Highly Enantioselective Michael Addition of Aromatic Ketones to Nitroolefins Promoted by Chiral Bifunctional Primary Amine-Thiourea Catalysts Based on Saccharides

Kun Liu, Han-Feng Cui, Jing Nie, Ke-Yan Dong, Xiao-Juan Li, Jun-An Ma*

*Department of Chemistry,
Tianjin University, Tianjin 300072, China
Majun_an68@tju.edu.cn*

Supporting Information

Table of Contents

Experimental Section	Page 1 — Page 5
Copies of HPLC data	Page 6 — Page 19
Copies of ^1H and ^{13}C NMR Spectra	Page 20 — Page 28

General information: ^1H and ^{13}C NMR were recorded on Bruker-300 and Varian-500 instruments. Chemical shifts were reported in ppm down field from internal Me_4Si . Optical rotations were determined using a Perkin Elmer Model 341 polarimeter. LRMS were recorded on a VG-7070E spectrometer. HRMS were done on a Bruker APEX III FTMS (7.0 TESLA). Chiral HPLC analyses were carried out on a Hewlett Packard Model HP 1200 instrument. All reactions were carried out in oven-dried glassware under air atmosphere unless otherwise noted.

Materials: All commercially available solvents and reagents were used as received unless otherwise noted. Analytical thin layer chromatography was performed on 0.20 mm Qingdao Haiyang silica gel plates. Silica gel (200-300 mesh) (from Qingdao Haiyang Chem. Company, Ltd.) was used for flash chromatography. Three saccharidyl isothiocyanates **1** were obtained following the literature procedure.^[1]

Preparation of catalysts:

To a solution of 1, 2-cyclohexyldiamine (3.6 mmol) in dichloromethane (20 mL) was added the corresponding sugar-derived isothiocyanates **1** (3 mmol). The mixture was stirred at room temperature for 3-24 h (TLC) and concentrated. The resulting residue was chromatographed with the eluent (AcOEt / Et₃N: 100 / 1) to give the crude solid. The crude solid was dissolved in a minimal amount of dichloromethane and slowly precipitated from solution by the addition of petroleum at 0 °C. Filtration afforded the desired thiourea products **2**.

Yield 60%, $[\alpha]_D^{20}$ -38.2° (c 1.0, CHCl₃), ¹H NMR (500 MHz, CDCl₃) δ0.88-0.95 (m, 1H, cyclohexane-H), 1.24-1.27 (m, 5H, cyclohexane-H), 1.72-1.75 (m, 2H, cyclohexane-H), 2.00-2.09 (m, 14H, 4CH₃, NHC(S)NH), 2.62 (br, 1H, NH), 3.18 (br, 1H, NH), 3.48 (m, 1H, cyclohexane-H), 3.82-3.85 (m, 1H, cyclohexane-H), 4.06-4.14 (m, 2H, CH₂), 4.28-4.33 (m, 1H, pyranose-H), 5.03-5.11(m, 2H, pyranose-H), 5.30-5.34 (m, 1H, pyranose-H), 5.81-5.82 (m, 1H, pyranose-H); ¹³C NMR (125 MHz, CDCl₃) δ180.1, 170.9, 170.5, 170.1, 169.8, 83.0, 73.7, 73.5, 70.9, 68.4, 61.8, 60.6, 53.6, 35.0, 32.1, 31.1, 24.7, 21.1, 21.0, 20.9, 20.8; IR (KBr): ν 3349, 2929, 1752, 1542, 1373, 1229, 1035, 907, 758, 654 cm⁻¹. MS (ESI): m/z 504.31 (M⁺+1). HRMS (MALDI): Calculated for [C₂₁H₃₃N₃O₉S+H]: 504.2025, found 504.2023.

