Supporting Information for

Organocatalyzed enantioselective one-pot three-component access to indoloquinolizidines by a Michael addition-Pictect-Spengler sequence

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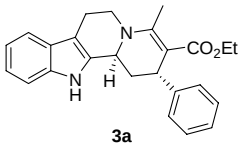
General Information: Thin-layer chromatography (TLC) carried out on 0.25 mm silica gel plates visualized with UV light and/or by staining with ethanolic phosphomolybdic acid (PMA) or iodine. Flash column chromatography was performed on silica gel H (10-40 μ). NMR spectra were recorded on Bruker AM500 (500MHz). Chemical shifts (δ) are given in ppm relative to TMS, coupling constants (J) in Hz. Optical rotations were taken on JASCO P1030. High-resolution mass spectra were recorded on Bruker ApeXIII 7.0 TESLA FTMS. Enantiomeric excesses were determined by chiral HPLC using a Waters instrument.

General Procedure for Multicatalytic Cascade Michael/Benzoin Reaction:

Step 1: To a mixture of catalyst **A** or **B** (0.01 mmol) and benzoic acid (0.01 mmol) in toluene (0.2 mL) was added β-ketonester (0.1 mmol) under an atmosphere of N_2. Followed by the addition of α,β-unsaturated aldehyde (0.2 mmol). The reaction was followed by TLC. ***Step 2***: After full conversion of β-ketoneester, the reaction mixture was diluted with toluene (2 mL). Then tryptamine (0.2 mmol) and benzoic acid (0.2 mmol) was added. The reaction mixture was heated at 50 ^{o}C for 12-24h. The reaction mixture was diluted with EA (20 mL) and washed with saturated $NaHCO_3$ (10 mL), brine (10 mL) sequentially. The organic layer was dried over Na_2SO_4 and concentrated in vacuo to afford a crude product which was purified by flash silica gel chromatography.



3a

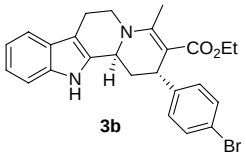
(2*S*,12b*S*)-Ethyl 4-methyl-2-phenyl-1,2,6,7,12,12b-hexahydroindolo[2,3α]quinolizine-3-carboxylate (**3a**):

The reaction was carried out at -15 ^{o}C for 12h using **A** as catalyst (step 1) and stirred for 12h at 50 ^{o}C (step 2), the title product was isolated as a white powder (78% yield, 92% ee):

^{1}H NMR ($CDCl_3$, 500M): δ 7.67 (s, 1H), 7.48 (d, J = 7.5 Hz, 1H), 7.30-7.35 (m, 2H), 7.23-7.28 (m, 3H), 7.20 (t, J = 7.5 Hz, 1H), 7.05-7.16 (m, 2H), 4.25-4.40 (m, 3H), 3.90-4.00 (m, 2H), 3.14-3.24 (m, 1H), 2.82-2.91 (m, 1H), 2.73-2.80 (m, 1H), 2.66 (s, 3H), 2.29 (dt, J = 12.5 Hz and 3.5 Hz, 1H), 2.07 (td, J = 12.5 Hz and 5 Hz, 1H), 0.99 (t, J = 7.5 Hz, 3H).

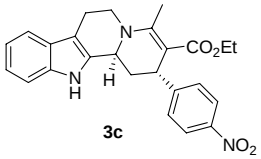
^{13}C NMR ($CDCl_3$, 125M): δ 169.1, 154.9, 147.1, 136.1, 134.1, 128.3, 127.9, 126.8, 126.0, 121.7,

119.6, 118.0, 110.9, 109.1, 96.8, 58.9, 49.6, 44.8, 38.1, 36.0, 22.4, 17.6, 14.3.
$[\alpha]^{25}_{D}$ 140.0 (*c* 0.70, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{25}H_{27}N_2O_2)^+$ 387.2064, found 387.2067.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 10.77 min (minor enantiomer), t_R = 28.06 min (major enantiomer).



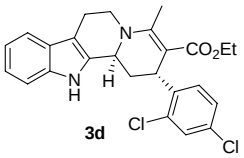
3b

(2*S*,12b*S*)-ethyl
2-(4-bromophenyl)-4-methyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3b**): The reaction was carried out at -15 °C for 24h using **A** as catalyst (step 1) and stirred for 20h at 50 °C (step 2), the title product was isolated as a yellow solid (76% yield, 93% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.75 (s, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.40-7.45 (m, 2H), 7.24-7.28 (m, 2H), 7.05-7.16 (m, 5H), 4.31-4.36 (m, 1H), 4.22-4.28 (m, 2H), 3.90-3.98 (m, 2H), 3.14-3.22 (m, 1H), 2.82-2.90 (m, 1H), 2.74-2.80 (m, 1H), 2.65 (s, 3H), 2.26 (ddd, *J* = 13 Hz 3.5 Hz and 2.5 Hz, 1H), 2.07 (td, *J* = 13 Hz and 4.5 Hz, 1H), 1.01 (t, *J* = 7 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ 168.9, 155.2, 146.3, 136.2, 133.8, 131.4, 129.7, 122.0, 119.8, 118.2, 111.0, 109.3, 96.4, 59.1, 49.6, 44.9, 37.9, 36.0, 22.5, 17.6, 14.4.
$[\alpha]^{25}_{D}$ 183.4 (*c* 0.65, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{25}H_{26}N_2O_2Br)^+$465.1194, found 465.1172.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 7.82 min (minor enantiomer), t_R = 30.76 min (major enantiomer).



3c

(2*S*,12b*S*)-ethyl
4-methyl-2-(4-nitrophenyl)-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3c**): The reaction was carried out at 10 °C for 48h using **B** as catalyst (step 1) and stirred for 24h at 50 °C (step 2), the title product was isolated as a yellow solid (91% yield, 93% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 8.15 (d, *J* = 3.5 Hz, 2H), 7.85 (s, 1H), 7.48 (d, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 8.5 Hz, 2H), 7.27 (d, *J* = 8 Hz, 1H), 7.08-7.18 (m, 2H), 4.35-4.42 (m, 2H), 4.26 (d, *J* = 7 Hz, 1H), 3.90-3.97 (m, 2H), 3.18-3.26 (m, 1H), 2.76 -2.94 (m, 2H), 2.70 (s, 3H), 2.31-2.38 (m, 1H), 2.15 (td, *J* = 12.5 Hz and 5.5 Hz, 1H),0.98 (t, *J* = 7 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ 168.6, 155.8, 155.4, 146.5, 136.3, 133.3, 129.2, 128.8, 126.8, 124.5, 123.7, 122.2, 119.9, 118.3, 111.1, 109.5, 95.6, 59.2, 49.6, 45.0, 38.6, 35.8, 22.4, 17.6, 14.4.
$[\alpha]^{25}_{D}$ 96.0 (*c* 0.33, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{25}H_{26}N_3O_4)^+$432.1909, found 432.1918.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 16.18 min (minor enantiomer), t_R = 45.13 min (major enantiomer).



3d

(2*R*,12b*S*)-Ethyl 2-(2,4-dichlorophenyl)-4-methyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3d**): The reaction was carried out at 10 °C for 48h using **B** as catalyst (step 1) and stirred for 24h at 50 °C (step 2), the title product was isolated as a yellow solid (86% yield, 95% ee):

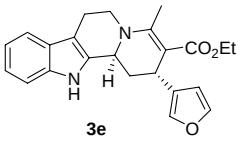
^{1}H NMR ($CDCl_3$, 500M): δ 7.73 (s, 1H), 7.83 (d, *J* = 7.5 Hz, 1H), 7.42 (d, *J* = 2 Hz, 1H), 7.26-7.29 (m, 1H), 7.07-7.19 (m, 4H), 4.65 (d, *J* = 4.5 Hz, 1H), 4.36 (dd, *J* = 13 Hz and 2 Hz, 1H), 4.25 (d, *J* = 13 Hz, 2H), 3.92 (q, *J* = 7.5 Hz, 3H), 3.16-3.25 (m, 1H), 2.84-2.94 (m, 1H), 2.76-2.83 (m, 1H), 2.68 (s, 3H), 2.31-2.37 (m, 1H), 2.15 (td, *J* = 13 Hz and 6 Hz, 1H), 0.98 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125M): δ 168.6, 155.8, 142.6, 136.2, 133.9, 133.6, 132.4, 130.7, 129.5, 126.9, 126.8, 126.8, 122.1, 119.9, 118.2, 111.1, 109.4, 96.2, 59.2, 49.7, 44.9, 35.3, 33.1, 22.5, 17.6, 14.3.

$[\alpha]^{25}_D$ 81.6 (*c* 1.75, $CHCl_3$).

HRMS (ESI) calcd. for $(C_{25}H_{25}N_2O_2Cl_2)^+$ 455.1289, found 455.1287.

HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 9.44 min (minor enantiomer), t_R = 28.50 min (major enantiomer).



3e

(2*S*,12b*S*)-Ethyl 2-(furan-3-yl)-4-methyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3e**): The reaction was carried out at 10 °C for 24h using **B** as catalyst (step 1) and stirred for 24h at 50 °C (step 2), the title product was isolated as a yellow solid (93% yield, 88% ee):

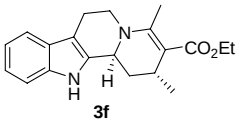
^{1}H NMR ($CDCl_3$, 500M): δ 7.80 (s, 1H), 7.48 (d, *J* = 8 Hz, 1H), 7.35 (s, 1H), 7.30 (d, *J* = 8 Hz, 1H), 7.09-7.18 (m, 2H), 6.27-6.30 (m, 1H), 5.96-5.99 (m, 1H), 4.24-4.40 (m, 3H), 4.00-4.12 (m, 2H), 3.20-3.30 (m, 1H), 2.72-2.90 (m, 2H), 2.59 (s, 3H), 2.53-2.57 (m, 1H), 1.91 (td, *J* = 12.5 Hz and 5 Hz, 1H), 1.13 (t, *J* = 7 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125M): δ 169.0, 159.2, 154.9, 141.2, 136.2, 134.0, 126.9, 122.1, 119.9, 118.2, 111.1, 110.3, 109.2, 106.6, 95.4, 59.2, 50.6, 44.9, 32.7, 32.4, 22.5, 17.7, 14.6.

$[\alpha]^{25}_D$ 19.2 (*c* 0.31, $CHCl_3$).

HRMS (ESI) calcd. for $(C_{23}H_{25}N_2O_3)^+$ 377.1857, found 377.1859.

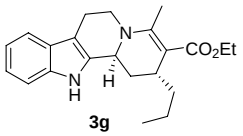
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R =13.44 min (minor enantiomer), t_R = 38.10 min (major enantiomer).



3f

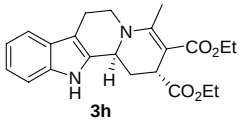
(2*R*,12b*S*)-ethyl 2,4-dimethyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3f**): The reaction was carried out at 10 °C for 24h using **B** as catalyst (step 1) and stirred for 24h at 50

^{o}C (step 2), the title product was isolated as a yellow oil (56% yield, 81% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.94 (s, 1H), 7.49 (d, *J* = 7.5 Hz, 1H), 7.33 (d, *J* = 7.5 Hz, 1H), 7.09-7.18 (m, 2H), 4.52 -4.58 (m, 1H), 4.07-4.30 (m, 3H), 3.06-3.24 (m, 2H), 2.72-2.90 (m, 2H), 2.49 (s, 3H), 2.05 -2.11 (m, 1H), 1.81 (td, *J* = 12.5 Hz and 5 Hz, 1H), 1.29 (t, *J* = 7 Hz, 3H), 1.17 (d, *J* = 7 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ 169.5, 153.1, 136.2, 134.6, 127.0, 121.9, 119.8, 118.2, 111.1, 109.1, 101.1, 59.1, 49.8, 44.9, 34.8, 26.5, 22.5, 22.2, 17.7, 14.7.
$[\alpha]^{25}_{D}$ -45.1 (*c* 1.7, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{20}H_{25}N_2O_2)^+$325.1923, found 325.1910.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R =11.07 min (minor enantiomer), t_R = 19.37 min (major enantiomer).



3g

(2*R*,12b*S*)-Ethyl
4-methyl-2-propyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3g**): The reaction was carried out at 10 ^{o}C for 48h using **B** as catalyst (step 1) and stirred for 24h at 50 ^{o}C (step 2), the title product was isolated as a yellow solid (68% yield, 85% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.82 (s, 1H), 7.49 (d, *J* = 8 Hz, 1H), 7.34 (d, *J* = 8 Hz, 1H), 7.09-7.19 (m, 2H), 4.49 -4.55 (m, 1H), 4.05-4.28 (m, 2H), 3.12 -3.20 (m, 1H), 2.93-2.99 (m, 1H), 2.72-2.8 0 (m, 2H), 2.47 (s, 3H), 2.20 -2.26 (m, 1H), 1.70 (td, *J* = 12.5 Hz and 5 Hz, 1H), 1.45-1.57 (m, 2H), 1.30-1.45 (m, 2H), 1.28 (t, *J* = 7 Hz, 3H), 0.97 (d, *J* = 6.5 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ 169.6, 152.8, 136.1, 134.7, 126.9, 121.8, 119.7, 118.1, 110.9, 108.9, 100.3, 59.0, 50.0, 45.0, 38.2, 36.1, 31.2, 30.9, 22.5, 20.4, 17.6, 14.6, 14.2.
$[\alpha]^{25}_{D}$ -44.8 (*c* 1.50, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{22}H_{28}N_2O_2Na)^+$375.2054, found 375.2043.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 9.36 min (minor enantiomer), t_R = 17.79 min (major enantiomer).

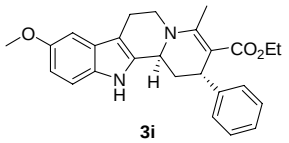


3h

(2*R*,12b*S*)-Diethyl 4-methyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-2,3-dicarboxylate (**3g**): The reaction was carried out at 10 ^{o}C for 2h using **B** as catalyst (step 1) and stirred for 18h at 50 ^{o}C (step 2), the title product was isolated as a yellow solid (59% yield, 69% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.90 (s, 1H), 7.49 (d, *J* = 8 Hz, 1H), 7.34 (d, *J* = 8 Hz, 1H), 7.10 -7.20 (m, 2H), 4.52 -4.58 (m, 1H), 4.05-4.30 (m, 3H), 3.83-3.87 (m, 1H), 3.15-3.24 (m, 1H), 2.74 -2.88 (m, 2H), 2.58 -2.61 (m, 1H), 2.56 (s, 3H), 1.88 (td, *J* = 13 Hz and 5.5 Hz, 1H), 1.31 (t, *J* = 7.5 Hz, 3H),1.24 (t, *J* = 7 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ 175.9, 168.8, 155.0, 136.3, 133.5, 126.8, 122.1, 119.8, 118.3, 111.2, 109.2, 93.8, 60.9, 59.4, 51.0, 44.8, 38.9, 30.5, 22.4, 17.5, 14.6, 14.3.
$[\alpha]^{25}_{D}$ 83.7 (*c* 0.33, $CHCl_3$).

HRMS (ESI) calcd. for $(C_{22}H_{27}N_2O_4)^+$383.1973, found 383.1965.

HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R =13.03 min (minor enantiomer), t_R = 32.37 min (major enantiomer).



3i

(2*S*,12b*S*)-Ethyl 9-methoxy-4-methyl-2-phenyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3i**): The reaction was carried out at 10 °C for 24h using **B** as catalyst (step 1) and stirred for 2h at 50 °C (step 2), the title product was isolated as a yellow solid (70% yield, 94% ee):

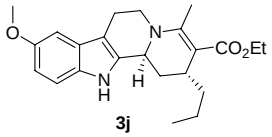
^{1}H NMR ($CDCl_3$, 500M): δ 7.58 (s, 1H), 7.10 -7.33 (m, 6H), 6.90-6.94 (m, 1H), 6.76-6.81 (m, 1H), 4.24 -4.38 (m, 3H), 3.90-3.98 (m, 2H), 3.84 (s, 3H), 3.12-3.22 (m, 1H), 2.80-2.88 (m, 1H), 2.68 -2.76 (m, 1H), 2.65 (s, 3H), 2.24-2.31 (m, 1H), 2.06 (td, *J* = 12.5 Hz and 5 Hz, 1H), 0.99 (t, *J* = 7.5 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125M): δ 169.1, 154.8, 154.3, 147.2, 135.1, 131.2, 128.4, 127.9, 127.3, 126.1, 122.1, 111.6, 109.1, 93.8, 100.5, 97.0, 59.1, 56.1, 49.7, 44.9, 38.2, 36.1, 30.5, 22.6, 17.7, 14.4.

$[\alpha]^{25}_D$ 119.4 (*c* 1.05, $CHCl_3$).

HRMS (ESI) calcd. for $(C_{26}H_{29}N_2O_3)^+$ 417.2188, found 417.2173.

HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R =16.32 min (minor enantiomer), t_R = 42.91 min (major enantiomer).



3j

(2*R*,12b*S*)-ethyl 9-methoxy-4-methyl-2-propyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3j**): The reaction was carried out at -15 °C for 48h using **A** as catalyst (step 1) and stirred for 1h at 50 °C (step 2), the title product was isolated as a yellow solid (93% yield, 93% ee):

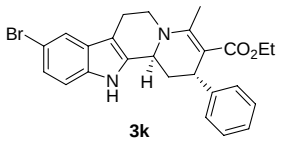
^{1}H NMR ($CDCl_3$, 500M): δ 7.81 (s, 1H), 7.22 (d, *J* = 8.5 Hz, 1H), 6.94 (d, *J* = 2.5 Hz, 1H), 6.82 (dd, *J* = 9 Hz and 2.5 Hz, 1H), 4.53 (d, *J* = 13 Hz, 1H), 4.09-4.30 (m, 3H), 3.88 (s, 3H), 3.14-3.23 (m, 1H), 2.95-3.02 (m, 1H), 2.80-2.89 (m, 1H), 2.70-2.77 (m, 1H), 2.50 (s, 3H), 2.24 (ddd, *J* = 13 Hz, 5 Hz and 2.5 Hz, 1H), 1.72 (td, *J* = 13 Hz and 5 Hz, 1H), 1.46-1.59 (m, 2H), 1.32-1.45 (m, 2H), 1.30 (t, *J* = 7.5 Hz, 3H), 0.99 (t, *J* = 7 Hz, 3H).

^{13}C NMR ($CDCl_3$, 125M): δ169.6, 154.2, 152.7, 135.6, 131.1, 127.4, 111.6, 111.5, 108.8, 100.4, 100.3, 58.9, 55.9, 50.1, 45.0, 38.2, 31.3, 30.9, 22.5, 20.4, 17.6, 14.6, 14.3.

$[\alpha]^{25}_D$ -22.8 (*c* 1.60, $CHCl_3$).

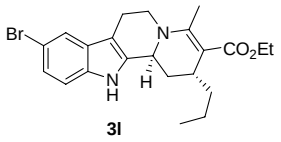
HRMS (ESI) calcd. for $(C_{23}H_{31}N_2O_3)^+$ 383.2323, found 383.2329.

HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R =16.30 min (minor enantiomer), t_R = 35.88 min (major enantiomer).



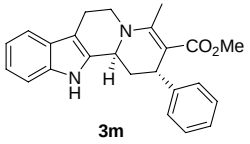
3k

(2*S*,12b*S*)-Ethyl
9-bromo-4-methyl-2-phenyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3k**): The reaction was carried out at 10 ^{o}C for 24h using **B** as catalyst (step 1) and stirred for 20h at 50 ^{o}C (step 2), the title product was isolated as a yellow solid (67% yield, 94% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.87 (s, 1H), 7.58 (d, *J* = 2 Hz, 1H), 7.26-7.33 (m, 2H), 7.17-7.25 (m, 4H), 7.09 (d, *J* = 4 Hz, 1H), 4.22 -4.35 (m, 3H), 3.89-3.97 (m, 2H), 3.10-3.19 (m, 1H), 2.74-2.85 (m, 1H), 2.65-2.72 (m, 1H), 2.63 (s, 3H), 2.28 (ddd, *J* = 12.5 Hz, 3.5 Hz and 2.5 Hz, 1H), 2.05 (td, *J* = 12.5 Hz and 5.5 Hz, 1H), 1.46-1.59 (m, 2H), 0.98 (t, *J* = 7 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ169.1, 154.8, 146.9, 135.6, 134.8, 131.1, 128.7, 128.4, 127.9, 126.2, 124.6, 120.8, 112.9, 112.4, 108.8, 97.2, 59.1, 49.6, 44.8, 38.2, 35.9, 22.4, 17.7, 14.4.
$[\alpha]^{25}_{D}$ 59.6 (*c* 0.3, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{25}H_{26}N_2O_2Br)^+$ 465.1194, found 465.1172.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 12.66 min (minor enantiomer), t_R = 38.74 min (major enantiomer).



3l

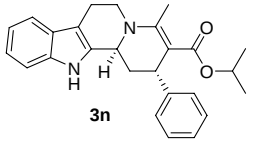
(2*R*,12b*S*)-ethyl
9-bromo-4-methyl-2-propyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3l**): The reaction was carried out at -15 ^{o}C for 48h using **A** as catalyst (step 1) and stirred for 18h at 50 ^{o}C (step 2), the title product was isolated as a yellow solid (57% yield, 92% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 8.06 (s, 1H), 7.60 (s, 1H), 7.17-7.25 (m, 2H), 4.51 (d, *J* = 13 Hz, 1H), 4.04-4.27 (m, 3H), 3.10-3.18 (m, 1H), 3.88 (s, 3H), 2.93-2.99 (m, 1H), 2.75-2.85 (m, 1H), 2.65-2.72 (m, 1H), 2.46 (s, 3H), 2.24 (ddd, *J* = 13 Hz, 4 Hz and 2.5 Hz, 1H), 1.69 (td, *J* = 13 Hz and 5 Hz, 1H), 1.45-1.56 (m, 2H), 1.29-1.43 (m, 2H), 1.27 (t, *J* = 7 Hz, 3H), 0.96 (t, *J* = 7 Hz, 3H).
^{13}C NMR ($CDCl_3$, 125M): δ169.5, 152.5, 136.1, 134.7, 128.7, 124.5, 120.8, 112.9, 112.3, 108.7, 100.6, 59.1, 49.9, 44.8, 38.1, 31.2, 30.9, 22.3, 20.4, 17.6, 14.6, 14.2.
$[\alpha]^{25}_{D}$ 55.3 (*c* 1.15, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{22}H_{28}N_2O_2Br)^+$ 431.1321, found 431.1328.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 11.89 min (minor enantiomer), t_R = 27.29 min (major enantiomer).



3m

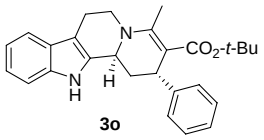
(2*S*,12b*S*)-Methyl

4-methyl-2-phenyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3m**): The reaction was carried out at -15 °C for 24h using **A** as catalyst (step 1) and stirred for 24h at 50 °C (step 2), the title product was isolated as a yellow solid (95% yield, 92% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.68 (s, 1H), 7.47 (d, *J* = 7.5 Hz, 1H), 7.29-7.35 (m, 2H), 7.19-7.28 (m, 4H), 7.06-7.17 (m, 2H), 4.35 (dd, *J* = 13 Hz and 3.5 Hz, 1H), 4.29-4.31 (m, 1H), 4.23-4.29 (m, 1H), 3.49 (s, 3H), 3.14-3.21 (m, 1H), 2.82-2.91 (m, 1H), 2.73-2.80 (m, 1H), 2.68 (s, 3H), 2.32 (ddd, *J* = 13 Hz, 3.5 Hz and 2.5 Hz, 1H), 2.06 (td, *J* = 13 Hz and 5 Hz, 1H).
^{13}C NMR ($CDCl_3$, 125M): δ 155.1, 146.7, 139.6, 136.1, 134.1, 128.5, 127.9, 126.9, 126.2, 121.9, 119.8, 118.2, 111.0, 109.2, 96.3, 50.8, 49.7, 45.0, 37.9, 36.2, 22.6, 17.6.
$[\alpha]^{25}_D$ 38.3 (*c* 1.05, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{24}H_{25}N_2O_2)^+$373.1903, found 373.1910.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 12.23 min (minor enantiomer), t_R = 39.82 min (major enantiomer).



3n

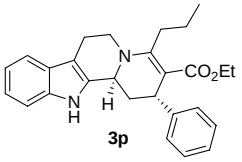
(2*S*,12b*S*)-Isopropyl
4-methyl-2-phenyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3n**): The reaction was carried out at -15 °C for 24h using **A** as catalyst (step 1) and stirred for 20h at 50 °C (step 2), the title product was isolated as a yellow solid (95% yield, 96% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.72 (s, 1H), 7.43-7.51 (m, 1H), 7.18-7.35 (m, 6H), 7.07-7.18 (m, 2H), 4.84 (m, *J* = 6.5 Hz, 1H), 4.25-4.35 (m, 3H), 3.15-3.24 (m, 1H), 2.73-2.91 (m, 2H), 2.65 (s, 3H), 2.24-2.32 (m, 1H), 2.07 (td, *J* = 13 Hz and 5.5 Hz, 1H), 1.13 (d, *J* =6.5 Hz, 1H), 0.77 (d, *J* =6.5 Hz, 1H).
^{13}C NMR ($CDCl_3$, 125M): δ 168.7, 154.8, 147.6, 139.6, 136.2, 134.2, 128.3, 128.1, 126.9, 126.0, 121.8, 119.7, 118.1, 111.0, 109.2, 97.8, 65.8, 49.5, 44.8, 38.5, 36.1, 22.5, 22.3, 21.5, 17.7.
$[\alpha]^{25}_D$ -85.9 (*c* 0.19, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{26}H_{29}N_2O_2)^+$ 401.2219, found 401.2223.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 10.21 min (minor enantiomer), t_R = 21.80 min (major enantiomer).



