Electronic Supplementary Information

Enantioselective Synthesis of Chiral α-Alkynylated Thiazolidones by Tandem *S*-Addition/Acetalization of Alkynyl Imines

Mei-Xin Wang,[†a] Juan Liu,[†a,b] Zhen Liu,[a] Yingcheng Wang,[a] Qi-Qiong Yang,[a] Wen-Yu Shan,[a] Yu-Hua Deng*[a] and Zhihui Shao*[a]

[†] These two authors contributed equally to this work.

E-mail: dengyuhua@ynu.edu.cn; zhihui_shao@hotmail.com

[a] Key Laboratory of Medicinal Chemistry for Natural Resource, Ministry of Education, School of Chemical Science and Technology, Yunnan University, Kunming, China, 650091

[b] Yunnan Baiyao Group CO., Ltd, Kunming, China, 650500.

Table of Contents

1. General Information

Unless otherwise specified, all reactions were carried out under argon atmosphere in anhydrous conditions. All the solvents were purified according to the standard procedures. All chemicals which are commercially available were used without further purification unless otherwise noted. All the chiral phosphoric acids were directly used for the reaction, after they are bought from the *Daicel Chiral Technologies (China) Co., LTD.* 1,4-dithiane-2,5-diol **2** (CAS: 40018-26-6) and 1,4-dioxane-2,5-diol **2′** (CAS: 23147-58-2) bought from the *Energy-Chemical* were directly used for the reaction. Thin-layer chromatography (TLC) was performed on silica gel plates (60F-254) using UV-light (254 and 365nm).

^{1}H-NMR and ^{13}C-NMR spectra were recorded at 400 MHz or 600 MHz spectrophotometer. Chemical shifts (δ) are expressed in ppm, and J values are given in Hz. NMR multiplicities are abbreviated as follows: s = singlet, br = broad signal, d = doublet, t = triplet, q = quartet, m = multiple, dd = doublet of doublet. Values of enantiomeric excess was determined by chiral HPLC (Agilent 1260 Infinity) with *n*-hexane and *i*-propanol as eluents. High resolution mass spectrometry (HRMS) was were recorded on an ESI-ion trap Mass spectrometer (Agilent 1100 series LC/MSD, SL model). Optical rotations were measured on a Jasco P-2000 polarimeter. All chemicals and solvents were used as received without further purification unless otherwise stated. Column chromatography was performed on silica gel (200–300 mesh).

2. Substrates of Alkynyl Imines

According to the known procedures, *N*-Boc-protected *C*-alkynyl *N,O*-acetals **1a-1k** and *N*-Cbz-protected *C*-alkynyl *N,O*-acetals **1l-1t** were synthesized (Figure S1).[1]

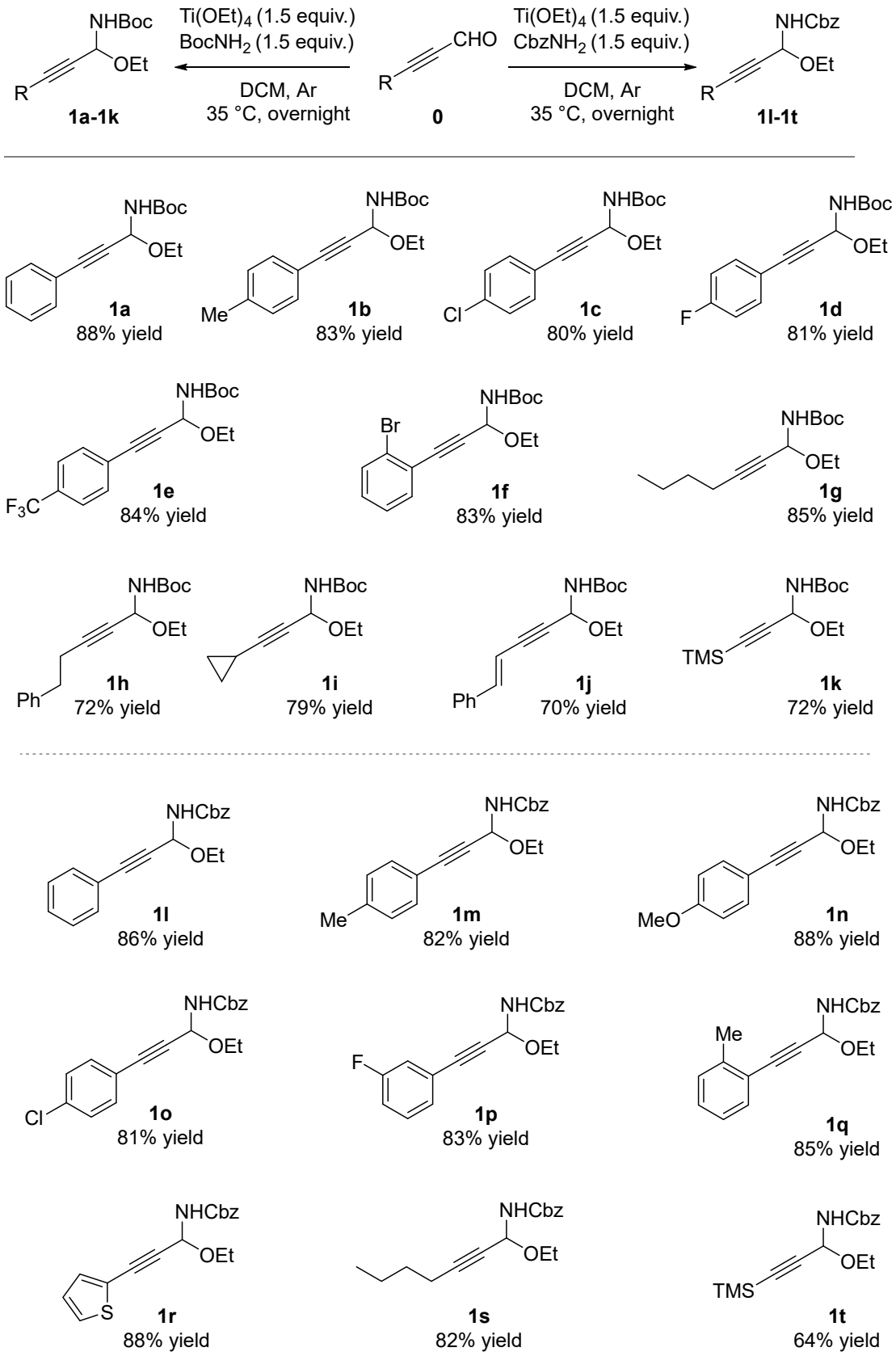
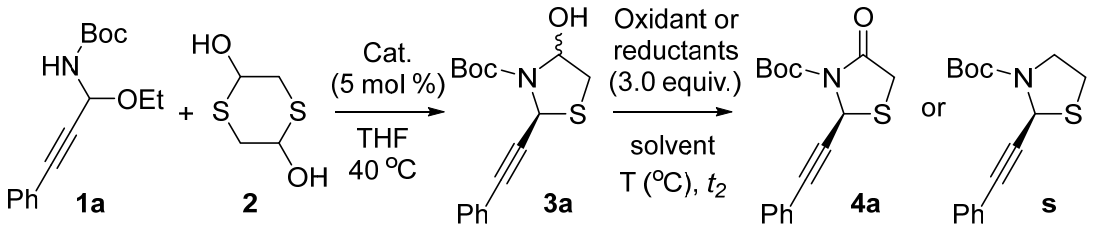
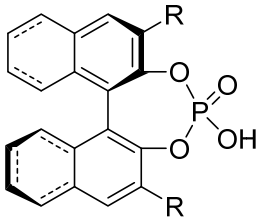


Figure S1. All Precursors of Alkynyl Imines Employed.

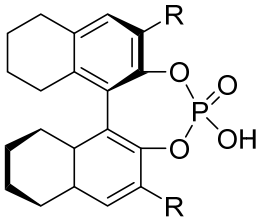
3. Optimization Study

Table S1. Preliminary Study & Catalysts Evaluation and Screening [a]

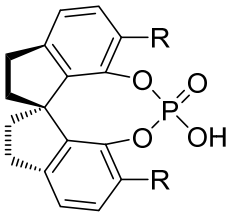




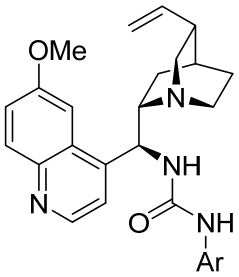
A1: R = Ph
A2: R = 9-anthryl
A3: R = 2,4,6-(^{i}Pr)$_3$-C$_6$H$_2$
A4: R = 2,4,6-(Me)$_3$-C$_6$H$_2$
A5: R = 3,5-(CF$_3$)$_2$-C$_6$H$_3$
A6: R = 3,5-(Me)$_2$-C$_6$H$_3$
A7: R = 4-^{t}Bu-C$_6$H$_4$
A9 R = 4-Ph-C$_6$H$_4$
A10: R = 3,5-(Ph)$_2$-C$_6$H$_3$
A11: R = 4-Cl-C$_6$H$_4$
A12: R = 4-CF$_3$-C$_6$H$_4$
A13: R = 4-NO$_2$-C$_6$H$_4$
A14: R = 4-OMe-C$_6$H$_4$



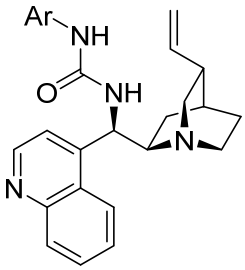
A8: R = *H8-2*,4,6-(Me)$_3$-C$_6$H$_2$



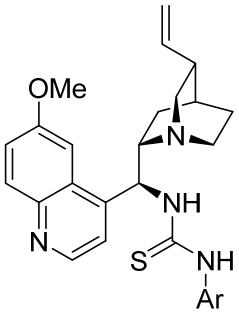
B1: R = 2,4,6-(^{i}Pr)$_3$C$_6$H$_2$
B2: R = 2,4,6-(Me)$_3$C$_6$H$_2$
B3: R = 9-phenanthryl
B4: R = SiPh$_3$



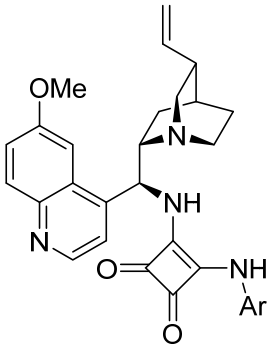
C1: Ar = 3,5-(CF$_3$)$_2$-C$_6$H$_3$



C2: Ar = 3,5-(CF$_3$)$_2$-C$_6$H$_3$



D1: Ar = 3,5-(CF$_3$)$_2$-C$_6$H$_3$



E1: Ar = 3,5-(CF$_3$)$_2$-C$_6$H$_3$
E2: Ar = 3,5-(CF$_3$)$_2$-C$_6$H$_3$CH$_2$

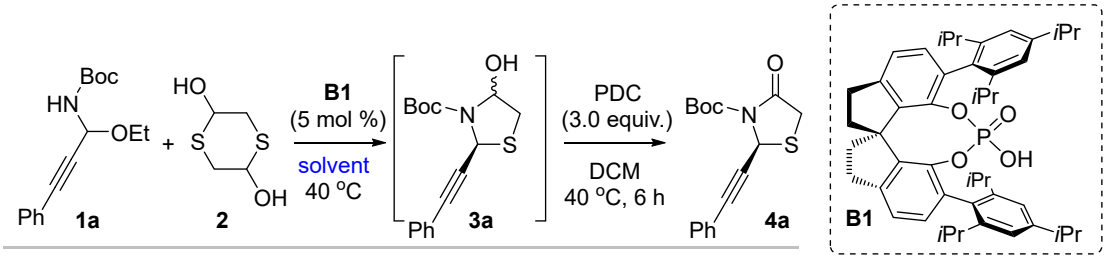
Entry	Cat.	Oxidant or Reductants	Solvent	T (°C)	t_2 (h)	yield (%)	ee (%)
1[b]	**A1**	-	-	-	-	41	0 (0)
2	**A1**	PCC	DCM	40	6	29	0

3	**A1**	PDC	DCM	40	6	32	0
4	**A1**	IBX	DCM	40	6	25	0
5	**A1**	PDC	DCM	RT	12	21	0
6	**A1**	$LiAlH_4$	THF	0	12	complex	-
7	**A1**	TFA/Et_3SiH	DCM	0	12	complex	-
8	**A1**	$BF_3.Et_2O/Et_3SiH$	DCM	0	12	complex	-
9	**A1**	$BF_3.Et_2O/Et_3SiH$	DCM	rt	12	complex	-
10	**A2**	PDC	DCM	40	6	50	13
11	**A3**	PDC	DCM	40	6	52	50
12	**A4**	PDC	DCM	40	6	47	32
13	**A5**	PDC	DCM	40	6	51	11
14	**A6**	PDC	DCM	40	6	46	15
15	**A7**	PDC	DCM	40	6	54	32
16	**A8**	PDC	DCM	40	6	53	11
17	**A9**	PDC	DCM	40	6	50	12
18	**A10**	PDC	DCM	40	6	34	9
19	**A11**	PDC	DCM	40	6	49	12
20	**A12**	PDC	DCM	40	6	46	11
21	**A13**	PDC	DCM	40	6	55	0
22	**A14**	PDC	DCM	40	6	34	27
23	**B1**	PDC	DCM	40	6	55	56
24	**B2**	PDC	DCM	40	6	49	50
25	**B3**	PDC	DCM	40	6	44	36
26	**B4**	PDC	DCM	40	6	30	0
27	**C1**	PDC	DCM	40	6	N.D.	-
28	**C2**	PDC	DCM	40	6	N.D.	-
29	**D1**	PDC	DCM	40	6	N.D.	-
30	**E1**	PDC	DCM	40	6	N.D.	-
31	**E2**	PDC	DCM	40	6	N.D.	-

[a] Unless otherwise noted, the reaction was performed with **1a** (0.1 mmol), **2** (0.06 mmol),

indicated catalyst (5 mol %) in the THF (1.0 mL) at 40 °C; then oxidant or reductant (3.0 equiv.) in the indicated solvent and temperature. Yields of isolated products **4a** are given. The ee values for **4a** are determined by HPLC. [b] Yield refers to the isolated product **3a**. The ee values are determined by HPLC for **3a**. dr = 1.4:1.

Table S2. Evaluation and Screening of Solvents [a]

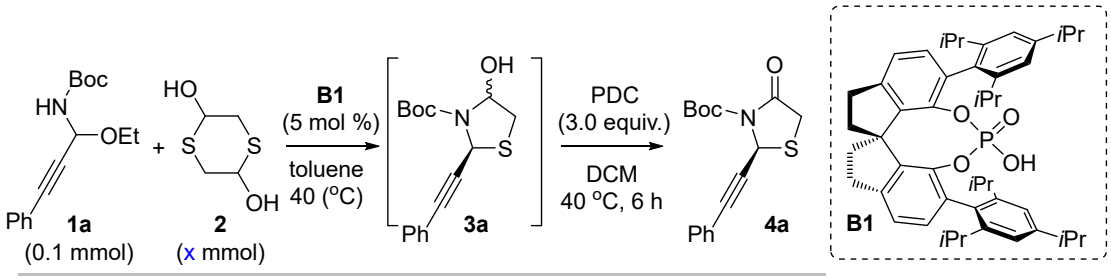


Entry	Solvent	yield of **4a** (%)	ee of **4a** (%)
1	THF	55	56
2	1,4-dioxane	40	0
3	PhOMe	52	80
4	toluene	61	90
5	xylene	56	89
6	EtC_6H_5	50	86
7	ClC_6H_5	56	84
8	BrC_6H_5	59	88
9	$CF_3C_6H_5$	57	85
10	DCE	56	72
11	DCM	42	77
12	$CHCl_3$	58	85
13	CCl_4	54	90
14	EtOAc	60	89
15	DMSO	32	0
16	DMF	39	14

[a] Unless otherwise noted, the reaction was performed with **1a** (0.1 mmol), **2** (0.06 mmol), **B1**

(5 mol %) in the indicated solvent (1.0 mL) at 40 °C; then PDC (3.0 equiv.) in the DCM (1.0 mL) at 40 °C. Yields of isolated products **4a** are given. The ee values for **4a** are determined by HPLC.

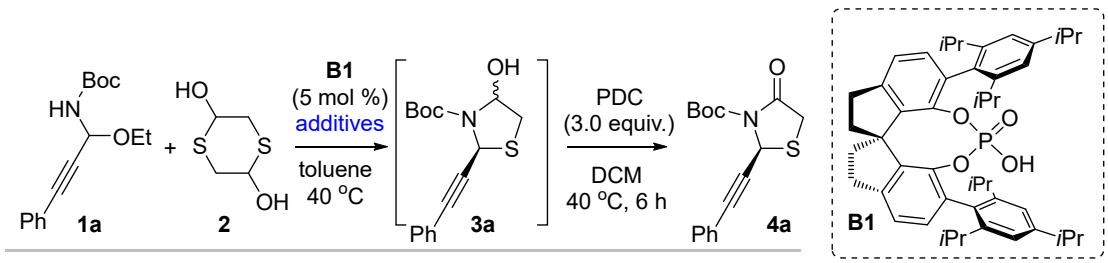
Table S3. Evaluation and Screening of the Loading of Substrates[a]



Entry	**2** (x mmol)	yield of **4a** (%)	ee of **4a** (%)
1	0.05	40	90
2	0.06	61	90
3	0.08	58	90
4	0.1	54	90

[a] Unless otherwise noted, the reaction was performed with **1a** (0.1 mmol), **2** (x mmol), **B1** (5 mol %) in the toluene (1.0 mL) at 40 °C; then PDC (3.0 equiv.) in the DCM (1.0 mL) at 40 °C. Yields of isolated products **4a** are given. The ee values for **4a** are determined by HPLC.

Table S4. Evaluation and Screening of Additives [a]

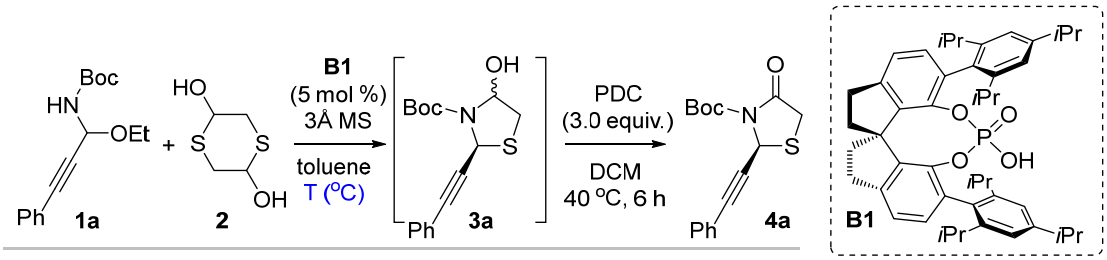


Entry	additives	yield of **4a** (%)	ee of **4a** (%)
1	none	61	90
2	3Å MS	72	90
3	4Å MS	70	89

4	5Å MS	70	89
5	$MgSO_4$	66	84

[a] Unless otherwise noted, the reaction was performed with **1a** (0.1 mmol), **2** (0.06 mmol), **B1** (5 mol %) and indicated additives (30 mg) in the toluene (1.0 mL) at 40 °C; then PDC (3.0 equiv.) in the DCM (1.0 mL) at 40 °C. Yields of isolated products **4a** are given. The ee values for **4a** are determined by HPLC.

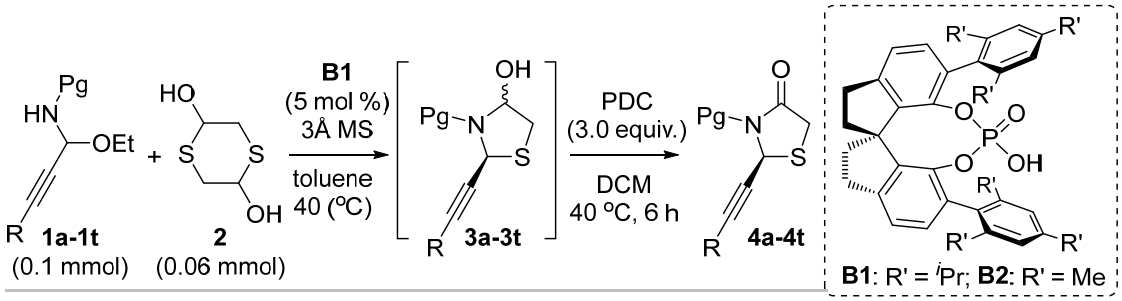
Table S5. Evaluation and Screening of Temperature [a]



Entry	T (°C)	*t* (h)	yield (%)	ee (%)
1	20	24	44	93
2	25	24	46	92
3	30	12	53	90
4	35	8	57	90
5	40	8	55	90
6	50	8	42	89

[a] Unless otherwise noted, the reaction was performed with **1a** (0.1 mmol), **2** (0.06 mmol), **B1** (5 mol %) and 3Å MS (30 mg) in the toluene (1.0 mL) at the indicated temperature; then PDC (3.0 equiv.) in the DCM (1.0 mL) at 40 °C. Yields of isolated products **4a** are given. The ee values for **4a** are determined by HPLC.

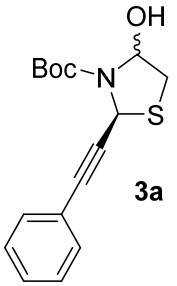
4. General Procedure for the Tandem Asymmetric Annulation



General procedure for annulation: The freshly dry toluene (1 mL) was added to the dry 25-mL tube containing with alkynyl imines **1** (0.1 mmol), the chiral catalyst **B1** (5 mol %), 2,5-dihydroxy-1,4-dithiane **2** (9.2 mg, 0.06 mmol) and 3Å MS (30 mg). The reacting mixture was stirred at 40 °C for 8-12 hours. Upon the completion of alkynyl imines monitored by TLC analysis (*about 1:1 dr*), the mixture was directly purified by flash column chromatography (elution: ethyl acetate/petroleum ether = 1/15) to afford the crude chiral thiazolidines **3**.

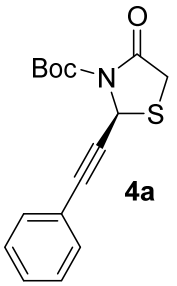
Note: Chiral thiazolidines **3** readily occurred the racemization of *N,O*-acetals motif and some undefined side reaction in the solvent. Most of the chiral thiazolidines **3** could not be purified carefully, and only three chiral thiazolidines have been provided the good quality spectrums or data. In order to facilitate the further analyses, the crude products **3** was directly used for the next oxidation procedure.

General procedure for PDC-mediated oxidation: The solution of the obtained crude products **3** (1.0 equiv.) in DCM (1.0 mL) was added PDC (3.0 equiv.). The mixture was stirred at 40 °C for 6 hours. Then, the mixture was cooled to rt and added an appropriate amount of silica gel, which gave a good solid residue after the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/25 to 1/20) to afford the desired products **4**.



3a

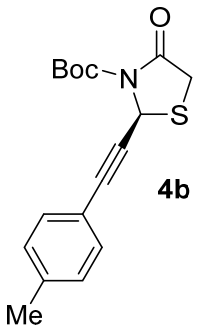
***Tert*-butyl (2*S*)-4-hydroxy-2-(phenylethynyl)thiazolidine-3-carboxylate.** Following the *General Procedure* (without PDC-mediated oxidation procedure), **3a** was obtained as clear colorless oil (8 h, 29.4 mg, 92% yield, 1.4:1 dr, 90% (90%) ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{20}$ = -160.1 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{16}H_{19}NO_3SNa$ $[M + Na]^+$: 328.0978, found: 328.0978; **^{1}H NMR** (400 MHz, acetone-d^6, ppm): δ 7.43-7.36 (m, 7H (5*1.4H) + 5H), 5.97 (d, J = 2.8 Hz, 1H), 5.88 (s, 1H), 5.82 (s, 1.4H), 5.70 (s, 1.4H), 5.17 (d, J = 4.6 Hz, 1H), 4.98 (d, J = 5.3 Hz, 1.4H), 3.58-3.53 (m, 1.4H), 3.43-3.39 (m, 1H), 3.23 (s, 1H), 3.04 (d, J = 12.1 Hz, 1.4H), 1.50 (s, 13H(9*1.4H) + 9H); **^{13}C NMR** (100 MHz, acetone-d^6, ppm): δ 152.1, 152.0, 131.44, 131.41, 128.52, 128.48, 122.8, 122.7, 88.5, 83.8, 82.6, 82.2, 81.9, 80.7, 80.5, 51.4, 50.5, 38.1, 27.6; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 90:10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (minor isomer) = 8.02 min, t_R (major isomer) = 9.05 min; t_R (minor isomer) = 13.08 min, t_R (major isomer) = 14.78 min.



4a

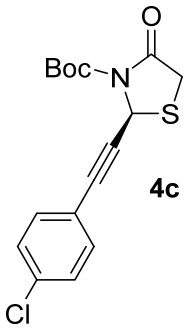
***Tert*-butyl (*S*)-4-oxo-2-(phenylethynyl)thiazolidine-3-carboxylate.** Following the *General Procedure*, **4a** was obtained as clear colorless oil (8 h, 23.5 mg, 72% yield of 2-steps, 90% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -5.3 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{16}H_{17}NO_3SNa$ $[M + Na]^+$: 326.0821, found: 326.0819; **^{1}H NMR** (400 MHz, $CDCl_3$,

ppm): δ 7.44-7.40 (m, 2H), 7.37-7.32 (m, 3H), 5.91 (s, 1H), 4.03 (d, J = 16.0 Hz, 1H), 3.61 (d, J = 16.0 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.6, 148.3, 131.7, 129.1, 128.4, 121.6, 85.4, 85.3, 84.7, 49.9, 33.6, 28.0; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.45 min, t_R (minor) = 14.89 min.



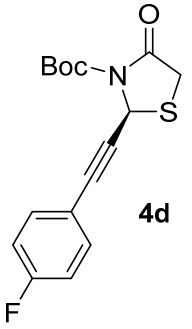
4b

***Tert*-butyl (*S*)-4-oxo-2-(*p*-tolylethynyl)thiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3b** was obtained as clear yellow oil (8 h, 25.5 mg, 80% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4b** was obtained as clear yellow oil (8 h, 22.3 mg, 69% yield of 2-steps, 93% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -4.3 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{17}H_{19}NO_3SNa$ $[M + Na]^+$: 340.0978, found: 340.0977; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 7.32-7.30 (m, 2H), 7.14-7.13 (m, 2H), 5.90 (s, 1H), 4.02 (d, J = 16.2 Hz, 1H), 3.60 (d, J = 16.2 Hz, 1H), 2.36 (s, 3H), 1.56 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 169.6, 148.3, 139.3, 131.6, 129.2, 118.6, 85.6, 84.73, 84.65, 50.0, 33.6, 28.0, 21.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.76 min, t_R (minor) = 14.93 min.



4c

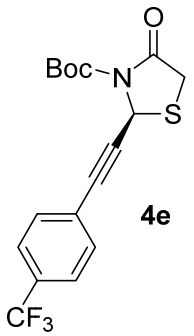
***Tert*-butyl (*S*)-2-((4-chlorophenyl)ethynyl)-4-oxothiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3c** was obtained as clear yellow oil (8 h, 31.5 mg, 93% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4c** was obtained as clear yellow oil (10 h, 23.0 mg, 68% yield of 2-steps, 91% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -11.6 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{16}H_{16}ClNO_3SNa$ $[M + Na]^+$: 360.0432, found: 360.0434; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 7.36-7.30 (m, 4H), 5.89 (s, 1H), 4.01 (d, *J* = 16.2 Hz, 1H), 3.61 (d, *J* = 16.2 Hz, 1H), 1.56 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 169.4, 148.3, 135.2, 133.0, 128.8, 120.1, 86.4, 84.8, 84.2, 49.7, 33.6, 28.0; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.28 min, t_R (minor) = 14.50 min.



4d

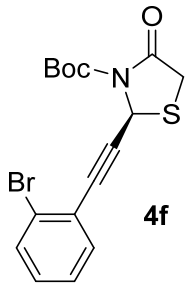
***Tert*-butyl (*S*)-2-((4-fluorophenyl)ethynyl)-4-oxothiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3d** was obtained as clear yellow oil (8 h, 31.3 mg, 97% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4d** was obtained as clear yellow oil (6 h, 20.0 mg, 62% yield of 2-steps, 91% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{20}$ = -292.1 (*c* 1, $CHCl_3$); **HRMS (ESI-TOF)**

calculated for $C_{16}H_{16}FNO_3SNa$ $[M + Na]^+$: 344.0727, found: 344.0729; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.43-7.38 (m, 2H), 7.06-7.00 (m, 2H), 5.89 (s, 1H), 4.02 (d, J = 16.0 Hz, 1H), 3.62 (d, J = 16.0 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.5, 162.9 (d, $^1J_{CF}$ = 249.1 Hz), 148.3, 133.7 (d, $^3J_{CF}$ = 8.44 Hz), 117.7 (d, $^4J_{CF}$ = 3.62 Hz), 115.8 (d, $^2J_{CF}$ = 21.9 Hz), 85.2, 84.8, 84.3, 49.8, 33.6, 28.0; **^{19}F NMR** (376 MHz, $CDCl_3$, ppm): δ -109.5 (s); **HPLC analysis** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.67 min, t_R (minor) = 15.08 min.



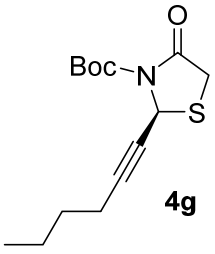
4e

***Tert*-butyl (*S*)-4-oxo-2-((4-(trifluoromethyl)phenyl)ethynyl)thiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3e** was obtained as clear yellow oil (8 h, 32.6 mg, 87% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4e** was obtained as clear yellow oil (10 h, 25.6 mg, 69% yield of 2-steps, 93% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -3.4 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{17}H_{16}F_3NO_3SNa$ $[M + Na]^+$: 394.0695, found: 394.0696; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.61-7.59 (m, 2H), 7.54-7.52 (m, 2H), 5.92 (s, 1H), 4.03 (d, J = 16.0 Hz, 1H), 3.63 (d, J = 16.4 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.3, 148.2, 132.0, 130.8 (q, $^2J_{CF}$ = 32.5 Hz), 125.4 (q, $^3J_{CF}$ = 3.9 Hz), 123.7 (q, $^1J_{CF}$ = 273.4 Hz), 87.8, 84.9, 83.8, 49.6, 33.5, 28.0; **^{19}F NMR** (376 MHz, $CDCl_3$, ppm): δ -63.0 (s); **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 9.27 min, t_R (minor) = 10.79 min.



4f

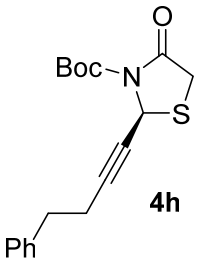
***Tert*-butyl (*S*)-2-((2-bromophenyl)ethynyl)-4-oxothiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3f** was obtained as clear yellow oil (8 h, 34.6 mg, 90% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4f** was obtained as clear yellow oil (8 h, 23.7 mg, 62% yield of 2-steps, 94% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -2.2 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{16}H_{16}BrNO_3SNa$ $[M + Na]^+$: 403.9926, found: 403.9923; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 7.60-7.58 (m, 1H), 7.45-7.44 (m, 1H), 7.29-7.28 (m, 1H), 7.23-7.20 (m, 1H), 5.92 (s, 1H), 4.08 (d, *J* = 16.1 Hz, 1H), 3.61 (d, *J* = 16.1 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 169.4, 148.2, 133.4, 132.5, 130.2, 127.1, 125.8, 123.9, 89.9, 84.8, 83.9, 50.0, 33.7, 28.0; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.51 min, t_R (minor) = 15.39 min.



4g

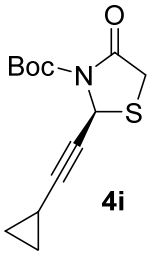
***Tert*-butyl (*S*)-2-(hex-1-yn-1-yl)-4-oxothiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3g** was obtained as clear yellow oil (8 h, 21.5 mg, 75% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4g** was obtained as clear yellow oil (10 h, 13.0 mg, 46% yield of 2-steps, 87% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -7.0 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{14}H_{21}NO_3SNa$

$[M + Na]^+$: 306.1134, found: 306.1133; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 5.67 (s, 1H), 3.95 (d, J = 16.2 Hz, 1H), 3.55 (d, J = 16.2 Hz, 1H), 2.23 (td, J = 6.9, 1.6 Hz, 2H), 1.55 (s, 9H), 1.51-1.45 (m, 2H), 1.44-1.36 (m, 2H), 0.91 (t, J = 7.3 Hz, 3H) ; **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.6, 148.3, 86.7, 84.4, 76.9, 49.7, 33.5, 30.3, 27.9, 21.9, 18.4, 13.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 220 nm, retention time: t_R (major) = 10.72 min, t_R (minor) = 12.65 min.



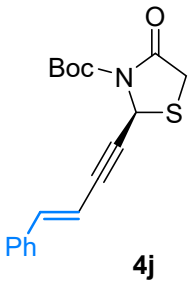
4h

***Tert*-butyl (*S*)-4-oxo-2-(4-phenylbut-1-yn-1-yl)thiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3h** was obtained as clear yellow oil (8 h, 27.4 mg, 82% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4h** was obtained as clear yellow oil (10 h, 25.1 mg, 55% yield of 2-steps, 84% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -9.2 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{18}H_{21}NO_3SNa$ $[M + Na]^+$: 354.1134, found: 354.1135; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 7.31-7.28 (m, 2H), 7.23-7.20 (m, 3H), 5.62 (d, J = 2.0, 1H), 3.85 (dd, J = 16.1, 2.2 Hz, 1H), 3.51 (dd, J = 16.1, 2.3 Hz, 1H), 2.81 (t, J = 7.5 Hz, 2H), 2.53 (tt, J = 7.5, 2.0 Hz, 2H), 1.53 (s, 9H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 169.5, 148.4, 140.1, 128.5, 128.4, 126.5, 85.8, 84.4, 77.8, 49.6, 34.6, 33.6, 28.0, 20.9; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.42 min, t_R (minor) = 15.02 min.



4i

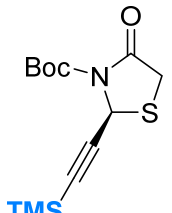
***Tert*-butyl (*S*)-2-(cyclopropylethynyl)-4-oxothiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3i** was obtained as clear colorless oil (8 h, 21.2 mg, 79% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4i** was obtained as clear colorless oil (12 h, 14.2 mg, 53% yield of 2-steps, 77% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -5.6 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{13}H_{17}NO_3SNa$ $[M + Na]^+$: 290.0821, found: 290.0820; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 5.63 (d, *J* = 1.8 Hz, 1H), 3.94 (d, *J* = 16.1 Hz, 1H), 3.54 (d, *J* = 16.2 Hz, 1H), 1.55 (s, 9H), 1.30-1.25 (m, 1H), 0.83-0.80 (m, 2H), 0.70-0.68 (m, 2H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 170.2, 148.9, 90.2, 85.0, 72.4, 50.2, 34.1, 28.5, 8.94, 8.92, -0.0; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 220 nm, retention time: t_R (major) = 12.84 min, t_R (minor) = 15.38 min.



4j

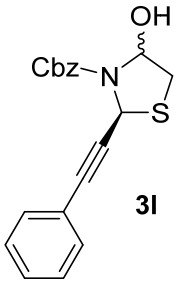
***Tert*-butyl (*S,E*)-4-oxo-2-(4-phenylbut-3-en-1-yn-1-yl)thiazolidine-3-carboxylate**. Following the *General Procedure*, the crude product **3j** was obtained as clear colorless oil (8 h, 30.5 mg, 92% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4j** was obtained as clear colorless oil (12 h, 23.4 mg, 71% yield of 2-steps, 86% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -222.5 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)**

calculated for $C_{18}H_{19}NNaO_3S$ $[M + Na]^+$: 352.0978, found: 352.0999; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.42-7.28 (m, 5H), 6.97 (d, J = 16.3 Hz, 1H), 6.17 (dd, J = 16.3, 1.7 Hz, 1H), 5.86 (d, J = 1.7 Hz, 1H), 4.01 (d, J = 16.1 Hz, 1H), 3.59 (d, J = 16.1 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.4, 148.3, 143.0, 135.7, 129.1, 128.8, 126.4, 106.5, 87.3, 84.73, 84.69, 50.0, 33.6, 28.0; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 14.88 min, t_R (minor) = 16.71 min.



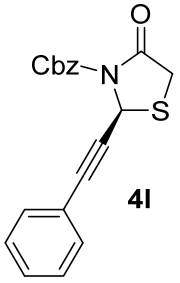
4k

***Tert*-butyl (*S*)-4-oxo-2-((trimethylsilyl)ethynyl)thiazolidine-3-carboxylate.** Following the *General Procedure*, the crude product **3k** was obtained as clear colorless oil (8 h, 29.5 mg, 98% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4k** was obtained as clear colorless oil (12 h, 18.0 mg, 60% yield of 2-steps, 91% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -233.2 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{13}H_{21}NNaO_3SSi$ $[M + Na]^+$: 322.0904, found: 322.0899; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 5.62 (s, 1H), 3.93 (d, J = 16.1 Hz, 1H), 3.53 (d, J = 16.1 Hz, 1H), 1.53 (s, 9H), 0.15 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 170.0, 148.4, 101.3, 91.0, 84.9, 49.7, 33.7, 28.3, -0.004; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 210 nm, retention time: t_R (major) = 9.15 min, t_R (minor) = 10.83 min.



3l

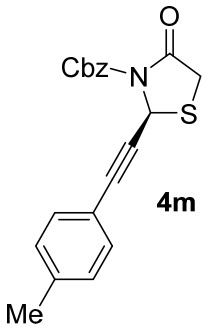
Benzyl (2*S*)-4-hydroxy-2-(phenylethynyl)thiazolidine-3-carboxylate. Following the *General Procedure* (without PDC-mediated oxidation procedure), **3l** was obtained as clear colorless oil (8 h, 32.0 mg, 94% yield, 1:1 dr, 93% (93%) ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{20}$ = -194.0 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{19}H_{17}NO_3SNa$ $[M + Na]^+$: 362.0821, found: 362.0822; **^{1}H NMR** (400 MHz, acetone-d^6, ppm): δ 7.47-7.31 (m, 20H), 6.06 (s, 1H), 5.98 (s, 1H), 5.90 (d, *J* = 3.6 Hz, 1H), 5.83 (s, 1H), 5.63 (s, 2H), 5.30 (d, *J* = 12.4 Hz, 2H), 5.14 (d, *J* = 12.8 Hz, 2H), 3.61-3.58 (m, 1H), 3.45 (dd, *J* = 11.2, 4.8 Hz, 1H), 3.28-3.25 (m, 1H), 3.09 (d, *J* = 12.0 Hz, 1H); **^{13}C NMR** (100 MHz, acetone-d^6, ppm): δ 152.81, 152.76, 136.8, 136.7, 131.5, 128.6, 128.5, 128.4, 128.3, 127.9, 127.8, 127.7, 122.6, 122.5, 121.3, 120.0, 88.13, 88.08, 83.1, 82.4, 67.0, 66.9, 54.1, 34.0, 26.2, 24.4, 23.6; **HPLC analysis** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major of isomer A) = 27.55 min, t_R (minor of isomer A) = 30.40 min; t_R (minor of isomer B) = 70.64 min, t_R (major of isomer B) = 76.50 min.



4l

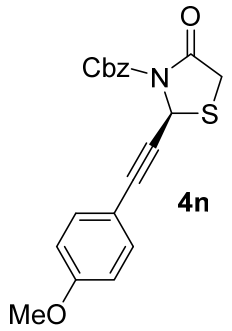
Benzyl (*S*)-4-oxo-2-(phenylethynyl)thiazolidine-3-carboxylate. Following the *General Procedure*, **4l** was obtained as a pale yellow solid (10 h, 21.2 mg, 63% yield of 2-steps, 93% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -255.4 (*c* 1.0, $CHCl_3$); m.p. 80-81 °C; **HRMS**

(ESI-TOF) calculated for $C_{19}H_{15}NO_3SNa$ $[M + Na]^+$: 360.0665, found: 360.0663; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.45-7.44 (m, 2H), 7.38-7.33 (m, 8H), 5.98 (s, 1H), 5.42 (d, J = 12.3 Hz, 1H), 5.30 (d, J = 12.2 Hz, 1H), 4.07 (d, J = 16.3 Hz, 1H), 3.63 (d, J = 16.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.3, 149.9, 134.7, 131.9, 129.1, 128.7, 128.6, 128.35, 128.29, 121.4, 85.9, 85.0, 69.0, 49.8, 33.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 24.44 min, t_R (minor) = 27.10 min.

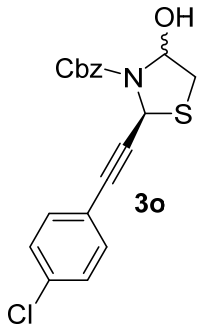


4m

Benzyl (*S*)-4-oxo-2-(*p*-tolylethynyl)thiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3m** was obtained as clear yellow oil (8 h, 32.0 mg, 91% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4m** was obtained as clear yellow oil (10 h, 22.9 mg, 65% yield of 2-steps, 92% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -274.3 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{20}H_{17}NO_3SNa$ $[M + Na]^+$: 374.0821, found: 374.0816; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.45-7.43 (m, 2H), 7.34-7.32 (m, 3H), 7.27-7.25 (m, 2H), 7.13-7.11 (m, 2H), 5.97 (s, 1H), 5.41 (d, J = 12.3 Hz, 1H), 5.29 (d, J = 12.3 Hz, 1H), 4.06 (d, J = 16.3 Hz, 1H), 3.62 (d, J = 16.2 Hz, 1H), 2.36 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.3, 149.9, 139.4, 134.7, 131.8, 129.1, 128.7, 128.6, 128.3, 118.4, 86.1, 84.3, 68.9, 49.9, 33.5, 21.6; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 25.22 min, t_R (minor) = 27.80 min.

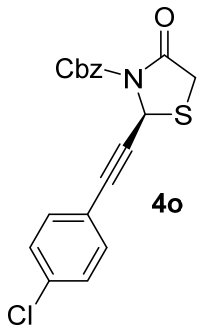


Benzyl (*S*)-2-((4-methoxyphenyl)ethynyl)-4-oxothiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3n** was obtained as clear colorless oil (8 h, 34.2 mg, 93% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4n** was obtained as a pale white solid (10 h, 23.8 mg, 65% yield of 2-steps, 90% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -287.2 (*c* 1.0, $CHCl_3$); m.p. 92-93 °C; **HRMS (ESI-TOF)** calculated for $C_{20}H_{17}NO_4SNa$ $[M + Na]^+$: 390.0770, found: 390.0766; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 7.45-7.43 (m, 2H), 7.33-7.30 (m, 5H), 6.84-6.83 (m, 2H), 5.96 (s, 1H), 5.41 (d, *J* = 12.6 Hz, 1H), 5.29 (d, *J* = 12.0 Hz, 1H), 4.05 (d, *J* = 16.2 Hz, 1H), 3.81 (s, 3H), 3.61 (d, *J* = 16.2 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 169.3, 160.2, 149.9, 134.8, 133.4, 128.64, 128.55, 128.2, 114.0, 113.5, 86.0, 83.8, 68.9, 55.3, 50.0, 33.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 36.67 min, t_R (minor) = 41.63 min.



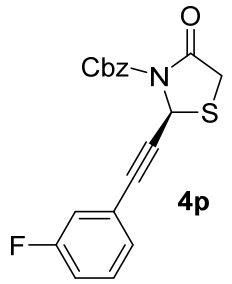
Benzyl (2*S*)-2-((4-chlorophenyl)ethynyl)-4-hydroxythiazolidine-3-carboxylate. Following the *General Procedure* (without PDC-mediated oxidation procedure), **3o** was obtained as clear colorless oil (8 h, 34.3 mg, 92% yield, 1.3:1 dr, 97% (97%) ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20);

$[\alpha]_D^{20}$ = -181.7 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{19}H_{16}ClNO_3SNa$ [M + Na]$^+$: 396.0432, found: 396.0431; **^{1}H NMR** (400 MHz, acetone-d^6, ppm): δ 7.47-7.32 (m, 18H), 6.06 (s, 1H), 5.97 (s, 1H), 5.90 (d, *J* = 4.0 Hz, 1H), 5.82 (s, 1H), 5.63 (s, 2H), 5.30 (d, *J* = 12.4 Hz, 2H), 5.13 (d, *J* = 12.8 Hz, 2H), 3.59 (d, *J* = 12.0 Hz, 1H), 3.45 (dd, *J* = 11.2, 4.4 Hz, 1H), 3.28-3.25 (m, 1H), 3.09 (d, *J* = 12.0 Hz, 1H); **^{13}C NMR** (100 MHz, acetone-d^6, ppm): δ 152.7, 146.8, 136.8, 136.7, 134.1, 133.1, 128.7, 128.40, 128.36, 128.0, 127.9, 127.7, 121.4, 121.3, 89.3, 89.2, 67.0, 66.9, 54.1, 34.0, 26.2, 24.4, 23.6; **HPLC analysis** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 95:5, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major of isomer A) = 22.28 min, t_R (minor of isomer A) = 24.50 min; t_R (minor of isomer B) = 63.07 min, t_R (major of isomer B) = 74.68 min.



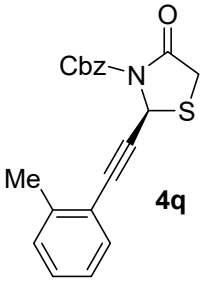
4o

Benzyl (*S*)-2-((4-chlorophenyl)ethynyl)-4-oxothiazolidine-3-carboxylate. Following the *General Procedure*, **4o** was obtained as a pale yellow solid (10 h, 24.6 mg, 63% yield of 2-steps, 97% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -162.2 (*c* 1.0, $CHCl_3$); m.p. 102-103 °C; **HRMS (ESI-TOF)** calculated for $C_{19}H_{14}ClNO_3SNa$ [M + Na]$^+$: 394.0275, found: 394.0273; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.43-7.39 (m, 2H), 7.34-7.29 (m, 7H), 5.96 (s, 1H), 5.42 (d, *J* = 12.2 Hz, 1H), 5.29 (d, *J* = 12.2 Hz, 1H), 4.05 (d, *J* = 16.3 Hz, 1H), 3.63 (d, *J* = 16.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.1, 149.9, 135.3, 134.7, 133.1, 128.74, 128.67, 128.3, 119.9, 86.0, 84.7, 69.0, 49.7, 33.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 23.71 min, t_R (minor) = 26.37 min.



