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**Supporting Information for –**

**Characterization of the Pharmacophore Properties of Novel Selective Estrogen Receptor  
Downregulators (SERDs)**

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## I. Synthesis and Spectroscopic Characterization

**Synthesis Materials and Methods.** Reactions using moisture- or airsensitive reagents were performed in anhydrous solvents, which were collected using a solvent dispensing system built by J. C. Meyer based on a design developed by Pangborn et al. Reaction progress was followed by TLC on 0.25 mm silica gel glass plates containing F-254 indicator. Visualization on TLC was monitored by UV light or phosphomolybdic acid indicator. Reactions were performed under a nitrogen atmosphere unless noted otherwise.  $^1\text{H}$  spectra were obtained on 400 or 500 MHz spectrometers.  $^{13}\text{C}$  NMR spectra were acquired at 100 or 125 MHz. The chemical shifts were reported in parts per million and were referenced to the internal solvent peaks. Coupling constants were reported in hertz. Mass spectra were recorded under electron impact conditions at 70 eV. McMurry coupled compounds **7** and **8** were synthesized according to published procedures.<sup>14</sup>

All compounds assayed were >95% pure in two different HPLC systems (normal and reversed phase); see below. All compounds prepared had spectroscopic data ( $^1\text{H}$  and  $^{13}\text{C}$  NMR) fully consistent with their assigned structures.

**General Procedure for *O*-Alkylation Reaction.** To the mixture of McMurry coupled compound (1.0 equiv) and  $\text{Cs}_2\text{CO}_3$  or  $\text{K}_2\text{CO}_3$  (3 equiv) in  $\text{CH}_3\text{CN}$  (15 mL) was added each bromo-ester (1.0 equiv). The mixture was stirred at room temperature for 24 h. The reaction mixture was poured into 1 N HCl (100 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. Flash column chromatography (20% EtOAc/hexane) gave the alkylated products.

**General Procedure for Heck Coupling Reaction.** To the mixture of triflate compound (**11** or **38**, 1.0 equiv),  $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$  (0.2 equiv) and triethylamine (1.0 mL) in DMF (3.0 mL) was added each alkene-ester (10 equiv) at room temperature under  $\text{N}_2$  atmosphere. The mixture was stirred at 120 °C for 24 h. The reaction mixture was cooled to room temperature and was poured into 1 N HCl (30 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. Flash column chromatography (20% EtOAc/hexane) gave the Heck-coupled products.

**General Procedure for Hydrolysis of Ester.** To the solution of the ester (1.0 equiv) in  $\text{CH}_3\text{OH}$  (4.0 mL) was added 2N KOH (2.0 mL). The mixture was stirred at room temperature for 24 h. The reaction mixture was poured into 1 N HCl (4.0 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. Flash column chromatography (10%  $\text{CH}_3\text{OH}/\text{CH}_2\text{Cl}_2$ ) gave the acid products.

**9-[(4-hydroxyphenyl)(phenyl)methylene]bicyclo[3.3.1]nonane (9).** Titanium(IV) chloride (1.76 mL, 16.06 mmol) was added slowly at -10 °C to a stirred suspension of zinc powder (2.1 g, 32.12 mmol) in 25

mL of anhydrous THF under nitrogen atmosphere. Addition of titanium(IV) chloride released a yellow fume, and the reaction mixture turned a yellow-green color. The cooling was removed, and the reaction mixture was refluxed for 2.5 h. The reaction mixture was then cooled to room temperature. A solution of each 4-hydroxybenzophenone (0.86 g, 4.34 mmol) and bicyclo[3.3.1]nonan-9-one (0.6 g, 4.34 mmol) dissolved in THF (20 mL) was injected by syringe, and the reaction was refluxed for 2 h. The reaction mixture was cooled to room temperature and was poured slowly into a NaHCO<sub>3</sub> solution (300 mL). Et<sub>2</sub>O (150 mL) was added to the aqueous solution with vigorous stirring, and the heterogeneous solution was filtered through Celite. After the organic layer was decanted and saved, the aqueous layer was extracted with additional Et<sub>2</sub>O (150 mL). The ether layer was dried (MgSO<sub>4</sub>) and evaporated under reduced pressure. Flash column chromatography (30% Et<sub>2</sub>OAc/hexane) gave **9** (1.15 g, 87%): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.30-7.26 (m, 2H), 7.21-7.15 (m, 3H), 7.05-7.02 (m, 2H), 2.09-1.98 (m, 2H), 1.88-1.70 (m, 8H), 1.63-1.56 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 153.67, 145.45, 143.29, 135.70, 130.54, 129.26, 127.91, 125.87, 114.96, 114.76, 34.13, 34.03, 33.76, 21.66; MS (EI) *m/z* 304 (M<sup>+</sup>, 100), 261, 222. HRMS (EI) *m/z* calcd for C<sub>22</sub>H<sub>24</sub>O 304.1827, found 304.1820.

**9-[(4-*tert*-Butyldimethylsilyloxyphenyl) (4-hydroxyphenyl) methylene]bicyclo[3.3.1]-nonane (10).** To the mixture of **7** (1.50 g, 4.68 mmol) and imidazole (0.48 g, 7.02 mmol) in THF (40 mL) was added TBDMSCl (1.06 g, 7.02 mmol) at room temperature. The mixture was stirred at room temperature for 24 h. The reaction mixture was poured into water (200 mL) and extracted from the aqueous phase with EtOAc (200 mL). The organic layer was dried with MgSO<sub>4</sub> and evaporated under reduced pressure. The residue was purified by flash column chromatography (20% EtOAc/hexane) to provide **10** (0.91 g, 45%): <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.03-7.00 (m, 4H), 6.77-6.73 (m, 4H), 5.64 (br, 1H), 2.72 (m, 2H), 2.09-1.98 (m, 2H), 1.87-1.78 (m, 8H), 1.61-1.58 (m, 2H), 1.00 (s, 9H), 0.22 (s, 6H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 153.67, 153.579, 144.77, 136.31, 135.86, 130.48, 130.26, 130.23, 119.26, 114.73, 34.06, 34.01, 33.73, 25.62, 21.66, 18.09, -4.44 ; MS (EI) *m/z* 434 (M<sup>+</sup>), 132, 75 (100). HRMS (EI) *m/z* calcd for C<sub>28</sub>H<sub>38</sub>O<sub>2</sub>Si 434.2641, found 434.2643.

**9-[(4-Trifluoromethanesulfonylphenyl) (4-hydroxyphenyl) methylene]bicyclo[3.3.1]-nonane (11).** To the mixture of **10** (2.10 g, 4.83 mmol) and triflic anhydride (1.06 mL, 6.28 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (50 mL) was added triethylamine (0.88 mL, 6.28 mmol) at 0 °C. The mixture was stirred at room temperature for 8 h. The reaction mixture was poured into water (200 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with MgSO<sub>4</sub> and evaporated under reduced pressure. The residue was purified by flash column chromatography (20% EtOAc/hexane) to provide **11** (1.73 g, 79%) as a colorless oil: <sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>) δ 7.21-7.15 (m, 4H), 7.00-6.98 (m, 2H), 6.76-6.74 (m, 2H), 4.80 (br, 1H), 2.70-2.60 (m,

2H), 2.07-2.00 (m, 2H), 1.82-1.78 (m, 8H), 1.61-1.57 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.05, 147.72, 147.30, 143.59, 134.83, 130.98, 130.56, 128.93, 120.77, 118.70 (q,  $J = 318.8$  Hz), 115.02, 34.21, 34.19, 33.79, 33.72, 21.52; MS (EI)  $m/z$  452 ( $\text{M}^+$ , 100), 319, 237. HRMS (EI)  $m/z$  calcd for  $\text{C}_{23}\text{H}_{23}\text{O}_3\text{SF}_3$  452.1269, found 452.1264.

**2,2'-[4,4'-(Bicyclo[3.3.1]non-9-ylidenemethylene)bis(4,1-phenylene)]bis(oxy)diacetic acid (12).** To the mixture of **7** (0.12 g, 0.37 mmol) and  $\text{Cs}_2\text{CO}_3$  (0.29 g, 0.88 mmol) in  $\text{CH}_3\text{CN}$  (15 mL) was added methyl bromoacetate (0.08 mL, 0.08 mmol). The mixture was stirred at room temperature for 24 h. The reaction mixture was poured into 1 N HCl (100 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. 2N KOH (2.0 mL) was added to the reaction residue dissolved in  $\text{CH}_3\text{OH}$  (4.0 mL). The mixture was stirred at room temperature for 24 h, and poured into 1 N HCl (4.0 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. Flash column chromatography ( $\text{CH}_3\text{COOH}/\text{CH}_3\text{OH}/\text{CH}_2\text{Cl}_2 = 1/9/90$ ) gave **12** (0.12 g, 75%):  $^1\text{H}$  NMR (400 MHz,  $\text{DMSO}-d_4$ )  $\delta$  6.95 (d,  $J = 8.4$  Hz, 4H), 6.77 (d,  $J = 8.0$  Hz, 2H), 4.49 (s, 4H), 2.57 (m, 2H), 1.98-1.89 (m, 2H), 1.71 (m, 8H), 1.52-1.50 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, methanol- $d_4$ )  $\delta$  171.00, 156.38, 143.83, 135.19, 129.98, 129.78, 114.03, 65.29, 33.60, 33.26, 21.26; MS (ESI)  $m/z$  437 ( $\text{M}^+ + 1$ , 100), 379. HRMS (ESI)  $m/z$  calcd for  $\text{C}_{26}\text{H}_{29}\text{O}_6$  437.1964, found 437.1952.

**Methyl 2-[4-[bicyclo[3.3.1]non-9-ylidene[4-(tert-butyl)dimethylsilyloxyphenyl]methyl]phenoxy]acetate (14).** According to the general procedure for *O*-alkylation reaction with **10** (0.15 g, 0.34 mmol) and methyl 2-bromoacetate (0.1 mL, 1.02 mmol), the isolated yield of product **14** was 0.14 g (82%) as a colorless oil.  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.10-6.98 (m, 4H), 6.84-6.72 (m, 4H), 4.62 (s, 2H), 3.81 (s, 3H), 2.70-2.69 (m, 2H), 2.06-1.99 (m, 2H), 1.85-1.74 (m, 8H), 1.61-1.57 (m, 2H), 0.99 (s, 9H), 0.21 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.51, 155.94, 153.68, 145.04, 136.93, 136.08, 130.39, 130.22, 130.12, 119.24, 133.95, 65.29, 52.15, 34.02, 33.73, 33.70, 25.60, 21.62, 18.06, -4.45; MS (EI)  $m/z$  506 ( $\text{M}^+$ , 100), 463, 299. HRMS (EI)  $m/z$  calcd for  $\text{C}_{31}\text{H}_{42}\text{O}_4\text{Si}$  506.2852, found 506.2853.

**2-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]acetic acid (15).** Starting from 0.1 g (0.20 mmol) of the ester **14** and using the general procedure for hydrolysis of ester, 0.07 g (93%) of acid product **15** was obtained:  $^1\text{H}$  NMR (400 MHz, methanol- $d_4$ )  $\delta$  6.98 (d,  $J = 6.8$  Hz, 2H), 6.88 (d,  $J = 6.4$  Hz, 2H), 6.80 (d,  $J = 6.8$  Hz, 2H), 6.66 (d,  $J = 6.8$  Hz, 2H), 4.40 (s, 2H), 2.65-2.62 (m, 2H), 2.03-1.97 (m, 2H), 1.77-1.70 (m, 8H), 1.55-1.53 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz, methanol- $d_4$ )  $\delta$  177.06, 157.95, 156.62, 145.22,

137.68, 135.78, 132.31, 131.27, 131.21, 1115.69, 115.37, 68.14, 35.39, 34.77, 34.73, 22.72; MS (EI)  $m/z$  387 ( $M^+$ ), 334, 150 (100). HRMS (EI)  $m/z$  calcd for  $C_{24}H_{26}O_4$  378.1831, found 378.1826.

**(E)-Ethyl 3-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]acrylate (16).** Following the general procedure for Heck coupling reaction and using the triflate **11** and ethyl acrylate as the reactants, 0.14 g (72%) of **16** was obtained as a white solid: mp 69-70 °C;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.66 (d,  $J = 16.4$  Hz, 1H), 7.41 (d,  $J = 8.0$  Hz, 2H), 7.16 (d,  $J = 8.0$  Hz, 2H), 7.00 (d,  $J = 8.4$  Hz, 2H), 6.79-6.75 (m, 2H), 6.39 (d,  $J = 16.4$  Hz, 1H), 4.26 (q,  $J = 7.1$  Hz, 2H), 2.71-2.68 (m, 2H), 2.06-1.99 (m, 2H), 1.33 (t,  $J = 7.0$  Hz, 3H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  167.52, 154.32, 146.58, 145.71, 144.80, 134.74, 131.94, 130.54, 130.08, 129.84, 144.80, 134.74, 131.94, 130.54, 130.08, 129.84, 127.78, 117.10, 114.95, 60.58, 34.26, 34.17, 33.79, 33.74, 21.56, 14.25; MS (EI)  $m/z$  402 ( $M^+$ , 100), 281, 107. HRMS (EI)  $m/z$  calcd for  $C_{27}H_{30}O_3$  402.2195, found 402.2190.

**(E)-3-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]acrylic acid (17).** Starting from 0.09 g (0.22 mmol) of the ester **16** and using the general procedure for hydrolysis of ester, 0.07 g (88%) of acid product **17** was obtained:  $^1H$  NMR (400 MHz, methanol- $d_4$ )  $\delta$  7.63 (d,  $J = 16.0$  Hz, 1H), 7.48 (d,  $J = 8.0$  Hz, 2H), 7.15 (d,  $J = 8.0$  Hz, 2H), 6.93 (d,  $J = 8.4$  Hz, 2H), 6.71-6.68 (m, 2H), 6.42 (d,  $J = 15.6$  Hz, 1H), 2.69-2.63 (m, 2H), 2.09-2.01 (m, 2H), 1.87-1.75 (m, 8H), 1.59-1.56 (m, 2H);  $^{13}C$  NMR (100 MHz, methanol- $d_4$ )  $\delta$  170.56, 157.01, 147.16, 146.88, 146.22, 134.99, 133.52, 132.28, 131.41, 130.94, 128.90, 118.58, 115.85, 35.69, 35.53, 34.81, 34.76, 22.86; MS (EI)  $m/z$  374 ( $M^+$ ), 149, 135 (100). HRMS (EI)  $m/z$  calcd for  $C_{25}H_{26}O_3$  374.1882, found 374.1888.

**(E)-Methyl 4-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]but-3-enoate (18).** Following the general procedure for Heck coupling reaction and using the triflate **11** and methyl 3-butenolate as the reactants, 0.11 g (61%) of **18** was obtained as a colorless oil:  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.27 (d,  $J = 8.4$  Hz, 2H), 7.08 (d,  $J = 8.4$  Hz, 2H), 7.02-6.99 (m, 2H), 6.75-6.72 (m, 2H), 6.46 (d,  $J = 16.0$  Hz, 1H), 6.28-6.21 (m, 1H), 4.98 (s, 1H), 3.71 (s, 3H), 3.24 (d,  $J = 7.2$  Hz, 2H), 2.67 (m, 2H), 2.06-1.98 (m, 2H), 1.86-1.74 (m, 8H), 1.61-1.56 (m, 2H);  $^{13}C$  NMR (100 MHz,  $CDCl_3$ )  $\delta$  173.25, 154.84, 146.72, 143.78, 136.44, 135.39, 134.36, 131.56, 131.28, 130.54, 126.87, 115.80, 52.97, 39.29, 35.21, 35.11, 34.77, 22.63; MS (EI)  $m/z$  402 ( $M^+$ , 100). HRMS (EI)  $m/z$  calcd for  $C_{27}H_{30}O_3$  402.2195, found 402.2200.

**(E)-Ethyl 5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]pent-4-enoate (19).** Following the general procedure for Heck coupling reaction and using the triflate **11** and methyl 4-pentenoate as the reactants, 0.13 g (41%) of **19** was obtained as a colorless oil:  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.24 (d,  $J = 8.5$  Hz, 2H), 7.08 (d,  $J = 8.5$  Hz, 2H), 7.00 (d,  $J = 8.5$  Hz, 2H), 6.74 (d,  $J = 8.5$  Hz, 2H), 6.39

(d,  $J = 16.0$  Hz, 1H), 6.19-6.13 (m, 1H), 4.14 (q,  $J = 7.2$  Hz, 2H), 2.74-2.70 (m, 2H), 2.54-2.45 (m, 4H), 2.06-1.97 (m, 2H), 1.80-1.77 (m, 8H), 1.59-1.56 (m, 2H), 1.26 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  173.22, 153.96, 145.56, 142.36, 135.42, 134.97, 130.76, 130.57, 130.37, 129.53, 127.81, 125.61, 114.80, 60.46, 34.17, 34.12, 34.08, 33.76, 28.27, 21.63, 14.20; MS (EI)  $m/z$  430 ( $\text{M}^+$ , 100), 310, 170. HRMS (EI)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{34}\text{O}_3$  430.2508, found 430.2511.

**(E)-Methyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hex-5-enoate (20).**

Following the general procedure for Heck coupling reaction and using the triflate **11** and methyl 5-hexenoate as the reactants, 0.17 g (38%) of **20** was obtained as a colorless oil:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.25 (d,  $J = 8.0$  Hz, 2H), 7.08 (d,  $J = 12.0$  Hz, 2H), 7.01 (d,  $J = 8.8$  Hz, 2H), 6.75 (d,  $J = 8.8$  Hz, 2H), 6.37 (d,  $J = 16.0$  Hz, 1H), 6.17-6.10 (m, 1H), 3.68 (s, 3H), 2.72 (m, 2H), 2.37 (t,  $J = 7.6$  Hz, 2H), 2.24 (q,  $J = 6.8$  Hz, 2H), 2.08-1.97 (m, 2H), 1.85-1.78 (m, 8H), 1.61-1.56 (m, 2H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  175.46, 154.98, 146.42, 143.21, 136.33, 136.10, 131.64, 131.51, 130.48, 146.42, 143.21, 136.33, 136.10, 131.64, 131.51, 130.48, 130.20, 129.75, 126.49, 115.78, 52.62, 35.17, 35.08, 34.75, 34.34, 33.26, 25.49, 22.62; MS (EI)  $m/z$  430 ( $\text{M}^+$ , 100), 332, 107. HRMS (EI)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{34}\text{O}_3$  430.2508, found 430.2501.

**(E)-Ethyl 7-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hept-6-enoate (21).**

Following the general procedure for Heck coupling reaction and using the triflate **11** and methyl 6-heptenoate as the reactants, 0.09 g (35%) of **21** was obtained as a colorless oil:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.25 (d,  $J = 8.5$  Hz, 2H), 7.08 (d,  $J = 7.5$  Hz, 2H), 7.02 (d,  $J = 8.5$  Hz, 2H), 6.75 (d,  $J = 9.0$  Hz, 2H), 6.36 (d,  $J = 16.0$  Hz, 1H), 6.20-6.13 (m, 1H), 4.15 (q,  $J = 7.2$  Hz, 2H), 2.72 (br, 2H), 2.34 (t,  $J = 7.5$  Hz, 2H), 2.25-2.20 (m, 2H), 2.03-1.98 (m, 2H), 1.87-1.46 (m, 14H), 1.26 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  175.16, 154.96, 146.37, 143.07, 136.39, 136.30, 131.46, 130.98, 130.56, 130.47, 128.89, 126.45, 115.77, 61.41, 35.21, 35.16, 35.10, 35.07, 34.76, 33.57, 29.82, 25.43, 22.62, 15.17; MS (ESI)  $m/z$  459 ( $\text{M}^+ + 1$ ), 364 (100). HRMS (ESI)  $m/z$  calcd for  $\text{C}_{31}\text{H}_{39}\text{O}_3$  459.2899, found 459.2899.

**(E)-4-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]but-3-enoic acid (22).** Starting from 0.11 g (0.27 mmol) of the ester **18** and using the general procedure for hydrolysis of ester, 0.09 g (86%) of acid product **22** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.28 (d,  $J = 8.0$  Hz, 2H), 7.05 (d,  $J = 8.0$  Hz, 2H), 6.94-6.92 (m, 2H), 6.70-6.67 (m, 2H), 6.46 (d,  $J = 16.0$  Hz, 2H), 6.32-6.26 (m, 1H), 3.20 (d,  $J = 7.0$  Hz, 2H), 2.69-2.66 (m, 2H), 2.10-2.03 (m, 2H), 1.85-1.77 (m, 8H), 1.60-1.57 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  176.03, 156.83, 145.90, 144.00, 136.29, 135.49, 133.98, 132.64, 131.36, 130.49, 126.82, 123.04, 115.75, 39.32, 35.58, 35.48, 34.79, 22.72; MS (EI)  $m/z$  388 ( $\text{M}^+$ , 100), 149, 57. HRMS (EI)  $m/z$  calcd for  $\text{C}_{26}\text{H}_{28}\text{O}_3$  388.2039, found 388.2042.

**(E)-5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]pent-4-enoic acid (23).** Starting from 0.10 g (0.23 mmol) of the ester **19** and using the general procedure for hydrolysis of ester, 0.08 g (87%) of acid product **23** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.21 (d,  $J = 8.0$  Hz, 2H), 7.01 (d,  $J = 8.0$  Hz, 2H), 6.92 (d,  $J = 8.5$  Hz, 2H), 6.68 (d,  $J = 8.0$  Hz, 2H), 6.38 (d,  $J = 15.5$  Hz, 2H), 6.19-6.15 (m, 1H), 2.68-2.61 (m, 2H), 2.44 (m, 4H), 2.06-1.99 (m, 2H), 1.78-1.73 (m, 8H), 1.56-1.53 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.72, 156.73, 145.75, 143.58, 136.57, 135.49, 132.60, 131.88, 131.35, 130.41, 129.00, 126.63, 115.72, 35.51, 35.41, 34.77, 34.75, 29.50, 22.71; MS (EI)  $m/z$  402 ( $\text{M}^+$ ), 332 (100), 107. HRMS (EI)  $m/z$  calcd for  $\text{C}_{27}\text{H}_{30}\text{O}_3$  402.2195, found 402.2197.

**(E)-6-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hex-5-enoic acid (24).** Starting from 0.13 g (0.30 mmol) of the ester **20** and using the general procedure for hydrolysis of ester, 0.10 g (79%) of acid product **24** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.21 (d,  $J = 8.0$  Hz, 2H), 7.01 (d,  $J = 8.0$  Hz, 2H), 6.92 (d,  $J = 8.5$  Hz, 2H), 6.68 (d,  $J = 8.0$  Hz, 2H), 6.34 (d,  $J = 16.0$  Hz, 2H), 6.18-6.12 (m, 1H), 2.68-2.65 (m, 2H), 2.31 (t,  $J = 7.0$  Hz, 2H), 2.21 (q,  $J = 6.8$  Hz, 2H), 2.04-1.99 (m, 2H), 1.79-1.73 (m, 8H), 1.64-1.55 (m, 2H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.73, 156.74, 145.72, 143.46, 136.76, 135.53, 132.64, 131.80, 131.35, 130.41, 130.08, 126.58, 115.71, 35.52, 35.43, 34.78, 34.46, 33.46, 25.83, 22.72; MS (ESI)  $m/z$  417 ( $\text{M}^+ + 1$ , 100), 373. HRMS (ESI)  $m/z$  calcd for  $\text{C}_{28}\text{H}_{33}\text{O}_3$  417.2430, found 417.2420.

**(E)-7-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hept-6-enoic acid (25).** Starting from 0.10 g (0.22 mmol) of the ester **21** and using the general procedure for hydrolysis of ester, 0.08 g (88%) of acid product **25** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.23 (d,  $J = 8.5$  Hz, 2H), 7.02 (d,  $J = 8.0$  Hz, 2H), 6.92 (d,  $J = 9.0$  Hz, 2H), 6.68 (d,  $J = 8.5$  Hz, 2H), 6.36 (d,  $J = 15.5$  Hz, 1H), 6.22-6.17 (m, 1H), 2.69-2.66 (m, 2H), 2.29 (t,  $J = 7.5$  Hz, 2H), 2.25-2.19 (m, 2H), 2.10-2.02 (m, 2H), 2.00-1.75 (m, 8H), 1.68-1.47 (m, 6H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.89, 156.75, 145.68, 143.38, 136.92, 135.55, 132.68, 131.36, 130.68, 130.40, 130.12, 126.53, 115.72, 35.53, 35.44, 35.38, 34.95, 34.77, 33.75, 30.00, 25.69, 22.72; MS (ESI)  $m/z$  431 ( $\text{M}^+ + 1$ , 100), 358. HRMS (ESI)  $m/z$  calcd for  $\text{C}_{29}\text{H}_{35}\text{O}_3$  431.2586, found 431.2585.

**Methyl hydrogen 4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]styrylphosphonate (26).** To the mixture of **11** (0.22 g, 0.49 mmol),  $\text{Pd}(\text{PPh}_3)_2\text{Cl}_2$  (0.07 mmol, 0.01 mmol) and triethylamine (1.0 mL) in DMF (3.0 mL) was added dimethyl vinylphosphonate (0.67 g, 4.9 mmol) at room temperature under  $\text{N}_2$  atmosphere. The mixture was stirred at 120  $^\circ\text{C}$  for 24 h. The reaction mixture was cooled to room temperature and was poured into 1 N HCl (30 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. 2N KOH (2.0 mL) was added

to the reaction residue dissolved in CH<sub>3</sub>OH (4.0 mL). The mixture was stirred at room temperature for 24 h, and poured into 1 N HCl (4.0 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with MgSO<sub>4</sub> and evaporated under reduced pressure. Flash column chromatography (10% CH<sub>3</sub>OH /CH<sub>2</sub>Cl<sub>2</sub>) gave **26** (0.12 g, 57 %) as a white solid: mp 145-146 °C; <sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>6</sub>) δ 7.49 (d, *J* = 7.5 Hz, 2H), 7.25-7.17 (m, 1H), 7.06 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.5 Hz, 2H), 6.67 (d, *J* = 8.5 Hz, 2H), 6.42-6.35 (m, 1H), 3.49 (d, *J* = 11.0 Hz, 3H), 2.61-2.54 (m, 2H), 2.00-1.94 (m, 2H), 1.72-1.71 (m, 8H), 1.53-1.51 (m, 2H); <sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>) δ 155.90, 144.65, 144.42, 133.22, 133.00, 132.69, 130.33, 130.00, 129.32, 127.34, 117.38 (d, *J* = 232.4 Hz), 114.97, 51.30 (d, *J* = 5.8 Hz), 33.80, 33.61, 33.29, 33.25, 21.20; MS (ESI) *m/z* 425 (M<sup>+</sup> + 1, 100). HRMS (ESI) *m/z* calcd for C<sub>25</sub>H<sub>30</sub>O<sub>4</sub>P 425.1882, found 425.1868.

