Supporting information

Enantioselective Peroxidation of *C*-Alkynyl Imines Enabled by Chiral BINOL Calcium Phosphate

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Contents:

1.General information……..…S2

2. General procedure for synthesis of *C*-alkynyl-*N*-Boc-*N,O*-acetals **1**…… S2

3. General procedure for synthesis of *hydroperoxides* **2**…….……..S2

4.Catalytic asymmetric synthesis of chiral *C*-alkynyl *α*-amino peroxides **3**, **4**…..S3

5.Characterization data of the products **3**, **4** ……..…S4

6. Catalytic asymmetric synthesis of chiral *C*-aryl *α*-amino peroxides 6……...….S25

7. Characterization data of the products **6**…….…….……..…..S26

8.Large-Scale catalytic asymmetric reactions……..…….....S27

9. General procedure for synthesis and characterization data of the products 7….....S27

10. The experiments of non-linear effect and product ee dependence on time……..S29

11. Proposed transition states for the asymmetric peroxidation of C-alkynyl imines.....S30

12. Determination of the Product Stereochemistry……S30

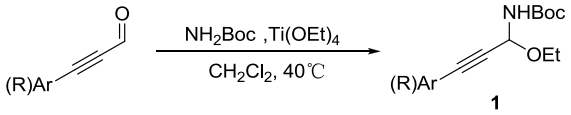
13. References…….....….....S32

14. Copies of NMR and HPLC……..S33

General information

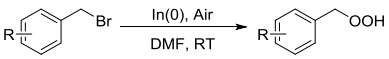
^{1}H-NMR, ^{13}C-NMR and ^{19}F-NMR spectra were recorded on Bruker Avance 400 MHz or spectrophotometer. Chemical shift (δ) are expressed in ppm, and *J* values are given in Hz. The enantiomeric excess was determined by HPLC using Chiralpak AD-H, Chiralpak IC-3, Chiralpak ID-3 columns with *n*-Hexane and 2-propanol as eluents. High-resolution mass spectrometry (HRMS) was recorded on a VG Auto Spec-3000 spectrometer. Optical rotations were measured on a JASCO DIP-370p olarimeter at 589 nm at 20℃. Flash column chromatography was performed on silica gel (200-300 mesh, Qingdao Marine Chemical Inc.). Toluene, diethyl ether, and THF was distilled from sodium benzophenone ketyl immediately prior to use. MeCN, $CHCl_3$, and CH_2Cl_2 were all distilled from CaH_2 immediately prior to use. Unless otherwide noted, all chemicals and solvents were purchased from Adama-beta[@], Energy Chemical *et al.* and used as received without further purification. Chiral phosphoric acids were purchased from Daicel Chiral Technologies Co., LTD. *C*-alkynyl-*N*-Boc-*N,O*-acetals **1**[1]were prepared according to the reported procedures. Hydroperoxides **2** were purchased directly or prepared according to the reported procedures[2].

General procedure for synthesis of *C*-alkynyl-*N*-Boc-*N,O*-acetals 1



To a solution of ynal (10 mmol) in dichloromethane (80 ml) under an argon atmosphere was added $Ti(OEt)_4$ (4.26 g, 15 mmol) and $BocNH_2$ (1.76 g, 15 mmol) at room temperature. The reaction mixture was stirred under an argon atmosphere at 40℃ for 12 h. The reaction was quenched by the addition of saturated Na_2SO_4 solution (20 mL). The resulting white precipitate was isolated, and the aqueous fraction was further extracted with CH_2Cl_2 (20 mL x 3). The combined organic fraction was dried over Na_2SO_4 and the solvent was evaporated to give a residue. The residue was purified by flash column chromatography using AcOEt/hexane as an eluent to afford *C*-alkynyl-*N*-Boc-*N,O*-acetal **1**.

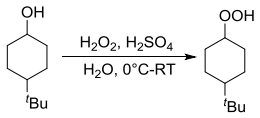
General procedure for synthesis of hydroperoxides 2



To a 5 ml vial equipped with a stirring bar, benzyl bromide (0.3 mmol) was dissolved in DMF (1 ml), indium powder (45 mg, 0.39 mmol) was added into the mixture. The resulted mixture was

then stirred at room temperature and monitored by TLC. After consumption of starting material, the reaction mixture was quenched with water, extracted with ethyl acetate, purified by flash chromatography (silica gel, *n*-hexanes/EtOAc) to provide the desired product.

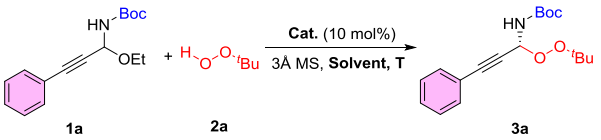
General procedure for synthesis of hydroperoxides 2p.



To a cooled (0 ℃) solution of H_2O_2 (65 ml, 1.1 mol, 50% wt in H_2O) and H_2SO_4 (1.0 mL, 19 mmol) was added 4-(tert-butyl)cyclohexan-1-ol (42 mmol). The reaction mixture was stirred vigorously at ambient temperature for 14 hours and then was partitioned between Et_2O (100 mL) and water (100 mL). The aqueous layer was extracted with Et_2O (2 × 50 mL) and the combined organic layers were washed with 1 N NaOH (3 × 25 mL) and brine (50 mL). The resulting solution was dried over $MgSO_4$ and concentrated *in vacuo* to yield hydroperoxide as a white solid.

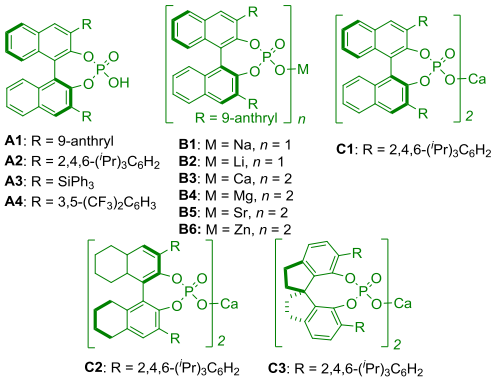
Catalytic asymmetric synthesis of chiral *C*-alkynyl *α*-amino peroxides 3 and 4

Table S1. Screening of optimal condition for the *oxa*-Mannich reaction



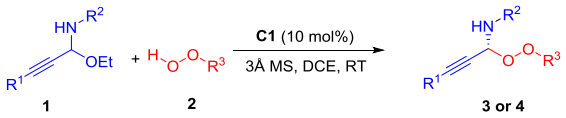
entry	catalyst	solvent	Yield (%)[b]	ee (%)[c]
1	A1	DCM	90	2
2	A2	DCM	93	5
3	A3	DCM	92	4
4	A4	DCM	91	3
5	B1	DCM	65	21
6	B2	DCM	77	20
7	B3	DCM	81	41
8	B4	DCM	91	22
9	B5	DCM	80	14
10	B6	DCM	75	18
11	C1	DCM	92	77
12	C2	DCM	90	70
13	C3	DCM	82	69
14	C1	$CHCl_3$	79	81
15	C1	DCE	94	90
16	C1	CCl_4	86	66
17	C1	toluene	90	72
18	C1	*m*-xylene	81	77
19	C1	THF	N.R.	N.D.

20	C1	MCPE	83	82
21	C1	ether	46	80
22[d]	C1	DCE	75	55
23[e]	C1	DCE	60	90
24[f]	C1	DCE	94	92
25[g]	C1	DCE	88	90
26[h]	C1	DCE	90	89



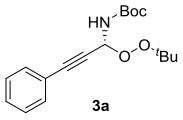
[a]General reaction condition: **1a** (0.1 mmol), **2a** (0.2 mmol), catalyst (10 mol%), solvent (1 mL), and 3Å M.S. (100mg), rt, 12h. [b] Isolated yield. [c] Determined by HPLC analysis. [d] Na_2SO_4 replaced 3 Å M. S.. [e] 4 Å M. S. replaced 3 Å M. S.. [f] Volume of solvent was 2 mL. [g] Reaction was carried out at 0 ℃. [h] the catalyst loading was reduced to 5 mol%. Boc = *tert*-butoxycarbonyl, DCM = dichloromethane, DCE = 1,2-dichloroethane, M. S. = molecular sieves.

General procedure for synthesis of chiral C-alkynyl α-amino peroxides 3, 4



To a solution of **1** (0.1 mmol) and **2** (0.2 mmol) in DCE (1.0 mL) was added the catalyst **C1** (10 mol %) and 3Å MS (100 mg) at room temperature. After *C*-alkynyl-*N*-Boc-*N*,*O*-acetal **1** was consumed, the mixture was directly purified by silica gel chromatography (ethyl acetate/petroleum ether = 1/40 to 1/20) to afford the product **3** or **4** (the racemic product **3** or **4** was obtained by using 1,1'-binaphthyl-2,2'-diyl hydrogenphosphate as the catalyst).

Characterization data of the products 3, 4



tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **3a**

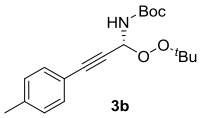
White Solid, 94% yield, 30.1 mg, $[\alpha]_D^{20}$= +62.3 (c 1.00 $CHCl_3$).

^{1}H-NMR(400 MHz, CDCl3): δ (ppm): 7.46 (d, 2H, J = 6.4 Hz), 7.37-7.30 (m, 3H), 6.23 (d, 1H, J = 9.2 Hz), 5.59 (d, 1H, J = 7.2 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, CDCl3): δ (ppm): 153.91, 131.91, 128.94, 128.29, 121.73, 85.15, 83.20, 81.08, 80.61, 75.55, 28.25, 26.40.

HRMS calcd.for C18H25NO4Na [M+Na]+: 342.1676, found: 342.1672.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 5.7 min (minor), t_R = 12.3 min (major).



3b

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(p-tolyl)prop-2-yn-1-yl)carbamate **3b**

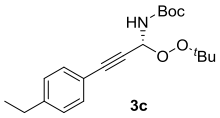
White Solid, 84% yield, 28.3 mg, $[\alpha]_D^{20}$= +51.2 (c 1.25 $CHCl_3$).

^{1}H-NMR(400 MHz, CDCl3): δ (ppm): 7.35 (d, 2H, *J* = 8.0 Hz), 7.11 (d, 2H, *J* = 8.0 Hz), 6.22 (d, 1H, *J* = 8.8 Hz), 5.58 (d, 1H, *J* = 7.2 Hz), 2.35 (s, 3H), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, CDCl3): δ (ppm): 153.96, 139.15, 131.81, 129.05, 118.64, 85.41, 82.53, 81.03, 80.53, 75.58, 28.26, 26.40, 21.50.

HRMS calcd.for C19H27NO4Na [M+Na]+: 356.1832, found: 356.1827.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 7.7 min (minor), t_R = 23.6 min (major).



3c

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-ethylphenyl)prop-2-yn-1-yl)carbamate **3c**

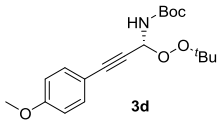
White Solid, 80% yield, 27.8 mg, $[\alpha]_D^{20}$= +80.2 (c 1.00 $CHCl_3$).

^{1}H-NMR(400 MHz, CDCl3): δ (ppm): 7.37 (d, 2H, *J* = 6.8 Hz), 7.14 (d, 2H, *J* = 8.0 Hz), 6.22 (d, 1H, *J* = 9.2 Hz), 5.59 (d, 1H, *J* = 7.2 Hz), 2.64 (dd, 2H, *J* = 7.6 Hz, *J* = 7.6 Hz), 1.48 (s, 9H), 1.29 (s, 9H), 1.22 (t, 3H, *J* = 7.6 Hz, *J* = 7.6 Hz).

^{13}C-NMR (100 MHz, CDCl3): δ (ppm): 153.98, 145.43, 131.90, 127.86, 118.86, 85.43, 82.51, 81.03, 80.53, 75.59, 28.82, 28.26, 26.40, 15.42.

HRMS calcd.for C20H29NO4Na [M+Na]+: 370.1989, found: 370.1984.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 6.9 min (minor), t_R = 20.7 min (major).



3d

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-methoxyphenyl)prop-2-yn-1-yl)carbamate **3d**

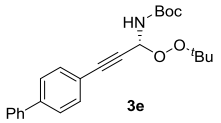
White Solid, 90% yield, 31.5 mg, $[\alpha]_D^{20}$= +56.3 (c 1.90 $CHCl_3$).

^{1}H-NMR(400 MHz, CDCl3): δ (ppm): 7.39 (d, 2H, *J* = 8.8 Hz), 6.84 (d, 2H, *J* = 8.8 Hz), 6.22 (d, 1H, *J* = 9.2 Hz), 5.59 (d, 1H, *J* = 8.0 Hz), 3.91 (S, 3H), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, CDCl3): δ (ppm): 160.09, 153.99, 133.42, 113.94, 113.73, 85.29, 81.84, 81.03, 80.49, 75.60, 55.28, 28.25, 26.40.

HRMS calcd.for C19H27NO5Na [M+Na]+: 372.1781, found: 372.1778.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 15.3 min (minor), t_R = 21.7 min (major).



3e

tert-butyl (*R*)-(3-([1,1'-biphenyl]-4-yl)-1-(*tert*-butylperoxy)prop-2-yn-1-yl)carbamate **3e**

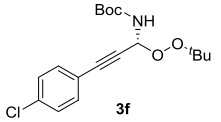
White Solid, 97% yield, 38.3 mg, $[\alpha]_D^{20}$= +83.0 (c 0.20 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.59-7.51 (m, 6H), 7.44 (t, 2H, *J* = 7.2 Hz, *J* = 7.6 Hz), 7.36 (t, 1H, *J* = 7.6 Hz, *J* = 7.2 Hz), 6.26 (d, 1H, *J* = 9.6 Hz), 5.64 (d, 1H, *J* = 9.2 Hz), 1.49 (s, 9H), 1.31 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.00, 141.71, 140.17, 132.36, 128.89, 127.79, 127.05, 126.99, 120.56, 85.06, 83.83, 81.14, 80.63, 75.53, 28.27, 26.43.

HRMS calcd.for C24H29NO4Na [M+Na]+: 418.1989, found: 418.1983.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 15.3 min (minor), t_R = 21.7 min (major).



3f

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-chlorophenyl)prop-2-yn-1-yl)carbamate **3f**

white solid, 90% yield, 32.0 mg, $[\alpha]_D^{20}$= +89.1 (c 1.30 $CHCl_3$).

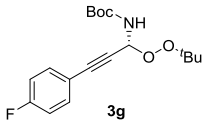
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.39 (d, 2H, *J* = 8.8 Hz), 7.29 (d, 2H, *J* = 8.4 Hz), 6.22 (d, 1H, *J* = 9.2 Hz), 5.61 (d, 1H, *J* = 8.4 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.92, 135.10, 133.14, 128.69, 120.20, 84.22, 83.97,

81.16, 80.69, 75.38, 28.24, 26.38.

HRMS calcd.for C18H24ClNO4Na [M+Na]+: 376.1286, found: 376.1286.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 5.7 min (minor), t_R = 14.7 min (major).



3g

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-fluorophenyl)prop-2-yn-1-yl)carbamate **3g**

White solid, 92% yield, 31.3 mg, $[\alpha]_D^{20}$= +59.9 (c 1.60 $CHCl_3$).

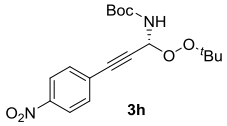
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.39-7.35 (m, 2H), 6.96-6.92 (m, 2H), 6.15 (d, 1H, *J* = 9.2 Hz), 5.53 (d, 1H, *J* = 8.0 Hz), 1.41 (s, 9H), 1.22 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 162.89 (d, *J* = 249 Hz), 153.95, 133.90 (d, *J* = 8 Hz), 117.80 (d, *J* = 3 Hz), 115.66 (d, *J* = 22 Hz), 84.09, 82.99, 81.12, 80.65, 75.41, 28.24, 26.38.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -109.75.

HRMS calcd.for C18H24FNO4Na [M+Na]+: 360.1582, found: 360.1587.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R = 6.1 min (minor), t_R = 16.7 min (major).



3h

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-nitrophenyl)prop-2-yn-1-yl)carbamate **3h**

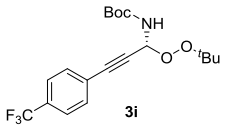
White solid, 98% yield, 35.7 mg, $[\alpha]_D^{20}$= +60.6 (c 1.60 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 8.20 (d, 2H, *J* = 8.8 Hz), 7.62 (d, 2H, *J* = 8.8 Hz), 6.27 (d, 1H, *J* = 9.2 Hz), 5.65 (d, 1H, *J* = 8.0 Hz), 1.49 (s, 9H), 1.30 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm):153.84, 147.61, 132.74, 128.54, 123.56, 88.40, 82.91, 81.33, 80.97, 75.20, 28.22, 26.37.

HRMS calcd.for C18H24N2O6Na [M+Na]+: 387.1527, found: 387.1523.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =13.4 min (minor), t_R = 28.6 min (major).



3i

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-(trifluoromethyl)phenyl)prop-2-yn-1-yl)carbamate **3i**

White solid, 92% yield, 35.6 mg, $[\alpha]_D^{20}$= +68.9 (c 0.85 $CHCl_3$).

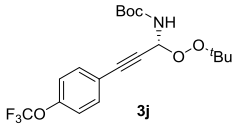
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.58 (t, 4H, *J* = 9.6 Hz, *J* = 9.6 Hz), 6.25 (d, 1H, *J* = 9.6 Hz), 5.55 (d, 1H, *J* = 9.2 Hz), 1.49 (s, 9H), 1.30 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.91, 132.19, 130.70 (d, *J* = 33 Hz), 125.25 (t, *J* = 4 Hz, *J* = 13 Hz), 122.41, 85.65, 83.56, 81.25, 80.82, 75.25, 28.23, 26.37.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -62.94.

HRMS calcd.for C19H24F3NO4Na [M+Na]+: 410.1550, found: 410.1553.

HPLC analysis: Daicel CHIRALCEL AD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =8.4 min (minor), t_R = 11.6 min (major).



3j

tert-butyl (*R*)-(1-*(tert*-butylperoxy)-3-(4-(trifluoromethoxy)phenyl)prop-2-yn-1-yl)carbamate **3j**

White solid, 84% yield, 34 mg, $[\alpha]_D^{20}$= +58.6 (c 2.00 $CHCl_3$).

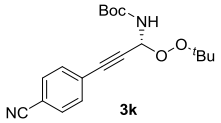
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.51-7.47 (m, 2H), 7.17 (d, 2H, *J* = 8.0 Hz)， 6.23 (d, 1H, *J* = 9.2 Hz), 5.62 (d, 1H, *J* = 8.4 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.94, 149.40, 133.50, 121.62, 120.62 (d, *J* = 31 Hz), 119.05, 84.17, 83.64, 81.17, 80.73, 75.33, 28.23, 26.37.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -57.81.

HRMS calcd.for C19H24F3NO5Na [M+Na]+: 426.1499, found: 426.1495.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =4.4 min (minor), t_R = 10.4 min (major).



3k

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(4-cyanophenyl)prop-2-yn-1-yl)carbamate **3k**

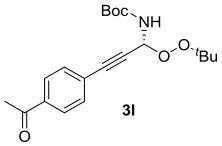
White solid, 91% yield, 31.3 mg, $[\alpha]_D^{20}$= +123.4 (c 1.54 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.62 (d, 2H, *J* = 8.4 Hz), 7.55 (d, 2H, *J* = 8.4 Hz), 6.25 (d, 1H, *J* = 9.6 Hz), 5.64 (d, 1H, *J* = 9.2 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.86, 132.45, 132.03, 126.61, 118.22, 112.44, 87.60, 83.17, 81.31, 80.92, 75.18, 28.22, 26.37.

HRMS calcd.for C19H24N2O4Na [M+Na]+: 367.1628, found: 367.1621.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.3 min (minor), t_R = 17.3 min (major).



3l

tert-butyl (*R*)-(3-(4-acetylphenyl)-1-(*tert*-butylperoxy)prop-2-yn-1-yl)carbamate **3l**

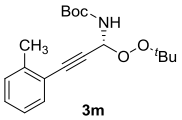
White solid, 95% yield, 34.3mg, $[\alpha]_D^{20}$= +82.8 (c 1.60 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.91 (d, 2H, *J* = 8.0 Hz), 7.55 (d, 2H, *J* = 8.4 Hz), 6.26 (d, 1H, *J* = 9.6 Hz), 5.67 (d, 1H, *J* = 8.0 Hz), 2.61 (s, 3H), 1.49 (s, 9H), 1.30 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 197.26, 153.92, 136.83, 132.08, 128.18, 126.51, 86.41, 84.12, 81.24, 80.87, 75.31, 28.24, 26.38.

HRMS calcd.for C20H27NO5Na [M+Na]+: 384.1781, found: 384.1777.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =13.0 min (minor), t_R = 31.3 min (major).



3m

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(o-tolyl)prop-2-yn-1-yl)carbamate **3m**

White solid, 99% yield, 33.0 mg, $[\alpha]_D^{20}$= +51.7 (c 1.35 $CHCl_3$).

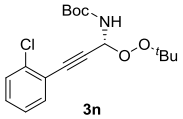
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.42 (d, 1H, *J* = 7.6 Hz), 7.25-7.18 (m, 2H), 7.13 (t, 1H, *J* = 7.6 Hz, *J* = 7.2 Hz), 6.27 (d, 1H, *J* = 9.6 Hz), 5.61 (d, 1H, 8.4 Hz), 2.43 (s, 3H), 1.49 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.02, 140.72, 132.20, 129.46, 128.95, 125.51, 121.49, 86.93, 84.19, 80.98, 80.58, 75.59, 28.26, 26.40, 20.59.

HRMS calcd.for C19H27NO4Na [M+Na]+: 356.1832, found: 356.1830.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ

= 254 nm, retention time: t_R =5.2 min (minor), t_R = 9.7 min (major).



3n

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(2-chlorophenyl)prop-2-yn-1-yl)carbamate **3n**

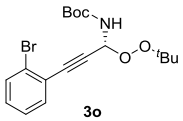
White solid, 83% yield, 29.4 mg, $[\alpha]_D^{20}$= +26.8 (c 1.40 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.49 (dd, 1H, *J* = 8.0 Hz, *J* = 7.6 Hz), 7.39 (dd, 1H, *J* = 8.0 Hz, *J* = 7.2 Hz), 7.30-7.25 (m, 1H), 7.21 (td, 1H, *J* = 7.6 Hz, *J* = 7.6 Hz), 6.28 (d, 1H, *J* = 9.6 Hz), 5.66 (d, 1H, *J* = 8.4 Hz), 1.49 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.96, 136.330, 133.69, 129.99, 129.32, 126.41, 121.78, 88.36, 81.79, 81.10, 80.67, 75.44, 28.25, 26.40.

HRMS calcd.for C18H24ClNO4Na [M+Na]+: 376.1286, found: 376.1288.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =4.5 min (minor), t_R = 11.3 min (major).



3o

tert-butyl (*R*)-(3-(2-bromophenyl)-1-(*tert*-butylperoxy)prop-2-yn-1-yl)carbamate **3o**

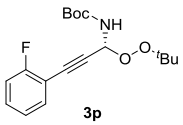
White solid, 91% yield, 36.4 mg, $[\alpha]_D^{20}$= +34.1 (c 1.50 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.58 (d, 1H, *J* = 7.6 Hz), 7.50 (dd, 1H, *J* = 7.6 Hz, *J* = 7.6 Hz), 7.27 (t, 1H, *J* = 7.2 Hz, *J* = 8.0 Hz), 7.21 (td, 1H, *J* = 8.0 Hz, *J* = 7.6 Hz), 6.28 (d, 1H, *J* = 10.0 Hz), 5.67 (d, 1H, *J* = 9.6 Hz), 1.48 (s, 9H), 1.30 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.98, 133.78, 132.48, 130.15, 127.00, 125.75, 123.95, 87.69, 83.49, 81.14, 80.69, 75.43, 28.26, 26.41.

HRMS calcd.for C18H24BrNO4Na [M+Na]+: 420.0781, found: 420.0779.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.8 min (minor), t_R = 18.3 min (major).



3p

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(2-fluorophenyl)prop-2-yn-1-yl)carbamate **3p**

White solid, 92% yield, 31.0 mg, $[\alpha]_D^{20}$= +85.3 (c 0.80 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45 (td, 1H, *J* = 7.6 Hz, *J* = 7.2 Hz), 7.36-7.30 (m, 1H),

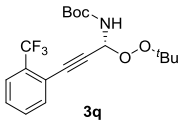
7.12-7.05 (m, 2H), 6.26 (d, 1H, *J* = 10.0 Hz), 5.66 (d, 1H, *J* = 9.2 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 163.0 (d, *J* = 251 Hz), 153.95, 133.80, 130.78 (d, *J* = 8 Hz), 123.95 (d, *J* = 3 Hz), 115.58 (d, *J* = 21 Hz), 110.39 (d, *J* = 16 Hz), 88.37, 81.14, 80.66, 78.60, 75.38, 28.24, 26.38.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -109.33.

HRMS calcd.for C18H24FNO4Na [M+Na]+: 360.1582, found: 360.1579.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =7.8 min (minor), t_R = 40.3 min (major).



3q

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(2-(trifluoromethyl)phenyl)prop-2-yn-1-yl)carbamate **3q**

White solid, 99% yield, 38.3 mg, **$[\alpha]_D^{20}$**= +71.2 (c 1.80 $CHCl_3$).

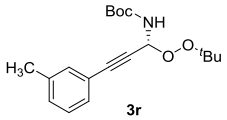
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.66 (d, 1H, *J* = 7.6 Hz), 7.61 (d, 1H, *J* = 7.6 Hz), 7.50 (t, 1H, *J* = 7.2 Hz, *J* = 7.6 Hz), 7.46 (t, 1H, *J* = 7.2 Hz, *J* = 7.6 Hz), 6.26 (d, 1H, *J* = 9.6 Hz), 5.61 (d, 1H, *J* = 8.4 Hz), 1.49 (s, 9H), 1.28 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.96, 134.27, 131.38(m), 128.75, 125.87 (q, *J* = 5 Hz, *J* = 10 Hz), 124.67, 121.95, 120.00, 88.82, 81.06, 80.70, 77.21, 75.35, 28.23, 26.31.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -62.12.

HRMS calcd.for C19H24F3NO4Na [M+Na]+: 410.1550, found:410.1548.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =4.6 min (minor), t_R = 7.4 min (major).



3r

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(m-tolyl)prop-2-yn-1-yl)carbamate **3r**

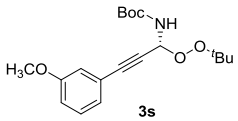
White solid, 96% yield, 32.0 mg, **$[\alpha]_D^{20}$**= +40.2 (c 1.35 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.27 (dd, 2H, *J* = 8.8 Hz, *J* = 7.6 Hz), 7.21 (t, 1H, *J* = 7.6 Hz, *J* = 7.2 Hz), 7.15 (d, 1H, *J* = 7.6 Hz), 6.23 (d, 1H, *J* = 9.6 Hz), 5.59 (d, 1H, *J* = 8.0 Hz), 2.32 (s, 3H), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.96, 138.01, 132.47, 129.85, 128.98, 128.20, 121.50, 85.35, 82.83, 81.06, 80.56, 75.53, 28.25, 26.40, 21.17.

HRMS calcd.for C19H27NO4Na [M+Na]+: 356.1832, found: 356.1833.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =7.0 min (minor), t_R = 19.3 min (major).



3s

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(3-methoxyphenyl)prop-2-yn-1-yl)carbamate **3s**

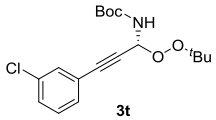
White solid, 94% yield, 33.0 mg, $[\alpha]_D^{20}$= +74.9 (c 1.50 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.16 (dd, 1H, *J* = 9.6 Hz, *J* = 8.0 Hz), 6.98 (d, 1H, *J* = 7.6 Hz), 6.91 (s, 1H), 6.83 (dd, 1H, *J* = 8.4 Hz, *J* = 8.0 Hz), 6.16 (d, 1H, *J* = 9.2 Hz), 5.55 (d, 1H, *J* = 8.4 Hz), 3.72 (s, 9H), 1.41 (s, 9H), 1.22 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 159.25, 153.97, 129.39, 124.43, 122.67, 116.67, 115.64, 85.06, 82.99, 81.10, 80.60, 75.44, 55.29, 28.25, 26.39.

HRMS calcd.for C19H27NO5Na [M+Na]+: 372.1781, found: 372.1583.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.0 min (minor), t_R = 21.1 min (major).



3t

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(3-chlorophenyl)prop-2-yn-1-yl)carbamate **3t**

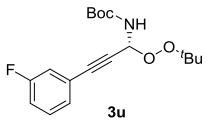
White solid, 90% yield, 31.8 mg, $[\alpha]_D^{20}$= +39.4 (c 1.40 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45 (s, 1H), 7.35-7.32 (m, 2H), 7.27 (t, 1H, *J* = 7.6 Hz, *J* = 6.0 Hz), 6.23 (d, 1H, *J* = 9.6 Hz), 5.62 (d, 1H, *J* = 8.8 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.92, 134.18, 131.77, 130.04, 129.58, 129.29, 123.42, 84.46, 83.60, 81.19, 80.74, 75.31, 28.24, 26.39.

HRMS calcd.for C18H24ClNO4Na [M+Na]+: 376.1286, found: 376.1283.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.9 min (minor), t_R = 11.4 min (major).



3u

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(3-fluorophenyl)prop-2-yn-1-yl)carbamate **3u**

White solid, 91% yield, 31.0 mg, $[\alpha]_D^{20}$= +57.7 (c 1.00 $CHCl_3$).

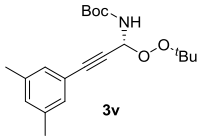
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.30-7.23 (m, 2H), 7.16 (dd, 1H, *J* = 9.2 Hz, *J* = 9.6 Hz), 7.08-7.03 (m, 1H), 6.23 (d, 1H, *J* = 9.6 Hz), 5.62 (d, 1H, *J* = 8.8 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 162.24 (d, *J* = 245 Hz), 153.93, 129.95 (d, *J* = 8 Hz), 128.82 (d, *J* = 3 Hz), 123.53 (d, *J* = 10 Hz), 118.74 (d, *J* = 23 Hz), 116.39 (d, *J* = 21 Hz), 84.17, 83.76, 81.18, 75.31, 28.24, 26.38.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -112.66.

HRMS calcd.for C18H24FNO4Na [M+Na]+: 360.1582, found: 360.1584.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.8 min (minor), t_R = 10.4 min (major).



3v

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(3,5-dimethylphenyl)prop-2-yn-1-yl)carbamate **3v**

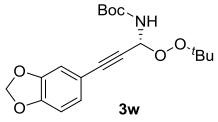
White solid, 89% yield, 31.0 mg, $[\alpha]_D^{20}$= +74.4 (c 1.20 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.09 (s, 2H), 6.97 (s, 1H), 6.22 (d, 1H, *J* = 9.6 Hz), 5.58 (d, 1H, *J* = 8.0 Hz), 2.28 (s, 6H), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.99, 137.88, 130.87, 129.58, 121.30, 85.54, 82.43, 81.04, 80.51, 75.55, 28.26, 26.40, 21.06.

HRMS calcd.for C20H29NO4Na [M+Na]+: 370.1989, found: 370.1989.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.4 min (minor), t_R = 12.9 min (major).



3w

tert-butyl (*R*)-(3-(benzo[d][1,3]dioxol-5-yl)-1-(*tert*-butylperoxy)prop-2-yn-1-yl)carbamate **3w**

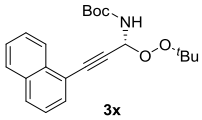
White solid, 99% yield, 36.0 mg, $[\alpha]_D^{20}$= +50.8 (c 1.70 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 6.99 (d, 1H, *J* = 8.0 Hz), 6.89 (s, 1H), 6.75 (d, 1H, *J* = 8.0 Hz), 6.21 (d, 1H, *J* = 9.6 Hz), 5.98 (s, 2H), 5.61 (d, 1H, *J* = 8.8 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.98, 148.43, 147.37, 126.79, 114.85, 111.77, 108.44, 101.40, 85.09, 81.58, 81.07, 80.55, 75.50, 28.25, 26.39.

HRMS calcd.for C19H25NO6Na [M+Na]+: 386.1574, found: 386.1569.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =8.7 min (minor), t_R = 23.5 min (major).



3x

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(naphthalen-1-yl)prop-2-yn-1-yl)carbamate **3x**

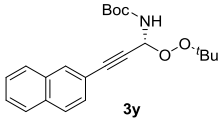
White solid, 86% yield, 31.8 mg, $[\alpha]_D^{20}$= +63.7 (c 1.20 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 8.33 (d, 1H, *J* = 8.4 Hz), 7.85 (d, 2H, *J* = 8.4 Hz), 7.69 (d, 1H, *J* = 7.2 Hz), 7.59-7.50 (m, 2H), 7.42 (t, 1H, *J* = 8.0 Hz, *J* = 7.2 Hz), 6.38 (d, 1H, *J* = 9.6 Hz), 5.74 (d, 1H, *J* = 8.4 Hz), 1.50 (s, 9H), 1.33 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.07, 133.37, 133.07, 130.92, 129.48, 126.96, 126.54, 126.13, 125.09, 119.36, 88.06, 83.43, 81.11, 80.07, 75.66, 28.29, 26.46.

HRMS calcd.for C22H27NO4Na [M+Na]+: 370.2013, found: 370.2008.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =6.4 min (minor), t_R = 11.9 min (major).



3y

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(naphthalen-2-yl)prop-2-yn-1-yl)carbamate **3y**

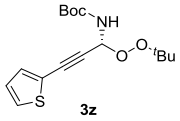
White solid, 88% yield, 32.6 mg, $[\alpha]_D^{20}$= +73.0 (c 1.58 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.99 (s, 1H), 7.82-7.76 (m, 3H), 7.50 (t, 3H, *J* = 9.6 Hz, *J* = 5.6 Hz), 6.29 (d, 1H, *J* = 10.0 Hz)， 5.67 (d, 1H, *J* = 9.2 Hz), 1.49 (s, 9H), 1.31 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.01, 133.10, 132.78, 132.20, 128.26, 128.04, 127.86, 127.79, 127.02, 126.66, 118.96, 85.51, 83.47, 81.16, 80.63, 75.56, 28.28, 26.44.

HRMS calcd.for C22H27NO4Na [M+Na]+: 370.2013, found: 370.2011.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.8 min (minor), t_R = 12.9 min (major).



3z

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(thiophen-2-yl)prop-2-yn-1-yl)carbamate **3z**

White solid, 97% yield, 31.5 mg, $[\alpha]_D^{20}$= +41.4 (c 1.30 $CHCl_3$).

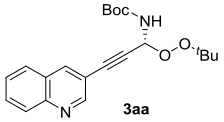
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.29 (d, 1H, *J* = 5.2 Hz), 7.26 (d, 1H, *J* = 3.6 Hz), 6.98 (dd,

1H, *J* = 5.2 Hz, *J* = 5.2 Hz), 6.24 (d, 1H, *J* = 9.6 Hz), 5.61 (d, 1H, *J* = 8.8 Hz), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.91, 133.10, 127.99, 126.99, 121.53, 87.04, 81.19, 80.68, 78.64, 75.50, 28.25, 26.39.

HRMS calcd.for C16H23NO4SNa [M+Na]+: 348.1240, found: 348.1235.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =7.2 min (minor), t_R = 18.0 min (major).



3aa

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(quinolin-3-yl)prop-2-yn-1-yl)carbamate **3aa**

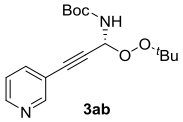
White solid, 91% yield, 33.7 mg, $[\alpha]_D^{20}$= +118.3 (c 1.30 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 8.93 (s, 1H), 8.23 (s, 1H), 8.11 (d, 1H, *J* = 8.4 Hz), 7.80-7.60 (m, 2H), 7.58 (t, 1H, *J* = 7.2 Hz, *J* = 7.6 Hz), 6.32 (d, 1H, *J* = 9.6 Hz), 5.88 (s, 1H), 1.50 (s, 9H), 1.31 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.99, 151.92, 147.13, 139.22, 130.49, 129.45, 127.68, 127.43, 126.99, 115.87, 86.63, 82.32, 81.28, 80.81, 75.36, 28.26, 26.41.

HRMS calcd.for C21H26N2O4Na [M+Na]+: 371.1965, found: 371.1963.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =11.8 min (minor), t_R = 17.0 min (major).



3ab

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(pyridin-3-yl)prop-2-yn-1-yl)carbamate **3ab**

White solid, 89% yield, 28.5 mg, $[\alpha]_D^{20}$= +16.5 (c 1.15 $CHCl_3$).

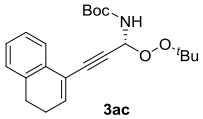
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 8.71 (d, 1H, *J* = 1.2 Hz), 8.58 (dd, 1H, *J* = 4.8 Hz, *J* = 4.8 Hz), 7.77-7.74 (m, 1H), 7.27 (t, 1H, *J* = 7.6 Hz, *J* = 5.2 Hz), 6.26 (d, 1H, *J* = 9.6 Hz), 5.90 (s, 1H), 1.49 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.98, 152.49, 149.24, 138.91, 123.02, 118.97, 86.81, 81.66, 81.25, 80.75, 75.24, 28.24, 26.37.

HRMS calcd.for C17H24N2O4Na [M+Na]+: 321.1809, found: 321.1810.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 70/30, flow rate = 1.0 mL/min, λ

= 254 nm, retention time: t_R =8.4 min (minor), t_R = 10.4 min (major).



3ac

tert-butyl (*R*)-(1-(*tert*-butylperoxy)-3-(3,4-dihydronaphthalen-1-yl)prop-2-yn-1-yl)carbamate **3ac**

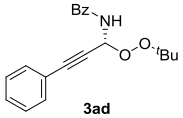
Colorless oil, 85% yield, 31.5 mg, $[\alpha]_D^{20}$= +87.3 (c 0.50 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.55 (d, 1H, *J* = 7.6 Hz), 7.24-7.16 (m, 2H), 7.10 (d, 1H, *J* = 7.2 Hz), 6.51 (t, 1H, *J* = 4.8 Hz, *J* = 4.8 Hz), 6.23 (d, 1H, *J* = 9.6 Hz), 5.61 (d, 1H, *J* = 8.0 Hz), 2.79 (t, 2H, *J* = 8.0 Hz, *J* = 8.4 Hz), 2.41-2.36 (m, 2H), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.02, 137.01, 134.84, 132.11, 127.82, 127.39, 126.64, 125.08, 120.69, 84.11, 83.26, 81.00, 80.57, 75.51, 28.26, 26.97, 26.41, 23.62.

HRMS calcd.for C22H29NO4Na [M+Na]+: 394.1989, found: 394.1982.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.4 min (minor), t_R = 11.0 min (major).



3ad

(*R*)-N-(1-(*tert*-butylperoxy)-3-phenylprop-2-yn-1-yl)benzamide **3ad**

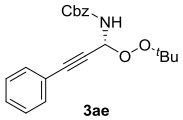
White solid, 90% yield, 29.0 mg, $[\alpha]_D^{20}$= +106.5 (c 0.85 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.86 (d, 2H, *J* = 7.2 Hz), 7.56-7.53 (m, 1H), 7.50-7.45 (m, 4H), 7.38-7.30 (m, 3H), 7.14 (d, 1H, *J* = 9.2 Hz) 6.76 (d, 1H, *J* = 9.2 Hz), 1.30 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 166.16, 133.43, 132.17, 131.95, 129.08, 128.71, 128.36, 127.33, 121.61, 85.52, 83.39, 81.40, 73.86, 26.47.

HRMS calcd.for C20H21NO3Na [M+Na]+: 346.1414, found: 346.1410.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =13.2 min (minor), t_R = 22.6 min (major).



3ae

benzyl (*R*)-(1-(*tert*-butylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **3ae**

White solid, 90% yield, 31.8 mg, $[\alpha]_D^{20}$= +86.8 (c 1.38 $CHCl_3$).

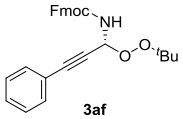
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.44 (t, 2H, *J* = 6.4 Hz, *J* = 8.0 Hz), 7.37 (d, 3H, *J* = 6.4 Hz), 7.35-7.29 (m, 4H), 6.30 (d, 1H, *J* = 9.6 Hz), 5.84 (d, 1H, *J* = 8.8 Hz), 5.17 (s, 2H), 1.27 (s,

9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.80, 135.96, 131.93, 129.09, 128.56, 128.35, 128.31, 128.28, 121.53, 85.54, 82.80, 81.28, 75.90, 67.32, 26.38.

HRMS calcd.for C21H23NO4Na [M+Na]+: 376.1519, found: 376.1515.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =13.9 min (major), t_R = 15.7 min (minor).



3af

(9H-fluoren-9-yl)methyl (*R*)-(1-(*tert*-butylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **3af**

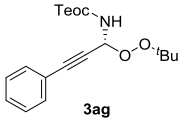
White solid, 91% yield, 40.1 mg, **$[\alpha]_D^{20}$**= +70.9 (c 1.50 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.77 (d, 2H, *J* = 7.6 Hz), 7.62 (d, 2H, *J* = 7.2 Hz), 7.47 (t, 2H, *J* = 6.8 Hz, *J* = 7.6 Hz), 7.41 (t, 2H, *J* = 7.2 Hz, *J* = 7.6 Hz), 7.35-7.30 (m, 5H), 6.31 (d, 1H, *J* = 9.6 Hz), 5.88 (d, 2H, *J* = 9.6 Hz), 4.57-4.40 (m, 2H), 4.26 (t, 1H, *J* = 6.8 Hz, *J* = 6.8 Hz), 1.28 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.86, 143.71, 141.34, 131.95, 129.13, 128.38, 127.79, 127.14, 125.12, 121.54, 120.03, 85.63, 82.82, 81.31, 75.92, 67.33, 47.07, 26.44.

HRMS calcd.for C28H27NO4Na [M+Na]+: 464.1832, found: 464.1832.

HPLC analysis: Daicel CHIRALCEL AD-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =10.1 min (major), t_R = 13.6 min (minor).



3ag

2,2,2-trichloroethyl (*R*)-(1-(*tert*-butylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **3ag**

White solid, 90% yield, 35.6 mg, **$[\alpha]_D^{20}$**= +115.2 (c 1.64 $CHCl_3$).

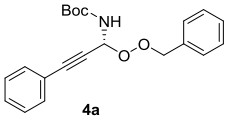
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.41 (d, 2H, *J* = 6.8 Hz), 7.31-7.26 (m, 3H), 6.23 (d, 1H, *J* = 9.6 Hz), 5.65 (d, 1H, *J* = 8.4 Hz), 4.17 (t, 2H, *J* = 8.4 Hz, *J* = 8.0 Hz), 1.25 (s, 9H), 0.97 (t, 2H, *J* = 8.0 Hz, *J* = 8.0 Hz), 0.00 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 156.56, 133.38, 130.50, 129.80, 123.09, 86.79, 84.53, 82.70, 77.31, 65.35, 27.88, 19.15, 0.00.

HRMS calcd.for C19H29NO4SiNa [M+Na]+: 386.1758, found: 386.1760.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ

= 254 nm, retention time: t_R =8.0 min (major), t_R = 11.6 min (minor).



4a

tert-butyl (*R*)-(1-(benzylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **4a**

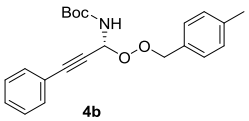
White solid, 89% yield, 31.4 mg, $[\alpha]_D^{20}$= +54.5 (c 1.10 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45 (dd, 2H, *J* = 7.6 Hz, *J* = 8.0 Hz), 7.40 (dd, 2H, *J* = 8.0 Hz, *J* = 7.2 Hz), 7.38-7.30 (m, 6H), 6.32 (d, 1H, *J* = 8.8 Hz), 5.67 (d, 1H, *J* = 4.8 Hz), 5.13 (d, 2H, *J* = 2.0 Hz), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.84, 135.25, 131.92, 129.26, 129.13, 128.52, 128.46, 128.39, 121.50, 85.45, 82.87, 80.91, 77.84, 75.55, 28.26.

HRMS calcd.for C21H23NO4Na [M+Na]+: 376.1519, found: 376.1517.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =8.8 min (minor), t_R = 11.3 min (major).



4b

tert-butyl (*R*)-(1-((4-methylbenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4b**

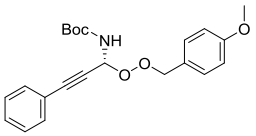
White solid, 90% yield, 33.0 mg, $[\alpha]_D^{20}$= +18.9 (c 0.80 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.46-7.38 (m, 2H), 7.37-7.28 (m, 5H), 7.15 (d, 2H, *J* = 7.6 Hz), 6.31 (d, 1H, *J* = 8.8 Hz), 5.67 (d, 1H, *J* = 5.6 Hz), 5.09 (q, 2H, *J* = 10.4 Hz, *J* = 10.8 Hz), 2.33 (s, 3H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.82, 138.38, 132.12, 131.91, 129.42, 129.16, 129.10, 128.38, 121.55, 85.35, 82.98, 80.85, 77.72, 75.52, 28.26, 21.25.

HRMS calcd.for C22H25NO4Na [M+Na]+: 390.1676, found: 390.1672.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.4 min (minor), t_R = 13.8 min (major).



tert-butyl (*R*)-(1-((4-methoxybenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4c**

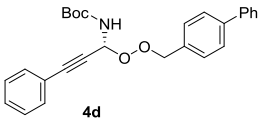
White solid, 91% yield, 35.0 mg, $[\alpha]_D^{20}$= +81.2 (c 1.63 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.46-7.44 (m, 2H), 7.38-7.29 (m, 5H), 6.86 (d, 1H, *J* = 8.8 Hz), 6.30 (d, 1H, *J* = 8.4 Hz), 5.06 (q, 2H, *J* = 10.4 Hz, *J* = 10.4 Hz), 3.78 (s, 3H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 159.92, 153.84, 131.90, 131.04, 129.10, 128.38, 127.18, 121.54, 113.88, 85.35, 83.01, 80.84, 77.46, 75.51, 55.25, 28.25.

HRMS calcd.for C22H25NO5Na [M+Na]+: 406.1625, found: 406.1626.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.2 min (minor), t_R = 14.4 min (major).



4d

tert-butyl (*R*)-(1-(([1,1'-biphenyl]-4-ylmethyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4d**

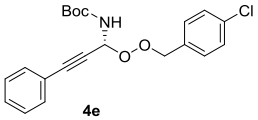
White solid, 97% yield, 41.7 mg, **$[\alpha]_D^{20}$**= +48.2 (c 1.65 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.57 (d, 4H, *J* = 7.6 Hz), 7.49-7.41 (m, 6H), 7.37-7.29 (m, 4H), 6.35 (d, 1H, *J* = 9.2 Hz), 5.72 (d, 1H, *J* = 7.6 Hz), 5.18 (t, 2H, *J* = 12.4 Hz, *J* = 11.2 Hz), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.89, 141.48, 140.78, 134.23, 131.93, 129.75, 129.16, 128.80, 128.42, 127.43, 127.25, 127.17, 121.49, 85.49, 82.89, 80.93, 77.55, 75.58, 28.28.

HRMS calcd.for C27H27NO4Na [M+Na]+: 452.1832, found: 452.1836.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.6 min (minor), t_R = 15.1 min (major).



4e

tert-butyl (*R*)-(1-((4-chlorobenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4e**

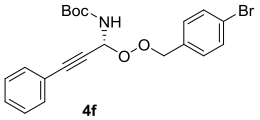
White solid, 90% yield, 35.0 mg, **$[\alpha]_D^{20}$**= +76.5 (c 1.20 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.46-7.44 (m, 2H), 7.39-7.35 (m, 2H), 7.34-7.30 (m, 5H), 6.30 (d, 1H, *J* = 8.8 Hz), 5.66 (d, 1H, *J* = 6.0 Hz), 5.09 (s, 2H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.80, 134.39, 133.97, 131.90, 130.53, 129.20, 128.64, 128.41, 121.38, 85.60, 82.65, 80.99, 77.36, 28.24.

HRMS calcd.for C21H22ClNO4Na [M+Na]+: 410.1130, found: 410.1131.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =6.6 min (minor), t_R = 11.3 min (major).



4f

tert-butyl (*R*)-(1-((4-bromobenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4f**

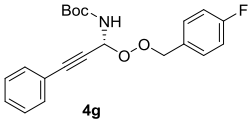
White solid, 85% yield, 36.7 mg, $[\alpha]_D^{20}$= +38.8 (c 1.45 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.47-7.43 (m, 4H), 7.36-7.30 (m, 3H), 7.27 (d, 2H, *J* = 8.0 Hz), 6.29 (d, 1H, *J* = 9.2 Hz), 5.66 (d, 1H, *J* = 7.6 Hz), 5.06 (s, 2H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.80, 134.49, 131.90, 131.60, 130.82, 129.20, 128.42, 122.58, 121.36, 85.60, 82.63, 80.99, 76.90, 75.54, 28.25.

HRMS calcd.for C21H22BrNO4Na [M+Na]+: 454.0624, found: 454.0626.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =6.7 min (minor), t_R = 11.8 min (major).



4g

tert-butyl (*R*)-(1-((4-fluorobenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4g**

White solid, 91% yield, 33.8 mg, $[\alpha]_D^{20}$= +63.4 (c 1.45 $CHCl_3$).

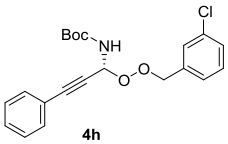
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45-7.43 (m, 2H), 7.39-7.30 (m, 5H), 7.02 (td, 2H, *J* = 8.8 Hz, *J* = 8.8 Hz), 6.30 (d, 1H, *J* = 8.4 Hz), 5.67 (s, 1H), 5.08 (s, 2H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 162.90 (d, *J* = 245 Hz), 153.81, 131.90, 131.15 (d, *J* = 8 Hz), 129.17, 128.40, 121.42, 115.36 (d, *J* = 21 Hz), 85.55, 82.73, 80.95, 76.95, 75.54, 28.24.

^{19}F NMR (376 MHz, $CDCl_3$): δ (ppm): -113.40.

HRMS calcd.for C21H22FNO4Na [M+Na]+: 394.1425, found: 394.1425.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =6.7 min (minor), t_R = 10.3 min (major).



4h

tert-butyl (*R*)-(1-((3-chlorobenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4h**

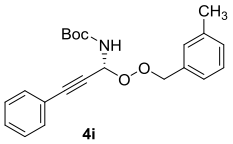
White solid, 92% yield, 35.7 mg, $[\alpha]_D^{20}$= +44.7 (c 1.48 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.47-7.44 (m, 2H), 7.41 (s, 1H), 7.38-7.31 (m, 3H), 7.30-7.26 (m, 3H), 6.31 (d, 1H, *J* = 8.8 Hz), 5.57 (d, 1H, *J* = 8.0 Hz), 5.09 (s, 2H), 1.48 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.81, 137.53, 134.32, 131.93, 129.71, 129.19, 129.08, 128.58, 128.40, 127.06, 121.36, 85.68, 82.60, 81.03, 76.87, 75.57, 28.25.

HRMS calcd.for C21H22ClNO4Na [M+Na]+: 410.1130, found: 410.1128.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =6.7 min (minor), t_R = 11.0 min (major).



4i

tert-butyl (*R*)-(1-((3-methylbenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4i**

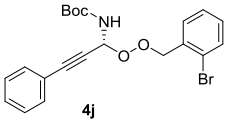
White solid, 90% yield, 33.0 mg, $[\alpha]_D^{20}$= +81.0 (c 1.20 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.46 (d, 2H, *J* = 7.6 Hz), 7.37-7.30 (m, 3H), 7.24-7.19 (m, 3H), 7.13 (d, 1H, *J* = 7.2 Hz), 6.32 (d, 1H, *J* = 8.8 Hz), 5.70 (s, 1H), 5.10 (t, 2H, *J* = 12.0 Hz, *J* = 12.0 Hz), 2.32 (s, 3H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.84, 138.09, 135.03, 131.92, 130.03, 129.30, 129.12, 128.39, 128.36,126.36, 121.53, 85.41, 82.97, 80.88, 77.91, 75.54, 28.26, 21.30.

HRMS calcd.for C22H25NO4Na [M+Na]+: 390.1676, found: 390.1677.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =8.0 min (minor), t_R = 10.9 min (major).



4j

tert-butyl (*R*)-(1-((2-bromobenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4j**

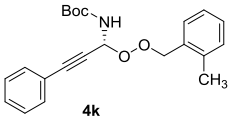
White solid, 99% yield, 42.8 mg, $[\alpha]_D^{20}$= +67.2 (c 1.50 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.53 (t, 2H, *J* = 7.2 Hz, *J* = 6.0 Hz), 7.45 (d, 2H, *J* = 7.2 Hz), 7.37-7.25 (m, 4H), 7.16 (t, 1H, *J* = 7.6 Hz, *J* = 7.6 Hz), 6.33 (d, 1H, *J* = 8.4 Hz), 5.74 (s, 1H), 5.26 (s, 2H), 1.48 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.79, 135.16, 132.73, 131.94, 130.89, 129.77, 129.12, 128.36, 127.41, 123.70, 121.47, 85.55, 82.76, 80.97, 76.95, 75.66, 28.26.

HRMS calcd.for C21H22BrNO4Na [M+Na]+: 454.0624, found: 454.0624.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.3 min (minor), t_R = 18.1 min (major).



4k

tert-butyl (*R*)-(1-((2-methylbenzyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4k**

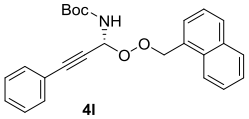
White solid, 95% yield, 35.0 mg, $[\alpha]_D^{20}$= +71.6 (c 1.35 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45 (d, 2H, *J* = 7.2 Hz), 7.32 (q, 4H, *J* = 7.2 Hz, *J* = 6.8 Hz), 7.23 (t, 1H, *J* = 6.0 Hz, *J* = 7.2 Hz), 7.16 (d, 2H, *J* = 4.4 Hz), 6.31 (d, 1H, *J* = 8.4 Hz), 5.67 (s, 1H), 5.17 (q, 2H, *J* = 10.4 Hz, *J* = 10.4 Hz), 2.40 (s, 3H), 1.46 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.80, 137.93, 132.94, 131.91, 130.77, 130.35, 129.10, 128.88, 128.37, 125.93, 121.54, 85.34, 83.08, 80.90, 75.88, 75.56, 28.25, 19.02.

HRMS calcd.for C22H25NO4Na [M+Na]+: 390.1676, found: 390.1675.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =8.6 min (minor), t_R = 10.4 min (major).



4l

tert-butyl (*R*)-(1-((naphthalen-1-ylmethyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4l**

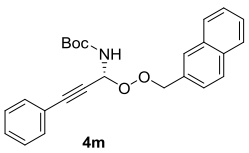
White solid, 95% yield, 38.3 mg, $[\alpha]_D^{20}$= +59.4 (c 1.65 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 8.22 (d, 1H, *J* = 6.8 Hz), 7.84 (d, 2H, *J* = 8.4 Hz), 7.54 (d, 1H, *J* = 6.8 Hz), 7.49-7.41 (m, 5H), 7.38-7.30 (m, 3H), 6.37 (d, 1H, *J* = 8.4 Hz), 5.70 (s, 1H), 5.61 (dd, 2H, *J* = 10.4 Hz, *J* = 10.8 Hz), 1.44 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.79, 133.73, 132.26, 131.96, 130.66, 129.63, 129.14, 128.86, 128.53, 128.39, 126.57, 125.87, 125.26, 124.13, 121.49, 85.33, 83.22, 80.92, 75.87, 75.60, 28.23.

HRMS calcd.for C25H25NO4Na [M+Na]+: 402.1711, found: 402.1712.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.8 min (minor), t_R = 18.8 min (major).



4m

tert-butyl (*R*)-(1-((naphthalen-2-ylmethyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4m**

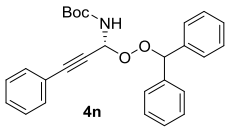
White solid, 95% yield, 38.3 mg, $[\alpha]_D^{20}$= +57.3 (c 1.75 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.81 (q, 4H, *J* = 8.0 Hz, *J* = 6.8 Hz), 7.53 (d, 1H, *J* = 8.4 Hz), 7.49-7.43 (m, 4H), 7.37-7.29 (m, 3H), 6.35 (d, 1H, *J* = 8.8 Hz), 5.67 (s, 1H), 5.29 (s, 2H), 1.45 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.85, 133.37, 133.24, 132.83, 131.94, 129.14, 128.52, 128.39, 128.22, 128.07, 127.72, 126.78, 126.26, 126.15, 121.49, 85.52, 82.88, 80.90, 77.94, 75.59, 28.24.

HRMS calcd.for C25H25NO4Na [M+Na]+: 402.1711, found: 402.1708.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.0 min (minor), t_R = 17.2 min (major).



4n

tert-butyl (*R*)-(1-(benzhydrylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **4n**

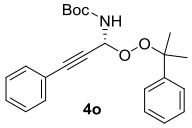
White solid, 80% yield, 34.4 mg, $[\alpha]_D^{20}$= +38.1 (c 1.90 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.44 (dd, 2H, *J* = 7.6 Hz, *J* = 7.6 Hz), 7.41 (d, 2H, *J* = 6.8 Hz), 7.37-7.28 (m, 11H), 6.36 (d, 1H, *J* = 9.2 Hz), 6.30 (s, 1H), 5.68 (d, 1H, *J* = 7.6 Hz), 1.46 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.79, 139.32, 139.13, 131.92, 129.11, 128.39, 128.36, 128.35, 128.14, 128.01, 127.99, 127.75, 121.57, 88.11, 85.51, 83.17, 80.87, 75.76, 28.25.

HRMS calcd.for C27H27NO4Na [M+Na]+: 452.1832, found: 452.1831.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.6 min (major), t_R = 10.4 min (minor).



4o

tert-butyl (*R*)-(3-phenyl-1-((2-phenylpropan-2-yl)peroxy)prop-2-yn-1-yl)carbamate **4o**

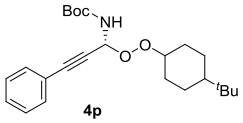
White solid, 83% yield, 31.6 mg, $[\alpha]_D^{20}$= +73.5 (c 1.80 $CHCl_3$).

^{1}H-NMR (600 MHz, $CDCl_3$): δ (ppm): 7.51 (d, 2H, *J* = 7.8 Hz), 7.44 (d, 2H, *J* = 6.6 Hz), 7.43-7.30 (m, 4H), 7.26 (d, 1H, *J* = 8.4 Hz), 6.27 (d, 1H, *J* = 8.4 Hz), 5.57 (s, 1H), 1.65 (d, 6H, *J* = 7.8 Hz), 1.49 (s, 9H).

^{13}C-NMR (150 MHz, $CDCl_3$): δ (ppm): 153.99, 144.96, 131.90, 128.99, 128.33, 128.08, 127.20, 125.57, 121.69, 85.31, 83.75, 83.06, 80.62, 75.56, 28.27, 26.58.

HRMS calcd.for C23H27NO4Na [M+Na]+: 404.1832, found: 404.1829.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =10.0min (major), t_R = 13.2 min (minor).



4p

tert-butyl (*R*)-(1-((4-(tert-butyl)cyclohexyl)peroxy)-3-phenylprop-2-yn-1-yl)carbamate **4p**

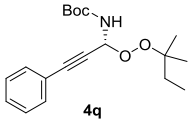
White solid, 81% yield, 32.5 mg, **$[\alpha]_D^{20}$**= +92.7 (c 0.60 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.46 (d, 2H, *J* = 6.4 Hz), 7.37-7.30 (m, 3H), 6.55 (d, 1H, *J* = 10.8 Hz), 5.71 (d, 1H, *J* = 10.4 Hz), 2.37-2.32 (m, 2H), 1.68 (q, 2H, *J* = 10.4 Hz, *J* = 10.8 Hz), 1.57 (dd, 1H, *J* = 4.0 Hz, *J* = 4.4 Hz), 1.49 (s, 9H), 1.35-1.25 (m, 3H), 1.08-1.02 (m, 1H), 0.87 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 155.99, 132.02, 129.42, 128.40, 120.98, 87.08, 82.21, 82.16, 81.08, 76.95, 47.49, 32.34, 29.78, 28.18, 27.66, 23.62, 22.98.

HRMS calcd.for C24H35NO4Na [M+Na]+: 424.2458, found: 424.2462.

HPLC analysis: Daicel CHIRALCEL AD-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =5.8 min (major), t_R = 11.8 min (minor).



4q

tert-butyl (*R*)-(1-(tert-pentylperoxy)-3-phenylprop-2-yn-1-yl)carbamate **4q**

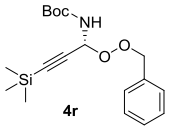
White solid, 82% yield, 27.3 mg, **$[\alpha]_D^{20}$**= +98.8 (c 0.64 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45 (dd, 2H, *J* = 7.2 Hz, *J* = 8.0 Hz), 7.37-7.29 (m, 3H), 6.23 (d, 1H, *J* = 9.6 Hz), 5.59 (d, 1H, 7.6 Hz), 1.60 (q, 2H, *J* = 7.6 Hz, *J* = 7.6 Hz), 1.48 (s, 9H), 1.24 (s, 9H), 0.91 (t, 3H, *J* = 7.6 Hz, *J* = 7.6 Hz).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.98, 131.90, 128.94, 128.30, 121.74, 85.12, 83.33, 83.21, 80.55, 75.41, 31.73, 28.25, 23.91, 8.39.

HRMS calcd.for C19H27NO4Na [M+Na]+: 356.1832, found: 356.1838.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =4.4 min (minor), t_R = 6.8 min (major).



4r

tert-butyl (*R*)-(1-(benzylperoxy)-3-(trimethylsilyl)prop-2-yn-1-yl)carbamate **4r**

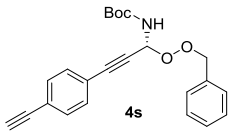
Colorless oil, 87% yield, 30.5 mg, $[\alpha]_D^{20}$= +183.5 (c 0.40 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.40-7.37 (m, 2H), 7.36-7.31 (m, 3H), 6.07 (d, 1H, *J* = 8.8 Hz), 5.55 (d, 1H, *J* = 6.0 Hz), 5.09 (q, 2H, *J* = 10.8 Hz, *J* = 10.4 Hz), 1.44 (s,9H), 0.20 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 154.19, 135.58, 129.64, 128.91, 128.82, 98.95, 91.94, 81.26, 78.13, 75.51, 28.63, 0.00.

HRMS calcd.for C18H27NO4SiNa [M+Na]+: 372.1602, found: 372.1608.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 220 nm, retention time: t_R =6.1 min (minor), t_R = 7.6 min (major).



4s

tert-butyl (*R*)-(1-(benzylperoxy)-3-(4-ethynylphenyl)prop-2-yn-1-yl)carbamate **4s**

White solid, 82% yield, 31.0 mg, $[\alpha]_D^{20}$= +155.3 (c 1.35 $CHCl_3$).

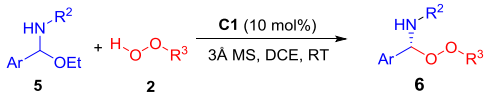
^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.45 (d, 2H, *J* = 8.4 Hz), 7.41-7.39 (m, 4H), 7.38-7.32 (m, 3H), 6.32 (d, 1H, *J* = 12.0 Hz), 5.67 (d, 1H, *J* = 6.4 Hz), 5.13 (s, 2H), 3.19 (s, 1H), 1.47 (s, 9H).

^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 153.80, 135.20, 132.08, 131.78, 129.23, 128.55, 128.47, 122.92, 121.88, 84.75, 82.95, 81.00, 79.37, 77.86, 75.47, 28.24.

HRMS calcd.for C23H23NO4Na [M+Na]+: 400.1519, found: 400.1516.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 85/15, flow rate = 1.0 mL/min, λ = 254 nm, retention time: t_R =9.0 min (minor), t_R = 13.5 min (major).

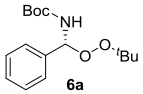
Catalytic asymmetric synthesis of chiral *C*-aryl *α*-amino peroxides 6



To a solution of **5** (0.1 mmol) and **2** (0.2 mmol) in DCE (1.0 mL) was added the catalyst **C1** (10 mol %) and 3Å MS (100 mg) at room temperature. After *C*-aryl-*N*-Boc-*N*,*O*-acetal **5** was consumed, the mixture was directly purified by silica gel chromatography (ethyl acetate/petroleum

ether = 1/40 to 1/20) to afford the product **6** (the racemic product **6** was obtained by using 1,1'-binaphthyl-2,2'-diyl hydrogenphosphate as the catalyst).

Characterization data of the products 6



6a

tert-butyl (*R*)-((tert-butylperoxy)(phenyl)methyl)carbamate **6a**

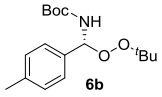
White solid, 90% yield, 26.6 mg, $[\alpha]_D^{20}$= +17.8 (c 0.90 $CHCl_3$).

^{1}H-NMR (600 MHz, $CDCl_3$): δ (ppm): 7.36 (d, 2H, *J* = 7.2 Hz), 7.31-7.19 (m, 3H), 6.34 (d, 1H, *J* = 9.6 Hz), 5.24 (d, 1H, *J* = 7.8 Hz), 1.41 (s, 9H), 1.22 (s, 9H).

^{13}C-NMR (150 MHz, $CDCl_3$): δ (ppm): 154.98, 136.78, 128.79, 128.53, 126.49, 85.51, 80.90, 80.06, 28.31, 26.42.

HRMS calcd.for C16H25NO4Na [M+Na]+: 318.1676, found: 318.1680.

HPLC analysis: Daicel CHIRALCEL ID-3, *n*-hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min, λ = 215 nm, retention time: t_R =8.1 min (minor), t_R = 10.3 min (major).



6b

tert-butyl (*R*)-((tert-butylperoxy)(p-tolyl)methyl)carbamate **6b**

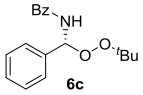
White solid, 92% yield, 28.5 mg, $[\alpha]_D^{20}$= +39.3 (c 0.96 $CHCl_3$).

