Asymmetric Aziridination of Chalcones Catalyzed by a Novel Backbone 1,8-BisoxazolinylAnthracene (AnBOX)-Copper Complex

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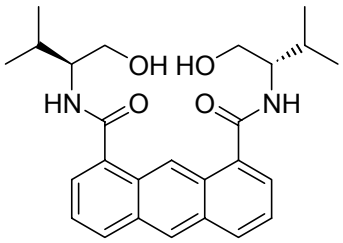
Electronic Supplementary Information

Experimental Section

General Methods. IR spectra were recorded on a Bruker Vector 22 FT-IR spectrophotometer. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Varian Mercury 200 (200 MHz) and Mercury Plus 300 (300 MHz) spectrometer in $CDCl_3$ solution with TMS as an internal standard and chemical shifts are reported in ppm. Mass spectra were obtained on a VG-ZAB-HS spectrometer. CH analyses were performed on an Elementar Vario EL analyzer. Optical rotations were measured on a Perkin-Elmer Model 341LC polarimeter with a thermally jacketed 10 cm cell (concentration c given as g/100 mL). HPLC analyses were performed on an HP1100 HPLC equipment. The e.e. values were determined by HPLC analysis with chiralcel AS, OD or OD-H columns (4.6×250 mm) with a mixture of hexane-isopropanol as an eluent at an eluent rate of 0.5 or 0.8 mL/min at monitoring wave 254 nm. All chalcones were prepared according to the literature procedure.[1] TsN=IPh was prepared according to the literature method.[2] CuOTf·1/2PhH was purchased from Aldrich. Benzene was heated under reflux over sodium and distilled prior to use. Dichloromethane and acetonitrile were refluxed and distilled from calcium hydride prior to use. All reactions were carried out under an atmosphere of nitrogen in gas burner-dried glassware with magnetic stirring.

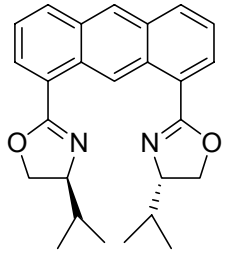
Synthesis of chiral ligand AnBOX: Anthracene-1,8-dicarboxylic acid was prepared as previously reported[3] and anthracene-1,8-dicarbonyl dichloride was synthesized according to the literature procedure.[4]

(*S,S*)-*N,N*'-Bis[1-(hydroxymethyl)-2-methylpropyl]-anthracene-1,8-dicarboxamide



A 250 mL flask fitted with a magnetic stir bar was charged with a solution of 0.87 g (2.9 mmol) anthracene-1,8-dicarbonyl dichloride in 50 mL of dry THF. The solution was cooled in an ice bath, and a solution of 0.89 g (8.7) L-valinol in 30 mL of dry THF was added dropwise. Some yellow solid was observed. After addition, the ice bath was removed and the yellow suspension was stirred at room temperature overnight, then filtered to give yellow crude product, which was recrystallized from ethanol to afford 1.04 g of yellow needle crystals in 83% yield. mp 252-254 ^{o}C; TLC ethyl acetate, R_f = 0.47; $[\alpha]^{24}_{D}$ = -65.9 (*c* 0.41, CH_3OH); IR (KBr) *v* cm^{-1} 3314 (O-H), 1635 (C=O): ^{1}H NMR (400 MHz, DMSO-d_6/TMS) δ 0.95 (d, *J* = 6.8 Hz, 6H, $2CH_3$), 1.01 (d, *J* = 6.8 Hz, 6H, $2CH_3$), 1.97-2.02 [m, 2H, 2C**H**$(CH_3)_2$], 3.55-3.58 (m, 4H, $2CH_2O$), 3.93-3.97 (m, 2H, 2C**H**NH), 4.62 (t, *J* =5.6 Hz, 2H, 2OH), 7.53-7.61 (m, 4H, 2NH, 2ArH), 8.15-8.19 (m, 2H, ArH), 8.66 (s, 1H, ArH), 9.18 (s, 1H, ArH); ^{13}C NMR (100 MHz, DMSO-d_6/TMS) δ 18.33, 19.84, 28.38, 56.21, 61.48, 123.09, 124.76, 124.97, 126.79, 128.19, 129.56, 131.04, 135.96, 168.54; MS (EI) *m/z* (relative intensity, %): 436 (9, M^+), 418 (6, M-H_2O), 406 (38), 388 (17), 357 (4), 348 (5), 334 (33), 316 (27), 307 (23), 289 (35), 272 (35), 248 (61), 233 (13), 221 (16), 204 (36), 190 (5), 176 (33); Anal. Calcd for $C_{26}H_{32}N_2O_4$: C, 71.53; H, 7.39; N, 6.42. Found: C, 71.72; H, 7.28; N, 6.12.

(*S,S*)-1,8-Bis[4-(1-methylethyl)oxazolin-2-yl]anthracene (AnBOX)

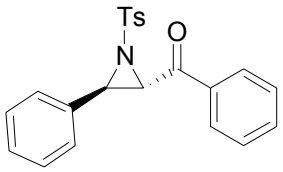


A 250 mL flask fitted with a magnetic stir bar was charged with a solution of 1.04 g (2.39 mmol) above dihydroxy diamide in 80 mL of dry THF, 12.1 mL (166 mmol) of $SOCl_2$ was added and the reaction mixture was brought to reflux for 4 h to give clear yellow solution. The resulting solution was evaporated *in vacuo* to remove THF and excess $SOCl_2$. The residue was dissolved in 120 mL of ethyl acetate, then washed with 48 mL of 2 M K_2CO_3, water, and brine, respectively. The organic layer was dried over Na_2SO_4, filtered, and concentrated *in vacuo* to yield yellow solid, which was used without purification. The yellow solid was dissolved in 112 mL of acetonitrile, then 24.2 g (175 mmol) K_2CO_3 and 9.7 mL of water were added. After refluxing for 8 h, the resulting solution was cooled to room temperature and concentrated, then 640 mL of ethyl acetate and 160 mL of water were added. The organic extract was dried over Na_2SO_4, filtered, and concentrated to give crude product, which was purified by column chromatography (silica gel 50 g, ethyl acetate: petroleum 1:4 (v/v)) to afford 0.77 g of yellow crystals in 80% yield. mp 122-124 ^{o}C; TLC ethyl acetate: petroleum 1:2 (v/v), R_f = 0.35; $[\alpha]^{20}_{D}$ = +64.7 (*c* 0.97, $CHCl_3$); IR (KBr) *v* cm^{-1} 1645 (C=N); ^{1}H NMR δ 1.03 (d, *J* = 6.9 Hz, 6H, $2CH_3$), 1.15 (d, *J* = 6.9 Hz, 6H,

2CH_3), 2.00-2.06 (m, 2H, 2C**H**(CH_3)$_2$), 4.23-4.33 (m, 4H, 2CH_2O), 4.51-4.56 (m, 2H, 2CHN), 7.48 (dd, *J* = 8.4, 6.9 Hz, 2H, ArH), 8.08-8.13 (m, 4H, ArH), 8.47 (s, 1H, ArH), 10.64 (s, 1H, ArH); ^{13}C NMR δ 18.09, 19.37, 32.84, 69.47, 73.24, 124.47, 124.88, 125.90, 127.54, 129.38, 129.48, 131.46, 131.74, 163.80; MS (EI) *m/z* (rel. intensity): 400 (22.2, M^+), 357 (18.7, M-C_3H_7), 330 (100.0), 287 (7.9), 271 (72.4), 244 (24.0), 228 (6.0), 216 (11.5), 202 (11.9), 190 (4.1), 176 (4.3); Anal. cacld for $C_{26}H_{28}N_2O_2$: C 77.97, H 7.05, N 6.99; Found: C 77.99, H 7.26, N 6.76.

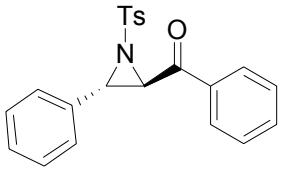
Asymmetric aziridination of chalcones, general procedure: A three-necked flask (25 mL) is charged with chalcone **2a** (312 mg, 1.50 mmol), AnBOX **1** (24 mg, 0.06 mmol) and the CuOTf·1/2PhH (13 mg, 0.05 mmol) under nitrogen atmosphere. Dichloromethane (8 mL) is added by syringe and the resulting mixture is stirred for 1 h at 24 °C. PhI=NTs (373 mg, 1.00 mmol) is added portionwise to the mixture over 2 h. After the addition, the reaction mixture is kept stirring for another 3 h. The aziridine product is obtained after flash silica gel chromatography with a mixture of petroleum ether (60-90 °C) and ethyl acetate (6:1, v/v) as an eluent.

(2*S*,3*R*)-2-Benzoyl-3-phenyl-1-(*p*-toluenesulfonyl)aziridine (3a) (Entry 3):



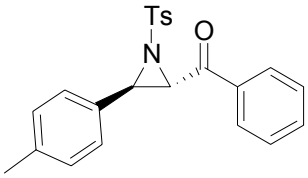
Colorless crystal, yield 80%; mp 112-114 °C [Lit[5]. (2*R*,3*S*)-enantiomer, 86% ee, mp 109.0-110.5], TLC ethyl acetate: petroleum 1:5 (v/v), R_f 0.20; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 30.5 min; τ_{minor} = 35.9 min). $[\alpha]^{20}_D$ = +5.70 (*c* 1.00, $CHCl_3$, 96% ee) [Lit[3]. (2*R*,3*S*)-enantiomer, $[\alpha]^{24}_D$ = -5.24 (*c* 0.85, $CHCl_3$, 86% ee)]; IR (KBr) *v* cm^{-1} 1688 (s), 1332 (s), 1161 (s); ^{1}H NMR δ 2.40 (s, 3H, CH_3), 4.29 (d, *J* = 5.2 Hz, 1H, CH), 4.52 (d, *J* = 5.2 Hz, 1H, CH), 7.22-7.26 (m, 2H, ArH), 7.34-7.38 (m, 5H, ArH), 7.46-7.51 (m, 2H, ArH), 7.60-7.65 (m, 1H, ArH), 7.72 (d, *J* = 8.5 Hz, 2H, ArH), 8.06 (d, *J* = 8.5 Hz, 2H, ArH); ^{13}C NMR δ 21.60, 47.47, 50.18, 127.51, 127.67, 128.63, 128.78, 128.93, 129.46, 132.88, 134.10, 135.92, 136.56, 144.36, 190.33. MS (EI) *m/z* (relative intensity, %): 377 (M^+, 6), 221 (M^+-Ts, 99), 105 ($PhCO^+$, 100), 77 (Ph^+, 79). Anal. Calcd for $C_{22}H_{19}NO_3S$: C, 70.00; H, 5.07; N, 3.71. Found: C, 70.06; H, 5.12; N, 3.58.

(2*R*,3*S*)-2-Benzoyl-3-phenyl-1-(*p*-toluenesulfonyl)aziridine (3'a) (Entry 9):



Colorless crystal, yield 38%; mp 109-111 °C; [Lit[5]. mp 109.0-110.5, 86% ee], TLC ethyl acetate: petroleum 1:5 (v/v), R_f 0.20; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{minor} = 28.9 min; τ_{major} = 33.1 min). $[\alpha]^{20}_D$ = -5.21 (*c* 1.01, $CHCl_3$, 86% ee) [Lit[3]. $[\alpha]^{24}_D$ = -5.24 (*c* 0.85, $CHCl_3$, 86% ee)].

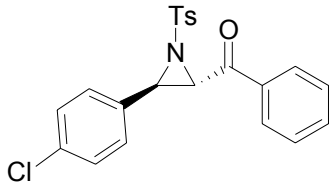
(2*S*,3*R*)-2-Benzoyl-3-(*p*-tolyl)-1-(*p*-toluenesulfonyl)aziridine (3b):



Colorless crystal, yield 86%; mp 133-135 ^{o}C; TLC ethyl acetate: petroleum 1:6 (v/v), R_f 0.20; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 23.7 min; τ_{minor} = 27.4 min): $[\alpha]^{20}_{D}$ = -3.09 (*c* 0.91, $CHCl_3$, 98% ee); IR (KBr) *v* cm^{-1} 1687 (s), 1332 (s), 1162 (s); ^{1}H NMR δ 2.35 (s, 3H, CH_3), 2.40 (s, 3H, CH_3), 4.33 (d, *J* = 4.3 Hz, 1H, CH), 4.46 (d, *J* = 4.3 Hz, 1H, CH), 7.15 (d, *J* = 7.8 Hz, 2H, ArH), 7.22-7.26 (m, 4H, ArH), 7.46-7.51 (m, 2H, ArH, 7.60-7.62 (m, 1H, ArH), 7.72 (d, *J* = 8.1 Hz, 2H, ArH), 8.04-8.06 (m, 2H, ArH); ^{13}C NMR δ 21.24, 21.60, 47.89, 49.74, 127.66, 128.79, 128.89, 129.31, 129.45, 129.57, 134.06, 135.90, 136.70, 138.89, 144.29, 190.53. MS (EI) *m/z* (relative intensity, %): 391 (M^+, 14), 235 (M^+-Ts-H, 100), 130 (M^+-Ts-H-PhCO, 16) 105 ($PhCO^+$, 65), 91 ($CH_3C_6H_4^+$, 35), 77 (Ph^+, 58). Anal. Calcd for $C_{23}H_{21}NO_3S$: C, 70.56; H, 5.41; N, 3.58. Found: C, 70.51; H, 5.47; N, 3.40.

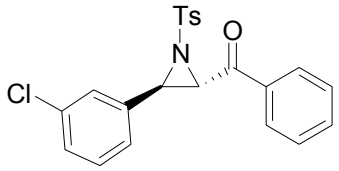
Note: 2-(*p*-Toluoyl)-3-(*p*-tolyl)-1-(*p*-toluenesulfonyl)aziridine (**3b**) was assigned as (2*S*,3*R*) configuration based on the same reaction mechanism and relative retention times although it has a minus sign of specific rotation.

(2*S*,3*R*)-2-Benzoyl-3-(4-chlorophenyl)-1-(*p*-toluenesulfonyl)aziridine (3c):



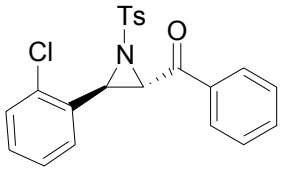
Colorless crystal, yield 70%; mp 153-154 ^{o}C ; TLC ethyl acetate: petroleum 1:6, R_f 0.26; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 32.0 min; τ_{minor} = 47.7 min): $[\alpha]^{20}_{D}$ +3.11 (*c* 1.03 , $CHCl_3$, 76% ee); IR (KBr) *v* cm^{-1} 1688 (s), 1332 (s), 1162 (s); ^{1}H NMR δ 2.40 (s, 3H, CH_3), 4.24 (d, *J* = 4.3 Hz, 1H, CH), 4.48 (d, *J* = 4.3 Hz, 1H, CH), 7.22-7.31 (m, 6H, ArH), 7.45-7.52 (m, 2H, ArH), 7.59-7.63 (m, 1H, ArH), 7.69-7.73 (m, 2H, ArH), 8.01-8.06 (m, 2H, ArH); ^{13}C NMR δ 21.56, 46.62, 50.20, 127.72, 128.81, 128.88, 128.91, 128.95, 129.56, 131.56, 134.13, 134.91, 135.93, 136.53, 144.54, 190.02. MS (EI) *m/z* (relative intensity, %): 411 (M^+, 6), 255 (M^+-Ts-H, 100), 105 ($PhCO^+$, 78), 77 (Ph^+, 70). Anal. Calcd for $C_{22}H_{18}ClNO_3S$: C, 64.15; H, 4.40; N, 3.40. Found: C, 64.12; H, 4.44; N, 3.14.

(2*S*,3*R*)-2-Benzoyl-3-(3-chlorophenyl)-1-(*p*-toluenesulfonyl)aziridine (3d):



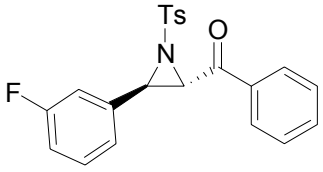
Colorless crystal, yield 76%; mp 140-142 ^{o}C ; TLC ethyl acetate: petroleum 1:6 (v/v), R_f 0.33; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 28.6 min; τ_{minor} = 38.1 min): $[\alpha]^{20}_{D}$ = +12.2 (*c* 1.06, $CHCl_3$, 84% ee); IR (KBr) *v* cm^{-1} 1689 (s), 1333 (s), 1161 (s); ^{1}H NMR δ 2.41 (s, 3H, CH_3), 4.20 (d, *J* = 4.2 Hz, 1H, CH), 4.50 (d, *J* = 4.2 Hz, 1H, CH), 7.21-7.23 (m, 1H, ArH), 7.25-7.32 (m, 5H, ArH), 7.46-7.52 (m, 2H, ArH), 7.61-7.66 (m, 1H, ArH), 7.69-7.72 (m, 2H, ArH), 8.03-8.06 (m, 2H, ArH); ^{13}C NMR δ 21.63, 46.04, 50.48, 125.70, 127.37, 127.76, 128.81, 128.98, 129.03, 129.58, 129.91, 134.20, 134.63, 135.21, 135.81, 136.16, 144.65, 189.87. MS (EI) *m/z* (relative intensity, %): 411 (M^+, 5), 255 (M^+-Ts-H, 100), 105 ($PhCO^+$, 67). Anal. Calcd for $C_{22}H_{18}ClNO_3S$: C, 64.15; H, 4.40; N, 3.40. Found: C, 64.11; H, 4.30; N, 3.35.