4p

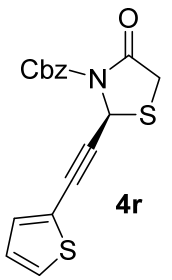
Benzyl (*S*)-2-((3-fluorophenyl)ethynyl)-4-oxothiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3p** was obtained as clear yellow oil (8 h, 31.1 mg, 87% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4p** was obtained as clear yellow oil (8 h, 19.6 mg, 55% yield of 2-steps, 92% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -284.6 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{19}H_{14}FNO_3SNa$ $[M + Na]^+$: 378.0571, found: 378.0569; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.45-7.43 (m, 2H), 7.34-7.28 (m, 4H), 7.16-7.04 (m, 3H), 5.96 (s, 1H), 5.43 (d, *J* = 12.2 Hz, 1H), 5.29 (d, *J* = 12.2 Hz, 1H), 4.05 (d, *J* = 16.3 Hz, 1H), 3.63 (d, *J* = 16.3 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.1, 162.2 (d, $^1J_{C\text{-}F}$ = 245.8 Hz), 149.9, 134.6, 130.0 (d, $^3J_{CF}$ = 8.5 Hz), 128.70, 128.68, 128.4, 127.8 (d, $^4J_{CF}$ = 3.0 Hz), 123.2 (d, $^3J_{CF}$ = 9.6 Hz), 118.7 (d, $^2J_{CF}$ = 22.9 Hz), 116.6 (d, $^2J_{CF}$ = 21.1 Hz), 85.9, 84.5 (d, $^4J_{CF}$ = 3.3 Hz), 69.0, 49.6, 33.4; **^{19}F NMR** (376 MHz, $CDCl_3$, ppm): δ -112.5 (s); **HPLC analysis** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 23.26 min, t_R (minor) = 25.87 min.



4q

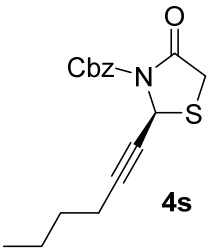
Benzyl (*S*)-4-oxo-2-(*o*-tolylethynyl)thiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3q** was obtained as clear colorless oil (8 h, 32.6 mg, 92% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum

ether = 1/20); **4q** was obtained as clear colorless oil (10 h, 23.0 mg, 65% yield of 2-steps, 90% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -331.5 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{20}H_{17}NO_3SNa$ $[M + Na]^+$: 374.0821, found: 374.0822; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.45-7.43 (m, 2H), 7.34-7.32 (m, 4H), 7.27-7.12 (m, 3H), 6.01 (s, 1H), 5.39 (d, *J* = 12.4 Hz, 1H), 5.32 (d, *J* = 12.8 Hz, 1H), 4.06 (d, *J* = 16.4 Hz, 1H), 3.64 (d, *J* = 16.2 Hz, 1H), 2.33 (s, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.2, 149.9, 140.6, 134.7, 132.1, 129.5, 129.2, 128.7, 128.6, 128.3, 125.6, 121.2, 88.8, 85.0, 69.0, 50.0, 33.5, 20.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 90:10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 35.86 min, t_R (minor) = 42.61 min.



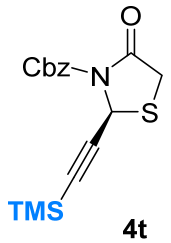
4r

Benzyl (*S*)-4-oxo-2-(thiophen-2-ylethynyl)thiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3r** was obtained as clear yellow oil (8 h, 31.6 mg, 91% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4r** was obtained as clear yellow oil (10 h, 19.1 mg, 56% yield of 2-steps, 93% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/25); $[\alpha]_D^{20}$ = -301.1 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{17}H_{13}NO_3S_2Na$ $[M + Na]^+$: 366.0229, found: 366.0226; **^{1}H NMR** (600 MHz, $CDCl_3$, ppm): δ 7.39-7.35 (m, 2H), 7.29-7.23 (m, 4H), 7.15-7.13 (m, 1H), 6.94-6.92 (s, 1H), 5.91 (s, 1H), 5.34 (d, *J* = 12.0 Hz, 1H), 5.23 (d, *J* = 12.0 Hz, 1H), 3.99 (d, *J* = 16.2 Hz, 1H), 3.55 (d, *J* = 16.2 Hz, 1H); **^{13}C NMR** (150 MHz, $CDCl_3$, ppm): δ 169.1, 149.8, 134.7, 133.3, 128.7, 128.6, 128.30, 128.25, 127.1, 121.3, 88.7, 79.4, 69.0, 49.9, 33.5; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 27.52 min, t_R (minor) = 30.23 min.



4s

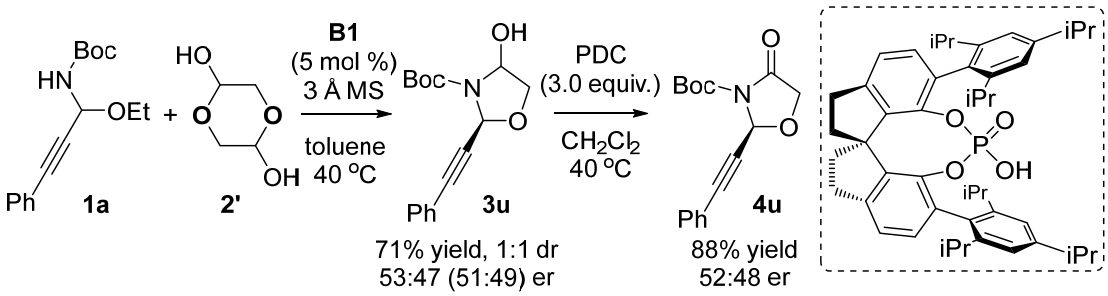
Benzyl (*S*)-2-(hex-1-yn-1-yl)-4-oxothiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3s** was obtained as clear yellow oil (8 h, 25.0 mg, 78% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4s** was obtained as clear yellow oil (10 h, 13.5 mg, 43% yield of 2-steps, 90% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{20}$ = -206.6 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{17}H_{19}NO_3SNa$ [M + Na]$^+$: 340.0978, found: 340.0979; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.45-7.32 (m, 5H), 5.73 (t, *J* = 1.6, 1H), 5.37 (d, *J* = 12.4 Hz, 1H), 5.29 (d, *J* = 12.4 Hz, 1H), 3.98 (d, *J* = 16.4 Hz, 1H), 3.56 (d, *J* = 16.0 Hz, 1H), 2.20 (dt, *J* = 7.2, 1.6 Hz, 2H), 1.51-1.42 (m, 2H), 1.40-1.31 (m, 2H), 0.89 (t, *J* = 7.2, 3H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.4, 150.0, 134.8, 128.62, 128.57, 128.2, 87.4, 76.5, 68.8, 49.7, 33.5, 30.2, 21.9, 18.5, 13.6; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 220 nm, retention time: t_R (major) = 19.95 min, t_R (minor) = 22.19 min.



4t

Benzyl (*S*)-4-oxo-2-((trimethylsilyl)ethynyl)thiazolidine-3-carboxylate. Following the *General Procedure*, the crude product **3t** was obtained as clear colorless oil (8 h, 33.2 mg, 98% yield) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); **4t** was obtained as clear colorless oil (12 h, 20.0 mg, 60% yield of 2-steps, 92% ee) after flash chromatography (elution gradient: ethyl acetate/petroleum ether = 1/20); $[\alpha]_D^{25}$ = -318.2 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)**

calculated for $C_{16}H_{19}NNaO_3SSi$ $[M + Na]^+$: 356.0747, found: 356.0752; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.46-7.42 (m, 2H), 7.40-7.32 (m, 3H), 5.71 (s, 1H), 5.37 (d, J = 12.3 Hz, 1H), 5.28 (d, J = 12.3 Hz, 1H), 3.99 (d, J = 16.2 Hz, 1H), 3.56 (d, J = 16.2 Hz, 1H), 0.16 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 169.7, 150.1, 135.1, 129.01, 128.98, 128.6, 100.8, 91.8, 69.3, 49.8, 33.8, -0.004; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 210 nm, retention time: t_R (major) = 14.12 min, t_R (minor) = 16.22 min.

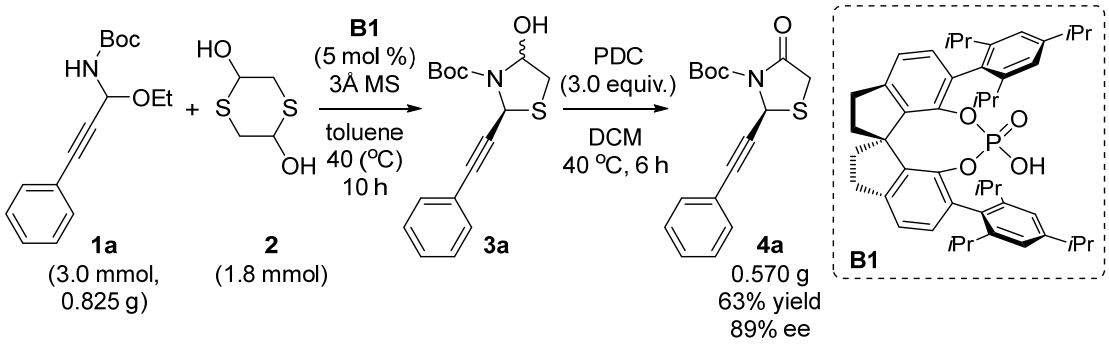


General procedure for annulation: The freshly dry toluene (1 mL) was added to the dry 25-mL tube containing with alkynyl imines **1a** (27.5 mg, 0.1 mmol), the chiral catalyst **B1** (3.6 mg, 5 mol %), 1,4-dioxane-2,5-diol **2'** (7.2 mg, 0.06 mmol) and 3Å MS (30 mg). The reacting mixture was stirred at 40 °C for 8 hours. Upon the completion of alkynyl imines monitored by TLC analysis, the mixture was directly purified by flash column chromatography (elution: ethyl acetate/petroleum ether = 1/15) to afford the crude chiral oxazolidine **3u** [20.6 mg, 71% yield, 1:1 dr, ca. 5% (3%) ee]. **HRMS (ESI-TOF)** calculated for $C_{16}H_{19}NNaO_4$ $[M + Na]^+$: 312.1206, found: 312.1210; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 90:10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (minor, isomer A) = 9.22 min, t_R (major, isomer A) = 9.66 min; t_R (minor, isomer B) = 13.07 min, t_R (major, isomer B) = 20.77 min.

General procedure for PDC-mediated oxidation: The solution of the obtained crude products **3u** (20.0 mg, 1.0 equiv.) in DCM (1.0 mL) was added PDC (78 mg, 3.0 equiv.). The mixture was stirred at 40 °C for 10 hours. Then, the mixture was cooled to rt and added an appropriate amount of silica gel, which gave a good solid residue after the solvent was removed under reduced pressure. The residue was purified by silica gel

column chromatography (ethyl acetate/petroleum ether = 1/20) to afford the desired product **4u** (18.2 mg, 88% yield, 4% ee) as a colorless oil. ***Tert*-butyl (*S*)-4-oxo-2-(phenylethynyl)oxazolidine-3-carboxylate (4u): HRMS (ESI-TOF)** calculated for $C_{16}H_{17}NO_4Na$ $[M + Na]^+$: 310.1050, found: 310.1052; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.46-7.44 (m, 2H), 7.41-7.32 (m, 3H), 6.30 (s, 1H), 4.52 (d, J = 14.8 Hz, 1H), 4.35 (d, J = 14.8 Hz, 1H), 1.57 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 168.4, 147.1, 131.9, 129.4, 128.5, 121.0, 86.7, 84.7, 82.9, 80.3, 67.2, 28.0; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (minor) = 11.71 min, t_R (major) = 15.24 min.

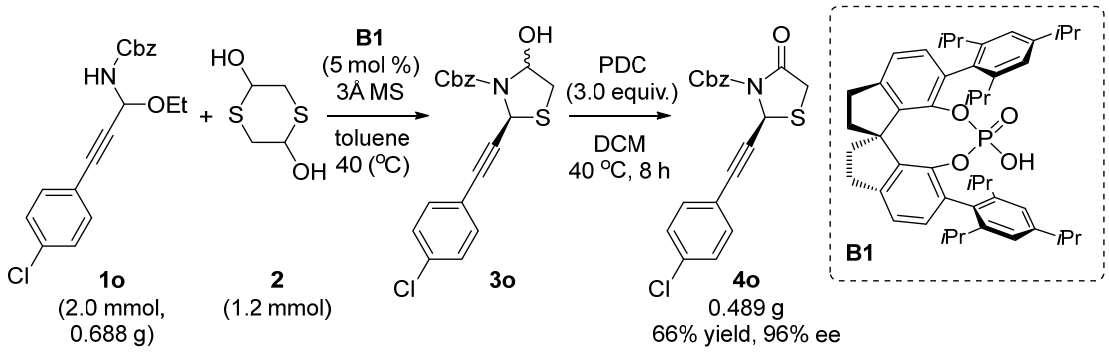
5. General Procedure for the Gram-Scale Syntheses



General Procedure for the Large-Scale Synthesis of 4a: To a stirred mixture of alkynyl imine precursor **1a** (0.825 g, 3.0 mmol), the chiral catalyst **B1** (107.7 mg, 5 mol %) and 3 Å MS (90 mg) in toluene (30 mL) was added 2,5-dihydroxy-1,4-dithiane **2** (274 mg, 1.8 mmol). The resulting mixture was stirred at 40 °C for 10 hours. The solvent was concentrated and the residue was purified by flash column chromatography (ethyl acetate/petroleum ether = 1/15) to afford crude thiazolidine **3a** (861.2 mg, 94% yield).

Then, crude thiazolidine **3a** (861.2 mg, 2.82 mmol) was dissolved in DCM (25 mL), followed by the addition of PDC (3.16 g, 8.4 mmol, 3.0 equiv). The mixture was stirred at 40 °C for 8 hours. Then, the reacting mixture was cooled to room temperature

and added an appropriate amount of silica gel, which gave a good solid residue after the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/25 to 1/20) to afford chiral thiazolidone **4a** (0.570 g, 63% yield of 2-steps, 89% ee). **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 12.44 min, t_R (minor) = 14.72 min.



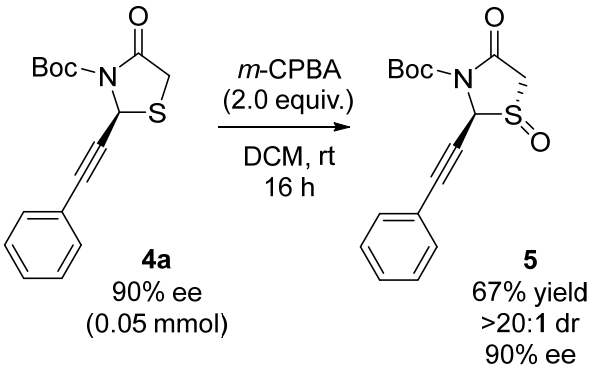
General Procedure for the Large-Scale Synthesis of 4m: To a stirred mixture of alkynyl imines **1o** (0.688 g, 2.0 mmol), the chiral catalyst **B1** (72 mg, 5 mol %) and 3Å MS (60 mg) in toluene (20 mL) was added 2,5-dihydroxy-1,4-dithiane **2** (185 mg, 1.2 mmol). The resulting mixture was stirred at 40 °C for 10 h. The solvent was concentrated and the residue was purified by flash column chromatography (ethyl acetate/petroleum ether = 1/15) to afford the crude thiazolidine **3o** (718.8 mg, 90% yield).

Then, the solution of crude **3o** (718.8 mg, 1.8 mmol) in DCM (18 mL) was added PDC (2.03 g, 5.4 mmol, 3.0 equiv). The mixture was stirred at 40 °C for 8 hours. Then, the reacting mixture was cooled to room temperature and added an appropriate amount of silica gel, which gave a good solid residue after the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/25 to 1/20) to afford the chiral thiazolidone **4o** (0.489 g, 66% yield of 2-steps, 96% ee). **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH

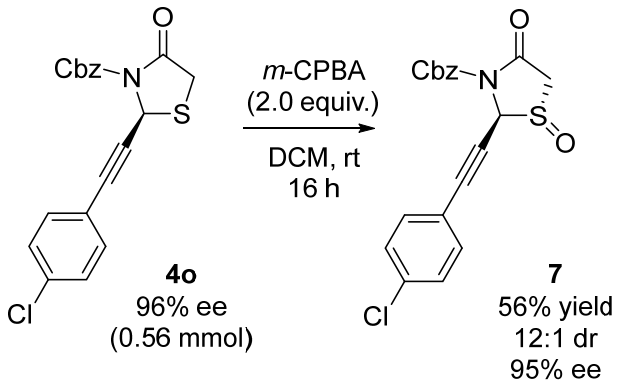
= 80:20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 23.73 min, t_R (minor) = 26.41 min.

6. Synthetic Applications

6.1 General Procedure of Oxidation

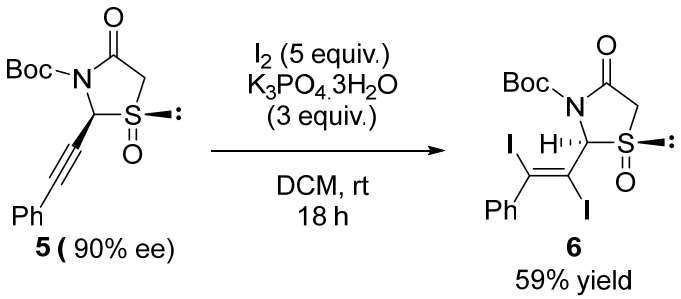


General Procedure for the Synthesis of Chiral Sulfoxide 5: The solution of **4a** (15.7 mg, 0.05 mmol, 90% ee) in DCM (1 mL) was added *m*-CPBA (2.0 equiv.). The mixture was stirred overnight at room temperature. After removal of *m*-chlorobenzoic acid by filtration, the filtrate was washed with aqueous $KHCO_3$ and then aqueous NaCl, the organic layer was dried over anhydrous $MgSO_4$, concentrated, and purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/5) to afford the desired product **5** as a clear yellow oil (16 h, 11.0 mg, 67% yield, >20:1 dr, 90% ee). ***Tert*-butyl (1*S*, 2*S*)-4-oxo-2-(phenylethynyl)thiazolidine-3-carboxylate 1-oxide (5):** $[\alpha]_D^{20}$ = -227.4 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{16}H_{17}NO_4SNa$ $[M + Na]^+$: 342.0770, found: 342.0769; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.44-7.34 (m, 5H), 5.85 (d, *J* = 1.44 Hz, 1H), 4.00 (d, *J* = 16.8 Hz, 1H), 3.68 (dd, *J* = 16.8, 1.6 Hz, 1H), 1.59 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.3, 148.7, 132.0, 130.0, 128.6, 120.3, 91.9, 85.6, 69.5, 55.0, 53.5, 28.0; **HPLC analysis** Daicel CHIRALCEL OD-H, *n*-Hexane/*i*-PrOH = 70:30, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 15.36 min, t_R (minor) = 20.29 min.



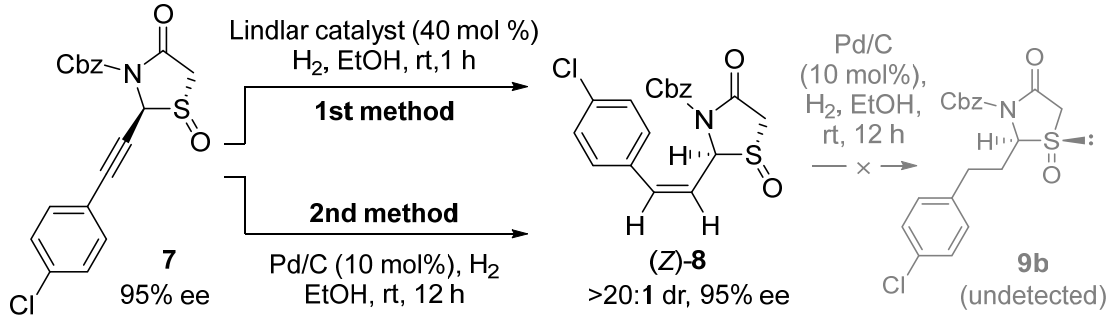
General Procedure for the Synthesis of Chiral Sulfoxide 7: The solution of **4o** (208.0 mg, 0.56 mmol, 96% ee) in DCM (10 mL) was added *m*-CPBA (2.0 equiv.). The mixture was stirred overnight at room temperature. After removal of *m*-chlorobenzoic acid by filtration, the filtrate was washed with aqueous $KHCO_3$ and then aqueous NaCl, the organic layer was dried over anhydrous $MgSO_4$, concentrated, and purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/5) to afford the desired product **7** as a pale yellow solid (16 h, 122.0 mg, 56% yield, 12:1 dr, 95% (95%) ee). **Benzyl (1*S*, 2*S*)-2-((4-chlorophenyl)ethynyl)-4-oxothiazolidine-3-carboxylate 1-oxide (7).** $[\alpha]_D^{20}$ = -222.4 (*c* 1.0, $CHCl_3$); m.p. 47-48 °C; **HRMS (ESI-TOF)** calculated for $C_{19}H_{14}ClNO_4SNa$ $[M + Na]^+$: 410.0224, found: 410.0220; *For major diastereomer* **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.46-7.43 (m, 2H), 7.37-7.33 (m, 7H), 5.89 (d, *J* = 1.44 Hz, 1H), 5.42 (d, *J* = 12.2 Hz, 1H), 5.35 (d, *J* = 12.2 Hz, 1H), 4.00 (d, *J* = 16.8 Hz, 1H), 3.70 (dd, *J* = 16.8, 1.72 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.0, 150.4, 136.4, 134.3, 133.2, 129.0, 128.8, 128.7, 128.3, 118.5, 91.2, 78.0, 69.5, 69.2, 54.9; **HPLC analysis:** Daicel CHIRALCEL OD-H, *n*-Hexane/*i*-PrOH = 70:30, flow rate = 1.0 mL/min, λ = 254 nm, retention time: *minor diastereomer* t_R (minor) = 19.03 min, t_R (major) = 21.52 min, *major diastereomer* t_R (minor) = 33.38 min, t_R (major) = 47.65 min.

6.2 General Procedure of I_2-Addition



General Procedure for the Synthesis of 6: The solution of **5** (23.0 mg, 0.07 mmol, 90% ee) was dissolved in DCM (1 mL), followed by the addition of $K_3PO_4{\cdot}3H_2O$ (57.5 mg, 0.22 mmol), I_2 (91.4 mg, 0.36 mmol). The mixture was refluxed for another 18h. Then the reaction was quenched by aqueous $Na_2S_2O_3$ and extracted with CH_2Cl_2 for three times. The organic layers were combined, dried over Na_2SO_4, filtered and evaporated under reduced pressure. The residue was purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/5) to afford the desired product **6** as clear colorless oil (18 h, 24.2 mg, 59% yield, >20:1 dr). ***Tert*-butyl (2*S*)-2-((*E*)-1,2-diiodo-2-phenylvinyl)-4-oxothiazolidine-3-carboxylate 1-oxide (6).** $[\alpha]_D^{20}$ = -157.4 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{16}H_{17}I_2NO_4SNa$ $[M + Na]^+$: 595.8860, found: 595.8865; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.44-7.36 (m, 3H), 7.21-7.19 (m, 2H), 5.93 (s, 1H), 3.86 (d, J = 17.2 Hz, 1H), 3.73 (d, J = 17.6 Hz, 1H), 1.61 (s, 9H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 167.6, 148.0, 146.8, 129.3, 128.9, 127.7, 104.0, 96.3, 91.0, 85.5, 56.7, 28.1.

6.3 General Procedure of Hydrogenation



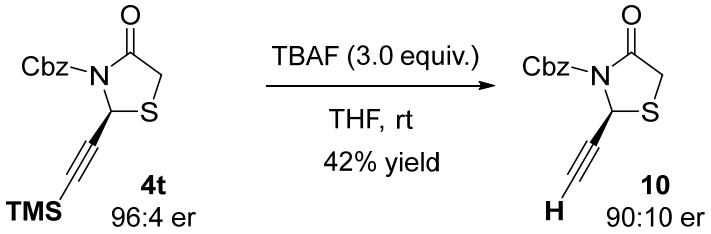
General Procedure for the Synthesis of 8 (1st method)[2]: Lindlar catalyst ($Pd/CaCO_3$)

(313.9 mg, 40 mol %) was suspended in EtOH (10 mL), **7** (147.4 mg, 0.38 mmol, 95% ee) and quinoline (34.4 mg, 0.7 equiv.) were added. The reaction mixture was stirred at room temperature for 1 hour under hydrogen atmosphere. Lindlar catalyst was removed by filtration over a pad of celite, the filtrate was concentrated in vacuo, and purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/2) to afford the desired product **8** as a clear colorless oil (1 h, 82.7 mg, 56% yield, >20:1 dr, 95% ee).

General Procedure for the Synthesis of 8 (2nd method): Pd/C (38.4 mg, 10 mol %) was suspended in EtOH (5 mL) and **7** (70.0 mg, 0.18 mmol, 95% ee) were added. The reaction mixture was stirred at room temperature for 12 hours under hydrogen atmosphere. Catalyst was removed by filtration over a pad of celite, the filtrate was concentrated in vacuo, and purified by silica gel column chromatography (ethyl acetate/petroleum ether = 1/2) to afford the desired product **8** as a clear colorless oil (12 h, 28.1 mg, 40% yield, >20:1 dr, 95% ee). During this procedure, we didn't detect or isolate the compounds **9b**. Notably, even if **8** was conducted under the procedure once again, there are still no compounds **9b** detected or isolated.

Benzyl (1*S*, 2*S*)-2-((*Z*)-4-chlorostyryl)-4-oxothiazolidine-3-carboxylate 1-oxide (8). $[\alpha]_D^{20}$ = -196.3 (*c* 1.0, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{19}H_{16}ClNO_4SNa$ $[M + Na]^+$: 412.0381, found: 412.0379; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.45-7.30 (m, 6H), 7.25-7.17 (m, 3H), 6.82 (d, *J* = 11.5 Hz, 1H), 6.02 (d, *J* = 9.5 Hz, 1H), 5.28 (dd, *J* = 11.5, 9.5 Hz, 1H), 5.22 (d, *J* = 2.9 Hz, 2H), 3.83 (d, *J* = 17.2 Hz, 1H), 3.69 (dd, *J* = 17.2, 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 166.6, 150.4, 137.3, 134.9, 134.1, 132.8, 129.7, 129.4, 128.8, 128.7, 128.3, 121.2, 78.0, 69.3, 54.5; **HPLC analysis** Daicel CHIRALCEL OD-H, *n*-Hexane/*i*-PrOH = 70:30, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R (major) = 26.18 min, t_R (minor) = 37.16 min.

6.4 General Procedure of Desilication



General procedure for the synthesis of 10: To the solution of **4t** (17.0 mg, 0.05 mmol) in THF (1.0 mL) in an ice-water bath was added the tetrabutylammonium fluoride (TBAF, 44 uL, 0.15 mmol, 3.0 equiv). The reaction mixture was stirred at room temperature for 2 h and then the solution was poured into saturated aq. NH_4Cl with rapid stirring. The resulting suspension was transferred to a separatory funnel and extracted three times with Et_2O. The combined organic portions were dried (Na_2SO_4), filtered, and concentrated. The residue was purified by silica gel column chromatography with the eluent of ethyl acetate/petroleum ether 1/20-1/15 to afford compound **10** as a colorless oil (5.6 mg, 42% yield, 80% ee), which contains a few of inseparable and unknown impurity (δ = 4.71 (d, J = 5.5 Hz, 0.4 H)).

Benzyl *(S)*-2-ethynyl-4-oxothiazolidine-3-carboxylate (10)*:* $[\alpha]_D^{20}$ = -117.9 (*c* 0.1, $CHCl_3$); **HRMS (ESI-TOF)** calculated for $C_{13}H_{11}NNaO_3S$ $[M + Na]^+$: 284.0352, found: 284.0355; **^{1}H NMR** (400 MHz, $CDCl_3$, ppm): δ 7.46-7.41 (m, 2H), 7.41-7.33 (m, 3H), 5.72 (d, J = 1.6 Hz, 1H), 5.36 (d, J = 17.8 Hz, 1H), 5.33 (d, J = 17.8 Hz, 1H), 4.01 (d, J = 16.3 Hz, 1H), 3.59 (d, J = 16.3 Hz, 1H), 2.66 (d, J = 1.6 Hz, 1H); **^{13}C NMR** (100 MHz, $CDCl_3$, ppm): δ 168.8, 149.9, 134.6, 128.7, 128.6, 127.0, 79.9, 74.3, 69.1, 48.9, 33.3; **HPLC analysis:** Daicel CHIRALPAK IC, *n*-Hexane/*i*-PrOH = 90:10, flow rate = 1.0 mL/min, λ = 210 nm, retention time: t_R (minor) = 43.019 min, t_R (major) = 45.302 min.

7. Assignment of Absolute Configuration for Products

Experimental: Single crystals of $C_{19}H_{14}ClNO_3S$ **[4o (szh_wmx2_0m)]** obtained from *n*-Pentane and DCM. A suitable crystal was selected and measured on a

diffractometer. The crystal was kept at 100.(2) K during data collection.

Crystal Data for $C_{19}H_{14}ClNO_3S$ (M =371.82 g/mol): monoclinic, space group $P2_1$ (no. 4), a = 5.5623(3) Å, b = 8.7434(4) Å, c = 17.6707(8) Å, β = 96.868(2)°, V = 853.22(7) Å^3, Z = 2, T = 100.(2) K, μ(Cu Kα) = 3.285 mm^{-1}, *Dcalc* = 1.447 g/cm^3, 12452 reflections measured (5.04° ≤ 2Θ ≤ 144.88°), 3319 unique (R_{int} = 0.0647, R_{sigma} = 0.0577) which were used in all calculations. The final R_1 was 0.0423 (I > 2σ(I)) and wR_2 was 0.1071 (all data).

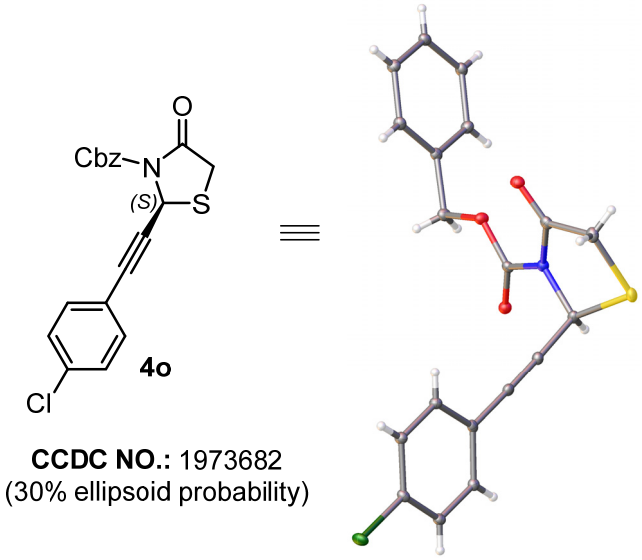


Figure S2. View of **4o**.

Table S6. Crystal data and structure refinement for 4o (szh_wmx2_0m)

Identification code	**4o (szh_wmx2_0m)**
Empirical formula	$C_{19}H_{14}ClNO_3S$
Formula weight	371.82
Temperature/K	100.(2)
Crystal system	monoclinic
Space group	$P2_1$
a/Å	5.5623(3)
b/Å	8.7434(4)
c/Å	17.6707(8)

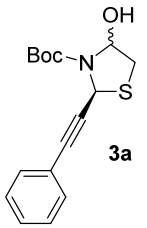
α/°	90
β/°	96.868(2)
γ/°	90
Volume/Å^3	853.22(7)
Z	2
ρ_{calc}g/cm^3	1.447
μ/mm^{-1}	3.285
F(000)	384.0
Crystal size/mm^3	0.250 × 0.180 × 0.010
Radiation	Cu Kα (λ = 1.54178)
2Θ range for data collection/°	5.04 to 144.88
Index ranges	-5 ≤ h ≤ 6, -10 ≤ k ≤ 10, -21 ≤ l ≤ 21
Reflections collected	12452
Independent reflections	3319 [R_{int} = 0.0647, R_{sigma} = 0.0577]
Data/restraints/parameters	3319/1/226
Goodness-of-fit on F^2	1.050
Final R indexes [I>=2σ (I)]	R_1 = 0.0423, wR_2 = 0.1046
Final R indexes [all data]	R_1 = 0.0442, wR_2 = 0.1071
Largest diff. peak/hole / e Å^{-3}	0.40/-0.32
Flack parameter	**0.121(11)**

8. References

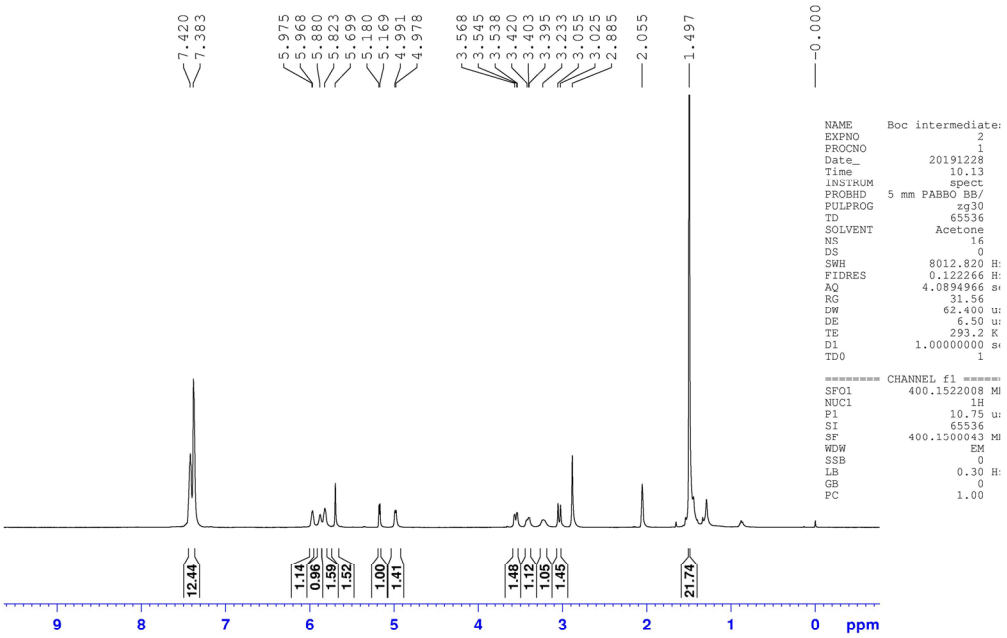
1 (a) Y. Wang, M. Mo, K. Zhu, C. Zheng, H. Zhang, W. Wang and Z. Shao, Asymmetric synthesis of *syn*-propargylamines and unsaturated beta-amino acids under Brønsted base catalysis, *Nat. Commun.*, 2015, **6**, 8544; (b) Y. Wang, S. Wang, W. Shan and Z. Shao, Direct asymmetric N-propargylation of indoles and carbazoles catalyzed by lithium SPINOL phosphate, *Nat. Commun.*, 2020, **11**, 226.

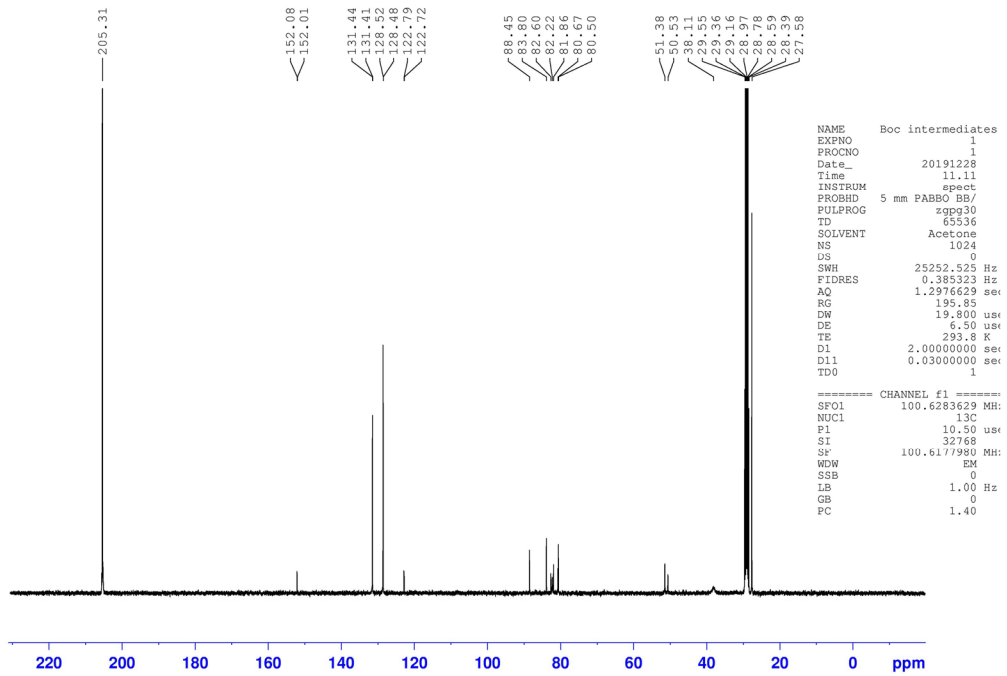
2 X. Meng, B. Yang, L. Zhang, G. Pan, X. Zhang and Z. Shao, Rh(II)/Brønsted Acid Catalyzed General and Highly Diastereo- and Enantioselective Propargylation of *in Situ* Generated Oxonium Ylides and C-Alkynyl N-Boc N,O-Acetals: Synthesis of Polyfunctional Propargylamines, *Org. Lett.*, 2019, **21**, 1292.

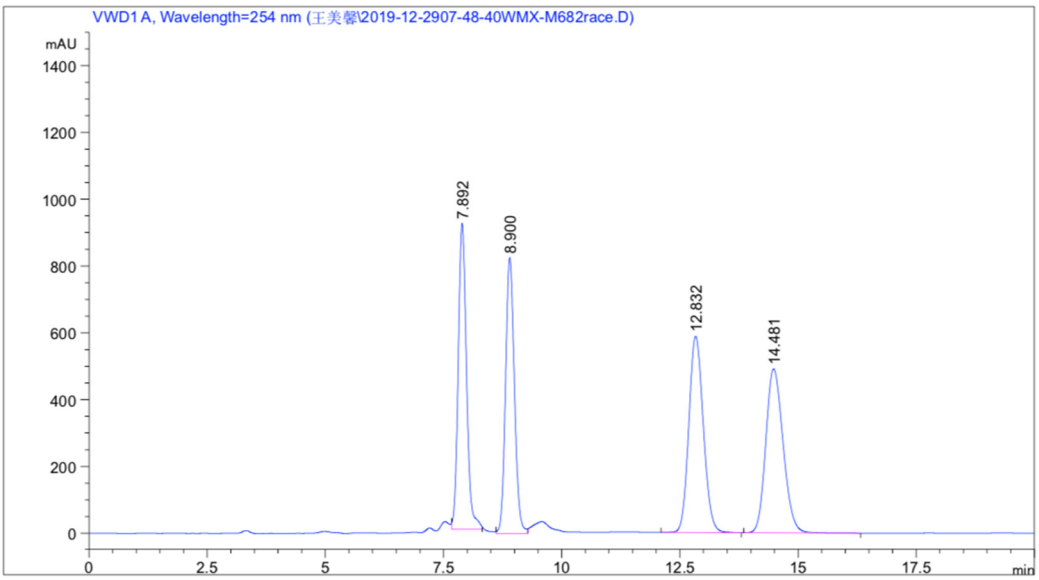
9. Copies of NMR and HPLC Spectrums



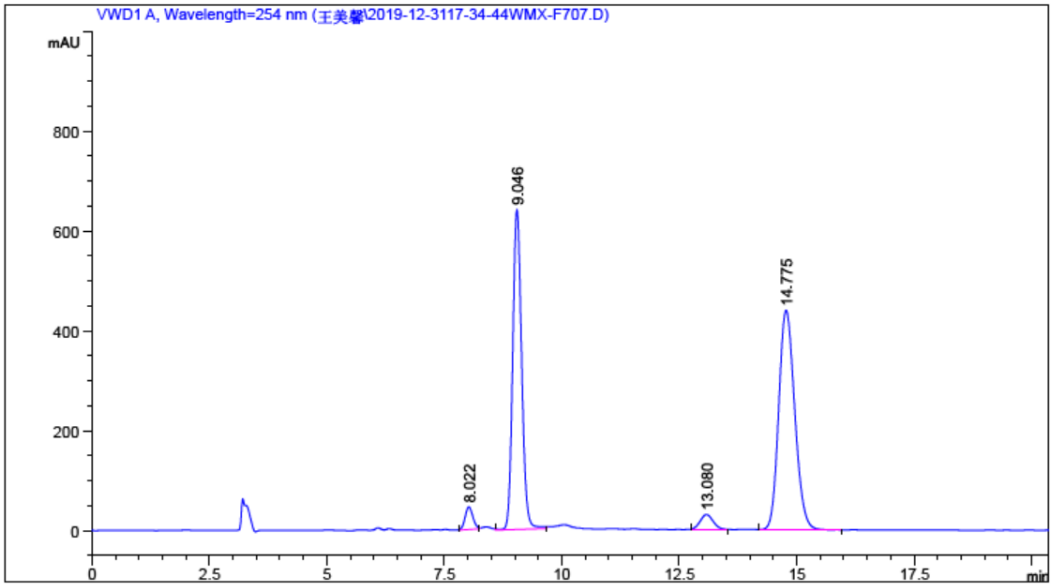
3a



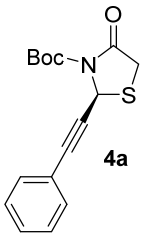




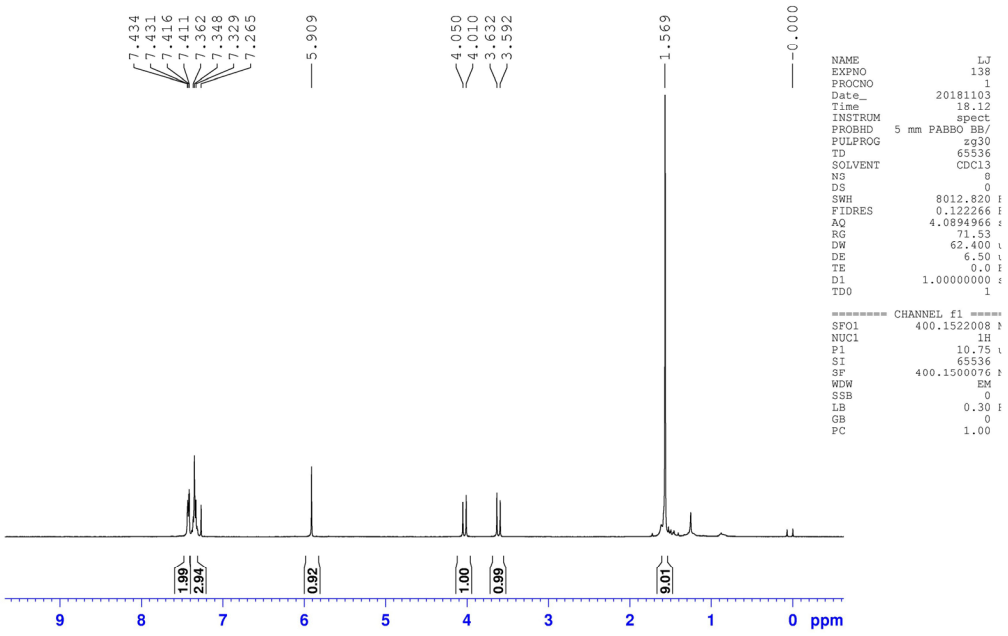
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	7.892	FM	0.2027	1.11349e4	915.63257	23.7302
2	8.900	MF	0.2176	1.07782e4	825.61517	22.9700
3	12.832	BB	0.3326	1.25215e4	587.70587	26.6852
4	14.481	BB	0.3958	1.24883e4	490.83417	26.6146



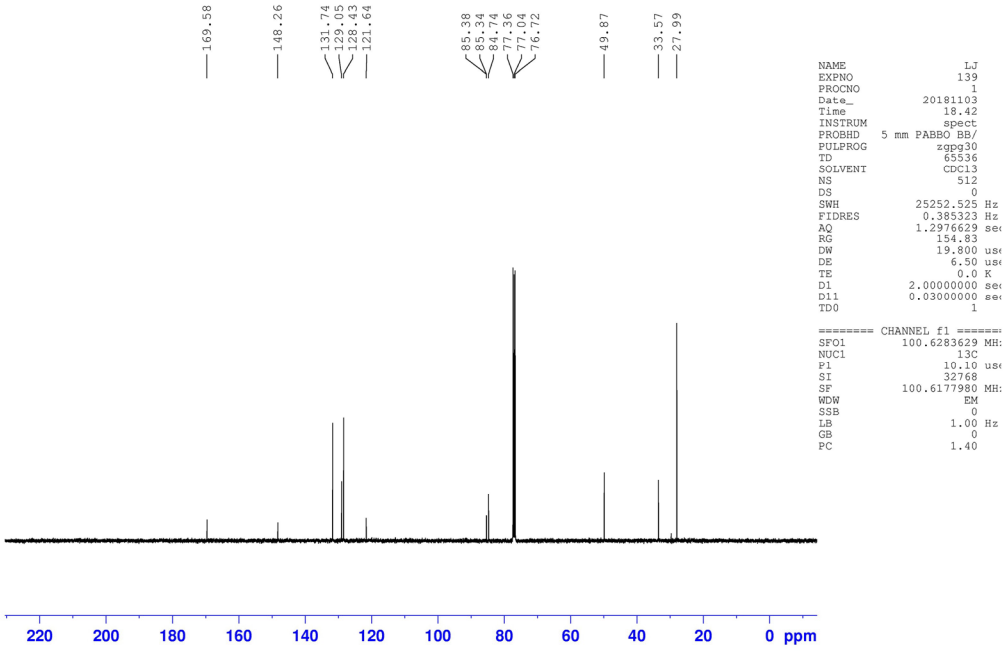
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	8.022	MM	0.1750	438.60156	41.76659	2.2052
2	9.046	MM	0.2202	8460.81641	640.38739	42.5393
3	13.080	MM	0.3121	551.65039	29.45602	2.7736
4	14.775	MM	0.3955	1.04383e4	439.83322	52.4819



