Supporting Information

Chemoenzymatic One-Pot Reaction of Noncompatible Catalysts: Combining Enzymatic Ester Hydrolysis with Cu(I) /Bipyridine Catalyzed Oxidation in Aqueous Medium

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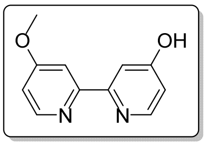
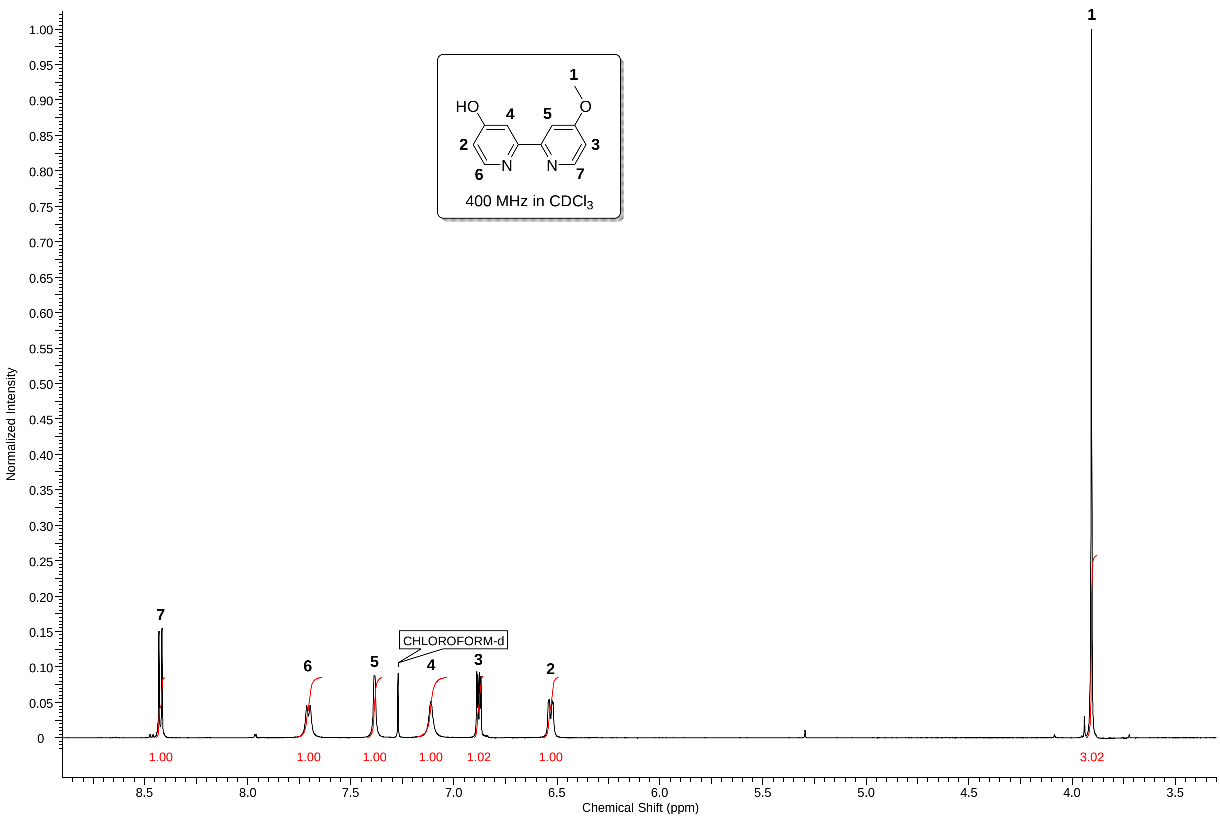
1. ^{1}H-, ^{13}C-NMR- and ESI-MS data of synthesized monomers

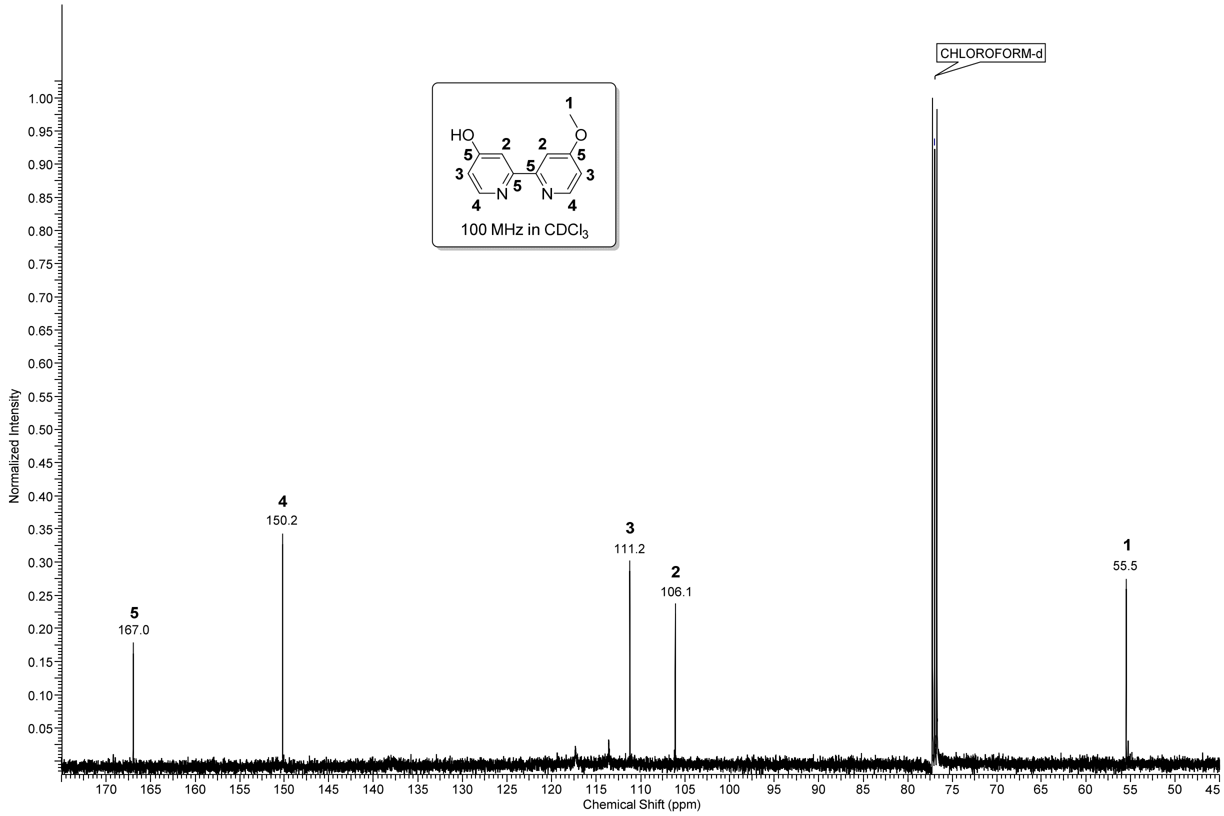
4'-Methoxy-[2,2'-bipyridin]-4-ol:

To a solution of 4,4’-dimethoxy-2,2’-bipyridine (500 mg, 2.31mmol, 1.0 eq.) in 30 ml absolute acetic acid was given 48% HBr_{aq} (0.31 ml, 2.77 mmol, 1.15 eq.) while stirring. After heating for reflux the solution was neutralized with aqueous ammonia solution up to pH 8–9 under ice bath cooling and stirred for 30 min at rt. The solution was extracted with dichloromethane (4 x 30 ml) and the organic layers were dried with MgSO4. The solvent was removed under reduced pressure and the residue was washed with a small amount of chloroform. 4'-Methoxy-[2,2'-bipyridin]-4-ol was yielded as white crystals (328 mg, 1.62 mmol, 70%).



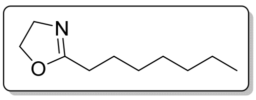
^{1}H-NMR (400 MHz, $CDCl_3$): δ = 3.91 ppm (s, C*H*3O), 6.53 (dd, *J* = 7.2 Hz, CHC*H*COCH3), 6.88 (dd, *J* = 5.8 Hz, CHC*H*COH), 7.11 (s, br, CC*H*COH), 7.39 (s, CC*H*COCH3), 7.71 (d, *J* = 7 Hz, C*H*CHCOH), 8.42 (d, *J* = 5.8 Hz, C*H*CHCOCH3): **^{13}C-NMR (100 MHz, $CDCl_3$):** δ = 55.5 ppm (*C*H3O), 106.1 (2 x C*C*HCOR), 111.2 (2 x CH*C*HCOR), 150.2 (2 x *C*HCHCOR), 167.0 (2 x N*C*CH, 2 x COR); **HR-ESI-MS**: $M_{calculated}$ = 202.0742 [M = $C_{11}H_{10}N_2O_2$]; $M_{measured}$ = 203.0816 $[M+H]^+$ Known compound.[S1]



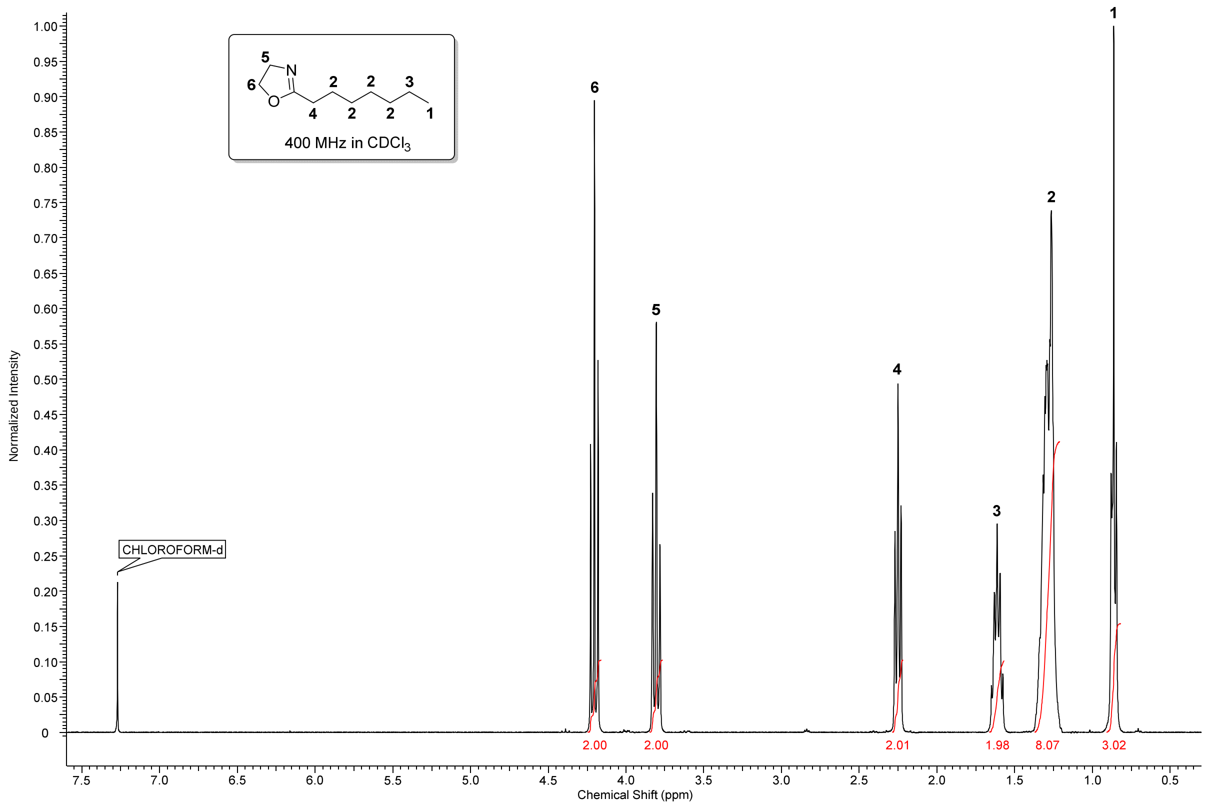


2-*n*-Heptyl-2-Oxazolin:

To octanonitrile (24.57 ml, 154.94 mmol, 1.0 eq.) ethanolamine (11.24 ml, 185.93 mmol, 1.20 eq.) and $Cd(OAc)_2{\cdot}2H_2O$ (825.91 mg, 3.10 mmol, 0.02 eq.) was added. The solution was stirred 30 h at 130 °C and the product was purified by distillation (88 °C, $1.6 \cdot 10^{-1}$ mbar). After addition of CaH_2 the product was distillated again and yielded as colorless liquid (15.5 g, 91.57 mmol, 60%).



^{1}H-NMR (400 MHz, $CDCl_3$): δ = 0.86 ppm (m, C*H₃*), 1.30 (m, 4xC*H₂*), 1.61 (quin, *J* = 7.4 Hz, $CH_3C\mathit{H_2}$), 2.25 (t, *J* = 7.7 Hz, $CC\mathit{H_2}$), 3.80 (t, *J* = 9.4 Hz, $C\mathit{H_2}N$), 4.20 (t, *J* = 9.5 Hz, $C\mathit{H_2}O$); **^{13}C-NMR (100 MHz, $CDCl_3$):** δ = 14.0 ppm ($\mathit{C}H_3$), 22.6 ($CH_3\mathit{C}H_2$), 25.9 ($\mathit{C}H_2CH_2CO$), 27.9 ($\mathit{C}H_2CH_2CH_2CH_3$), 28.9 ($\mathit{C}H_2CN$), 29.2 ($\mathit{C}H_2CH_2CH_2CN$), 31.6 ($\mathit{C}H_2CH_2CH_3$), 54.3 ($\mathit{C}H_2N$), 67.1 ($\mathit{C}H_2O$), 168.6 ($\mathit{C}O$); **HR-ESI-MS**: $M_{calculated}$ = 169.1467 [M = $C_{10}H_{19}NO$]; $M_{measured}$ = 170.1538 $[M+H]^+$ Known compound.[S2]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Chemical Shift (ppm)
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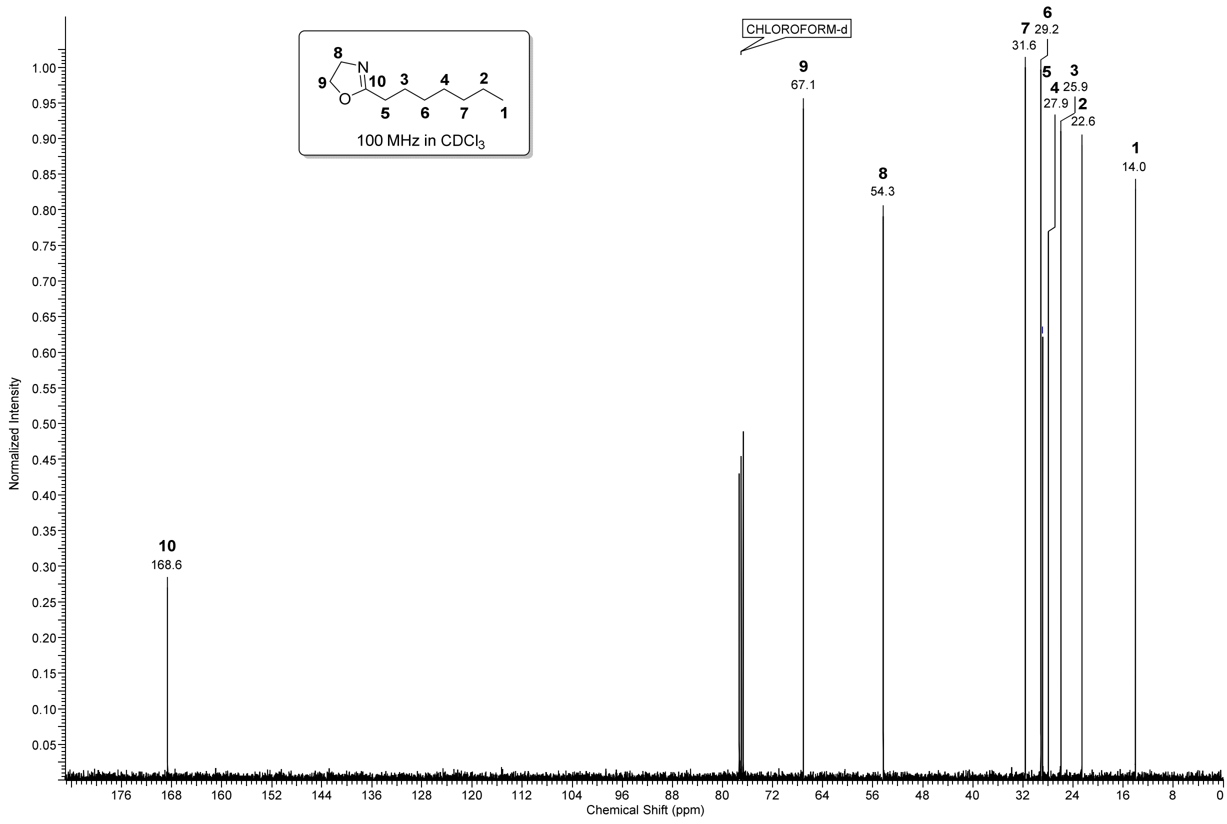
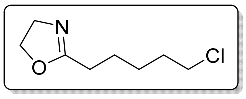
2.00
2.01
1.98
8.07
3.02

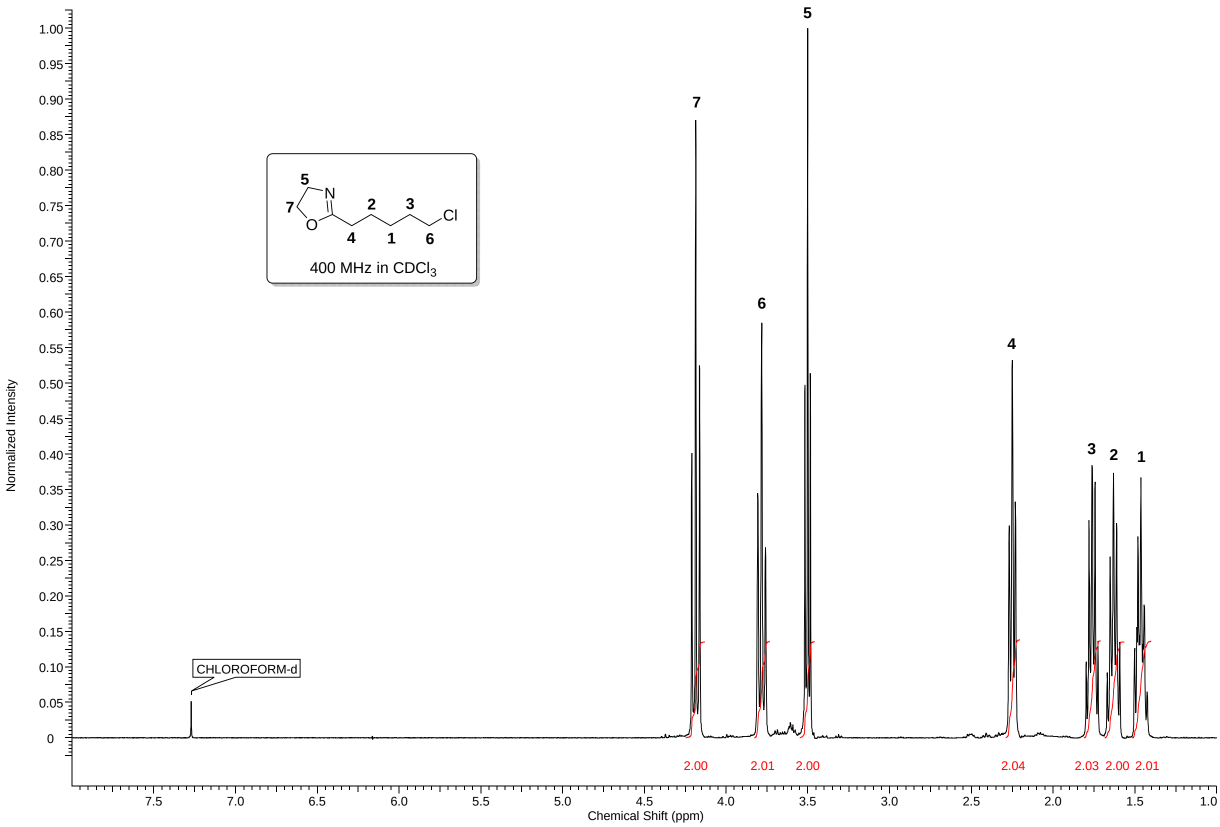

100 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
168.6
67.1
54.3
31.6
29.2
27.9
25.9
22.6
14.0

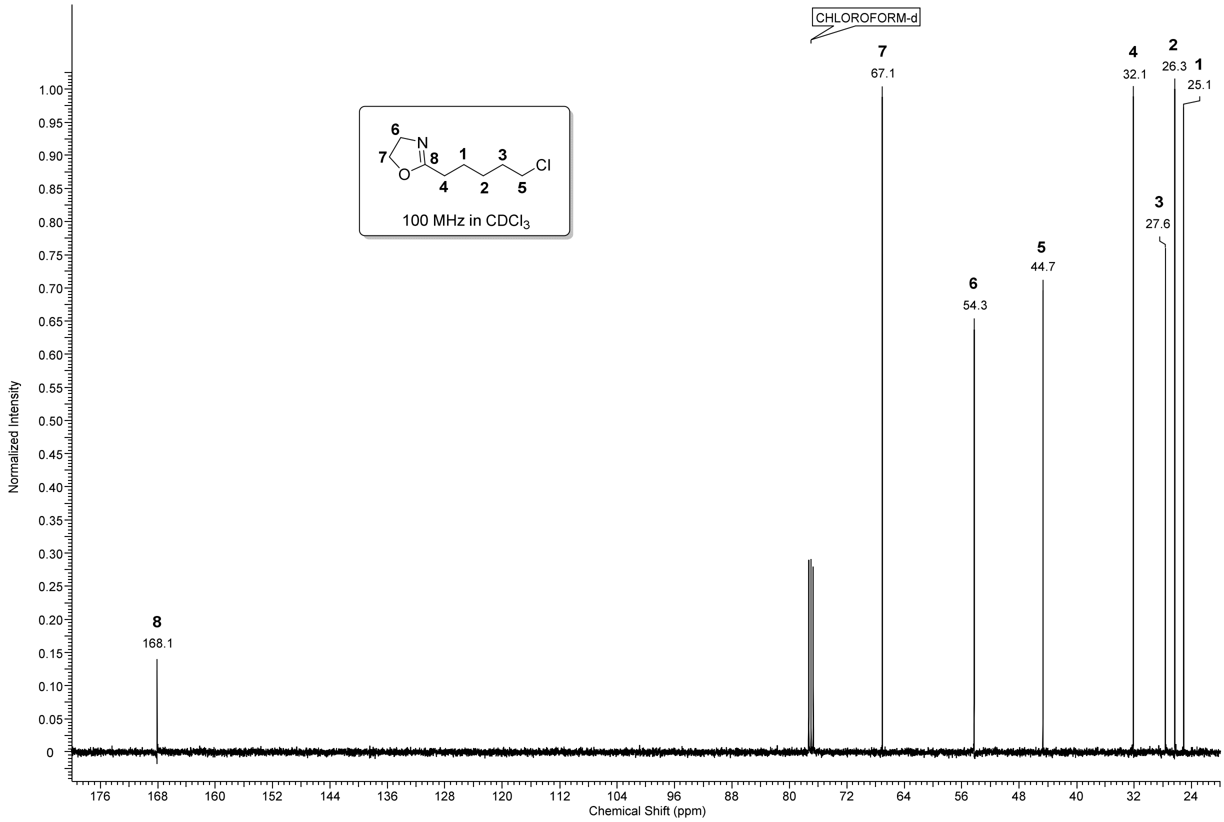
2-(5-Chlorpentyl)-2-oxazolin:

The synthesis was carried out according to *Litt et al.*[S15] Starting from 3-caprolactone (10 g, 87.61 mmol, 1.0 eq.) 2-(5-chlorpentyl)-2-oxazolin was obtained after three steps with an overall yield of 66%.



