

# Nucleophilic iron catalysis in transesterifications – scope and limitations.

*Silja Magens, and Bernd Plietker\*.*

Institut für Organische Chemie, Universität Stuttgart, Pfaffenwaldring 55, D-70569  
Stuttgart Germany.

[bernd.plietker@oc.uni-stuttgart.de](mailto:bernd.plietker@oc.uni-stuttgart.de)

|              |   |             |
|--------------|---|-------------|
| <b>S-I</b>   | <b>General remarks</b>                              | <b>S-2</b>  |
| <b>S-II</b>  | <b>Preparation of 4-Chlorophenyl ester</b>          | <b>S-2</b>  |
| <b>S-III</b> | <b>General procedure for transesterifications</b>   | <b>S-6</b>  |
| <b>S-IV</b>  | <b>Spectroscopic data</b>                           | <b>S-7</b>  |
| <b>S-V</b>   | <b><math>^1\text{H-NMR}</math> spectra of ester</b> | <b>S-23</b> |
| <b>S-VI</b>  | <b>Chirale HPLC chromatogram of chirale ester</b>   | <b>S-48</b> |
| <b>S-VII</b> | <b>References</b>                                   | <b>S-51</b> |

## S-I. General Remarks

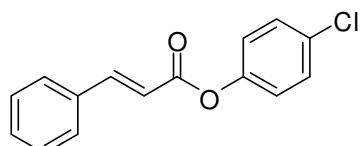
All solvents were purified by distillation over  $\text{CaH}_2$  prior to use. Flash-chromatography was done on silicagel 60 (230-400 mesh) using head pressure by means of compressed air. Infrared spectra (IR) were recorded as a thin film between KBr-plates. Proton and carbon nuclear magnetic resonance spectra were recorded in chloroform ( $d_1$ ) and referenced to the solvent signal or to the internal standard TMS. The multiplicities of the signals are given as d (doublet), t (triplet), q (quartet) and m (multiplet). GC-yields were obtained using *n*-dodecane as internal standard that was added in an amount equal to quantitative yield to the reaction.

## S-II Preparation of chlorophenyl ester:

### General Procedure 1:

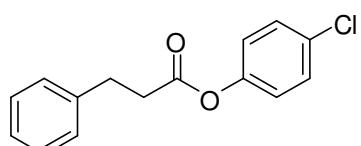
To a solution of the acid, chloro phenol (0.8 equiv.) and 4-dimethylaminopyridine (0.1 equiv.) in dichlormethane (0.5 mmol/ml) was added *N,N*-dicyclohexylcarbodiimide (0.8 equiv) at 0°C. After the reaction mixture was allowed to stir at ambient temperature overnight, it was filtered through a pad of silica gel and purified by flash chromatography (silica gel).

### Cinnamic acid 4-chlorophenyl ester (27)<sup>2</sup>



Ester **27** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 92%);  $R_f$  0.63 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.88 (d, 1 H,  $J$  = 16.0 Hz), 7.61-7.58 (m, 2H), 7.45-7.43 (m, 3 H), 7.39-7.36 (m, 2 H), 7.14-7.11 (m, 2 H) 6.62 (d, 1 H,  $J$  = 16.2 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 165.2, 149.3, 147.0, 134.0, 131.2, 130.9, 129.5, 129.0, 128.4, 123.0, 116.9 ppm; IR (KBr):  $\nu$  = 3055 (w), 1727 (s), 1629 (m), 1575 (m), 1481(m), 1447 (m), 1402 (w), 1335 (m), 1305 (m), 1280 (m), 1206 (m), 1133 (s), 1086 (m), 996 (m), 968 (m), 842 (s), 802 (m), 762 (s), 707 (m), 681 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV):  $m/z$  = 258 ( $M^+$ )<sup>(2)</sup>, 131 ( $\text{C}_9\text{H}_7\text{O}^+$ )<sup>(100)</sup>, 103 ( $\text{C}_8\text{H}_7^+$ )<sup>(45)</sup>, 77 ( $\text{C}_6\text{H}_5^+$ )<sup>(30)</sup>, 51 ( $\text{C}_4\text{H}_3^+$ )<sup>(10)</sup>.

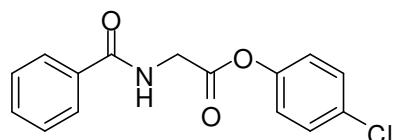
### 3-Phenylpropionic acid 4-chlorophenyl ester (49)<sup>3</sup>



Ester **49** was prepared acc. to GP-I and obtained as a colorless crystals; (yield: 87%)  $R_f$  0.59 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.34-7.29 (m, 4 H), 7.26-7.22 (m, 3H), 6.95-6.92 (m, 2 H), 3.06 (t, 2 H,  $J$  = 7.6 Hz), 2.88 (t, 2 H,  $J$  = 7.6 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  =

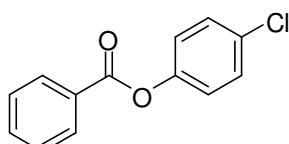
171.2, 149.1, 139.9, 131.2, 129.4, 128.6, 128.4, 126.5, 122.9, 35.9, 30.9 ppm;IR (KBr):  $\nu$  = 3057 (w), 3027 (w), 2925 (w), 1759 (s), 1602 (w), 1485 (m), 1451 (m), 1428 (m), 1379 (m), 1324 (w), 1293 (w), 1265 (w), 1200 (m), 1161 (m), 1119 (s), 1080 (s), 1010 (m), 943 (m), 925 (m), 911 (m), 844 (m), 780 (m), 732 (m), 717 (m), 694 (m)  $\text{cm}^{-1}$ ;GC-MS (EI 70 eV): m/z = 260 ( $M^+$ )<sup>(10)</sup>, 133 ( $C_9H_9O$ )<sup>(45)</sup>, 128 ( $C_6H_5OCl$ )<sup>(40)</sup>, 105 ( $C_8H_9$ )<sup>(100)</sup>, 91 ( $C_7H_7$ )<sup>(90)</sup>, 77 ( $C_6H_5$ )<sup>(20)</sup>, 65 ( $C_5H_5$ )<sup>(15)</sup>, 51 ( $C_4H_3$ )<sup>(10)</sup>.

### Hippuric acid 4-chlorophenyl ester (50)



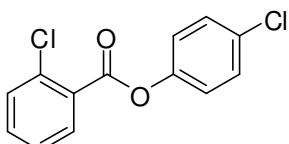
Ester **50** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 85 %);  $R_f$  0.23 (i-hexane/ethyl acetate 1:1);<sup>1</sup>H-NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.84-7.82 (m, 2H), 7.56-7.53 (m, 1 H), 7.48-7.45 (m, 2H), 7.38-7.35 (m, 2H), 7.11-7.08 (m, 2 H), 6.69 (s, 1H), 4.51 (d, 2H,  $J$  = 5.0 Hz) ppm;<sup>13</sup>C-NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 168.6, 167.6, 148.7, 133.5, 132.0, 131.7, 129.7, 128.7, 127.1, 122.7, 42.0 ppm; IR (KBr):  $\nu$  = 3338 (m), 3059 (w), 2937 (w), 1767 (s), 1642 (s), 1600 (w), 1578 (w), 1531 (s), 1486 (s), 1400 (m), 1377 (m), 1312 (m), 1270 (w), 1222 (s), 1174 (w), 1148 (s); 1079 (s), 1000 (m), 943 (w), 915 (m), 838 (s), 802 (m), 736 (m), 716 (m), 692 (s), 630 (s)  $\text{cm}^{-1}$ ;GC-MS (EI 70 eV): m/z = 253 ( $M\text{-HCl}$ )<sup>(2)</sup>, 207 ( $M\text{-C}_5H_8N$ )<sup>(15)</sup>, 162 ( $C_9H_8NO_2$ )<sup>(45)</sup>, 134 ( $C_8H_8NO$ )<sup>(20)</sup>, 105 ( $C_7H_5O$ )<sup>(100)</sup>, 77 ( $C_6H_5$ )<sup>(40)</sup>, 51 ( $C_4H_3$ )<sup>(15)</sup>.EA found: C: 62.05 %, H: 4.26 %, N: 4.86 %, Cl: 12.10 %;calcul.: C: 62.19 %, H: 4.17 %, N: 4.83, Cl: 12.24 %.

### Benzoic acid 4-chlorophenyl ester (51)



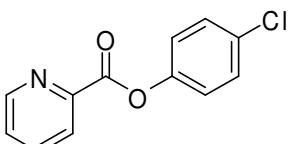
Ester **51** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 91%);  $R_f$  0.82 (i-hexane/ethyl acetate 3:1);<sup>1</sup>H-NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.20-8.18 (m, 2H), 7.66-7.63 (m, 1H), 7.54-7.50 (m, 2H), 7.41-7.38 (m, 2H), 7.18-7.15 (m, 2H) ppm;<sup>13</sup>C-NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 165.0, 149.4, 133.8, 131.3, 130.2, 129.6, 129.2, 128.6, 123.1 ppm;IR (KBr):  $\nu$  = 3063 (w), 1730 (s), 1594 (w), 1485 (m), 1450 (m), 1404 (w), 1353(w), 1314 (w), 1261 (m), 1200 (m), 1158 (m), 1012 (m), 936 (m), 874 (m), 807 (m), 701 (s), 683 (m)  $\text{cm}^{-1}$ ;GC-MS (EI 70 eV): m/z = 232 ( $M^+$ )<sup>(5)</sup>, 105 ( $C_7H_5O$ )<sup>(100)</sup>, 77 ( $C_6H_5$ )<sup>(40)</sup>, 51 ( $C_4H_3$ )<sup>(10)</sup>. EA:found: C: 67.06, H: 4.00, Cl: 15.18; calcul: C: 67.11, H: 3.90, Cl:15.24.

### 2-Chlorobenzoic acid 4-chlorophenyl ester (52)



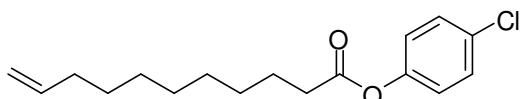
Ester **52** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 87 %);  $R_f$  0.79 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.04-8.02 (m, 1H) 7.54-7.48 (m, 2H), 7.41-7.38 (m, 3H), 7.21-7.18 (m, 2H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 163.8, 149.1, 134.5, 133.4, 131.9, 131.5, 131.4, 129.6, 128.9, 126.8, 123.0 ppm; IR (KBr):  $\nu$  = 3103 (w), 1751 (s), 1591 (m), 1572 (w), 1470 (m), 1435 (m), 1404 (w), 1293 (m), 1265 (m), 1244 (m), 1196 (m), 1164 (m), 1134 (m), 1082 (m), 1035 (s), 1008 (m), 950 (w), 872 (m), 803 (m), 775 (w), 731 (s), 688 (m), 664 (m), 638 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 266 ( $\text{M}^+$ )<sup>(2)</sup>, 139 ( $\text{C}_7\text{H}_4\text{ClO}^+$ )<sup>(100)</sup>, 111( $\text{C}_6\text{H}_4\text{Cl}^+$ )<sup>(30)</sup>, 99 ( $\text{C}_5\text{H}_4\text{Cl}^+$ )<sup>(5)</sup>, 85 ( $\text{C}_4\text{H}_2\text{Cl}^+$ )<sup>(2)</sup>, 75 ( $\text{C}_6\text{H}_3^+$ )<sup>(20)</sup>, 63 ( $\text{C}_5\text{H}_3^+$ )<sup>(5)</sup>, 50 ( $\text{C}_4\text{H}_2^+$ )<sup>(5)</sup>; HRMS (ESI  $\text{C}_{13}\text{H}_8\text{O}_2\text{Cl}_2+\text{Na}$ ) calcd.: 288.9794; found: 288.9805.

#### Picolinic acid 4-chlorophenyl ester (53)



Ester **53** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 78 %);  $R_f$  0.34 (i-hexane/ethyl acetate 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.86-8.85 (m, 1H), 8.28-8.26 (m, 1H), 7.95-7.91 (m, 1H), 7.59-7.56 (m, 1H), 7.42-7.39 (m, 2H), 7.23-7.20 (m, 2H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 163.6, 150.2, 149.4, 147.1, 137.3, 131.6, 129.6, 127.6, 126.0, 123.1 ppm; IR (KBr):  $\nu$  = 3093 (w), 3052 (w), 3036 (w), 1749 (s), 1581 (m), 1485 (m), 1435 (w), 1403 (w), 1303 (m), 1281 (m), 1237 (m), 1192 (s), 1155 (m), 1114 (m), 1073 (s), 1010 (m), 993 (m), 885 (m), 810 (m), 744 (m), 719 (m), 694 (m), 649 (w), 619 (m), 512 (s)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 233 ( $\text{M}^+$ )<sup>(1)</sup>, 189 ( $\text{M-CO}_2^+$ )<sup>(20)</sup>, 106 ( $\text{C}_6\text{H}_5\text{NO}^+$ )<sup>(50)</sup>, 78 ( $\text{C}_5\text{H}_5\text{N}^+$ )<sup>(100)</sup>, 63 ( $\text{C}_5\text{H}_3^+$ )<sup>(5)</sup>, 51 ( $\text{C}_4\text{H}_3^+$ )<sup>(15)</sup>. HRMS (ESI  $\text{C}_{12}\text{H}_8\text{O}_2\text{ClN}+\text{H}$ ) calcd.: 234.0316; found: 234.0307.

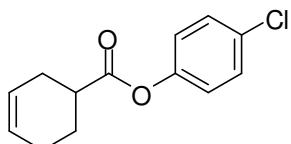
#### 10-Undecenic acid 4-chlorophenyl ester (54)



Ester **54** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 89 %);  $R_f$  0.74 (i-hexane/diethyl ether 6:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.35-7.32 (m, 2H), 7.03-7.01 (m, 2H), 5.81 (tdd, 1 H,  $J$  = 17.0, 10.3, 6.8 Hz), 5.02-4.97 (m, 1H), 4.95-4.92 (m, 1H), 2.54 (t, 2 H,  $J$  = 7.5 Hz), 2.07-2.02 (m, 2H), 1.77-1.71 (m, 2H), 1.42-1.30 (m, 10H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 172.1, 149.2, 139.2, 131.1, 129.4, 123.0, 114.2, 34.3, 33.8, 29.3, 29.2, 29.1, 29.0, 28.9, 24.9 ppm; IR (KBr):  $\nu$  = 3076 (w), 2925 (m), 2854 (m), 1758 (s), 1640 (w), 1486 (s), 1464 (w), 1361 (w), 1199 (s), 1162 (m), 1132 (m), 1084 (s), 1013 (m), 993 (w), 908 (m), 839 (w), 804 (w), 724 (w)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 294 ( $\text{M}^+$ )<sup>(1)</sup>, 167 ( $\text{C}_{11}\text{H}_{19}\text{O}^+$ )<sup>(10)</sup>, 149 ( $\text{C}_9\text{H}_9\text{O}_2^+$ )<sup>(25)</sup>, 128 ( $\text{C}_6\text{H}_5\text{OCl}^+$ )<sup>(100)</sup>, 99 ( $\text{C}_7\text{H}_{15}^+$ )<sup>(15)</sup>, 83

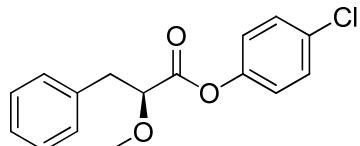
$(C_6H_{11})^+(25)$ , 67  $(C_5H_7)^+(35)$ , 55  $(C_4H_7)^+(95)$ ; HRMS (ESI  $C_{17}H_{23}O_2Cl+Na$ ) calcd.: 317.1279; found: 317.1278.

### Cyclohex-3-enic acid 4-Chlorophenyl ester (55)



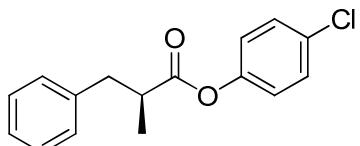
Ester **55** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 90 %);  $R_f$  0.58 (i-hexane/diethyl ether 6:1);  $^1H$ -NMR (500 MHz,  $CDCl_3$ ):  $\delta$  = 7.35-7.32 (m, 2H), 7.04-7.01 (m, 2H), 5.76-5.71 (m, 2H), 2.84-2.78 (m, 1H), 2.40-2.38 (m, 2H), 2.22-2.12 (m, 3H), 1.87-1.79 (m, 1H) ppm;  $^{13}C$ -NMR (125 MHz,  $CDCl_3$ ):  $\delta$  = 174.1, 149.3, 131.1, 129.4, 126.8, 124.9, 122.9, 39.4, 27.3, 25.0, 24.3 ppm; IR (KBr):  $\nu$  = 3027 (w), 2927 (w), 2841 (w), 1751 (s), 1652 (w), 1590 (w), 1486 (s), 1437 (w), 1403 (w), 1374 (w), 1302 (w), 1193 (s), 1161 (m), 1128 (s), 1091 (s), 1058 (m), 1013 (m), 993 (m), 949 (w), 918 (m), 873 (w), 853 (m), 807 (w), 775 (w), 725 (w), 644 (m)  $cm^{-1}$ ; GC-MS (EI 70 eV): m/z = 236 ( $M^+$ )(5), 128 ( $C_6H_5OCl^+$ )(10), 109 ( $C_7H_9O^+$ )(40), 81 ( $C_6H_9^+$ )(100), 53 ( $C_4H_4^+$ )(10); HRMS (ESI  $C_{13}H_{13}O_2Cl+Na$ ) calcd.: 259.0496; found: 259.0492.

### 2S-Methoxy-3phenylpropionic acid 4-chlorophenyl ester (56)



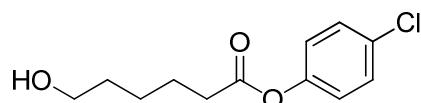
The corresponding acid was prepared acc. to ref. 4. Ester **56** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 84 %);  $[\alpha]_d^{20}$  ( $c=1$ , acetone) -8.7;  $R_f$  0.23 (i-hexane/diethyl ether 2:1);  $^1H$ -NMR (500 MHz,  $CDCl_3$ ):  $\delta$  = 7.34-7.26 (m, 7H), 6.89-6.86 (m, 2H), 4.22 (dd, 1H,  $J$  = 6.9, 5.7 Hz), 3.47 (s, 3H), 3.19-3.17 (m, 2H) ppm;  $^{13}C$ -NMR (125 MHz,  $CDCl_3$ ):  $\delta$  = 170.3, 148.7, 136.3, 131.5, 129.5, 128.5, 127.0, 122.7, 81.6, 58.5, 39.2 ppm; IR (KBr):  $\nu$  = 3029(w), 2930 (w), 2830 (w), 1765 (s), 1588 (w), 1485 (s), 1454 (m), 1403 (w), 1363 (w), 1196 (s), 1162 (s), 1140 (m), 1112 (s), 1084 (s), 1012 (m), 949(w), 907 (w), 880 (w), 824 (w), 806 (w), 744 (m), 698 (s)  $cm^{-1}$ ; GC-MS (EI 70 eV): m/z = 290 ( $M^+$ )(1), 135 ( $C_9H_{11}O^+$ )(100), 127 ( $C_6H_4ClO^+$ )(2), 103 ( $C_8H_7^+$ )(100), 91 ( $C_7H_7^+$ )(50), 77 ( $C_6H_5^+$ )(20), 65 ( $C_5H_5^+$ )(15), 51 ( $C_4H_3^+$ )(10); HRMS (ESI  $C_{16}H_{15}O_3Cl+Na$ ) calcd.: 313.0602; found: 313.0604.

### 2S-Methyl-3phenylpropionic 4-chlorophenyl ester (57)



The corresponding acid was prepared acc. to ref. 5 .Ester **57** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 86 %);  $\alpha_d^{20}$  ( $c=1$ , acetone) -98.6;  $R_f$  0.53 (i-hexane/diethyl ether 2:1);  $^1H$ -NMR (500 MHz,  $CDCl_3$ ):  $\delta$  = 7.33-7.27 (m, 4H), 7.25-7.22 (m, 3H), 6.86-6.83 (m, 2H), 3.12-3.08 (m, 1H), 2.99 (tq, 1H,  $J$  = 7.1 Hz), 2.85-2.81 (m, 1H), 1.32 (d, 3H,  $J$  = 7.1 Hz) ppm; $^{13}C$ -NMR (125 MHz,  $CDCl_3$ ):  $\delta$  = 174.4, 149.2, 138.9, 131.2, 129.4, 129.1, 128.5, 126.6, 122.9, 41.6, 39.8, 17.0 ppm; IR (KBr):  $\nu$  = 3028 (w), 2975 (w), 2935 (w), 1753 (s), 1604 (w), 1485 (s), 1454 (m), 1403 (w), 1360 (w), 1279 (w), 1196 (s), 1161 (m), 1133 (s), 1082 (s), 1011 (m), 939 (w), 910 (w), 874 (w), 803 (m), 740 (m), 698 (s) 668 (m)  $cm^{-1}$ ;GC-MS (EI 70 eV): m/z = 274 (M) $^+(20)$ , 147 ( $C_{10}H_{11}O$ ) $^+(45)$ , 128 ( $C_6H_5ClO$ ) $^+(10)$ , 119 ( $C_9H_{11}$ ) $^+(90)$ , 91 ( $C_7H_7$ ) $^+(100)$ , 77 ( $C_6H_5$ ) $^+(5)$ , 65 ( $C_5H_5$ ) $^+(5)$ , 51 ( $C_4H_3$ ) $^+(5)$ ; HRMS (ESI  $C_{16}H_{15}O_2Cl+Na$ ) calcd.: 297.0653; found: 297.0639.

### **6-Hydroxycapronic acid 4-chlorophenylester (77)**



Ester **77** was prepared acc. to GP-I and obtained as a colorless crystals (yield: 72 %);  $R_f$  0.26 (i-hexane/ethyl acetate 1:1);  $^1H$ -NMR (500 MHz,  $CDCl_3$ ):  $\delta$  = 7.35-7.32 (m, 2H), 7.04-7.01 (m, 2H), 3.66 (t, 2H,  $J$  = 6.2 Hz), 2.57 (t, 2H,  $J$  = 7.4 Hz), 1.81 (m, 2H), 1.65-1.60 (m, 2H), 1.52-1.46 (m, 2H) ppm; $^{13}C$ -NMR (125 MHz,  $CDCl_3$ ):  $\delta$  = 172.0, 149.1, 131.1, 129.5, 122.9, 62.6, 34.2, 32.3, 25.3, 24.6 ppm;IR (KBr):  $\nu$  = 3332 (b), 2935 (m), 2864 (w), 1754 (s), 1486 (s), 1366 (w), 1198(s), 1162 (m), 1129 (s), 1084 (s), 1013 (m), 839 (w)  $cm^{-1}$ ;GC-MS (EI 70 eV): m/z = 242 (M) $^+(1)$ , 170 ( $C_8H_6ClO_2$ ) $^+(2)$ , 128 ( $C_6H_5ClO$ ) $^+(100)$ , 115 ( $C_6H_{11}O_2$ ) $^+(70)$ , 97 ( $C_6H_9O$ ) $^+(30)$ , 69 ( $C_4H_5O$ ) $^+(40)$ ; HRMS (ESI  $C_{12}H_{15}O_3Cl+Na$ ) calcd.:265.0602; found: 265.0600.

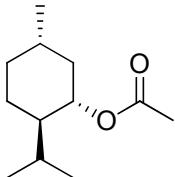
## **S-III. General Procedure for Transesterifications**

### **General Procedure 2:**

A 10mL-Schlenk tube was charged at room temperature with  $[Bu_4N][Fe(CO)_3NO]$  (2.5-10 mol%) and molsieves 4 Å (50-100 mg) under a  $N_2$ -atmosphere. Dry *n*-hexane (conc. 1mmol/mL) was added. After stirring for 15 min at room temperature the alcohol and the activated ester was added. The tube was immediately closed and heated to 80 °. After cooling to room temperature the reaction mixture was directly purified by means of flash chromatography.

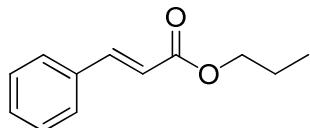
## **S-IV. Spectroscopic Data**

**Acetic acid *L*-mentyl ester (16)<sup>6</sup>**



Ester **16** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 92%);  $[\alpha]_d^{20}$  ( $c=1$ , acetone) -70.8;  $R_f$  0.59 (i-hexane/ethyl acetate, 20:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 4.67 (dt, 1H,  $J$  = 11.0 Hz, 4.5), 2.04 (s, 3H), 2.01-1.97 (m, 1H), 1.89-1.83 (m, 1H), 1.70-1.64 (m, 2H), 1.53-1.44 (m, 1H) 1.39-1.33 (m, 1H), 1.10-1.01 (m, 1H), 1.00- 0.93 (m, 1H), 0.90 (dd, 6H,  $J$  = 2.7, 6.5 Hz), 0.87-0.82 (m, 1H), 0.76 (d, 3H,  $J$  = 7.0 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 170.4, 74.2, 47.0, 40.9, 34.3, 31.4, 26.3, 23.5, 22.0, 21.4, 20.7, 16.4 ppm; IR (film):  $\nu$  = 2954 (m), 2928 (m), 2870(w), 1733(s) 1455 (w), 1369 (m), 1241 (s), 1023 (m), 904 (m) 730 (m) cm<sup>-1</sup>; MS (CI,  $\text{CH}_4$ ): m/z 199 ( $M+\text{H}$ )<sup>+</sup> (10), 155 ( $M-\text{C}_2\text{H}_3\text{O}$ )<sup>+</sup> (89), 139 ( $M-\text{C}_2\text{H}_3\text{O}_2$ )<sup>+</sup> (139), 138 ( $M-\text{C}_2\text{H}_4\text{O}_2$ )<sup>+</sup> (100), 123 ( $M-\text{C}_3\text{H}_7\text{O}_2$ )<sup>+</sup> (30), 95 ( $C_7\text{H}_{11}$ )<sup>+</sup> (60), 83 ( $\text{C}_6\text{H}_{11}$ )<sup>+</sup> (30), 81 ( $\text{C}_6\text{H}_9$ )<sup>+</sup> (45), 43 ( $\text{C}_3\text{H}_7$ )<sup>+</sup>.

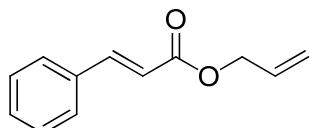
**Cinnamic acid propyl ester (38)<sup>7</sup>**



Ester **38** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 82% );  $R_f$  0.40(i-hexane/diethyl ether 5:1);

$^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.69 (d, 1H,  $J$  = 15.4 Hz), 7.54- 7.51 (m, 2H), 7.40-7.37 (m, 3H), 6.46 (d, 1 H,  $J$  = 15.8 Hz), 4.17 (t, 2H,  $J$  = 6.9 Hz), 1.74 (tq, 2H,  $J$  = 7.0), 1.00 (t, 3H,  $J$  = 7.4 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 167.1, 144.6, 134.5, 130.2, 128.9, 128.1, 118.3, 66.2, 22.1, 10.5 ppm; IR (Film):  $\nu$  = 3061 (w), 3028 (w), 2966 (m), 2878 (w), 1708 (s), 1636 (m), 1578 (w), 1496 (w), 1449 (m), 1389 (w), 1309 (m), 1269 (m), 1254 (m), 1201 (m), 1164 (s), 1061 (m), 978 (m), 936 (m), 863 (m), 765 /s), 709 (m), 683 (m) cm<sup>-1</sup>; GC-MS (EI 70 eV): m/z = 190 ( $M$ )<sup>+</sup> (20), 148 ( $M-\text{C}_3\text{H}_6$ )<sup>+</sup> (60), 131 ( $M-\text{C}_3\text{H}_7\text{O}$ )<sup>+</sup> (100), 103 ( $\text{C}_8\text{H}_7$ )<sup>+</sup> (60), 77 ( $\text{C}_6\text{H}_5$ )<sup>+</sup>(30), 51 ( $\text{C}_4\text{H}_3$ )<sup>+</sup> (15).

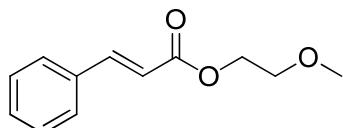
**Cinnamic acid allyl ester (39)<sup>8</sup>**



Ester **39** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 86%);  $R_f$  0.42(i-hexane/diethyl ether 5:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.72 (d, 1H,  $J$  = 15.9 Hz), 7.55-7.52 (m, 2H),

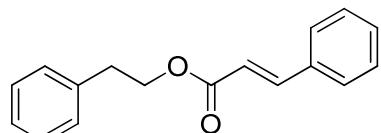
7.40-7.38 (m, 3H), 6.48 (d, 1H,  $J$  = 16.3 Hz), 6.00 (ddt, 1H,  $J$  = 16.4, 11.0 , 5.7 Hz), 5.38 (dd, 1H,  $J$  = 17.0, 1.9 Hz), 5.28 (dd, 1H,  $J$  = 10.7, 1.3 Hz), 4.72 (d, 2H,  $J$  = 5.7 Hz) ppm;  $^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 166.6, 145.1, 134.4, 132.3, 130.3, 128.9, 128.1, 118.3, 117.9, 65.2 ppm; IR (Film):  $\nu$  = 3062 (w), 3027 (w), 2941 (w), 1961 (w), 1708 (s), 1635 (m), 1577 (w), 1496 (w), 1449 (w), 1359 (w), 1307 (m), 1272 (m), 1251 (m), 1201 (m), 1157 (s), 977 (m), 921 (m), 863 (m), 765 (s), 709 (m), 682 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 188 ( $\text{M}^+$ )(10), 143 ( $\text{M-C}_2\text{H}_5\text{O}^+$ )(15), 131( $\text{C}_9\text{H}_7\text{O}^+$ )(100), 103 ( $\text{C}_8\text{H}_7$ ) $^+$ (75), 77 ( $\text{C}_6\text{H}_6$ ) $^+$  (50), 51 ( $\text{C}_4\text{H}_3$ ) $^+$ (15).

#### Cinnamic acid 2-methoxyethyl ester (40)<sup>9</sup>



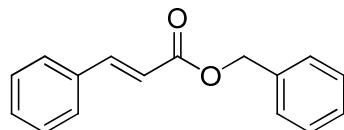
Ester **40** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 72%);  $R_f$  0.47(i-hexane/diethyl ether 1:1);  $^1\text{H}$ -NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.72 (d, 1H,  $J$  = 12.0 Hz), 7.54-7.51 (m, 2H), 7.40-7.37 (m, 3H), 6.50 (d, 1H,  $J$  = 12.0 Hz), 4.37 (t, 2H,  $J$  = 4.7 Hz), 3.67 (t, 2H,  $J$  = 4.8 Hz), 3.43 (s, 3H) ppm;  $^{13}\text{C}$ -NMR (125 Hz,  $\text{CDCl}_3$ ):  $\delta$  = 167.0, 145.2, 134.4, 130.3, 128.9, 128.1, 117.9, 70.6, 63.6, 59.1 ppm; IR (KBr):  $\nu$  = 2885 (w), 1708 (s), 1636 (m), 1578 (w), 1496 (w), 1450 (m), 1368 (w), 1309 (m), 1255 (m), 1201 (m), 1166 (s), 1125 (m), 1040 (m), 978 (m), 864 (m), 766 (s), 710 (m) 683 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 206 ( $\text{M}^+$ )(1), 171 ( $\text{M-CH}_4\text{O}^+$ )(5), 148 ( $\text{M-C}_3\text{H}_6\text{O}^+$ )(55), 131 ( $\text{C}_9\text{H}_7\text{O}^+$ )(100), 103 ( $\text{C}_8\text{H}_7$ ) $^+$ (60), 77 ( $\text{C}_6\text{H}_5$ ) $^+$ (40), 58 ( $\text{C}_3\text{H}_6\text{O}^+$ )(30), 51 ( $\text{C}_4\text{H}_3$ ) $^+$  (15).

#### Cinnamic acid 2-phenylethyl ester (41)<sup>6</sup>



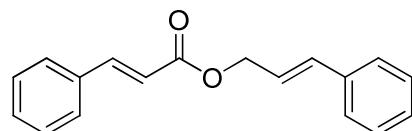
Ester **41** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 91%);  $R_f$  0.81 (i-hexane/ethyl acetate 3:1);  $^1\text{H}$ -NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.68 (d, 1H,  $J$  = 16.1 Hz), 7.52-7.50 (m, 2H), 7.38-7.37 (m, 3H), 7.34-7.30 (m, 2H), 7.27-7.22 (m, 3 H), 6.43 (d, 1H,  $J$  = 16.1 Hz), 4.43 (t, 2H,  $J$  = 7.0 Hz), 3.02 (t, 2H,  $J$  = 7.0 Hz) ppm;  $^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 166.9, 144.8, 137.9, 134.4, 130.3, 128.9, 128.8, 128.5, 128.1, 126.6, 118.1, 65.0, 35.2 ppm; IR (KBr):  $\nu$  = 3060 (w), 3028 (w), 2949 (w), 2980 (w), 1705 (s) 1635 (m), 1465 (w), 1449 (m), 1385 (w), 1311 (s), 1282 (m), 1170 (s), 1072 (m), 982 (m) 868 (m), 767 (s) 731 (m) 698 (s), 681 (s), 600 (w) 570 (w)  $\text{cm}^{-1}$ ; MS (ESI): m/z 275 ( $\text{M+Na}^+$ ).

