

Supplementary Material

for

**The Organocatalytic Synthesis of Enantioenriched 1, 2,
4-Triazolines Containing Chiral Quaternary Carbon Centers**

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I . General experimental information

Unless otherwise noted, commercial reagents were used as received and all reactions were carried out directly in air atmosphere. All reactions were monitored by TLC with silica gel coated plates. ^1H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 600 spectrometer. HRMS (Bio TOF Q) spectra were recorded on P-SIMS-Gly of Bruker Daltonics Inc. Chemical shifts are reported in ppm from tetramethylsilane (TMS) with the solvent resonance as the internal standard. Proton signal multiplicities are given as s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), br (broad) or a combination of them. *J*-values are in Hz. Enantiomer ratios were determined by HPLC with chiral columns (Chiralpak IA 、IC and AD-H was purchased from Daicel Industries). Optical rotations were measured on a Perkin Elmer 341 Polarimeter at $\lambda = 589$ nm. The substituted α -isothiocyanatos ^[1] and catalysts ^[2] were prepared according to the literatures.

References:

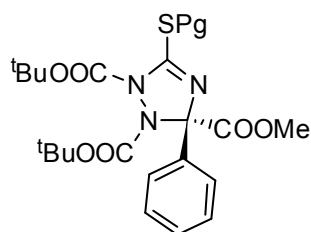
1. (a) K. Tani , K. Suwa , E. Tanigawa , T. Lse , T. Yamagate , Y. Tatsuno , S. Osaka , *Jouranal of Organmetallic Chemistry* 1989, 370, 203. (b) Claudia Bello , Michele Cea , Giovanna Dal Bello , Anna Garuti , Ilaria Rocco , Gabriella Cirmena , Eva Moran , Aimable Nahimana , Michel A. Duchosal , Floriana Fruscione , Paolo Pronzato , Francesco Grossi , Franco Patrone , Alberto Ballestrero , Marc Dupuis , Bernard Sordat , Alessio Nencioni , Pierre Vogel , *Bioorganic & Medicinal Chemistry* 2010 ,18 , 3320–3334.
2. (a) Mukkanti Amere , Marie-Claire Lasne and Jacques Rouden , *Org. Lett.* 2007, 9 , 2621-2614. (b) Stefan France , Harald Wack , Andrew E. Taggi , Ahmed M. Hafez , Ty R. Wagerle, Meha H. Shah , Crystal L. Dusich and Thomas Lectka, *J. Am. Chem. Soc.* 2004 , 126, 4245-4255.

II. The general procedure for the Asymmetric reaction.

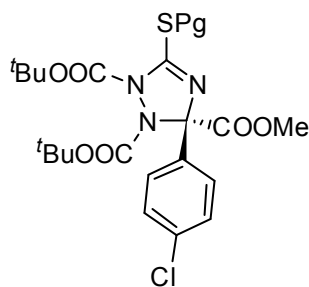


General procedure:

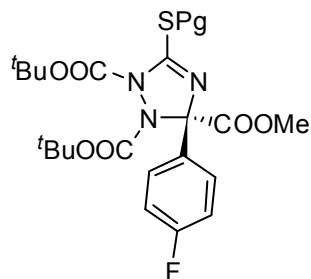
A reaction tube was charged with **1** (0.1 mmol), **2** (0.16 mmol), catalyst **QD-3f** (4.3 mg, 0.01 mmol), Na₂SO₄ (20 mg, 0.06 mmol) and xylene (1 mL). The solution was stirred at -20°C until the reaction completed (monitored by TLC). One drop of CH₃COOH was added to the mixture. The solvent was concentrated and the residue was subjected to silica gel column chromatography to afford the desired products **4** with using ethyl acetate/petroleum as eluents.



4a white solid (41 mg, 77%) ; the enantiomeric excess was determined to be 89% by HPLC analysis on Daicel Chirapak AD-H column (hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 4.74 min, t_R (major) 6.07 min; $[\alpha]_D^{20} = +124$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.59 (s, 2H), 7.35 (s, 3H), 7.22 (s, 1H), 3.75 (s, 3H), 1.52-1.47 (m, 36H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 168.10, 155.71, 154.39, 153.89, 137.87, 128.57, 127.88, 127.27, 95.25, 86.34, 83.95, 83.76, 81.59, 53.10, 28.19, 28.16, 27.94, 27.76; HRMS (ESI): calcd. for C₃₀H₄₆N₅O₁₀S (M⁺+H): 668.2960, found: 668.2956.

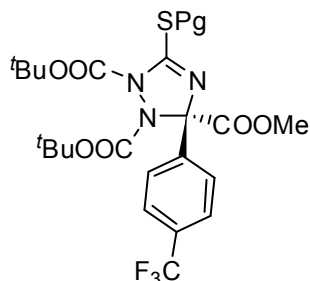


4b white solid (42 mg, 75 %); the enantiomeric excess was determined to be 87% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 95/5, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 6.85 min, t_R (major) 10.11 min; $[\alpha]_D^{20} = +105$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.52 (s, 2H), 7.33 (s, 2H), 7.17 (s, 1H), 3.75 (s, 3H), 1.53-1.48 (m, 36H); ¹³C NMR (151 MHz, CDCl₃) δ(ppm) 167.78, 159.12, 154.28, 153.79, 136.54, 134.59, 128.73, 128.05, 94.73, 86.61, 84.01, 81.65, 53.19, 28.15, 27.92, 27.90, 27.73; HRMS (ESI): calcd. for C₃₀H₄₅ClN₅O₁₀S (M⁺+H): 702.2570, found: 702.2548.



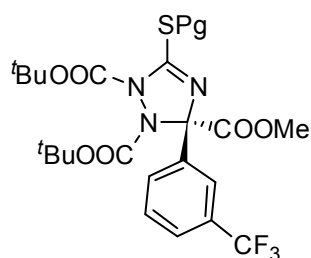
4c white solid (37 mg, 68 %) ; the enantiomeric excess was determined to be 88% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 4.72 min, t_R (major) 6.18 min; $[\alpha]_D^{20} = +124$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.57 (s, 2H), 7.17 (s, 1H), 7.04 (s, 2H), 3.75 (s, 3H), 1.58-1.39 (m, 36H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 167.95, 163.69-162.05 (d, ¹J_{F-C} = 247.6 Hz, 1C), 159.02, 154.34, 153.85, 133.81,

129.23, 129.17, 114.86-114.71 (d, $^2J_{F-C} = 22.65$ Hz, 1C), 94.73, 86.59, 84.03, 83.96, 81.67, 53.18, 28.17, 27.94, 27.93, 27.74; HRMS (ESI): calcd. for $C_{30}H_{45}FN_5O_{10}S$ ($M^+ + H$): 686.2866, found: 686.2850.



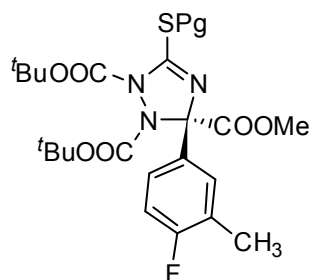
4d white solid (38 mg, 65 %) ; the enantiomeric excess was determined to be 82% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 4.19 min, t_R (major) 5.16 min; $[\alpha]_D^{20} = +109$ (c = 0.1, CH_3CH_2OH); 1H NMR (600 MHz, $CDCl_3$)

δ (ppm) 7.67 (d, $J = 58.7$ Hz, 4H), 7.17 (s, 1H), 3.76 (s, 3H), 1.64-1.37 (m, 36H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 167.63, 159.46, 154.27, 153.82, 141.83, 131.07-130.43 (q, $^2J_{F-C} = 31.7$ Hz, 1C), 127.79, 126.74-121.33 (q, $^1J_{F-C} = 271.8$ Hz, 1C), 124.89, 94.54, 86.80, 84.23, 84.11, 81.76, 53.29, 28.16, 27.93, 27.91, 27.73; HRMS (ESI): calcd. for $C_{31}H_{45}F_3N_5O_{10}S$ ($M^+ + H$): 736.2834, found: 736.2825.



4e white solid (41 mg, 70%) ; the enantiomeric excess was determined to be 71% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 95/5, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 6.04 min, t_R (major) 8.65 min; $[\alpha]_D^{20} = +81$ (c = 0.1, CH_3CH_2OH); 1H NMR (600 MHz, $CDCl_3$) δ (ppm)

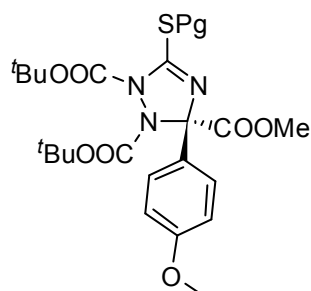
7.85-7.80 (m, 2H), 7.61 (d, $J = 7.2$ Hz, 1H), 7.50 (s, 1H), 7.15 (s, 1H), 3.77 (s, 3H), 1.56-1.48 (m, 36H); ^{13}C NMR (151 MHz, $CDCl_3$) δ (ppm) 167.60, 154.24, 153.81, 153.59, 138.96, 130.92, 130.68-130.04 (q, $^2J_{F-C} = 31.7$ Hz, 1C), 128.43, 126.78-121.37 (q, $^1J_{F-C} = 274.8$, 1C), 125.45, 125.43, 124.18, 94.73, 86.68, 84.22, 84.08, 81.66, 53.29, 28.13, 27.90, 27.84, 27.79, 27.75, 27.69; HRMS (ESI): calcd. for $C_{31}H_{45}F_3N_5O_{10}S$ ($M^+ + H$): 736.2834, found: 736.2828.



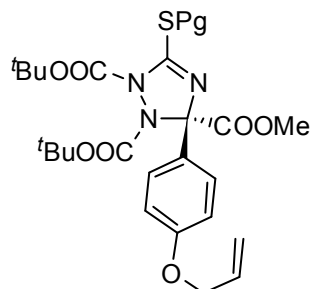
4g white solid (38 mg, 70 %) ; the enantiomeric excess was determined to be 85% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 3.62 min, t_R (major) 4.08 min; $[\alpha]_D^{20} = +110$ (c = 0.1, CH_3CH_2OH); 1H NMR (600 MHz, $CDCl_3$) δ

(ppm) 7.39 (s, 2H), 7.20 (s, 1H), 7.00-6.97 (m, 1H), 3.75 (s, 3H), 2.26 (s, 3H), 1.58-1.37 (m, 36H);

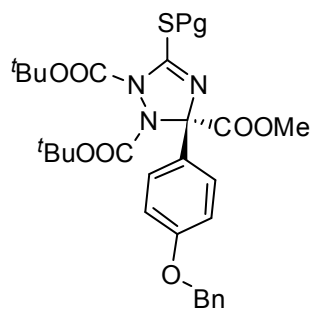
^{13}C NMR (151 MHz, CDCl_3) δ (ppm) 167.67, 162.23-160.60 (d, $J_{\text{F-C}} = 246.1$ Hz, 1C), 154.40, 153.87, 133.40, 130.57, 126.32, 126.28, 124.33-124.21 (d, $^2J_{\text{F-C}} = 18.1$ Hz, 1C), 114.51-114.35 (d, $^2J_{\text{F-C}} = 22.6$ Hz, 1C), 94.46, 86.51, 83.98, 83.89, 81.61, 53.14, 28.18, 28.01, 27.95, 27.94, 27.75, 14.63; HRMS (ESI): calcd. for $\text{C}_{31}\text{H}_{47}\text{FN}_5\text{O}_{10}\text{S}$ ($\text{M}^+ + \text{H}$): 700.3022, found: 700.2993.



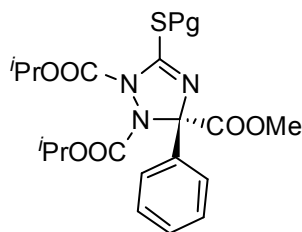
4h white solid (30 mg, 54%) ; the enantiomeric excess was determined to be 85% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 70/30, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 4.01 min, t_R (major) 4.58 min; $[\alpha]_{\text{D}}^{20} = +79$ (c = 0.1, $\text{CH}_3\text{CH}_2\text{OH}$); ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.50 (s, 2H), 7.20 (s, 1H), 6.89-6.87 (m, 2H), 3.81 (s, 3H), 3.74 (s, 3H), 1.55-1.43 (m, 36H); ^{13}C NMR (151 MHz, CDCl_3) δ (ppm) 172.48, 168.22, 159.80, 159.68, 154.40, 153.86, 130.06, 129.11, 128.74, 128.53, 114.35, 113.31, 94.97, 86.33, 83.92, 83.66, 81.53, 55.24, 53.06, 28.17, 27.94, 27.93, 27.75; HRMS (ESI): calcd. for $\text{C}_{31}\text{H}_{48}\text{N}_5\text{O}_{11}\text{S}$ ($\text{M}^+ + \text{H}$): 698.3066, found: 698.3055.



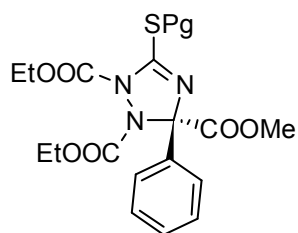
4i white solid (30 mg, 53%) ; the enantiomeric excess was determined to be 84% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 7.39 min, t_R (major) 11.37 min; $[\alpha]_{\text{D}}^{20} = +92$ (c = 0.1, $\text{CH}_3\text{CH}_2\text{OH}$); ^1H NMR (600 MHz, CDCl_3) δ (ppm) 7.49 (s, 2H), 7.22 (s, 1H), 6.90 (d, $J = 7.5$ Hz, 2H), 6.05 (ddd, $J = 16.0, 10.3, 5.1$ Hz, 1H), 5.35 (dd, $J = 77.6, 13.9$ Hz, 2H), 4.54 (t, $J = 8.3$ Hz, 2H), 3.74 (s, 3H), 1.57-1.42 (m, 36H); ^{13}C NMR (151 MHz, CDCl_3) δ (ppm) 171.02, 168.26, 158.85, 155.69, 154.40, 153.88, 133.24, 130.20, 128.53, 117.61, 114.10, 94.96, 86.34, 83.92, 83.66, 81.54, 68.84, 53.06, 28.17, 28.15, 27.94, 27.93, 27.75; HRMS (ESI): calcd. for $\text{C}_{33}\text{H}_{50}\text{N}_5\text{O}_{11}\text{S}$ ($\text{M}^+ + \text{H}$): 724.3222, found: 724.3206.



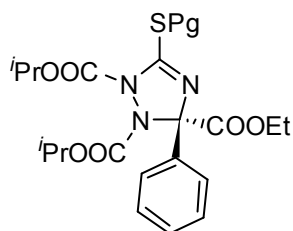
4j white solid (35 mg, 56%) ; the enantiomeric excess was determined to be 84% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 90/10, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 10.22 min, t_R (major) 16.30 min; $[\alpha]_D^{20} = +126$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.50 (s, 2H), 7.44-7.31 (m, 6H), 6.96 (d, J =8.7 Hz, 2H), 5.06 (d, J =11.5 Hz, 2H), 3.74 (s, 3H), 1.63-1.42 (m, 36H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 171.05, 168.28, 159.07, 154.41, 153.90, 136.96, 130.34, 128.58, 127.96, 127.47, 114.22, 94.92, 86.37, 83.95, 83.69, 81.57, 70.07, 53.09, 28.19, 27.95, 27.76; HRMS (ESI): calcd. for C₃₇H₅₂N₅O₁₁S (M⁺+H): 774.3379, found: 774.3362.



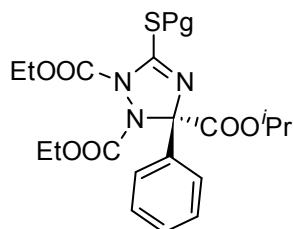
4l white solid (35 mg, 72%) ; the enantiomeric excess was determined to be 38% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 95/5, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 12.18 min, t_R (major) 15.79 min; $[\alpha]_D^{20} = +65$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.62-7.56 (m, 2H), 7.36 (s, 4H), 5.05-4.92 (m, 4H), 3.76 (s, 3H), 1.37-1.25 (m, 24H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 167.69, 158.73, 155.44, 154.82, 154.59, 149.79, 137.49, 130.19, 128.76, 127.96, 127.14, 126.67, 95.77, 73.84, 73.14, 72.07, 70.17, 53.31, 21.94, 21.89, 21.73, 21.69, 21.67, 21.64, 21.61, 21.59; HRMS (ESI): calcd. for C₂₆H₃₈N₅O₁₀S (M⁺+H): 612.2334, found: 612.2337.



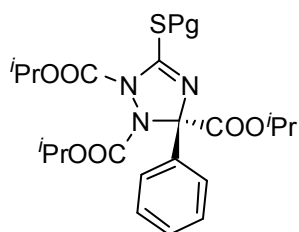
4m white solid (33 mg, 76%) ; the enantiomeric excess was determined to be 14% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 7.33 min, t_R (major) 8.34 min; $[\alpha]_D^{20} = +58$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.56 (s, 2H), 7.37 (s, 4H), 4.34-4.23 (m, 8H), 3.76 (s, 3H), 1.35-1.28 (m, 12H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 167.69, 158.55, 155.88, 155.37, 155.23, 154.92, 137.39, 128.88, 127.88, 128.04, 127.97, 127.40, 127.09, 95.75, 64.85, 64.77, 64.57, 63.65, 53.49, 14.39, 14.21, 14.17, 14.08; HRMS (ESI): calcd. for C₂₂H₃₀N₅O₁₀S (M⁺+H): 556.1708, found: 556.1705.



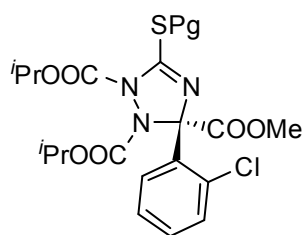
4n white solid (24 mg, 49%) ; the enantiomeric excess was determined to be 40% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 4.30 min, t_R (major) 4.80 min; $[\alpha]_D^{20}=+56$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.62-7.50 (m, 2H), 7.35-7.22 (m, 4H), 5.06-4.92 (m, 4H), 4.31-4.08 (m, 2H), 1.41-1.20 (m, 27H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.98, 158.53, 155.47, 154.77, 154.50, 149.83, 137.48, 128.68, 127.87, 127.18, 95.73, 73.81, 73.12, 71.99, 70.11, 62.68, 21.91, 21.68, 21.60, 13.87; HRMS (ESI): calcd. for C₂₇H₄₀N₅O₁₀S (M⁺+H): 626.2490, found: 626.2480.



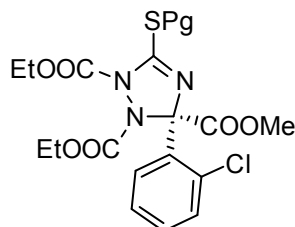
4o white solid (40 mg, 78%) ; the enantiomeric excess was determined to be 62% by HPLC analysis on Daicel Chirapak IC column (hexane/isopropanol = 70/30, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 22.71 min, t_R (major) 26.04 min; $[\alpha]_D^{20}=+61$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.63-7.56 (m, 2H), 7.44-7.29 (m, 4H), 5.03 (s, 1H), 4.36-4.22 (m, 8H), 1.35-1.20 (m, 18H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.27, 158.29, 155.94, 155.26, 154.84, 150.47, 137.45, 128.80, 127.97, 127.24, 96.16, 71.08, 64.86, 64.82, 63.48, 62.34, 21.52, 21.43, 14.40, 14.28, 14.19, 14.13; HRMS (ESI): calcd. for C₂₄H₃₄N₅O₁₀S (M⁺+H): 584.2021, found: 584.2013.



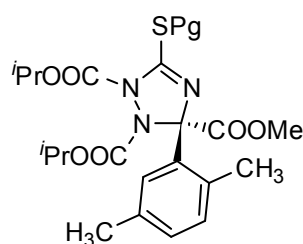
4p white solid (28 mg, 55%) ; the enantiomeric excess was determined to be 76% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 95/5, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 8.40 min, t_R (major) 11.02 min; $[\alpha]_D^{20}=+90$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.62-7.56 (m, 2H), 7.34 (s, 4H), 5.04-4.89 (m, 5H), 1.37-1.20 (m, 30H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.33, 158.37, 155.49, 154.70, 154.47, 149.95, 137.55, 128.62, 127.82, 127.24, 96.59, 73.79, 73.12, 71.87, 70.88, 70.08, 21.95, 21.92, 21.68, 21.60, 21.53, 21.37, 21.29, 21.26; HRMS (ESI): calcd. for C₂₈H₄₂N₅O₁₀S (M⁺+H): 640.2647, found: 640.2636.



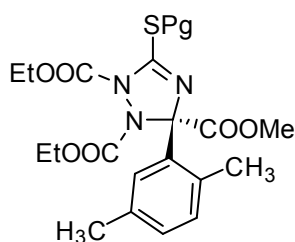
4q white solid (29 mg, 58%) ; the enantiomeric excess was determined to be 95% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 4.72 min, t_R (major) 5.36 min; $[\alpha]_D^{20} = +85$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.56 (s, 2H), 7.36 (s, 3H), 5.05-4.91 (m, 4H), 3.75 (s, 3H), 1.36-1.24 (m, 24H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 165.99, 158.94, 155.41, 154.86, 154.77, 154.46, 131.28, 130.25, 128.81, 128.00, 127.18, 126.72, 95.86, 73.88, 73.18, 73.07, 72.40, 72.12, 53.34, 21.98, 21.84, 21.76, 21.73, 21.70, 21.65, 21.60; HRMS (ESI): calcd. for C₂₆H₃₇ClN₅O₁₀S (M⁺+H): 646.1944, found: 646.1929.



4r white solid (41 mg, 87%) ; the enantiomeric excess was determined to be 83% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 95/5, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 6.36 min, t_R (major) 8.37 min; $[\alpha]_D^{20} = 56$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.68 (s, 2H), 7.43 (s, 1H), 7.31 (s, 2H), 4.36-4.21 (m, 8H), 3.78 (s, 3H), 1.38-1.25 (m, 12H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.10, 155.80, 155.00, 154.92, 149.96, 134.39, 133.43, 131.32, 130.31, 127.96, 126.77, 96.10, 64.92, 64.76, 63.92, 62.23, 53.49, 14.40, 14.15, 14.11; HRMS (ESI): calcd. for C₂₂H₂₉ClN₅O₁₀S (M⁺+H): 590.1318, found: 590.1326.



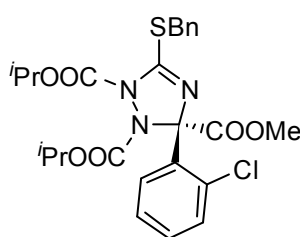
4s white solid (20 mg, 41%) ; the enantiomeric excess was determined to be 77% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 4.05 min, t_R (major) 4.72 min; $[\alpha]_D^{20} = +89$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.40 (s, 1H), 7.06 (s, 3H), 5.07-5.02 (m, 4H), 3.77 (s, 3H), 2.31 (s, 6H), 1.38-1.27 (m, 24H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 167.31, 157.73, 155.36, 154.66, 154.59, 150.01, 134.96, 134.55, 133.97, 131.88, 129.52, 126.95, 96.85, 73.55, 73.03, 72.08, 70.13, 53.20, 21.94, 21.85, 21.76, 21.70, 21.62, 21.10, 20.81; HRMS (ESI): calcd. for C₂₈H₄₂N₅O₁₀S (M⁺+H): 640.2647, found: 640.2637.



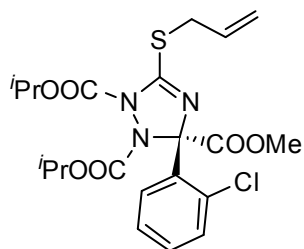
4t white solid (20 mg, 44%) ; the enantiomeric excess was determined to be 72% by HPLC analysis on Daicel Chirapak IA column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 5.31 min, t_R (major) 6.70 min; $[\alpha]_D^{20} = +71$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.40 (s, 2H), 7.08 (s, 2H), 4.39-4.18 (m, 8H), 3.78 (s, 3H), 2.32 (s, 6H), 1.38-1.26 (m, 12H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 167.36, 155.87, 155.18, 154.94, 150.35, 135.12, 134.36, 133.95, 131.94, 129.67, 126.91, 97.17, 64.77, 64.73, 64.08, 63.79, 53.43, 21.15, 20.76, 14.43, 14.35, 14.21, 14.15; HRMS (ESI): calcd. for C₂₄H₃₄N₅O₁₀S (M⁺+H): 584.2021, found: 584.2000.

III. General Procedure for the synthesis of **5a**, **5b** and **5c**.

The synthesis of 5a and 5b: To mixture of **4q** (0.1 mmol) and anhydrous K₂CO₃ (0.3 mmol) in 2.0 mL acetone was added BnBr or 3-bromopropene (0.2 mmol) at room temperature. Then the reaction was stirred at room temperature for 8 hours and then concentrated in vacuo. The residue mixture was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 8:1) to give compound **5a** or **5b**.

