# Supporting Information

# for

# A Lewis acid-promoted Pinner reaction

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## Experimental section and NMR spectra of all synthesized compounds.

Content:

## A. Experimental Section

**General.** Flash column chromatography was carried out using Merck $SiO_2$ 60 (230–400 mesh),[1] and thin layer chromatography (TLC) was carried out using commercially available Merck F254 precoated sheets. $^1H$ and $^{13}C$ NMR spectra were recorded on a Bruker AVANCE 300 or on an AM-400 spectrometer. Chemical shifts are given in ppm and were referenced using residual signals of the solvent as internal standard ($^1H$: $CHCl_3$, 7.26, acetone, 2.05; $^{13}C$: $CDCl_3$, 77.16). IR spectra were recorded on a Bruker IFS-88 spectrometer. Mass spectra were recorded on a Finnigan MAT-90 mass spectrometer.

**General procedure (GP 1) for reactions with acetonitrile or acrylonitrile.**[2] TMSOTf (336 mg, 1.51 mmol) is added to a solution of the alcohol (0.755 mmol) in the respective nitrile (3 mL) and the mixture is stirred at rt for 65 h. $H_2O$ (25 mL) and brine (25 mL) are added, and the mixture is extracted with EtOAc (3 × 30 mL). The combined organic layers are dried ($Na_2SO_4$) and concentrated. The crude product is purified by flash column chromatography (silica gel).

**General procedure (GP 2) for reactions with acrylonitrile in the presence of nitrobenzene.** TMSOTf (336 mg, 1.51 mmol) is added to a solution of the alcohol (0.755 mmol) and nitrobenzene (93 mg, 0.755 mmol) in the respective nitrile (3 mL) and the mixture is stirred at rt for 65 h. $H_2O$ (25 mL) and brine (25 mL) are added, and the mixture is extracted with EtOAc (3 × 30 mL). The combined organic layers are dried ($Na_2SO_4$) and concentrated. The crude product is purified by flash column chromatography (silica gel).

**General procedure (GP 3) for reactions with benzyl cyanide or benzonitrile.**[2] TMSOTf (336 mg, 1.51 mmol) is added to a solution of the alcohol (0.755 mmol) in the respective nitrile (3 mL) and the mixture is stirred at rt for 65 h. $H_2O$ (25 mL) and brine (25 mL) are added, and the mixture is extracted with EtOAc (3 × 30 mL). The combined organic layers are dried ($Na_2SO_4$) and concentrated, and excess nitrile is distilled off at reduced pressure (90 °C, 0.1 mbar). The crude product is purified by flash column chromatography (silica gel).

**(9*H*-Fluoren-9-yl)methyl acetate (2).** 9*H*-Fluoren-9-ylmethanol (**1**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **2** (152 mg, 83%) as a yellow solid. $R_f$ = 0.53 (hexanes/EtOAc, 3:1). $^1H$ NMR (300 MHz, $CDCl_3$) δ 2.16 (s, 3H, Me), 4.22 (t, $^3J$ = 7.2 Hz, 1H, 9'-H), 4.37 (d, $^3J$ = 7.2 Hz, 2H, $CH_2$), 7.33 (t, $^3J$ = 7.4 Hz, 2H, 2'-H, 7'-H), 7.42 (t, $^3J$ = 7.4 Hz, 2H, 3'-H,

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6'-H), 7.61 (d, $^3J$ = 7.4 Hz, 2H, 1'-H, 8'-H), 7.78 (d, $^3J$ = 7.4 Hz, 2H, 4'-H, 5'-H). The spectrum is in full agreement with published data.[3]

**(9*H*-Fluoren-9-yl)methyl phenylacetate (3).** 9*H*-Fluoren-9-ylmethanol (**1**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **3** (207 mg, 86%) as a yellow oil. $R_f$ = 0.59 (hexanes/EtOAc, 3:1). IR (KBr): $\tilde{\nu}$ (cm$^{-1}$) = 3452 (w), 3064 (m), 3032 (m), 2949 (m), 2891 (w), 1732 (s, C=O), 1451 (s), 1255 (s), 1148 (s), 1007 (m), 758 (s), 740 (s), 543 (m), 426 (m). $^1$H NMR (400 MHz, $CDCl_3$) δ 3.71 (s, 2H, $CH_2$Ph), 4.18 (t, $^3J$ = 7.2 Hz, 1H, 9'-H), 4.39 (d, $^3J$ = 7.2 Hz, 2H, $OCH_2$), 7.22–7.42 (m, 11H, $H_{ar}$), 7.75 (d, $^3J$ = 7.5 Hz, 4'-H, 5'-H). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 41.8 (t), 46.8 (d), 66.9 (t), 120.1 (2 d), 125.2 (d), 127.2 (2 d), 127.3 (2 d), 127.9 (2 d), 128.8 (2 d), 129.5 (2 d), 134.0 (s), 141.4 (2 s), 143.8 (2 s), 171.6 (s). MS (EI, 90 °C): *m*/*z* (%) = 314 (10) [M$^+$], 179 (16), 178 (100), 165 (19), 91 (19). HRMS ($^{12}C_{22}{}^1H_{18}{}^{16}O_2$, EI): calcd. 314.1307 amu; found 314.1309 amu. Anal. calcd for $C_{22}H_{18}O_2$ (314.13): C 84.05, H 5.77; found C 83.64, H 5.64.

**(9*H*-Fluoren-9-yl)methyl benzoate (4).** 9*H*-Fluoren-9-ylmethanol (**1**) and benzonitrile were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **4** (100 mg, 44%) as a yellow solid. $R_f$ = 0.59 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 4.40 (t, $^3J$ = 7.2 Hz, 1H, 9'-H), 4.62 (d, $^3J$ = 7.2 Hz, 2H, $CH_2$), 7.33 (t, $^3J$ = 7.4 Hz, 2H, 2'-H, 7'-H), 7.43 (t, $^3J$ = 7.4 Hz, 2H, 3'-H, 6'-H), 7.50 (t, $^3J$ = 7.5 Hz, 2H, Ph), 7.61 (t, $^3J$ = 7.4 Hz, 1H, Ph), 7.67 (d, $^3J$ = 7.4 Hz, 2H, 1'-H, 8'-H), 7.80 (d, $^3J$ = 7.4 Hz, 2H, 4'-H, 5'-H), 8.11 (d, $^3J$ = 7.8 Hz, 2H, Ph). The spectrum is in full agreement with published data.[4]

**(9*H*-Fluoren-9-yl)methyl acrylate (5).** 9*H*-Fluoren-9-ylmethanol (**1**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **5** (100 mg, 52%) as a yellow oil. A similar reaction performed according to GP 2 yielded **5** (127 mg, 67%). $R_f$ = 0.56 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 4.28 (t, $^3J$ = 7.3 Hz, 1H, 9'-H), 4.46 (d, $^3J$ = 7.3 Hz, 2H, $CH_2$), 5.91 (d, $^3J$ = 10.3 Hz, 1H, 3-$H_{trans}$), 6.23 (dd, $^3J$ = 17.3 Hz, $^3J$ = 10.3 Hz, 1H, 2-H), 6.48 (d, $^3J$ = 17.3 Hz, 1H, 3-$H_{cis}$), 7.33 (t, $^3J$ = 7.4 Hz, 2H, 2'-H, 7'-H), 7.42 (t, $^3J$ = 7.4 Hz, 2H, 3'-H, 6'-H), 7.61 (d, $^3J$ = 7.4 Hz, 2H, 1'-H, 8'-H), 7.78 (d, $^3J$ = 7.4 Hz, 2H, 4'-H, 5'-H). The spectrum is in full agreement with published data.[5]

**1-Decyl acetate (7).** 1-Decanol (**6**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **7** (121 mg, 80%) as a colourless oil. $R_f$ = 0.63 (hexanes/EtOAc, 3:1).

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$^1$H NMR (300 MHz, $CDCl_3$) δ 0.88 (t, $^3J$ = 6.6 Hz, 3H, 10'-$H_3$), 1.20–1.36 [m, 14H, Me($CH_2$)$_7$], 1.61 (tt, $^3J$ = 6.8 Hz, $^3J$ = 6.4 Hz, 2H, 2'-$H_2$), 2.04 (s, 3H, OAc), 4.05 (t, $^3J$ = 6.8 Hz, 2H, 1'-$H_2$). The spectrum is in full agreement with published data.[6]

**1-Decyl phenylacetate (8).** 1-Decanol (**6**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **8** (177 mg, 85%) as a yellowish oil. $R_f$ = 0.69 (hexanes/EtOAc 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 0.89 (t, $^3J$ = 6.7 Hz, 3H, Me), 1.21–1.35 [m, 14H, Me($CH_2$)$_7$], 1.55–1.65 (m, 2H, 2'-$H_2$), 3.61 (s, 2H, $CH_2$Ph), 4.08 (t, $^3J$ = 6.7 Hz, 2H, 1'-$H_2$), 7.22–7.38 (m, 5H, Ph). The spectrum is in full agreement with published data.[7]

**1-Decyl benzoate (9).** 1-Decanol (**6**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 19:1) afforded **9** (45 mg, 23%) as a yellowish oil. $R_f$ = 0.69 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 0.88 (t, $^3J$ = 6.4 Hz, 3H, Me), 1.20−1.45 [m, 14H, Me($CH_2$)$_7$], 1.77 (tt, $^3J$ = 7.1 Hz, $^3J$ = 6.5 Hz, 2H, 2'-$H_2$), 4.31 (t, $^3J$ = 6.5 Hz, 2H, 1'-$H_2$), 7.44 (t, $^3J$ = 7.5 Hz, 2H, Ph), 7.55 (t, $^3J$ = 7.2 Hz, 1H, Ph), 8.05 (d, $^3J$ = 7.3 Hz, 2H, Ph). The spectrum is in full agreement with published data.[8]

**1-Decyl acrylate (10).** 1-Decanol (**6**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **10** (47 mg, 29%) as a colourless oil. A similar reaction performed according to GP 2 yielded **10** (64 mg, 40%). $R_f$ = 0.69 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 0.88 (t, $^3J$ = 6.6 Hz, 3H, Me), 1.23–1.40 [m, 14H, $H_3C(CH_2)_7$], 1.66 (tt, $^3J$ = 7.1 Hz, $^3J$ = 6.7 Hz, 2H, 2'-$H_2$), 4.15 (t, $^3J$ = 6.7 Hz, 2H, 1'-$H_2$), 5.81 (dd, $^2J$ = 1.6 Hz, $^3J$ = 10.4 Hz, 1H, 3-$H_{trans}$), 6.12 (dd, $^3J$ = 10.4 Hz, $^3J$ = 17.3 Hz, 1H, 2'-$H_2$), 6.40 (dd, $^2J$ = 1.6 Hz, $^3J$ = 17.3 Hz, 1H, 3-$H_{cis}$). The spectrum is in full agreement with published data.[9]

**6-Chlorohexyl acetate (12).** 6-Chlorohexanol (**11**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **12** (114 mg, 84%) as a colourless oil. $R_f$ = 0.56 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.31–1.53 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.64 (tt, $^3J$ = 6.8 Hz, $^3J$ = 7.2 Hz, 2H, 2'-$H_2$), 1.78 (tt, $^3J$ = 6.7 Hz, $^3J$ = 7.3 Hz, 2H, 5'-$H_2$), 2.05 (s, 3H, Me), 3.53 (t, $^3J$ = 6.7 Hz, 2H, 6'-$H_2$), 4.06 (t, $^3J$ = 6.7 Hz, 2H, 1'-$H_2$). The spectrum is in full agreement with published data.[10]

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**6-Chlorohexyl phenylacetate (13).** 6-Chlorohexanol (**11**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **13** (174 mg, 90%) as a yellowish oil. $R_f$ = 0.59 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.23–1.49 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.63 (tt, $^3J$ = 6.8 Hz, $^3J$ = 7.3 Hz, 2H, 5'-$H_2$), 1.75 (tt, $^3J$ = 6.8 Hz, $^3J$ =7.3 Hz, 2H, 2'-$H_2$), 3.51 (t, $^3J$ = 6.7 Hz, 2H, 6'-$H_2$), 3.62 (s, 2H, $CH_2$Ph), 4.09 (t, $^3J$ = 6.6 Hz, 2H, 1'-$H_2$), 7.23–7.40 (m, 5H, Ph). The spectrum is in full agreement with published data.[11]

**6-Chlorohexyl benzoate (14).** 6-Chlorohexanol (**11**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **14** (50 mg, 27%) as a yellowish oil. $R_f$ = 0.62 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.42–1.60 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.73–1.88 (m, 4H, 2'-$H_2$, 5'-$H_2$), 3.55 (t, $^3J$ = 6.6 Hz, 2H, 6'-$H_2$), 4.33 (t, $^3J$ = 6.6 Hz, 2H, 1'-$H_2$), 7.44 (t, $^3J$ = 7.5 Hz, 2H, Ph), 7.56 (t, $^3J$ = 7.4 Hz, 1H, Ph), 8.04 (d, $^3J$ = 7.8 Hz, 2H, Ph). The spectrum is in full agreement with published data.[12]

**6-Chlorohexyl acrylate (15).** 6-Chlorohexanol (**11**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **15** (55 mg, 38%) as a colourless oil. A similar reaction performed according to GP 2 yielded **15** (23 mg, 16%). $R_f$ = 0.64 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.33–1.55 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.69 (tt, $^3J$ = 6.7 Hz, $^3J$ = 7.2 Hz, 2H, 5'-$H_2$), 1.79 (tt, $^3J$ = 6.6 Hz, $^3J$ = 7.2 Hz, 2H, 2'-$H_2$), 3.54 (t, $^3J$ = 6.6 Hz, 2H, 6'-$H_2$), 4.16 (t, $^3J$ = 6.6 Hz, 2H, 1'-$H_2$), 5.82 (dd, $^2J$ = 1.1 Hz, $^3J$ = 10.4 Hz, 1H, 3-$H_{trans}$), 6.12 (dd, $^3J$ = 10.4 Hz, $^3J$ = 17.3 Hz, 1H, 2-H), 6.40 (dd, $^3J$ = 1.1 Hz, $^3J$ = 17.3 Hz, 1H, 3-$H_{cis}$). The spectrum is in full agreement with published data.[13]

**2-(2-Ethoxyethoxy)ethyl acetate (17)**. 2-(2-Ethoxyethoxy)ethanol (**16**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 4:1) afforded **17** (101 mg, 75%) as a colourless oil. $R_f$ = 0.16 (hexanes/EtOAc 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.21 (t, $^3J$ = 7.0 Hz, 3H, $OCH_2CH_3$), 2.07 (s, 3H, OAc), 3.53 (q, $^3J$ = 7.0 Hz, 2H, $OCH_2CH_3$), 3.56–3.67 (m, 4H, $EtOCH_2CH_2O$), 3.70 (t, $^3J$ = 4.8 Hz, 2H, $OCH_2CH_2OAc$), 4.23 (t, $^3J$ = 4.8 Hz, 2H, $OCH_2CH_2OAc$). The spectrum is in full agreement with published data.[14]

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**2-(2-Ethoxyethoxy)ethyl phenylacetate (18).** 2-(2-Ethoxyethoxy)ethanol (**16**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 4:1) afforded **18** (162 mg, 85%) as a colourless oil. $R_f$ = 0.20 (hexanes/EtOAc, 3:1). IR (KBr): $\tilde{\nu}$ ($cm^{-1}$) = 3450 (w), 2974 (m), 2870 (m), 1737 (m, C=O), 1454 (w), 1253 (m), 1114 (m), 1043 (w), 697 (w). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.21 (t, $^3J$ = 7.0 Hz, 3H, Me), 3.52 (q, $^3J$ = 7.0 Hz, 2H, $CH_2$Me), 3.53–3.62 (m, 4H, EtO$CH_2CH_2$O), 3.65 (s, 2H, $CH_2$Ph), 3.69 (t, $^3J$ = 4.8 Hz, 2H, O$CH_2$$CH_2$OAc), 4.26 (t, $^3J$ = 4.8 Hz, 2H, O$CH_2$$CH_2$OAc), 7.23–7.35 (m, 5H, Ph). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 15.3 (q), 41.4 (t), 64.2 (t), 66.8 (t), 69.2 (t), 69.9 (t), 70.8 (t), 127.2 (d), 128.7 (2 d), 129.4 (2 d), 134.1 (s), 171.7 (s). MS (EI, 20 °C): *m*/*z* (%) = 252 (36) [$M^+$], 163 (34), 119 (15), 118 (59), 92 (10), 91 (100), 73 (14), 72 (35), 59 (22), 45 (26). HRMS ($^{12}C_{14}{}^1H_{20}{}^{16}O_4$, EI): calcd. 252.1362 amu; found 252.1364 amu. Anal. calcd for $C_{14}H_{20}O_4$ (252.14): C 66.65, H 7.99; found C 66.79, H 7.89.

**2-(2-Ethoxyethoxy)ethyl benzoate (19).** 2-(2-Ethoxyethoxy)ethanol (**16**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 4:1) afforded **19** (47 mg, 26%) as a colourless oil. $R_f$ = 0.23 (hexane/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.20 (t, $^3J$ = 7.0 Hz, 3H, Me), 3.53 (q, $^3J$ = 7.0 Hz, 2H, $CH_2$Me), 3.58–3.73 (m, 4H, EtO$CH_2CH_2$O), 3.85 (t, $^3J$ = 4.8 Hz, 2H, O$CH_2$$CH_2$OAc), 4.49 (t, $^3J$ = 4.8 Hz, 2H, O$CH_2$$CH_2$OAc), 7.43 (t, $^3J$ = 7.5 Hz, 2H, Ph), 7.56 (t, $^3J$ = 7.4 Hz, 1H, Ph), 8.06 (d, $^3J$ = 7.7 Hz, 2H, Ph). The spectrum is in full agreement with published data.[15]

**2-(2-Ethoxyethoxy)ethyl acrylate (20).** 2-(2-Ethoxyethoxy)ethanol (**16**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 4:1) afforded **20** (32 mg, 23%) as a colourless oil. A similar reaction performed according to GP 2 yielded **20** (27 mg, 19%). $R_f$ = 0.27 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.21 (t, $^3J$ = 7.0 Hz, 3H, Me), 3.53 (q, $^3J$ = 7.0 Hz, 2H, $CH_2$Me), 3.57–3.69 (m, 4H, EtO$CH_2CH_2$O), 3.75 (t, $^3J$ = 4.8 Hz, 2H, O$CH_2$$CH_2$OAc), 4.32 (t, $^3J$ = 4.8 Hz, 2H, O$CH_2$$CH_2$OAc), 5.83 (dd, $^2J$ = 1.3 Hz, $^3J$ = 10.4, 1H, 3-$H_{trans}$), 6.15 (dd, $^3J$ = 10.4 Hz, $^3J$ = 17.3, 1H, 2-H), 6.43 (dd, $^2J$ = 1.3 Hz, $^3J$ = 17.3 Hz, 1H, 3-$H_{cis}$). The spectrum is in full agreement with published data.[16]

**4-Nitrobenzyl acetate** (**22**)**.** 4-Nitrobenzyl alcohol (**21**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 4:1) afforded **22** (133 mg, 90%) as a yellowish solid. $R_f$ = 0.36 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 2.15 (s, 3H, Me), 5.20 (s, 2H, $CH_2$), 7.52 (d, $^3J$ = 8.8 Hz, 2H, Ar), 8.22 (d, $^3J$ = 8.8 Hz, 2H, Ar). The spectrum is in full agreement with published data.[17]

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**4-Nitrobenzyl phenylacetate (23).** 4-Nitrobenzyl alcohol (**21**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **23** (160 mg, 78%) as a white solid. $R_f$ = 0.39 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 3.72 (s, 2H, Ph*C*$H_2$), 5.22 (s, 2H, $OCH_2$), 7.24–7.38 (m, 5H, Ph), 7.42 (d, $^3J$ = 8.7 Hz, 2H, Ar), 8.18 (d, $^3J$ = 8.7 Hz, 2H, Ar). The spectrum is in full agreement with published data.[18]

**4-Nitrobenzyl benzoate (24).** 4-Nitrobenzyl alcohol (**21**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **24** (77 mg, 39%) as a white solid. $R_f$ = 0.39 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 5.46 (s, 2H, $CH_2$), 7.47 (t, $^3J$ = 7.6 Hz, 2H, Ph), 7.60 (t, $^3J$ = 7.0 Hz, 1H, Ph), 7.61 (d, $^3J$ = 8.6 Hz, 2H, Ar), 8.09 (d, $^3J$ = 7.9 Hz, 2H, Ph), 8.25 (d, $^3J$ = 8.6 Hz, 2H, Ar). The spectrum is in full agreement with published data.[19]

**4-Nitrobenzyl acrylate (25).** 4-Nitrobenzyl alcohol (**21**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **25** (133 mg, 85%) as a yellowish solid. $R_f$ = 0.36 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 5.30 (s, 2H, *C*$H_2$Ar), 5.92 (d, $^3J$ = 10.4 Hz, 1H, 3-$H_{trans}$), 6.20 (dd, $^3J$ = 10.4 Hz, $^3J$ = 17.3 Hz, 1H, 2-H), 6.49 (d, $^3J$ = 17.3 Hz, 1H, 3-$H_{cis}$), 7.54 (d, $^3J$ = 8.6 Hz, 2H, Ar), 8.23 (d, $^3J$ = 8.6 Hz, 2H, Ar). The spectrum is in full agreement with published data.[20]

**4-(Acetoxymethyl)benzoic acid (27).** 4-(Hydroxymethyl)benzoic acid (**26**) and MeCN were reacted according to GP 1. Chromatography ($CH_2Cl_2$/MeOH/AcOH, 100:10:1) afforded **27** (130 mg, 88%) as a white solid. $R_f$ = 0.31 (MeOH/$CH_2Cl_2$, 19:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 2.15 (s, 3H, Me), 5.18 (s, 2H, $CH_2$), 7.46 (d, $^3J$ = 8.2 Hz, 2H, Ar), 8.12 (d, $^3J$ = 8.2 Hz, 2H, Ar), 11.0 (bs, 1H, $CO_2H$). The spectrum is in full agreement with published data.[21]

**4-[(Phenylacetoxy)methyl]benzoic acid (28).** 4-(Hydroxymethyl)benzoic acid (**26**) and BnCN were reacted according to GP 3. Chromatography ($CH_2Cl_2$/MeOH/AcOH, 100:10:1) afforded **28** (179 mg, 87%) as a white solid. $R_f$ = 0.29 (MeOH/$CH_2Cl_2$, 19:1). IR (ATR): $\tilde{\nu}$ ($cm^{-1}$) = 2943 (m), 2557 (m), 1719 (s, C=O), 1682 (s, C=O), 1613 (m), 1427 (m), 1291 (s), 1266 (s), 1144 (s), 1127 (s), 1029 (m), 750 (s), 718 (s), 692 (s). $^1$H NMR (400 MHz, $CDCl_3$) δ 3.71 (s, 2H, Ph*C*$H_2$), 5.21 (s, 2H, $OCH_2$), 7.27–7.37 (m, 5H, Ph), 7.38 (d, $^3J$ = 8.4 Hz, 2H, Ar), 8.08 (d, $^3J$ = 8.4 Hz, 2H, Ar), 11.1 (bs, 1H, COOH). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 41.5 (t), 65.9 (t), 127.4 (d), 127.7 (d), 128.8 (d), 129.1 (s), 129.4 (d), 130.6 (d),

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133.8 (s), 142.0 (s), 171.4 (s), 171.4 (s). MS (EI, 110 °C): *m*/*z* (%) = 270 (6) [$M^+$], 136 (4), 135 (35), 107 (25), 92 (10), 91 (100), 90 (11), 89 (9), 79 (4), 77 (9), 65 (14). HRMS ($^{12}C_{16}{}^{1}H_{14}{}^{16}O_4$, EI): calcd. 270.0892 amu; found 270.0893 amu. $C_{16}H_{14}O_4$ (270.09): calcd. C 71.10, H 5.22; found C 70.72, H 5.02.

**4-(Benzoyloxymethyl)benzoic acid (29).** 4-(Hydroxymethyl)benzoic acid (**26**) and PhCN were reacted according to GP 3. Chromatography ($CH_2Cl_2$/MeOH/AcOH, 100:10:1) afforded **29** (60 mg, 31%) as a white solid. $R_f$ = 0.31 (MeOH/$CH_2Cl_2$, 19:1) IR (ATR): $\tilde{\nu}$ ($cm^{-1}$) = 2955 (w), 2660 (w), 2540 (w), 1715 (w, C=O), 1677 (w, C=O), 1425 (w), 1264 (m), 1095 (w), 1069 (w), 935 (w), 756 (w), 699 (w). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.45 (s, 2H, $CH_2$), 7.47 (t, $^3J$ = 7.7 Hz, 2H, Ph), 7.55 (d, $^3J$ = 8.3 Hz, 2H, Ar), 7.59 (t, $^3J$ = 7.4 Hz, $^4J$ = 1.5 Hz, 1H, Ph), 8.10 (dd, $^3J$ = 8.4 Hz, $^4J$ = 1.4 Hz, 2H, Ph), 8.14 (d, $^3J$ = 8.3 Hz, 2H, Ar),10,8 (bs, 1H, COOH). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 66.0 (t), 127.8 (d), 128.6 (d), 129.2 (s), 129.9 (d), 130.7 (d), 133.4 (d), 142.2 (s), 166.4 (s), 171.9 (s), one signal (s) covered. MS (EI, 100 °C): *m*/*z* (%) = 257 (6), 256 (42) [$M^+$], 135 (30), 121 (10), 107 (26), 106 (13), 105 (100), 90 (7), 89 (10), 79 (9), 78 (6), 77 (52), 51 (15), 43 (13). HRMS ($^{12}C_{15}{}^{1}H_{12}{}^{16}O_4$, EI): calcd. 256.0736 amu; found 256.0734 amu.

**4-(Acryloyloxymethyl)benzoic acid (30).** 4-(Hydroxymethyl)benzoic acid (**26**) and acrylonitrile were reacted according to GP 1. Chromatography ($CH_2Cl_2$/MeOH/AcOH, 100:10:1) afforded **30** (98 mg, 64%) as a white solid. $R_f$ = 0.27 (MeOH/$CH_2Cl_2$, 19:1). $^1$H NMR (400 MHz, $CDCl_3$) δ 5.28 (s, 2H, $OCH_2$), 5.90 (d, $^3J$ = 10.4 Hz, 1H, 3-$H_{cis}$), 6.20 (dd, $^3J$ = 10.4 Hz, $^3J$ = 17.3 Hz, 1H, 2-H), 6.49 (d, $^3J$ = 17.3 Hz, 1H, 3-$H_{trans}$), 7.48 (d, $^3J$ = 7.8 Hz, 2H, Ar), 8.12 (d, $^3J$ = 7.8 Hz, 2H, Ar), 11.4 (bs, 1H, COOH). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 65.6 (t), 127.9 (d), 128.1 (d), 129.2 (s), 130.7 (d), 131.8 (t), 142.1 (s), 166.0 (s), 171.6 (s).

**Hexane-1,6-diyl diacetate (32) and 6-hydroxyhexyl acetate (33).** 1,6-Hexanediol (**31**) and MeCN were reacted according to GP 1 with 4 equivalents of TMSOTf. Chromatography (hexanes/EtOAc, 9:1) afforded **32** (48 mg, 32%) as a colourless oil. $R_f$ = 0.45 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.33–1.42 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.56–1.68 (m, 4H, 2'-$H_2$, 5'-$H_2$), 2.04 (s, 6H, Me), 4.05 (t, $^3J$ = 6.7 Hz, 4H, 1'-$H_2$, 6'-$H_2$). The spectrum is in full agreement with published data.[22] **Side product: 6-Hydroxyhexyl acetate (33).** Chromatography (hexanes/EtOAc, 9:1) afforded **33** (22 mg, 19%) as a colourless oil. $R_f$ = 0.29 (hexanes/EtOAc, 1:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.36–1.45 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.53–1.71 (m, 4H, 2'-$H_2$, 5'-$H_2$), 1.90 (br s, 1H, OH), 2.05 (s, 3H, OAc), 3.65 (t, $^3J$ = 6.5 Hz, 2H, 6'-$H_2$), 4.06 (t, $^3J$ = 6.7 Hz, 2H, 1'-$H_2$). The spectrum is in full agreement with published data.[23]

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**Hexane-1,6-diyl bis(phenylacetate) (34) and 6-hydroxyhexyl phenylacetate (35).** 1,6 Hexanediol (**31**) and BnCN were reacted according to GP 3 with 4 equivalents of TMSOTf. Chromatography (hexanes/EtOAc, 9:1) afforded **34** (122 mg, 46%) as a colourless oil. $R_f$ = 0.45 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.24–1.36 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.52–1.64 (m, 4H, 2'-$H_2$, 5'-$H_2$), 3.61 (s, 4H, $CH_2$Ph), 4.06 (t, $^3J$ = 6.6 Hz, 2H, 1'-$H_2$, 6'-$H_2$), 7.22–7.36 (m, 10H, Ph). The spectrum is in full agreement with published data.[24] **Side product: 6-Hydroxyhexyl phenylacetate (35):** Chromatography (hexanes/EtOAc, 9:1) afforded **35** (66 mg, 37%) as a colourless oil. $R_f$ = 0.29 (hexanes/EtOAc, 1:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.29–1.40 (m, 4H, 3'-$H_2$, 4'-$H_2$), 1.49–1.58 (m, 2H, 5'-$H_2$), 1.63 (tt, $^3J$ = 6.9 Hz, $^3J$ = 6.8 Hz, 2H, 2'-$H_2$), 3.61 (s, 2H, $CH_2$Ph), 3.62 (t, $^3J$ = 6.5 Hz, 2H, 6'-$H_2$), 4.09 (t, $^3J$ = 6.6 Hz, 2H, 1'-$H_2$), 7.23−7.36 (m, 5H, Ph). The spectrum is in full agreement with published data.[25]

**Ethyl 6-acetoxyhexanoate (37).** Ethyl 6-hydroxyhexanoate (**36**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **37** (32 mg, 21%) as a colourless oil. $R_f$ = 0.47 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.25 (t, $^3J$ = 7.1 Hz, 3H, $CH_2CH_3$), 1.32–1.44 (m, 2H, 4-$H_2$), 1.59–1.71 (m, 4H, 3-$H_2$, 5-$H_2$), 2.04 (s, 3H, OAc), 2.30 (t, $^3J$ = 7.5 Hz, 2H, 2-$H_2$), 4.05 (t, $^3J$ = 6.6 Hz, 2H, 6-$H_2$), 4.12 (q, $^3J$ = 7.1 Hz, 2H, $CH_2CH_3$). The spectrum is in full agreement with published data.[26]

**Ethyl 6-(phenylacetoxy)hexanoate (38).** Ethyl 6-hydroxyhexanoate (**36**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **38** (33 mg, 16%) as a yellowish oil. $R_f$ = 0.44 (hexanes/EtOAc, 3:1). IR (KBr): $\tilde{\nu}$ ($cm^{-1}$) = 3444 (w), 2940 (m), 1733 (s, C=O), 1455 (w), 1249 (m), 1110 (s), 1031 (m), 698 (w), 617 (m). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.25 (t, $^3J$ = 7.1 Hz, 3H, Me), 1.29–1.39 (m, 2H, 4-$H_2$), 1.58−1.67 (m, 4H, 3-$H_2$, 5-$H_2$), 2.27 (t, $^3J$ = 7.5 Hz, 2H, 2-$H_2$), 3.61 (s, 2H, $CH_2$Ph), 4.08 (t, $^3J$ = 6.6 Hz, 2H, 6-$H_2$), 4.12 (q, $^3J$ = 7.1 Hz, 2H, $CH_2CH_3$), 7.23–7.35 (m, 5H, Ph). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.4 (q), 24.7 (t), 25.6 (t), 28.4 (t), 34.3 (t), 41.6 (t), 60.4 (t), 64.8 (t), 127.2 (d), 128.7 (2 d), 129.4 (2 d), 134.3 (s), 171.8 (s), 173.7 (s). MS (EI, 40 °C): *m*/*z* (%) = 278 (15) [$M^+$], 118 (100), 115 (15), 91 (60), 69 (16). HRMS ($^{12}C_{16}{}^1H_{22}{}^{16}O_4$, EI): calcd. 278.1518 amu; found 278.1516. amu.