**Ethyl 4-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]butanoate (27)**. According to the general procedure for *O*-alkylation reaction with **7** and ethyl 4-bromobutanoate, the isolated yield of product **27** was 0.07 g (40%) as a white solid: 78-79 °C; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.06-6.98 (m, 4H), 6.80-6.72 (m, 4H), 4.91 (s, 1H), 4.14 (q, *J* = 7.2 Hz, 2H), 3.97 (t, *J* = 6.0 Hz, 2H), 2.69 (m, 2H), 1.84-2.12-1.98 (m, 4H), 1.85-1.73 (m, 8H), 1.60-1.56 (m, 2H), 1.25 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 174.46, 157.99, 154.71, 145.95, 136.93, 136.86, 131.49, 131.30, 131.08, 115.73, 114.78, 67.51, 61.48, 35.10, 34.76, 31.85, 25.66, 22.67, 15.18; MS (EI) *m/z* 434 (M<sup>+</sup>), 227, 115 (100). HRMS (EI) *m/z* calcd for C<sub>28</sub>H<sub>34</sub>O<sub>4</sub> 434.2457, found 434.2452.

**Methyl 5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]pentanoate (28)**. According to the general procedure for *O*-alkylation reaction with **7** and methyl 5-bromopentanoate, the isolated yield of product **28** was 0.07 g (41%) as a colorless oil: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.06-6.99 (m, 4H), 6.80-6.72 (m, 4H), 3.94 (t, *J* = 5.6 Hz, 2H), 2.07-1.97 (m, 2H), 1.84-1.73 (m, 25H), 1.60-1.56 (m, 2H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 175.27, 158.03, 154.81, 145.82, 136.78, 131.46, 131.28, 131.11, 115.71, 114.73, 88.79, 68.12, 52.64, 35.07, 34.74, 34.67, 29.63, 22.66, 22.61; MS (EI) *m/z* 434 (M<sup>+</sup>, 100). HRMS (EI) *m/z* calcd for C<sub>28</sub>H<sub>34</sub>O<sub>4</sub> 434.2457, found 434.2451.

**Ethyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoate (29)**. According to the general procedure for *O*-alkylation reaction with **7** and ethyl 6-bromohexanoate, the yield of compound **29** was 0.21 g (45%) as a colorless oil: <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.06-6.99 (m, 4H), 6.81-6.74 (m, 4H), 5.90 (br, 1H), 4.15 (q, *J* = 7.1 Hz, 2H), 3.93 (t, *J* = 6.4 Hz, 2H), 2.71 (br, 2H), 2.35 (t, *J* = 7.4 Hz, 2H), 2.08-1.97 (m, 2H), 1.86-1.67 (m, 12H), 1.60-1.46 (m, 4H), 1.26 (t, *J* = 7.0 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 175.18, 158.09, 154.96, 145.70, 136.74, 136.62, 131.41, 131.26, 131.18, 115.71, 114.73, 68.42, 61.45,



35.24, 35.04, 34.72, 29.90, 26.60, 25.64, 22.63, 15.14; MS (EI)  $m/z$  462 ( $M^+$ , 100). HRMS (EI)  $m/z$  calcd for  $C_{30}H_{38}O_4$  462.2770, found 462.2766.

**Ethyl 7-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]heptanoate (30).** According to the general procedure for *O*-alkylation reaction with **7** and ethyl 7-bromoheptanoate, the isolated yield of product **30** was 0.18 g (38%) as a colorless oil:  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.06 (d,  $J = 8.5$  Hz, 2H), 7.02 (d,  $J = 9.0$  Hz, 2H), 6.81 (d,  $J = 8.5$  Hz, 2H), 6.76 (d,  $J = 8.5$  Hz, 2H), 5.94 (s, 1H), 4.16 (q,  $J = 7.0$  Hz, 2H), 3.93 (t,  $J = 6.5$  Hz, 2H), 2.73 (br, 2H), 2.34 (t,  $J = 7.5$  Hz, 2H), 2.08-1.99 (m, 2H), 1.87-1.75 (m, 10H), 1.71-1.58 (m, 4H), 1.51-1.37 (m, 4H), 1.27 (t,  $J = 7.2$  Hz, 3H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  175.32, 158.14, 154.95, 145.67, 136.70, 136.63, 131.40, 131.24, 131.20, 115.71, 114.74, 68.60, 61.42, 35.25, 35.04, 34.71, 30.04, 29.76, 26.67, 25.79, 22.62, 15.14; MS (EI)  $m/z$  476 ( $M^+$ ), 446 (100), 255. HRMS (EI)  $m/z$  calcd for  $C_{31}H_{40}O_4$  476.2927, found 476.2918.

**Ethyl 11-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]nudecanoate (31).** According to the general procedure for *O*-alkylation reaction with **7** and ethyl 11-bromoundecanoate, the isolated yield of product **31** was 0.24 g (36%) as a colorless oil:  $^1H$  NMR (500 MHz,  $CDCl_3$ )  $\delta$  7.08-7.00 (m, 4H), 6.84-6.76 (m, 4H), 4.16 (q,  $J = 7.2$  Hz, 2H), 3.94 (t,  $J = 6.5$  Hz, 2H), 2.73 (br, 2H), 2.33 (t,  $J = 7.7$  Hz, 2H), 2.08-1.99 (m, 2H), 1.87-1.58 9 (m 14H), 1.48-1.42 (m, 2H), 1.38-1.26 (m, 13H);  $^{13}C$  NMR (125 MHz,  $CDCl_3$ )  $\delta$  175.51, 158.71, 155.04, 145.56, 136.67, 136.53, 131.36, 131.27, 131.22, 115.72, 114.75, 68.79, 61.39, 35.35, 35.04, 35.01, 34.70, 30.38, 30.26, 30.22, 30.11, 30.00, 26.97, 25.87, 22.62, 15.12; MS (EI)  $m/z$  532 ( $M^+$ ), 446 (100), 223. HRMS (EI)  $m/z$  calcd for  $C_{35}H_{48}O_4$  532.3553, found 532.3558.

**4-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]butanoic acid (32).** Starting from 0.15 g (0.34 mmol) of the ester **27** and using the general procedure for hydrolysis of ester, 0.14 g (97%) of acid product **32** was obtained:  $^1H$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.26 (s 1H), 7.05-6.99 (m, 4H), 6.80-6.71 (m, 4H), 3.98 (t,  $J = 6.0$  Hz, 2H), 2.69 (br, 2H), 2.58 (t,  $J = 7.5$  Hz, 2H), 2.12-1.98 (m, 4H), 1.84-1.77 (m, 8H), 1.60-1.54 (m, 2H);  $^{13}C$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.16, 158.67, 156.69, 145.12, 137.26, 135.92, 132.49, 131.29, 131.27, 115.66, 114.96, 67.98, 35.48, 35.45, 34.77, 31.52, 25.94, 22.75; MS (EI)  $m/z$  406 ( $M^+$ ), 285, 141 (100). HRMS (EI)  $m/z$  calcd for  $C_{26}H_{30}O_4$  406.2144, found 406.2143.

**5-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]pentanoic acid (33).** Starting from 0.12 g (0.28 mmol) of the ester **28** and using the general procedure for hydrolysis of ester, 0.11 g (92%) of acid product **33** was obtained:  $^1H$  NMR (400 MHz, methanol- $d_4$ )  $\delta$  7.00-6.90 (m, 4H), 6.78-6.66 (m, 4H), 3.91 (t,  $J = 5.2$  Hz, 2H), 2.66 (br, 2H), 2.35 (br, 2H), 2.08-1.98 (m, 2H), 1.84-1.72 (m, 12H), 1.58-1.54 (m, 2H);  $^{13}C$  NMR (100 MHz, methanol- $d_4$ )  $\delta$  177.43, 158.70, 156.62, 145.07, 137.07, 135.91, 132.42, 131.31,

131.25, 115.65, 114.89, 68.50, 35.41, 30.77, 29.85, 22.84, 22.76; MS (EI)  $m/z$  420 ( $M^+$ , 100), 285, 107. HRMS (EI)  $m/z$  calcd for  $C_{27}H_{32}O_4$  420.2301, found 420.2301.

**6-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoic acid (34).** Starting from 0.09 g (0.19 mmol) of the ester **29** and using the general procedure for hydrolysis of ester, 0.08 g (98%) of acid product **34** was obtained:  $^1H$  NMR (400 MHz, methanol- $d_4$ )  $\delta$  6.98 (d,  $J = 6.8$  Hz, 2H), 6.91 (d,  $J = 6.8$  Hz, 2H), 6.76 (d,  $J = 6.8$  Hz, 2H), 6.67 (d,  $J = 6.8$  Hz, 2H), 3.89 (t,  $J = 5.0$  Hz, 2H), 2.66 (br, 2H), 2.29 (t,  $J = 5.8$  Hz, 2H), 2.07-1.98 (m, 2H), 1.80-1.44 (m, 16H);  $^{13}C$  NMR (100 MHz, methanol- $d_4$ )  $\delta$  177.73, 158.80, 156.67, 145.07, 137.05, 135.93, 132.48, 131.34, 131.27, 115.66, 114.90, 68.70, 35.41, 34.90, 34.76, 30.13, 26.76, 25.85, 22.76; MS (ESI)  $m/z$  435 ( $M^+ + 1$ , 100). HRMS (ESI)  $m/z$  calcd for  $C_{28}H_{35}O_4$  435.2535, found 435.2556.

**7-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]heptanoic acid (35).** Starting from 0.11 g (0.23 mmol) of the ester **30** and using the general procedure for hydrolysis of ester, 0.10 g (96%) of acid product **35** was obtained:  $^1H$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  6.98 (d,  $J = 8.5$  Hz, 2H), 6.91 (d,  $J = 8.5$  Hz, 2H), 6.77 (d,  $J = 8.5$  Hz, 2H), 6.67 (d,  $J = 8.5$  Hz, 2H), 4.89 (br, 1H), 3.90 (t,  $J = 6.5$  Hz, 2H), 2.67-2.66 (m, 2H), 2.80 (t,  $J = 7.2$  Hz, 2H), 2.08 -1.99 (m, 2H), 1.84-1.70 (m, 10H), 1.64-1.53 (m, 4H), 1.49-1.35 (m, 4H);  $^{13}C$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.65, 158.82, 156.64, 145.06, 137.04, 135.94, 132.49, 131.31, 131.24, 115.65, 114.92, 68.84, 35.45, 35.43, 34.87, 34.77, 30.28, 29.96, 26.88, 26.01, 22.75; MS (EI)  $m/z$  448 ( $M^+$ , 100), 284, 141. HRMS (EI)  $m/z$  calcd for  $C_{29}H_{36}O_4$  448.2614, found 448.2619.

**11-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]nudecanoic acid (36).** Starting from 0.15 g (0.28 mmol) of the ester **31** and using the general procedure for hydrolysis of ester, 0.14 g (99%) of acid product **36** was obtained:  $^1H$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  6.99-6.90 (m, 4H), 6.76-6.66 (m, 4H), 3.87 (t,  $J = 6.5$  Hz, 2H), 2.66 (br, 2H), 2.25 (t,  $J = 7.5$  Hz, 2H), 2.05 -1.98 (m, 2H), 1.78-1.68 (m, 10H), 1.57-1.56 (m, 4H), 1.42-1.29 (m, 12H);  $^{13}C$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.87, 158.80, 156.62, 145.02, 136.98, 135.92, 132.48, 131.31, 131.24, 115.66, 114.91, 68.92, 35.42, 35.06, 34.77, 30.66, 30.54, 30.49, 30.46, 30.41, 30.24, 27.17, 26.12, 22.76; MS (EI)  $m/z$  504 ( $M^+$ ), 446 (100), 141. HRMS (EI)  $m/z$  calcd for  $C_{33}H_{44}O_4$  504.3240, found 504.3234.

**(4-tert-Butyldimethylsilyloxyphenyl) (4-hydroxyphenyl) methylenecyclohexane (37).** To the mixture of **8** (2.17 g, 7.74 mmol) and imidazole (0.74 g, 10.84 mmol) in THF (50 mL) was added TBDMSCl (1.63 g, 10.84 mmol) at room temperature. The mixture was stirred at room temperature for 24 h. The reaction mixture was poured into water (200 mL) and extracted from the aqueous phase with EtOAc (200 mL). The organic layer was dried with  $MgSO_4$  and evaporated under reduced pressure. The residue was purified by

flash column chromatography (20% EtOAc/hexane) to provide **37** (1.22 g, 40%):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  6.99-6.97 (m, 4H), 6.77-6.74 (m, 4H), 6.40 (br, 1H), 2.27-2.26 (m, 4H), 1.61 (br, 6H), 1.00 (s, 9H), 0.22 (s, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  153.90, 153.65, 138.11, 136.44, 135.72, 133.56, 131.01, 130.83, 119.22, 114.71, 32.43, 28.62, 25.61, 18.08, -4.47; MS (EI)  $m/z$  394 ( $\text{M}^+$ , 100), 337, 187. HRMS (EI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{34}\text{O}_2\text{Si}$  394.2328, found 394.2329.

**(4-Hydroxyphenyl) (4-trifluoromethanesulfonylphenyl) methylenecyclohexane (38)**. To the mixture of **37** (0.33 g, 0.84 mmol) and triflic anhydride (0.18 mL, 1.09 mmol) in  $\text{CH}_2\text{Cl}_2$  (50 mL) was added triethylamine (0.15 mL, 1.09 mmol) at 0 °C. The mixture was stirred at room temperature for 8 h. The reaction mixture was poured into water (200 mL) and extracted from the aqueous phase with EtOAc. The organic layer was dried with  $\text{MgSO}_4$  and evaporated under reduced pressure. The residue was purified by flash column chromatography (20% EtOAc/hexane) to provide **38** (0.28 g, 82%) as a colorless oil:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.17-6.95 (m, 6H), 6.77-6.76 (m, 2H), 4.98 (s, 1H), 2.26-2.19 (m, 4H), 1.61-1.59 (m, 6H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  154.02, 147.90, 143.67, 140.75, 134.85, 132.04, 131.88, 131.06, 120.90, 118.80 (q,  $J = 318.9$  Hz), 114.82, 32.43, 28.46, 26.54; MS (EI)  $m/z$  412 ( $\text{M}^+$ , 100), 279, 211. HRMS (EI)  $m/z$  calcd for  $\text{C}_{20}\text{H}_{19}\text{O}_4\text{SF}_3$  412.0956, found 412.0960.

**(E)-ethyl 3-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenyl]acrylate (39)**. Following the general procedure for Heck coupling reaction and using the triflate **38** and methyl acrylate as the reactants, 0.12 g (65%) of **39** was obtained as a colorless oil:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.66 (d,  $J = 16.0$  Hz, 1H), 7.40 (d,  $J = 8.4$  Hz, 2H), 7.12 (d,  $J = 8.0$  Hz, 2H), 6.97-6.94 (m, 2H), 6.79-6.76 (m, 2H), 6.39 (d,  $J = 16.0$  Hz, 1H), 4.26 (q,  $J = 7.1$  Hz, 2H), 2.27-2.24 (m, 4H), 1.60 (m, 6H), 1.33 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  167.56, 154.39, 145.79, 144.82, 139.82, 134.76, 133.34, 131.96, 131.06, 130.36, 127.67, 117.16, 114.87, 60.61, 32.47, 28.61, 26.70, 14.24; MS (EI)  $m/z$  362 ( $\text{M}^+$ , 100), 333, 101. HRMS (EI)  $m/z$  calcd for  $\text{C}_{24}\text{H}_{26}\text{O}_3$  362.1882, found 362.1879.

**Ethyl 6-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoate (40)**. According to the general procedure for *O*-alkylation reaction with **8** and ethyl 6-bromohexanoate, the isolated yield of product **40** was 0.18 g (34%) as a colorless oil:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.02-6.95 (m, 4H), 6.80-6.73 (m, 4H), 5.85 (s, 1H), 4.15 (q,  $J = 7.2$  Hz, 2H), 3.93 (t,  $J = 6.2$  Hz, 2H), 2.35 (t,  $J = 7.5$  Hz, 2H), 2.25-2.24 (m, 4H), 1.99-1.67 (m, 4H), 1.59-1.57 (m, 6H), 1.53-1.46 (m, 2H), 1.26 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  174.15, 157.14, 153.97, 138.11, 135.77, 135.70, 133.43, 130.98, 130.81, 114.63, 113.67, 67.46, 60.44, 34.25, 32.42, 28.90, 28.62, 26.81, 25.60, 24.64, 14.15; MS (EI)  $m/z$  422 ( $\text{M}^+$ , 100), 279, 143. HRMS (EI)  $m/z$  calcd for  $\text{C}_{27}\text{H}_{34}\text{O}_4$  422.2457, found 422.2466.

**Methyl 2-[4-[cyclohexylidene(4-*tert*-butyldimethylsilyloxyphenyl)methyl]phenoxy]acetate (41).**

According to the general procedure for *O*-alkylation reaction with **37** and methyl 2-bromoacetate, the isolated yield of product **41** was 0.26 g (77%) as a colorless oil:  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.03 (d,  $J = 8.8$  Hz, 2H), 6.95 (d,  $J = 8.4$  Hz, 2H), 6.81 (d,  $J = 8.8$  Hz, 2H), 6.74 (d,  $J = 8.4$  Hz, 2H), 4.62 (s, 2H), 3.81 (s, 3H), 2.22 (m, 4H), 1.59 (m, 6H), 0.98 (s, 9H), 0.20 (s, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  169.50, 155.95, 153.75, 138.44, 136.98, 136.14, 133.33, 130.95, 130.77, 119.20, 113.86, 65.29, 52.18, 32.41, 28.62, 26.80, 25.61, 18.09, -4.45; MS (EI)  $m/z$  466 ( $\text{M}^+$ , 100), 446, 275. HRMS (EI)  $m/z$  calcd for  $\text{C}_{28}\text{H}_{38}\text{O}_4\text{Si}$  466.2539, found 466.2544.

**(E)-3-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenyl]acrylic acid (42).** Starting from 0.11 g (0.30 mmol) of the ester **39** and using the general procedure for hydrolysis of ester, 0.09 g (85%) of acid product **42** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.63 (d,  $J = 15.6$  Hz, 1H), 7.48 (d,  $J = 8.4$  Hz, 2H), 7.10 (d,  $J = 8.0$  Hz, 2H), 6.90-6.86 (m, 2H), 6.71-6.67 (m, 2H), 6.43 (d,  $J = 16.0$  Hz, 1H), 2.25-2.20 (m, 4H), 1.59 (m, 6H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  170.54, 157.02, 147.26, 146.21, 140.34, 135.35, 135.11, 133.54, 131.94, 131.46, 128.80, 118.64, 115.76, 33.51, 29.73, 27.87; MS (EI)  $m/z$  334 ( $\text{M}^+$ , 100), 253, 150. HRMS (EI)  $m/z$  calcd for  $\text{C}_{22}\text{H}_{22}\text{O}_3$  334.1569, found 334.1573.

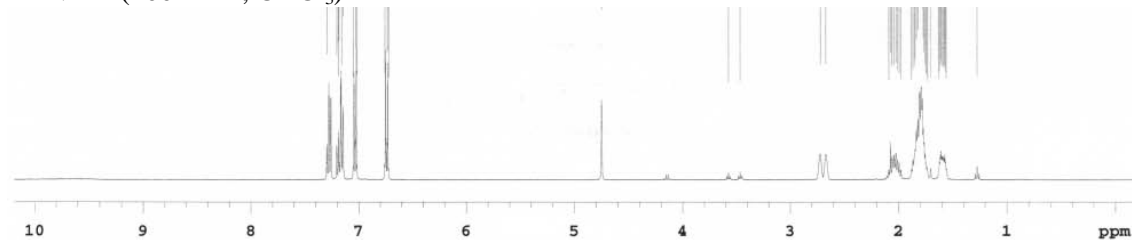
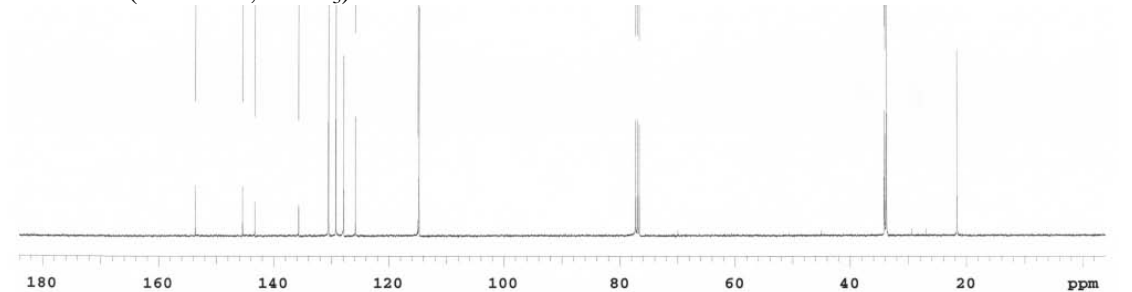
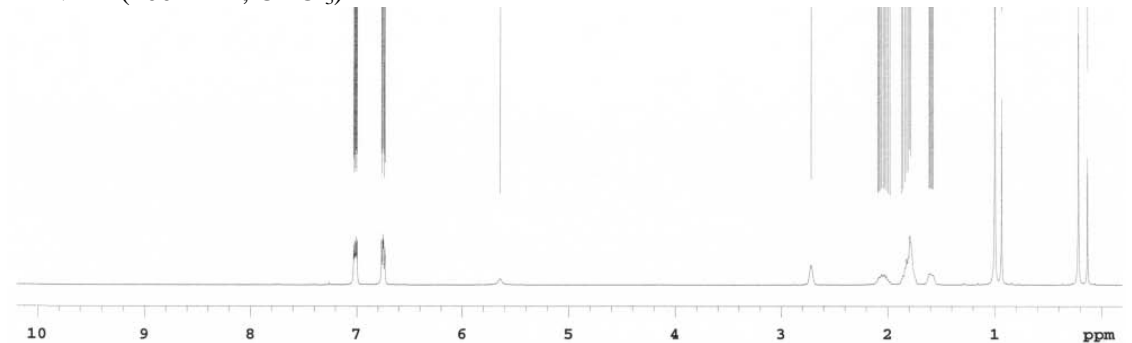
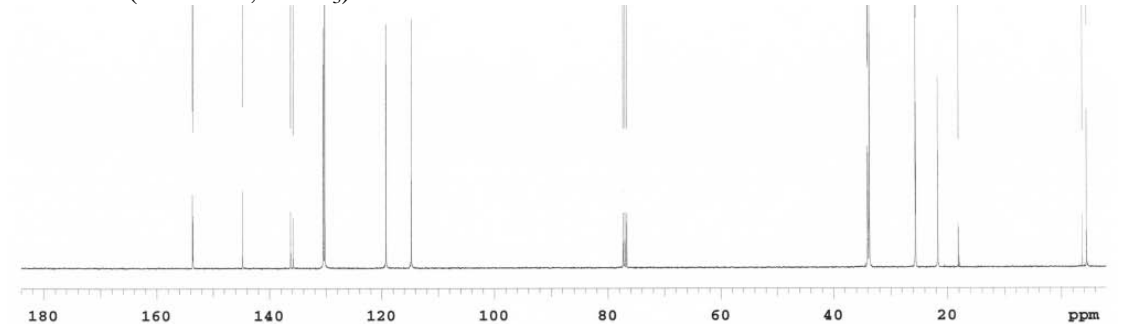
**6-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoic acid (43).** Starting from 0.16 g (0.38 mmol) of the ester **40** and using the general procedure for hydrolysis of ester, 0.14 g (93%) of acid product **43** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  6.94-6.85 (m, 4H), 6.77-6.66 (m, 4H), 3.90 (t,  $J = 6.2$  Hz, 2H), 2.30 (t,  $J = 7.5$  Hz, 2H), 2.22-2.19 (m, 4H), 1.77-1.46 (m, 12H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.68, 158.77, 156.64, 138.58, 137.12, 136.00, 135.52, 131.86, 131.80, 115.55, 114.80, 68.71, 34.91, 33.47, 30.12, 29.75, 27.97, 26.75, 25.85; MS (ESI)  $m/z$  395 ( $\text{M}^+ + 1$ ), 228 (100), 115. HRMS (ESI)  $m/z$  calcd for  $\text{C}_{25}\text{H}_{31}\text{O}_4$  395.2222, found 395.2216.