#### Cinnamic acid benzyl ester (42)<sup>10</sup>



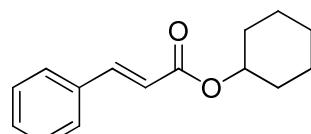
Ester **42** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 96%);  $R_f$  0.41 (i-hexane/diethyl ether 5:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.74 (d, 1H,  $J$  = 15.8 Hz), 7.54-7.52 (m, 2H), 7.43-7.33 (m, 8H), 6.50 (d, 1H,  $J$  = 16.1 Hz), 5.26 (s, 2H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 166.8, 145.2, 136.0, 134.4, 130.4, 128.9, 128.6, 128.3, 128.1, 117.9, 66.4 ppm; IR (KBr):  $\nu$  = 3062 (w), 3030 (w), 2951 (w), 2889 (w), 1965 (w), 1707 (s), 1634 (m), 1577 (w), 1496 (m), 1449(m), 1375 (m), 1306 (m), 1268 (m), 1252 (m), 1201 (m), 1154 (s), 1071 (w), 976 (m), 908 (m), 862 (m), 765 (m), 731 (m), 695 (m), 682 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 238 ( $M^+$ )<sup>(15)</sup>, 220 ( $M-\text{H}_2\text{O}^+$ )<sup>(5)</sup>, 192 ( $M-\text{C}_2\text{H}_6\text{O}^+$ )<sup>(60)</sup>, 147 ( $M-\text{C}_7\text{H}_7^+$ )<sup>(5)</sup>, 131( $\text{C}_9\text{H}_7\text{O}^+$ )<sup>(90)</sup>, 115 ( $\text{C}_9\text{H}_7^+$ )<sup>(10)</sup>, 103 ( $\text{C}_8\text{H}_7^+$ )<sup>(40)</sup>, 91 ( $\text{C}_7\text{H}_7^+$ )<sup>(100)</sup>, 77 ( $\text{C}_6\text{H}_5^+$ )<sup>(50)</sup>, 65 ( $\text{C}_5\text{H}_5^+$ )<sup>(25)</sup>, 51 ( $\text{C}_4\text{H}_3^+$ )<sup>(25)</sup>.

#### Cinnamic acid cinnamyl ester (**43**)<sup>11</sup>



Ester **43** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 88%);  $R_f$  0.41 (i-hexane/diethyl ether 5:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.74 (d, 1H,  $J$  = 16.2 Hz), 7.55-7.53 (m, 2H), 7.42-7.39 (m, 5H), 7.35-7.32 (m, 2H), 7.28-7.25 (m, 1H), 6.72 (d, 1H,  $J$  = 15.8 Hz), 6.49 (d, 1H,  $J$ =16.3 Hz), 6.37 (td, 1H,  $J$  = 16.0, 6.3 Hz), 4.88 (dd, 2H,  $J$  = 6.3, 1.2 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 166.7, 145.1, 136.2, 134.4, 134.3, 130.4, 128.9, 128.6, 128.1, 128.0, 126.6, 123.3, 117.9, 65.2 ppm; IR (KBr):  $\nu$  = 3060 (w), 3027 (w), 2942 (w), 2873 (w), 1965 (w), 1705 (s), 1635 (m), 1577 (w), 1495 (m), 1449 (m), 1376 (w), 1308 (m), 1252 (m), 1201 (m), 1158 (s), 1112 (m), 1070 (w), 964 (m), 907 (m), 863 (m), 766 (m), 728 (s), 683 (s)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 264 ( $M^+$ )<sup>(2)</sup>, 219 ( $M-\text{CHO}_2$ ), 173 ( $M-\text{C}_7\text{H}_7^+$ )<sup>(2)</sup> 131 ( $\text{C}_9\text{H}_7\text{O}^+$ )<sup>(100)</sup>, 115 ( $\text{C}_9\text{H}_7^+$ )<sup>(40)</sup>, 103 ( $\text{C}_8\text{H}_7^+$ )<sup>(30)</sup>, 91 ( $\text{C}_7\text{H}_7^+$ )<sup>(15)</sup>, 77 ( $\text{C}_6\text{H}_5^+$ )<sup>(20)</sup>, 51 ( $\text{C}_4\text{H}_3^+$ )<sup>(10)</sup>.

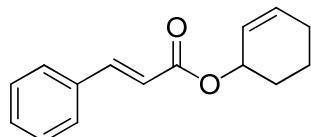
#### Cinnamic acid cyclohexyl ester (**44**)<sup>12</sup>



Ester **44** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 79%);  $R_f$  0.68 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.69 (d, 1H,  $J$  = 16.1 Hz), 7.54-7.54 (m, 2H), 7.40-7.37 (m, 3H), 6.46 (d, 1H,  $J$  = 15.9 Hz), 6.02-5.98 (m, 1H), 5.80-5.77 (m, 1H), 5.43-5.39 (m, 1H), 2.16-2.10 (m, 1H), 2.06-1.99 (m, 1H), 1.98-1.91 (m, 1H), 1.85-1.77 (m, 2H), 1.72-1.64 (m, 1H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 166.6, 144.5, 134.5, 132.8, 130.2, 128.9, 128.1, 125.8, 118.6,

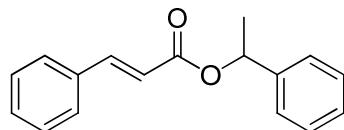
68.2, 28.4, 24.9, 18.9 ppm; IR (KBr):  $\nu$  = 3029 (w), 2933 (m), 2866 (w), 1702 (s), 1635 (m), 1577 (w), 1496 (w), 1449 (m), 1330 (m), 1302 (m), 1269 (m), 1249 (m), 1201 (m), 1157 (s), 1096 (w), 1070 (m), 1009 (m), 976 (m), 915 (m), 863 (m), 765 (s), 707 (m), 682 (m).  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 228 (M)<sup>+</sup>(5), 183 (M-C<sub>2</sub>H<sub>5</sub>O)<sup>+</sup> (85), 174 (M-C<sub>6</sub>H<sub>9</sub>)<sup>+</sup>(5), 147 (C<sub>9</sub>H<sub>7</sub>O<sub>2</sub>)<sup>+</sup>(10), 131 (C<sub>9</sub>H<sub>7</sub>O)<sup>+</sup> (100), 103 (C<sub>8</sub>H<sub>7</sub>)<sup>+</sup> (60), 81 (C<sub>6</sub>H<sub>9</sub>)<sup>+</sup> (85), 77 (C<sub>6</sub>H<sub>5</sub>)<sup>+</sup> (50), 51 (C<sub>4</sub>H<sub>3</sub>)<sup>+</sup> (15).

#### Cinnamic acid cyclohex-2-enyl ester (**45**)<sup>13</sup>



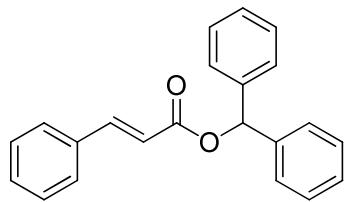
Ester **45** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 86%); R<sub>f</sub> 0.68 (i-hexane/diethyl ether 3:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.69 (d, 1H, J = 16.1 Hz), 7.54-7.54 (m, 2H), 7.40-7.37 (m, 3H), 6.46 (d, 1H, J = 15.9 Hz), 6.02-5.98 (m, 1H), 5.80-5.77 (m, 1H), 5.43-5.39 (m, 1H), 2.16-2.10 (m, 1H), 2.06-1.99 (m, 1H), 1.98-1.91 (m, 1H), 1.85-1.77 (m, 2H), 1.72-1.64 (m, 1H) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  = 166.6, 144.5, 134.5, 132.8, 130.2, 128.9, 128.1, 125.8, 118.6, 68.2, 28.4, 24.9, 18.9 ppm; IR (KBr):  $\nu$  = 3029 (w), 2933 (m), 2866 (w), 1702 (s), 1635 (m), 1577 (w), 1496 (w), 1449 (m), 1330 (m), 1302 (m), 1269 (m), 1249 (m), 1201 (m), 1157 (s), 1096 (w), 1070 (m), 1009 (m), 976 (m), 915 (m), 863 (m), 765 (s), 707 (m), 682 (m).  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 228 (M)<sup>+</sup>(5), 183 (M-C<sub>2</sub>H<sub>5</sub>O)<sup>+</sup> (85), 174 (M-C<sub>6</sub>H<sub>9</sub>)<sup>+</sup>(5), 147 (C<sub>9</sub>H<sub>7</sub>O<sub>2</sub>)<sup>+</sup>(10), 131 (C<sub>9</sub>H<sub>7</sub>O)<sup>+</sup> (100), 103 (C<sub>8</sub>H<sub>7</sub>)<sup>+</sup> (60), 81 (C<sub>6</sub>H<sub>9</sub>)<sup>+</sup> (85), 77 (C<sub>6</sub>H<sub>5</sub>)<sup>+</sup> (50), 51 (C<sub>4</sub>H<sub>3</sub>)<sup>+</sup> (15).

#### Cinnamic acid 1-phenylethyl ester (**46**)<sup>14</sup>



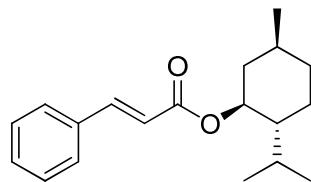
Ester **46** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 76%); R<sub>f</sub> 0.65 (i-hexane/diethyl ether 3:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.70 (d, 1H, J = 16.0 Hz), 7.54-7.51 (m, 2H), 7.42-7.35 (m, 7H), 7.32-7.28 (m, 1H), 6.48 (d, 1H, J = 15.8 Hz), 6.03 (q, 1H, J = 6.5 Hz), 1.62 (d, 3H, J = 6.4 Hz) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  = 166.3, 144.9, 141.7, 134.4, 130.3, 128.9, 128.5, 128.1, 127.9, 126.1, 118.4, 72.4, 22.3 ppm; IR (KBr):  $\nu$  = 3062 (w), 3030 (w), 2979 (w), 2932 (w), 1966 (w), 1704 (s), 1635 (m), 1578 (w), 1495 (m), 1449 (m), 1333 (m), 1303 (m), 1268 (m), 1253 (m), 1201 (m), 1164 (s), 1061 (m), 1028 (m), 977 (m), 907 (m), 863 (w), 763 (m), 729 (s), 696 (s), 682 (s), 647 (w)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 252 (M)<sup>+</sup>(1), 237 (M-CH<sub>3</sub>)<sup>+</sup>(1), 206 (M-C<sub>2</sub>H<sub>6</sub>O)<sup>+</sup>(30), 174 (M-C<sub>6</sub>H<sub>6</sub>)<sup>+</sup>(3), 161 (M-C<sub>7</sub>H<sub>7</sub>)<sup>+</sup> (10), 147 (M-C<sub>7</sub>H<sub>5</sub>O)<sup>+</sup>(3), 131 (C<sub>9</sub>H<sub>7</sub>O)<sup>+</sup>(60), 105 (C<sub>7</sub>H<sub>5</sub>O)<sup>+</sup>(100), 77 (C<sub>6</sub>H<sub>5</sub>)<sup>+</sup>(50), 51 (C<sub>4</sub>H<sub>3</sub>)<sup>+</sup> (15).

#### Cinnamic acid diphenylmethyl ester (**47**)<sup>15</sup>



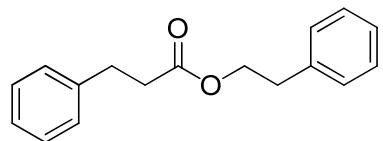
Ester **47** was prepared acc. to GP-II and obtained as a colorless crystals (yield 73%);  $R_f$  0.70 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.79 (d, 1H,  $J$  = 16.1 Hz), 7.57-7.54 (m, 2H), 7.43-7.36 (m, 11H), 7.33-7.29 (m, 2H), 7.05 (s, 1H), 6.59 (d, 1H,  $J$  = 16.3 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 165.7, 145.4, 140.2, 134.3, 130.4, 128.9, 128.5, 128.2, 127.9, 127.2, 118.0, 77.0 ppm; IR (KBr):  $\nu$  = 3056 (w), 3027 (w), 2922 (w), 1960 (w), 1711 (s), 1635 (m), 1598 (w), 1575 (w), 1493 (m), 14446 (m), 1357 (w), 1329 (m), 1309 (m), 1296 (m), 1281 (m), 1202 (m), 1161 (s), 1077 (m), 1030 (w), 979 (s), 915 (w), 866 (m), 765 (m), 746 (s), 693 (s), 644 (m), 604 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV):  $m/z$  = 314 ( $M^+$ )<sup>2</sup>, 268 ( $M-\text{C}_2\text{H}_6\text{O}$ )<sup>+</sup>(18), 254 ( $M-\text{C}_2\text{H}_4\text{O}_2$ )<sup>+</sup>(15), 236 ( $M-\text{C}_6\text{H}_6$ )<sup>+</sup>(10), 223 ( $M-\text{C}_7\text{H}_7$ )<sup>+</sup>(8), 183 ( $M-\text{C}_9\text{H}_7\text{O}$ )<sup>+</sup>(10), 167 ( $\text{C}_{13}\text{H}_{11}$ )<sup>+</sup>(100), 152 ( $\text{C}_{12}\text{H}_8$ )<sup>+</sup>(30), 131 ( $\text{C}_9\text{H}_7\text{O}$ )<sup>+</sup>(55), 115 ( $\text{C}_9\text{H}_7$ )<sup>+</sup>(5), 103 ( $\text{C}_8\text{H}_7$ )<sup>+</sup>(25), 77 ( $\text{C}_6\text{H}_5$ )<sup>+</sup>(45), 51 ( $\text{C}_4\text{H}_3$ )<sup>+</sup>(15).

#### Cinnamic acid *L*-mentyl ester (**48**)<sup>16</sup>



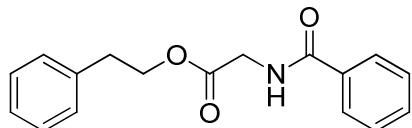
Ester **48** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 67% );  $[\alpha]_d^{20}$  (c=1, acetone) -57.7;  $R_f$  0.73 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.68 (d, 1H,  $J$  = 16.1 Hz), 7.54-7.51 (m, 2H), 7.40-7.36 (m, 3H), 6.44 (d, 1H,  $J$  = 16.3 Hz), 4.83 (dt, 1H,  $J$  = 11.0, 4.4 Hz), 2.09-2.05 (m, 1H), 1.96-1.90 (m, 1H), 1.73-1.68 (m, 2H), 1.57-1.50 (m, 1H), 1.49-1.42 (m, 1H), 1.14-1.01 (m, 2H), 0.94-0.87 (m, 1H), 0.92 (dd, 6H,  $J$  = 6.9, 4.1 Hz), 0.79 (d, 3H,  $J$  = 7.0 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 166.6, 144.4, 134.5, 130.1, 128.8, 128.0, 118.7, 74.3, 47.2, 41.0, 34.3, 31.4, 26.4, 23.5, 22.1, 20.8, 16.4 ppm; IR (Film):  $\nu$  = 3061 (w), 2953 (m), 2925 (m), 2868 (m), 2361 (w), 1704 (s), 1637 (m), 1449 (m), 1306 (m), 1268 (m), 1201 (m), 1169 (s), 1095 (w), 979 (m), 917 (w), 863 (w), 765 (m), 709 (m), 683 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV):  $m/z$  = 286 ( $M^+$ )<sup>5</sup>, 149 ( $\text{C}_9\text{H}_9\text{O}_2$ )<sup>+</sup>(10), 138 ( $\text{C}_{10}\text{H}_{18}$ )<sup>+</sup>(60), 131 ( $\text{C}_9\text{H}_7\text{O}$ )<sup>+</sup>(100), 123 ( $\text{C}_9\text{H}_{15}$ )<sup>+</sup>(15), 103 ( $\text{C}_8\text{H}_7$ )<sup>+</sup>(45), 95 ( $\text{C}_7\text{H}_{11}$ )<sup>+</sup>(50), 81 ( $\text{C}_6\text{H}_9$ )<sup>+</sup>(30), 55 ( $\text{C}_3\text{H}_3\text{O}$ )<sup>+</sup>(15).

#### 3-Phenylpropionic acid 2-phenylethyl ester (**58**)<sup>17</sup>



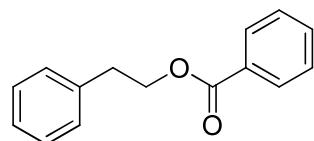
Ester **58** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 89%);  $R_f$  0.45 (i-hexane/diethyl ether 5:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.31-7.16 (m, 10H), 4.28 (t, 2H,  $J$  = 7.4 Hz), 2.93-2.89 (m, 4H), 2.61 (t, 2H, 7.7 Hz) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  = 172.8, 140.5, 137.8, 128.9, 128.5, 128.3, 126.6, 126.2, 64.9, 35.9, 35.1, 30.9 ppm; IR (KBr):  $\nu$  = 3061(w), 3030(w), 2959 (w), 2936 (w), 2895 (w), 2868 (w), 1721(s), 1602 (w), 1491 (m), 1471 (m), 1452 (m), 1417 (m), 1390 (s), 1362 (m), 1291 (m), 1164, 1078 (m), 1048 (m), 1027 (m), 1000 (m), 970 (m), 939 (m), 910(m), 782 (m), 746 (s), 698 (s) cm<sup>-1</sup>; GC-MS (EI 70 eV): m/z = 133 (C<sub>9</sub>H<sub>9</sub>O)<sup>+</sup>(2), 104 (C<sub>8</sub>H<sub>8</sub>)<sup>+</sup>(100), 91 (C<sub>7</sub>H<sub>7</sub>)<sup>+</sup>(30), 77 (C<sub>6</sub>H<sub>5</sub>)<sup>+</sup>(10), 65 (C<sub>5</sub>H<sub>5</sub>)<sup>+</sup>(8), 51 (C<sub>4</sub>H<sub>3</sub>)<sup>+</sup>(5).

#### **Hippuric acid 2-phenylethyl ester (59)**<sup>6</sup>



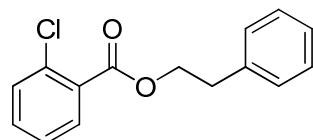
Ester (**59**) was prepared acc. to GP-II and obtained as a colorless crystals (yield: 82%);  $R_f$  0.19 (i-hexane/ethyl acetate 3:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 7.80-7.78 (m, 2H), 7.52-7.49 (m, 1H), 7.44-7.41 (m, 2H), 7.31-7.28 (m, 2H), 7.25-7.20 (m, 3H), 6.74 (s, 1H), 4.39 (t, 2H,  $J$  = 6.9 Hz), 4.20 (d, 2H,  $J$  = 5.5 Hz), 2.97 (t, 2H,  $J$  = 6.9 Hz) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  = 169.7, 167.4, 137.3, 133.7, 131.8, 128.9, 128.6, 127.1, 126.7, 66.0, 41.9, 34.3 ppm; IR (KBr):  $\nu$  = 3317(m), 3061 (w), 2933 (w), 2850 (w), 1734 (s), 1621 (m), 1577 (m), 1536 (s) 1488 (m), 1408 (w), 1355 (m), 1310 (m), 1253 (m), 1197 (s), 1075 (m) 977 (m), 741 (m), 699 (s), 599 (m) cm<sup>-1</sup>; HRMS (ESI C<sub>17</sub>H<sub>17</sub>NO<sub>3</sub>+Na): calcd. 306,1101; found: 306,1106.

#### **Benzoic acid 2-phenylethyl ester (60)**<sup>6</sup>



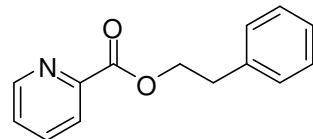
Ester **60** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 84%);  $R_f$  0.69 (i-hexane/diethyl ether 1:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>):  $\delta$  = 8.02-8.00 (m, 2H), 7.56-7.53 (m, 1H), 7.45-7.41 (m, 2H), 7.34-7.28 (m, 4H), 7.26-7.22 (m, 1H), 4.53 (t, 2H,  $J$  = 6.9 Hz), 3.08 (t, 2H,  $J$  = 6.9 Hz) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>):  $\delta$  = 166.6, 137.9, 132.9, 130.3, 129.6, 129.0, 128.6, 128.4, 126.6, 65.5, 35.3 ppm; IR (film):  $\nu$  = 3063 (w), 3029 (w), 2956 (w), 1714 (s), 1602 (w) 1583 (w), 1496 (w), 1452 (m), 1382 (w), 1313 (m), 1268 (s), 1175 (m), 1109 (s), 1069 (m), 1026 (m), 749 (m) cm<sup>-1</sup>; MS (ESI): m/z 249 (M+Na)<sup>+</sup>.

**2-Chloro-benzoic acid -2phenylethyl ester (61)<sup>18</sup>**



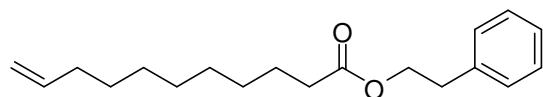
Ester **61** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 84%);  $R_f$  0.59 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.76-7.73 (m, 1H), 7.45-7.38 (m, 2H), 7.34-7.23 (m, 6H), 4.56 (t, 2H,  $J$  = 7.0 Hz), 3.09 (t, 2H,  $J$  = 6.9 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 165.6, 137.7, 133.8, 132.5, 131.4, 131.1, 130.2, 129.0, 128.6, 126.7, 126.5, 66.0, 35.1 ppm; IR (KBr):  $\nu$  = 3064 (w), 3028 (w), 2856 (w), 1726 (s), 1592 (m), 1496 (w), 1472 (m), 1454 (m), 1435 (m), 1380 (w), 1289 (s), 1246 (s), 1198 (w), 1162 (w), 1115 (s), 1086 (w), 1048 (s), 989 (w), 964 (m), 908 (w), 866 (w), 834 (w), 789 (w), 743 (s), 696 (s), 649 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 139 ( $\text{C}_7\text{H}_4\text{ClO}$ )<sup>+</sup>(35), 111 ( $\text{C}_6\text{H}_4\text{Cl}$ )<sup>+</sup>(20), 104 ( $\text{C}_8\text{H}_8$ )<sup>+</sup>(100), 91 ( $\text{C}_7\text{H}_7$ )<sup>+</sup>(10), 65 ( $\text{C}_5\text{H}_5$ )<sup>+</sup>(5), 51 ( $\text{C}_4\text{H}_3$ )<sup>+</sup>(5).

**Picolinic acid 2-phenylethyl ester (62)<sup>19</sup>**



Ester **62** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 82%);  $R_f$  0.14 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 8.81-8.79 (m, 1H), 8.13-8.11 (m, 1H), 7.90-7.87 (m, 1H), 7.54-7.51 (m, 1H), 7.33-7.22 (m, 5H), 4.63 (t, 2H,  $J$  = 7.5 Hz), 3.15 (t, 2H,  $J$  = 7.4 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 164.7, 149.6, 147.6, 137.6, 137.4, 129.0, 128.6, 127.1, 126.7, 125.3, 66.5, 35.1 ppm; IR (KBr):  $\nu$  = 3060 (w), 3028 (w), 2956 (w), 1737 (m), 1715 (s), 1583 (m), 1572 (w), 1497 (w), 1454 (m), 1436 (m), 1383 (w), 1303 (s), 1279 (s), 1242 (s), 1126 (s), 1086 (m), 1044 (m), 1030 (w), 992 (m), 968 (m), 909 (w), 821 (w), 744 (s), 697 (s), 618 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 106 ( $\text{C}_6\text{H}_4\text{NO}$ )<sup>+</sup> (15), 104 ( $\text{C}_8\text{H}_8$ )<sup>+</sup>(100), 91 ( $\text{C}_7\text{H}_7$ )<sup>+</sup>(10), 78 ( $\text{C}_5\text{H}_4\text{N}$ )<sup>+</sup>(50), 65 ( $\text{C}_5\text{H}_5$ )<sup>+</sup>(5), 51 ( $\text{C}_4\text{H}_3$ )<sup>+</sup>(15).

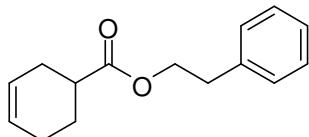
**10-Undecenic acid 2-phenylethylester(63)**



Ester **63** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 83%);  $R_f$  0.55 (i-hexane/diethyl ether 10:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.32-7.28 (m, 2H), 7.24-7.21 (m, 3H), 5.81 (tdd, 1H,  $J$  = 13.5, 10.2, 6.7 Hz), 5.01-4.97 (m, 1H), 4.95-4.91 (m, 1H), 4.29 (t, 2H,  $J$  = 7.1 Hz), 2.94 (t, 2H,  $J$  = 7.1 Hz), 2.28 (t, 2H,  $J$  = 7.6 Hz), 2.06-2.01 (m, 2H), 1.61-1.56 (m, 2H), 1.39-1.34 (m, 2H), 1.30-1.25 (m, 8H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 173.8, 139.2, 137.9, 128.9, 128.5, 126.5, 114.2,

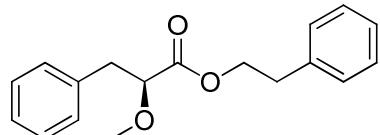
64.7, 35.2, 34.3, 33.8, 29.3, 29.2, 29.1, 29.0, 28.9, 24.9 ppm; IR (KBr):  $\nu$  = 3065 (w), 3029 (w), 2925 (m), 2854 (m), 1734 (s), 1640 (w), 1497 (w), 1454 (w), 1387 (w), 1352 (w), 1237 (w), 1164 (m), 1114 (w), 1087 (w), 995 (m), 908 (m), 732 (m), 697 (s)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 288 ( $M^+$ )<sup>(1)</sup>, 167 ( $C_{11}H_{19}O$ )<sup>(1)</sup>, 121 ( $C_8H_9O$ )<sup>(1)</sup>, 104 ( $C_8H_8$ )<sup>(100)</sup>, 91 ( $C_7H_7$ )<sup>(5)</sup>, 77 ( $C_6H_5$ )<sup>(5)</sup>, 55 ( $C_4H_7$ )<sup>(10)</sup>. HRMS (ESI  $C_{19}H_{28}O_2+Na$ ) calcd.: 311.1982; found: 311.1982.

### Cyclohex-3-enic acid 2-phenylethylester(64)



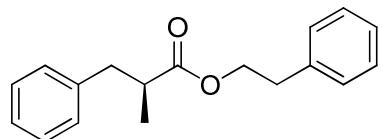
Ester **64** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 86%);  $R_f$  0.51 (i-hexane/diethyl ether 10:1); <sup>1</sup>H-NMR (500 MHz,  $CDCl_3$ ):  $\delta$  = 7.32-7.28 (m, 2H), 7.25-7.21 (m, 3H), 5.68-5.66 (m, 2H), 4.31 (t, 2H,  $J$  = 6.9 Hz), 2.94 (t, 2H,  $J$  = 7.0 Hz), 2.56-2.50 (m, 1H), 2.23-2.20 (m, 2H), 2.09-2.04 (m, 2H), 1.99-1.93 (m, 1H), 1.69-1.62 (m, 1H) ppm; <sup>13</sup>C-NMR (125 MHz,  $CDCl_3$ ):  $\delta$  = 175.8, 137.9, 128.9, 128.5, 126.7, 126.5, 125.2, 64.8, 39.3, 35.2, 27.4, 25.0, 24.4 ppm; IR (KBr):  $\nu$  = 3063 (w), 3027 (w), 2924 (w), 2840 (w), 1728 (s), 1652 (w), 1604 (w), 1497 (w); 1454 (m), 1437 (w), 1389 (w), 1304 (m), 1261 (w), 1221 (m), 1159 (s), 1087 (w), 1063 (w), 1012 (m), 917 (w), 746 (m), 731 (m), 697 (s), 648 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 230 ( $M^+$ )<sup>(1)</sup>, 184 ( $M-C_2H_6O$ )<sup>(2)</sup>, 105 ( $C_8H_9$ )<sup>(100)</sup>, 91 ( $C_7H_7$ )<sup>(15)</sup>, 79 ( $C_6H_7$ )<sup>(25)</sup>, 65 ( $C_5H_5$ )<sup>(10)</sup>, 53 ( $C_4H_5$ )<sup>(10)</sup>. HRMS (ESI  $C_{15}H_{18}O_2+Na$ ) calcd.: 253.1199; found: 253.1208.

### 2S-Methoxy 3-phenylpropionic acid 2-phenylethyl ester (65)



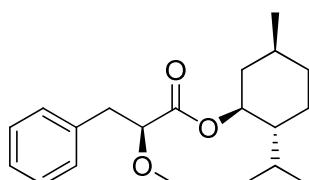
Ester **65** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 72%);  $[\alpha]_d^{20}$  ( $c=1$ , acetone)-21.7;  $R_f$  0.43 (i-hexane/diethyl ether 2:1); <sup>1</sup>H-NMR (500 MHz,  $CDCl_3$ ):  $\delta$  = 7.31-7.16 (m, 10H), 4.33 (t, 2H,  $J$  = 6.9 Hz), 3.93 (dd, 1H,  $J$  = 6.9, 5.7 Hz), 3.29 (s, 3H), 2.98-2.95 (m, 2H), 2.90 (t, 2H,  $J$  = 7.0 Hz) ppm; <sup>13</sup>C-NMR (125 MHz,  $CDCl_3$ ):  $\delta$  = 172.0, 137.5, 137.0, 129.3, 128.9, 128.5, 128.3, 126.7, 81.7, 65.3, 58.3, 39.1, 35.0 ppm; IR (KBr):  $\nu$  = 3063 (w), 3028 (w), 2929 (w), 2828 (w), 1745 (s), 1604 (w), 1496 (m), 1454 (m), 1353 (w), 1273 (m), 1176 (s), 1115 (s), 1079 (m), 1051 (w), 1016 (m), 908 (w), 832 (w), 744 (s), 696 (s)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 252 ( $M-CH_4O$ )<sup>(1)</sup>, 206 ( $C_{12}H_{14}O_3$ )<sup>(2)</sup>, 148 ( $C_9H_8O_2$ )<sup>(3)</sup>, 135 ( $C_9H_{11}O$ )<sup>(100)</sup>, 104 ( $C_8H_8$ )<sup>(100)</sup>, 91 ( $C_7H_7$ )<sup>(50)</sup>, 77 ( $C_6H_5$ )<sup>(20)</sup>, 65 ( $C_5H_5$ )<sup>(15)</sup>, 51 ( $C_4H_3$ )<sup>(10)</sup>; HRMS (ESI  $C_{18}H_{20}O_3+Na$ ) calcd.: 307.1305; found: 307.1308.

### 2S-Methyl 3-phenylpropionic acid 2-phenylethyl ester (66)



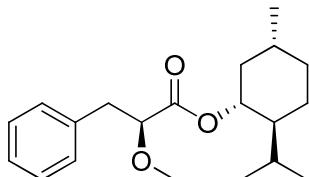
Ester **66** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 69%);  $[\alpha]_d^{20}$  ( $c=1$ , acetone) -22.9;  $R_f$  0.71 (i-hexane/diethyl ether 2:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.30-7.19 (m, 6H), 7.18-7.16 (m, 2H), 7.13-7.11 (m, 2H), 4.28-4.23 (m, 2H), 3.00-2.96 (m, 1H), 2.86 (t, 2H,  $J$  = 7.0 Hz), 2.70 (tq, 1H,  $J$  = 7.1 Hz), 2.66-2.61 (m, 1H), 1.12 (d, 3H,  $J$  = 7.1 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 176.0, 139.4, 137.9, 129.0, 120.9, 128.5, 128.4, 126.5, 126.3, 64.8, 41.5, 39.7, 35.1, 16.8 ppm; IR (KBr):  $\nu$  = 3028 (w), 2971 (w), 1729 (s), 1603 (w), 1495 (m), 1453 (m), 1385 (w), 1353 (w), 1280 (w), 1248 (m), 1161 (s), 1116 (m), 1087 (8m), 1030 (m), 982 (w), 909 (w), 843 (w), 743 (s9; 696 (s)  $\text{cm}^{-1}$ ; MS (APCI):  $m/z$  = 269 ( $M+\text{H})^+(1)$ , 251 ( $M-\text{H}_2\text{O})^+(40)$ , 105 ( $\text{C}_8\text{H}_9)^+(100)$ ; HRMS (APCI  $\text{C}_{18}\text{H}_{20}\text{O}_2+\text{H}$ ) calcd.: 269.1536; found: 269.1535.

### **2S-Methoxy 3-phenyl propionic acid L-menthyl ester (67a)**



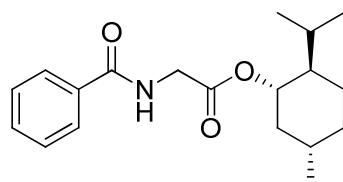
Ester **67a** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 69%);  $[\alpha]_d^{20}$  ( $c=1$ , acetone) -50.0;  $R_f$  0.68 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.29-7.20 (m, 5H), 4.76 (dt, 1H,  $J$  = 10.9, 4.5Hz), 3.94 (t, 1H,  $J$  = 6.8 Hz), 3.34 (s, 3H), 3.01 (d, 2H,  $J$  = 6.8 Hz), 1.86-1.77 (m, 2H), 1.70-1.64 (m, 2H), 1.51-1.43 (m, 1H), 1.41-1.36 (m, 1H), 1.09-0.99 (m, 1H), 0.91-0.82 (m, 8H), 0.75 (d, 3H,  $J$  = 7.0 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 171.8, 137.0, 129.4, 128.3, 126.7, 82.0, 74.9, 58.1, 46.8, 40.7, 39.2, 34.2, 31.4, 26.2, 23.3, 22.0, 20.8, 16.2 ppm; IR (KBr):  $\nu$  = 3029 (w), 2953 (m), 2925 (m), 2869 (m), 1740 (s), 1604 (w), 1495 (w), 1254 (m), 1369 (w), 1274 (m), 1190 (s), 1151 (m), 1116 (s), 1079 (m), 1012 (m), 983 (m), 960 (m), 913 (w), 843 (w), 742 (m), 697 (s)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV):  $m/z$  = 318 ( $M)^+(1)$ , 286 ( $M-\text{CH}_4\text{O})^+(20)$ , 138 ( $\text{C}_{10}\text{H}_{18})^+(30)$ , 135 ( $\text{C}_9\text{H}_{11}\text{O})^+(100)$ , 103 ( $\text{C}_8\text{H}_7)^+(10)$ , 91 ( $\text{C}_7\text{H}_7)^+(10)$ , 55 ( $\text{C}_4\text{H}_9)^+(10)$ ; HRMS (ESI  $\text{C}_{20}\text{H}_{30}\text{O}_3+\text{Na}$ ) calcd.: 341.2087; found 341.2085.