**6-Ethoxy-6-oxohexyl benzoate (39).** Ethyl 6-hydroxyhexanoate (**36**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **39** (21 mg, 10%) as a yellowish oil. $R_f$ = 0.44 (hexanes/EtOAc, 3:1). IR (KBr): $\tilde{\nu}$ ($cm^{-1}$) = 3440 (w), 2940 (w), 1721 (m, C=O), 1275 (m), 1113 (s),

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713 (m), 618 (m). $^{1}$H NMR (400 MHz, $CDCl_3$) δ 1.25 (t, $^{3}J$ = 7.1 Hz, 3H, $CH_2CH_3$), 1.44–1.54 (m, 2H, 4'-$H_2$), 1.71 (tt, $^{3}J$ = 7.5 Hz, $^{3}J$ = 7.7 Hz, 2H, 3'-$H_2$), 1.79 (tt, $^{3}J$ = 6.6 Hz, $^{3}J$ = 7.6 Hz, 2H, 5'-$H_2$), 2.33 (t, $^{3}J$ = 7.5 Hz, 2H, 2'-$H_2$), 4.12 (q, $^{3}J$ = 7.1 Hz, 2H, $CH_2CH_3$), 4.32 (t, $^{3}J$ = 6.6 Hz, 2H, 6'-$H_2$), 7.44 (t, $^{3}J$ = 7.7 Hz, 2H, Ph), 7.55 (t, $^{3}J$ = 7.4 Hz, 1H, Ph), 8.04 (d, $^{3}J$ = 7.9 Hz, 2H, Ph). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.4 (q), 24.8 (t), 25.8 (t), 28.6 (t), 34.4 (t), 60.4 (t), 64.9 (t), 128.5 (2 d), 129.7 (2 d), 130.5 (s), 133.0 (d), 166.8 (s), 173.7 (s). MS (EI, 40 °C): *m*/*z* (%) = 264 (8) [$M^+$], 142 (49), 105 (100), 88 (12), 77 (14). HRMS ($^{12}C_{15}{}^{1}H_{20}{}^{16}O_4$, EI): calcd. 264.1362 amu; found 264.1360 amu.

**Ethyl 6-(acryloyloxy)hexanoate (40).** Ethyl 6-hydroxyhexanoate (**36**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **40** (12 mg, 7%) as a colourless oil. $R_f$ = 0.38 (hexanes/EtOAc, 3:1). IR (KBr): $\tilde{\nu}$ ($cm^{-1}$) = 3439 (w), 2942 (m), 1729 (s, C=O), 1409 (m), 1192 (m), 811 (w), 618 (w). $^{1}$H NMR (400 MHz, $CDCl_3$) δ 1.25 (t, $^{3}J$ = 7.1 Hz, 3H, $CH_2CH_3$), 1.36–1.45 (m, 2H, 4-$H_2$), 1.61–1.73 (m, 4H, 3-$H_2$, 5-$H_2$), 2.31 (t, $^{3}J$ = 7.5 Hz, 2H, 2-$H_2$), 4.12 (q, $^{3}J$ = 7.1 Hz, 2H, $CH_2CH_3$), 4.15 (t, $^{3}J$ = 6.7 Hz, 2H, 6-$H_2$), 5.81 (dd, $^{3}J$ = 10.4 Hz, $^{2}J$ = 1.3 Hz, 1H, 3'-$H_{trans}$), 6.11 (dd, $^{3}J$ = 17.3 Hz, $^{3}J$ = 10.4 Hz, 1H, 2'-$H_α$), 6.39 (dd, $^{3}J$ = 17.3 Hz, $^{2}J$ = 1.3 Hz, 1H, 3'-$H_{cis}$). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 14.4 (q), 24.7 (t), 25.7 (t), 28.4 (t), 34.3 (t), 60.4 (t), 64.5 (t), 128.7 (d), 130.7 (t), 166.4 (s), 173.7 (s). MS (EI, 30 °C): *m*/*z* (%) = 214 (1) [$M^+$], 169 (21) [$M^+ - OCH_2CH_3$], 142 (58), 114 (11), 113 (13), 88 (25), 73 (14), 69 (16), 68 (26), 55 (100) [$OCHCH_2$]. HRMS ($^{12}C_{11}{}^{1}H_{18}{}^{16}O_4$, EI): calcd. 214.1205 amu; found 214.1208 amu.

**4-(Benzyloxycarbonylamino)butyl acetate (42).** Benzyl 4-hydroxybutylcarbamate (**41**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 2:1) afforded **42** (27 mg, 13%) as a colourless oil. $R_f$ = 0.55 (hexanes/EtOAc, 1:2). $^{1}$H NMR (300 MHz, $CDCl_3$) δ 1.50–1.73 (m, 4H, 2'-$H_2$, 3'-$H_2$), 2.04 (s, 3H, OAc), 3.23 (q, $^{3}J$ = 6.4 Hz, 2H, 1'-$H_2$), 4.07 (t, $^{3}J$ = 6.1 Hz, 2H, 4'-$H_2$), 4.79 (s, 1H, NH), 5.10 (s, 2H, $CH_2$Ph), 7.28–7.39 (m, 5H, Ph). The spectrum is in full agreement with published data.[27]

**4-(Benzyloxycarbonylamino)butyl phenylacetate (43).** Benzyl 4-hydroxybutylcarbamate (**41**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 3:1) afforded **43** (34 mg, 13%) as a yellowish oil. $R_f$ = 0.71 (hexanes/EtOAc, 1:2). IR (KBr): $\tilde{\nu}$ ($cm^{-1}$) = 3347 (m), 2951 (m), 1727 (s, C=O), 1530 (m), 1455 (m), 1253 (s), 1141 (m), 1027 (m), 727 (m), 697 (m). $^{1}$H NMR (400 MHz, $CDCl_3$) δ 1.51 (tt, $^{3}J$ = 7.0 Hz, $^{3}J$ = 7.5 Hz, 2H, 2'-$H_2$), 1.64 (tt, $^{3}J$ = 7.4 Hz, $^{3}J$ = 6.5 Hz, 2H, 3'-$H_2$), 3.18 (q, $^{3}J$ = 6.6 Hz, 2H, 1'-$H_2$), 3.61 (s, 2H, C$CH_2$Ph), 4.10 (t, $^{3}J$ = 6.4 Hz, 2H, 4'-$H_2$), 4.72 (s,

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1H, NH), 5.10 (s, 2H, O*CH*$_2$Ph), 7.23–7.40 (m, 10H, Ph). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 25.9 (t), 26.7 (t), 40.7 (t), 41.6 (t), 64.5 (t), 66.8 (t), 127.2 (2 d), 128.3 (2 d), 128.7 (2 d), 128.7 (2 d), 129.4 (2 d), 134.2 (s), 136.7 (s), 156.5 (s), 171.7 (s). MS (EI, 80 °C): *m*/*z* (%) = 341 (5) [$M^+$], 180 (12), 118 (11), 108 (64), 107 (23), 92 (15), 91 (100). HRMS ($^{12}C_{20}{}^{1}H_{23}{}^{14}N^{16}O_4$, EI): calcd. 341.1627 amu; found 341.1625 amu.

**4-(Benzyloxycarbonylamino)butyl acrylate (44).** Benzyl 4-hydroxybutylcarbamate (**41**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 1:1) afforded **44** (30 mg, 14%) as a yellowish oil. $R_f$ = 0.62 (hexanes/EtOAc, 1:2). IR (KBr): $\tilde{\nu}$ ($cm^{-1}$) = 3346 (m), 2954 (m), 1723 (m, C=O), 1532 (m), 1454 (m), 1410 (m), 1256 (m), 1194 (m), 1135 (m), 1062 (w), 1027 (w), 985 (w), 812 (w), 739 (w), 698 (w). $^1$H NMR (400 MHz, $CDCl_3$) δ 1.52–1.76 (m, 4H, 2'-$H_2$, 3'-$H_2$), 3.24 (q, $^3J$ = 6.6 Hz, 2H, 1'-$H_2$), 4.17 (t, $^3J$ = 6.3 Hz, 2H, 4'-$H_2$), 4.81 (s, 1H, NH), 5.09 (s, 2H, *CH*$_2$Ph), 5.82 (dd, $^3J$ = 10.4 Hz, $^2J$ = 1.4 Hz, 1H, 3-$H_{trans}$), 6.11 (dd, $^3J$ = 17.3 Hz, $^3J$ = 10.4 Hz, 1H, 2-H), 6.40 (dd, $^3J$ = 17.3 Hz, $^2J$ = 1.4 Hz, 1H, 3-$H_{cis}$), 7.28−7.40 (m, 5H, Ph). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 26.1 (t), 26.8 (t), 40.8 (t), 64.2 (t), 66.8 (t), 128.0 (2 d), 128.3 (d), 128.6 (d), 128.7 (2 d), 130.9 (t), 136.7 (s), 156.5 (s), 166.3 (s). MS (EI, 50 °C): *m*/*z* (%) = 277 (1) [$M^+$], 205 (51), 160 (10), 98 (14), 92 (12), 91 (100), 65 (9). HRMS ($^{12}C_{15}{}^{1}H_{19}{}^{14}N^{16}O_4$, EI): calcd. 277.1314 amu; found 277.1317 amu.

**Cyclohexyl acetate (46).** Cyclohexanol (**45**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **46** (16 mg, 15%) as a colourless oil. $R_f$ = 0.50 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.19–1.42 (m, 5H, 3'-$H_2$, 4'-$H_a$$H_b$, 5'-$H_2$), 1.48–1.58 (m, 1H, 4'-$H_a$$H_b$), 1.65–1.76 (m, 2H, 2'-$H_2$ and/or 6'-$H_2$), 1.79–1.90 (m, 2H, 2'-$H_2$ and/or 6'-$H_2$), 2.02 (s, 3H, Me), 4.72 (tt, $^3J$ = 4.2 Hz, $^3J$ = 9.1 Hz, 1H, 1'-H). The spectrum is in full agreement with published data.[28]

**Cyclohexyl phenylacetate (47).** Cyclohexanol (**45**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 9:1) afforded **47** (42 mg, 25%) as a colourless oil. $R_f$ = 0.59 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 1.19–1.56 (m, 6H, 3'-$H_2$, 4'-$H_2$, 5'-$H_2$), 1.61–1.71 (m, 2H, 2'-$H_2$ and/or 6'-$H_2$), 1.76–1.88 (m, 2H, 2'-$H_2$ and/or 6'-$H_2$), 3.60 (s, 2H, 2-$H_2$), 4.77 (tt, $^3J$ = 3.7 Hz, $^3J$ = 8.8 Hz, 1H, 1'-H), 7.22–7.36 (m, 5H, Ph). The spectrum is in full agreement with published data.[29]

**2-(4-Hydroxyphenyl)ethyl acetate (54).** 4-(2-Hydroxyethyl)phenol (**53**) and MeCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 4:1) afforded **54** (89 mg, 65%) as a yellowish oil.

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29 Yang, C.-G.; He, C. *J. Am. Chem. Soc.*, **2005**, *127*, 6966–6967.

$R_f$ = 0.24 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 2.04 (s, 3H, Me), 2.86 (t, $^3J$ = 7.1 Hz, 2H, 2'-$H_2$), 4.24 (t, $^3J$ = 7.1 Hz, 2H, 1'-$H_2$), 6.77 (d, $^3J$ = 8.4 Hz, 2H, Ar), 7.08 (d, $^3J$ = 8.4 Hz, 2H, Ar). The spectrum is in full agreement with published data.[30]

**2-(4-Hydroxyphenyl)ethyl phenylacetate, monaspilosin** (**55**). 4-(2-Hydroxyethyl)phenol (**53**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 4:1) afforded **55** (142 mg, 73%) as a colourless wax. $R_f$ = 0.24 (hexanes/EtOAc, 3:1). IR (ATR): $\tilde{\nu}$ ($cm^{-1}$) = 3315 (m), 2955 (w), 1690 (s, C=O), 1594 (m), 1514 (m), 1452 (m), 1221 (s), 1147 (s), 1015 (s). $^1$H NMR (400 MHz, $CDCl_3$) δ 2.84 (t, $^3J$ = 7.0 Hz, 2H, 2'-$H_2$), 3.61 (s, 2H, PhC$H_2$), 4.26 (t, $^3J$ = 7.0 Hz, 2H, 1'-$H_2$), 5.21 (s, 1H, OH), 6.72 (d, $^3J$ = 8.5 Hz, 2H, Ar), 6.99 (d, $^3J$ = 8.5 Hz, 2H, Ar), 7.22−7.35 (m, 5H, Ph). $^{13}$C NMR (100 MHz, $CDCl_3$) δ 34.3 (t), 41.6 (t), 65.8 (t), 115.5 (2 d), 127.2 (d), 128.7 (2 d), 129.4 (2 d), 129.9 (s), 130.2 (2 d), 134.1 (s), 154.4 (s), 171.9 (s). MS (EI, 100 °C): *m*/*z* (%) = 256 (4) [$M^+$], 121 (40), 120 (100), 107 (49), 91 (72), 77 (16). HRMS ($^{12}C_{16}{}^1H_{16}{}^{16}O_3$, EI): calcd. 256.1099 amu; found 256.1101 amu. $C_{16}H_{16}O_3$ (256.11): calcd. C 74.98, H 6.29; found C 74.90, H 6.28.

**Benzyl acetate (57) and *N*-benzylacetamide (58).** Benzyl alcohol (**56**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 9:1) afforded **57** (47 mg, 41%) as a colourless oil. $R_f$ = 0.50 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 2.11 (s, 3H, Me), 5.11 (s, 2H, $CH_2$), 7.28–7.40 (m, 5H, Ph). The spectrum is in full agreement with published data.[31] **Side product: *N*-Benzylacetamide (58):** Chromatography (hexanes/EtOAc, 1:3) afforded **58** (20 mg, 18%) as a white solid. $R_f$ = 0.21 (hexanes/EtOAc, 1:2). $^1$H NMR (300 MHz, $CDCl_3$) δ 2.00 (s, 3H, $CH_3$), 4.41 (d, $^3J$ = 5.7 Hz, 2H, $CH_2$), 5.97 (s, 1H, NH), 7.23–7.40 (m, 5H, Ph). The spectrum is in full agreement with published data.[32]

**Benzyl phenylacetate (59) and *N*-benzyl-phenylacetamide (60)**. Benzyl alcohol (**56**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 1:2) afforded amide **60** (100 mg, 59%) as a white solid. $R_f$ = 0.71 (hexanes/EtOAc, 1:2). $^1$H NMR (300 MHz, $CDCl_3$) δ 3.63 (s, 2H, Ph$CH_2$CO), 4.41 (d, $^3J$ = 5.8 Hz, 2H, NH$CH_2$Ph), 5.71 (s, 1H, NH), 7.18 (d, $^3J$ = 6.5 Hz, 2H, Ph), 7.21–7.40 (m, 8H, Ph). The spectrum is in full agreement with published data.[33] **Side product: Benzyl phenylacetate (59):** Chromatography (hexanes/EtOAc, 9:1) afforded **59** (30 mg, 18%) as a yellowish oil. $R_f$ = 0.53

30 Seidel, G.; Laurich, D.; Fürstner, A. *J. Org. Chem.*, **2004**, *69*, 3950–3952.
31 Débieux, J.-L.; Cosandey, A.; Helgen, C.; Bochet, C. G. *Eur. J. Org. Chem.*, **2007**, 2073–2077.
32 Bia, N.-M.; Rena, M.-G.; Song, Q.-H. *Synth. Commun.*, **2010**, *40*, 2617–2623.
33 Chen, Z.-W.; Jiang, H.-F.; Pan, X.-Y., He, Z.-J. *Tetrahedron*, **2011**, *67*, 5920–5927.

(hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 3.68 (s, 2H, Ph$CH_2$CO), 5.14 (s, 2H, $OCH_2$), 7.19–7.45 (m, 10H, Ph). The spectrum is in full agreement with published data.[34]

***N*-Benzylbenzamide** (**61**). Benzyl alcohol (**56**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 1:2) afforded **61** (105 mg, 66%) as a white solid. $R_f$ = 0.70 (hexanes/EtOAc, 1:2). $^1$H NMR (300 MHz, $CDCl_3$) δ 4.66 (d, $^3J$ = 5.6 Hz, 2H, $CH_2$), 6.39 (s, 1H, NH), 7.28–7.53 (m, 8H, Ph), 7.79 (d, $^3J$ = 7.6 Hz, 2H, Ph). The spectrum is in full agreement with published data.[35]

**Benzyl acrylate (62) and *N*-benzylacrylamide (63).** Benzyl alcohol (**56**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 1:1) afforded **63** (110 mg, 90%) as a white solid. $R_f$ = 0.40 (hexanes/EtOAc, 1:2). $^1$H NMR (300 MHz, $CDCl_3$) δ 4.52 (d, $^3J$ = 5.8 Hz, 2H, NH$CH_2$Ph), 5.67 (dd, $^3J$ = 10.2 Hz, $^2J$ = 1.4 Hz, 1H, 3-$H_{trans}$), 5.93 (s, 1H, NH), 6.11 (dd, $^3J$ = 17.0 Hz, 10.2 Hz, 1H, 2-H), 6.33 (dd, $^3J$ = 17.0 Hz, $^2J$ = 1.4 Hz), 7.27−7.38 (m, 5H, Ph). The spectrum is in full agreement with published data.[36] **Side product: Benzyl acrylate (62):** Chromatography (hexanes/EtOAc, 1:1) afforded **62** (5 mg, 4%) as a colourless oil. $R_f$ = 0.60 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 5.20 (s, 2H, $CH_2$), 5.85 (dd, $^2J$ = 1.5 Hz, $^3J$ = 10.4 Hz, 1H, 3-$H_{trans}$), 6.17 (dd, $^3J$ = 10.4 Hz, $^3J$ = 17.3 Hz, 1H, 2-H), 6.46 (dd, $^3J$ = 1.5 Hz, $^3J$ = 17.3 Hz, 1H, 3-$H_{cis}$), 7.28−7.41 (m, 5H, Ph). The spectrum is in full agreement with published data.[37]

**4-Fluorobenzyl acetate (65) and *N*-(4-fluorobenzyl)acetamide (66).** 4-Fluorobenzyl alcohol (**64**) and MeCN were reacted according to GP 1. Chromatography (hexanes/EtOAc, 3:1) afforded **66** (80 mg, 63%) as a white solid. $R_f$ = 0.13 (hexanes/EtOAc, 1:2). $^1$H NMR (300 MHz, acetone-$D_6$) δ 1.93 (s, 3H, $CH_3$), 4.34 (d, $^3J$ = 6.0 Hz, 2H, $CH_2$Ar), 7.05 (t, $^3J$ = 8.8 Hz, 2H, Ar), 7.33 (dd, $^4J$ = 5.6 Hz, $^3J$ = 8.5 Hz, 2H, Ar), 7.64 (s, 1H, NH). The spectrum is in full agreement with published data.[38] **Side product: 4-Fluorobenzyl Acetate (65):** Chromatography (hexanes/EtOAc, 2:1) afforded **65** (37 mg, 29%) as a colourless oil. $R_f$ = 0.44 (hexanes/EtOAc, 3:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 2.09 (s, 3H, Me), 5.07 (s, 2H, $CH_2$), 7.05 (t, $^3J$ = 8.6 Hz, 2H, Ar), 7.34 (dd, $^3J$ = 8.3 Hz, $^4J$ = 5.6 Hz, 2H, Ar). The spectrum is in full agreement with published data.[39]

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34 Ginisty, M.; Roy, M.-N.; Charette, A. B. *J. Org. Chem.*, **2008**, *73*, 2542–2547.
35 Matsumoto, K.; Hashimoto, S.; Uchida, T.; Okamoto, T.; Otani, S. *Chem. Ber.*, **1989**, *122*, 1357–1363.
36 Miyabe, H.; Ueda, M.; Nishimura, A.; Naito, T. *Tetrahedron* **2004**, *60*, 4227–4235.
37 Grasa, G. A.; Güveli, T.; Singh, R.; Nolan, S. P. *J. Org. Chem.*, **2003**, *68*, 2812–2819.
38 Shine, S. J.; Yueh, W. *J. Org. Chem.*, **1994**, *59*, 3553–3559.
39 Kadam, S. T.; Kim, S. S. *Synthesis*, **2008**, 267–271.

***N*-(4-Fluorobenzyl)phenylacetamide (67).** 4-Fluorobenzyl alcohol (**64**) and BnCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 3:1) afforded **67** (146 mg, 79%) as a white solid. $R_f$ = 0.18 (hexanes/EtOAc, 2:1). IR (ATR): $\tilde{\nu}$ ($cm^{-1}$) = 3280 (m), 3061 (w), 1641 (s, C=O), 1603 (m), 1548 (m), 1504 (m), 1491 (m), 1452 (m), 1432 (m), 1224 (m), 1028 (m), 823 (m), 752 (m), 716 (m), 694 (s), 569 (m). $^1$H NMR (400 MHz, $CDCl_3$) δ 3.63 (s, 2H, $CH_2$Ph), 4.37 (d, $^3J$ = 5.8 Hz, 2H, $CH_2$Ar), 5.67 (s, 1H, NH), 6.97 (t, $^3J$ = 8.6 Hz, 2H, Ar), 7.15 (dd, $^4J$ = 5.6 Hz, $^3J$ = 8.1 Hz, 2H, Ar), 7.24–7.39 (m, 5H, Ph). $^{13}$C NMR (75 MHz, $CDCl_3$) δ 43.0 (t), 43.9 (t), 115.6 (2 d), 127.6 (d), 129.2 (2 d), 129.3 (2 d), 129.6 (2 d), 134.1 (s), 134.8 (s), 162.2 (s), 171.0 (s). MS (EI, 80 °C): *m*/*z* (%) = 244 (12), 243 (97) [$M^+$], 109 (100), 92 (15), 91 (49). HRMS ($^{12}C_{15}{}^1H_{14}{}^{19}F^{14}N^{16}O$, EI): calcd. 243.1059 amu; found 243.1057 amu. Anal. calcd for $C_{15}H_{14}FNO$ (243.11): C 74.06, H 5.80, N 5.76; found C 74.29, H 5.78, N 5.56.

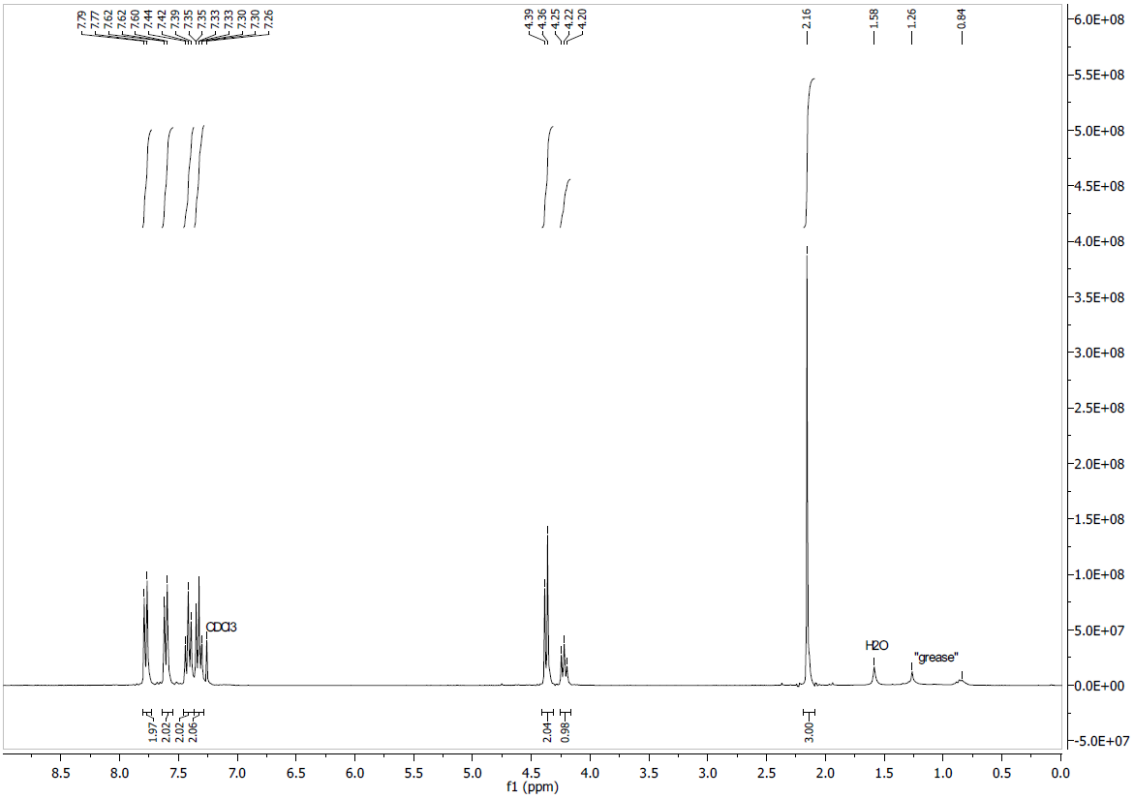
***N*-(4-Fluorobenzyl)benzamide (68).** 4-Fluorobenzyl alcohol (**64**) and PhCN were reacted according to GP 3. Chromatography (hexanes/EtOAc, 1:1) afforded **68** (122 mg, 70%) as a white solid. $R_f$ = 0.22 (hexanes/EtOAc, 2:1). $^1$H NMR (300 MHz, $CDCl_3$) δ 4.63 (d, $^3J$ = 5.7 Hz, 2H, $CH_2$), 6.40 (s, 1H, NH), 7.04 (t, $^3J$ = 8.5 Hz, 2H, Ar), 7.34 (dd, $^4J$ = 5.6 Hz, $^3J$ = 8.1 Hz, 2H, Ar), 7.39−7.55 (m, 3H, Ph), 7.79 (d, $^3J$ = 7.9 Hz, 2H, Ph). The spectrum is in full agreement with published data.[40]

***N*-(4-Fluorobenzyl)acrylamide (69).** 4-Fluorobenzyl alcohol (**64**) and acrylonitrile were reacted according to GP 1. Chromatography (hexanes/EtOAc, 1:2) afforded **69** (121 mg, 89%) as a white solid. $R_f$ = 0.29 (hexanes/EtOAc, 1:1). IR (ATR): $\tilde{\nu}$ ($cm^{-1}$) = 3270 (w), 1654 (m, C=O), 1620 (m), 1543 (m), 1509 (m), 1213 (m), 980 (m), 949 (m), 834 (m), 698 (m), 553 (m), 486 (m). $^1$H NMR (400 MHz, $CDCl_3$) δ 4.49 (d, $^3J$ = 5.8 Hz, 2H, Ar$CH_2$), 5.69 (dd, $^3J$ = 10.3 Hz, $^2J$ = 1.3 Hz, 1H, 3-$H_{trans}$), 6.10 (s, 1H, NH), 6.14 (dd, $^3J$ = 17.0 Hz, $^3J$ = 10.3 Hz, 1H, 2-H), 6.33 (dd, $^3J$ = 17,0 Hz, $^2J$ = 1.3 Hz, 1H, 3-$H_{cis}$), 7.03 (t, $^3J$ = 8.6 Hz, 2H, Ar), 7.25–7.32 (m, 2H, Ar). $^{13}$C NMR (400 MHz, $CDCl_3$) δ 43.1 (t), 115.7 (2 d), 127.1 (t), 129.7 (2 d), 130.7 (d), 134.0 (s), 162.3 (s), 165.6 (s). MS (EI, 60 °C): *m*/*z* (%) = 180 (14), 179 (100) [$M^+$], 178 (18), 136 (9), 135 (38), 124 (37), 122 (10), 109 (45), 55 (31). HRMS ($^{12}C_{10}{}^1H_{10}{}^{19}F^{14}N^{16}O$, EI): calcd. 179.0746 amu; found 179.0744 amu. $C_{10}H_{10}FNO$ (179.07): calcd. C 67.03, H 5.62, N 7.82; found C 66.84, H 5.58, N 7.69.

