Highly Chemoselective and Enantioselective Synthesis of 3,4-2H-Pyrindin-2-ones by an NHC-Catalyzed [3+3] Cyclization

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General Information:

Analytical thin layer chromatography (TLC) was performed using Merck 60 F254 precoated silica gel plate (0.2 mm thickness). Subsequent to elution, plates were visualized using UV radiation (254 nm) on Spectroline Model ENF-24061/F 254 nm.

Flash column chromatography was performed using silica gel column chromatography with freshly distilled solvents. Columns were typically packed as slurry and equilibrated with the appropriate solvent system prior to use.

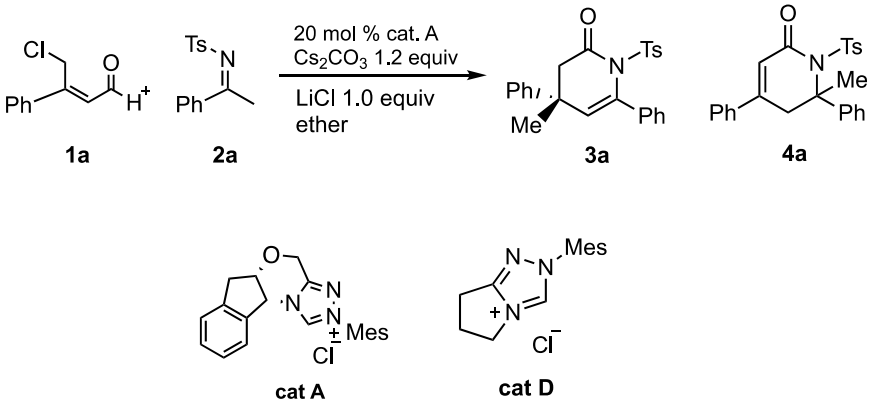
Proton nuclear magnetic resonance spectra (^{1}H NMR) were recorded on Bruker AMX 500 spectrophotometer ($CDCl_3$ as solvent). Chemical shifts for ^{1}H NMR spectra are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 7.26, singlet). Multiplicities were given as: s (singlet), d (doublet), t (triplet), dd (doublets of doublet) or m (multiplets). The number of protons (n) for a given resonance is indicated by nH. Coupling constants are reported as a *J* value in Hz. Carbon nuclear magnetic resonance spectra (^{13}C NMR) are reported as δ in units of parts per million (ppm) downfield from $SiMe_4$ (δ 0.0) and relative to the signal of chloroform-d (δ 77.0, triplet).

Enantiomeric excesses were determined by high performance liquid chromatography (HPLC) analysis on a chiral stationary phase, CHIRALPAK AD-H, CHIRALCEL IB, CHIRALCEL IA and CHIRALPAK OD-H. Optical rotations were measured in $CHCl_3$ on a Schmidt + Haensdchpolarimeter (Polartronic MH8) with a 10 cm cell (c given in 0.5 g/100 mL). Absolute configuration of the products was determined by X-ray

crystallography. High resolution mass spectrometry (HRMS) was recorded on QTOF perimer for ESI^+ and ESI^-.

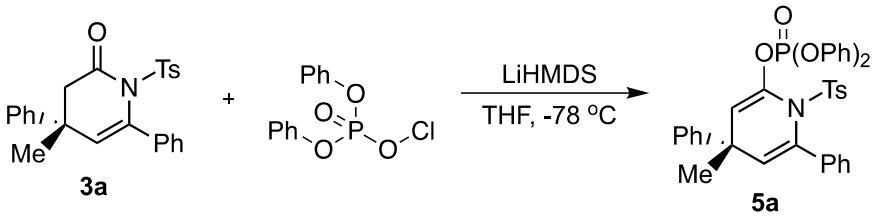
The racemic products used to determine the e.e. values were synthesized using cat D.

General procedure 1 for the catalytic synthesis of products 3a:



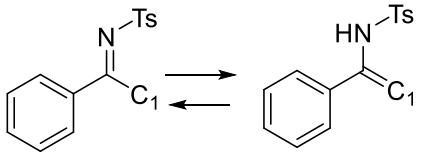
To an oven dried 10 mL vial was added 2.0 mL solvent, ketimines **2** (27.4 mg, 0.10 mmol, 1.0 equiv) and LiCl (0.10 mmol, 4.2 mg, 1.0 equiv), the mixture stirred 30 mins. Then aldehyde **1** (0.10 mmol, 18.0 mg, 1.0 equiv), cat **A** (7.4 mg, 0.02 mmol, 20 mol %), Cs_2CO_3 (38.88 mg, 0.10 mmol, 1.2 equiv) was added in the mixture. The reaction was stirred until complete disappearance of the starting material monitored by TLC. The resulting solution was concentrated under reduced pressure and the residue was subjected to column chromatography using EA/PE = 1:30 as eluent to afford the desired product **3a**. *And, used for the preparation of the corresponding racemic products for HPLC analysis was synthesized by cat **D**.*

Transformation of disubstituted 3a to trisubstituted 5a:



To a solution of compound **3a** (0.13 mmol, 53.3 mg) in THF (4 mL) at -78 °C, LiHMDS (0.22 mmol, 1 M in THF, 0.22 mL) was added dropwise. After stirring for 1.5 h, a solution of diphenyl chlorophosphate (0.19 mmol, 15.1μL) was added in THF (2.0 mL) at -78 °C. After 2 h, the reaction was quenched by the slow addition of H_2O at -78 °C. Ethyl acetate was then added, the organic layer was separated and dried ($MgSO_4$), and the solvent was evaporated under reduced pressure. The residue was purified by flash column chromatography (silica gel, EA/PE = 1:20) to afford the vinyl phosphate **5a** (50.9 mg, 61% yield) as a colorless oil.

Table 1: The conversion ratio between imine and enamine[a]



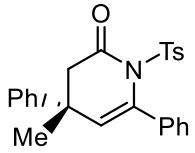
6 **7**

Entry	Ketamine	Additive	Ratios（6:7）
1	6	-	87% : 13%
2	6	LiCl	25% : 75%
3	6	LiBr	83% : 17%
4	6	^{t}BuCOOH	84% : 16%
5	6	NaCl	92% : 8%

[a] 0.05 mmol imine **6** and 0.05 mmol additive were dissolved in 1ml d-DMSO. After 15 min, absorbed 0.7 ml the mixture was detected by ^{1}H NMR. The ratios were calculated by ^{1}H peek area of C_1 in NMR.

Characterization data

(R)-4-methyl-4,6-diphenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one 3a



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:30). The compound 3a was obtained (70 % yield, 29.2 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54 (d, *J* = *7.1* Hz, 2H), 7.43 - 7.35 (m, 5H), 7.26 - 7.23 (m, 5H), 6.99 (d, *J* = 8.1 Hz, 2H), 6.03 (s, 1H), 3.05 (d, *J* = 16.3 Hz, 1H), 2.72 (d, *J* = 16.3 Hz, 1H), 2.34 (s, 3H), 1.41 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.0, 145.5, 144.4, 140.3, 137.8, 135.8, 129.2, 129.1, 129.0, 128.6, 128.5, 127.7, 127.0, 126.0, 125.8, 47.1, 39.0, 30.5, 21.7.

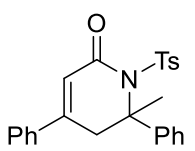
HRMS (ESI^+) calcd for $C_{25}H_{24}NO_3S$, m/z 418.1471, found 418.1482.

HPLC: Chiralcel AD (n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 14.321 min, t_R (minor) = 17.855 min; >99% ee.

$[\alpha]_D^{25}$ = -6.32 (c = 0.5, $CHCl_3$).

Melting point: 83 - 84°C.

6-methyl-4,6-diphenyl-1-tosyl-5,6-dihydropyridin-2(1H)-one 4a



The compound 4a purified by flash chromatography on silica gel (EA/PE = 1:20).

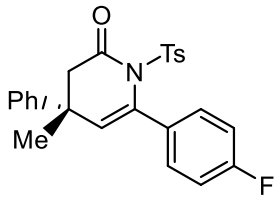
^{1}H NMR (500 MHz, $CDCl_3$) δ 7.83 (d, *J* = 8.1 Hz, 2H), 7.58 (d, *J* = 7.5 Hz, 2H), 7.46 - 7.31 (m, 8H), 7.25 - 7.26 (m, 2H), 6.28 (s, 1H), 3.28 (d, *J* = 17.3 Hz, 1H), 2.95 (d, *J* = 17.2 Hz, 1H), 2.42 (s, 3H), 2.27 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 165.0, 150.0, 145.8, 144.4, 137.9, 136.4, 130.5, 129.7, 129.1, 129.0, 128.6, 127.6, 126.1, 125.6, 118.8, 68.3, 46.8, 25.8, 21.8.

HRMS (ESI^-) calcd for $C_{25}H_{22}NO_3S$, m/z 416.1326, found 418.1315.

(R)-6-(4-fluorophenyl)-4-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3b



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:25). The compound 3b was obtained (70 % yield, 30.5 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 - 7.48 (m, 2H), 7.36 - 7.33 (m, 2H), 7.25 (s, 5H), 7.08 (t, *J* = 8.6 Hz, 2H), 6.99 (d, *J* = 8.0 Hz, 2H), 5.98 (s, 1H), 3.05 (d, *J* = 16.4 Hz, 1H), 2.71 (d, *J* = 16.4 Hz, 1H), 2.35 (s, 3H), 1.41 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.7, 162.7 (d, *J* = 247.9 Hz), 145.3, 144.3, 139.3, 135.5, 133.7 (d, *J* = 3.4 Hz), 129.0, 128.8, 127.7(d, *J* = 8.2 Hz), 127.4 126.9 125.6, 115.5 (d, *J* = 21.9 Hz), 47.0, 38.8, 30.4, 21.6.

HRMS (ESI^+) calcd for $C_{25}H_{23}FNO_3S$, m/z 436.1377, found 436.1389.

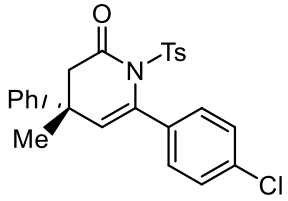
HPLC: Chiralcel AD (n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 15.469 min, t_R (minor) = 18.806 min; >99% ee.

$[\alpha]_D^{25}$ = -12.36 (c = 0.5, $CHCl_3$).

Melting point: 77 - 78°C.

(R)-6-(4-chlorophenyl)-4-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-on

3c



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:20). The compound 3c was obtained (72 % yield, 32.5 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 - 7.44 (m, 2H), 7.41 - 7.33 (m, 4H), 7.26 - 7.22 (m, 5H), 6.99 (d, *J* = 8.1 Hz, 2H), 6.01 (s, 1H), 3.05 (d, *J* = 17.2 Hz, 1H), 2.70 (d, *J* = 16.3 Hz, 1H), 2.35 (s, 3H), 1.40 (s, 3H).

^{13}C NMR (126 MHz, CDCl3) δ 170.8, 145.4, 144.5, 139.3, 136.3, 135.6, 134.3, 129.1, 129.1, 129.0, 128.8, 128.0, 127.3, 127.1, 125.7, 47.0, 39.0, 30.5, 21.8.

HRMS (ESI^+) calcd for $C_{25}H_{23}ClNO_3S$, m/z 452.1082, found 452.1097.

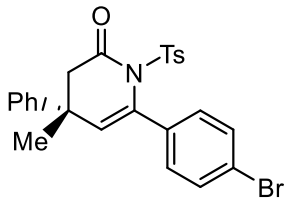
HPLC: Chiralcel AD (n-hexane/i-PrOH, 95/5, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 54.992 min, t_R (minor) = 62.529 min; >99% ee.

$[\alpha]_D^{25}$ = -5.92 (c = 0.5, $CHCl_3$).

Melting point: 80 - 81°C.

(R)-6-(4-bromophenyl)-4-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3d



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:15). The compound 3d was obtained (74 % yield, 36.5 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.55 - 7.50 (m, 2H), 7.44 - 7.40 (m, 2H), 7.39 - 7.35 (m, 2H), 7.26 - 7.22 (m, 5H), 6.99 (d, *J* = 8.1 Hz, 2H), 6.02 (s, 1H), 3.05 (d, *J* = 16.4 Hz, 1H), 2.70 (d, *J* = 16.3 Hz, 1H), 2.35 (s, 3H), 1.40 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.8, 145.3, 144.5, 139.4, 136.8, 135.5, 131.8, 129.1, 129.1, 129.0, 128.1, 127.6, 127.1, 125.7, 122.5, 47.0, 39.0, 30.5, 21.8.

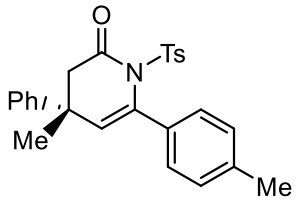
HRMS (ESI^+) calcd for $C_{25}H_{23}BrNO_3$ S, m/z 496.0577, found 496.0587.

HPLC: Chiralcel IA (n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ= 254 nm), t_R(major) = 19.656 min, t_R (minor) = 18.276 min; >99% ee.

$[\alpha]_D^{25}$ = -40.46 (c = 0.5, $CHCl_3$).

Melting point: 88 - 89°C.

(R)-4-methyl-4-phenyl-6-(p-tolyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one 3e



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:30). The compound 3e was obtained (51 % yield, 22.0 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 - 7.42 (m, 2H), 7.40 - 7.35 (m, 2H), 7.26 - 7.19 (m, 7H), 6.98 (d, *J* = 8.1 Hz, 2H), 5.99 (s, 1H), 3.04 (d, *J* = 16.3 Hz, 1H), 2.70 (d, *J* = 16.3 Hz, 1H), 2.36 (s, 3H), 2.35 (s, 3H), 1.39 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 171.3, 145.9, 144.5, 140.5, 138.7, 136.0, 135.2, 129.5, 129.3, 129.2, 129.1, 127.2, 127.1, 126.1, 126.0, 47.3, 39.1, 30.8, 21.9, 21.7.

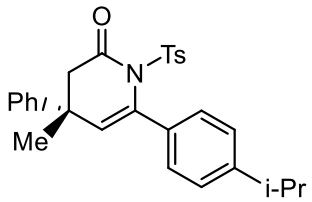
HRMS (ESI^+) calcd for $C_{26}H_{26}NO_3S$, m/z 432.1628, found 432.1634.

HPLC: Chiralcel AD n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R (major) = 16.042 min, t_R (minor) = 19.709 min; >99% ee.

$[\alpha]_D^{25}$ = -36.5 (c = 0.5, $CHCl_3$).

Melting point: 64 - 65°C.

(R)-6-(4-isopropylphenyl)-4-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one 3f



The title compound was prepared according to the GP1, purified by flash

chromatography on silica gel (EA/PE = 1:25). The compound 3f was obtained (50 % yield, 23.0 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, *J* = 8.3 Hz, 2H), 7.35 (d, *J* = 8.1 Hz, 2H), 7.28 - 7.22 (m, 7H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.00 (s, 1H), 3.04 (d, *J* = 16.4 Hz, 1H), 2.95 (p, *J* = 6.9 Hz, 1H), 2.70 (d, *J* = 16.3 Hz, 1H), 2.34 (s, 3H), 1.39 (s, 3H), 1.28 (d, *J* = 6.9 Hz, 6H).

^{13}C NMR (126 MHz, CDCl3) δ 171.1, 149.3, 145.6, 144.3, 140.3, 135.8, 135.2, 129.1, 129.0, 128.9, 127.0, 126.7, 125.9, 125.8, 47.2, 38.9, 34.0, 30.6, 24.1, 24.0, 21.7.

HRMS (ESI^+) calcd for $C_{28}H_{30}NO_3S$, m/z 460.1941, found 460.1944.

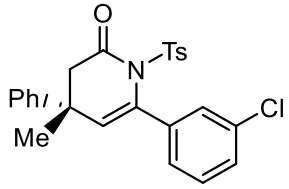
HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 11.350 min, t_R (minor) = 13.881 min; >99% ee.

$[\alpha]_D^{25}$ = -40.12 (c = 0.5, $CHCl_3$).

Melting point: 71 - 72°C.

(R)-6-(3-chlorophenyl)-4-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3g



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:20). The compound 3g was obtained (73 % yield, 33.1 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46 - 7.3 (m, 4H), 7.36 - 7.31 (m, 2H), 7.26 (s, 5H),

7.03 (d, J = 8.2 Hz, 2H), 6.05 (s, 1H), 3.06 (d, J = 17.1 Hz, 1H), 2.71 (d, J = 16.4 Hz, 1H), 2.36 (s, 3H), 1.43 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 169.7, 144.0, 143.5, 138.3, 137.9, 134.4, 133.4, 128.7, 128.0, 127.9, 127.9, 127.5, 127.4, 126.0, 124.9, 124.6, 123.2, 46.0, 37.8, 29.0, 20.6.

HRMS (ESI^+) calcd for $C_{25}H_{23}ClNO_3S$, m/z 452.1082, found 452.1091.

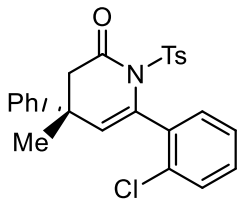
HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 11.522 min, t_R (minor) = 13.586 min; 98% ee.

$[\alpha]_D^{25}$ = -14.76 (c = 0.5, $CHCl_3$).

Melting point: 71 - 72°C.

(R)-6-(2-chlorophenyl)-4-methyl-4-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3h



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:15). The compound 3h was obtained (83 % yield, 37.4 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (m, 1H), 7.45 - 7.19 (m, 10H), 7.02 (d, J = 8.1 Hz, 2H), 5.99 (s, 1H), δ 3.08 (d, J = 16.2 Hz, 1H), 2.78 (d, J = 16.3 Hz, 1H), 2.35 (s, 3H), 1.46 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.8, 144.7, 135.9, 135.7, 133.0, 130.8, 130.3, 129.8, 129.4, 129.4, 129.4, 129.2, 129.1, 128.9, 127.3, 127.0, 126.0, 47.9, 39.2, 30.0, 22.0.

HRMS (ESI$^+$) calcd for $C_{25}H_{23}ClNO_3S$, m/z 452.1082, found 452.1099.

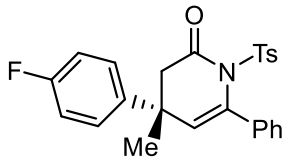
HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R (major) = 12.647 min, t_R (minor) = 19.282 min; >99% ee.

$[\alpha]_D^{25}$ = -4.12 (c = 0.5, $CHCl_3$).

Melting point: 62 - 63°C.

(R)-4-(4-fluorophenyl)-4-methyl-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3i



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:25). The compound 3i was obtained (53 % yield, 23.1 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 - 7.49 (m, 2H), 7.46 - 7.30 (m, 5H), 7.23 - 7.14 (m, 2H), 7.06 - 6.99 (m, 2H), 6.88 (t, *J* = 8.6 Hz, 2H), 6.01 (s, 1H), 3.01 (d, *J* = 17.3 Hz, 1H), 2.72 (d, *J* = 16.2 Hz, 1H), 2.37 (s, 3H), 1.38 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.9, 161.9 (d, *J* = 245.8 Hz), 144.7, 141.2 (d, *J* = 3.2 Hz), 140.7, 137.7, 135.6, 129.2, 128.8, 128.6, 127.5 (d, *J* = 5.5 Hz), 127.4, 125.9, 115.8 (d, *J* = 21.3 Hz), 47.2, 38.5, 30.8, 21.7.

HRMS (ESI$^+$) calcd for $C_{25}H_{23}FNO_3S$, m/z 436.1377, found 436.1390.

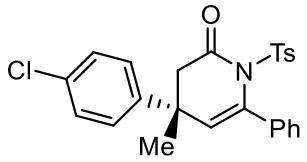
HPLC: Chiralcel AD n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 15.330 min, t_R (minor) = 21.545 min; >99% ee.

$[\alpha]_D^{25}$ = -32.48 (c = 0.5, $CHCl_3$).

Melting point: 169 - 170°C.

(R)-4-(4-chlorophenyl)-4-methyl-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3j



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:20). The compound 3j was obtained (52 % yield, 23.5 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.61 - 7.51 (m, 2H), 7.46 - 7.32 (m, 5H), 7.14 (s, 4H), 7.03 (d, *J* = 8.0 Hz, 2H), 6.02 (s, 1H), 3.00 (d, *J* = 16.2 Hz, 1H), 2.72 (d, *J* = 16.2 Hz, 1H),, 2.39 (s, 3H), 1.37 (s, 3H).

^{13}C NMR (126 MHz, CDCl3) δ 170.6, 150.9, 144.6, 144.0, 140.8, 137.6, 135.3, 132.9, 129.1, 128.9, 128.7, 128.5, 127.1, 127.0, 125.7, 46.6, 38.5, 30.8, 21.6.

HRMS (ESI^+) calcd for $C_{25}H_{23}ClNO_3S$, m/z 452.1082, found 452.1091.

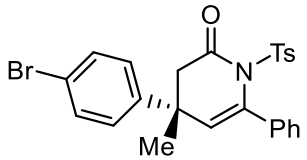
HPLC: Chiralcel AD n-hexane/i-PrOH, 90/10, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 28.532 min, t_R (minor) = 35.038 min; 98% ee.

$[\alpha]_D^{25}$ = -13.94 (c = 0.5, $CHCl_3$).

Melting point: 172 - 173°C.

(R)-4-(4-bromophenyl)-4-methyl-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3k



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:20). The compound 3k was obtained (51 % yield, 25.3 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 - 7.52 (m, 2H), 7.46 - 7.33 (m, 5H), 7.32 - 7.26 (m, 2H), 7.14 - 6.98 (m, 4H), 6.01 (s, 1H), 2.99 (d, *J* = 17.1 Hz, 1H), 2.71 (d, *J* = 16.3 Hz, 1H)., 2.40 (s, 3H), 1.36 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.9, 145.0, 144.9, 141.1, 137.9, 135.6, 132.5, 132.2, 129.4, 129.0, 128.9, 127.8, 127.2, 126.0, 121.4, 46.9, 38.9, 31.1, 22.0.

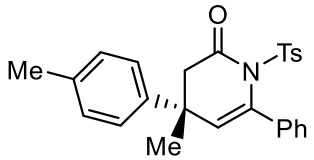
HRMS (ESI^+) calcd for $C_{25}H_{23}BrNO_3S$, m/z 496.0577, found 496.0585.

HPLC: Chiralcel IA n-hexane/i-PrOH, 70/30, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 11.573 min, t_R (minor) = 13.132 min; >99% ee.

$[\alpha]_D^{25}$ = -21.62 (c = 0.5, $CHCl_3$).

Melting point: 179 - 180°C.

(R)-4-methyl-6-phenyl-4-(p-tolyl)-1-tosyl-3,4-dihydropyridin-2(1H)-one 3l



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:25). The compound 3l was obtained (55 %

yield, 23.7 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 - 7.50 (m, 2H), 7.49 - 7.29 (m, 5H), 7.16 - 7.09 (m, 2H), 7.03 (d, *J* = 7.9 Hz, 2H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.02 (s, 1H), 3.02 (d, *J* = 16.2 Hz, 1H), 2.69 (d, *J* = 16.2 Hz, 1H), 2.36, (s, 3H), 2.35 (s, 3H), 1.38 (s, 3H).

^{13}C NMR (126 MHz, CDCl3) δ 171.0, 144.1, 142.4, 140.1, 137.8, 136.5, 135.7, 129.5, 129.1, 128.6, 128.5, 128.3, 127.8, 125.8, 125.5, 47.0, 38.5, 30.5, 21.6, 21.1.

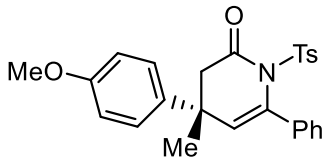
HRMS (ESI^+) calcd for $C_{26}H_{26}NO_3S$, m/z 432.1628, found 432.1647.

HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 14.480 min, t_R (minor) = 11.373 min; >99% ee.

$[\alpha]_D^{25}$ = 19.18 (c = 0.5, $CHCl_3$).

Melting point: 133 - 134°C.

(R)-4-(4-methoxyphenyl)-4-methyl-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one 3m



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:20). The compound 3m was obtained (60 % yield, 26.8 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 - 7.51 (m, 2H), 7.45 - 7.32 (m, 5H), 7.19 - 7.12 (m, 2H), 6.99 (d, *J* = 8.1 Hz, 2H), 6.75 (d, *J* = 8.8 Hz, 2H), 6.01 (s, 1H), 3.82 (s, 3H), , 3.00 (d, *J* = 16.3 Hz, 1H), 2.69 (d, *J* = 16.2 Hz, 1H), 2.35 (s, 3H), 1.37 (s, 3H).

^{13}C NMR (126 MHz, CDCl3) δ 170.9, 158.5, 144.1, 140.1, 137.8, 137.3, 135.7, 129.1, 128.7, 128.5, 128.4, 127.8, 126.7, 125.8, 114.2, 55.2, 47.1, 38.2, 30.6, 21.6.

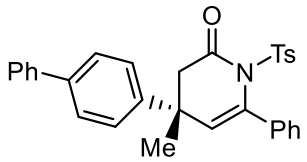
HRMS (ESI$^+$) calcd for $C_{26}H_{26}NO_4S$, m/z 448.1577, found 448.1592.

HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 11.699 min, t_R (minor) = 12.965 min; >99% ee.

$[\alpha]_D^{25}$ = 14.9 (c = 0.5, $CHCl_3$).

Melting point: 89 - 90°C.

(R)-4-([1,1'-biphenyl]-4-yl)-4-methyl-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one 3n



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:20). The compound 3n was obtained (71 % yield, 35.0 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 - 7.56 (m, 4H), 7.51 - 7.35 (m, 10H), 7.33 - 7.28 (m, 2H), 6.88 (d, *J* = 8.1 Hz, 2H), 6.07 (s, 1H), 3.08 (d, *J* = 17.1 Hz, 1H), 2.75 (d, *J* = 16.2 Hz, 1H), 2.12 (s, 3H), 1.44 (s, 3H).

^{13}C NMR (126 MHz, CDCl3) δ 170.8, 144.5, 144.2, 140.4, 140.2, 139.7, 137.8, 135.6, 129.0, 129.0, 128.6, 128.5, 128.4, 127.6, 127.4, 127.3, 126.8, 126.1, 125.8, 46.9, 38.6, 30.6, 21.3.

HRMS (ESI$^+$) calcd for $C_{31}H_{28}NO_3S$, m/z 494.1784, found 494.1790.

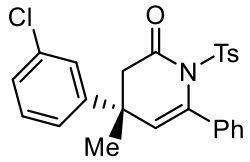
HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R (major) = 20.884 min, t_R (minor) = 17.457 min; >99% ee.

$[\alpha]_D^{25}$ = 25.76 (c = 0.5, $CHCl_3$).

Melting point: 155 - 156°C.