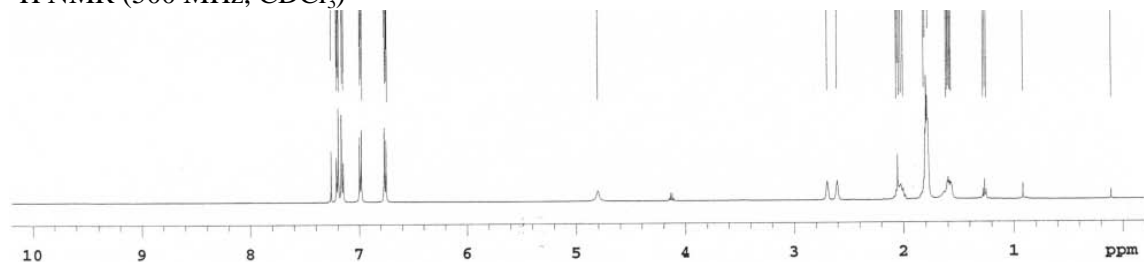
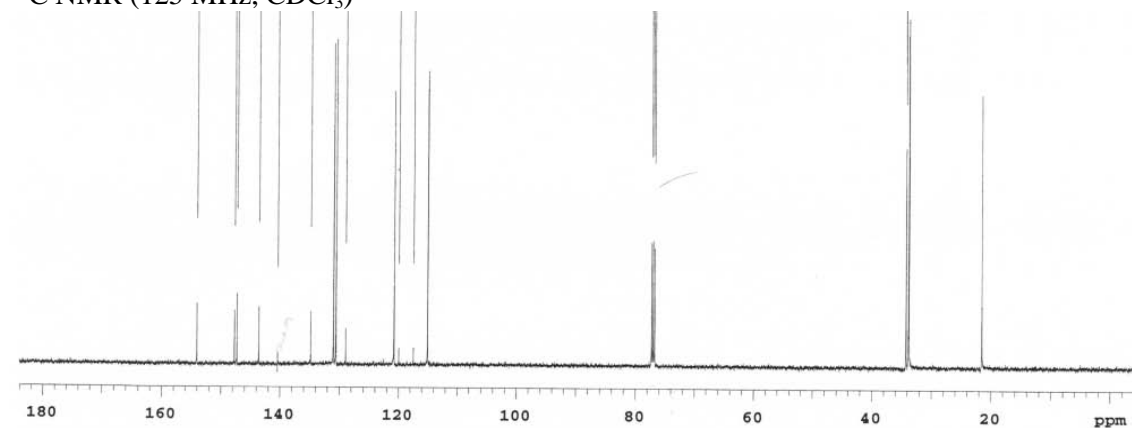
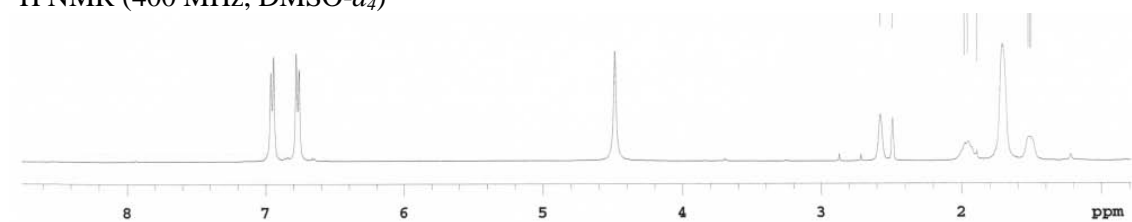
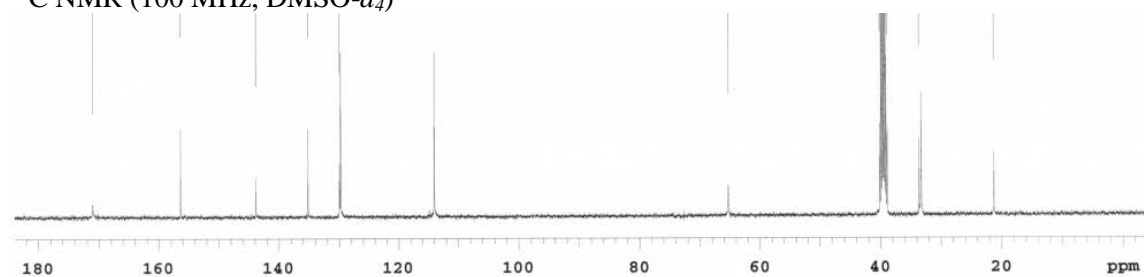
**2-[4-[Cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]acetic acid (44).** Starting from 0.20 g (0.43 mmol) of the ester **41** and using the general procedure for hydrolysis of ester, 0.14 g (95%) of acid product **44** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  6.97 (d,  $J = 8.0$  Hz, 2H), 6.84 (d,  $J = 8.5$  Hz, 2H), 6.81 (d,  $J = 8.0$  Hz, 2H), 6.67 (d,  $J = 8.0$  Hz, 2H), 4.57 (s, 2H), 2.20 (m, 4H), 1.55 (m, 6H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  173.62, 157.72, 156.67, 138.87, 138.02, 135.84, 135.30, 131.83, 115.58, 114.98, 66.25, 33.42, 29.71, 27.93; MS (ESI)  $m/z$  339 ( $\text{M}^+ + 1$ ), 250 (100). HRMS (ESI)  $m/z$  calcd for  $\text{C}_{21}\text{H}_{23}\text{O}_4$  339.1596, found 339.1602.

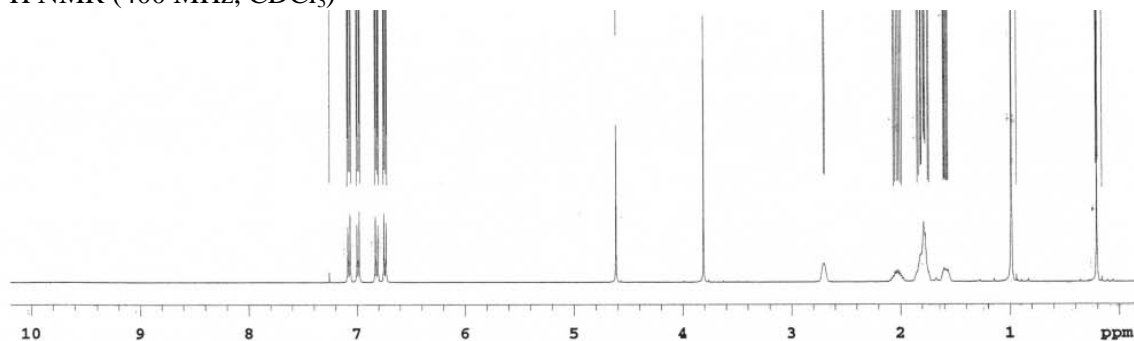
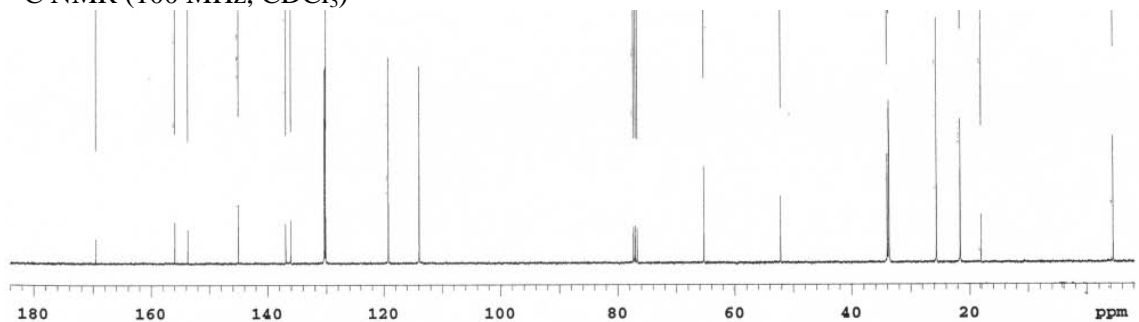
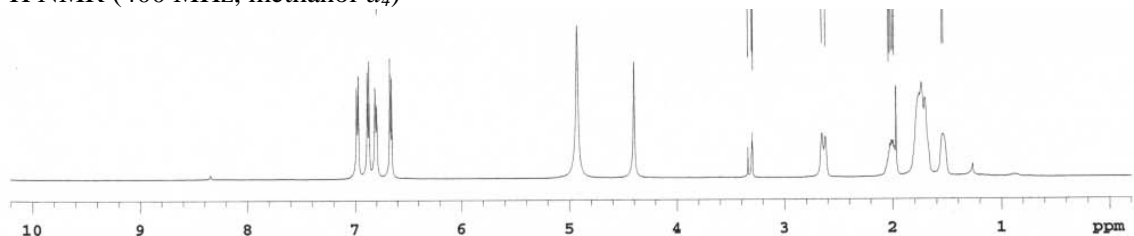
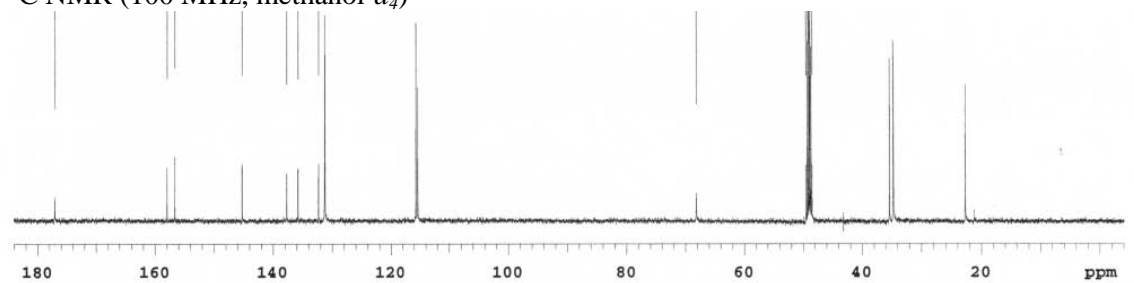
**Ethyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(phenyl)methyl]phenoxy]hexanoate (45).** According to the general procedure for *O*-alkylation reaction with **9** and ethyl 6-bromohexanoate, the yield of compound **29**

was 0.34 g (99%) as a colorless oil:  $^1\text{H}$  NMR (500 MHz,  $\text{CDCl}_3$ )  $\delta$  7.30-7.26 (m, 2H), 7.21-7.17 (m, 3H), 7.09 (d,  $J = 8.5$  Hz, 2H), 6.82 (d,  $J = 8.5$  Hz, 2H), 4.15 (q,  $J = 7.2$  Hz, 2H), 3.95 (t,  $J = 6.2$  Hz, 2H), 2.76-2.70 (m, 2H), 2.35 (t,  $J = 7.5$  Hz, 2H), 2.10-2.01 (m, 2H), 1.90-1.49 (m, 16H), 1.28 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (125 MHz,  $\text{CDCl}_3$ )  $\delta$  174.54, 158.24, 146.16, 144.32, 136.31, 131.76, 131.24, 130.20, 128.82, 126.78, 114.74, 68.35, 61.13, 35.17, 35.09, 34.99, 34.72, 29.93, 26.61, 25.64, 22.62, 15.17; MS (EI)  $m/z$  446( $\text{M}^+$ , 100), 303, 143. HRMS (EI)  $m/z$  calcd for  $\text{C}_{30}\text{H}_{38}\text{O}_3$  446.2821, found 446.2820.

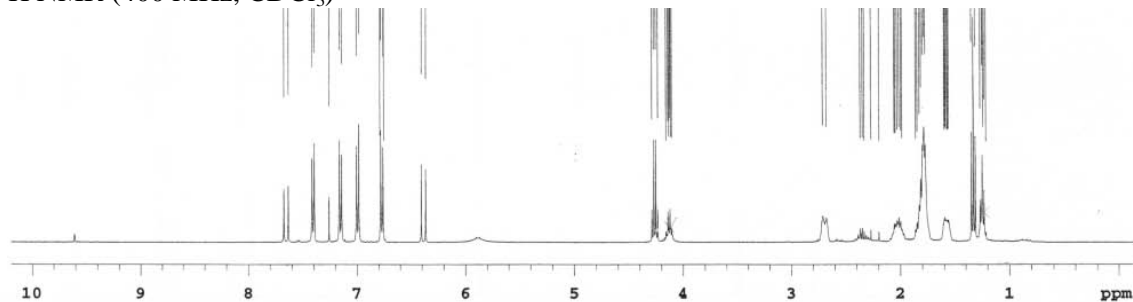
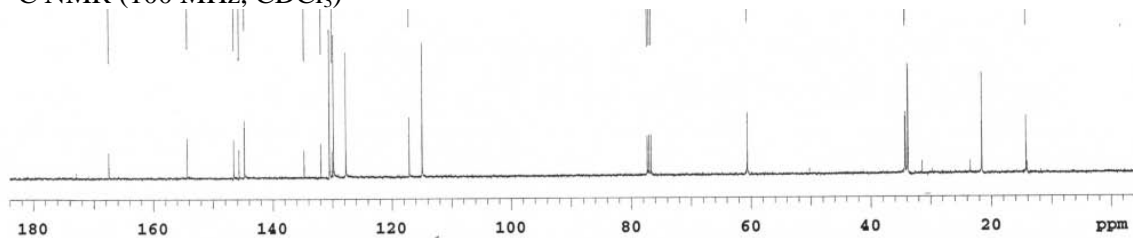
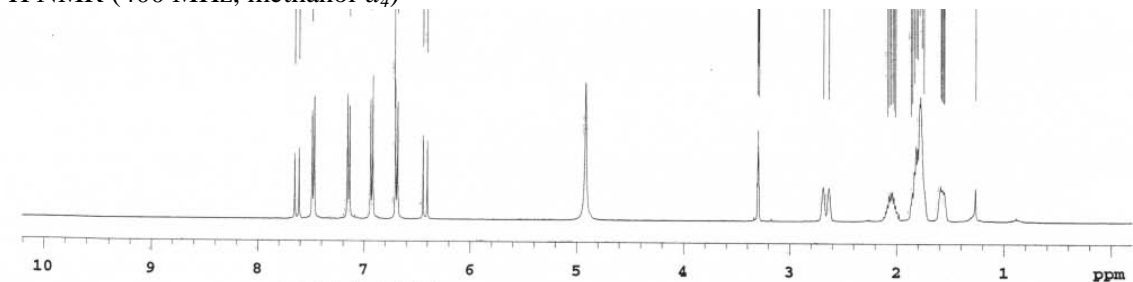
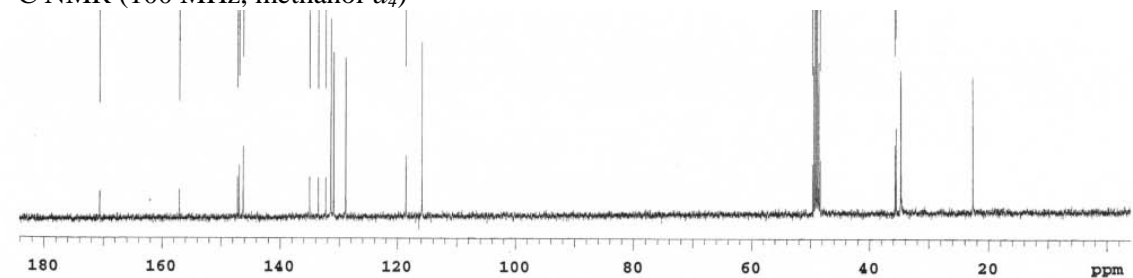
**6-[4-[bicyclo[3.3.1]non-9-ylidene(phenyl)methyl]phenoxy]hexanoic acid (46).** Starting from 0.30 g (0.67 mmol) of the ester **45** and using the general procedure for hydrolysis of ester, 0.27 g (97%) of acid product **46** was obtained:  $^1\text{H}$  NMR (500 MHz, methanol- $d_4$ )  $\delta$  7.23-7.20 (m, 2H), 7.14-7.09 (m, 3H), 7.00 (d,  $J = 9.0$  Hz, 2H), 6.77 (d,  $J = 9.0$  Hz, 2H), 3.89 (t,  $J = 6.5$  Hz, 2H), 2.68-2.60 (m, 2H), 2.29 (t,  $J = 7.2$  Hz, 2H), 2.08-1.99 (m, 2H), 1.85-1.44 (m, 16H);  $^{13}\text{C}$  NMR (125 MHz, methanol- $d_4$ )  $\delta$  177.68, 158.90, 145.89, 144.63, 136.52, 132.74, 131.26, 130.22, 128.96, 127.03, 115.01, 68.74, 35.51, 35.38, 34.95, 34.76, 30.13, 26.78, 25.87, 22.72 ; MS (EI)  $m/z$  418 ( $\text{M}^+$ , 100), 221, 69. HRMS (EI)  $m/z$  calcd for  $\text{C}_{28}\text{H}_{34}\text{O}_3$  418.2508, found 418.2507.

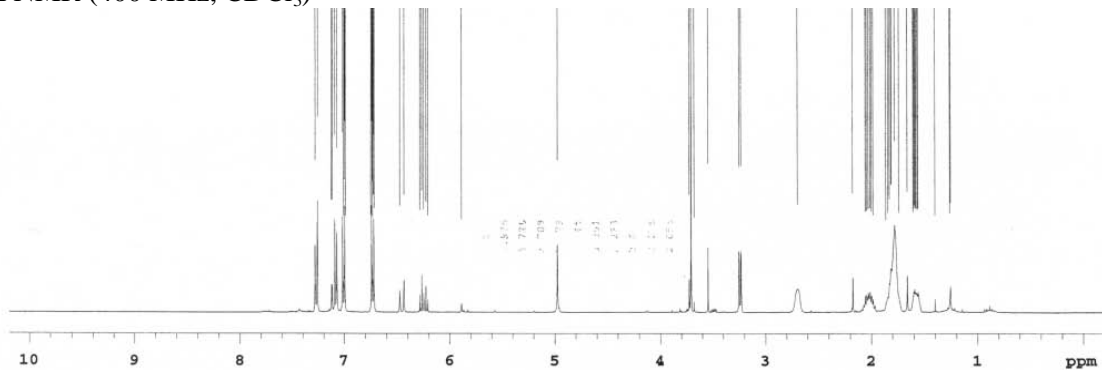
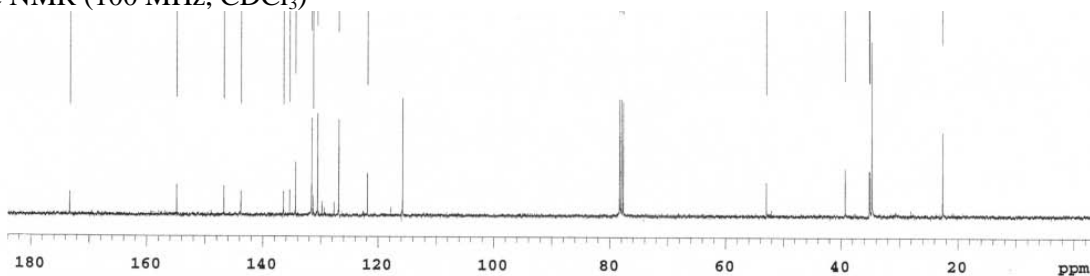
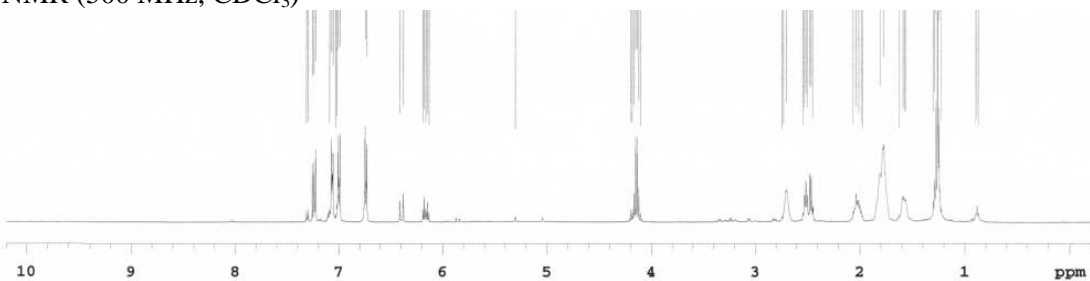
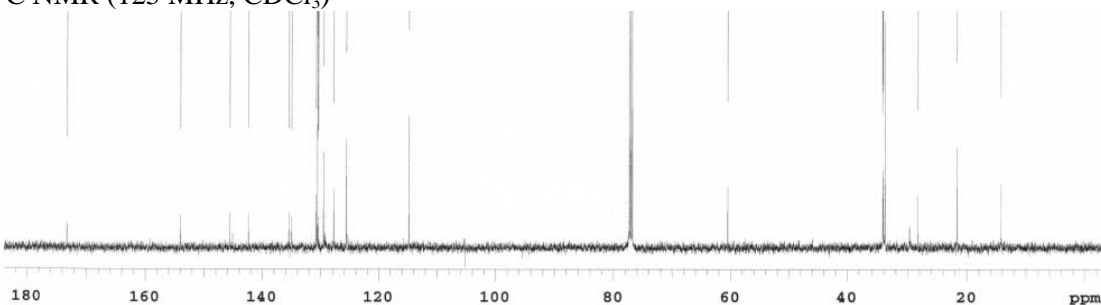
**II.  $^1\text{H}$  and  $^{13}\text{C}$  NMR Spectra****9-[(4-hydroxyphenyl)(phenyl)methylene]bicyclo[3.3.1]-nonane (9).** $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )**9-[(4-*tert*-Butyldimethylsilyloxyphenyl)(4-hydroxyphenyl)methylene]bicyclo[3.3.1]-nonane (10).** $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ ) $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )

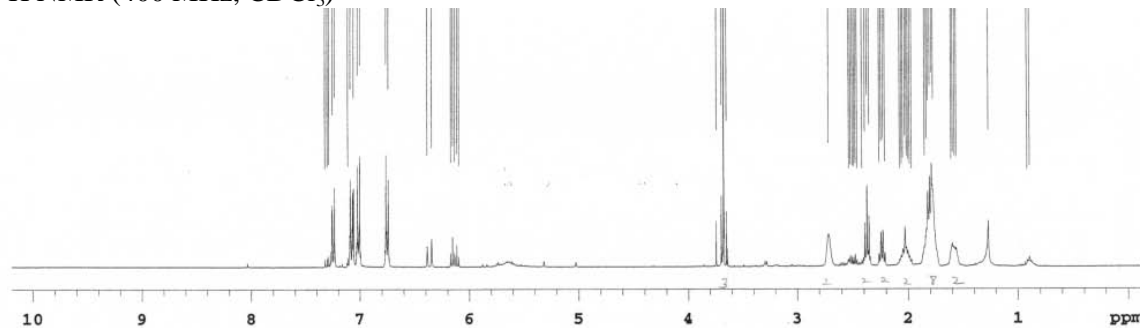
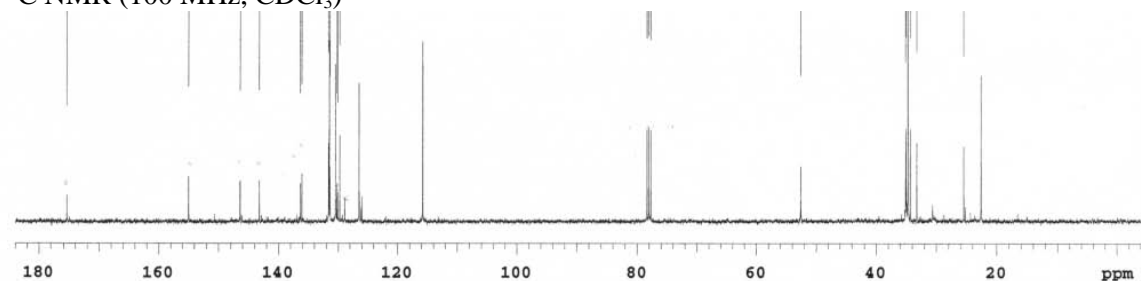
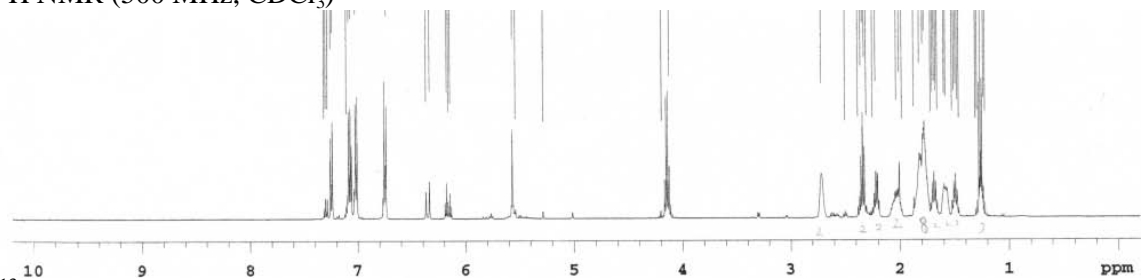
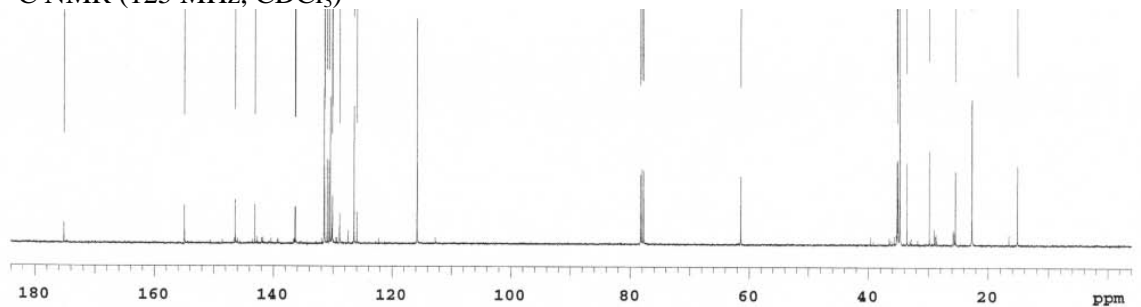
**9-[(4-Trifluoromethanesulfonylphenyl) (4-hydroxyphenyl) methylene]bicyclo[3.3.1]-nonane (11).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)**2,2'-[4,4'-(Bicyclo[3.3.1]non-9-ylidenemethylene)bis(4,1-phenylene)]bis(oxy)diacetic acid (12).**<sup>1</sup>H NMR (400 MHz, DMSO-*d*<sub>4</sub>)<sup>13</sup>C NMR (100 MHz, DMSO-*d*<sub>4</sub>)