^{1}H-NMR (600 MHz, $CDCl_3$): δ (ppm): 7.31 (d, 2H, *J* = 7.8 Hz), 7.17 (d, 2H, *J* = 7.8 Hz), 6.38 (d, 1H, *J* = 9.6 Hz), 5.29 (d, 1H, *J* = 7.2 Hz), 2.35 (s, 3H), 1.48 (s, 9H), 1.29 (s, 9H).

^{13}C-NMR (150 MHz, $CDCl_3$): δ (ppm): 154.98, 138.66, 133.77, 129.19, 126.39, 125.81, 85.47, 80.82, 79.94, 28.30, 26.41, 21.20.

HRMS calcd.for C17H27NO4Na [M+Na]+: 332.1832, found: 332.1829.

HPLC analysis: Daicel CHIRALCEL ID-3, *n*-hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min, λ = 220 nm, retention time: t_R =5.4 min (major), t_R = 6.4 min (minor).



6c

(*R*)-N-((tert-butylperoxy)(phenyl)methyl)benzamide **6c**

White solid, 94% yield, 28.2 mg, $[\alpha]_D^{20}$= +28.2 (c 0.20 $CHCl_3$).

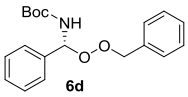
^{1}H-NMR (600 MHz, $CDCl_3$): δ (ppm): 7.85 (d, 2H, *J* = 7.2 Hz), 7.53-7.49 (m, 3H), 7.50-7.43 (m,

2H), 7.41-7.35 (m, 3H), 6.91 (d, 1H, *J* = 9.0 Hz), 6.86 (d, 1H, *J* = 9.0 Hz), 1.30 (s, 9H).

^{13}C-NMR (150 MHz, $CDCl_3$): δ (ppm): 166.96, 136.87, 134.01, 131.93, 128.97, 128.68, 127.18, 126.53, 84.37, 81.31, 26.47.

HRMS calcd.for C18H21NO3Na [M+Na]+: 322.1414, found: 322.1417.

HPLC analysis: Daicel CHIRALCEL ID-3, *n*-hexane/*i*-PrOH = 95/5, flow rate = 1.0 mL/min, λ = 230 nm, retention time: t_R =13.0 min (minor), t_R = 18.4 min (major).



6d

tert-butyl (*R*)-((benzylperoxy)(phenyl)methyl)carbamate **6d**

White solid, 91% yield, 30.0 mg, $[\alpha]_D^{20}$= +42.9 (c 0.57 $CHCl_3$).

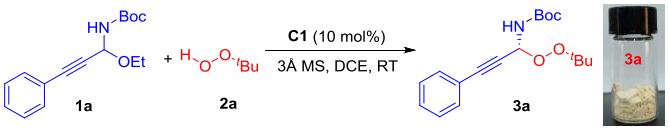
^{1}H-NMR (600 MHz, $CDCl_3$): δ (ppm): 7.38-7.32 (m, 10H), 6.50 (d, 1H, *J* = 9.6 Hz), 5.33 (d, 1H, *J* = 7.8 Hz), 5.06 (q, 2H, *J* = 11.4 Hz, *J* = 11.4 Hz), 1.48 (s, 9H).

^{13}C-NMR (150 MHz, $CDCl_3$): δ (ppm): 154.76, 136.33, 125.67, 129.22, 128.97, 128.59, 128.39, 126.51, 125.94, 85.13, 81.96, 80.39, 63.54, 28.31.

HRMS calcd.for C19H23NO4Na [M+Na]+: 352.1519, found: 352.1517.

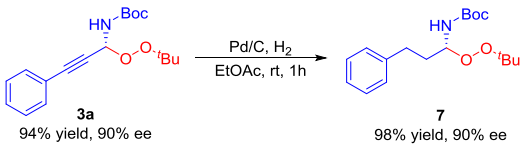
HPLC analysis: Daicel CHIRALCEL ID-3, *n*-hexane/*i*-PrOH = 80/20, flow rate = 1.0 mL/min, λ = 220 nm, retention time: t_R =14.3 min (major), t_R = 15.8 min (minor).

Large-Scale catalytic asymmetric reactions



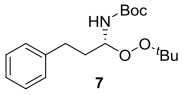
To a solution of **1a** (4.0 mmol) and **2a** (8.0 mmol) in DCE (40.0 mL) was added the catalyst **C1** (10 mol %) and 3Å MS (4g) at room temperature. After *C*-alkynyl-*N*-Boc-*N*,*O*-acetal **1a** was consumed, the mixture was directly purified by silica gel chromatography (ethyl acetate/petroleum ether = 1/40 to 1/20) to afford the product 3a (1.19 g, 94% yield and 90% ee)

General procedure for synthesis and characterization data of the products 7



An oven-dried 10 mL Schlenk tube equipped with a stirring bar and capped with a rubber septum

was charged with Pd/C (0.01 mmol Pd). The tube was degassed and backfilled with hydrogen gas (3 times). Under a positive hydrogen pressure (hydrogen-filled balloon), degassed EtOAc (1mL) was added into the tube via a syringe, followed by the addition of **3a** (32 mg, 0.1 mmol). The reaction mixture was stirred at room temperature under a hydrogen atmosphere (hydrogen-filled balloon). Upon completion of the reaction in 1 h (monitored by TLC), the reaction mixure was filtered with a pad of celite to remove Linder catalyst, and the filtrate was concentrated in vacuo. The organic layer was further purified by flash column chromatography on silica gel to give **7**.



7

tert-butyl (*R*)-(1-(tert-butylperoxy)-3-phenylpropyl)carbamate **7**

White solid, 98% yield, 31.7 mg, $[\alpha]_D^{20}$= +48.6 (c 1.50 $CHCl_3$).

^{1}H-NMR (400 MHz, $CDCl_3$): δ (ppm): 7.28 (d, 2H, *J* = 7.2 Hz), 7.19 (d, 3H, *J* = 6.0 Hz), 5.39 (d, 1H, *J* = 7.6 Hz), 5.03 (d, 1H, *J* = 9.2 Hz), 2.73 (t, 2H, *J* = 7.6 Hz, *J* = 7.2 Hz), 2.03 (q, 1H, *J* = 6.8 Hz, *J* = 6.8 Hz), 1.85-1.79 (m, 1H), 1.46 (S, 9H), 1.24 (s, 9H).

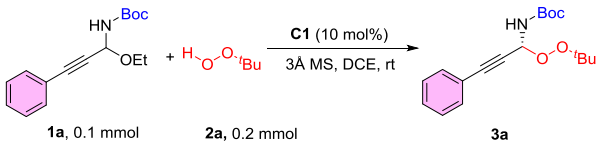
^{13}C-NMR (100 MHz, $CDCl_3$): δ (ppm): 155.14, 141.13, 128.45, 128.39, 126.04, 84.44, 80.29, 79.71, 34.22, 31.37, 28.30, 26.38.

HRMS calcd.for C18H29NO4Na [M+Na]+: 346.1989, found: 346.1992.

HPLC analysis: Daicel CHIRALCEL IC-3, *n*-hexane/*i*-PrOH = 90/10, flow rate = 1.0 mL/min, λ = 218 nm, retention time: t_R =4.3 min (minor), t_R = 5.3 min (major).

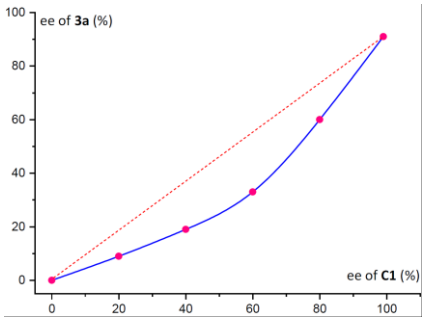
The experiments of non-linear effect and product ee dependence on time

(1) non-linear effect (NLE)



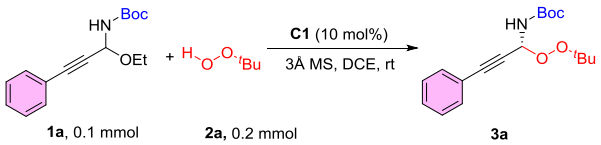
1a, 0.1 mmol **2a,** 0.2 mmol **3a**

entry	ee of **C1**%	ee of **3a**%
1	99	92
2	80	60
3	60	33
4	40	19
5	20	9
6	0	0

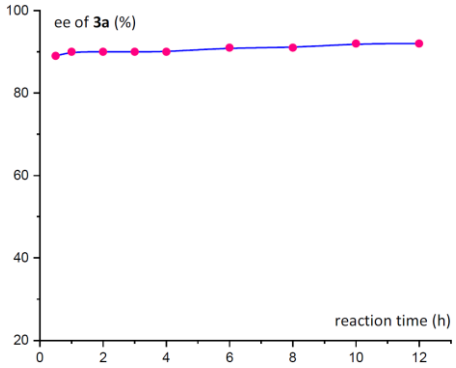


(2) **product ee dependence on time**

The enantiopurity of the product was kept with reaction time, indicating the reaction isn't a progress of smiple kinetic resolution.



1a, 0.1 mmol **2a,** 0.2 mmol **3a**



Proposed transition states for the asymmetric peroxidation of C-alkynyl imines.

A plausible transition state model was proposed on the basis of the absolute configuration of the product, the control experiments and the pervious works of the chiral calcium phosphate catalysts[4] (Figure 1), demonstrating to account for the chemistry and stereochemistry of the reaction. The chiral calcium phosphate as a bifunctional catalyst is able to simultaneously activate both *C*-alkynyl imine *in situ* generated and hydroperoxide by multiple hydrogen bonds. Subsequently, the interaction could provide an enantioselective environment to urge to hydroperoxide attack the *Si* face of carbon-nitrogen double bond in *C*-alkynyl imine , forming the product with high enantioselectivity.

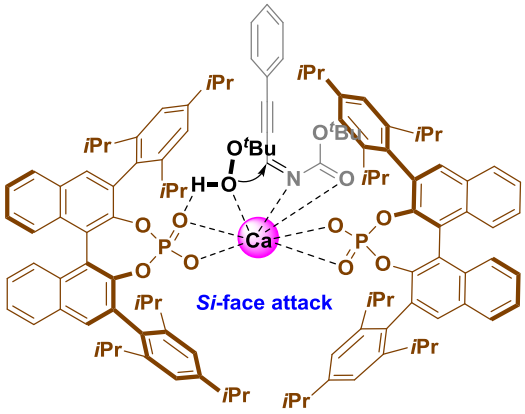


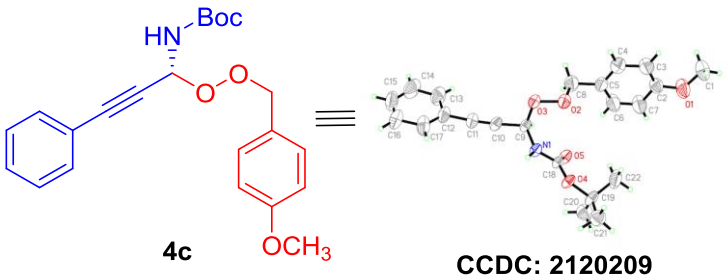
Figure 1. Plausible transition state model

Determination of the Product Stereochemistry

The absolute stereochemistry of the *C*-alkynynl **4** were based on single crystal X-ray crystallography of **4c.** The X-ray data have been deposited at the Cambridge Crystallographic Data Center (CCDC 2120219). The stereochemistry of other products was assumed by analogy. And the absolute configurations of **6** was comfirmed by comparing of the optical rotation of **6c** with literature value.

(i) X-ray crystallography.

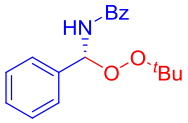
The proper chiral single crystal was obtained by slow evaporation of a ^{i}PrOH solution of **4c** at room temperature.



4c **CCDC: 2120209**

Identification code	2
Empirical formula	C22 H25 N O5
Formula weight	383.43
Temperature	298(2) K
Wavelength	1.54178 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 9.7745(14) A alpha = 90 deg.
	b = 9.5549(14) A beta = 91.267(9) deg.
	c = 11.5736(17) A gamma = 90 deg.
Volume	1080.6(3) A^3
Z, Calculated density	2, 1.178 Mg/m^3
Absorption coefficient	0.682 mm^-1
F(000)	408
Crystal size	0.220 x 0.180 x 0.160 mm
Theta range for data collection	3.820 to 65.403 deg.
Limiting indices	-11<=h<=9, -11<=k<=11, -13<=l<=13
Reflections collected / unique	9944 / 3553 [R(int) = 0.0909]
Completeness to theta = 65.403	97.5 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.897 and 0.863
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3553 / 21 / 258
Goodness-of-fit on F^2	1.099
Final R indices [I>2sigma(I)]	R1 = 0.0811, wR2 = 0.2370
R indices (all data)	R1 = 0.1146, wR2 = 0.2647
Absolute structure parameter	-0.03(17)
Extinction coefficient	0.040(12)
Largest diff. peak and hole	0.422 and -0.270 e.A^-3

(ii) Comparison of the optical rotation value with the literature data.



6c

The literature optical rotation value for (*R*)-**6c** in 86% ee was reported to be $[\alpha]_D^{20}$ =+32.9 (c = 1.43, $CHCl_3$).[3] The measured value of our product in 95% ee was $[\alpha]_D^{20}$:+28.2 (c = 0.20, $CHCl_3$). Thus, the absolute configuration of our product was assigned to be *R*.

References

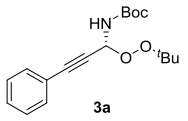
[1] Y.-C. Wang, M.-J. Mo, K.-X. Zhu, C. Zheng, H.-B. Zhang, W. Wang and Z.-H. Shao, *Nat. Commun.* 2015, **6**, 8544.

[2] (a) Y.-X. Hou, J.-J. Hu, R.-G. Xu, S.-L. Pan, X.-F. Zeng and G.-F. Zhong, *Org. Lett.* **2019**, *21*, 4428. (b) T. G. Driver, J. R. Hason and K. S. Woerpel, *J. Am. Chem. Soc.* **2007**, *129*, 3836.

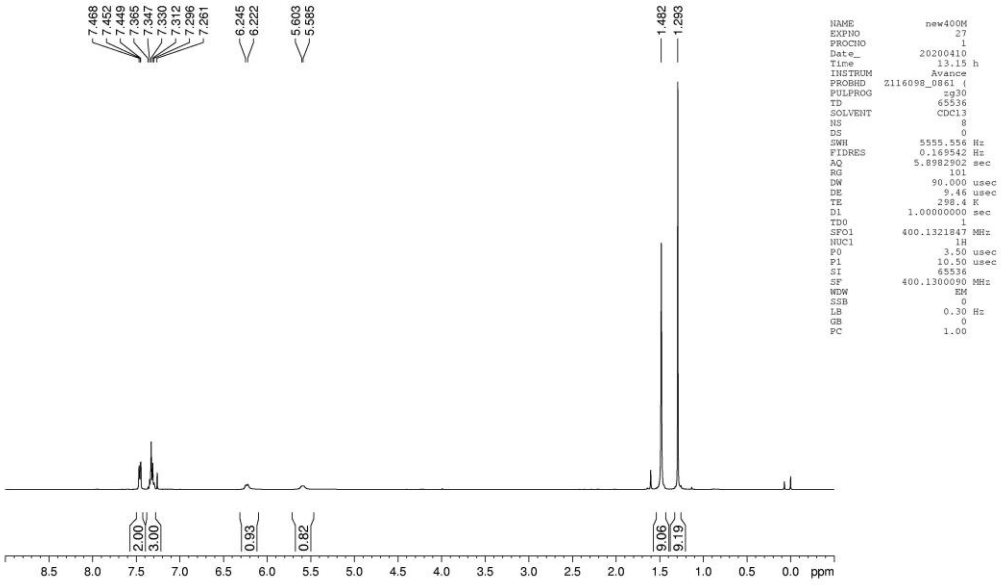
[3] W. H. Zheng, L. Wojtas and J. C. Antilla, *Angew. Chem. Int. Ed.* **2010**, *49*, 6589.

[4] (a) L. Simón and R. S. Paton, *J. Am. Chem. Soc.*, **2018**, *140*, 5412; (b) X.-T. Fang, Z.-H. Deng, W.-H. Zheng and J. C. Antilla, *ACS Catal.*, **2019**, ***9***, 1748; (c) R. Cao and J. C. Antilla, *Org. Lett.*, **2020**, ***22***, 5958; (d) R.-H. Liu, S. Krishnamurthy, Z.-W. Wu, K. S. S. Tummalapalli and J. C. Antilla, *Org. Lett.*, **2020**, *22*, 8101.

Copies of NMR and HPLC



3a

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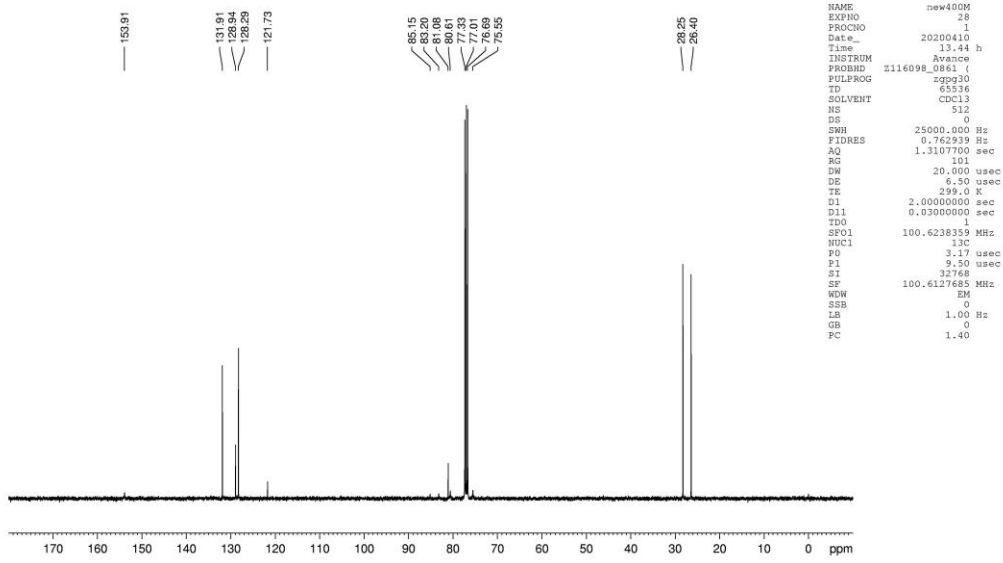


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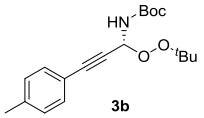
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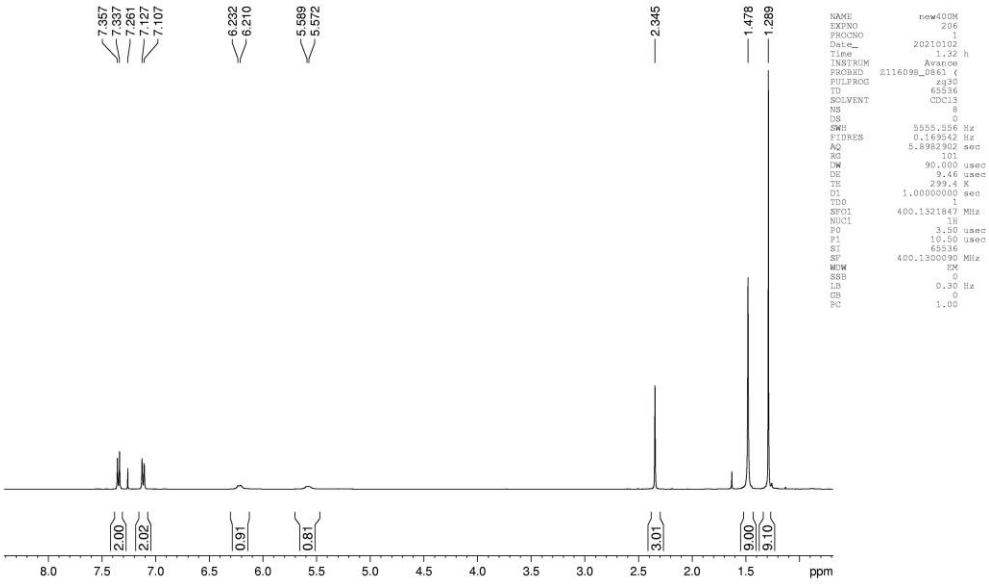
tBu

3b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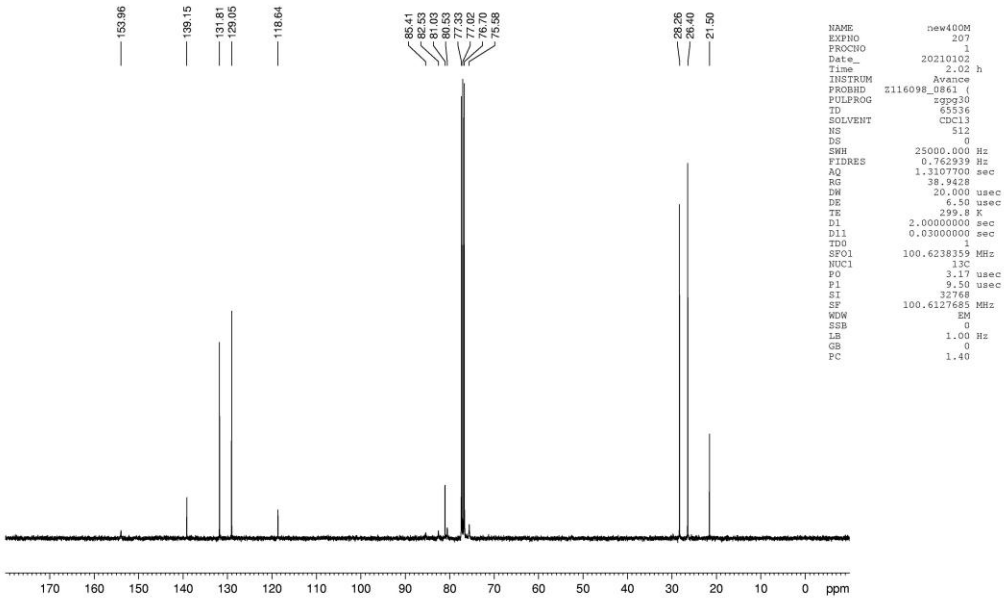
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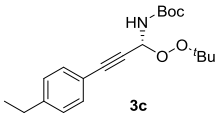
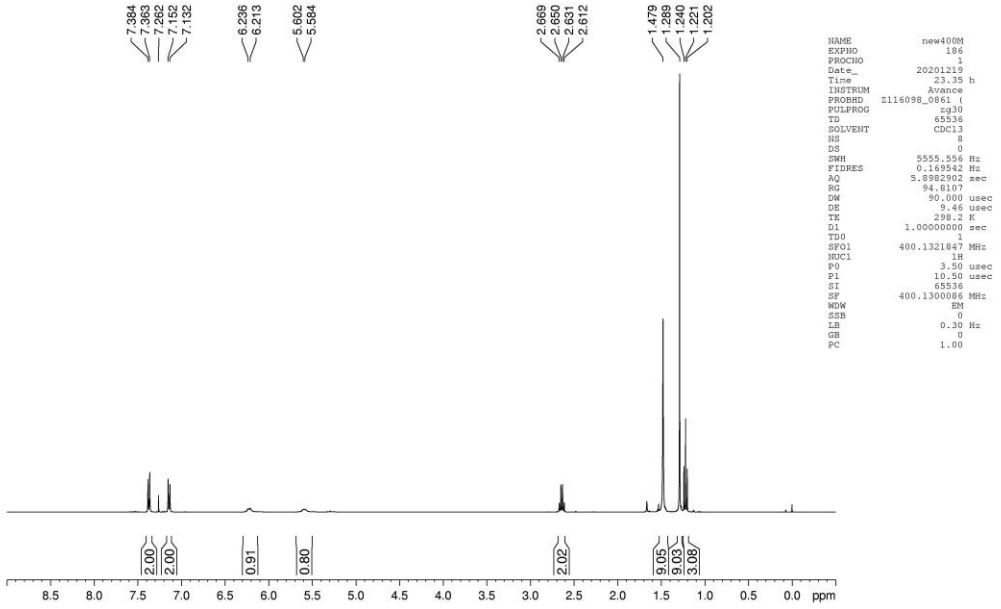


3c

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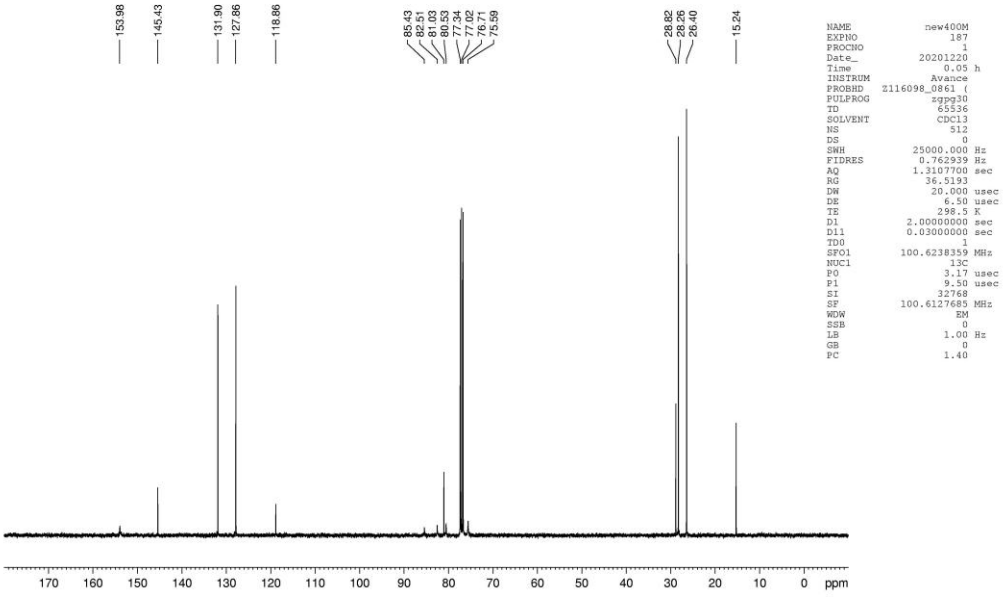


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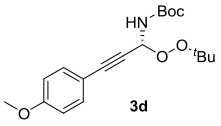
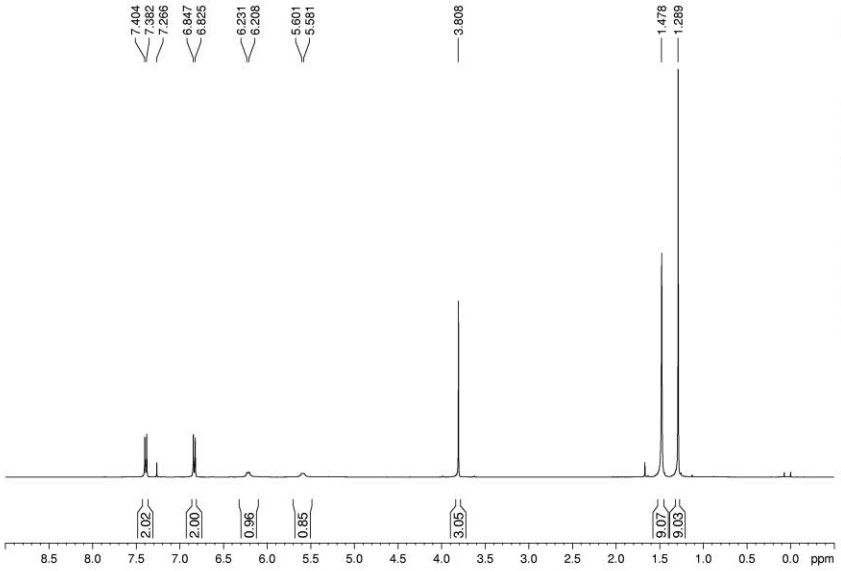
NAME            new400M
EXPNO               187
PROCNO                1
Date_          20201220
Time               0.05 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH           25000.000 Hz
FIDRES         0.762939 Hz
AQ             1.3107700 sec
RG              36.5193
DW                20.000 usec
DE                 6.50 usec
TE                298.5 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
```

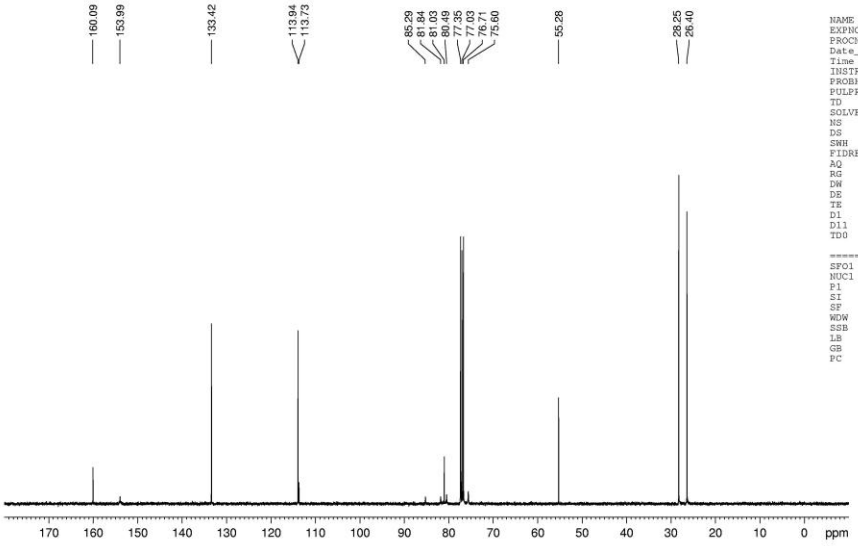


3d



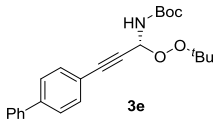

```
NAME                 old400M
EXPNO                    128
PROCNO                     1
Date_               20210102
Time                    3.29
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                         8
DS                         0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                     31.56
DW                    62.400 usec
DE                     6.50 usec
TE                    296.1 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1              400.1522008 MHz
NUC1                      1H
P1                     10.75 usec
SI                     65536
SF                400.1500072 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

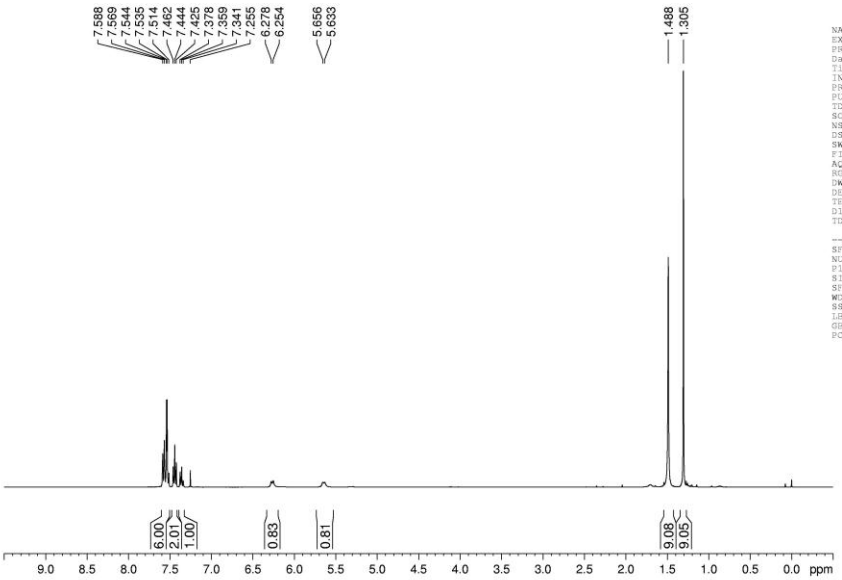



```
NAME                 old400M
EXPNO                    129
PROCNO                     1
Date_               20210102
Time                    3.59
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       512
DS                         0
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                    195.85
DW                    19.800 usec
DE                     6.50 usec
TE                    296.4 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                100.6177980 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

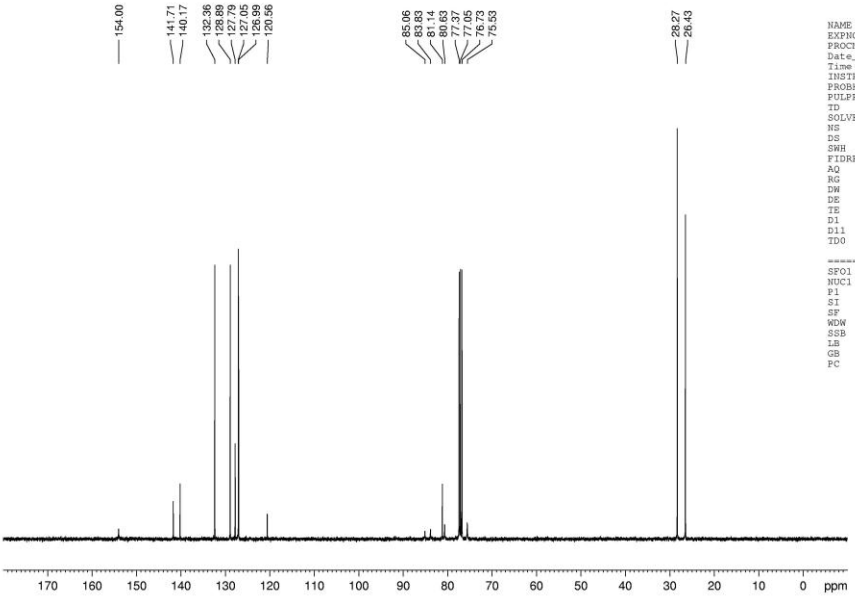


3e



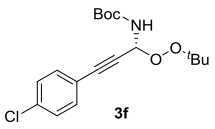

```
NAME                old1400M
EXPNO                    118
PROCNO                     1
Date_               20201228
Time                    0.57
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                      31.56
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
SI                     65536
SF                400.1500116 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

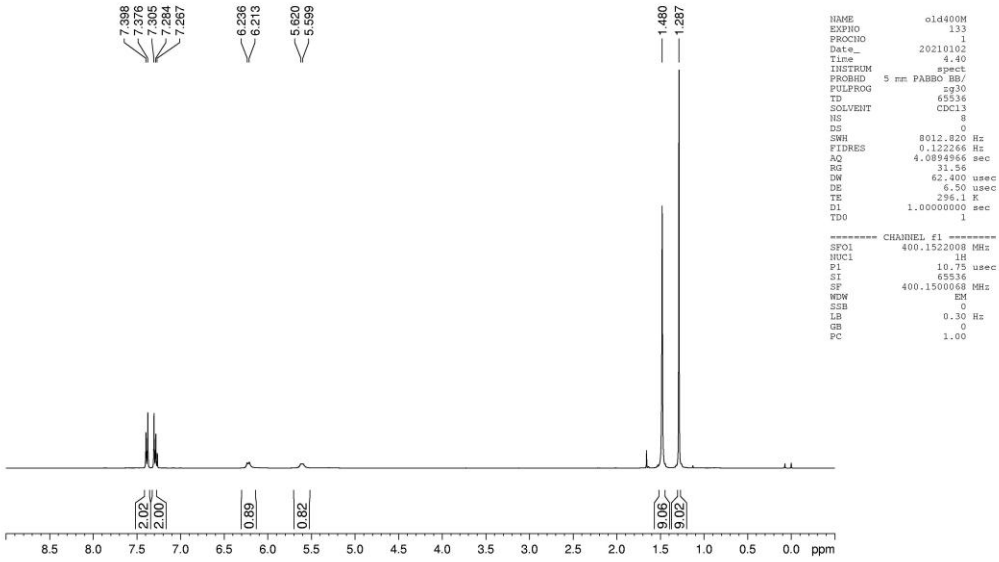



```
NAME                old1400M
EXPNO                    119
PROCNO                     1
Date_               20201228
Time                    1.27
INSTRUM                 spect
PROBHD   5 mm PABBO BB/
PULPROG                 zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       512
DS                         0
SWH                25252.525 Hz
FIDRES              0.385323 Hz
AQ                  1.2976629 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
SI                     32768
SF                100.6177980 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

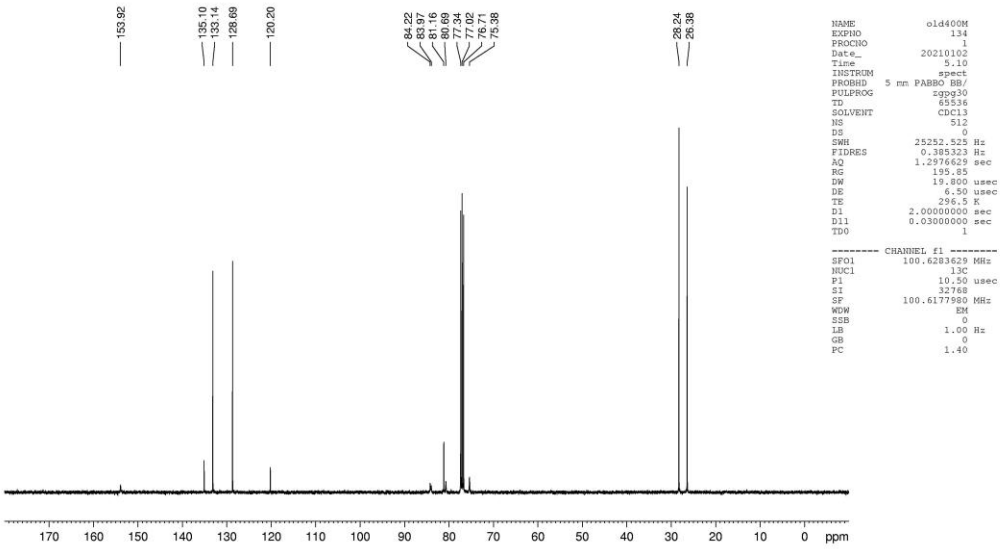


3f



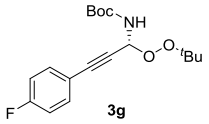

```
NAME              old400M
EXPNO                 133
PROCNO                  1
Date_            20210102
Time                 4.40
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                 31.56
DW                62.400 usec
DE                 6.50 usec
TE                296.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1          400.1522008 MHz
NUC1                    1H
P1                  10.75 usec
SI                  65536
SF            400.1500068 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

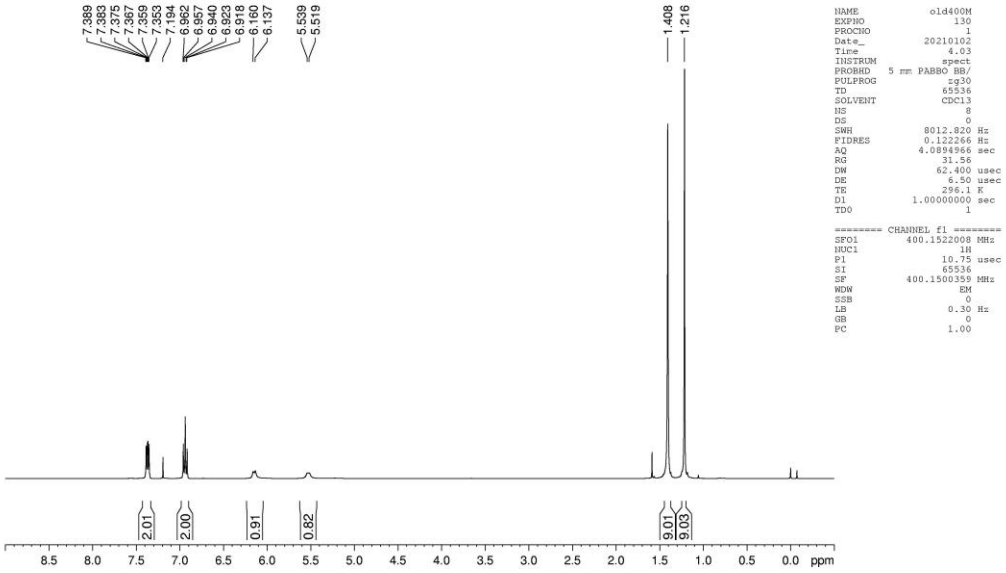



```
NAME              old400M
EXPNO                 134
PROCNO                  1
Date_            20210102
Time                 5.10
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ              1.2976629 sec
RG                195.85
DW                19.800 usec
DE                 6.50 usec
TE                296.5 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            100.6177980 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

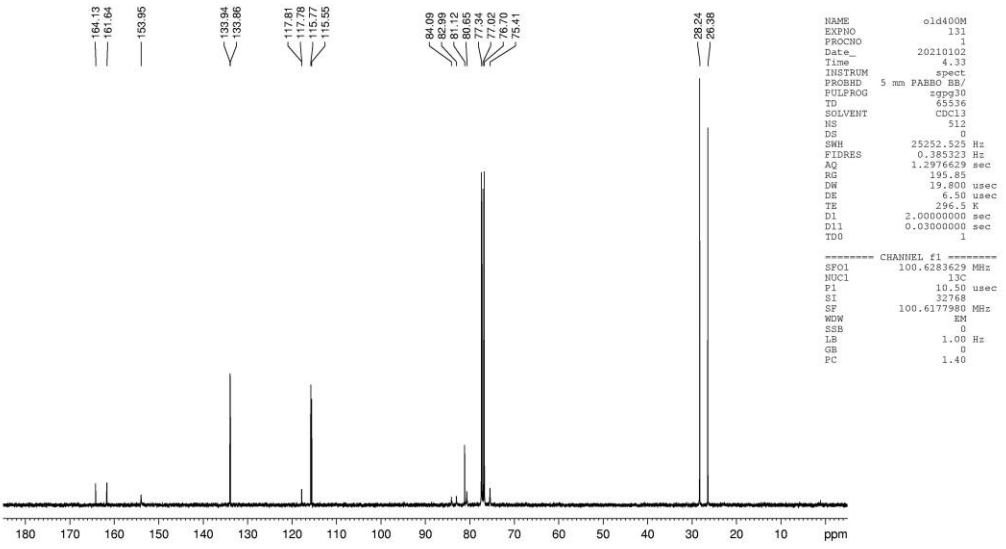


3g



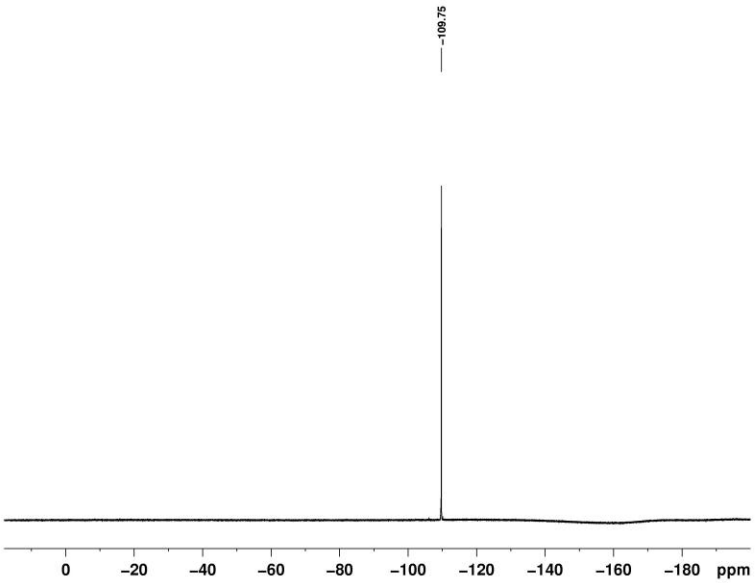

```
NAME              old400M
EXPNO                 130
PROCNO                  1
Date_            20210102
Time                4.03
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8012.820 Hz
FIDRES          0.122266 Hz
AQ              4.0894966 sec
RG                 31.56
DW                62.400 usec
DE                 6.50 usec
TE                296.1 K
D1             1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
SFO1           400.1522008 MHz
NUC1                   1H
P1                 10.75 usec
SI                 65536
SF             400.1500359 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



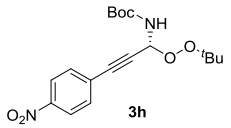

```
NAME              old400M
EXPNO                 131
PROCNO                  1
Date_            20210102
Time                4.33
INSTRUM            spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH            25252.525 Hz
FIDRES          0.385323 Hz
AQ              1.2976629 sec
RG                 195.85
DW                19.800 usec
DE                 6.50 usec
TE                296.5 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             100.6177980 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

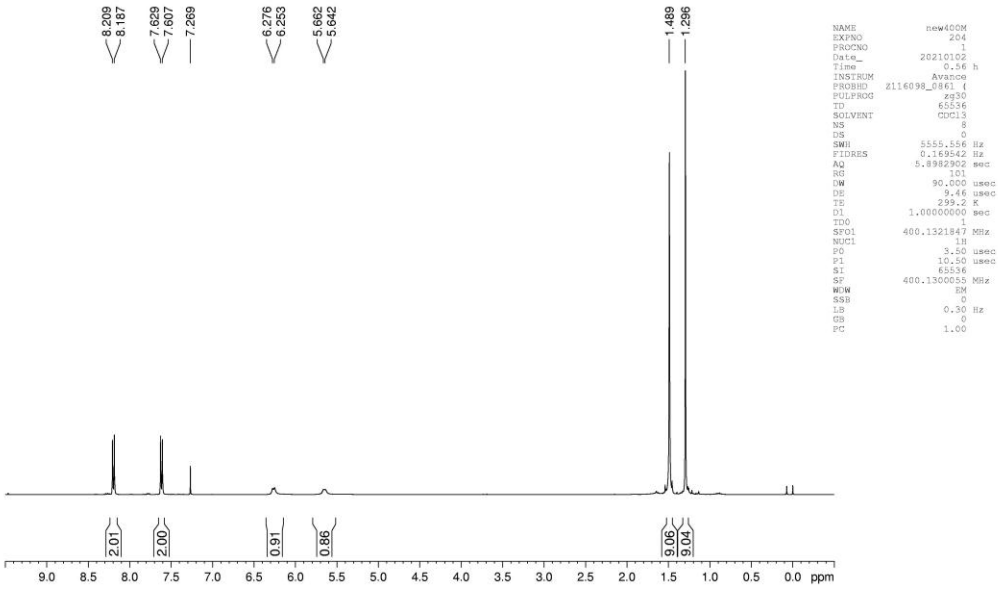



```
NAME                 old400M
EXPNO                    132
PROCNO                     1
Date_               20210102
Time                    4.35
INSTRUM                spect
PROBHD    5 mm PABBO BB/
PULPROG          zgfhigqn.2
TD                     131072
SOLVENT                CDCl3
NS                        16
DS                         4
SWH                89285.711 Hz
FIDRES                 0.681196 Hz
AQ                     0.7340532 sec
RG                       195.85
DW                       5.600 usec
DE                       6.50 usec
TE                       296.4 K
D1                  1.00000000 sec
D11                 0.03000000 sec
D12                 0.00002000 sec
TD0                        1

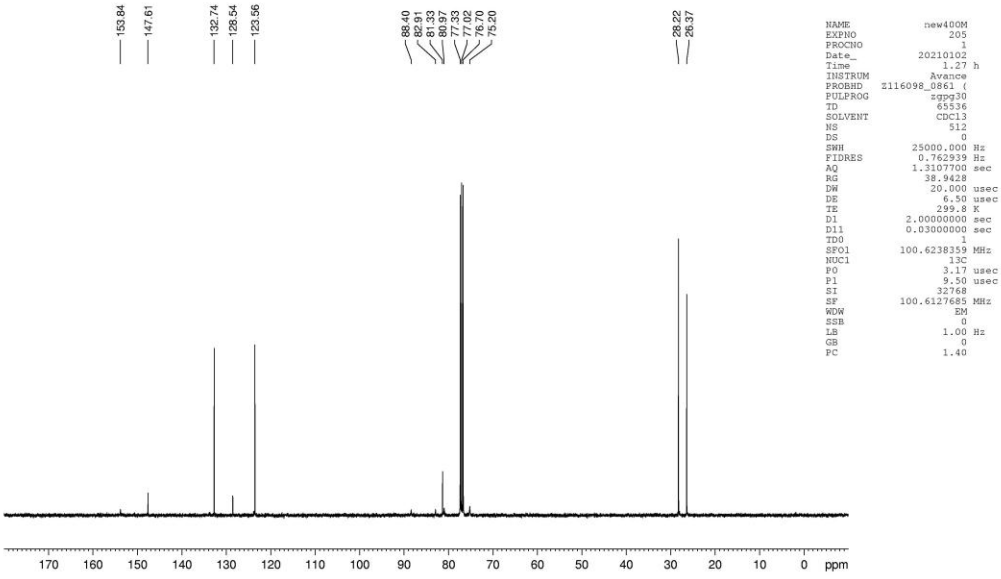
======== CHANNEL f1 ========
SFO1               376.4795333 MHz
NUC1                       19F
P1                       14.00 usec
SI                        65536
SF                 376.5171850 MHz
WDW                       EM
SSB                        0
LB                       0.30 Hz
GB                         0
PC                       1.00
```



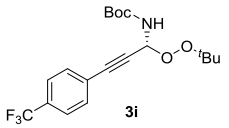
3h



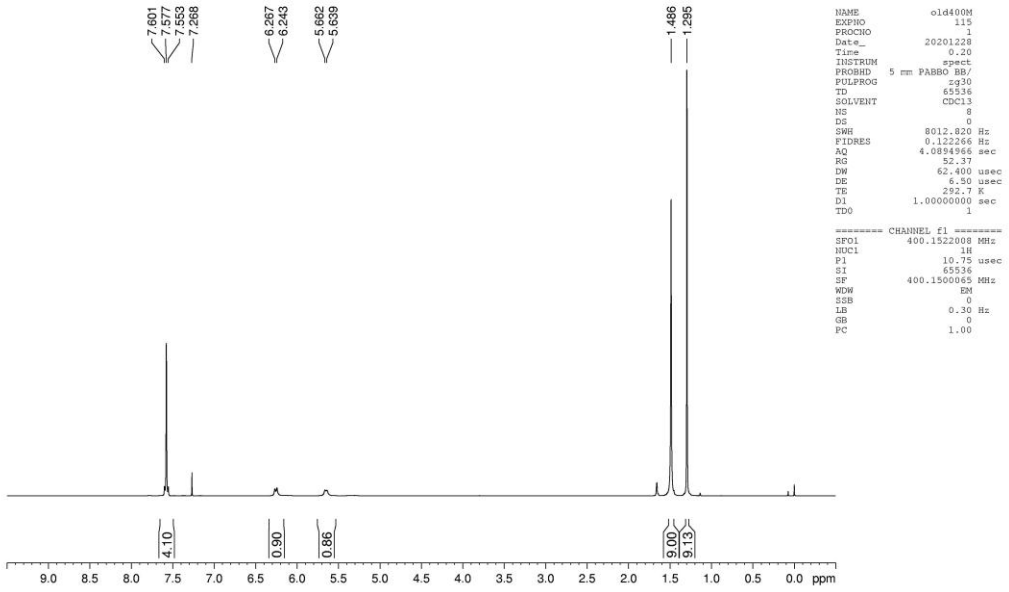
NAME	new400M
EXPNO	204
PROCNO	1
Date_	20210102
Time	0.56 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	101
DW	90.000 usec
DE	9.46 usec
TE	299.2 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.50 usec
P1	10.50 usec
SI	65536
SF	400.1300055 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

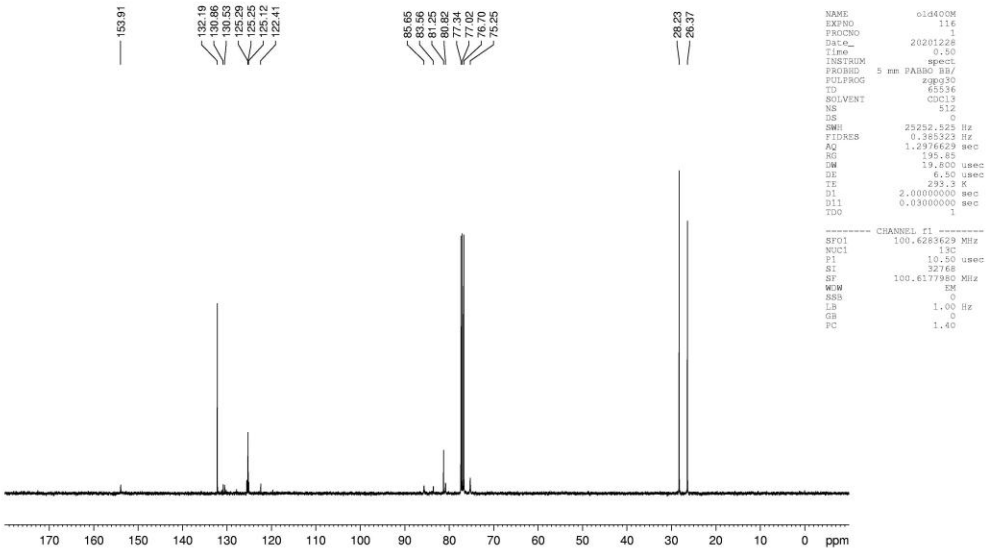


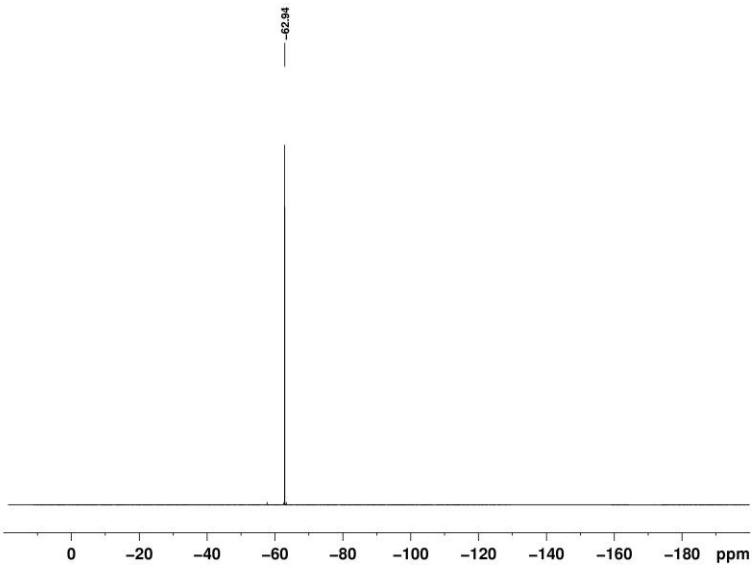
NAME	new400M
EXPNO	205
PROCNO	1
Date_	20210102
Time	1.27 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	38.9428
DW	20.000 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238359 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


Boc
NH
O
O
tBu
F3C

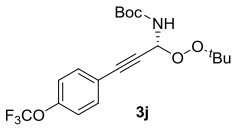
3i


7.601
7.577
7.553
7.268
6.267
6.243
5.662
5.639
1.486
1.295
4.10
0.90
0.86
9.00
9.13
9.0 8.5 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
NAME old400M
EXPNO 115
PROCNO 1
Date_ 20201220
Time 0.20
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 sec
RG 52.37
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
SI 65536
SF 400.1500065 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

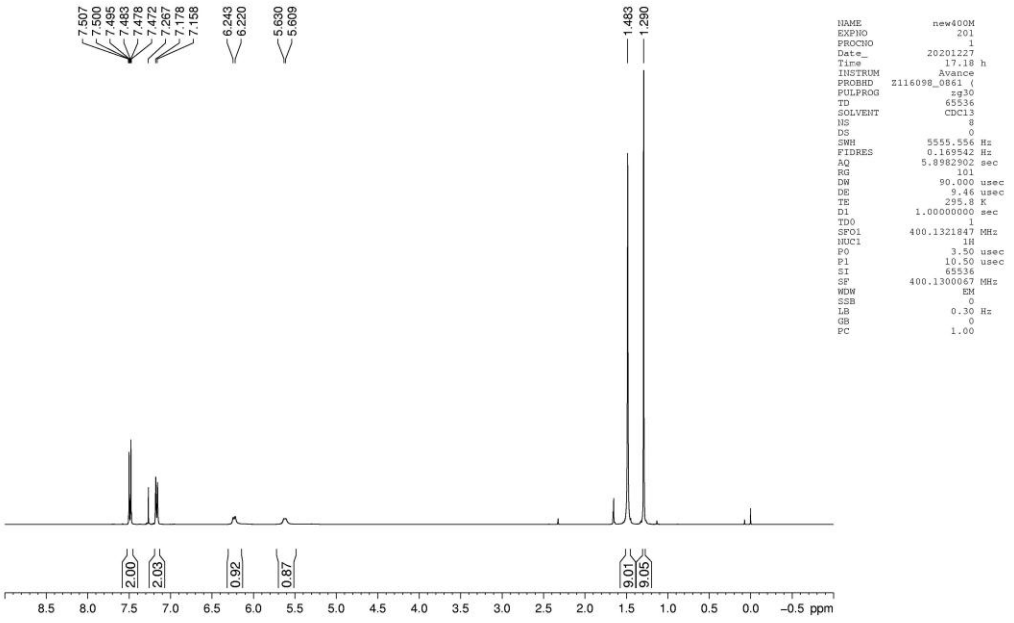

153.91
132.19
130.86
130.53
125.29
125.25
125.12
122.41
85.65
83.56
81.25
80.82
77.34
77.02
76.70
75.25
28.23
26.37
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
NAME old400M
EXPNO 116
PROCNO 1
Date_ 20201228
Time 0.50
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 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
SI 32768
SF 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



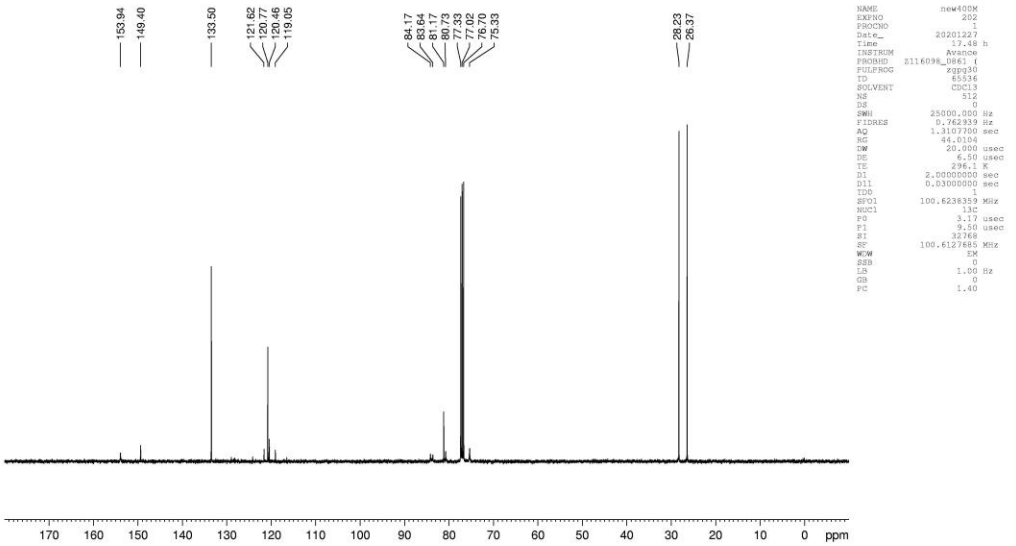
NAME	old400M
EXPNO	117
PROCNO	1
Date_	20201228
Time	0.52
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgfhigqn.2
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	89285.711 Hz
FIDRES	0.681196 Hz
AQ	0.7340532 sec
RG	195.85
DW	5.600 usec
DE	6.50 usec
TE	293.1 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	376.4795333 MHz
NUC1	19F
P1	14.00 usec
SI	65536
SF	376.5171850 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



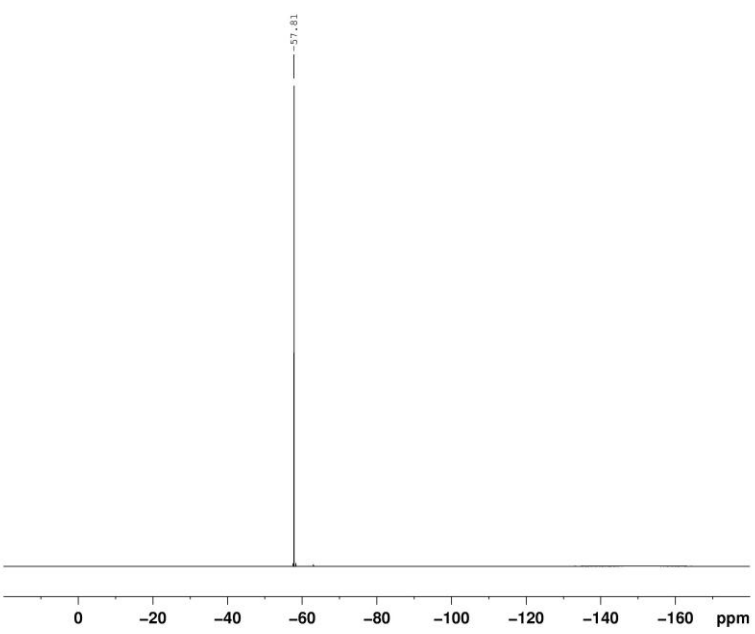
3j



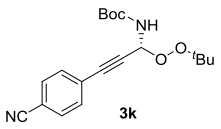

```
NAME            new400M
EXPNO               201
PROCNO                1
Date_          20201227
Time              17.18 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                    8
DS                    0
SWH            5555.556 Hz
FIDRES        0.169542 Hz
AQ             5.8982902 sec
RG                  101
DW              90.000 usec
DE               9.46 usec
TE              295.8 K
D1             1.00000000 sec
TD0                   1
SFO1       400.1321847 MHz
NUC1                1H
P0               3.50 usec
P1              10.50 usec
SI               65536
SF         400.1300067 MHz
WDW                 EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 1.00
```



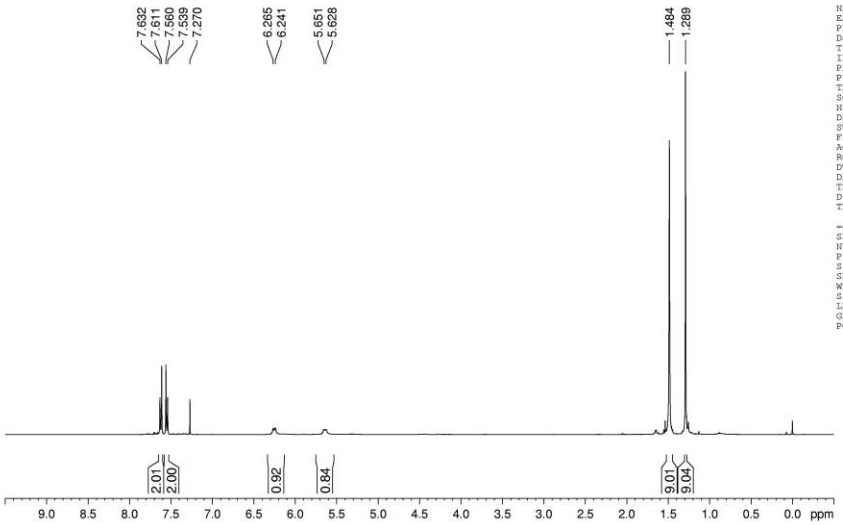

```
NAME            new400M
EXPNO               202
PROCNO                1
Date_          20201227
Time              17.48 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD               65536
SOLVENT          CDCl3
NS                  512
DS                    0
SWH            25000.000 Hz
FIDRES         0.762939 Hz
AQ             1.3107700 sec
RG                44.0104
DW              20.000 usec
DE               6.50 usec
TE              296.1 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	new400M
EXPNO	203
PROCNO	1
Date_	20201227
Time	17.50 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	296.0 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	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

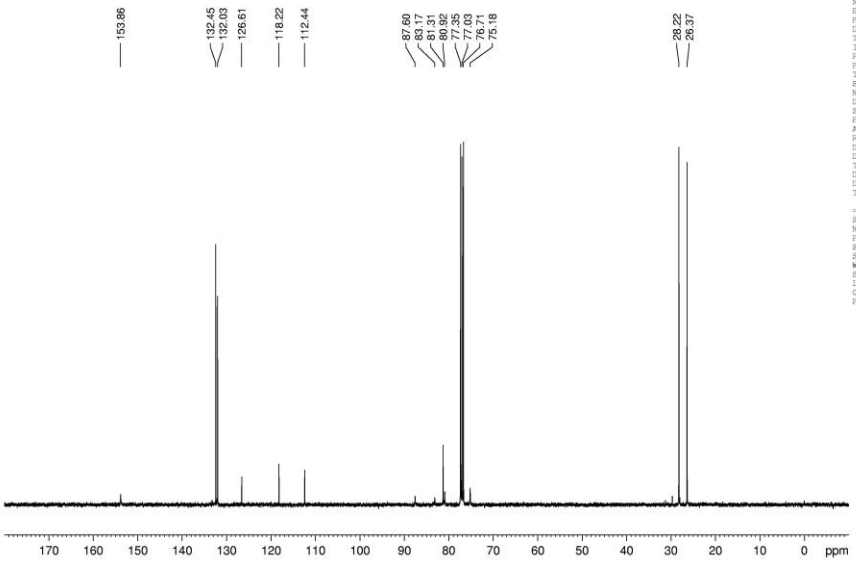


3k



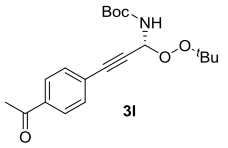

```
NAME                old400M
EXPNO                   169
PROCNO                    1
Date_              20210310
Time                  1.50
INSTRUM              spect
PROBHD     5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                     63.8
DW                62.400 usec
DE                 6.50 usec
TE                 294.8 K
D1                1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
SFO1           400.1522008 MHz
NUC1                     1H
P1                 10.75 usec
SI                   65536
SF             400.1500058 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

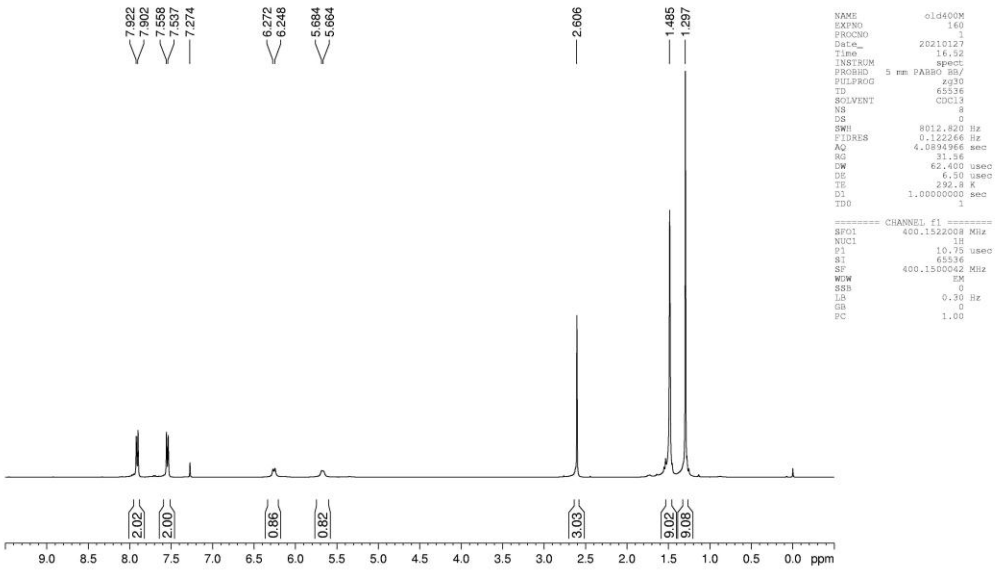