(R)-4-(3-chlorophenyl)-4-methyl-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3o



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:25). The compound 3o was obtained (41 % yield, 18.5 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ7.58 - 7.51 (m, 2H), 7.48 - 7.34 (m, 5H), 7.24 - 7.09 (m, 4H), 7.01 (d, *J* = 8.1 Hz, 2H), 5.99 (s, 1H), 3.00 (d, *J* = 16.4 Hz, 1H), 2.72 (d, *J* = 16.4 Hz, 1H), 2.37 (s, 3H), 1.39 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.7, 148.0, 145.0, 141.2, 137.9, 135.6, 135.2, 130.5, 129.4, 129.1, 128.9, 128.9, 128.8, 127.5, 126.9, 126.1, 124.5, 47.0, 39.1, 30.8, 22.0.

HRMS (ESI^+) calcd for $C_{25}H_{23}ClNO_3S$, m/z 452.1082, found 452.1090.

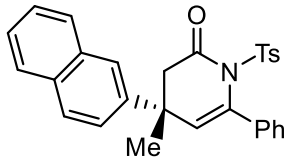
HPLC: Chiralcel AD n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R (major) = 20.934 min, t_R (minor) = 23.993 min; >99% ee.

$[\alpha]_D^{25}$ = -14.54 (c = 0.5, $CHCl_3$).

Melting point: 160 - 161°C.

(R)-4-methyl-4-(naphthalen-2-yl)-6-phenyl-1-tosyl-3,4-dihydropyridin-2(1H)-one

3p



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:30). The compound 3p was obtained (45 % yield, 21.1 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (d, *J* = 7.9 Hz, 1H), 7.78 (d, *J* = 8.6 Hz, 1H), 7.66 - 7.62 (m, 2H), 7.59 (d, *J* = 7.9 Hz, 1H), 7.53 - 7.37 (m, 7H), 7.15 - 7.06 (m, 2H), 6.27 (d, *J* = 8.0 Hz, 2H), 6.16 (s, 1H), 3.20 (d, *J* = 16.3 Hz, 1H), 2.80 (d, *J* = 16.3 Hz, 1H), 1.99 (s, 3H), 1.45 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ 170.9, 144.2, 143.1, 140.8, 138.0, 135.2, 133.6, 132.6, 128.9, 128.9, 128.7, 128.6, 128.3, 128.2, 127.6, 127.4, 126.5, 126.2, 125.8, 124.8, 123.7, 46.4, 39.0, 31.1, 21.5.

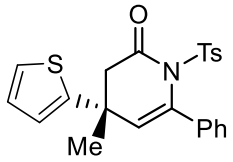
HRMS (ESI^+) calcd for $C_{29}H_{26}NO_3S$, m/z 468.1633, found 468.1636.

HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 12.904 min, t_R (minor) = 14.444 min; >99% ee.

$[\alpha]_D^{25}$ = 25.22 (c = 0.5, $CHCl_3$).

Melting point: 168 - 169°C.

(R)-4-methyl-6-phenyl-4-(thiophen-2-yl)-1-tosyl-3,4-dihydropyridin-2(1H)-one 3q



The title compound was prepared according to the GP1, purified by flash chromatography on silica gel (EA/PE = 1:30). The compound 3q was obtained (42 % yield, 17.8 mg), as a white solid.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.53 (d, *J* = 8.4 Hz, 2H), 7.50 - 7.45 (m, 2H), 7.41 - 7.34 (m, 3H), 7.18 - 7.19 (m, 1H), 7.12 (d, *J* = 8.1 Hz, 2H), 6.84 - 6.85 (m, 1H), 6.79 - 6.80 (m, 1H), 5.94 (s, 1H), 2.98 (d, *J* = 17.0 Hz, 1H), 2.77 (d, *J* = 16.4 Hz, 1H), 2.38 (s, 3H), 1.51 (s, 3H)

^{13}C NMR (126 MHz, CDCl3) δ 170.4, 150.1, 144.5, 140.6, 137.4, 135.8, 129.2, 128.9, 128.5, 128.4, 127.0, 126.3, 126.0, 124.1, 123.3, 48.7, 37.2, 30.5, 21.7.

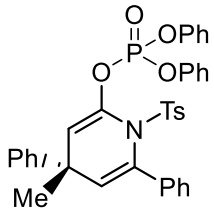
HRMS (ESI Na^+) calcd for $C_{23}H_{21}NO_3SNa$, m/z 446.0855, found 446.0857.

HPLC: Chiralcel IA n-hexane/i-PrOH, 80/20, flow rate 1.0 mL/min, λ = 254 nm), t_R(major) = 18.588 min, t_R (minor) = 13.255 min; 98% ee.

$[\alpha]_D^{25}$ = -10.79 (c = 0.5, $CHCl_3$).

Melting point: 171 - 172°C.

4-methyl-4,6-diphenyl-1-tosyl-1,4-dihydropyridin-2-yl diphenyl phosphate 5a



The title compound was purified by flash chromatography on silica gel (EA/PE = 1:20).

The compound 5a was obtained (61 % yield, 50.9 mg), as a colourless oil.

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 - 7.32 (m, 8H), 7.31 - 7.16 (m, 14H), 6.95 (d, *J* = 8.0 Hz, 2H), 5.65 (s, 1H), 5.56 (s, 1H), 2.29 (s, 3H), 1.37 (s, 3H).

^{13}C NMR (126 MHz, $CDCl_3$) δ151.0 (dd, *J* = 7.4, 1.9 Hz), 148.1, 144.9, 140.6 (d, *J* = 6.7 Hz), 138.5, 137.5, 135.3, 130.5, 129.6, 129.2, 129.1, 128.8, 128.6, 128.0, 127.7, 126.8, 126.5, 126.2, 120.8 (dd, *J* = 7.6, 5.0 Hz), 112.3 (d, *J* = 3.7 Hz), 42.0, 42.0, 22.2.

^{31}P NMR (202 MHz, $CDCl_3$) δ -18.4.

HRMS (ESI+) calcd for $C_{37}H_{33}NO_6PS$, m/z 650.1761, found 650.1758.

HPLC: Chiralcel AD-H n-hexane/i-PrOH, 75/25, flow rate 1.0 mL/min, λ = 254 nm), t_R (major) = 34.309 min, t_R (minor) = 28.093 min; >99% ee.

$[\alpha]_D^{25}$ = -11.34 (c = 0.5, $CHCl_3$).

Computational details

To explore the origins of stereoselectivity, density-functional theory (DFT) calculations were performed with Gaussian 09 package.[S1] The geometries were fully optimized at wB97xd/6-31G(d) level of theory with the SMD implicit solvation model to account for the solvation effects of diethylether. All of the optimized geometries were verified by frequency computations as minima (zero imaginary frequencies) or transition structures (a single imaginary frequency). Single-point energies of the optimized geometries were evaluated using the same accurate density functional and a larger 6-311++G(d,p) basis set. The free energies were corrected using Truhlar's quasi harmonic correction, by raising vibrational frequencies that are below 100 cm^{-1}.

S1. Gaussian 09, Revision A.2, Frisch, M. J.; Trucks, G. W.; Schlegel, H. B.; Scuseria, G. E.; Robb, M. A.; Cheeseman, J. R.; Scalmani, G.; Barone, V.; Mennucci, B.; Petersson, G. A.; Nakatsuji, H.; Caricato, M.; Li, X.; Hratchian, H. P.; Izmaylov, A. F.; Bloino, J.; Zheng, G.; Sonnenberg, J. L.; Hada, M.; Ehara, M.; Toyota, K.; Fukuda, R.; Hasegawa, J.; Ishida, M.; Nakajima, T.; Honda, Y.; Kitao, O.; Nakai, H.; Vreven, T.; Montgomery, Jr., J. A.; Peralta, J. E.; Ogliaro, F.; Bearpark, M.; Heyd, J. J.; Brothers, E.; Kudin, K.
N.; Staroverov, V. N.; Kobayashi, R.; Normand, J.; Raghavachari, K.; Rendell, A.; Burant, J. C.; Iyengar, S. S.; Tomasi, J.; Cossi, M.; Rega, N.; Millam, N. J.; Klene, M.; Knox, J. E.; Cross, J. B.; Bakken, V.; Adamo, C.; Jaramillo, J.; Gomperts, R.; Stratmann, R. E.; Yazyev, O.; Austin, A. J.; Cammi, R.; Pomelli, C.; Ochterski, J. W.; Martin, R. L.; Morokuma, K.; Zakrzewski, V. G.; Voth, G. A.; Salvador, P.; Dannenberg, J. J.; Dapprich, S.; Daniels, A. D.; Farkas, Ö.; Foresman, J. B.; Ortiz, J. V.; Cioslowski, J.; Fox, D. J. Gaussian, Inc., Wallingford CT, 2009

Cartesian coordinates (in Å) of related structures

enone

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enamine

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O	2.95167100	0.20738000	-1.22806100
O	2.91661000	-1.62250400	0.51302700
H	0.46099800	-1.13457800	0.62860200
C	2.78210600	0.88855000	1.30680400
H	2.41795900	0.49892100	2.25938600
H	3.86157400	1.05222300	1.35083600
H	2.27188700	1.82013500	1.05245400
C	-1.51375200	0.20257800	-0.15801600
C	-1.97188800	-1.08709000	-0.45373800
C	-2.41491000	1.14041000	0.35709400
C	-3.30597400	-1.42487300	-0.24809700
H	-1.28836600	-1.82281800	-0.86856600
C	-3.74814700	0.80027900	0.56030200
H	-2.06313000	2.13535800	0.61426200
C	-4.19784700	-0.48321100	0.25852400
H	-3.64912200	-2.42612300	-0.49182600
H	-4.43577900	1.53757900	0.96419900
H	-5.23854500	-0.74811400	0.42042900

TSa

C	-2.88740000	-1.15760000	-0.48200000
C	-3.77980000	-1.89190000	-1.51070000
N	-1.69250000	-1.96870000	-0.22610000
C	-2.73880000	-4.00520000	-1.20740000
H	-2.92910000	-4.97240000	-0.73840000
H	-2.44570000	-4.18140000	-2.25310000
C	-1.60750000	-3.30170000	-0.52030000
C	-0.47170000	-1.57760000	0.17860000
N	0.28010000	-2.67640000	0.16930000
N	-0.41570000	-3.76430000	-0.27010000
O	-3.91990000	-3.25330000	-1.12570000
C	-0.05220000	-0.17640000	0.57970000
C	1.19160000	0.18690000	0.06100000
O	-0.84380000	0.43750000	1.32400000
H	-2.53770000	-0.21170000	-0.90180000

H	-3.38820000	-1.81260000	-2.53200000
H	1.62690000	-0.49270000	-0.65540000
C	1.96670000	1.33220000	0.42740000
C	3.42830000	1.19230000	0.03940000
C	4.44640000	1.52290000	0.93740000
C	3.79270000	0.67280000	-1.21130000
C	5.78530000	1.31610000	0.60830000
H	4.21140000	1.93070000	1.91360000
C	5.12600000	0.46960000	-1.54130000
H	3.02780000	0.42460000	-1.94160000
C	6.13170000	0.78560000	-0.62890000
H	6.55670000	1.57250000	1.32850000
H	5.38060000	0.06210000	-2.51530000
H	7.17490000	0.62370000	-0.88360000
C	1.71680000	1.92220000	1.80540000
H	0.65140000	2.01640000	2.00230000
H	2.19360000	2.89790000	1.93080000
H	2.12780000	1.23650000	2.55540000
C	0.10770000	2.95900000	-0.62330000
C	1.48910000	2.80550000	-0.76600000
H	2.10600000	3.59500000	-0.34950000
H	1.86100000	2.38250000	-1.69100000
N	-0.75830000	2.14850000	-1.29780000
S	-0.57000000	1.45240000	-2.84970000
O	0.71200000	0.78390000	-2.94480000
O	-1.80710000	0.70880000	-3.02080000
H	-1.74730000	2.26080000	-1.08720000
C	-0.55600000	2.87460000	-3.91200000
H	-1.49690000	3.41450000	-3.79290000
H	-0.44880000	2.51110000	-4.93730000
H	0.29660000	3.50330000	-3.64470000
C	-5.14940000	-1.24690000	-1.30230000
H	-5.22170000	-0.32520000	-1.89250000
C	1.66160000	-2.78670000	0.55760000
C	1.98280000	-2.66650000	1.91000000
C	2.61930000	-2.93280000	-0.45160000
C	3.33710000	-2.67170000	2.24330000
C	3.95470000	-2.93550000	-0.06310000
C	4.33190000	-2.79230000	1.27460000
H	3.61770000	-2.57200000	3.28890000
H	4.72320000	-3.02950000	-0.82620000
C	2.22930000	-3.03560000	-1.90200000
H	1.69320000	-3.96870000	-2.10600000
H	3.11770000	-3.00880000	-2.53800000

H	1.57470000	-2.21050000	-2.20770000
C	5.78960000	-2.74310000	1.64710000
H	6.35080000	-3.54680000	1.15880000
H	5.93210000	-2.83340000	2.72760000
H	6.23120000	-1.79250000	1.32540000
C	0.92340000	-2.50590000	2.96780000
H	0.10610000	-3.22290000	2.83320000
H	0.49080000	-1.49830000	2.95300000
H	1.35140000	-2.66440000	3.96080000
C	-3.83980000	-0.93830000	0.67670000
C	-5.97070000	-0.51340000	2.38630000
C	-3.58270000	-0.71600000	2.02150000
C	-5.14580000	-0.95420000	0.18030000
C	-6.21990000	-0.73470000	1.03130000
C	-4.66570000	-0.50890000	2.87700000
H	-2.56390000	-0.68410000	2.39020000
H	-7.23820000	-0.75210000	0.65250000
H	-4.48790000	-0.34160000	3.93510000
H	-6.80170000	-0.35190000	3.06680000
H	-5.95090000	-1.91820000	-1.62010000
C	-0.48510000	3.88150000	0.36310000
C	-1.59250000	3.51320000	1.14210000
C	0.07500000	5.15610000	0.52300000
C	-2.12920000	4.41160000	2.05500000
H	-1.98260000	2.50100000	1.09110000
C	-0.47070000	6.05040000	1.43600000
H	0.91990000	5.46030000	-0.08680000
C	-1.57310000	5.68080000	2.20250000
H	-2.97400000	4.11140000	2.66700000
H	-0.03430000	7.03810000	1.54610000
H	-1.99310000	6.37870000	2.92040000

TSb

N	0.16880000	-2.49160000	0.71830000
C	0.88080000	-1.39620000	0.47600000
N	2.14710000	-1.70170000	0.81930000
C	0.38890000	-0.14420000	-0.21540000
C	0.79210000	1.09010000	0.29750000
O	-0.32130000	-0.34020000	-1.22270000
H	1.21230000	1.13010000	1.29610000
C	0.60710000	2.30330000	-0.43870000
C	1.14080000	3.53920000	0.24960000
C	1.86730000	4.50630000	-0.45220000

C	0.93650000	3.73180000	1.62330000
C	2.38740000	5.62200000	0.20030000
H	2.05350000	4.39470000	-1.51390000
C	1.44950000	4.84730000	2.27290000
H	0.34570000	3.01550000	2.18600000
C	2.18140000	5.79820000	1.56370000
H	2.95630000	6.35390000	-0.36560000
H	1.27180000	4.97740000	3.33640000
H	2.58430000	6.67020000	2.07010000
C	0.87450000	2.24110000	-1.93620000
H	0.39920000	1.36920000	-2.37900000
H	0.53140000	3.14020000	-2.45370000
H	1.95630000	2.15220000	-2.08910000
C	-2.14140000	1.55720000	-0.57500000
C	-1.36050000	2.71410000	-0.45010000
H	-1.42040000	3.44110000	-1.25330000
H	-1.31770000	3.15550000	0.53810000
N	-2.39140000	0.82100000	0.56210000
S	-2.86630000	1.47480000	2.06600000
O	-1.73600000	2.15970000	2.66760000
O	-3.49360000	0.35940000	2.75010000
H	-2.76680000	-0.11840000	0.44890000
C	-4.11260000	2.66930000	1.63910000
H	-4.93580000	2.15310000	1.14190000
H	-4.45280000	3.11140000	2.57880000
H	-3.69000000	3.44290000	0.99660000
C	-2.77380000	1.10090000	-1.82580000
C	-3.85920000	0.21220000	-1.79800000
C	-2.33210000	1.57650000	-3.06860000
C	-4.45840000	-0.21290000	-2.97730000
H	-4.26950000	-0.14480000	-0.85800000
C	-2.93210000	1.14930000	-4.24450000
H	-1.50760000	2.27620000	-3.12500000
C	-3.99320000	0.24730000	-4.20530000
H	-5.29680000	-0.90130000	-2.93360000
H	-2.56770000	1.52300000	-5.19630000
H	-4.46050000	-0.08730000	-5.12630000
N	0.93660000	-3.49520000	1.23040000
C	-1.25040000	-2.66910000	0.57000000
C	-2.04920000	-2.39370000	1.68230000
C	-1.74980000	-3.12220000	-0.65180000
C	-3.42650000	-2.53600000	1.52330000
C	-3.13410000	-3.24840000	-0.75690000
C	-3.98290000	-2.95290000	0.31100000

H	-4.07670000	-2.31130000	2.36350000
H	-3.55760000	-3.58440000	-1.69980000
C	-0.83980000	-3.43730000	-1.80500000
H	-0.02140000	-4.09870000	-1.49960000
H	-0.40340000	-2.51260000	-2.19560000
H	-1.39130000	-3.92900000	-2.61020000
C	-5.47600000	-3.09990000	0.17430000
H	-6.00250000	-2.26990000	0.65720000
H	-5.81960000	-4.02340000	0.65500000
H	-5.78070000	-3.13960000	-0.87560000
C	-1.43860000	-1.95840000	2.98700000
H	-0.91520000	-1.00110000	2.88290000
H	-0.71390000	-2.69820000	3.34490000
H	-2.20710000	-1.83150000	3.75060000
C	2.13530000	-2.99070000	1.28070000
C	3.39060000	-3.61200000	1.80870000
H	3.42170000	-4.67340000	1.55860000
H	3.40560000	-3.51090000	2.90430000
C	3.37380000	-0.90000000	0.69160000
H	3.17760000	0.07560000	1.14130000
O	4.50520000	-2.99220000	1.22140000
C	4.53600000	-1.59240000	1.44970000
H	4.50140000	-1.38720000	2.52710000
C	5.78480000	-1.04630000	0.75550000
H	6.10300000	-0.11380000	1.23610000
H	6.60850000	-1.76110000	0.82760000
C	5.31330000	-0.79970000	-0.65730000
C	6.06800000	-0.62920000	-1.81030000
C	3.91950000	-0.75150000	-0.71470000
C	5.41040000	-0.41810000	-3.02170000
H	7.15290000	-0.67240000	-1.77150000
C	3.25750000	-0.54470000	-1.91790000
C	4.01750000	-0.38150000	-3.07610000
H	5.98680000	-0.29100000	-3.93330000
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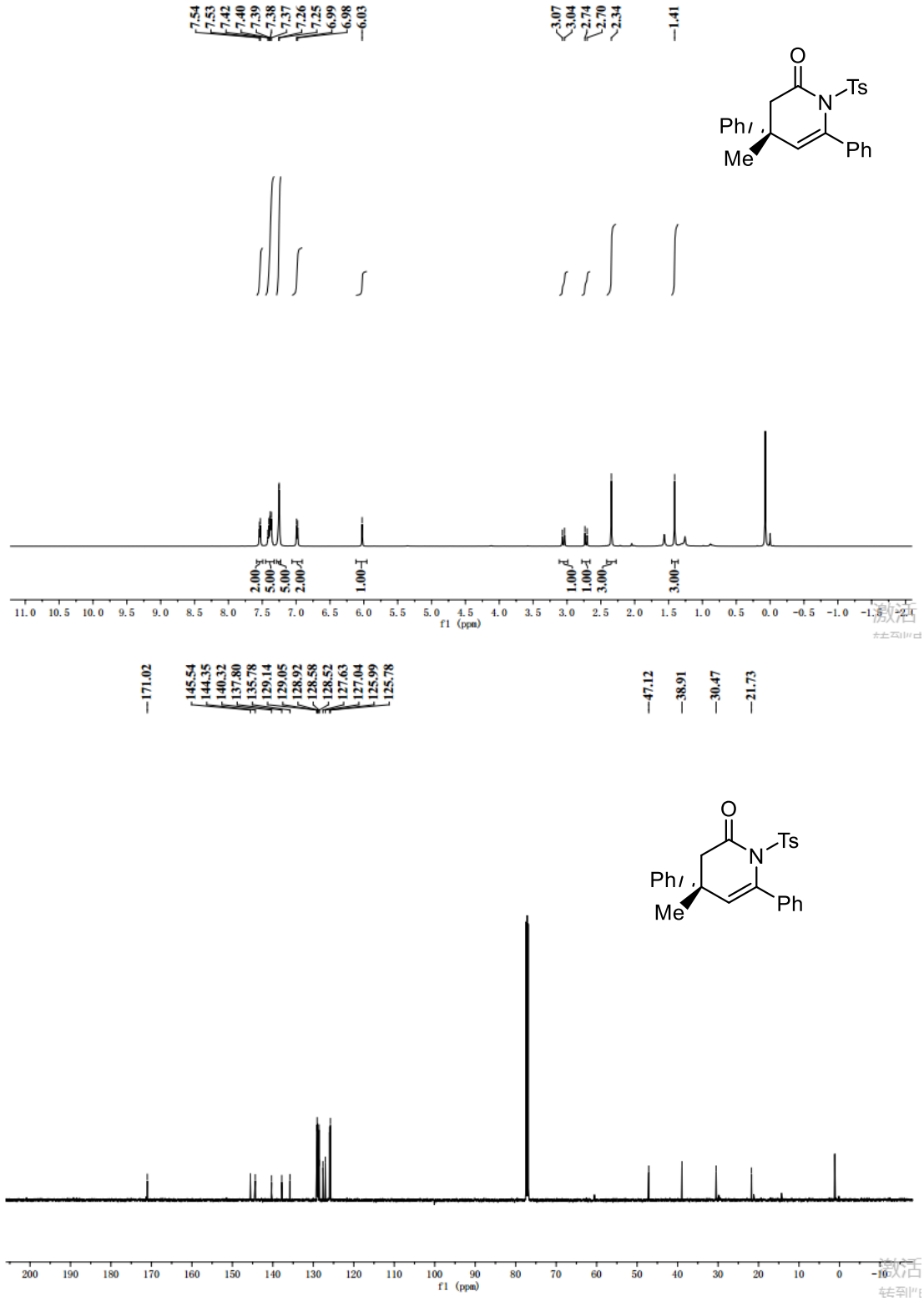
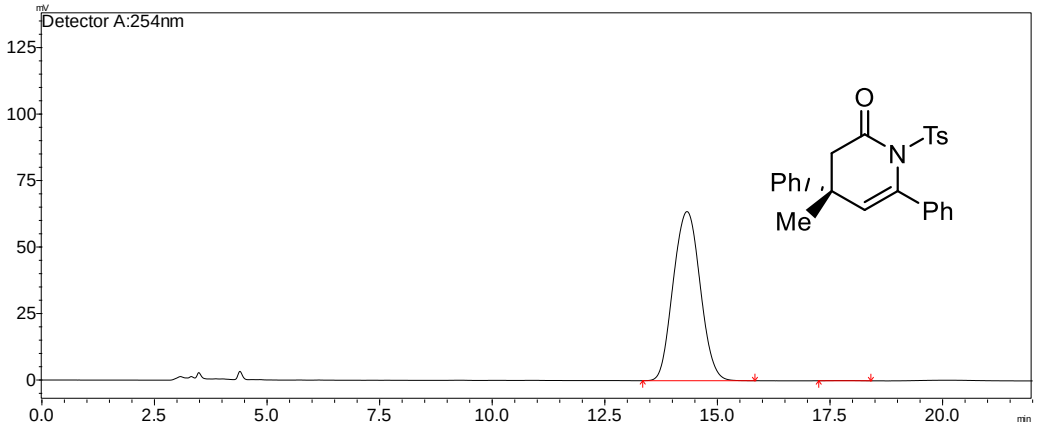
H	3.51670000	-0.22900000	-4.02730000

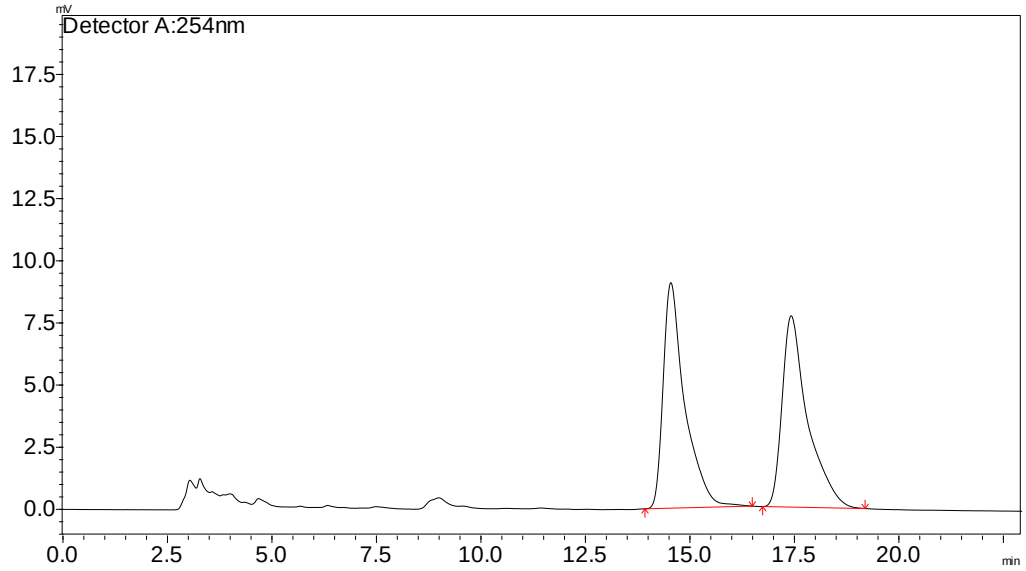
NMR and HPLC spectra

3a



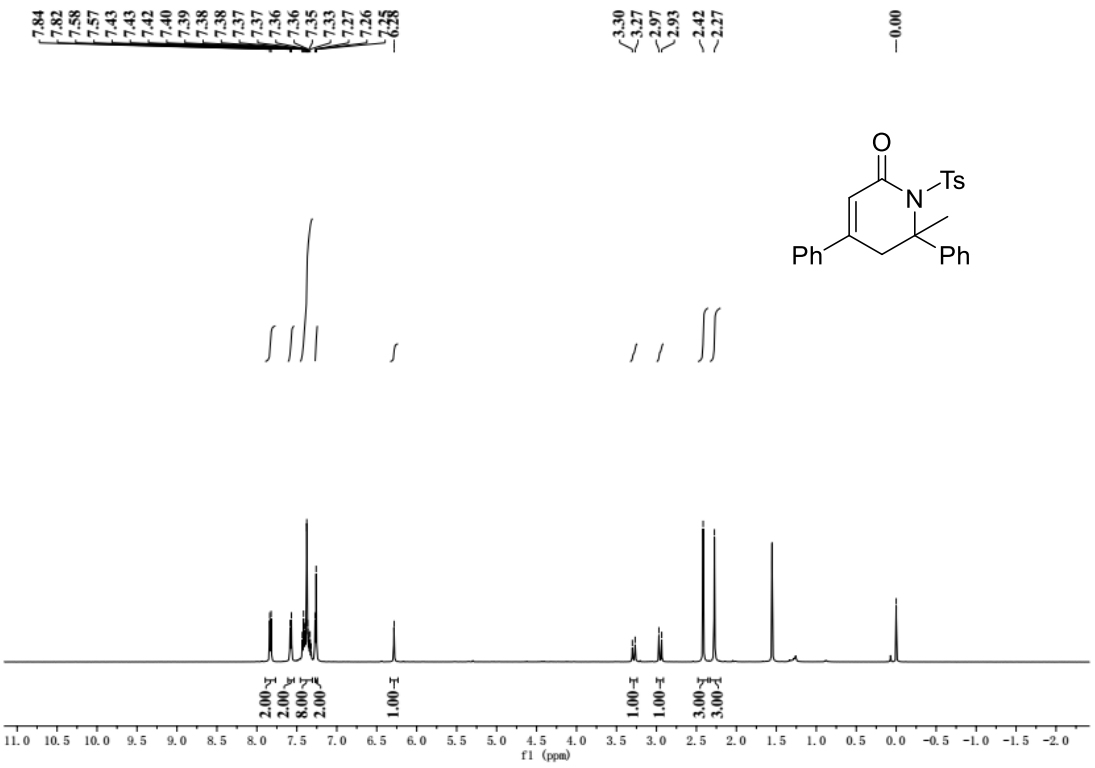


Peak	Ret .Time	Area	Height	Area%	Height%
1	14.321	26451478	63583	99.870	99.870
2	17.855	3449	83	0.130	0.130
Total		2654928	63666	100.000	100.000