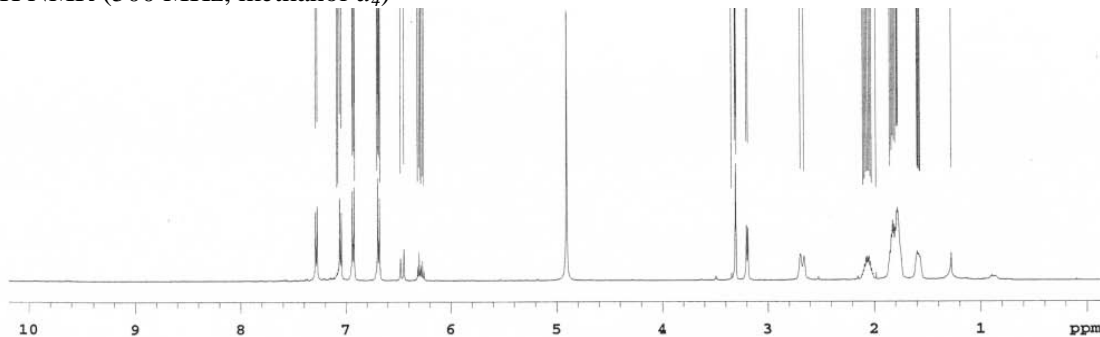
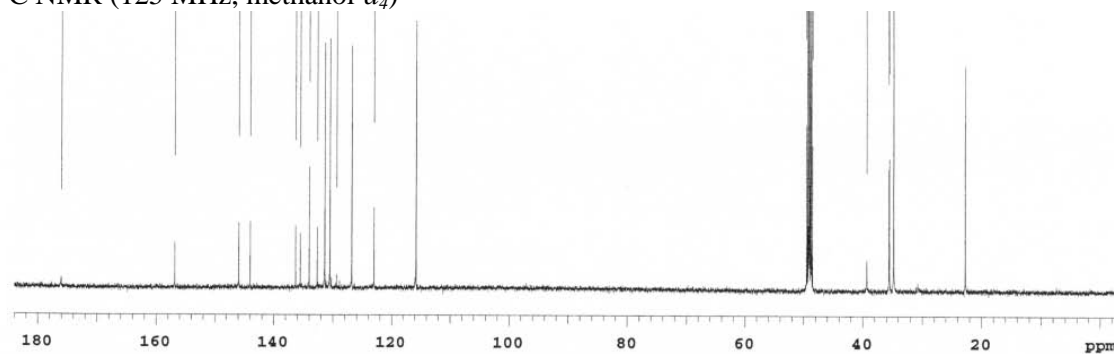
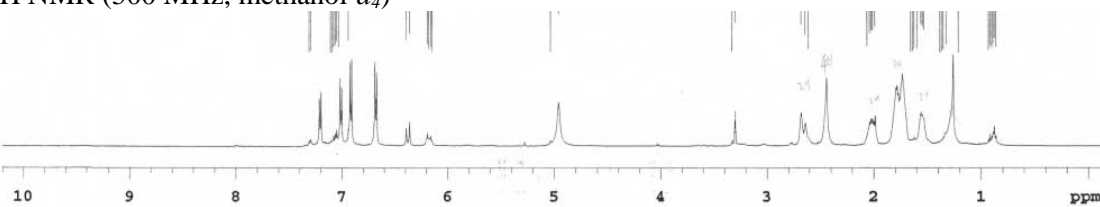
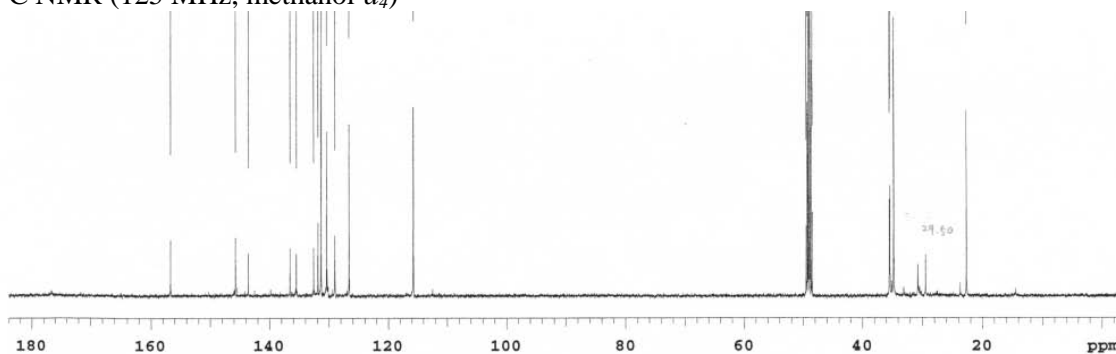
**Methyl 2-[4-[bicyclo[3.3.1]non-9-ylidene[4-(*tert*-butyldimethylsilyloxyphenyl)methyl]phenoxy]acetate (14).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**2-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]acetic acid (15).**<sup>1</sup>H NMR (400 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (100 MHz, methanol-*d*<sub>4</sub>)

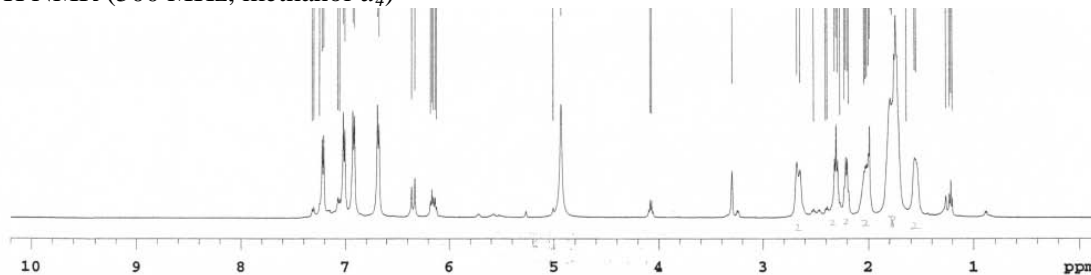
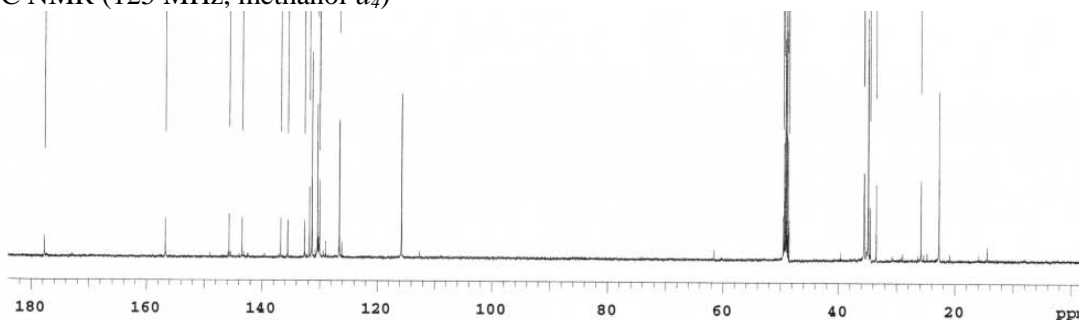
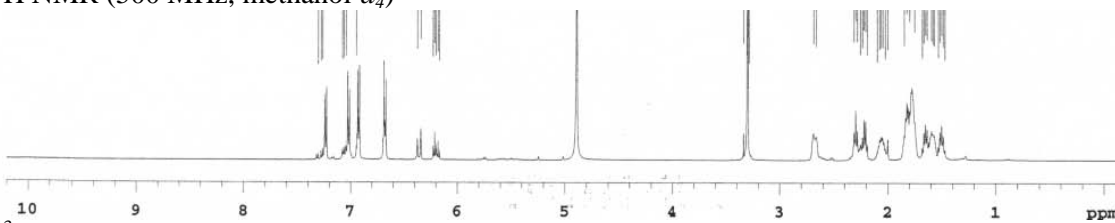
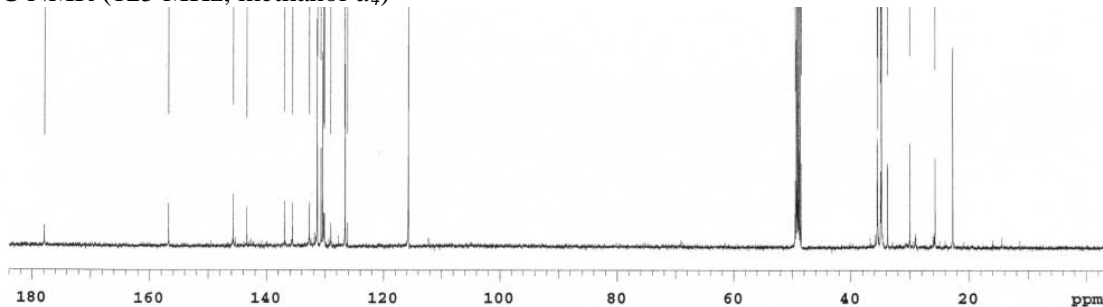


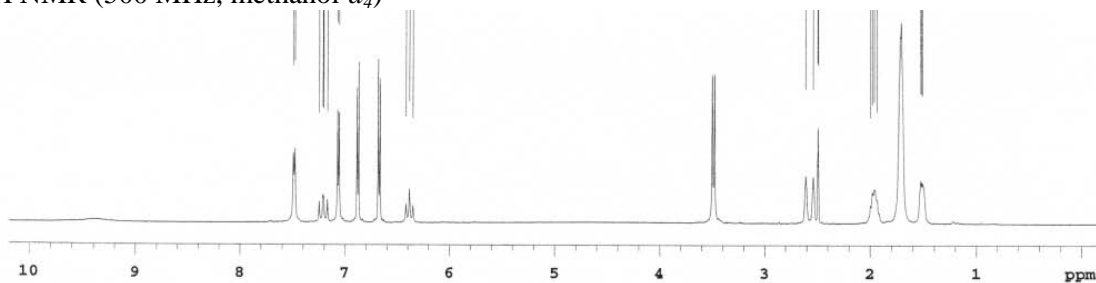
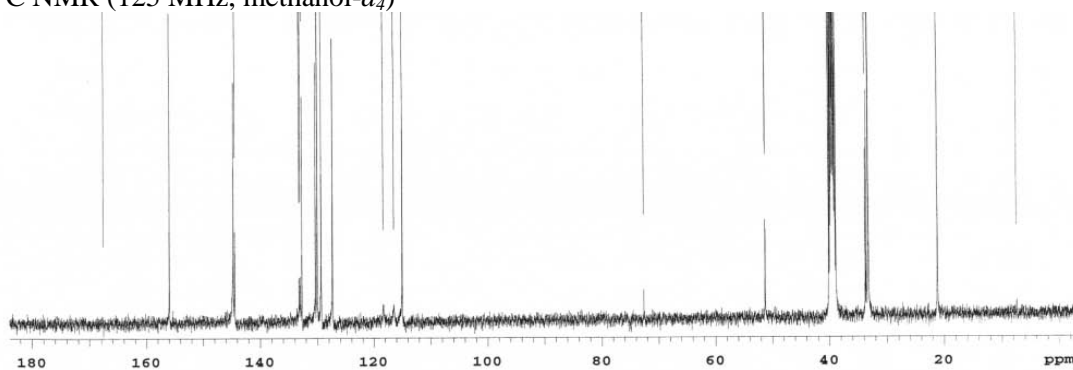
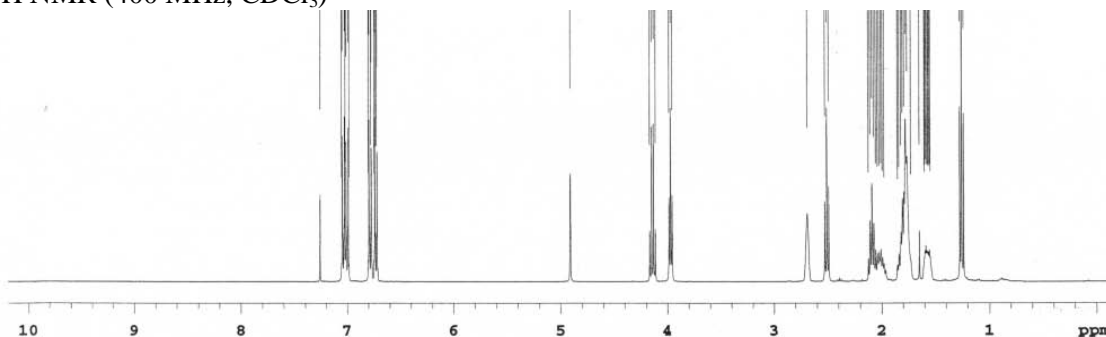
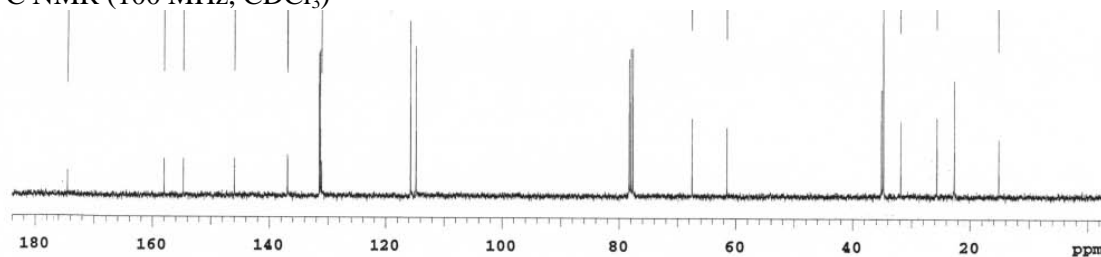
**(E)-Ethyl 3-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]acrylate (16).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**(E)-3-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]acrylic acid (17).**<sup>1</sup>H NMR (400 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (100 MHz, methanol-*d*<sub>4</sub>)

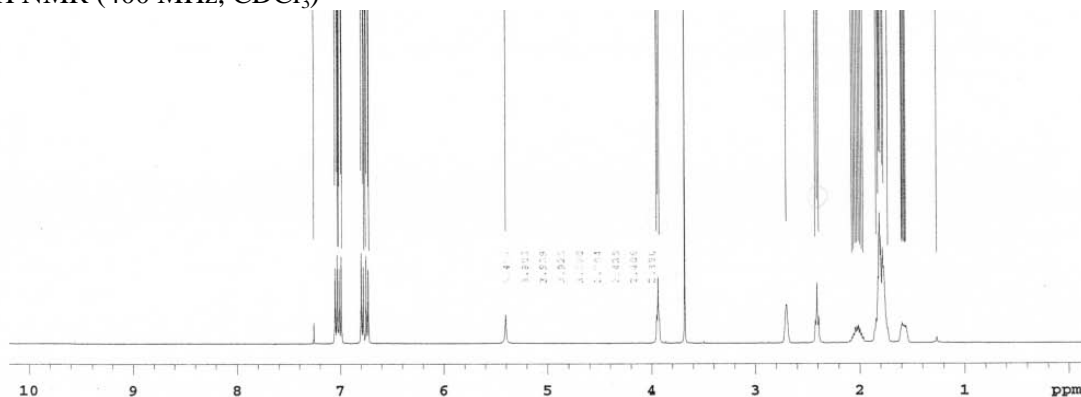
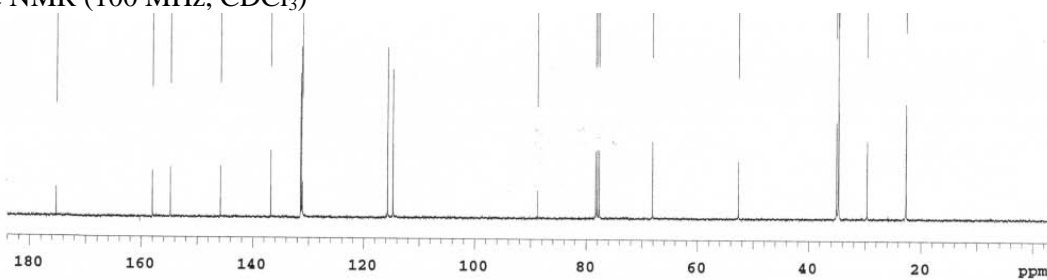
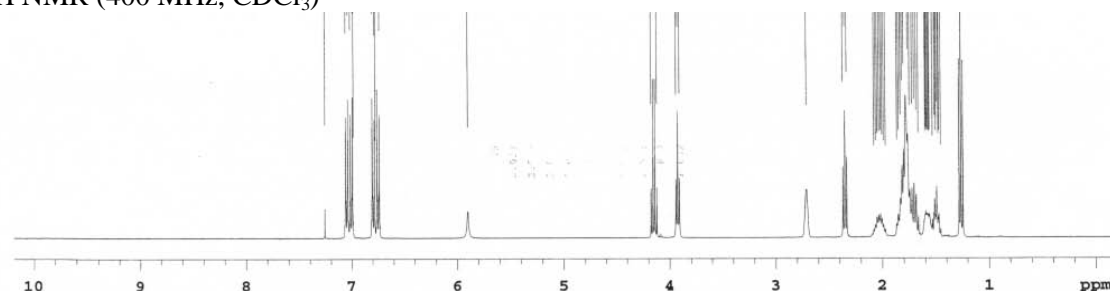
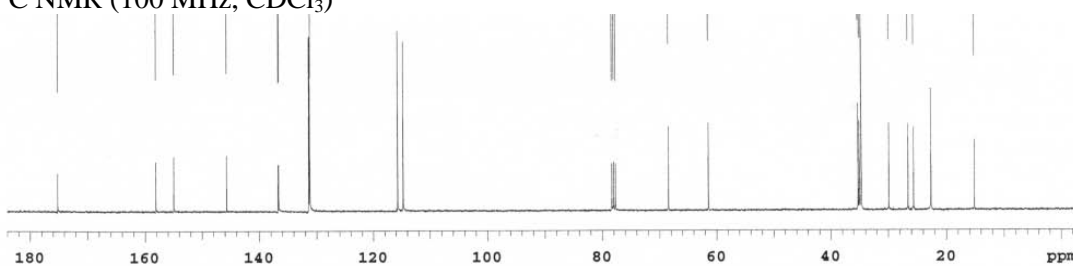
**(E)-Methyl 4-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]but-3-enoate (18).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**(E)-Ethyl 5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]pent-4-enoate (19).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

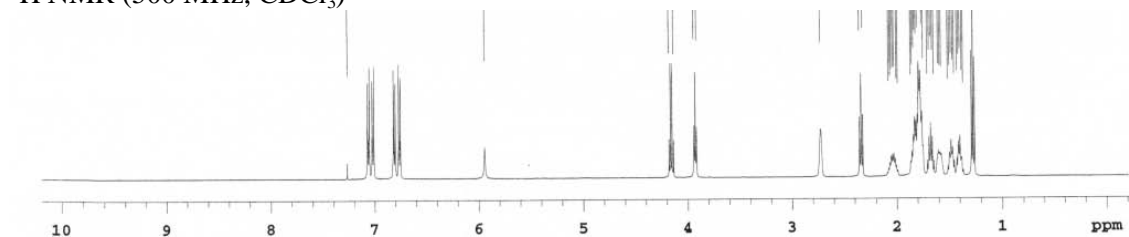
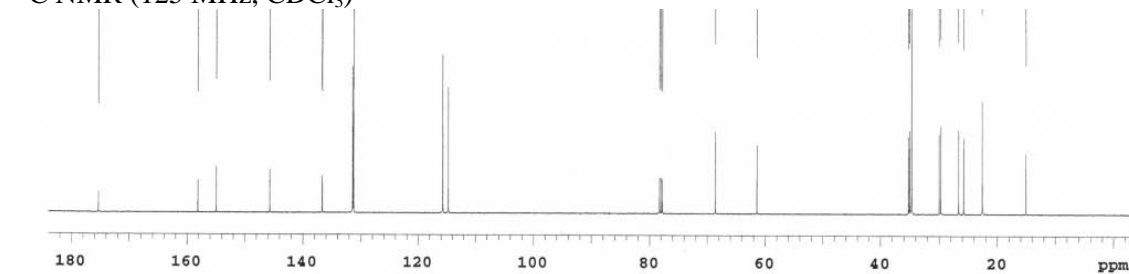
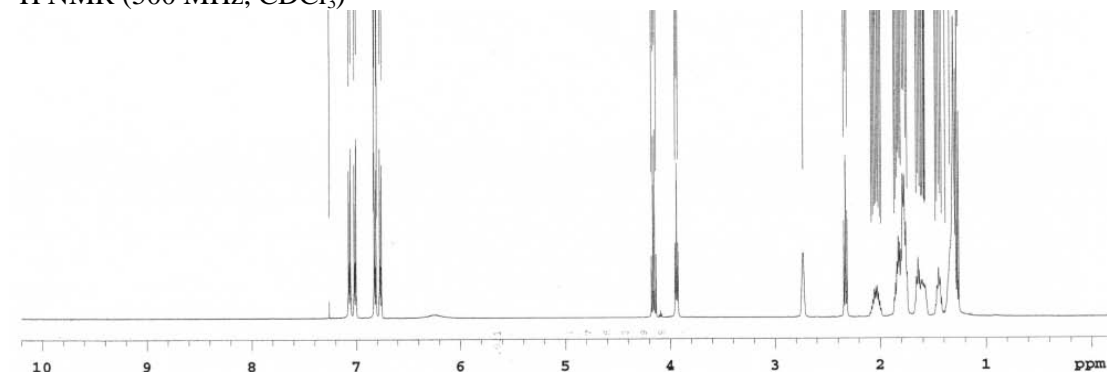
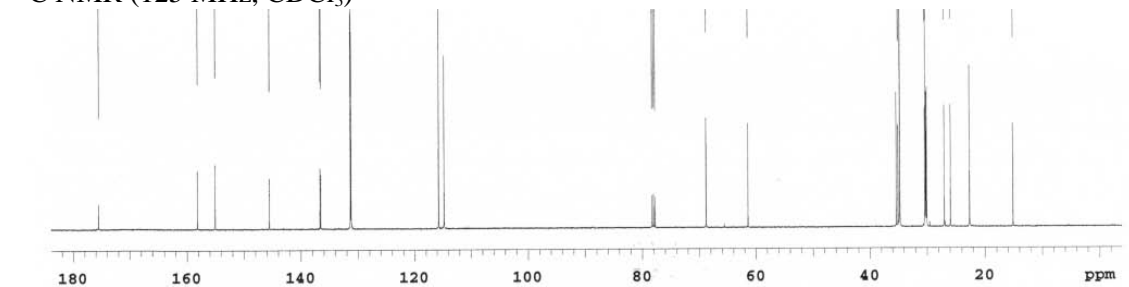
**(E)-Methyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hex-5-enoate (20).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**(E)-Ethyl 7-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hept-6-enoate (21).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

**(E)-4-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]but-3-enoic acid (22).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**(E)-5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]pent-4-enoic acid (23).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)

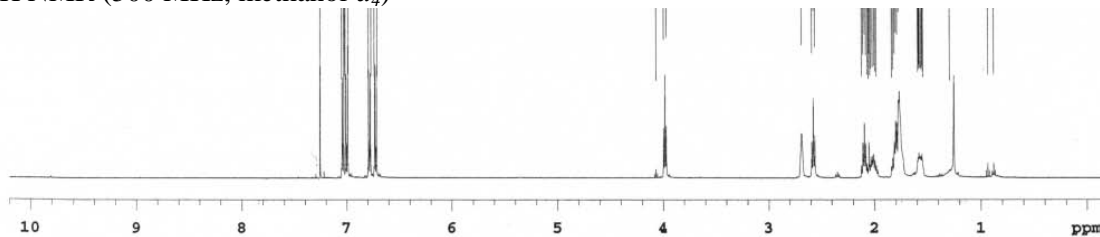
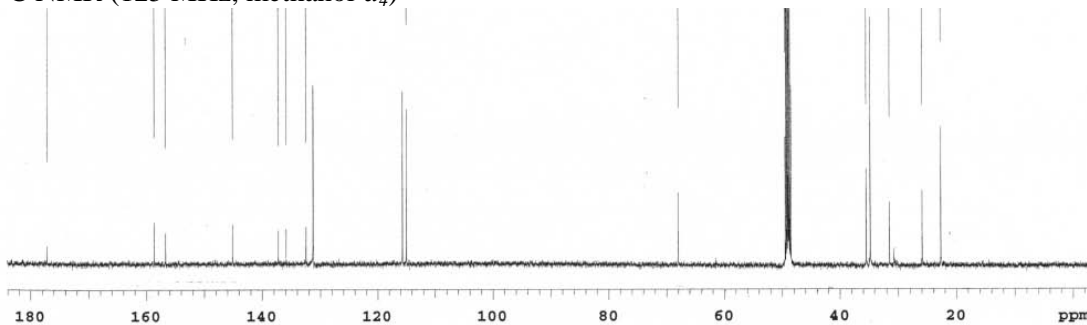
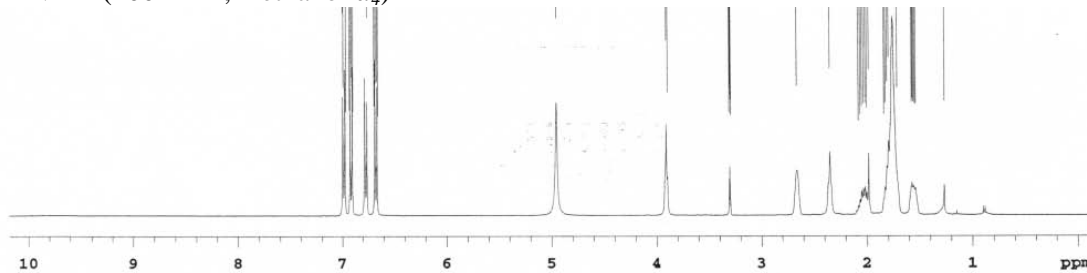
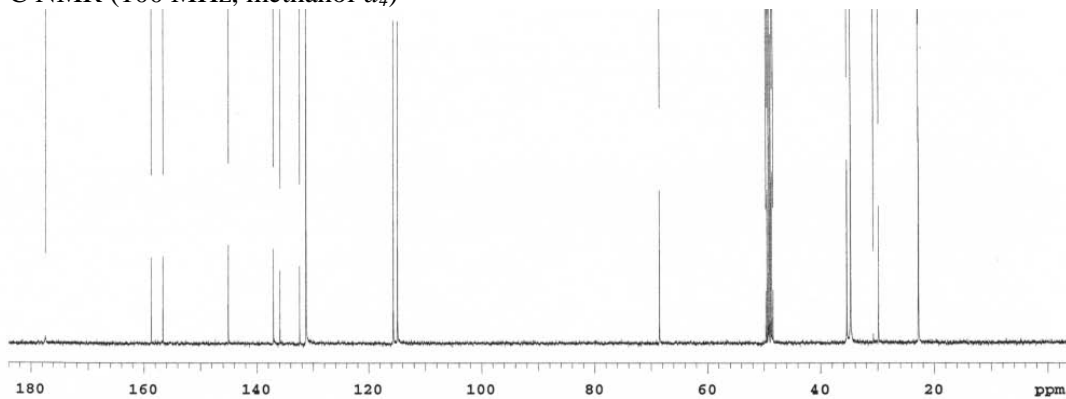
**(E)-6-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hex-5-enoic acid (24).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**(E)-7-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hept-6-enoic acid (25).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)

