Supporting Information for

Synthesis of (*1R,2R*)-DPEN-Derived Triazolium Salts and Their Application in Asymmetric Intramolecular Stetter Reactions

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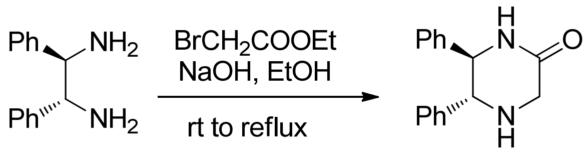
General Methods.

All reactions utilizing air- or moisture-sensitive reagents were carried out in flame-dried glassware under a dry Ar atmosphere. All solvents were purified and dried according to standard methods prior to use.

^{1}H NMR spectra were recorded on a VARIAN Mercury 300 MHz spectrometer in chloroform-d_3. Chemical shifts are reported in ppm with the internal TMS signal at 0.0 ppm as a standard. The data are reported as (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, brs = broad singlet, coupling constant (s) in Hz, integration). ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 MHz spectrometer in chloroform-d_3. Chemical shifts are reported in ppm with the internal chloroform signal at 77.0 ppm as a standard. ^{19}F NMR spectra were recorded on a VARIAN Mercury 282 MHz spectrometer. Chemical shifts are reported in ppm with CCl_3F signal at 0.00 ppm as an external standard.

Optical rotations ($[\alpha]_D$) were measured on a Perkin-Elmer 785A UV/VIS Detector polarimeter. Enantiomeric excesses were determined by HPLC analysis on chiral stationary phases [CHIRALPAK AD-H or CHIRALCEL OD-H or CHIRALCEL OB-H (Daicel Chemical Ind., Ltd., Φ 0.46mm × 25 cm)].

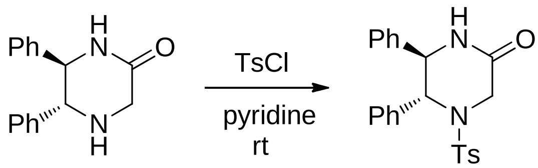
Preparation of (*5R*,*6R*)-5,6-diphenylpiperazin-2-one 7[1]



7

To a solution of (*1R*, *2R*)-1,2-diphenyl-1,2-ethanediamine (2.12 g, 10 mmol) and NaOH 0.4 g (10 mmol) in 30 mL of absolute ethanol was added a solution of ethyl bromoacetate 1.65 g (1.1 mL, 10 mmol) in 20 mL of absolute ethanol over three hours. The reaction was stirred at room temperature for another two days, and then refluxed overnight. After removal of the solvent at reduced pressure, purification by silica gel column chromatography eluting with 5% MeOH in EtOAc afforded title compound **7** as a white solid (1.4 g, 54% yield). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.11-6.86 (m, 10H), 6.51 (s, 1H), 4.40 (d, J = 9.0 Hz, 2H), 3.63 (m, 3H), 2.19 (brs, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 169.7, 138.5, 138.4, 128.28, 128.24, 128.19, 128.10, 127.7, 127.4, 65.8, 64.4, 50.0.

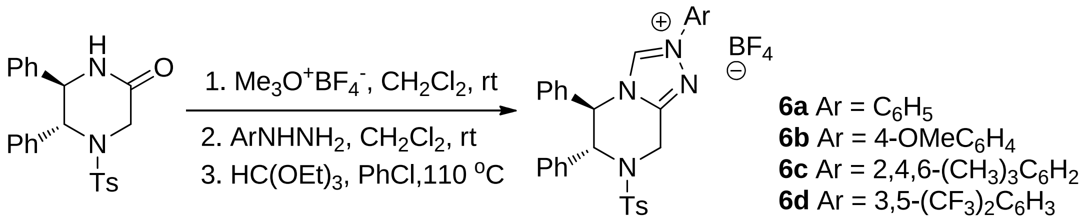
Preparation of (*5R*,*6R*)-5,6-diphenyl-4-tosylpiperazin-2-one 8



7 **8**

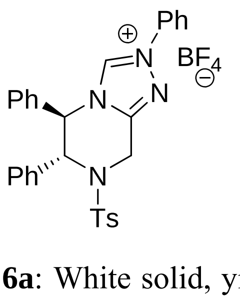
To a solution of compound **7** **(**1.0 g, 4 mmol) in pyridine (20 mL), *p*-toluenesulfonyl chloride (0.99 g, 5.2 mmol) was added slowly and the mixture was stirred at rt for 24 h. The solution was extracted with Et_2O and washed with HCl (1N), water and brine. The organic layer was dried over $NaSO_4$, filtered, and concentrated under reduced pressure. The crude residue was purified by flash chromatography on silica gel (EtOAc/n-hexane = 1/1) afforded title compound **8** as a white solid (1.4 g, 87% yield). $[\alpha]_D^{20}$ = +22.4 (c = 1.0, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.94-7.31 (m, 14H), 5.30 (s, 1H), 4.88 (s, 1H), 3.80 (dd, J = 5.6, 1.8 Hz, 2H), 2.27 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 167.4, 143.5, 139.8, 137.3, 135.2, 129.4, 128.9, 128.3, 128.0, 127.2, 127.1, 126.0, 61.2, 59.1, 45.0, 21.4. IR (thin film): ν_{max} (cm^{-1}) = 3431, 3423, 2914, 1685, 1344, 1162, 1096, 1056, 807, 762, 695, 668, 572; MS (ESI, *m/z*, rel. intensity) 407.2 (M+H); HRMS (MALDI) calcd for $C_{23}H_{23}N_2O_3S$ (M+H): 407.1424; Found: 407.1429. m.p. 178-179 ℃.

Preparation of triazolium salts 6a-d[2]

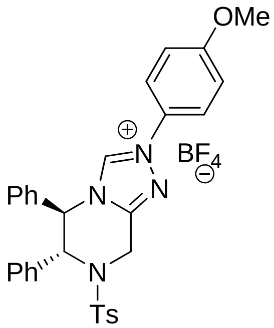


8

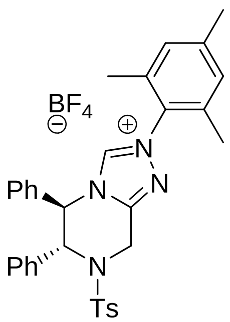
A flamed-dried 50 mL round bottom flask was charged with compound **8** (0.81 g, 2.0 mmol) and CH_2Cl_2 (15 mL). Trimethyloxonium tetrafluoroborate (0.36 g, 2.4 mmol) was added and the mixture was stirred for about 1 day at rt. The corresponding aryl hydrazine (2.4 mmol) was added and stirred for another 1 day. The solvent was evaporated and chlorobenzene (20 mL) was added, followed by triethyl orthoformate (2.5 mL / day, 15 mmol). The mixture was then heated to 110℃ and stirred at this temperature for about 3 d. After completion (monitored by NMR), the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography and further purified by recrystallization in hexane/ethyl acetate. All the yields indicated below refer to those obtained after recrystallization.



6a: White solid, yield 50%. $[\alpha]_D^{20}$ = +121.3^o (c = 0.20, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.05 (s, 1 H), 7.79 (d, *J* = 17.7 Hz, 2 H), 7.47-7.24 (m, 13 H), 7.15 (d, *J* = 8.1 Hz, 2 H), 7.02 (d, *J* = 7.5 Hz, 2 H), 6.78 (s, 1 H), 5.79 (d, *J* = 1.8 Hz, 1 H), 5.13 (d, *J* = 17.4 Hz, 1 H), 4.48 (d, *J* = 17.7 Hz, 1 H), 2.38 (s, 3 H); ^{19}F NMR (282 MHz, $CDCl_3$): δ -150.2, -150.3; ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.3, 144.7, 139.7, 135.2, 134.3, 133.0, 131.4, 130.2, 130.0, 129.7, 129.3, 127.22, 127.17, 126.4, 120.7, 62.1, 61.6, 38.0, 21.5; IR (thin film): ν_{max} (cm^{-1}) = 3064, 2918, 1595, 1455, 1353, 1162, 1055, 760, 689; MS (ESI, *m/z*, rel. intensity) 507.1 (M-BF_4); HRMS (ESI) calcd for $C_{30}H_{27}N_4O_2S$ (M-BF_4): 507.1849; Found: 507.1851. m.p. 213-216 ℃.

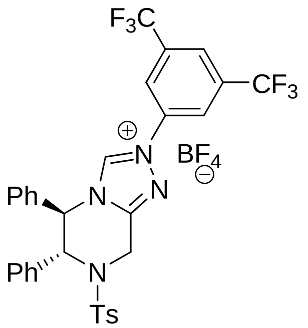


6b: Pale yellow solid, yield 27%; $[\alpha]_D^{20}$ = +24.3° (c = 0.20, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 10.00 (s, 1 H), 7.72 (d, *J* = 8.7 Hz, 2H), 7.36-6.93 (m, 16H), 6.77 (s, 1 H), 5.80 (s, 1 H), 5.09 (d, *J* = 17.4 Hz, 1 H), 4.46 (d, *J* = 17.4 Hz, 1 H), 3.80 (s, 3 H), 2.40 (s, 3 H); ^{19}F NMR (282 MHz, $CDCl_3$): δ -150.2, -150.3; ^{13}C NMR (75 MHz, $CDCl_3$) δ 161.6, 148.9, 144.6, 138.9, 135.4, 135.2, 133.0, 130.0, 129.61, 129.58, 129.3, 127.4, 127.2, 126.3, 122.3, 115.2, 61.9, 61.7, 55.7, 37.9, 21.5; IR (thin film): ν_{max} (cm^{-1}) = 3059, 2918, 2842, 1597, 1520, 1454, 1352, 1260, 1162, 1057, 835, 729, 699; MS (ESI, *m/z*, rel. intensity) 537.5 (M-BF_4); HRMS (ESI) calcd for $C_{31}H_{29}N_4O_3S$ (M-BF_4): 537.1955; Found: 537.1957. m.p. 124-126 ℃.



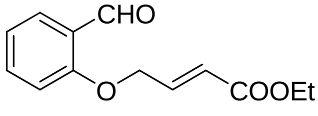
6c: White solid, yield 30%; $[\alpha]_D^{20}$ = +68.4° (c = 0.20, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 9.77 (s, 1 H), 7.42-7.31 (m, 10 H), 7.19 (d, *J* = 6.3 Hz, 2 H), 7.04 (d, *J* = 5.7 Hz, 2 H), 6.98 (s, 3 H), 5.86 (s, 1 H), 5.10 (d, *J* = 17.4 Hz, 1 H), 4.53 (d, *J* =17.4 Hz, 1 H), 2.43 (s, 3 H), 2.33 (s, 3 H), 1.96 (s, 6 H); ^{19}F NMR (282 MHz, $CDCl_3$): δ

-150.1, -150.2; ^{13}C NMR (75 MHz, $CDCl_3$) δ 149.7, 144.7, 144.4, 142.6, 135.4, 135.2, 133.2, 130.0, 129.8, 129.69, 129.65, 129.3, 127.2, 127.1, 126.1, 62.3, 61.7, 38.1, 21.6, 21.2, 17.0; IR (thin film): ν_{max} (cm^{-1}) = 3065, 2924, 2854, 1651, 1597, 1579, 1500, 1450, 1354, 1267, 1162, 1059, 930, 815, 757, 730, 699; MS (ESI, *m/z*, rel. intensity) 549.4 (M-BF_4); HRMS (ESI) calcd for $C_{33}H_{33}N_4O_2S$ (M-BF_4): 549.2319; Found: 549.2315. m.p. 221-223 ℃.



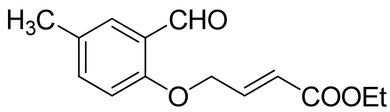
6d: White solid, yield 63%; $[\alpha]_D^{20}$ = +31.7° (c = 0.20, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 10.15 (s, 1 H), 8.41 (s, 2 H), 8.00 (s, 1 H), 7.39-7.10 (m, 14 H), 6.59 (d, *J* = 3.3 Hz, 1 H), 5.79 (d, *J* = 3.3 Hz, 1 H), 5.13 (d, *J* = 17.7 Hz, 1 H), 4.53 (d, *J* = 17.7 Hz, 1 H), 2.39 (s, 3 H); ^{19}F NMR (282 MHz, $CDCl_3$): δ -63.5, -150.1; ^{13}C NMR (75 MHz, $CDCl_3$) δ 150.4, 144.8, 141.5, 135.8, 135.3, 134.3, 134.1, 133.8, 133.1, 130.1, 130.0, 129.8, 129.5, 129.4, 127.5, 127.2, 126.9, 121.95, 121.92, 62.9, 61.6, 38.1, 21.5; IR (thin film): ν_{max} (cm^{-1}) = 3068, 2922, 1596, 1538, 1455, 1367, 1281, 1144, 1074, 898, 814, 756, 698; MS (ESI, *m/z*, rel. intensity) 643.2 (M-BF_4); HRMS (ESI) calcd for $C_{32}H_{25}F_6N_4O_2S$ (M-BF_4): 643.1597; Found: 643.1597. m.p. 162-163 ℃.

The substrates were prepared according to literatures precedent.[3-6] All spectral data was matched with literature description.



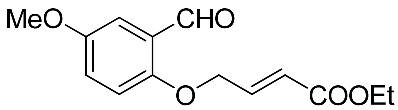
9a[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.57 (s, 1H), 7.88 (d, *J* = 7.5 Hz, 1H), 7.56 (dd, *J* = 6.9, 8.7 Hz, 1H), 7.15-7.06 (m, 2H), 6.95 (d, *J* = 8.4 Hz, 1H), 6.22 (d, *J* = 15.9 Hz, 1H), 4.84 (dd, *J* = 2.1, 3.9 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H).



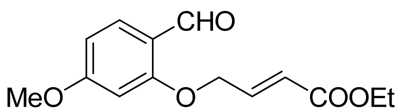
9b[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.53 (s, 1H), 7.67 (s, 1H), 7.35 (d, *J* = 8.4 Hz, 1H), 7.10 (dt, *J* = 4.2, 15.6 Hz, 1H), 6.84 (d, *J* = 8.7 Hz, 1H), 6.21 (dd, *J* = 2.1, 15.9 Hz, 2H), 4.81 (dd, *J* = 1.8, 3.6 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 2.33 (s, 3H) , 1.31 (t, *J* = 7.2 Hz, 3H).



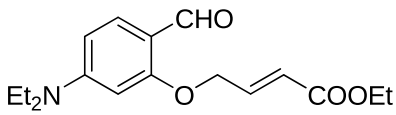
9c[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.53 (s, 1H), 7.36 (d, *J* = 3.0 Hz, 1H), 7.12-7.06 (m, 2H), 6.90 (d, *J* = 9.3 Hz, 1H), 6.20 (dt, *J* = 2.1, 15.6 Hz, 1H), 4.79 (dd, *J* = 2.1, 3.9 Hz, 2H), 4.23 (q, *J* = 6.9 Hz, 2H), 3.82 (s, 3H), 1.31 (t, *J* = 6.9 Hz, 3H).



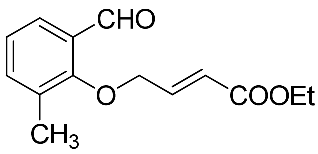
9d[5]

Yellow solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.33 (s, 1H), 7.85 (d, *J* = 9.0 Hz, 1H), 6.68 (dd, *J* = 1.5, 8.7 Hz, 1H), 6.57 (d, *J* = 5.7 Hz, 1H), 6.52 (d, *J* = 2.1 Hz, 1H), 5.25 (dd, *J* = 6.3, 12.9 Hz, 1H), 4.17 (q, *J* = 7.2 Hz, 2H), 3.87 (s, 3H), 3.31 (d, *J* = 7.2 Hz, 2H), 1.28 (t, *J* = 7.2 Hz, 3H).



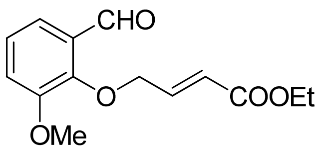
9e[5]

Orange solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.22 (s, 1H), 7.73 (d, *J* = 9.0 Hz, 1H), 7.11 (dt, *J* = 3.9, 15.9 Hz, 1H), 6.31 (d, *J* = 10.2 Hz, 1H), 6.25 (d, *J* = 15.9 Hz, 1H), 5.97 (d, *J* = 1.8 Hz, 1H), 4.79 (dd, *J* = 2.1, 6.9 Hz, 2H), 4.22 (q, *J* = 7.2 Hz, 2H), 3.41 (q, *J* = 7.2 Hz, 4H), 1.31 (t, *J* = 7.2 Hz, 3H), 1.21 (t, *J* = 7.2 Hz, 6H).



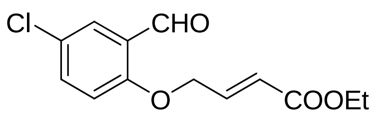
9f[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.33 (s, 1H), 7.70 (d, *J* = 7.5 Hz, 1H), 7.48 (d, *J* = 7.2 Hz, 1H), 7.19 (t, *J* = 7.5 Hz, 1H), 7.09 (dt, *J* = 4.2, 15.6 Hz, 1H), 6.34 (d, *J* = 15.6 Hz, 1H), 4.62 (dd, *J* = 1.8, 3.9 Hz, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 2.33 (s, 3H) , 1.33 (t, *J* = 7.2 Hz, 3H).



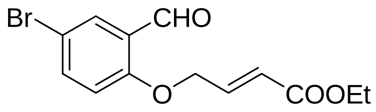
9g[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.44 (s, 1H), 7.44 (dd, *J* = 4.2, 4.8 Hz, 1H), 7.17 (d, *J* = 4.5 Hz, 2H), 7.09 (dt, *J* = 4.5, 15.9 Hz, 1H), 6.24 (dt, *J* = 1.5, 15.6 Hz, 1H), 4.83 (dd, *J* = 1.5, 4.2 Hz, 2H), 4.23 (q, *J* = 6.9 Hz, 2H), 3.91 (s, 3H), 1.31 (t, *J* = 6.9 Hz, 3H).



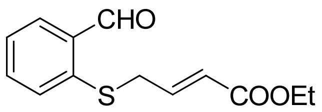
9h[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.49 (s, 1H), 7.82 (d, *J* = 2.4 Hz, 1H), 7.49 (dd, *J* = 2.4, 9.0 Hz, 1H), 7.08 (dt, *J* = 4.5, 15.9 Hz, 1H), 6.91 (d, *J* = 9.0 Hz, 1H), 6.19 (dt, *J* = 1.8, 15.9 Hz, 1H), 4.83 (dd, *J* = 1.8, 3.9 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.31 (t, *J* = 7.2 Hz, 3H).



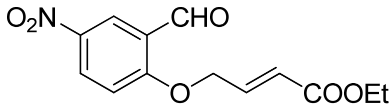
9i[5]

White solid. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.47 (s, 1H), 7.96 (d, *J* = 2.4 Hz, 1H), 7.63 (dd, *J* = 2.4, 8.7 Hz, 1H), 7.09 (dt, *J* = 4.5, 15.3 Hz, 1H), 6.85 (d, *J* = 8.7 Hz, 1H), 6.19 (d, *J* = 15.6 Hz, 1H), 4.83 (dd, *J* = 2.1, 4.2 Hz, 2H), 4.23 (q, *J* = 7.2 Hz, 2H), 1.32 (t, *J* = 7.2 Hz, 3H).



9j

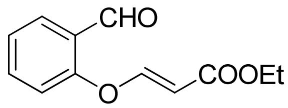
Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.31 (s, 1H), 7.83 (d, *J* = 7.5 Hz, 1H), 7.52 (t, *J* = 8.1 Hz, 1H), 7.39-7.31 (m, 2H), 6.97 (dt, *J* = 6.9, 15.6 Hz, 1H), 5.94 (d, *J* = 15.6 Hz, 1H), 4.15 (q, *J* = 7.2 Hz, 2H), 3.70 (d, *J* = 6.6 Hz, 2H), 1.25 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 191.1, 165.3, 141.4, 139.3, 133.8, 133.7, 132.3, 128.3, 125.7, 123.9, 60.2, 33.9, 13.9. IR (thin film): ν_{max} (cm^{-1}) = 3061, 2981, 2848, 2742, 1716, 1652, 1587, 1461, 1368, 1317, 1267, 1198, 1041, 978, 754, 679; MS (EI, *m/z*, rel. intensity) 250 (M^+, 2), 137 (100); HRMS (EI) calcd for $C_{13}H_{14}O_3S$ (M^+): 250.0664. Found: 250.0663.



9k

White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 10.53 (s, 1H), 8.71 (d, *J* = 2.8 Hz, 1H), 8.43 (dd, *J* = 3.2, 9.2 Hz, 1H), 7.13 (t, *J* = 4.4 Hz, 1H), 7.11-7.08 (m, 1H), 6.22 (dt, *J* = 2.0, 16.0 Hz, 1H), 5.00 (dd, *J* = 2.0, 4.4 Hz, 2H), 4.25 (q, *J* = 7.2 Hz, 2H), 1.32 (t, *J*

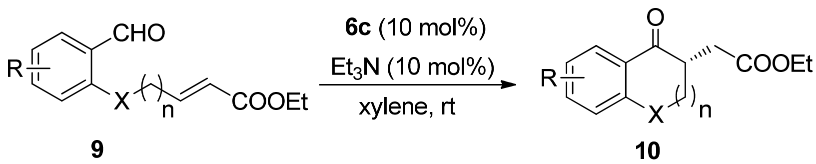
= 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 186.9, 165.3, 163.7, 142.0, 139.3, 130.5, 124.9, 124.8, 123.5, 113.1, 67.8, 60.9, 14.1. IR (thin film): ν_{max} (cm^{-1}) = 3080, 2966, 2903, 1694, 1588, 1524, 1368, 1342, 1273, 1193, 1025, 945, 749, 676; MS (EI, *m/z*, rel. intensity) 233 (24), 206 (33), 85 (100); HRMS (EI) calcd for $C_{13}H_{13}NO_6$ (M^+): 279.0743. Found: 279.0747; m.p. 71-73 °C.



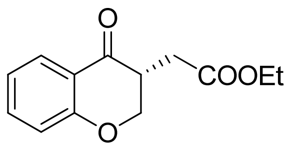
9l[6]

Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 10.37 (s, 1H), 7.93 (dd, *J* = 1.8, 7.8 Hz, 1H), 7.85 (d, *J* = 12.3 Hz, 1H), 7.69-7.63 (m, 1H), 7.33 (t, *J* = 7.5 Hz, 1H), 7.17 (d, *J* = 8.7 Hz, 1H), 5.64 (d, *J* = 12.0 Hz, 1H), 4.22 (q, *J* = 7.2 Hz, 2H), 1.30 (t, *J* = 7.2 Hz, 3H).