### **2S-Methoxy 3-phenyl propionic acid D-menthyl ester (67b)**



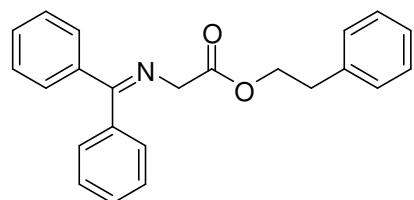
Ester **67b** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 69%)[ $\alpha$ ]<sub>d</sub><sup>20</sup> (c=1, acetone) +30.1;; R<sub>f</sub> 0.68 (i-hexane/diethyl ether 3:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.30-7.20 (m, 5H), 4.72 (dt, 1H, J = 11.1, 4.5 Hz), 3.95 (dd, 1H, J = 7.7, 5.4 Hz), 3.35 (s, 3H), 3.06-2.97 (m, 2H), 2.00-1.97 (m, 1H), 1.71-1.62 (m, 3H), 1.53-1.45 (m, 1H), 1.42-1.36 (m, 1H), 1.07-0.94 (m, 2H), 0.91-0.82 (m, 7H), 0.68 (d, 3H, J = 6.9 Hz) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>): δ = 171.8, 137.0, 129.4, 128.3, 126.6, 82.1, 75.2, 58.2, 46.7, 40.9, 39.2, 34.2, 31.4, 25.8, 23.0, 22.0, 20.8, 15.8 ppm; IR (KBr): ν = 2953 (m), 2827 (m), 2869 (m), 1740 (s), 1454 (m), 1369 (w), 1274 (m), 1190 (s), 1114 (s), 1013 (m), 982 (m), 959 (m), 913 (w), 844 (w), 743 (m), 697 (s) cm<sup>-1</sup>; GC-MS (EI 70 eV): m/z = 318 (M)<sup>+</sup>(1), 286 (M-CH<sub>4</sub>O)<sup>+</sup>(20), 138 (C<sub>10</sub>H<sub>18</sub>)<sup>+</sup>(40), 135 (C<sub>9</sub>H<sub>11</sub>O)<sup>+</sup>(100), 83 (C<sub>5</sub>H<sub>7</sub>O)<sup>+</sup>(40); HRMS (ESI C<sub>20</sub>H<sub>30</sub>O<sub>3</sub>+Na) calcd.: 341.2087; found 341.2086.

### Hippuric acid L-menthylester (**73**)<sup>20</sup>



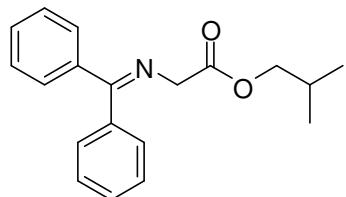
Ester **73** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 84%);[ $\alpha$ ]<sub>d</sub><sup>20</sup> (c=1, acetone) -42.5;; R<sub>f</sub> 0.41 (i-hexane/diethyl ether 1:1); <sup>1</sup>H-NMR (500 MHz, CDCl<sub>3</sub>): δ = 7.82-7.81 (m, 2H), 7.53-7.50 (m, 1H), 7.46-7.43 (m, 2H), 6.73 (s, 1H), 4.80 (dt, 1H, J = 11.0, 4.6 Hz), 4.22 (d, 2H, J = 4.8 Hz), 2.04-2.00 (m, 1H), 1089-1.83 (m, 1H), 1.74-1.68 (m, 2H), 1.54-1.39 (m, 2H), 1.11-1.01 (m, 2H), 0.93-0.85 (m, 7H), 0.77 (d, 3H, J = 6.9 Hz) ppm; <sup>13</sup>C-NMR (125 MHz, CDCl<sub>3</sub>): δ = 169.7, 167.4, 133.8, 131.7, 128.6, 127.0, 75.9, 46.9, 42.0, 40.8, 34.1, 31.4, 26.3, 23.4, 22.0, 20.7, 16.4 ppm; IR (KBr): ν = 3307 (m), 2958 (m), 2946 (m), 2924 (m), 2857 (m), 1962 (w), 1722 (s), 1638 (m), 1604 (w), 1579 (w), 1531 (s), 1489 (w), 1457 (w), 1422 (w), 1326 (m), 12.76 (m), 1203 (s), 1176 (w), 1162 (w), 1096 (w), 1072 (w), 1036 (w), 1014 (m), 981 (m), 907 (w), 851 (w), 802 (w), 747 (w), 717 (m), 639 (m) cm<sup>-1</sup>; GC-MS (EI 70 eV): m/z = 180 (C<sub>9</sub>H<sub>10</sub>NO<sub>3</sub>)<sup>+</sup>(25), 162 (C<sub>9</sub>H<sub>8</sub>NO<sub>2</sub>)<sup>+</sup>(5), 135 (C<sub>8</sub>H<sub>9</sub>NO)<sup>+</sup>(80), 123 (C<sub>9</sub>H<sub>15</sub>)<sup>+</sup>(5), 105 (C<sub>7</sub>H<sub>5</sub>O)<sup>+</sup>(100), 97 (C<sub>7</sub>H<sub>13</sub>)<sup>+</sup>(10), 83 (C<sub>6</sub>H<sub>11</sub>)<sup>+</sup>(80), 77 (C<sub>6</sub>H<sub>5</sub>)<sup>+</sup>(50), 69 (C<sub>5</sub>H<sub>9</sub>)<sup>+</sup>(20), 55 (C<sub>4</sub>H<sub>7</sub>)<sup>+</sup>(30).

### Benzophenone imine glycine 2-phenylethyl ester (**74**)



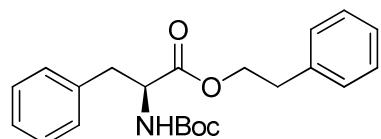
Ester **74** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 96%);  $R_f$  0.50 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.66-7.64 (m, 2H), 7.46-7.38 (m, 4H), 7.35-7.32 (m, 2H), 7.26-7.18 (m, 5H), 7.13-7.10 (m, 2H), 4.36 (t, 2H,  $J$  = 7.0 Hz), 4.30 (s, 2H), 2.95 (t, 2H,  $J$  = 7.0 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 171.9, 170.6, 139.3, 137.7, 136.0, 130.5, 128.9, 128.8, 128.7, 128.6, 128.5, 128.1, 127.6, 126.5, 65.3, 55.7, 35.1 ppm; (KBr):  $\nu$  = 3026 (w), 2954 (w), 1735 (s), 1624 (m), 1575 (w), 1494 (w), 1445 (m), 1385 (w), 1341 (w), 1314 (w), 1289 (m), 1266 (m), 1170 (s), 1054 (m), 1028 (m), 999 (m), 907 (m), 847 (w), 780 (m), 731 (m), 693 (s), 652 (m)  $\text{cm}^{-1}$ ; (APCI): m/z = 344 ( $\text{M+H}^+$ ) (100), 254 ( $\text{C}_{16}\text{H}_{16}\text{NO}_2$ ) (10), 240 ( $\text{C}_{15}\text{H}_{14}\text{NO}_2$ ), 194 ( $\text{C}_{14}\text{H}_{12}\text{N}$ ) $^+$ , 105 ( $\text{C}_8\text{H}_9$ ) $^+$ . HRMS (APCI)  $\text{C}_{23}\text{H}_{21}\text{NO}_2+\text{H}$  calcd.: 344.1645; found: 344.1644.

#### Benzophenone imine glycine isobutyl ester (75)



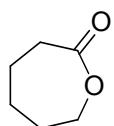
Ester **75** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 84%);  $R_f$  0.48 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.67-7.64 (m, 2H), 7.49-7.38 (m, 4H), 7.35-7.31 (m, 2H), 7.20-7.18 (m, 2H), 4.22 (s, 2H), 3.93 (d, 2H,  $J$  = 6.9 Hz), 1.99-1.91 (m, 1H), 0.93 (d, 6H,  $J$  = 6.3 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 171.8, 170.8, 139.4, 136.1, 130.4, 128.8, 128.7, 128.6, 128.1, 127.7, 70.1, 55.7, 27.7, 19.1 ppm; (KBr):  $\nu$  = 3058 (w), 2960 (m), 2874 (w), 1737 (s), 1625 (m), 1576 (w), 1490 (w), 1469 (m), 1445 (m), 1377 (m), 1339 (w), 1314 (w), 1288 (m), 1173 (s), 1074 (w), 1054 (w), 1028 (m), 1000 (m), 941 (w), 908 (w), 779 (m), 693 (s), 652 (m)  $\text{cm}^{-1}$ ; (EI 70 eV): m/z = 295 ( $\text{M}^+$ ) (15), 194 ( $\text{M-C}_5\text{H}_9\text{O}_2$ ) $^+$  (90), 91 ( $\text{C}_7\text{H}_7$ ) $^+$  (100); APCI  $\text{C}_{19}\text{H}_{21}\text{NO}_2+\text{H}$  calcd.: 296.1645; found: 296.1645.

#### 2S-tert-Butoxycarbonylamino 3-phenyl propionic acid 2- phenylethyl ester (76)



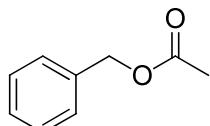
Ester **76** was prepared acc. to GP-II and obtained as a colorless crystals (yield: 98%);  $[\alpha]_d^{20}$  ( $c=1$ , acetone) + 3.5;  $R_f$  0.54 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.32\text{-}7.28$  (m, 2H), 7.27-7.21 (m, 4H), 7.20-7.18 (m, 2H), 7.03-7.00 (m, 2H), 4.96-4.92 (m, 1H), 4.58-4.54 (m, 1H), 4.30 (t, 2H,  $J = 7.0$  Hz), 3.07-2.96 (m, 2H), 2.89 (t, 2H,  $J = 7.0$  Hz), 1.41 (s, 9H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta = 171.8, 155.1, 137.5, 136.0, 129.3, 128.9, 128.6, 128.5, 127.0, 126.7, 79.9, 65.8, 54.5, 38.3, 35.0, 28.3$  ppm; IR (KBr):  $\nu = 3383$  (m), 3026 (w), 2976 (w), 1731 (m), 1689 (s), 1603 (w), 1521 (m), 1444 (m), 1389 (w), 1366 (m), 1268 (m), 1249 (m), 121 (m), 1158 (s), 1082 (w), 1057 (m), 1028 (m), 991 (m), 887 (w), 857 (w), 830 w, 750 (s), 699 (s), 658 (m)  $\text{cm}^{-1}$ ; MS (ESI):  $m/z = 392$  ( $\text{M+Na}^+$ ) (100), 336 ( $\text{M-C}_4\text{H}_8+\text{Na}^+$ ) (10), 292 ( $\text{C}_{17}\text{H}_{19}\text{NO}_2\text{Na}^+$ ) (10), 270 ( $\text{C}_{17}\text{H}_{20}\text{NO}_2$ ) (10), 105 ( $\text{C}_8\text{H}_9$ ) (5); HRMS (ESI  $\text{C}_{22}\text{H}_{27}\text{NO}_4+\text{Na}$ ) calcd.: 392.1832; found: 392.1844.

#### **$\epsilon$ -Caprolactone (78)<sup>21</sup>**



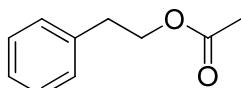
Lactone **78** was prepared acc to GP-II except using THF (conc. 0.5 mmol/ml) as solvent and obtained as a colorless liquid (yield: 75 %);  $R_f$  0.41 (i-hexane/ethyl acetate 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta = 4.24\text{-}4.22$  (m, 2H), 2.66-2.63 (m, 2H), 1.89-1.85 (m, 2H), 1.80-1.74 (m, 4H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta = 176.2, 65.3, 34.6, 29.3, 29.1, 23.0$  ppm; IR (KBr):  $\nu = 2933$  (m), 1722 (s), 1437 (w), 1391 (w), 1347 (m), 1327 (w), 1289 (m), 1250 (m), 1224 (w), 1162 (s), 1086 (m), 1051 (s), 1014 (m), 986 (m), 961 (m), 848 (m), 694 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV):  $m/z = 114$  ( $\text{M}^+$ ) (20), 84 ( $\text{C}_5\text{H}_8\text{O}^+$ ) (30), 70 ( $\text{C}_4\text{H}_6\text{O}^+$ ) (20), 55 ( $\text{C}_4\text{H}_7$ ) (100).

#### **Acetic acid benzyl ester (79)<sup>6</sup>**



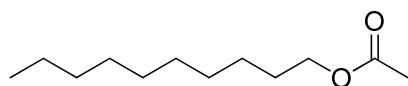
Ester **79** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.69 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta = 7.39\text{-}7.31$  (m, 5 H), 5.10 (s, 2 H), 2.11 (s, 3 H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta = 170.9, 136.0, 128.6, 128.3, 66.3, 21.0$  ppm; IR (film):  $\nu = 3034$  (w), 2954 (w), 1734 (s), 1455 (w), 1379 (m), 1362 (m), 1231 (s), 1024 (m), 735 (m), 695 (m)  $\text{cm}^{-1}$ ; MS (EI, 70 eV):  $m/z$  150 ( $\text{M}^+$ ) (60), 108 ( $\text{M-C}_2\text{H}_2\text{O}^+$ ) (100), 91 ( $\text{C}_7\text{H}_7$ ) (60), 79 ( $\text{C}_6\text{H}_7$ ) (18), 77 ( $\text{C}_6\text{H}_5$ ) (15), 43 ( $\text{C}_2\text{H}_3\text{O}^+$ ) (25).

#### **Acetic acid 2-phenylethyl ester (84)<sup>6</sup>**



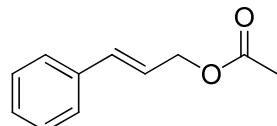
Ester **84** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.89 (i-hexane/ethyl acetate, 1:1);  $^1\text{H-NMR}$  (400 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.31-7.28 (m, 2H), 7.24-7.20 (m, 3H), 4.27 (t, 2H,  $J$  = 7.0 Hz), 2.93 (t, 2H,  $J$  = 7.0 Hz), 2.02 (s, 3H) ppm;  $^{13}\text{C-NMR}$  (100 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 171.0, 137.8, 128.9, 128.5, 126.6, 64.9, 35.1, 21.0 ppm; IR (film):  $\nu$  3029 (w), 2958 (w), 1738 (s), 1454 (w), 1364 (w), 1237 (s), 1031 (m), 700 (m)  $\text{cm}^{-1}$ ; MS (Cl,  $\text{CH}_4$ ): m/z 165 ( $M+\text{H}$ ) $_+$  (8), 149 ( $M-\text{CH}_3$ ) $_+$  (3), 119 ( $M-\text{C}_2\text{H}_5\text{O}$ ) $_+$  (5), 104 ( $M-\text{C}_2\text{H}_4\text{O}_2$ ) $_+$  (100), 91 ( $\text{C}_7\text{H}_7$ ) $_+$  (10), 77 ( $\text{C}_6\text{H}_5$ ) $_+$ , 43 ( $\text{C}_2\text{H}_3\text{O}$ ) $_+$  (12).

#### Acetic acid decylester (**85**)<sup>22</sup>



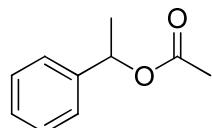
Ester **85** was prepared acc. to GP-II and obtained as a colorless liquid ;  $R_f$  0.70(i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 4.05 (t, 2 H,  $J$  = 6.8 Hz), 2.05 (s, 3H), 1.65-1.59 (m, 2H), 1.35-1.22 (m, 14 H), 0.88 (t, 3H,  $J$  = 6.9 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 171.3, 64.7, 31.9, 29.5, 29.3, 29.2, 28.6, 25.9, 22.7, 21.0, 14.1 ppm; IR (KBr):  $\nu$  = 2923 (m), 2854 (m), 1740 (s), 1466 (w), 1364 (m), 1231 (s), 1038 (m), 974 (w), 722 (w), 605 (w)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 140 ( $\text{C}_{10}\text{H}_{20}$ ) $_+$  (10), 116 ( $\text{C}_6\text{H}_{12}\text{O}_2$ ) $_+$  (10), 112 ( $\text{C}_8\text{H}_{16}$ ) $_+$  (40), 97 ( $\text{C}_7\text{H}_{13}$ ) $_+$  (50), 83 ( $\text{C}_6\text{H}_{11}$ ) $_+$  (60), 70 ( $\text{C}_5\text{H}_{10}$ ) $_+$  (85), 61( $\text{C}_2\text{H}_5\text{O}_2$ ) $_+$  (80), 55 ( $\text{C}_4\text{H}_7$ ) $_+$  (100).

#### Acetic acid 3-phenylallyl ester (**86**)<sup>6</sup>



Ester **86** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.65 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz $\text{CDCl}_3$ ):  $\delta$  = 7.42-7.25 (m, 5 H), 6.66 (d, 1 H,  $J$  = 16.0 Hz), 6.28 (dt, 1 H,  $J$  = 16.0 Hz, 6.5 Hz,), 4.73 (dd, 2 H,  $J$  = 6.5 Hz, 1.1 Hz), 2.11 (s, 3 H,  $\text{CH}_3$ ) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 170.9, 136.2, 134.2, 128.6, 128.1, 126.6, 123.2, 65.1, 21.0 ppm; IR (film):  $\nu$  3027 (w), 2942 (w), 1733 (s), 1494 (w), 1449 (m), 1379 (m), 1361 (m), 1223 (s), 1113 (w), 1068 (w), 1023 (m), 963 (m), 744 (m), 691 (m)  $\text{cm}^{-1}$ ; MS (EI, 70 eV): m/z 176 ( $M$ ) $_+$  (80), 134 ( $M-\text{C}_2\text{H}_2\text{O}$ ) $_+$  (80), 115 ( $\text{C}_9\text{H}_7$ ) $_+$  (100), 105 ( $\text{C}_8\text{H}_9$ ) $_+$  (50), 92 ( $\text{C}_7\text{H}_8$ ) $_+$  (40), 77 ( $\text{C}_6\text{H}_5$ ) $_+$  (15), 51 ( $\text{C}_4\text{H}_3$ ) $_+$  (8), 43 ( $\text{C}_2\text{H}_3\text{O}$ ) $_+$  (85).

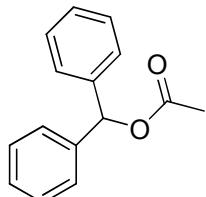
#### Acetic acid 1-phenylethyl ester (**87**)<sup>6</sup>



Ester **87** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.72 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.36-7.33 (m, 4 H), 7.31-7.27 (m, 1 H ), 5.88 (q, 1 H,  $J$  = 6.6 Hz), 2.07 (s, 3H), 1.53 (d, 3 H,  $J$  = 6.6 Hz) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 170.3, 141.7, 128.5, 127.9, 126.1, 72.3, 22.2, 21.4 ppm; IR (film):  $\nu$  = 3034 (w), 2981 (w), 1726 (s), 1451 (w), 1370 (m),

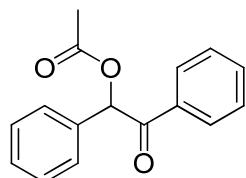
1232 (s), 1208 (m), 1963 (m), 1028 (m), 942 (m), 759 (m), 697 (s)  $\text{cm}^{-1}$ ; MS (EI, 70 eV): m/z 164 ( $M^+$ ) (50), 122 ( $M-\text{C}_2\text{H}_2\text{O}^+$ ) (85), 104 ( $\text{C}_8\text{H}_8^+$ ) (100), 77 ( $\text{C}_6\text{H}_5^+$ ) (35), 43 ( $\text{C}_2\text{H}_3\text{O}^+$ ) (55).

#### **Acetic acid diphenylmethyl ester (88)<sup>23</sup>**



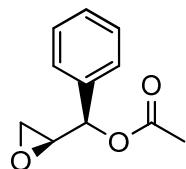
Ester **88** was prepared acc. to GP-II and obtained as a colorless crystals;  $R_f$  0.57(i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.35-7.32 (m, 8H), 7.30-7.27 (m, 2H), 6.88 (s, 1H), 2.16 (s, 3H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 170.0, 140.2, 128.5, 127.9, 127.1, 76.9, 21.3 ppm; IR (KBr):  $\nu$  = 3063 (w), 3031 (w), 2936 (w), 1736 (s), 1601 (w), 1586 (w), 1494 (m), 1452 (m), 1369 (m), 1225 (s), 1184 (m), 1080 (w), 1019 (m), 971 (m), 908 (m), 861 (w), 741 (m), 694 (s), 647 (m), 606 (m) 547 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 226 ( $M^+$ ) (15), 184 ( $M-\text{C}_2\text{H}_2\text{O}^+$ ) (25), 165 ( $\text{C}_{13}\text{H}_9^+$ ) (100), 152 ( $\text{C}_{12}\text{H}_8^+$ ) (15), 105 ( $\text{C}_7\text{H}_5\text{O}^+$ ) (20), 77 ( $\text{C}_6\text{H}_5^+$ ) (30) 51 ( $\text{C}_4\text{H}_3^+$ ) (10).

#### **O-Acetyl benzoin (89)<sup>24</sup>**



Ester **89** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.23(i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.94-7.92 (m, 2H), 7.52-7.45 (m, 3H), 7.41-7.32 (m, 5H), 6.87 (s, 1H), 2.21 (s, 3H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 193.7, 170.5, 134.6, 133.6, 133.5, 129.4, 129.2, 128.8, 128.7, 128.6, 77.7, 20.8 ppm; IR (KBr):  $\nu$  = 3064 (w), 3032 (w), 297 (w), 1724 (m), 1688 (s), 1595 (m), 1578 (m), 1493 (w), 1448 (m), 1371 (m), 1264 (m), 1222 (s), 1179 (m), 1079 (w), 1046 (m), 1001 (m), 964 (m), 928 (m), 859 (m), 752 (m), 693 (s), 637 (m), 583 (m)  $\text{cm}^{-1}$ ; GC-MS (EI 70 eV): m/z = 254 ( $M^+$ ) (<1), 149 ( $\text{C}_9\text{H}_9\text{O}_2$ ) (15), 105 ( $\text{C}_7\text{H}_5\text{O}^+$ ) (100), 77 ( $\text{C}_6\text{H}_5^+$ ) (30), 51 ( $\text{C}_4\text{H}_3^+$ ) (5).

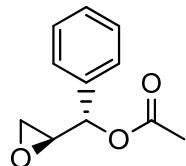
#### **Acetic acid syn2,3-epoxy 1-phenyl propyl ester (90a)<sup>25</sup>**



Ester **90a** was prepared acc. to GP-II and obtained as a colorless liquid (together with anti-Diastereomer);  $R_f$  0.30 (i-hexane/diethyl ether 3:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.40-7.05 (m, 5H), 5.85 (d, 1H,  $J$  = 3.7 Hz), 3.27-3.25 (m, 1H), 2.78 (dd, 1H,  $J$  = 5.5, 4.2 Hz), 2.67 (dd, 1H,  $J$  = 5.5, 2.5

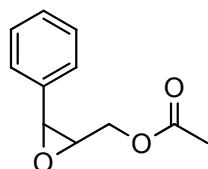
Hz), 2.10 (s, 3H) ppm;  $^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 169.8, 136.1, 128.7, 128.6, 127.4, 73.9, 52.8, 44.8, 21.0 ppm; IR (NaCl):  $\nu$  = 3065 (w), 3034 (w), 2999 (w), 2929 (w), 2552 (w), 2367 (w), 2174 (w), 1973 (w), 1736 (s), 1587 (w), 1496 (w), 1455 (w), 1370 (m), 1225 (s), 1201 (m), 1137 (w), 1079 (w), 1055 (w), 1021 (m), 956 (w), 904 (w), 866 (m), 831 (m), 748 (m), 698 (s), 672 (w), 606 (w), 552 (m), 530 (w)  $\text{cm}^{-1}$ ; MS (EI, 70 eV): m/z = 162 ( $\text{M-C}_2\text{H}_6^+$ ) (8), 150 ( $\text{M-C}_2\text{H}_2\text{O}^+$ ) (7), 132 ( $\text{M-C}_2\text{H}_4\text{O}_2^+$ ) (7), 120 ( $\text{M-C}_4\text{H}_8\text{O}^+$ ) (62), 101 ( $\text{M-C}_7\text{H}_7^+$ ) (20), 91 ( $\text{C}_7\text{H}_7^+$ ), 77 ( $\text{C}_6\text{H}_5^+$ ) (20), 51 ( $\text{C}_4\text{H}_3^+$ ) (16).

#### **Acetic acid anti2,3-epoxy 1-phenyl propyl ester (90b)<sup>25</sup>**



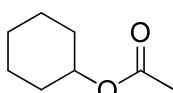
Ester **90b** was prepared acc. to GP-II and obtained as a colorless liquid (together with syn Diastereomer);  $R_f$  0.30 (i-hexane/diethyl ether 3:1);  $^1\text{H}$ -NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.46-7.21 (m, 5H), 5.50 (d, 1H,  $J$  = 6.7 Hz), 3.34-3.31 (m, 1H), 2.82 (dd, 1H,  $J$  = 4.7 Hz), 2.67 (dd, 1H,  $J$  = 5.0, 2.7 Hz), 2.09 (s, 3H) ppm;  $^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 169.9, 136.7, 128.7, 128.6, 126.9, 76.7, 53.7, 45.1, 21.1 ppm; IR (NaCl):  $\nu$  = 2998 (w), 2931 (w), 2364 (w), 2176 (w), 1963 (w), 1736 (s), 1586 (w), 1495 (w), 1454 (w), 1370 (m), 1226 (s), 1136 (w), 1077 (w), 1022 (m), 976 (w), 940 (w), 902 (w), 870 (m), 842 (m), 810 (w), 751 (m), 699 (s), 670 (w), 630 (w), 548 (w)  $\text{cm}^{-1}$ ; MS (EI, 70 eV): m/z = 195 ( $\text{M}^+$ ) (4) 162 ( $\text{M-C}_2\text{H}_6^+$ ) (8), 150 ( $\text{M-C}_2\text{H}_2\text{O}^+$ ) (7), 132 ( $\text{M-C}_2\text{H}_4\text{O}_2^+$ ) (7), 120 ( $\text{M-C}_4\text{H}_8\text{O}^+$ ) (62), 101 ( $\text{M-C}_7\text{H}_7^+$ ) (20), 91 ( $\text{C}_7\text{H}_7^+$ ), 77 ( $\text{C}_6\text{H}_5^+$ ) (20), 51 ( $\text{C}_4\text{H}_3^+$ ) (16).

#### **Acetic acid 2,3-epoxy 3-phenyl propyl ester (91)<sup>26</sup>**



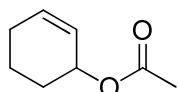
Ester **91** was prepared acc. to GP-II and obtained as a colorless liquid (yield: 82%);  $R_f$  0.43 (i-hexane/diethyl ether 3:1);  $^1\text{H}$ -NMR (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 7.33-7.18 (m, 5H), 4.40 (dd, 1H,  $J$  = 12.0, 3.2 Hz), 4.02 (dd, 1H,  $J$  = 12.1, 5.7 Hz), 3.73 (d, 1H,  $J$  = 2.0 Hz), 3.20-3.18 (m, 1H), 2.01 (s, 3H,  $\text{CH}_3$ ) ppm;  $^{13}\text{C}$ -NMR (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 169.7, 135.2, 127.9, 127.6, 124.7, 63.2, 58.2, 55.5, 14.3 ppm; IR (NaCl):  $\nu$  = 3419 (b), 2933 (b), 2368 (w), 2188 (w), 1964 (w), 1719 (s), 1495 (w), 1454 (w), 1371 (m), 1230 (s), 1038 (s), 1038 (s), 877 (w), 762 (w), 700 (m), 606 (w), 580 (w)  $\text{cm}^{-1}$ . GC-MS (EI 70 eV): m/z = 192 ( $\text{M}^+$ ) (5), 149 ( $\text{M-C}_2\text{H}_3\text{O}^+$ ) (65), 133 ( $\text{M-C}_2\text{H}_3\text{O}_2^+$ ) (25), 120 ( $\text{C}_8\text{H}_8\text{O}^+$ ) (10), 107 ( $\text{C}_7\text{H}_7\text{O}^+$ ) (100), 105 ( $\text{C}_7\text{H}_5\text{O}^+$ ) (90), 91 ( $\text{C}_7\text{H}_7^+$ ) (90), 86 ( $\text{C}_4\text{H}_6\text{O}_2^+$ ) (20), 77 ( $\text{C}_6\text{H}_5^+$ ) (55), 51 ( $\text{C}_4\text{H}_3^+$ ) (30)

#### **Acetic acid cyclohexanyl ester (92)<sup>6</sup>**



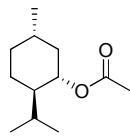
Ester **92** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.71 (i-hexane/diethyl ether 1:1);  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 4.76-4.71 (m, 1H), 2.03 (s, 3 H), 1.87-1.83 (m, 2 H), 1.75-1.70 (m, 2H), 1.57-1.53 (m, 1 H), 1.43-1.31 (m, 4 H) 1.28-1.21 (m, 1H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 170.6, 72.7, 31.7, 25.4, 23.8, 21.5 ppm; IR (film):  $\nu$  = 2935 (m), 2858 (w), 1732 (s), 1450(w), 1362 (m), 1231 (s), 1125 (w), 1043 (m), 1021(m), 967 (w), 905 (w) 607 (w) cm<sup>-1</sup>; MS (EI, 70 eV): m/z 142.1 (M)<sup>+</sup>(2), 127.1 (M-CH<sub>3</sub>)<sup>+</sup> (2), 99.1 (M-OC<sub>2</sub>H<sub>3</sub>)<sup>+</sup> (7), 82.1 (C<sub>6</sub>H<sub>10</sub>)<sup>+</sup> (100), 67.1 (C<sub>5</sub>H<sub>7</sub>)<sup>+</sup> (50), 54.1 (C<sub>4</sub>H<sub>6</sub>)<sup>+</sup> (15), 43.0 (C<sub>2</sub>H<sub>3</sub>O)<sup>+</sup> (75 ).

#### **Acetic acid cyclohexenyl ester (93)<sup>6</sup>**

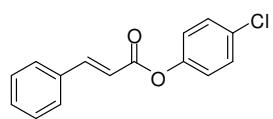
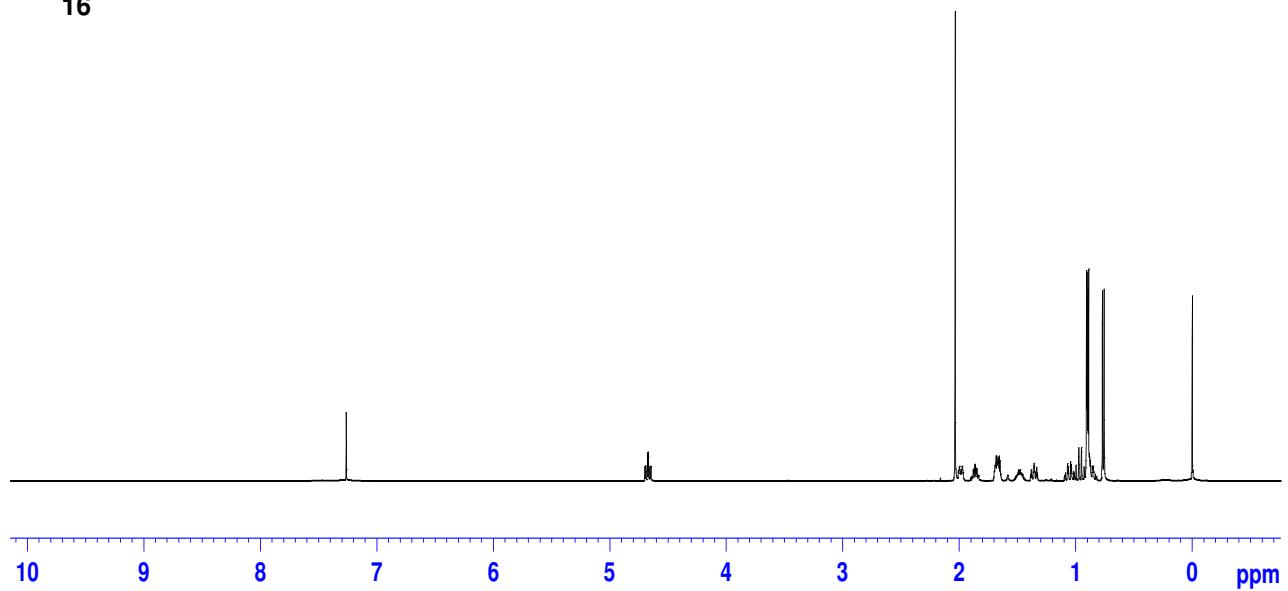


Ester **93** was prepared acc. to GP-II and obtained as a colorless liquid;  $R_f$  0.72  $^1\text{H-NMR}$  (500 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 5.98-5.94 (m, 1 H), 5.72-5.69 (m, 1 H), 5.27-5.24 (m, 1 H), 2-13-2.06 (m, 1 H), 2.06 (s, 3 H), 2.03-1.95 (m, 1 H), 1.90-1.84 (m, 1 H), 1.77-1.70 (m, 2 H), 1.66-1.61 (m, 1 H) ppm;  $^{13}\text{C-NMR}$  (125 MHz,  $\text{CDCl}_3$ ):  $\delta$  = 170.8, 132.7, 125.7, 68.1, 28.3, 24.9, 21.4, 18.9 ppm; IR (film):  $\nu$  = 3033 (w), 2938 (w), 2868 (w), 2836 (w), 1725 (s), 1433 (w), 1370 (m), 1233 (s), 1162 (w), 1097 (w), 1028 (m), 1007 (m), 952 (w), 906 (m), 729 (m), 626 (w) cm<sup>-1</sup>; MS (EI, 70 eV): m/z 140 (M)<sup>+</sup> (30), 98 (M-C<sub>2</sub>H<sub>2</sub>O)<sup>+</sup> (100), 83 (M-C<sub>3</sub>H<sub>5</sub>O)<sup>+</sup> (25), 79 (C<sub>6</sub>H<sub>7</sub>)<sup>+</sup> (90), 70 (C<sub>4</sub>H<sub>6</sub>O)<sup>+</sup> (35), 53 (C<sub>3</sub>H<sub>3</sub>O)<sup>+</sup> (8), 43 (C<sub>2</sub>H<sub>3</sub>O)<sup>+</sup> (65).

