



Supporting Information

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# A Stereocontrolled, Solid Phase Synthesis of 90 Membered Library of Indoline Alkaloid-like Polycyclics from an *Enantiorich* Aminoindoline Scaffold

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## General Methods

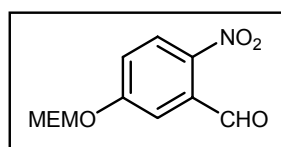
The materials were obtained from commercial suppliers and used without purification. THF, CH<sub>2</sub>Cl<sub>2</sub>, and DMF were passed through the activated alumina columns to remove impurities prior to use. 2,6-Lutidine was distilled from CaH<sub>2</sub> under N<sub>2</sub>. Column chromatography was performed using Silicycle Ultra Pure Silica Gel (230-400 mesh). Reactions were monitored by thin layer chromatography (TLC) using Merck 60 F<sub>254</sub> 0.25 mm silica gel plates.

Small-scale solid phase reactions (1-50 mg resin) were performed in 2 mL fritted polypropylene Bio-Spin® chromatography columns. Medium-scale solid phase reactions (50-500 mg) were performed in 10 mL polypropylene PD-10 columns. Agitation of solid phase reactions was performed using a Barnstead-Thermolyne™ Labquake shaker. Library synthesis was achieved by use of IRORI Technology (Accutag-100 Combinatorial Chemistry System, MicroKan Reactor Pk96, Radio frequency tag Pk500). The washing was carried out on a Vac-Man®laboratory Vacuum Manifold with 2-way Teflon stopcocks. The linker cleavage reactions (<50 mg of beads) were carried out in 1.5 mL eppendorf tubes. Vacuum removal of solvents for the linker cleavage reactions was accomplished using Genevac HT-4 Atlas Evaporator.

All NMR experiments were recorded on an AC-Bruker instrument (400 MHz). Unless otherwise noted, proton and carbon chemical shifts are reported in parts per million using residual CHCl<sub>3</sub> as an internal standard at 7.26 and 77.0 ppm, respectively. Analysis by mass spectrometry was performed on a VG Quattro I (Micromass) mass spectrometer equipped with a pneumatically assisted electrospray ionization source operating in positive mode. The enantiomeric excess (ee%) was determined by chiral HPLC using a Hewlett-Packard (Agilent) 1090 LC equipped with a diode array detector and Chiracel-OD column. The HPLC spectra were recorded on an Agilent 1100 Series HPLC system.

## Experimental Section

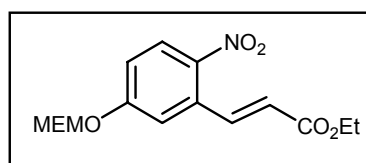
### Compound 9a



DIPEA (35.6 mL, 204 mmol) and MEM chloride (17.5 mL, 153 mmol) were added to a solution of 5-hydroxy-2-nitro-benzaldehyde (**9**, 13.9 g, 83.2 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (500 mL) at 0 °C. The mixture was warmed to room temperature and stirred for another 4 h. The reaction was quenched with saturated ammonium chloride solution and extracted with

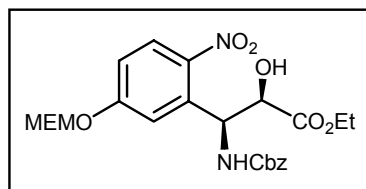
CH<sub>2</sub>Cl<sub>2</sub>. The organic layer was washed with brine, dried over Na<sub>2</sub>SO<sub>4</sub>, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:3 ethyl acetate/hexanes) to give the pure product **9a** (20.5 g, 97%) as pale yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  10.46 (s, 1H), 8.16 (d, *J* = 9.0 Hz, 1H), 7.48 (s, 1H), 7.34 (d, *J* = 8.9 Hz, 1H), 5.40 (s, 2H), 3.84 (d, *J* = 3.0 Hz, 2H), 3.56 (d, *J* = 2.9 Hz, 2H), 3.37 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  188.7, 162.1, 143.3, 134.7, 127.5, 120.2, 116.7, 93.9, 71.8, 68.9, 59.4; MS (ES<sup>+</sup>) *m/z* 256.0 (M+1).

### Compound 10



To a suspension of 60% NaH (3.0 g, 65 mmol) in THF (200mL) was added triethyl phosphono acetate (11.5 mL, 58 mmol) at 0 °C. The reaction mixture was stirred for 30 min at 0 °C. To the reaction mixture was added the above aldehyde **9a** (12.76 g, 50 mmol) at 0 °C. The reaction mixture was stirred for 6 h at 0 °C and quenched with water (75 mL). The mixture was extracted with EtOAc twice. The combined organic layers were washed with water and brine, dried over Na<sub>2</sub>SO<sub>4</sub>. The solvent was evaporated under reduced pressure and the residue was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the compound **10** (15.35 g, 94%) as yellow oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.18 (d, *J* = 15.7 Hz, 1H), 8.11 (d, *J* = 9.0 Hz, 1H), 7.21 (d, *J* = 2.6 Hz, 1H), 7.16 (dd, *J* = 9.0, 2.6 Hz, 1H), 6.32 (d, *J* = 15.7 Hz, 1H), 5.37 (s, 2H), 4.30 (q, *J* = 7.1 Hz, 2H), 3.84 (t, *J* = 4.6 Hz, 2H), 3.38 (t, *J* = 4.6 Hz, 2H), 3.38 (s, 3H), 1.36 (t, *J* = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  165.5, 161.5, 142.3, 141.1, 133.9, 127.8, 123.7, 117.4, 116.5, 93.8, 71.8, 68.6, 61.2, 59.4, 14.6; MS (ES<sup>+</sup>) *m/z* 326.1 (M+1).

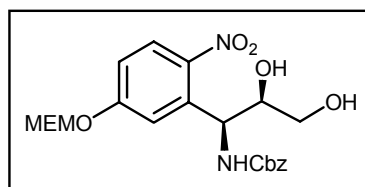
### Compound 11



Benzyl carbamate (3.63 g, 24 mmol) was dissolved in *n*-propyl alcohol (32 mL). To this stirred solution at 0 °C was added a freshly prepared solution of NaOH (NaOH (0.96 g, 24 mmol) in water (60 mL)), followed by a freshly prepared solution of *tert*-butyl hypochlorite (2.60 g, 24 mmol, ca. 2.8 mL). Then a solution of the ligand (DHQ)<sub>2</sub>PHAL (312 mg, 0.4 mmol, 5 mol%) in *n*-propyl alcohol (28 mL) was added. The solution was stirred in an ice bath for a few minutes. Then the olefin **10** (2.60 g, 8 mmol) was added, followed by potassium osmate dihydrate (118 mg, 0.32 mmol, 4 mmol%). The reaction was stirred for 2 h, and the dark green color of the solution have way to dark yellow at the end. After TLC analysis confirmed the absence of starting material, ethyl acetate (60 mL) was added and the phases were separated. The aqueous phase was extracted with ethyl acetate twice. The combined organic extracts were washed with water and brine, dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated to dryness to afford the crude product. Purification by column chromatography (1:1 ethyl acetate/hexanes) afforded the product **11** (2.93 g, 79%) as yellow syrup. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  8.13 (d, *J* = 9.1 Hz, 1H), 7.38-7.30 (m, 5H), 7.19 (d, *J* = 2.4 Hz, 1H), 7.11 (dd, *J* = 9.1, 2.4 Hz, 1H), 6.09 (d, *J* = 8.9 Hz, 1H), 5.88 (d, *J* = 8.9 Hz, 1H), 5.33 (s, 2H), 5.11 (d, *J* = 12.1 Hz, 1H), 5.03 (d, *J* = 11.9 Hz, 1H), 4.66 (s, 1H), 4.31 (q, *J* = 7.2 Hz, 2H), 3.82 (t, *J* = 4.6 Hz, 2H), 3.54 (t, *J* = 4.7 Hz, 2H), 3.36 (s, 3H), 3.31 (s, 1H), 1.28 (t, *J* = 7.2 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  173.1, 161.6, 155.7,

142.1, 138.5, 136.5, 128.9, 128.6, 128.5, 128.1, 117.4, 115.4, 93.7, 72.4, 71.8, 68.4, 67.5, 63.2, 59.4, 53.3, 14.4; MS (ES<sup>+</sup>) *m/z* 493.3 (M+1).

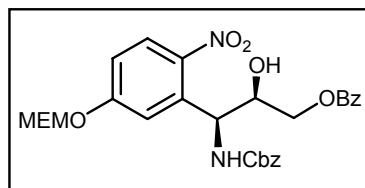
### Compound 11a



To a solution of the compound **11** (2.83 g, 6.12 mmol) in dry THF (75 mL) was added a solution of 2 M LiBH<sub>4</sub> in THF (3.06 mL, 6.12 mmol) under N<sub>2</sub> at 0°C. The solution was allowed to warm to room temperature, and then stirred overnight. The reaction was quenched by saturated ammonium chloride solution.

The aqueous phase was extracted with ethyl acetate. The combined organic layer was washed with brine, dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (3:1 ethyl acetate/hexanes) to give the product **11a** (1.81 g, 70%) as yellowish oil. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.04 (d, *J* = 9.0 Hz, 1H), 7.42-7.20 (m, 5H), 7.12-6.85 (m, 2H), 6.42 (d, *J* = 8.1 Hz, 1H), 5.53 (d, *J* = 5.6 Hz, 1H), 5.32-5.18 (m, 2H), 5.05 (s, 2H), 4.01 (s, 1H), 3.85-3.60 (m, 5H), 3.54 (m, 2H), 3.27 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 161.4, 156.5, 142.1, 140.2, 136.6, 128.9, 128.8, 128.7, 128.6, 128.5, 128.3, 128.2, 127.6, 116.5, 116.0, 115.5, 93.6, 73.8, 71.8, 68.2, 67.5, 64.7, 59.2, 52.5; MS (ES<sup>+</sup>) *m/z* 451.2 (M+1).

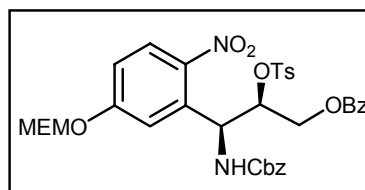
### Compound 11b



To a solution of diol **11a** (1.67 g, 3.71 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> (50 mL) was added dry pyridine (330 μL, 4.08 mmol). The solution was cooled to -25 °C. Benzoyl chloride (409 μL, 3.52 mmol) in CH<sub>2</sub>Cl<sub>2</sub> was added to the solution dropwise. The solution was stirred at -25 °C for 2 h. The reaction was quenched with methanol (0.5 mL), and washed with 1% HCl, brine, and

saturated NaHCO<sub>3</sub> solution. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product **11b** (1.80 g, 88%) as yellow syrup. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.11 (m, 3H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 1H), 7.40-7.20 (m, 7H), 7.06 (dd, *J* = 9.0, 2.1 Hz, 1H), 6.17 (d, *J* = 8.3 Hz, 1H), 5.78 (d, *J* = 8.3 Hz, 1H), 5.36 (d, *J* = 7.1 Hz, 1H), 5.31 (d, *J* = 7.1 Hz, 1H), 5.06 (s, 2H), 4.61 (dd, *J* = 11.4, 6.8 Hz, 1H), 4.51 (dd, *J* = 11.4, 4.8 Hz, 1H), 4.38 (m, 1H), 3.80 (m, 2H), 3.56 (m, 1H), 3.49 (m, 1H), 3.28 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 167.4, 161.3, 156.2, 142.0, 139.9, 136.5, 133.7, 130.2, 130.0, 129.0, 128.9, 128.6, 128.5, 128.3, 116.6, 115.6, 93.4, 71.8, 71.7, 68.0, 67.6, 66.9, 59.2, 52.5; MS (ES<sup>+</sup>) *m/z* 555.3.2 (M+1).

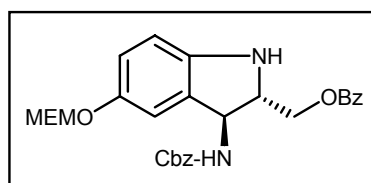
### Compound 11c



To a solution of the compound **11b** (1.63 g, 2.98 mmol) in dry CH<sub>2</sub>Cl<sub>2</sub> (30 mL) were added TsCl (852 mg, 4.47 mmol) and DMAP (728 mg, 5.96 mmol). The solution was stirred at room temperature for 24 h. The solution was washed with brine. The organic layer was dried over sodium sulfate, filtered, and

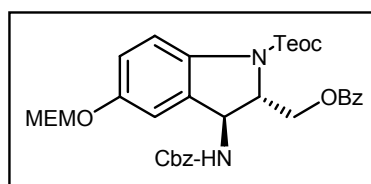
concentrated under reduced pressure. The crude product was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product **11c** (1.82 g, 87%) as yellow syrup. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.09 (d, *J* = 9.1 Hz, 1H), 8.02 (d, *J* = 7.6 Hz, 2H), 7.59-7.51 (m, 3H), 7.43 (t, *J* = 7.6 Hz, 2H), 7.35 (m, 5H), 7.11-7.04 (m, 4H), 6.02 (d, *J* = 9.2 Hz, 1H), 5.96 (d, *J* = 9.2 Hz, 1H), 5.35-5.28 (m, 3H), 5.07 (s, 2H), 4.68 (dd, *J* = 11.8, 5.9 Hz, 1H), 4.52 (dd, *J* = 11.8, 6.2 Hz, 1H), 3.83 (dd, *J* = 9.1, 4.4 Hz, 2H), 3.55 (dd, *J* = 9.1, 4.4 Hz, 2H), 3.36 (s, 3H), 2.30 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.2, 162.0, 155.8, 145.4, 141.7, 137.6, 136.2, 133.7, 132.9, 130.3, 130.2, 129.6, 128.8, 128.7, 128.6, 127.9, 93.8, 80.5, 71.8, 68.6, 67.9, 63.3, 59.4, 52.6, 22.0; MS (ES<sup>+</sup>) *m/z* 709.3 (M+1).

## Compound 12



To a solution of the compound **11c** (1.00 g, 1.41 mmol) in dry THF (25 mL) was added Lindlar catalyst (300 mg, 5% palladium over activated carbon). The reaction mixture was then subjected to hydrogenation under atmospheric pressure until TLC indicated the complete reduction of the nitro group. The mixture was filtered through celite. The organic solution was concentrated under reduced pressure. Then the intermediate was dissolved in dry THF (25 mL). Anhydrous potassium carbonate (390 mg, 2.82 mmol) was added to the solution. The mixture was stirred at 55 °C for 24 h. The mixture was diluted with ethyl acetate, and washed with brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product **12** (532 mg, 75%) as yellow syrup. <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.00 (d, *J* = 7.6 Hz, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.44-7.28 (m, 7H), 6.99 (s, 1H), 6.86 (dd, *J* = 8.4, 2.0 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 1H), 5.35 (bs, 1H), 5.21-5.11 (m, 5H), 4.62 (dd, *J* = 11.2, 3.7 Hz, 1H), 4.40 (dd, *J* = 11.0, 7.4 Hz, 1H), 4.10-3.90 (m, 2H), 3.83 (t, *J* = 4.5 Hz, 2H), 3.57 (t, *J* = 4.6 Hz, 2H), 3.38 (s, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 166.8, 156.3, 151.7, 145.4, 136.7, 133.5, 130.3, 130.1, 129.0, 128.8, 128.7, 128.6, 119.0, 114.4, 111.4, 95.1, 72.0, 67.9, 67.4, 67.1, 66.7, 59.4, 56.9; MS (ES<sup>+</sup>) *m/z* 507.3 (M+1).

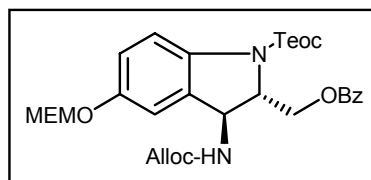
## Compound 12a



To a solution of 2-(trimethylsilyl)ethanol (295 μL, 2.06 mmol) and bis(trichloromethyl)carbonate (204 mg, 0.88 mmol) in dichloromethane (10 mL) cooled to 0 °C was added pyridine (167 μL, 2.06 mmol) dropwise. The solution was stirred at 0 °C for 2 h. Then a solution of the compound **12** (520 mg, 1.03 mmol) and pyridine (250 μL, 3.09 mmol) in dichloromethane (10 mL) was added dropwise to the solution of 2-trimethylsilylethoxycarbonyl chloride. The reaction was continued for 1 h at 0 °C. The solution was washed with 1% HCl solution, water, and brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:2 ethyl acetate/hexanes) to give the product **12a** (604 mg, 98%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.76 (bs, 1H), 7.70 (d, *J* = 7.4 Hz, 2H), 7.49 (t, *J* = 7.3 Hz, 1H), 7.38-7.29 (m, 7H), 7.10 (s, 1H), 7.04 (dd, *J* = 8.8, 2.0 Hz, 1H), 5.21-5.10 (m, 6H), 4.65-4.55 (m, 3H), 4.29 (m, 2H), 3.82 (t, *J* = 4.5 Hz, 2H), 3.55 (t, *J* = 4.6

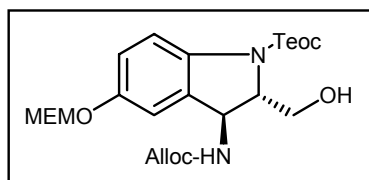
Hz, 2H), 3.36 (s, 3H), 1.09 (m, 2H), 0.06 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.5, 155.8, 154.1, 136.5, 134.0, 130.0, 129.0, 128.7, 128.3, 118.9, 116.8, 114.1, 94.7, 72.0, 68.0, 67.5, 66.5, 65.1, 64.8, 59.4, 18.2, 1.1; MS ( $\text{ES}^+$ )  $m/z$  651.4 ( $\text{M}+1$ ).

### Compound 13



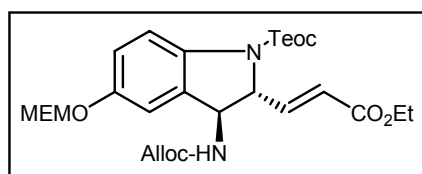
To a solution of the compound **12a** (790 mg, 1.21 mmol) in methanol (20 mL) was added Palladium catalyst (158 mg, 10% palladium over activated carbon). The reaction mixture was then subjected to hydrogenation under atmospheric pressure for 3 h. The mixture was filtered through celite. The organic solution was concentrated under reduced pressure. Then the intermediate was dissolved in dry THF (15 mL). Alloc chloride (161  $\mu\text{L}$ , 1.51 mmol) and pyridine (147  $\mu\text{L}$ , 1.82 mmol) was added to the solution at  $0^\circ\text{C}$ . The mixture was stirred at  $0^\circ\text{C}$  for 1 h. The mixture was diluted with ethyl acetate, and washed with 1% HCl solution and brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:2 ethyl acetate/hexanes) to give the product **13** (617 mg, 85%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.76 (bs, 1H), 7.68 (d,  $J = 7.7$  Hz, 2H), 7.49 (t,  $J = 7.3$  Hz, 1H), 7.33-7.28 (m, 2H), 7.09 (d,  $J = 2.2$  Hz, 1H), 7.03 (dd,  $J = 8.8, 2.0$  Hz, 1H), 5.92 (m, 1H), 5.34-5.05 (m, 5H), 4.62-4.57 (m, 5H), 4.29 (bs, 2H), 3.83 (t,  $J = 4.6$  Hz, 2H), 3.54 (t,  $J = 4.6$  Hz, 2H), 3.37 (s, 3H), 1.09 (bs, 2H), 0.05 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.5, 155.6, 154.1, 133.3, 132.9, 130.0, 128.6, 118.9, 188.4, 116.8, 114.1, 94.7, 72.0, 68.0, 66.5, 66.3, 65.1, 64.8, 59.4, 18.2, -1.1; MS ( $\text{ES}^+$ )  $m/z$  601.3 ( $\text{M}+1$ ).

### Compound 13a



To a solution of the compound **13** (586 mg, 0.98 mmol) in methanol (25 mL) was added potassium carbonate (135 mg, 0.98 mmol). The mixture was stirred at room temperature. After 2h, TLC showed the reaction was complete. The reaction was neutralized with Amberlite  $\text{H}^+$  resin to pH = 7. The mixture was then filtered. The filtrate was concentrated under reduced pressure. The crude product was diluted with ethyl acetate, and washed with brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product **13a** (480 mg, 99%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.68 (bs, 1H), 7.06 (d,  $J = 2.4$  Hz, 1H), 7.03 (dd,  $J = 8.8, 2.4$  Hz, 1H), 5.94 (m, 1H), 5.34 (d,  $J = 17.2$  Hz, 1H), 5.27-5.23 (m, 3H), 4.99 (d,  $J = 5.6$  Hz, 1H), 4.63 (d,  $J = 4.6$  Hz, 1H), 4.38-4.30 (m, 2H), 3.98 (bs, 1H), 3.83 (t,  $J = 4.6$  Hz, 2H), 3.70 (bs, 1H), 3.58 (t,  $J = 4.6$  Hz, 2H), 3.39 (s, 3H), 3.13 (bs, 1H), 1.13 (t,  $J = 8.6$  Hz, 2H), 0.08 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  156.3, 154.1, 132.7, 118.9, 118.7, 117.0, 113.9, 94.5, 72.0, 69.9, 68.0, 66.6, 64.8, 63.8, 59.4, 55.2, 18.4, -1.1; MS ( $\text{ES}^+$ )  $m/z$  497.4 ( $\text{M}+1$ ).

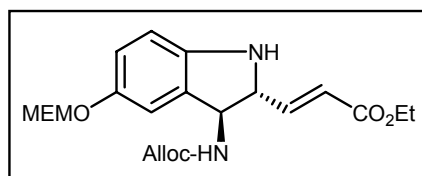
### Compound 14



The Dess-Martin periodinane (810 mg, 1.92 mmol) was added to a solution of the compound **13a** (475 mg, 0.96

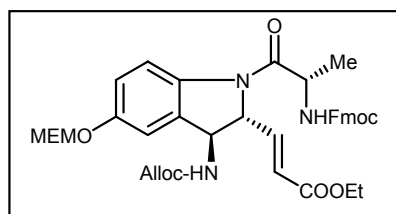
mmol) in dichloromethane (25 mL). The resulting suspension was stirred at room temperature. TLC showed complete conversion to the aldehyde after 1 h. Then (carbethoxymethylene)triphosphorane (836 mg, 2.4 mmol) was added to the reaction. The reaction was stirred for another 1 h. The mixture was washed with saturated sodium bicarbonate solution and brine sequentially. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (2:3 ethyl acetate/hexanes) to give the product **14** (500 mg, 93%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.83 (bs, 1H), 7.07-7.05 (m, 2H), 6.95 (dd,  $J$  = 15.6, 5.7 Hz, 1H), 5.98-5.89 (m, 2H), 5.33 (d,  $J$  = 17.2 Hz, 1H), 5.25 (d,  $J$  = 15.3 Hz, 1H), 5.23 (s, 2H), 5.06 (d,  $J$  = 6.7 Hz, 1H), 4.92 (bs, 1H), 4.84 (d,  $J$  = 6.7 Hz, 1H), 4.62 (bs, 2H), 4.30 (bs, 2H), 4.18 (q,  $J$  = 7.1 Hz, 2H), 3.83 (t,  $J$  = 4.6 Hz, 2H), 3.58 (t,  $J$  = 4.6 Hz, 2H), 3.39 (s, 3H), 1.28 (t,  $J$  = 7.1 Hz, 3H), 1.07 (bs, 2H), 0.07 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.3, 155.6, 154.2, 144.2, 132.8, 122.6, 119.3, 118.5, 117.0, 114.4, 94.5, 72.0, 68.3, 68.1, 66.4, 64.8, 61.0, 59.4, 57.2, 18.2, 14.6, -1.1; MS ( $\text{ES}^+$ )  $m/z$  565.4 ( $\text{M}+1$ ).

