

# Highly Enantioselective Direct Conjugate Addition of Ketones to Nitroalkenes Promoted by A Chiral Primary Amine-Thiourea Catalyst

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## Supporting Information

General Information: Nuclear magnetic resonance (NMR) spectra were recorded on a Varian Inova-500 or a Varian Mercury-400. Chemical shifts are reported in parts per million using the solvent resonance internal standard (chloroform, 7.26 and 77.0 ppm, unless specified otherwise). Data are reported as follows: chemical shift, multiplicity (app = apparent, par obsc = partially obscured, ovrlp = overlapping, s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, abq = ab quartet), coupling constant, and integration. FT-IR infrared spectra were recorded on a Mattson Galaxy Series FTIR 3000 spectrometer. Optical rotations were measured on a Jasco DIP 370 digital polarimeter using a 2mL, 1dm cell, and are reported as  $[\alpha]_D$  (concentration in grams/100 mL solvent). Melting points were measured on a Mel-Temp apparatus, and are uncorrected. The mass spectroscopic data were obtained at the Harvard University Mass Spectrometry Facility. Chiral HPLC analysis was performed on a Hewlett-Packard 1050 instrument. Chiral GC analysis was performed on a Hewlett-Packard 5890 gas chromatography. All reactions were carried out in oven-dried glassware under a nitrogen atmosphere unless otherwise noted. Analytical thin layer chromatography was performed on 0.25mm EMD Chemical silica gel plates. Silica gel 60 (230-400 mesh) from EMD Chemicals was used for flash chromatography.

### Experimental Procedures for Catalyzed Michael Addition of Ketone to Nitroolefin:

**General Procedure A.** The nitroolefin (1.0 mmol) and catalyst **1** (0.1 mmol) were placed in a 1.5 mL vial equipped with a Teflon-coated stirbar. Toluene (50  $\mu$ L) was added under  $N_2$ , followed by the addition of ketone (1.1 mmol). The vial was capped and the resulting mixture was stirred for 24 h. The reaction progress was monitored by  $^1H$  NMR analysis. Upon complete consumption of the nitroolefin, the crude reaction mixture was applied to silica gel and product isolated by flash chromatography.

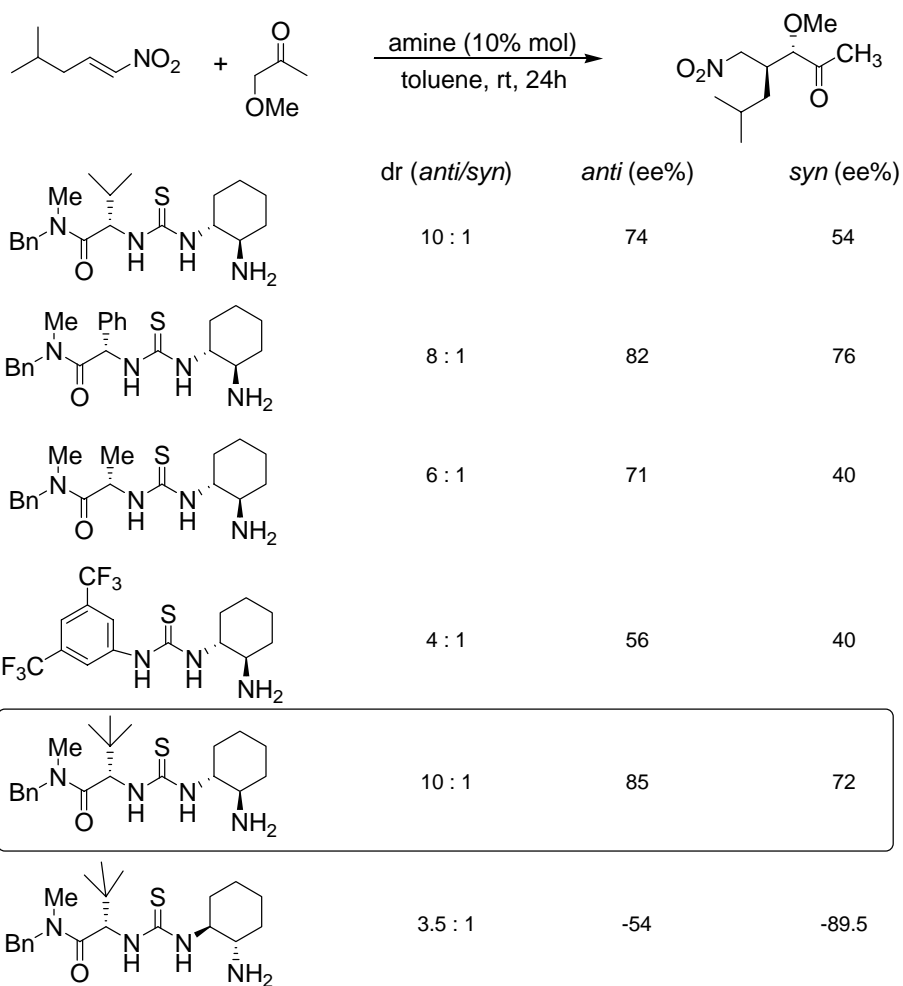
**General Procedure B.** The nitroolefin (1.0 mmol), benzoic acid (0.1 mmol) and catalyst **1** (0.1 mmol) were placed in a 1.5 mL vial equipped with a Teflon-coated stirbar. Toluene (50  $\mu$ L) was added under  $N_2$ , followed by the addition of ketone (5.0 mmol). The vial was capped and the resulting mixture was stirred for 24 h. The reaction progress was monitored by  $^1H$  NMR analysis. Upon complete consumption of the nitroolefin, the crude reaction mixture was applied to silica gel and product isolated by flash chromatography.

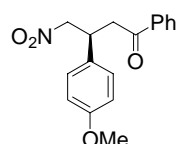
**General Procedure C.** The nitroolefin (0.5 mmol) and catalyst **1** (0.05 mmol) were placed in a 1.5 mL vial equipped with a Teflon-coated stirbar. Toluene (250  $\mu$ L) was added under  $N_2$ , followed by the addition of ketone (2.0 mmol). The resulting mixture was stirred at rt, and an addition portion of catalyst (0.05 mmol in 80  $\mu$ L of toluene) was added after 12 h. The resulting mixture was stirred for additional 24 h. The reaction progress was monitored by  $^1H$  NMR

analysis. Upon complete consumption of the nitroolefin, the crude reaction mixture was applied to silica gel and product isolated by flash chromatography.

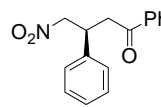
**General Procedure D.** The catalyst **1** (0.1 mmol) was placed in a 1.5 mL vial equipped with a Teflon-coated stirbar. Toluene (50  $\mu$ L) was added under  $N_2$ , followed by the addition of ketone (2.5 mmol). A solution of nitroolefin (0.5 mmol) in toluene (200  $\mu$ L) was added via syringe pump over 20 h. After the completion of the addition of nitroolefin, the resulting mixture was stirred for additional 24 h. The reaction progress was monitored by  $^1H$  NMR analysis. Upon complete consumption of the nitroolefin, the crude reaction mixture was applied to silica gel and product isolated by flash chromatography.

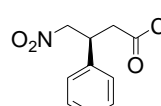
### Catalyst screen.

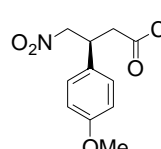


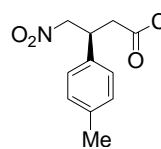
 Prepared from *trans*-4-methoxy- $\beta$ -nitrostyrene (180 mg, 1.0 mmol) and acetophenone (1.1 mmol, 128  $\mu$ L) according to General Procedure A, affording 212 mg of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20}$  -23.4 $^\circ$  (c 1.42,  $CHCl_3$ ); mp 70.5-72.5  $^\circ C$ ; IR (KBr) 3033, 2936, 2839, 1681, 1549, 1255, 1180, 1029, 760, 691, 560  $cm^{-1}$ ;  $^1H$  NMR (400 MHz,  $CDCl_3$ )  $\delta$  7.92 (d,  $J$  = 5.9 Hz, 2H), 7.58 (t,  $J$  = 7.3, 1H), 7.46 (t,  $J$  = 8.1 Hz, 2H), 7.22 (d,  $J$  = 8.8 Hz, 2H), 6.87 (d,  $J$  = 8.8 Hz, 1H), 4.83 (dd,  $J$  = 6.95, 12.3 Hz, 1H), 4.67 (dd,  $J$  = 8.1, 12.4 Hz, 1H), 4.18 (m, 1H), 3.78 (s, 3H), 3.48 (abq,  $J$  =

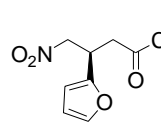
6.6, 16.5 Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  197.0, 159.0, 136.4, 133.5, 131.0, 128.7, 128.5, 128.0, 114.4, 79.8, 55.2, 41.6, 38.6; LRMS (CI)  $m/z$  calcd for  $\text{C}_{17}\text{H}_{17}\text{NO}_4$  299, found 299.

 Prepared from *trans*- $\beta$ -nitrostyrene (150 mg, 1.0 mmol) and acetophenone (1.1 mmol, 128  $\mu\text{L}$ ) according to General Procedure A, affording 223 mg (83% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$   $-23.2^\circ$  (c 2.51,  $\text{CHCl}_3$ ); mp 91-92.5  $^\circ\text{C}$ ; IR (KBr) 3081, 3060, 3029, 2921, 1688, 1546, 1373, 1269, 1225, 1184, 703, 686, 560  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.93 (m, 2H), 7.60 (m, 1H), 7.48 (m, 2H), 7.36-7.25 (m, 5H), 6.87 (d,  $J$  = 8.8 Hz, 1H), 4.86 (dd,  $J$  = 6.95, 12.4 Hz, 1H), 4.72 (dd,  $J$  = 8.1, 12.4 Hz, 1H), 4.27 (m, 1H), 3.52 (m, 2H), 3.48 (abq,  $J$  = 6.6, 16.5 Hz, 2H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  196.8, 139.1, 136.3, 133.6, 129.0, 128.7, 128.0, 127.9, 127.4, 79.5, 41.5, 39.2; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  287, found 287.

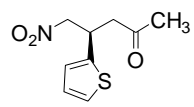
 Prepared from *trans*- $\beta$ -nitrostyrene (150 mg, 1.0 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) according to General Procedure B, affording 193mg (93% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$   $+2.4^\circ$  (c 1.03,  $\text{CHCl}_3$ ); mp 120-122.5  $^\circ\text{C}$ ; ( IR (KBr) 3064, 3035, 2968, 2948, 2919, 2902, 1714, 1548, 1384, 1361, 1163, 758, 696, 548  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.35 (m, 2H), 7.32 (m, 1H), 7.22 (m, 2H), 4.71 (dd,  $J$  = 7.3, 12.5 Hz, 1H), 4.62 (dd,  $J$  = 7.8, 12.2 Hz, 1H), 4.04 (m, 1H), 2.93 (d,  $J$  = 7.32, 1H), 2.12 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  205.4, 138.8, 129.0, 127.9, 127.3, 79.4, 55.5, 46.1, 39.0, 30.4; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  225, found 225.

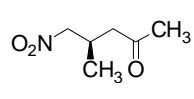
 Prepared from *trans*-4-methoxy- $\beta$ -nitrostyrene (180 mg, 1.0 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) according to General Procedure B, affording 208 mg of white solid after chromatography (19:10, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$   $-1.5^\circ$  (c 1.32,  $\text{CHCl}_3$ ); mp 93.5-94.5  $^\circ\text{C}$ ; IR (KBr) 3028, 3002, 2960, 2901, 1715, 1550, 1517, 1260, 1180, 1033, 813, 554  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.14 (d,  $J$  = 8.4 Hz, 2H), 6.86 (d,  $J$  = 8.8 Hz, 2H), 4.68 (dd,  $J$  = 6.6, 12.3 Hz, 1H), 4.58 (dd,  $J$  = 7.7, 12.1 Hz, 1H), 3.95 (m, 1H), 3.78 (s, 3H), 3.73 (d,  $J$  = 6.9 Hz, 2H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  205.5, 159.1, 130.6, 128.4, 114.4, 79.7, 55.5, 46.2, 38.4, 30.4; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  255, found 255.

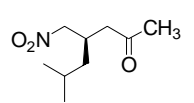
 Prepared from *trans*-4-methyl- $\beta$ -nitrostyrene (163 mg, 1.0 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) according to General Procedure B, affording 192 mg (87% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$   $+5.5^\circ$  (c 1.59,  $\text{CHCl}_3$ ). IR (KBr) 3056, 3029, 2976, 2944, 2924, 1717, 1551, 1378, 1365, 1163, 816, 544  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.14-7.08 (m, 1H), 4.69 (dd,  $J$  = 6.9, 12.3 Hz, 1H), 4.60 (dd,  $J$  = 7.7, 12.1 Hz, 1H), 4.0-3.93 (m, 1H), 2.91 (d,  $J$  = 6.9 Hz, 2H), 2.31 (s, 3H), 2.11 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  205.4, 137.4, 135.7, 129.6, 127.1, 79.5, 46.0, 38.5, 30.2, 20.9; LRMS (APCI)  $m/z$  calcd for  $\text{C}_{12}\text{H}_{15}\text{NO}_3$  221.1, found 221.4.