3o

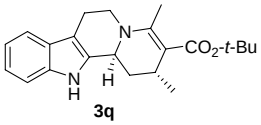
(2*S*,12b*S*)-*t*-butyl
4-methyl-2-phenyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3o**): The reaction was carried out at -15 °C for 72h using **A** as catalyst (step 1) and stirred for 24h at 50 °C (step 2), the title product was isolated as a yellow solid (73% yield, 96% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.62 (s, 1H), 7.45-7.50 (m, 1H), 7.18-7.35 (m, 6H), 7.07-7.18 (m, 2H), 4.24-4.34 (m, 1H), 4.18-4.23 (m, 1H), 3.15-3.24 (m, 1H), 2.72-2.92 (m, 2H), 2.61 (s, 3H), 2.21-2.28 (m, 1H), 2.08 (td, *J* = 12.5 Hz and 5.5 Hz, 1H), 1.19 (s, 9H).

^{13}C NMR ($CDCl_3$, 125M): δ 168.8, 154.3, 147.9, 136.2, 134.2, 128.3, 128.2, 126.9, 126.0, 121.9, 119.8, 118.2, 110.9, 109.3, 99.5, 49.4, 44.7, 39.1, 36.3, 28.3, 22.5, 17.8.
$[\alpha]^{25}_D$ 78.4 (*c* 0.60, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{27}H_{31}N_2O_2)^+$ 415.2385, found 415.2380.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 8.91 min (minor enantiomer), t_R = 12.87 min (major enantiomer).



3p

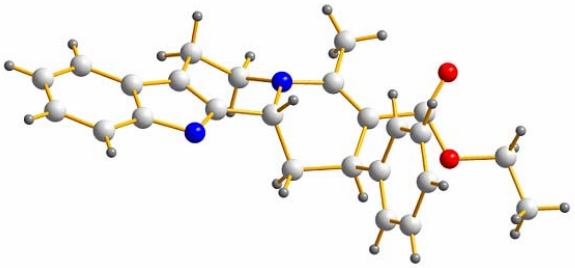
(2*S*,12b*S*)-ethyl 2-phenyl-4-propyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate (**3p**): The reaction was carried out at -15 °C for 24h using **A** as catalyst (step 1) and stirred for 20h at 50 °C (step 2), the title product was isolated as a yellow solid (92% yield, 94% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.72 (s, 1H), 7.44-7.52 (m, 2H), 7.30-7.36 (m, 2H), 7.20-7.28 (m, 3H), 7.08-7.18 (m, 2H), 4.23-4.35 (m, 3H), 4.18-4.23 (m, 1H), 3.96 (q, *J* = 7 Hz, 2H), 3.36-3.47 (m, 1H),3.18-3.27 (m, 1H), 2.71-2.95 (m, 3H), 2.26-2.33 (m, 1H), 2.07 (td, *J* = 13 Hz and 5 Hz, 1H), 1.83-1.95 (m, 1H), 1.62-1.74 (m, 1H), 1.14 (t, *J* = 7 Hz , 3H), 1.02 (t, *J* = 7 Hz , 3H).
^{13}C NMR ($CDCl_3$, 125M): δ 168.6, 159.1, 147.3, 136.2, 134.2, 128.3, 127.9, 126.9, 126.1, 121.9, 119.7, 118.1, 111.0, 109.2, 95.8, 58.9, 49.6, 44.6, 38.1, 36.2, 31.8, 22.8, 14.4.
$[\alpha]^{25}_D$ 62.3 (*c* 0.80, $CHCl_3$).
HRMS (ESI) calcd. for $(C_{27}H_{31}N_2O_2)^+$415.2374, found 415.2380.
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R = 9.76 min (minor enantiomer), t_R = 61.16 min (major enantiomer).

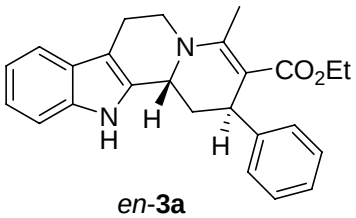


3q

(2*S*,12b*S*)-*t*-butyl 2,4-dimethyl-1,2,6,7,12,12b-hexahydroindolo[2,3*α*]quinolizine-3-carboxylate[10a] (**3q**): The reaction was carried out at -15 °C for 24h using **A** as catalyst (step 1) and stirred for 20h at 50 °C (step 2), the title product was isolated as a yellow solid (51% yield, 80% ee):
^{1}H NMR ($CDCl_3$, 500M): δ 7.82 (s, 1H), 7.49 (d, *J* = 8 Hz, 1H), 7.34 (d, *J* = 8 Hz, 1H), 7.08-7.20 (m, 2H), 4.44-4.51 (m, 1H), 4.15-4.23 (m, 1H), 3.11-3.21 (m, 1H), 2.96-3.06 (m, 1H), 2.72-2.89 (m, 2H), 2.43 (s, 3H), 2.00-2.08 (m, 1H), 1.81 (td, *J* = 12.5 Hz and 5.5 Hz, 1H), 1.50 (s, 9H).
$[\alpha]^{25}_D$ -11.4 (*c* 0.95, $CHCl_3$).
HPLC (Daicel Chiralpak ADH, Hexane : Isopropanol = 4 : 1, Flow rate = 0.7 mL/min, λ = 220 nm): t_R =10.09 min (minor enantiomer), t_R = 12.04 min (major enantiomer).

Crystal structure for *en*-**3a**:

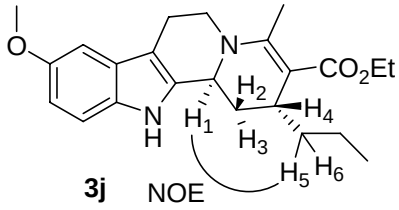




en-**3a**

Identification code	gd
Empirical formula	C25 H25 N2 O2
Formula weight	385.47
Temperature	296(2) K
Wavelength	0.71073 A
Crystal system, space group	Monoclinic, P2(1)
Unit cell dimensions	a = 8.356(3) A alpha = 90 deg.
	b = 7.930(2) A beta = 103.369(4) deg.
	c = 15.722(5) A gamma = 90 deg.
Volume	1013.6(5) A^3
Z, Calculated density	2, 1.263 Mg/m^3
Absorption coefficient	0.080 mm^-1
F(000)	410
Crystal size	0.27 x 0.22 x 0.15 mm
Theta range for data collection	2.51 to 25.25 deg.
Limiting indices	-8<=h<=10, -9<=k<=9, -18<=l<=12
Reflections collected / unique	5268 / 3536 [R(int) = 0.0305]
Completeness to theta = 25.25	99.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9880 and 0.9786
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	3536 / 1 / 265
Goodness-of-fit on F^2	1.046
Final R indices [I>2sigma(I)]	R1 = 0.0480, wR2 = 0.1095
R indices (all data)	R1 = 0.0665, wR2 = 0.1222
Absolute structure parameter	1.2(18)
Extinction coefficient	0.009(3)
Largest diff. peak and hole	0.271 and -0.164 e.A^-3

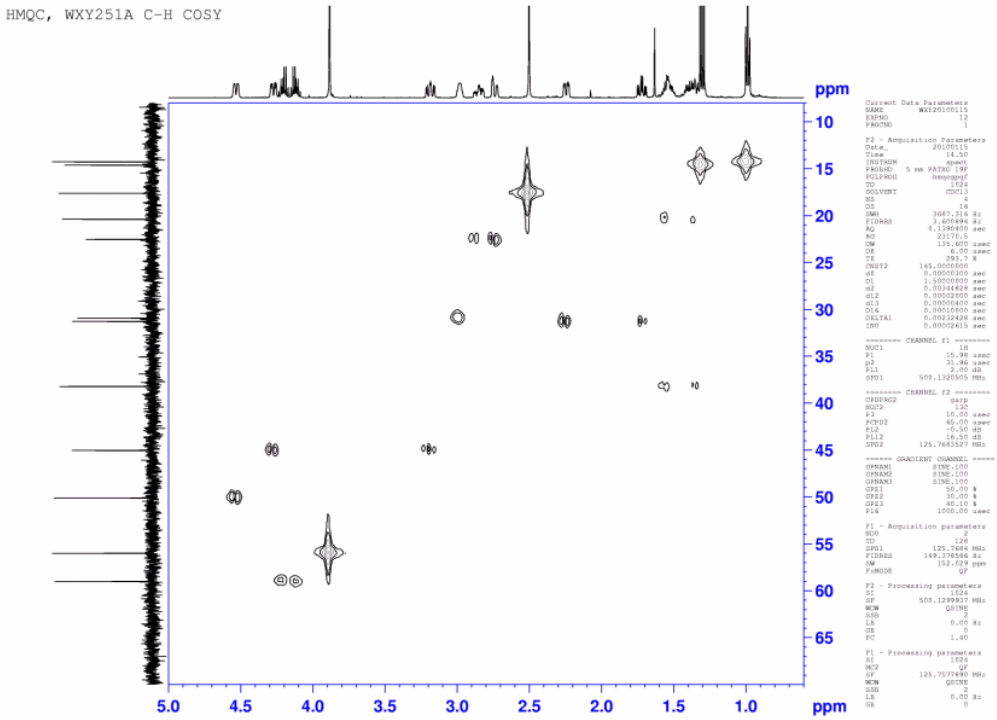
Determination of relative configuration of **3j**:

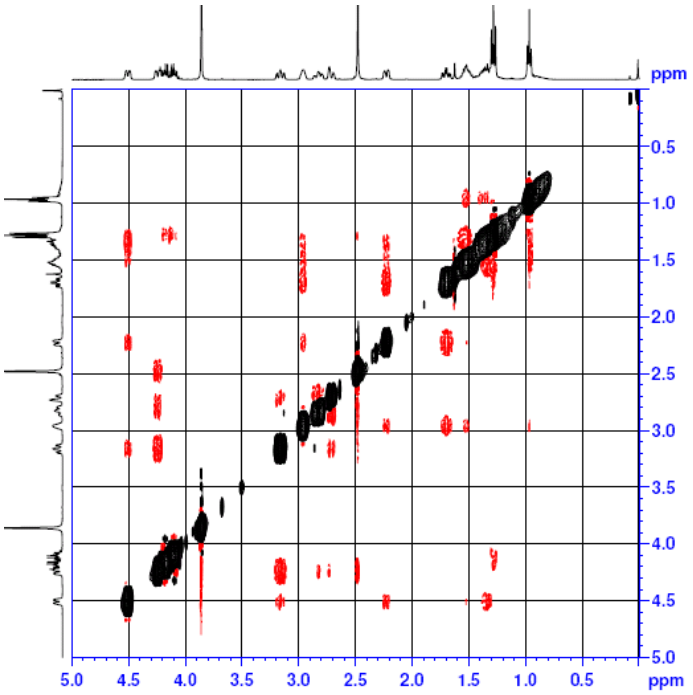


3j NOE

$^3J_{H1H2}$ = 13 HZ
$^2J_{H2H3}$ = 13 HZ
$^3J_{H2H4}$ = 5 HZ

H1 : 4.53 ppm
H2 : 1.72 ppm
H3 : 2.24 ppm
H4 : 2.96 ppm
H5,6 : 1.4-1.6 ppm



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EXPNO                   30
PROCNO                   1

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D1                2.00000000 sec
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IN0               0.00029750 sec

======== CHANNEL f1 ========
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F1 - Acquisition parameters
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F1 - Processing parameters
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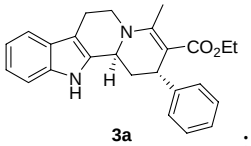
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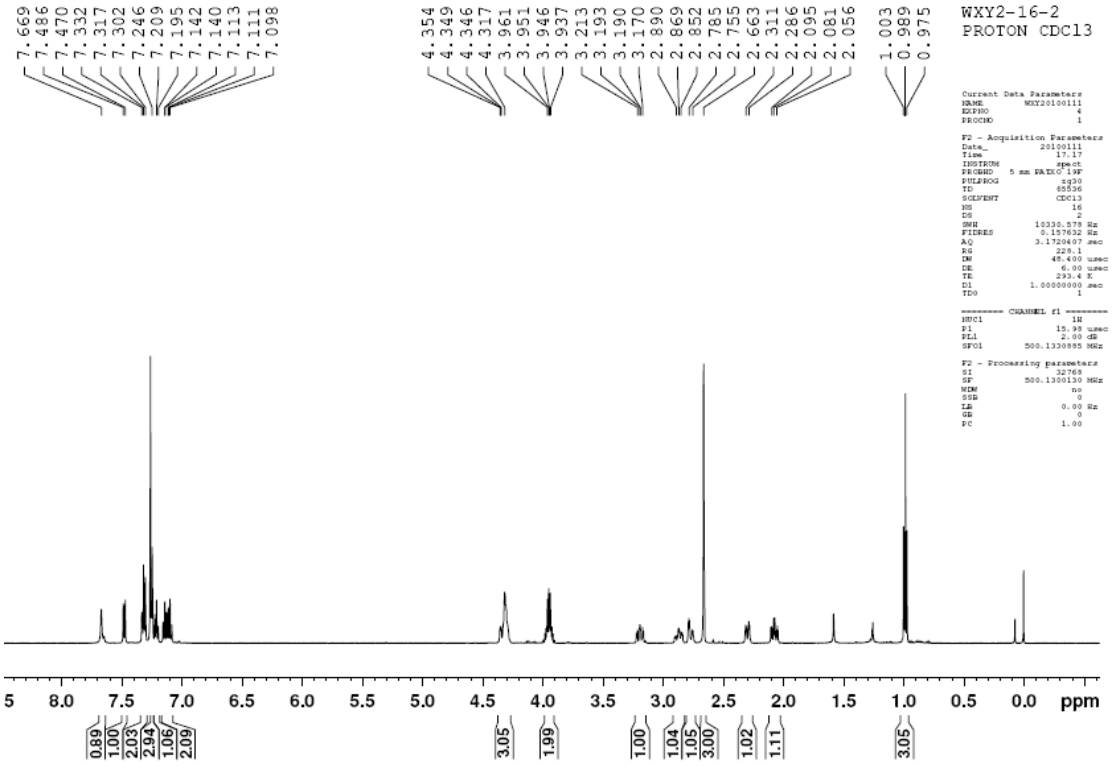
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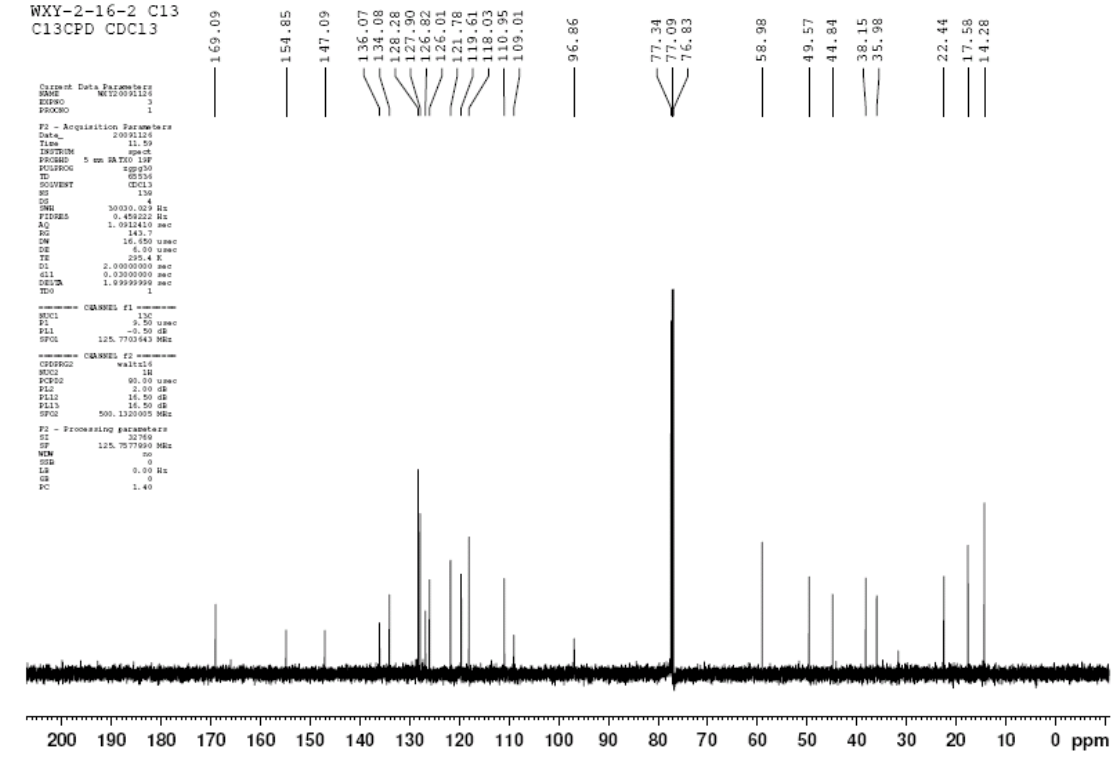
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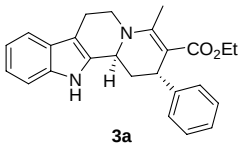
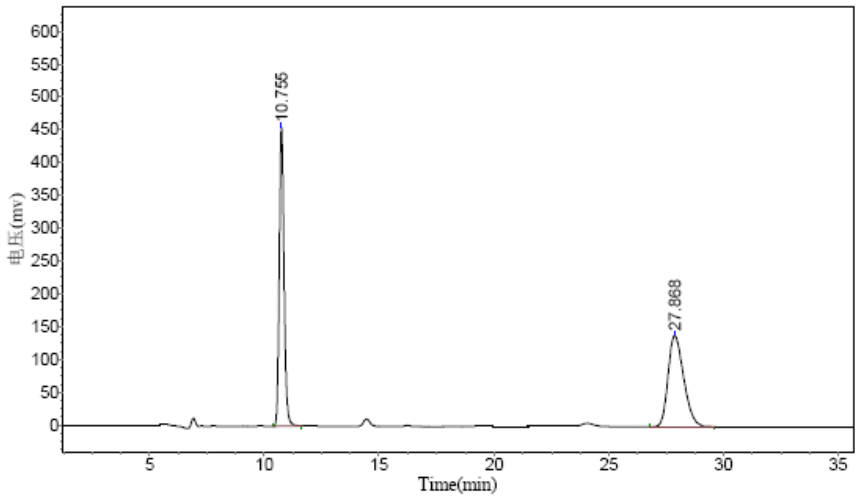


3a



Results

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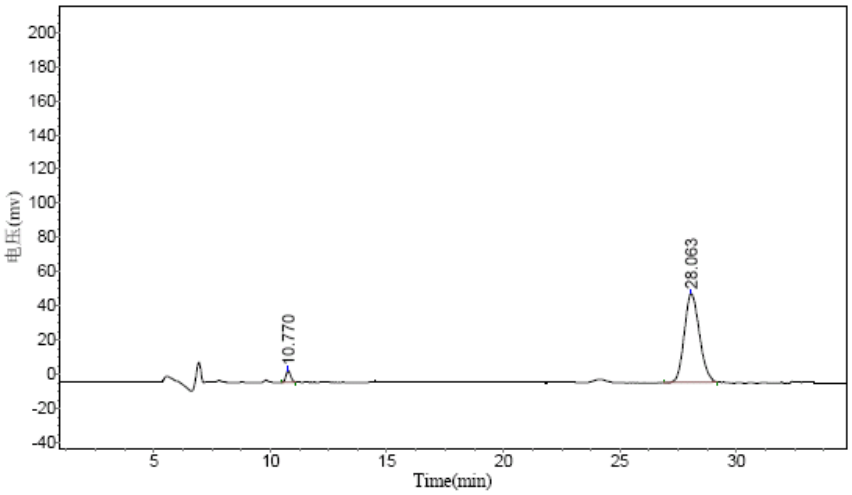

Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
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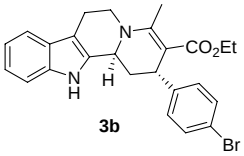
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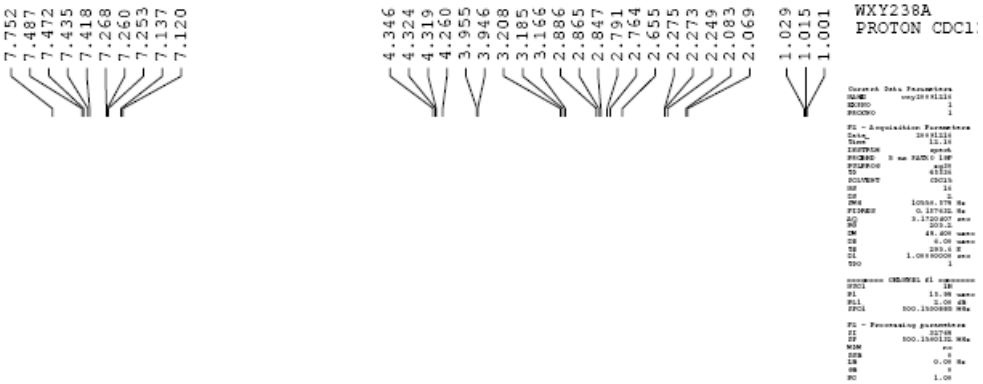
3b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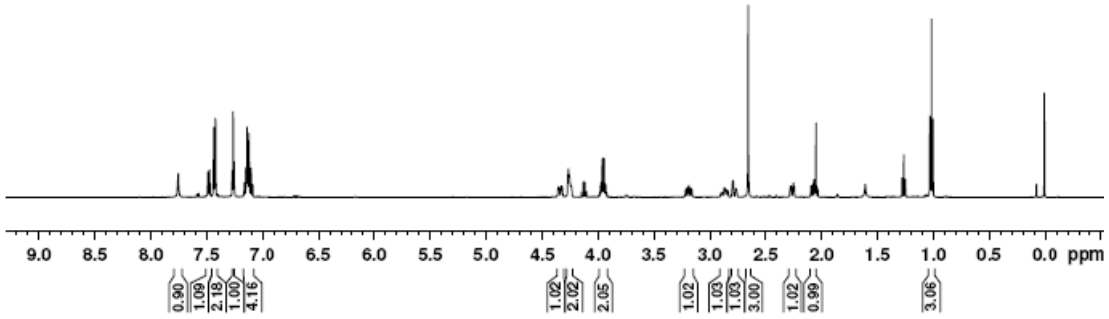
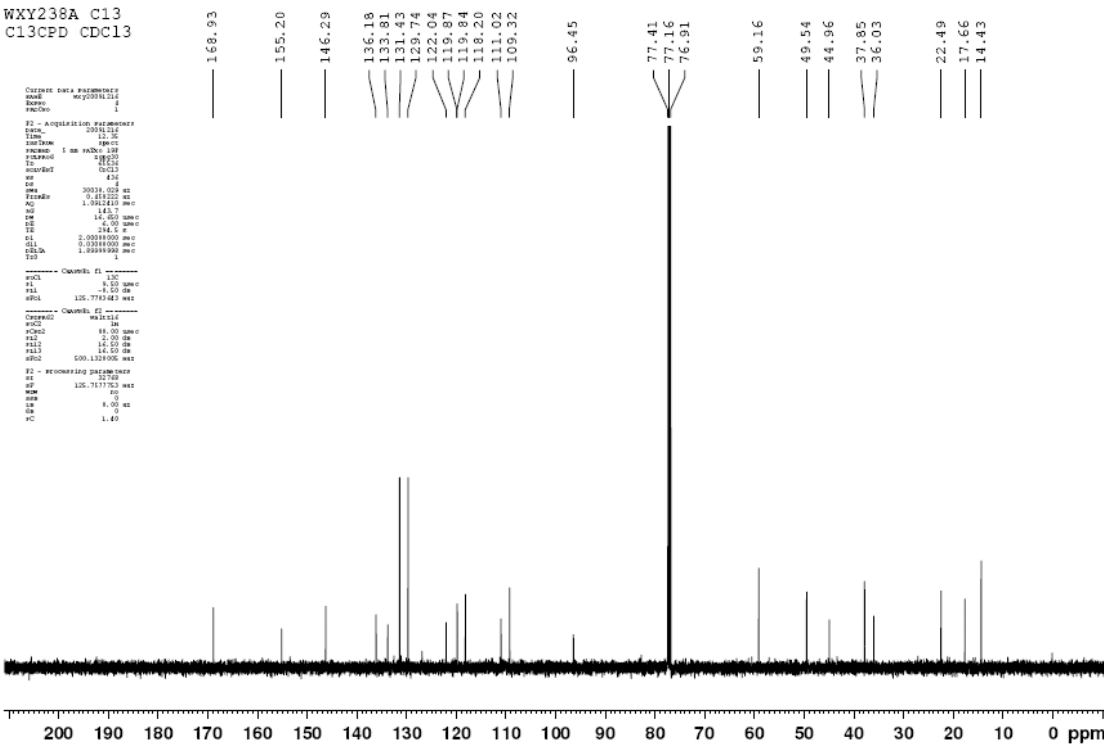
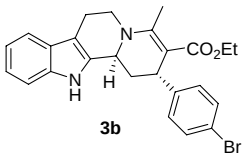
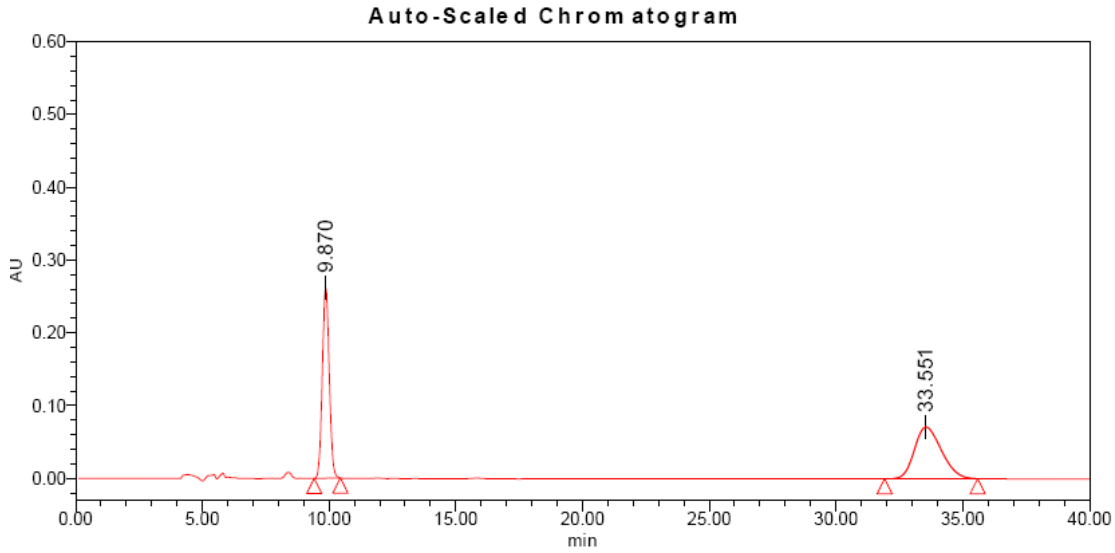
7.120
4.346
4.324
4.319
4.260
3.955
3.946
3.208
3.185
3.166
2.886
2.865
2.847
2.791
2.764
2.655
2.275
2.273
2.249
2.083
2.069
1.029
1.015
1.001
WXY238A
PROTON CDCl3