Yield 61%, $[\alpha]_D^{20}$ +43.1° (c 1.0, CHCl₃), ¹H NMR (500 MHz, CDCl₃) δ0.88-0.95 (m, 1H, cyclohexane-H), 1.24-1.28 (m, 5H, cyclohexane-H), 1.72-1.74 (m, 2H, cyclohexane-H), 2.00-2.10 (m, 14H, 4CH₃, NHC(S)NH), 2.62 (br, 1H, NH), 3.19 (br, 1H, NH), 3.45-3.49 (m, 1H, cyclohexane-H), 3.82-3.85 (m, 1H, cyclohexane-H), 4.06-4.33 (m, 3H, CH₂, pyranose-H), 5.03-5.10(m, 2H, pyranose-H), 5.30-5.34 (m, 1H, pyranose-H), 5.81-5.83 (m, 1H, pyranose-H); ¹³C NMR (125 MHz, CDCl₃) δ179.1, 170.9, 170.5, 170.1, 169.8, 83.0, 73.7, 73.5, 70.9, 68.4, 61.8,

60.6, 53.6, 35.0, 32.1, 31.1, 24.7, 21.1, 21.0, 20.9, 20.8; IR (KBr): ν 3349, 2929, 1752, 1542, 1373, 1229, 1035, 907, 758, 654 cm^{-1} . MS (ESI): m/z 504.30 (M^++1). HRMS (MALDI): Calculated for $[\text{C}_{21}\text{H}_{33}\text{N}_3\text{O}_9\text{S}+\text{H}]$: 504.2025, found 504.2024.

Yield 60%, $[\alpha]_D^{20} +103.6^\circ$ (c1.0, CHCl_3), ^1H NMR (500 MHz, CDCl_3) δ 1.24-1.27 (m, 5H, cyclohexane-H), 1.73-1.80 (m, 3H, cyclohexane-H), 1.97-2.16 (m, 23H, 7CH₃, NHC(S)NH), 3.19 (br, 2H, NH₂), 3.40-3.46 (m, 1H, cyclohexane-H), 3.69-3.90 (m, 4H, pyranose-3H, cyclohexane-H), 4.05-4.22 (m, 5H, pyranose-H), 4.47-4.53 (m, 1H, pyranose-H), 4.66-4.74 (m, 1H, pyranose-H), 4.94-5.02 (m, 1H, pyranose-H), 5.08-5.13 (m, 1H, pyranose-H), 5.20-5.36 (m, 2H, pyranose-H); ^{13}C NMR (125 MHz, CDCl_3) δ 184.2, 171.3, 171.0, 170.5, 170.3, 170.2, 169.7, 169.2, 101.1, 88.4, 76.4, 73.2, 71.9, 71.2, 69.3, 68.5, 66.9, 66.8, 63.9, 61.0, 60.5, 56.4, 33.7, 30.8, 25.1, 24.7, 21.3, 21.2, 21.1, 21.0, 20.9, 20.8, 20.6; IR (KBr): ν 3359, 2931, 2858, 1741, 1541, 1234, 1050, 600 cm^{-1} . MS (ESI): m/z 792.25 (M^++1) (85%), 733.2 (100%), 691.2 (78%), 649.2 (30%). HRMS (MALDI): Calculated for $[\text{C}_{33}\text{H}_{49}\text{N}_3\text{O}_{17}\text{S}]$: 791.2803, found 791.2792.

Yield 73%, $[\alpha]_D^{20} +11.2^\circ$ (c 1.0, CHCl_3), ^1H NMR (500 MHz, CDCl_3) δ 1.22-1.28 (m, 5H, cyclohexane-H), 1.73-1.82 (m, 3H, cyclohexane-H), 1.96-2.16 (m, 23H, 7CH₃, NHC(S)NH), 3.19 (br, 2H, NH₂), 3.40-3.47 (m, 1H, cyclohexane-H), 3.48-3.90 (m, 4H, pyranose-3H, cyclohexane-H), 4.09-4.35 (m, 5H, pyranose-H), 4.42-4.60 (m, 1H, pyranose-H), 4.62-4.79 (m, 1H, pyranose-H), 4.93-5.09 (m, 1H, pyranose-H), 5.10-5.13 (m, 1H, pyranose-H), 5.26-5.36 (m, 2H, pyranose-H); ^{13}C NMR (125 MHz, CDCl_3) δ 192.3, 171.3, 171.2, 170.8, 170.7, 170.0, 169.9, 169.6, 95.7, 82.7, 75.3, 74.1, 73.0, 71.3, 70.2, 69.4, 68.4, 68.1, 62.9, 61.6, 60.6, 53.7, 45.0, 31.0, 30.0, 29.2, 24.7, 27.1, 21.1, 21.0, 21.9, 20.8, 20.7; IR (KBr): ν 3310, 2950, 1751, 1536, 1239,

