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Supporting Information

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First Catalytic Enantio- and Diastereoselective Formation of β-Sultones: Ring-Strained Precursors for Enantioenriched β-Hydroxysulfonyl Derivatives

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Experimental

Except as otherwise indicated, all reactions were carried out in oven dried glassware under a positive pressure of nitrogen. Benzene (Fluka, >99.5%) was stored in crown-capped bottles under argon over 4Å molecular sieves. Dichloromethane was purified by distillation and dried by a passage over activated alumina under nitrogen atmosphere. Methanol (Fluka, HPLC grade), pentane (J.T. Baker, UV quality), *n*-hexane (Fluka, UV quality), cyclohexane (Thommen & Furler), ethyl acetate (Thommen & Furler) and triethylamine (Fluka, >99.5%) were used as purchased. Ethyl glyoxylate was purchased from Fluka as 50% solution in toluene and purified as detailed below. All other laboratory chemicals were purchased from *Fluka*, *Merck*, *J.T. Baker*, *ABCR* or *Aldrich* and were used without purification. For work-up procedures and flash-chromatography, distilled technical grade-solvents were used. Unless otherwise indicated, all liquids were added via syringe, solids were added neat against an argon flow. Solvents were removed at a heating-bath temperature of 40 °C and 600 - 30 mbar pressure by rotary evaporation. Non-volatile compounds were dried *in vacuo* at 0.01 mbar. Yields refer to purified compounds and are calculated in mol% of the used starting material. Except as otherwise indicated, reactions were magnetically stirred and monitored by thin layer-chromatography (TLC) using silica gel plates from *Merck (silica gel 60 F_{254})*. Visualization occurred by fluorescence quenching under UV light and by staining with $KMnO_4$ / NaOH. Purification by flash-chromatography was performed on silica gel 60 Å, 32-62, provided by *Fluka,* using a forced flow of eluent at 0.2-0.4 bar pressure. NMR-spectra were recorded on a *Varian Gemini 300* and a *Varian Mercury 300* spectrometer operating at 300 MHz (^{1}H), 75 MHz (^{13}C) or by the NMR service of the Laboratory of Organic Chemistry at ETHZ on a Bruker DRX400 spectrometer 400 MHz (^{1}H), 100 MHz (^{13}C). Chemical shifts δ are referred in terms of ppm and *J*-coupling constants are given in Hz. Abbreviations for multiplicity are as follows: *s* (singulet), *d* (doublet), *t* (triplet), *q* (quadruplet), *m* (multiplet), *b* (broad signal). IR-spectra were recorded on

a *Perkin Elmer Spectrum RX I FT-IR* and the signals are given by wave numbers (cm^{-1}). Optical rotation was measured on a *Jasco DIP-100 digital Polarimeter* operating at the sodium D line with a 100 mm path length cell. Melting points were measured using a Büchi 535 melting point apparatus in open glass capillaries and are uncorrected. Mass spectra were obtained from the ETH Zürich MS Service. High resolution EI mass spectra were performed on a Micromass AutoSpec Ultima and were calibrated with perfluorotributylamine (PFTBA) prior to data acquisition. High resolution ESI mass spectra were performed on an Ion Spec Ultima 2 FTICR. ESI mass spectra were performed on a Finnigan TSQ7000. Combustion analysis was performed by the Mikroelementaranalytisches Laboratorium at ETH Zürich. The single crystal X-ray analysis was performed by Paul Seiler at the ETH Zürich.

Purification of ethyl glyoxylate

To a flame-dried 25 mL round bottom flask equipped with a magnetic stirring bar and a short path distillation apparatus was added 10 mL of commercial ethyl glyoxylate solution (50% in toluene) and 3 g of phosphorus pentoxide. The distillation pot was warmed to 80–90 °C at 250 mbar to remove most of the toluene and to depolymerize the technical ethyl glyoxylate. The pressure was reduced to 200 mbar and the distillation pot slowly warmed to 120–130 °C to collect a glyoxylate/toluene mixture. The concentration of the distilled solution of monomeric glyoxylate was determined by ^{1}H-NMR to be typically between 55 and 75% (m/m).

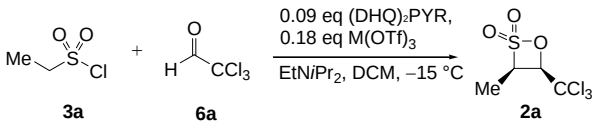
Activation of Lewis acid triflates

All Lewis acids employed were dried using a Büchi Kugelrohr oven. The metal triflate salts were placed in a flame dried round bottomed flask and heated to 140 °C at 0.01 mbar until weight constancy (usually overnight). The activated Lewis acids were stored and handled in a glove box.

General procedure for the formation of β-trichloromethyl β-sultones (GP1)

A suspension of $M(OTf)_3$ (0.137 mmol, 0.36 equiv.; M = Bi: 90 mg; M = In: 77 mg) in 1 mL DCM was sonicated at rt for 15 min and was subsequently cooled to –15 °C (EtOH/ice bath). Then a solution of chloral (**6a**, 99 μL, 1.018 mmol, 2.75 equiv.) in DCM (0.5 mL) was added. After 5 min a solution of $(DHQ)_2PYR$ (diydroquinine 2,5-diphenyl-4,6-pyrimidinediyl diether, 30 mg, 0.034 mmol, 0.091 equiv.) in DCM (0.5 mL) was added followed after additional 5 min by a solution of PMP (1,2,2,6,6-pentamethylpiperidine, 89 μL, 0.493 mmol, 1.32 equiv.) in DCM (0.5 mL). After another 15 min of stirring at –15 °C a solution of the corresponding sulfochloride **3** (0.371 mmol, 1 equiv.) in DCM (0.5 mL) was added dropwise by syringe pump over a period of 2.5 h. After complete addition the reaction mixture was stirred at –15 °C for additional 15 min. Prior to aqueous work up the suspension was diluted with MTBE. The precipitate was filtered off and washed 4 times with MTBE. The organic layer was subsequently sequentially washed with saturated aqueous Na_2EDTA (25 mL), 0.05 M H_2SO_4 (2 x 20 mL) and H_2O (2 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered and the solvent removed *in vacuo*.

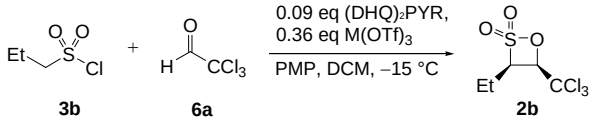
3-Methyl-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2a)



3-Methyl-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (**3a**) was prepared according to GP1, but using Et*N*i*P*r$_2$ instead of PMP and 0.18 equiv. of M(OTf)$_3$ instead of 0.36 equiv.. **2a** was furnished as colorless needles (M = Bi: 63 mg, 0.263 mmol, yield: 78%, *ee* = 67%, *dr* = 96:1; M = In: 53 mg, 0.210 mmol, yield: 61%, *ee* = 78%, *dr* > 100:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min) after ring opening with 2-phenethyl alcohol providing phenethyl sulfonate **11a** (*vide infra*).

$C_4H_5Cl_3O_3S$, **MW:** 239.51 g/mol. **Mp:** 118.1 - 119.7 °C. **$[\alpha]_D^{24.9°C}$** (c = 0.950, $CHCl_3$) = +17.75 ± 0.33 (sample with *ee* = 80%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 5.21 (*m*, 1 H, C*H*-S); 5.08 (*d*, *J* = 8.2, 1 H, C*H*-O); 1.92 (*d*, *J* = 7.4, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 94.3 (*C*Cl_3); 73.5 (C_{ring}); 72.6 (C_{ring}); 9.4 (*C*H_3). **IR (film):** ν = 2977, 2360, 1367, 1212, 1178, 945. **MS (EI) *m/z*:** 205 (0.8), 203 (1), 125 (35), 123 (57), 121 (73), 119 (12), 117 (14), 92 (12), 87 (20), 65 (12), 63 (16), 57 (40), 51 (14), 41 (26), 29 (100), 27 (30). **Anal. Calcd. for $C_4H_5Cl_3O_3S$**: C, 20.06; H, 2.10; O, 20.04; S, 13.39; Cl, 44.41. Found: C, 20.35; H, 2.10; O, 19.90; S, 13.21; Cl, 44.53.

3-Ethyl-4-trichlormethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2b)

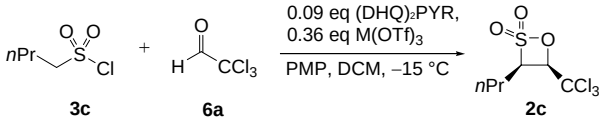


3b **6a** **2b**

3-Ethyl-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (**2b**) was prepared according to GP1 In case of M = In only 0.18 equiv. of $M(OTf)_3$ instead of 0.36 equiv. were used. **3b** was furnished as colorless needles (M = Bi: 62 mg, 0.245 mmol, yield: 65%, *ee* = 92%, *dr* > 100:1; M = In: 27 mg, 0.107 mmol, yield: 29%, *ee* = 97%, *dr* > 100:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min) after ring opening with 2-phenethyl alcohol providing phenethyl sulfonate **11b** (*vide infra*).

$C_5H_7Cl_3O_3S$, **MW:** 253.53 g/mol. **Mp:** 114.5-115.2 °C. **[α]** $_D^{24.9°C}$ (c = 1.125, $CHCl_3$) = +48.89 ± 0.39 (sample with *ee* = 92%). **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 5.06 (*d*, *J* = 8.3, 1 H, C*H*-O); 4.94-5.00 (*m*, 1 H, C*H*-S); 2.65-2.77 (*m*, 1 H, C*H*H); 2.16-2.26 (*m*, 1 H, CH*H*); 1.19 (*t*, *J* = 7.3, 3 H, CH_3). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 94.4 (*C*Cl_3); 80.2 (C_{ring}); 73.9 (C_{ring}); 18.1 (*C*H_2); 12.8 (*C*H_3). **IR ($CHCl_3$):** ν = 1461, 1382, 1206. **MS (ESI) *m/z*:** 309 $[M+MeOH+Na]^+$. **Anal. Calcd. for $C_5H_7Cl_3O_3S$**: C, 23.69; H, 2.78. Found: C, 23.79; H, 2.88.

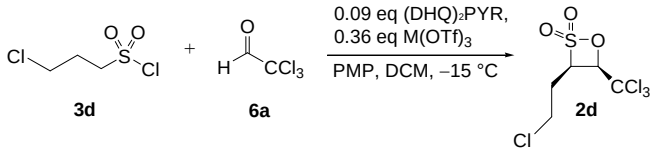
3-Propyl-4-trichlormethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2c)



3-Propyl-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (**2c**) was prepared according to GP1 as colorless solid (M = Bi: 47 mg, 0.176 mmol, yield: 47%, *ee* = 83%, *dr* > 50:1; M = In: 36 mg, 0.134 mmol, yield: 36%, *ee* = 89%, *dr* > 50:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min) after ring opening with 2-phenethyl alcohol providing phenethyl sulfonate **11c** (*vide infra*).

$C_6H_9Cl_3O_3S$, **MW:** 267.56 g/mol. **Mp:** 79.0-83.0 °C. **[α]** $_D^{24.9°C}$ (c = 1.55, $CHCl_3$) = +17.64 ± 0.16 (sample with *ee* = 83%). **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 4.99-5.06 (*m*, 2 H, C*H*-S, C*H*-O); 2.58-2.70 (*m*, 1 H, CH_2-CH_{ring}); 2.10-2.18 (*m*, 1 H, CH_2-CH_{ring}); 1.44-1.67 (*m*, 2 H, CH_2-CH_3); 2.64 (*t*, *J* = 7.4, 3 H, CH_3). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 94.5 (CCl_3); 78.4 (C_{ring}); 73.7 (C_{ring}); 26.1 (CH_2); 21.7 (CH_2); 13.4 (CH_3). **IR ($CHCl_3$):** ν = 2968, 2938, 2879, 1466, 1381. **MS (EI) *m/z*:** 187 $[MH\text{-}SO_3]^+$ (0.45), 149 $[MH\text{-}CCl_3]^+$ (54), 109 (37), 85 (44), 67 (55), 55 (97). **HRMS (EI) *m/z*:** Calc. for $[MH]^+$: 266.9411. Found: 266.9423. **Anal. Calcd. for $C_6H_9Cl_3O_3S$**: C, 26.93; H, 3.39. Found: C, 26.94; H, 3.45.

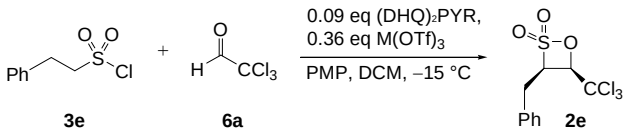
3-(2-Chloroethyl)-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2d)



3-(2-Chloroethyl)-4-trichlormethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (**2d**) was prepared according to GP1 as colorless needles (M = Bi: 93 mg, 0.323 mmol, yield: 87%, *ee* = 95%, *dr* > 50:1; M = In: 62 mg, 0.215 mmol, yield: 58%, *ee* = 81%, *dr* = 15:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min) after ring opening with phenylmagnesium bromide providing phenyl sulfone **14d** (*vide infra*).

$C_5H_6Cl_4O_3S$, **MW**: 287.98 g/mol. **Mp:** 71.5-73.0 °C. **$[\alpha]_D^{24.9°C}$** (c = 0.950, $CHCl_3$) = +65.35 ± 0.22. **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 5.36 (*ddd*, *J* = 11.5, 8.4, 2.9, 1 H, C*H*-S); 5.14 (*d*, *J* = 8.4, 1 H, C*H*-O); 3.78 (*ddd*, *J* = 11.7, 6.0, 4.8, 1 H, CH_2-Cl); 3.64 (*ddd*, *J* = 11.7, 9.1, 3.9, 1 H, CH_2-Cl); 3.17 (*dddd*, *J* = 15.5, 11.5, 6.0, 3.9, 1 H, CH_2-CH_{ring}); 2.69 (*dddd*, *J* = 15.5, 9.1, 4.8, 3.0, 1 H, CH_2-CH_{ring}). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 94.2 (*C*Cl_3); 74.9 (C_{ring}); 73.2 (C_{ring}); 41.6 (*C*H_2Cl); 27.4 (*C*H_2C_{ring}). **IR ($CHCl_3$):** ν = 3052, 2952, 2845, 1467, 1433, 1023. **MS (ESI) *m/z*:** 663 $[2xM+2xMeOH+Na]^+$, 343 $[M+MeOH+Na]^+$. **Anal. Calcd. for $C_5H_6Cl_4O_3S$**: C, 20.85; H, 2.10. Found: C, 21.08; H, 2.14.

3-Benzyl-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2e)

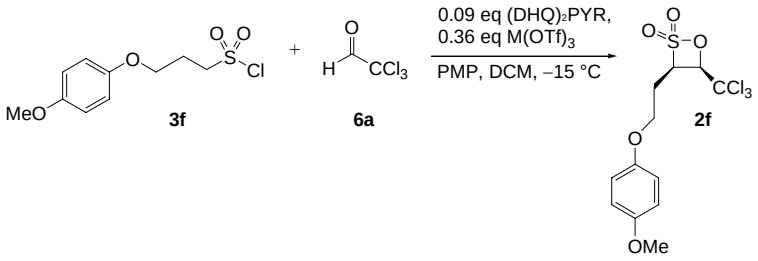


3e **6a** **2e**

3-Benzyl-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide **2e** was prepared according to GP1 as colorless solid (M = Bi: 70 mg, 0.223 mmol, yield: 60%, *ee* = 91%, *dr* = 22:1; M = In: 67 mg, 0.211 mmol, yield: 57%, *ee* = 84%, *dr* > 12:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 98:2 *n*-hexane/*i*PrOH, 1.0 mL/min).

$C_{10}H_9Cl_3O_3S$, **MW:** 315.60 g/mol. **Mp:** 142.1-142.7 °C. **$[\alpha]_D^{24.1°C}$** (c = 0.350, $CHCl_3$) = +53.98 ± 1.20 (sample with *ee* > 99.5% after recrystalization). **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 7.39-7.27 (*m*, 5 H, CH_{Ph}); 5.27 (*ddd*, *J* = 12.3, 8.3, 3.1, 1 H, C*H*-S); 5.13 (*d*, *J* = 8.3, 1 H, C*H*-O); 3.96 (*dd*, *J*=15.0, 12.3, 1 H, CH_2); 3.52 (*dd*, *J* = 15.0, 3.1, 1 H, CH_2). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 135.2 (C_{Ph}); 129.4 (C_{Ph}); 128.5 (C_{Ph}); 128.0 (C_{Ph}); 94.4 (CCl_3); 79.7 (C_{ring}); 73.9 (C_{ring}); 30.5 (CH_2). **IR ($CHCl_3$):** ν = 1603, 1498 , 1456, 1385. **MS (EI) *m/z*:** 314 $[M]^+$, 197 $[M\text{-}CCl_3]^+$(18), 133, 91 $[C_7H_7]^+$, 77 $[C_6H_5]^+$. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 313.9338. Found: 313.9334. **Anal. Calcd. for $C_{10}H_9Cl_3O_3S$**: C, 38.06; H, 2.87. Found: C, 38.15; H, 2.85.

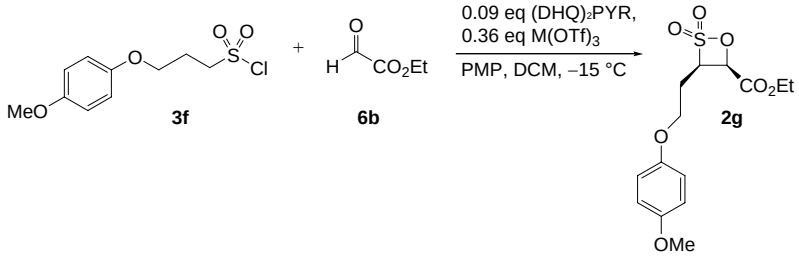
3-(2-(4-Methoxyphenoxy)ethyl)-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2f)



3-(2-(4-Methoxyphenoxy)ethyl)-4-trichloromethyl-1-oxa-2-thiacyclobutan-2,2-dioxide **2f** was prepared according to GP1 as colorless needles (M = Bi: 116 mg, 0.308 mmol, yield: 83%, *ee* = 99.4%, *dr* > 100:1; M = In: 96 mg, 0.256 mmol, yield: 69%, *ee* = 99.2%, *dr* > 100:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 96:4 *n*-hexane/EtOH, 1.0 mL/min).