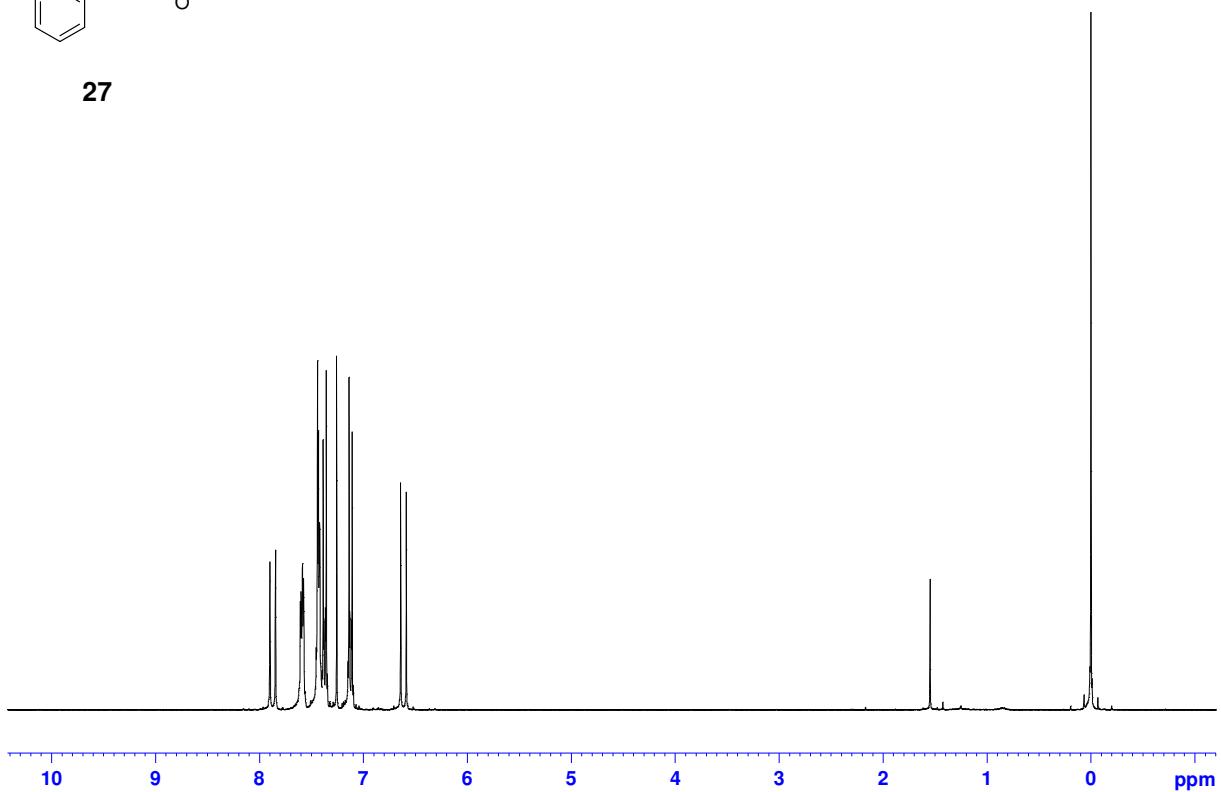
#### **S-IV $^1\text{H-NMR}$ spectra of ester**

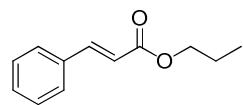


16

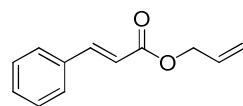
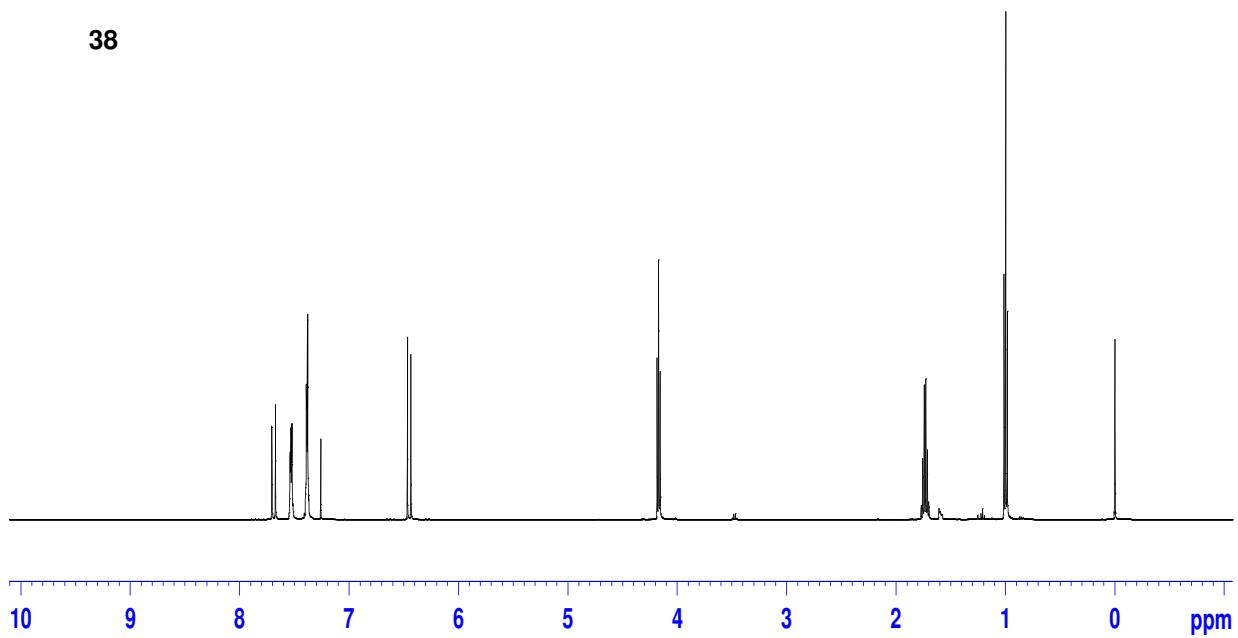


27

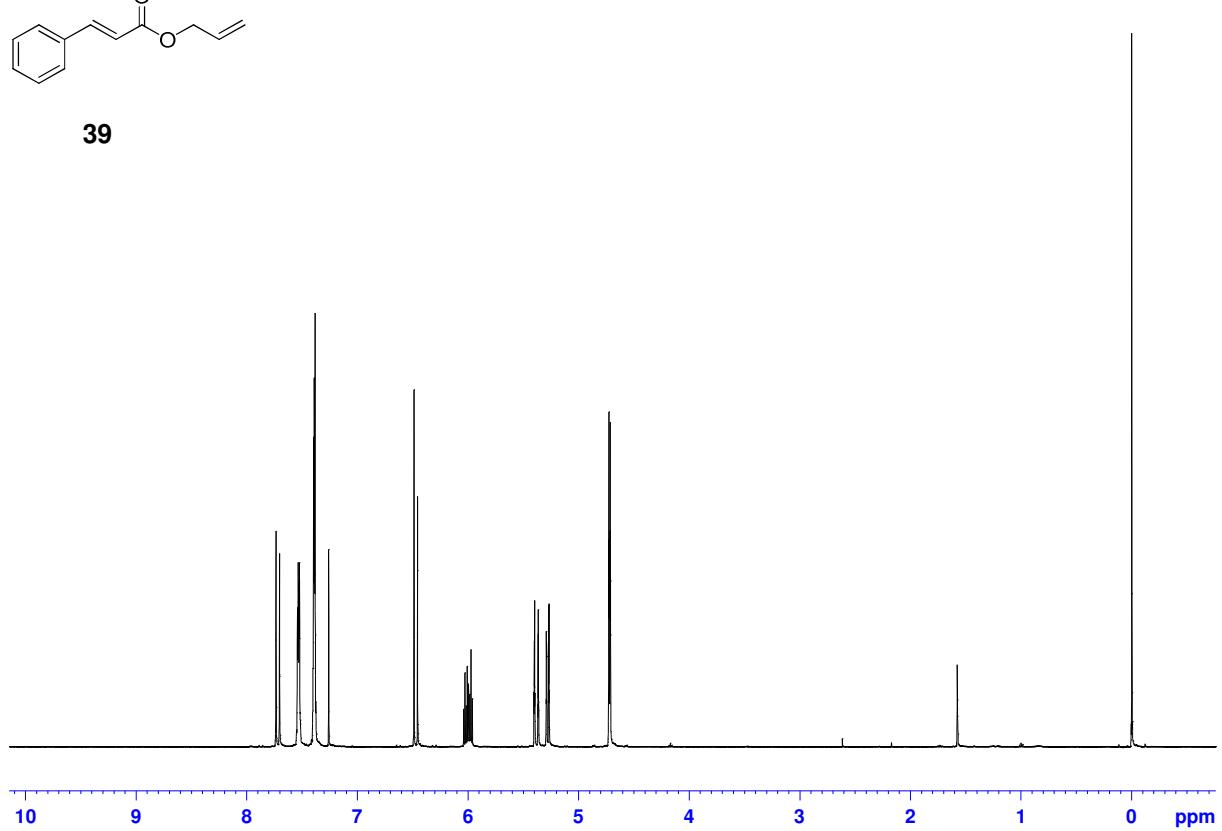


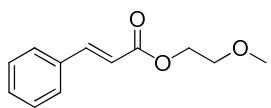


**38**

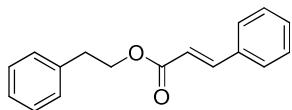
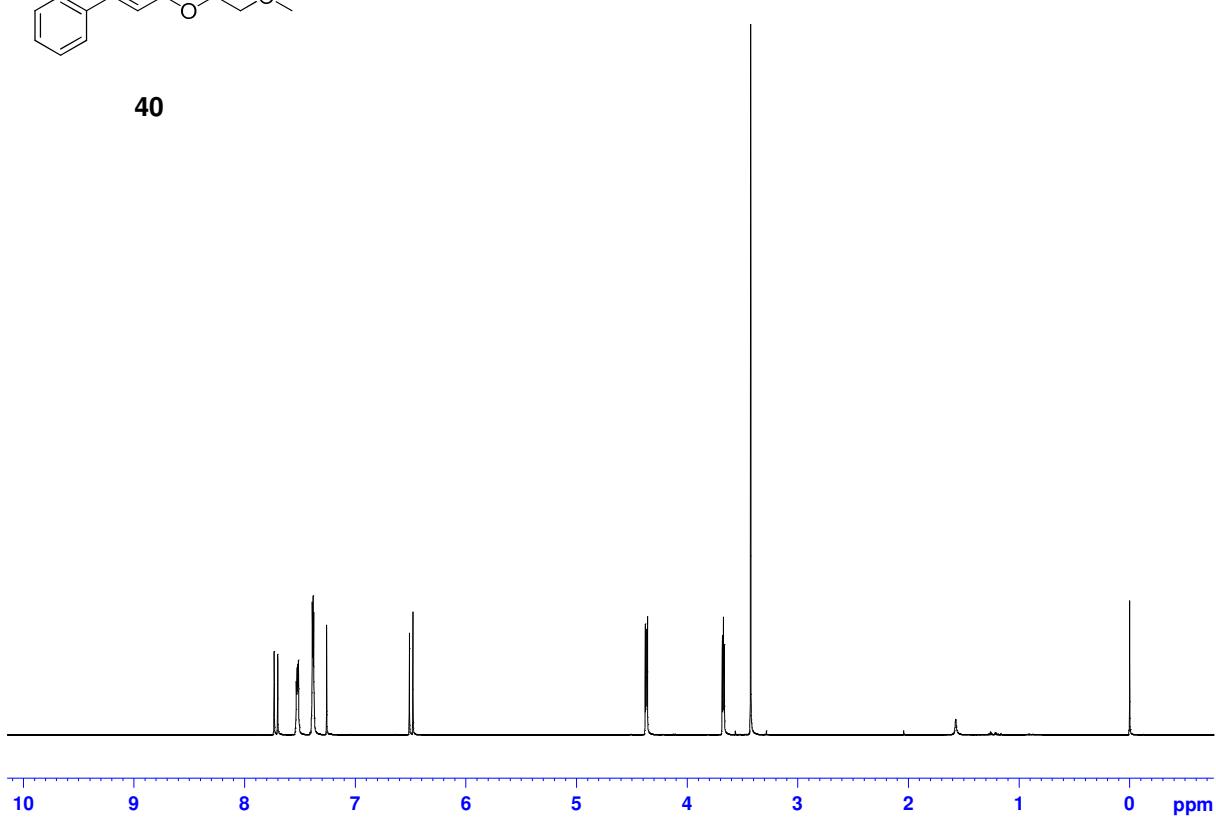


**39**

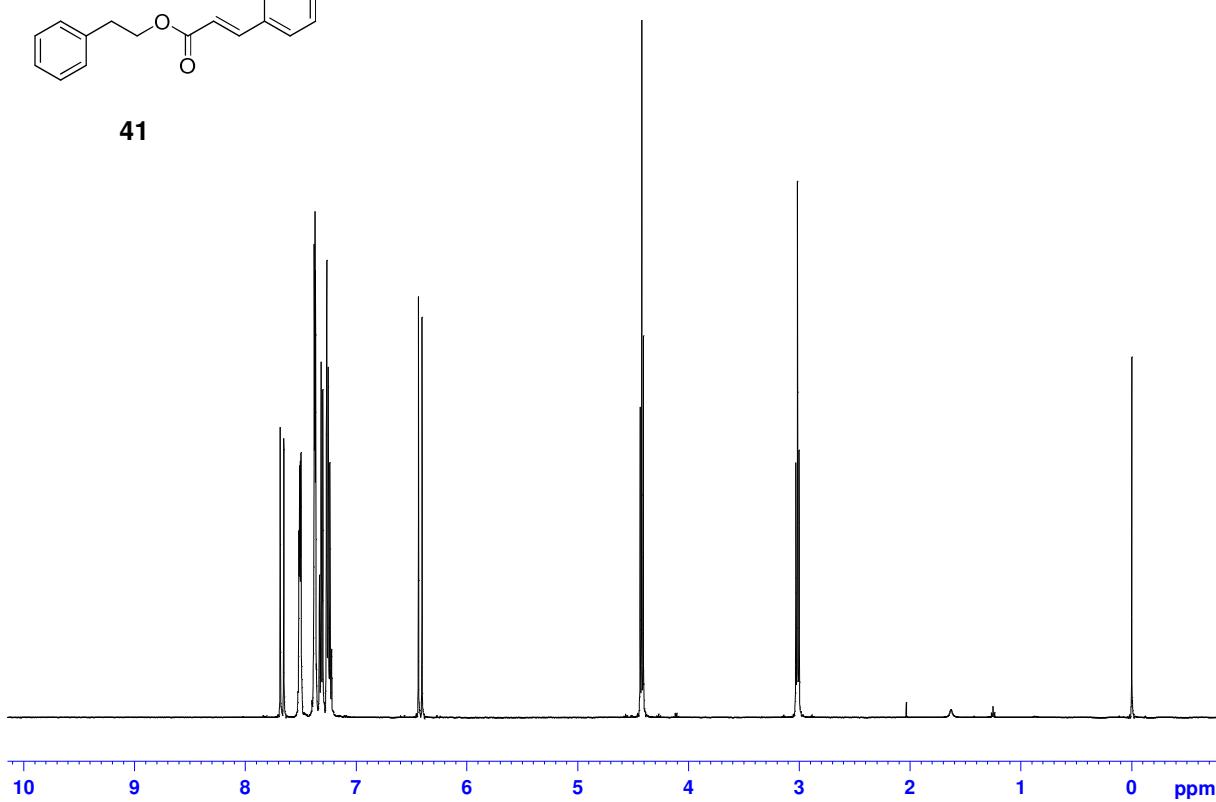


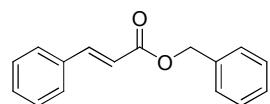


**40**

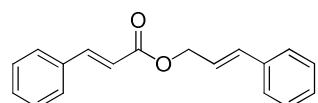
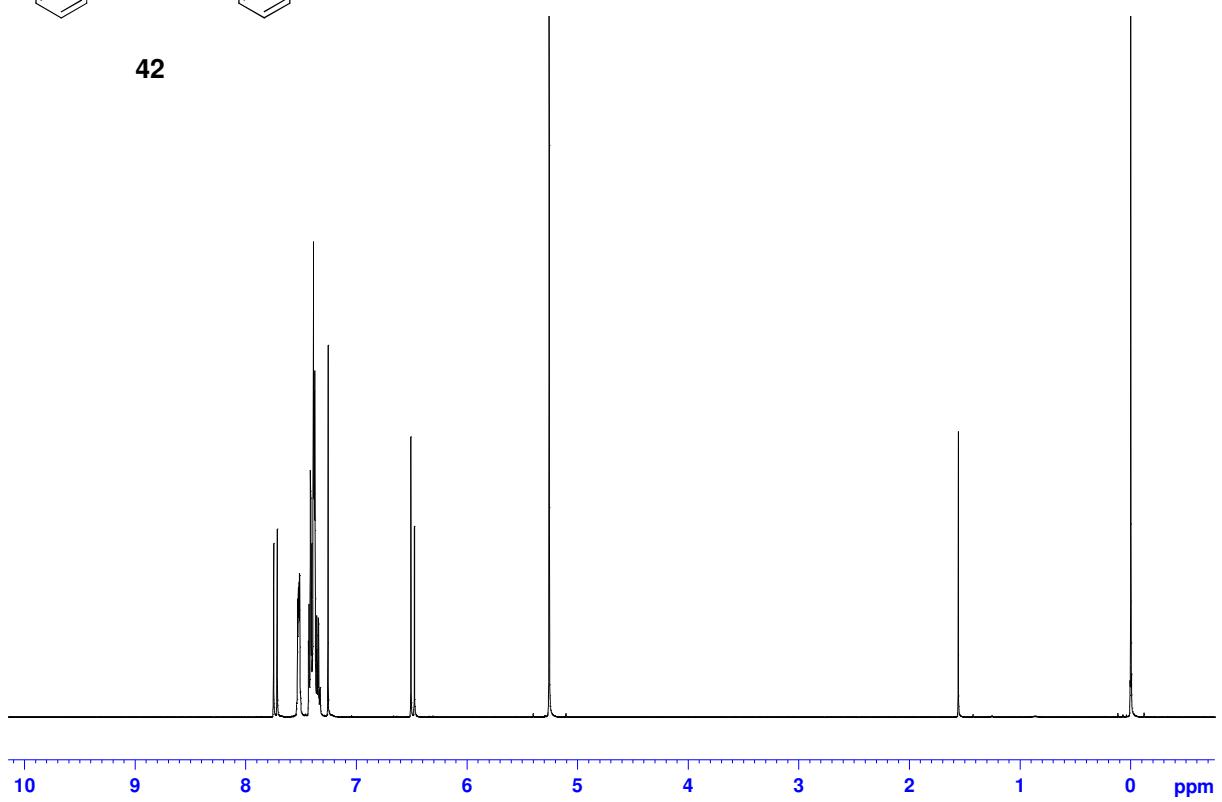


**41**

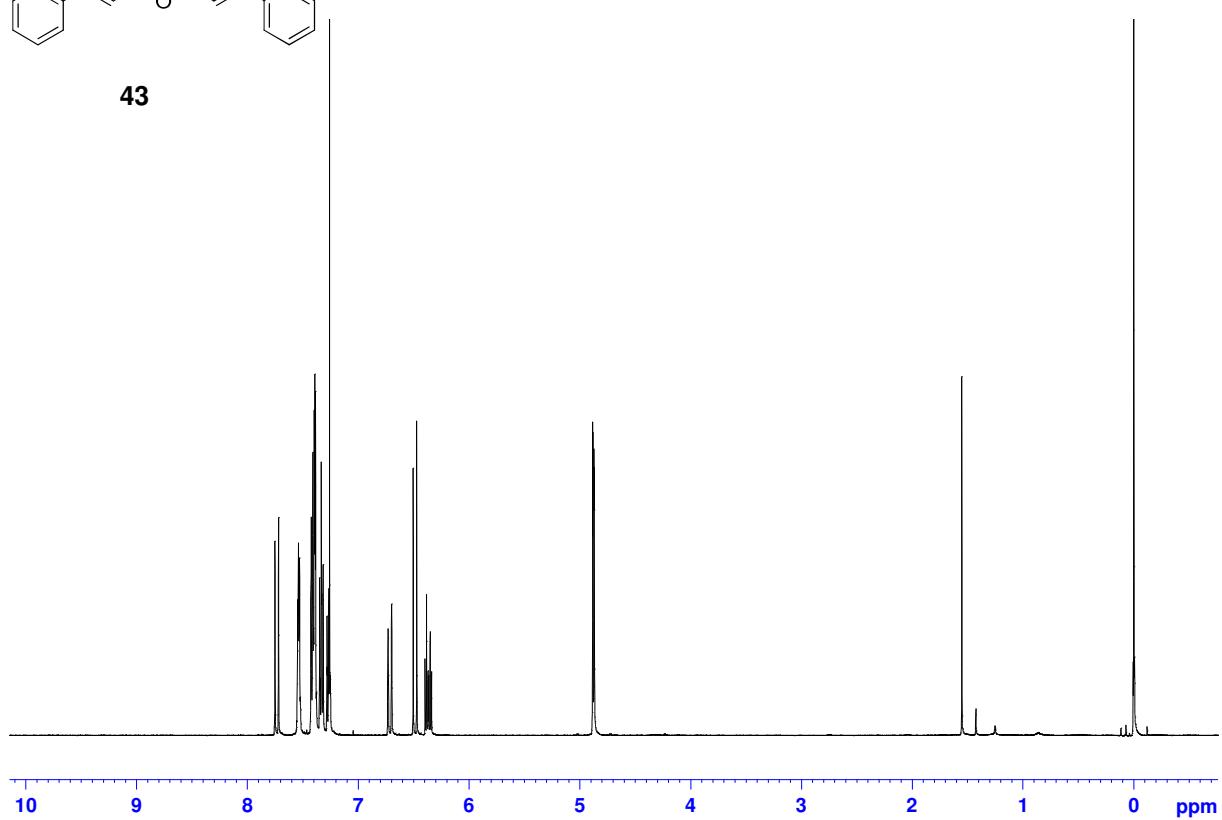


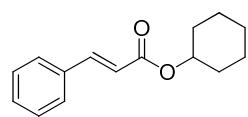


**42**

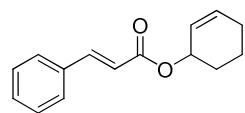
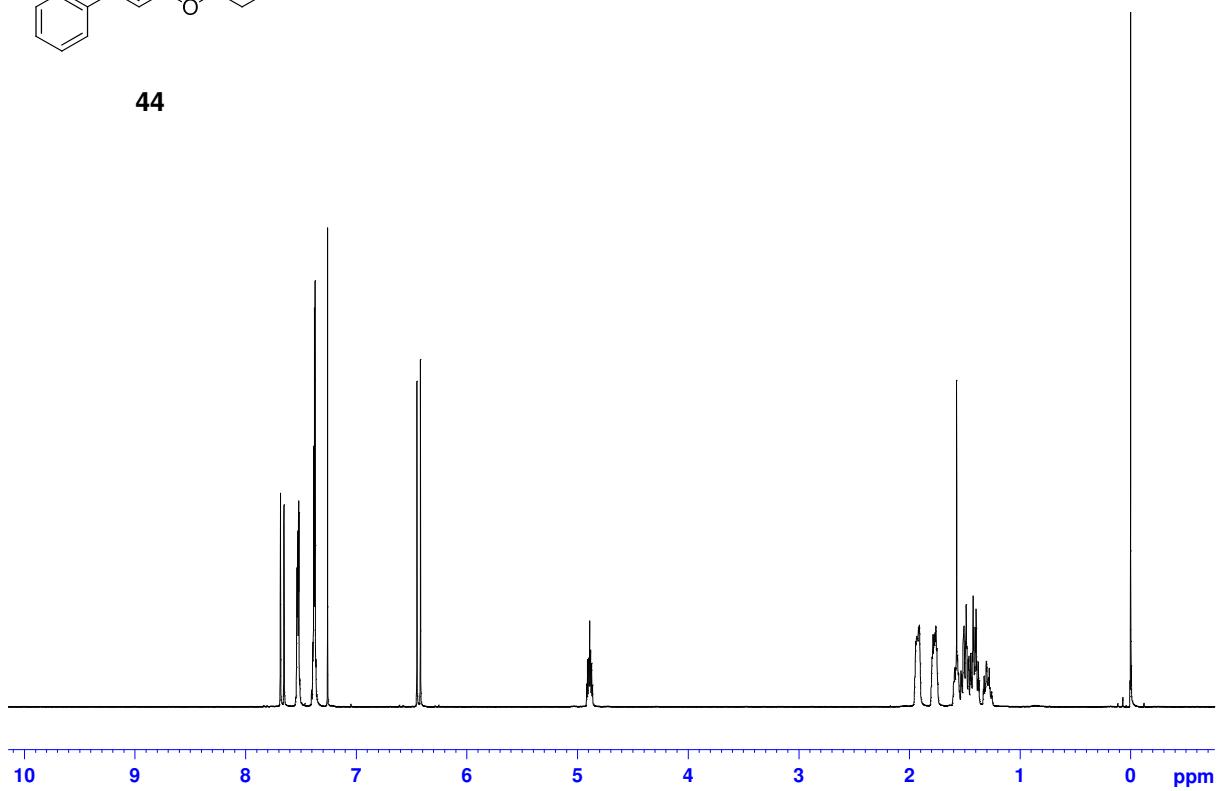


**43**

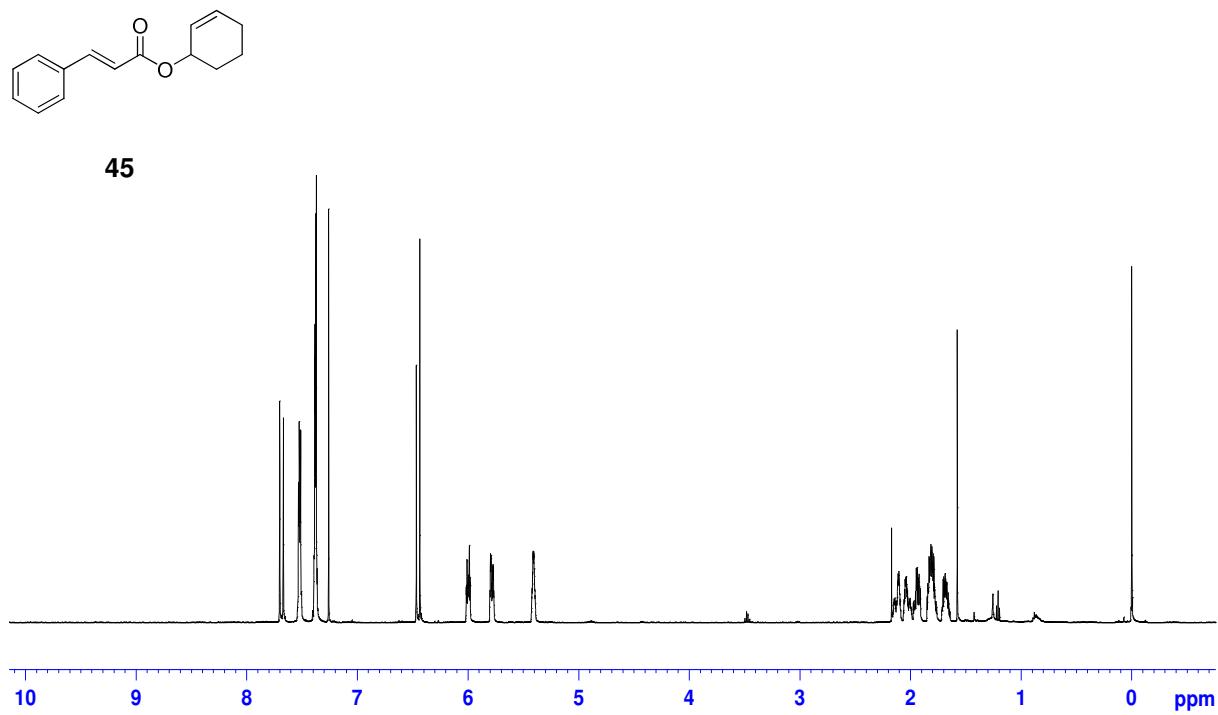


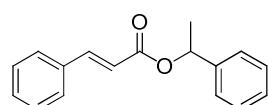


**44**

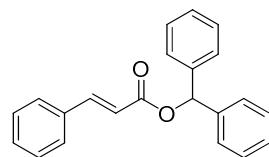
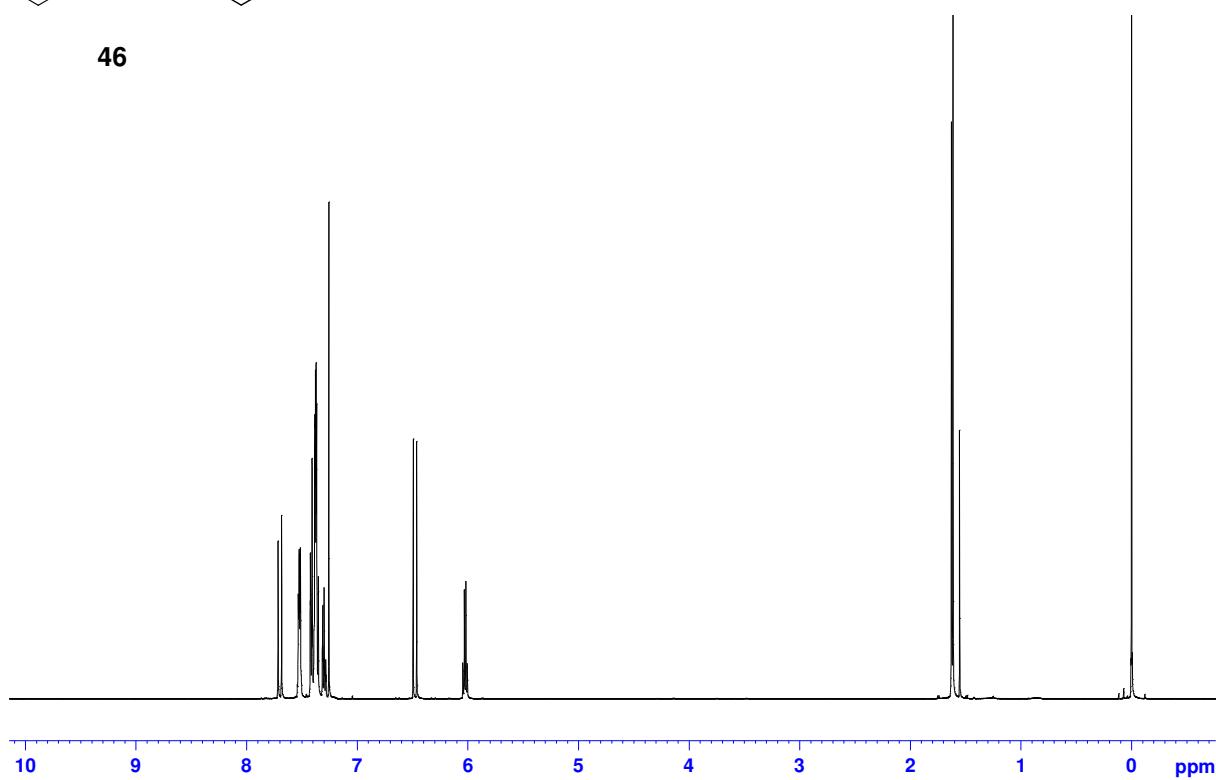