Peak	Ret .Time	Area	Height	Area%	Height%
1	14.539	326269	9037	50.026	54.099
2	17.419	325934	7698	49.974	45.901
Total		652203	16771	100.000	100.000

4a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3.27
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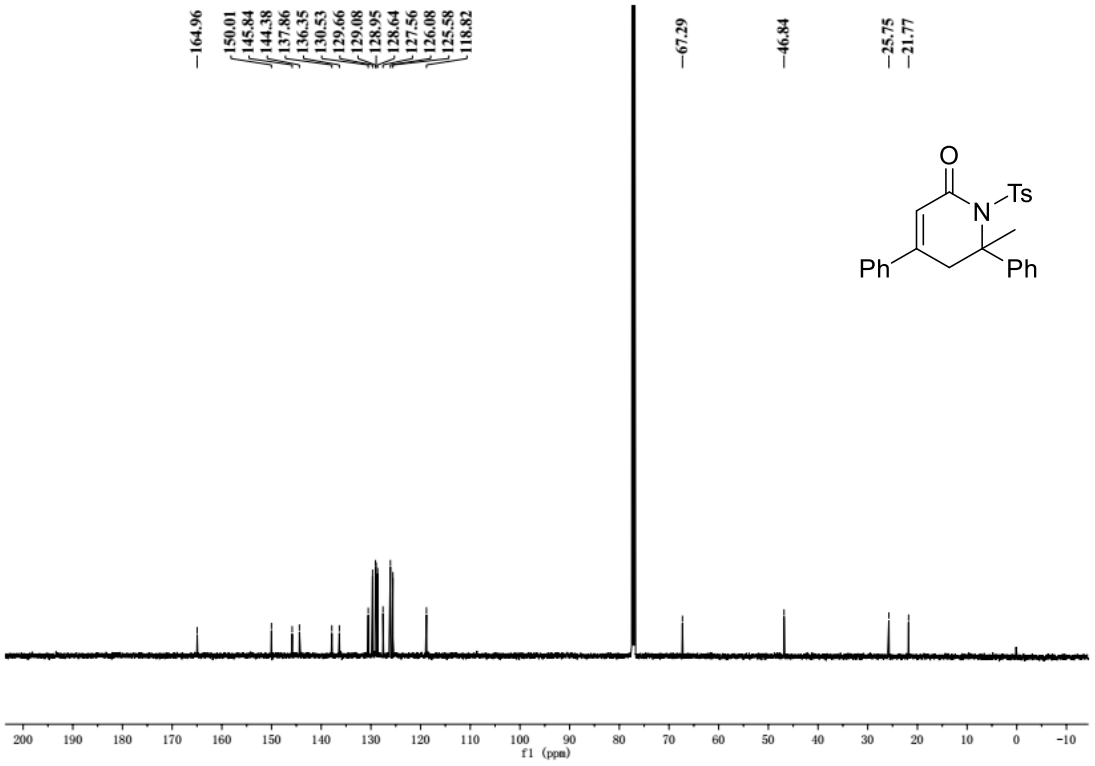
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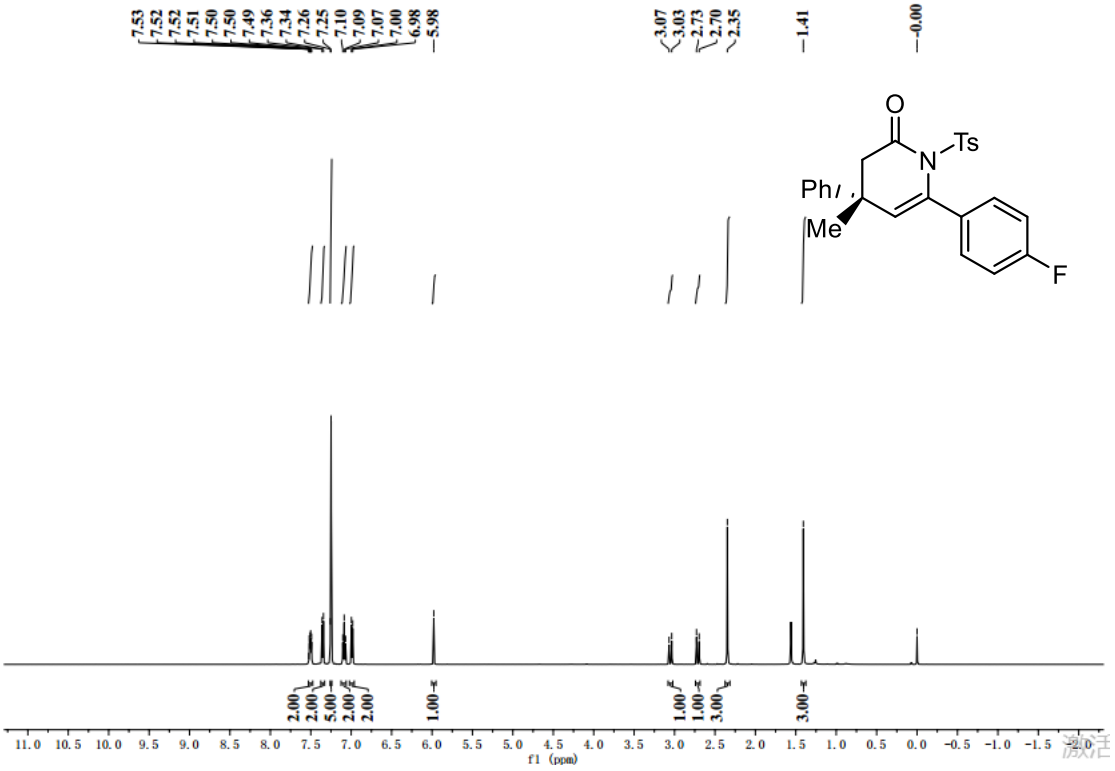
O
N
Ts
Ph
Ph
f1 (ppm)

3b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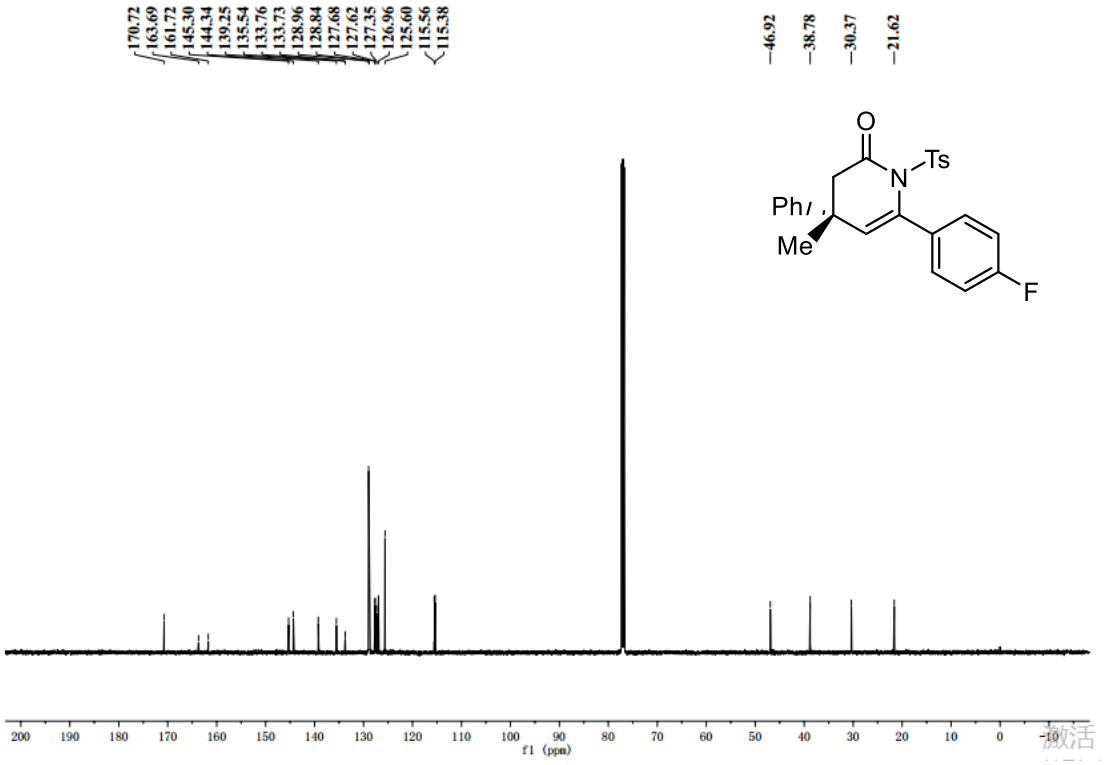
f1 (ppm)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127.35
126.96
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46.92
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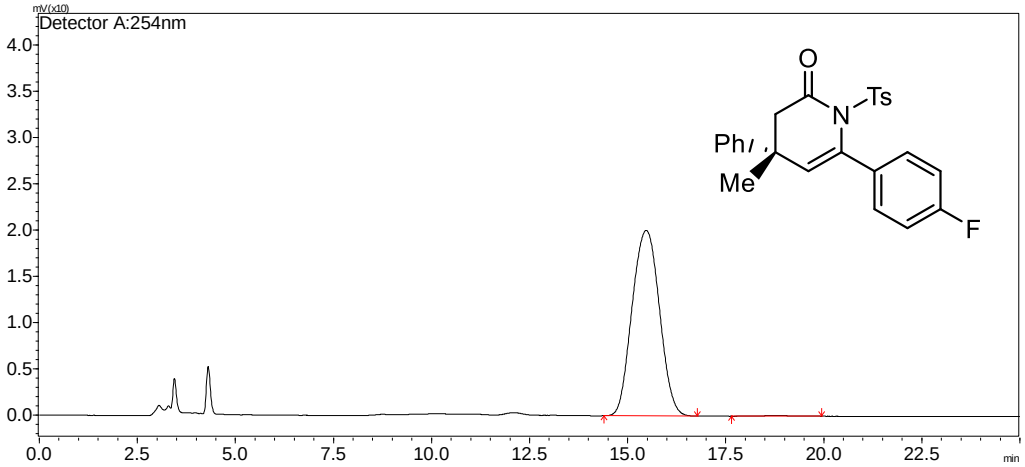
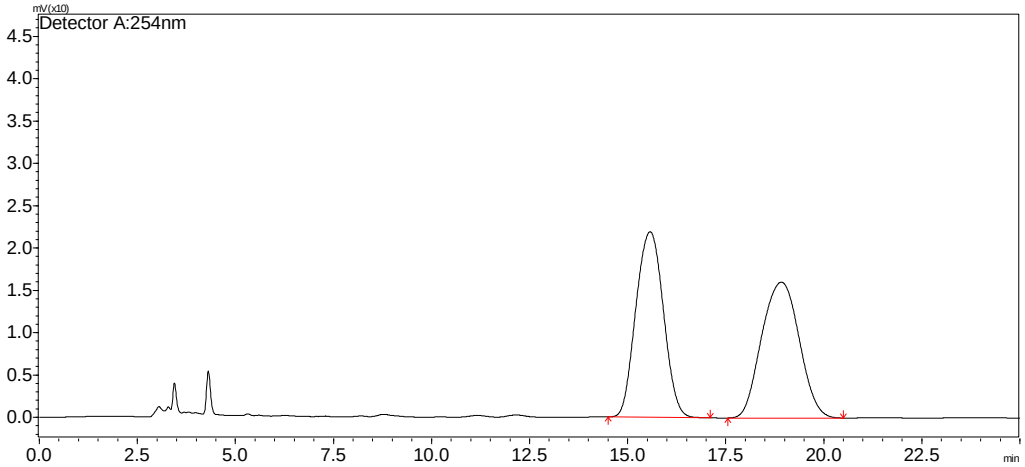
30.37
21.62
O
N
Ts
Ph
Me
F
f1 (ppm)

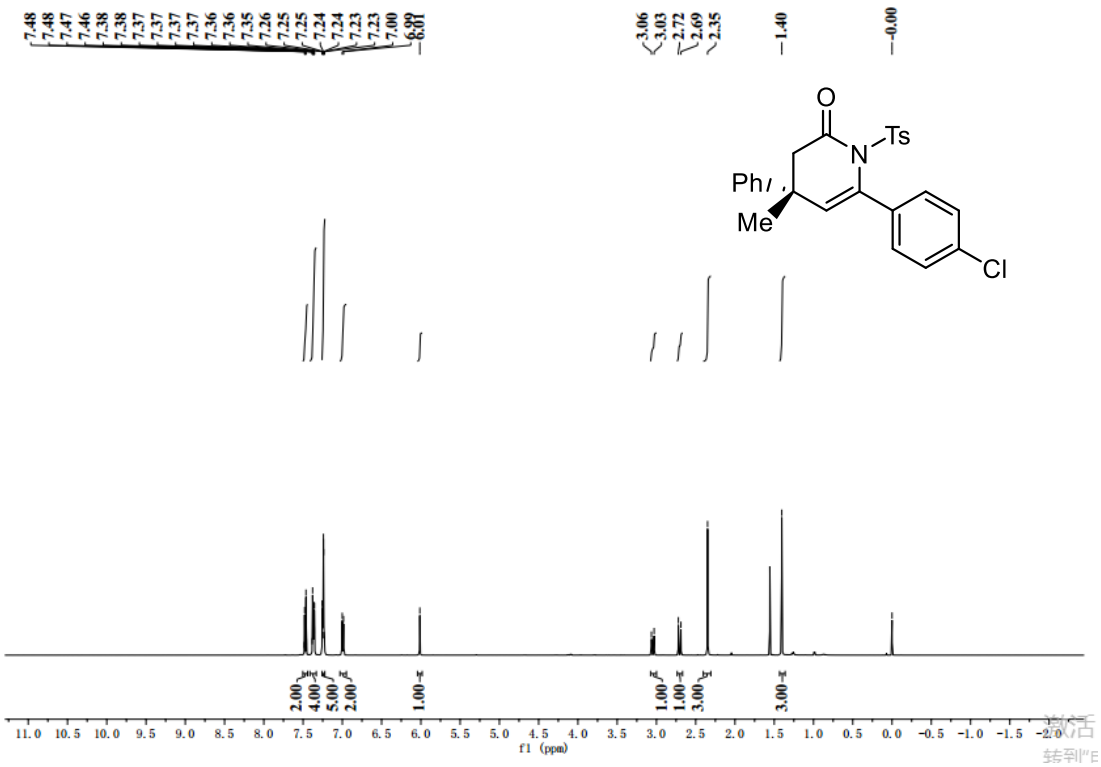


Peak	Ret .Time	Area	Height	Area %	Height %
1	15.469	974774	2005	99.626	99.693
2	18.806	3664	62	0.374	0.307
Total		978438	20116	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	15.568	1071455	21907	49.970	57.709
2	18.912	1072749	16054	50.030	42.291
Total		2144204	37961	100.000	100.000

3c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7.23
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3.06
3.03
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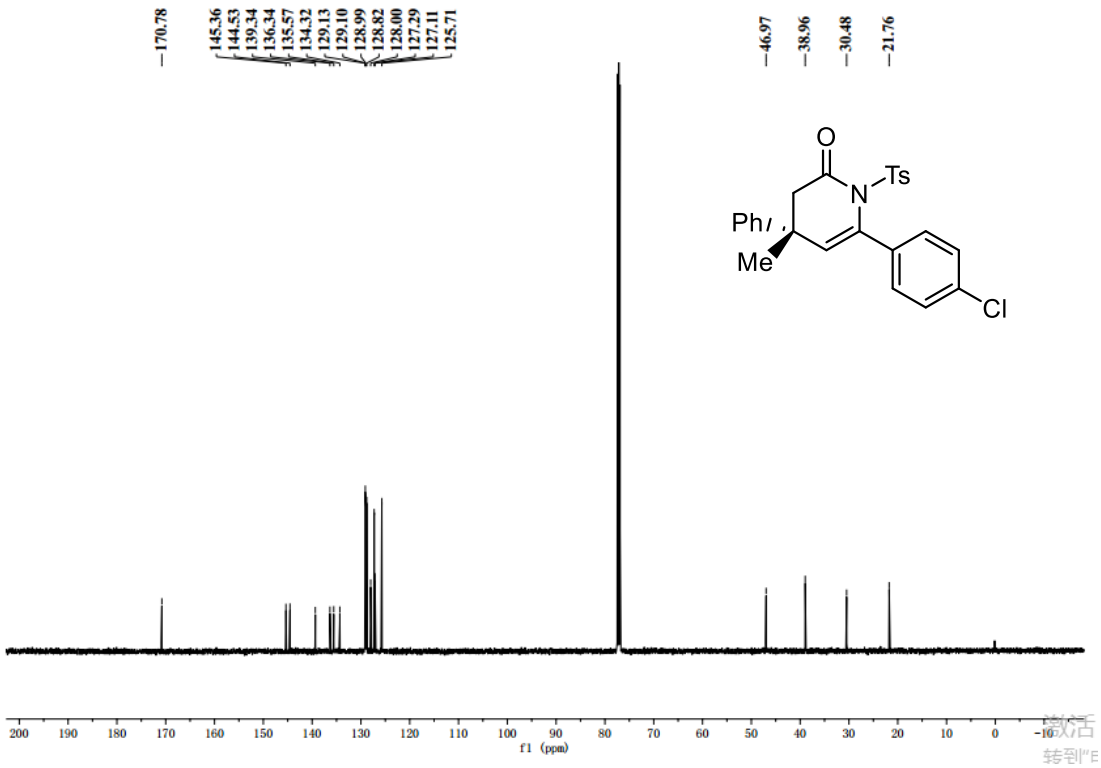
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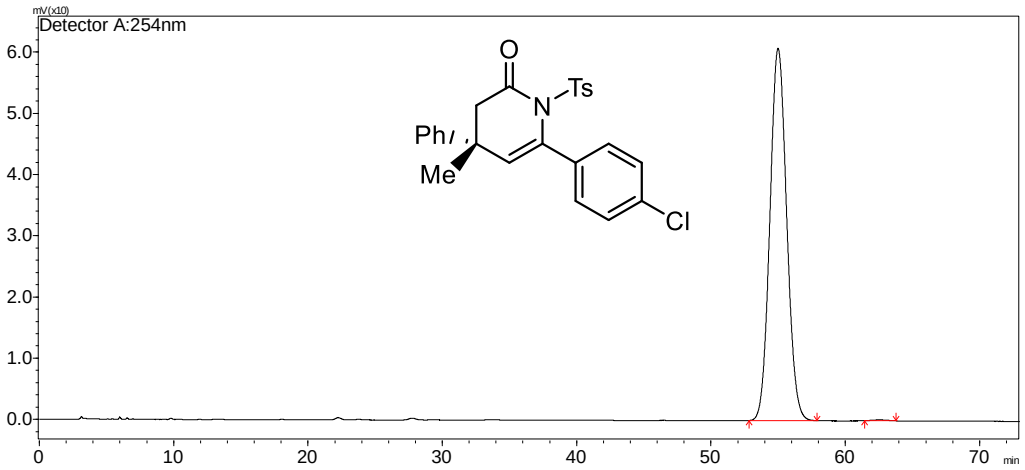
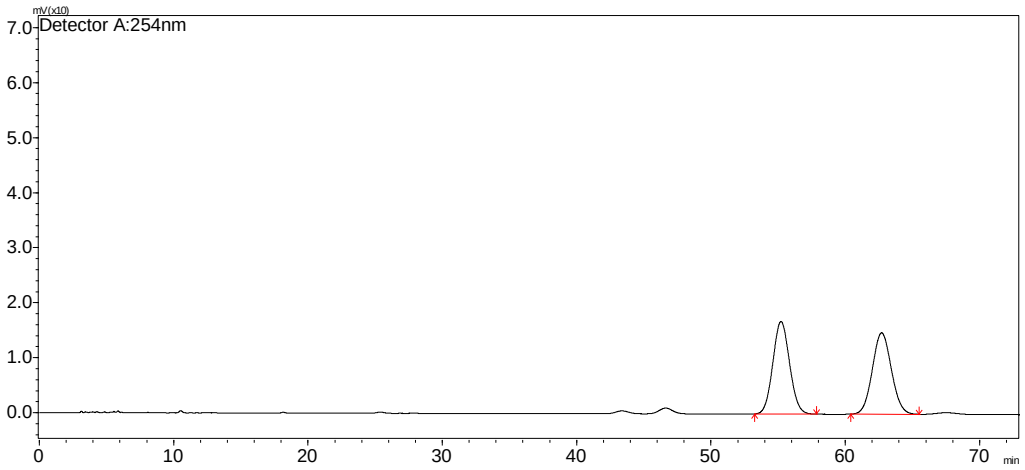
O
N
Ts
Ph
Me
Cl
f1 (ppm)

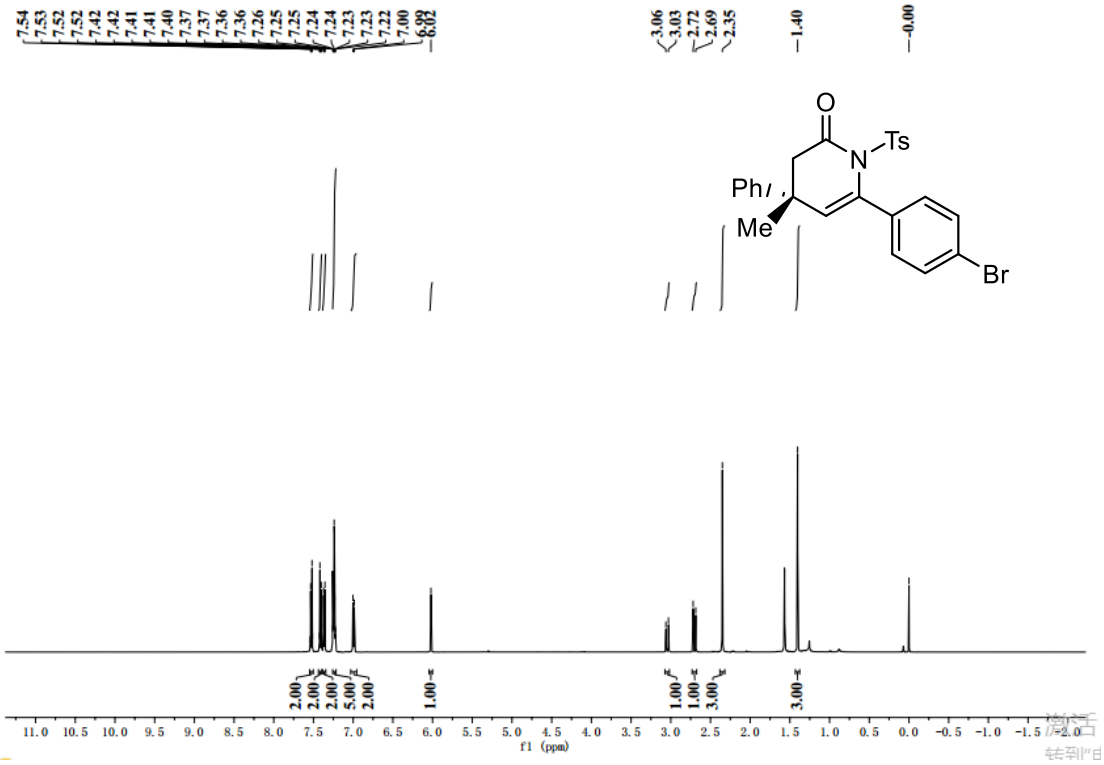


Peak	Ret .Time	Area	Height	Area %	Height %
1	54.992	5286001	60844	99.778	99.749
2	62.529	11768	153	0.222	0.251
Total		5297769	60997	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	55.212	1460040	16830	49.991	53.209
2	62.705	1460571	14800	50.009	46.791
Total		2920611	31629	100.000	100.000