1031, 600 cm^{-1} . MS (ESI): m/z 792.18 ($M^+ + 1$) (20%), 724.2 (100%), 710.2 (19%), 636.2 (24%), 619.2 (22%). HRMS (MALDI): Calculated for $[\text{C}_{33}\text{H}_{49}\text{N}_3\text{O}_{17}\text{S} + \text{H}]$: 792.2856, found 792.2856.

Yield 70%, $[\alpha]_{\text{D}}^{20} +130.1^\circ$ (c 1.0, CHCl_3), ^1H NMR (500 MHz, CDCl_3) δ 0.80-1.40 (m, 2H, cyclohexane-H), 1.80-2.32 (m, 24H, 6 CH_3 , cyclohexane-H x 6), 4.07 (m, 1H, pyranose-H), 4.10-4.30 (m, 3H, pyranose-CH, cyclohexane-CH x 2), 4.78 (dd, $J = 3.9$ and 10.0 Hz, 1H, pyranose-H), 4.91-5.27 (m, 2H, pyranose-H), 5.48 (t, $J = 9.28$ Hz, 1H, pyranose-H), 6.55 (d, $J = 4.0$ Hz, 1H, pyranose-H); ^{13}C NMR (125 MHz, CDCl_3) δ 180.8, 170.6, 170.0, 169.8, 169.5, 88.8, 74.3, 72.3, 70.7, 70.3, 67.3, 66.4, 61.1, 40.0, 20.81, 20.79, 20.76, 20.70; IR (KBr): ν 3320, 2975, 2942, 2780, 1754, 1541, 1433, 1372, 1236, 1112, 1038, 911, 752, 602 cm^{-1} . MS (ESI): m/z 532.20 ($M^+ + 1$).

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

The nitroolefin (0.3 mmol), aromatic ketone (3.0 mmol) and catalyst **2b** (0.045 mmol) were placed in a 5 mL vial equipped with a Teflon-coated stirbar. The solvent (1 mL) was added under air. The vial was capped with a white polyethylene stopper and the resulting mixture was stirred at room temperature for the stated time. Then the reaction solution was concentrated *in vacuo* and the crude was purified by flash chromatography to afford the product. Compounds **3a**,^[2, 3, 4] **3b**,^[3] **3c**,^[3, 4] **3d**,^[3] **3e**,^[3] **3f**,^[3, 5] **3i**,^[3] **3j**,^[3] **3k**,^[2, 3] and **3n**,^[3] were known.

3g $[\alpha]_{\text{D}}^{20} -22.3^\circ$ (c 1.0, CHCl_3), ^1H NMR (500 MHz, CDCl_3) δ 7.94-7.92 (m, 1H, Ar-H), 7.85-7.74 (m, 4H, Ar-H), 7.59-7.40 (m, 7H, Ar-H), 4.92 (dd, $J = 4.8, 9.3$ Hz, 1H, CH_2), 4.78 (dd, $J = 6.0, 9.6$ Hz, 1H, CH_2), 4.44-4.37 (m, 1H, CH), 3.55 (dd, $J = 5.1, 9.6$ Hz, 2H, CH_2); ^{13}C NMR (75 MHz, CDCl_3) δ 197.0, 136.7, 136.6, 133.8, 133.6, 133.1, 129.2, 129.1, 128.9, 128.3, 128.2, 128.0, 127.9, 126.7, 126.6, 126.4, 125.3, 79.7, 41.8, 39.6; IR (KBr): ν 3063, 2904, 1675, 1542, 1357, 1209, 748, 481 cm^{-1} . MS (ESI): m/z 318.6 (M^+) (10%), 105 (100%), 77 (56%), 51 (25%). HRMS (MALDI): Calculated for $[\text{C}_{20}\text{H}_{17}\text{NO}_3]$: 319.1208, found 319.1208.