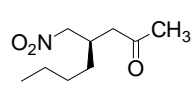
 Prepared from 2-(2-nitrovinyl)furan (139 mg, 1.0 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) according to General Procedure B, affording 175 mg (89% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$   $+5.7^\circ$  (c 1.32,  $\text{CHCl}_3$ ); mp 54.5-56  $^\circ\text{C}$ ; IR (film) 3151, 3124, 2920, 1714, 1552, 1430, 1377, 1165, 1015, 741  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.34 (dd,  $J$  = 0.73, 1.8 Hz, 1H), 6.30 (dd,  $J$  = 1.8, 5.7 Hz, 2H), 6.15 (d,  $J$  = 6.2 Hz, 1H), 4.72 (dd,  $J$  = 6.2, 10.6 Hz, 1H), 4.67 (dd,  $J$  = 3.3, 9.9 Hz, 1H), 4.14-4.07 (m, 1H), 3.01 (dd,  $J$  = 6.6, 18.1 Hz, 1H), 2.93 (dd,  $J$  = 7.3, 13.2 Hz, 1H), 2.18

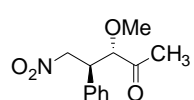
(s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  205.0, 151.6, 142.3, 110.5, 107.0, 77.0, 43.4, 32.8, 30.2; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  215, found 215.

 Prepared from *trans*-2-(2-nitrovinyl)thiophene (155 mg, 1.0 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) according to General Procedure B, affording 201 mg (94% yield) of pale yellow oil after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$  -11.8° (c 1.76,  $\text{CHCl}_3$ ). IR (film) 3111, 3005, 2919, 1714, 1552, 1429, 1378, 1166, 850, 707  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.21 (dd,  $J$  = 1.1, 4.8 Hz, 1H), 6.95-6.90 (m, 2H), 6.15 (d,  $J$  = 6.2 Hz, 1H), 4.74 (dd,  $J$  = 6.6, 12.4 Hz, 1H), 4.67 (dd,  $J$  = 7.3, 12.4 Hz, 1H), 4.35-4.29 (m, 1H), 3.02 (d,  $J$  = 6.6 Hz, 1H), 2.16 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  205.0, 141.6, 127.1, 125.4, 124.6, 79.6, 46.7, 34.4, 30.2; LRMS (CI)  $m/z$  calcd for  $\text{C}_9\text{H}_{11}\text{NO}_3\text{S}$  213, found 213.

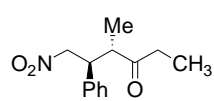
 Prepared from *trans*-1-nitropropene (44 mg, 0.5 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) using 15 mol% of catalyst (0.075 mmol) and 2 mol% of benzoic acid (0.01 mmol) according to General Procedure B, affording 55 mg (70% yield) of pale yellow oil after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$  -4.8° (c 1.0,  $\text{CHCl}_3$ ). IR (film) 2972, 2938, 1716, 1551, 1517, 1369, 1172  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.44 (dd,  $J$  = 6.2, 11.7 Hz, 1H), 4.37 (dd,  $J$  = 6.6, 11.9 Hz, 1H), 2.83 (m, 1H), 2.81 (dd,  $J$  = 6.3, 13.1 Hz, 1H), 2.64 (dd,  $J$  = 6.6, 17.9 Hz, 1H), 2.17 (s, 3H), 1.07 (d,  $J$  = 6.9 Hz, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  206.3, 80.1, 46.4, 30.3, 28.1, 17.3; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  163, found 163.

 Prepared from *trans*-4-methyl-1-nitro-pent-1-ene (72 mg, 0.5 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) using 15 mol% of catalyst (0.075 mmol) and 2 mol% of benzoic acid (0.01 mmol) according to General Procedure B, affording 82 mg (81% yield) of pale yellow oil after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$  -9.0° (c 1.0,  $\text{CHCl}_3$ ). IR (film) 2961, 2934, 2872, 1717, 1551, 1369, 735  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.48 (m, 2H), 2.71-2.55 (m, 2H), 2.54 (dd,  $J$  = 4.8, 17.6 Hz, 1H), 2.17 (s, 3H), 1.68-1.58 (m, 1H), 1.30-1.17 (m, 2H), 0.93 (d,  $J$  = 6.6 Hz, 3H), 0.91 (d,  $J$  = 6.6 Hz, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  206.7, 78.5, 44.7, 40.5, 30.8, 30.4, 25.0, 22.4, 22.3; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  205, found 205.

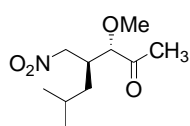
 Prepared from *trans*-1-nitro-hex-1-ene (72 mg, 0.5 mmol) and acetone (5.0 mmol, 368  $\mu\text{L}$ ) using 15 mol% of catalyst (0.075 mmol) and 2 mol% of benzoic acid (0.01 mmol) according to General Procedure B, affording 78 mg (78% yield) of pale yellow oil after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_{\text{D}}^{20}$  -5.0° (c 0.8,  $\text{CHCl}_3$ ). IR (film) 2934, 2864, 1719, 1551, 1435, 1379, 1167, 733  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.45 (d,  $J$  = 5.1 Hz, 2H), 2.65-2.50 (m, 3H), 2.17 (s, 3H), 1.41-1.29 (m, 5H), 0.91 (m, 4H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  206.7, 78.3, 44.6, 32.9, 31.1, 30.4, 28.7, 22.5, 13.8; LRMS (CI/ $\text{NH}_3$ )  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  205, found 205.

 Prepared from *trans*- $\beta$ -nitrostyrene (150 mg, 1.0 mmol) and  $\alpha$ -methoxyacetone (1.1 mmol, 101  $\mu\text{L}$ ) according to General Procedure A, affording 178 mg (75% yield) of white solid after chromatography (4:1, hexanes:EtOAc). IR (KBr) 3066, 3031, 3010, 2933, 2831, 1715, 1554, 1380, 1226, 1196, 1106, 764, 703, 653, 534  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.36-7.30 (m, 3H), 7.23 (m, 2H), 4.89 (dd,  $J$  = 4.8, 12.7 Hz, 1H), 4.74 (dd,  $J$  = 8.4, 13.0 Hz, 1H), 3.90-80 (m, 2H), 3.38 (s, 3H), 2.04 (s, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$

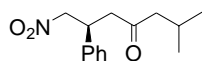
208.1, 135.3, 129.1, 128.5, 128.1, 88.1, 76.7, 58.8, 45.9, 26.2; LRMS (CI) m/z calcd for [M + NH<sub>4</sub>] 255, found 255.



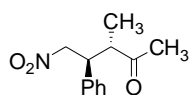
Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-pentanone (0.75 mmol, 80  $\mu$ L) according to General Procedure B using 2 mol% benzoic acid (0.01 mmol, 1.2 mg), affording 76 mg (65% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} + 13.4^\circ$  (c 1.55, CHCl<sub>3</sub>). IR (KBr) 3062, 3032, 2972, 2940, 2882, 1716, 1544, 1457, 1436, 1382, 759, 703 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32-7.23 (m, 3H), 7.19-7.16 (m, 2H), 4.82 (dd, J = 5.1, 12.8 Hz, 1H), 4.74 (dd, J = 9.8, 12.8 Hz, 1H), 3.81-3.75 (m, 1H), 3.03-2.9 (m, 1H), 2.37-2.27 (m, 1H), 2.19-2.04 (m, 1H), 1.20 (d, J = 6.95 Hz, 3H), 0.85 (t, J = 7.32 Hz, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  212.5, 138.0, 128.8, 77.5, 49.0, 45.8, 35.7, 14.3, 7.2; LRMS (APCI) m/z calcd for C<sub>13</sub>H<sub>17</sub>NO<sub>3</sub> 235.1, found 235.2.



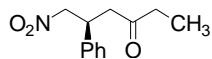
Prepared from *trans*-4-methyl-1-nitro-pent-1-ene (65 mg, 0.5 mmol) and  $\alpha$ -methoxyacetone (2.0 mmol, 92  $\mu$ L) General Procedure C, affording 94 mg (78% yield) of colorless oil after chromatography (9:1, hexanes:EtOAc). IR (film) 2960, 2935, 2874, 1720, 1554, 1468, 1382, 1356, 1110, 756, 740 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  4.45 (dd, J = 5.9, 13.2, 1H), 4.37 (dd, J = 6.2, 13.2, 1H), 3.59 (d, J = 5.5 Hz, 1H), 3.38 (s, 3H), 2.69-2.63 (m, 1H), 2.21 (s, 3H), 1.68-1.58 (m, 1H), 1.68-1.60 (m, 1H), 1.34-1.17 (m, 2H), 0.93 (d, J = 2.9 Hz, 3H), 0.92 (d, J = 3.3 Hz, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  209.4, 87.0, 75.6, 58.9, 37.4, 37.3, 26.1, 22.5, 22.1; LRMS (CI) m/z calcd for [M + NH<sub>4</sub>] 235, found 235.



Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 4-methyl-pent-2-one (1.1 mmol, 69  $\mu$ L) according to General Procedure A, affording 88 mg (71% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} + 3.5^\circ$  (c 1.02, CHCl<sub>3</sub>); mp 37-39 °C; IR (KBr) 3065, 3032, 2960, 2932, 2873, 1712, 1552, 1378, 761, 701 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.35-7.20 (m, 5H), 4.72 (dd, J = 7.0, 12.4 Hz, 1H), 4.63 (dd, J = 7.7, 12.5 Hz, 1H), 4.05 (m, 1H), 2.91 (abq, J = 6.6, 15.4 Hz, 2H), 2.28 (abq, J = 7.3, 13.9 Hz, 2H), 2.12 (m, 1H), 0.85 (d, J = 6.3 Hz, 3H), 0.84 (d, J = 6.3 Hz, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  207.5, 138.9, 129.0, 127.8, 127.4, 79.4, 52.2, 45.7 39.0, 24.4, 22.4, 22.3.; LRMS (CI) m/z calcd for [M + NH<sub>4</sub>] 267, found 267.

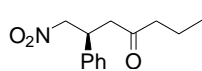


Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-hexanone (2.5 mmol, 225  $\mu$ L) according to General Procedure B using only 2 mol% of benzoic acid (0.01 mmol, 1.2 mg), affording 59 mg (53% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} - 4.8^\circ$  (c 0.8, CHCl<sub>3</sub>). IR (KBr) 3084, 3064, 3035, 2986, 1710, 1544, 1437, 1385, 1282, 1170, 1089, 759, 703, 536 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.33-7.19 (m, 5H), 4.80 (dd, J = 4.9, 12.7 Hz, 1H), 4.73 (dd, J = 9.3, 12.9 Hz, 1H), 3.81 (m, 1H), 3.02 (m, 1H), 1.95 (s, 3H); 1.23 (d, J = 7.3, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  209.8, 137.9, 128.9, 127.9, 127.8, 77.5, 49.7, 45.7, 29.4, 14.2; LRMS (ES) m/z calcd for [M + NH<sub>4</sub>] 239, found 239.

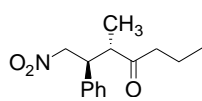


Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-hexanone (2.5 mmol, 225  $\mu$ L) according to General Procedure B using only 2 mol% of benzoic acid (0.01 mmol, 1.2 mg), affording 28 mg (25% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} + 7.06^\circ$  (c 0.75, CHCl<sub>3</sub>). IR (KBr) 3085, 3062, 3032, 2986, 2949, 2906, 1718, 1548, 1379, 1120, 762, 697, 571, 543 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz,

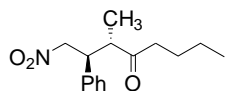
CDCl<sub>3</sub>)  $\delta$  7.35-7.20 (m, 5H), 4.72 (dd,  $J = 7.0, 12.4$  Hz, 1H), 4.63 (dd,  $J = 7.7, 12.1$  Hz, 1H), 4.06 (m, 1H), 2.89 (d,  $J = 7.0$ , 2H), 2.46, 2.24 (m, 2H), 1.02 (t,  $J = 7.3$ , 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  208.1, 138.9, 129.0, 127.8, 127.4, 79.5, 44.9, 39.1, 36.5, 7.5; LRMS (ES)  $m/z$  calcd for [M + NH<sub>4</sub>] 239, found 239.



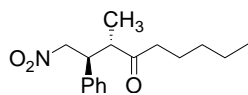
Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-hexanone (2.0 mmol, 110  $\mu$ L) according to General Procedure A affording 78 mg (67% yield) of colorless oil after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} +6.0^\circ$  (c 0.58, CHCl<sub>3</sub>). IR (KBr) 3065, 3033, 2965, 2935, 2877, 1712, 1551, 1378, 764, 701 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.35-7.20 (m, 5H), 4.72 (dd,  $J = 6.8, 12.2$  Hz, 1H), 4.63 (dd,  $J = 7.8, 12.2$  Hz, 1H), 4.05 (m, 1H), 2.92 (abq,  $J = 6.8, 17.6$  Hz, 2H), 2.39 (m, 2H), 1.58 (m, 2H), 0.86 (t,  $J = 7.32$ , 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  207.8, 138.9, 129.0, 127.8, 127.4, 79.5, 45.2, 45.1, 39.0, 17.0, 13.5; LRMS (ES)  $m/z$  calcd for [M + NH<sub>4</sub>] 253, found 253.



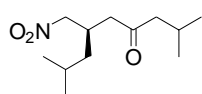
Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-hexanone (1.5 mmol, 92  $\mu$ L) according to General Procedure B using only 2 mol% of benzoic acid (0.01 mmol, 1.2 mg), affording 64 mg (51% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} +13.0^\circ$  (c 0.42, CHCl<sub>3</sub>); mp 61.5-63.0  $^\circ$ C; IR (KBr) 3064, 3032, 2966, 2880, 1714, 1542, 1434, 1384, 759, 703 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.32-7.17 (m, 5H), 4.81 (dd,  $J = 5.4, 12.7$  Hz, 1H), 4.73 (dd,  $J = 9.8, 12.7$  Hz, 1H), 3.81 (m, 1H), 2.99 (m, 1H), 2.30 (dt,  $J = 7.3, 17.1$  Hz, 1H), 2.08 (dt,  $J = 7.3, 17.6$  Hz, 1H), 1.41 (m, 2H), 1.20 (d,  $J = 6.8$  Hz, 3H), 0.72 (t,  $J = 7.32$ , 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  211.8, 138.0, 128.9, 127.9, 127.8, 77.6, 49.2, 45.8, 44.4, 19.5, 16.5, 14.4, 13.4; LRMS (CI)  $m/z$  calcd for [M + NH<sub>4</sub>] 267, found 267.



Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-heptanone (1.5 mmol, 105  $\mu$ L) according to General Procedure B using only 2 mol% of benzoic acid (0.01 mmol, 1.2 mg), affording 69 mg (53% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} +11.9^\circ$  (c 0.39, CHCl<sub>3</sub>); mp 64-65  $^\circ$ C; IR (KBr) 2955, 2934, 2870, 1708, 1542, 1437, 1383, 755, 701 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.31-7.28 (m, 2H), 7.26-7.23 (m, 1H), 7.18 (d,  $J = 6.8$  Hz, 2H), 4.81 (dd,  $J = 4.9, 12.7$  Hz, 1H), 4.73 (dd,  $J = 9.8, 12.7$  Hz, 1H), 3.81 (m, 1H), 3.01 (m, 1H), 2.31 (dt,  $J = 7.3, 17.1$  Hz, 1H), 2.10 (dt,  $J = 7.3, 17.6$  Hz, 1H), 1.34 (m, 2H), 1.20 (d,  $J = 6.8$  Hz, 3H), 1.14 (m, 2H), 0.79 (t,  $J = 7.3$ , 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  212.0, 138.0, 128.9, 127.9, 77.6, 49.2, 45.8, 42.2, 25.1, 22.0, 14.4, 13.4; LRMS (CI)  $m/z$  calcd for [M + NH<sub>4</sub>] 281, found 281.

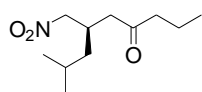


Prepared from *trans*- $\beta$ -nitrostyrene (0.5 mmol, 75 mg) and 3-heptanone (1.5 mmol, 117  $\mu$ L) according to General Procedure B using only 2 mol% of benzoic acid (0.01 mmol, 1.2 mg), affording 70 mg (50% yield) of white solid after chromatography (4:1, hexanes:EtOAc).  $[\alpha]_D^{20} +13.2^\circ$  (c 0.93, CHCl<sub>3</sub>); mp 61.5-63.0  $^\circ$ C; IR (KBr) 3065, 3012, 2956, 2872, 1709, 1543, 1459, 1438, 1382, 700 cm<sup>-1</sup>; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)  $\delta$  7.31-7.28 (m, 2H), 7.26-7.23 (m, 1H), 7.19 (d,  $J = 6.8$  Hz, 2H), 4.81 (dd,  $J = 5.4, 12.7$  Hz, 1H), 4.73 (dd,  $J = 9.8, 12.7$  Hz, 1H), 3.81 (m, 1H), 3.00 (m, 1H), 2.30 (dt,  $J = 7.3, 17.1$  Hz, 1H), 2.10 (dt,  $J = 7.3, 17.6$  Hz, 1H), 1.36 (m, 2H), 1.20 (d,  $J = 6.8$  Hz, 3H), 1.18 (partially overlapped, m, 2H), 1.05 (m, 2H), 0.82 (t,  $J = 7.3$ , 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>)  $\delta$  212.0, 138.0, 128.8, 127.9, 127.8, 77.6, 49.2, 45.8, 42.5, 31.0, 22.7, 22.3, 14.4, 13.8; LRMS (CI)  $m/z$  calcd for [M + NH<sub>4</sub>] 295, found 295.



Prepared from *trans*-4-methyl-1-nitro-pent-1-ene (65 mg, 0.5 mmol) and 4-methyl-pent-2-one (5.0 mmol, 312  $\mu$ L) General Procedure D, affording 64 mg (56% yield) of colorless oil after chromatography (95:5, hexanes:EtOAc).

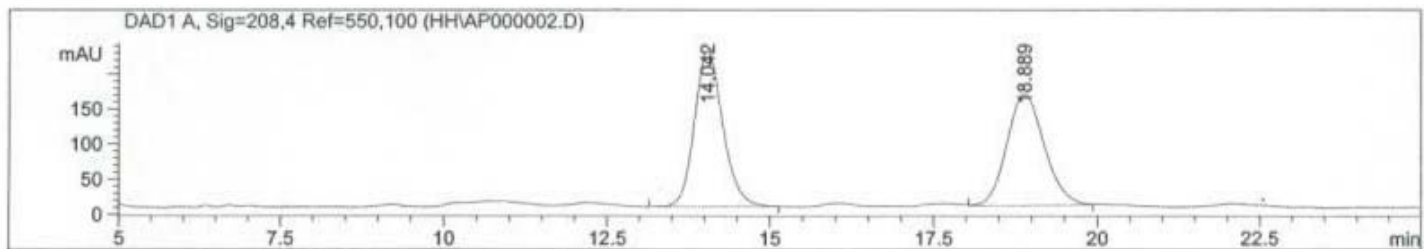
$[\alpha]_D^{20}$   $-8.6^\circ$  (c 0.5,  $\text{CHCl}_3$ ). IR (KBr) 2960, 2873, 1712, 1552, 1468, 1369, 1171  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.45 (d,  $J = 5.5$  Hz, 2H), 2.72 (m, 1H), 2.61 (dd,  $J = 7.3, 17.9$  Hz, 1H), 2.49 (dd,  $J = 5.5, 17.9$  Hz, 1H), 2.30 (d,  $J = 6.6$  Hz, 2H), 2.19 (m, 1H), 1.68 (m, 1H), 1.33 (m, 2H), 0.93 (m, 12H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  208.9, 78.6, 52.3, 49.2, 44.4, 40.6, 30.8, 25.1, 24.5, 22.5, 22.4, 22.3; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  247, found 247.



Prepared from *trans*-4-methyl-1-nitro-pent-1-ene (65 mg, 0.5 mmol) and 2-pentanone (5.0 mmol, 270  $\mu$ L) General Procedure D, affording 53 mg (50% yield) of colorless oil after chromatography (9.5:0.5, hexanes:EtOAc).  $[\alpha]_D^{20}$   $-$

$8.3^\circ$  (c 0.3,  $\text{CHCl}_3$ ). IR (KBr) 2962, 2935, 2875, 1713, 1551, 1381, 913, 735  $\text{cm}^{-1}$ ;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  4.4 (d,  $J = 5.9$  Hz, 2H), 2.72 (m, 1H), 2.61 (dd,  $J = 7.3, 18.1$  Hz, 1H), 2.50 (dd,  $J = 5.4, 18.1$  Hz, 1H), 2.41 (t,  $J = 7.3$  Hz, 2H), 1.66 (m, 3H), 1.29 (m, 2H), 0.93 (m, 9H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  209.1, 78.6, 45.2, 43.9, 40.6, 30.9, 25.1, 22.5, 22.3, 17.1, 13.7; LRMS (CI)  $m/z$  calcd for  $[\text{M} + \text{NH}_4]$  233, found 233.

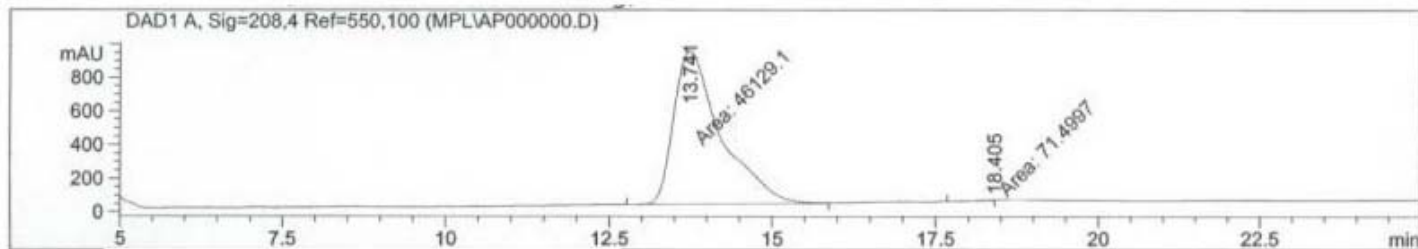
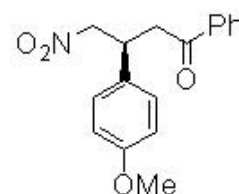
AD, 20% ipa/hex, 1 mL/min



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.042	PB	0.4463	6748.23437	222.23282	51.5525
2	18.889	VV	0.5575	6341.78955	158.94890	48.4475

Totals : 1.30900e4 381.18172

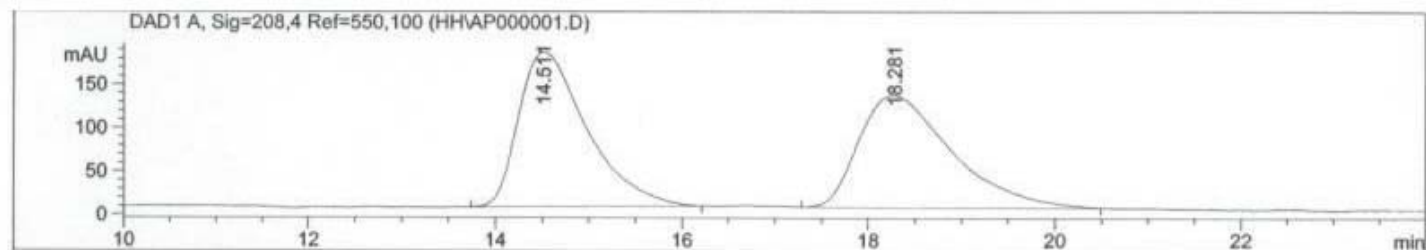


Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.741	MM	0.8394	4.61291e4	915.88409	99.8452
2	18.405	MM	0.2137	71.49969	5.57584	0.1548

Totals : 4.62006e4 921.45994

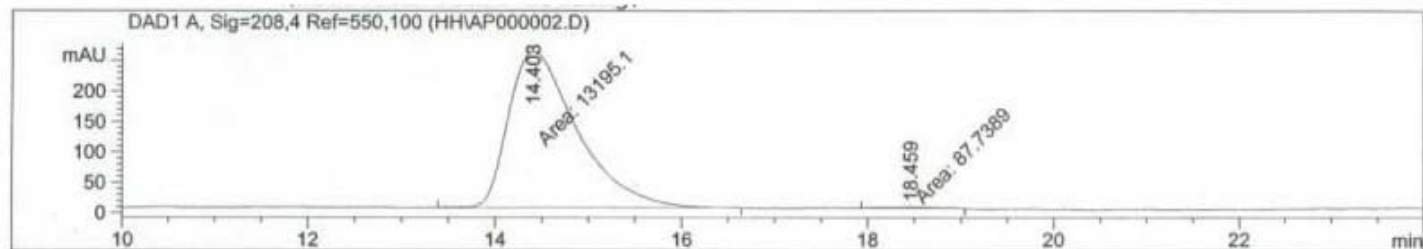
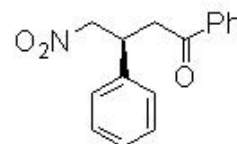
15% ipa/hex, 0.9 ml/min AS



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.511	BB	0.6879	9122.14355	178.59947	50.4062
2	18.281	VB	0.8366	8975.10645	129.49702	49.5938

Totals : 1.80973e4 308.09650

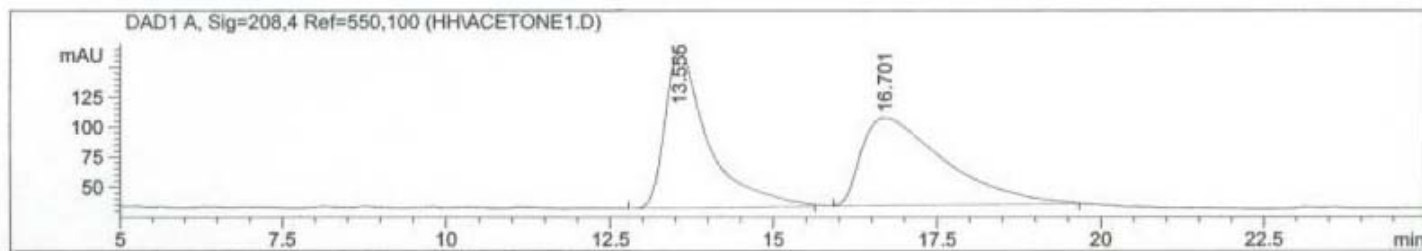


Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.403	MM	0.8561	1.31951e4	256.89523	99.3395
2	18.459	MM	0.6565	87.73894	2.22759	0.6605

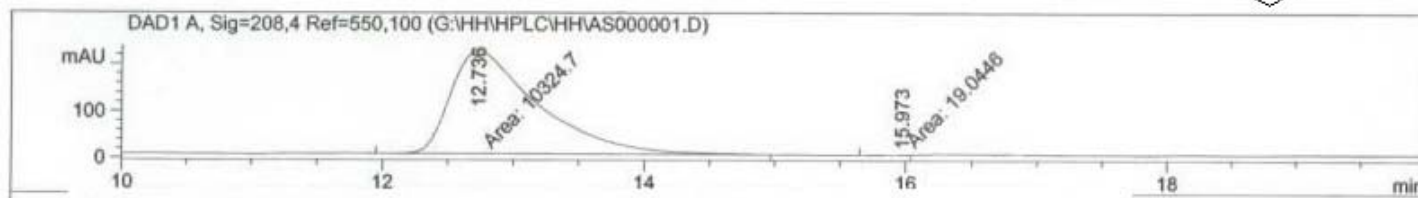
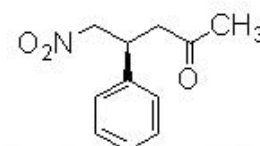
Totals : 1.32828e4 259.12282