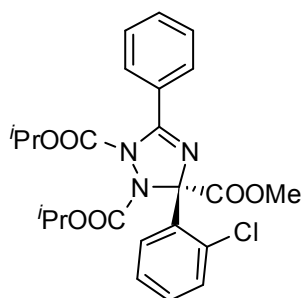


5a white solid (34 mg, 92%) ; the enantiomeric excess was determined to be 95% by HPLC analysis on Daicel Chirapak IC column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30 °C), UV 254 nm, t_R (minor) 8.16 min, t_R (major) 10.58 min; $[\alpha]_D^{20} = +112$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.73 (s, 1H), 7.44 (d, J = 7.6 Hz, 1H), 7.39 (d, J = 7.0 Hz, 2H), 7.32-7.23 (m, 5H), 5.06 – 4.98 (m, 2H), 4.38 (d, J = 13.2 Hz, 1H), 4.24 (d, J = 13.2 Hz, 1H), 3.76 (s, 3H), 1.34-1.26 (m, 12H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.73, 158.28, 154.32, 150.40, 136.20, 135.05, 133.92, 131.23, 129.94, 129.39, 128.45, 128.21, 127.51, 126.49, 95.03, 72.72, 71.84, 53.19, 37.11, 21.80, 21.72, 21.63; HRMS (ESI): calcd. for C₂₅H₂₉ClN₃O₆S (M⁺+H): 534.1460, found: 534.1455.



5b: 4t white solid (34 mg, 101%); the enantiomeric excess was determined to be 95% by HPLC analysis on Daicel Chirapak IC column (hexane/isopropanol = 70/30, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 7.39 min, t_R (major) 9.15min; $[\alpha]_D^{20} = +45$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.73 (d, J =7.2 Hz, 1H), 7.42 (d, J =7.6 Hz, 1H), 7.30-7.24 (m, 2H), 5.95 (ddt, J =17.1, 10.0, 7.1 Hz, 1H), 5.30 (dd, J =17.0, 0.9 Hz, 1H), 5.13 (d, J =10.0 Hz, 1H), 5.08-4.99 (m, 2H), 3.78 (s, 3H), 3.72 (ddd, J =20.2, 13.4, 6.7 Hz, 2H), 1.36-1.26 (m, 12H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.87, 158.08, 154.38, 150.48, 135.10, 133.91, 132.16, 131.20, 129.93, 129.39, 128.14, 126.48, 119.04, 95.08, 72.70, 71.84, 53.23, 35.61, 21.83, 21.76, 21.66; HRMS (ESI): calcd. for C₂₁H₂₇ClN₃O₆S (M⁺+H): 484.1304, found: 484.1303.

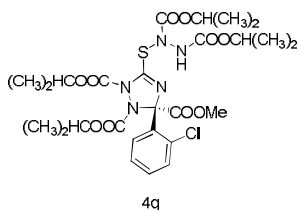
The synthesis of 5c: To a flame dried test tube were added **4q** (0.20 mmol), phenylboronic acid (0.40 mmol), Cu(thiophene-2-carboxylate) (0.60 mmol), and Pd(PPh₃)₄ (0.02 mmol). The test tube was flashed with argon. Then, THF (2.0 mL) was added via syringe, and the resulting mixture was stirred at 50°C for 3h. After it was cooled to ambient temperature, THF was removed by evaporation. Sat. aq. NaHCO₃ was added, and the mixture was extracted with CH₂Cl₂ three times. Combined organic layers were dried over Na₂SO₄ and filtered. After evaporation of solvent, the crude residue was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 8:1) to give compound **5c**.



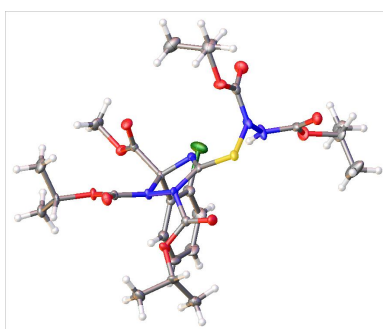
5c white solid (70 mg, >99%); the enantiomeric excess was determined to be 94% by HPLC analysis on Daicel Chirapak IC column (hexane/isopropanol = 80/20, flow rate 1.0 mL/min, T = 30°C), UV 254 nm, t_R (minor) 8.76 min, t_R (major) 9.57 min; $[\alpha]_D^{20} = -43$ (c = 0.1, CH₃CH₂OH); ¹H NMR (600 MHz, CDCl₃) δ (ppm) 7.84 (d, J = 7.3 Hz, 2H), 7.80 (dd, J = 7.7, 1.2 Hz, 1H), 7.51-7.46 (m, 2H), 7.40 (t, J = 7.7 Hz, 2H), 7.34-7.30 (m, 1H), 7.28-7.25 (m, 1H), 5.07-4.99 (m, 1H), 4.92-4.86 (m, 1H), 3.80 (s, 3H), 1.33-1.29 (m, 6H), 1.19 (d, J = 6.2 Hz, 3H), 1.12 (d, J = 6.2 Hz, 3H); ¹³C NMR (151 MHz, CDCl₃) δ (ppm) 166.67, 158.91, 154.30, 151.73, 134.59, 134.08, 131.88, 131.26, 130.29, 130.11, 128.58, 128.54, 128.42, 127.78, 126.51, 94.46, 72.28, 71.58,

53.38, 21.98, 21.95, 21.75, 21.59, 21.49; HRMS (ESI): calcd. for $C_{24}H_{27}ClN_3O_6$ ($M^+ + H$): 488.1583, found: 488.1581.

IV. Determination of the absolute configuration of compounds 4q.



(S)-1,2-diisopropyl 3-methyl 5-(1,2-bis(isopropoxycarbonyl)hydrazinylthio)-3-(2-chlorophenyl)-1H-1,2,4-triazole-1,2,3(3H)-tricarboxylate

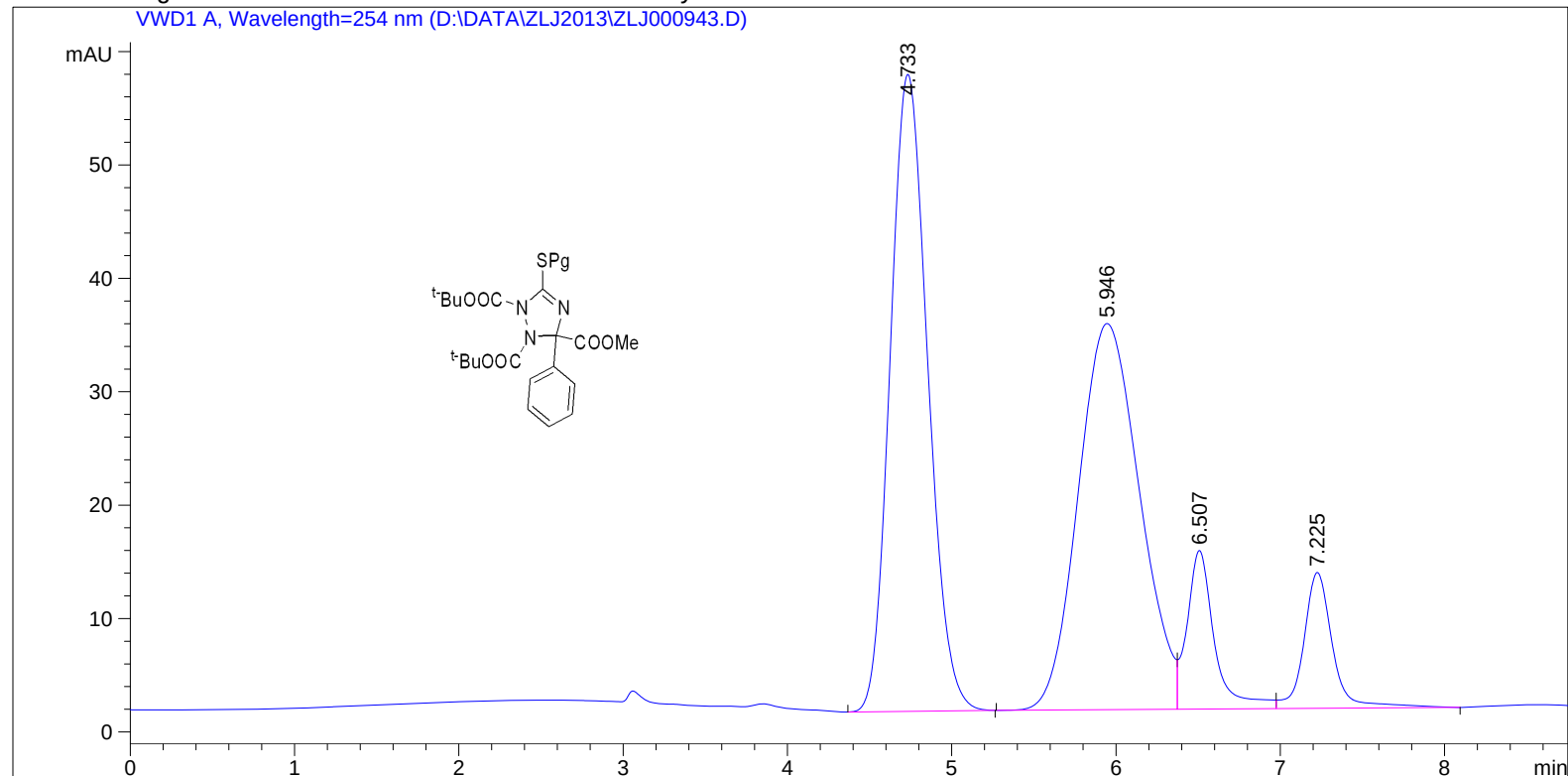


Chemical Formula	$C_{26}H_{36}ClN_5O_{10}S$
Formula weight	645.12
Temperature	293(2) K
Wavelength	1.54184 Å
Crystal system, space group	monoclinic, P 21
a, Å	10.3480 (5)
b, Å	15.6841 (6)
c, Å	10.7299 (5)
α , °	90.00
β , °	109.506 (5)
γ , °	90.00
Volume/Å ³	1641.50 (14)
Flack parameter	-0.001(8)
Z, Calculated density	2, 1.305 g/cm ³

V. The spectrums of 1H NMR, ^{13}C NMR and HPLC.

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Area Percent Report
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Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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1	4.733	BB	0.2478	891.52612	56.17606	42.8229
2	5.946	BV	0.4044	882.82587	34.05005	42.4050
3	6.507	VV	0.1705	162.47496	13.98409	7.8042
4	7.225	VB	0.1820	145.06435	11.98144	6.9679

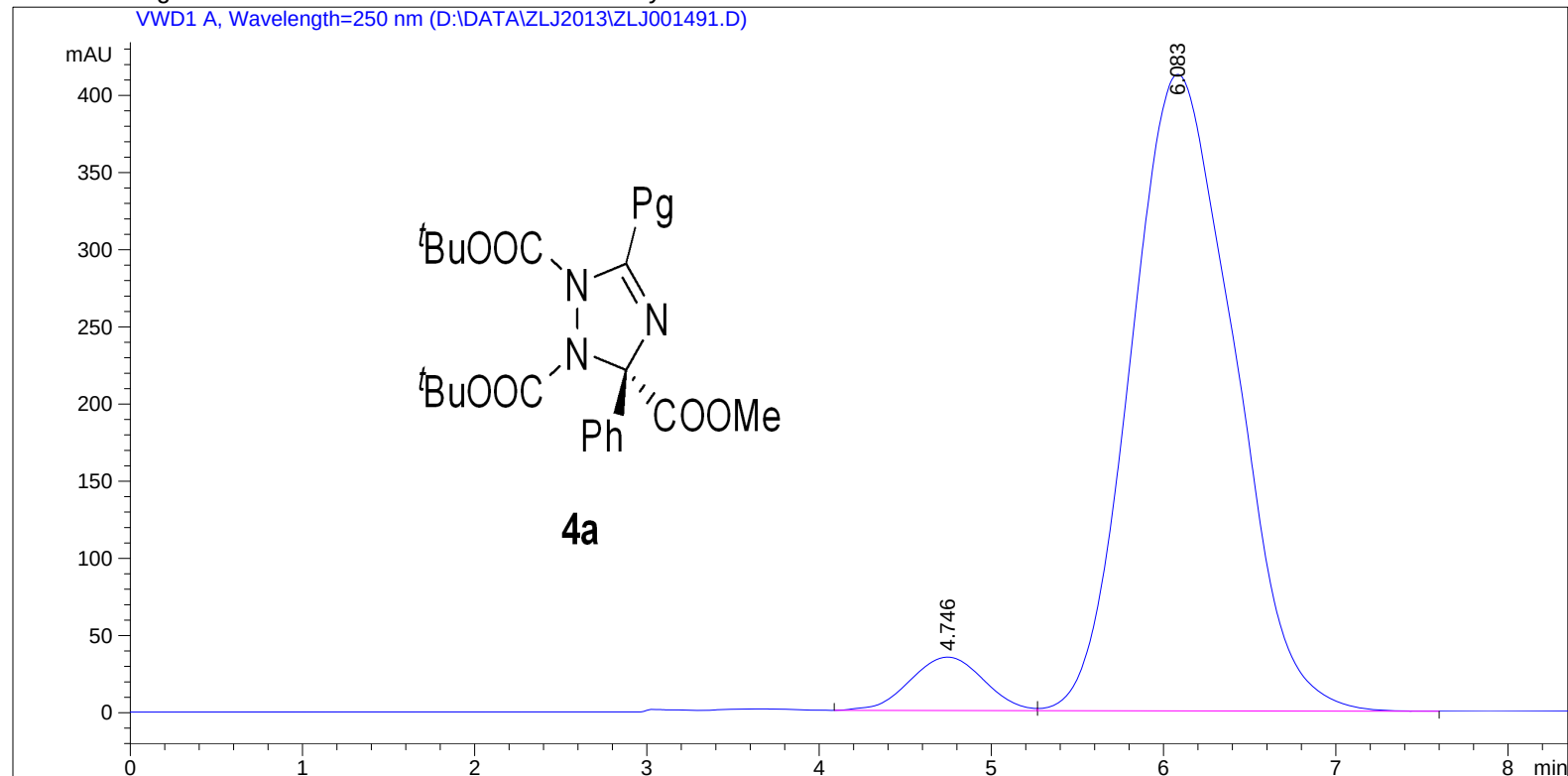
Totals : 2081.89130 116.19164

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S11

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Area Percent Report
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Use Multiplier & Dilution Factor with ISTDs

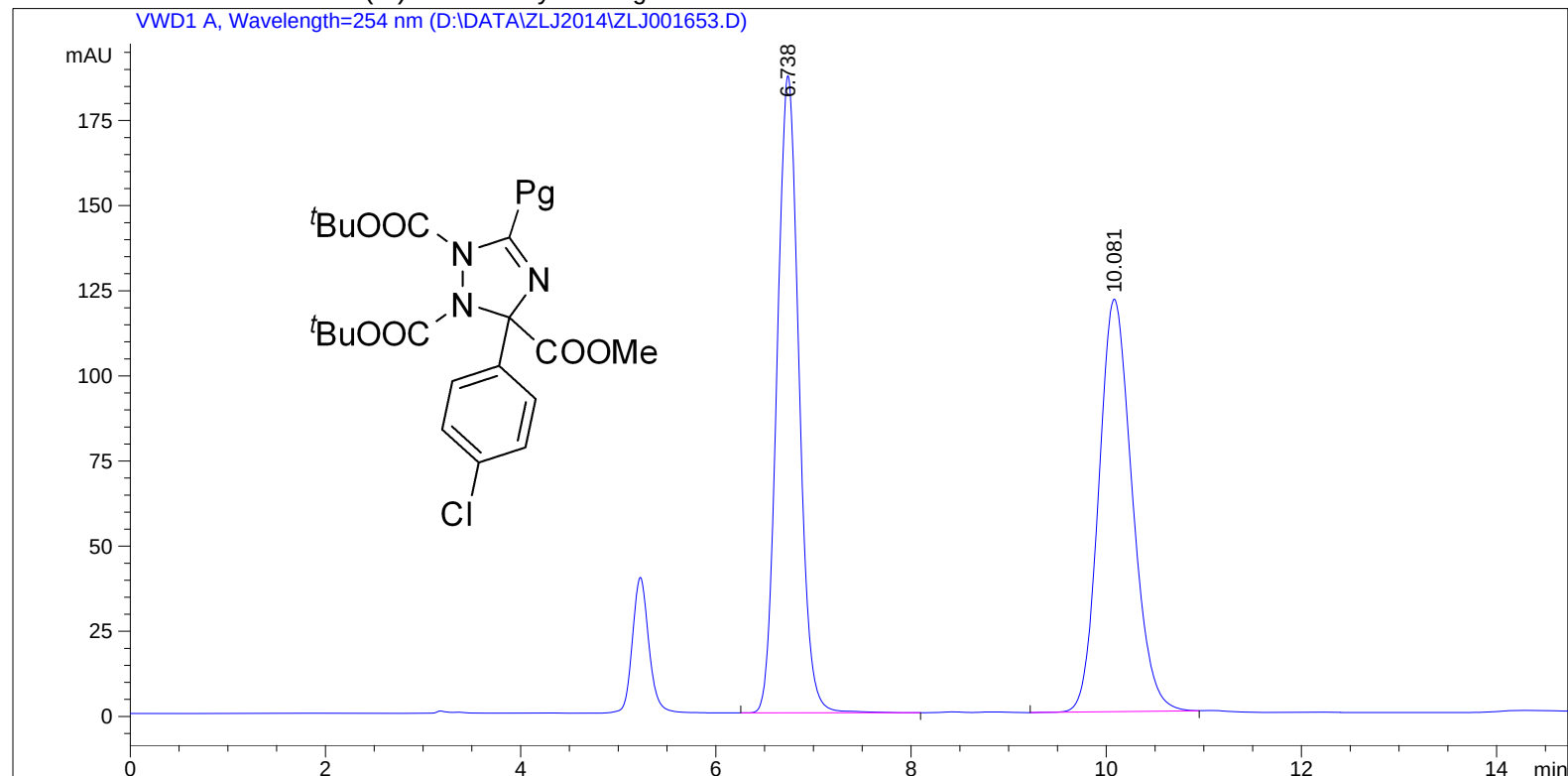
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.746	BV	0.4738	1024.43799	34.57751	5.6553
2	6.083	VB	0.6130	1.70903e4	412.43887	94.3447

Totals : 1.81148e4 447.01638

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*** End of Report ***

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Injection Date : 4/18/2014 10:02:25 AM
Inj Volume : No inj
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Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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: VWD1 A, Wavelength=254 nm

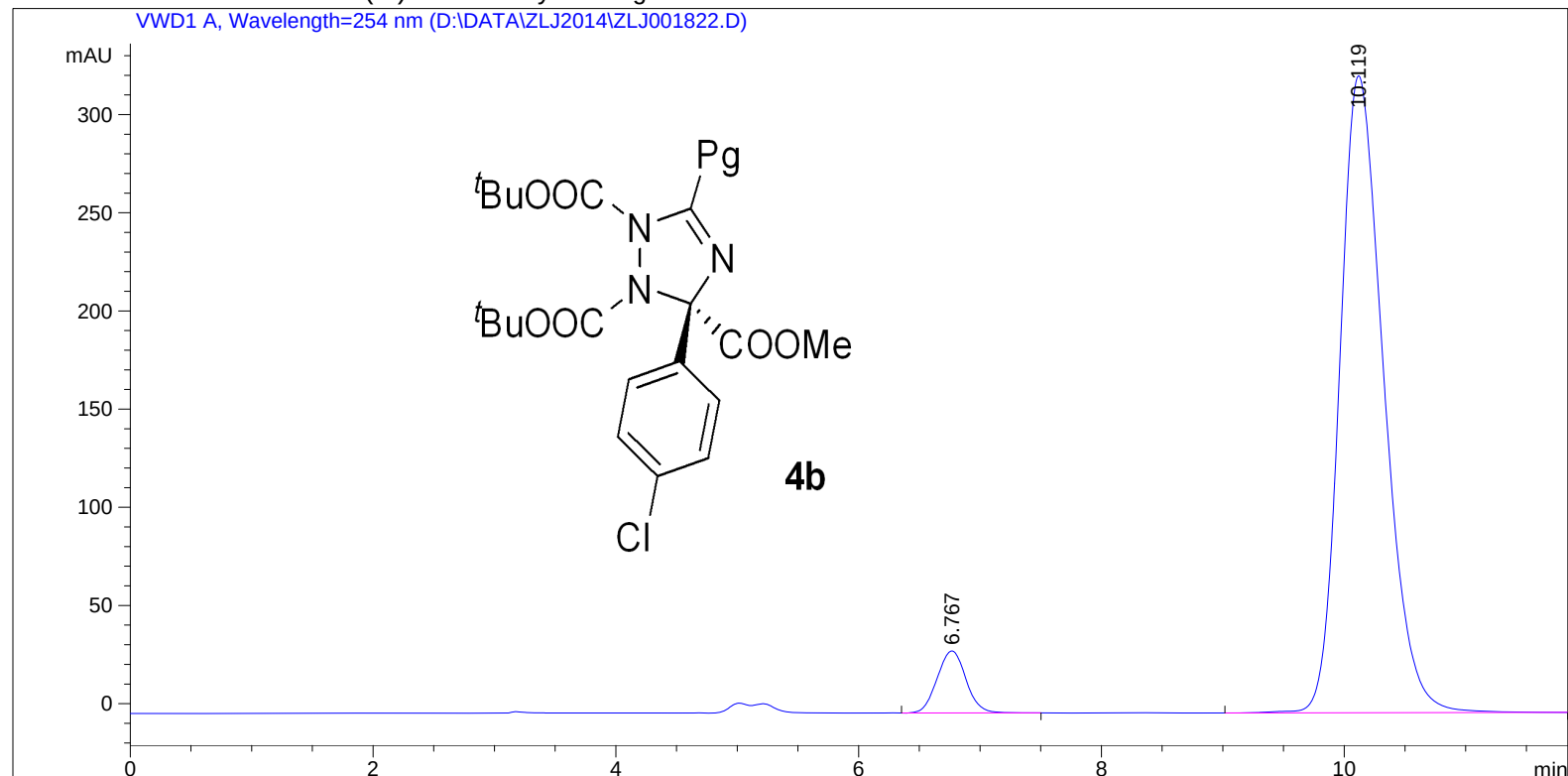
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.738	BB	0.2424	2906.68896	187.11411	50.2754
2	10.081	BB	0.3656	2874.84009	121.16588	49.7246

Totals : 5781.52905 308.27998

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*** End of Report ***

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Additional Info : Peak(s) manually integrated



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Area Percent Report
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Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

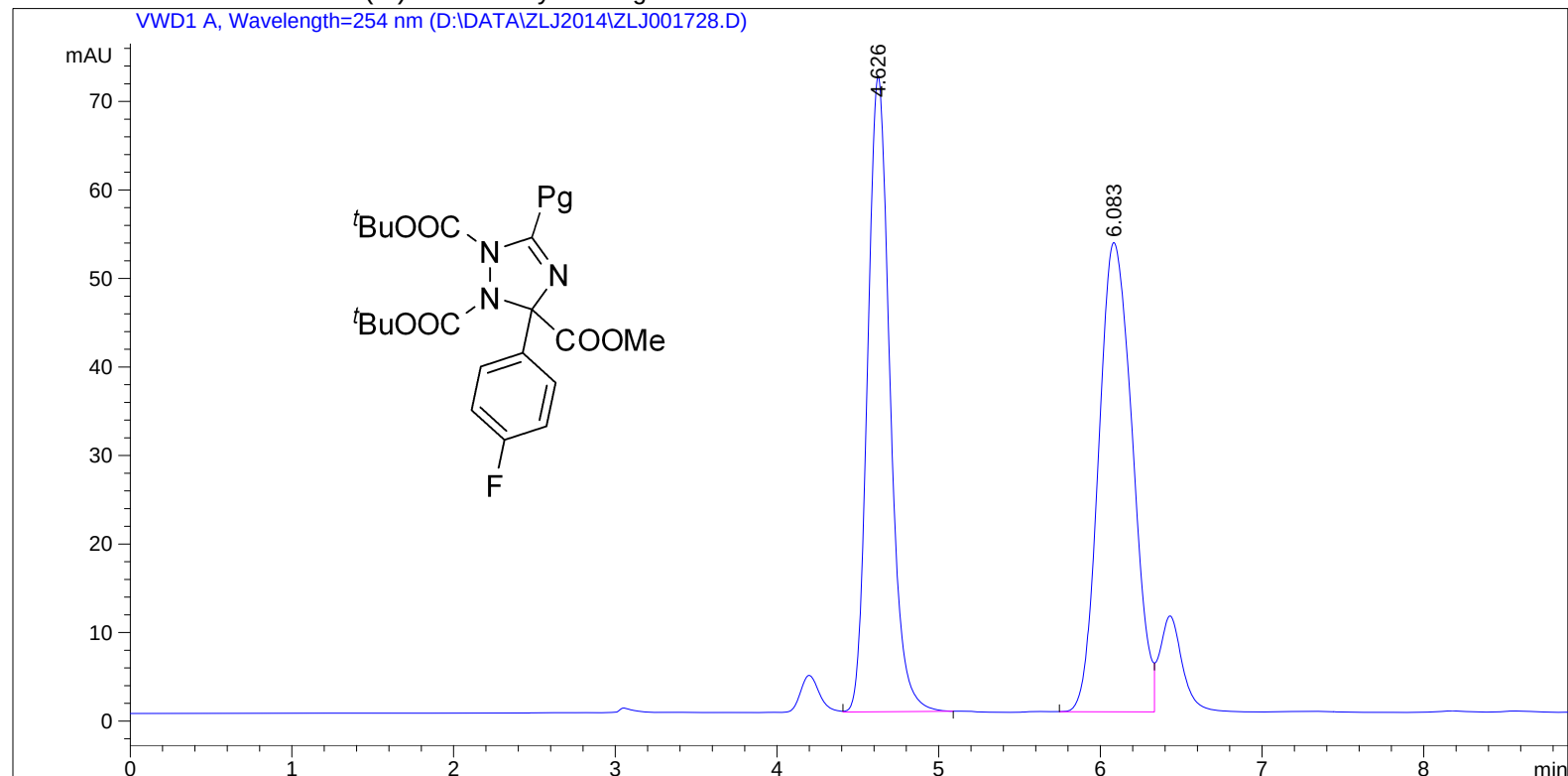
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Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.767	BB	0.2590	519.73267	31.57753	6.0135
2	10.119	BBA	0.3853	8123.09033	324.41058	93.9865