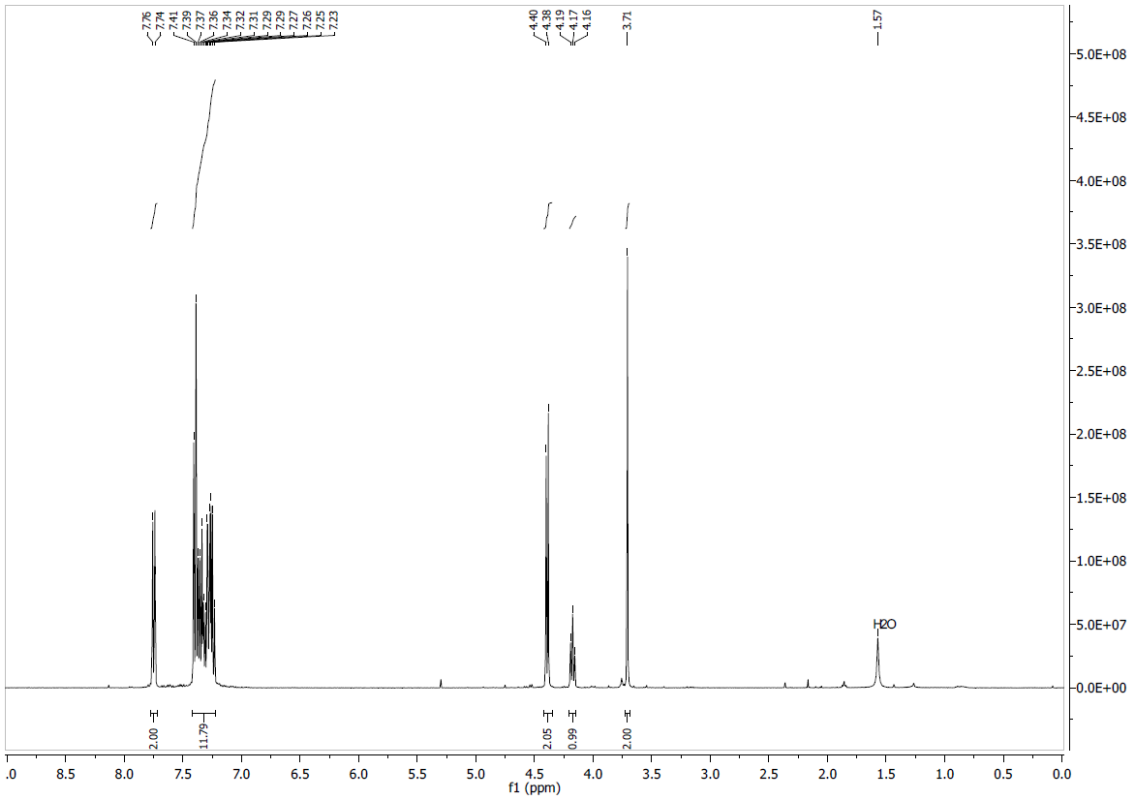
[40] Khalafi-Nezhad, A.; Foroughi, H. O.; Doroodmand, M. M.; Panahi, F. *J. Mater. Chem.*, **2011**, *21*, 12842–12851.

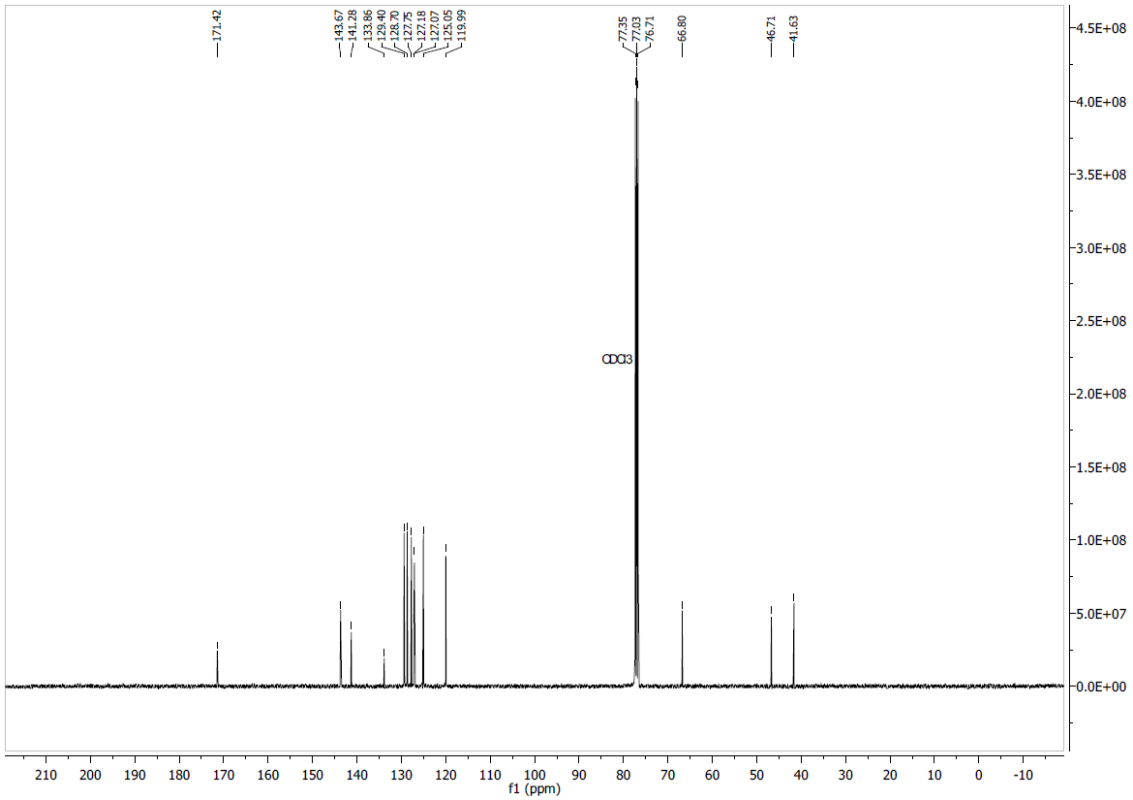
# B. $^{1}$H and $^{13}$C NMR spectra

## 1. (9*H*-Fluoren-9-yl)methyl acetate (2).

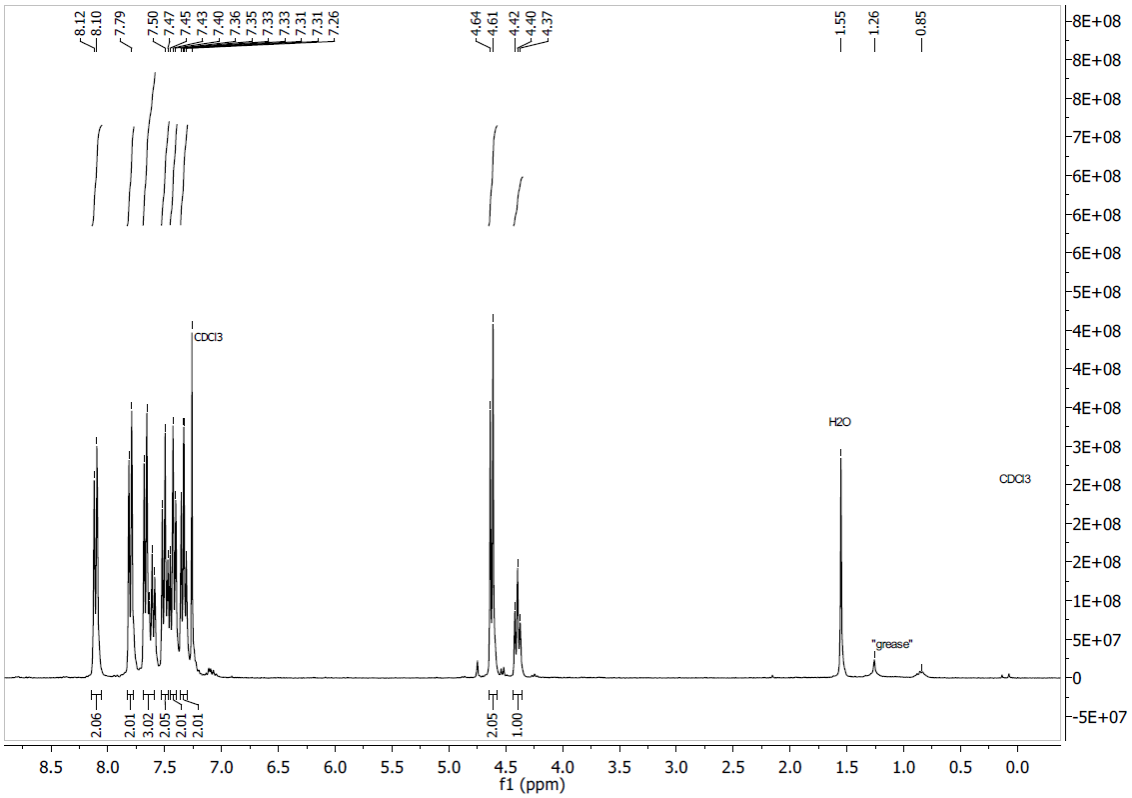


## 2. (9*H*-Fluoren-9-yl)methyl phenylacetate (3).

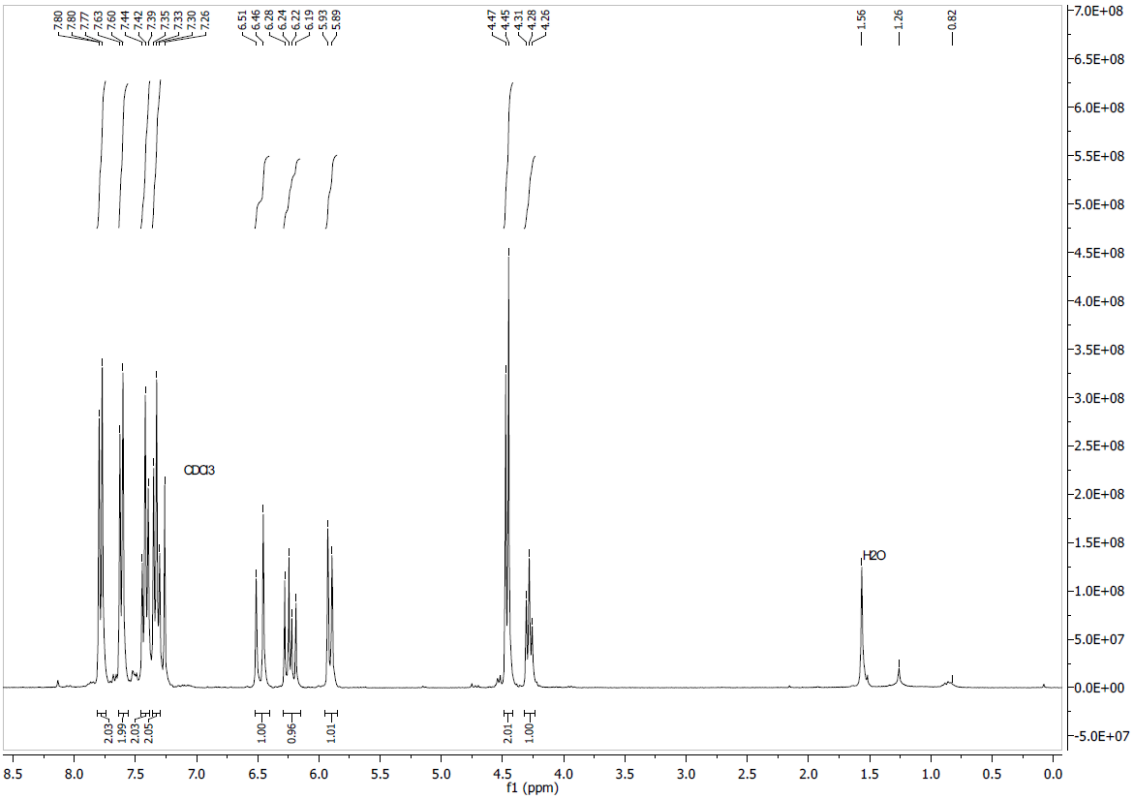


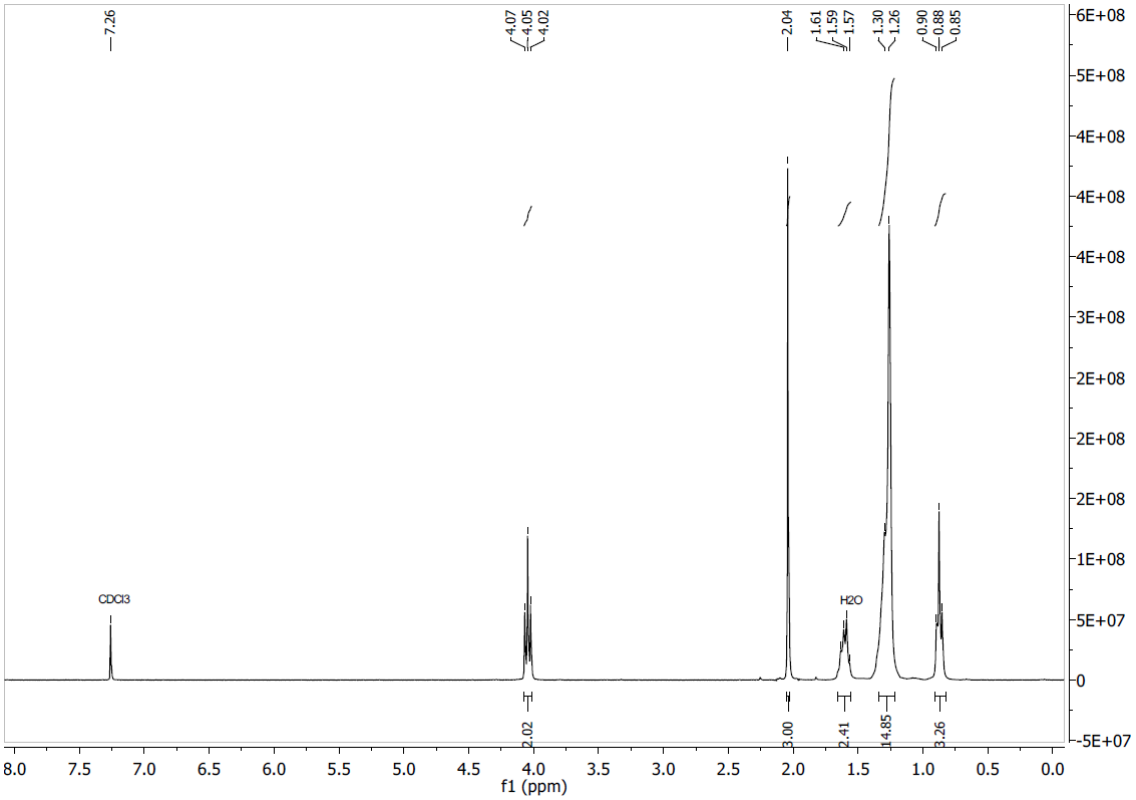
## 3. (9*H*-Fluoren-9-yl)methyl benzoate (4).



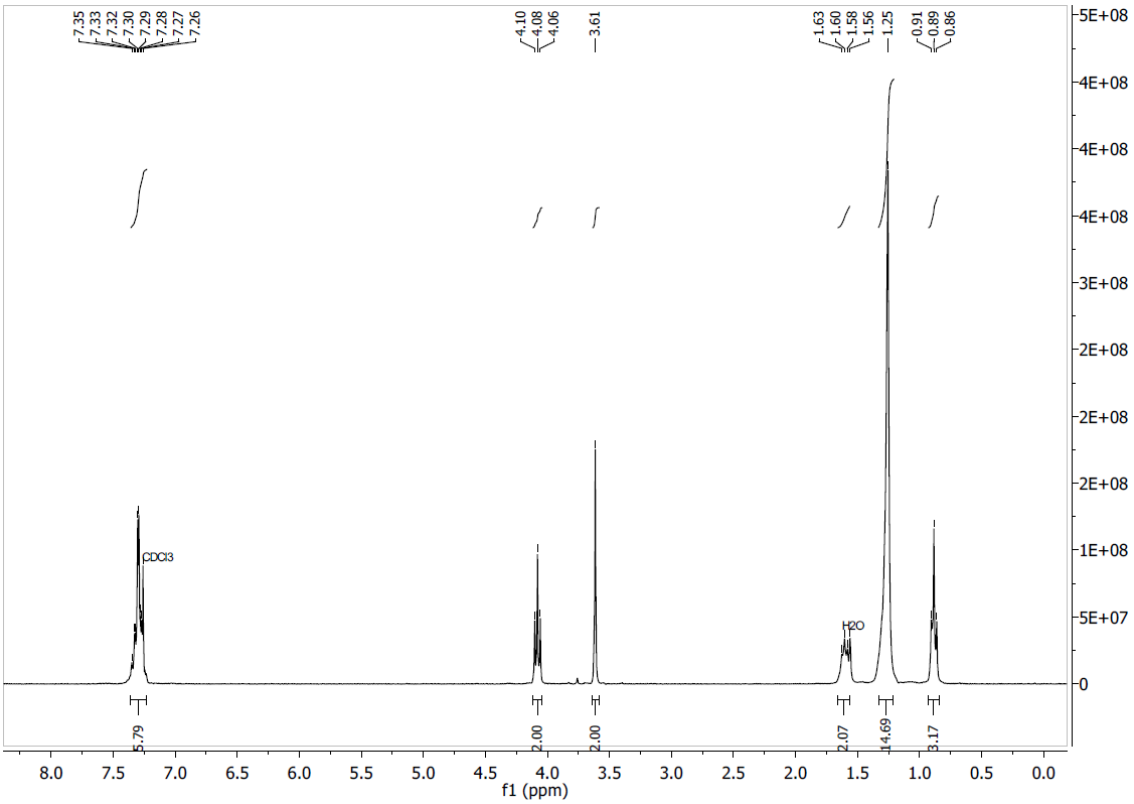

## 4. (9*H*-Fluoren-9-yl)methyl acrylate (5).



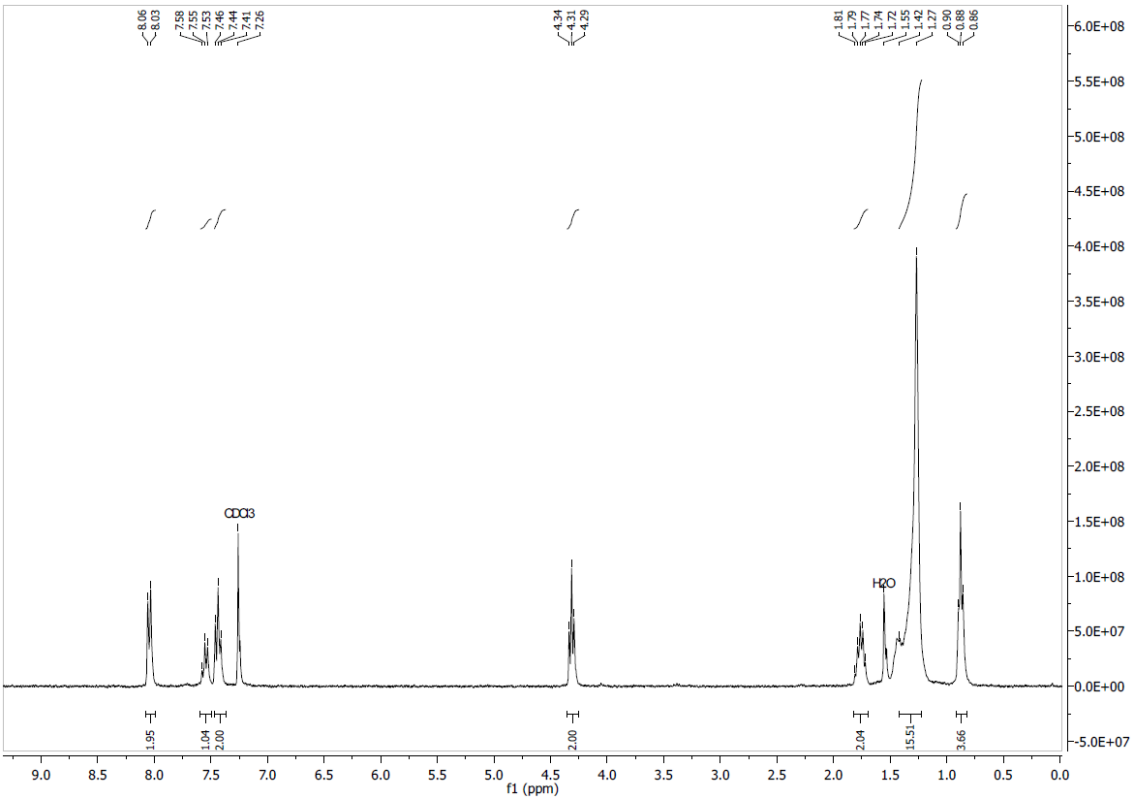
## 5. 1-Decyl acetate (7).



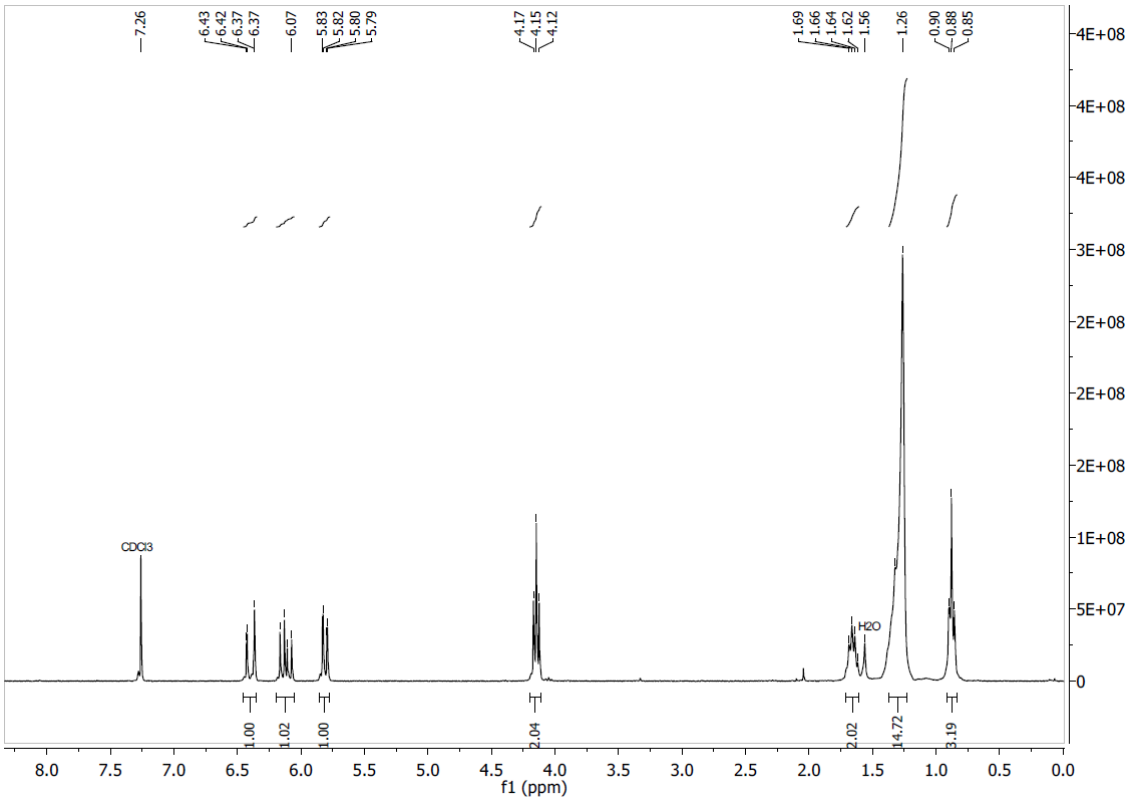

## 6. 1-Decyl phenylacetate (8).



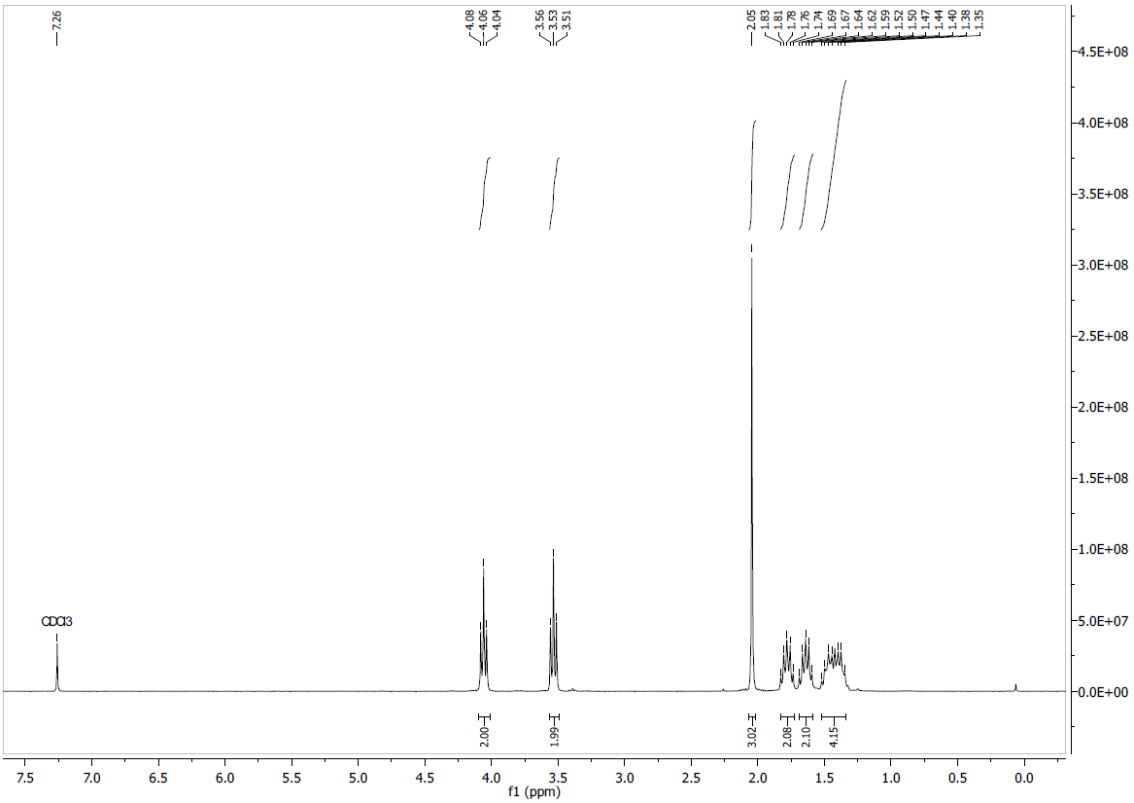
## 7. 1-Decyl benzoate (9).



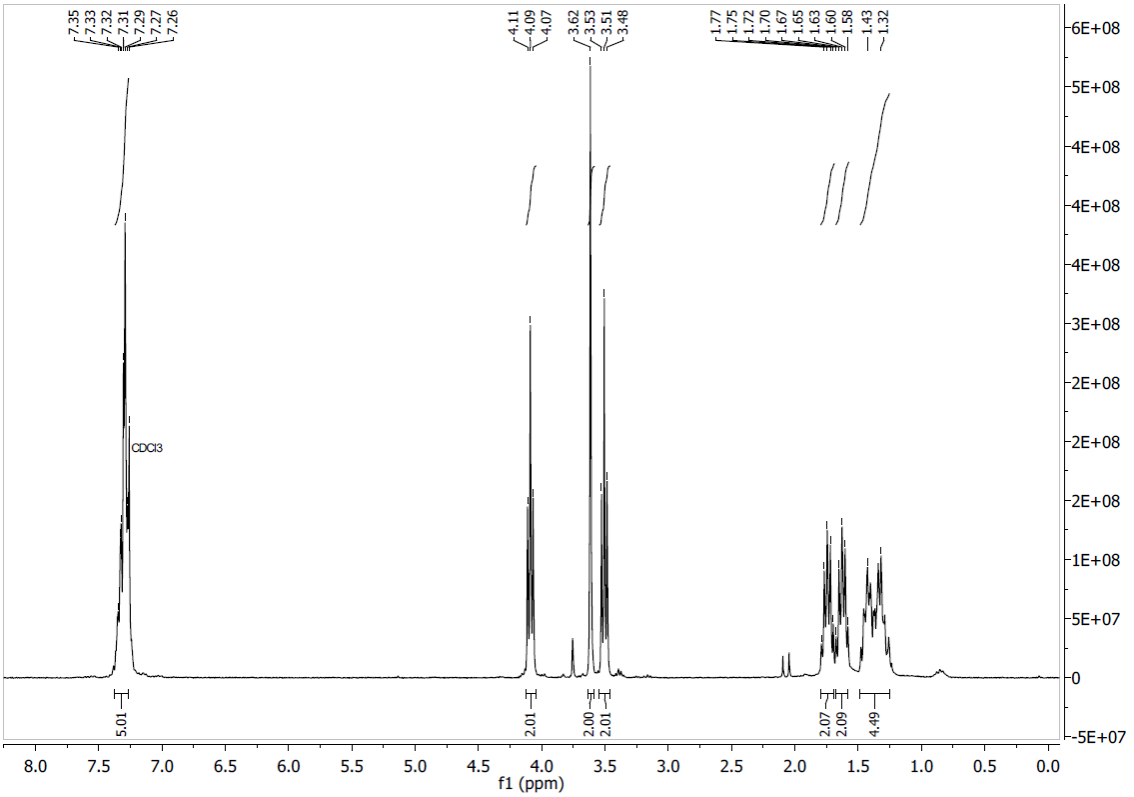

## 8. 1-Decyl acrylate (10).



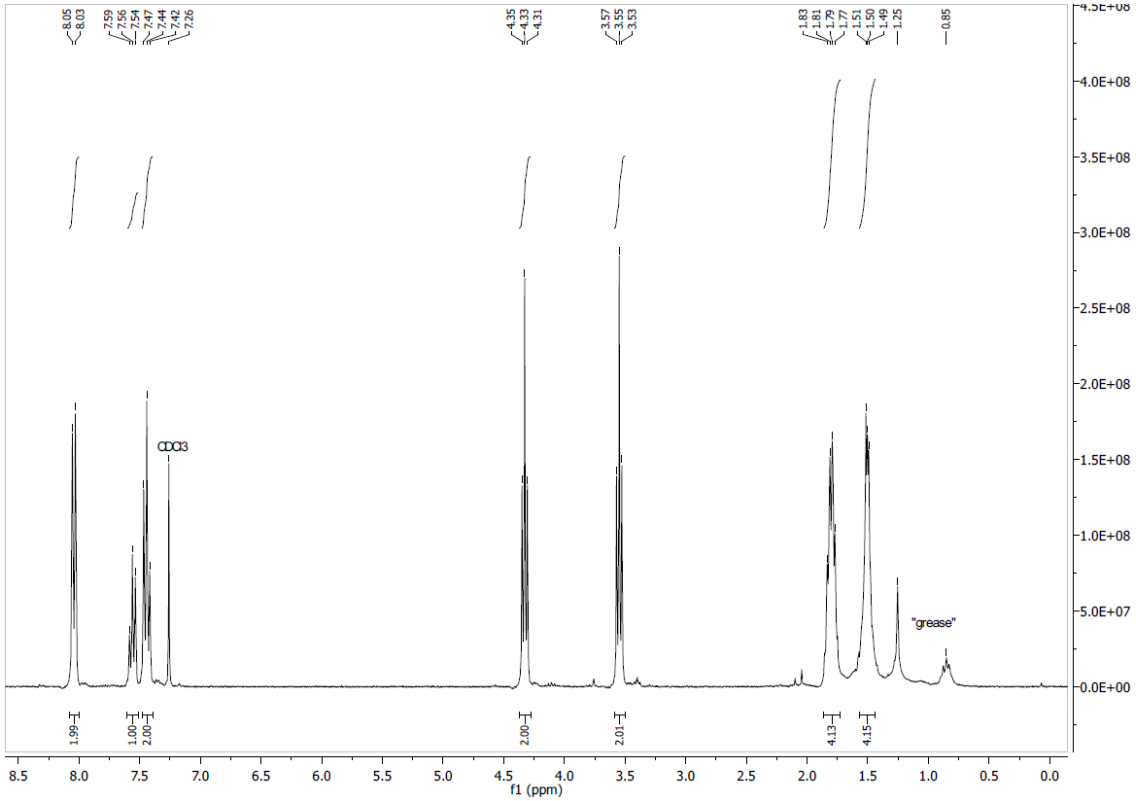
## 9. 6-Chlorohexyl Acetate (12).



