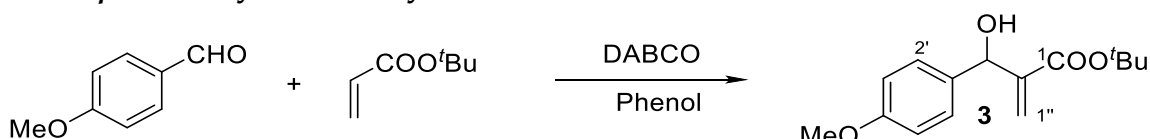


## Materials and Methods

The reactions performed with air sensitive reagents were conducted under dry argon. The solvents were previously distilled under nitrogen over calcium hydride or sodium filaments. Melting points were determined in a Koffler apparatus and are not corrected. NMR spectra were recorded Both proton NMR ( $^1\text{H}$  NMR) and carbon NMR ( $^{13}\text{C}$  NMR) were recorded in deuterated solvents, on spectrometers working on 200 MHz or 400 MHz for proton and 50 MHz or 100 MHz for carbon. Data shown below is represented as follows: chemical shift, multiplicity, coupling constant, integral and assignment. Chemical shifts are reported in ppm with respect to residual solvent protons, coupling constants ( $J_{\text{X-X'}}$ ) are reported in Hz. Mass spectra were taken in Mass spectra was recorded using APCI (Atmospheric Pressure Chemical Ionization). Rotatory power data was recorded using  $\text{CHCl}_3$  as solvent and sodium D line as polarized light ray: IR data was recorded using liquid IR spectrometer and NaCl crystal as supporting material. Silica column chromatography was performed using silica gel 60A (0.060-0.200 mm).

### 1. Baylis-Hillman reaction between *tert*-butyl acrylate and *p*-methoxybenzaldehyde.



Over 1.01 g (7.50 mmol) of *p*-anisaldehyde (CAS: 123-11-5, light yellow liquid), 3.40 g of DABCO (CAS: 280-57-9, white powder) (7.50 mmol), 1.45 g (7.50 mmol) of phenol (CAS: 108-95-2, colourless crystalline solid) and 4.0 mL (46.3 mmol) of *tert*-butyl acrylate (CAS: 1663-39-4, colourless liquid) were added under Ar atmosphere. After 29 days, the reaction crude was dissolved in EtOAc and washed with 1 M HCl,  $\text{H}_2\text{O}$ , 10% aqueous  $\text{NaHCO}_3$ ,  $\text{H}_2\text{O}$  and saturated aqueous NaCl. Then, 10 mL of a 40%  $\text{NaHSO}_3$  aqueous solution were added to the organic phase and then stirred for 2 hours. The phases were decanted and the organic phase was washed with  $\text{H}_2\text{O}$  three times and saturated aqueous NaCl and dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and the solvent was evaporated, obtaining 1.77 g (6.20 mmoles), 82.0% of **3**.

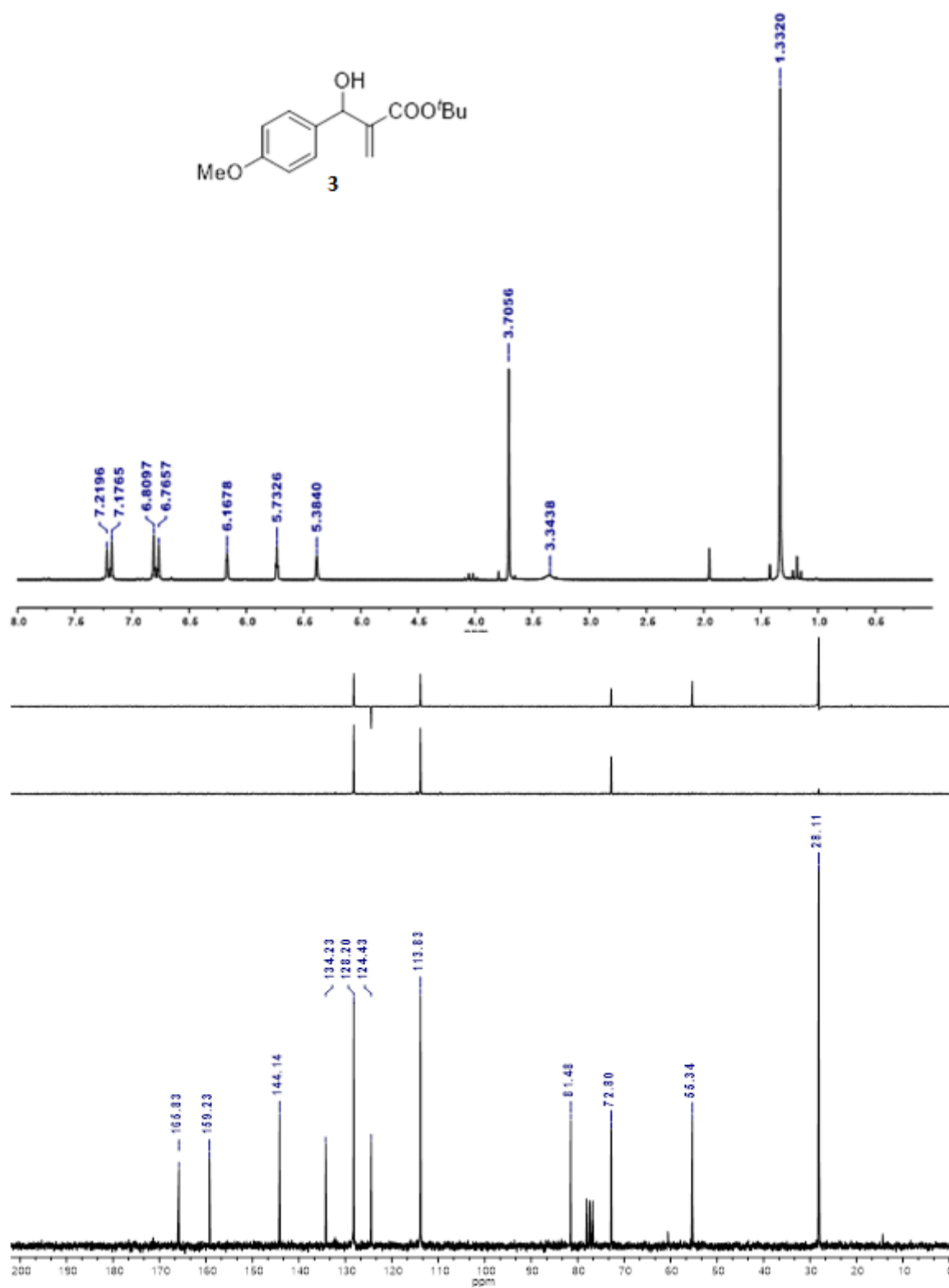
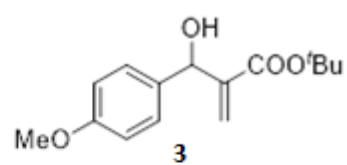
**3**: *tert*-Butyl 3-(4-methoxyphenyl)-3-hydroxy-2-methylene-propanoate.

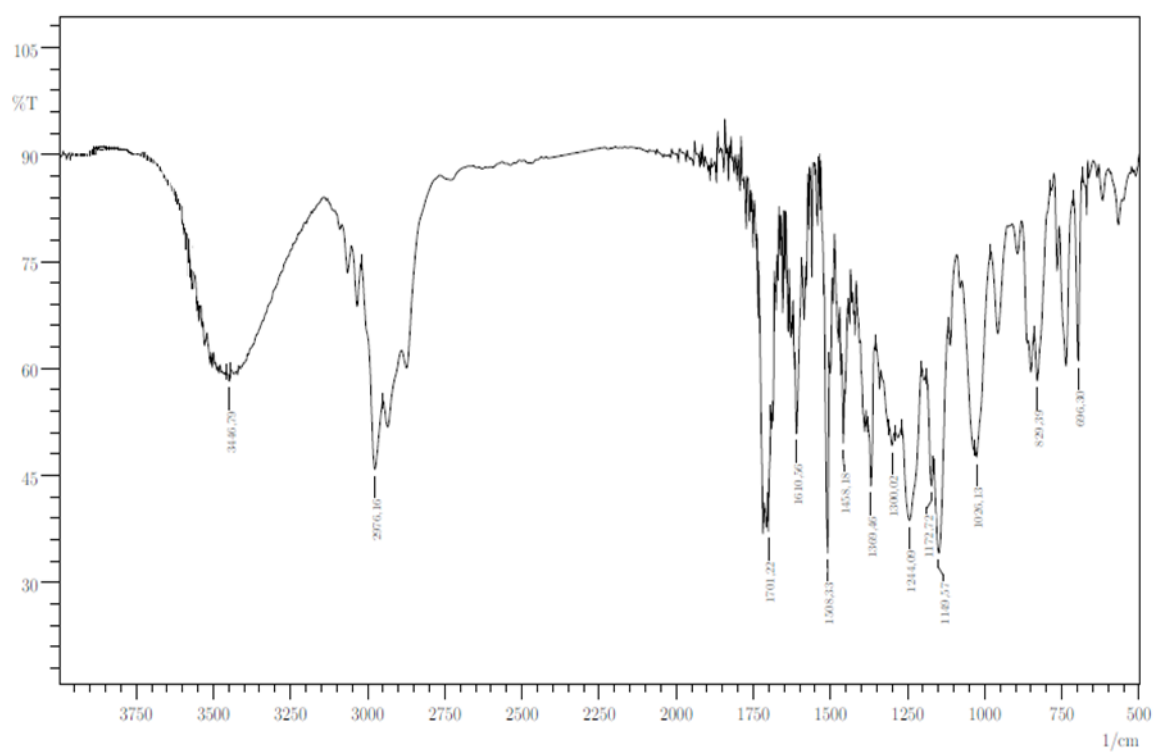
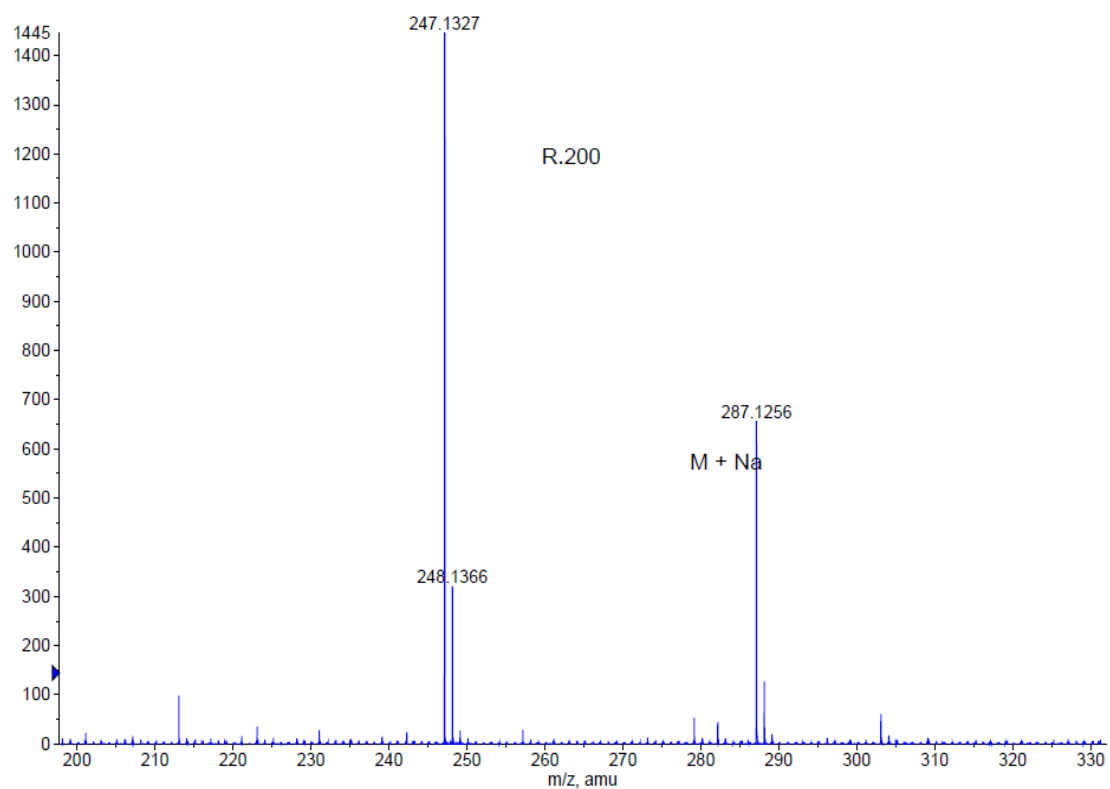
$^1\text{H}$  NMR (200 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.20 (d,  $J = 8.7$  Hz, 2H, H-2'), 6.79 (d,  $J = 8.7$  Hz, 2H, H-3'), 6.17 (s, 1H, H-1''B), 5.73 (s, 1H, H-1''A), 5.38 (s, 1H, H-3), 3.71 (s, 3H, -OMe), 1.33 (s, 9H, -*t*Bu).