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
0.90
1.09
2.18
1.00
4.16
1.02
2.02
2.05
1.02
1.03
1.03
3.00
1.02
0.99
3.06


WXY238A C13
C13CPD CDCl3
168.93
155.20
146.29
136.18
133.81
131.43
129.74
122.04
119.87
119.84
118.20
111.02
109.32
96.45
77.41
77.16
76.91
59.16
49.54
44.96
37.85
36.03
22.49
17.66
14.43
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

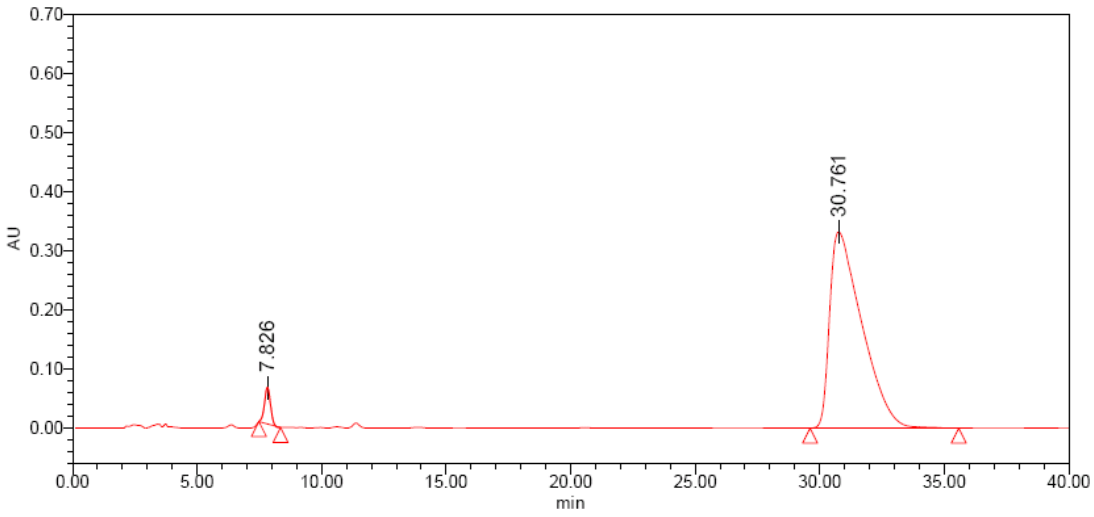


3b



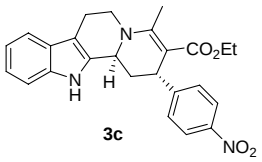
Peak Results

	Name	RT	Area	Height	% Height	% Area
1		9.870	4962590	261493	78.61	48.52
2		33.551	5264667	71137	21.39	51.48

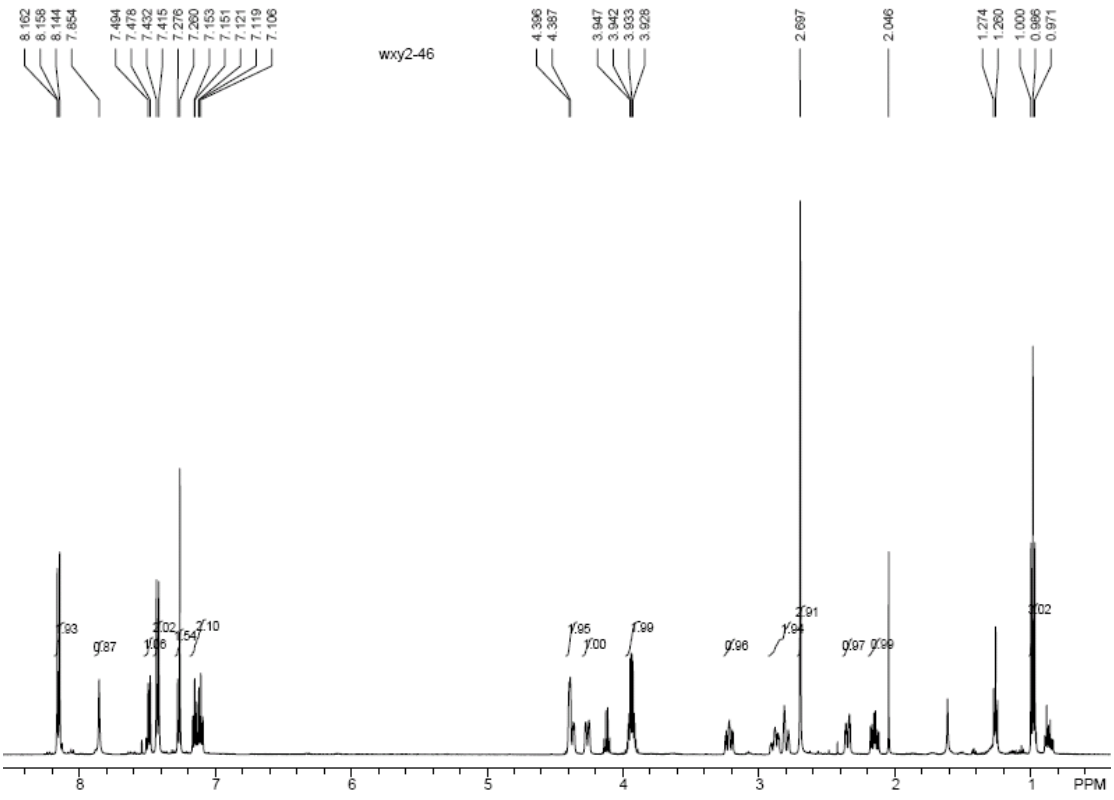


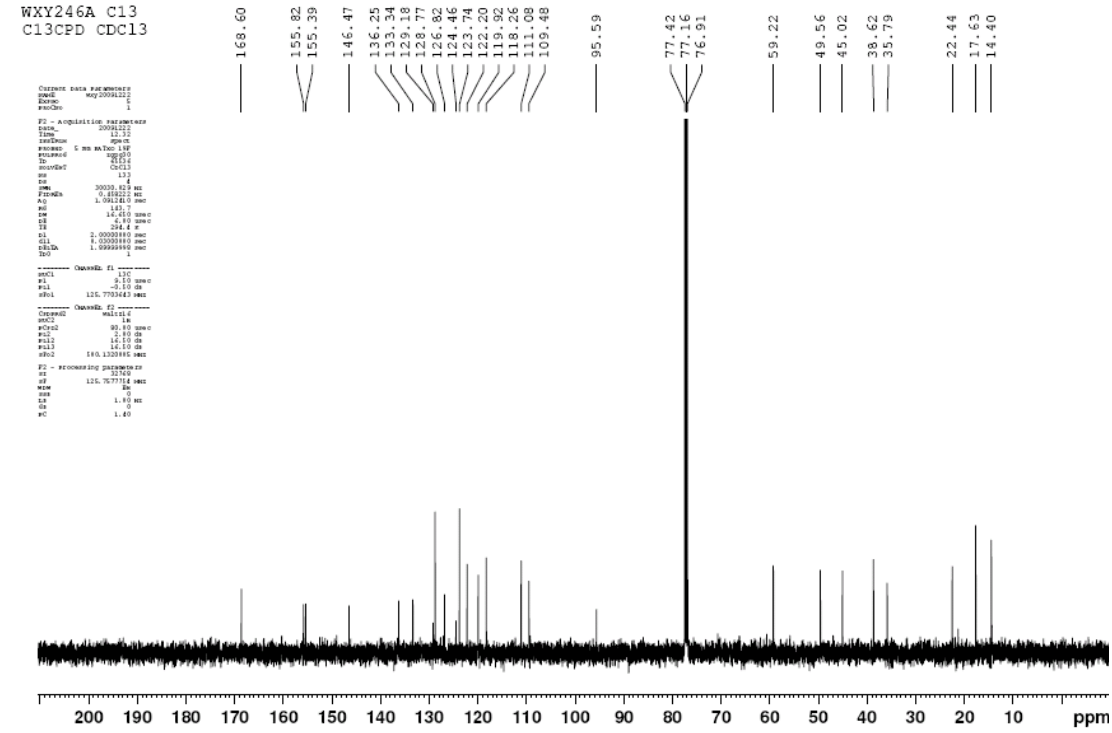
Peak Results

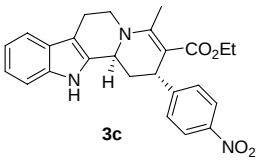
	Name	RT	Area	Height	% Height	% Area
1		7.826	1044936	62048	15.73	3.39
2		30.761	29814616	332407	84.27	96.61


N
H
N
H
CO2Et
NO2

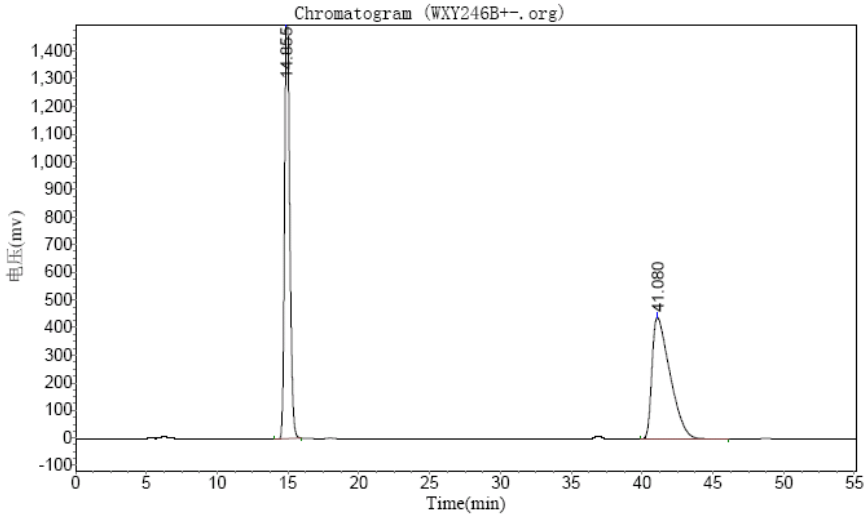
3c


8.162
8.158
8.144
7.854
7.494
7.478
7.432
7.415
7.276
7.260
7.153
7.151
7.121
7.119
7.106
wxy2-46
4.396
4.387
3.947
3.942
3.933
3.928
2.697
2.046
1.274
1.260
1.000
0.986
0.971
PPM


WXY246A C13
C13CPD CDCl3
168.60
155.82
155.39
146.47
136.25
133.34
129.18
128.77
126.82
124.46
123.74
122.20
119.92
118.26
111.08
109.48
95.59
77.42
77.16
76.91
59.22
49.56
45.02
38.62
35.79
22.44
17.63
14.40
ppm

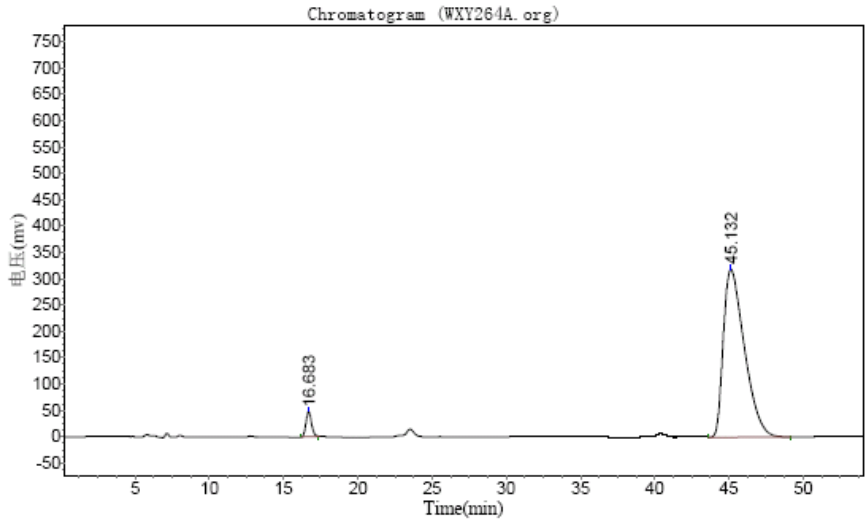


3c



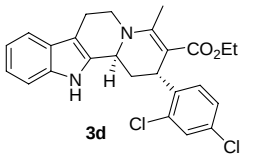
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.855	1491209.750	38885500.000	49.9417
2		41.080	440323.031	38976280.000	50.0583
Total			1931532.781	77861780.000	100.0000

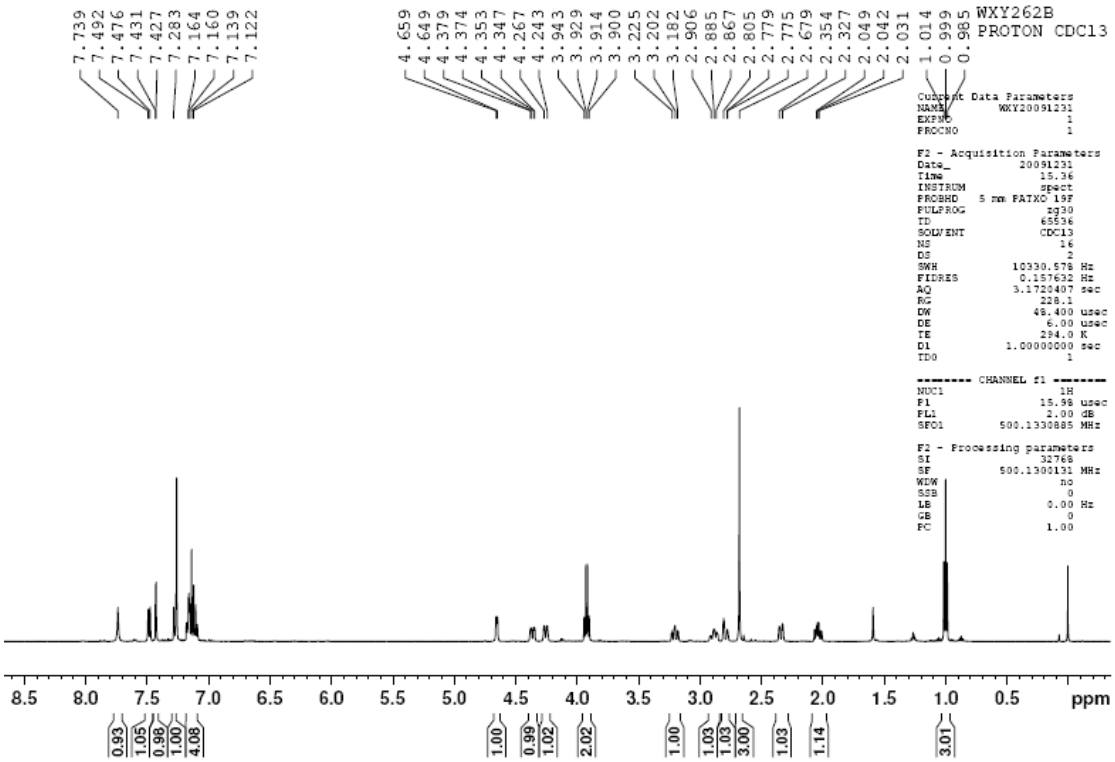


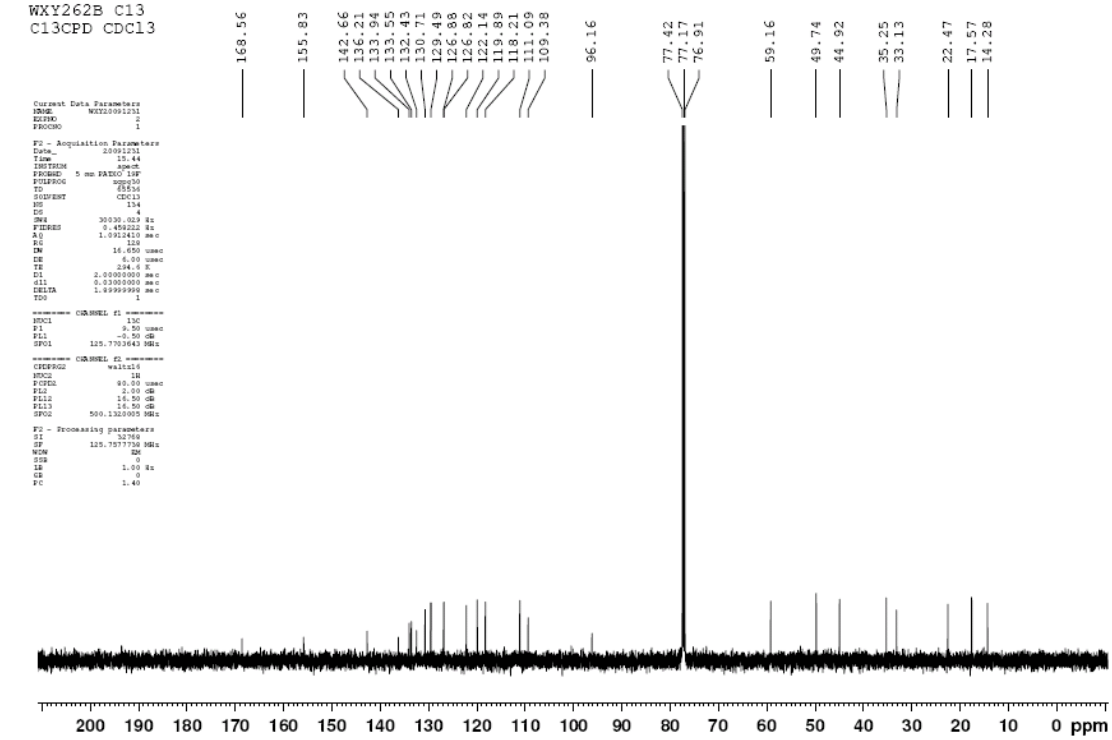
Results

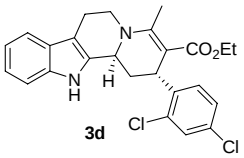
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.683	47871.648	1234859.375	3.7885
2		45.132	318240.594	31359720.000	96.2115
Total			366112.242	32594579.375	100.0000


N
CO2Et
N
H
H
Cl
Cl

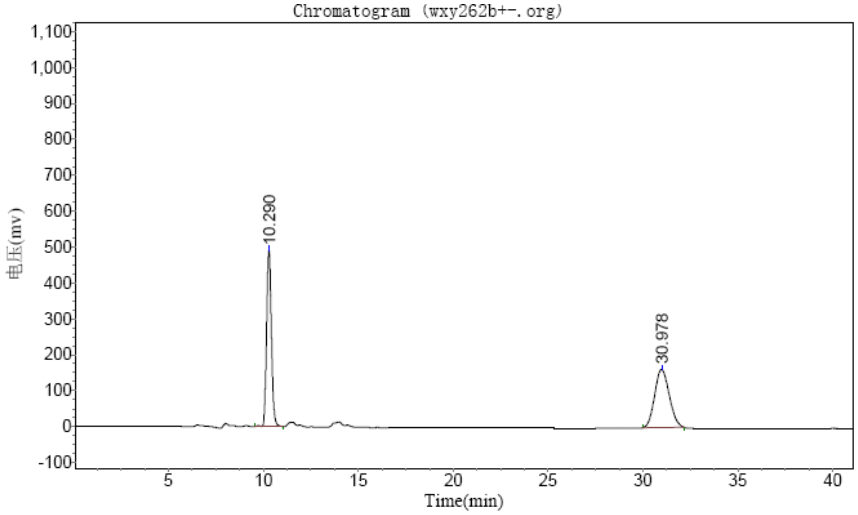
3d


WXY262B
PROTON CDC13
7.739
7.492
7.476
7.431
7.427
7.283
7.164
7.160
7.139
7.122
4.659
4.649
4.379
4.374
4.353
4.347
4.267
4.243
3.943
3.929
3.914
3.900
3.225
3.202
3.182
2.906
2.885
2.867
2.805
2.779
2.775
2.679
2.354
2.327
2.049
2.042
2.031
1.014
0.999
0.985
ppm
0.93
1.05
0.98
1.00
4.08
1.00
0.99
1.02
2.02
1.00
1.03
1.03
3.00
1.03
1.14
3.01


WXY262B C13
C13CPD CDCl3
168.56
155.83
142.66
136.21
133.94
133.55
132.43
130.71
129.49
126.88
126.82
122.14
119.89
118.21
111.09
109.38
96.16
77.42
77.17
76.91
59.16
49.74
44.92
35.25
33.13
22.47
17.57
14.28
ppm

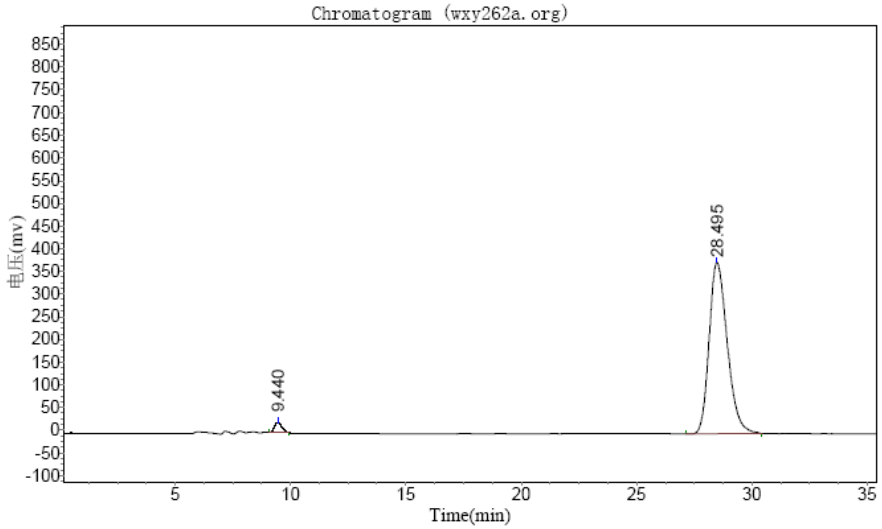


3d



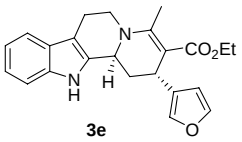
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		10.290	491468.375	8622626.000	49.9362
2		30.978	161966.938	8644662.000	50.0638
Total			653435.313	17267288.000	100.0000

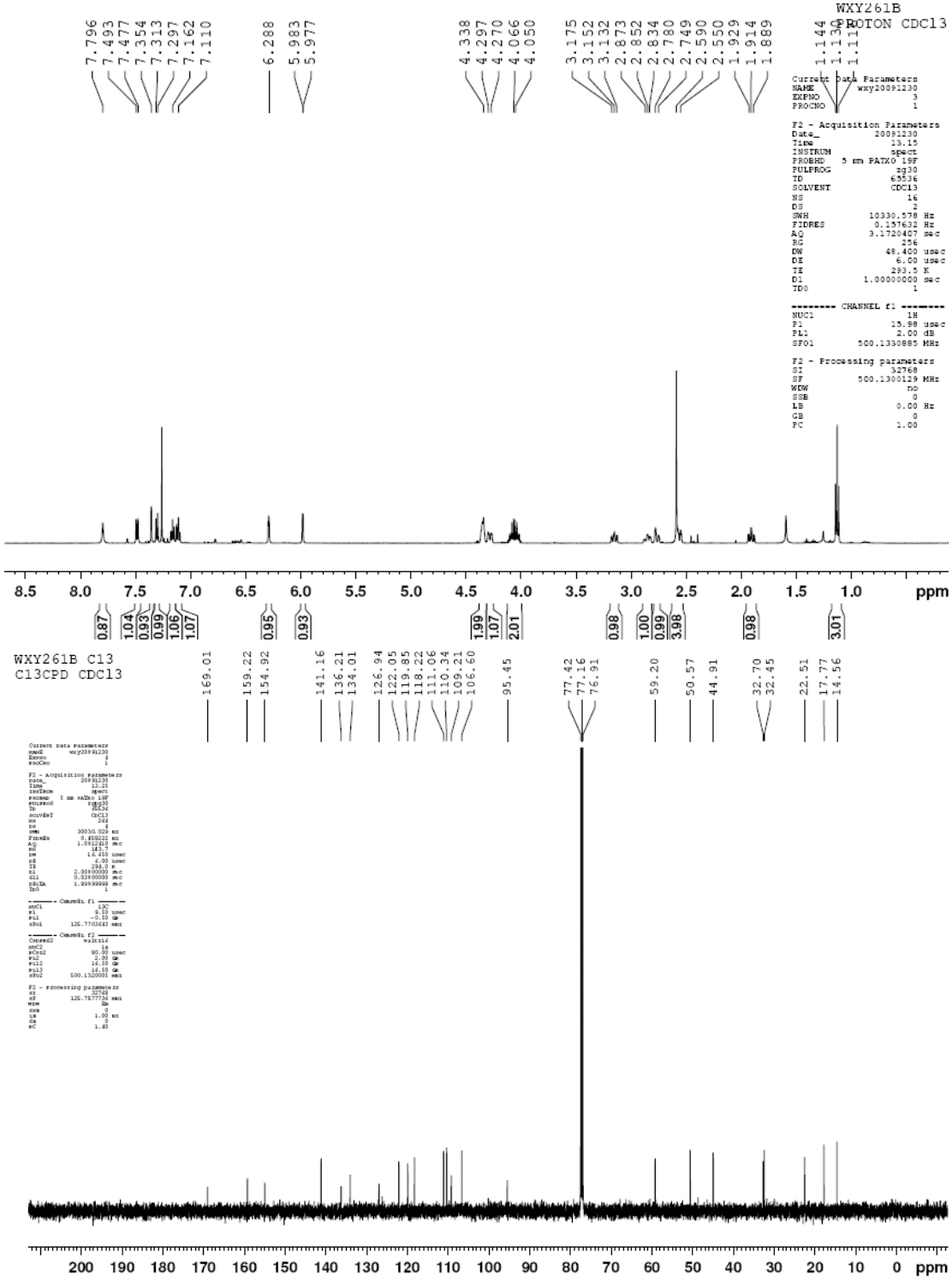


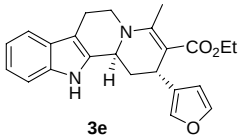
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.440	22591.154	508269.688	2.3991
2		28.495	377960.875	20677882.000	97.6009
Total			400552.029	21186151.688	100.0000