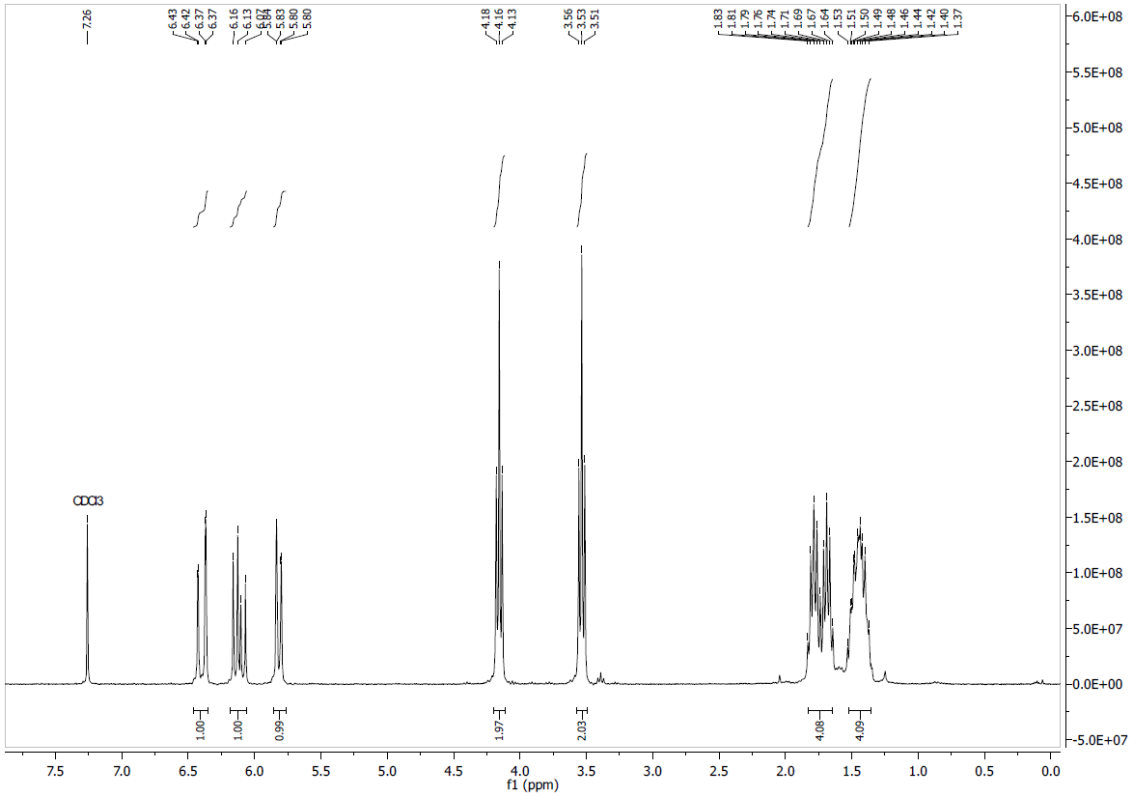

## 10. 6-Chlorohexyl phenylacetate (13).



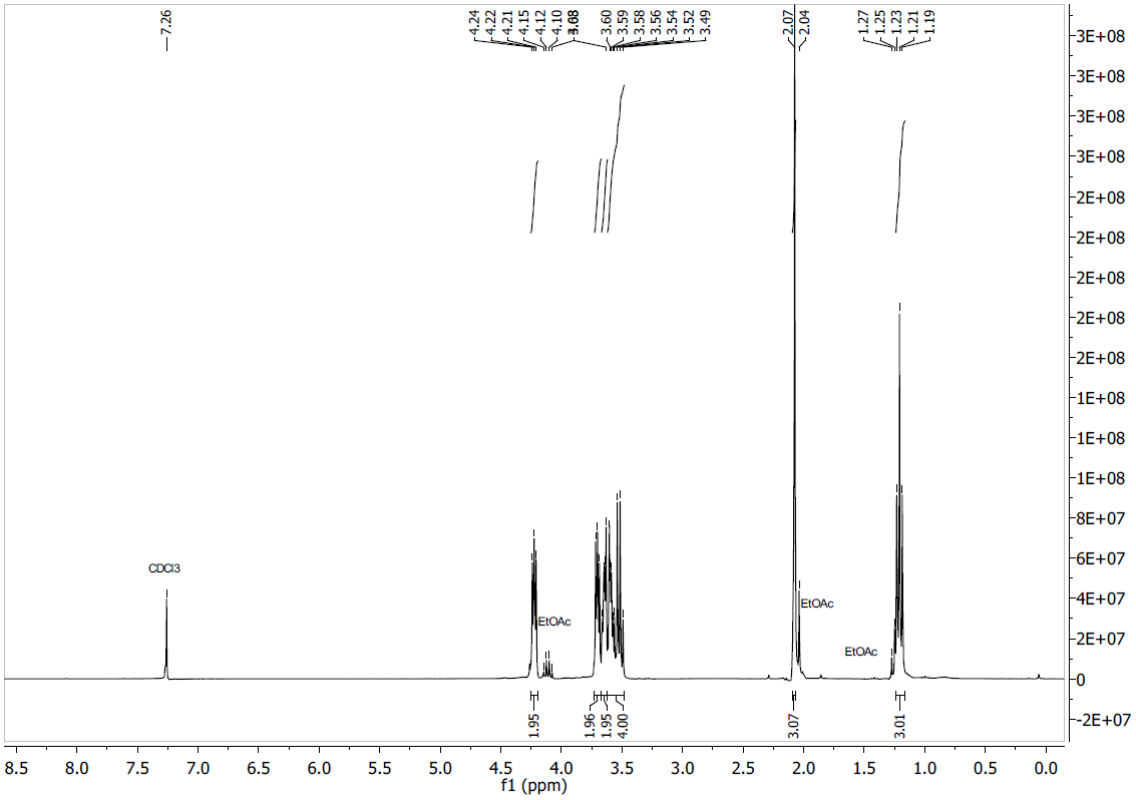
## 11. 6-Chlorohexyl benzoate (14).



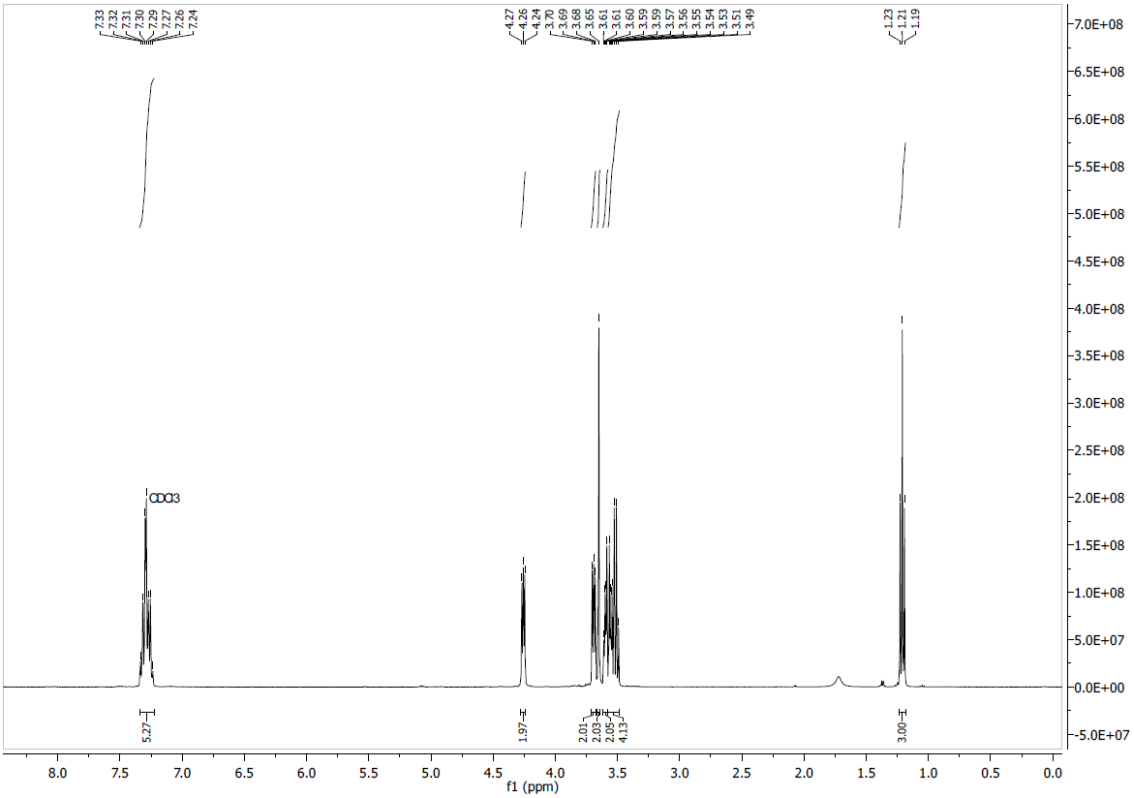

## 12. 6-Chlorohexyl acrylate (15).

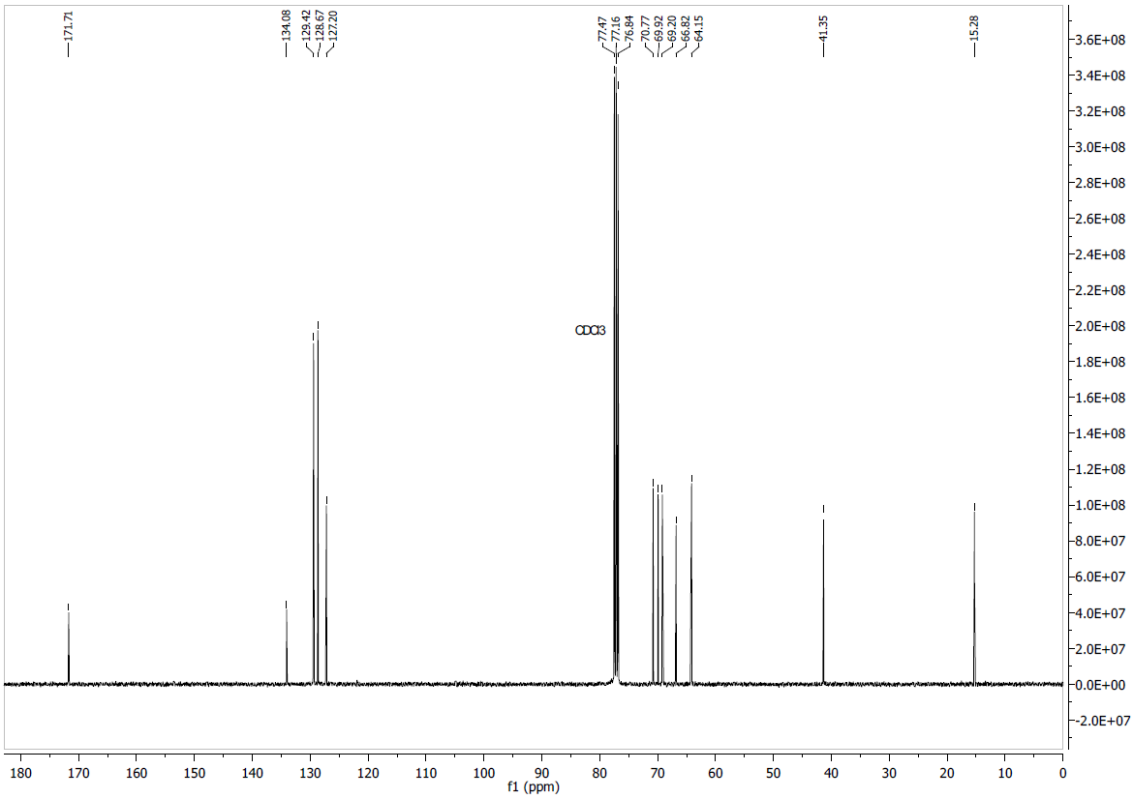


## 13. 2-(2-Ethoxyethoxy)ethyl acetate (17).

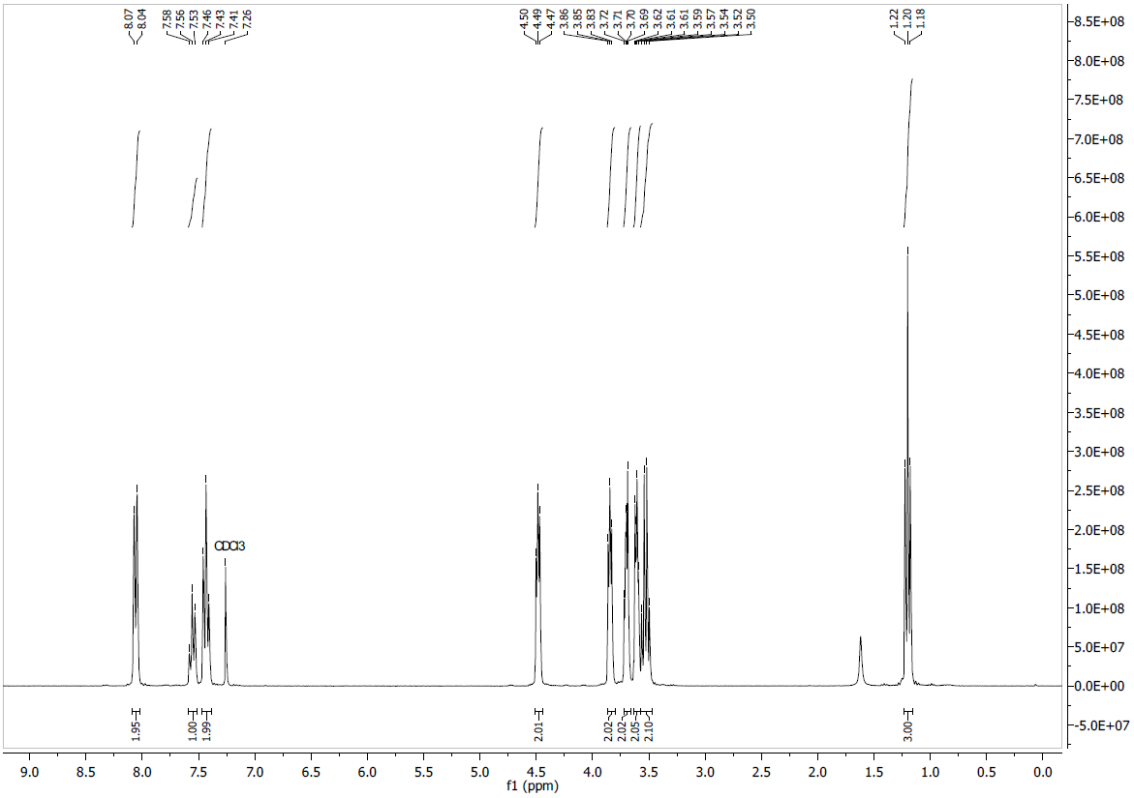


## 14. 2-(2-Ethoxyethoxy)ethyl phenylacetate (18).

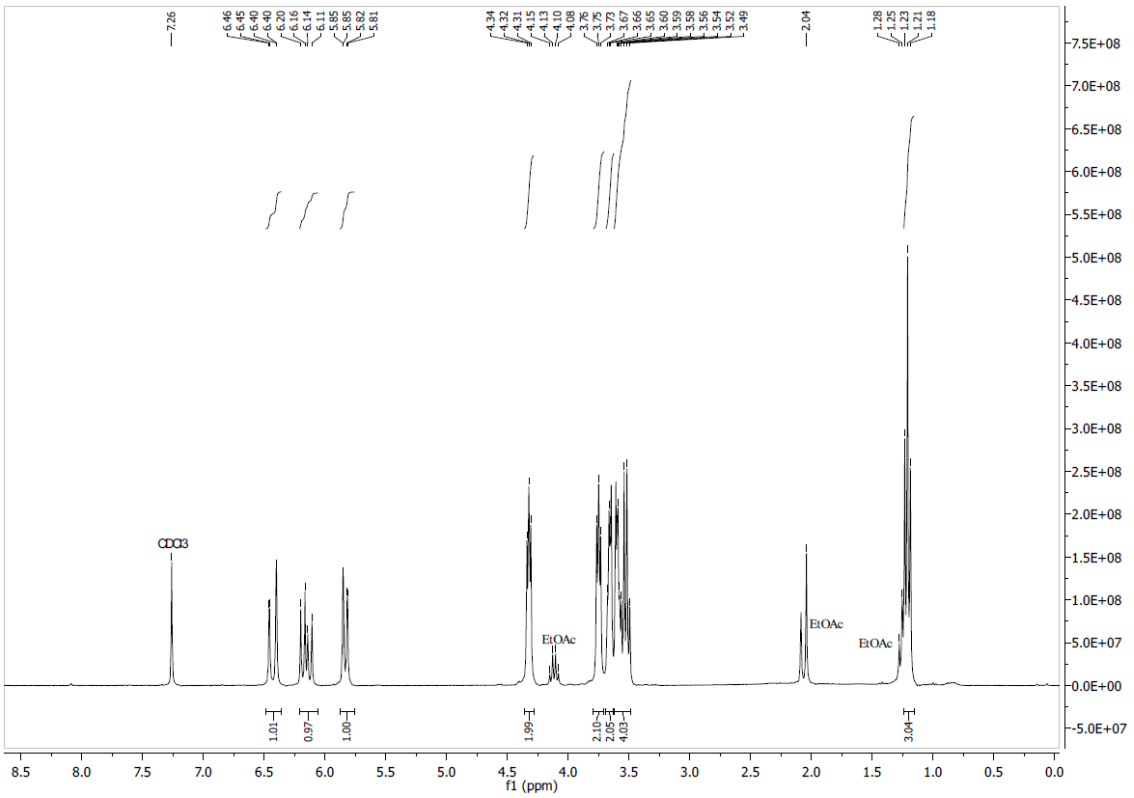


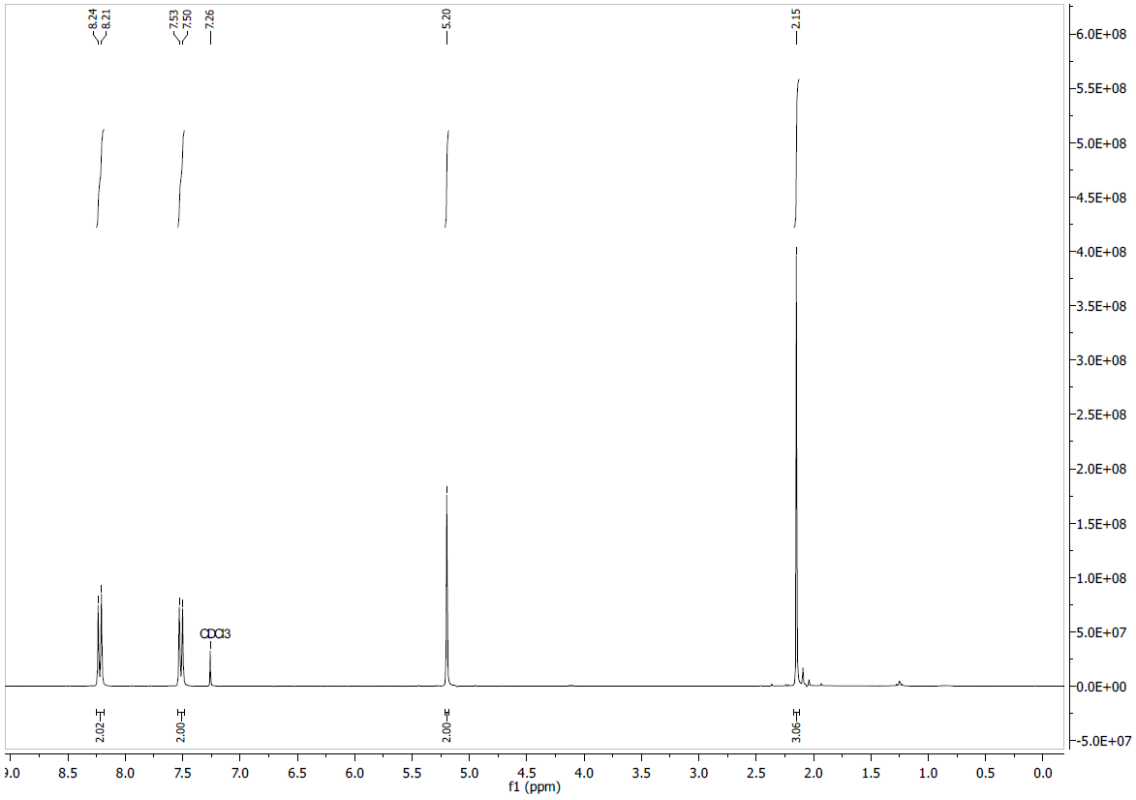
## 15. 2-(2-Ethoxyethoxy)ethyl benzoate (19).



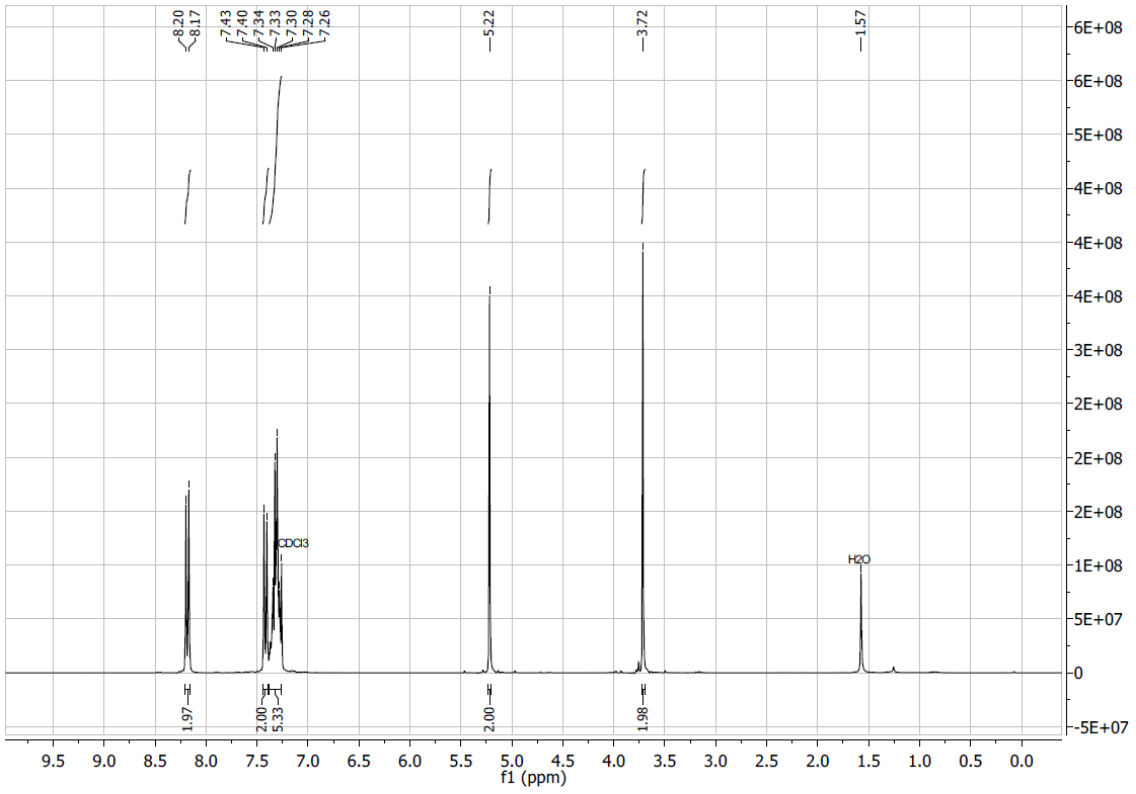

## 16. 2-(2-Ethoxyethoxy)ethyl acrylate (20).



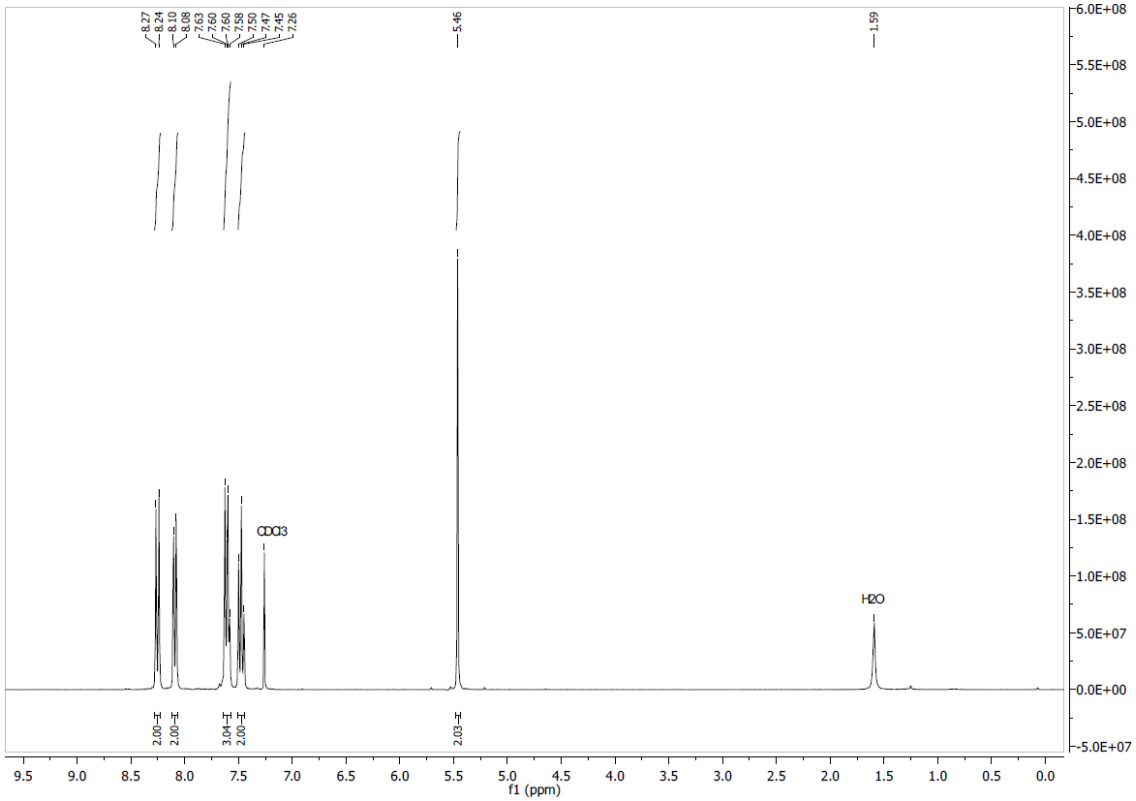
**17. 4-Nitrobenzyl acetate** (**22**).