$^{13}\text{C}$  NMR (50 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 165.83 (C,  $\text{COO}^t\text{Bu}$ ), 159.23 (C, C-4'), 144.14 (C, C-2), 134.23 (C, C-1'), 128.20 (CH, C-2'), 124.43 ( $\text{CH}_2$ , C-1''), 113.82 (CH, C-3'), 81.47 (C, *t*Bu), 72.80 (CH, C-3), 55.34 ( $\text{CH}_3$ , OMe), 28.10 ( $\text{CH}_3$ ,  $\text{C}(\text{CH}_3)_3$ ).

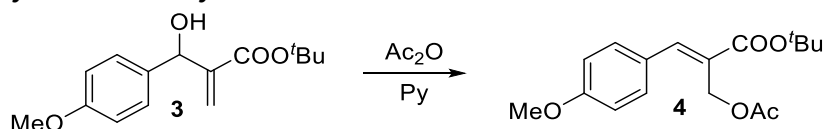
IR $_{\text{max}}$  (film) ( $\text{cm}^{-1}$ ) 831, 851, 1034, 1150, 1173, 1252, 1304, 1369, 1393, 1458, 1512, 1605, 1701, 2978.

E.M.A.R., FAB, calculated for  $\text{C}_{15}\text{H}_{20}\text{O}_4$  [ $\text{M} + \text{Na}$ ]: 287.1253. Found: 287.1256  $\Delta = 0.76$  ppm.





## 2. Acetylation of Baylis-Hillman adduct 3.



4 mL (49.50 mmol) of pyridine (CAS: 110-86-1, colourless liquid) and 3 mL (31.50 mmol) of acetic anhydride (CAS: 108-24-7, colourless liquid) were added to 1.56 g (5.92 mmol) of **3** under Ar atmosphere and at 0 °C. The ice bath was then removed and the mixture was allowed to stir at room temperature. After 72 hours, the reaction crude was hydrolysed with ice. It was extracted with EtOAc and washed with 1 M HCl, H<sub>2</sub>O, 5% aqueous NaHCO<sub>3</sub>, H<sub>2</sub>O and saturated aqueous NaCl. The organic phase was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. The solvent was evaporated obtaining 1,68 g of crude reaction that were chromatographed on silica "flash". The desired product was eluted with hexane/AcOEt 95: 5, 1.58 g of **4** (87%) were obtained.

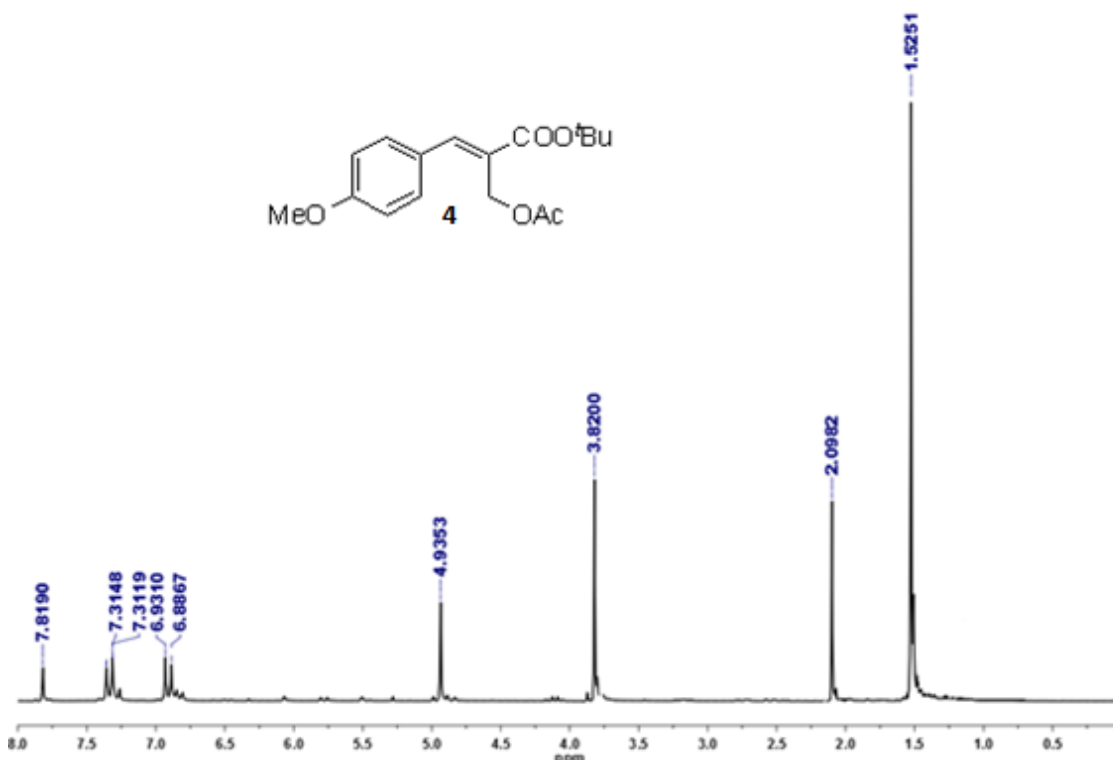
**4**: *tert*-butyl (*E*)-2-(acetoxymethyl)-3-(4-methoxyphenyl)acrylate.

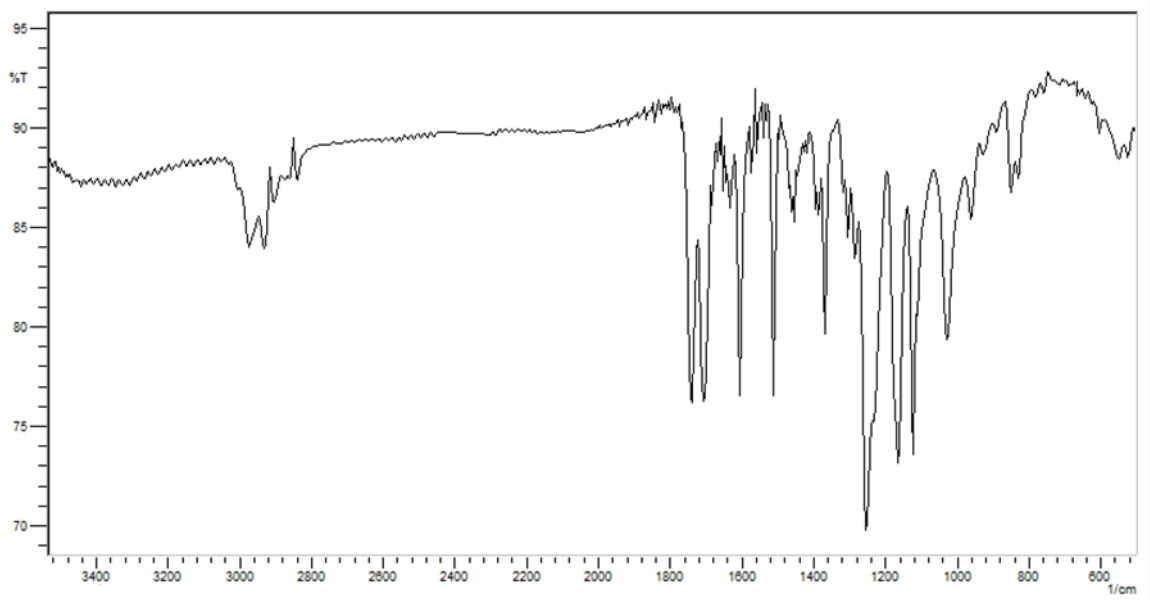
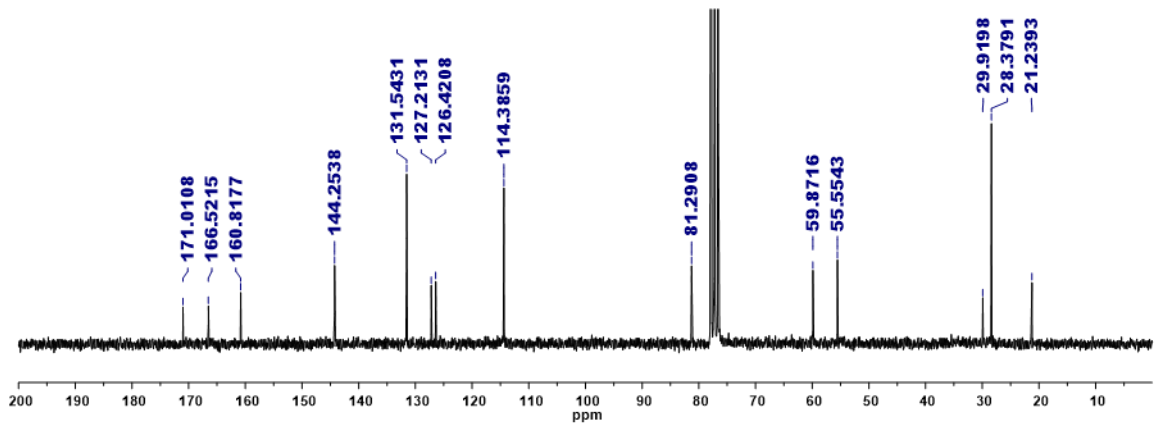
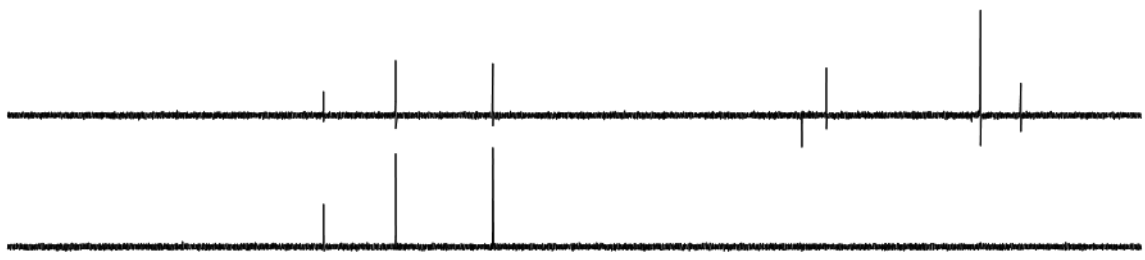
<sup>1</sup>H NMR (200 MHz, CDCl<sub>3</sub>) δ (ppm): 7.33(d, *J* = 8.8 Hz, 2H, H-2'), 6.91 (d, *J* = 8.8 Hz, 2H, H-3'), 7.82 (s, 1H, H-3), 4.94 (s, 2H, H-1''), 3.82 (s, 3H, -OMe), 2.10 (s, 3H, -OAc), 1.53 (s, 9H, COOtBu).