### Compound 14a



TBAF solution (1 M, 1.76 mL, 1.76 mmol) was added to the solution of the compound **14** (495 mg, 0.88 mmol) in THF (15 mL). The solution was stirred at room temperature for 2 h. The solution was washed with saturated  $\text{NH}_4\text{Cl}$  solution, dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (2:3 ethyl acetate/hexanes) to give the product **14a** (359 mg, 97%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  6.98 (dd,  $J$  = 15.6, 6.7 Hz, 1H), 6.92 (d,  $J$  = 2.1 Hz, 1H), 6.85 (dd,  $J$  = 2.1, 8.5 Hz, 1H), 6.56 (d,  $J$  = 8.5 Hz, 1H), 6.05 (d,  $J$  = 15.6 Hz, 1H), 5.92 (m, 1H), 5.39 (d,  $J$  = 8.3 Hz, 1H), 5.31 (d,  $J$  = 15.6 Hz, 1H), 5.22 (d,  $J$  = 10.7 Hz, 1H), 5.14 (s, 2H), 4.98 (t,  $J$  = 6.8 Hz, 1H), 4.59 (d,  $J$  = 5.3 Hz, 1H), 4.20-4.14 (m, 3H), 3.81 (t,  $J$  = 4.6 Hz, 2H), 3.56 (t,  $J$  = 4.6 Hz, 2H), 3.37 (s, 3H), 1.27 (t,  $J$  = 7.1 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.6, 156.0, 151.7, 147.0, 145.1, 133.0, 128.3, 122.6, 119.0, 118.3, 114.5, 111.3, 95.1, 72.0, 68.5, 67.9, 66.2, 60.9, 59.5, 59.4, 14.6; MS ( $\text{ES}^+$ )  $m/z$  421.3 ( $\text{M}+1$ ).

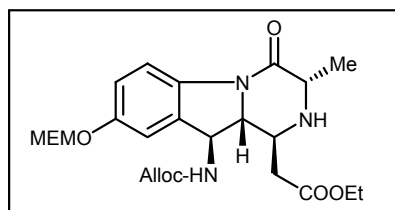
### Compound 15



To a solution of the compound **14a** (34 mg, 0.081 mmol) in  $\text{CH}_2\text{Cl}_2$  (2 mL) were added Fmoc-Ala-Cl (66.8 mg, 0.20 mmol) and pyridine (19.4  $\mu\text{L}$ , 0.24 mmol). The solution was stirred at room temperature for 2h. The solution was diluted with ethyl acetate, washed with 1% HCl solution and brine, dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product **15** (51 mg, 88%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.19 (d,  $J$  = 8.7 Hz, 1H), 7.78 (d,  $J$  = 7.4 Hz, 2H), 7.66-7.52 (m, 2H), 7.42 (t,  $J$  = 7.4 Hz, 2H), 7.33 (t,  $J$  = 7.2 Hz, 2H), 7.18-6.90 (m, 3H), 6.00 (d,  $J$  = 15.9 Hz, 1H), 5.93 (m, 1H), 5.79 (d,  $J$  = 7.5 Hz, 1H), 5.37-5.21 (m, 4H), 5.15 (bs, 1H), 4.97 (bs, 1H), 4.85 (bs, 1H), 4.65 (bs, 2H), 4.46-4.30 (m, 3H), 4.28-4.08 (m, 3H), 3.83 (t,  $J$  = 4.5 Hz, 2H), 3.57 (t,  $J$  = 4.5 Hz, 2H), 3.39 (s, 3H), 1.43 (d,  $J$  = 6.2 Hz, 3H),

1.23 (t,  $J = 7.0$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.1, 165.7, 155.7, 155.6, 155.5, 144.4, 144.2, 142.8, 141.7, 137.4, 132.7, 129.4, 128.1, 127.5, 125.6, 123.8, 120.4, 119.6, 119.2, 118.7, 118.3, 114.0, 94.3, 72.0, 68.2, 67.4, 66.5, 61.2, 59.4, 58.1, 49.3, 47.6, 20.3, 14.5; MS ( $\text{ES}^+$ )  $m/z$  714.5 ( $\text{M}+1$ ).

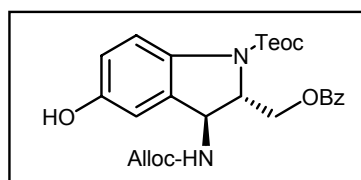
### Compound 16



To the compound **15** (45 mg, 0.063 mmol) was added 20% piperidine of  $\text{CH}_2\text{Cl}_2$  solution (2.5 mL). The solution was stirred at room temperature for 1 h. The solution was concentrated under reduced pressure. The crude product was purified by column chromatography (ethyl acetate) to give the mixture of two diastereomers **16** and **17** (>10:1, the ratio was judged by  $^1\text{H}$  NMR) (30 mg, 96%). Compound **16**:  $^1\text{H}$  NMR

(400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.00 (d,  $J = 8.6$  Hz, 1H), 7.00-6.93 (m, 2H), 5.95 (m, 1H), 5.49 (d,  $J = 9.7$  Hz, 1H), 5.36 (dd,  $J = 17.2, 1.1$  Hz, 1H), 5.29 (t,  $J = 9.4$  Hz, 1H), 5.27 (dd,  $J = 10.4, 1.1$  Hz, 1H), 5.21 (s, 2H), 4.66 (d,  $J = 4.5$  Hz, 2H), 4.17 (m, 2H), 3.90 (t,  $J = 9.4$  Hz, 1H), 3.80 (dd,  $J = 5.6, 3.6$  Hz, 2H), 3.70 (q,  $J = 7.1$  Hz, 1H), 3.55 (dd,  $J = 5.6, 3.6$  Hz, 2H), 3.48 (dt,  $J = 9.1, 3.0$  Hz, 1H), 3.38 (s, 3H), 2.88 (dd,  $J = 16.6, 2.9$  Hz, 1H), 2.57 (dd,  $J = 16.6, 8.2$  Hz, 1H), 1.43 (d,  $J = 7.1$  Hz, 3H), 1.35 1.28 (t,  $J = 7.1$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.8, 169.9, 156.4, 155.1, 136.3, 132.8, 132.0, 118.6, 118.4, 117.8, 112.4, 94.2, 71.9, 70.5, 68.1, 66.7, 61.2, 59.4, 56.5, 53.3, 50.6, 36.3, 19.2, 14.6; MS ( $\text{ES}^+$ )  $m/z$  492.2 ( $\text{M}+1$ ).

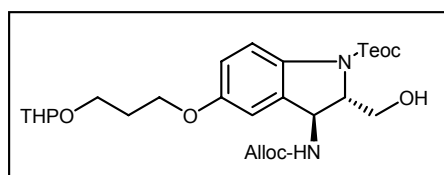
### Compound 13b



To a solution of the compound **13** (2.65g, 4.41 mmol) in EtOH (265 mL) was added *p*-TSA (840 mg, 4.41 mmol). The solution was stirred at  $50^\circ\text{C}$  for 24 h. The solution was diluted with ethyl acetate (50 mL), and washed with sodium bicarbonate solution and brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude

product was purified by column chromatography (2:3 ethyl acetate/hexanes) to give the product **13b** (2.12 g, 94%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.69 (d,  $J = 7.5$  Hz, 2H), 7.47 (t,  $J = 7.1$  Hz, 1H), 7.31 (dd,  $J = 8.6, 7.5$  Hz, 2H), 6.86 (s, 1H), 6.81 (d,  $J = 8.6$  Hz, 1H), 5.90 (m, 1H), 5.50 (d,  $J = 6.7$  Hz, 1H), 5.31 (d,  $J = 17.1$  Hz, 1H), 5.22 (d,  $J = 10.4$  Hz, 1H), 5.06 (d,  $J = 7.2$  Hz, 1H), 4.64-4.55 (m, 4H), 4.49 (d,  $J = 8.4$  Hz, 1H), 4.26 (bs, 2H), 1.07 (bs, 2H), 0.04 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.8, 156.0, 153.5, 153.1, 133.4, 132.8, 130.0, 129.9, 128.7, 118.5, 117.0, 112.8, 66.4, 66.3, 65.1, 64.9, 55.0, 18.2, -1.1; MS ( $\text{ES}^+$ )  $m/z$  513.3 ( $\text{M}+1$ ).

### Compound 13c

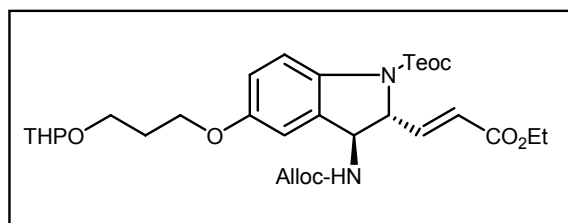


The compound **13b** (2.10 g, 4.10 mmol), 3-(tetrahydro-2H-pyran-2-yloxy)propyl 4-methylbenzenesulfonate (1.61 g, 5.13 mmol), and cesium carbonate (2.00g, 6.15 mmol) were added to DMF (30 mL). The mixture was stirred at  $40^\circ\text{C}$  for 12 h. Then DMF was removed under reduced



pressure. The mixture was diluted with ethyl acetate (50 mL) and washed with brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:2 ethyl acetate/hexanes). The intermediate was directly used in the next step. To a solution of this intermediate in methanol (50 mL) was added potassium carbonate (567 mg, 4.10 mmol). The mixture was stirred at room temperature for 2 h. The solution was diluted with ethyl acetate and washed with brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (2:3 ethyl acetate/hexanes) to give the product **13c** (1.91 g, 85%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.70 (bs, 1H), 6.91-6.63 (m, 2H), 5.92 (m, 1H), 5.35-5.13 (m, 3H), 4.98 (bs, 1H), 4.60 (m, 3H), 4.31 (m, 3H), 4.10-3.47 (m, 8H), 2.74 (bs, 1H), 2.04 (m, 2H), 1.86-1.48 (m, 6H), 0.94 (t,  $J = 7.4$  Hz, 2H), 0.07 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  156.3, 156.0, 155.7, 132.8, 118.6, 116.9, 116.8, 116.7, 111.8, 99.3, 66.8, 66.5, 66.0, 64.4, 64.3, 62.7, 60.5, 55.2, 31.1, 30.1, 25.8, 20.0, 18.3, -1.1; MS ( $\text{ES}^+$ )  $m/z$  551.3 ( $\text{M}+1$ ).

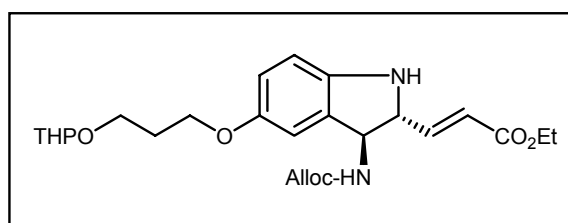
### Compound 13d



Sodium bicarbonate (2.27g, 27.04 mmol) and the Dess-Martin periodinane (2.86 g, 6.76 mmol) were added to a solution of the compound **13c** (1.86 g, 3.38 mmol) in dichloromethane (50 mL). The resulting suspension was stirred at room temperature. TLC showed complete conversion to the aldehyde after 5h. Then

(carbethoxymethylene)triphosphorane (2.94 g, 8.45 mmol) was added to the mixture. The mixture was stirred for another 2h. The mixture was washed with brine. The organic layer was dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:3 ethyl acetate/hexanes) to give the product **13d** (1.52 g, 73%).  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.81 (bs, 1H), 6.97-6.88 (m, 3H), 5.97-5.89 (m, 2H), 5.33 (d,  $J = 17.2$  Hz, 1H), 5.19 (d,  $J = 10.3$  Hz, 1H), 5.12 (d,  $J = 6.7$  Hz, 1H), 4.90 (bs, 1H), 4.84 (d,  $J = 7.1$  Hz, 1H), 4.61 (m, 3H), 4.29 (bs, 2H), 4.17 (q,  $J = 7.2$  Hz, 2H), 4.05 (t,  $J = 6.2$  Hz, 2H), 3.95-3.83 (m, 2H), 3.60-3.49 (m, 2H), 2.06 (t,  $J = 6.2$  Hz, 2H), 1.83-1.47 (m, 6H), 1.27 (t,  $J = 7.2$  Hz, 3H), 0.06 (s, 9H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  166.3, 156.1, 155.6, 144.3, 132.8, 122.5, 118.5, 117.2, 117.1, 117.0, 112.2, 99.4, 68.2, 66.4, 66.0, 64.7, 64.3, 62.7, 60.9, 57.4, 31.1, 30.1, 25.8, 20.0, 18.2, 14.6, -1.1; MS ( $\text{ES}^+$ )  $m/z$  619.5 ( $\text{M}+1$ ).

### Compound 13e

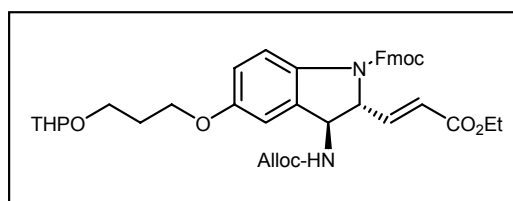


TBAF solution (1 M, 4.76 mL, 4.76 mmol) was added to the solution of the compound **13d** (1.47 g, 2.38 mmol) in THF (20 mL). The solution was stirred at room temperature for 1 h. The organic solution was washed with brine, dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified

by column chromatography (1:2 ethyl acetate/hexanes) to give the product **13e** (0.97 g, 86%).  $^1\text{H}$

NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  6.97 (dd,  $J$  = 15.6, 6.7 Hz, 1H), 6.77 (s, 1H), 6.70 (dd,  $J$  = 8.4, 2.1 Hz, 1H), 6.56 (d,  $J$  = 8.4 Hz, 1H), 6.04 (d,  $J$  = 15.6 Hz, 1H), 5.92 (m, 1H), 5.48 (d,  $J$  = 8.4 Hz, 1H), 5.32 (d,  $J$  = 17.2 Hz, 1H), 5.21 (d,  $J$  = 10.4 Hz, 1H), 4.97 (t,  $J$  = 7.0 Hz, 1H), 4.58 (m, 3H), 4.16 (q,  $J$  = 7.1 Hz, 3H), 3.97 (t,  $J$  = 6.3 Hz, 2H), 3.92-3.78 (m, 3H), 3.57-3.45 (m, 2H), 2.01 (t,  $J$  = 6.3 Hz, 2H), 1.85-1.43 (m, 6H), 1.26 (t,  $J$  = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.5, 156.0, 146.0, 133.0, 129.1, 123.2, 118.4, 117.0, 112.8, 112.6, 99.4, 68.4, 66.9, 66.3, 64.5, 62.8, 61.0, 59.6, 31.1, 30.1, 25.8, 20.0, 14.6; MS (ES<sup>+</sup>)  $m/z$  475.4 (M+1).

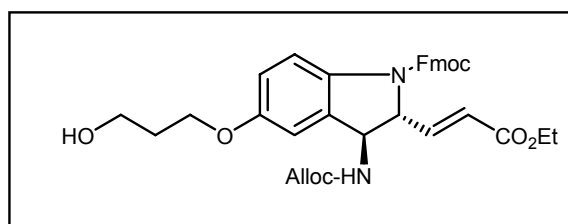
### Compound 13f



To a solution of the compound **13e** (940 mg, 1.98 mmol) in ethyl acetate (20 mL) was added 5% sodium bicarbonate solution (20 mL) and Fmoc-chloride (640 mg, 2.48 mmol). The mixture was stirred at room temperature for 3h. The mixture was separated. The organic phase was washed with brine, dried over

sodium sulfate, filtered, and concentrated under reduced pressure. The crude product was purified by column chromatography (1:2 ethyl acetate/hexanes) to give the product **13f** (1.20 g, 87%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 (m, 3H), 7.57 (d,  $J$  = 6.1 Hz, 2H), 7.41 (t,  $J$  = 7.3 Hz, 2H), 7.32 (t,  $J$  = 7.3 Hz, 2H), 6.95-6.50 (m, 3H), 5.96 (m, 1H), 5.79 (m, 1H), 5.43-5.22 (m, 2H), 5.03 (m, 1H), 4.92-4.50 (m, 7H), 4.28 (m, 1H), 4.18 (q,  $J$  = 7.1 Hz, 2H), 4.03 (m, 2H), 3.94-3.82 (m, 2H), 3.61-3.48 (m, 2H), 2.06 (t,  $J$  = 6.3 Hz, 2H), 1.90-1.48 (m, 6H), 1.29 (t,  $J$  = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.2, 155.6, 144.0, 141.9, 132.8, 128.2, 127.6, 125.3, 125.1, 122.6, 120.5, 118.6, 117.2, 117.0, 112.1, 99.4, 68.3, 67.8, 66.4, 66.0, 64.3, 62.8, 60.9, 57.6, 47.6, 31.1, 30.1, 25.8, 20.0, 14.6; MS (ES<sup>+</sup>)  $m/z$  697.3 (M+1).

### Compound 21a

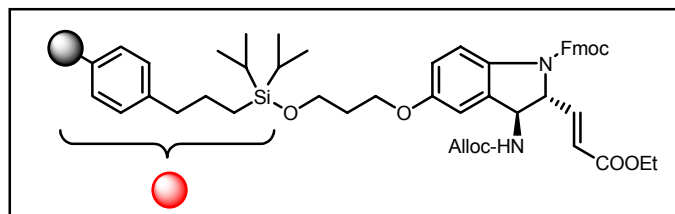


PPTS (42.2 mg, 0.168 mmol) was added to a solution of the compound **13f** (1.17 g, 1.68 mmol) in ethanol (20 mL). The solution was stirred at 55 °C for 8 h. The solution was diluted with ethyl acetate (40 mL), washed with brine, dried over sodium sulfate, filtered, and concentrated under reduced pressure. The crude

product was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product **21a** (1.01 g, 98%). <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.79 (m, 3H), 7.57 (d,  $J$  = 6.2 Hz, 2H), 7.42 (t,  $J$  = 7.3 Hz, 2H), 7.30 (t,  $J$  = 7.3 Hz, 2H), 6.97-6.50 (m, 3H), 5.96 (m, 1H), 5.78 (m, 1H), 5.44-5.18 (m, 2H), 4.95-4.50 (m, 6H), 4.28 (m, 1H), 4.18 (q,  $J$  = 7.1 Hz, 2H), 4.07 (m, 2H), 3.86 (t,  $J$  = 6.0 Hz, 2H), 2.03 (m, 2H), 1.78 (s, 1H), 1.29 (t,  $J$  = 7.1 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)  $\delta$  166.2, 156.0, 155.6, 152.9, 144.1, 141.9, 132.8, 128.2, 127.6, 125.3, 125.1, 122.6, 120.5, 120.4, 118.6, 117.2, 117.0, 112.1, 68.3, 67.8, 66.6, 66.4, 61.0, 60.7, 57.5, 47.6, 32.4, 14.6; MS (ES<sup>+</sup>)  $m/z$  613.3 (M+1).

## Solid-Phase Synthesis

### Compound 21b

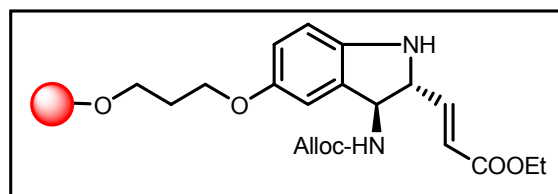


**Loading:** 3-[Diisopropyl(*p*-methoxyphenyl)silyl]propyl functionalized resin (110 mg, 0.157 mmol) was swollen in CH<sub>2</sub>Cl<sub>2</sub> (1.5 mL) under N<sub>2</sub> for 30 min. The solvent was then drained under positive N<sub>2</sub> pressure.

A solution of trifluoromethanesulfonic acid in CH<sub>2</sub>Cl<sub>2</sub> (4%, 2.1 mL) was added by syringe. The resin was then gently agitated for 30 min under N<sub>2</sub>. The acid solvent was drained under positive N<sub>2</sub> pressure. The activated resin was treated with 2,6-lutidine (0.15 mL) for 15 min and followed by addition of a solution of compound **21a** (254 mg, 0.415 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (1.5 mL). The resin was gently shaken overnight. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was then dried under vacuum overnight to give 183 mg of the loaded resin **21b**.

**Cleavage:** The loaded resin (20 mg) in Eppendorf® tube was swelled in THF (0.5 mL) for 30 min, and treated with HF-pyridine solution (15  $\mu$ L). The reaction tube was shaken for 3 h. Methoxytrimethylsilane (100  $\mu$ L) was added and the tube was shaken for another 30 min. The solution was removed and the resin was washed with THF. All solvents were combined and concentrated. The crude sample was purified by column chromatography (1:1 ethyl acetate/hexanes) to give the product (8.5 mg, 81% loading).

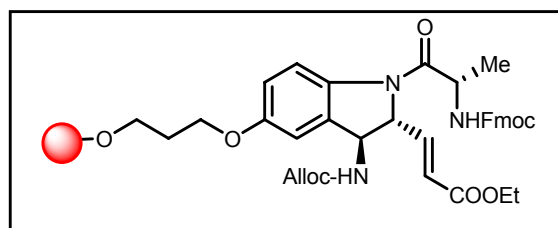
### Compound 21c



The resin (163 mg) **21b** was swelled in DMF (1.6 mL) for 30 min. Piperidine (0.4 mL) was added to the mixture. The mixture was shaken for 0.5 h. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was then dried under vacuum overnight to give 137 mg of the resin **21c**.

The dried resin (5 mg) was cleaved as the above method using HF-pyridine solution. The resulting compound was checked by MS. LRMS (MS ES<sup>+</sup>) calcd for C<sub>20</sub>H<sub>27</sub>N<sub>2</sub>O<sub>6</sub> 391.2 *m/z* (M+1)<sup>+</sup>; observed 391.2.