Totals : 8642.82300 355.98811

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*** End of Report ***

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Acq. Instrument : Instrument 1
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Location : Vial 1
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Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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Area Percent Report
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Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

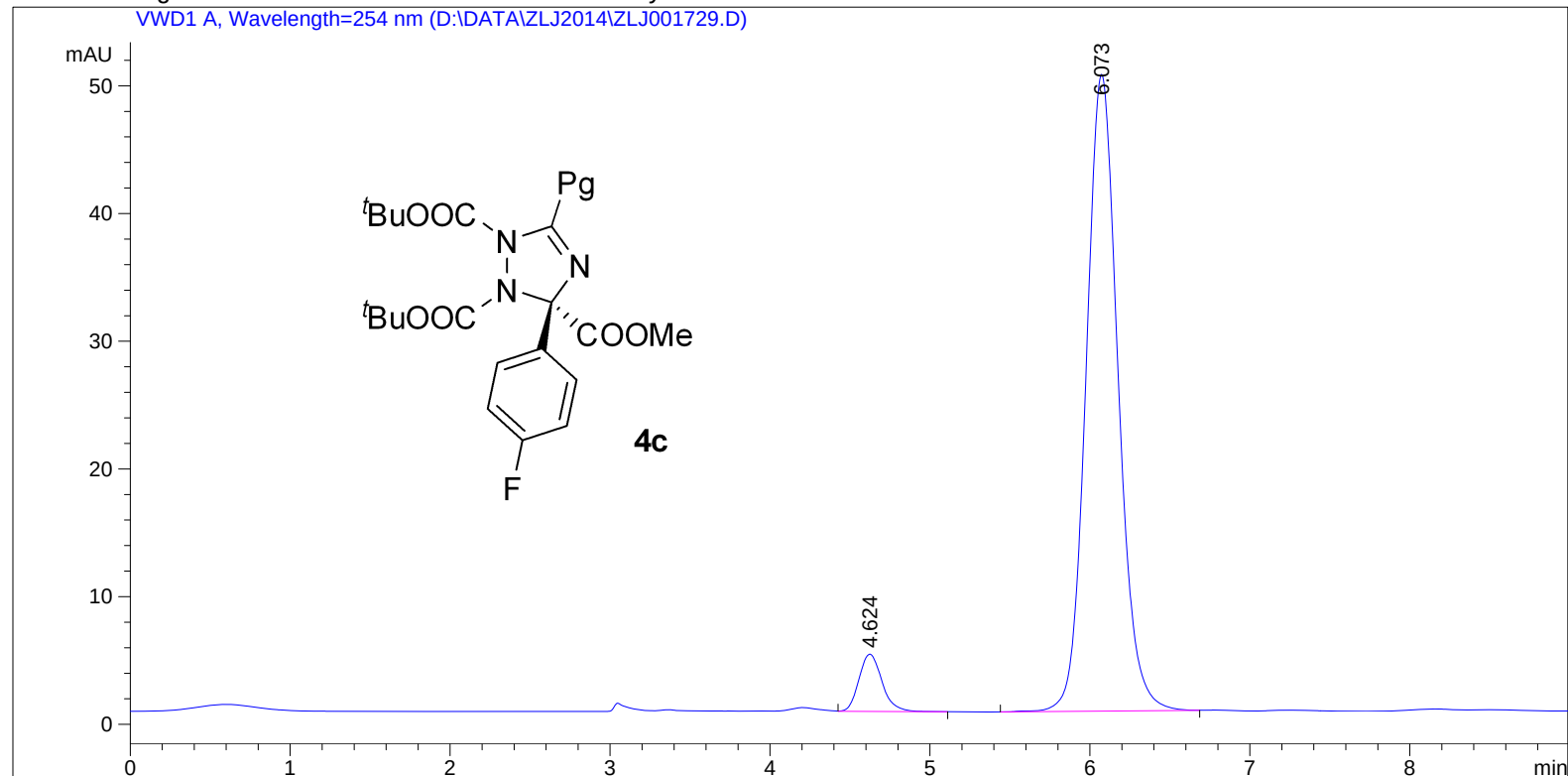
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1	4.626	VB	0.1480	687.45642	71.85688	47.2690
2	6.083	BV	0.2328	766.89417	53.01987	52.7310

Totals : 1454.35059 124.87675

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*** End of Report ***

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Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 5/4/2014 5:12:00 PM Inj Volume : No inj

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Last changed : 5/4/2014 5:10:15 PM by LNF
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Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF



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Area Percent Report
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Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

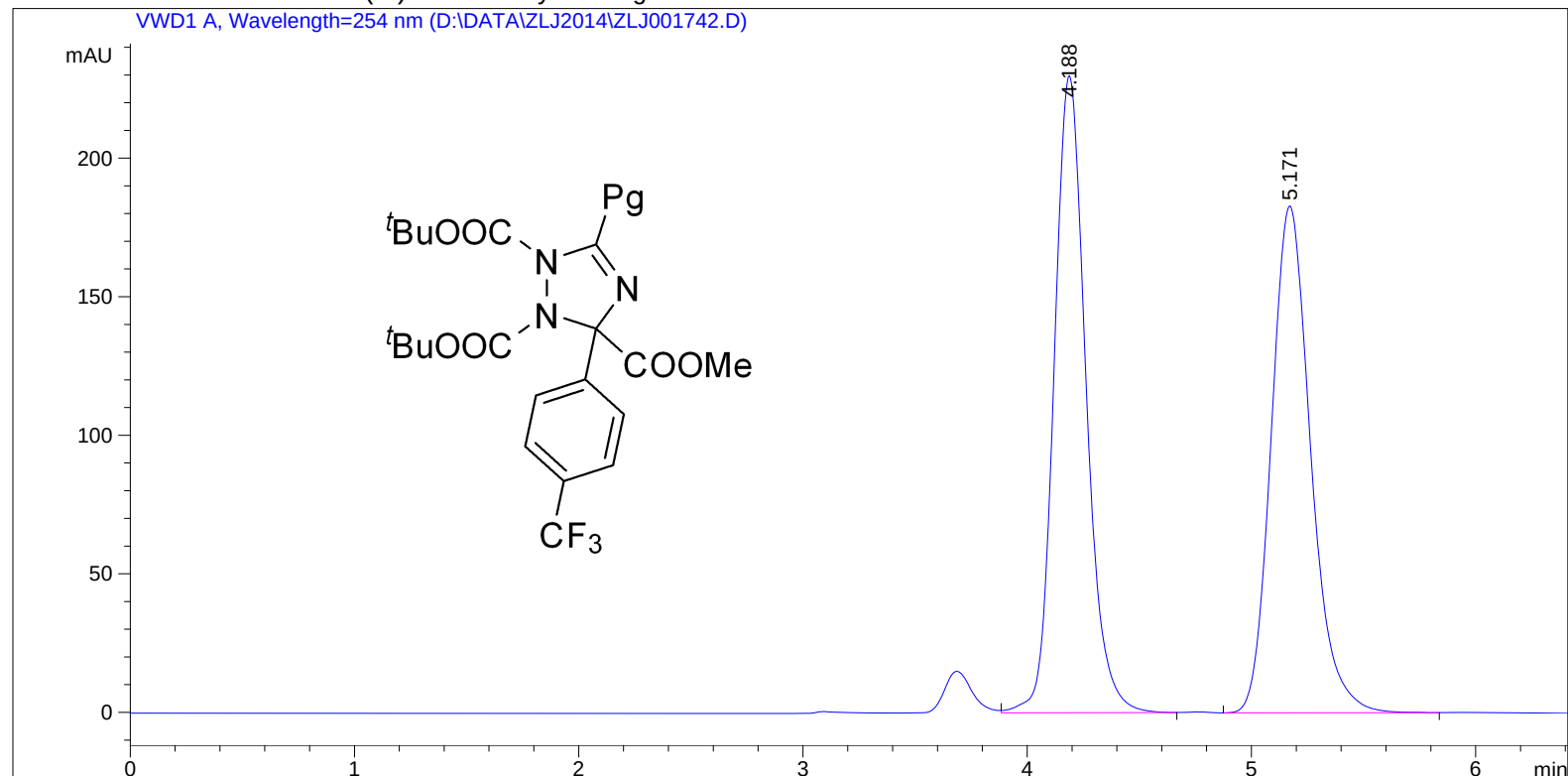
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.624	VB	0.1564	45.06038	4.48883	6.1149
2	6.073	BB	0.2128	691.84021	49.86996	93.8851

Totals : 736.90059 54.35879

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*** End of Report ***

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Location : Vial 1
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Additional Info : Peak(s) manually integrated



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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

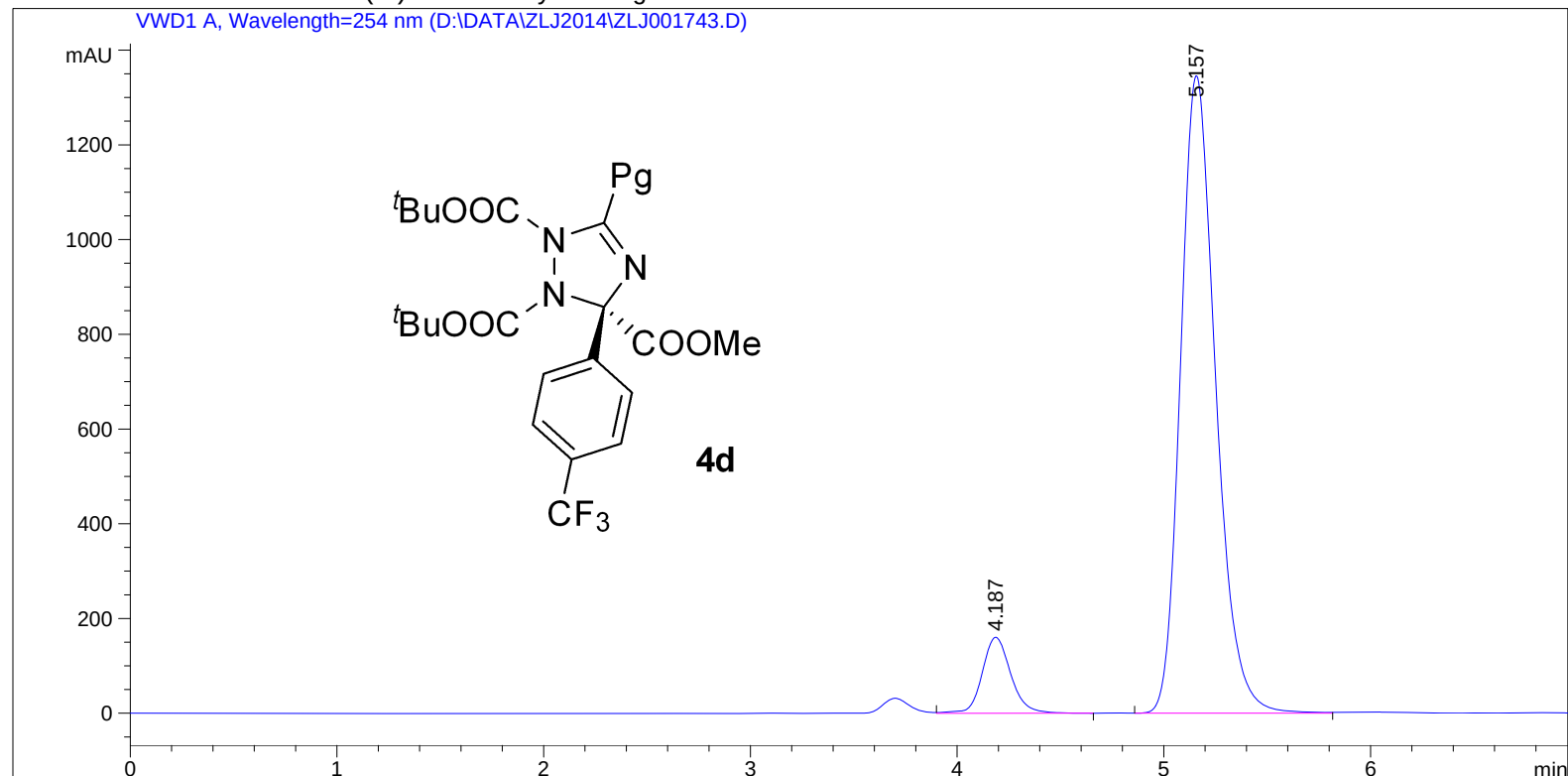
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.188	VB	0.1533	2275.24023	229.90190	50.4552
2	5.171	BB	0.1876	2234.18481	182.92796	49.5448

Totals : 4509.42505 412.82986

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*** End of Report ***

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Acq. Instrument : Instrument 1
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Location : Vial 1
Inj Volume : No inj
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Last changed : 5/6/2014 4:55:03 PM by LNF
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Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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Area Percent Report
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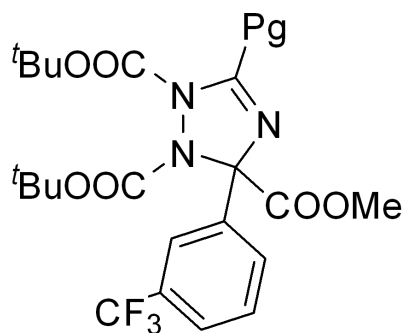
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Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.187	VB	0.1470	1540.01611	160.35957	8.8369
2	5.157	BV	0.1814	1.58872e4	1346.20776	91.1631

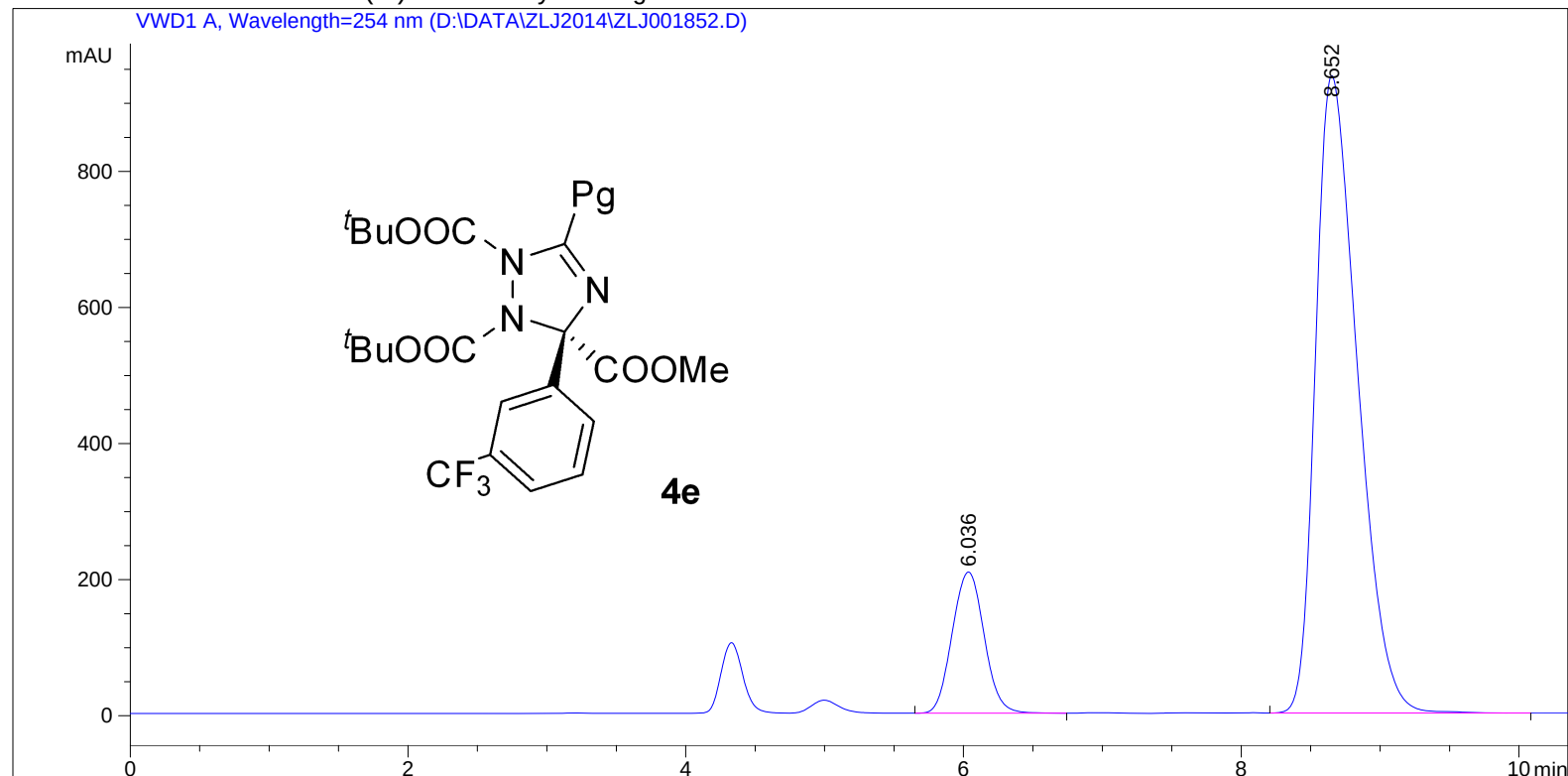
Totals : 1.74272e4 1506.56734

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*** End of Report ***



Page 1 of 1

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Injection Date : 7/8/2014 9:45:56 AM
Location : Vial 1
Inj Volume : No inj
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(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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: VWD1 A, Wavelength=254 nm

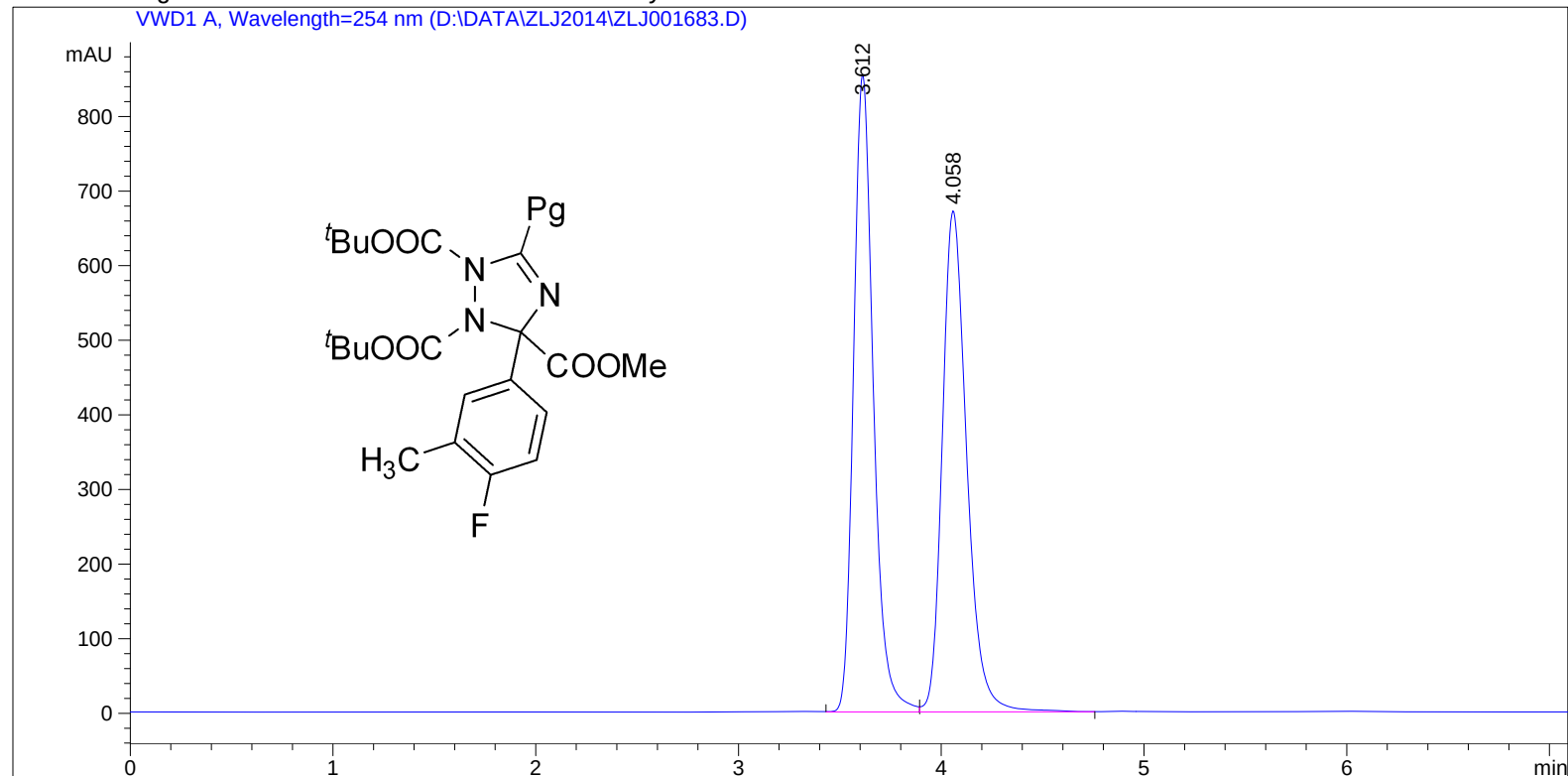
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.036	BB	0.2535	3316.97510	207.42863	14.4322
2	8.652	VB	0.3250	1.96662e4	936.65894	85.5678

Totals : 2.29831e4 1144.08757

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*** End of Report ***

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Last changed : 3/25/2013 8:45:53 AM by LNF
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Area Percent Report
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Dilution: : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=254 nm

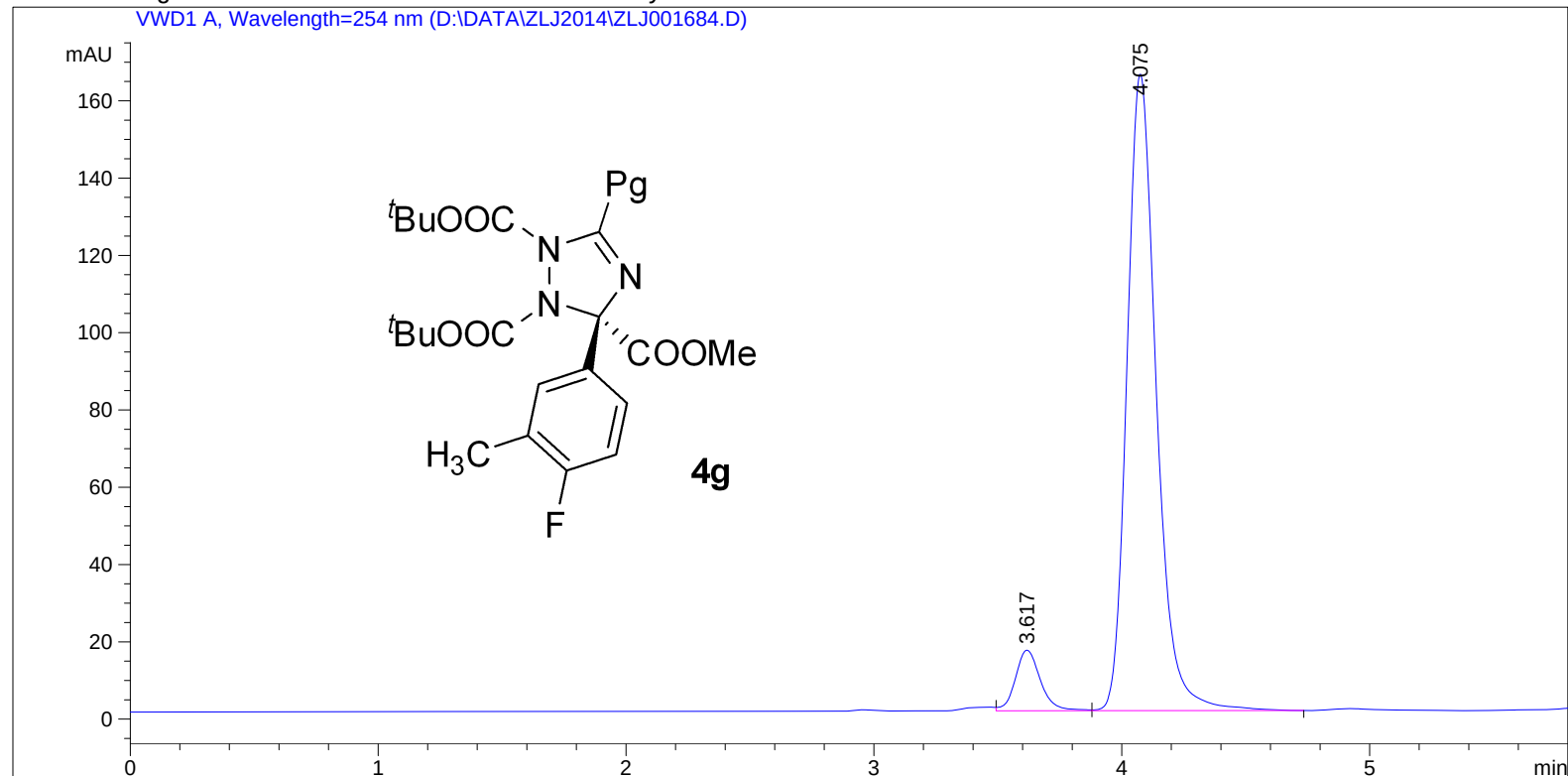
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.612	VV	0.1037	5788.96484	855.16876	50.6448
2	4.058	VV	0.1286	5641.56250	671.79053	49.3552