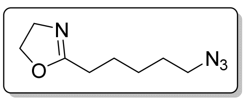
^{1}H-NMR (400 MHz, CDCl$_3$): δ = 1.47 pp (m, CH_2CH$_2$CH$_2$Cl), 1.63 (quin, J = 7.6 Hz, CH_2CH$_2$C(O)N), 1.76 (quin, J = 7.2 Hz, CH_2CH$_2$Cl), 2.25 (t, J = 7.5 Hz, CH_2C(O)N), 3.5 (t, J = 6.7 Hz, NCH_2CH$_2$O), 3.78 (t, J = 9.7 Hz, CH_2Cl), 4.19 (t, J = 9.5 Hz, NCH$_2$CH_2O); **^{13}C-NMR (100 MHz, CDCl$_3$):** δ = 25.1 ppm (*C*H$_2$CH$_2$C(O)N), 26.3 (*C*H$_2$CH$_2$CH$_2$C(O)N), 27.6 (CH$_2$*C*H$_2$CH$_2$Cl), 32.1 (*C*H$_2$C(O)N), 44.7 (*C*H$_2$N), 54.3 (CH$_2$*C*H$_2$*C*H$_2$Cl), 67.1 (NCH$_2$*C*H$_2$O), 168.1 (*C*(N)O); **HR-ESI-MS**: $M_{calculated}$ = 175.0764 [M = $C_8H_{14}ClNO$]; $M_{measured}$ = 176.0838 $[M+H]^+$ Known compound.[S1]



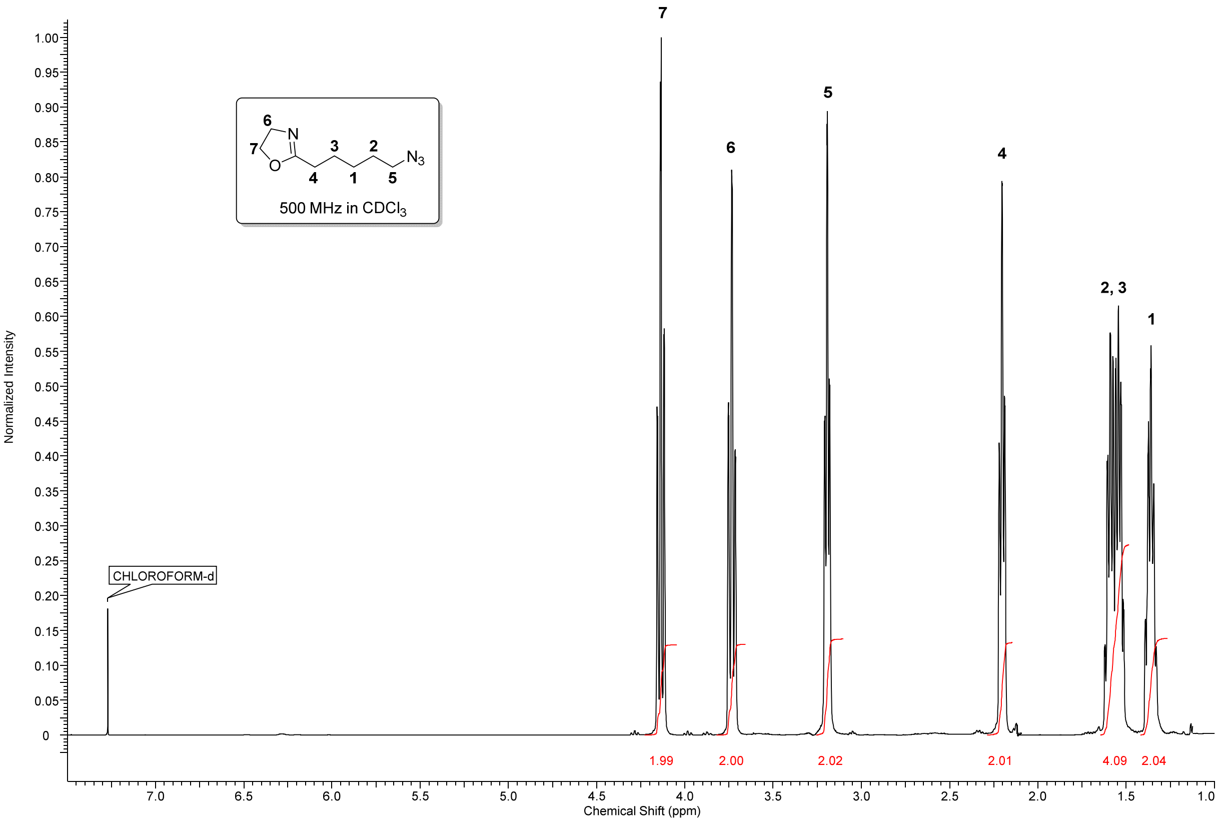


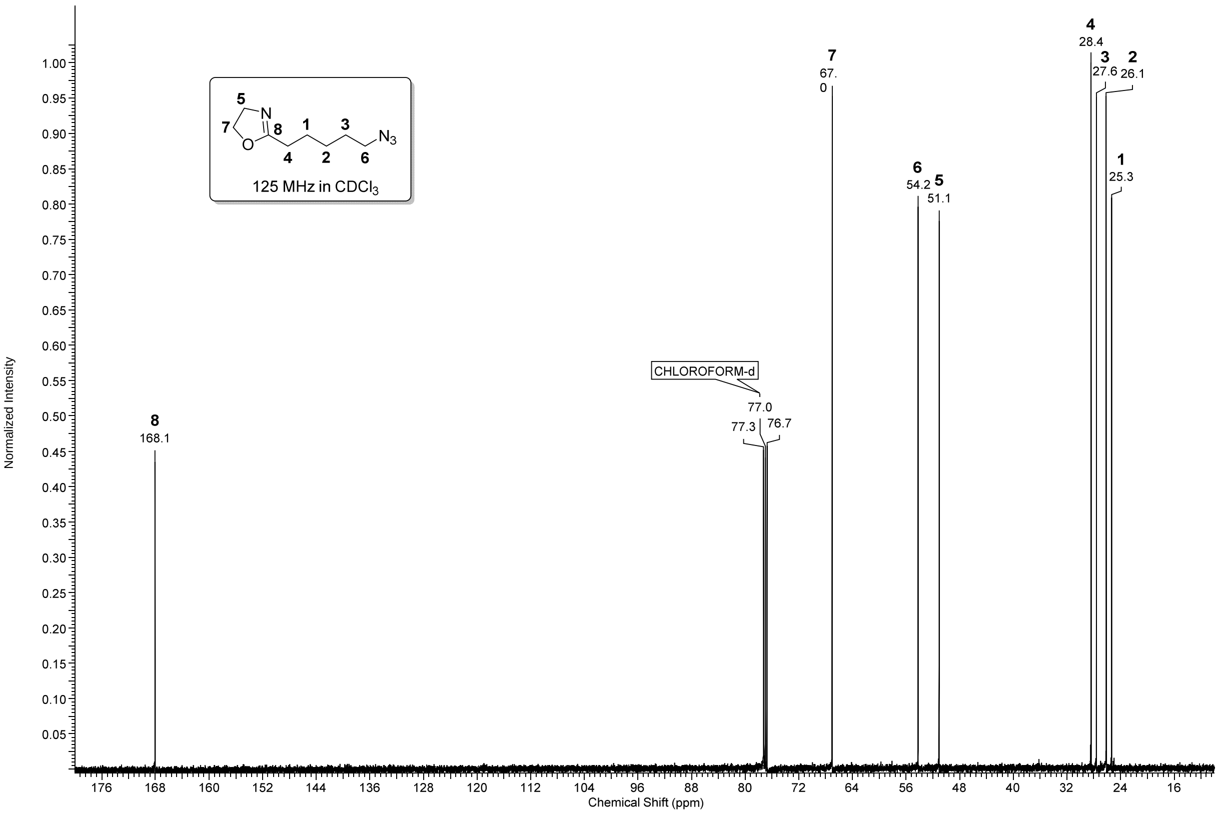
2-(5-Azidopentyl)-2-oxazolin:

The synthesis was carried out according to *Lav et al.*[S3] Starting from 6-bromohexanoic acid (25 g, 128.17 mmol, 1.0 eq) 2-(5-azidopentyl)-2-oxazolin was obtained after three steps with an overall yield of 43%.

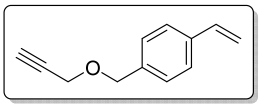


^{1}H-NMR (500 MHz, CDCl$_3$): δ = 1.28 - 1.42ppm (m, $CCH_2CH_2C\mathit{H_2}$), 1.48 - 1.64 (m, $CCH_2C\mathit{H_2}CH_2C\mathit{H_2}CH_2N_3$), 2.20 (td, J = 7.46/1.22 Hz, $CC\mathit{H_2}$), 3.19 (td, J = 6.85/1.47 Hz, $C\mathit{H_2}N_3$), 3.66 - 3.82 (m, $C\mathit{H_2}O$), 4.14 (td, J = 9.42/1.71 Hz, $C\mathit{H_2}N$); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 25.3 ppm ($\mathit{C}H_2CH_2C(O)N$), 26.1 ($\mathit{C}H_2CH_2CH_2C(O)N$), 27.6 ($CH_2\mathit{C}H_2CH_2N_3$), 28.4 ($\mathit{C}H_2C(O)N$), 51.1 ($\mathit{C}H_2N$), 54.2 ($CH_2\mathit{C}H_2\mathit{C}H_2N_3$), 67.0 ($NCH_2\mathit{C}H_2O$), 168.1 ($\mathit{C}(O)N$); **HR-ESI-MS**: $M_{calculated}$ = 182.1168 [M = $C_8H_{14}N_4O$]; $M_{measured}$ = 183.1246 $[M+H]^+$ Known compound.[S3]

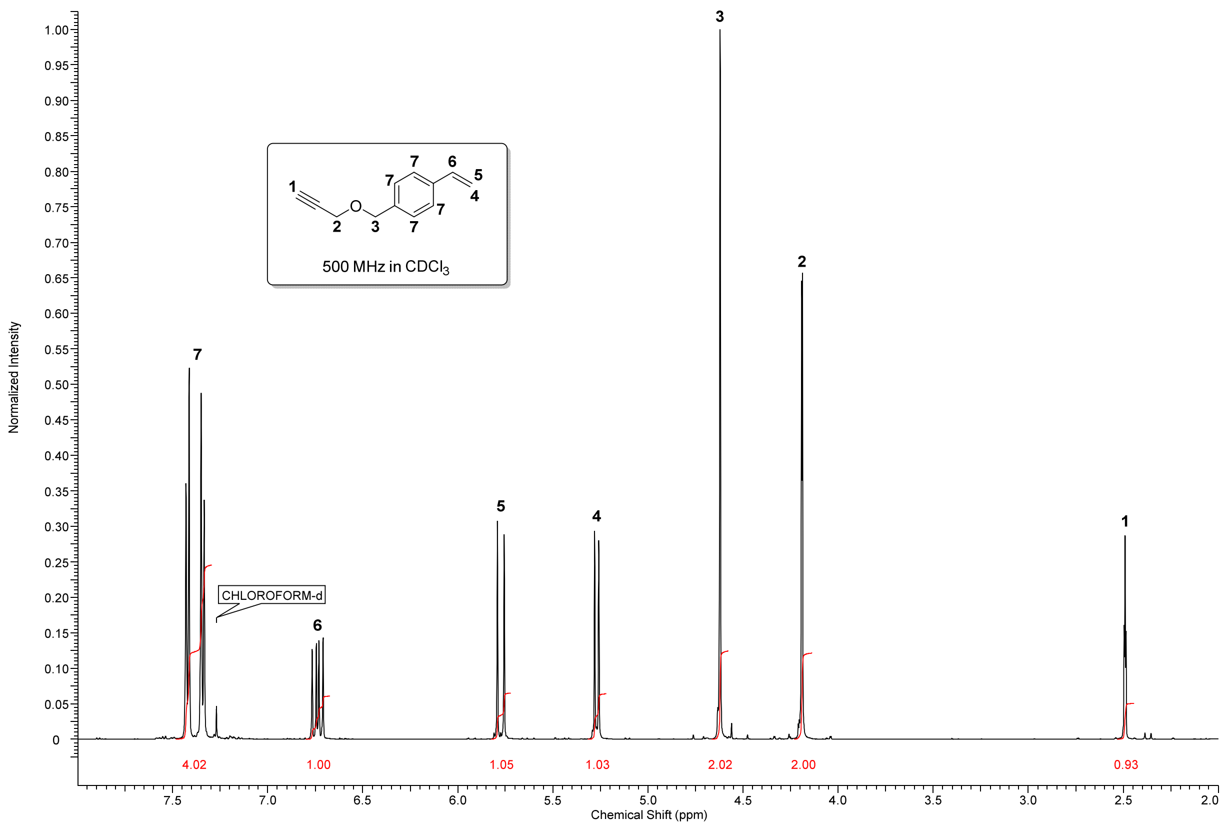

500 MHz in CDCl3
CHLOROFORM-d
7
6
5
4
2, 3
1
1.99
2.00
2.02
2.01
4.09
2.04
Chemical Shift (ppm)
Normalized Intensity

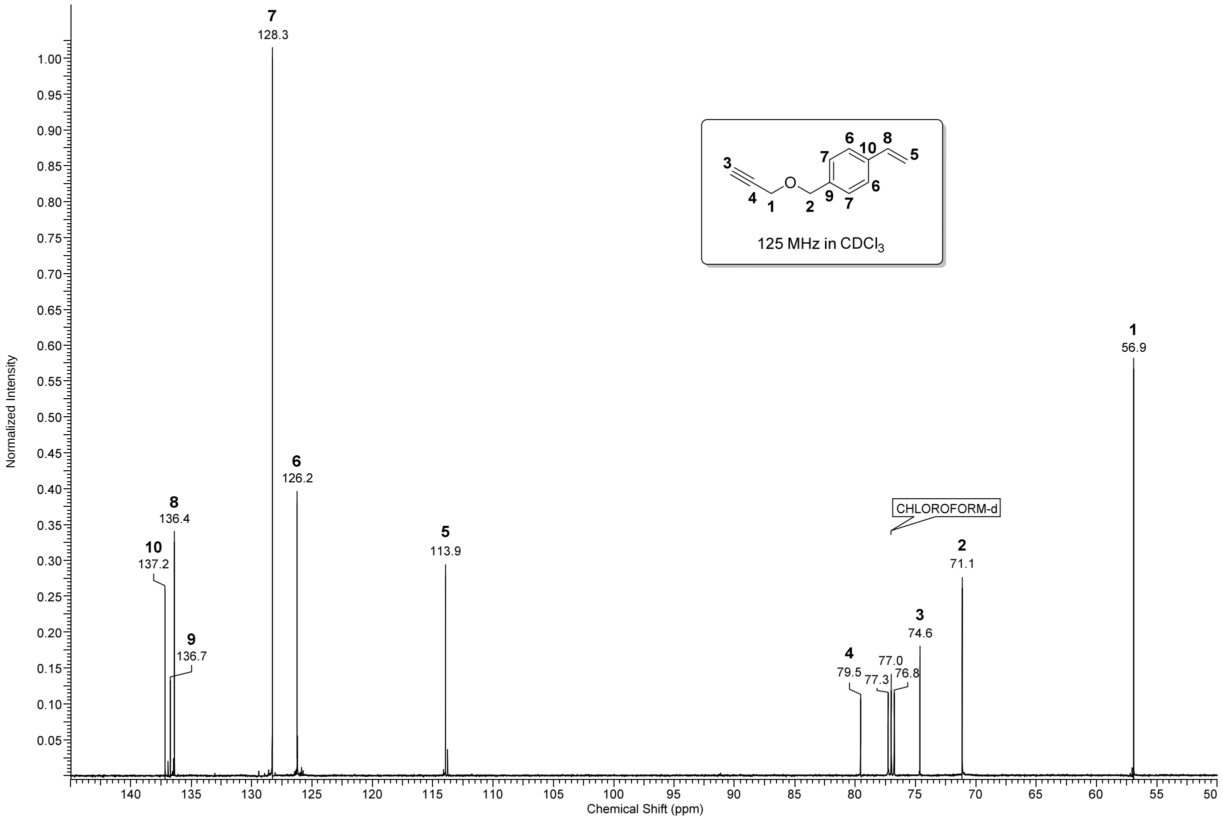

125 MHz in CDCl3
8
168.1
CHLOROFORM-d
77.0
77.3
76.7
7
67.0
6
54.2
5
51.1
4
28.4
3
27.6
2
26.1
1
25.3
Chemical Shift (ppm)
Normalized Intensity

1-((Prop-2-yn-1-yloxy)methyl)-4-vinylbenzene:

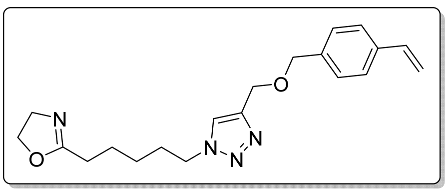


^{1}H-NMR (500 MHz, $CDCl_3$): δ = 2.49 ppm (t, J = 2.2 Hz, C*H*C), 4.19 (d, J = 2.4, CHCC*H₂*), 4.63 (s, OC*H₂*Ar), 5.27 (d, J = 10.8 Hz, C*H₂*CH), 5.77 (d, J = 17.6 Hz, C*H₂*CH), 6.74 (dd, J = 17.6 Hz/11.2 Hz, CH_2C*H*), 7.31-7.45 (m, *Ar*); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 56.9 (*C*H_2CCH), 71.1 (O*C*H_2Ar), 74.6 (CH_2C*C*H), 79.5 (CH_2*C*CH), 113.9 (*C*H_2CH), 126.2 (2xCHC*C*H), 128.3 (2xCH_2C*C*H), 136.4 (CH_2*C*H), 136.7 (CH_2*C*(Ar)), 137.2 (CH*C*(Ar)). Known compound.[S4]

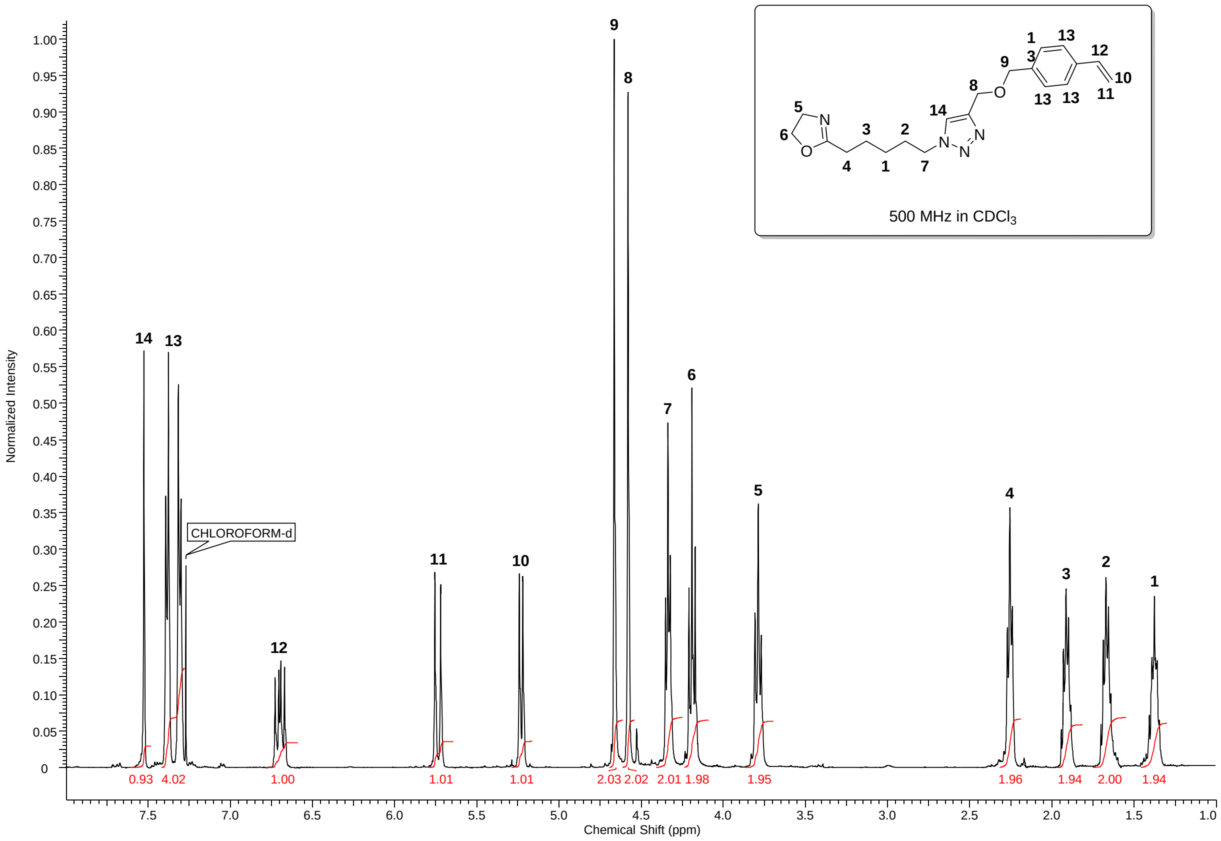




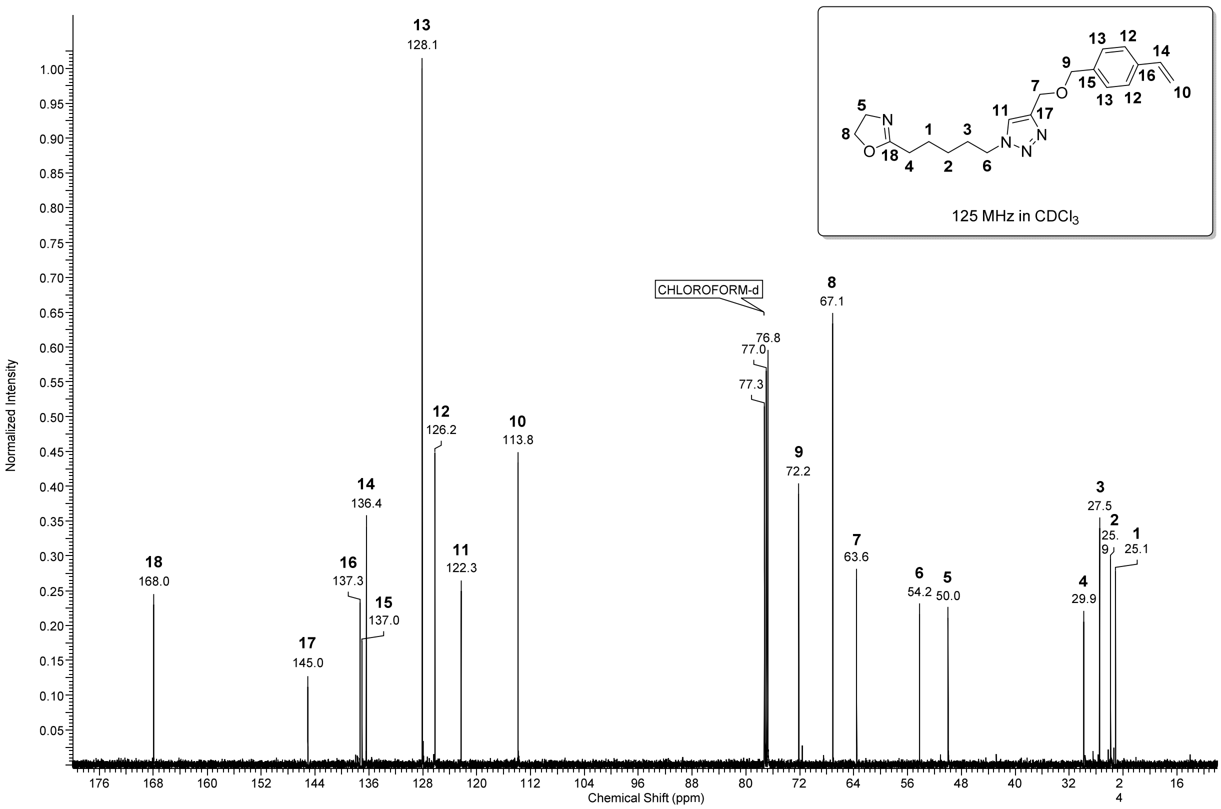
2-(5-(4-(((4-Vinylbenzyl)-oxy)-methyl)-1H-1,2,3-triazol-1-yl)pentyl)-2-oxazolin:



^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.32 - 1.42 (m, $CCH_2CH_2C\mathit{H_2}$), 1.62 - 1.71 (m, $CCH_2CH_2CH_2C\mathit{H_2}$), 1.86 - 1.96 (m, $CCH_2C\mathit{H_2}$), 2.26 (t, *J* = 7.48 Hz, $CC\mathit{H_2}CH_2$), 3.79 (t, *J*=9.46 Hz, $OC\mathit{H_2}CH_2N$), 4.18 (td, *J* = 9.42/1.71 Hz, $OCH_2C\mathit{H_2}N$), 4.28 - 4.38 (m, $C\mathit{H_2}NN$), 4.58 (s, $CHCC\mathit{H_2}$), 4.66 (s, $OC\mathit{H_2}Ar$), 5.23 (d, *J* = 10.8 Hz, $C\mathit{H_2}CH$), 5.74 (dd, *J* = 17.70, 0.61 Hz, $C\mathit{H_2}CH$), 6.70 (dd, *J* = 17.6 Hz/11.2 Hz, $CH_2C\mathit{H}$), 7.28 - 7.42 (m, *Ar*), 7.53 (s, $C\mathit{H}N$); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 25.1 ppm ($\mathit{C}H_2CH_2C(O)N$), 25.9 ($\mathit{C}H_2CH_2CH_2C(O)N$), 27.5 ($CH_2\mathit{C}H_2CH_2N$), 29.9 ($\mathit{C}H_2C(O)N$), 50.0 ($\mathit{C}H_2N$), 54.2 ($CH_2CH_2\mathit{C}H_2N$), 63.6 ($O\mathit{C}H_2CN$), 67.1 ($NCH_2\mathit{C}H_2O$), 72.2 ($O\mathit{C}H_2Ar$), 113.8 ($\mathit{C}H_2CH$), 122.3 ($\mathit{C}HN$), 126.2 (2x$CHC\mathit{C}H$), 128.1 (2x$CH_2C\mathit{C}H$), 136.4 ($CH_2\mathit{C}H$), 137.0 ($CH_2\mathit{C}$(Ar)), 137.3 ($CH\mathit{C}$(Ar)), 145.0 ($OCH_2\mathit{C}N$), 168.0 ($\mathit{C}(O)N$); **HR-ESI-MS**: $M_{calculated}$ = 354.2056 [M = $C_{20}H_{26}N_4O_2$]; $M_{measured}$ = 355.2136 $[M+H]^+$

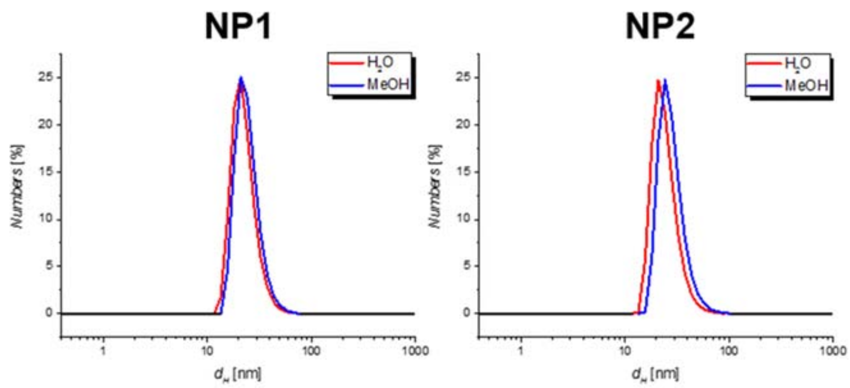


500 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)

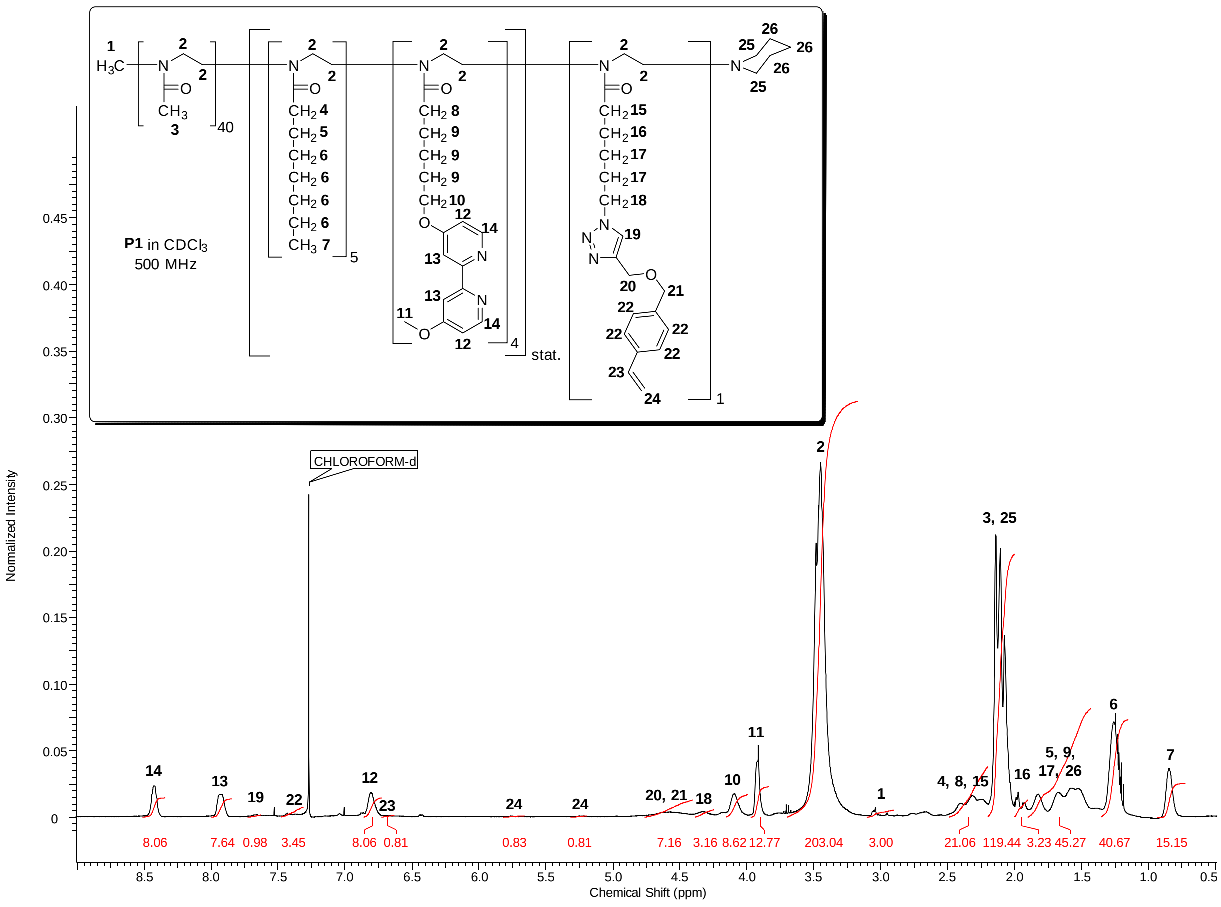


125 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)

2. Fig. S1. DLS size graph of **NP1** and **NP2** in methanol and water. 1mM solution at room temperature.

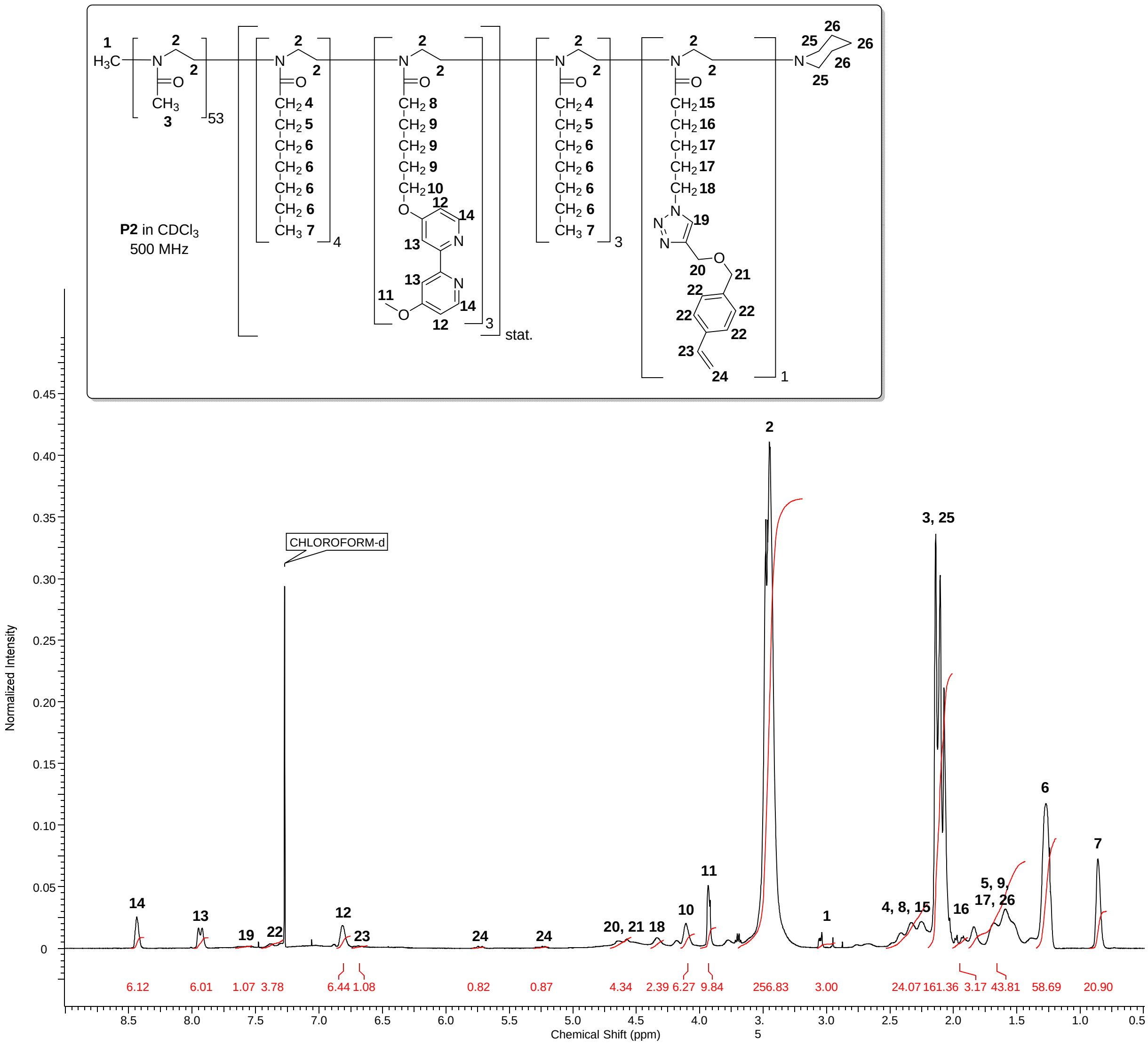


3. Fig S2. ^{1}H-NMR data of **P1** in $CDCl_3$.



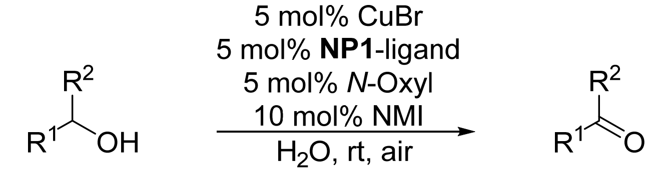
[1]H-NMR (500 MHz, $CDCl_3$): δ = 0.85 ppm (s(br), $CH_{3(HepOx)}$), 1.26 (s(br), 4x$CH_{2(HepOx)}$), 1.43 – 1.89 (m, $CH_{2(HepOx)}$, 3x$CH_{2(BiPyOx)}$, 2x$CH_{2(StyOx)}$, 3x$CH_{2(Pip)}$), 1.89 – 2.06 (s(br), $CH_{2(StyOx)}$), 2.06 – 2.20 (m, $CH_{3(MeOx)}$, 2x$CH_{2(Pip)}$), 2.20 – 2.50 (m, $CH_{2(HepOx)}$, $CH_{2(BiPyOx)}$, $CH_{2(StyOx)}$), 2.94/3.01 ($CH_{3(In)}$), 3.19 – 3.75 (m, CH_2-$CH_{2(backbone)}$), 3.93 (s(br), O$CH_{3(BiPyOx)}$), 4.10 (s(br), O$CH_{2(BiPyOx)}$), 4.34 (s(br), $CH_{2(StyOx)}$), 4.59 – 4.71 (m, 2x$CH_{2(StyOx)}$), 5.20 – 5.26 (m, $CH_{(StyOx)}$), 5.68 – 5.79 (m, $CH_{(StyOx)}$), 6.65 – 6.74 (m, $CH_{(StyOx)}$), 6.82 (s(br), 2x$CH_{(BiPyOx)}$), 7.28 – 7.43 (m, 4x$CH_{Ar(StyOx)}$), 7.49 – 7.65 (s(br), $CH_{Triazol(StyOx)}$), 7.94 (s(br), 2x$CH_{(BiPyOx)}$),), 8.44 (s(br), 2x$CH_{(BiPyOx)}$).

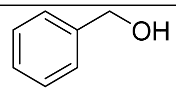
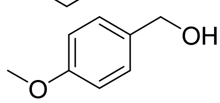
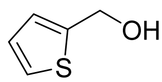
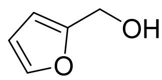
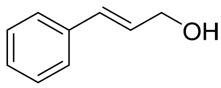
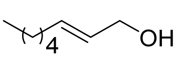
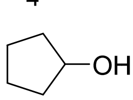
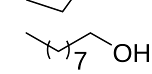
4. Fig S3. [1]H-NMR data of **P2** in $CDCl_3$.



^{1}H-NMR (500 MHz, CDCl$_3$): δ = 0.85 ppm (s(br), C$H_{3(\text{HepOx})}$), 1.25 (s(br), 4xC$H_{2(\text{HepOx})}$), 1.45 – 1.89 (m, C$H_{2(\text{HepOx})}$, 3xC$H_{2(\text{BiPyOx})}$, 2xC$H_{2(\text{StyOx})}$, 3xC$H_{2(\text{Pip})}$), 1.89 – 2.01 (s(br), C$H_{2(\text{StyOx})}$), 2.01 – 2.20 (m, C$H_{3(MeOx)}$, 2xC$H_{2(Pip)}$), 2.20 – 2.54 (m, C$H_{2(\text{HepOx})}$, C$H_{2(\text{BiPyOx})}$, C$H_{2(\text{StyOx})}$), 2.94/3.01 (C$H_{3(\text{In})}$), 3.18 – 3.70 (m, CH_2-C$H_{2(\text{backbone})}$), 3.92 (s(br), OC$H_{3(\text{BiPyOx})}$), 4.10 (s(br), OC$H_{2(\text{BiPyOx})}$), 4.34 (s(br), C$H_{2(\text{StyOx})}$), 4.52 – 4.72 (m, 2xC$H_{2(\text{StyOx})}$), 5.19 – 5.28 (m, C$H_{(\text{StyOx})}$), 5.69 – 5.79 (m, C$H_{(\text{StyOx})}$), 6.64 – 6.74 (m, C$H_{(\text{StyOx})}$), 6.82 (s(br), 2xC$H_{(\text{BiPyOx})}$), 7.28 – 7.42 (m, 4xC$H_{\text{Ar(StyOx)}}$), 7.49 – 7.65 (s(br), C$H_{\text{Triazol(StyOx)}}$), 7.93 (d(br), 2xC$H_{(\text{BiPyOx})}$),), 8.44 (s(br), 2xC$H_{(\text{BiPyOx})}$).

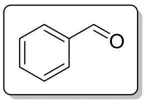
5. Table S1. Representative examples of functionalized alcohols in the micellar Cu(I) / *N*-oxyl catalysed aerobic oxidation with **P1** as polymeric ligand.[a]



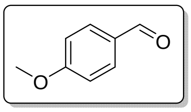
entry	substrate	*N*-oxyl	*t* [h]	conversion[b] [%]	yield[c] [%]
1a		ABNO	2	95 (± 3)	92
1b		TEMPO	3	99 (± 0)	96
2a		ABNO	2	96 (± 3)	91
2b		TEMPO	3	94 (± 4)	90
3a		ABNO	2	90 (± 3)	85
3b		TEMPO	3	62 (± 2)	56
4a		ABNO	2	79 (± 3)	72
4b		TEMPO	3	58 (± 0)	54
5a		ABNO	2	91 (± 3)	89
5b		TEMPO	3	87 (± 4)	84
6a		ABNO	2	79 (± 3)	71
6b		TEMPO	3	73 (± 2)	64
7a		ABNO	2	44 (± 2)	37
7b		TEMPO	3	3 (± 1)	n. d.
8a		ABNO	2	67 (± 2)	59
8b		TEMPO	3	9 (± 1)	5

[a] 40 mol of CuBr, 5 mM solution of **P1**; [b] average conversion (3 runs) after work-up and isolation determined by ^{1}H-NMR-spectroscopy; [c] average isolated yield (3 runs) after purification.

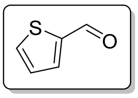
5.1 ^{1}H- and ^{13}C-NMR data of the synthesized aldehydes.