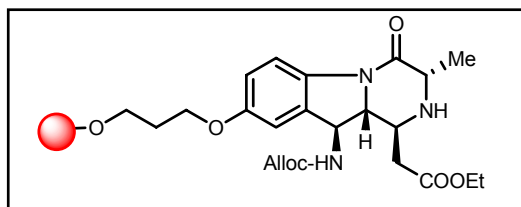
### Compound 22a



The resin **21c** (163 mg) was swelled in CH<sub>2</sub>Cl<sub>2</sub> (2.5 mL) under N<sub>2</sub> for 30 min. Collidine (145  $\mu$ L, 1.1 mmol) and Fmoc-Ala-Cl (181 mg, 0.55 mmol) were added to the mixture. The mixture was shaken for 24 h. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was

then dried under vacuum overnight to give 163 mg of the resin **22a**. The dried resin (10 mg) was cleaved as the above method using HF-pyridine solution. The resulting compound was checked by MS. LRMS (MS ES<sup>+</sup>) calcd for C<sub>38</sub>H<sub>42</sub>N<sub>3</sub>O<sub>9</sub> 684.3 *m/z* (M+1)<sup>+</sup>; observed 684.2.

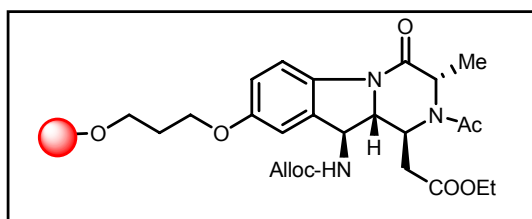
### Compound 23a



The resin **22a** (153 mg) was swelled in DMF (1.6 mL) for 30 min. Piperidine (0.4 mL) was added to the mixture. The mixture was shaken for 24 h. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was then dried under vacuum overnight to give 128 mg of the resin **23a**.

The dried resin (5 mg) was cleaved as the above method using HF-pyridine solution. The resulting compound was checked by MS. LRMS (MS ES<sup>+</sup>) calcd for C<sub>23</sub>H<sub>32</sub>N<sub>3</sub>O<sub>7</sub> 462.2 *m/z* (M+1)<sup>+</sup>; observed 462.3.

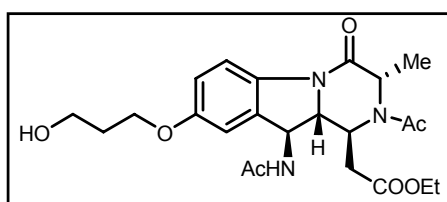
### Compound 24a



The resin **23a** (123 mg) was swelled in DMF (1.6 mL) for 30 min. Pyridine (162  $\mu$ L, 2 mmol) and acetic anhydride (95  $\mu$ L, 1 mmol) were added to the mixture. The mixture was shaken for 24 h. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was then dried under vacuum overnight to give 125 mg of the resin **24a**.

The dried resin (10 mg) was cleaved as the above method using HF-pyridine solution. The resulting compound was checked by MS. LRMS (MS ES<sup>+</sup>) calcd for C<sub>25</sub>H<sub>34</sub>N<sub>3</sub>O<sub>8</sub> 504.2 *m/z* (M+1)<sup>+</sup>; observed 504.2.

### Compound 25a



The resin **24a** (43 mg) was swelled in CH<sub>2</sub>Cl<sub>2</sub> (1.6 mL) for 30 min. Morpholine (70  $\mu$ L, 0.8 mmol) and tetrakis(triphenylphosphine) palladium (0) (46 mg, 0.04 mmol) were added to the mixture. The mixture was shaken for 2 h. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was then dried

under vacuum for 4 h. The resin was then swelled in CH<sub>2</sub>Cl<sub>2</sub> (1.5 mL) for 30 min. Pyridine (65  $\mu$ L, 0.80 mmol) and acetic anhydride (38  $\mu$ L, 0.40 mmol) were added to the mixture. The mixture was shaken for 24 h. The resin was washed with DMF (3 x), THF (3 x), and CH<sub>2</sub>Cl<sub>2</sub> (3 x). The resin was then dried under vacuum overnight. The dried resin was cleaved as the above method using HF-pyridine solution. The crude product was purified by column chromatography (ethyl acetate) to give the mixture of two diastereomers (13.2 mg, overall yield: ~84%) (4.5:1, the ratio was judged by <sup>1</sup>H NMR). LRMS (MS ES<sup>+</sup>) calcd for C<sub>23</sub>H<sub>32</sub>N<sub>3</sub>O<sub>7</sub> 462.2 *m/z* (M+1)<sup>+</sup>; observed 462.3;

Compound **25** (major product):  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.78 (d,  $J$  = 8.7 Hz, 1H), 7.13 (d,  $J$  = 9.6 Hz, 1H), 6.78 (dd,  $J$  = 8.7, 2.3 Hz, 1H), 6.74 (d,  $J$  = 1.7 Hz, 1H), 5.55 (t,  $J$  = 9.6 Hz, 1H), 4.43 (q,  $J$  = 7.1 Hz, 1H), 4.20-4.00 (m, 5H), 3.93 (dt,  $J$  = 10.4, 4.7 Hz, 1H), 3.85 (t,  $J$  = 5.8 Hz, 2H), 3.66 (dd,  $J$  = 16.6, 10.4 Hz, 1H), 2.94 (dd,  $J$  = 16.6, 4.7 Hz, 1H), 2.53 (bs, 1H), 2.15 (s, 3H), 2.02 (t,  $J$  = 5.9 Hz, 2H), 1.90 (s, 3H), 1.63 (d,  $J$  = 7.1 Hz, 3H), 1.27 (t,  $J$  = 7.1 Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  171.9, 170.9, 170.8, 166.8, 157.5, 134.3, 133.5, 119.0, 115.2, 110.4, 67.8, 66.4, 61.2, 60.2, 57.7, 54.0, 53.4, 33.6, 32.4, 23.6, 23.5, 17.1, 14.6.

### **Library Synthesis (90 compounds/2 diastereomers per well) by Parallel and IRORI Split-and-Mix Approach**

**Loading of Compound 21a.** The *p*-methoxyphenylsilane beads (500 mg, 1.29 mmol/g of silane, ICCB batch MX-19) were swollen in  $\text{CH}_2\text{Cl}_2$  (2 mL) under  $\text{N}_2$  for 30 min. The solvent was then drained under positive  $\text{N}_2$  pressure. A solution of trifluoromethanesulfonic acid in  $\text{CH}_2\text{Cl}_2$  (4%, 8.5 mL, 3.87 mmol) was added by syringe. The resin was then left to sit for 2 h under  $\text{N}_2$ . The acid solvent was drained under positive  $\text{N}_2$  pressure. The activated resin was treated with 2,6-lutidine (0.60 mL, 5.16 mmol) for 30 min and followed by addition of a solution of compound **21a** (790 mg, 1.29 mmol) in  $\text{CH}_2\text{Cl}_2$  (1.5 mL). The resin was gently shaken overnight. The resin was washed with  $\text{CH}_2\text{Cl}_2$  (3 x), THF (3 x), and  $\text{CH}_2\text{Cl}_2$  (3 x). The beads were then dried on the lyophilizer overnight to give the loaded beads (776 mg, 87% loading).

**Fmoc Removal.** The beads (776 mg, 0.56 mmol) were swelled in DMF (4 mL) for 30 min. The solvent was then drained. A solution of 20% piperidine in DMF (6 mL) was added to the beads. The beads were shaken for 0.5 h. The resin was washed with  $\text{CH}_2\text{Cl}_2$  (3 x), THF (3 x),  $\text{CH}_2\text{Cl}_2$  (3 x) and then dried on the lyophilizer overnight.

**Amino acid coupling (1<sup>st</sup> Diversity Point).** The beads from above were split into 5 parts (each 0.112 mmol), each of which was mixed with dry  $\text{CH}_2\text{Cl}_2$  (5 mL), collidine (291  $\mu\text{L}$ , 2.20 mmol), and 5 different Fmoc amino acid chlorides (0.825 mmol, Fmoc-Gly-Cl, Fmoc-Ala-Cl, Fmoc-Val-Cl, Fmoc-Leu-Cl, and Fmoc-Phe-Cl) respectively. The mixtures were shaken independently at room temperature overnight. Each reaction mixture was washed with  $\text{CH}_2\text{Cl}_2$  (3 x), THF (3 x)  $\text{CH}_2\text{Cl}_2$  (3 x) and the beads were then dried on the lyophilizer overnight.

**Fmoc Removal and Cyclization.** To each portion of the dried beads was added a solution of 20% piperidine in DMF (4 mL). All the reaction mixtures were shaken for 24 h. After filtration, the beads were washed with  $\text{CH}_2\text{Cl}_2$  (3 x), THF (3 x),  $\text{CH}_2\text{Cl}_2$  (3 x) and then finally dried on the lyophilizer overnight respectively.

**Acid Coupling (2<sup>nd</sup> Diversity Point).** The beads resulted from the above reactions were distributed into 45 MicroKans (9 MicroKans for each amino acid, about 16 mg beads in each Kan) and encoded with 45 RF tags. The 45 MicroKans were sorted into 3 pools (15 Kans in each pool). Each of these 3 pools was mixed with pyridine (40 equiv.) and one of the 3 corresponding acid chloride (20 equiv., phenylacetyl chloride, 4-methoxyphenylacetyl chloride, and hydrocinnamoyl chloride) in  $\text{CH}_2\text{Cl}_2$  (50 mL). Each reaction mixture was gently stirred overnight. After filtration, the MicroKans were washed with  $\text{CH}_2\text{Cl}_2$  (3 x), THF (3 x),  $\text{CH}_2\text{Cl}_2$  (3 x) and then dried under high vacuum overnight.

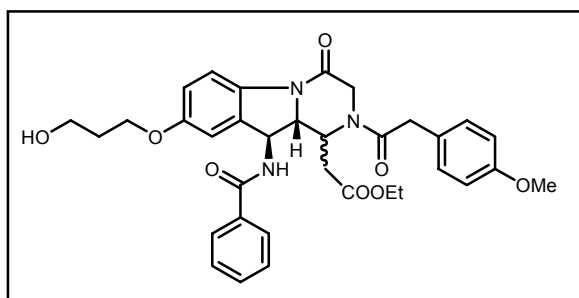
**Alloc Removal.** The 45 MicroKans were mixed with morpholine (1.91 mL, 22 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (150 mL). Tetrakis(triphenylphosphine) palladium(0) (1.27 g, 1.10 mmol) was added, and the mixture was allowed to stir gently overnight. After filtration, the MicroKans were washed with CH<sub>2</sub>Cl<sub>2</sub> (3 x), THF (3 x), CH<sub>2</sub>Cl<sub>2</sub> (3 x) and then dried under high vacuum overnight.

**Acid Coupling (3<sup>rd</sup> Diversity Point).** The 45 MicroKans were sorted into 3 pools (15 Kans in each pool). Each of these 3 pools was mixed with pyridine (40 equiv.) and one of the 3 corresponding acid chloride (20 equiv., 3,4-dimethoxybenzoyl chloride, isobutyryl chloride, and benzoyl chloride) in CH<sub>2</sub>Cl<sub>2</sub> (50 mL). Each reaction mixture was gently stirred overnight. After filtration, the MicroKans were washed with CH<sub>2</sub>Cl<sub>2</sub> (3 x), THF (3 x), CH<sub>2</sub>Cl<sub>2</sub> (3 x) and then dried under high vacuum overnight.

**Cleavage.** The beads in each MicroKan were transferred into Eppendorf® tube respectively, and cleaved under the similar conditions using HF-pyridine solution.

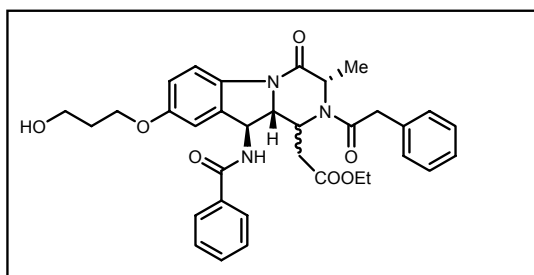
All the library members were analyzed by HPLC, MS and the following compounds were purified by column chromatography:

#### Gly1 (Mixture of Two Diastereomers)



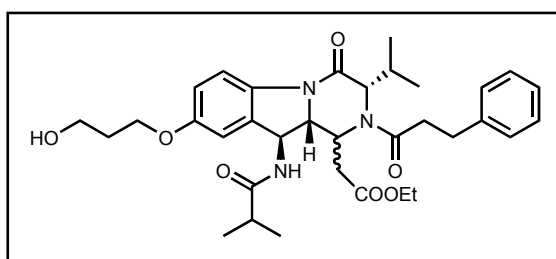
<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.07-6.37 (m, 13H), 5.83 (t, *J* = 8.4 Hz, 0.52H), 5.52 (t, *J* = 8.4 Hz, 0.48H), 5.14 (m, 0.52H), 4.78 (m, 0.48H), 4.45-3.40 (m, 14H), 3.19-2.65 (m, 2H), 2.03 (t, *J* = 5.3 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H); LRMS (ES<sup>+</sup>) for C<sub>34</sub>H<sub>37</sub>N<sub>3</sub>O<sub>8</sub>: 616.4 (M+1).

#### Ala7 (Mixture of Two Diastereomers)



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.02-6.69 (m, 14H), 5.83 (t, *J* = 8.7 Hz, 0.83H), 5.54 (t, *J* = 8.7 Hz, 0.17H), 4.58 (q, *J* = 7.0 Hz, 1H), 4.45 (t, *J* = 10.1 Hz, 1H), 4.18-3.56 (m, 10H), 3.40-2.65 (m, 2H), 1.99 (t, *J* = 5.6 Hz, 2H), 1.42 (d, *J* = 7.1 Hz, 3H), 1.16 (t, *J* = 7.1 Hz, 3H); LRMS (ES<sup>+</sup>) for C<sub>34</sub>H<sub>37</sub>N<sub>3</sub>O<sub>7</sub>: 600.3 (M+1).

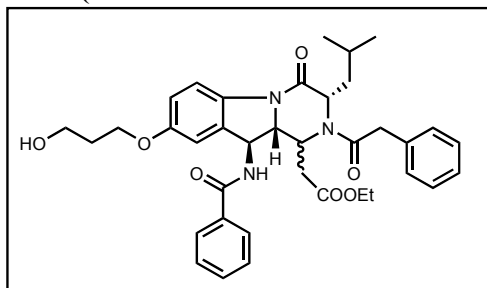
#### Val2 (Mixture of Two Diastereomers)



<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 8.11 (d, *J* = 8.8 Hz, 0.22H), 7.90 (d, *J* = 8.8 Hz, 0.78H), 7.30-7.13 (m, 4H), 6.99-6.79 (m, 2H), 6.71 (s, 1H), 6.05 (d, *J* =

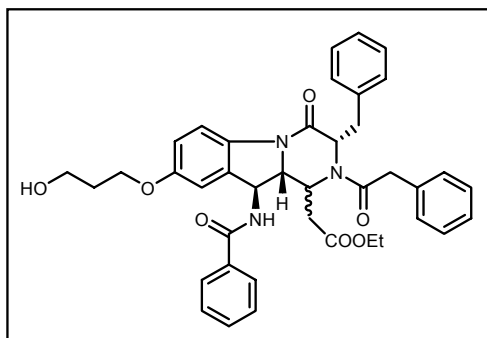
9.2 Hz, 0.78H), 5.94 (d,  $J = 9.2$  Hz, 0.22H), 5.59 (t,  $J = 7.1$  Hz, 0.78H), 5.33 (t,  $J = 7.1$  Hz, 0.22H), 4.21-3.83 (m, 10H), 3.54 (bs, 1H), 3.06-2.34 (m, 7H), 2.02 (m, 2H), 1.29-0.96 (m, 15H); LRMS ( $\text{ES}^+$ ) for  $\text{C}_{34}\text{H}_{45}\text{N}_3\text{O}_7$ : 608.2 ( $\text{M}+1$ ).

#### Leu8 (Mixture of Two Diastereomers)



$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03-6.69 (m, 14H), 5.80 (t,  $J = 8.7$  Hz, 0.75H), 5.52 (t,  $J = 8.7$  Hz, 0.25H), 4.55-3.60 (m, 12H), 3.32-2.73 (m, 2H), 2.09-1.88 (m, 5H), 1.38-0.95 (m, 9H); LRMS ( $\text{ES}^+$ ) for  $\text{C}_{37}\text{H}_{43}\text{N}_3\text{O}_7$ : 642.4 ( $\text{M}+1$ ).

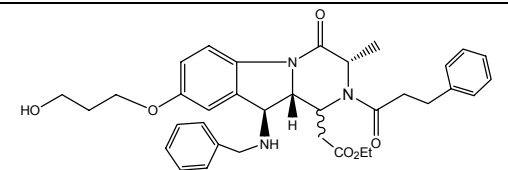
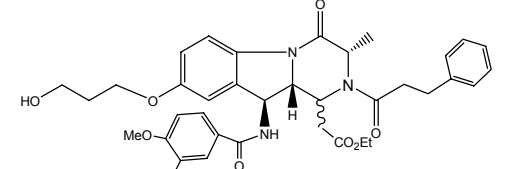
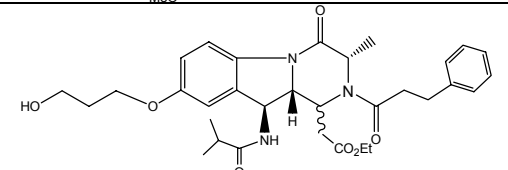
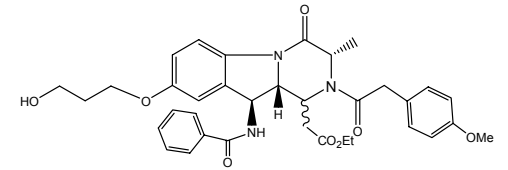
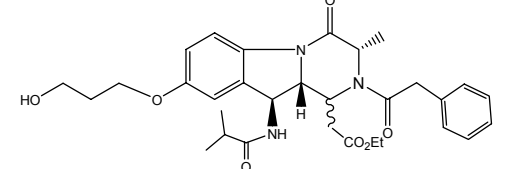
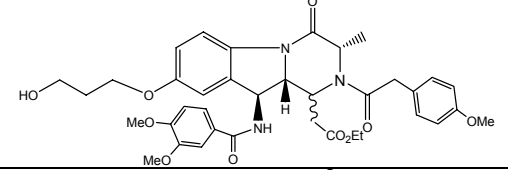
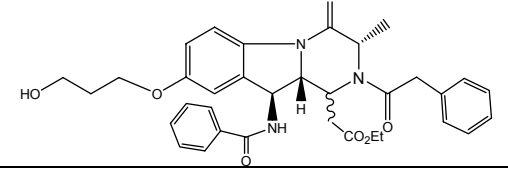
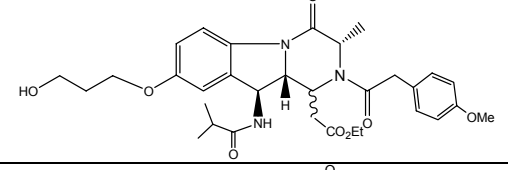
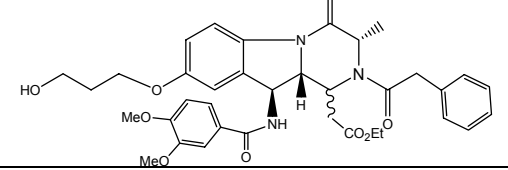
#### Phe5 (Mixture of Two Diastereomers)



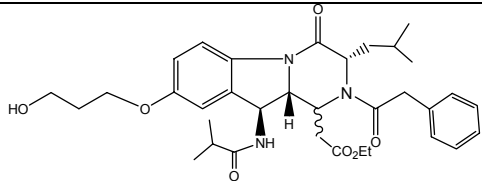
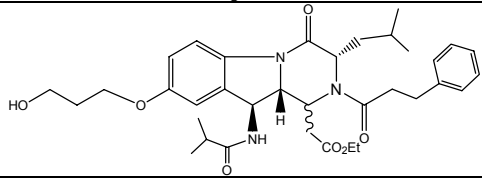
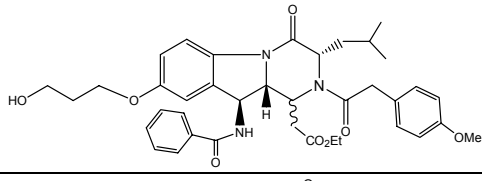
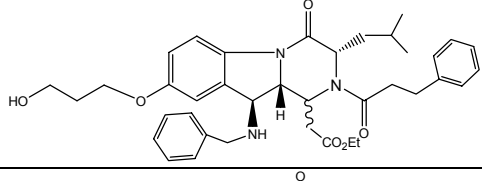
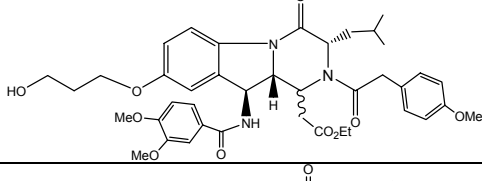
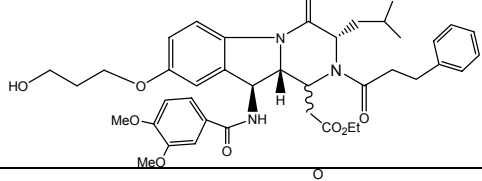
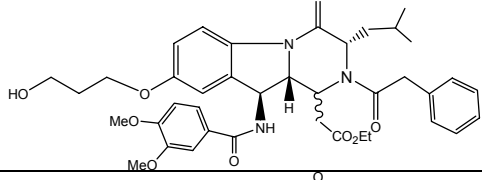
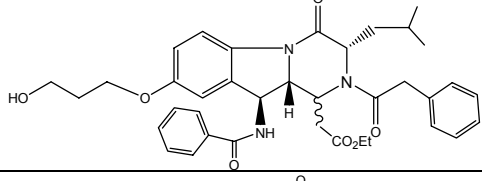
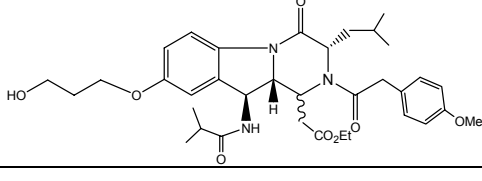
$^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.03-6.64 (m, 19H), 5.88 (t,  $J = 8.4$  Hz, 0.77H), 5.53 ( $J = 8.4$  Hz, 0.23H), 4.57-2.76 (m, 15H), 2.01 (t,  $J = 5.8$  Hz, 2H), 1.15 (t,  $J = 7.1$  Hz, 3H); LRMS ( $\text{ES}^+$ ) for  $\text{C}_{40}\text{H}_{41}\text{N}_3\text{O}_7$ : 676.3 ( $\text{M}+1$ ).