$C_{12}H_{13}Cl_3O_5S$, **MW:** 375.66 g/mol. **Mp:** 150.3-151.5 °C. **$[\alpha]_D^{27.9°C}$** (c = 0.923, $CHCl_3$) = +42.44 ± 0.52. **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 6.89-6.81 (*m*, 4 H, C*H*$_{Ph}$); 5.35-5.30 (*m*, 1 H, C*H*-S); 5.11 (*d*, *J* = 8.4, 1 H, C*H*-O); 4.10-4.14 (*m*, 1 H, CH_2-OAr); 4.02 (*ddd*, *J* = 9.3, 7.9, 4.2, 1 H, CH_2-OAr); 4.02 (*s*, 3 H, OCH_3); 3.10-3.20 (*m*, 1 H, CH_2-C$_{ring}$); 2.63-2.70 (*m*, 1 H, CH_2-C$_{ring}$). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 154.6 (*C*$_{Ph}$); 152.1 (*C*$_{Ph}$); 115.6 (2x *C*H$_{Ph}$); 114.8 (2x *C*H$_{Ph}$); 94.6 (*C*Cl_3); 75.8 (*C*$_{ring}$); 73.4 (*C*$_{ring}$); 65.4 (*C*H_2O); 55.7 (O*C*H_3); 24.8 (*C*H_2C$_{ring}$). **IR ($CHCl_3$):** ν = 1602, 1509, 1386. **MS (EI) *m/z*:** 374 $[M]^+$, 294 $[M\text{-}SO_3]^+$, 137, 123. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 373.9549. Found: 373.9544. **Anal. Calcd. for $C_{12}H_{13}Cl_3O_5S$**: C, 38.87; H, 3.49. Found: C, 38.87; H, 3.15.

3-(2-(4-Methoxyphenyloxy)ethyl)-4-ethyloxycarbonyl-1-oxa-2-thiacyclobutan-2,2-dioxide (2g)



A suspension of $M(OTf)_3$ (0.144 mmol, 0.36 equiv., M = Bi: 90 mg; M = In: 77 mg) in DCM (1 mL) was sonicated at room temperature for 15 min and was subsequently cooled to –15 °C (EtOH/ice bath). Then a solution of $(DHQ)_2PYR$ (30 mg, 0.034 mmol, 0.091 equiv.) in DCM (0.5 mL) was added followed after additional 5 min by a solution of PMP (89 μL, 0.493 mmol, 1.32 equiv.) in DCM (0.5 mL). After another 15 min of stirring at –15 °C a solution of sulfochloride **3f** (101 mg, 0.383 mmol, 1 equiv.) in DCM (0.5 mL) and ethyl glyoxylate (**6b**, 1.029 mmol, 2.69 equiv.) as solution in a mixture of toluene (see purification of ethyl glyoxylate) and DCM (solvent amount in total 0.5 mL) were simultaneously added dropwise by syringe pump over a period of 2.5 h. Finally the reaction mixture was stirred at –15 °C for additional 15 min. Prior to aqueous work up the suspension was diluted with MTBE. The precipitate was filtered off and washed 4 times with MTBE. The organic layer was afterwards sequentially washed with saturated aqueous Na_2EDTA (25 mL), 0.05 M H_2SO_4 (2 x 20 mL) and H_2O (2 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered and the solvent removed *in vacuo*.

Flash column chromatography on silica (eluent DCM) furnished analytically pure sultone **2g** as a hygroscopic colorless solid (M = Bi: 65 mg, 0.193 mmol, yield: 52%, *ee* = 90%, *dr* = 2:1; M = In: 78 mg, 0.236 mmol, yield: 62%, *ee* = 90%, *dr* = 2.5:1). The *ee* values were determined by HPLC (Chiralcel OD-H, 96:4 *n*-hexane/EtOH, 1.0 mL/min). **2g** was found to be both acid and base labile. Even traces of acid in the isolated product initiate an autocatalytic decomposition /

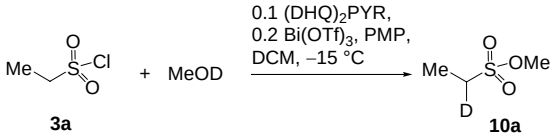
oligomerization whereas bases like triethylamine lead to elimination products. Therefore excessive drying was not possible and analytics were carried out immediately after purification.

$C_{14}H_{18}O_7S$, **MW:** 330.36 g/mol. **Mp:** 88.5-93.1 °C. **$[\alpha]_D^{27.9°C}$** (c = 0.150, CH_2Cl_2) = +38.57 ± 0.71 (sample with *ee* = 90%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 6.83 (*s*, 4 H, CH_{Ph}); 5.36 (*m*, 1 H, C*H*-S); 4.99 (*d*, *J* = 8.7, 1 H, C*H*-O); 4.38 (*qd*, *J* = 7.2, 1.9, 2 H, CH_2-O_{ester}); 4.11-3.97 (*m*, 2 H, CH_2-OAr); 3.77 (*s*, 3 H, OCH_3); 2.62-2.50 (*m*, 1 H, CH_2-C_{ring}); 2.36-2.26 (*m*, 1 H, CH_2-C_{ring}); 1.38 (*t*, *J* = 7.2, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 165.2 (*C*=O); 154.4 (C_{Ph}); 152.1 (C_{Ph}); 115.4 (2x CH_{Ph}); 114.7 (2x CH_{Ph}); 75.3 (C_{ring}); 64.5 (CH_2O); 63.2 (C_{ring}); 63.0 (CH_2O); 55.7 (OCH_3); 26.2 (CH_2C_{ring}); 14.2 (CH_3CH_2). **IR ($CHCl_3$):** ν = 2937, 1766, 1739, 1509, 1378, 1042. **MS (EI) *m/z*:** 330 $[M]^+$ (62), 207 $[M\text{-}OPhOMe]^+$ (43), 179 (46), 127 (100), 124 $[HOPhOMe]^+$ (63), 123 $[OPhOMe]^+$ (51), 99 (86). **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 330.0773. Found: 330.0771.

General procedure for the deuteration experiments (GP2)

A suspension of $Bi(OTf)_3$ (50 mg, 0.076 mmol, 0.18 equiv.) in DCM (0.5 mL) was sonicated at room temperature for 15 min and was subsequently cooled to –15 °C (EtOH/ice bath). Then a solution of d1-MeOD (31 µL, 0.756 mmol, 1.85 equiv.) in DCM (0.5 mL) was added. After 5 min a solution of $(DHQ)_2PYR$ (33 mg, 0.038 mmol, 0.09 equiv.) in DCM (0.5 mL) was added followed after additional 5 min by a solution of PMP (99 µL, 0.416 mmol, 1.33 equiv.) in DCM (0.5 mL). After another 15 min of stirring at –15 °C a solution of the corresponding sulfochloride **3** (0.416 mmol, 1 equiv.) in DCM (0.5 mL) was added dropwise by syringe pump over a period of 2.5 h. After complete addition the reaction mixture was stirred at –15 °C for additional 15 min. Prior to aqueous work up the suspension was diluted with MTBE. The precipitate was filtered off and washed 4 times with MTBE. The organic layer was subsequently sequentially washed with saturated aqueous Na_2EDTA (25 mL), 0.05 M H_2SO_4 (2 x 20 mL) and H_2O (2 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered and the solvent removed *in vacuo*.

α-monodeutero ethanesulfonic acid methyl ester (10a)

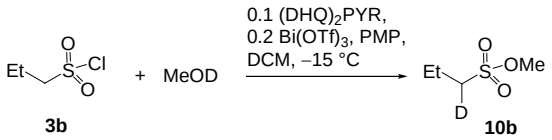


3a **10a**

α-Monodeutero ethanesulfonic acid methyl ester **10a** was obtained according to GP2 from ethanesulfonyl chloride (**3a**, 39 µL) as a pink volatile liquid (crude: 60 mg, 0.087 mmol of **10a** as determined by ^{1}H-NMR, yield: 21%, deuteration level 57% (determined by GC-MS)) containing 13% of unreacted sulfochloride **3a** and 69% of MTBE. Excessive drying was avoided

to prevent further loss of the volatile methyl ester **10a**. The analytical data were in accordance with literature precedent.[1]

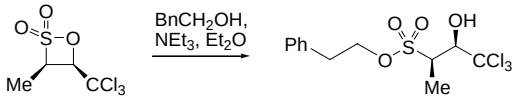
α-monodeutero propanesulfonic acid methyl ester (10b)



α-Monodeutero ethanesulfonic acid methyl ester **10b** was obtained according to GP2 from propanesulfonyl chloride (**3b**, 47 µL) as a pink volatile liquid (crude: 50 mg, 0.146 mmol, yield: 35% of **10b** as determined by 1H-NMR, deuteration level 88% (determined by GC-MS)) containing 46% of unreacted sulfochloride **3a** and 13% of MTBE. Excessive drying was avoided to prevent further loss of the volatile methyl ester **10b**. The analytical data were in accordance with literature precedent.[1]

[1] a) J. F. King, T. Durst, *J. Am. Chem. Soc.* **1965**, *87*, 5684; b) W. E. Truce, R. W. Campbell, *J. Am. Chem. Soc.* **1966**, *88*, 3599.

4,4,4-Trichloro-3-hydroxybutane-2-sulfonic acid phenethyl ester (11a)

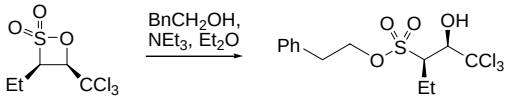


2a **11a**

To a stirred solution of β-sultone **2a** (23 mg, 0.086 mmol, 1 equiv.) in Et_2O (0.25 mL) a solution of 2-phenethyl alcohol (26 µL, 0.216 mmol, 2.5 equiv.) in Et_2O (0.15 mL) was added at room temperature followed by NEt_3 (12 µL, 0.086 mmol, 1 equiv.) in Et_2O (0.15 mL). After stirring the reaction mixture at room temperature for 14 h, the solvent was removed *in vacuo*. Flash column chromatography on silica (cyclohexane / EtOAc 4 : 1) furnished pure phenethyl ester **11a** as colorless oil (26 mg, 0.72 mmol, 84%, *ee* = 78%). The *ee* value was determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min).

$C_{12}H_{15}Cl_3O_4S$, **MW:** 361.67 g/mol. **[α]** $_D^{24.9°C}$ (c = 1.300, $CHCl_3$) = +4.33 ± 0.06 (sample with *ee* = 78%). **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** *δ* = 7.36-7.22 *(m*, 5 H, C*H*$_{Ph}$*);* 4.75-4.74 *(m,* 1 H, C*H*-OH*);* 4.50 *(dt, J* = 6.9, 0.7, 2 H, C*H*$_2$-O); 3.85 (*dq*, *J* = 7.0, 1.1, 1 H, C*H*-S); 3.23-3.21 (*m*, 1 H, -O*H*); 3.08 *(t, J* = 6.9, 2 H, C*H*$_2$-Ph); 1.57 (*d*, *J* = 7.0, 3 H, C*H*$_3$). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 136.1 (C_{Ph}); 129.0 (2x CH_{Ph}); 128.8 (2x CH_{Ph}); 127.2 (CH_{Ph}); 101.3 (CCl_3); 78.5 (C_{ring}); 70.6 (CH_2O); 57.8 (C_{ring}); 35.7 (CH_2Ph); 9.0 (CH_3). **IR ($CHCl_3$):** ν = 3588, 3370, 1456, 1355, 1336, 1172. **HRMS (ESI) *m/z*:** Calc. for $[M+Na]^+$: 382.9649. Found: 382.9640. **Anal. Calcd. for $C_{12}H_{15}Cl_3O_4S$**: C, 39.85; H, 4.18. Found: C, 39.73; H, 4.19.

1,1,1-Trichloro-2-hydroxypentane-3-sulfonic acid phenethyl ester (11b)

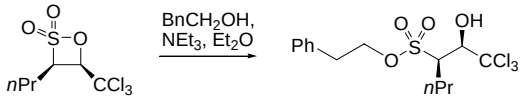


2b **11b**

To a stirred solution of β-sultone **2b** (25 mg, 0.094 mmol, 1 equiv.) in Et_2O (2 mL) a solution of 2-phenethyl alcohol (28 µL, 0.234 mmol, 2.5 equiv.) in Et_2O (0.25 mL) was added at room temperature followed by NEt_3 (13 µL, 0.094 mmol, 1 equiv.) in Et_2O (0.25 mL). The flask was subsequently closed. After stirring the reaction mixture at 50 °C for 3 h, the solvent was removed *in vacuo*. Excess of phenethyl alcohol was removed by flash column chromatography on silica (cyclohexane / EtOAc 4 : 1). Unconverted sultone was removed afterwards by flash column chromatography on silica (DCM), furnishing the pure phenethylester **11b** as colorless oil (22 mg, 0.059 mmol, 62%, *ee* = 90%). The *ee* value was determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min).

$C_{13}H_{17}Cl_3O_4S$, **MW:** 375.70 g/mol. **$[\alpha]_D^{24.9°C}$** (c = 0.750, $CHCl_3$) = +9.29 ± 0.21 (sample with *ee* = 90%). **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 7.36-7.23 (*m*, 4 H, C*H*$_{Ph}$); 4.72-4.71 (*m*, 1 H, C*H*-OH); 4.51 (*t*, 2 H, C*H*$_2$-O); 3.57 (*ddd*, *J* = 8.5, 2.3, 2.2, 1 H, C*H*-S); 3.11-3.07 (*m*, 3 H, C*H*$_2$-Ph, -O*H*); 2.39-2.29 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 1.98-1.86 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 1.19 (*t*, *J* = 7.5, 3 H, C*H*$_3$). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 136.1 (*C*$_{Ph}$); 129.0 (2x *C*H$_{Ph}$); 128.8 (2x *C*H$_{Ph}$); 127.2 (*C*H$_{Ph}$); 101.3 (*C*Cl$_3$); 79.2 (*C*$_{ring}$); 70.1 (*C*H$_2$O); 64.2 (*C*$_{ring}$); 35.7 (*C*H$_2$Ph); 17.7 (*C*H$_2$-C$_{ring}$); 13.3 (*C*H$_3$). **IR ($CHCl_3$):** ν = 3587, 1604, 1498 , 1456, 1334, 1164 cm^{-1}. **MS (ESI) *m/z*:** 773 [2xM+Na]$^+$, 399 [M+Na]$^+$. **Anal. Calcd. for $C_{13}H_{17}Cl_3O_4S$**: C, 41.56; H, 4.56. Found: C, 41.60; H, 4.60.

1,1,1-Trichloro-2-hydroxyhexane-3-sulfonic acid phenethyl ester (11c)

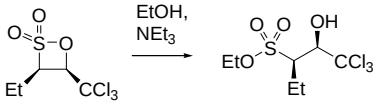


2c **11c**

To a stirred solution of β-sultone **2c** (28 mg, 0.094 mmol, 1 equiv.) in Et_2O (1 mL) a solution of 2-phenethyl alcohol (56 µL, 0.471 mmol, 5 equiv.) in Et_2O (0.25 mL) was added at room temperature followed by NEt_3 (13 µL, 0.094 mmol, 1 equiv.) in Et_2O (0.25 mL). The flask was subsequently closed. After stirring the reaction mixture at 50 °C for 18 h, the solvent was removed *in vacuo*. Excess of phenethyl alcohol was removed by flash column chromatography on silica (cyclohexane / EtOAc 4 : 1). Unconverted sultone was removed afterwards by flash column chromatography on silica (DCM), furnishing the pure phenethylester as colorless oil (28 mg, 0.72 mmol, 76%, *ee* = 89%). The *ee* value was determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min).

$C_{14}H_{19}Cl_3O_4S$, **MW:** 389.73 g/mol. **$[\alpha]_D^{24.9°C}$** (c = 0.550, $CHCl_3$) = +7.38 ± 0.13 (sample with *ee* = 89%). **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 7.30-7.16 (*m*, 5 H, C*H*$_{Ph}$); 4.66 (*dd*, *J* = 5.3, 1.0, 1 H, C*H*-OH); 4.44 (*t*, *J* = 6.9, 2 H, C*H*$_2$-O); 3.61 (*ddd*, *J* = 8.6, 2.2, 1.0, 1 H, C*H*-S); 3.07 (*d*, *J* = 5.3, 1 H, -O*H*); 3.03 (*t*, *J* = 6.9, 2 H, C*H*$_2$-Ph); 2.21-2.12 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 1.85-1.75 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 1.72-1.60 (*m*, 1 H, C*H*$_2$CH$_3$); 1.47-1.34 (*m*, 1 H, C*H*$_2$CH$_3$); 0.86 (*t*, *J* = 7.3, 3 H, C*H*$_3$). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 136.1 (*C*$_{Ph}$); 129.0 (2x *C*H$_{Ph}$); 128.8 (2x *C*H$_{Ph}$); 127.2 (*C*H$_{Ph}$); 101.3 (*C*Cl$_3$); 79.2 (*C*$_{ring}$); 70.1 (*C*H$_2$O); 62.4 (*C*$_{ring}$); 35.7 (*C*H$_2$Ph); 26.0 (*C*H$_2$-C$_{ring}$); 21.8 (*C*H$_2$CH$_3$); 14.0 (*C*H$_3$). **IR ($CHCl_3$):** ν = 3586, 2966, 2877, 1456, 1337, 1170. **MS (ESI) *m/z*:** 413 $[M+Na]^+$. **Anal. Calcd. for $C_{14}H_{19}Cl_3O_4S$**: C, 43.15; H, 4.91. Found: C, 43.22; H, 5.01.

1,1,1-Trichloro-2-hydroxypentane-3-sulfonic acid ethyl ester (12)

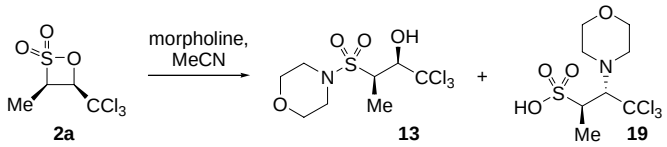


2b **12**

To a stirred solution of β-sultone **2b** (27 mg, recrystallized from hexane, *ee* = 95% (determined as phenethyl sulfonate **11b**), 0.106 mmol, 1 equiv.) in Et_2O (0.50 mL) EtOH (0.5 mL) was added at room temperature followed by NEt_3 (148 µL, 1.065 mmol, 10 equiv.). After stirring the reaction mixture at room temperature for 17 h, the solvent was removed *in vacuo*. Flash column chromatography on silica (DCM) furnished pure ethyl ester **12** as colorless oil (17 mg, 0.057 mmol, 54%, *ee* = 96%). The *ee* value was determined by HPLC (Chiralcel OD-H, 98:2 *n*-hexane/EtOH, 1.0 mL/min).