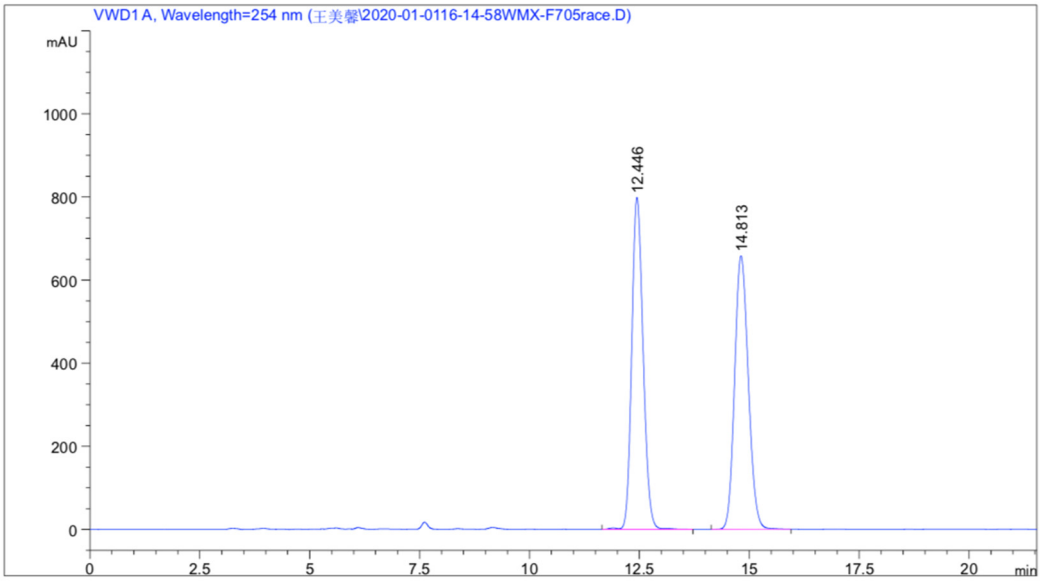
4a



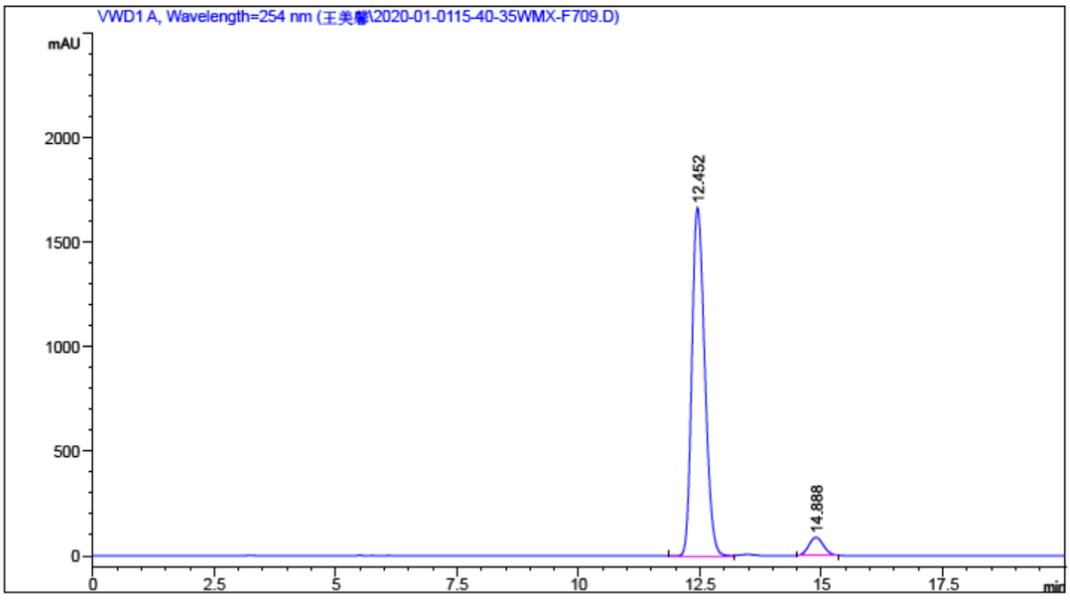

```
NAME                  LJ
EXPNO                138
PROCNO                 1
Date_          20181103
Time               18.12
INSTRUM            spect
PROBHD  5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 H
FIDRES          0.122266 H
AQ              4.0894966 s
RG                 71.53
DW                 62.400 u
DE                  6.50 u
TE                   0.0 K
D1              1.00000000 s
TD0                    1

======== CHANNEL f1 =====
SFO1        400.1522008 M
NUC1                1H
P1                10.75 u
SI                65536
SF          400.1500076 M
WDW                  EM
SSB                   0
LB                 0.30 H
GB                    0
PC                 1.00
```



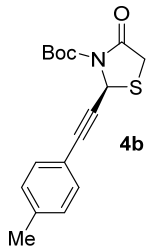

```
NAME                  LJ
EXPNO                139
PROCNO                 1
Date_          20181103
Time               18.42
INSTRUM            spect
PROBHD  5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH          25252.525 Hz
FIDRES         0.385323 Hz
AQ             1.2976629 se
RG                154.83
DW                19.800 us
DE                 6.50 us
TE                  0.0 K
D1             2.00000000 se
D11            0.03000000 se
TD0                    1

======== CHANNEL f1 =======
SFO1       100.6283629 MH
NUC1                13C
P1                10.10 us
SI                32768
SF         100.6177980 MH
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



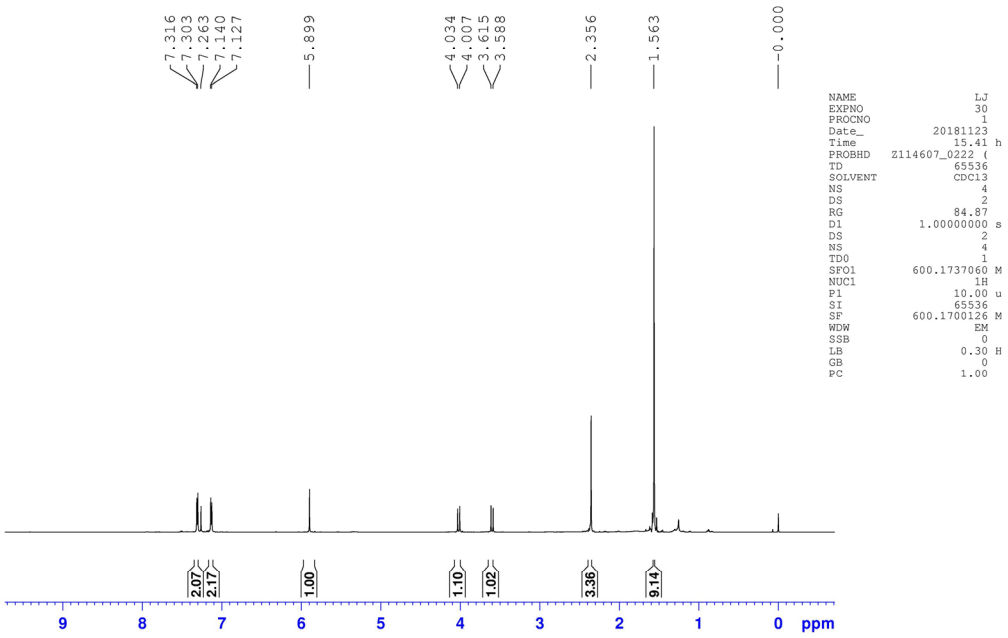
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.446	VV R	0.2774	1.43580e4	799.18805	50.1694
2	14.813	BB	0.3387	1.42610e4	658.31189	49.8306



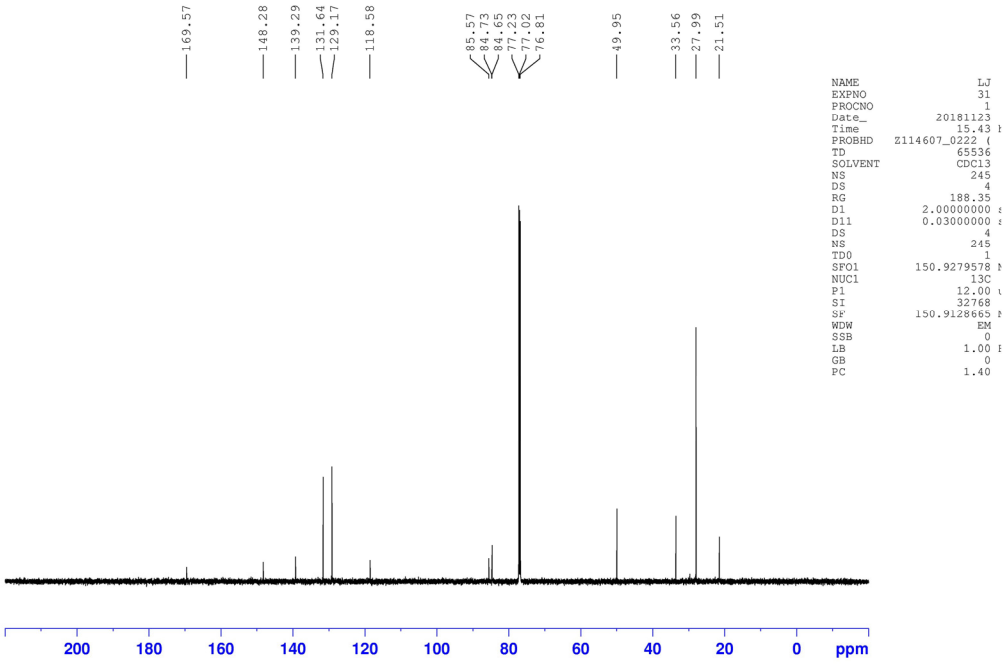
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.452	MM	0.3071	3.06646e4	1664.33569	95.1820
2	14.888	MM	0.3214	1552.20911	80.48049	4.8180

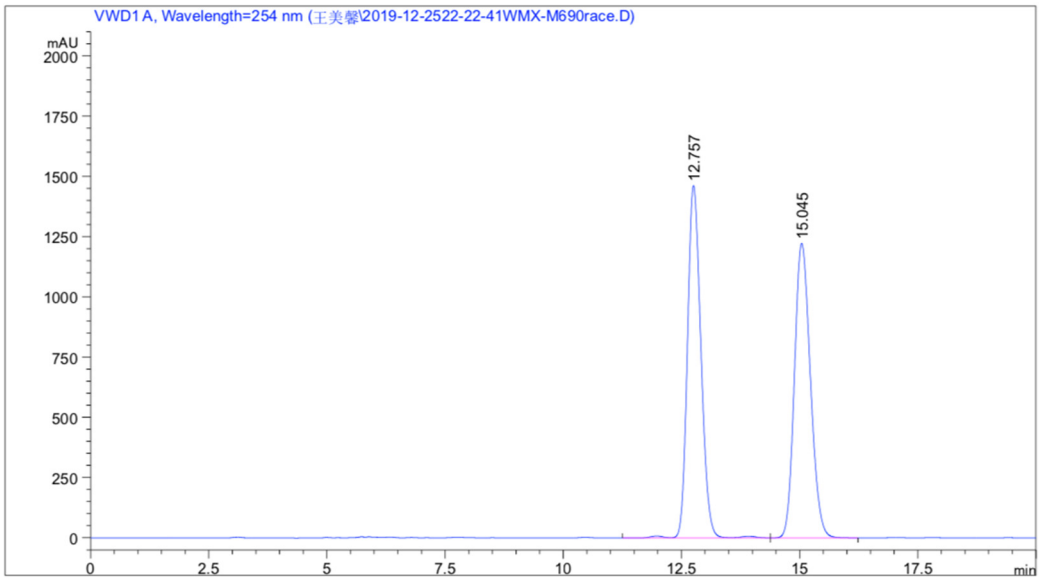


4b

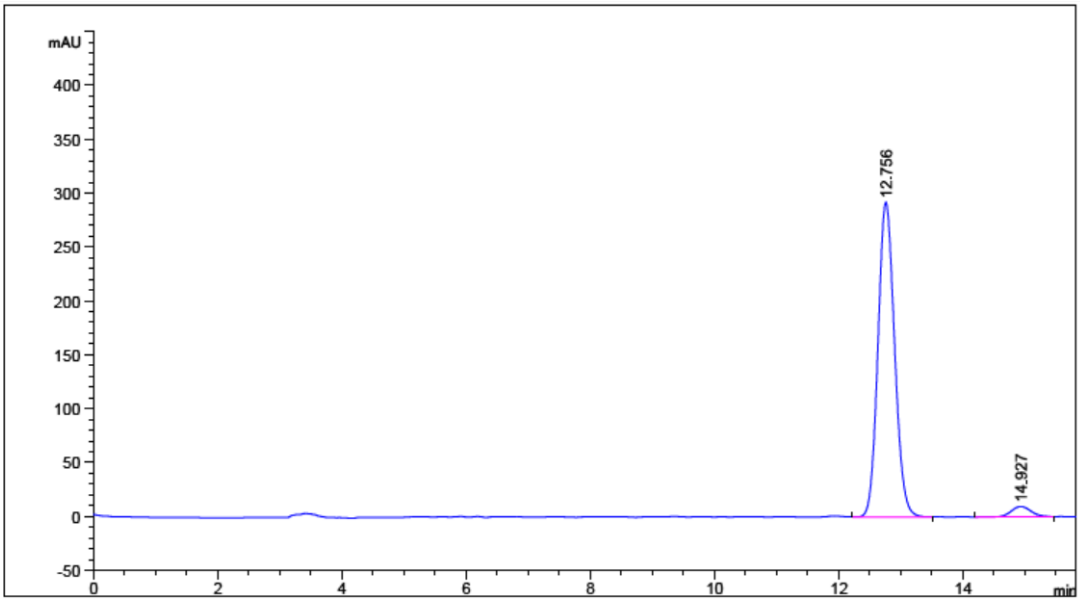



```
NAME                  LJ
EXPNO                 30
PROCNO                 1
Date_           20181123
Time               15.41 h
PROBHD    Z114607_0222 (
TD                 65536
SOLVENT            CDCl3
NS                     4
DS                     2
RG                 84.87
D1           1.00000000 s
DS                     2
NS                     4
TD0                    1
SFO1       600.1737060 M
NUC1                  1H
P1                10.00 u
SI                 65536
SF         600.1700126 M
WDW                   EM
SSB                    0
LB                 0.30 H
GB                     0
PC                  1.00
```

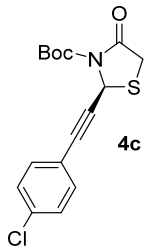

169.57
148.28
139.29
131.64
129.17
118.58
85.57
84.73
84.65
77.23
77.02
76.81
49.95
33.56
27.99
21.51
NAME LJ
EXPNO 31
PROCNO 1
Date_ 20181123
Time 15.43
PROBHD Z114607_0222
TD 65536
SOLVENT CDC13
NS 245
DS 4
RG 188.35
D1 2.00000000
D11 0.03000000
DS 4
NS 245
TD0 1
SFO1 150.9279578
NUC1 13C
P1 12.00
SI 32768
SF 150.9128665
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



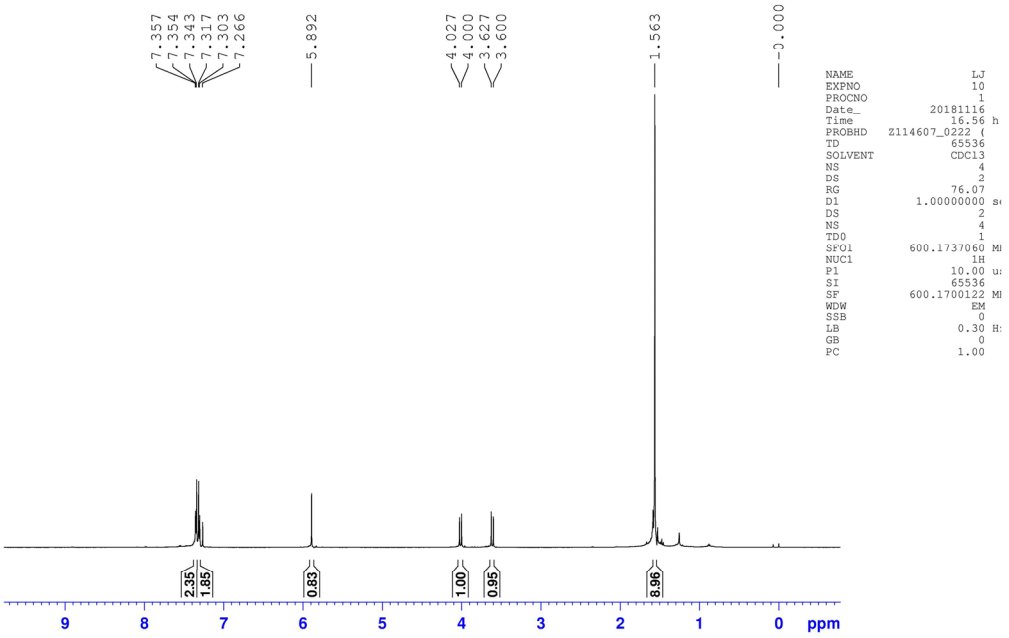
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.757	VV R	0.3037	2.89732e4	1462.34119	50.2256
2	15.045	BB	0.3640	2.87129e4	1222.58435	49.7744



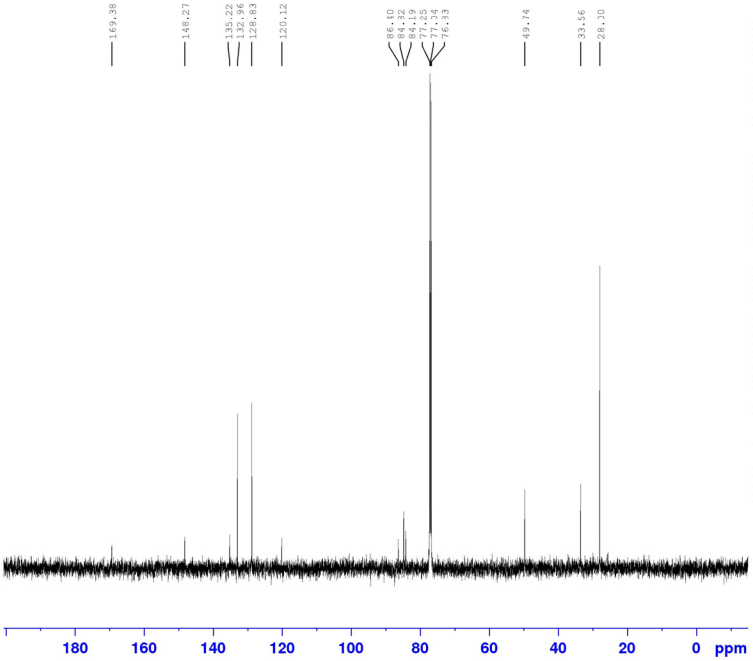
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.756	VB R	0.2890	5453.68555	291.91327	96.3107
2	14.927	BB	0.3435	208.91211	9.54073	3.6893



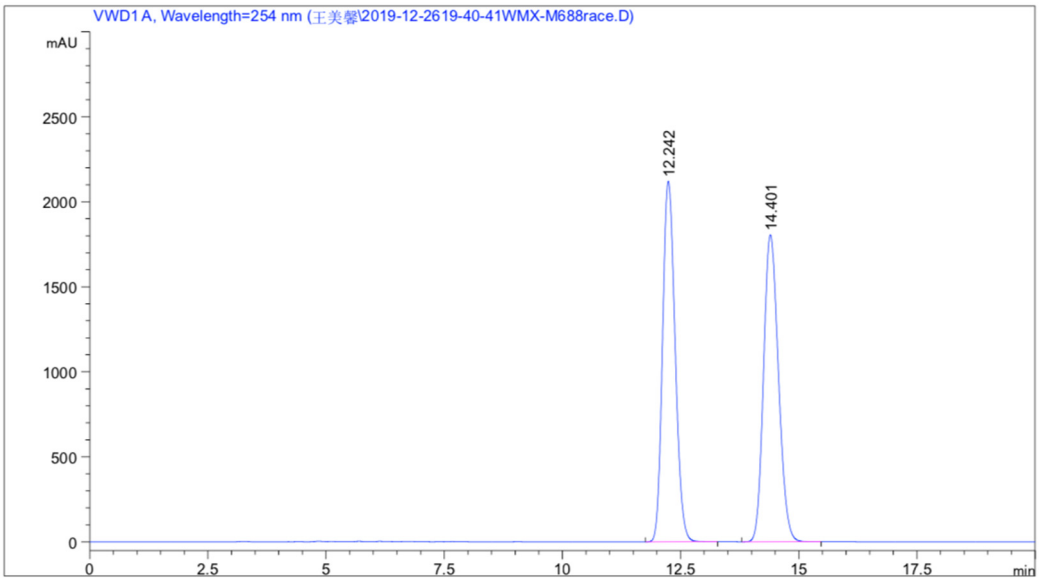
4c



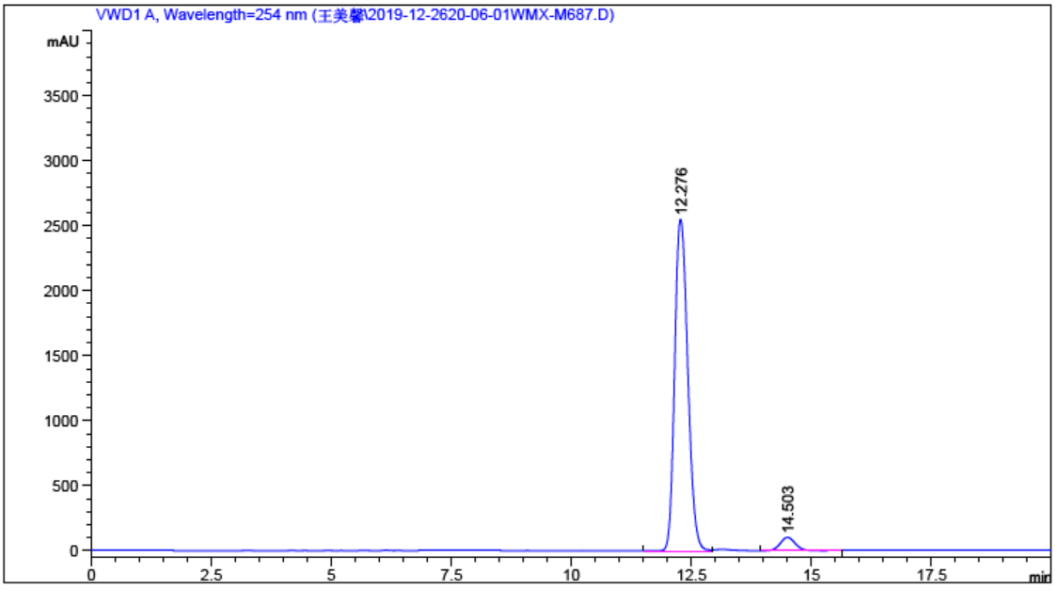

```
NAME                 LJ
EXPNO                10
PROCNO                1
Date_          20181116
Time              16.56 h
PROBHD    Z114607_0222 (
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    2
RG                76.07
D1          1.00000000 s
DS                    2
NS                    4
TD0                   1
SFO1        600.1737060 MH
NUC1                  1H
P1                10.00 u
SI                65536
SF          600.1700122 MH
WDW                  EM
SSB                   0
LB                 0.30 H
GB                    0
PC                 1.00
```



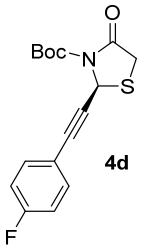

```
Current Data Parameters
NAME      Nov16-2018-liujuan
EXPNO                     11
PROCNO                     1

F2 - Acquisition Parameters
Date_           20181116
Time                16.58 h
PROBHD    Z114607_0222 (
TD                  65536
SOLVENT             CDCl3
NS                     37
DS                      4
RG                 188.35
D1           2.00000000 sec
D11          0.03000000 sec
DS                      4
NS                     37
TD0                     1
SFO1         150.9279578 MHz
NUC1                  13C
P1                  12.00 usec
PLW1         97.67099762 W
SFO2         600.1724007 MHz
NUC2                  1H
CPDPRG[2          waltz16
PCPD2               80.00 usec
PLW2         26.09399986 W
PLW12         0.40399119 W
PLW13         0.20248041 W

F2 - Processing parameters
SI                  32768
SF           150.9128665 MHz
WDW                    EM
SSB       0
LB                   2.00 Hz
GB        0
PC                   1.40
```



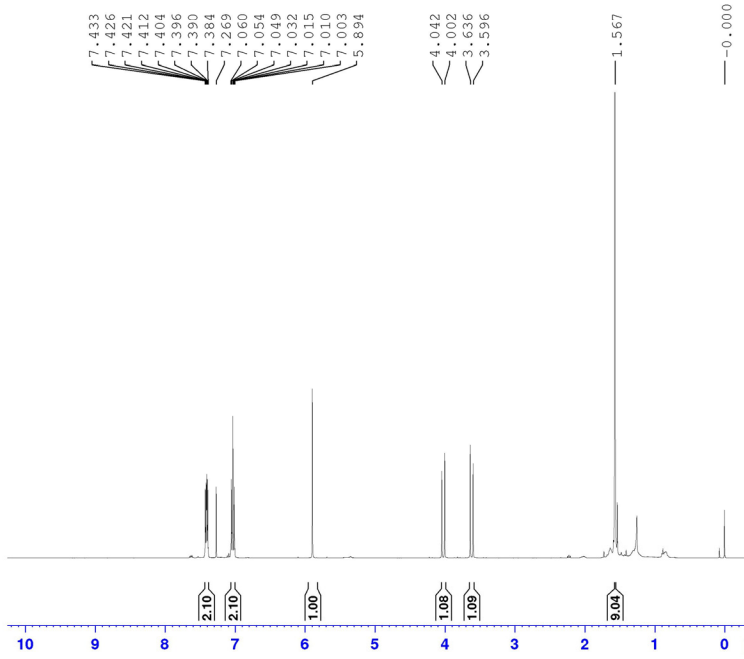
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.242	BB	0.2886	3.91841e4	2120.74512	49.7666
2	14.401	BB	0.3426	3.95516e4	1805.54846	50.2334



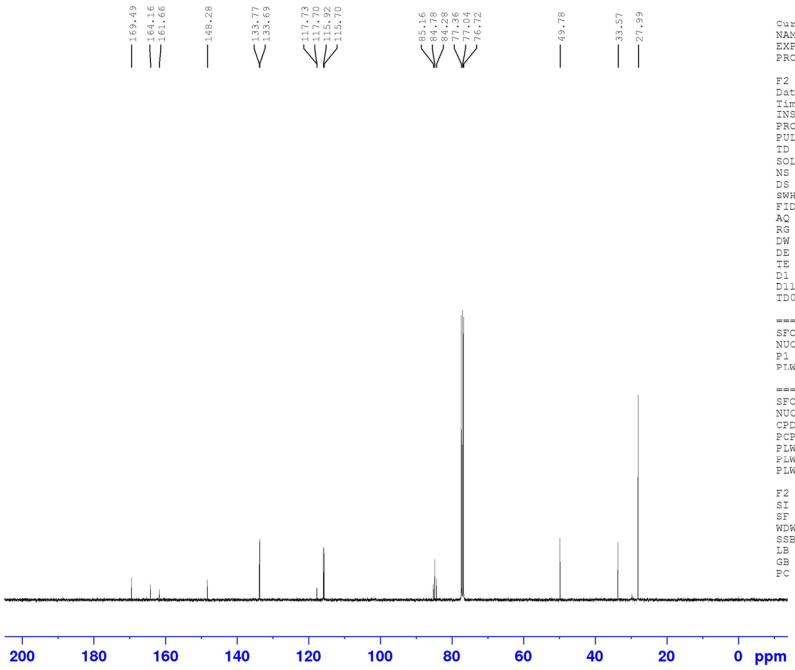
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.276	VV R	0.2963	4.83841e4	2549.40576	95.6356
2	14.503	BB	0.3350	2208.06079	101.80534	4.3644



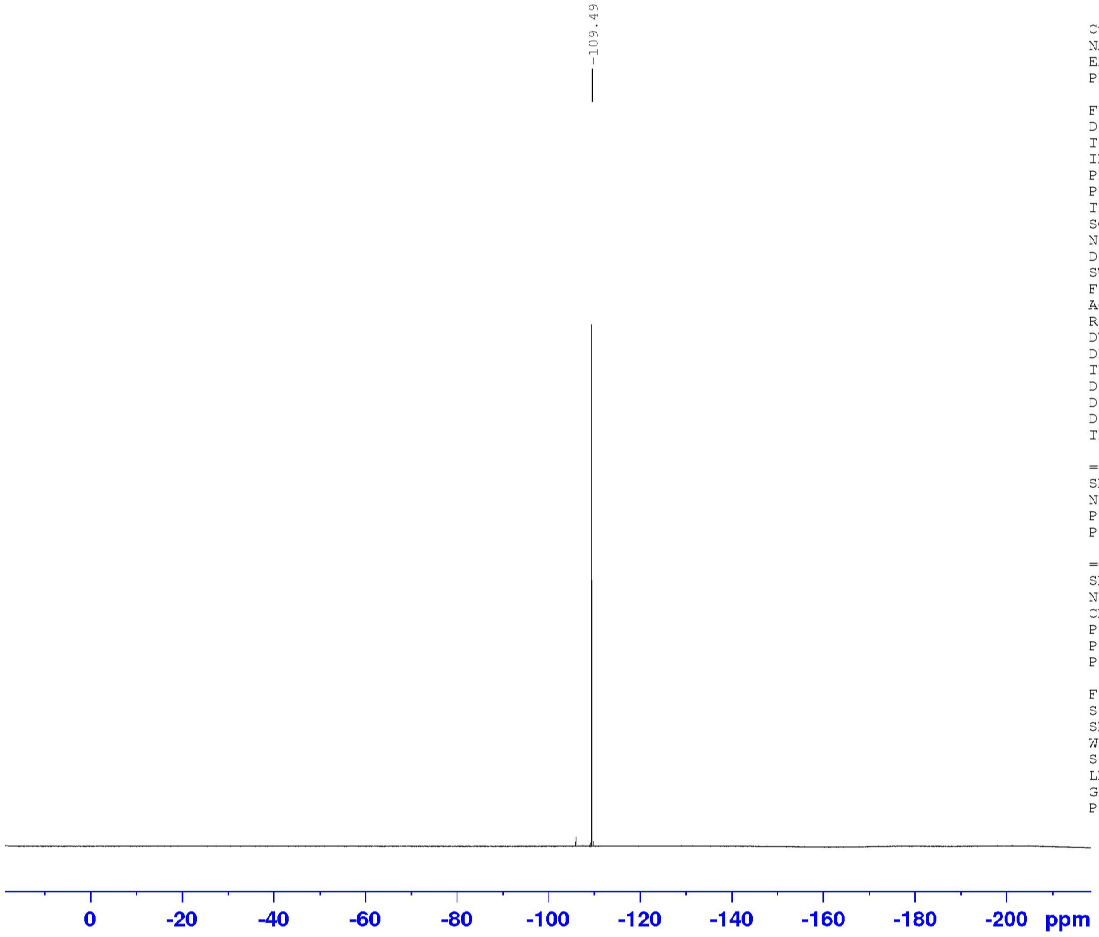
4d



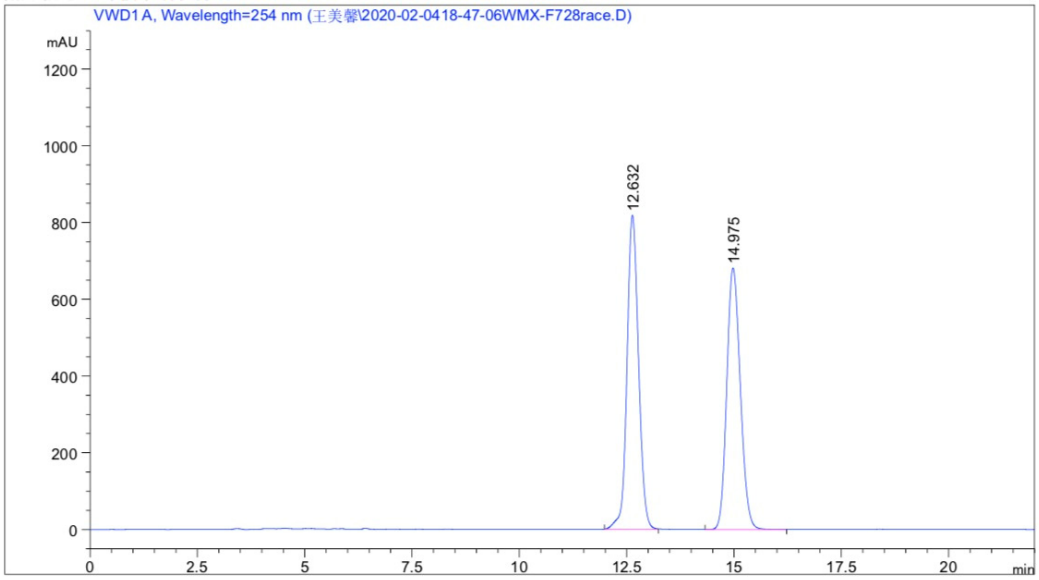

```
Current Data Parameters
NAME                  2020.2.15
EXPNO                       341
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200214
Time                      23.38
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                   8012.820 Hz
FIDRES                0.122266 Hz
AQ                    4.0894465 sec
RG                        78.11
DW                       62.400 usec
DE                        6.50 usec
TE                       292.2 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                       10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                       65536
SF                  400.1500063 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```



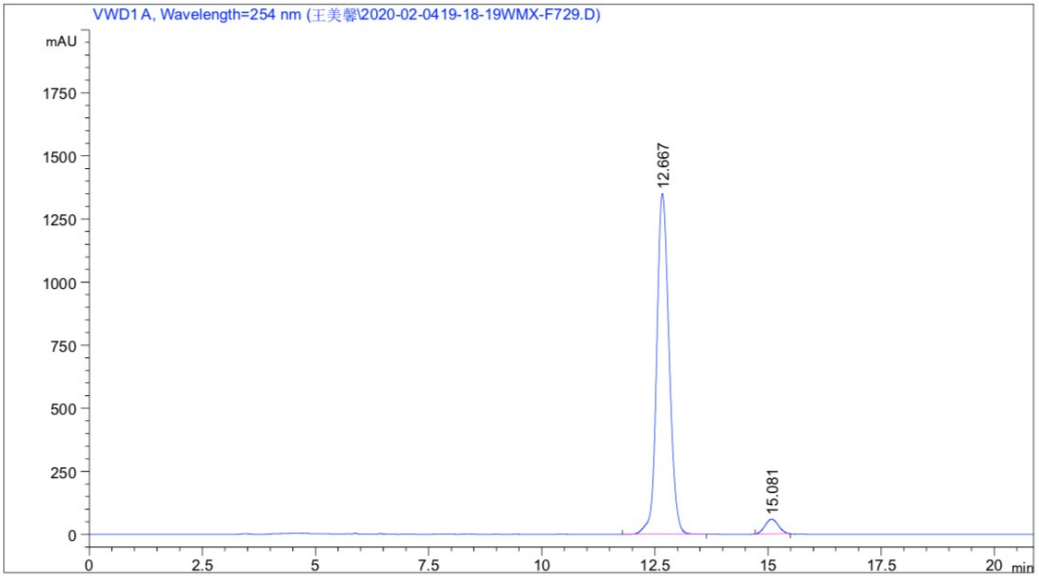

```
Current Data Parameters
NAME                  2020.2.15
EXPNO                       342
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200215
Time                      0.08
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         512
DS                           0
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976128 sec
RG                       195.85
DW                       19.800 usec
DE                        6.50 usec
TE                       292.7 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                       10.50 usec
PLW1                74.00000000 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W
PLW13                0.13149001 W

F2 - Processing parameters
SI                       32768
SF                  100.6177980 MHz
WDW                         EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```



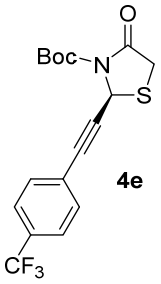

```
Current Data Parameters
NAME            2020.2.15
EXPNO                 344
PROCNO                  1

F2 - Acquisition Parameters
Date_           20200215
Time                 0.26
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG       zgfhigqn.2
TD                 131072
SOLVENT              CDCl3
NS                      16
DS                       4
SWH             89285.711 Hz
FIDRES           0.681196 Hz
AQ               0.7340032 sec
RG                  195.85
DW                   5.600 usec
DE                   6.50 usec
TE                   292.4 K
D1              1.00000000 sec
D11             0.03000000 sec
D12             0.00002000 sec
TD0                     1

======== CHANNEL f1 ========
SFO1          376.4795333 MHz
NUC1                   19F
P1                   14.00 usec
PLW1          24.85000038 W

======== CHANNEL f2 ========
SFO2          400.1516006 MHz
NUC2                   1H
CPDPRG[2           waltz16
PCPD2                90.00 usec
PLW2          17.50000000 W
PLW12          0.26142001 W

F2 - Processing parameters
SI                    65536
SF            376.5171850 MHz
WDW                     EM
SSB       0
LB                    0.30 Hz
GB        0
PC                    1.00
```



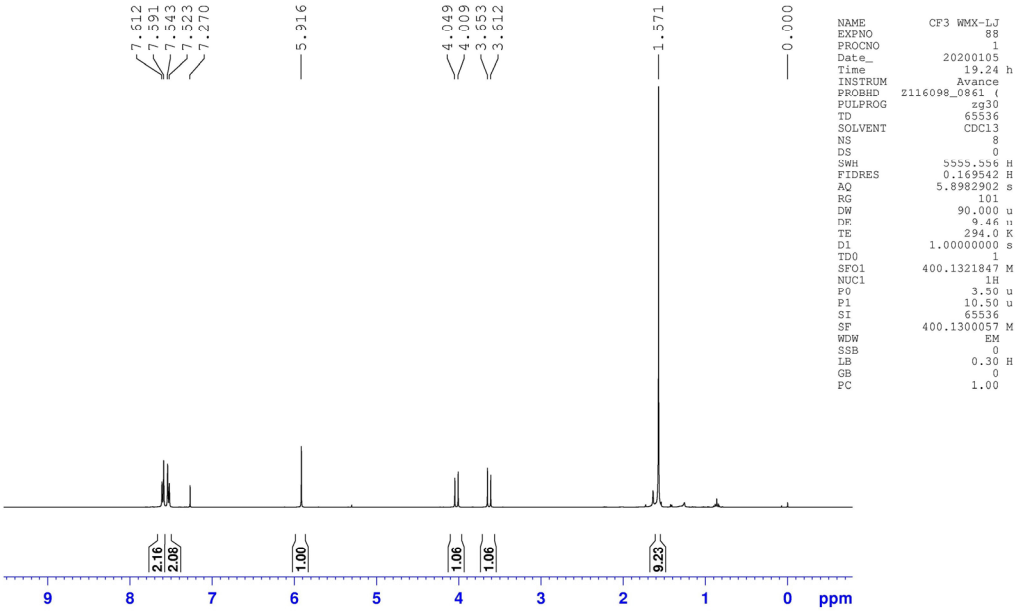
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.632	MM	0.3106	1.52335e4	817.50391	50.3751
2	14.975	MM	0.3668	1.50066e4	681.87213	49.6249



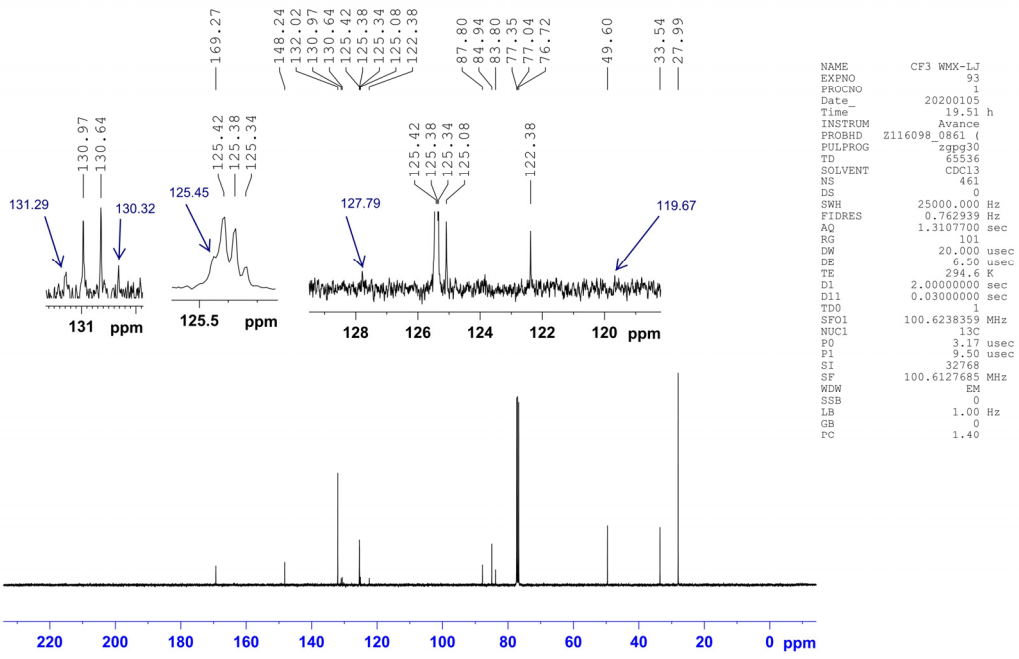
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.667	MM	0.3174	2.57169e4	1350.46411	95.5278
2	15.081	MM	0.3422	1203.94409	58.63163	4.4722



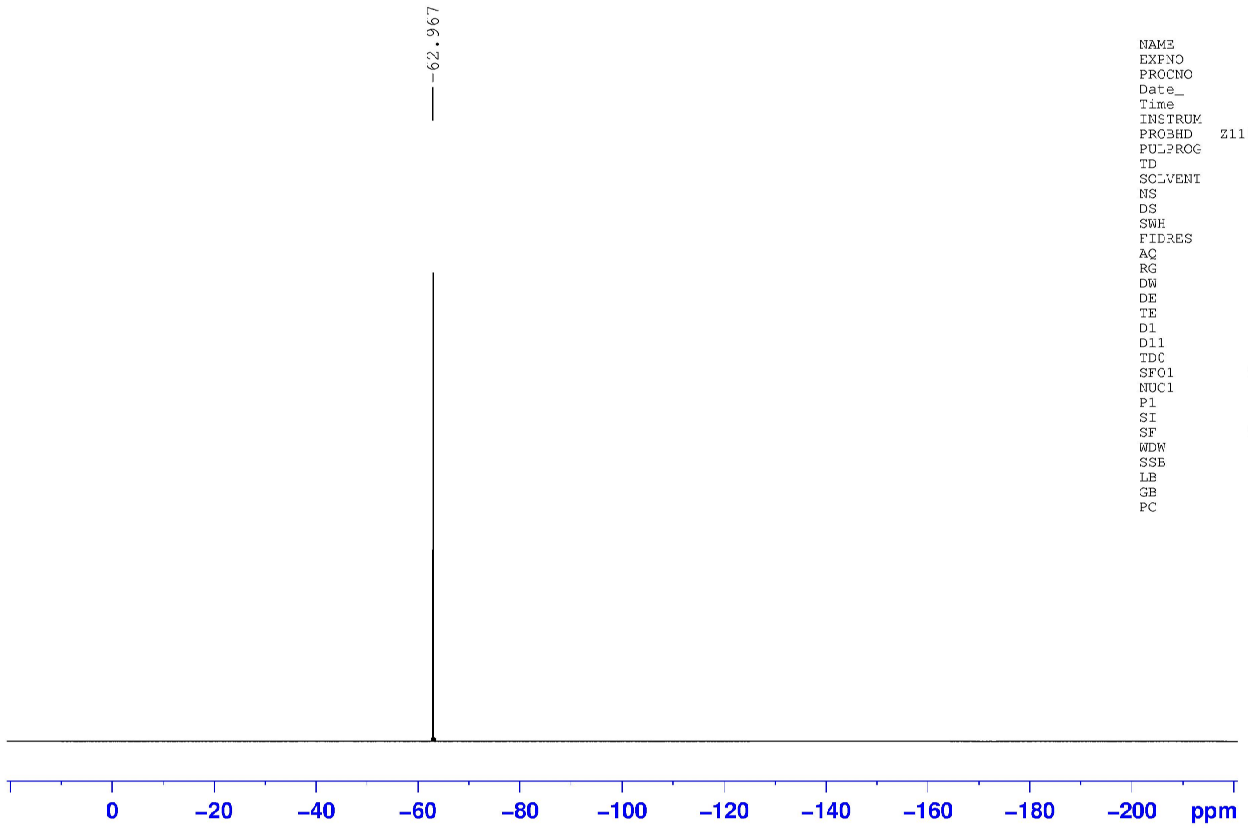
4e



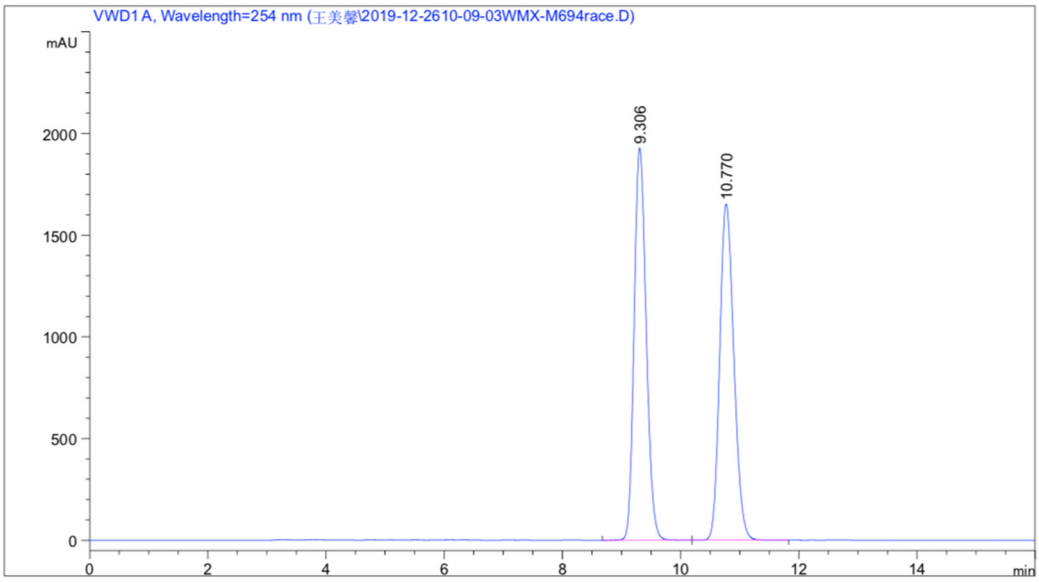