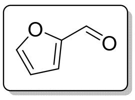
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.52 – 7.57 ppm (m, 2xCHC*H*), 7.63 – 7.67 (m, CHCHC*H*), 7.88 – 7.90 (m, 2xCC*H*), 10.03 (s, OC*H*); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 128.9 ppm (2xCCHC*H*), 129.7 (2xC*C*HCH), 134.4 (CCHCHC*H*), 136.3 (*C*CH), 192.4 (H*C*O). Known compound.[S5]



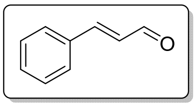
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 3.88 ppm (s, 3H, OCH_3), 7.00 (d, *J* = 8.0 Hz, 2xCHCC*H*), 7.83 (d, *J* = 8.0 Hz, 2xOCC*H*), 9.87 (s, OC*H*); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 55.5 ppm (O*C*H3), 114.2 (2xOC*C*H), 129.8 (2xCHC*C*HCH), 131.9 (CH*C*CHCH), 164.5 (O*C*CH), 190.8 (H*C*O). Known compound.[S5]



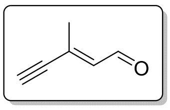
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 7.23 ppm (dd, *J* = 4.8, 3.9 Hz, CC*H*), 7.76 - 7.81 (m, SC*H*C*H*), 9.94 (s, OC*H*); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 128.3 ppm (S*C*HCH), 135.1 (C*C*HCH), 136.3 (SCH*C*H), 143.9 (*C*S), 183.0 (H*C*O). Known compound.[S5]



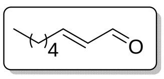
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 6.63 – 6.64 ppm (m, CC*H*), 7.28 – 7.30 (m, CCHC*H*), 7.72 – 7.75 (m, OC*H*CH), 9.68 (s, CC*H*O); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 127.9 (O*C*HCH), 134.8 (C*C*HCH), 136.1 (OCH*C*H), 142.7 (*C*OCH), 182.6 (H*C*O). Known compound.[S6]



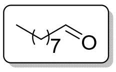
^{1}H-NMR (400 MHz, $CDCl_3$): δ = 6.73 ppm (dd, *J* = 15.9, 7.5 Hz, OCHC*H*), 7.38 - 7.53 (m, 2xCC*H*C*H*), 7.58 (m, C*H*CHCHCC*H*), 9.72 (d, *J* = 7.65 Hz, OC*H*); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 128.4 ppm (CCHCHC*H*), 128.4 (2xC*C*H*C*HCH), 129.0 (CCH*C*HC(H)O), 131.2 (*C*CHCHC(H)O), 152.8 (C*C*HCHC(H)O), 193.8 (H*C*O). Known compound.[S6]



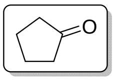
^{1}H-NMR (400 MHz, $CDCl_3$): δ = 2.30 (m, CH_3), 3.69 (s, CCC*H*), 6.29 (dd, *J* = 8.2, 1.6 Hz, OCHC*H*), 10.02 (d, *J* = 8 Hz, OC*H*); **^{13}C-NMR (100 MHz, $CDCl_3$):** δ = 24.6 ppm (CH_3), 80.1 (*C*HCC), 88.2 (CH*C*C), 136.8 (*C*HC(H)O), 140.9 (CHC*C*), 192.3 (H*C*O). Known compound.[S7]



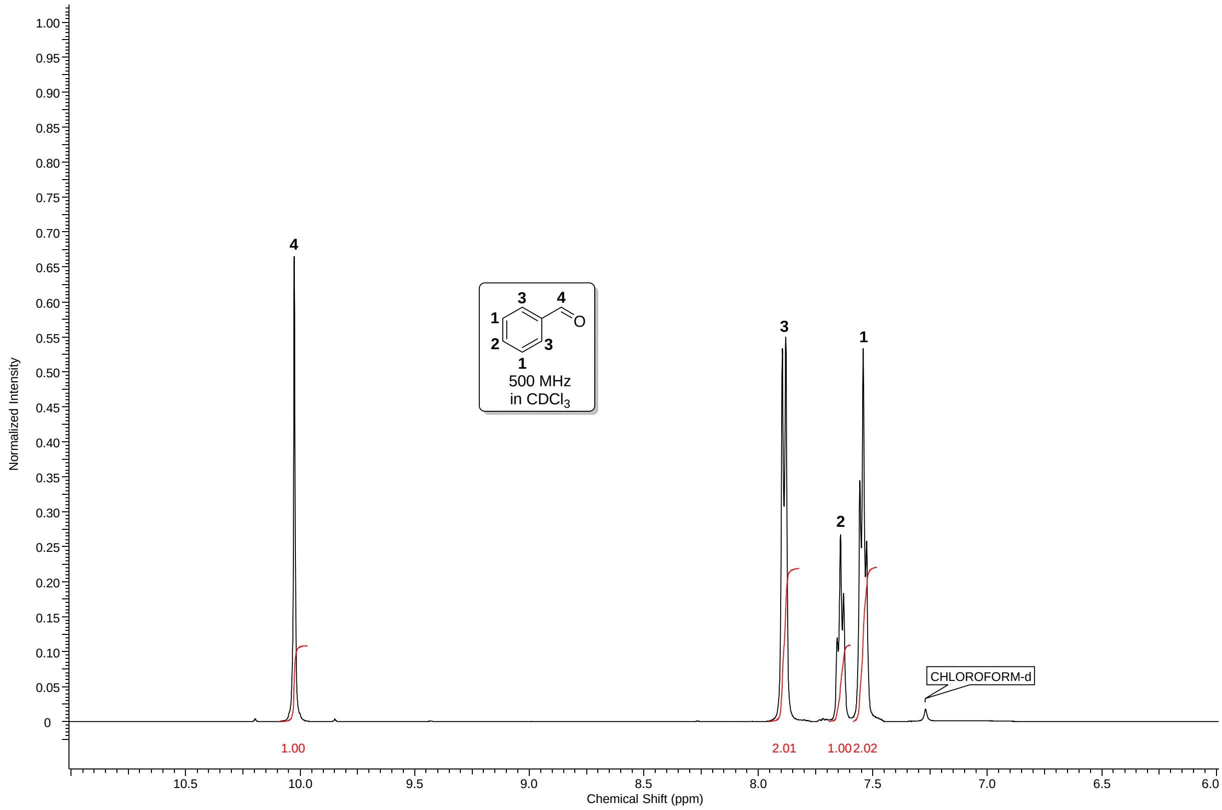
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 0.84 ppm (t, *J* = 6.4 Hz, CH_2CH_3), 1.20 – 1.36 (m, $CH_3CH_2CH_2CH_2$), 1.40 – 1.52 (m, CH_3CH_2), 2.28 (q, *J* =7 Hz, $CHCH_2CH_2$), 6.50 (dd, *J* = 15.6 Hz, 7.9 Hz, CH_2CH_2CH), 6.82 (dt, *J* = 15.2, 6.6 Hz, OCHC*H*), 9.44 (d, *J* = 8.1 Hz, OC*H*); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 14.0 ppm (CH_3), 22.5 (CH_2CH_3), 28.3 ($CH_2CH_2CH_2CH_3$), 31.3 ($CH_2CH_2CH_3$), 32.6 ($CHCH_2$), 132.9 (CH*C*HC(H)O), 158.9 (*C*HCHC(H)O), 193.9 (H*C*O). Known compound.[S8]

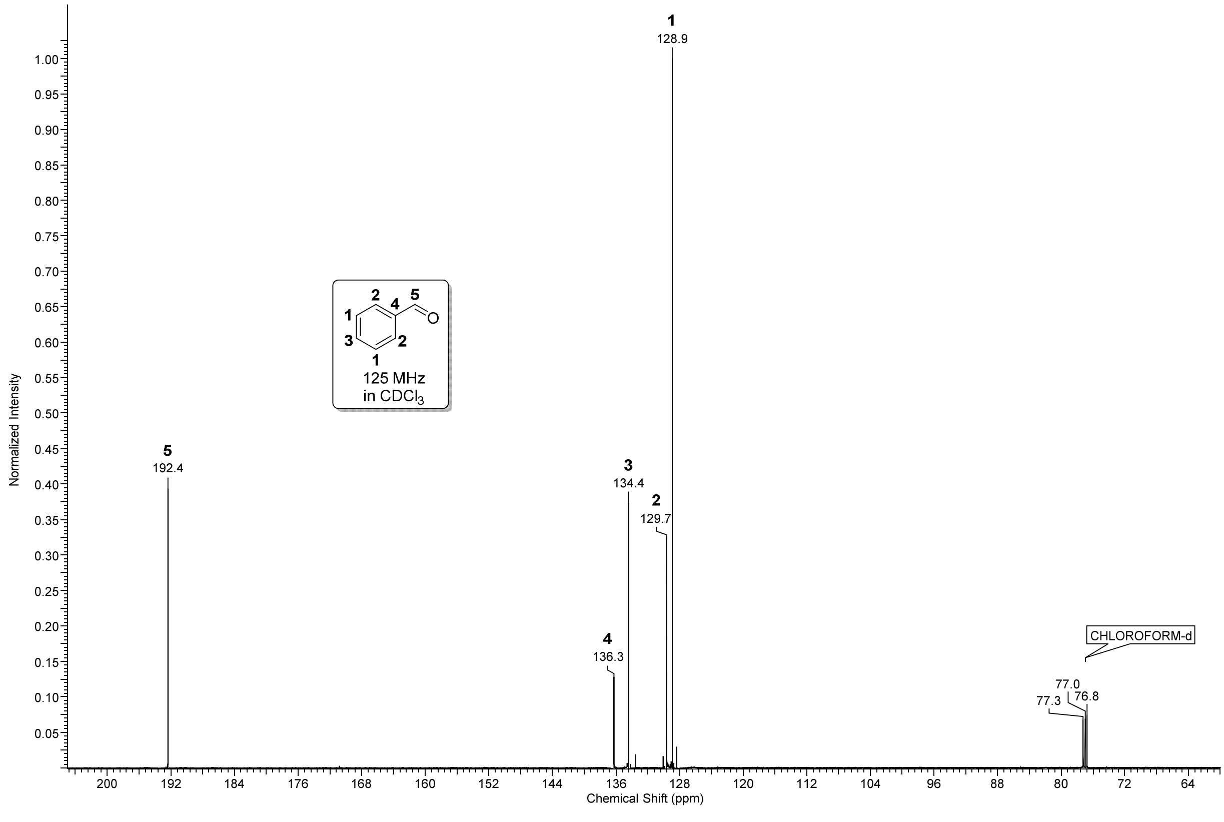


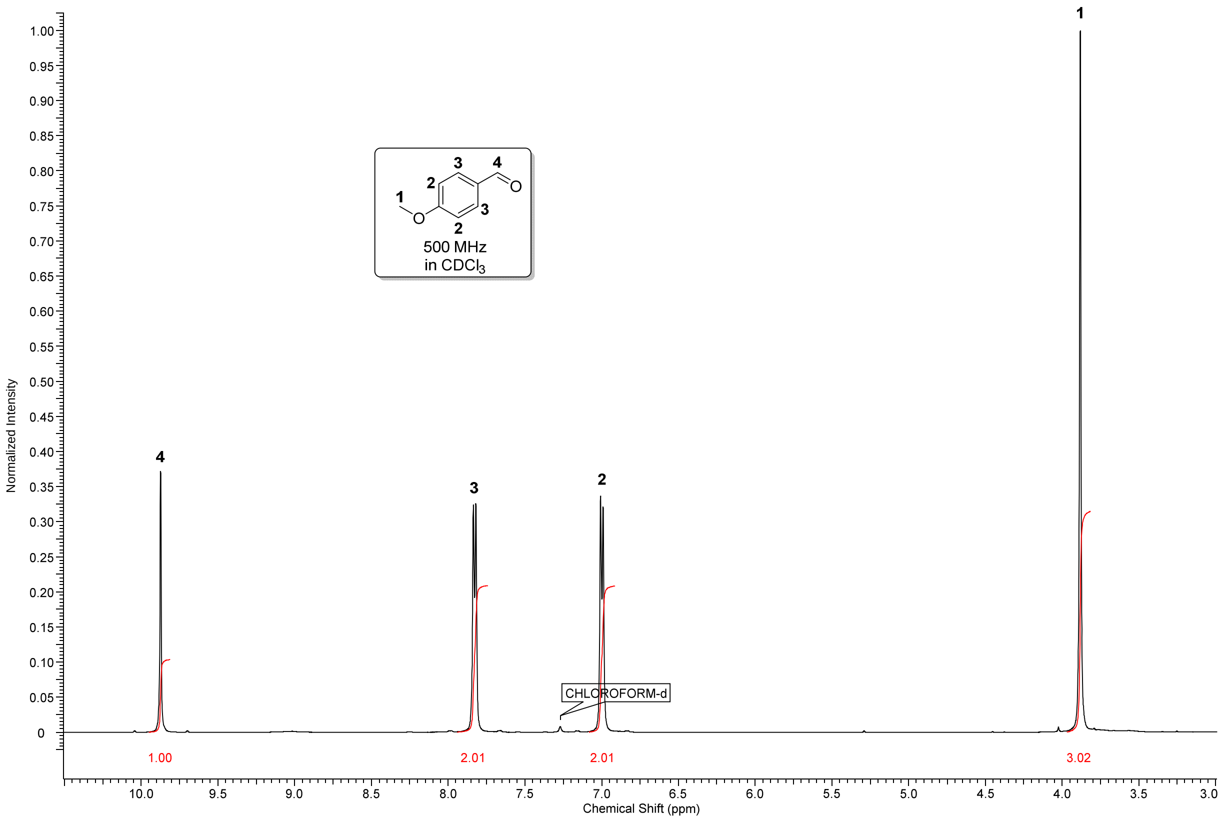
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 0.89 ppm (t, J = 6.9 Hz, CH_3), 1.18 – 1.45(m, $CH_3CH_2CH_2CH_2CH_2CH_2$), 1.58 (t, J = 7.4 Hz, OCH_2CH_2), 2.43 (dt, J = 7.3, 1.9, $CHCH_2$), 9.74 (t, J = 1.9 Hz, OCH); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 14.0 ppm (CH_3), 22.6 (CH_2CH_3), 25.9 ($O(H)CCH_2CH_2$), 29.1 ($CH_2CH_2CH_2CH_2CH_2CH_3$), 29.3 ($CH_2CH_2CH_2CH_3$), 31.3 ($CH_2CH_2CH_3$), 44.0 ($CH_2C(H)O$), 203.2 (HCO). Known compound.[S8]



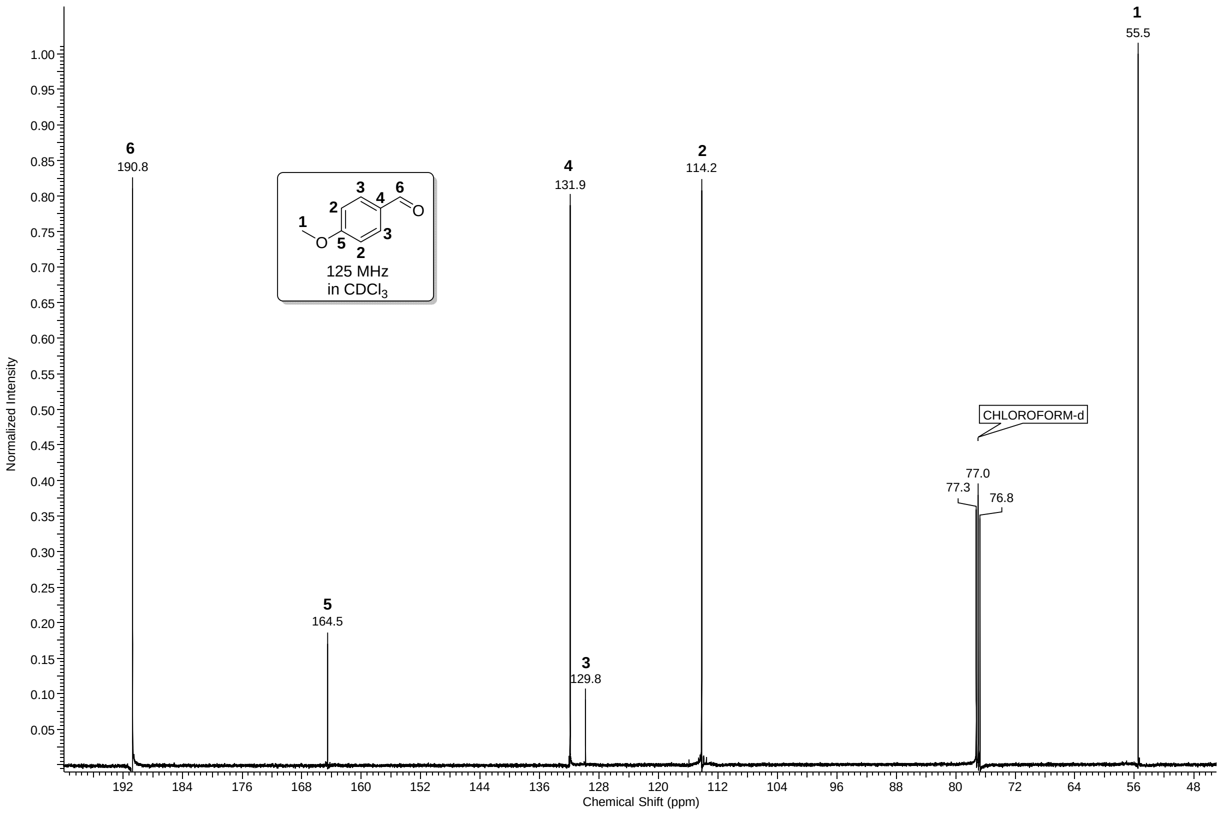
^{1}H-NMR (500 MHz, $CDCl_3$): δ = 1.84 – 1.95 ppm (m, 2xCH_2CH_2C), 2.04 – 2.16 (m, 2xCH_2C); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 23.1 ppm (2xCH_2CH_2C), 38.2 (2xCH_2CH_2C), 220.5 (C). Known compound.[S9]


Normalized Intensity
500 MHz
in $CDCl_3$
CHLOROFORM-d
1.00
2.01
1.00
2.02
Chemical Shift (ppm)

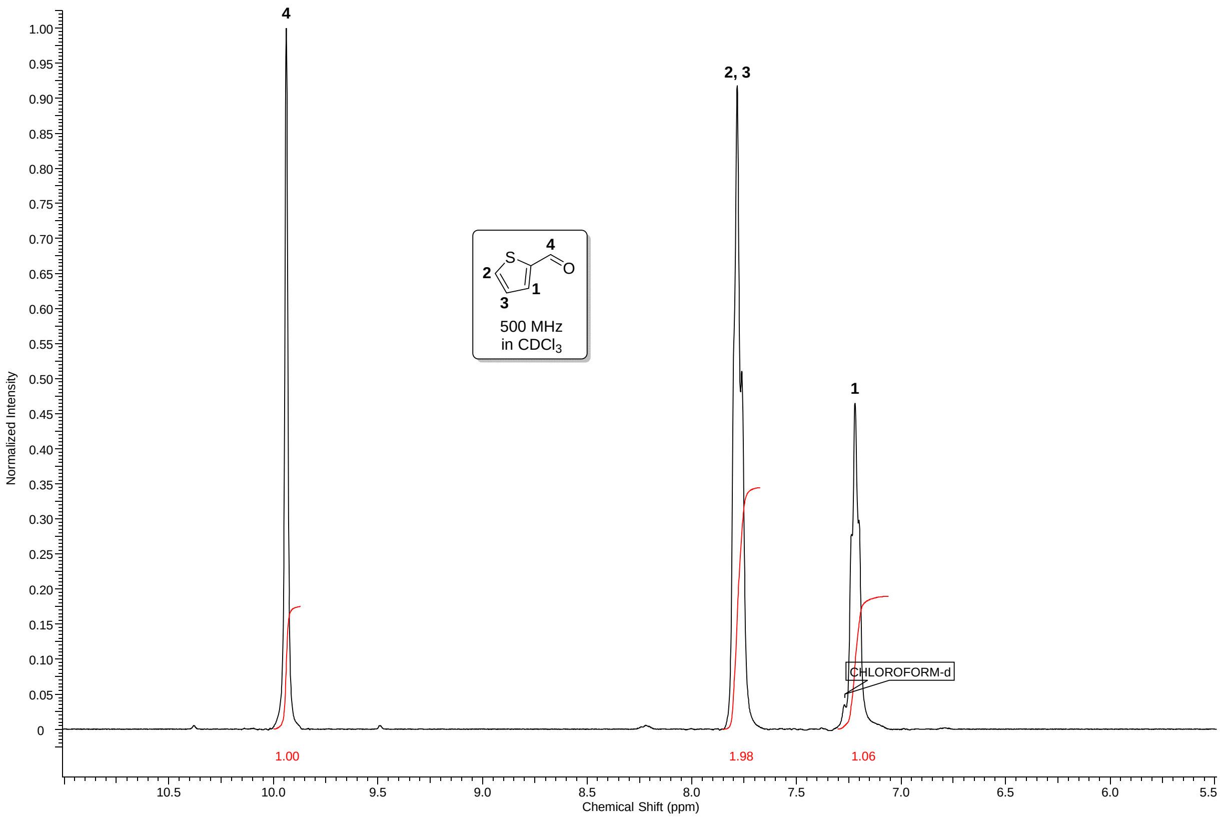

Normalized Intensity
125 MHz
in $CDCl_3$
192.4
136.3
134.4
129.7
128.9
CHLOROFORM-d
77.3
77.0
76.8
Chemical Shift (ppm)

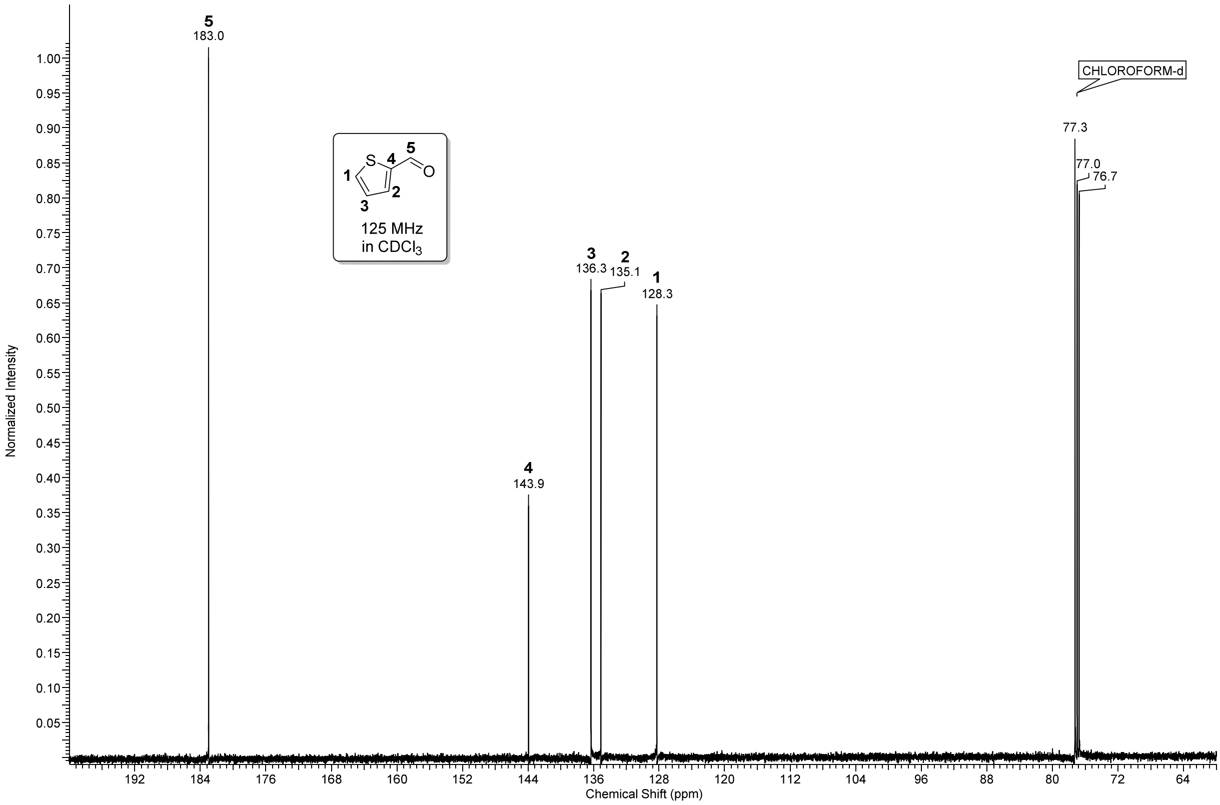


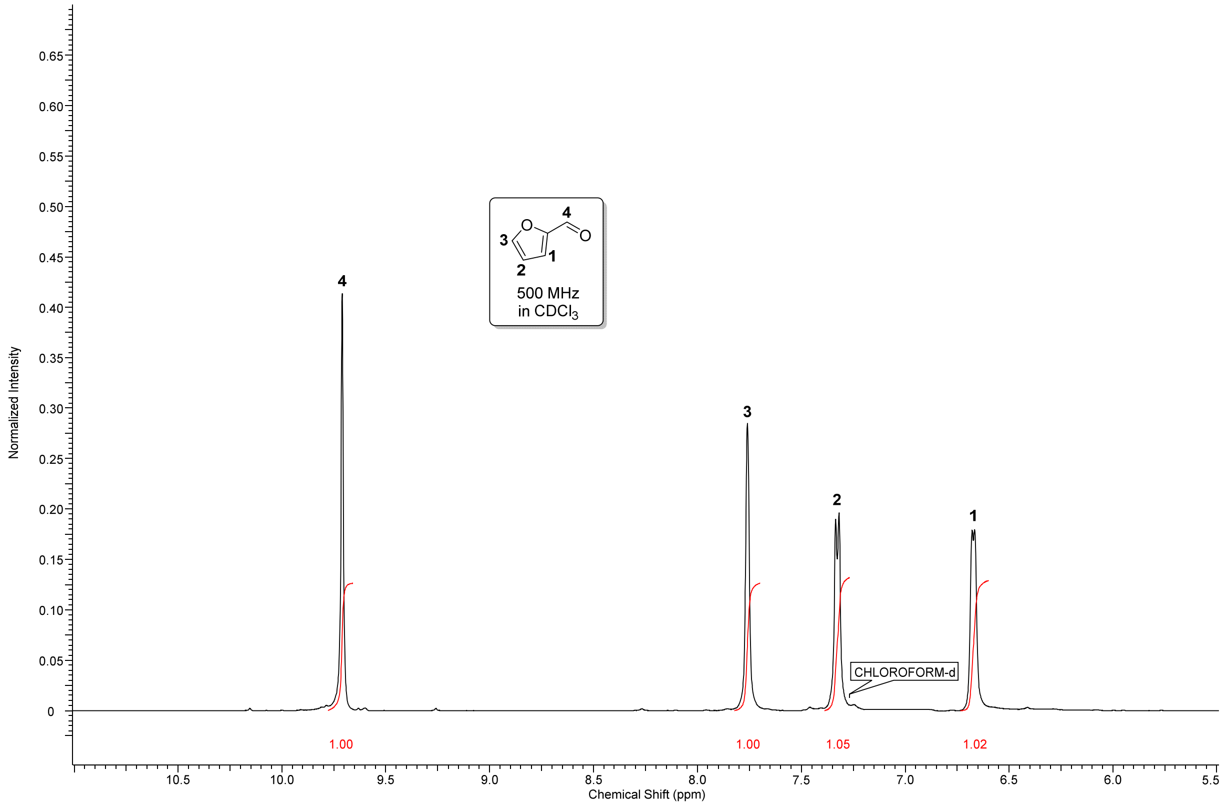
500 MHz
in $CDCl_3$
CHLOROFORM-d
1.00
2.01
2.01
3.02
Normalized Intensity
Chemical Shift (ppm)