General Procedure for Enantioselective Intramolecular Stetter Reactions:

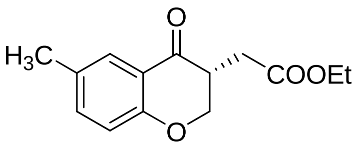


A flame dried Schlenk tube was cooled to room temperature and filled with argon. To this tube were added triazolium salt **6c** (6.4 mg, 0.01 mmol, 10 mol%) and xylene (1.0 mL). Then to this solution was added Et_3N (1.4 μL, 0.01 mmol, 10 mol%), the solution was stirred at room temperature for 30 minutes. The substrate **9** (0.1 mmol) was then added. After the reaction was complete (monitored by TLC), the reaction mixture was cooled to 0 ℃ immediately, quenched with 5 mL H_2O, The organic layer was separated, and the aqueous layer was extracted three times with ethyl acetate. The combined organic layers were dried over Na_2SO_4, filtered, and concentrated. The residue was purified by silica gel column chromatography (*n*-Hexane/EtOAc = 10/1) to afford the product **10**.



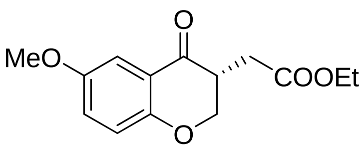
10a[5]

Colorless oil, 95% yield, 93% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 97/3, v = 0.7 mL·min^{-1}, λ = 254 nm, t (major) = 23.1 min, t (minor) = 34.9 min]; $[\alpha]_D^{20}$ = -6.8 (c = 1.0, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.89 (dd, J = 1.8, 7.8 Hz, 1H), 7.51-7.45 (m, 1H), 7.05-6.96 (m, 2H), 4.60 (dd, J = 5.4, 11.1 Hz, 1H), 4.30 (t, J = 11.7 Hz, 1H), 4.19 (q, J = 7.2 Hz, 2H), 3.39-3.29 (m, 1H), 2.94 (dd, J = 4.8, 17.1 Hz, 1H), 2.42 (dd, J = 8.1, 17.1 Hz, 1H), 1.28 (t, J = 7.2 Hz, 3H).



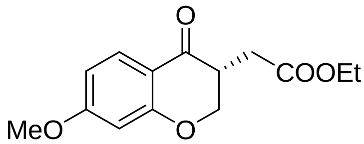
10b[5]

Colorless oil, 98% yield, 95% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 95/5, v = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 13.4 min, t (minor) = 20.8 min]; $[\alpha]_D^{20}$ = -20.5 (c = 1.0, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.67 (s, J = 1H), 7.28 (d, J = 8.1 Hz, 1H), 6.87 (d, J = 8.4 Hz, 1H), 4.57 (dd, J = 5.1, 11.1 Hz, 1H), 4.17-4.26 (m, 3H), 3.35-3.25 (m, 1H), 2.92 (dd, J = 4.8, 16.8 Hz, 1H), 2.41 (dd, J = 8.7, 17.1 Hz, 1H), 2.30 (s, 3H), 1.28 (t, J = 7.2 Hz, 3H).



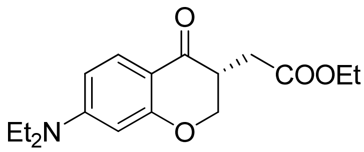
10c[5]

Yellow oil, 95% yield, 88% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 95/5, v = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 19.9 min, t (minor) = 28.6 min]; $[\alpha]_D^{20}$ = -24.2 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.31 (d, J = 3.0 Hz, 1H), 7.08 (dd, 3.3, 9.0 Hz, 1H), 6.91 (d, J = 9.0 Hz, 1H), 4.56 (dd, J = 5.1, 11.1 Hz, 1H), 4.30-4.16 (m, 3H), 3.77 (s, 3H), 3.36-3.26 (m, 1H), 2.90 (dd, *J* = 4.5, 16.8 Hz, 1H), 2.43 (dd, *J* = 8.1, 17.1 Hz, 1H), 1.27 (t, *J* = 7.2 Hz, 3H).



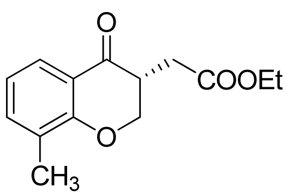
10d[5]

Yellow oil, 87% yield, 97% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 95/5, *v* = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 21.4 min, t (minor) = 27.5 min]; $[\alpha]_D^{20}$ = -10.8 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.82 (d, *J* = 8.7 Hz, 1H), 6.59 (dd, *J* = 2.4, 8.7 Hz, 1H), 6.41 (d, *J* = 2.4 Hz, 1H), 4.59 (dd, *J* = 5.4, 11.1 Hz, 1H), 4.28 (t, *J* = 11.4 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.83 (s, 3H), 3.31-3.24 (m, 1H), 2.94 (dd, *J* = 4.8, 17.1Hz, 1H), 2.39 (dd, *J* = 8.4, 16.8 Hz, 1H), 1.29 (t, *J* = 7.2 Hz, 3H).



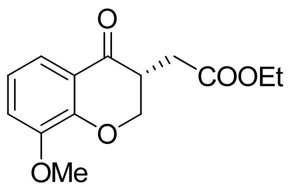
10e[5]

Yellow oil, 56% yield, 95% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 90/10, *v* = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 18.9 min, t (minor) = 20.7 min]; $[\alpha]_D^{20}$ = +9.5 (c = 1.0, $CHCl_3$). ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 9.0 Hz, 1H), 6.34 (dd, *J* = 2.4, 9.0 Hz, 1H), 6.04 (d, *J* = 2.4 Hz, 1H), 4.52 (dd, *J* = 5.1, 11.1 Hz, 1H), 4.26-4.14 (m, 3H), 3.38 (q, *J* = 7.2 Hz, 4H), 3.23-3.15 (m, 1H), 2.95 (dd, *J* = 4.8, 17.1 Hz, 1H), 2.35 (dd, *J* = 8.7, 16.8 Hz, 1H), 1.28 (t, *J* = 7.2 Hz, 3H), 1.20 (t, *J* = 7.2 Hz, 6H).



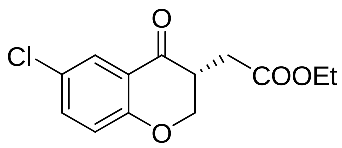
10f[5]

Yellow oil, 98% yield, 80% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 97/3, *v* = 0.7 mL·min^{-1}, λ = 254 nm, t (major) = 17.9 min, t (minor) = 24.0 min]; $[\alpha]_D^{20}$ = -11.2 (c = 1.0, CH_2Cl_2). ^1H NMR (300 MHz, $CDCl_3$) δ 7.74 (d, *J* = 7.5 Hz, 1H), 7.34 (d, *J* = 7.2 Hz, 1H), 6.92 (t, *J* = 7.5 Hz, 1H), 4.64 (dd, *J* = 5.4, 10.8 Hz, 1H), 4.29 (t, *J* = 12.0 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.35-3.28 (m, 1H), 2.94 (dd, *J* = 4.5, 17.1 Hz, 1H), 2.40 (dd, *J* = 8.1, 16.8 Hz, 1H), 2.24 (s, 3H), 1.29 (t, *J* = 7.2 Hz, 3H).



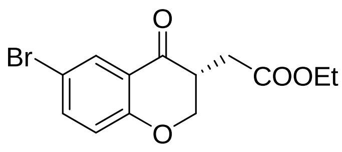
10g[5]

Yellow oil, 98% yield, 81% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 90/10, *v* = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 16.2 min, t (minor) = 22.1 min]; $[\alpha]_D^{20}$ = -26.1 (c = 1.0, CH_2Cl_2). ^1H NMR (300 MHz, $CDCl_3$) δ 7.47 (dd, *J* = 1.5, 7.8 Hz, 1H), 7.04 (dd, *J* = 1.5, 7.8 Hz, 1H), 6.95 (t, *J* = 8.1 Hz, 1H), 4.69 (dd, *J* = 5.4, 11.4 Hz, 1H), 4.35 (t, *J* = 11.1 Hz, 1H), 4.16 (q, *J* = 7.2 Hz, 2H), 3.89 (s, 3H), 3.38-3.28 (m, 1H), 2.91 (dd, *J* = 4.8, 17.1 Hz, 1H), 2.45 (dd, *J* = 8.7, 16.8 Hz, 1H), 1.28 (t, *J* = 7.2 Hz, 3H).



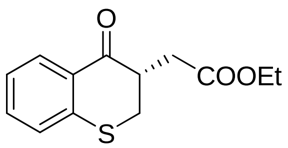
10h[5]

Yellow oil, 98% yield, 78% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 95/5, *v* = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 14.8 min, t (minor) = 21.6 min]; $[\alpha]_D^{20}$ = -13.7 (c = 1.1, $CHCl_3$). ^1H NMR (300 MHz, $CDCl_3$) δ 7.83 (d, *J* = 2.7 Hz, 1H), 7.39-7.43 (m, 1H), 6.94 (d, *J* = 9.0 Hz, 1H), 4.61 (dd, *J* = 5.4, 11.4 Hz, 1H), 4.30 (t, *J* = 11.7 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.36-3.26 (m, 1H), 2.92 (dd, *J* = 4.8, 17.1 Hz, 1H), 2.43 (dd, *J* = 8.1, 17.4 Hz, 1H), 1.28 (t, *J* = 7.2 Hz, 3H).



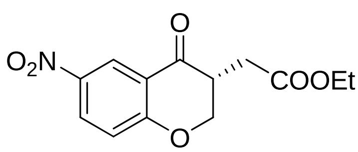
10i[5]

Yellow oil, 93% yield, 78% ee [Daicel Chiralcel OD-H, hexane/2-propanol = 97/3, *v* = 0.5 mL·min^{-1}, λ = 254 nm, t (minor) = 29.1 min, t (major) = 32.1 min]; $[\alpha]_D^{20}$ = -12.9 (c = 1.0, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.98 (d, *J* = 2.1 Hz, 1H), 7.54 (dd, *J* = 2.7, 9.0 Hz, 1H), 6.88 (d, *J* = 8.7 Hz, 1H), 4.60 (dd, *J* = 5.4, 11.1 Hz, 1H), 4.29 (t, *J* = 11.4 Hz, 1H), 4.18 (q, *J* = 7.2 Hz, 2H), 3.36-3.26 (m, 1H), 2.91 (dd, *J* = 4.5, 17.1 Hz, 1H), 2.43 (dd, *J* = 8.1, 17.1 Hz, 1H), 1.26 (t, *J* = 7.2 Hz, 3H).



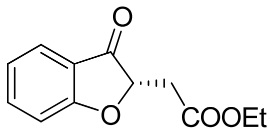
10j

Yellow oil, 70% yield, 89% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 97/3, *v* = 0.7 mL·min^{-1}, λ = 254 nm, t (major) = 21.0 min, t (minor) = 25.6 min]; $[\alpha]_D^{20}$ = -62.2 (c = 1.0, CH_2Cl_2). ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.09 (d, *J* = 8.1 Hz, 1H), 7.37 (t, *J* = 7.2 Hz, 1H), 7.26 (d, *J* = 6.6 Hz, 1H), 7.20 (t, *J* = 7.5 Hz, 1H), 4.19 (q, *J* = 7.2 Hz, 2H), 3.37-3.34 (m, 2H), 3.13-3.10 (m, 1H), 2.98 (dd, *J* = 5.1, 16.8 Hz, 1H), 2.60 (dd, *J* = 6.9, 16.8 Hz, 1H), 1.29 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.5, 171.6, 141.7, 133.2, 130.3, 129.6, 127.3, 124.9, 60.8, 44.4, 34.3, 30.8, 14.1. IR (thin film): ν_{max} (cm^{-1}) = 2980, 1732, 1681, 1588, 1437, 1177, 1028, 765, 737; MS (EI, *m/z*, rel. intensity) 250 (M^+, 5), 163 (100); HRMS (EI) calcd for $C_{13}H_{14}O_3S$ (M^+): 250.0664. Found: 250.0662.



10k

White solid, 90% yield, 0% ee [Daicel Chiralpak AD-H, hexane/2-propanol = 70/30, v = 1.0 mL·min^{-1}, λ = 254 nm, t (major) = 12.7 min, t (minor) = 20.3 min]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.78 (d, J = 2.7 Hz, 1H), 8.34 (dd, J = 2.7, 9.0 Hz, 1H), 7.13 (d, J = 9.0 Hz, 1H), 4.76 (dd, J = 5.4, 11.4 Hz, 1H), 4.44 (t, J = 11.7 Hz, 1H), 4.20 (q, J = 7.2 Hz, 1H), 3.45-3.35 (m, 1H), 2.96 (dd, J = 4.8, 17.1 Hz, 1H), 2.51 (dd, J = 7.5, 17.1 Hz, 1H), 1.30 (t, J = 7.2 Hz, 1H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 190.6, 170.8, 165.6, 142.1, 130.3, 123.9, 120.0, 119.2, 70.6, 61.2, 42.1, 29.8, 14.1. IR (thin film): ν_{max} (cm^{-1}) = 2986, 1731, 1692, 1439, 1339, 1012, 856, 750, 622; MS (EI, *m/z*, rel. intensity) 279 (M^+, 1), 192 (100); HRMS (EI) calcd for $C_{13}H_{13}NO_6$ (M^+): 279.0743. Found: 279.0745; m.p. 86-88 ℃.



10l

Yellow oil, 59% yield, 0% ee [Daicel Chiralcel OB-H, hexane/2-propanol = 90/10, v = 1.0 mL·min^{-1}, λ = 254 nm, t (minor) = 21.3 min, t (major) = 37.9 min]. ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69 (d, J = 7.5 Hz, 1H), 7.62 (t, J = 7.5 Hz, 1H), 7.15-7.08 (m, 2H), 4.89 (dd, J = 3.9, 7.5 Hz, 1H), 4.18-4.11 (m, 2H), 3.09 (dd, J = 3.6, 17.1 Hz, 1H), 2.83 (dd, J = 7.5, 16.8 Hz, 1H), 1.20 (t, J = 7.2 Hz, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 200.4, 172.4, 169.3, 138.0, 124.2, 122.1, 120.9, 113.5, 81.0, 61.2, 36.0, 13.9. IR (thin film): ν_{max} (cm^{-1}) = 2983, 2935, 1724, 1615, 1464, 1327, 1191, 1026, 891, 760; MS (EI, *m/z*, rel. intensity) 220 (M^+, 28), 147 (100); HRMS (EI) calcd for $C_{12}H_{12}O_4$ (M^+): 220.0736. Found: 220.0739.

References

[1] (a) L. Darko and J. Karliker, *J. Org. Chem.*,1971, **36,** 3810. (b) R. R.R. Taylor, H. C. Twin and R. A. Batey et al., Tetrahedron, 2010, **66**, 3370.
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[4] X. Han, L.-W. Ye, X.-L. Sun and Y. Tang, *J. Org. Chem*., 2009, **74**, 3394.

[5] J. Read de Alaniz, M. S. Kerr, J. L. Moore and T. Rovis, *J. Org. Chem.*, 2008, **73**, 2033.
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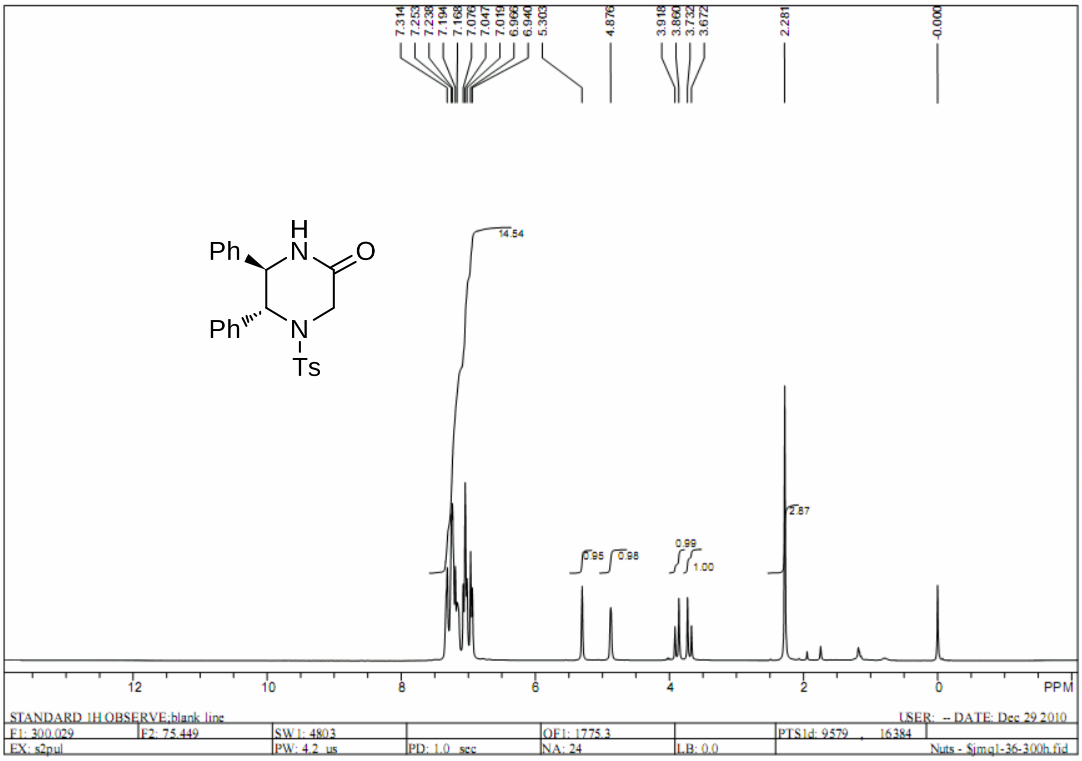

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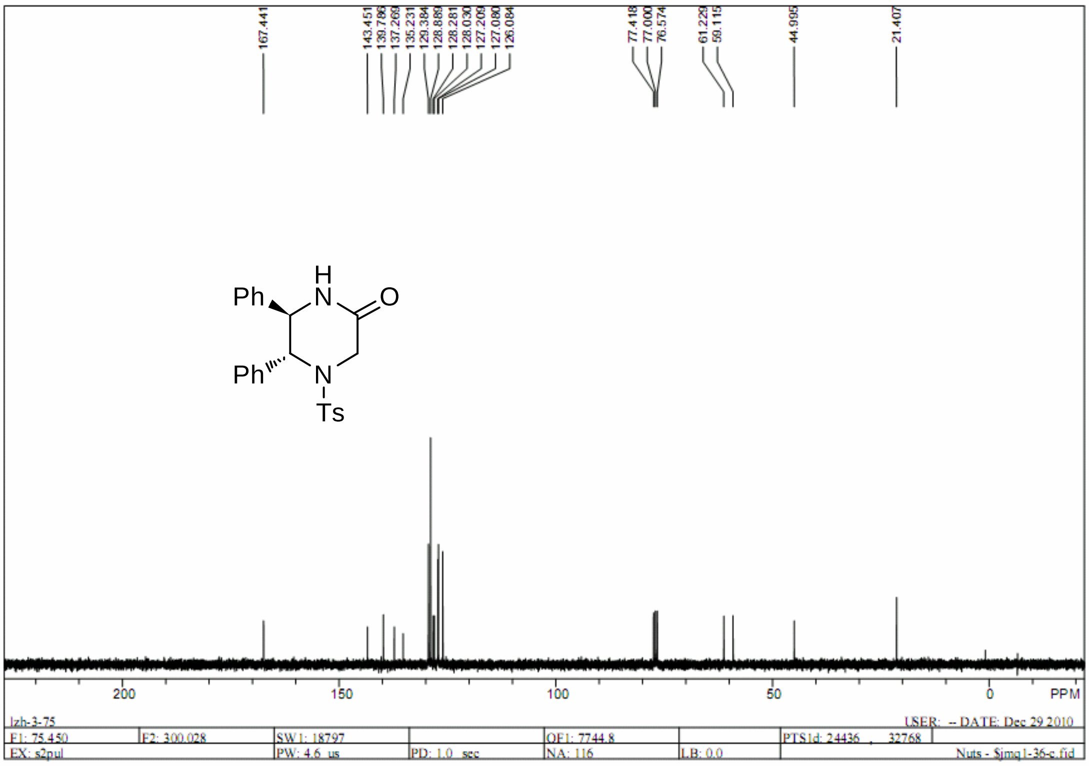

