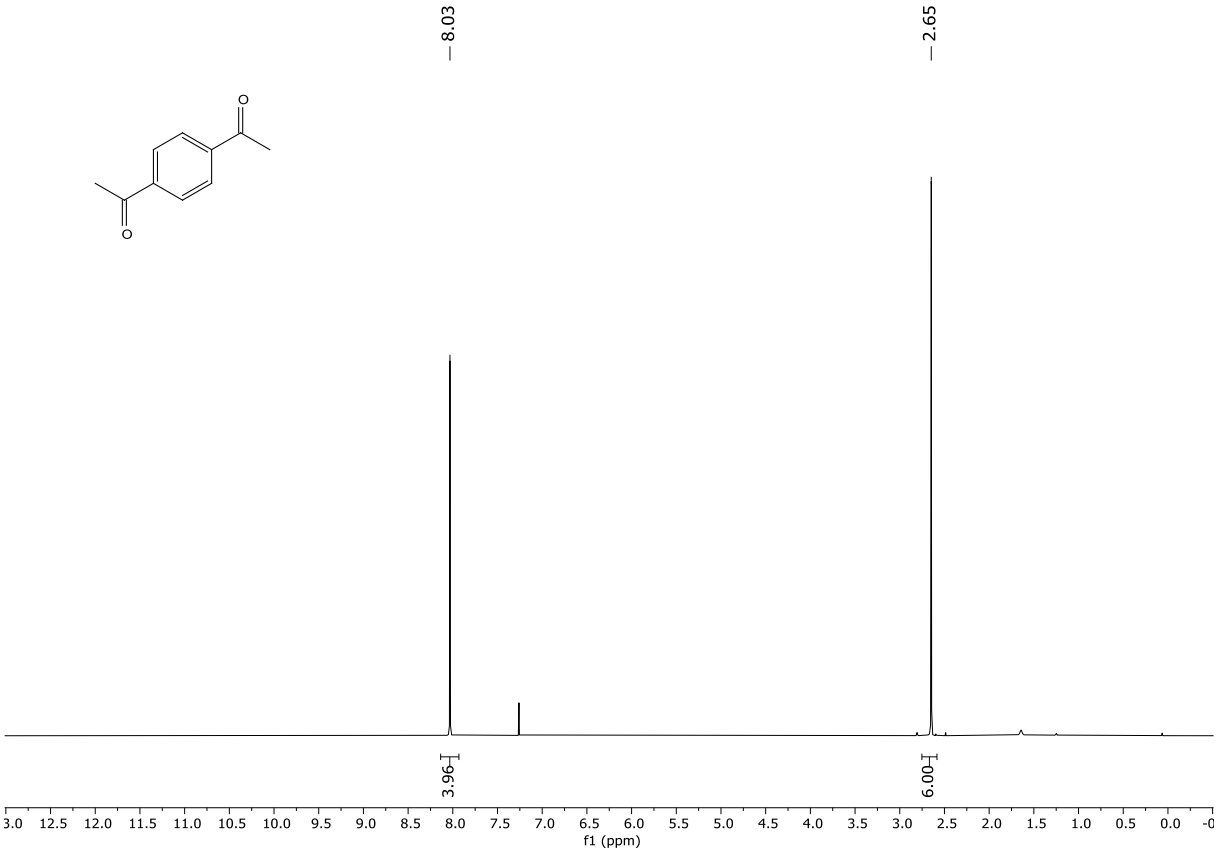
1-(4-((Triisopropylsilyl)oxy)phenyl)ethan-1-one (6j)



1-(4-((*tert*-Butyldimethylsilyl)oxy)phenyl)ethan-1-one (6k)