3h $[\alpha]_{\text{D}}^{20} -14.0^\circ$ (c 1.0, CHCl_3), ^1H NMR (300 MHz, CDCl_3) δ 7.94(m, 2H), 7.60(m, 1H), 7.50(m, 2H), 7.34(m, 1H), 6.20(d, $J = 3$ Hz, 2H), 4.77(m, 2H), 4.33(m, 1H), 3.48(m, 2H); ^{13}C NMR (75 MHz, CDCl_3) δ 196.8, 152.2, 142.5, 136.5, 133.9, 129.0 (2 $\times$ C), 128.3 (2 $\times$ C), 110.7, 107.4, 77.6,

39.2, 33.4; IR (KBr): ν 3063, 2914, 1675, 1557, 1388, 1199, 753, 599 cm^{-1} . MS (ESI): m/z 259.0 (M^+).

3l $[\alpha]_D^{20}$ -30.0° (c 0.1, CHCl_3), ^1H NMR (500 MHz, CDCl_3) δ 7.91 (d, J = 5.7 Hz, 1H, Ar-H), 7.61-7.25 (m, 8H, Ar-H), 4.86-4.79 (m, 1H, CH_2), 4.71-4.66 (m, 1H, CH_2), 4.25-4.18 (m, 1H, CH), 3.47-3.39 (m, 2H, CH_2); ^{13}C NMR (75 MHz, CDCl_3) δ 197.0, 151.0, 139.3, 136.6, 133.8, 132.3, 129.7, 129.3 (2 $\times$ C), 129.0, 128.3, 128.1, 127.7, 79.8, 41.7, 39.5; IR (KBr): ν 3062, 2924, 1685, 1574, 1383, 1234, 753, 692 cm^{-1} . MS (ESI): m/z 346.8 (M^+ -1) (45%), 318.6 (100%), 179.1 (36%), 101.1 (25%).

3m $[\alpha]_D^{20}$ -29.4° (c 1.0, CHCl_3), ^1H NMR (500 MHz, CDCl_3) δ 7.85 (d, J = 6.3 Hz, 1H, Ar-H), 7.44-7.22 (m, 8H, Ar-H), 4.94-4.77 (m, 1H, CH_2), 4.71-4.66 (m, 1H, CH_2), 4.22-4.17 (m, 1H, CH), 3.45-3.41 (m, 2H, CH_2); ^{13}C NMR (75 MHz, CDCl_3) δ 195.8, 140.3, 139.1, 134.9, 132.3, 130.8, 129.6, 129.3, 129.2, 128.2, 127.8, 127.6, 127.2, 79.7, 41.7, 39.5; IR (KBr): ν 3088, 3017, 2909, 1680, 1552, 1378, 1204, 978, 692, 564 cm^{-1} . MS (ESI): m/z 303.2 (M^+) (100%), 243.1 (85%), 225.3 (20%).