25% ipa/hex, 1mL/min, AS



Area Percent Report

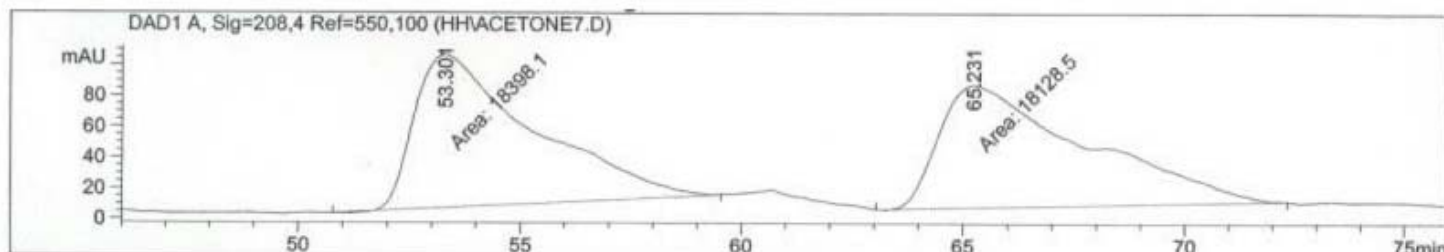
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.555	PB	0.6399	6038.68750	130.47807	48.1324
2	16.701	BB	1.0525	6507.30225	73.24842	51.8676
Totals :				1.25460e4	203.72649	



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.736	MM	0.7849	1.03247e4	219.23091	99.8159
2	15.973	MM	0.3146	19.04464	1.00907	0.1841
Totals :				1.03438e4	220.23998	

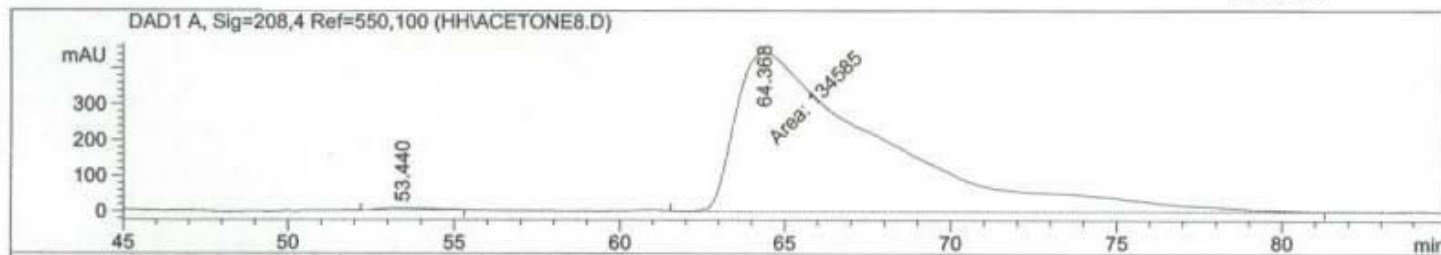
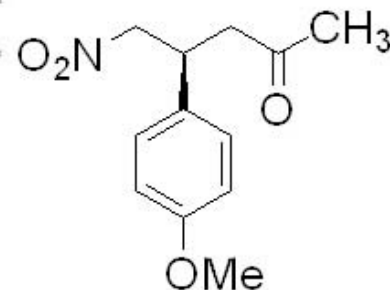
OD, 8% IPA/Hex, 0.6 mL/min



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	53.301	MM	3.0810	1.83981e4	99.52593	50.3691
2	65.231	MM	3.7879	1.81285e4	79.76414	49.6309

Totals : 3.65266e4 179.29007

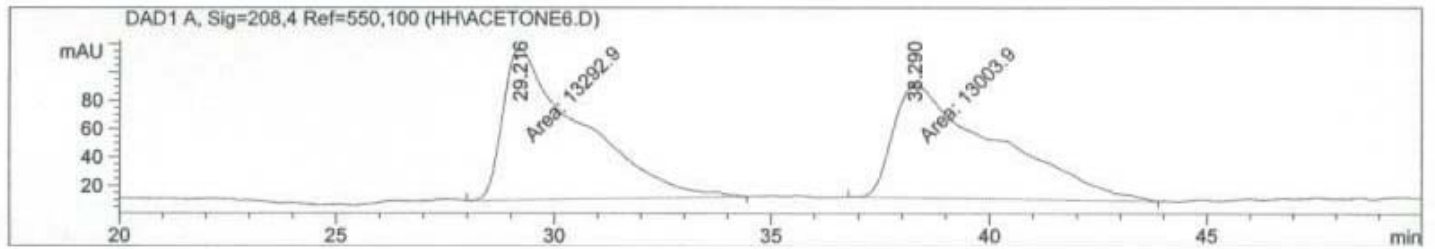


Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	53.440	PB	1.2515	750.42645	7.08618	0.5545
2	64.368	MM	5.0522	1.34585e5	443.98282	99.4455

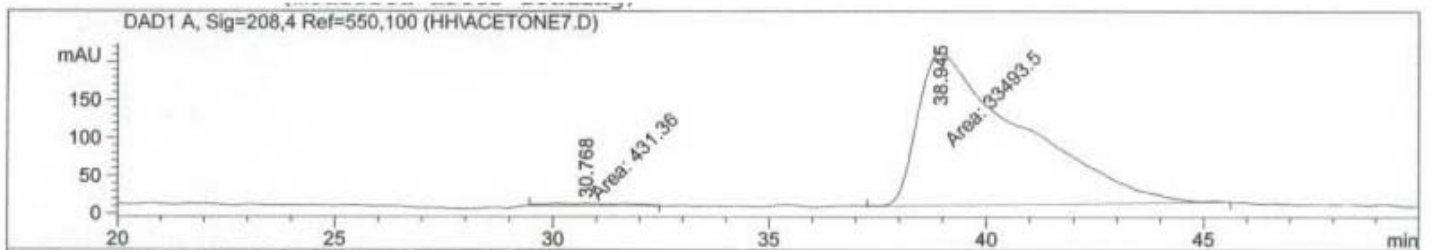
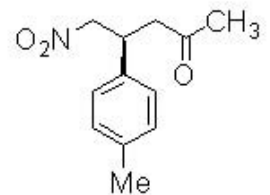
Totals : 1.35336e5 451.06899

OD, 8% IPA/Hex, 0.8 mL/min



Area Percent Report

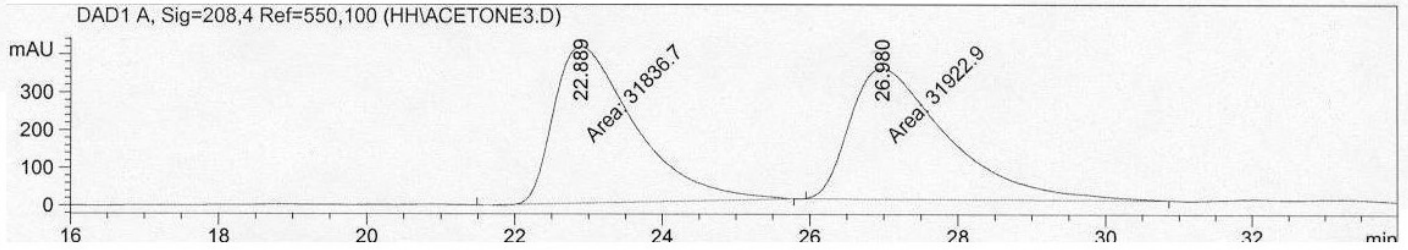
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.216	MM	2.0664	1.32929e4	107.21199	50.5495
2	38.290	MM	2.7132	1.30039e4	79.87917	49.4505
Totals :				2.62967e4	187.09116	



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.768	MM	2.3191	431.35980	3.10005	1.2715
2	38.945	MM	2.7690	3.34935e4	201.59592	98.7285
Totals :				3.39248e4	204.69597	

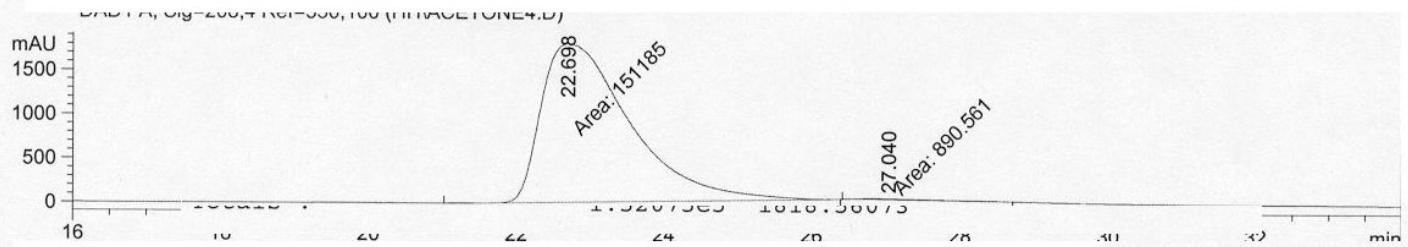
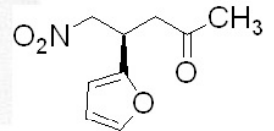
AD, 6% IPA/Hex, 0.6 mL/min



Signal 1: DAD1 A, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.889	MM	1.2808	3.18367e4	414.29419	49.9324
2	26.980	MM	1.5407	3.19229e4	345.32077	50.0676

Totals : 6.37596e4 759.61496

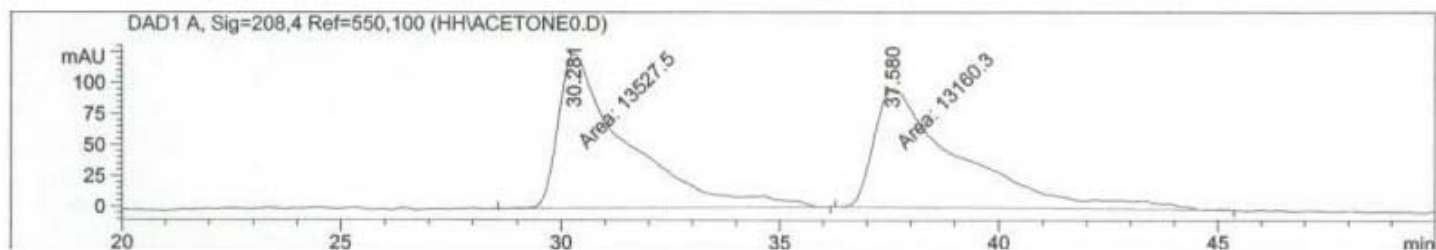


Signal 1: DAD1 A, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.698	MM	1.3936	1.51185e5	1808.12354	99.4144
2	27.040	MM	1.4221	890.56140	10.43719	0.5856

Totals : 1.52075e5 1818.56073

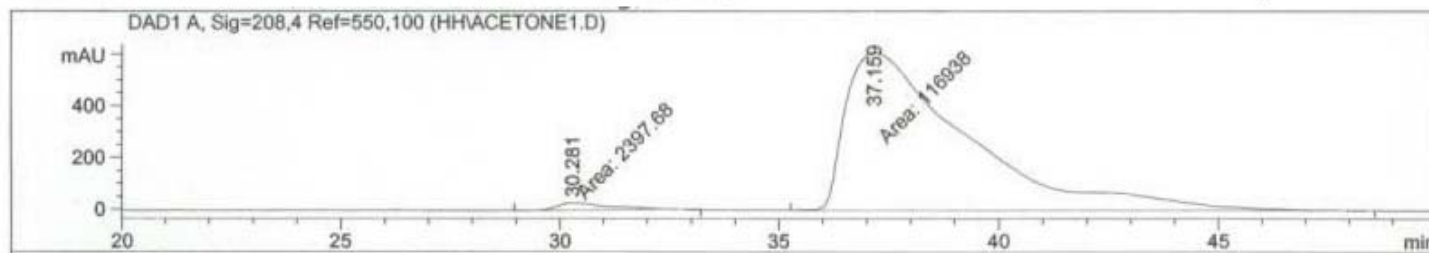
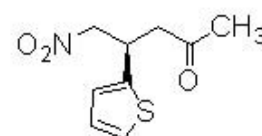
OD, 8% EtOH/Hex, 0.8 mL/min



Area Percent Report

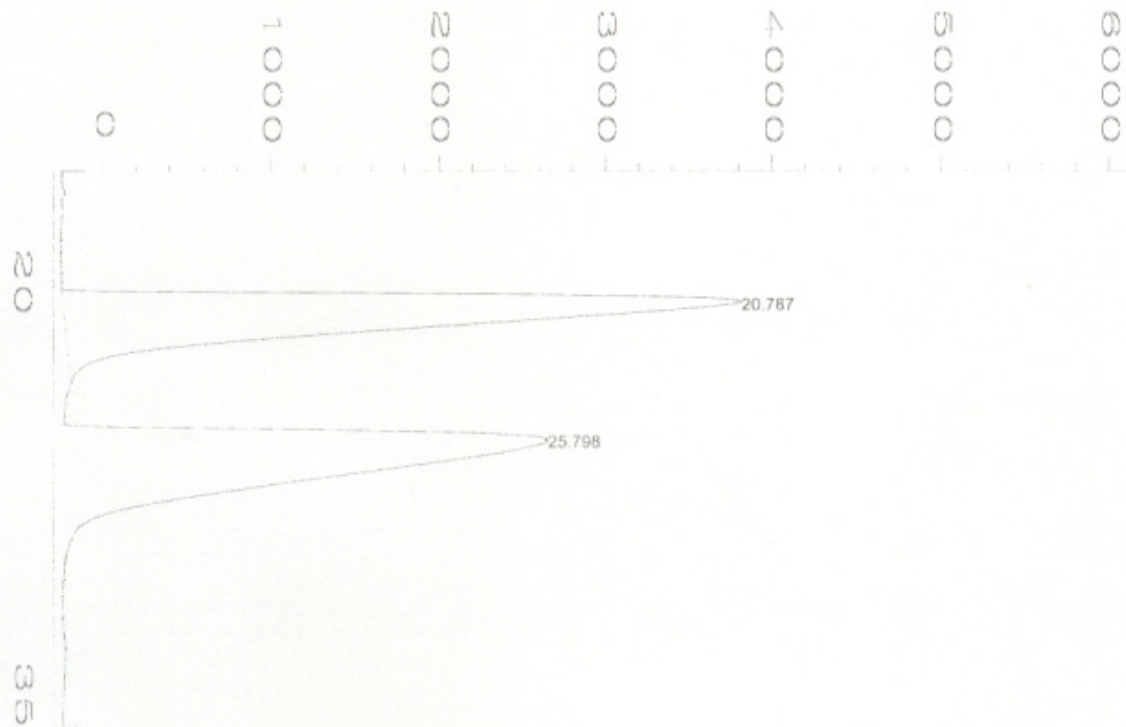
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.281	MM	1.8035	1.35275e4	125.00785	50.6879
2	37.580	MM	2.2809	1.31603e4	96.16085	49.3121