(2*S*,3*R*)-2-Benzoyl-3-(2-chlorophenyl)-1-(*p*-toluenesulfonyl)aziridine (3e):



Colorless crystal, yield 91%; mp 129-130 ^{o}C ; TLC ethyl acetate: petroleum 1:5 (v/v), R_f 0.33; The ee of the product was determined by HPLC with Chiralpak AS column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 67.2 min; τ_{minor} = 83.2 min): $[\alpha]^{20}_{D}$ = +12.3 (*c* 1.06, $CHCl_3$, 79% ee); IR (KBr) *v* cm^{-1} 1688 (s), 1334 (s), 1162 (s); ^{1}H NMR δ 2.42 (s, 3H, CH_3), 4.21 (d, *J* = 4.0 Hz, 1H, CH), 4.78 (d, *J* = 4.0 Hz, 1H, CH), 7.20-7.29 (m, 5H, ArH), 7.34-7.38 (m, 1H, ArH), 7.45-7.52 (m, 2H, ArH), 7.59-7.66 (m, 1H, ArH), 7.75 (d, *J* = 8.4 Hz, 2H, ArH), 8.07 (d, *J* = 7.0 Hz, 2H, ArH); ^{13}C NMR δ 21.62, 45.59, 49.42, 126.86, 127.92, 128.24, 128.75, 128.99, 129.46, 129.54, 129.90, 131.45, 134.02, 134.84, 136.04, 144.55, 155.02, 189.73. MS (EI) *m/z* (relative intensity, %): 411 (M^+, 21), 376 (M^+-Cl, 72), 255 (M^+-Ts-H, 100), 220 (M^+-Ts-H-Cl, 6), 150 (M^+-Ts-H-PhCO, 14), 105 ($PhCO^+$, 61), 77 (Ph^+, 78). Anal. Calcd for $C_{22}H_{18}ClNO_3S$: C, 64.15; H, 4.40; N, 3.40. Found: C, 63.97; H, 4.32; N, 3.18.

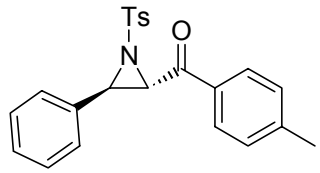
(2*S*,3*R*)-2-Benzoyl-3-(3-fluorophenyl)-1-(*p*-toluenesulfonyl)aziridine (3f):



Colorless crystal, yield 85%; mp 108-111 ^{o}C ; TLC ethyl acetate: petroleum 1:6 (v/v), R_f 0.18; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 27.1 min; τ_{minor} = 36.2 min): $[\alpha]^{20}_{D}$ = +12.0 (*c* 1.03, $CHCl_3$, 71% ee); IR (KBr) *v* cm^{-1} 1684 (s), 1333 (s), 1161 (s); ^{1}H NMR δ 2.41 (s, 3H, CH_3), 4.19 (d, *J* = 4.2 Hz, 1H, CH), 4.53 (d, *J* = 4.2 Hz,

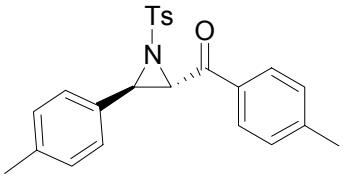
1H, CH), 6.98-7.06 (m, 2H, CH), 7.13-7.17 (m, 1H, ArH), 7.23-7.33 (m, 3H, ArH), 7.45-7.53 (m, 2H, ArH), 7.60-7.65 (m, 1H, ArH), 7.70-7.74 (m, 2H, ArH), 8.02-8.06 (m, 2H, ArH); ^{13}C NMR δ 21.59, 46.22, 50.71, 114.22 (d, $^{2}J_{F\text{-}C\text{-}C}$ = 22.9 Hz), 115.88 (d, $^{2}J_{F\text{-}C\text{-}C}$ = 21.0 Hz), 123.26, 123.31, 127.76, 128.81, 128.99, 129.58, 130.37, 133.00 (d, $^{1}J_{F\text{-}C}$ = 288.8 Hz), 134.13, 135.98, 136.49, 144.57, 189.88. MS (EI) *m/z* (relative intensity, %): 395 (M^+, 5), 240 (M^+-Ts, 48), 105 ($PhCO^+$, 100), 77 (Ph^+, 67). Anal. Calcd for $C_{22}H_{18}FNO_3S$: C, 66.82; H, 4.59; N, 3.54. Found: C, 66.81, H, 4.72, N, 3.40.

(2*S*,3*R*)-3-Phenyl-2-(*p*-toluoyl)-1-(*p*-toluenesulfonyl)aziridine (3g):



Colorless needle crystal, yield 92%; mp 138-139 ^{o}C; TLC ethyl acetate: petroleum 1:6 (v/v), R_f 0.23; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 27.8 min): $[\alpha]^{20}_{D}$ = +4.00 (*c* 0.93, $CHCl_3$, >99.9% ee); IR (KBr) *v* cm^{-1} 1683 (s), 1331 (s), 1162 (s); ^{1}H NMR δ 2.40 (s, 3H, CH_3), 2.43 (s, 3H, CH_3), 4.28 (d, *J* = 4.2 Hz, 1H, CH), 4.50 (d, *J* = 4.2 Hz, 1H, CH), 7.20 (m, 3H, ArH), 7.24-7.26 (m, 1H, ArH), 7.30-7.33 (m, 5H, ArH), 7.72 (d, *J* = 8.6 Hz, 2H, ArH), 7.96 (d, *J* = 8.2 Hz, 2H, ArH); ^{13}C NMR δ 21.55, 21.76, 47.48, 50.13, 127.56, 127.71, 128.58, 128.80, 129.06, 129.45, 129.46, 133.02, 133.62, 136.76, 144.25, 145.14, 189.76. MS (EI) *m/z* (relative intensity, %): 391 (M^+, 13), 236 (M^+-Ts, 77), 119 ($CH_3C_6H_4CO^+$, 100), 91 ($CH_3C_6H4^+$, 68), 77 (Ph^+, 10). Anal. Calcd for $C_{23}H_{21}NO_3S$: C, 70.56; H, 5.41; N, 3.58. Found: C, 70.50; H, 5.40; N, 3.32.

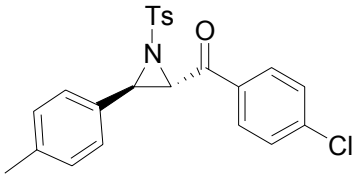
(2*S*,3*R*)-2-(*p*-Toluoyl)-3-(*p*-tolyl)-1-(*p*-toluenesulfonyl)aziridine (3h):



Colorless crystal, yield 59%; mp 168-170 ^{o}C; TLC ethyl acetate: petroleum 1:5 (v/v), R_f 0.27; The ee of the product was determined by HPLC with Chiralcel OD column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.8 mL/min (τ_{major} = 24.5 min); $[\alpha]^{20}_{D}$ = –5.83 (*c* 0.60, $CHCl_3$, > 99.9% ee); IR (KBr) *v* cm^{-1} 1680 (s), 1331 (s), 1161 (s); ^{1}H NMR δ 2.34 (s, 3H, CH_3), 2.39 (s, 3H, CH_3), 2.43 (s, 3H, CH_3), 4.32 (d, *J* = 4.4 Hz, 1H, CH), 4.44 (d, *J* = 4.4 Hz, 1H, CH), 7.14 (d, *J* = 8.1 Hz, 2H, ArH), 7.22 (d, *J* = 5.4 Hz, 2H, ArH), 7.24 (d, *J* = 5.4 Hz, 2H, ArH), 7.28 (d, *J* = 8.4 Hz, 2H, ArH), 7.72 (d, *J* = 8.4 Hz, 2H, ArH), 7.95 (d, *J* = 8.1 Hz, 2H, ArH); ^{13}C NMR δ 21.24, 21.60, 21.81, 47.86, 49.73, 127.63, 129.02, 129.28, 129.45, 129.65, 133.49, 136.75, 138.82, 144.22, 145.17, 189.98. MS (EI) *m/z* (relative intensity, %): 405 (M^+, 36), 249 (M^+-Ts-H, 100), 119 ($CH_3C_6H_4CO^+$, 67), 91 ($CH_3C_6H_4^+$, 75). Anal. Calcd for $C_{24}H_{23}NO_3S$: C, 71.09; H, 5.72; N, 3.45. Found: C, 70.98; H, 5.77; N, 3.28.

Note: 2-(*p*-Toluoyl)-3-(*p*-tolyl)-1-(*p*-toluenesulfonyl)aziridine (**3h**) was assigned as (2*S*,3*R*) configuration based on the same reaction mechanism and relative retention times in chiral column although it has a minus sign of specific rotation.

(2*S*,3*R*)-2-(4-Chlorobenzoyl)-3-(*p*-tolyl)-1-(*p*-toluenesulfonyl)azridine (3i):



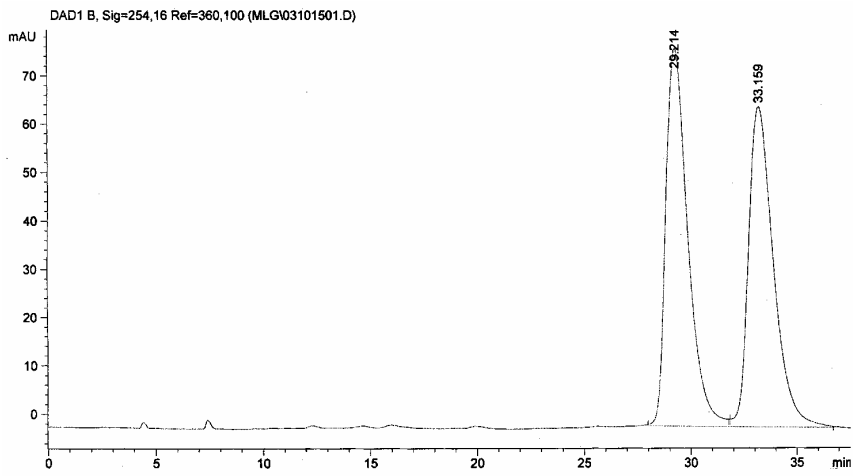
Colorless needle crystal, yield 51%; mp 159-161 ^{o}C ; TLC ethyl acetate: petroleum 1:6 (v/v), R_f 0.39; The ee of the product was determined by HPLC with chiral OD-H column with hexane/2-propanol (90:10, v/v) as an eluent at flow rate 0.5 mL/min (τ_{major} = 39.9 min; τ_{minor} = 45.5 min): $[\alpha]^{20}_{D}$ = +2.00 (*c* 0.55, $CHCl_3$, 68% ee); IR (KBr) *v* cm^{-1} 1686 (s), 1325 (s), 1161 (s); ^{1}H NMR δ 2.34 (s, 3H, CH_3), 2.41 (s, 3H, CH_3), 4.21 (d, *J* = 4.5 Hz, 1H, CH), 4.47 (d, *J* = 4.5 Hz, 1H, CH), 7.12- 7.26 (m, 6H, ArH), 7.45 (d, *J* = 9.0 Hz, 2H, ArH), 7.71 (d, *J* = 8.2 Hz, 2H, ArH), 8.00 (d, *J* = 8.6 Hz, 2H, ArH); ^{13}C NMR δ 21.22, 21.60, 47.51, 49.87, 127.41, 127.63, 128.87, 129.11, 129.32, 129.50, 130.27, 134.24, 136.47, 138.91, 140.64, 144.45, 189.39. MS (EI) *m/z* (relative intensity, %): 425 (M^+, 46), 270 (M^+-Ts, 55), 139 ($ClC_6H_4CO^+$, 100), 91 ($CH_3C_6H_4^+$, 26). Anal. Calcd for $C_{23}H_{20}ClNO_3S$: C, 64.86; H, 4.73; N, 3.29. Found: C, 64.95; H, 4.82; N, 2.97.

References

1. Dippy, J. F. J.; Lewis, R. H. *Rec. Trav. Chim*. **1937**, *56*, 1000-1006.
2. Yamada, Y.; Yamamoto, T.; Okawara, M. *Chem. Lett.* **1975,** 361-362.
3. Roger, M. E.; Averill, B. A. *J. Org. Chem.*, **1986**, *51*, 3308-3314.
4. Waldmann, H.; Stengl, R. *Chem. Ber.*, **1950**, *83*, 167-170.
5. Suga, H.; kakehi, A.; Ito, S.; Ibata, T.; Fudo, T.; Watanabe, Y.; Kinoshita, Y. *Bull. Chem. Soc. Jpn.* **2003**, *76*, 189-199.

3a

Racemate



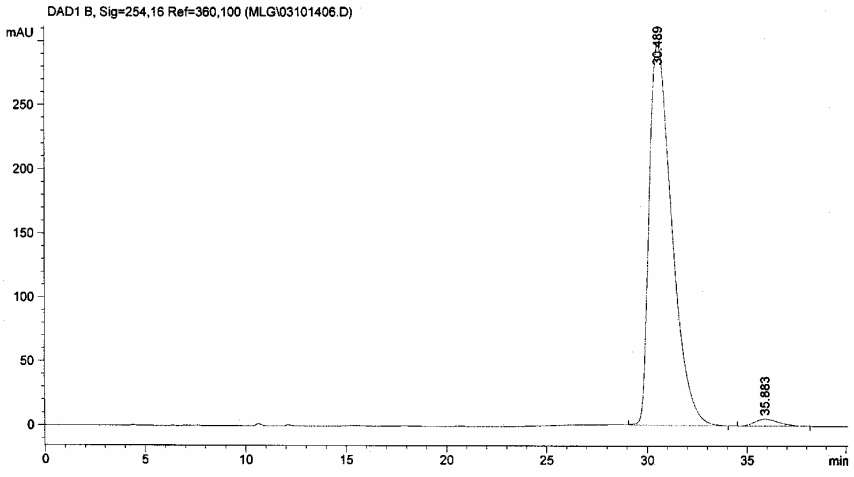
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.214	BB	1.0909	5594.80225	78.18256	50.6270
2	33.159	BB	1.2603	5456.21777	66.28526	49.3730
Totals :				1.10510e4	144.46781	

(2*S*,3*R*)-Enantiomer (major)



Area Percent Report

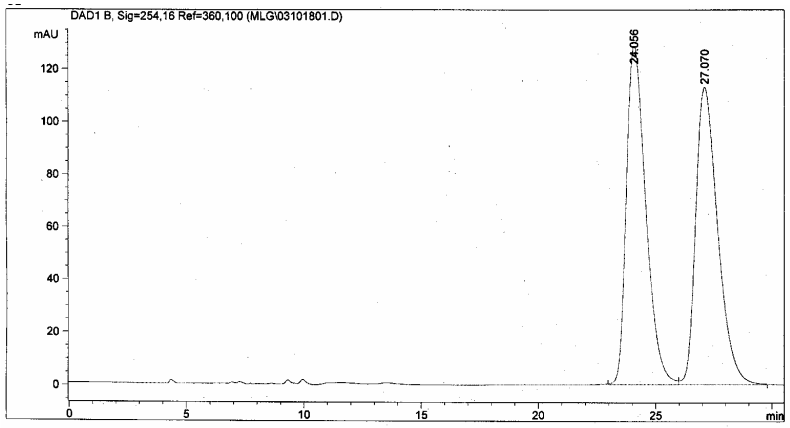
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	30.489	BB	1.2089	2.32998e4	296.45493	98.0230
2	35.883	PB	1.2527	469.91885	5.43495	1.9770
Totals :				2.37698e4	301.88987	

3b

Racemate



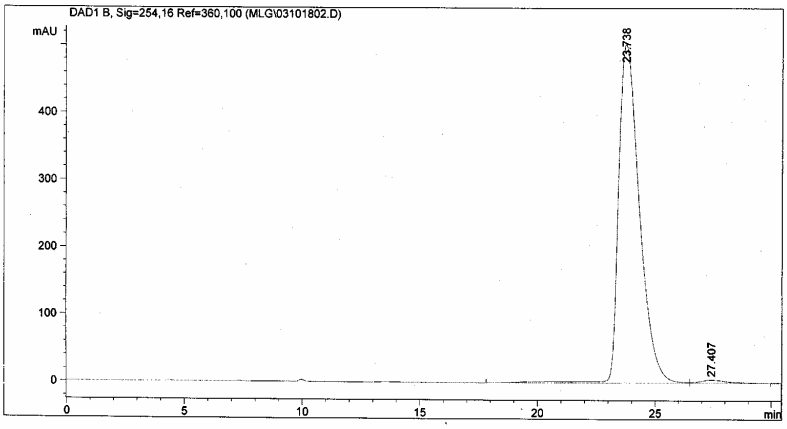
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	24.056	BV	0.8885	7396.33008	128.31975	49.8960
2	27.070	VB	1.0214	7427.16895	112.61421	50.1040
Totals :				1.48235e4	240.93396	

(2*S*,3*R*)-Enantiomer



Area Percent Report

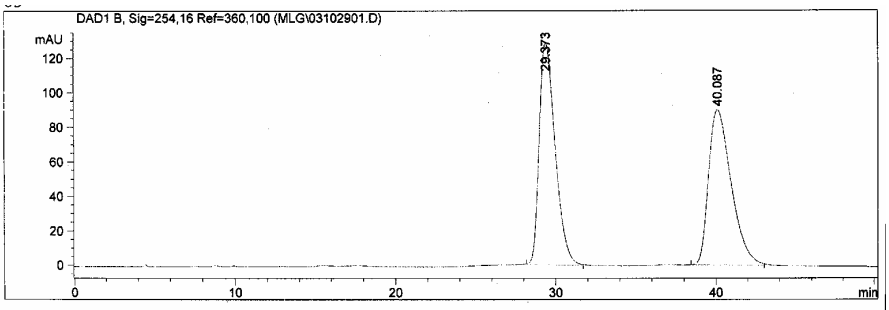
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	23.738	BV	0.9501	3.11078e4	502.60941	98.9505
2	27.407	VB	0.9942	329.95178	4.59240	1.0495
Totals :				3.14377e4	507.20180	