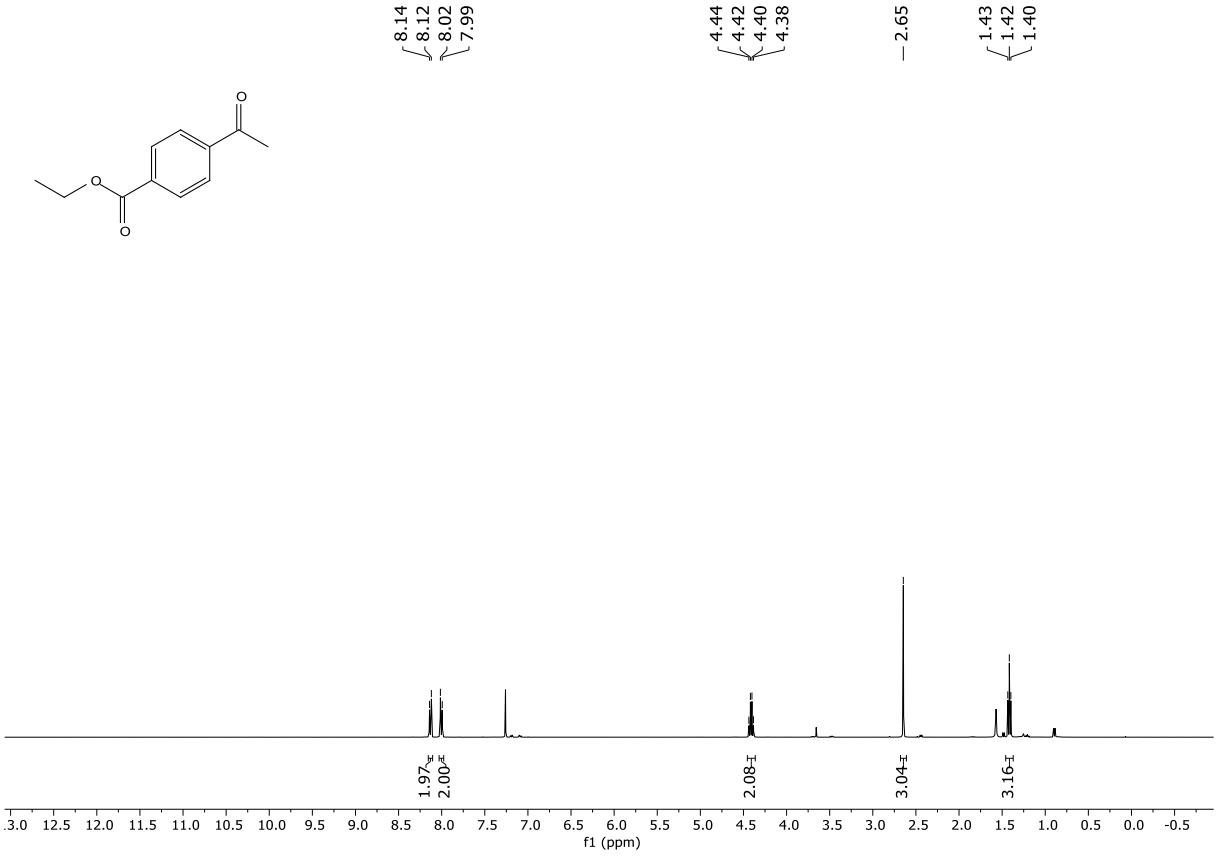


1,1'-(1,4-Phenylene)bis(ethan-1-one) (6l)