Compound Code	Compound Structure	Molecular Formula	Exact Mass	Found (M <sup>+</sup> +1)
Gly1		C <sub>34</sub> H <sub>37</sub> N <sub>3</sub> O <sub>8</sub>	615.3	616.5
Gly2		C <sub>35</sub> H <sub>39</sub> N <sub>3</sub> O <sub>9</sub>	645.3	646.4
Gly3		C <sub>33</sub> H <sub>35</sub> N <sub>3</sub> O <sub>7</sub>	585.3	586.3
Gly4		C <sub>30</sub> H <sub>37</sub> N <sub>3</sub> O <sub>7</sub>	551.3	552.4
Gly5		C <sub>34</sub> H <sub>37</sub> N <sub>3</sub> O <sub>7</sub>	599.3	600.4
Gly6		C <sub>36</sub> H <sub>41</sub> N <sub>3</sub> O <sub>10</sub>	675.3	676.4
Gly7		C <sub>36</sub> H <sub>41</sub> N <sub>3</sub> O <sub>9</sub>	659.3	660.4
Gly8		C <sub>31</sub> H <sub>39</sub> N <sub>3</sub> O <sub>7</sub>	565.3	566.4
Gly9		C <sub>31</sub> H <sub>39</sub> N <sub>3</sub> O <sub>8</sub>	581.3	582.4

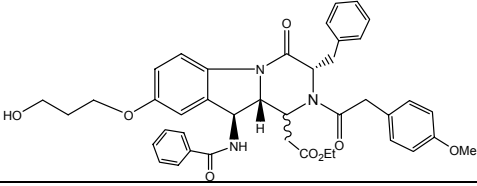


<b>Ala1</b>		$C_{35}H_{39}N_3O_7$	613.3	614.4
<b>Ala2</b>		$C_{37}H_{43}N_3O_9$	673.3	674.4
<b>Ala3</b>		$C_{32}H_{41}N_3O_7$	579.3	580.4
<b>Ala4</b>		$C_{35}H_{39}N_3O_8$	629.3	630.4
<b>Ala5</b>		$C_{31}H_{39}N_3O_7$	565.3	566.4
<b>Ala6</b>		$C_{37}H_{43}N_3O_{10}$	689.3	690.4
<b>Ala7</b>		$C_{34}H_{37}N_3O_7$	599.3	600.4
<b>Ala8</b>		$C_{32}H_{41}N_3O_8$	595.3	596.4
<b>Ala9</b>		$C_{36}H_{41}N_3O_9$	659.3	660.4



<b>Leu1</b>		$C_{34}H_{45}N_3O_7$	607.3	608.4
<b>Leu2</b>		$C_{35}H_{47}N_3O_7$	621.3	622.5
<b>Leu3</b>		$C_{38}H_{45}N_3O_8$	671.3	672.4
<b>Leu4</b>		$C_{38}H_{47}N_3O_6$	641.3	642.4
<b>Leu5</b>		$C_{40}H_{49}N_3O_{10}$	731.3	732.5
<b>Leu6</b>		$C_{40}H_{49}N_3O_9$	715.3	716.4
<b>Leu7</b>		$C_{39}H_{47}N_3O_9$	701.3	702.4
<b>Leu8</b>		$C_{37}H_{43}N_3O_7$	641.3	642.4
<b>Leu9</b>		$C_{35}H_{47}N_3O_8$	637.3	638.5

<b>Phe1</b>		$C_{41}H_{45}N_3O_6$	675.3	676.4
<b>Phe2</b>		$C_{38}H_{45}N_3O_7$	655.3	656.5
<b>Phe3</b>		$C_{43}H_{47}N_3O_9$	749.3	750.5
<b>Phe4</b>		$C_{42}H_{45}N_3O_9$	735.3	736.4
<b>Phe5</b>		$C_{40}H_{41}N_3O_7$	675.3	676.4
<b>Phe6</b>		$C_{43}H_{47}N_3O_{10}$	765.3	766.3
<b>Phe7</b>		$C_{37}H_{43}N_3O_7$	641.3	642.5
<b>Phe8</b>		$C_{38}H_{45}N_3O_8$	671.3	672.5

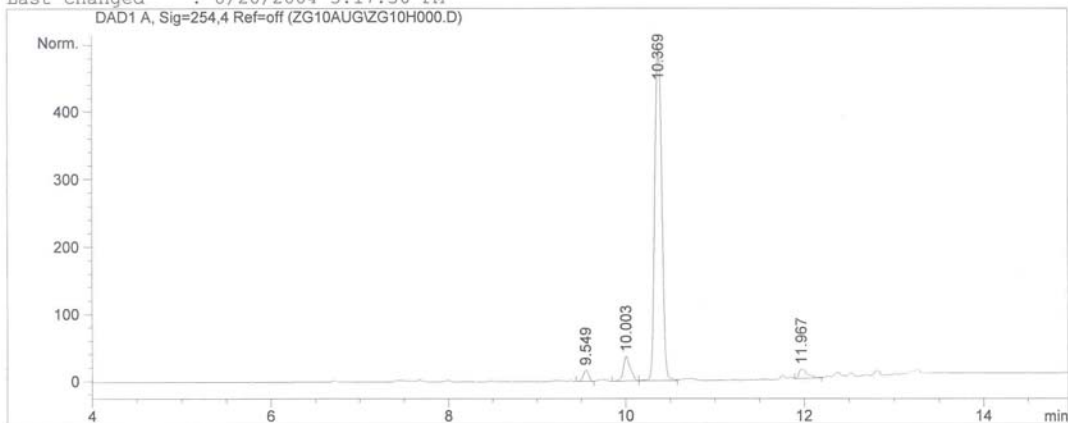
<b>Phe9</b>		$C_{41}H_{43}N_3O_8$	705.3	706.4
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Zorbax SB-C18 60A, 4.6x50 mm, 3.5um  
1-50% MeCN/water/0.1% TFA in 10 min,  
1mL/min

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Injection Date	: 8/10/2004 2:29:43 PM	Location	: P1-A-01
Sample Name	: Gly 1	Inj Volume	: 1 µl
Acq. Operator	: Malgosia		
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 2:29:43 PM by Malgosia		
	(modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:17:36 PM		

DAD1 A, Sig=254,4 Ref=off (ZG10AUG\ZG10H000.D)



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Area Percent Report

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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.549	VV	0.0639	70.02956	17.06966	2.2600
2	10.003	VV	0.0774	199.65639	36.75245	6.4433
3	10.369	VV	0.0887	2730.58081	492.14438	88.1206
4	11.967	VB	0.0920	98.41796	14.71386	3.1761

Totals : 3098.68472 560.68034

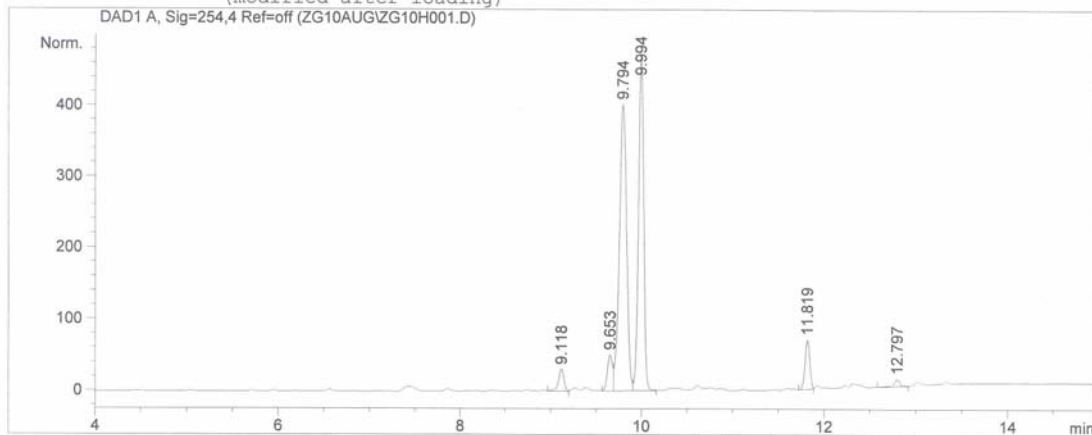
Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 3:44:27 PM      Seq. Line :    2
Sample Name      : Gly 2                    Location  : P1-A-02
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:33:50 PM by Malgosia
                  (modified after loading)
=====
```



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                        Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.118	VV	0.0654	129.58182	30.62739	2.7325
2	9.653	BV	0.0595	196.92184	50.44310	4.1525
3	9.794	VV	0.0866	2231.70361	403.03238	47.0601
4	9.994	VB	0.0623	1884.28503	475.03201	39.7341
5	11.819	VV	0.0580	248.96083	69.20084	5.2499
6	12.797	VV	0.0668	50.78490	10.80781	1.0709

Totals : 4742.23804 1039.14352

Results obtained with enhanced integrator!

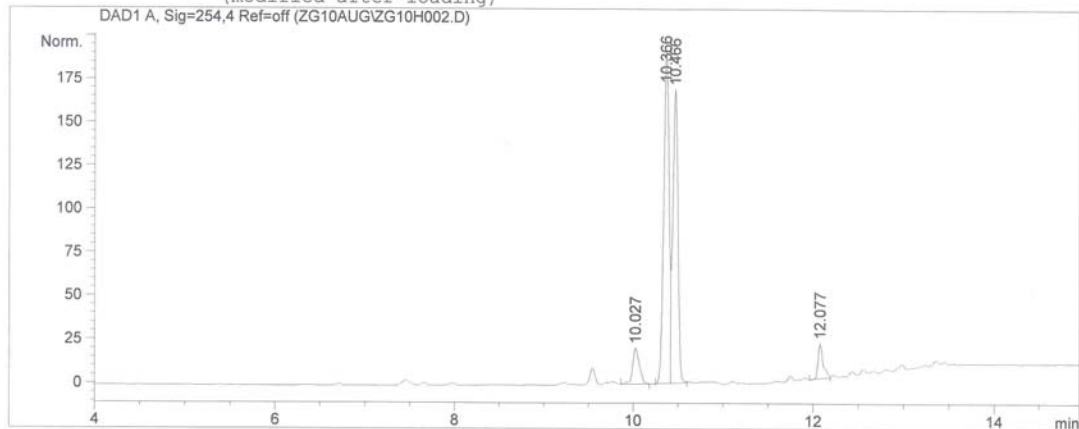
```
=====
*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/10/2004 4:08:32 PM	Seq. Line	: 3
Sample Name	: Gly 3	Location	: Pl-A-03
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:34:11 PM by Malgosia (modified after loading)		

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Area Percent Report

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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.027	VB	0.0774	108.38375	20.58184	6.5174
2	10.366	PV	0.0647	797.32721	190.96729	47.9457
3	10.466	VB	0.0628	678.94470	169.09985	40.8270
4	12.077	VV	0.0577	78.32435	19.99950	4.7099

Totals : 1662.98001 400.64848

Results obtained with enhanced integrator!

=====

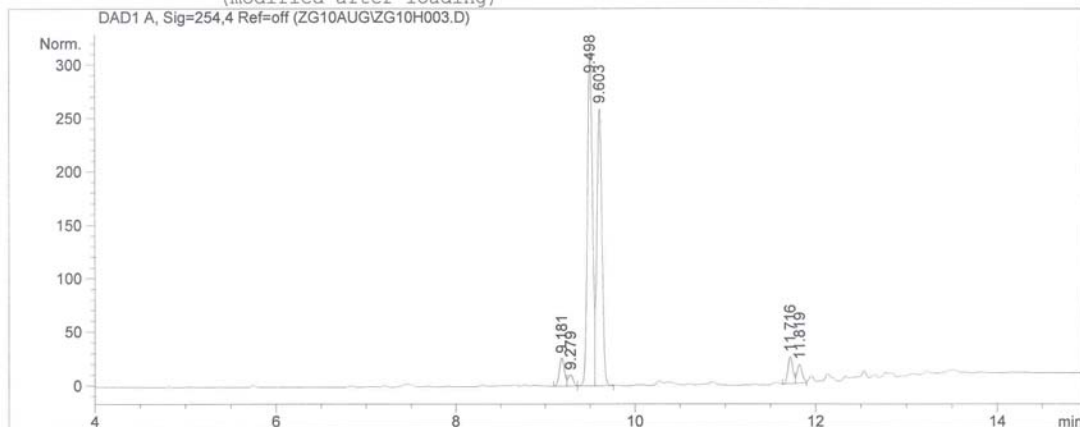
\*\*\* End of Report \*\*\*



Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 4:32:37 PM      Seq. Line :    4
Sample Name      : Gly 4                     Location  : Pl-A-04
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:34:31 PM by Malgosia
                  (modified after loading)
=====
```



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=====
Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.181	PV	0.0626	103.88903	26.03044	4.0699
2	9.279	VV	0.0591	40.90902	10.57800	1.6026
3	9.498	VV	0.0597	1227.06641	312.91394	48.0708
4	9.603	VB	0.0598	1018.12238	259.61212	39.8853
5	11.716	VV	0.0577	93.45545	24.94477	3.6612
6	11.819	VV	0.0601	69.18102	17.51953	2.7102

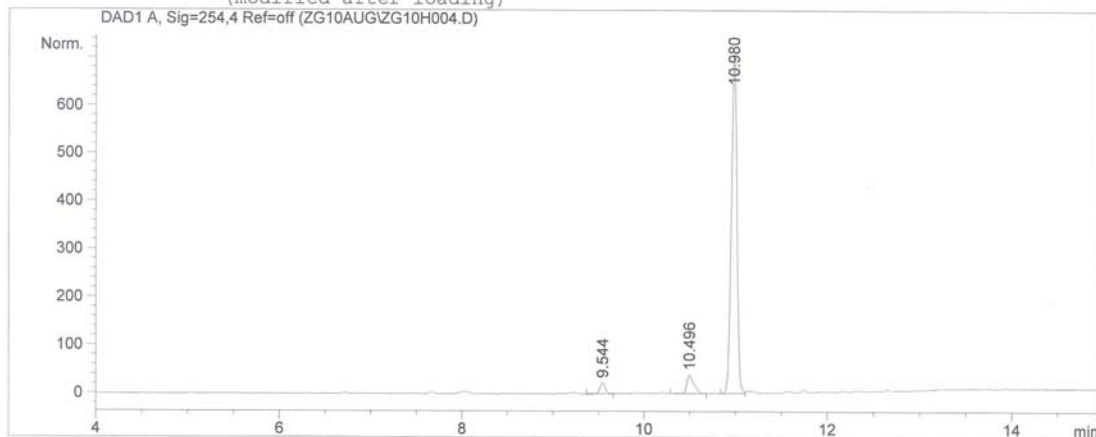
Totals : 2552.62329 651.59880

Results obtained with enhanced integrator!

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*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 4:56:41 PM      Seq. Line :    5
Sample Name      : Gly 5                    Location  : Pl-A-05
Acq. Operator    : Malgosia                 Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:34:53 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.544	VV	0.0660	100.20441	23.39717	3.0328
2	10.496	VP	0.0767	207.99893	38.70428	6.2953
3	10.980	VV	0.0673	2995.84570	708.27527	90.6720

Totals : 3304.04905 770.37673

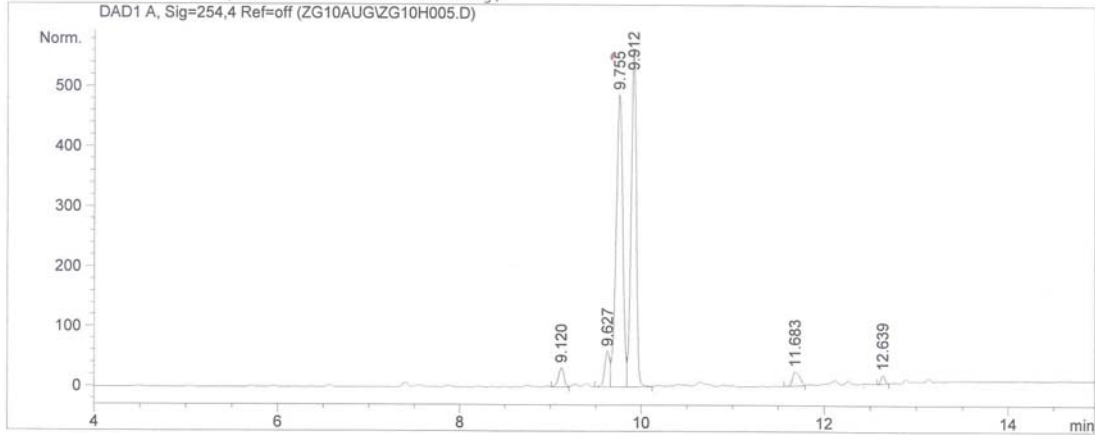
Results obtained with enhanced integrator!

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*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 5:20:48 PM      Seq. Line :    6
Sample Name      : Gly 6                     Location  : P1-A-06
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:15 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
Area Percent Report
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```

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.120	VV	0.0632	137.92001	32.71040	2.6133
2	9.627	VV	0.0589	231.48347	60.09352	4.3862
3	9.755	VV	0.0796	2493.55298	488.37338	47.2482
4	9.912	VB	0.0620	2237.95093	567.36273	42.4050
5	11.683	PV	0.0760	132.09290	24.06330	2.5029
6	12.639	VV	0.0474	44.56087	14.70423	0.8443

Totals : 5277.56116 1187.30756

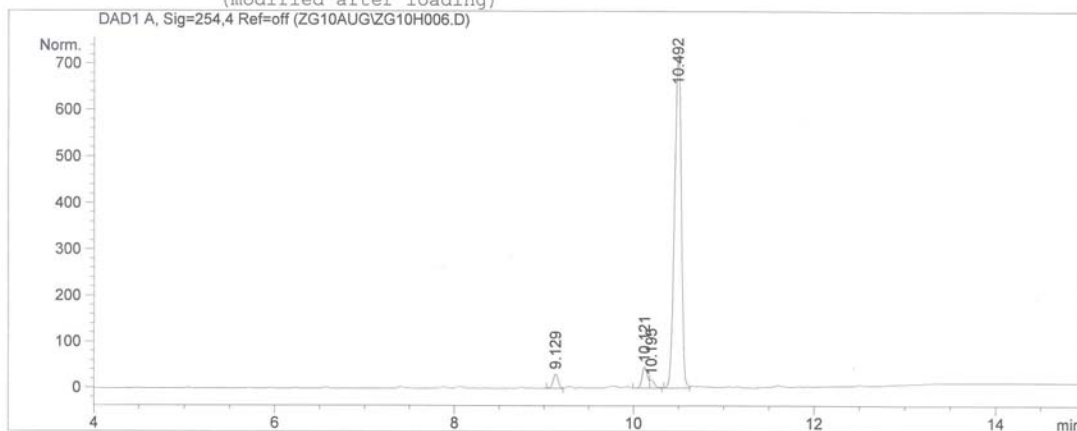
Results obtained with enhanced integrator!

```
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*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 5:44:54 PM      Seq. Line :    7
Sample Name     : Gly 7                    Location  : P1-A-07
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:30 PM by Malgosia
                  (modified after loading)
=====
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                        Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.129	BV	0.0604	125.04546	31.45512	2.9951
2	10.121	VV	0.0652	194.02309	45.98740	4.6473
3	10.195	VB	0.0536	65.19171	18.26244	1.5615
4	10.492	BV	0.0831	3790.74219	724.21503	90.7962

Totals : 4175.00245 819.91998

Results obtained with enhanced integrator!

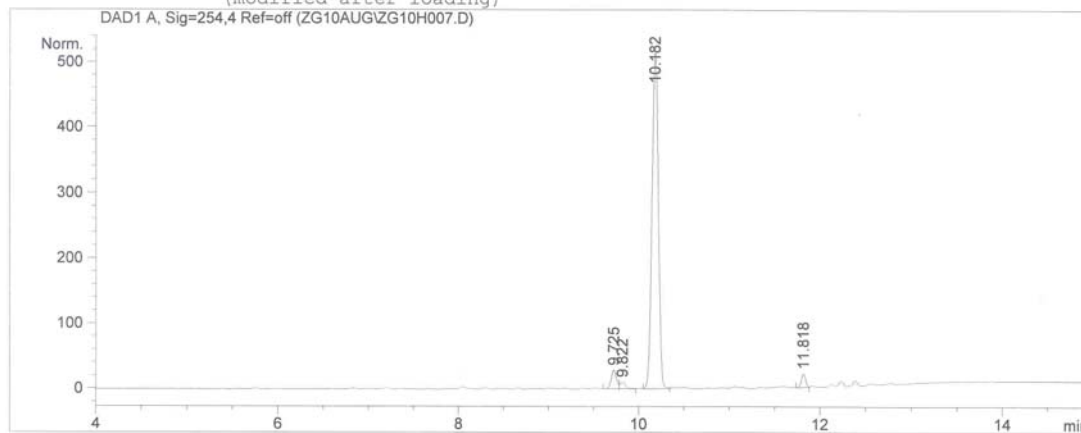
```
=====
                        *** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/10/2004 6:09:00 PM	Seq. Line	: 8
Sample Name	: Gly 8	Location	: Pl-A-08
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

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Area Percent Report

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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.725	VV	0.0642	118.47385	28.69500	4.2902
2	9.822	VB	0.0643	46.83952	10.87446	1.6962
3	10.182	BB	0.0770	2525.45923	516.80145	91.4519
4	11.818	VV	0.0533	70.74353	21.00987	2.5618

Totals : 2761.51614 577.38078

Results obtained with enhanced integrator!

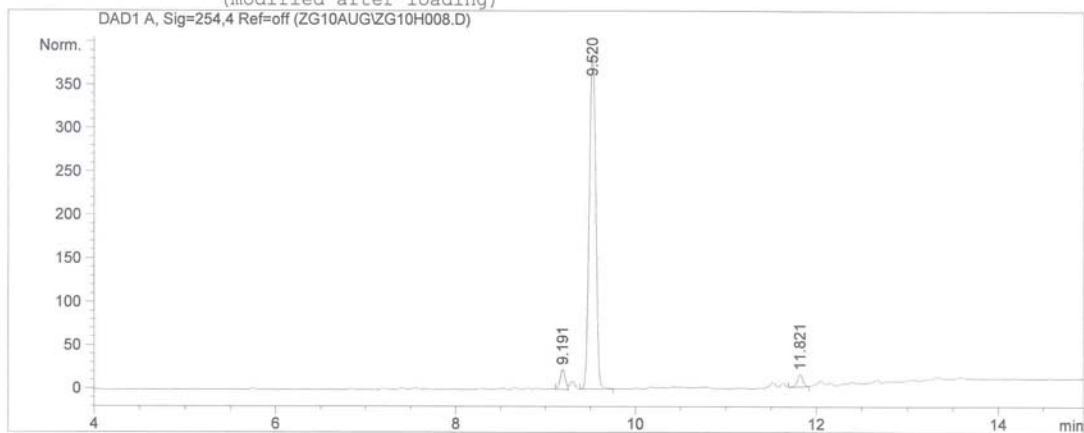
=====

\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 6:33:06 PM      Seq. Line :    9
Sample Name      : Gly 9                    Location  : P1-A-09
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.191	BV	0.0612	88.64692	22.88506	4.2029
2	9.520	VB	0.0812	1958.55115	386.16025	92.8583
3	11.821	VV	0.0669	61.98492	14.22009	2.9388

Totals :                      2109.18299   423.26540

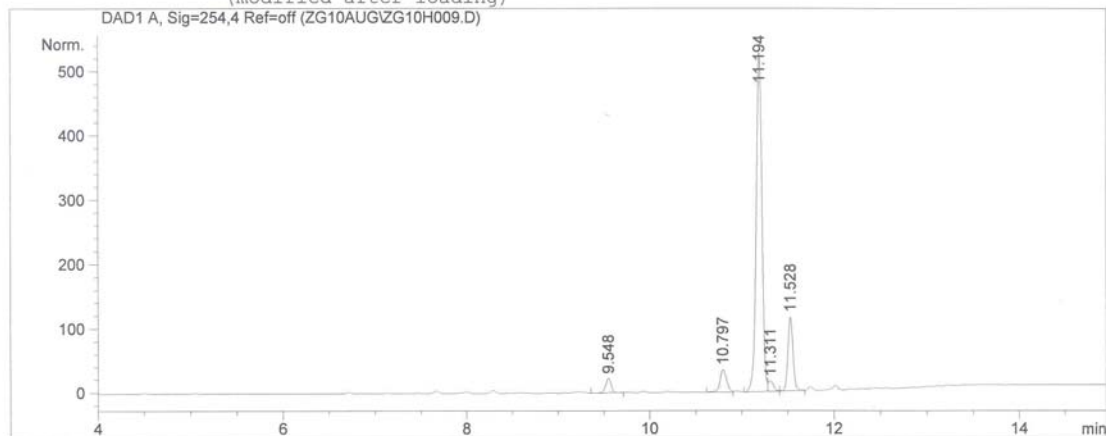
Results obtained with enhanced integrator!