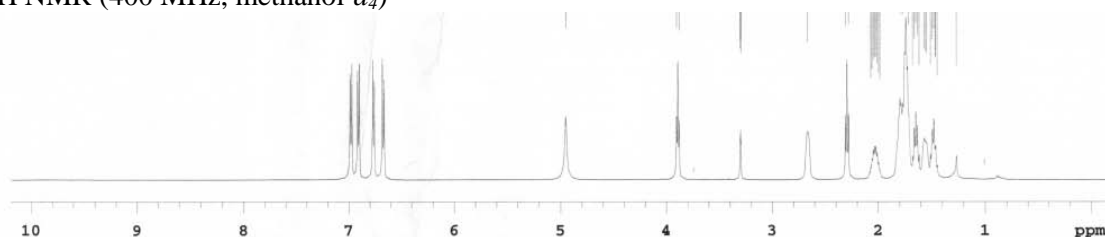
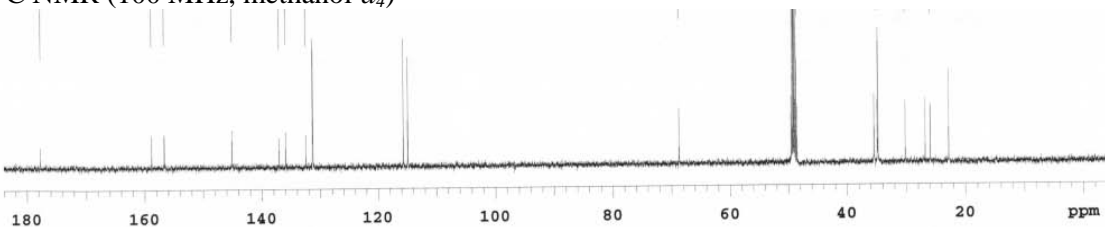
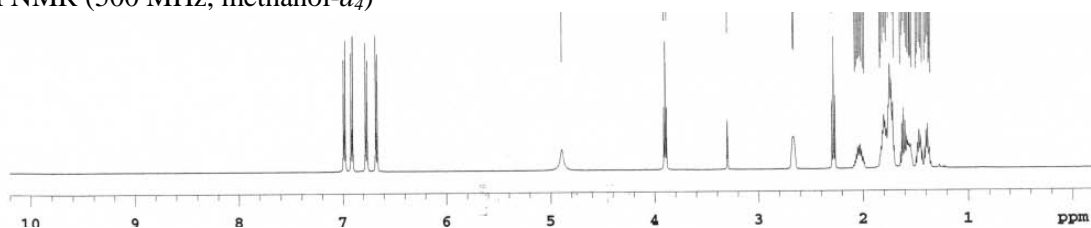
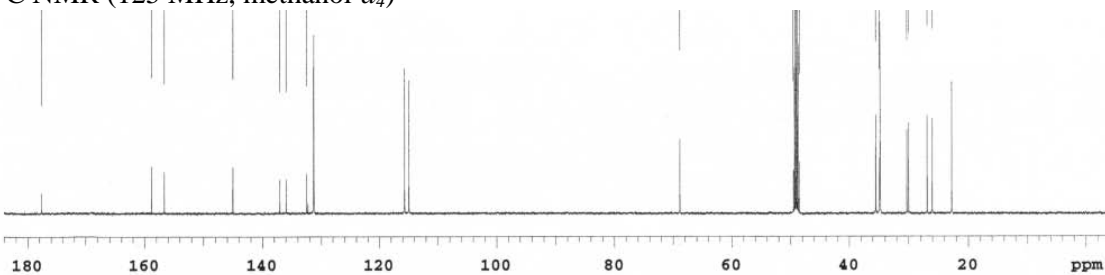
**Methyl hydrogen 4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]styrylphosphonate (26).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**Ethyl 4-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]butanoate (27).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

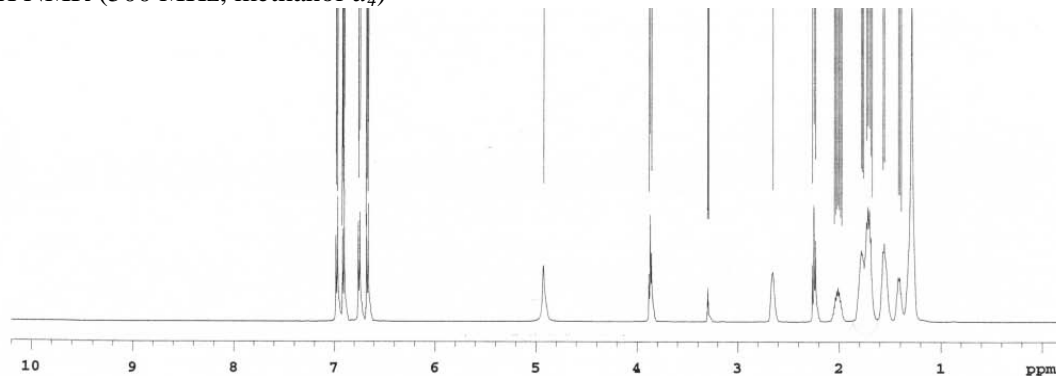
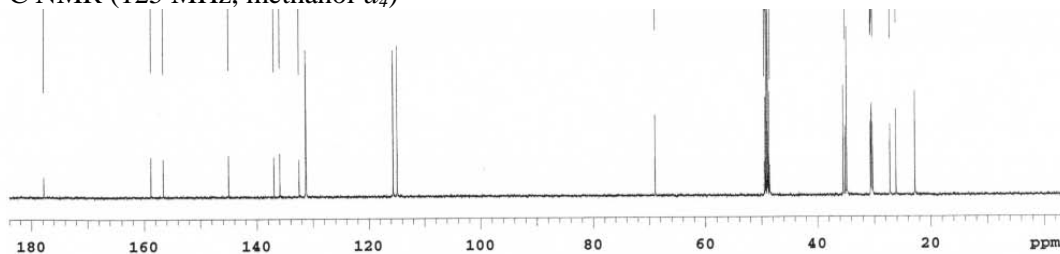
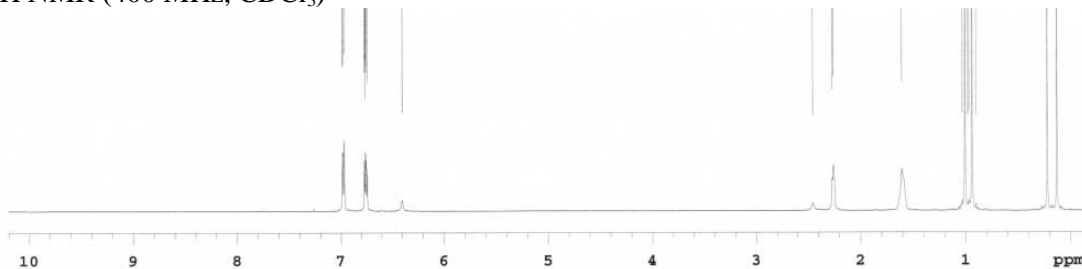
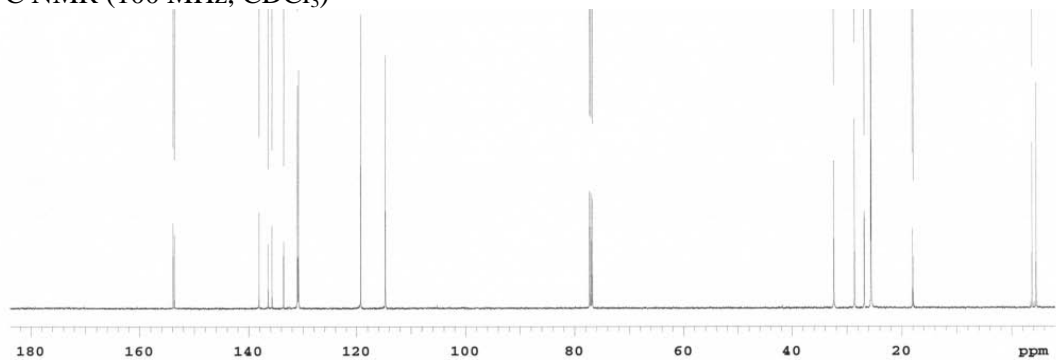
**Methyl 5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]pentanoate (28).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)**Ethyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoate (29).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

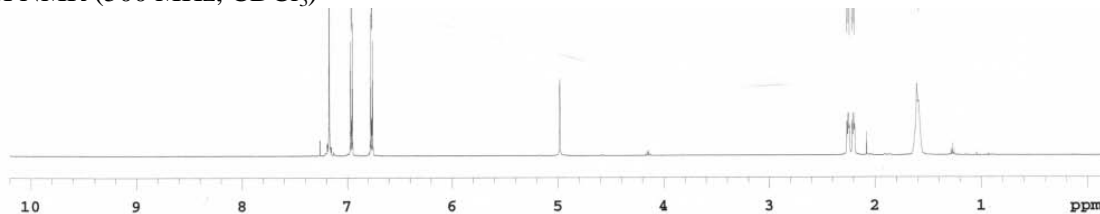
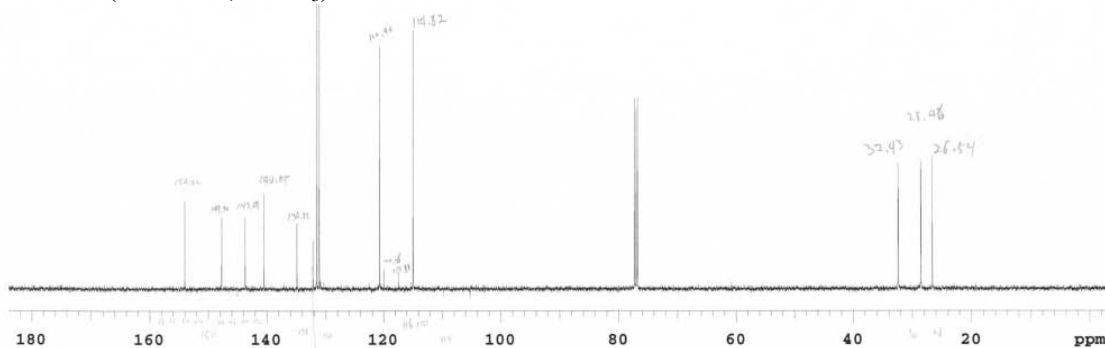
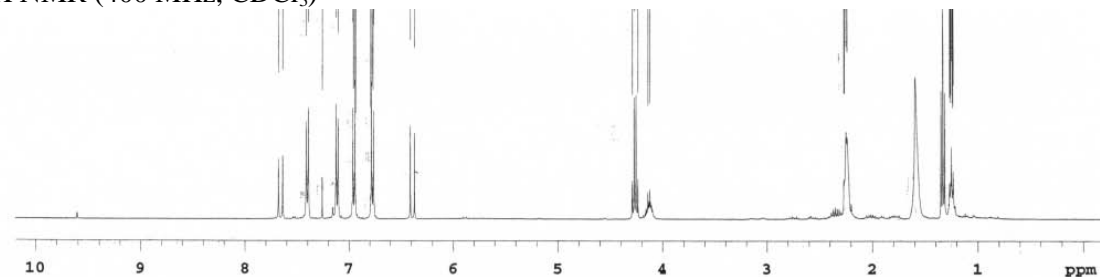
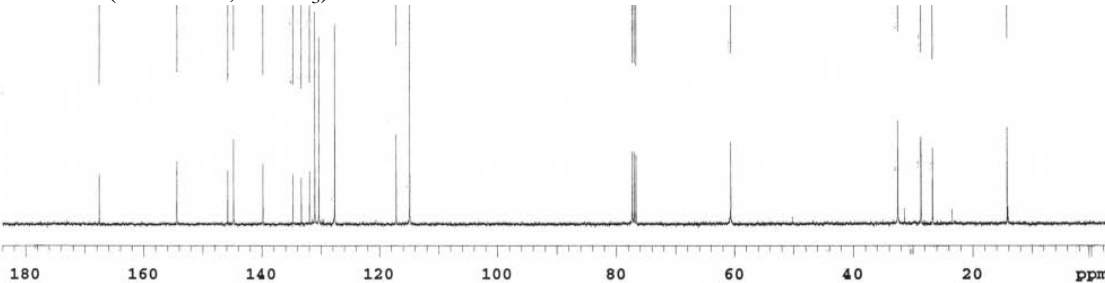
**Ethyl 7-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]heptanoate (30).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)**Ethyl 11-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]nudecanoate (31).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

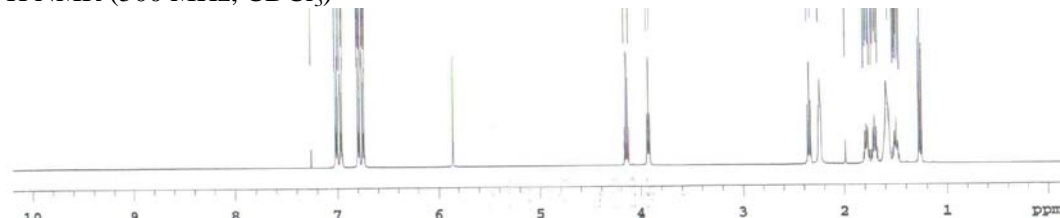
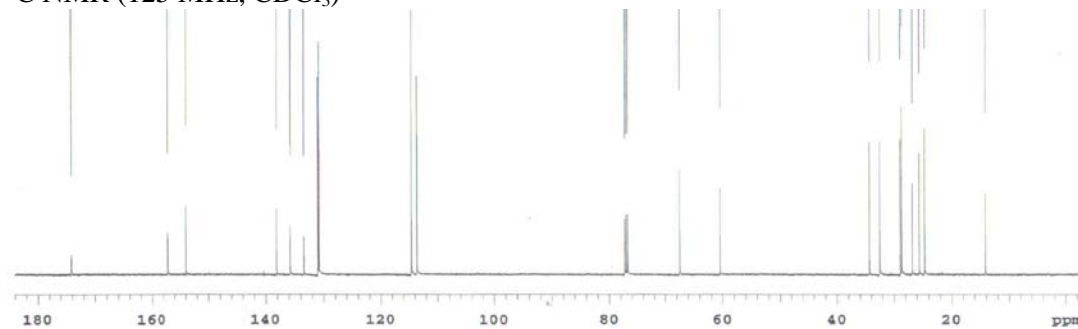
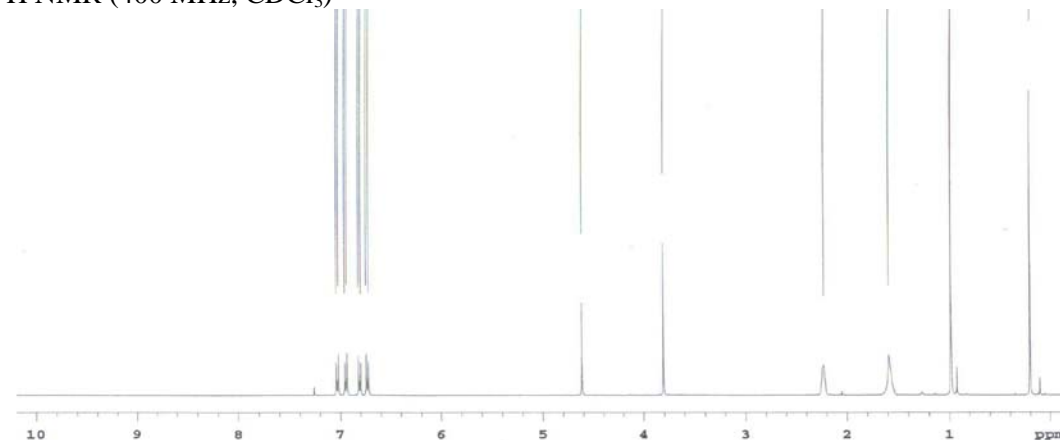
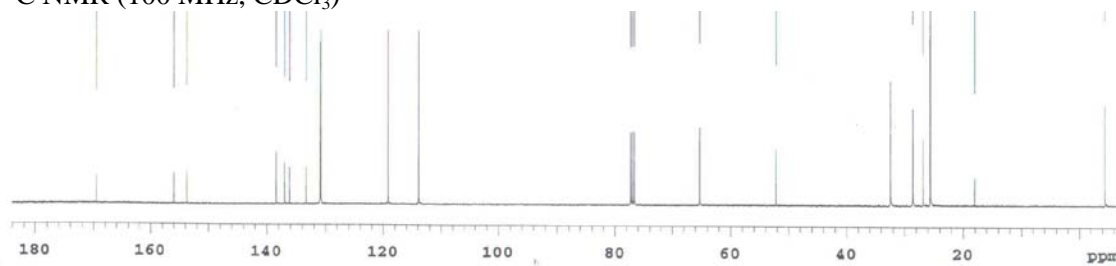


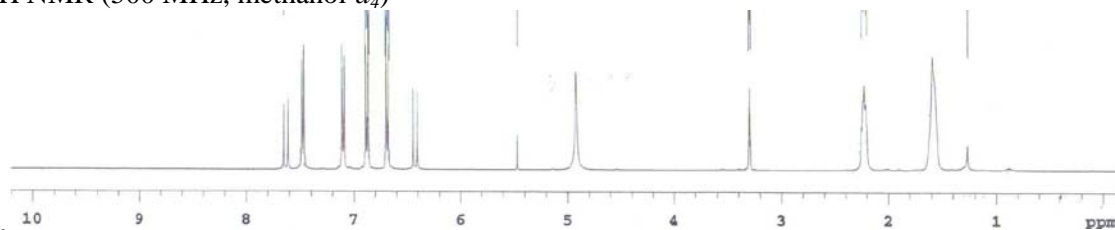
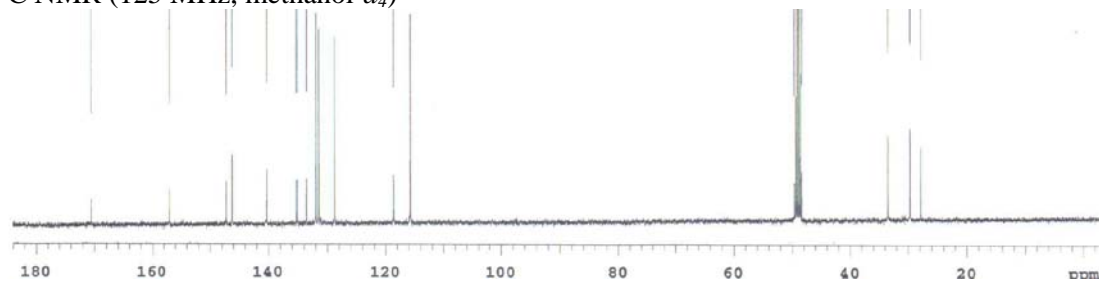
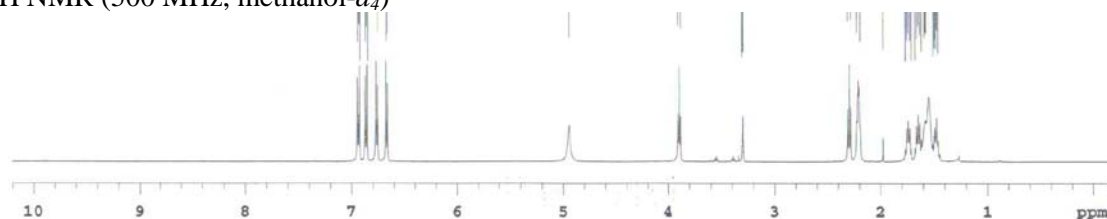
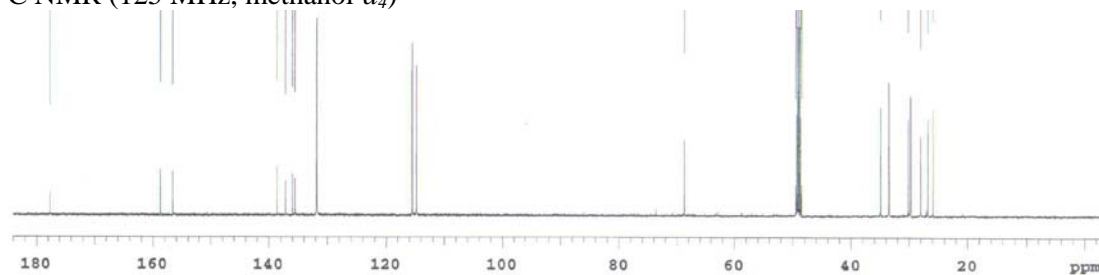
**4-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]butanoic acid (32).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**5-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]pentanoic acid (33).**<sup>1</sup>H NMR (400 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (100 MHz, methanol-*d*<sub>4</sub>)

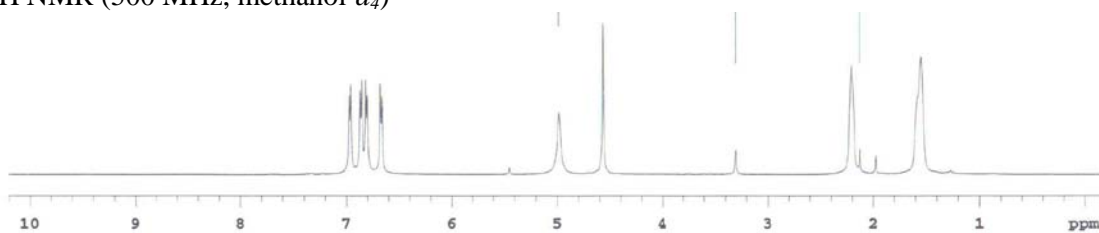
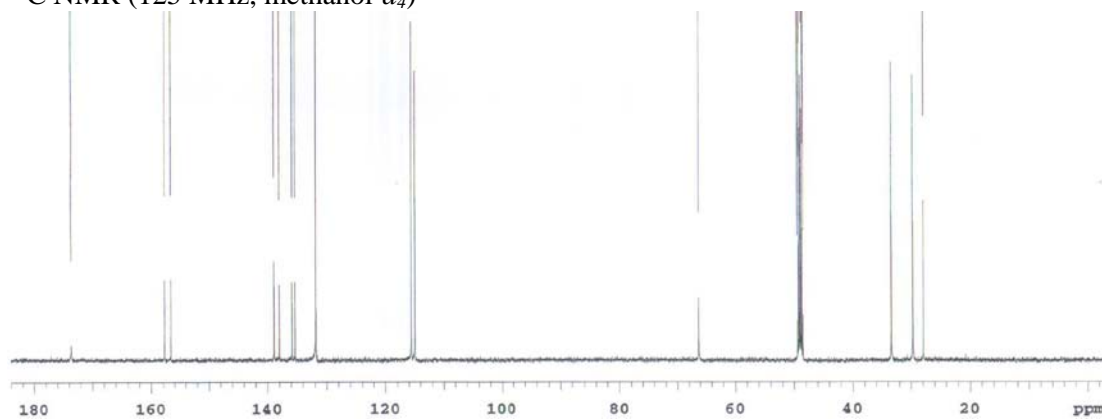
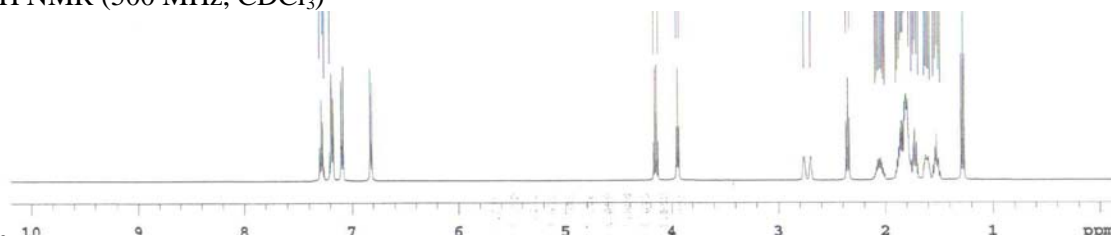
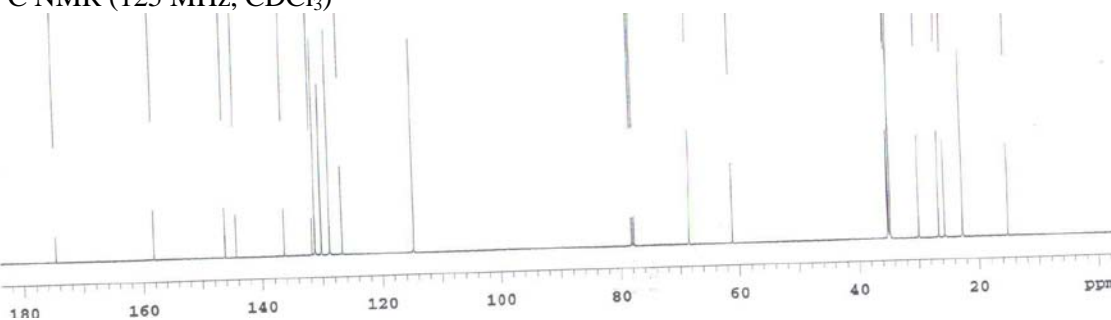
**6-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoic acid (34).**<sup>1</sup>H NMR (400 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (100 MHz, methanol-*d*<sub>4</sub>)**7-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]heptanoic acid (35).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)

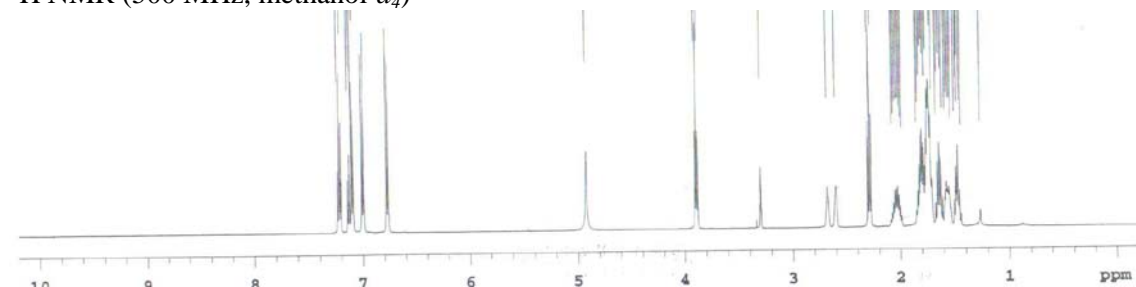
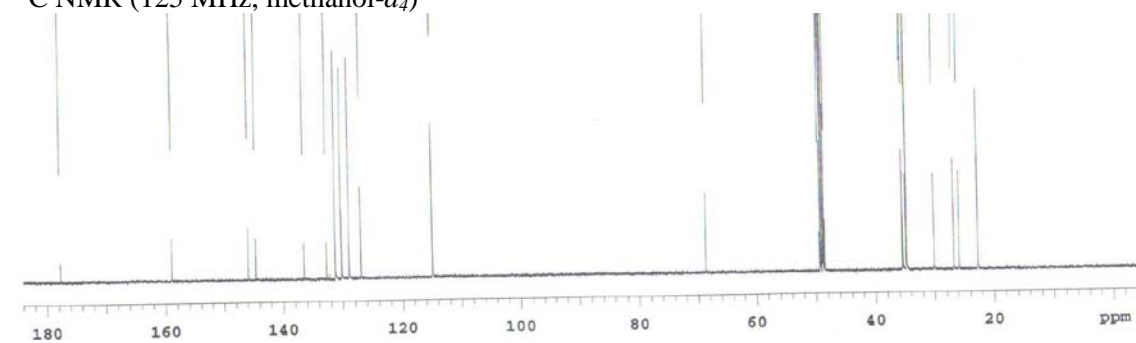
**11-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]nudecanoic acid (36).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**(4-*tert*-Butyldimethylsilyloxyphenyl) (4-hydroxyphenyl) methylenecyclohexane (37).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