**45**

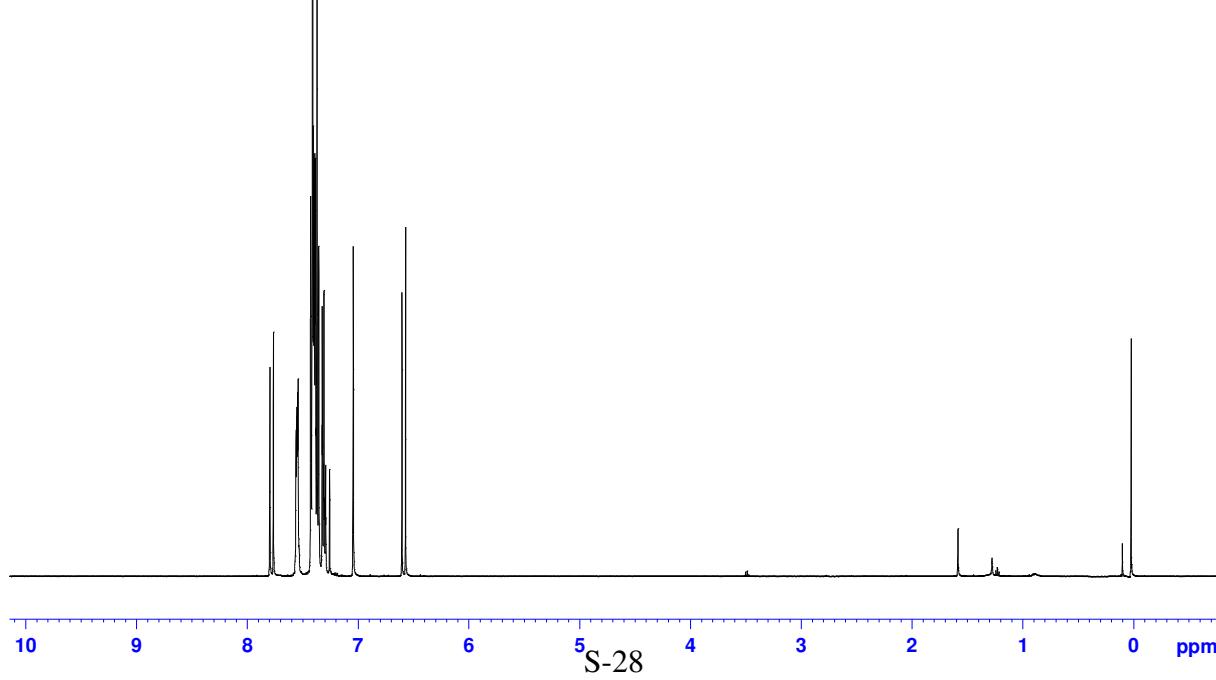


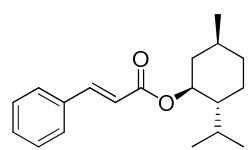


**46**

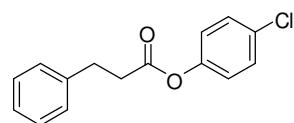
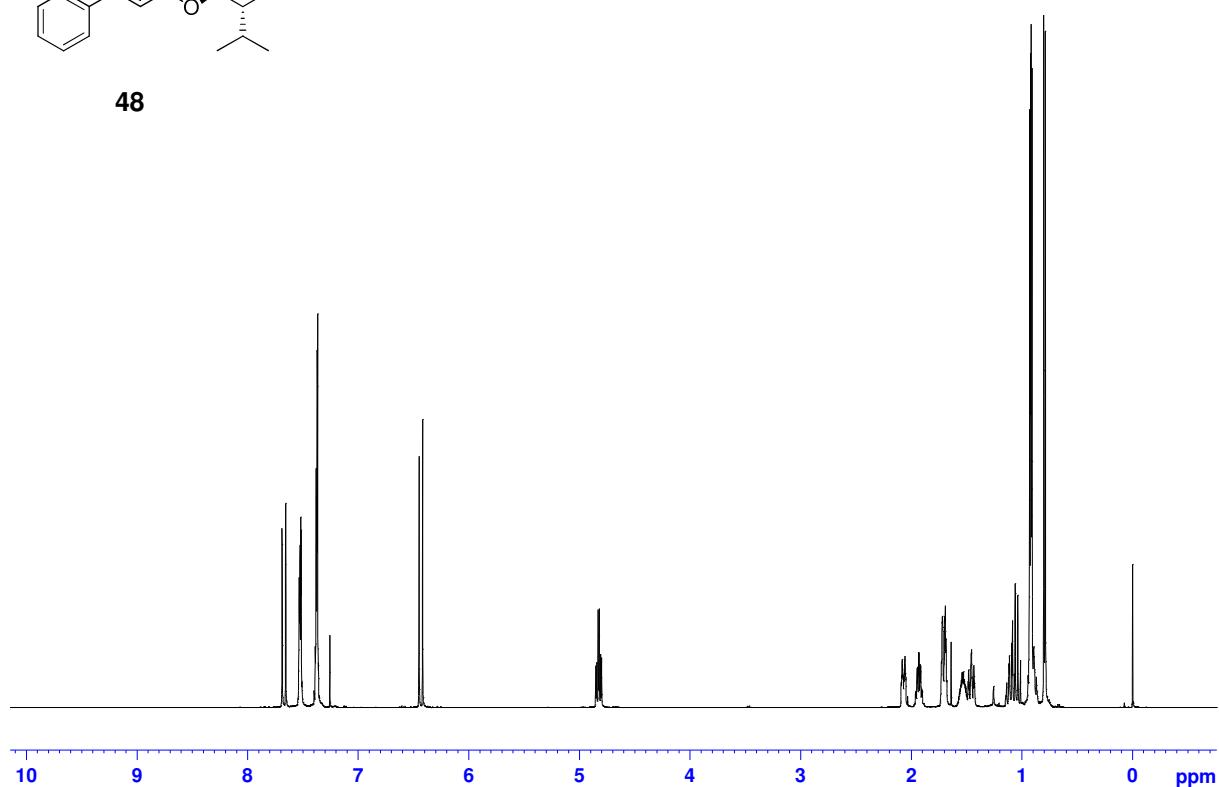


**47**

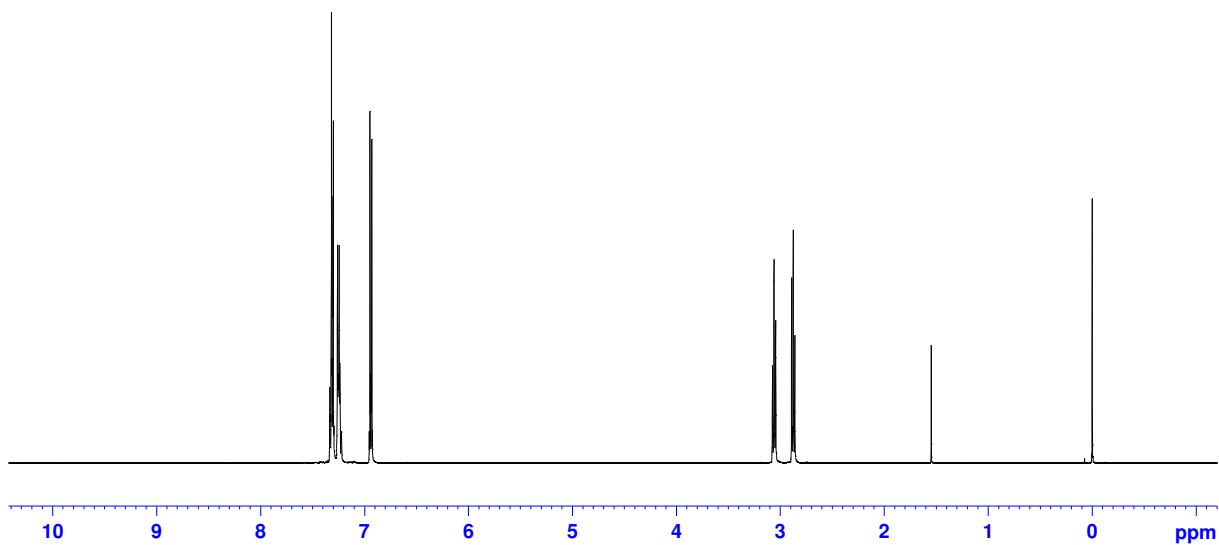


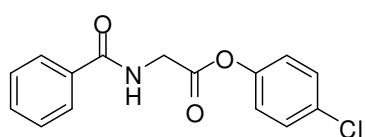


**48**

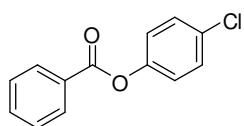
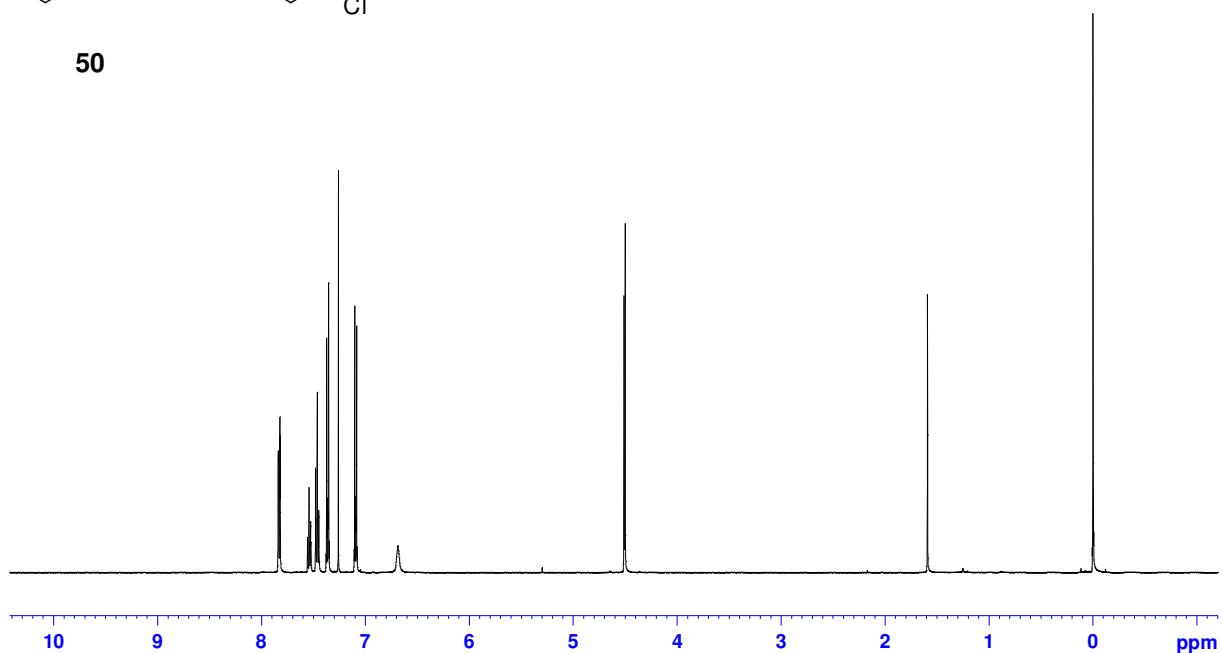


**49**

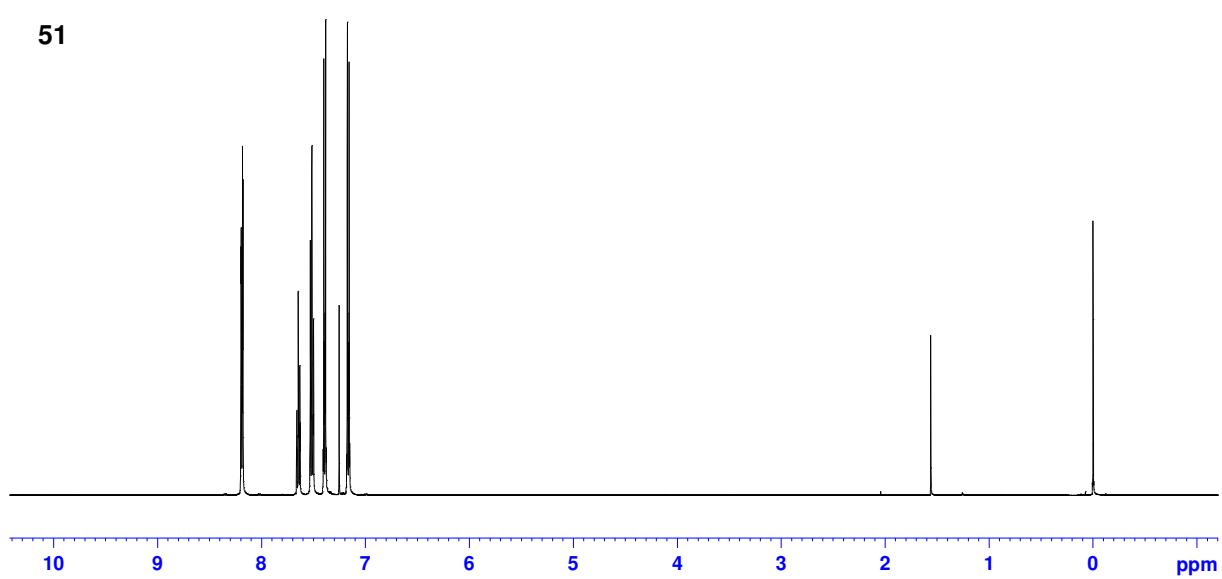


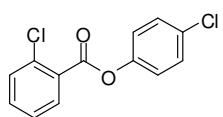


**50**

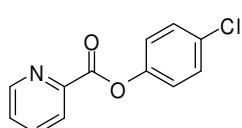
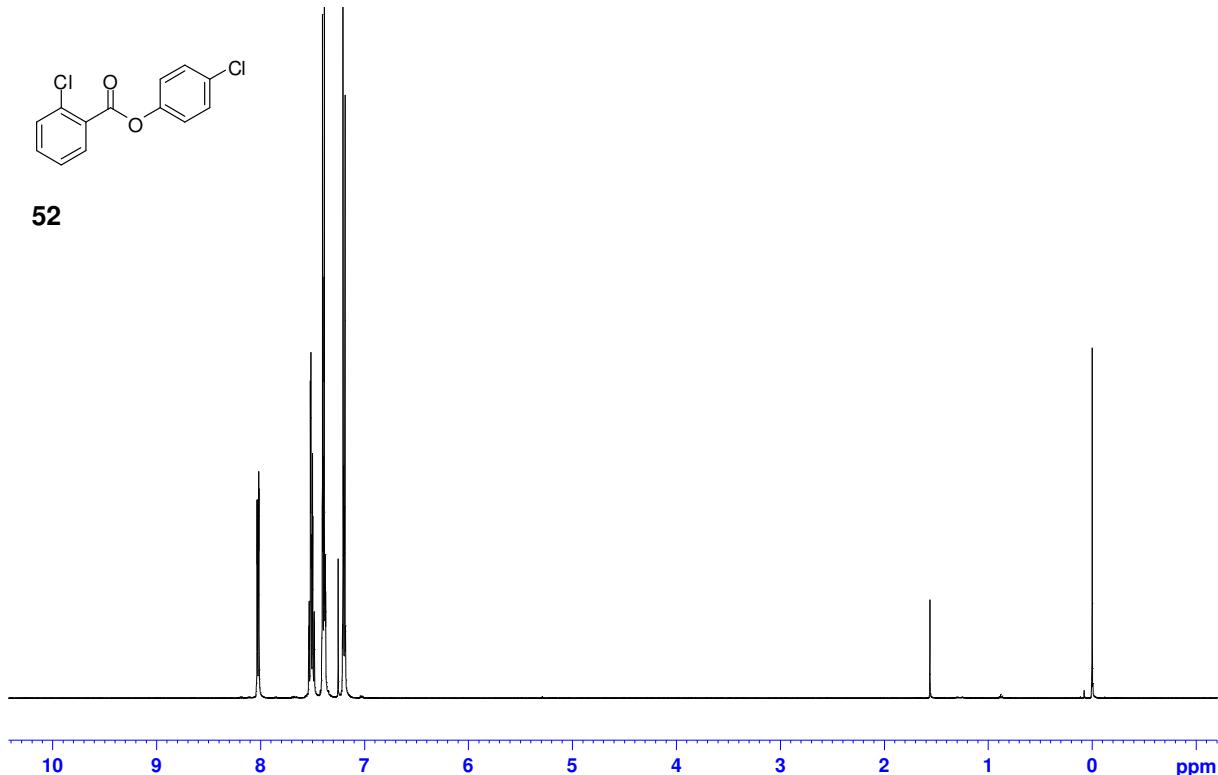


**51**

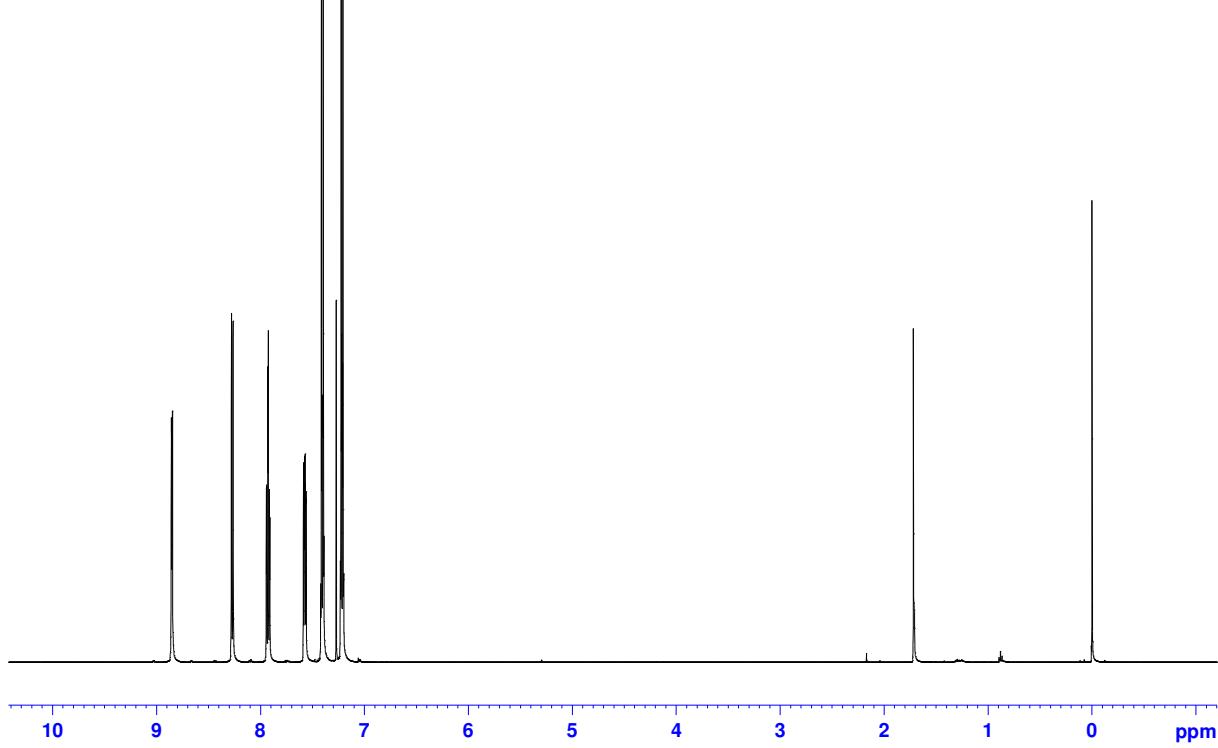


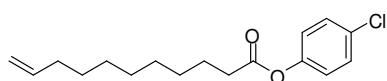


**52**

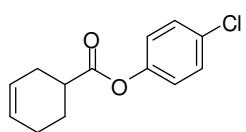
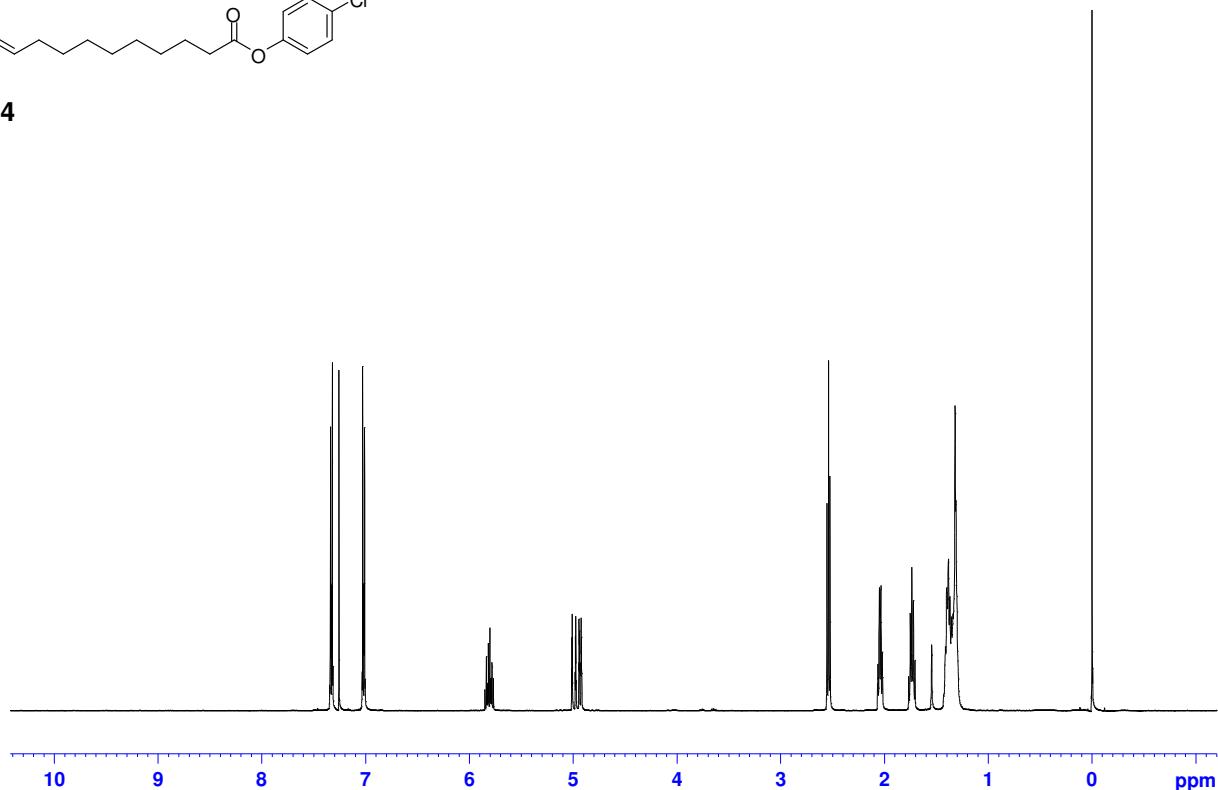


**53**

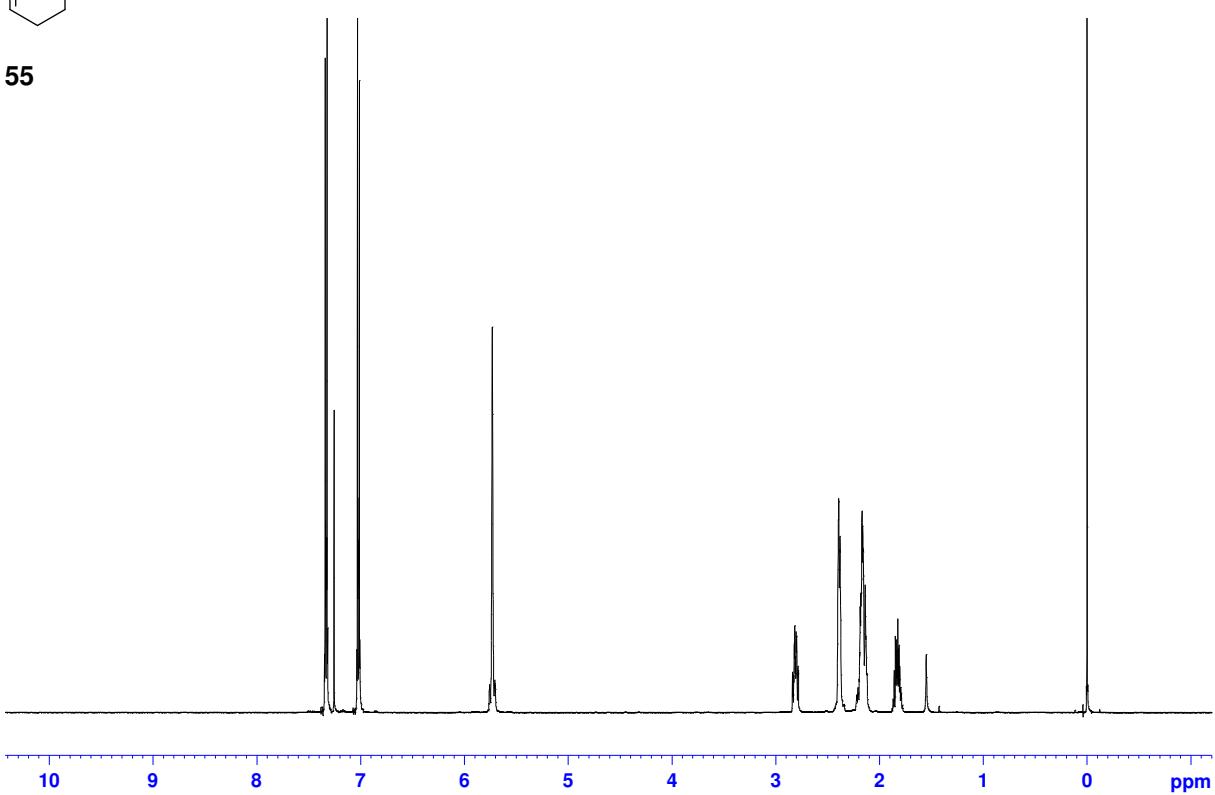


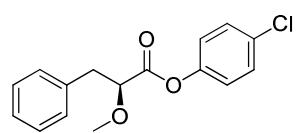


**54**

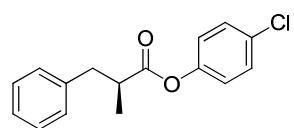
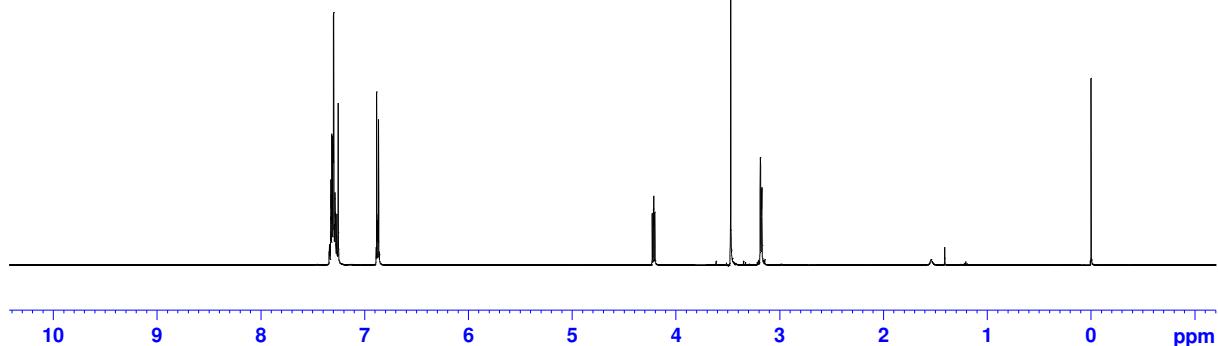


**55**

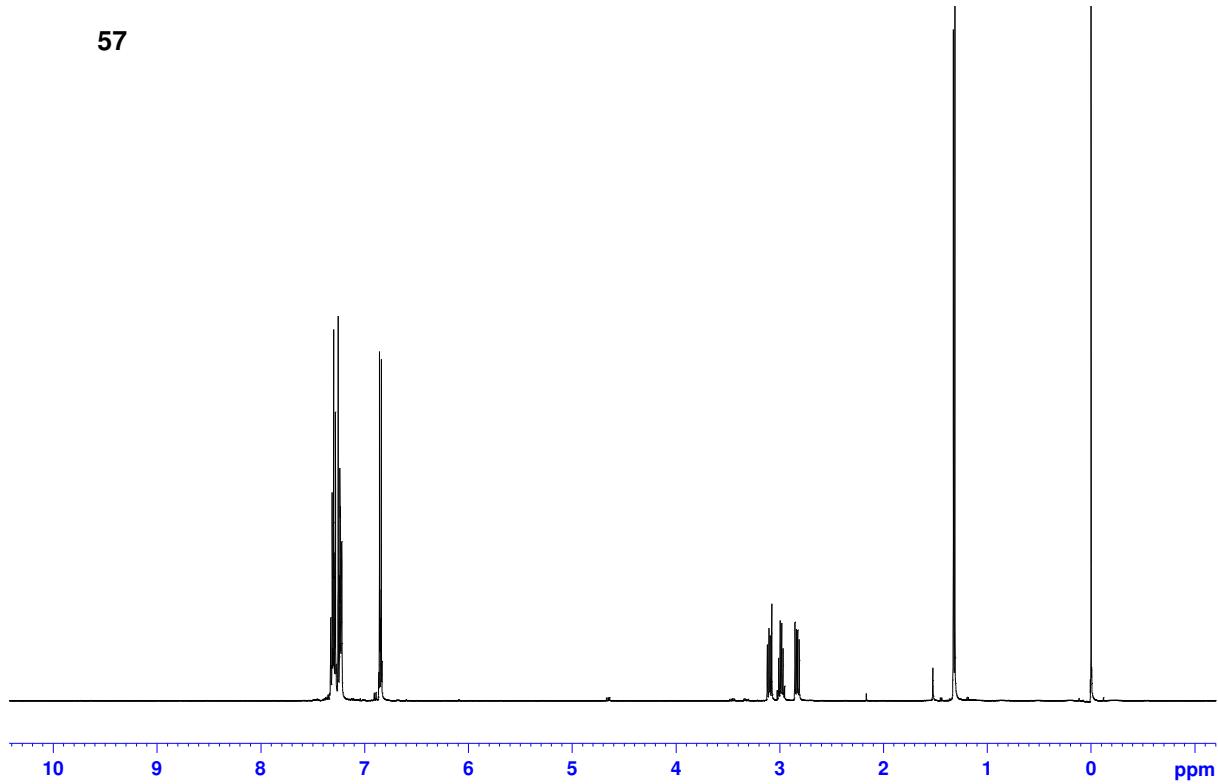


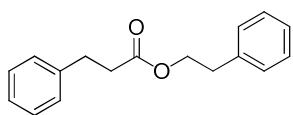


**56**

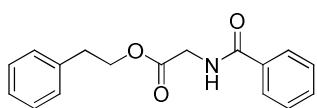
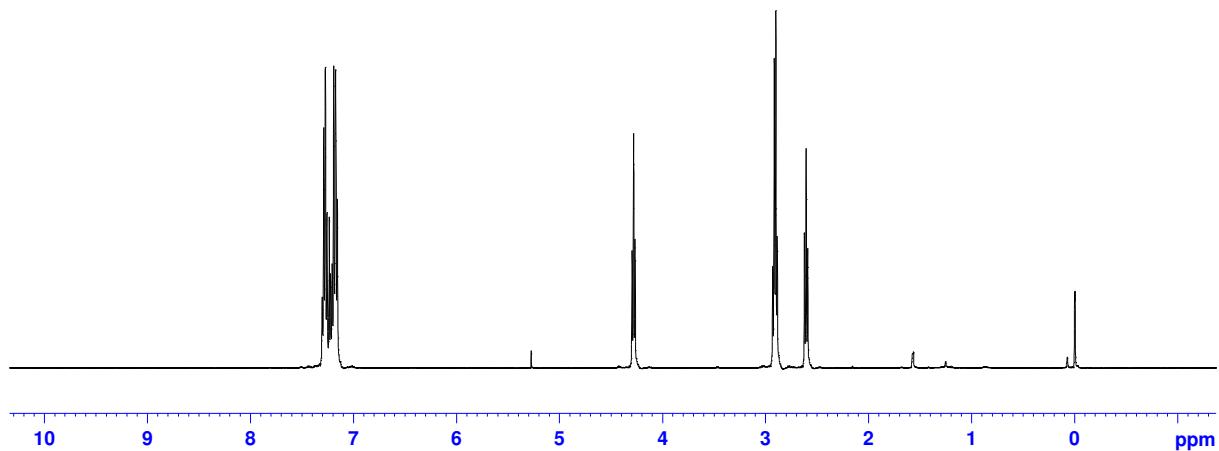