```
NAME                old400M
EXPNO                   170
PROCNO                    1
Date_              20210310
Time                  2.36
INSTRUM              spect
PROBHD     5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     800
DS                       0
SWH              28252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                   195.85
DW                19.800 usec
DE                 6.50 usec
TE                 295.3 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             100.6177980 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.40
```

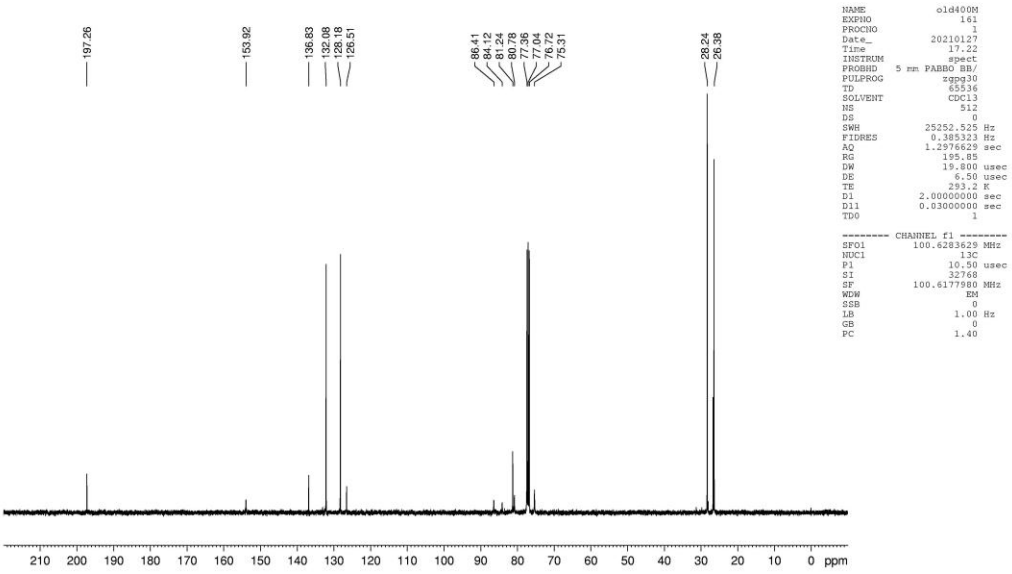


3l



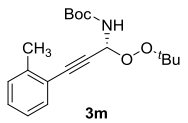

```
NAME                old400M
EXPNO                   160
PROCNO                    1
Date_              20210127
Time                  16.52
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                   292.8 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                     1H
P1                    10.75 usec
SI                    65536
SF               400.1500042 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

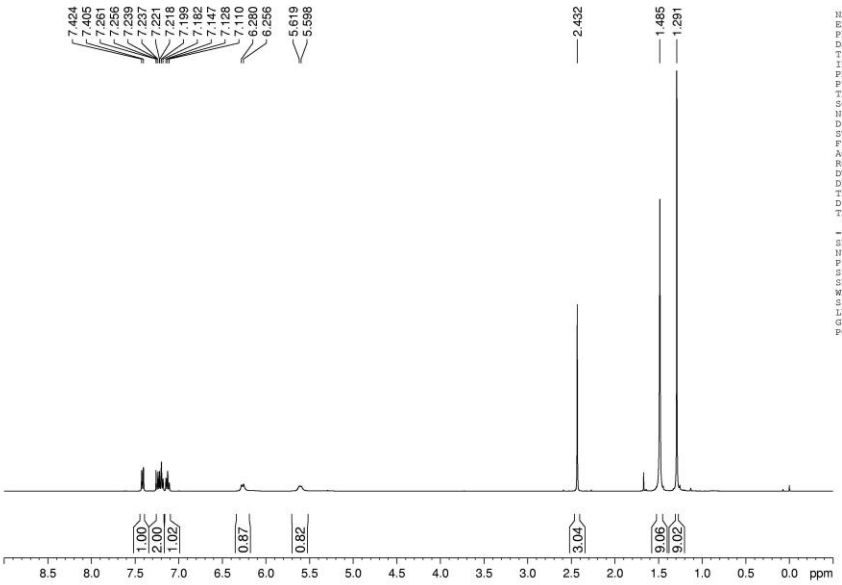



```
NAME                old400M
EXPNO                   161
PROCNO                    1
Date_              20210127
Time                  17.22
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                    195.85
DW                   19.800 usec
DE                    6.50 usec
TE                   293.2 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               100.6177980 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

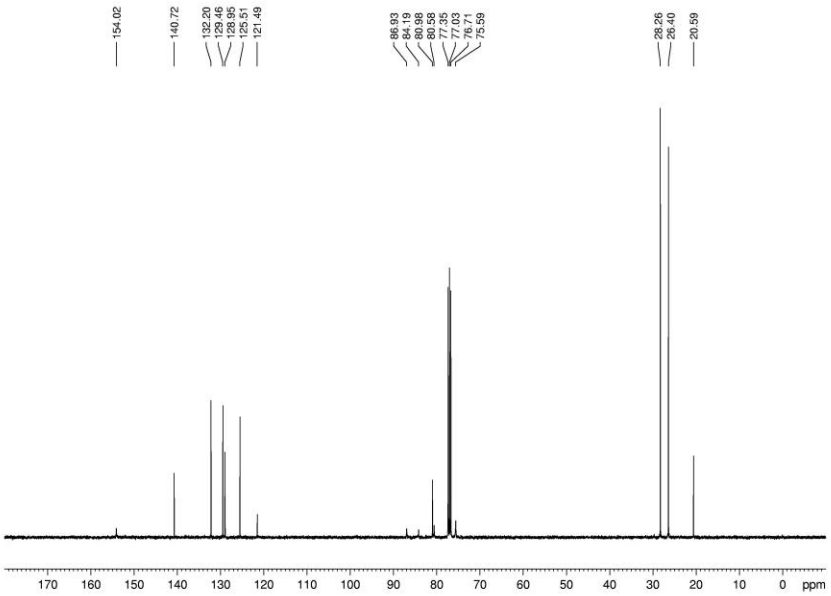


3m



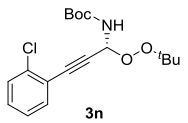

```
NAME                old400M
EXPNO                   122
PROCNO                    1
Date_              20210102
Time                  1.47
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                   295.9 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                       1H
P1                    10.75 usec
SI                     65536
SF               400.1500091 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                    1.00
```

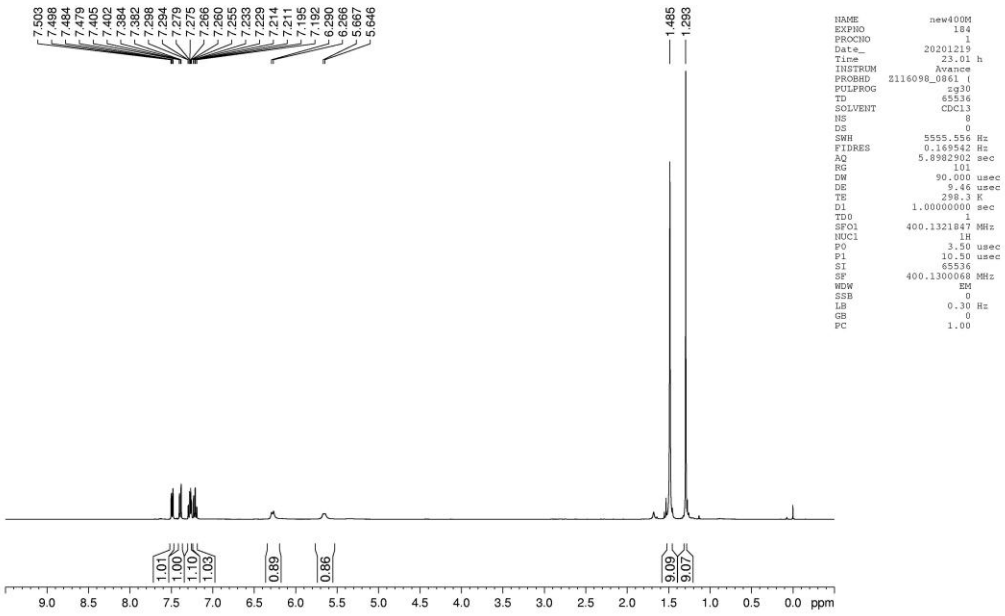



```
NAME                old400M
EXPNO                   123
PROCNO                    1
Date_              20210102
Time                  2.16
INSTRUM              spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                   195.85
DW                   19.800 usec
DE                    6.50 usec
TE                   296.3 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

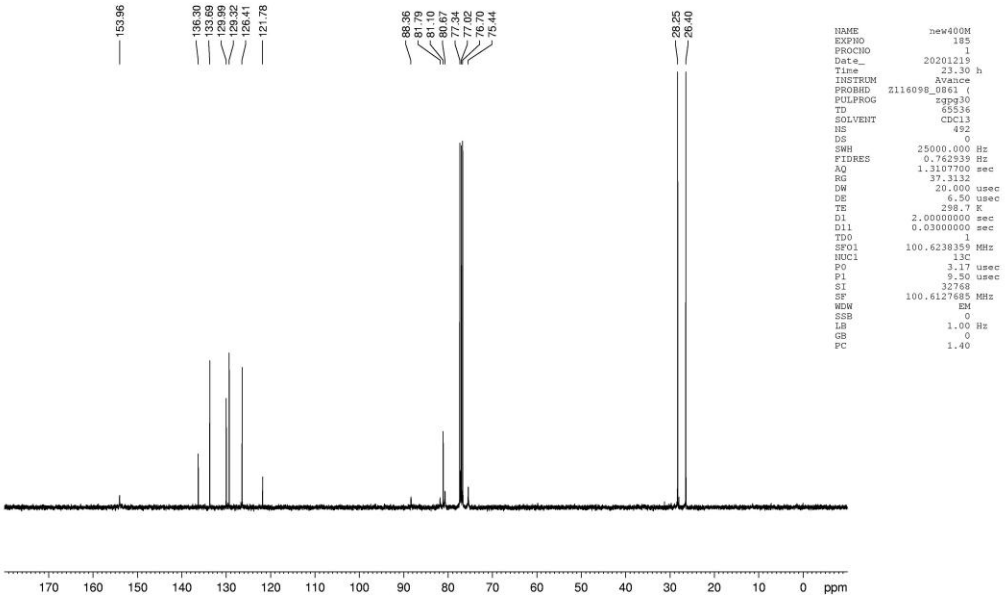
======== CHANNEL f1 ========
SFO1             100.6283629 MHz
NUC1                      13C
P1                    10.50 usec
SI                     32768
SF               100.6177980 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                       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



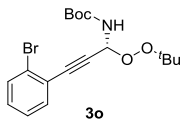
3n



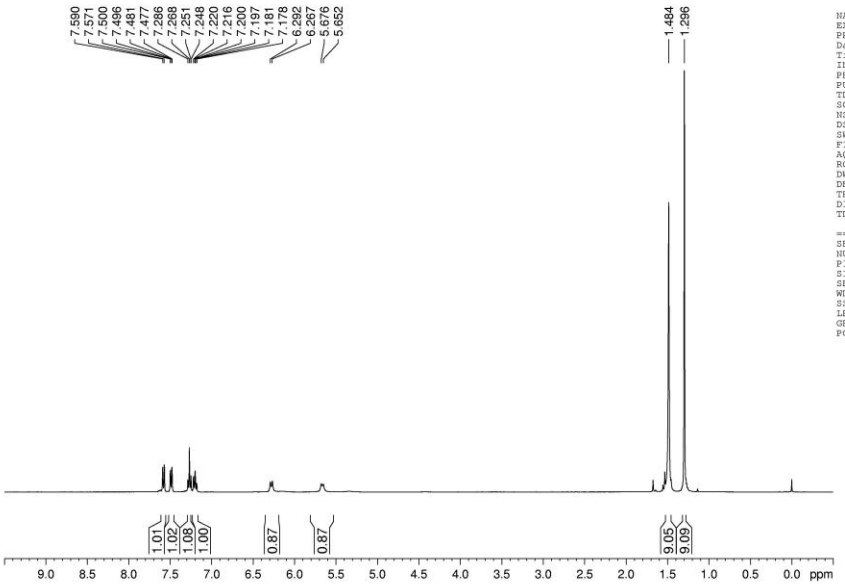

```
NAME            new400M
EXPNO               184
PROCNO                1
Date_          20201219
Time              23.01 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             5555.556 Hz
FIDRES          0.169542 Hz
AQ              5.8982902 sec
RG                   101
DW                90.000 usec
DE                 9.46 usec
TE                298.3 K
D1           1.00000000 sec
TD0                    1
SFO1        400.1321847 MHz
NUC1                1H
P0                 3.50 usec
P1                10.50 usec
SI                65536
SF          400.1300068 MHz
WDW                  EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```




```
NAME            new400M
EXPNO               185
PROCNO                1
Date_          20201219
Time              23.30 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   492
DS                     0
SWH            25000.000 Hz
FIDRES          0.762939 Hz
AQ              1.3107700 sec
RG                37.3132
DW                20.000 usec
DE                 6.50 usec
TE                298.7 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
```

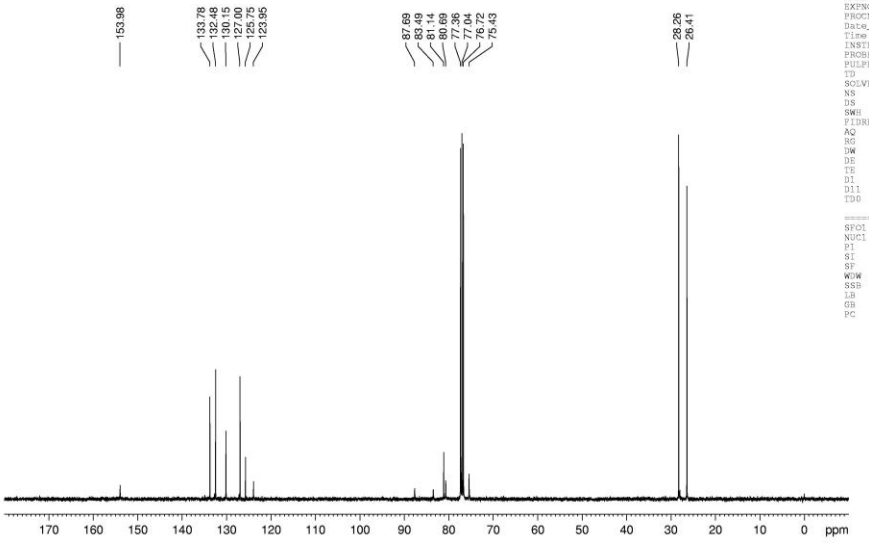


3o



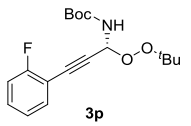

```
NAME                old400M
EXPNO                   139
PROCNO                    1
Date_              20210116
Time                  22.12
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                     31.56
DW                    62.400 usec
DE                     6.50 usec
TE                    291.4 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1               400.1522008 MHz
NUC1                      1H
P1                    10.75 usec
SI                     65536
SF                 400.1500064 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

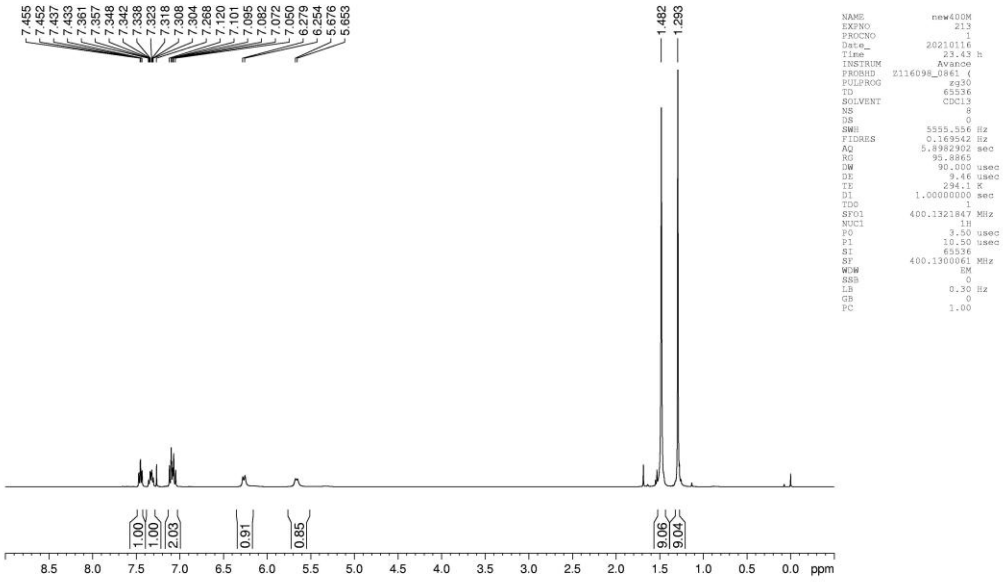



```
NAME                old400M
EXPNO                   140
PROCNO                    1
Date_              20210116
Time                  22.30
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES                0.385323 Hz
AQ                 1.2976629 sec
RG                    195.85
DW                    19.800 usec
DE                     6.50 usec
TE                    291.7 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

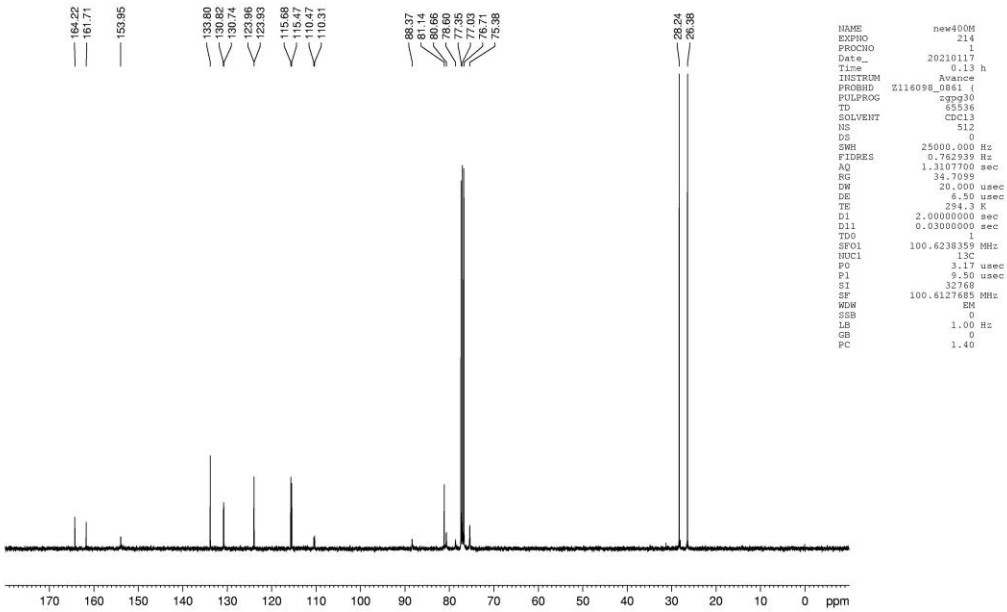
======== CHANNEL f1 ========
SFO1               100.6283629 MHz
NUC1                      13C
P1                    10.50 usec
SI                     32768
SF                 100.6177980 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                   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



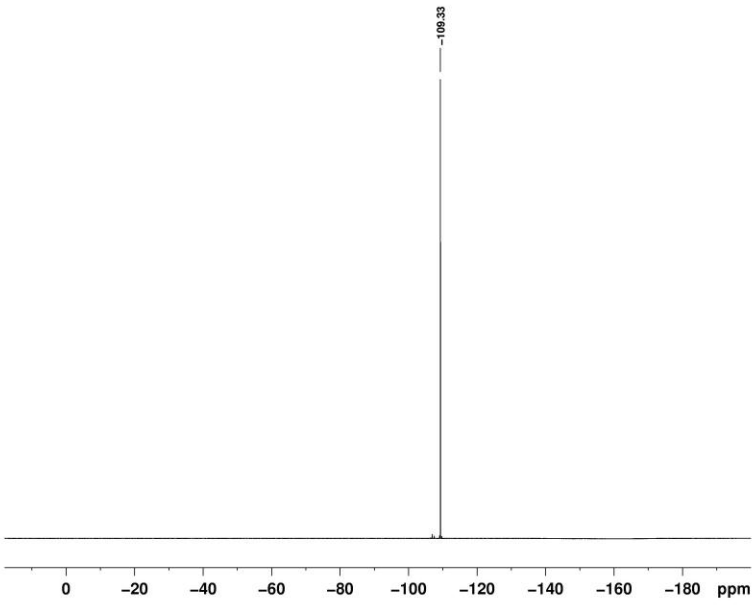
3p



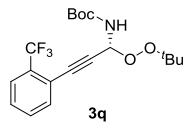

```
NAME            new400M
EXPNO               213
PROCNO                1
Date_          20210116
Time              23.43 h
INSTRUM           Avance
PROBHD    Z116098_0861 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             5555.556 Hz
FIDRES          0.169542 Hz
AQ              5.8982302 sec
RG                 95.8865
DW                90.000 usec
DE                 9.46 usec
TE                294.1 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1321847 MHz
NUC1                  1H
P0                 3.50 usec
P1                10.50 usec
SI                 65536
SF            400.1300061 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



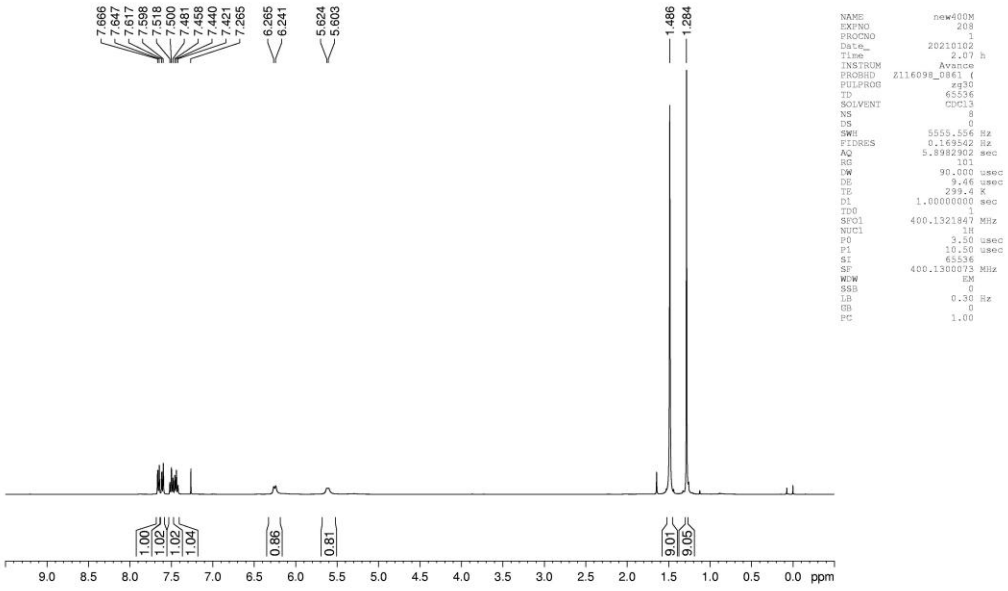

```
NAME            new400M
EXPNO               214
PROCNO                1
Date_          20210117
Time              0.13 h
INSTRUM           Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH            25000.000 Hz
FIDRES          0.762939 Hz
AQ              1.3107700 sec
RG                 34.7099
DW                20.000 usec
DE                 6.50 usec
TE                294.3 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
```



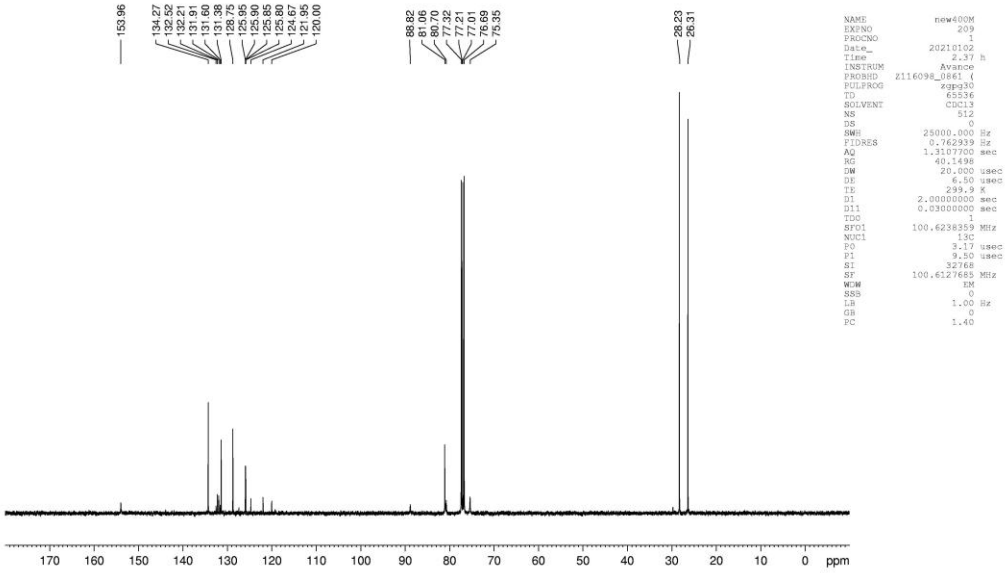
NAME	new400M
EXPNO	215
PROCNO	1
Date_	20210117
Time	0.15 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	294.4 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 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



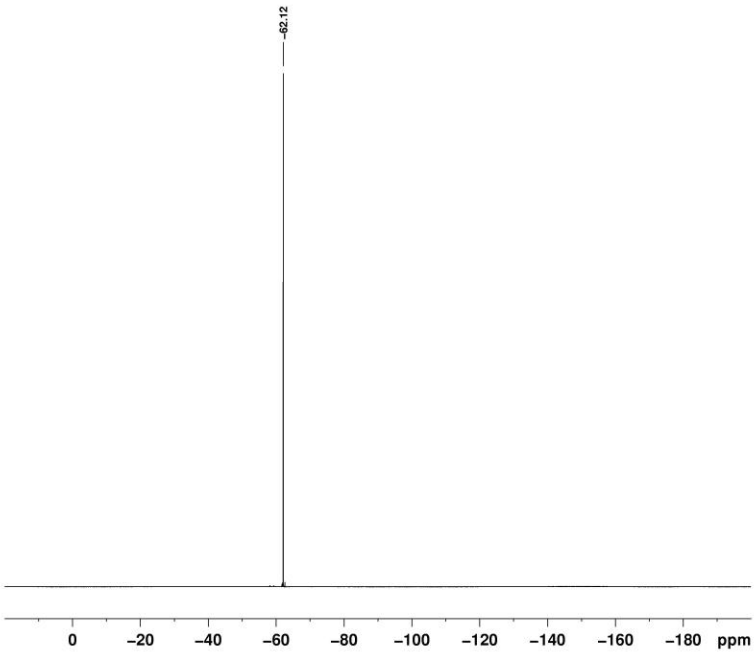
3q



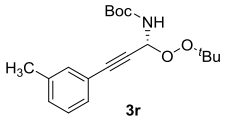

```
NAME            new400M
EXPNO               208
PROCNO                1
Date_          20210102
Time               2.07 h
INSTRUM         Avance
PROBHD     Z116098_0861 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            5555.556 Hz
FIDRES        0.169542 Hz
AQ             5.8982902 sec
RG                  101
DW               90.000 usec
DE                9.46 usec
TE               299.4 K
D1            1.00000000 sec
TD0                   1
SFO1         400.1321847 MHz
NUC1                1H
P0                3.50 usec
P1               10.50 usec
SI                65536
SF           400.1300073 MHz
WDW                 EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



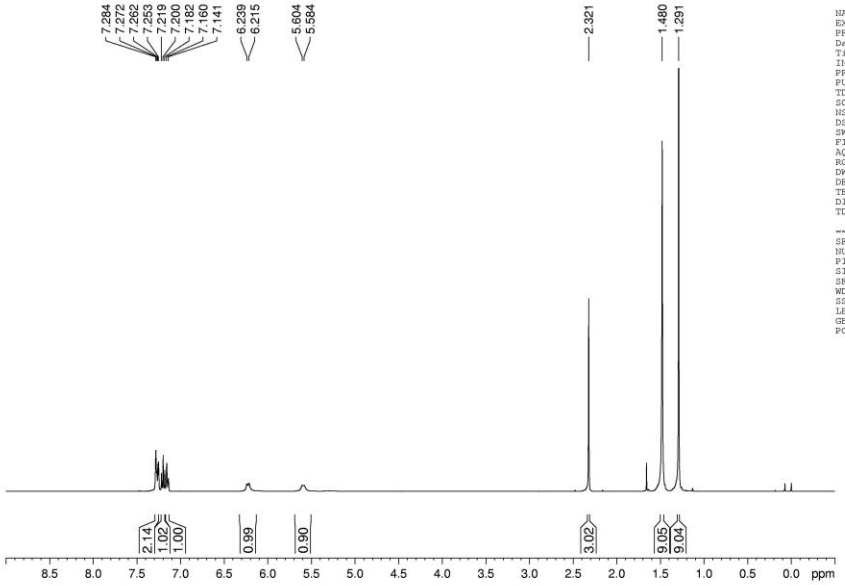

```
NAME            new400M
EXPNO               209
PROCNO                1
Date_          20210102
Time               2.37 h
INSTRUM         Avance
PROBHD     Z116098_0861 (
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    0
SWH           25000.000 Hz
FIDRES        0.762939 Hz
AQ             1.31077700 sec
RG                40.1498
DW               20.000 usec
DE                6.50 usec
TE               299.9 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	new400M
EXPNO	210
PROCNO	1
Date_	20210102
Time	2.39 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	299.7 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	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

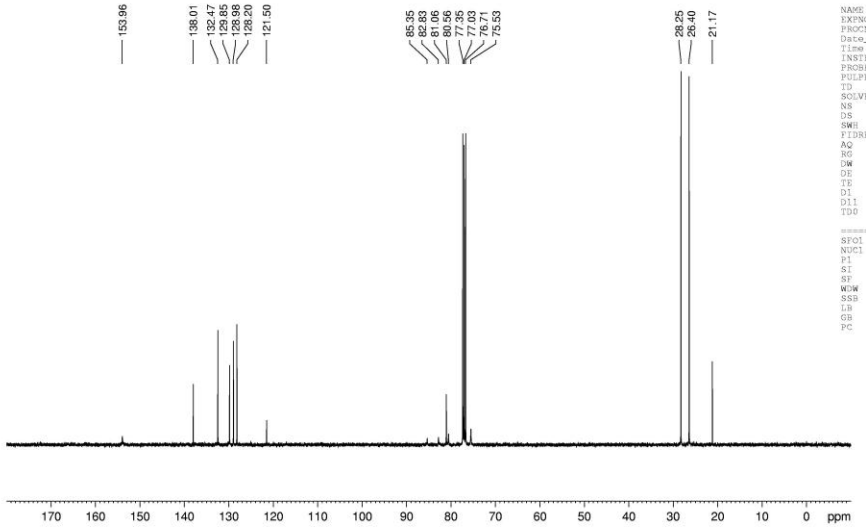


3r



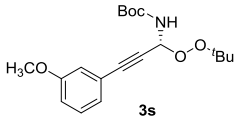

```
NAME                old400M
EXPNO                   124
PROCNO                    1
Date_              20210102
Time                   2.21
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                  295.9 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                     1H
P1                   10.75 usec
SI                    65536
SF               400.1500086 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

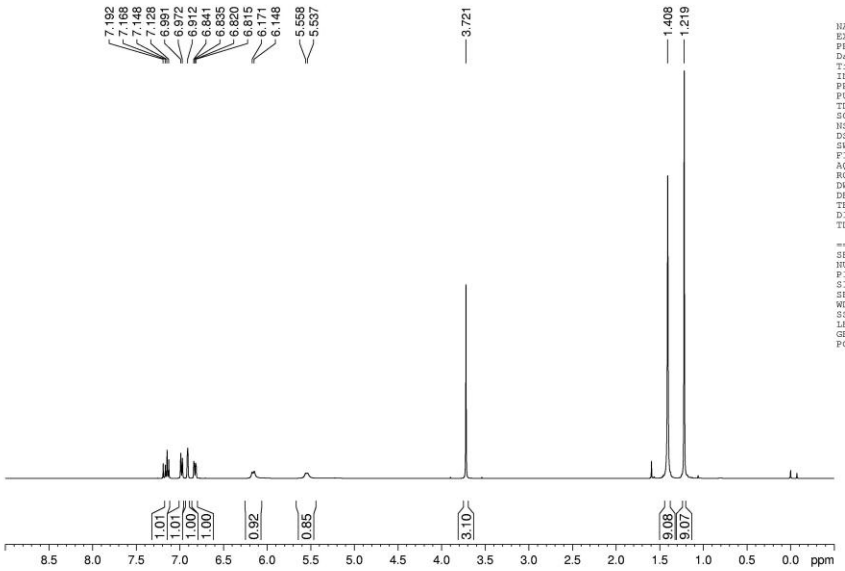



```
NAME                old400M
EXPNO                   125
PROCNO                    1
Date_              20210102
Time                   2.51
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                   195.85
DW                   19.800 usec
DE                    6.50 usec
TE                  296.3 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               100.6177980 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40
```

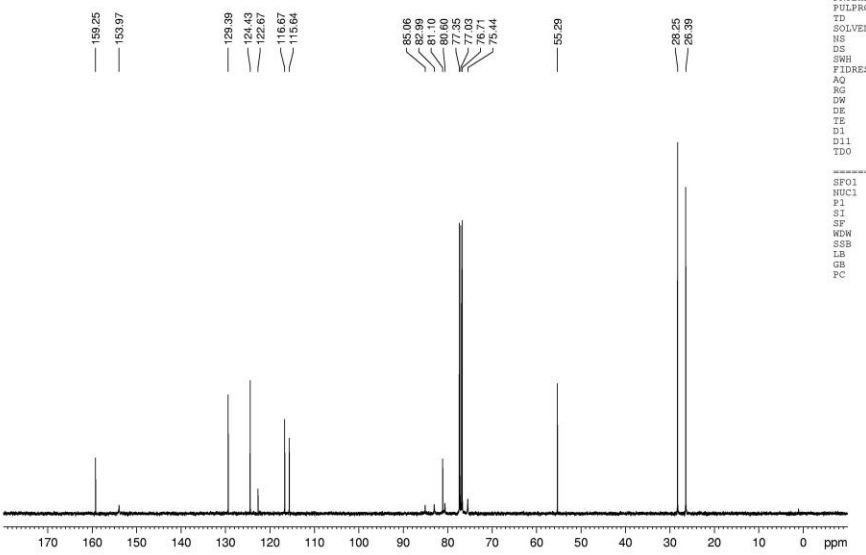


3s



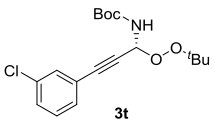

```
NAME                old400M
EXPNO                   126
PROCNO                    1
Date_              20210102
Time                   2.55
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                   296.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1522008 MHz
NUC1                      1H
P1                    10.75 usec
SI                    65536
SF              400.1500366 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

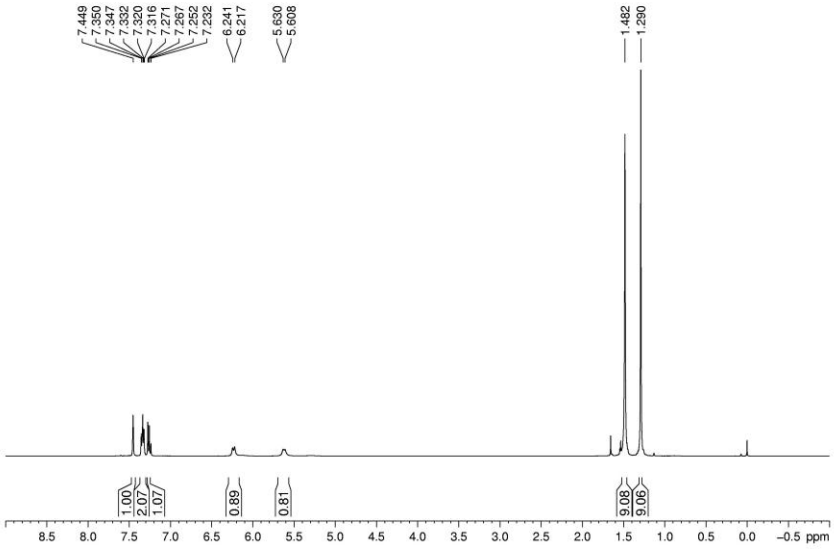



```
NAME                old400M
EXPNO                   127
PROCNO                    1
Date_              20210102
Time                   3.25
INSTRUM               spect
PROBHD    5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                    195.85
DW                   19.800 usec
DE                    6.50 usec
TE                   296.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

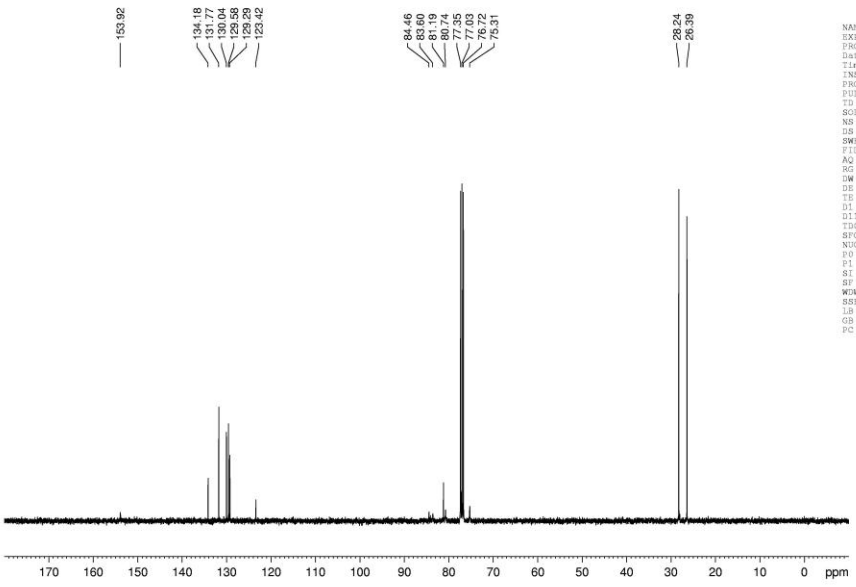
======== CHANNEL f1 ========
SFO1            100.6283629 MHz
NUC1                      13C
P1                    10.50 usec
SI                    32768
SF              100.6177980 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC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



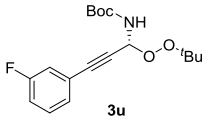
3t



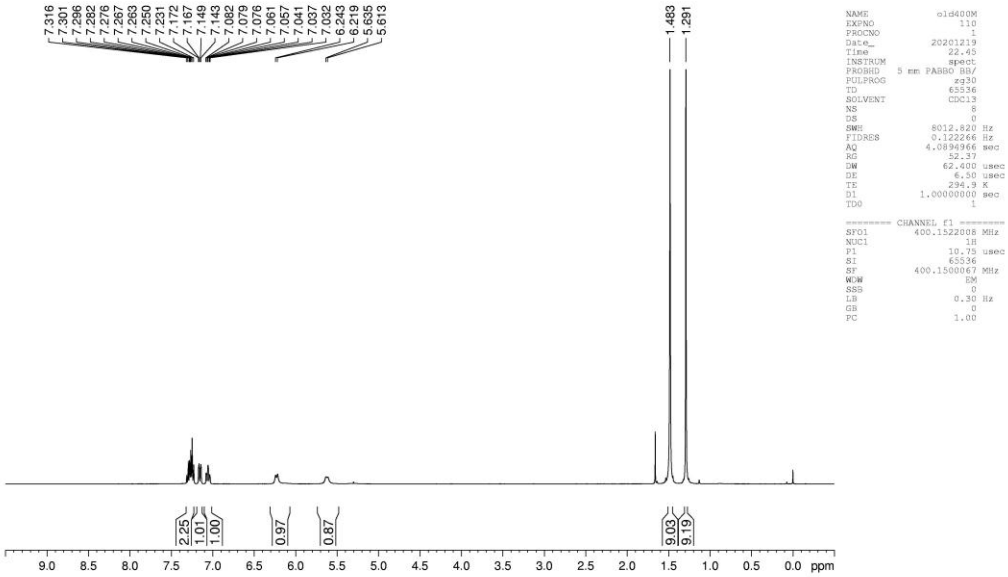

```
NAME            new400M
EXPNO               222
PROCNO                1
Date_          20210117
Time              17.17 h
INSTRUM           Avance
PROBHD   Z116098_0861 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                    8
DS                    0
SWH           5555.556 Hz
FIDRES        0.169542 Hz
AQ            5.8982902 sec
RG                  101
DW             90.000 usec
DE              9.46 usec
TE             293.6 K
D1            1.00000000 sec
TD0                   1
SFO1        400.1321847 MHz
NUC1                 1H
P0              3.50 usec
P1             10.50 usec
SI               65536
SF          400.1300065 MHz
WDW                  EM
SSB                   0
LB               0.30 Hz
GB                    0
PC                 1.00
```




```
NAME            new400M
EXPNO               223
PROCNO                1
Date_          20210117
Time              17.31 h
INSTRUM           Avance
PROBHD   Z116098_0861 (
PULPROG        zgpg30
TD               65536
SOLVENT          CDCl3
NS                  216
DS                    0
SWH          25000.000 Hz
FIDRES        0.762939 Hz
AQ            1.3107700 sec
RG                33.1673
DW             20.000 usec
DE              6.50 usec
TE             294.0 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
SI               32768
SF          100.6127685 MHz
WDW                  EM
SSB                   0
LB               1.00 Hz
GB                    0
PC                 1.40
```

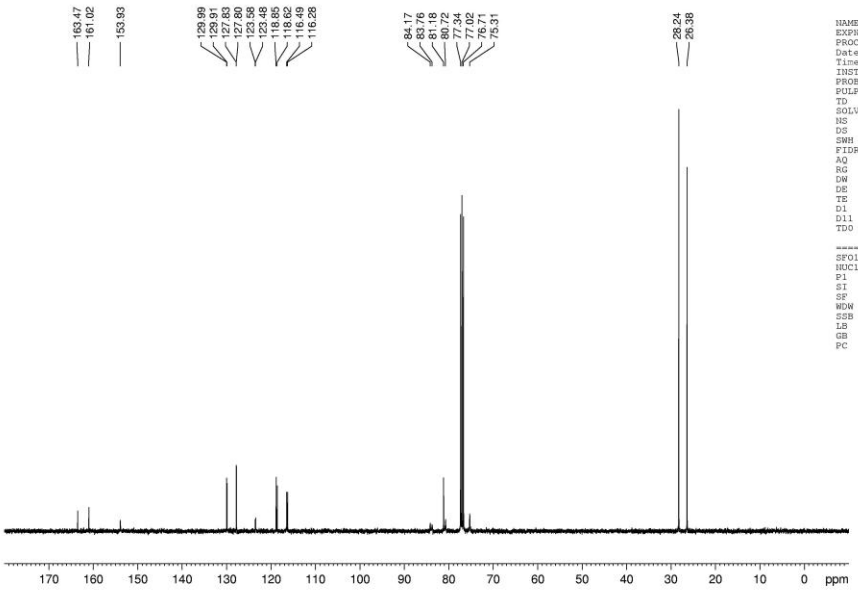


3u



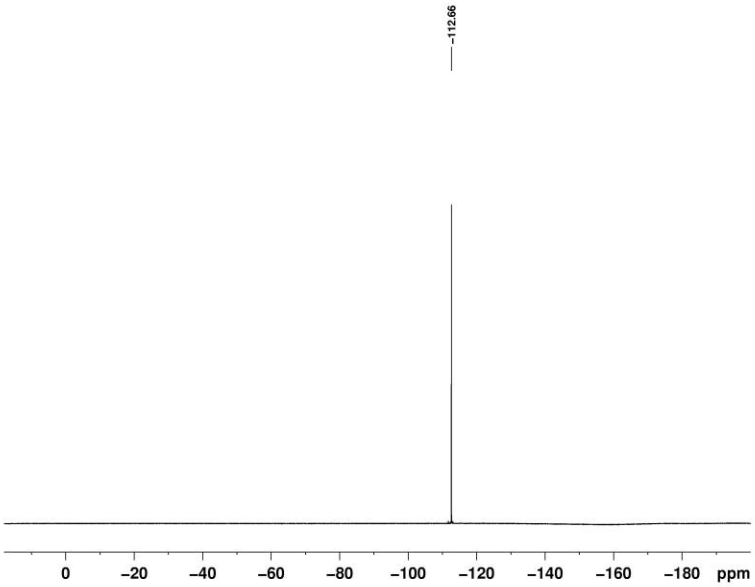

```
NAME            old400M
EXPNO               110
PROCNO                1
Date_          20201219
Time              22.45
INSTRUM           spect
PROBHD  5 mm PABBO BB/
PULPROG           zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.122266 Hz
AQ             4.0894966 sec
RG                52.37
DW               62.400 usec
DE                6.50 usec
TE               294.9 K
D1             1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
SFO1          400.1522008 MHz
NUC1                  1H
P1                10.75 usec
SI                65536
SF            400.1500067 MHz
WDW                   EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

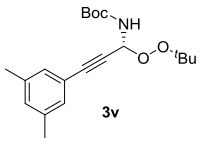



```
NAME            old400M
EXPNO               111
PROCNO                1
Date_          20201219
Time              22.50
INSTRUM           spect
PROBHD  5 mm PABBO BB/
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  351
DS                    0
SWH           25252.525 Hz
FIDRES         0.385323 Hz
AQ             1.2976629 sec
RG                195.85
DW               19.800 usec
DE                6.50 usec
TE               295.1 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                   1

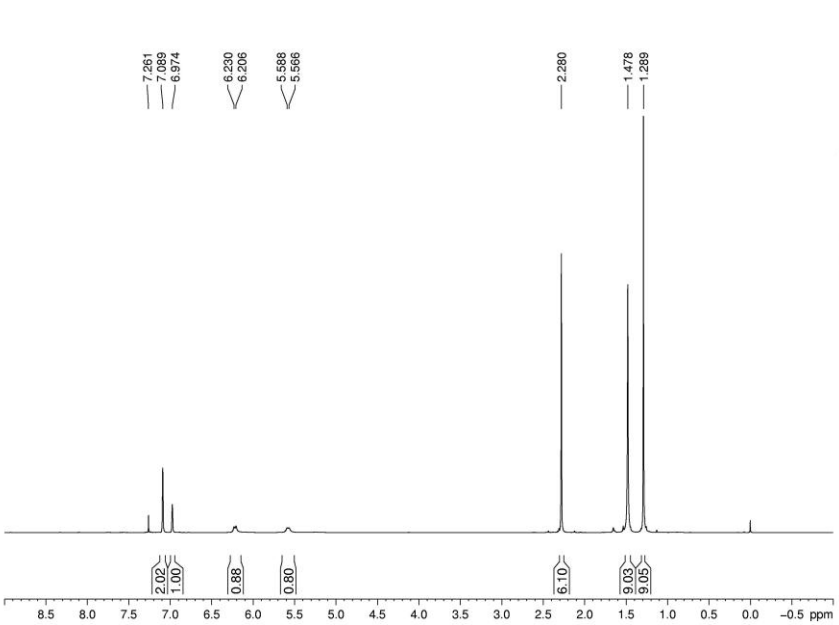
======== CHANNEL f1 ========
SFO1          100.6283629 MHz
NUC1                  13C
P1                10.50 usec
SI                32768
SF            100.6177980 MHz
WDW                   EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```



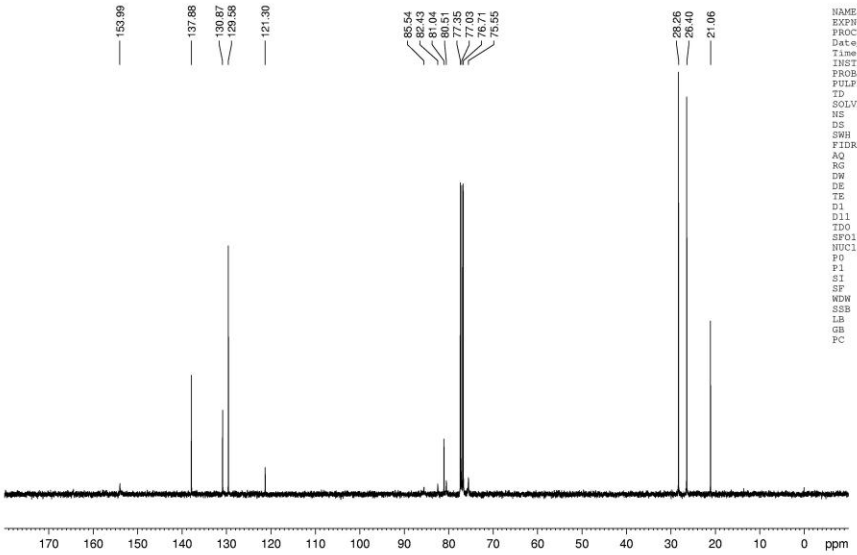
NAME	old400M
EXPNO	112
PROCNO	1
Date_	20201219
Time	23.08
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgfhigqn.2
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	89285.711 Hz
FIDRES	0.681196 Hz
AQ	0.7340532 sec
RG	195.85
DW	5.600 usec
DE	6.50 usec
TE	295.2 K
D1	1.00000000 sec
D11	0.03000000 sec
D12	0.00002000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	376.4795333 MHz
NUC1	19F
P1	14.00 usec
SI	65536
SF	376.5171850 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



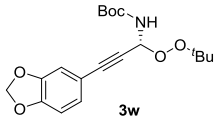
3v



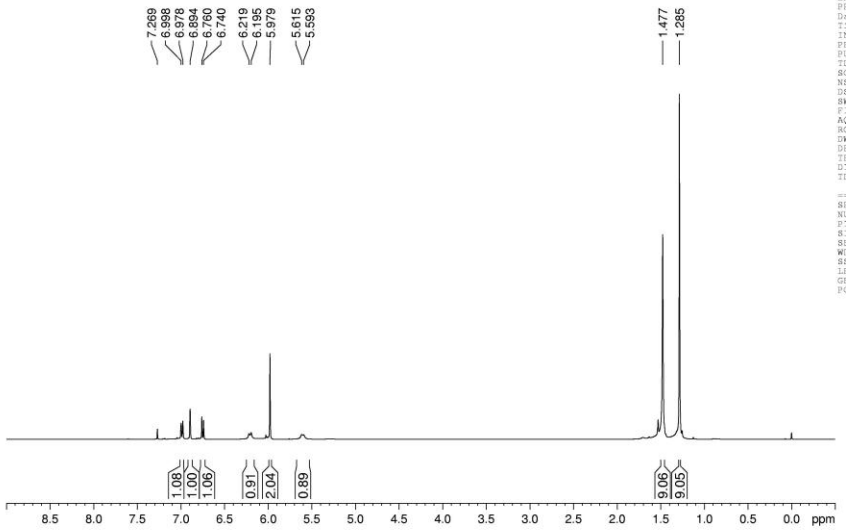
NAME	new400M
EXPNO	242
PROCNO	1
Date_	20210126
Time	22.17 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	101
DW	90.000 usec
DE	9.46 usec
TE	295.4 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.50 usec
P1	10.50 usec
SI	65536
SF	400.1300088 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	new400M
EXPNO	243
PROCNO	1
Date_	20210126
Time	22.36 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	306
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	39.0092
DW	20.000 usec
DE	6.50 usec
TE	295.6 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

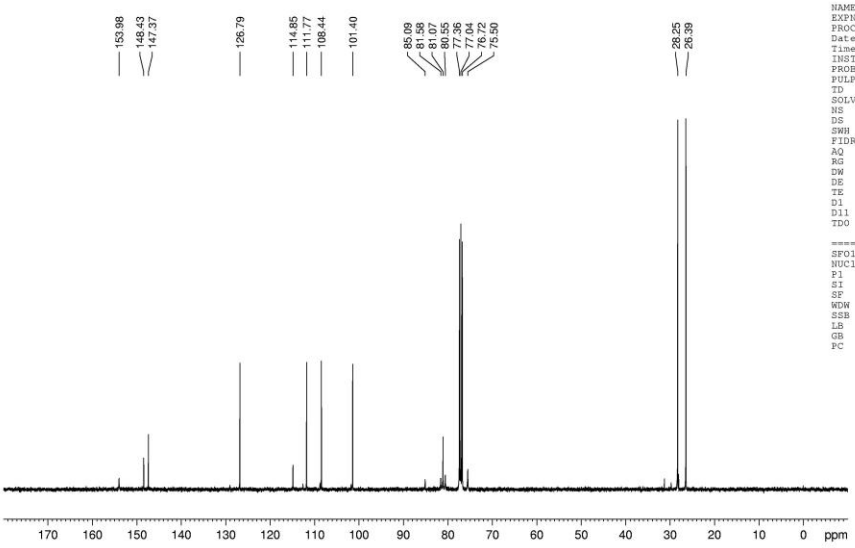


3w



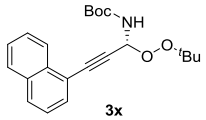

```
NAME                old400M
EXPNO                   158
PROCNO                    1
Date_              20210127
Time                  16.17
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                     65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                    31.56
DW                    62.400 usec
DE                     6.50 usec
TE                    292.7 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1322008 MHz
NUC1                      1H
P1                     10.75 usec
SI                     65536
SF               400.1300060 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

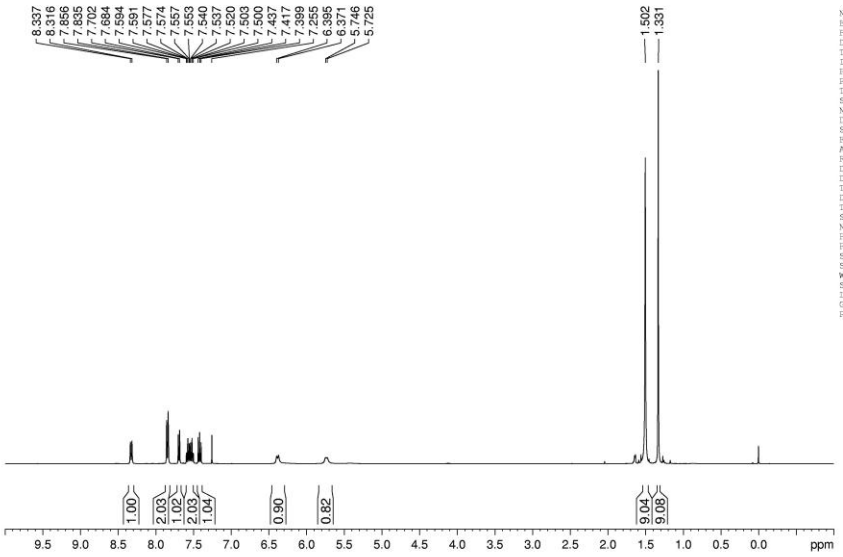



```
NAME                old400M
EXPNO                   159
PROCNO                    1
Date_              20210127
Time                  16.38
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                     65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 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

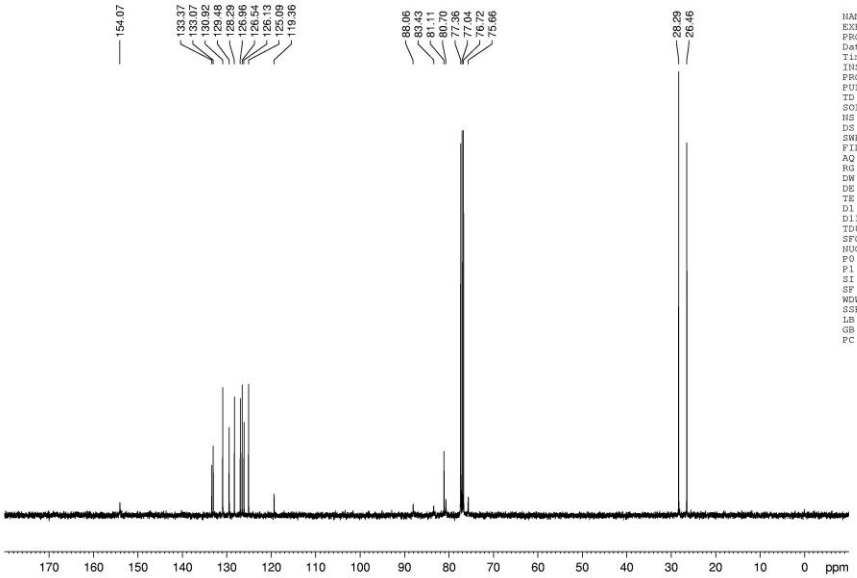
======== CHANNEL f1 ========
SFO1             100.6283629 MHz
NUC1                      13C
P1                     10.50 usec
SI                     32768
SF               100.6177980 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                       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



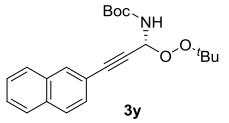
3x



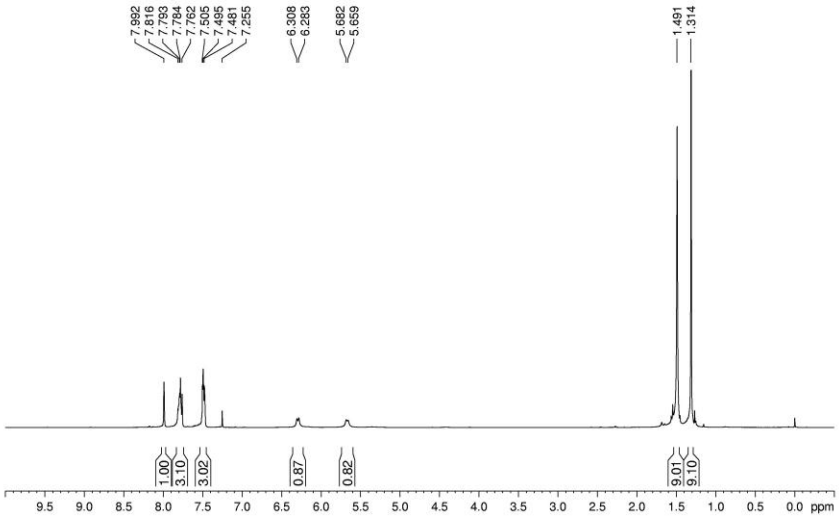

```
NAME            new400M
EXPNO               228
PROCNO                1
Date_          20210122
Time              22.47 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH           5555.556 Hz
FIDRES        0.169542 Hz
AQ            5.8982902 sec
RG                   101
DW              90.000 usec
DE               9.46 usec
TE              295.0 K
D1            1.00000000 sec
TD0                    1
SFO1        400.1321847 MHz
NUC1                  1H
P0               3.50 usec
P1              10.50 usec
SI                 65536
SF          400.1300112 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                1.00
```




```
NAME            new400M
EXPNO               229
PROCNO                1
Date_          20210122
Time              23.04 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   271
DS                     0
SWH          25000.000 Hz
FIDRES        0.762939 Hz
AQ            1.3107700 sec
RG               39.9164
DW              20.000 usec
DE               6.50 usec
TE              295.1 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
```

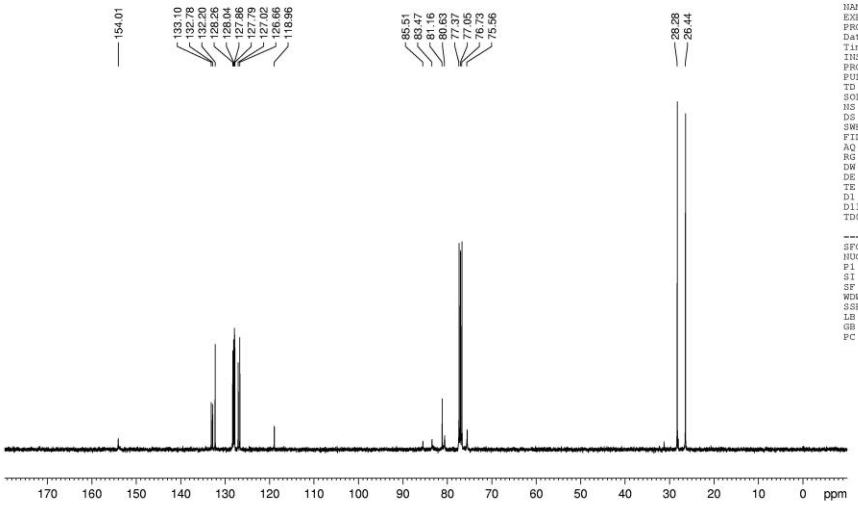


3y



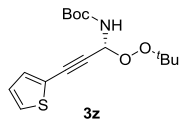

```
NAME                old400M
EXPNO                   154
PROCNO                    1
Date_              20210126
Time                 18.41
INSTRUM              spect
PROBHD     5 mm PABBO BB/
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.122266 Hz
AQ               4.0894966 sec
RG                   31.56
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
SI                  65536
SF              400.1500121 MHz
WDW                     EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

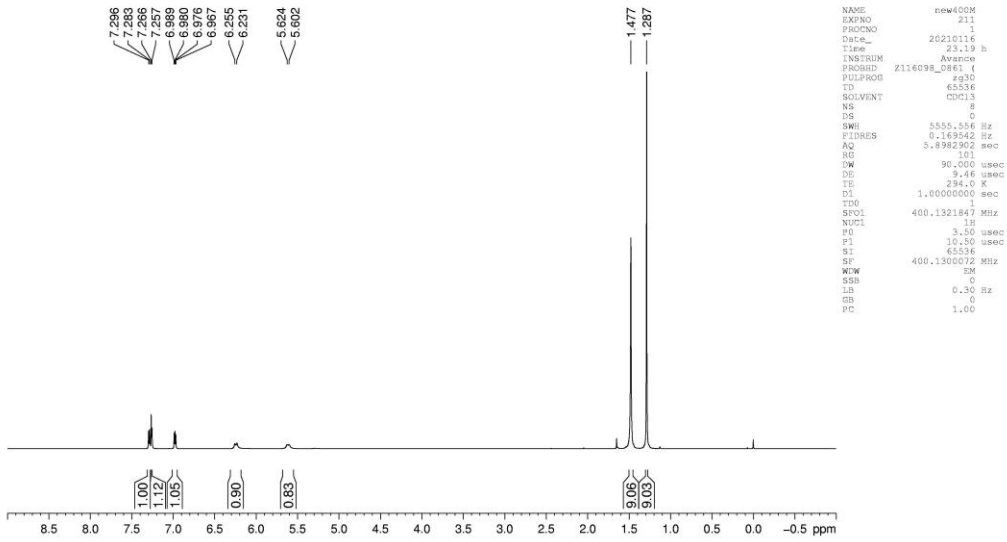



```
NAME                old400M
EXPNO                   155
PROCNO                    1
Date_              20210126
Time                 18.48
INSTRUM              spect
PROBHD     5 mm PABBO BB/
PULPROG             zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    335
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                 293.1 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1

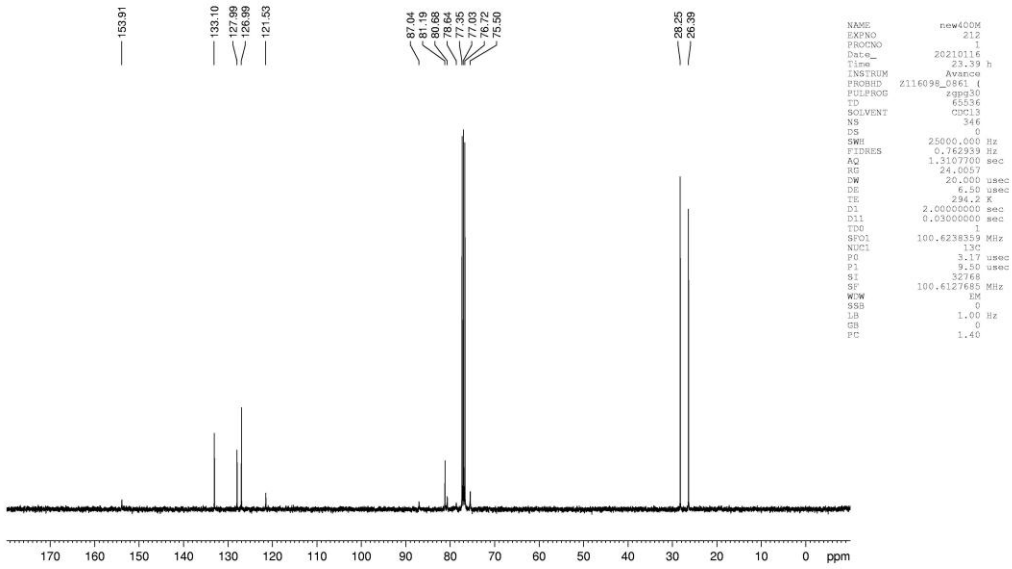
-------- CHANNEL f1 --------
SFO1            100.6283629 MHz
NUC1                    13C
P1                  10.50 usec
SI                  32768
SF              100.6177980 MHz
WDW                     EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC            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



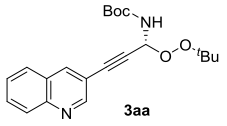
3z



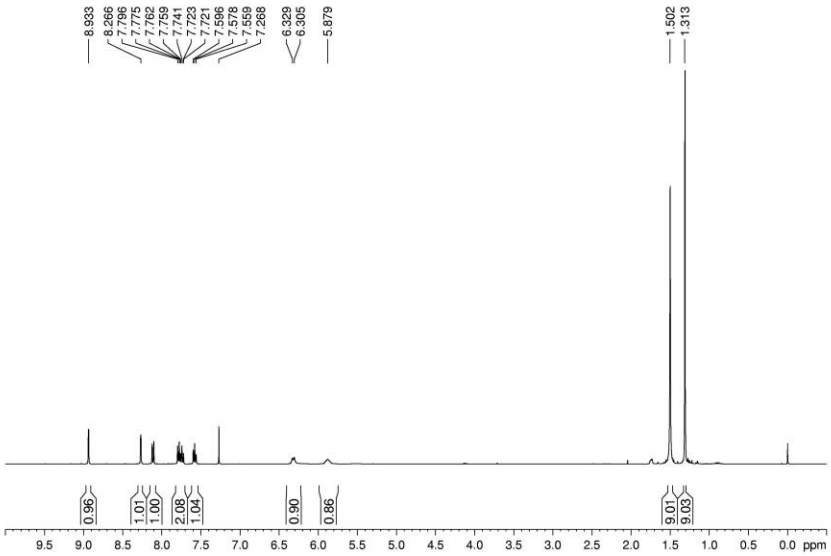