```
NAME          CF3 WMX-LJ
EXPNO             88
PROCNO             1
Date_          20200105
Time           19.24 h
INSTRUM         Avance
PROBHD    Z116098_0861 (
PULPROG         zg30
TD             65536
SOLVENT        CDCl3
NS                 8
DS                 0
SWH          5555.556 H
FIDRES       0.169542 H
AQ           5.8982902 s
RG               101
DW            90.000 u
DE             9.46 u
TE           294.0 K
D1           1.00000000 s
TD0                1
SFO1       400.1321847 M
NUC1              1H
P0             3.50 u
P1            10.50 u
SI             65536
SF         400.1300057 M
WDW               EM
SSB                0
LB            0.30 H
GB                 0
PC              1.00
```



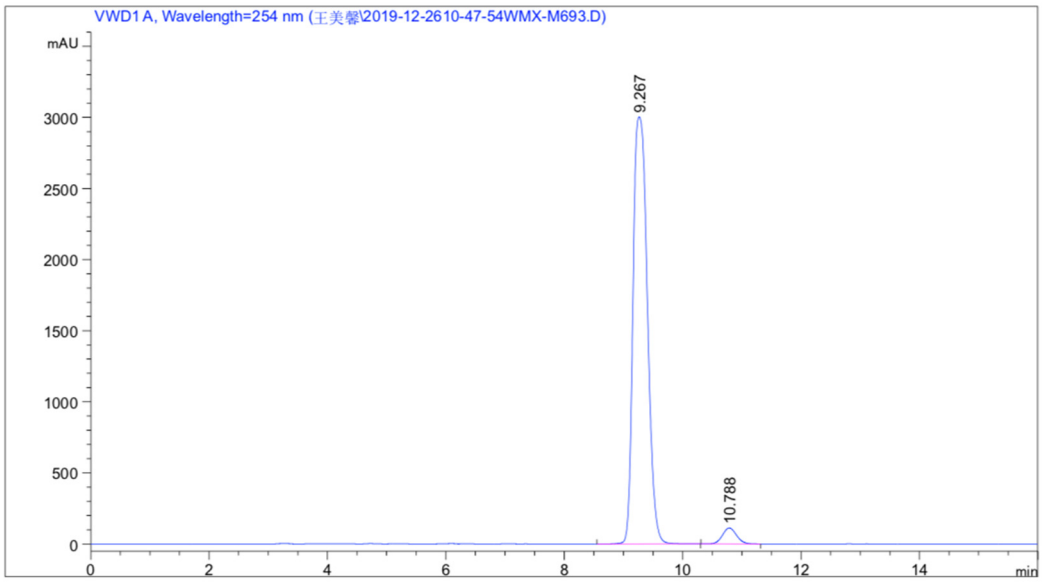

```
NAME          CF3 WMX-LJ
EXPNO             93
PROCNO             1
Date_          20200105
Time           19.51 h
INSTRUM         Avance
PROBHD    Z116098_0861 (
PULPROG        zgpg30
TD             65536
SOLVENT        CDCl3
NS               461
DS                 0
SWH          25000.000 Hz
FIDRES        0.762939 Hz
AQ            1.3107700 sec
RG               101
DW            20.000 usec
DE             6.50 usec
TE           294.6 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                1
SFO1       100.6238359 MHz
NUC1              13C
P0             3.17 usec
P1             9.50 usec
SI             32768
SF         100.6127685 MHz
WDW               EM
SSB                0
LB            1.00 Hz
GB                 0
PC              1.40
```



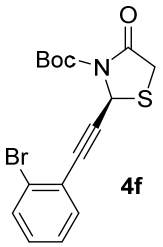
NAME	CF3 WMX-LJ
EXPNO	96
PROCNO	1
Date_	20200105
Time	19.53 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	294.5 K
D1	1.00000000 sec
D11	0.03000000 sec
TDC	1
SFO1	376.4607164 MHz
NUC1	19F
P1	18.00 usec
SI	65536
SF	376.4983662 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



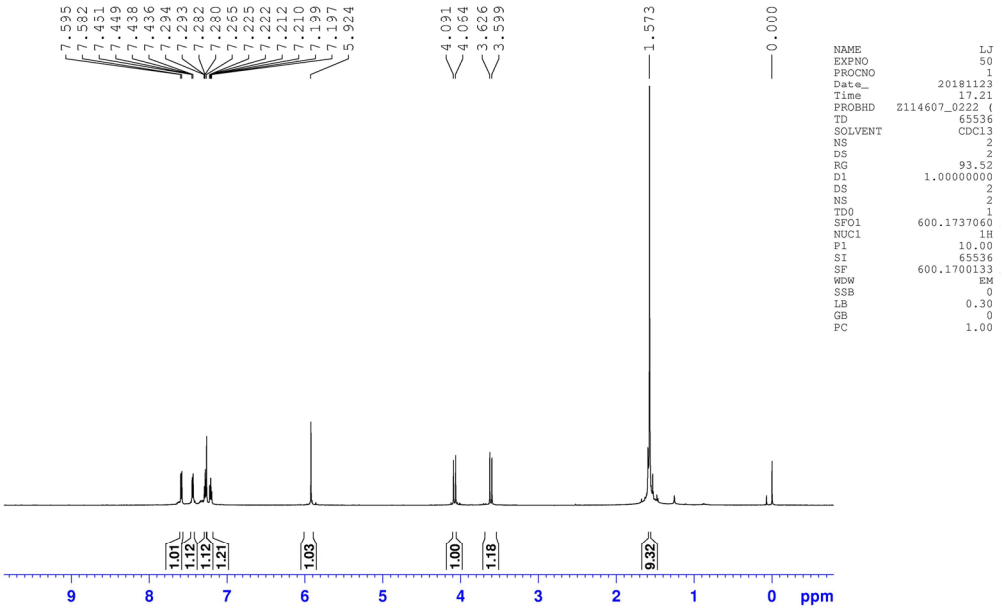
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.306	BB	0.2206	2.73713e4	1927.84119	49.8533
2	10.770	BB	0.2604	2.75325e4	1652.50415	50.1467



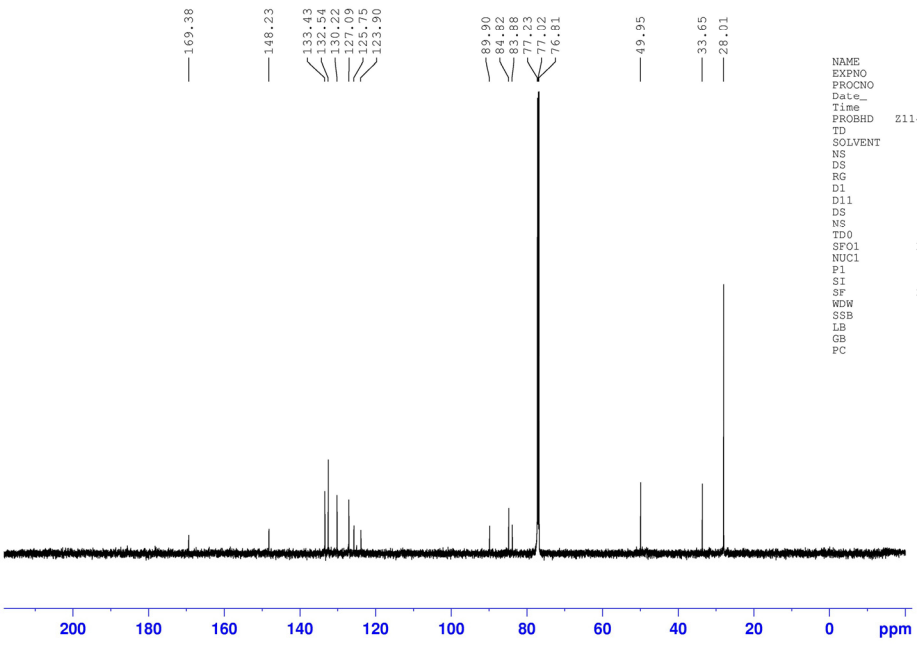
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.267	BB	0.2652	5.02570e4	3003.53687	96.4719
2	10.788	BB	0.2555	1837.96069	111.94482	3.5281



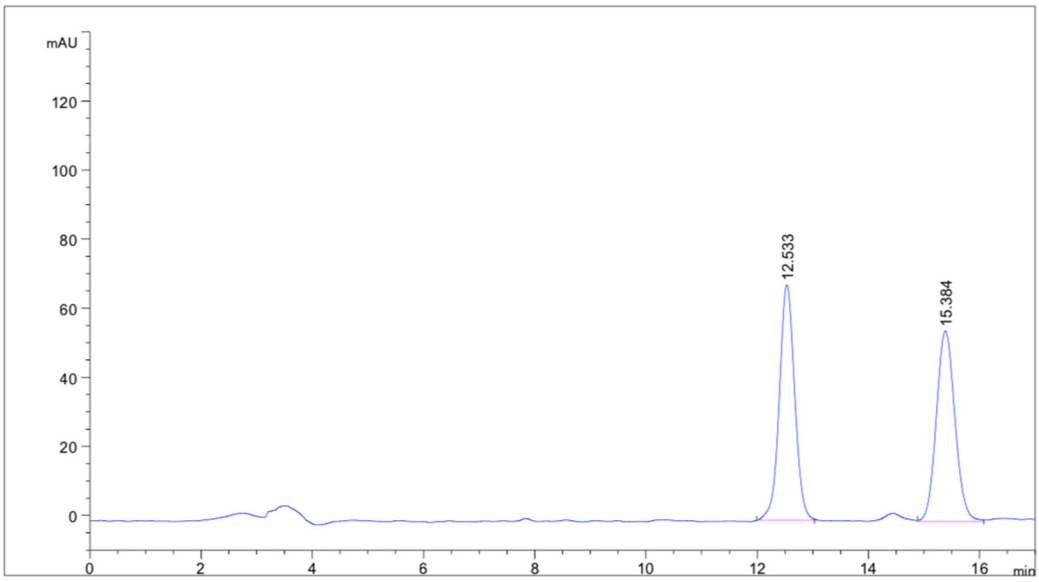
4f



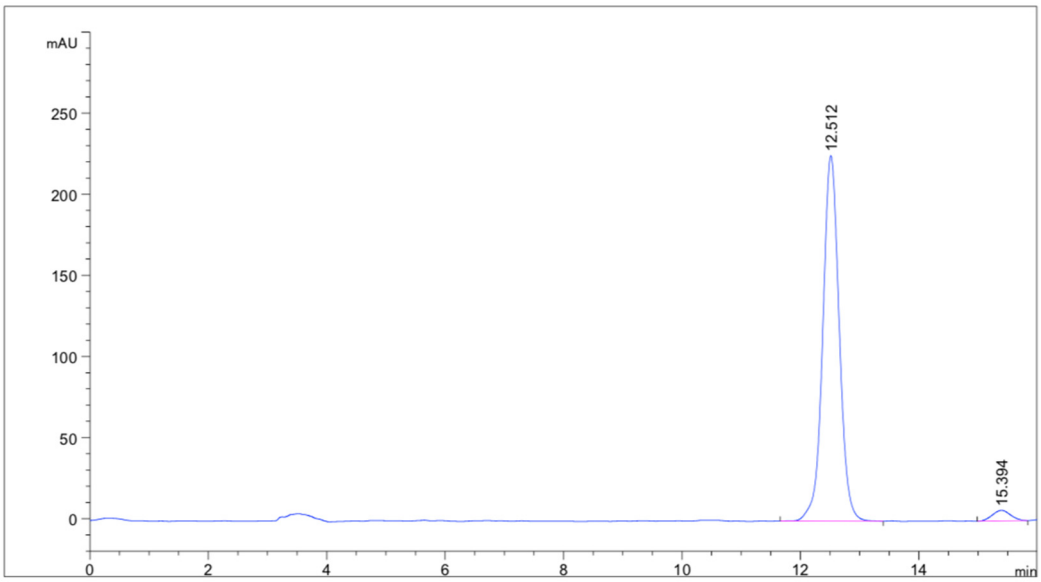
NAME	LJ
EXPNO	50
PROCNO	1
Date_	20181123
Time	17.21
PROBHD	Z114607_0222 (
TD	65536
SOLVENT	CDCl3
NS	2
DS	2
RG	93.52
D1	1.00000000
DS	2
NS	2
TD0	1
SFO1	600.1737060
NUC1	1H
P1	10.00
SI	65536
SF	600.1700133
WDW	EM
SSB	0
LB	0.30
GB	0
PC	1.00



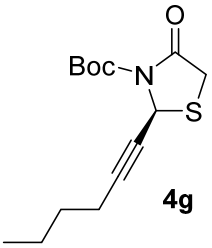
NAME	LJ
EXPNO	51
PROCNO	1
Date_	20181123
Time	17.29
PROBHD	Z114607_0222 (
TD	65536
SOLVENT	CDCl3
NS	400
DS	4
RG	188.35
D1	2.00000000
D11	0.03000000
DS	4
NS	400
TD0	1
SFO1	150.9279578
NUC1	13C
P1	12.00
SI	32768
SF	150.9128665
WDW	EM
SSB	0
LB	1.00
GB	0
PC	1.40



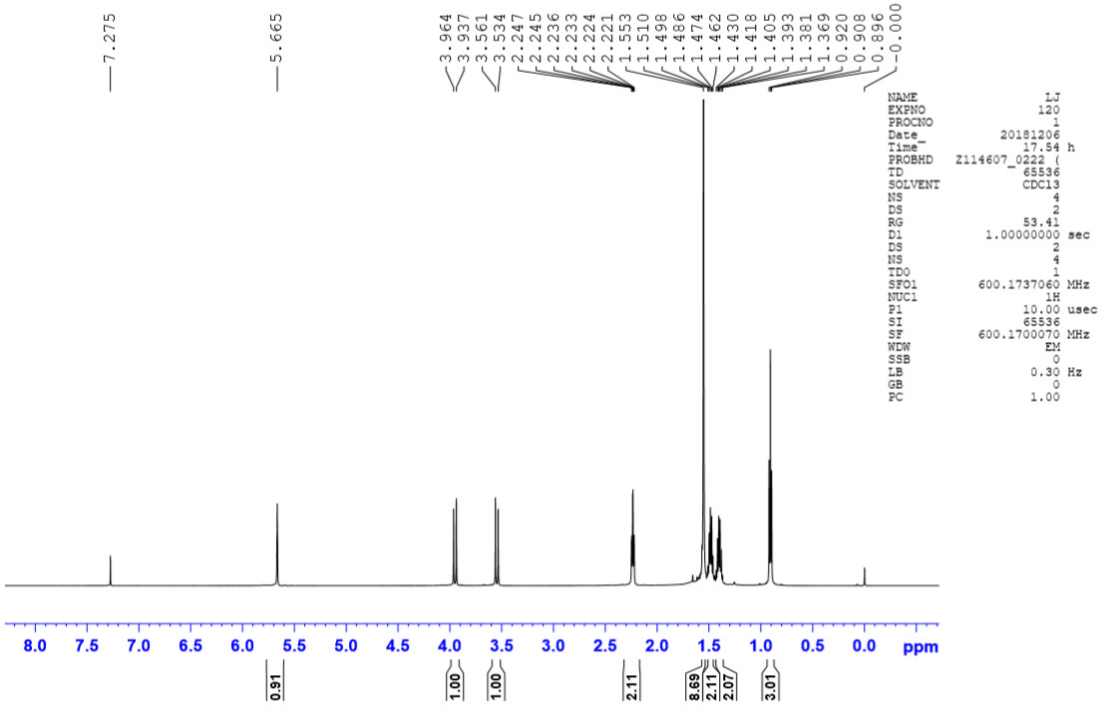
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.533	MM	0.3192	1305.25928	68.16132	50.4618
2	15.384	MM	0.3872	1281.36731	55.15733	49.5382

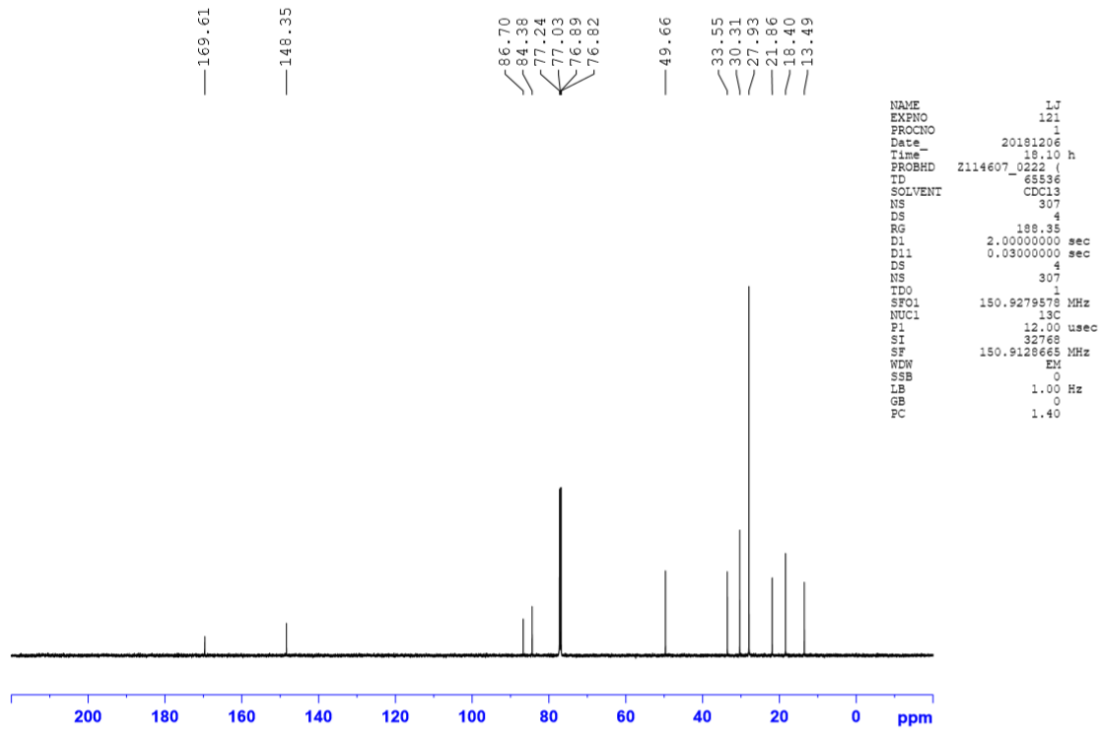


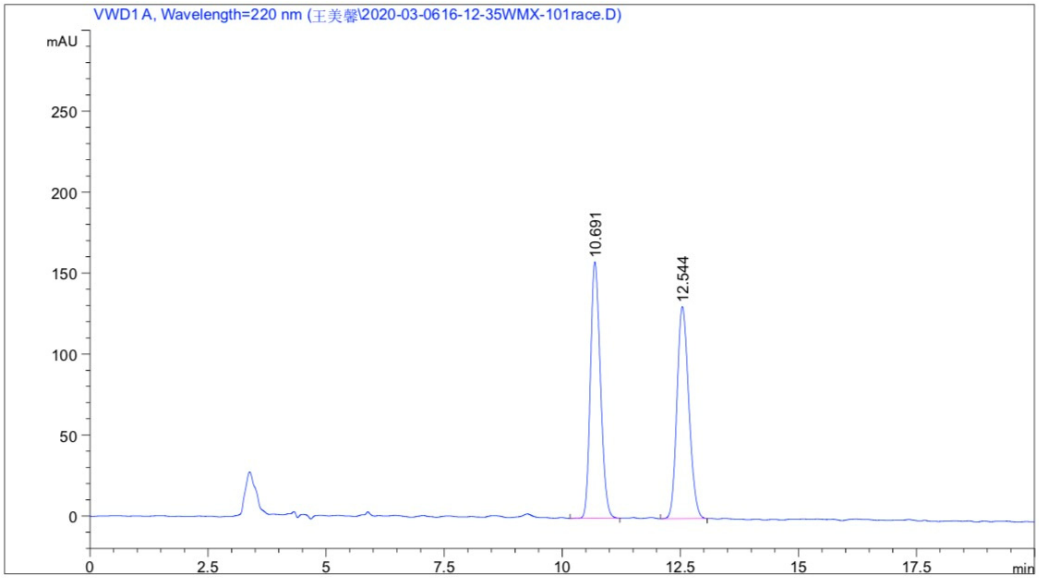
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.512	BB	0.2972	4350.56592	225.38519	96.9098
2	15.394	BB	0.3320	138.73010	6.52540	3.0902


O
Boc
N
S

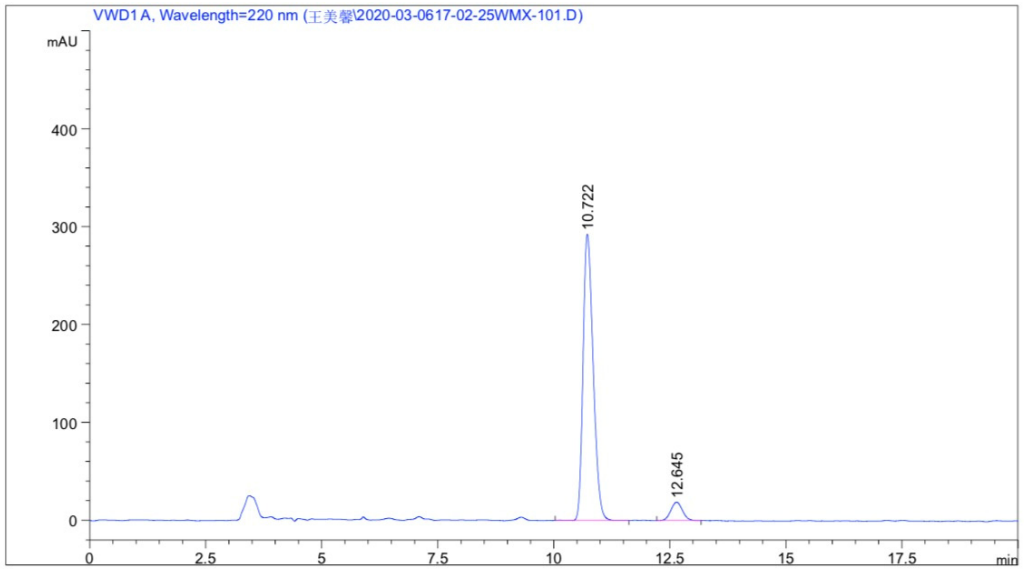
4g


7.275
5.665
3.964
3.937
3.561
3.534
2.247
2.245
2.236
2.233
2.224
2.221
1.553
1.510
1.498
1.486
1.474
1.462
1.430
1.418
1.405
1.393
1.381
1.369
0.920
0.908
0.896
0.000
NAME LJ
EXPNO 120
PROCNO 1
Date_ 20181206
Time 17.54 h
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 4
DS 2
RG 53.41
D1 1.00000000 sec
DS 2
NS 4
TD0 1
SFO1 600.1737060 MHz
NUC1 1H
P1 10.00 usec
SI 65536
SF 600.1700070 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 ppm
0.91
1.00
1.00
2.11
8.69
2.11
2.07
3.01

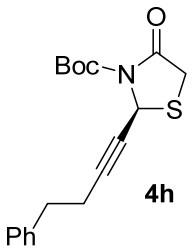

169.61
148.35
86.70
84.38
77.24
77.03
76.89
76.82
49.66
33.55
30.31
27.93
21.86
18.40
13.49
NAME LJ
EXPNO 121
PROCNO 1
Date_ 20181206
Time 18.10 h
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 307
DS 4
RG 188.35
D1 2.00000000 sec
D11 0.03000000 sec
DS 4
NS 307
TD0 1
SFO1 150.9279578 MHz
NUC1 13C
P1 12.00 usec
SI 32768
SF 150.9128665 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



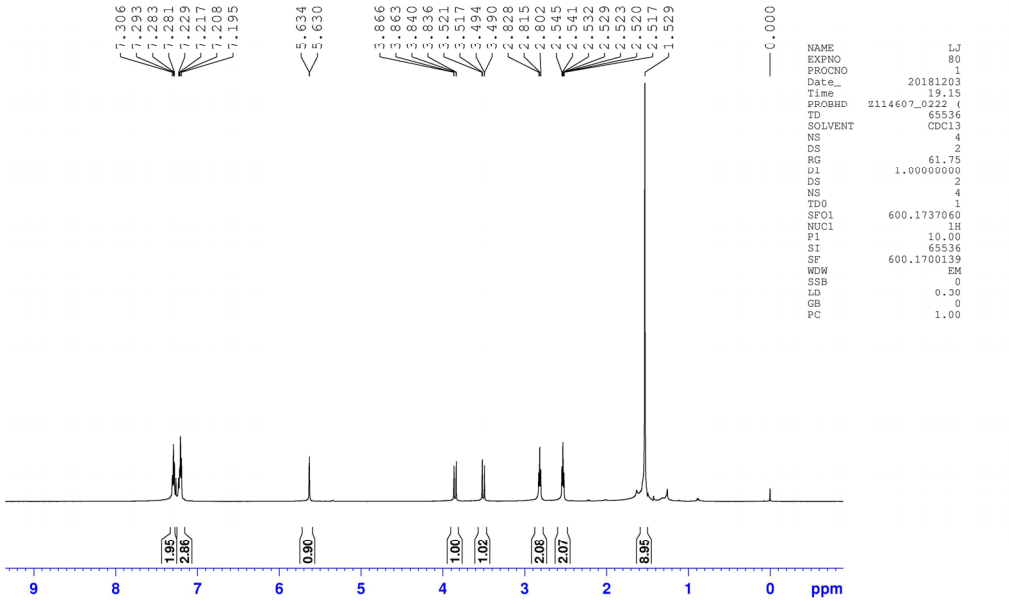
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.691	VB R	0.2287	2331.62866	158.42418	50.4440
2	12.544	BB	0.2721	2290.58643	130.92555	49.5560

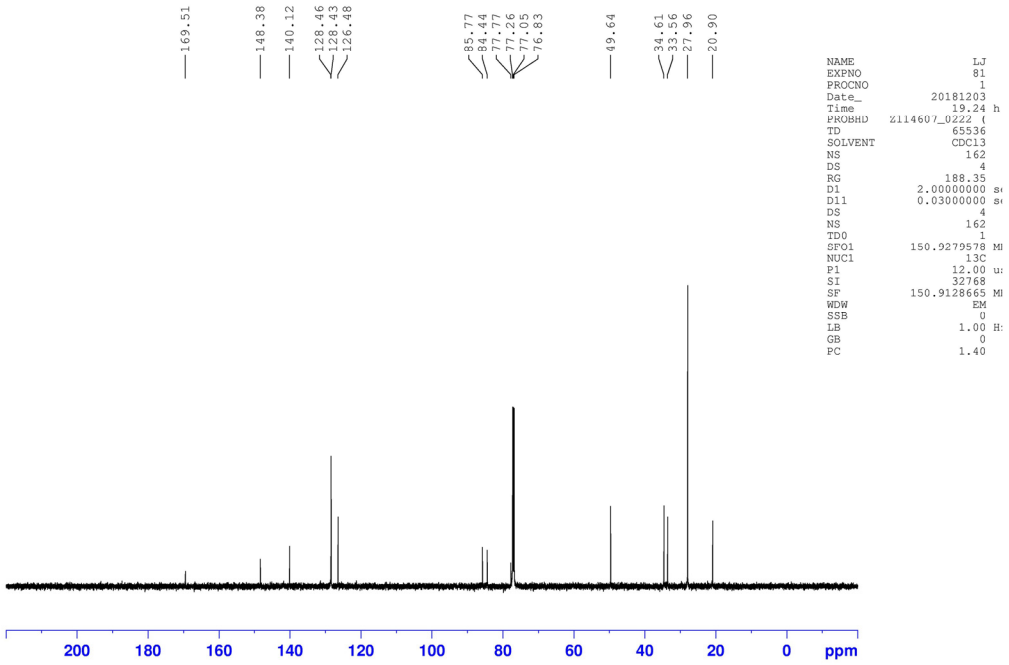


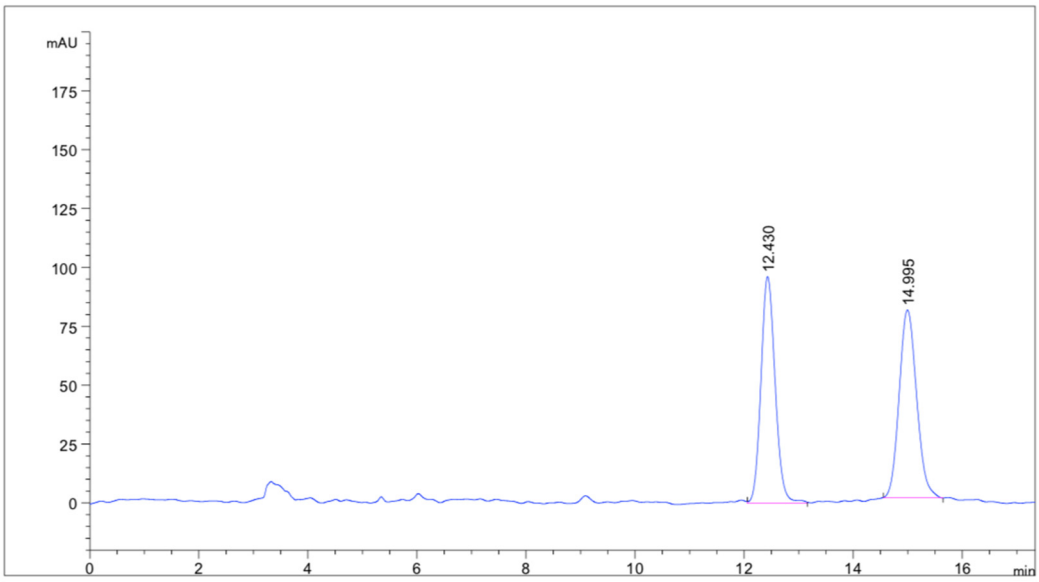
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.722	MM	0.2518	4414.31738	292.15646	93.3627
2	12.645	MM	0.2828	313.81985	18.49163	6.6373


O
Boc
N
S
Ph

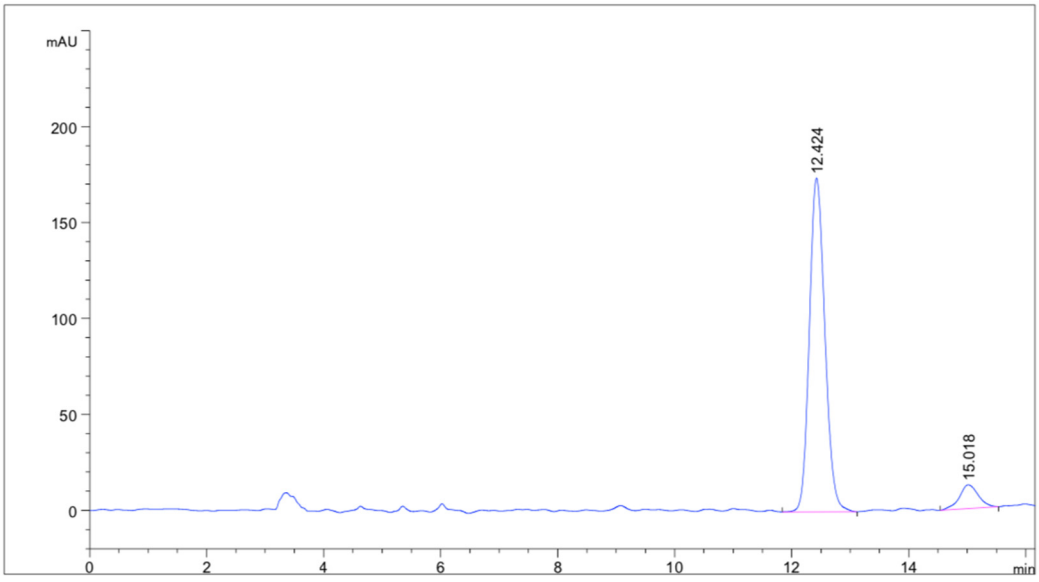
4h


7.306
7.293
7.283
7.281
7.229
7.217
7.208
7.195
5.634
5.630
3.866
3.863
3.840
3.836
3.521
3.517
3.494
3.490
2.828
2.815
2.802
2.545
2.541
2.532
2.529
2.523
2.520
2.517
1.529
0.000
NAME LJ
EXPNO 80
PROCNO 1
Date_ 20181203
Time 19.15
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 4
DS 2
RG 61.75
D1 1.00000000
DS 2
NS 4
TD0 1
SFO1 600.1737060
NUC1 1H
P1 10.00
SI 65536
SF 600.1700139
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
1.95
2.86
0.90
1.00
1.02
2.08
2.07
8.95
9
8
7
6
5
4
3
2
1
0
ppm

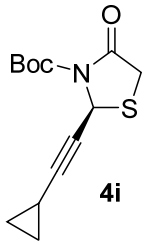

169.51
148.38
140.12
128.46
128.43
126.48
85.77
84.44
77.77
77.26
77.05
76.83
49.64
34.61
33.56
27.96
20.90
NAME LJ
EXPNO 81
PROCNO 1
Date_ 20181203
Time 19.24 h
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 162
DS 4
RG 188.35
D1 2.00000000 s
D11 0.03000000 s
DS 4
NS 162
TD0 1
SFO1 150.9279578 M
NUC1 13C
P1 12.00 u
SI 32768
SF 150.9128665 M
WDW EM
SSB 0
LB 1.00 H
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



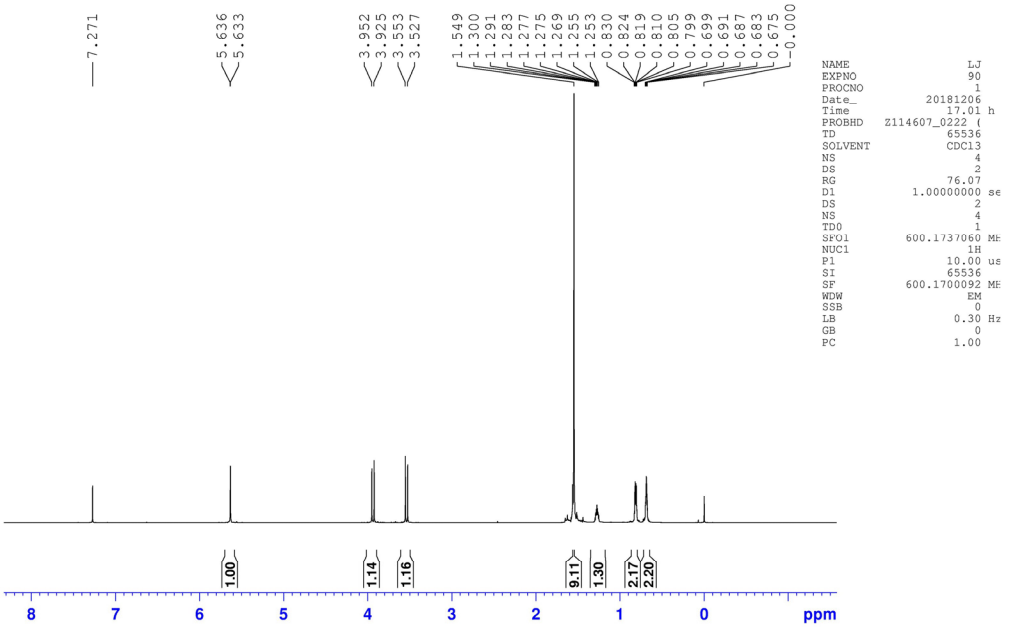
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.430	MM	0.3015	1741.25098	96.26540	49.5341
2	14.995	MM	0.3705	1774.00842	79.80426	50.4659

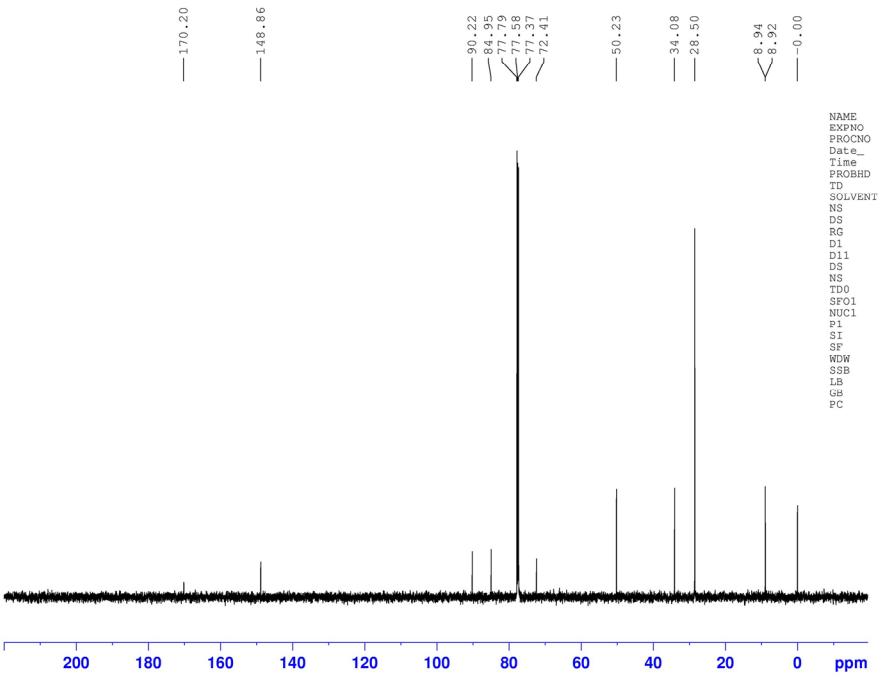


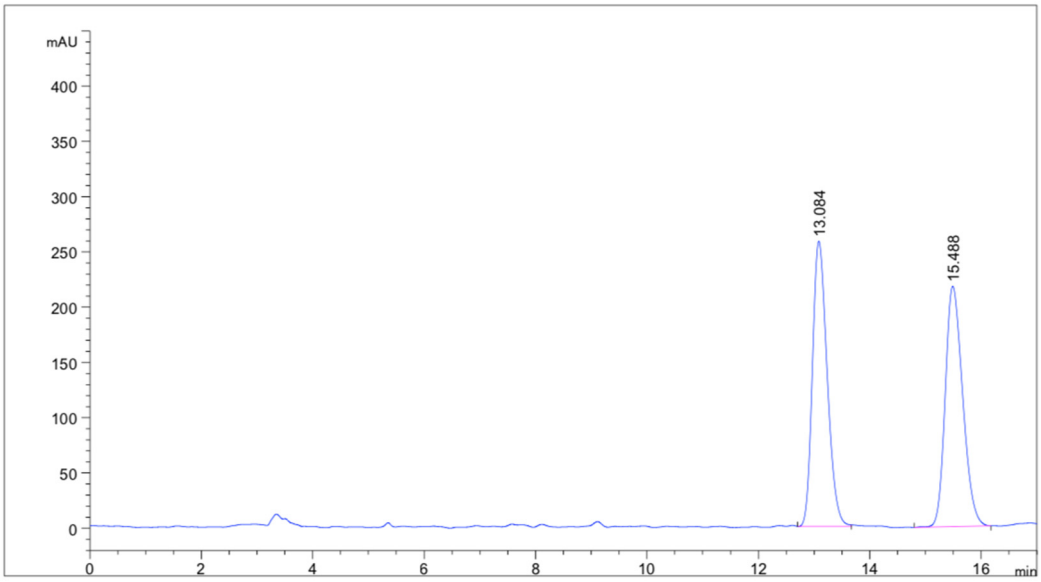
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.424	BB	0.2869	3203.03784	173.91127	91.9834
2	15.018	BB	0.3428	279.15509	12.39468	8.0166


O
Boc
N
S

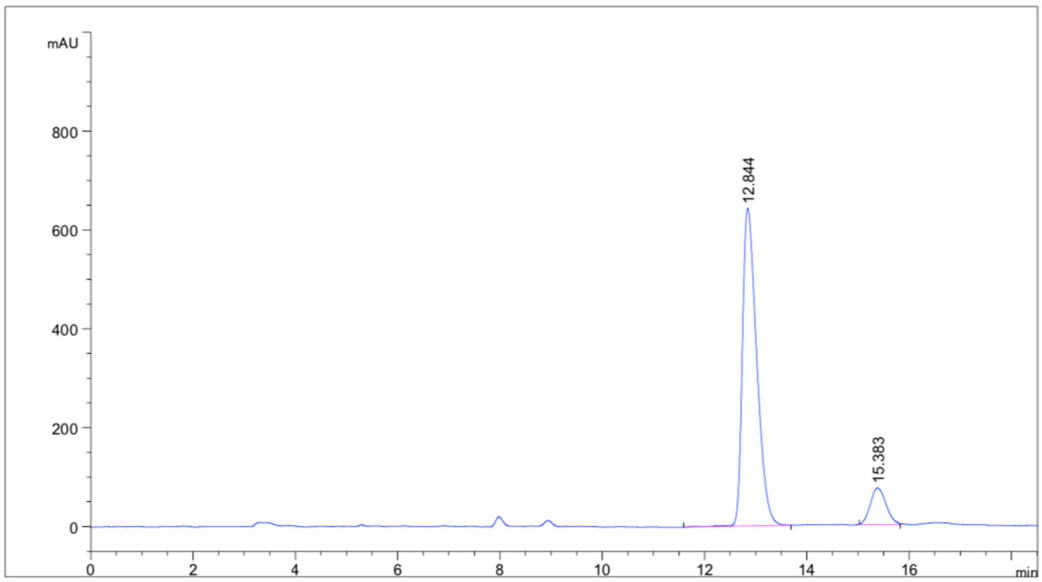
4i


7.271
5.636
5.633
3.952
3.925
3.553
3.527
1.549
1.300
1.291
1.283
1.277
1.275
1.269
1.255
1.253
0.830
0.824
0.819
0.810
0.805
0.799
0.699
0.691
0.687
0.683
0.675
0.000
1.00
1.14
1.16
9.11
1.30
2.17
2.20
8
7
6
5
4
3
2
1
0
ppm

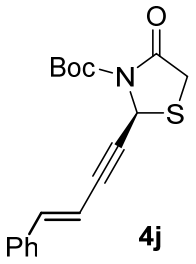

170.20
148.86
90.22
84.95
77.79
77.58
77.37
72.41
50.23
34.08
28.50
8.94
8.92
0.00
200
180
160
140
120
100
80
60
40
20
0
ppm



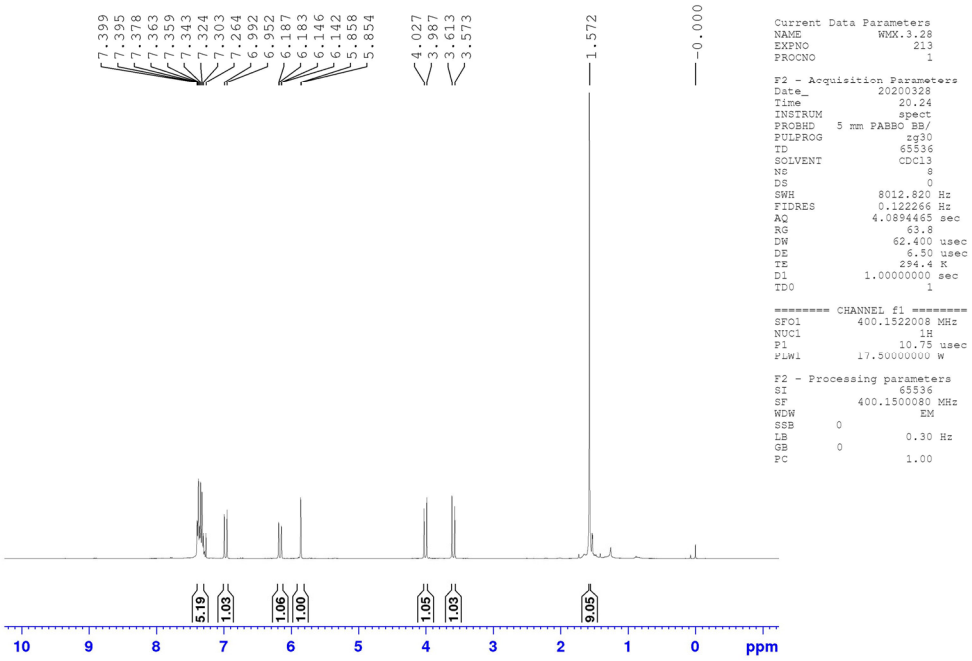
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	13.084	MM	0.3058	4734.38672	258.02844	50.0002
2	15.488	BB	0.3379	4734.35449	217.55765	49.9998



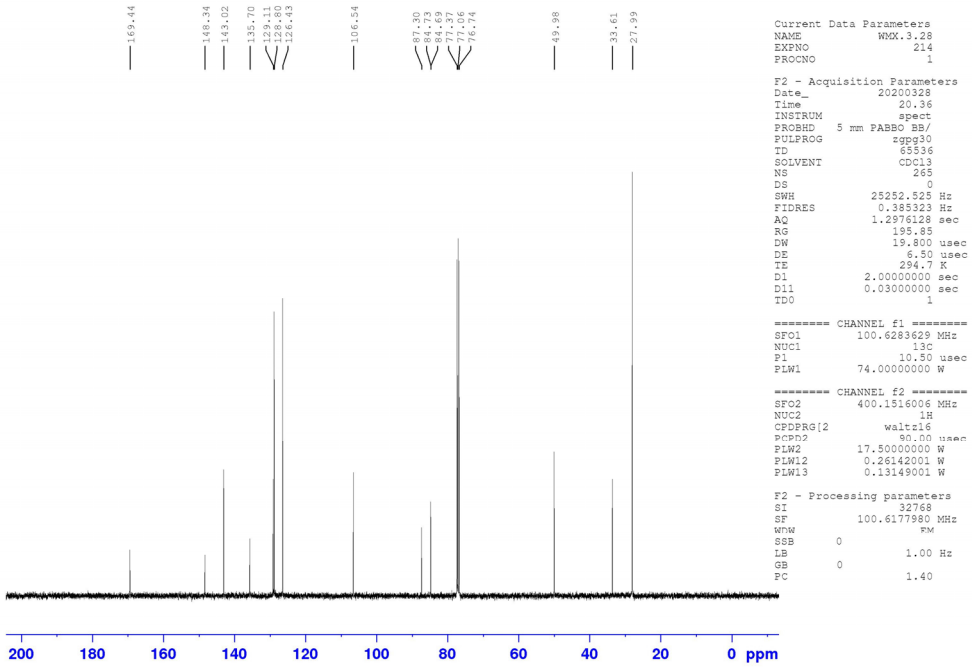
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	12.844	VB R	0.2970	1.24586e4	643.07721	88.6488
2	15.383	MM	0.3566	1595.27161	74.55279	11.3512



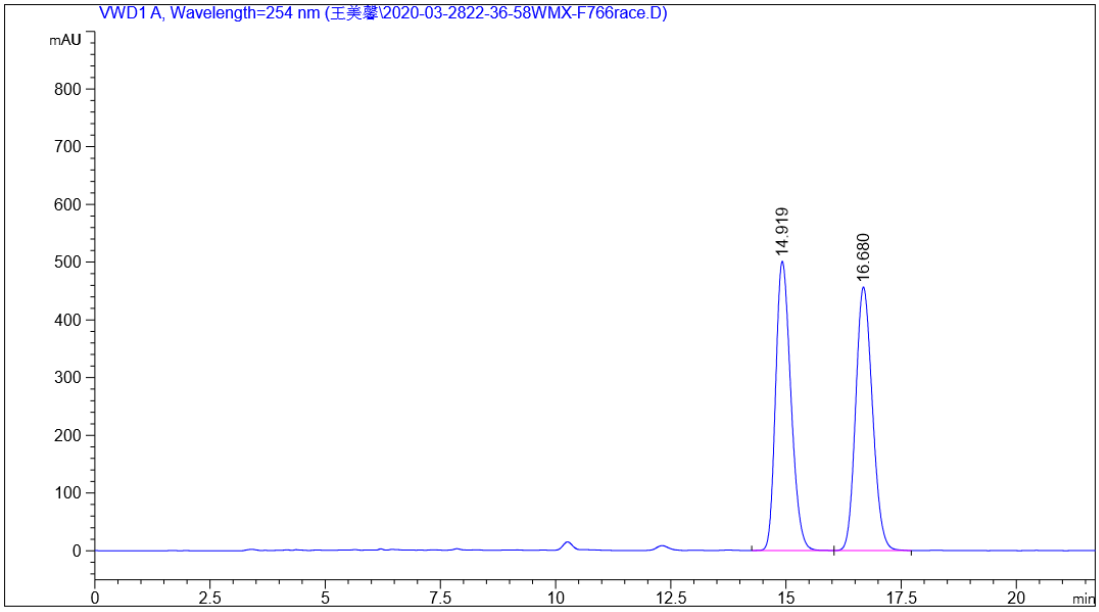
4j



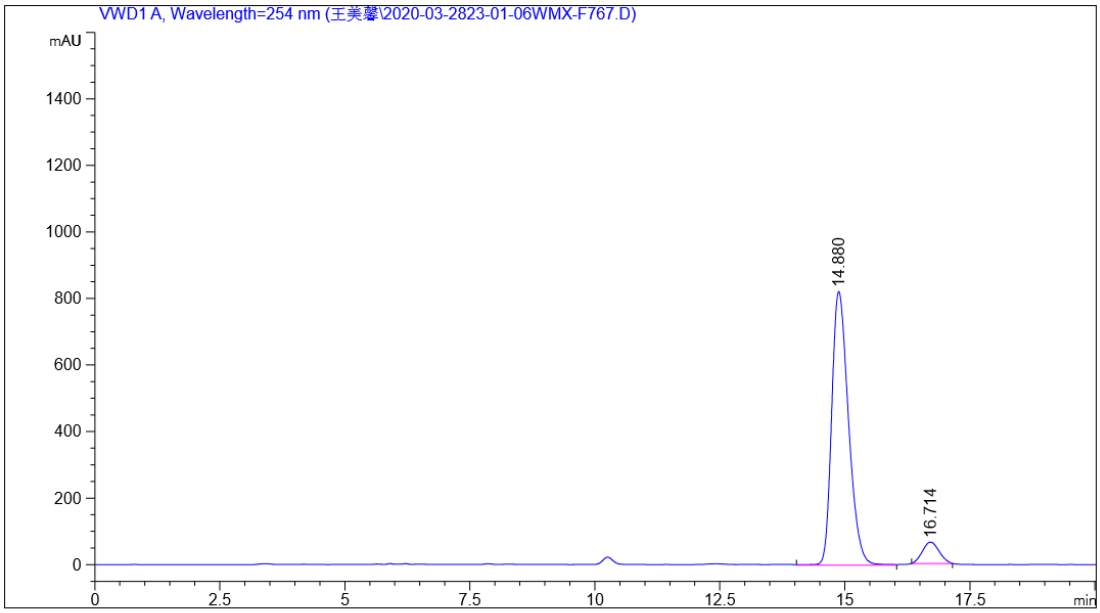