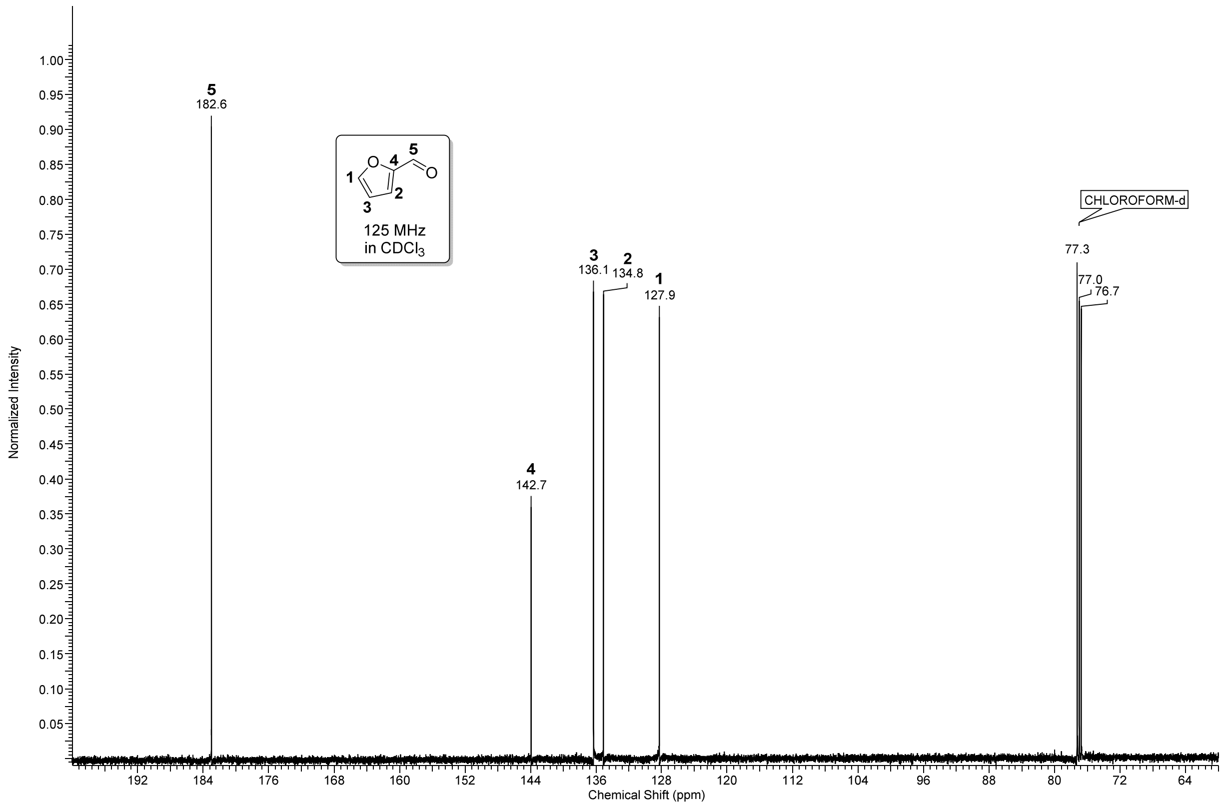


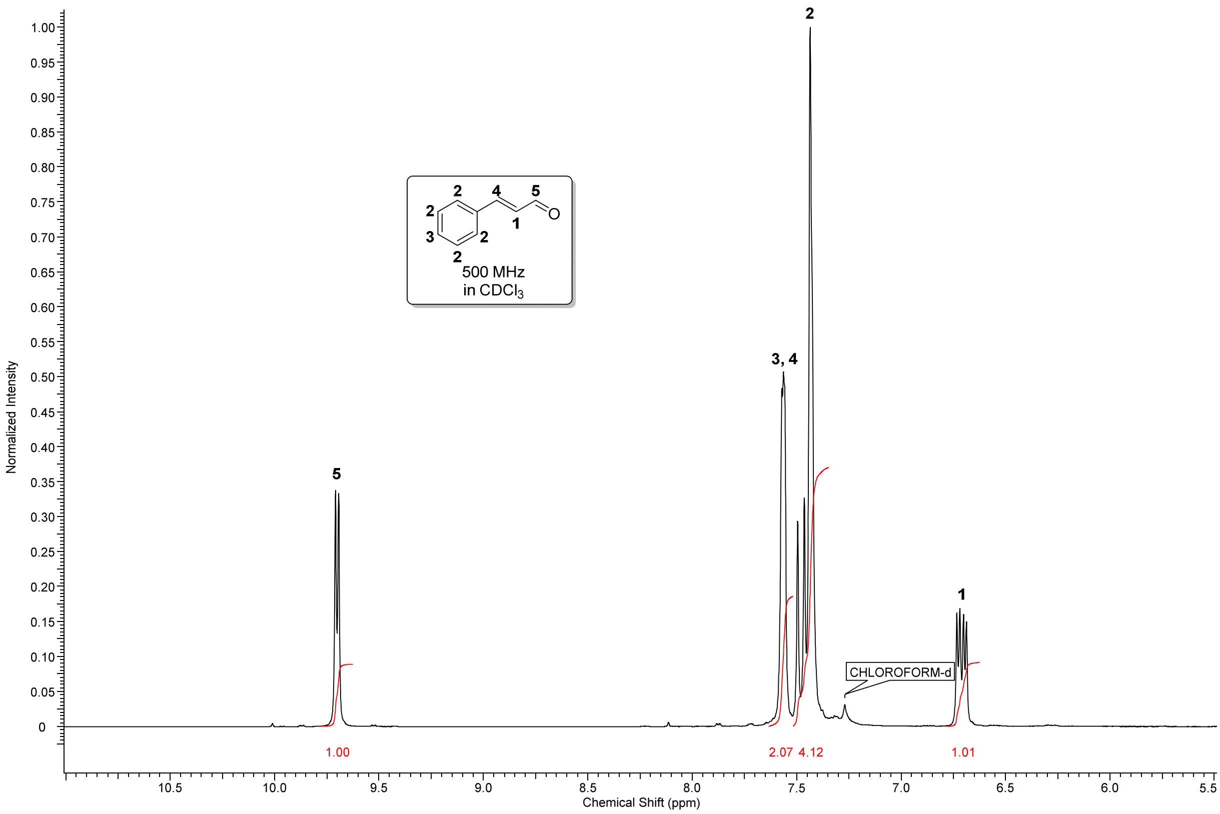
125 MHz
in $CDCl_3$
190.8
164.5
131.9
129.8
114.2
77.3
77.0
76.8
55.5
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)


4
2, 3
1
500 MHz
in CDCl3
CHLOROFORM-d
1.00
1.98
1.06
Normalized Intensity
Chemical Shift (ppm)

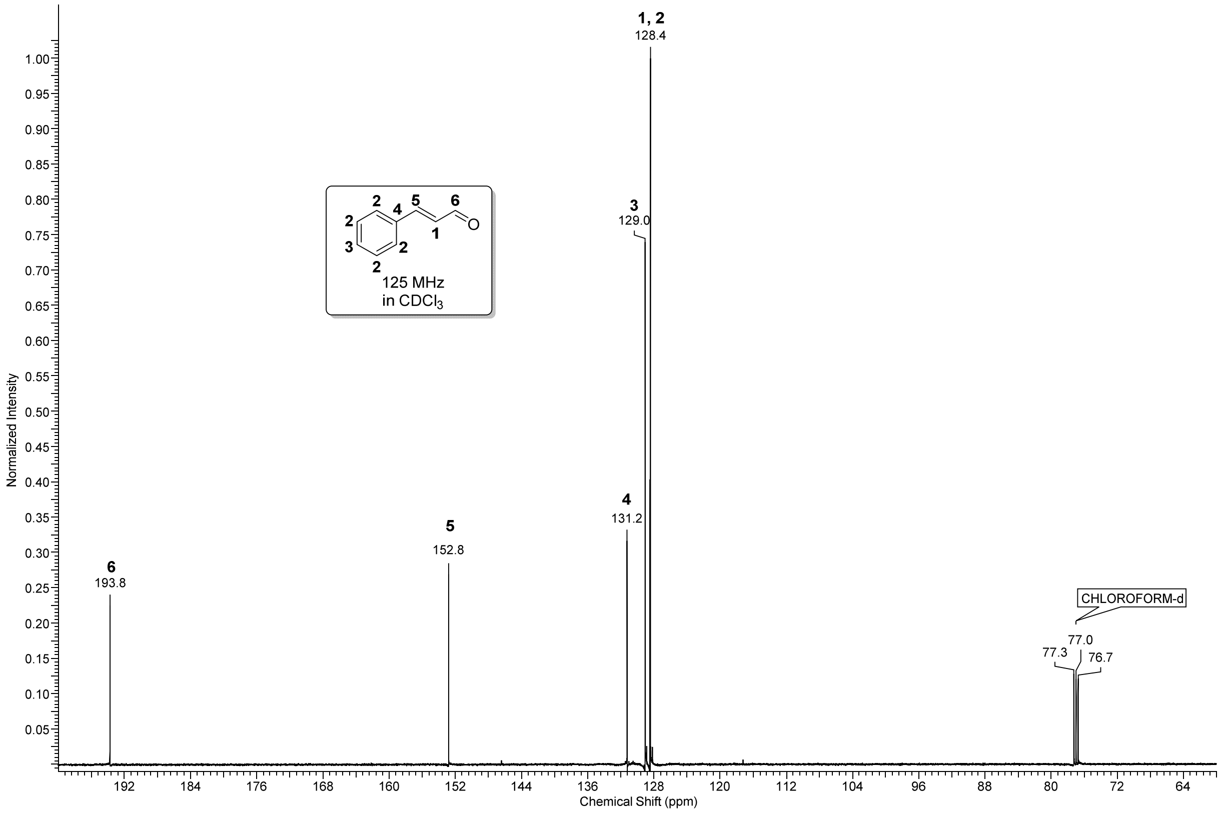

5
183.0
125 MHz
in CDCl3
CHLOROFORM-d
77.3
77.0
76.7
3
136.3
2
135.1
1
128.3
4
143.9
Normalized Intensity
Chemical Shift (ppm)


500 MHz
in $CDCl_3$
CHLOROFORM-d
1.00
1.00
1.05
1.02
Chemical Shift (ppm)
Normalized Intensity

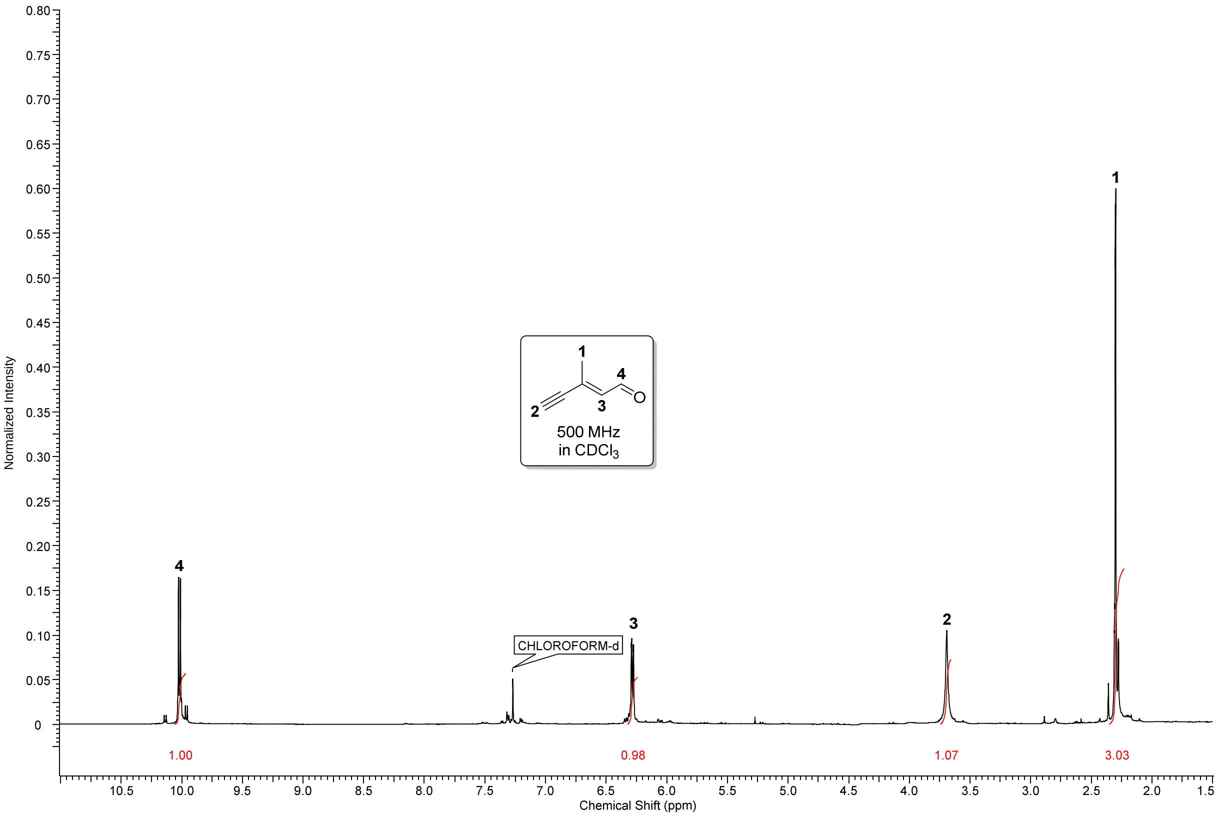

125 MHz
in $CDCl_3$
182.6
142.7
136.1
134.8
127.9
CHLOROFORM-d
77.3
77.0
76.7
Chemical Shift (ppm)
Normalized Intensity



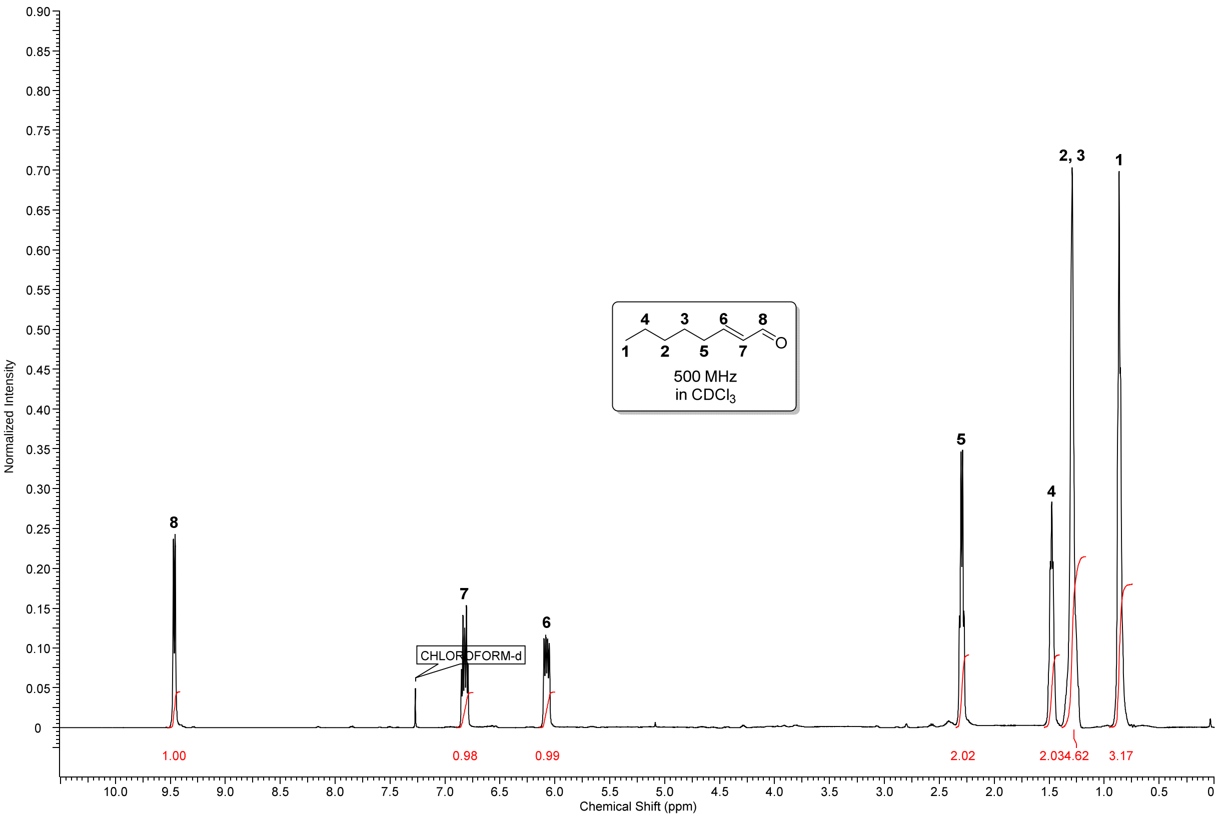
2
3, 4
5
1
500 MHz
in CDCl3
CHLOROFORM-d
1.00
2.07 4.12
1.01
Normalized Intensity
Chemical Shift (ppm)



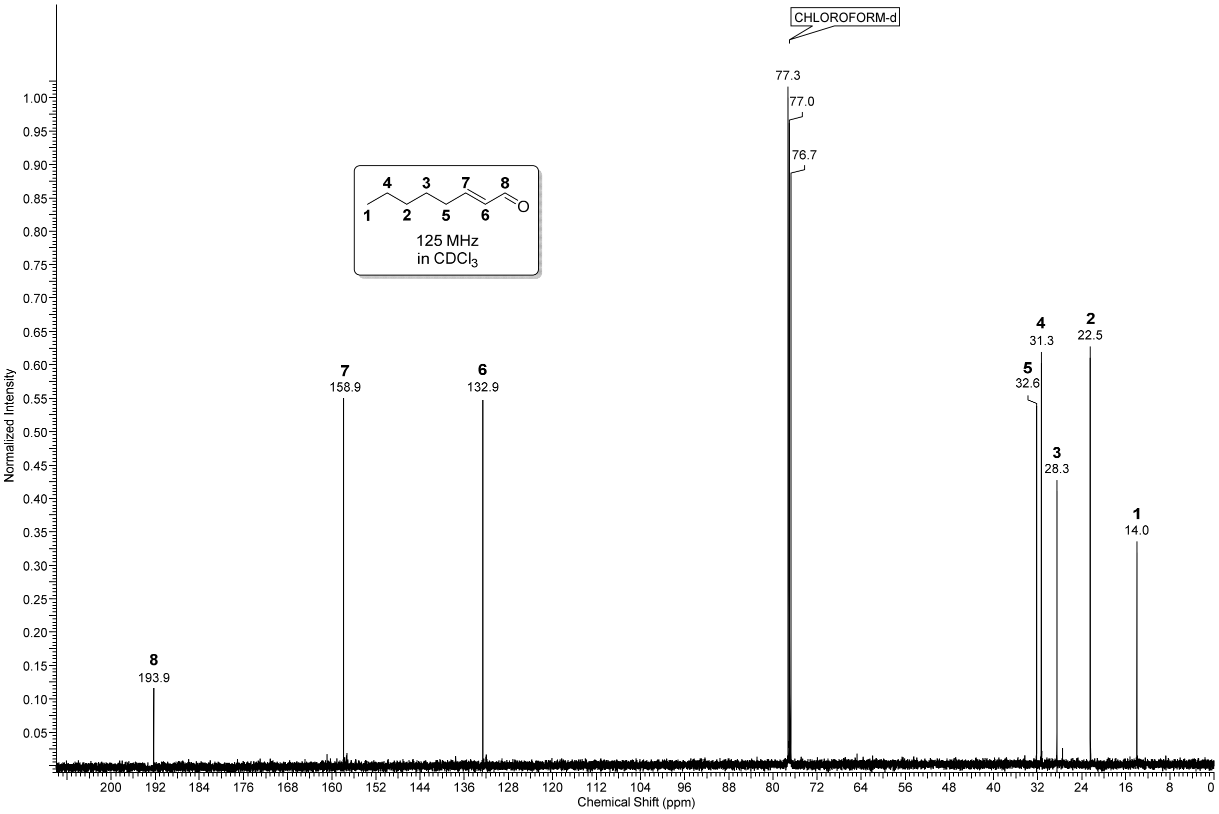
1, 2
128.4
3
129.0
4
131.2
5
152.8
6
193.8
125 MHz
in CDCl3
CHLOROFORM-d
77.0
77.3
76.7
Normalized Intensity
Chemical Shift (ppm)