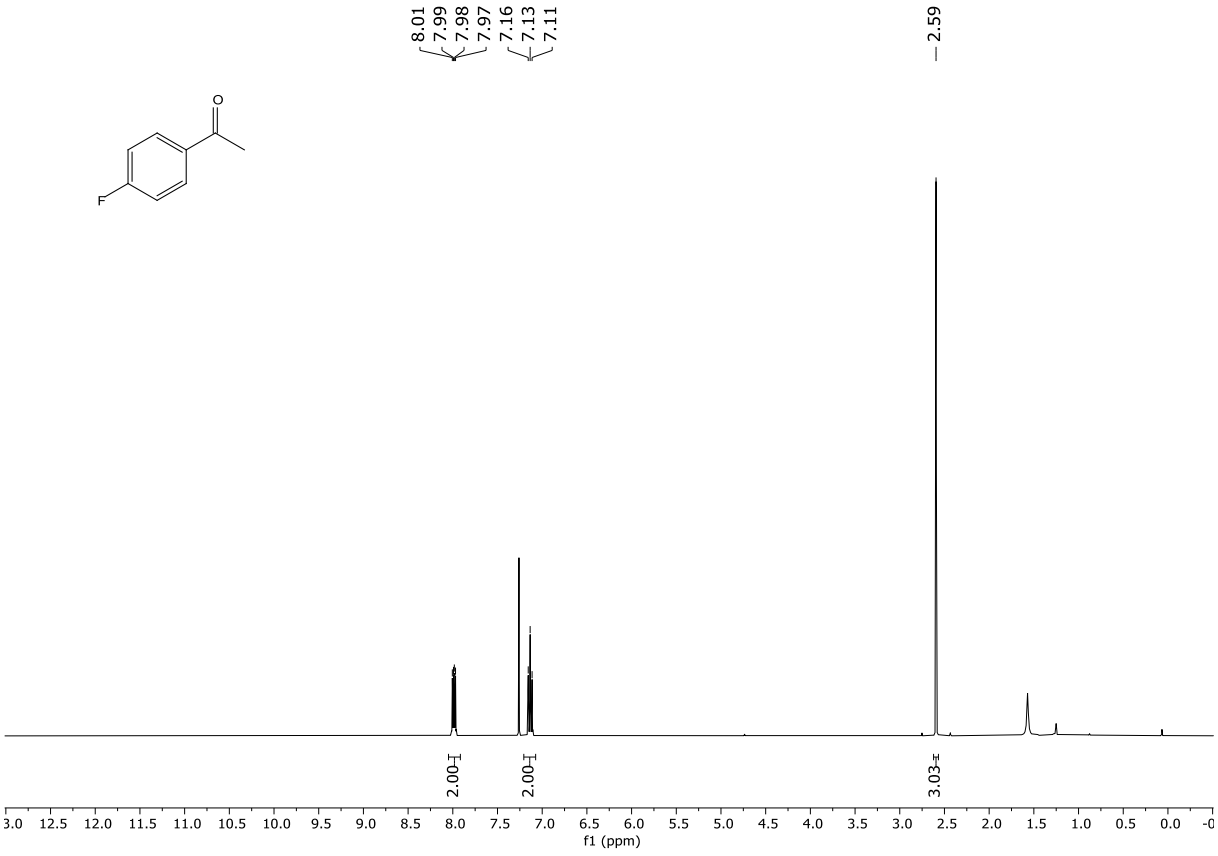


Methyl 4-acetylbenzoate (6m)