**57**

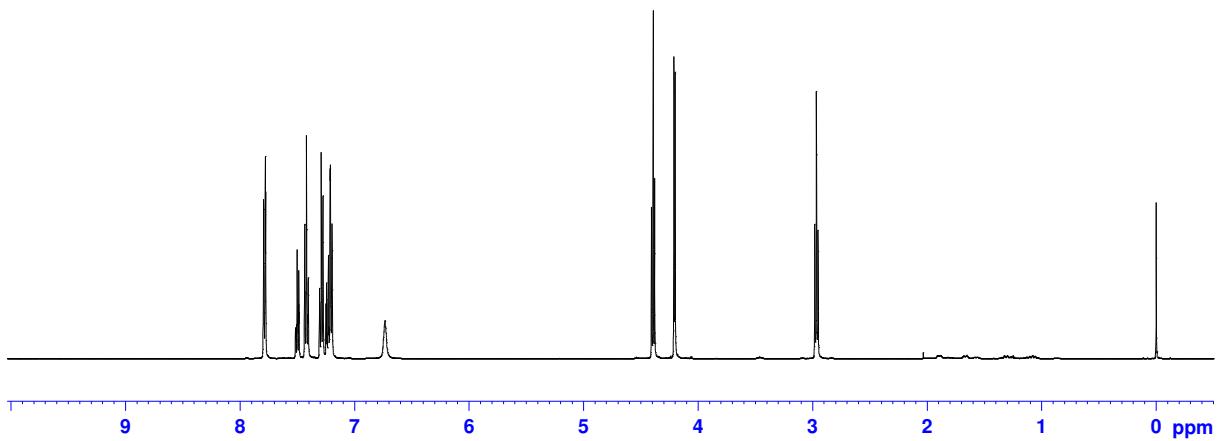


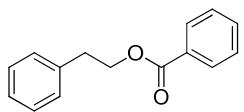


**58**

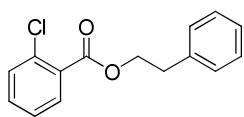
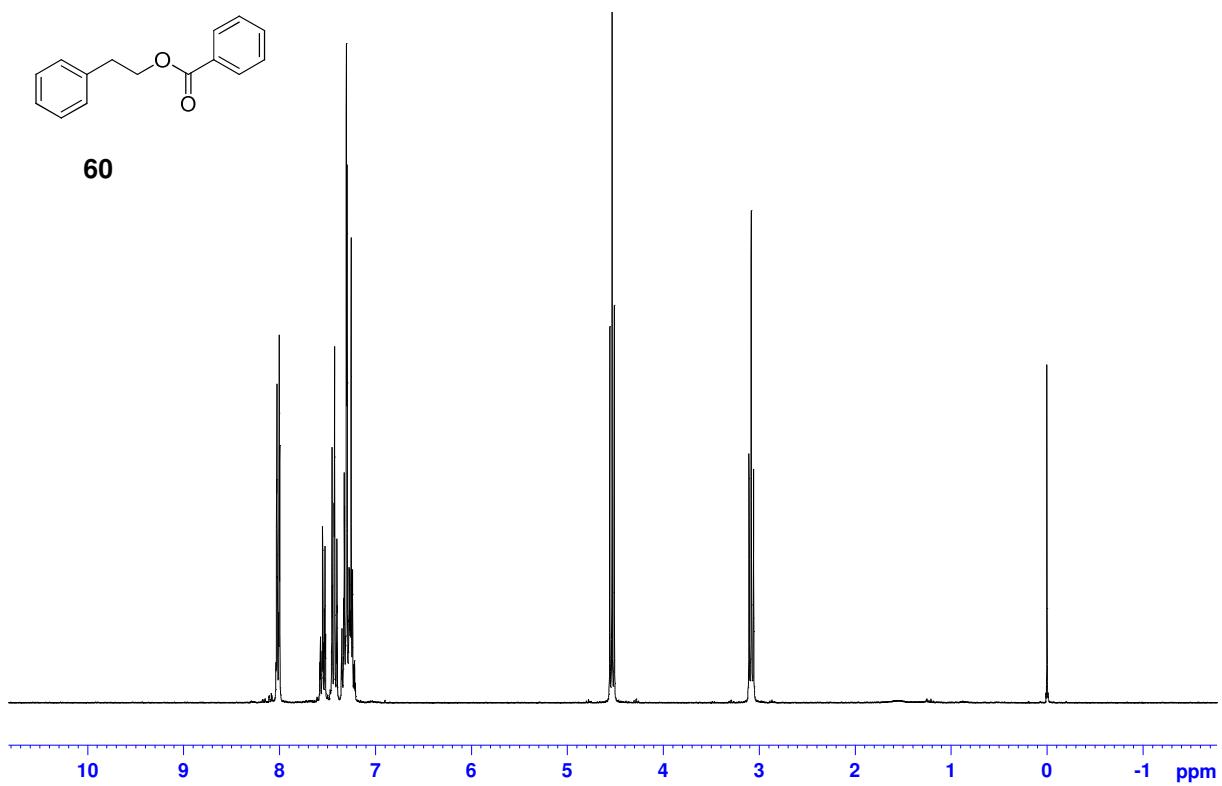


**59**

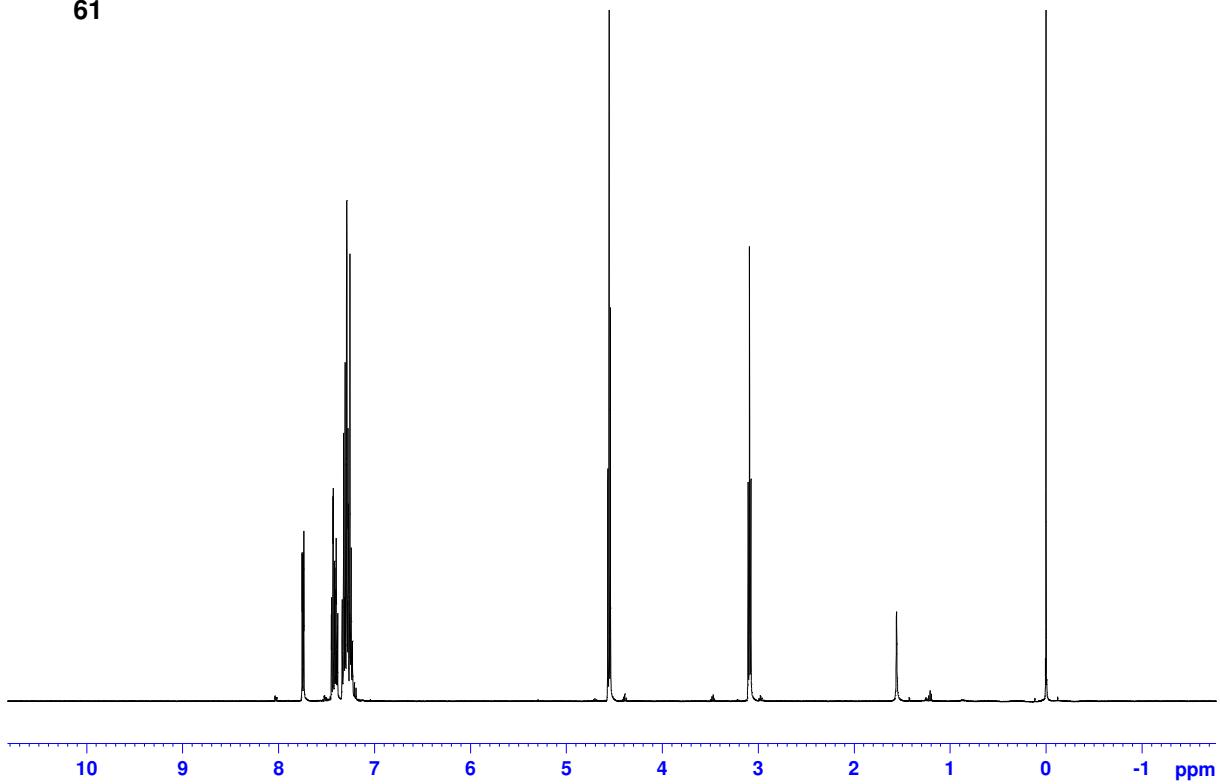


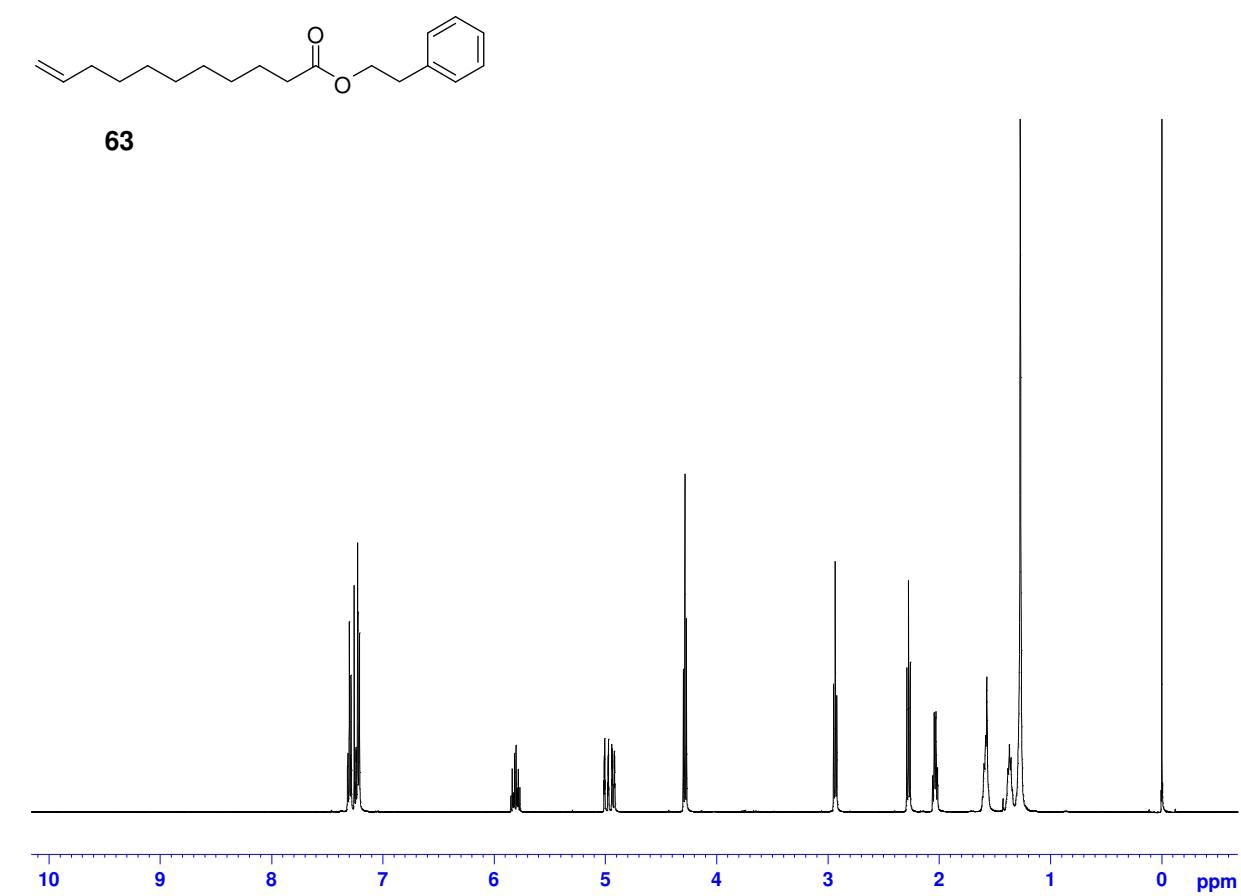
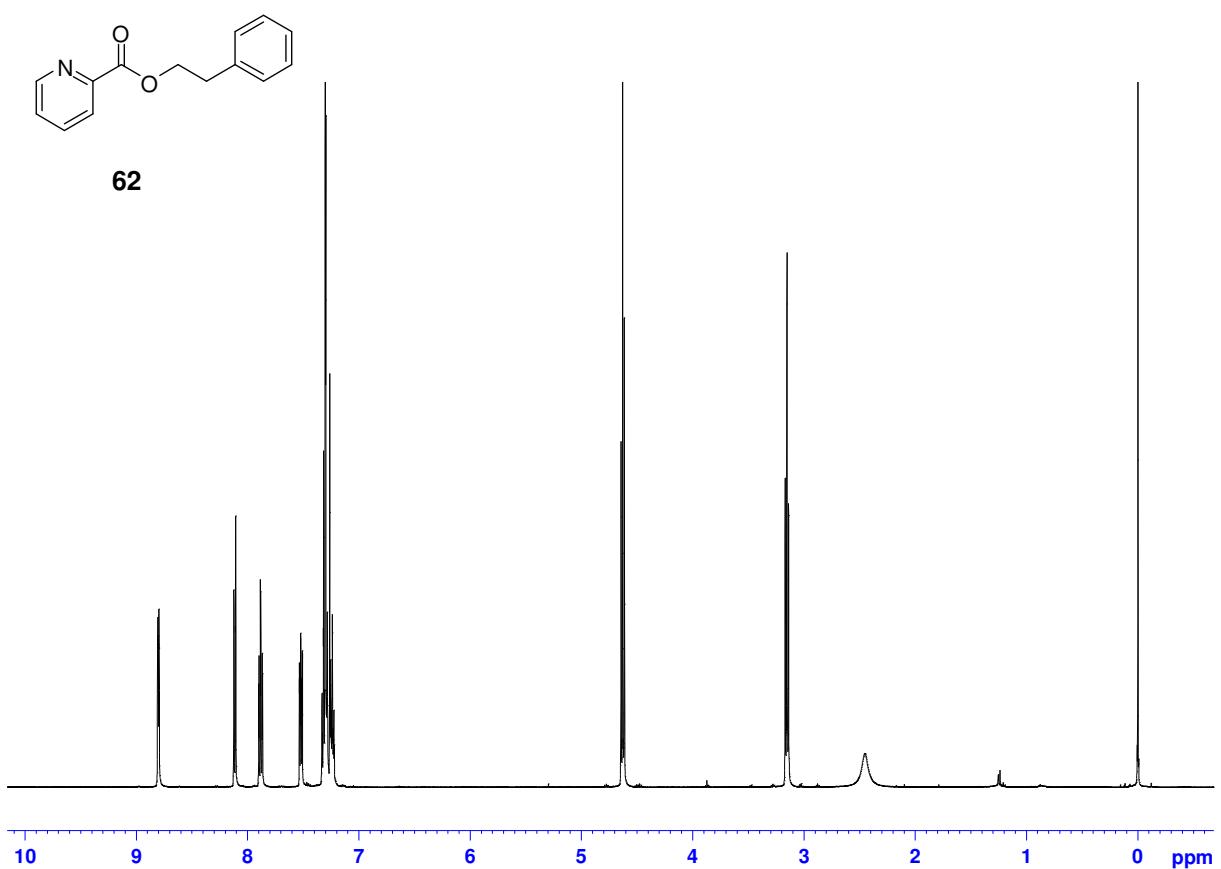


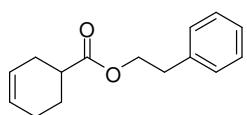
**60**



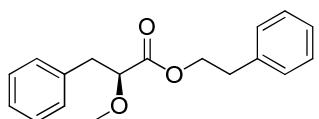
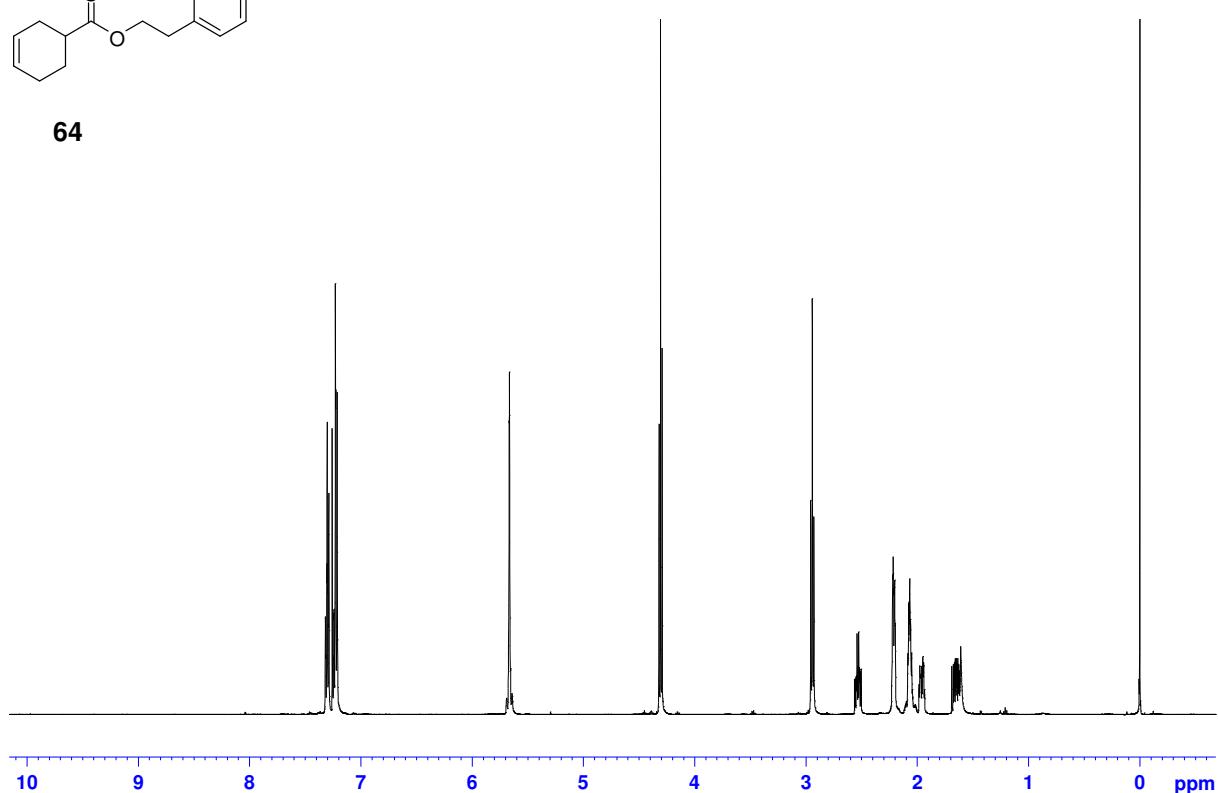
**61**



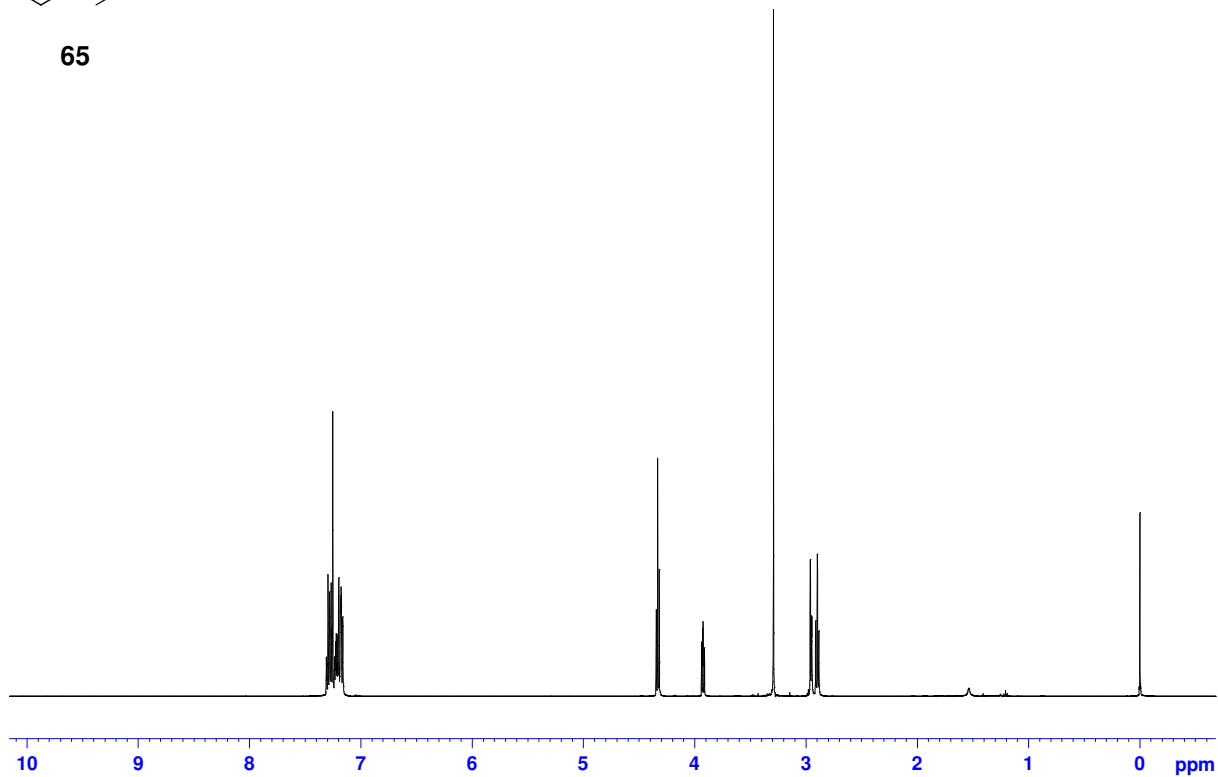


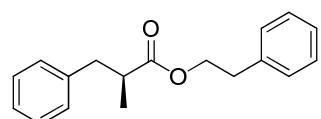


**64**

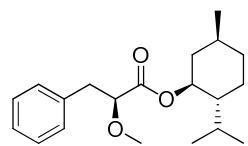
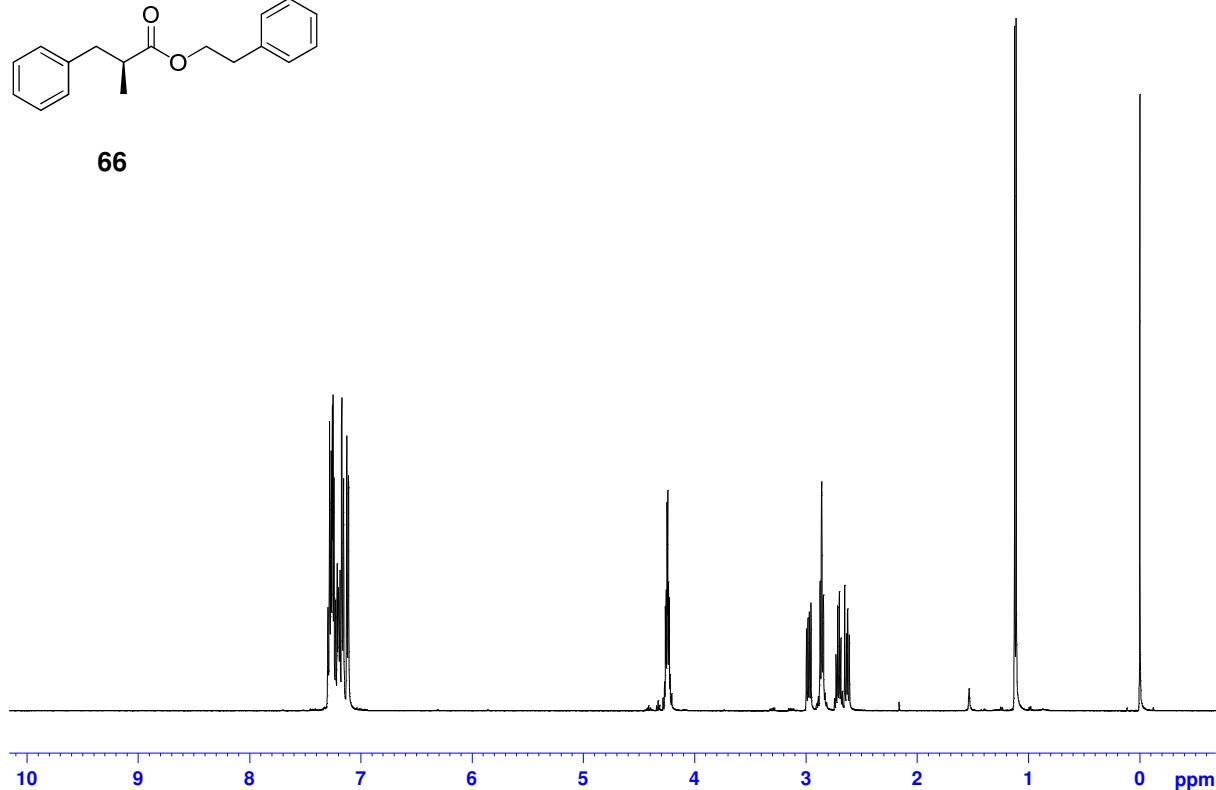


**65**

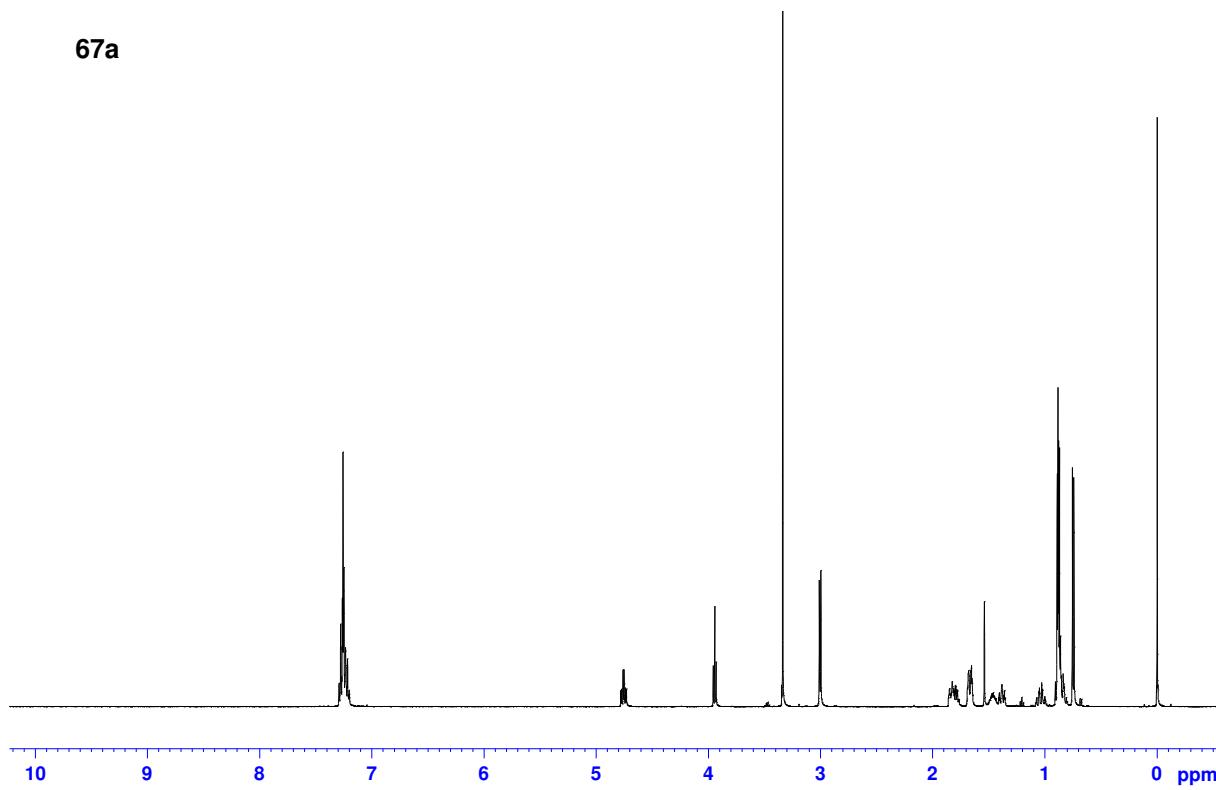


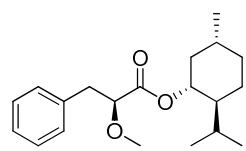


**66**

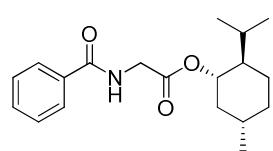
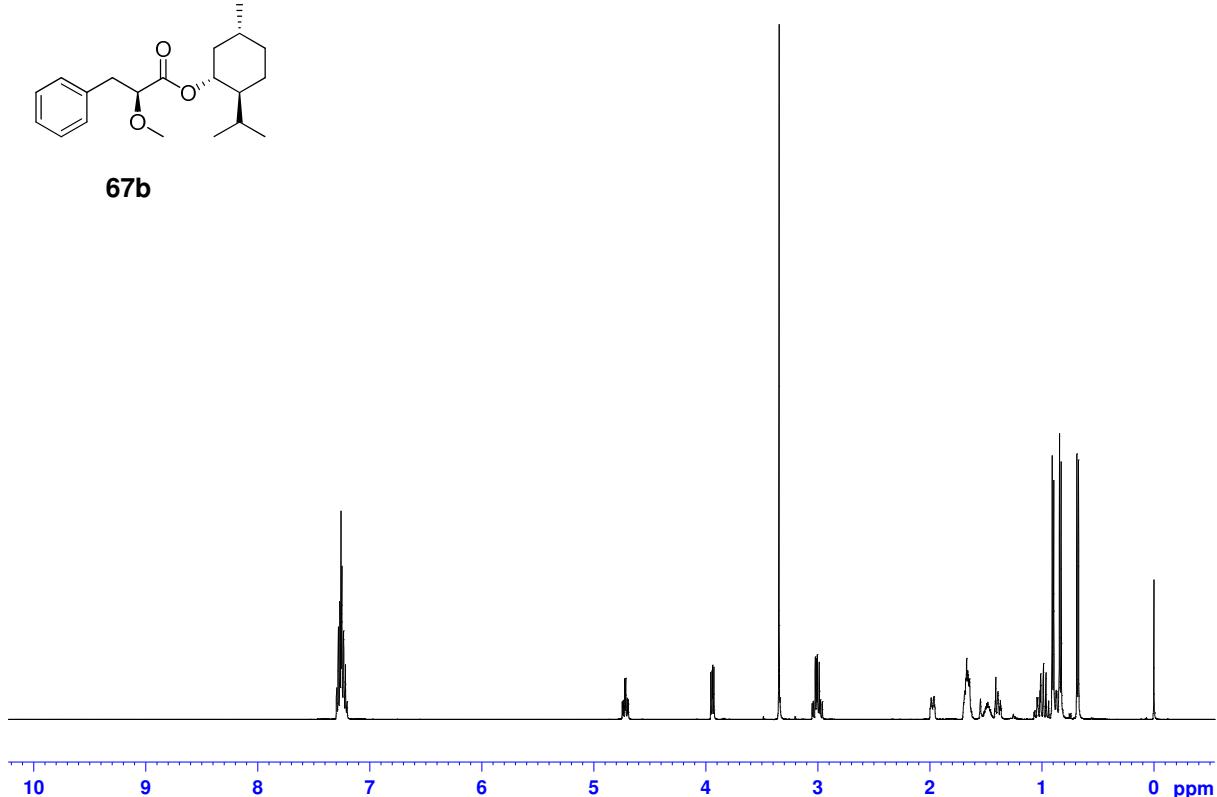