**(4-Hydroxyphenyl) (4-trifluoromethanesulfonylphenyl) methylenecyclohexane (38).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)**(E)-ethyl 3-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenyl]acrylate (39).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

**Ethyl 6-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoate (40).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)**Methyl 2-[4-[cyclohexylidene(4-*tert*-butyldimethylsilyloxyphenyl)methyl]phenoxy]acetate (41).**<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

**(E)-3-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenyl]acrylic acid (42).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**6-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoic acid (43).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)

**2-[4-[Cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]acetic acid (44).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)**Ethyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(phenyl)methyl]phenoxy]hexanoate (45).**<sup>1</sup>H NMR (500 MHz, CDCl<sub>3</sub>)<sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>)

**6-[4-[bicyclo[3.3.1]non-9-ylidene(phenyl)methyl]phenoxy]hexanoic acid (46).**<sup>1</sup>H NMR (500 MHz, methanol-*d*<sub>4</sub>)<sup>13</sup>C NMR (125 MHz, methanol-*d*<sub>4</sub>)



**HPLC Spectra**

**9-[(4-Trifluoromethanesulfonylphenyl) (4-hydroxyphenyl) methylene]bicyclo[3.3.1]-nonane (11).**

Reverse Phase (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

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Printed: 05/22/2006

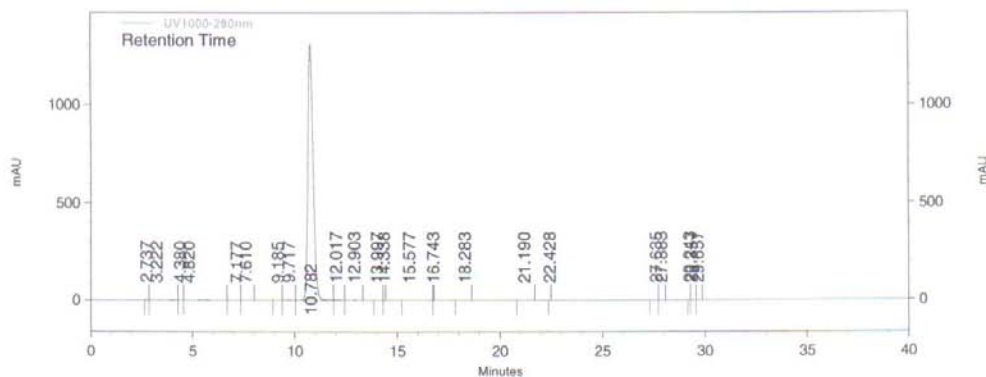
03:11:09 AM

Analyst: System

Sample ID: dwk-ii-47

Vial: N/A

Injection Volume: 0



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 (5/5/2006  
 12:22:50 AM)  
 (Reprocessed))

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	4.820	73886	0.316	VD
	7.177	6645	0.028	VV
	7.610	5533	0.024	VB
	9.185	2809	0.012	BV
	9.717	13601	0.058	VV
	10.782	23135761	98.986	VD
	12.017	13090	0.056	VV
	12.903	10258	0.044	VB
	13.997	722	0.003	BB
	14.338	79	0.000	BB
	15.577	2208	0.009	BD
	16.743	36	0.000	VB
	18.283	3371	0.014	BB

**Normal Phase** (Method EtOAc:Hx:AcOH 10:90:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

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 04:07:15 PM

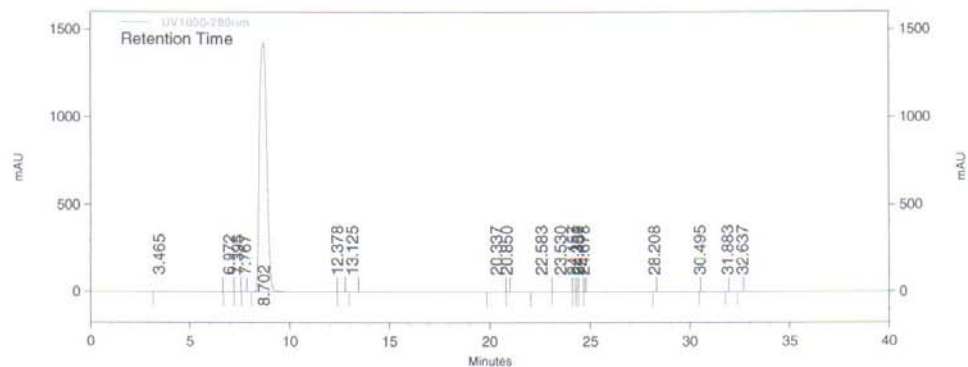
Printed: 05/10/2006

Analyst: System

Sample ID: dwk-ii-47

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/10/2006  
 4:02:25 PM)  
 (Original))

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	7.767	554	0.002	BB
	8.702	35829871	99.596	BD
	12.378	1152	0.003	VB
	13.125	1073	0.003	BB
	20.337	9400	0.026	BD
	20.850	303	0.001	VB
	22.583	18037	0.050	BV
	23.530	21888	0.061	VV
	24.157	992	0.003	VV
	24.350	578	0.002	VV
	24.437	747	0.002	VV
	24.678	101	0.000	VB
	28.208	152	0.000	BB

**2-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]acetic acid (15).****Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 20:80:0.1, Flow rate 1mL/min)**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-i-195rp-5.dat

Acquired: 5/2/2006 11:33:51 PM

Printed: 05/03/2006

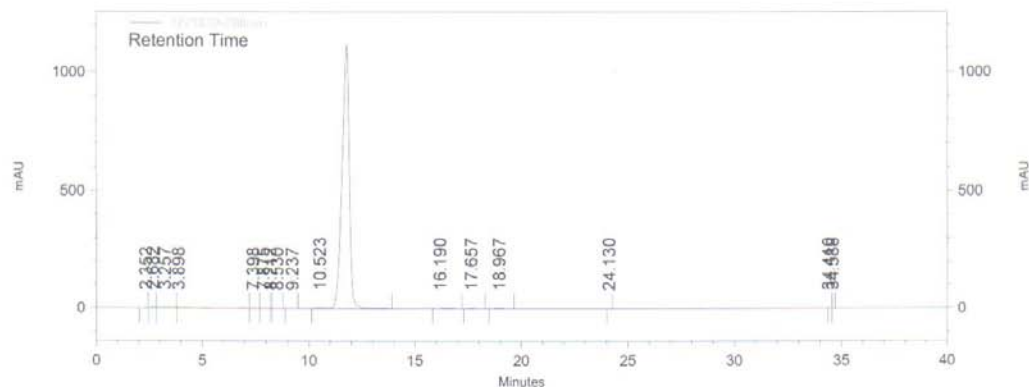
12:39:00 AM

Analyst: System

Sample ID: dwk-i-195

Vial: N/A

Injection Volume: 0



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 Results (System  
 (5/3/2006  
 12:33:58 AM)  
 (Original))

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	3.898	79825	0.301	VD
	7.398	6391	0.024	VV
	7.875	4595	0.017	VV
	8.212	225	0.001	VV
	8.530	2328	0.009	VB
	9.237	2443	0.009	BB
	10.523	26281173	99.065	BD
	16.190	21817	0.082	BD
	17.657	9342	0.035	BB
	18.967	13668	0.052	BB
	24.130	206	0.001	BD
	34.410	146	0.001	BV
	34.588	120	0.000	VB

Normal Phase (Method *i*-PrOH:CH<sub>2</sub>Cl<sub>2</sub>:AcOH 10:90:0.5, Flow rate 1mL/min)

## Area Percent Report

Page 1 of 2

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Printed: 05/22/2006

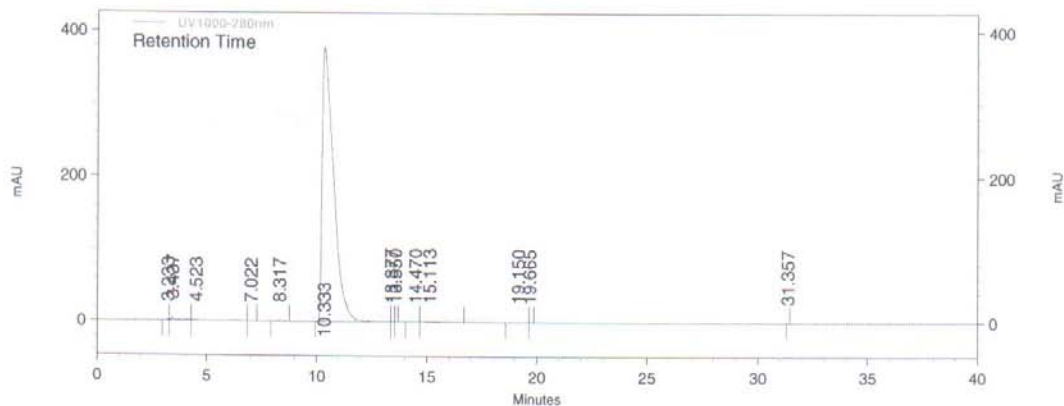
03:04:51 AM

Analyst: System

Sample ID: dwk-i-195

Vial: N/A

Injection Volume: 0



UV1000-280nm  
Results (System  
(5/22/2006  
3:04:33 AM)  
(Reprocessed)  
(Aborted Run))

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	7.022	1545	0.011	VB
	8.317	13786	0.101	BB
	10.333	13570550	99.047	BD
	13.377	564	0.004	VV
	13.550	155	0.001	VB
	14.470	5086	0.037	BV
	15.113	14119	0.103	VD
	19.150	7007	0.051	BV
	19.665	283	0.002	VD
	31.357	129	0.001	BB

**(E)-3-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]acrylic acid (17).****Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 20:80:0.1, Flow rate 1mL/min)**Area Percent Report**

Page 1 of 2

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Acquired: 5/3/2006 12:16:03 PM

Printed: 05/22/2006

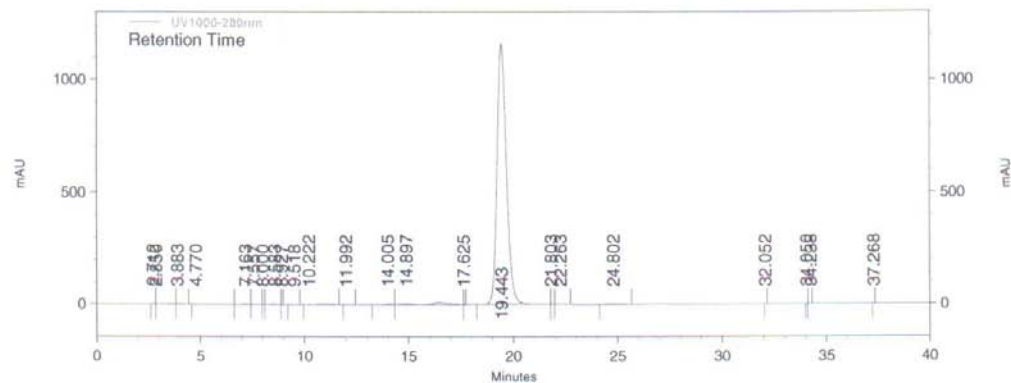
03:10:14 AM

Analyst: System

Sample ID: dwk-i-211

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/3/2006  
 1:16:11 PM)  
 (Original))

Name	Retention Time	Area	Area Percent	Integration Codes
	2.712	14809	0.041	BD
	2.850	42540	0.117	BD
	3.883	15494	0.042	VD
	4.770	5579	0.015	BD
	7.163	4823	0.013	VV
	7.557	2345	0.006	VV
	8.000	123	0.000	VB
	8.583	4164	0.011	BV
	8.927	77	0.000	VB
	9.518	942	0.003	BB
	10.222	31022	0.085	BD
	11.992	1322	0.004	BB
	14.005	7406	0.020	BV
	14.897	365259	1.002	VD
	17.625	94	0.000	VB
	19.443	35933002	98.537	BD

**Normal Phase** (Method *i*-PrOH:CH<sub>2</sub>Cl<sub>2</sub>:Hx:AcOH 5:35:60:0.5, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-i-211np-3.dat

Acquired: 5/12/2006 9:38:02 PM

03:09:05 AM

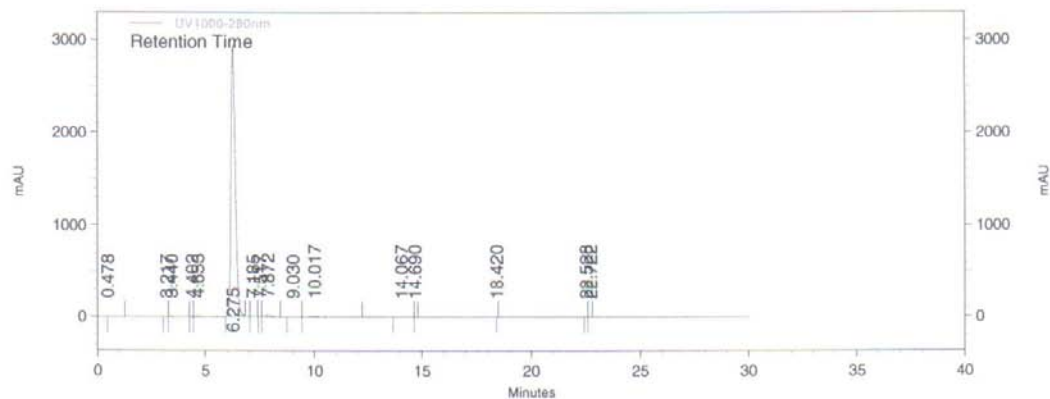
Printed: 05/22/2006

Analyst: System

Sample ID: dwk-i-211

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/12/2006  
 10:08:09 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	0.478	226	0.001	BD
	3.217	8070	0.020	BD
	3.440	103539	0.254	BD
	4.402	18350	0.045	VV
	4.653	228953	0.562	VD
	6.275	40043971	98.232	MM
	7.185	10776	0.026	BB
	7.517	1230	0.003	BV
	7.872	234001	0.574	VB
	9.030	11088	0.027	BV
	10.017	94591	0.232	VD
	14.067	9461	0.023	BV
	14.690	161	0.000	VB
	18.420	52	0.000	BD

**(E)-4-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]but-3-enoic acid (22).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 15:85:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-41rp-p-1.dat

Acquired: 5/4/2006 12:58:52 PM

Printed: 05/04/2006

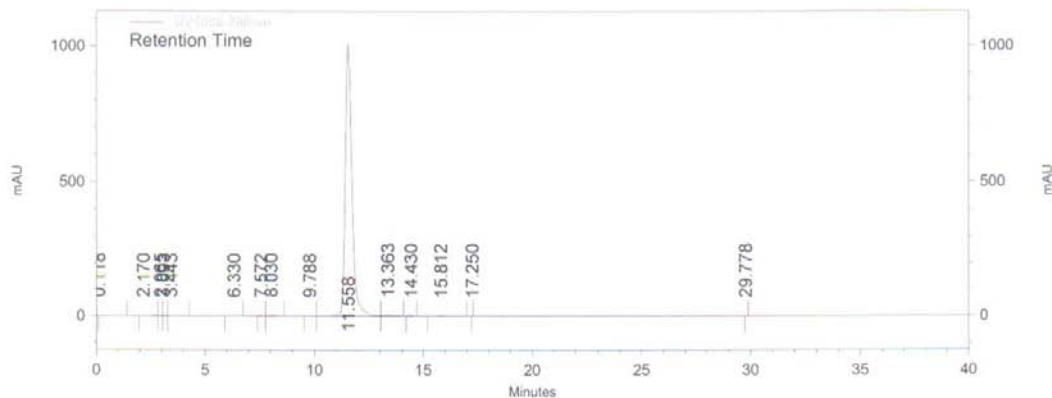
01:45:07 PM

Analyst: System

Sample ID: dwk-ii-41

Vial: N/A

Injection Volume: 0



UV1000-280nm

Results

(Acquisition in progress)

Name	Retention Time	Area	Area Percent	Integration Codes
	0.118	2338	0.011	BD
	2.170	25613	0.119	BD
	2.965	5025	0.023	BV
	3.093	5250	0.024	VV
	3.443	9471	0.044	VD
	6.330	3791	0.018	BD
	7.572	1433	0.007	BV
	8.030	15827	0.074	VB
	9.788	2289	0.011	BV
	11.558	21351224	99.360	VD
	13.363	56321	0.262	VB
	14.430	2264	0.011	BB
	15.812	7842	0.036	BD
	17.250	36	0.000	BB
	29.778	63	0.000	BD

**Normal Phase** (Method *i*-PrOH:CH<sub>2</sub>Cl<sub>2</sub>:Hx:AcOH 5:35:60:0.5, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

Data File: D:\DWKIM\dwk-ii-41-np-3.dat

Acquired: 5/15/2006 12:09:11 PM

Printed: 05/22/2006

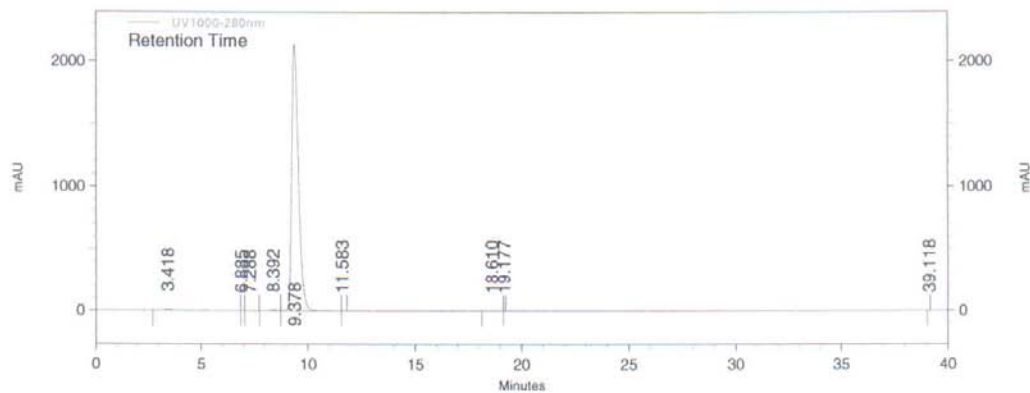
03:11:50 AM

Analyst: System

Sample ID: dwk-ii-41

Vial: N/A

Injection Volume: 0



**UV1000-280nm**  
**Results (System**  
**(5/15/2006**  
**12:49:40 PM)**  
**(Reprocessed)**  
**(Aborted Run))**

Name	Retention Time	Area	Area Percent	Integration Codes
	3.418	249790	0.539	BD
	6.885	1843	0.004	VV
	7.288	5679	0.012	VB
	8.392	126201	0.272	BV
	9.378	45970496	99.154	VD
	11.583	401	0.001	VB
	18.610	8262	0.018	BD
	19.177	64	0.000	VB
	39.118	71	0.000	BD

Totals		46362807	100.000	
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**(E)-5-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]pent-4-enoic acid (23).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-i-265rp-1.dat

Acquired: 5/3/2006 11:55:50 PM

Printed: 05/04/2006

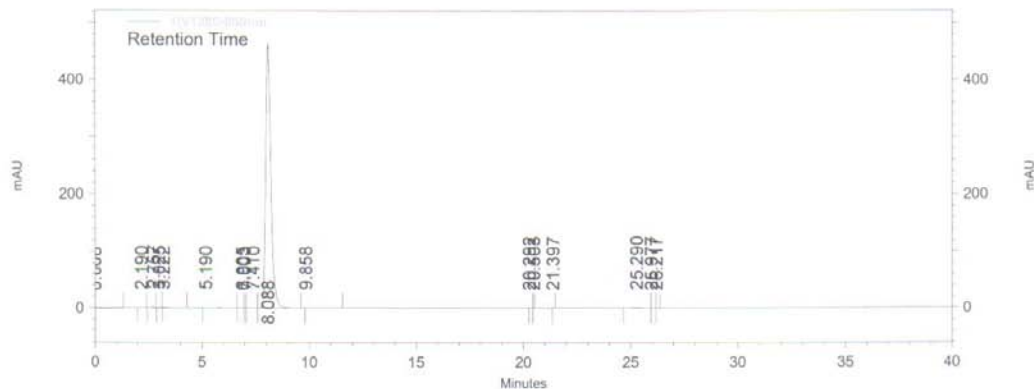
12:36:02 AM

Analyst: System

Sample ID: dwk-i-265

Vial: N/A

Injection Volume: 0



**UV1000-280nm**

**Results**

(Acquisition in progress)

Name	Retention Time	Area	Area Percent	Integration Codes
	0.008	71034	0.897	BD
	2.190	1847	0.023	BD
	2.757	14166	0.179	BB
	3.025	11370	0.144	BV
	3.222	19221	0.243	VD
	5.190	11619	0.147	BD
	6.905	591	0.007	BV
	7.003	154	0.002	VV
	7.410	2653	0.034	VV
	8.088	7747487	97.841	VB
	9.858	3194	0.040	BD
	20.292	93	0.001	BD
	20.503	30	0.000	BB
	21.397	82	0.001	BB
	25.290	34261	0.433	BV
	25.977	466	0.006	VV
	26.217	189	0.002	VB

**Normal Phase** (Method *i*-PrOH:CH<sub>2</sub>Cl<sub>2</sub>:Hx:AcOH 3:27:70:0.5, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

Data File: D:\DWKIM\dwk-i-265-np-6.dat  
 Acquired: 5/15/2006 3:32:48 PM  
 03:13:03 AM

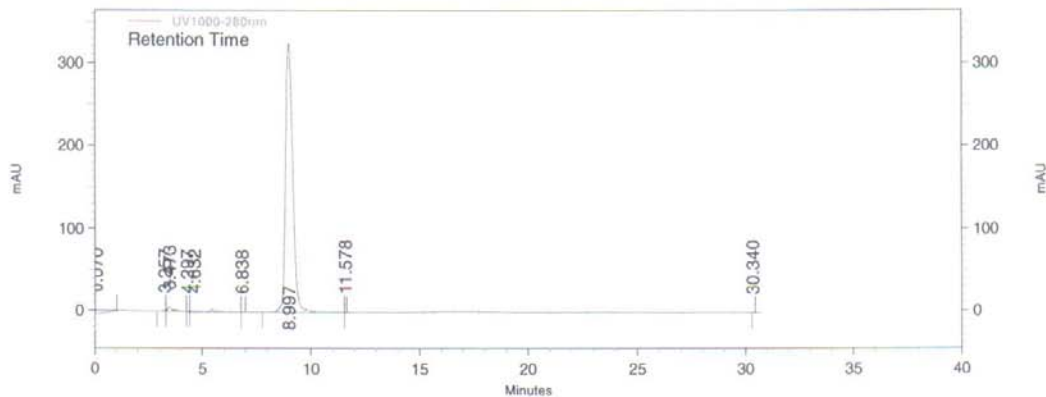
Printed: 05/22/2006

Analyst: System

Sample ID: dwk-i-265

Vial: N/A

Injection Volume: 0



**UV1000-280nm**  
**Results (System**  
**(5/15/2006**  
**4:03:40 PM)**  
**(Reprocessed)**  
**(Aborted Run)**

Name	Retention Time	Area	Area Percent	Integration Codes
	0.070	147301	1.887	MM
	3.257	10024	0.128	BV
	3.473	90064	1.154	VD
	4.297	2108	0.027	VV
	4.632	46016	0.589	VD
	6.838	143	0.002	VB
	8.997	7510726	96.210	BD
	11.578	78	0.001	VB
	30.340	106	0.001	BD

Totals		7806566	100.000	
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**(E)-6-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hex-5-enoic acid (24).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-87rp-1.dat

Acquired: 5/1/2006 11:17:35 AM

Printed: 05/01/2006

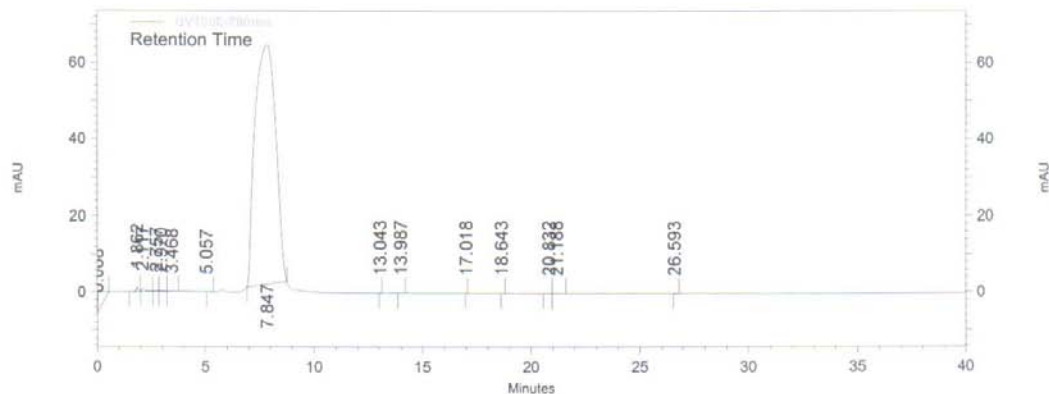
01:13:38 PM

Analyst: System

Sample ID: dwk-ii-87

Vial: N/A

Injection Volume: 0



UV1000-280nm  
Results (System  
(5/1/2006  
1:13:14 PM)  
(Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	0.008	82148	1.941	MM
	1.862	8225	0.194	BV
	2.117	11056	0.261	VD
	2.757	3512	0.083	VV
	2.970	2888	0.068	VV
	3.468	737	0.017	VD
	5.057	307	0.007	MM
	7.847	4122572	97.404	MM
	13.043	80	0.002	BB
	13.987	413	0.010	BB
	17.018	51	0.001	BD
	18.643	132	0.003	BB
	20.832	71	0.002	MM
	21.188	56	0.001	MM
	26.593	195	0.005	BB