```
Current Data Parameters
NAME                  WMX.3.28
EXPNO                      213
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20200328
Time                     20.24
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                           8
DS                           0
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                        63.8
DW                      62.400 usec
DE                       6.50 usec
TE                      294.4 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               400.1522008 MHz
NUC1                       1H
P1                      10.75 usec
PLW1               17.50000000 W

F2 - Processing parameters
SI                       65536
SF                 400.1500080 MHz
WDW                         EM
SSB          0
LB                       0.30 Hz
GB           0
PC                       1.00
```



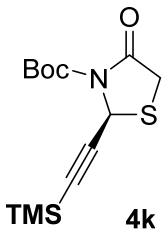

```
Current Data Parameters
NAME                  WMX.3.28
EXPNO                      214
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20200328
Time                     20.36
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                         265
DS                           0
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976128 sec
RG                       195.85
DW                      19.800 usec
DE                       6.50 usec
TE                      294.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1               100.6283629 MHz
NUC1                       13C
P1                      10.50 usec
PLW1               74.00000000 W

======== CHANNEL f2 ========
SFO2               400.1516006 MHz
NUC2                       1H
CPDPRG[2             waltz16
PCPD2                  90.00 usec
PLW2               17.50000000 W
PLW12               0.26142001 W
PLW13               0.13149001 W

F2 - Processing parameters
SI                       32768
SF                 100.6177980 MHz
WDW                         EM
SSB          0
LB                       1.00 Hz
GB           0
PC                       1.40
```



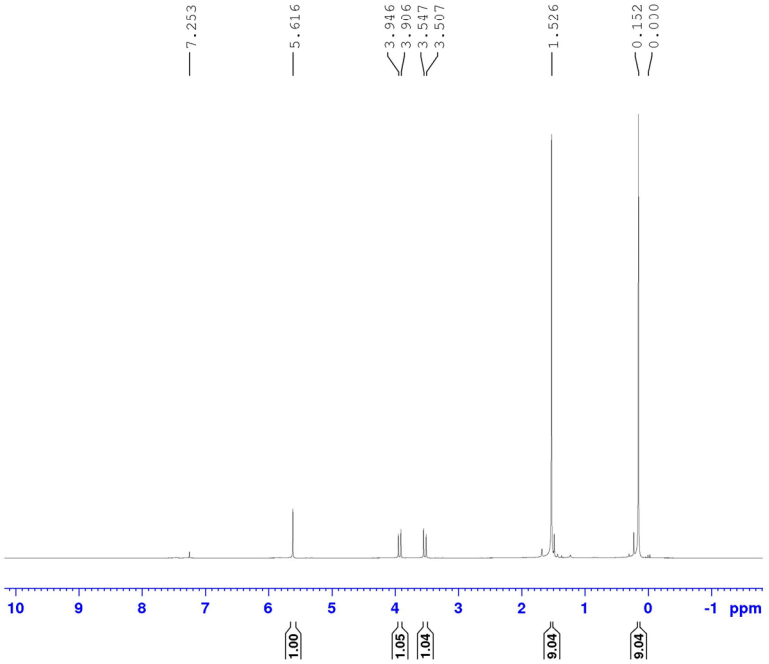
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.919	BB	0.3567	1.15439e4	501.17773	49.9907
2	16.680	BB	0.3941	1.15482e4	456.62704	50.0093



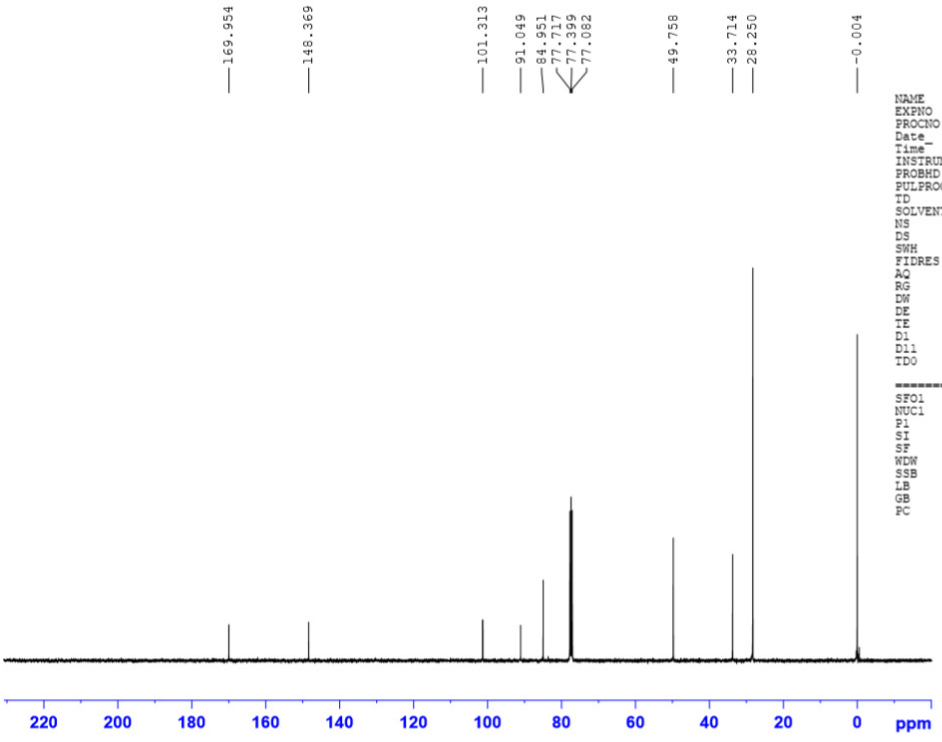
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.880	MM	0.3894	1.92179e4	822.62415	92.7945
2	16.714	MM	0.3866	1492.27856	64.33423	7.2055



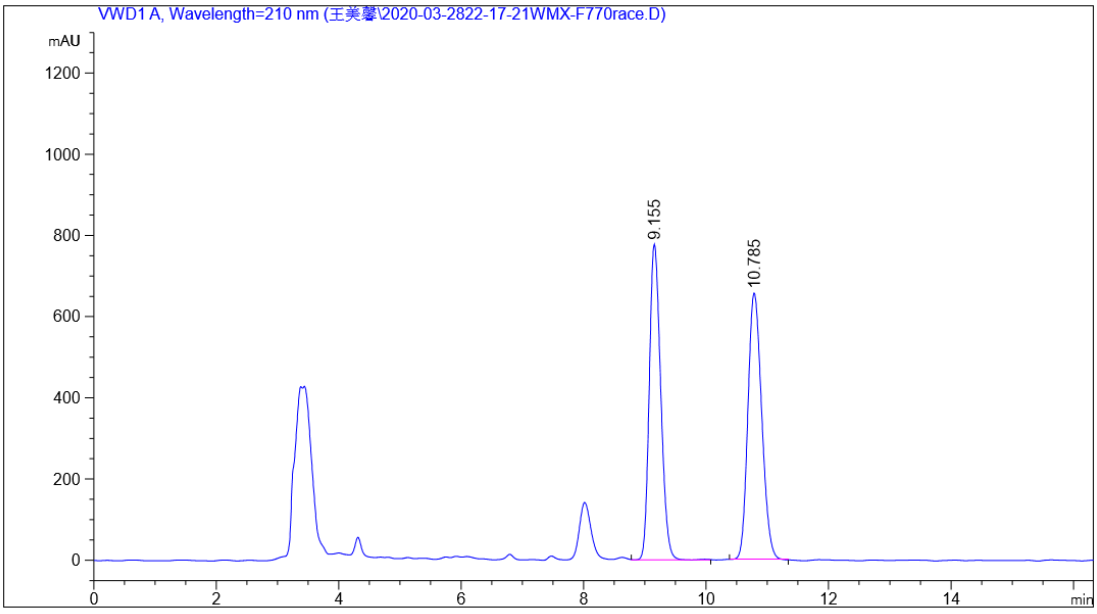
4k



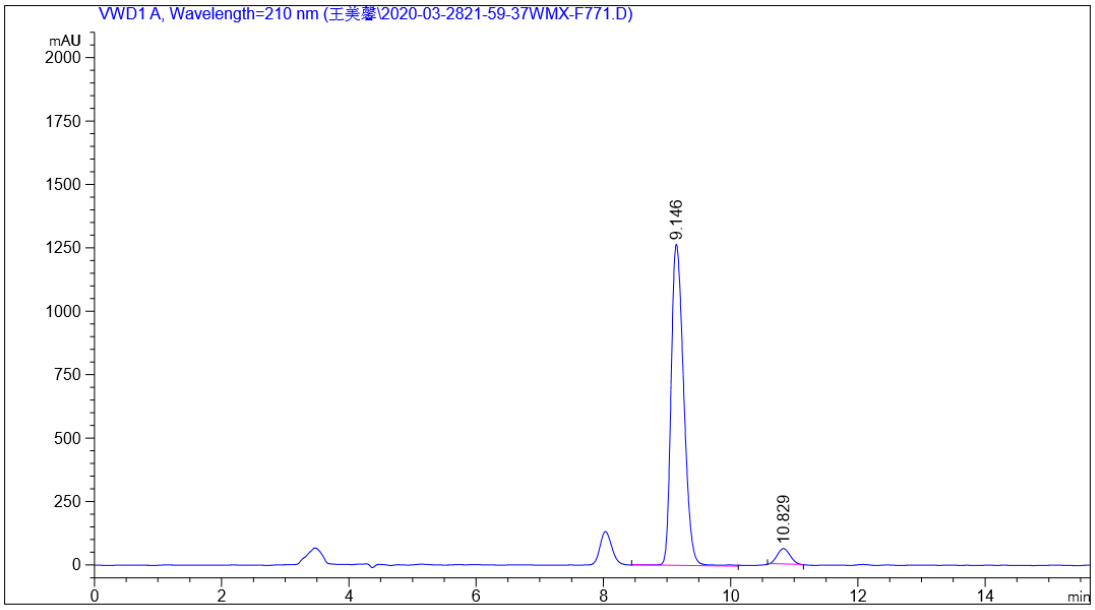

```
Current Data Parameters
NAME                  WMX.3.28
EXPNO                      210
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20200328
Time                     19.48
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                       31.56
DW                      62.400 usec
DE                       6.50 usec
TE                      294.0 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                      10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                          65536
SF                  400.1500129 MHz
WDW                            EM
SSB                 0
LB                       0.30 Hz
GB                  0
PC                       1.00
```



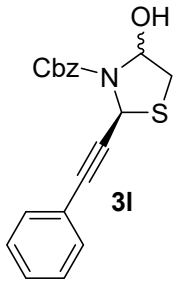

```
NAME                      WMX-2
EXPNO                       211
PROCNO                        1
Date_                  20200328
Time                      20.06
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         385
DS                           0
SWH                  25252.525 Hz
FIDRES               0.385323 Hz
AQ                   1.2976629 sec
RG                      195.85
DW                      19.800 usec
DE                       6.50 usec
TE                      294.7 K
D1                   2.00000000 sec
D11                  0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                      10.50 usec
SI                         32768
SF                  100.6177634 MHz
WDW                            EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```



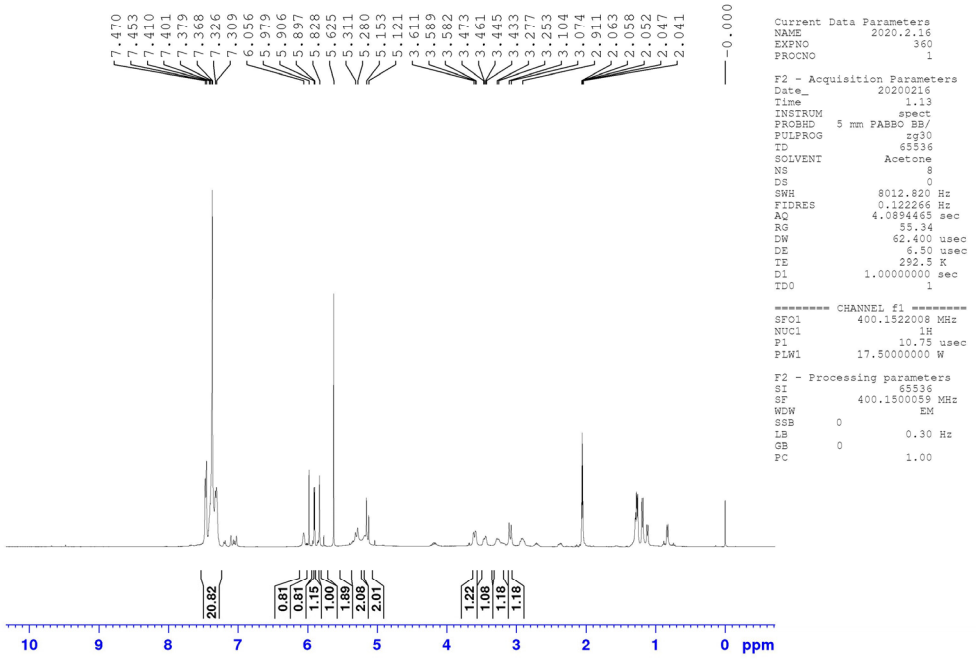
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.155	MM	0.2168	1.01121e4	777.45612	49.7221
2	10.785	MM	0.2602	1.02251e4	655.01587	50.2779



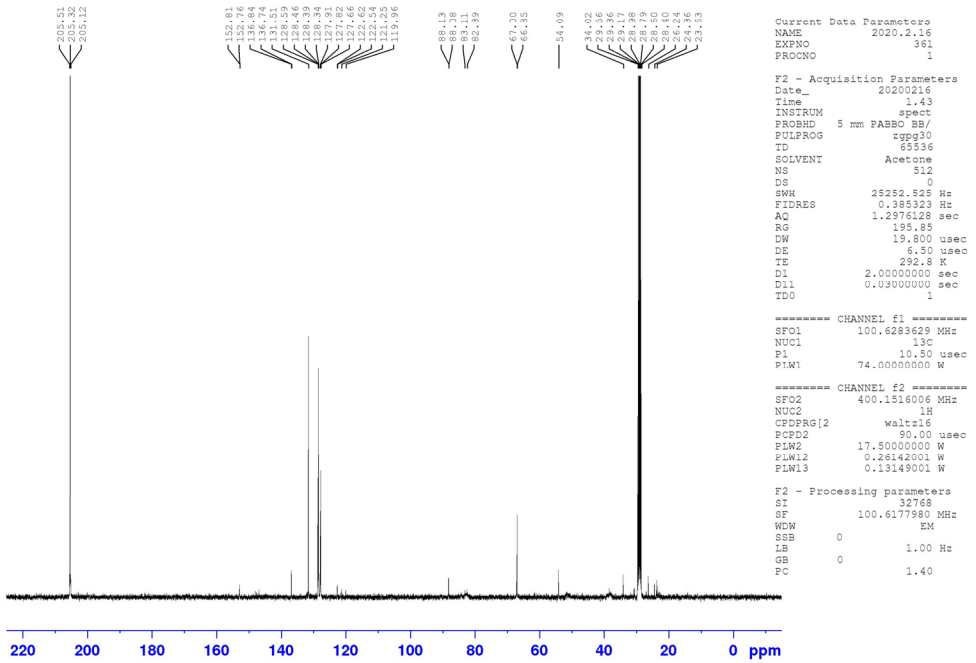
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.146	MM	0.2294	1.74089e4	1264.93054	95.4199
2	10.829	MM	0.2309	835.61603	60.32272	4.5801



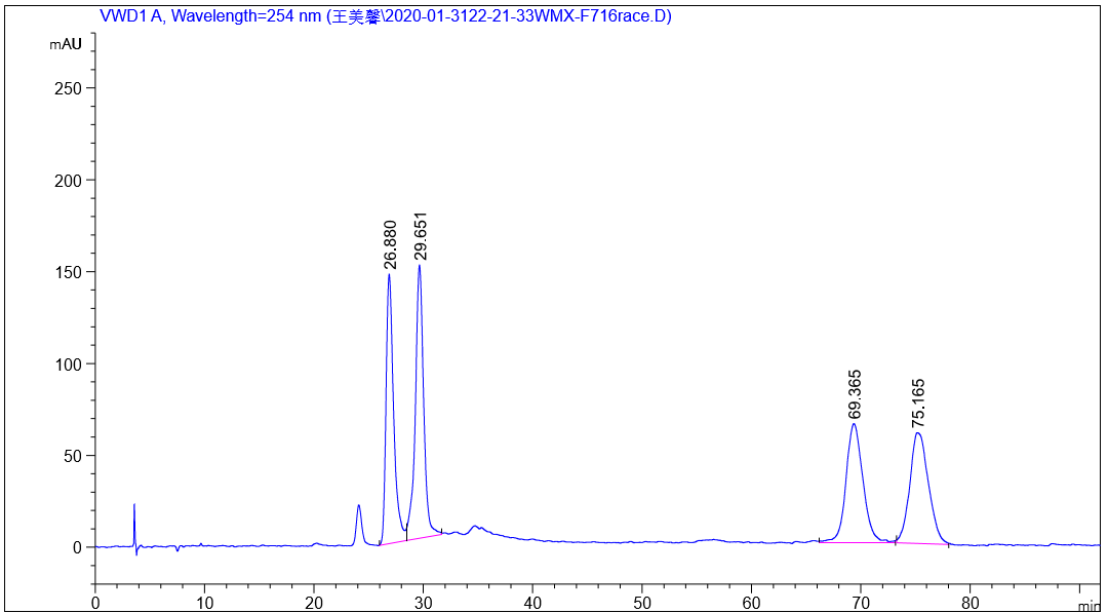
3l



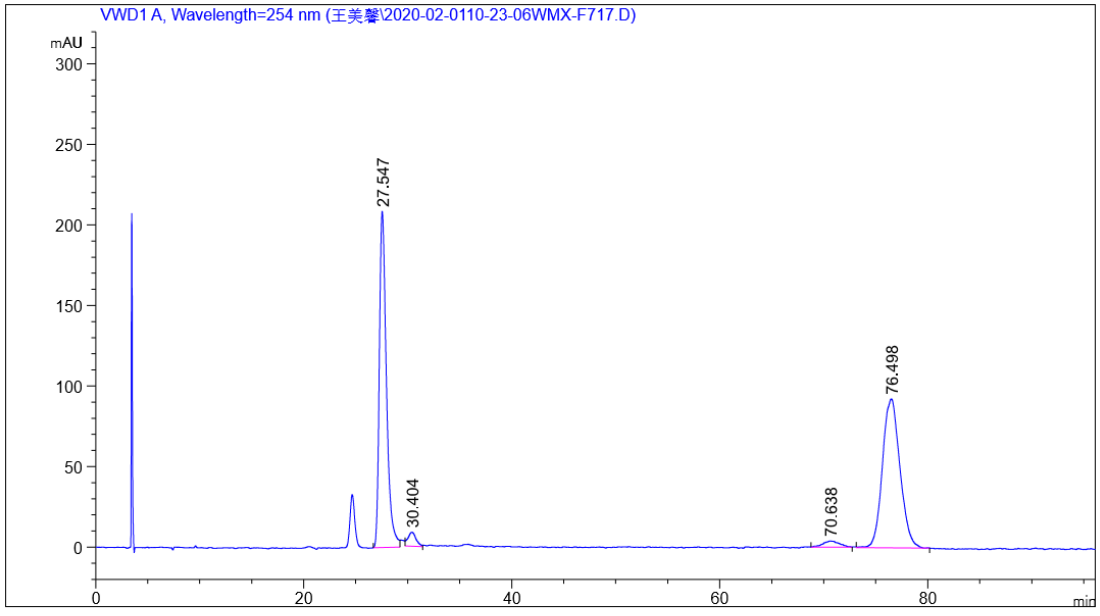

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       360
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20200216
Time                      1.13
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                Acetone
NS                           8
DS                           0
SWH                   8012.820 Hz
FIDRES                0.122266 Hz
AQ                    4.0894465 sec
RG                        55.34
DW                       62.400 usec
DE                        6.50 usec
TE                       292.5 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                       10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                        65536
SF                  400.1500059 MHz
WDW                          EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```



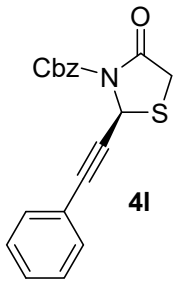

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       361
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20200216
Time                      1.43
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                Acetone
NS                           512
DS                           0
SWH                   25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976128 sec
RG                        195.85
DW                       19.800 usec
DE                        6.50 usec
TE                       292.8 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                       10.50 usec
PLW1                74.00000000 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W
PLW13                0.13149001 W

F2 - Processing parameters
SI                        32768
SF                  100.6177980 MHz
WDW                          EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```



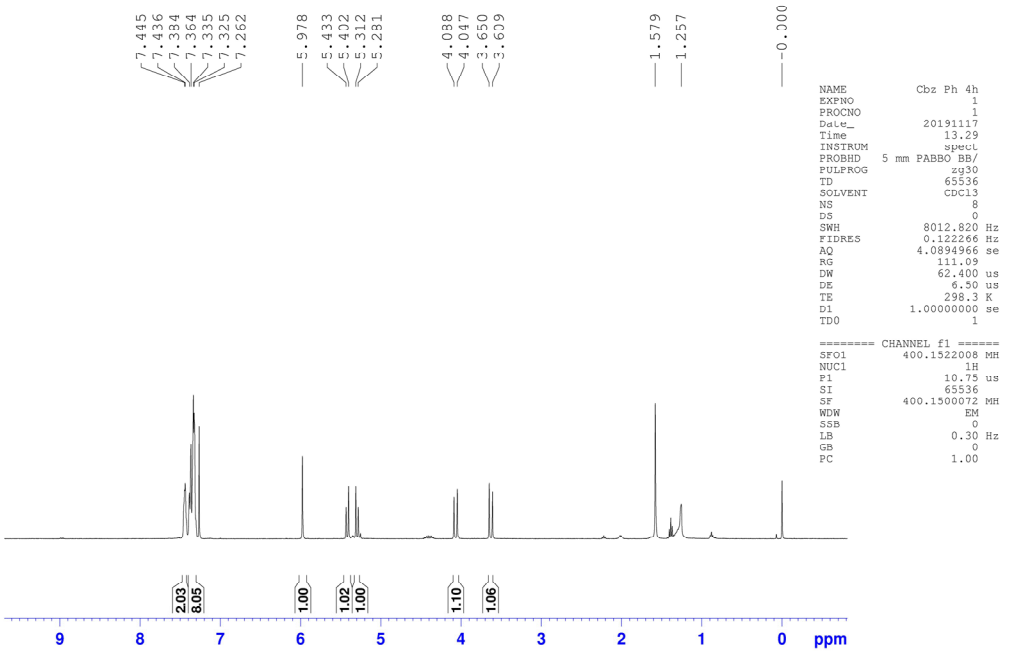
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.880	MM	0.7717	6709.38428	144.89612	24.2136
2	29.651	MM	0.8034	6857.87256	142.27571	24.7494
3	69.365	MM	1.8045	7016.55029	64.80614	25.3221
4	75.165	MM	1.9727	7125.39844	60.19931	25.7149



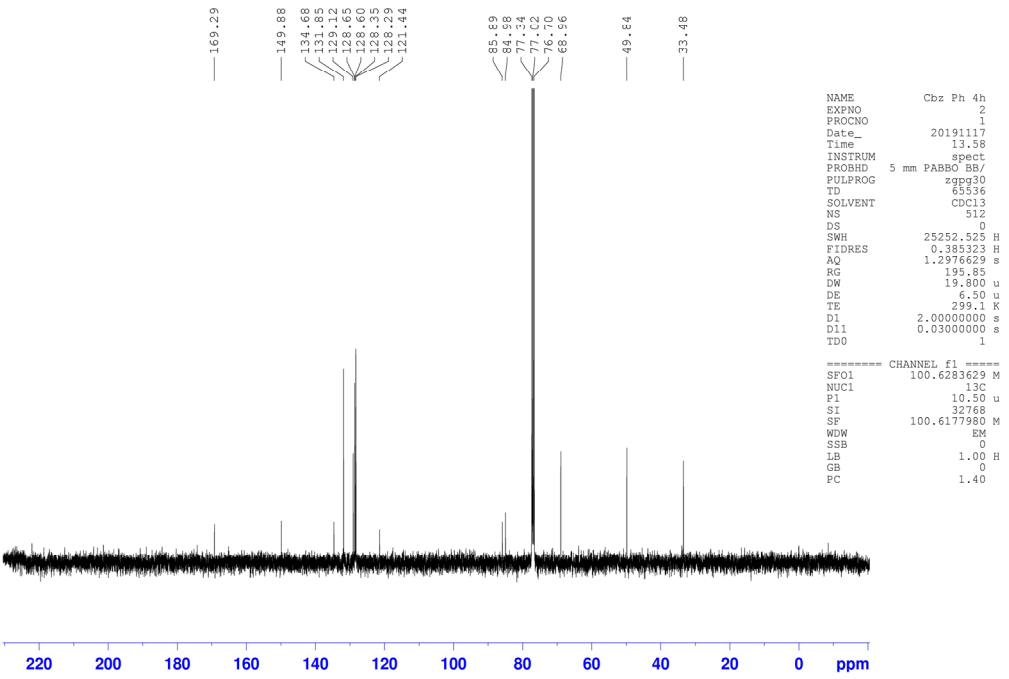
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.547	MM	0.7566	9314.26465	205.17818	44.5912
2	30.404	MM	0.7769	328.19852	7.04086	1.5712
3	70.638	MM	1.8456	425.89789	3.84614	2.0389
4	76.498	MM	1.9510	1.08198e4	92.42960	51.7987



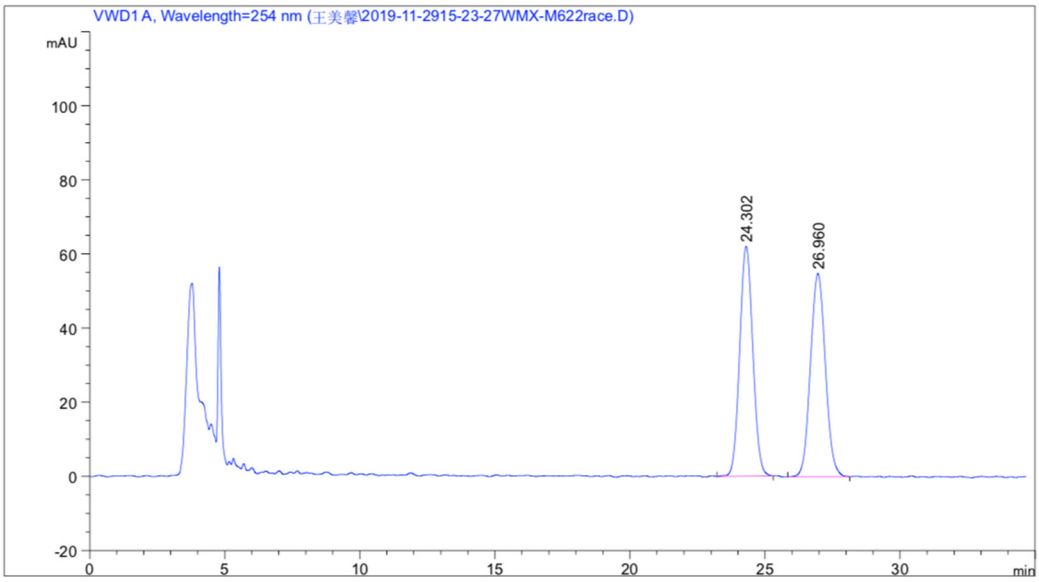
4l



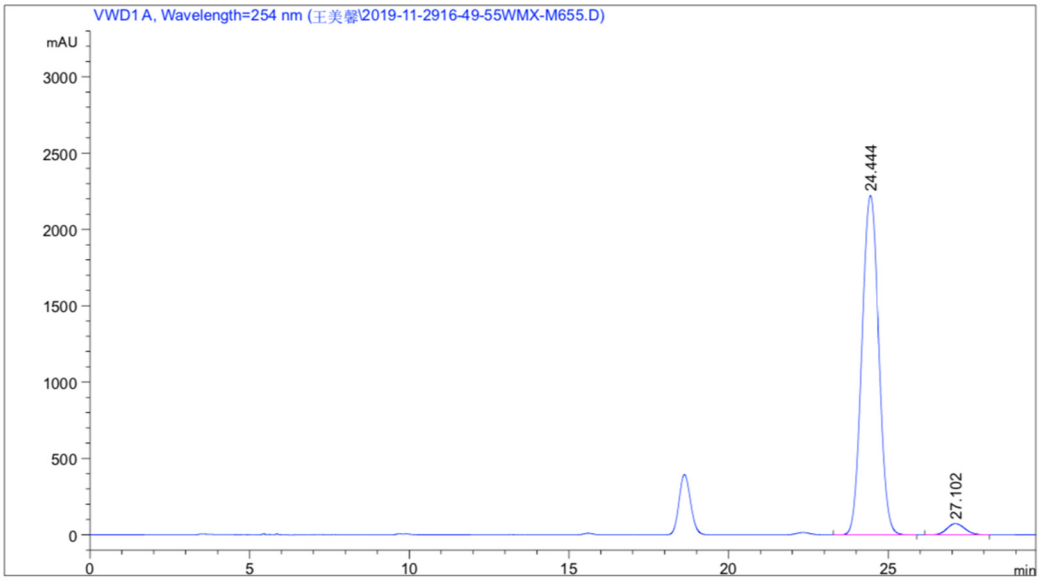

```
NAME            Cbz Ph 4h
EXPNO                   1
PROCNO                  1
Date_            20191117
Time                13.29
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 se
RG                  111.09
DW                  62.400 us
DE                    6.50 us
TE                  298.3 K
D1               1.00000000 se
TD0                     1

======== CHANNEL f1 ======
SFO1          400.1522008 MH
NUC1                   1H
P1                 10.75 us
SI                  65536
SF            400.1300072 MH
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



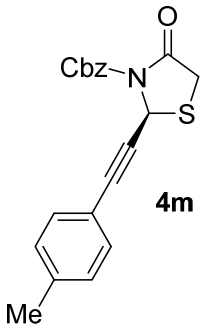

```
NAME            Cbz Ph 4h
EXPNO                   2
PROCNO                  1
Date_            20191117
Time                13.58
INSTRUM             spect
PROBHD    5 mm PABBO BB/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      0
SWH             25252.525 H
FIDRES           0.385323 H
AQ               1.2976629 s
RG                  195.85
DW                  19.800 u
DE                    6.50 u
TE                  299.1 K
D1               2.00000000 s
D11              0.03000000 s
TD0                     1

======== CHANNEL f1 ======
SFO1          100.6283629 M
NUC1                   13C
P1                 10.50 u
SI                  32768
SF            100.6177980 M
WDW                    EM
SSB                     0
LB                   1.00 H
GB                      0
PC                   1.40
```



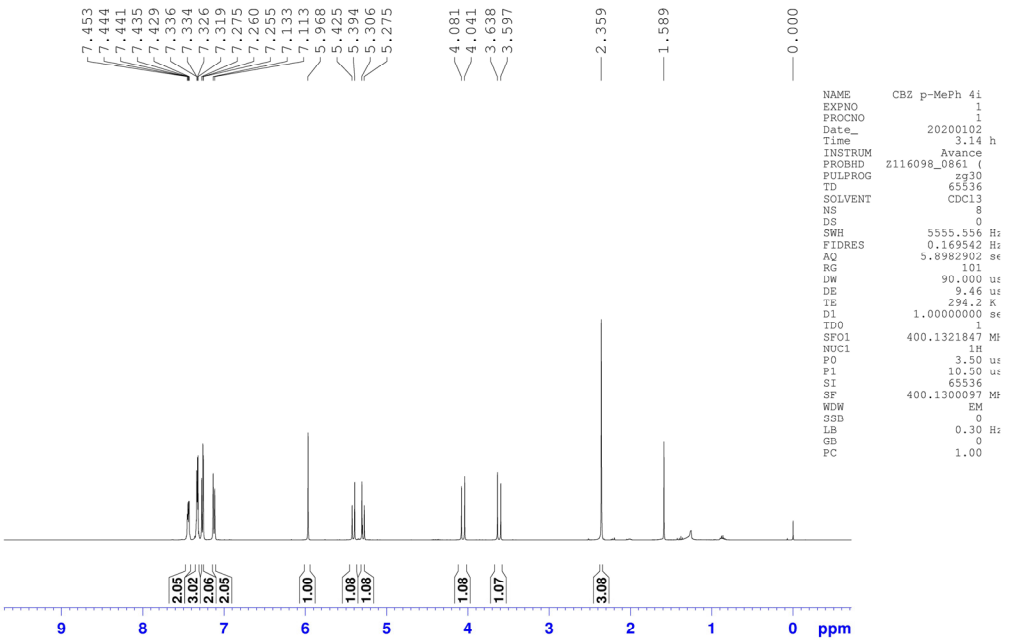
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.302	BB	0.5282	2096.36499	62.04695	49.8972
2	26.960	BB	0.6146	2105.00684	54.87020	50.1028



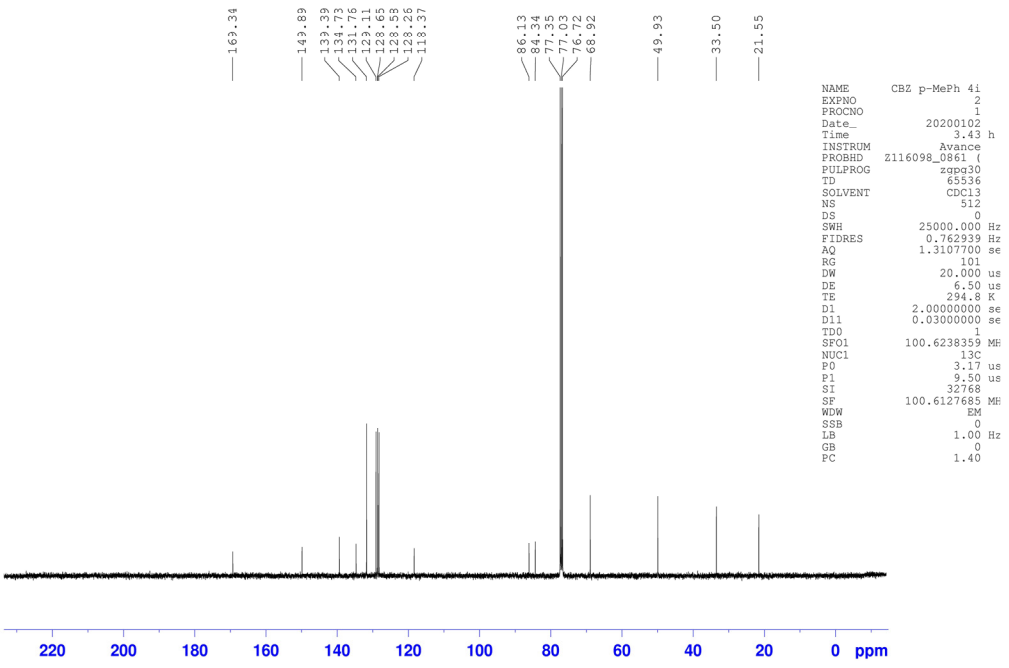
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.444	BB	0.5825	8.14698e4	2223.58545	96.6077
2	27.102	BB	0.6141	2860.73145	73.17535	3.3923



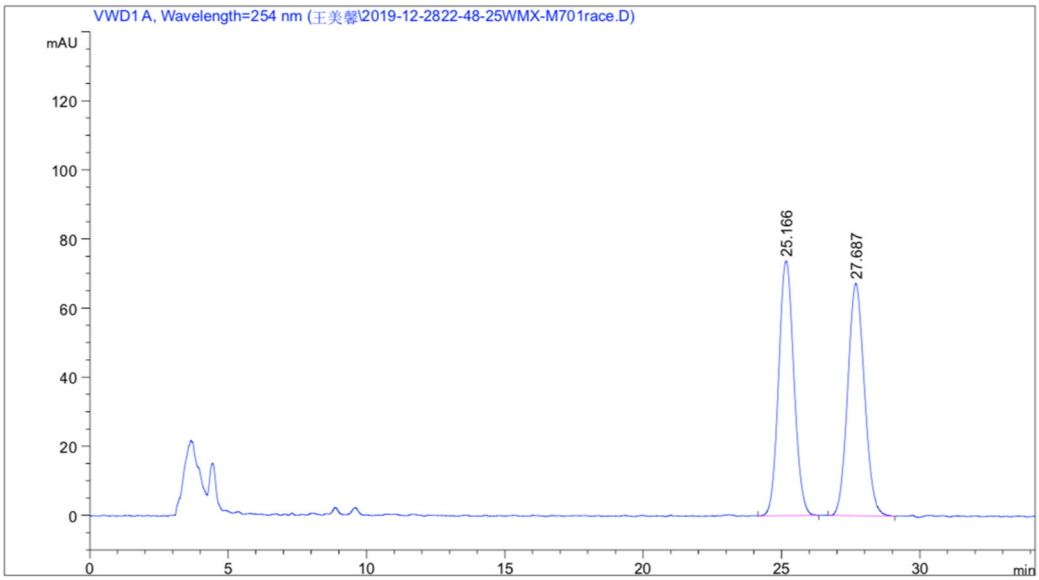
4m



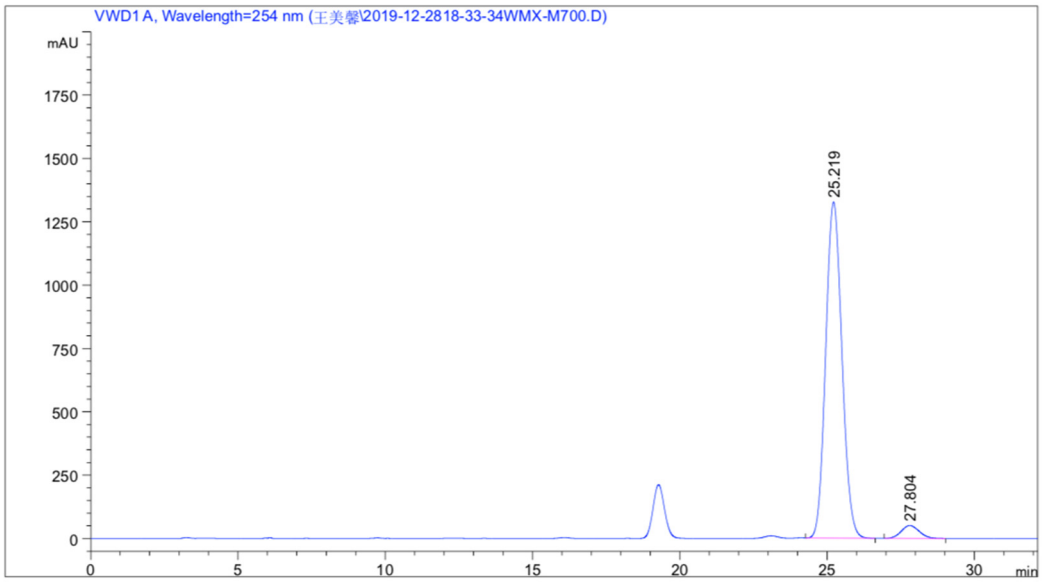

```
NAME          CBZ p-MePh 4i
EXPNO                    1
PROCNO                   1
Date_             20200102
Time                 3.14 h
INSTRUM              Avance
PROBHD       Z116098_0861 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                5555.556 Hz
FIDRES            0.169542 Hz
AQ                5.8982902 se
RG                      101
DW                   90.000 us
DE                    9.46 us
TE                   294.2 K
D1                1.00000000 se
TD0                       1
SFO1             400.1321847 MH
NUC1                     1H
P0                    3.50 us
P1                   10.50 us
SI                    65536
SF               400.1300097 MH
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                     1.00
```



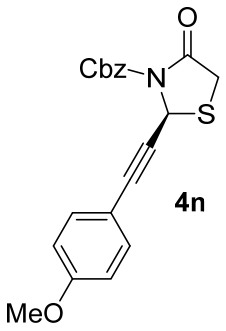