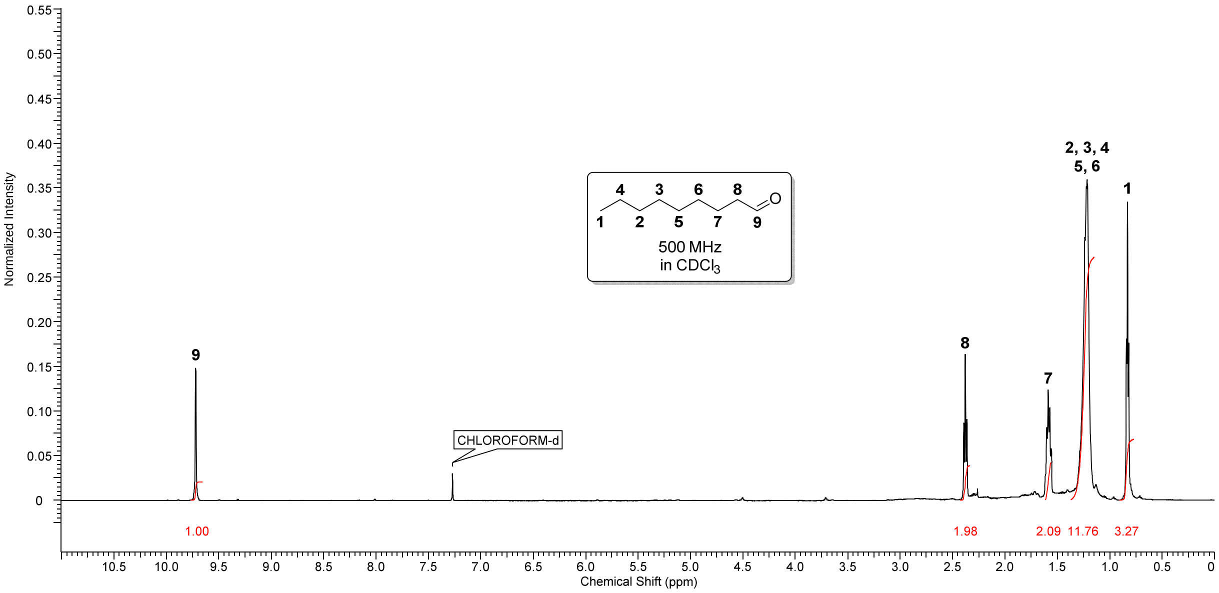


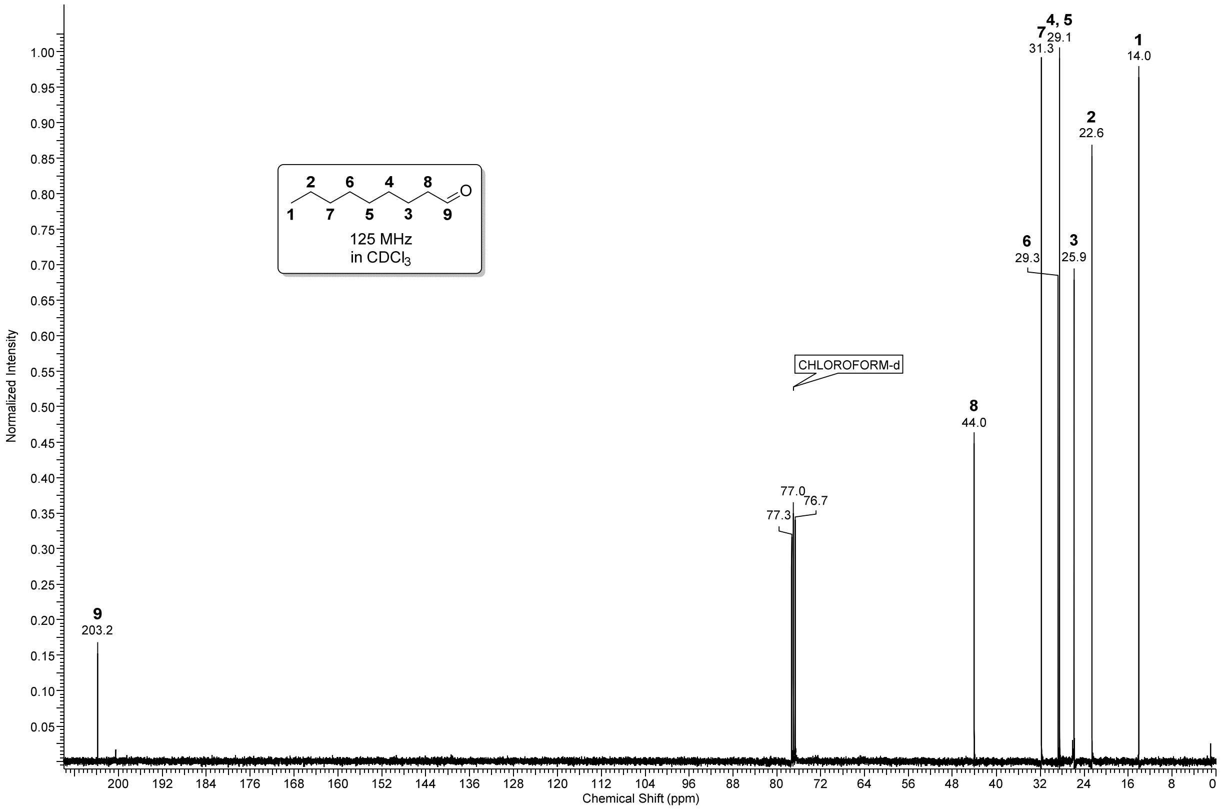
1
4
2
3
500 MHz
in $CDCl_3$
CHLOROFORM-d
1.00
0.98
1.07
3.03
Normalized Intensity
Chemical Shift (ppm)



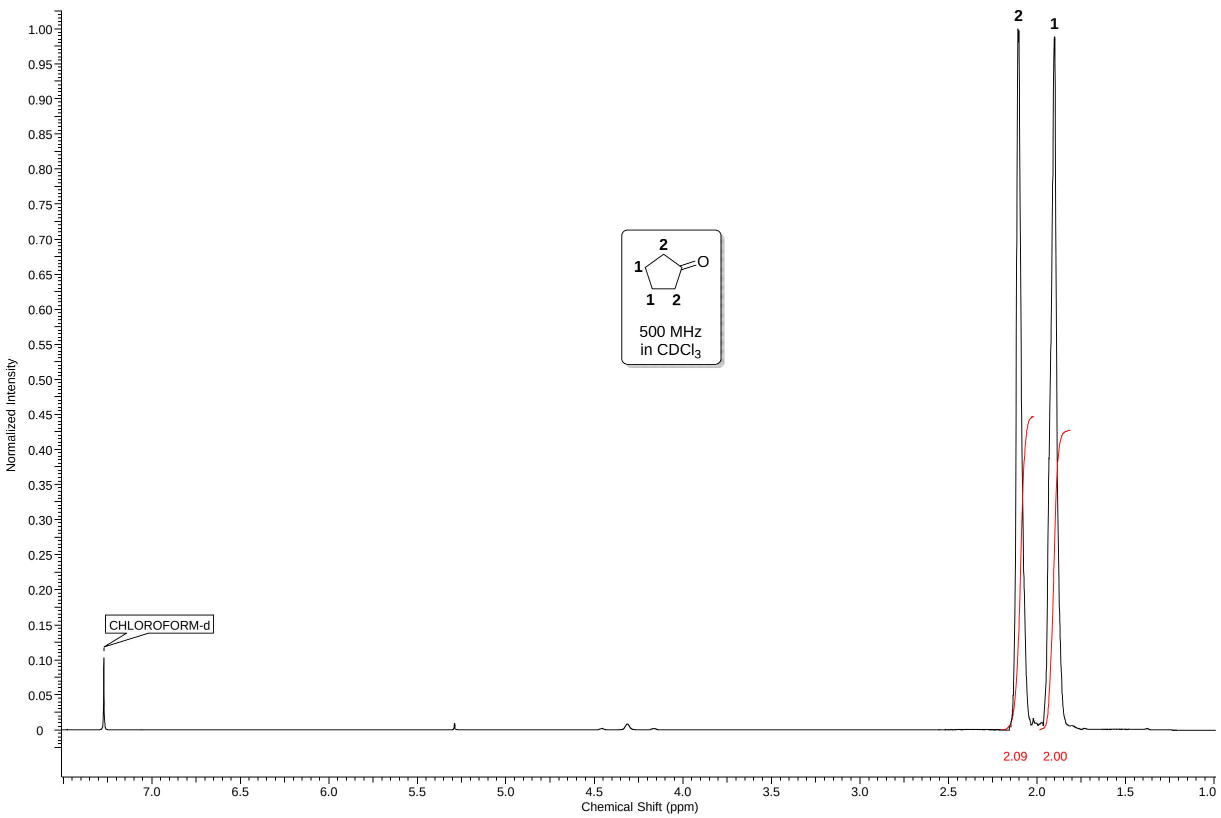
4
3
6
8
1
2
5
7
500 MHz
in $CDCl_3$
2, 3
CHLOROFORM-d
1.00
0.98
0.99
2.02
2.03
4.62
3.17
Normalized Intensity
Chemical Shift (ppm)


CHLOROFORM-d
77.3
77.0
76.7
125 MHz
in CDCl3
8
193.9
7
158.9
6
132.9
5
32.6
4
31.3
3
28.3
2
22.5
1
14.0
Normalized Intensity
Chemical Shift (ppm)

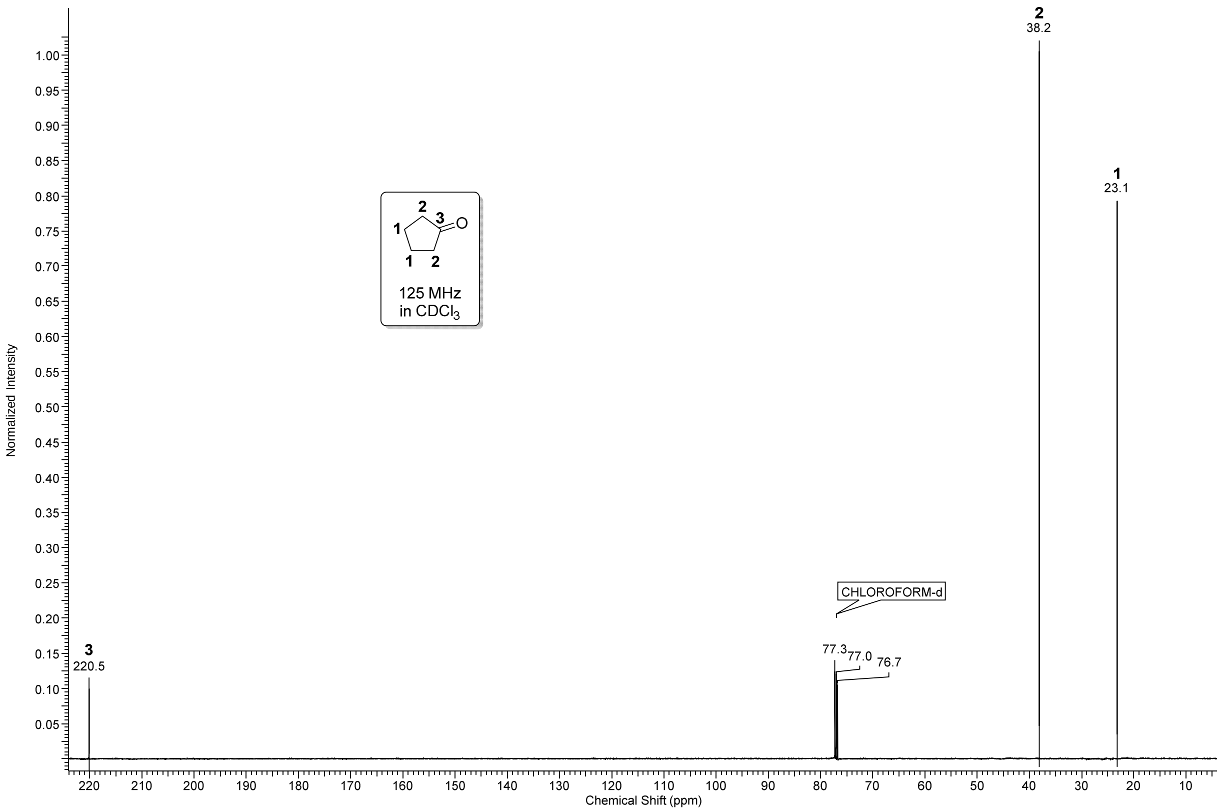

500 MHz
in CDCl3
9
CHLOROFORM-d
8
7
2, 3, 4
5, 6
1
1.00
1.98
2.09
11.76
3.27
Normalized Intensity
Chemical Shift (ppm)



125 MHz
in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)

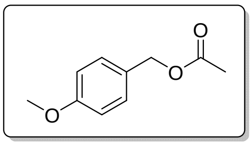


500 MHz
in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
2.09
2.00

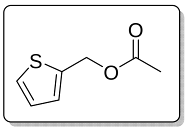


125 MHz
in $CDCl_3$
CHLOROFORM-d
3
220.5
77.3
77.0
76.7
2
38.2
1
23.1
Normalized Intensity
Chemical Shift (ppm)

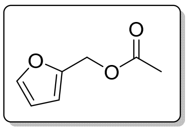
6. ^{1}H- and ^{13}C-NMR data of the synthesized acetyl esters.



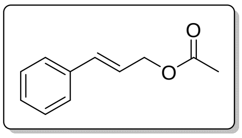
(0.99 g, 5.52 mmol, 95%); **^{1}H-NMR (500 MHz, CDCl$_3$):** δ = 2.09 ppm (s, $\mathrm{CC}H_3$), 3.82 (s, $\mathrm{OC}H_3$), 5.05 (s, $\mathrm{OC}H_2$), 6.90 (d, J = 8 Hz, $\mathrm{2xCH_2CC}H$), 7.31 (d, J = 8 Hz, $\mathrm{2xOCC}H$); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 21.1 ppm ($\mathrm{C}C\mathrm{H_3}$), 55.3 ($\mathrm{O}C\mathrm{H_3}$), 66.1 ($\mathrm{O}C\mathrm{H_2}$), 113.9 ($\mathrm{2xCH_2C}C\mathrm{H}$), 128.0 ($\mathrm{2xOC}C\mathrm{H}$), 130.1 ($\mathrm{CH_2}C\mathrm{CH}$), 159.6 ($\mathrm{O}C\mathrm{CH}$), 171.0 ($C\mathrm{CH_3}$). Known compound.[S10]



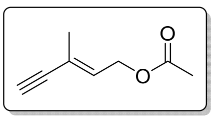
(0.86 g, 5.51 mmol, 95%); **^{1}H-NMR (500 MHz, CDCl$_3$):** δ = 2.10 ppm (s, $\mathrm{C}H_3$), 5.27 (s, $\mathrm{C}H_2$), 7.0 (dd, J = 4.9, J = 3.7 Hz, $\mathrm{CC}H$), 7.10 (d, J = 3.4 Hz, $\mathrm{CCHC}H$), 7.33 (d, J = 4.9, 0.9 Hz, $\mathrm{SC}H$); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 21.0 ppm ($\mathrm{C}C\mathrm{H_3}$), 60.4 ($\mathrm{O}C\mathrm{H_2}$), 126.8 ($\mathrm{C}C\mathrm{H}C\mathrm{HCH}$), 128.2 ($\mathrm{S}C\mathrm{H}$), 137.9 ($\mathrm{S}C\mathrm{CH_2}$), 170.7 ($C\mathrm{CH_3}$). Known compound.[S11]



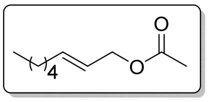
(0.65 g, 4.47 mmol, 77%); **^{1}H-NMR (500 MHz, CDCl$_3$):** δ = 2.08 ppm (s, $\mathrm{C}H_3$), 5.06 (s, $\mathrm{C}H_2$), 6.36 (dd, J = 2.9 Hz, 2 Hz, $\mathrm{CC}H$), 6.41 (d, J = 2.9 Hz, $\mathrm{CCHC}H$), 7.42 (d, J = 1.5 Hz, $\mathrm{OC}H$); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 20.8 ($\mathrm{C}C\mathrm{H_3}$), 58.0 ($\mathrm{O}C\mathrm{H_2}$), 110.5 ($\mathrm{C}C\mathrm{H}C\mathrm{HCH}$), 143.2 ($\mathrm{O}C\mathrm{H}$), 149.4 ($\mathrm{O}C\mathrm{CH_2}$), 170.6 ($C\mathrm{CH_3}$). Known compound.[S10]



(1.01 g, 5.74 mmol, 99%); **^{1}H-NMR (500 MHz, CDCl$_3$):** δ = 2.12 ppm (s, $\mathrm{CCH_3}$), 4.74 (dd, J = 6.4, 0.9 Hz, $\mathrm{OC}H_2$), 6.30 (dt, J = 15.9, 6.4, 6.4 Hz, $\mathrm{OCH_2C}H$), 6.66 (d, J = 15.9 Hz, $\mathrm{CH_2CHC}H$), 7.22 – 7.45 (m, 5xCHAromat); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 20.9 ppm ($\mathrm{C}C\mathrm{H_3}$), 65.0 ($\mathrm{O}C\mathrm{H_2}$), 123.0 ($\mathrm{OCH_2}C\mathrm{H}$), 126.5 ($\mathrm{CCHCH}C\mathrm{H}$), 128.0 ($\mathrm{2xCCH}C\mathrm{H}$), 128.5 ($\mathrm{2xC}C\mathrm{HCH}$), 134.1 ($\mathrm{C}C\mathrm{H}$), 136.1 ($C\mathrm{CH}$), 170.8 ($C\mathrm{CH_3}$). Known compound.[S11]

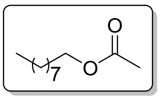


(0.74 g, 5.34 mmol, 92%); **^{1}H-NMR (500 MHz, CDCl$_3$):** δ = 1.88 ppm (s, $\mathrm{CHCC}H_3$), 2.06 (s, $\mathrm{OCC}H_3$), 2.87 (s, $\mathrm{CCC}H$), 4.64 (d, J = 6.8 Hz, $\mathrm{OC}H_2$), 6.00 (t, J =4 Hz, $\mathrm{OCH_2C}H$); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 17.5 ($\mathrm{CHC}C\mathrm{H_3}$), 20.8 ($\mathrm{C}C\mathrm{H_3}$), 60.3 ($C\mathrm{H_2}$), 76.0 ($\mathrm{CC}C\mathrm{H}$), 85.3 ($\mathrm{C}C\mathrm{CH}$), 131.8 ($C\mathrm{HC}C\mathrm{CH}$), 170.7 ($C\mathrm{CH_3}$). Known compound.[S12]