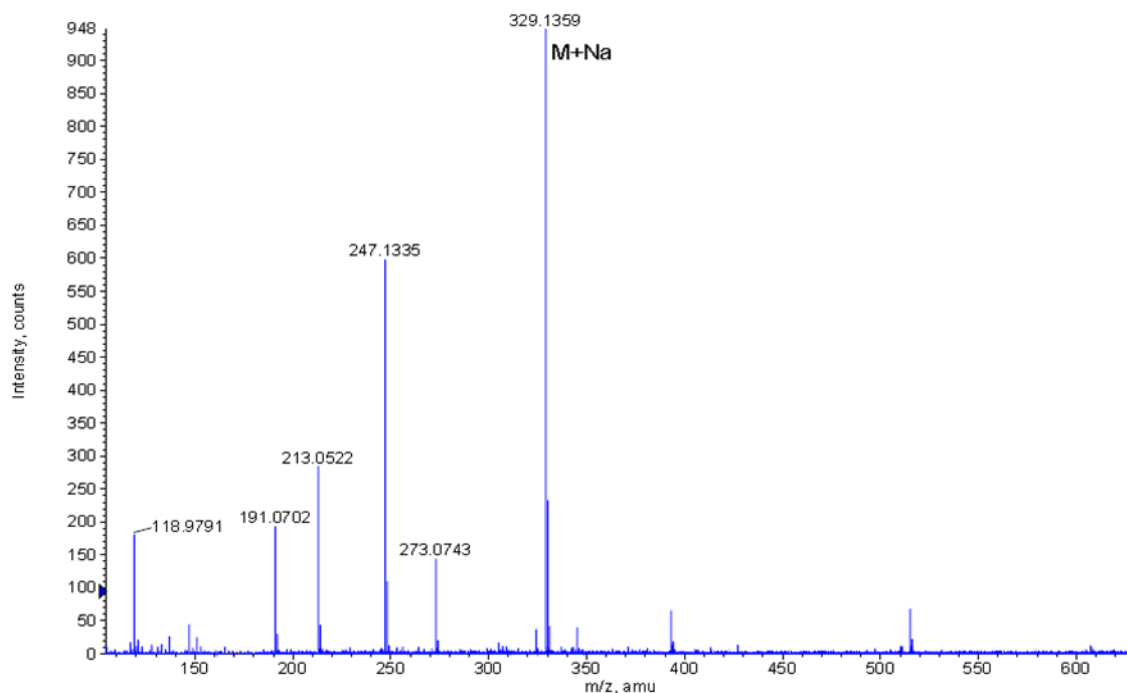
<sup>13</sup>C NMR (50 MHz, CDCl<sub>3</sub>) δ (ppm): 171.62 (C, OCOMe), 166.52 (C, COOtBu), 160.82 (C, C-4'), 144.25 (C, C-2'), 131.54 (C, C-3), 127.21 (CH, C-1'), 126.42 (C, C-2'), 114.39 (CH, C-3'), 81.29 (C, C(CH<sub>3</sub>)<sub>3</sub>), 59.87 (CH<sub>2</sub>, C1''), 55.55 (CH<sub>3</sub>, -OMe), 28.38 (CH<sub>3</sub>, C(CH<sub>3</sub>)<sub>3</sub>), 21.24 (CH<sub>3</sub>, OCOMe).

IR<sub>max</sub> (film) (cm<sup>-1</sup>): 829, 1028, 1150, 1175, 1202, 1231, 1252, 1288, 1395, 1512, 1611, 1699, 1719, 2936, 2978.

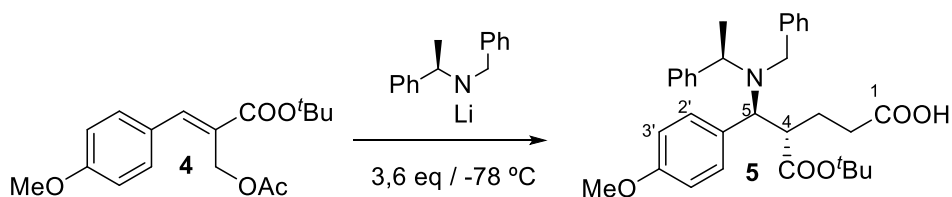
E.M.AR., FAB, calcd for C<sub>17</sub>H<sub>22</sub>O<sub>5</sub> [M + Na]: 329.1359. Found: 329.1360. Δ = -0.14 ppm.







### 3. Addition of chiral amide (R)-2 to 4.



On a (*R*)-( $\alpha$ -methylbenzyl)benzylamine solution, 1.41 g (6.69 mmol) in 5 mL of dry THF, under Ar atmosphere, in a heart flask, 3.94 mL (6.32 mmol) of *n*-BuLi were carefully added (CAS: 109-72-8, 1.6 M commercial solution in hexanes) at  $-78\text{ }^{\circ}\text{C}$ . The mixture was stirred for 15 minutes. Then, it was transferred to an ice bath for another 15 minutes. After this time, the reaction mixture was cooled back to  $-78\text{ }^{\circ}\text{C}$ . In parallel, 0.569 g of **4** (1.58 mmol) was dissolved in 5 mL of dry THF and cooled to  $-78\text{ }^{\circ}\text{C}$  under Ar atmosphere. The solution from the heart flask was then added via "cannula" onto the dissolved substrate. The temperature was maintained at  $-78\text{ }^{\circ}\text{C}$  and it was allowed to stir for an hour. Subsequently, 3 mL of saturated ammonium chloride were added and the mixture was allowed to stir until it reached room temperature. THF was evaporated under reduced pressure. The reaction mixture was extracted with EtOAc and washed with 10% aqueous citric acid,  $\text{H}_2\text{O}$  and saturated aqueous NaCl. The organic phase was dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and the solvent was evaporated, obtaining 0.835 g, which were chromatographed on flash silica and eluted with hexane/EtOAc 9:1 to obtain 0.563 g of **5** (59%).

**5**: (4*S*,5*S*)-5-(Benzyl ((*R*)-1-phenylethyl) amino)-4-(*tert*-butoxycarbonyl)-5-(4-methoxyphenyl)-pentanoic acid.

$[\alpha]_{\text{D}}^{20} = -62.3$  (c 1.24,  $\text{CHCl}_3$ ).

**$^1\text{H}$  NMR** (200 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm)\*: 7.54-7.16 (m, 12H, ArH), 6.98 (d,  $J = 8.3$  Hz, 2H, H-2'), 4.14 (q,  $J = 6.3$  Hz, 1H, C( $\alpha$ )H), 3.98 (AB,  $J_{\text{AB}} = 13.7$  Hz, 1H,  $\text{NCH}_\text{B}\text{HPh}$ ), 3.80 (m, 1H, H-5), 3.83 (s, 3H, OMe), 3.57 (AB,  $J_{\text{AB}} = 13.7$  Hz, 1H,  $\text{NCH}_\text{A}\text{HPh}$ ), 3.04 (td, 1H,  $J = 10.8$  Hz and 3.0 Hz, H-4), 2.92-2.60 (m, 1H, H-3<sub>A</sub>), 2.20-2.10 (m, 2H, H-2), 1.61-1.58 (m, 1H, H-3<sub>B</sub>), 1.04 (s, 9H,  $\text{COOtBu}$ ), 0.98 (d,  $J = 6.7$  Hz, 3H, C( $\alpha$ )H).