Optical rotation and chiral-phase HPLC data

Product	$[\alpha]_D^{20}$	Daicel Chiralpak (AD-H)
3a	-23.0° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 16.4 min (major), 23.2 min
3b	-22.0° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 20/80, 254 nm, 1.0 mL/min, t_R = 9.3 min (major), 12.2 min
3c	-21.1° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 20/80, 254 nm, 1.0 mL/min, t_R = 13.8 min (major), 18.9 min
3d	-23.3° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 17.6 min (major), 25.9 min
3e	-26.1° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 15/85, 254 nm, 1.0 mL/min, t_R = 12.0 min (major), 14.5 min
3f	-29.4° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 15/85, 254 nm, 1.0 mL/min, t_R = 11.1 min (major), 13.4 min
3g	-22.3° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 23.3 min (major), 29.0 min
3h	-14.0° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 12.1 min (major), 14.7 min
3i	-2.4° (c0.8, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 6.7 min (major), 7.3 min
3j	-32.1° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 18.5 min (major), 30.7 min
3k	-28.4° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 15/85, 254 nm, 1.0 mL/min, t_R = 24.3 min (major), 39.7 min
3l	-30.0° (c0.1, CHCl_3)	<i>i</i> -PrOH/hexane: 15/85, 254 nm, 1.0 mL/min, t_R = 12.2 min (major), 16.6 min 18.9 min (major), 24.8 min
3m	-29.4° (c1.0, CHCl_3)	<i>i</i> -PrOH/hexane: 15/85, 254 nm, 1.0 mL/min, t_R = 16.7 min (major), 22.2 min
3n	-58.7° (c0.8, CHCl_3)	<i>i</i> -PrOH/hexane: 10/90, 254 nm, 1.0 mL/min, t_R = 25.7 min (major), 49.7 min

[1] a) Lindhorst, T.; Kieburg, C. *Synthesis* **1995**, 10, 1228; b) Selkti, M.; Kassab, R.; Lopez, H. P.; Villain, F.; de Rango, C. *J. Carbohydr. Chem.* **1999**, 18, 1019; c) Pearson, M. S. M.; Robin, A.; Bourgougnon, N.; Meslin, J. C.; Deniaud, D. *J. Org. Chem.* **2003**, 68, 8583.

[2] Vakylya, B.; Varga, S.; Csapmpai, A.; Soós, T. *Org. Lett.* **2005**, 7, 1967.

[3] Dixon, D. J.; Richardson, R. D.; *Synlett.* **2006**, 1, 81.

[4] Huang, H.; Jacobsen, E. N. *J. Am. Chem. Soc.* **2006**, 128, 7170.

[5] Corey, E. J.; Zhang, F.-Y. *Org. Lett.* **2000**, 2, 4257.

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.501	VV	0.3519	1921.47742	82.91228	49.6538
2	23.243	VV	0.4993	1948.26880	59.36709	50.3462

Totals : 3869.74622 142.27937

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.422	VV	0.3702	1.30465e4	538.06995	98.4865
2	23.197	VV	0.4908	200.49246	6.15389	1.5135

Totals : 1.32470e4 544.22384

Sample Info : 254nm,i-PrOH:hexane=20:80,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.342	VV	0.2194	6549.32129	449.72693	50.2293
2	12.261	VV	0.2797	6489.52197	351.12842	49.7707

Totals : 1.30388e4 800.85535

Sample Info : 254nm,i-PrOH:hexane=20:80,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.315	VV	0.2151	1.03338e4	728.22595	98.2986
2	12.249	VV	0.2893	178.86186	9.14608	1.7014

Totals : 1.05126e4 737.37204

Sample Info : 254nm,i-PrOH:hexane=20:80,AD-H,1.0ml/min

Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.932	VV	0.2880	9430.15820	497.83713	50.9575
2	17.541	VV	0.3888	9075.78125	358.13947	49.0425

Totals : 1.85059e4 855.97659

Sample Info : 254nm,i-PrOH:hexane=20:80,AD-H,1.0ml/min

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.894	VB	0.3181	1.41761e4	682.38989	97.7750
2	18.994	BB	0.4229	322.58795	11.62095	2.2250

Totals : 1.44987e4 694.01084

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	17.714	VV	0.3804	2669.28247	106.28538	48.6187
2	25.923	VV	0.5752	2820.95020	74.59035	51.3813

Totals : 5490.23267 180.87573

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	17.591	VB	0.4048	1.56391e4	591.00964	97.3943
2	25.944	VB	0.5563	418.40488	11.40176	2.6057

Totals : 1.60575e4 602.41140

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	12.007	VV	0.2784	1.21955e4	663.94922	49.4205
2	14.467	VV	0.3376	1.24815e4	562.28192	50.5795

Totals : 2.46770e4 1226.23114

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	12.047	VV	0.2770	9704.26758	531.74530	98.6708
2	14.549	VB	0.3383	130.72420	5.74436	1.3292