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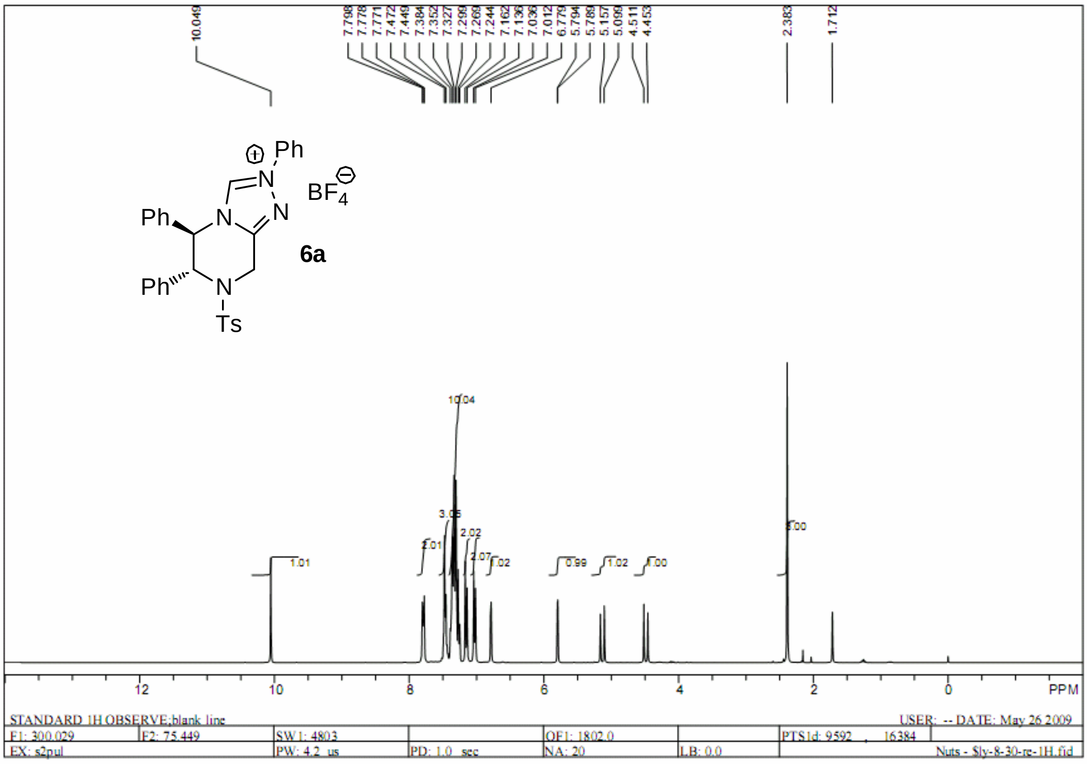
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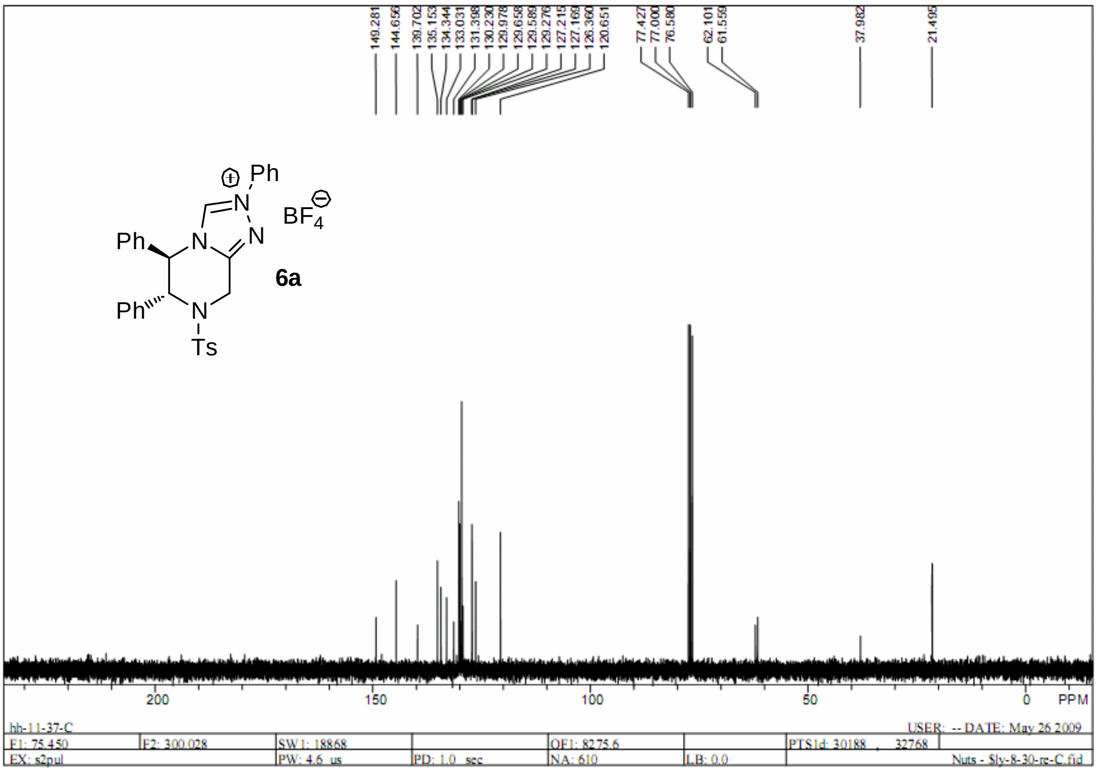
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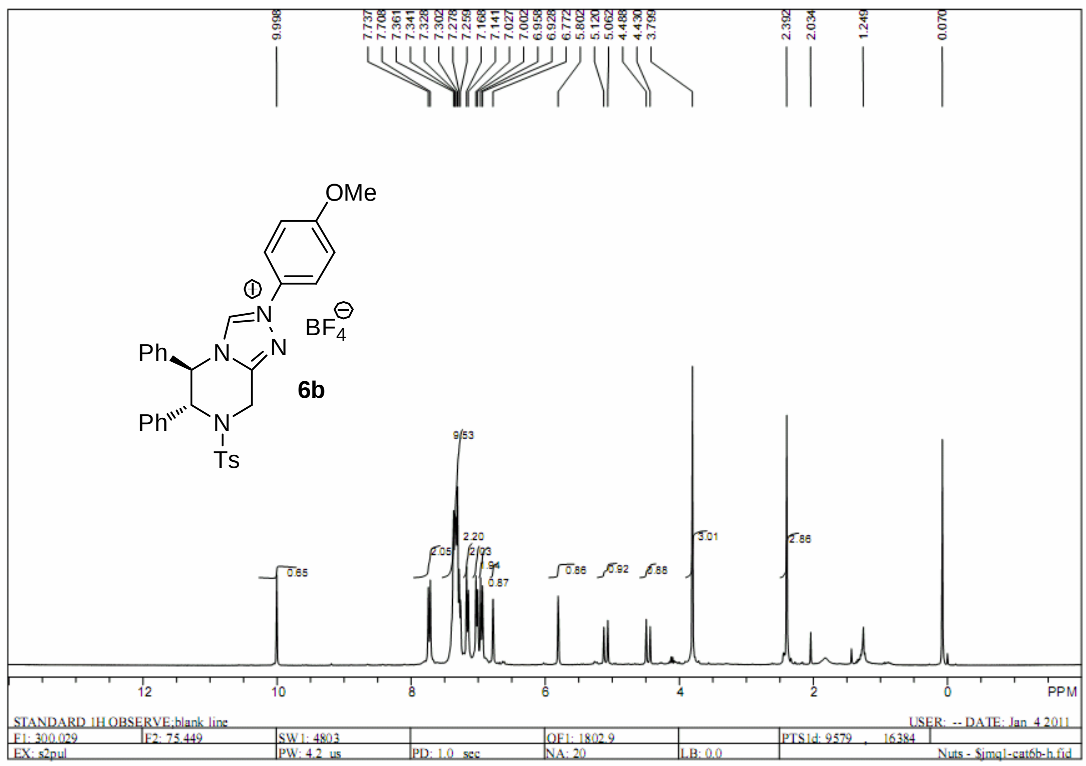
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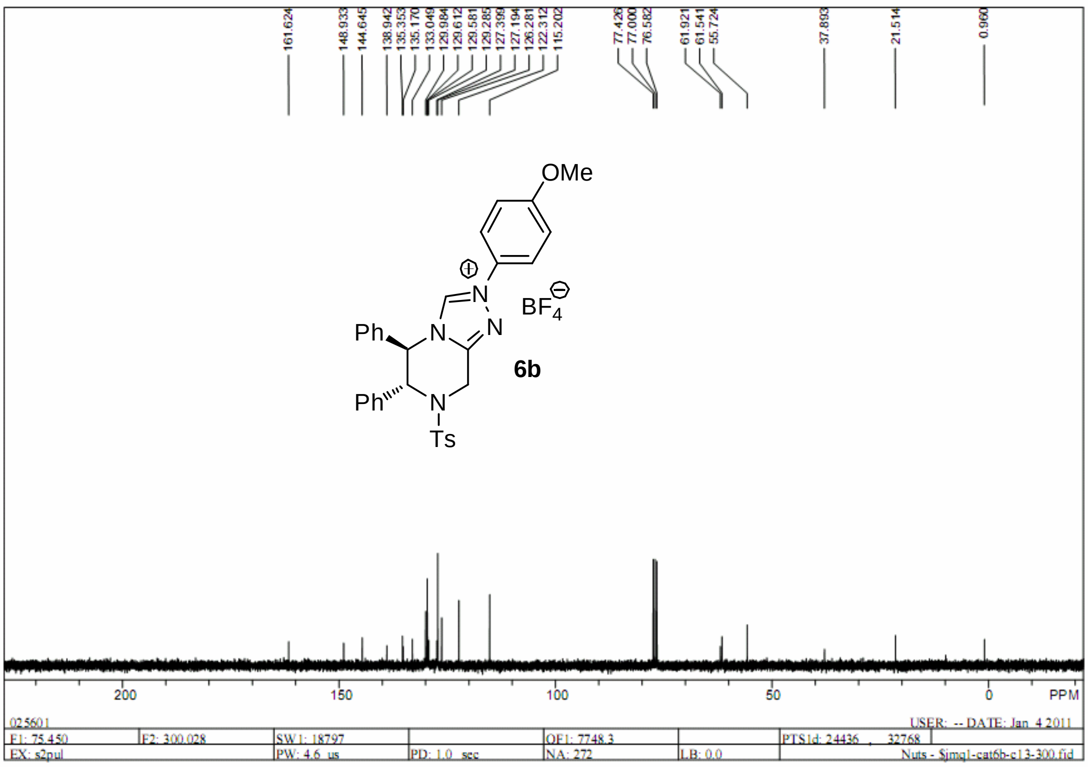
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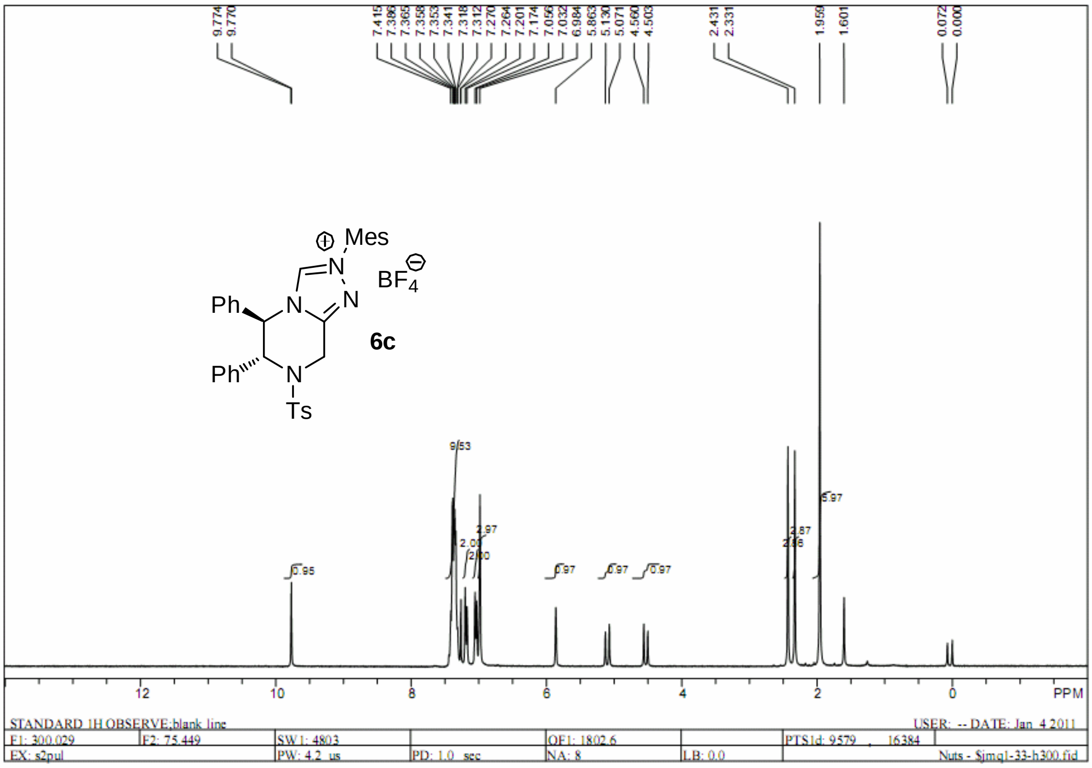

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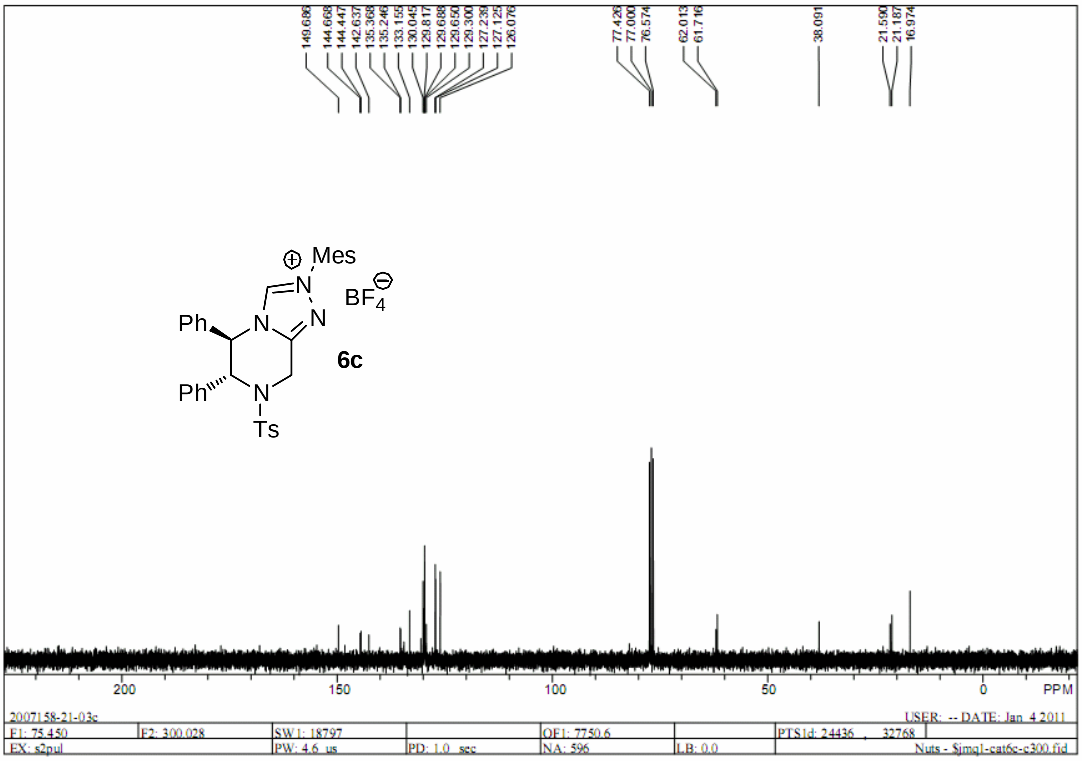
2.331
1.959
1.601
0.072
0.000
Mes
BF4
Ph
N
N
N
Ph
N
Ts
6c
9.53
2.00
2.00
2.97
0.95
0.97
0.97
0.97
2.86
2.87
5.97
12
10
8
6
4
2
0
PPM
STANDARD 1H OBSERVE,blank line
USER: -- DATE: Jan 4 2011
F1: 300.029
F2: 75.449
SW1: 4803
OF1: 1802.6
PTS1d: 9579 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $jmql-33-h300.fid



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144.668
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135.246
133.155
130.045
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129.688
129.650
129.300
127.239
127.125
126.076
77.426
77.000
76.574
62.013
61.716
38.091
21.590
21.187
16.974
Mes
BF4
Ph
N
N
N
Ph
N
Ts
6c
200
150
100
50
0
PPM
2007158-21-03c
USER: -- DATE: Jan 4 2011
F1: 75.450
F2: 300.028
SW1: 18797
OF1: 7750.6
PTS1d: 24436 , 32768
EX: s2pul
PW: 4.6 us
PD: 1.0 sec
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LB: 0.0
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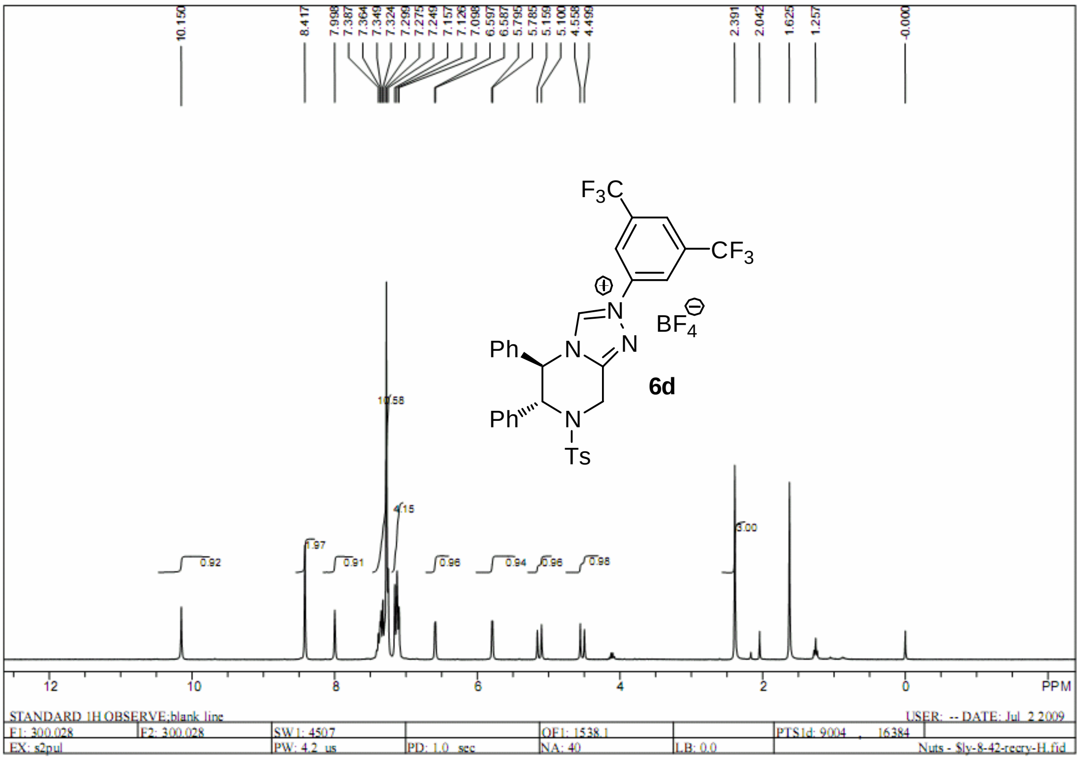