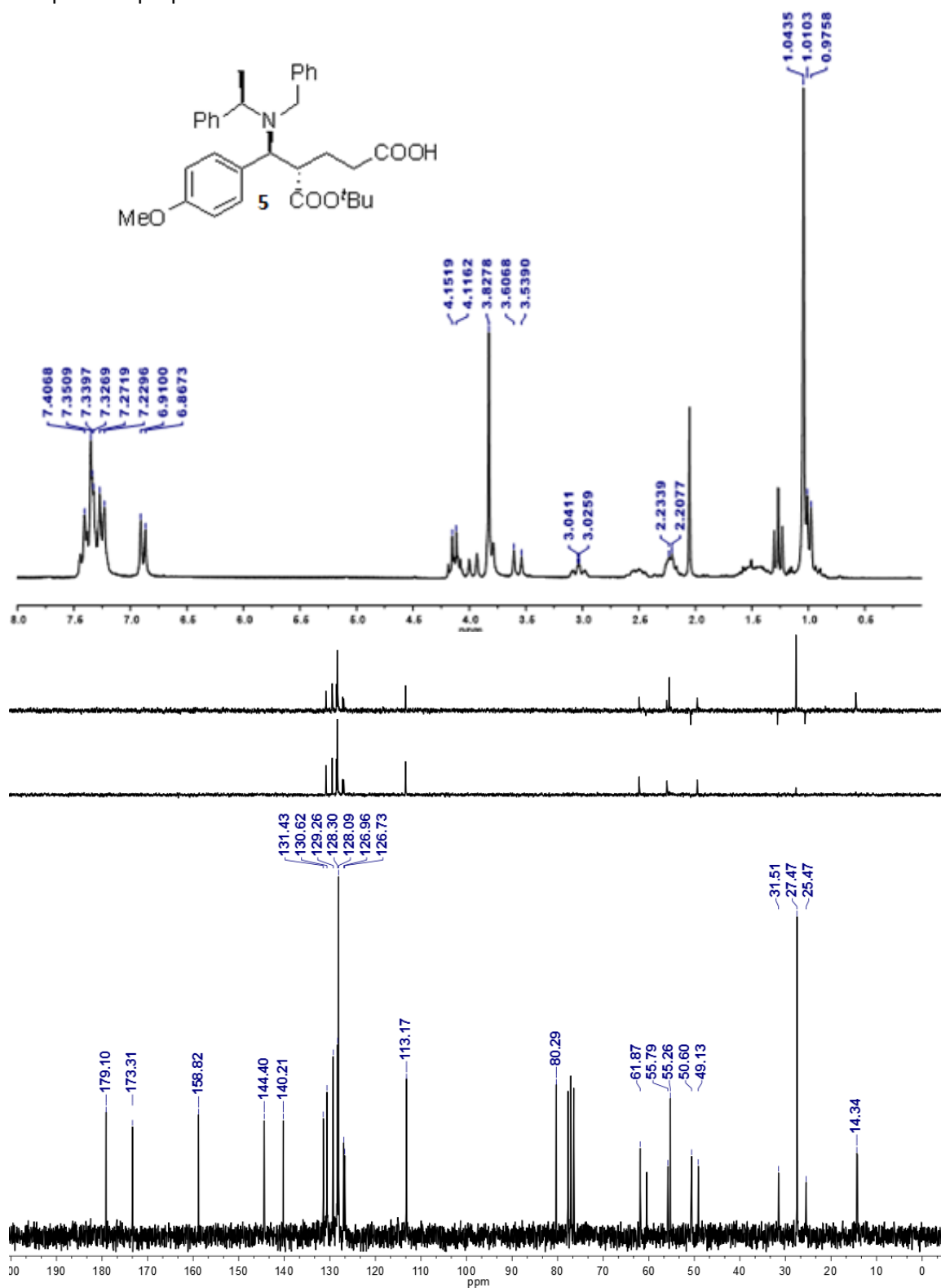
**$^{13}\text{C}$  NMR** (50 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm)\*: 179.10 (C, COOH), 173.31 (C,  $\text{COOtBu}$ ), 158.82 (C, C-4'), 144.40 (C,  $\text{C}_{\text{ipso}}$ ), 140.21 (C,  $\text{C}_{\text{ipso}}$   $\text{CH}_2\text{Ph}$ ), 131.43 (C, C-1'), 130.62 (CH, Ar), 129.26 (CH, Ar), 128.30 (CH, Ar), 128.09 (CH, Ar), 126.96 (CH, Ar), 126.73 (CH, C-2'), 113.17 (CH, C-3'), 80.29 (C,

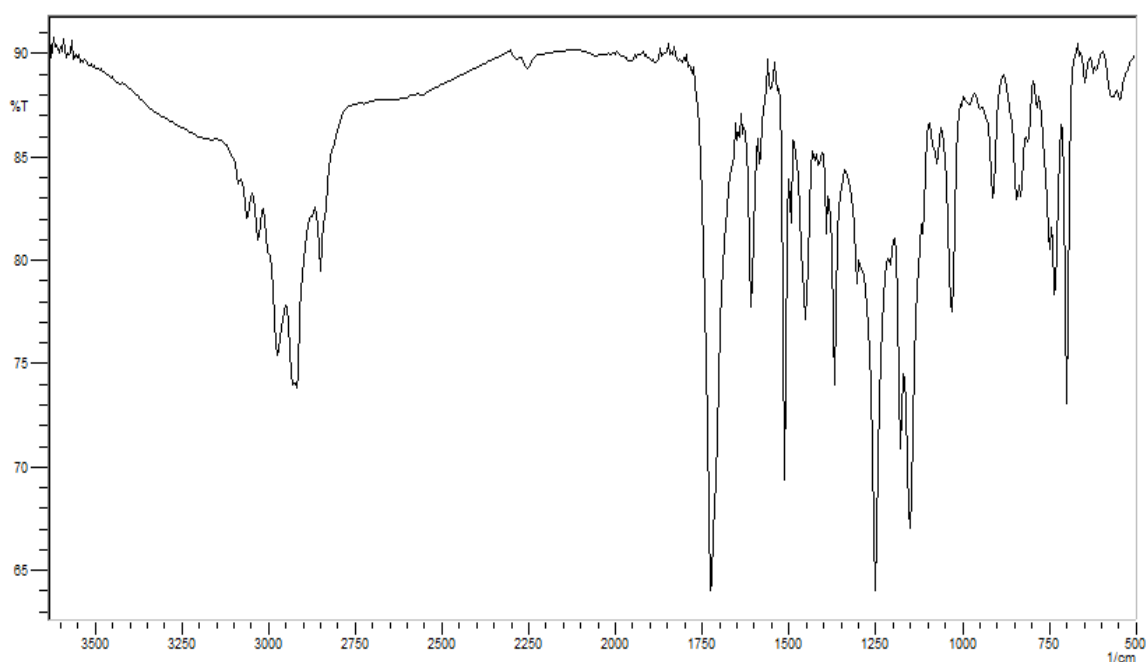
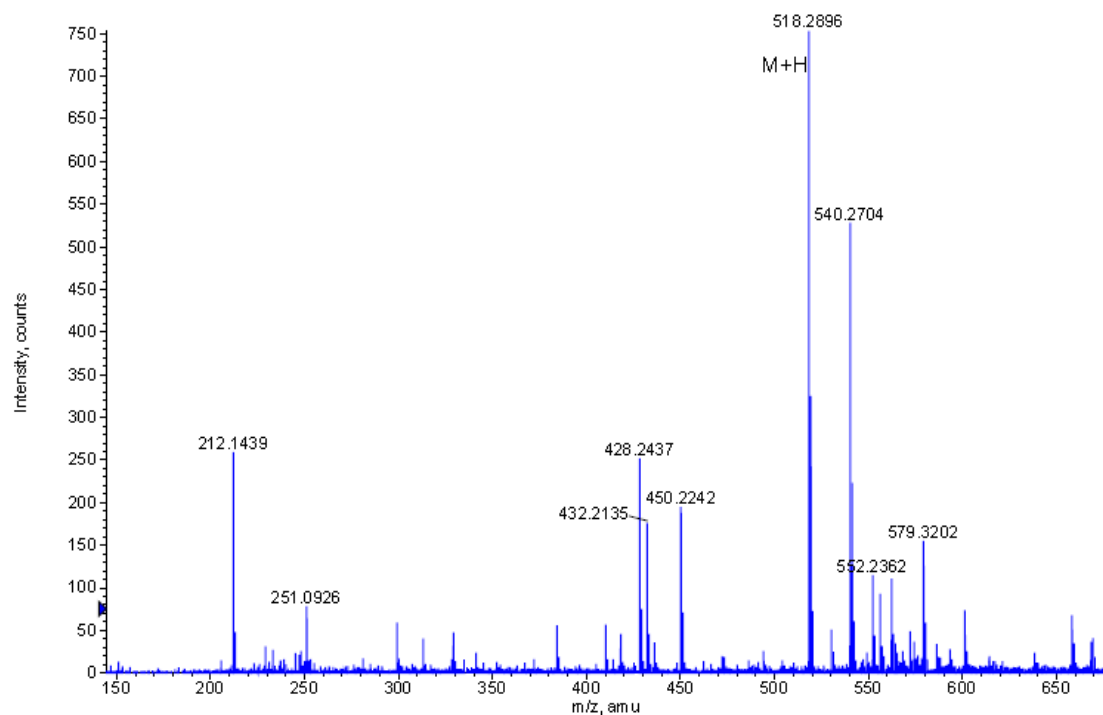
$\text{C(CH}_3)_3$ ), 61.95 (CH, C-5), 55.79 (CH, C( $\alpha$ )), 55.26 (CH<sub>3</sub>, OCH<sub>3</sub>), 50.60 (CH<sub>2</sub>, NCH<sub>2</sub>Ph), 49.13 (CH, C-4), 31.51 (CH<sub>2</sub>, C-2), 27.47 ((CH<sub>3</sub>, C(CH<sub>3</sub>)<sub>3</sub>), 25.47 (CH<sub>2</sub>, C-3), 14.34 (CH<sub>3</sub>, C( $\alpha$ )Me).

**IR**<sub>max</sub> (film) (cm<sup>-1</sup>): 700, 733, 1030, 1150, 1179, 1207, 1250, 1304, 1368, 1452, 1512, 1609, 1724, 2849, 2928, 2974.

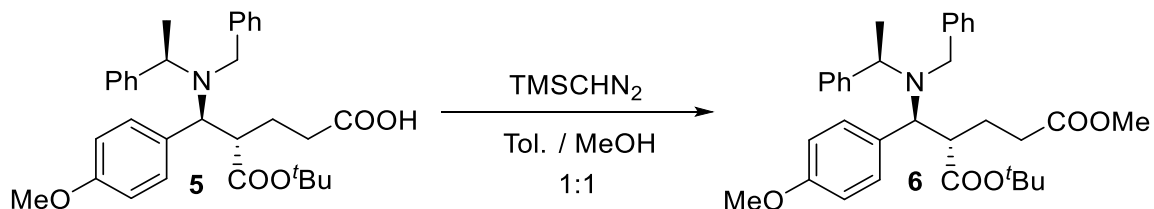
**E.M.A.R.**, FAB, calcd for C<sub>32</sub>H<sub>40</sub>NO<sub>5</sub> [M+H]: 518.2901 Found: 518.2896  $\Delta$  = -0.77 ppm.

\*Compounds **6-8** keep same numeration than **5** in the spectroscopic assignation for comparative purposes.





#### 4. Reaction of $\delta$ -amino acid **5** with trimethylsilyldiazomethane.



To a solution of **5** (57,6 mg; 0,111 mmol) in 2 mL of toluene-methanol 1:1, 90  $\mu\text{L}$  (0,183 mmol) of trimethylsilyldiazomethane (CAS: 18107-18-1, commercial solution 2M in hexanes, yellow) were added. The mixture was allowed to stir at room temperature for 30 minutes. Then, the crude was evaporated and the desired product was obtained, 59 mg of **6** (100%).