```
=====
*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 6:57:09 PM      Seq. Line :   10
Sample Name      : Ala 1                    Location  : P1-B-01
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.548	VB	0.0653	96.76212	22.88712	3.1432
2	10.797	BV	0.0749	177.50027	35.15320	5.7659
3	11.194	VV	0.0667	2295.42456	528.72522	74.5638
4	11.311	VV	0.0585	63.09015	16.53822	2.0494
5	11.528	VV	0.0589	445.69263	115.81772	14.4777

Totals : 3078.46973 719.12148

Results obtained with enhanced integrator!

=====  
\*\*\* End of Report \*\*\*

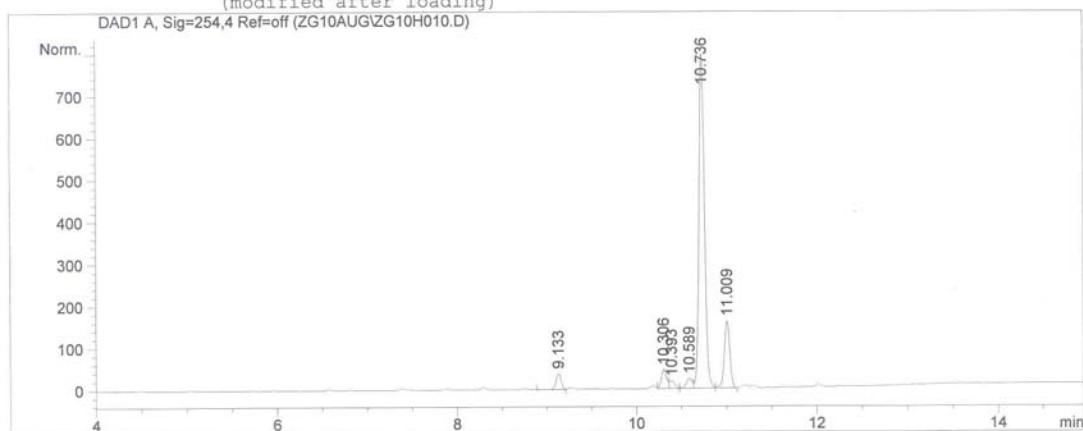


Zorbax SB-C18, 4.6x50mm, 3.5um  
grad. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/10/2004 7:21:13 PM	Seq. Line	: 11
Sample Name	: Ala 2	Location	: P1-B-02
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

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Area Percent Report

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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.133	VV	0.0607	155.26125	38.82611	3.3820
2	10.306	VV	0.0626	191.05287	45.85803	4.1616
3	10.393	VV	0.0598	72.89963	18.55335	1.5879
4	10.589	VV	0.0686	109.58826	24.32212	2.3871
5	10.736	VV	0.0653	3385.40723	800.68982	73.7426
6	11.009	VV	0.0650	676.63678	161.12260	14.7388

Totals : 4590.84602 1089.37203

Results obtained with enhanced integrator!

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\*\*\* End of Report \*\*\*

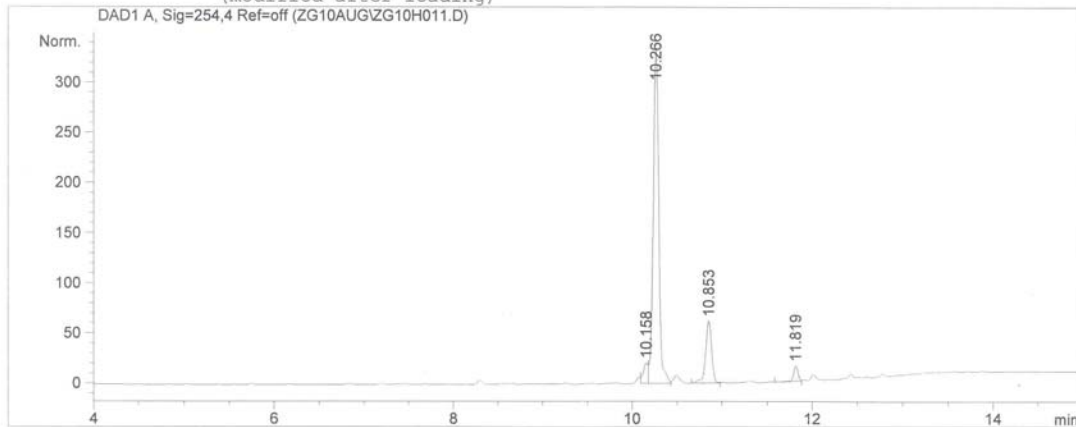


Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/10/2004 7:45:17 PM	Seq. Line	: 12
Sample Name	: Ala 3	Location	: P1-B-03
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

=====



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Area Percent Report

=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.158	VV	0.0545	75.46846	20.71795	4.0593
2	10.266	VV	0.0662	1440.06873	334.78223	77.4576
3	10.853	BB	0.0730	291.49664	61.86292	15.6789
4	11.819	VV	0.0522	52.13573	15.13500	2.8042

Totals : 1859.16956 432.49810

Results obtained with enhanced integrator!

=====

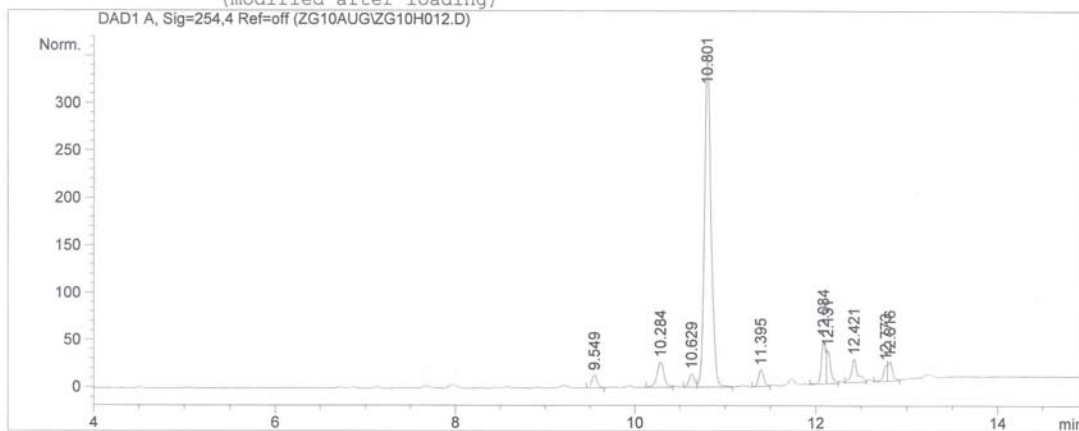
\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```

=====
Injection Date   : 8/10/2004 8:09:24 PM      Seq. Line :   13
Sample Name     : Ala 4                     Location  : Pl-B-04
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.549	BB	0.0643	56.22199	13.57302	2.1431
2	10.284	BB	0.0839	147.95401	27.03072	5.6397
3	10.629	BV	0.0659	60.81625	14.23206	2.3182
4	10.801	VB	0.0748	1780.04285	353.17493	67.8515
5	11.395	VV	0.0641	71.59211	17.37978	2.7289
6	12.084	BV	0.0481	150.30034	46.01387	5.7291
7	12.131	VB	0.0467	109.52878	34.84853	4.1750
8	12.421	BV	0.0697	121.79704	24.62722	4.6426
9	12.773	VV	0.0457	52.23829	17.15676	1.9912
10	12.816	VB	0.0536	72.94880	20.44430	2.7807

Totals :                    2623.44044   568.48120

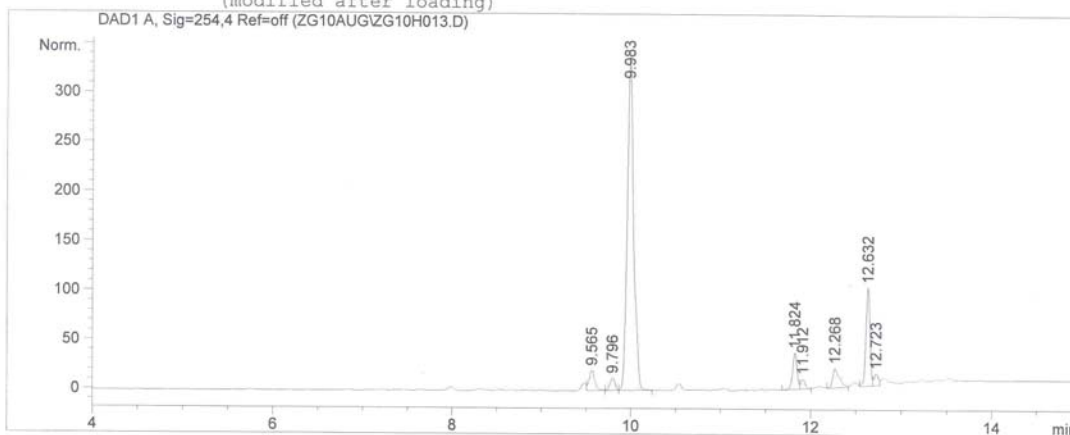
Results obtained with enhanced integrator!

=====  
 \*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 8:33:31 PM      Seq. Line :   14
Sample Name      : Ala 5                    Location  : P1-B-05
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By          :      Signal
Multiplier          :      1.0000
Dilution            :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.565	VV	0.0648	90.73383	20.84722	3.7204
2	9.796	VV	0.0626	50.88973	12.72773	2.0867
3	9.983	VP	0.0737	1680.13574	339.54532	68.8922
4	11.824	VV	0.0546	128.86581	37.04315	5.2840
5	11.912	VV	0.0531	36.07967	10.23720	1.4794
6	12.268	VV	0.0721	99.81856	19.36242	4.0930
7	12.632	VV	0.0503	310.76953	99.86330	12.7428
8	12.723	VV	0.0515	41.49717	12.26344	1.7015

Totals : 2438.79004 551.88978

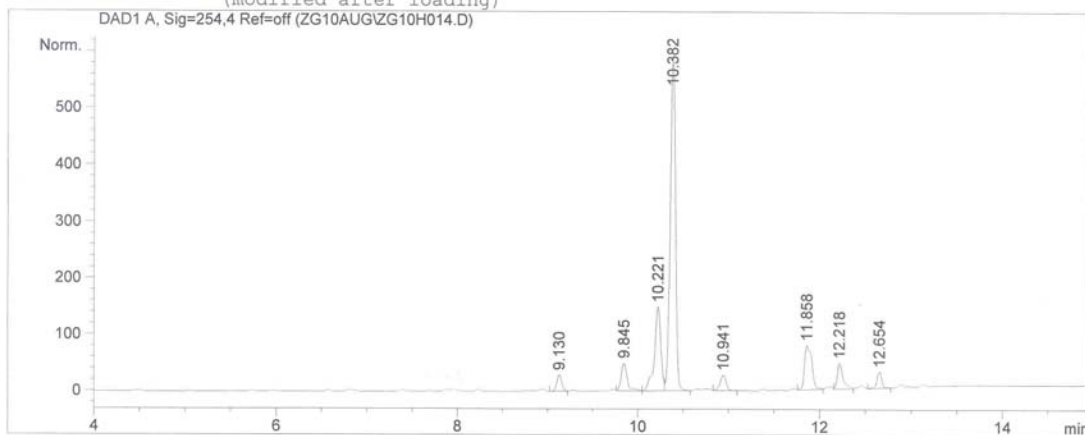
Results obtained with enhanced integrator!

```
=====
*** End of Report ***
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 8:57:38 PM      Seq. Line :   15
Sample Name     : Ala 6                     Location  : P1-B-06
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                          Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.130	VV	0.0610	112.36643	29.12068	2.5855
2	9.845	VV	0.0706	231.83678	49.55153	5.3346
3	10.221	VV	0.0732	731.78845	149.21597	16.8384
4	10.382	VV	0.0629	2390.20923	594.29803	54.9987
5	10.941	VP	0.0719	125.83982	27.22087	2.8956
6	11.858	VV	0.0780	446.22485	78.92790	10.2676
7	12.218	VV	0.0654	202.42863	45.99020	4.6579
8	12.654	VV	0.0564	105.24555	28.96079	2.4217

Totals : 4345.93975 1003.28598

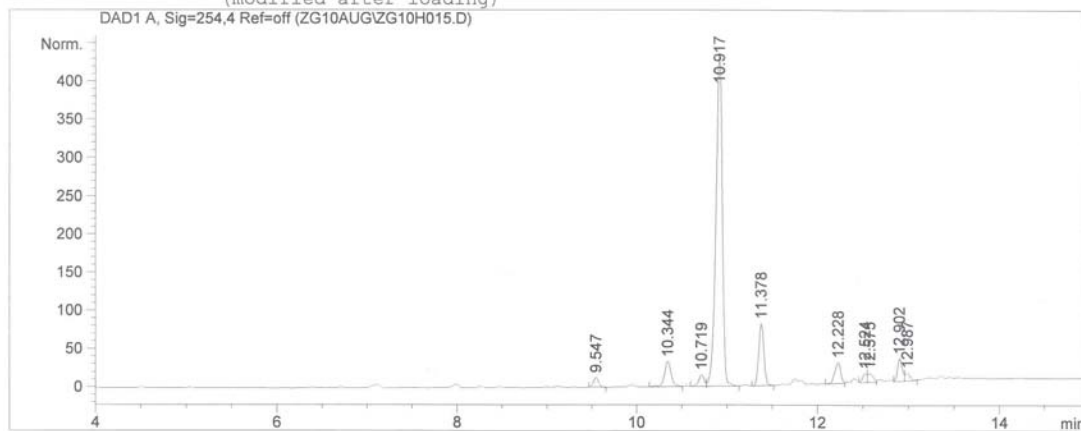
Results obtained with enhanced integrator!

```
=====
*** End of Report ***
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 9:21:43 PM      Seq. Line :   16
Sample Name     : Ala 7                     Location  : P1-B-07
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



=====  
 Area Percent Report  
 =====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.547	BP	0.0625	51.43306	12.90779	1.6512
2	10.344	BV	0.0815	173.84550	32.95494	5.5810
3	10.719	VV	0.0642	67.01646	15.58866	2.1515
4	10.917	VB	0.0728	2136.90405	438.74176	68.6020
5	11.378	VB	0.0624	323.25006	81.19262	10.3774
6	12.228	VV	0.0694	124.60268	27.24085	4.0002
7	12.524	VV	0.0518	41.00568	12.01957	1.3164
8	12.575	VV	0.0617	45.96645	11.23927	1.4757
9	12.902	VV	0.0565	107.95164	29.60454	3.4656
10	12.987	VB	0.0555	42.95488	11.00528	1.3790

Totals : 3114.93046 672.49528

Results obtained with enhanced integrator!

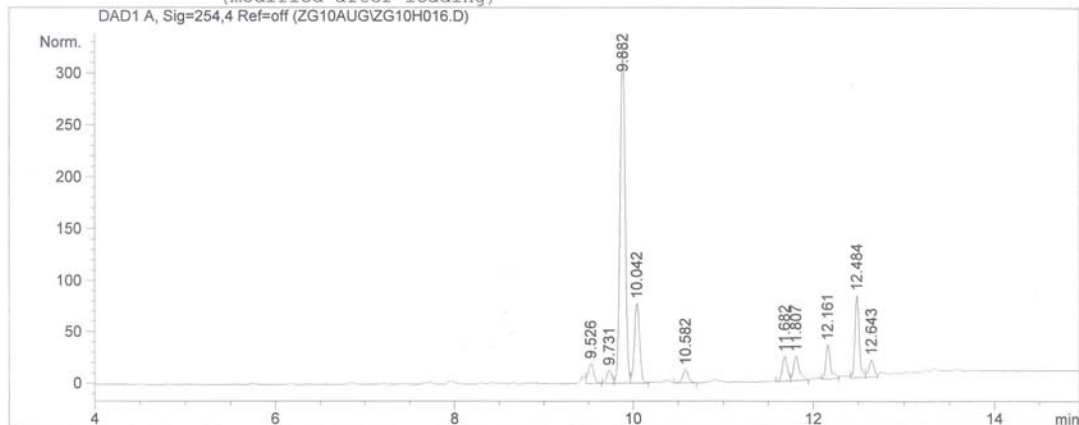
=====  
 \*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/10/2004 9:45:48 PM	Seq. Line	: 17
Sample Name	: Ala 8	Location	: P1-B-08
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

=====



=====

Area Percent Report

=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.526	VB	0.0664	84.82645	19.64726	3.4584
2	9.731	BV	0.0611	51.95520	12.86605	2.1183
3	9.882	VV	0.0626	1293.51672	323.49792	52.7377
4	10.042	VB	0.0635	316.93207	77.86670	12.9216
5	10.582	VB	0.0675	56.48164	12.78326	2.3028
6	11.682	VV	0.0616	93.36176	23.91072	3.8064
7	11.807	VV	0.0653	103.34872	23.51532	4.2136
8	12.161	VV	0.0556	124.11214	33.20705	5.0602
9	12.484	VV	0.0501	262.47272	80.32182	10.7012
10	12.643	VV	0.0580	65.72768	16.66154	2.6798

Totals : 2452.73509 624.27762

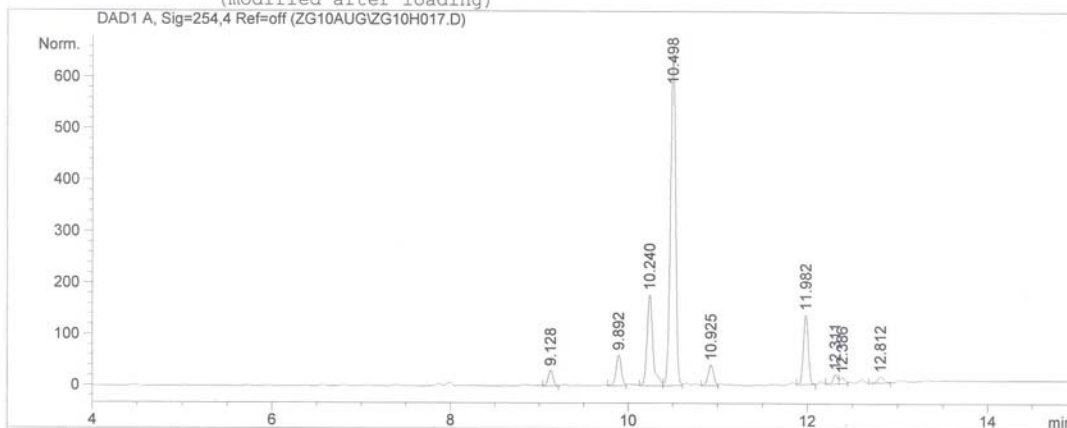
Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 10:09:52 PM      Seq. Line :   18
Sample Name      : Ala 9                      Location  : Pl-B-09
Acq. Operator    : Malgosia                   Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.128	VV	0.0612	117.59017	30.32862	2.5131
2	9.892	VV	0.0640	250.91589	60.92342	5.3625
3	10.240	VV	0.0690	809.04297	177.92598	17.2908
4	10.498	VV	0.0632	2625.24072	648.28192	56.1063
5	10.925	VV	0.0669	175.90619	40.30844	3.7594
6	11.982	VV	0.0605	519.15149	135.98509	11.0952
7	12.311	VV	0.0563	71.23461	18.75966	1.5224
8	12.386	VV	0.0581	49.01361	12.39562	1.0475
9	12.812	VV	0.0783	60.95170	12.19308	1.3027

Totals : 4679.04736 1137.10183

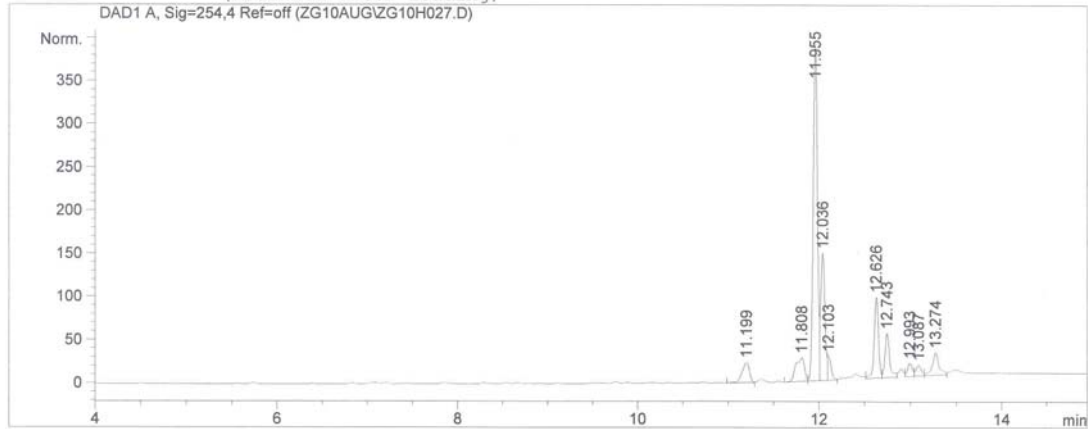
Results obtained with enhanced integrator!

```
=====
*** End of Report ***
```



Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====  
Injection Date : 8/11/2004 2:10:44 AM      Seq. Line : 28  
Sample Name : Leu 1      Location : P1-D-01  
Acq. Operator : Malgosia      Inj : 1  
                                 Inj Volume : 1 µl  
  
Acq. Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/10/2004 3:55:50 PM by Malgosia  
                                 (modified after loading)  
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/20/2004 3:35:49 PM by Malgosia  
                                 (modified after loading)  
=====



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.199	BV	0.0833	132.07397	23.61199	4.6235
2	11.808	VV	0.0917	181.07794	27.17311	6.3389
3	11.955	VV	0.0499	1264.29199	389.60019	44.2586
4	12.036	VV	0.0479	480.03339	147.59726	16.8044
5	12.103	VV	0.0406	71.77789	27.37761	2.5127
6	12.626	VV	0.0496	301.88156	93.74487	10.5679
7	12.743	VV	0.0539	182.85159	50.90263	6.4010
8	12.993	VV	0.0547	56.67868	15.47900	1.9841
9	13.087	VV	0.0608	50.41922	12.56709	1.7650
10	13.274	VV	0.0729	135.51331	25.96420	4.7439

Totals : 2856.59954 814.01794

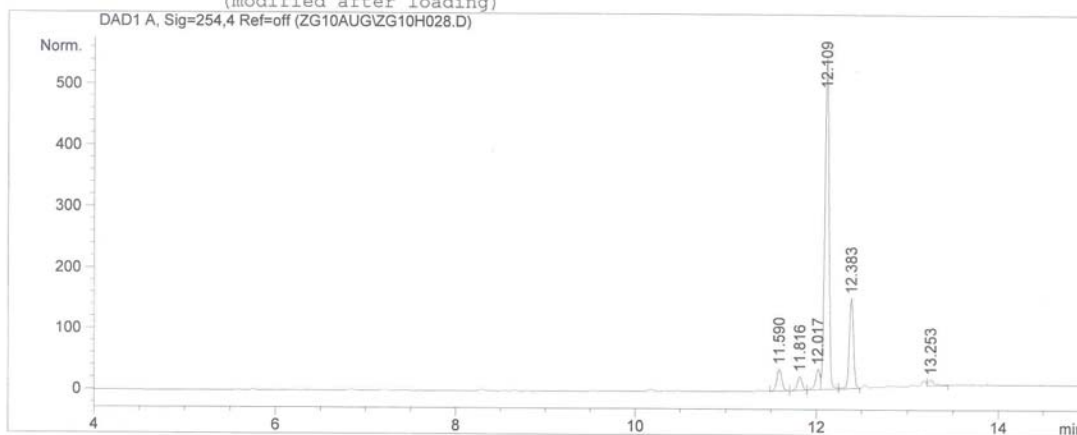
Results obtained with enhanced integrator!