3c

Racemate



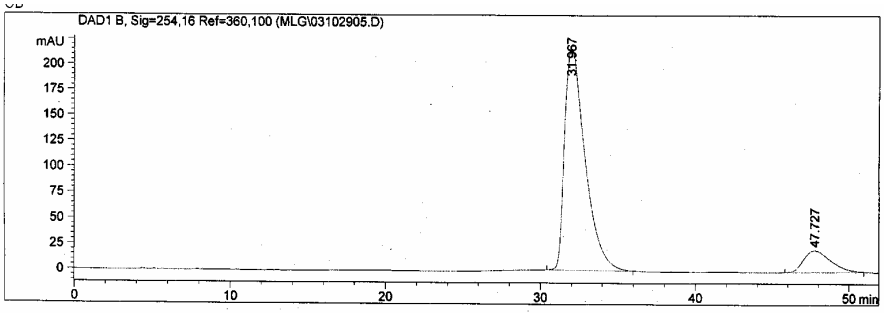
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	29.373	BB	1.0761	8951.78418	128.59756	50.1407
2	40.087	BB	1.5202	8901.55078	90.13540	49.8593
Totals :				1.78533e4	218.73296	

(2*S*,3*R*)-Enantiomer



Area Percent Report

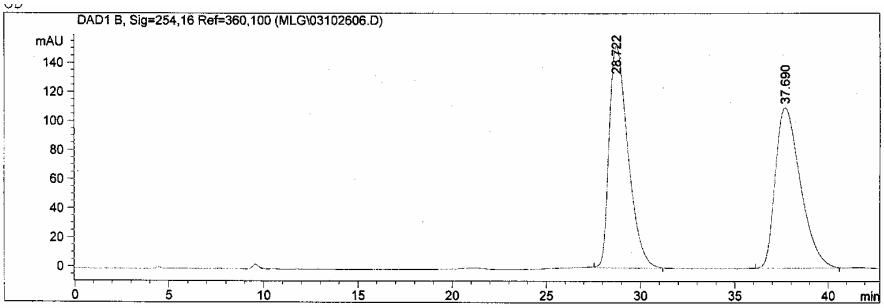
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	31.967	BB	1.3891	1.99454e4	216.85042	88.2261
2	47.727	BB	1.5069	2661.74341	21.37369	11.7739
Totals :				2.26071e4	238.22410	

3d

Racemate



Area Percent Report

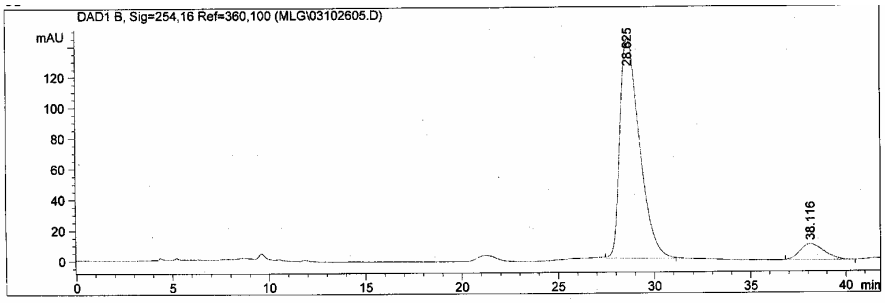
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.722	BB	1.0674	1.05851e4	152.19102	50.6737
2	37.690	BB	1.4583	1.03036e4	110.25172	49.3263

Totals : 2.08887e4 262.44274

(2*S*,3*R*)-Enantiomer



Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

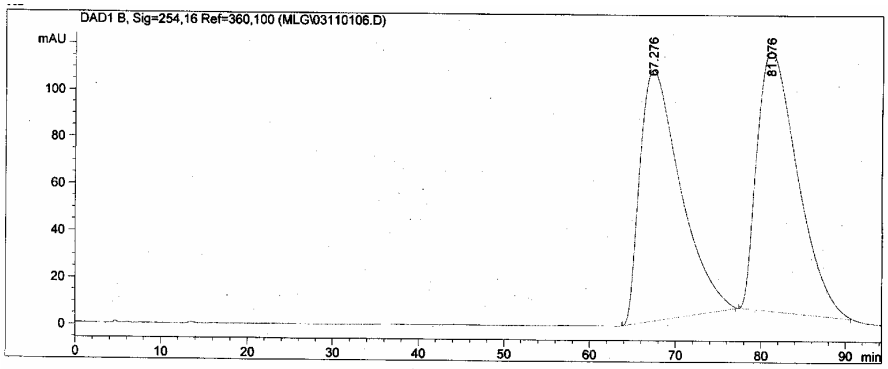
Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	28.625	BB	1.0886	1.01151e4	143.10440	91.7460
2	38.116	BP	1.3770	910.00647	10.35755	8.2540

Totals : 1.10251e4 153.46195

3e

Racemate

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================================================================================
                            Area Percent Report
================================================================================

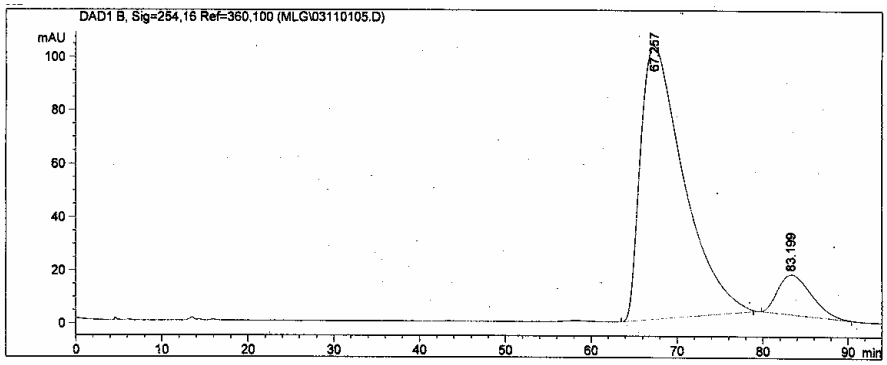
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  67.276 BB    3.8656 3.51550e4   107.09793  48.8130
   2  81.076 BB    3.9023 3.68648e4   111.01113  51.1870

Totals :                  7.20198e4   218.10906
```

(2*S*,3*R*)-Enantiomer

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================================================================================
                            Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

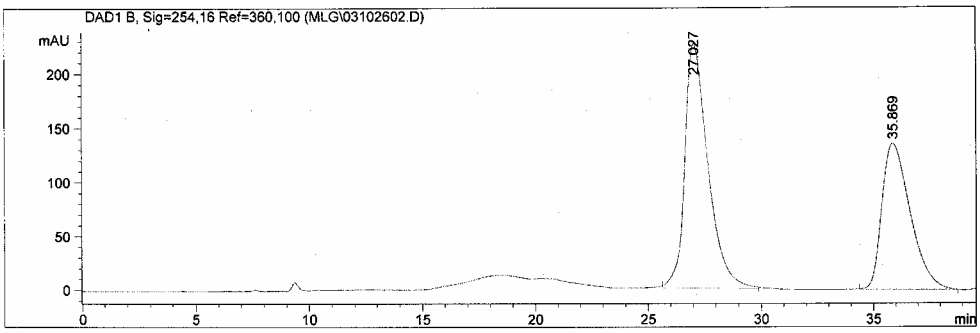
Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  67.257 BB    4.1223 3.58817e4   102.59443  89.5922
   2  83.199 BB    3.2357 4168.34424   15.09465  10.4078

Totals :                  4.00501e4   117.68907
```

3f

Racemate



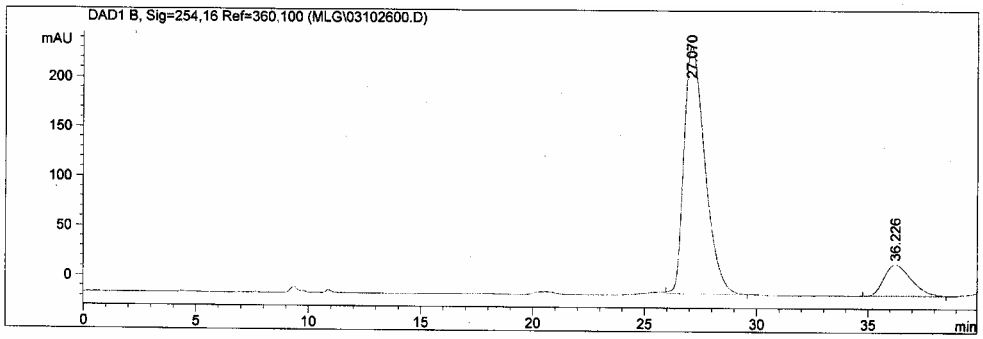
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.027	BB	1.0745	1.61520e4	225.82196	57.4041
2	35.869	BP	1.3709	1.19853e4	135.11165	42.5959
Totals :				2.81373e4	360.93361	

(2*S*,3*R*)-Enantiomer



Area Percent Report

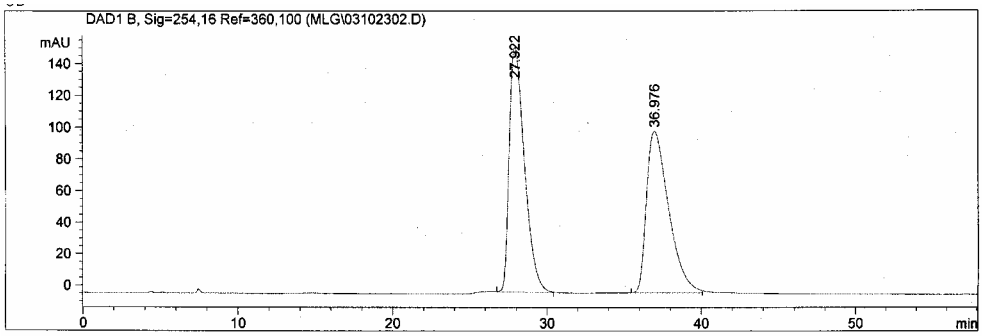
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	27.070	BB	1.0099	1.61933e4	247.97229	85.6869
2	36.226	PP	1.3423	2704.93188	31.35977	14.3131
Totals :				1.88982e4	279.33206	

3g

Racemate

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================================================================================
                          Area Percent Report
================================================================================

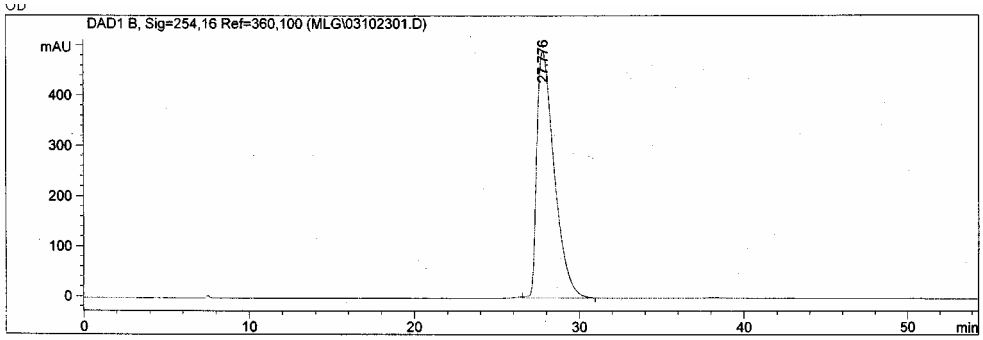
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.922 BB    1.0618 1.06847e4   154.69186  51.0566
   2  36.976 BB    1.5296 1.02425e4   102.16978  48.9434

Totals :                  2.09272e4   256.86164
```

(2*S*,3*R*)-Enantiomer

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================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

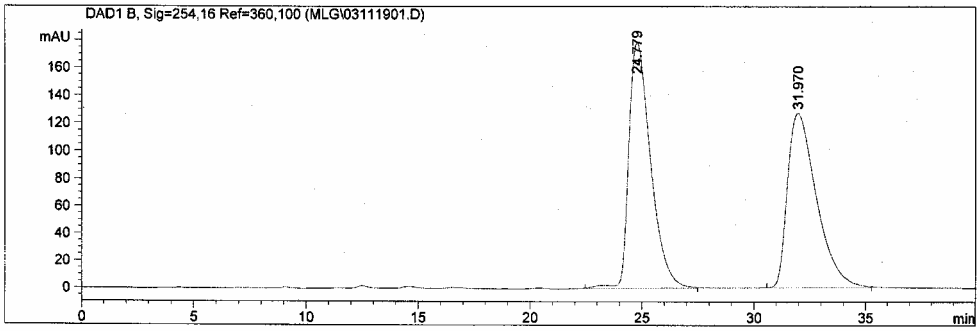
Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.776 BB    1.1344 3.62390e4   490.22571 100.0000

Totals :                  3.62390e4   490.22571
```

3h

Racemate




```
================================================================================
                          Area Percent Report
================================================================================

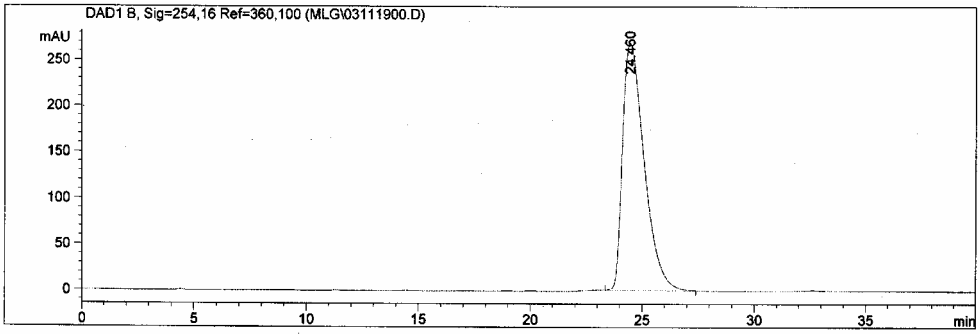
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.779 BB    1.0499 1.22219e4   178.70891  51.4587
   2  31.970 BB    1.4023 1.15290e4   127.09128  48.5413

Totals :                  2.37509e4   305.80019
```

(2*S*,3*R*)-Enantiomer




```
================================================================================
                          Area Percent Report
================================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

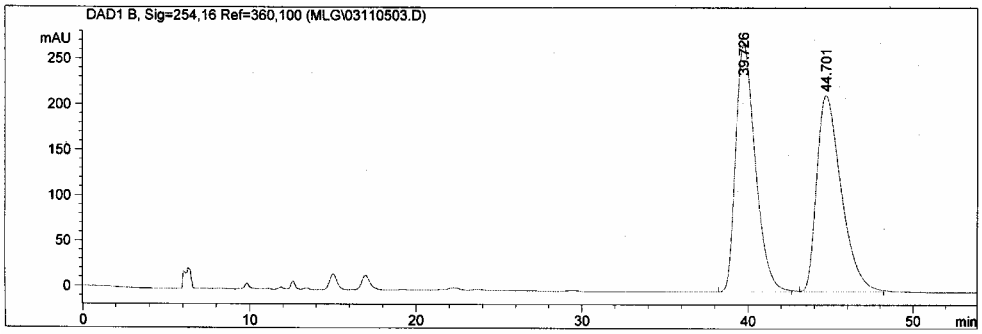

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]    [mAU*s]    [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  24.460 BB    1.0328 1.80803e4   268.78641 100.0000

Totals :                  1.80803e4   268.78641
```

3i

Racemate



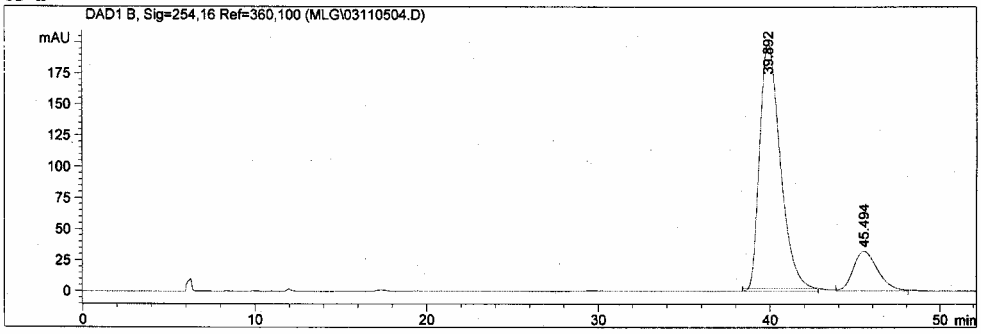
Area Percent Report

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	39.726	BB	1.2775	2.24892e4	271.75940	50.1621
2	44.701	BB	1.5832	2.23438e4	215.94388	49.8379
Totals :				4.48330e4	487.70328	

(2*S*,3*R*)-Enantiomer



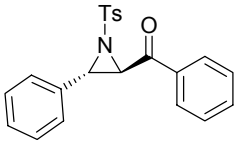
Area Percent Report

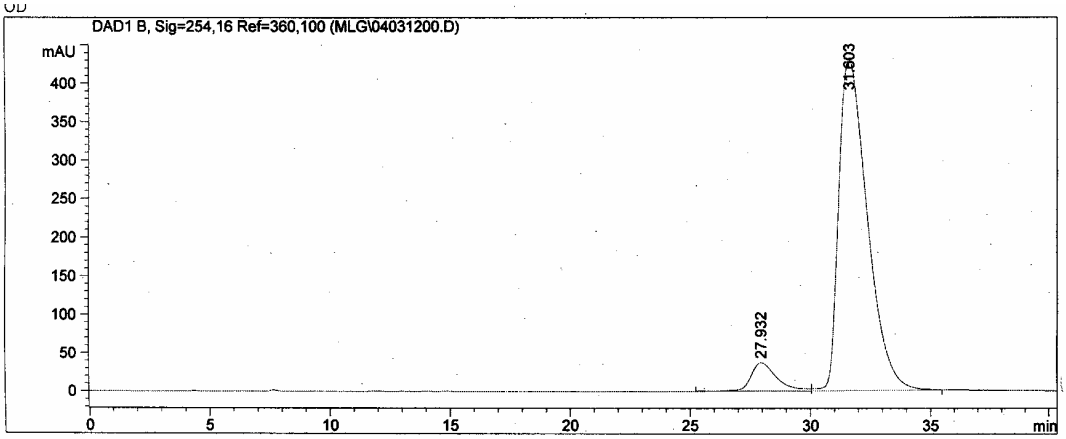
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000

Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	39.892	BB	1.3005	1.68783e4	199.20110	83.9060
2	45.494	BB	1.5554	3237.42017	32.24350	16.0940
Totals :				2.01157e4	231.44460	

3a'



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=====================================================================
                      Area Percent Report
=====================================================================

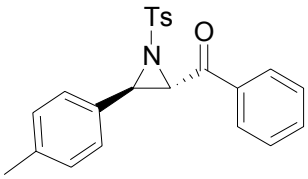
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

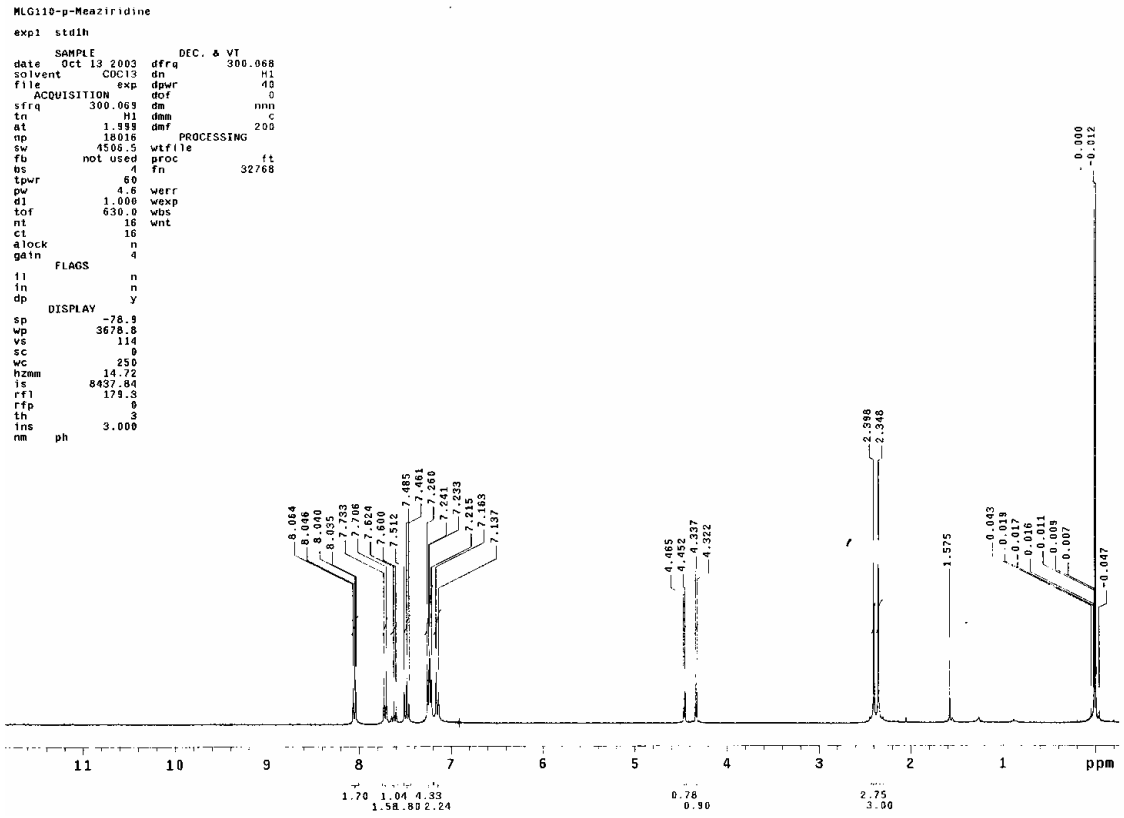
Signal 1: DAD1 B, Sig=254,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]   [mAU*s]     [mAU]       %
----|-------|----|-------|----------|----------|--------|
   1  27.932 PV    1.0976 2769.52002   37.07144   7.2198
   2  31.603 VB    1.2704 3.55907e4   429.66614  92.7802