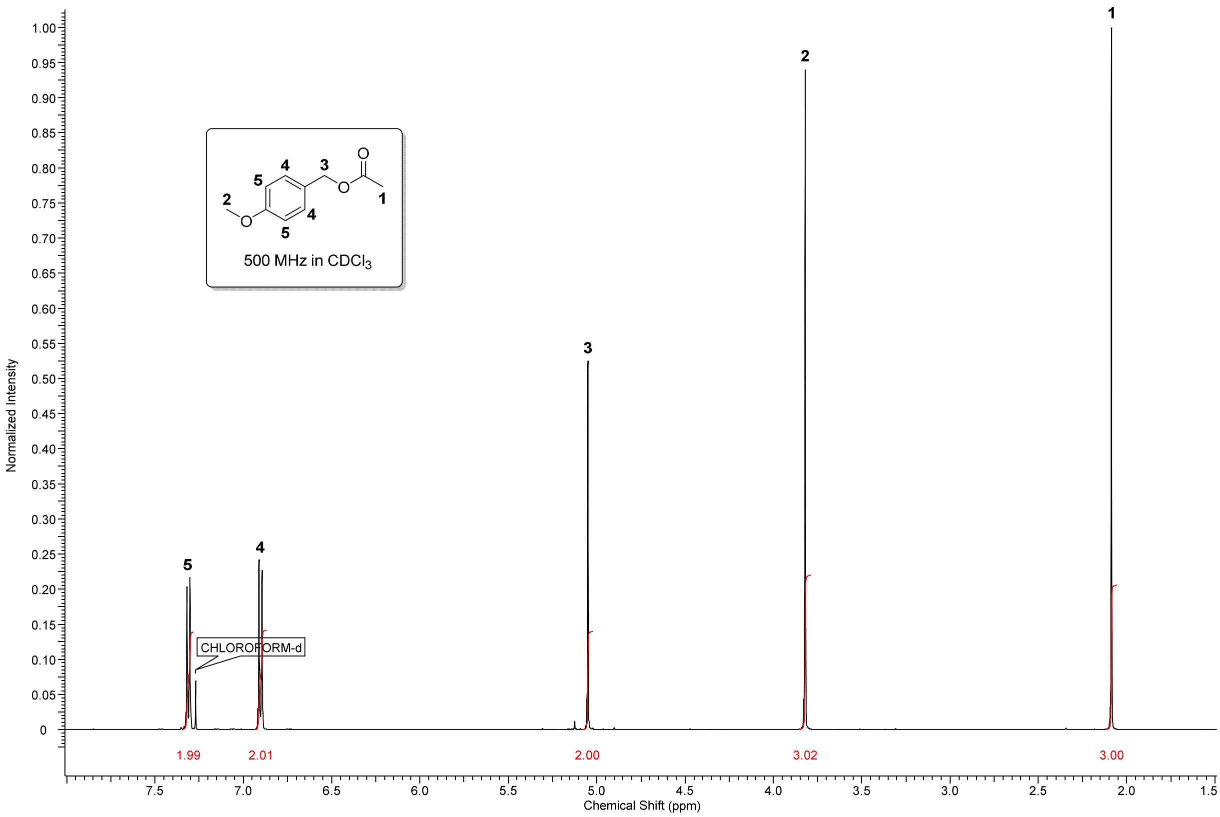


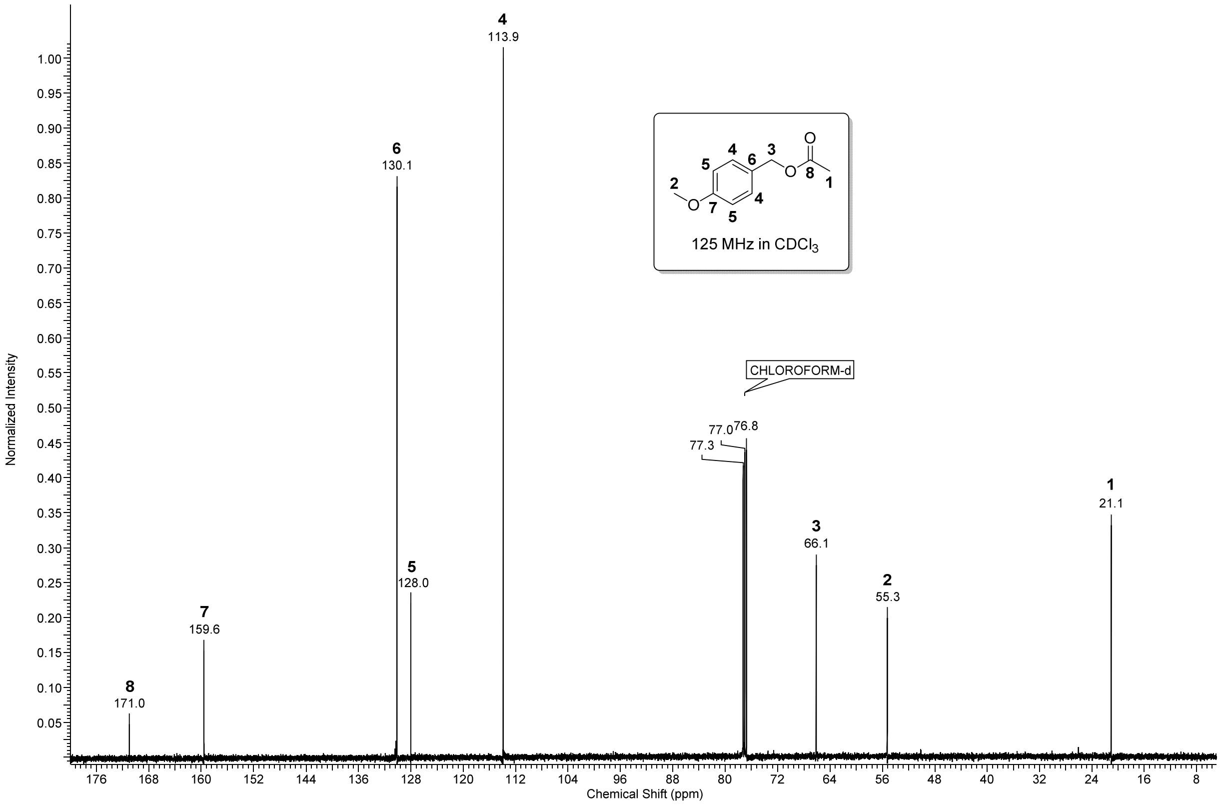
(0.55 g, 3.25 mmol, 56%); **^{1}H-NMR (500 MHz, CDCl$_3$):** δ = 0.89 ppm (t, J = 7.1 Hz, $\mathrm{CH_2C}H_3$), 1.23 – 1.36 (m, $\mathrm{CH_3CH_2C}H_2\mathrm{C}H_2$), 1.39 (dt, J = 6 Hz, $\mathrm{CH_3C}H_2$), 2.03 (d, J =4 Hz, $\mathrm{CHC}H_2\mathrm{CH_2}$), 2.06 (s, $\mathrm{C}H_3$), 4.51 (d, J = 6.8 Hz, $\mathrm{OCH_2}$), 5.56 (dt, J = 12.1, 3.7 Hz, $\mathrm{CH_2CH_2C}H$), 5.77 (dt, J = 12.1, 3.7 Hz, $\mathrm{OCH_2C}H$); **^{13}C-NMR (125 MHz, CDCl$_3$):** δ = 14.0 ($\mathrm{CH_2}C\mathrm{H_3}$), 21.0 ($\mathrm{C}C\mathrm{H_3}$), 22.5 ($C\mathrm{H_2CH_3}$), 28.5

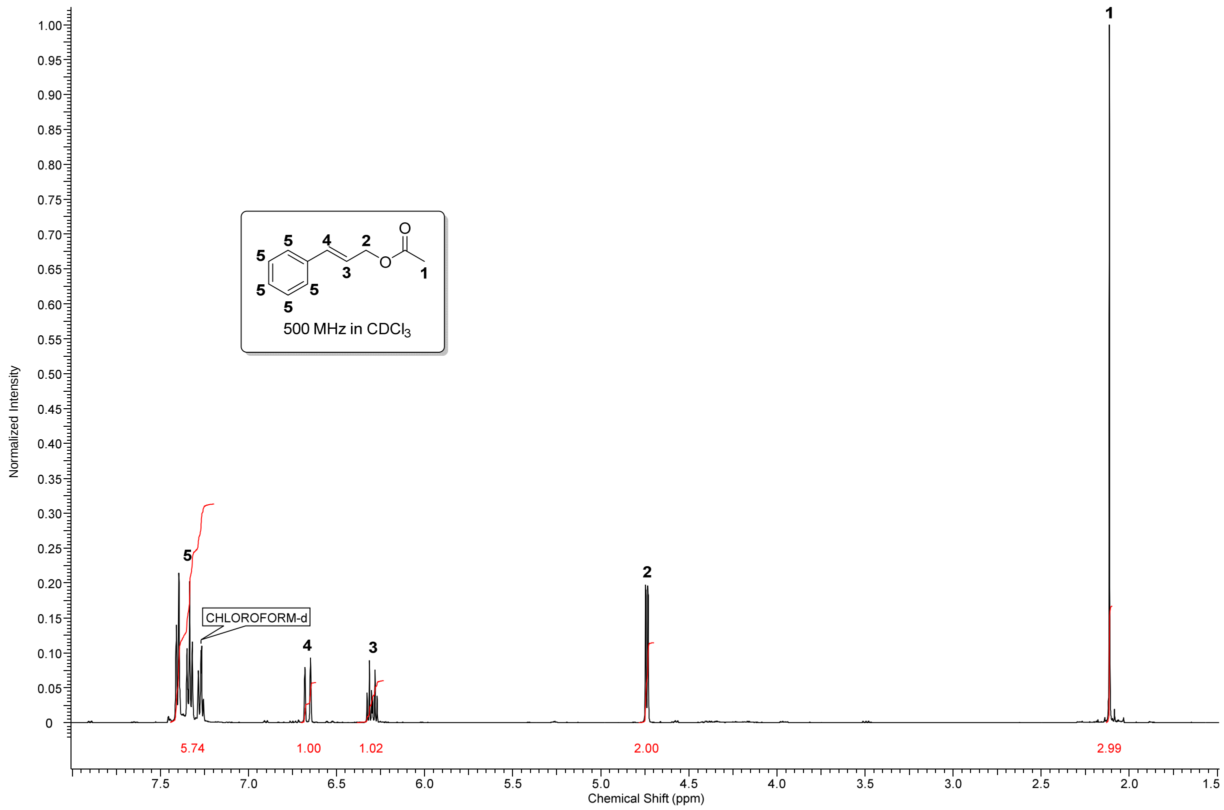
($\mathit{C}H_2CH_2CH_2CH_3$), 31.3 ($\mathit{C}H_2CH_2CH_3$), 32.2 ($\mathit{C}H_2CH_2CH_2CH_2CH_3$), 65.3 ($O\mathit{C}H_2$), 123.6 ($OCH_2CH\mathit{C}H$), 136.8 ($OCH_2\mathit{C}H$), 170.9 ($\mathit{C}CH_3$). Known compound.[S13]

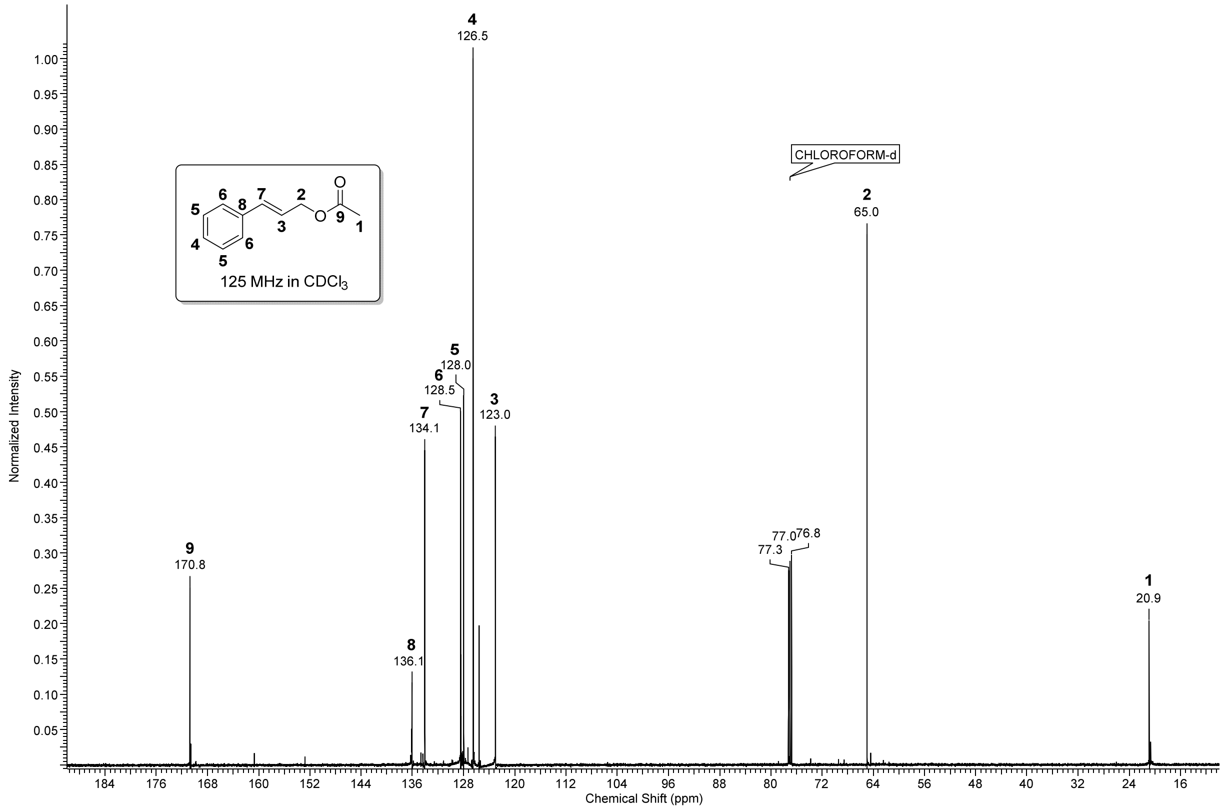


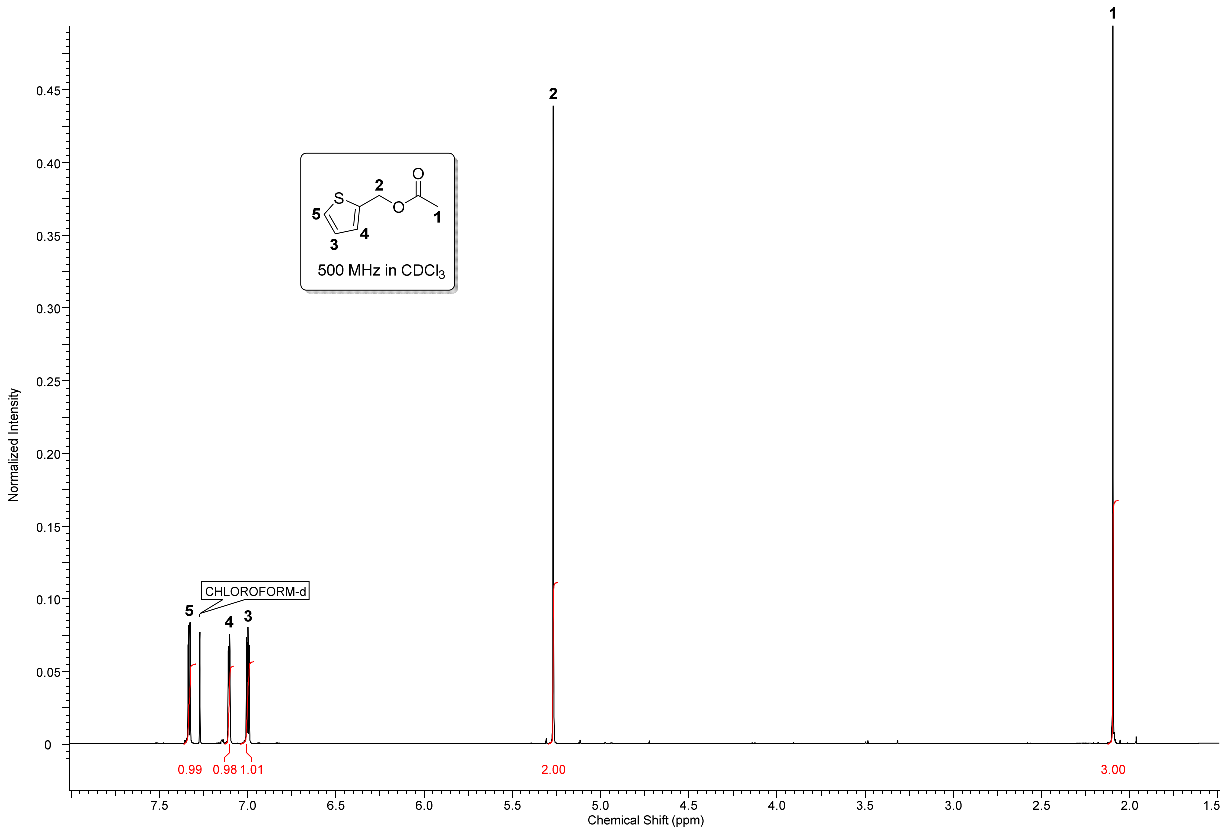
(0.54 g, 2.90 mmol, 50%); **^{1}H-NMR (500 MHz, $CDCl_3$):** δ = 0.89 ppm (t, J = 6.8 Hz, $C\mathit{H_3}$), 1.18 – 1.42(m, $CH_3C\mathit{H_2}C\mathit{H_2}C\mathit{H_2}C\mathit{H_2}C\mathit{H_2}C\mathit{H_2}$), 1.62 (qin, J = 6.8 Hz, $OCH_2C\mathit{H_2}$), 2.05 (s, $CC\mathit{H_3}$), 4.06 (t, J = 6.8 Hz, $OC\mathit{H_2}$); **^{13}C-NMR (125 MHz, $CDCl_3$):** δ = 14.0 ($CH_2\mathit{C}H_3$), 20.9 ($C\mathit{C}H_3$), 22.6 ($\mathit{C}H_2CH_3$), 25.9 ($OCH_2CH_2\mathit{C}H_2$), 28.6 ($OCH_2\mathit{C}H_2$), 29.2 ($OCH_2CH_2CH_2\mathit{C}H_2\mathit{C}H_2$), 29.4 ($\mathit{C}H_2CH_2CH_2CH_3$), 31.8 ($\mathit{C}H_2CH_2CH_3$), 64.6 ($O\mathit{C}H_2$), 171.2 ($\mathit{C}CH_3$). Known compound.[S14]

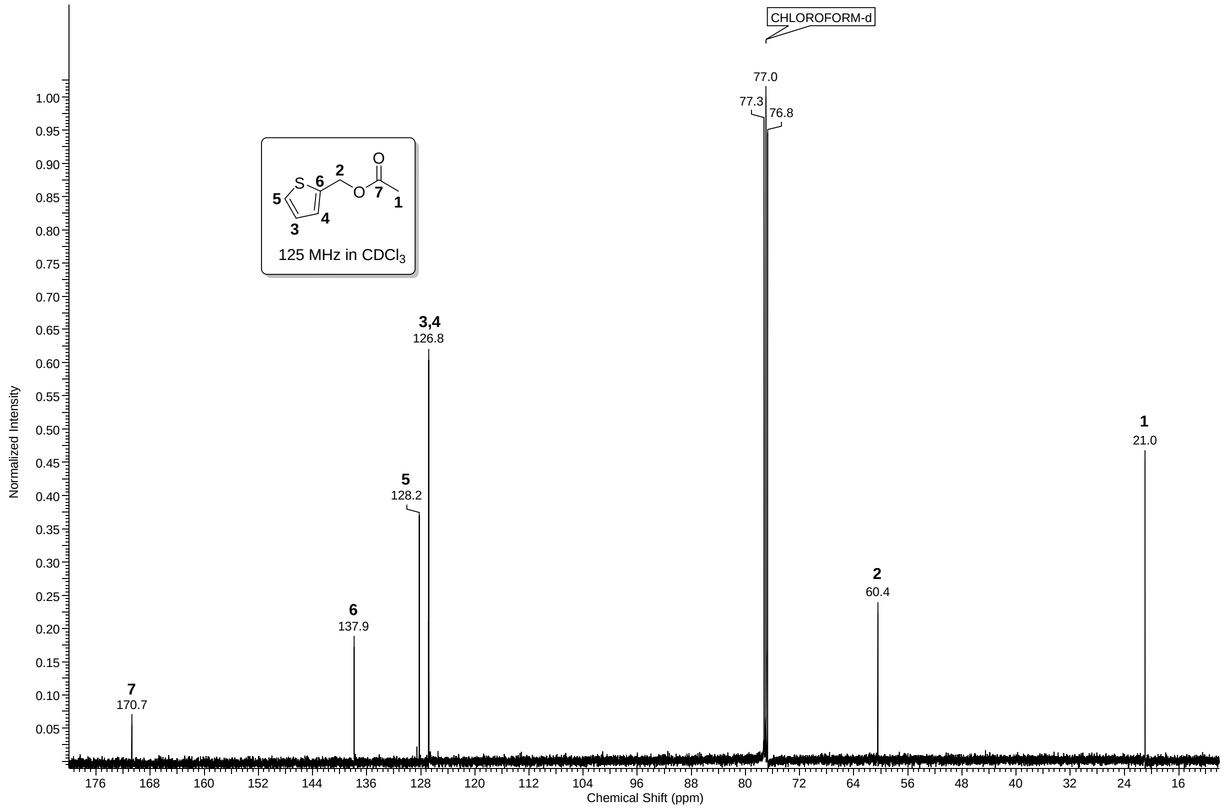

500 MHz in CDCl3
CHLOROFORM-d
1.99
2.01
2.00
3.02
3.00
Normalized Intensity
Chemical Shift (ppm)

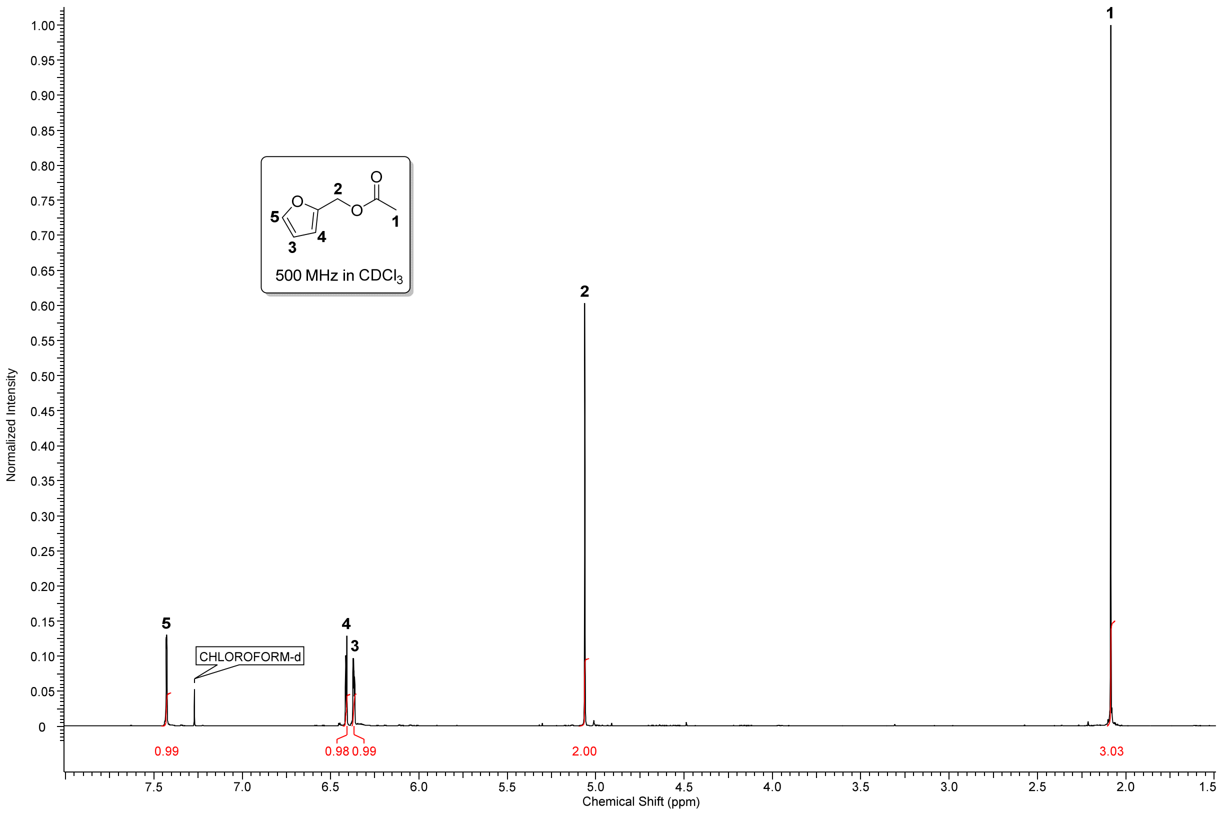

125 MHz in CDCl3
CHLOROFORM-d
171.0
159.6
130.1
128.0
113.9
77.3
77.0
76.8
66.1
55.3
21.1
Normalized Intensity
Chemical Shift (ppm)