=====  
\*\*\* End of Report \*\*\*



Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====  
Injection Date : 8/11/2004 2:34:51 AM      Seq. Line : 29  
Sample Name : Leu 2      Location : Pl-D-02  
Acq. Operator : Malgosia      Inj : 1  
                                 Inj Volume : 1 µl  
  
Acq. Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/10/2004 3:55:50 PM by Malgosia  
                 (modified after loading)  
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/20/2004 3:35:49 PM by Malgosia  
                 (modified after loading)  
=====



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.590	VV	0.0584	135.74805	35.64218	5.2356
2	11.816	VP	0.0549	78.84872	22.51501	3.0411
3	12.017	VV	0.0527	116.16936	33.30010	4.4805
4	12.109	VV	0.0512	1732.51538	543.88379	66.8208
5	12.383	VV	0.0519	482.50562	148.54759	18.6096
6	13.253	VB	0.0630	46.99184	10.74885	1.8124

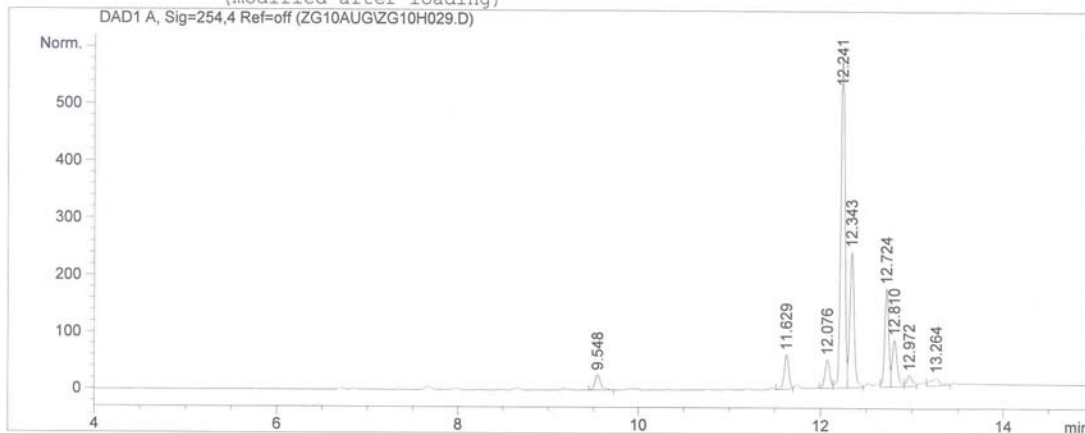
Totals :                                      2592.77897    794.63752

Results obtained with enhanced integrator!

=====  
\*\*\* End of Report \*\*\*  
=====

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 2:58:54 AM      Seq. Line :   30
Sample Name     : Leu 3                    Location  : P1-D-03
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl
Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254.4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.548	BB	0.0677	118.48042	26.73413	2.8160
2	11.629	VV	0.0566	222.91714	61.10519	5.2983
3	12.076	VV	0.0571	187.39674	50.72004	4.4540
4	12.241	VV	0.0484	1841.92310	589.70233	43.7785
5	12.343	VV	0.0533	806.96387	239.68605	19.1798
6	12.724	VV	0.0499	534.29645	173.58124	12.6991
7	12.810	VV	0.0571	307.00146	83.05581	7.2968
8	12.972	VV	0.0659	84.54302	19.00002	2.0094
9	13.264	VV	0.0987	103.84311	13.96640	2.4681

Totals : 4207.36532 1257.55120

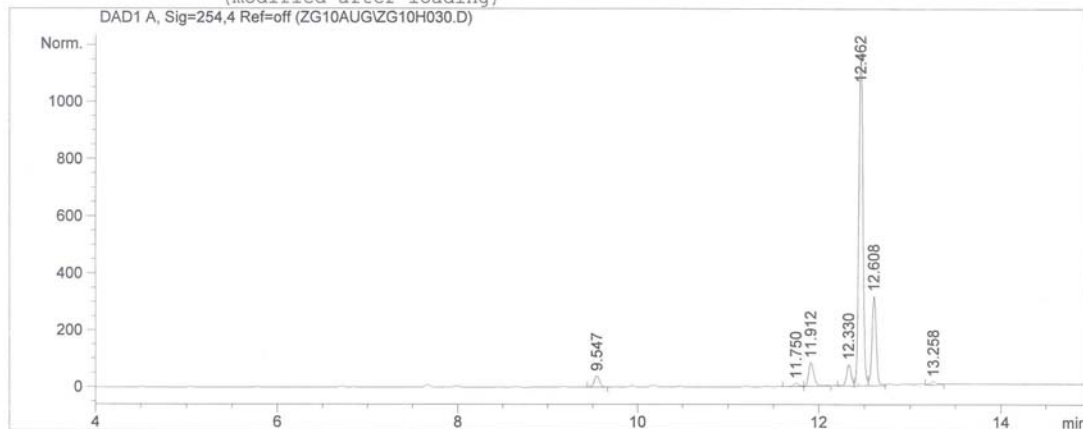
Results obtained with enhanced integrator!

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*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 3:22:59 AM      Seq. Line :   31
Sample Name      : Leu 4                    Location  : P1-D-04
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.547	BB	0.0612	163.15477	40.31819	2.9024
2	11.750	VV	0.0662	53.31617	11.92685	0.9484
3	11.912	VV	0.0659	358.22882	83.76402	6.3725
4	12.330	VV	0.0543	270.41675	74.65365	4.8104
5	12.462	VV	0.0484	3674.85962	1177.24463	65.3721
6	12.608	VV	0.0519	1026.30249	315.86118	18.2569
7	13.258	VV	0.0856	75.17248	11.91678	1.3372

Totals : 5621.45110 1715.68529

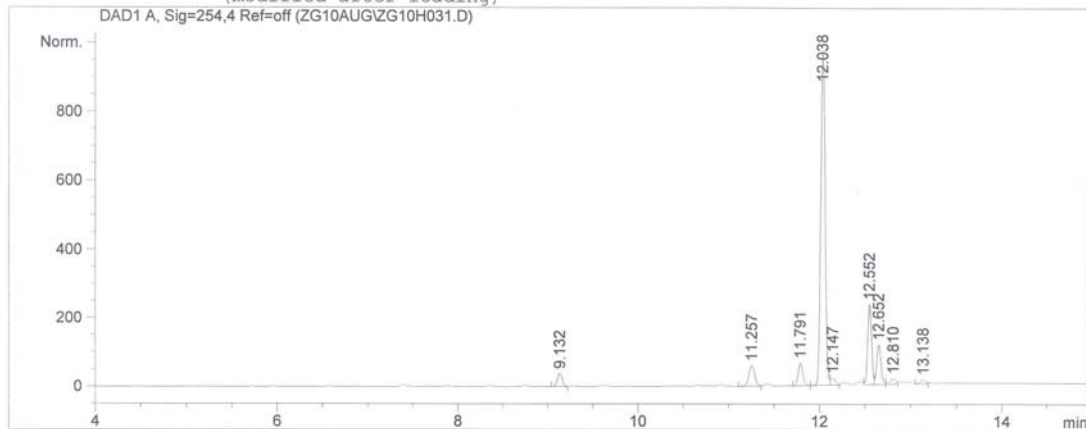
Results obtained with enhanced integrator!

```
=====
*** End of Report ***
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date : 8/11/2004 3:47:06 AM      Seq. Line : 32
Sample Name    : Leu 5                    Location  : P1-D-05
Acq. Operator  : Malgosia                 Inj       : 1
                                           Inj Volume: 1 µl

Acq. Method    : C:\HPCHEM\1\METHODS\ZG01.M
Last changed   : 8/10/2004 3:55:50 PM by Malgosia
                (modified after loading)
Analysis Method: C:\HPCHEM\1\METHODS\ZG01.M
Last changed   : 8/20/2004 3:35:49 PM by Malgosia
                (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.132	BV	0.0609	149.18658	38.72206	2.8592
2	11.257	BV	0.0720	279.38953	60.39709	5.3546
3	11.791	PV	0.0558	237.07278	66.14194	4.5436
4	12.038	VV	0.0532	3282.22900	976.87482	62.9051
5	12.147	VV	0.0535	73.64734	20.71803	1.4115
6	12.552	VV	0.0470	704.42468	234.91624	13.5006
7	12.652	VV	0.0516	372.89740	115.74183	7.1467
8	12.810	VV	0.0628	73.91893	16.96847	1.4167
9	13.138	VV	0.0556	44.97823	12.02390	0.8620

Totals : 5217.74448 1542.50439

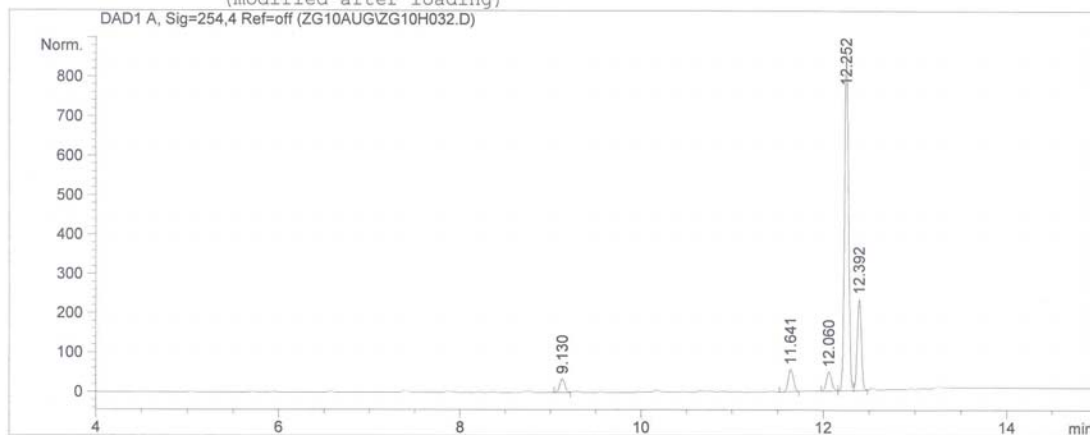
Results obtained with enhanced integrator!

```
=====
*** End of Report ***
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 4:11:10 AM      Seq. Line :   33
Sample Name      : Leu 6                    Location  : Pl-D-06
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.130	BV	0.0616	135.48473	34.66608	3.4359
2	11.641	VV	0.0650	228.11041	56.57514	5.7850
3	12.060	VV	0.0628	193.74406	48.30569	4.9134
4	12.252	VV	0.0480	2659.53564	861.45544	67.4467
5	12.392	VV	0.0483	726.29193	233.22002	18.4190

Totals : 3943.16678 1234.22236

Results obtained with enhanced integrator!

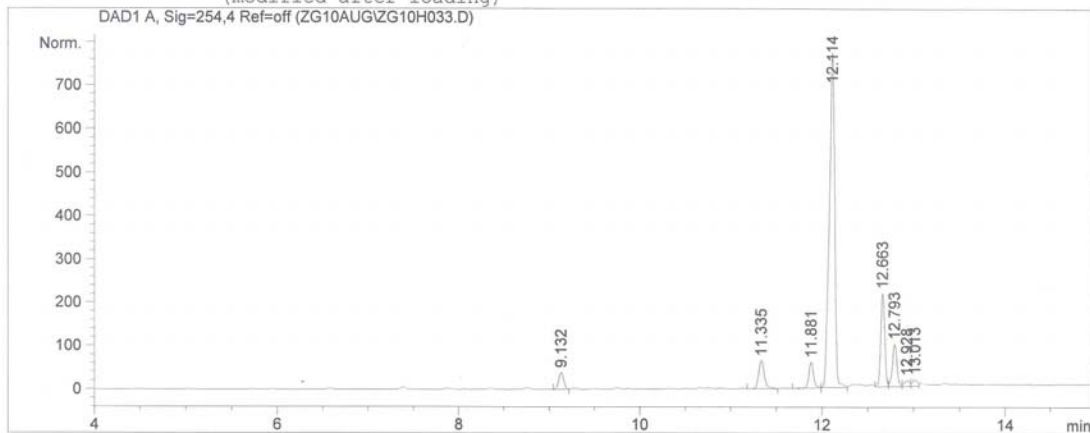
```
=====
*** End of Report ***
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/11/2004 4:35:17 AM	Seq. Line	: 34
Sample Name	: Leu 7	Location	: P1-D-07
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

=====



=====

Area Percent Report

=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.132	BV	0.0613	151.65341	39.09852	3.0911
2	11.335	VP	0.0667	279.59772	64.29184	5.6989
3	11.881	VV	0.0554	210.89502	59.39983	4.2985
4	12.114	VV	0.0616	3162.27002	775.30872	64.4544
5	12.663	VV	0.0493	655.62811	216.61996	13.3632
6	12.793	VV	0.0519	336.58127	98.31401	6.8603
7	12.928	VV	0.0560	53.10948	14.08821	1.0825
8	13.013	VV	0.0532	56.47400	15.99573	1.1511

Totals : 4906.20902 1283.11682

Results obtained with enhanced integrator!

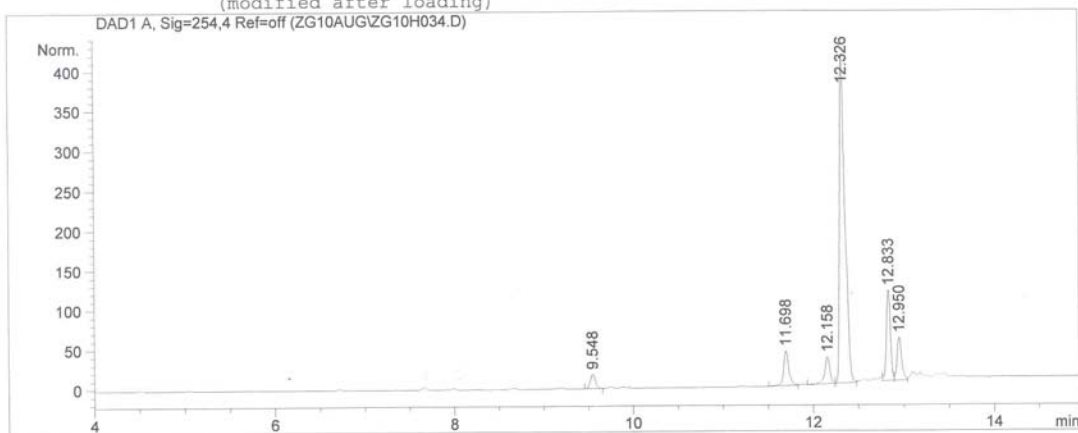
=====

\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 4:59:23 AM      Seq. Line :   35
Sample Name     : Leu 8                    Location  : Pl-D-08
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.548	BB	0.0625	73.24783	18.36710	2.7901
2	11.698	PB	0.0589	175.36244	43.64214	6.6797
3	12.158	VV	0.0596	132.01231	33.80830	5.0285
4	12.326	VV	0.0599	1703.65039	414.89273	64.8934
5	12.833	VV	0.0498	350.18765	114.15073	13.3389
6	12.950	VV	0.0530	190.84584	54.26984	7.2695

Totals : 2625.30647 679.13083

Results obtained with enhanced integrator!

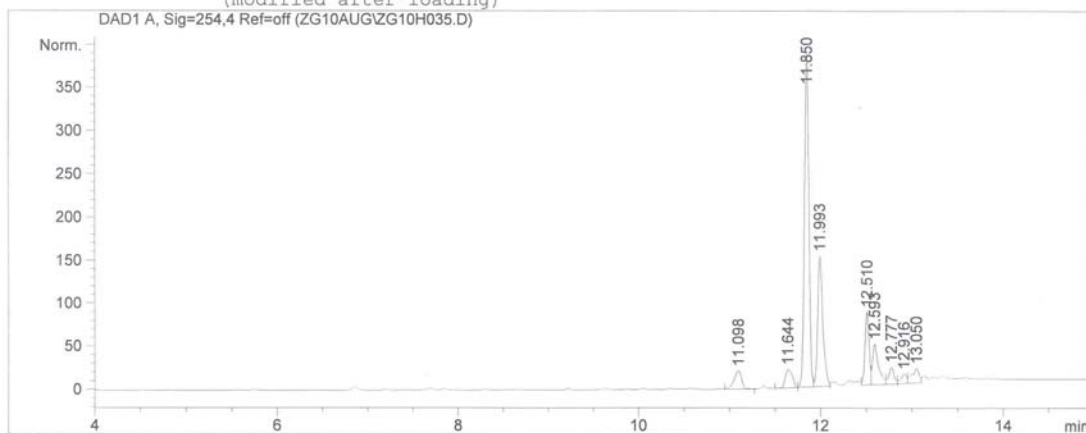
```
=====
*** End of Report ***
```



Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 5:23:30 AM      Seq. Line :   36
Sample Name     : Leu 9                    Location  : Pl-D-09
Acq. Operator   : Malgosia                 Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.098	BP	0.0899	123.30206	21.17747	4.3468
2	11.644	VV	0.0895	114.42693	21.02774	4.0339
3	11.850	VV	0.0536	1311.52673	386.74081	46.2356
4	11.993	VV	0.0573	590.85443	152.03613	20.8296
5	12.510	VV	0.0503	263.19016	84.64189	9.2783
6	12.593	VV	0.0646	210.88676	46.76837	7.4344
7	12.777	VV	0.0611	78.38555	19.42210	2.7633
8	12.916	VV	0.0628	48.20404	11.06828	1.6994
9	13.050	VV	0.0779	95.83968	16.97755	3.3787

Totals : 2836.61634 759.86036

Results obtained with enhanced integrator!

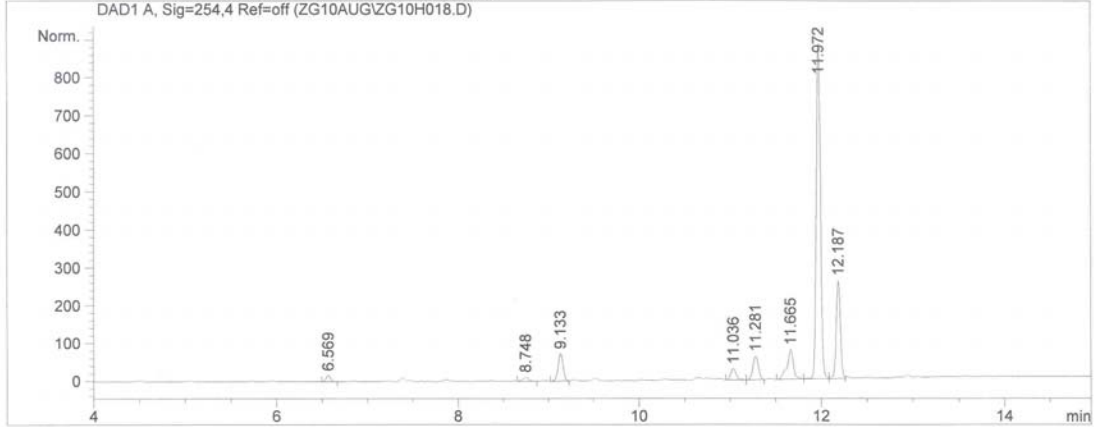
```
=====
*** End of Report ***
```



Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 10:33:56 PM      Seq. Line :   19
Sample Name      : Val 1                      Location  : P1-C-01
Acq. Operator    : Malgosia                   Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



Area Percent Report