```
NAME            new400M
EXPNO               211
PROCNO                1
Date_          20210116
Time              23.19 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG           zg30
TD               65536
SOLVENT          CDCl3
NS                    8
DS                    0
SWH            5555.556 Hz
FIDRES        0.169542 Hz
AQ            5.8982902 sec
RG                  101
DW               90.000 usec
DE                9.46 usec
TE               294.0 K
D1            1.00000000 sec
TD0                   1
SFO1       400.1321847 MHz
NUC1                1H
P0                3.50 usec
P1               10.50 usec
SI               65536
SF         400.1300072 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```




```
NAME            new400M
EXPNO               212
PROCNO                1
Date_          20210116
Time              23.39 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG         zgpg30
TD               65536
SOLVENT          CDCl3
NS                  346
DS                    0
SWH           25000.000 Hz
FIDRES        0.762939 Hz
AQ            1.31077700 sec
RG                24.0057
DW               20.000 usec
DE                6.50 usec
TE               294.2 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
```

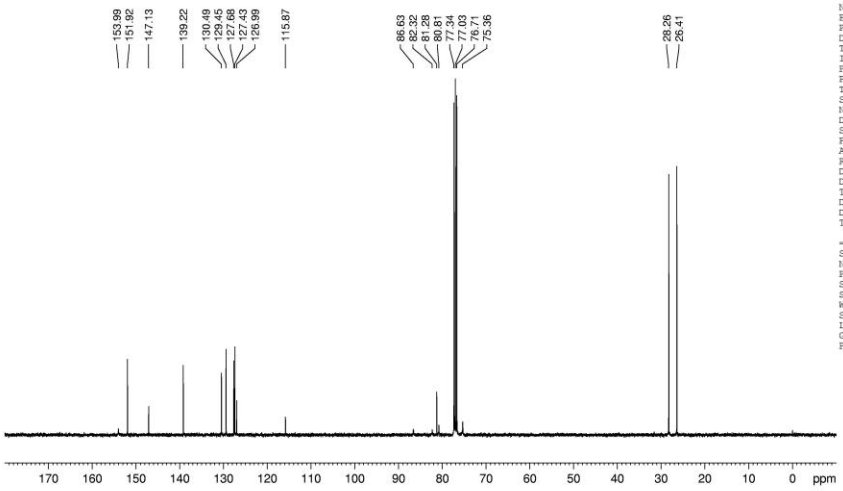


3aa



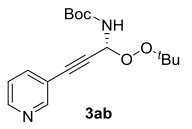

```
NAME                old400M
EXPNO                   171
PROCNO                    1
Date_              20210310
Time                   2.40
INSTRUM               spect
PROBHD  5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                    71.53
DW                   62.400 usec
DE                    6.50 usec
TE                   295.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                       1H
P1                    10.75 usec
SI                     65536
SF               400.1500064 MHz
WDW                       EM
SSB                        0
LB                    0.30 Hz
GB                         0
PC                      1.00
```

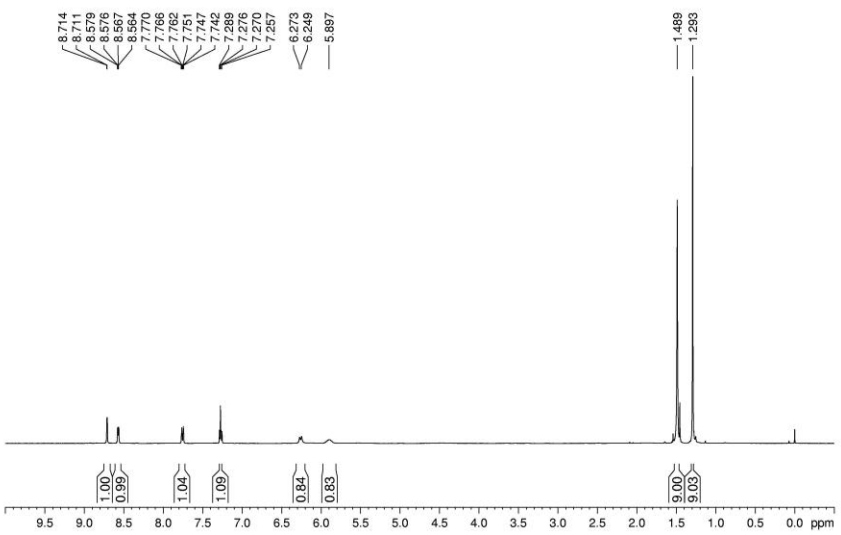



```
NAME                old400M
EXPNO                   172
PROCNO                    1
Date_              20210310
Time                   3.26
INSTRUM               spect
PROBHD  5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     800
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                   295.3 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               100.6177980 MHz
WDW                       EM
SSB                        0
LB                    1.00 Hz
GB                         0
PC                      1.40
```

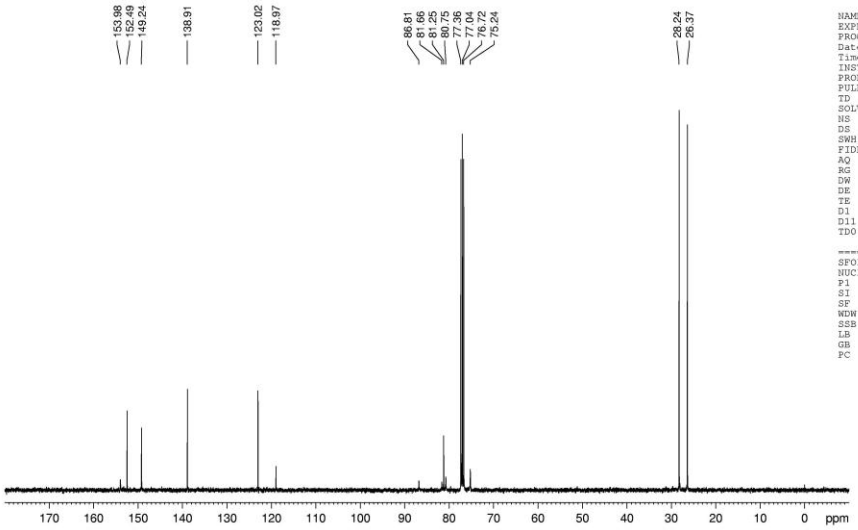


3ab



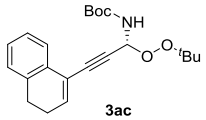

```
NAME                old400M
EXPNO                   143
PROCNO                    1
Date_              20210123
Time                   0.51
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                     55.34
DW                    62.400 usec
DE                     6.50 usec
TE                    292.6 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1               400.1522008 MHz
NUC1                       1H
P1                        10.75 usec
SI                        65536
SF                 400.1500031 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

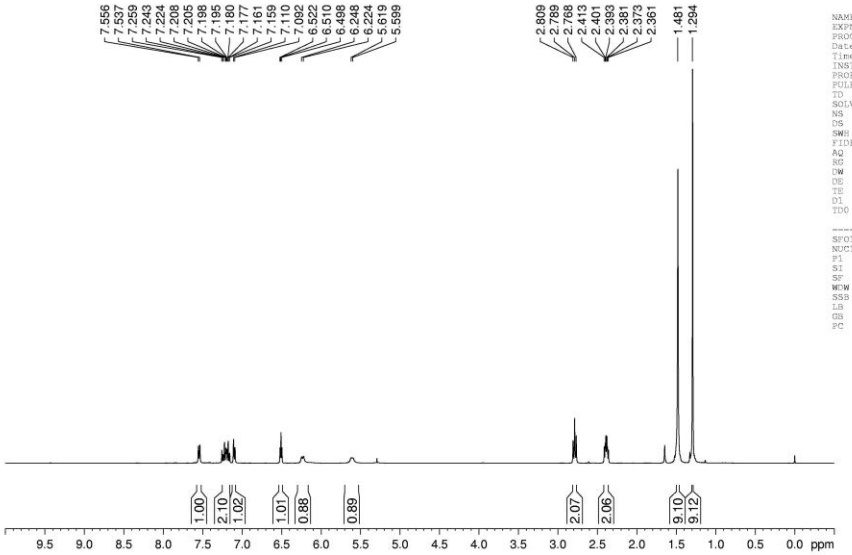



```
NAME                old400M
EXPNO                   144
PROCNO                    1
Date_              20210123
Time                   1.21
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES             0.385323 Hz
AQ                 1.2976629 sec
RG                    195.85
DW                   19.800 usec
DE                     6.50 usec
TE                    292.9 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                 100.6177980 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB                         0
PC                      1.40
```

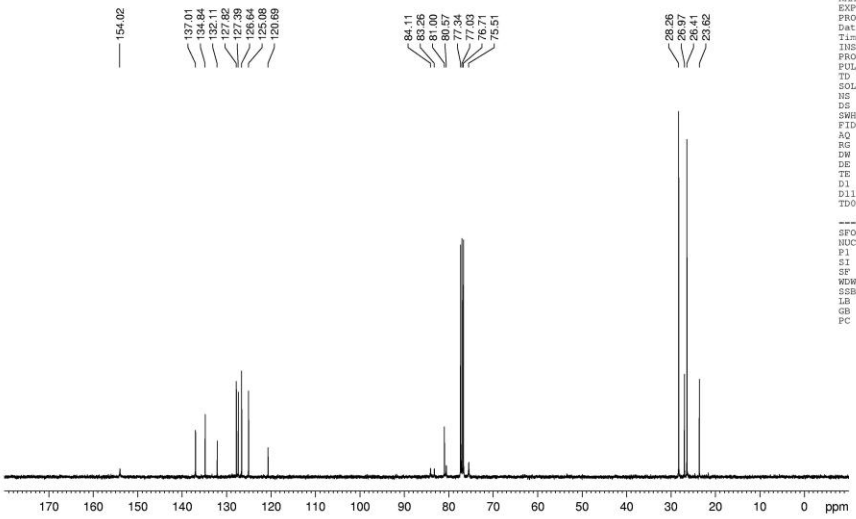


3ac



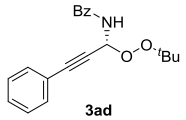

```
NAME                 old400M
EXPNO                    208
PROCNO                     1
Date_             20210408
Time                   22.52
INSTRUM                spect
PROBHD     5 mm PABBO BB/
PULPROG             zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                        31.56
DW                    62.400 usec
DE                     6.50 usec
TE                    295.6 K
D1                 1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
SFO1               400.1522008 MHz
NUC1                       1H
P1                        10.75 usec
SI                     65536
SF                 400.1500102 MHz
WDW                       EM
SSB                        0
LB                      0.30 Hz
GB                         0
PC                      1.00
```

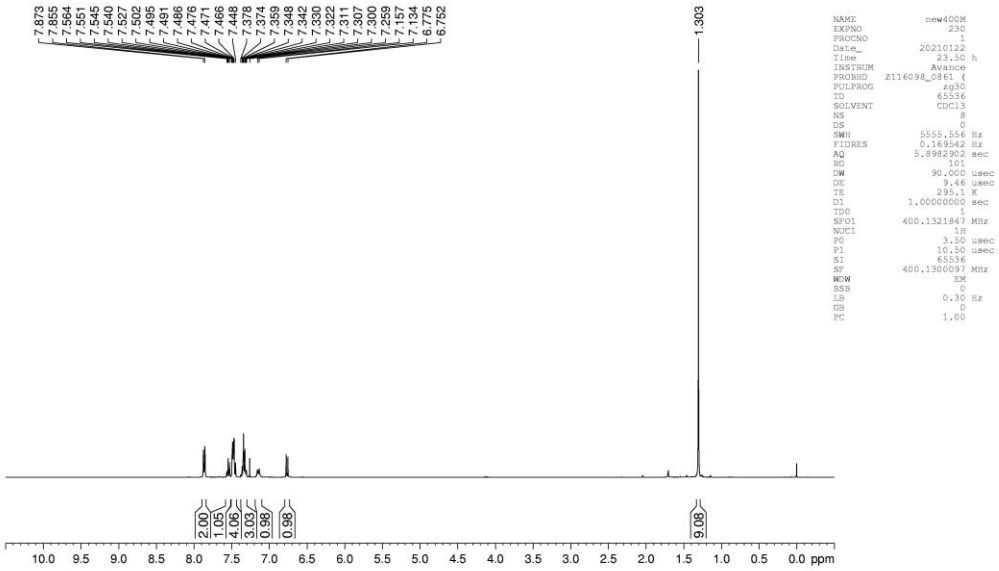



```
NAME                 old400M
EXPNO                    209
PROCNO                     1
Date_             20210408
Time                   22.55
INSTRUM                spect
PROBHD     5 mm PABBO BB/
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       700
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                    295.6 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                        1

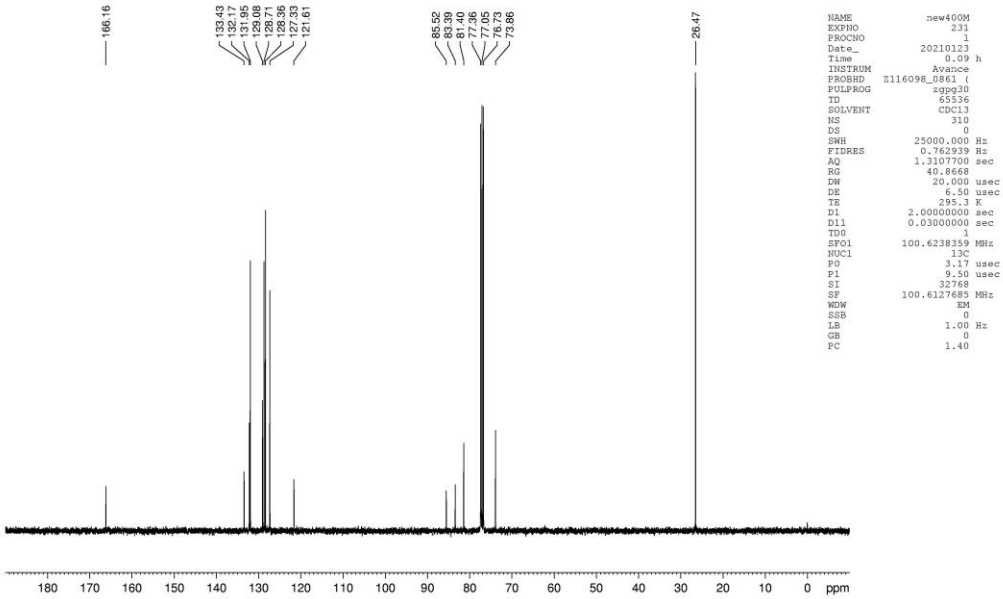
======== CHANNEL f1 ========
SFO1               100.6283629 MHz
NUC1                       13C
P1                        10.50 usec
SI                     32768
SF                 100.6177980 MHz
WDW                       EM
SSB                        0
LB                      1.00 Hz
GB            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



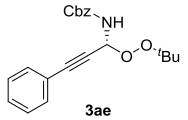
3ad



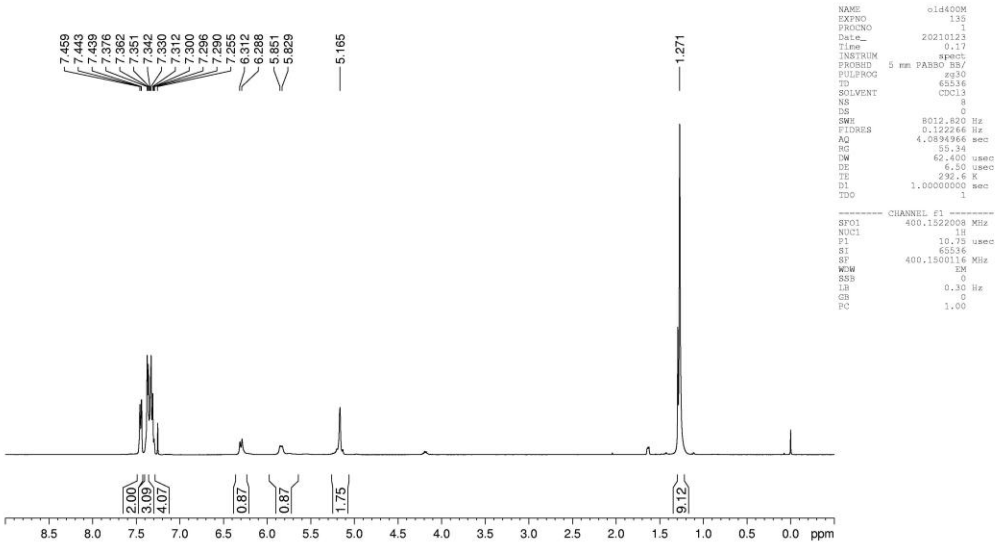
NAME	new400M
EXPNO	230
PROCNO	1
Date_	20210122
Time	23.50 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	101
DW	90.000 usec
DE	9.46 usec
TE	295.1 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.50 usec
P1	10.50 usec
SI	65536
SF	400.1300097 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

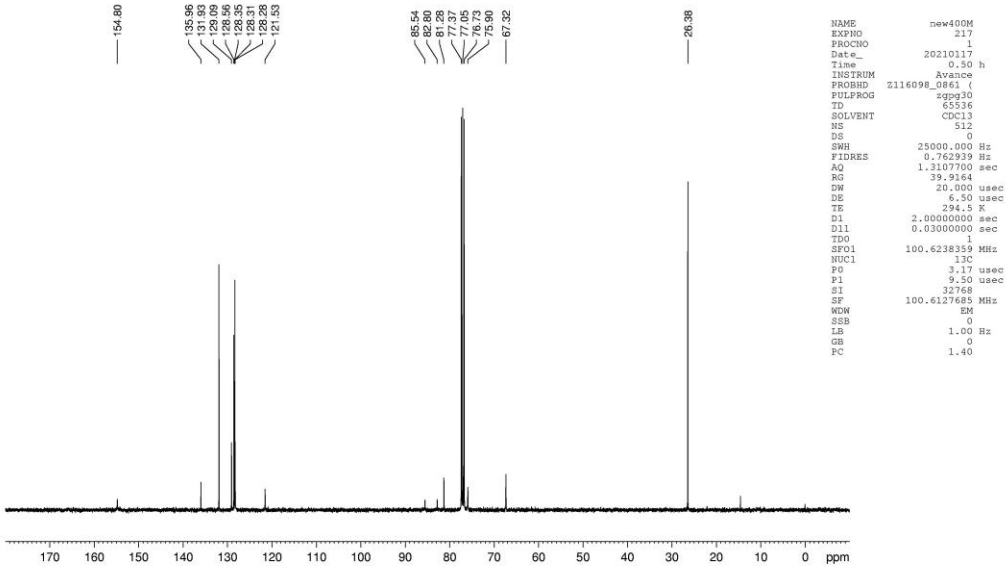


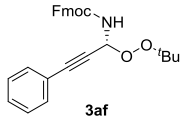
NAME	new400M
EXPNO	231
PROCNO	1
Date_	20210123
Time	0.09 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	310
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	40.8668
DW	20.000 usec
DE	6.50 usec
TE	295.3 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238359 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


Cbz
NH
O
O
tBu

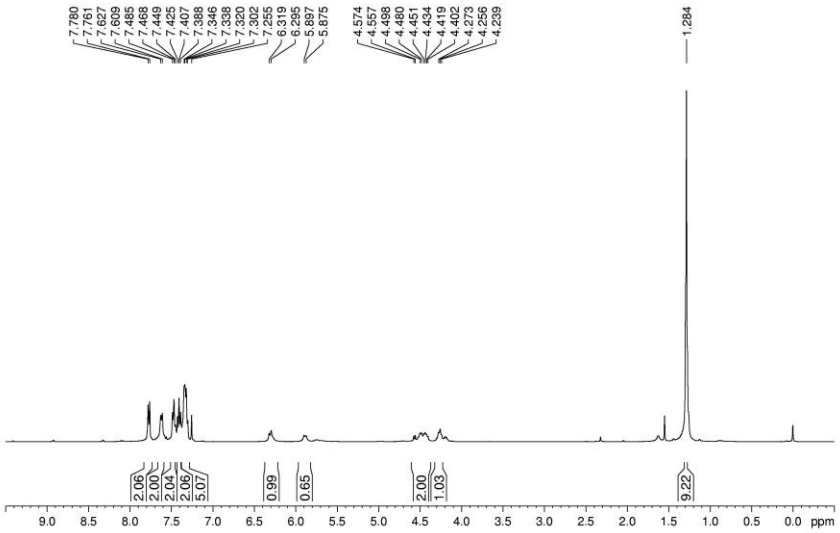
3ae


7.459
7.443
7.439
7.376
7.362
7.351
7.342
7.330
7.312
7.300
7.296
7.290
7.255
6.312
6.288
5.851
5.829
5.165
1.271
2.00
3.09
4.07
0.87
0.87
1.75
9.12
8.5 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
NAME old400M
EXPNO 135
PROCNO 1
Date_ 20210123
Time 0.17
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 sec
RG 55.34
DW 62.400 usec
DE 6.50 usec
TE 292.6 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
SFO1 400.1522008 MHz
NUC1 1H
P1 10.75 usec
SI 65536
SF 400.1500116 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

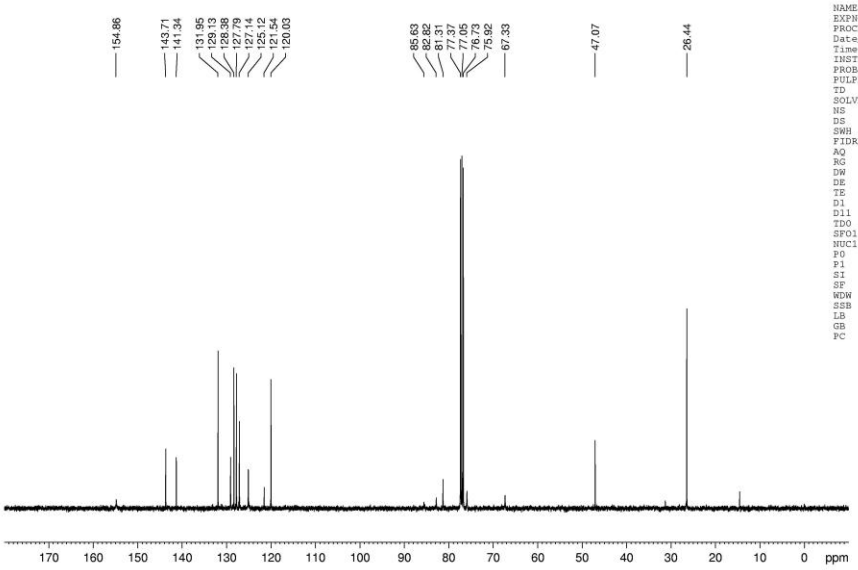

154.80
135.96
131.93
129.09
128.56
128.35
128.31
128.28
121.53
85.54
82.80
81.28
77.37
77.05
76.73
75.90
67.32
26.38
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
NAME new400M
EXPNO 217
PROCNO 1
Date_ 20210117
Time 0.50 h
INSTRUM Avance
PROBHD Z116098_0861 (
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 512
DS 0
SWH 25000.000 Hz
FIDRES 0.762939 Hz
AQ 1.3107700 sec
RG 39.9164
DW 20.000 usec
DE 6.50 usec
TE 294.5 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



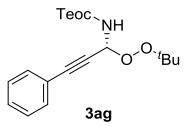
3af



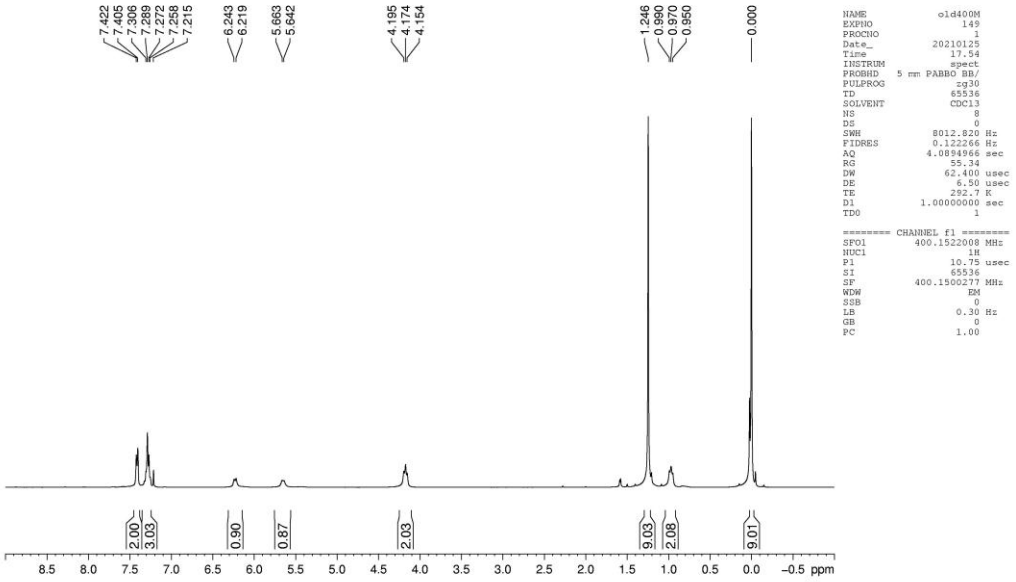
NAME	old400M
EXPNO	147
PROCNO	1
Date_	20210125
Time	17.33
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	63.8
DW	62.400 usec
DE	6.50 usec
TE	292.7 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1522008 MHz
NUC1	1H
P1	10.75 usec
SI	65536
SF	400.1500117 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

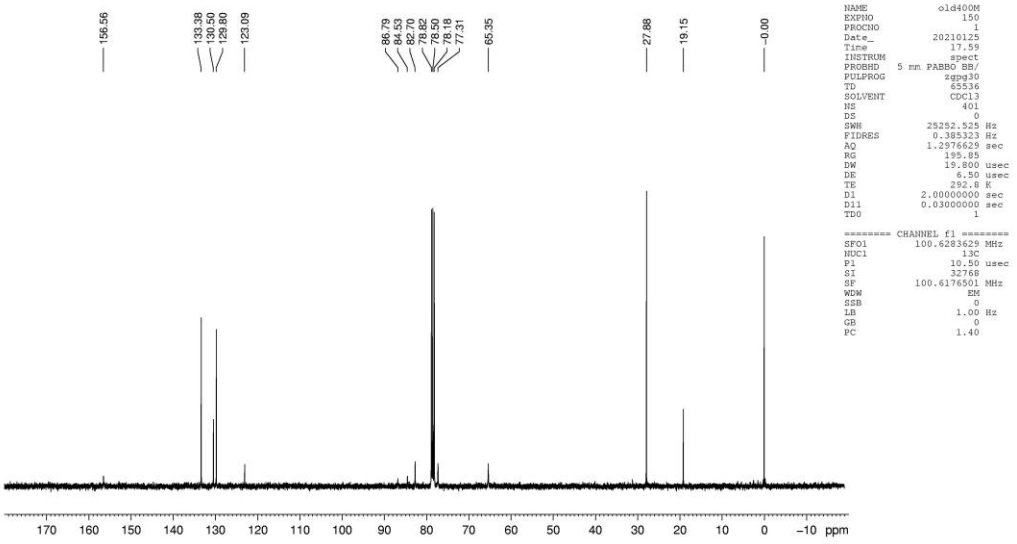


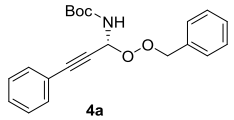
NAME	new400M
EXPNO	227
PROCNO	1
Date_	20210122
Time	22.43 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	411
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	38.0578
DW	20.000 usec
DE	6.50 usec
TE	295.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238359 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


Teoc
NH
O
O
tBu

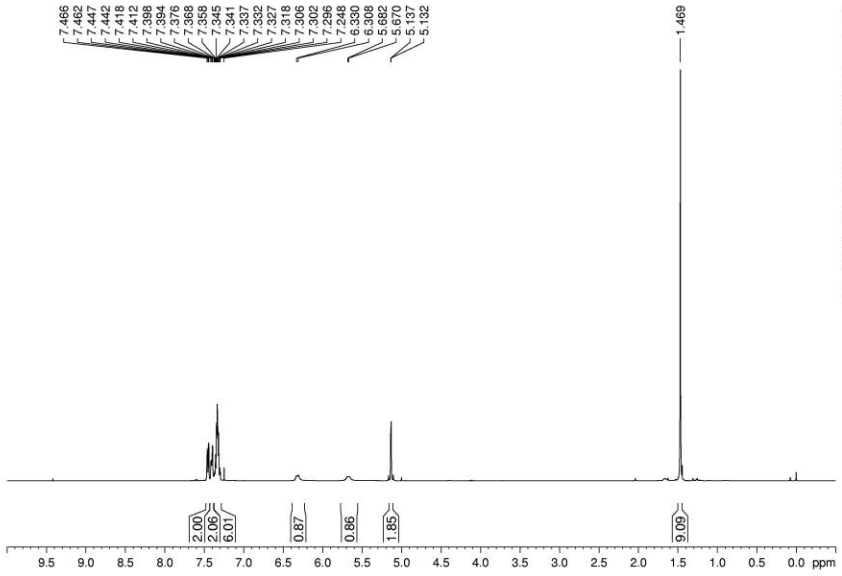
3ag


7.422
7.405
7.306
7.289
7.272
7.258
7.215
6.243
6.219
5.663
5.642
4.195
4.174
4.154
1.246
0.990
0.970
0.950
0.000
2.00
3.03
0.90
0.87
2.03
9.03
2.08
9.01
8.5 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 −0.5 ppm
NAME old400M
EXPNO 149
PROCNO 1
Date_ 20210125
Time 17.54
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 sec
RG 55.34
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
SI 65536
SF 400.1500277 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

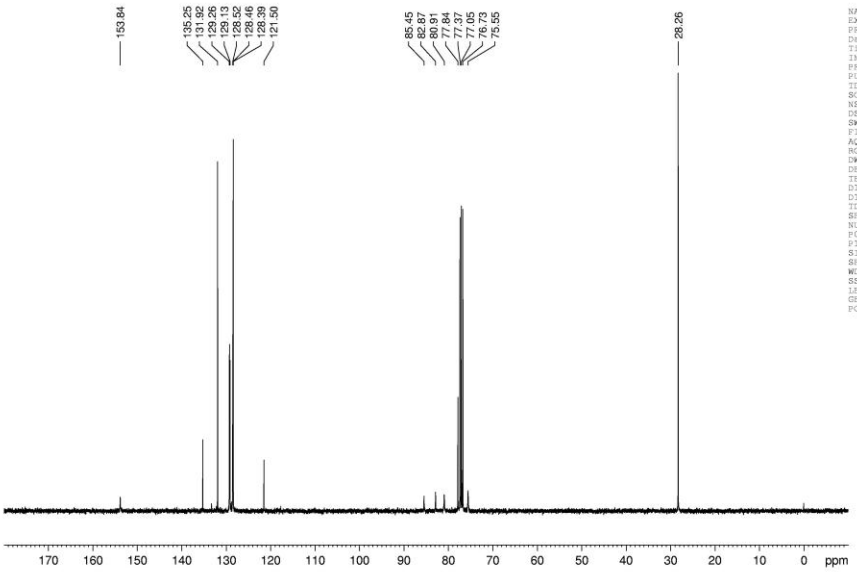

156.56
133.38
130.50
129.60
123.09
86.79
84.53
82.70
78.82
78.50
78.18
77.31
65.35
27.88
19.15
0.00
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 −10 ppm
NAME old400M
EXPNO 150
PROCNO 1
Date_ 20210125
Time 17.59
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 401
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 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
SI 32768
SF 100.6176501 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



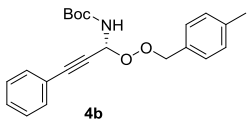
4a



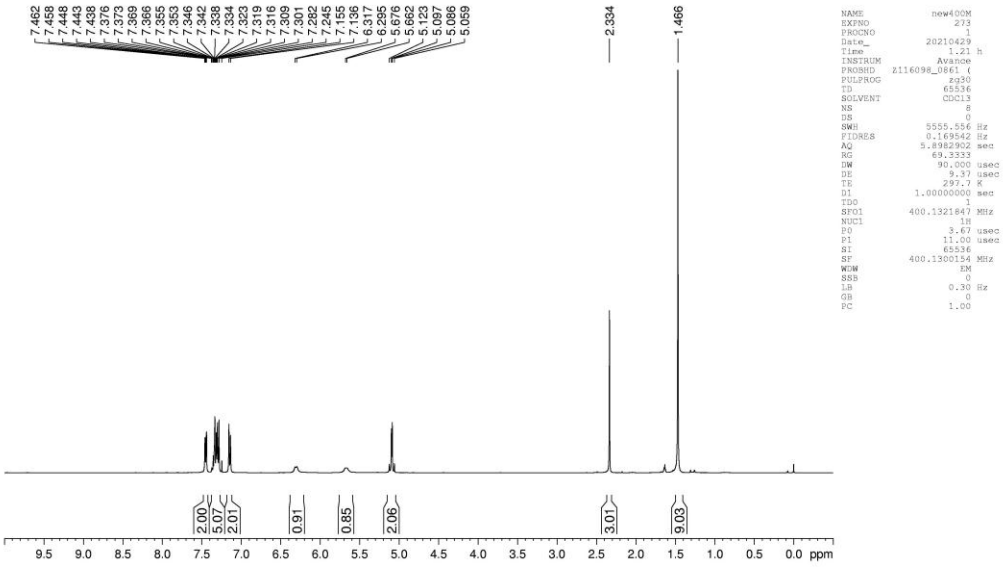

```
NAME            new400M
EXPNO               271
PROCNO                1
Date_          20210429
Time               0.47 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            5555.556 Hz
FIDRES         0.169942 Hz
AQ             5.8982902 sec
RG               93.8889
DW               90.000 usec
DE                9.37 usec
TE                297.6 K
D1            1.00000000 sec
TD0                   1
SFO1       400.1321847 MHz
NUC1                  1H
P0                3.67 usec
P1               11.00 usec
SI                65536
SF         400.1300141 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



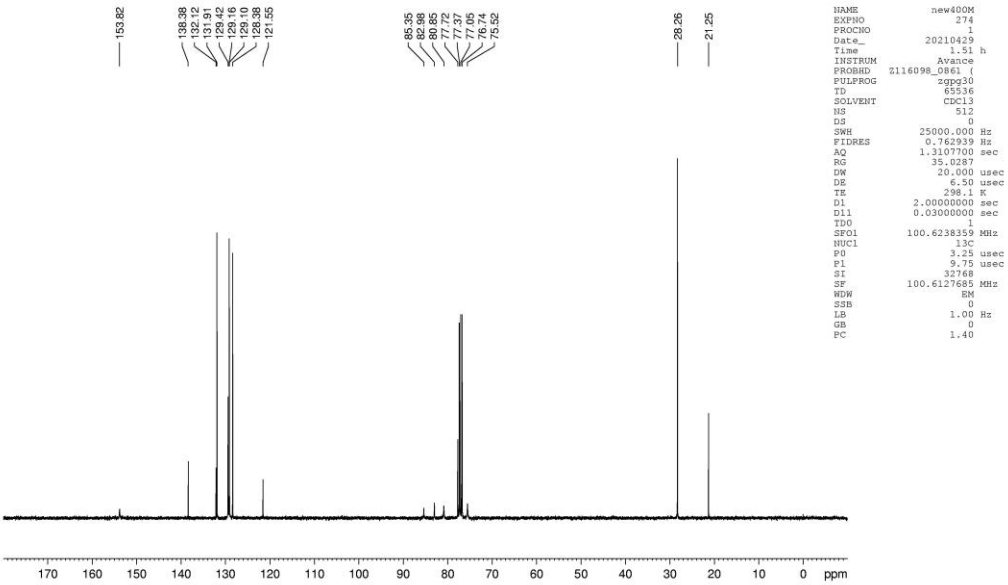

```
NAME            new400M
EXPNO               272
PROCNO                1
Date_          20210429
Time               1.17 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                  512
DS                    0
SWH           25000.000 Hz
FIDRES         0.762939 Hz
AQ             1.3107700 sec
RG               35.9269
DW               20.000 usec
DE                6.50 usec
TE                297.9 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1       100.6238359 MHz
NUC1                 13C
P0                3.25 usec
P1                9.75 usec
SI                32768
SF         100.6127685 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



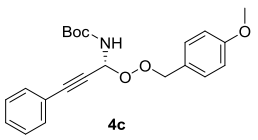
4b



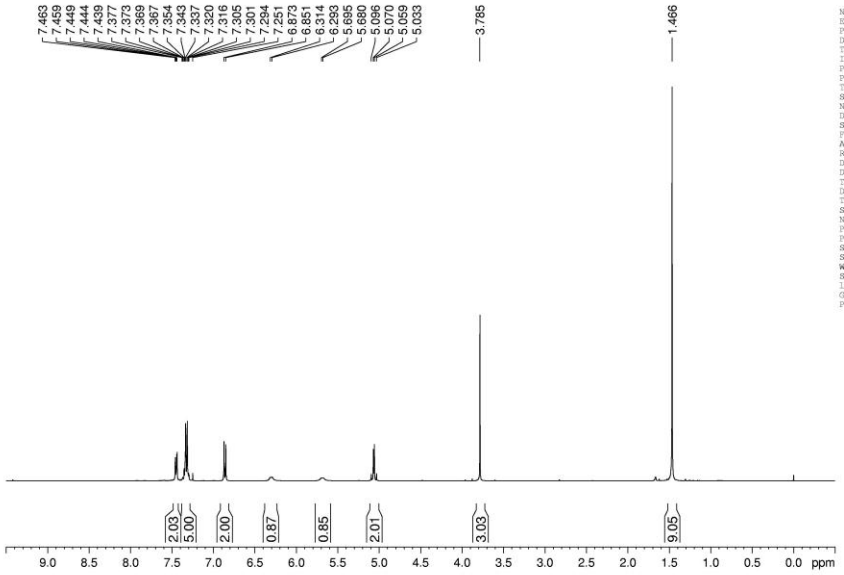

```
NAME            new400M
EXPNO               273
PROCNO                1
Date_          20210429
Time               1.21 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG           zg30
TD               65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            5555.556 Hz
FIDRES        0.169542 Hz
AQ            5.8982902 sec
RG               69.3333
DW              90.000 usec
DE               9.37 usec
TE              297.7 K
D1            1.00000000 sec
TD0                   1
SFO1       400.1321847 MHz
NUC1                1H
P0                3.67 usec
P1               11.00 usec
SI               65536
SF         400.1300154 MHz
WDW                 EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



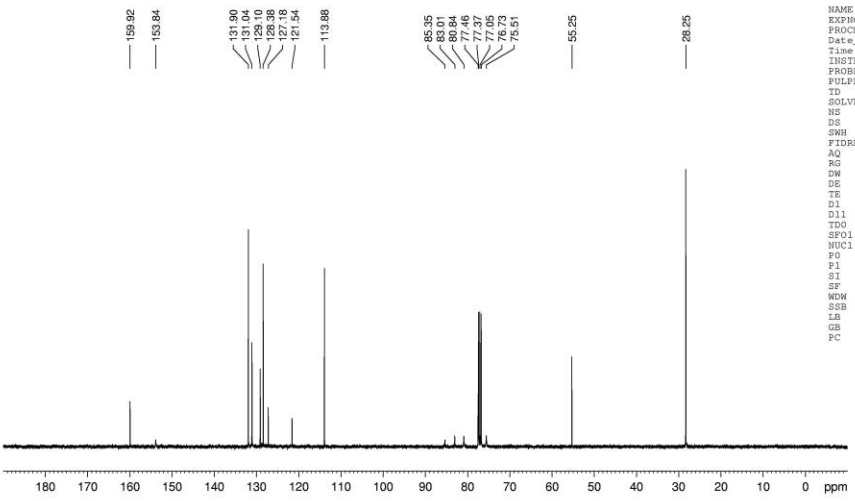

```
NAME            new400M
EXPNO               274
PROCNO                1
Date_          20210429
Time               1.51 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG         zgpg30
TD               65536
SOLVENT           CDCl3
NS                  512
DS                    0
SWH           25000.000 Hz
FIDRES        0.762939 Hz
AQ            1.3107700 sec
RG               35.0287
DW              20.000 usec
DE               6.50 usec
TE              298.1 K
D1            2.00000000 sec
D11           0.03000000 sec
TD0                   1
SFO1       100.6238359 MHz
NUC1                13C
P0                3.25 usec
P1                9.75 usec
SI               32768
SF         100.6127685 MHz
WDW                 EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```



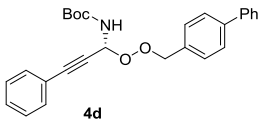
4c



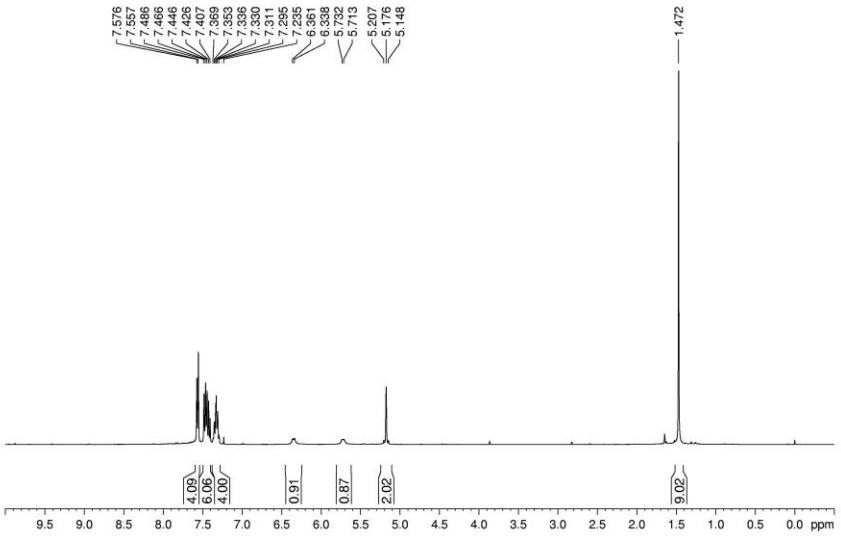
NAME	new400M
EXPNO	289
PROCNO	1
Date_	20210605
Time	0.23 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	65
DW	90.000 usec
DE	9.37 usec
TE	299.3 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.67 usec
P1	11.00 usec
SI	65536
SF	400.1300132 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	new400M
EXPNO	290
PROCNO	1
Date_	20210605
Time	0.38 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	250
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	37.4557
DW	20.000 usec
DE	6.50 usec
TE	299.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238359 MHz
NUC1	13C
P0	3.25 usec
P1	9.75 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

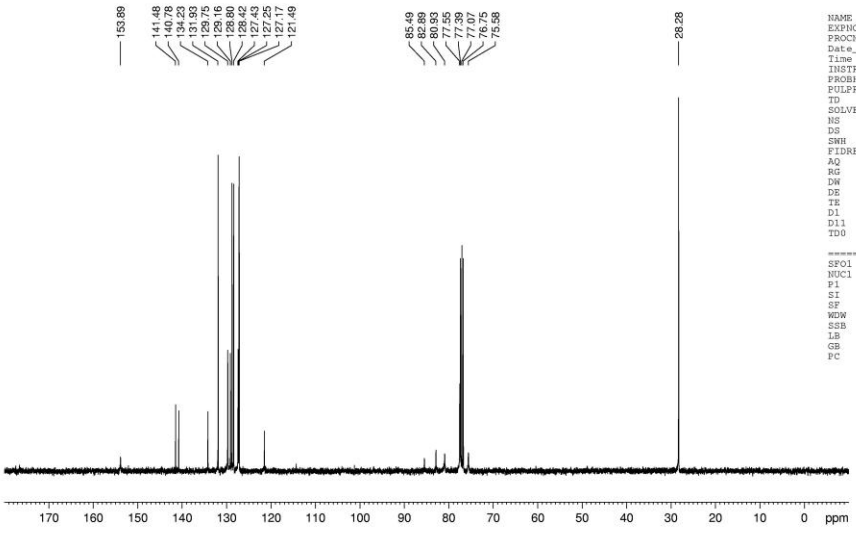


4d



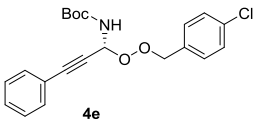

```
NAME                old400M
EXPNO                   266
PROCNO                    1
Date_              20210605
Time                   9.16
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                     65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                     31.56
DW                    62.400 usec
DE                     6.50 usec
TE                   296.0 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                     1H
P1                    10.75 usec
SI                     65536
SF               400.1500189 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

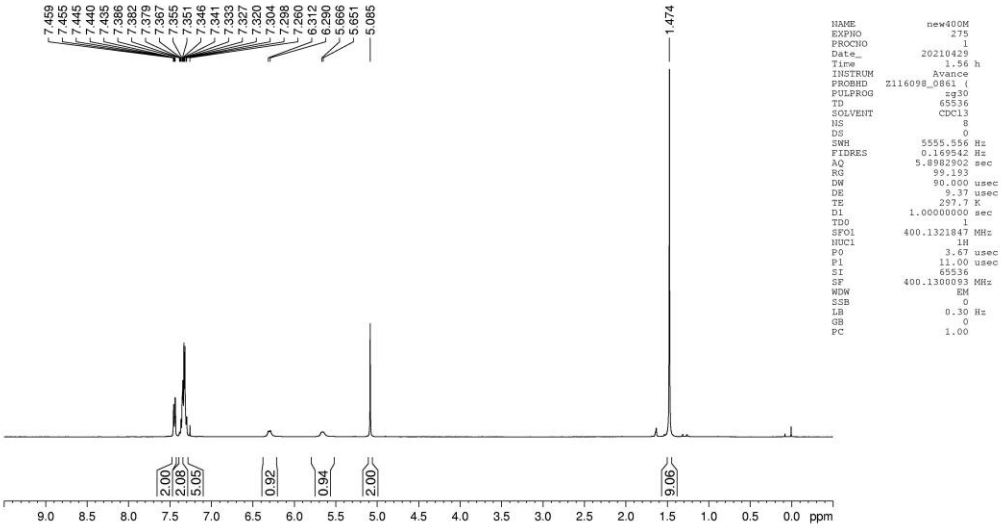



```
NAME                old400M
EXPNO                   269
PROCNO                    1
Date_              20210605
Time                   9.18
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG           zgpg30
TD                     65536
SOLVENT               CDCl3
NS                      212
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                   296.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

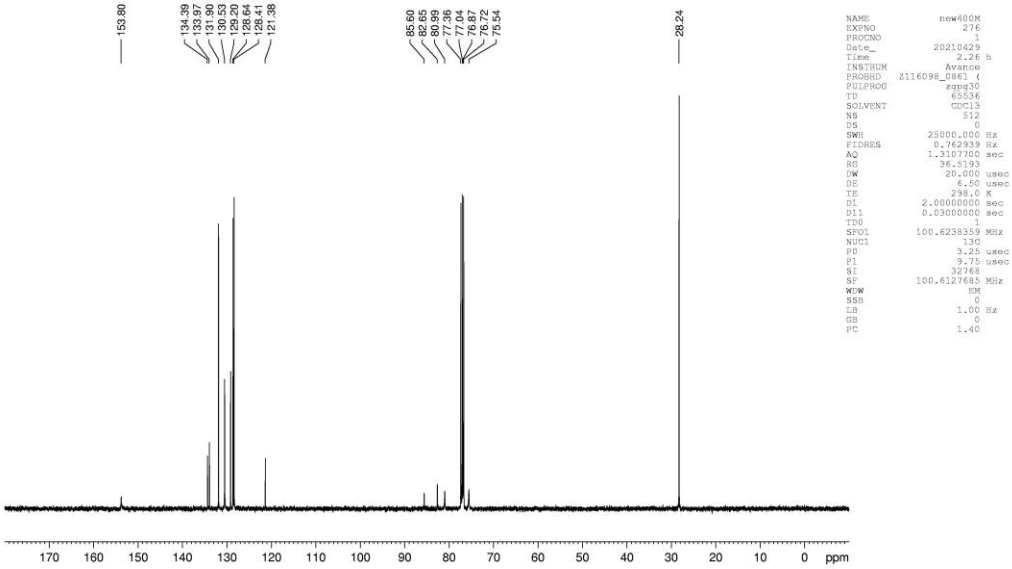
======== CHANNEL f1 ========
SFO1             100.6283629 MHz
NUC1                     13C
P1                    10.50 usec
SI                     32768
SF               100.6177980 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0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



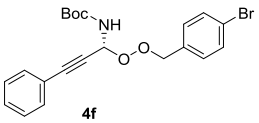
4e



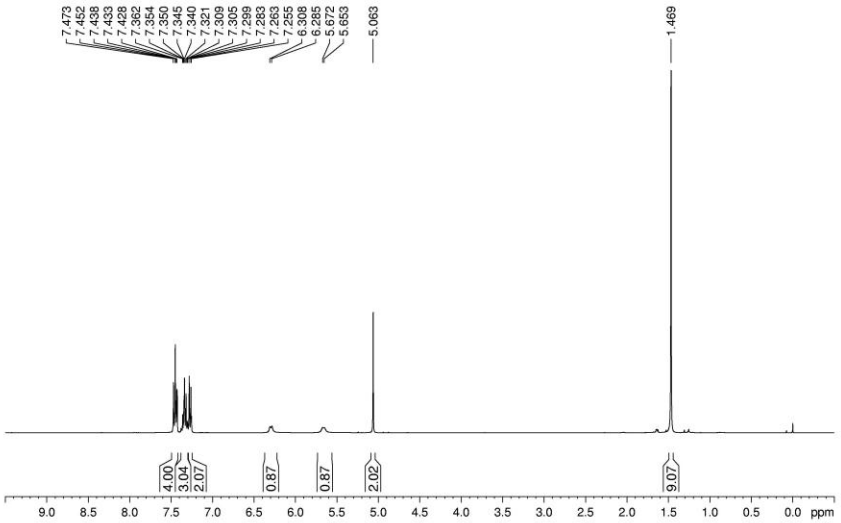
NAME	new400M
EXPNO	275
PROCNO	1
Date_	20210429
Time	1.56 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	99.193
DW	90.000 usec
DE	9.37 usec
TE	297.7 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.67 usec
P1	11.00 usec
SI	65536
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	new400M
EXPNO	276
PROCNO	1
Date_	20210429
Time	2.26 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 sec
RG	36.5193
DW	20.000 usec
DE	6.50 usec
TE	298.0 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238359 MHz
NUC1	13C
P0	3.25 usec
P1	9.75 usec
NS	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

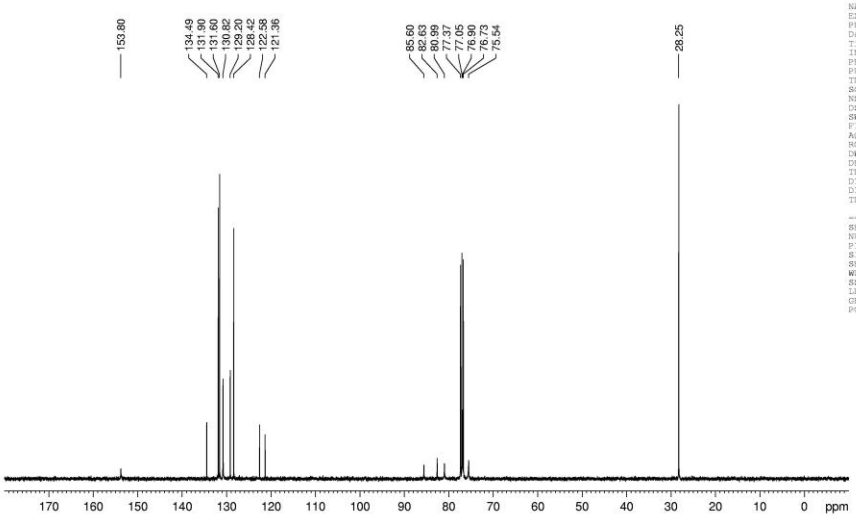


4f



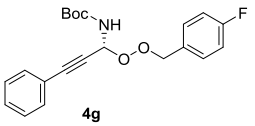

```
NAME                old400M
EXPNO                   241
PROCNO                    1
Date_              20210430
Time                  19.16
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES             0.122266 Hz
AQ                 4.0894966 sec
RG                     31.56
DW                    62.400 usec
DE                     6.50 usec
TE                    296.1 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1             400.1522008 MHz
NUC1                     1H
P1                    10.75 usec
SI                    65536
SF               400.1500112 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

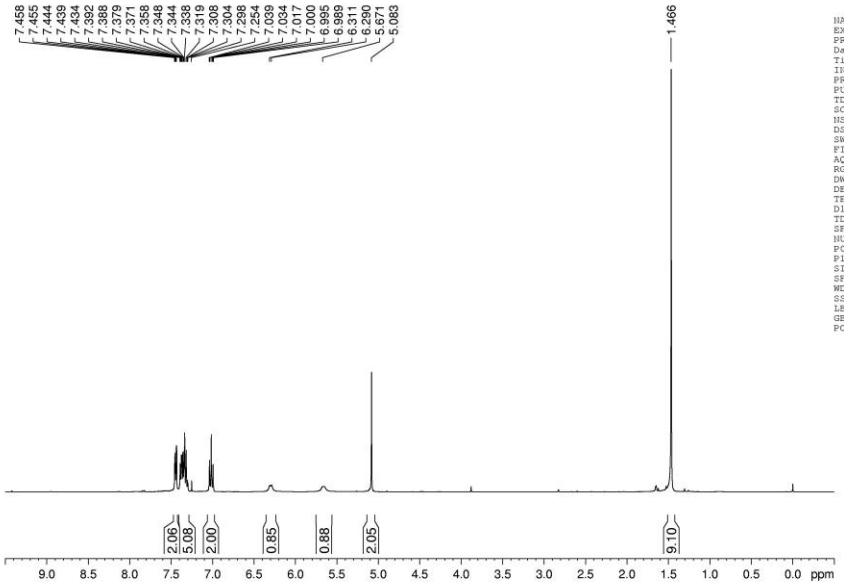



```
NAME                old400M
EXPNO                   242
PROCNO                    1
Date_              20210430
Time                  19.19
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      353
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                    296.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

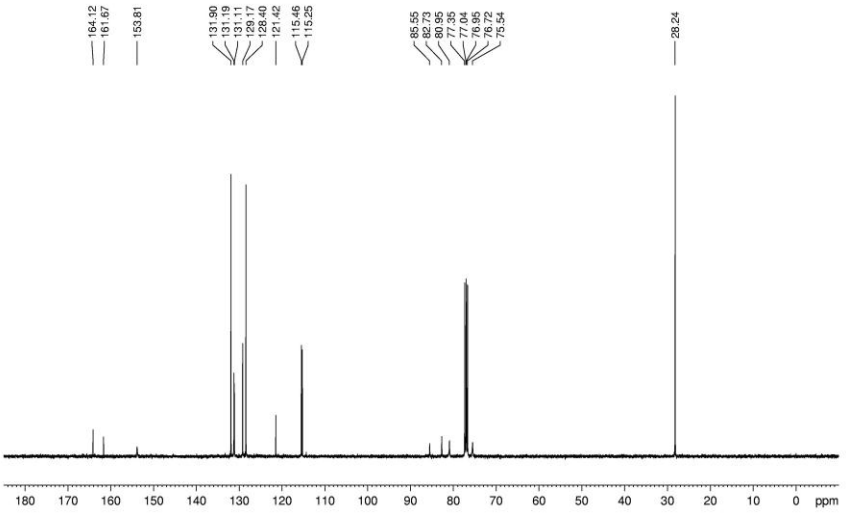
======== CHANNEL f1 ========
SFO1             100.6283629 MHz
NUC1                     13C
P1                    10.50 usec
SI                    32768
SF               100.6177980 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC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



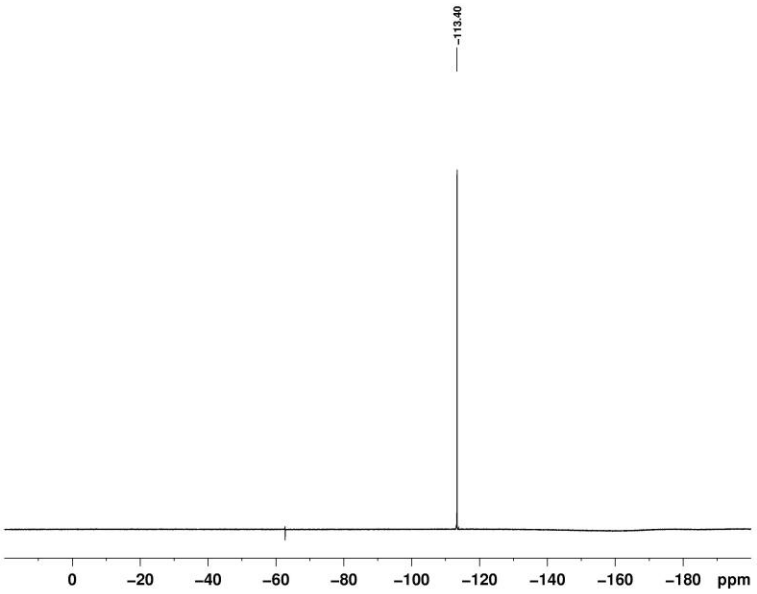
4g



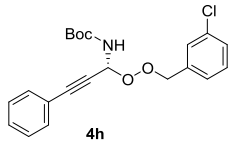
NAME	new400M
EXPNO	286
PROCNO	1
Date_	20210604
Time	23.46 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	92.7934
DW	90.000 usec
DE	9.37 usec
TE	299.3 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.67 usec
P1	11.00 usec
SI	65536
SF	400.1300121 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



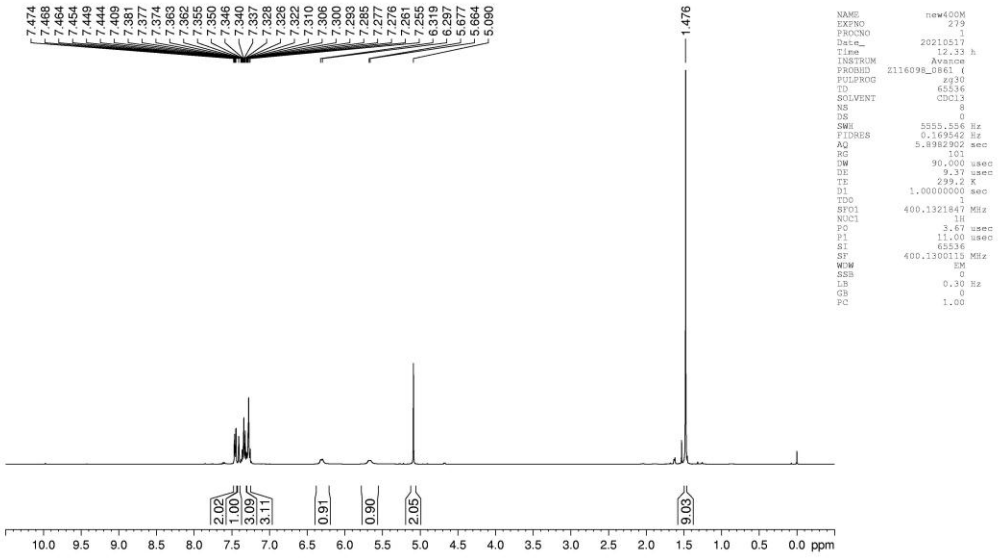
NAME	new400M
EXPNO	287
PROCNO	1
Date_	20210605
Time	0.16 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	512
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	39.1204
DW	20.000 usec
DE	6.50 usec
TE	299.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238339 MHz
NUC1	13C
P0	3.25 usec
P1	9.75 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



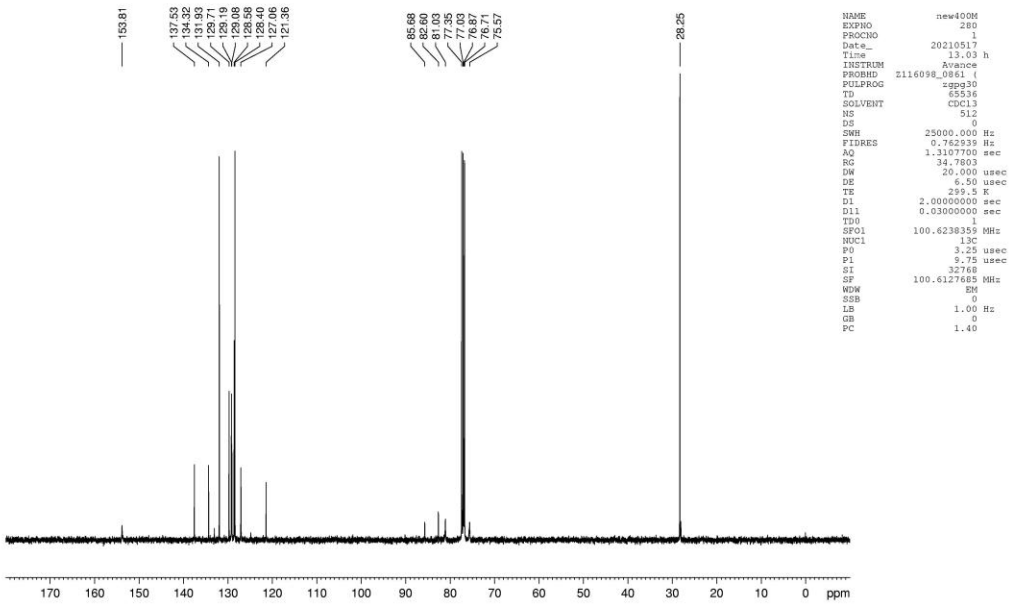
NAME	new400M
EXPNO	288
PROCNO	1
Date_	20210605
Time	0.18 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgig
TD	131072
SOLVENT	CDCl3
NS	16
DS	4
SWH	90909.094 Hz
FIDRES	1.387163 Hz
AQ	0.7209460 sec
RG	101
DW	5.500 usec
DE	6.50 usec
TE	299.7 K
D1	1.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	376.4607164 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



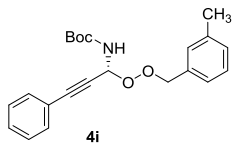
4h



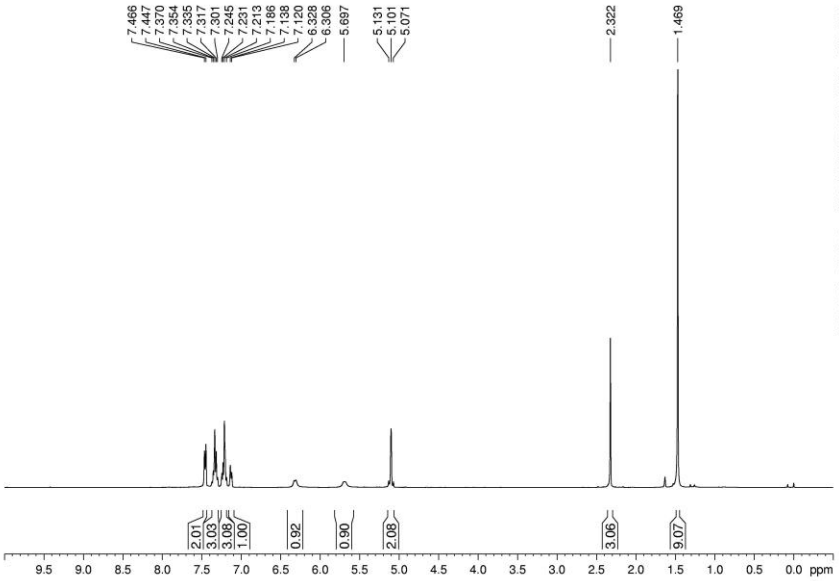

```
NAME            new400M
EXPNO               279
PROCNO                1
Date_          20210517
Time              12.33 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             5555.556 Hz
FIDRES          0.169542 Hz
AQ              5.8982902 sec
RG                   101
DW                90.000 usec
DE                 9.37 usec
TE                299.2 K
D1             1.00000000 sec
TD0                    1
SFO1          400.1321847 MHz
NUC1                  1H
P0                  3.67 usec
P1                 11.00 usec
SI                 65536
SF            400.1300115 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



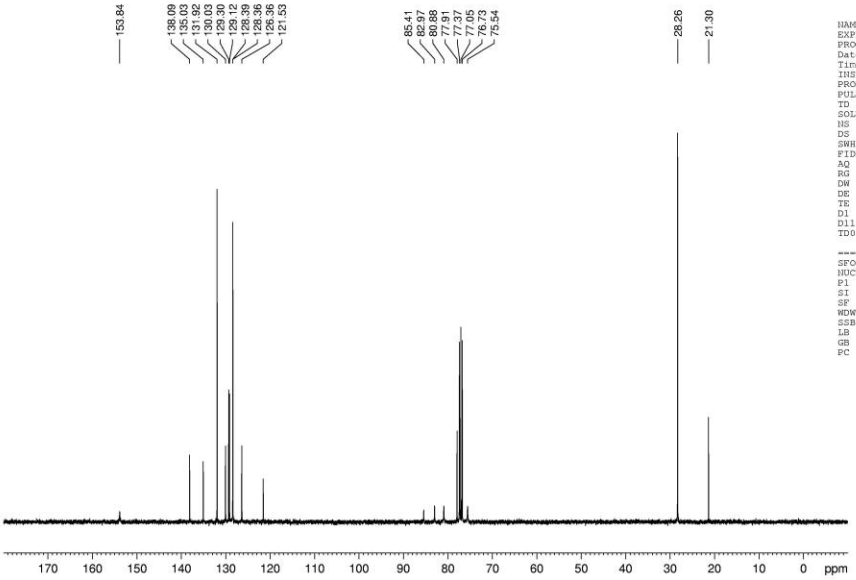