500 MHz in CDCl3
CHLOROFORM-d
5.74
1.00
1.02
2.00
2.99
Normalized Intensity
Chemical Shift (ppm)

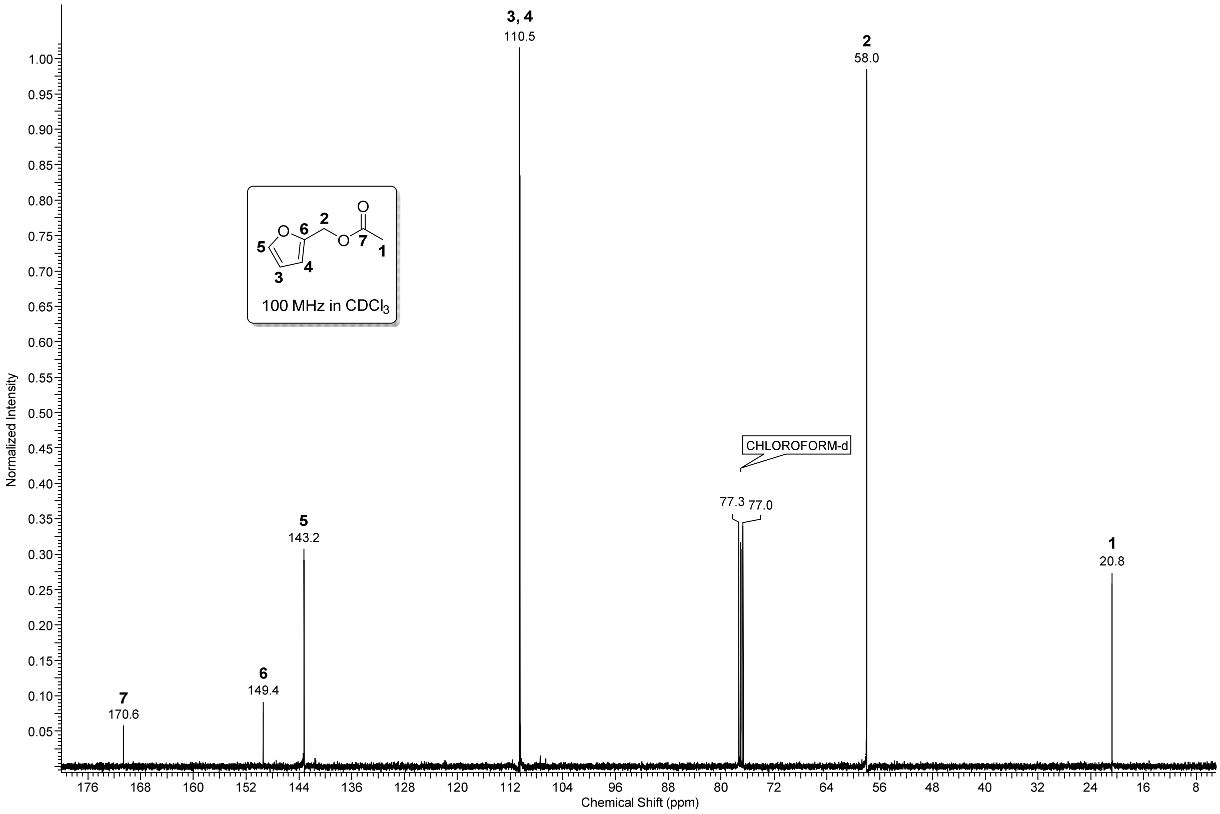

125 MHz in CDCl3
CHLOROFORM-d
126.5
65.0
128.0
128.5
134.1
123.0
170.8
136.1
77.3
77.0
76.8
20.9
Normalized Intensity
Chemical Shift (ppm)


500 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
0.99
0.98
1.01
2.00
3.00

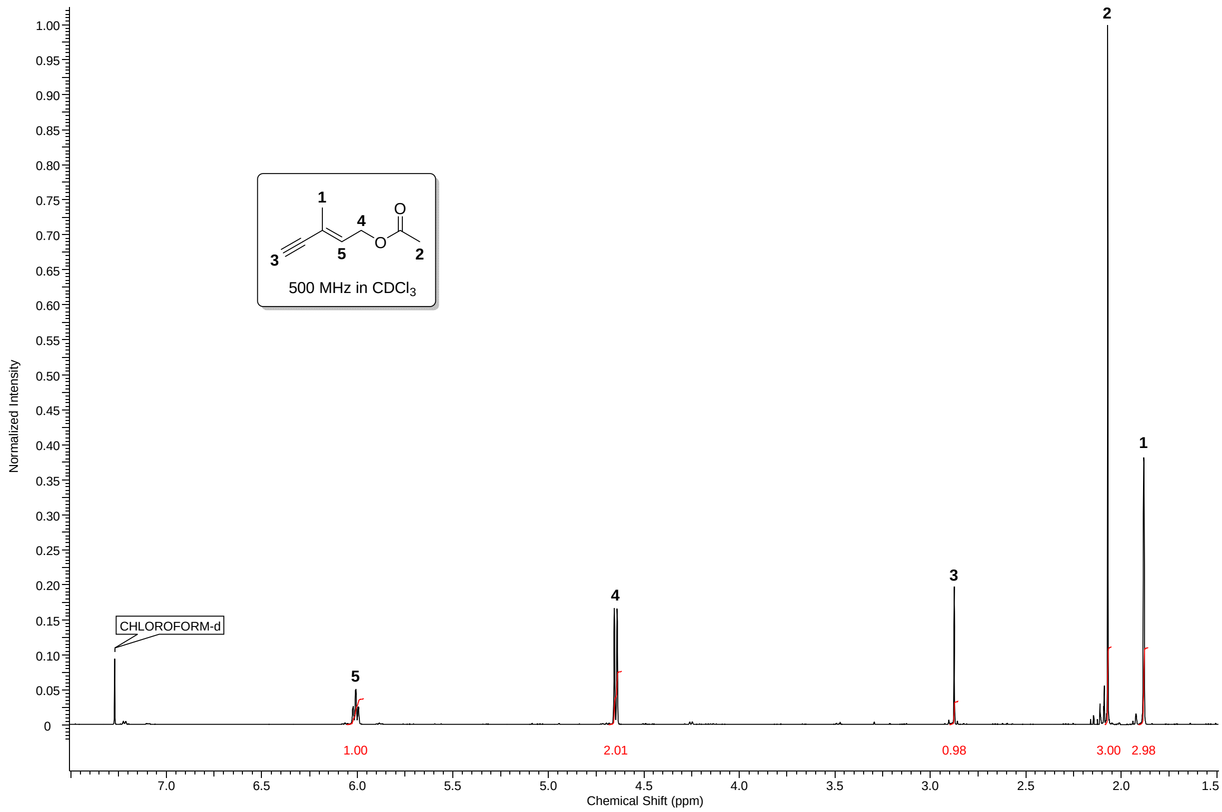

125 MHz in CDCl3
CHLOROFORM-d
77.0
77.3
76.8
170.7
137.9
128.2
126.8
60.4
21.0
Normalized Intensity
Chemical Shift (ppm)



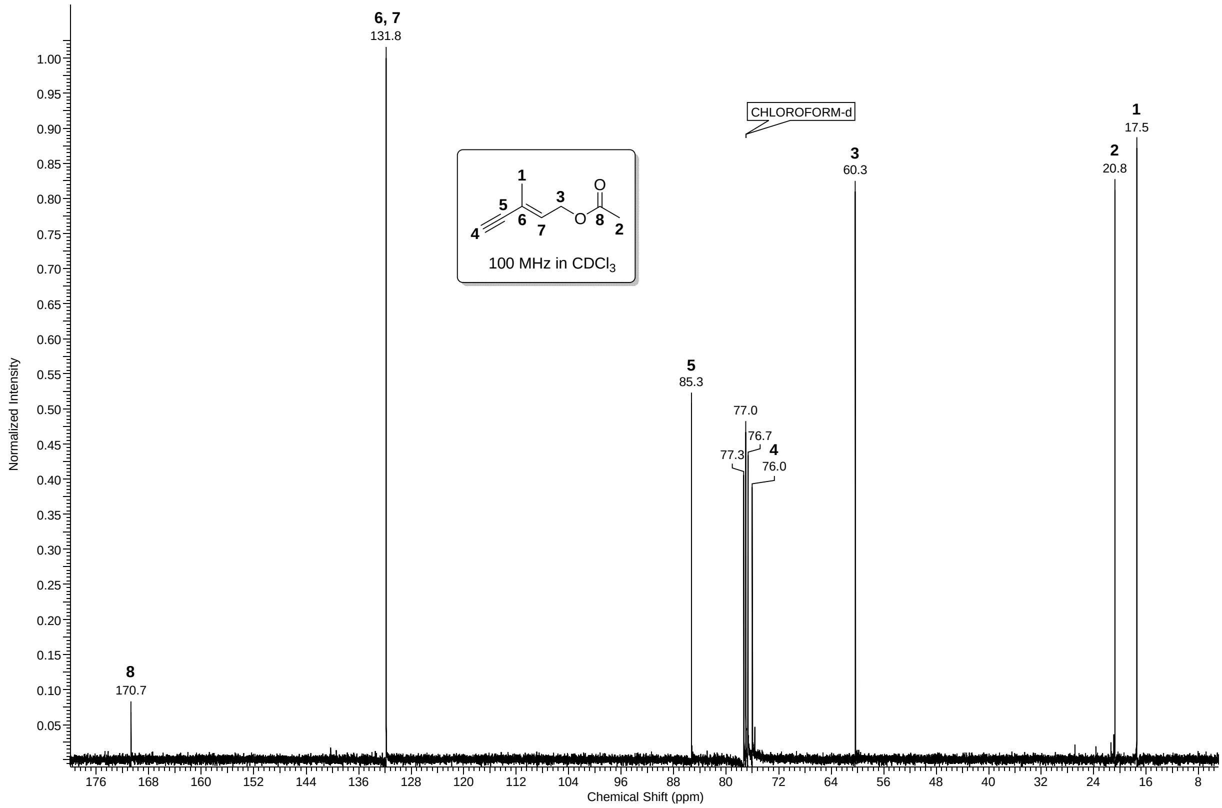
500 MHz in CDCl3
CHLOROFORM-d
0.99
0.98
0.99
2.00
3.03
Normalized Intensity
Chemical Shift (ppm)



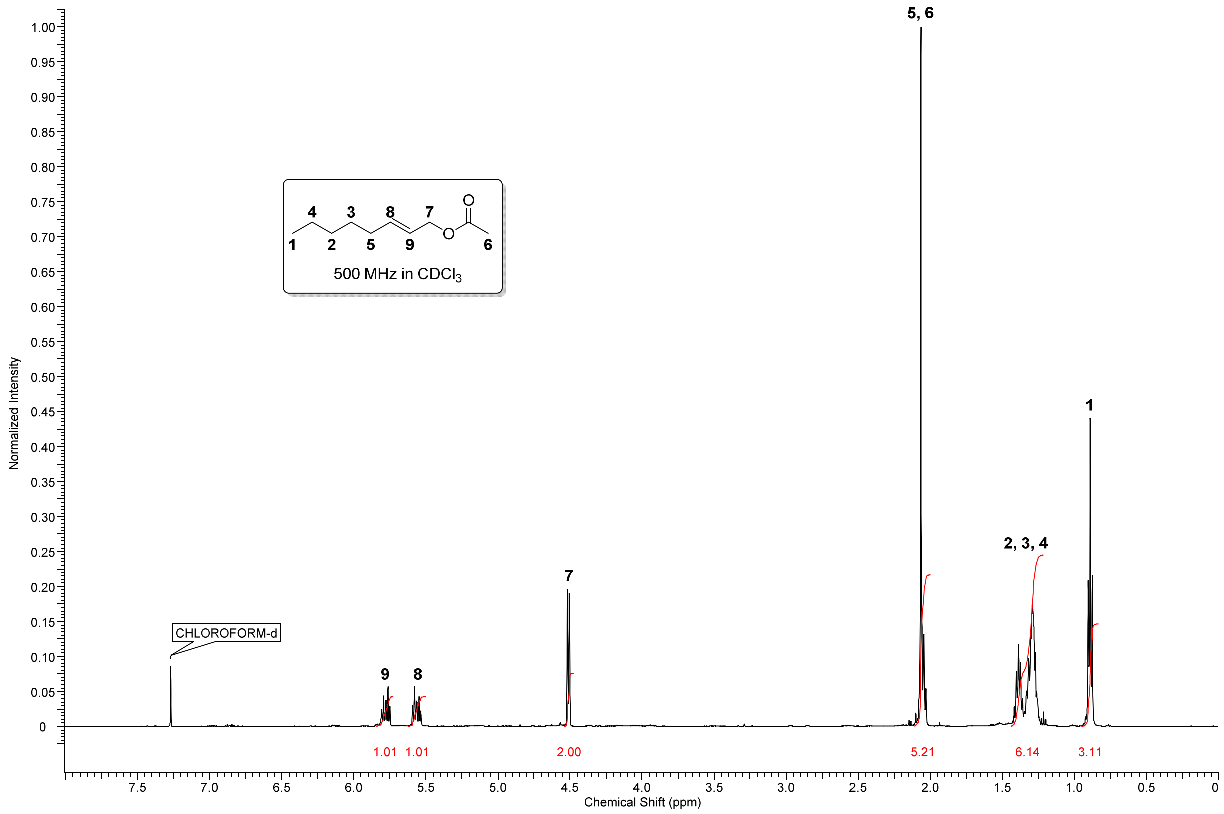
100 MHz in CDCl3
3, 4
110.5
2
58.0
CHLOROFORM-d
77.3
77.0
5
143.2
1
20.8
6
149.4
7
170.6
Normalized Intensity
Chemical Shift (ppm)

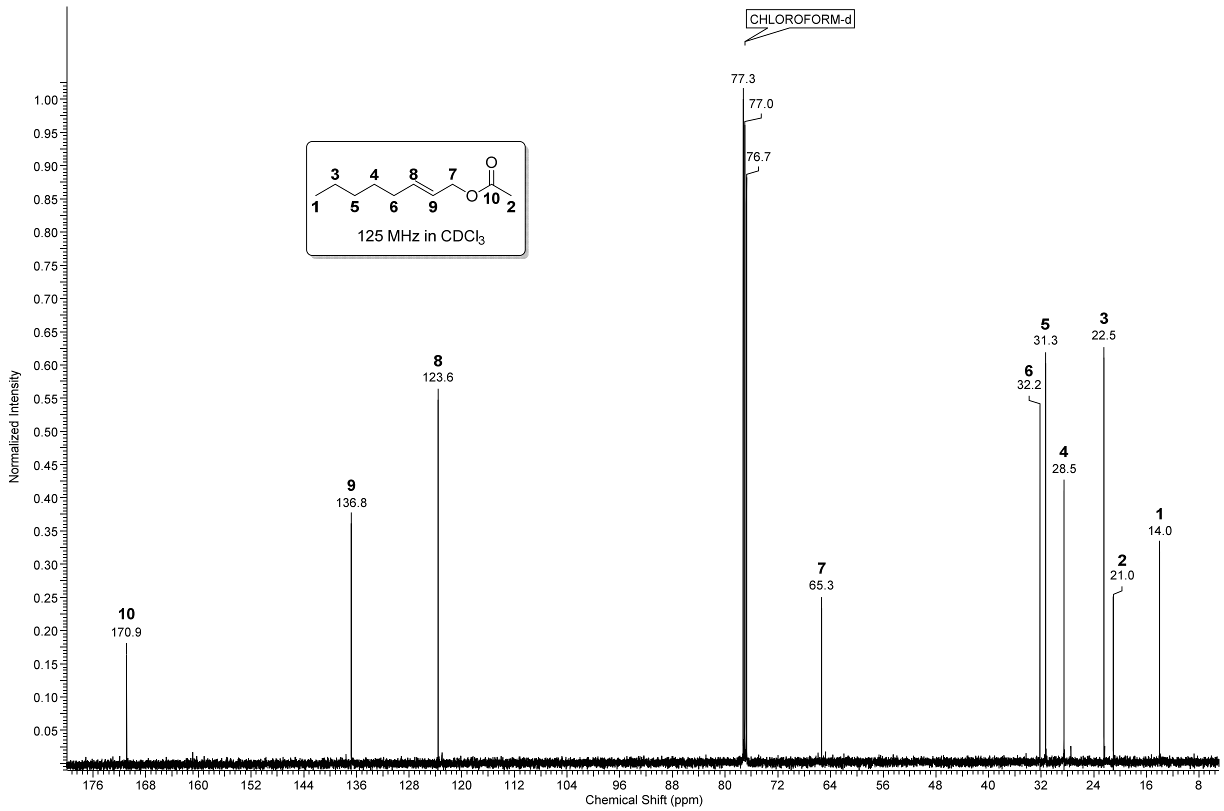


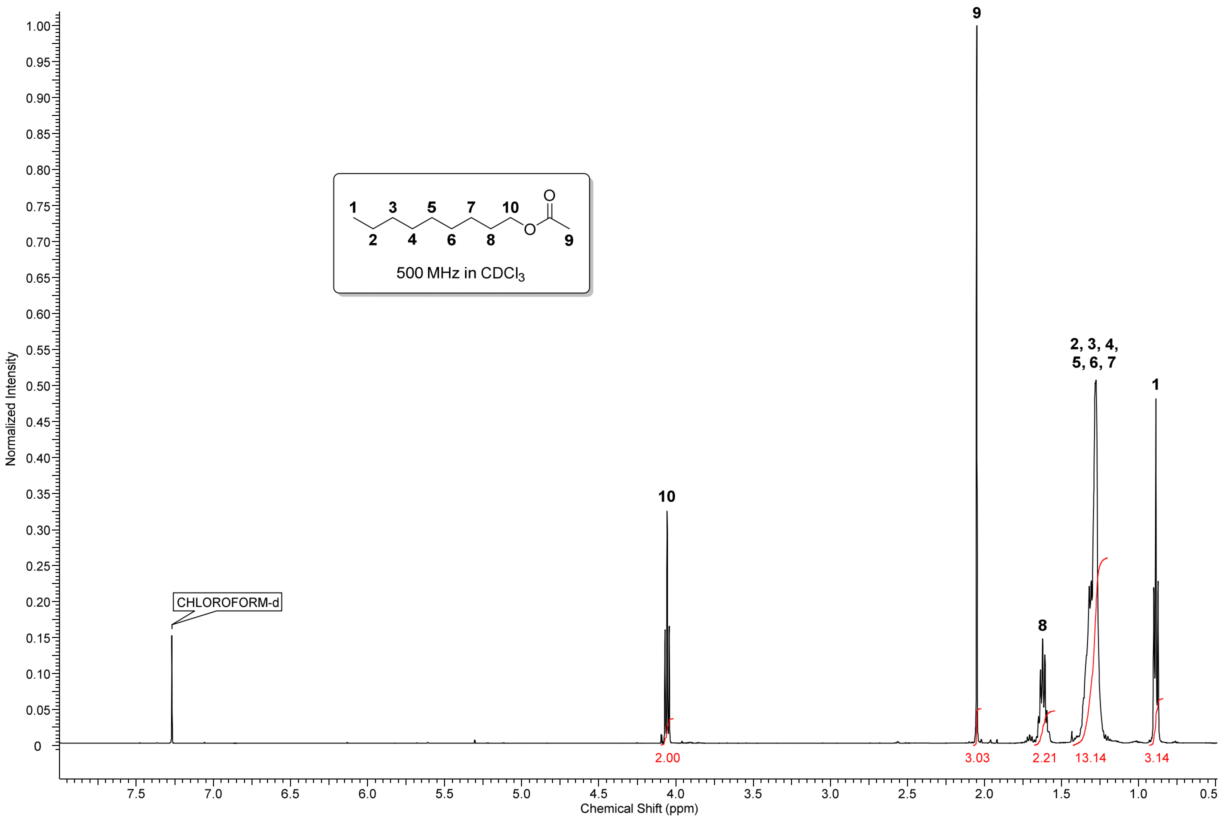
500 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
1.00
2.01
0.98
3.00
2.98



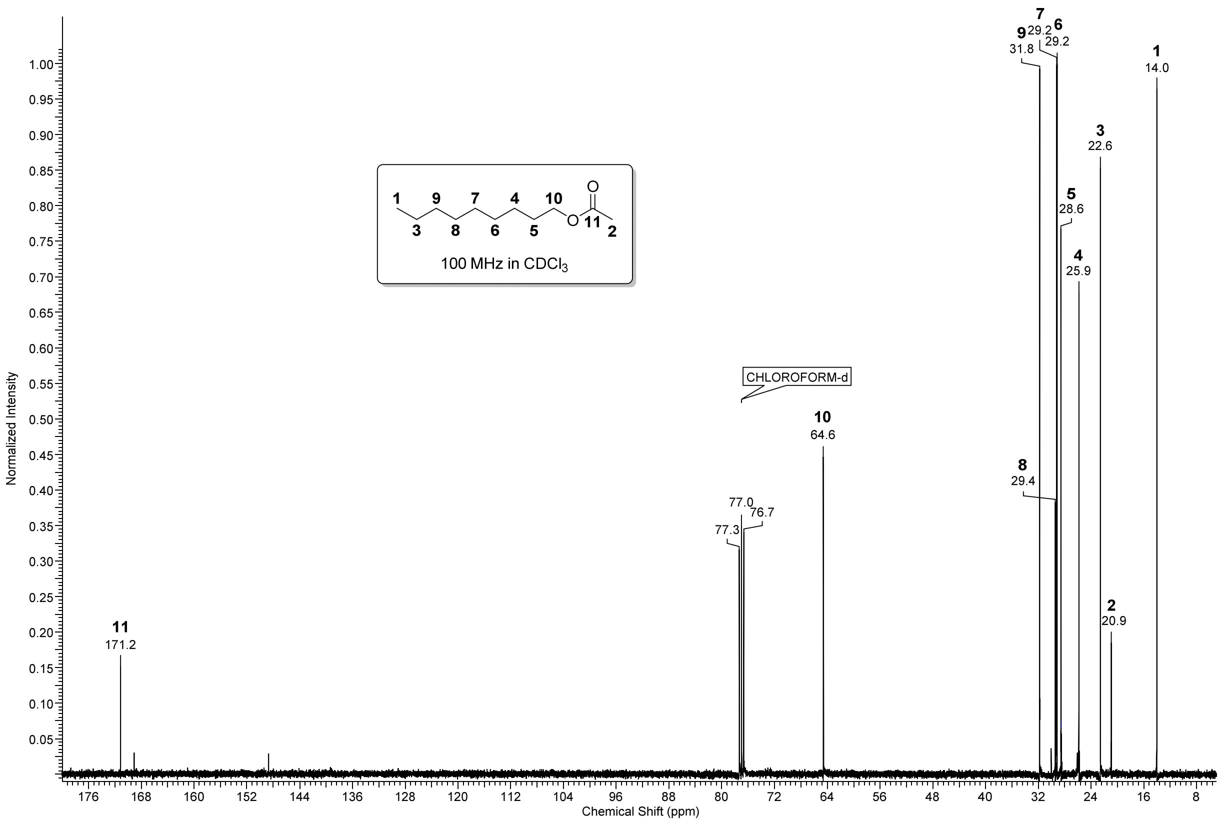
100 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
170.7
131.8
85.3
77.3
77.0
76.7
76.0
60.3
20.8
17.5


500 MHz in CDCl3
CHLOROFORM-d
5, 6
2, 3, 4
Normalized Intensity
Chemical Shift (ppm)
1.01
1.01
2.00
5.21
6.14
3.11


125 MHz in CDCl3
CHLOROFORM-d
77.3
77.0
76.7
170.9
136.8
123.6
65.3
32.2
31.3
28.5
22.5
21.0
14.0
Normalized Intensity
Chemical Shift (ppm)



500 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
2, 3, 4,
5, 6, 7
2.00
3.03
2.21
13.14
3.14



100 MHz in CDCl3
CHLOROFORM-d
Normalized Intensity
Chemical Shift (ppm)
171.2
77.3
77.0
76.7
64.6
31.8
29.4
29.2
29.2
28.6
25.9
22.6
20.9
14.0

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