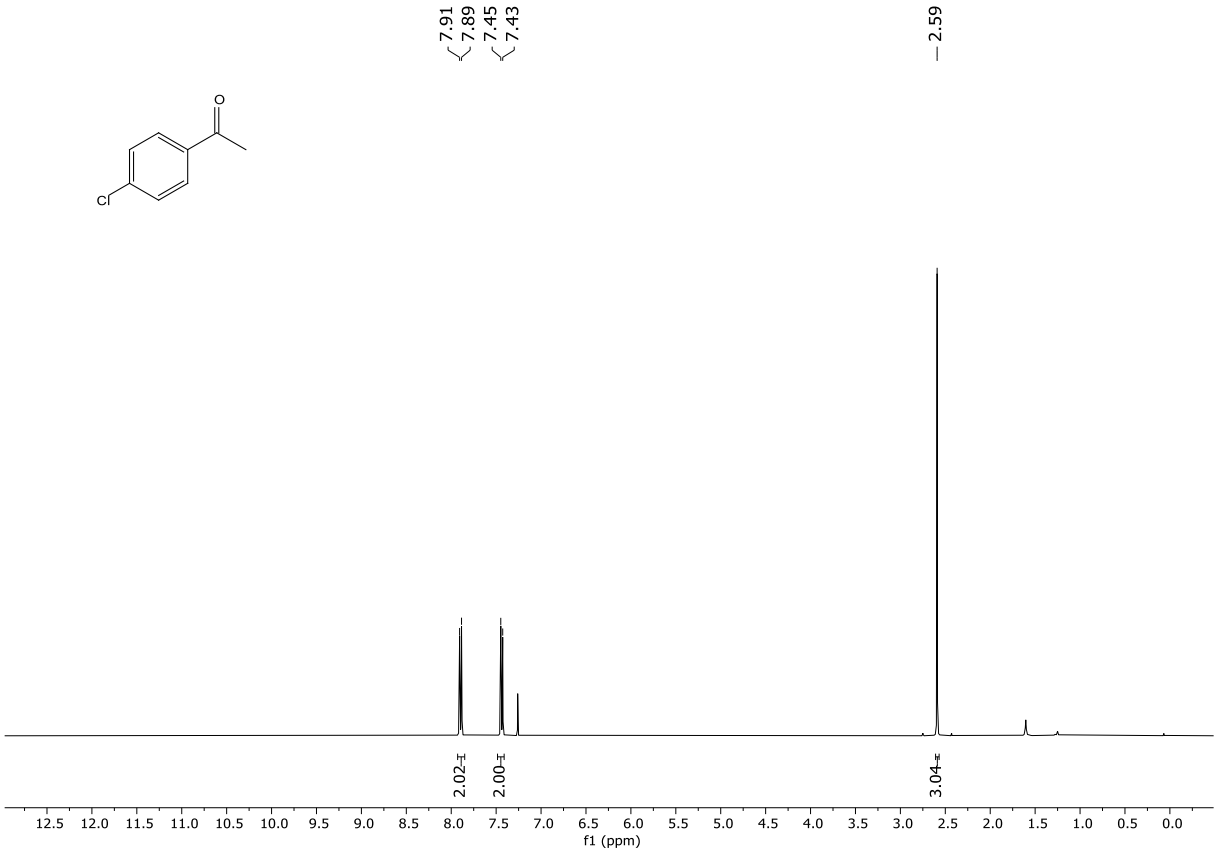
Ethyl 4-acetylbenzoate (6n)



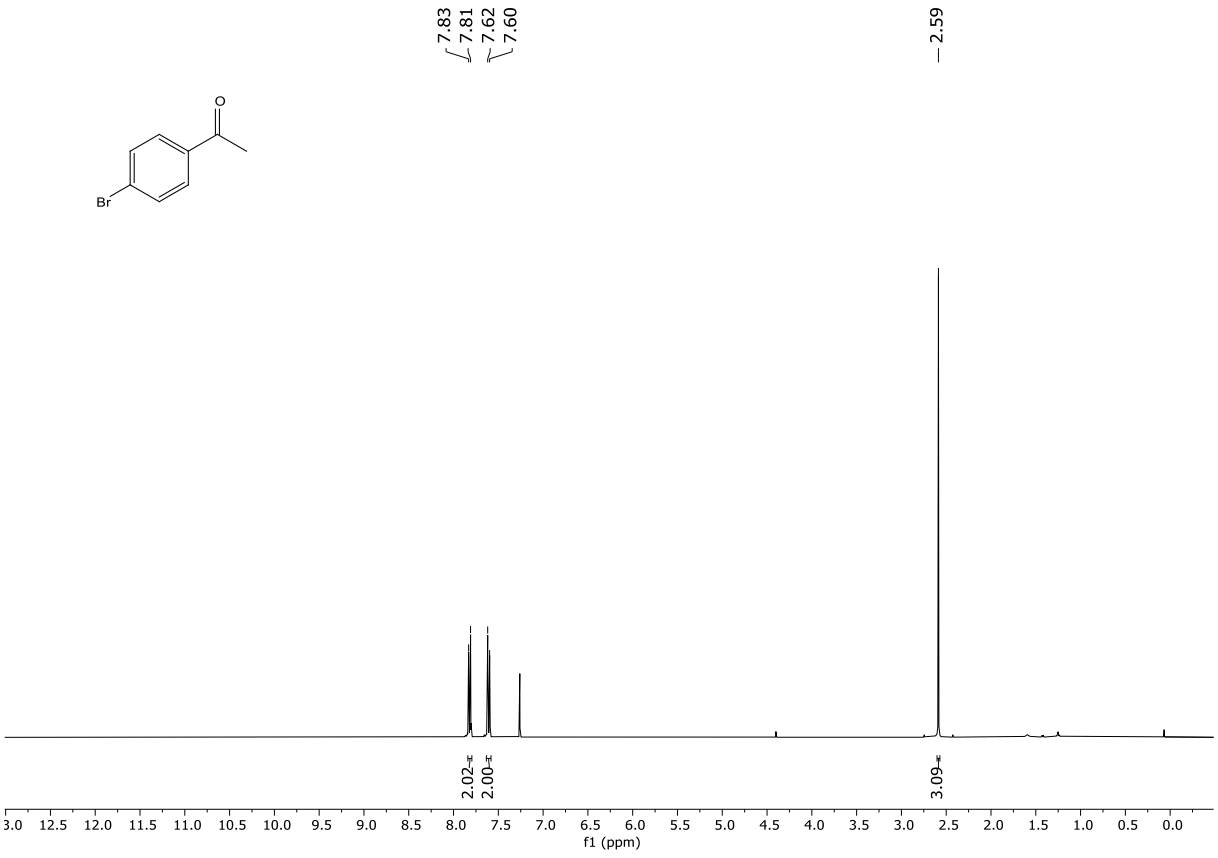
1-(4-Fluorophenyl)ethan-1-one (6o)