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

Data File: D:\DWKIM\dwk-ii-87np-4.dat

Acquired: 5/11/2006 10:45:40 AM

Printed: 05/11/2006

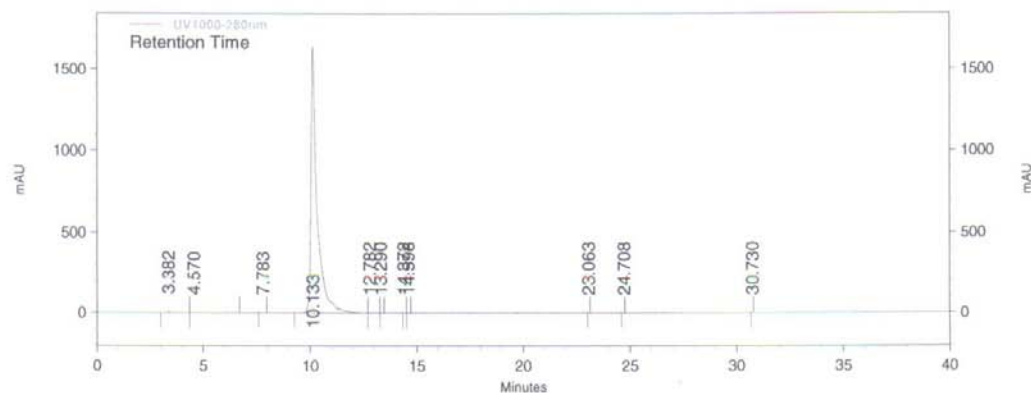
11:57:24 AM

Analyst: System

Sample ID: dwk-ii-87

Vial: N/A

Injection Volume: 0



UV1000-280nm  
Results (System  
5/11/2006  
11:57:16 AM)  
(Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.382	53641	0.162	BD
	4.570	19388	0.058	VD
	7.783	615	0.002	BB
	10.133	33081151	99.746	BD
	12.782	9968	0.030	VV
	13.290	235	0.001	VB
	14.372	124	0.000	BV
	14.598	131	0.000	VB
	23.063	54	0.000	BD
	24.708	106	0.000	BB
	30.730	69	0.000	BB

Totals		33165482	100.000	
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**(E)-7-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenyl]hept-6-enoic acid (25).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

Data File: D:\DWKIM\dwk-ii-85-4.dat

Acquired: 4/20/2006 5:01:57 PM

Printed: 04/20/2006

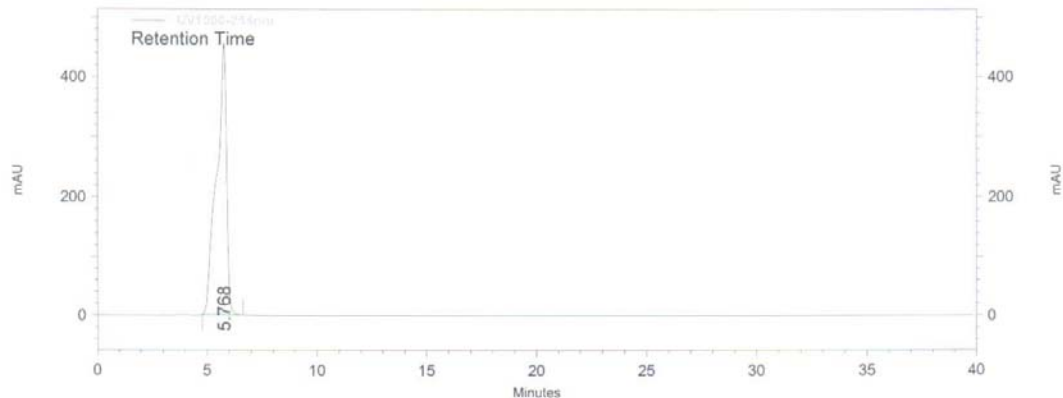
05:42:18 PM

Analyst: System

Sample ID: dwk-ii-85

Vial: N/A

Injection Volume: 0



UV1000-254nm

Results (System

(4/20/2006

5:42:09 PM)

(Reprocessed)

(Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	5.768	14330803	100.000	BB
<b>Totals</b>				
		14330803	100.000	

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

Data File: D:\DWKIM\dwk-ii-85np-2.dat

Acquired: 5/10/2006 10:15:58 PM

10:46:50 PM

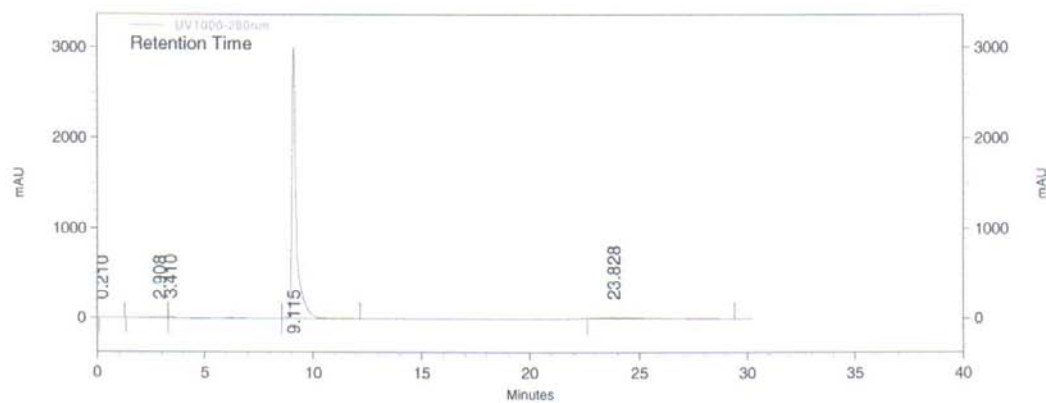
Printed: 05/10/2006

Analyst: System

Sample ID: dwk-ii-85

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/10/2006  
 10:46:13 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	0.210	7470	0.016	BD
	2.908	429139	0.946	BD
	3.410	547726	1.207	VD
	9.115	43415673	95.663	BD
	23.828	984085	2.168	BD

Totals		45384093	100.000	
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**Methyl hydrogen 4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]styrylphosphonate (26).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-i-259rp.dat

Acquired: 5/3/2006 2:45:50 PM

Printed: 05/22/2006 03:15:12

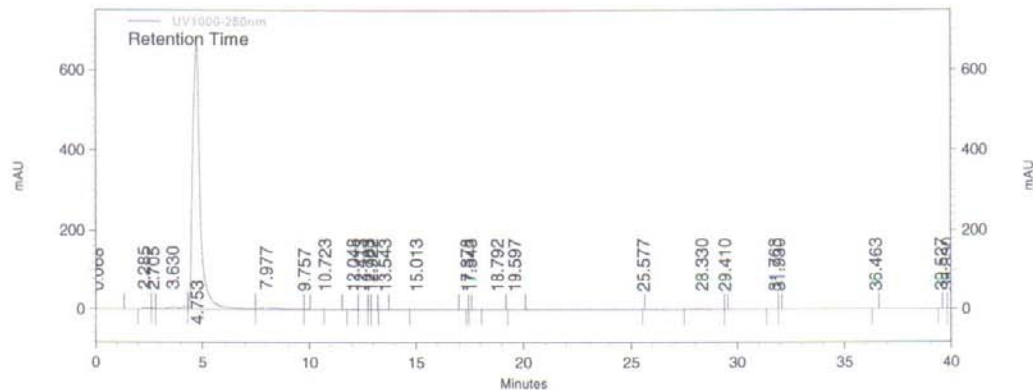
AM

Analyst: System

Sample ID: dwk-i-259

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/3/2006  
 3:33:11 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	0.068	1085	0.006	BD
	2.285	86461	0.516	BD
	2.705	29120	0.174	VV
	3.630	300808	1.795	VD
	4.753	15986247	95.378	VD
	7.977	262738	1.568	VV
	9.757	1274	0.008	VB
	10.723	50	0.000	BD
	12.048	5494	0.033	BV
	12.413	3286	0.020	VV
	12.783	89	0.001	VB
	12.922	254	0.002	BB
	13.543	566	0.003	BB
	15.013	12268	0.073	BD

**Normal Phase** (Method EtOAc:Hx:AcOH 20:79:1.0, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

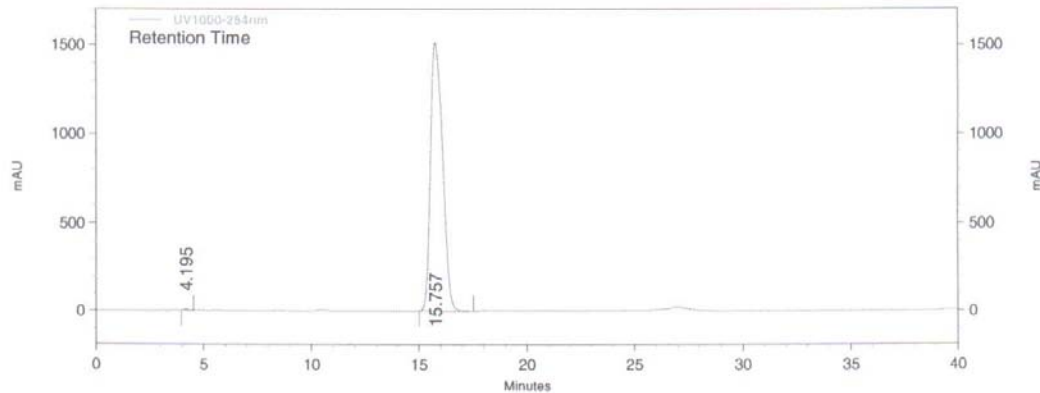
Data File: D:\DWKIM\dwk-i-259-np.dat  
 Acquired: 4/19/2006 11:35:37 AM  
 03:16:37 AM

Printed: 05/22/2006

Analyst: System  
 Sample ID: test-13

Vial: N/A

Injection Volume: 0



UV1000-254nm  
 Results (System  
 (4/19/2006  
 1:03:36 PM)  
 (Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	4.195	146713	0.246	BB
	15.757	59566058	99.754	BB
<b>Totals</b>				
		59712771	100.000	



**Ethyl 6-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoate (29).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 5:95:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

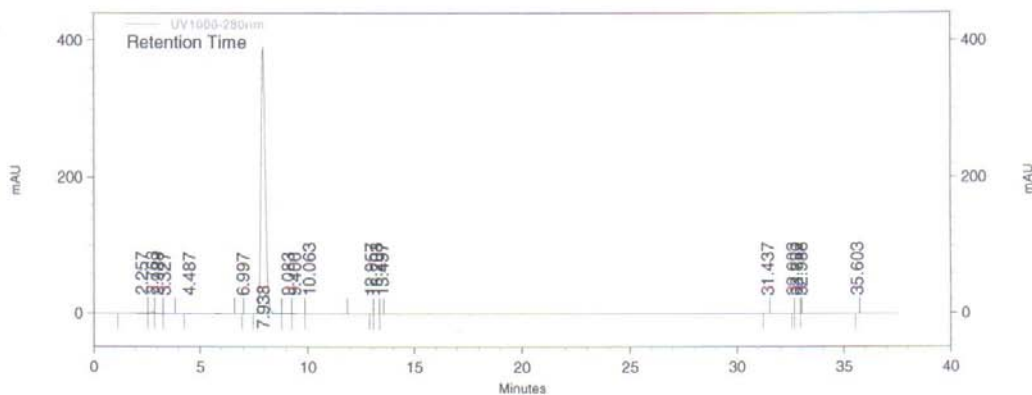
Data File: D:\DWKIM\dwk-ii-35p-1.dat  
 Acquired: 5/4/2006 9:16:14 PM  
 AM

Printed: 05/22/2006 03:06:33

Analyst: System  
 Sample ID: dwk-ii-35

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/4/2006  
 9:53:51 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	2.257	35276	0.632	BD
	2.763	18563	0.333	VV
	2.988	7404	0.133	VV
	3.327	2279	0.041	VD
	4.487	18228	0.327	BD
	6.997	40	0.001	BB
	7.938	5478263	98.127	BV
	9.083	10999	0.197	VV
	9.400	7870	0.141	VB
	10.063	2598	0.047	BD
	12.957	115	0.002	BB
	13.203	266	0.005	BV
	13.497	121	0.002	VB
	31.437	242	0.004	BD

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-35-np.dat

Acquired: 5/8/2006 3:09:04 PM

PM

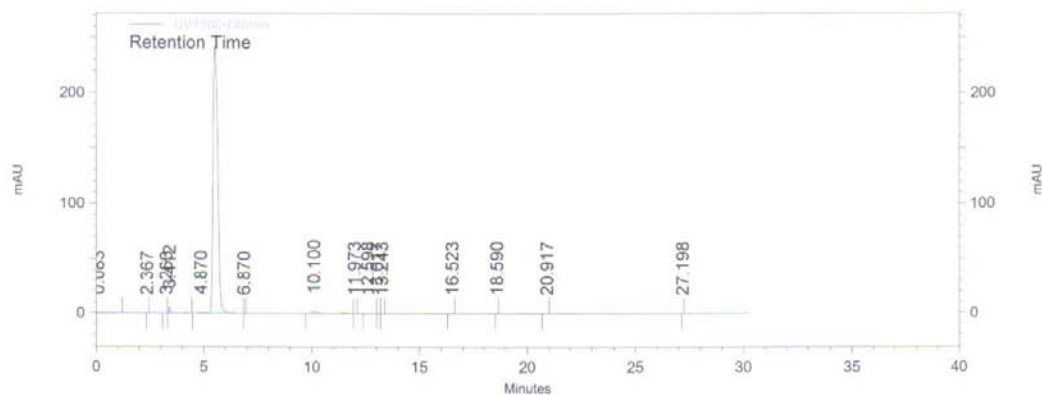
Printed: 05/08/2006 03:39:31

Analyst: System

Sample ID: dwk-ii-35

Vial: N/A

Injection Volume: 0



UV1000-280nm

Results (System

5/8/2006

3:39:25 PM)

(Reprocessed)

(Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	0.083	21229	0.566	BD
	2.367	43	0.001	BD
	3.263	873	0.023	BB
	3.412	24969	0.666	BD
	4.870	3659222	97.642	BD
	6.870	69	0.002	VB
	10.100	37739	1.007	BD
	11.973	281	0.007	VB
	12.598	2237	0.060	BV
	13.017	214	0.006	VV
	13.243	107	0.003	VB
	16.523	146	0.004	BD
	18.590	126	0.003	BB
	20.917	277	0.007	BD

**4-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]butanoic acid (32).****Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-253-rp.dat

Acquired: 4/28/2006 11:53:14 AM

12:54:19 PM

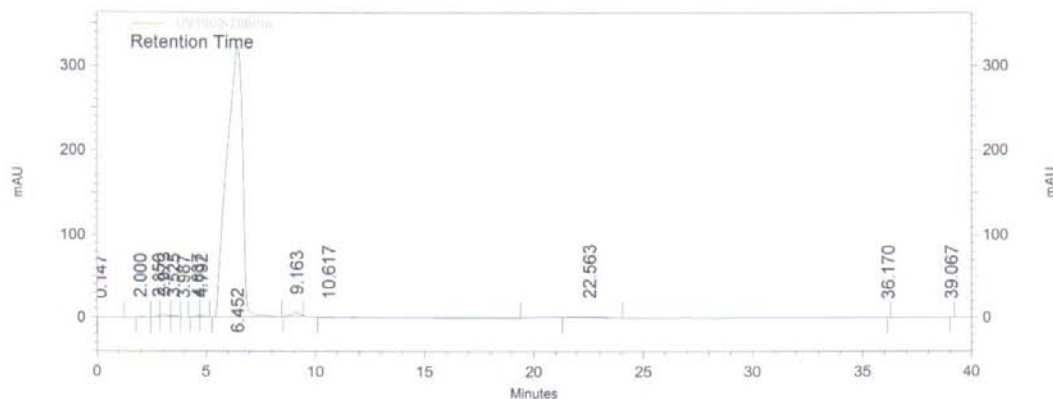
Printed: 04/28/2006

Analyst: System

Sample ID: dwk-ii-253

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (4/28/2006  
 12:53:59 PM)  
 (Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	0.147	1931	0.012	BD
	2.000	8807	0.053	BD
	2.850	14500	0.088	VV
	3.073	63232	0.383	VV
	3.525	23963	0.145	VV
	3.987	1507	0.009	VD
	4.637	17483	0.106	BV
	4.792	17852	0.108	VB
	6.452	16098807	97.631	BB
	9.163	124309	0.754	MM
	10.617	48974	0.297	BD
	22.563	67917	0.412	BD
	36.170	93	0.001	BB
	39.067	137	0.001	BB

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

### Area Percent Report

Page 1 of 2

Data File: D:\DWKIM\dwk-i-253-np-1.dat  
 Acquired: 5/8/2006 2:25:43 PM  
 PM

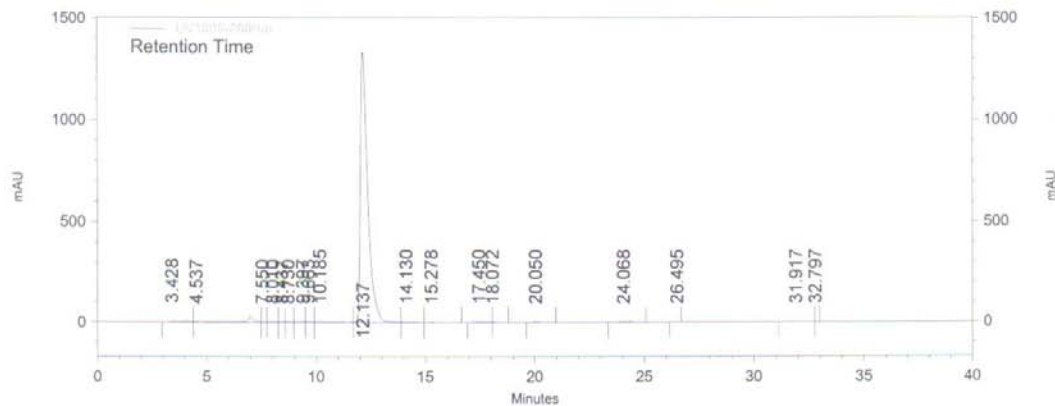
Printed: 05/08/2006 03:07:40

Analyst: System

Sample ID: dwk-i-253

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/8/2006  
 3:07:34 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.428	151528	0.473	BD
	4.537	571285	1.783	VD
	7.550	9371	0.029	VV
	8.010	22740	0.071	VV
	8.437	16431	0.051	VV
	8.730	16788	0.052	VV
	9.397	35304	0.110	VV
	9.663	39544	0.123	VV
	10.185	81140	0.253	VD
	12.137	30876296	96.341	VV
	14.130	52282	0.163	VV
	15.278	18353	0.057	VD
	17.450	71041	0.222	BV
	18.072	7209	0.022	VB

**5-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]pentanoic acid (33).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-33-rp-1.dat

Acquired: 4/28/2006 11:08:11 AM

Printed: 04/28/2006

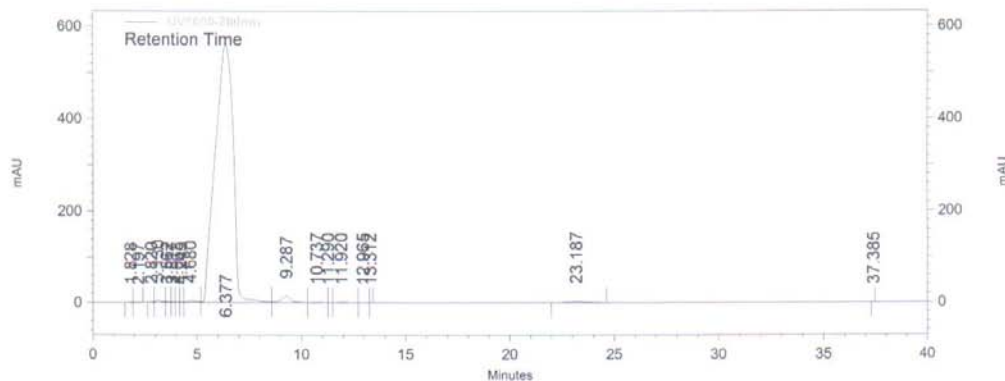
11:51:34 AM

Analyst: System

Sample ID: dwk-ii-33

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (4/28/2006  
 11:51:30 AM)  
 (Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	1.828	3276	0.010	BV
	2.197	2918	0.008	VB
	2.820	15017	0.044	BV
	3.130	94719	0.275	VV
	3.562	26909	0.078	VV
	3.817	13092	0.038	VV
	4.063	12137	0.035	VV
	4.245	11248	0.033	VV
	4.680	121223	0.352	VV
	6.377	33409500	97.101	VV
	9.287	474127	1.378	VV
	10.737	32151	0.093	VV
	11.290	2938	0.009	VV
	11.920	27697	0.080	VV
	12.965	2903	0.008	VV
	13.312	154	0.000	VB

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-33-np-1.dat

Acquired: 5/9/2006 9:14:21 AM

Printed: 05/09/2006 09:57:29

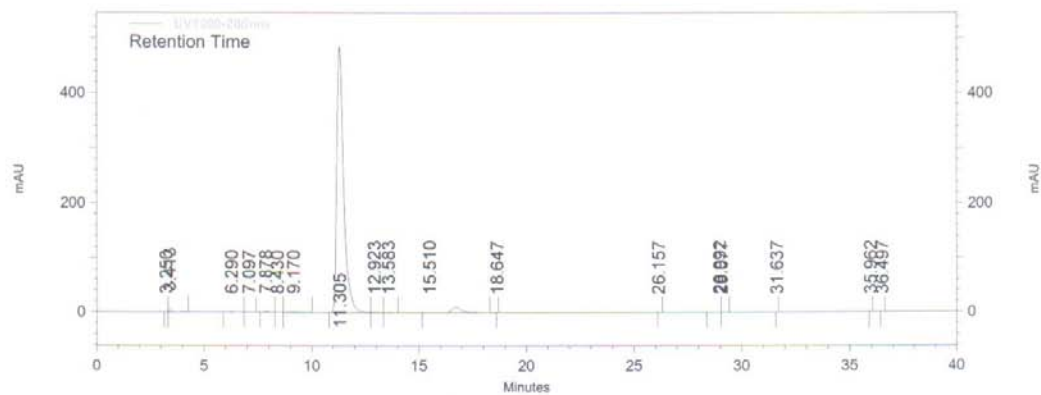
AM

Analyst: System

Sample ID: dwk-ii-33

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 5/9/2006  
 9:57:21 AM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.250	403	0.004	BD
	3.413	14232	0.128	BD
	6.290	11496	0.104	BD
	7.097	4347	0.039	VB
	7.878	17499	0.158	BV
	8.430	1498	0.013	VV
	9.170	20287	0.183	VB
	11.305	10719772	96.581	BD
	12.923	8363	0.075	VV
	13.583	4495	0.040	VB
	15.510	290777	2.620	BD
	18.647	57	0.001	BB
	26.157	137	0.001	BD
	28.992	3763	0.034	BV

**6-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoic acid (34).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-43-rp-2.dat  
 Acquired: 4/27/2006 9:41:28 PM  
 09:01:57 AM

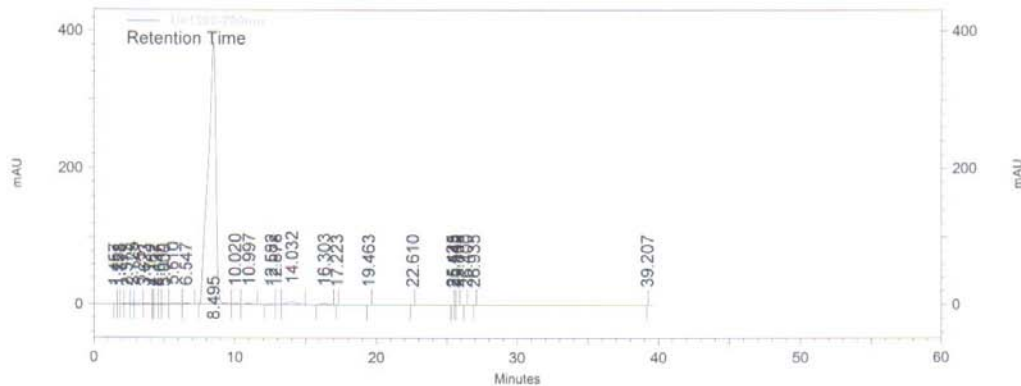
Printed: 04/28/2006

Analyst: System

Sample ID: dwk-ii-43

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (4/27/2006  
 10:22:08 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	1.457	916	0.006	BV
	1.758	3012	0.020	VV
	1.873	5984	0.039	VV
	2.373	15829	0.103	VV
	2.725	20371	0.133	VB
	3.257	62192	0.406	BV
	3.623	32049	0.209	VV
	4.162	4089	0.027	VV
	4.437	13057	0.085	VV
	4.645	7322	0.048	VV
	5.000	14942	0.098	VV
	5.610	53034	0.346	VV
	6.547	33876	0.221	VB
	8.495	14754225	96.357	BV

**Normal Phase** (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-43-np.dat  
 Acquired: 5/9/2006 9:59:05 AM  
 AM

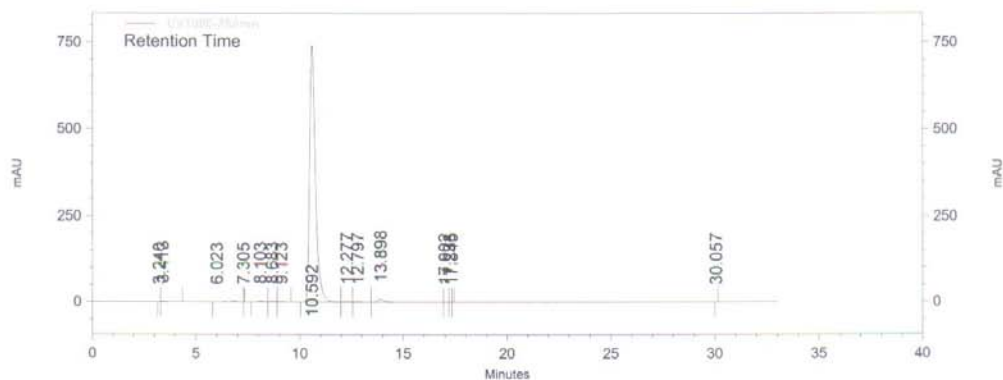
Printed: 05/09/2006 10:32:15

Analyst: System

Sample ID: dwk-ii-43

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 5/9/2006  
 10:32:10 AM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.240	248	0.002	BD
	3.413	12522	0.084	BD
	6.023	67940	0.453	BD
	7.305	56	0.000	VB
	8.103	34177	0.228	BV
	8.683	27731	0.185	VV
	9.123	24141	0.161	VB
	10.592	14570342	97.201	BD
	12.277	31013	0.207	VV
	12.797	34176	0.228	VV
	13.898	186078	1.241	VD
	17.002	1058	0.007	VV
	17.233	313	0.002	VV
	17.345	126	0.001	VB