$C_7H_{13}Cl_3O_4S$, **MW**: 299.60 g/mol. **[α]** $_D^{24.9°C}$ (c = 0.80, $CHCl_3$) = +16.26 ± 0.90 (sample with *ee* = 96%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.85 *(m, 1 H,* C*H*-OH*);* 4.41 *(dq,* J = 7.2, 2.8, 1 H, CH*H*-O); 4.36 *(dq, J* = 6.85, 2.8, 1 H, C*H*H-O); 3.61 *(ddd, J* = 8.4, 2.2, 1.0, 1 H, C*H*-S); 3.42 (*m*, 1 H, -O*H*); 2.48-2.35 (*m*, 1 H, CH_2-C_{ring}); 2.06-1.91 (*m*, 1 H, CH_2-C_{ring}); 1.44 *(dd, J* = 7.2, 6.9, 3 H, CH_3); 1.22 (*t*, *J* = 7.5, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 101.4 (*C*Cl_3); 79.1 (C_{ring}); 67.1 (O*C*$H_2$$CH_3$); 63.9 ($C_{ring}$); 17.7 (*C*$H_2$$C_{ring}$); 15.2 (*C*$H_3$); 13.3 (*C*$H_3$). **IR ($CHCl_3$):** ν = 3590, 3500, 2987, 1466, 1355. **HRMS (ESI) *m/z*:** Calc. for $[M+Na]^+$: 320.9492. Found: 320.9493. **Anal. Calcd. for $C_7H_{13}Cl_3O_4S$**: C, 28.06; H, 4.37. Found: C, 28.42; H, 4.48.

1,1,1-Trichloro-3-(morpholine-4-sulfonyl)-butan-2-ol (13) and 4,4,4-Trichloro-3-morpholin-4-yl-butane-2-sulfonic acid (19)



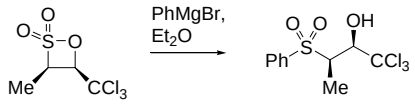
To a stirred solution of β-sultone **2a** (17 mg, batch with *ee* = 80% (determined as phenethyl sulfonate **11a**), 0.071 mmol, 1 equiv.) in DCM (0.75 mL) a solution of morpholine (62 µL, 0.714 mmol, 10 equiv.) in DCM (0.25 mL) was added room temperature. The reaction mixture was stirred for 3 h at room temperature before removing the solvent *in vacuo*. Then the crude product mixture (regioselectivity 2:1) was dissolved in MeOH (50 µL) and Et_2O was added until precipitation of the minor regioisomer, the hygroscopic taurine derivative **19**, occurred. After standing overnight at 4 °C the supernatant liquid was removed, diluted with 15 mL of Et_2O and extracted twice with 1M HCl. The combined organic layers were washed with H_2O until neutral, dried over Na_2SO_4 and filtered. After removal of the solvent *in vacuo*, sulfonamide **13** was obtained as colorless solid (15 mg, 0.045 mmol, 65%). Taurine derivative **19** was furnished without further purification as slightly brown solid (8 mg, 0.025 mmol, 35%).

Sulfonamide 13: $C_8H_{14}Cl_3NO_4S$, MW: 326.63 g/mol. **$[\alpha]_D^{24.9°C}$** (c =0.75, $CHCl_3$) = +13.28 ± 0.55 (sample with *ee* = 80%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 4.83-4.81 (*m*, 1 H, C*H*-OH); 3.84 (*dq*, *J* = 7.2, 0.9, 1 H, C*H*-S); 3.78-3.72 (*m*, 4 H, 2x CH_2-O); 3.44-3.39 (*m*, 4 H, 2x CH_2-N); 3.37 (*b*, 1H, -O*H*); 1.6 (*d*, *J* = 7.2, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 101.7 (*C*Cl_3); 79.0 (C_{ring}); 66.9 (*C*H_2O); 58.7 (C_{ring}); 46.5 (*C*H_2N); 9.1 (*C*H_3). **IR ($CHCl_3$):** ν = 3581, 2925, 2865, 1454, 1340, 1225, 1158. **HRMS (ESI) *m/z*:** Calc. for $[M+Na]^+$: 347.9601. Found: 347.9603. **Anal. Calcd. for $C_8H_{14}Cl_3NO_4S$**: C, 29.42; H, 4.32. Found: C, 29.05; H, 4.23.

Taurine derivative 19: $C_8H_{14}Cl_3NO_4S$, MW: 326.63 g/mol. **^{1}H NMR (400 MHz, CD_3OD, 21 °C):** δ = 4.84 (*d*, *J* = 1.2, 1 H, C*H*-N); 3.90-3.86 (*m*, 4 H, 2x CH_2-O); 3.50 (*dq*, *J* = 7.20, 1.2, 1

H, C*H*-S); 3.25-3.21 (*m*, 4 H, 2x C*H*$_2$-N); 1.51 (*d*, *J* = 7.0, 3 H, C*H*$_3$). **^{13}C NMR (100 MHz, CD_3OD, 21 °C):** δ = 105.2 (*C*Cl_3); 81.1 (C_{ring}); 64.9 (*C*H_2O); 57.7 (C_{ring}); 44.7 (*C*H_2N); 10.7 (*C*H_3). **IR ($CHCl_3$):** ν = 1807, 1666, 1603. **Anal. Calcd. for $C_{12}H_{15}Cl_3O_4S$**: a satisfactory microanalysis could not be obtained due to hygroscopicity.

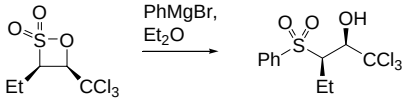
3-Benzenesulfonyl-1,1,1-trichlorobutan-2-ol (14a)



A stirred solution of β-sultone **2a** (20 mg, batch with *ee* = 84% (determined as phenethyl sulfonate **11a**), 0.089 mmol, 1 equiv.) in Et_2O (1.5 mL) was cooled to 4 °C. Then a solution of phenyl magnesium bromide (44 μL, 0.133 mmol, 3M in Et_2O, 1.5 equiv.) in Et_2O (0.5 mL) was added. The solution was allowed to warm up to room temperature and the flask was subsequently closed. After stirring the reaction mixture at 50 °C for 15 h, it was allowed to cool down to room temperature. Then ice and HCl (1M, 3 mL) were successively added. The aqueous layer was extracted three times with Et_2O, the combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed *in vacuo*. Flash column chromatography on silica (DCM) furnished pure phenyl sulfone **14a** as colorless oil (26 mg, 0.72 mmol, 84%, *ee* = 84%). The *ee* value was determined by HPLC (Chiralcel OD-H, 99:1 → 98.5:1.5 *n*-hexane/*i*PrOH, 1.5 mL/min).

$C_{10}H_{11}Cl_3O_3S$, **MW**: 317.62 g/mol. **$[\alpha]_D^{24.9°C}$** (c =0.3, $CHCl_3$) = +21.15 ± 0.50 (sample with *ee* = 84%) **^{1}H NMR (400 MHz, $CDCl_3$, 21 °C):** δ = 7.95-7.91 (*m*, 2 H, C*H*$_{Ph}$); 7.73-7.68 (*m*, 1 H, C*H*$_{Ph}$); 7.63-7.57 (*m*, 2 H, C*H*$_{Ph}$); 4.97 (*b*, 1 H, C*H*-OH); 3.84 (*dq*, *J* = 7.0, 1.1, 1 H, C*H*-S); 3.43 (*b*, 1 H, -O*H*); 1.51 (*d*, *J* = 7.0, 1 H, C*H*$_3$). **^{13}C NMR (100 MHz, $CDCl_3$, 21 °C):** δ = 136.7 (*C*$_{Ph}$); 134.3 (*C*H$_{Ph}$); 129.4 (2x*C*H$_{Ph}$); 129.2 (2x*C*H$_{Ph}$); 101.7 (*C*Cl_3); 77.7 (*C*$_{ring}$); 61.2 (*C*$_{ring}$); 8.6 (*C*H_3). **IR ($CHCl_3$):** ν = 3586, 3467, 1448, 1308,1151. **HRMS (ESI) *m/z*:** Calc. for $[M+Na]^+$: 338.9387. Found: 338.9394. **Anal. Calcd. for $C_{10}H_{11}Cl_3O_3S$**: C, 37.82; H, 3.49. Found: C, 38.29; H, 3.57.

3-Benzenesulfonyl-1,1,1-trichloropentan-2-ol (14b)

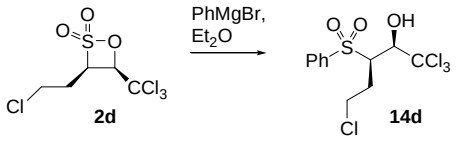


2b **14b**

A stirred solution of β-sultone **2b** (19 mg recrystallized from hexane, *ee* = 99.4% (determined as phenethyl sulfonate **11b**), 0.075 mmol, 1 equiv.) in Et_2O (2 mL) was cooled to 4 °C. Then a solution of phenyl magnesium bromide (37 μL, 0.112 mmol, 3M in Et_2O, 1.5 equiv.) in Et_2O (0.25 mL) was added. The solution was allowed to warm up to room temperature and the flask was subsequently closed. After stirring the reaction mixture at 50 °C for 2 h, it was allowed to cool down to room temperature. Then ice and HCl (1M, 3 mL) were successively added. The aqueous layer was extracted three times with Et_2O, the combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed *in vacuo*. Flash column chromatography on silica (DCM) furnished pure phenyl sulfone **14b** as colorless oil (23 mg, 0.69 mmol, 92%, *ee* = 99.5%). The *ee* value was determined by HPLC (Chiralcel OD-H, 99:1 → 98.8:1.2 *n*-hexane/*i*PrOH, 1.5 mL/min).

$C_{11}H_{13}Cl_3O_3S$, **MW:** 331.65 g/mol. **[α]** $_D^{24.9°C}$ (c =0.32, $CHCl_3$) = +23.02 ± 0.79 (sample with *ee* = 99.5%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.98-7.93 (*m*, 2 H, C*H*$_{Ph}$); 7.75-7.57 (*m*, 3 H, C*H*$_{Ph}$); 4.88-4.84 (*m*, 1 H, C*H*-OH); 3.56 (*dd*, *J* = 7.8, 1.9, 1 H, C*H*-S); 3.28 (*d*, *J* = 4.7, 1 H, -O*H*); 2.46-2.30 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 1.95-1.79 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 0.99 (*t*, *J* = 7.5, 3 H, C*H*$_3$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 137.1 (*C*$_{Ph}$); 134.2 (*C*H$_{Ph}$); 129.3 (2x *C*H$_{Ph}$); 129.0 (2x *C*H$_{Ph}$); 101.4 (*C*Cl_3); 78.4 (*C*$_{ring}$); 67.6 (*C*$_{ring}$); 16.9 (*C*H$_2$*C*$_{ring}$); 13.8 (*C*H$_3$). **IR ($CHCl_3$):** ν = 3586, 2974, 2931, 1711, 1308, 1150. **MS (EI) *m/z*:** 330 $[M]^+$, 189 $[M\text{-}PhSO_2]^+$. **HRMS (EI) *m/z*:** Calc. for $[M]^+$: 329.9651. Found: 329.9644. **Anal. Calcd. for $C_{11}H_{13}Cl_3O_3S$**: C, 39.84; H, 3.95. Found: C, 40.11; H, 4.01.

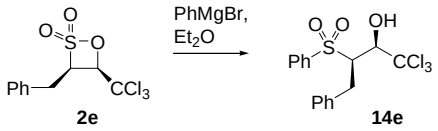
3-Benzenesulfonyl-1,1,1,5-tetrachloropentan-2-ol (14d)



A stirred solution of β-sultone **2d** (15 mg recrystallized from hexane, 0.050 mmol, 1 equiv.) in Et_2O (0.5 mL) was cooled to 4 °C. Then a solution of phenyl magnesium bromide (50 μL, 0.150 mmol, 3M in Et_2O, 3 equiv.) in Et_2O (0.25 mL) was added. The solution was allowed to warm up to room temperature and the flask was subsequently closed. After stirring the reaction mixture at 50 °C for 15 h, it was allowed to cool down to room temperature. Then ice and HCl (1M, 3 mL) were successively added. The aqueous layer was extracted three times with Et_2O, the combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed *in vacuo*. Flash column chromatography on silica (DCM) furnished pure phenyl sulfone **14d** as colorless oil (15 mg, 0.41 mmol, 82%, *ee* > 99.5%). The *ee* value was determined by HPLC (Chiralcel OD-H, 99:1 → 98.4:1.6 *n*-hexane/*i*PrOH, 1.5 mL/min).

$C_{11}H_{12}Cl_4O_3S$, **MW**: 366.09 g/mol. **[α]** $[\alpha]_D^{24.9°C}$ (c =0.6, $CHCl_3$) = +29.37 ± 0.15 (sample with *ee* > 99.5%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.99-7.93 (*m*, 2 H, C*H*$_{Ph}$); 7.77-7.59 (*m*, 3 H, C*H*$_{Ph}$); 4.82-4.78 (*m*, 1 H, C*H*-OH); 4.01 (*dd*, *J* = 8.7, 2.2, 1 H, C*H*-S); 3.85-3.70 (*m*, 2 H, C*H*$_2$Cl); 3.42 (*d*, *J* = 4.4, 1 H, -O*H*); 2.86-2.73 (*m*, 1 H, C*H*$_2$-C$_{ring}$); 2.48-2.35 (*m*, 1 H, C*H*$_2$-C$_{ring}$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 136.7 (*C*$_{Ph}$); 134.5 (*C*H$_{Ph}$); 129.6 (2x*C*H$_{Ph}$); 128.9 (2x *C*H$_{Ph}$); 100.9 (*C*Cl$_3$); 78.4 (*C*$_{ring}$); 62.4 (*C*$_{ring}$); 42.7 (*C*H$_2$Cl); 26.7 (*C*H$_2$-C$_{ring}$). **IR ($CHCl_3$):** ν = 3585, 1448, 1309, 1140. **HRMS (ESI) *m/z*:** Calc. for $[M+Na]^+$: 388.9124. Found: 388.9126. **Anal. Calcd. for $C_{11}H_{12}Cl_4O_3S$**: C, 36.09; H, 3.30. Found: C, 36.39; H, 3.57.

1,1,1-trichloro-4-phenyl-3-(phenylsulfonyl)butan-2-ol (14e)

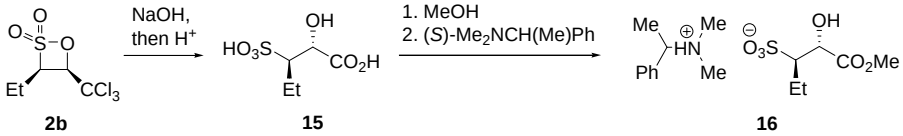


2e **14e**

A stirred solution of β-sultone **2e** (11 mg recrystallized from hexane, *ee* = 96%, 0.033 mmol, 1 equiv.) in Et_2O (1.5 mL) was cooled to 4 °C. Then a solution of phenyl magnesium bromide (33 μL, 0.099 mmol, 3M in Et_2O, 3 equiv.) in Et_2O (0.3 mL) was added. The solution was allowed to warm up to room temperature and the flask was subsequently closed. After stirring the reaction mixture at 50 °C for 17 h, it was allowed to cool down to room temperature. Then ice and HCl (1M, 1 mL) were successively added. The aqueous layer was extracted three times with Et_2O, the combined organic layers were dried over Na_2SO_4, filtered and the solvent was removed *in vacuo*. Flash column chromatography on silica (DCM) furnished pure phenyl sulfone **14e** as colorless oil (12 mg, 0.030 mmol, 90%, *ee* = 98%). The *ee* value was determined by HPLC (Chiralcel OD-H, 98:2 *n*-hexane/*i*PrOH, 1.0 mL/min).

$C_{16}H_{15}Cl_3O_3S$, **MW**: 393.72 g/mol. **$[\alpha]_D^{24.9°C}$** (c =0.65, $CHCl_3$) = +4.84 ± 1.12 (sample with *ee*=98%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.70-7.65 (*m*, 2 H, C*H*$_{Ph}$); 7.55-7.48 (*m*, 1 H, C*H*$_{Ph}$); 7.41-7.33 (*m*, 2 H, C*H*$_{Ph}$); 7.15-7.01 (*m*, 5 H, C*H*$_{Ph}$); 5.21-5.17 (*m*, 1 H, C*H*-OH); 4.29-4.22 (*m*, 1 H, C*H*-S); 3.78 (*dd*, *J* = 16.8, 2.2, 1 H, C*H*$_2$-C$_{ring}$); 3.58 (*d*, *J* = 4.9, 1 H, -OH); 3.17 (*dd*, *J* = 16.8, 9.3, 1 H, C*H*$_2$-C$_{ring}$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 137.8 (*C*$_{Ph}$); 137.4 (*C*$_{Ph}$); 133.8 (*C*H$_{Ph}$); 129.0 (2x *C*H$_{Ph}$); 128.5 (4x *C*H$_{Ph}$); 128.2 (2x *C*H$_{Ph}$); 126.3 (*C*H$_{Ph}$); 101.7 (*C*Cl$_3$); 77.8 (*C*$_{ring}$); 66.8 (*C*$_{ring}$); 29.7 (*C*H$_2$-C$_{ring}$). **IR ($CHCl_3$):** ν = 3690, 3586, 1604, 1498, 1448, 1308, 1148 cm^{-1}. **HRMS (ESI) *m/z*:** Calc. for [M+Na]$^+$: 416.9670. Found: 416.9666. **Anal. Calcd. for $C_{16}H_{15}Cl_3O_3S$**: C, 48.81; H, 3.84. Found: C, 49.02; H, 4.14.