```
NAME          CBZ p-MePh 4i
EXPNO                    2
PROCNO                   1
Date_             20200102
Time                 3.43 h
INSTRUM              Avance
PROBHD       Z116098_0861 (
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25000.000 Hz
FIDRES            0.762939 Hz
AQ                1.3107700 se
RG                      101
DW                   20.000 us
DE                    6.50 us
TE                   294.8 K
D1                2.00000000 se
D11               0.03000000 se
TD0                       1
SFO1             100.6238359 MH
NUC1                     13C
P0                    3.17 us
P1                    9.50 us
SI                    32768
SF               100.6127685 MH
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                     1.40
```



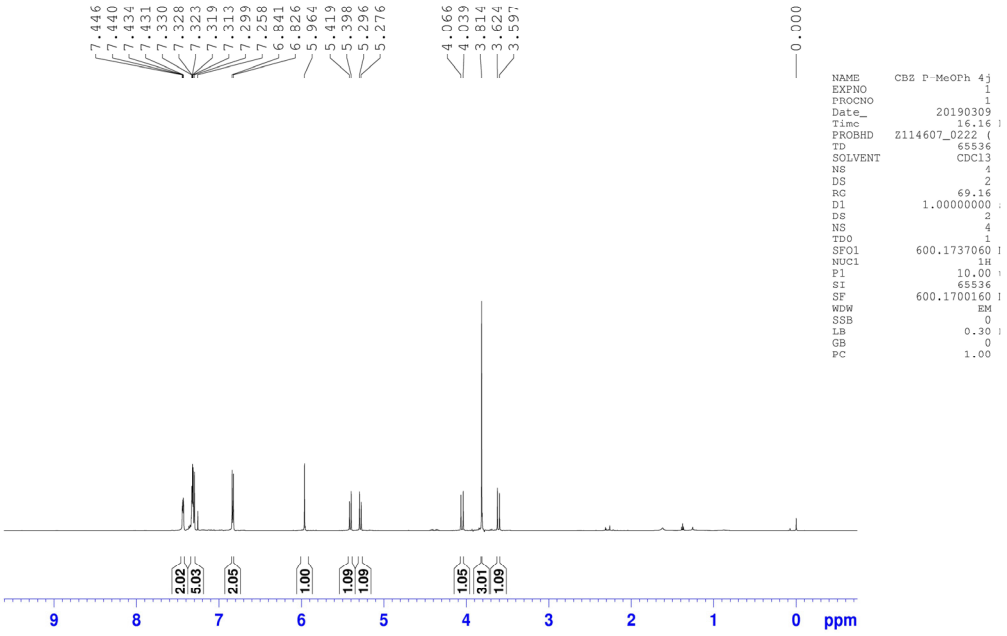
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.166	BB	0.5903	2829.81299	73.80706	50.0084
2	27.687	BB	0.6586	2828.86084	67.27695	49.9916

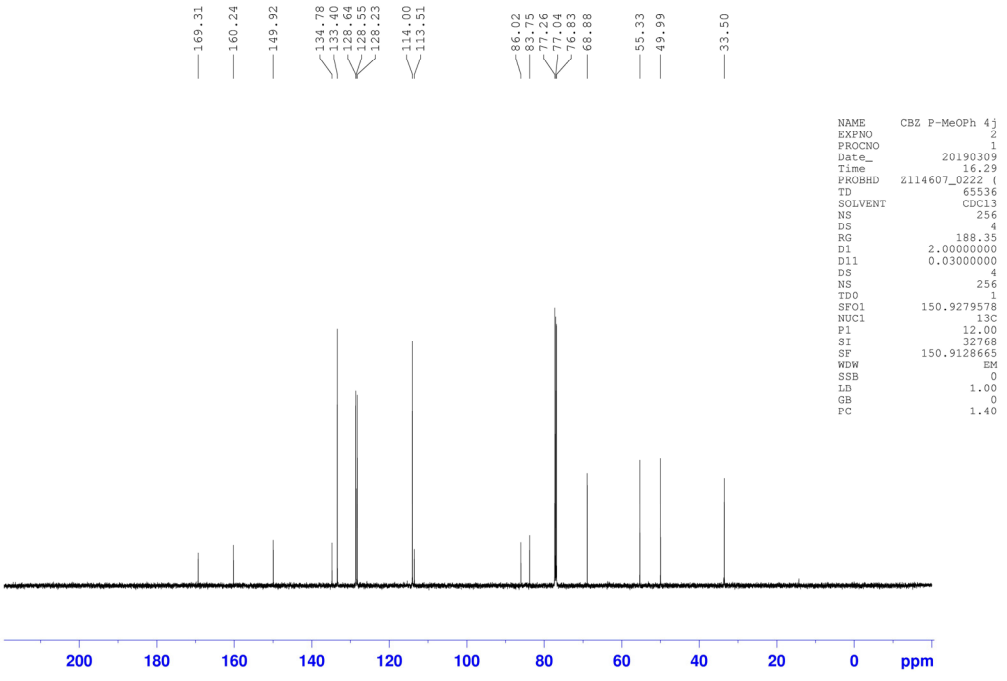


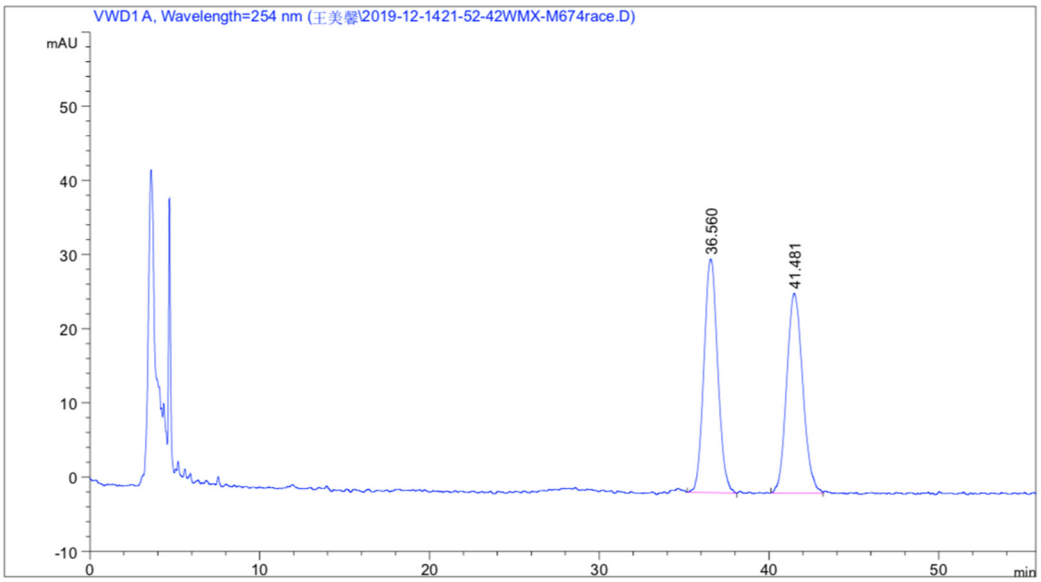
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	25.219	BB	0.6059	5.08205e4	1326.67664	95.9448
2	27.804	BB	0.6700	2147.97046	51.04313	4.0552


O
Cbz
N
S
MeO

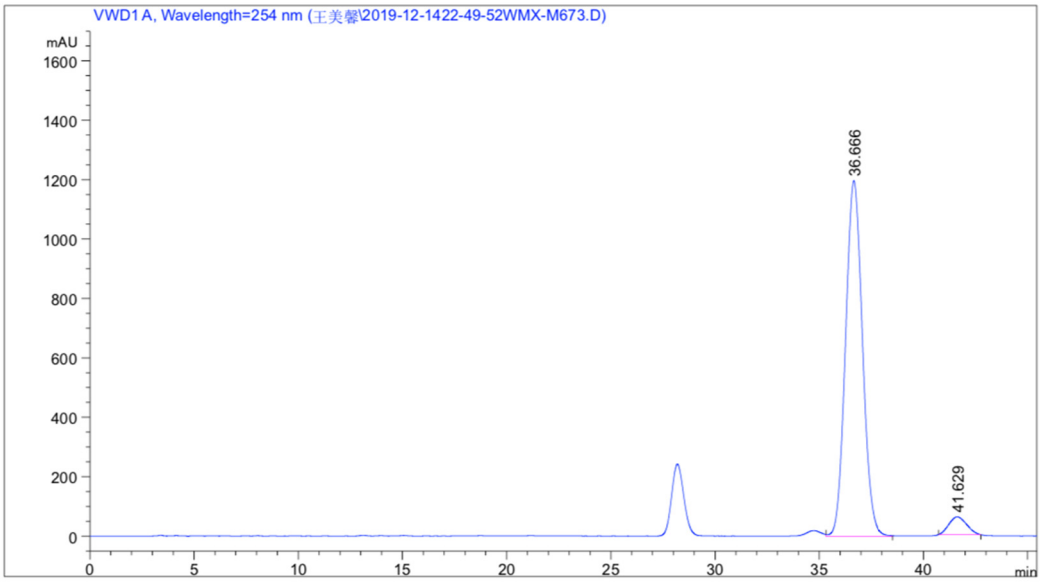
4n


7.446
7.440
7.434
7.431
7.330
7.328
7.323
7.319
7.313
7.299
7.258
6.841
6.826
5.964
5.419
5.398
5.296
5.276
4.066
4.039
3.814
3.624
3.597
0.000
NAME CBZ P-MeOPh 4j
EXPNO 1
PROCNO 1
Date_ 20190309
Time 16.16
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 4
DS 2
RG 69.16
D1 1.00000000
DS 2
NS 4
TD0 1
SFO1 600.1737060
NUC1 1H
P1 10.00
SI 65536
SF 600.1700160
WDW EM
SSB 0
LB 0.30
GB 0
PC 1.00
2.02
5.03
2.05
1.00
1.09
1.09
1.05
3.01
1.09
9
8
7
6
5
4
3
2
1
0
ppm

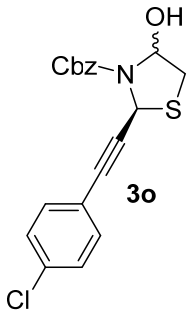

169.31
160.24
149.92
134.78
133.40
128.64
128.55
128.23
114.00
113.51
86.02
83.75
77.26
77.04
76.83
68.88
55.33
49.99
33.50
NAME CBZ P-MeOPh 4j
EXPNO 2
PROCNO 1
Date_ 20190309
Time 16.29
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 256
DS 4
RG 188.35
D1 2.00000000
D11 0.03000000
DS 4
NS 256
TD0 1
SFO1 150.9279578
NUC1 13C
P1 12.00
SI 32768
SF 150.9128665
WDW EM
SSB 0
LB 1.00
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



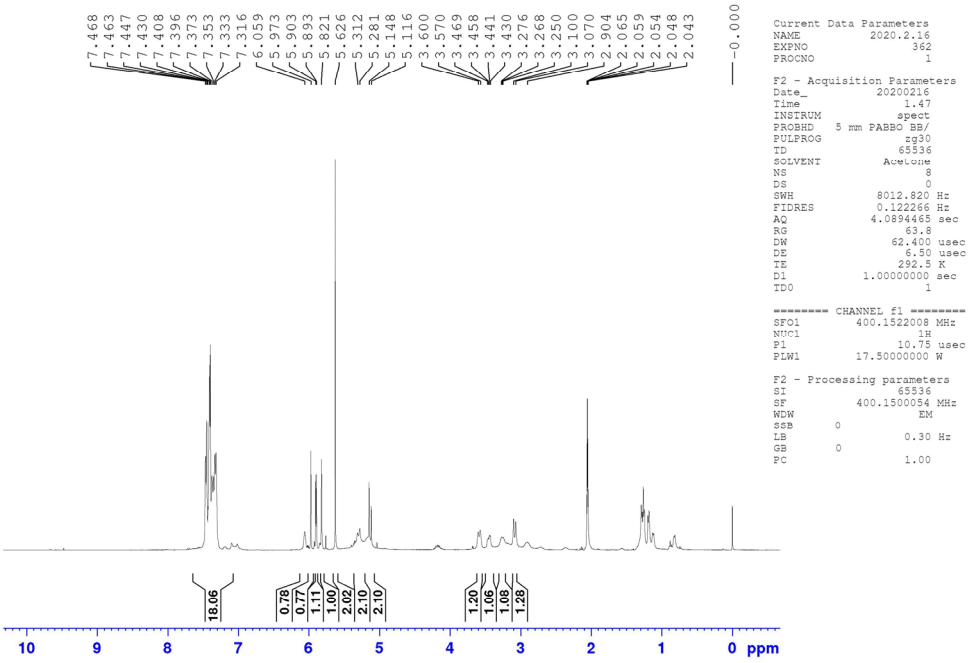
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.560	BB	0.8179	1781.79517	31.49991	50.1557
2	41.481	MM	1.0949	1770.73193	26.95395	49.8443



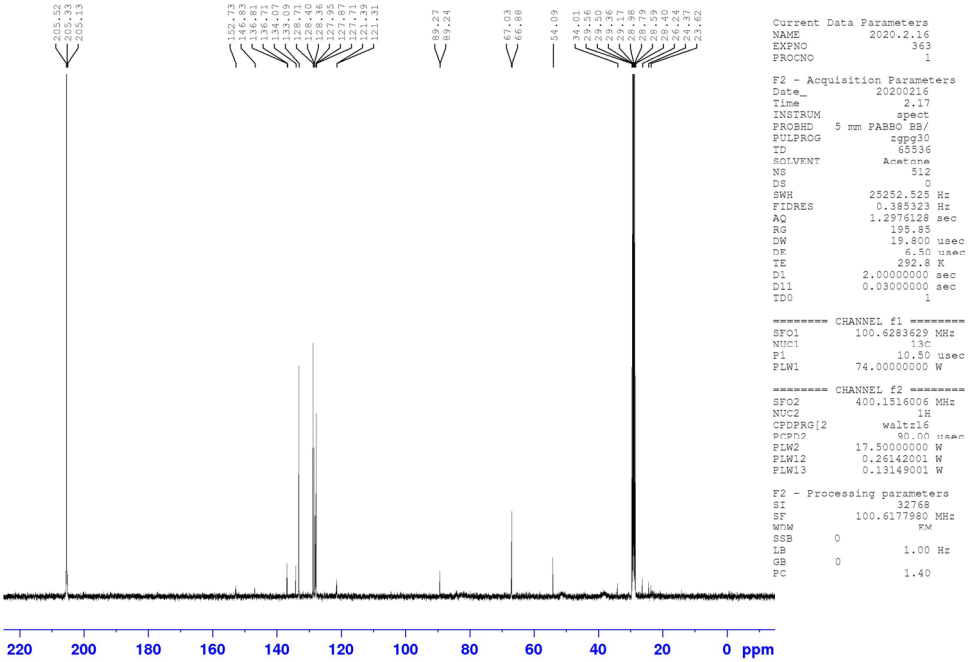
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	36.666	MM	0.9421	6.76844e4	1197.45667	95.0630
2	41.629	MM	0.9825	3515.12427	59.62617	4.9370



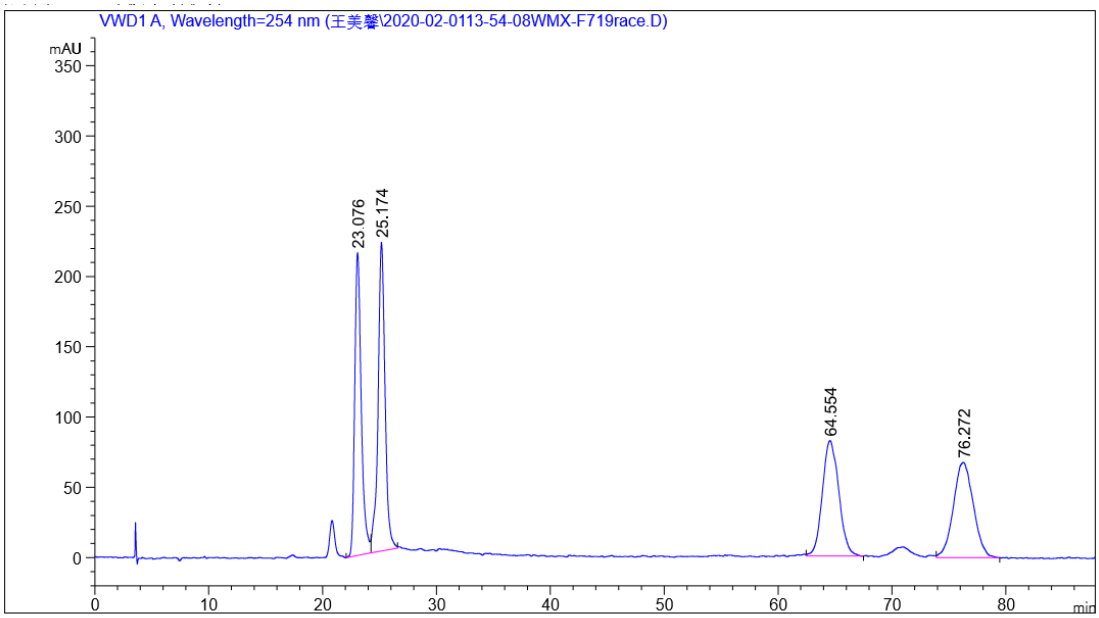
3o



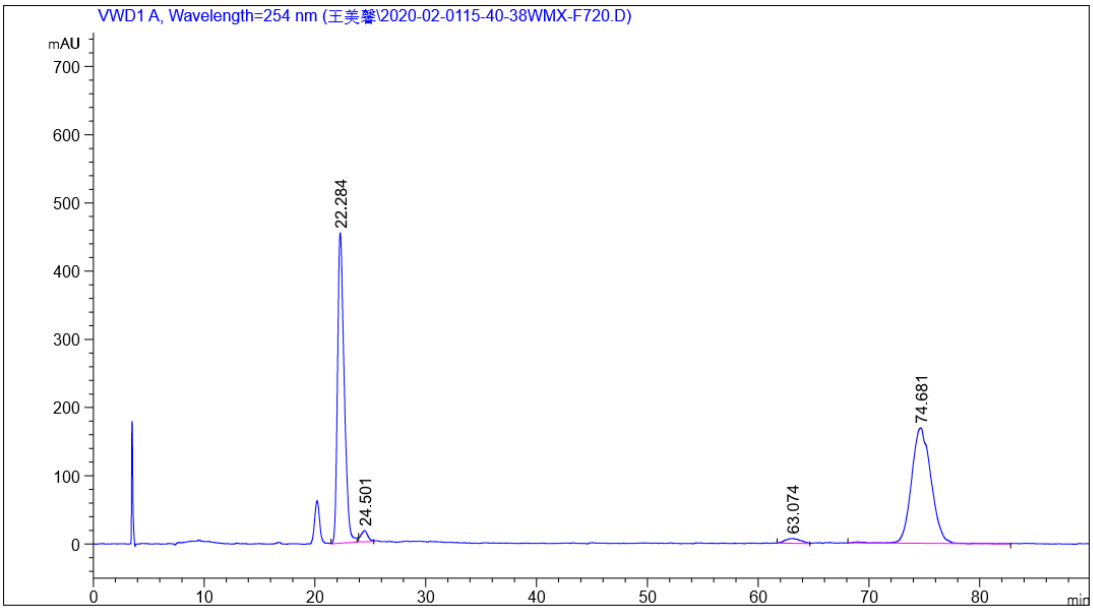

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       362
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                       1.47
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                Acetone
NS                             8
DS                             0
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894465 sec
RG                         63.8
DW                    62.400 usec
DE                     6.50 usec
TE                     292.5 K
D1                  1.00000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                    10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                         65536
SF                  400.1500054 MHz
WDW                           EM
SSB                 0
LB                     0.30 Hz
GB                  0
PC                     1.00
```



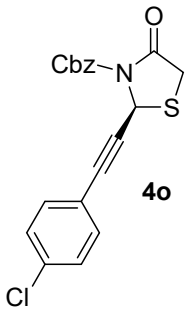

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       363
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                       2.17
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                Acetone
NS                           512
DS                             0
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976128 sec
RG                        195.85
DW                    19.800 usec
DE                     6.50 usec
TE                     292.8 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                            1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                    10.50 usec
PLW1                74.00000000 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                 90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W
PLW13                0.13149001 W

F2 - Processing parameters
SI                         32768
SF                  100.6177980 MHz
WDW                           EM
SSB                 0
LB                     1.00 Hz
GB                  0
PC                     1.40
```



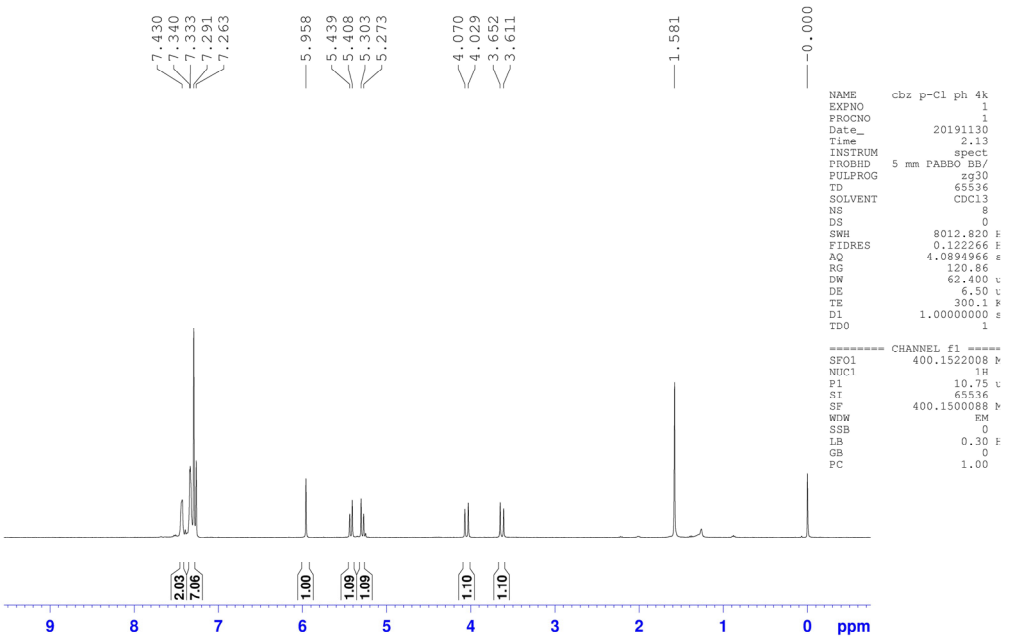
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.076	MM	0.6355	8070.10498	211.65628	24.5891
2	25.174	MM	0.6540	8260.32813	210.49434	25.1686
3	64.554	MM	1.6765	8242.50586	81.94035	25.1143
4	76.272	MM	2.0328	8246.97266	67.61681	25.1280



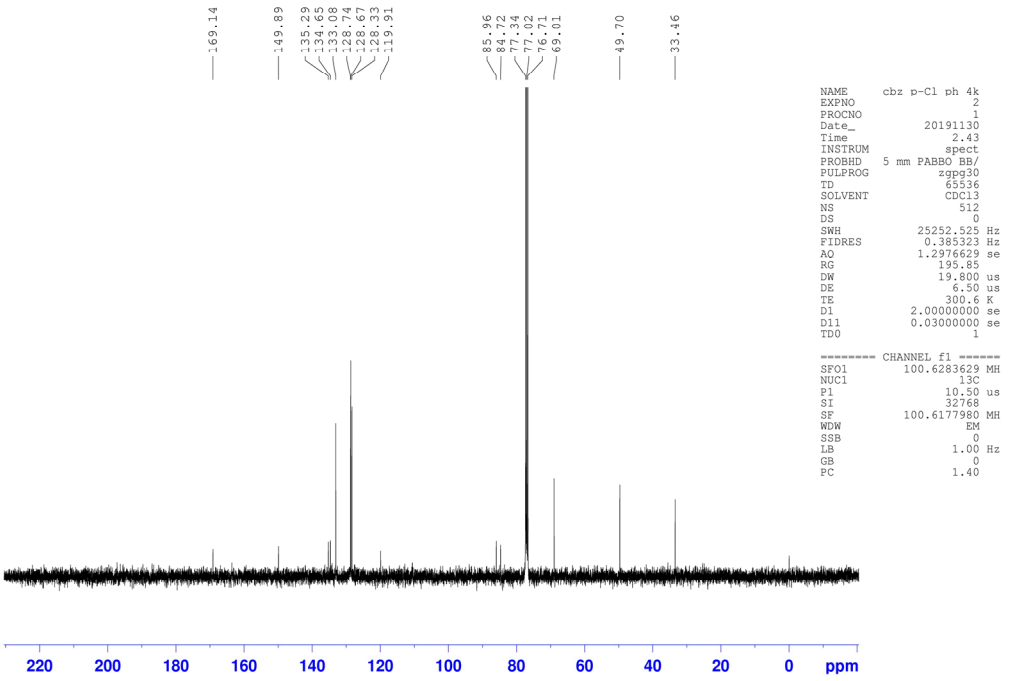
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	22.284	MM	0.6812	1.85191e4	453.08725	46.1384
2	24.501	MM	0.5457	379.21011	11.58216	0.9448
3	63.074	MM	1.2365	389.83884	5.25474	0.9712
4	74.681	MM	2.0552	2.08500e4	169.08481	51.9456



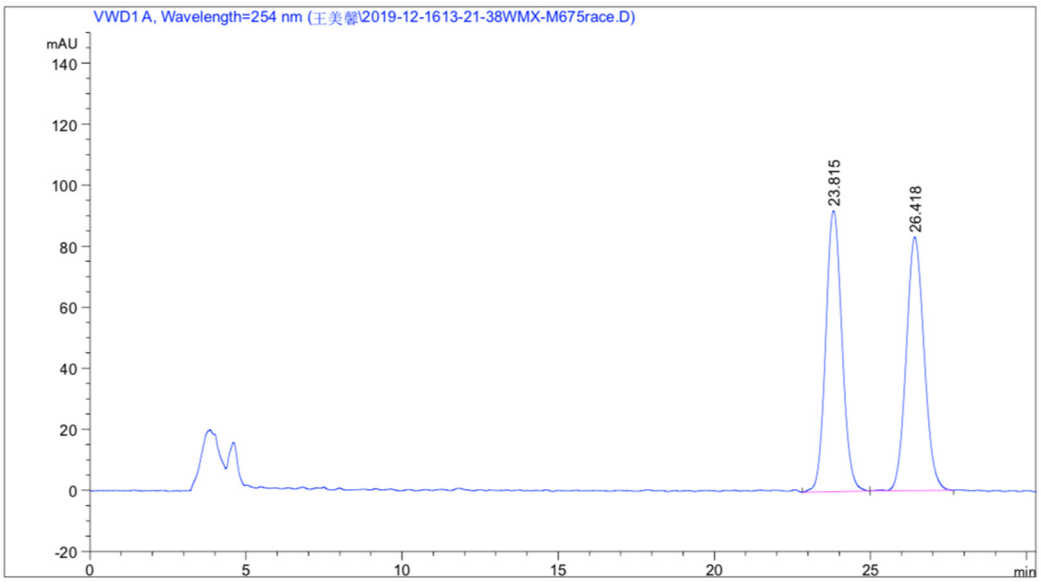
4o



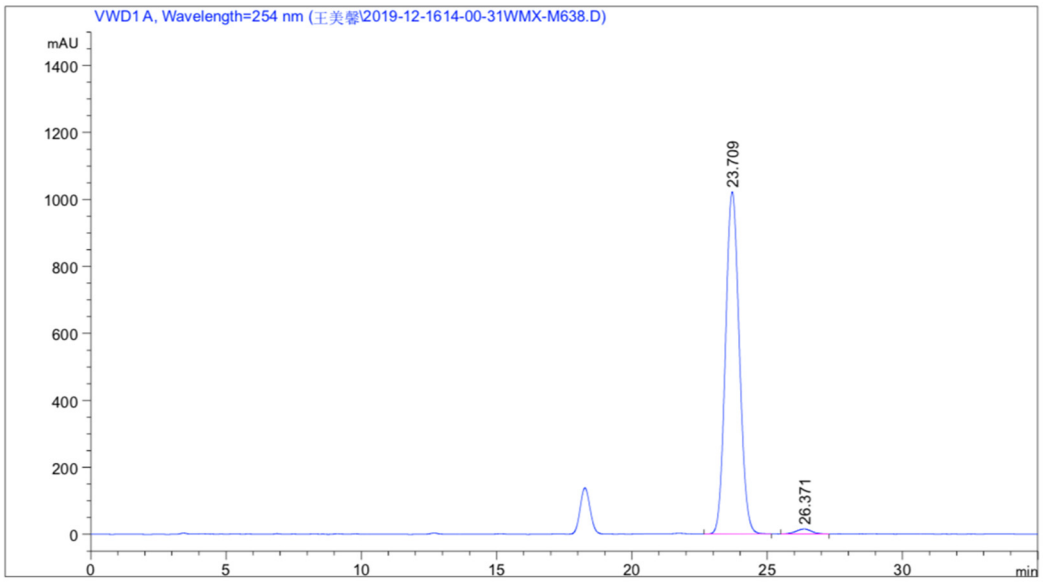

```
NAME        cbz p-Cl ph 4k
EXPNO                    1
PROCNO                   1
Date_             20191130
Time                 2.13
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                       8
DS                       0
SWH               8012.820 H
FIDRES            0.122266 H
AQ                4.0894966 s
RG                   120.86
DW                   62.400 u
DE                    6.50 u
TE                   300.1 K
D1                1.00000000 s
TD0                      1

======== CHANNEL f1 ======
SFO1           400.1522008 M
NUC1                    1H
P1                   10.75 u
SI                   65536
SF             400.1500088 M
WDW                    EM
SSB                     0
LB                   0.30 H
GB                      0
PC                   1.00
```



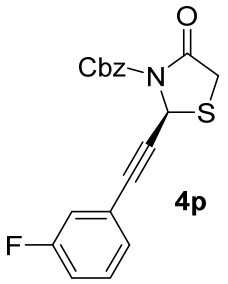

```
NAME        cbz p-Cl ph 4k
EXPNO                    2
PROCNO                   1
Date_             20191130
Time                 2.43
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                     512
DS                       0
SWH              25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 se
RG                   195.85
DW                   19.800 us
DE                    6.50 us
TE                   300.6 K
D1                2.00000000 se
D11               0.03000000 se
TD0                      1

======== CHANNEL f1 ======
SFO1           100.6283629 MH
NUC1                    13C
P1                   10.50 us
SI                   32768
SF             100.6177980 MH
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



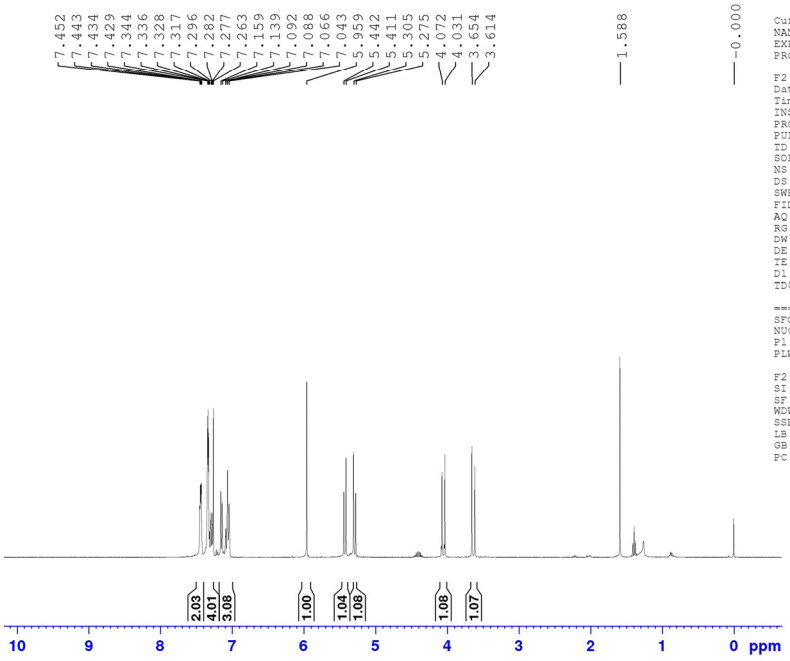
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.815	BB	0.5650	3330.57300	92.08092	50.1609
2	26.418	VB R	0.6233	3309.20361	83.15253	49.8391



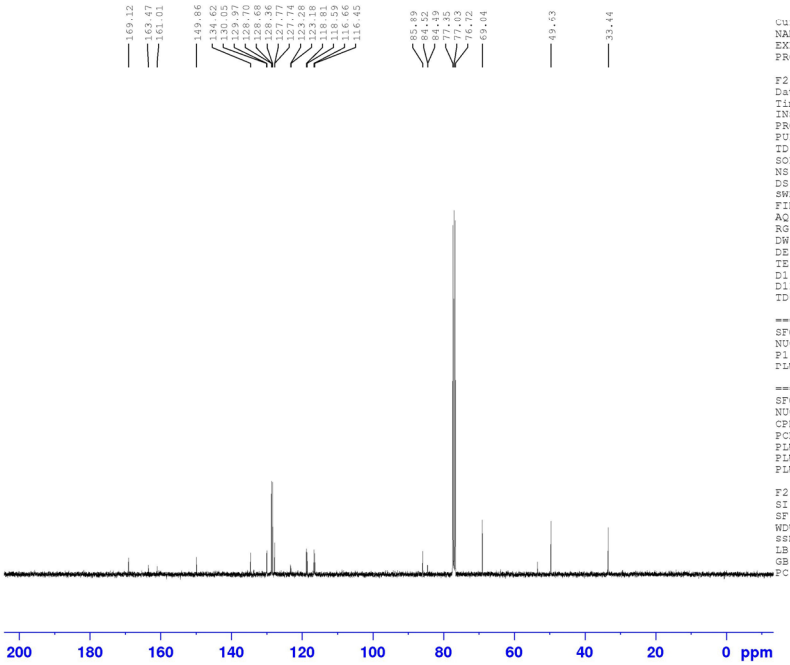
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.709	BB	0.5506	3.62134e4	1021.53662	98.4130
2	26.371	BB	0.5881	583.99274	15.10233	1.5870



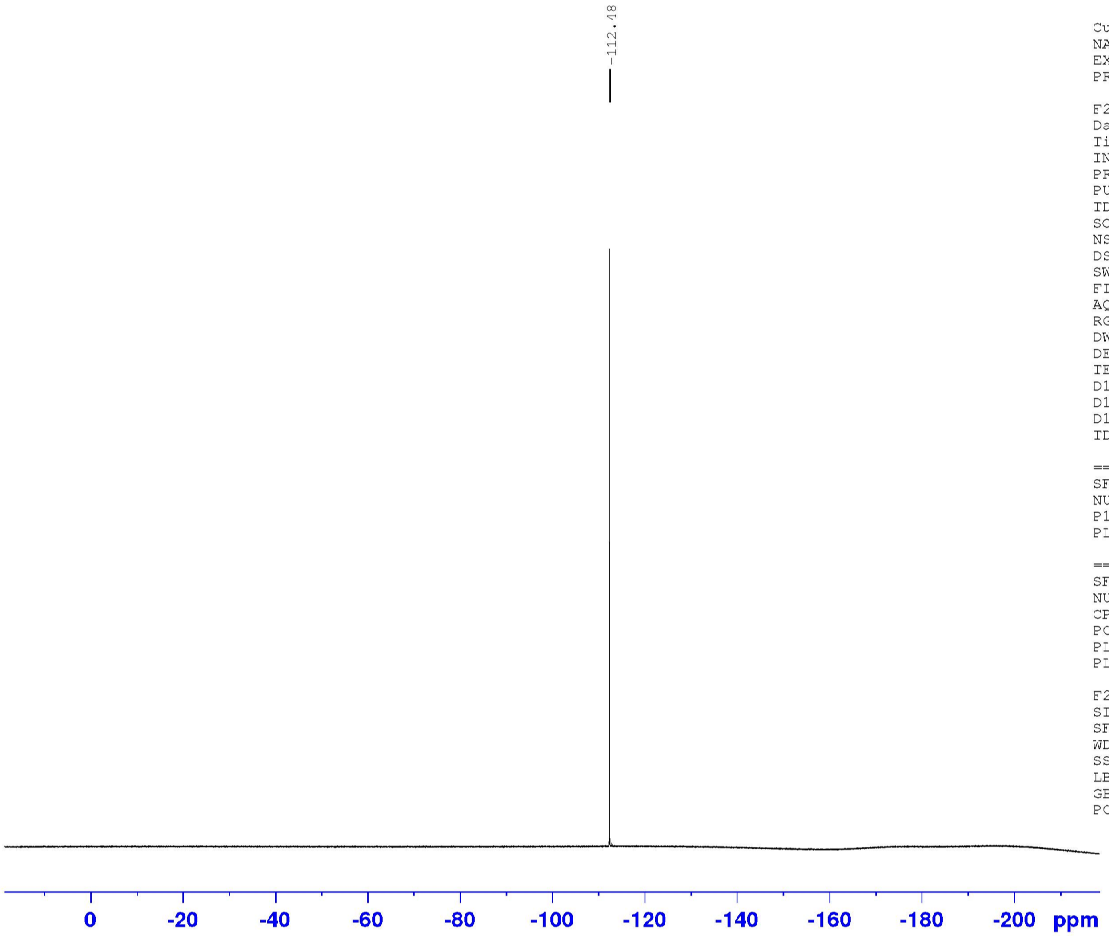
4p



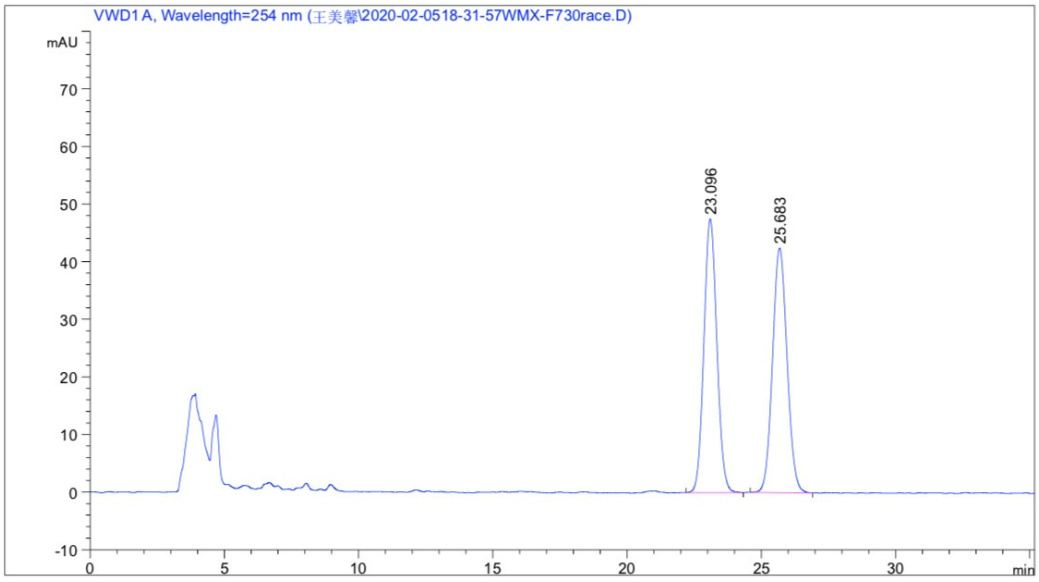

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       353
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                       0.03
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                   8012.820 Hz
FIDRES                0.122266 Hz
AQ                    4.0894465 sec
RG                        97.57
DW                       62.400 usec
DE                        6.50 usec
TE                       292.4 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                       10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                           65536
SF                  400.1500088 MHz
WDW                             EM
SSB                          0
LB                            0.30 Hz
GB                           0
PC                            1.00
```



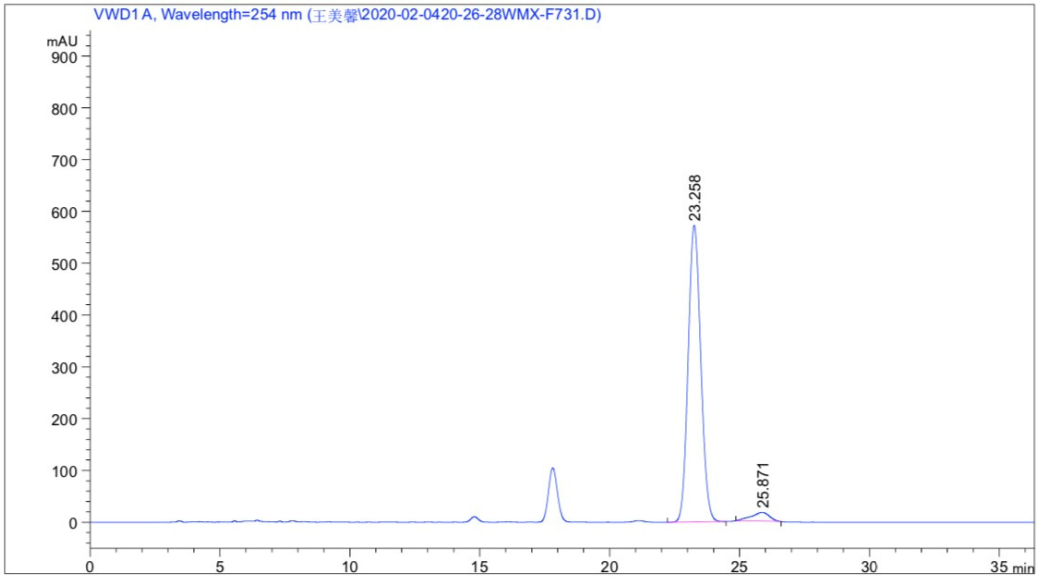

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       356
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                       0.33
INSTRUM                   spect
PROBHD     5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         512
DS                           0
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976128 sec
RG                       195.85
DW                       19.800 usec
DE                        6.50 usec
TE                       292.8 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                       10.50 usec
PLW1                74.00000000 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W
PLW13                0.13149001 W

F2 - Processing parameters
SI                           32768
SF                  100.6177980 MHz
WDW                             EM
SSB                          0
LB                            1.00 Hz
GB                           0
PC                            1.40
```



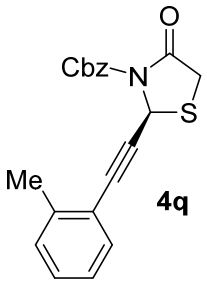

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       357
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                       0.35
INSTRUM                    spect
PROBHD    5 mm PABBO BB/
PULPROG             zgfhigqn.2
TD                        131072
SOLVENT                   CDCl3
NS                           16
DS                            4
SWH                   89285.711 Hz
FIDRES                 0.681196 Hz
AQ                    0.7340032 sec
RG                       195.85
DW                        5.600 usec
DE                        6.50 usec
TE                       292.7 K
D1                    1.00000000 sec
D11                   0.03000000 sec
D12                   0.00002000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                376.4795333 MHz
NUC1                       19F
P1                       14.00 usec
PLW1                24.85000038 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                       1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W