Totals : 2.66877e4 221.16870



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.281	MM	1.3810	2397.67505	28.93632	2.0092
2	37.159	MM	3.1981	1.16938e5	609.41809	97.9908

Totals : 1.19336e5 638.35441

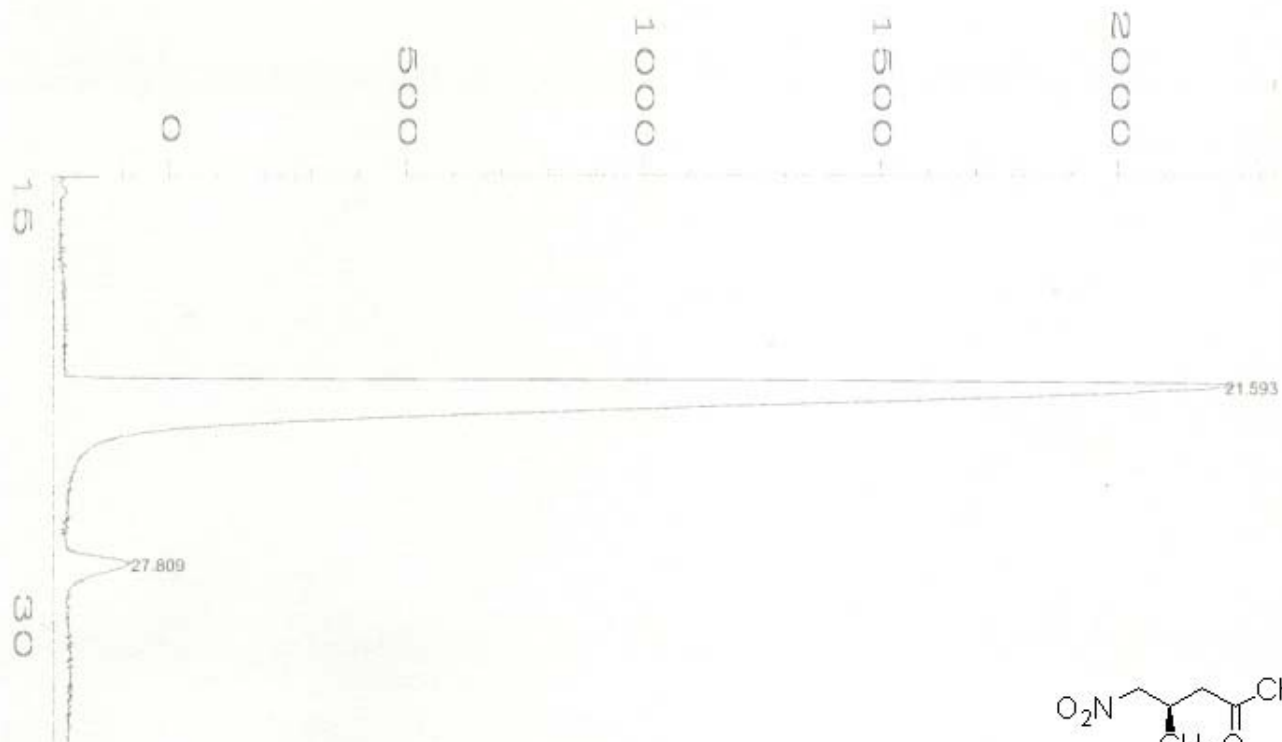


=====  
 Area Percent Report  
 =====

Sig. 1 in C:\PEAK\EXPORT2\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	20.787	322072	4058	BF	0.945	50.2808
2	25.798	318474	2862	MM	1.854	49.7192

Total area = 640546

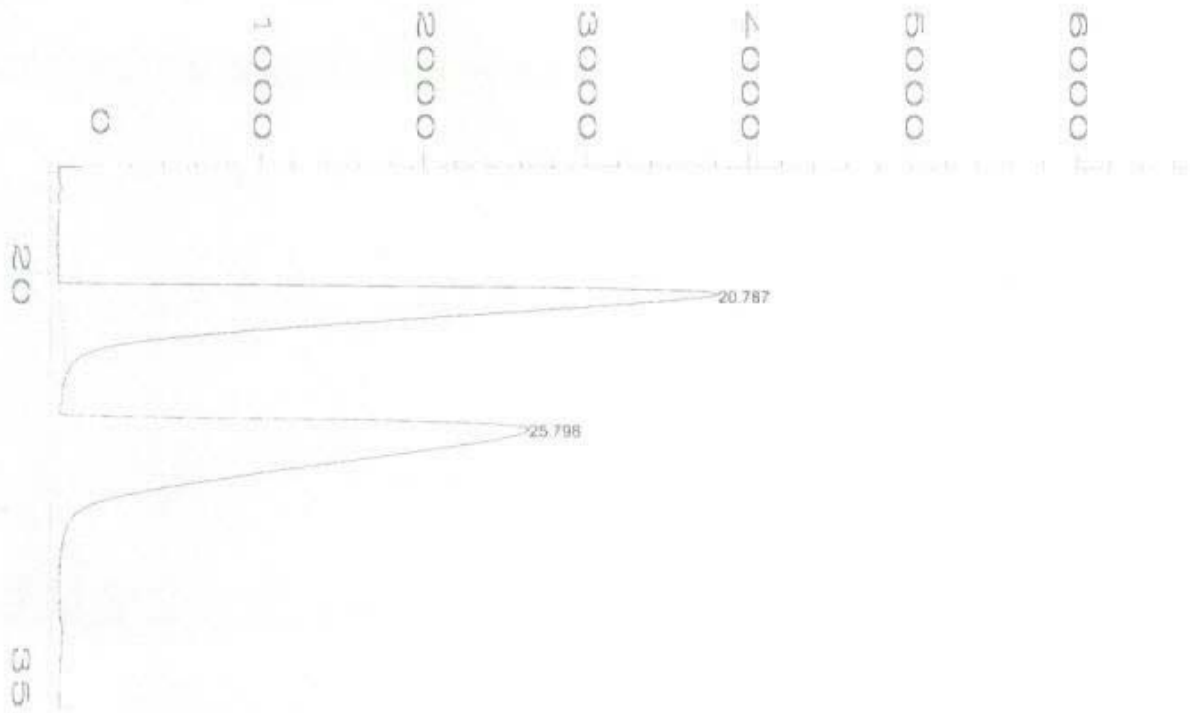


=====  
 Area Percent Report  
 =====

Sig. 1 in C:\PEAK\EXPORT2\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	21.593	158702	2467	MM	1.072	96.6305
2	27.809	5534	137	MM	0.673	3.3695

Total area = 164236



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

Data File Name : C:\PEAK\EXPORT2\SIGNAL.D  
 Operator :  
 Instrument : HP5890A  
 Sample Name :  
 Run Time Bar Code:  
 Acquired on : JAN 15, 1905 10:43:55  
 Report Created on: 15 Jan 05 11:45 AM

Page Number : 1  
 Vial Number : 0  
 Injection Number :  
 Sequence Line :  
 Instrument Method:  
 Analysis Method : QC15MIN.MTH

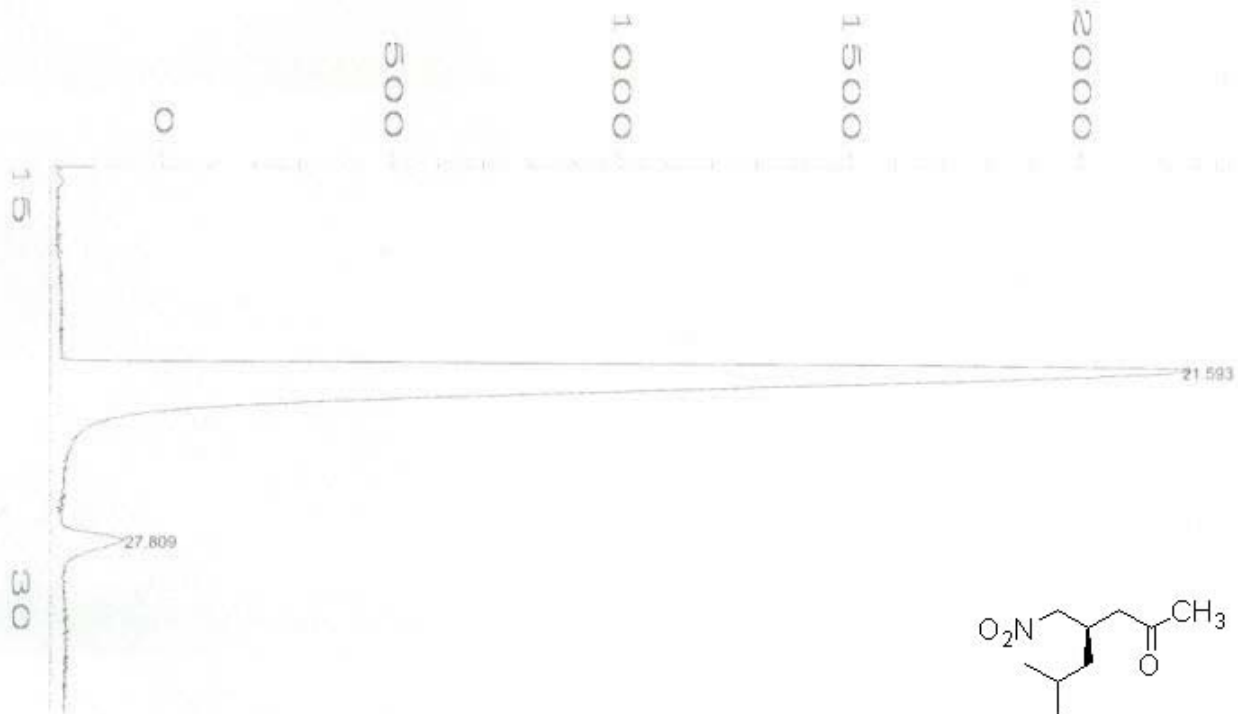
Sig. 1 in C:\PEAK\EXPORT2\SIGNAL.D

Pk#	Ret Time	Area	Height	Type	Width	Area %
1	20.787	322072	4058	BF	0.945	50.2808
2	25.798	318474	2862	MM	1.854	49.7192

Total area = 640546

User Modified

=====



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

Data File Name : C:\PEAK\EXPORT2\SIGNAL.D  
 Operator :  
 Instrument : HP5890A  
 Sample Name :  
 Run Time Bar Code:  
 Acquired on : JAN 15, 1905 11:49:33  
 Report Created on: 15 Jan 05 12:29 PM

Page Number : 1  
 Vial Number : 0  
 Injection Number :  
 Sequence Line :  
 Instrument Method:  
 Analysis Method : QC15MIN.MTH

Sig. 1 in C:\PEAK\EXPORT2\SIGNAL.D

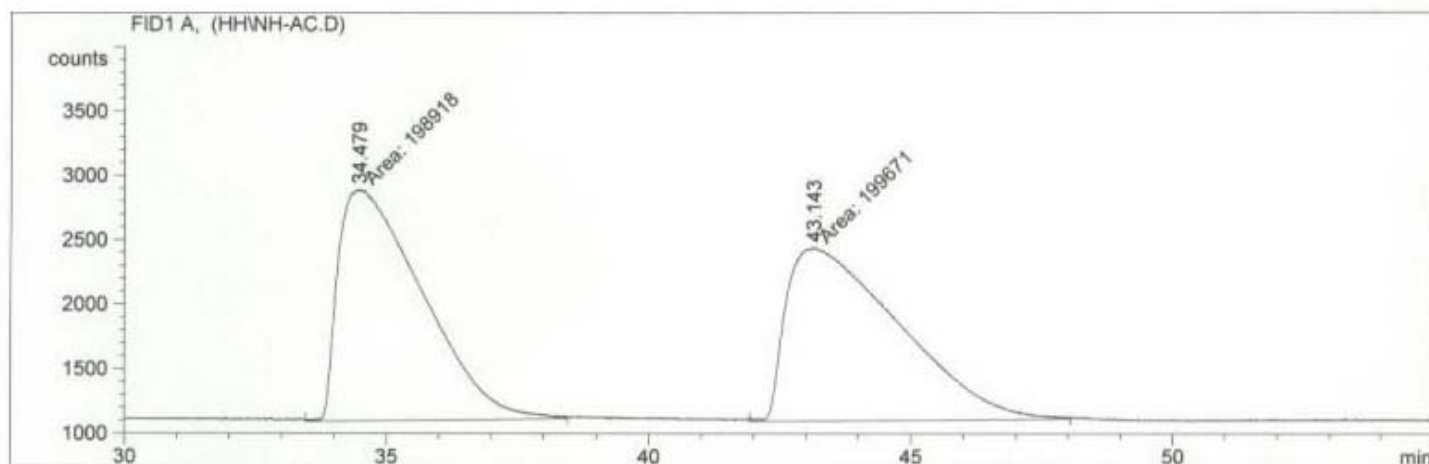
Pk#	Ret Time	Area	Height	Type	Width	Area %
1	21.593	158702	2467	MM	1.072	96.6305
2	27.809	5534	137	MM	0.673	3.3695