**6:** 1-(*tert*-butyl) 5-methyl (*S*)-2-((*S*)-(benzyl(*R*)-1-phenylethyl)amino)(4-methoxyphenyl)methyl)glutarate

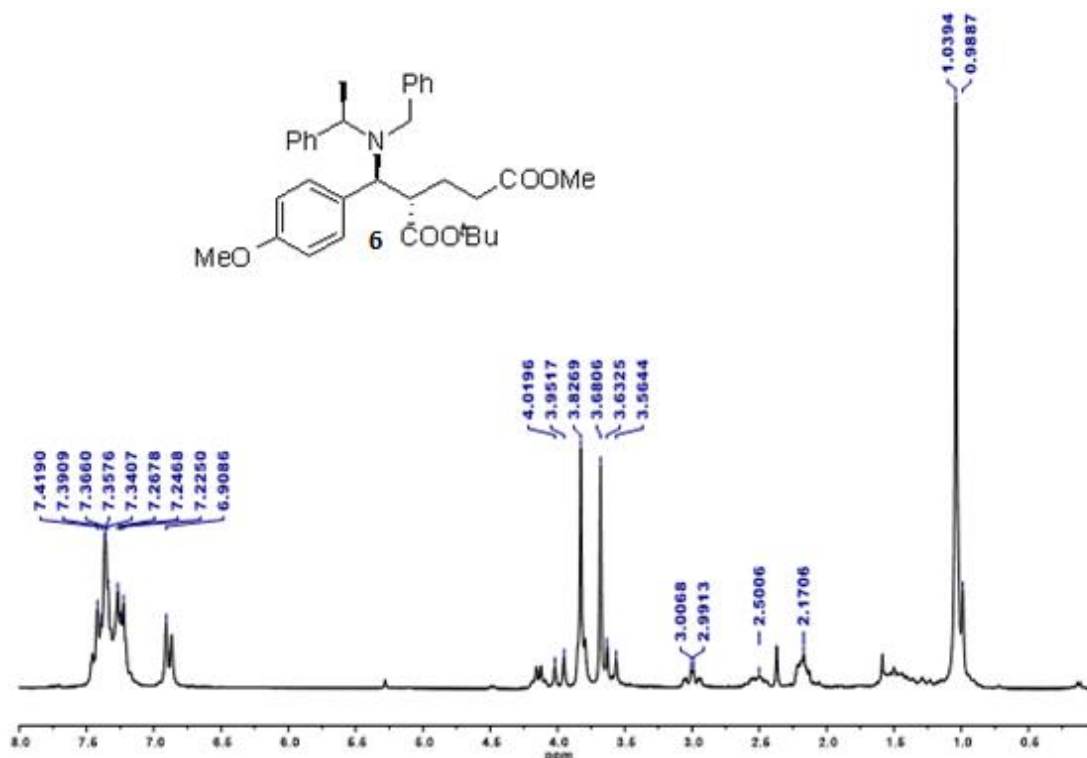
$[\alpha]_D^{20} = -54.3$  (c 1.02, CHCl<sub>3</sub>).

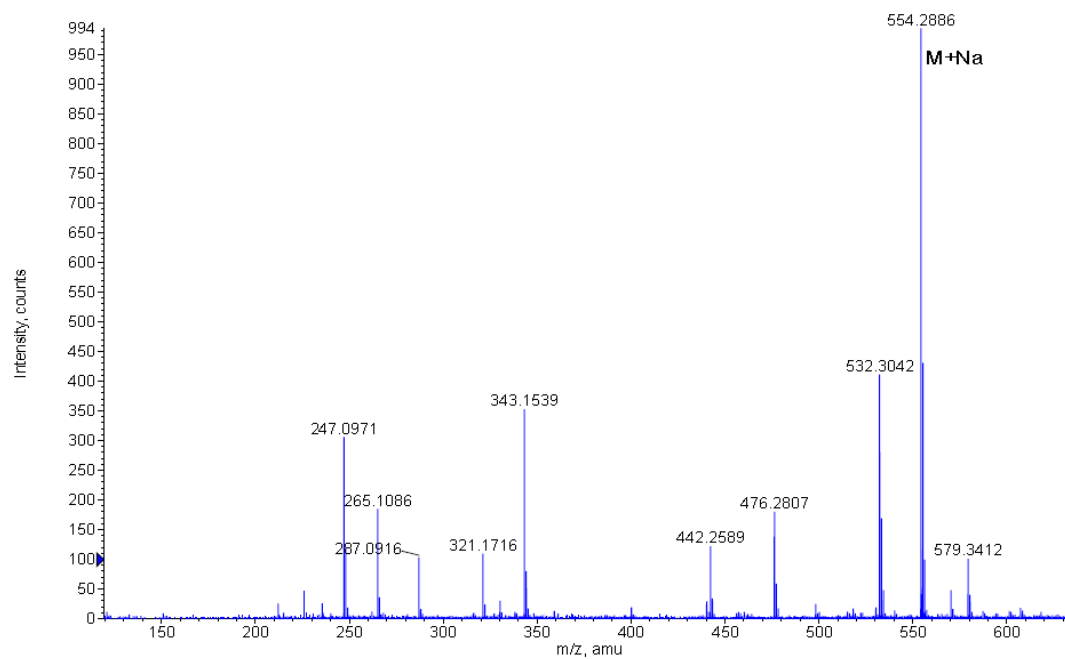
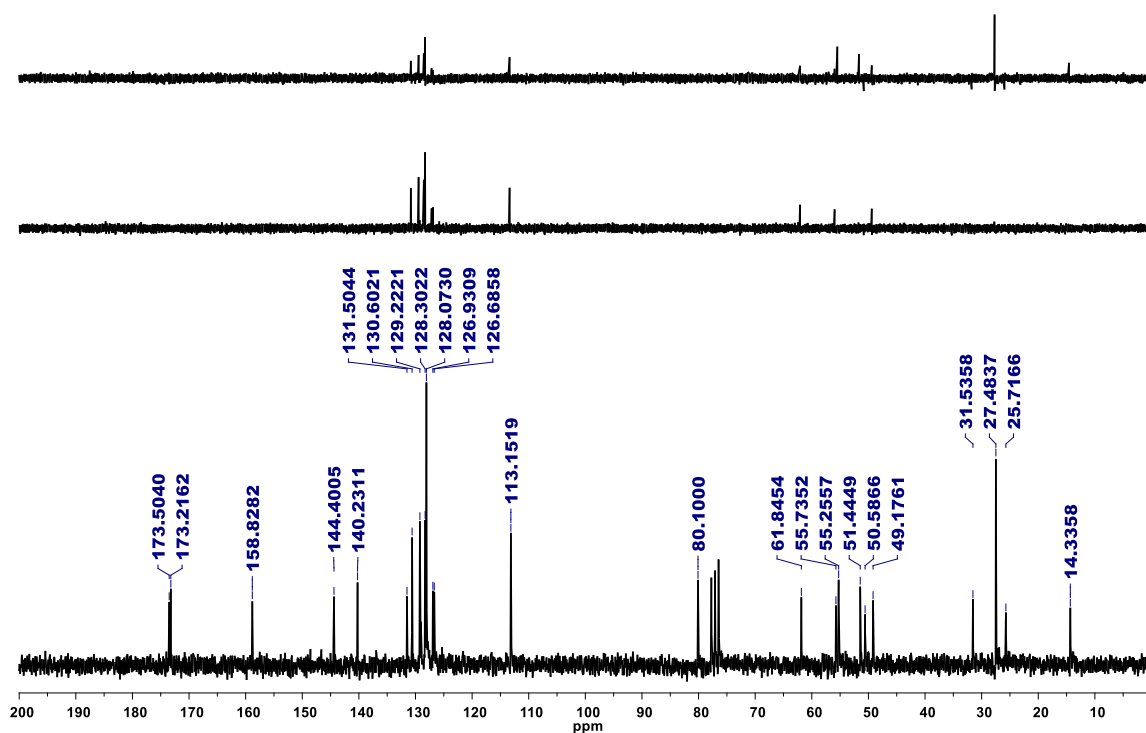
**<sup>1</sup>H NMR** (200 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 7.58-7.02 (m, 12H, ArH), 6.89 (d,  $J = 8.3$  Hz, 2H, H-3'), 4.14 (q,  $J = 6.5$  Hz, 1H, C( $\alpha$ )H), 3.97 (AB,  $J_{AB} = 13.7$  Hz, 1H, NCH<sub>B</sub>HPh), 3.83 (s, 3H, OMe), 3.82 (d, 1H,  $J = 10.8$  Hz, H-5), 3.68 (s, 3H, COOMe), 3.58 (AB,  $J_{AB} = 13.7$  Hz, 1H, NCH<sub>A</sub>HPh), 2.98 (td,  $J = 11.0$ , 3.0 Hz, 1H, H-4), 2.71-2.43 (m, 1H, H-3<sub>A</sub>), 2.23-2.08 (m, 2H, H-2), 1.76-1.48 (m, 1H, H-3<sub>B</sub>), 1.02 (s, 9H, *t*Bu), 0.97 (s, 3H, C( $\alpha$ )H).

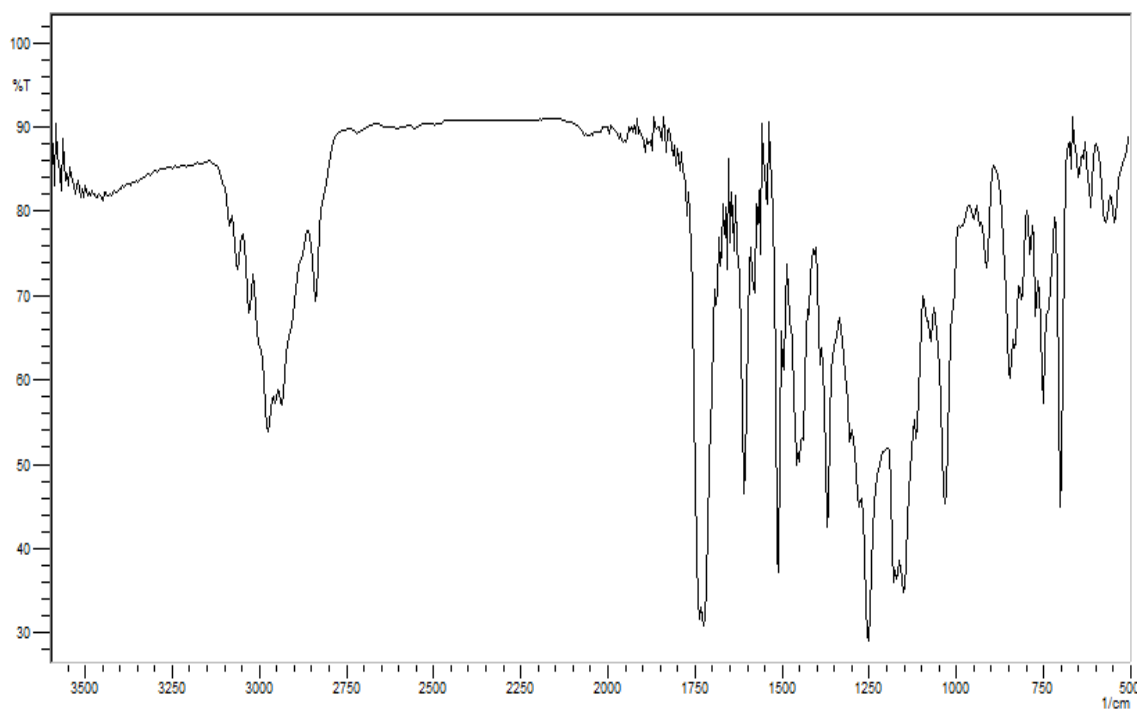
**<sup>13</sup>C NMR** (50 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 173.51 (C, COOMe), 173.22 (C, COO*t*Bu), 158.83 (C, C-4'), 144.40 (C, C<sub>ipso</sub> CHPh), 140.23 (C, C<sub>ipso</sub> CH<sub>2</sub>Ph), 131.50 (C, C-1'), 130.60 - 126.60 (CHx12, Ar), 113.15 (CH, C-2'), 80.10 (C, C(CH<sub>3</sub>)<sub>3</sub>), 61.85 (CH, C-5), 55.74 (CH, C( $\alpha$ )), 55.24 (CH<sub>3</sub>, OCH<sub>3</sub>), 51.45 (CH<sub>3</sub>, COOMe), 50.59 (CH<sub>2</sub>, NCH<sub>2</sub>Ph), 49.18 (CH, C-4), 31.54 (CH<sub>2</sub>, C-2), 27.49 (CH<sub>3</sub>, C(CH<sub>3</sub>)<sub>3</sub>), 25.72 (CH<sub>2</sub>, C-3), 14.34 (CH<sub>3</sub>, C( $\alpha$ )Me).