3d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7.22
7.00
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6.02
3.06
3.03
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2.35
1.40
0.00
O
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Ts
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Me
Br
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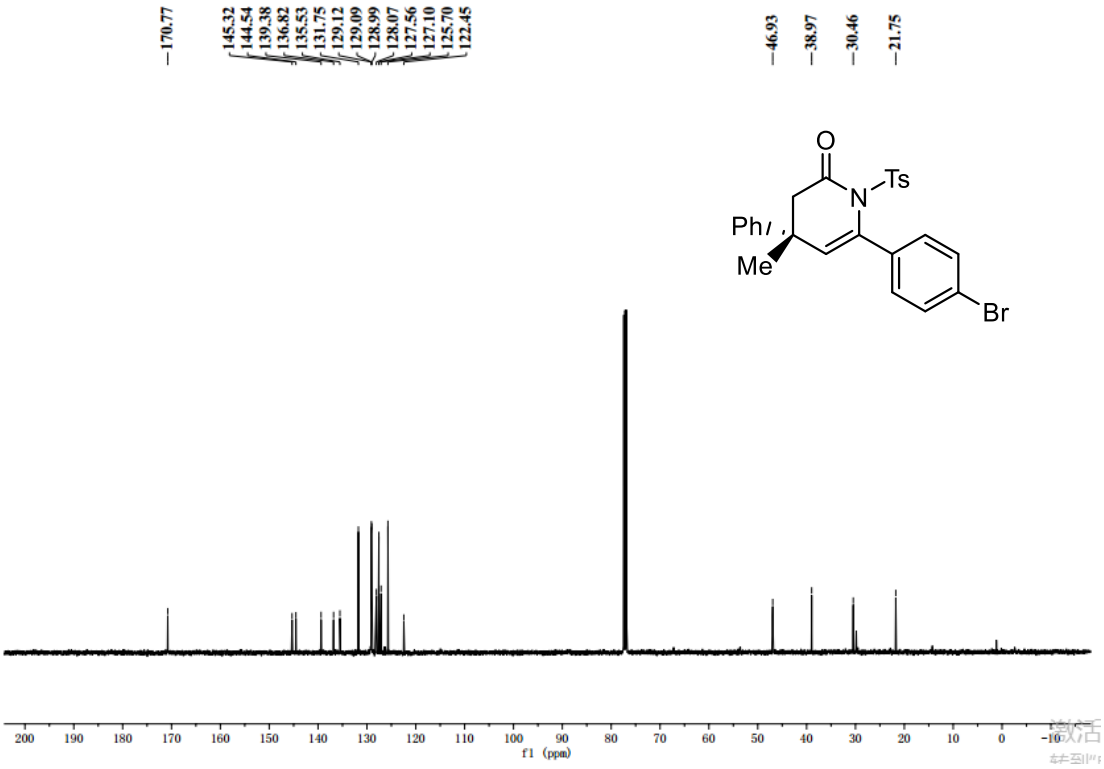
2.00
1.00
1.00
1.00
3.00
3.00
f1 (ppm)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122.45
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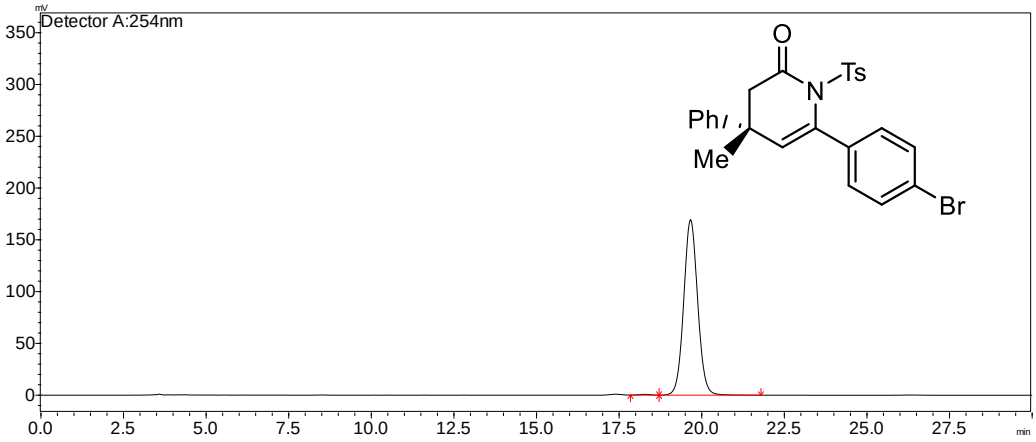
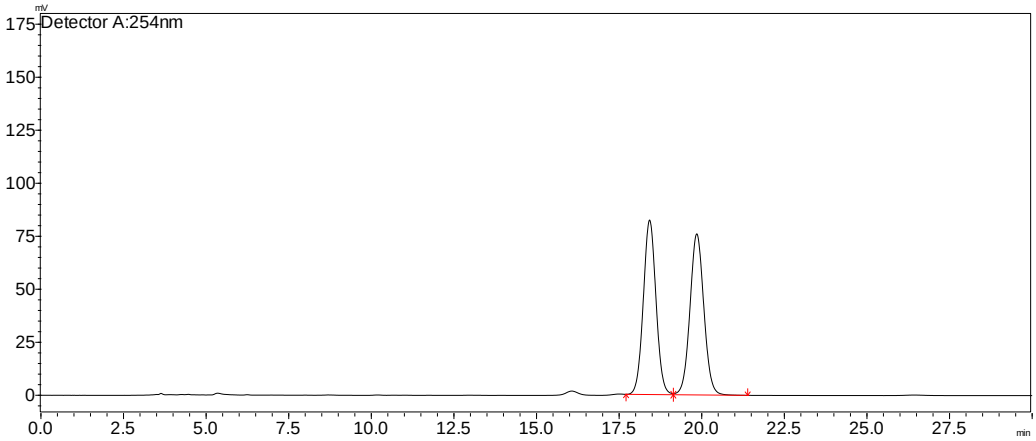
38.97
30.46
21.75
O
N
Ts
Ph
Me
Br
f1 (ppm)

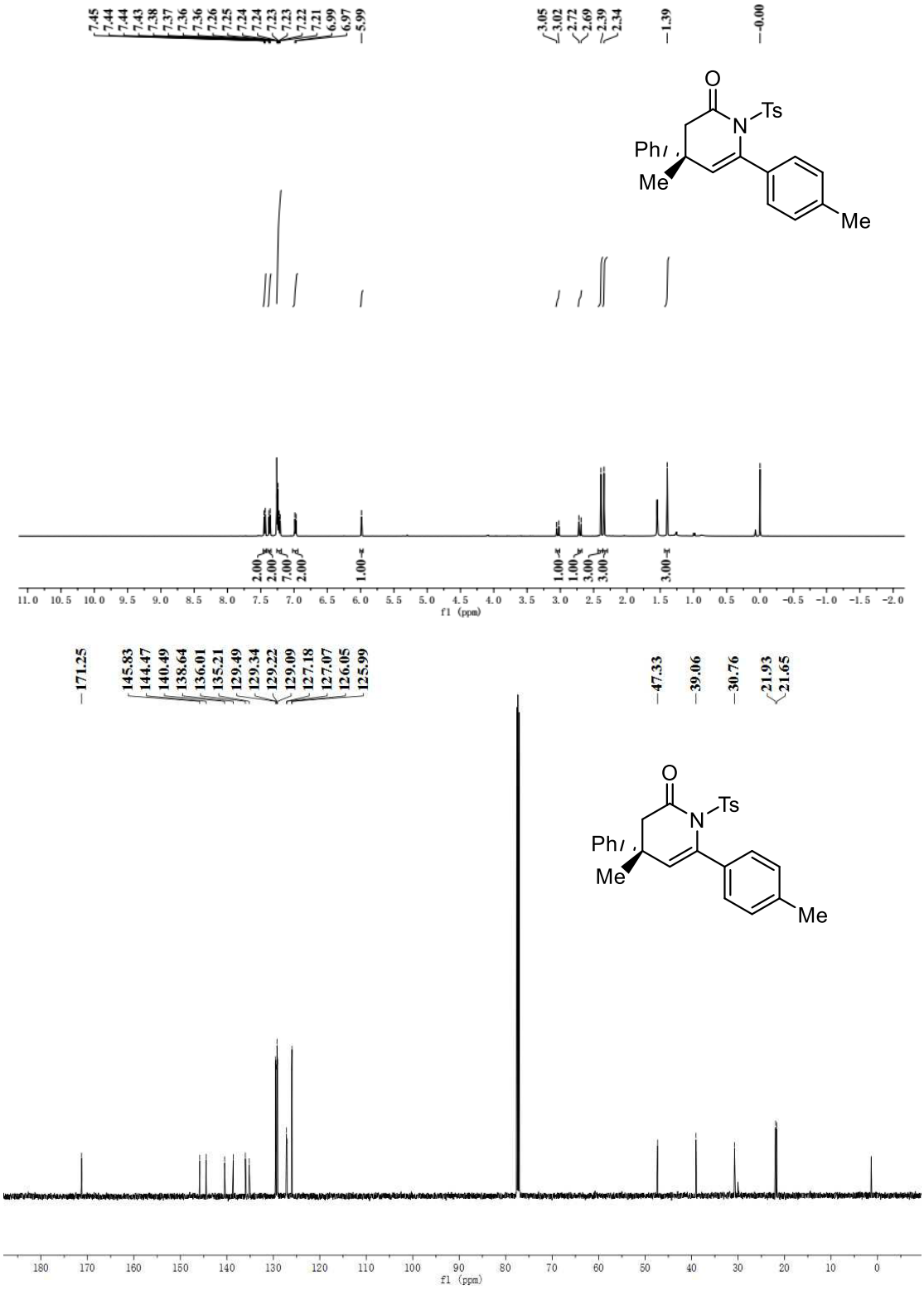


Peak	Ret .Time	Area	Height	Area%	Height%
1	18.276	16615	712	0.333	0.419
2	19.656	4977762	169415	99.667	99.581
Total		4994377	170127	100.000	100.000



Peak	Ret .Time	Area	Height	Area%	Height%
1	18.417	2200314	82299	49.713	51.998
2	19.845	2225742	75974	50.287	48.002
Total		4426056	158273	100.000	100.000

3e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7.23
7.23
7.22
7.21
6.99
6.97
5.99
3.05
3.02
2.72
2.69
2.39
2.34
1.39
0.00
O
N
Ts
Ph
Me
Me
2.00
2.00
7.00
2.00
1.00
1.00
1.00
3.00
3.00
3.00
f1 (ppm)
171.25
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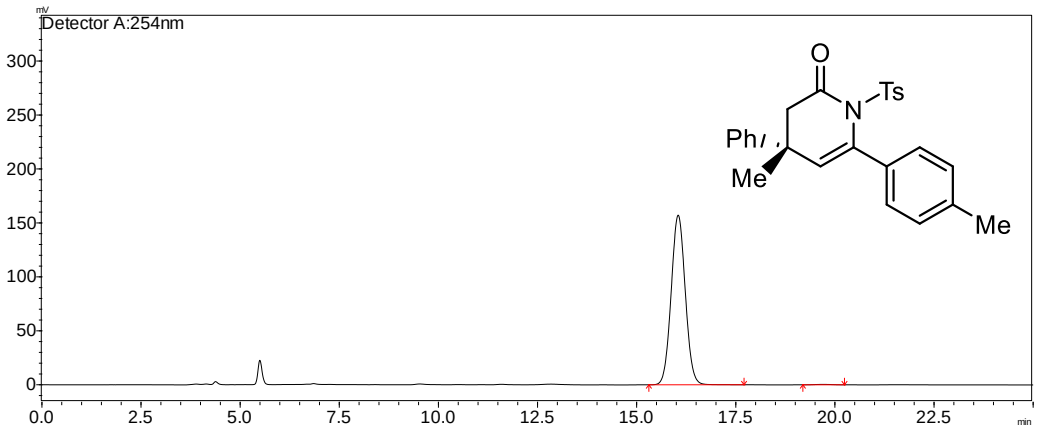
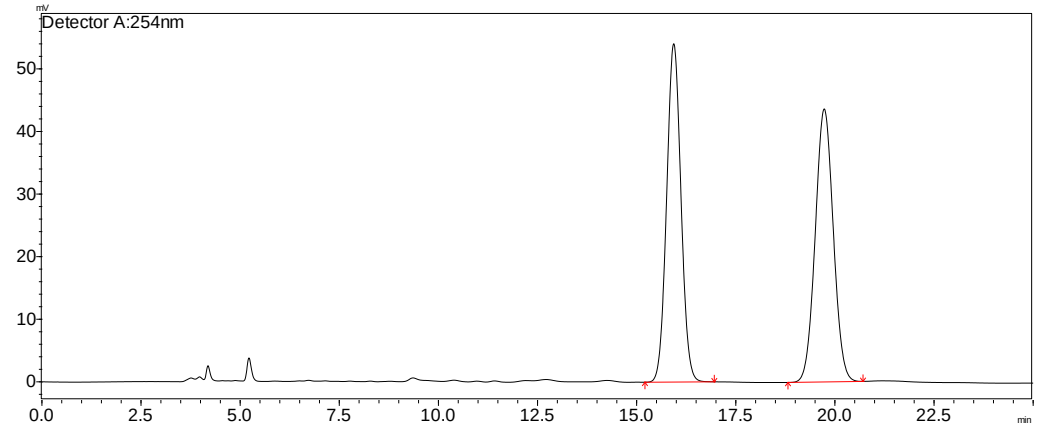
O
N
Ts
Ph
Me
Me
f1 (ppm)

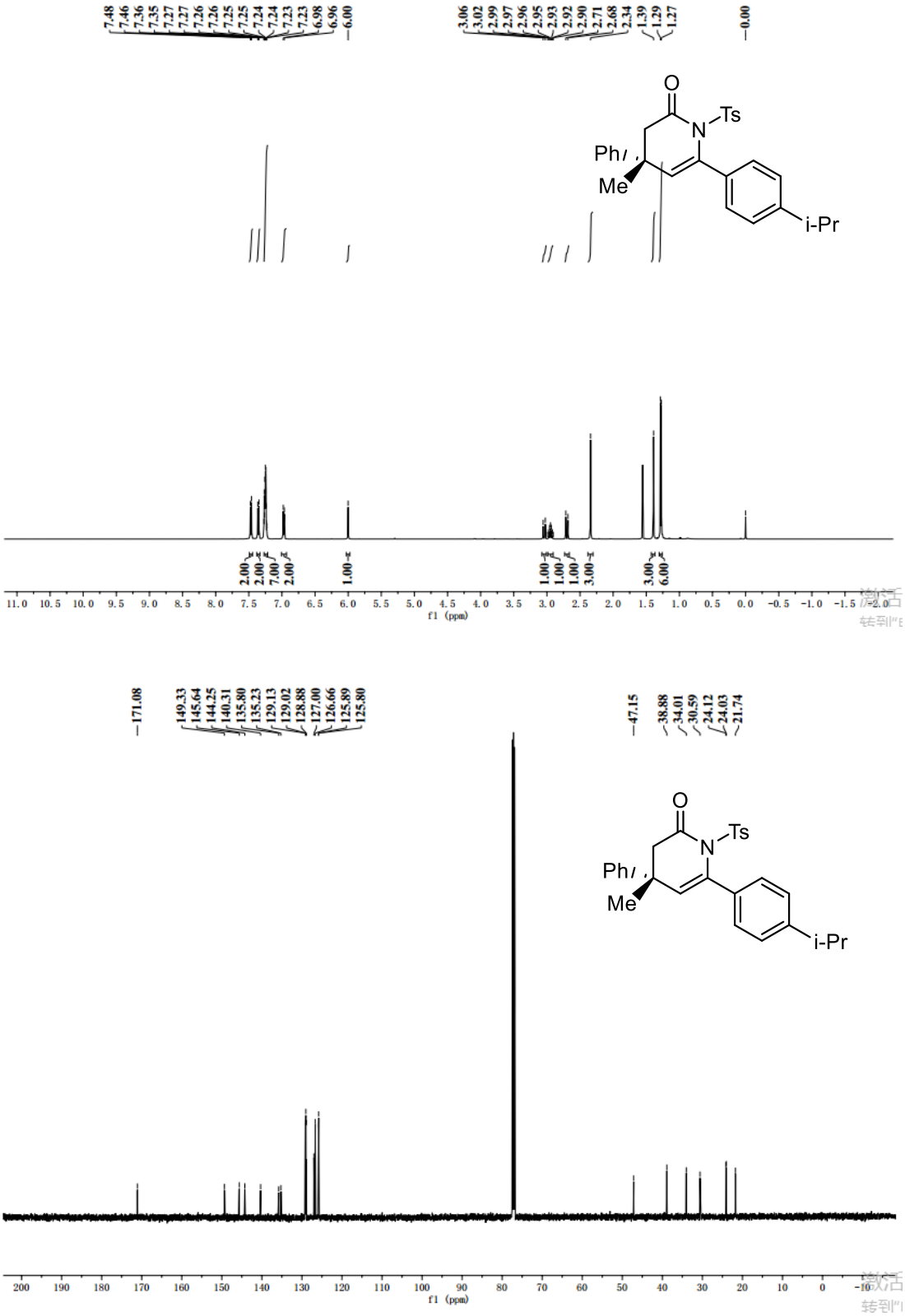


Peak	Ret .Time	Area	Height	Area%	Height%
1	16.042	3892839	157179	99.716	99.755
2	19.709	11070	386	0.284	0.245
Total		3903909	157565	100.000	100.000



Peak	Ret .Time	Area	Height	Area%	Height%
1	15.931	1359059	54055	49.579	55.338
2	19.727	1382140	43626	50.421	44.662
Total		274119	97681	100.000	100.000

3f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N
Ts
Ph
Me
i-Pr
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2.00
7.00
2.00
1.00
1.00
1.00
1.00
3.00
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6.00
f1 (ppm)
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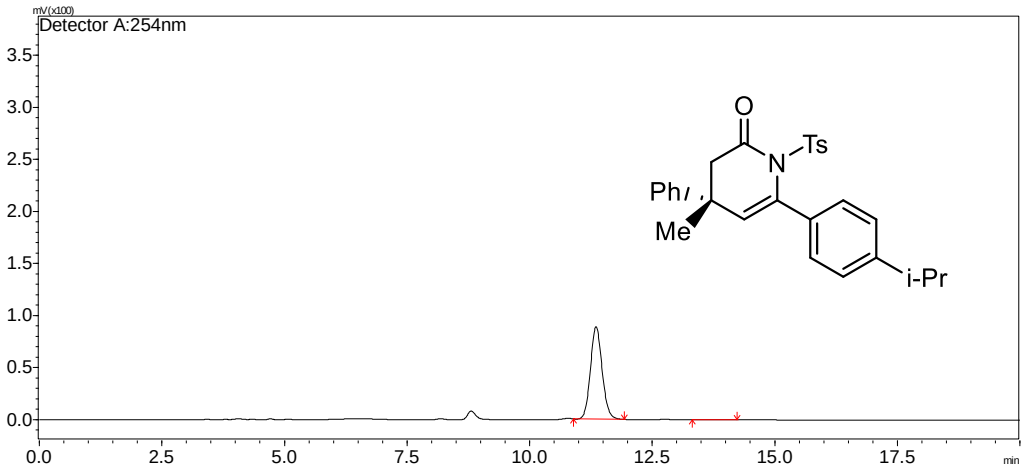
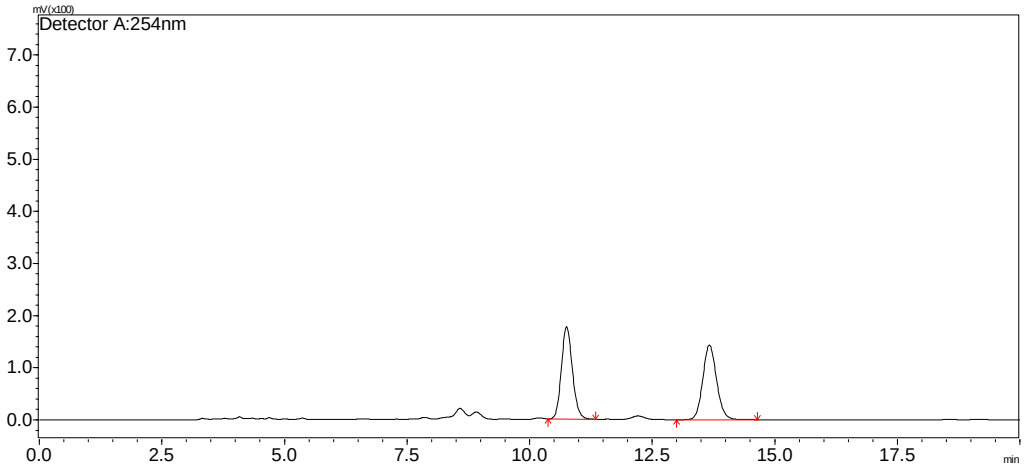
Ts
Ph
Me
i-Pr
f1 (ppm)

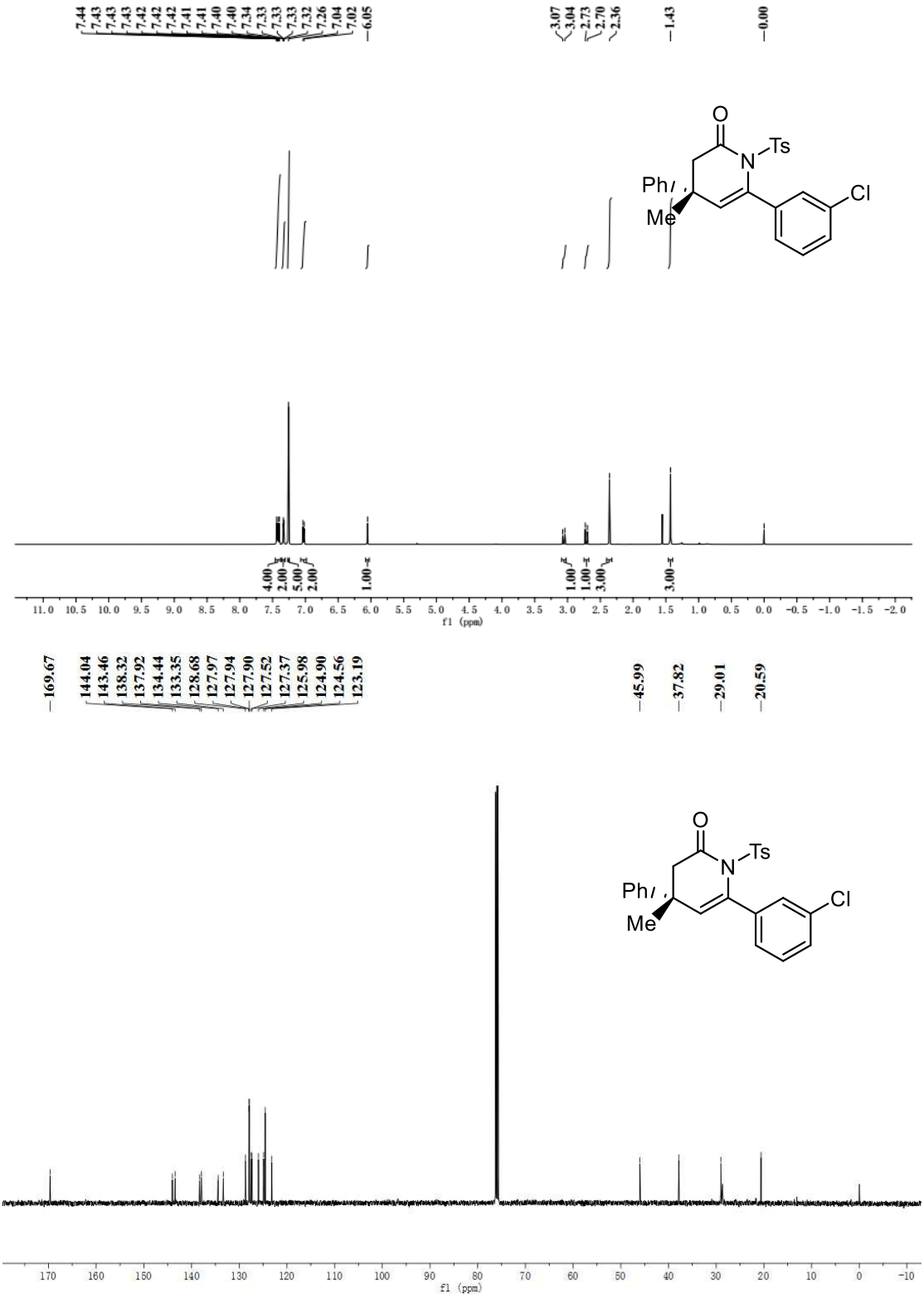


Peak	Ret .Time	Area	Height	Area %	Height %
1	11.350	1477646	88638	99.764	99.832
2	13.881	3498	149	0.236	0.168
Total		1481144	88787	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	10.749	2776618	177259	49.744	55.315
2	13.662	2805188	143195	50.256	44.685
Total		5581805	320454	100.000	100.000

3g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6.05
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3.04
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2.36
1.43
0.00
O
N
Ts
Ph
Me
Cl
4.00
2.00
5.00
2.00
1.00
1.00
1.00
3.00
3.00
f1 (ppm)
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37.82
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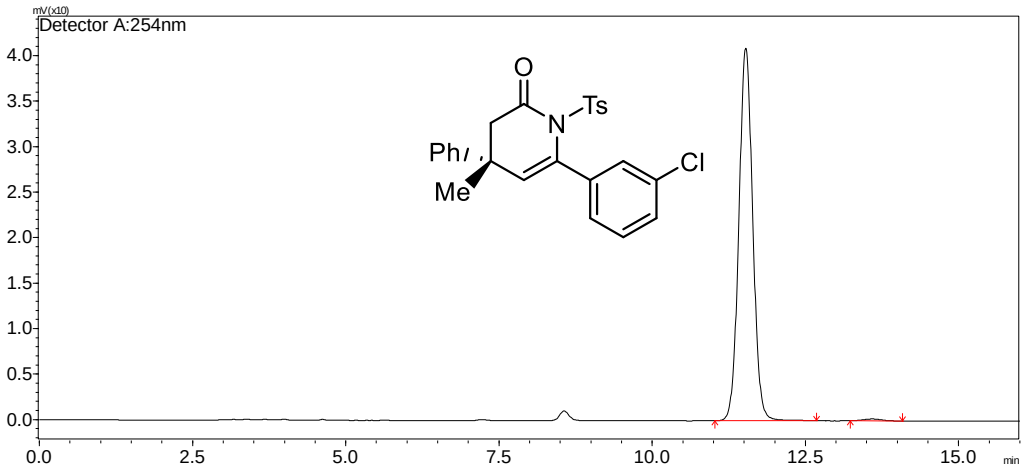
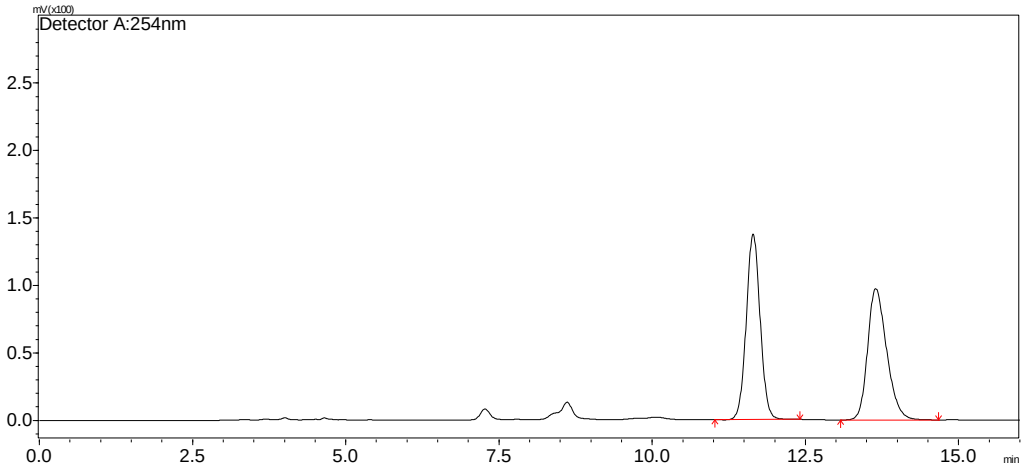
20.59
f1 (ppm)