Total area = 164236

User Modified

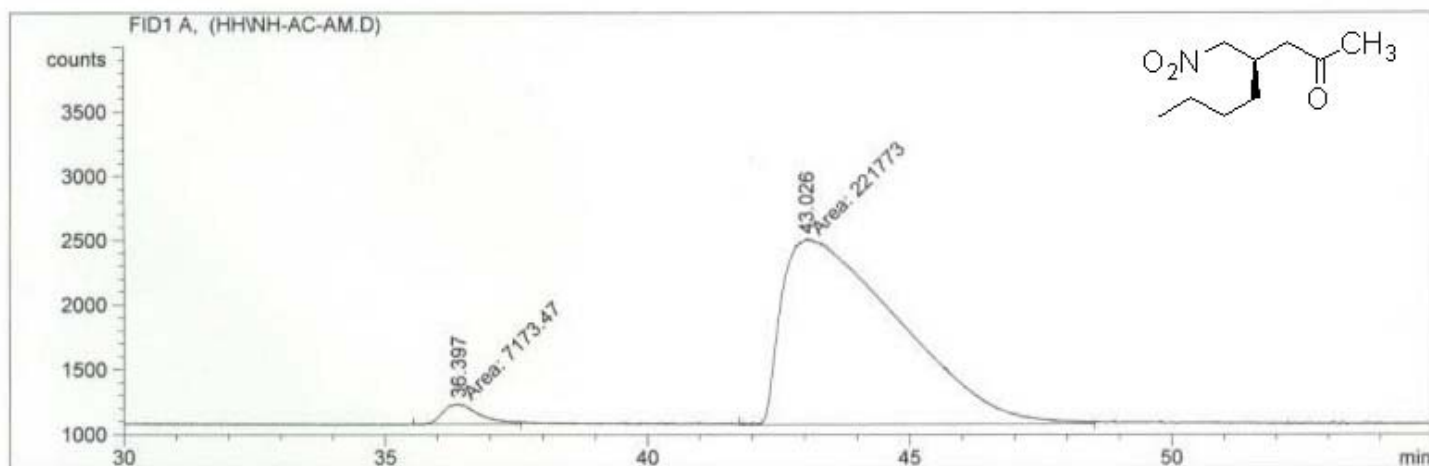
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gamma TA, 110 isothermic, front column



Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	34.479	MM	1.8498	1.98918e5	1792.21704	49.90562
2	43.143	MM	2.4814	1.99671e5	1341.12720	50.09438

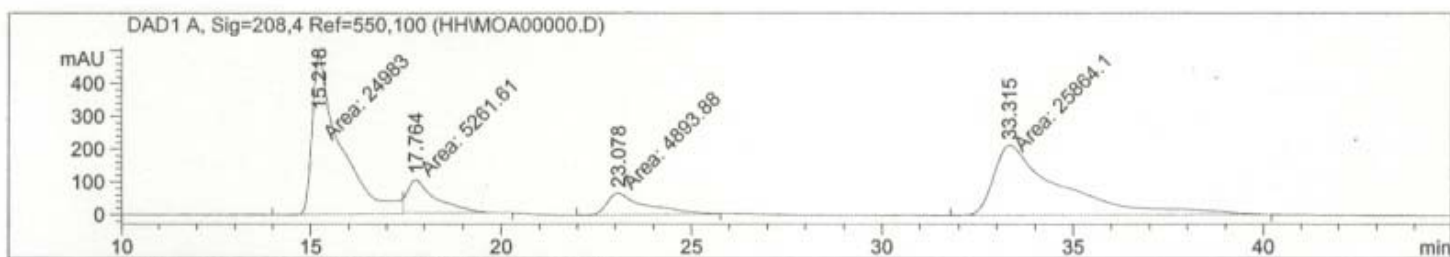
Totals : 3.98589e5 3133.34424



Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	36.397	MM	0.7806	7173.47412	153.15279	3.13325
2	43.026	MM	2.5787	2.21773e5	1433.36658	96.86675

Totals : 2.28947e5 1586.51936

OD, 10% EtOH/Hex, 0.7 mL/min

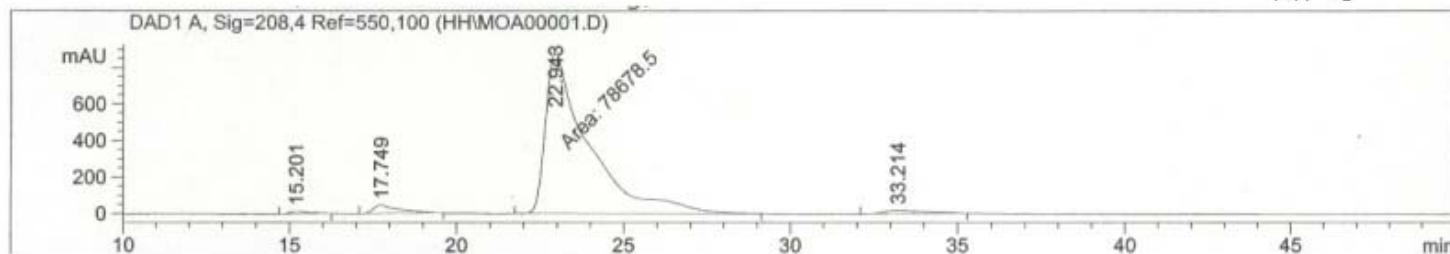
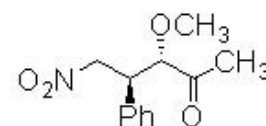


Area Percent Report

Signal 1: DAD1 A, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.218	MM	0.8594	2.49830e4	484.51413	40.9540
2	17.764	MM	0.8697	5261.61133	100.82719	8.6252
3	23.078	MM	1.2216	4893.88330	66.77068	8.0224
4	33.315	MM	2.0332	2.58641e4	212.01376	42.3983

Totals : 6.10026e4 864.12577



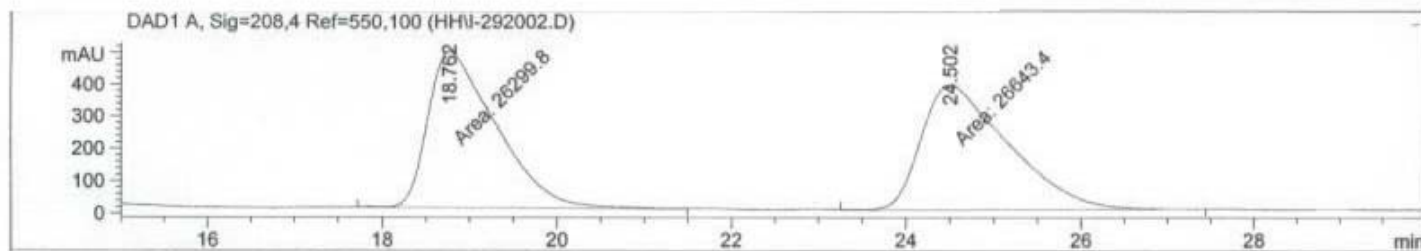
Area Percent Report

Signal 1: DAD1 A, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.201	PB	0.4882	528.36194	12.94510	0.6337
2	17.749	VB	0.6963	2526.48437	45.18521	3.0303
3	22.943	MM	1.4911	7.86785e4	879.42926	94.3682
4	33.214	BB	1.0189	1640.60608	19.23919	1.9678

Totals : 8.33740e4 956.79876

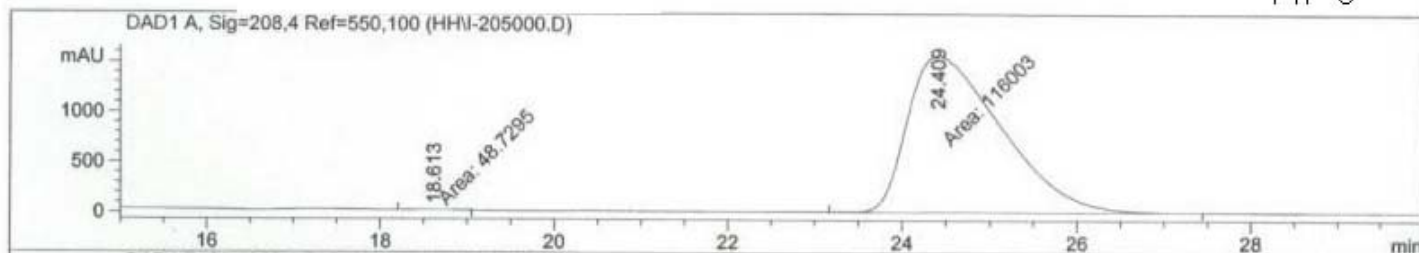
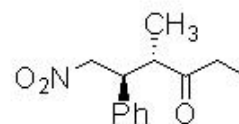
OD, 15% ipa/hex, 0.6 mL/min



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.762	MM	0.9027	2.62998e4	485.58133	49.6755
2	24.502	MM	1.1492	2.66434e4	386.40411	50.3245

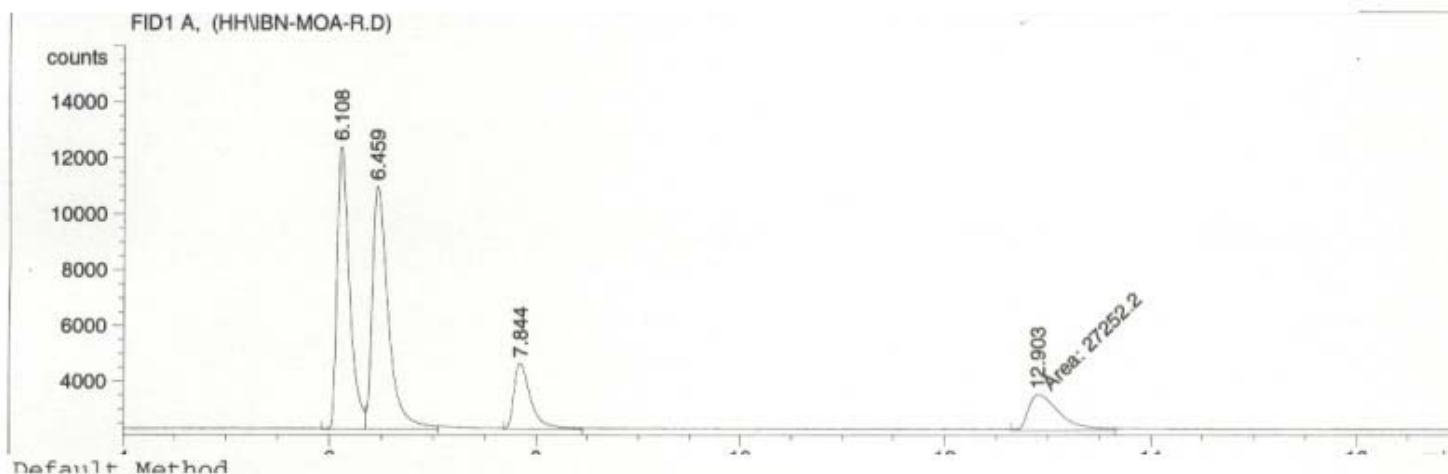
Totals : 5.29433e4 871.98544



Area Percent Report

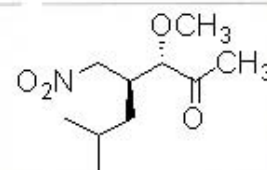
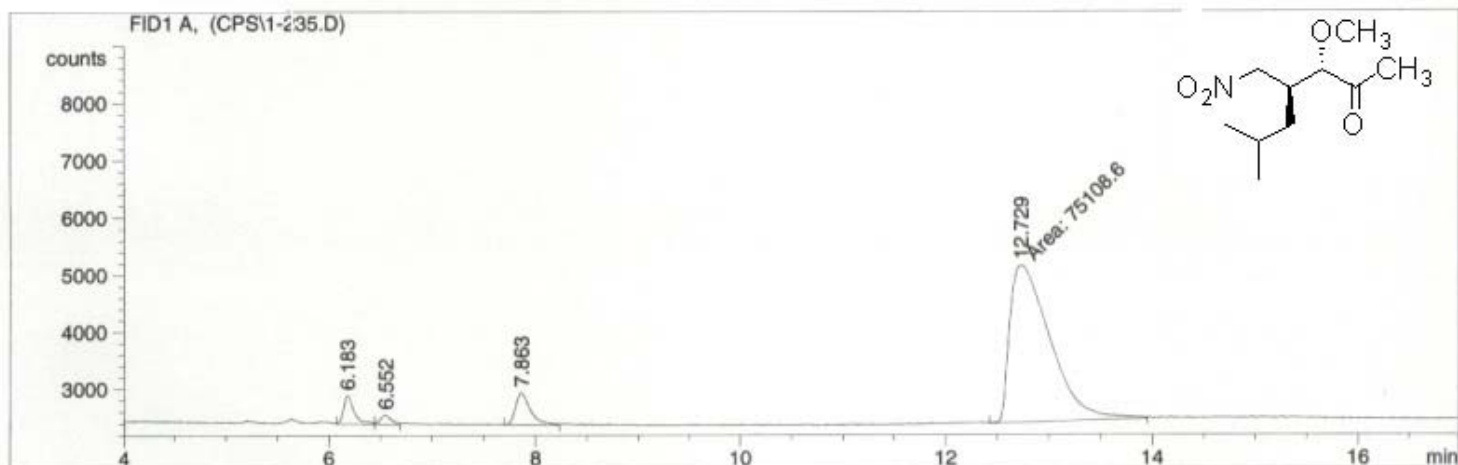
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	18.613	MM	0.4716	48.72953	1.72220	0.0420
2	24.409	MM	1.2357	1.16003e5	1564.64490	99.9580