```
=====
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
=====
```

Signal 1: DAQ1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.569	BB	0.0531	56.48395	16.84506	1.1680
2	8.748	VV	0.0641	47.04040	10.95423	0.9727
3	9.133	VV	0.0593	282.36530	72.76206	5.8387
4	11.036	VP	0.0639	122.21551	29.78621	2.5272
5	11.281	VV	0.0663	259.69742	62.69995	5.3700
6	11.665	VV	0.0709	383.20380	78.55480	7.9239
7	11.972	VV	0.0516	2855.16455	886.83215	59.0391
8	12.187	VV	0.0511	829.89014	260.98032	17.1605

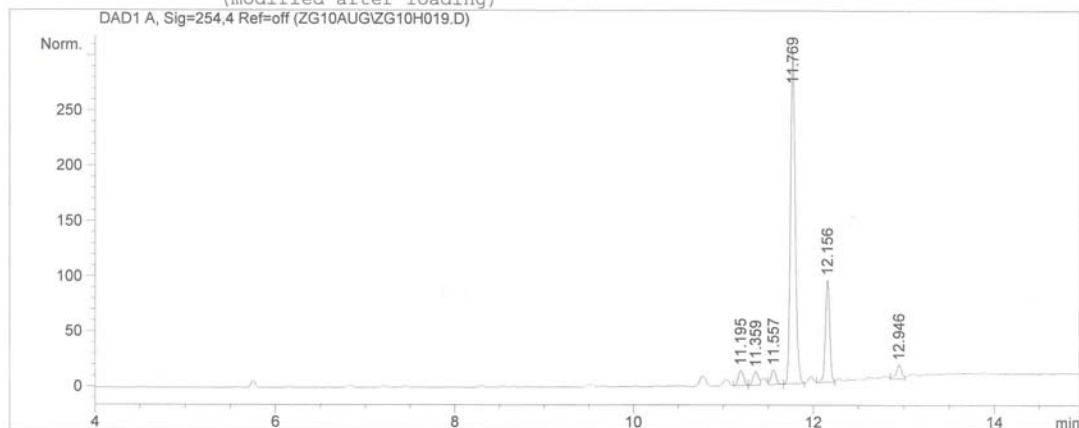
Totals : 4836.06105 1419.41478

Results obtained with enhanced integrator!

\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====  
Injection Date : 8/10/2004 10:58:02 PM Seq. Line : 20  
Sample Name : Val 2 Location : Pl-C-02  
Acq. Operator : Malgosia Inj : 1  
Inj Volume : 1 µl  
Acq. Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/10/2004 3:55:50 PM by Malgosia  
(modified after loading)  
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/20/2004 3:35:49 PM by Malgosia  
(modified after loading)  
=====



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.195	VV	0.0626	56.16360	14.07022	3.4712
2	11.359	VV	0.0620	49.49796	12.02201	3.0593
3	11.557	VP	0.0585	52.04745	13.63344	3.2168
4	11.769	VV	0.0568	1100.23474	299.66940	68.0011
5	12.156	VV	0.0525	306.56320	93.01751	18.9475
6	12.946	VV	0.0612	53.45824	12.67004	3.3040

Totals : 1617.96520 445.08262

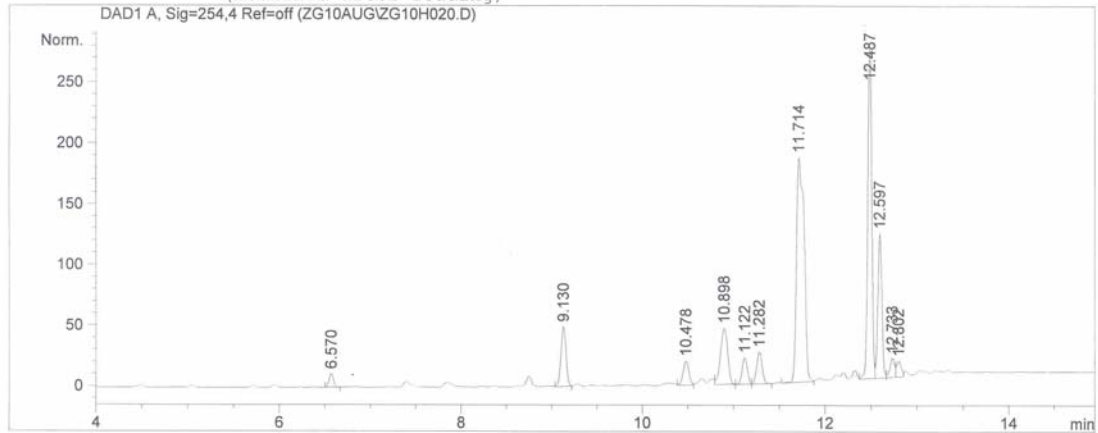
Results obtained with enhanced integrator!

=====  
\*\*\* End of Report \*\*\*  
=====

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 11:22:07 PM      Seq. Line : 21
Sample Name      : Val 3                      Location  : P1-C-03
Acq. Operator    : Malgosia                   Inj       : 1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



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=====
Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.570	BP	0.0527	37.58251	11.32343	1.1973
2	9.130	BV	0.0592	191.29089	49.32756	6.0940
3	10.478	VV	0.0695	88.06834	19.96274	2.8056
4	10.898	VP	0.0859	255.04140	46.57156	8.1250
5	11.122	VV	0.0630	88.28615	21.90060	2.8126
6	11.282	VP	0.0650	110.43542	26.28268	3.5182
7	11.714	VV	0.0795	1072.17175	185.33948	34.1566
8	12.487	VV	0.0472	828.32013	274.44122	26.3881
9	12.597	VV	0.0496	364.24060	119.40856	11.6038
10	12.733	VV	0.0534	56.79278	16.00327	1.8093
11	12.802	VV	0.0509	46.75917	13.35042	1.4896

Totals : 3138.98913 783.91152

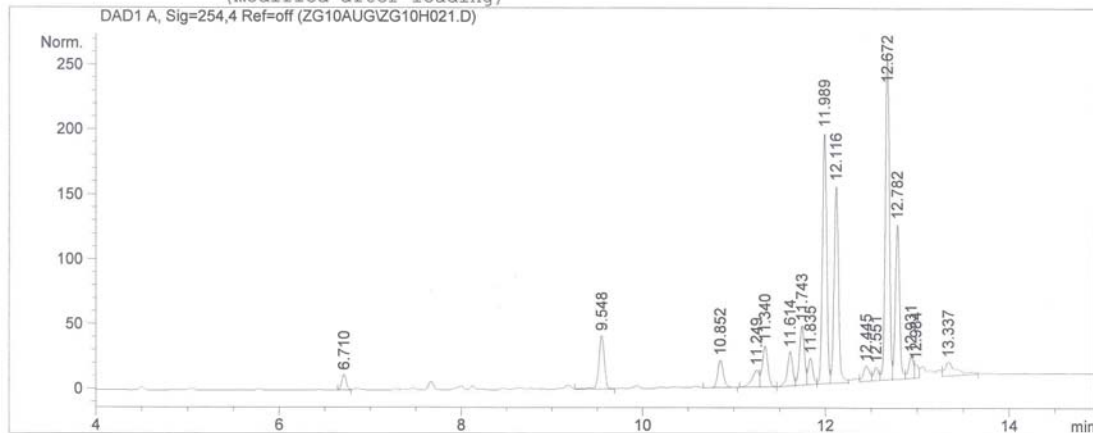
Results obtained with enhanced integrator!

```
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*** End of Report ***
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/10/2004 11:46:13 PM      Seq. Line :   22
Sample Name     : Val 4                      Location  : Pl-C-04
Acq. Operator   : Malgosia                   Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



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=====
Area Percent Report
=====
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```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

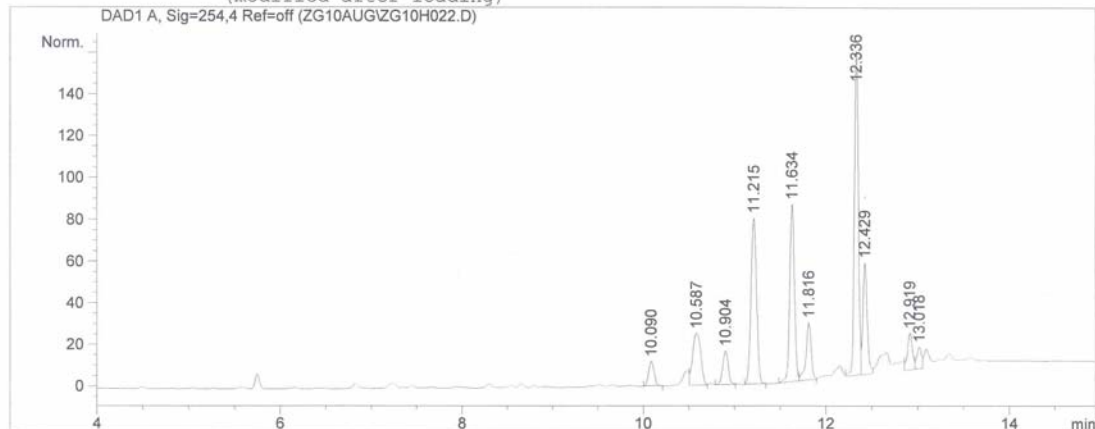
Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.710	BV	0.0534	39.82188	11.79706	1.1629
2	9.548	VB	0.0666	179.90872	41.45250	5.2537
3	10.852	VB	0.0660	90.91088	21.21736	2.6548
4	11.249	BV	0.0769	71.20436	13.20817	2.0793
5	11.340	VP	0.0652	133.49580	31.66972	3.8984
6	11.614	VV	0.0601	104.47399	26.41590	3.0509
7	11.743	VV	0.0575	171.69504	46.00222	5.0138
8	11.835	VV	0.0538	73.62534	20.55972	2.1500
9	11.989	VV	0.0495	624.97247	194.41766	18.2505
10	12.116	VB	0.0509	507.58783	152.35880	14.8226
11	12.445	VV	0.0729	55.26421	11.74286	1.6138
12	12.551	VV	0.0560	38.22228	10.13382	1.1162
13	12.672	VV	0.0494	768.49866	253.14821	22.4417
14	12.782	VV	0.0487	378.33554	120.23834	11.0482
15	12.931	VV	0.0553	62.53636	16.83182	1.8262
16	12.984	VV	0.0405	30.02151	10.77663	0.8767
17	13.337	VB	0.1164	93.84051	10.89988	2.7403

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 12:10:18 AM      Seq. Line :   23
Sample Name      : Val 5                      Location  : P1-C-05
Acq. Operator    : Malgosia                   Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
Area Percent Report
=====
```

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.090	PP	0.0652	49.32955	11.70509	2.7673
2	10.587	VV	0.1001	153.52362	24.85683	8.6124
3	10.904	VB	0.0652	68.27924	16.18014	3.8303
4	11.215	PP	0.0653	336.84558	79.77705	18.8964
5	11.634	PV	0.0563	308.20163	85.09615	17.2896
6	11.816	VV	0.0580	108.92026	27.61339	6.1102
7	12.336	VV	0.0476	477.57327	156.51382	26.7910
8	12.429	VV	0.0502	176.09044	53.80845	9.8784
9	12.919	VV	0.0557	66.55541	17.76235	3.7336
10	13.018	VV	0.0532	37.26761	10.56170	2.0906

Totals : 1782.58661 483.87496

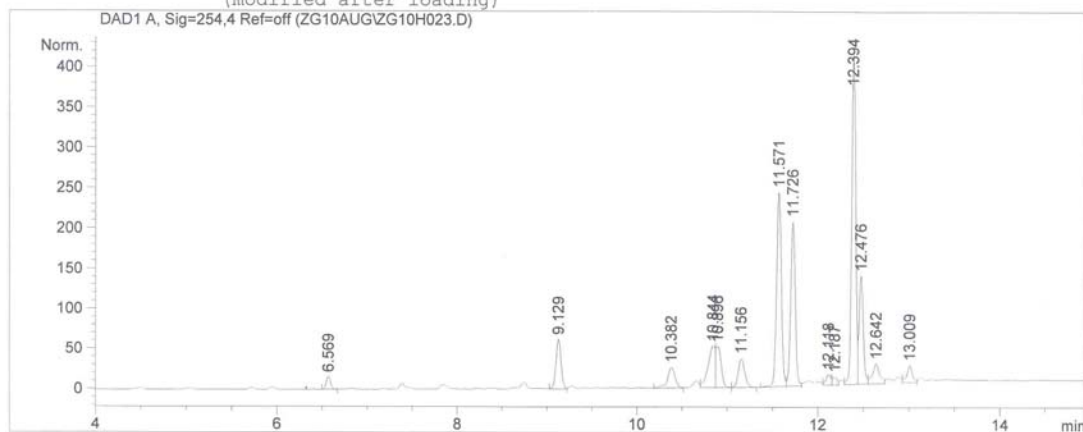
Results obtained with enhanced integrator!

```
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*** End of Report ***
=====
```

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 12:34:25 AM      Seq. Line :   24
Sample Name      : Val 6                      Location  : P1-C-06
Acq. Operator    : Malgosia                   Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



=====  
 Area Percent Report  
 =====

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.569	BB	0.0531	53.01356	15.83399	1.1224
2	9.129	BV	0.0599	243.50172	61.84081	5.1555
3	10.382	BV	0.0876	150.32268	25.93875	3.1827
4	10.844	VV	0.0780	285.22284	52.00510	6.0388
5	10.896	VV	0.0608	203.75783	50.78392	4.3140
6	11.156	VB	0.0763	179.41197	35.91370	3.7985
7	11.571	PV	0.0570	892.69672	242.39584	18.9003
8	11.726	VV	0.0527	717.86365	205.54945	15.1987
9	12.118	VV	0.0561	50.59891	13.39499	1.0713
10	12.187	VV	0.0506	35.38861	10.15692	0.7493
11	12.394	VV	0.0478	1257.16565	410.22025	26.6169
12	12.476	VV	0.0490	428.78204	135.36388	9.0782
13	12.642	VV	0.0775	135.26405	24.85947	2.8638
14	13.009	VV	0.0618	90.19321	21.11224	1.9096

Totals : 4723.18344 1305.36929

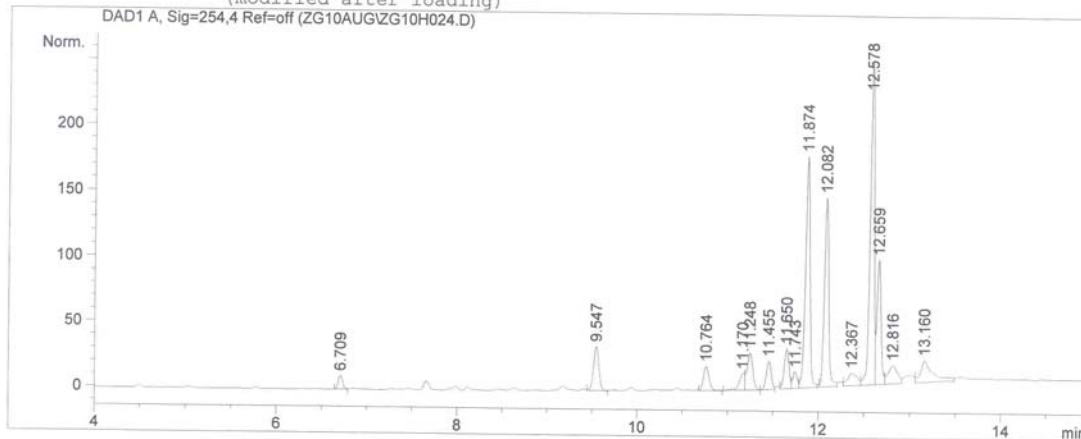


Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/11/2004 12:58:31 AM	Seq. Line	: 25
Sample Name	: Val 7	Location	: P1-C-07
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

=====



=====  
Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

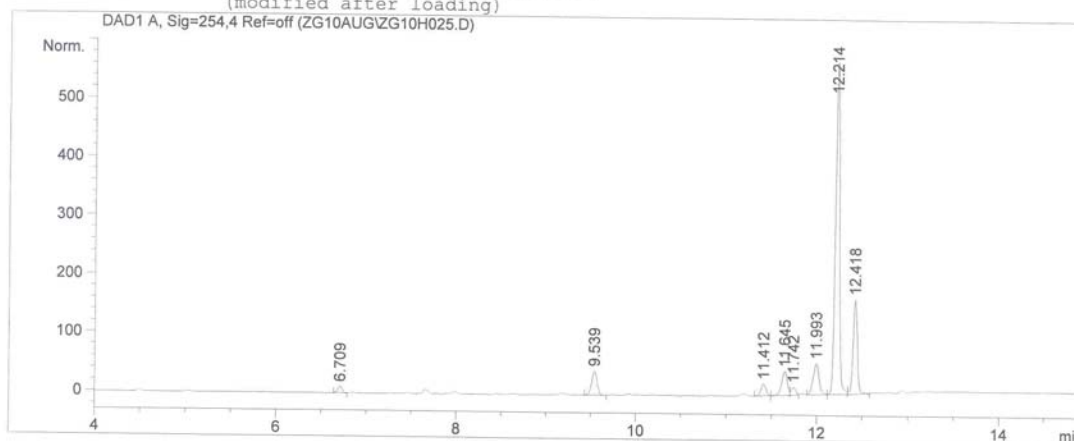
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.709	BV	0.0535	35.54205	10.49449	1.1146
2	9.547	VB	0.0639	140.67128	34.23788	4.4113
3	10.764	BP	0.0664	80.83566	18.71599	2.5349
4	11.170	BV	0.0628	53.41060	12.77990	1.6749
5	11.248	VP	0.0679	127.37429	28.62568	3.9943
6	11.455	VV	0.0603	82.73176	21.76798	2.5944
7	11.650	VV	0.0563	111.24227	30.67770	3.4884
8	11.743	VV	0.0519	43.68406	12.76275	1.3699
9	11.874	VB	0.0550	620.78308	176.73744	19.4670
10	12.082	BV	0.0529	482.94009	144.93358	15.1444
11	12.367	VV	0.0960	72.71994	10.09654	2.2804
12	12.578	VV	0.0483	777.15820	249.65233	24.3707
13	12.659	VV	0.0486	300.24640	95.63408	9.4153
14	12.816	VV	0.0962	101.01217	14.33323	3.1676
15	13.160	VV	0.1220	158.55237	16.81430	4.9720

Totals : 3188.90423 878.26386

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 1:22:36 AM      Seq. Line :   26
Sample Name      : Val 8                    Location  : P1-C-08
Acq. Operator    : Malgosia                 Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



```
=====
                        Area Percent Report
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```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.709	BV	0.0534	36.75970	10.87261	1.1871
2	9.539	BB	0.0651	171.46045	40.70677	5.5372
3	11.412	VP	0.0611	77.54405	20.06119	2.5042
4	11.645	VV	0.0673	179.18158	40.71922	5.7866
5	11.742	VV	0.0549	47.43760	13.54222	1.5320
6	11.993	VV	0.0704	247.09482	53.02968	7.9798
7	12.214	VV	0.0489	1812.13721	572.61578	58.5218
8	12.418	VV	0.0499	524.90076	161.62784	16.9513

Totals :                      3096.51616   913.17531

Results obtained with enhanced integrator!

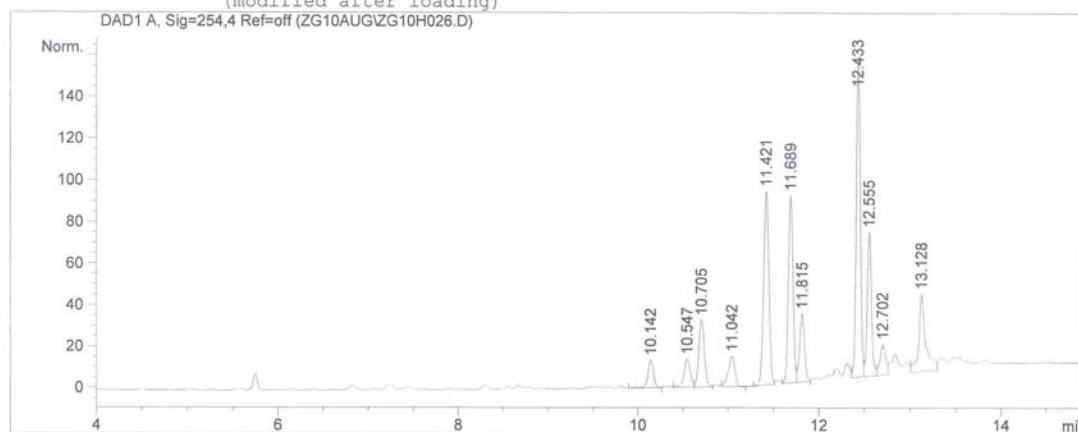
```
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*** End of Report ***
```



Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date : 8/11/2004 1:46:40 AM      Seq. Line : 27
Sample Name    : Val 9                    Location  : P1-C-09
Acq. Operator  : Malgosia                 Inj       : 1
                                           Inj Volume: 1 µl

Acq. Method    : C:\HPCHEM\1\METHODS\ZG01.M
Last changed   : 8/10/2004 3:55:50 PM by Malgosia
                (modified after loading)
Analysis Method: C:\HPCHEM\1\METHODS\ZG01.M
Last changed   : 8/20/2004 3:35:49 PM by Malgosia
                (modified after loading)
=====
```



```
=====
Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.142	VB	0.0666	57.83676	13.33506	2.7099
2	10.547	BV	0.0748	66.62510	13.68512	3.1217
3	10.705	VB	0.0687	140.44400	32.31869	6.5805
4	11.042	BP	0.0751	74.56674	14.71864	3.4938
5	11.421	PV	0.0634	379.31808	93.44712	17.7729
6	11.689	VV	0.0554	320.58743	90.45161	15.0211
7	11.815	VV	0.0580	124.16922	32.92220	5.8179
8	12.433	VV	0.0502	479.90634	154.61864	22.4859
9	12.555	VV	0.0500	226.79257	69.67086	10.6263
10	12.702	VV	0.0650	65.09702	14.90108	3.0501
11	13.128	VV	0.0759	198.91031	37.47134	9.3199

Totals : 2134.25359 567.54036

Results obtained with enhanced integrator!