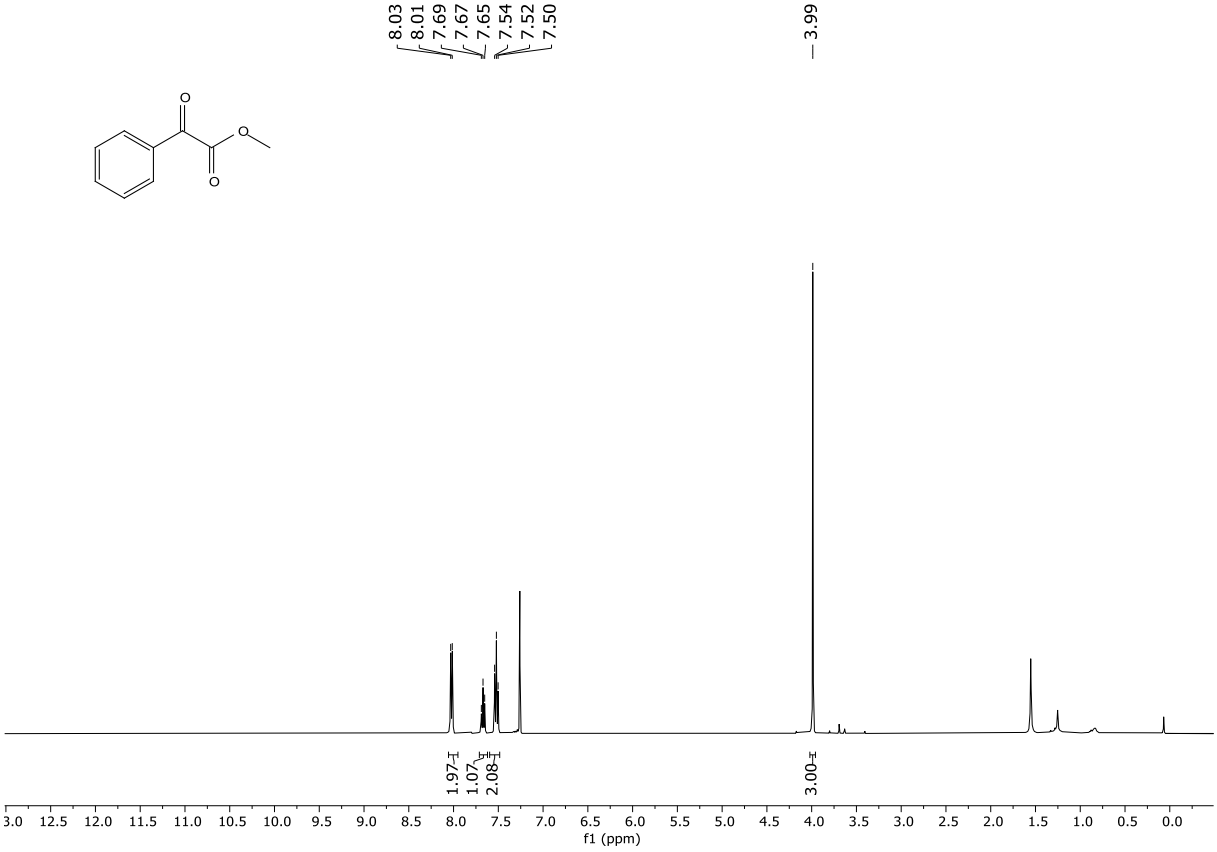
1-(4-Chlorophenyl)ethan-1-one (6p)