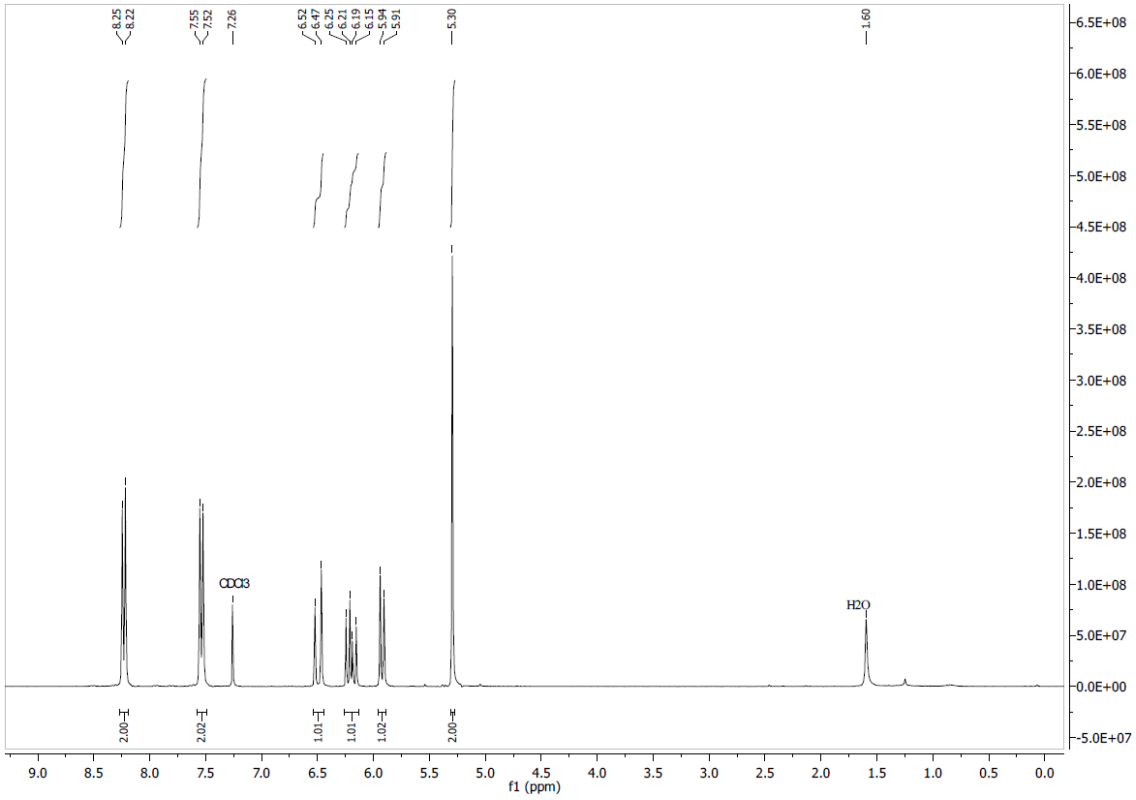

**18. 4-Nitrobenzyl phenylacetate (23).**



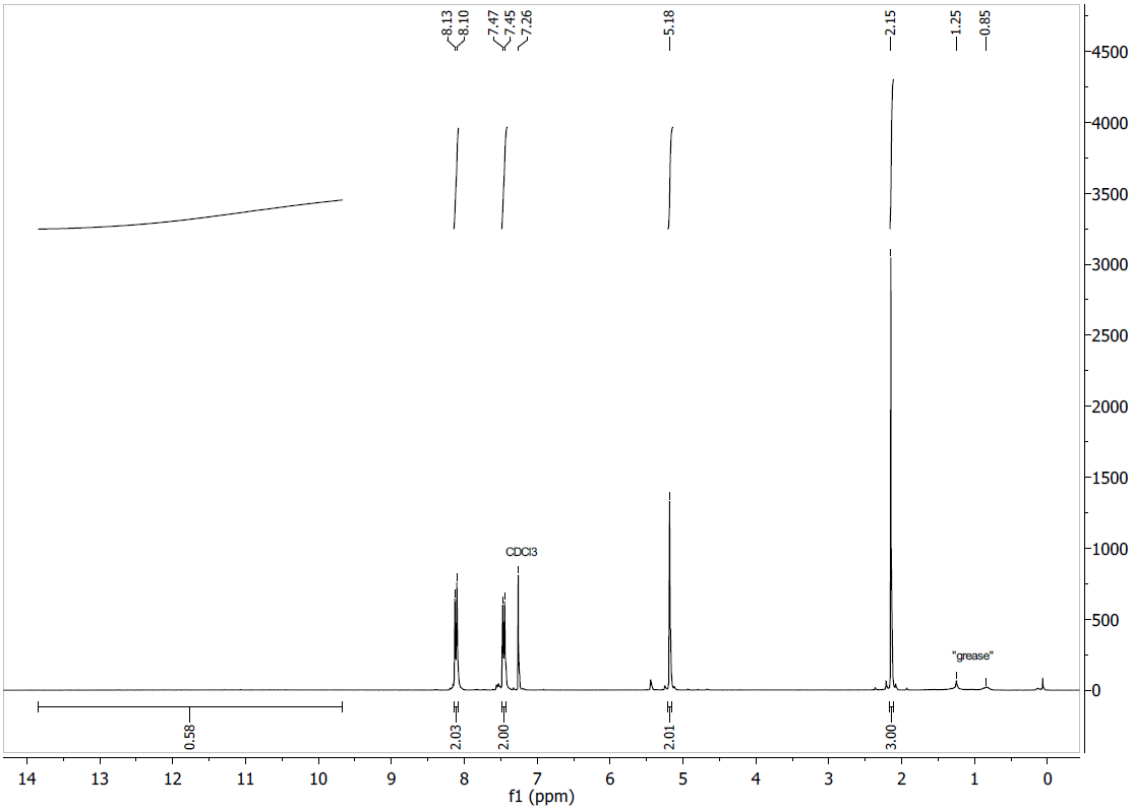
## 19. 4-Nitrobenzyl benzoate (24).



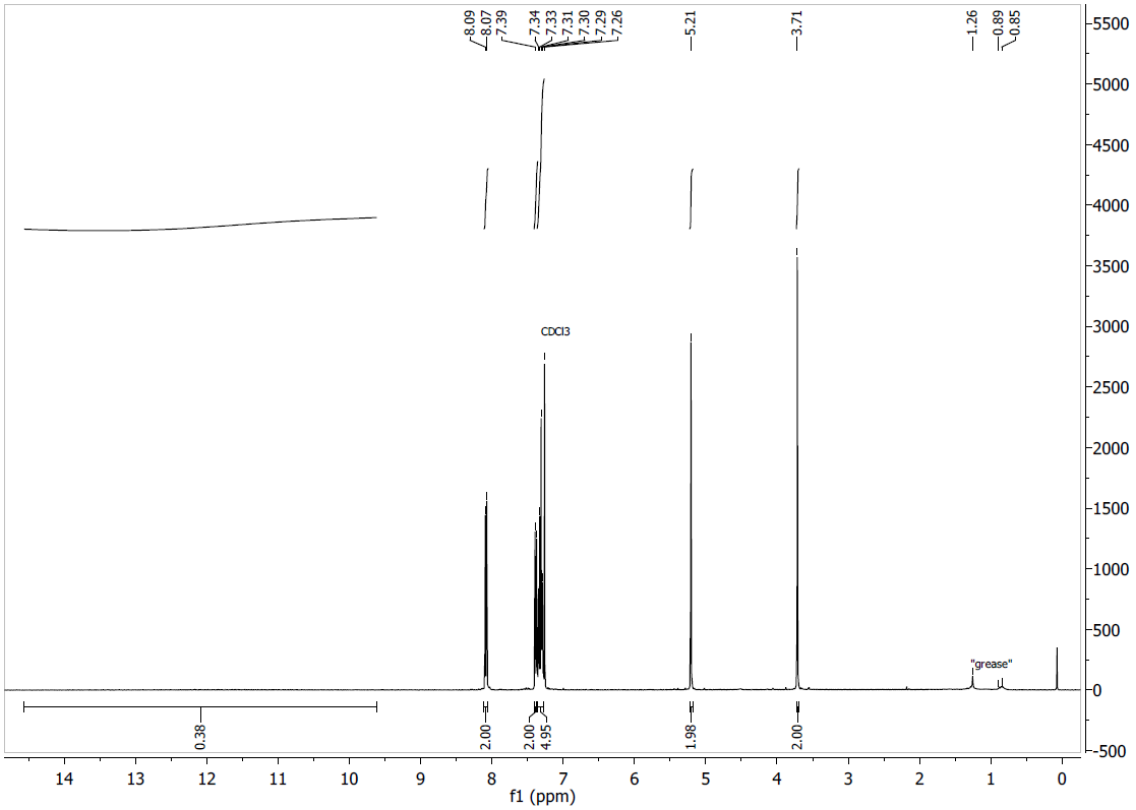

## 20. 4-Nitrobenzyl acrylate (25).

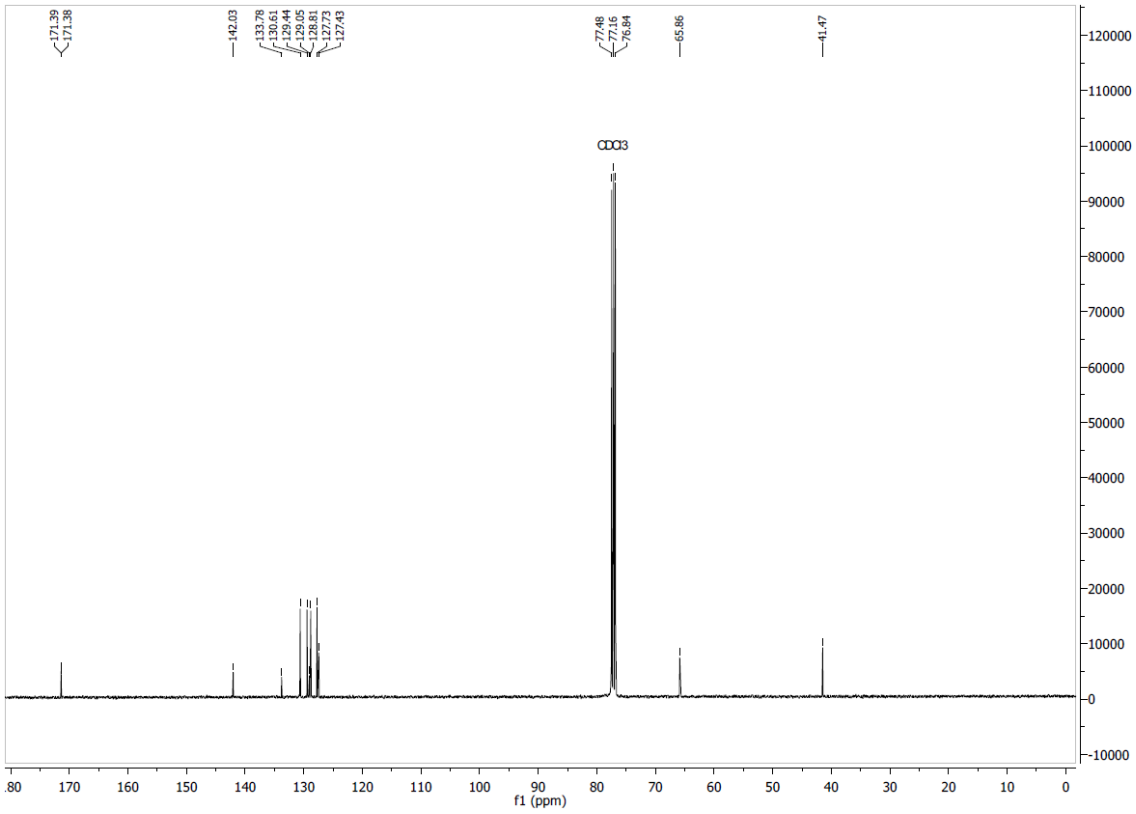


## 21. 4-(Acetoxymethyl)benzoic acid (27).

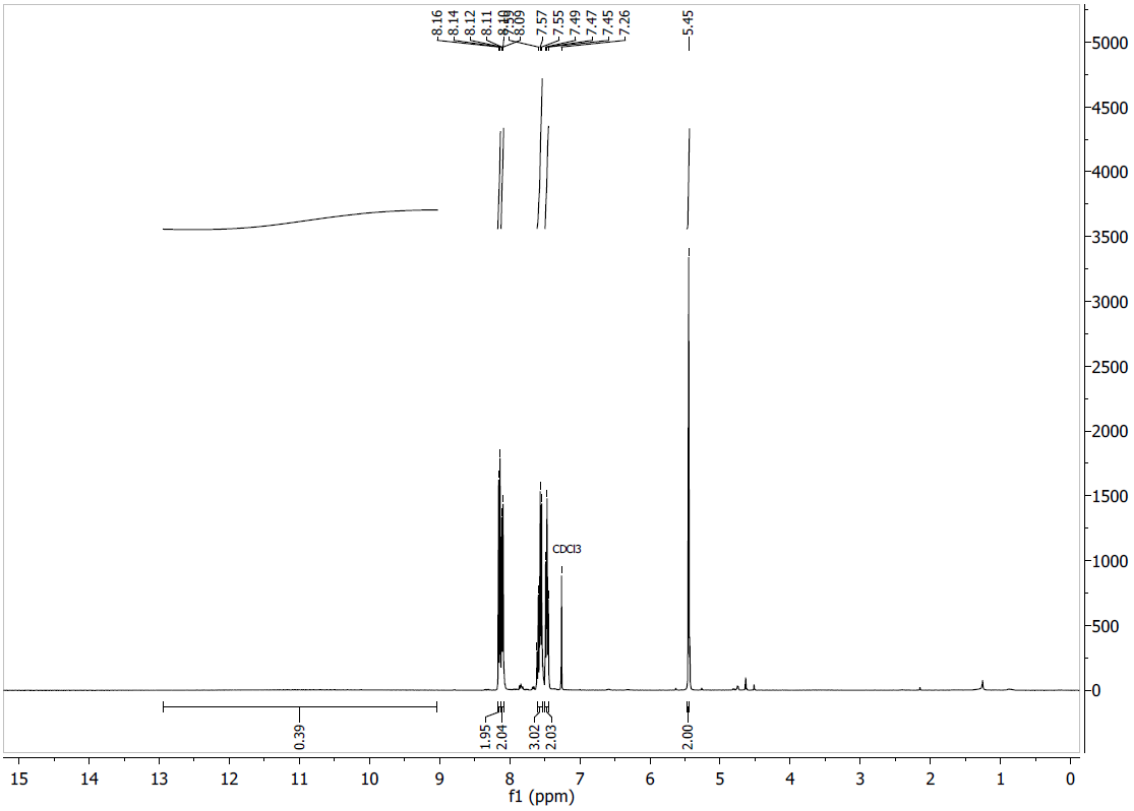


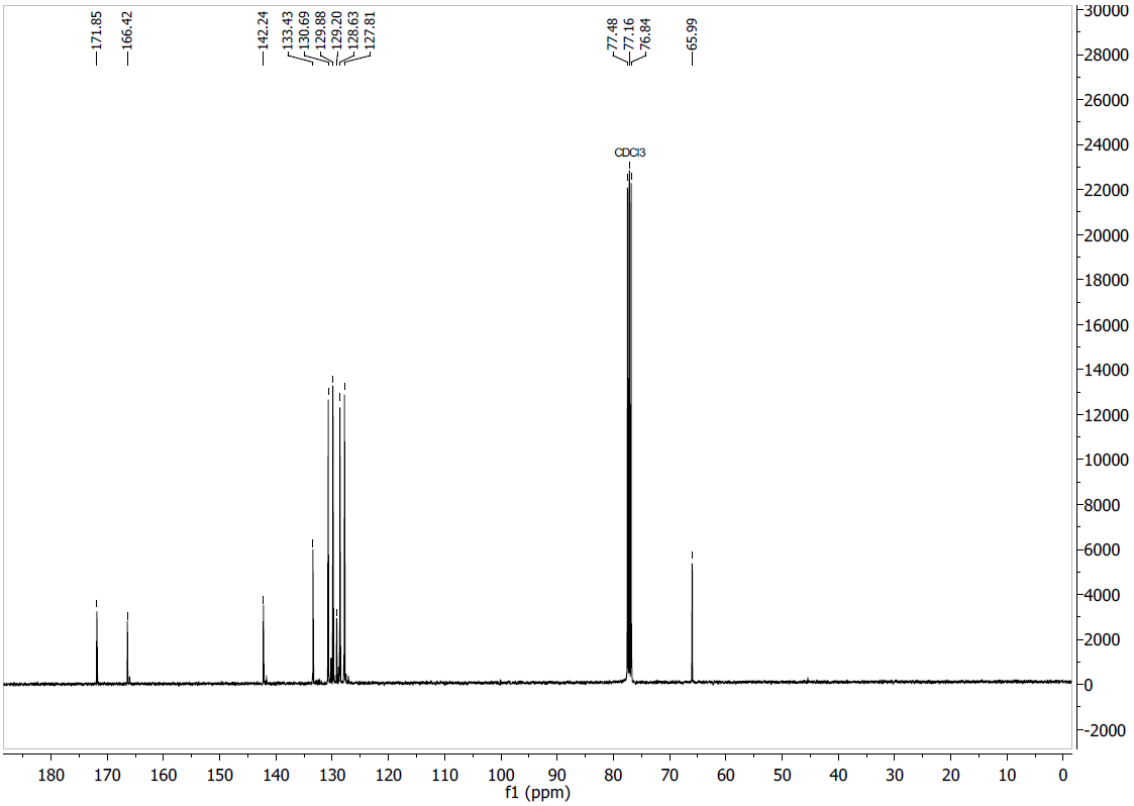
## 22. 4-[(Phenylacetoxy)methyl]benzoic acid (28).



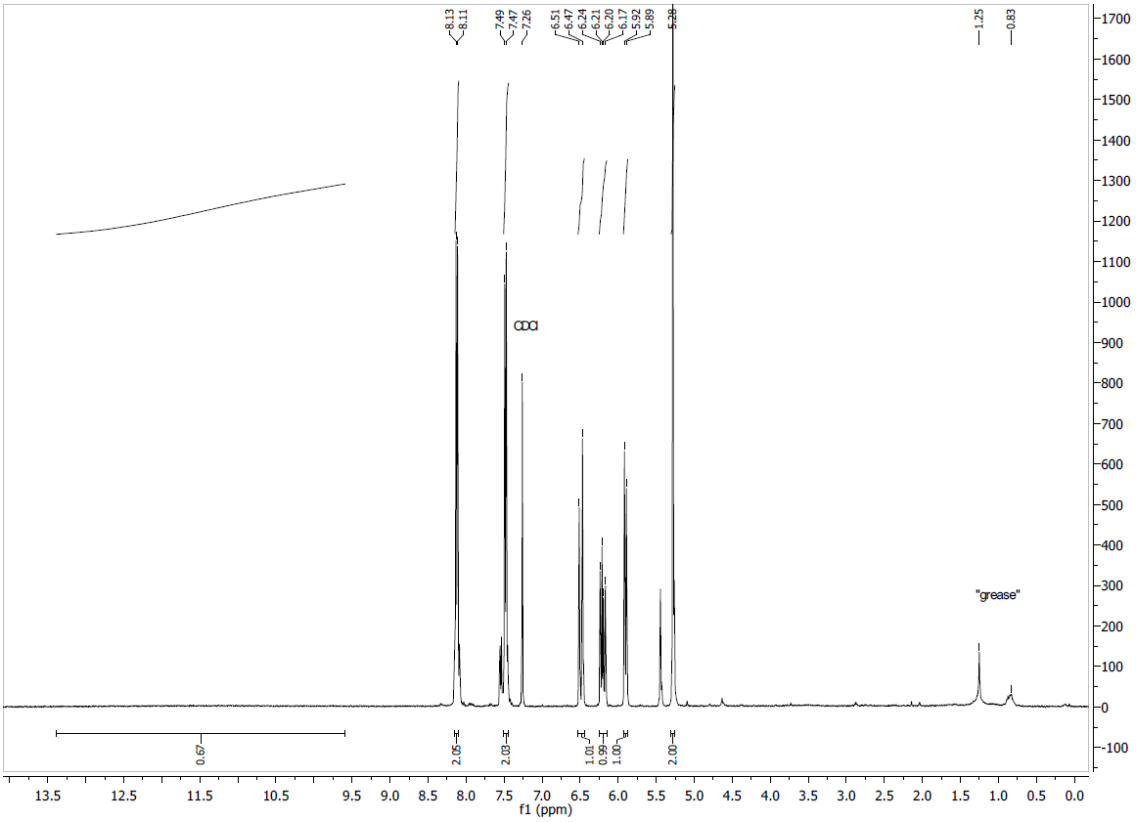



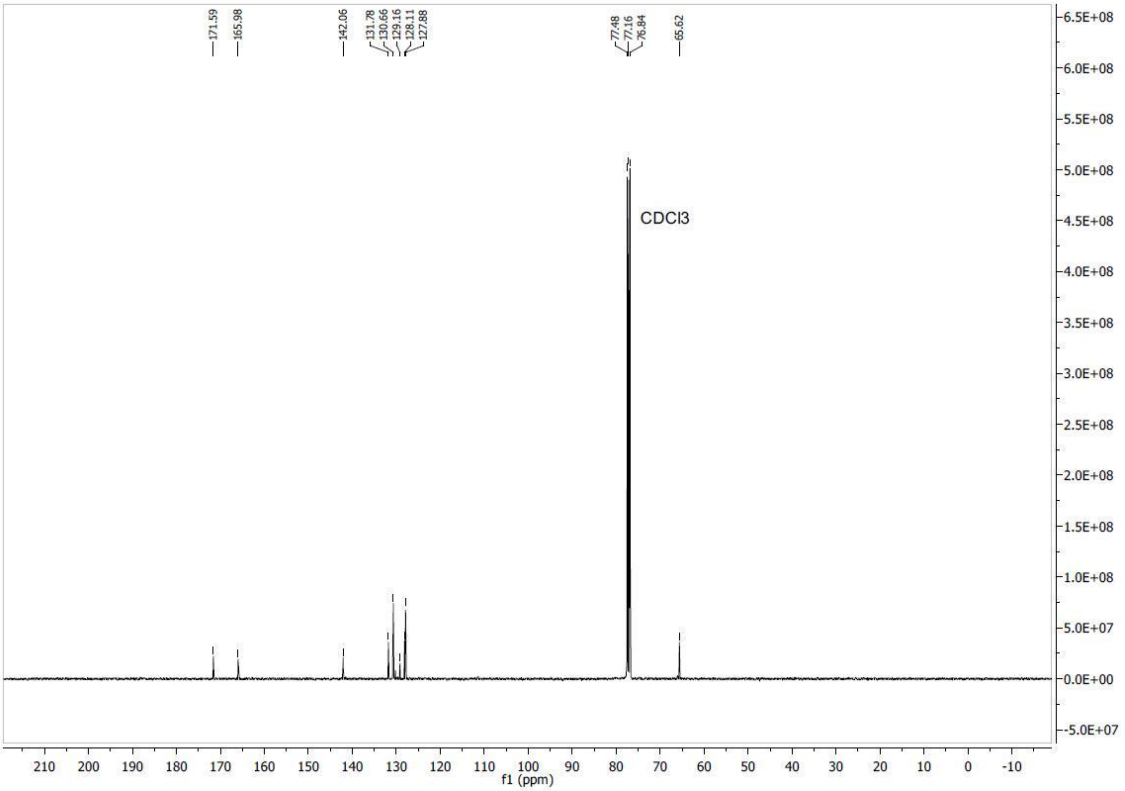
## 23. 4-(Benzoyloxymethyl)benzoic acid (29).



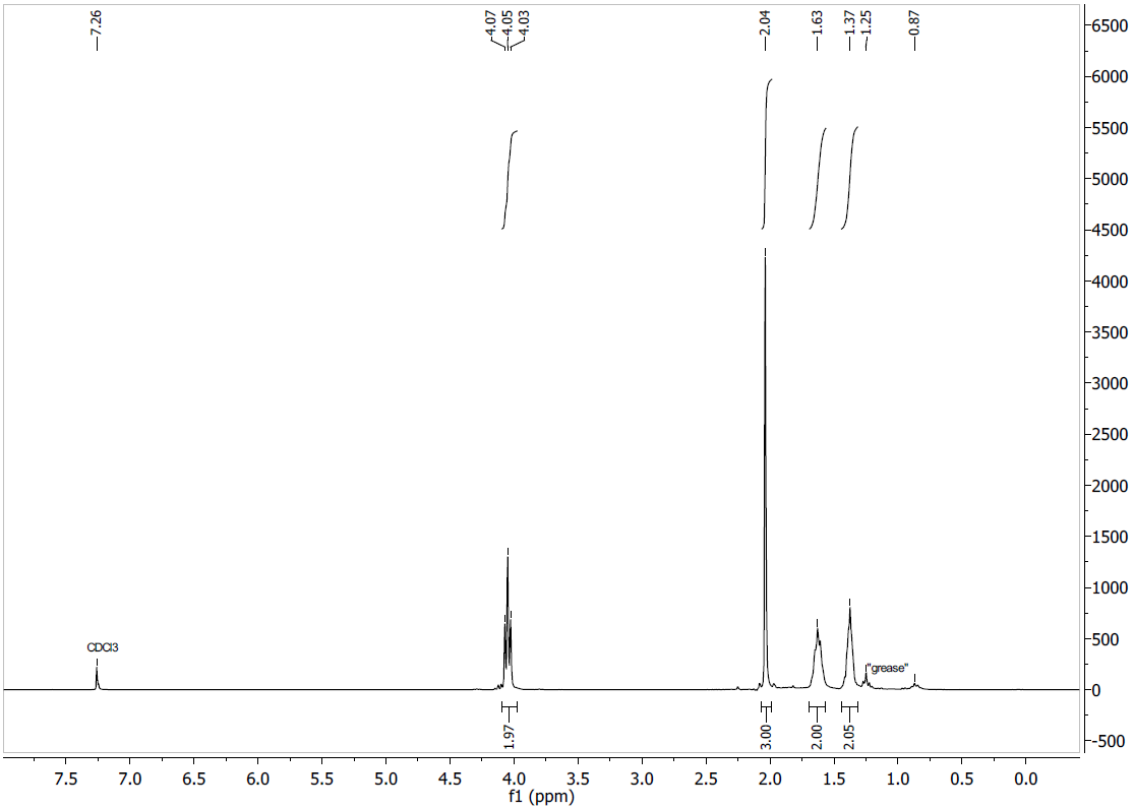



## 24. 4-(Acryloyloxymethyl)benzoic acid (30).

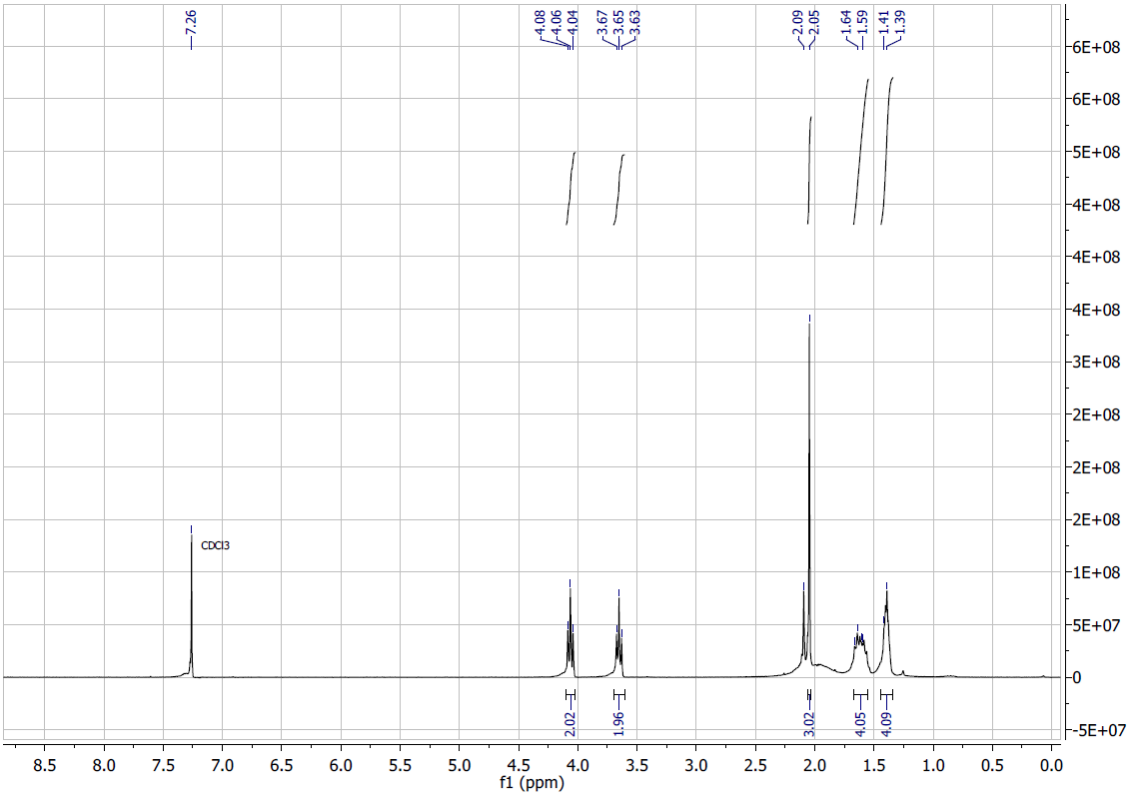


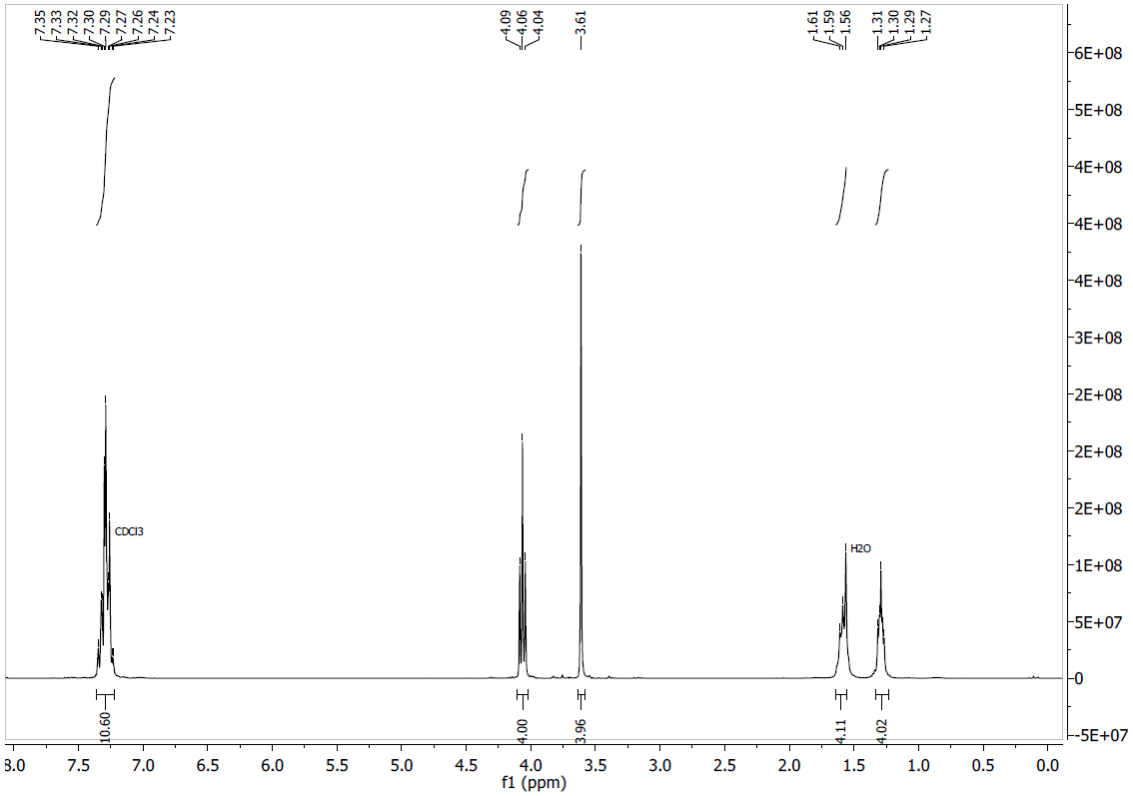
## 25. Hexane-1,6-diyl diacetate (32).