## 7-[4-[Bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]heptanoic acid (35).

Reverse Phase (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

## Area Percent Report

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-63-rp.dat

Acquired: 4/28/2006 9:52:21 AM

Printed: 04/28/2006

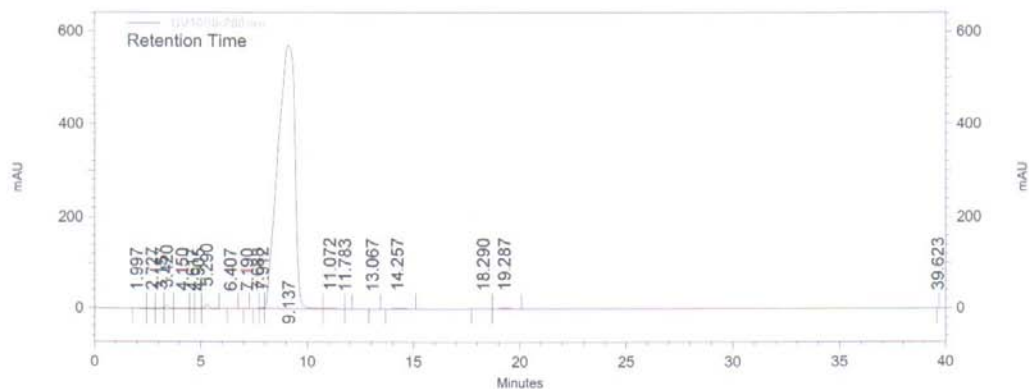
10:41:28 AM

Analyst: System

Sample ID: dwk-ii-63

Vial: N/A

Injection Volume: 0



UV1000-280nm

Results (System

(4/28/2006

10:41:23 AM)

(Reprocessed)

(Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	1.997	13125	0.041	BV
	2.727	26108	0.081	VB
	3.157	32949	0.102	BV
	3.420	87442	0.271	VV
	4.150	35715	0.111	VV
	4.617	6725	0.021	VV
	4.905	8456	0.026	VV
	5.290	124958	0.387	VB
	6.407	1183	0.004	BB
	7.190	329	0.001	BB
	7.688	6375	0.020	BV
	7.912	28579	0.088	VV
	9.137	31790074	98.370	VV
	11.072	47221	0.146	VV

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

## Area Percent Report

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-63-np-2.dat

Acquired: 5/8/2006 12:32:26 PM

Printed: 05/08/2006

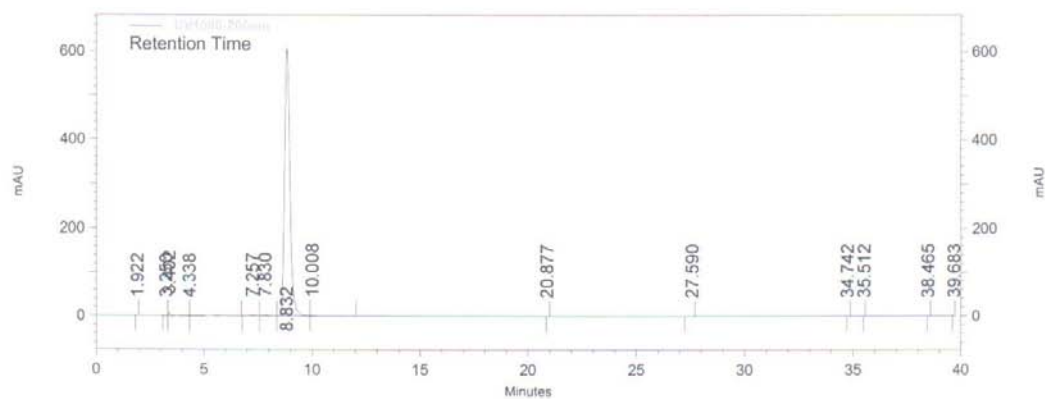
01:12:22 PM

Analyst: System

Sample ID: dwk-ii-63

Vial: N/A

Injection Volume: 0



UV1000-280nm  
Results (System  
(5/8/2006  
1:12:15 PM)  
(Reprocessed)  
(Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	1.922	115	0.001	BD
	3.250	1485	0.014	BD
	3.402	17593	0.166	BD
	4.338	23444	0.221	VD
	7.257	32096	0.303	BV
	7.830	37014	0.349	VV
	8.832	10462241	98.649	VV
	10.008	30691	0.289	VD
	20.877	110	0.001	BD
	27.590	428	0.004	BB
	34.742	98	0.001	BB
	35.512	56	0.001	BB
	38.465	104	0.001	BB
	39.683	56	0.001	BE

**11-[4-[bicyclo[3.3.1]non-9-ylidene(4-hydroxyphenyl)methyl]phenoxy]nudecanoic acid (36).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 5:95:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-73rp-6.dat  
 Acquired: 5/4/2006 2:58:12 PM  
 PM

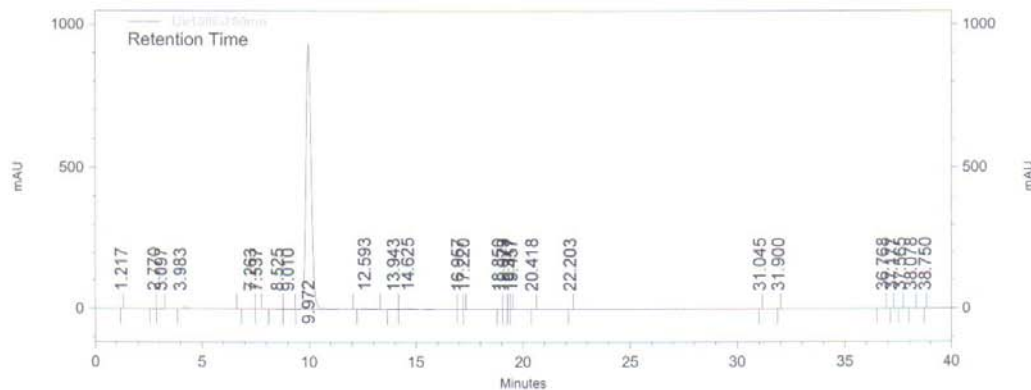
Printed: 05/04/2006 03:43:39

Analyst: System

Sample ID: dwk-ii-73

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System)  
 (5/4/2006  
 3:43:33 PM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	1.217	73	0.000	BD
	2.770	8868	0.055	BD
	3.097	5335	0.033	BB
	3.983	108462	0.669	BD
	7.263	4369	0.027	BV
	7.537	965	0.006	VB
	8.525	2980	0.018	BV
	9.010	2941	0.018	VB
	9.972	15924817	98.252	BD
	12.593	62412	0.385	BB
	13.943	3225	0.020	BV
	14.625	80567	0.497	VD
	16.957	1063	0.007	VV
	17.220	82	0.001	VB

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-73-np.dat  
 Acquired: 5/8/2006 3:40:33 PM  
 PM

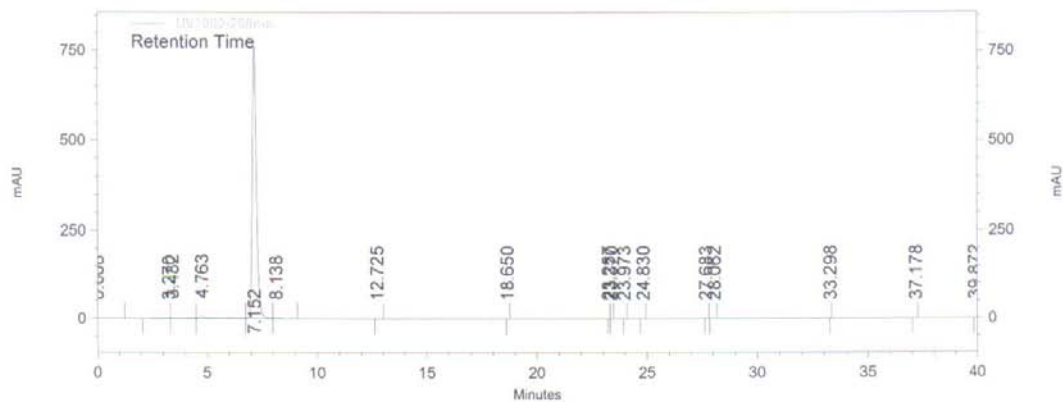
Printed: 05/08/2006 04:28:19

Analyst: System

Sample ID: dwk-ii-73

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System)  
 (5/8/2006  
 4:28:11 PM)  
 (Reprocessed)  
 (Aborted Run)

Name	Retention Time	Area	Area Percent	Integration Codes
	0.008	2866	0.028	BD
	3.270	14452	0.139	BD
	3.432	59622	0.574	VD
	4.763	219460	2.114	VD
	7.152	10048538	96.813	VV
	8.138	32687	0.315	VB
	12.725	505	0.005	BD
	18.650	95	0.001	BD
	23.257	46	0.000	BD
	23.370	65	0.001	VB
	23.973	123	0.001	BB
	24.830	207	0.002	BB
	27.683	108	0.001	BB
	28.062	221	0.002	BB

**(E)-3-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenyl]acrylic acid (42).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 20:80:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-i-207rp-1.dat

Acquired: 5/3/2006 11:36:05 AM

Printed: 05/22/2006

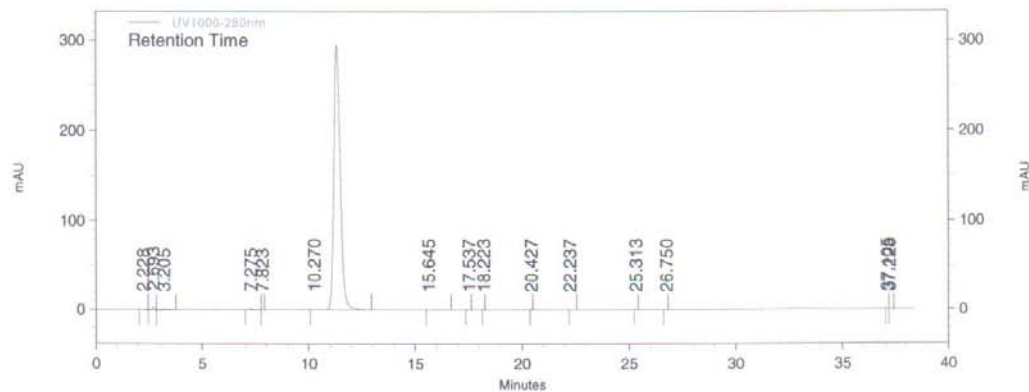
03:21:12 AM

Analyst: System

Sample ID: dwk-i-207

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 5/3/2006  
 12:14:47 PM)  
 (Reprocessed)  
 (Aborted Run)

Name	Retention Time	Area	Area Percent	Integration Codes
	2.228	8710	0.139	BD
	2.693	33609	0.538	VB
	3.205	32598	0.521	BD
	7.275	12333	0.197	BD
	7.823	128	0.002	VB
	10.270	6163427	98.572	BD
	15.645	668	0.011	BD
	17.537	308	0.005	BB
	18.223	97	0.002	BB
	20.427	60	0.001	BD
	22.237	217	0.003	BB
	25.313	136	0.002	BB
	26.750	182	0.003	BB
	37.105	120	0.002	BB

Normal Phase (Method *i*-PrOH:CH<sub>2</sub>Cl<sub>2</sub>:Hx:AcOH 5:35:60:0.5, Flow rate 1mL/min)

### Area Percent Report

Page 1 of 1

Data File: D:\DWKIM\dwk-i-209np.dat

Acquired: 5/13/2006 5:06:37 PM

Printed: 05/22/2006

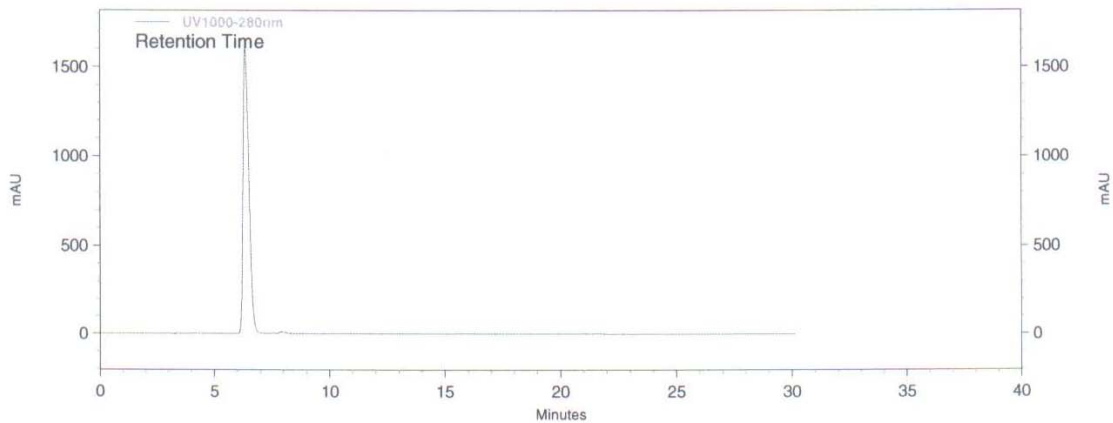
03:21:27 AM

Analyst: System

Sample ID: dwk-i-209

Vial: N/A

Injection Volume: 0



Area percent : >99%

**6-[4-[cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]hexanoic acid (43).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 10:90:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 1

Data File: D:\DWKIM\test-4.dat  
 Acquired: 4/17/2006 1:08:12 PM  
 03:23:13 AM

*DWK-ii-97-rp*

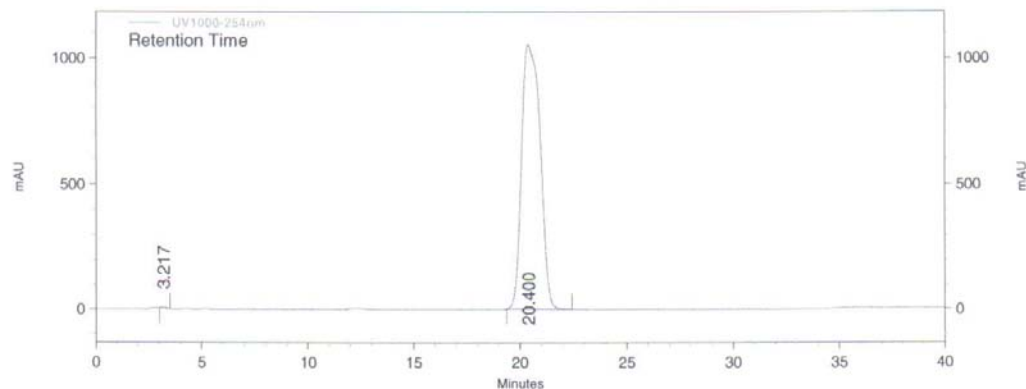
Printed: 05/22/2006

Analyst: System

Sample ID: test-4 *DWK-ii-97*

Vial: N/A

Injection Volume: 0



UV1000-254nm  
 Results (System  
 (4/17/2006  
 1:53:17 PM)  
 (Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.217	93796	0.148	BB
	20.400	63447438	99.852	BB
Totals		63541234	100.000	

Normal Phase (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

Area Percent Report

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-97-np.dat

Acquired: 5/9/2006 11:23:23 AM

Printed: 05/09/2006

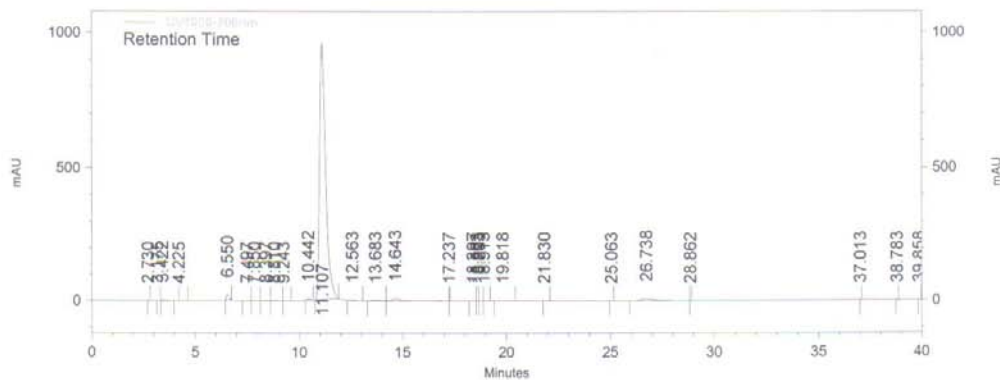
12:54:12 PM

Analyst: System

Sample ID: dwk-ii-97

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 (5/9/2006  
 12:53:19 PM)  
 (Reprocessed))

Name	Retention Time	Area	Area Percent	Integration Codes
	2.730	46	0.000	BD
	3.175	399	0.002	BB
	3.422	9429	0.046	BD
	4.225	4443	0.022	MM
	6.550	192373	0.939	MM
	7.497	8145	0.040	BV
	7.850	6541	0.032	VB
	8.397	18787	0.092	BV
	8.810	16942	0.083	VV
	9.243	3137	0.015	VB
	10.442	47954	0.234	MM
	11.107	19513304	95.293	MM
	12.563	33331	0.163	BB
	13.683	57531	0.281	BV
	14.643	223687	1.092	VD
	17.237	27	0.000	VB



**2-[4-[Cyclohexylidene(4-hydroxyphenyl)methyl]phenoxy]acetic acid (44).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 20:80:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-i-199rp.dat  
 Acquired: 5/3/2006 10:56:24 AM  
 03:18:34 AM

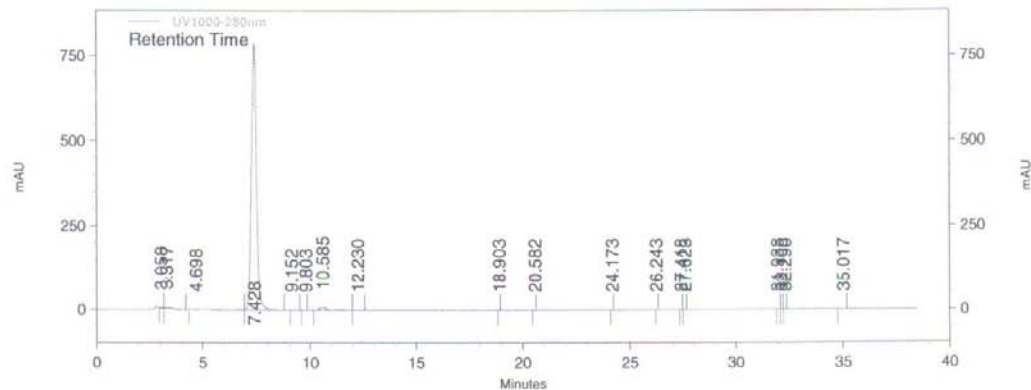
Printed: 05/22/2006

Analyst: System

Sample ID: dwk-i-199

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System)  
 (5/3/2006  
 11:34:57 AM)  
 (Reprocessed)  
 (Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.058	13849	0.107	BV
	3.317	15498	0.120	VD
	4.698	45724	0.354	BD
	7.428	12668276	98.061	VB
	9.152	939	0.007	BB
	9.803	245	0.002	BB
	10.585	168149	1.302	BD
	12.230	4789	0.037	BB
	18.903	75	0.001	BD
	20.582	107	0.001	BD
	24.173	69	0.001	BB
	26.243	59	0.000	BB
	27.418	79	0.001	BB
	27.623	123	0.001	BB

Normal Phase (Method *i*-PrOH:CH<sub>2</sub>Cl<sub>2</sub>:AcOH 10:90:0.5, Flow rate 1mL/min)

### Area Percent Report

Page 1 of 2

Data File: D:\DWKIM\dwk-i-199np.dat

Acquired: 5/12/2006 4:17:30 PM

Printed: 05/22/2006

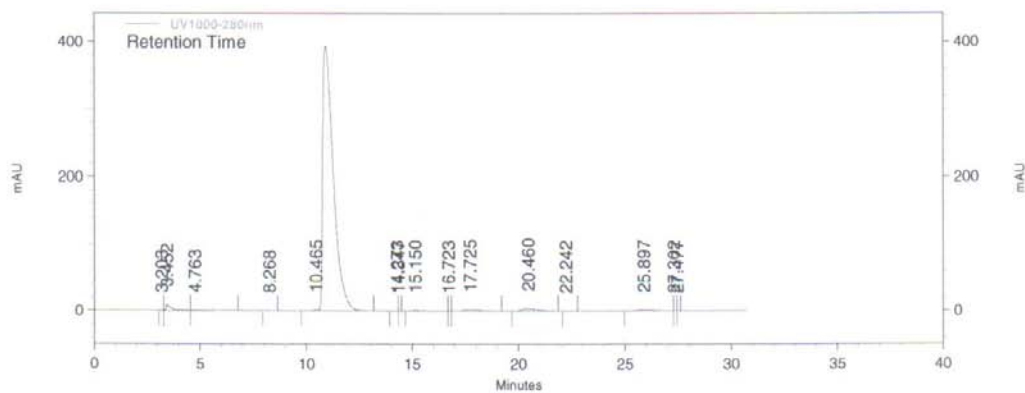
03:19:13 AM

Analyst: System

Sample ID: dwk-i-199

Vial: N/A

Injection Volume: 0



UV1000-280nm

Results (System

5/12/2006

4:48:25 PM)

(Reprocessed)

(Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.203	3545	0.025	BD
	3.452	182366	1.280	BD
	4.763	37876	0.266	VD
	8.268	3274	0.023	BB
	10.465	13652989	95.858	BD
	14.273	984	0.007	BV
	14.347	253	0.002	VB
	15.150	22620	0.159	BD
	16.723	62	0.000	BB
	17.725	98306	0.690	BB
	20.460	160063	1.124	BD
	22.242	2043	0.014	BB
	25.897	77998	0.548	BV
	27.302	482	0.003	VV

**6-[4-[bicyclo[3.3.1]non-9-ylidene(phenyl)methyl]phenoxy]hexanoic acid (46).**

**Reverse Phase** (Method H<sub>2</sub>O:MeOH:AcOH 5:95:0.1, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-93rp-6.dat  
 Acquired: 5/4/2006 3:45:00 PM  
 PM

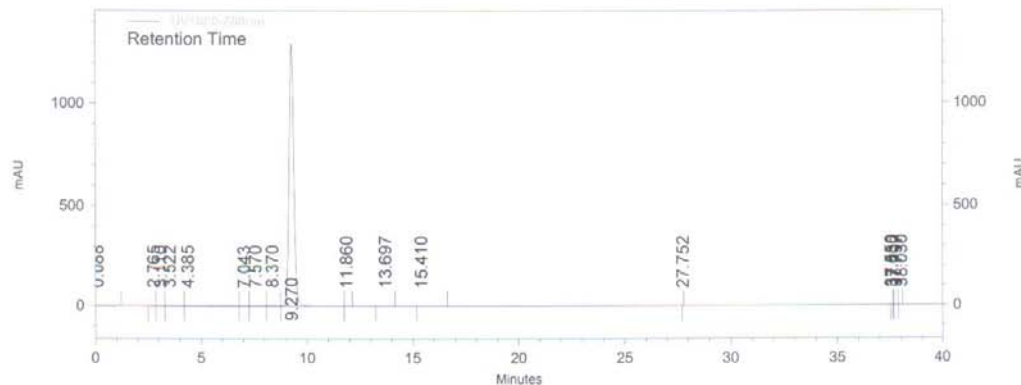
Printed: 05/04/2006 04:25:16

Analyst: System

Sample ID: dwk-ii-93

Vial: N/A

Injection Volume: 0



UV1000-280nm  
 Results (System  
 5/4/2006  
 4:25:10 PM)  
 (Reprocessed)  
 (Aborted Run)

Name	Retention Time	Area	Area Percent	Integration Codes
	0.088	27366	0.135	BD
	2.765	7896	0.039	BD
	3.110	10759	0.053	BV
	3.522	35802	0.176	VD
	4.385	70705	0.348	VD
	7.043	8268	0.041	VV
	7.570	23610	0.116	VV
	8.370	47902	0.236	VV
	9.270	20088064	98.760	VD
	11.860	2114	0.010	VB
	13.697	16472	0.081	BB
	15.410	869	0.004	BD
	27.752	61	0.000	BD
	37.550	83	0.000	BV

**Normal Phase** (Method EtOAc:Hx:AcOH 25:75:0.2, Flow rate 1mL/min)

**Area Percent Report**

Page 1 of 2

Data File: D:\DWKIM\dwk-ii-93-np.dat  
 Acquired: 5/9/2006 2:29:17 PM  
 AM

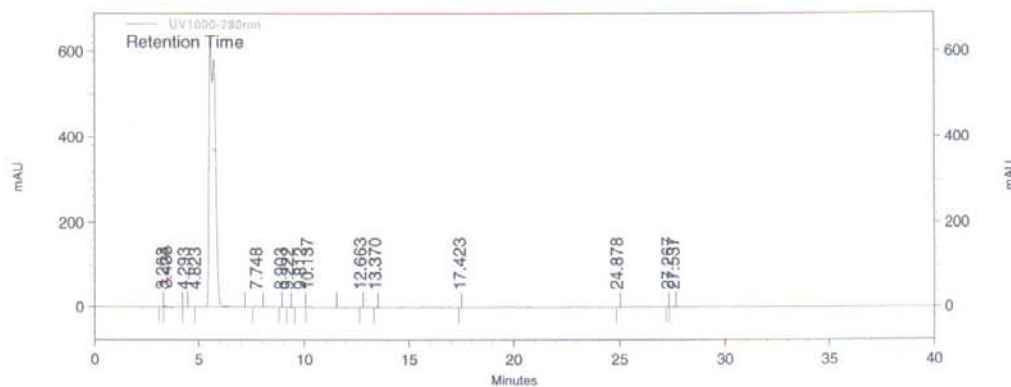
Printed: 05/22/2006 03:24:40

Analyst: System

Sample ID: dwk-ii-93

Vial: N/A

Injection Volume: 0



**UV1000-280nm**

**Results (System**

(5/22/2006

3:24:22 AM)

(Reprocessed)

(Aborted Run))

Name	Retention Time	Area	Area Percent	Integration Codes
	3.263	371	0.003	BD
	3.435	17457	0.139	BD
	4.293	826	0.007	VB
	4.823	12576776	99.789	BD
	7.748	1485	0.012	BB
	8.903	105	0.001	BB
	9.222	105	0.001	BB
	9.812	5262	0.042	BV
	10.137	294	0.002	VD
	12.663	131	0.001	BB
	13.370	115	0.001	BB
	17.423	68	0.001	BD
	24.878	114	0.001	BD
	27.267	66	0.001	BB