Totals : 1.14305e4 1526.95929

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*** End of Report ***
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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/23/2014 11:27:31 AM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/23/2014 11:25:48 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
=====



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

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

Signal 1: VWD1 A, Wavelength=254 nm

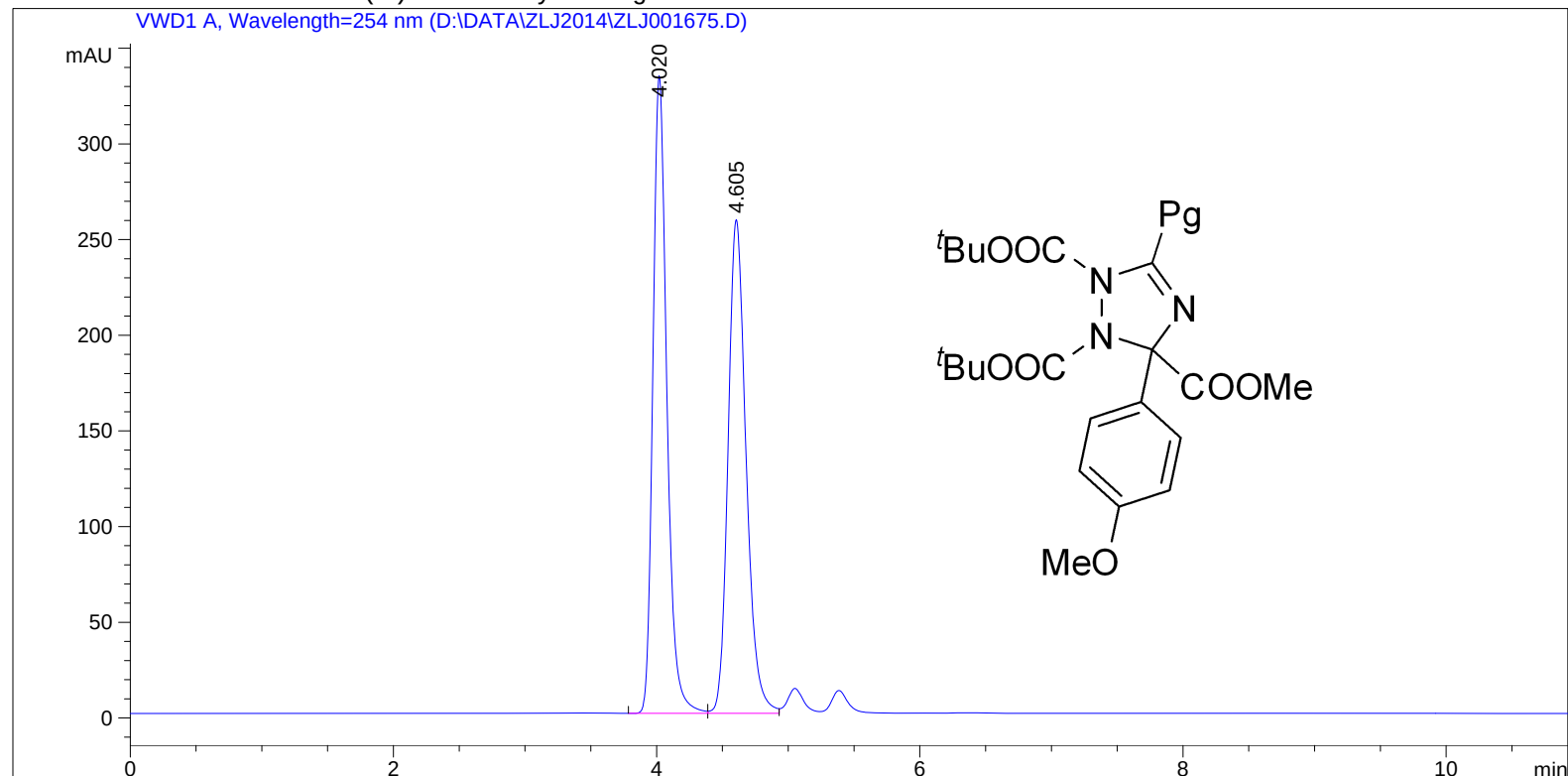
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.617	VV	0.1081	110.13467	15.69066	7.5387
2	4.075	VB	0.1247	1350.79871	164.81862	92.4613

Totals : 1460.93337 180.50928

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/21/2014 1:23:11 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/21/2014 1:20:55 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

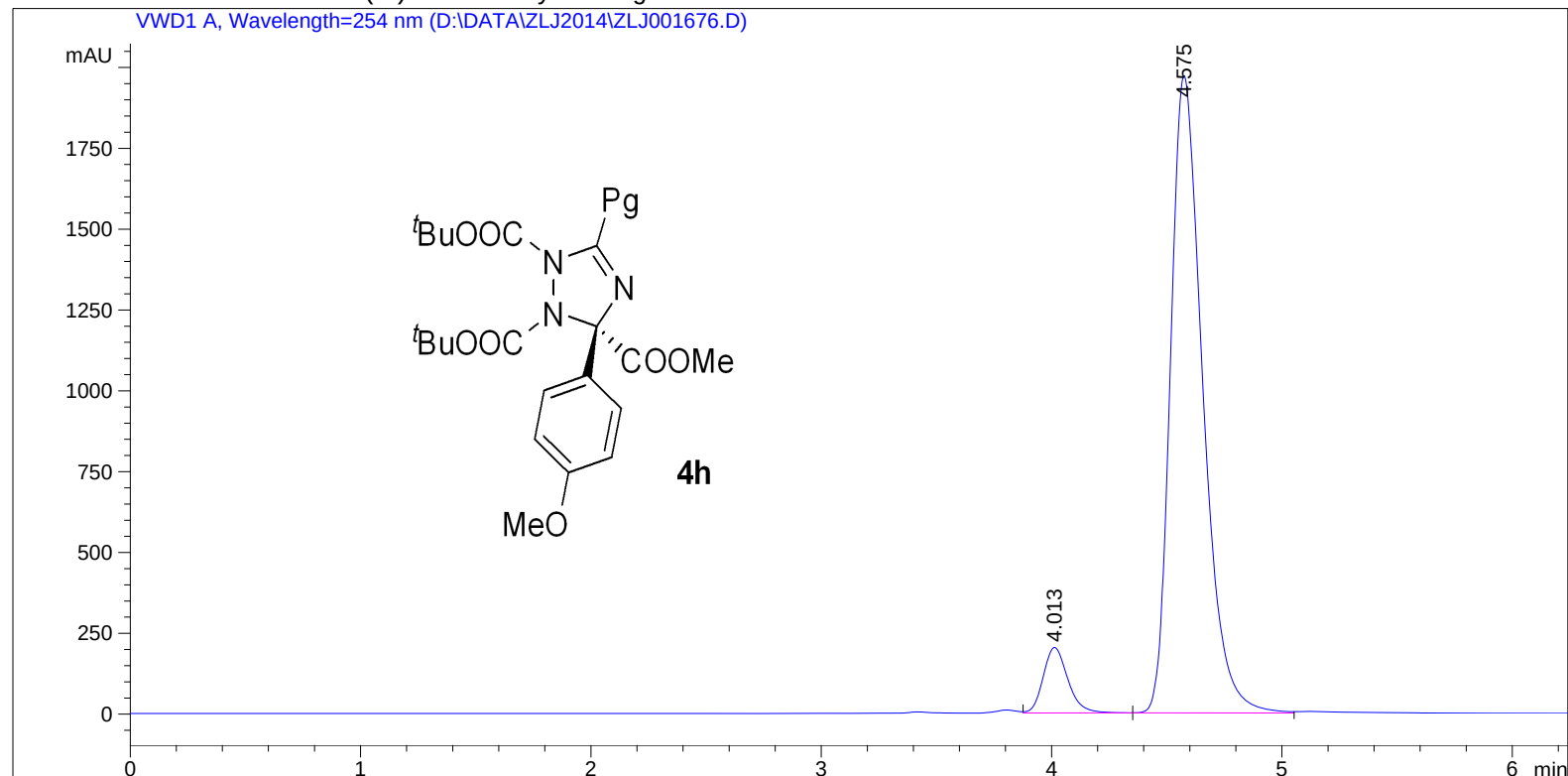
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.020	BV	0.1138	2460.87354	333.32416	50.0361
2	4.605	VV	0.1475	2457.32007	258.00714	49.9639

Totals : 4918.19360 591.33130

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/21/2014 1:36:22 PM
Inj Volume : No inj
Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/21/2014 1:34:13 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

Signal 1: VWD1 A, Wavelength=254 nm

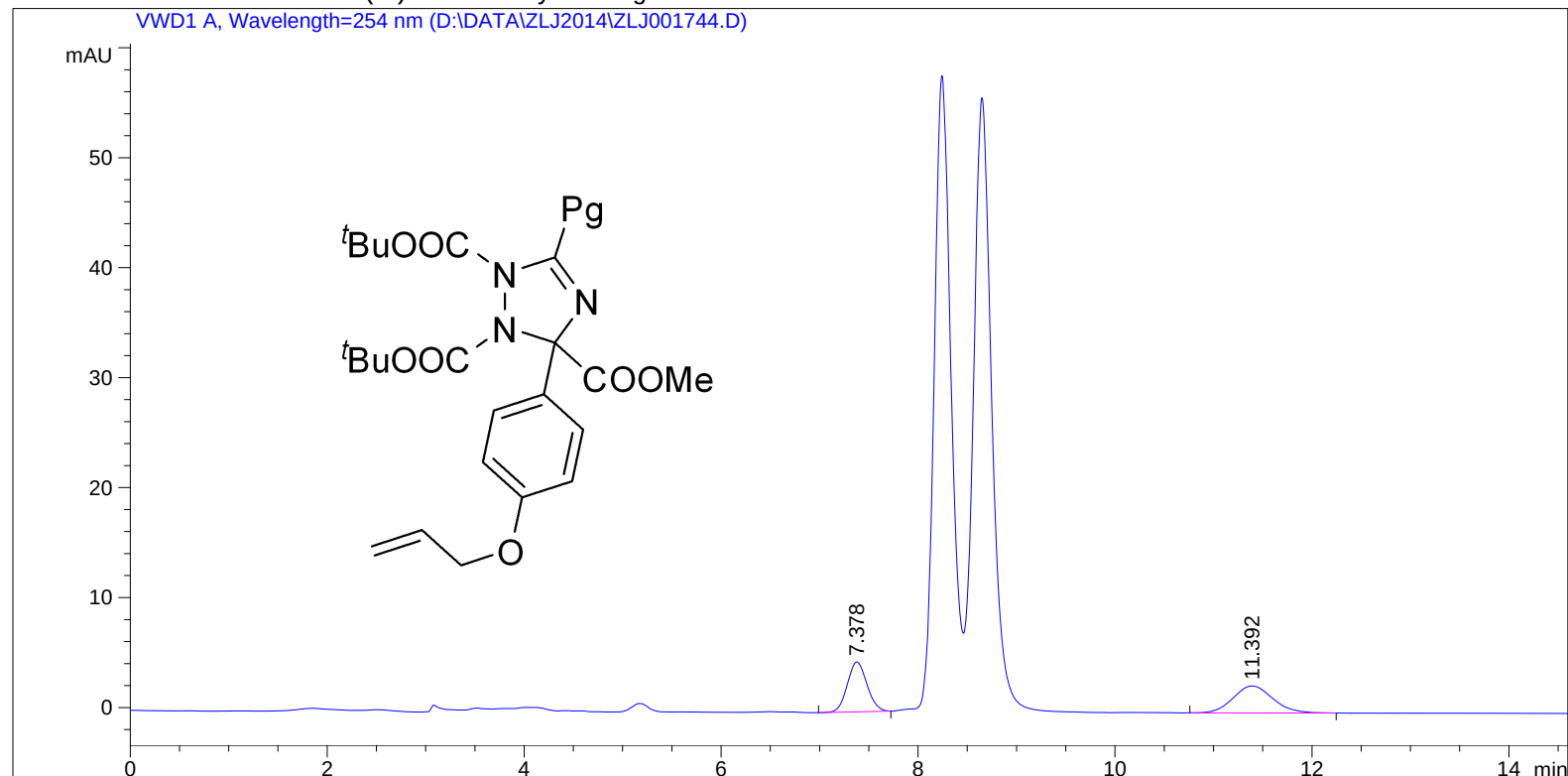
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.013	VB	0.1143	1504.24976	202.65331	7.2878
2	4.575	BV	0.1496	1.91363e4	1971.55127	92.7122

Totals : 2.06406e4 2174.20457

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 5/6/2014 5:03:01 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 5/6/2014 5:02:20 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

Signal 1: VWD1 A, Wavelength=254 nm

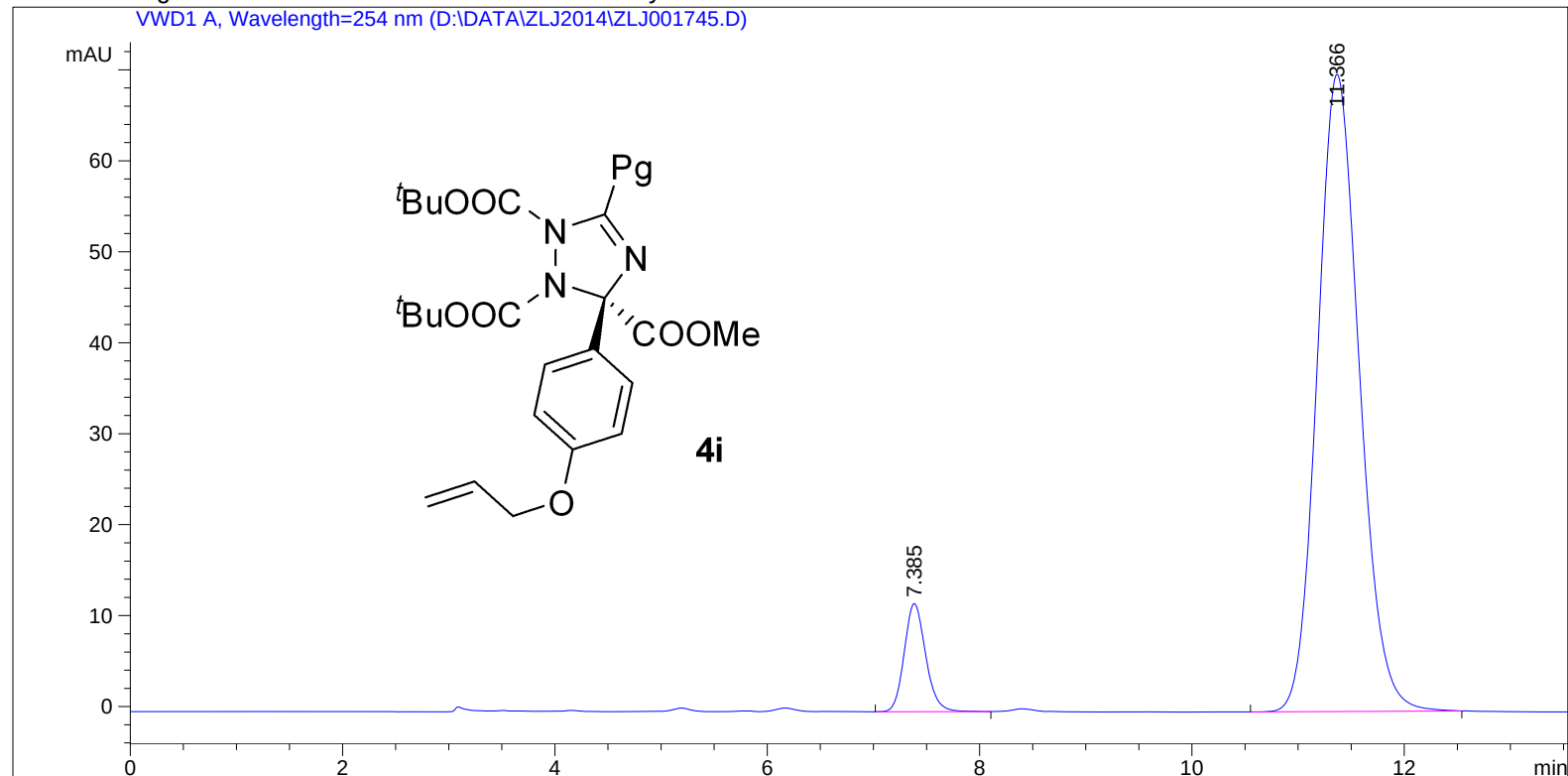
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.378	BB	0.2137	62.58542	4.52855	47.6268
2	11.392	BB	0.4256	68.82261	2.45827	52.3732

Totals : 131.40803 6.98683

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 5/6/2014 5:18:43 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 5/6/2014 5:17:41 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF



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Area Percent Report
=====

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

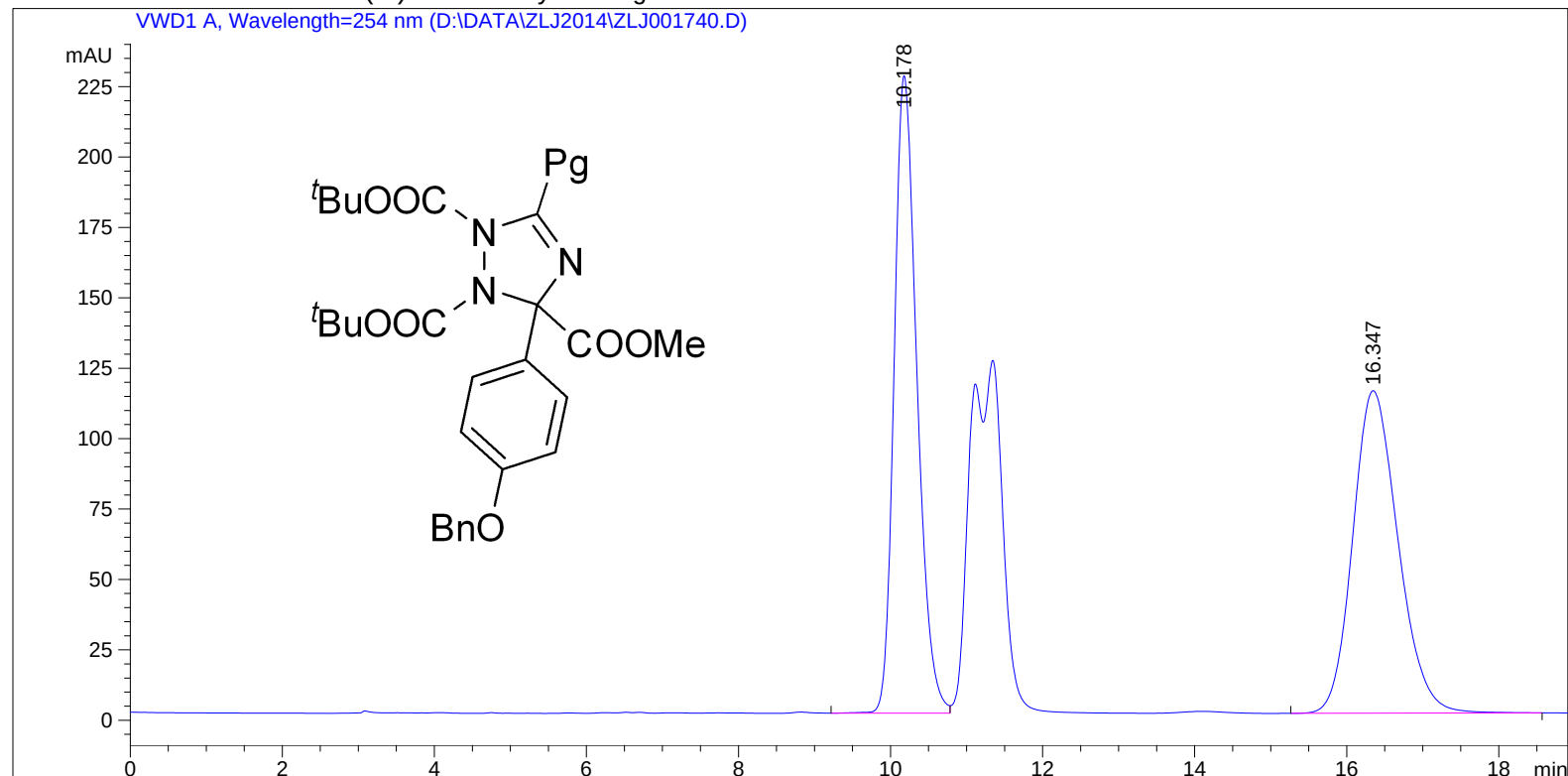
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.385	BB	0.2158	166.41368	11.88510	7.9572
2	11.366	BB	0.4254	1924.94958	70.05244	92.0428

Totals : 2091.36327 81.93754

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1
Injection Date : 5/6/2014 3:57:10 PM
Location : Vial 1
Inj Volume : No inj
Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 5/6/2014 3:56:50 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

Signal 1: VWD1 A, Wavelength=254 nm

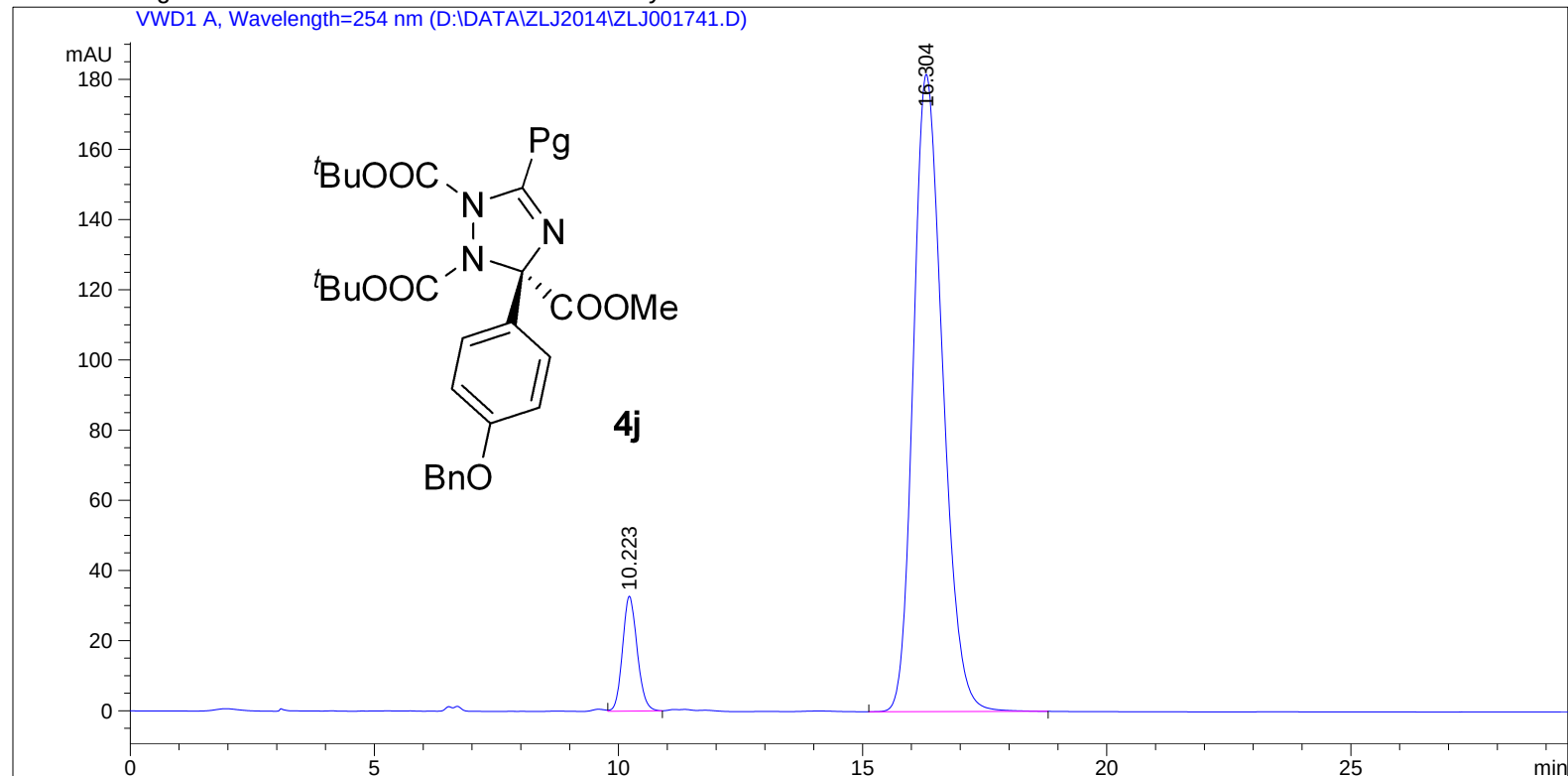
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.178	BV	0.3204	4718.20703	226.33710	49.8848
2	16.347	BB	0.6431	4740.00293	114.49794	50.1152