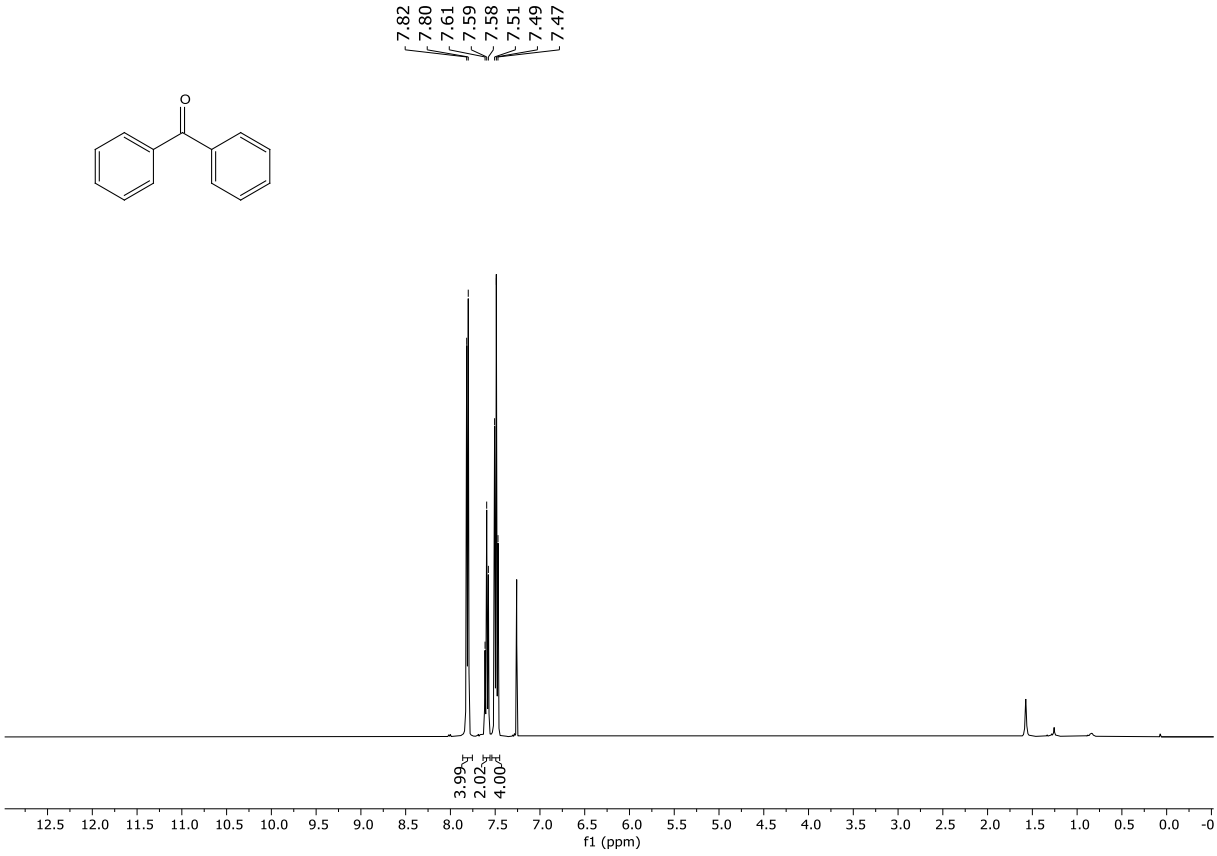
1-(4-Bromophenyl)ethan-1-one (6q)