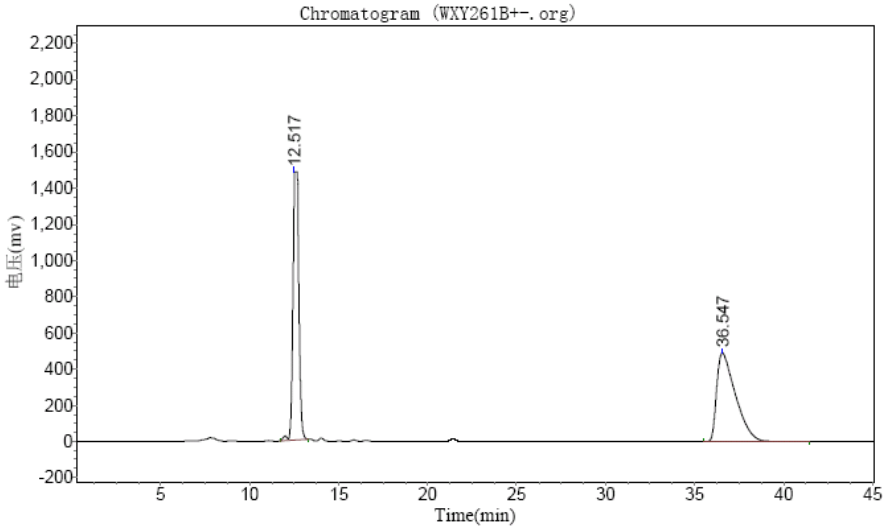

N
H
H
CO2Et
O

3e


WXY261B
PROTON CDC13
7.796
7.493
7.477
7.354
7.313
7.297
7.162
7.110
6.288
5.983
5.977
4.338
4.297
4.270
4.066
4.050
3.175
3.152
3.132
2.873
2.852
2.834
2.780
2.749
2.590
2.550
1.929
1.914
1.889
1.144
1.130
1.115
Current Data Parameters
NAME wxy20091230
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091230
Time 13.15
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 256
DW 48.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
0.87 1.04 0.93 0.99 1.06 1.07 0.95 0.93 1.99 1.07 2.01 0.98 1.00 0.99 3.98 0.98 3.01
WXY261B C13
C13CPD CDC13
169.01
159.22
154.92
141.16
136.21
134.01
126.94
122.05
119.85
118.22
111.06
110.34
109.21
106.60
95.45
77.42
77.16
76.91
59.20
50.57
44.91
32.70
32.45
22.51
17.77
14.56
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

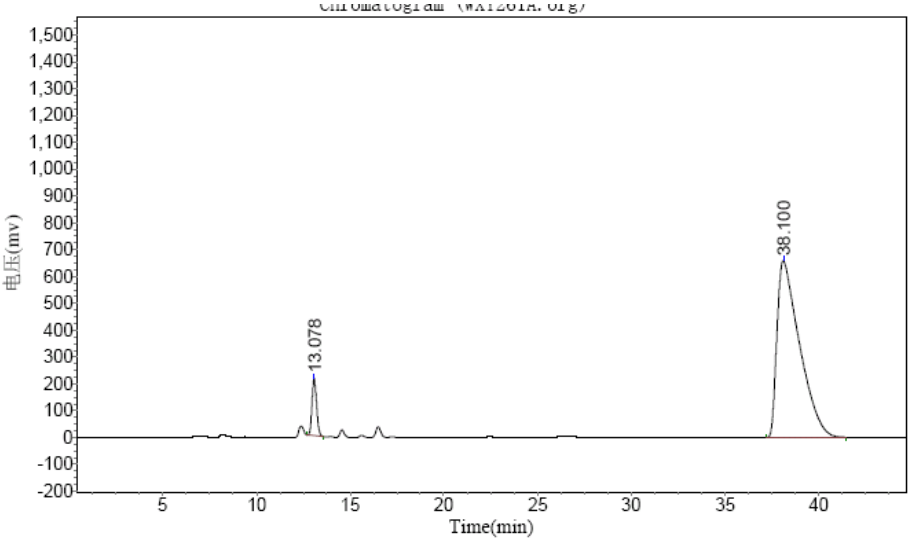


3e



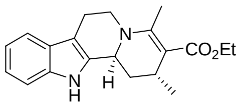
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		12.517	1480795.625	34284956.000	48.8683
2		36.547	490019.094	35872900.000	51.1317
Total			1970814.719	70157856.000	100.0000

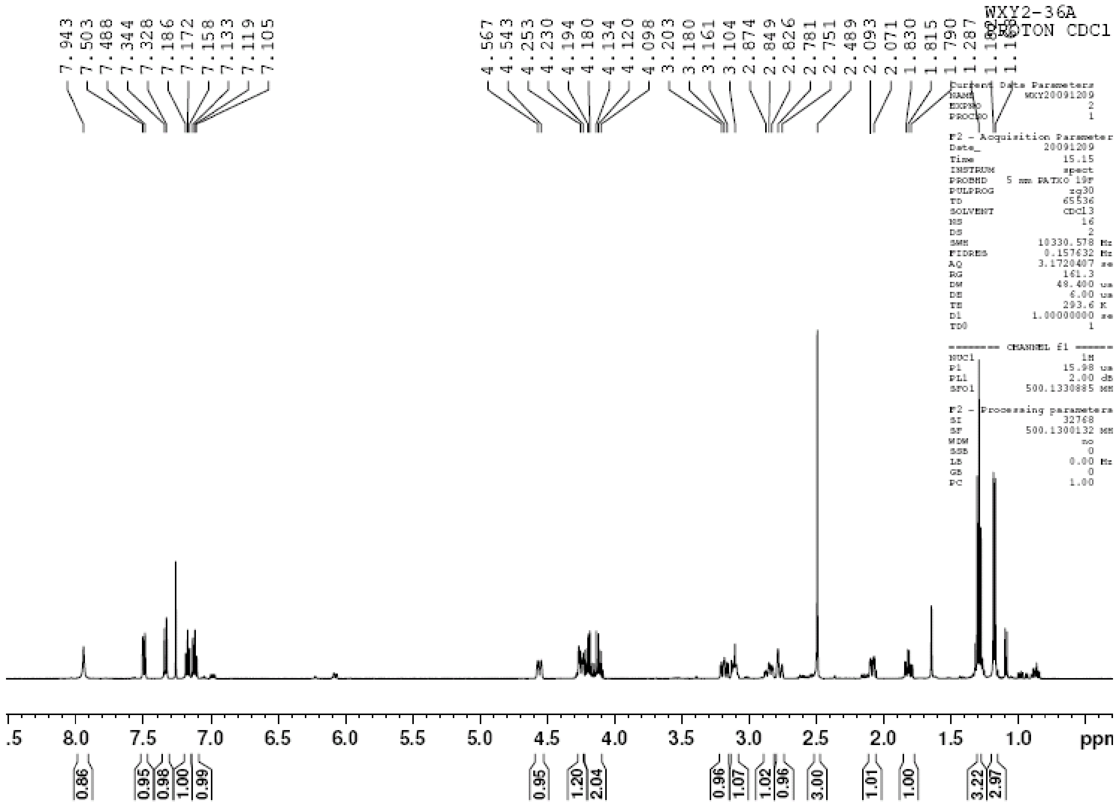


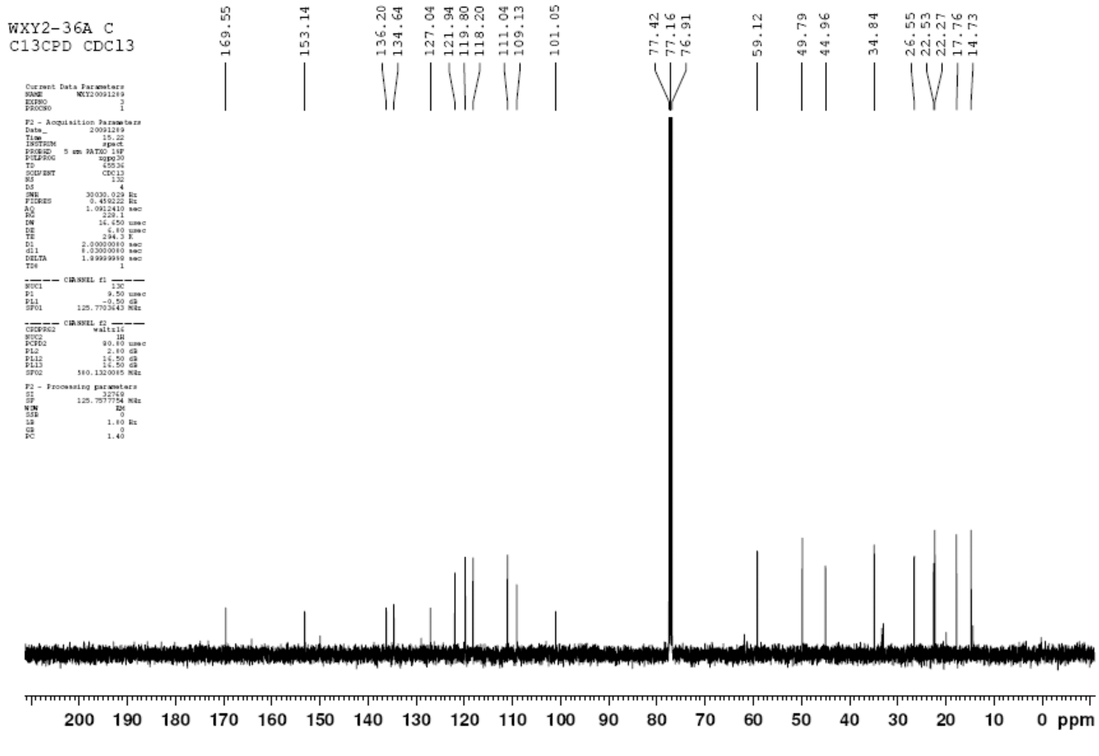
Results

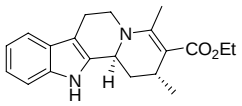
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.078	213329.328	3629038.250	6.1879
2		38.100	658998.313	55018396.000	93.8121
Total			872327.641	58647434.250	100.0000


N
CO2Et
H
N
H

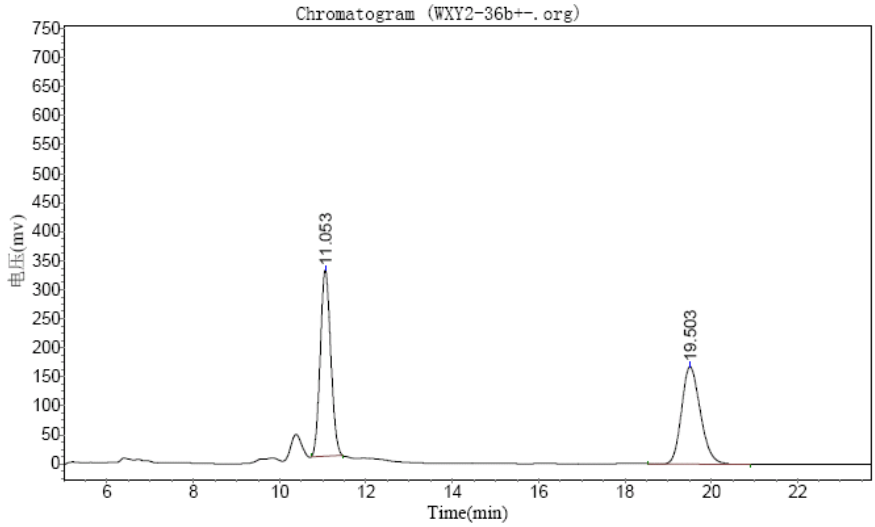
3f


WXY2-36A
PROTON CDCl
7.943
7.503
7.488
7.344
7.328
7.186
7.172
7.158
7.133
7.119
7.105
4.567
4.543
4.253
4.230
4.194
4.180
4.134
4.120
4.098
3.203
3.180
3.161
3.104
2.874
2.849
2.826
2.781
2.751
2.489
2.093
2.071
1.830
1.815
1.790
1.287
Current Data Parameters
F2 - Acquisition Parameter
CHANNEL f1
F2 - Processing parameters
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
ppm
0.86
0.95
0.98
1.00
0.99
0.95
1.20
2.04
0.96
1.07
1.02
0.96
3.00
1.01
1.00
3.22
2.97


WXY2-36A C
C13CPD CDCl3
169.55
153.14
136.20
134.64
127.04
121.94
119.80
118.20
111.04
109.13
101.05
77.42
77.16
76.91
59.12
49.79
44.96
34.84
26.55
22.53
22.27
17.76
14.73
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0 ppm

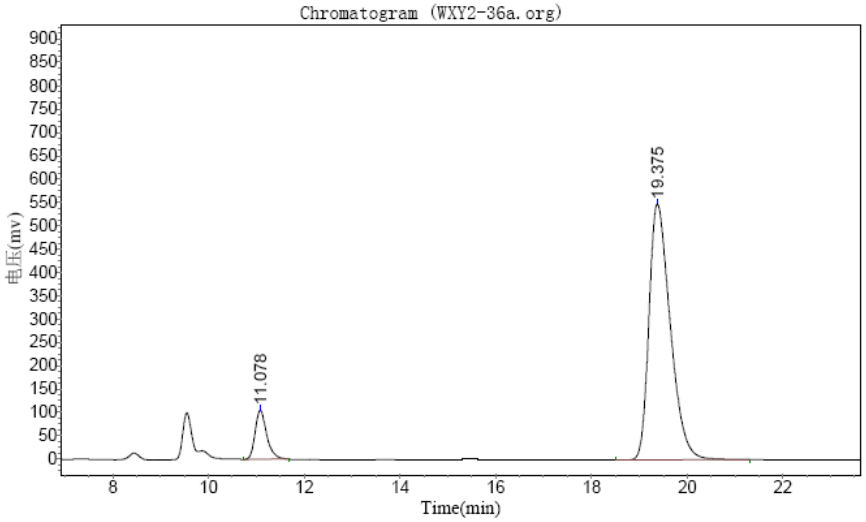


3f



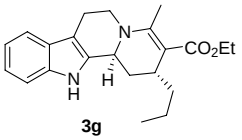
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.053	320045.563	5401052.000	51.6955
2		19.503	167341.031	5046762.000	48.3045
Total			487386.594	10447814.000	100.0000

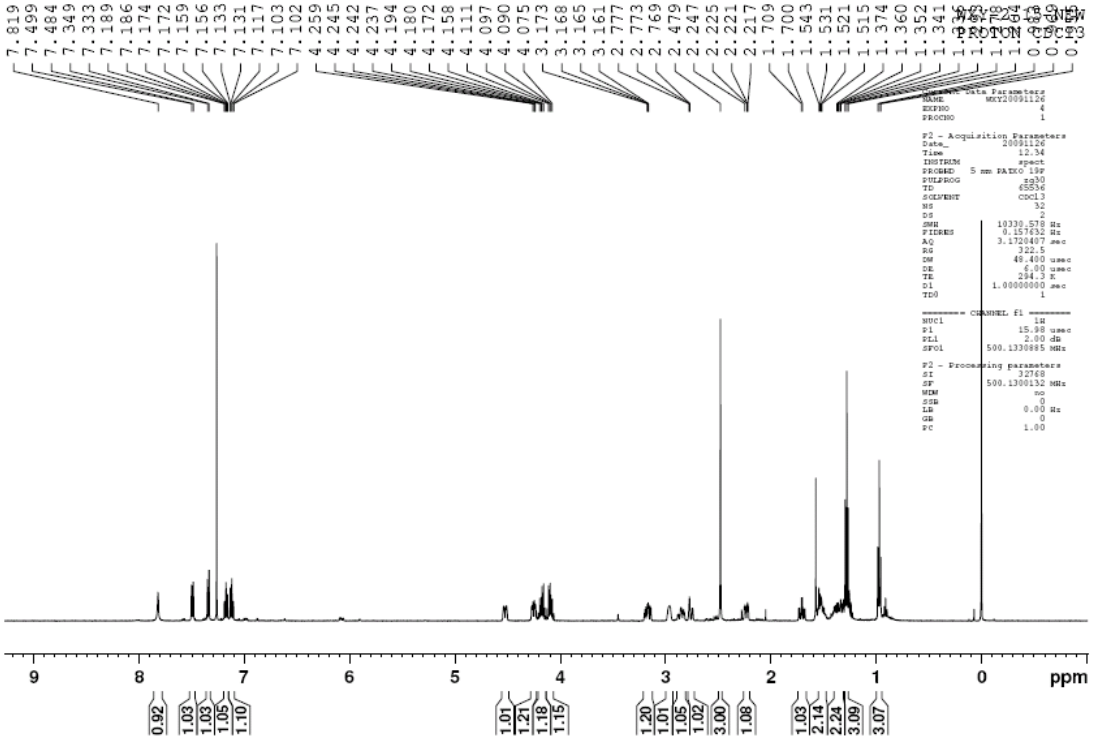


Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.078	104756.438	1758797.375	9.3884
2		19.375	548933.250	16974906.000	90.6116
Total			653689.688	18733703.375	100.0000


N
CO2Et
N
H
H

3g


7.819
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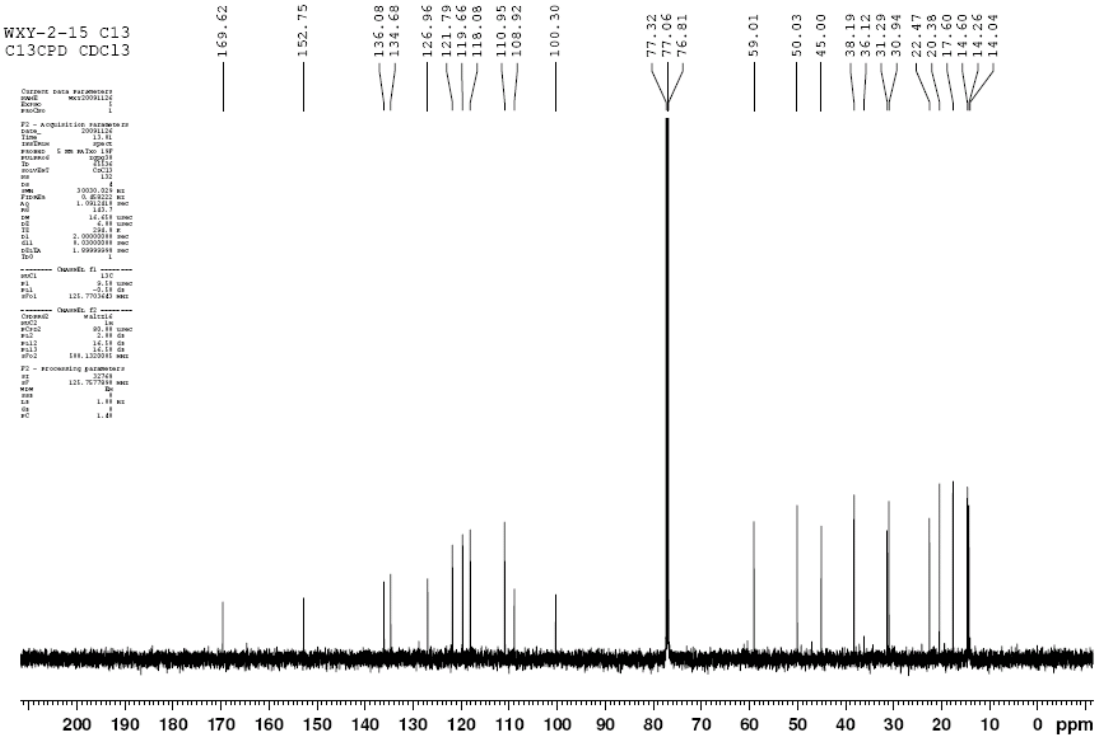
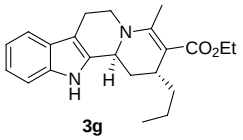
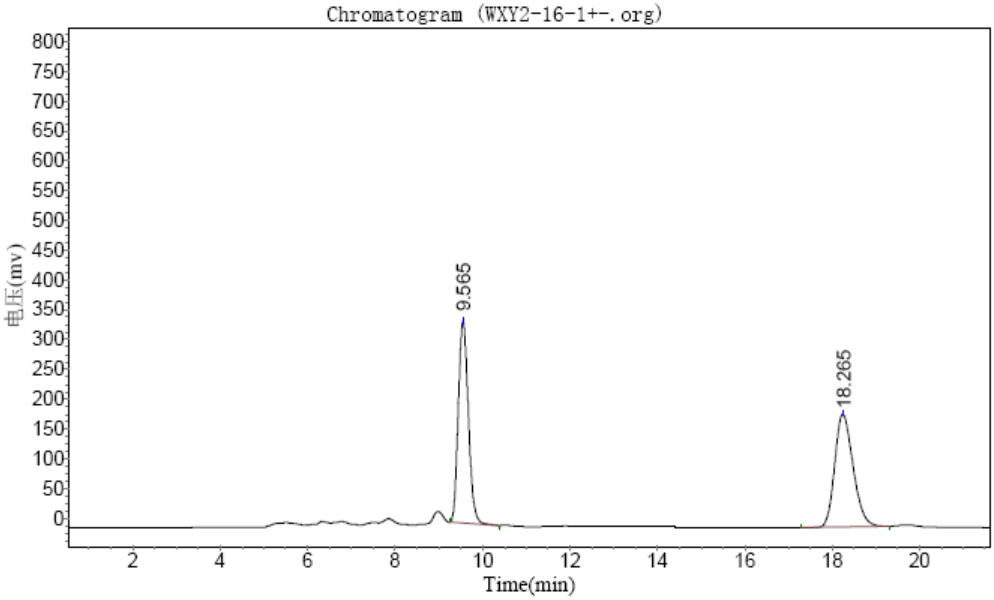
7.484
7.349
7.333
7.189
7.186
7.174
7.172
7.159
7.156
7.133
7.131
7.117
7.103
7.102
4.259
4.245
4.242
4.237
4.194
4.180
4.172
4.158
4.111
4.097
4.090
4.075
3.173
3.168
3.165
3.161
2.777
2.773
2.769
2.479
2.247
2.225
2.221
2.217
1.709
1.700
1.543
1.531
1.521
1.515
1.374
1.360
1.352
1.341
WXY-2-15 CNEW
PROTON CDCl3
9
8
7
6
5
4
3
2
1
0
ppm
0.92
1.03
1.03
1.05
1.10
1.01
1.21
1.18
1.15
1.20
1.01
1.05
1.02
3.00
1.08
1.03
2.14
2.24
3.09
3.07


WXY-2-15 C13
C13CPD CDCl3
169.62
152.75
136.08
134.68
126.96
121.79
119.66
118.08
110.95
108.92
100.30
77.32
77.06
76.81
59.01
50.03
45.00
38.19
36.12
31.29
30.94
22.47
20.38
17.60
14.60
14.26
14.04
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

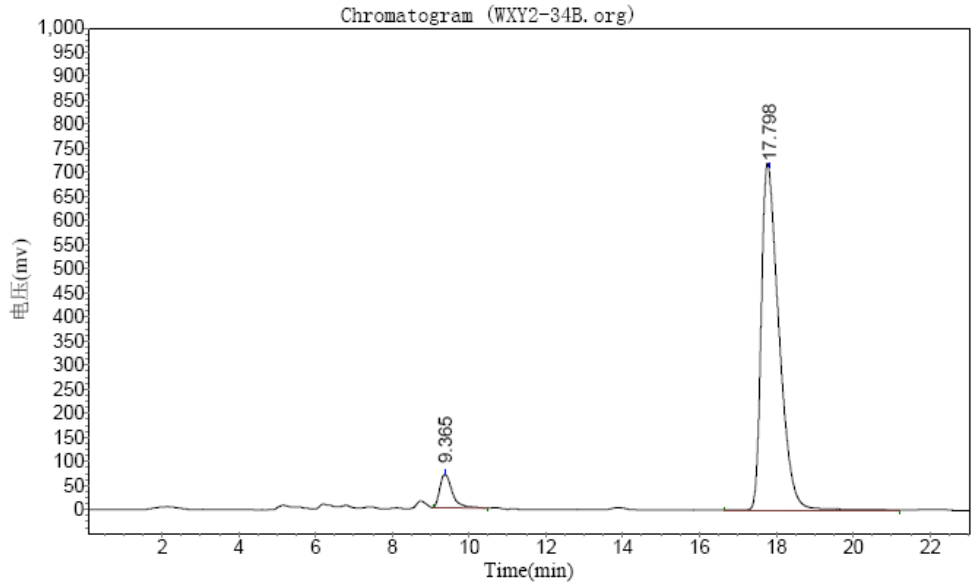


3g



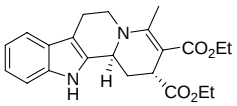
Results

Peak ID	Ret Time	Height	Area	Conc.
	9.565	345203.906	5828632.500	50.7954
	18.265	189737.922	5646092.500	49.2046
		534941.828	11474725.000	100.0000