F2 - Processing parameters
SI                        65536
SF                  376.5171850 MHz
WDW                          EM
SSB                 0
LB                         0.30 Hz
GB                  0
PC                         1.00
```



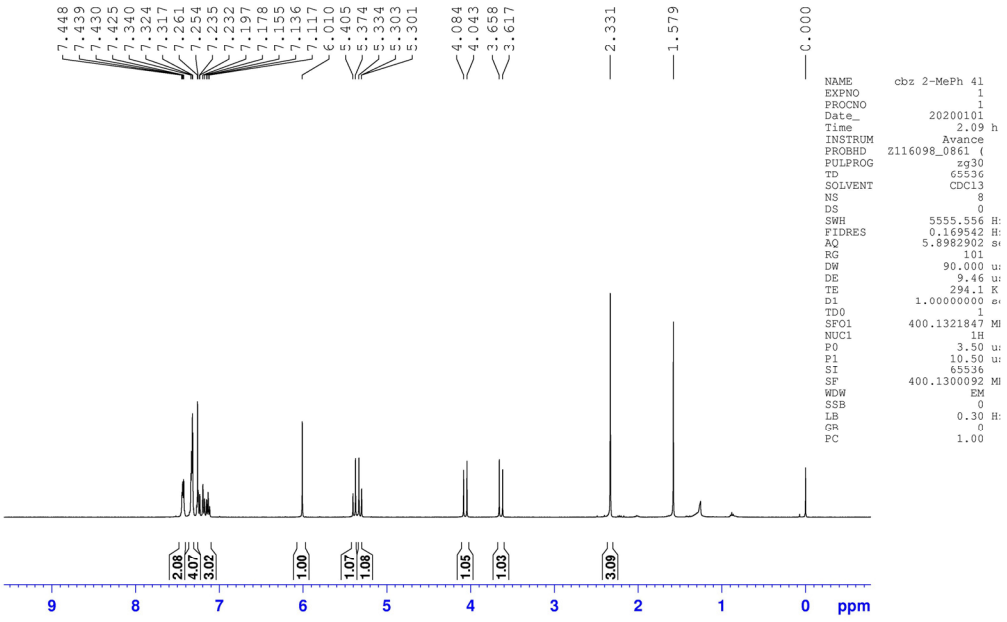
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.096	BB	0.5223	1597.97937	47.52560	49.9958
2	25.683	BB	0.5831	1598.24939	42.46276	50.0042

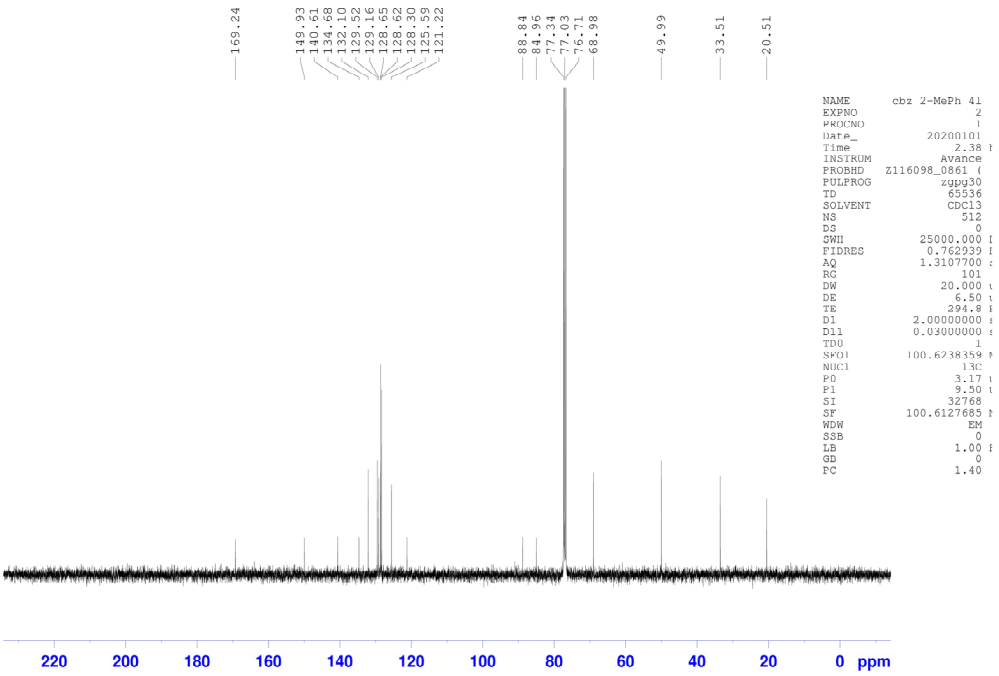


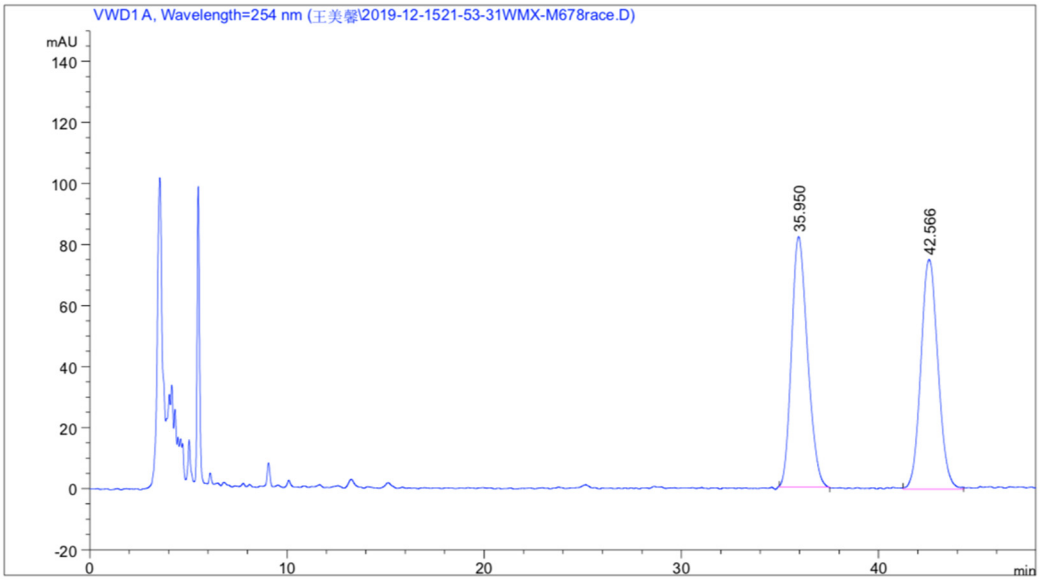
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.258	MM	0.5624	1.93082e4	572.22876	95.9263
2	25.871	MM	0.8075	819.96735	16.92495	4.0737


O
Cbz
N
S
Me

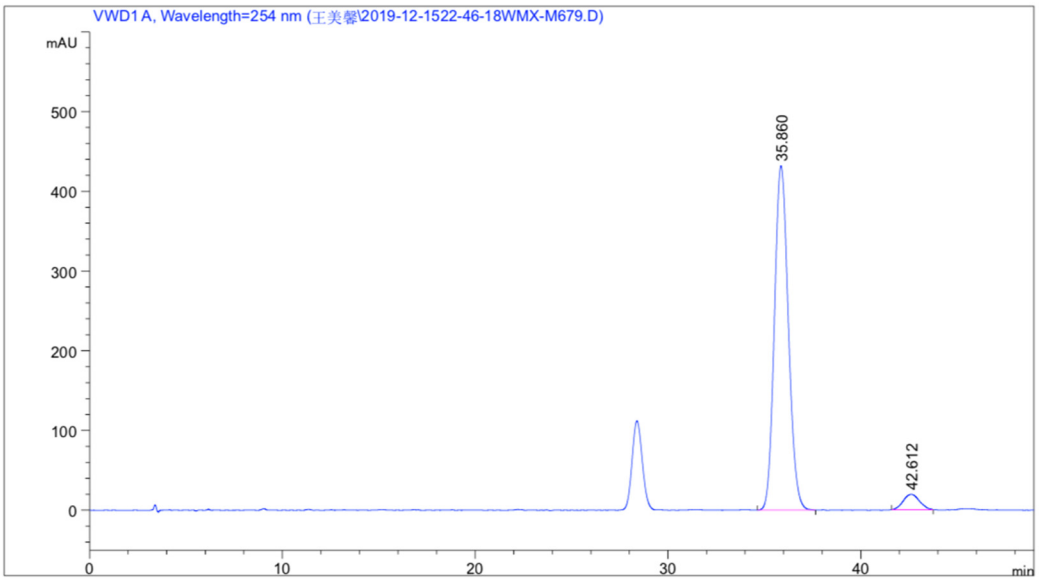
4q


7.448
7.439
7.430
7.425
7.340
7.324
7.317
7.261
7.254
7.235
7.232
7.197
7.178
7.155
7.136
7.117
6.010
5.405
5.374
5.334
5.303
5.301
4.084
4.043
3.658
3.617
2.331
1.579
0.000
2.08
4.07
3.02
1.00
1.07
1.08
1.05
1.03
3.09
9
8
7
6
5
4
3
2
1
0
ppm

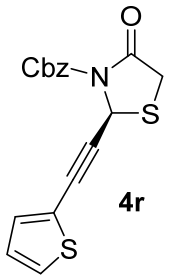

169.24
149.93
140.61
134.68
132.10
129.52
129.16
128.65
128.62
128.30
125.59
121.22
88.84
84.96
77.34
77.03
76.71
68.98
49.99
33.51
20.51
220
200
180
160
140
120
100
80
60
40
20
0
ppm



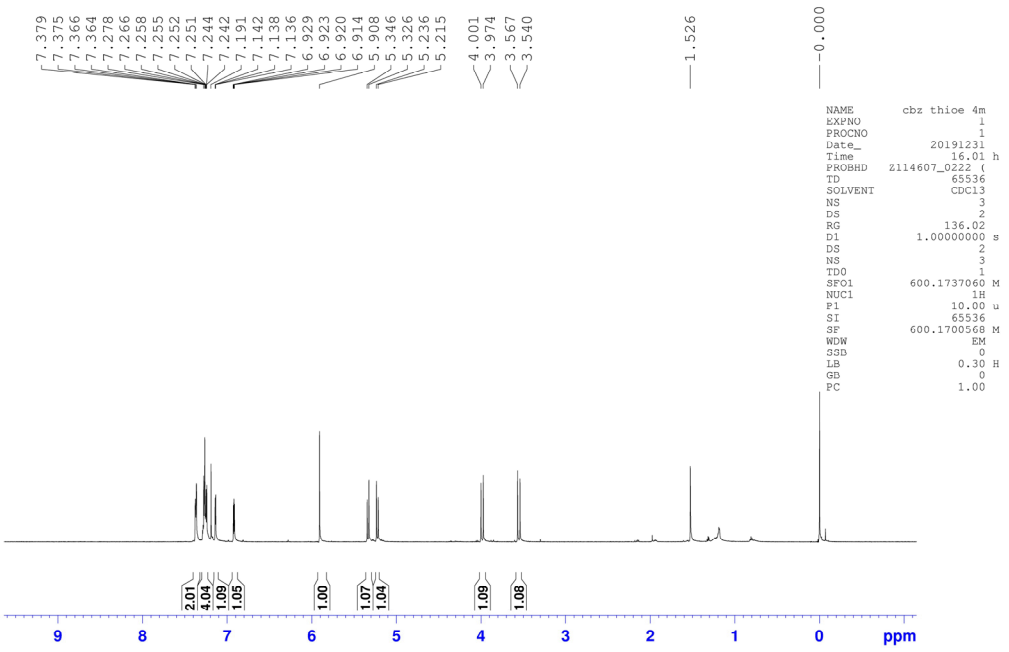
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.950	MM	0.9366	4609.15430	82.02331	50.2875
2	42.566	MM	1.0086	4556.45361	75.29333	49.7125

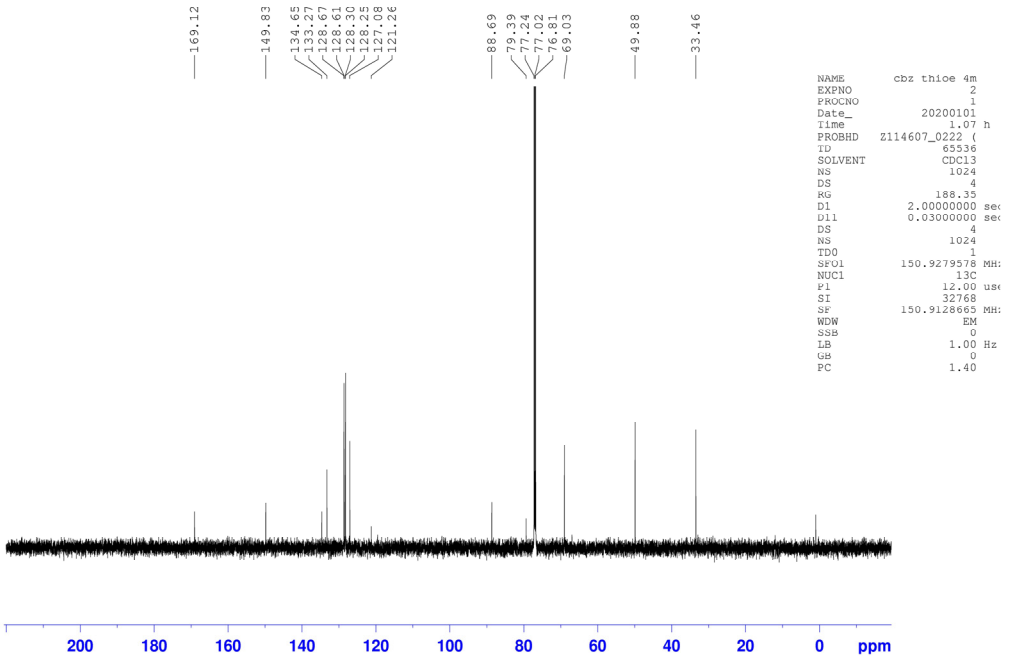


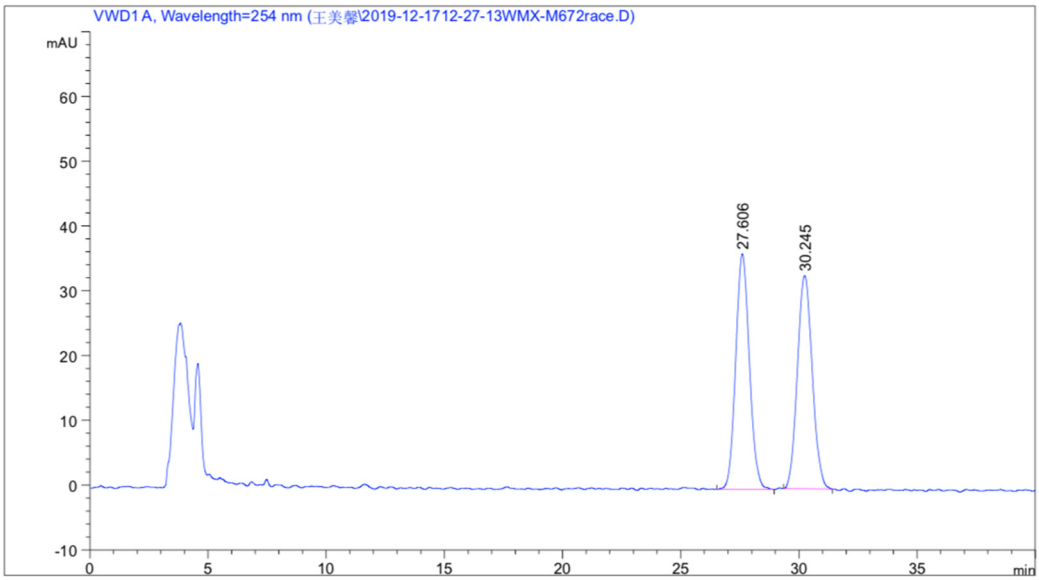
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	35.860	BB	0.7747	2.17116e4	431.74435	95.1075
2	42.612	MM	0.9726	1116.89209	19.13901	4.8925


O
Cbz
N
S
S

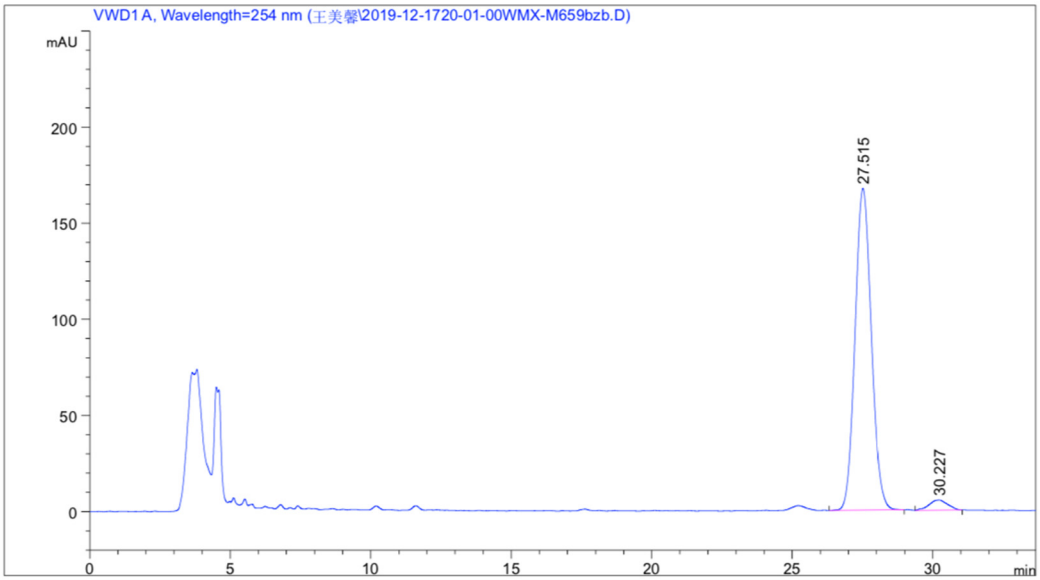
4r


7.379
7.375
7.366
7.364
7.278
7.266
7.258
7.255
7.252
7.251
7.244
7.242
7.191
7.142
7.138
7.136
6.929
6.923
6.920
6.914
5.908
5.346
5.326
5.236
5.215
4.001
3.974
3.567
3.540
1.526
0.000
NAME cbz thioe 4m
EXPNO 1
PROCNO 1
Date_ 20191231
Time 16.01 h
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 3
DS 2
RG 136.02
D1 1.00000000 s
DS 2
NS 3
TD0 1
SFO1 600.1737060 M
NUC1 1H
P1 10.00 u
SI 65536
SF 600.1700568 M
WDW EM
SSB 0
LB 0.30 H
GB 0
PC 1.00
2.01
4.04
1.09
1.05
1.00
1.07
1.04
1.09
1.08
9
8
7
6
5
4
3
2
1
0
ppm

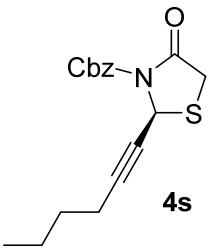

169.12
149.83
134.65
133.27
128.67
128.61
128.30
128.25
127.08
121.26
88.69
79.39
77.24
77.02
76.81
69.03
49.88
33.46
NAME cbz thioe 4m
EXPNO 2
PROCNO 1
Date_ 20200101
Time 1.07 h
PROBHD Z114607_0222 (
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
RG 188.35
D1 2.00000000 sec
D11 0.03000000 sec
DS 4
NS 1024
TD0 1
SFO1 150.9279578 MH
NUC1 13C
P1 12.00 use
SI 32768
SF 150.9128665 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.606	BB	0.6281	1467.72363	36.42567	50.4730
2	30.245	BB	0.6934	1440.21423	32.92329	49.5270

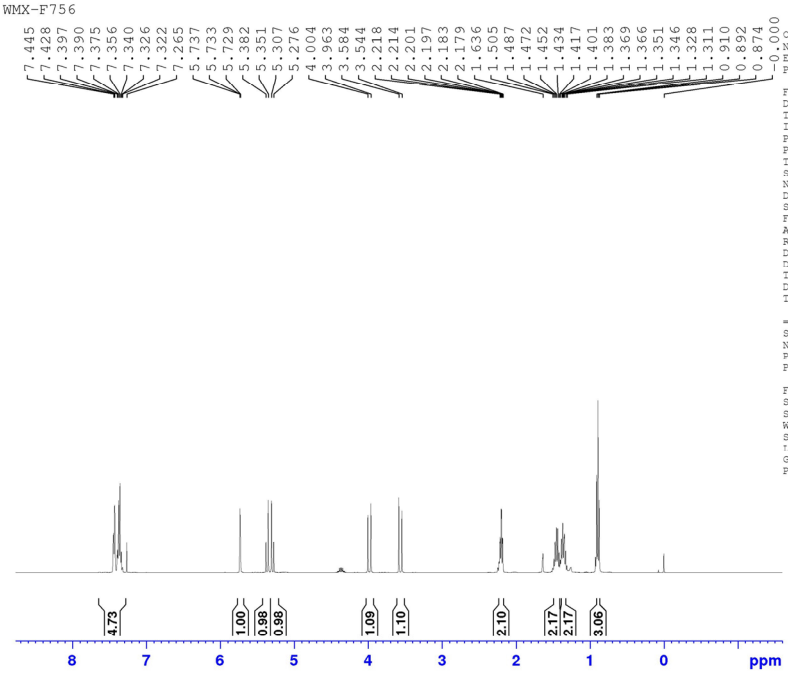


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.515	BB	0.6266	6753.86914	167.42610	96.5429
2	30.227	BB	0.6684	241.85080	5.25987	3.4571



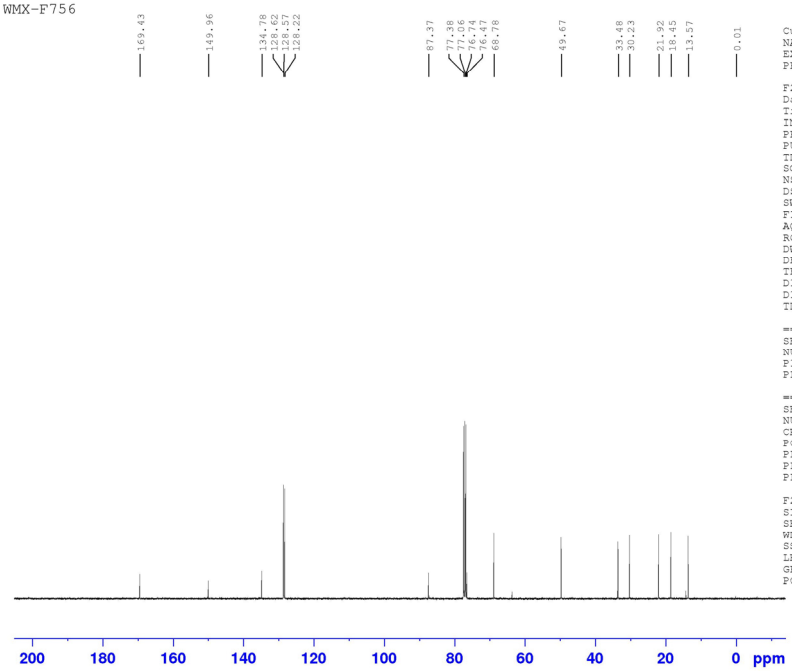
4s

WMX-F756

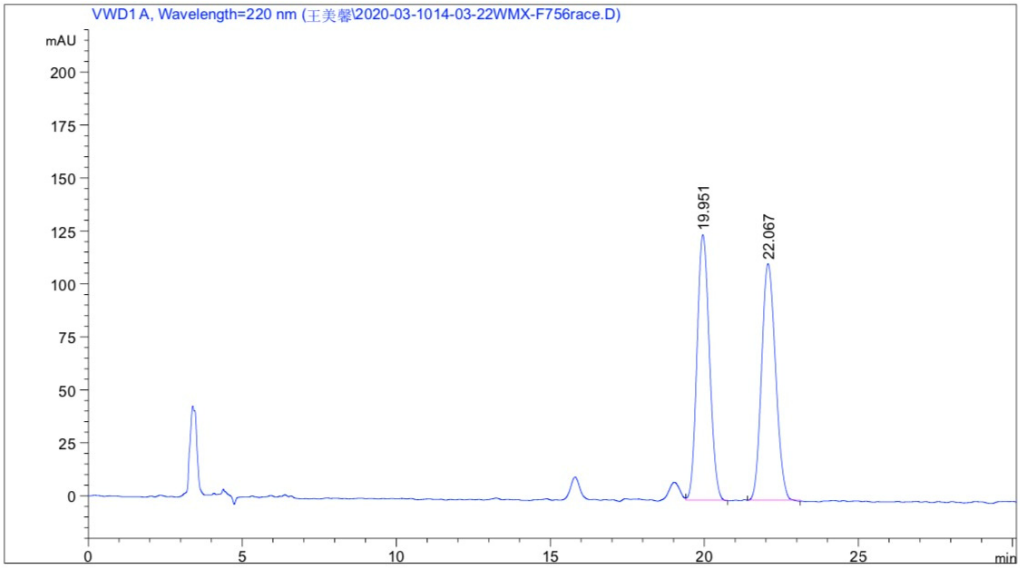



```
Current Data Parameters
NAME                  2020.3.10
EXPNO                       397
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20200310
Time                      12.26
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                   8012.820 Hz
FIDRES                0.122266 Hz
AQ                    4.0894465 sec
RG                       55.34
DW                       62.400 usec
DE                        6.50 usec
TE                       292.9 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                       10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                       65536
SF                  400.1500075 MHz
WDW                         EM
SSB                  0
LB                           0.30 Hz
GB                   0
PC                           1.00
```

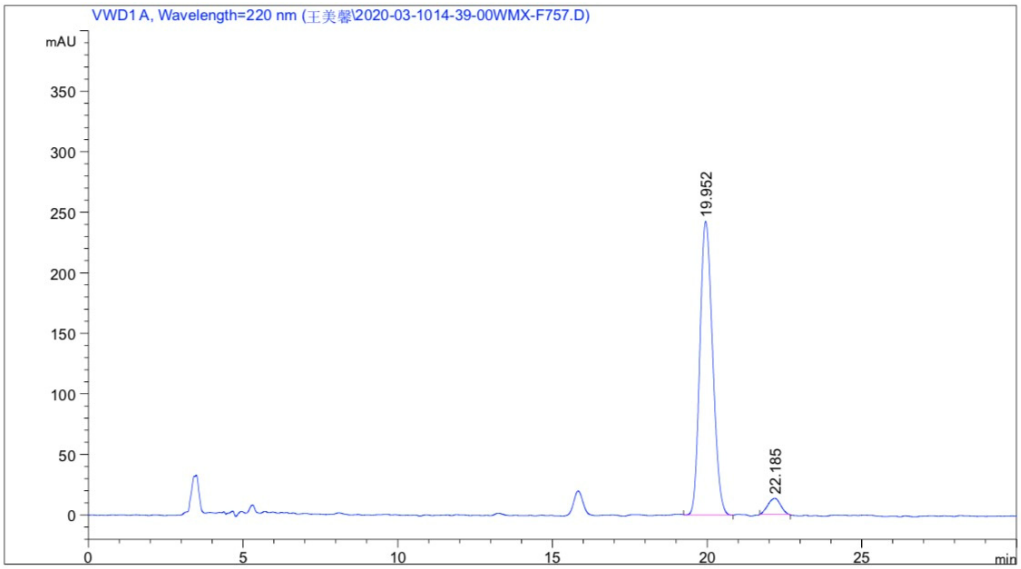
WMX-F756



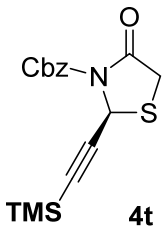

```
Current Data Parameters
NAME                  2020.3.10
EXPNO                       398
PROCNO                        1

F2 - Acquisition Parameters
Date_                 20200310
Time                      12.56
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         512
DS                           0
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976128 sec
RG                      195.85
DW                       19.800 usec
DE                        6.50 usec
TE                       293.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                       10.50 usec
PLW1                74.00000000 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W
PLW13                0.13149001 W

F2 - Processing parameters
SI                       32768
SF                  100.6177980 MHz
WDW                         EM
SSB                  0
LB                           1.00 Hz
GB                   0
PC                           1.40
```



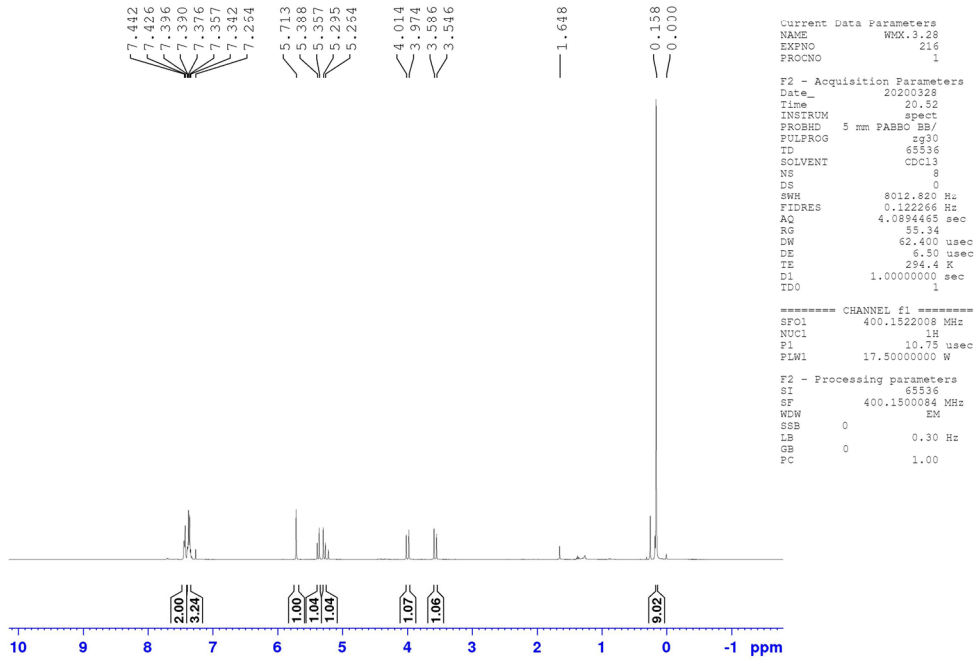
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.951	MM	0.4668	3509.93872	125.31471	49.9484
2	22.067	MM	0.5251	3517.19653	111.63673	50.0516



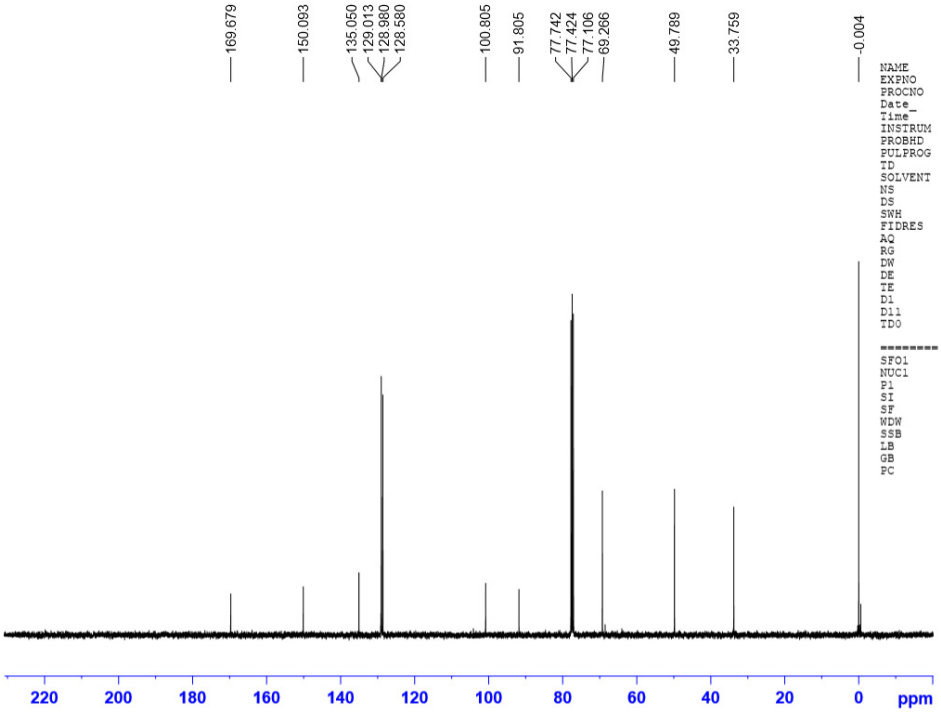
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.952	MM	0.4769	6932.55420	242.29329	94.8412
2	22.185	MM	0.4724	377.08838	13.30504	5.1588



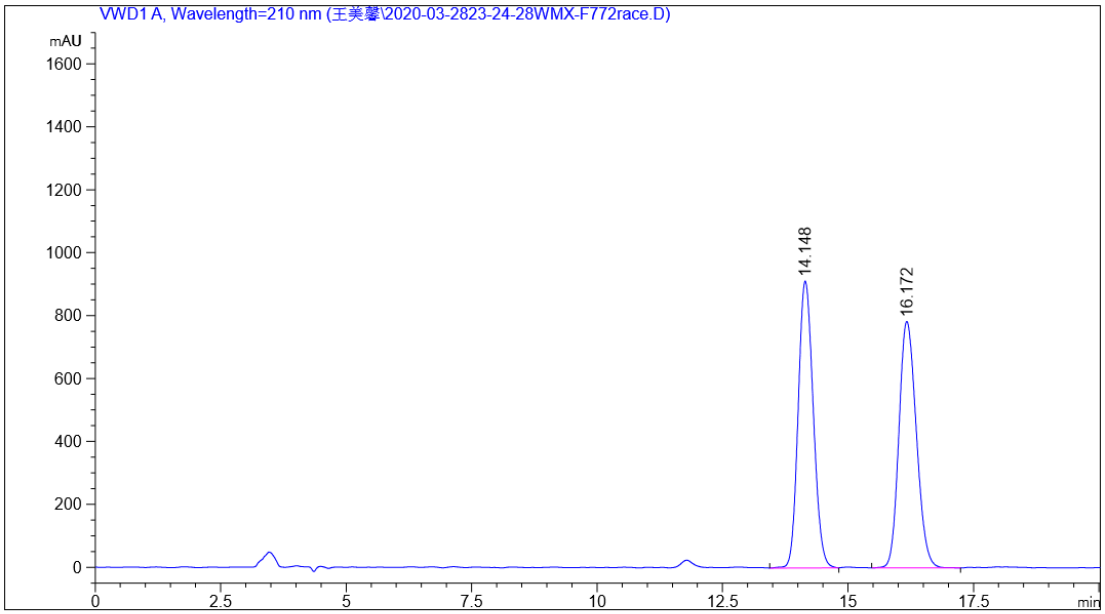
4t



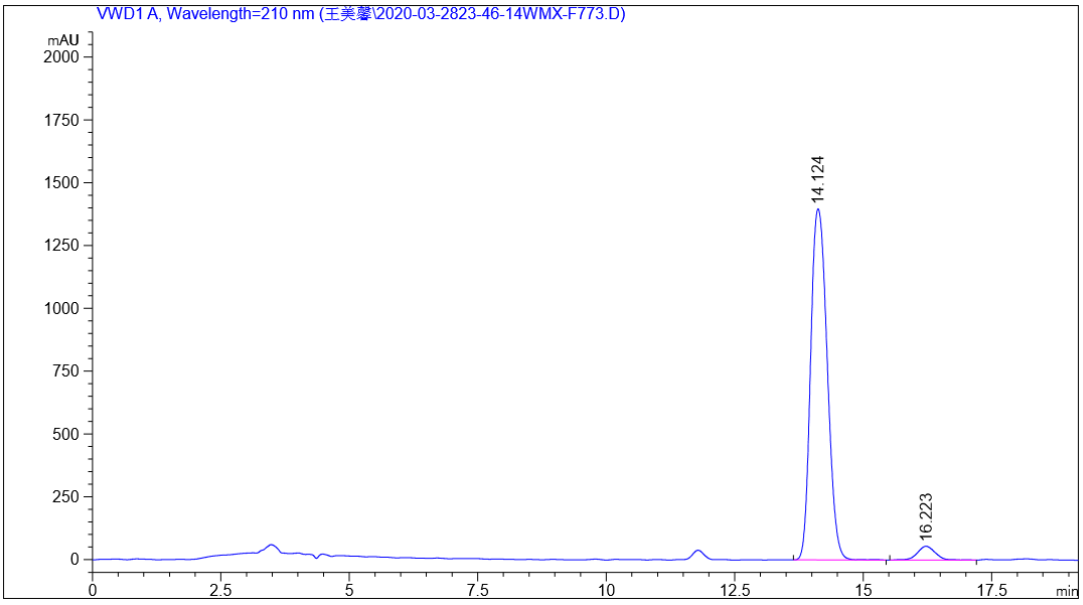

```
Current Data Parameters
NAME                  WMX.3.28
EXPNO                      216
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20200328
Time                     20.52
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                 CDCl3
NS                          8
DS                          0
SWH                  8012.820 Hz
FIDRES               0.122266 Hz
AQ                   4.0894465 sec
RG                       55.34
DW                      62.400 usec
DE                       6.50 usec
TE                      294.4 K
D1                   1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1            400.1522008 MHz
NUC1                       1H
P1                      10.75 usec
PLW1            17.50000000 W

F2 - Processing parameters
SI                       65536
SF              400.1500084 MHz
WDW                         EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```



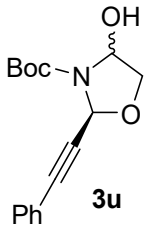

```
NAME                    WMX-2
EXPNO                     217
PROCNO                      1
Date_                20200328
Time                    21.05
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                      65536
SOLVENT                 CDCl3
NS                        220
DS                          0
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 sec
RG                      195.85
DW                      19.800 usec
DE                       6.50 usec
TE                      294.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
SFO1            100.6283629 MHz
NUC1                       13C
P1                      10.50 usec
SI                       32768
SF              100.6177610 MHz
WDW                         EM
SSB                          0
LB                       1.00 Hz
GB                           0
PC                       1.40
```



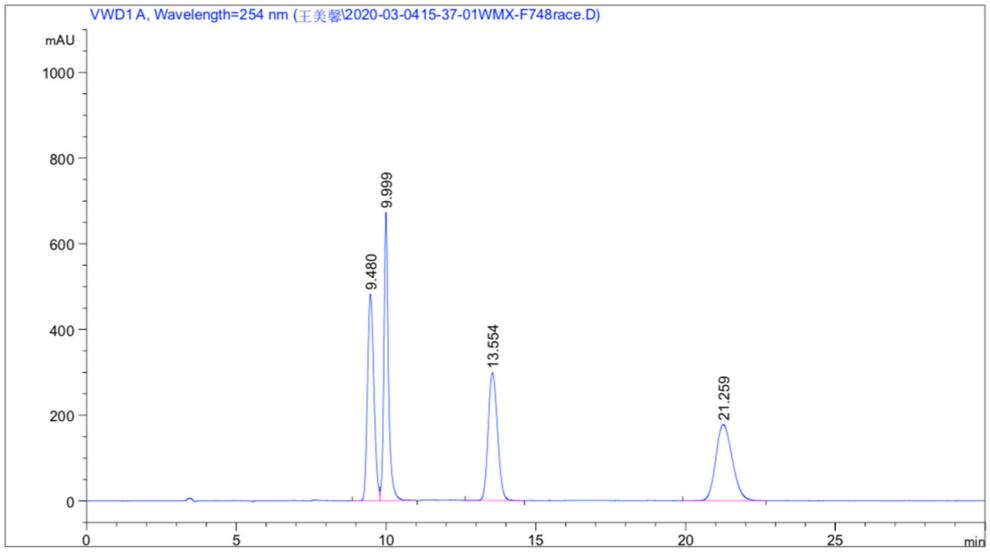
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.148	BB	0.3265	1.90256e4	911.53906	50.1020
2	16.172	BB	0.3781	1.89481e4	783.70044	49.8980



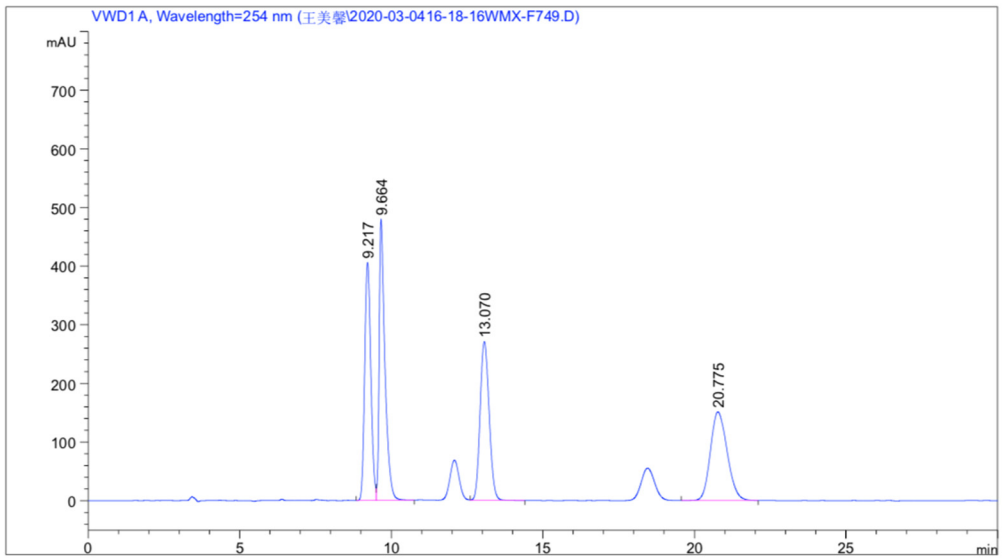
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.124	BV R	0.3561	3.12926e4	1397.09912	95.8703
2	16.223	BV R	0.3753	1347.96045	54.92122	4.1297



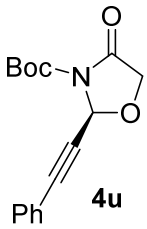
3u



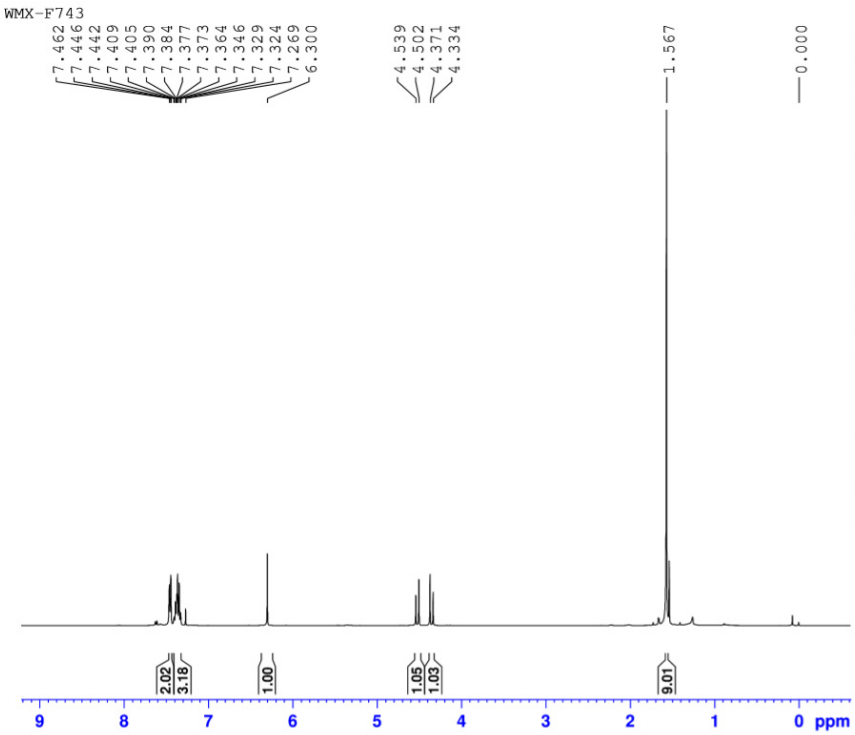
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.480	BV	0.2347	7221.99268	482.30569	26.2441
2	9.999	VB	0.1543	7051.52930	672.68518	25.6246
3	13.554	BB	0.3402	6446.92969	298.25671	23.4276
4	21.259	BB	0.5920	6798.10791	177.81673	24.7037



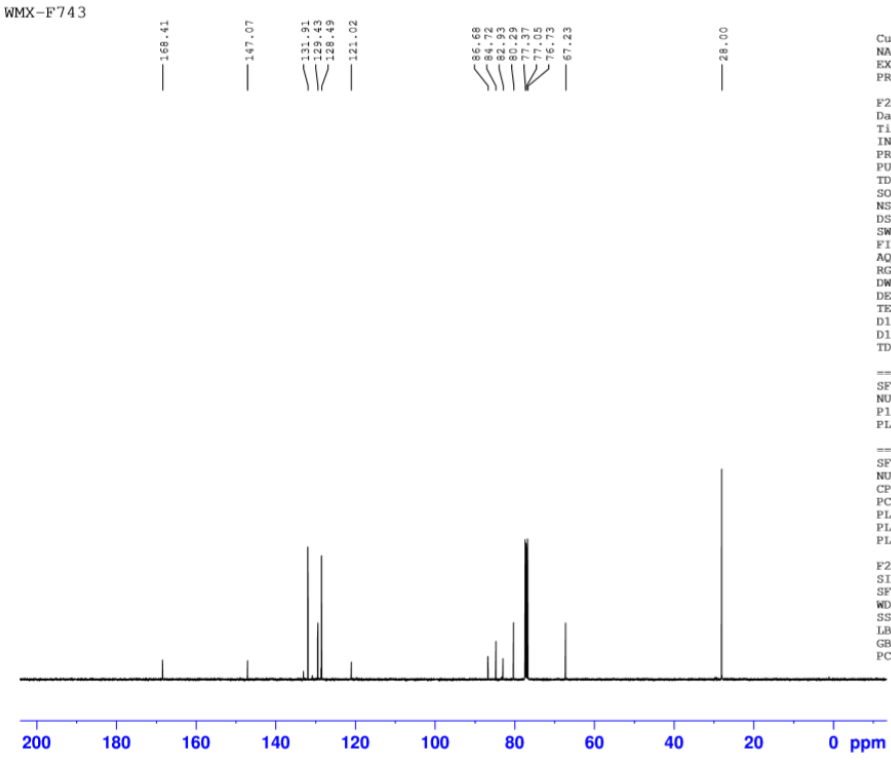
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.217	BV	0.2216	5723.58203	405.72955	24.3750
2	9.664	VB	0.1912	6288.98096	478.80304	26.7829
3	13.070	VB	0.3289	5640.17822	270.86707	24.0198
4	20.775	BB	0.6110	5828.62207	151.44980	24.8223



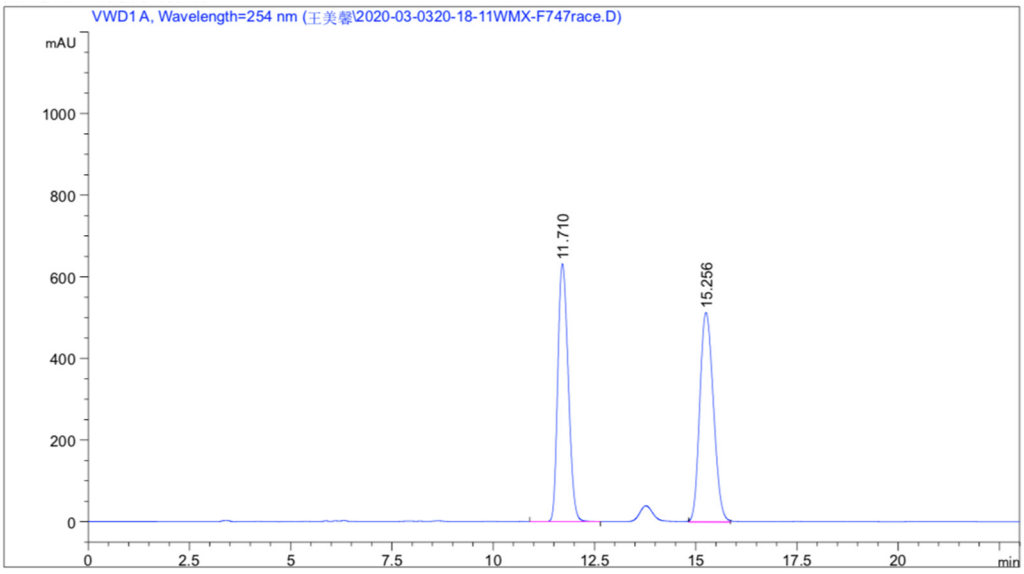
4u



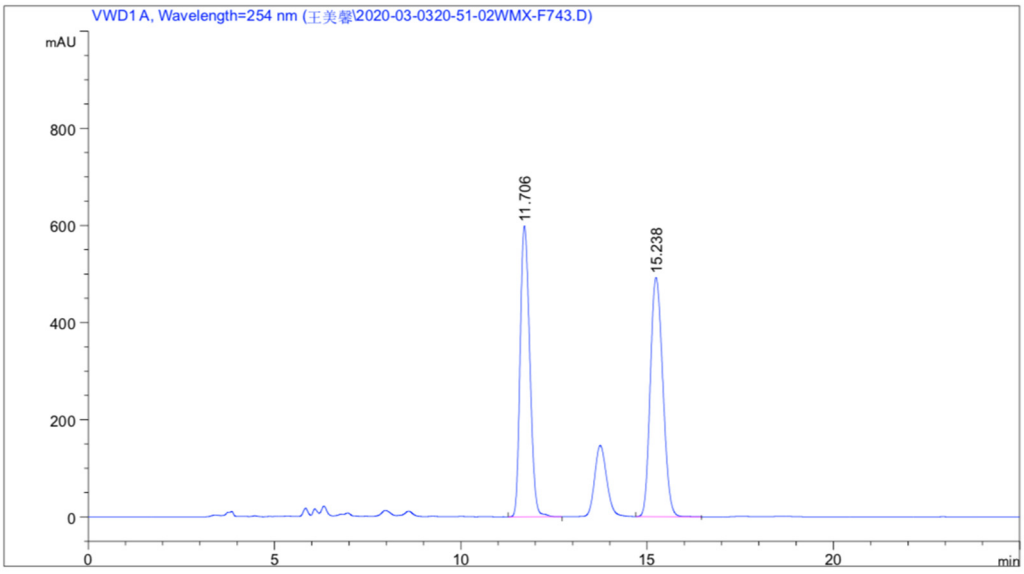

```
Current Data Parameters
NAME                  2020.3
EXPNO                    370
PROCNO                     1

F2 - Acquisition Parameters
Date_              20200302
Time                  14.30
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894465 sec
RG                    55.34
DW                    62.400 usec
DE                     6.50 usec
TE                    292.4 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                       1H
P1                     10.75 usec
PLW1            17.50000000 W

F2 - Processing parameters
SI                        65536
SF               400.1500062 MHz
WDW                         EM
SSB          0
LB                      0.30 Hz
GB           0
PC                      1.00
```