```
NAME            new400M
EXPNO               280
PROCNO                1
Date_          20210517
Time              13.03 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH            25000.000 Hz
FIDRES          0.762939 Hz
AQ              1.3107700 sec
RG                34.7803
DW                20.000 usec
DE                 6.50 usec
TE                299.5 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                    1
SFO1          100.6238359 MHz
NUC1                  13C
P0                  3.25 usec
P1                  9.75 usec
SI                 32768
SF            100.6127685 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



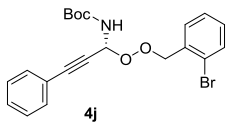
4i



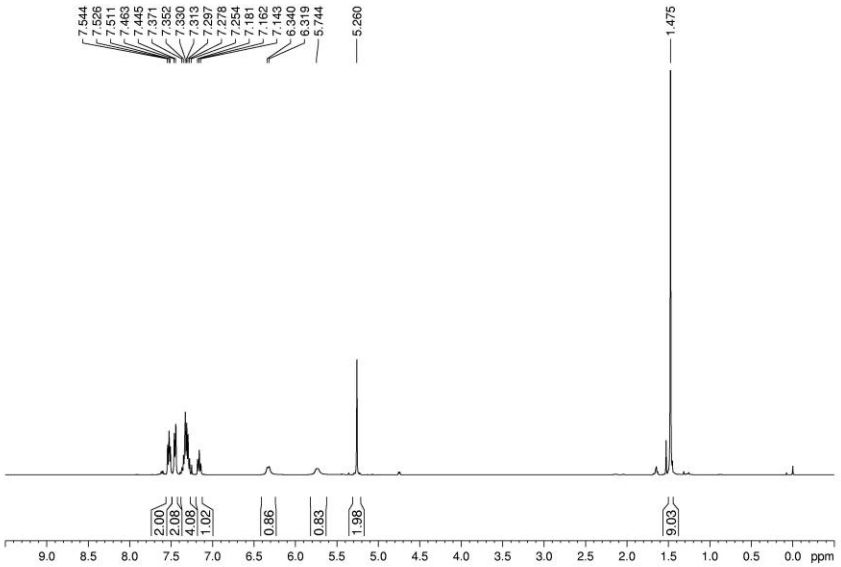
NAME	old400M
EXPNO	264
PROCNO	1
Date_	20210603
Time	22.18
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	31.56
DW	62.400 usec
DE	6.50 usec
TE	298.8 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1522008 MHz
NUC1	1H
P1	10.75 usec
SI	65536
SF	400.1500165 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



NAME	old400M
EXPNO	265
PROCNO	1
Date_	20210603
Time	22.21
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	344
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	298.8 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	100.6177980 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

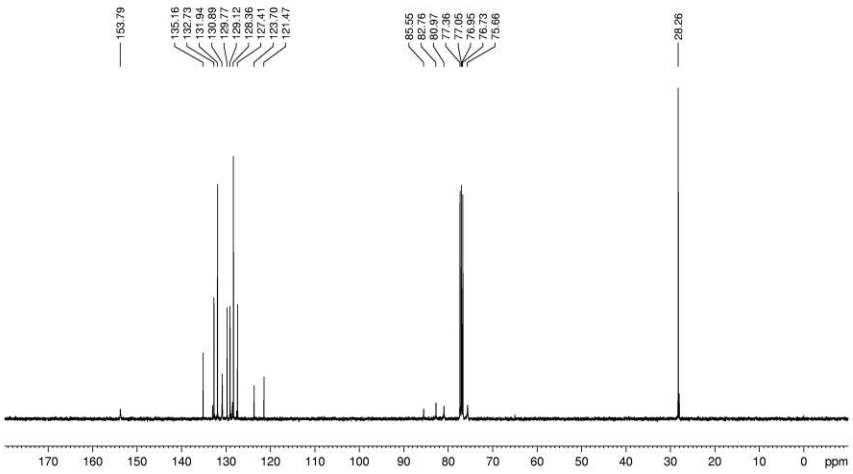


4j



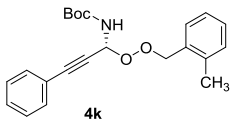

```
NAME                old400M
EXPNO                   256
PROCNO                    1
Date_              20210516
Time                  17.22
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG             zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                   297.7 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1               400.1522008 MHz
NUC1                      1H
P1                   10.75 usec
SI                    65536
SF                 400.1500126 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                     1.00
```

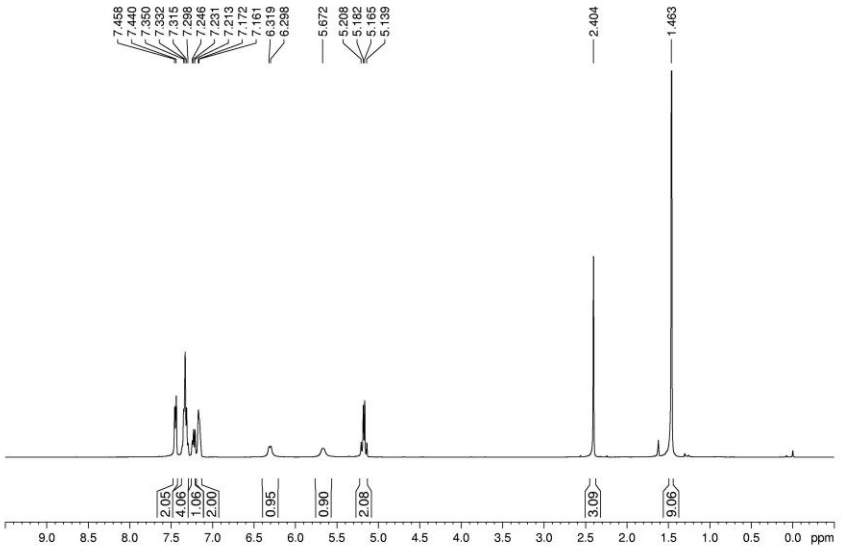



```
NAME                old400M
EXPNO                   257
PROCNO                    1
Date_              20210516
Time                  17.52
INSTRUM               spect
PROBHD     5 mm PABBO BB/
PULPROG           zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH               25252.525 Hz
FIDRES                0.385323 Hz
AQ                 1.2976629 sec
RG                   195.85
DW                   19.800 usec
DE                    6.50 usec
TE                   298.2 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                 100.6177980 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                     1.40
```

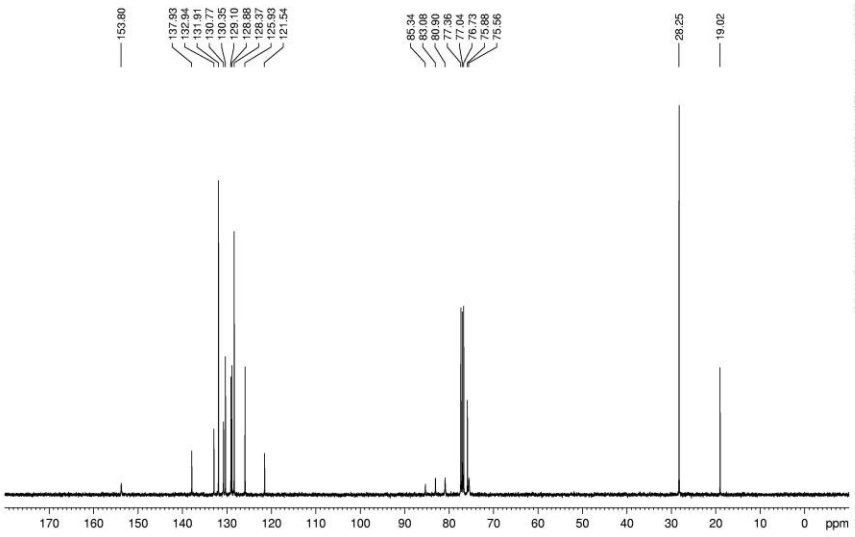


4k



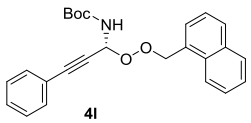

```
NAME                old400M
EXPNO                   258
PROCNO                    1
Date_              20210603
Time                  20.38
INSTRUM               spect
PROBHD  5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ                4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                   299.2 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1              400.1522008 MHz
NUC1                      1H
P1                    10.75 usec
SI                    65536
SF                400.1500167 MHz
WDW                       EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

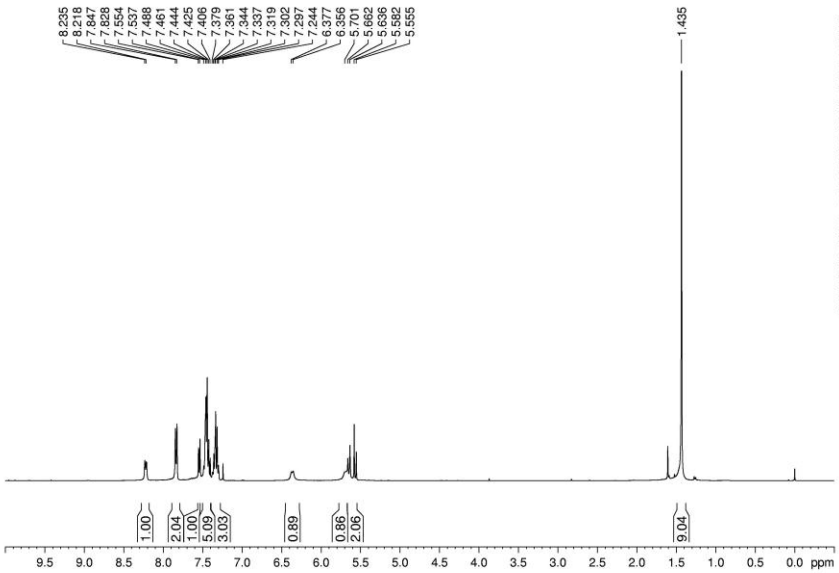



```
NAME                old400M
EXPNO                   259
PROCNO                    1
Date_              20210603
Time                  20.45
INSTRUM               spect
PROBHD  5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     512
DS                       0
SWH               25252.525 Hz
FIDRES            0.385323 Hz
AQ                1.2976629 sec
RG                    195.85
DW                   19.800 usec
DE                    6.50 usec
TE                   299.3 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                100.6177980 MHz
WDW                       EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

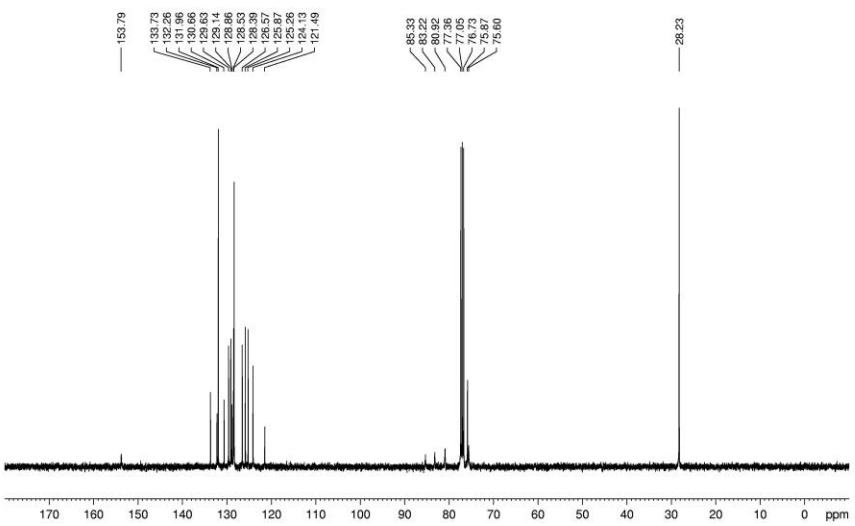


4l



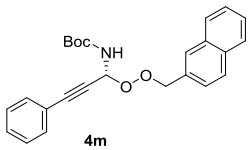

```
NAME                  old400M
EXPNO                     266
PROCNO                      1
Date_               20210605
Time                    8.51
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH                8012.820 Hz
FIDRES                0.122266 Hz
AQ                 4.0894966 sec
RG                     52.37
DW                    62.400 usec
DE                     6.50 usec
TE                    296.1 K
D1                 1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1               400.1522008 MHz
NUC1                       1H
P1                      10.75 usec
SI                      65536
SF                 400.1500154 MHz
WDW                        EM
SSB                         0
LB                       0.30 Hz
GB                          0
PC                       1.00
```

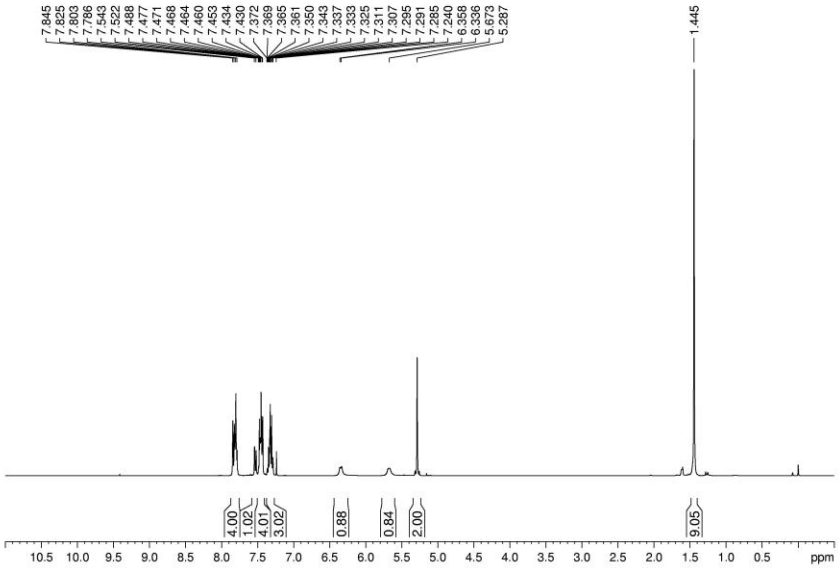



```
NAME                  old400M
EXPNO                     267
PROCNO                      1
Date_               20210605
Time                    8.54
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      343
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                    296.1 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                       1

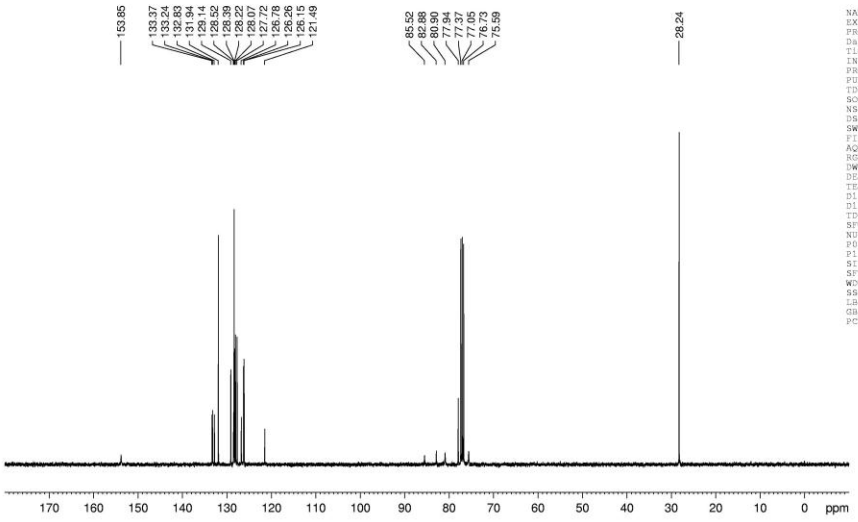
======== CHANNEL f1 ========
SFO1               100.6283629 MHz
NUC1                       13C
P1                      10.50 usec
SI                      32768
SF                 100.6177980 MHz
WDW                        EM
SSB                         0
LB                       1.00 Hz
GB          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



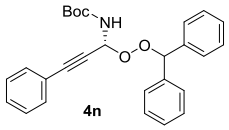
4m



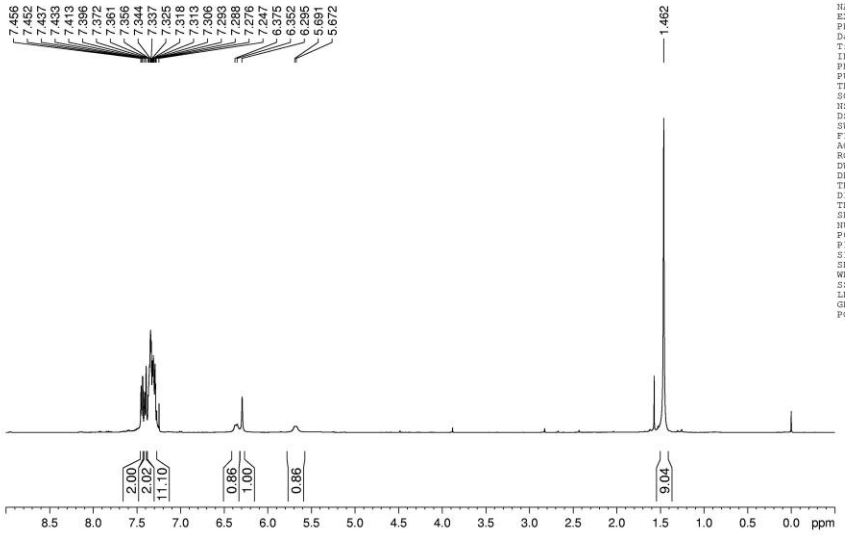

```
NAME                new400M
EXPNO                   277
PROCNO                    1
Date_              20210429
Time                   2.30 h
INSTRUM                Avance
PROBHD     Z116098_0861 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        0
SWH               5555.556 Hz
FIDRES               0.169542 Hz
AQ                5.8982902 sec
RG                   95.8865
DW                   90.000 use
DE                    9.37 use
TE                   297.6 K
D1                1.00000000 sec
TD0                       1
SFO1            400.1321847 MHz
NUC1                     1H
P0                    3.67 use
P1                   11.00 use
SI                    65536
SF              400.1300175 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



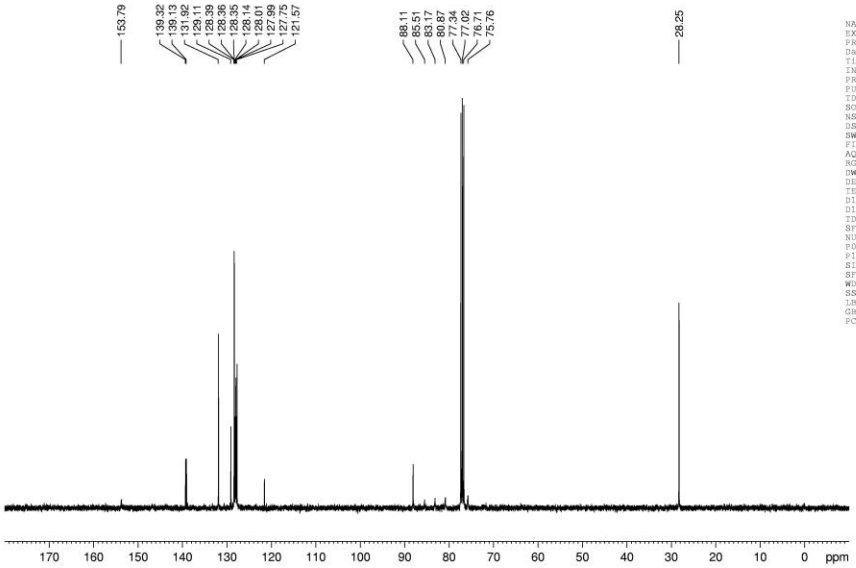

```
NAME                new400M
EXPNO                   278
PROCNO                    1
Date_              20210429
Time                   3.00 h
INSTRUM                Avance
PROBHD     Z116098_0861 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      512
DS                        0
SWH              25000.000 Hz
FIDRES               0.762939 Hz
AQ                1.3107700 sec
RG                   36.6754
DW                   20.000 usec
DE                    6.50 usec
TE                   298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1            100.6238359 MHz
NUC1                     13C
P0                    3.25 usec
P1                    9.75 usec
SI                    32768
SF              100.6127685 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```



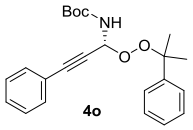
4n



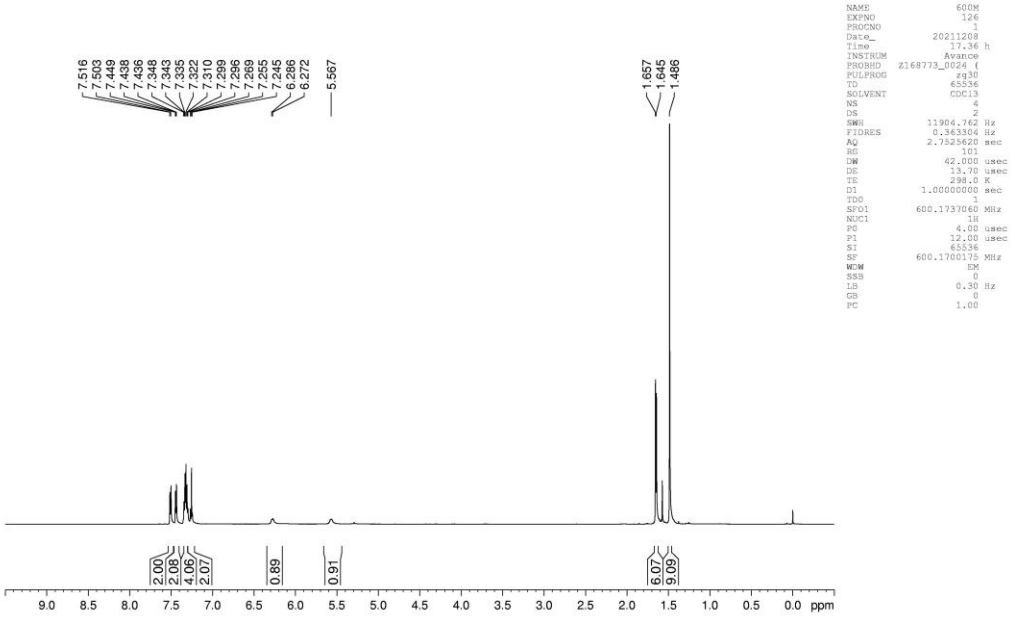

```
NAME            new400M
EXPNO               284
PROCNO                1
Date_          20210604
Time              23.10 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH           5555.556 Hz
FIDRES        0.169542 Hz
AQ            5.8982902 sec
RG                   101
DW               90.000 usec
DE                9.37 usec
TE                299.2 K
D1           1.00000000 sec
TD0                    1
SFO1       400.1321847 MHz
NUC1                  1H
P0                3.67 usec
P1               11.00 usec
SI                 65536
SF         400.1300147 MHz
WDW                   EM
SSB                    0
LB                0.30 Hz
GB                     0
PC                  1.00
```



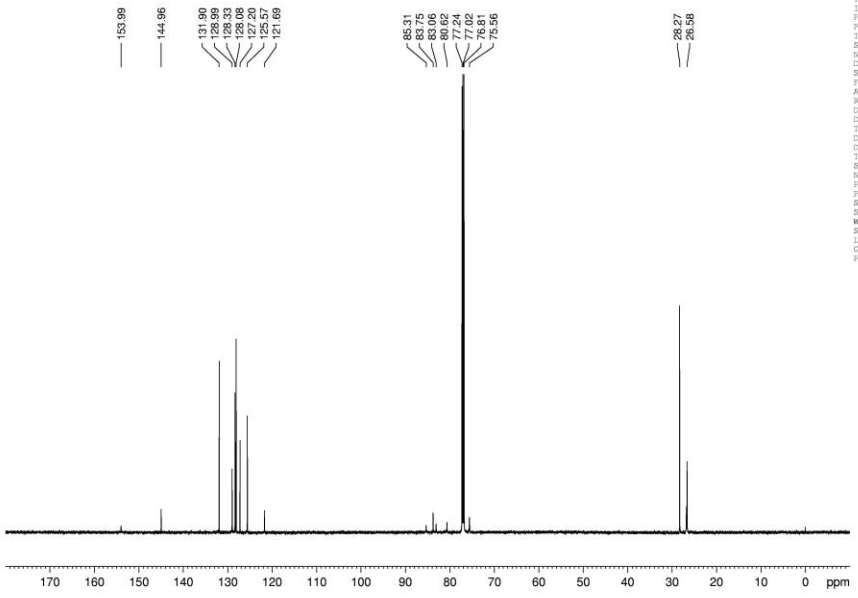

```
NAME            new400M
EXPNO               285
PROCNO                1
Date_          20210604
Time              23.41 h
INSTRUM          Avance
PROBHD    Z116098_0861 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   512
DS                     0
SWH          25000.000 Hz
FIDRES        0.762939 Hz
AQ            1.3107700 sec
RG                34.1743
DW               20.000 usec
DE                6.50 usec
TE                299.8 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1
SFO1       100.6238359 MHz
NUC1                  13C
P0                3.25 usec
P1                9.75 usec
SI                 32768
SF         100.6127685 MHz
WDW                   EM
SSB                    0
LB                1.00 Hz
GB                     0
PC                  1.40
```



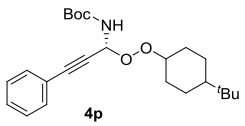
4o



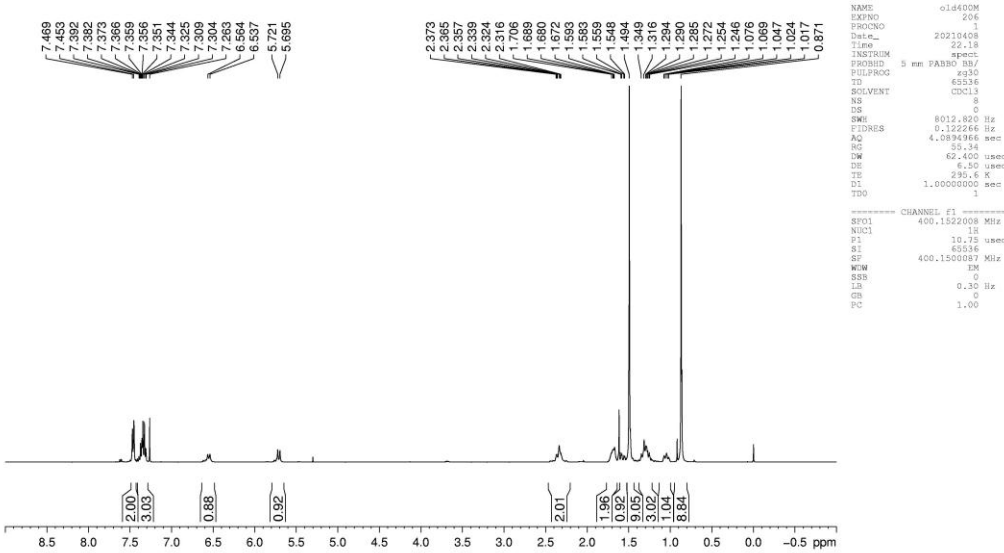

```
NAME                  600M
EXPNO                  126
PROCNO                   1
Date_             20211208
Time                17.36 h
INSTRUM               Avance
PROBHD    Z168773_0024 (
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        4
DS                        2
SWH               11904.762 Hz
FIDRES            0.363304 Hz
AQ                2.7525620 sec
RG                      101
DW                 42.000 usec
DE                 13.70 usec
TE                 298.0 K
D1                1.00000000 sec
TD0                       1
SFO1          600.1737060 MHz
NUC1                    1H
P0                 4.00 usec
P1                 12.00 usec
SI                    65536
SF            600.1700175 MHz
WDW                     EM
SSB                      0
LB                  0.30 Hz
GB                       0
PC                    1.00
```

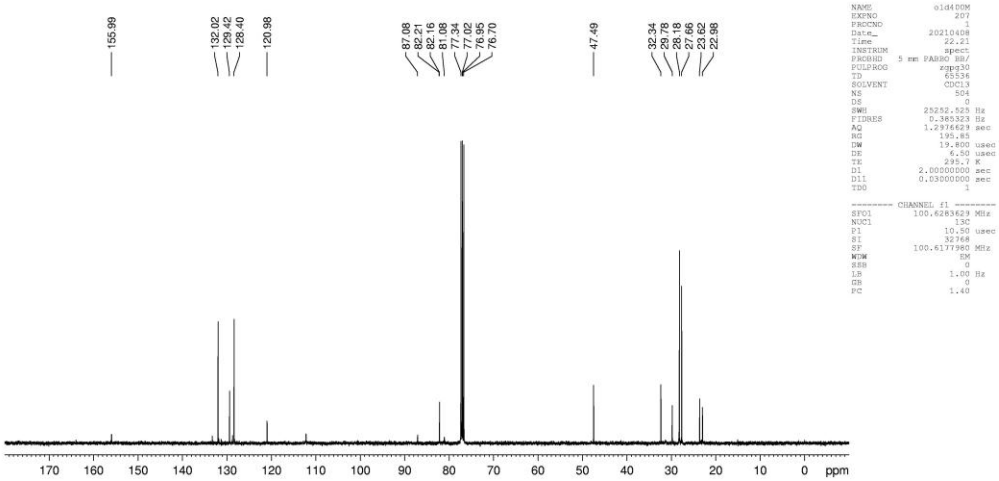


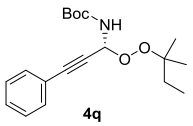

```
NAME                  600M
EXPNO                  127
PROCNO                   1
Date_             20211208
Time                17.50 h
INSTRUM               Avance
PROBHD    Z168773_0024 (
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      256
DS                        4
SWH               39714.285 Hz
FIDRES            1.089913 Hz
AQ                0.9175540 sec
RG                      101
DW                 14.000 usec
DE                 18.00 usec
TE                 298.0 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1
SFO1          150.9279578 MHz
NUC1                    13C
P0                 3.33 usec
P1                 10.00 usec
SI                    32768
SF            150.9128665 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                    1.40
```


Boc
NH
O
O
tBu

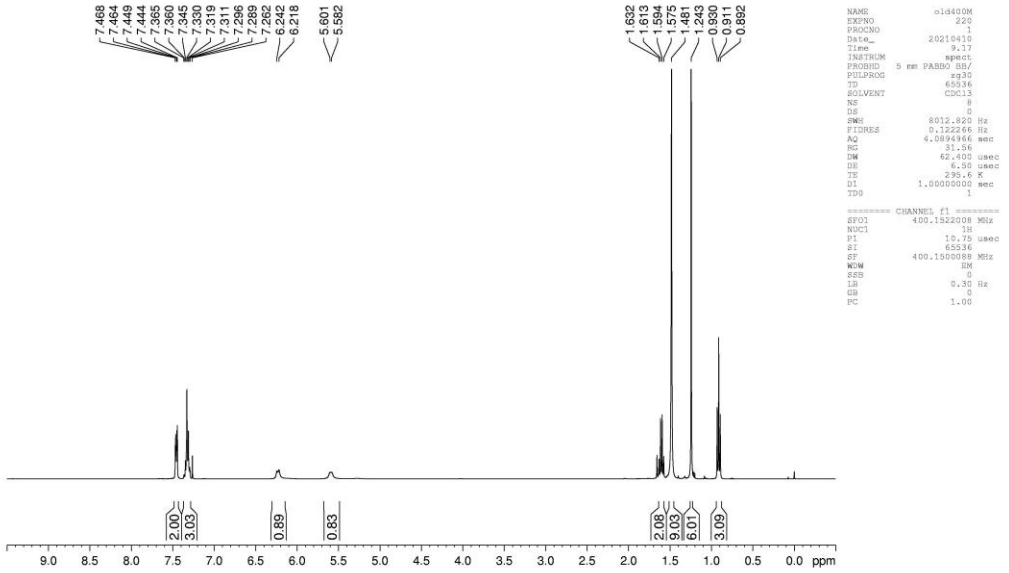
4p


7.469
7.453
7.392
7.382
7.373
7.366
7.359
7.356
7.351
7.344
7.325
7.309
7.304
7.263
6.564
6.537
5.721
5.695
2.373
2.365
2.357
2.339
2.324
2.316
2.306
1.698
1.680
1.672
1.593
1.583
1.555
1.548
1.494
1.349
1.316
1.294
1.290
1.285
1.272
1.254
1.246
1.076
1.069
1.047
1.024
1.017
0.871
2.00
3.03
0.88
0.92
2.01
1.96
0.92
9.05
3.02
1.04
8.84
8.5 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 −0.5 ppm
NAME old400M
EXPNO 206
PROCNO 1
Date_ 20210408
Time 22.18
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 sec
RG 59.34
DW 62.400 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1522008 MHz
NUC1 1H
P1 10.75 usec
SI 65536
SF 400.1500087 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00


155.99
132.02
129.42
128.40
120.98
87.08
82.21
82.16
81.08
77.34
77.02
76.95
76.70
47.49
32.34
29.78
28.18
27.66
25.52
22.98
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
NAME old400M
EXPNO 207
PROCNO 1
Date_ 20210408
Time 22.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 504
DS 4
SWH 25252.525 Hz
FIDRES 0.385323 Hz
AQ 1.2976629 sec
RG 195.85
DW 19.800 usec
DE 6.50 usec
TE 295.7 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 100.6177980 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

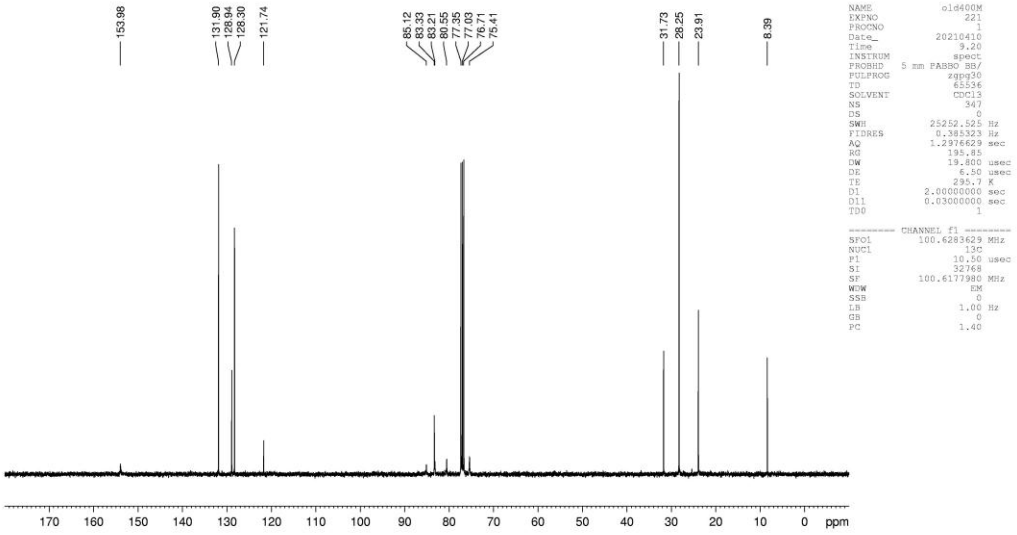


4q



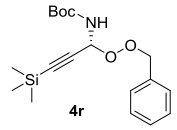

```
NAME                 old400M
EXPNO                    220
PROCNO                     1
Date_               20210410
Time                    9.17
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                     65536
SOLVENT                CDCl3
NS                         8
DS                         0
SWH                 8012.820 Hz
FIDRES              0.122266 Hz
AQ                  4.0894966 sec
RG                     31.56
DW                    62.400 usec
DE                     6.50 usec
TE                    299.6 K
D1                  1.00000000 sec
TD0                        1

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

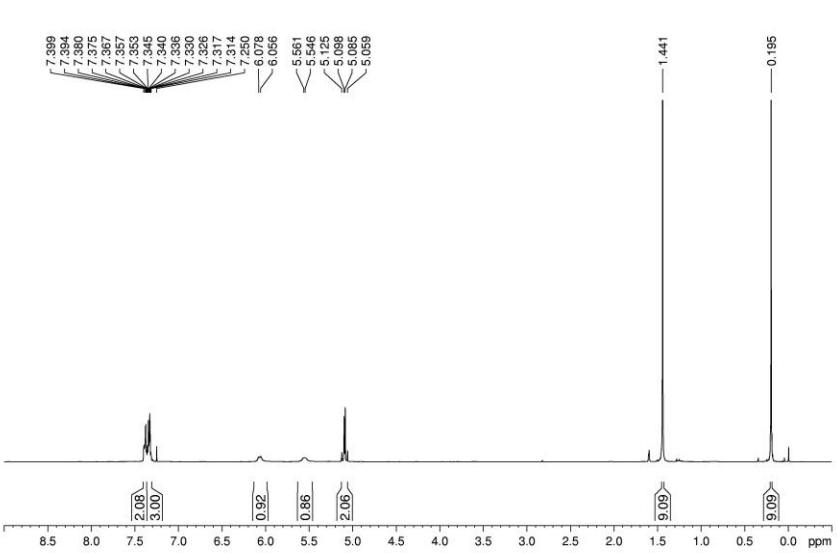



```
NAME                 old400M
EXPNO                    221
PROCNO                     1
Date_               20210410
Time                    9.20
INSTRUM                spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                     65536
SOLVENT                CDCl3
NS                       347
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                    295.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                        1

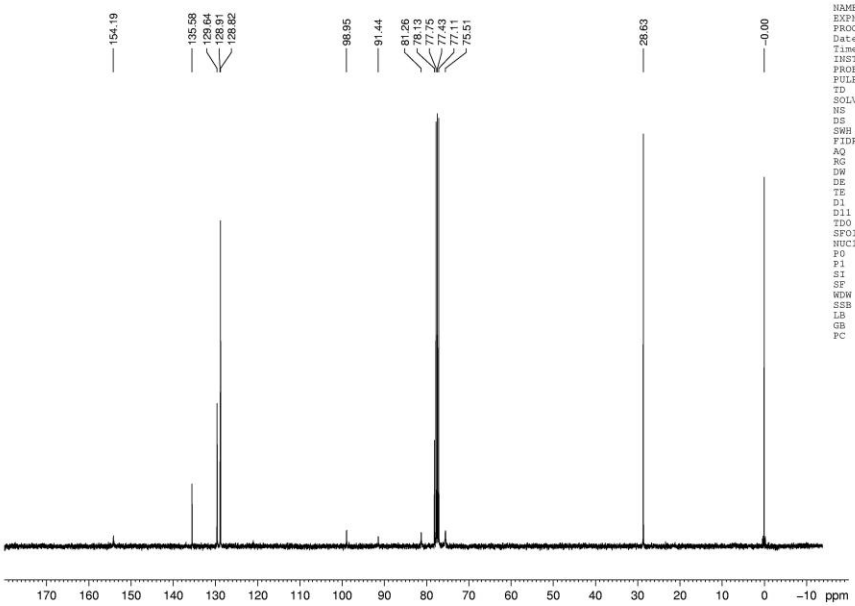
======== CHANNEL f1 ========
SFO1                100.6283629 MHz
NUC1                       13C
P1                    10.50 usec
SI                     32768
SF                  100.6177980 MHz
WDW                        EM
SSB                        0
LB                      1.00 Hz
GB             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



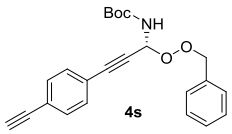
4r



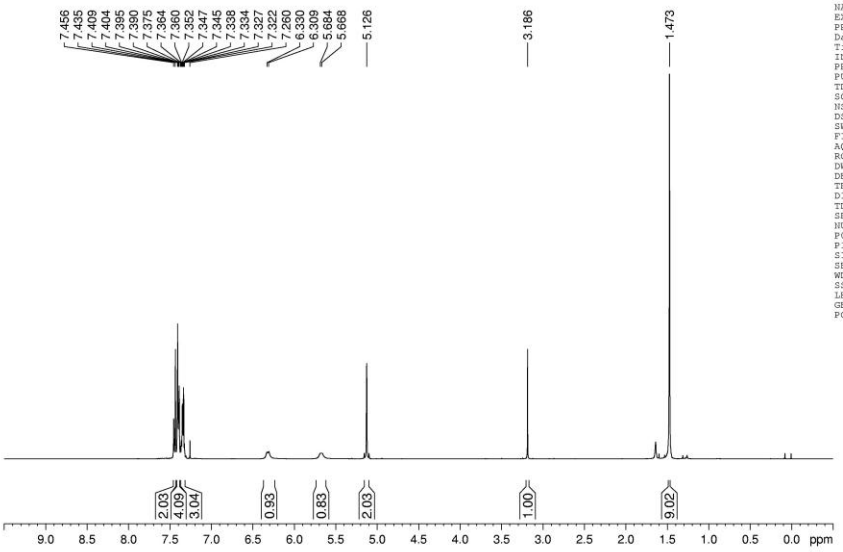

```
NAME              new400M
EXPNO                 291
PROCNO                  1
Date_            20210605
Time                23.15 h
INSTRUM           Avance
PROBHD    Z116098_0861 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              5555.556 Hz
FIDRES           0.169542 Hz
AQ               5.8982902 sec
RG                    101
DW                 90.000 usec
DE                  9.37 usec
TE                 299.8 K
D1              1.00000000 sec
TD0                     1
SFO1           400.1321847 MHz
NUC1                   1H
P0                  3.67 usec
P1                 11.00 usec
SI                  65536
SF             400.1300135 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```



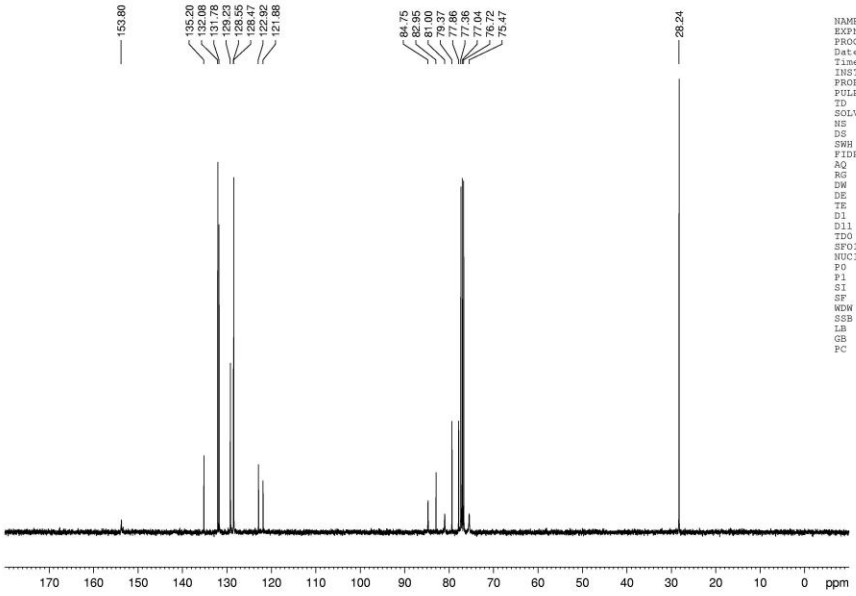

```
NAME              new400M
EXPNO                 292
PROCNO                  1
Date_            20210605
Time                23.45 h
INSTRUM           Avance
PROBHD    Z116098_0861 (
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    512
DS                      0
SWH             25000.000 Hz
FIDRES           0.762939 Hz
AQ               1.3107700 sec
RG                 36.6754
DW                 20.000 usec
DE                  6.50 usec
TE                 299.9 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                     1
SFO1           100.6238359 MHz
NUC1                   13C
P0                  3.25 usec
P1                  9.75 usec
SI                  32768
SF             100.6127272 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```



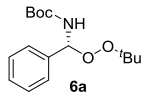
4s



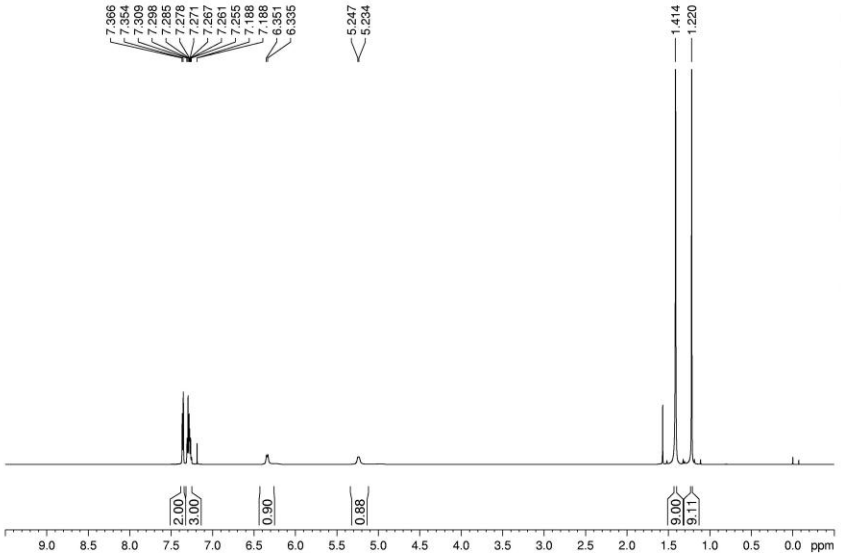
NAME	new400M
EXPNO	297
PROCNO	1
Date_	20210729
Time	21.59 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	5555.556 Hz
FIDRES	0.169542 Hz
AQ	5.8982902 sec
RG	101
DW	90.000 usec
DE	9.37 usec
TE	298.1 K
D1	1.00000000 sec
TD0	1
SFO1	400.1321847 MHz
NUC1	1H
P0	3.67 usec
P1	11.00 usec
SI	65536
SF	400.1300096 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00



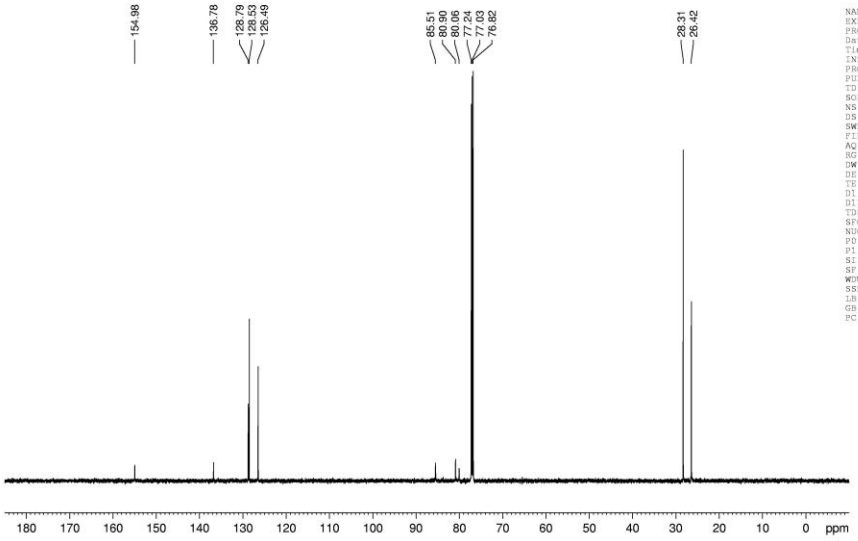
NAME	new400M
EXPNO	298
PROCNO	1
Date_	20210729
Time	22.21 h
INSTRUM	Avance
PROBHD	Z116098_0861 (
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	376
DS	0
SWH	25000.000 Hz
FIDRES	0.762939 Hz
AQ	1.3107700 sec
RG	16.25
DW	20.000 usec
DE	6.50 usec
TE	298.2 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
SFO1	100.6238359 MHz
NUC1	13C
P0	3.25 usec
P1	9.75 usec
SI	32768
SF	100.6127685 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



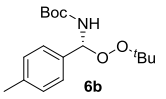
6a



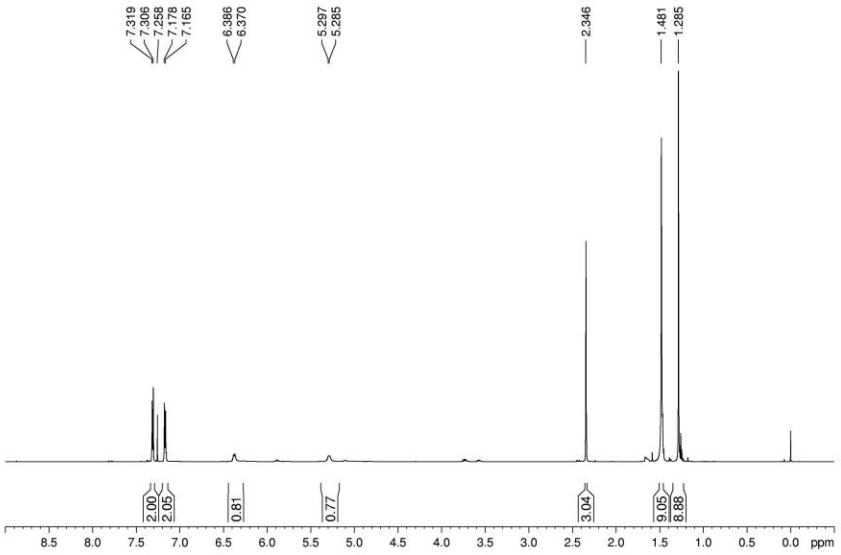

```
NAME                600M
EXPNO                 61
PROCNO                 1
Date_           20210917
Time                2.26 h
INSTRUM          Avance
PROBHD    Z168773_0024 (
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                    16
DS                     2
SWH             11904.762 Hz
FIDRES          0.363304 Hz
AQ              2.7525620 sec
RG                   101
DW                42.000 usec
DE                13.70 usec
TE                298.0 K
D1              1.00000000 sec
TD0                    1
SFO1          600.1737060 MHz
NUC1                  1H
P0                 4.00 usec
P1                12.00 usec
SI                 65536
SF            600.1700576 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



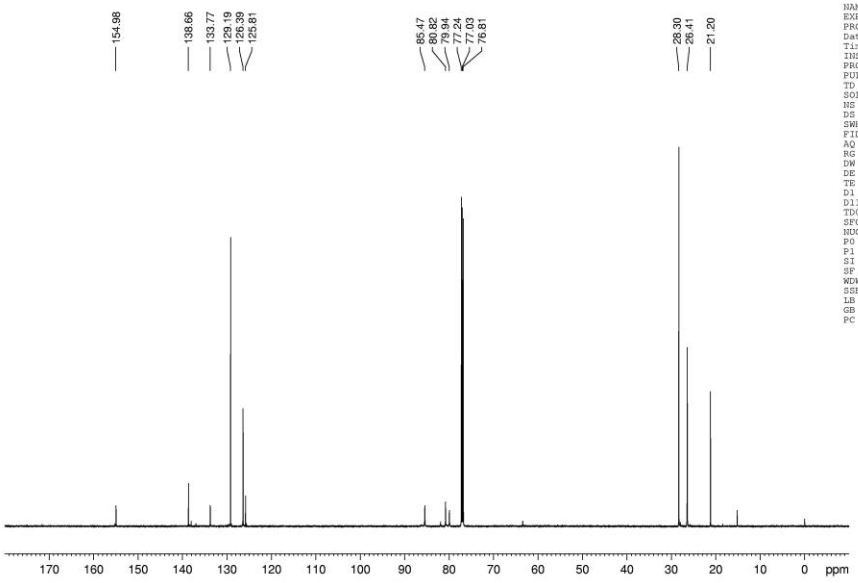

```
NAME                600M
EXPNO                 62
PROCNO                 1
Date_           20210917
Time                2.28 h
INSTRUM          Avance
PROBHD    Z168773_0024 (
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    32
DS                     4
SWH             35714.285 Hz
FIDRES          1.089913 Hz
AQ              0.9175540 sec
RG                   101
DW                14.000 usec
DE                18.00 usec
TE                298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                    1
SFO1          150.9279578 MHz
NUC1                  13C
P0                 3.33 usec
P1                10.00 usec
SI                 32768
SF            150.9128665 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```



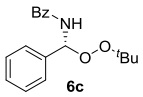
6b



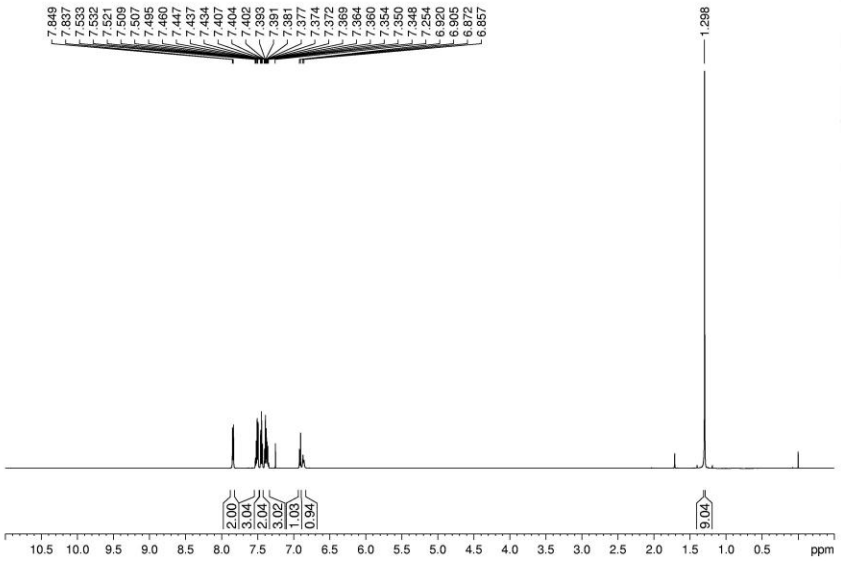

```
NAME                 600M
EXPNO                 116
PROCNO                  1
Date_             20211116
Time                15.30 h
INSTRUM             Avance
PROBHD    Z168773_0024 (
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       4
DS                       2
SWH              11904.762 Hz
FIDRES           0.363304 Hz
AQ               2.7525620 sec
RG                  67.0968
DW                  42.000 usec
DE                  13.70 usec
TE                  298.0 K
D1               1.00000000 sec
TD0                      1
SFO1           600.1737060 MHz
NUC1                    1H
P0                   4.00 usec
P1                  12.00 usec
SI                   65536
SF             600.1700154 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```



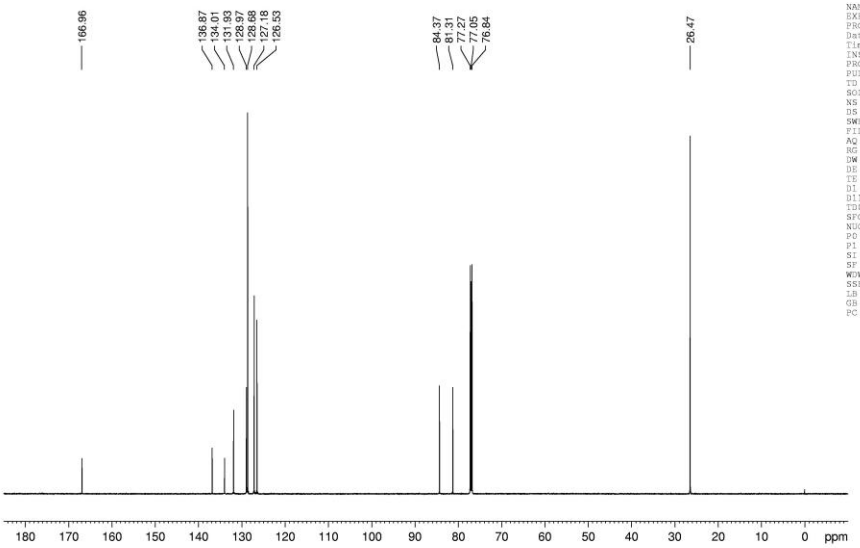

```
NAME                 600M
EXPNO                 117
PROCNO                  1
Date_             20211116
Time                15.44 h
INSTRUM             Avance
PROBHD    Z168773_0024 (
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH              35714.285 Hz
FIDRES           1.089913 Hz
AQ               0.9175540 sec
RG                     101
DW                  14.000 usec
DE                  18.00 usec
TE                  298.0 K
D1               2.00000000 sec
D11              0.03000000 sec
TD0                      1
SFO1           150.9279578 MHz
NUC1                   13C
P0                   3.33 usec
P1                  10.00 usec
SI                   32768
SF             150.9128680 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```



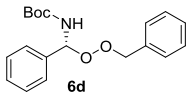
6c



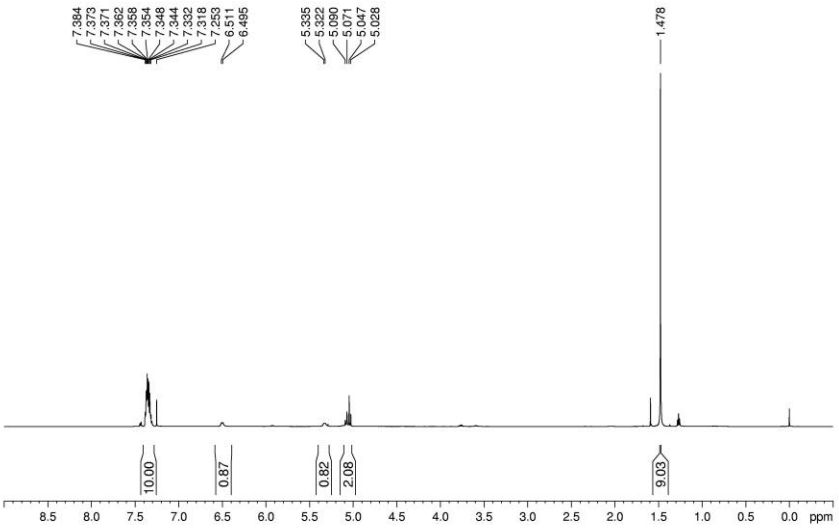

```
NAME                 600M
EXPNO                 110
PROCNO                  1
Date_            20211110
Time                20.18 h
INSTRUM             Avance
PROBHD    Z168773_0024 (
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       2
SWH             11904.762 Hz
FIDRES          0.363304 Hz
AQ              2.7525620 sec
RG                  69.3333
DW                 42.000 usec
DE                 13.70 usec
TE                 298.0 K
D1              1.00000000 sec
TD0                      1
SFO1        600.1737060 MHz
NUC1                    1H
P0                 4.00 usec
P1                12.00 usec
SI                   65536
SF          600.1700179 MHz
WDW                     EM
SSB                      0
LB                0.30 Hz
GB                       0
PC                1.00
```



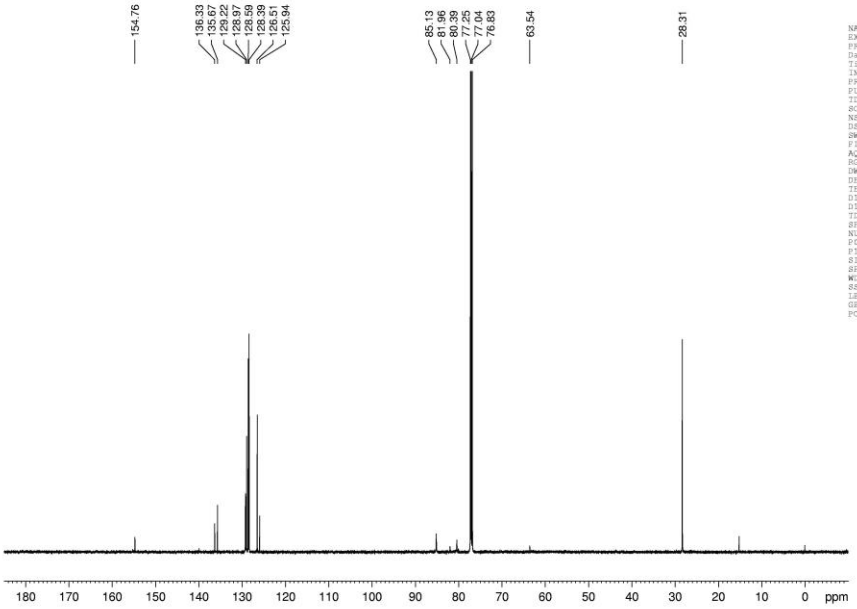

```
NAME                 600M
EXPNO                 111
PROCNO                  1
Date_            20211110
Time                20.32 h
INSTRUM             Avance
PROBHD    Z168773_0024 (
PULPROG           zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     256
DS                       4
SWH             35714.285 Hz
FIDRES          1.089913 Hz
AQ              0.9175540 sec
RG                     101
DW                 14.000 usec
DE                 18.00 usec
TE                 298.0 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                      1
SFO1        150.9279578 MHz
NUC1                    13C
P0                  3.33 usec
P1                 10.00 usec
SI                   32768
SF          150.9128665 MHz
WDW                     EM
SSB                      0
LB                1.00 Hz
GB                       0
PC                1.40
```



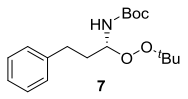
6d



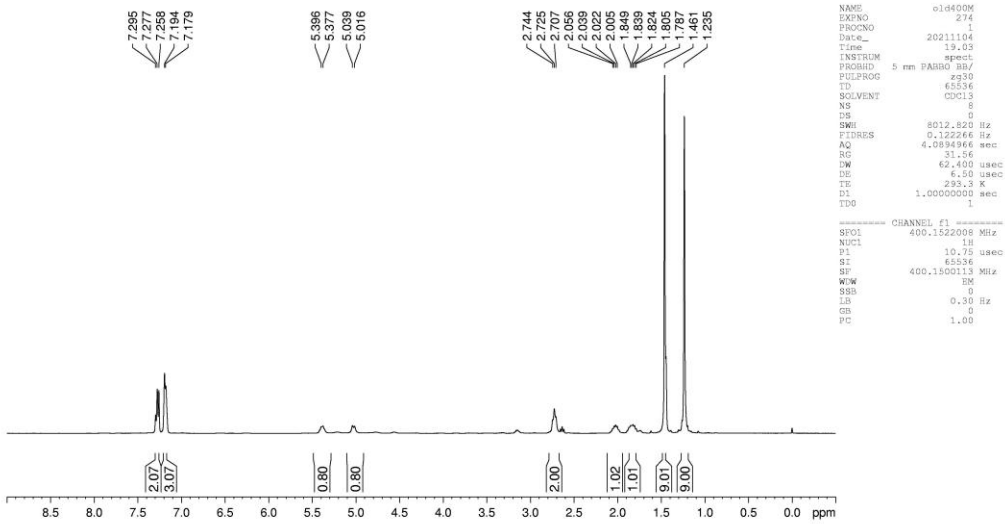

```
NAME                  600M
EXPNO                  122
PROCNO                   1
Date_           20211122
Time                15.08 h
INSTRUM             Avance
PROBHD   Z168773_0024 (
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      4
DS                      2
SWH             11904.762 Hz
FIDRES          0.363304 Hz
AQ              2.7525620 sec
RG                    101
DW                 42.000 usec
DE                 13.70 usec
TE                298.0 K
D1             1.00000000 sec
TD0                     1
SFO1           600.1737060 MHz
NUC1                   1H
P0                 4.00 usec
P1                12.00 usec
SI                  65536
SF             600.1700184 MHz
WDW                    EM
SSB                     0
LB                 0.30 Hz
GB                      0
PC                   1.00
```




```
NAME                  600M
EXPNO                  123
PROCNO                   1
Date_           20211122
Time                15.16 h
INSTRUM             Avance
PROBHD   Z168773_0024 (
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    128
DS                      4
SWH             35714.285 Hz
FIDRES          1.089913 Hz
AQ              0.9175540 sec
RG                    101
DW                 14.000 usec
DE                 18.00 usec
TE                298.0 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                     1
SFO1           150.9279578 MHz
NUC1                   13C
P0                 3.33 usec
P1                10.00 usec
SI                  32768
SF             150.9128665 MHz
WDW                    EM
SSB                     0
LB                 1.00 Hz
GB                      0
PC                   1.40
```

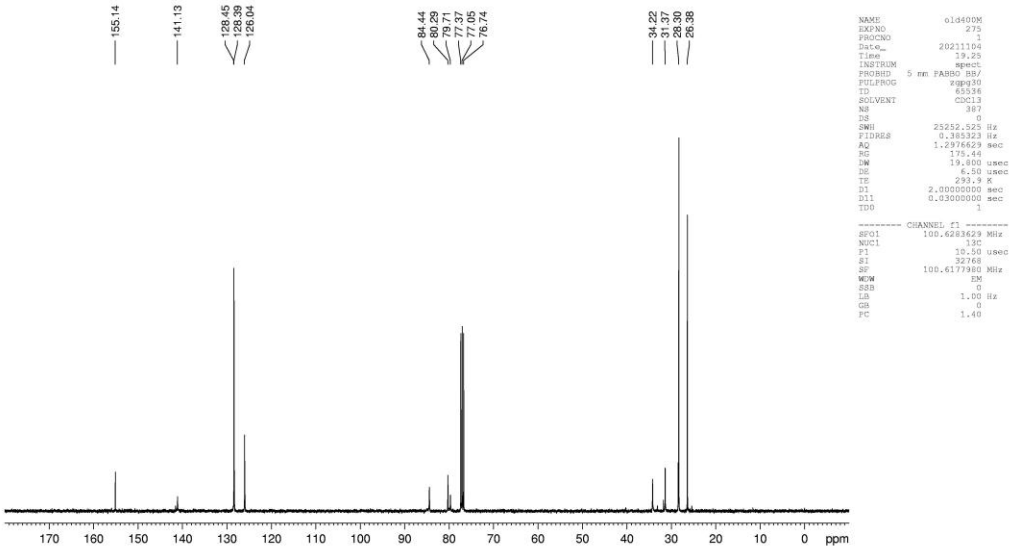


7



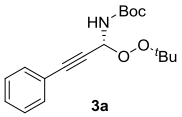