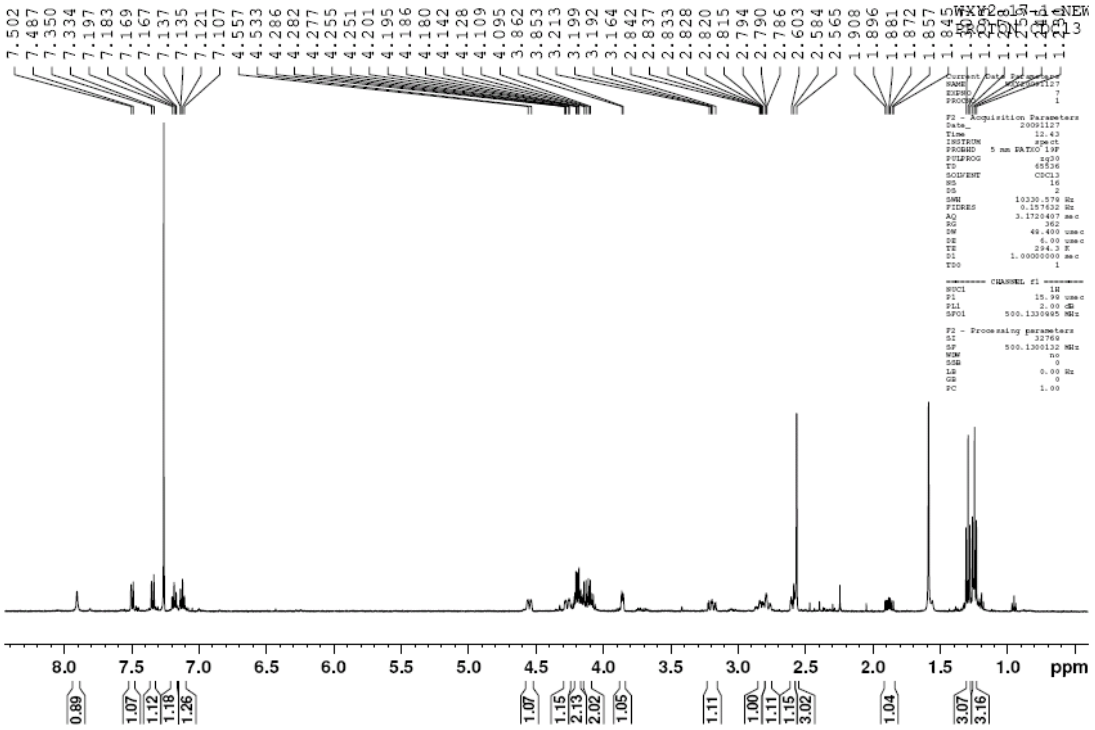


Results

Peak ID	Ret Time	Height	Area	Conc.
	9.365	73574.047	1915280.875	7.7127
	17.798	717168.813	22917378.000	92.2873
		790742.859	24832658.875	100.0000


N
H
H
N
CO2Et
CO2Et

3h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7.183
7.169
7.167
7.137
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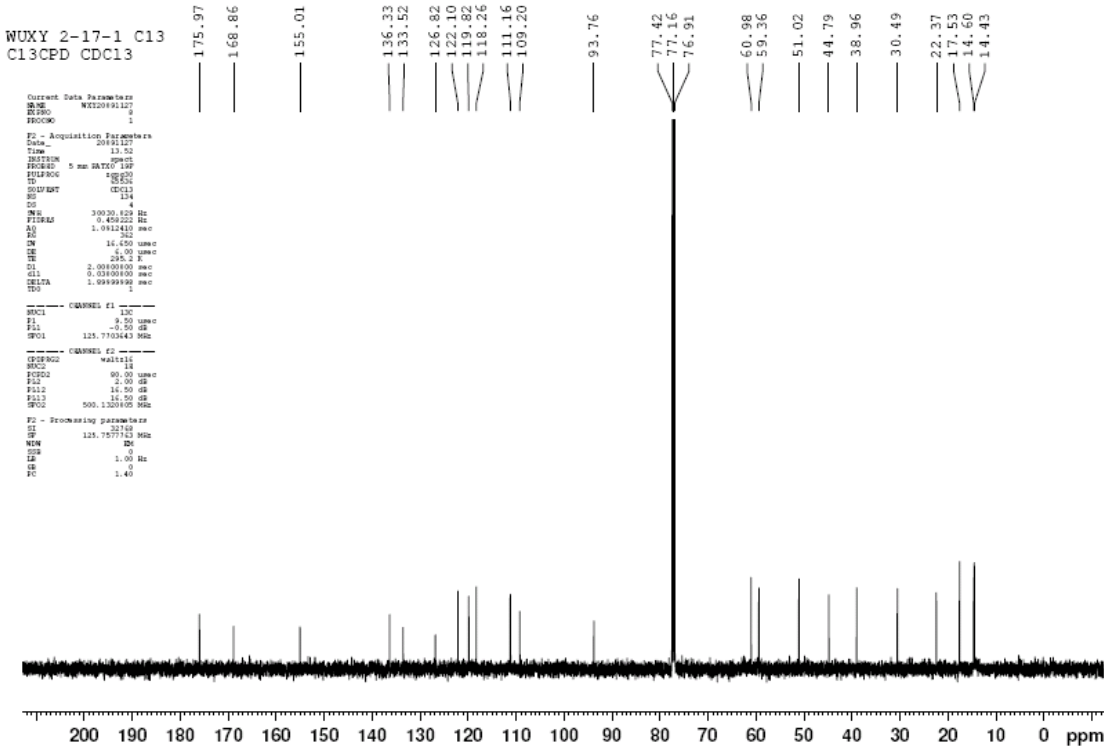
7.121
7.107
4.557
4.533
4.286
4.282
4.277
4.255
4.251
4.201
4.195
4.186
4.180
4.142
4.128
4.109
4.095
3.862
3.853
3.213
3.199
3.192
3.164
2.842
2.837
2.833
2.828
2.820
2.815
2.794
2.790
2.786
2.603
2.584
2.565
1.908
1.896
1.881
1.872
1.857
1.845
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.89
1.07
1.12
1.18
1.26
1.07
1.15
2.13
2.02
1.05
1.11
1.00
1.11
1.15
3.02
1.04
3.07
3.16


WUXY 2-17-1 C13
C13CPD CDCl3
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168.86
155.01
136.33
133.52
126.82
122.10
119.82
118.26
111.16
109.20
93.76
77.42
77.16
76.91
60.98
59.36
51.02
44.79
38.96
30.49
22.37
17.53
14.60
14.43
200
190
180
170
160
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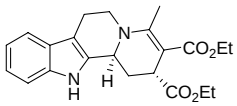
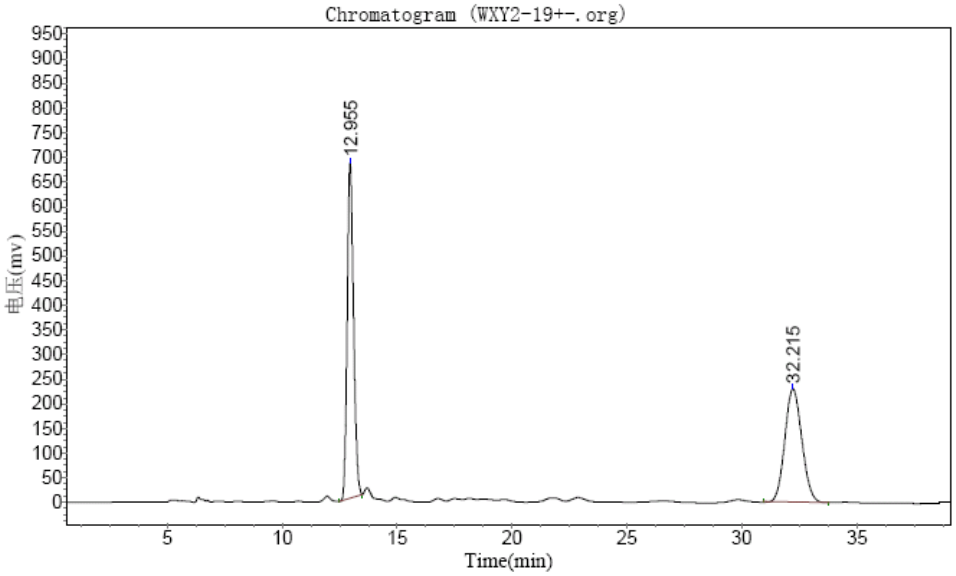
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

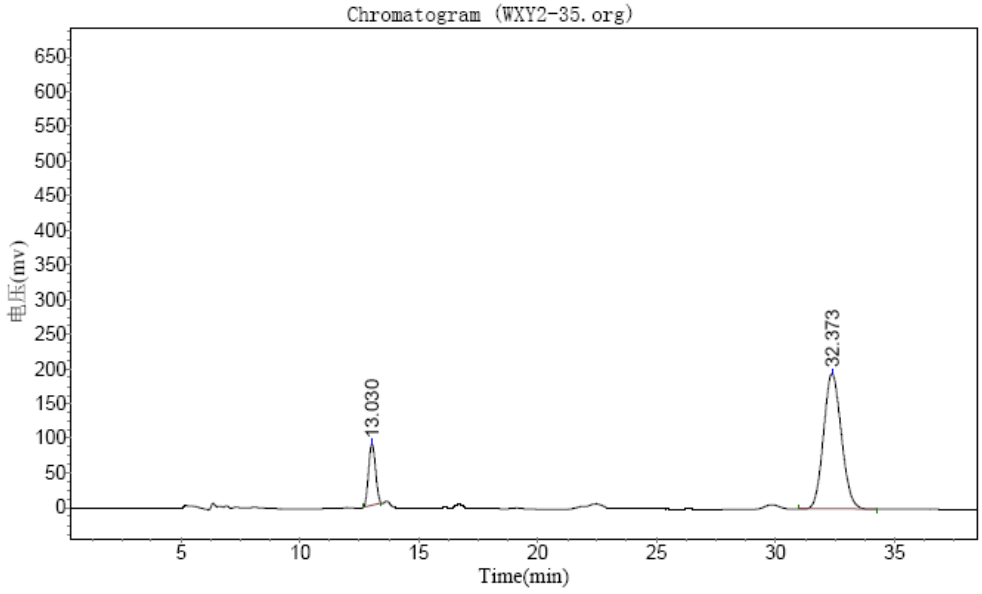


3h



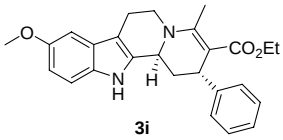
Results

Peak ID	Ret Time	Height	Area	Conc.
	12.955	680230.563	13009961.000	52.4020
	32.215	230055.875	11817257.000	47.5980
		910286.438	24827218.000	100.0000

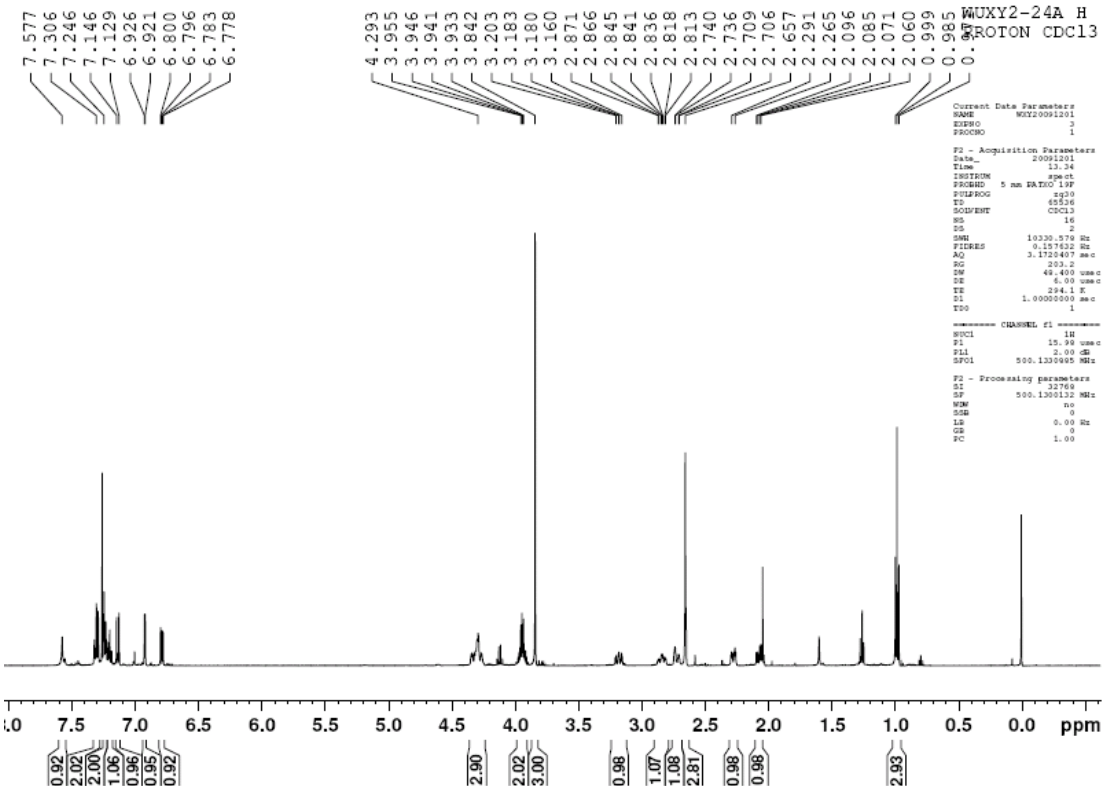


Results

Peak ID	Ret Time	Height	Area	Conc.
	13.030	88502.859	1798164.500	15.0449
	32.373	195223.328	10153804.000	84.9551
		283726.188	11951968.500	100.0000


O
N
CO2Et
N
H
H

3i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PROTON CDC13
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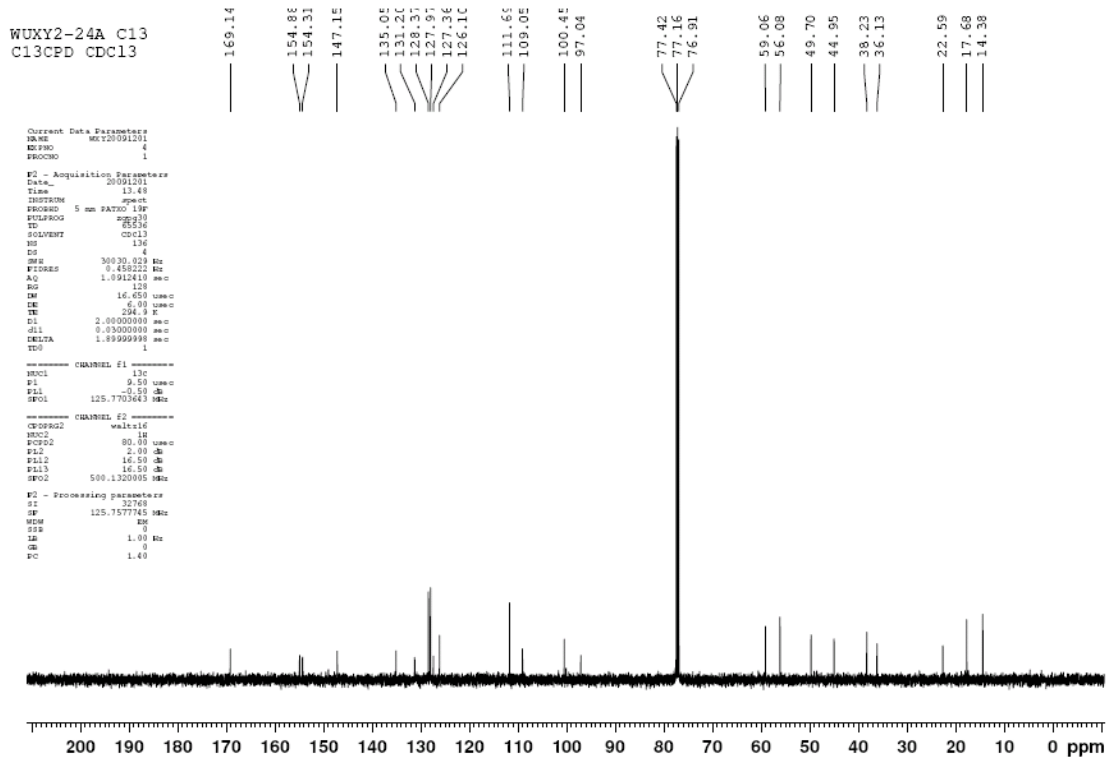
7.246
7.146
7.129
6.926
6.921
6.800
6.796
6.783
6.778
4.293
3.955
3.946
3.941
3.933
3.842
3.203
3.183
3.180
3.160
2.871
2.866
2.845
2.841
2.836
2.818
2.813
2.740
2.736
2.709
2.706
2.657
2.291
2.265
2.096
2.085
2.071
2.060
0.999
0.985
Current Data Parameters
NAME WXY20091201
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091201
Time 13.34
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.579 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203.2
DW 49.400 usec
DE 6.00 usec
TE 294.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.99 usec
PL1 2.00 dB
SFO1 500.1330995 MHz
F2 - Processing parameters
SI 32768
SF 500.1300132 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
ppm
0.92
2.02
2.00
1.06
0.96
0.95
0.92
2.90
2.02
3.00
0.98
1.07
1.08
2.81
0.98
0.98
2.93


WUXY2-24A C13
C13CPD CDCl3
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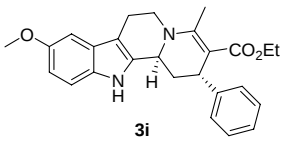
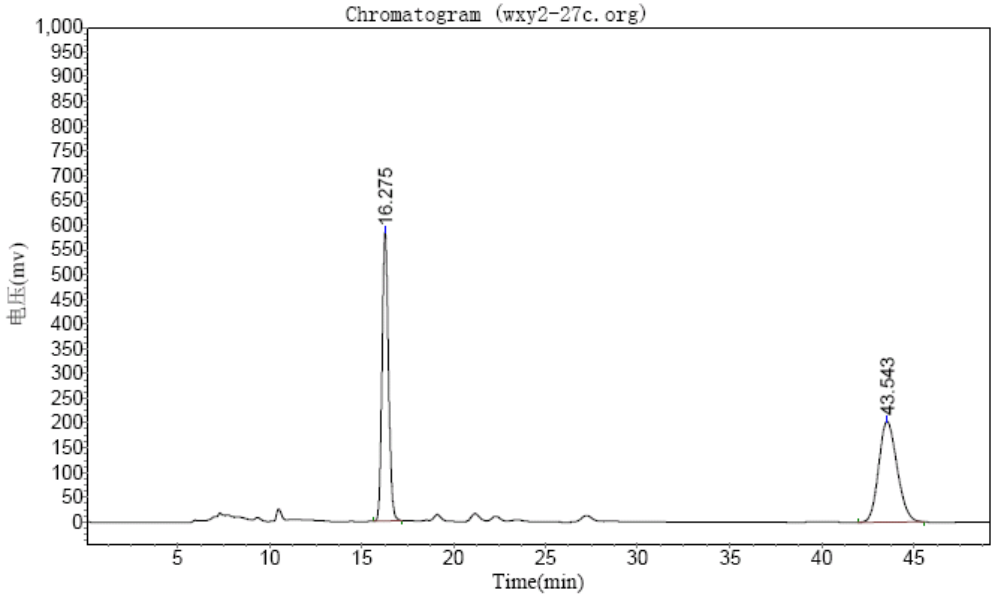
154.88
154.31
147.15
135.05
131.20
128.37
127.97
127.36
126.10
111.65
109.05
100.45
97.04
77.42
77.16
76.91
59.06
56.08
49.70
44.95
38.23
36.13
22.59
17.68
14.38
Current Data Parameters
NAME WXY20091201
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091201
Time 13.49
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 136
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 128
DW 16.650 usec
DE 6.00 usec
TE 294.9 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13c
P1 9.50 usec
PL1 -0.59 dB
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 16.59 dB
PL13 16.50 dB
SFO2 500.1320005 MHz
F2 - Processing parameters
SI 32768
SF 125.7577745 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

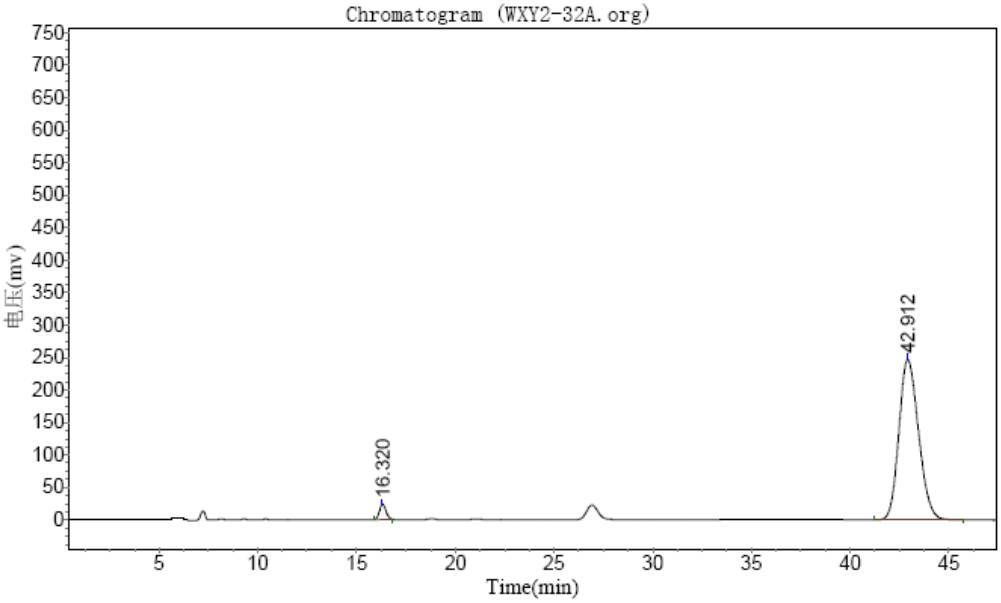


3i



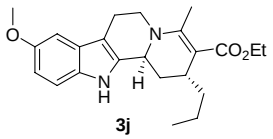
Results

Peak ID	Ret Time	Height	Area	Conc.
	16.275	583587.938	14128806.000	49.9025
	43.543	203547.875	14183990.000	50.0975
		787135.813	28312796.000	100.0000

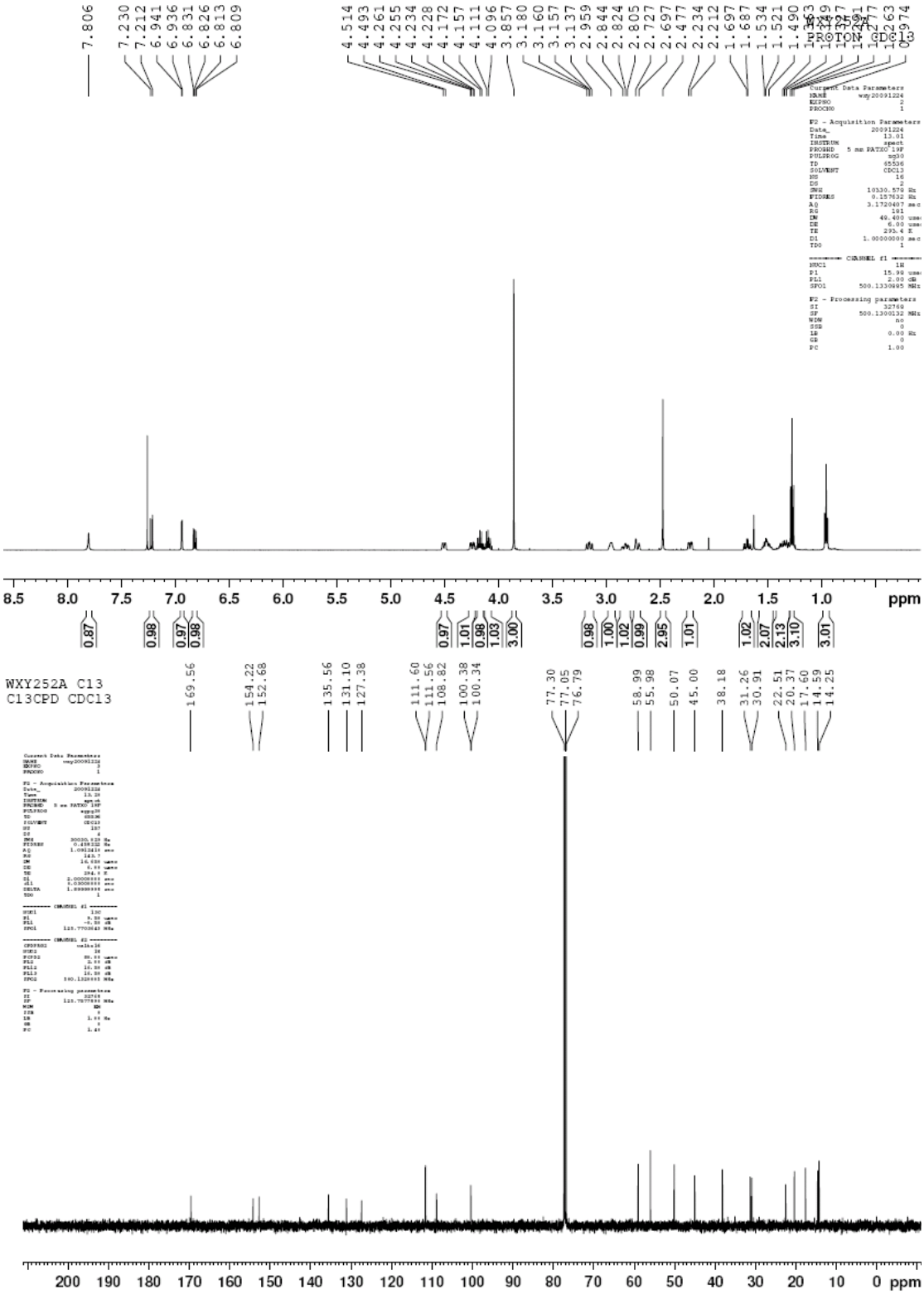


Results

Peak ID	Ret Time	Height	Area	Conc.
	16.320	23443.834	531187.500	3.0053
	42.912	247497.219	17143704.000	96.9947
		270941.053	17674891.500	100.0000