Totals : 1.16052e5 1566.36710



Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	6.108	VV	0.1245	8.45588e4	1.01182e4	36.49162
2	6.459	VV	0.1516	9.30046e4	8709.16602	40.13643
3	7.844	VV	0.1576	2.69056e4	2367.97339	11.61119
4	12.903	MM	0.3530	2.72522e4	1286.87415	11.76076

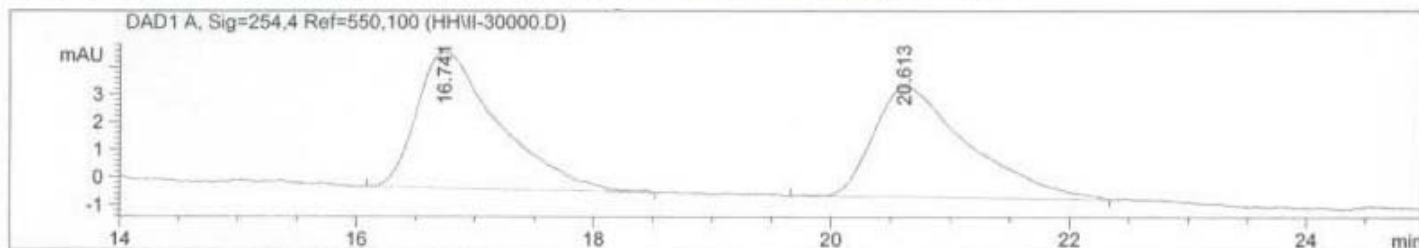
Totals : 2.31721e5 2.24823e4



Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	6.183	VV	0.1041	3572.90186	489.54092	4.18672
2	6.552	VV	0.0926	1141.19470	155.53624	1.33725
3	7.863	VV	0.1217	5516.20850	561.30994	6.46388
4	12.729	MM	0.4525	7.51086e4	2766.36792	88.01215

Totals : 8.53389e4 3972.75502

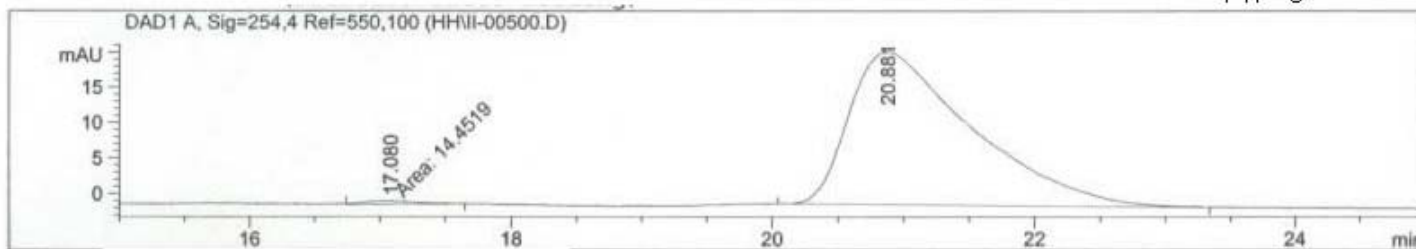
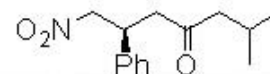
OD, 10% IPA/hex, 1.0 mL/min



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	16.741	BV	0.6234	236.73672	4.96291	51.7190
2	20.613	BV	0.6789	221.00014	3.98975	48.2810

Totals : 457.73686 8.95266

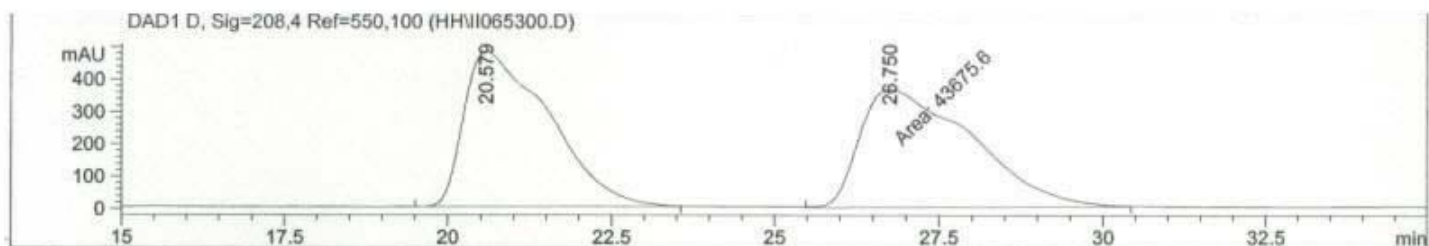


Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	17.080	MM	0.5279	14.45186	4.56298e-1	1.0247
2	20.881	BV	0.8874	1395.83899	21.53515	98.9753

Totals : 1410.29085 21.99145

10% ipa/hex, 1 mL/min, OD

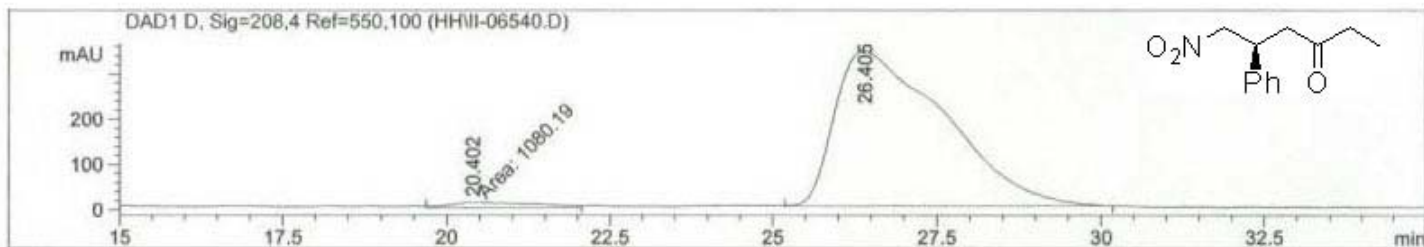


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Area Percent Report  
=====

Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.579	PV	1.0650	4.33025e4	480.00055	49.7855
2	26.750	MM	1.9849	4.36756e4	366.73294	50.2145

Totals :                                    8.69781e4    846.73349



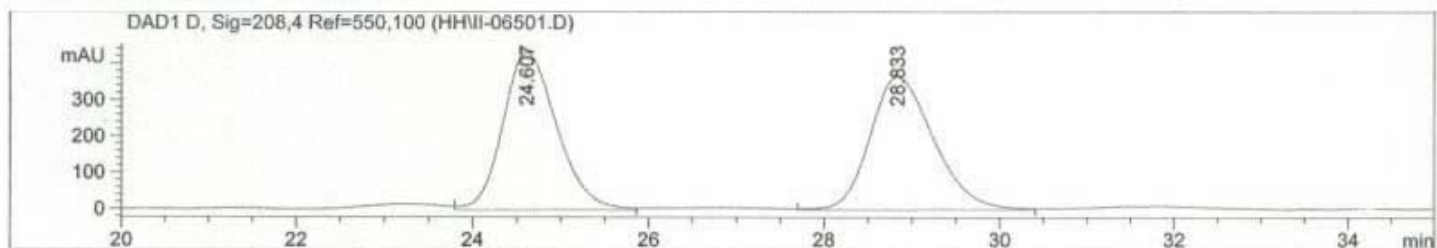
=====  
Area Percent Report  
=====

Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.402	MM	1.5678	1080.19092	11.48280	2.6390
2	26.405	VV	1.3787	3.98512e4	342.39297	97.3610

Totals :                                    4.09314e4    353.87577

10% ipa/hex, 1 mL/min, OD

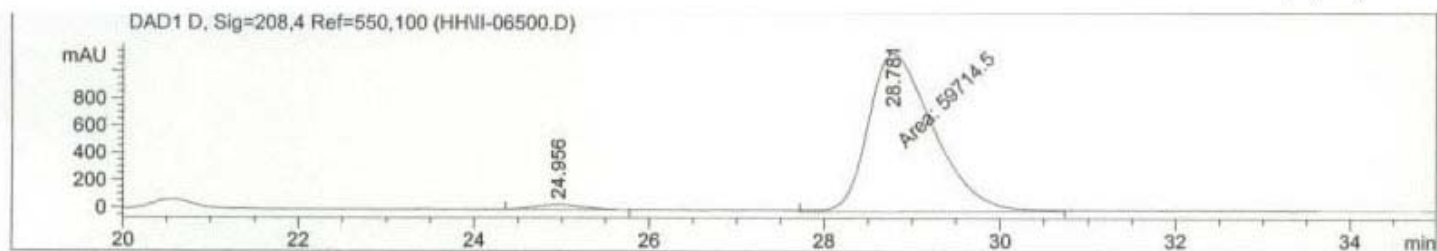
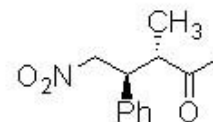


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Area Percent Report  
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Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.607	VV	0.6133	1.85121e4	433.48022	49.8917
2	28.833	VV	0.7499	1.85925e4	367.60391	50.1083

Totals :                    3.71046e4    801.08414



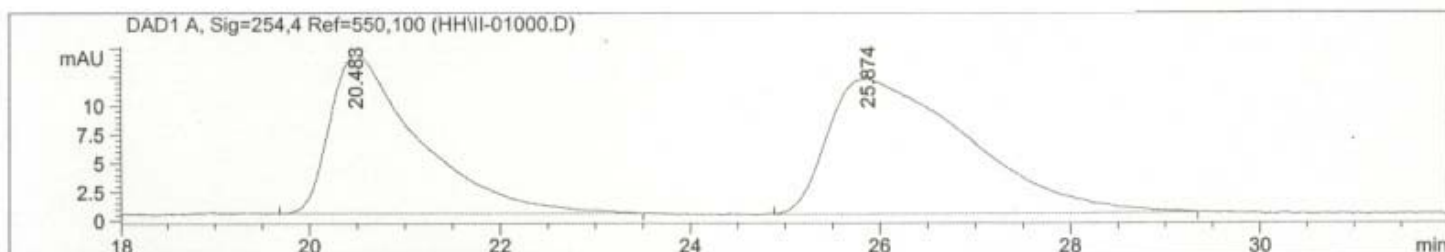
=====  
Area Percent Report  
=====

Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.956	VV	0.4924	1418.16736	35.52486	2.3198
2	28.781	MM	0.8589	5.97145e4	1158.70398	97.6802

Totals :                    6.11327e4    1194.22884

OD, 10% ipa/hex, 1.0 mL/min

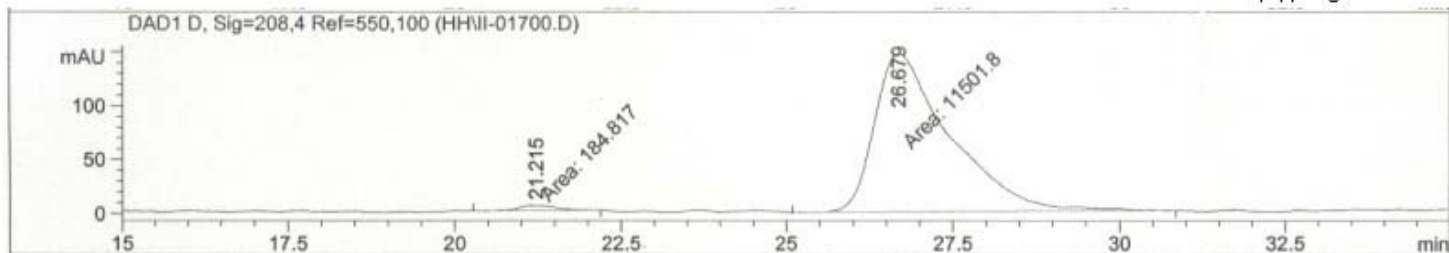
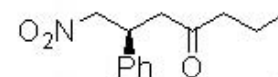


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Area Percent Report  
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Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	20.489	MM	1.0833	4.46746e4	687.29510	50.9226
2	25.770	VV	1.0288	4.30558e4	545.30322	49.0774

Totals :                                    8.77304e4    1232.59833



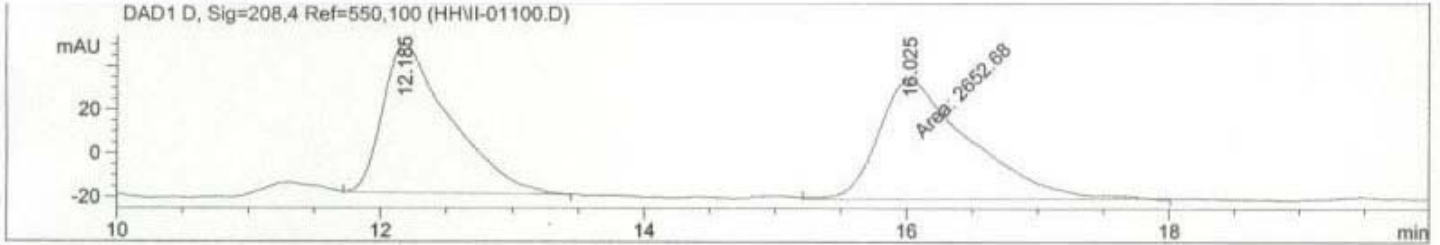
=====  
Area Percent Report  
=====

Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	21.215	MM	0.6185	184.81703	4.98020	1.5814
2	26.679	MM	1.3092	1.15018e4	146.42561	98.4186