```
NAME                old400M
EXPNO                   274
PROCNO                    1
Date_              20211104
Time                  19.03
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8012.820 Hz
FIDRES               0.122266 Hz
AQ                4.0894966 sec
RG                    31.56
DW                   62.400 usec
DE                    6.50 usec
TE                   293.3 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
SFO1            400.1522008 MHz
NUC1                      1H
P1                    10.75 usec
SI                    65536
SF              400.1500113 MHz
WDW                      EM
SSB                       0
LB                    0.30 Hz
GB                        0
PC                    1.00
```

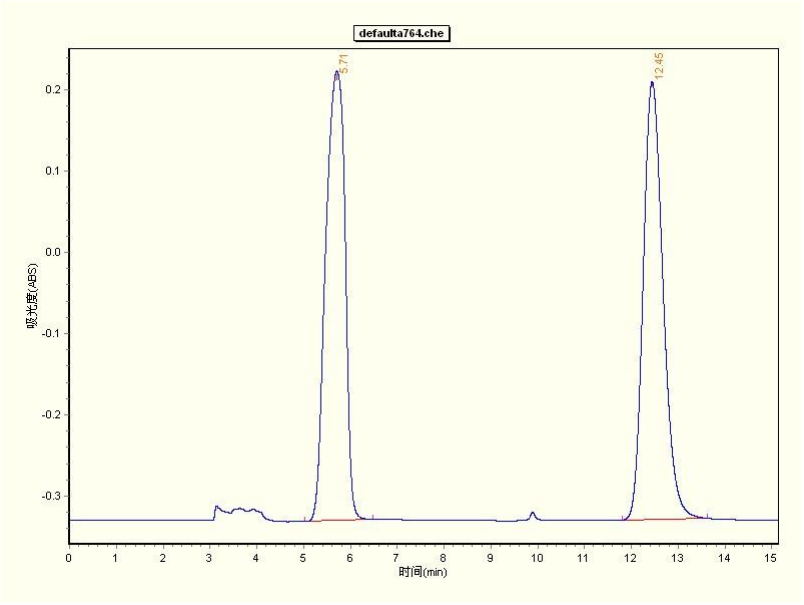



```
NAME                old400M
EXPNO                   275
PROCNO                    1
Date_              20211104
Time                  19.29
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG             zgpg30
TD                   65536
SOLVENT              CDCl3
NS                     367
DS                       0
SWH              25252.525 Hz
FIDRES               0.385323 Hz
AQ                1.2976629 sec
RG                   175.44
DW                   19.800 usec
DE                    6.50 usec
TE                   293.9 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

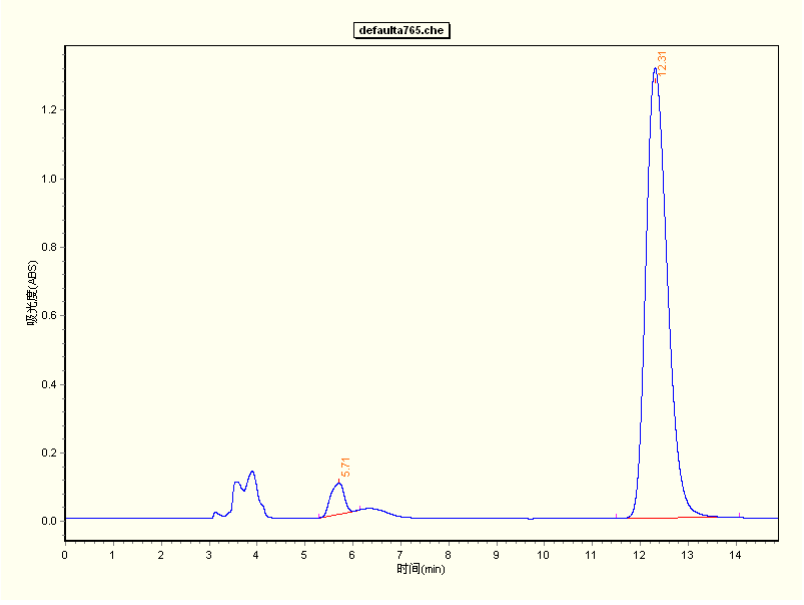
======== CHANNEL f1 ========
SFO1            100.6283629 MHz
NUC1                      13C
P1                    10.50 usec
SI                    32768
SF              100.6177980 MHz
WDW                      EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC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



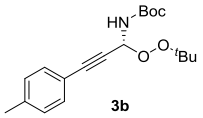
3a



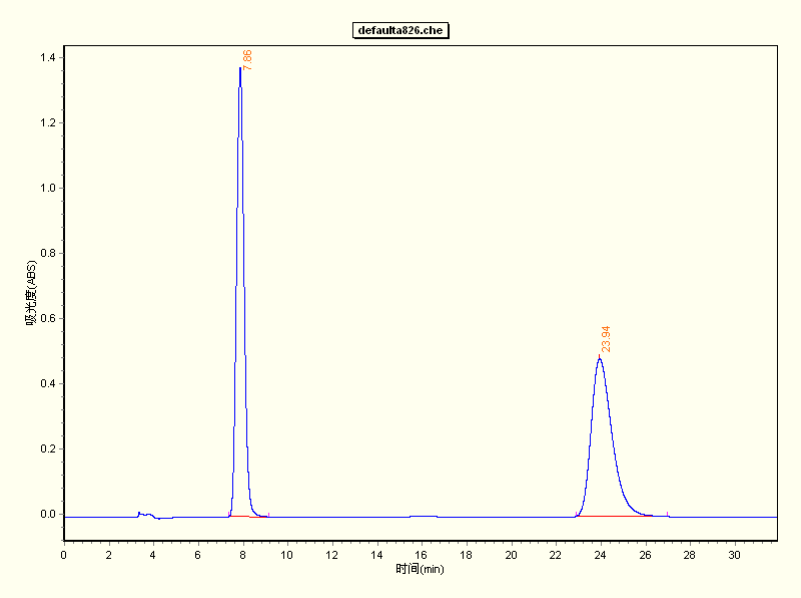
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.71	7921954	276397	49.89	1.483	BB
2	12.45	7958419	269596	50.11	2.350	BB



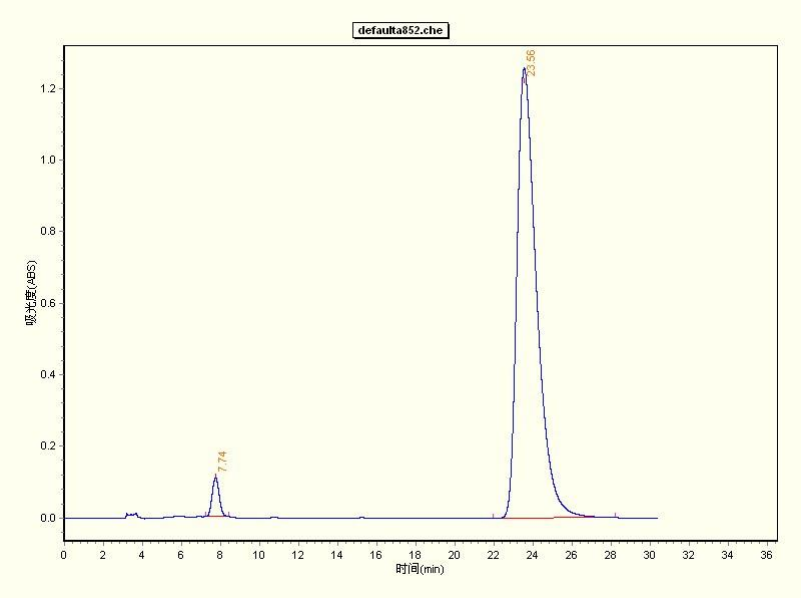
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.71	876573	45151	4.11	0.761	BB
2	12.31	20474346	656756	95.89	2.584	BB



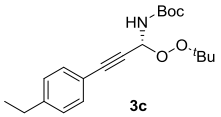
3b



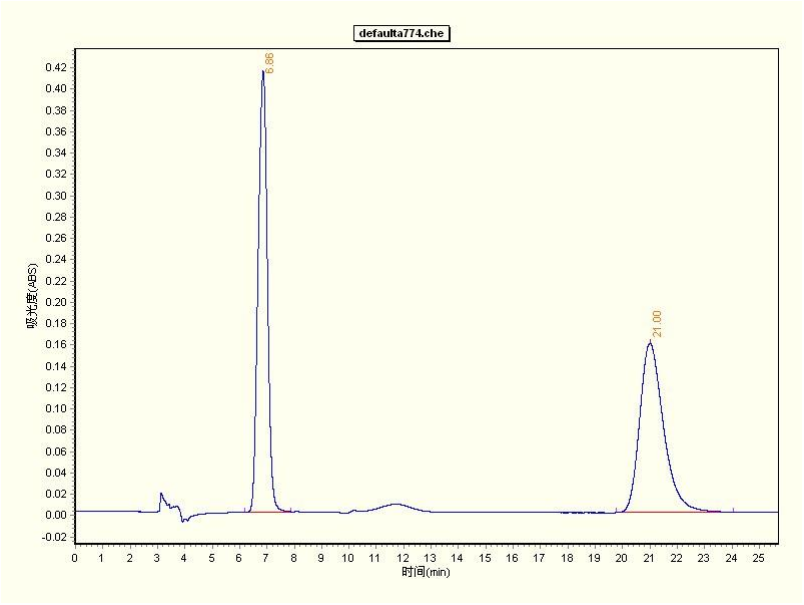
Entry	Retention time	Area	Height	Area%	Width	Type
1	7.86	16147332	686988	50.79	1.782	BB
2	23.94	15647870	241729	49.21	4.057	BB



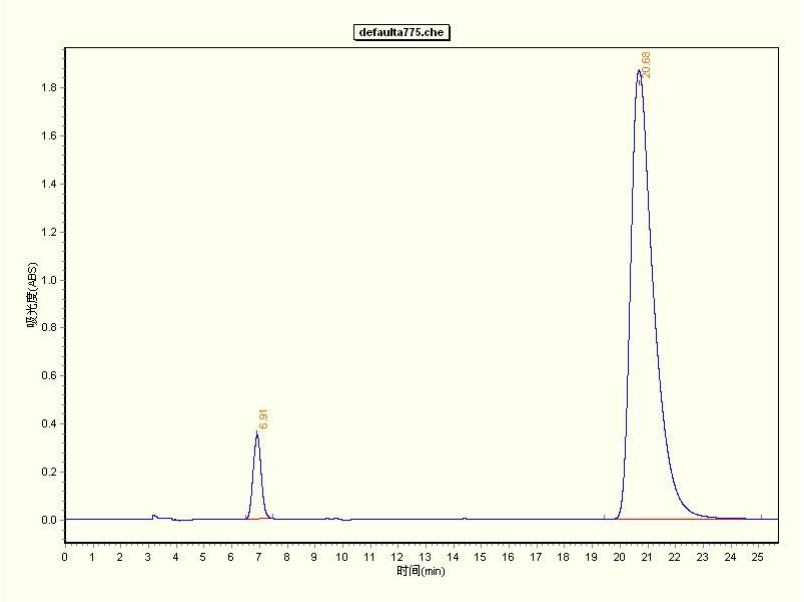
Entry	Retention time	Area	Height	Area%	Width	Type
1	7.74	1362506	54450	2.99	1.194	BB
2	23.56	4134927	628800	97.01	6.626	BB



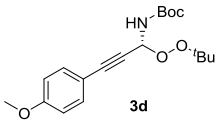
3c



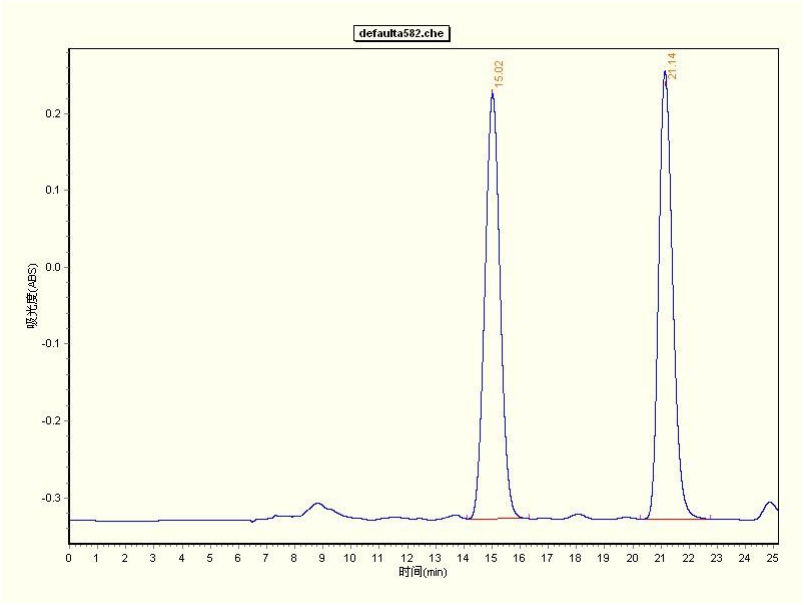
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.86	4875471	206846	50.35	1.667	BB
2	21.00	4808935	79333	49.65	4.292	BB



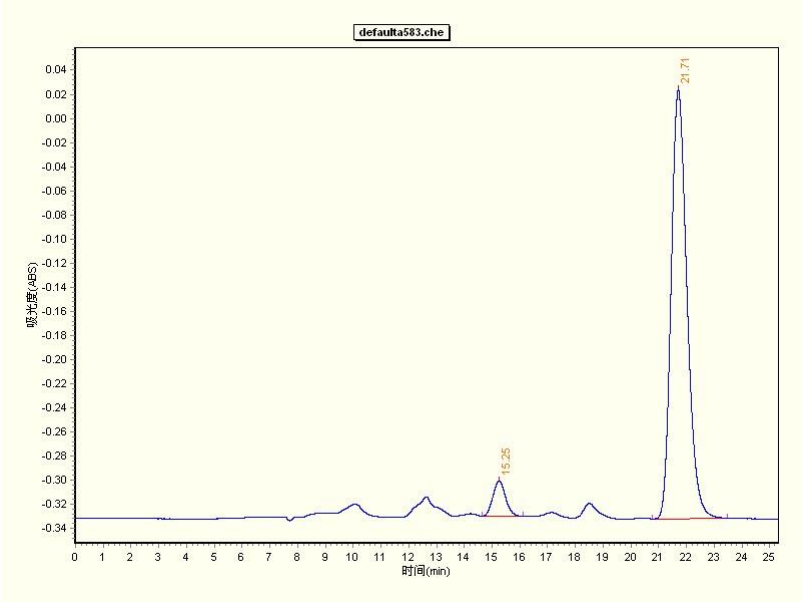
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.91	3507020	173828	6.10	0.974	BB
2	20.68	54013567	935743	93.90	5.688	BB



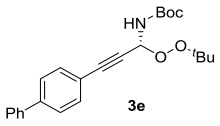
3d



Entry	Retention time	Area	Height	Area%	Width	Type
1	15.02	10390246	276483	50.83	2.199	BB
2	21.14	10052915	291278	49.17	2.488	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	15.25	455070	14558	6.08	1.46	BB
2	21.71	7027282	177627	93.92	2.707	BB



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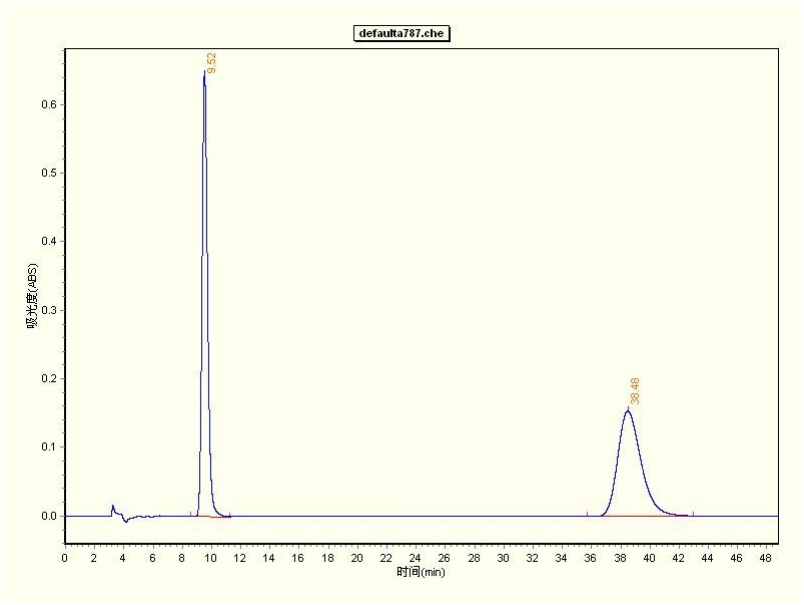
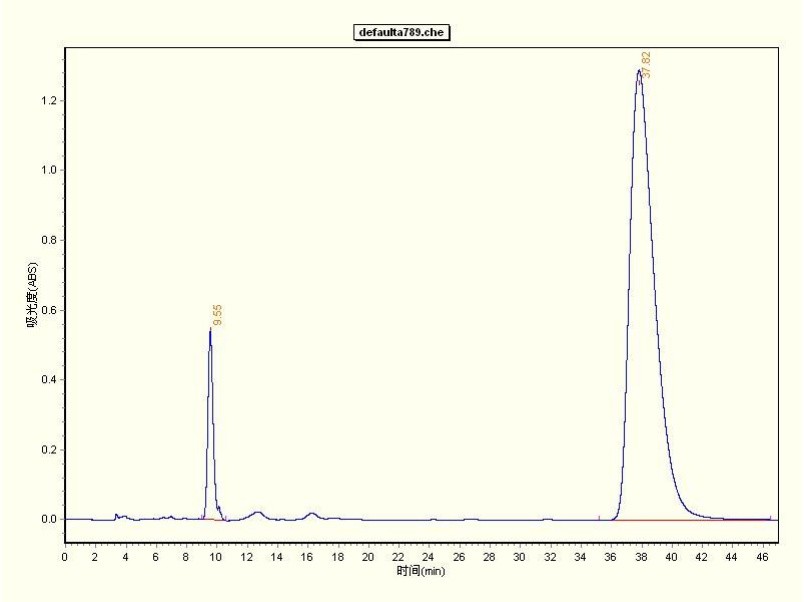
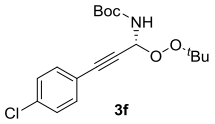


Entry	Retention time	Area	Height	Area%	Width	Type
1	9.52	8477123	324619	49.86	2.660	BB
2	38.48	8523634	76766	50.14	7.302	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	9.55	6693770	269642	8.30	1.565	BB
2	37.82	73943429	644134	91.70	11.293	BB



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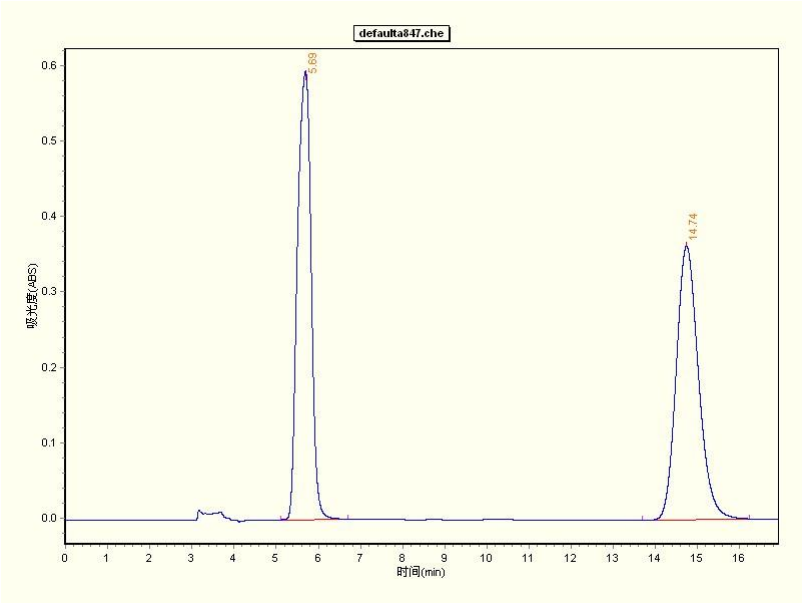
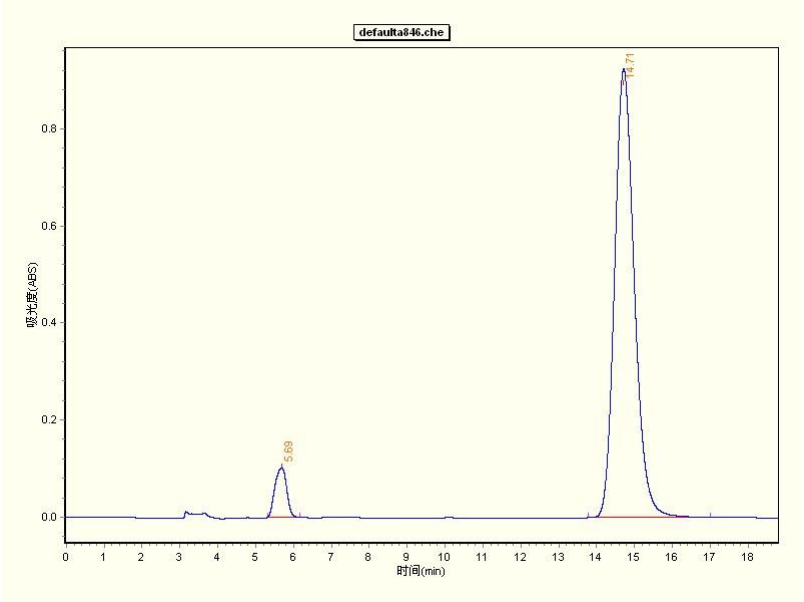
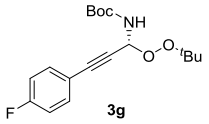


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.69	6671936	297092	50.13	1.589	BB
2	14.74	6638164	181380	49.87	2.535	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.69	1097137	51022	6.16	0.859	BB
2	14.71	16714001	462841	93.84	3.223	BB



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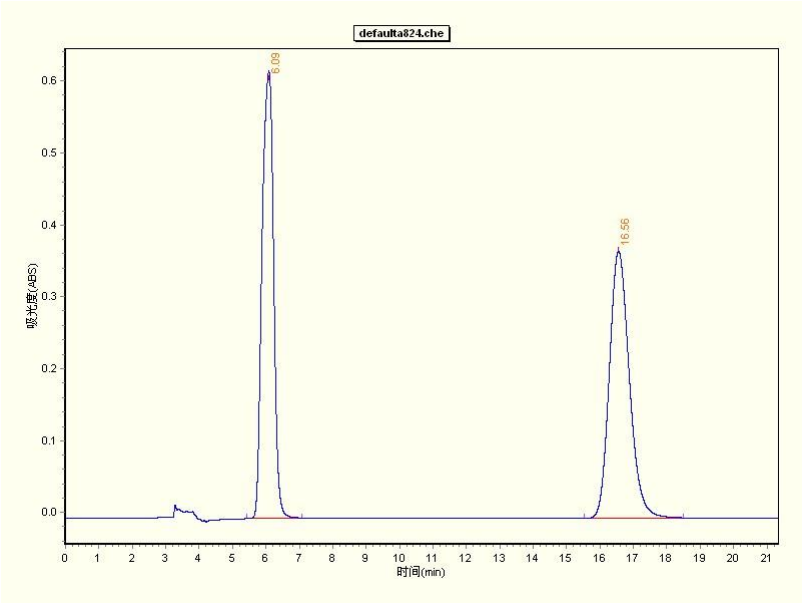
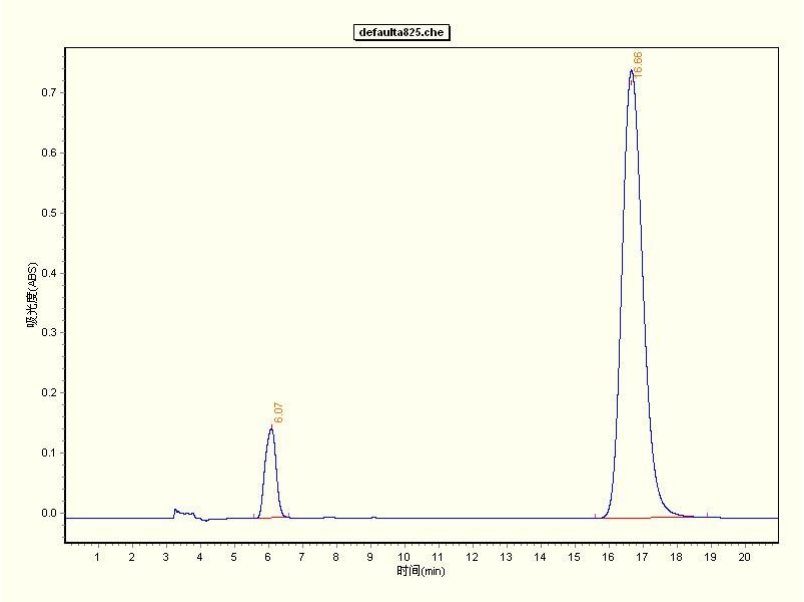
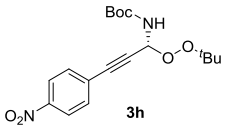


Entry	Retention time	Area	Height	Area%	Width	Type
1	6.09	7416039	310816	49.07	1.650	BB
2	16.56	7697130	185725	50.93	2.961	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	6.07	1664606	74411	9.45	1.038	BB
2	16.66	15947145	372948	90.55	3.323	BB



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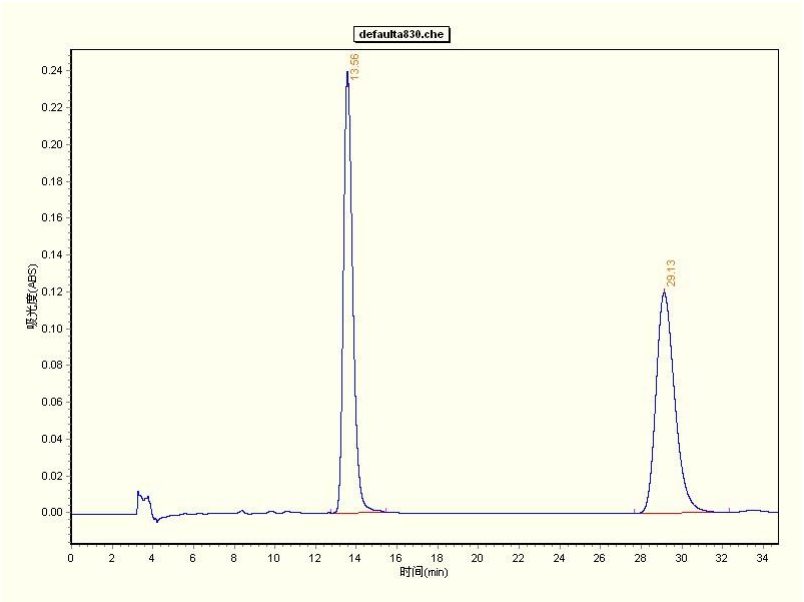
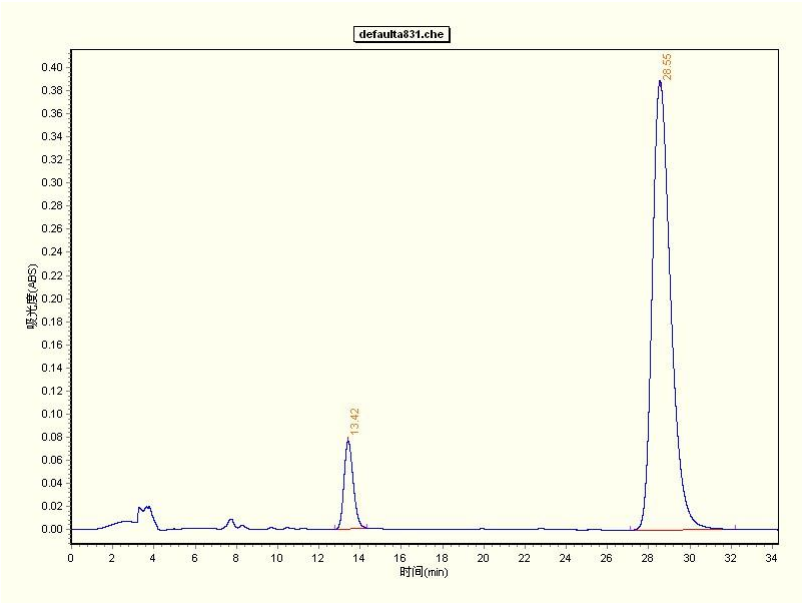
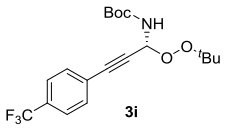


Entry	Retention time	Area	Height	Area%	Width	Type
1	13.56	3795529	119708	49.71	2.702	BB
2	29.13	3839309	59875	50.29	4.643	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	13.42	1179914	38176	8.92	1.579	BB
2	28.55	12046658	194568	91.08	5.116	BB



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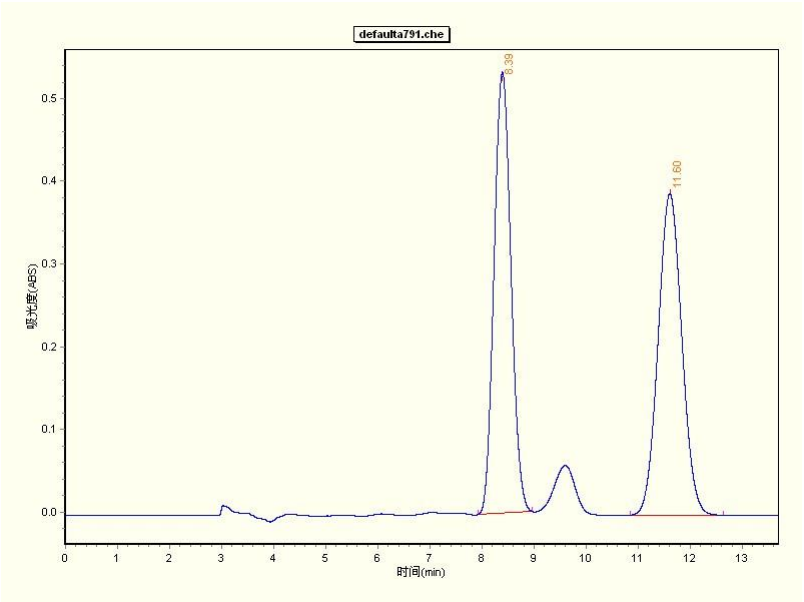
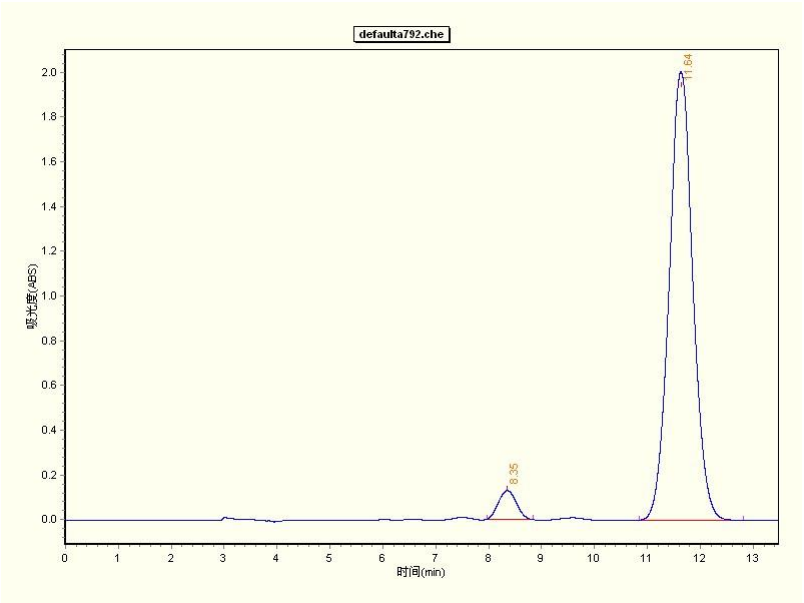
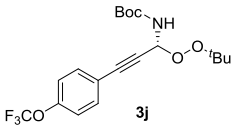


Entry	Retention time	Area	Height	Area%	Width	Type
1	8.39	6025940	266586	49.56	1.044	BB
2	11.60	6131945	194164	50.44	1.791	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.35	1527669	64434	4.68	0.885	BB
2	11.64	31138132	1002283	95.32	1.977	BB



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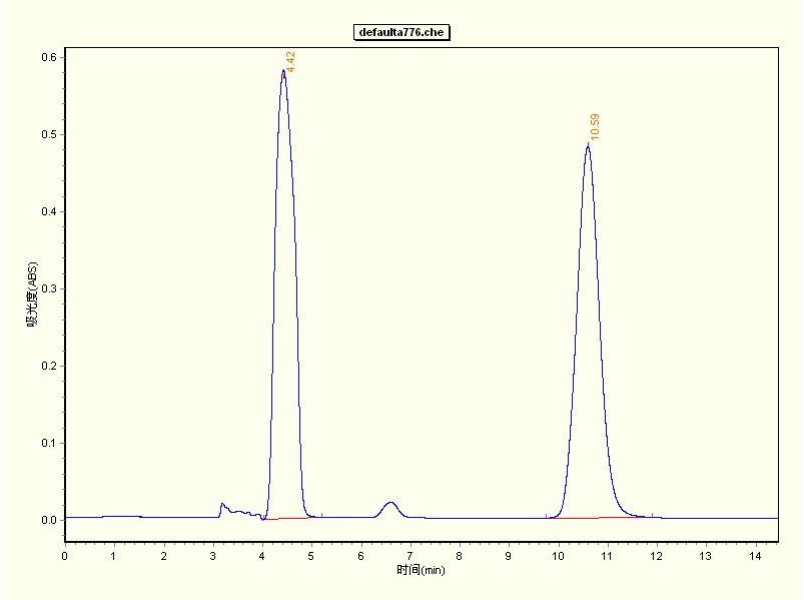
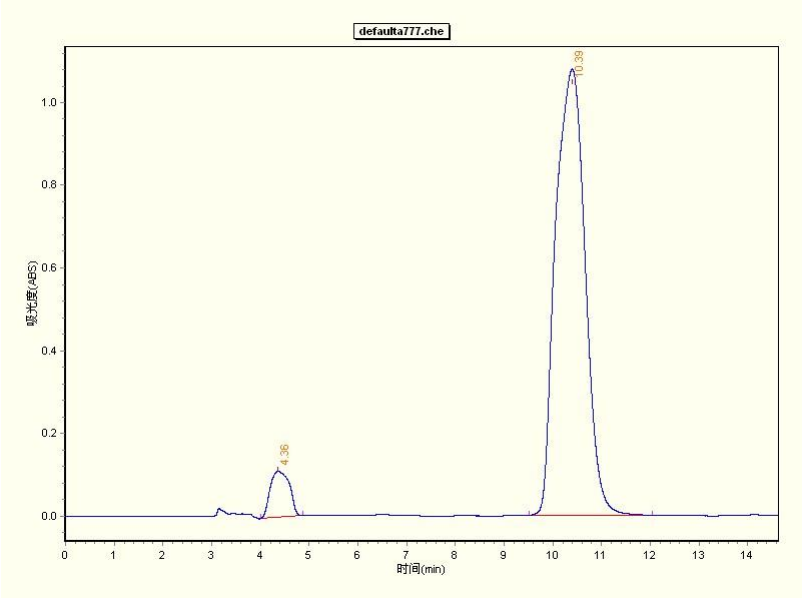
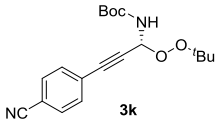


Entry	Retention time	Area	Height	Area%	Width	Type
1	4.42	7738112	291542	49.87	1.189	BB
2	10.59	7777113	241322	50.13	2.149	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	4.36	1516974	55760	6.26	0.856	BB
2	10.39	22722310	539337	93.74	2.537	BB



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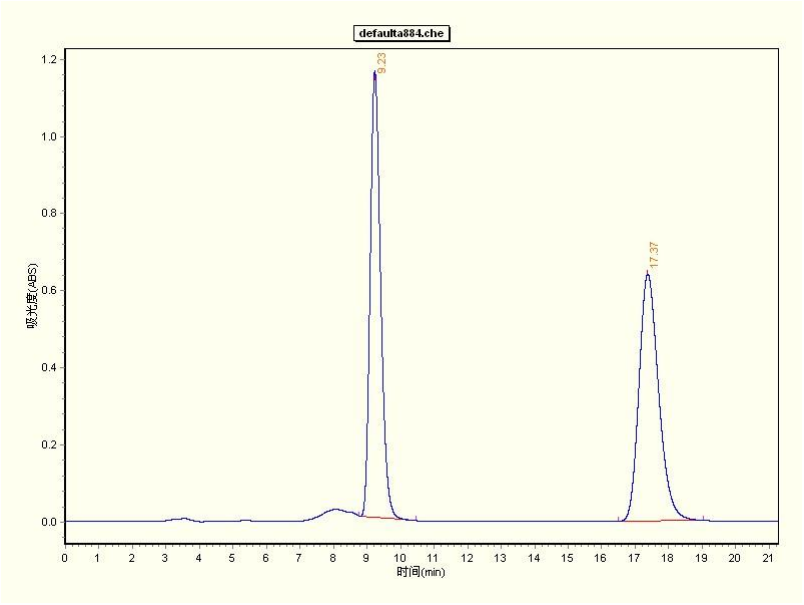
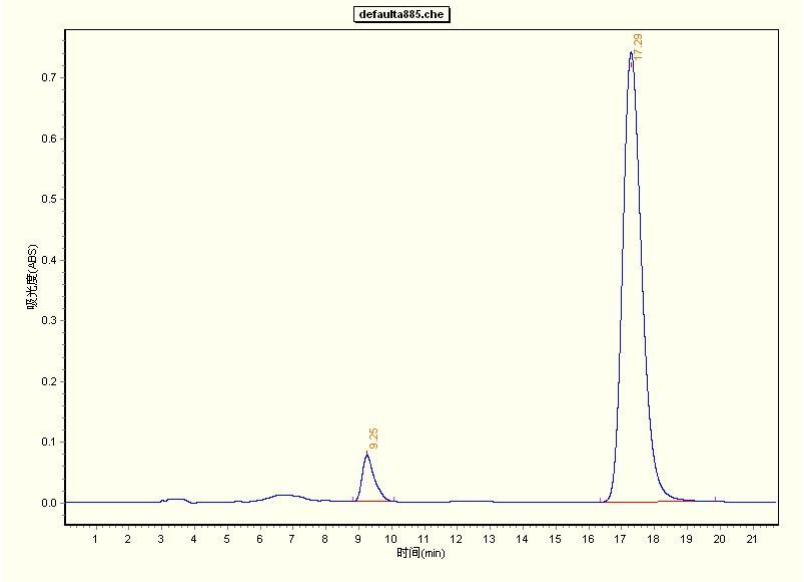
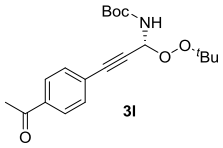


Entry	Retention time	Area	Height	Area%	Width	Type
1	9.23	12532025	577998	49.03	1.695	BB
2	17.37	13029540	320149	50.97	2.530	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	9.25	993971	38109	6.20	1.248	BB
2	17.29	15049998	370154	93.80	3.508	BB



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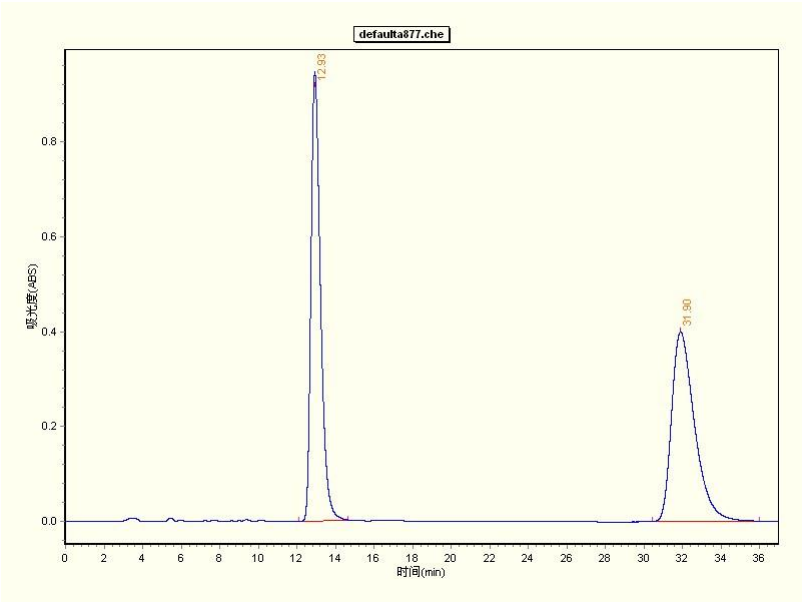
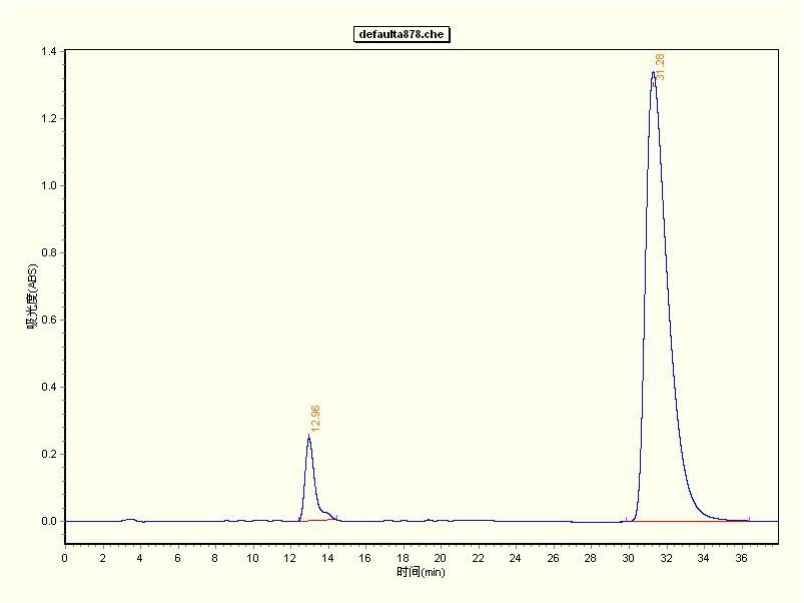
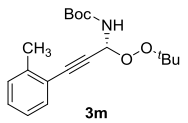


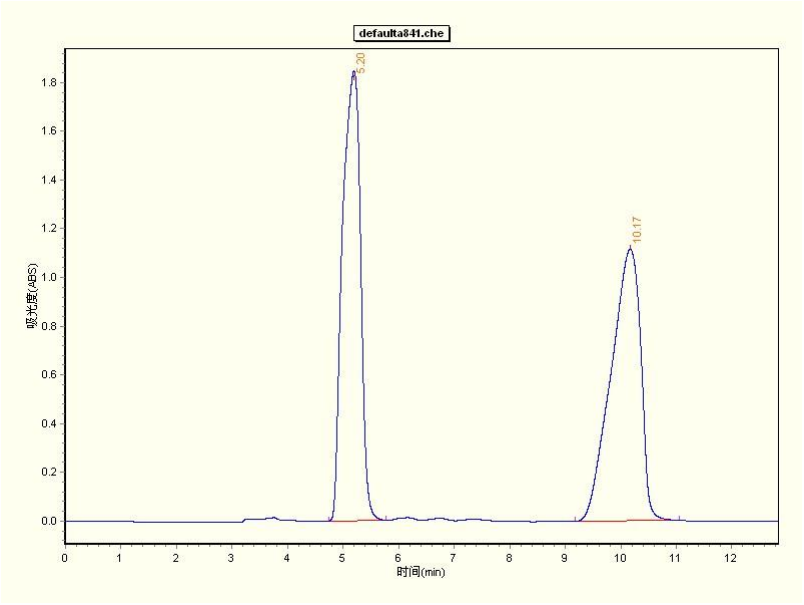
Entry	Retention time	Area	Height	Area%	Width	Type
1	12.93	16216567	473300	49.46	2.566	BB
2	31.90	16568516	199958	50.54	5.571	BB



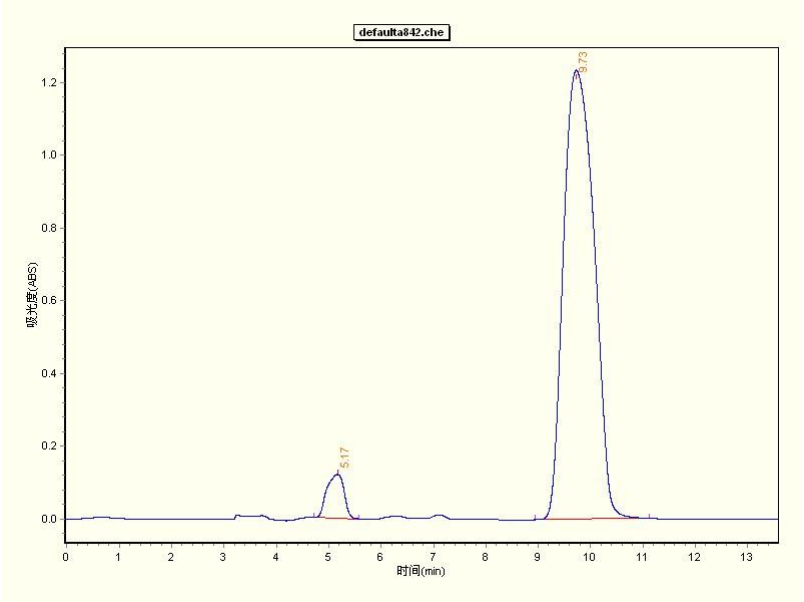
Entry	Retention time	Area	Height	Area%	Width	Type
1	12.96	4488181	122824	7.45	2.020	BB
2	31.28	55790503	670010	92.55	6.543	BB



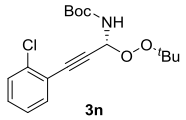
3m



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.20	20815849	922013	50.07	1.030	BB
2	10.17	20760499	557557	49.93	1.870	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.17	1347345	59654	5.10	0.850	BB
2	9.73	25070532	616917	94.90	2.173	BB



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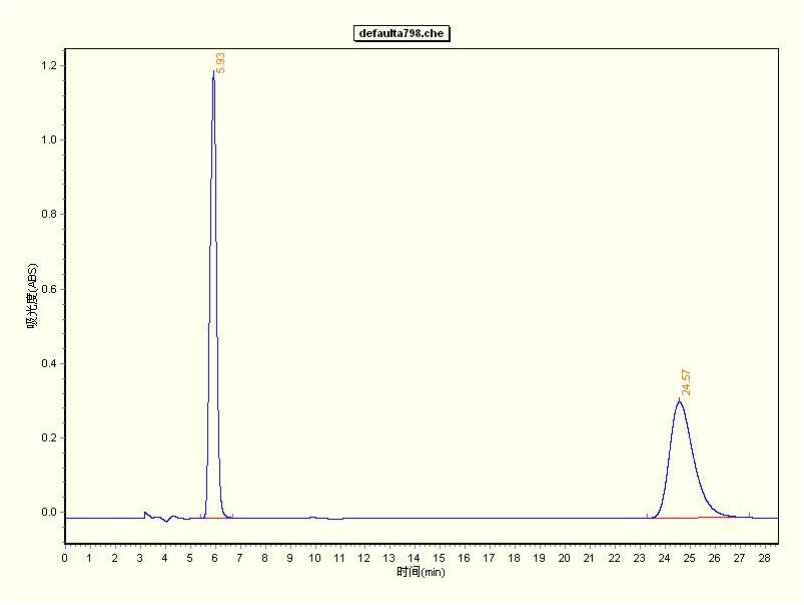
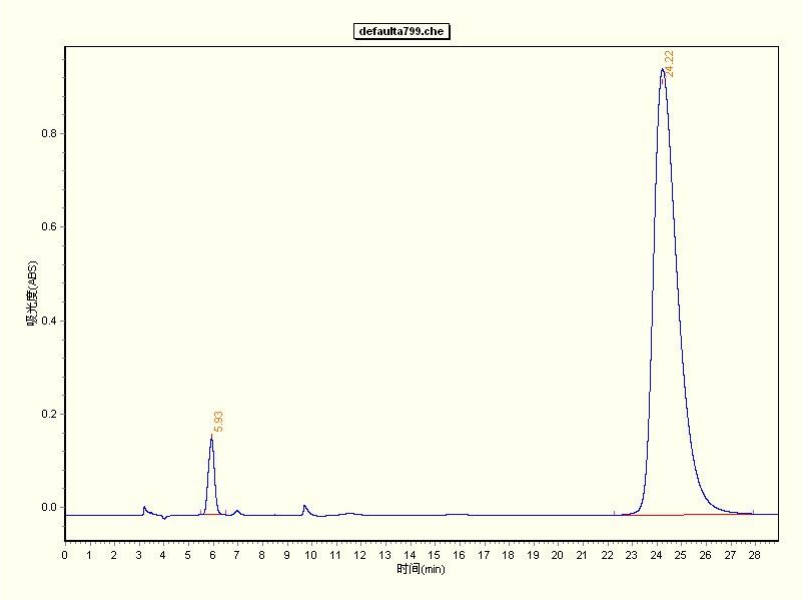
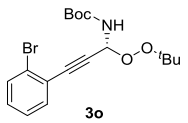


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.93	10820196	600121	50.66	1.290	BB
2	24.57	10536706	156180	49.34	4.073	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.93	1408840	81295	4.15	1.032	BB
2	24.22	32563273	476403	95.85	5.642	BB



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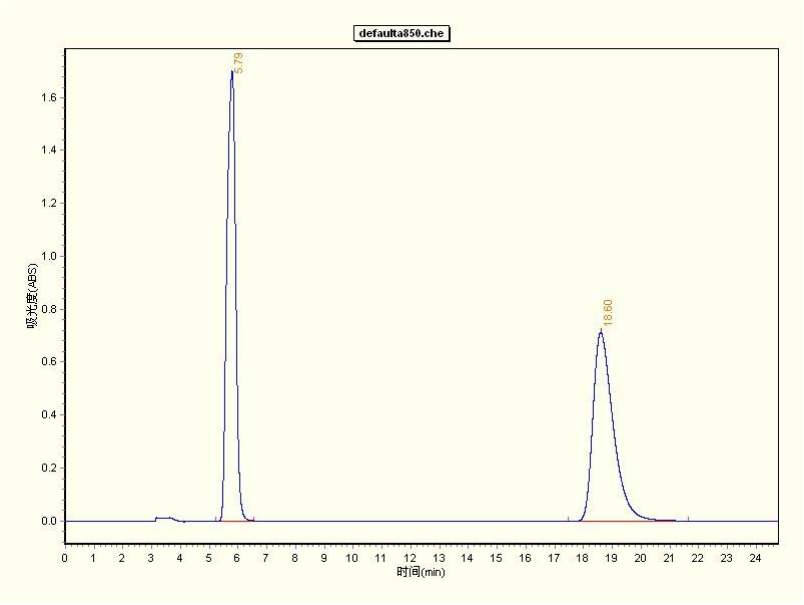
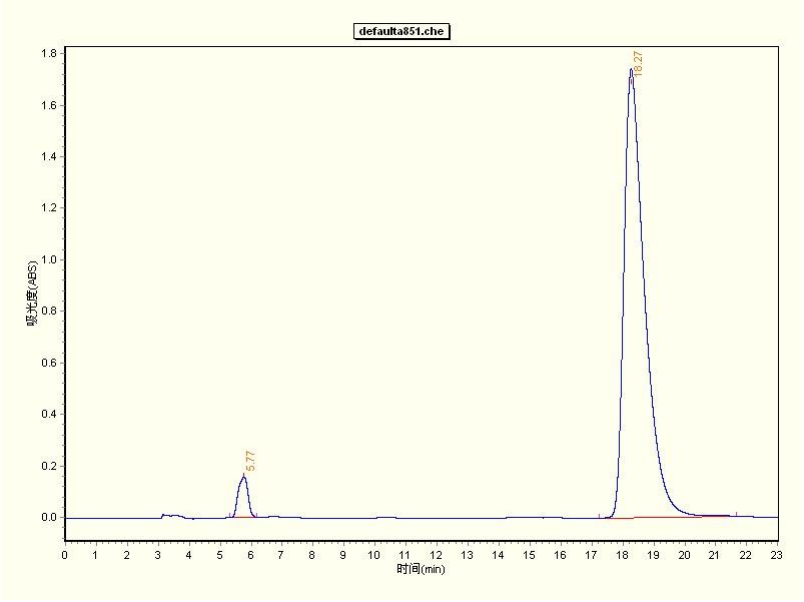
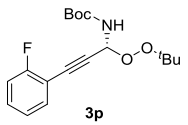


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.79	18000276	850218	50.67	1.351	BB
2	18.60	17522051	356892	49.33	4.149	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.77	1706660	77881	4.05	0.844	BB
2	18.27	40393317	870572	95.95	4.427	BB



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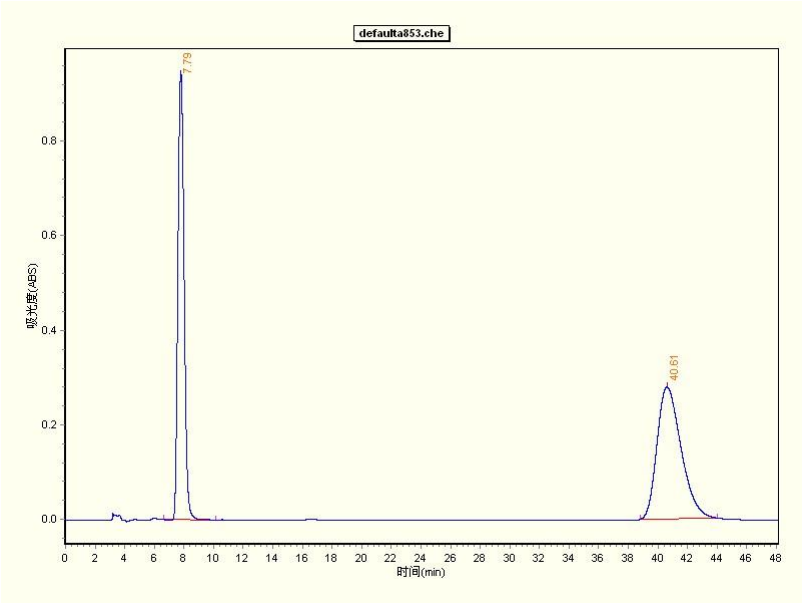
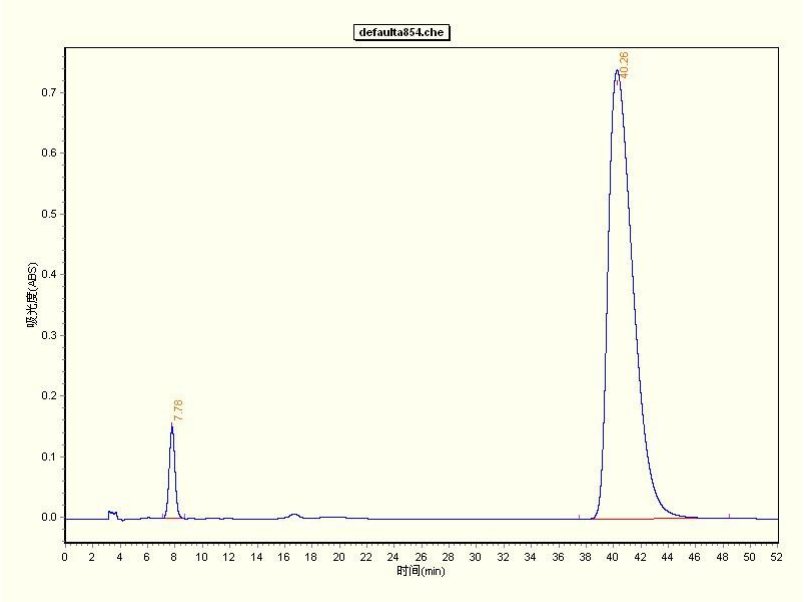
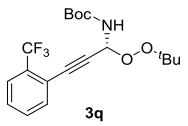


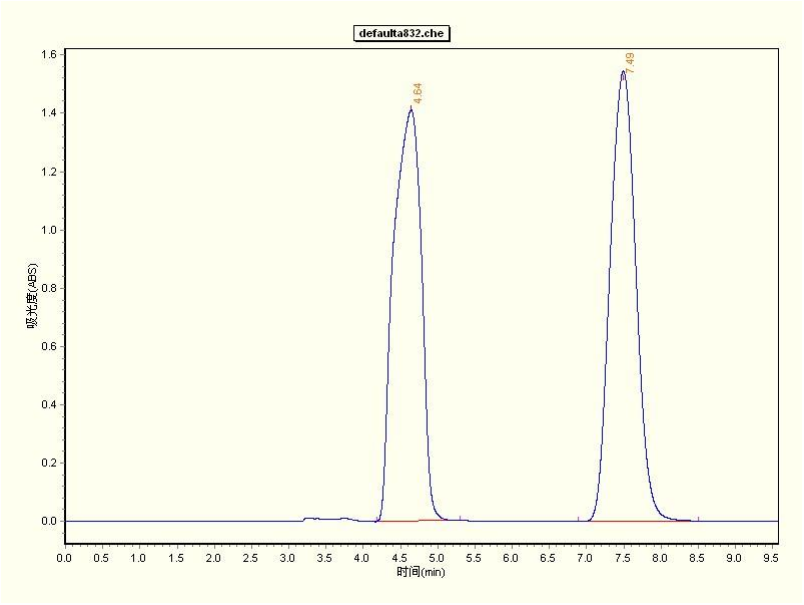
Entry	Retention time	Area	Height	Area%	Width	Type
1	7.79	13709906	474291	46.47	3.531	BB
2	40.61	15792885	139515	53.53	5.248	BB



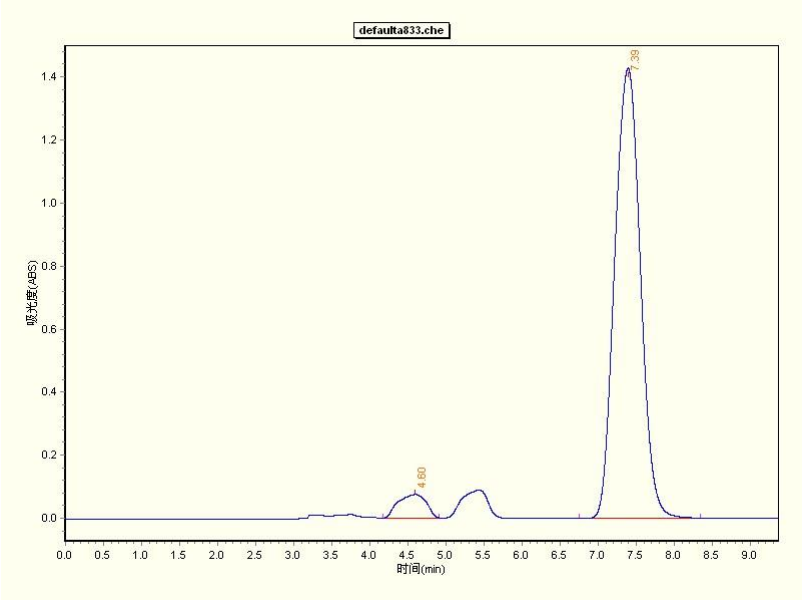
Entry	Retention time	Area	Height	Area%	Width	Type
1	7.78	2213968	75553	4.53	4.548	BB
2	40.26	46635735	369756	95.47	10.934	BB



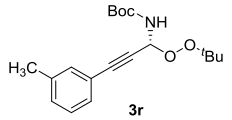
3q



Entry	Retention time	Area	Height	Area%	Width	Type
1	4.64	18202619	705163	49.52	1.116	BB
2	7.49	18556755	772074	50.48	1.606	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	4.60	952693	37085	5.41	0.747	BB
2	7.39	16660627	713359	94.59	1.593	BB



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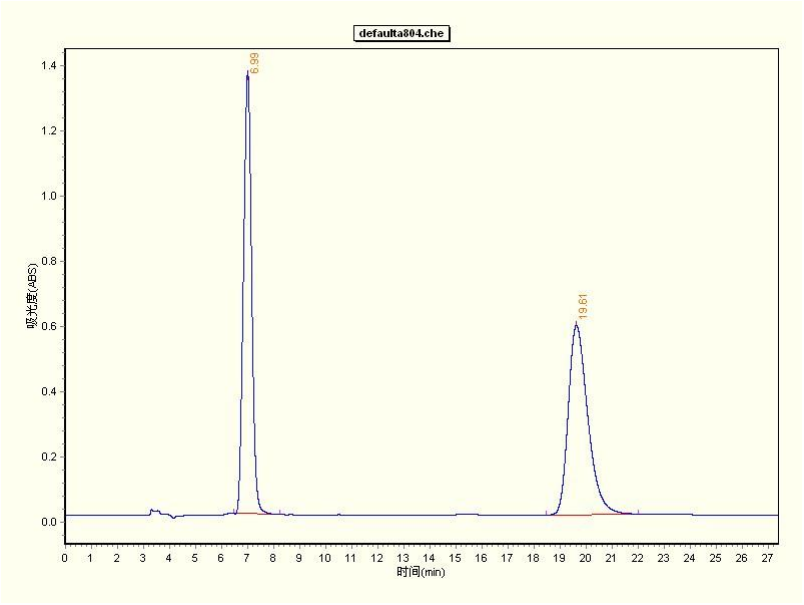
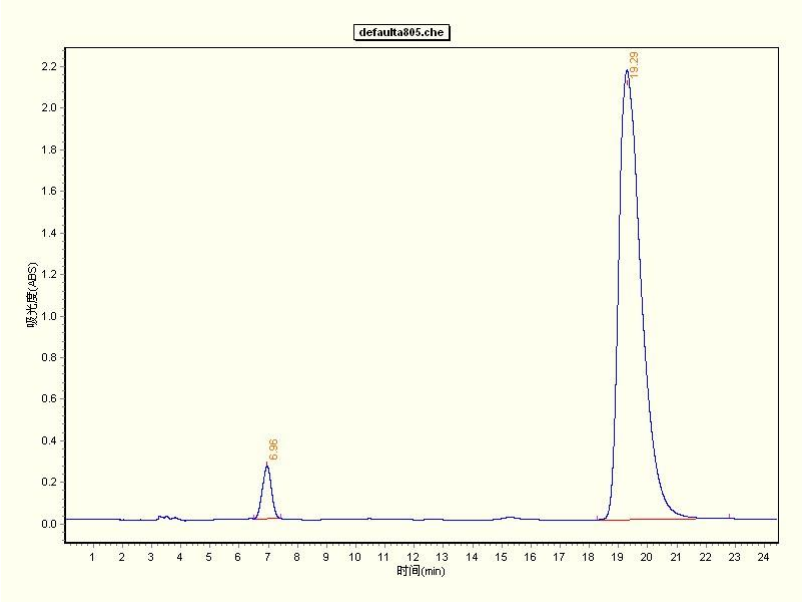
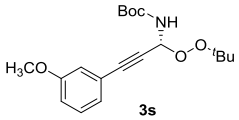


Entry	Retention time	Area	Height	Area%	Width	Type
1	6.99	14771902	678919	49.41	1.763	BB
2	19.61	15124083	290460	50.59	3.553	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	6.96	2722215	126246	4.48	0.897	BB
2	19.29	58099962	1079898	95.52	4.553	BB



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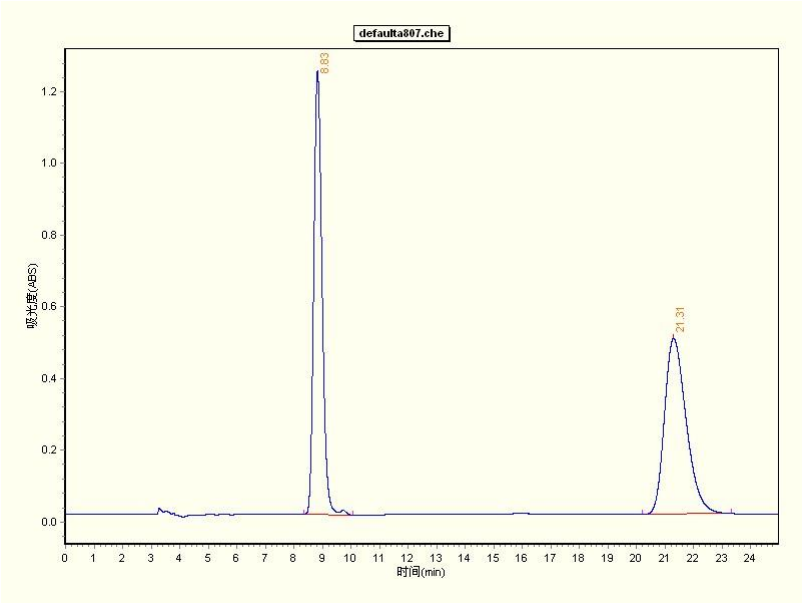
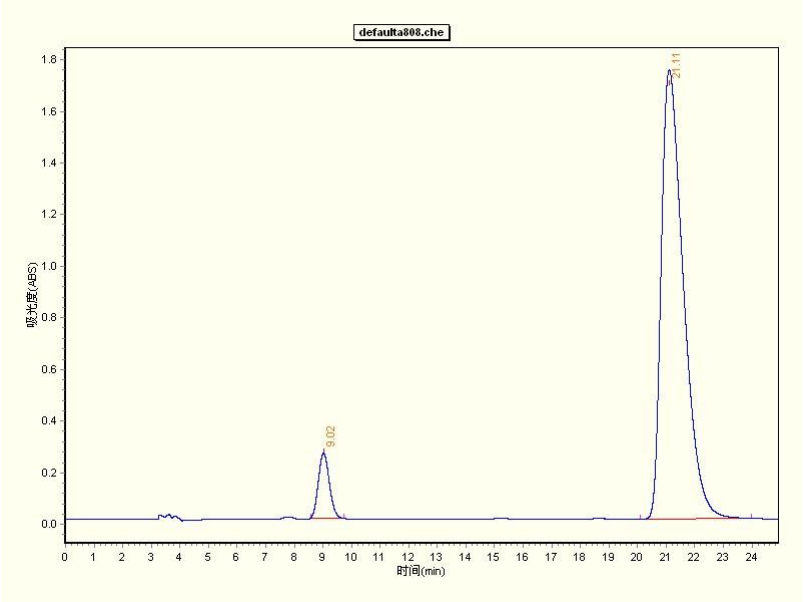
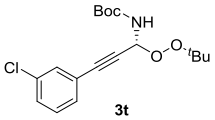