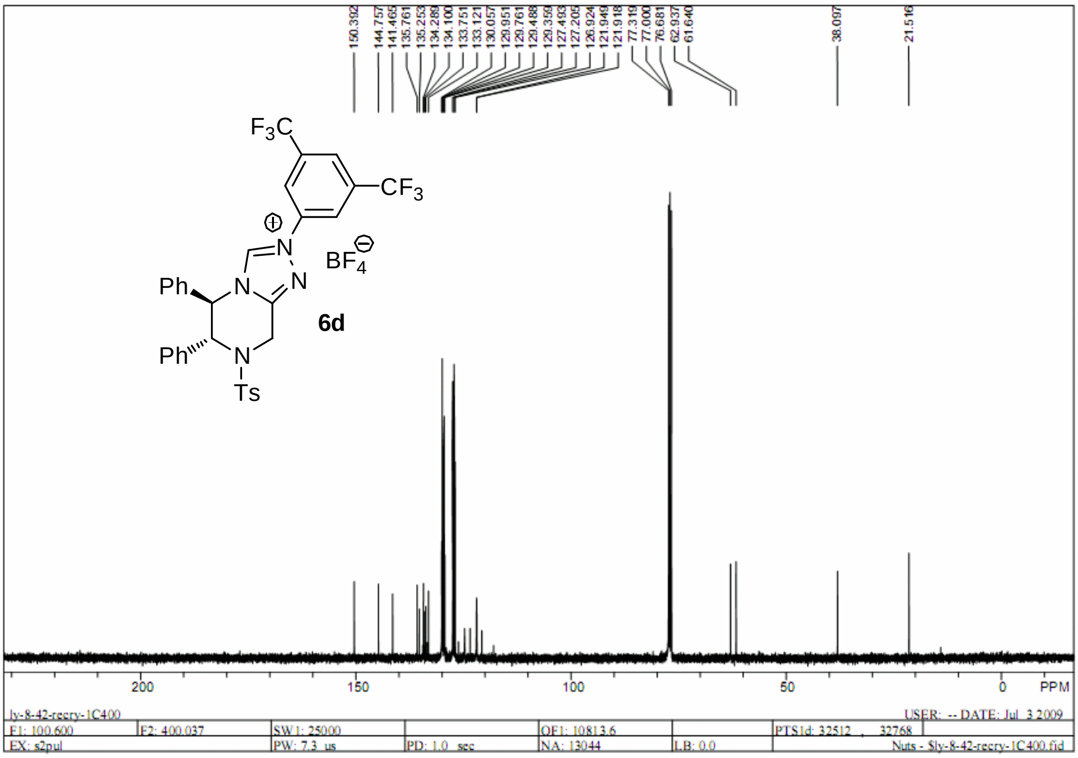

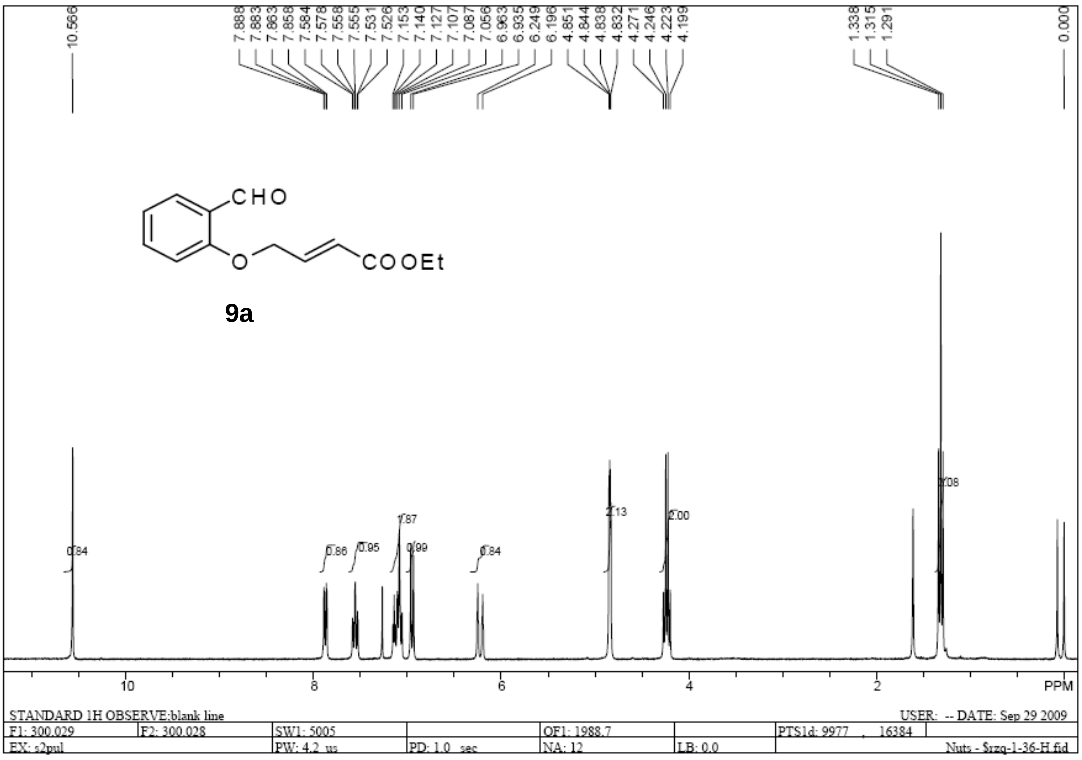
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USER: -- DATE: Sep 29 2009
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SW1: 5005
OF1: 1988.7
PTS1d: 9977 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 12
LB: 0.0
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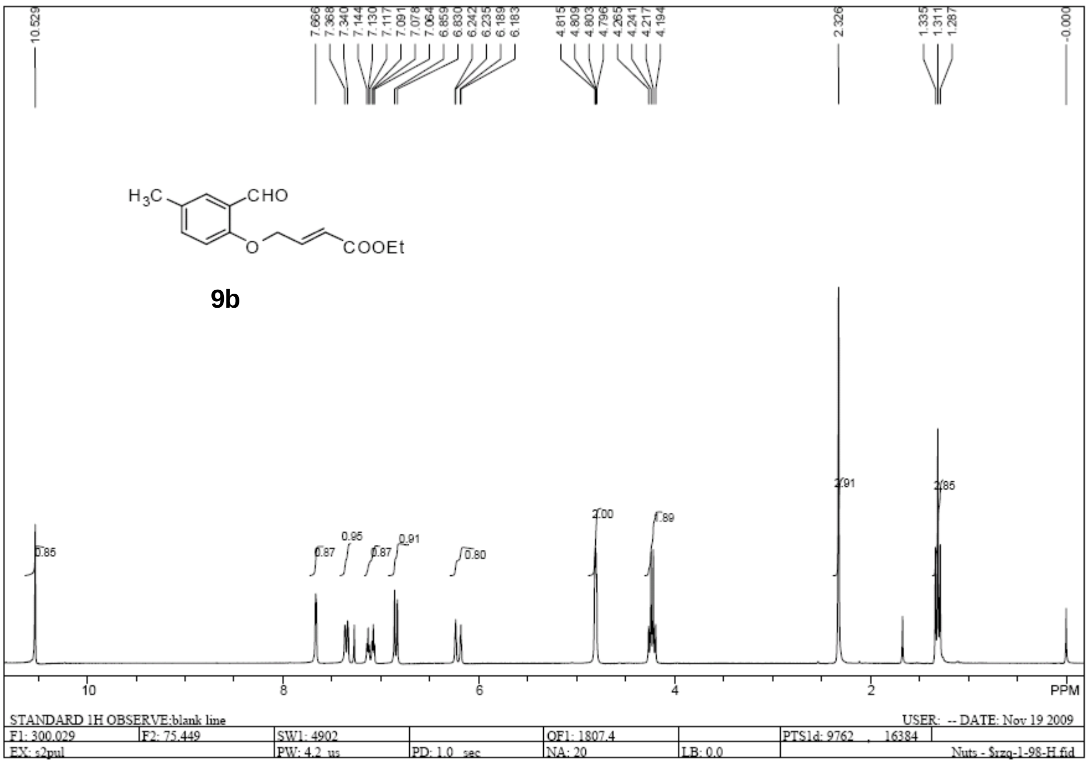
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CHO
COOEt
9b
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Nov 19 2009
F1: 300.029
F2: 75.449
SW1: 4902
OF1: 1807.4
PTS1d: 9762 , 16384
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PW: 4.2 us
PD: 1.0 sec
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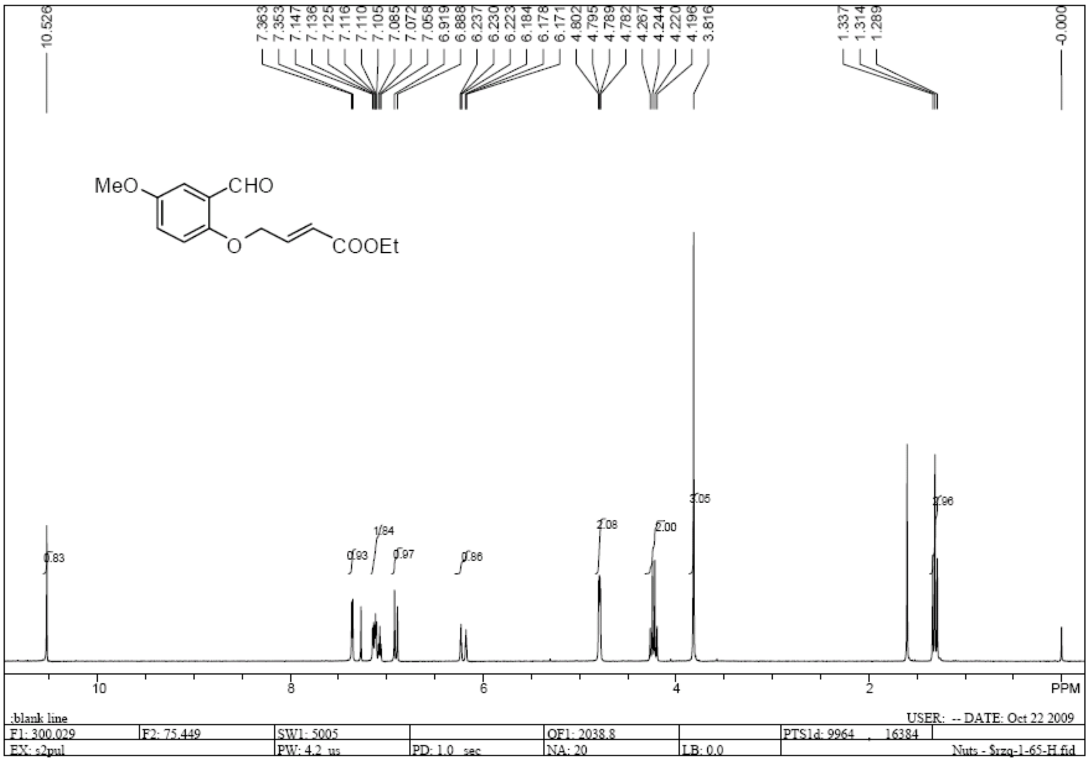
LB: 0.0
Nuts - $rzq-1-98-H.fid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7.072
7.058
6.919
6.888
6.237
6.230
6.223
6.184
6.178
6.171
4.802
4.795
4.789
4.782
4.267
4.244
4.220
4.196
3.816
1.337
1.314
1.289
-0.000
MeO
CHO
O
COOEt
0.83
0.93
1.84
0.97
0.86
2.08
2.00
3.05
2.96
10
8
6
4
2
PPM
;blank line
USER: -- DATE: Oct 22 2009
F1: 300.029
F2: 75.449
SW1: 5005
OF1: 2038.8
PTS1d: 9964 , 16384
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PW: 4.2 us
PD: 1.0 sec
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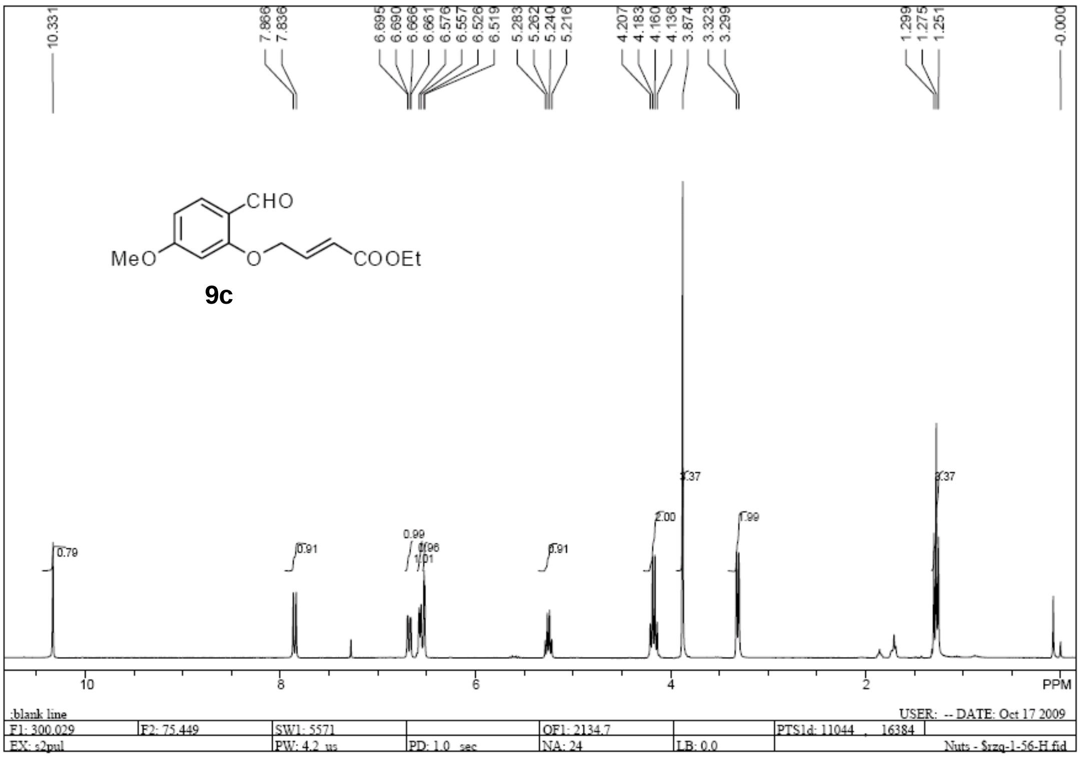
LB: 0.0
Nuts - $rzq-1-65-H.fid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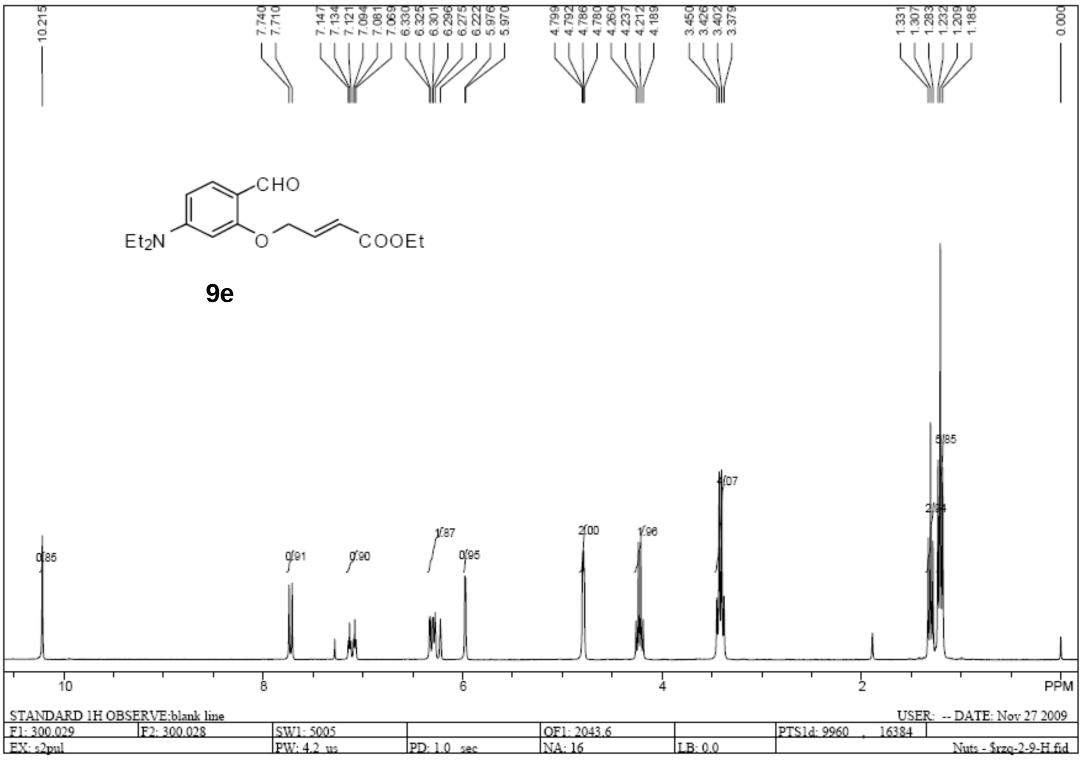
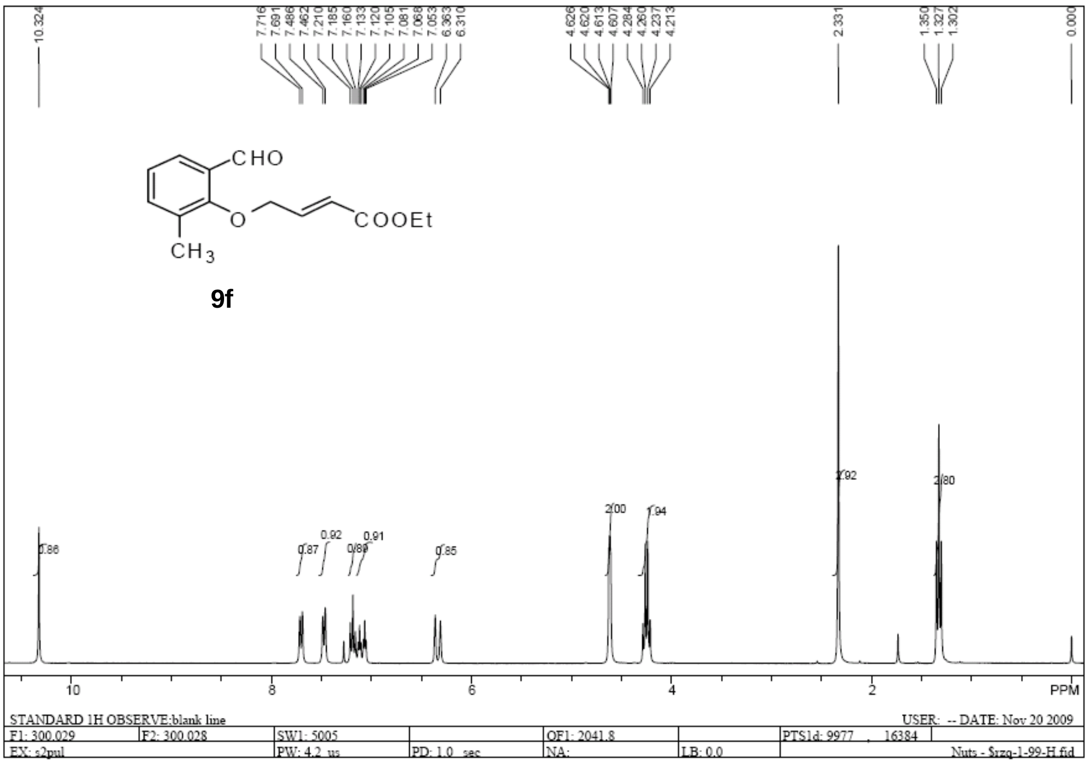
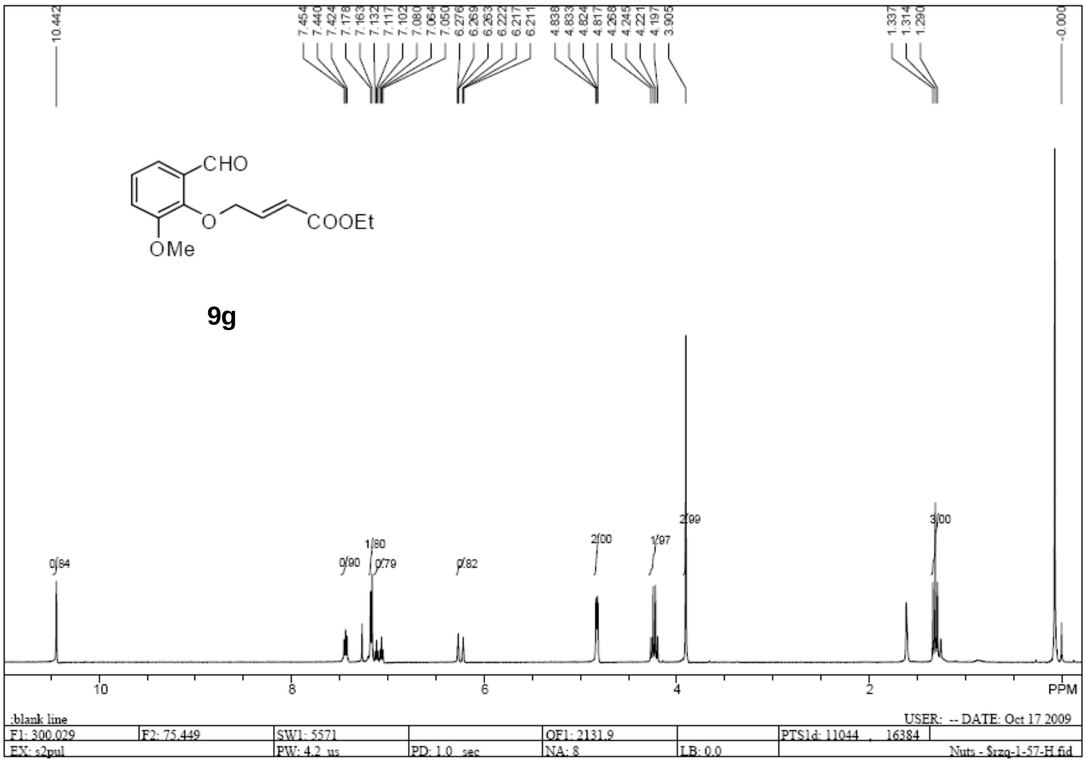
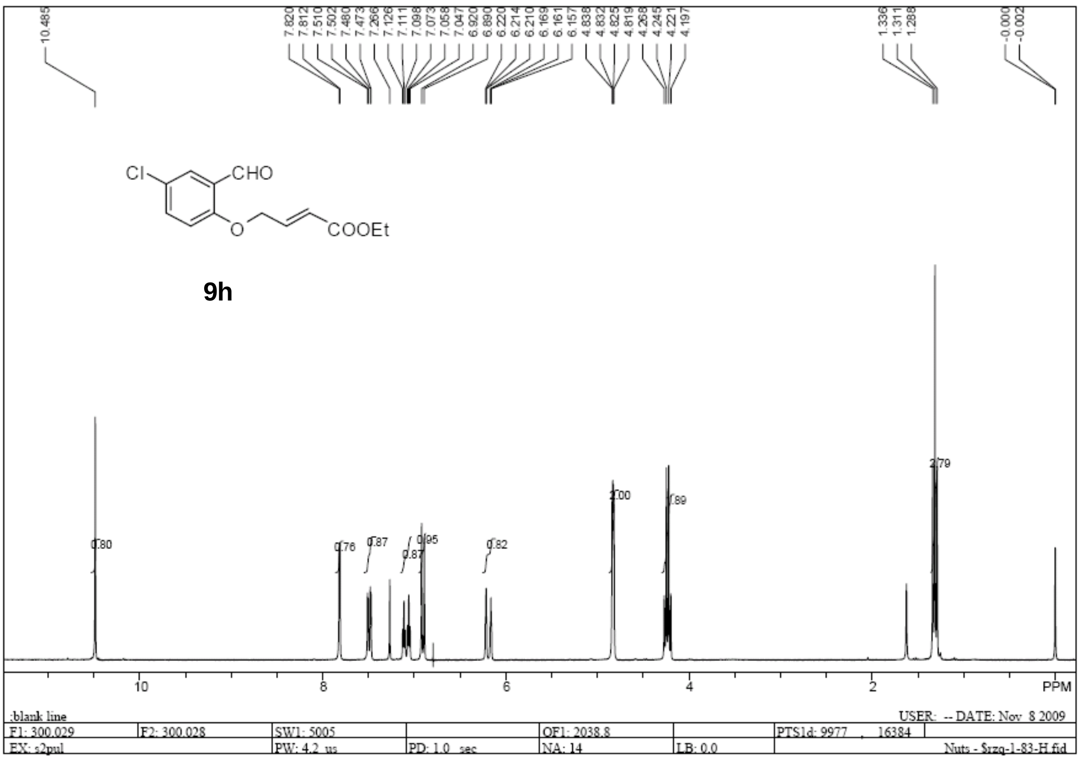
6.661
6.576
6.557
6.526
6.519
5.283
5.262
5.240
5.216
4.207
4.183
4.160
4.136
3.874
3.323
3.299
1.299
1.275
1.251
-0.000
CHO
MeO
O
COOEt
0.79
0.91
0.99
0.96
1.01
0.91
2.00
3.37
1.99
3.37
10
8
6
4
2
PPM
;blank line
USER: -- DATE: Oct 17 2009
F1: 300.029
F2: 75.449
SW1: 5571
OF1: 2134.7
PTS1d: 11044 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 24
LB: 0.0
Nuts - $rzq-1-56-H.fid