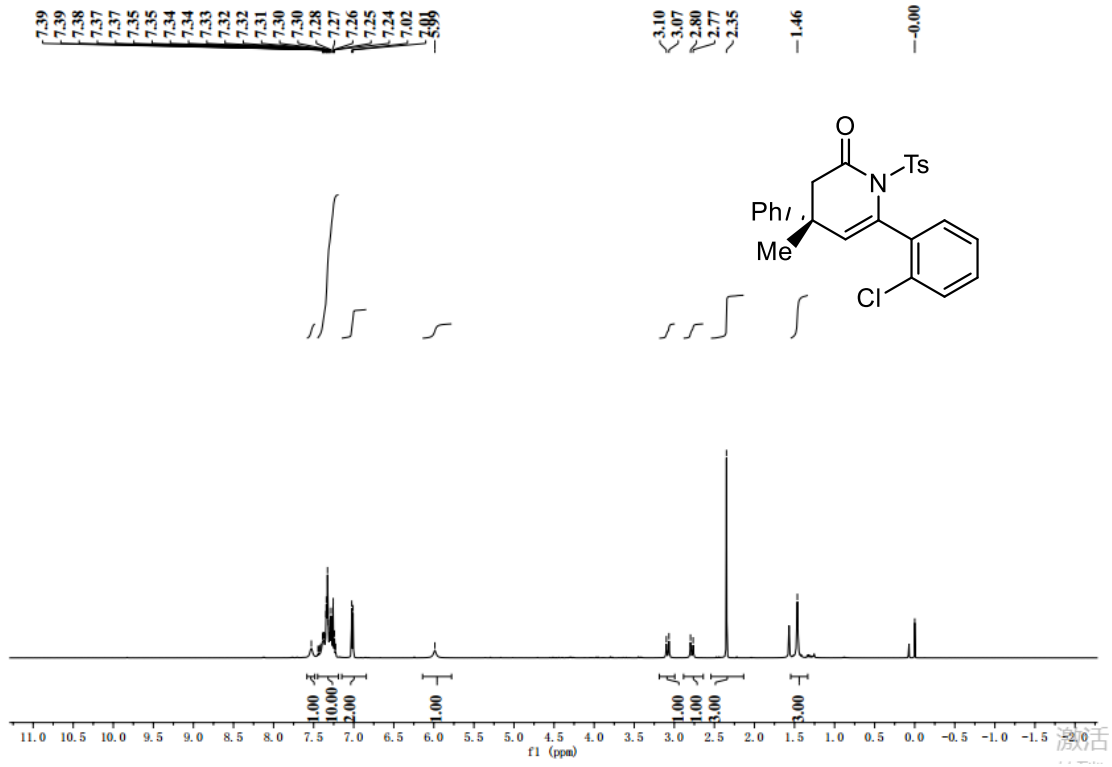


Peak	Ret .Time	Area	Height	Area %	Height %
1	11.522	645260	40950	99.362	99.500
2	13.586	4143	206	0.638	0.500
Total		649403	41156	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	11.642	2167248	137472	50.663	58.537
2	13.643	2110531	97375	49.337	41.463
Total		4277779	234847	100.000	100.000

3h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7.24
7.02
7.01
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3.07
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2.35
1.46
0.00
O
Ts
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Ph
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Cl
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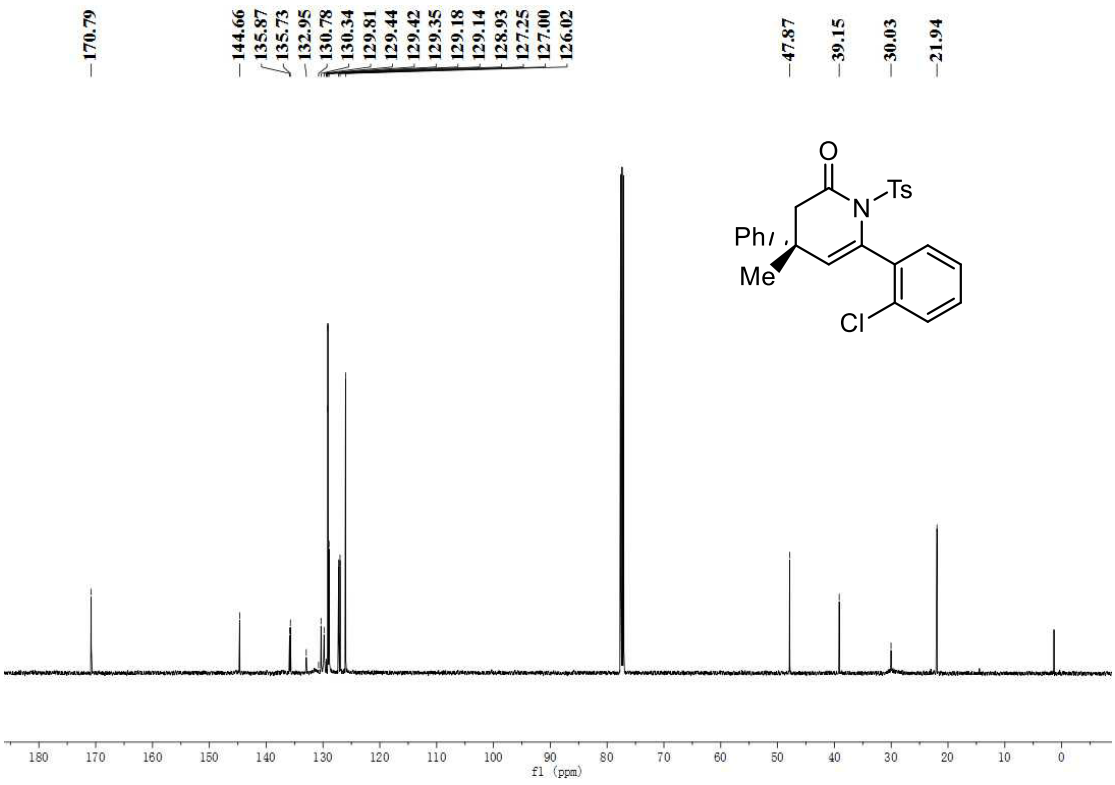
1.00
1.00
1.00
3.00
3.00
f1 (ppm)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129.14
128.93
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126.02
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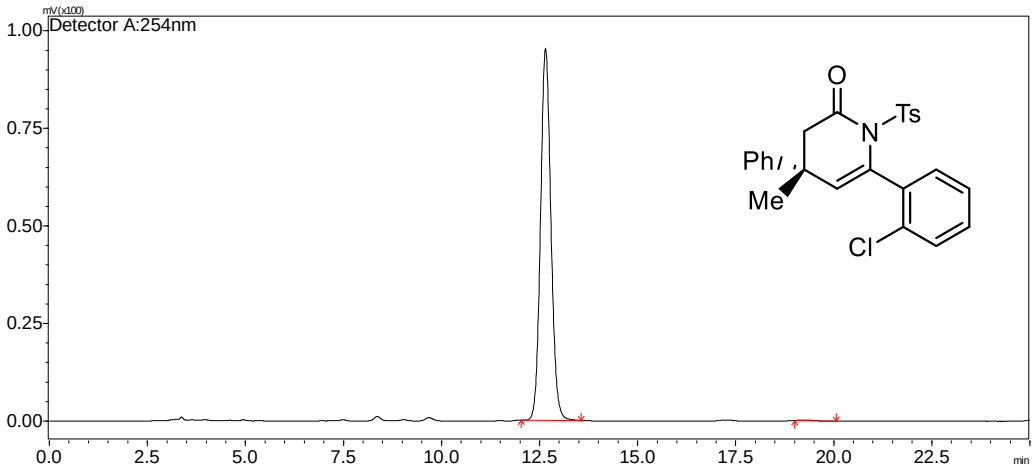
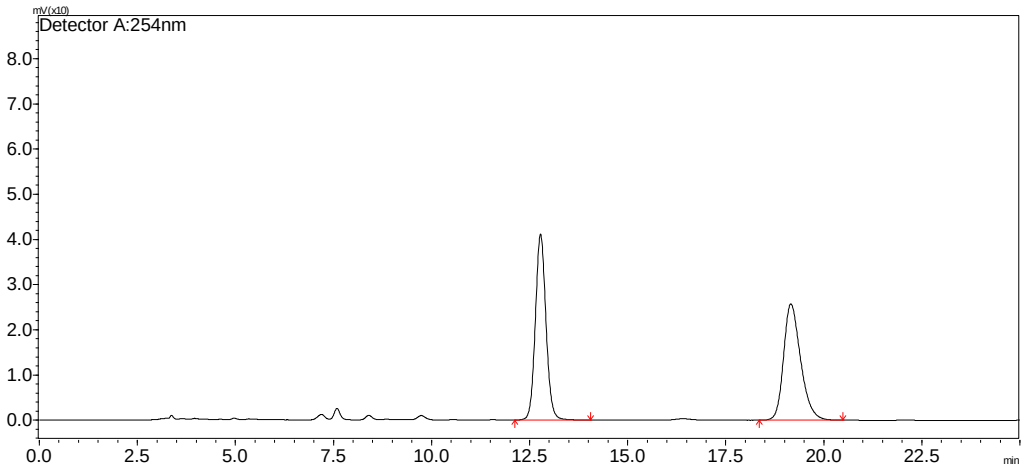
21.94
O
Ts
N
Ph
Me
Cl
f1 (ppm)

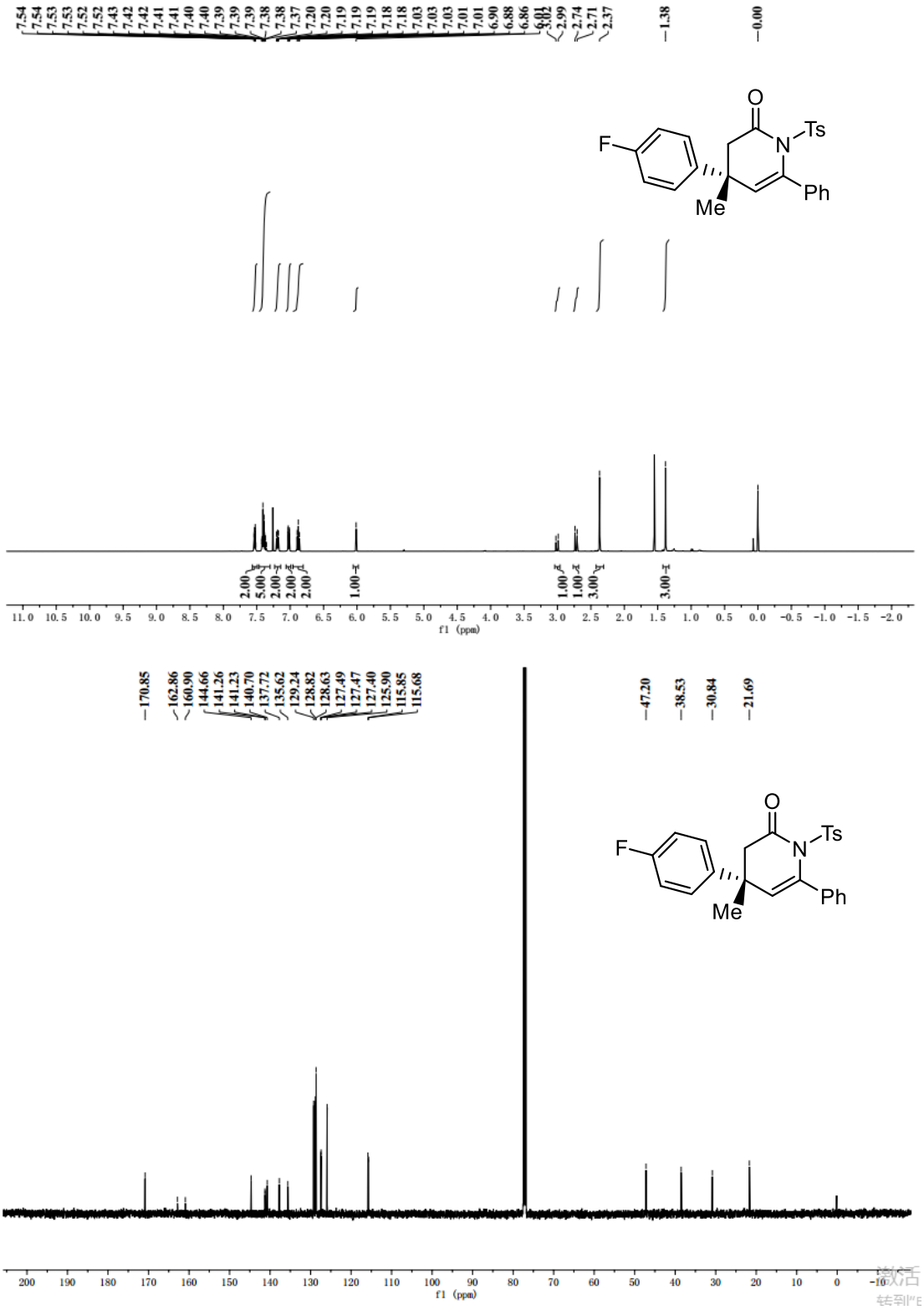


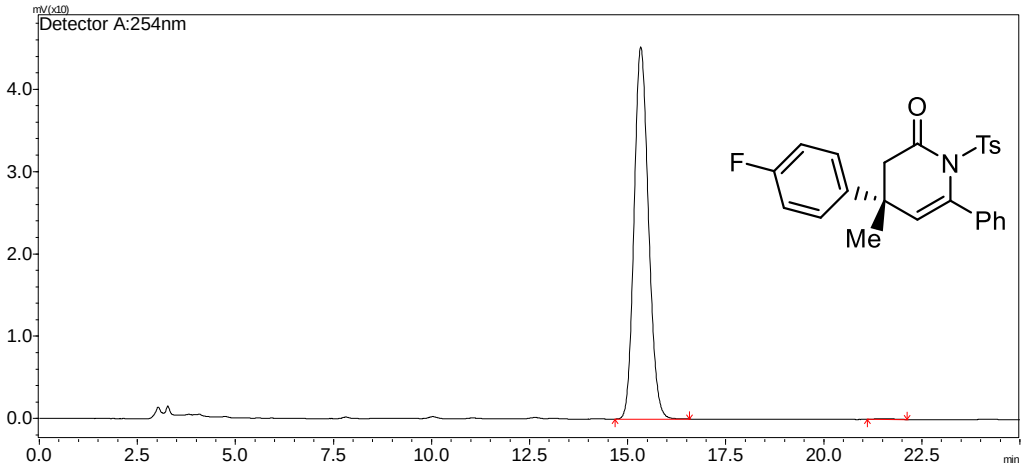
Peak	Ret .Time	Area	Height	Area %	Height %
1	12.647	1759915	95346	99.866	99.878
2	19.282	2360	116	0.134	0.122
Total		1762274	95462	100.000	100.000



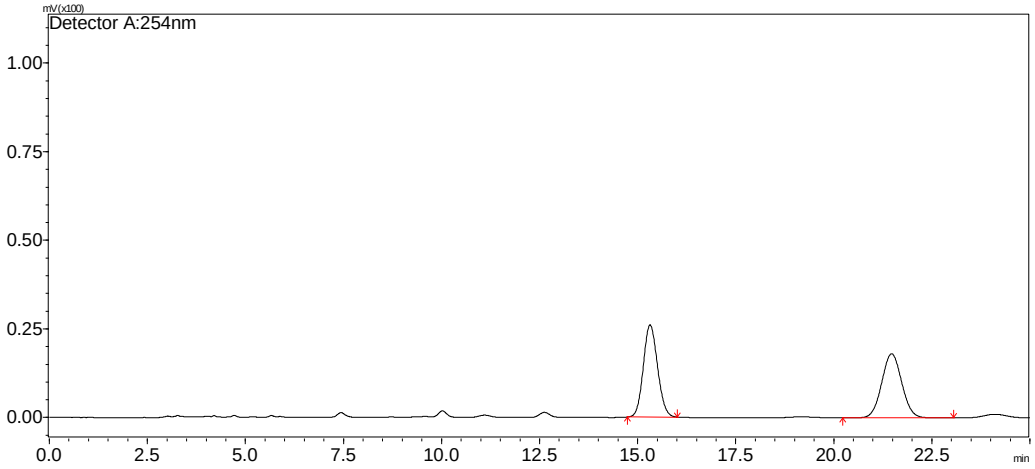
Peak	Ret .Time	Area	Height	Area %	Height %
1	12.773	776275	41206	49.904	61.523
2	19.155	779272	25771	50.096	38.477
Total		1555547	66977	100.000	100.000

3i


7.54 7.54 7.53 7.53 7.52 7.52 7.43 7.42 7.42 7.41 7.41 7.40 7.40 7.39 7.39 7.39 7.38 7.38 7.37 7.20 7.20 7.19 7.19 7.19 7.18 7.18 7.03 7.03 7.03 7.01 7.01 6.90 6.88 6.86 6.01 3.02 2.99 2.74 2.71 2.37 1.38 0.00
F
O
N
Ts
Ph
Me
2.00 5.00 2.00 2.00 2.00 1.00 1.00 1.00 3.00 3.00
f1 (ppm)
170.85 162.86 160.90 144.66 141.26 141.23 140.70 137.72 135.62 129.24 128.82 128.63 127.49 127.47 127.40 125.90 115.85 115.68 47.20 38.53 30.84 21.69
f1 (ppm)

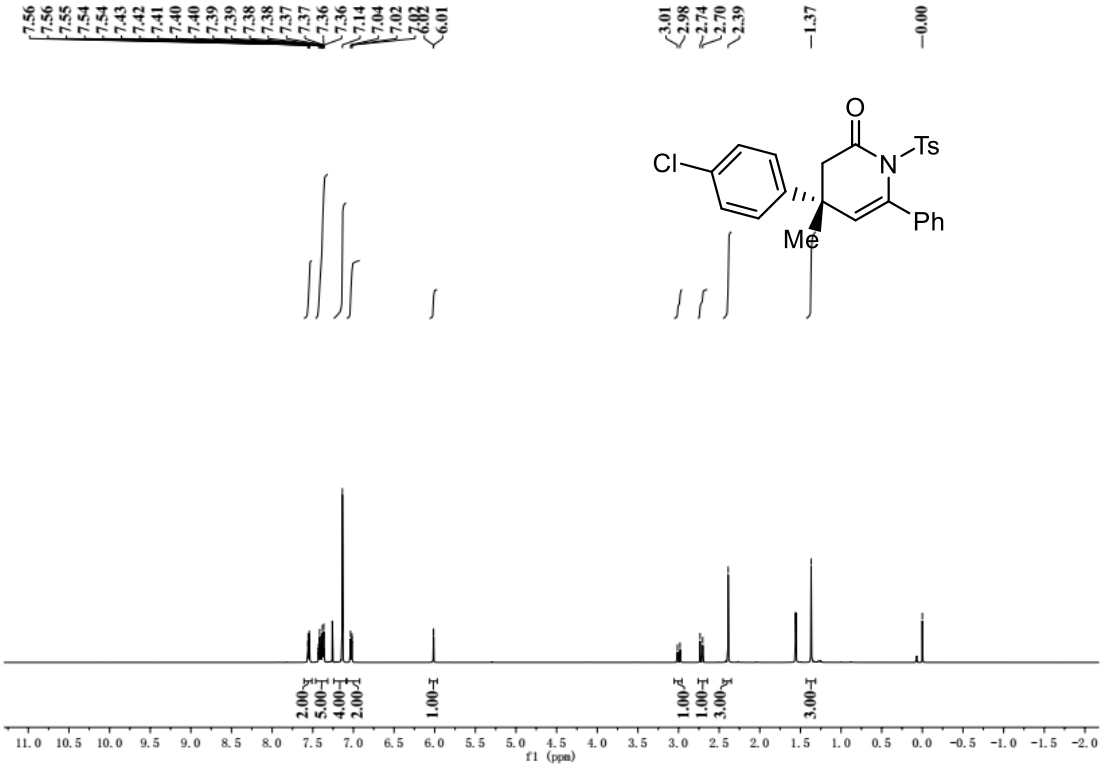


Peak	Ret .Time	Area	Height	Area %	Height %
1	15.330	1154381	45221	99.721	99.768
2	21.545	3229	102	0.279	0.232
Total		1157610	45326	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	15.311	657690	26045	50.662	49.028
2	21.470	640511	18078	49.33840.972	42.291
Total		1298201	44124	100.000	100.000

3j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7.02
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6.01
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2.70
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N
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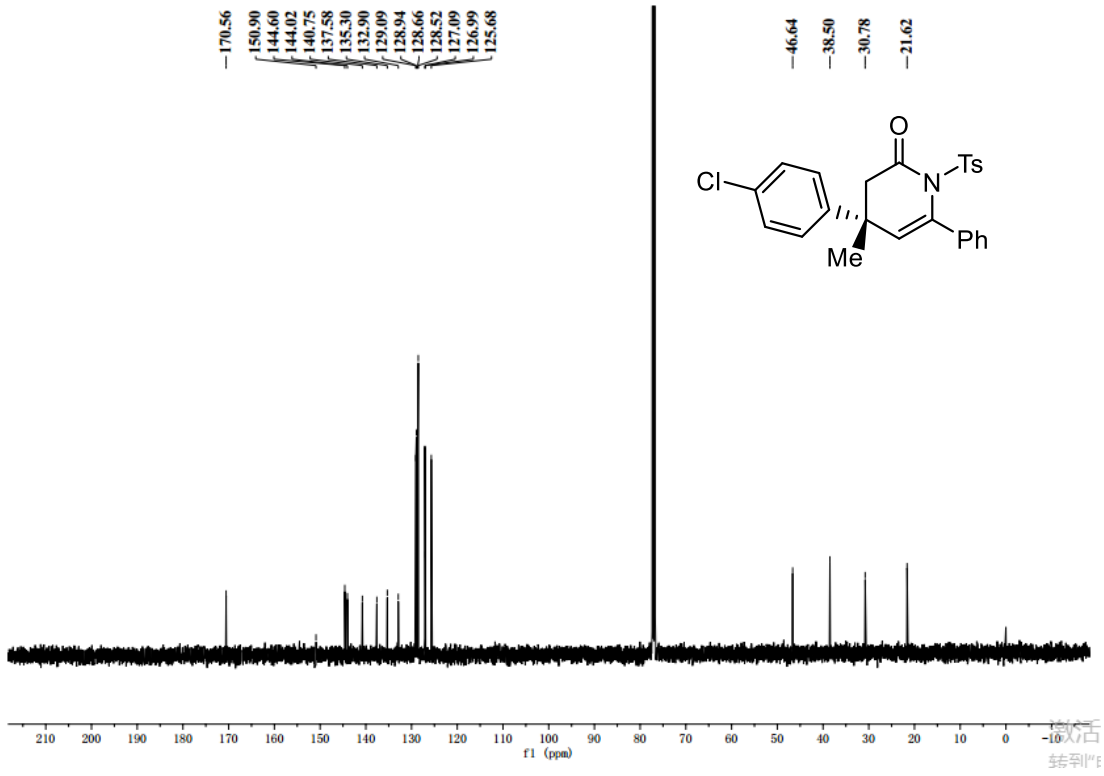
1.00
3.00
3.00
f1 (ppm)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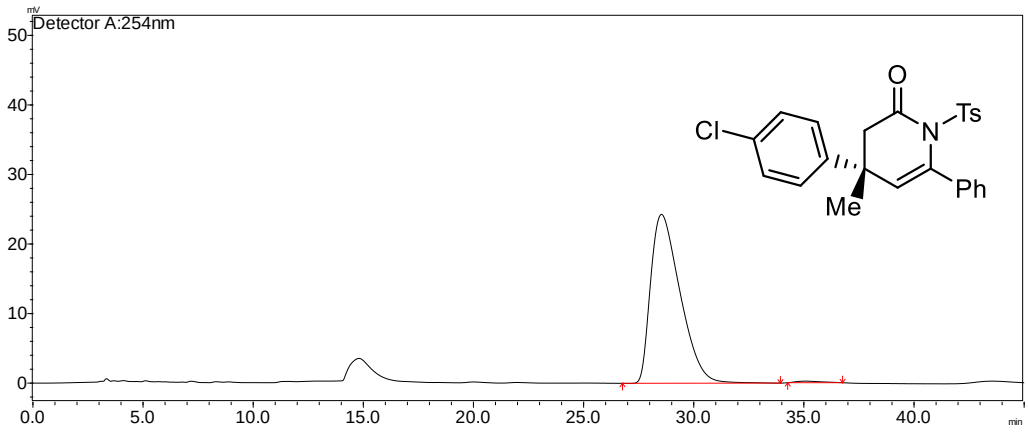
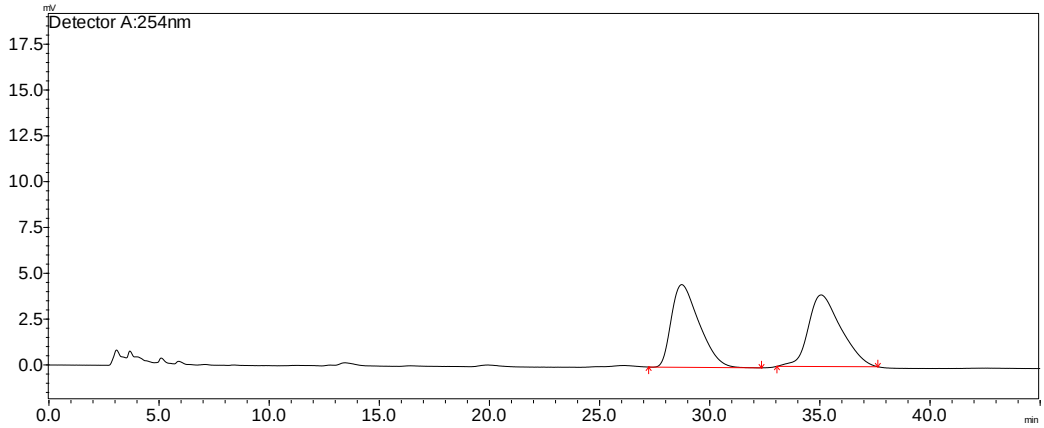
Cl
Ph
Me
f1 (ppm)