2-Hydroxy-3-sulfopentanoic acid methyl ester (*S*)-(–)-*N*,*N*-dimethyl-1-phenylethylammonium salt (16)

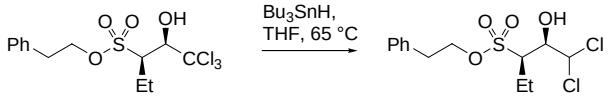


β-sultone **2b** (19 mg, batch with *ee* = 95% (determined as phenethyl sulfonate **11b**), 0.075 mmol, 1 equiv.) was stirred in aqueous NaOH (466 µL, 0.466 mmol, 6.25 equiv.) at room temperature for 18h. The reaction mixture was filtered over an acidic cation exchanger resin (Amberlite IR-120, 2.5 g) and eluted with water until neutral. Concentrating the filtrate *in vacuo* furnished diacid **15** as slightly yellow oil (16 mg, 0.075 mmol, 1 equiv.), which was dissolved in 1 mL of MeOH and stirred for 14 h at room temperature. Afterwards the solvent was removed *in vacuo* and the residue was dried using azeotropic evaporation with toluene and subsequently with MeOH (twice) to furnish the corresponding monomethylester as pale yellow oil, which was employed in the next step without purification. The ester was dissolved in MeOH (1 mL) and a solution of (*S*)-(–)-*N*,*N*-dimethyl-1-phenylethylamine (13 µL, 0.075 mmol, 1 equiv.) in MeOH (1 mL) was added at room temperature. After 5 h the solvent was removed *in vacuo*. The residue was dissolved in a minimum amount of DCM and hexane was added until precipitation of the corresponding ammonium salt **16**, which was obtained as a pale yellow oil (24 mg, 0.066 mmol, 88% over 3 steps).

$C_{16}H_{27}NO_6S$, **MW:** 361.45 g/mol. **[α]** $[\alpha]_D^{24.9°C}$ (c =1.15, $CHCl_3$) = –5.36 ± 0.75. **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.45 (*b*, 5 H, C*H*$_{Ph}$); 4.86 (*b*, 1 H, N*H*$^+$); 4.44, 4.33 (*b*, 2 H, C*H*-OH, NC*H*CH_3); 3.76 (*b*, 3 H, COOC*H*$_3$); 3.49 (*b*, 1 H, C*H*-S); 3.32 (*b*, 1 H, -O*H*); 2.83 (*b*, 3 H, NC*H*$_3$); 2.67 (*b*, 3 H, NC*H*$_3$); 2.19 (*b*, 1 H, C*H*$_2$$CH_3$); 1.99 (*b*, 1 H, C*H*$_2$$CH_3$); 1.78 (*b*, 3 H, CHC*H*$_3$); 1.11 (*b*, 3 H, CH_2C*H*$_3$). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 174.5 (*C*OOMe); 132.8 (*C*$_{Ph}$); 130.4 (*C*H$_{Ph}$); 129.4 (2x*C*H$_{Ph}$); 129.1 (2x *C*H$_{Ph}$); 69.2 (*C*H-O); 66.3 (N*C*HCH_3);

63.3 (*C*H-S); 52.2 (COO*C*H_3); 42.3 (N*C*H_3); 39.1 (N*C*H_3); 20.3 (CH*C*H_3); 16.8 (*C*H_2CH$_3$); 11.7 (*C*H_3). **IR ($CHCl_3$):** ν = 3423, 1745, 1459, 1243, 1144, 909. **HRMS (ESI) *m/z*:** Calc. for $[Cation]^+$: 150.1277. Found: 150.1280. Calc. for $[Anion]^+$: 211.0282. Found: 211.0285. **Anal. Calcd. for $C_{16}H_{27}NO_6S$**: a satisfactory microanalysis could not be obtained due to hygroscopicity.

1,1-Dichloro-2-hydroxypentane-3-sulfonic acid phenethyl ester (17)

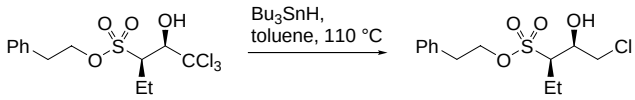


11b **17**

To a solution of sulfonate **11b** (18 mg, batch with *ee* = 90%, 0.046 mmol, 1 equiv.) in THF (1.5 mL) a solution of tributyltin hydride (55 µL, 0.205 mmol, 4.5 equiv.) in THF (0.5 mL) was added at room temperature and the flask was closed. After stirring the reaction mixture at 60 °C for 14 h, the solvent was removed *in vacuo*. The residue was dissolved in MeCN and extracted three times with hexane to remove all tin derivatives. Flash column chromatography on silica (DCM) furnished pure dichloro sulfonate **17** as colorless oil (11 mg, 0.034 mmol, 70%).

$C_{13}H_{18}Cl_2O_4S$, **MW:** 341.26 g/mol. **$[\alpha]_D^{24.9°C}$** (c =0.7, $CHCl_3$) = +12.82 ± 0.52 (sample with *ee* = 90%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.39-7.20 (*m*, 5 H, CH_{Ph}); 5.80 (*d*, *J* = 5.9, 1 H, CHCl$_2$); 4.52-4.44 (*m*, 2 H, CH_2-O); 4.42-4.36 (*m*, 1 H, C*H*-OH); 3.46-3.41 (*m*, 1 H, C*H*-S); 3.08 (*t*, *J* = 6.9, 3 H, CH_2-Ph); 2.83 (*d*, *J* = 4.4, 1 H, -O*H*); 2.06-1.89 (*m*, 2 H, CH_2-C_{ring}); 1.14-1.06 (*m*, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 135.9 (C_{Ph}); 128.8 (2x CH_{Ph}); 128.7 (2x CH_{Ph}); 127.1 (CH_{Ph}); 74.4, 73.8 ($CHCl_2$, C_{ring}); 70.3 (CH_2O); 63.5 (C_{ring}); 35.7 (CH_2Ph); 18.0 (CH_2-C_{ring}); 12.3 (CH_3). **IR ($CHCl_3$):** ν = 3584, 2967, 1404, 1456, 1351. **HRMS (ESI) *m/z*:** Calc. for $[M+Na]^+$: 363.0195. Found: 363.0202. **Anal. Calcd. for $C_{13}H_{18}Cl_2O_4S$**: C, 45.76; H, 5.32. Found: C, 45.91; H, 5.26.

1-Chloro-2-hydroxypentane-3-sulfonic acid phenethyl ester (18)



11b **18**

To a solution of sulfonate **11b** (23 mg, batch with *ee* = 80%, 0.058 mmol, 1 equiv.) in toulene (2 mL) a solution of tributyltin hydride (55 µL, 0.205 mmol, 4.5 equiv.) in toluene (0.5 mL) was added at room temperature and the flask was closed. After stirring the reaction mixture at 110 °C for 14 h, the solvent was removed *in vacuo*. The residue was dissolved in MeCN and extracted three times with hexane to remove all tin derivatives. Flash column chromatography on silica (DCM) furnished pure monochloro sulfonate **18** as colorless oil (15 mg, 0.049 mmol, 83%).

$C_{13}H_{19}ClO_4S$, **MW:** 306.81 g/mol. **[α]** $_D^{24.9°C}$ (c =0.8, $CHCl_3$) = +7.50 ± 0.42 (sample with *ee* = 80%). **^{1}H NMR (300 MHz, $CDCl_3$, 21 °C):** δ = 7.38-7.20 (*m*, 5 H, CH_{Ph}); 4.53-4.41 (*m*, 2 H, CH_2-O); 4.33-4.25 (*m*, 1 H, C*H*-OH); 3.63 (*dd*, *J* = 11.2, 6.5, 1 H, CH_2Cl); 3.50 (*dd*, *J* = 11.2, 6.5, 1 H, CH_2Cl); 3.25-3.18 (*m*, 1 H, C*H*-S); 3.08 (*t*, *J* = 6.9, 3 H, CH_2-Ph); 2.75 (*d*, *J* = 4.7, 1 H, -O*H*); 2.04-1.73 (*m*, 2 H, CH_2-C_{ring}); 1.14-1.07 (*m*, 3 H, CH_3). **^{13}C NMR (75 MHz, $CDCl_3$, 21 °C):** δ = 133.1 (C_{Ph}); 128.9 (2x *C*H$_{Ph}$); 128.8 (2x *C*H$_{Ph}$); 127.12 (*C*H$_{Ph}$); 70.2 (*C*H_2O); 69.7 (C_{ring}); 64.1 (C_{ring}); 45.2 (*C*H_2Cl); 35.6 (*C*H_2Ph); 17.5 (*C*H_2-C_{ring}); 12.6 (*C*H_3). **IR ($CHCl_3$):** ν = 3568, 2964, 1456, 1350, 1168. **HRMS (ESI) *m/z*:** Calc. for [M+Na] $^+$: 329.0585. Found: 329.0592. **Anal. Calcd. for $C_{13}H_{19}ClO_4S$**: C, 50.89; H, 6.24. Found: C, 50.74; H, 6.37.

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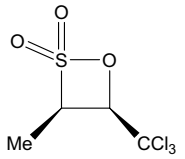
F.Koch/Peters FK003-4 OPR:PZ
400 MHz 1H-NMR



2a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PROCNO                   1

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1D NMR plot parameters
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F.Koch/Peters FK003-4 OPR:PZ
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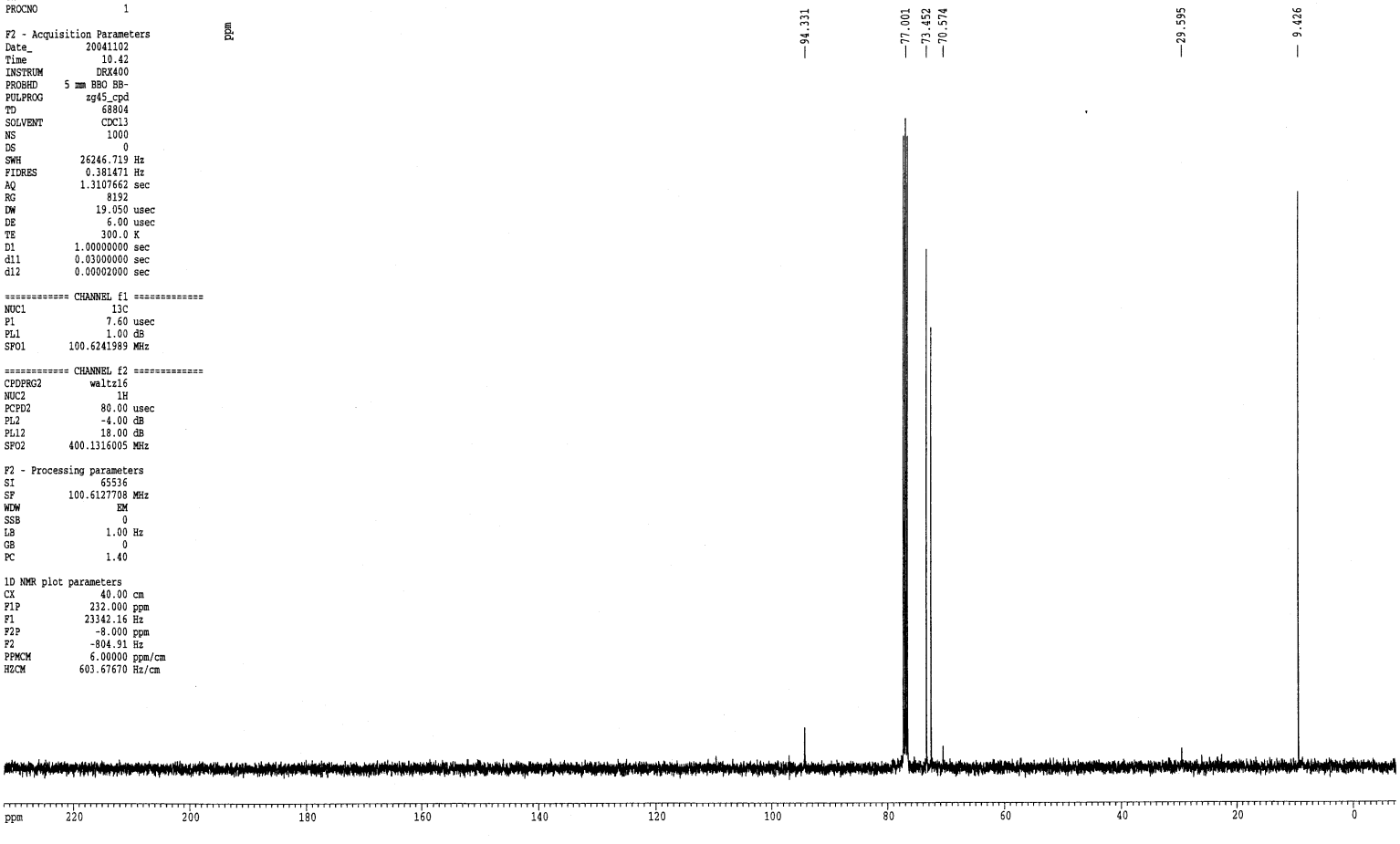

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PROCNO 1

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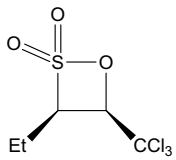
Koch/Peters FK058-1 OPR:PZ
400 MHz 1H-NMR



2b

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PROCNO 1

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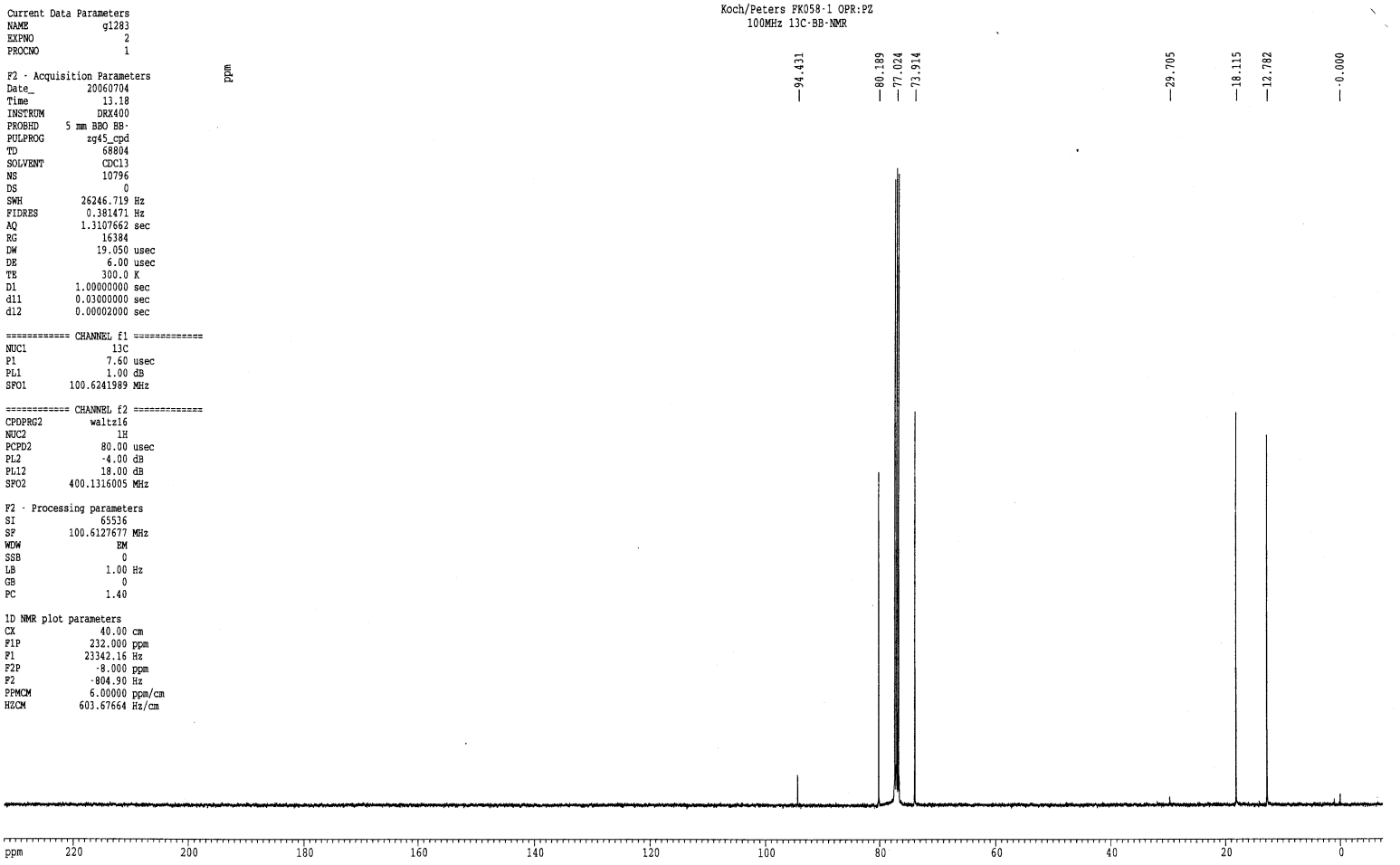
Koch/Peters FK058-1 OPR:PZ
100MHz 13C-BB-NMR



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========== CHANNEL f1 ==========
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1D NMR plot parameters
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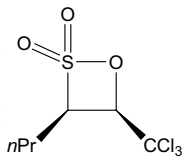
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.03250 Hz/cm

Koch/Peters FK074-1K1 OPR:PZ
400 MHz 1H-NMR



2c

Current Data Parameters
NAME g1282
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060704
Time 1.23
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg45_cpd
TD 68804
SOLVENT CDCl3
NS 14000
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 16384
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec

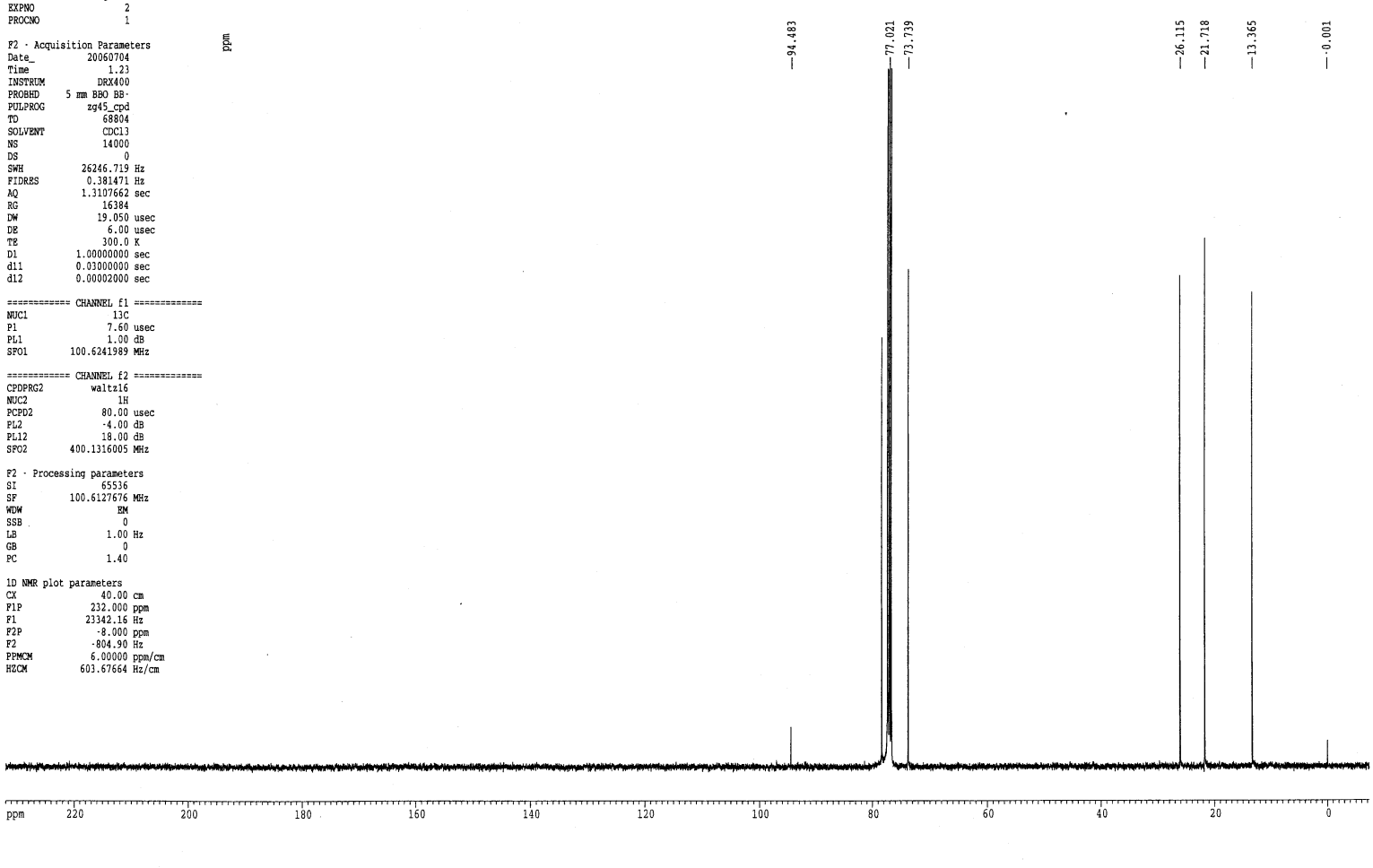
========== CHANNEL f1 ==========
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz

========== CHANNEL f2 ==========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 65536
SF 100.6127676 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

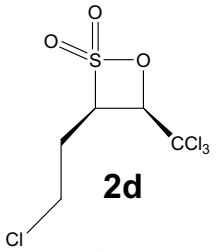
1D NMR plot parameters
CX 40.00 cm
F1P 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.90 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67664 Hz/cm

Koch/Peters FK074-1K1 OPR:PZ
100MHz 13C-BB-NMR



```
Current Data Parameters
NAME                  g1291
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060713
Time                  8.46
INSTRUM             DRX400
PROBHD      5 mm BBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      64
DS                       0
SWH               6410.256 Hz
FIDRES           0.097813 Hz
AQ                5.1118579 sec
RG                    322.5
DW                   78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1             1.00000000 sec

============ CHANNEL f1 =============
NUC1                    1H
P1                    8.20 usec
PL1                  -4.00 dB
SFO1          400.1325608 MHz

F2 - Processing parameters
SI                   65536
SF            400.1300085 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   40.00 cm
F1P                   9.500 ppm
F1                 3801.24 Hz
F2P                  -0.500 ppm
F2                 -200.07 Hz
PPMCM              0.25000 ppm/cm
HZCM             100.03250 Hz/cm
```

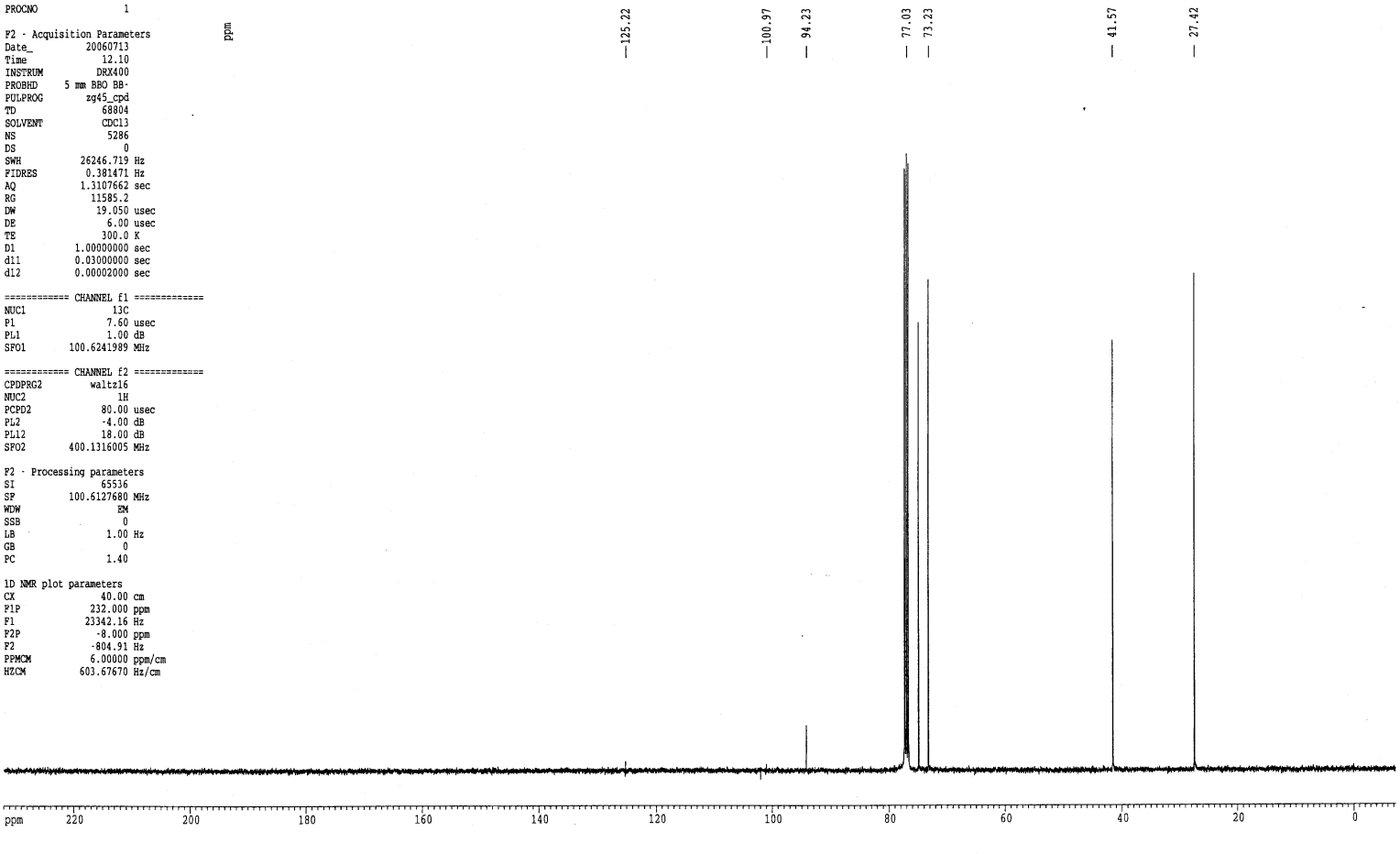
F.Koch/Peters FK062-2-K2 OPR:PZ
400 MHz 1H-NMR



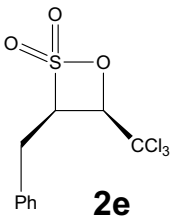
2d


```
Current Data Parameters
NAME                  g1291
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060713
Time                 12.10
INSTRUM             DRX400
PROBHD      5 mm BBO BB-
PULPROG           zg45_cpd
TD                   68804
SOLVENT              CDCl3
NS                    5286
DS                       0
SWH              26246.719 Hz
FIDRES           0.381471 Hz
AQ                1.3107662 sec
RG                 11585.2
DW                   19.050 usec
DE                    6.00 usec
TE                   300.0 K
D1             1.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                    7.60 usec
PL1                   1.00 dB
SFO1          100.6241989 MHz

============ CHANNEL f2 =============
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 18.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   65536
SF            100.6127680 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   40.00 cm
F1P                 232.000 ppm
F1                23342.16 Hz
F2P                  -8.000 ppm
F2                 -804.91 Hz
PPMCM              6.00000 ppm/cm
HZCM             603.67670 Hz/cm
```

F.Koch/Peters FK062-2-K2 OPR:PZ
100MHz 13C-BB-NMR



F.Koch/Peters FK102-1K1 OPR:PZ
400 MHz 1H-NMR

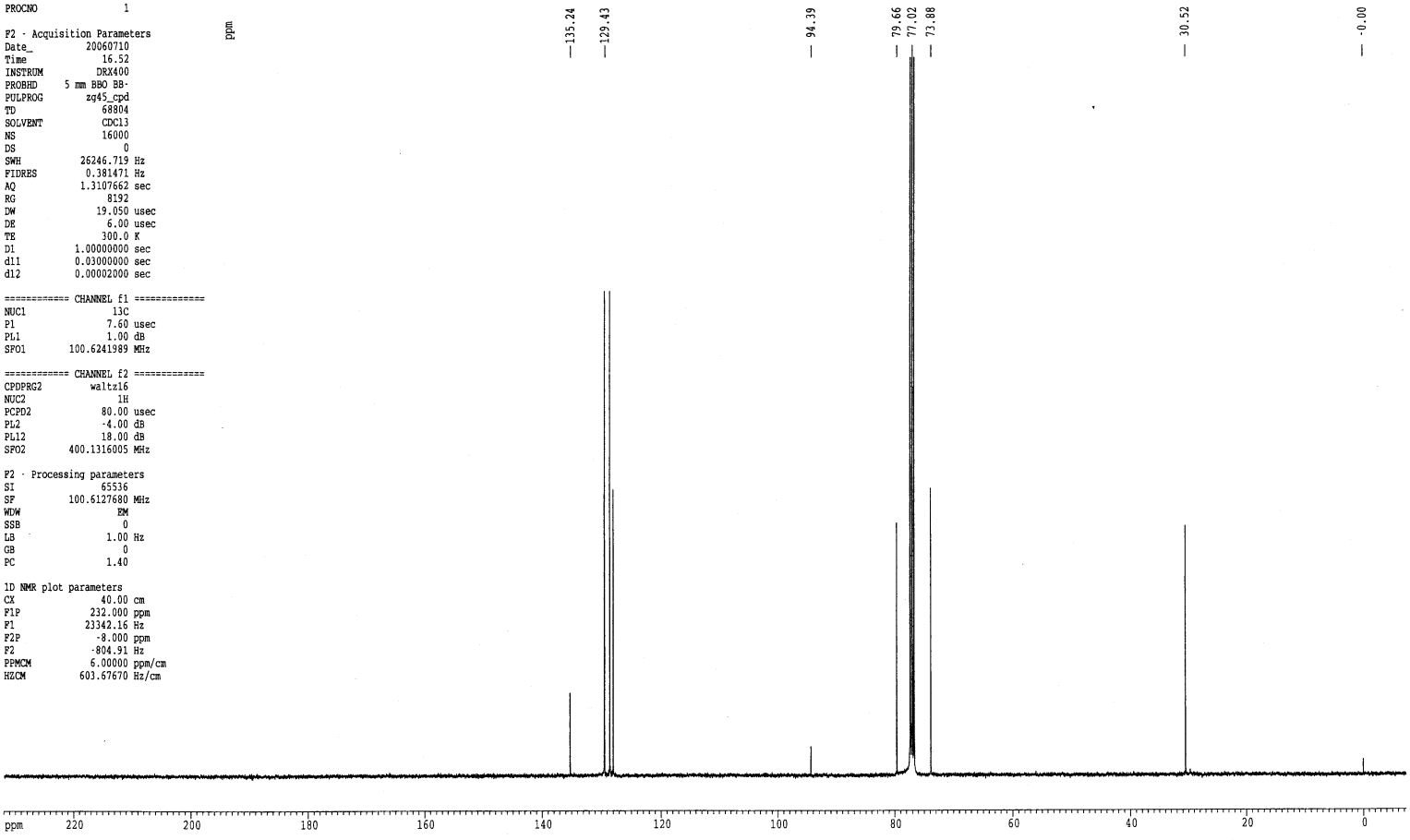


2e

```
Current Data Parameters
NAME                  g1289
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060710
Time                 13.36
INSTRUM              DRX400
PROBHD     5 mm BBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        0
SWH               6410.256 Hz
FIDRES               0.097813 Hz
AQ                5.1118579 sec
RG                     574.7
DW                  78.000 usec
DE                   6.00 usec
TE                   300.0 K
D1              1.00000000 sec

============ CHANNEL f1 =============
NUC1                     1H
P1                    8.20 usec
PL1                  -4.00 dB
SFO1           400.1325608 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300101 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    40.00 cm
F1P                   9.500 ppm
F1                  3801.24 Hz
F2P                  -0.500 ppm
F2                  -200.07 Hz
PPMCM              0.25000 ppm/cm
HZCM             100.03250 Hz/cm
```

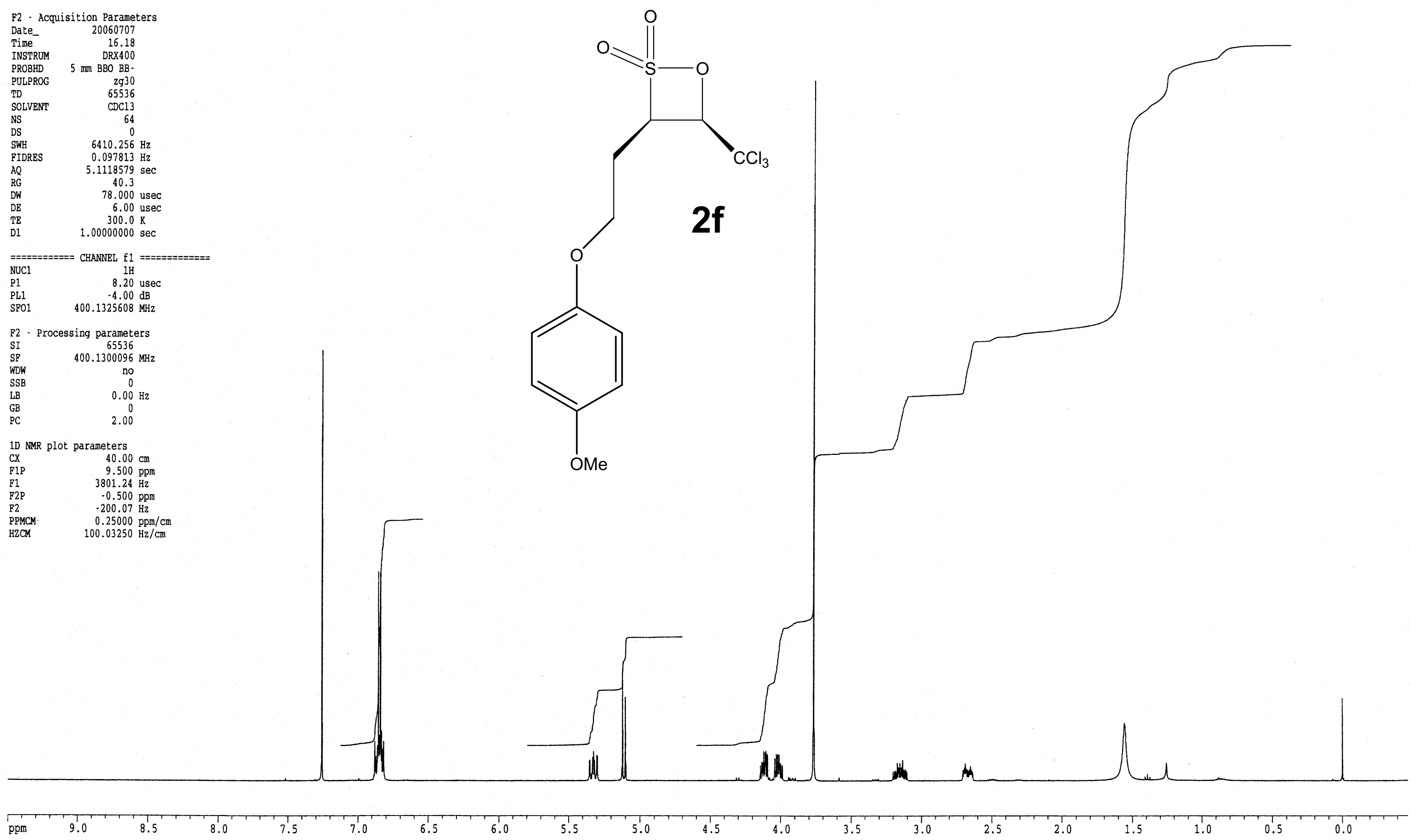

F.Koch/Peters FK102-1K1 OPR:PZ
100MHz 13C-BB-NMR

```
Current Data Parameters
NAME                  g1289
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20060710
Time                 16.52
INSTRUM              DRX400
PROBHD     5 mm BBO BB-
PULPROG             zg45_cpd
TD                    68804
SOLVENT               CDCl3
NS                    16000
DS                        0
SWH              26246.719 Hz
FIDRES               0.381471 Hz
AQ                1.3107662 sec
RG                     8192
DW                  19.050 usec
DE                   6.00 usec
TE                   300.0 K
D1              1.00000000 sec
d11             0.03000000 sec
d12             0.00002000 sec

============ CHANNEL f1 =============
NUC1                    13C
P1                    7.60 usec
PL1                   1.00 dB
SFO1           100.6241989 MHz

============ CHANNEL f2 =============
CPDPRG2             waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                    65536
SF             100.6127680 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    40.00 cm
F1P                  232.000 ppm
F1                 23342.16 Hz
F2P                   -8.000 ppm
F2                  -804.91 Hz
PPMCM              6.00000 ppm/cm
HZCM             603.67670 Hz/cm
```