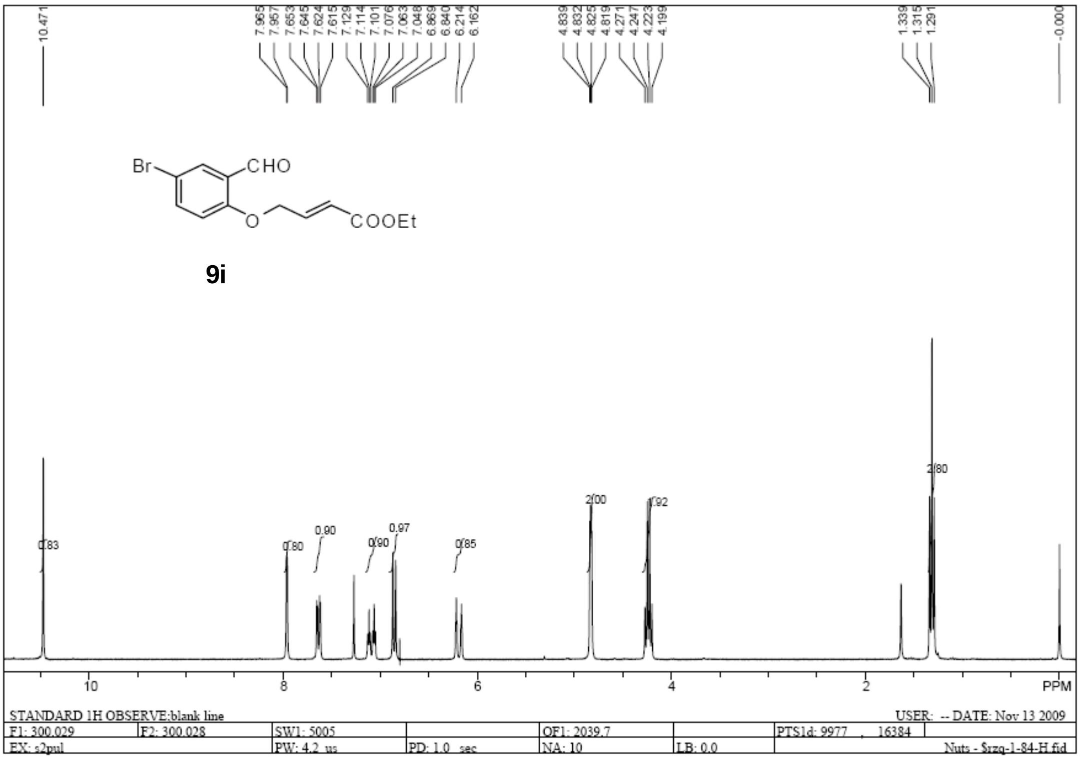
9c

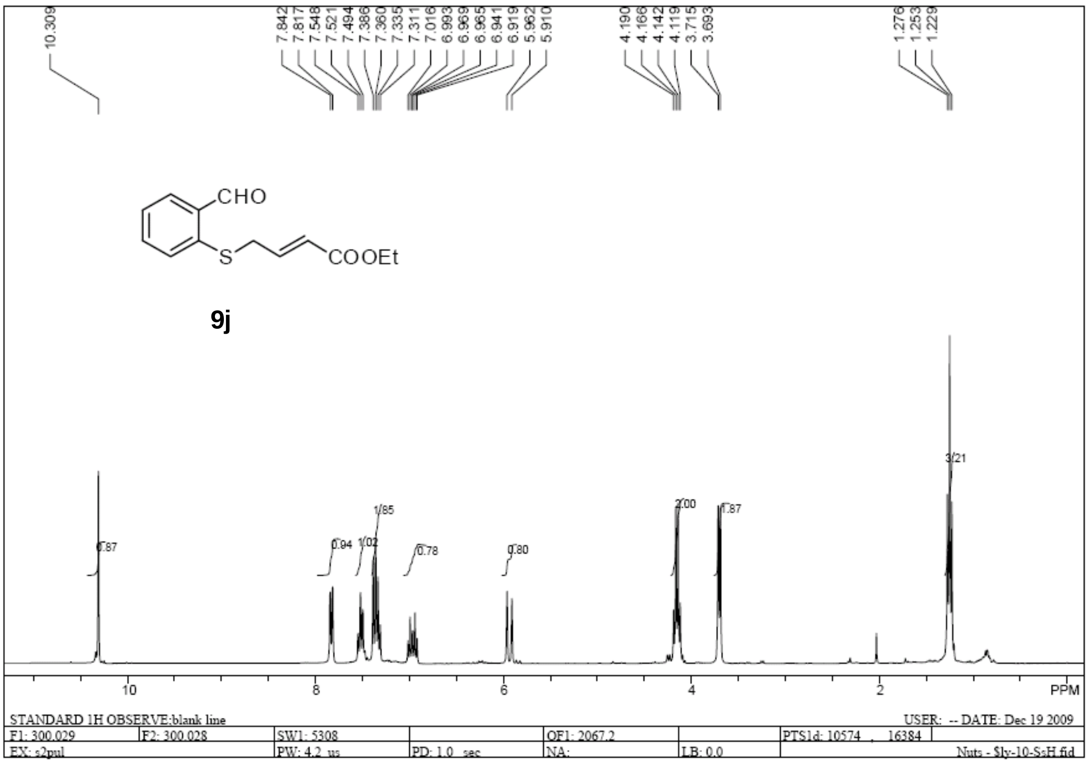


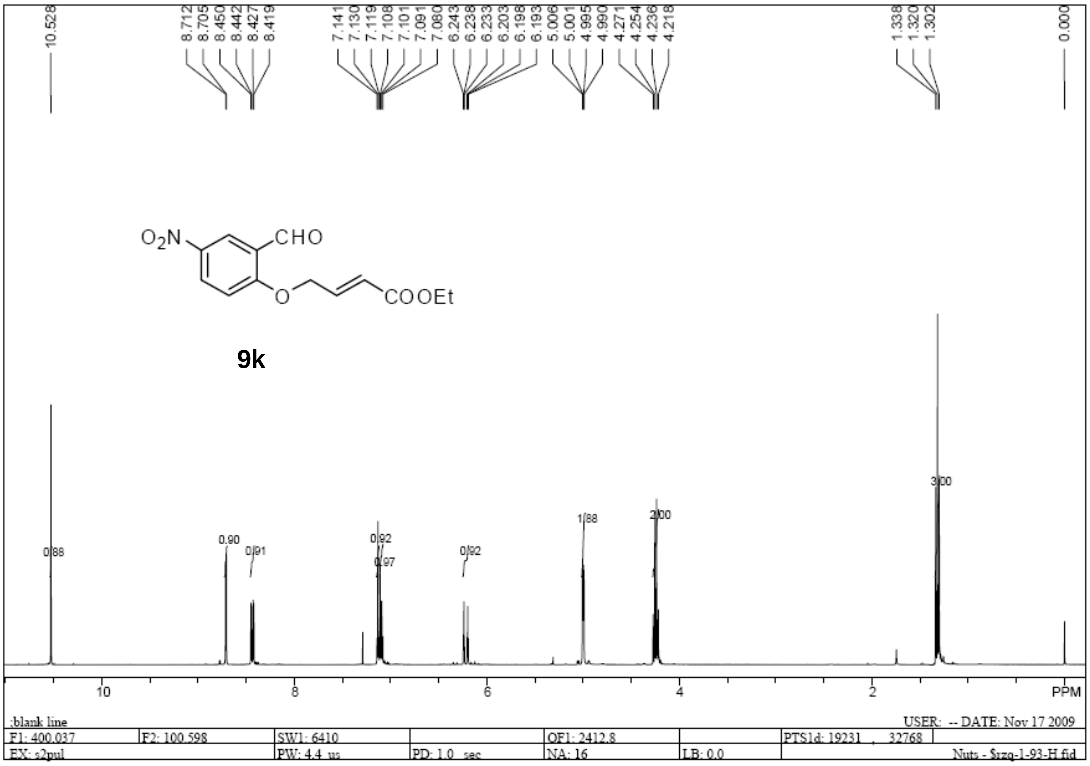



CHO
O
COOEt
OMe
9g
PPM
:blank line
USER: -- DATE: Oct 17 2009
F1: 300.029
F2: 75.449
SW1: 5571
OF1: 2131.9
PTS1d: 11044 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 8
LB: 0.0
Nuts - $rzq-1-57-H.fid


Cl
CHO
O
COOEt
9h
PPM
:blank line
USER: -- DATE: Nov 8 2009
F1: 300.029
F2: 300.028
SW1: 5005
OF1: 2038.8
PTS1d: 9977 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 14
LB: 0.0
Nuts - $rzq-1-83-H.fid


Br
CHO
O
COOEt
9i
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Nov 13 2009
F1: 300.029
F2: 300.028
SW1: 5005
OF1: 2039.7
PTS1d: 9977 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 10
LB: 0.0
Nuts - $rzq-1-84-H.fid
PPM


CHO
S
COOEt
9j
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Dec 19 2009
F1: 300.029
F2: 300.028
SW1: 5308
OF1: 2067.2
PTS1d: 10574 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA:
LB: 0.0
Nuts - $ly-10-SsH.fid
PPM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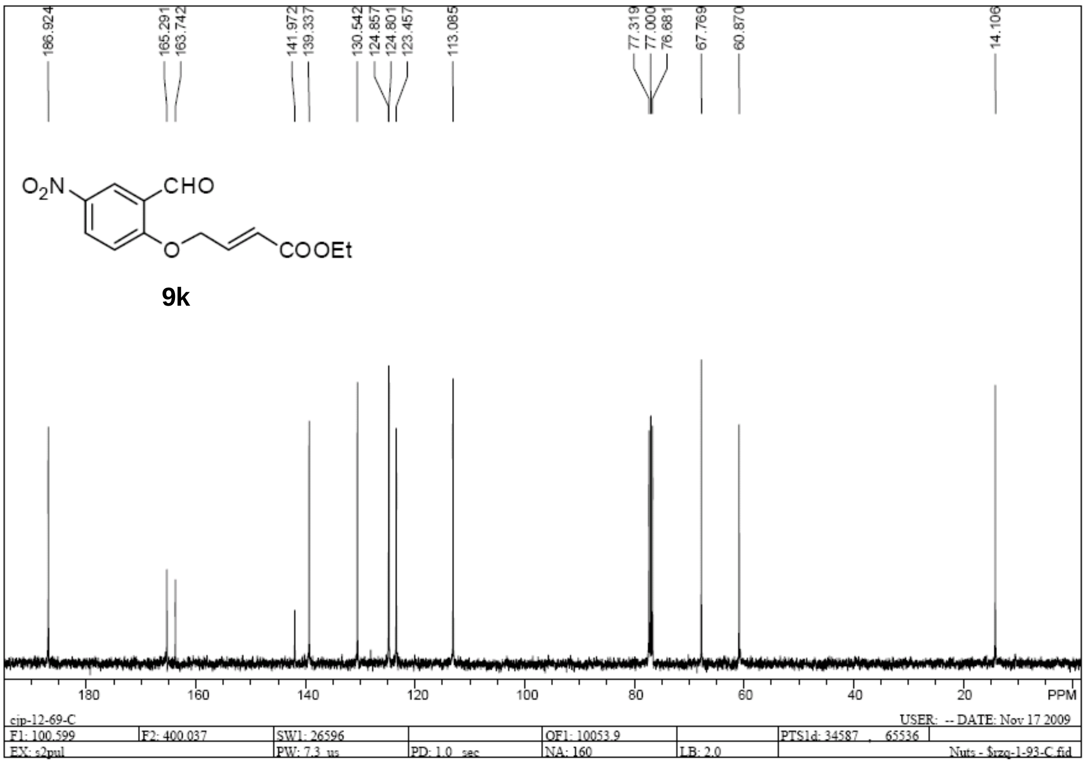
8.712 8.705 8.450 8.442 8.427 8.419
7.141 7.130 7.119 7.108 7.101 7.091 7.080
6.243 6.238 6.233 6.203 6.198 6.193
5.006 5.001 4.995 4.990
4.271 4.254 4.236 4.218
1.338 1.320 1.302
0.000
O_2N
CHO
O
COOEt
9k
0.88
0.90
0.91
0.92
0.97
0.92
1.88
2.00
3.00
10
8
6
4
2
PPM
:blank line
USER: -- DATE: Nov 17 2009
F1: 400.037
F2: 100.598
SW1: 6410
OF1: 2412.8
PTS1d: 19231 , 32768
EX: s2pul
PW: 4.4 us
PD: 1.0 sec
NA: 16
LB: 0.0
Nuts - $rzq-1-93-H.fid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165.291 163.742
141.972 139.337
130.542 124.857 124.801 123.457
113.085
77.319 77.000 76.681
67.769
60.870
14.106
O_2N
CHO
O
COOEt
9k
180
160
140
120
100
80
60
40
20
PPM
cjp-12-69-C
USER: -- DATE: Nov 17 2009
F1: 100.599
F2: 400.037
SW1: 26596
OF1: 10053.9
PTS1d: 34587 , 65536
EX: s2pul
PW: 7.3 us
PD: 1.0 sec
NA: 160
LB: 2.0
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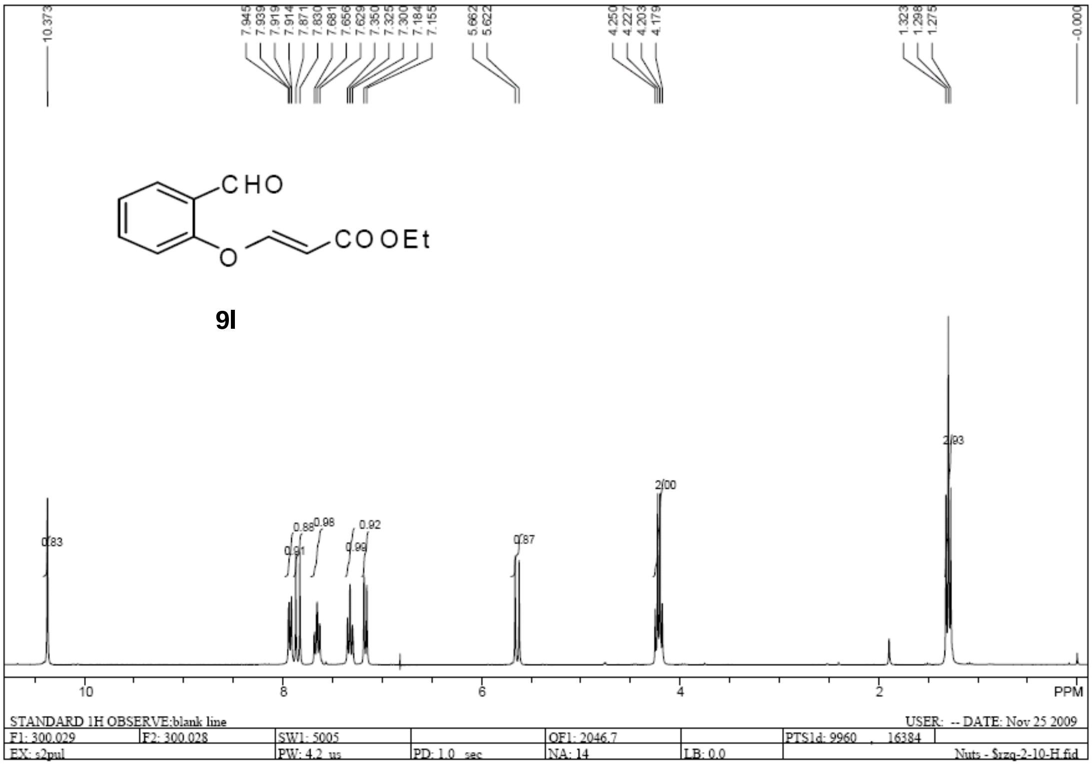
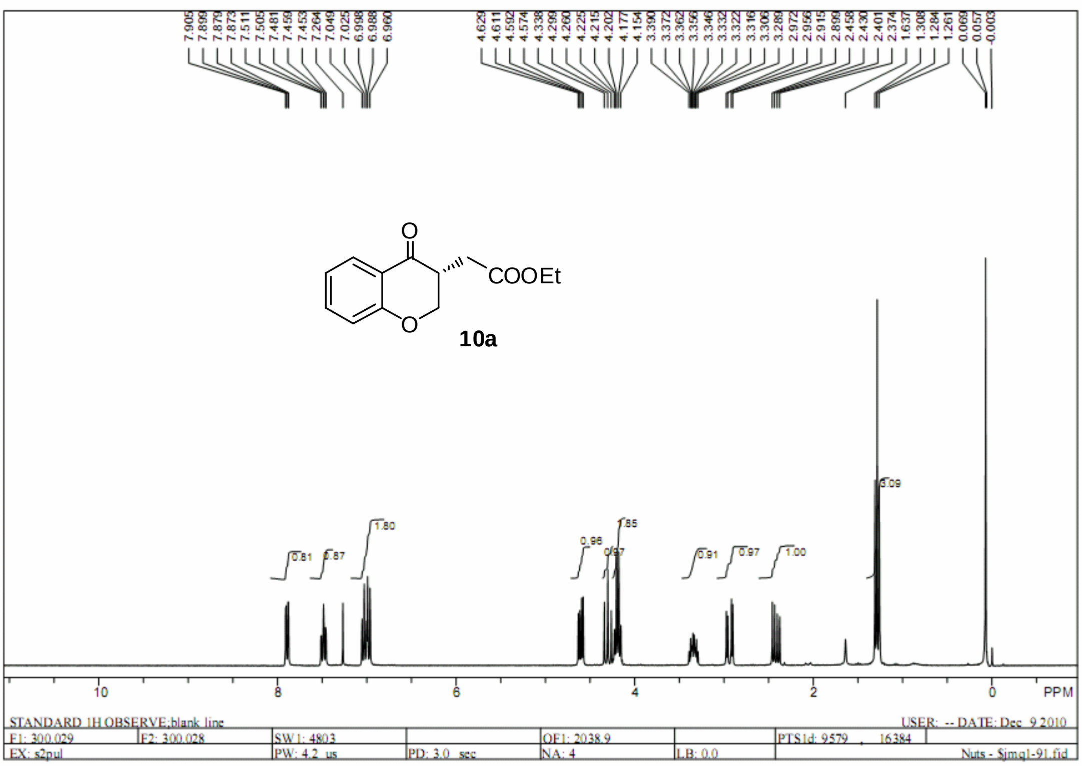
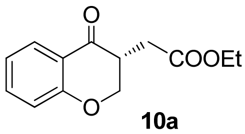
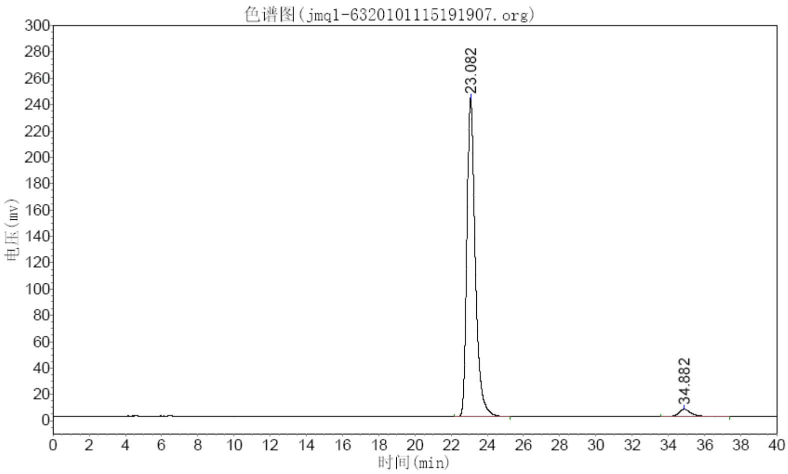

CHO
COOEt
O
9l
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Nov 25 2009
F1: 300.029
F2: 300.028
SW1: 5005
OF1: 2046.7
PTS1d: 9960 , 16384
EX: s2pul
PW: 4.2 us
PD: 1.0 sec
NA: 14
LB: 0.0
Nuts - $rzq-2-10-H.fid


O
COOEt
O
10a
PPM
STANDARD 1H OBSERVE:blank line
USER: -- DATE: Dec 9 2010
F1: 300.029
F2: 300.028
SW1: 4803
OF1: 2038.9
PTS1d: 9579 , 16384
EX: s2pul
PW: 4.2 us
PD: 3.0 sec
NA: 4
LB: 0.0
Nuts - $jmq1-91.fid



10a

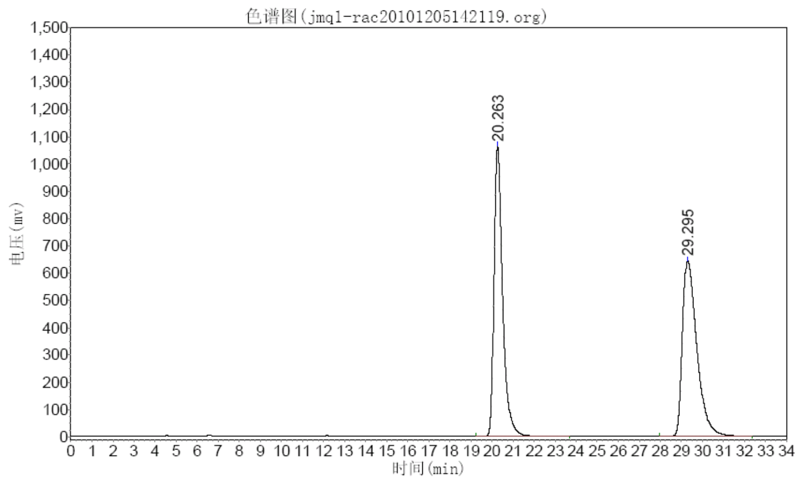
实验时间：2010-11-15,19:59:14　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-6320101115191907.org　　　　报告时间：2011-01-03,23:24:55



分析结果表

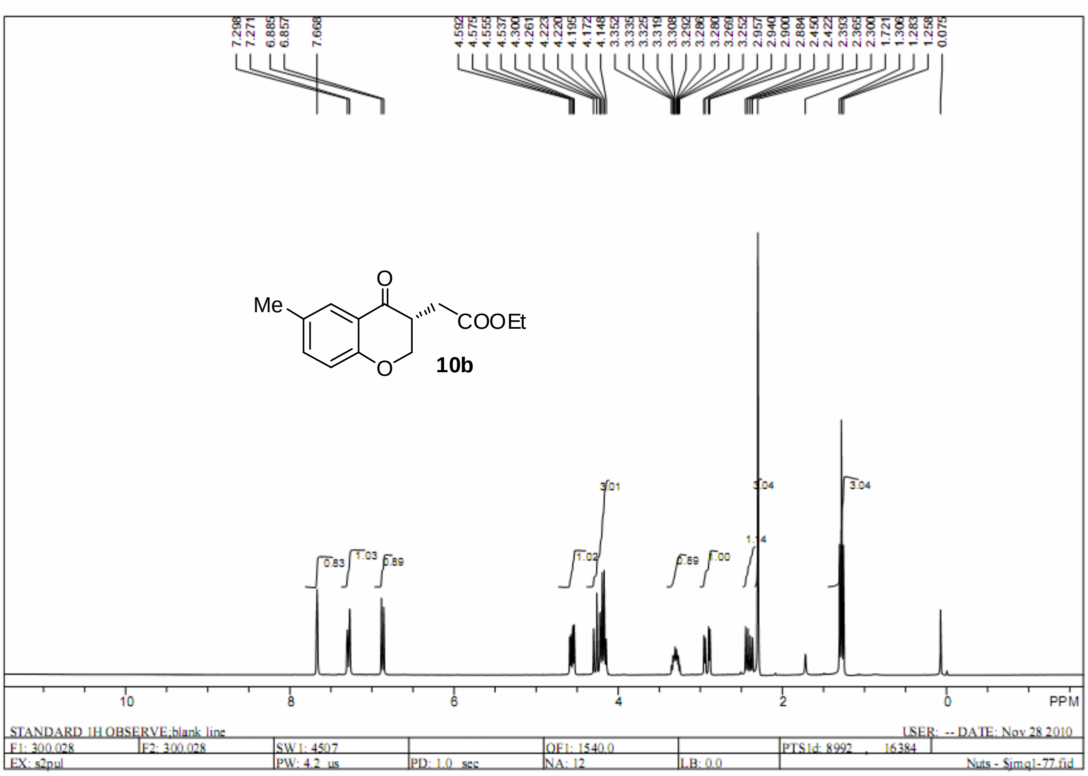
峰号	峰名	保留时间	峰高	峰面积	含量
1		23.082	241988.906	7704997.500	96.6335
2		34.882	5620.678	268429.313	3.3665
总计			247609.584	7973426.813	100.0000

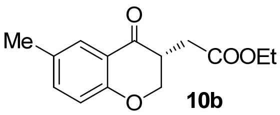
实验时间：2010-12-5,14:55:57　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-rac20101205142119.org　　　　报告时间：2010-12-5,14:57:49



分析结果表

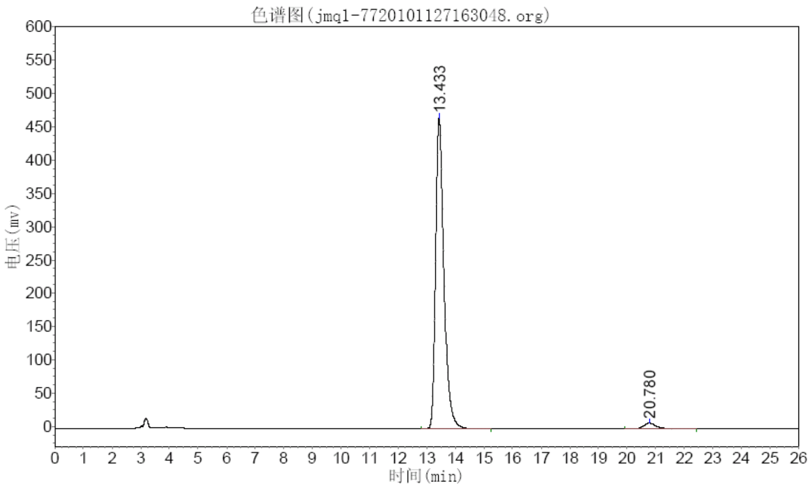
峰号	峰名	保留时间	峰高	峰面积	含量
1		20.263	1063086.500	30161286.000	49.7534
2		29.295	642151.813	30460318.000	50.2466
总计			1705238.313	60621604.000	100.0000