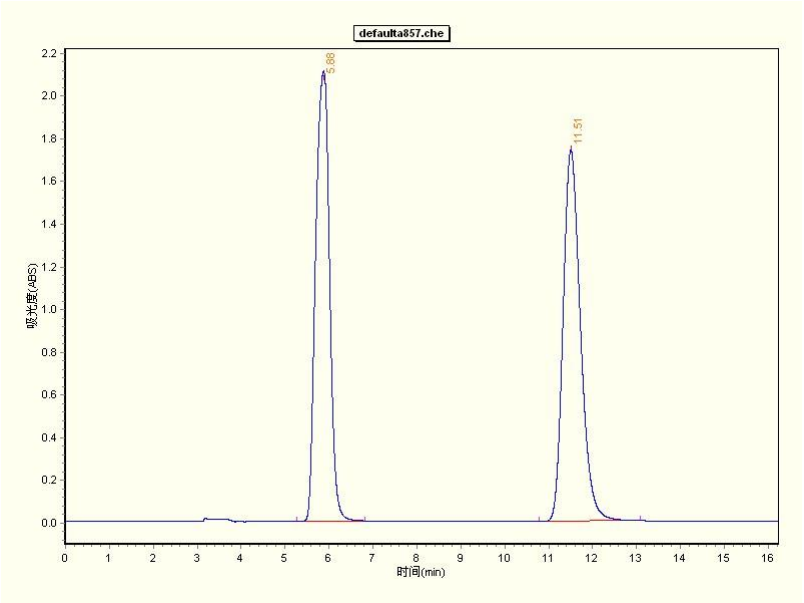
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.83	12552145	618835	49.12	1.708	BB
2	21.31	13002713	245436	50.88	3.119	BB



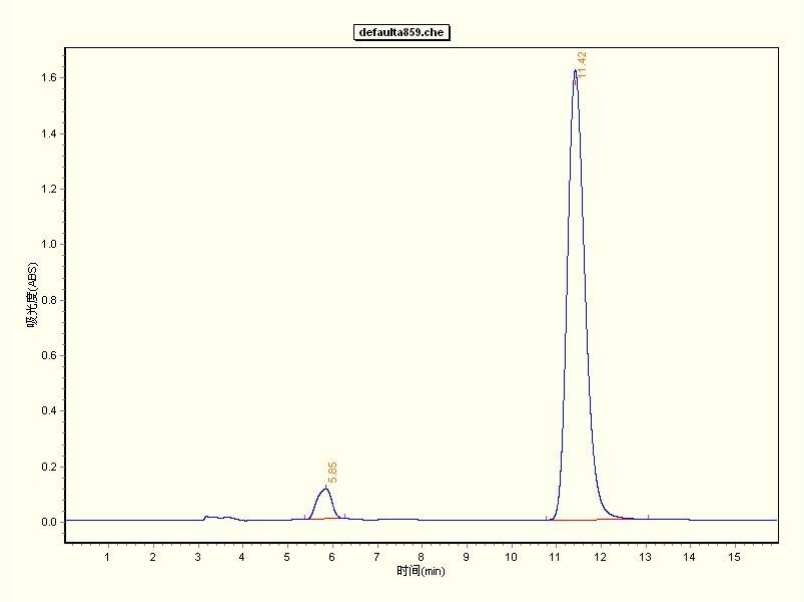
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.02	3351692	126183	6.69	1.162	BB
2	21.11	46772600	869324	93.31	3.878	BB



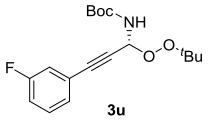
3t



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.88	23040545	1053499	49.10	1.528	BB
2	11.51	23880639	869649	50.90	2.316	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.85	1247563	54033	5.33	0.891	BB
2	11.42	22157128	809603	94.67	2.292	BB



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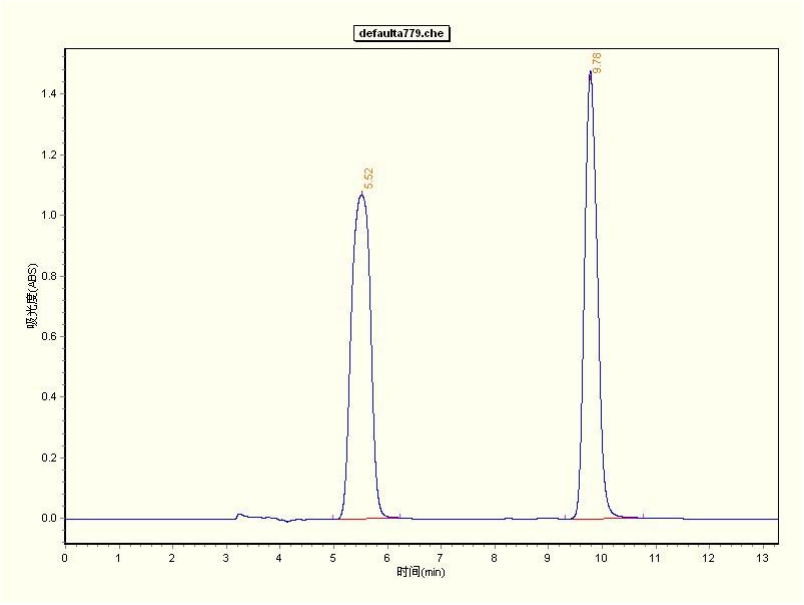
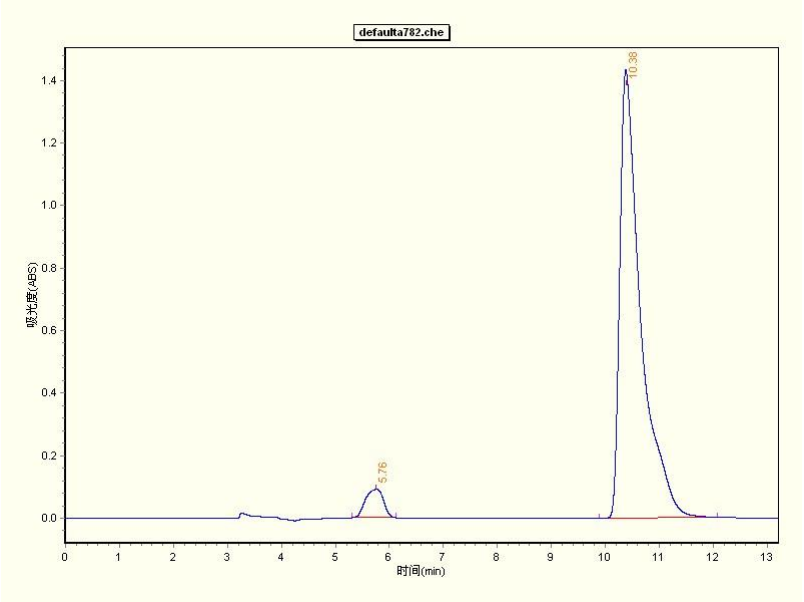
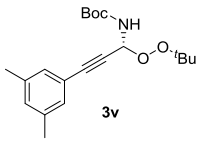


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.52	13136234	534868	51.90	1.264	BB
2	9.78	12172193	738658	48.10	1.462	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.76	1045119	45303	5.17	0.798	BB
2	10.38	19170543	716611	94.83	2.173	BB



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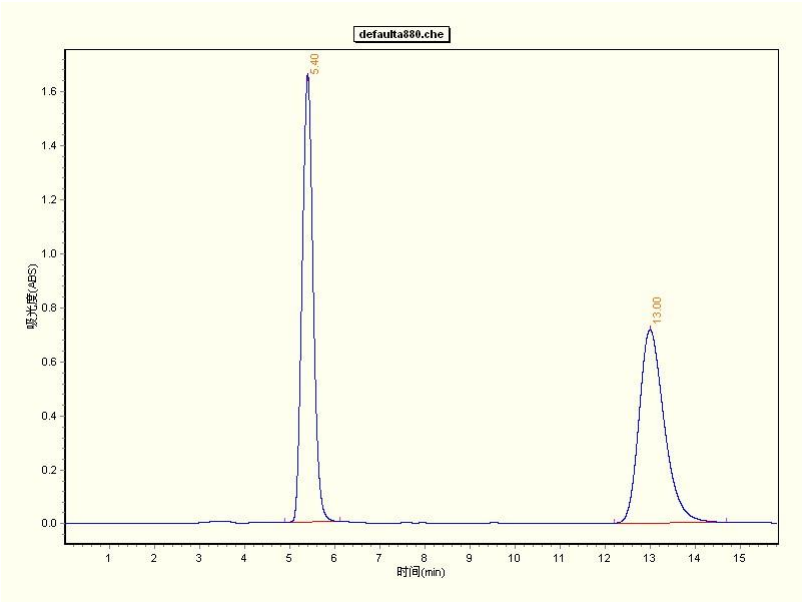
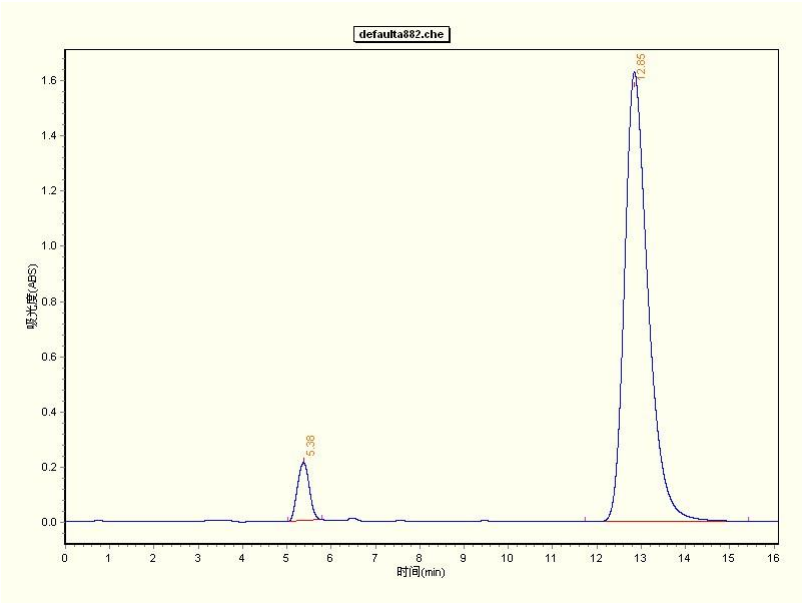
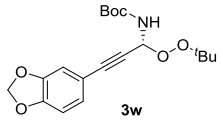


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.40	14054940	829681	50.37	1.224	BB
2	13.00	13849656	357561	49.63	2.495	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.38	1958669	105143	6.13	0.782	BB
2	12.85	30001825	813765	93.87	3.702	BB



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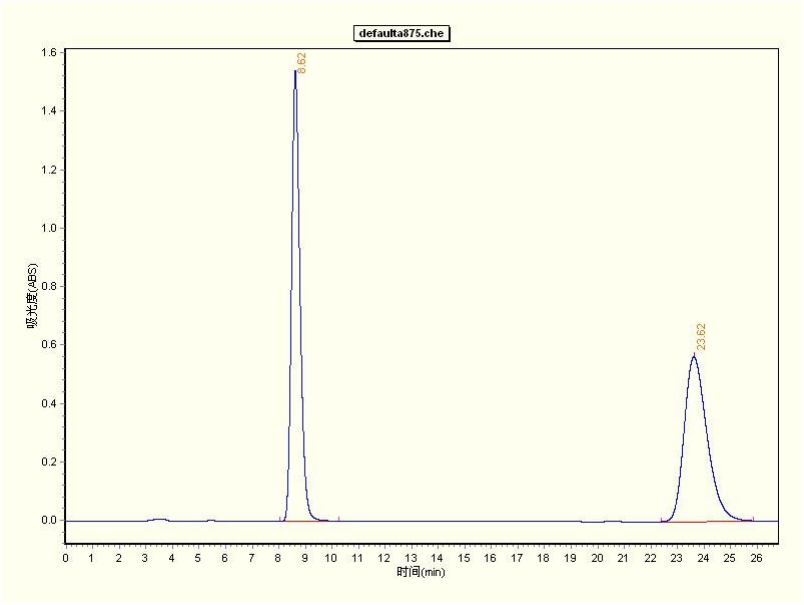
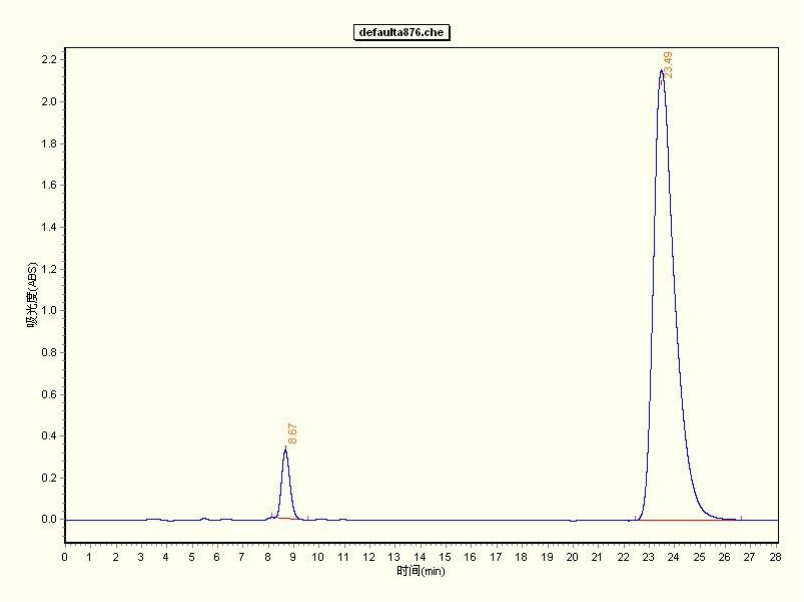
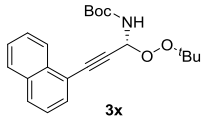


Entry	Retention time	Area	Height	Area%	Width	Type
1	8.62	16990933	769520	50.28	2.223	BB
2	23.62	16800591	281491	49.72	3.483	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.67	3704873	163850	5.55	1.420	BB
2	23.49	63098099	1077646	94.45	4.177	BB



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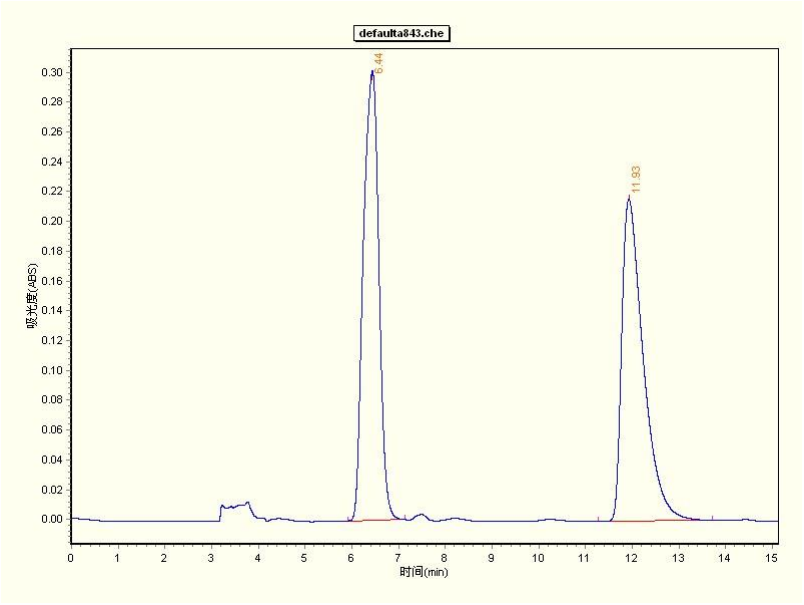
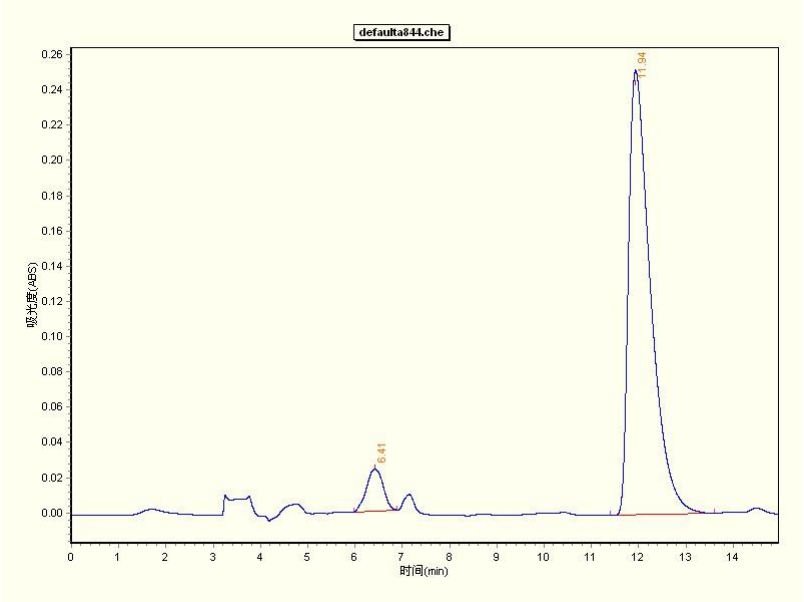
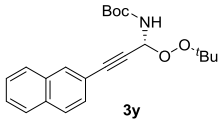


Entry	Retention time	Area	Height	Area%	Width	Type
1	6.44	3477413	150652	50.10	1.213	BB
2	11.93	3463812	108100	49.90	2.444	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	6.41	289685	11842	6.62	0.910	BB
2	11.94	4085960	126238	93.38	2.192	BB



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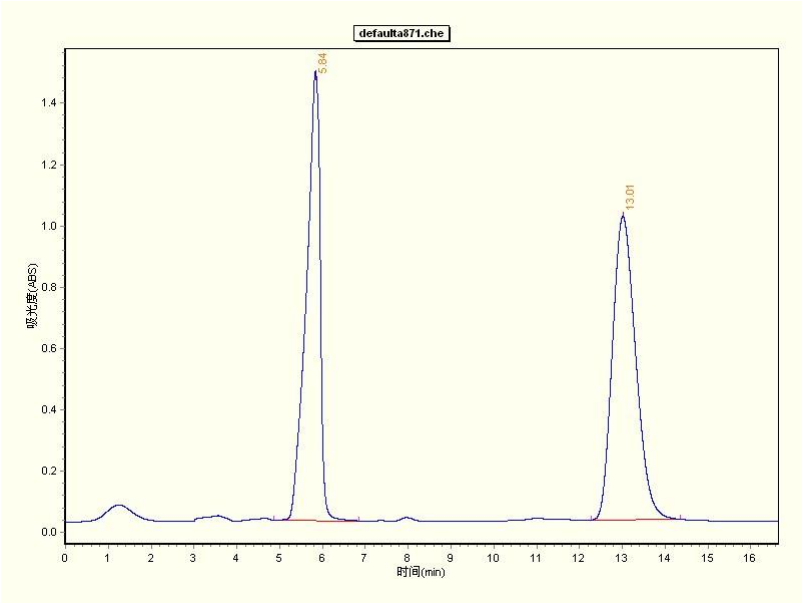
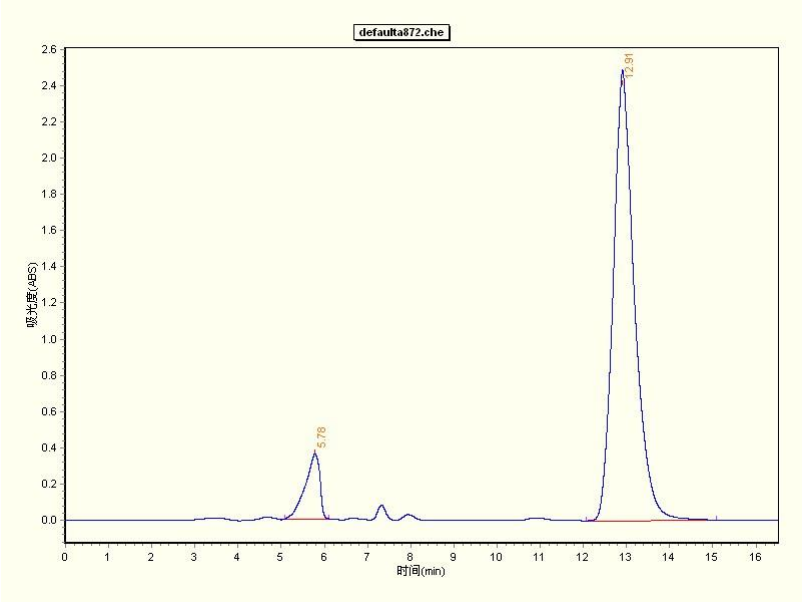
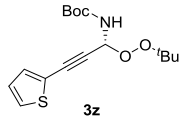


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.84	16554146	733430	46.96	1.979	BB
2	13.01	18700321	495006	53.04	2.078	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.78	4194206	178346	9.01	1.022	BB
2	12.91	42366344	1242389	90.99	3.026	BB



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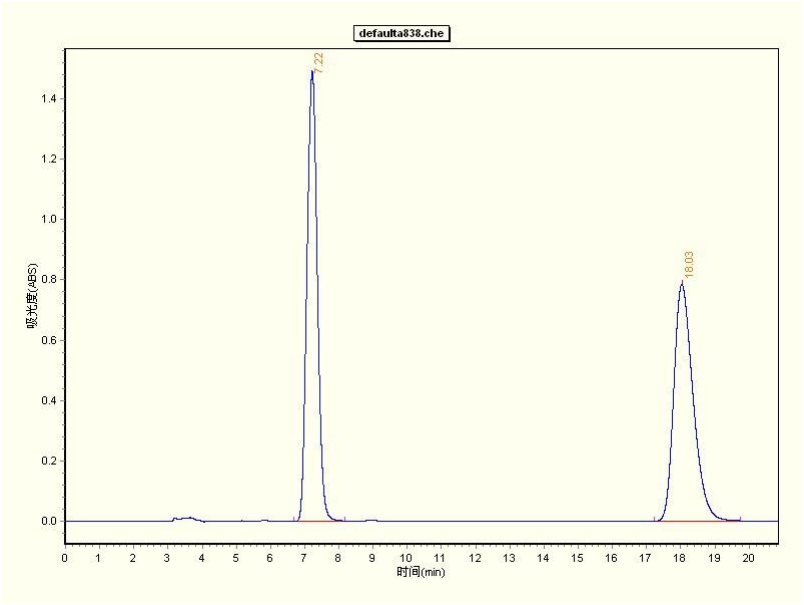
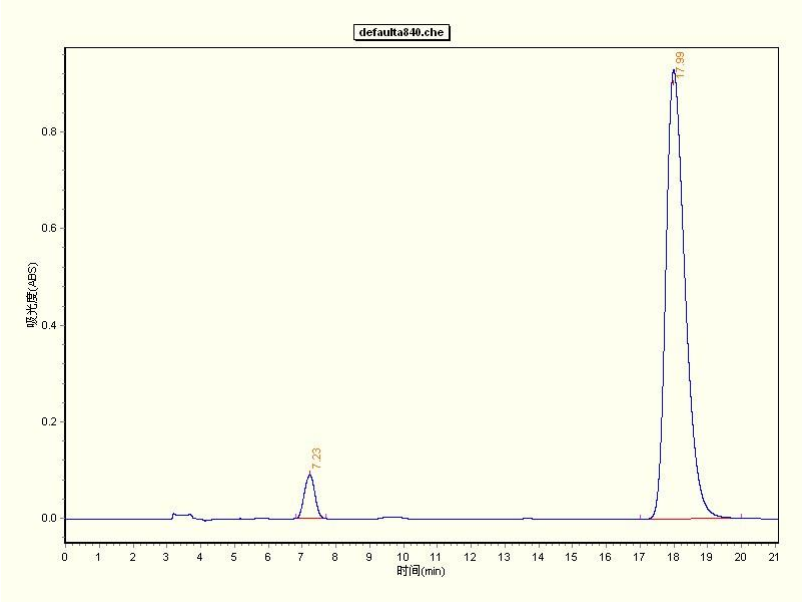
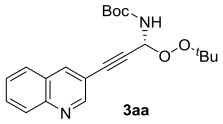


Entry	Retention time	Area	Height	Area%	Width	Type
1	7.22	15733076	745610	50.60	1.488	BB
2	18.03	15359638	392742	49.40	2.523	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	7.23	969994	45120	5.04	0.878	BB
2	17.99	18268271	464228	94.96	2.989	BB



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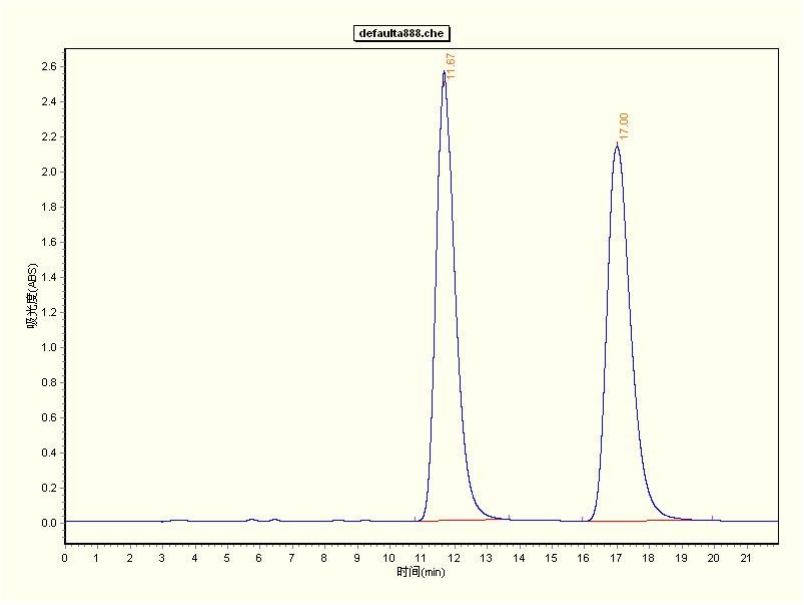
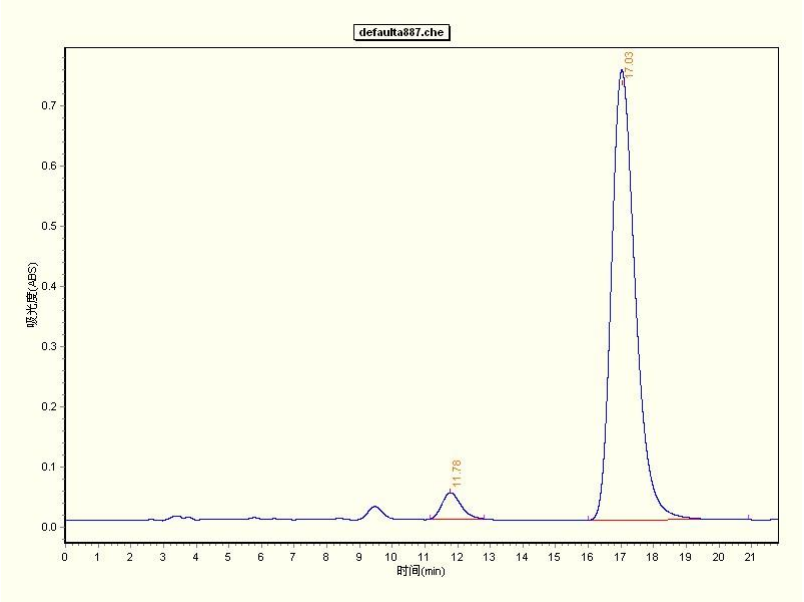
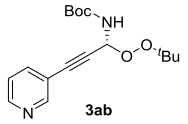


Entry	Retention time	Area	Height	Area%	Width	Type
1	11.67	52729123	1278594	49.04	2.896	BB
2	17.00	54794459	1065647	50.96	4.007	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	11.78	881513	21861	4.45	1.666	BB
2	17.03	18933290	373695	95.55	4.888	BB



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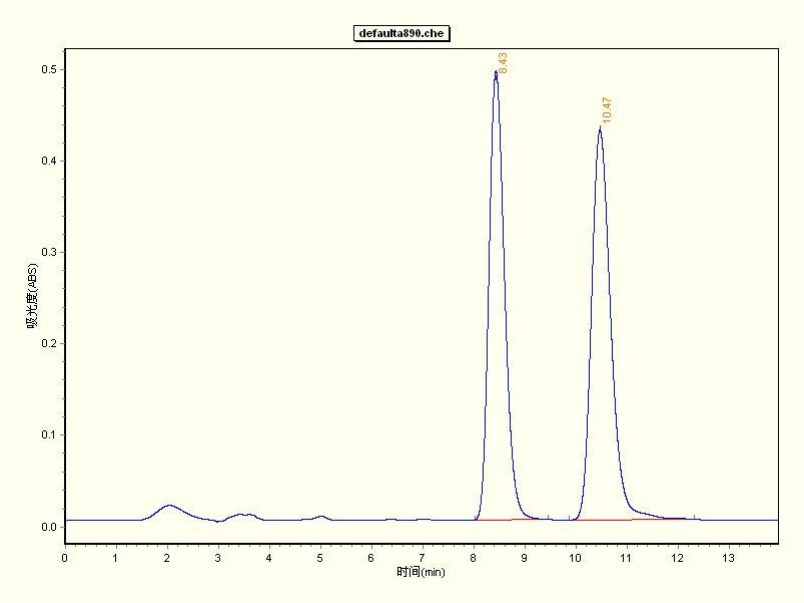
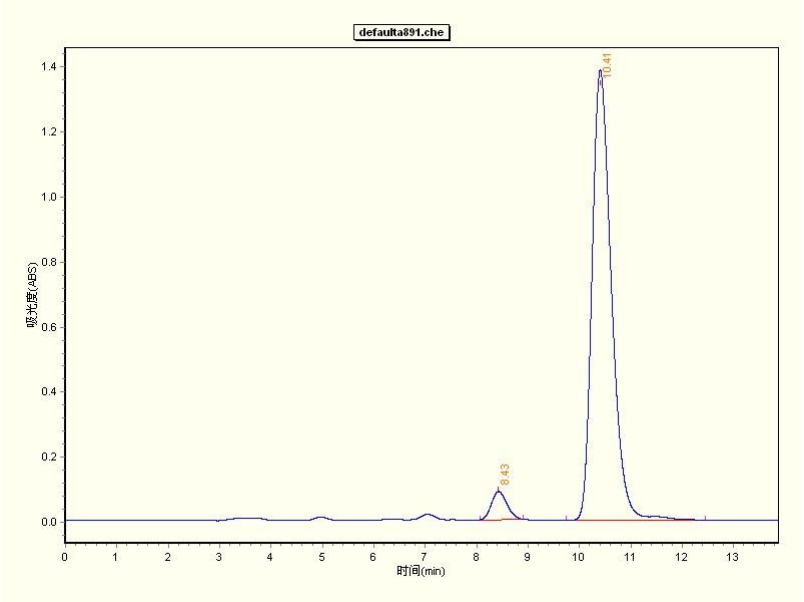
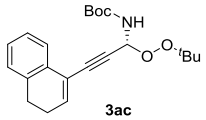


Entry	Retention time	Area	Height	Area%	Width	Type
1	8.43	5204531	245289	48.18	1.439	BB
2	10.47	5597415	213006	51.82	2.449	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.43	897173	43169	4.74	0.838	BB
2	10.41	18040978	694496	95.26	2.709	BB



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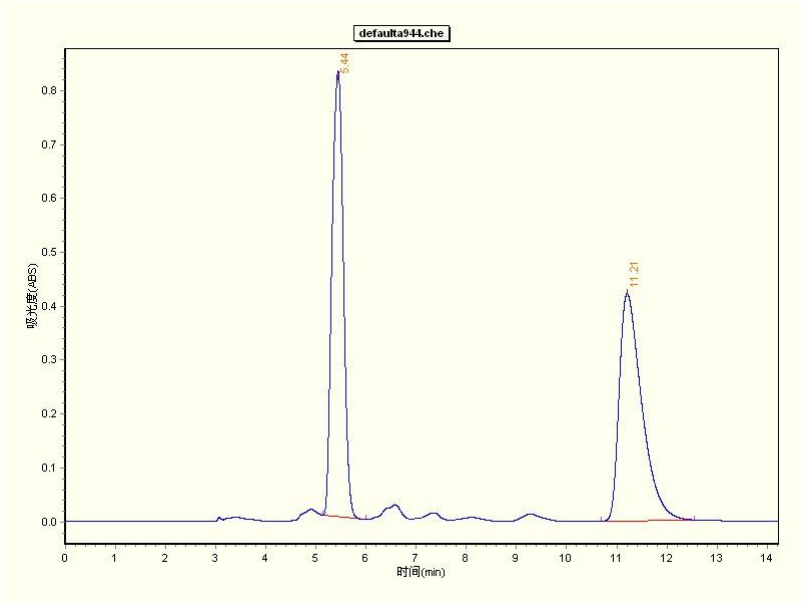
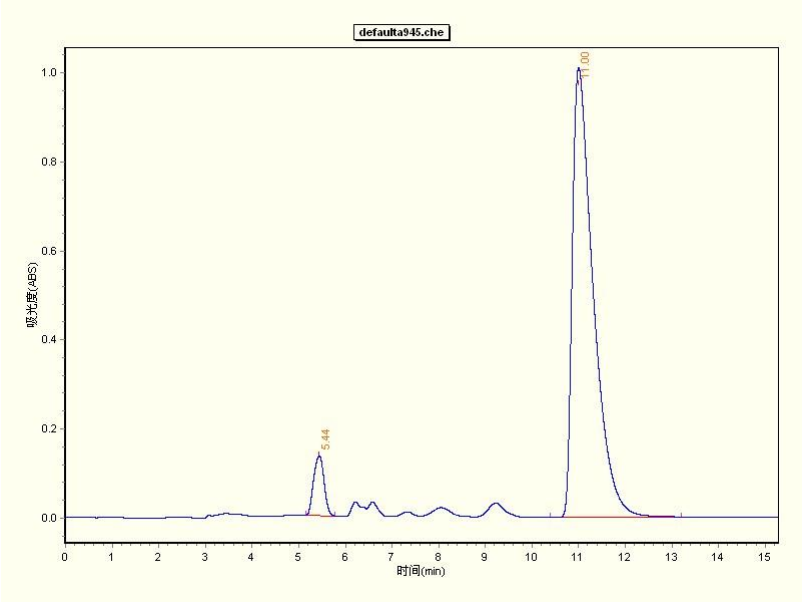
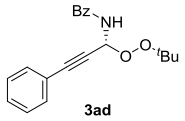


Entry	Retention time	Area	Height	Area%	Width	Type
1	5.44	6528822	413562	49.69	0.860	BB
2	11.21	6611568	211030	50.31	1.862	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	5.44	1043292	67259	6.21	0.621	BB
2	11.00	15766441	506100	93.79	2.803	BB



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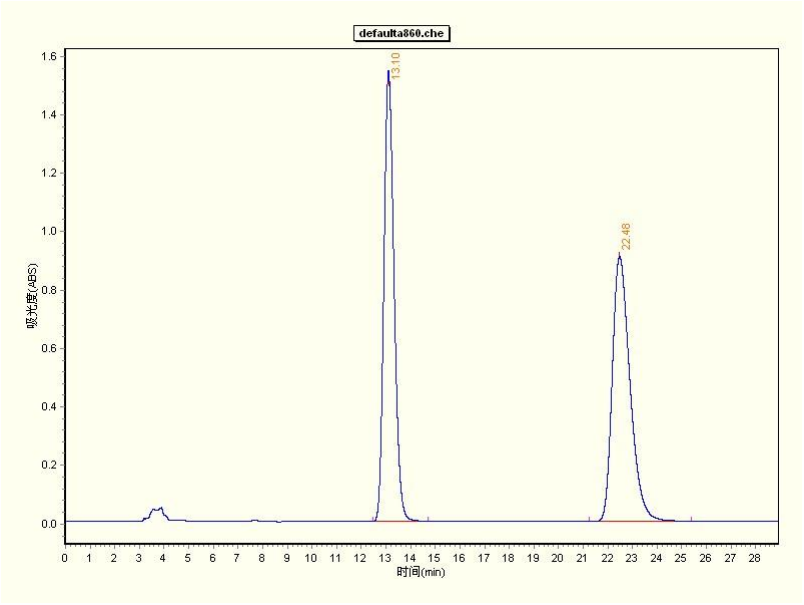
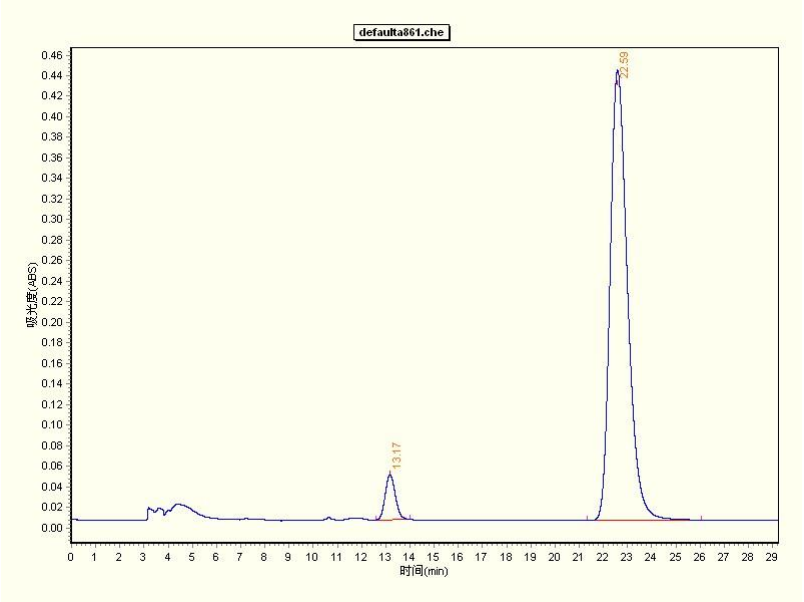
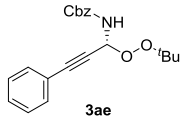


Entry	Retention time	Area	Height	Area%	Width	Type
1	13.10	21998816	770133	49.35	2.236	BB
2	22.48	22578704	453168	50.65	4.156	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	13.17	632344	21830	5.46	1.399	BB
2	22.59	10950558	219050	94.54	4.752	BB



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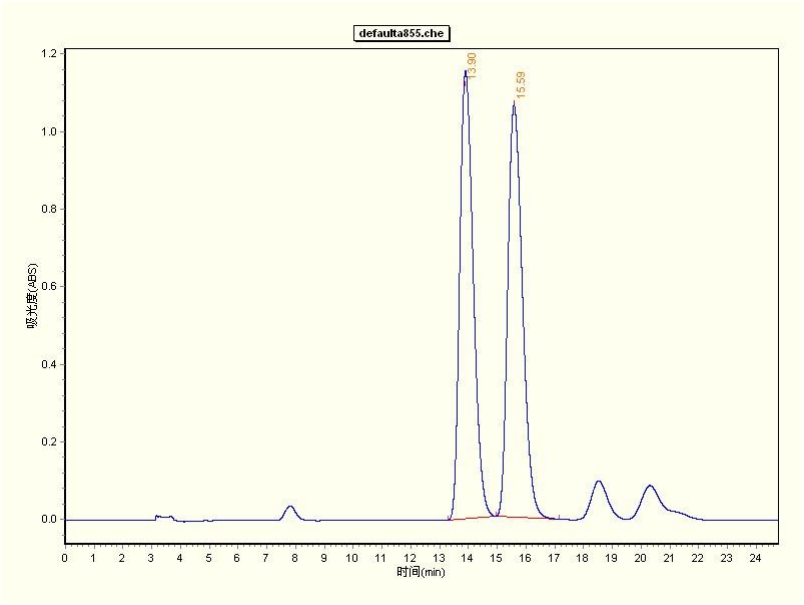
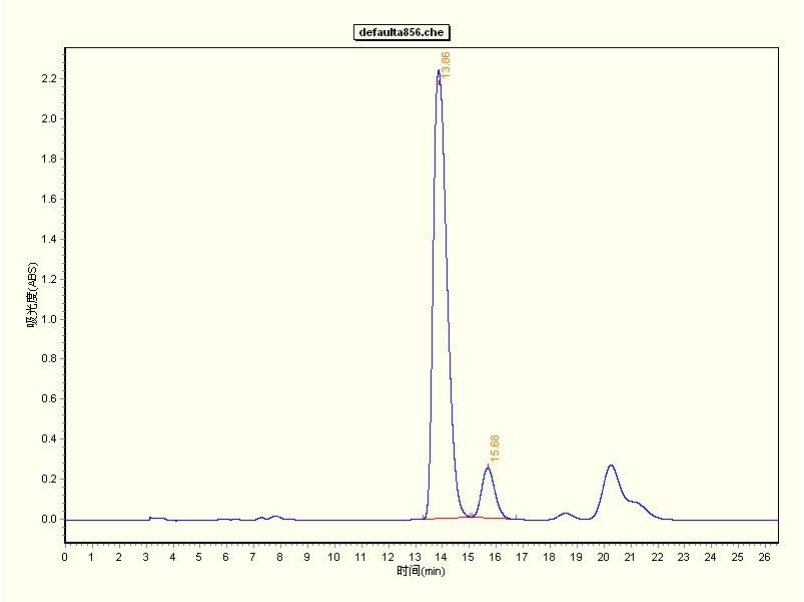
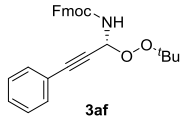


Entry	Retention time	Area	Height	Area%	Width	Type
1	13.90	18833788	576842	50.02	1.645	BB
2	15.59	18819072	530926	49.98	2.161	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	13.86	39522839	1118975	90.26	1.733	BB
2	15.68	4265388	125511	9.74	1.653	BB



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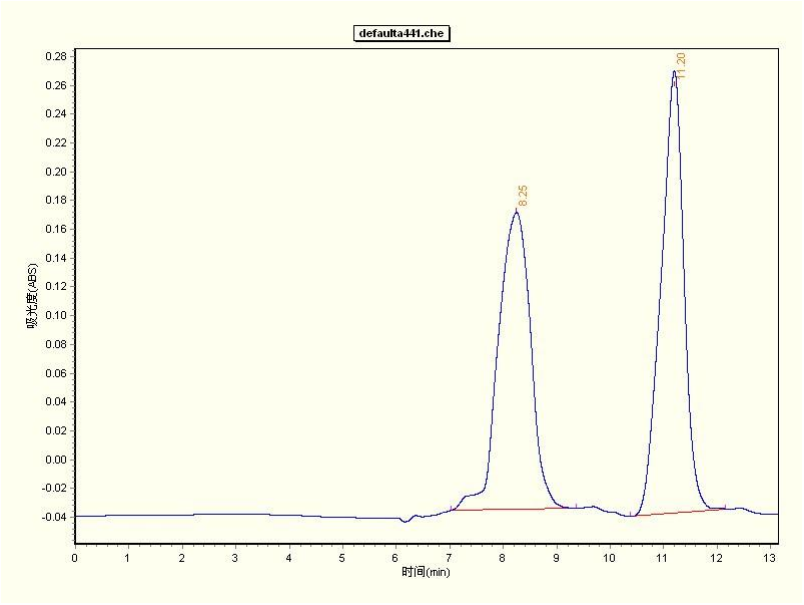
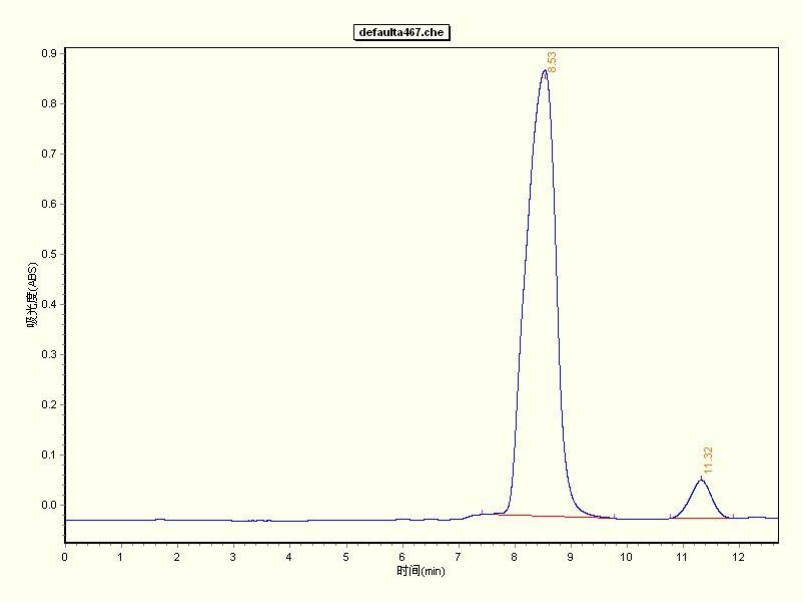
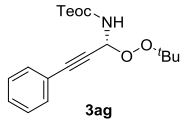


Entry	Retention time	Area	Height	Area%	Width	Type
1	8.25	4330919	103042	48.66	2.343	BB
2	11.20	4568608	153514	51.34	1.789	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.53	16323432	445050	94.14	2.351	BB
2	11.32	1015871	38256	5.86	1.119	BB



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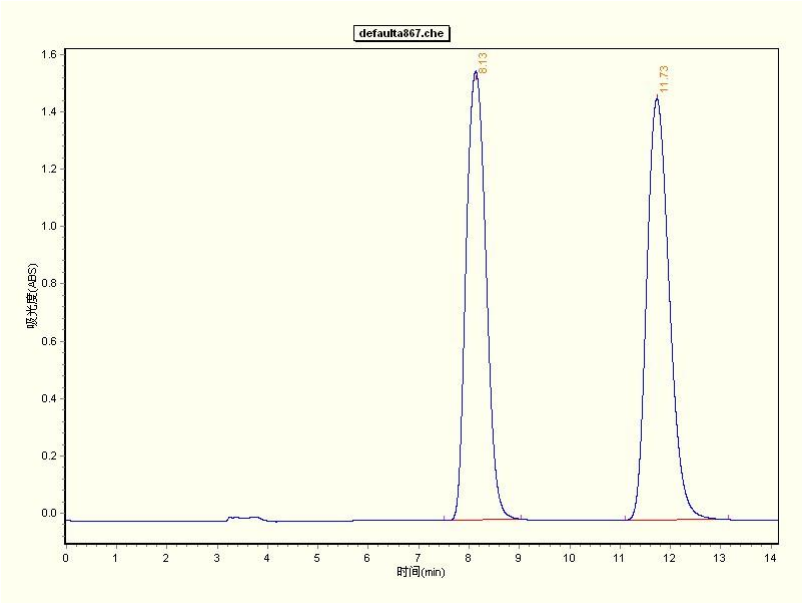
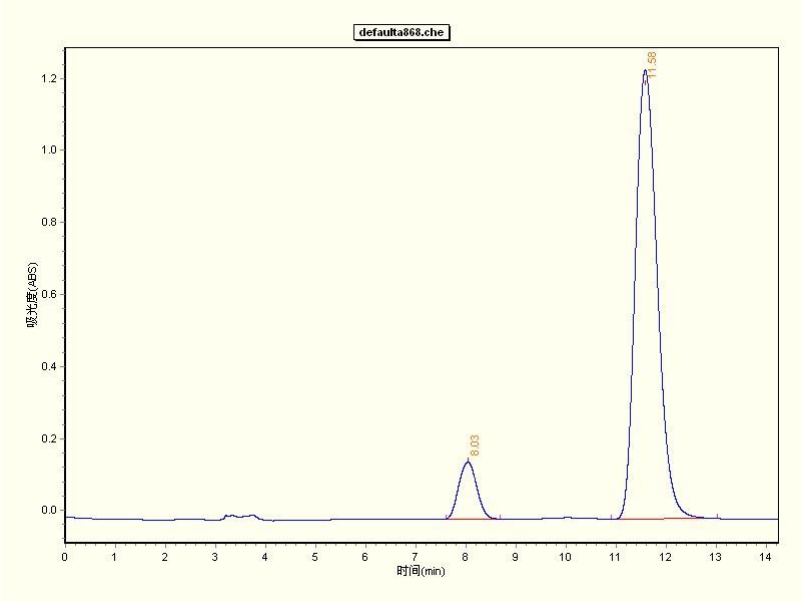
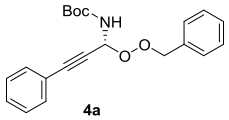


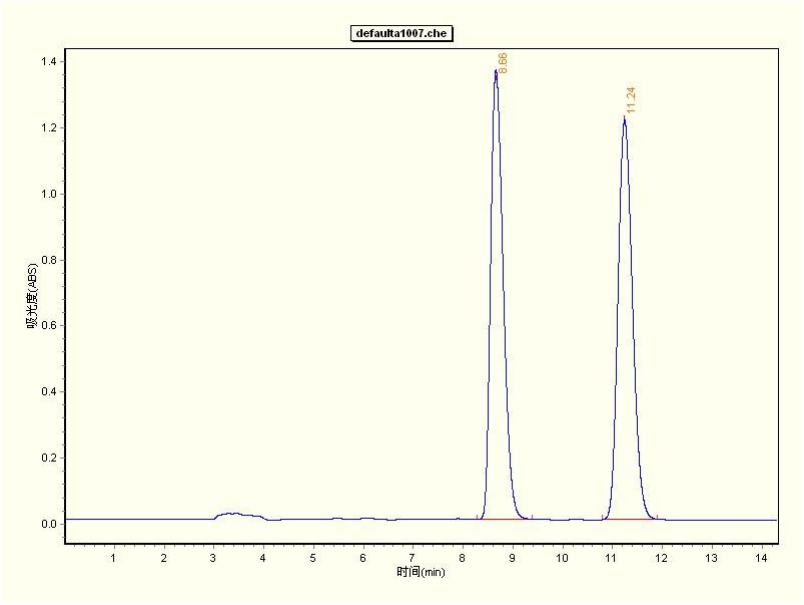
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.13	21372095	782509	48.37	1.530	BB
2	11.73	22808799	734661	51.63	2.049	BB



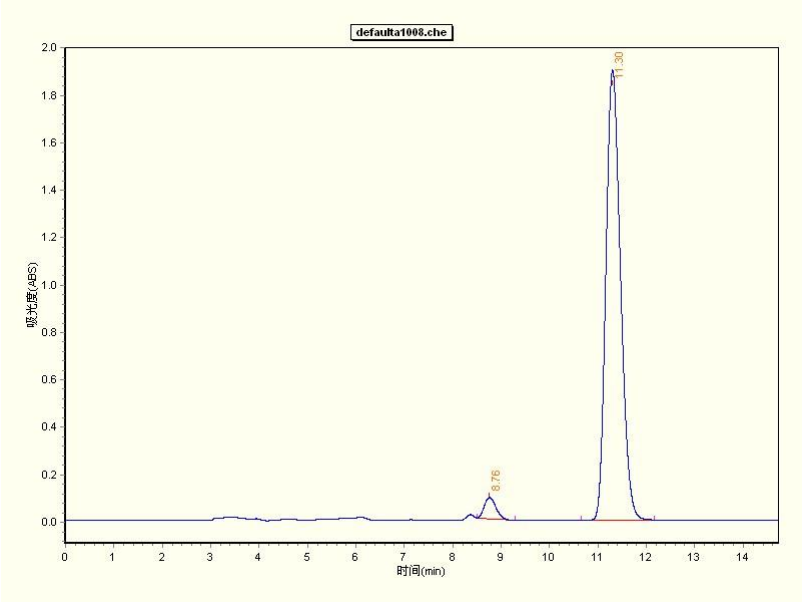
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.03	1970686	78807	9.41	1.072	BB
2	11.58	18968257	623045	90.59	2.117	BB



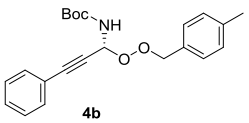
4a



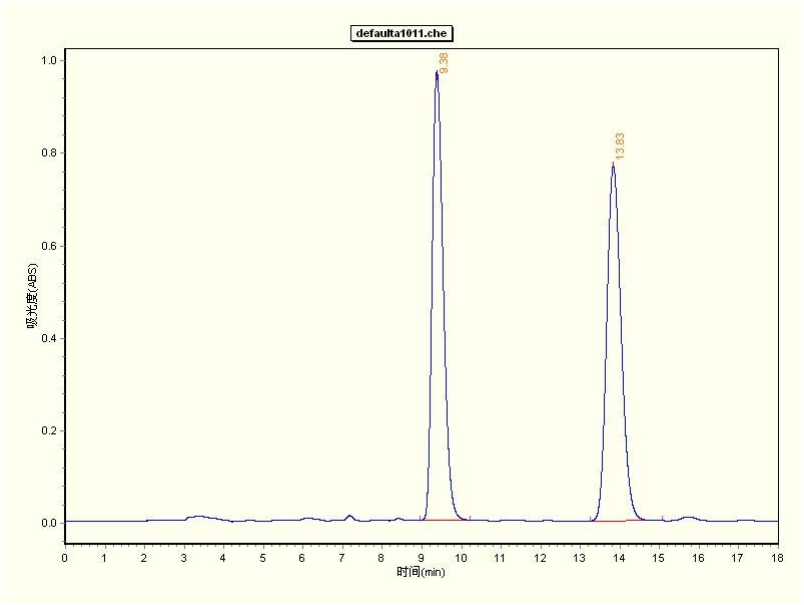
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.66	11898085	680367	49.34	1.121	BB
2	11.24	12215920	604577	50.66	1.107	BB



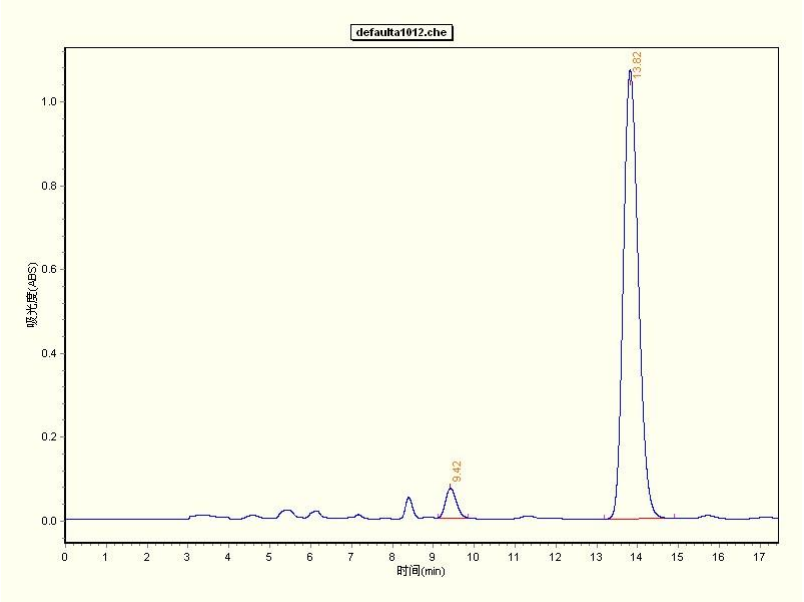
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.76	735583	44812	3.56	0.788	BB
2	11.30	19911820	949117	96.44	1.507	BB



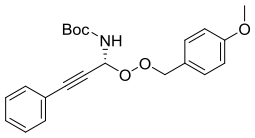
4b

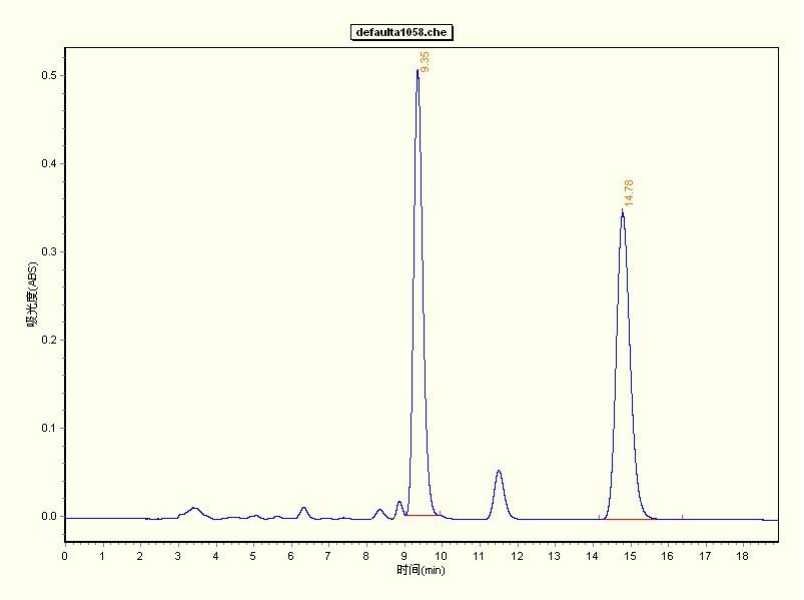


Entry	Retention time	Area	Height	Area%	Width	Type
1	9.38	9399997	485525	49.18	1.285	BB
2	13.83	9712728	383838	50.82	1.820	BB

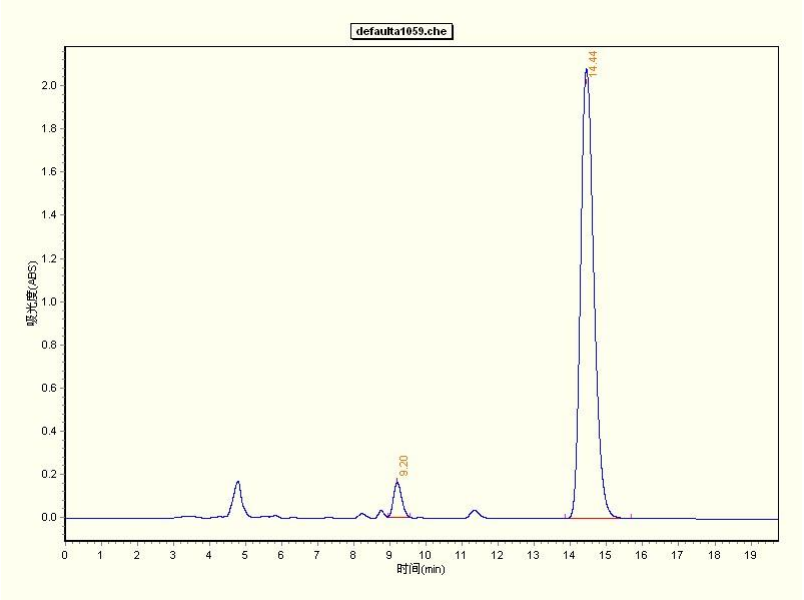


Entry	Retention time	Area	Height	Area%	Width	Type
1	9.42	643229	35338	4.49	0.726	BB
2	13.82	13678397	535990	95.51	1.712	BB

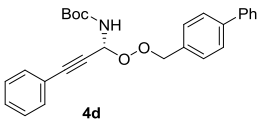




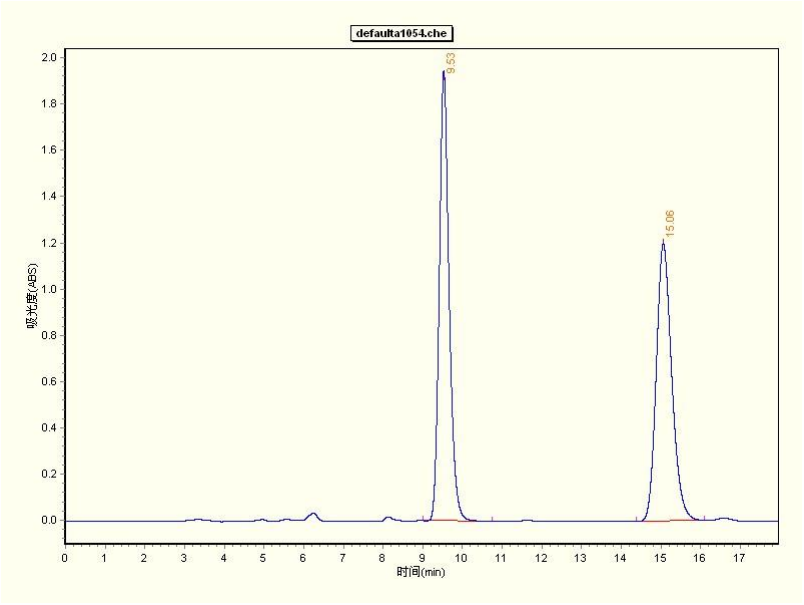
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.35	4236453	252562	48.77	0.913	BB
2	14.78	4449512	174089	51.23	2.205	BB



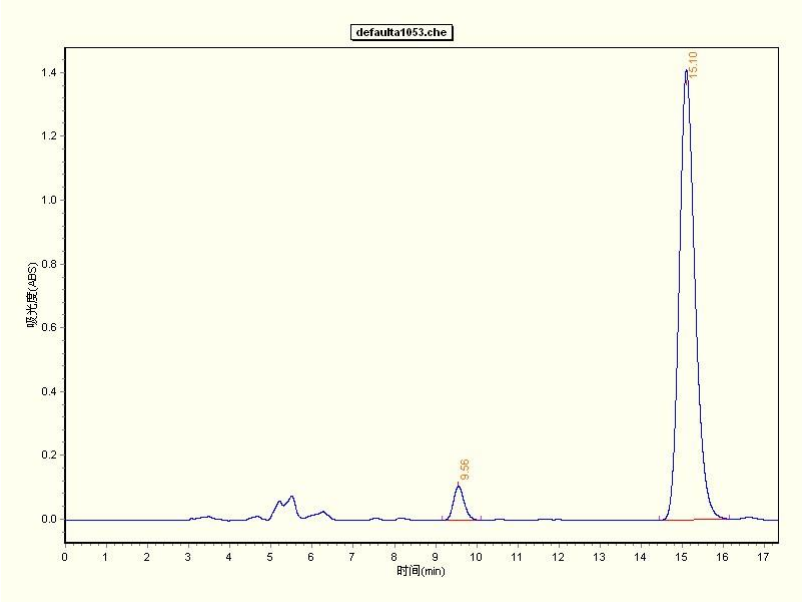
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.20	1259912	79725	4.51	0.630	BB
2	14.44	26694009	1039811	95.49	1.828	BB



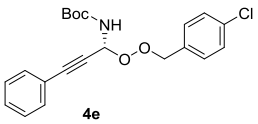
4d



Entry	Retention time	Area	Height	Area%	Width	Type
1	9.53	16776959	972474	50.88	1.730	BB
2	15.06	16199593	599814	49.12	1.730	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	9.56	969084	53170	4.91	0.945	BB
2	15.10	18777371	705091	95.09	1.718	BB



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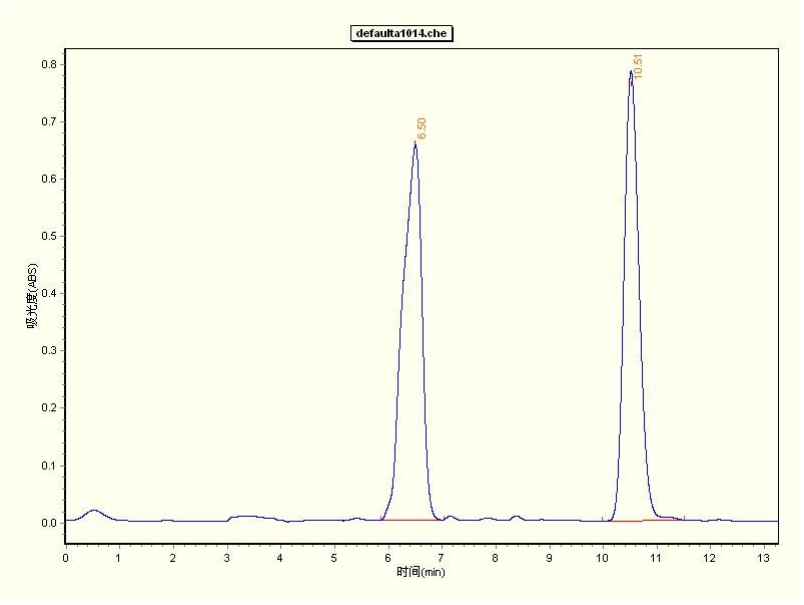
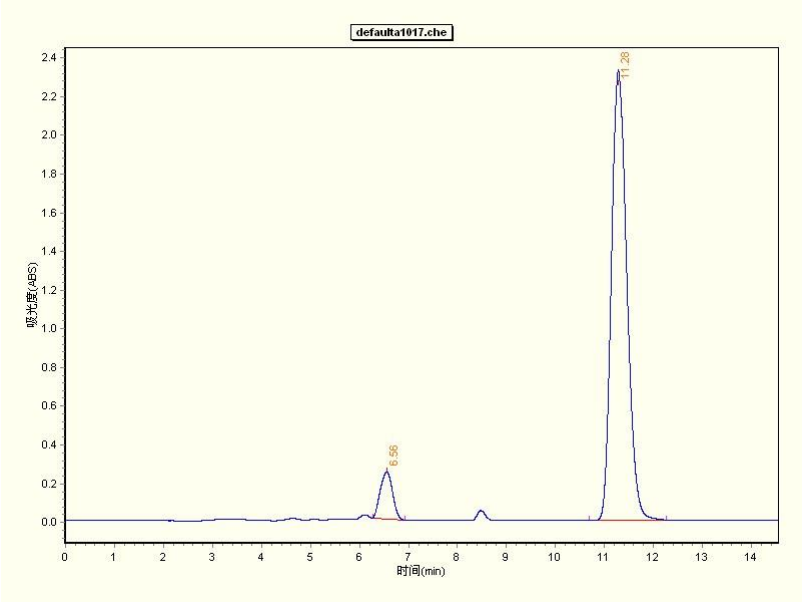
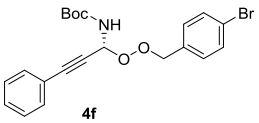