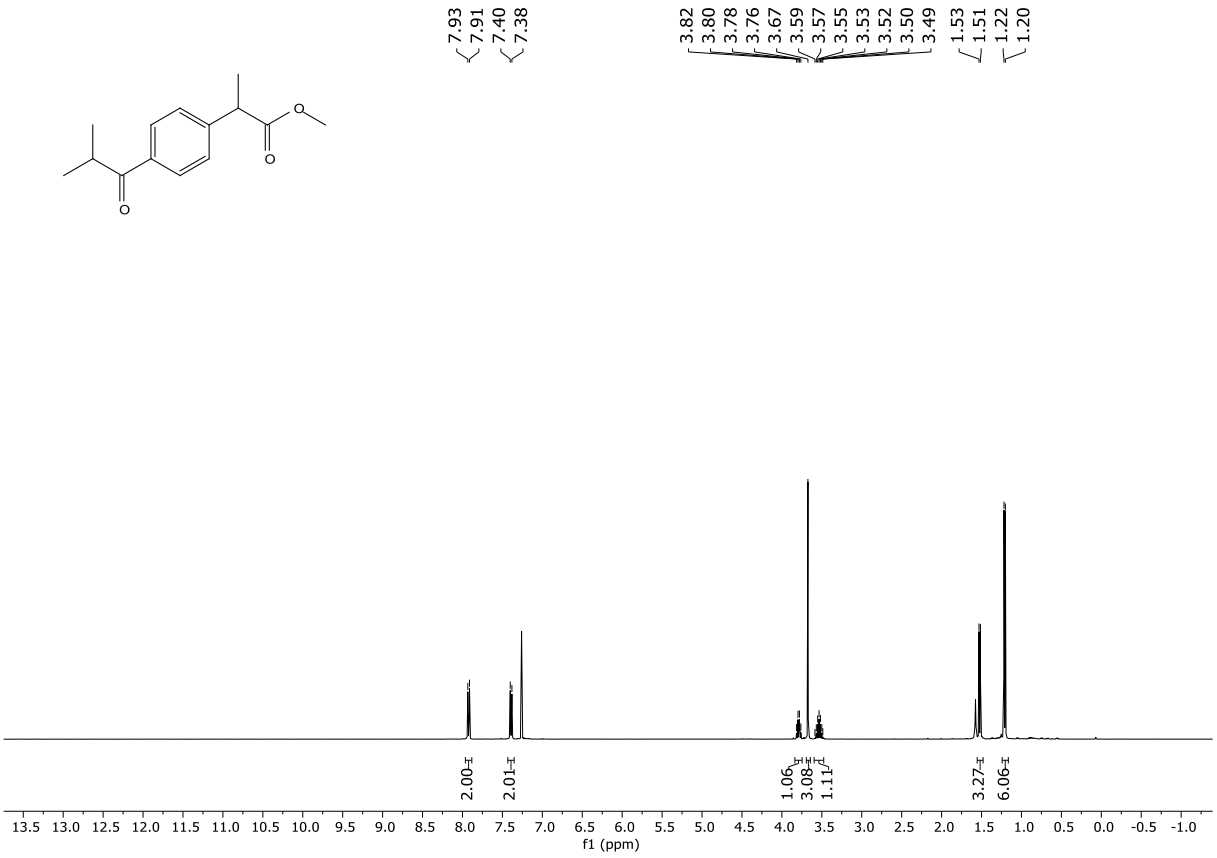
Methyl 2-oxo-2-phenylacetate (6r)



Benzophenone (6s)



Methyl 2-(4-isobutyrylphenyl)propanoate (6t)



(6,7-Dimethoxyisoquinolin-1-yl)(3,4-dimethoxyphenyl)methanone (6u)