10b

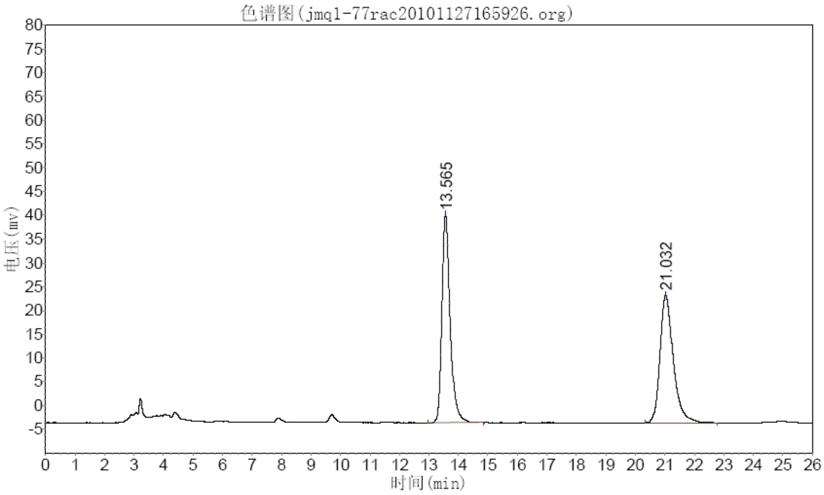
实验时间：2010-11-27, 16:57:11　　　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-7720101127163048.org　　　　　　报告时间：2011-01-03, 23:39:10



分析结果表

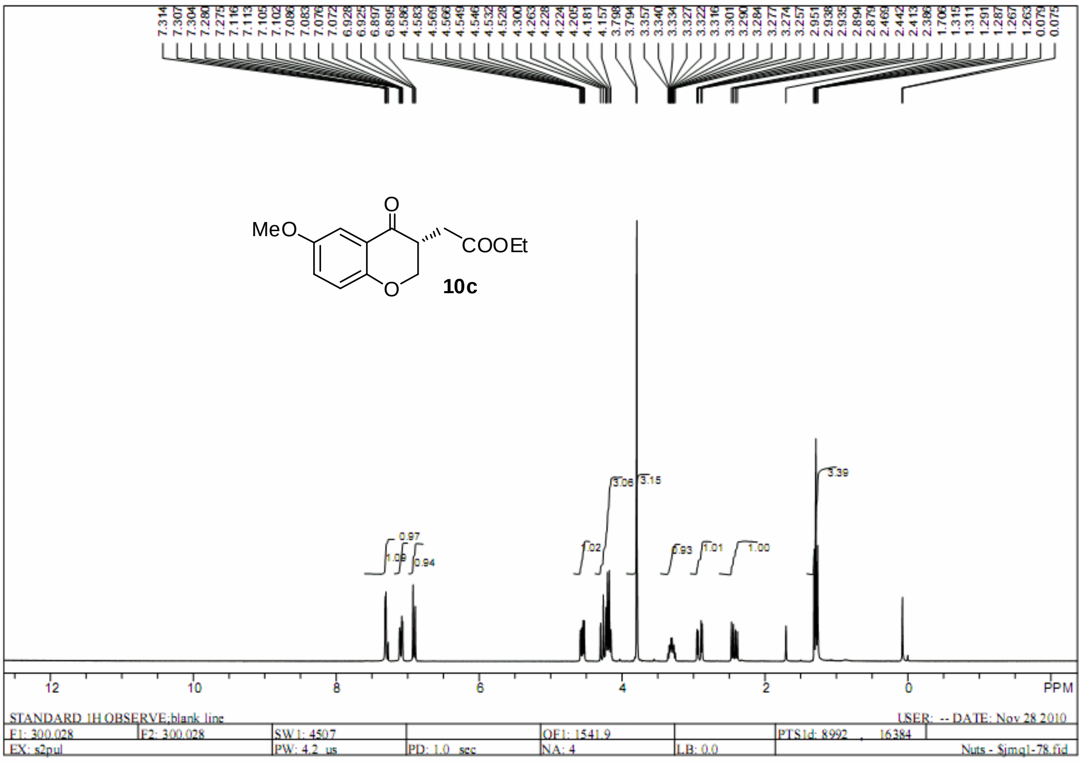
峰号	峰名	保留时间	峰高	峰面积	含量
1		13.433	466598.063	9521052.000	97.2881
2		20.780	8720.130	265401.313	2.7119
总计			475318.192	9786453.313	100.0000

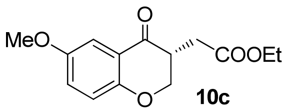
实验时间：2010-11-27,17:28:54　　　　　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-77rac20101127165926.org　　　　报告时间：2011-01-03,23:40:31



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		13.565	43694.875	863654.875	50.7774
2		21.032	26724.771	837208.938	49.2226
总计			70419.646	1700863.813	100.0000





10c

实验时间：2010-11-27,18:05:09　　　　实验者：jmq
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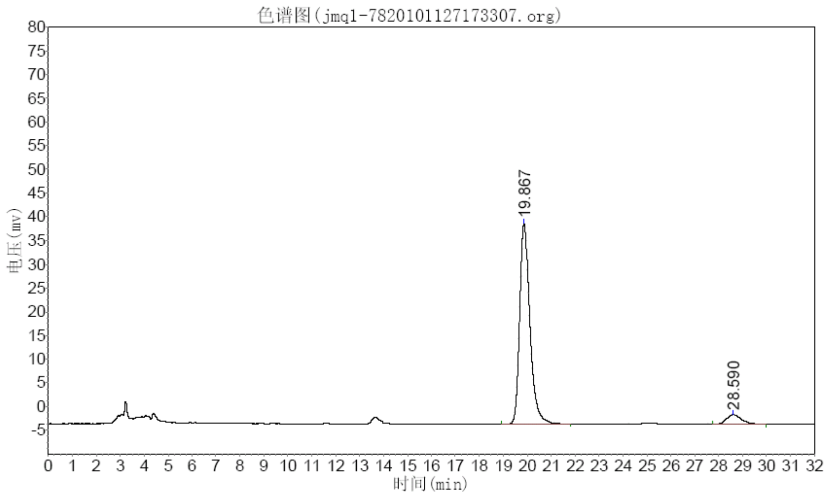

分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		19.867	42243.957	1244112.750	93.8639
2		28.590	1939.420	81330.930	6.1361
总计			44183.377	1325443.680	100.0000

实验时间：2010-11-27,18:44:55　　　　实验者：jmq
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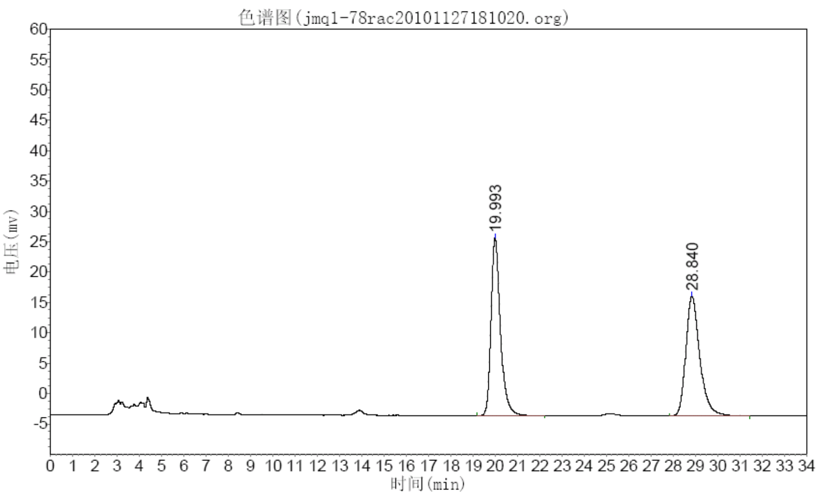
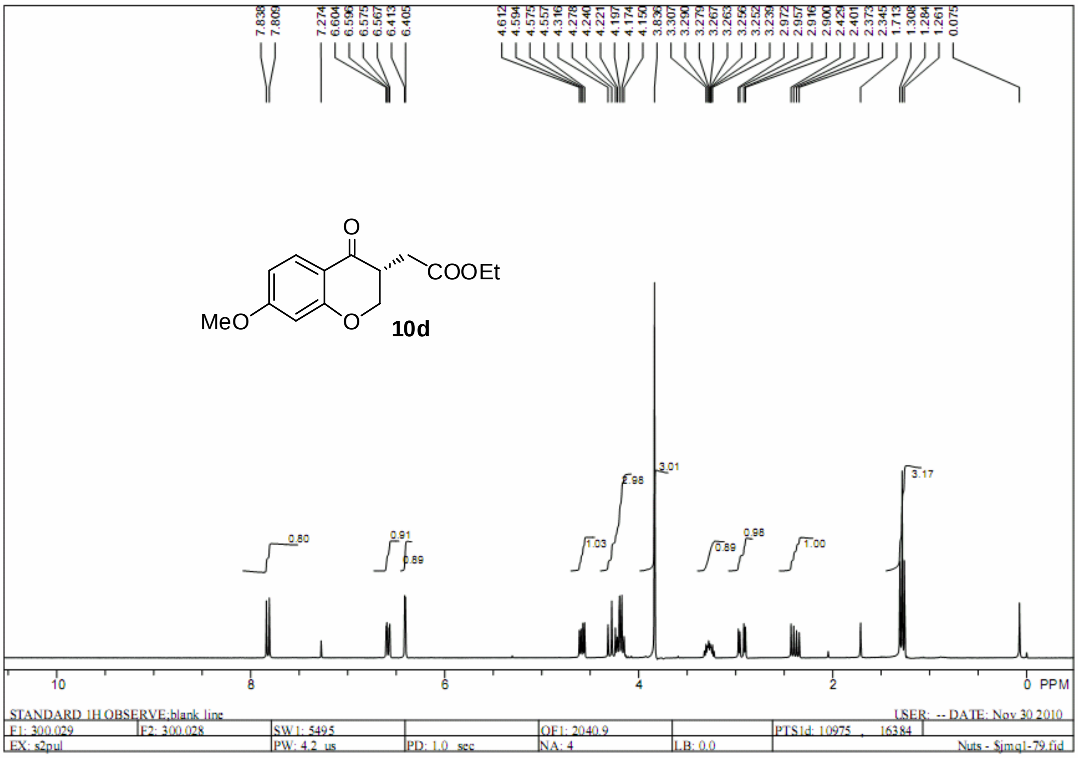
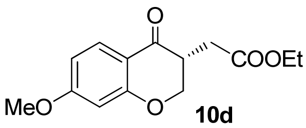


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		19.993	29288.502	869811.625	50.1339
2		28.840	19751.318	865163.750	49.8661
总计			49039.820	1734975.375	100.0000

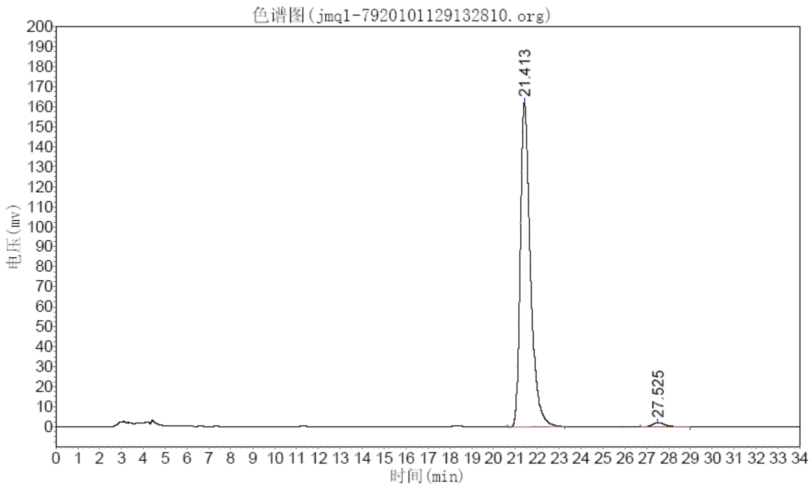




10d

实验时间：2010-11-29, 14:03:47
谱图文件:D:\date\jmq\jmql-7920101129132810. org

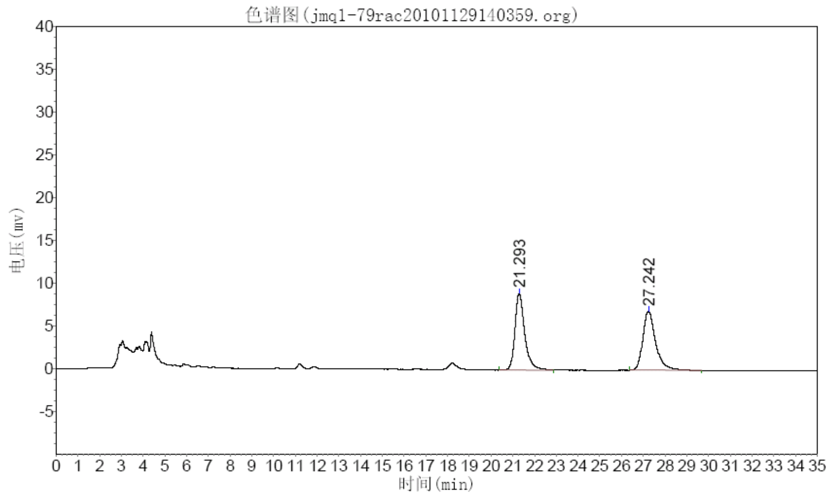
实验者：jmq
报告时间：2011-01-03, 23:42:45



分析结果表

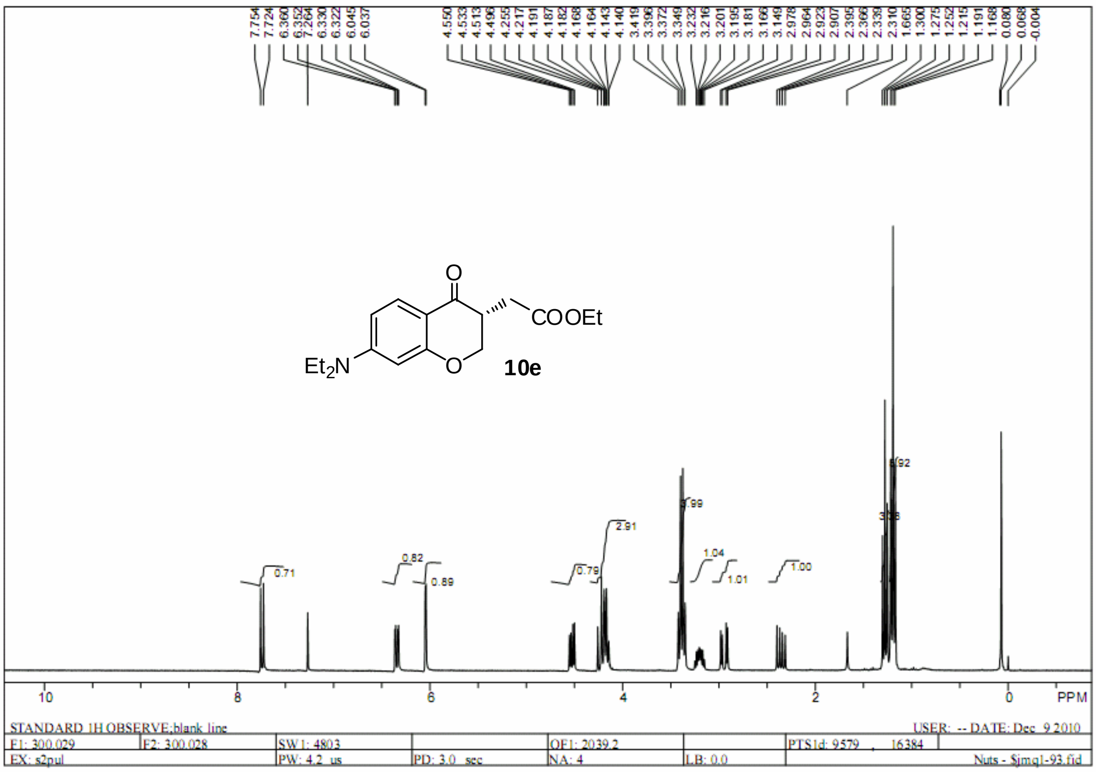
峰号	峰名	保留时间	峰高	峰面积	含量
1		21.413	162249.422	5262747.000	98.3419
2		27.525	2216.440	88731.500	1.6581
总计			164465.862	5351478.500	100.0000

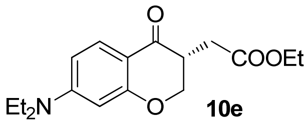
实验时间：2010-11-29, 14:48:05　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-79rac20101129140359.org　　　　报告时间：2010-11-29, 14:53:35



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		21.293	8996.183	281985.188	49.8550
2		27.242	6954.303	280161.156	49.5325
3		37.310	75.651	3464.700	0.6126
总计			16026.137	565611.043	100.0000





10e

实验时间：2010-12-9,11:18:01　　　　实验者：jmq
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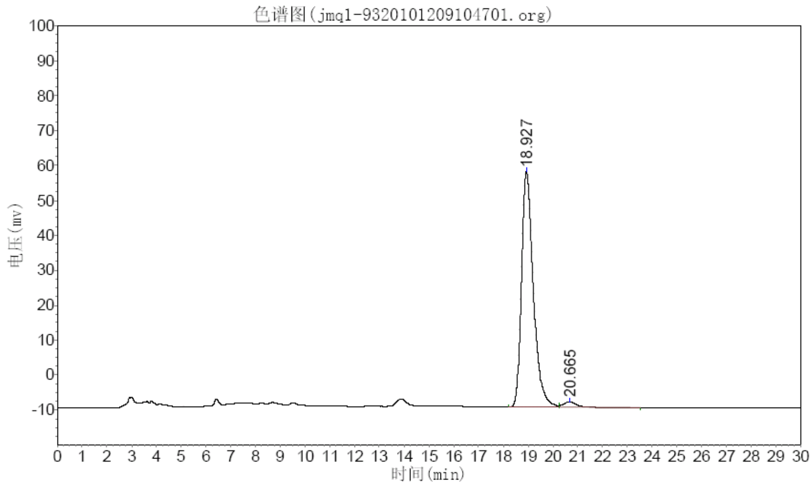
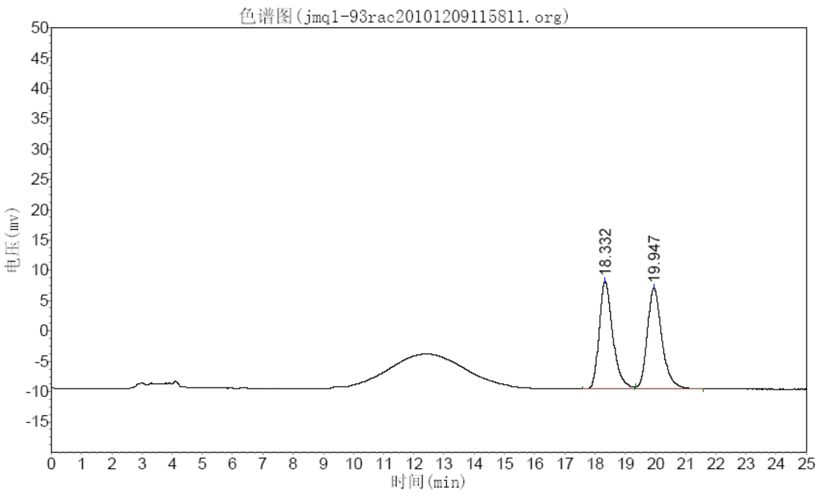


分析结果表

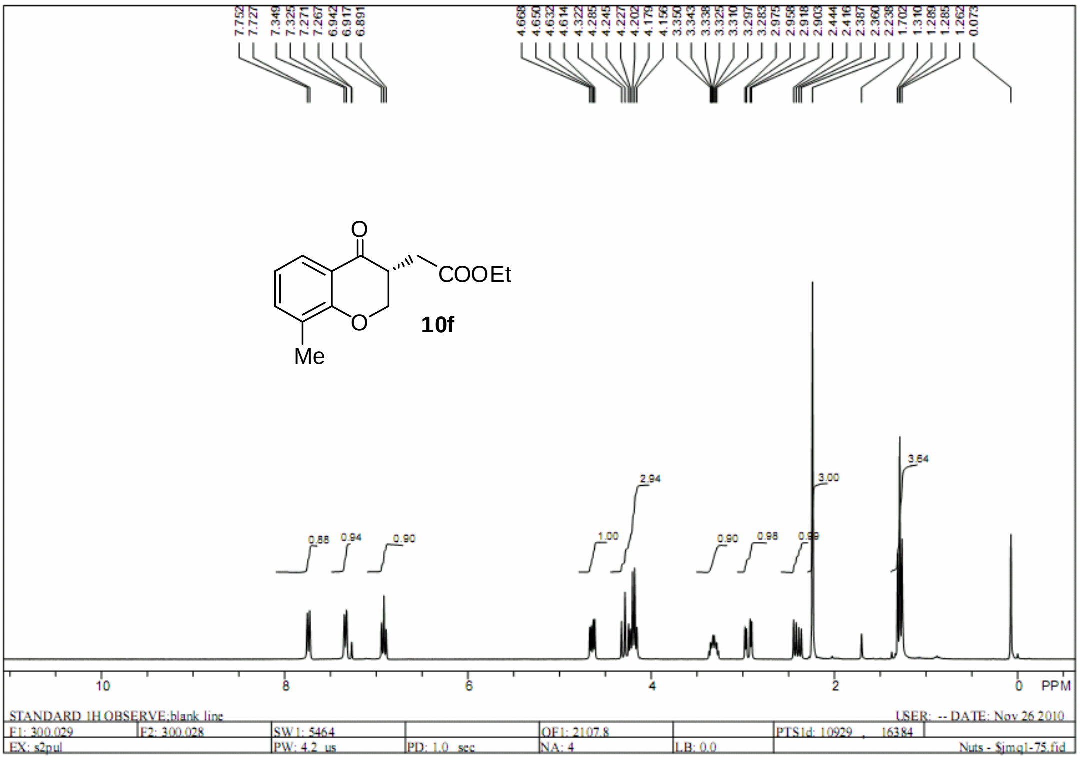
峰号	峰名	保留时间	峰高	峰面积	含量
1		18.927	67457.563	2243350.500	97.6320
2		20.665	1586.243	54411.020	2.3680
总计			69043.805	2297761.520	100.0000