Totals : 9458.20996 340.83504

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*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 5/6/2014 4:16:26 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 5/6/2014 4:16:10 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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Area Percent Report
=====

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

Signal 1: VWD1 A, Wavelength=254 nm

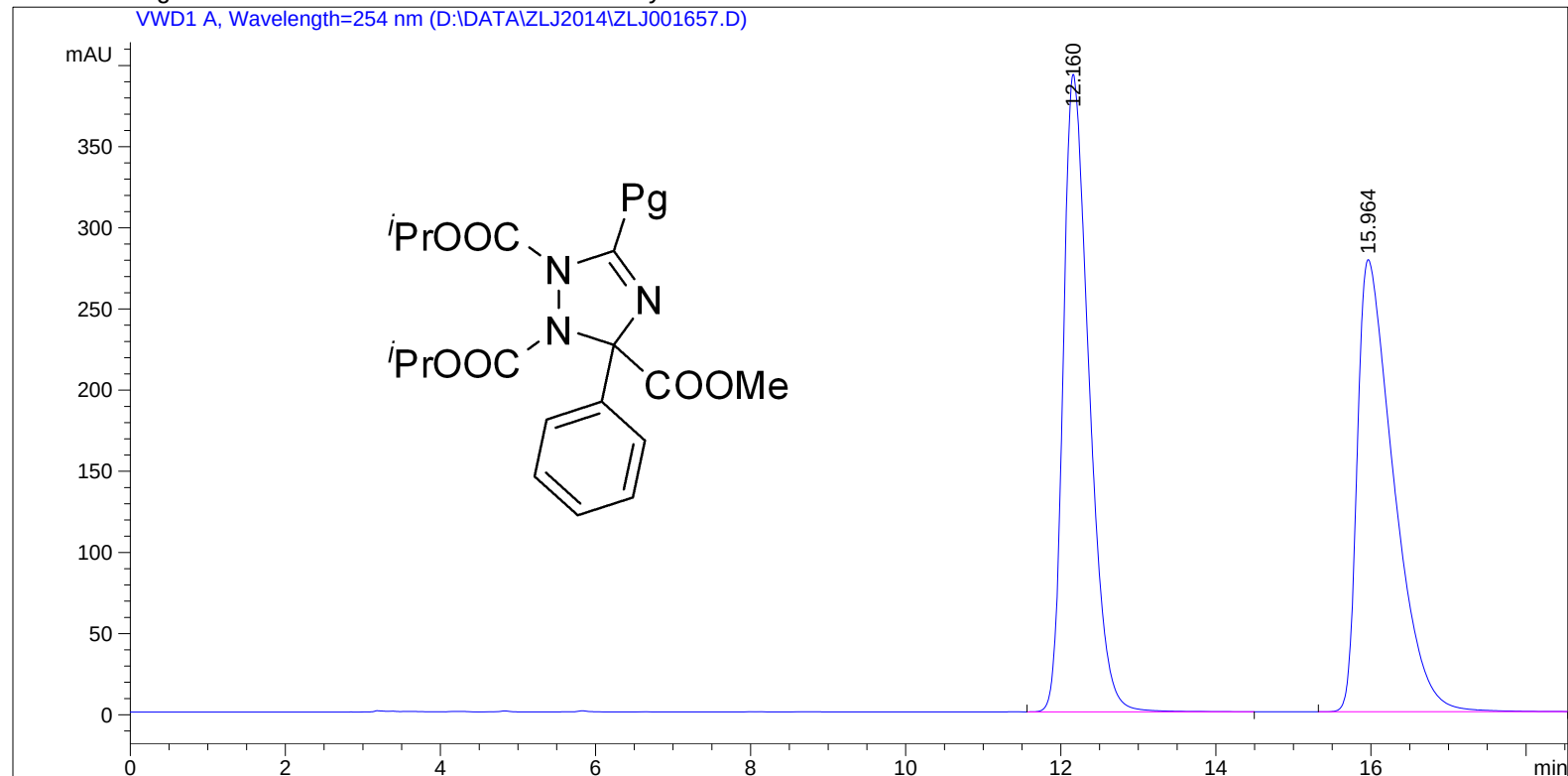
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.223	VB	0.3204	682.43591	32.73026	8.2476
2	16.304	BB	0.6449	7591.90771	181.64870	91.7524

Totals : 8274.34363 214.37896

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*** End of Report ***
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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/18/2014 10:57:07 AM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/18/2014 10:56:36 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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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: VWD1 A, Wavelength=254 nm

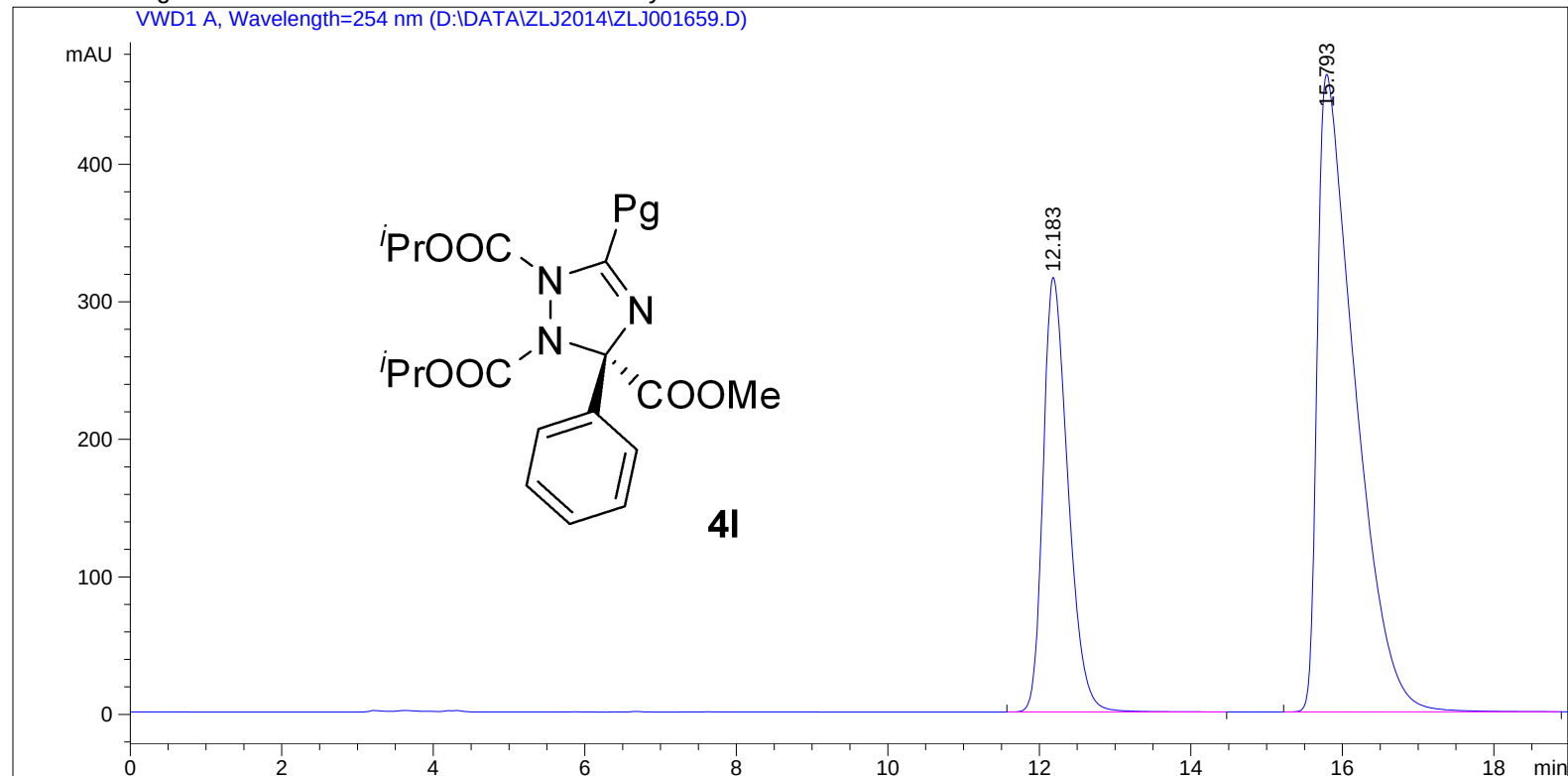
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.160	BB	0.3542	9081.00781	392.79745	50.0846
2	15.964	BBA	0.4897	9050.34277	278.54248	49.9154

Totals : 1.81314e4 671.33994

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*** End of Report ***
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=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/18/2014 11:33:27 AM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/18/2014 11:33:07 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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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

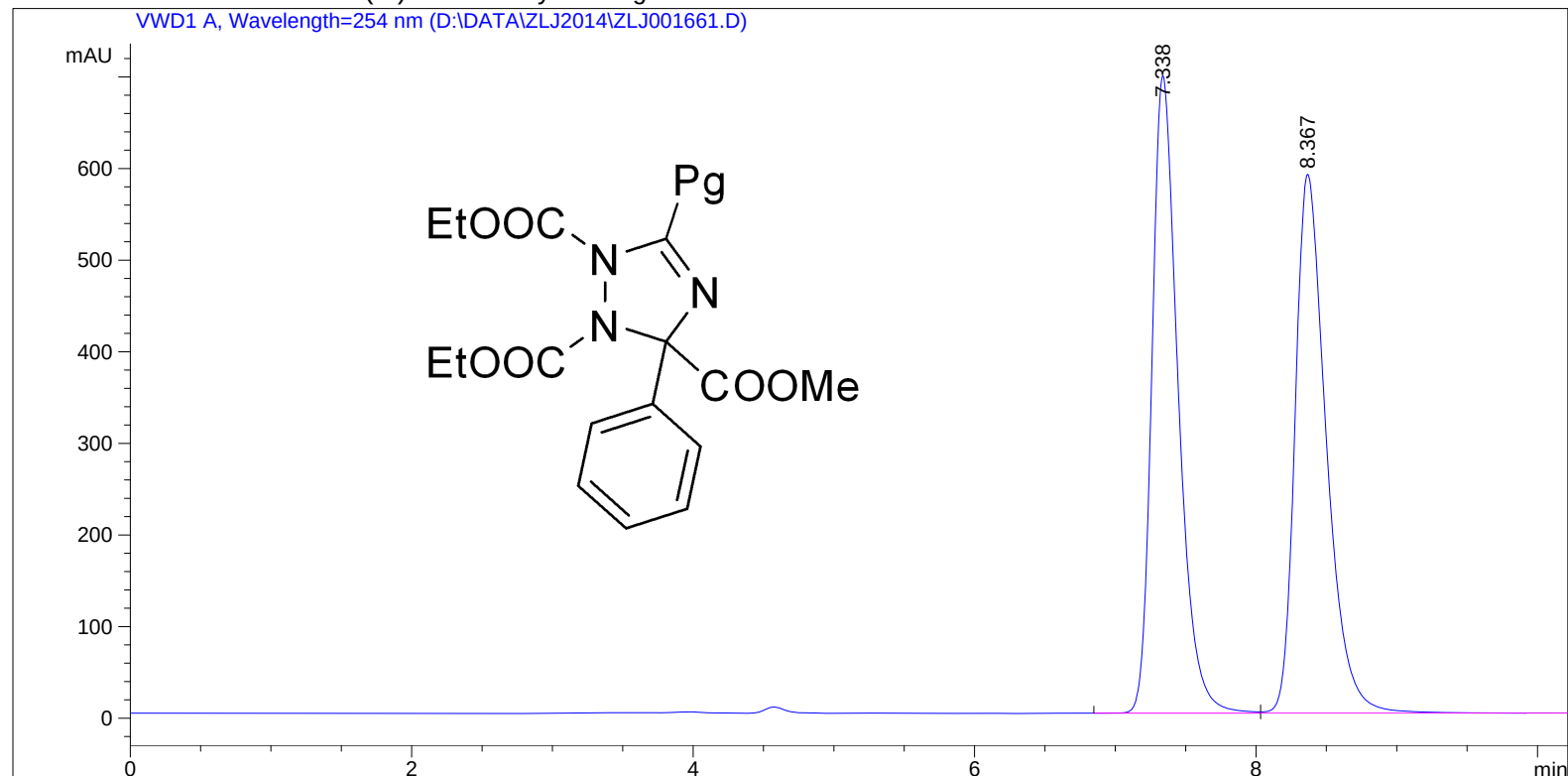
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.183	BB	0.3510	7221.13672	316.02963	31.1135
2	15.793	BB	0.5028	1.59879e4	463.60297	68.8865

Totals : 2.32090e4 779.63260

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*** End of Report ***
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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/20/2014 11:13:18 AM
Inj Volume : No inj
Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/20/2014 11:07:07 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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Area Percent Report
=====

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

Signal 1: VWD1 A, Wavelength=254 nm

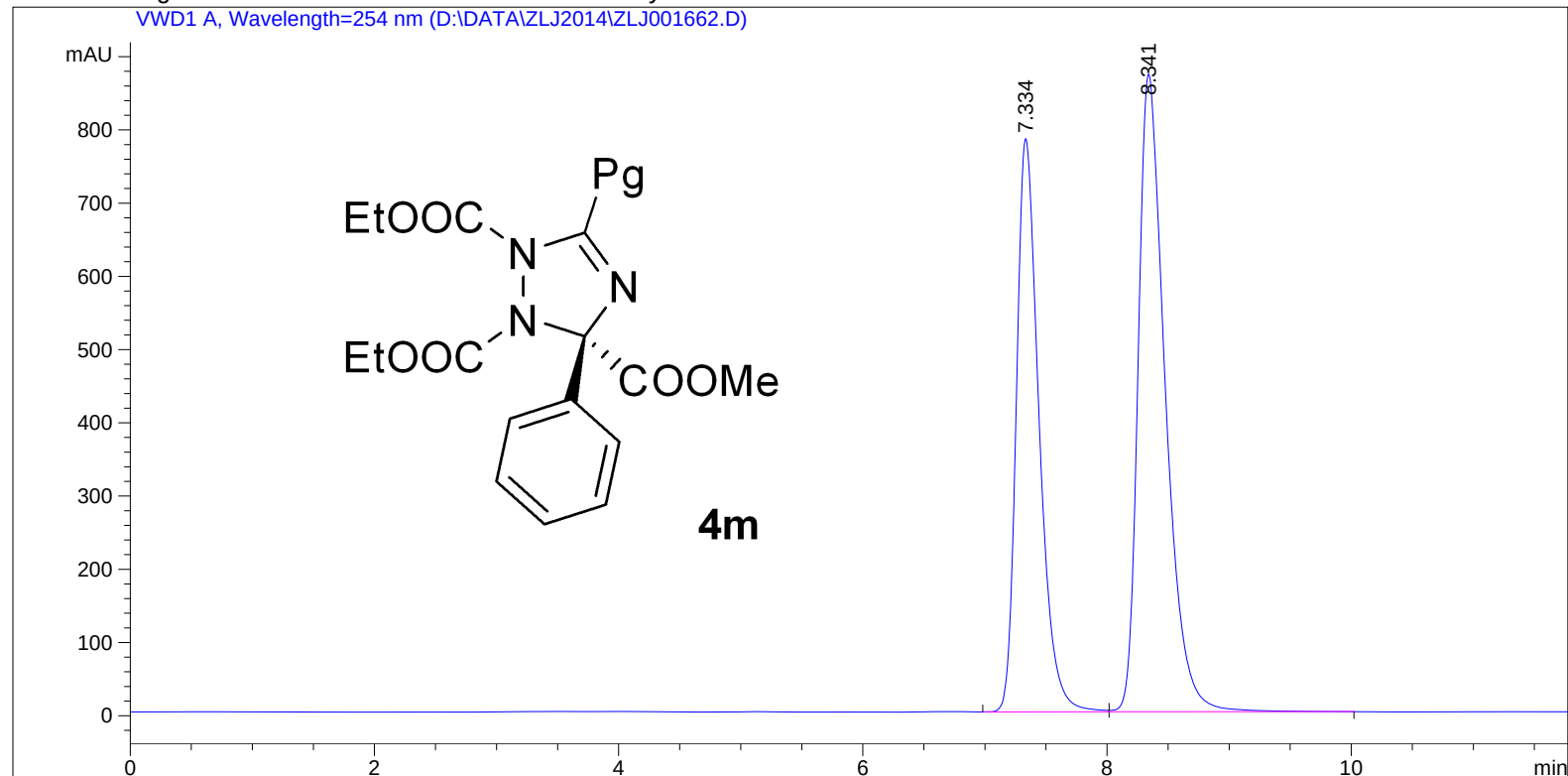
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.338	BV	0.1983	9047.70020	695.78363	49.9341
2	8.367	VBA	0.2338	9071.59473	588.21442	50.0659

Totals : 1.81193e4 1283.99805

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 4/20/2014 11:24:06 AM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 4/20/2014 11:23:35 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
=====



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Area Percent Report
=====

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

Signal 1: VWD1 A, Wavelength=254 nm

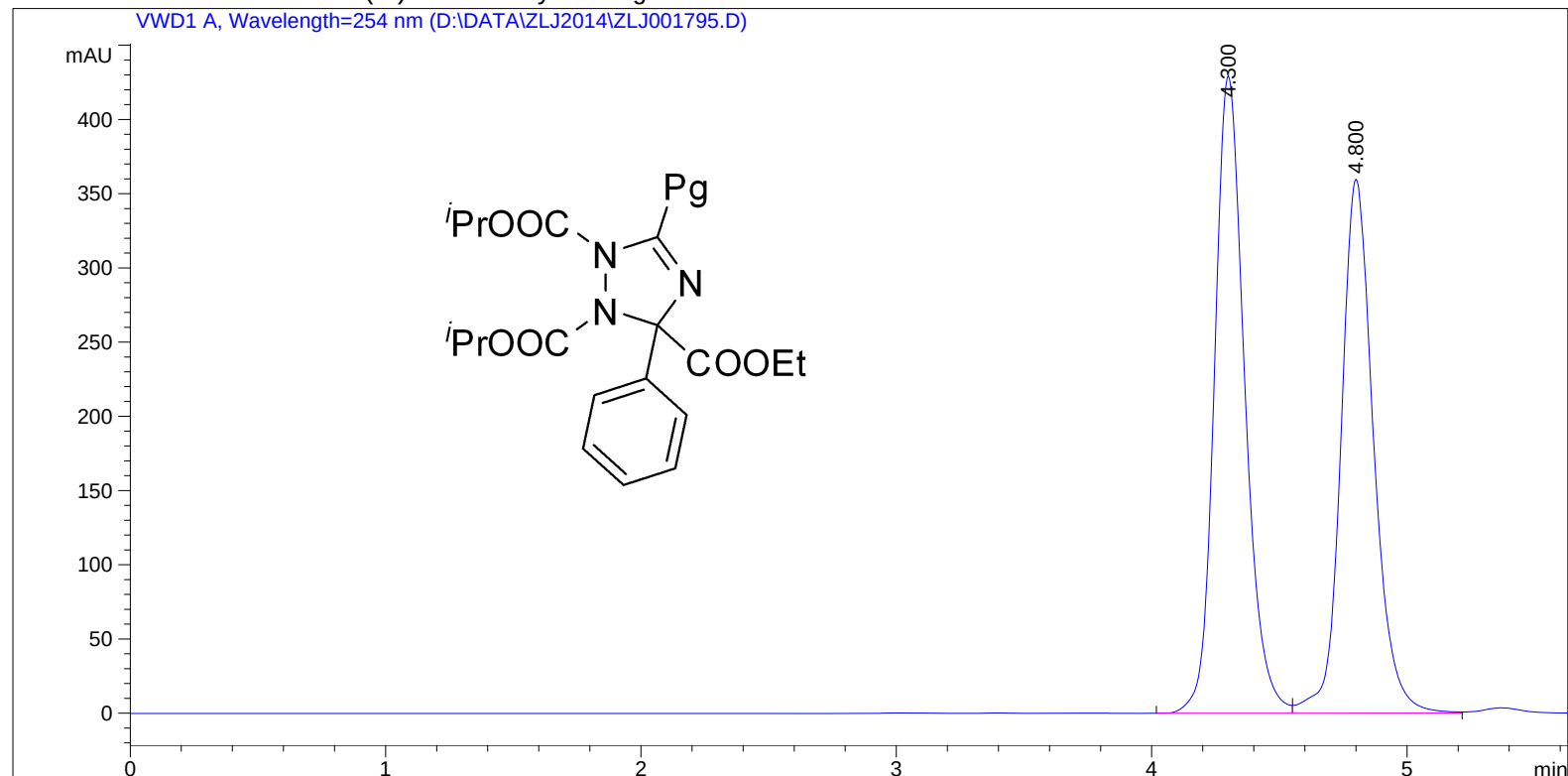
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.334	VV	0.1958	1.01129e4	782.96552	42.9071
2	8.341	VB	0.2327	1.34564e4	870.77081	57.0929

Totals : 2.35694e4 1653.73633

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 6/3/2014 3:59:14 PM
Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 6/3/2014 3:56:08 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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Area Percent Report
=====

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

Signal 1: VWD1 A, Wavelength=254 nm

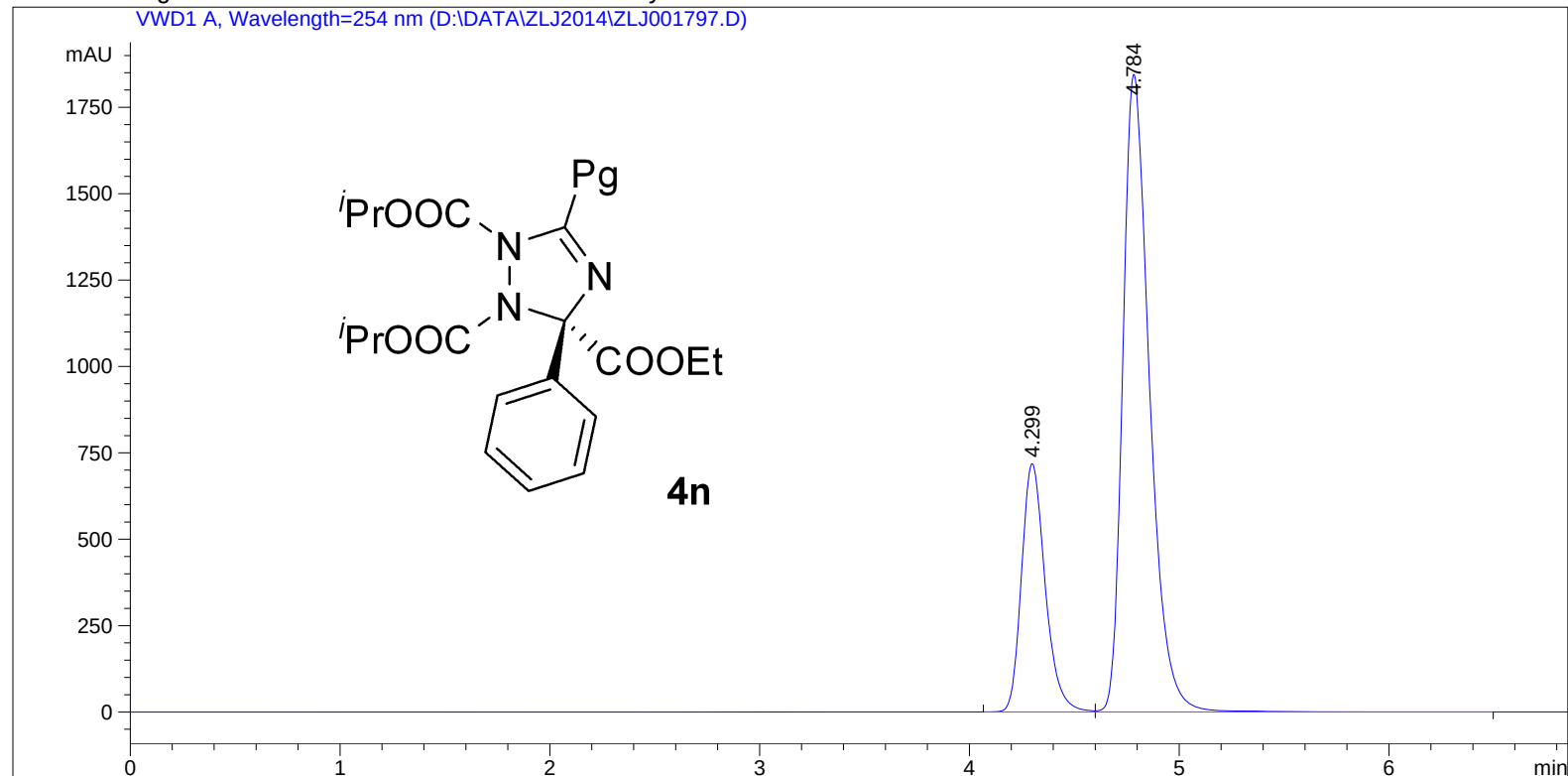
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.300	BV	0.1266	3536.51465	429.48553	52.5212
2	4.800	VV	0.1343	3196.98828	359.71152	47.4788