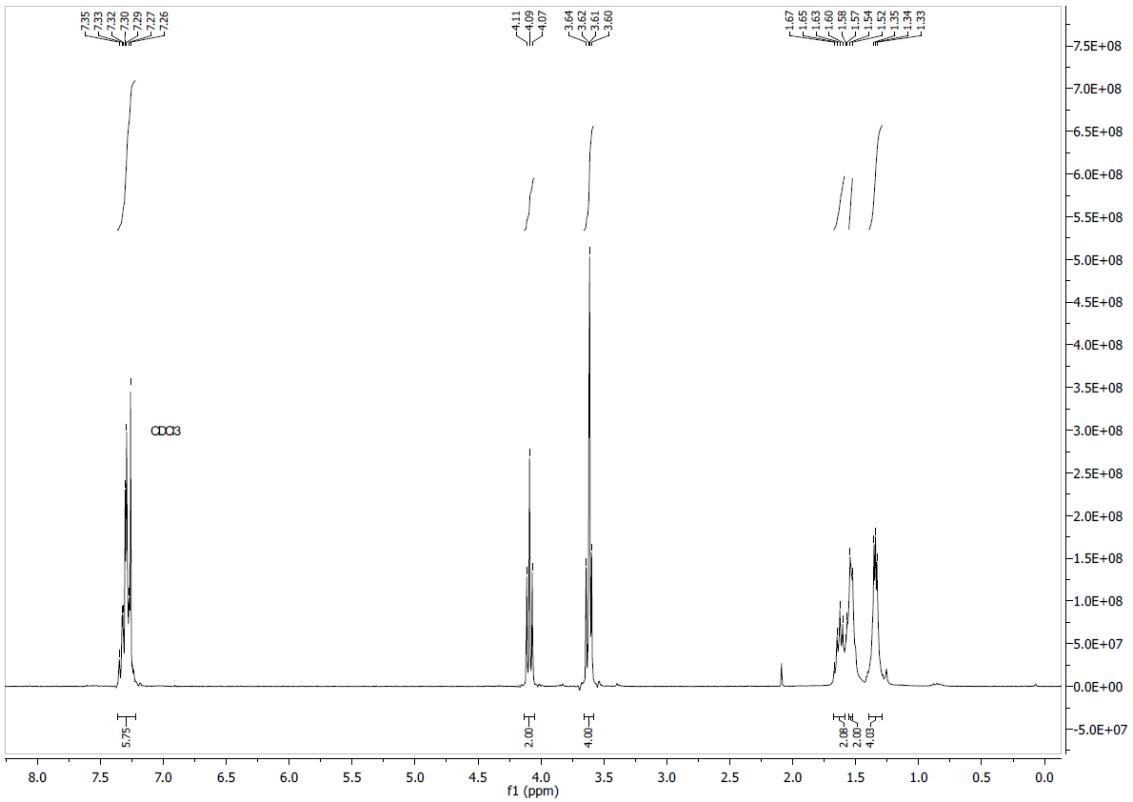

## 26. 6-Hydroxyhexyl acetate (33).



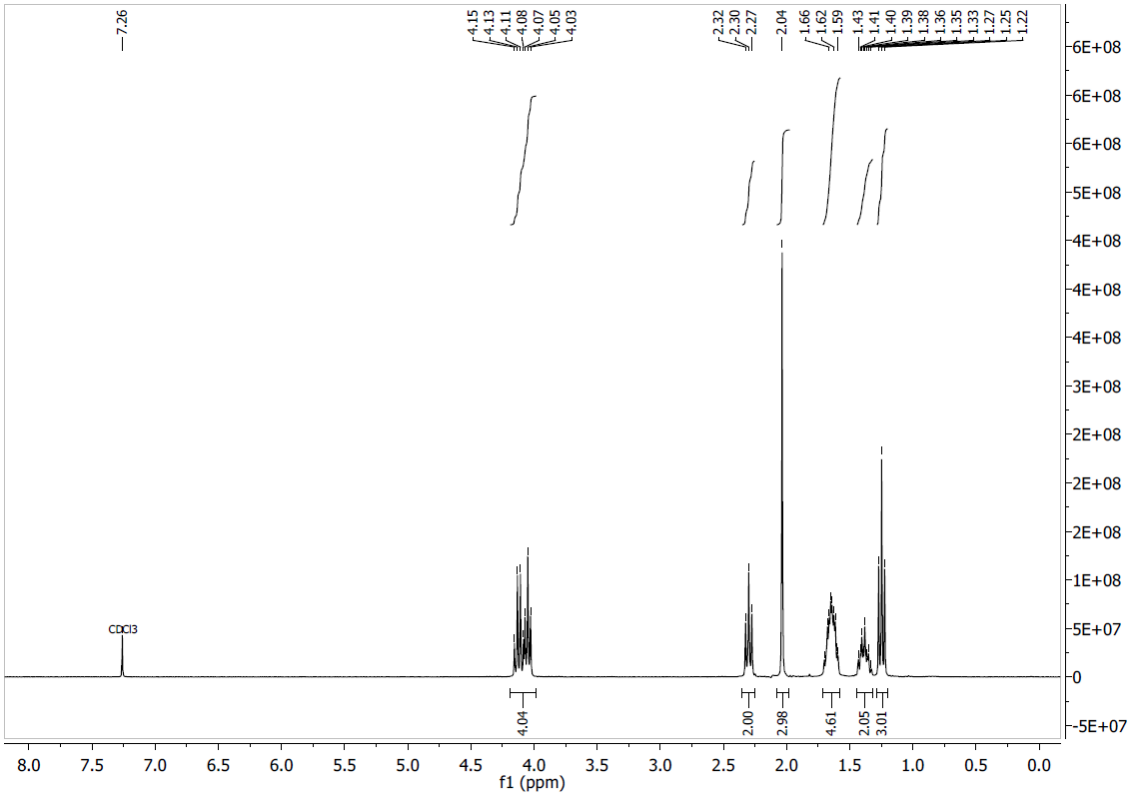
## 27. Hexane-1,6-diyl bis(phenylacetate) (34).



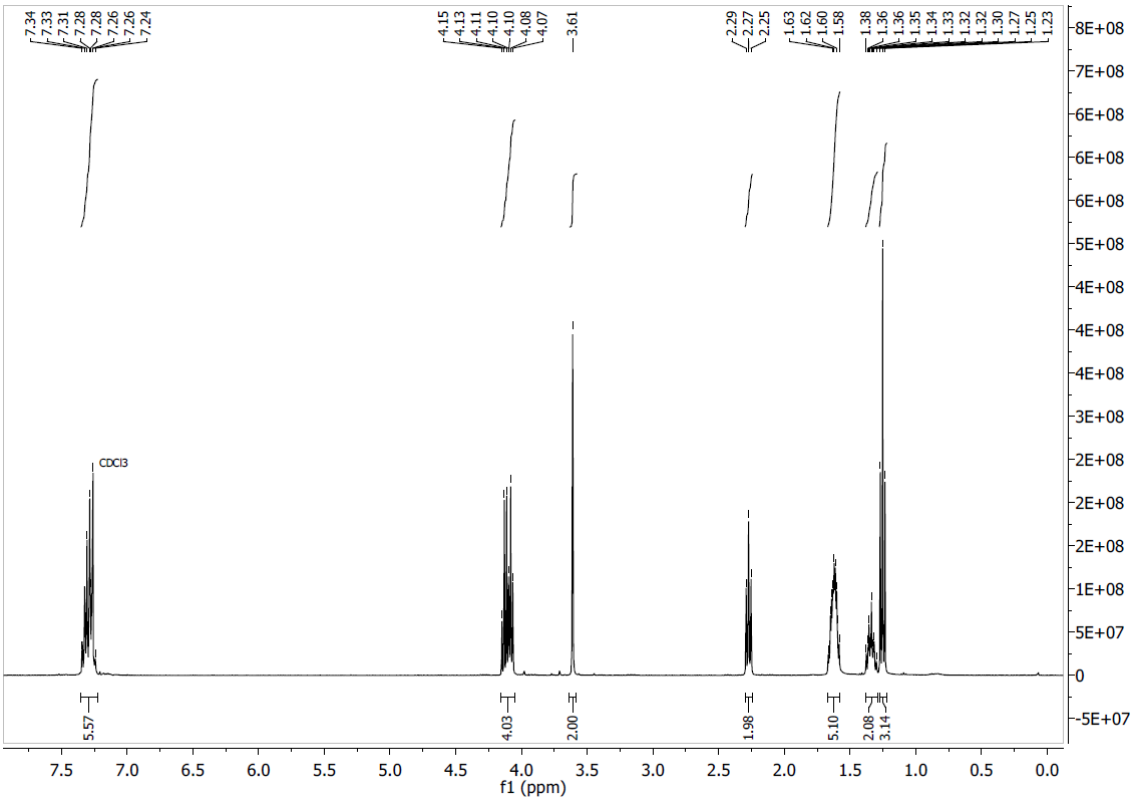

## 28. 6-Hydroxyhexyl phenylacetate (35).

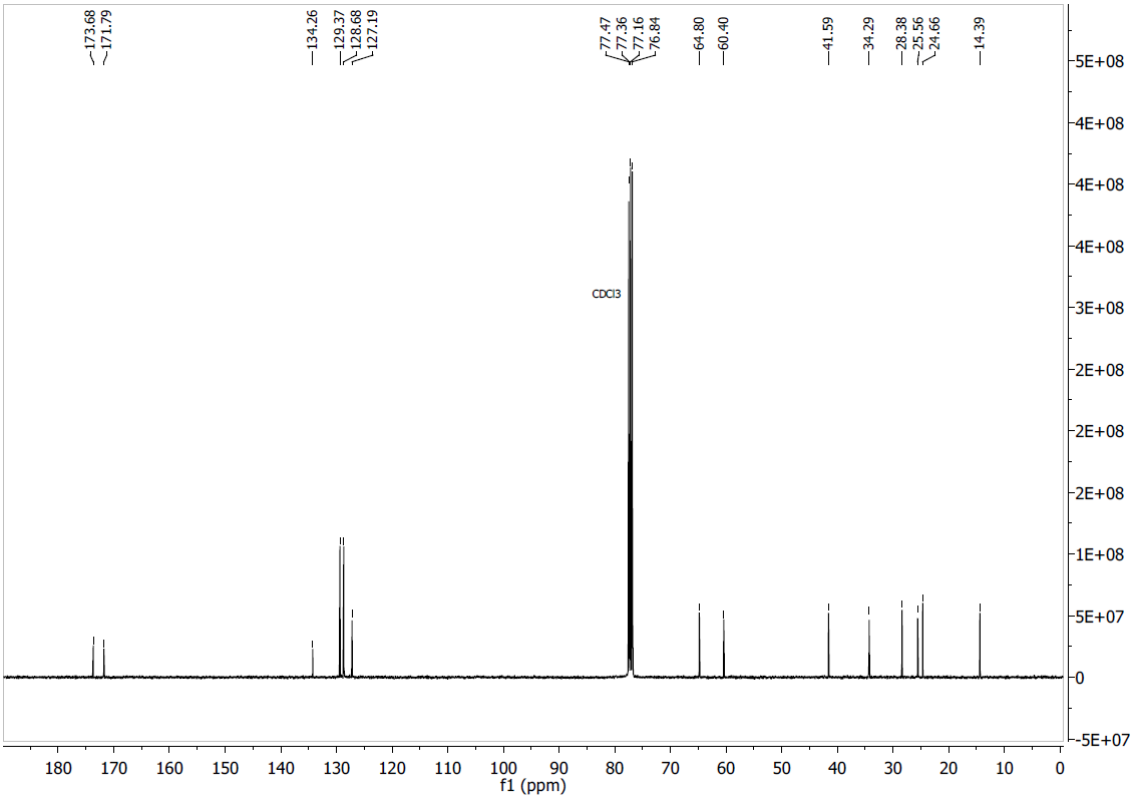


## 29. Ethyl 6-acetoxyhexanoate (37).

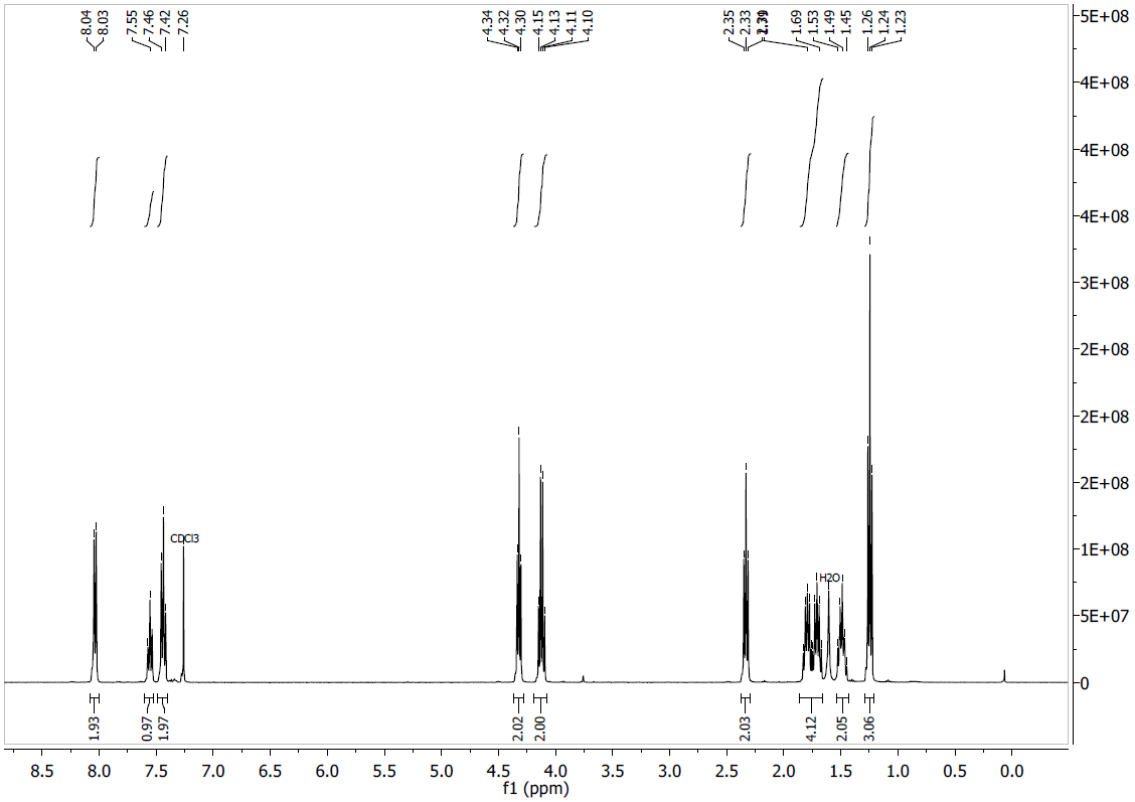


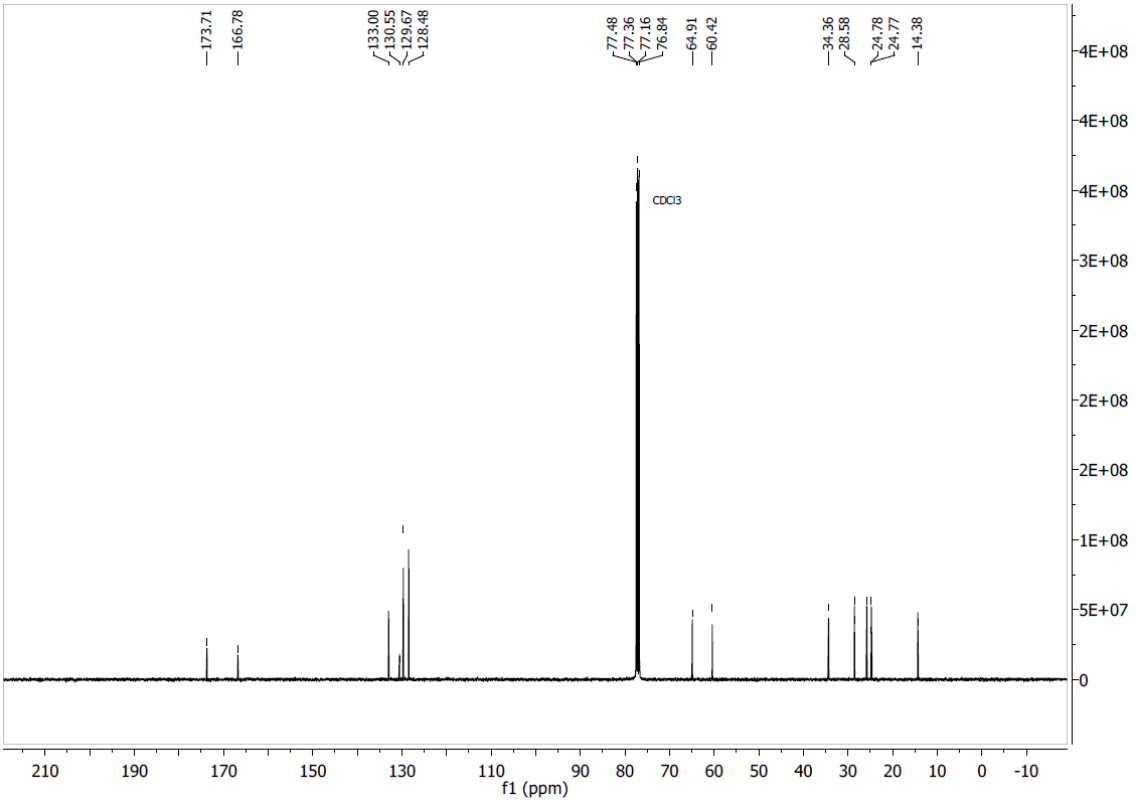
## 30. Ethyl 6-(phenylacetoxy)hexanoate (38).



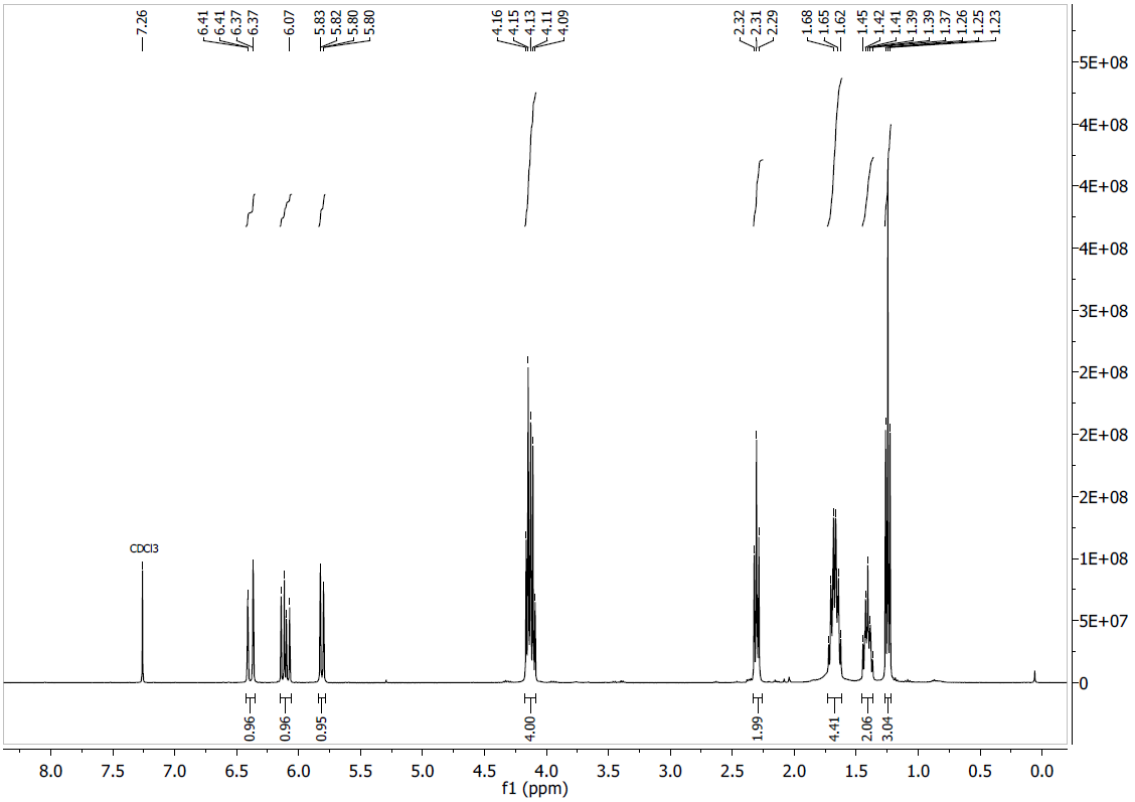



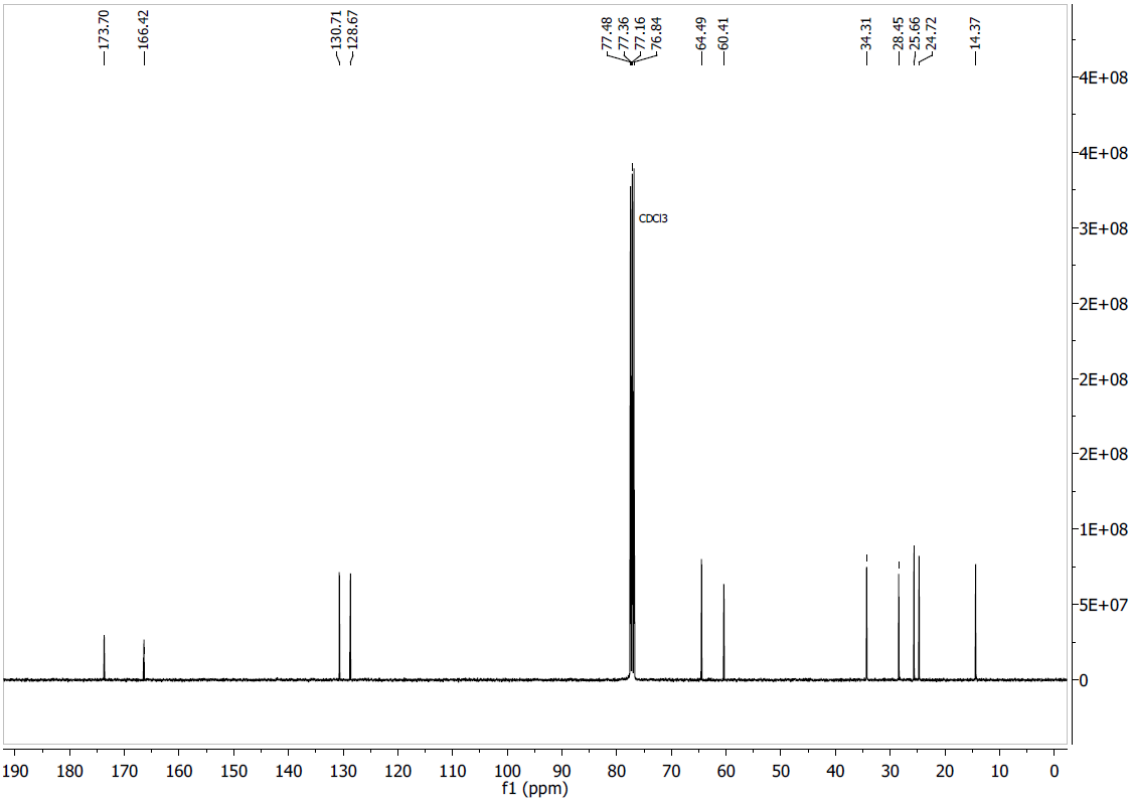
## 31. 6-Ethoxy-6-oxohexyl benzoate (39).