O
N
CO2Et
N
H
H

3j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6.936
6.831
6.826
6.813
6.809
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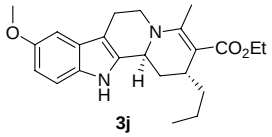
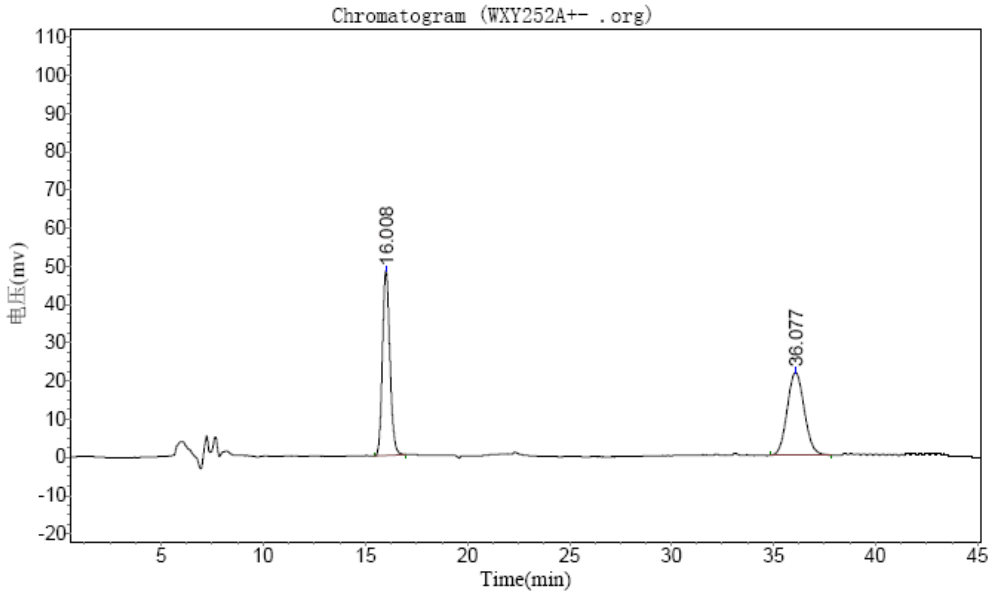
4.493
4.261
4.255
4.234
4.228
4.172
4.157
4.111
4.096
3.857
3.180
3.160
3.157
3.137
2.959
2.844
2.824
2.805
2.727
2.697
2.477
2.234
2.212
1.697
1.687
1.534
1.521
1.490
WXY252A
PROTON CDCl3
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 ppm
0.87 0.98 0.97 0.98 0.97 1.01 0.98 1.03 3.00 0.98 1.00 1.02 0.99 2.95 1.01 1.02 2.07 2.13 3.10 3.01
WXY252A C13
C13CPD CDCl3
169.56
154.22
152.68
135.56
131.10
127.38
111.60
111.56
108.82
100.38
100.34
77.30
77.05
76.79
58.99
55.98
50.07
45.00
38.18
31.26
30.91
22.51
20.37
17.60
14.59
14.25
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

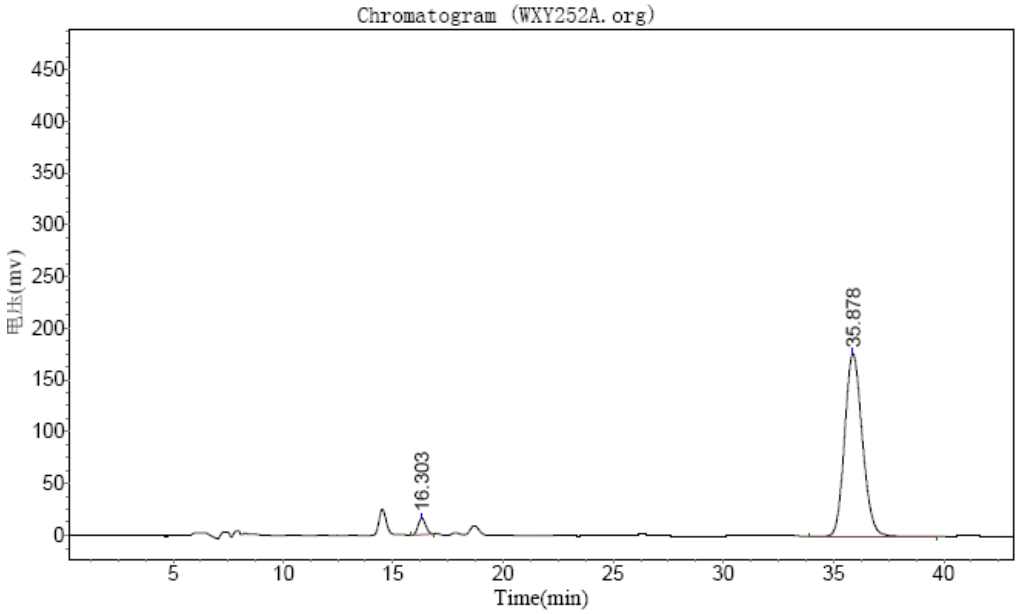


3j



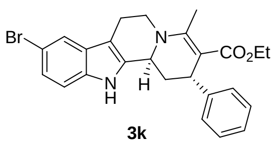
Results

Peak ID	Ret Time	Height	Area	Conc.
	16.008	48183.598	1219790.000	49.0305
	36.077	21746.527	1268027.250	50.9695
		69930.125	2487817.250	100.0000

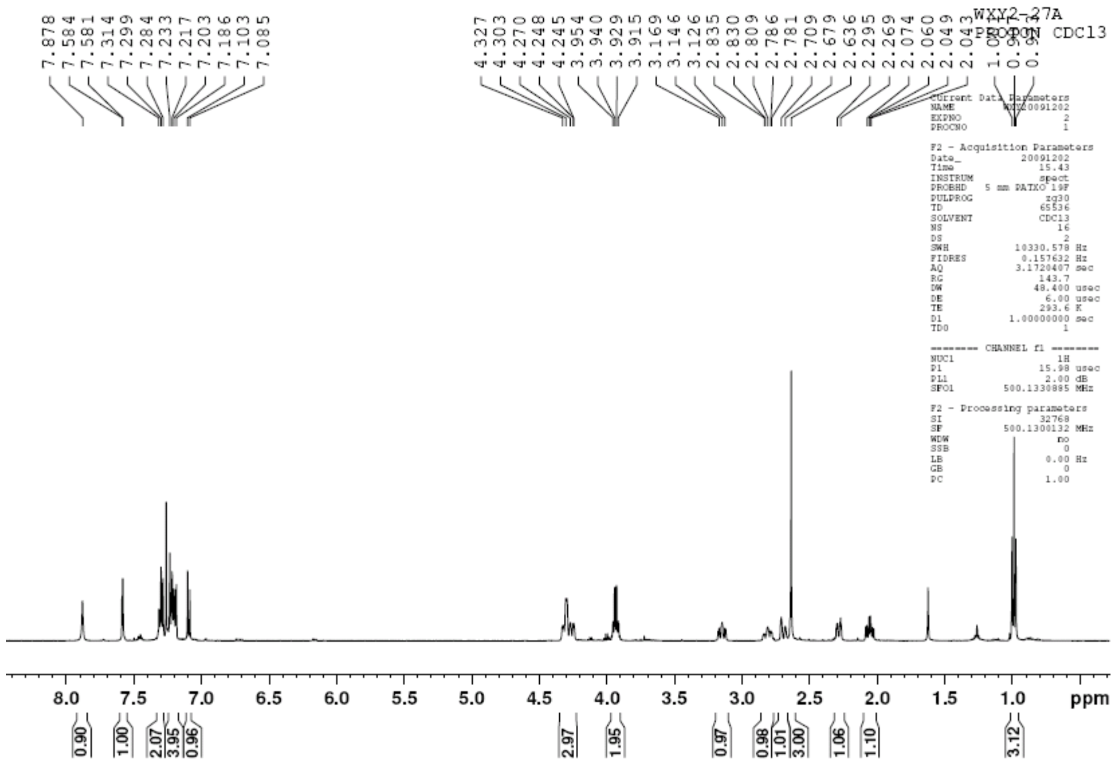


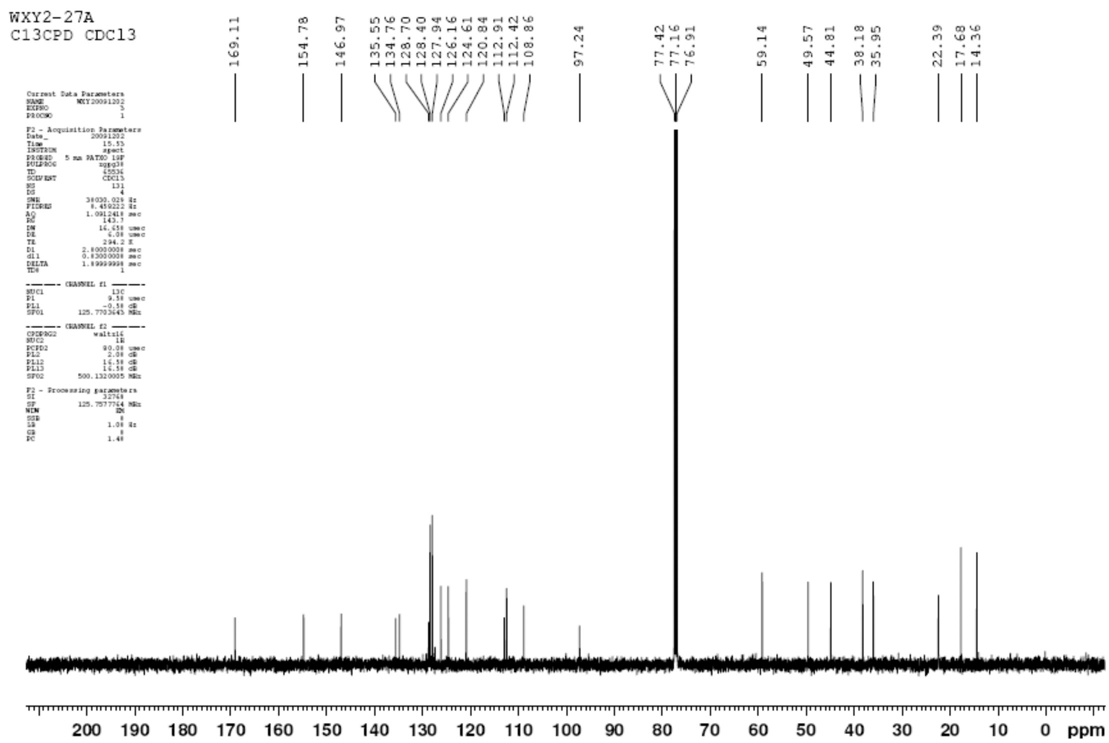
Results

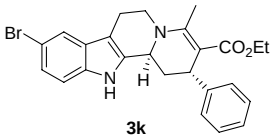
Peak ID	Ret Time	Height	Area	Conc.
	16.303	15853.293	376273.813	3.6692
	35.878	176466.641	9878745.000	96.3308
		192319.934	10255018.813	100.0000


Br
N
H
H
CO2Et

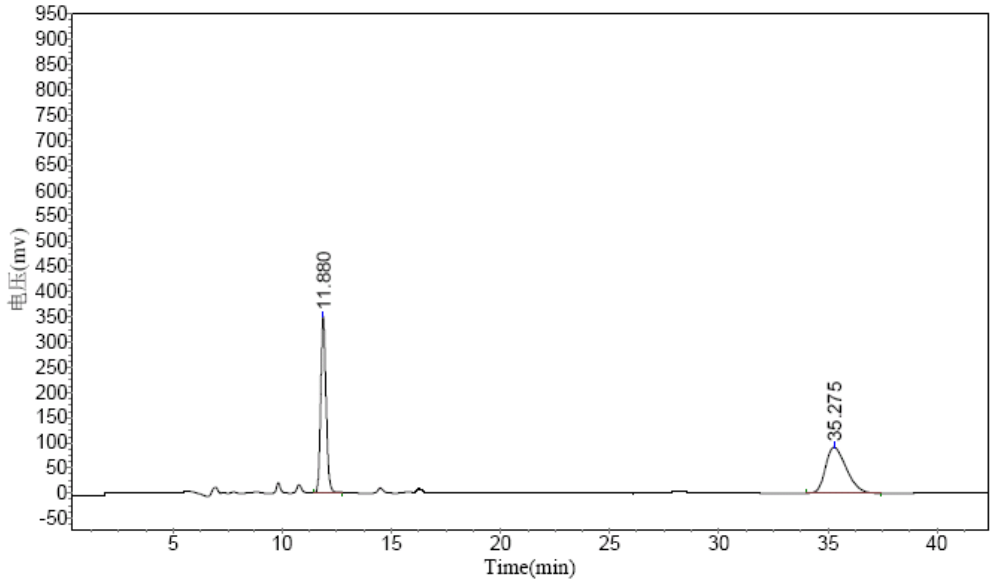
3k


WXY2-27A
PROTON CDCl3
ppm


WXY2-27A
C13CPD CDCl3
ppm

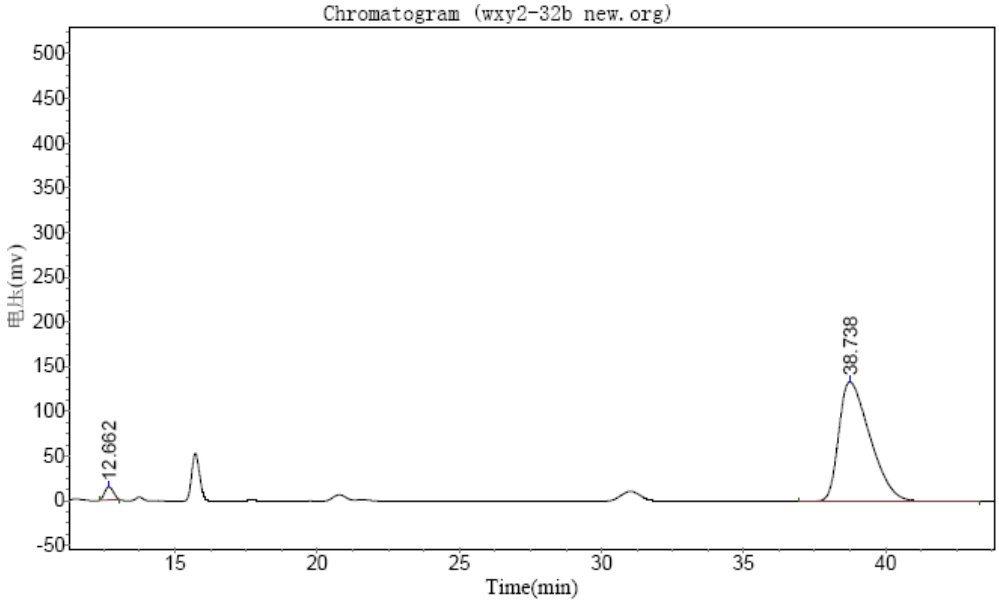


3k



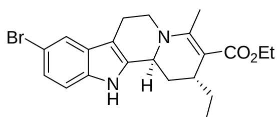
Results

Peak ID	Ret Time	Height	Area	Conc.
	11.880	349567.094	5976516.000	49.8360
	35.275	90973.883	6015850.500	50.1640
		440540.977	11992366.500	100.0000

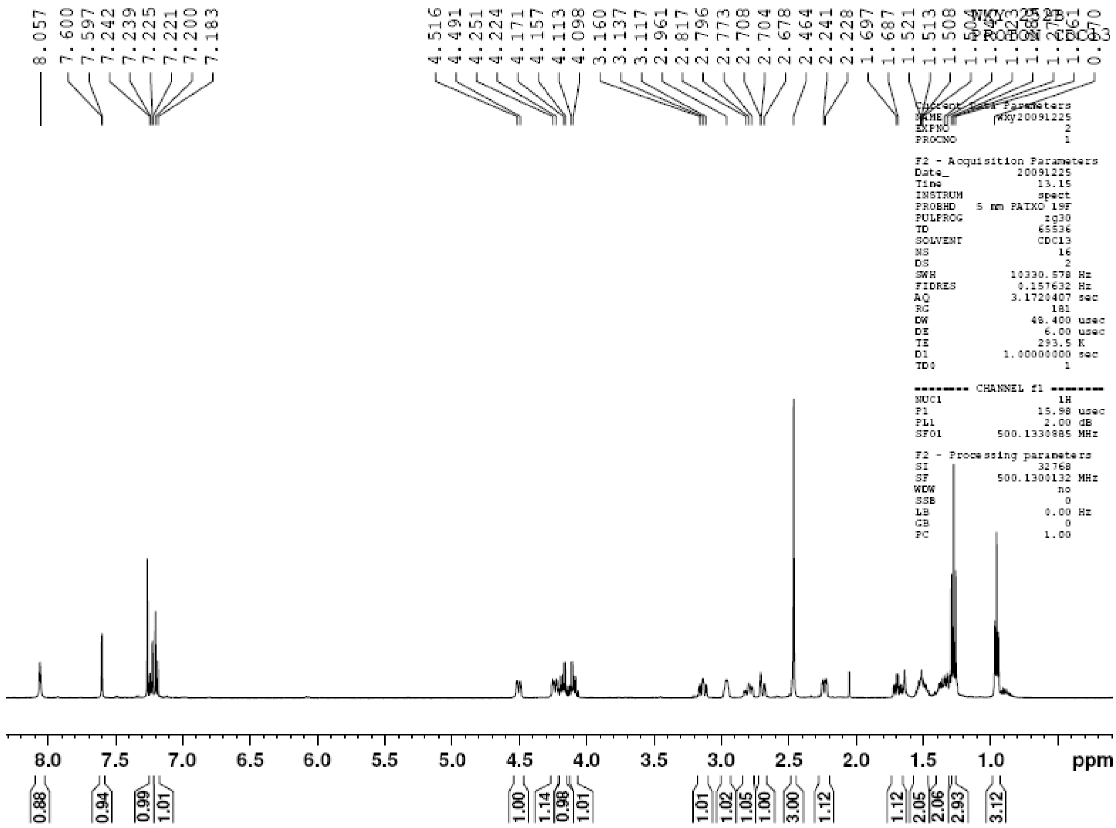


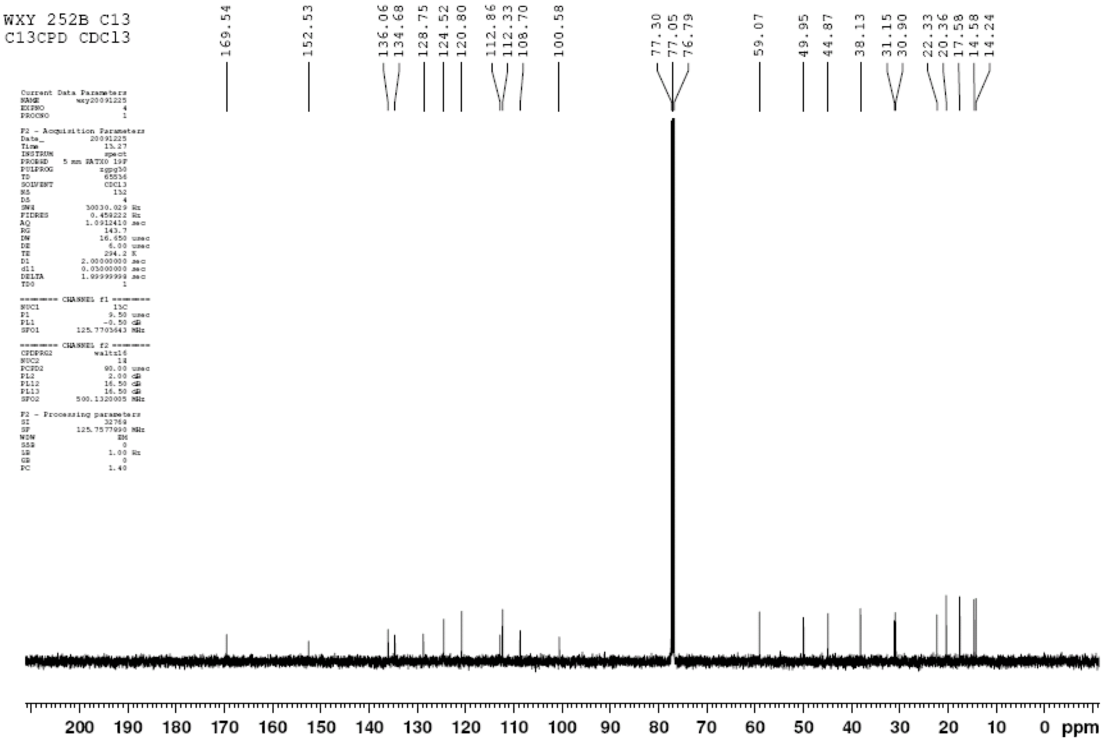
Results

Peak ID	Ret Time	Height	Area	Conc.
	12.662	14737.358	319092.406	3.0196
	38.738	134027.313	10248239.000	96.9804
		148764.671	10567331.406	100.0000


Br
N
H
H
N
CO2Et

3l


WXY 252B
PROTON CDCl3
ppm


WXY 252B C13
C13CPD CDCl3
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152.53
136.06
134.68
128.75
124.52
120.80
112.86
112.33
108.70
100.58
77.30
77.05
76.79
59.07
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44.87
38.13
31.15
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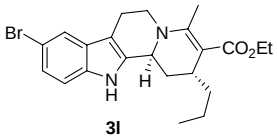
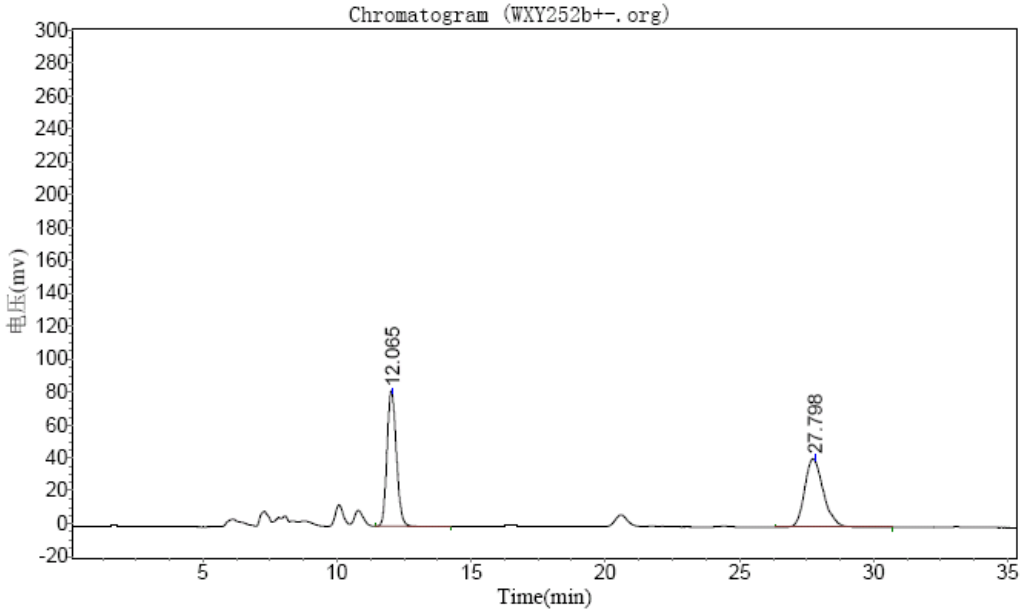
20.36
17.58
14.58
14.24
ppm