Totals : 6733.50293 789.19705

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 6/3/2014 4:13:42 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 6/3/2014 4:13:27 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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=====
Area Percent Report
=====

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

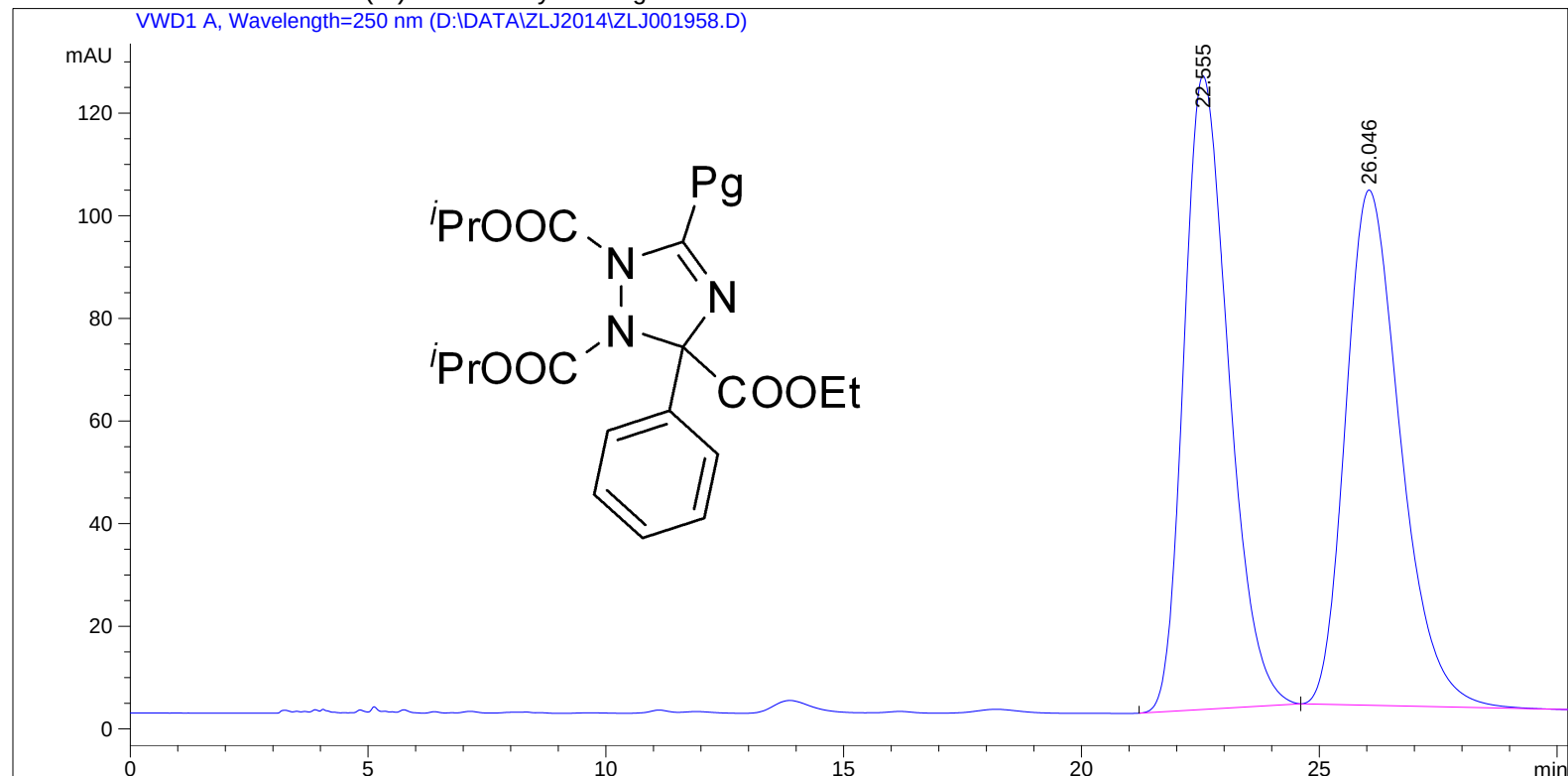
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.299	BV	0.1205	5634.46191	719.10120	25.6071
2	4.784	VB	0.1369	1.63691e4	1845.90002	74.3929

Totals : 2.20035e4 2565.00122

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*** End of Report ***
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=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 6:04:54 PM
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 6:04:43 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

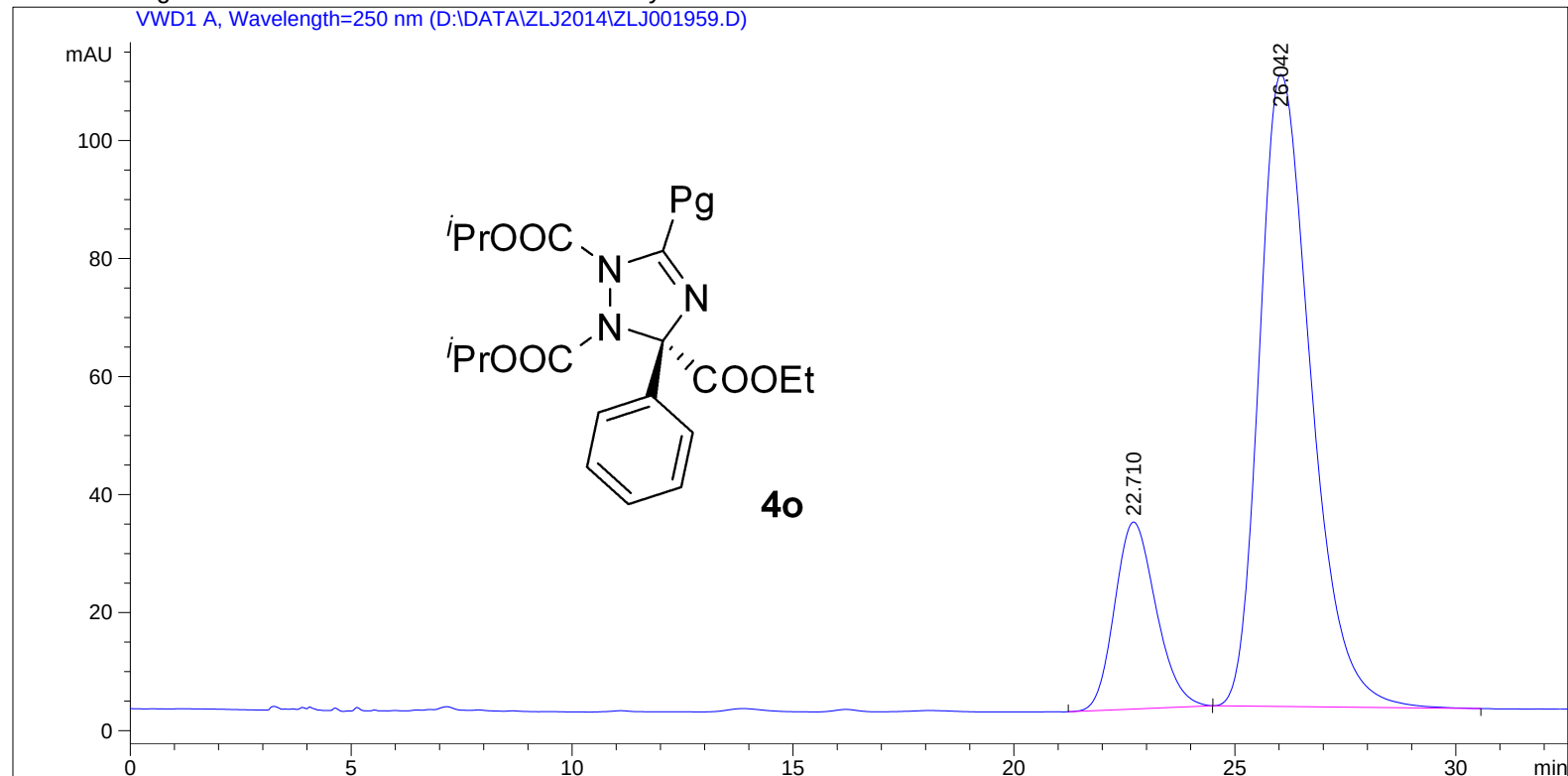
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.555	BB	1.0095	8096.26074	123.48978	50.1934
2	26.046	BBA	1.2305	8033.86816	100.40849	49.8066

Totals : 1.61301e4 223.89827

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 6:35:31 PM Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 6:35:13 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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=====
Area Percent Report
=====

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

Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.710	BB	1.0048	2060.61987	31.68546	19.1537
2	26.042	BB	1.2239	8697.69238	107.11949	80.8463

Totals : 1.07583e4 138.80495

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

Acq. Operator : LNF

Location : Vial 1

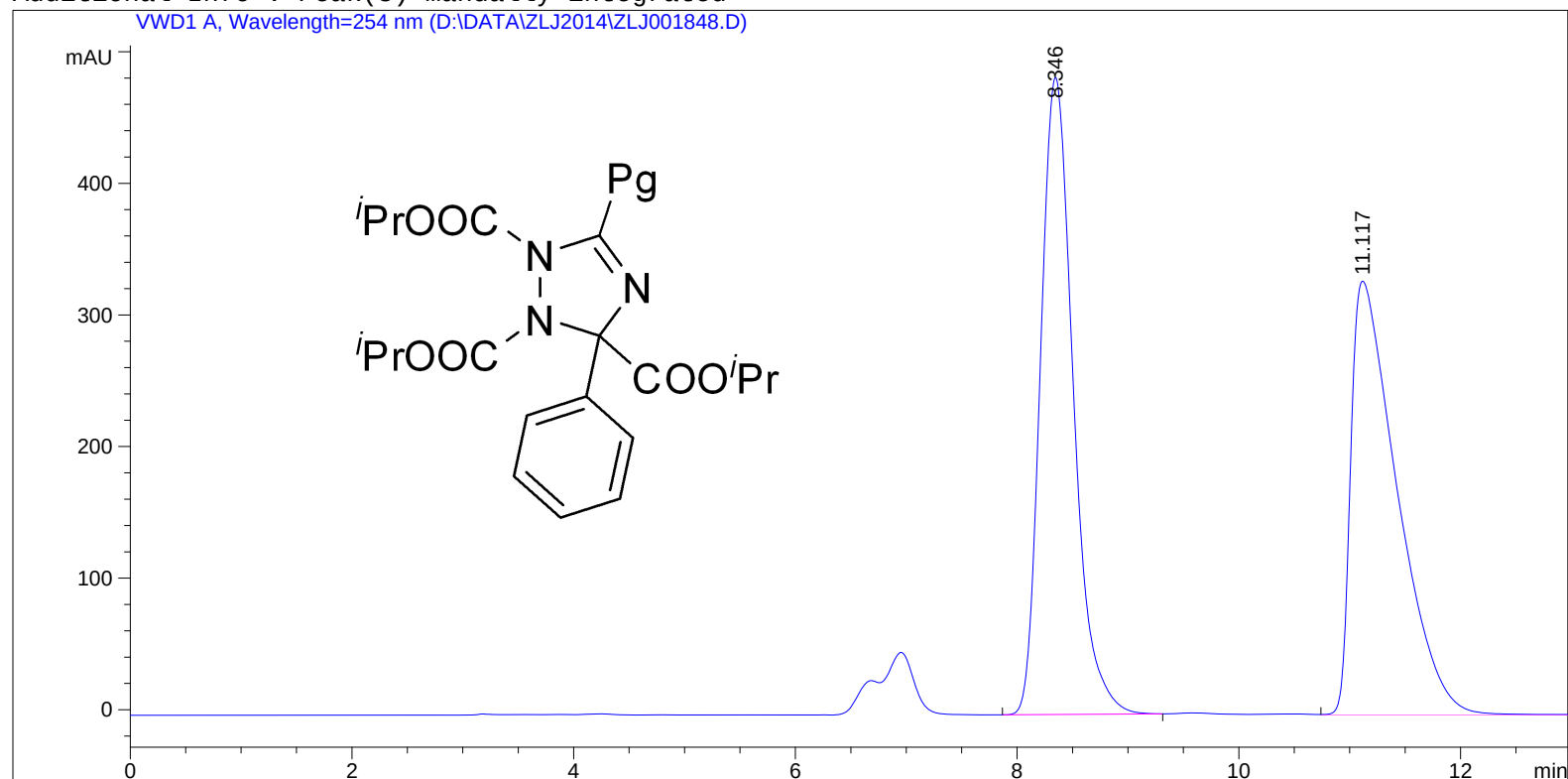
Injection Date : 7/4/2014 9:20:50 PM

Acq. Method : IA_20_1.M

Analysis Method : D:\METHOD\OD_20_1.M

Last changed : 3/25/2013 8:45:53 AM by LNF

Additional Info : Peak(s) manually integrated



Area Percent Report

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

Signal 1: VWD1 A, Wavelength=254 nm

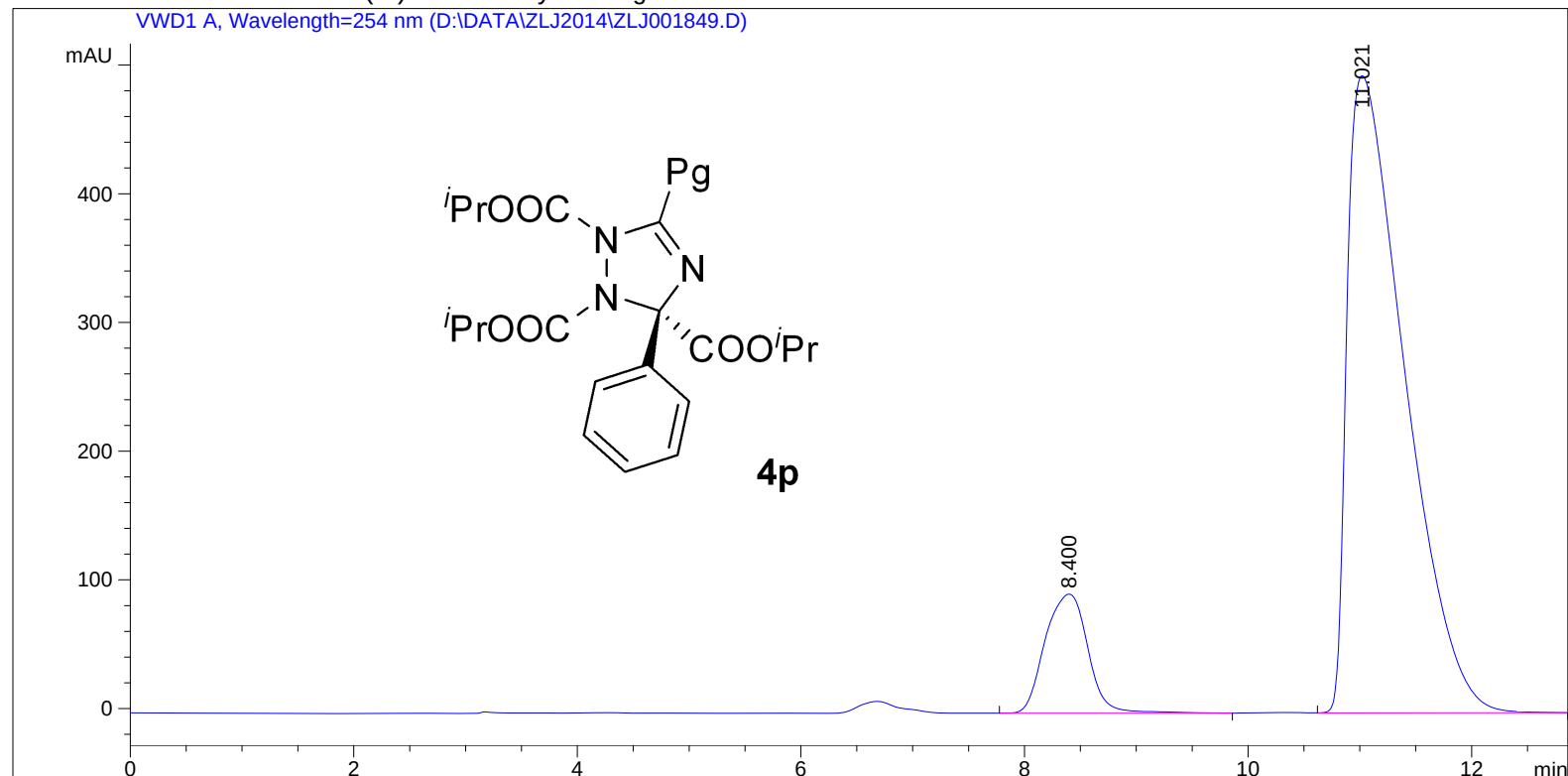
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.346	BB	0.3181	9874.78320	484.03918	51.0249
2	11.117	BBA	0.4261	9478.08887	329.28870	48.9751

Totals : 1.93529e4 813.32788

*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 7/4/2014 9:34:17 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 7/4/2014 9:33:53 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

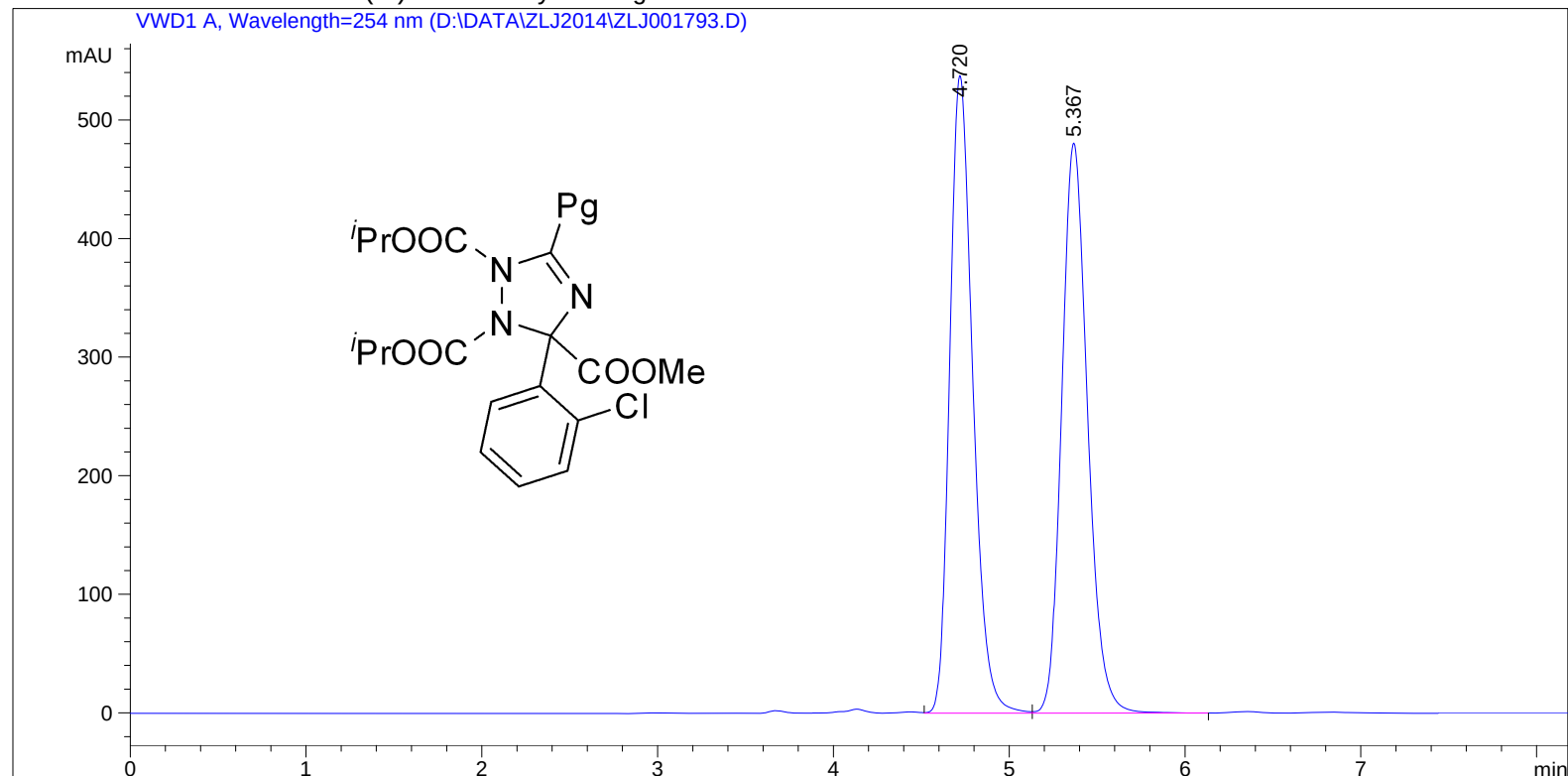
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.400	BB	0.4356	2460.02515	92.45815	12.1770
2	11.021	VBA	0.5535	1.77422e4	495.08545	87.8230

Totals : 2.02022e4 587.54359

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*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1
Injection Date : 6/3/2014 3:35:17 PM
Location : Vial 1
Inj Volume : No inj
Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 6/3/2014 3:30:59 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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

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

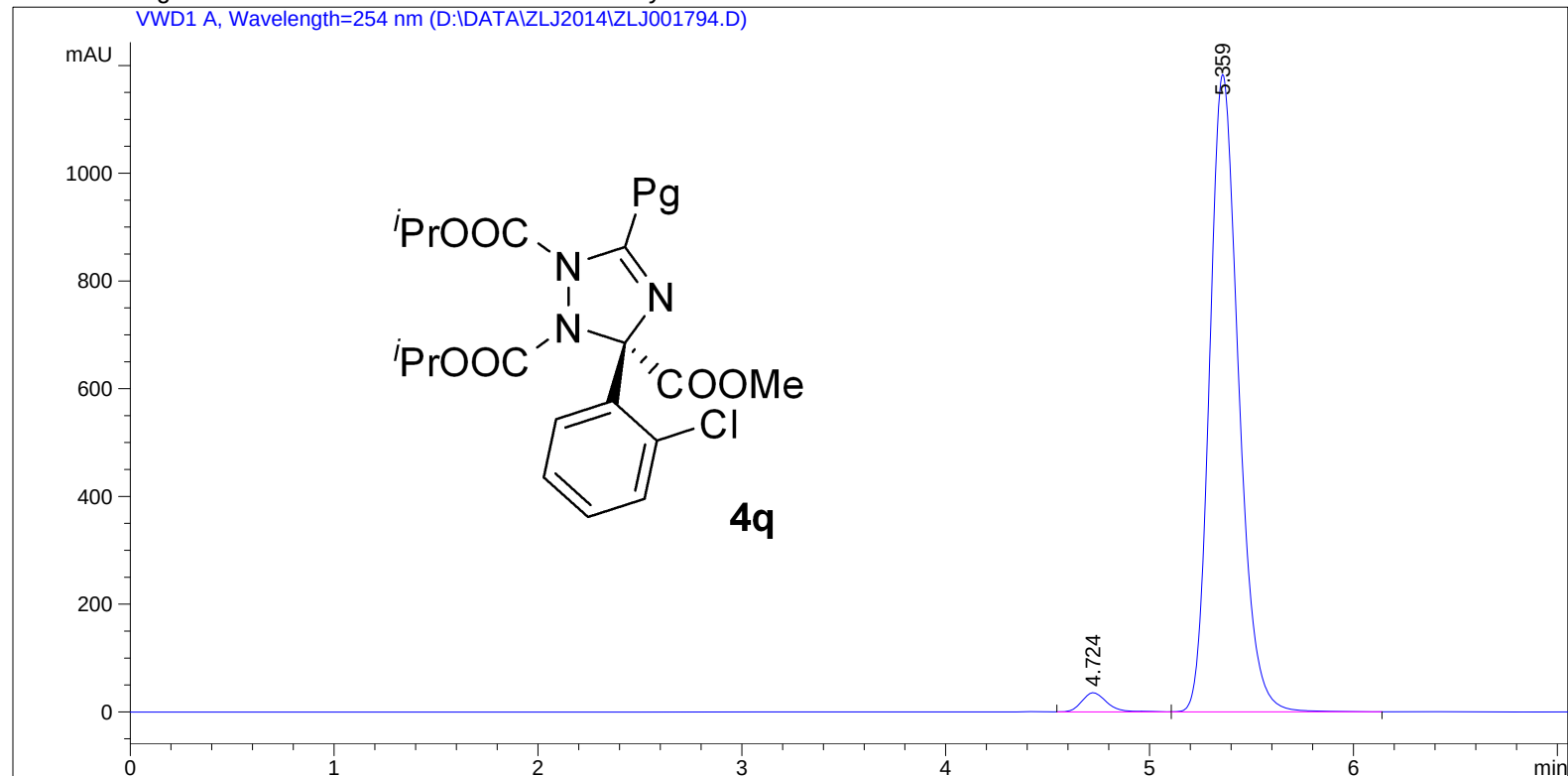
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.720	VV	0.1399	4903.77100	537.83289	49.9735
2	5.367	VB	0.1584	4908.96680	480.83133	50.0265