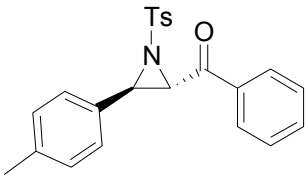
Totals :                  3.83603e4   466.73758
```

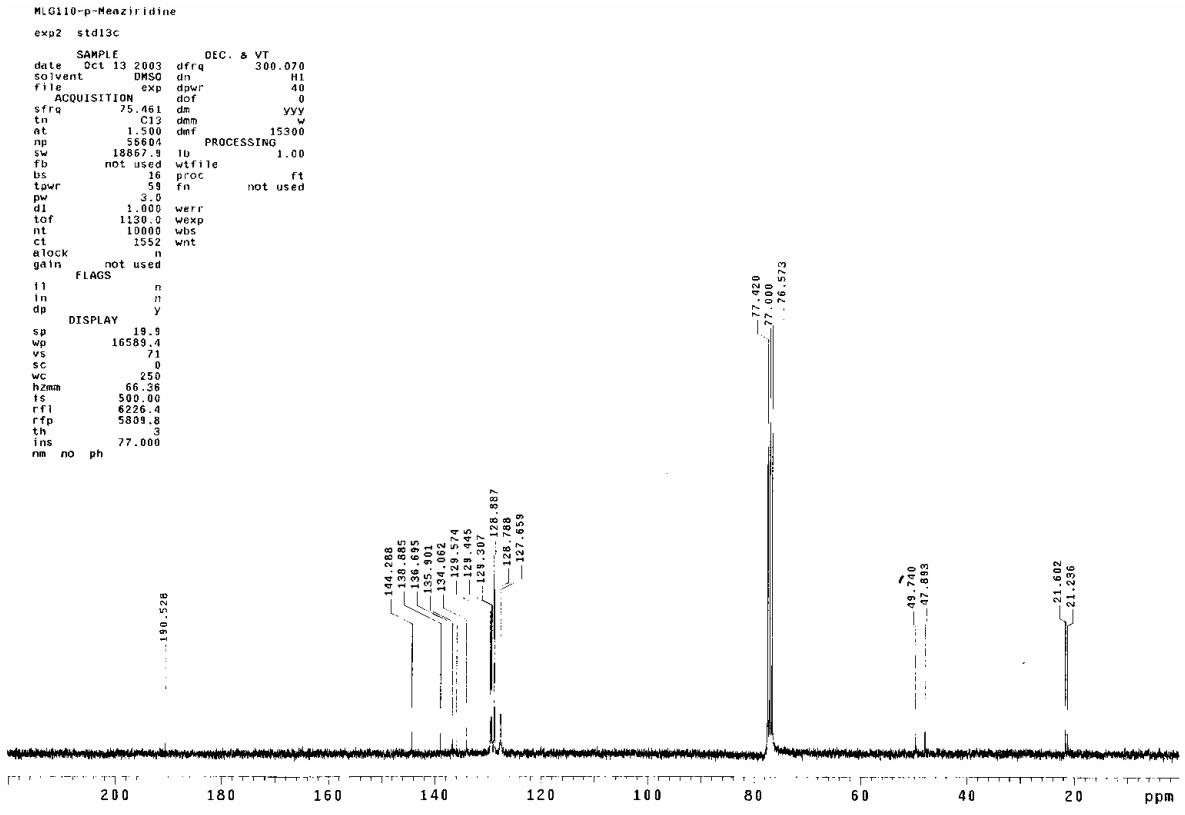
3b


Ts
N
O

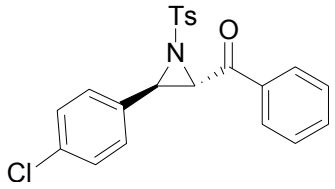

MLG110-p-Meaziridine
exp1 std1h
SAMPLE
date Oct 13 2003
solvent CDCl3
file exp
ACQUISITION
sfrq 300.069
tn H1
at 1.998
np 18016
sw 4506.5
fb not used
bs 4
tpwr 60
pw 4.6
d1 1.000
tof 630.0
nt 16
ct 16
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -78.9
wp 3678.8
vs 114
sc 0
wc 250
hzmm 14.72
is 8437.84
rfl 179.3
rfp 0
th 3
ins 3.000
nm ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 48
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn 32768
werr
wexp
wbs
wnt
8.064
8.046
8.040
8.035
7.733
7.706
7.624
7.600
7.512
7.485
7.461
7.260
7.241
7.233
7.215
7.163
7.137
4.465
4.452
4.337
4.322
2.598
2.348
1.575
0.043
0.019
0.017
0.016
0.011
0.009
0.007
0.000
-0.012
-0.047
11
10
9
8
7
6
5
4
3
2
1
ppm
1.70
1.04
4.33
1.58
1.80
2.24
0.78
0.90
2.75
3.00

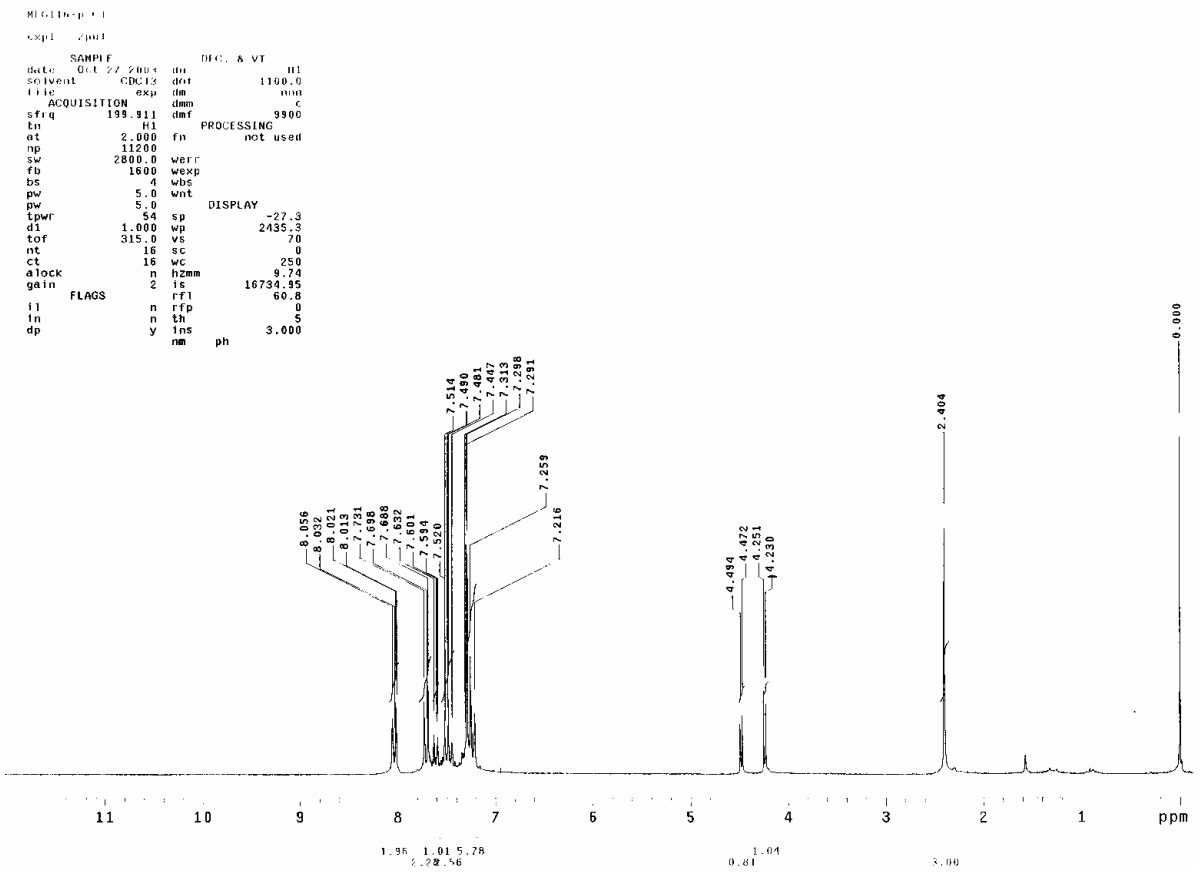
3b


Ts
N
O

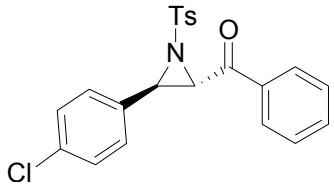

MLG110-p-Meaziridine
exp2 stdl3c
SAMPLE
date Oct 13 2003
solvent DMSO
file exp
ACQUISITION
sfrq 75.461
tn C13
at 1.500
np 58604
sw 18867.8
fb not used
bs 16
tpwr 59
pw 3.0
dl 1.000
tof 1130.0
nt 10000
ct 1552
alock n
gain not used
FLAGS
fl n
in n
dp y
DISPLAY
sp 19.9
wp 16589.4
vs 71
sc 0
wc 250
hzmm 66.38
fs 500.00
rfl 6226.4
rfp 5809.8
th 3
ins 77.000
nm no ph
DEC. & VT
dfrq 300.070
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 15300
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
190.528
144.288
138.885
136.695
135.901
134.062
129.574
129.445
129.307
128.887
128.788
127.659
77.420
77.000
76.573
49.740
47.893
21.602
21.236
200
180
160
140
120
100
80
60
40
20
ppm

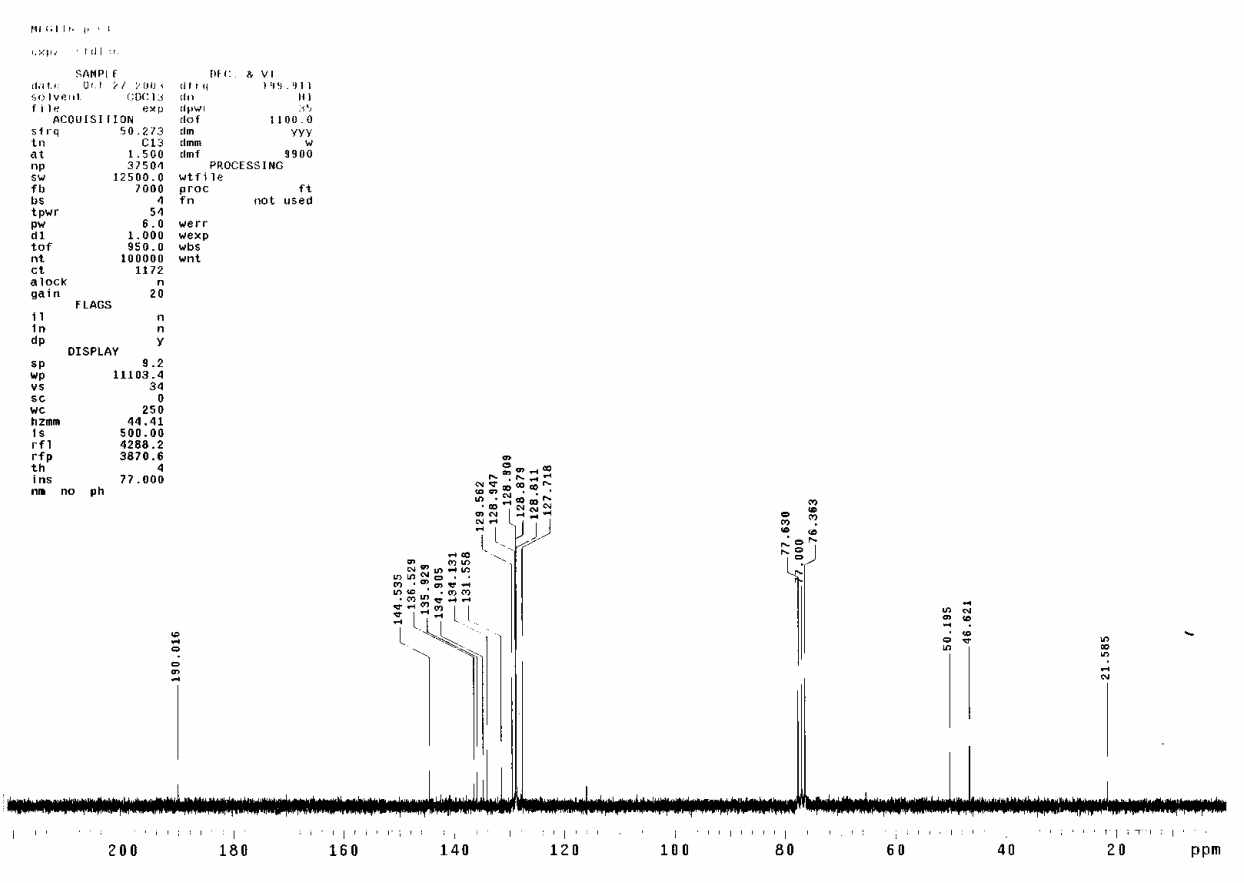
3c


Ts
N
O
Cl

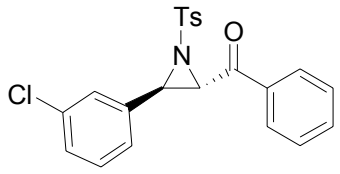

8.056
8.032
8.021
8.013
7.701
7.688
7.632
7.601
7.594
7.520
7.514
7.490
7.481
7.447
7.313
7.298
7.291
7.259
7.216
4.494
4.472
4.251
4.230
2.404
0.000
11
10
9
8
7
6
5
4
3
2
1
ppm
1.96
1.01
5.78
2.56
1.04
0.81
3.00

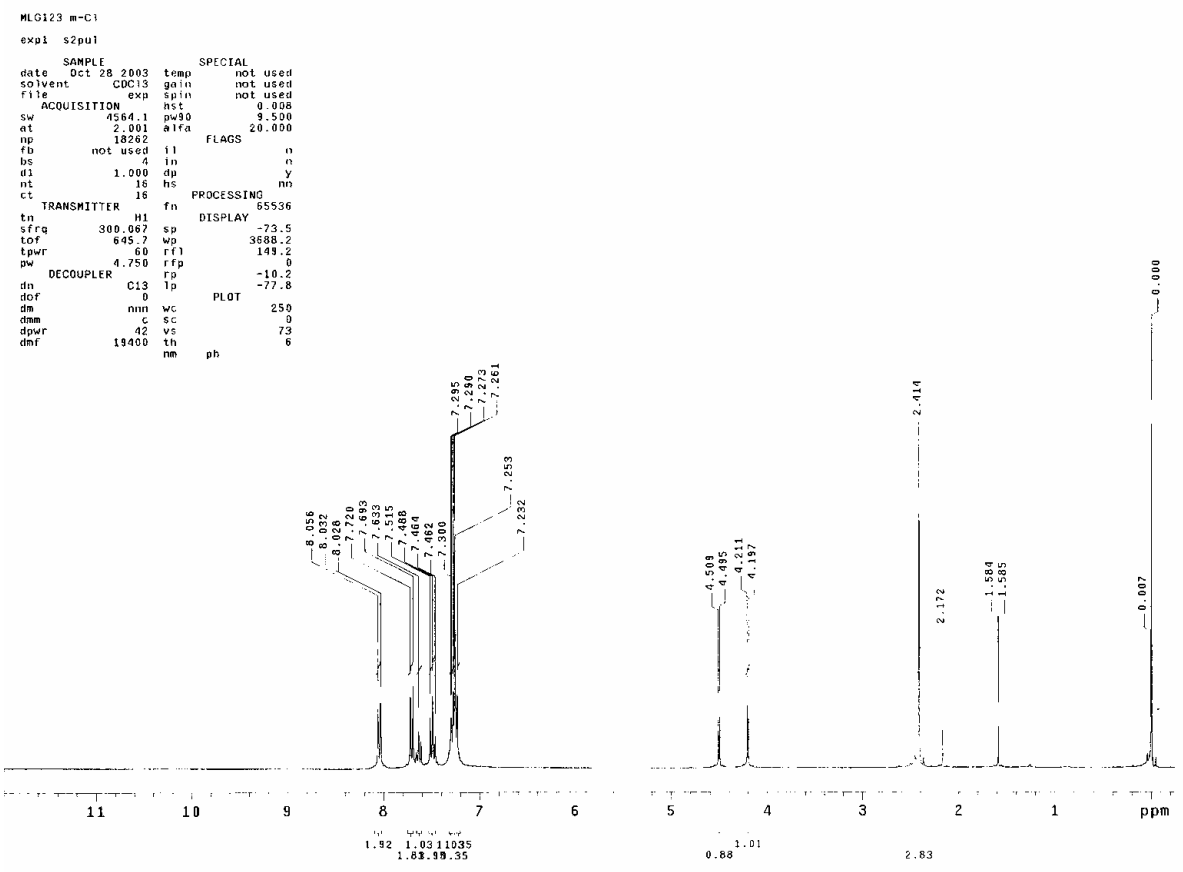
3c


Ts
N
O
Cl

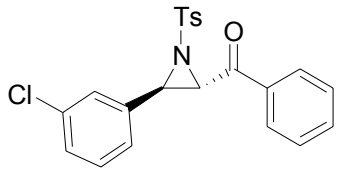

SAMPLE
date Oct 27 2005
solvent CDCl3
file exp
ACQUISITION
sfrq 50.273
tn C13
at 1.500
np 37504
sw 12500.0
fb 7000
bs 4
tpwr 54
pw 6.0
d1 1.000
tof 950.0
nt 100000
ct 1172
alock n
gain 20
FLAGS
il n
in n
dp y
DISPLAY
sp 9.2
wp 11103.4
vs 34
sc 0
wc 250
hzmm 44.41
is 500.00
rfl 4288.2
rfp 3870.6
th 4
ins 77.000
nm no ph
PROC. & FT
dfrq 199.911
dn H1
dpwr 35
dof 1100.0
dm yyy
dmm w
dmf 9900
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
144.535
136.529
135.928
134.905
134.131
131.558
129.562
128.947
128.809
128.879
128.811
127.718
77.630
77.000
76.363
50.195
46.621
21.585
190.016
200
180
160
140
120
100
80
60
40
20
ppm

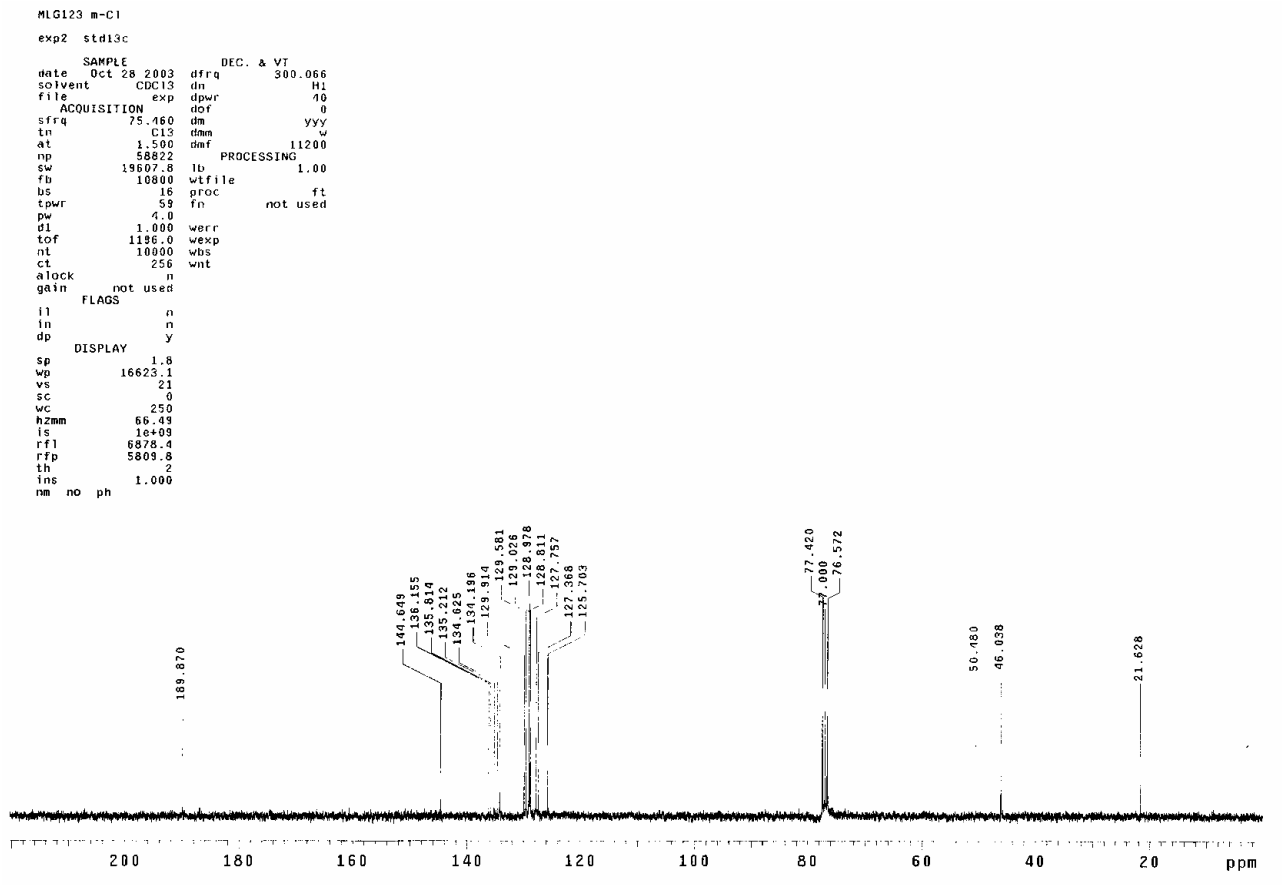
3d


Ts
N
O
Cl

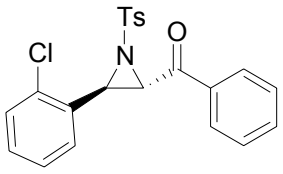

MLG123 m-Cl
expl s2pul
SAMPLE
date Oct 28 2003
solvent CDCl3
file exp
ACQUISITION
sw 4564.1
at 2.001
np 18262
fb not used
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
nst 0.006
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -73.5
wp 3688.2
rfl 149.2
rfp 0
rp -10.2
lp -77.8
PLOT
wc 250
sc 0
vs 73
th 6
nm ph
8.056
8.032
8.026
7.720
7.693
7.633
7.515
7.488
7.464
7.462
7.300
7.295
7.290
7.273
7.261
7.253
7.232
4.509
4.495
4.211
4.197
2.414
2.172
1.584
1.565
0.007
0.000
11
10
9
8
7
6
5
4
3
2
1
ppm
1.92
1.03
1.83
1.99
1.35
0.88
1.01
2.83

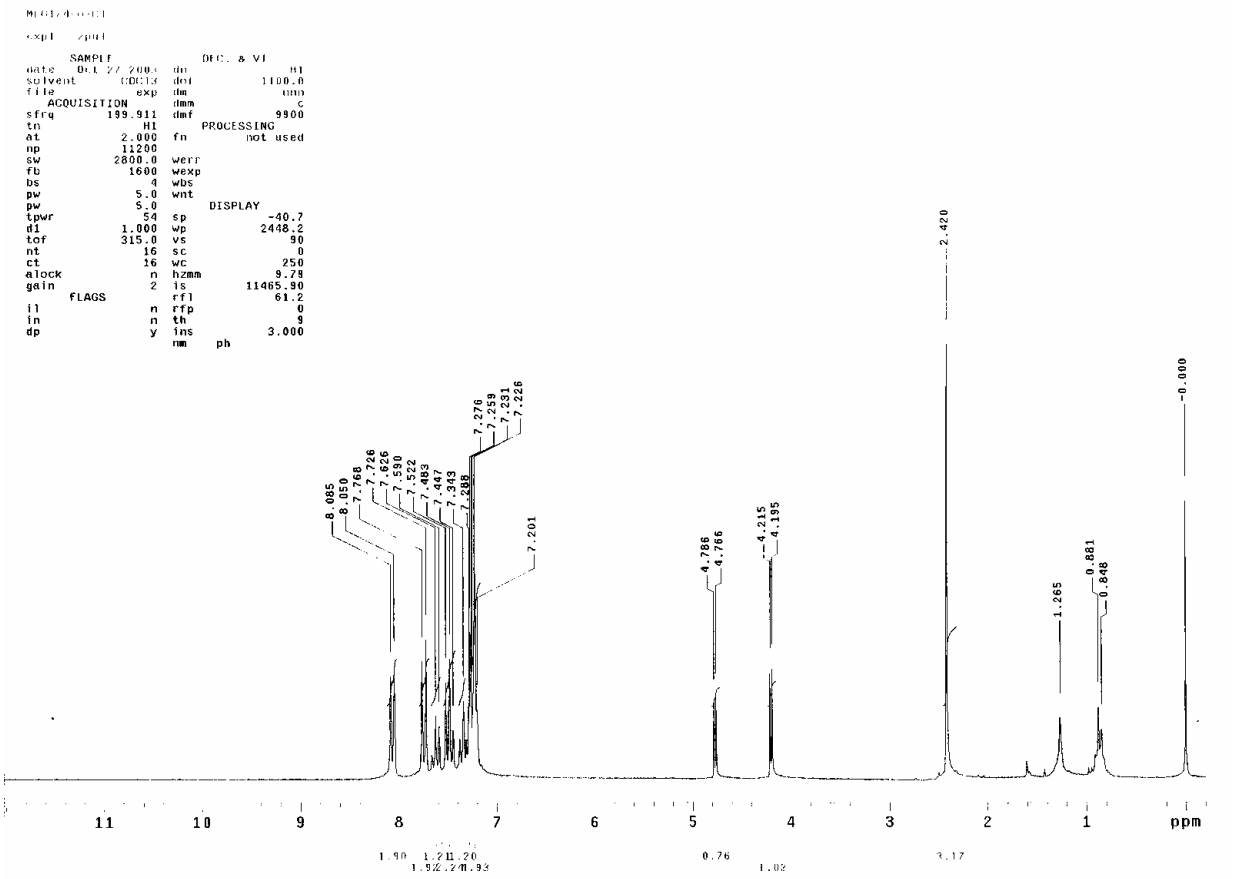
3d


Ts
N
O
Cl

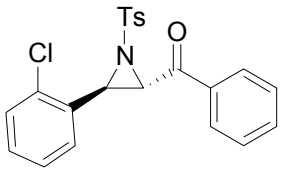

MLG123 m-Cl
exp2 std13c
SAMPLE
date Oct 28 2003
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 16
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 10000
ct 256
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp 1.8
wp 16623.1
vs 21
sc 0
wc 250
hzmm 66.49
is 1e+09
rfl 6878.4
rfp 5809.8
th 2
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
189.870
144.649
136.155
135.814
135.212
134.625
134.196
129.914
129.581
129.026
128.978
128.811
127.757
127.366
125.703
77.420
77.000
76.572
50.480
46.038
21.628
200
180
160
140
120
100
80
60
40
20
ppm

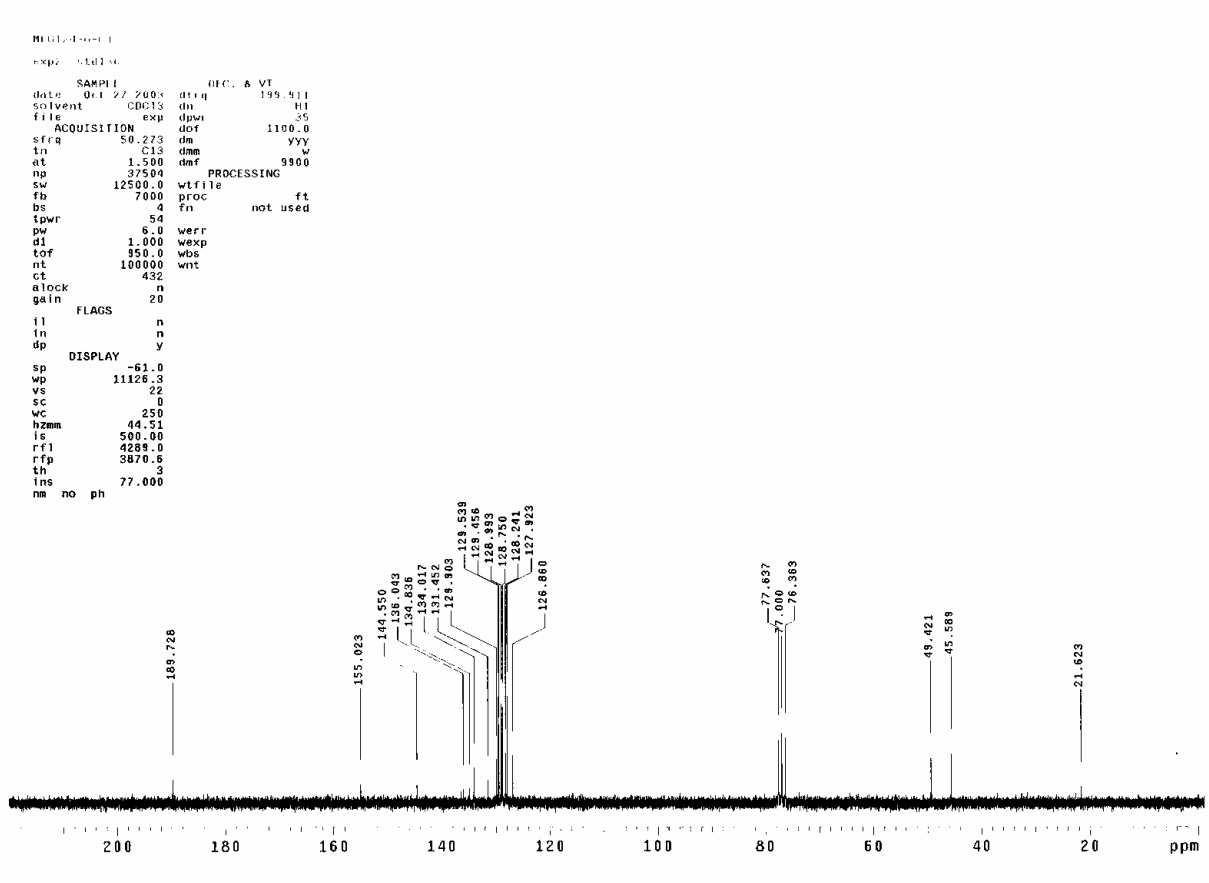
3e


Ts
O
Cl
N

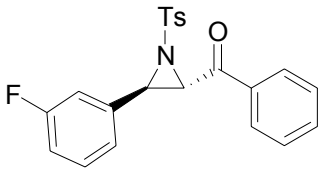

expt zpul
SAMPLE
date Oct 27 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 199.911
tn H1
at 2.000
np 11200
sw 2800.0
fb 1600
bs 4
pw 5.0
pw 5.0
tpwr 54
d1 1.000
tof 315.0
nt 16
ct 16
alock n
gain 2
FLAGS
il n
in n
dp y
DEC. & VT
dn H1
dof 1100.0
dm nnn
dmm c
dmf 9900
PROCESSING
fn not used
werr
wexp
wbs
wnt
DISPLAY
sp -40.7
wp 2448.2
vs 90
sc 0
wc 250
hzmm 9.79
is 11465.90
rfl 61.2
rfp 0
th 9
ins 3.000
mm ph
8.085
8.056
7.768
7.726
7.626
7.590
7.522
7.483
7.447
7.343
7.288
7.276
7.259
7.231
7.226
7.201
4.786
4.766
4.215
4.195
2.420
1.265
0.881
0.848
0.000
11
10
9
8
7
6
5
4
3
2
1
ppm
1.90
1.21
1.20
1.92
2.21
1.93
0.76
1.02
3.17

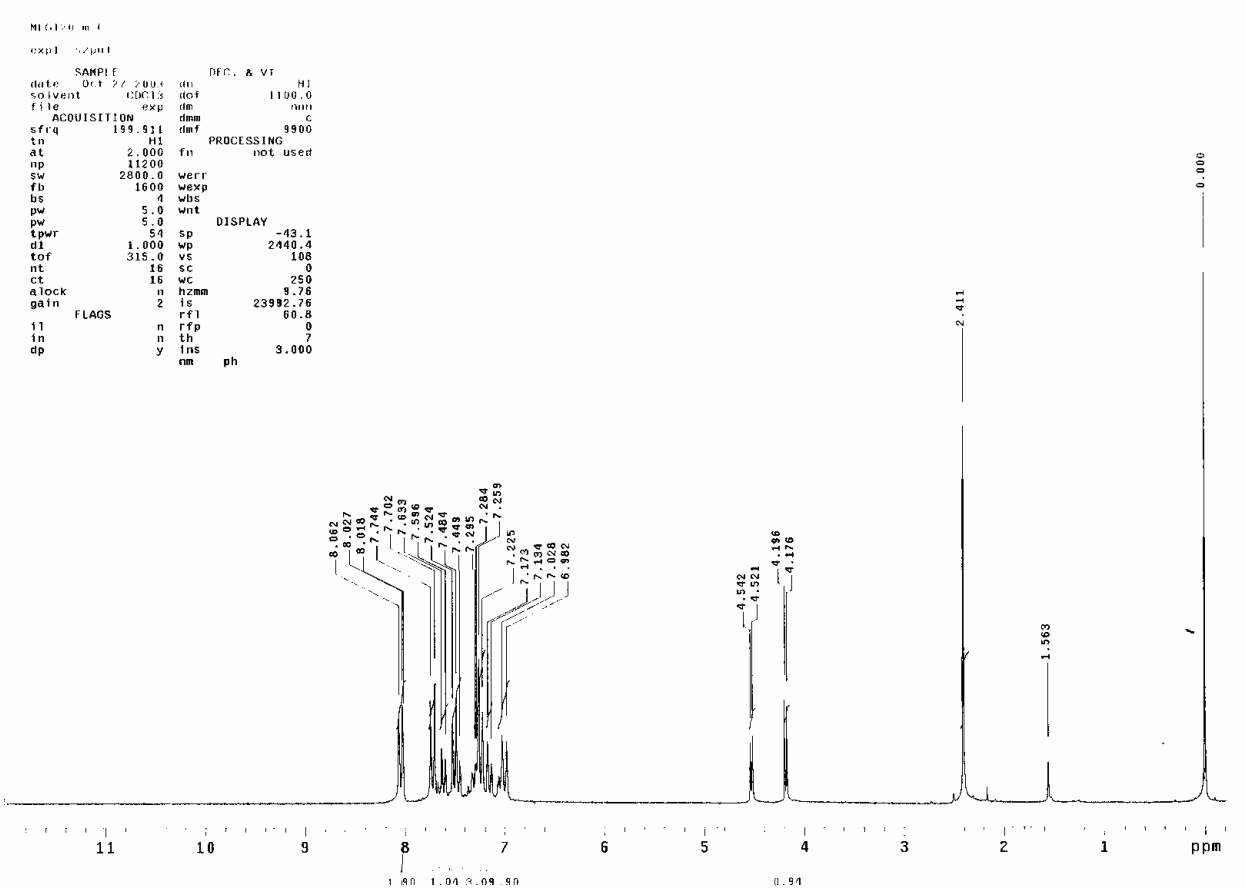
3e


Ts
Cl
N
O

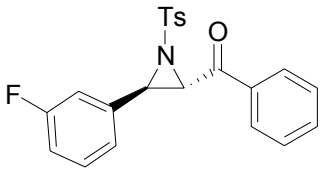

SAMPLE
date Oct 27 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 50.273
tn C13
at 1.500
np 37504
sw 12500.0
fb 7000
bs 4
tpwr 54
pw 6.0
d1 1.000
tof 950.0
nt 100000
ct 432
alock n
gain 20
FLAGS
il n
in n
dp y
DISPLAY
sp -61.0
wp 11126.3
vs 22
sc 0
wc 250
hzmm 44.51
is 500.00
rfl 4289.0
rfp 3870.6
th 3
ins 77.000
nm no ph
OFC. & VT
dfrq 199.911
dn H1
dpwr 35
dof 1100.0
dm yyy
dmm w
dmf 9900
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
189.728
155.023
144.550
136.043
134.836
134.017
131.452
128.903
129.539