Entry	Retention time	Area	Height	Area%	Width	Type
1	6.50	8083302	326850	51.31	1.158	BB
2	10.51	7671308	392171	48.69	1.526	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	6.56	2118393	121535	7.67	0.657	BB
2	11.28	25483805	1163536	92.33	1.558	BB



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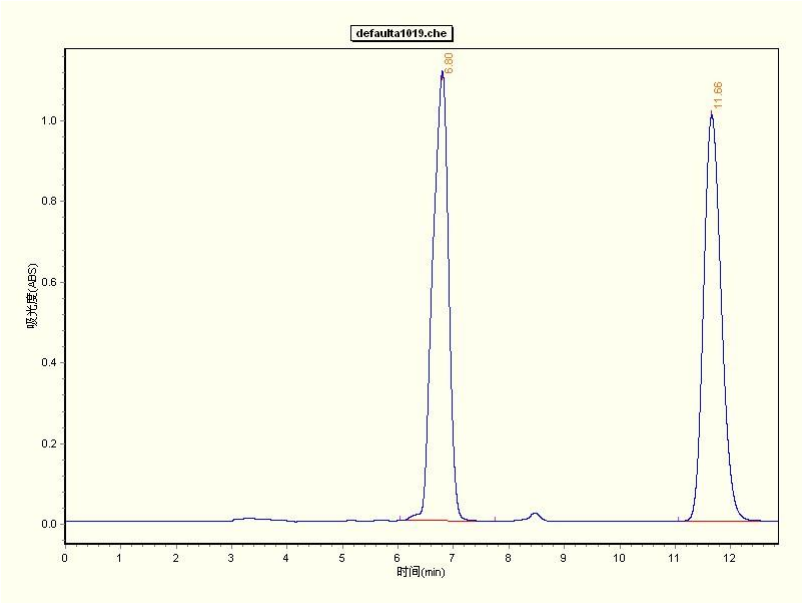
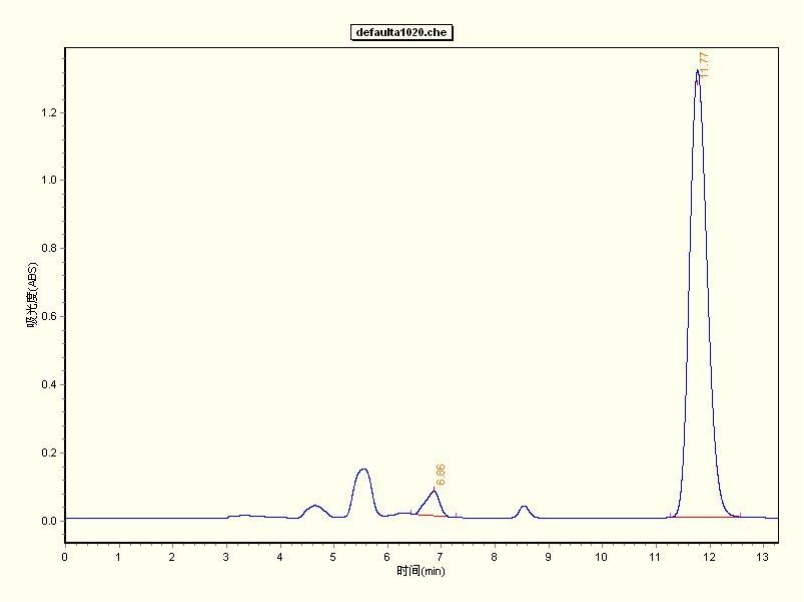
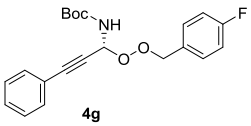


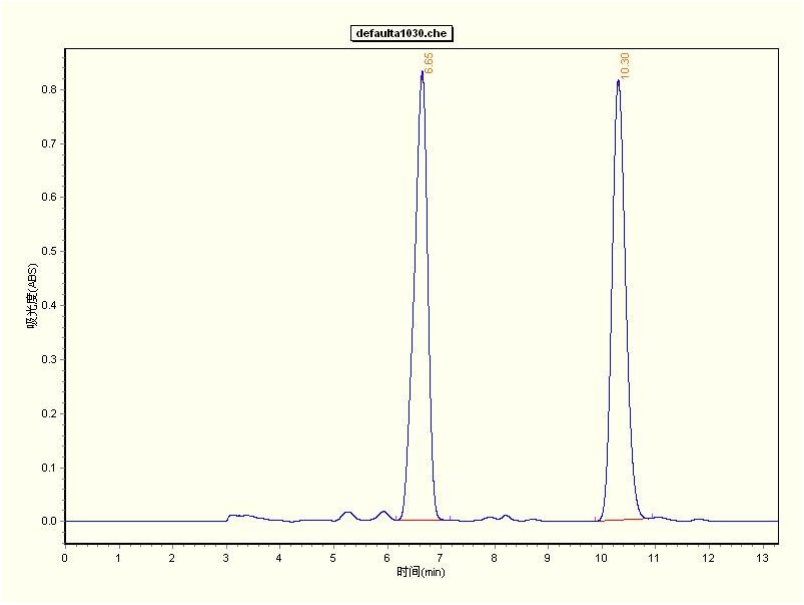
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.80	11038914	556794	49.74	1.708	BB
2	11.66	11156367	502777	50.26	1.928	BB



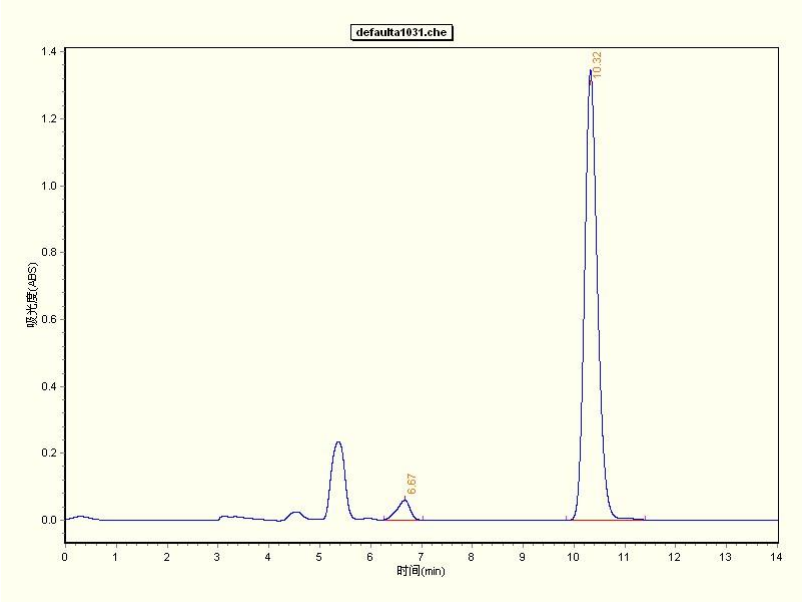
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.86	626578	35400	3.99	0.821	BB
2	11.77	15079913	656860	96.01	1.311	BB



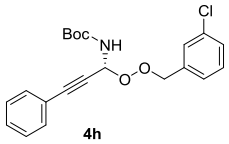
4g



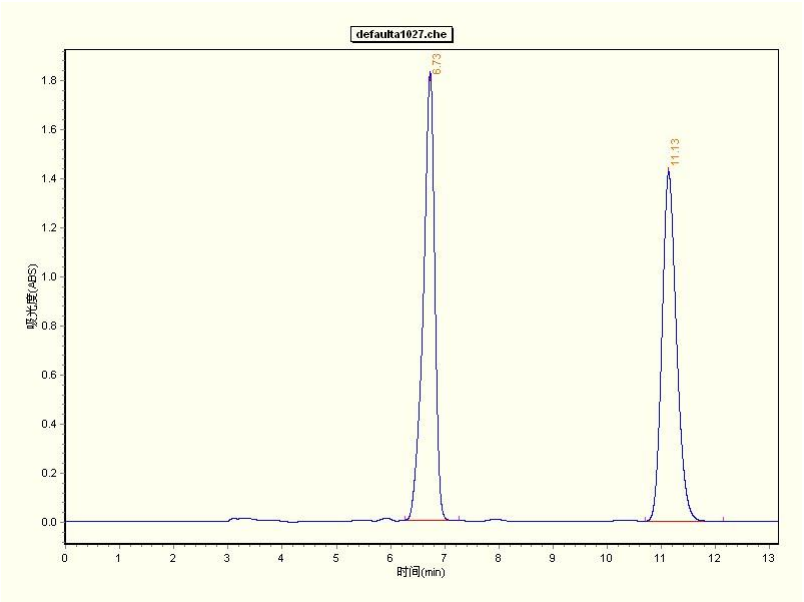
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.65	7251364	415780	50.21	0.996	BB
2	10.30	7191191	407562	49.79	1.059	BB



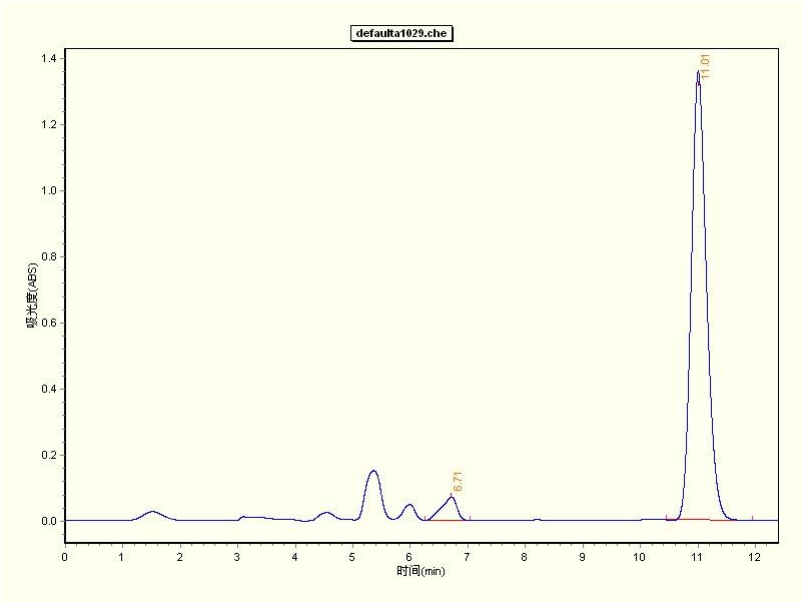
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.67	522899	29441	4.25	0.767	BB
2	10.32	11788292	672375	95.75	1.550	BB



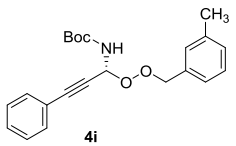
4h



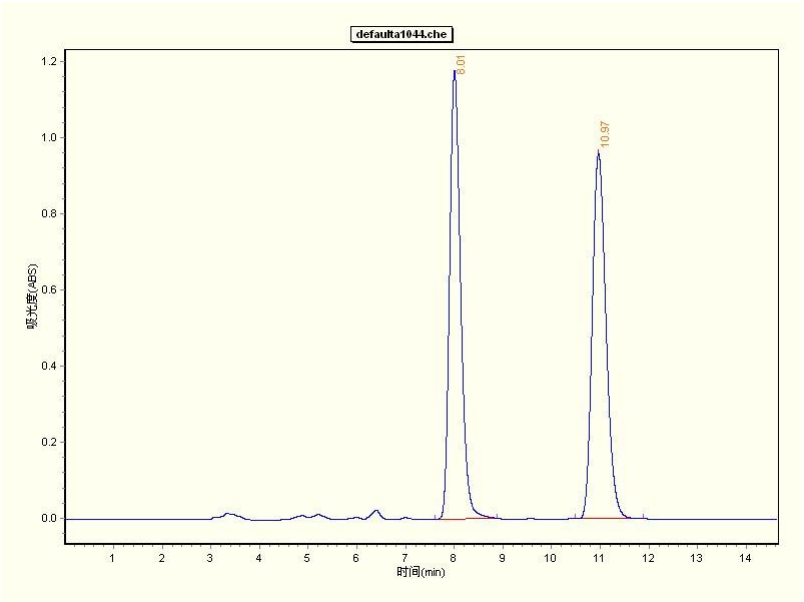
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.73	13427126	914641	50.34	0.991	BB
2	11.13	13245445	712370	49.66	1.434	BB



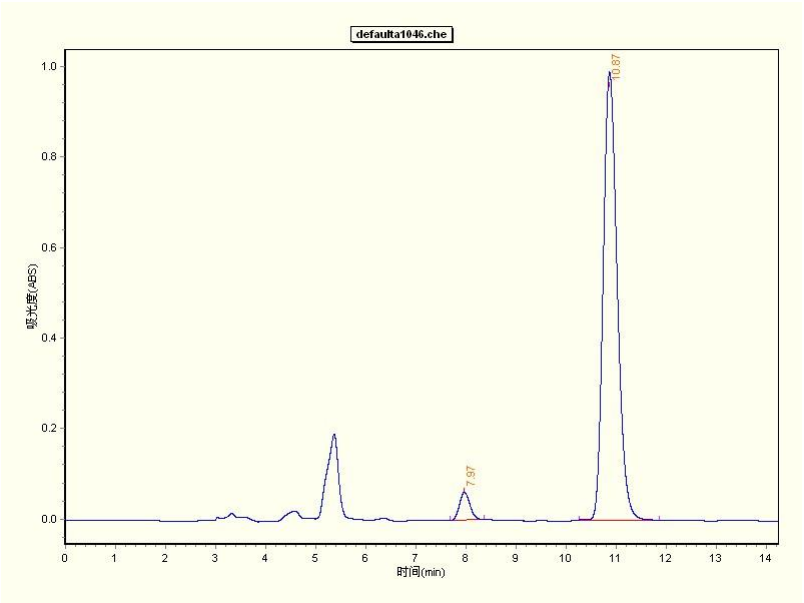
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.71	646683	34991	4.92	0.796	BB
2	11.01	12499744	679180	95.08	1.504	BB



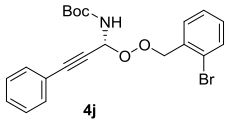
4i



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.01	9254821	588662	49.63	1.278	BB
2	10.97	9391481	479441	50.37	1.379	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	7.97	437254	30754	4.35	0.692	BB
2	10.87	9605712	495286	95.65	1.596	BB



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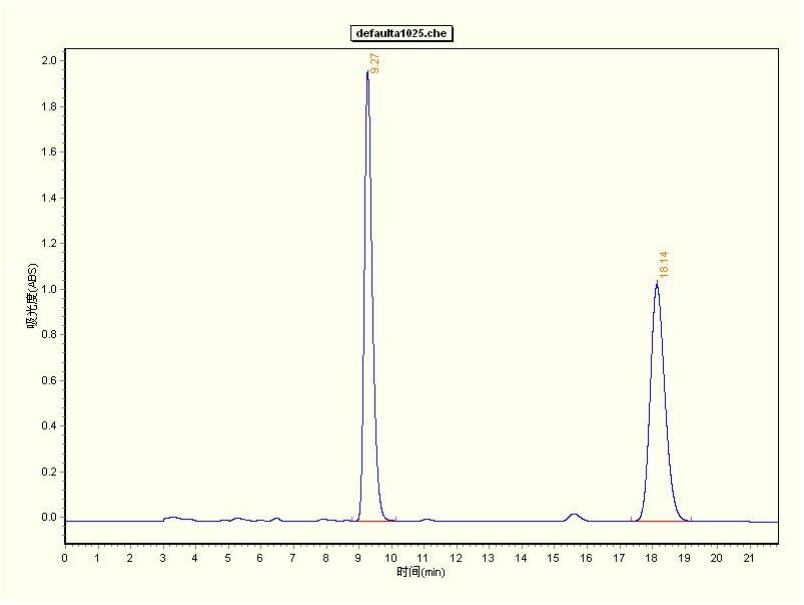
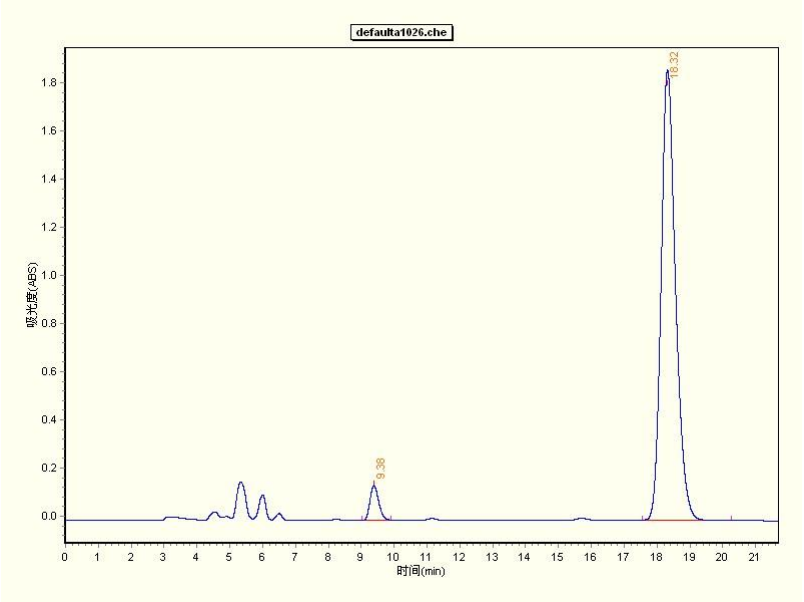
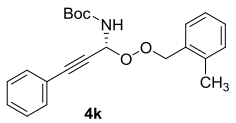


Entry	Retention time	Area	Height	Area%	Width	Type
1	9.27	16781861	984898	50.94	1.343	BB
2	18.14	16165514	517834	49.06	1.863	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	9.38	1331692	72337	4.47	0.882	BB
2	18.32	28469021	934607	95.53	2.731	BB



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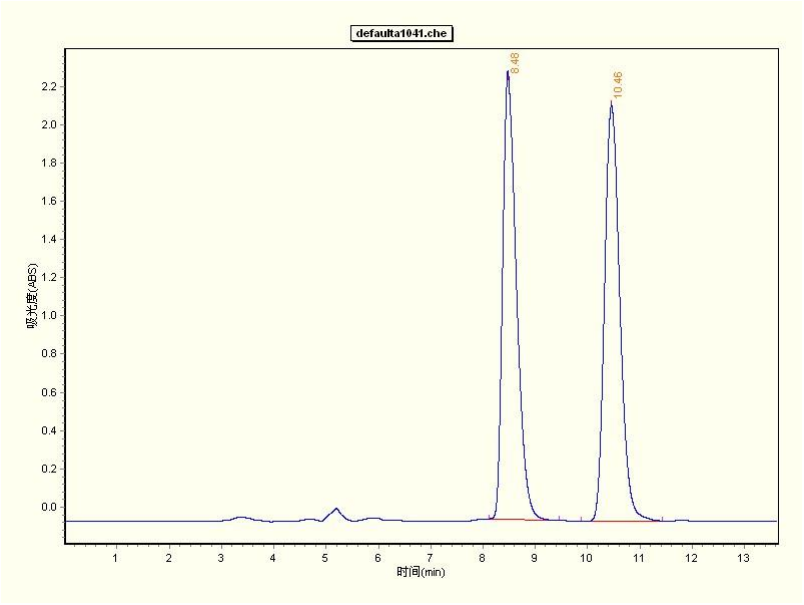
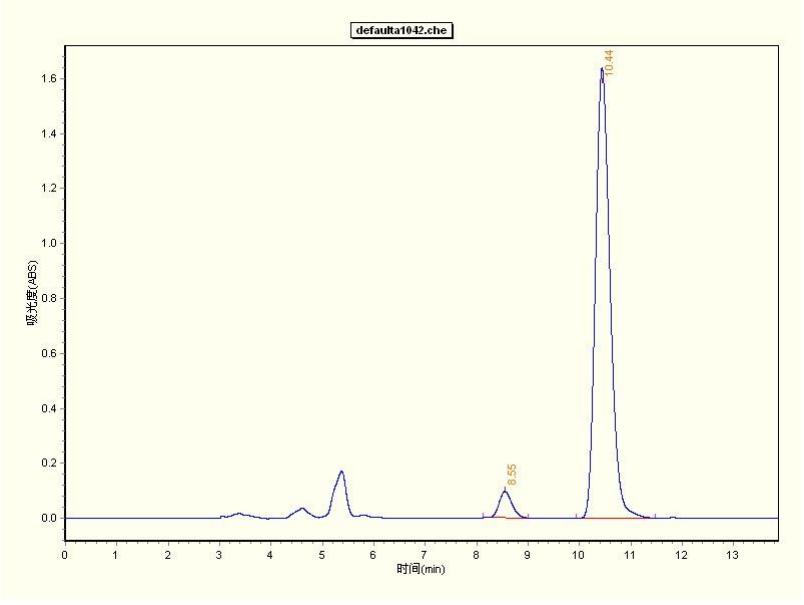
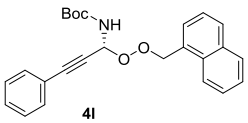


Entry	Retention time	Area	Height	Area%	Width	Type
1	8.48	21380820	1171178	49.14	1.338	BB
2	10.46	22130952	1087902	50.86	1.554	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.55	822791	47824	4.86	0.886	BB
2	10.44	16114982	818780	95.14	1.539	BB



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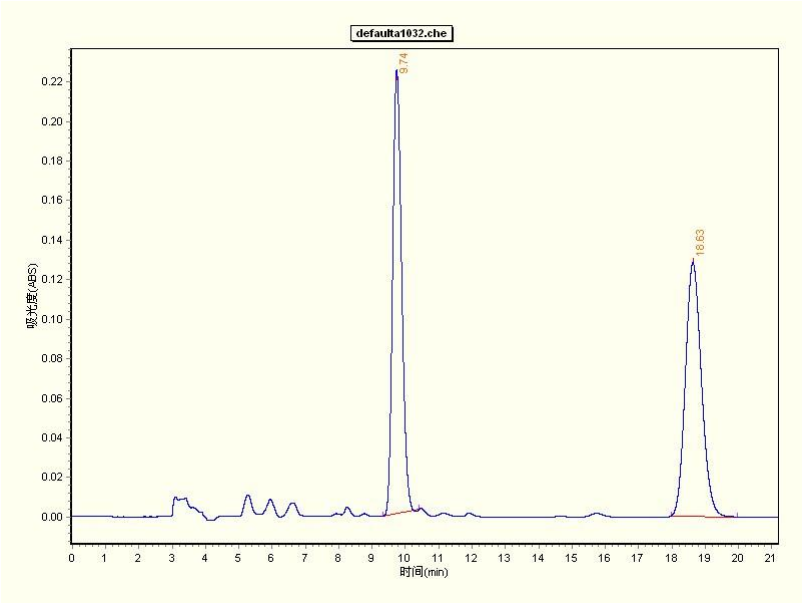
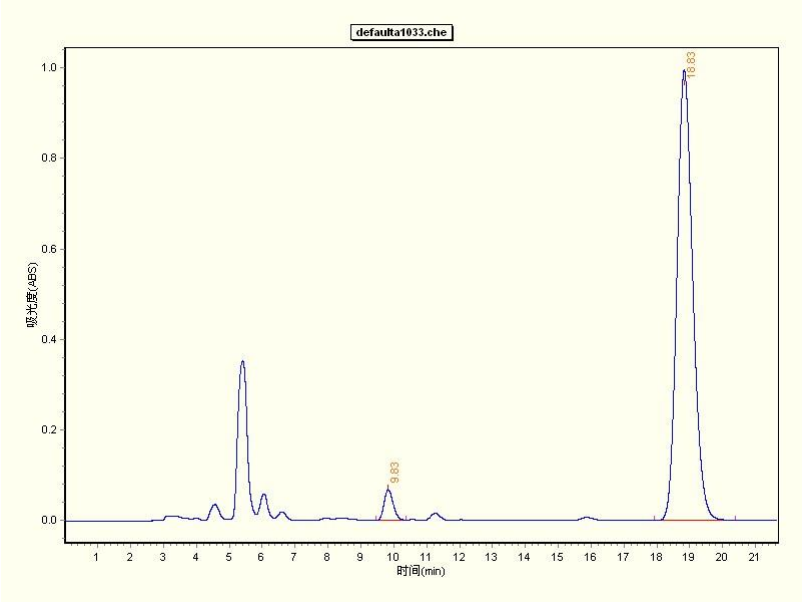
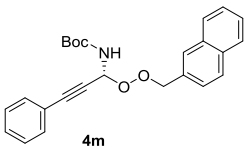


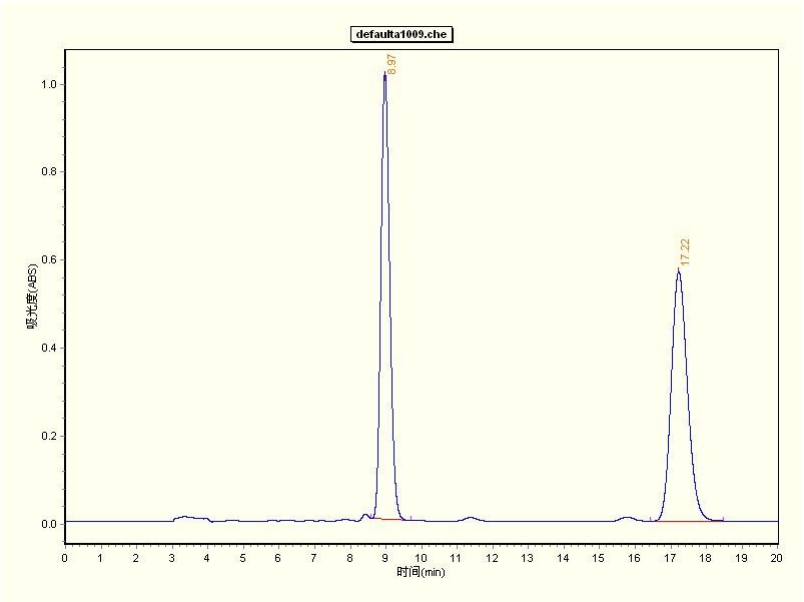
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.74	2093911	112178	49.44	1.092	BB
2	18.63	2141654	64090	50.56	2.005	BB



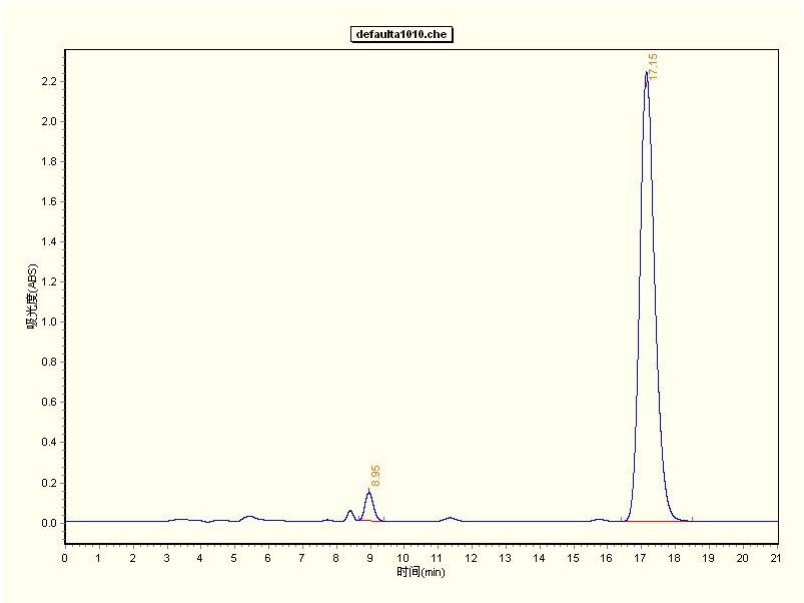
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.83	642506	33808	3.72	0.902	BB
2	18.83	16627433	497012	96.28	2.475	BB



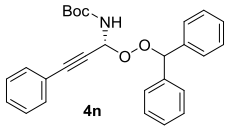
4m



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.97	8752327	508067	49.45	1.143	BB
2	17.22	8945685	283526	50.55	2.048	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	8.95	127068	70783	3.43	0.751	BB
2	17.15	34537575	1119426	96.57	2.077	BB



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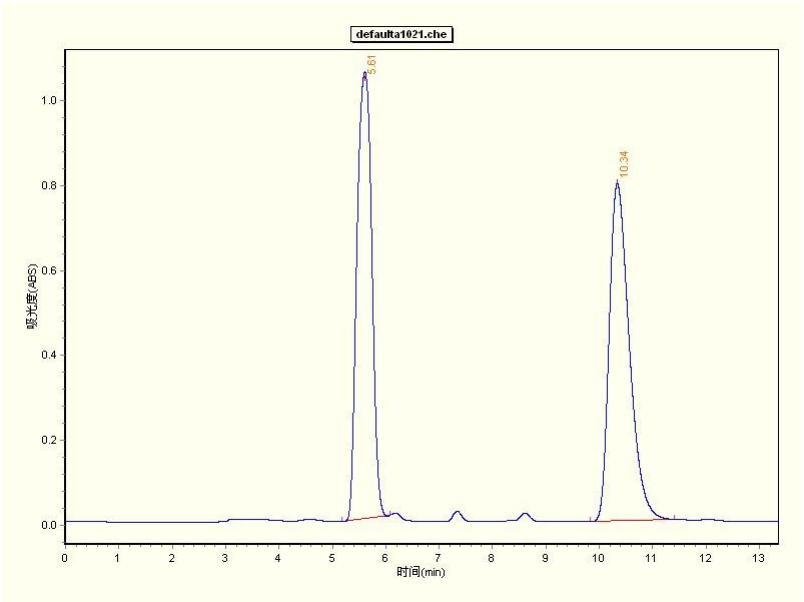
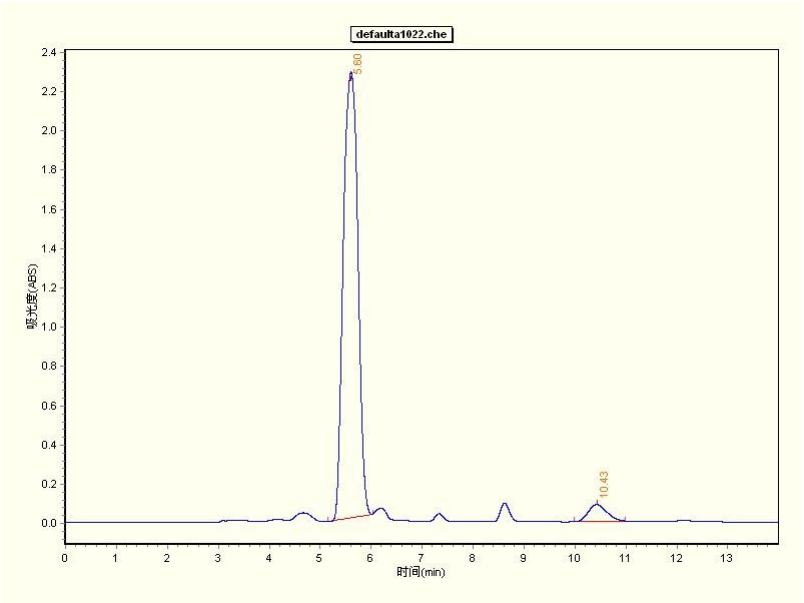
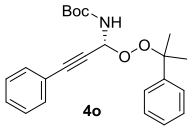


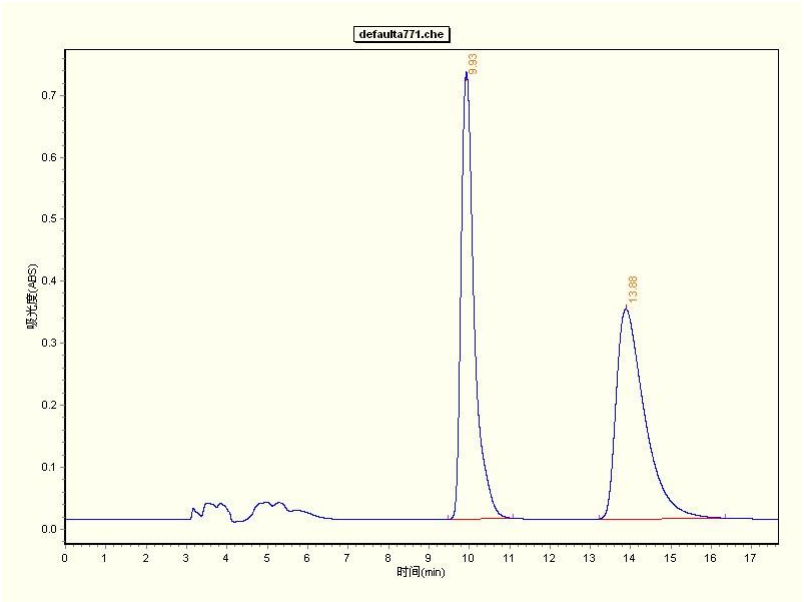
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.61	10129388	525904	49.45	0.900	BB
2	10.34	10355933	397616	50.55	1.562	BB



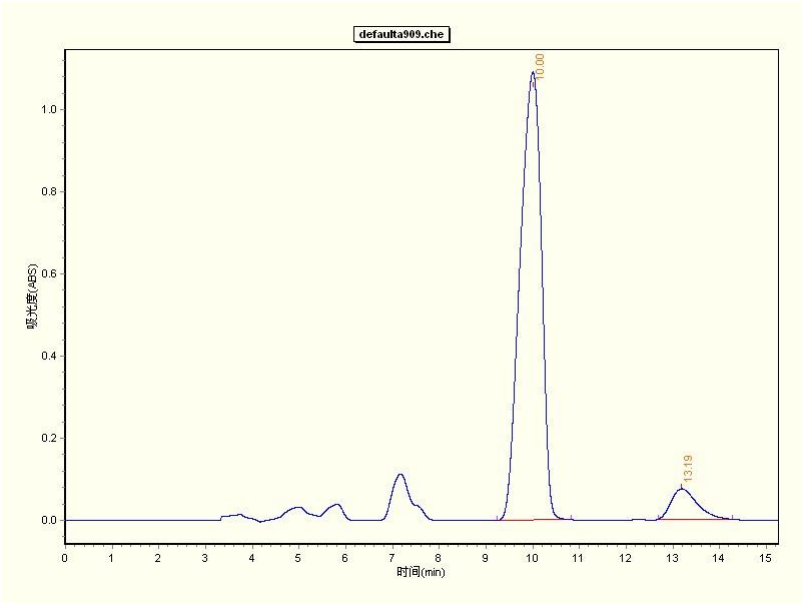
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.60	22564577	1135252	95.44	0.887	BB
2	10.43	1078184	42750	4.56	0.998	BB



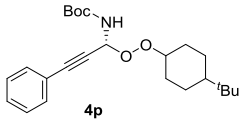
4o



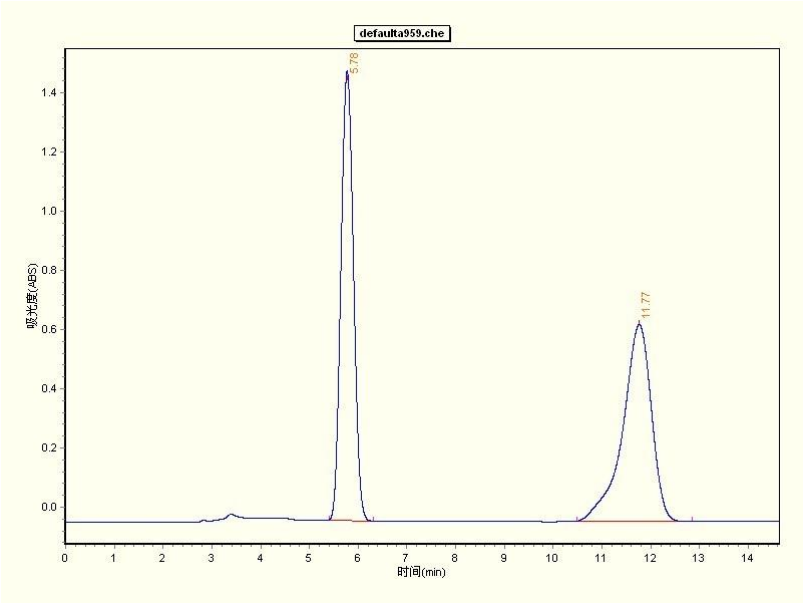
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.93	8157730	360508	49.64	1.618	BB
2	13.88	8275513	169235	50.36	3.108	BB



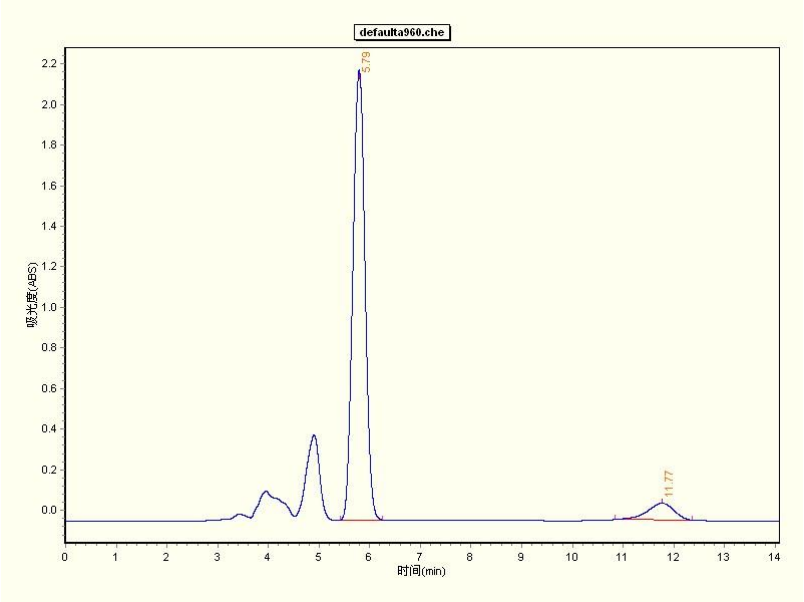
Entry	Retention time	Area	Height	Area%	Width	Type
1	10.00	17308990	546022	92.39	1.578	BB
2	13.19	1425097	37113	7.61	1.597	BB



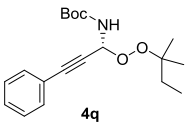
4p



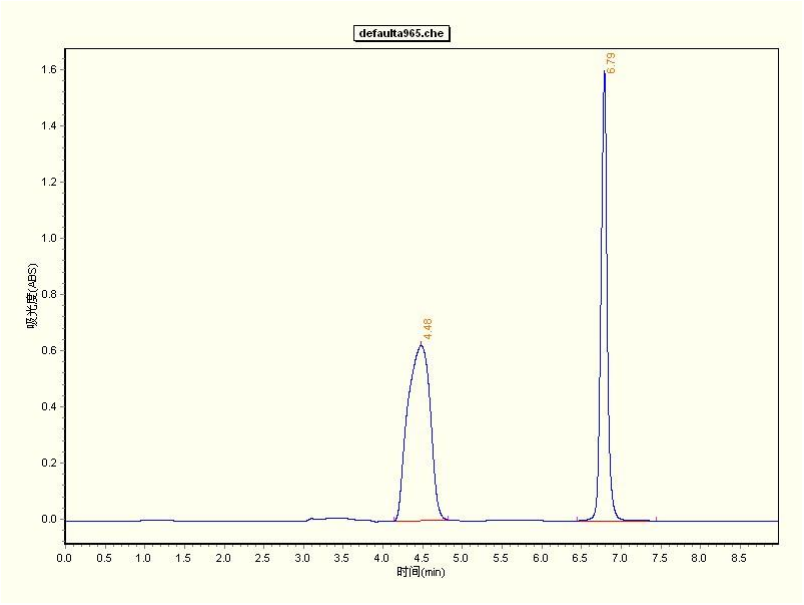
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.78	13558828	760316	49.80	0.922	BB
2	11.77	13666577	333002	50.20	2.368	BB



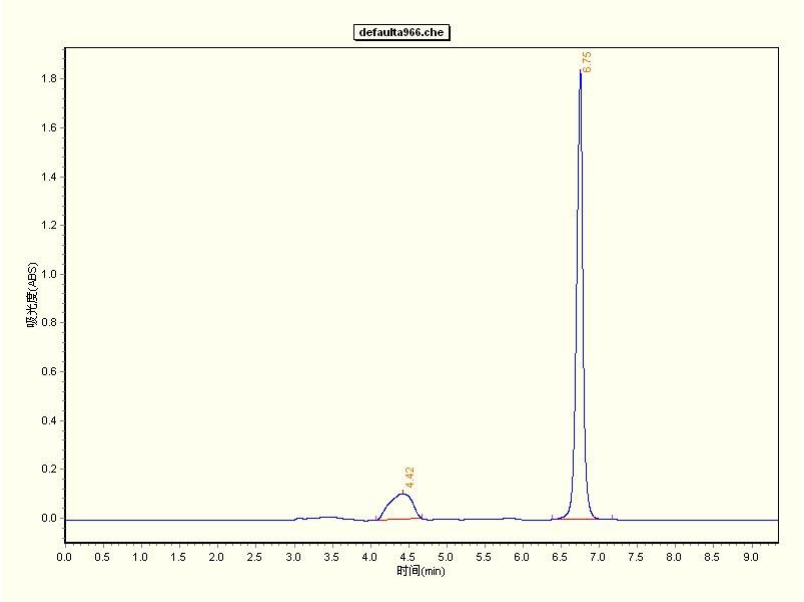
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.79	18393855	1107981	92.43	0.838	BB
2	11.77	1505405	41011	7.57	1.524	BB



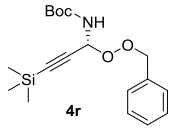
4q



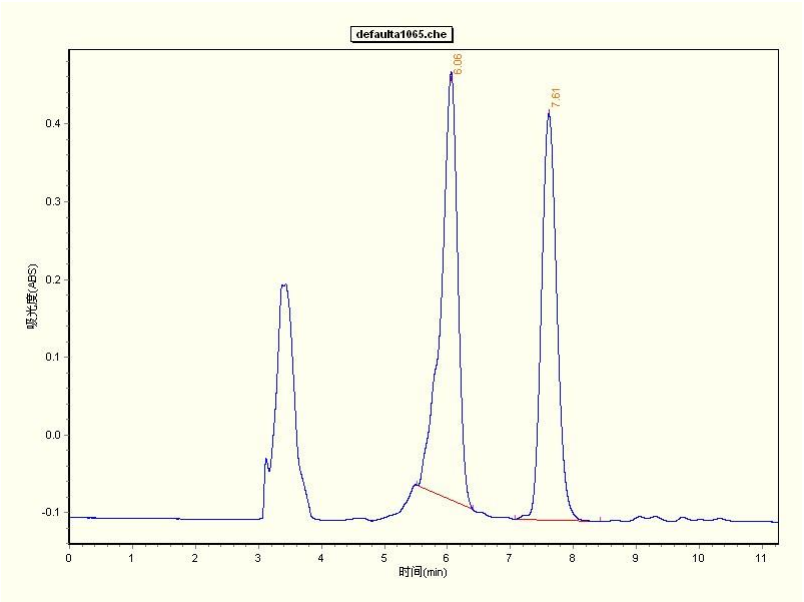
Entry	Retention time	Area	Height	Area%	Width	Type
1	4.48	6126489	311121	58.38	0.683	BB
2	6.79	4366814	799779	41.62	1.003	BB



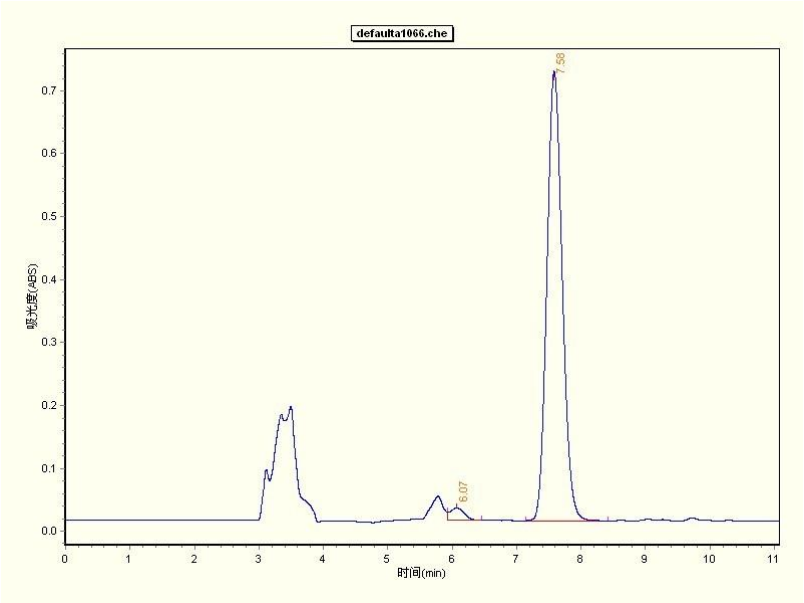
Entry	Retention time	Area	Height	Area%	Width	Type
1	4.42	1095112	53629	16.94	0.603	BB
2	6.75	5369605	921016	83.06	0.787	BB



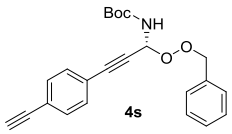
4r



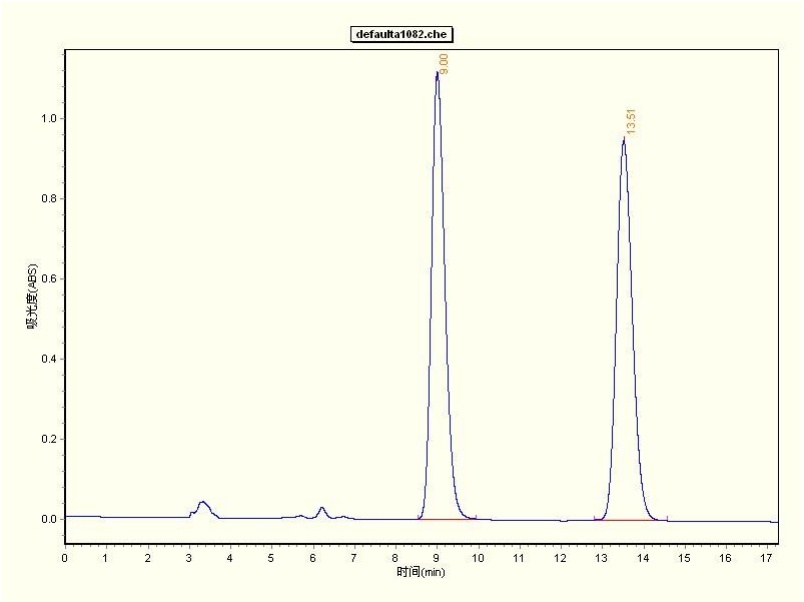
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.06	5103117	275568	53.45	0.892	BB
2	7.61	4444661	261556	46.55	1.354	BB



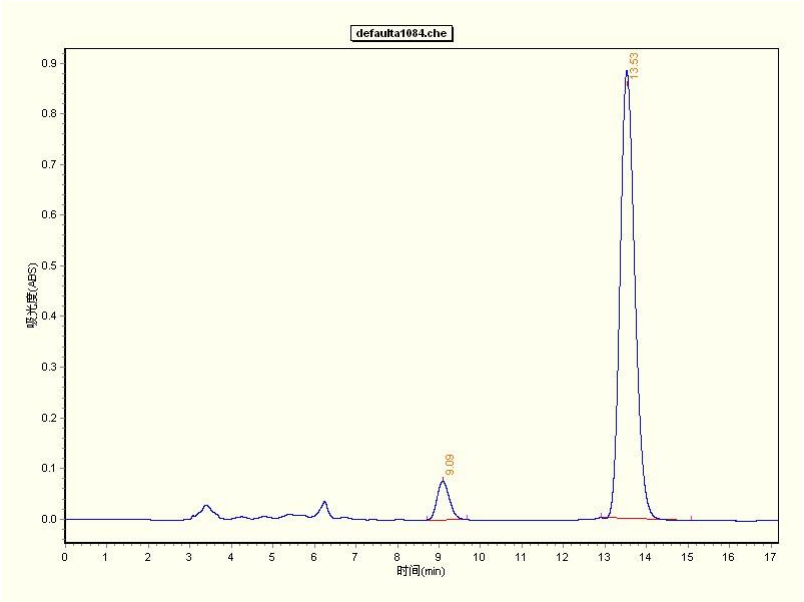
Entry	Retention time	Area	Height	Area%	Width	Type
1	6.07	432105	18677	6.83	0.553	BB
2	7.58	5892391	356865	93.17	1.278	BB



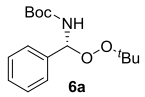
4s



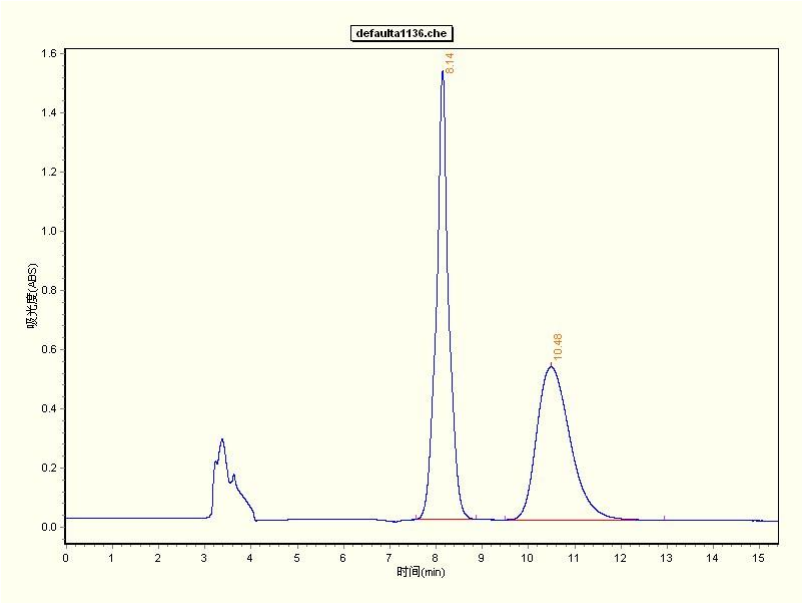
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.00	12640017	557604	49.92	1.407	BB
2	13.51	12682036	473436	50.08	1.762	BB



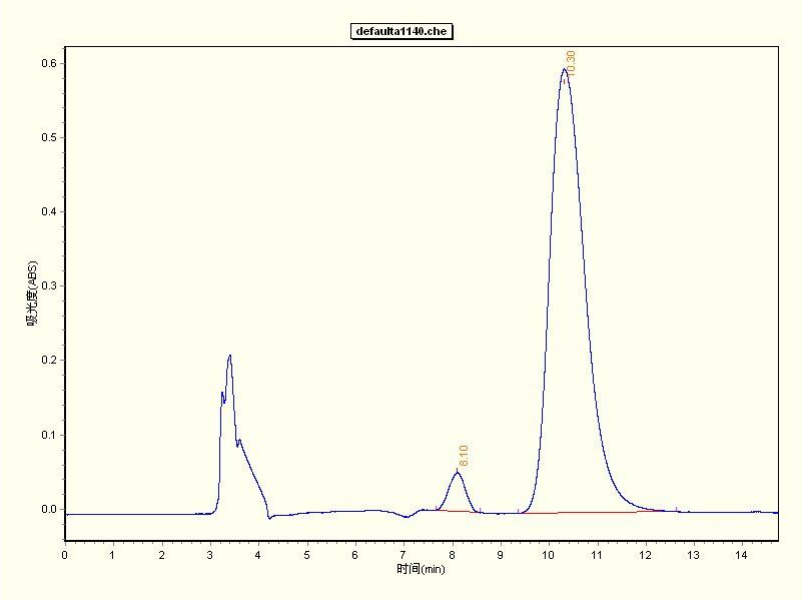
Entry	Retention time	Area	Height	Area%	Width	Type
1	9.09	787165	38220	6.76	0.970	BB
2	13.53	10857826	441855	93.24	2.173	BB



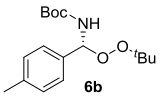
6a



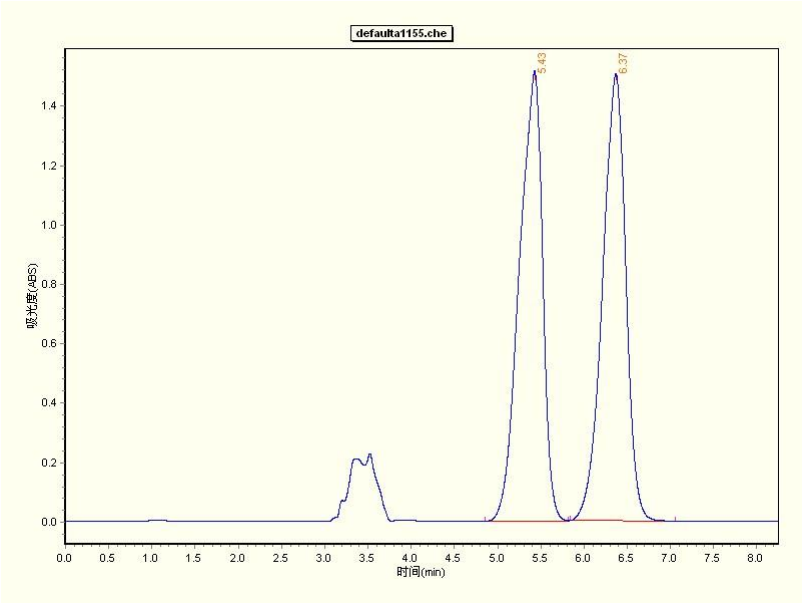
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.14	14159486	775720	52.25	1.307	BB
2	10.48	12939361	258307	47.75	3.441	BB



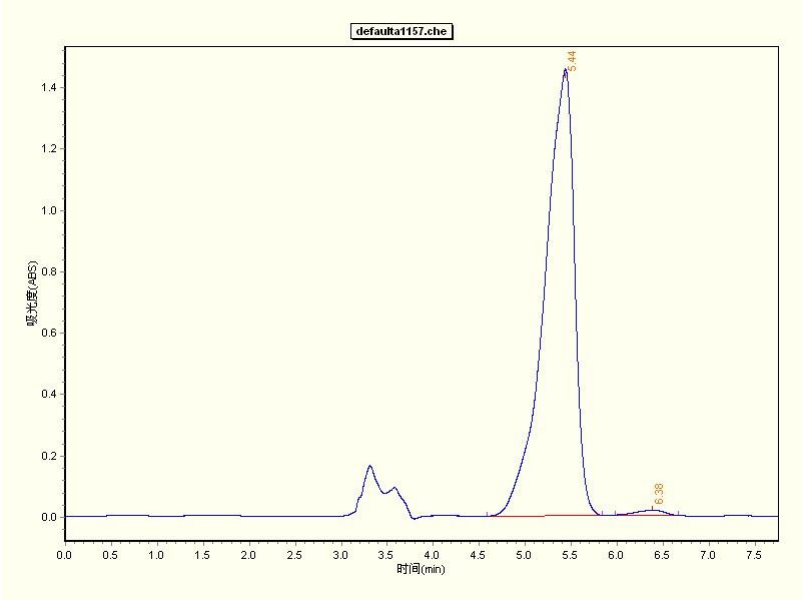
Entry	Retention time	Area	Height	Area%	Width	Type
1	8.10	627677	26254	3.97	0.907	BB
2	10.30	15180824	298971	96.03	3.278	BB



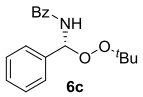
6b



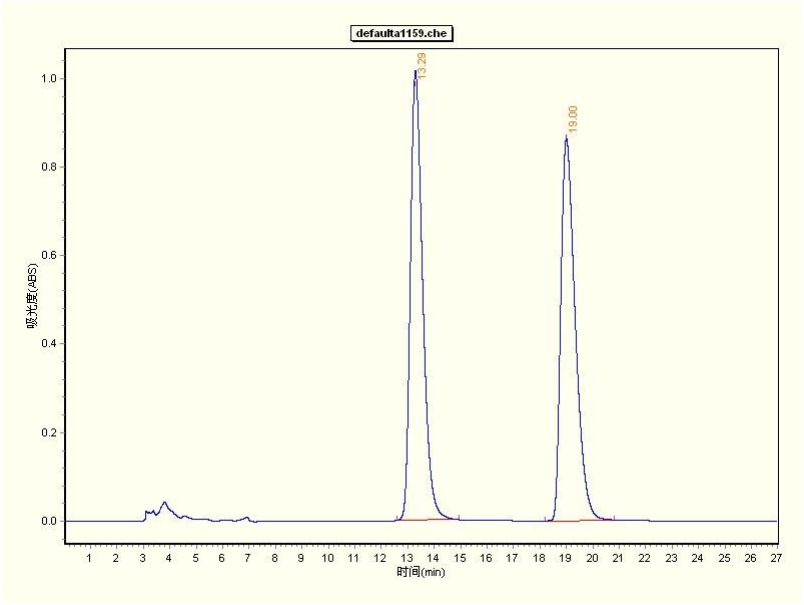
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.43	14087316	756610	50.08	0.963	BB
2	6.37	14041212	752312	49.98	1.213	BB



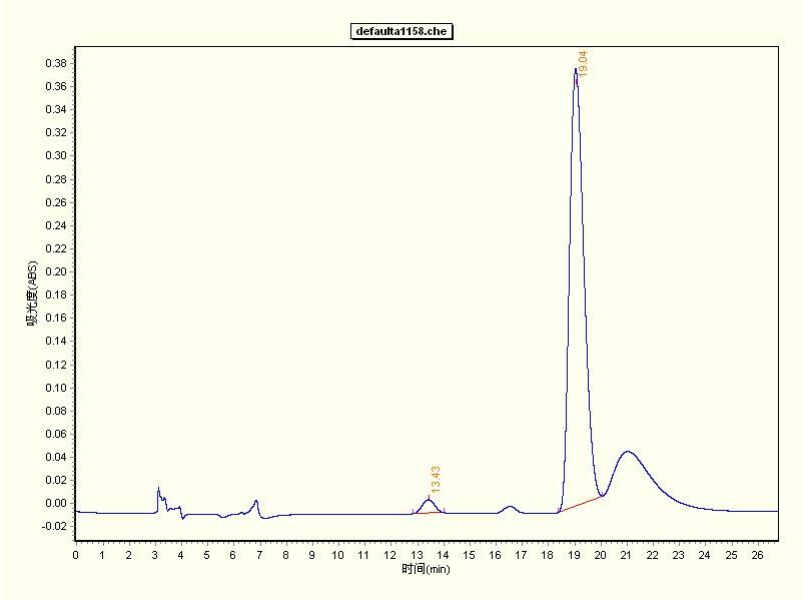
Entry	Retention time	Area	Height	Area%	Width	Type
1	5.44	16554007	727840	98.95	1.254	BB
2	6.38	176318	8394	1.05	0.692	BB



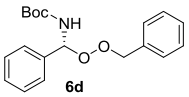
6c



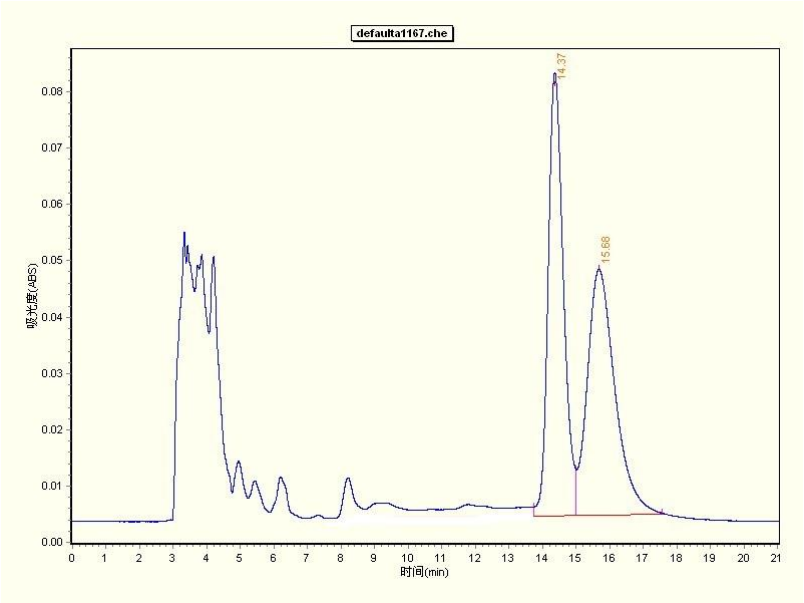
Entry	Retention time	Area	Height	Area%	Width	Type
1	13.29	16452627	507804	50.08	2.333	BB
2	19.00	16398860	432046	49.92	2.610	BB



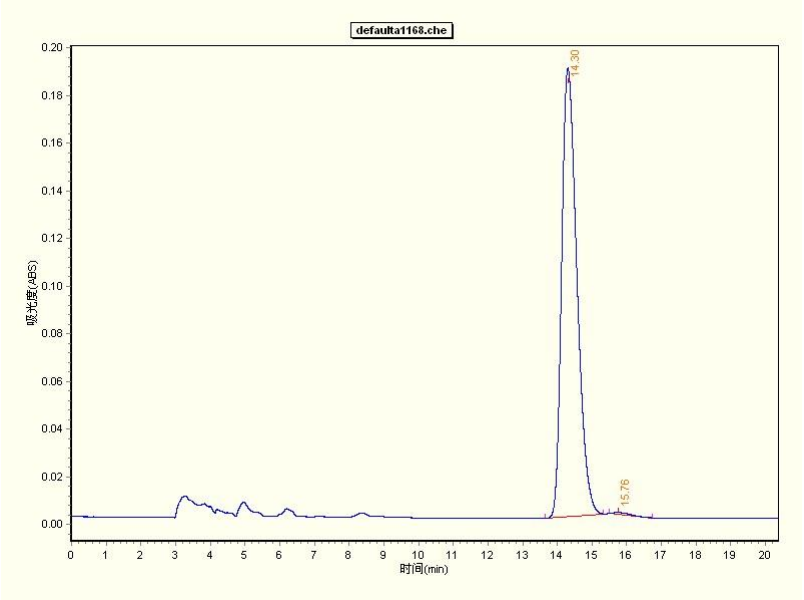
Entry	Retention time	Area	Height	Area%	Width	Type
1	13.43	194658	5847	2.64	1.208	BB
2	19.04	7189295	1888699	97.36	1.692	BB



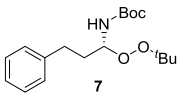
6d



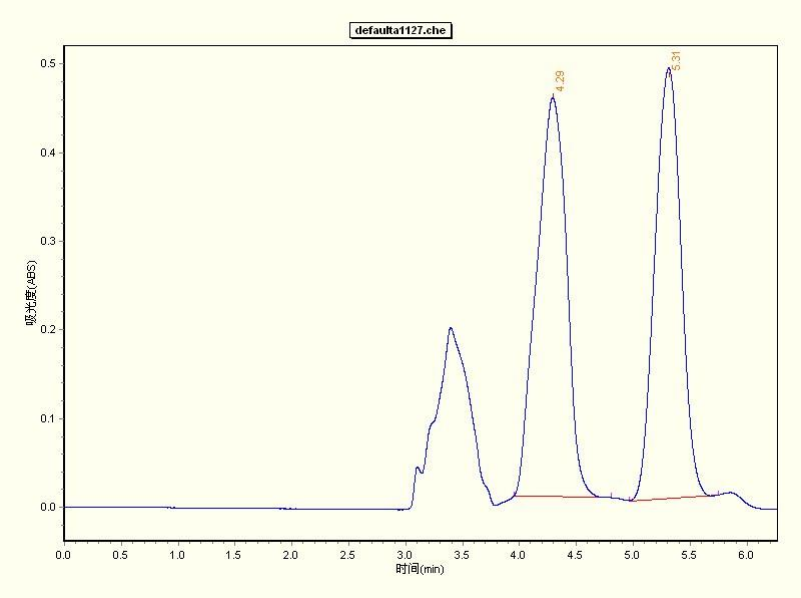
Entry	Retention time	Area	Height	Area%	Width	Type
1	14.37	1237712	39283	49.88	1.279	BB
2	15.68	1243819	21792	50.12	2.543	BB



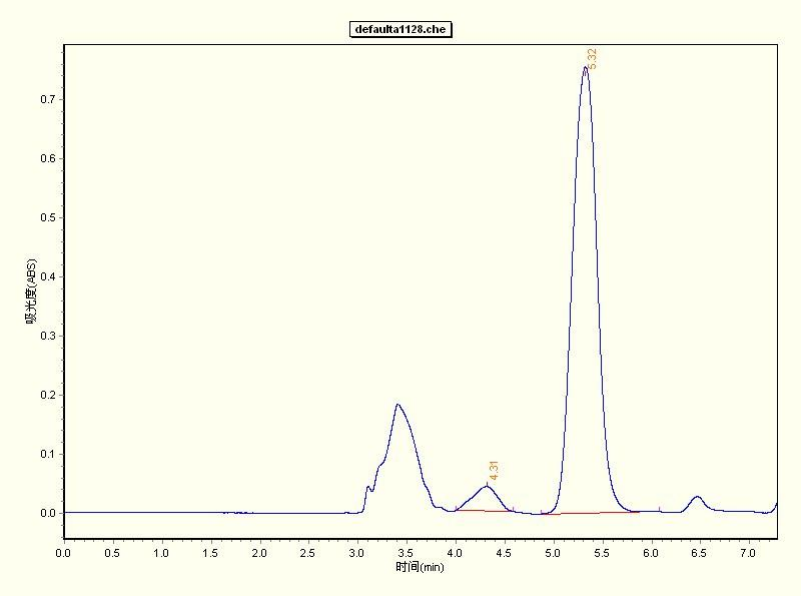
Entry	Retention time	Area	Height	Area%	Width	Type
1	14.30	2869484	94041	99.31	1.667	BB
2	15.76	19961	553	0.69	1.278	BB



7



Entry	Retention time	Area	Height	Area%	Width	Type
1	4.29	4108205	225148	51.45	0.852	BB
2	5.31	3876196	242504	48.55	0.789	BB



Entry	Retention time	Area	Height	Area%	Width	Type
1	4.31	364815	20666	5.23	0.585	BB
2	5.32	6616835	377933	94.77	1.210	BB