Totals : 9812.73779 1018.66422

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*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 6/3/2014 3:49:02 PM
Inj Volume : No inj
Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 6/3/2014 3:43:35 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF



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

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

Signal 1: VWD1 A, Wavelength=254 nm

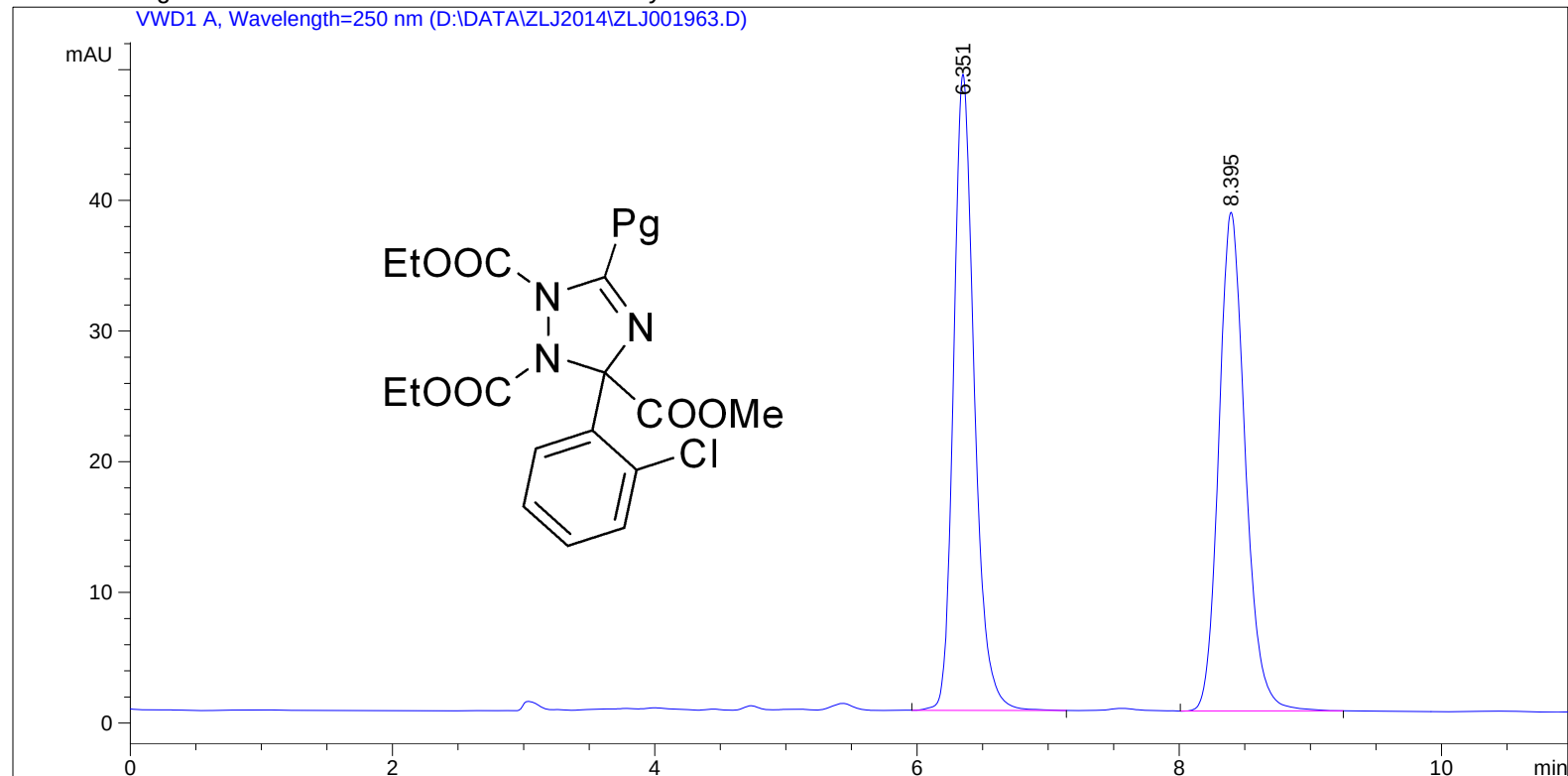
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.724	VV	0.1308	306.16940	35.65111	2.5589
2	5.359	VB	0.1513	1.16585e4	1183.51917	97.4411

Totals : 1.19647e4 1219.17028

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

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 8:07:00 PM Inj Volume : No inj

Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 8:06:42 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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Area Percent Report
=====

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

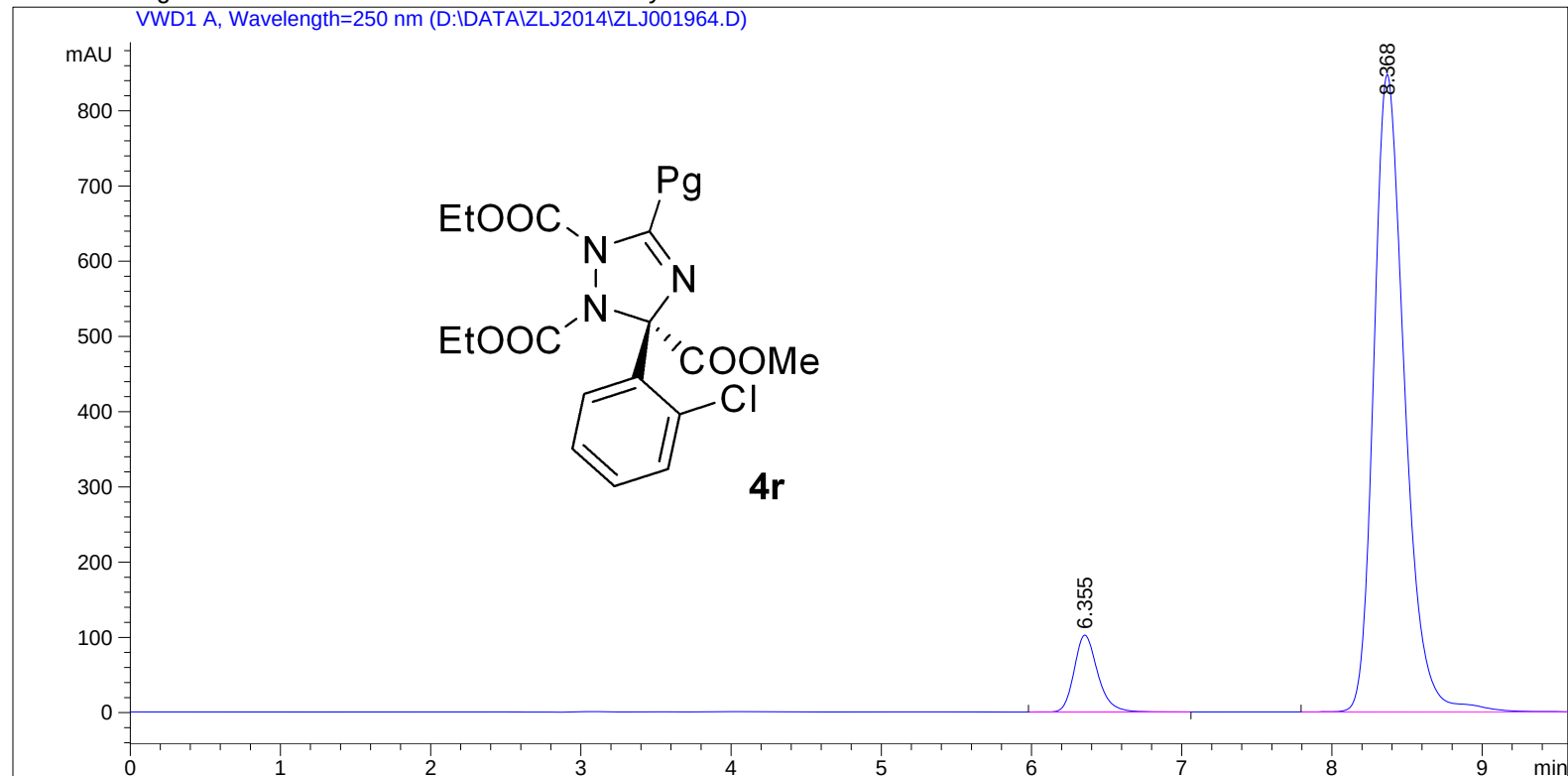
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.351	BB	0.1675	535.24805	48.70113	49.9308
2	8.395	BB	0.2165	536.73218	38.17142	50.0692

Totals : 1071.98022 86.87255

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*** End of Report ***
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=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 8:18:15 PM Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 8:18:03 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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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: VWD1 A, Wavelength=250 nm

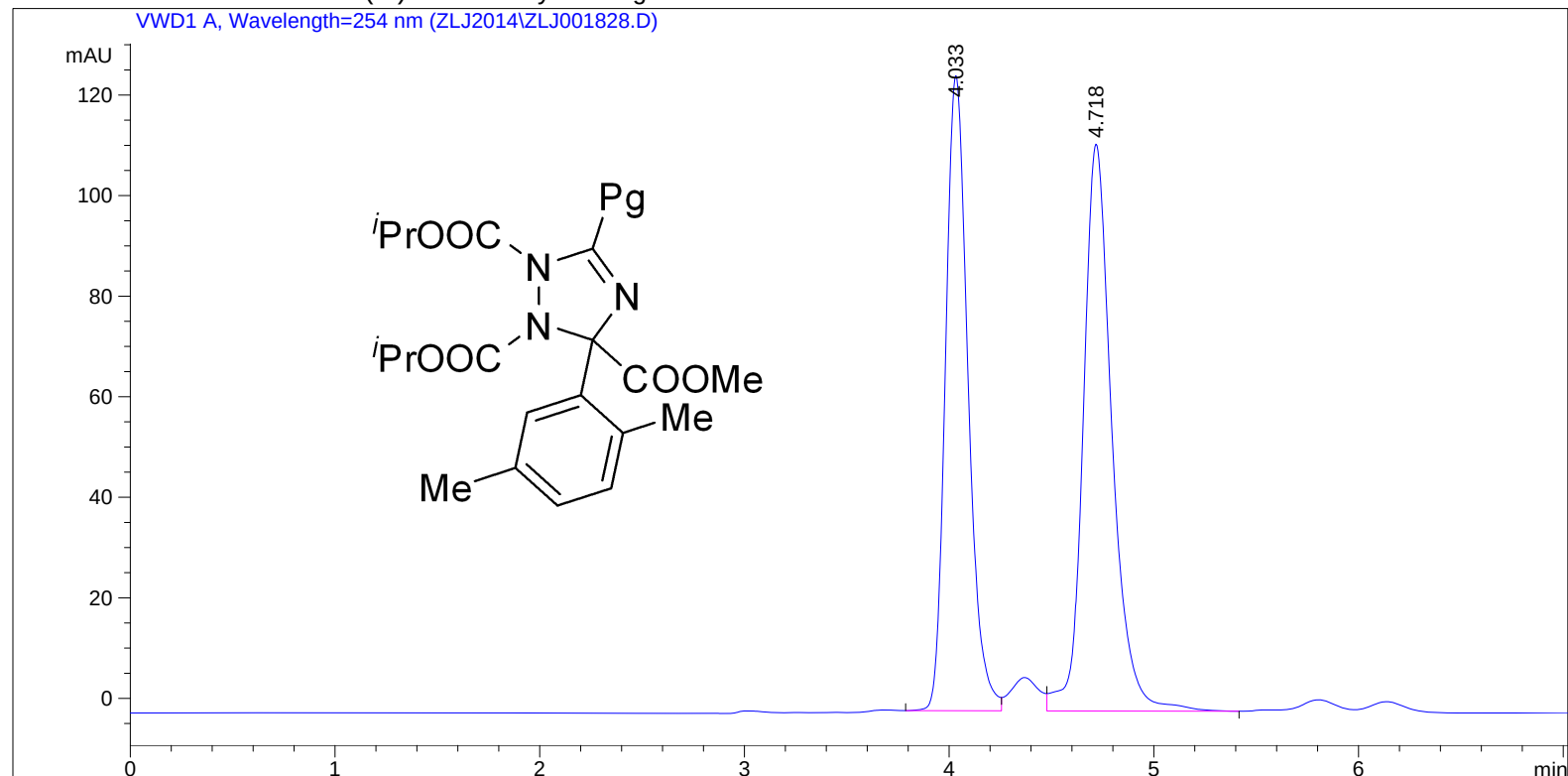
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	6.355	BB	0.1661	1110.12952	102.19118	8.4013
2	8.368	BBA	0.2190	1.21037e4	847.46783	91.5987

Totals : 1.32138e4 949.65902

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*** End of Report ***
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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 6/16/2014 5:04:54 PM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 6/16/2014 5:02:26 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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: VWD1 A, Wavelength=254 nm

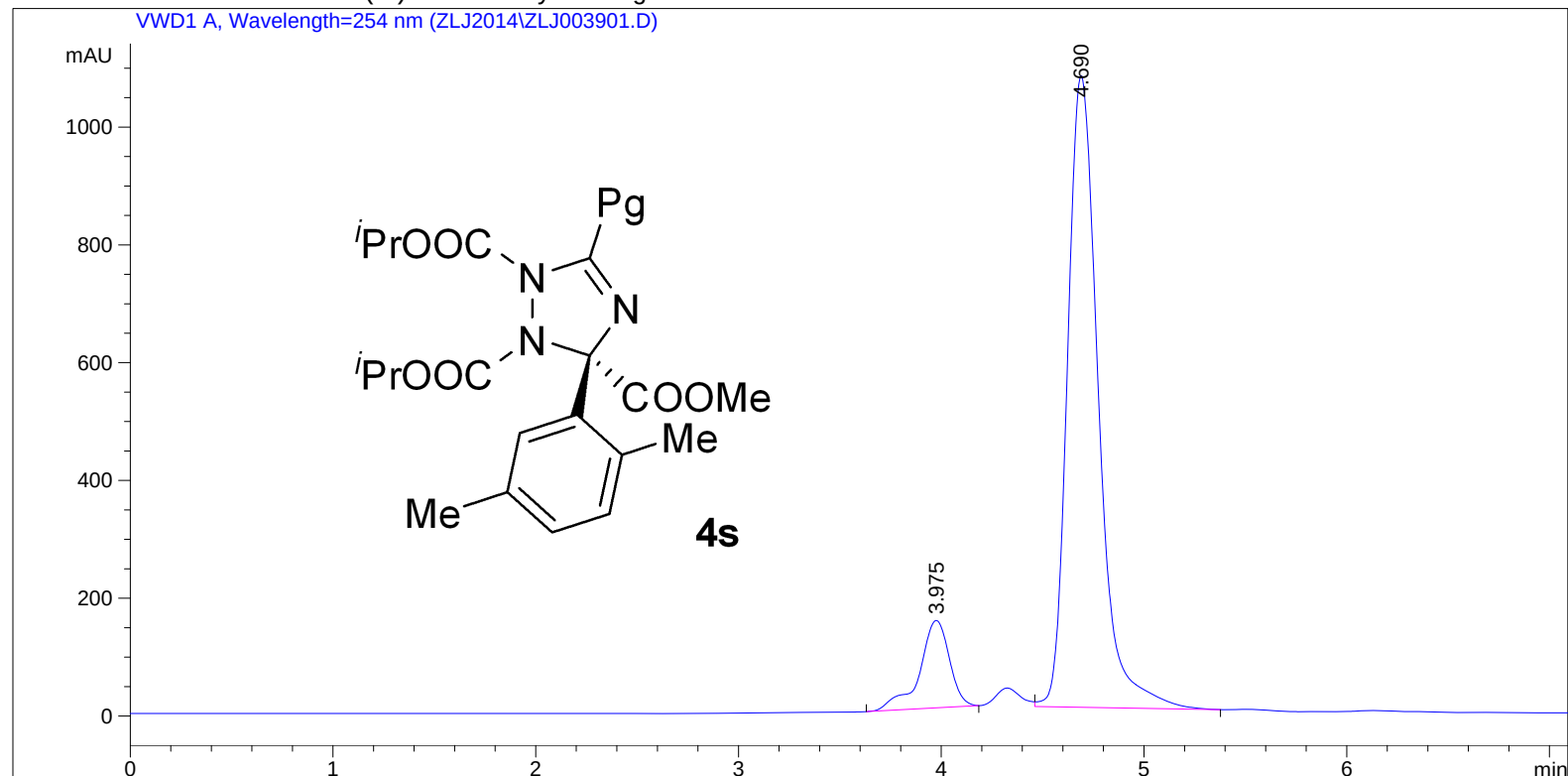
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	4.033	BV	0.1186	968.36328	126.30608	46.7133
2	4.718	VB	0.1478	1104.62927	112.76656	53.2867

Totals : 2072.99255 239.07263

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*** End of Report ***

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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 3/10/2017 2:38:11 PM Inj Volume : No inj

Acq. Method : D:\METHOD\OD_20_1.M
Last changed : 3/10/2017 2:23:02 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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

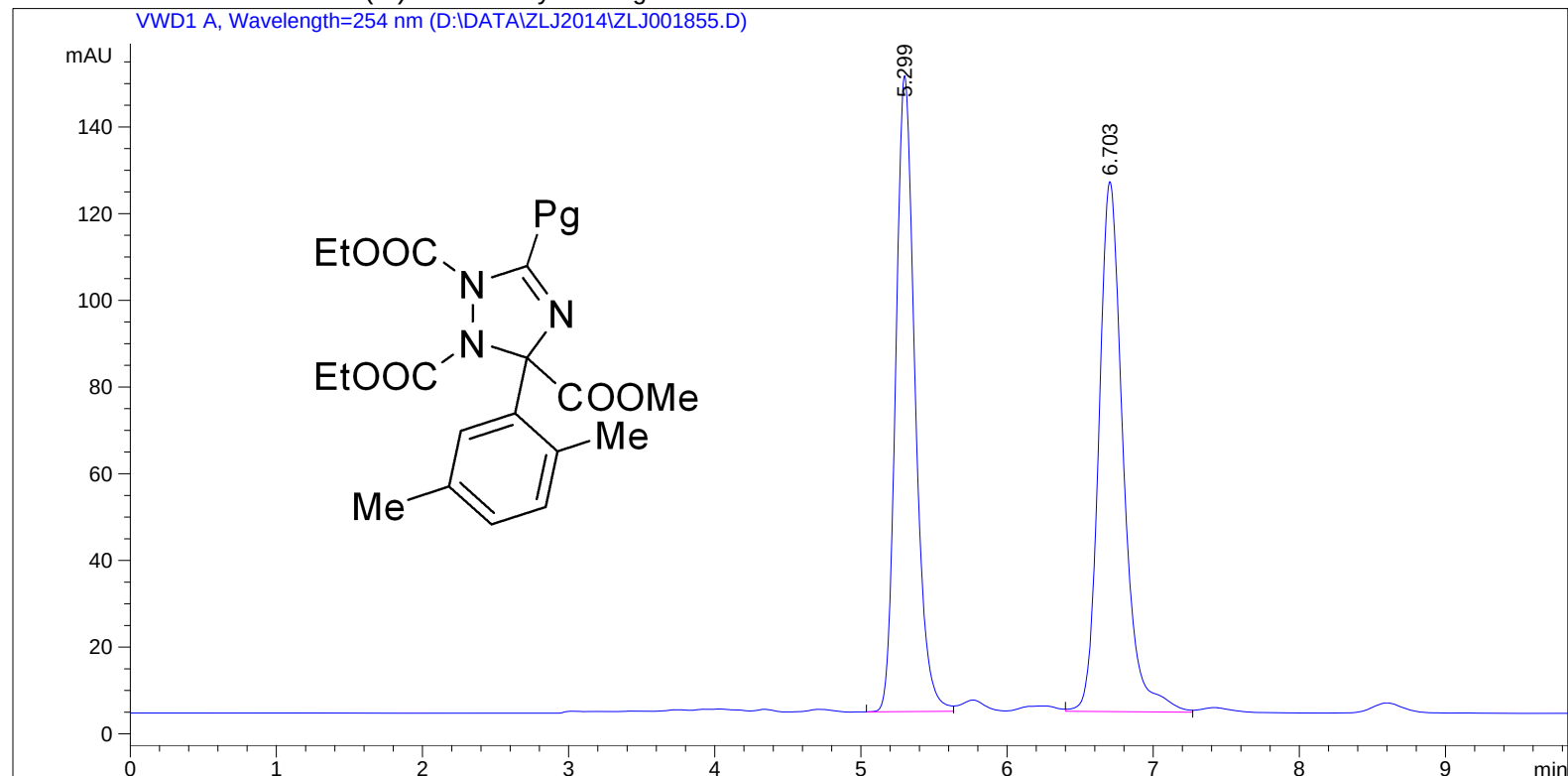
Signal 1: VWD1 A, Wavelength=254 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	3.975	BB	0.1561	1522.31824	148.42172	11.8427
2	4.690	VB	0.1640	1.13322e4	1072.77380	88.1573

Totals : 1.28545e4 1221.19553

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*** End of Report ***

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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 7/8/2014 10:50:12 AM
Inj Volume : No inj
Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 7/8/2014 10:48:20 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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: VWD1 A, Wavelength=254 nm

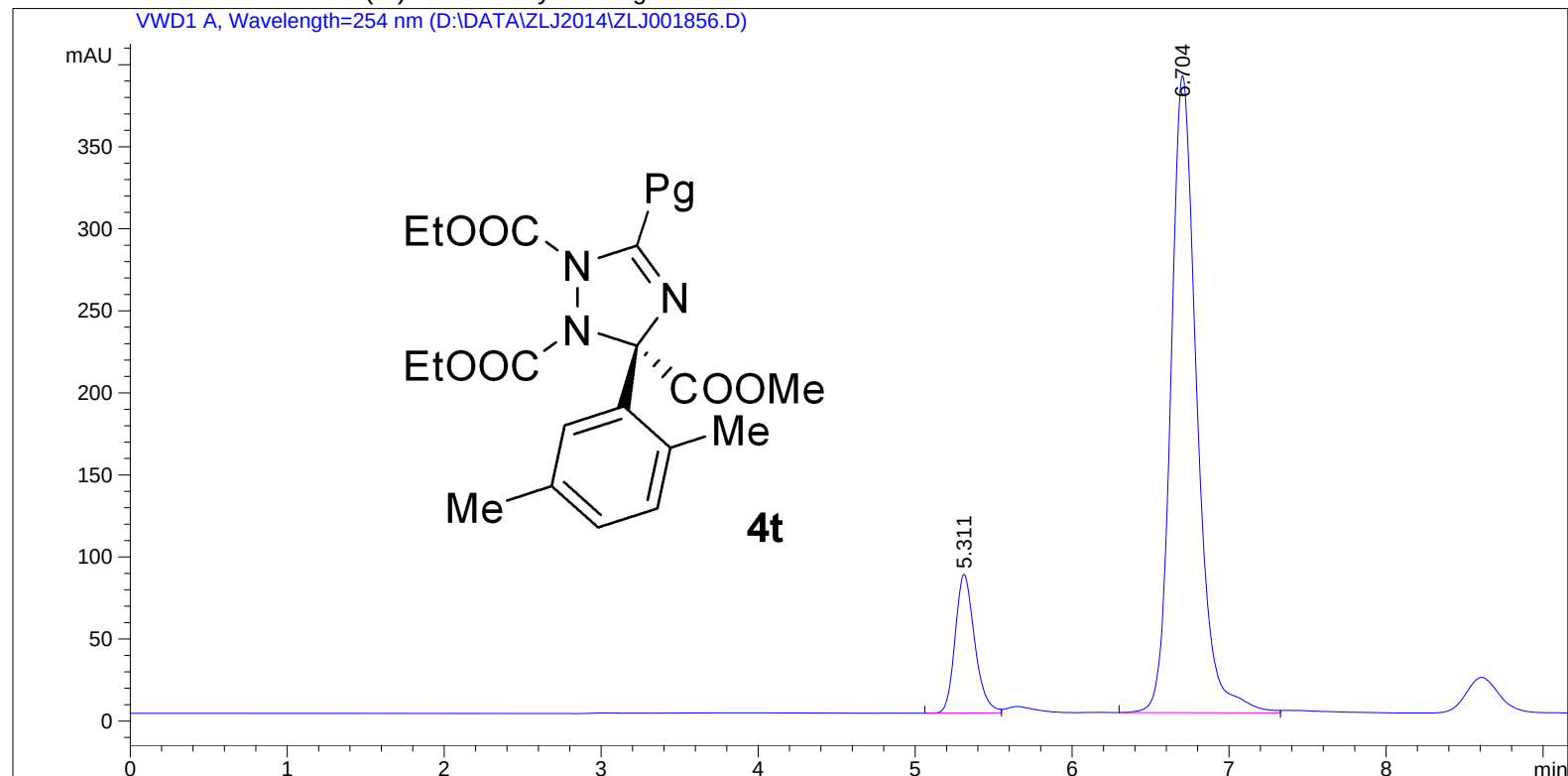
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.299	BV	0.1404	1343.94873	146.71457	48.6928
2	6.703	VV	0.1759	1416.11023	122.28600	51.3072

Totals : 2760.05896 269.00056

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*** End of Report ***

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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 7/8/2014 11:01:40 AM Inj Volume : No inj

Acq. Method : D:\METHOD\IA_20_1.M
Last changed : 7/8/2014 11:00:07 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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: VWD1 A, Wavelength=254 nm