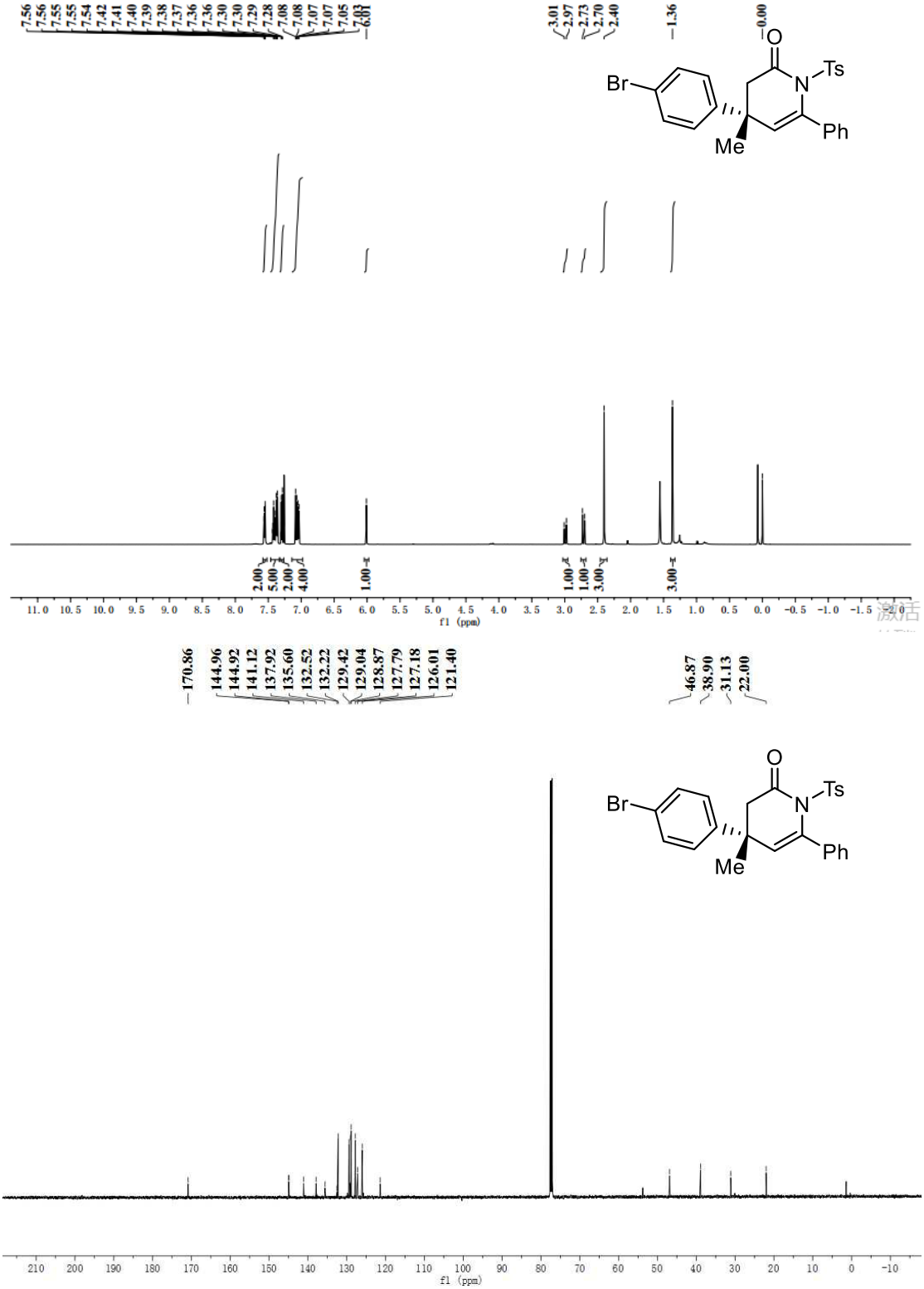


Peak	Ret .Time	Area	Height	Area%	Height%
1	28.532	2261604	24289	99.182	99.078
2	35.038	18642	226	0.818	0.922
Total		652203	16771	100.000	100.000



Peak	Ret .Time	Area	Height	Area%	Height%
1	28.721	395656	4512	49.506	53.587
2	35.064	403557	3908	50.494	46.413
Total		799212	8419	100.000	100.000

3k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7.08
7.07
7.07
7.05
7.03
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N
Ts
Ph
Me
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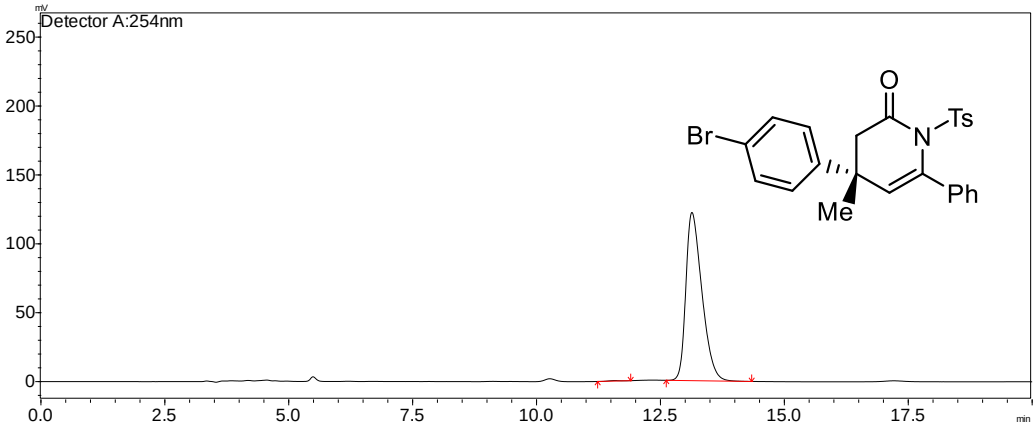
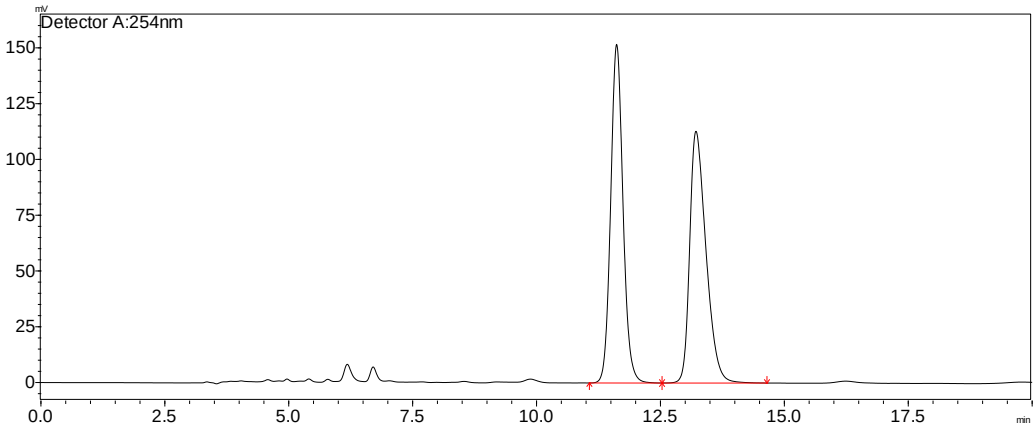
f1 (ppm)

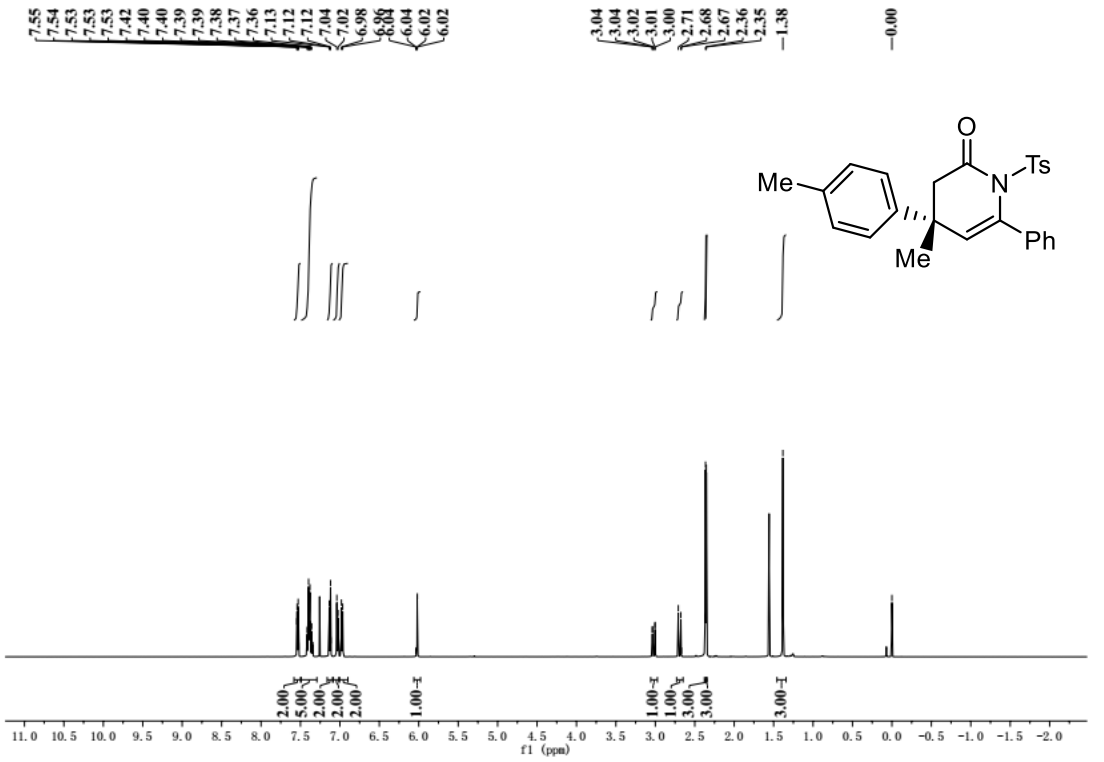


Peak	Ret .Time	Area	Height	Area%	Height%
1	11.573	5017	352	0.182	0.287
2	13.132	2752777	122083	99.818	99.713
Total		2757794	122435	100.000	100.000



Peak	Ret .Time	Area	Height	Area%	Height%
1	11.612	2626460	151794	50.463	57.355
2	13.213	2578313	112861	49.537	42.645
Total		5204773	264655	100.000	100.000

3l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3.02
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2.35
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Ts
N
Me
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1.00
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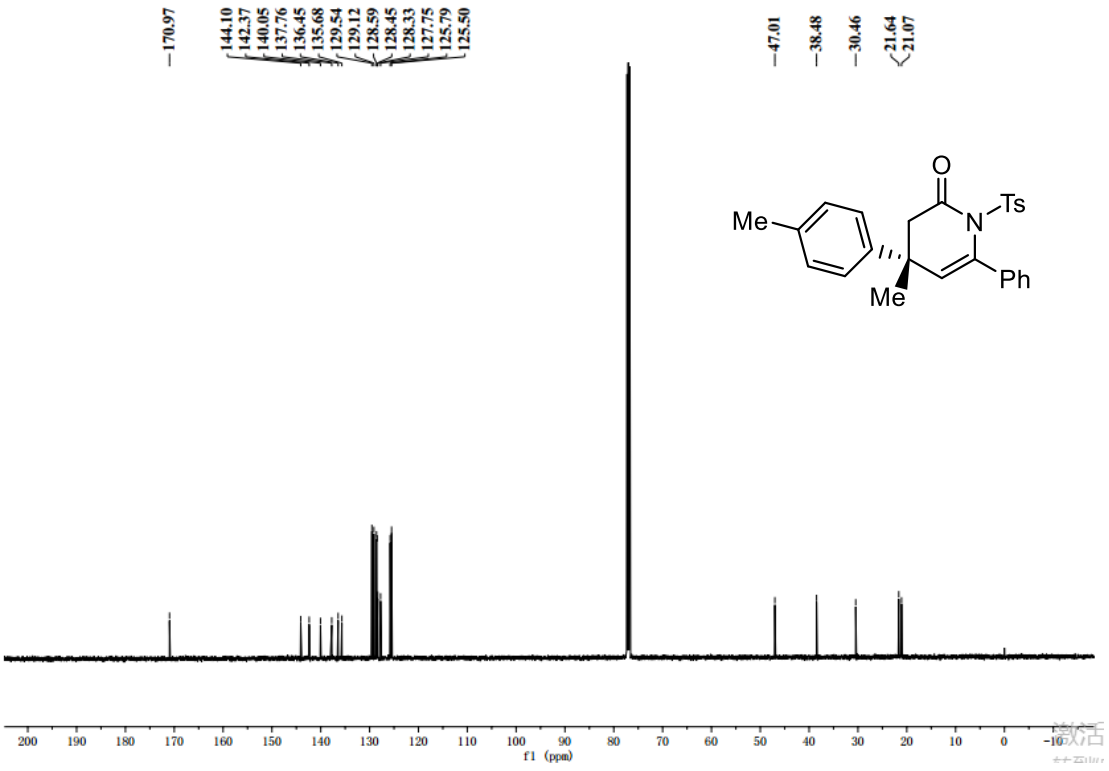
3.00
f1 (ppm)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125.50
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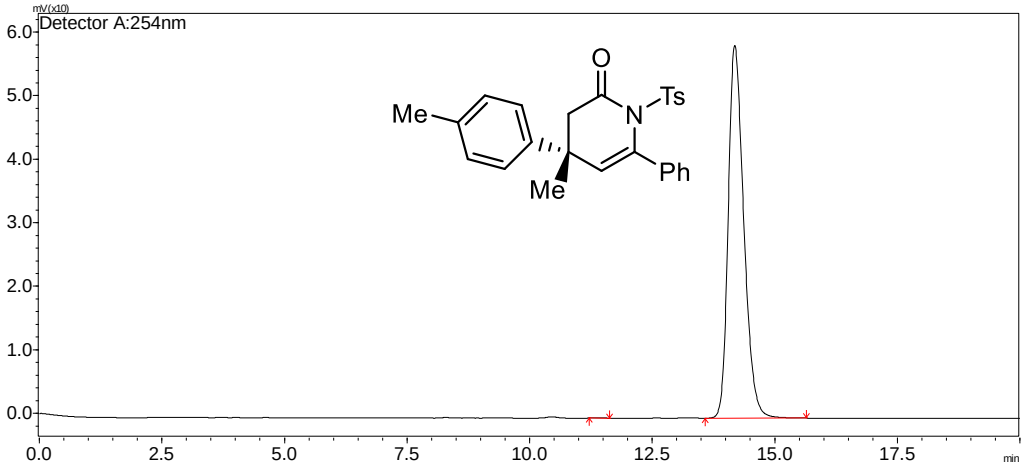
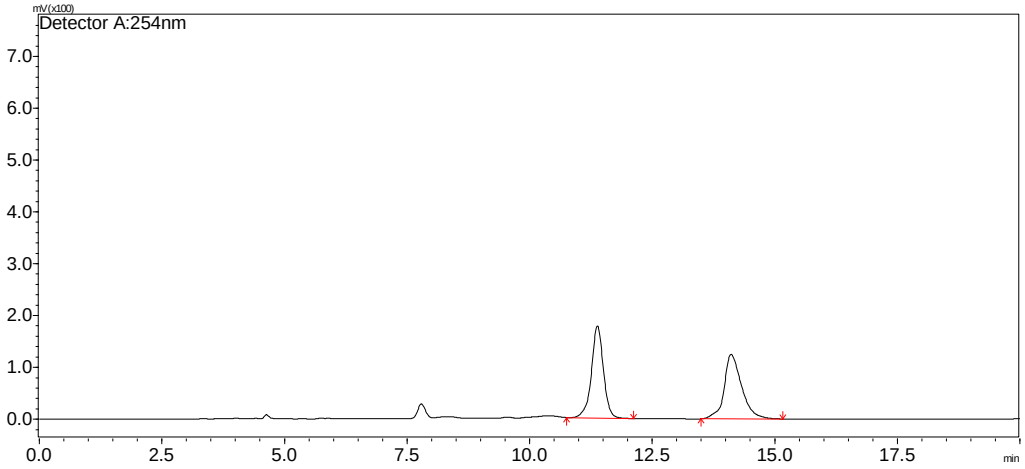
38.48
30.46
21.64
21.07
O
Ts
N
Me
Me
Ph
f1 (ppm)

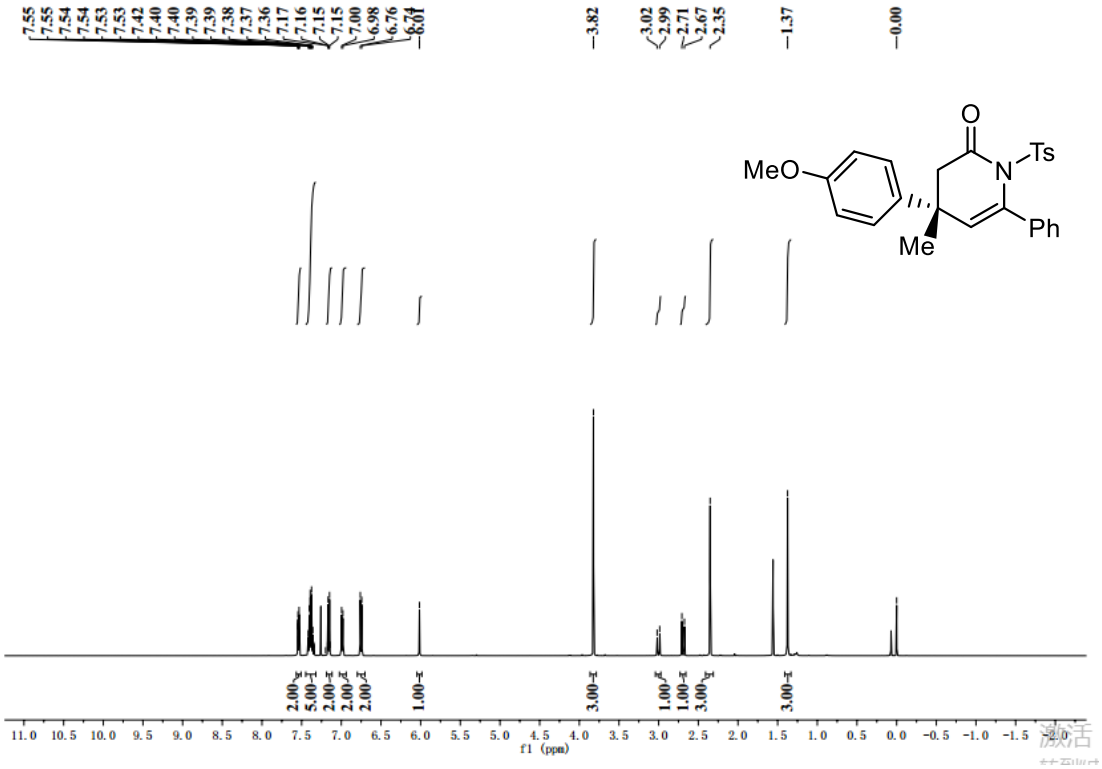


Peak	Ret .Time	Area	Height	Area %	Height %
1	11.373	654	51	0.050	0.086
2	14.180	1296296	58668	99.950	99.914
Total		1296950	58719	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	11.379	3035399	177662	49.588	58.834
2	14.106	3085826	124310	50.412	41.166
Total		6121224	301972	100.000	100.000

3m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7.54
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7.53
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7.40
7.40
7.39
7.39
7.38
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7.17
7.16
7.15
7.15
7.00
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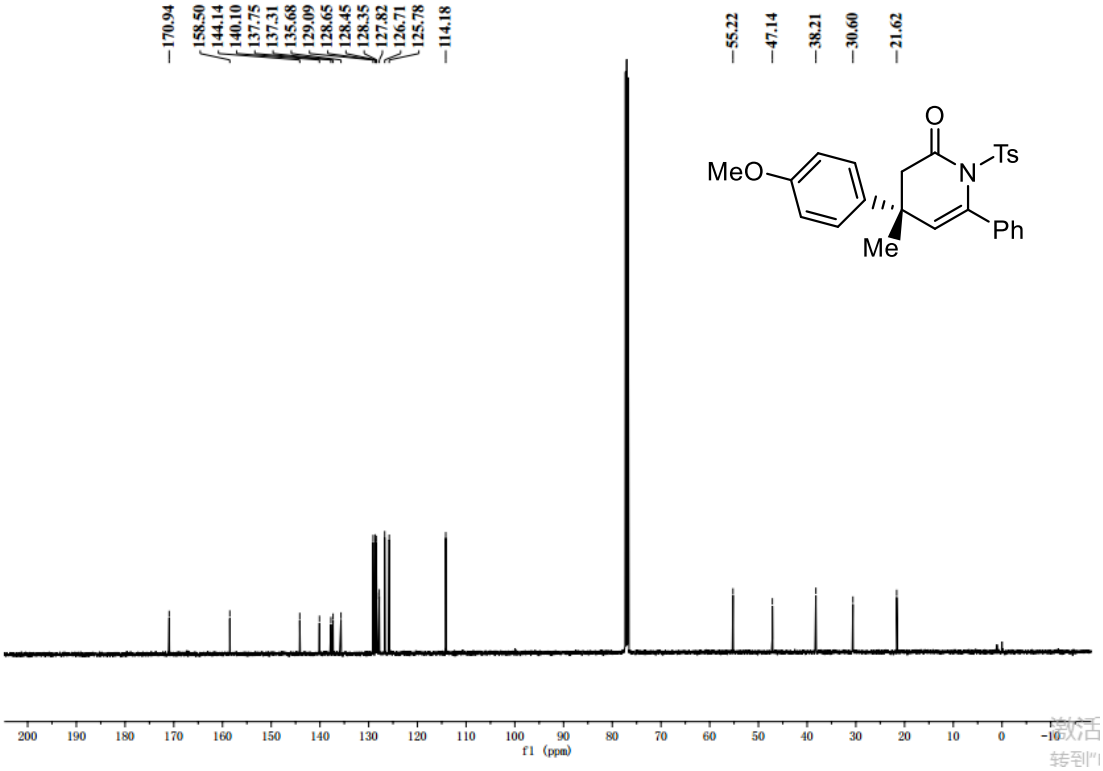
6.76
6.74
6.01
3.82
3.02
2.99
2.71
2.67
2.35
1.37
0.00
MeO
O
N
Ts
Ph
Me
2.00
5.00
2.00
2.00
2.00
1.00
3.00
1.00
1.00
3.00
3.00
f1 (ppm)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128.45
128.35
127.82
126.71
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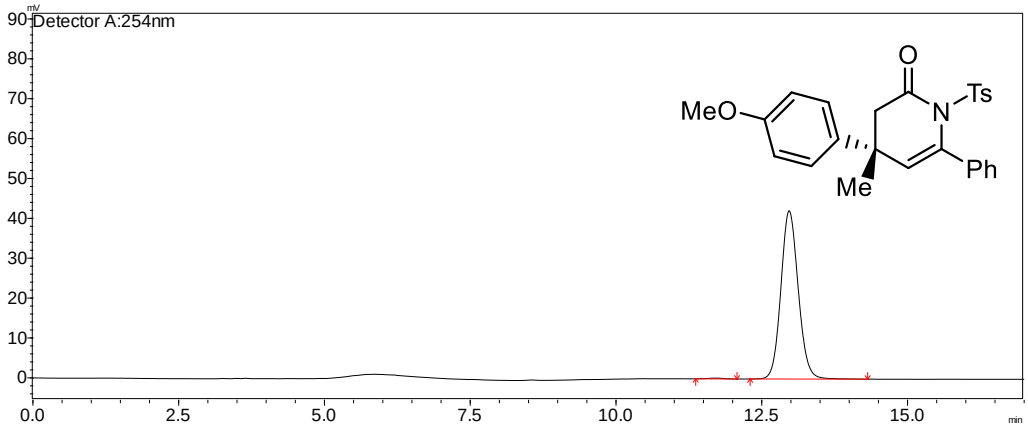
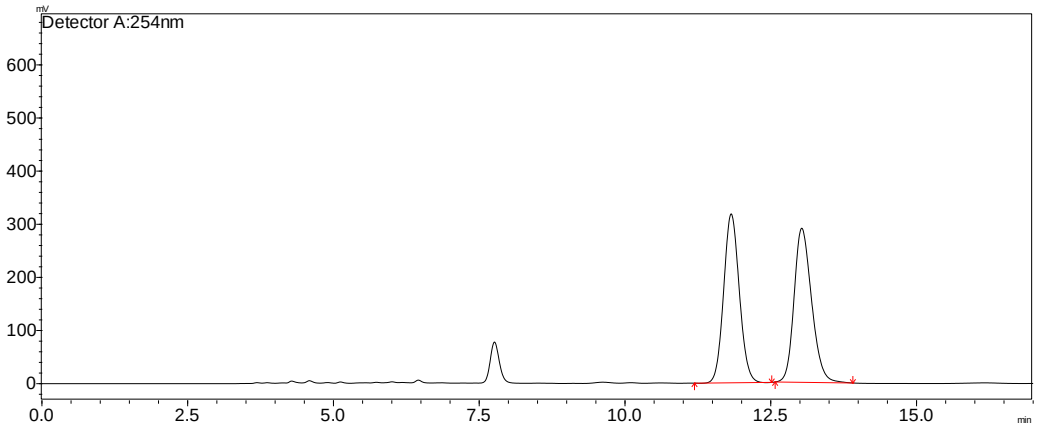
30.60
21.62
MeO
O
N
Ts
Ph
Me
f1 (ppm)

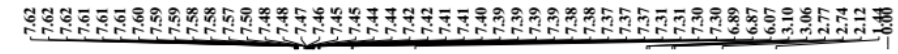


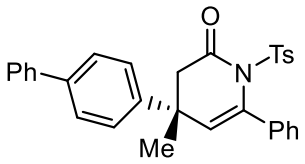
Peak	Ret .Time	Area	Height	Area%	Height%
1	11.699	3717	199	0.424	0.469
2	12.965	873102	42215	99.576	99.531
Total		876819	42414	100.000	100.000

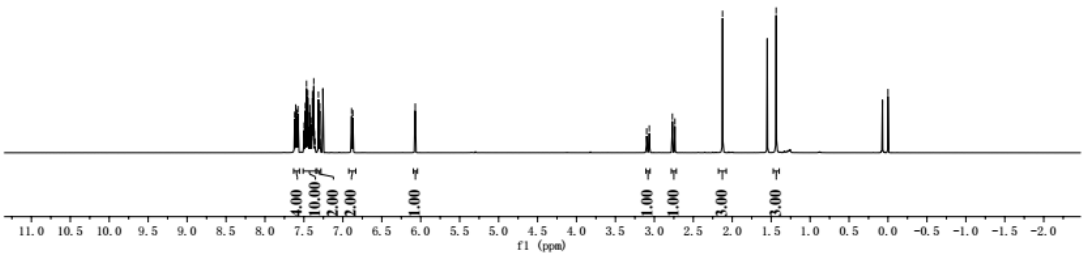


Peak	Ret .Time	Area	Height	Area%	Height%
1	11.817	5956964	318235	49.311	52.252
2	13.027	6123335	290798	50.689	47.748
Total		12080298	609033	100.000	100.000