F.Koch/Peters FK106-1 OPR:RF
400 MHz 1H-NMR

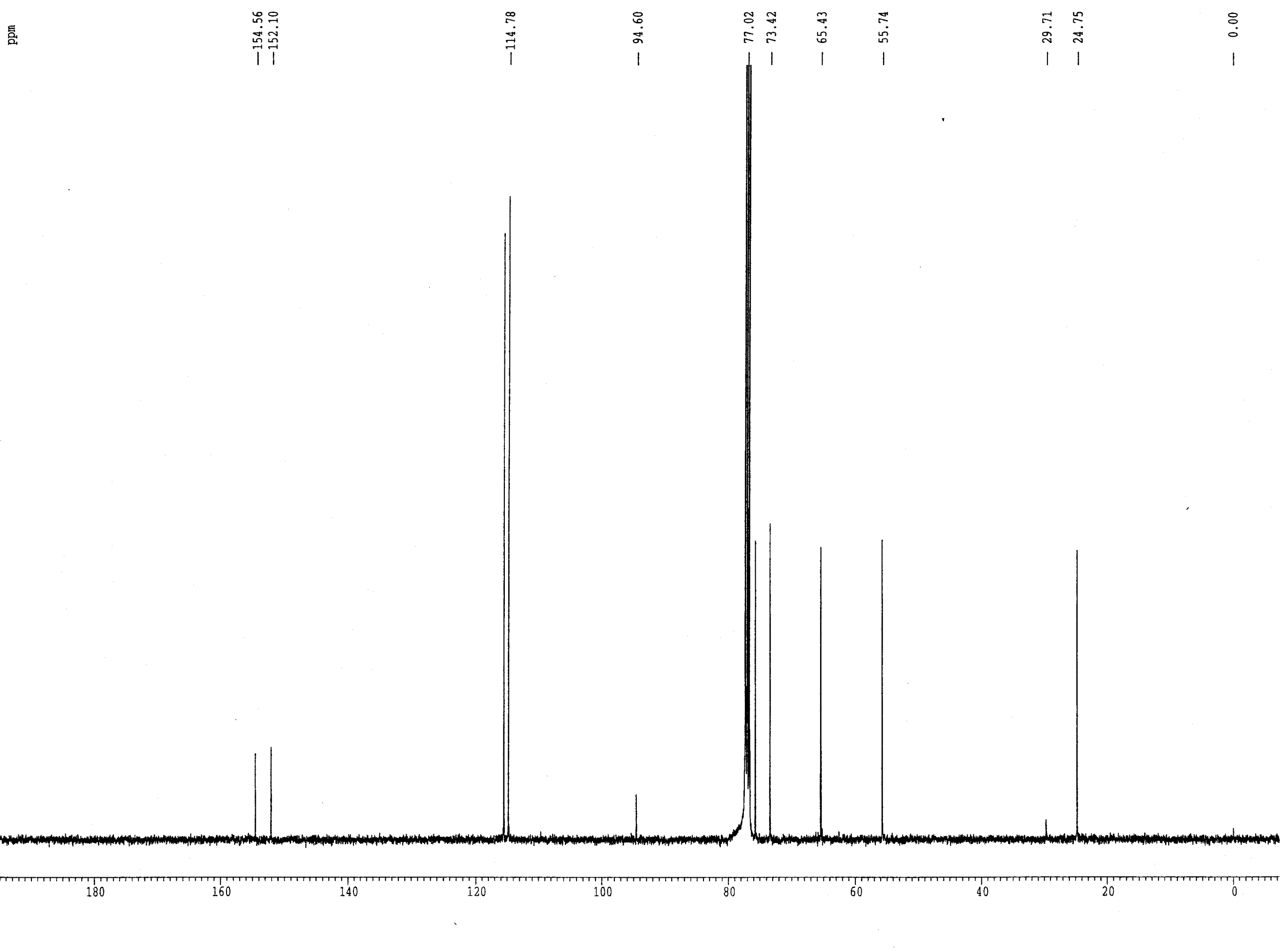
```
Current Data Parameters
NAME            g1288
EXPNO               1
PROCNO              1

F2 - Acquisition Parameters
Date_        20060707
Time            16.18
INSTRUM        DRX400
PROBHD   5 mm BBO BB-
PULPROG          zg30
TD              65536
SOLVENT         CDCl3
NS                 64
DS                  0
SWH          6410.256 Hz
FIDRES       0.097813 Hz
AQ          5.1118579 sec
RG               40.3
DW             78.000 usec
DE               6.00 usec
TE              300.0 K
D1         1.00000000 sec

============ CHANNEL f1 ============
NUC1               1H
P1               8.20 usec
PL1             -4.00 dB
SFO1      400.1325608 MHz

F2 - Processing parameters
SI              65536
SF        400.1300096 MHz
WDW                no
SSB                 0
LB               0.00 Hz
GB                  0
PC               2.00

1D NMR plot parameters
CX              40.00 cm
F1P             9.500 ppm
F1            3801.24 Hz
F2P            -0.500 ppm
F2            -200.07 Hz
PPMCM         0.25000 ppm/cm
HZCM        100.03250 Hz/cm
```



2f

F.Koch/Peters FK106-1 OPR:RF
100MHz 13C-BB-NMR

```
Current Data Parameters
NAME            g1288
EXPNO               2
PROCNO              1

F2 - Acquisition Parameters
Date_        20060707
Time            16.38
INSTRUM        DRX400
PROBHD   5 mm BBO BB-
PULPROG      zg45_cpd
TD              68804
SOLVENT         CDCl3
NS              30000
DS                  0
SWH         26246.719 Hz
FIDRES       0.381471 Hz
AQ          1.3107662 sec
RG               8192
DW             19.050 usec
DE               6.00 usec
TE              300.0 K
D1         1.00000000 sec
d11        0.03000000 sec
d12        0.00002000 sec

============ CHANNEL f1 ============
NUC1              13C
P1               7.60 usec
PL1              1.00 dB
SFO1      100.6241989 MHz

============ CHANNEL f2 ============
CPDPRG2       waltz16
NUC2               1H
PCPD2           80.00 usec
PL2             -4.00 dB
PL12            18.00 dB
SFO2      400.1316005 MHz

F2 - Processing parameters
SI              65536
SF        100.6127676 MHz
WDW                EM
SSB                 0
LB               1.00 Hz
GB                  0
PC               1.40

1D NMR plot parameters
CX              40.00 cm
F1P           232.000 ppm
F1           23342.16 Hz
F2P            -8.000 ppm
F2            -804.91 Hz
PPMCM         6.00000 ppm/cm
HZCM        603.67670 Hz/cm
```



LOC ETHZ NMR Mercury 300MHz Nr.4 11/14/06 21:07:33 USER:rkoch GROUP:peters SAMPLE:fk130-8-f7-10

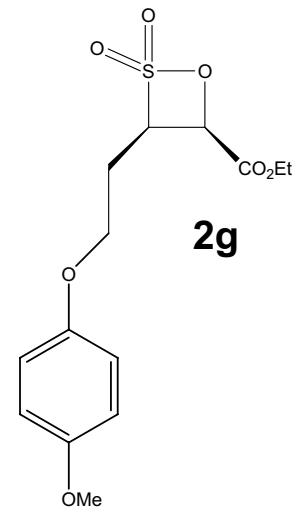
STANDARD 1H OBSERVE

Sample directory: fk130-8-f7-10

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk130-8-f7-10
INOVA-500 "nmroc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
256 repetitions
OBSERVE H1, 299.9012507 MHz
DATA PROCESSING
FT size 32768
Total time 28 min, 10 sec



2g

LOC ETHZ NMR Gemini 300 MHz Nr.3 11/14/06 21:10:05 USER:rkoch GROUP:peters SAMPLE:fk130-8-f2-6

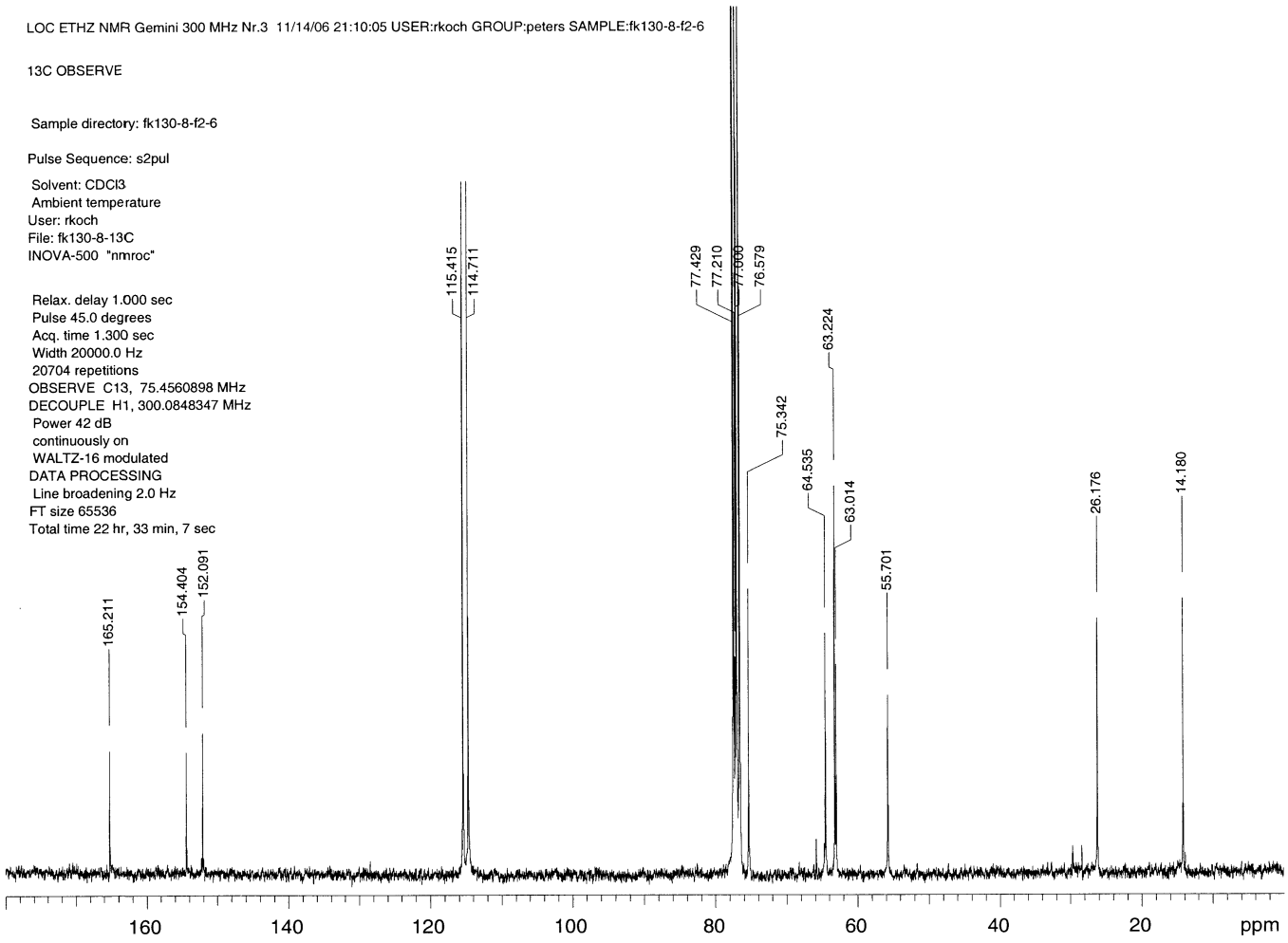
13C OBSERVE

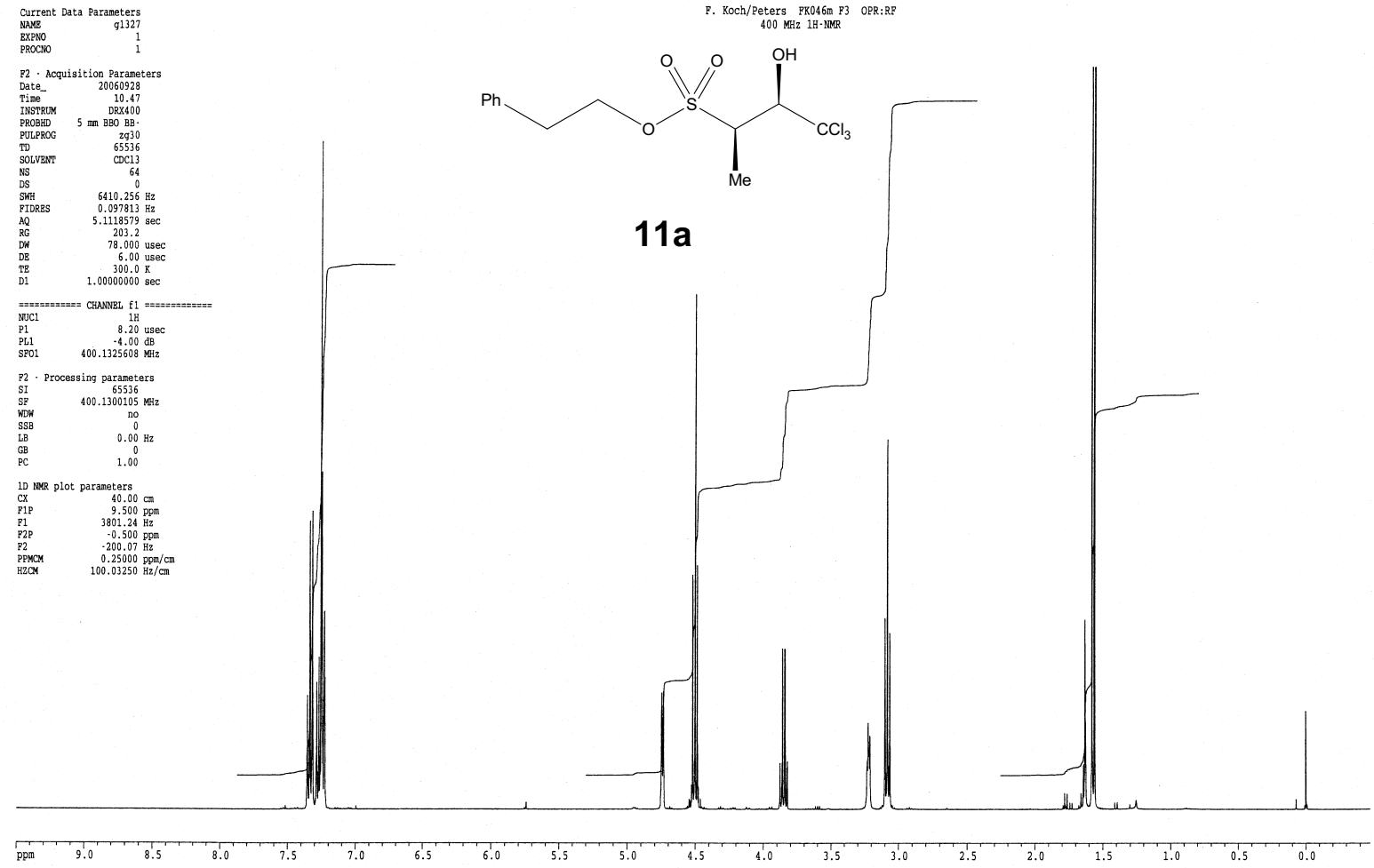
Sample directory: fk130-8-f2-6

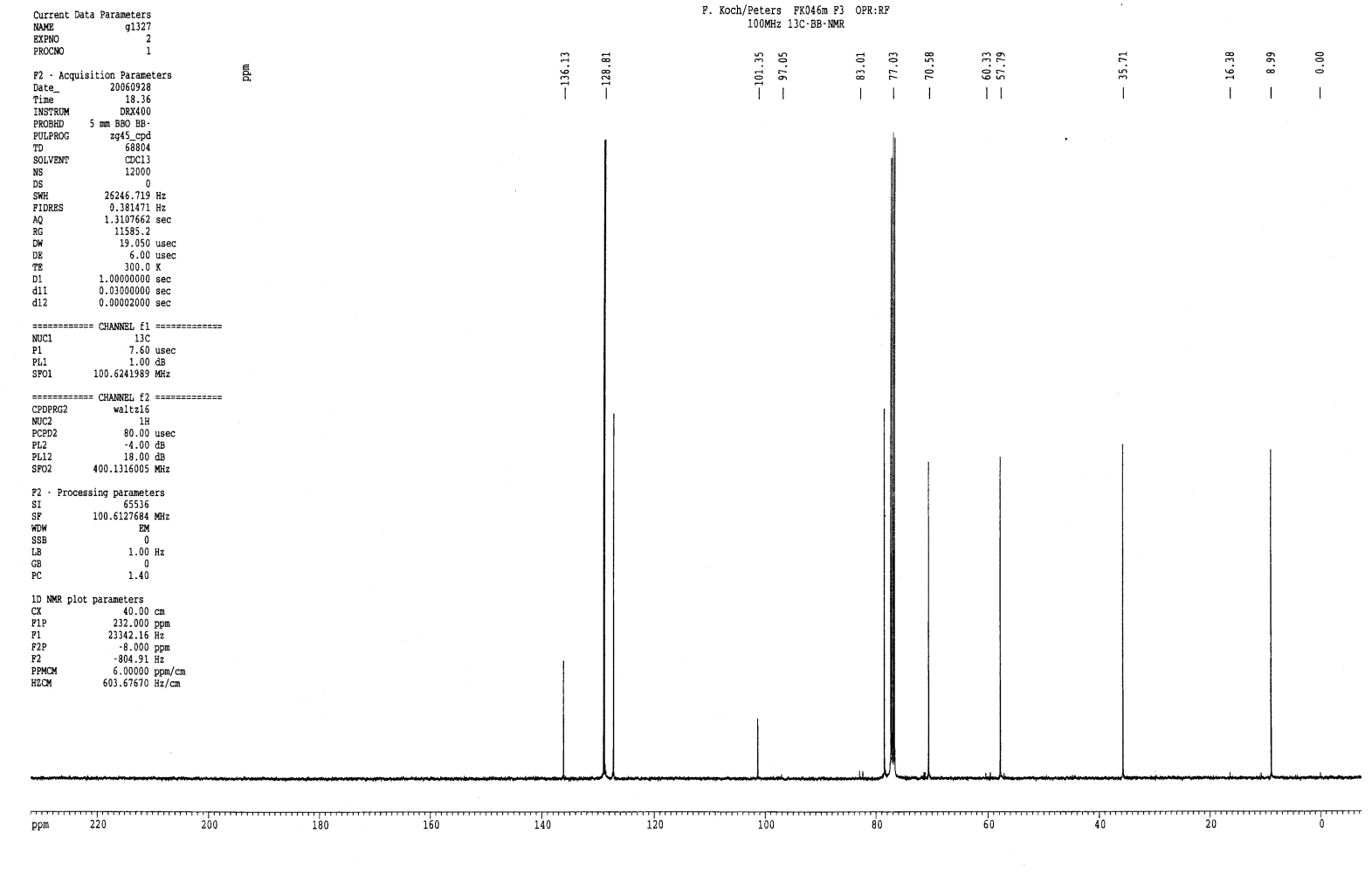
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk130-8-13C
INOVA-500 "nmroc"