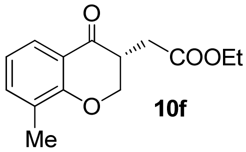
实验时间：2010-12-9,12:23:25　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-93rac20101209115811.org　　　　报告时间：2010-12-9,12:27:49



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		18.332	17764.010	555142.813	49.8923
2		19.947	16584.771	557539.688	50.1077
总计			34348.781	1112682.500	100.0000



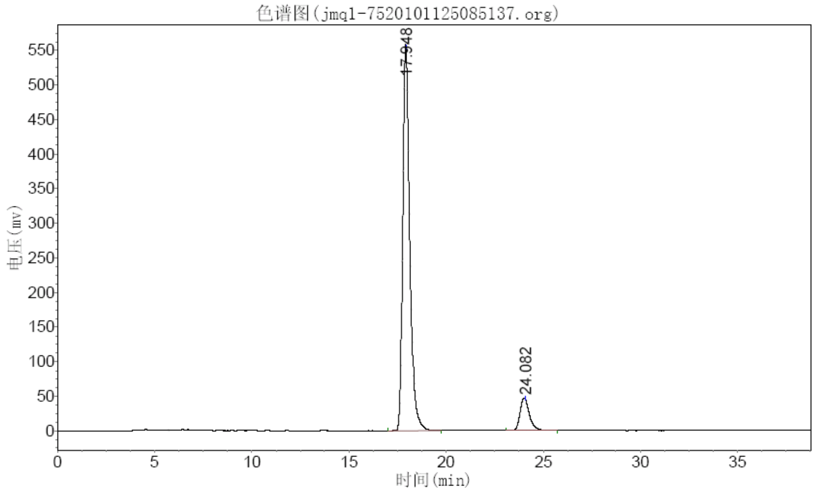


10f

实验时间：2010-11-25, 9:30:27　　　　实验者：jmq

谱图文件:D:\date\jmq\jmql-7520101125085137.org　　　　报告时间：2011-01-03, 23:34:06

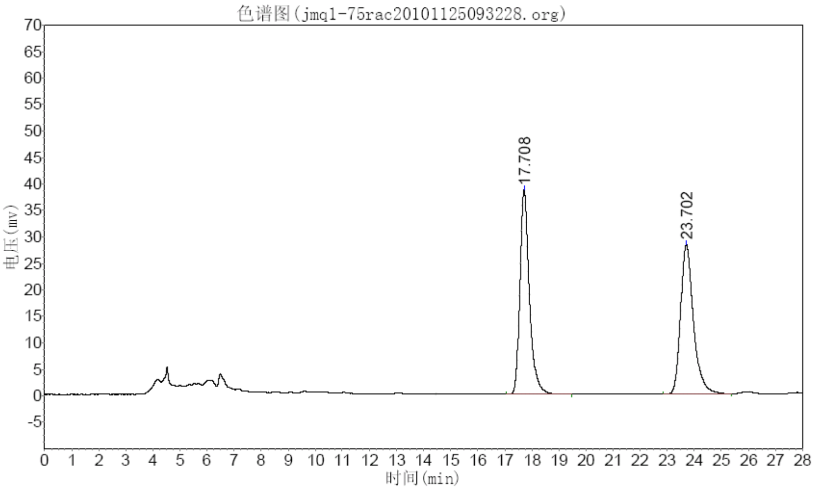
色谱图(jmql-7520101125085137.org)



分析结果表

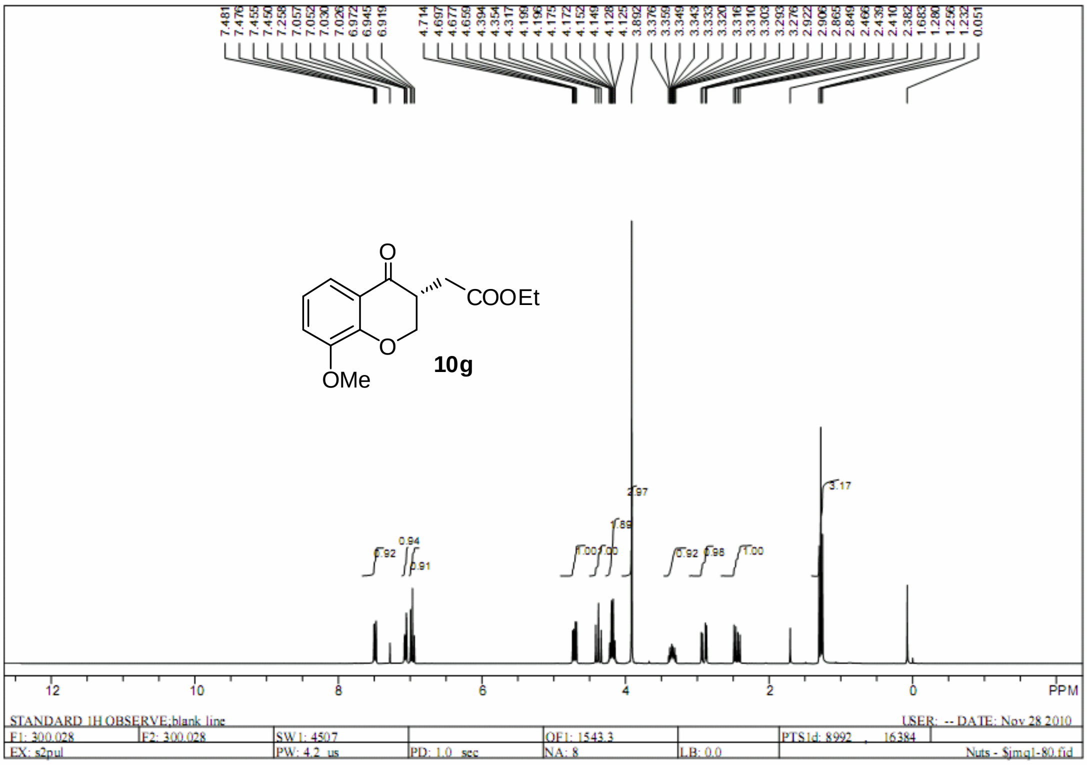
峰号	峰名	保留时间	峰高	峰面积	含量
1		17.948	555687.938	14029590.000	90.0028
2		24.082	45928.531	1558358.625	9.9972
总计			601616.469	15587948.625	100.0000

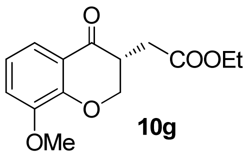
实验时间：2010-11-25,10:01:09　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-75rac20101125093228.org　　　　报告时间：2011-01-03,23:37:09



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		17.708	38563.473	943461.313	50.0665
2		23.702	28310.521	940956.750	49.9335
总计			66873.994	1884418.063	100.0000

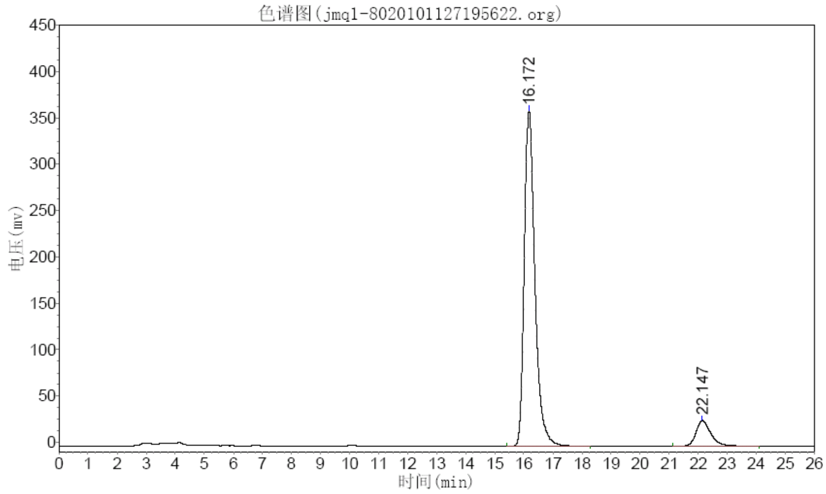




10g

实验时间：2010-11-27,20:22:31
谱图文件:D:\date\jmq\jmq1-8020101127195622.org

实验者：jmq
报告时间：2010-11-27,20:27:19

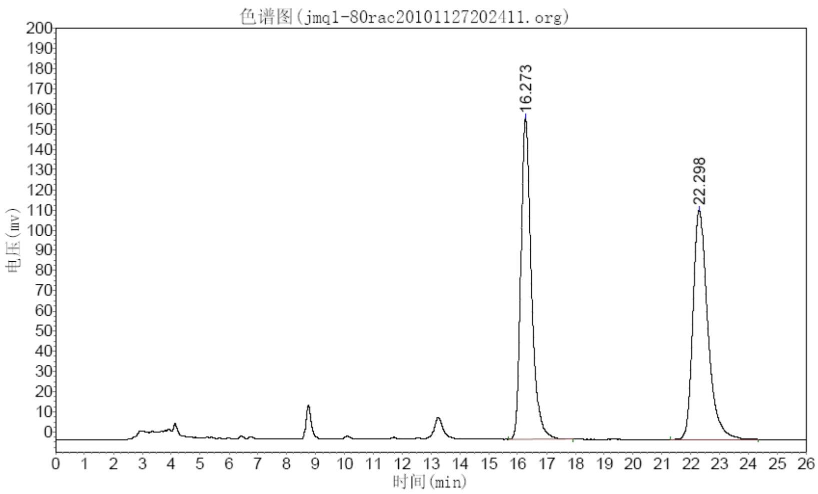


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.172	362048.375	9109131.000	90.4294
2		22.147	27882.139	964066.500	9.5706
总计			389930.514	10073197.500	100.0000

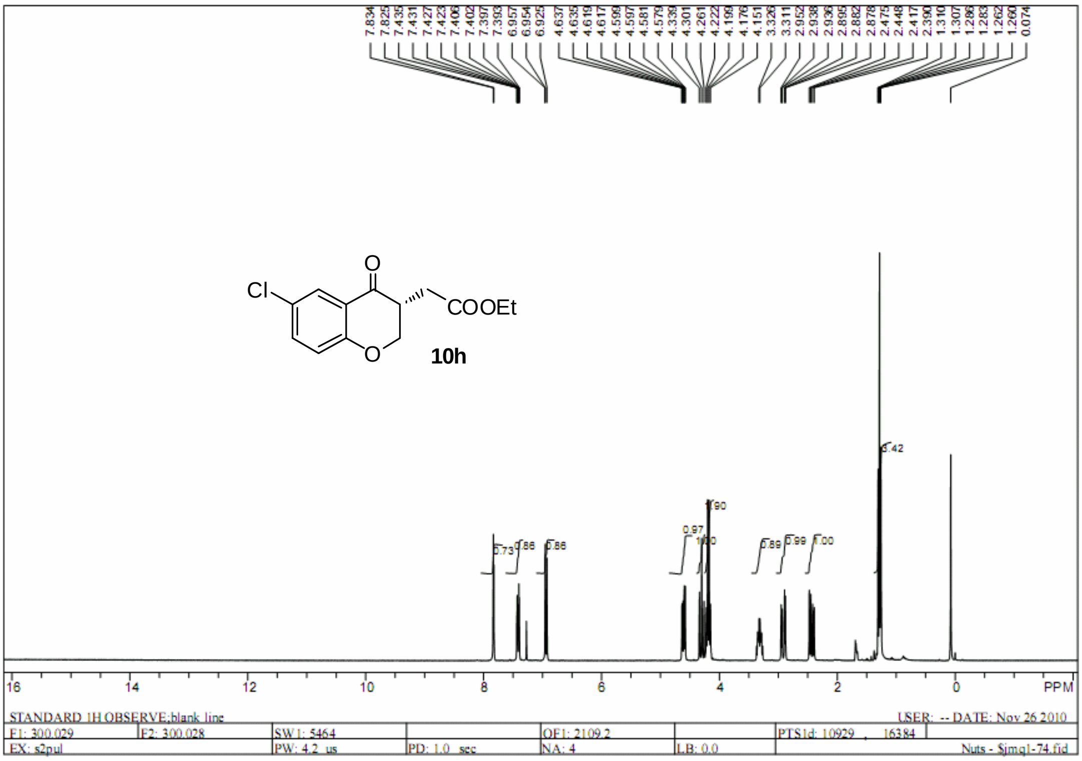
实验时间：2010-11-27,20:56:10
谱图文件:D:\date\jmq\jmq1-80rac20101127202411.org

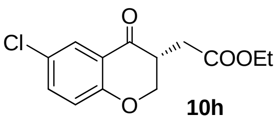
实验者：jmq
报告时间：2010-11-27,20:59:20



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		16.273	159079.484	4022908.500	50.0170
2		22.298	113539.344	4020169.000	49.9830
总计			272618.828	8043077.500	100.0000

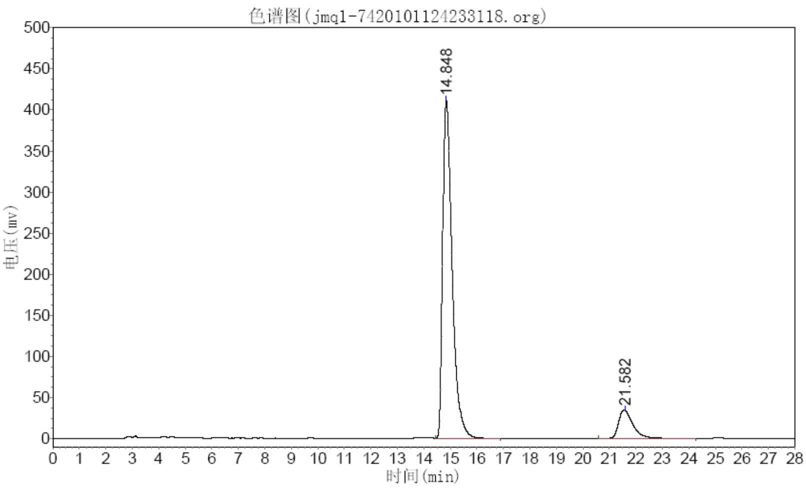




10h

实验时间：2010-11-25, 0:01:02　　　　实验者：jmq

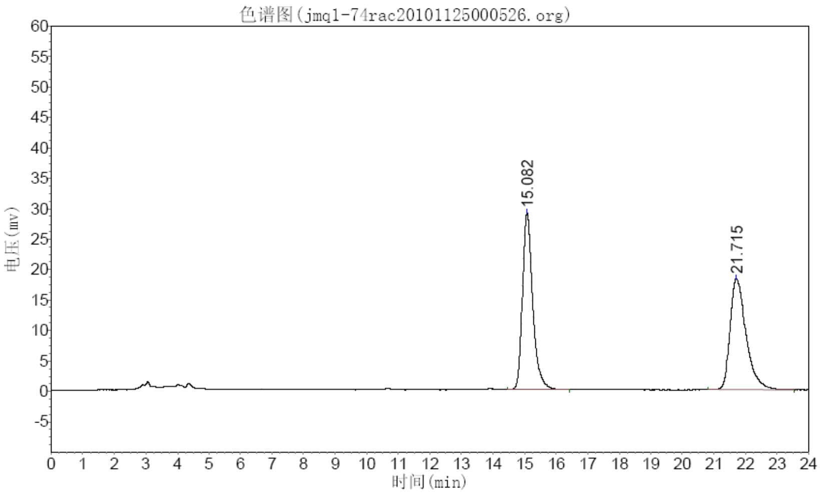
谱图文件:D:\date\jmq\jmq1-7420101124233118.org　　　　报告时间：2011-01-03, 23:27:08



分析结果表

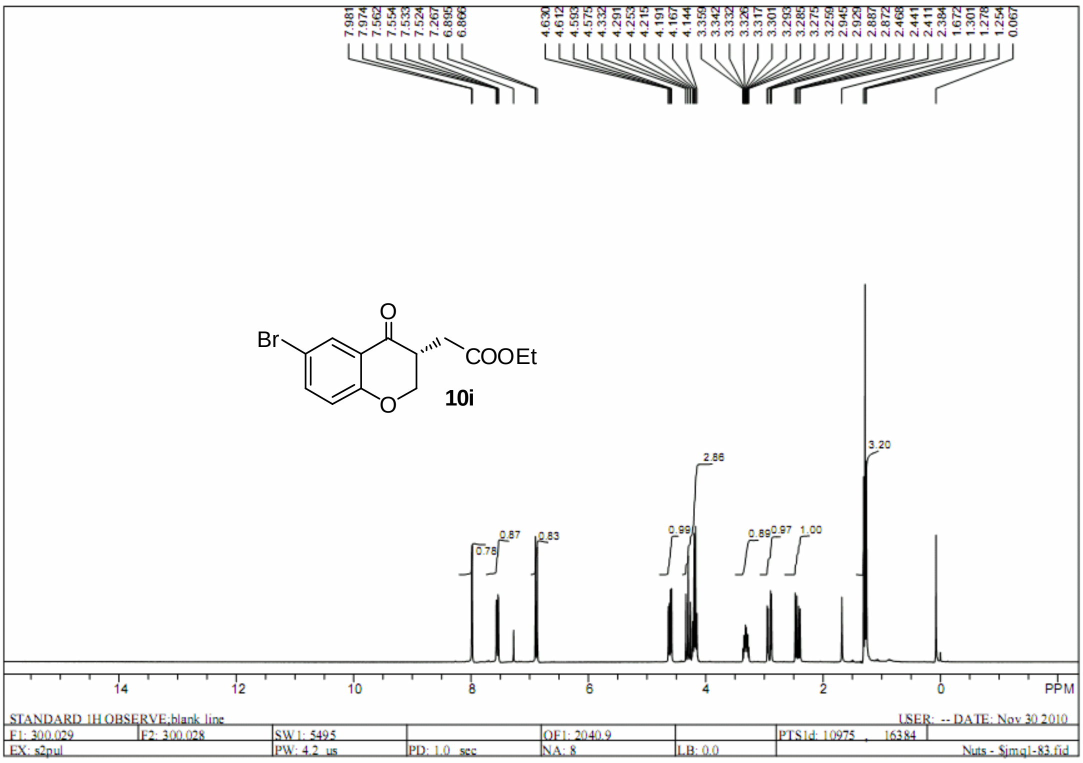
峰号	峰名	保留时间	峰高	峰面积	含量
1		14.848	410191.906	10131754.000	89.0507
2		21.582	34130.727	1245761.250	10.9493
总计			444322.633	11377515.250	100.0000

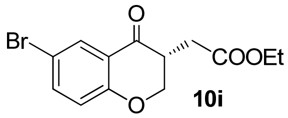
实验时间：2010-11-25,0:29:58　　　　　　　　　　　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-74rac20101125000526.org　　　　报告时间：2011-01-03,23:30:54



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		15.082	29080.645	657919.625	50.4974
2		21.715	18232.197	644958.813	49.5026
总计			47312.842	1302878.438	100.0000

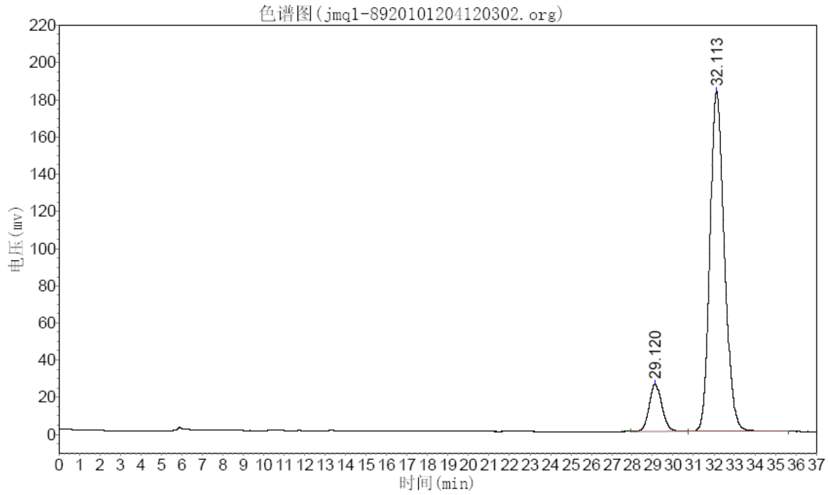




10i

实验时间：2010-12-4, 12:40:49
谱图文件:D:\date\jmq\jmq1-8920101204120302.org

实验者：jmq
报告时间：2010-12-4, 12:47:48

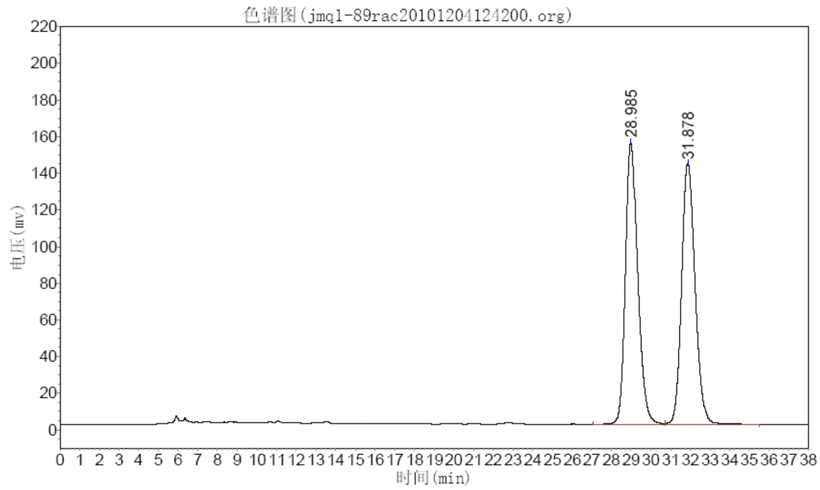


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		29.120	25340.496	1105027.375	10.9518
2		32.113	182760.219	8984932.000	89.0482
总计			208100.715	10089959.375	100.0000