**IR**<sub>max</sub> (film) (cm<sup>-1</sup>): 700.16, 1031.92, 1151.50, 1176.58, 1251.80, 1367.53, 1454.33, 1512.19, 1606.70, 1697.36, 1714.72, 1728.22, 2927.94

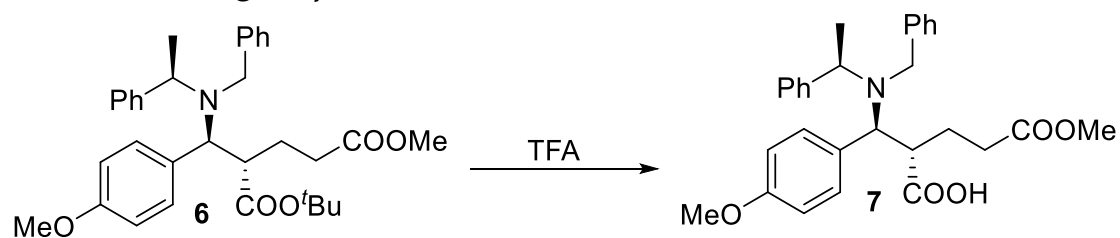
**E.M.AR.**, FAB, calcd for C<sub>33</sub>H<sub>41</sub>O<sub>5</sub>N [M+Na]: 554.2877. Found: 554.2886.  $\Delta = 1.63$  ppm.







## 5. Obtaining the $\beta$ -amino acid **7**.



0.9 mL of trifluoroacetic acid (CAS: 76-05-1, colourless liquid) were added to 50 mg of **6** (0.094 mmol) and the mixture was allowed to stir at room temperature for 4 h. After this time the volatile compounds were evaporate. The crude was dissolved in EtOAc and washed with 5% aqueous  $\text{NaHCO}_3$  and saturated aqueous NaCl. The organic phase was dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and the solvent was evaporated, obtaining 42 mg of **7** (95%).

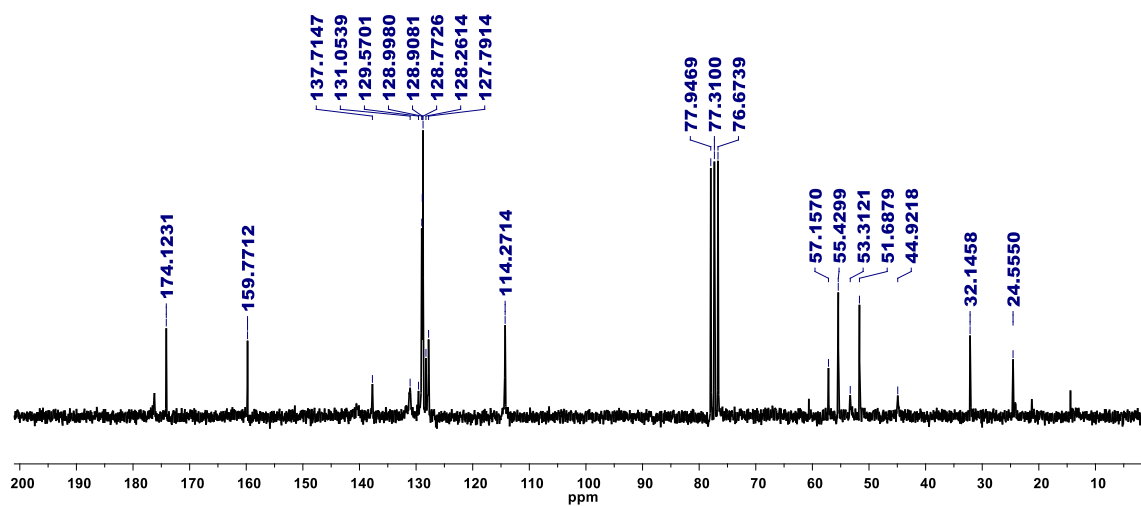
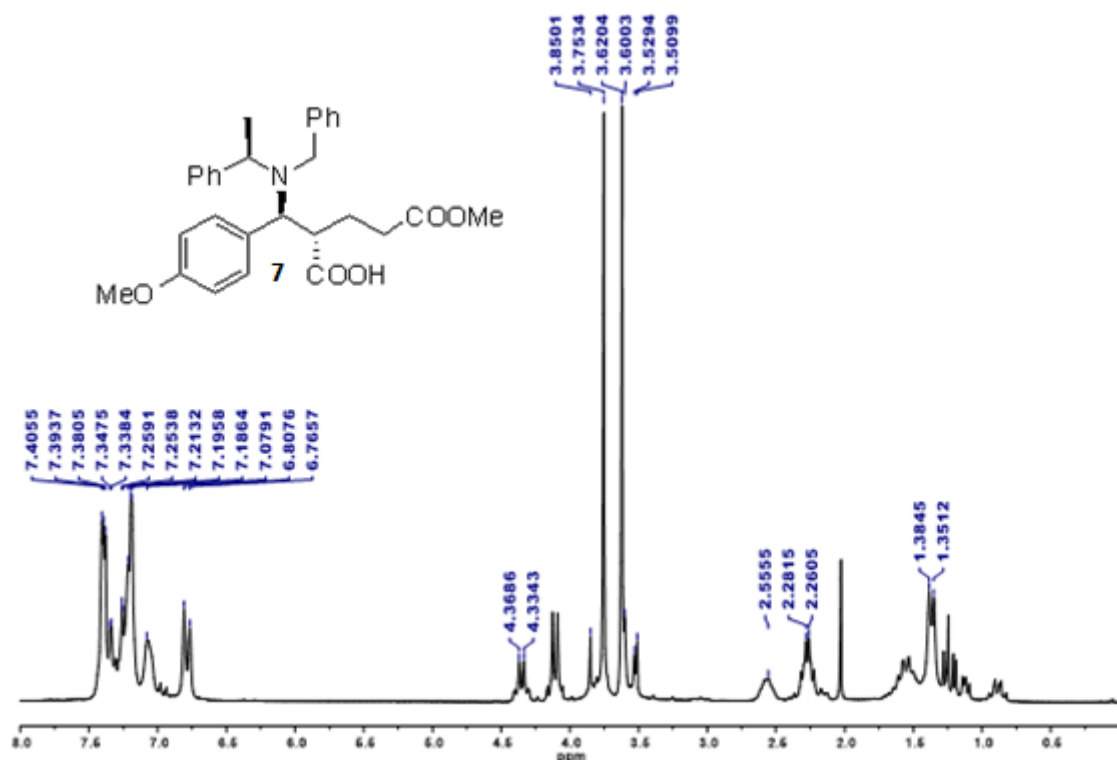
**7**: (S)-2-((S)-((benzyl((R)-1-phenylethyl)amino)(4-methoxyphenyl)methyl)-5-methoxy-5-oxopentanoic acid.

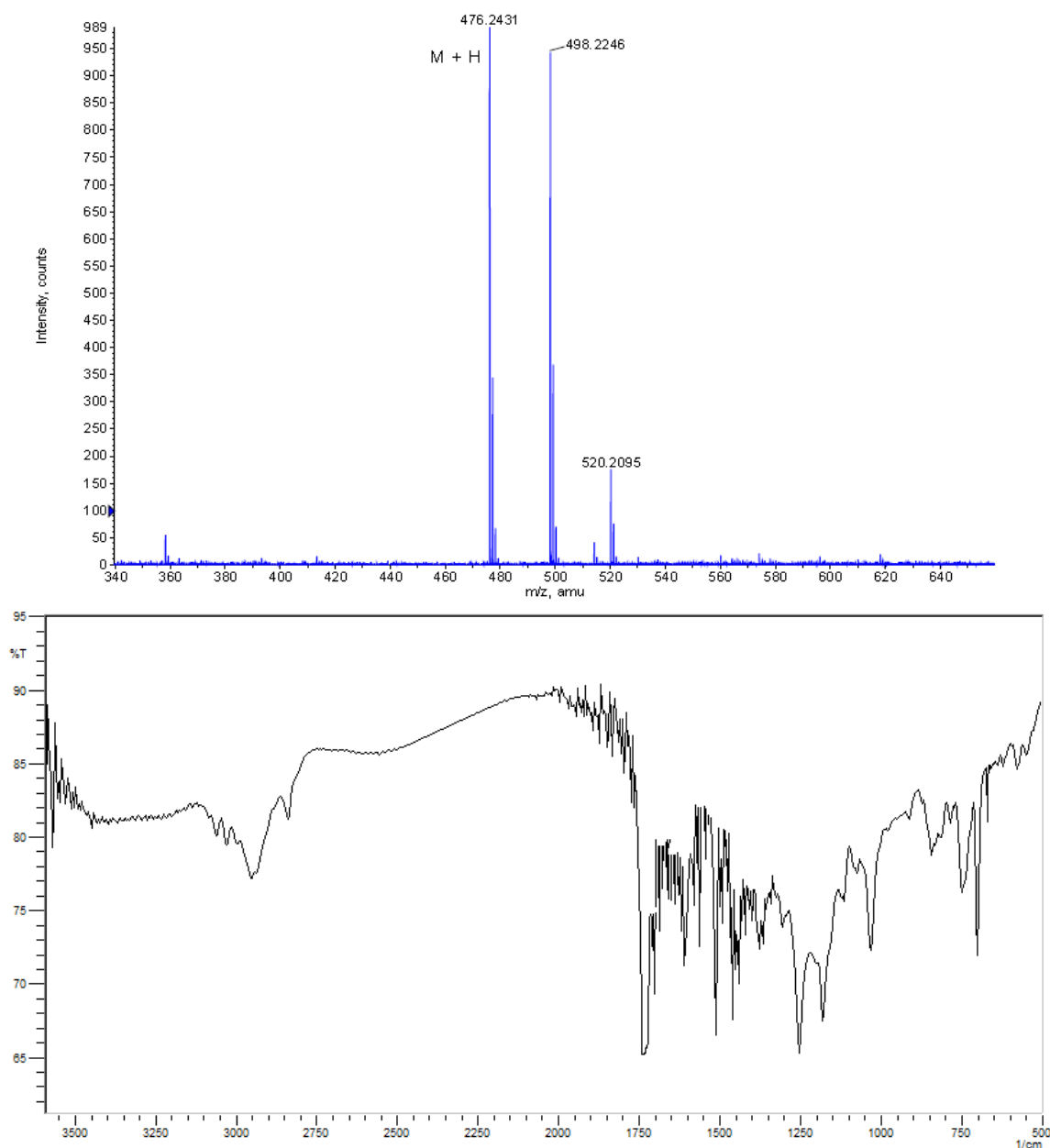
$[\alpha]_{\text{D}}^{20} = +17.7$  (c 1.27,  $\text{CHCl}_3$ ).