129.456
128.993
128.750
128.241
127.923
126.860
77.637
77.000
76.363
49.421
45.589
21.623
200
180
160
140
120
100
80
60
40
20
ppm

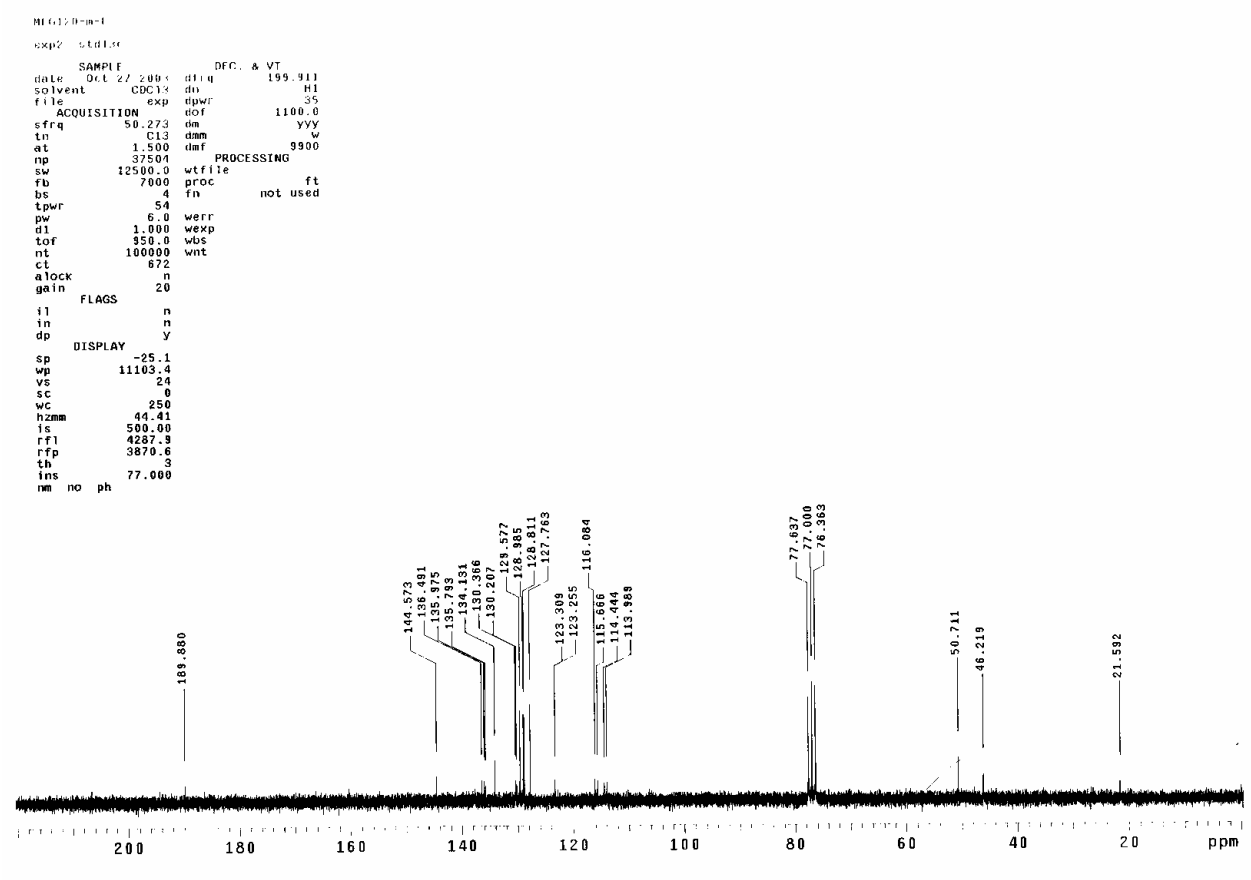
3f


Ts
N
O
F

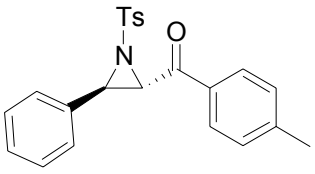

8.062
8.027
8.018
7.744
7.702
7.633
7.596
7.524
7.484
7.449
7.295
7.284
7.259
7.225
7.173
7.134
7.028
6.982
4.542
4.521
4.196
4.176
2.411
1.563
0.000
11
10
9
8
7
6
5
4
3
2
1
ppm

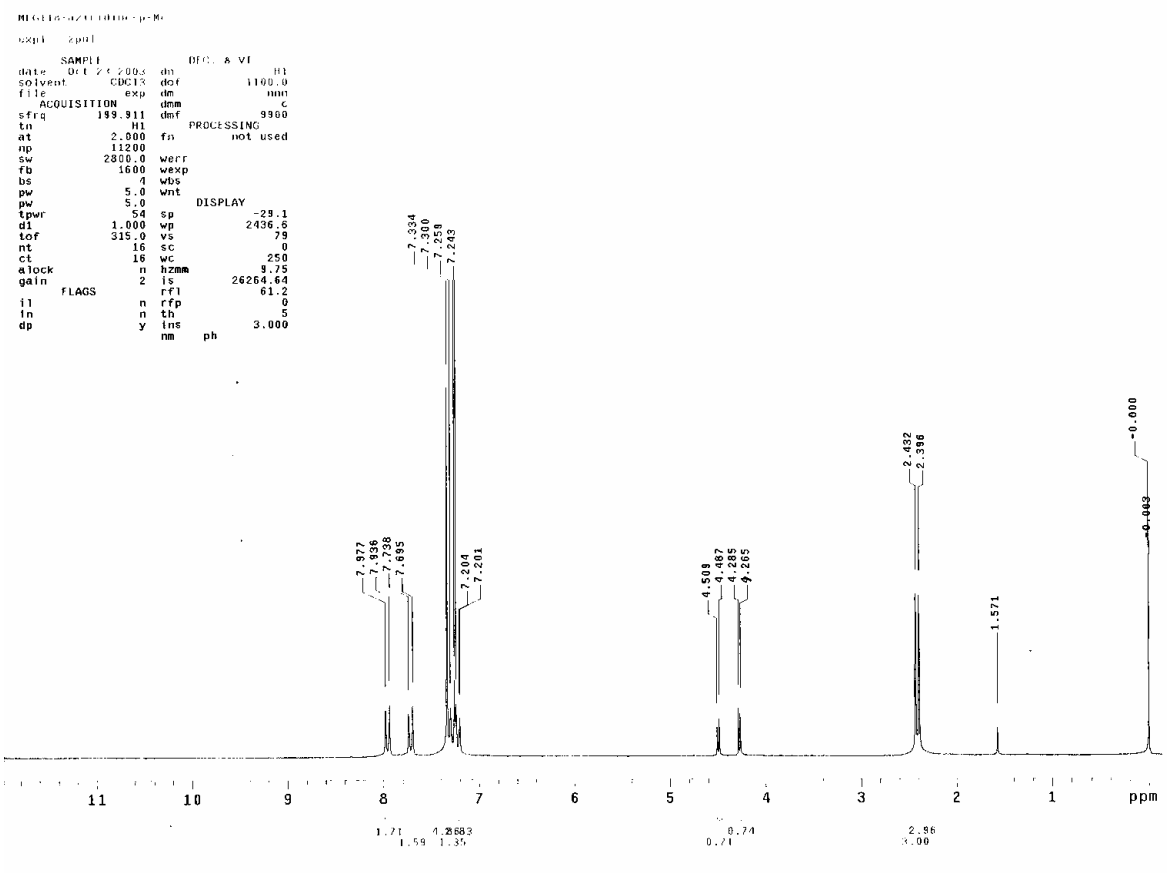
3f


Ts
N
O
F

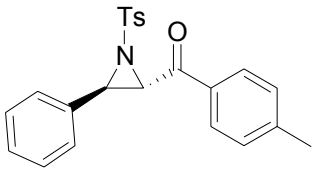

MEG170-m-1
exp2 std1c
SAMPLE
date Oct 27 2008
solvent CDCl3
file exp
ACQUISITION
sfrq 50.273
tn C13
at 1.500
np 37504
sw 12500.0
fb 7000
bs 4
tpwr 54
pw 6.0
d1 1.000
tof 950.0
nt 100000
ct 672
alock n
gain 20
FLAGS
il n
in n
dp y
DISPLAY
sp -25.1
wp 11103.4
vs 24
sc 0
wc 250
hzmm 44.41
is 500.00
rfl 4287.9
rfp 3870.6
th 3
ins 77.000
nm no ph
DEC. & VT
dfrq 199.911
dn H1
dpwr 35
dof 1100.0
dm yyy
dmm w
dmf 9900
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
189.880
144.573
136.491
135.975
135.793
134.131
130.366
130.207
129.577
128.985
128.811
127.763
123.309
123.255
116.084
115.666
114.444
113.989
77.637
77.000
76.363
50.711
46.219
21.592
200
180
160
140
120
100
80
60
40
20
ppm

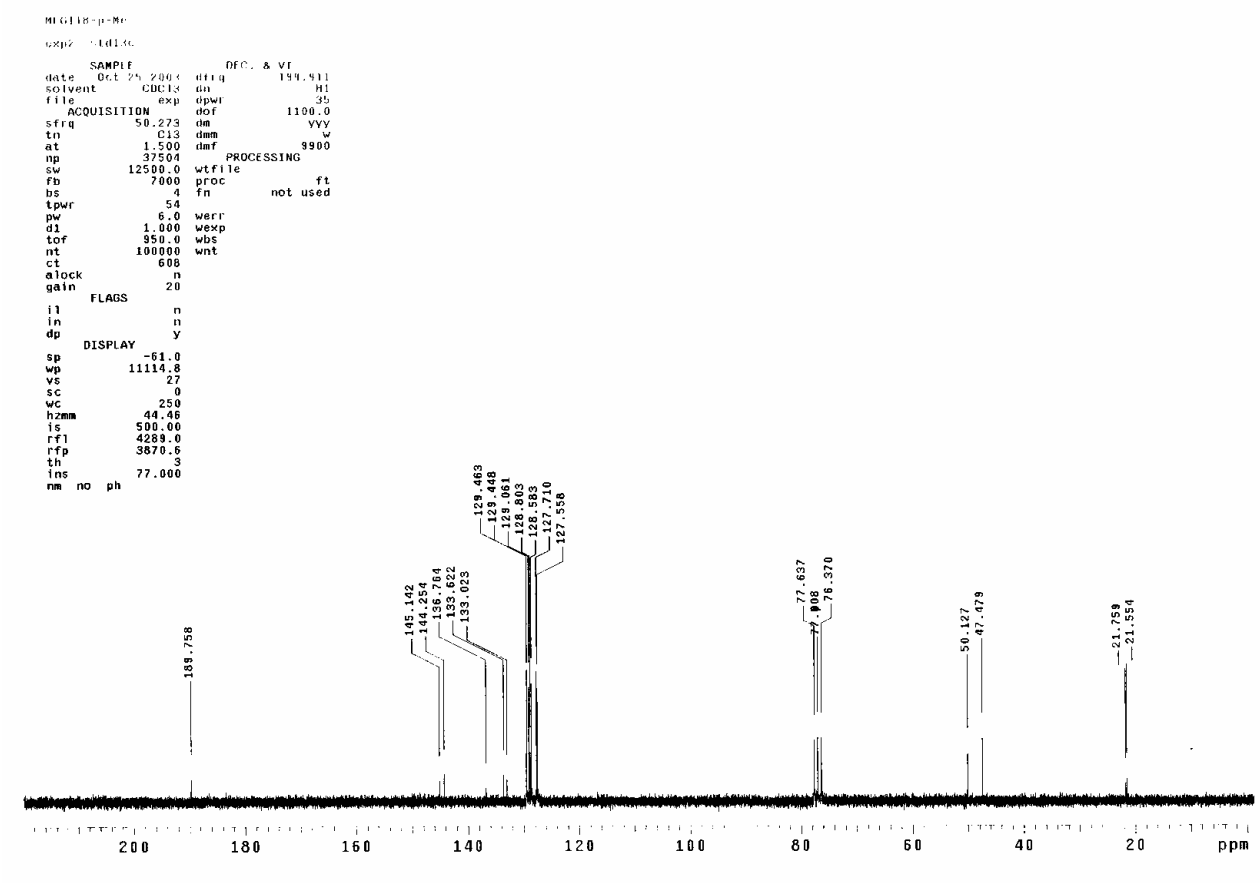
3g


Ts
N
O

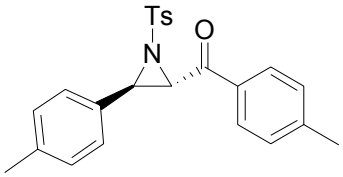

7.977
7.936
7.738
7.695
7.334
7.300
7.259
7.243
7.204
7.201
4.509
4.487
4.285
4.265
2.432
2.396
1.571
0.000
0.083
11
10
9
8
7
6
5
4
3
2
1
ppm

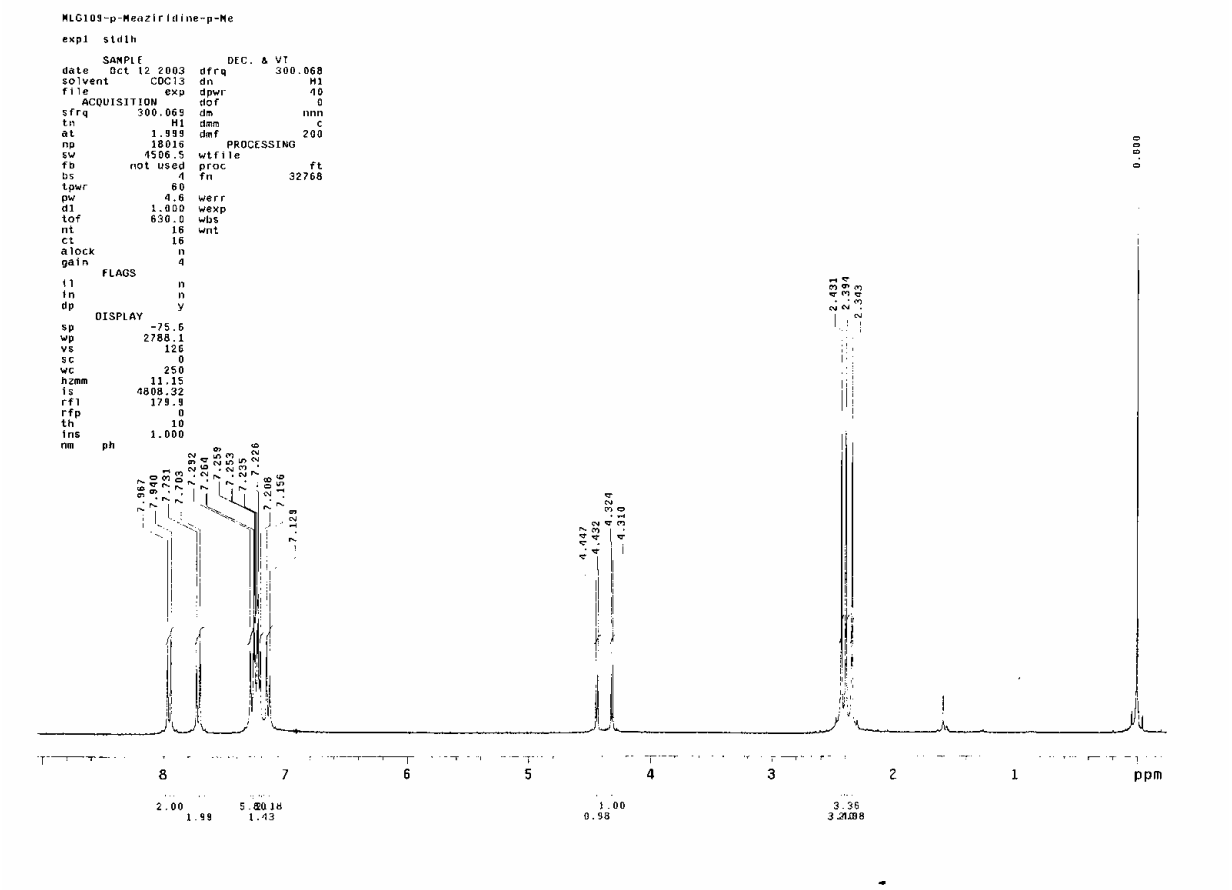
3g


Ts
N
O

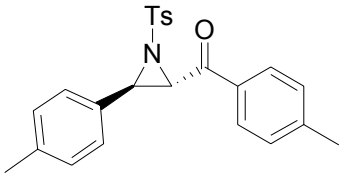

MIG118-p-Me
SAMPLE
date Oct 25 2004
solvent CDCl3
file exp
ACQUISITION
sfrq 50.273
tn C13
at 1.500
np 37504
sw 12500.0
fb 7000
bs 4
tpwr 54
pw 6.0
d1 1.000
tof 950.0
nt 100000
ct 608
alock n
gain 20
FLAGS
il n
in n
dp y
DISPLAY
sp -61.0
wp 11114.8
vs 27
sc 0
wc 250
hzmm 44.46
is 500.00
rfl 4289.0
rfp 3870.6
th 3
ins 77.000
nm no ph
DEC. & VT
dfrq 199.911
dn H1
dpwr 35
dof 1100.0
dm yyy
dmm w
dmf 9900
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
189.758
145.142
144.254
136.764
133.622
133.023
129.463
129.448
129.061
128.803
128.583
127.710
127.558
77.637
77.308
76.370
50.127
47.479
21.759
21.554
200
180
160
140
120
100
80
60
40
20
ppm

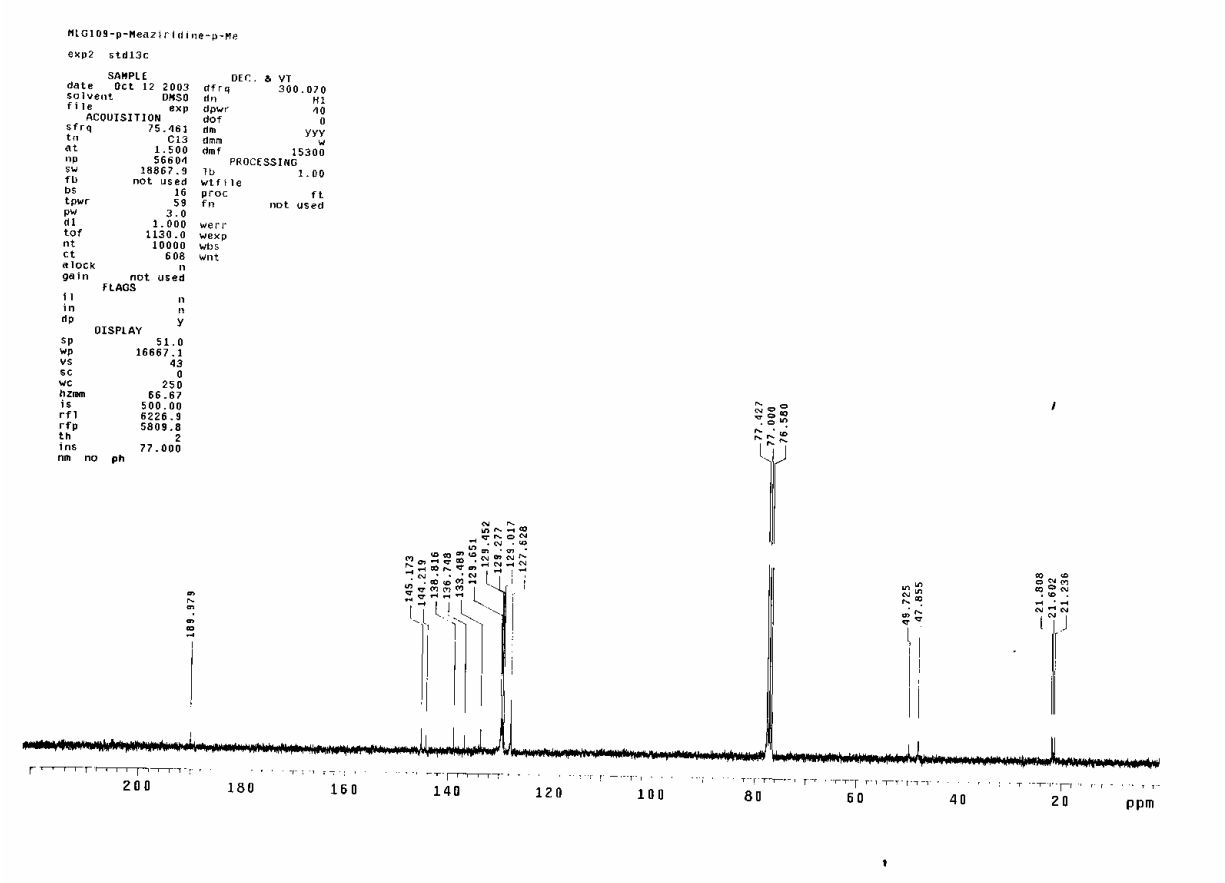
3h


Ts
N
O

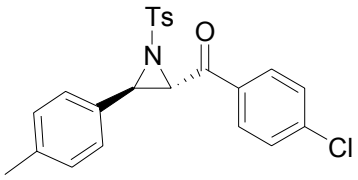

MLG109-p-Meaziridine-p-Me
expl stdlh
SAMPLE
date Oct 12 2003
solvent CDCl3
file exp
ACQUISITION
sfrq 300.069
tn H1
at 1.998
np 18016
sw 4506.5
fb not used
bs 4
tpwr 60
pw 4.6
d1 1.000
tof 630.0
nt 16
ct 16
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -75.6
wp 2788.1
vs 126
sc 0
wc 250
hzmm 11.15
is 4808.32
rfl 179.9
rfp 0
th 10
ins 1.000
nm ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 40
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn 32768
werr
wexp
wbs
wnt
7.967
7.940
7.731
7.703
7.282
7.264
7.259
7.253
7.235
7.226
7.208
7.156
7.129
4.447
4.432
4.324
4.310
2.431
2.394
2.343
0.800
8
7
6
5
4
3
2
1
ppm
2.00
1.99
5.20
1.43
1.00
0.98
3.35
3.00

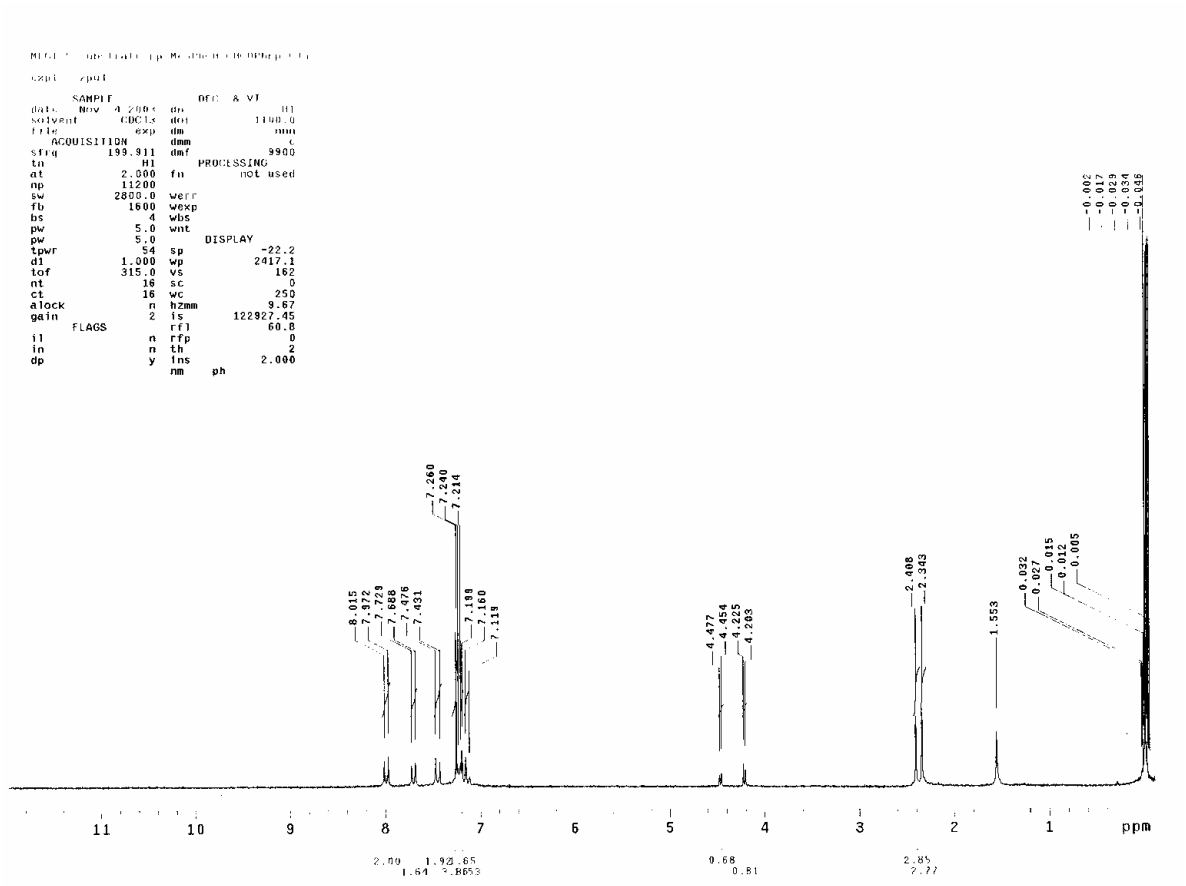
3h


Ts
N
O

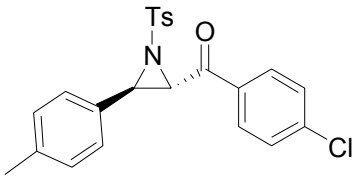

MLG109-p-Meaziridine-p-Me
exp2 std13c
SAMPLE
date Oct 12 2003
solvent DMSO
file exp
ACQUISITION
sfrq 75.461
tn C13
at 1.500
np 56604
sw 18867.9
fb not used
bs 16
tpwr 59
pw 3.0
d1 1.000
tof 1130.0
nt 10000
ct 608
alock n
gain not used
FLAGS
fl n
in n
dp y
DISPLAY
sp 51.0
wp 16667.1
vs 43
sc 0
wc 250
hzmm 66.67
is 500.00
rfl 6226.9
rfp 5809.8
th 2
ins 77.000
nm no ph
DEC. & VT
dfrq 300.070
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 15300
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
189.979
145.173
144.219
138.816
136.748
133.489
129.652
129.277
129.017
127.628
77.427
77.000
76.580
49.725
47.855
21.808
21.602
21.236
200
180
160
140
120
100
80
60
40
20
ppm

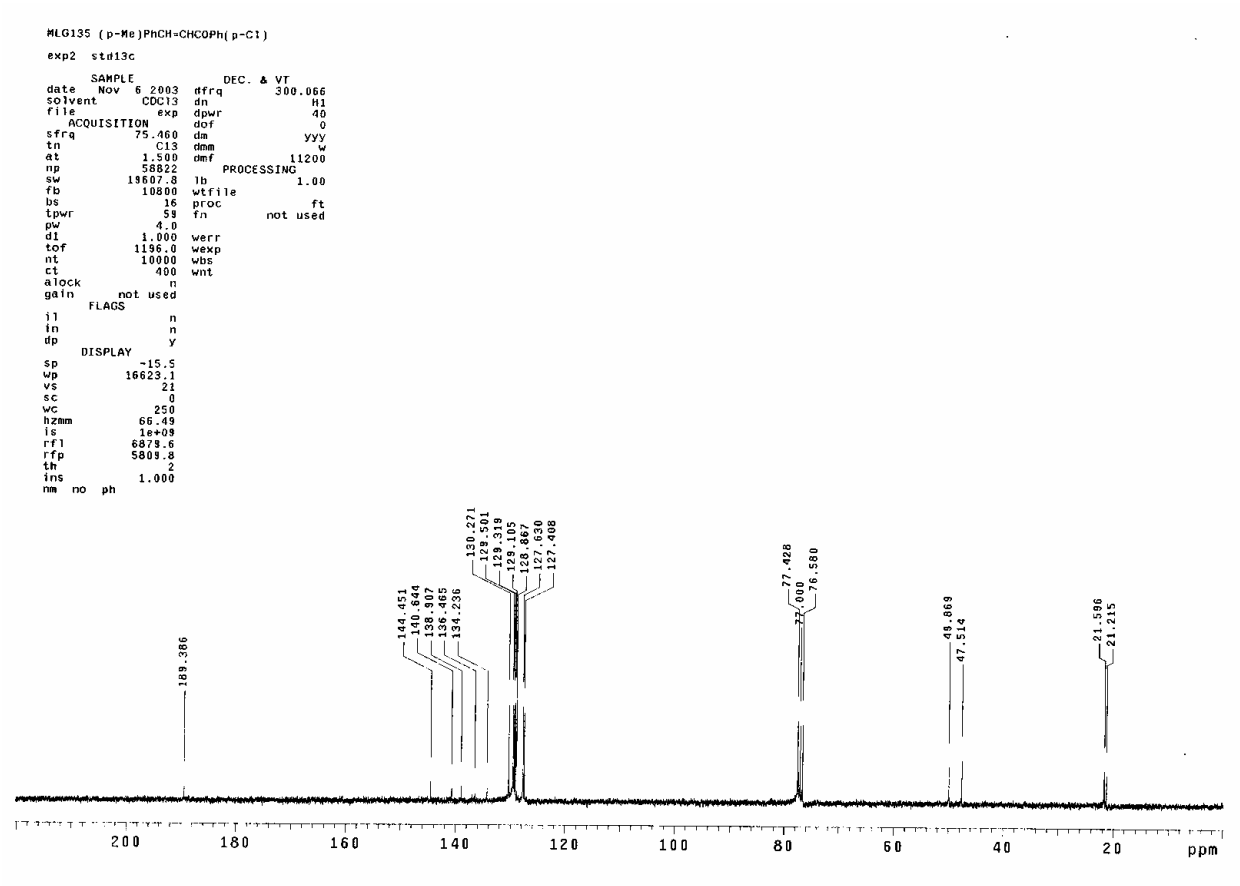
3i


Ts
N
O
Cl

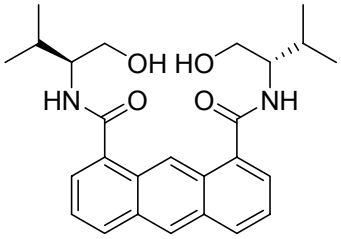

8.015
7.472
7.729
7.688
7.476
7.431
7.260
7.240
7.214
7.188
7.160
7.119
4.477
4.454
4.225
4.203
2.408
2.343
1.553
0.032
0.027
0.015
0.012
0.005
-0.002
-0.017
-0.023
-0.034
-0.038
11
10
9
8
7
6
5
4
3
2
1
ppm
2.00
1.92
1.64
3.8653
0.68
0.81
2.85
2.77

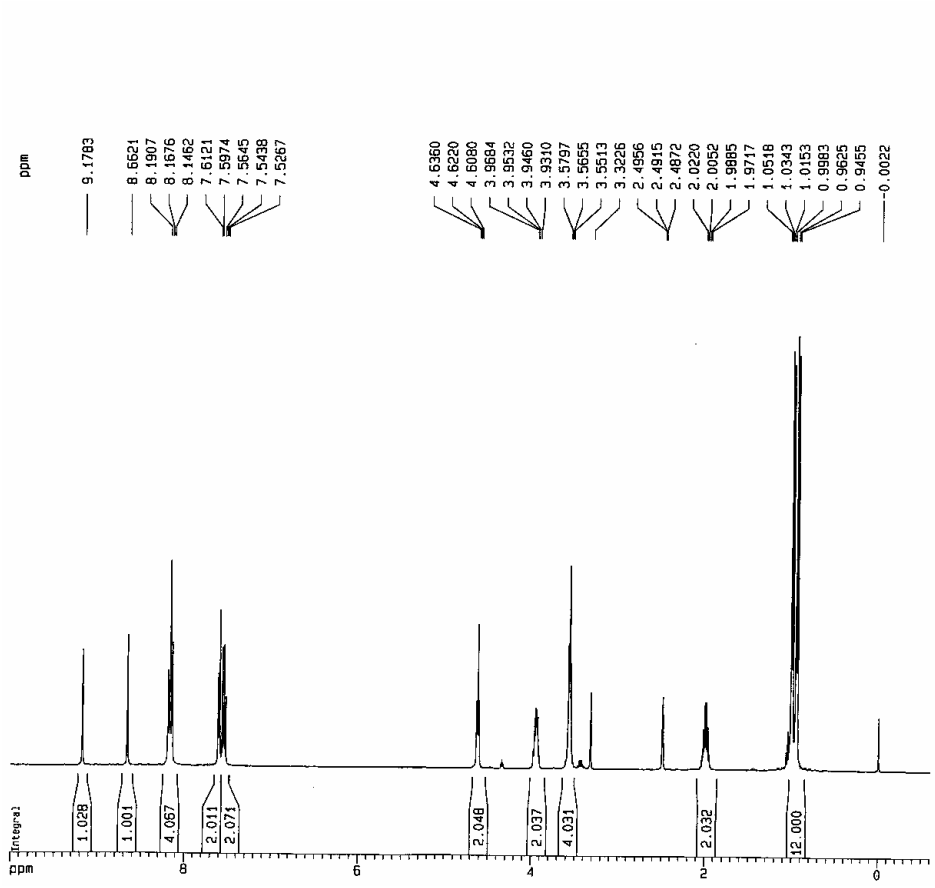
3i


Ts
N
O
Cl


MLG135 (p-Me)PhCH=CHCOPh(p-Cl)
exp2 std13c
SAMPLE
date Nov 6 2003
solvent CDCl3
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 16
tpwr 58
pw 4.0
d1 1.000
tof 1196.0
nt 10000
ct 400
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -15.5
wp 16623.1
vs 21
sc 0
wc 250
hzmm 66.49
is 1e+09
rf1 6879.6
rf2 5809.8
th 2
ins 1.000
nm no ph
DEC. & VT
dfrq 300.066
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
189.386
144.451
140.644
138.907
136.465
134.236
130.271
129.501
129.319
129.105
128.867
127.630
127.408
77.428
77.000
76.580
49.869
47.514
21.596
21.215
200
180
160
140
120
100
80
60
40
20
ppm

Diamide





Current Data Parameters

NAME	h22691
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040513
Time	7.59
INSTRUM	ARX400
PROBHD	5 mm Multinu
PULPROG	zg
TD	32768
SOLVENT	DMSO
NS	32
DS	0
SWH	6578.947 Hz
FIDRES	0.200774 Hz
AQ	2.4904180 sec
RG	715
DW	76.000 usec
DE	95.00 usec
TE	300.0 K
D1	2.00000000 sec
P1	3.00 usec
DE	95.00 usec
SFO1	400.1325569 MHz
NUCLEUS	1H

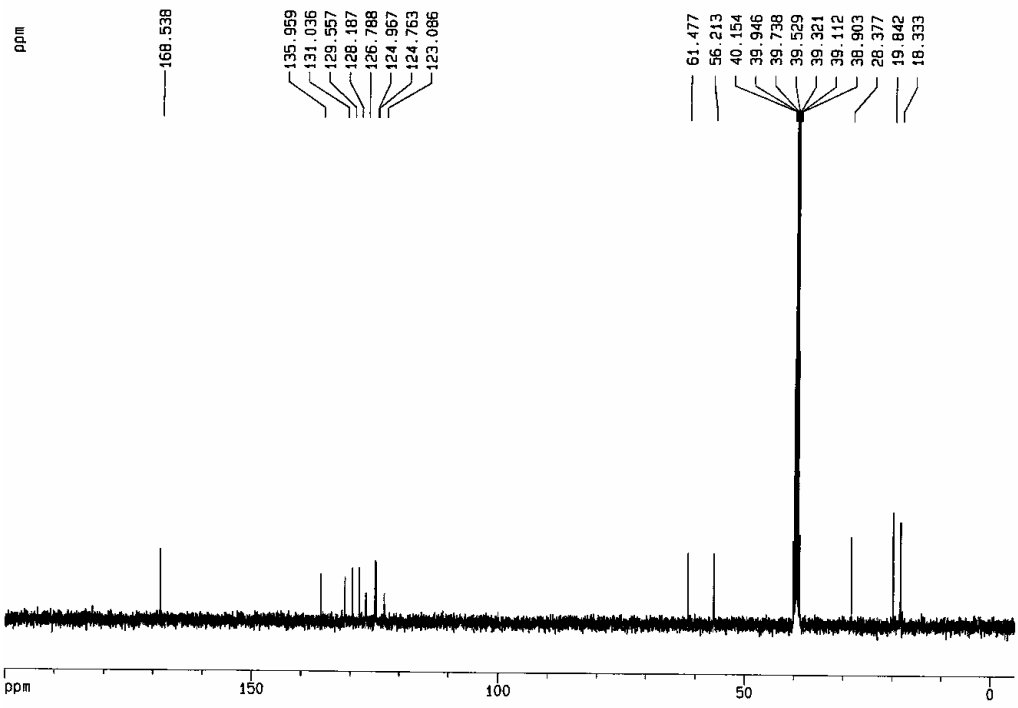
F2 - Processing parameters

SI	16384
SF	400.1300062 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	4.00

1D NMR plot parameters

CX	20.00 cm
F1P	10.000 ppm
F1	4001.30 Hz
F2P	-0.600 ppm
F2	-240.08 Hz
PPMCM	0.53000 ppm/cm
HZCM	212.06891 Hz/cm

Diamide



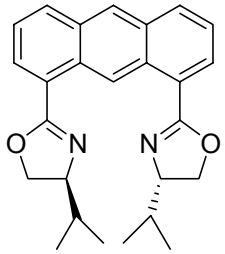