3n




O
Ts
N
Ph
Ph
Me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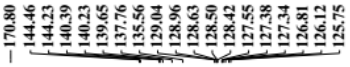
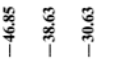
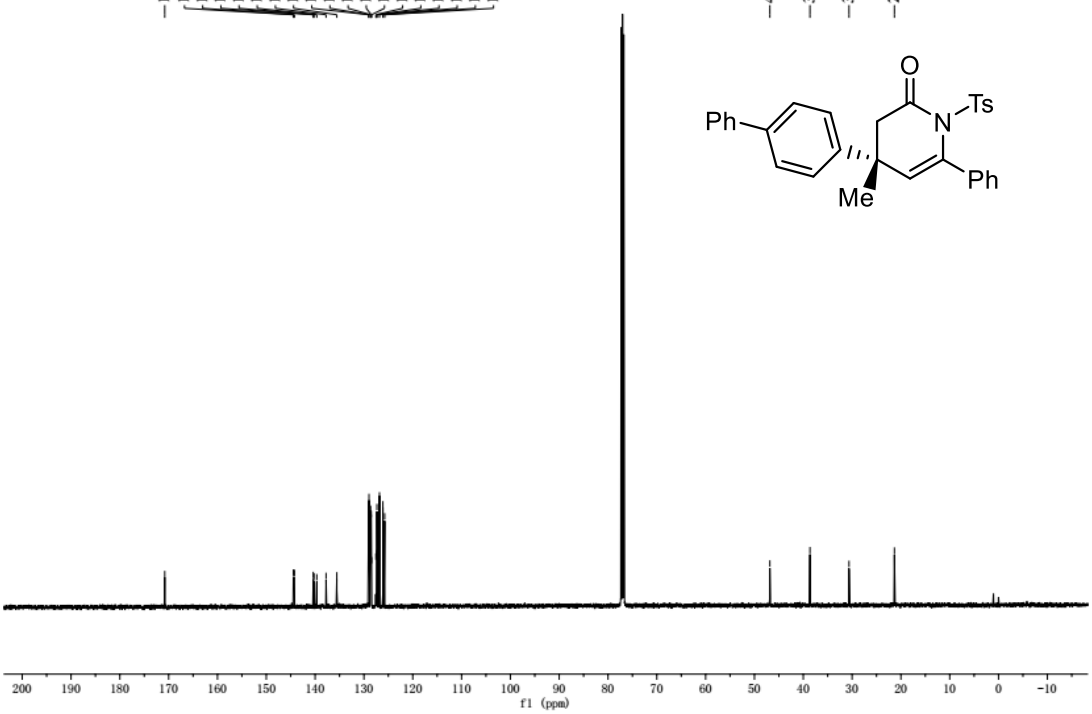
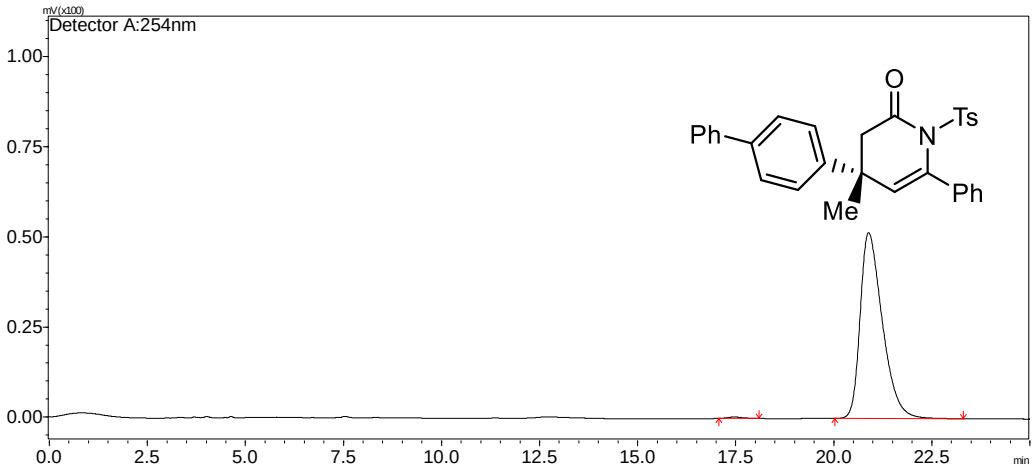
1.00
1.00
3.00
3.00
f1 (ppm)



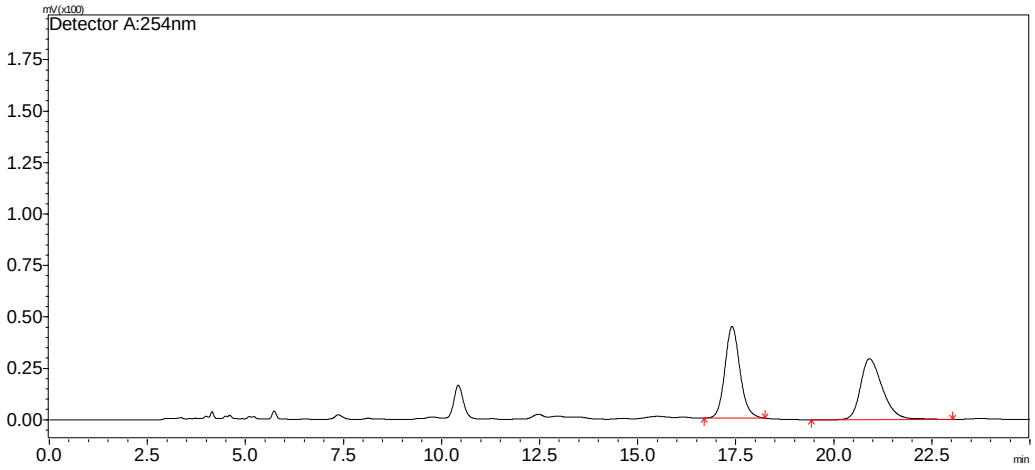



f1 (ppm)

O
Ts
N
Ph
Ph
Me

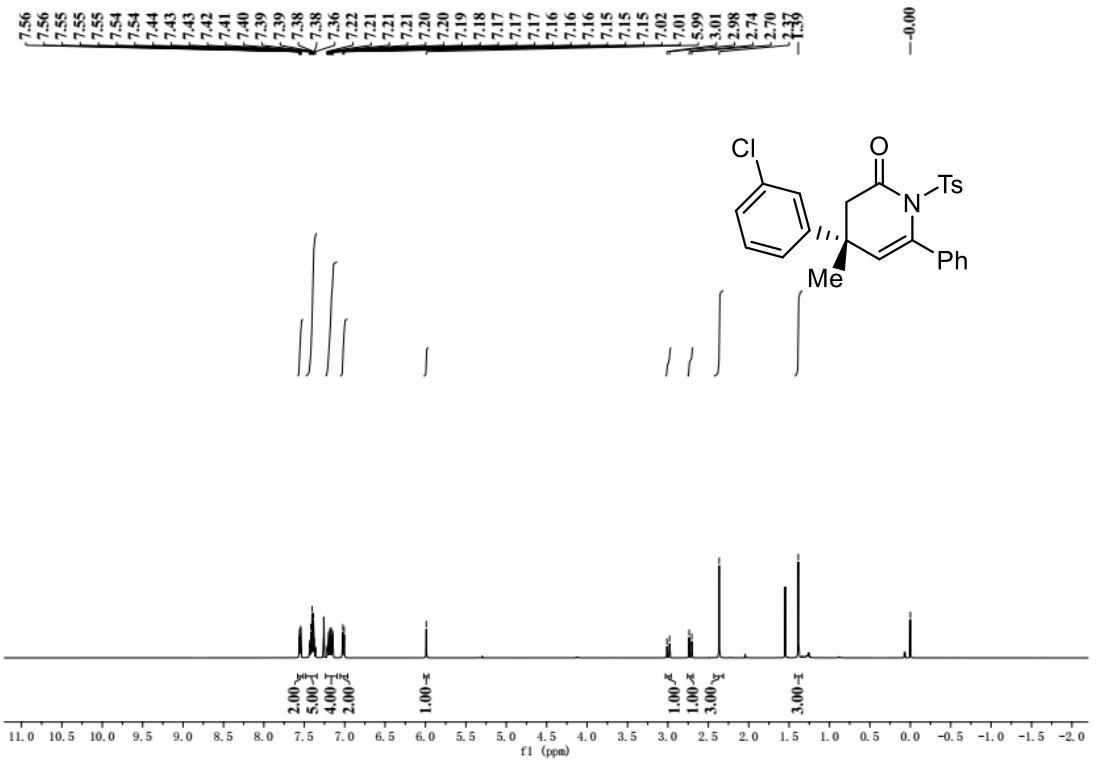


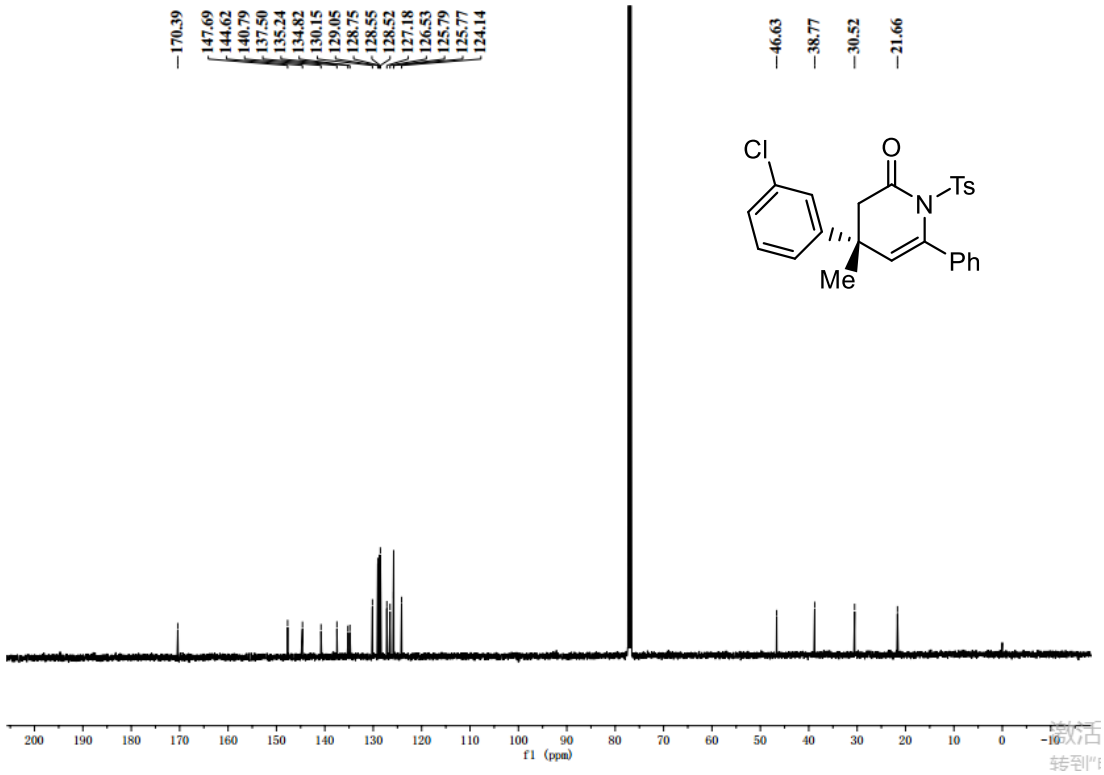
Peak	Ret .Time	Area	Height	Area %	Height %
1	17.457	7512	351	0.368	0.676
2	20.884	2034949	51576	99.632	99.324
Total		2042461	51927	100.000	100.000

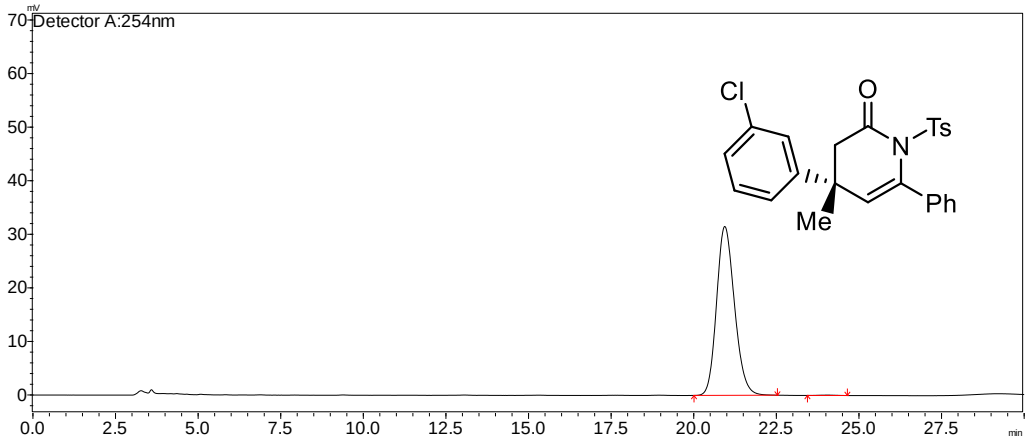


Peak	Ret .Time	Area	Height	Area %	Height %
1	17.403	1196023	44521	50.733	60.001
2	20.908	1161443	29680	49.267	39.999
Total		2357466	74200	100.000	100.000

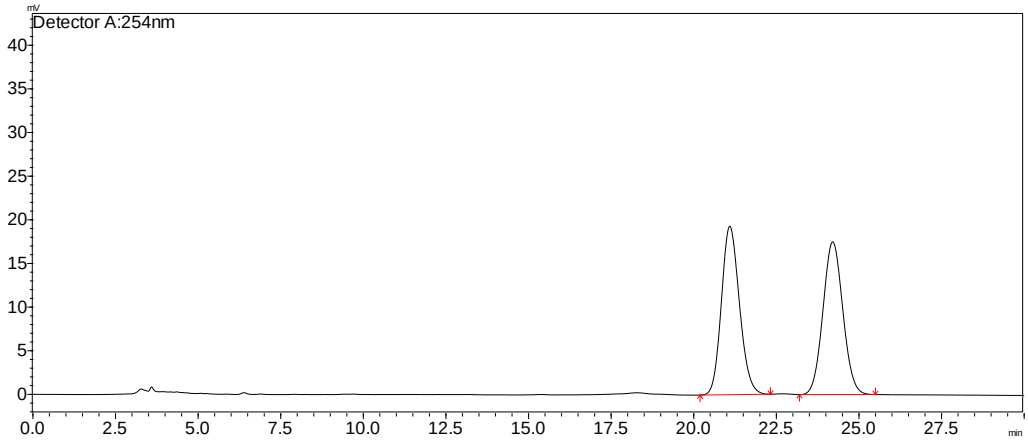
3o





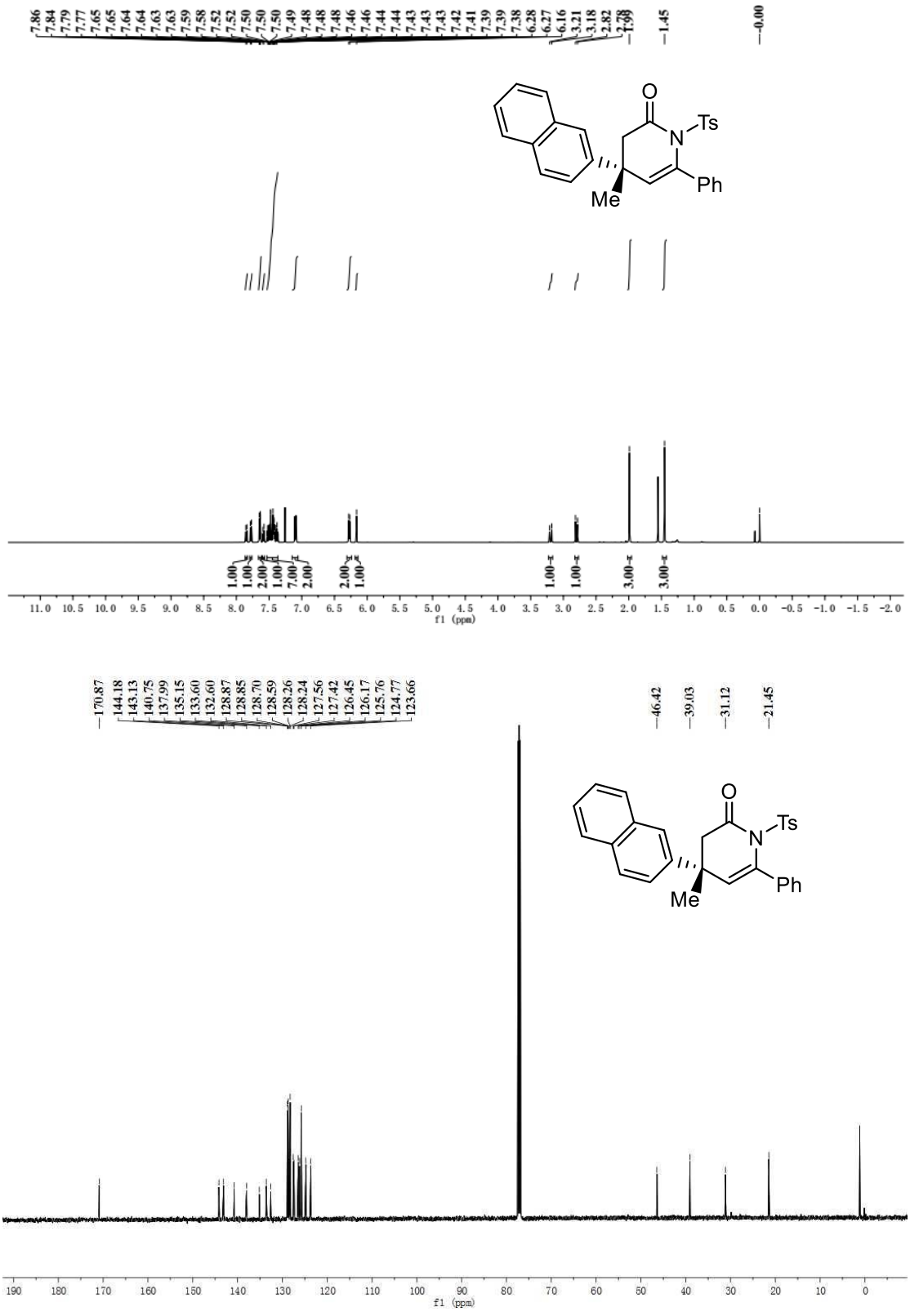


Peak	Ret .Time	Area	Height	Area%	Height%
1	20.934	11189601	31522	99.792	99.777
2	23.993	2482	70	0.208	0.223
Total		1192082	31593	100.000	100.000



Peak	Ret .Time	Area	Height	Area%	Height%
1	21.086	733323	19306	49.993	52.449
2	24.202	733535	17502	50.007	47.551
Total		1466858	36808	100.000	100.000

3p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7.41
7.39
7.39
7.38
6.28
6.27
6.16
3.21
3.18
2.82
1.99
1.45
0.00
O
N
Ts
Ph
Me
1.00
1.00
2.00
1.00
7.00
2.00
2.00
1.00
1.00
1.00
3.00
3.00
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f1 (ppm)
170.87
144.18
143.13
140.75
137.99
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133.60
132.60
128.87
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128.24
127.56
127.42
126.45
126.17
125.76
124.77
123.66
46.42
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21.45
O
N
Ts
Ph
Me
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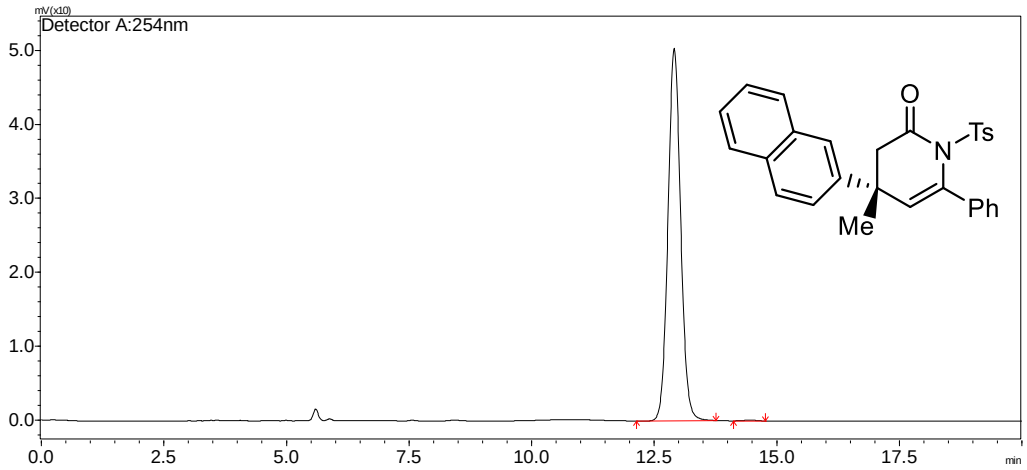
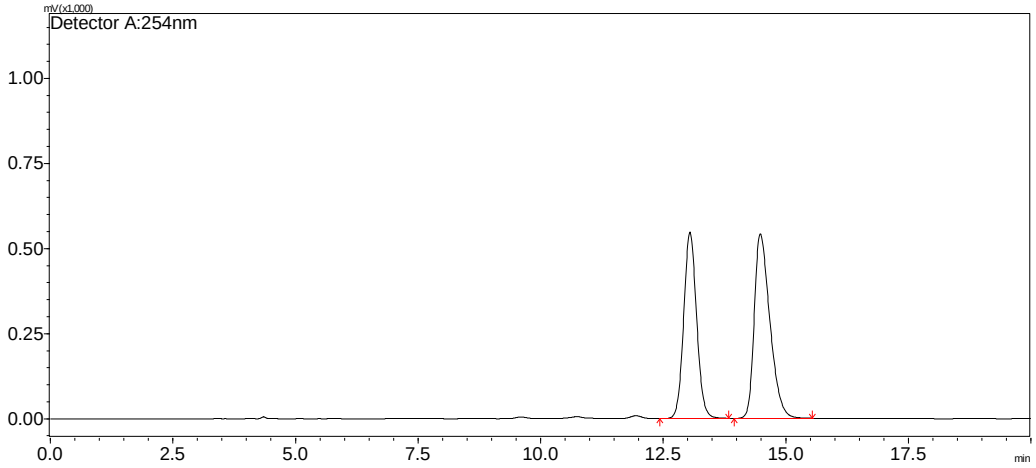
f1 (ppm)

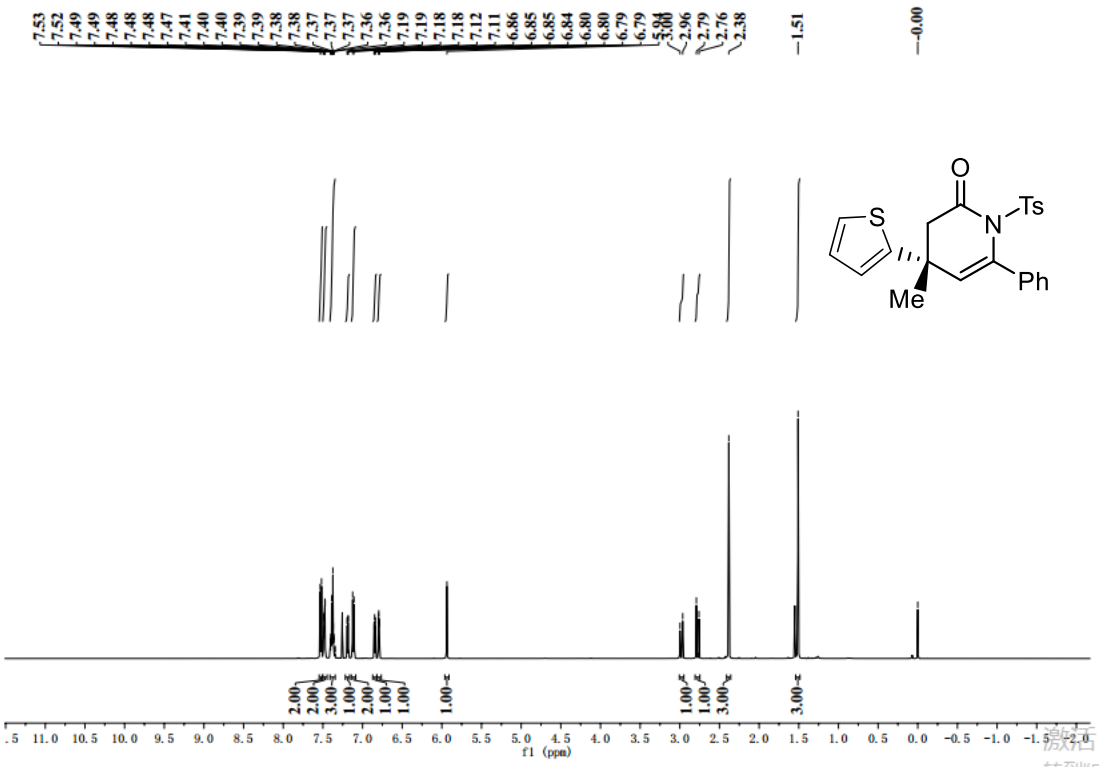


Peak	Ret .Time	Area	Height	Area %	Height %
1	12.904	909329	50386	99.838	99.814
2	14.444	1477	94	0.162	0.186
Total		910806	560480	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	13.042	1003281	547225	45.516	50.233
2	14.478	12009471	524141	54.484	49.767
Total		22042291	1089365	100.000	100.000

3q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7.11
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6.84
6.80
6.80
6.79
6.79
5.94
3.00
2.96
2.79
2.76
2.38
1.51
0.00
O
S
N
Ts
Ph
Me
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2.00
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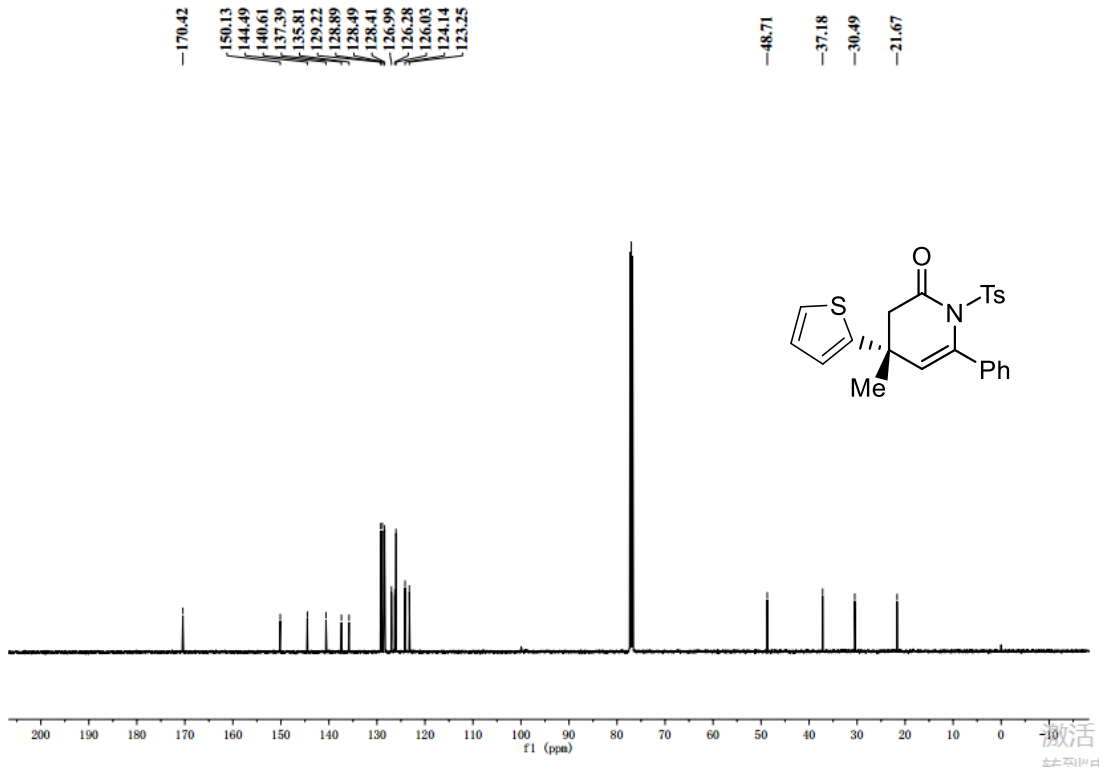
1.00
2.00
1.00
1.00
1.00
1.00
3.00
3.00
f1 (ppm)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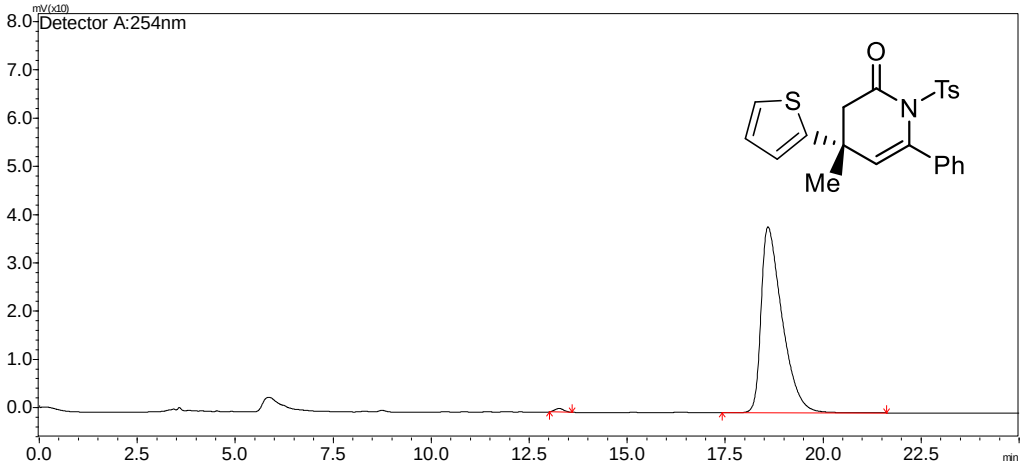
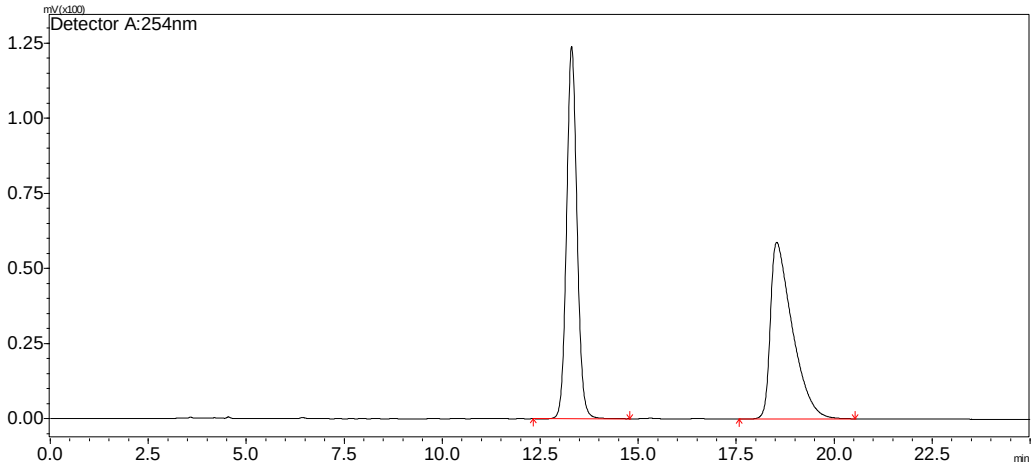
137.39
135.81
129.22
128.89
128.49
128.41
126.99
126.28
126.03
124.14
123.25
48.71
37.18
30.49
21.67
O
S
N
Ts
Ph
Me
f1 (ppm)