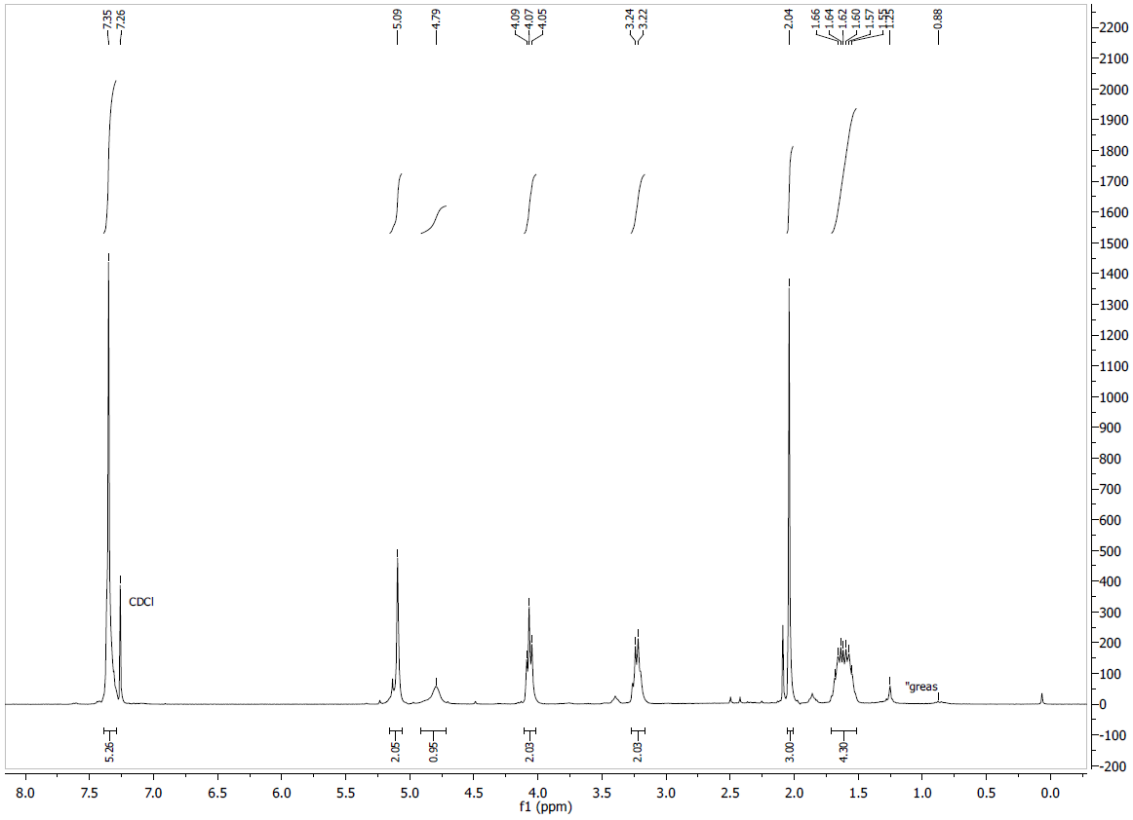



## 32. Ethyl 6-(acryloyloxy)hexanoate (40).

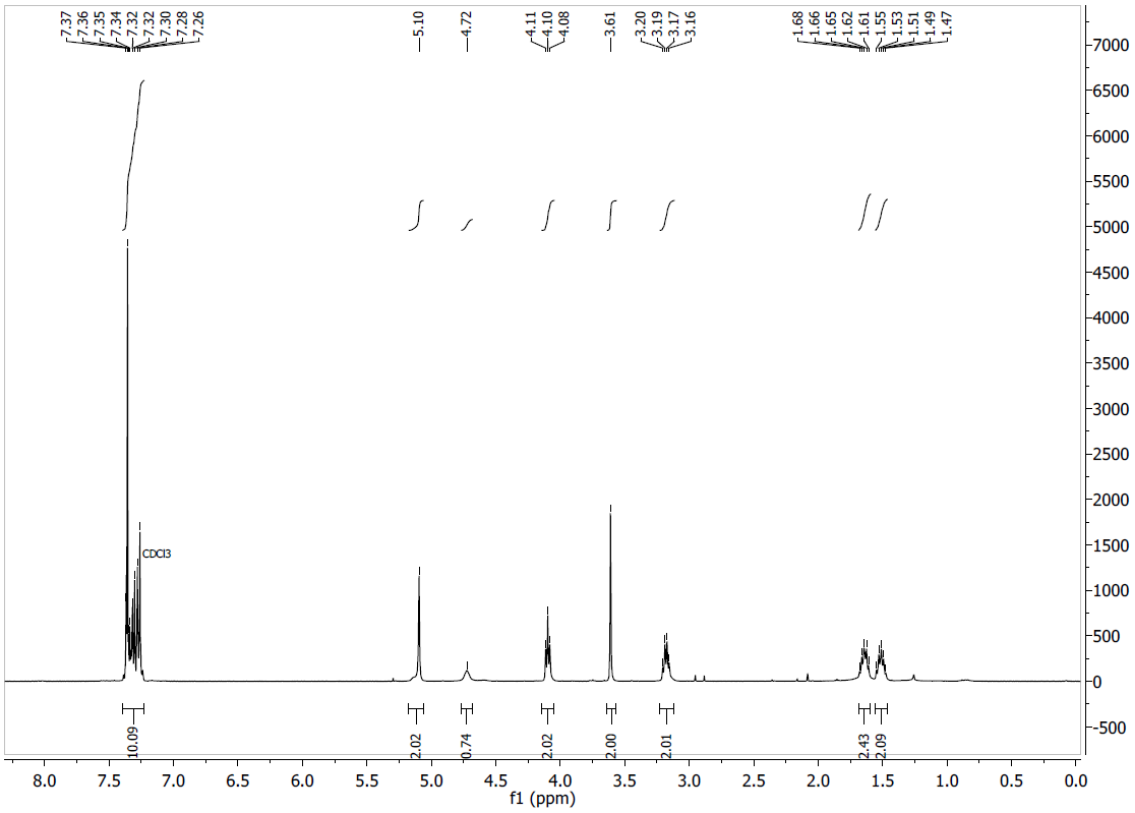


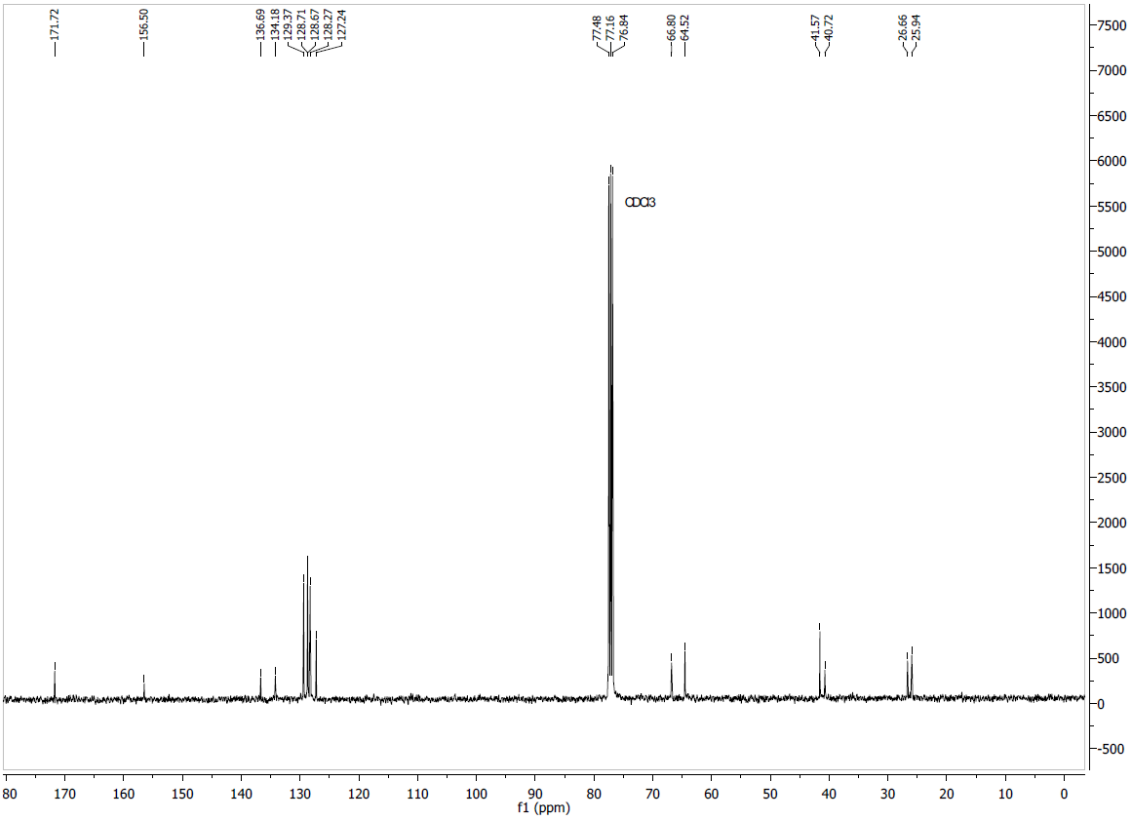



## 33. 4-(Benzyloxycarbonylamino)butyl acetate (42).

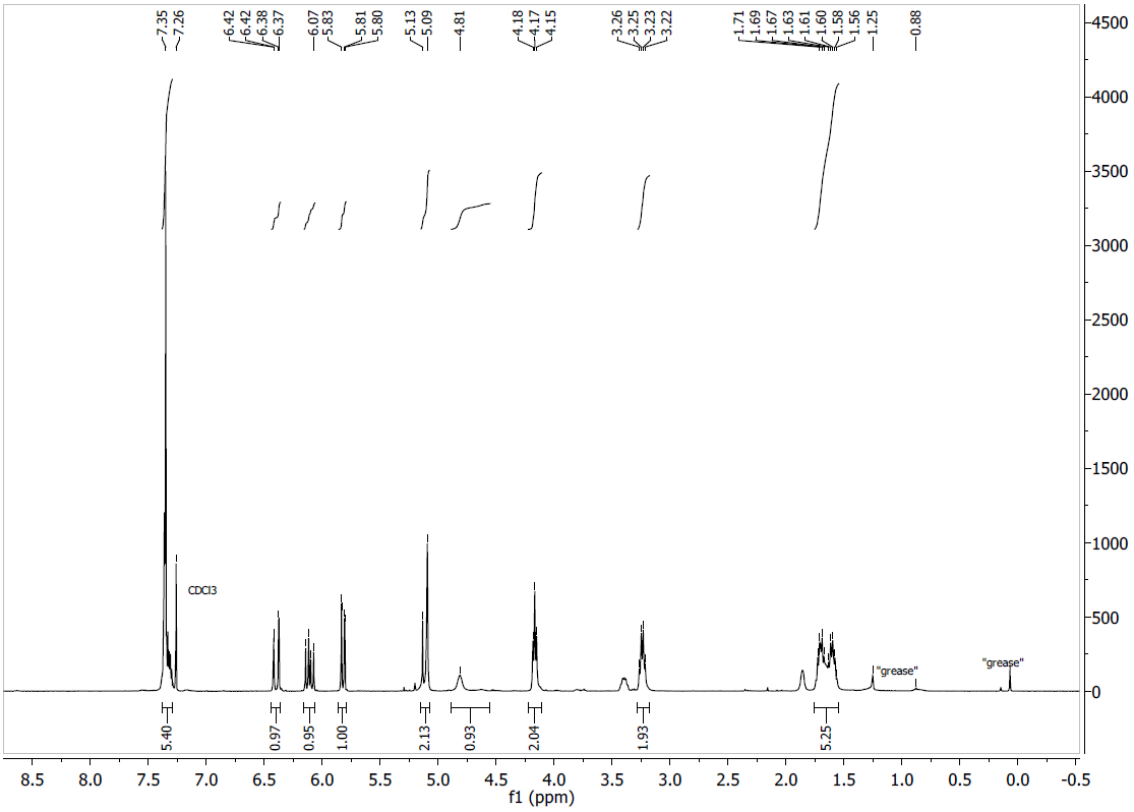


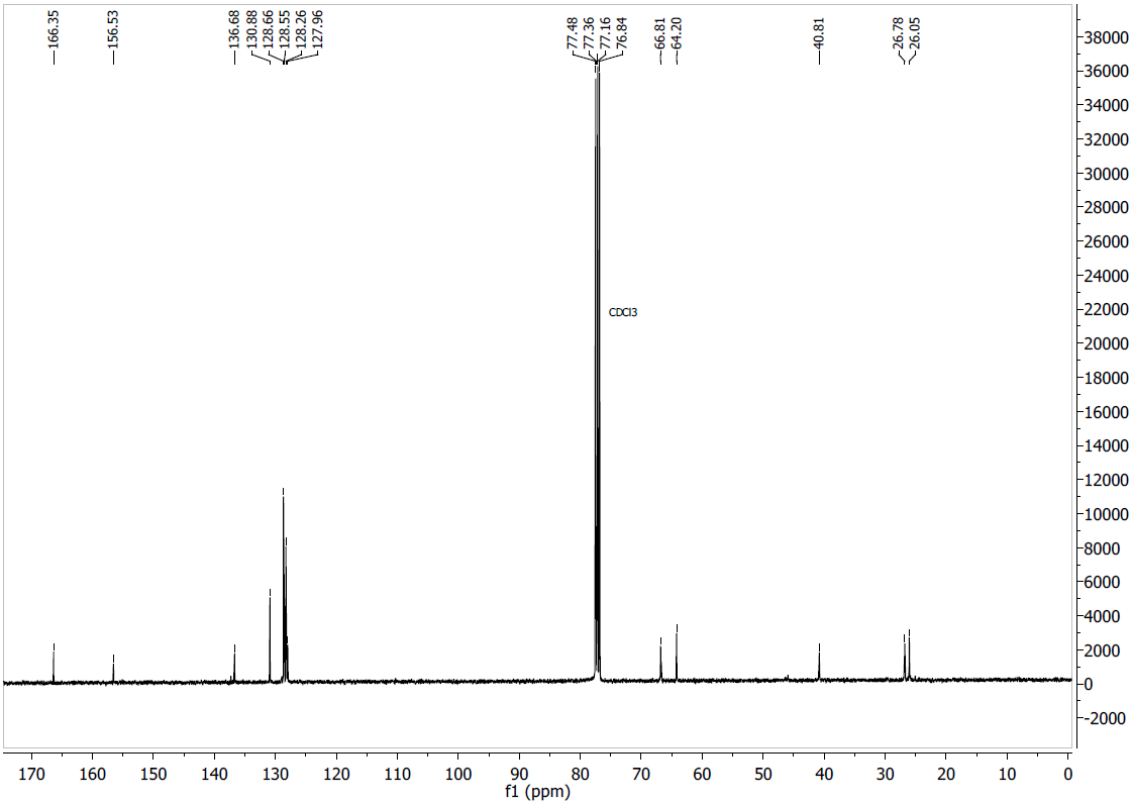
## 34. 4-(Benzyloxycarbonylamino)butyl phenylacetate (43).



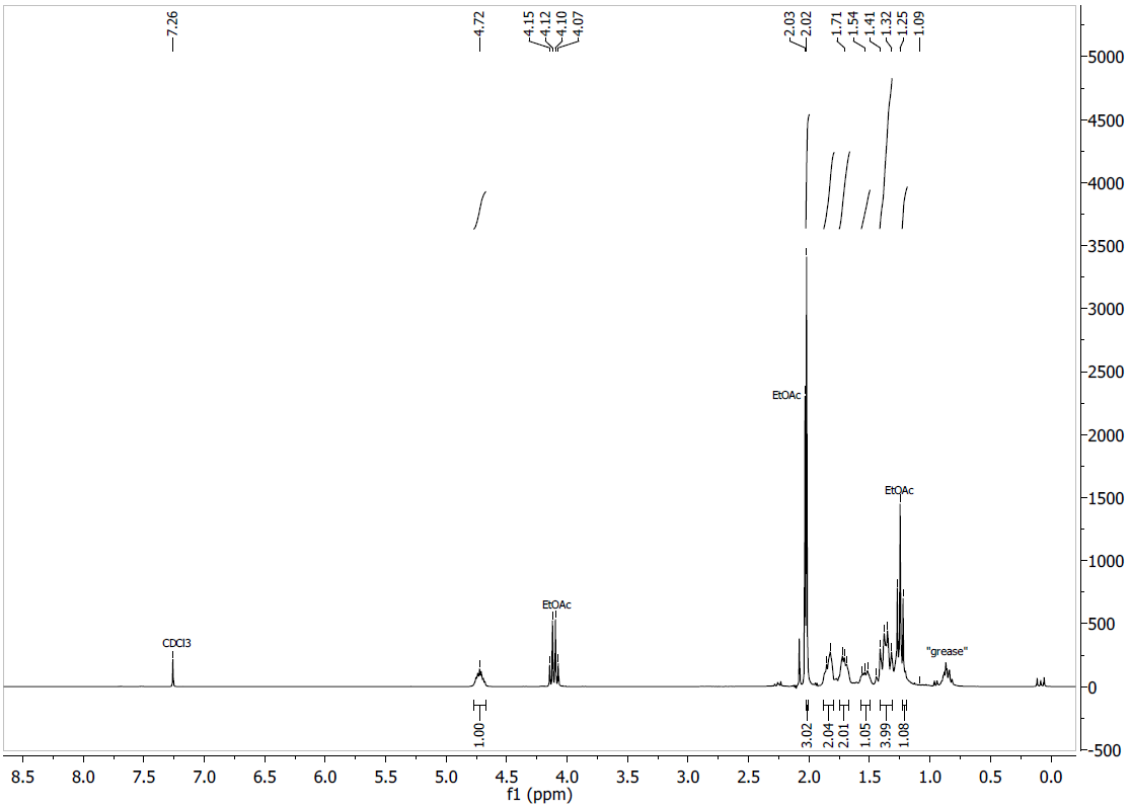



## 35. 4-(Benzyloxycarbonylamino)butyl acrylate (44).

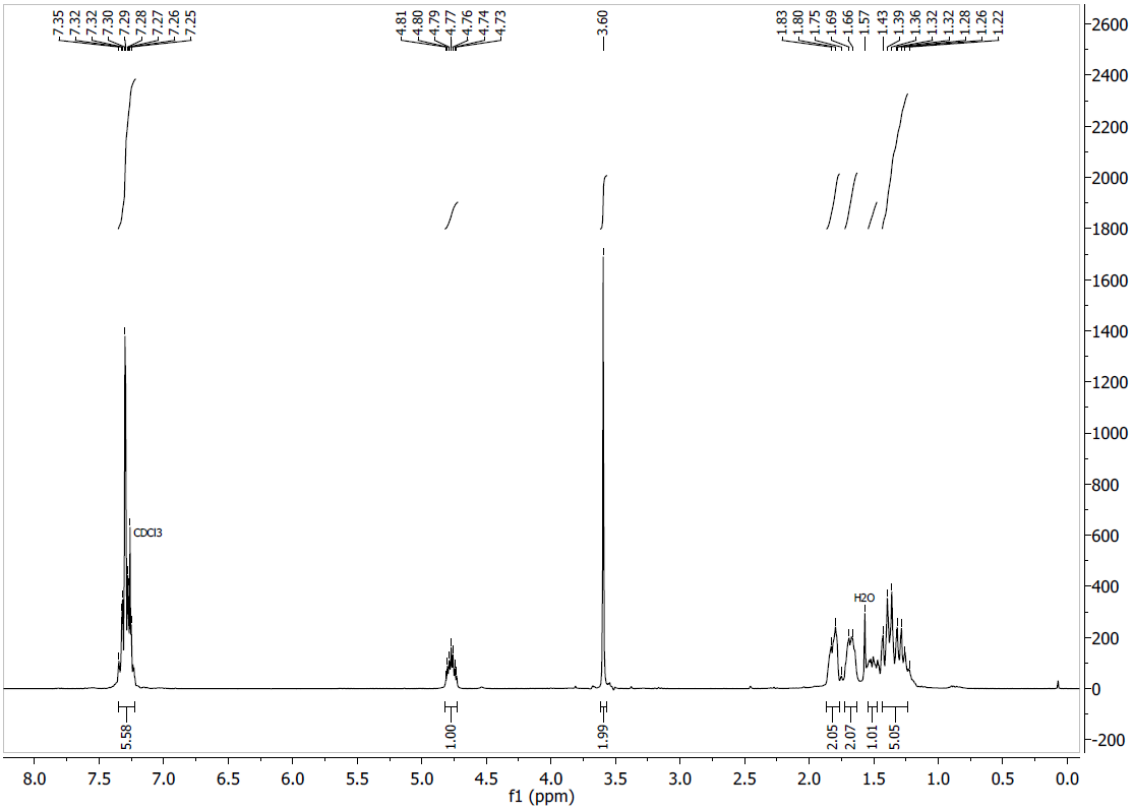


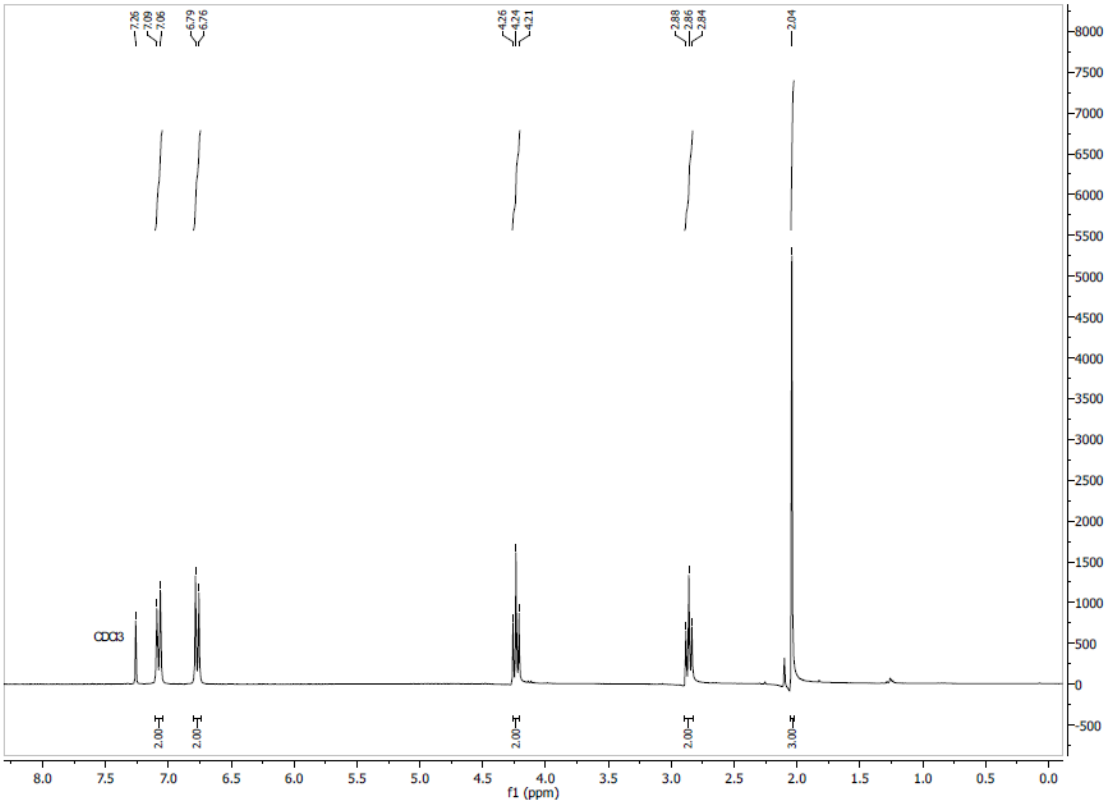
## 36. Cyclohexyl acetate (46).



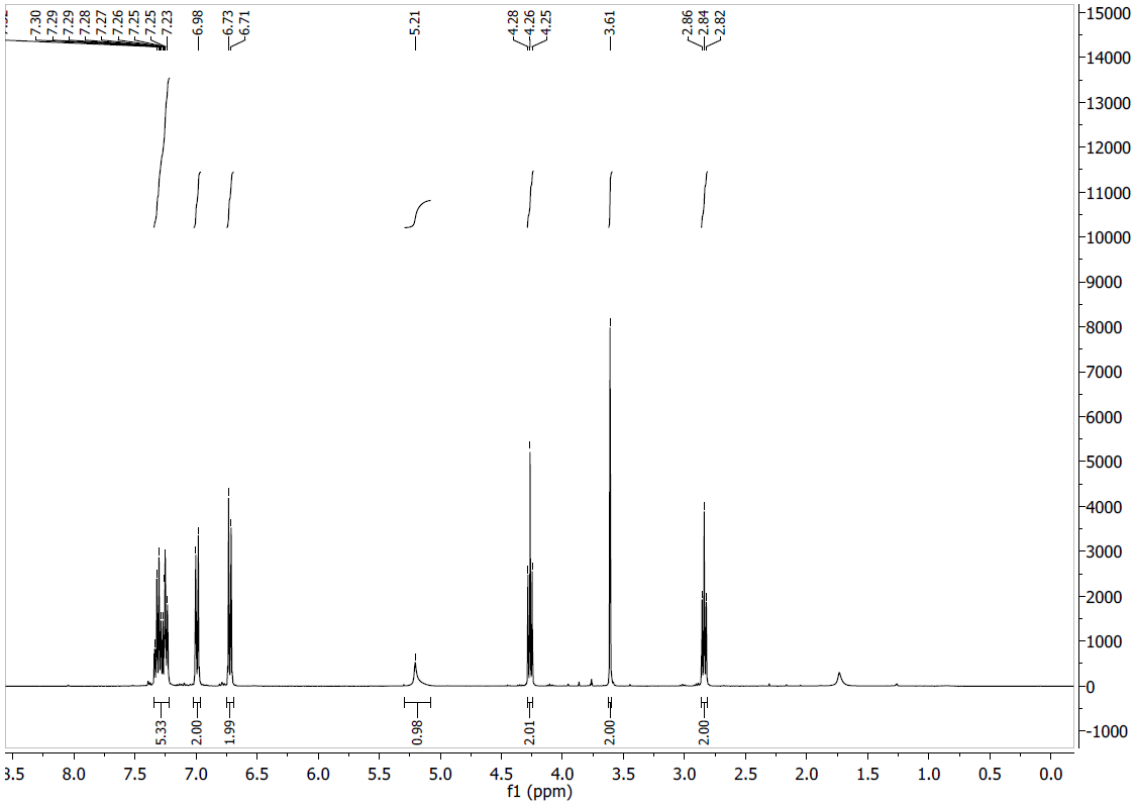

## 37. Cyclohexyl phenylacetate (47).

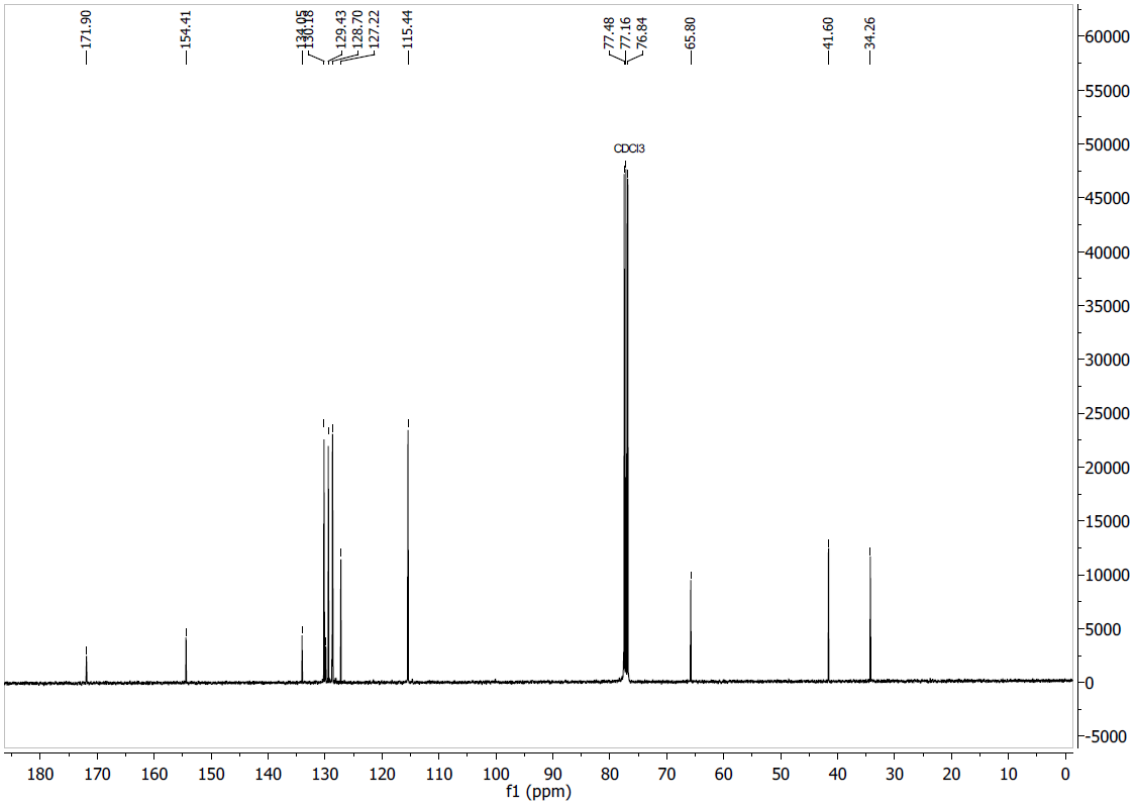


## 38. 2-(4-Hydroxyphenyl)ethyl acetate (54).

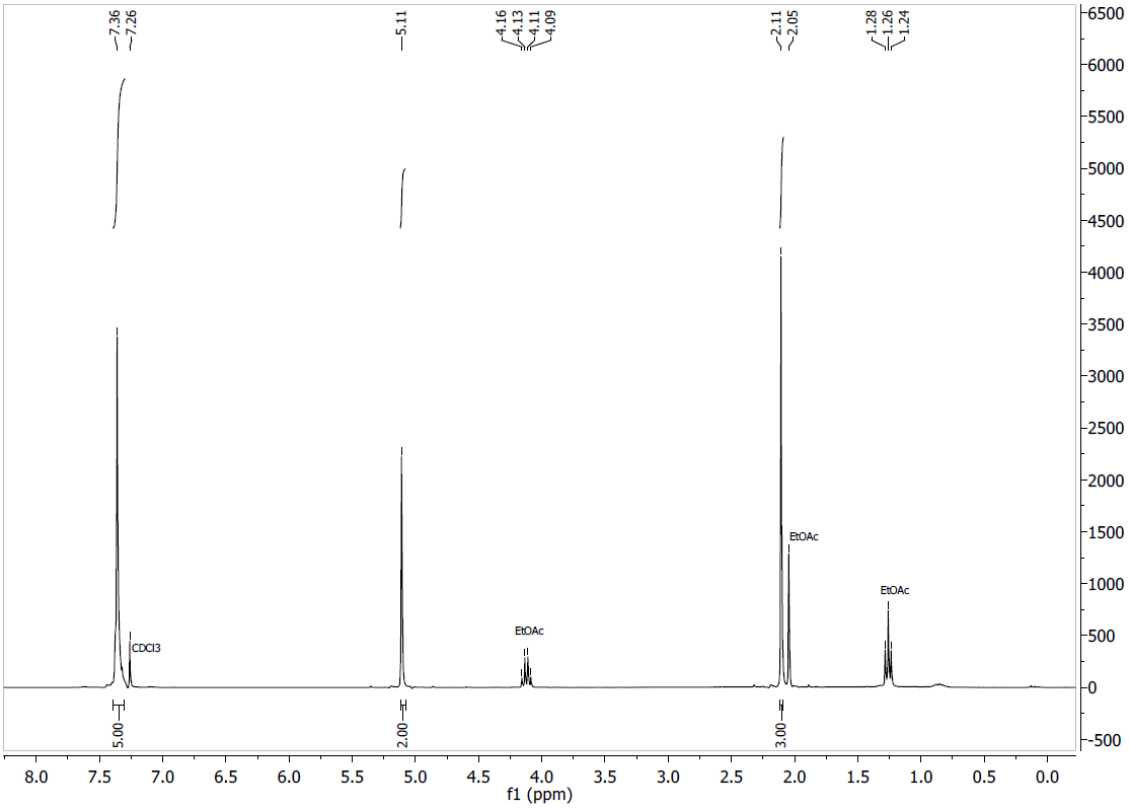


**39. 2-(4-Hydroxyphenyl)ethyl phenylacetate, monaspilosin (55).**

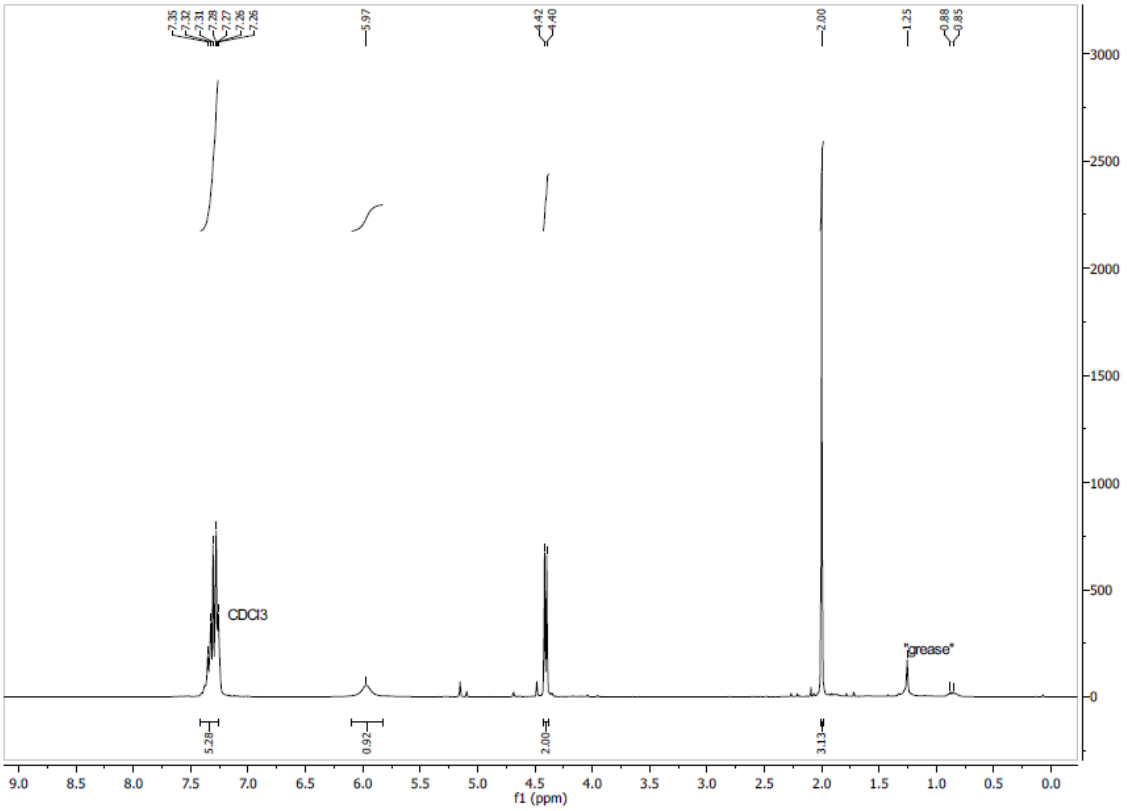


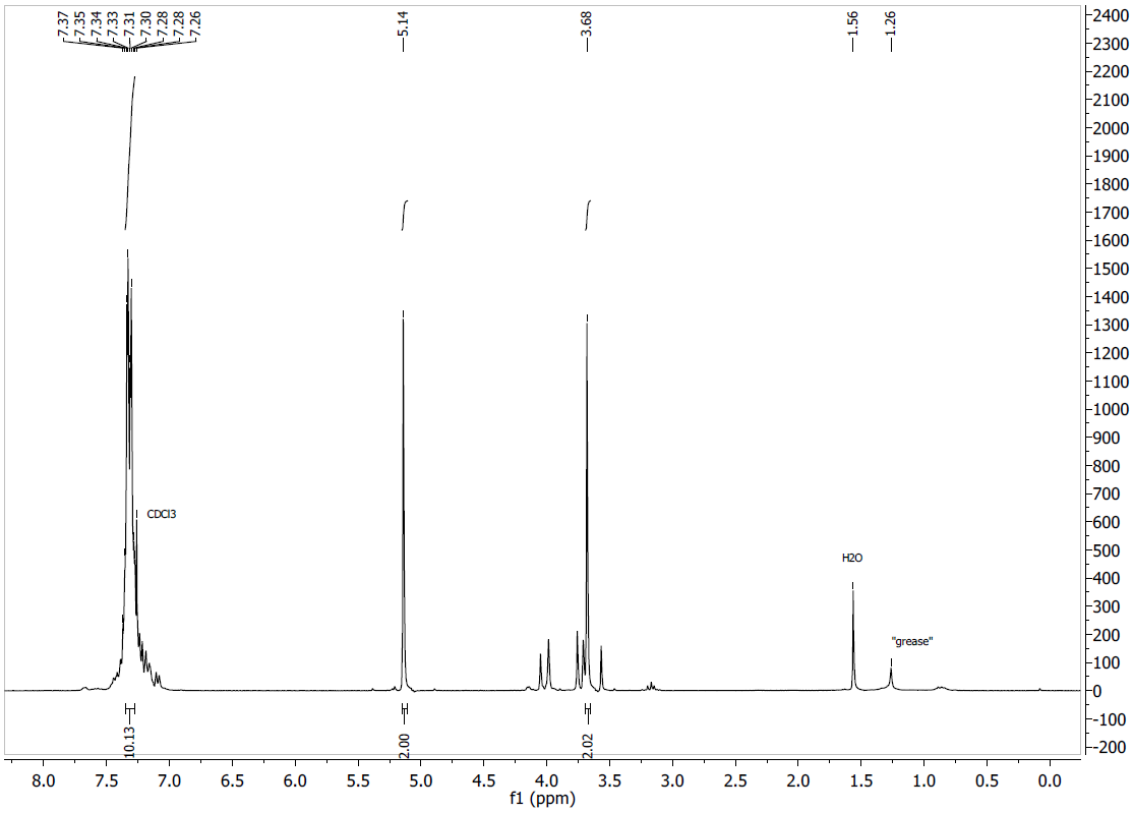
## 40. Benzyl acetate (57).