Totals : 9834.99178 537.48966

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.208	VV	0.2621	1.47276e4	855.03442	46.6272
2	13.453	VV	0.3200	1.68582e4	804.95410	53.3728
Totals :				3.15858e4	1659.98853	

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.162	VV	0.2731	2.78421e4	1553.76257	98.7639
2	13.438	VB	0.3144	348.47189	16.43091	1.2361
Totals :				2.81906e4	1570.19348	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.100	VB	0.5235	1321.26965	38.42133	48.8879
2	28.620	VV	0.6550	1381.38171	32.00338	51.1121
Totals :				2702.65137	70.42471	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.384	VV	0.5257	6109.02197	176.66077	97.0026
2	29.003	VB	0.6517	188.77295	4.45411	2.9974
Totals :				6297.79492	181.11488	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.160	BV	0.2562	2676.48145	157.74576	48.4290
2	14.665	VB	0.2979	2850.12866	145.84634	51.5710
Totals :				5526.61011	303.59210	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.095	VV	0.2779	1.94220e4	1059.62793	98.7292
2	14.699	VV	0.3026	249.99739	12.37747	1.2708
Totals :				1.96720e4	1072.00540	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.503	VV	0.1773	840.97498	68.93568	52.4569
2	8.177	VV	0.1781	762.19916	64.76085	47.5431
Totals :				1603.17413	133.69653	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.770	VV	0.1513	7831.65088	785.56830	96.7334
2	7.339	VV	0.1705	264.46710	22.75403	3.2666
Totals :				8096.11798	808.32233	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	19.073	VV	0.4207	1.32086e4	474.70197	49.5383
2	31.386	VV	0.6912	1.34548e4	297.24155	50.4617
Totals :				2.66633e4	771.94351	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	18.530	VB	0.4210	5.23673e4	1880.57556	98.0741
2	30.782	BB	0.6586	1028.35498	23.93153	1.9259
Totals :				5.33956e4	1904.50709	

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.649	VV	0.5881	4979.80371	127.91352	50.2772
2	39.631	VB	0.8973	4924.89258	84.26532	49.7228
Totals :				9904.69629	212.17884	

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	24.366	VB	0.5997	2.75504e4	690.16364	98.5012
2	39.746	VB	0.8811	419.20840	7.28353	1.4988
Totals :				2.79696e4	697.44717	

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.281	VV	0.2757	9515.34570	524.64630	20.6983
2	16.630	VV	0.3817	1.05465e4	418.07480	22.9413
3	18.905	VV	0.4388	1.27932e4	439.25192	27.8285
4	24.795	VV	0.5749	1.31166e4	347.05167	28.5319

Totals : 4.59716e4 1729.02469

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

Area Percent Report

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.282	VV	0.2782	1.21790e4	672.72821	56.5573
2	16.680	VB	0.3630	255.66861	10.70726	1.1873
3	18.936	BV	0.4385	8886.56152	310.73959	41.2678
4	24.888	VB	0.6162	212.66685	5.08309	0.9876

Totals : 2.15339e4 999.25815

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.530	VV	0.3782	1.01324e4	406.44333	49.4292
2	21.757	VB	0.4972	1.03664e4	317.63647	50.5708
Totals :				2.04988e4	724.07980	

Sample Info : 254nm,i-PrOH:hexane=15:85,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	16.762	VV	0.4140	7.57673e4	2806.64844	98.0076
2	22.238	VB	0.5074	1540.26062	45.97182	1.9924
Totals :				7.73076e4	2852.62025	

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	25.530	BV	0.5909	681.80896	17.40832	50.5377
2	49.037	BB	1.1100	667.29980	9.30964	49.4623

Totals : 1349.10876 26.71795

Sample Info : 254nm,i-PrOH:hexane=10:90,AD-H,1.0ml/min

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

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	25.743	VB	0.6117	1.28484e4	321.60965	97.3742
2	49.772	BB	1.1182	346.47461	4.62702	2.6258

Totals : 1.31949e4 326.23667