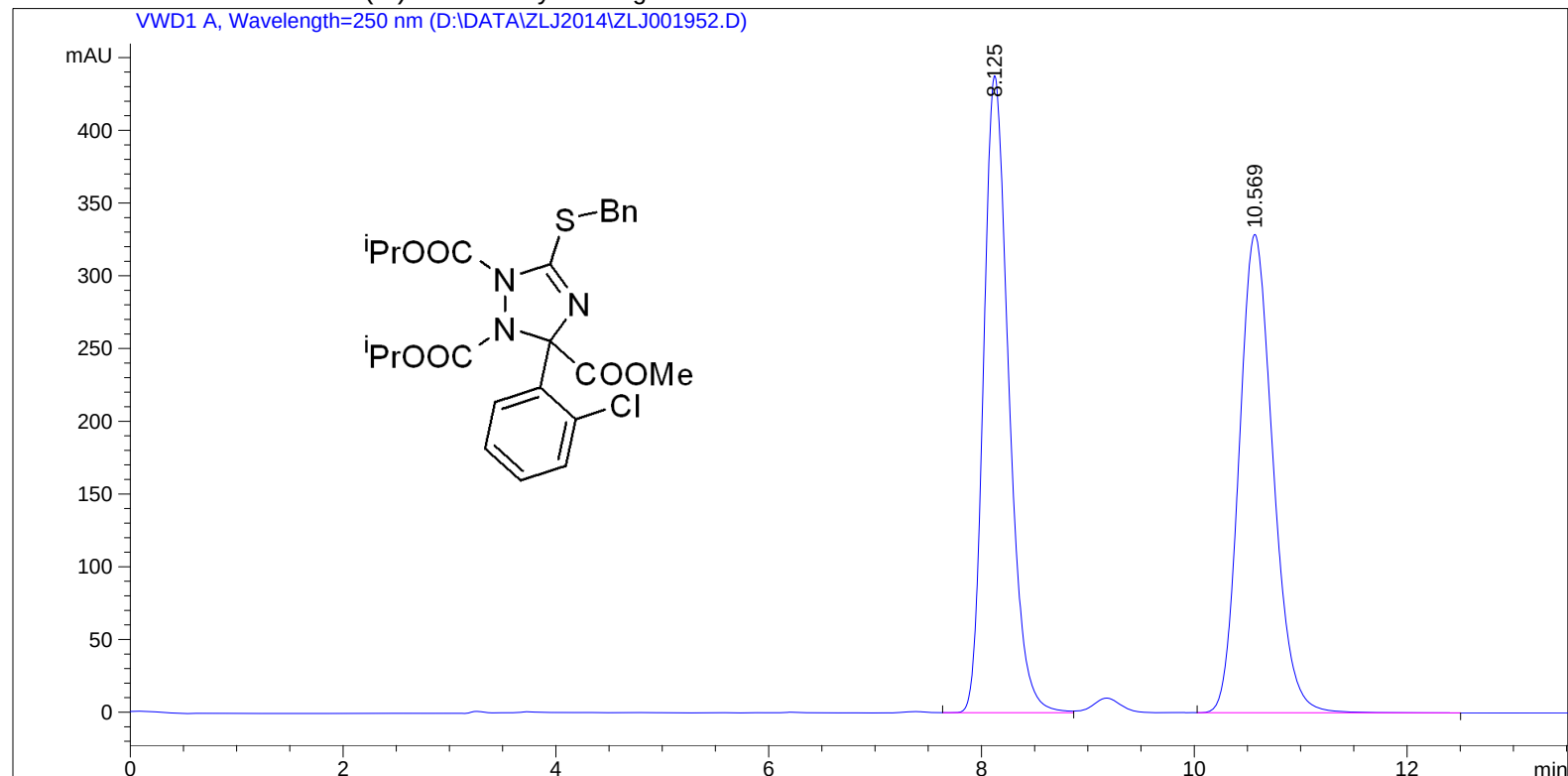
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	5.311	BV	0.1330	731.33856	84.51580	14.0974
2	6.704	BV	0.1747	4456.39844	388.16531	85.9026

Totals : 5187.73700 472.68111

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*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 3:53:54 PM Inj Volume : No inj

Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 3:53:32 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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

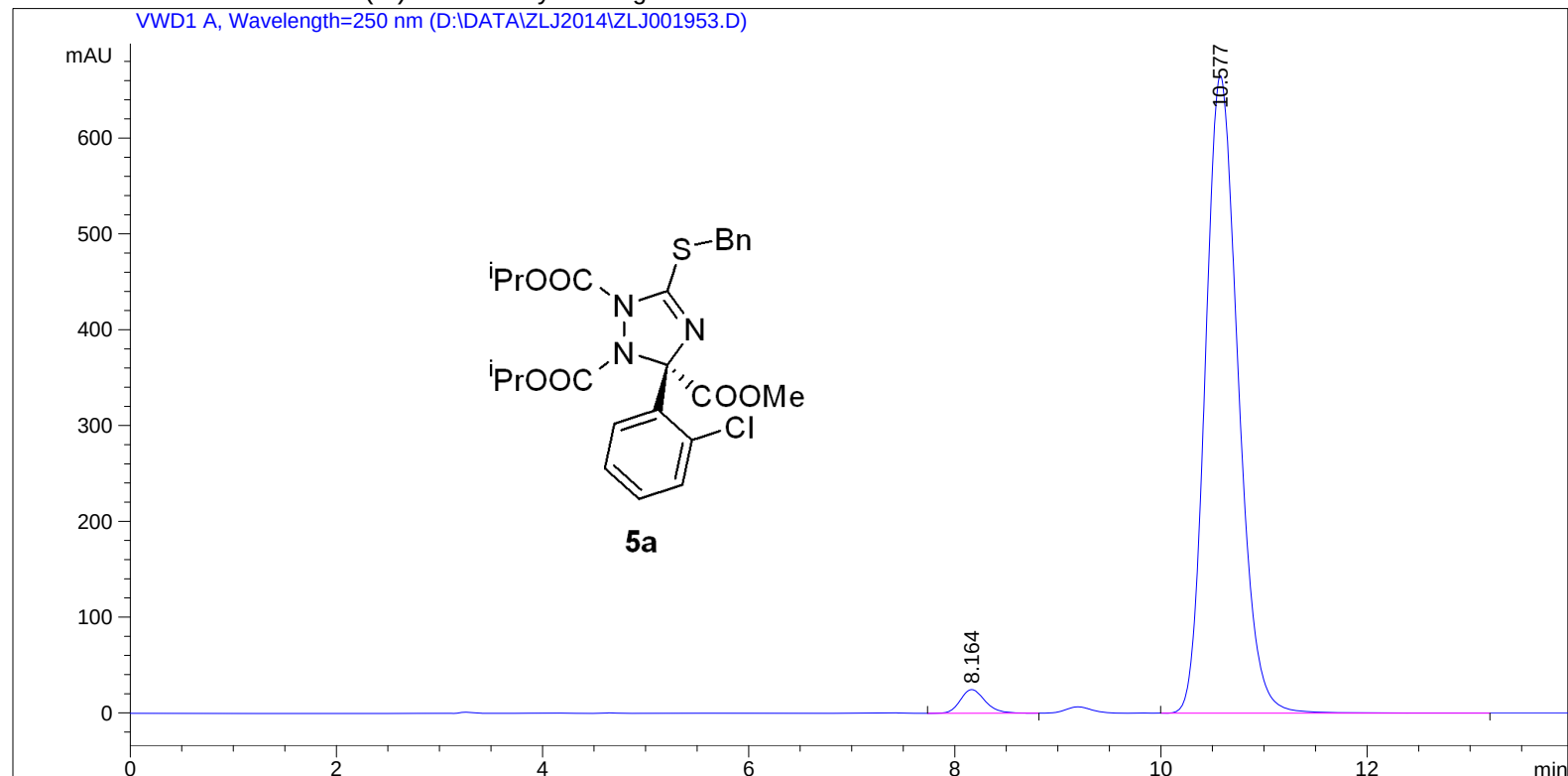
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.125	BV	0.2523	7172.14502	437.82642	49.8008
2	10.569	BB	0.3410	7229.51807	328.78741	50.1992

Totals : 1.44017e4 766.61383

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*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 4:09:49 PM
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 4:07:30 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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

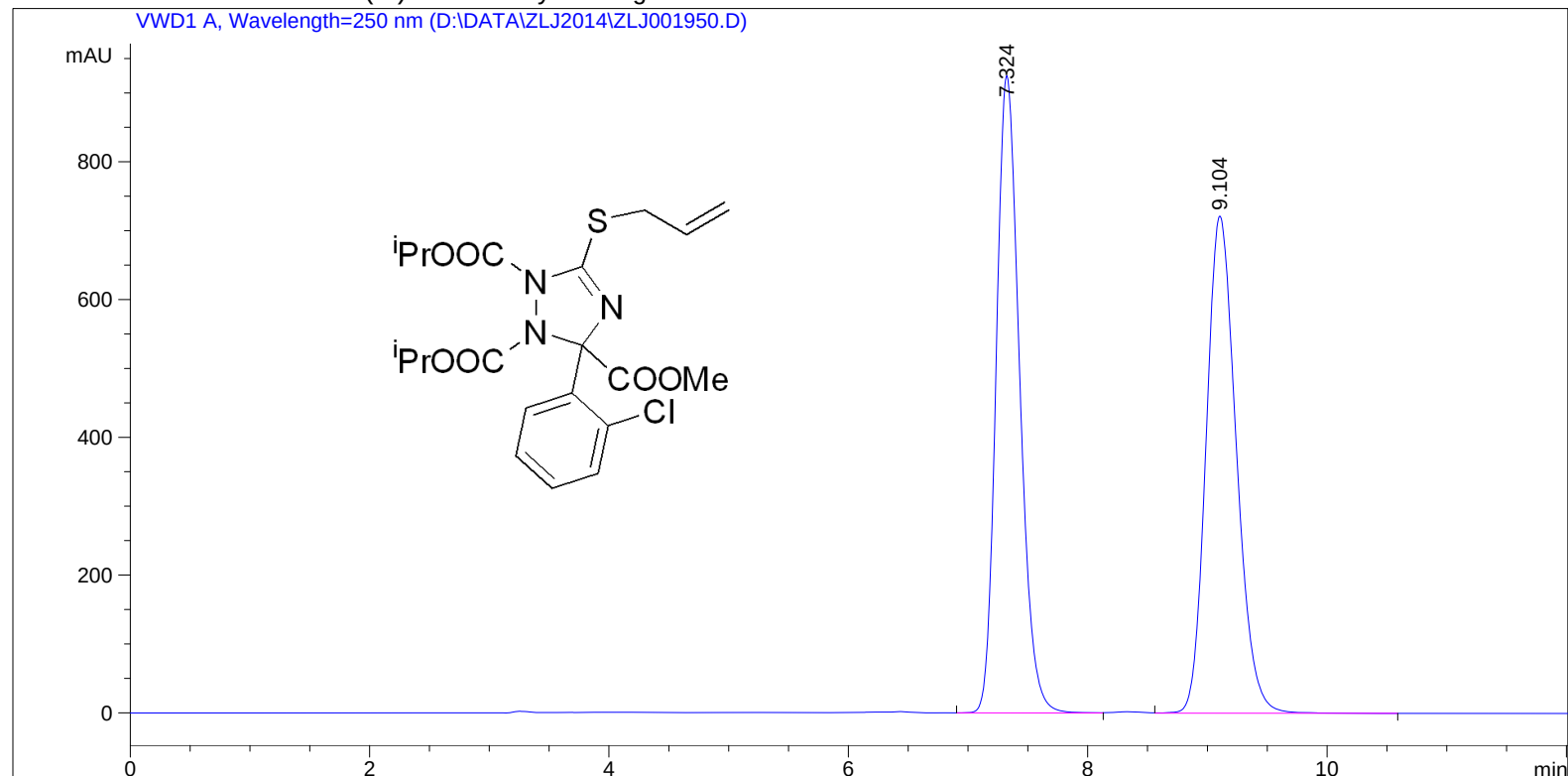
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.164	BB	0.2564	410.91287	24.74135	2.7250
2	10.577	BB	0.3417	1.46686e4	665.36407	97.2750

Totals : 1.50796e4 690.10542

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*** End of Report ***

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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 3:27:42 PM
Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 11:47:39 AM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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

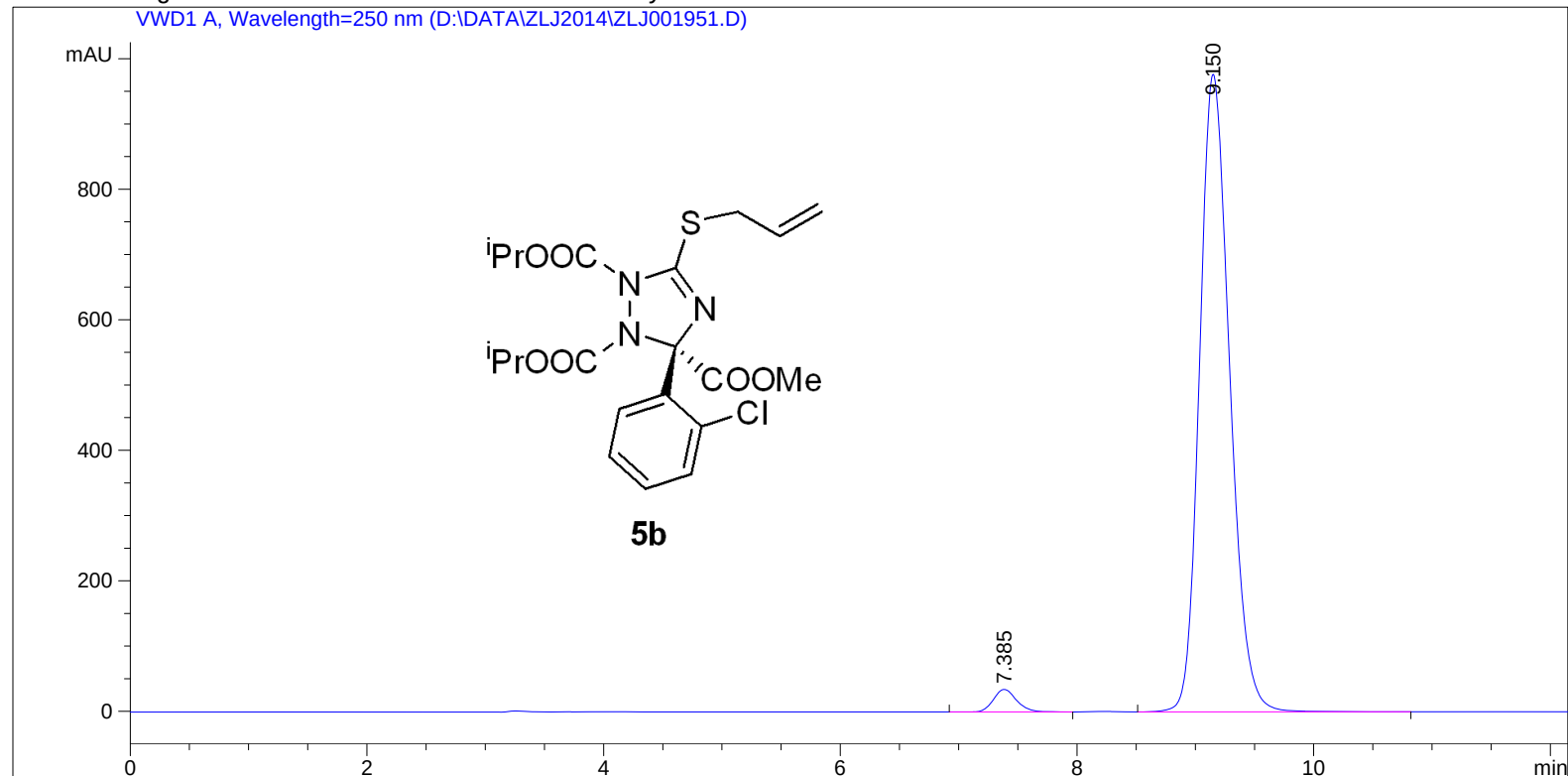
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.324	BV	0.2107	1.25543e4	925.12500	49.8701
2	9.104	VB	0.2710	1.26197e4	721.72253	50.1299

Totals : 2.51741e4 1646.84753

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*** End of Report ***

=====
Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 3:41:18 PM Inj Volume : No inj
Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 3:39:48 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
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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: VWD1 A, Wavelength=250 nm

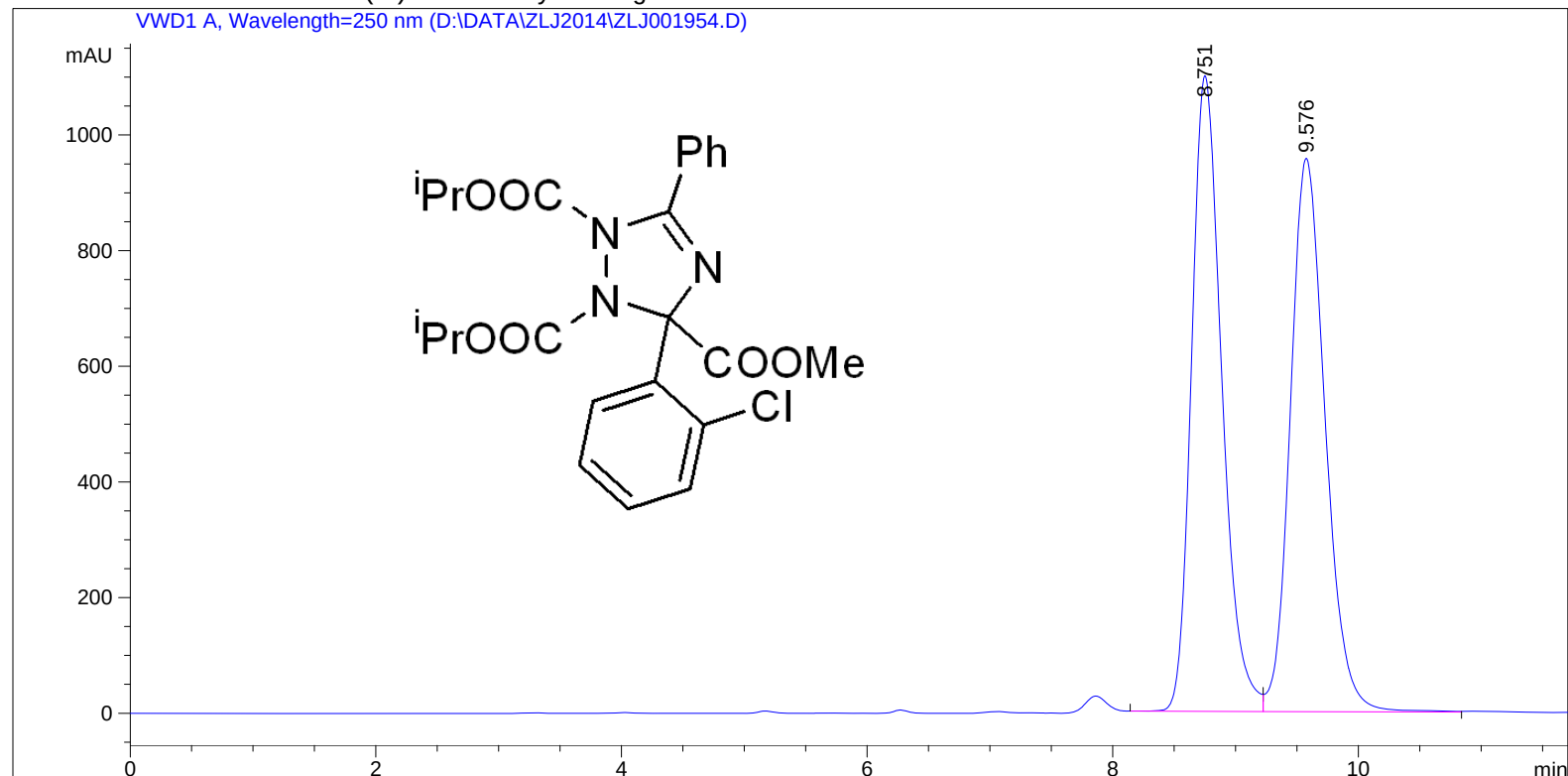
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.385	BB	0.2092	469.25818	34.58874	2.6472
2	9.150	BB	0.2745	1.72574e4	976.91528	97.3528

Totals : 1.77266e4 1011.50402

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*** End of Report ***
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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 4:25:54 PM Inj Volume : No inj

Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 4:23:50 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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Area Percent Report
=====

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

Signal 1: VWD1 A, Wavelength=250 nm

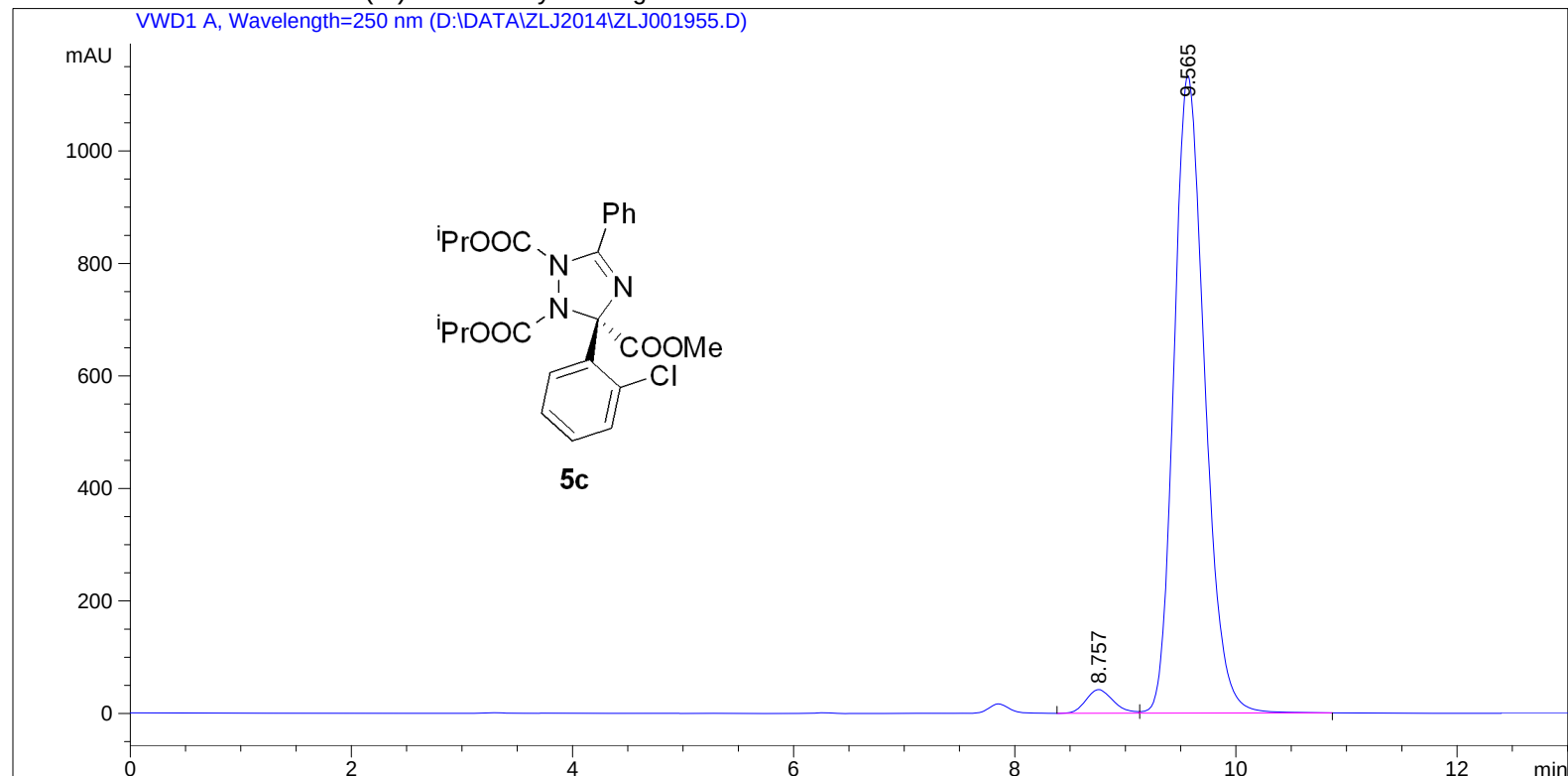
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.751	BV	0.2639	1.88275e4	1099.14551	49.8797
2	9.576	VV	0.3049	1.89183e4	956.87341	50.1203

Totals : 3.77458e4 2056.01892

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*** End of Report ***

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Acq. Operator : LNF
Acq. Instrument : Instrument 1 Location : Vial 1
Injection Date : 8/31/2014 4:37:58 PM Inj Volume : No inj

Acq. Method : C:\CHEM32\1\METHODS\DEF_LC.M
Last changed : 8/31/2014 4:37:43 PM by LNF
(modified after loading)
Analysis Method : D:\METHOD\OD_20_1.M
Last changed : 3/25/2013 8:45:53 AM by LNF
Additional Info : Peak(s) manually integrated



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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: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.757	BV	0.2655	721.92944	42.12500	3.1924
2	9.565	VB	0.2982	2.18924e4	1133.08325	96.8076

Totals : 2.26143e4 1175.20825

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*** End of Report ***