**$^1\text{H}$  NMR** (200 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.58 - 7.02 (m, 12H, ArH), 6.78 (d,  $J = 8.3$  Hz, 2H, H-2'), 4.12 (q,  $J = 6.8$  Hz, 1H, C( $\alpha$ )H), 3.80 (AB,  $J_{\text{AB}} = 13.7$  Hz, 1H,  $\text{NCH}_2\text{HPh}$ ), 3.76 (s, 3H, OMe), 3.62 (s, 3H, COOMe), 3.61 (AB,  $J_{\text{AB}} = 13.7$  Hz, 1H,  $\text{NCH}_2\text{HPh}$ ), 2.62 (td,  $J = 11.0, 3.0$  Hz, 1H), 2.30-2.20 (m, 2H, H-2), 1.76-1.48 (m, 2H), 1.37 (d,  $J = 6.8$  Hz, 3H, C( $\alpha$ )H).

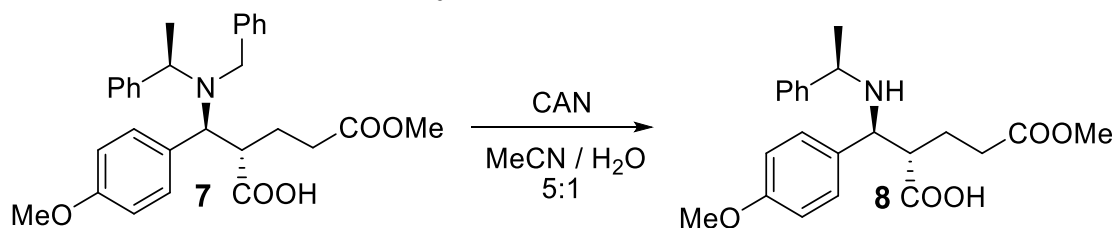
**$^{13}\text{C}$  NMR** (50 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 176.22 (C, COOH), 174.13 (C, COOMe), 159.77 (C, C-4'), 144.61 (C,  $\text{C}_{\text{ipso}} \text{CH}_2\text{Ph}$ ), 137.72 (C,  $\text{C}_{\text{ipso}}$ ), 131.05 (C, C1'), 129.00-127.79 (CHx12, Ar), 114.28 (CH, C-2'), 62.55 (CH, C-5), 57.16 (CH, C( $\alpha$ )), 55.43 ( $\text{CH}_3$ , OCH<sub>3</sub>), 51.69 ( $\text{CH}_3$ , COOMe), 50.80 ( $\text{CH}_2$ ,  $\text{NCH}_2\text{Ph}$ ), 44.92 (CH, C-4), 32.15 ( $\text{CH}_2$ , C-2), 24.56 ( $\text{CH}_2$ , C-3), 14.42 ( $\text{CH}_3$ , C( $\alpha$ )Me).

**E.M.A.R.**, FAB, calcd for  $\text{C}_{29}\text{H}_{34}\text{NO}_5$   $[\text{M}+\text{H}]$ : 476.2437. Found: 476.2431.  $\Delta = 1.03$  ppm.





## 6. Chemoselective debenzoylation of 7.



To a solution of **7** (58.3 mg; 0.124 mmol) in 12 mL of acetonitrile water 5:1, 211 mg (0.498 mmol) of CAN (CAS: 16774-21-3, orange solid) were added. The mixture was kept under stirring at room temperature under Ar atmosphere for 15 minutes. After this time, 5 mL of saturated aqueous NaHCO<sub>3</sub> were added and the mixture was left to stir for another 15 minutes. The reaction mixture is extracted with DCM. The organic phase was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>, filtered and the solvent was evaporated, obtaining 62 mg, which were chromatographed on flash silica. The desired product was eluted with hexane/EtOAc 7:3, 40 mg of **8** (84.0%)

**8: (S)-5-methoxy-2-((S)-(4-methoxyphenyl)((R)-1-phenylethyl)amino)methyl)-5-oxopentanoic acid.**

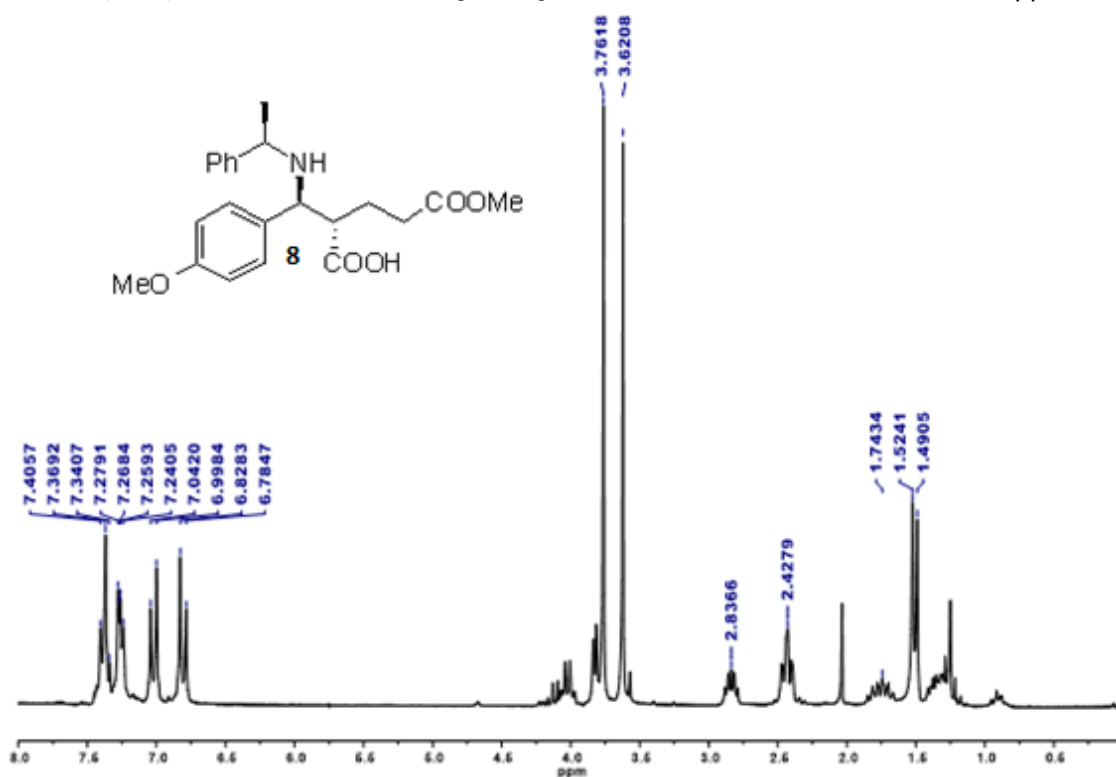
$[\alpha]_D^{20} = +19.5$  (c 1.12,  $\text{CHCl}_3$ ).

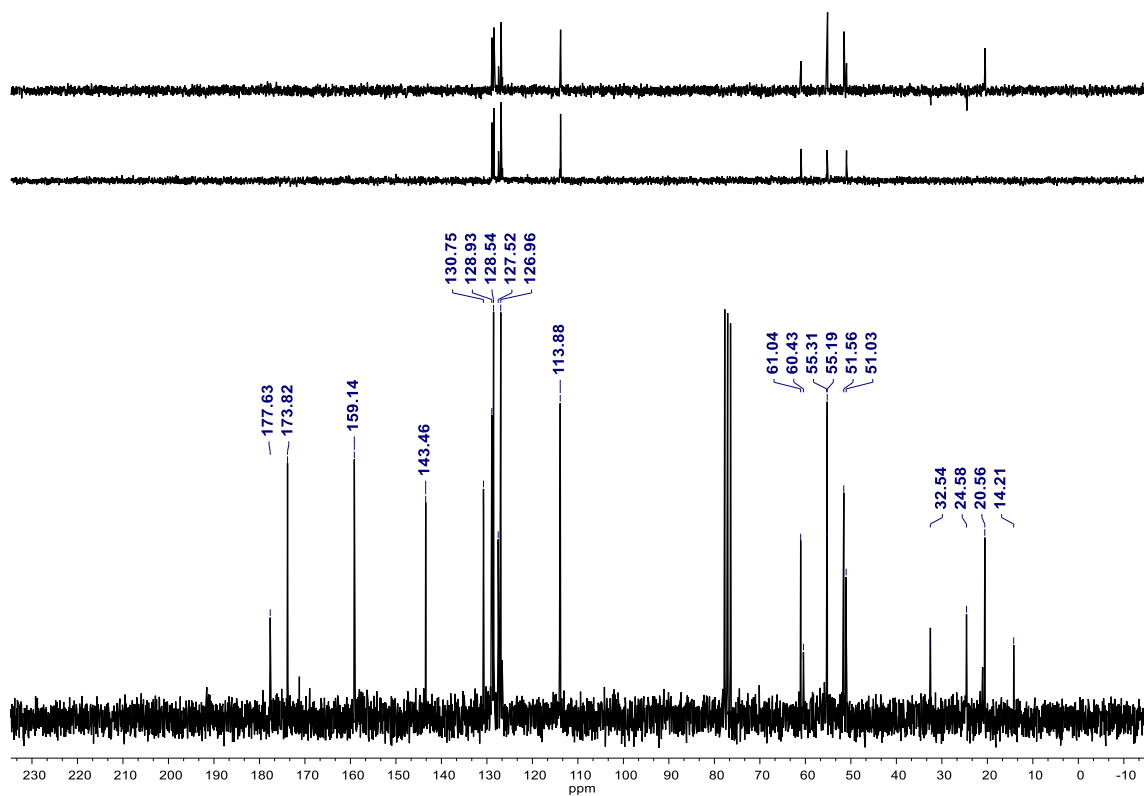
$^1\text{H NMR}$  (200 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 7.41-7.26 (m, 5H, ArH), 7.04 (d,  $J = 4$  Hz, 2H, H2'), 6.80 (d,  $J = 8.4$  Hz, 2H, H-3'), 4.03 (q,  $J = 6.8$  Hz, 1H, C( $\alpha$ )H), 3.83 (AB,  $J_{AB} = 13.7$  Hz, 1H,  $\text{NCH}_B\text{HPh}$ ), 3.76 (s, 3H, OMe), 3.62 (s, 3H COOMe), 3.58 (AB,  $J_{AB} = 13.7$  Hz, 1H,  $\text{NCH}_A\text{HPh}$ ), 2.84 (td,  $J = 11.0$  and 3.0 Hz, 1H, H-4), 2.43 (t,  $J = 7.6$ , 2H, H-2), 1.82-1.65 (m, 1H), 1.51 (d,  $J = 6.8$  Hz, 3H, C( $\alpha$ )H), 1.42-1.25 (m, 2H).