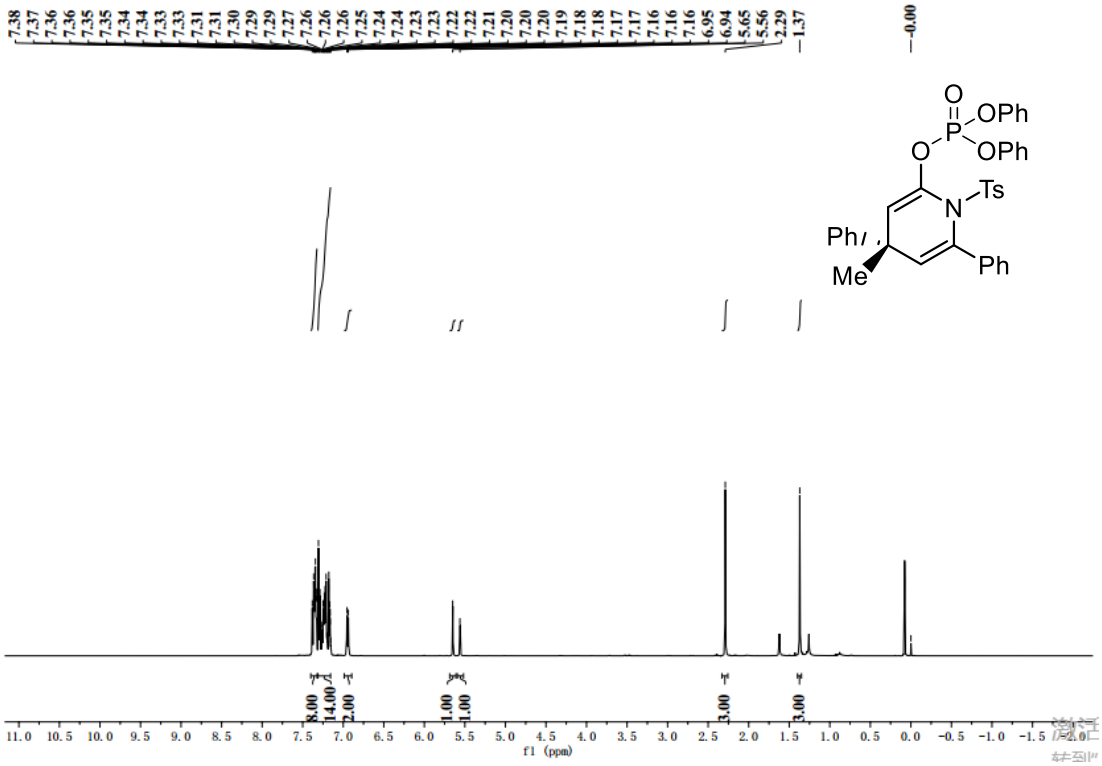


Peak	Ret .Time	Area	Height	Area %	Height %
1	13.255	11961	741	0.826	1.884
2	18.588	1436024	38584	99.174	98.116
Total		1447985	39324	100.000	100.000



Peak	Ret .Time	Area	Height	Area %	Height %
1	13.298	2293646	123913	50.022	67.800
2	18.531	2291673	58849	49.978	32.200
Total		4585319	182761	100.000	100.000

5a


7.38 7.37 7.36 7.36 7.35 7.35 7.34 7.34 7.33 7.33 7.31 7.31 7.30 7.29 7.29 7.27 7.26 7.26 7.26 7.25 7.24 7.24 7.23 7.23 7.22 7.22 7.21 7.20 7.20 7.20 7.19 7.18 7.18 7.17 7.17 7.16 7.16 7.16 6.95 6.94 5.65 5.56 2.29 1.37
0.00
O
OPh
P
O
OPh
Ts
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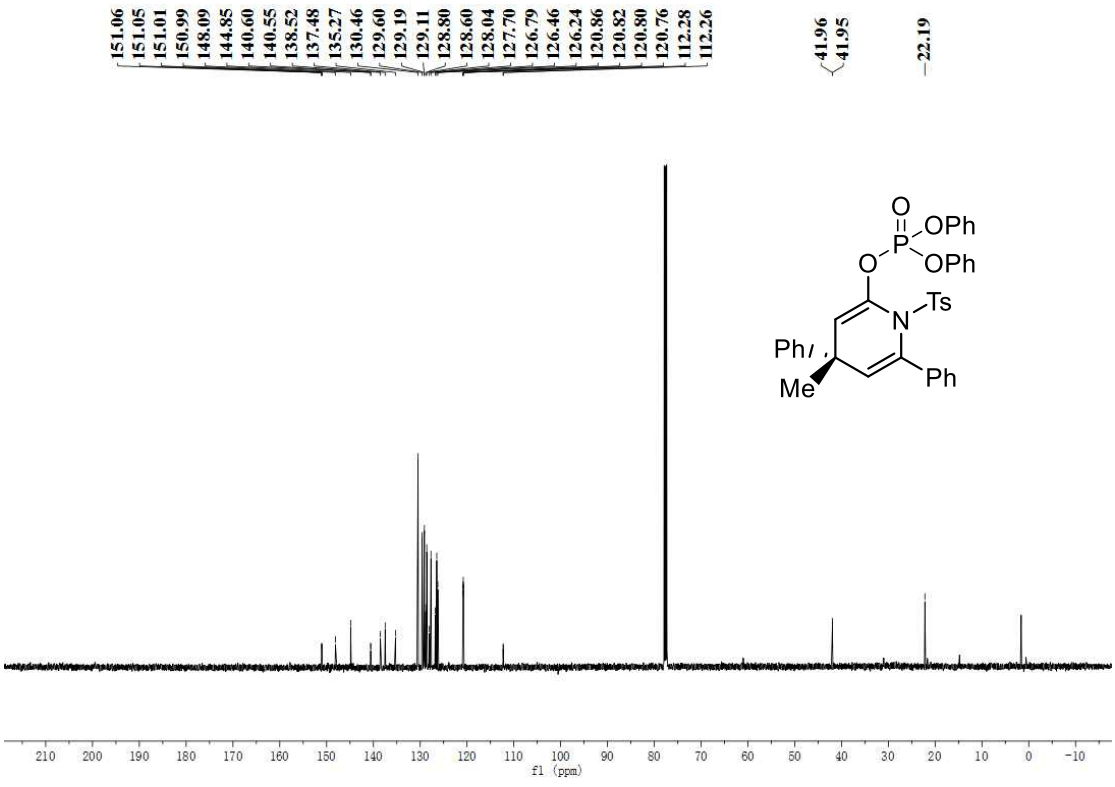
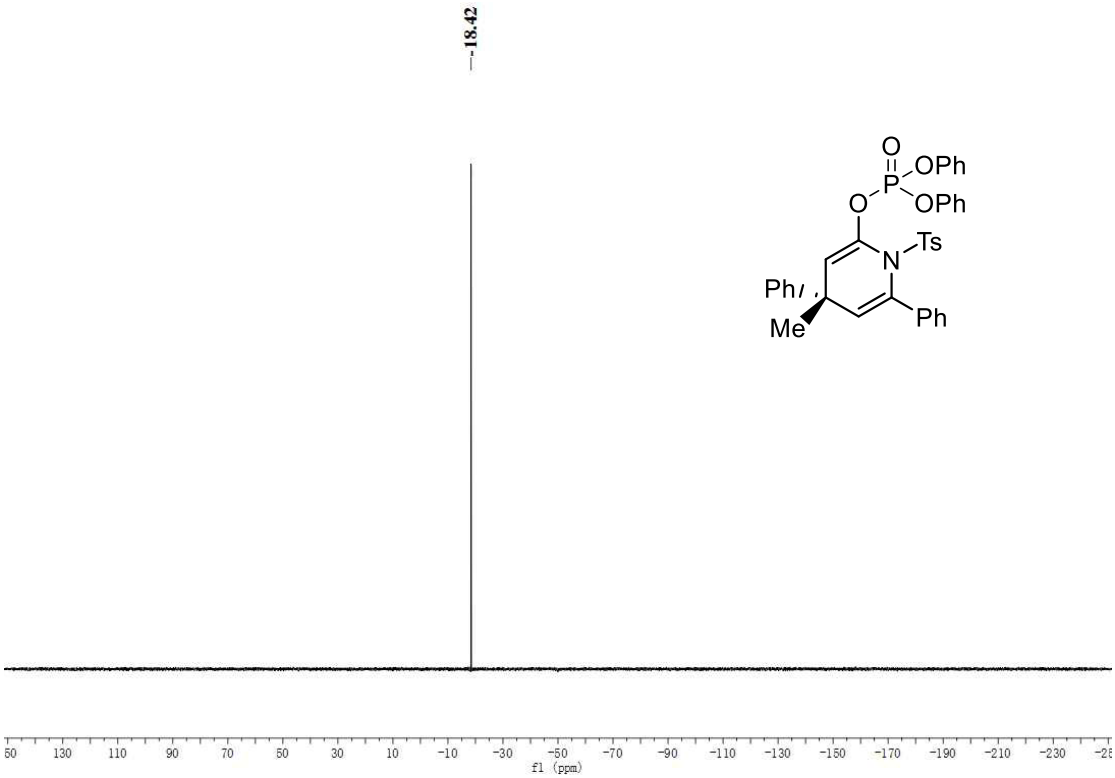
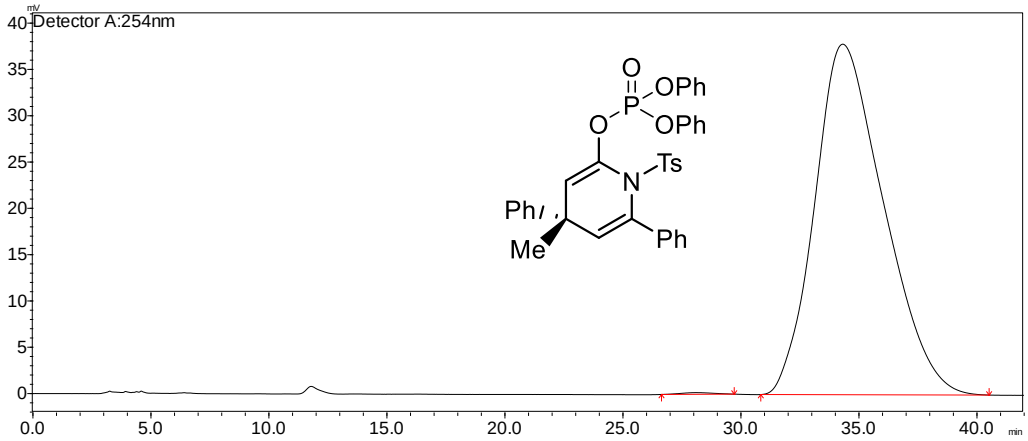
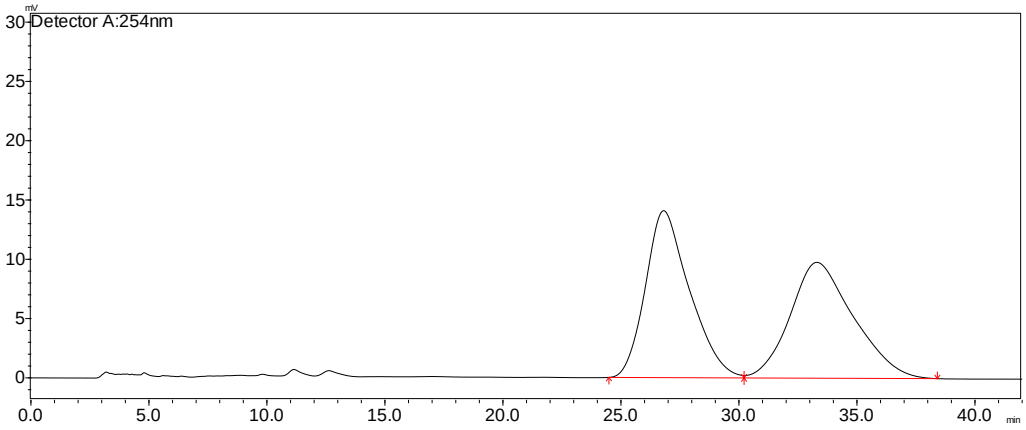
Ph
Me
Ph
8.00 14.00 2.00 1.00 1.00 3.00 3.00
11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5 -2.0
f1 (ppm)


151.06 151.05 151.01 150.99 148.09 144.85 140.60 140.55 138.52 137.48 135.27 130.46 129.60 129.19 129.11 128.80 128.60 128.04 127.70 126.79 126.46 126.24 120.86 120.82 120.80 120.76 112.28 112.26
41.96 41.95
22.19
O
OPh
P
O
OPh
Ts
N
Ph
Me
Ph
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)



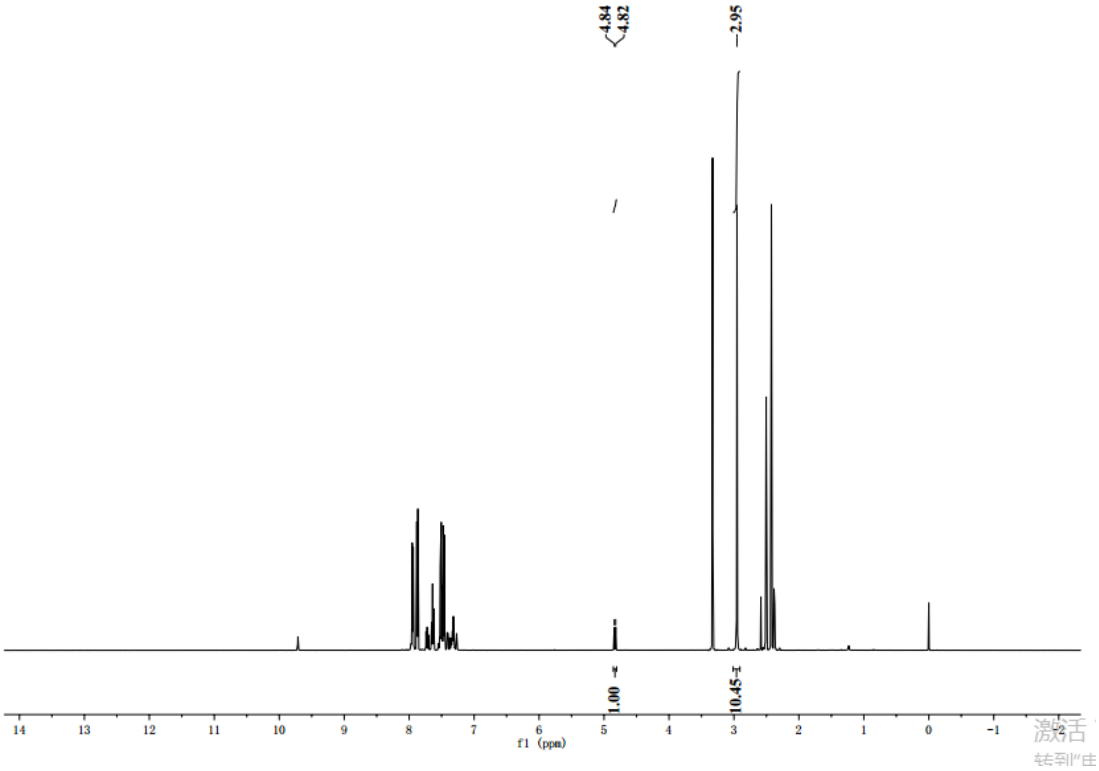


Peak	Ret .Time	Area	Height	Area%	Height%
1	28.093	17401	182	0.225	0.477
2	34.309	7705068	37857	99.775	99.523
Total		7722469	38038	100.000	100.000

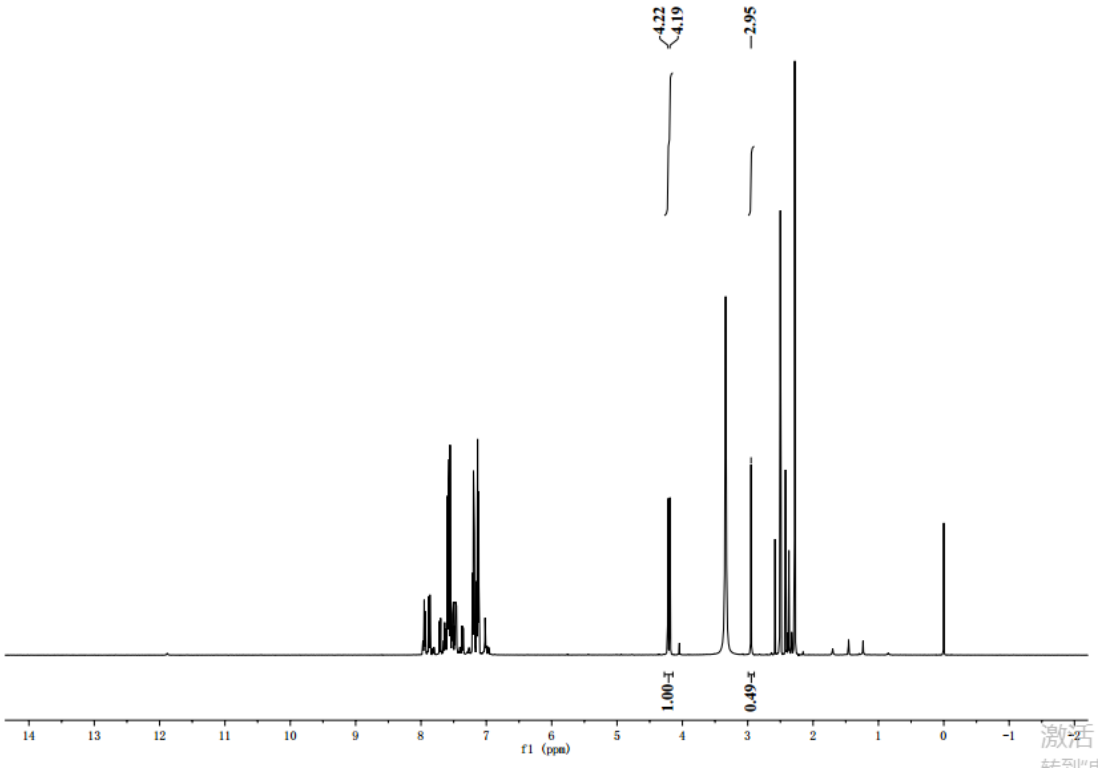


Peak	Ret .Time	Area	Height	Area%	Height%
1	26.806	1794060	13992	50.596	59.292
2	33.297	1751807	9607	49.404	40.708
Total		3545867	23599	100.000	100.000

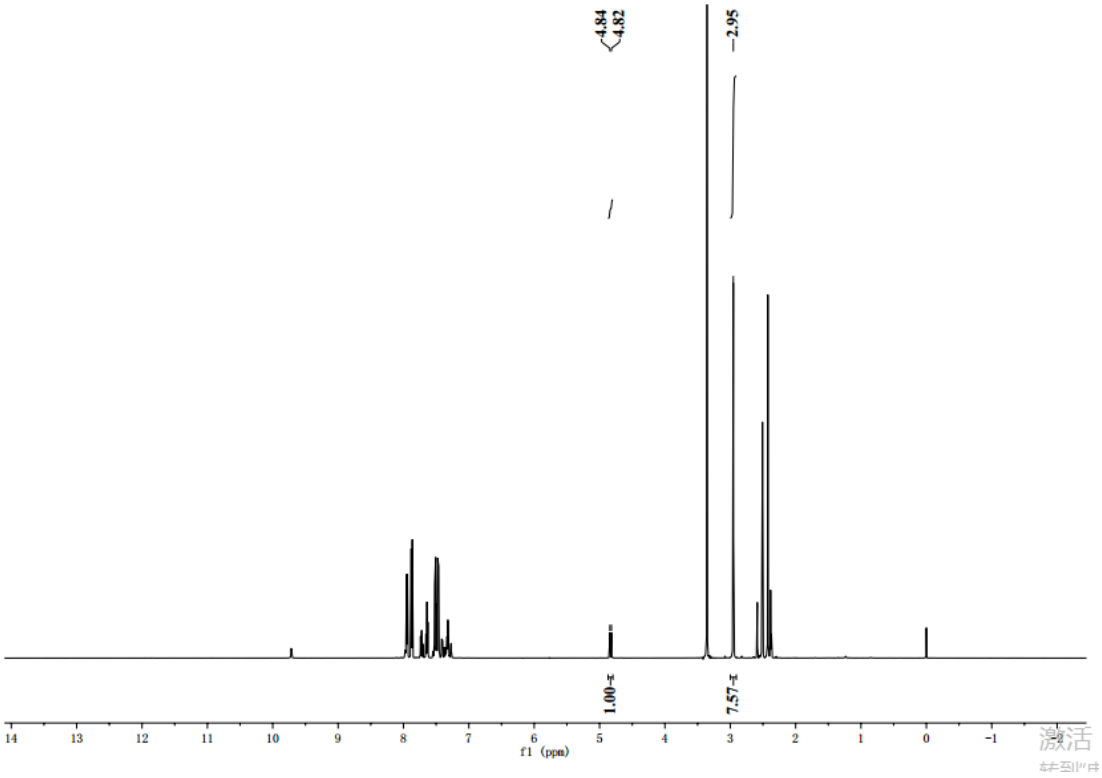
Imine 6 dissolved in d-DMSO



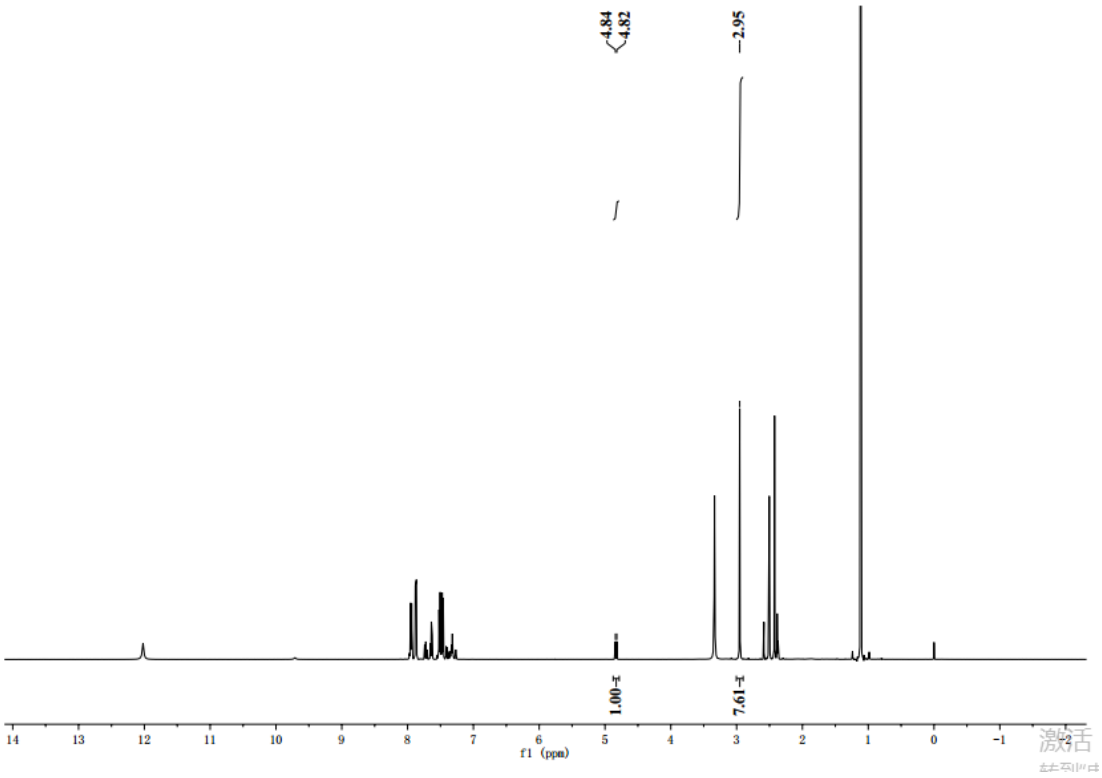
Imine 6 and LiCl dissolved in d-DMSO



Imine 6 and LiBr dissolved in d-DMSO



Imine 6 and ^{t}BuCOOH dissolved in d-DMSO



Imine 6 and NaCl dissolved in d-DMSO