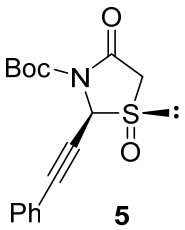

```
Current Data Parameters
NAME                  2020.3
EXPNO                    371
PROCNO                     1

F2 - Acquisition Parameters
Date_              20200302
Time                  14.55
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      436
DS                        0
SWH               25252.525 Hz
FIDRES                0.385323 Hz
AQ                 1.2976128 sec
RG                    195.85
DW                    19.800 usec
DE                     6.50 usec
TE                    293.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1             100.6283629 MHz
NUC1                       13C
P1                     10.50 usec
PLW1            74.00000000 W

======== CHANNEL f2 ========
SFO2             400.1516006 MHz
NUC2                       1H
CPDPRG[2               waltz16
PCPD2                  90.00 usec
PLW2            17.50000000 W
PLW12            0.26142001 W
PLW13            0.13149001 W

F2 - Processing parameters
SI                        32768
SF               100.6177980 MHz
WDW                         EM
SSB          0
LB                      1.00 Hz
GB           0
PC                      1.40
```



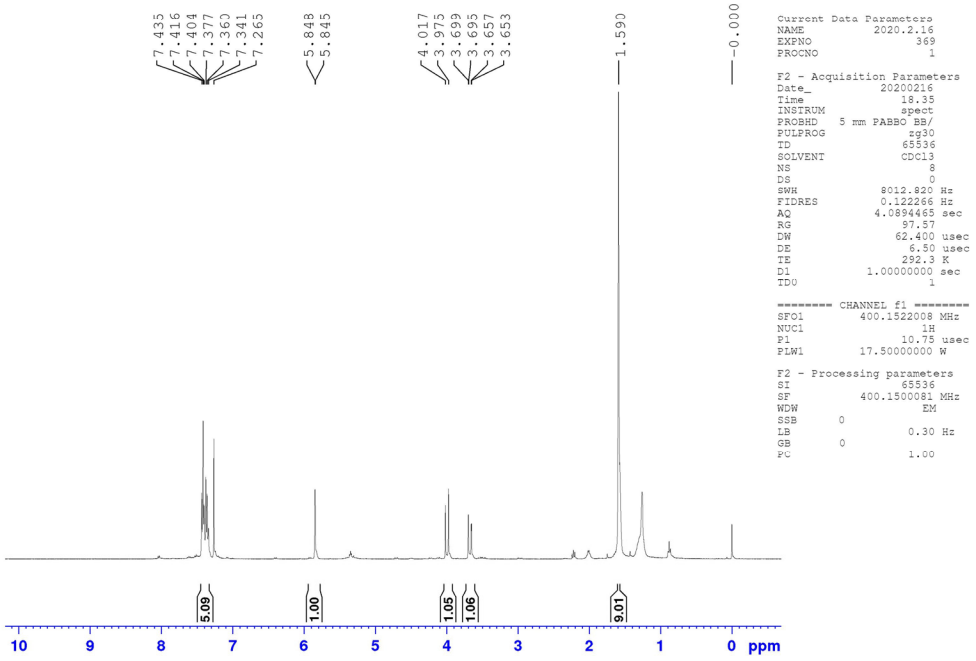
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.710	MM	0.3004	1.14039e4	632.80438	49.3528
2	15.256	MM	0.3839	1.17030e4	508.09836	50.6472

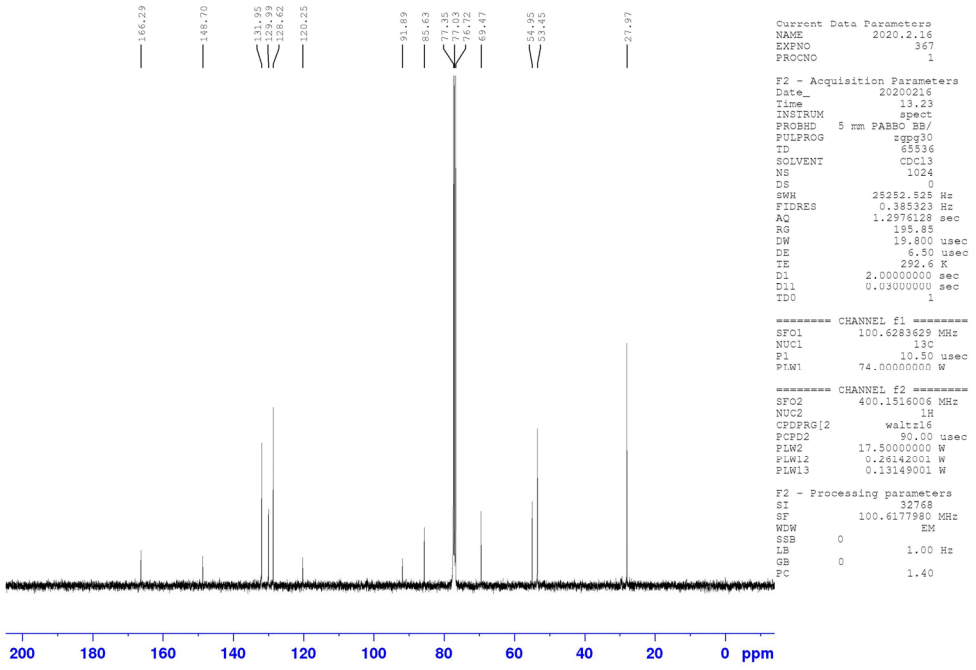


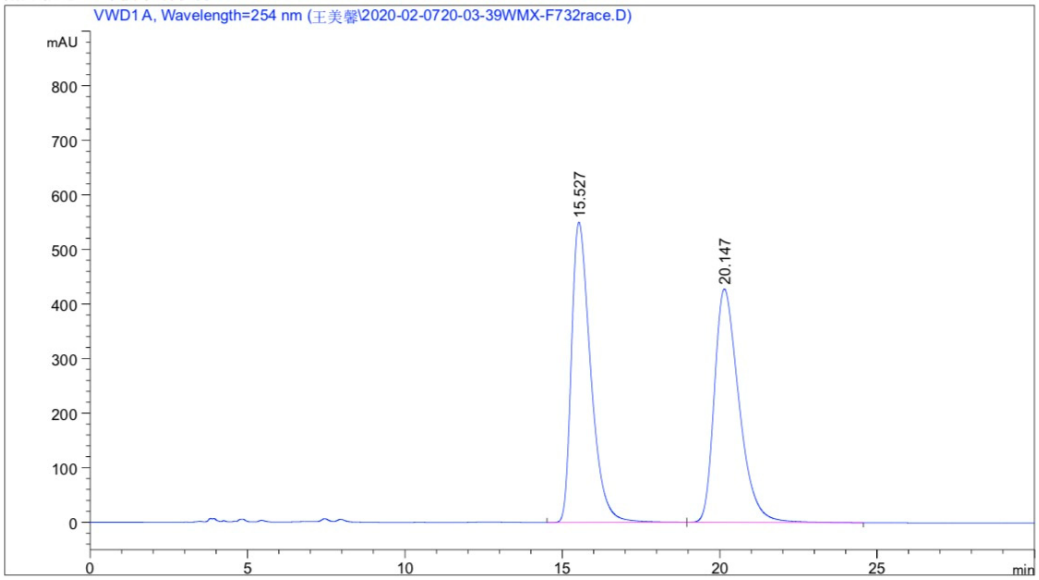
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	11.706	BB	0.2846	1.07054e4	598.63831	48.1397
2	15.238	VB	0.3731	1.15329e4	492.73874	51.8603


O
Boc
N
S
O
Ph

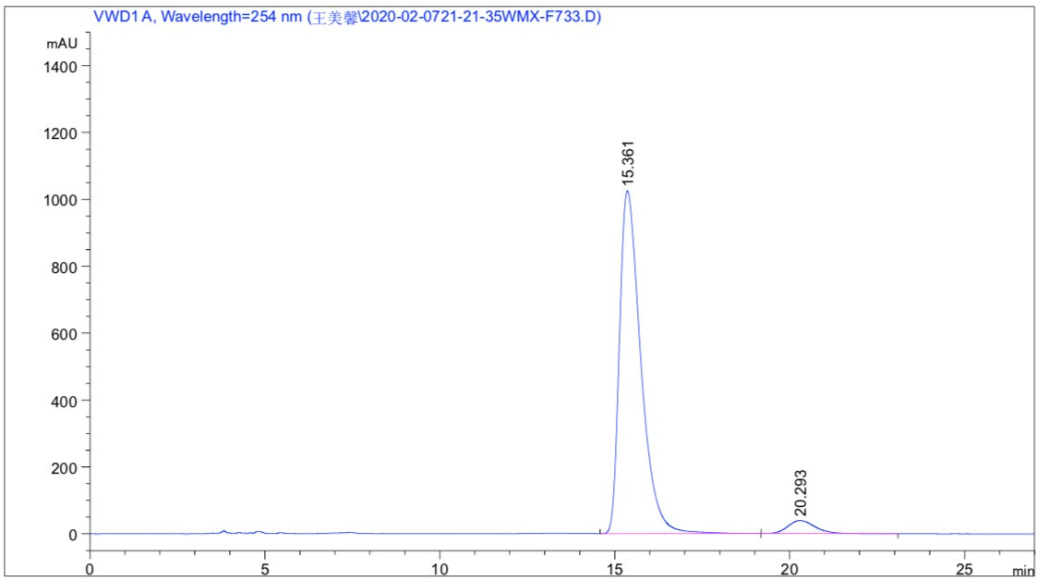
5


7.435
7.416
7.404
7.377
7.360
7.341
7.265
5.848
5.845
4.017
3.975
3.699
3.695
3.657
3.653
1.590
0.000
5.09
1.00
1.05
1.06
9.01
10 9 8 7 6 5 4 3 2 1 0 ppm
Current Data Parameters
NAME 2020.2.16
EXPNO 369
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200216
Time 18.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 97.57
DW 62.400 usec
DE 6.50 usec
TE 292.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1522008 MHz
NUC1 1H
P1 10.75 usec
PLW1 17.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1500081 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

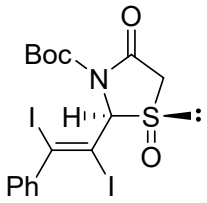

166.29
148.70
131.95
131.39
128.62
120.25
91.89
85.63
77.35
77.03
76.72
69.47
54.95
53.45
27.97
200 180 160 140 120 100 80 60 40 20 0 ppm
Current Data Parameters
NAME 2020.2.16
EXPNO 367
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200216
Time 13.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976128 sec
RG 195.85
DW 19.800 usec
DE 6.50 usec
TE 292.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6283629 MHz
NUC1 13C
P1 10.50 usec
PLW1 74.00000000 W
======== CHANNEL f2 ========
SFO2 400.1516006 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 17.50000000 W
PLW12 0.26142001 W
PLW13 0.13149001 W
F2 - Processing parameters
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



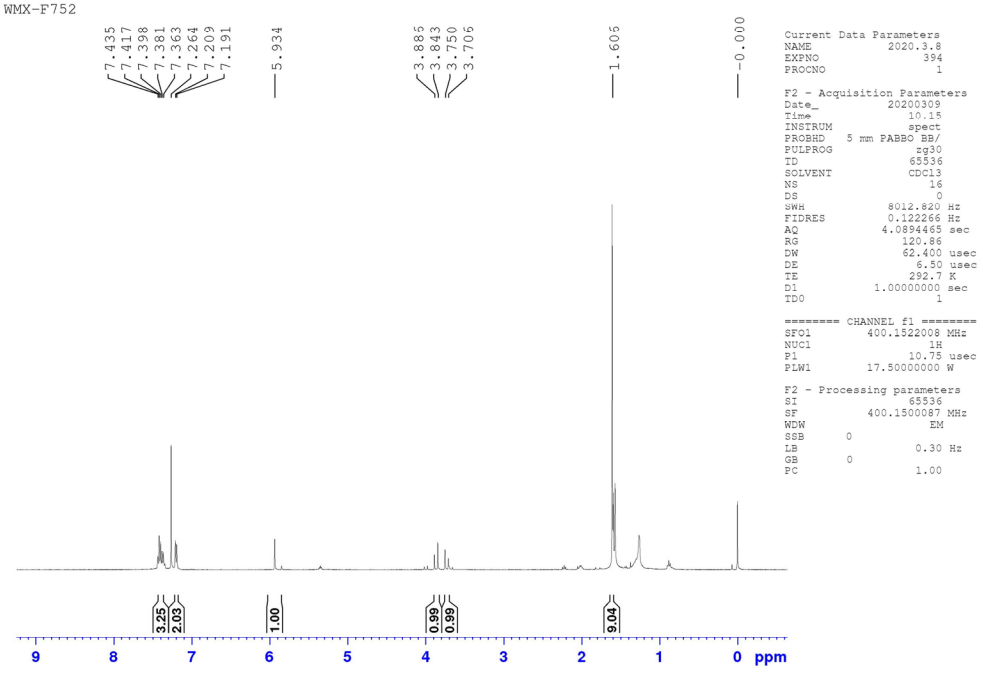
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.527	BB	0.6470	2.32995e4	550.25037	50.0258
2	20.147	BB	0.8296	2.32754e4	427.88611	49.9742

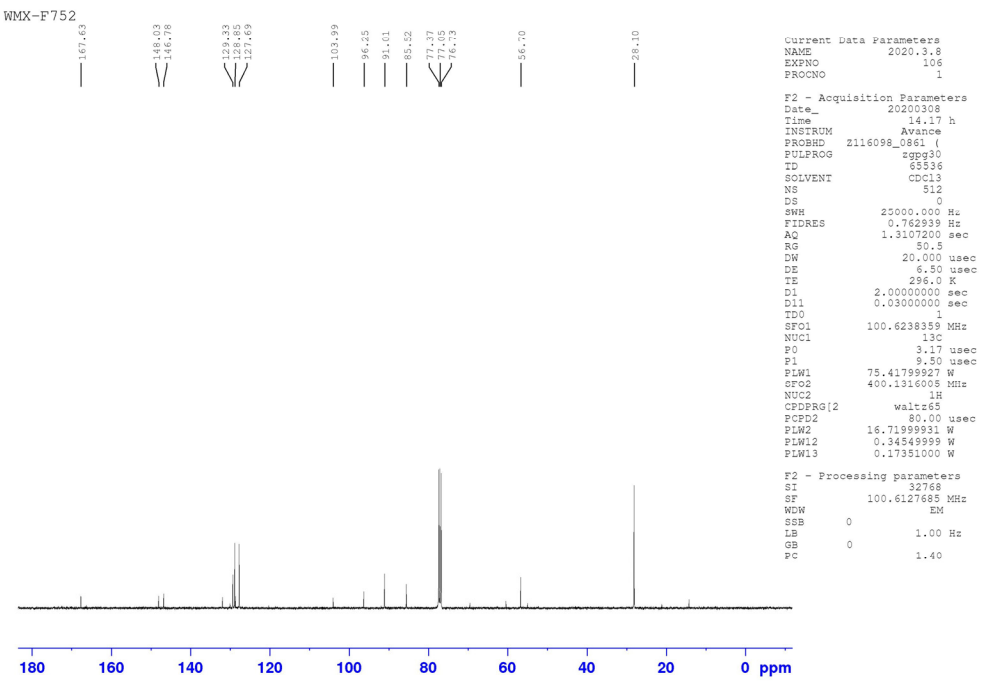


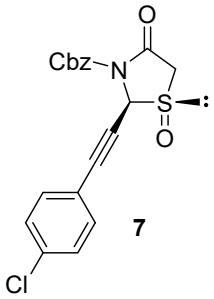
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.361	BB	0.6489	4.34084e4	1025.39343	95.3171
2	20.293	BB	0.8372	2132.66113	38.92548	4.6829


O
Boc
N
H
S
O
I
Ph
I

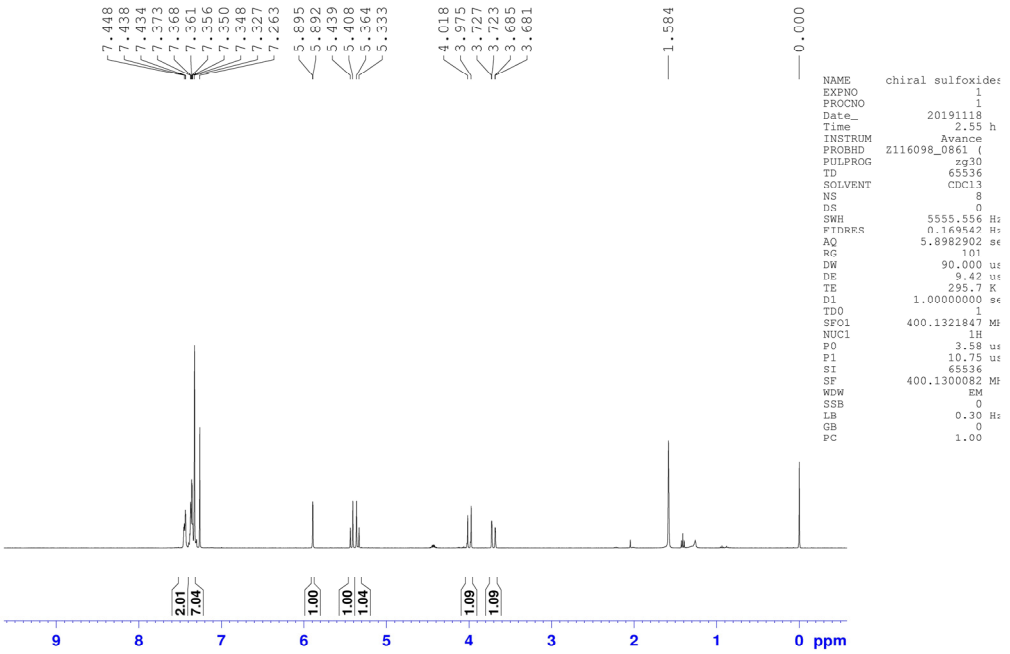
6


WMX-F752
7.435
7.417
7.398
7.381
7.363
7.264
7.209
7.191
5.934
3.885
3.843
3.750
3.706
1.605
0.000
3.25
2.03
1.00
0.99
0.99
9.04
9 8 7 6 5 4 3 2 1 0 ppm
Current Data Parameters
NAME 2020.3.8
EXPNO 394
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200309
Time 10.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 0
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 120.86
DW 62.400 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1522008 MHz
NUC1 1H
P1 10.75 usec
PLW1 17.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1500087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

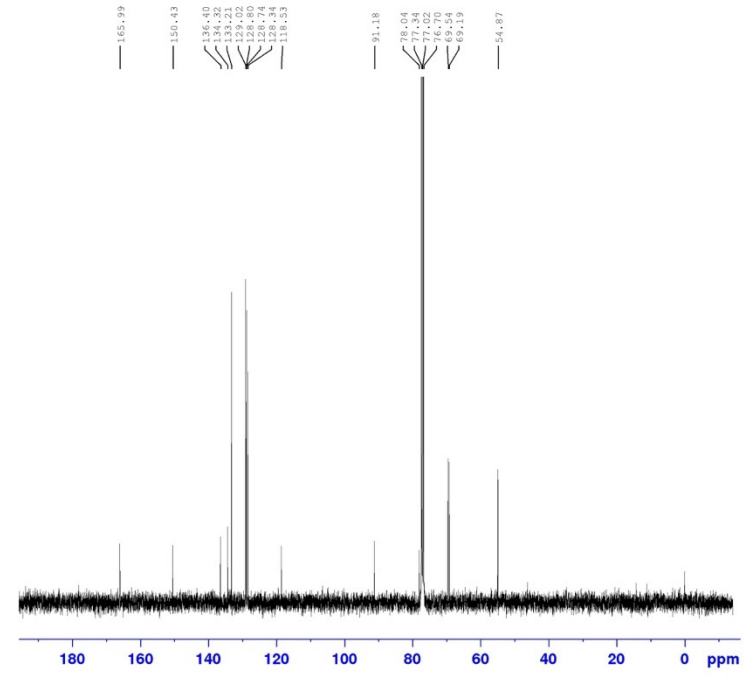

WMX-F752
167.63
148.03
146.78
129.33
128.85
127.69
103.99
96.25
91.01
85.52
77.37
77.05
76.73
56.70
28.10
180 160 140 120 100 80 60 40 20 0 ppm
Current Data Parameters
NAME 2020.3.8
EXPNO 106
PROCNO 1
F2 - Acquisition Parameters
Date_ 20200308
Time 14.17 h
INSTRUM Avance
PROBHD Z116098_0861 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 1.3107200 sec
RG 50.5
DW 20.000 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6238359 MHz
NUC1 13C
P0 3.17 usec
P1 9.50 usec
PLW1 75.41799927 W
SFO2 400.1316005 MHz
NUC2 1H
CPDPRG[2 waltz65
PCPD2 80.00 usec
PLW2 16.71999931 W
PLW12 0.34549999 W
PLW13 0.17351000 W
F2 - Processing parameters
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



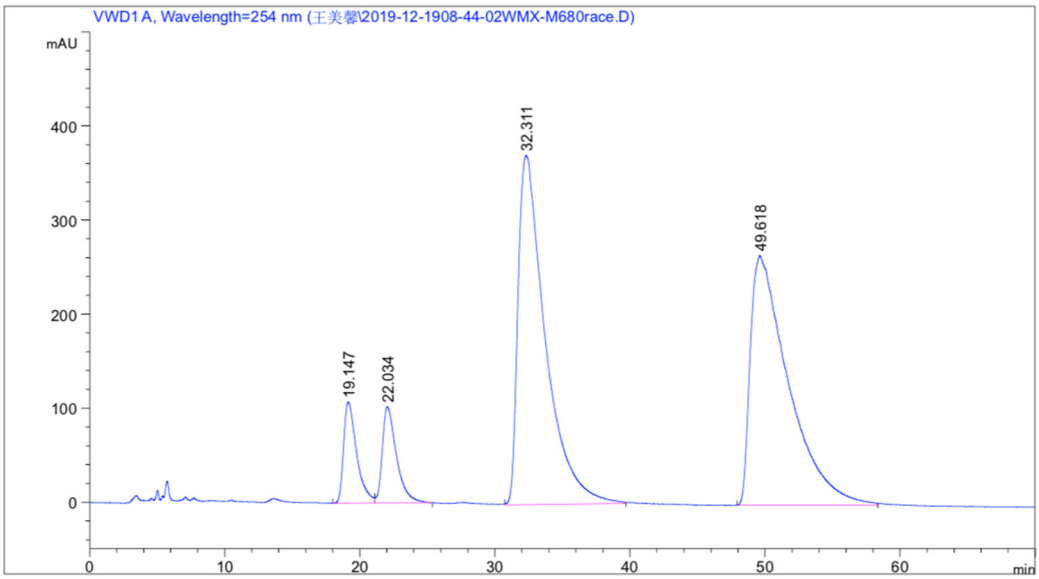
7



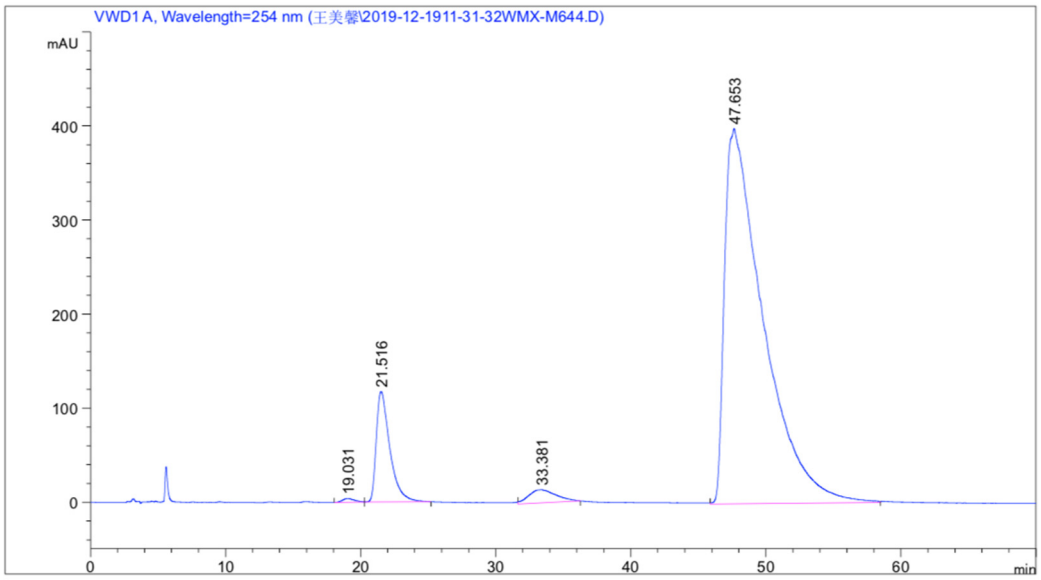

```
NAME      chiral sulfoxides
EXPNO                 1
PROCNO                1
Date_          20191118
Time               2.55 h
INSTRUM          Avance
PROBHD   Z116098_0861 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            5555.556 Hz
FIDRES        0.169542 Hz
AQ             5.8982902 sec
RG                  101
DW               90.000 usec
DE                9.42 usec
TE               295.7 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1321847 MHz
NUC1                 1H
P0                3.58 usec
P1               10.75 usec
SI                65536
SF           400.1300082 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                 1.00
```



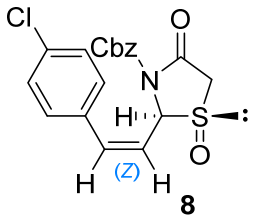

```
Current Data Parameters
NAME                 WMX-LJ
EXPNO                    77
PROCNO                    1

F2 - Acquisition Parameters
Date_              20191118
Time                  3.24 h
INSTRUM              Avance
PROBHD       Z116098_0861 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH             25000.000 Hz
FIDRES              0.762939 Hz
AQ                  1.3107200 sec
RG                      101
DW                   20.000 usec
DE                    6.50 usec
TE                   296.4 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                       1
SFO1           100.6238359 MHz
NUC1                    13C
P0                    3.33 usec
P1                   10.00 usec
PLW1           75.41799927 W
SFO2           400.1316005 MHz
NUC2                    1H
CPDPRG[2             waltz65
PCPD2                80.00 usec
PLW2           16.71999931 W
PLW12           0.37619999 W
PLW13           0.18892001 W

F2 - Processing parameters
SI                    32768
SF             100.6127685 MHz
WDW                      EM
SSB          0
LB                    1.00 Hz
GB           0
PC                    1.40
```



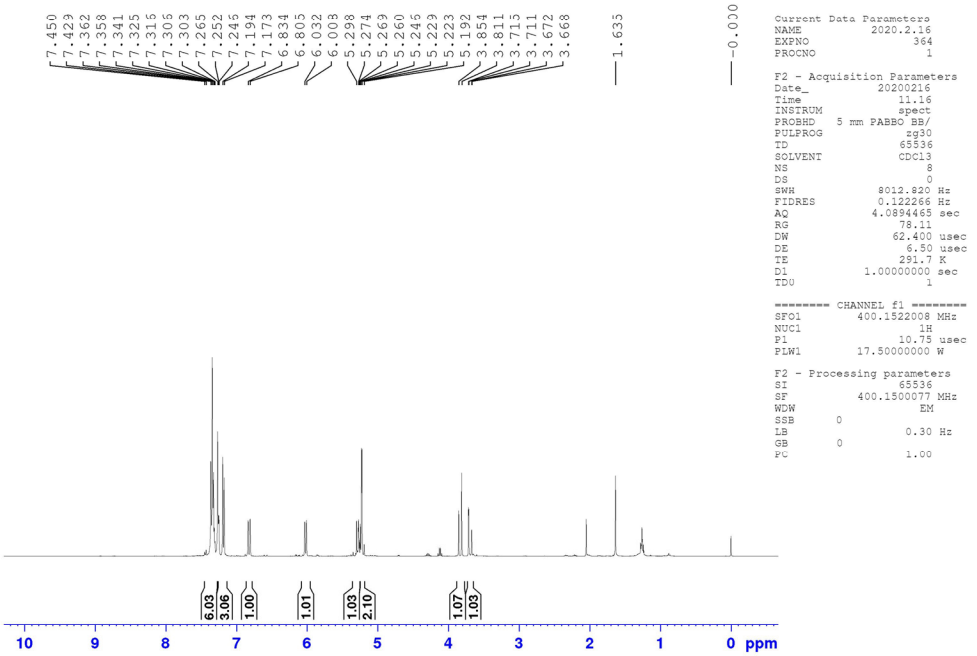
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.147	BV	0.9995	7229.78125	107.24380	6.0940
2	22.034	VB	1.1002	7473.35889	102.19559	6.2993
3	32.311	MM	2.3371	5.20481e4	371.16742	43.8717
4	49.618	MM	3.2601	5.18860e4	265.25854	43.7350



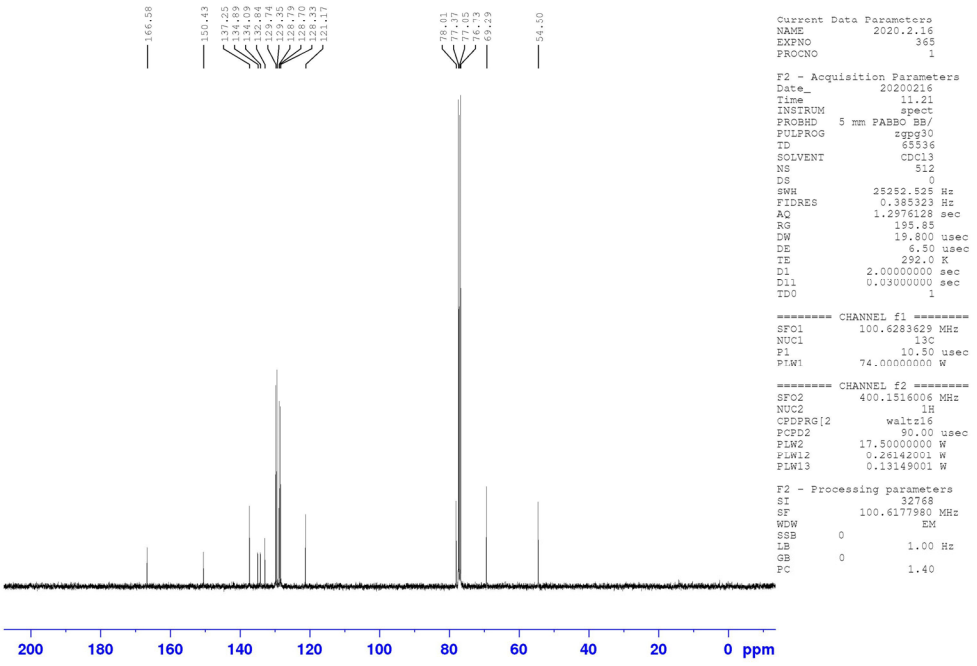
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	19.031	BB	0.7389	235.23035	3.91565	0.2623
2	21.516	BB	1.0036	8187.18652	117.25873	9.1282
3	33.381	MM	2.3505	1965.86218	13.93932	2.1918
4	47.653	MM	3.3133	7.93029e4	398.90836	88.4177



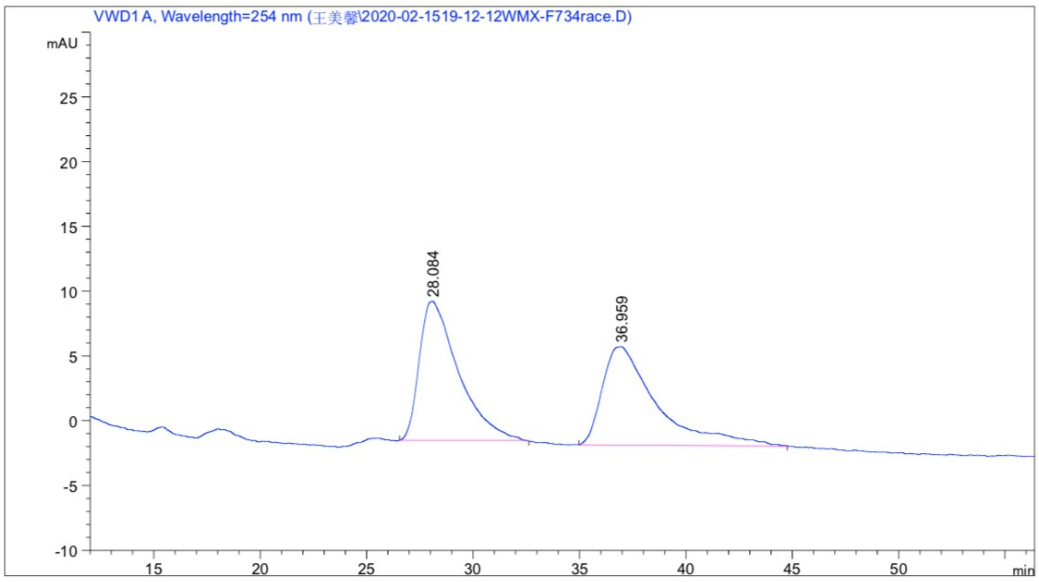
8



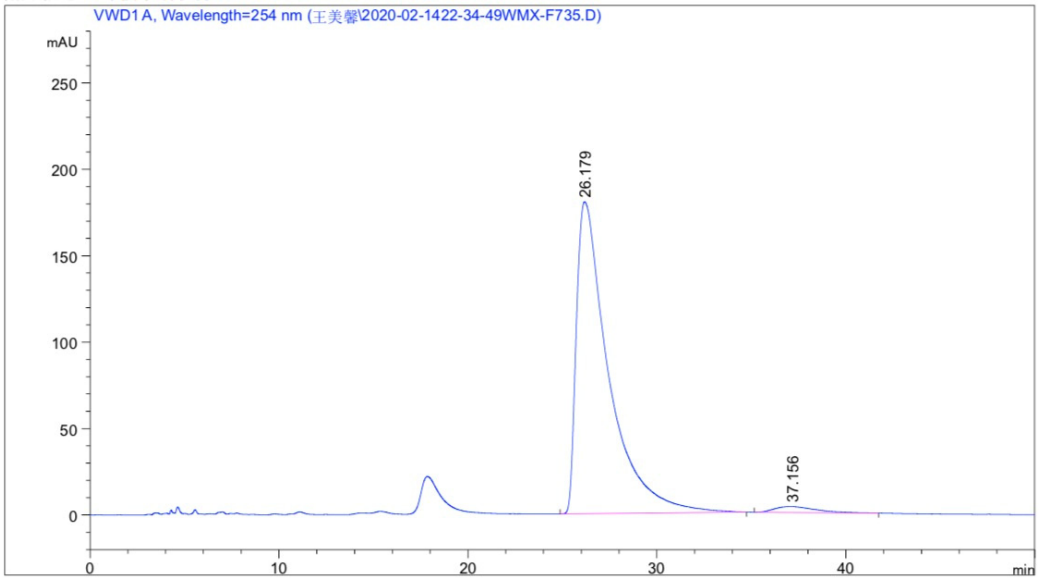

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       364
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                      11.16
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           0
SWH                   8012.820 Hz
FIDRES                0.122266 Hz
AQ                    4.0894465 sec
RG                        78.11
DW                       62.400 usec
DE                        6.50 usec
TE                       291.7 K
D1                    1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                400.1522008 MHz
NUC1                        1H
P1                       10.75 usec
PLW1                17.50000000 W

F2 - Processing parameters
SI                       65536
SF                  400.1500077 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```



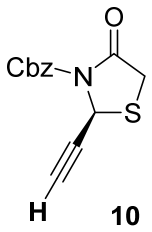

```
Current Data Parameters
NAME                  2020.2.16
EXPNO                       365
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20200216
Time                      11.21
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         512
DS                           0
SWH                  25252.525 Hz
FIDRES                0.385323 Hz
AQ                    1.2976128 sec
RG                       195.85
DW                       19.800 usec
DE                        6.50 usec
TE                       292.0 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                        13C
P1                       10.50 usec
PLW1                74.00000000 W

======== CHANNEL f2 ========
SFO2                400.1516006 MHz
NUC2                        1H
CPDPRG[2                waltz16
PCPD2                    90.00 usec
PLW2                17.50000000 W
PLW12                0.26142001 W
PLW13                0.13149001 W

F2 - Processing parameters
SI                       32768
SF                  100.6177980 MHz
WDW                         EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```



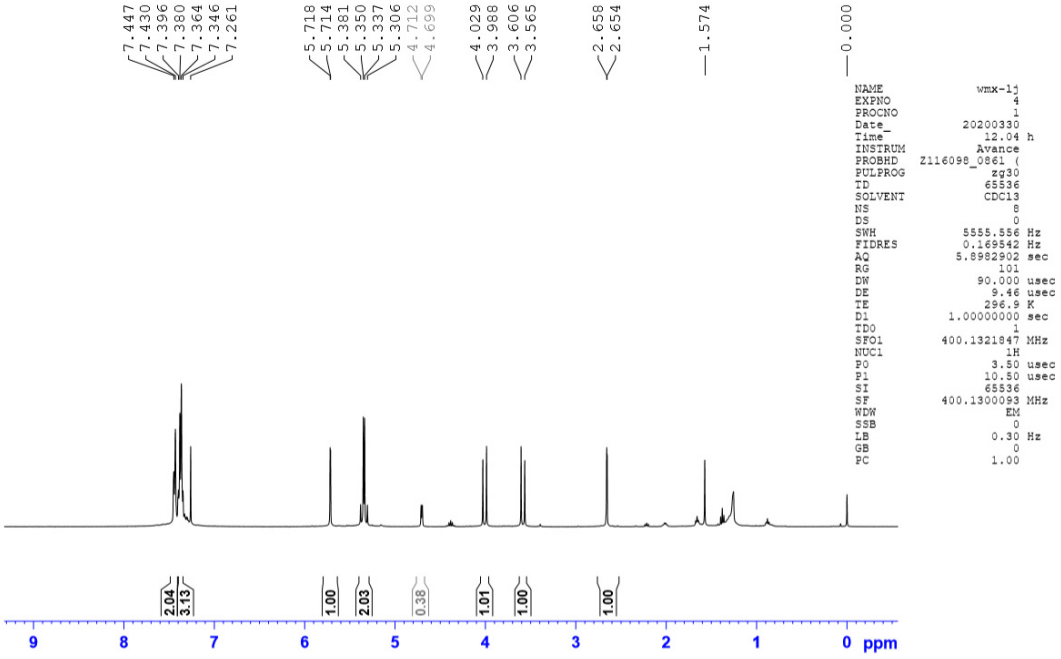
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.084	MM	2.1298	1371.97095	10.73640	50.3942
2	36.959	MM	2.9604	1350.50488	7.60326	49.6058

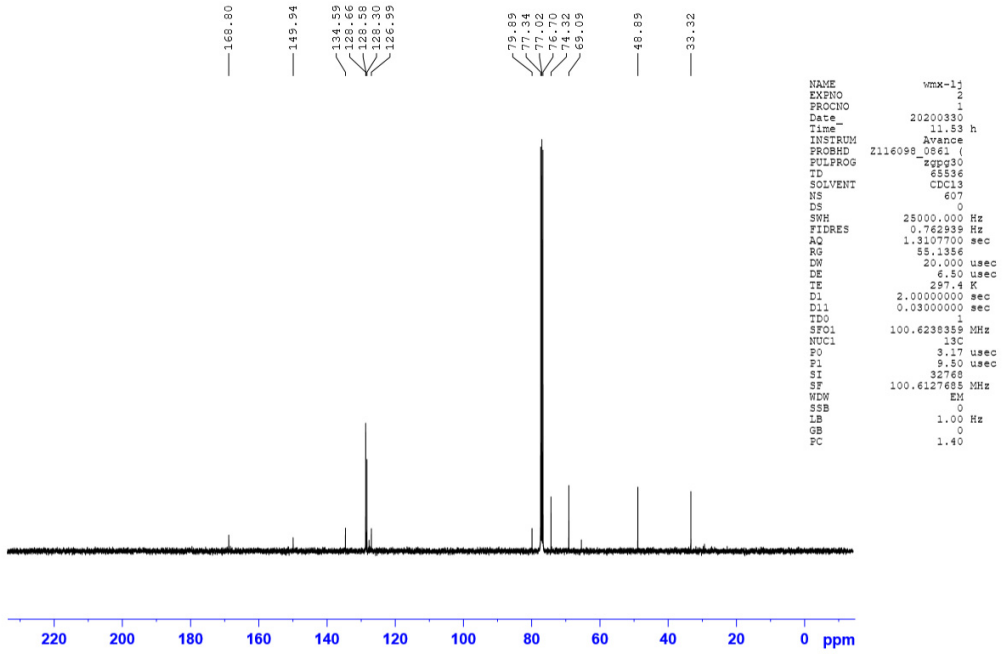


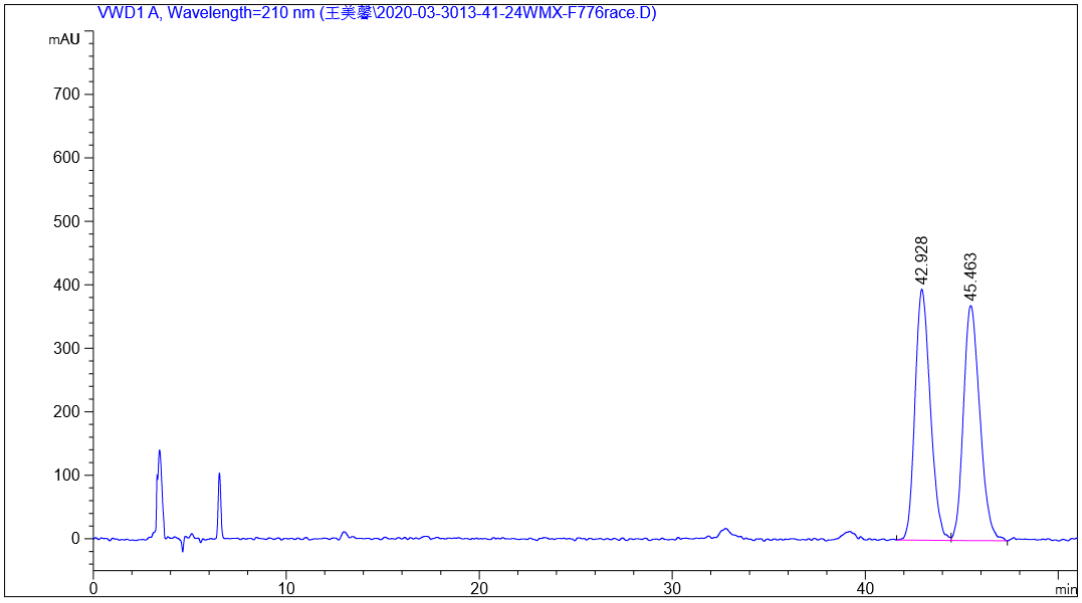
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	26.179	BB	1.6495	2.14007e4	180.47250	97.5867
2	37.156	BB	1.7846	529.22498	3.51686	2.4133


O
Cbz
N
S
H

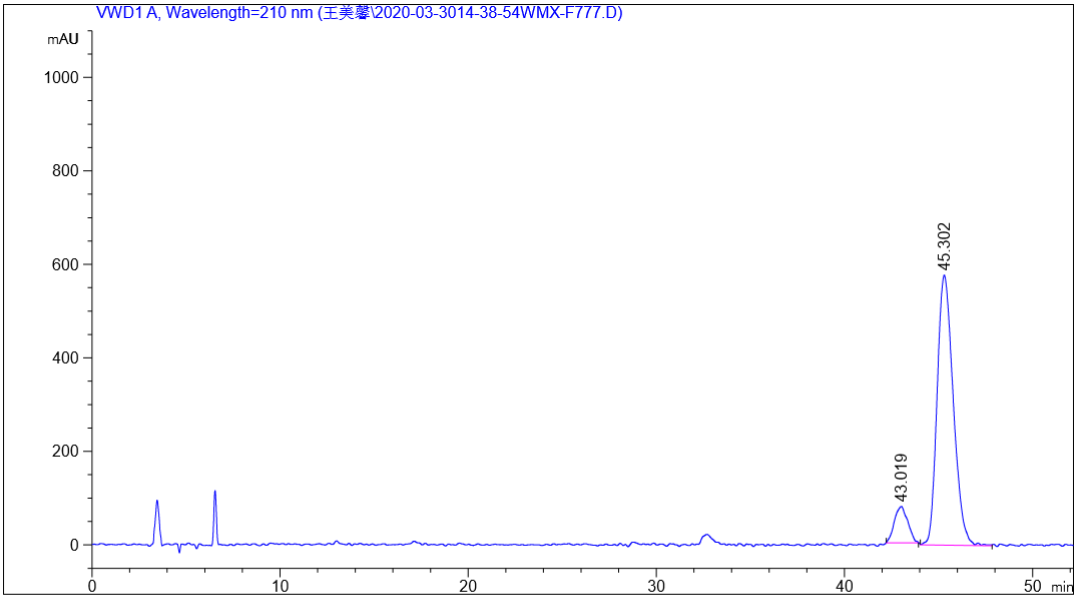
10


7.447
7.430
7.396
7.380
7.364
7.346
7.261
5.718
5.714
5.381
5.350
5.337
5.306
4.712
4.699
4.029
3.988
3.606
3.565
2.658
2.654
1.574
0.000
NAME wmx-1j
EXPNO 4
PROCNO 1
Date_ 20200330
Time_ 12.04 h
INSTRUM Avance
PROBHD Z116098_0861 (
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 5555.556 Hz
FIDRES 0.169542 Hz
AQ 5.8982902 sec
RG 101
DW 90.000 usec
DE 9.46 usec
TE 296.9 K
D1 1.00000000 sec
TD0 1
SFO1 400.1321847 MHz
NUC1 1H
P0 3.50 usec
P1 10.50 usec
SI 65536
SF 400.1300093 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
2.04
3.13
1.00
2.03
0.38
1.01
1.00
1.00
9
8
7
6
5
4
3
2
1
0 ppm


168.80
149.94
134.59
128.66
128.58
128.30
126.99
79.89
77.34
77.02
76.70
74.32
69.09
48.89
33.32
NAME wmx-1j
EXPNO 2
PROCNO 1
Date_ 20200330
Time_ 11.53 h
INSTRUM Avance
PROBHD Z116098_0861 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 607
DS 0
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 1.3107700 sec
RG 55.1356
DW 20.000 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 100.6238359 MHz
NUC1 13C
P0 3.17 usec
P1 9.50 usec
SI 32768
SF 100.6127685 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
220
200
180
160
140
120
100
80
60
40
20
0 ppm



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	42.928	BV	0.8494	2.17864e4	395.03601	49.8800
2	45.463	VV R	0.9016	2.18912e4	369.90900	50.1200



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	43.019	MM	0.8253	3860.05103	77.95095	10.0980
2	45.302	MM	0.9918	3.43657e4	577.50226	89.9020