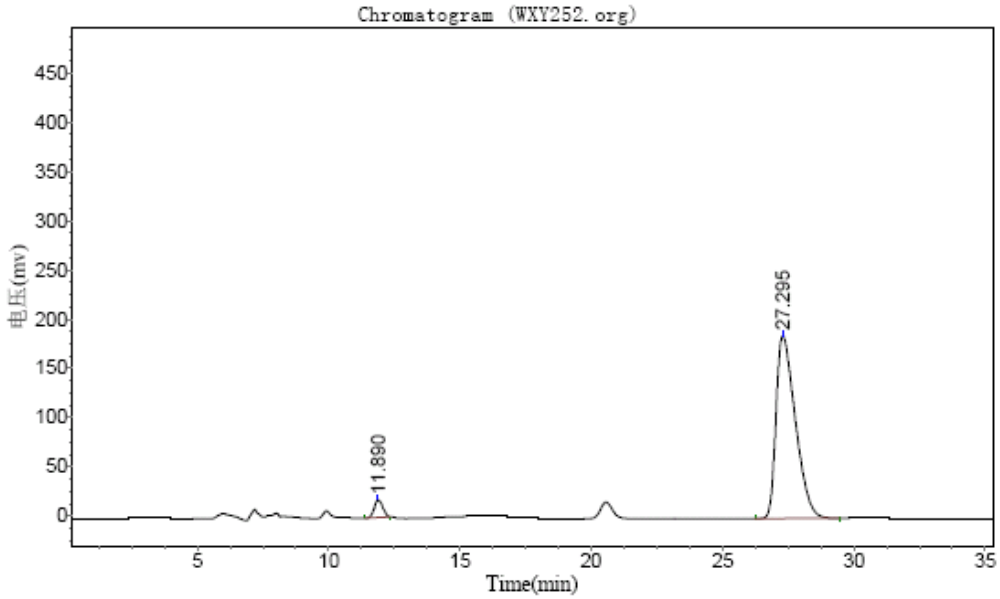


3l



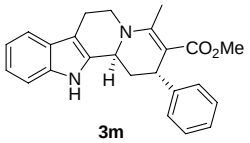
Results

Peak ID	Ret Time	Height	Area	Conc.
	12.065	82431.680	2081647.375	49.9454
	27.798	41552.148	2086199.750	50.0546
		123983.828	4167847.125	100.0000

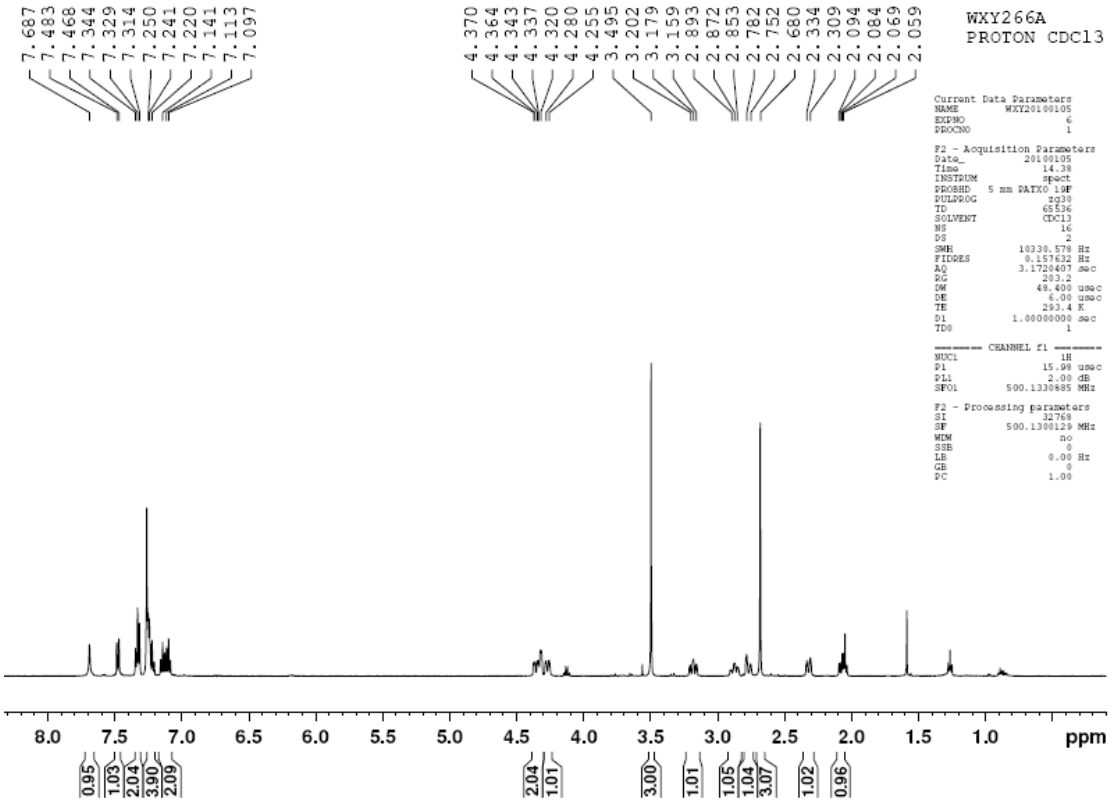


Results

Peak ID	Ret Time	Height	Area	Conc.
	11.890	17737.182	399702.188	4.0456
	27.295	185627.391	9480238.000	95.9544
		203364.572	9879940.188	100.0000


N
CO2Me
N
H
H

3m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7.314
7.250
7.241
7.220
7.141
7.113
7.097
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2.782
2.752
2.680
2.334
2.309
2.094
2.084
2.069
2.059
WXY266A
PROTON CDCl3
Current Data Parameters
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EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100105
Time 14.38
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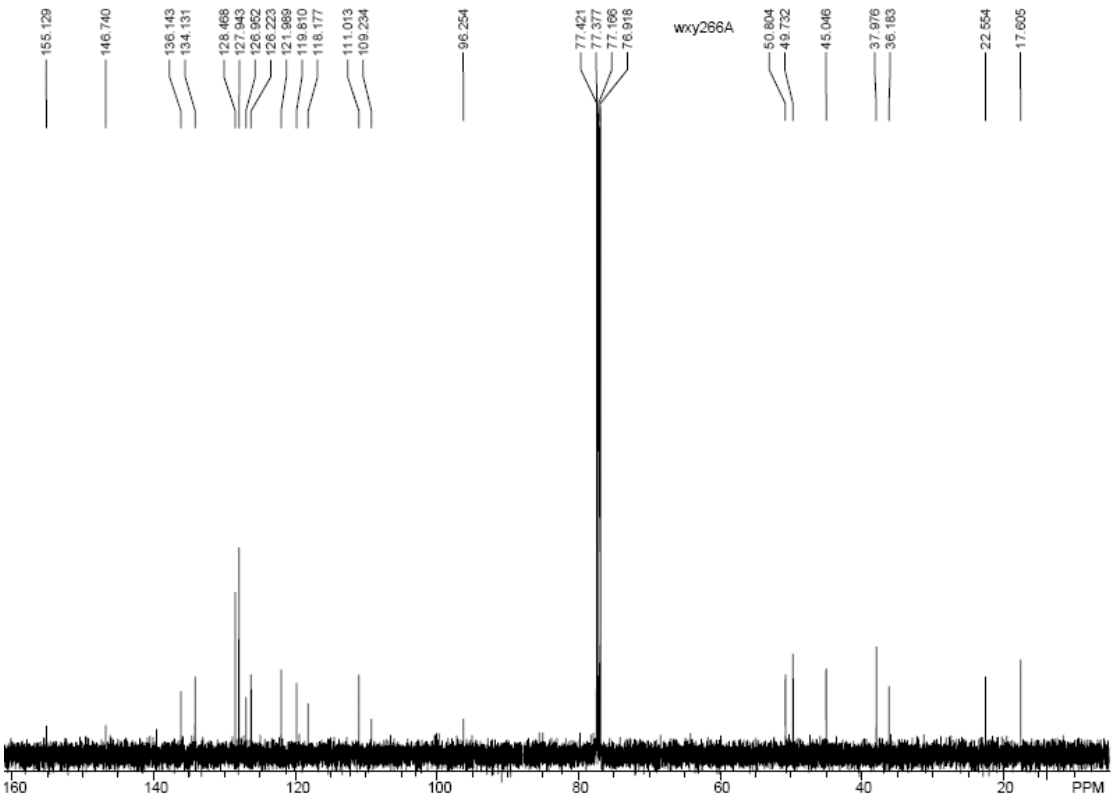
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 203.2
DW 48.400 usec
DE 6.00 usec
TE 293.4 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.95
1.03
2.04
3.90
2.09
2.04
1.01
3.00
1.01
1.05
1.04
3.07
1.02
0.96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146.740
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134.131
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127.943
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126.223
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119.810
118.177
111.013
109.234
96.254
77.421
77.377
77.166
76.918
wxy266A
50.804
49.732
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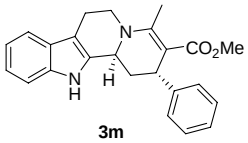
37.976
36.183
22.564
17.605
160
140
120
100
80
60
40
20
PPM



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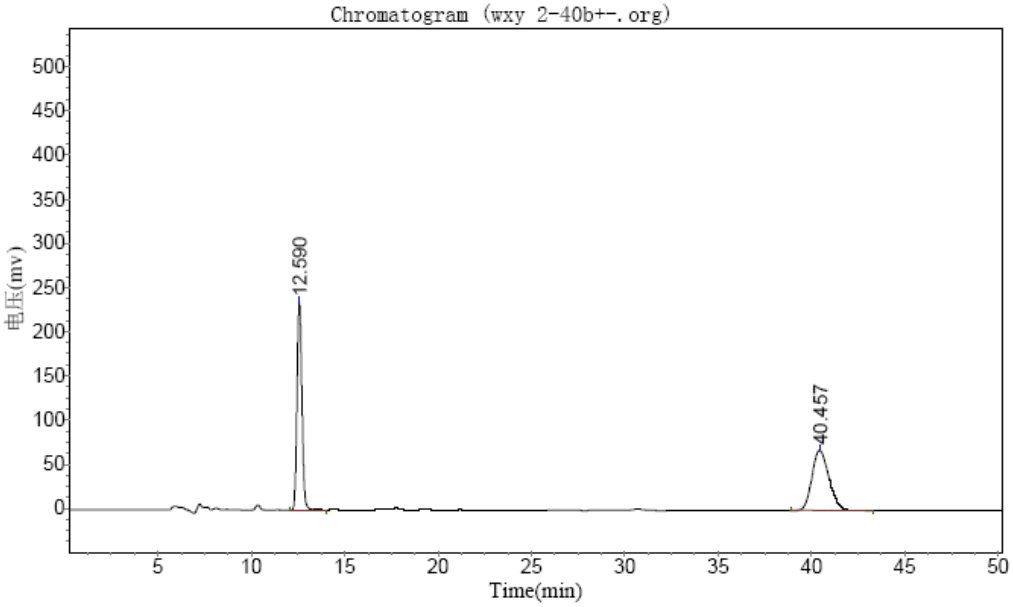
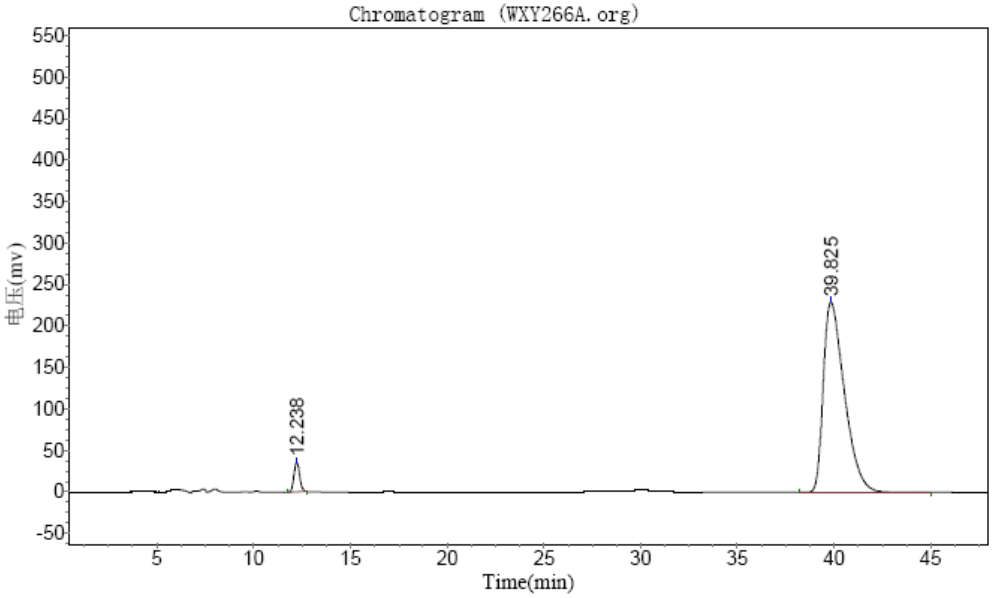


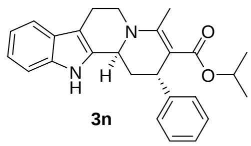
Results

Peak ID	Ret Time	Height	Area	Conc.
	12.590	234035.969	4313317.000	49.5764
	40.457	68382.773	4387030.500	50.4236
		302418.742	8700347.500	100.0000

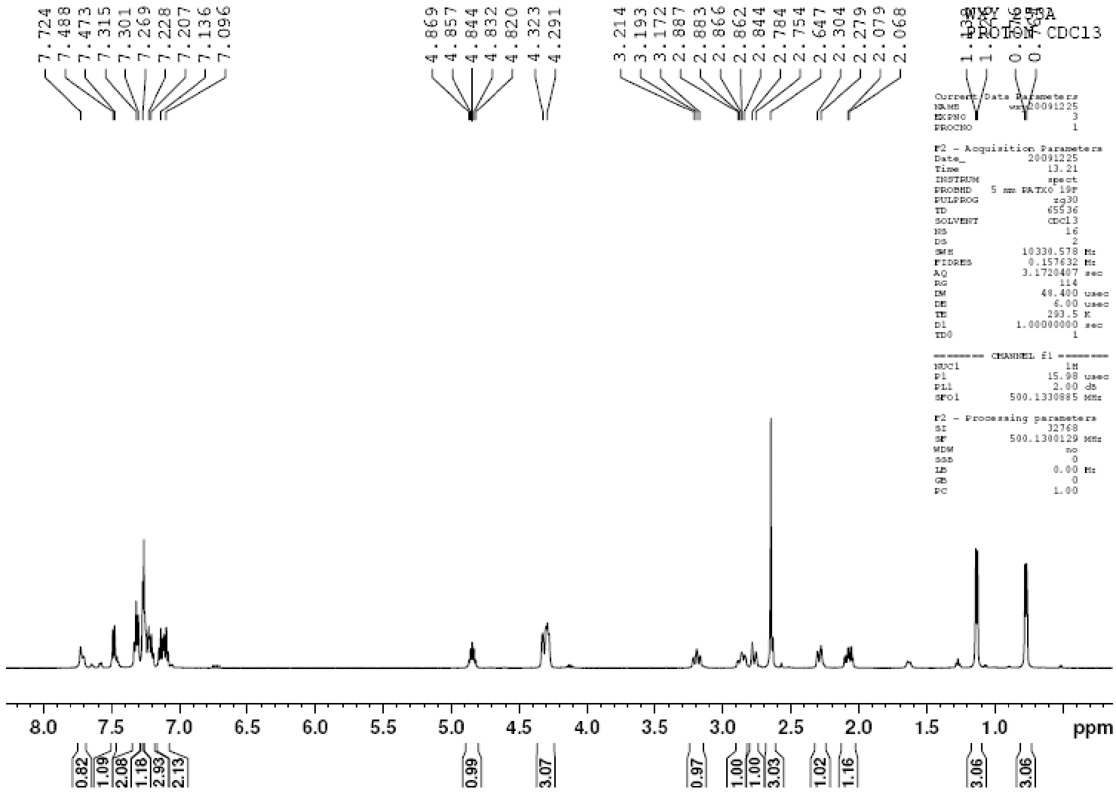


Results

Peak ID	Ret Time	Height	Area	Conc.
	12.238	35515.270	721684.188	3.9736
	39.825	229514.078	17440088.000	96.0264
		265029.348	18161772.188	100.0000


N
O
O
N
H
H

3n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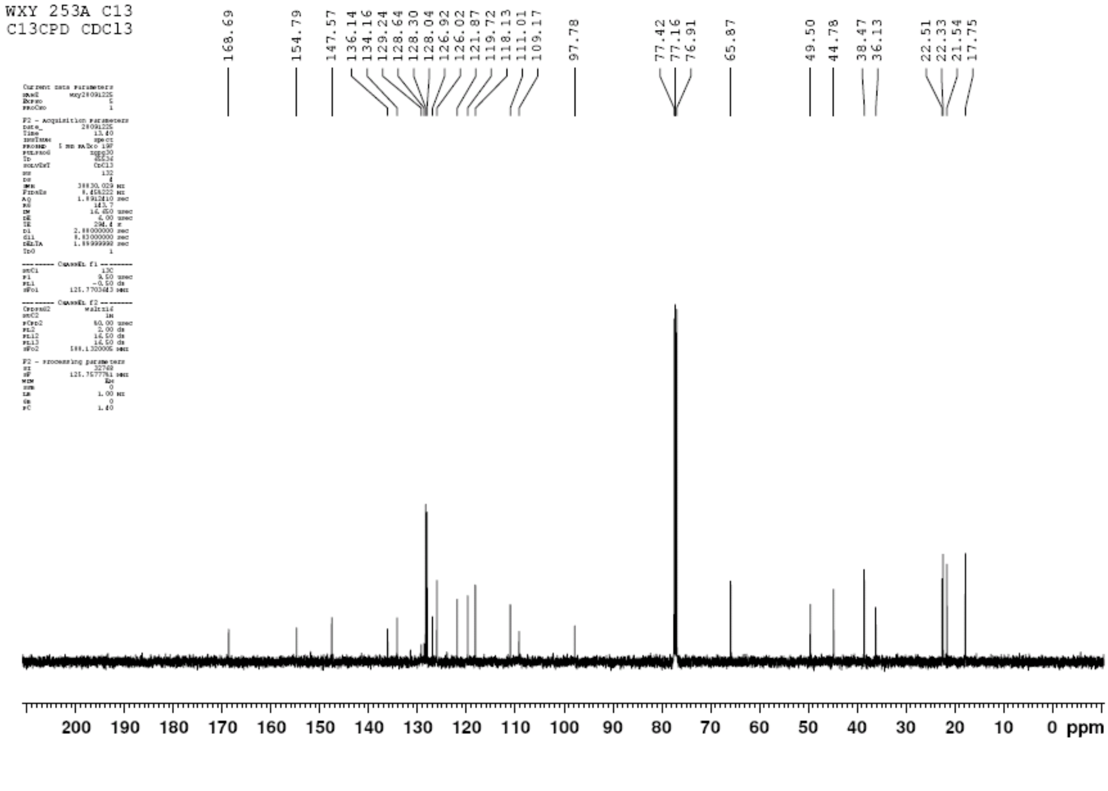
7.488
7.473
7.315
7.301
7.269
7.228
7.207
7.136
7.096
4.869
4.857
4.844
4.832
4.820
4.323
4.291
3.214
3.193
3.172
2.887
2.883
2.866
2.862
2.844
2.784
2.754
2.647
2.304
2.279
2.079
2.068
WXY 253A
PROTON CDCl3
Current Data Parameters
NAME wxy20091225
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091225
Time 13.21
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 114
DW 48.400 usec
DE 6.00 usec
TE 293.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SFO1 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
ppm
0.82
1.09
2.08
1.18
2.93
2.13
0.99
3.07
0.97
1.00
1.00
3.03
1.02
1.16
3.06
3.06


WXY 253A C13
C13CPD CDCl3
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154.79
147.57
136.14
134.16
129.24
128.64
128.30
128.04
126.92
126.02
121.87
119.72
118.13
111.01
109.17
97.78
77.42
77.16
76.91
65.87
49.50
44.78
38.47
36.13
22.51
22.33
21.54
17.75
200
190
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170
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150
140
130
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110
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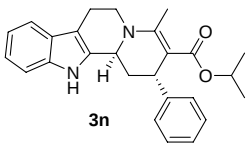
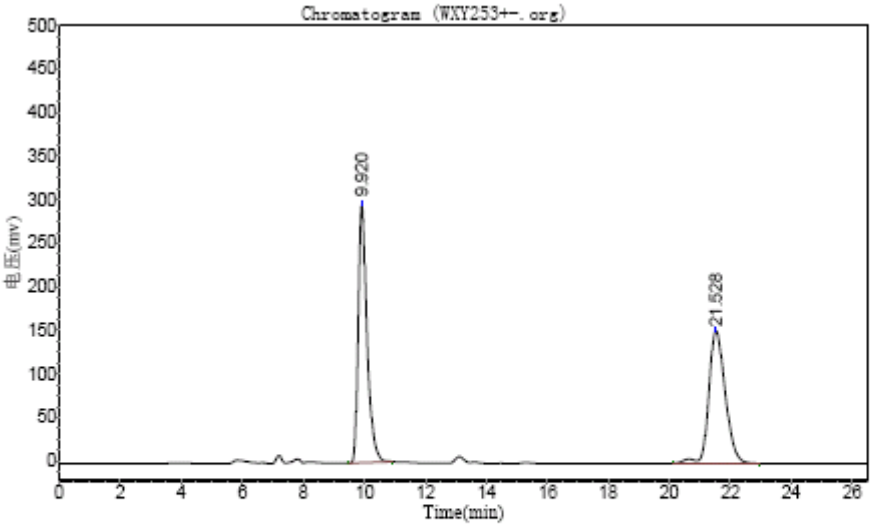
90
80
70
60
50
40
30
20
10
0 ppm

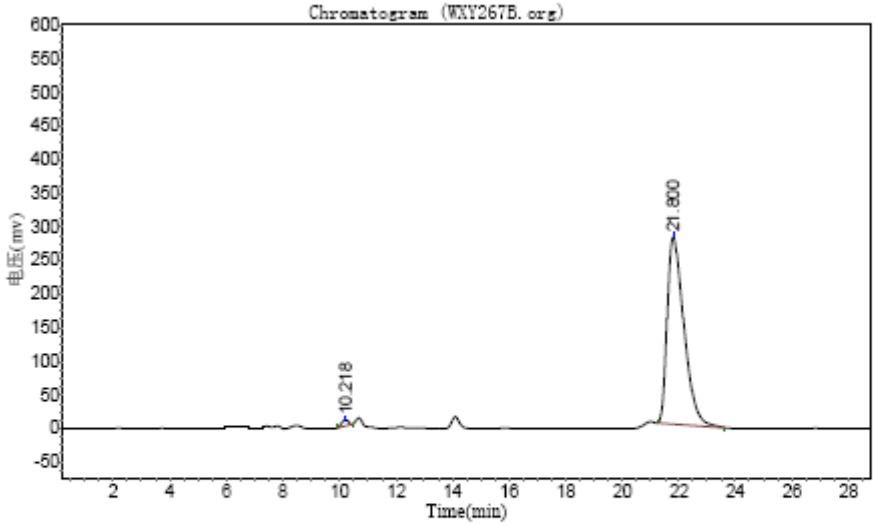


3n



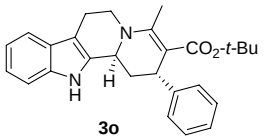
Results

Peak ID	Ret Time	Height	Area	Conc.
	9.920	295421.375	5942029.500	50.7882
	21.528	152728.734	5757605.500	49.2118
		448150.109	11699635.000	100.0000

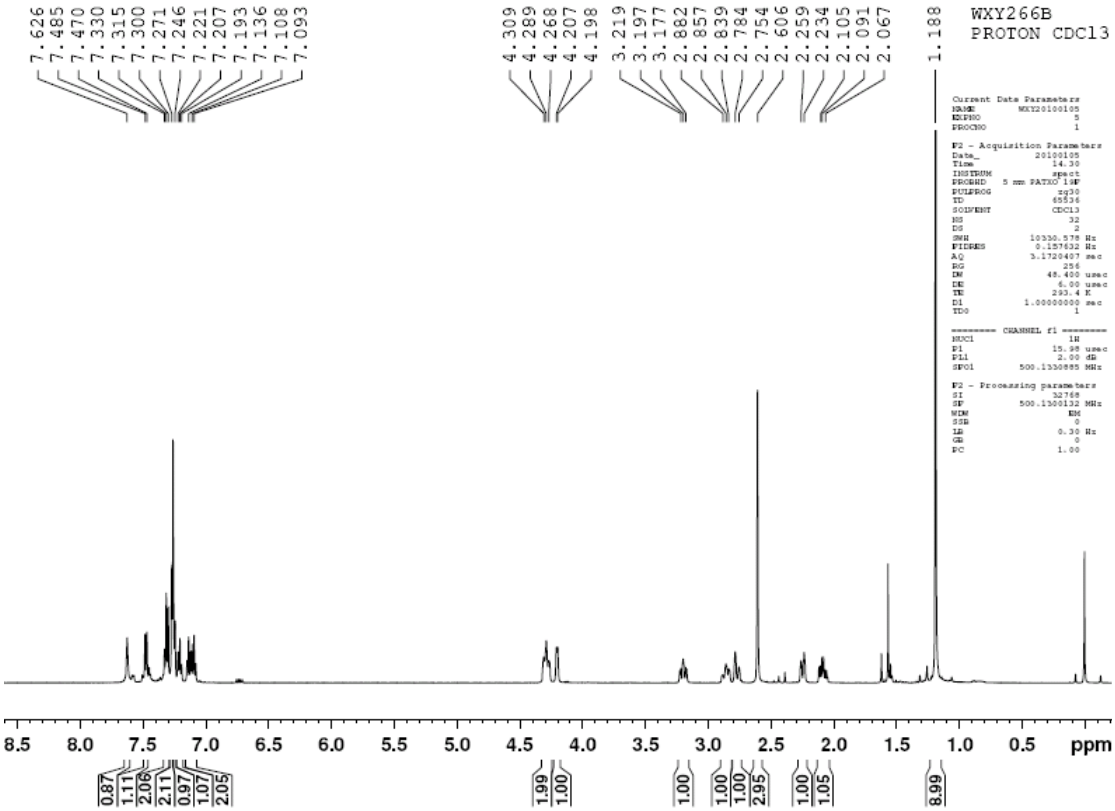


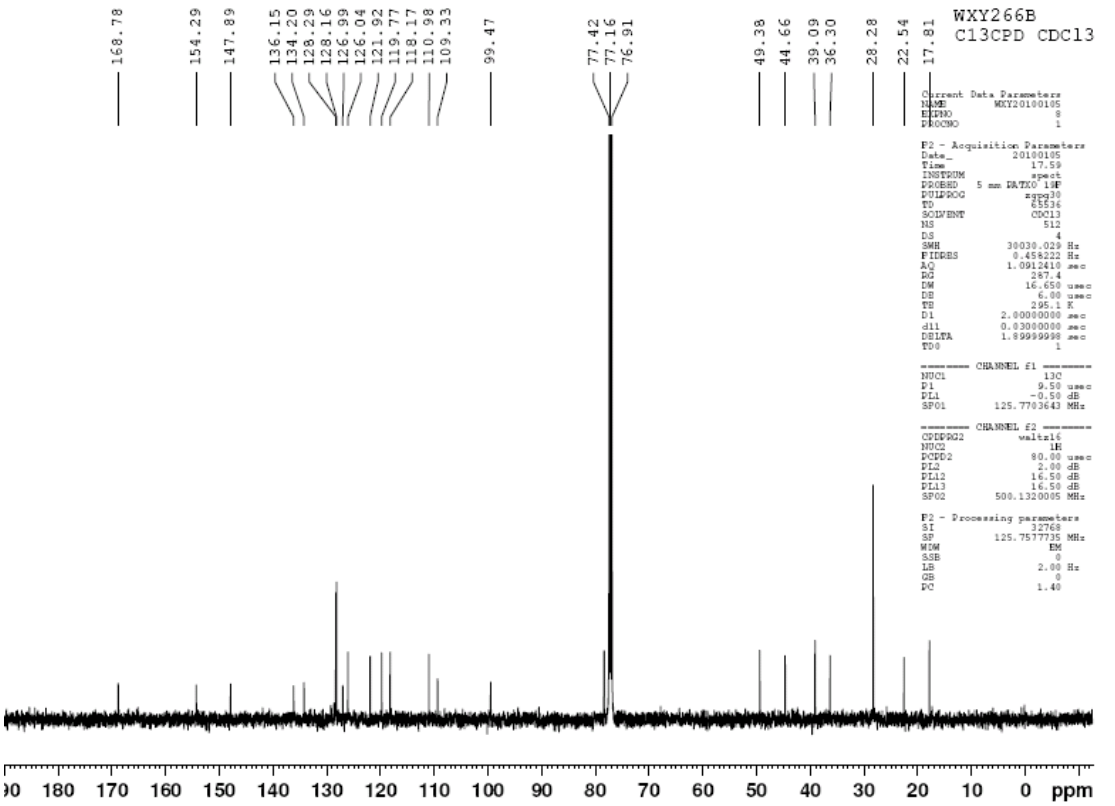
Results

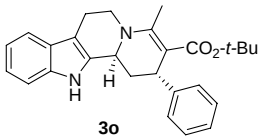
Peak ID	Ret Time	Height	Area	Conc.
	10.218	9253.499	146482.844	1.2600
	21.800	278388.563	11479469.000	98.7400
		287642.062	11625951.844	100.0000