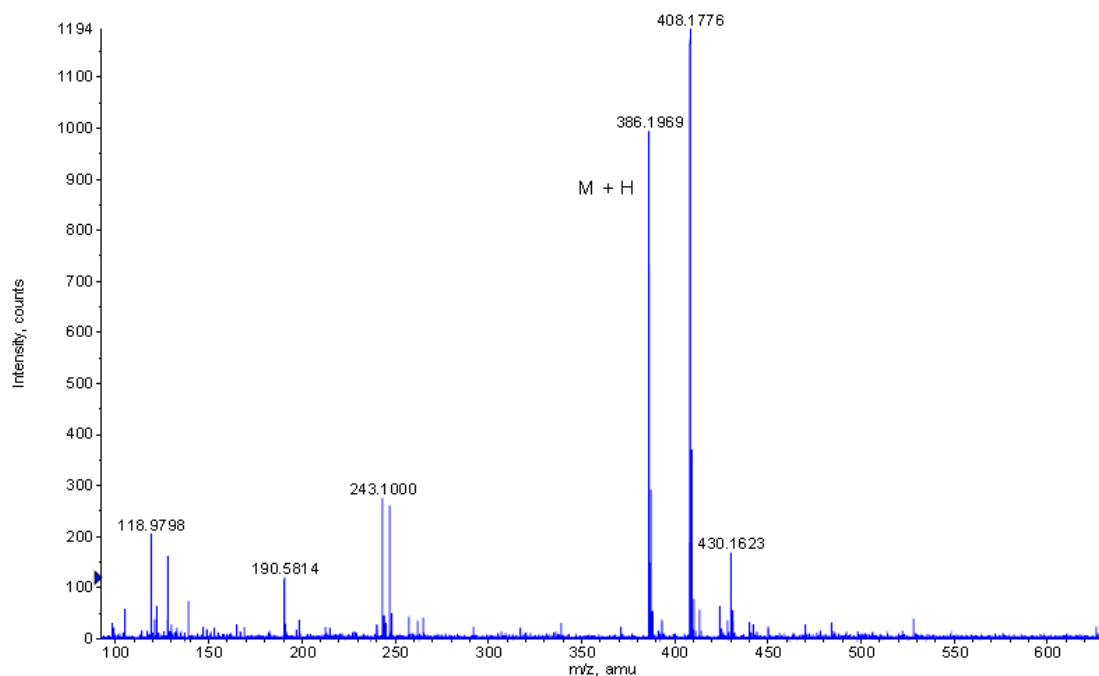
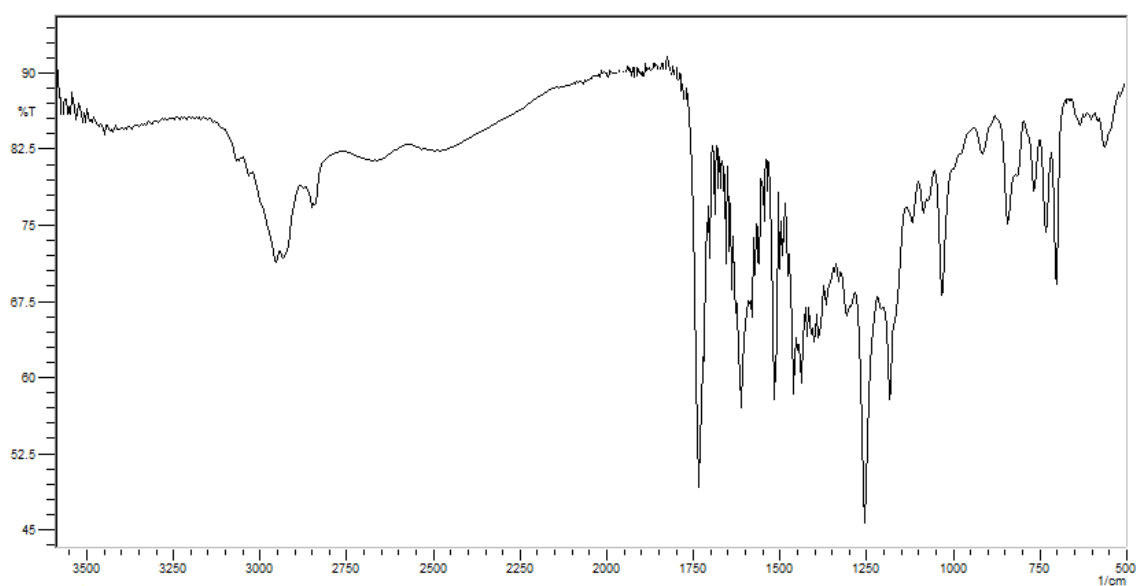
$^{13}\text{C NMR}$  (50 MHz,  $\text{CDCl}_3$ )  $\delta$  (ppm): 177.63 (C, COOH), 173.82 (C, COOMe), 159.14 (C, C-4'), 143.46 (C,  $C_{ipso}$   $\text{CH}_2\text{Ph}$ ), 130.75 (C,  $C_{ipso}$ ), 128.93 (C, C-1'), 128.54-126.96 ( $\text{CH} \times 9$ , Ar), 113.88 (CH, C-3'), 61.04 (CH, C-5), 55.31 (CH, C( $\alpha$ )), 55.19 ( $\text{CH}_3$ , OCH<sub>3</sub>), 51.56 ( $\text{CH}_3$ , COOMe), 51.03 ( $\text{CH}_2$ ,  $\text{NCH}_2\text{Ph}$ ), 32.54 (CH, C-4), 24.58 ( $\text{CH}_2$ , C-2), 20.56 ( $\text{CH}_2$ , C-3), 14.21 ( $\text{CH}_3$ , C( $\alpha$ )Me).

$\text{IR}_{\text{max}}$  (film) ( $\text{cm}^{-1}$ ): 702, 733, 831, 1034, 1180, 1211, 1252, 1306, 1400, 1508, 1611, 1701, 1719, 1730, 2837, 2953.

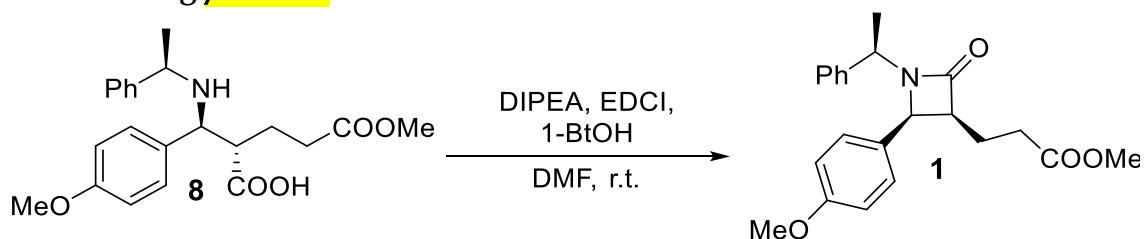
**E.M.AR.**, FAB, calcd for  $\text{C}_{22}\text{H}_{27}\text{NO}_5\text{Na}$  [ $\text{M}+\text{Na}$ ]: 408.1781. Found: 408.1777.  $\Delta = -1.09$  ppm.







## 7. Obtaining $\beta$ -lactam **1**.



40 mg (0.109 mmol) of **8** were dissolved in 20 mL of DMF. Then, 79 mg (0.24 mmol) of EDCI (CAS: 25952-53-8, white powder), 55.8 mg (0.24 mmol) of hydroxybenzotriazole hydrate (CAS: 123333-53-9, white powder) and 64  $\mu$ L (0.24 mmol) of DIPEA (CAS: 7087-68-5, colourless liquid) were added. Stirring was maintained for 16 h, sealing the balloon with a  $\text{CaCl}_2$  tube. The crude was dissolved in EtOAc and washed with small portions of  $\text{H}_2\text{O}$  and saturated aqueous NaCl. The organic phase was dried over anhydrous  $\text{Na}_2\text{SO}_4$ , filtered and the solvent was

evaporated, obtaining 38 mg, which were chromatographed on "flash" silica. The desired product was eluted with hexane/EtOAc 9:1, 33mg of **1** (86.7%) were obtained.

**1:** (3*S*,4*S*)-*N*-[(*R*)- $\alpha$ -methylbenzylamine]-4-*p*-methoxyphenyl-3-methoxycarbonylethyl- $\beta$ -lactam.

$[\alpha]_D^{20} = + 103.5$  (c 1.19, CHCl<sub>3</sub>).

**<sup>1</sup>H NMR** (200 MHz, CDCl<sub>3</sub>)  $\delta$  (ppm): 6.90-6.79 (m, 5H, ArH), 6.75 (d,  $J = 8.7$  Hz, 2H, H-2'), 6.49 (d,  $J = 8.7$  Hz, 2H, H-3'), 4.57 (q,  $J = 7.2$  Hz, 1H, C( $\alpha$ )), 4.12 (d,  $J = 5.6$  Hz, 1H, H-4), 3.36 (s, 3H, COOMe), 3.15 (s, 3H, OMe), 2.83 (td,  $J = 8.3, 5.6$  Hz, 1H, H-3), 1.82-1.72 (m, 2H, H-2''), 1.45-1.31 (m, 2H, H-1''), 1.00 (d,  $J = 7.2$  Hz, 3H, C( $\alpha$ )Me).

**<sup>13</sup>C NMR** (50 MHz, CDCl<sub>3</sub>)  $\delta$  173.24 (C, COOMe), 170.548 (C, C-2), 159.71 (C, C-4'), 140.56 (C, *C<sub>ipso</sub>* CHPh), 129.05 (CH, Ar), 128.81 (CH, Ar), 128.42 (CH, Ar), 127.86 (CH, Ar), 127.40 (CH, Ar), 113.77 (CH, C-2'), 57.56 (CH, C( $\alpha$ )), 55.36 (CH<sub>3</sub>, OCH<sub>3</sub>), 53.48 (CH, C-4), 52.69 (CH, C-3), 51.63 (CH<sub>3</sub>, COOCH<sub>3</sub>), 31.30 (CH<sub>2</sub>, C-2''), 21.08 (CH<sub>2</sub>, C-1''), 19.65 (CH<sub>3</sub>, C( $\alpha$ )Me).

**IR**<sub>max</sub> (film) (cm<sup>-1</sup>): 703, 733, 1032, 1182, 1256, 1292, 1306, 1356, 1389, 1400, 1516, 1611, 1734, 2934, 2953.

**E.M.AR.**, FAB, calcd para C<sub>22</sub>H<sub>25</sub>NO<sub>4</sub>Na [M+Na]: 390.1676. Found: 390.1679.  $\Delta = 0.82$  ppm.