Totals :                                    1.16866e4    151.40582

OD, 10% ipa/hex, 1.0 mL/min

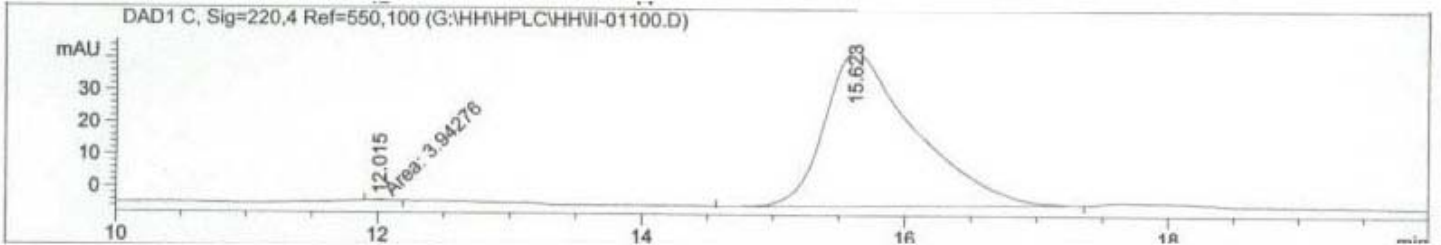
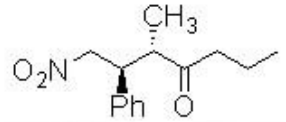


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Area Percent Report  
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Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.185	PV	0.4885	2413.46436	68.30708	47.6391
2	16.025	MM	0.8044	2652.68115	54.96444	52.3609

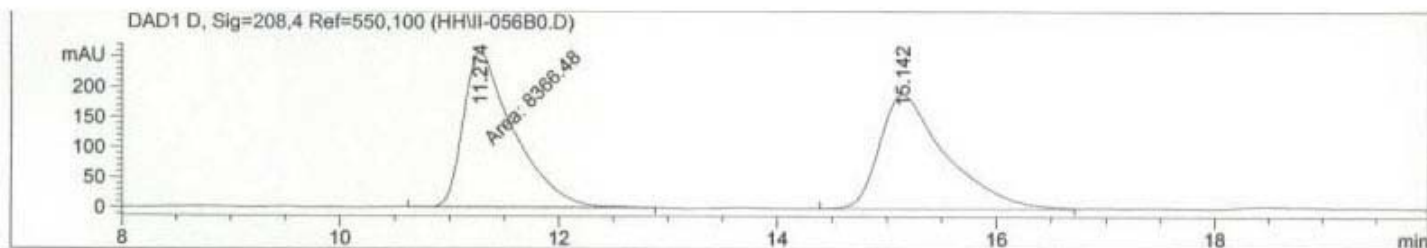
Totals :                                    5066.14551    123.27152



=====  
Area Percent Report  
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Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.015	MM	0.1981	3.94276	3.31634e-1	0.1716
2	15.623	PV	0.6516	2294.07373	48.26081	99.8284

10% ipa/hex, 1 mL/min, OD

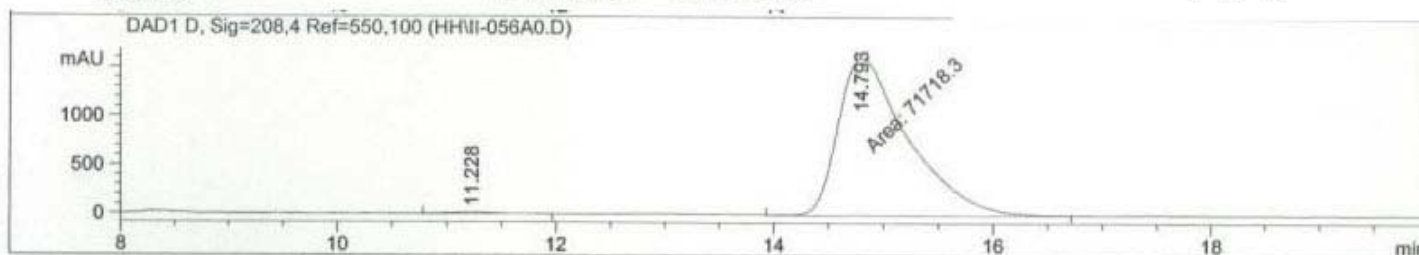
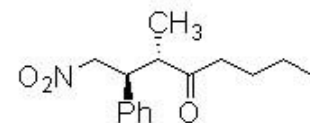


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Area Percent Report  
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Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.274	MM	0.5399	8366.47559	258.28879	50.9729
2	15.142	VV	0.5788	8047.09766	190.41586	49.0271

Totals : 1.64136e4 448.70465



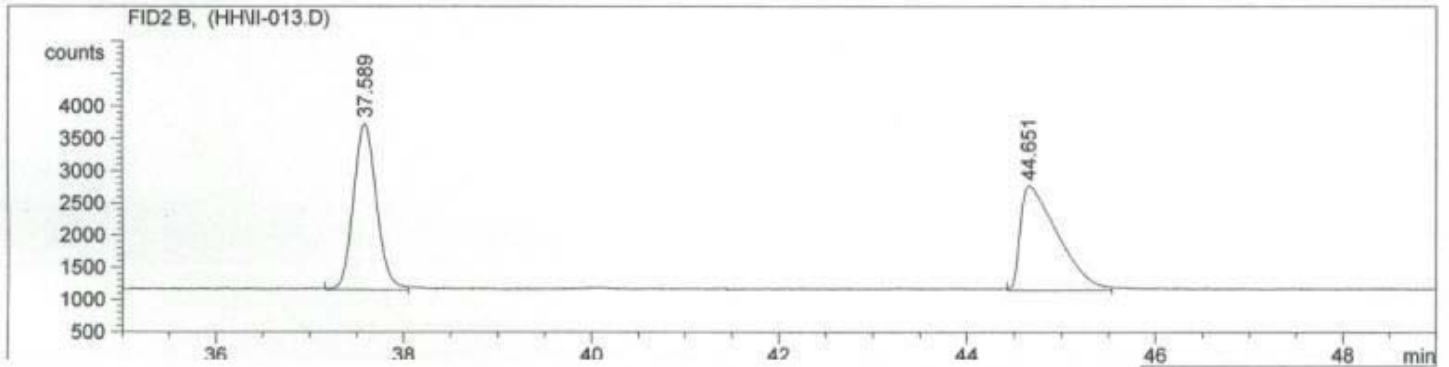
=====  
Area Percent Report  
=====

Signal 3: DAD1 D, Sig=208,4 Ref=550,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.228	VV	0.4008	527.28949	16.21629	0.7299
2	14.793	MM	0.7419	7.17183e4	1611.21106	99.2701

Totals : 7.22456e4 1627.42735

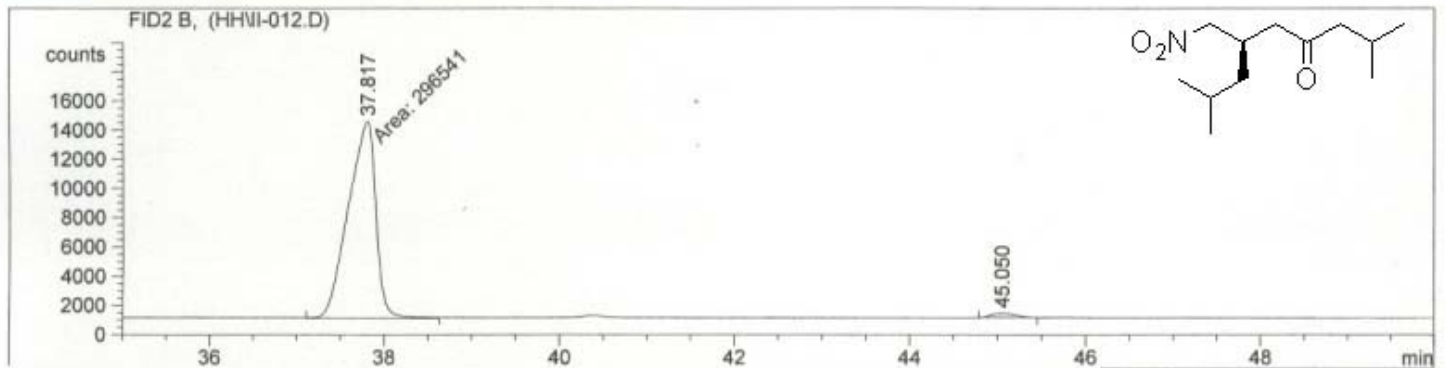
g-TA, 120 isothermal, signal2



Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	37.589	VV	0.2031	4.40940e4	2584.13892	49.45674
2	44.651	VV	0.3291	4.50627e4	1627.39514	50.54326

Totals : 8.91567e4 4211.53406

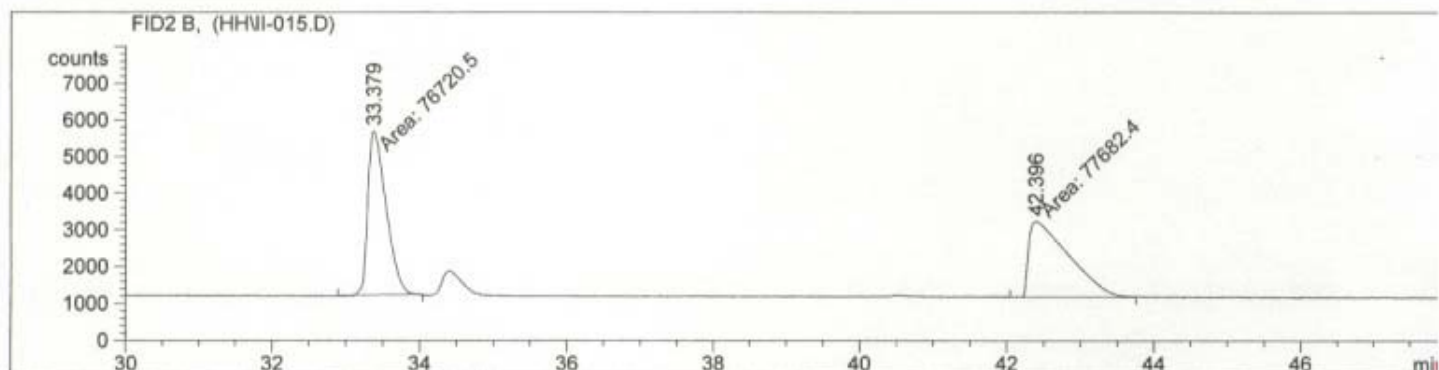


Area Percent Report

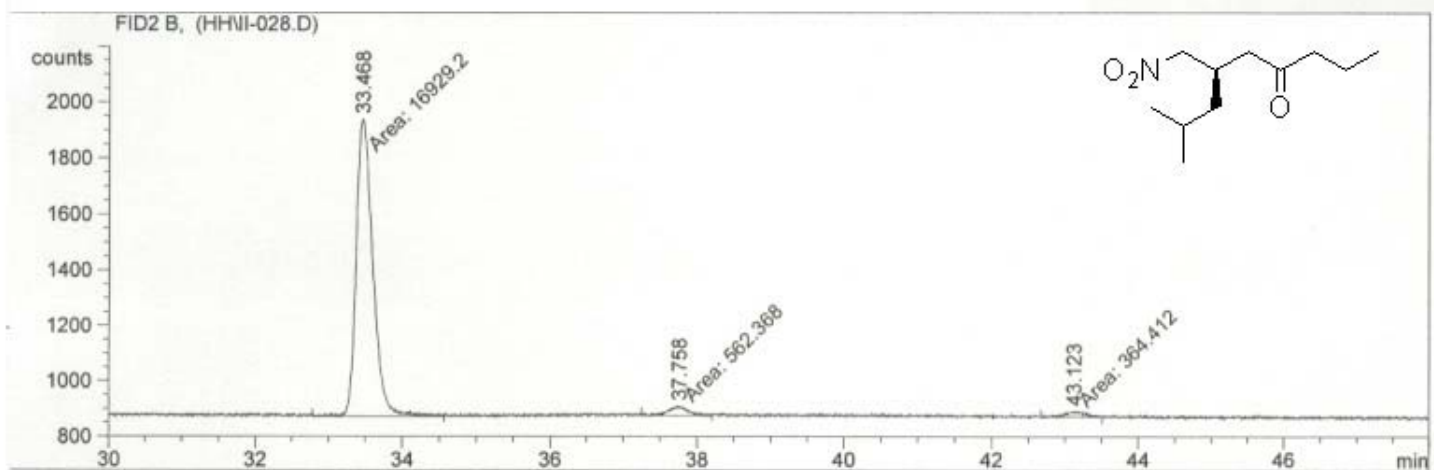
Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	37.817	MM	0.3666	2.96541e5	1.34831e4	98.39439
2	45.050	VV	0.2140	4838.98389	269.15033	1.60561

Totals : 3.01380e5 1.37522e4

g-TA, 120 isothermal



Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	33.379	MM	0.2864	7.67205e4	4465.04443	49.68851
2	42.396	MM	0.6301	7.76824e4	2054.86279	50.31149
Totals :				1.54403e5	6519.90723	



Peak #	RetTime [min]	Type	Width [min]	Area counts*s	Height [counts]	Area %
1	33.468	MM	0.2643	1.69292e4	1067.37781	94.80969
2	37.758	MM	0.2877	562.36786	32.58337	3.14947
3	43.123	MM	0.3032	364.41156	20.02933	2.04084
Totals :				1.78560e4	1119.99050	