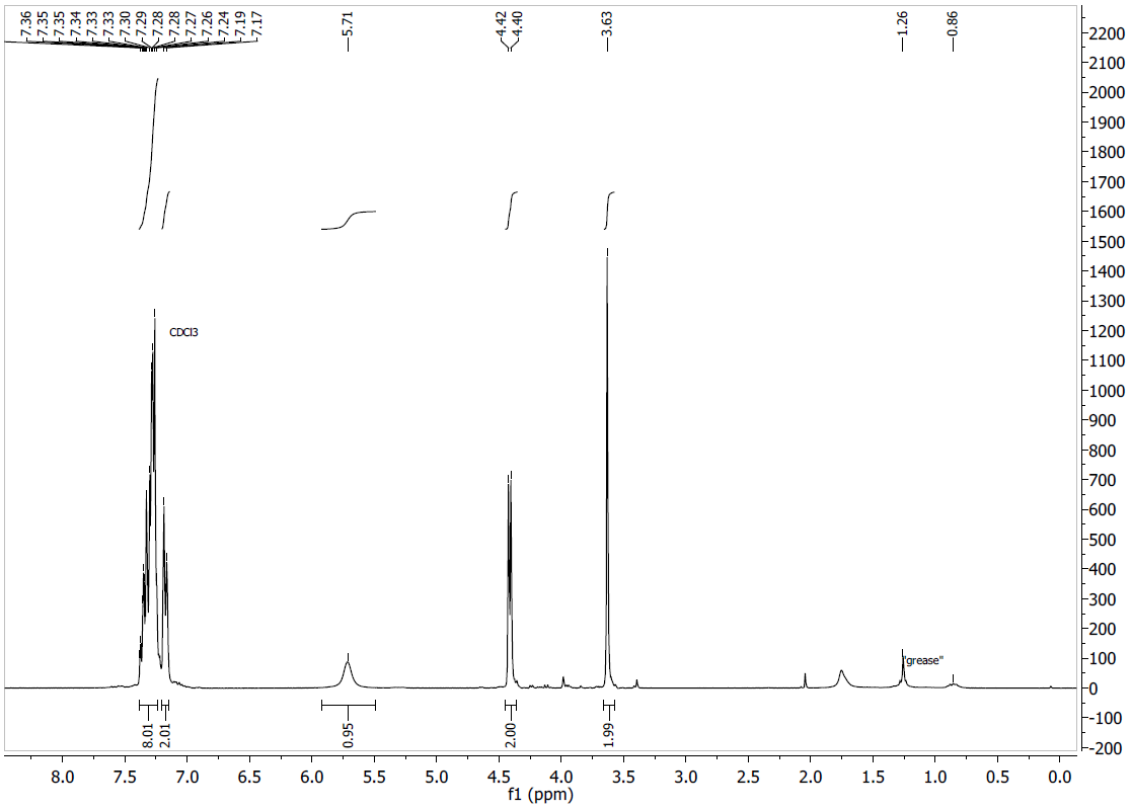

## 41. *N*-Benzylacetamide (58)



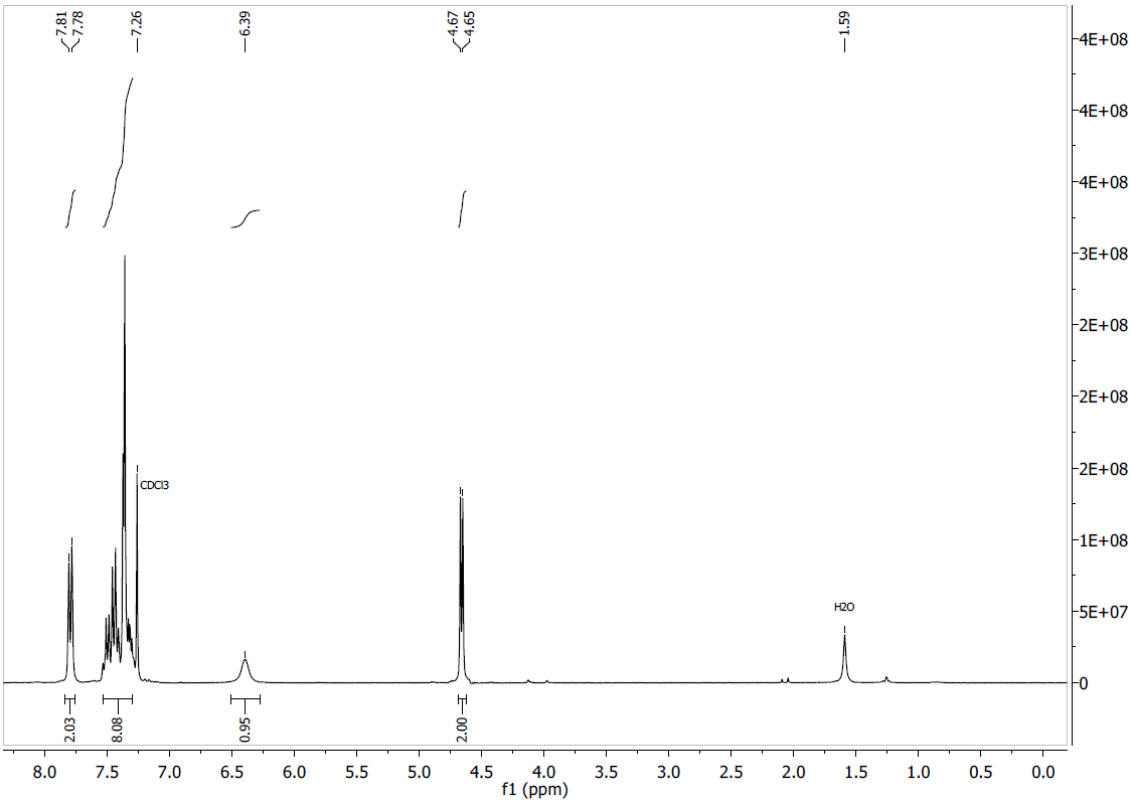
## 42. Benzyl phenylacetate (59).



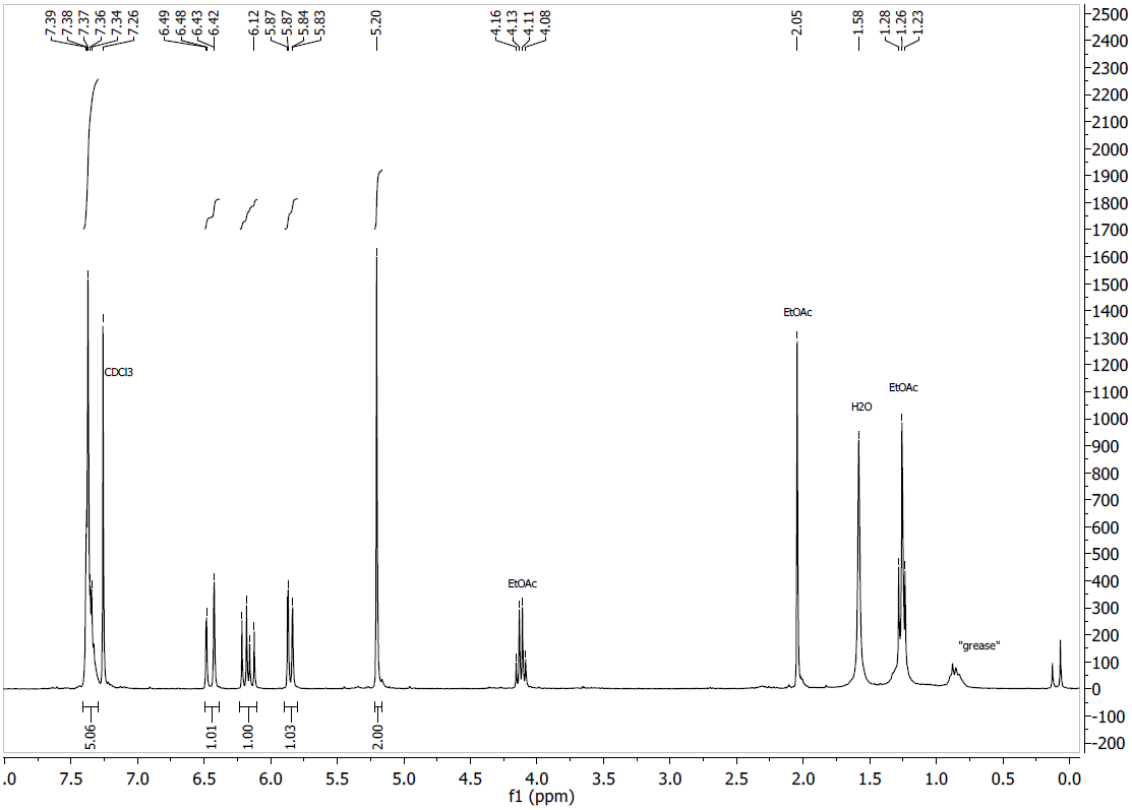
**43. *N*-Benzyl-phenylacetamide (60).**



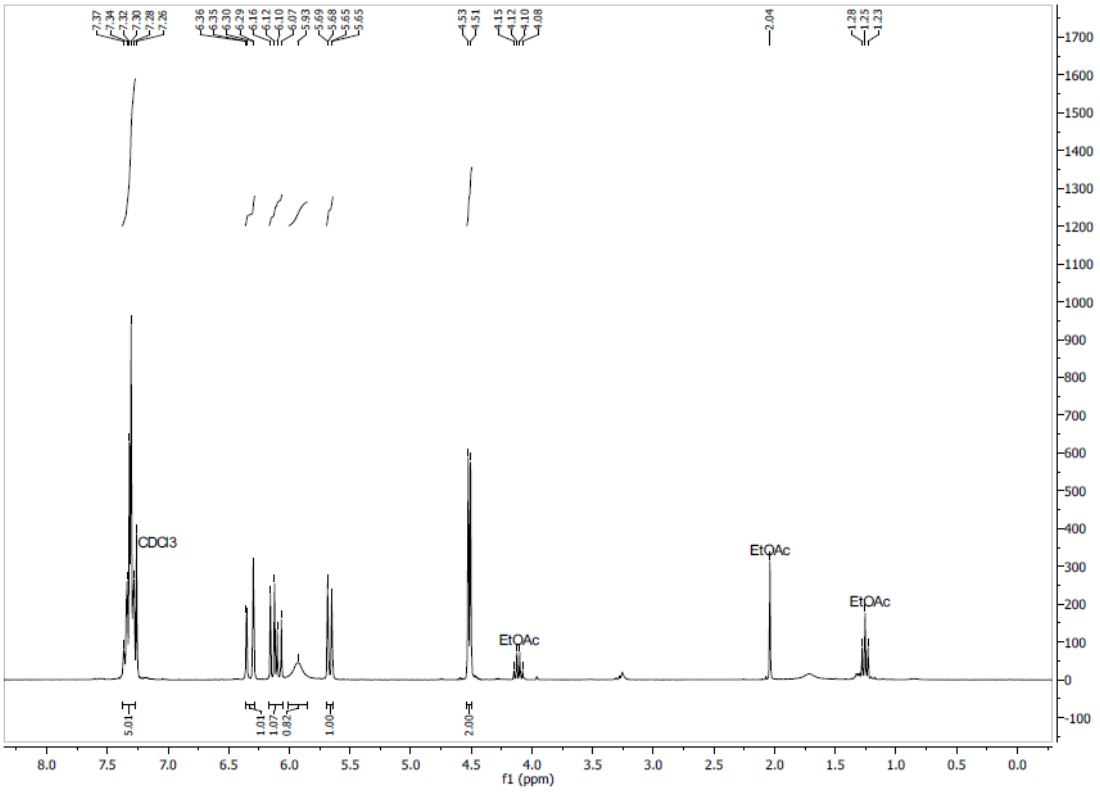

**44. *N*-Benzylbenzamide (61).**



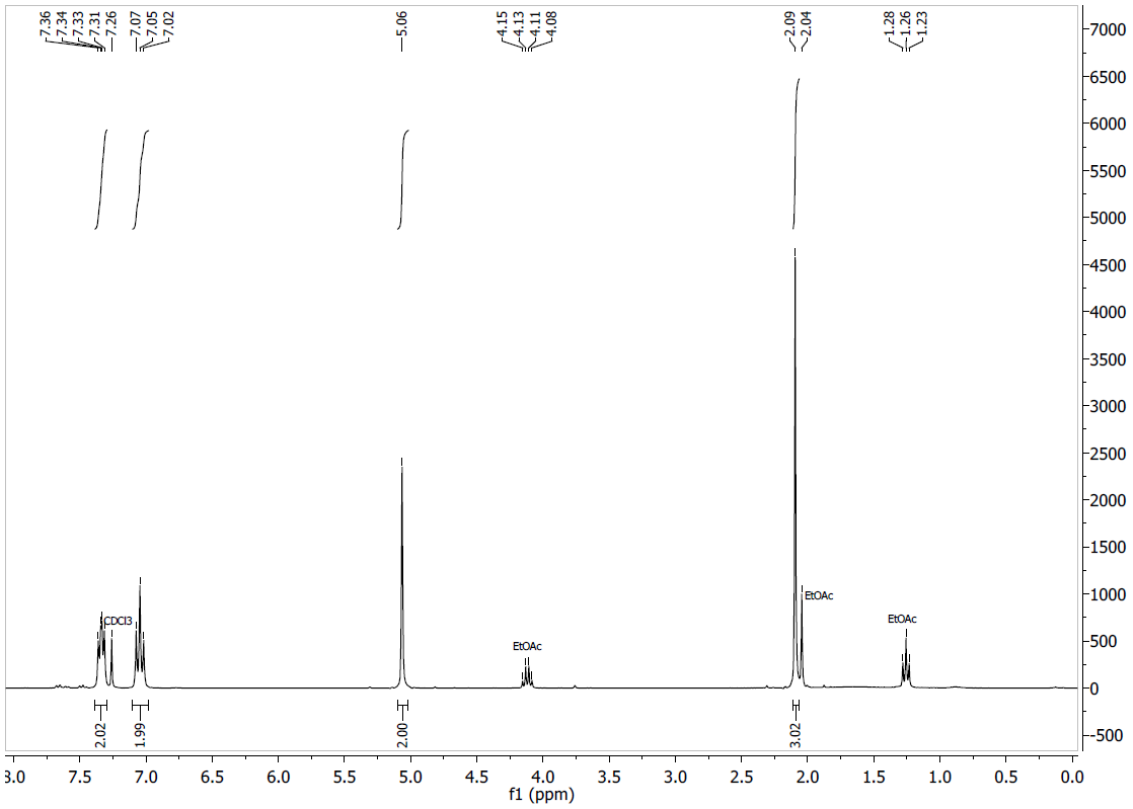
## 45. Benzyl acrylate (62).



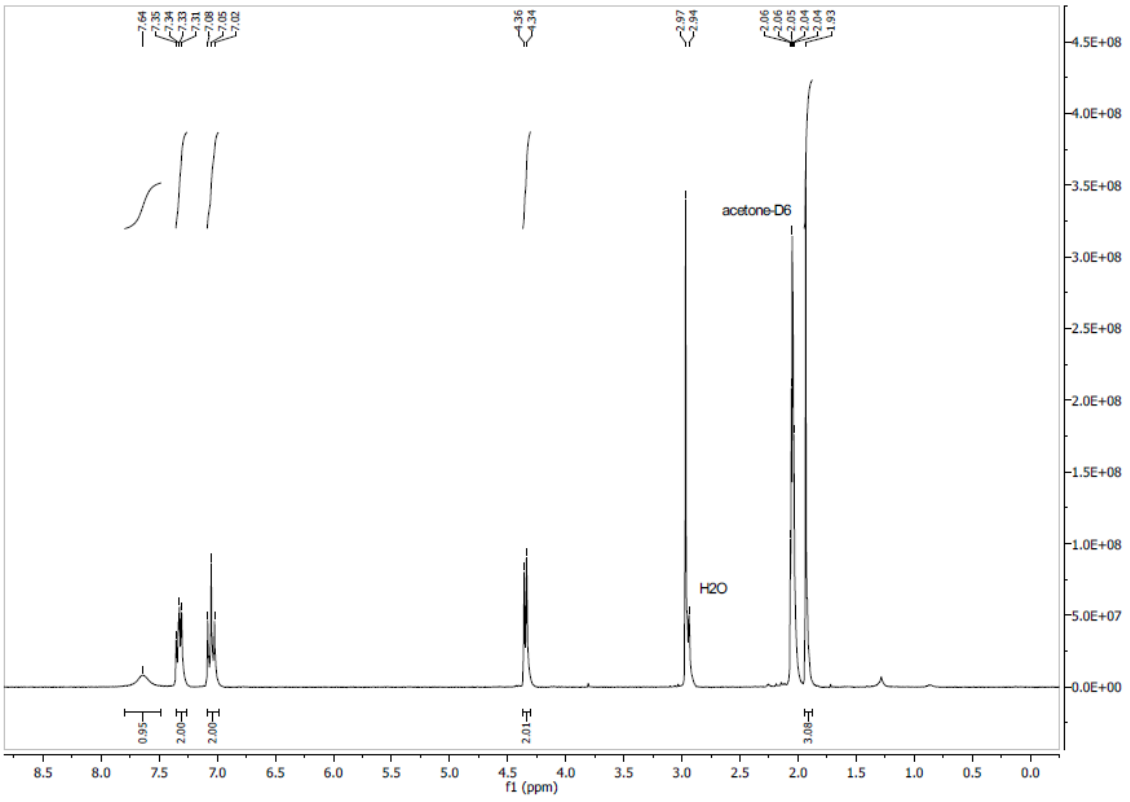

## 46. N-Benzylacrylamide (63)



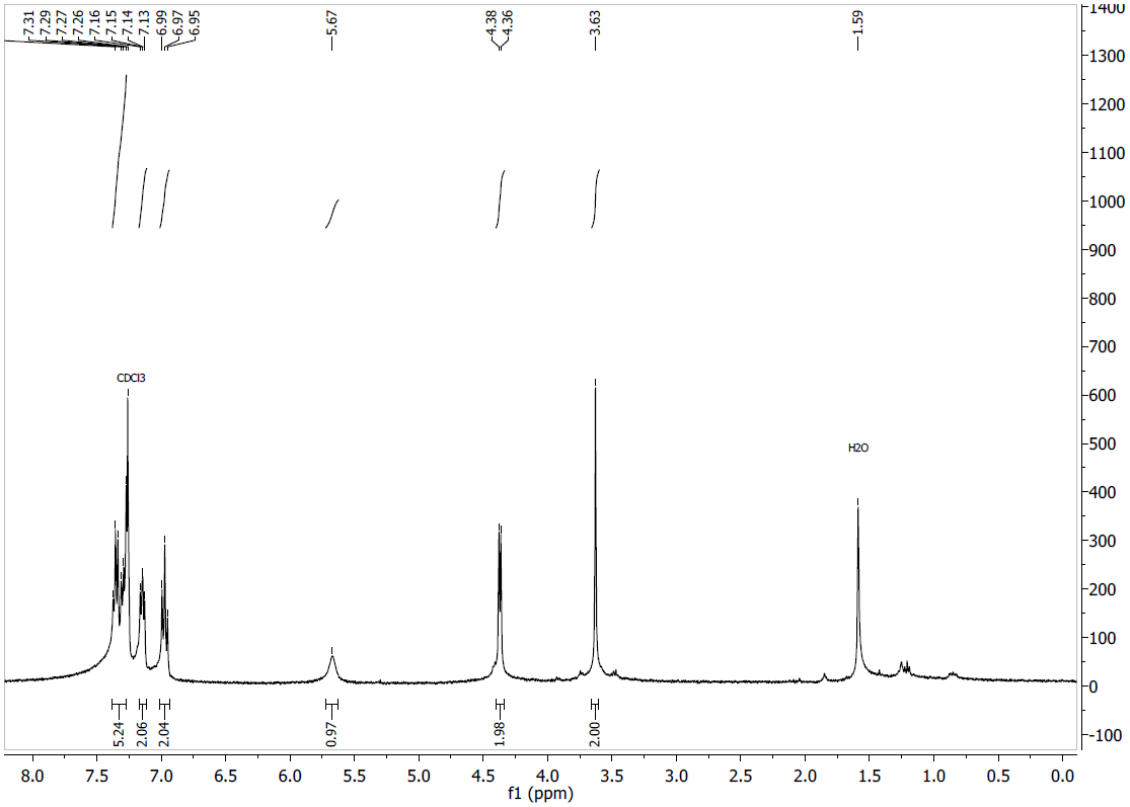
## 47. 4-Fluorobenzyl acetate (65).

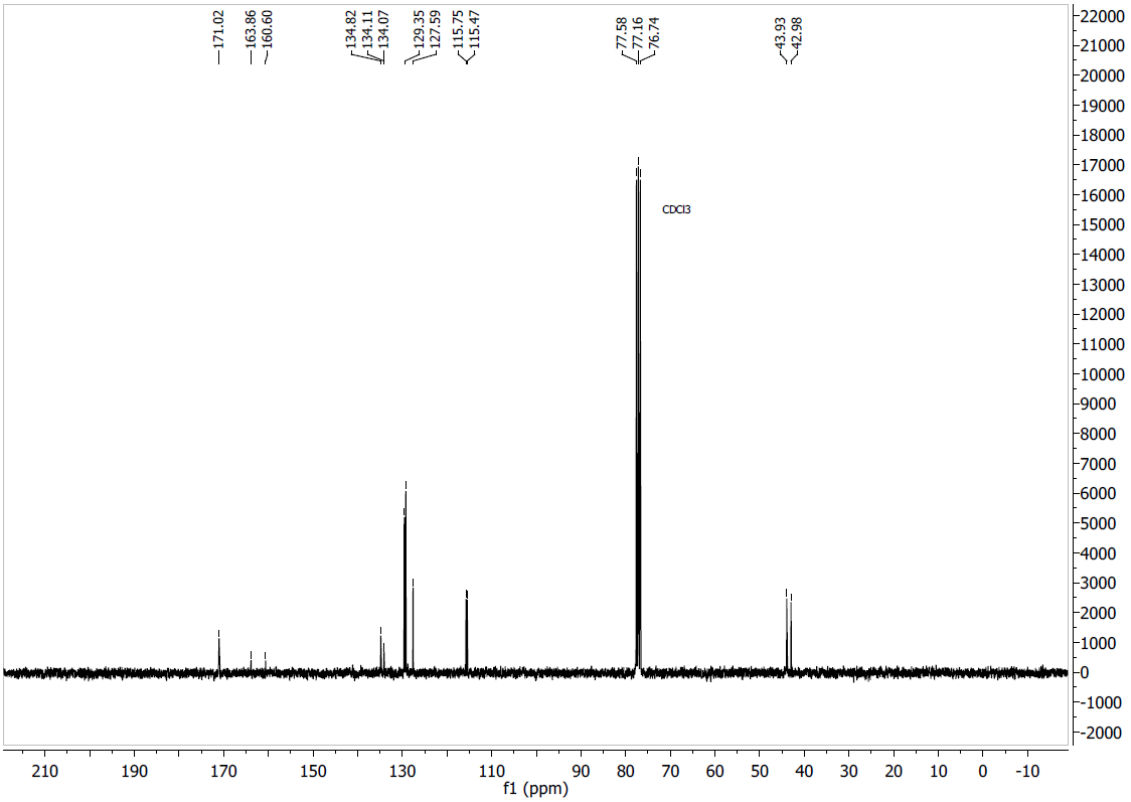



## 48. *N*-4-(Fluorobenzyl)acetamide (66).

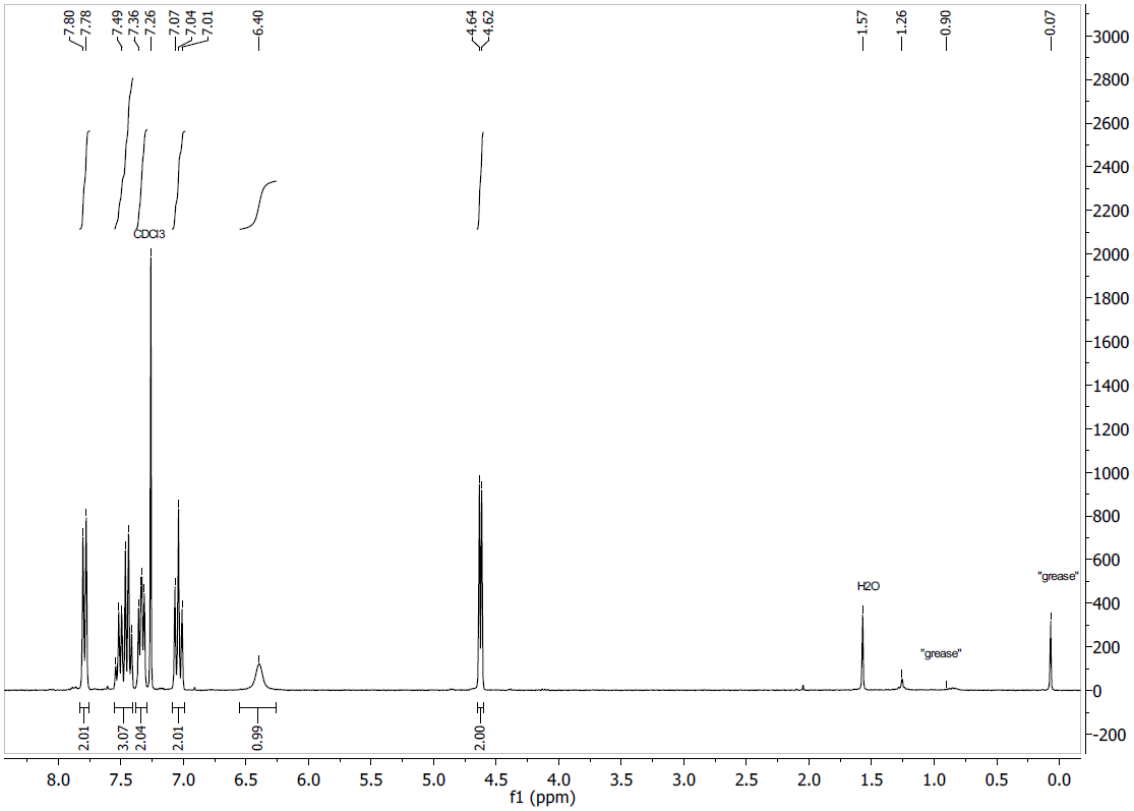


**49. *N*-(4-Fluorobenzyl)phenylacetamide (67).**



**50. *N*-(4-Fluorobenzyl)benzamide (68).**



**51. *N*-(4-Fluorobenzyl)acrylamide (69).**