CO2-t-Bu
N
H
H

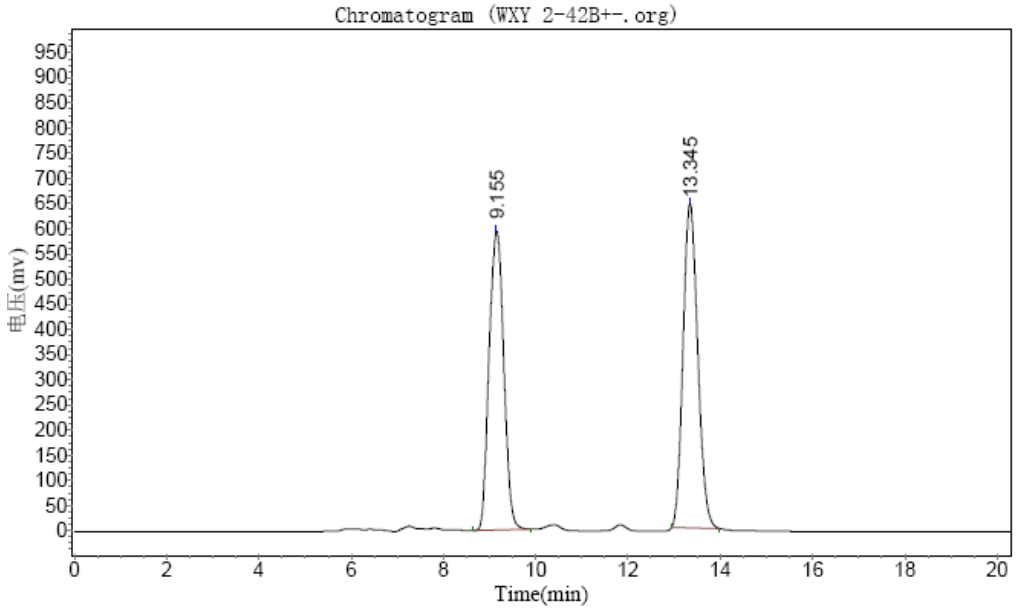
3o


WXY266B
PROTON CDCl3
7.626
7.485
7.470
7.330
7.315
7.300
7.271
7.246
7.221
7.207
7.193
7.136
7.108
7.093
4.309
4.289
4.268
4.207
4.198
3.219
3.197
3.177
2.882
2.857
2.839
2.784
2.754
2.606
2.259
2.234
2.105
2.091
2.067
1.188
0.87
1.11
2.06
2.11
0.97
1.07
2.05
1.99
1.00
1.00
1.00
1.00
2.95
1.00
1.05
8.99
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 ppm


WXY266B
C13CPD CDCl3
168.78
154.29
147.89
136.15
134.20
128.29
128.16
126.99
126.04
121.92
119.77
118.17
110.98
109.33
99.47
77.42
77.16
76.91
49.38
44.66
39.09
36.30
28.28
22.54
17.81
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

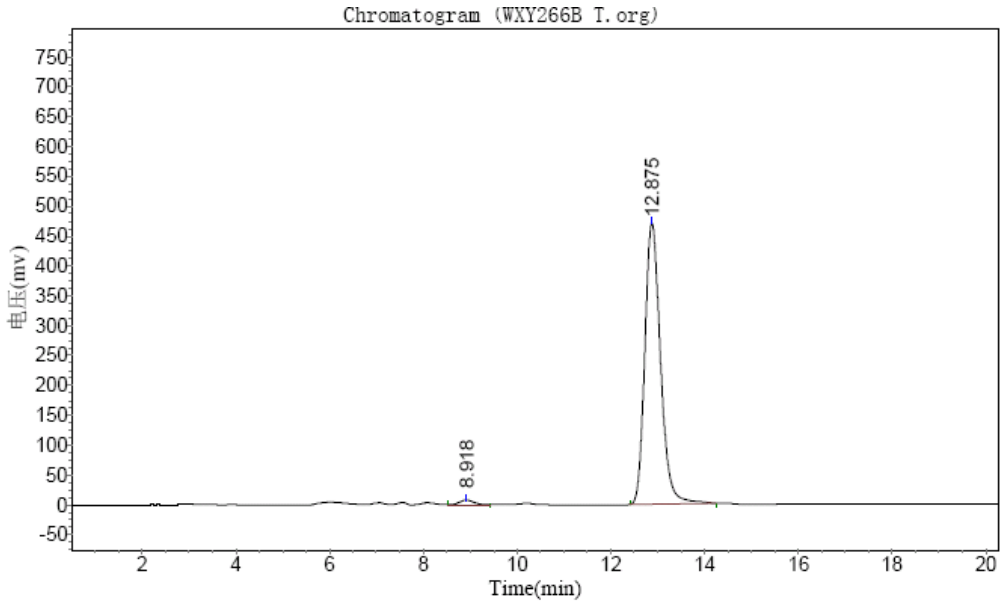


3o



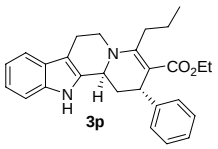
Results

Peak ID	Ret Time	Height	Area	Conc.
	9.155	593530.688	13087335.000	47.9745
	13.345	644479.000	14192422.000	52.0255
		1238009.688	27279757.000	100.0000

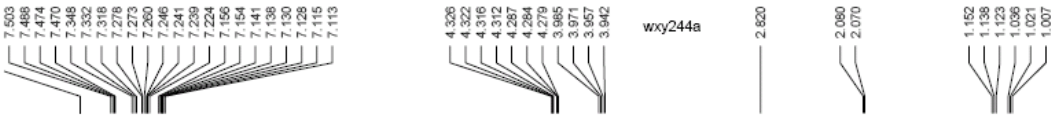


Results

Peak ID	Ret Time	Height	Area	Conc.
	8.918	8485.617	185728.391	1.6671
	12.875	470855.250	10955171.000	98.3329
		479340.867	11140899.391	100.0000


CO2Et
N
H
H

3p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7.332
7.316
7.278
7.273
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7.246
7.241
7.239
7.224
7.156
7.154
7.141
7.138
7.130
7.128
7.115
7.113
4.306
4.322
4.316
4.312
4.287
4.284
4.279
3.965
3.971
3.957
3.942
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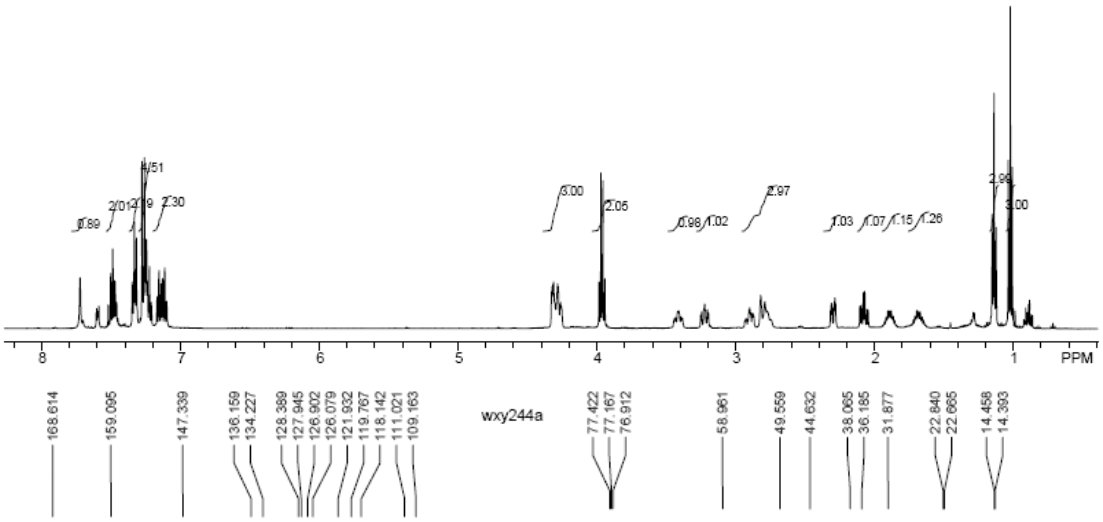
1.138
1.123
1.036
1.021
1.007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3.00
2.05
0.98
1.02
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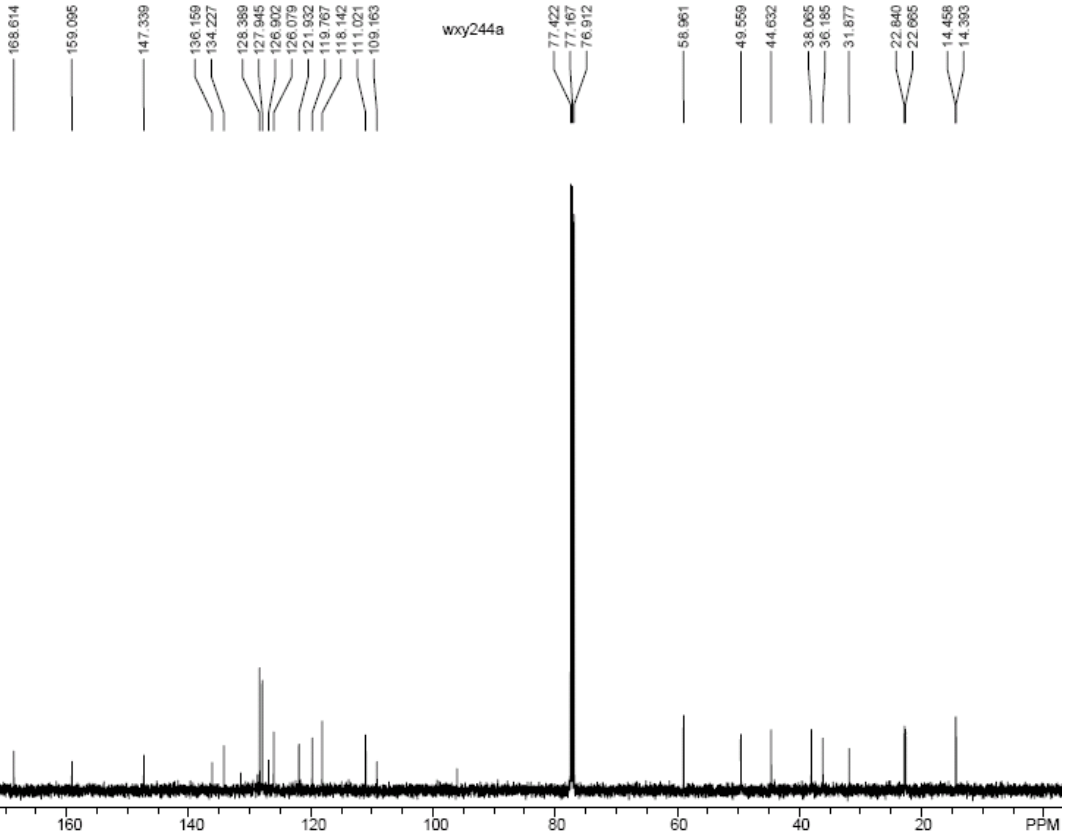
1.03
1.07
1.15
1.26
2.99
3.00
8
7
6
5
4
3
2
1
PPM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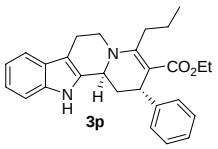
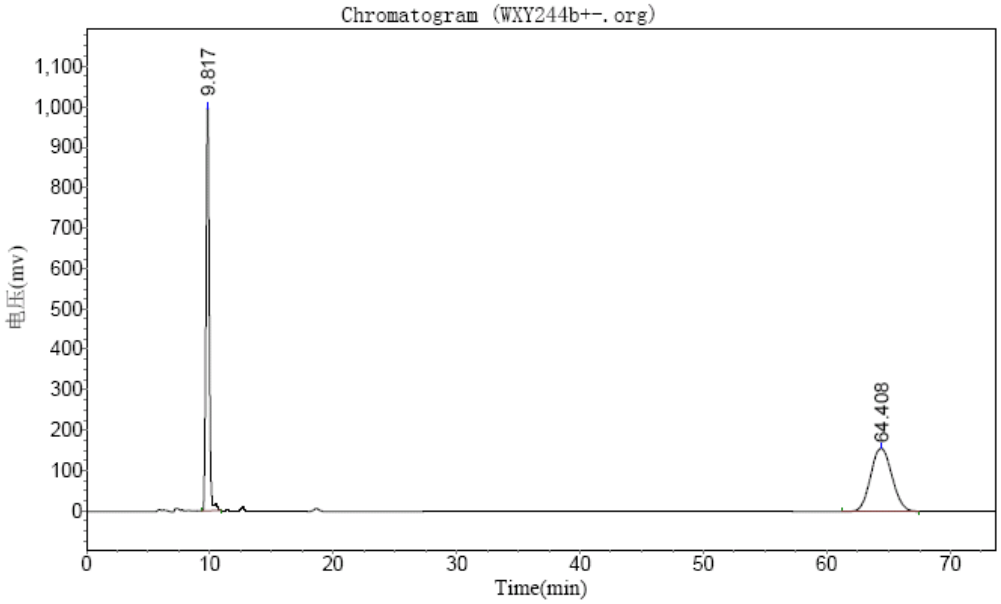
136.159
134.227
128.389
127.945
126.902
126.079
121.932
119.767
118.142
111.021
109.163
wxy244a
77.422
77.167
76.912
58.961
49.559
44.632
38.065
36.185
31.877
22.840
22.665
14.458
14.393
160
140
120
100
80
60
40
20
PPM

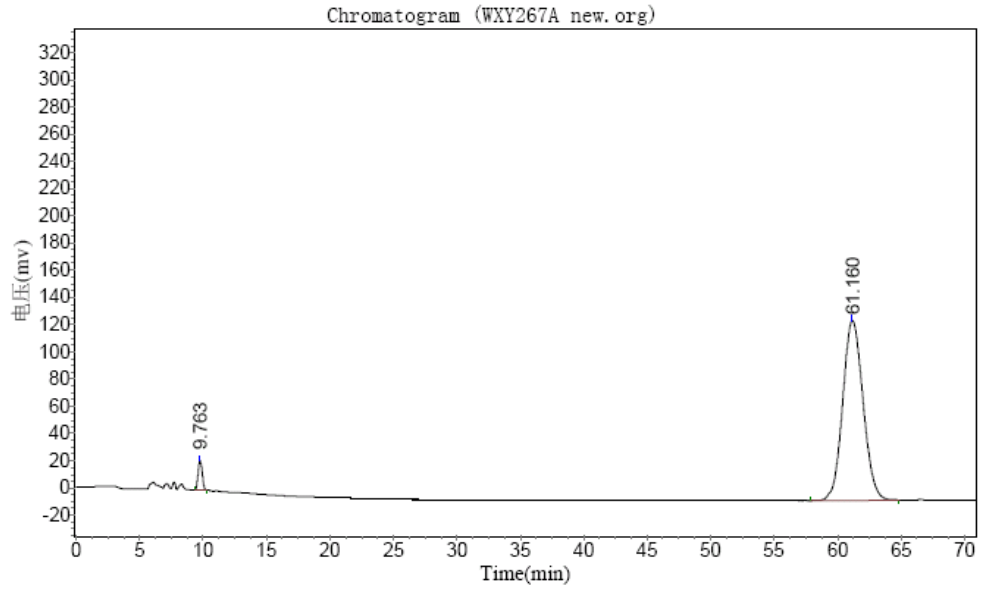


3p



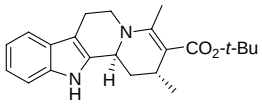
Results

Peak ID	Ret Time	Height	Area	Conc.
	9.817	997086.750	18544504.000	49.5718
	64.408	156493.313	18864882.000	50.4282
		1153580.063	37409386.000	100.0000

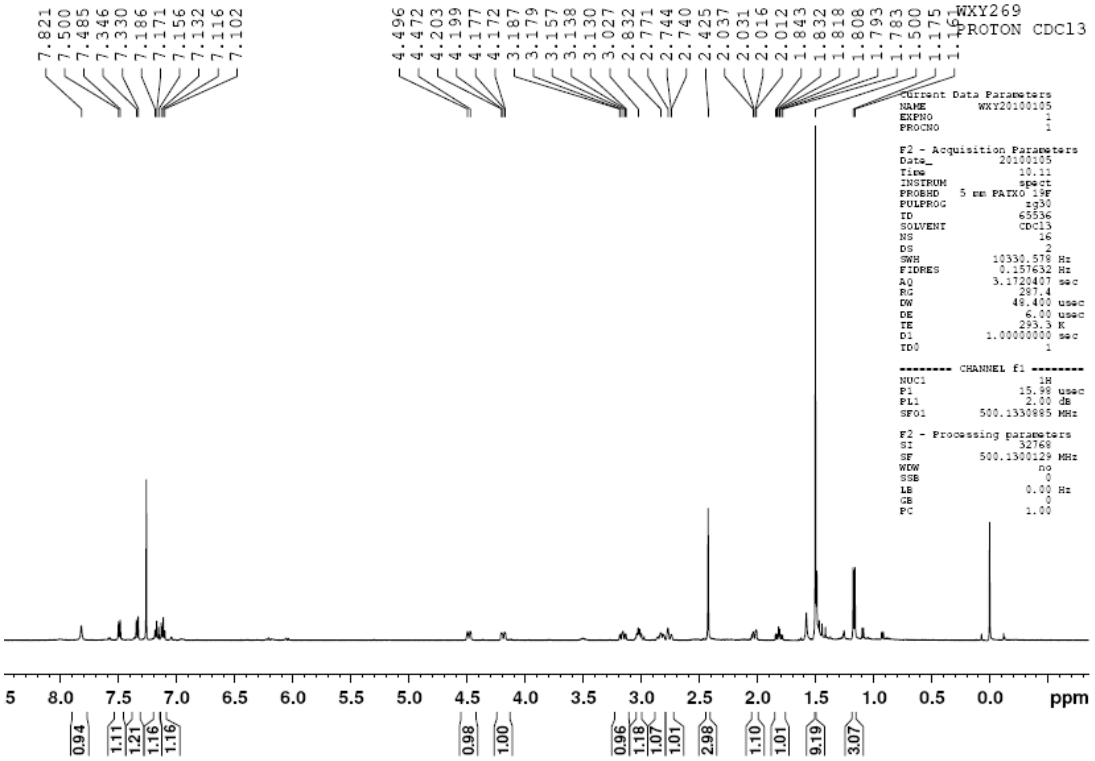


Results

Peak ID	Ret Time	Height	Area	Conc.
	9.763	21432.580	481845.375	3.1625
	61.160	132374.922	14754438.000	96.8375
		153807.502	15236283.375	100.0000


N
CO2-t-Bu
N
H
H

3q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7.171
7.156
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7.116
7.102
4.496
4.472
4.203
4.199
4.177
4.172
3.187
3.179
3.157
3.138
3.130
3.027
2.832
2.771
2.744
2.740
2.425
2.037
2.031
2.016
2.012
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1.818
1.808
1.793
1.783
1.500
1.175
1.161
WXY269
PROTON CDCl3
Current Data Parameters
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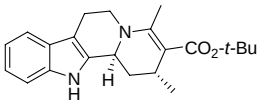
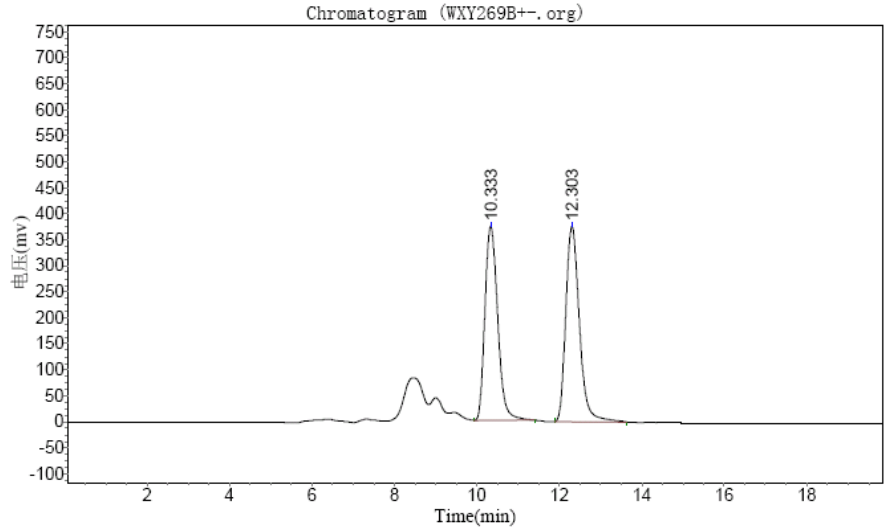
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100105
Time 10.11
INSTRUM spect
PROBHD 5 mm PATXO 19F
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 287.4
DW 48.400 usec
DE 6.00 usec
TE 293.3 K
D1 1.00000000 sec
TD0 1
CHANNEL f1
NUC1 1H
P1 15.98 usec
PL1 2.00 dB
SF01 500.1330885 MHz
F2 - Processing parameters
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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
0.94
1.11
1.21
1.16
1.16
0.98
1.00
0.96
1.18
1.07
1.01
2.98
1.10
1.01
9.19
3.07

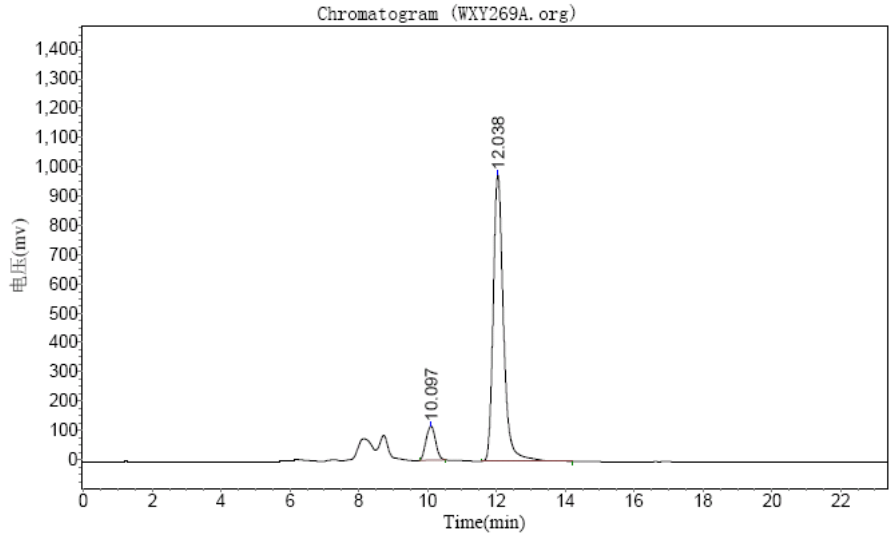


3q



Results

Peak ID	Ret Time	Height	Area	Conc.
	10.333	372825.344	8012541.500	48.1830
	12.303	375958.688	8616841.000	51.8170
		748784.031	16629382.500	100.0000



Results

Peak ID	Ret Time	Height	Area	Conc.
	10.097	115815.563	2228290.000	9.8723
	12.038	979336.313	20342834.000	90.1277
		1095151.875	22571124.000	100.0000