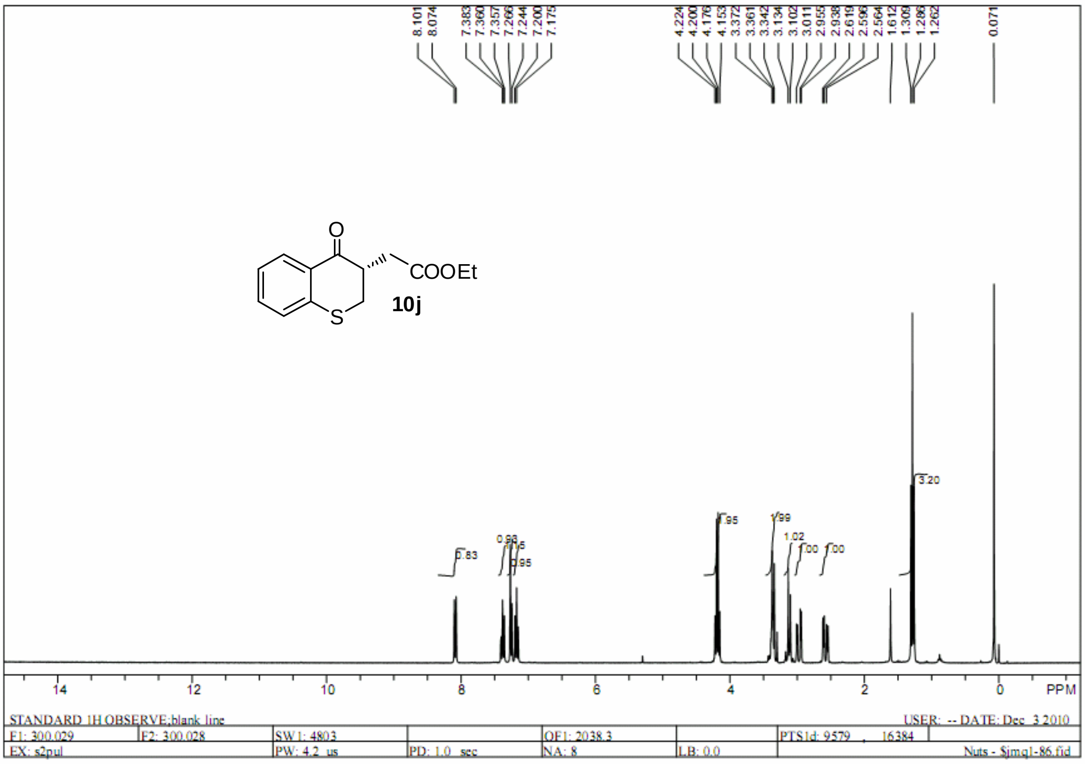
实验时间：2010-12-04, 13:20:16
谱图文件:D:\date\jmq\jmq1-89rac20101204124200.org

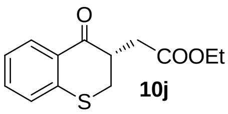
实验者：jmq
报告时间：2010-12-04, 13:23:59



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		28.985	153544.859	6703626.500	49.9836
2		31.878	141922.813	6708016.000	50.0164
总计			295467.672	13411642.500	100.0000

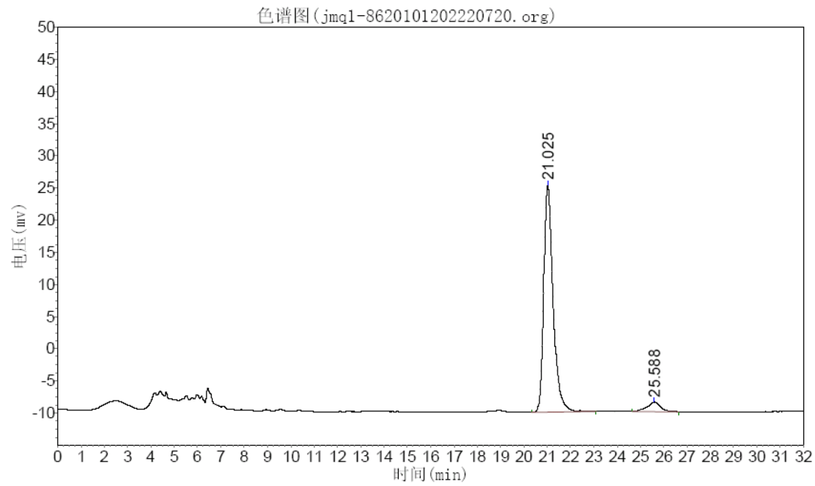




10j

实验时间：2010-12-02, 22:39:25
谱图文件:D:\date\jmq\jmq1-8620101202220720.org

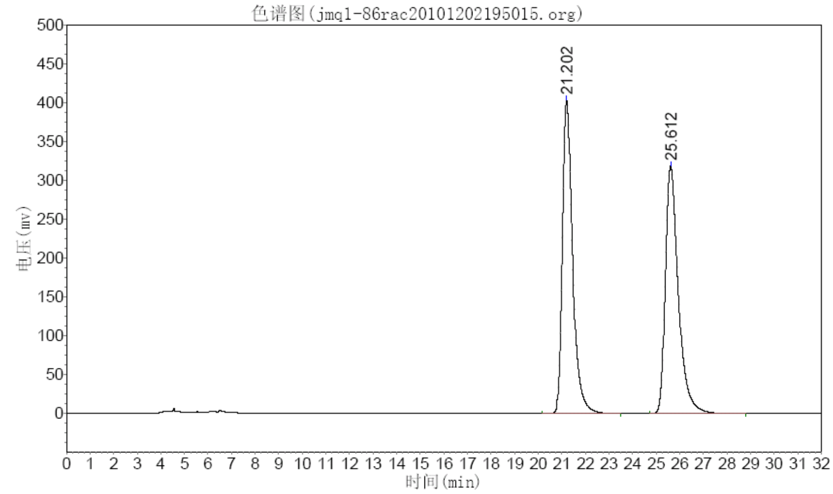
实验者：jmq
报告时间：2010-12-02, 22:42:55



分析结果表

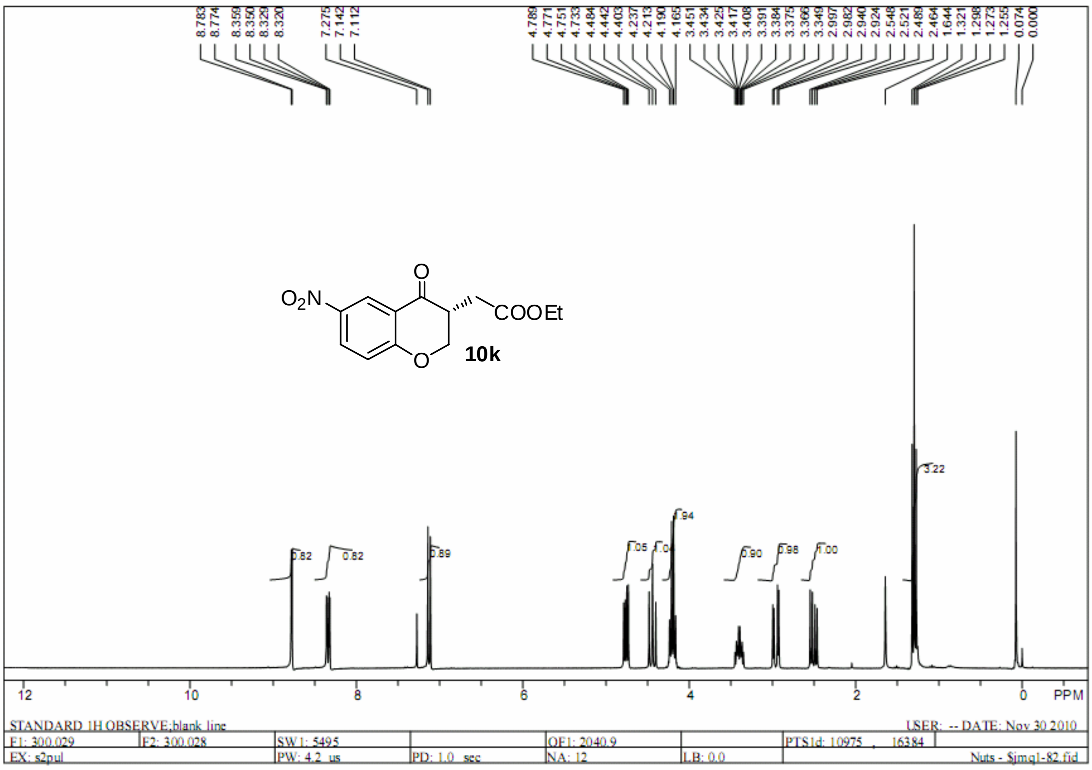
峰号	峰名	保留时间	峰高	峰面积	含量
1		21.025	35291.648	988533.813	94.3072
2		25.588	1518.530	59672.734	5.6928
总计			36810.178	1048206.547	100.0000

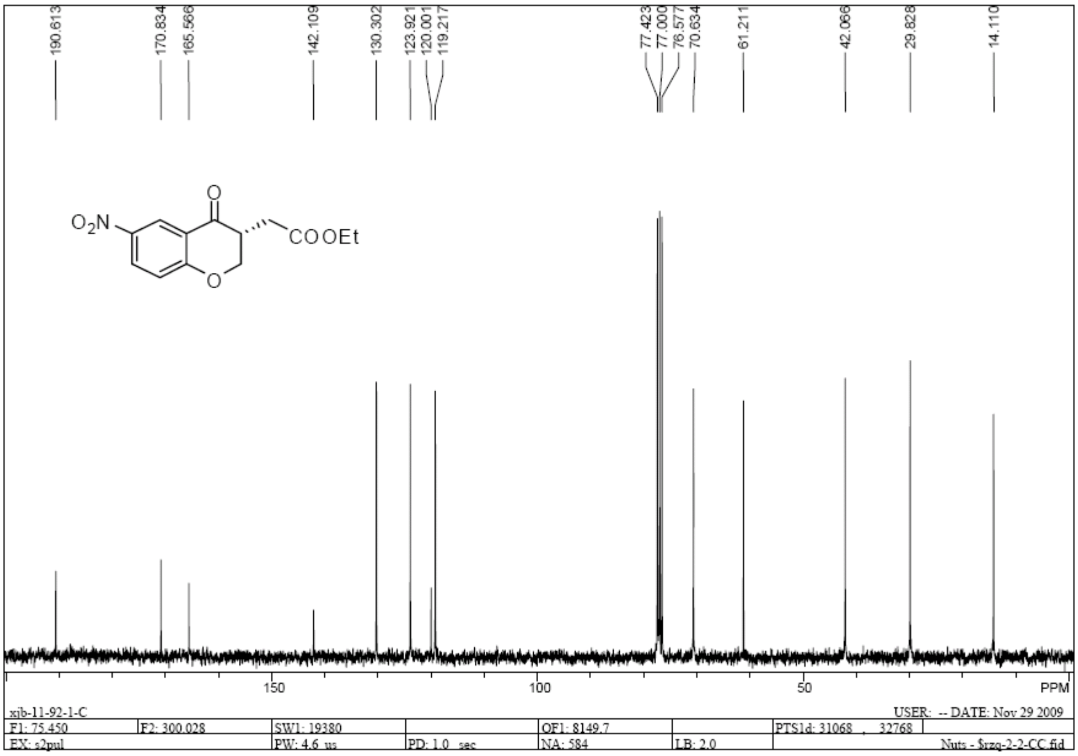
实验时间：2010-12-2,20:29:03　　　　　　　　　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-86rac20101202195015.org　　　报告时间：2010-12-2,22:44:54

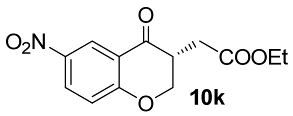


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		21.202	403654.688	12132979.000	49.9236
2		25.612	319292.219	12170135.000	50.0764
总计			722946.906	24303114.000	100.0000



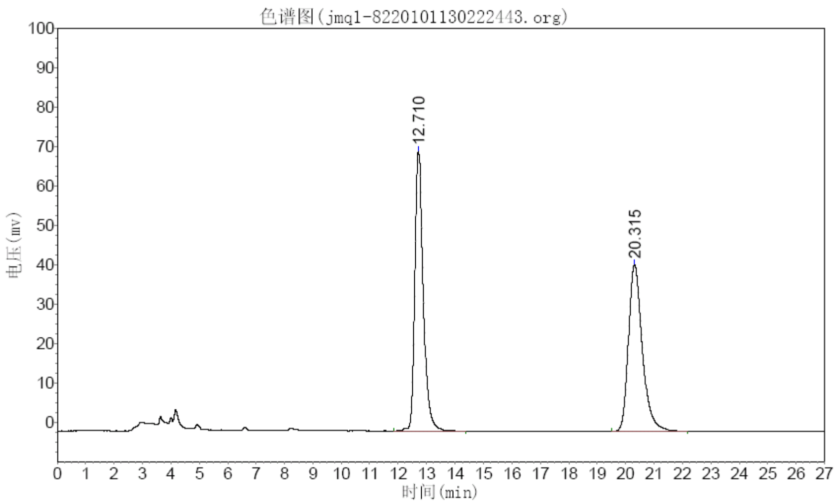




10k

实验时间：2010-11-30,23:00:07
谱图文件:D:\date\jmq\jmq1-8220101130222443.org

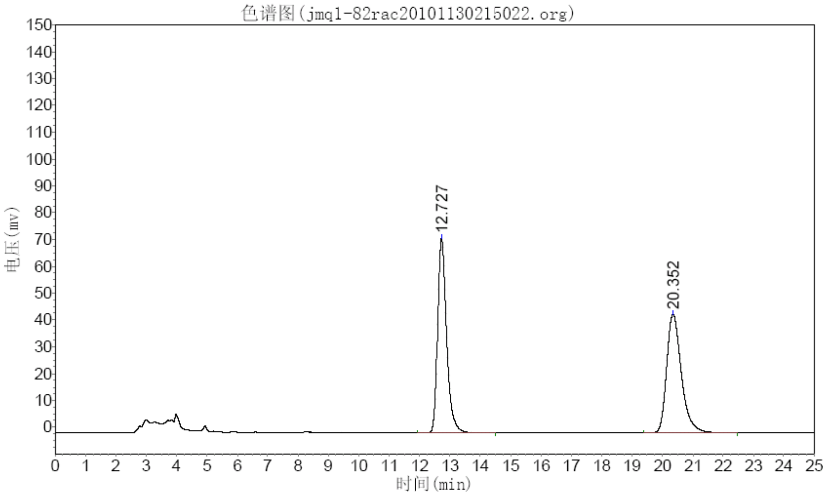
实验者：jmq
报告时间：2010-12-5,18:28:37



分析结果表

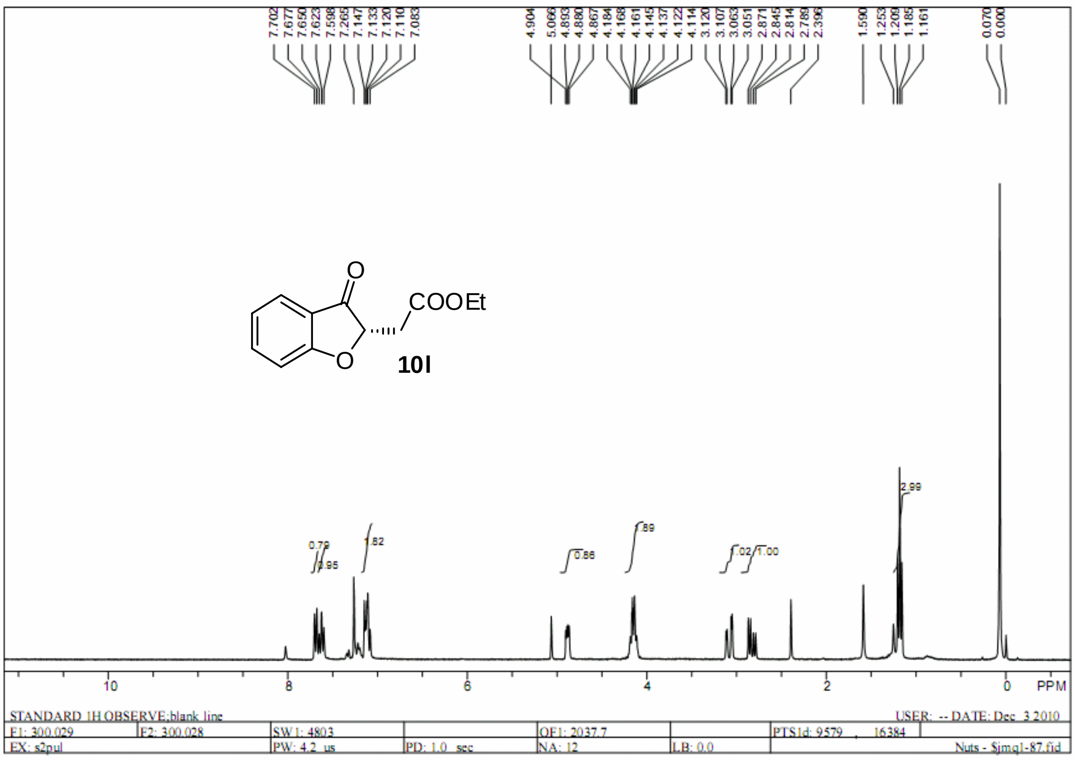
峰号	峰名	保留时间	峰高	峰面积	含量
1		12.710	71117.141	1473330.375	50.7944
2		20.315	42378.586	1427246.750	49.2056
总计			113495.727	2900577.125	100.0000

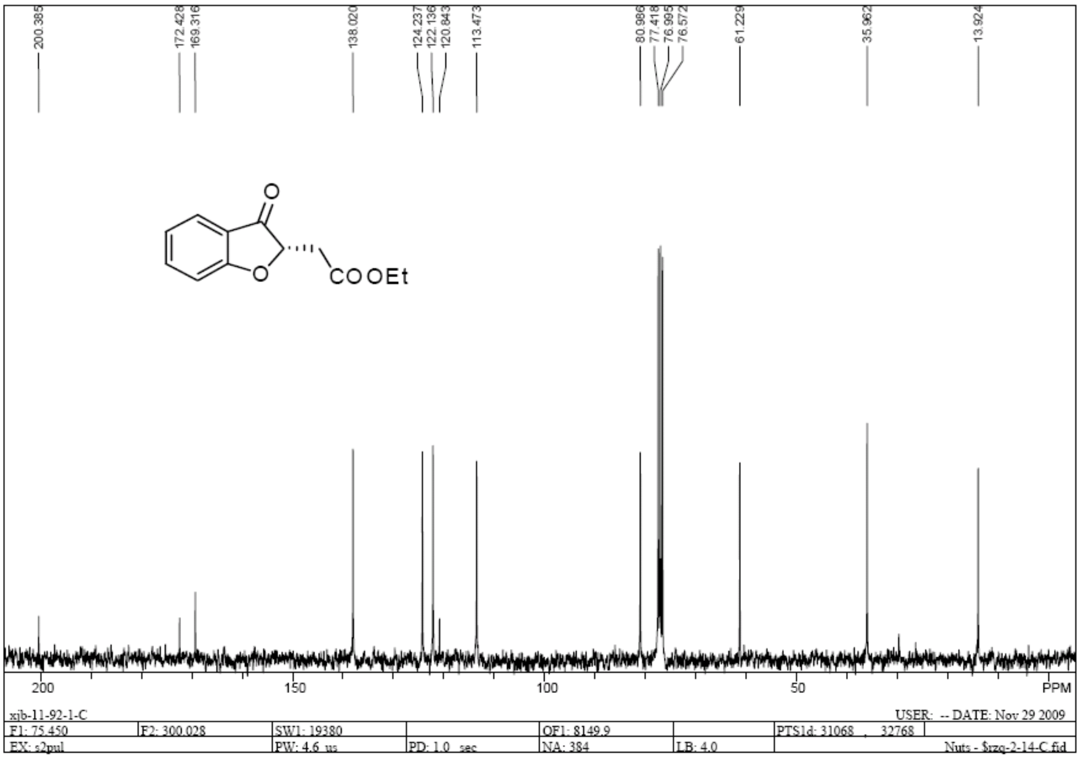
实验时间：2010-11-30, 22:16:38　　　　　　　　　　　　　　实验者：jmq
谱图文件:D:\date\jmq\jmql-82rac20101130215022.org　　　　　报告时间：2010-11-30, 23:04:47

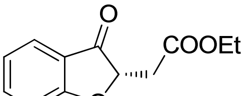


分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		12.727	72572.391	1495748.250	50.0605
2		20.352	44116.188	1492132.875	49.9395
总计			116688.578	2987881.125	100.0000

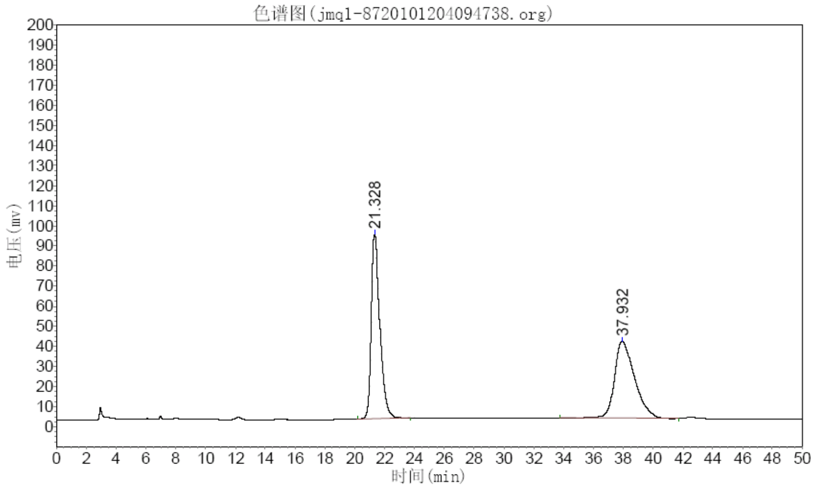






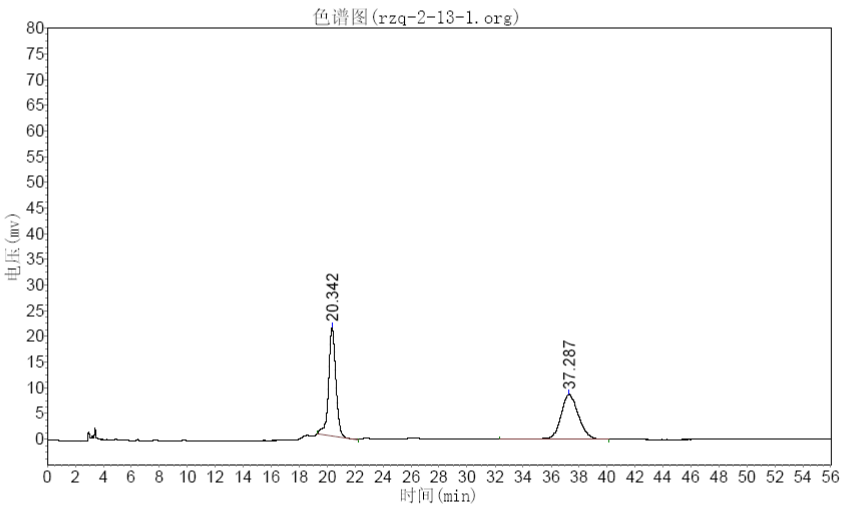
10l

实验时间：2010-12-4,10:37:45　　　　实验者：jmq
谱图文件:D:\date\jmq\jmq1-8720101204094738.org　　　　报告时间：2010-12-4,12:24:50



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		21.328	92007.492	3675771.250	50.7610
2		37.932	38220.797	3565559.500	49.2390
总计			130228.289	7241330.750	100.0000



分析结果表

峰号	峰名	保留时间	峰高	峰面积	含量
1		20.342	21179.414	771577.500	49.9817
2		37.287	8756.937	772141.625	50.0183
总计			29936.351	1543719.125	100.0000