**67a**

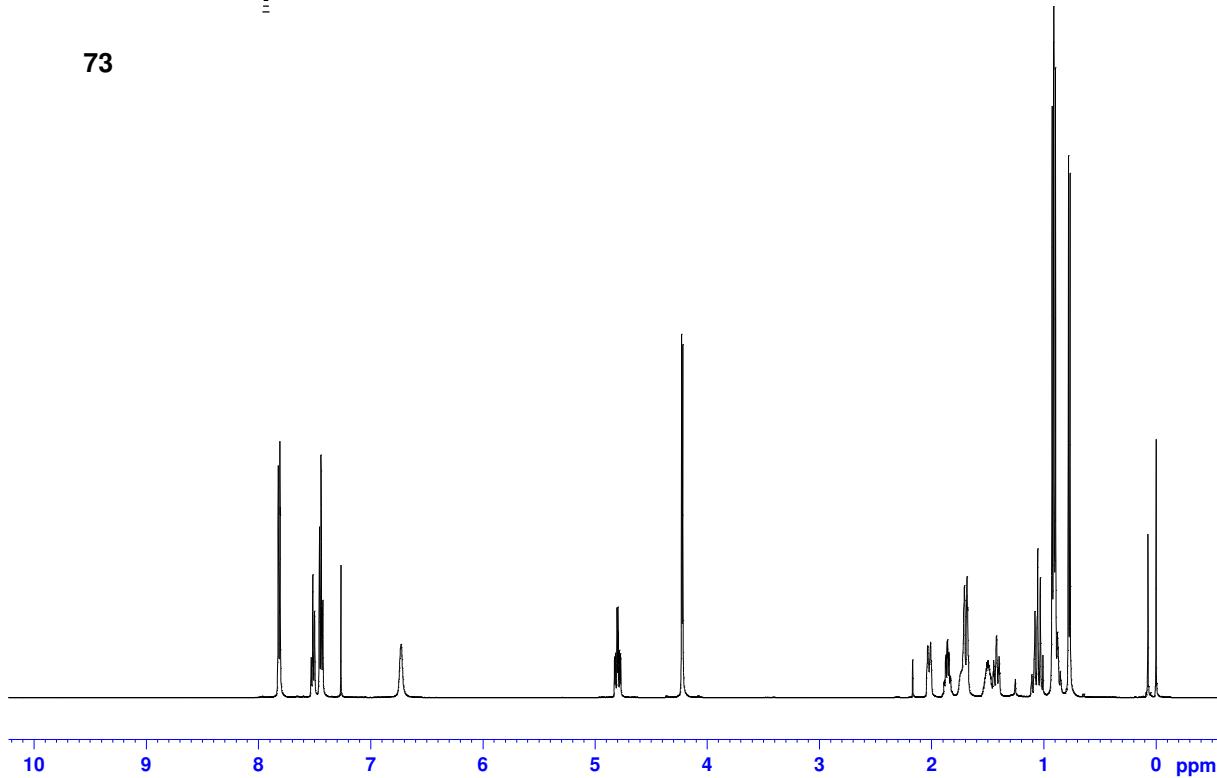


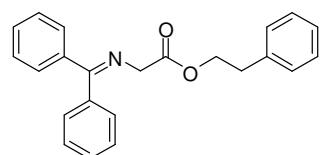


**67b**

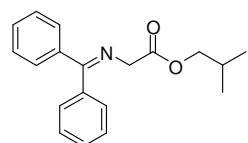
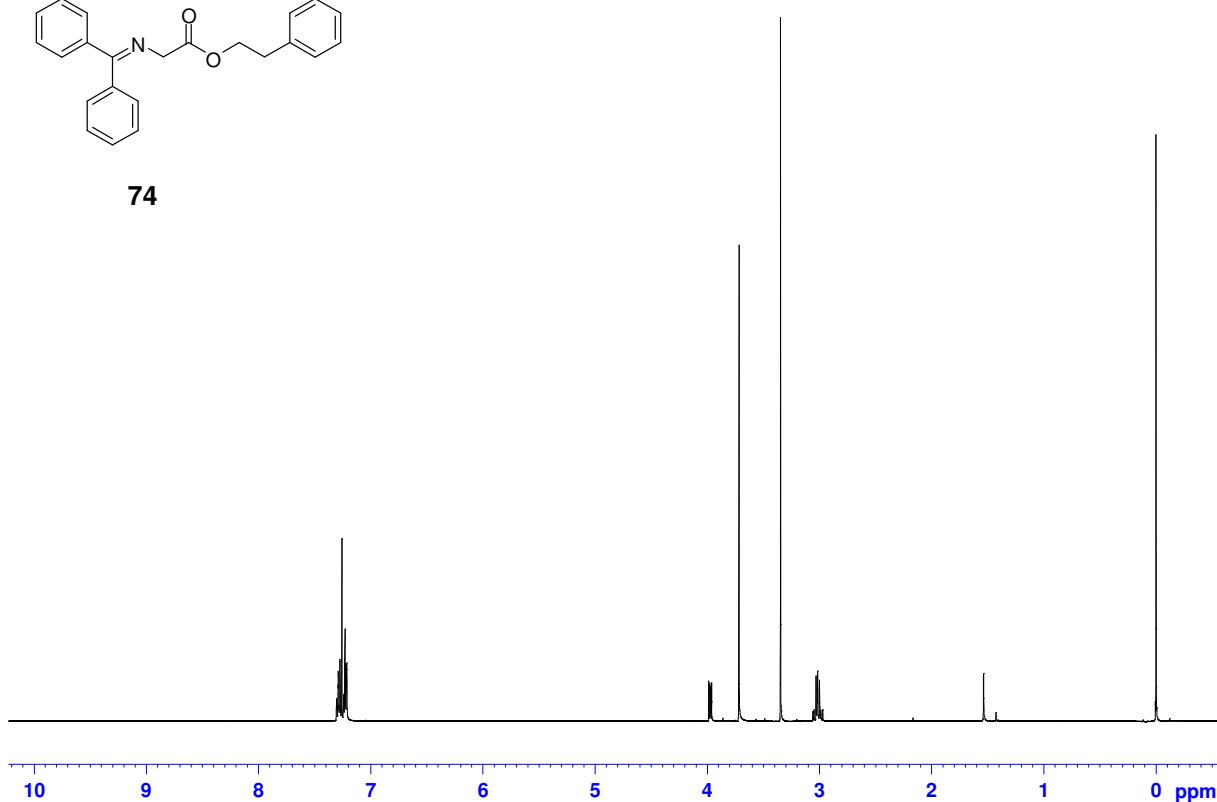


**73**

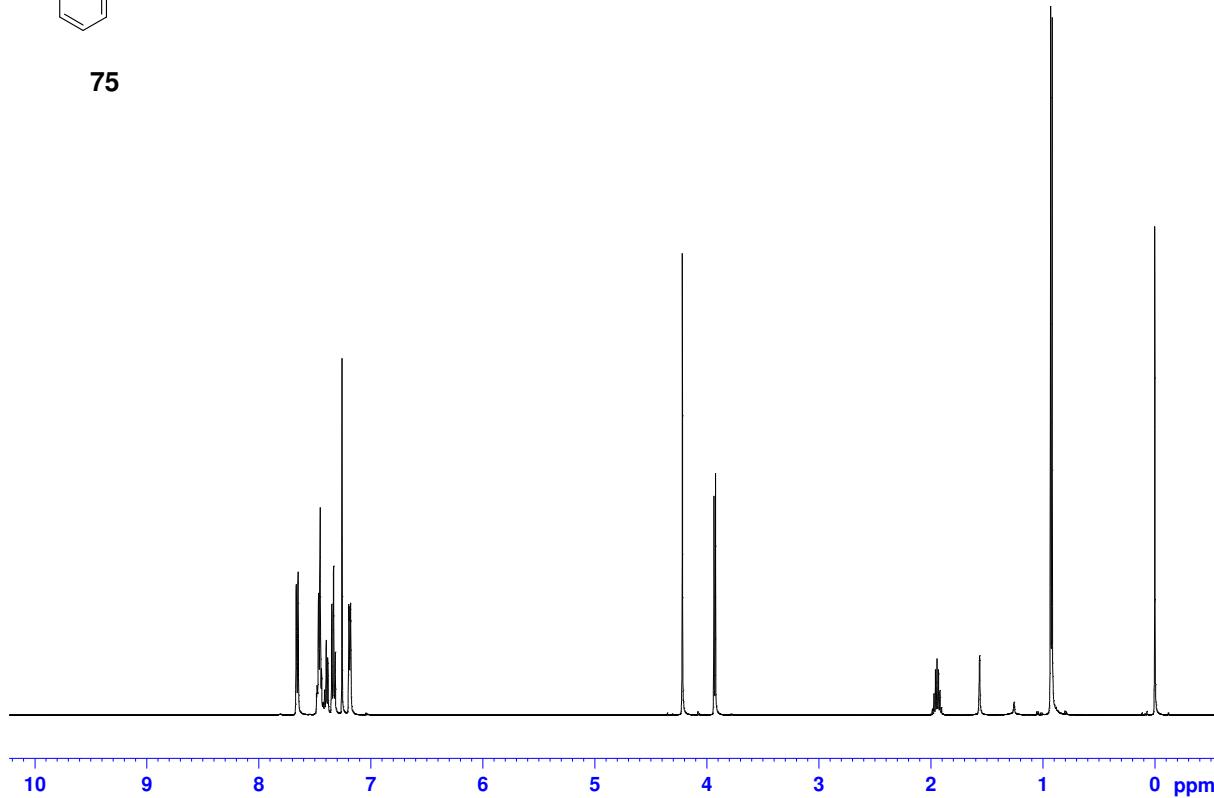


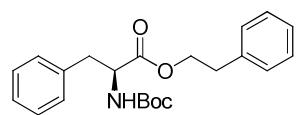


**74**

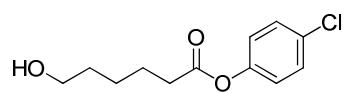
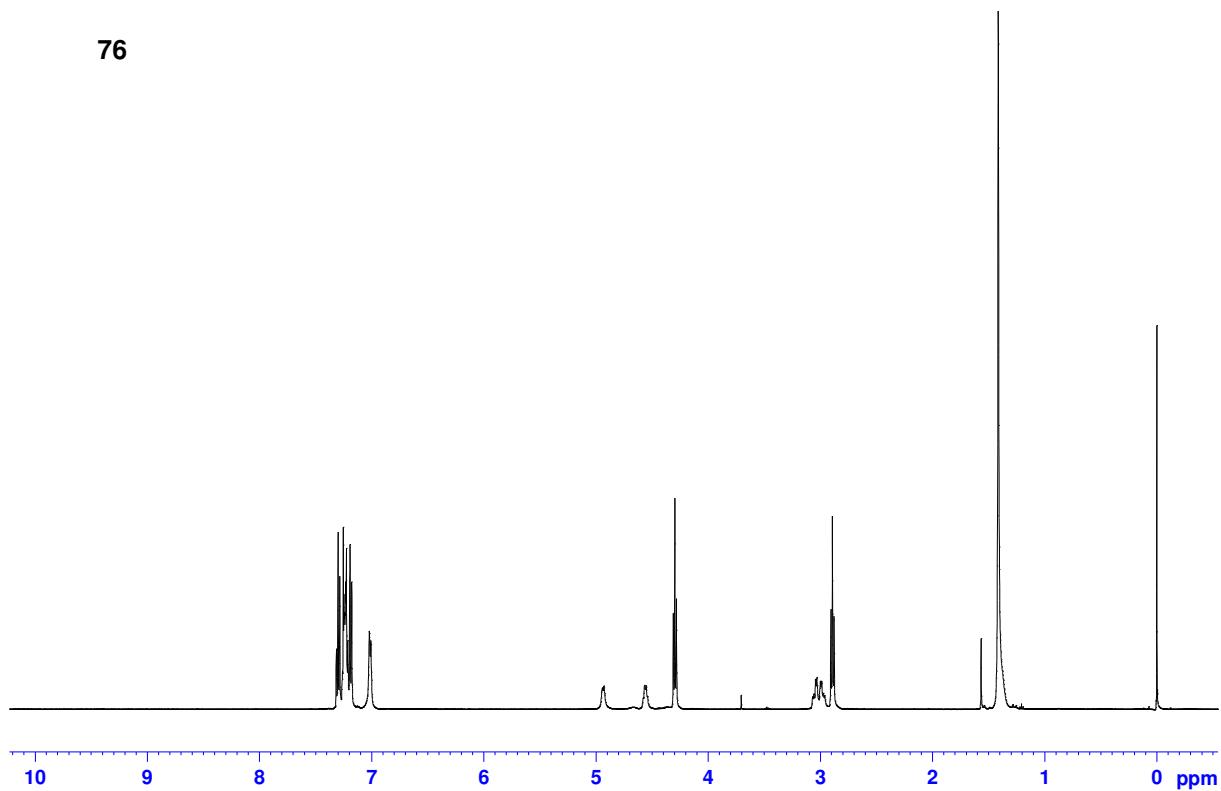


**75**

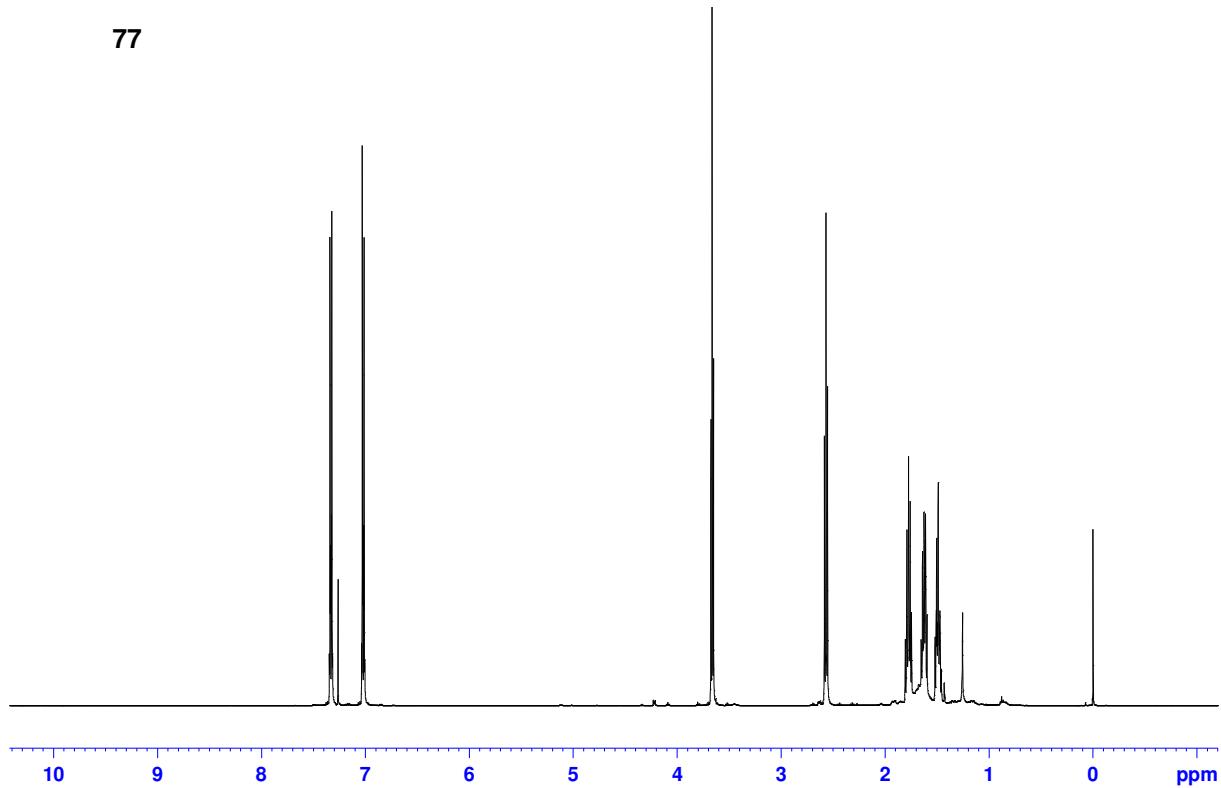


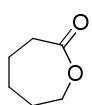


**76**

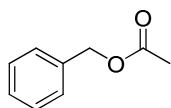
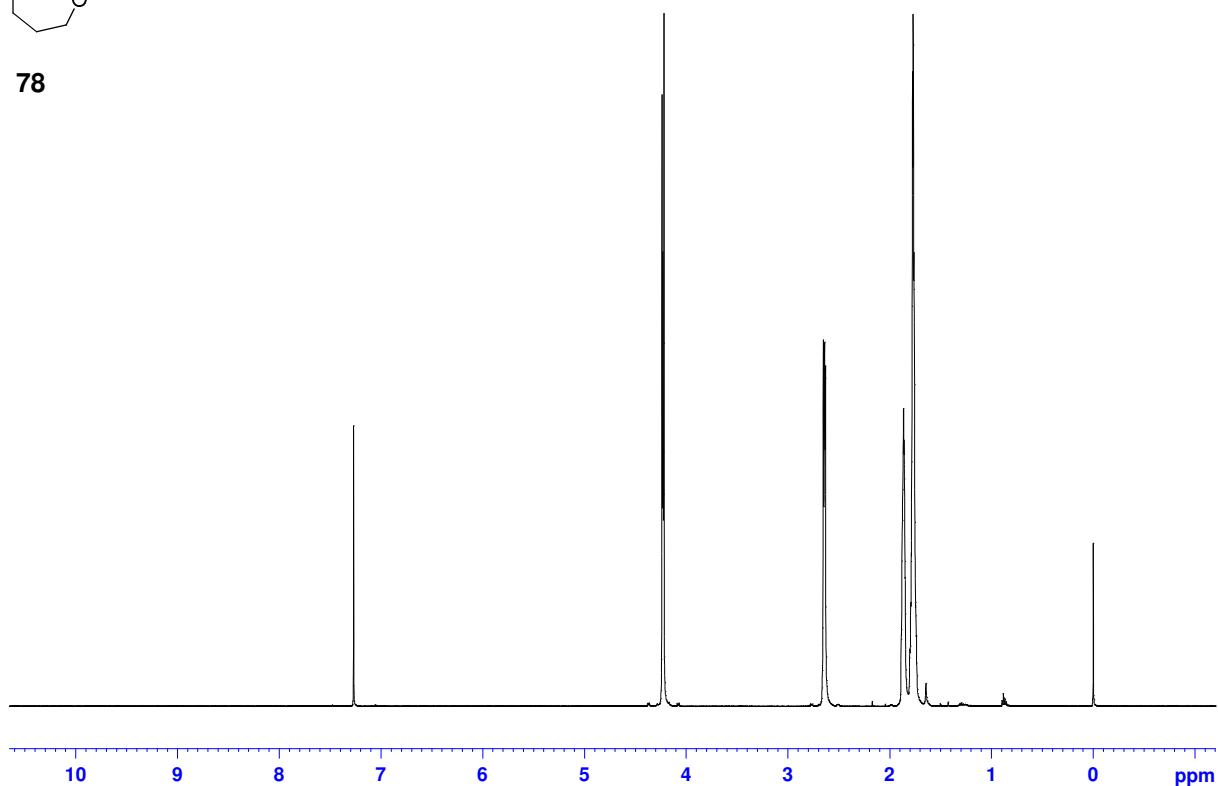


**77**

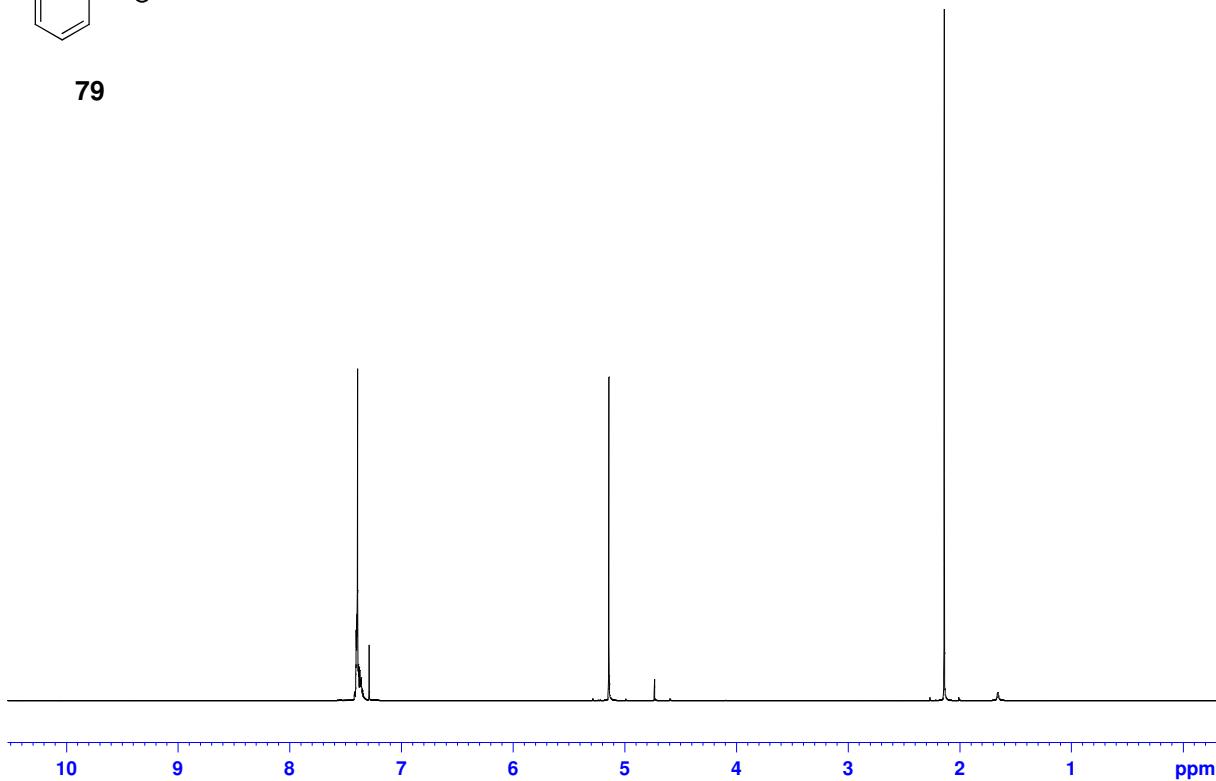


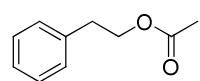


**78**

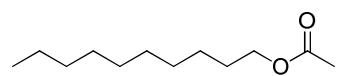
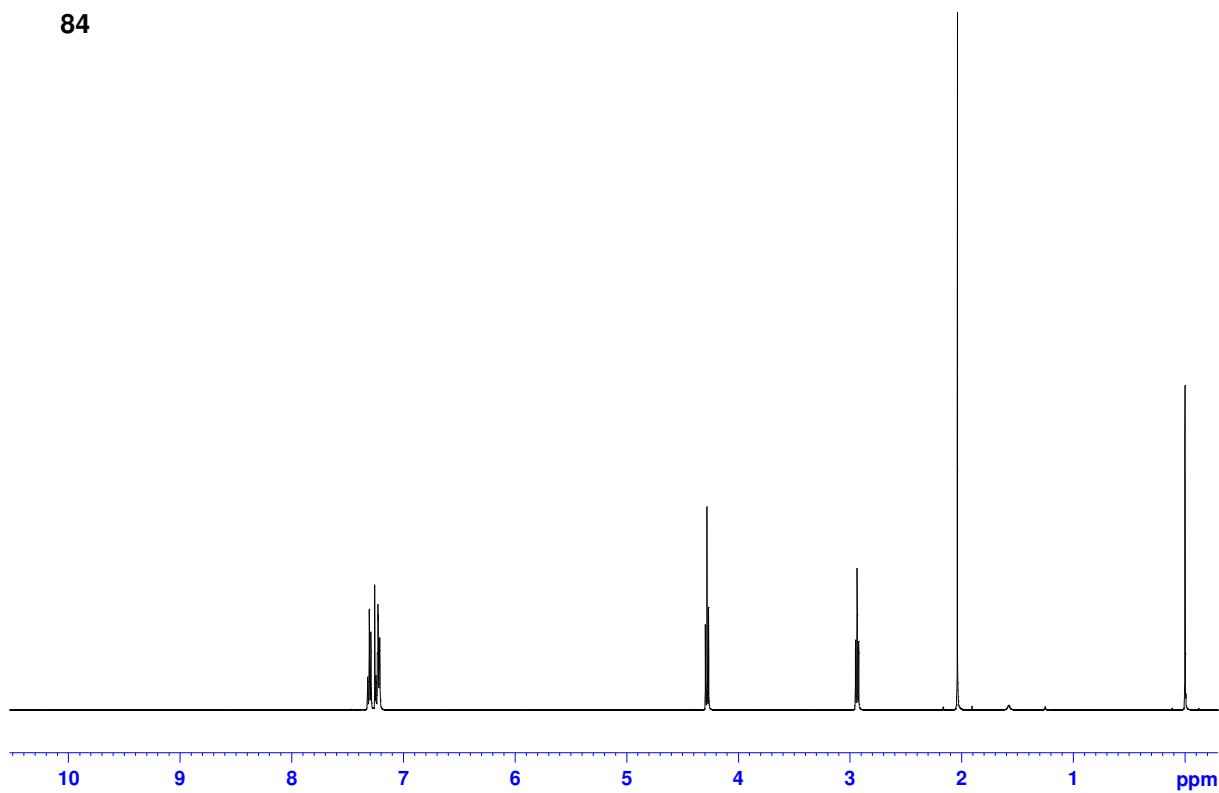


**79**

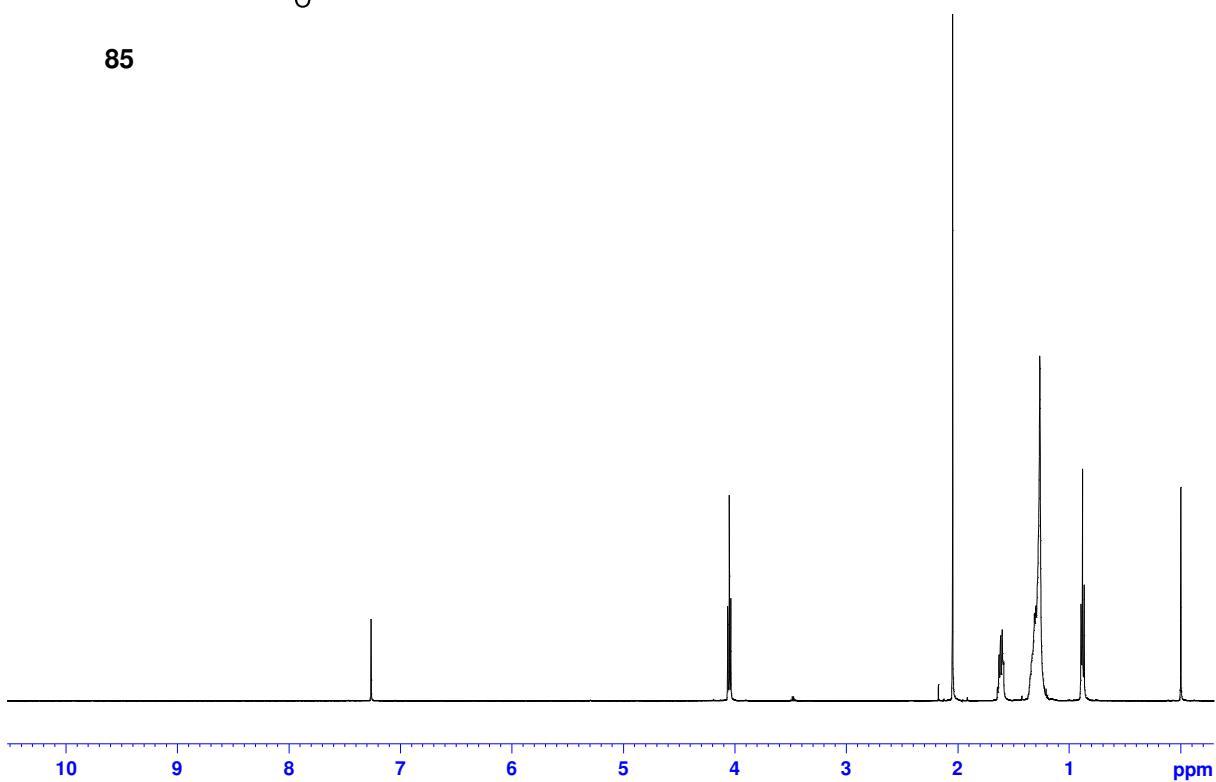


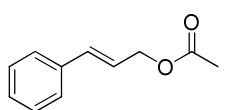


**84**

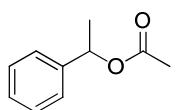
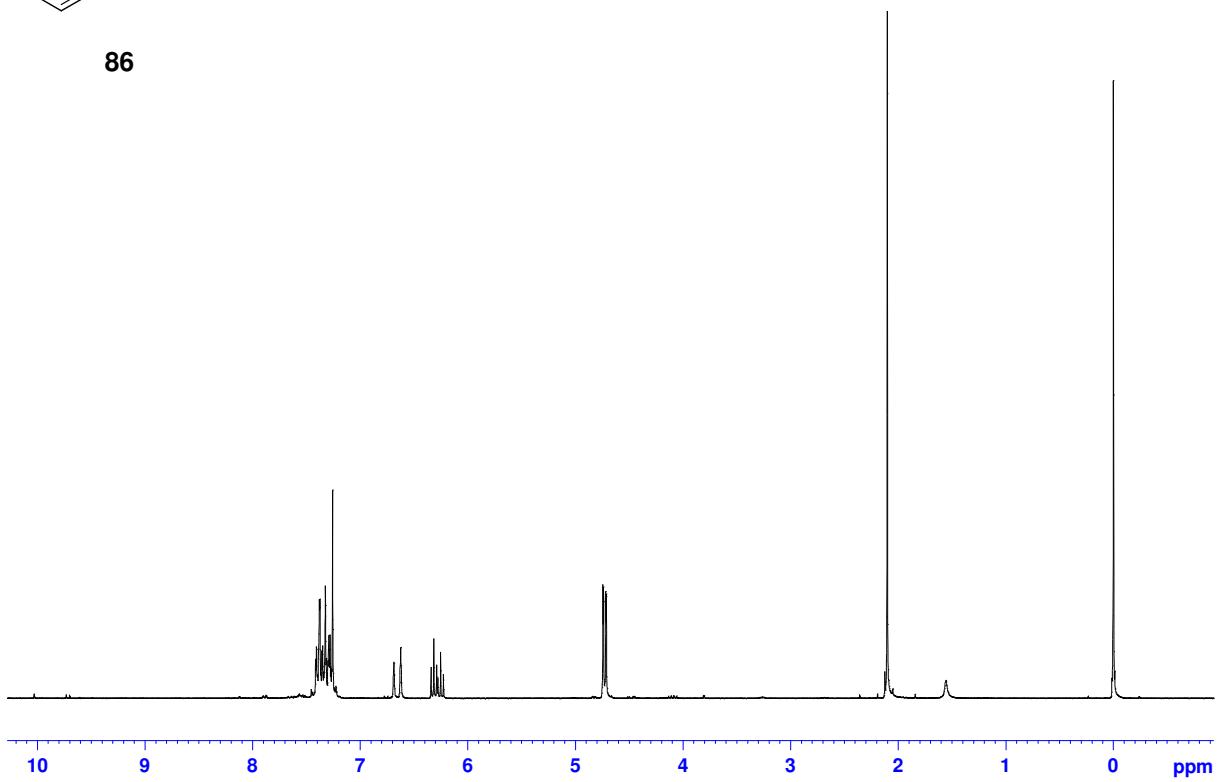


**85**

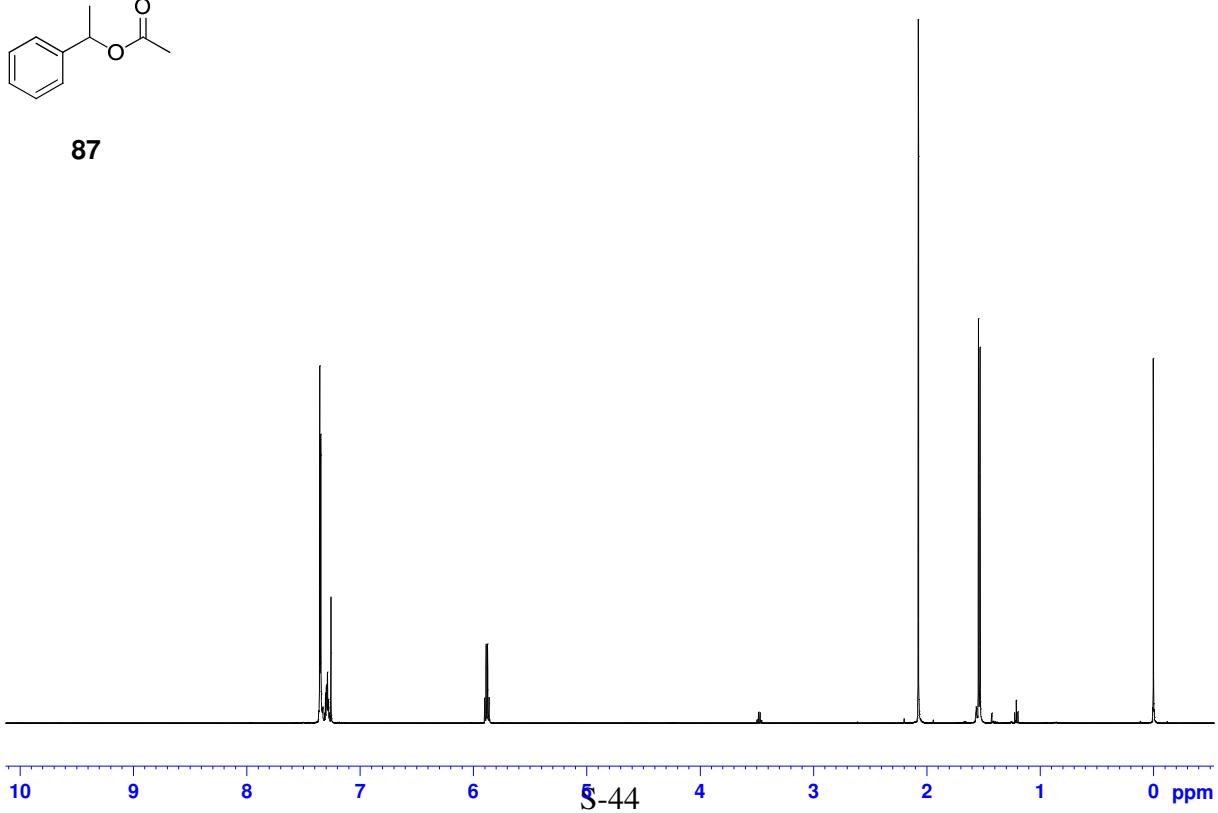


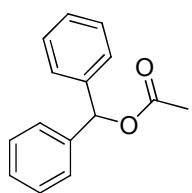


**86**

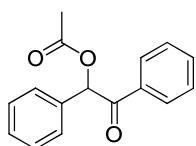
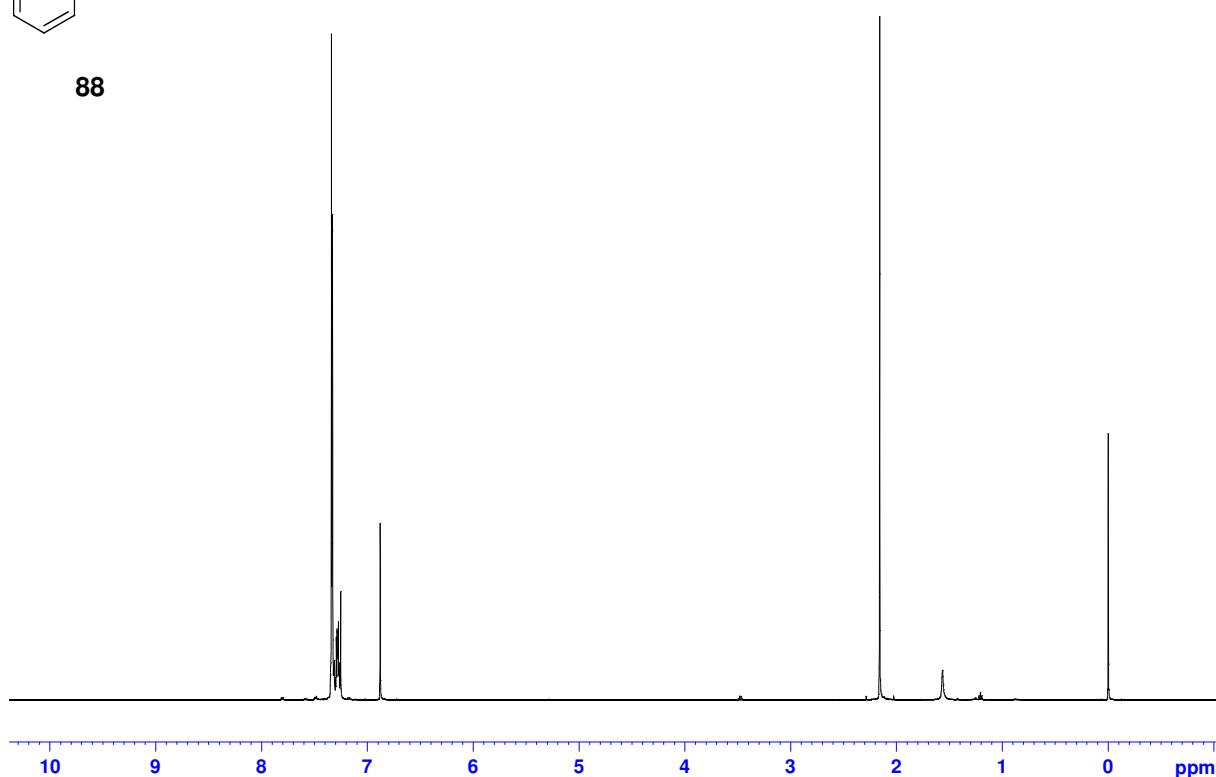