```
Current Data Parameters
NAME                 c22691
EXPNO                     1
PROCNO                    1

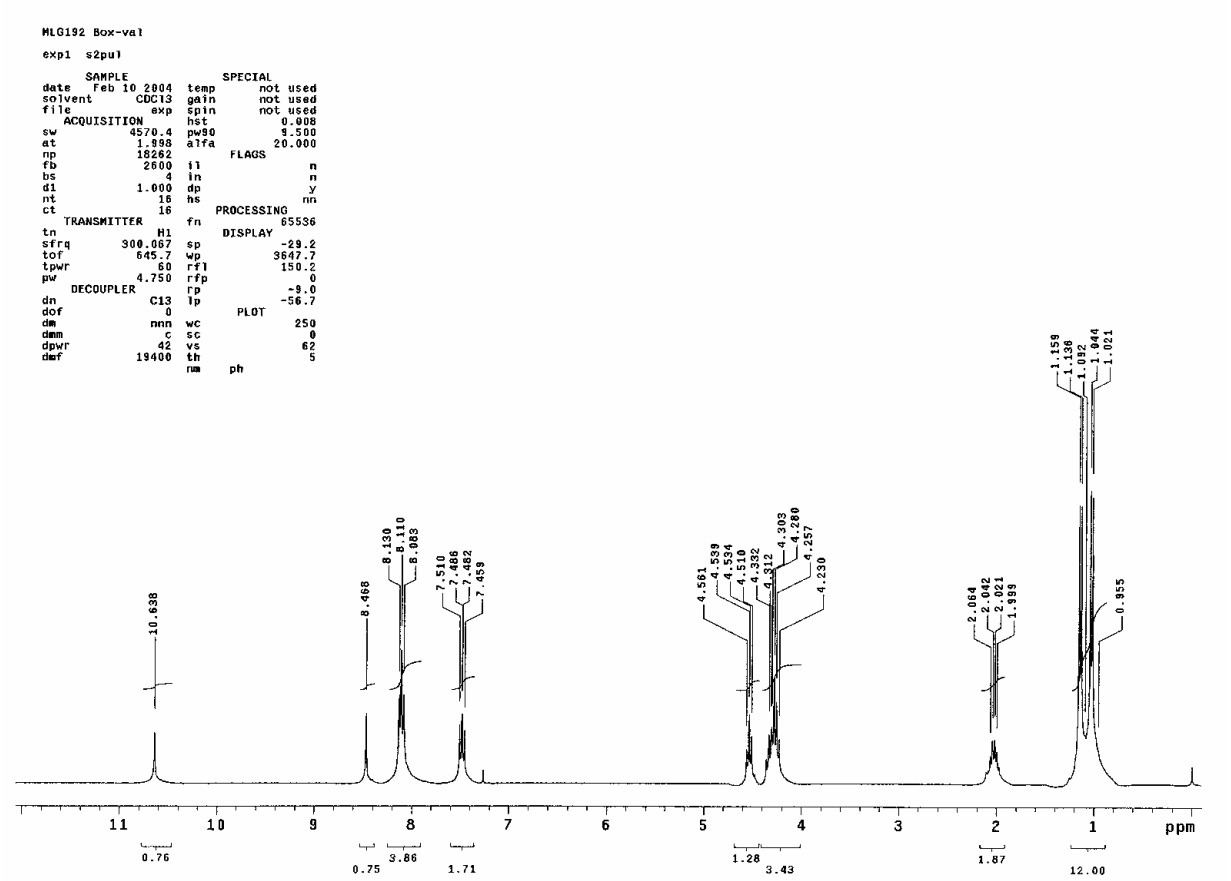
F2 - Acquisition Parameters
Date_              20040513
Time                   8.04
INSTRUM              ARX400
PROBHD     5 mm Multinu
PULPROG               zgdc
TD                   32768
SOLVENT               DMSO
NS                      241
DS                        2
SWH              29411.766 Hz
FIDRES           0.897576 Hz
AQ               0.5571060 sec
RG                   32768
DW                17.000 usec
DE                24.29 usec
TE                300.0 K
D12           0.00002000 sec
DL5                24.00 dB
CPDPRG              waltz16
P31               100.00 usec
D1              2.00000000 sec
P1                 4.00 usec
DE                24.29 usec
SFO1           100.6240000 MHz
NUCLEUS                 13C
D11           0.03000000 sec