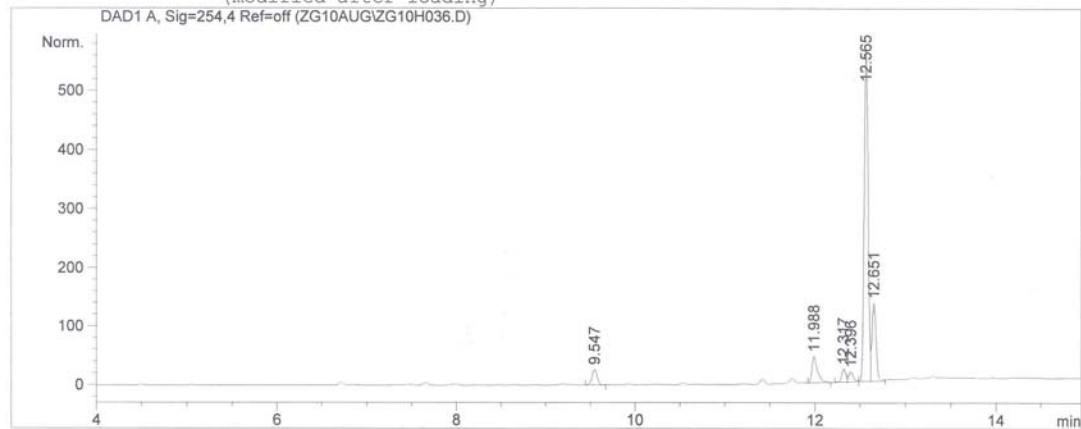
\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/11/2004 5:47:35 AM	Seq. Line	: 37
Sample Name	: Phe 1	Location	: P1-E-01
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

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Area Percent Report

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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.547	BB	0.0625	106.08964	26.59242	4.1439
2	11.988	BB	0.0615	192.12000	45.26096	7.5043
3	12.317	BV	0.0497	70.86447	21.93745	2.7680
4	12.396	VV	0.0534	62.26489	17.53522	2.4321
5	12.565	VV	0.0494	1708.27515	562.77350	66.7258
6	12.651	VV	0.0506	420.52747	133.88492	16.4259

Totals : 2560.14161 807.98447

Results obtained with enhanced integrator!

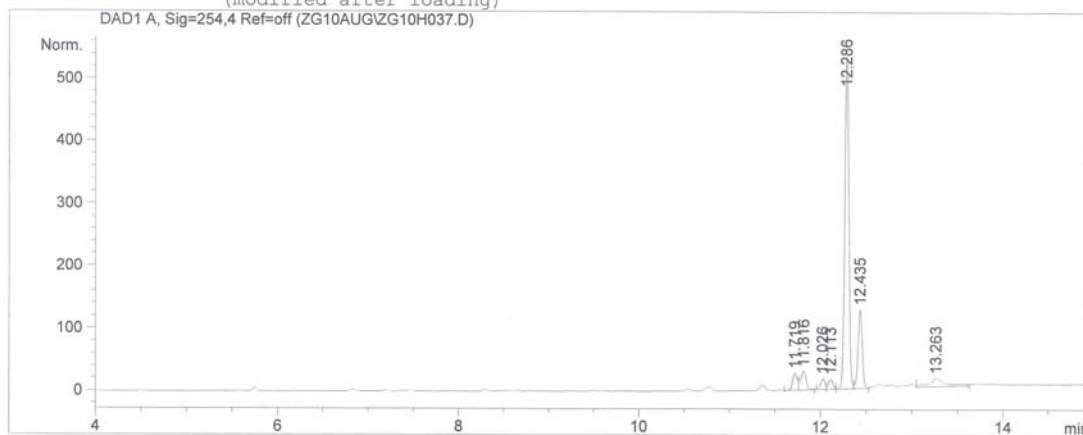
\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

=====

Injection Date	: 8/11/2004 6:11:40 AM	Seq. Line	: 38
Sample Name	: Phe 2	Location	: P1-E-02
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl
Acq. Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/10/2004 3:55:50 PM by Malgosia (modified after loading)		
Analysis Method	: C:\HPCHEM\1\METHODS\ZG01.M		
Last changed	: 8/20/2004 3:35:49 PM by Malgosia (modified after loading)		

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Area Percent Report

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Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.719	VV	0.0534	98.33421	27.68604	3.7838
2	11.816	VB	0.0645	130.46387	30.15449	5.0201
3	12.026	BV	0.0530	60.39660	18.07776	2.3240
4	12.113	VV	0.0509	53.74779	16.11356	2.0681
5	12.286	VV	0.0504	1669.73120	534.89520	64.2489
6	12.435	VV	0.0496	408.22049	126.70089	15.7077
7	13.263	VB	0.1755	177.95453	13.70429	6.8474

Totals : 2598.84868 767.33223

Results obtained with enhanced integrator!

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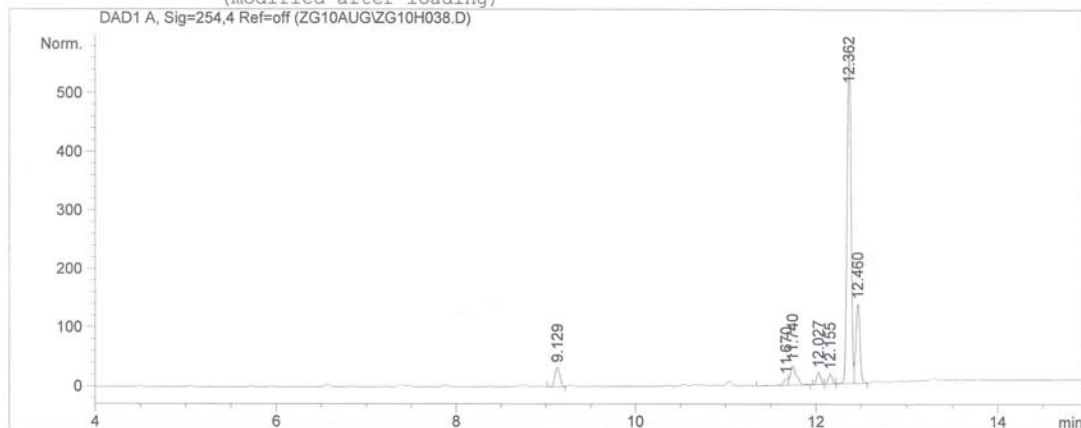
\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

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=====
Injection Date   : 8/11/2004 6:35:44 AM      Seq. Line :   39
Sample Name     : Phe 3                     Location  : P1-E-03
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
  
```



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 Area Percent Report  
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Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=254.4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.129	VV	0.0614	128.28217	32.96026	4.9575
2	11.670	VV	0.0520	40.49026	11.24190	1.5648
3	11.740	VB	0.0672	155.28490	32.79556	6.0010
4	12.027	BV	0.0485	65.37354	20.88184	2.5264
5	12.155	VV	0.0490	52.98052	16.72101	2.0474
6	12.362	VV	0.0475	1730.13074	567.84918	66.8615
7	12.460	VB	0.0477	415.09290	135.73886	16.0414

Totals :                    2587.63503   818.18861

Results obtained with enhanced integrator!

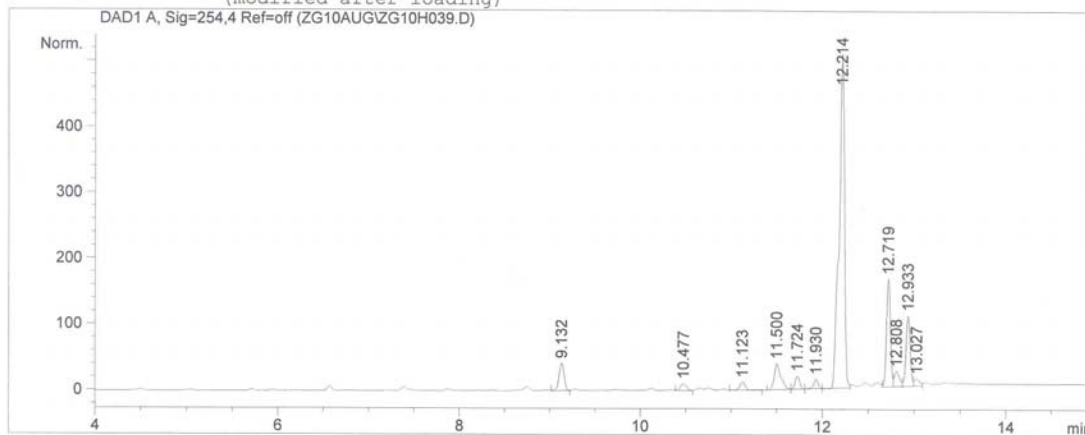
=====  
 \*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```

=====
Injection Date   : 8/11/2004 6:59:47 AM      Seq. Line :   40
Sample Name     : Phe 4                     Location  : P1-E-04
Acq. Operator   : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
  
```



=====  
 Area Percent Report  
 =====

```

Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
  
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.132	VV	0.0597	163.86299	41.87284	4.5296
2	10.477	VV	0.0704	47.77705	10.63588	1.3207
3	11.123	VB	0.0635	49.15244	12.08106	1.3587
4	11.500	PV	0.0737	199.36897	38.96297	5.5111
5	11.724	VV	0.0543	67.85409	19.66646	1.8757
6	11.930	VV	0.0542	49.40593	14.33948	1.3657
7	12.214	VV	0.0593	2071.95093	511.24939	57.2744
8	12.719	VV	0.0470	492.47156	164.14851	13.6133
9	12.808	VV	0.0569	89.05878	23.14269	2.4618
10	12.933	VV	0.0491	341.11298	107.34164	9.4293
11	13.027	VV	0.0586	45.56918	11.40068	1.2597

Totals : 3617.58488 954.84161

Results obtained with enhanced integrator!

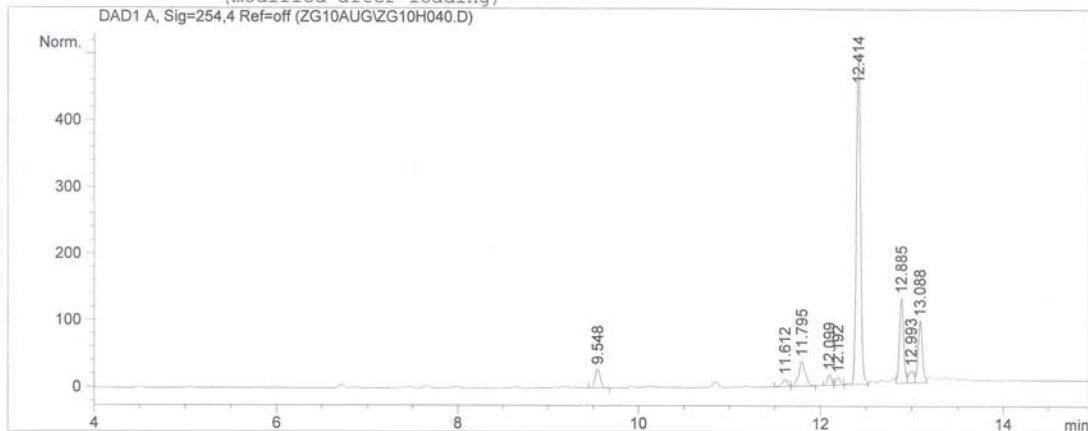
\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

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Injection Date	: 8/11/2004 7:23:54 AM	Seq. Line	: 41
Sample Name	: Phe 5	Location	: P1-E-05
Acq. Operator	: Malgosia	Inj	: 1
		Inj Volume	: 1 µl

Acq. Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/10/2004 3:55:50 PM by Malgosia  
(modified after loading)  
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M  
Last changed : 8/20/2004 3:35:49 PM by Malgosia  
(modified after loading)



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Area Percent Report  
=====

Sorted By : Signal  
Multiplier : 1.0000  
Dilution : 1.0000  
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAQ1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.548	BB	0.0635	114.63654	28.13693	4.1028
2	11.612	VV	0.0599	42.79906	10.87987	1.5317
3	11.795	VB	0.0722	193.44675	37.48305	6.9233
4	12.099	BV	0.0490	50.44863	15.91154	1.8055
5	12.192	VV	0.0512	39.39475	11.72768	1.4099
6	12.414	VV	0.0490	1586.86560	500.66327	56.7927
7	12.885	VV	0.0483	398.03973	127.83804	14.2455
8	12.993	VV	0.0557	69.32121	18.51730	2.4810
9	13.088	VV	0.0500	299.18619	91.83815	10.7076

Totals : 2794.13846 842.99583

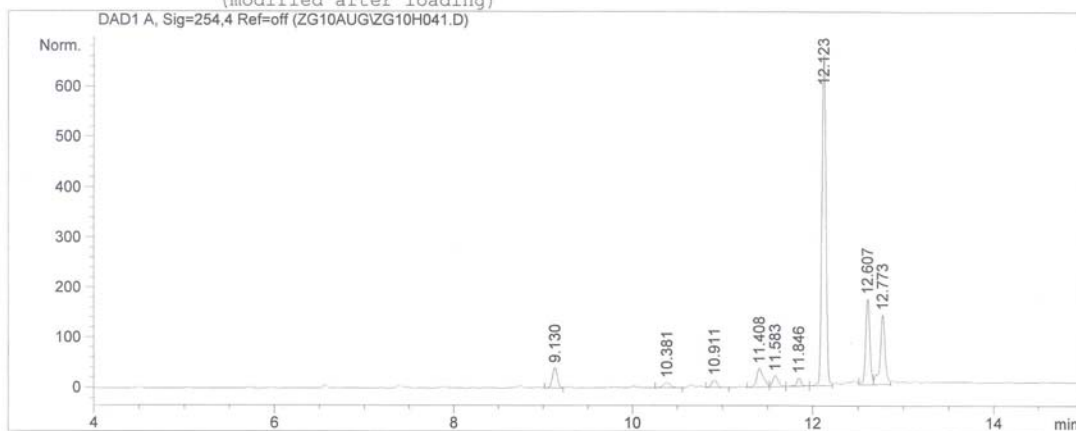
Results obtained with enhanced integrator!

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\*\*\* End of Report \*\*\*

Zorbax SB-C18, 4.6x50mm, 3.5um  
grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

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=====
Injection Date   : 8/11/2004 7:47:58 AM      Seq. Line :   42
Sample Name      : Phe 6                    Location  : Pl-E-06
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/10/2004 3:55:50 PM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
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=====
Area Percent Report
=====
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```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.130	VV	0.0596	159.20345	40.75895	4.2520
2	10.381	BP	0.0835	54.74224	10.38008	1.4621
3	10.911	VB	0.0657	64.67771	15.17095	1.7274
4	11.408	VV	0.0745	194.42311	37.47285	5.1927
5	11.583	VV	0.0670	94.87312	21.70265	2.5339
6	11.846	VV	0.0528	57.37926	16.41315	1.5325
7	12.123	VV	0.0501	2044.34070	660.30365	54.6004
8	12.607	VV	0.0498	555.84882	171.67789	14.8457
9	12.773	VV	0.0555	518.69568	139.24294	13.8534

Totals : 3744.18407 1113.12310

Results obtained with enhanced integrator!

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*** End of Report ***
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```

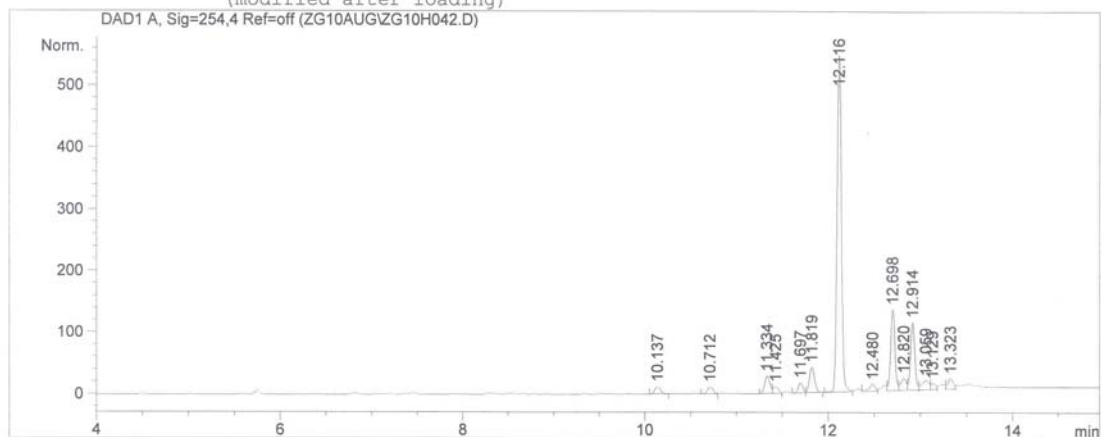


Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```

=====
Injection Date   : 8/11/2004 8:12:03 AM      Seq. Line :   43
Sample Name     : Phe 7                    Location  : P1-E-07
Acq. Operator   : Malgosia                 Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method     : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/11/2004 8:21:24 AM by Malgosia
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\ZG01.M
Last changed    : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
  
```



=====  
 Area Percent Report  
 =====

Sorted By : Signal  
 Multiplier : 1.0000  
 Dilution : 1.0000  
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.137	PB	0.0652	52.10426	12.34519	1.5487
2	10.712	VV	0.0649	48.89508	11.67382	1.4533
3	11.334	PV	0.0630	113.99520	28.29391	3.3882
4	11.425	VV	0.0577	41.15327	10.98063	1.2232
5	11.697	VV	0.0544	55.91812	16.14967	1.6620
6	11.819	VB	0.0620	161.24541	40.88228	4.7926
7	12.116	BV	0.0521	1788.70642	547.04895	53.1648
8	12.480	VV	0.0694	52.72071	11.09317	1.5670
9	12.698	VV	0.0491	421.54895	132.63889	12.5295
10	12.820	VV	0.0602	83.79563	20.26530	2.4906
11	12.914	VV	0.0518	357.93091	110.41520	10.6386
12	13.059	VV	0.0769	79.74725	15.28870	2.3703
13	13.129	VV	0.0525	36.71564	10.08673	1.0913
14	13.323	VV	0.0606	69.97810	17.51849	2.0799

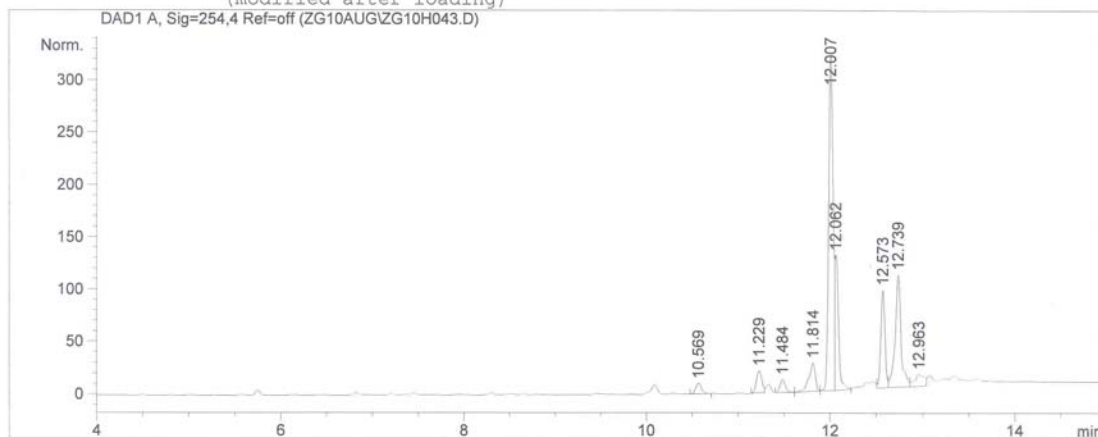
Totals : 3364.45496 984.68093



Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 8:36:09 AM      Seq. Line :   44
Sample Name      : Phe 8                    Location  : Pl-E-08
Acq. Operator    : Malgosia                 Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/11/2004 8:21:24 AM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



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                        Area Percent Report
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```

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.569	PB	0.0691	44.31501	10.11504	1.7114
2	11.229	PV	0.0619	87.34206	21.26828	3.3730
3	11.484	VP	0.0639	50.73627	12.34955	1.9593
4	11.814	VV	0.0691	132.00662	26.96475	5.0978
5	12.007	VV	0.0510	1079.43079	322.49768	41.6855
6	12.062	VB	0.0446	345.15503	128.95168	13.3292
7	12.573	VV	0.0484	291.95001	93.52307	11.2745
8	12.739	VV	0.0637	476.25388	107.52427	18.3920
9	12.963	VV	0.0954	82.27461	11.50961	3.1773

Totals : 2589.46428 734.70393

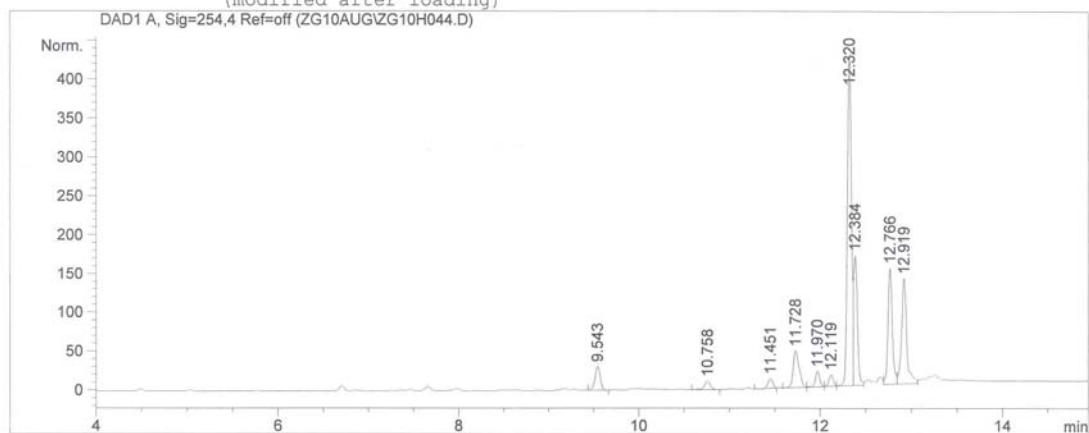
Results obtained with enhanced integrator!

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*** End of Report ***
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Zorbax SB-C18, 4.6x50mm, 3.5um  
 grd. 1-50% MeCN/water/0.1% TFA in 10min., 1mL/min

```
=====
Injection Date   : 8/11/2004 9:00:15 AM      Seq. Line :   45
Sample Name      : Phe 9                    Location  : P1-E-09
Acq. Operator    : Malgosia                  Inj       :    1
                                           Inj Volume: 1 µl

Acq. Method      : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/11/2004 8:21:24 AM by Malgosia
                  (modified after loading)
Analysis Method  : C:\HPCHEM\1\METHODS\ZG01.M
Last changed     : 8/20/2004 3:35:49 PM by Malgosia
                  (modified after loading)
=====
```



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=====
                        Area Percent Report
=====
```

```
Sorted By          :      Signal
Multiplier         :      1.0000
Dilution           :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: DAD1 A, Sig=254,4 Ref=off

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.543	BB	0.0633	123.71336	30.51867	3.6430
2	10.758	VP	0.0688	49.47570	10.93832	1.4569
3	11.451	VV	0.0588	47.78798	12.43223	1.4072
4	11.728	VV	0.0673	216.08466	47.26445	6.3630
5	11.970	VV	0.0522	67.69629	19.64822	1.9935
6	12.119	VV	0.0522	49.30229	15.07061	1.4518
7	12.320	VV	0.0487	1341.26282	426.94598	39.4961
8	12.384	VV	0.0455	481.24567	167.74873	14.1712
9	12.766	VV	0.0526	496.20767	149.93745	14.6118
10	12.919	VV	0.0590	523.15936	135.64822	15.4055

Totals : 3395.93579 1016.15289

Results obtained with enhanced integrator!

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                        *** End of Report ***
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```