**87**

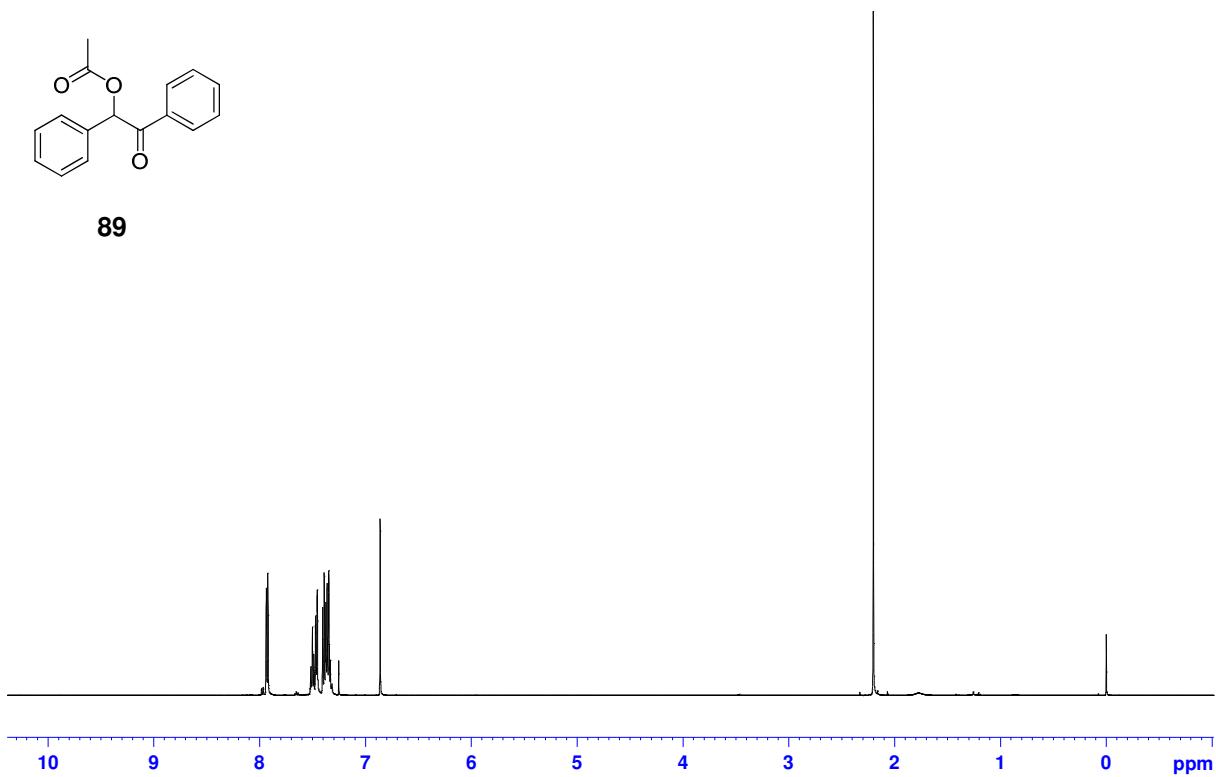


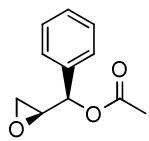


**88**

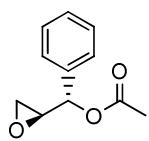
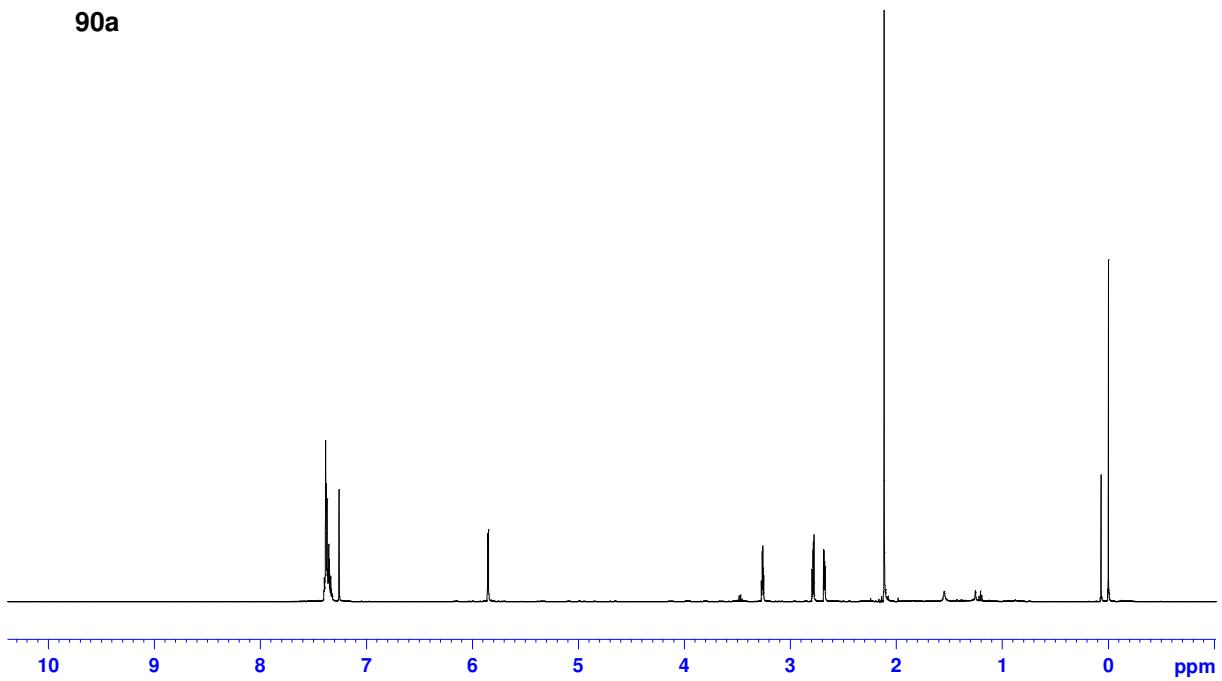


**89**

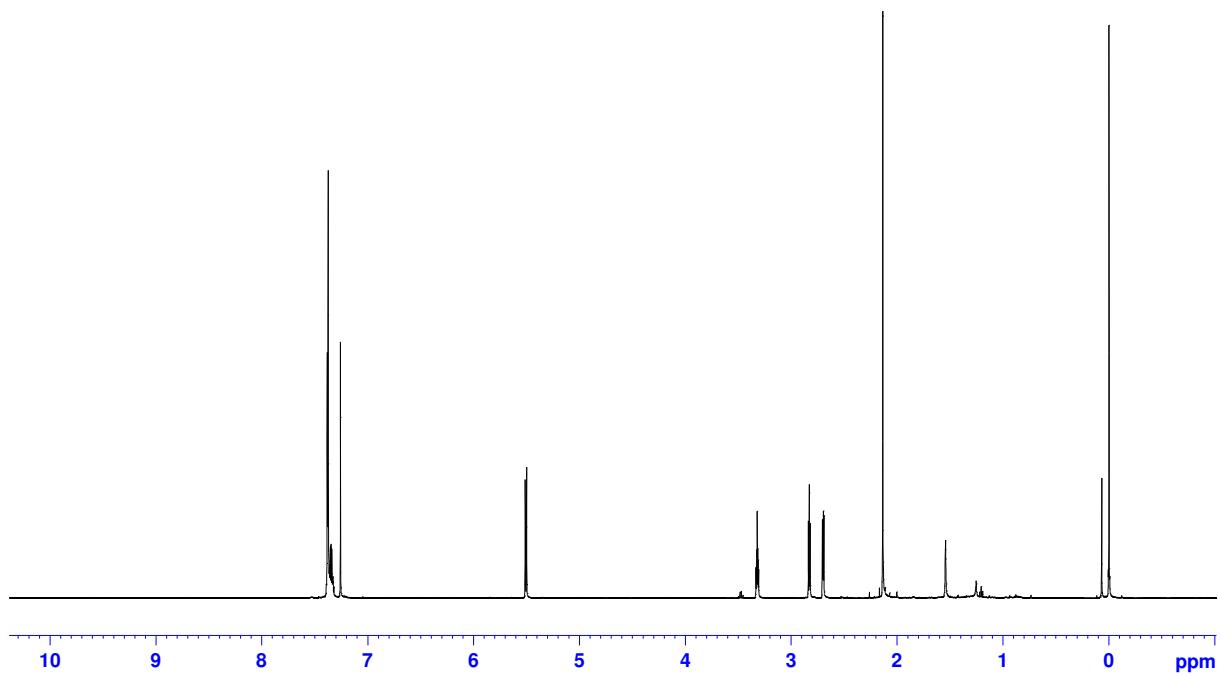


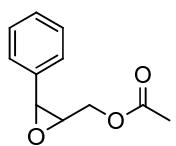


**90a**

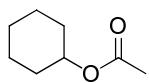
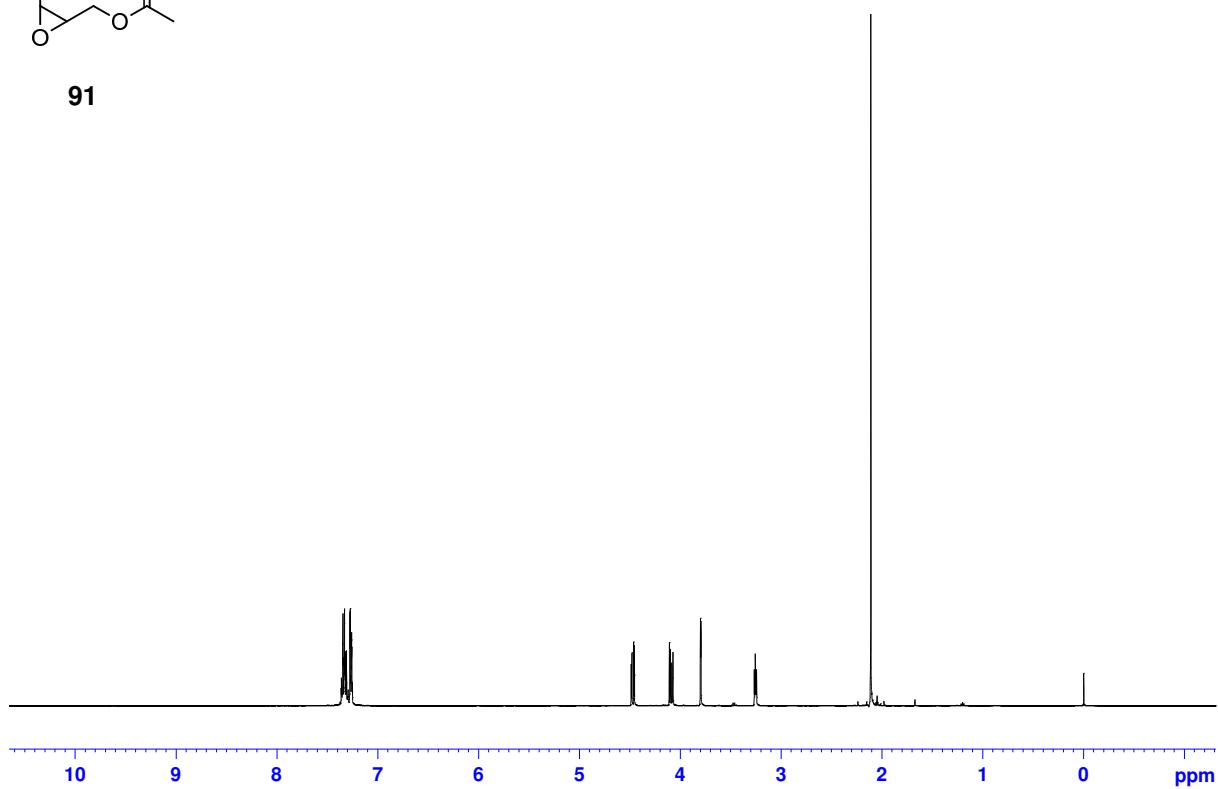


**90b**

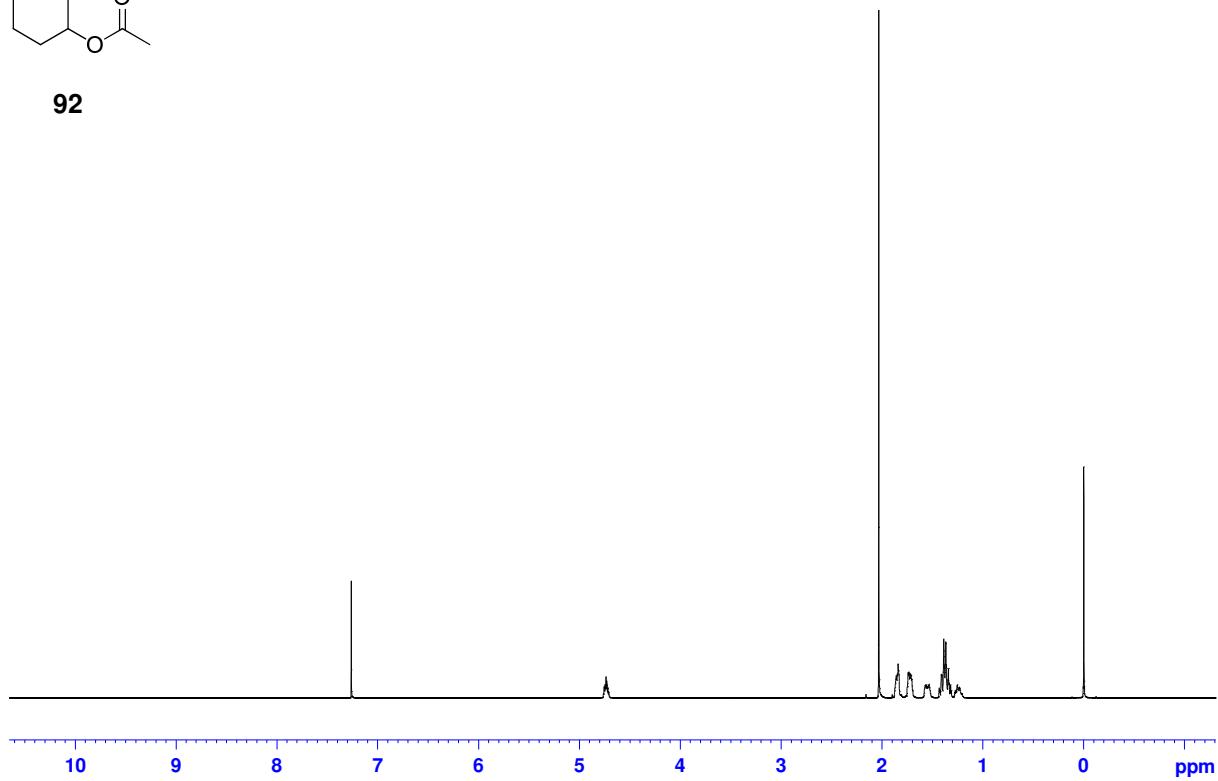


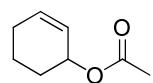


**91**

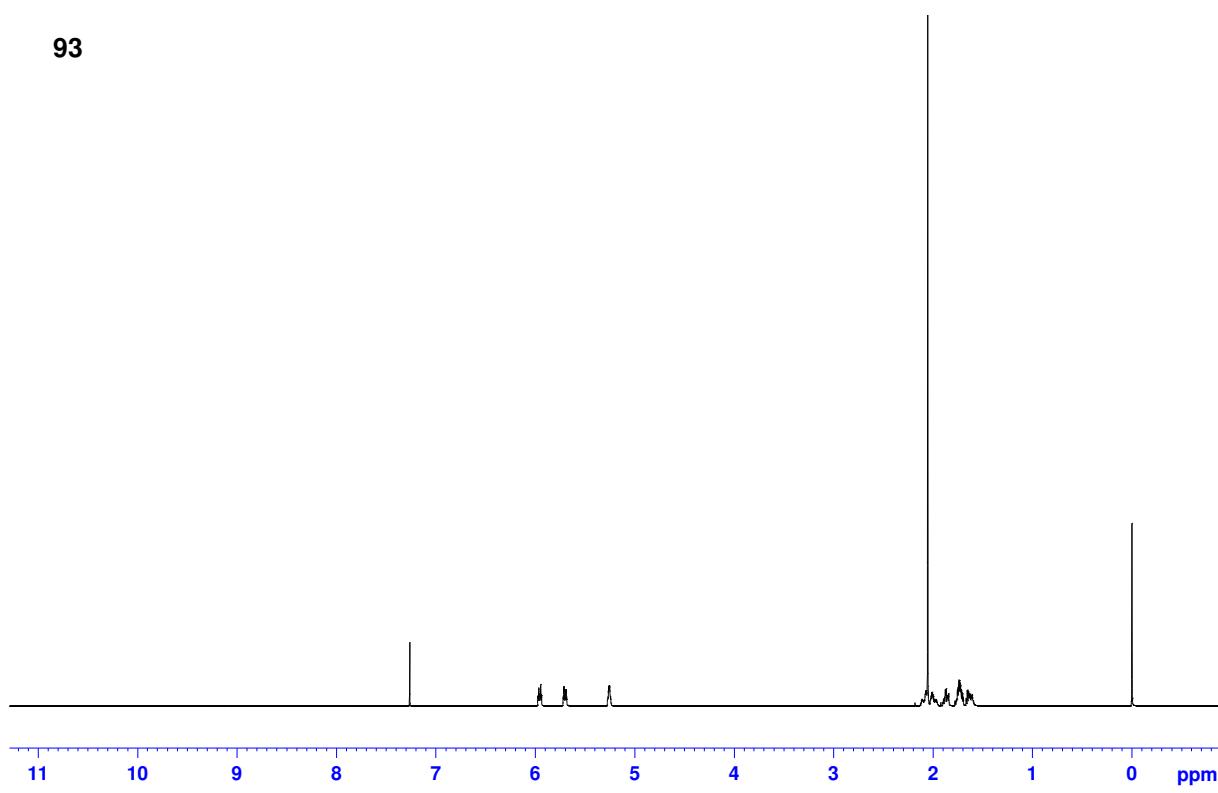


**92**

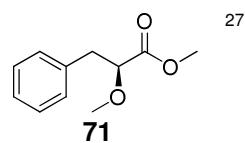




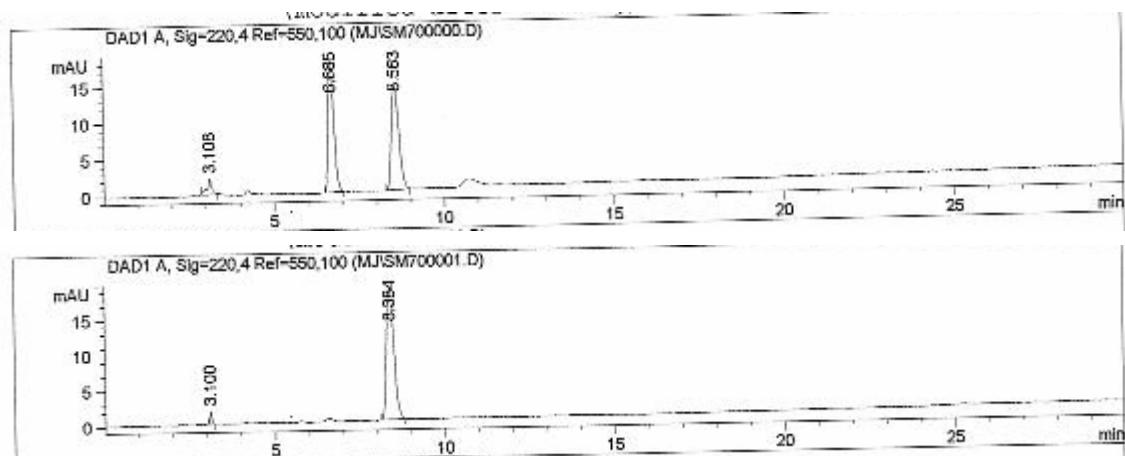
93

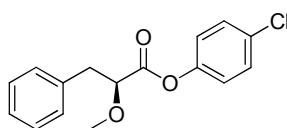


**S-V Chirale HPLC chromatogram of chirale ester**



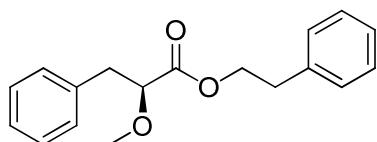
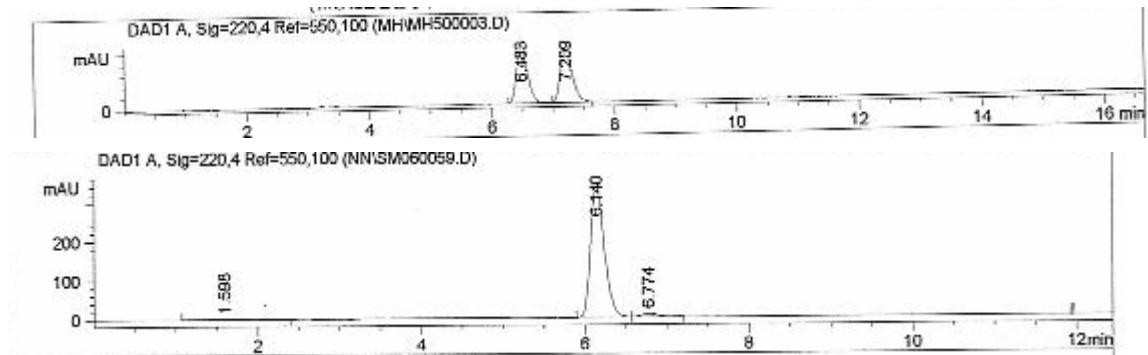
Chiracel OD-H, 95:5 (Heptane/isopropanol) 1ml/min





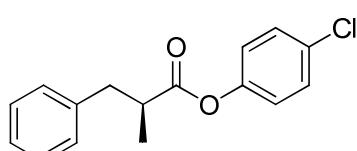
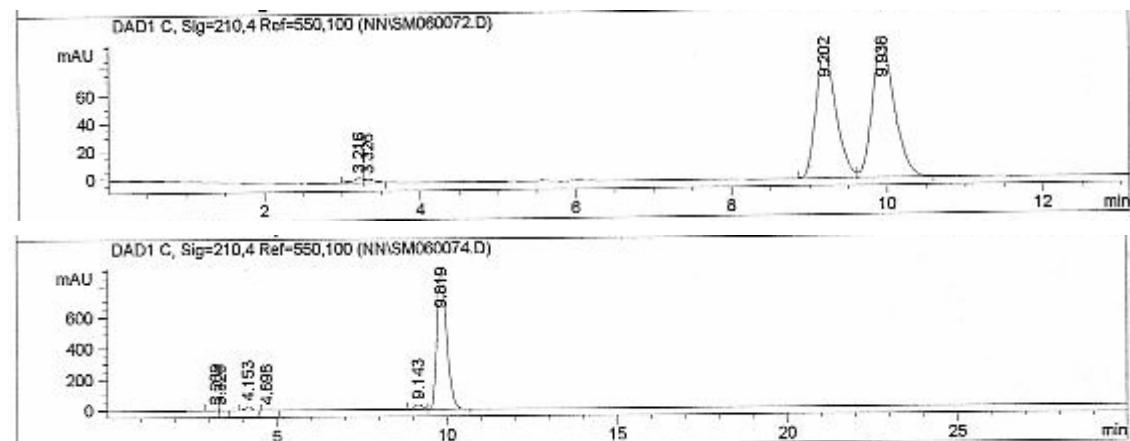
**57**

Chirale OD, 97 :3(Heptane/isopropanol) 1ml/min

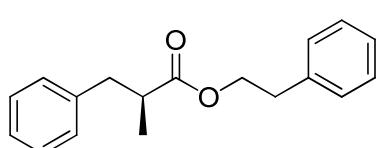


**65**

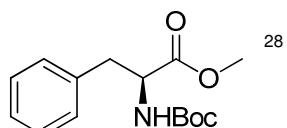
Chirale OD, 96:4 (Heptane/isopropanol) 1ml/min



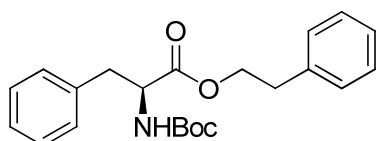
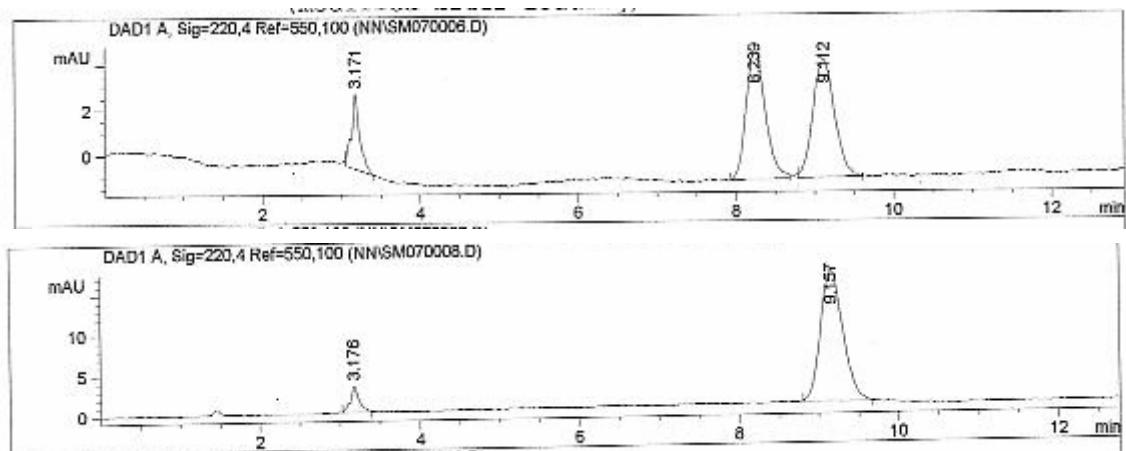
**57**



**66**

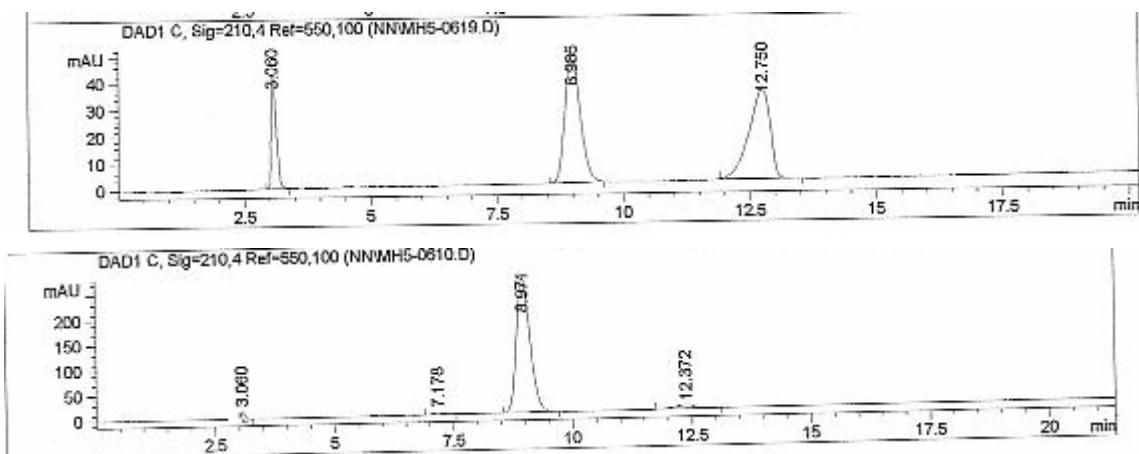


chiracel OD-H, 96:4 (Heptane/isopropanol) 1ml/min



**76**

Chiracel OD-H, 97:1 (Heptane/isopropanol) 1ml/min



- 1 Neises, B.; Steglich, W. *Angew. Chem.* **1978**, *90*, 556, *Angew. Chem., Int. Ed.* **1978**, *17*, 552.
- 2 Fuson T.; *J. Org. Chem.*, **1953**, *18*, 1762.
- 3 Miyashita, M.; Shiina, I.; Miyoshi, S.; Mukaiyama, T.; *Bull. Chem. Soc. Jpn*, 1993, *66*, 1516.
- 4 Hsin, Ling-Wei; Chang, Li-Te; Rothman, Richard B.; Dersch, Christina M.; Jacobson, Arthur E.; Rice, Kenner C.; *J. Med. Chem.*, 2008, *51*, 2795.
- 5 Studte, C.; Breit, B.; *Angew. Chem.* **2008**, *120*, 5531.
- 6 Magens, S.; Ertelt, M.; Jatsch, A.; Plietker, B.; *Org. Lett.*, **2008**, *10*, 53.
- 7 Castle, K.; Hau,C.S.; Sweeney, J.B.; Tindall, C.; *Org. Lett.*, **2003**, *5*, 757.
- 8 Crosignani, S.; White, P. D.; Steinauer, R.; Linclau, B.; *Org. Lett.*, **2003**, *5*, 853.
- 9 Sinha, A. K.; Sharma, A.; Swaroop, A.; Kumar, V.; *Tetrahedron*, **2007**, *63*, 1000.
- 10 Parrish, J. P.; Dueno, E. E.; Kirn, S.-I.; Jung, K. W.; *Synthetic Communications*, **2000**, *30*, 2687.
- 11 Mali, R. S.; Papalkar, A. S.; *Journal of Chemical Research, Synopses*, **2001**, *10*, 433.
- 12 Ohshima, T.; Iwasaki, T.; Maegawa, Y.; Yoshiyama, A.; Mashima, K.; *J. Am. Chem. Soc.*, **2008**, *130*, 2944.
- 13 Akermark, B.; Larsson, E. M.; Oslob, J. D.; *J. Org. Chem.*, **1994**, *59*, 5729.
- 14 Funasaka, S.; Mukaiyama, T.; *Bull. Chem. Soc. Jpn*, **2008**, *81*, 148.
- 15 Hanessian, S.; Gomtsyan, A.; Maike, N.; *J.Org.Chem*,**2000**, *65*, 5623.
- 16 Ye, S.; Tang, Y.; Dai, L.-X.; *J. Org. Chem.*, **2001**, *66*, 5717.
- 17 Liu, X.-G.;Hu, W.-X, *Journal of Chemical Research, Synopses*; **2004**, *8*, 564.
- 18 Suresh, V.; Suryakiran, N.; Venkateswarlu, Y. *Can. J. Chem.*, **2007**, *85*,1037.
- 19 Blaser, H.-U.; Diggelmann, M.; Meier, H.; Naud, F.; Scheppach, E.; Schnyder, A.; Studer, M.; *J. Org. Chem.*, **2003**, *68*, 3725.
- 20 Mehlfuehrer, M.; Berner, H.; Thirring, K.; *J. Chem. Soc., Chem. Commun.* **1994**, *11*, 1291.
- 21 Schomaker, J. M.; Travis, B. R.; Borhan, B.; *Org. Lett.*, **2003**, *5*, 3089.
- 22 Bosco, J. W. J.; Saikia, Anil K.; *Chem Commun.*, **2004**, *19*, 1116.
- 23 Sakai, N.; Moriya, T.; Konakahara, T.; *J. Org. Chem.*, **2007**, *72*, 5920.
- 24 Hattori, K.; Sajiki, H.; Hirota, K.; *Tetrahedron*, **2001**, *57*, 4817.
- 25 Takeshita, M.; Yaguchi, R.; Akutsu, N.; *Tetrahedron: Asymm.*, **1992**; *3*; 1369.
- 26 Man, K. T.; Klawonn, M.; Bhor, S.; Doebler, C.; Anilkumar, G.; Hugl, H.; Maegerlein, W.; Beller, M.; *Org Lett*, **2005**, *7*, 987.
- 27 Heck,R.F; *J. Am. Chem. Soc.*, **1968**, *90*, 5518.
- 28 Jackson, R. F. W.; Moore, R. J.; Dexter, C. S.; Elliott, J.; Mowbray, C. E.; *J. Org. Chem*,**1998**, *63*, 7875.