F2 - Processing parameters
SI                   32768
SF             100.6128180 MHz
WDW                     EM
SSB                      0
LB                  1.00 Hz
GB                       0
PC                  0.50

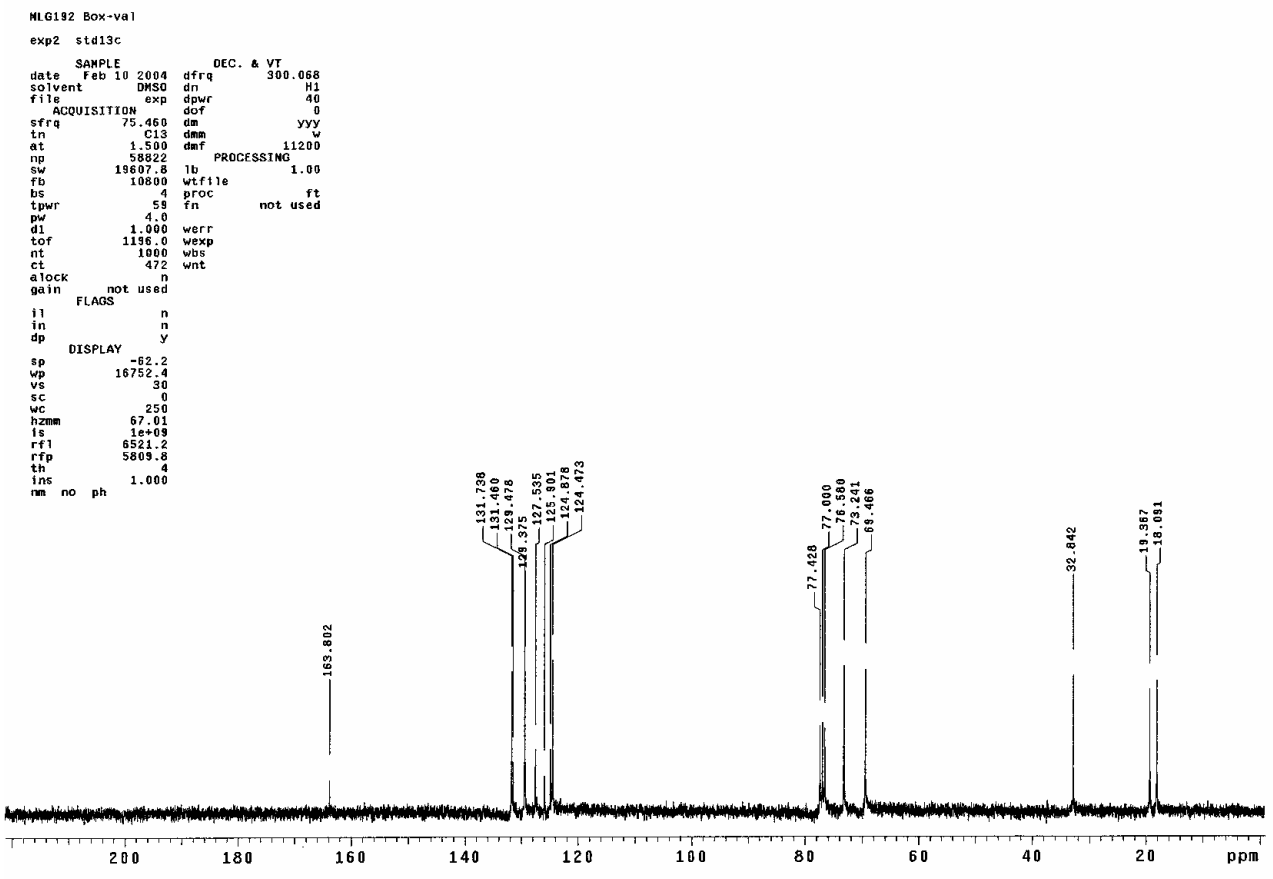
1D NMR plot parameters
CX                  20.00 cm
F1P               200.000 ppm
F1                20122.56 Hz
F2P                -5.000 ppm
F2                 -503.06 Hz
PPMCM            10.25000 ppm/cm
HZCM          1031.28149 Hz/cm
```

AnBOX


O
N
N
O


MLG192 Box-val
exp1 s2pul
SAMPLE
date Feb 10 2004
solvent CDCl3
file exp
ACQUISITION
sw 4570.4
at 1.998
np 18262
fb 2600
bs 4
d1 1.000
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 300.067
tof 645.7
tpwr 60
pw 4.750
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 42
dmf 19400
SPECIAL
temp not used
gain not used
spin not used
hst 0.008
pw90 9.500
alfa 20.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp -28.2
wp 3647.7
rfl 150.2
rfp 0
rp -9.0
lp -56.7
PLOT
wc 250
sc 0
vs 62
th 5
nm ph
10.638
8.468
8.130
8.110
8.083
7.510
7.486
7.482
7.459
4.561
4.539
4.534
4.510
4.332
4.312
4.303
4.280
4.257
4.230
2.064
2.042
2.021
1.999
1.159
1.139
1.102
1.044
1.021
0.955
11
10
9
8
7
6
5
4
3
2
1
ppm
0.76
0.75
3.86
1.71
1.28
3.43
1.87
12.00

AnBOX


MLG192 Box-val
exp2 std13c
SAMPLE
date Feb 10 2004
solvent DMSO
file exp
ACQUISITION
sfrq 75.460
tn C13
at 1.500
np 58822
sw 19607.8
fb 10800
bs 4
tpwr 59
pw 4.0
d1 1.000
tof 1196.0
nt 1000
ct 472
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -62.2
wp 16752.4
vs 30
sc 0
wc 250
hzmm 67.01
is 1e+09
rf1 6521.2
rfp 5809.8
th 4
ins 1.000
nm no ph
DEC. & VT
dfrq 300.068
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
163.802
131.738
131.460
129.478
126.375
127.535
125.801
124.878
124.473
77.428
77.000
76.580
73.241
69.466
32.842
19.367
18.091
200
180
160
140
120
100
80
60
40
20
ppm