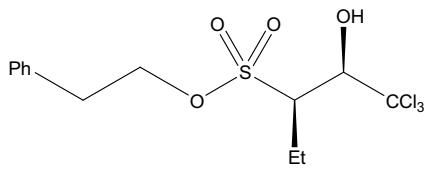
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
20704 repetitions
OBSERVE C13, 75.4560898 MHz
DECOUPLE H1, 300.0848347 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 2.0 Hz
FT size 65536
Total time 22 hr, 33 min, 7 sec




Current Data Parameters
NAME g1327
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060928
Time 10.47
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 203.2
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
============ CHANNEL f1 ============
NUC1 1H
P1 8.20 usec
PL1 -4.00 dB
SFO1 400.1325608 MHz
F2 - Processing parameters
SI 65536
SF 400.1300105 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 40.00 cm
F1P 9.500 ppm
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.03250 Hz/cm
F. Koch/Peters FK046m F3 OPR:RF
400 MHz 1H-NMR
Ph
O
O
S
O
OH
CCl3
Me
11a
ppm 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0


Current Data Parameters
NAME g1327
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060928
Time 18.36
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg45_cpd
TD 68804
SOLVENT CDCl3
NS 12000
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 11585.2
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
============ CHANNEL f1 ============
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz
============ CHANNEL f2 ============
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 65536
SF 100.6127684 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 40.00 cm
F1P 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cm
F. Koch/Peters FK046m F3 OPR:RF
100MHz 13C-BB-NMR
ppm
136.13
128.81
101.35
97.05
83.01
77.03
70.58
60.33
57.79
35.71
16.38
8.99
0.00
ppm 220 200 180 160 140 120 100 80 60 40 20 0

Koch/Peters FK059m-P6 OPR:RF
400 MHz 1H-NMR

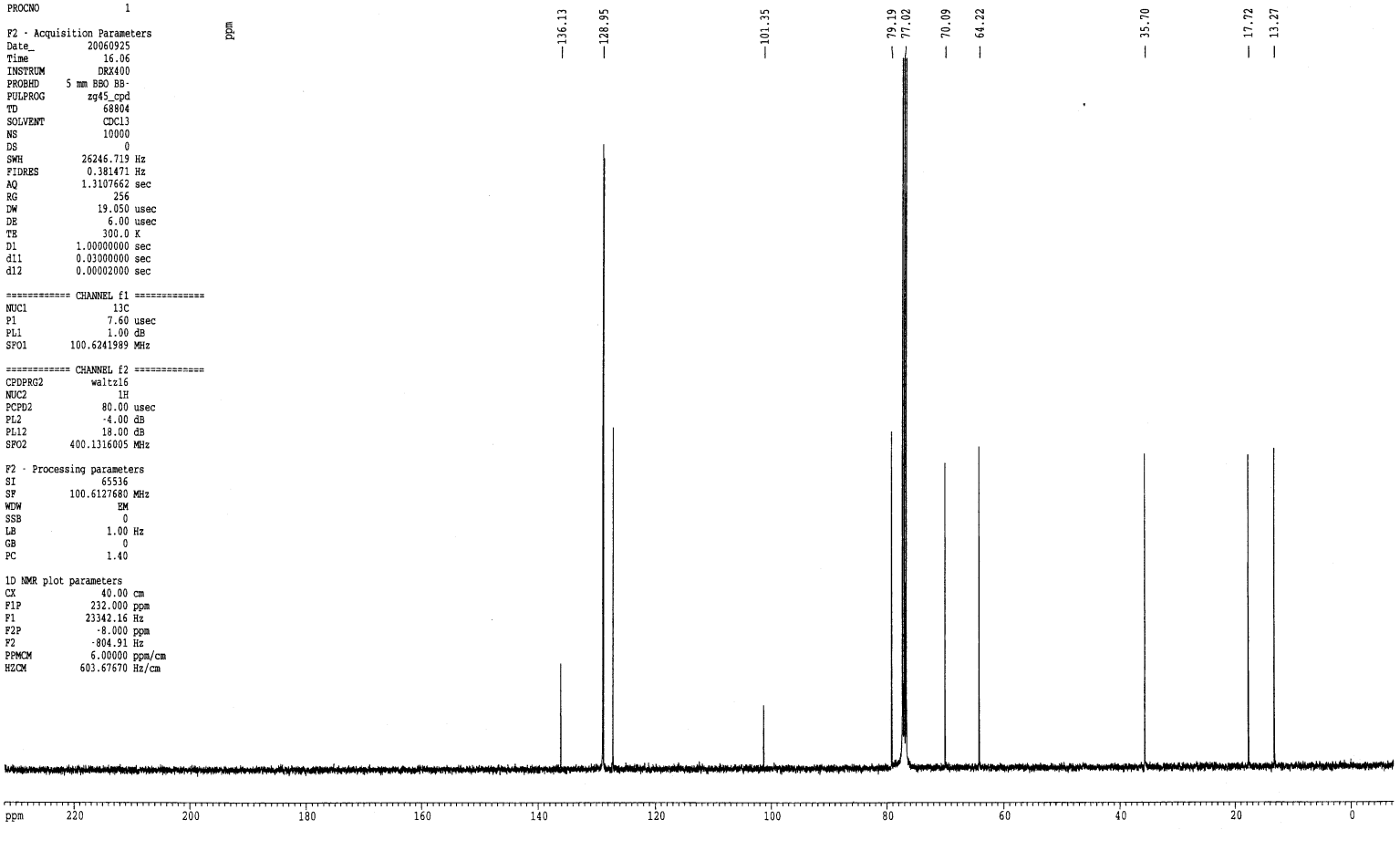


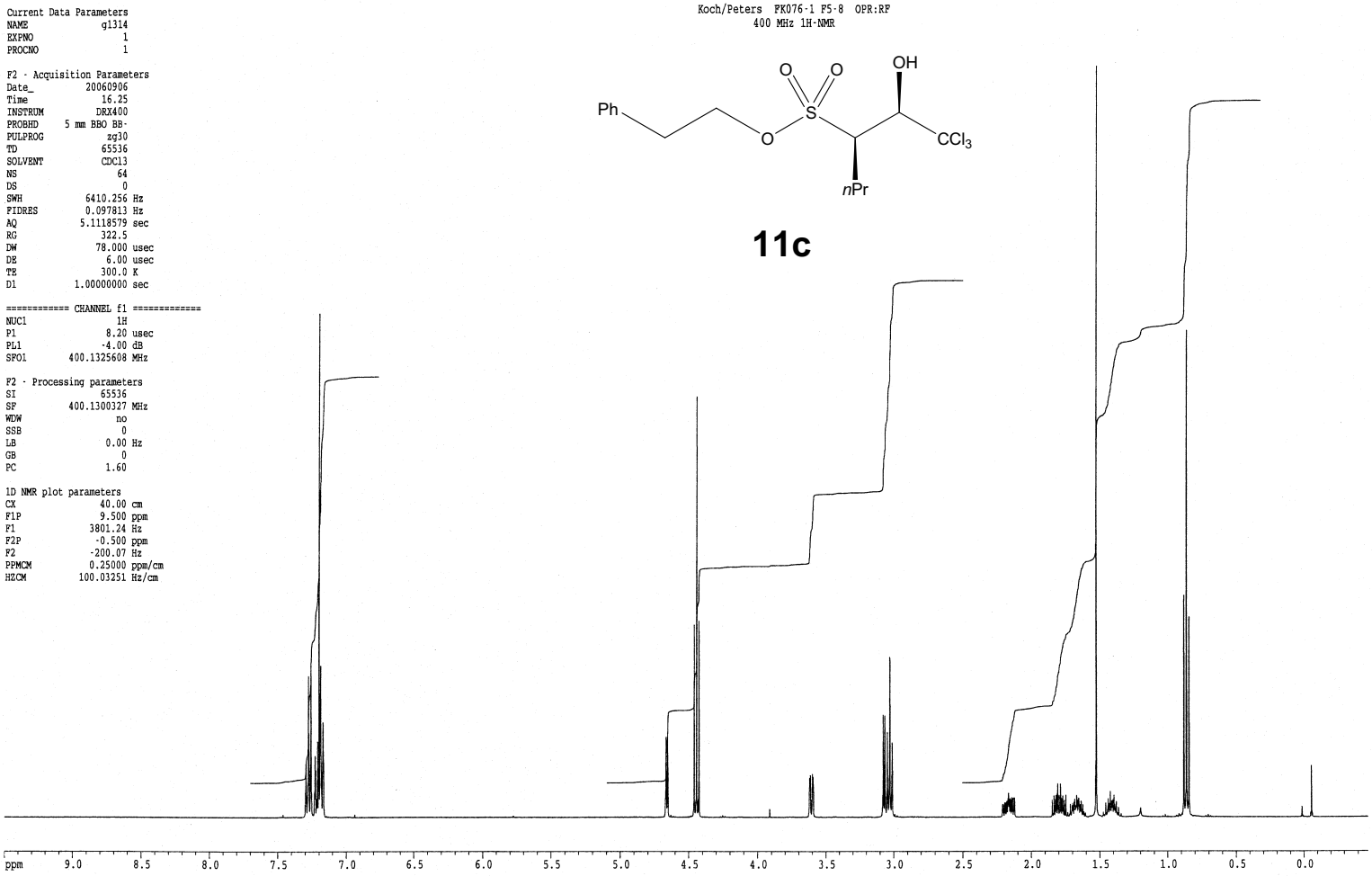
11b

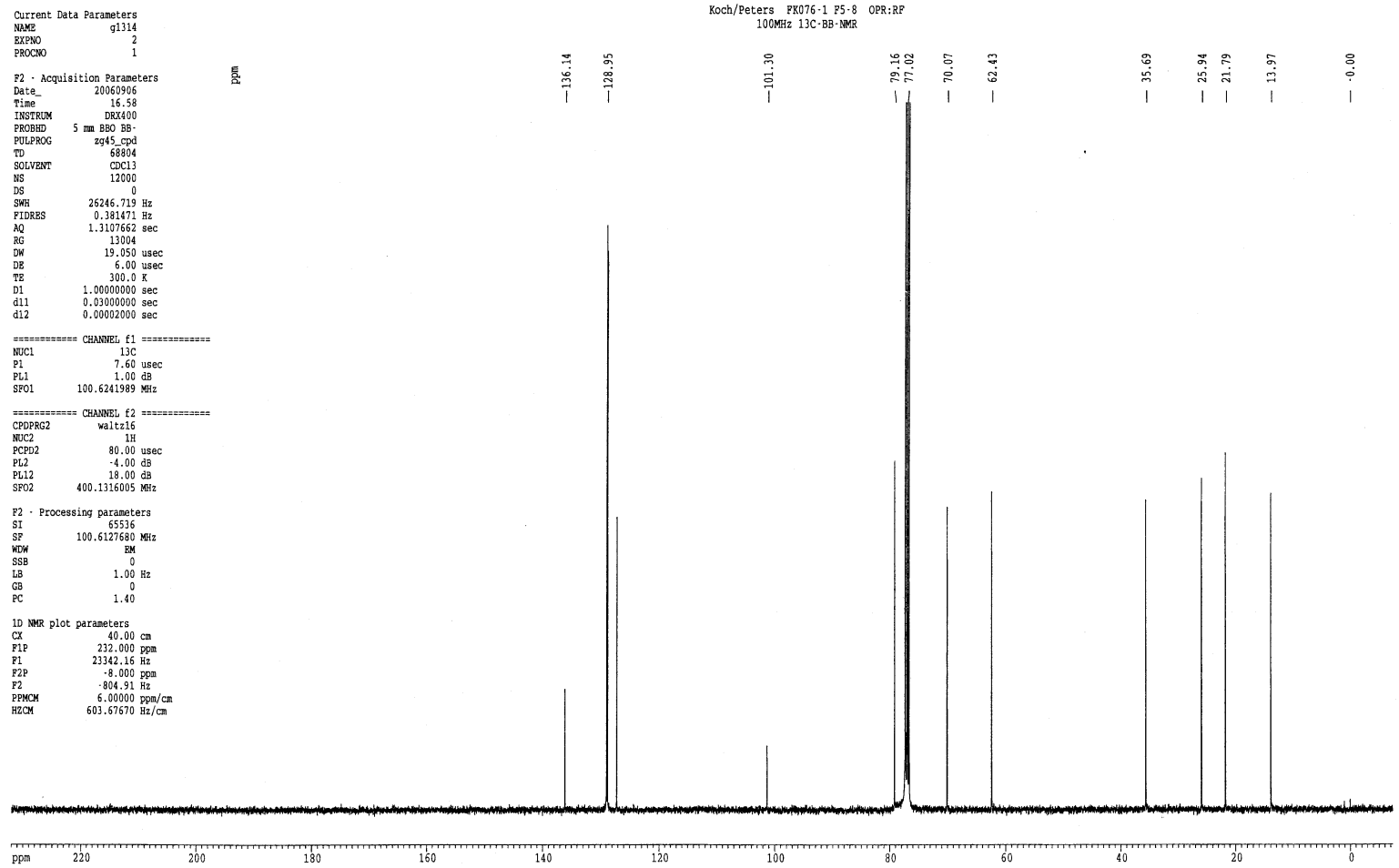
```
Current Data Parameters
NAME                  gl325
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060925
Time                  14.53
INSTRUM              DRX400
PROBHD      5 mm BBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        0
SWH                6410.256 Hz
FIDRES            0.097813 Hz
AQ                5.1118579 sec
RG                      362
DW                   78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 =============
NUC1                    1H
P1                    8.20 usec
PL1                  -4.00 dB
SFO1           400.1325608 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300101 MHz
WDW                      no
SSB                       0
LB                     0.00 Hz
GB                        0
PC                     2.00

1D NMR plot parameters
CX                    40.00 cm
F1P                    9.500 ppm
F1                  3801.24 Hz
F2P                   -0.500 ppm
F2                  -200.07 Hz
PPMCM                0.25000 ppm/cm
HZCM               100.03250 Hz/cm
```


Koch/Peters FK059m-P6 OPR:RF
100MHz 13C-BB-NMR

```
Current Data Parameters
NAME                  gl325
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20060925
Time                  16.06
INSTRUM              DRX400
PROBHD      5 mm BBO BB-
PULPROG            zg45_cpd
TD                    68804
SOLVENT               CDCl3
NS                    10000
DS                        0
SWH               26246.719 Hz
FIDRES            0.381471 Hz
AQ                1.3107662 sec
RG                      256
DW                   19.050 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                    7.60 usec
PL1                   1.00 dB
SFO1           100.6241989 MHz

============ CHANNEL f2 =============
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                    65536
SF             100.6127680 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    40.00 cm
F1P                  232.000 ppm
F1                 23342.16 Hz
F2P                   -8.000 ppm
F2                  -804.91 Hz
PPMCM                6.00000 ppm/cm
HZCM               603.67670 Hz/cm
```




Current Data Parameters
NAME g1314
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060906
Time 16.25
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 6410.256 Hz
FIDRES 0.097813 Hz
AQ 5.1118579 sec
RG 322.5
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
============ CHANNEL f1 =============
NUC1 1H
P1 8.20 usec
PL1 -4.00 dB
SFO1 400.1325608 MHz
F2 - Processing parameters
SI 65536
SF 400.1300327 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.60
1D NMR plot parameters
CX 40.00 cm
F1P 9.500 ppm
F1 3801.24 Hz
F2P -0.500 ppm
F2 -200.07 Hz
PPMCM 0.25000 ppm/cm
HZCM 100.03251 Hz/cm
Koch/Peters FK076-1 P5-8 OPR:RF
400 MHz 1H-NMR
Ph
O
O
S
O
OH
CCl3
nPr
11c
ppm
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0


Current Data Parameters
NAME g1314
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20060906
Time 16.58
INSTRUM DRX400
PROBHD 5 mm BBO BB-
PULPROG zg45_cpd
TD 68804
SOLVENT CDCl3
NS 12000
DS 0
SWH 26246.719 Hz
FIDRES 0.381471 Hz
AQ 1.3107662 sec
RG 13004
DW 19.050 usec
DE 6.00 usec
TE 300.0 K
D1 1.00000000 sec
d11 0.03000000 sec
d12 0.00002000 sec
============ CHANNEL f1 =============
NUC1 13C
P1 7.60 usec
PL1 1.00 dB
SFO1 100.6241989 MHz
============ CHANNEL f2 =============
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 -4.00 dB
PL12 18.00 dB
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 65536
SF 100.6127680 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
1D NMR plot parameters
CX 40.00 cm
F1P 232.000 ppm
F1 23342.16 Hz
F2P -8.000 ppm
F2 -804.91 Hz
PPMCM 6.00000 ppm/cm
HZCM 603.67670 Hz/cm
Koch/Peters FK076-1 P5-8 OPR:RF
100MHz 13C-BB-NMR
ppm
136.14
128.95
101.30
79.16
77.02
70.07
62.43
35.69
25.94
21.79
13.97
-0.00
ppm
220
200
180
160
140
120
100
80
60
40
20
0

LOC ETHZ NMR Mercury-vx 300MHz Nr.6 11/14/06 21:16:26 USER:rkoch GROUP:peters SAMPLE:fk135-2-f5-10

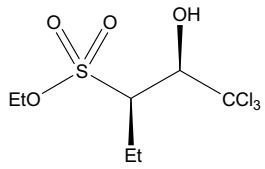
STANDARD 1H OBSERVE

Sample directory: fk135-2-f5-10

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk135-2-f5-10
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
16 repetitions
OBSERVE H1, 300.2230596 MHz
DATA PROCESSING
FT size 32768
Total time 0 min, 52 sec



12

LOC ETHZ NMR Mercury-vx 300MHz Nr.5 11/14/06 21:14:57 USER:rkoch GROUP:peters SAMPLE:

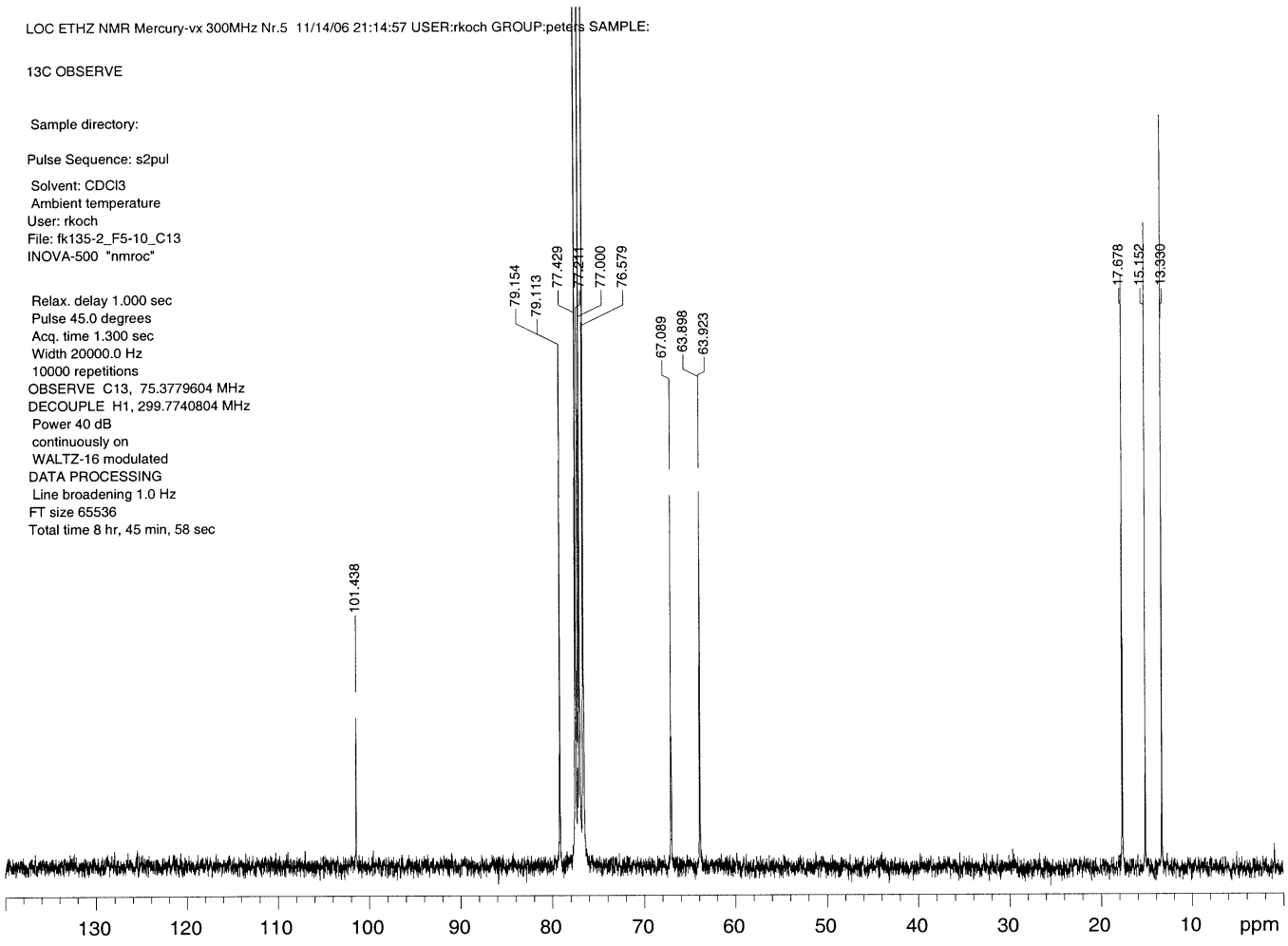
13C OBSERVE

Sample directory:

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk135-2_F5-10_C13
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
10000 repetitions
OBSERVE C13, 75.3779604 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 45 min, 58 sec



LOC ETHZ NMR Mercury 300MHz Nr.4 11/09/06 19:54:24 USER:rkoch GROUP:peters SAMPLE:fk052-4org

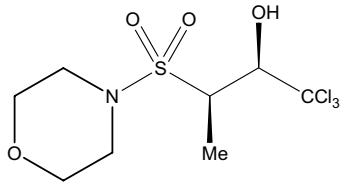
STANDARD 1H OBSERVE

File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
Mercury-300BB "gem4oc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
32 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec



13

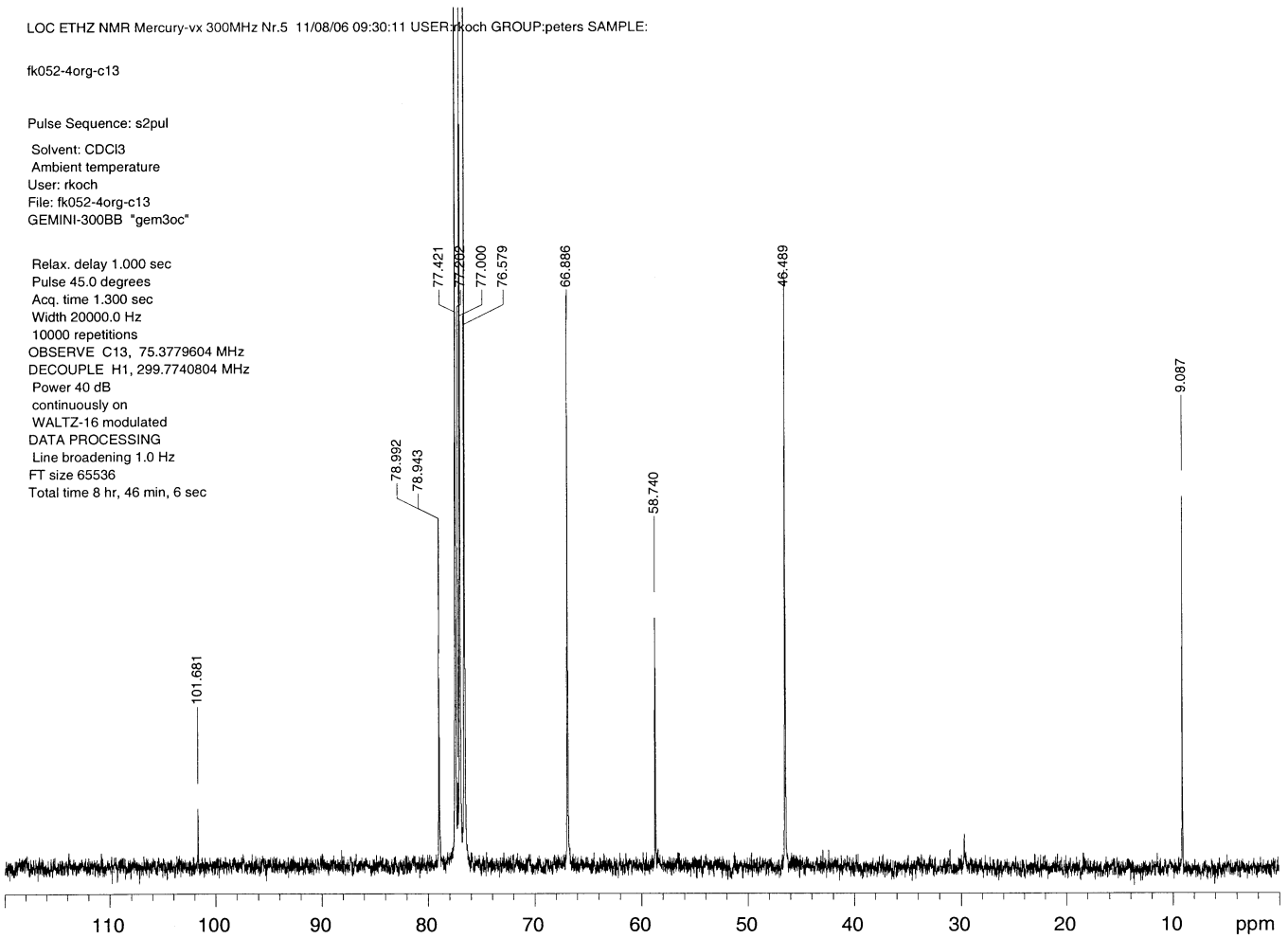
LOC ETHZ NMR Mercury-vx 300MHz Nr.5 11/08/06 09:30:11 USER:rkoch GROUP:peters SAMPLE:

fk052-4org-c13

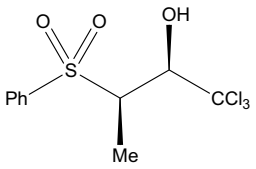
Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk052-4org-c13
GEMINI-300BB "gem3oc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
10000 repetitions
OBSERVE C13, 75.3779604 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 46 min, 6 sec



F.Koch/Peters FK136-1F11-21 OPR:PZ
400 MHz 1H-NMR

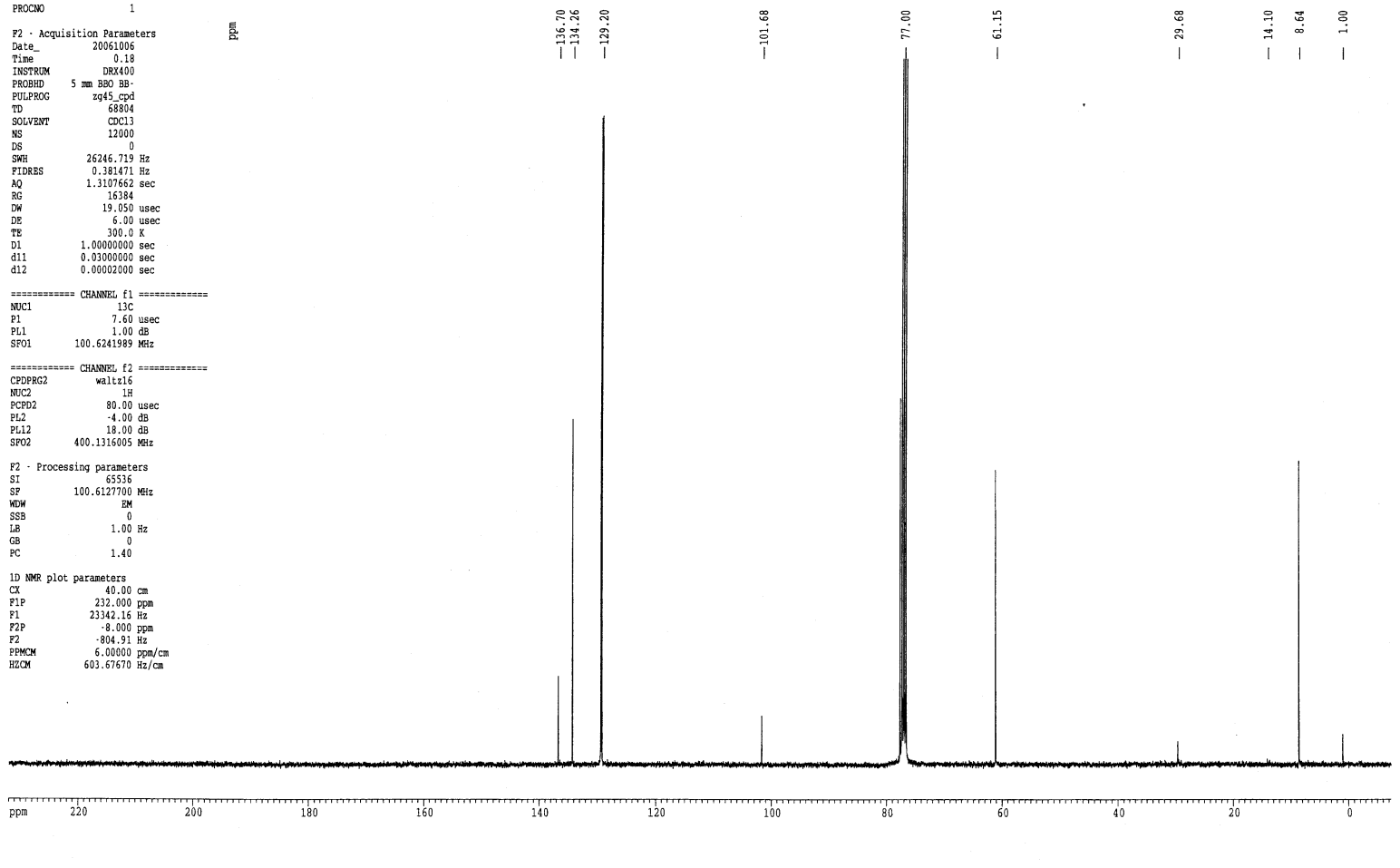


14a

```
Current Data Parameters
NAME                  g1331
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_              20061005
Time                 16.35
INSTRUM              DRX400
PROBHD     5 mm BBO BB-
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       64
DS                        0
SWH               6410.256 Hz
FIDRES            0.097813 Hz
AQ                5.1118579 sec
RG                      362
DW                   78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec

============ CHANNEL f1 =============
NUC1                    1H
P1                    8.20 usec
PL1                  -4.00 dB
SFO1           400.1325608 MHz

F2 - Processing parameters
SI                    65536
SF             400.1300089 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    1.40

1D NMR plot parameters
CX                   40.00 cm
F1P                   9.500 ppm
F1                 3801.24 Hz
F2P                  -0.500 ppm
F2                 -200.07 Hz
PPMCM                0.25000 ppm/cm
HZCM               100.03250 Hz/cm
```


F.Koch/Peters FK136-1F11-21 OPR:PZ
100MHz 13C-BB-NMR

```
Current Data Parameters
NAME                  g1331
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20061006
Time                  0.18
INSTRUM              DRX400
PROBHD     5 mm BBO BB-
PULPROG              zgpg45_cpd
TD                    68804
SOLVENT               CDCl3
NS                    12000
DS                        0
SWH              26246.719 Hz
FIDRES            0.381471 Hz
AQ                1.3107662 sec
RG                    16384
DW                   19.050 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
d11               0.03000000 sec
d12               0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                    7.60 usec
PL1                   1.00 dB
SFO1           100.6241989 MHz

============ CHANNEL f2 =============
CPDPRG2             waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 18.00 dB
SFO2           400.1316005 MHz

F2 - Processing parameters
SI                    65536
SF             100.6127700 MHz
WDW                     EM
SSB                       0
LB                    1.00 Hz
GB                        0
PC                    1.40

1D NMR plot parameters
CX                   40.00 cm
F1P                 232.000 ppm
F1                23342.16 Hz
F2P                 -8.000 ppm
F2                 -804.91 Hz
PPMCM                6.00000 ppm/cm
HZCM               603.67670 Hz/cm
```



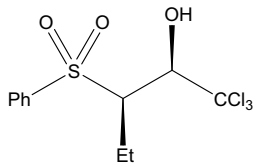
LOC ETHZ NMR Mercury 300MHz Nr.4 11/13/06 12:12:06 USER:rkoch GROUP:peters SAMPLE:fk125-1-K1

STANDARD 1H OBSERVE

Pulse Sequence: s2pul

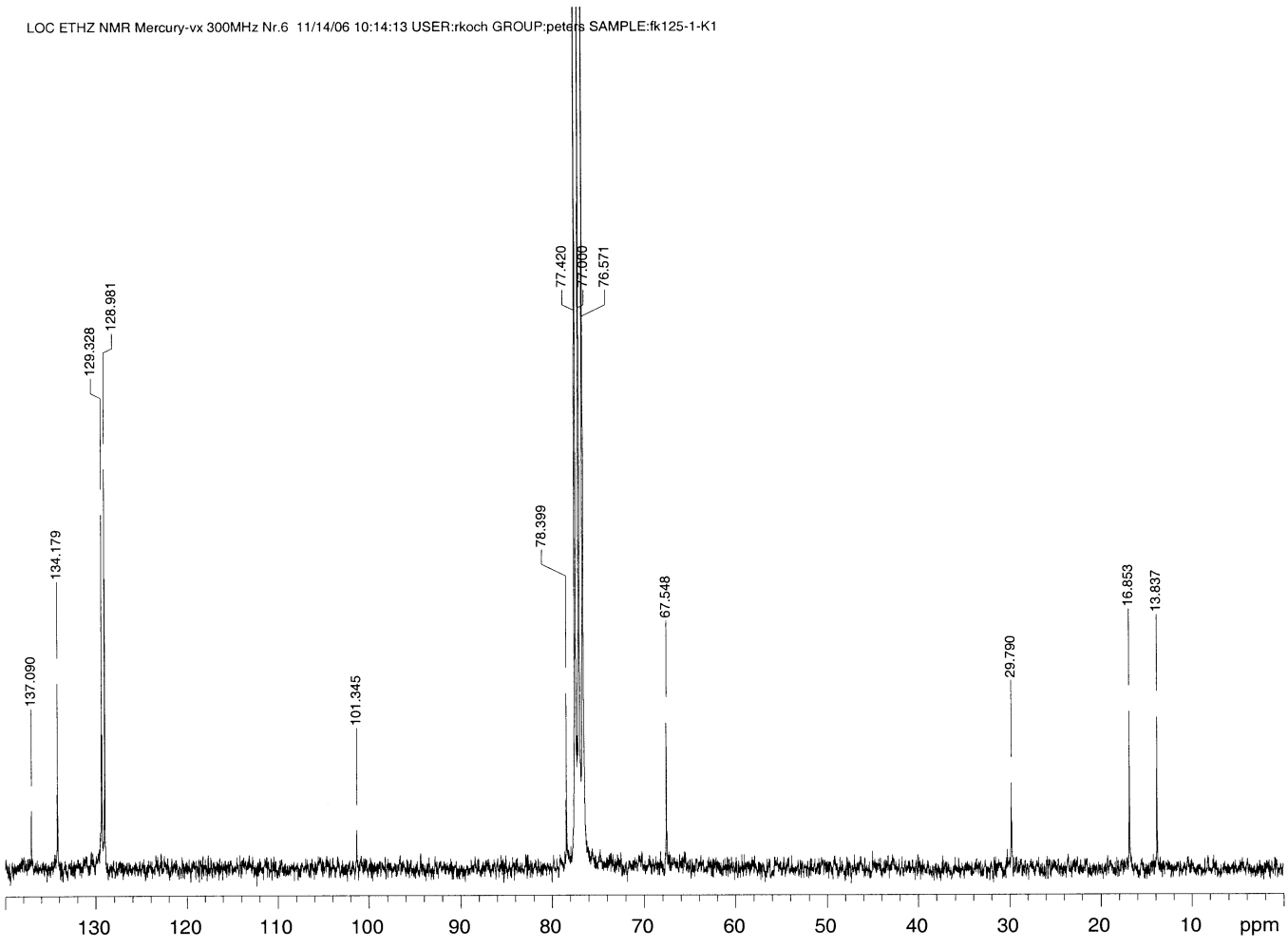
Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk125-1-k1-1H
GEMINI-300BB "gem3oc"

Pulse 18.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
66 repetitions
OBSERVE H1, 299.9012510 MHz
DATA PROCESSING
FT size 32768
Total time 1 hr, 28 min, 3 sec



14b

LOC ETHZ NMR Mercury-vx 300MHz Nr.6 11/14/06 10:14:13 USER:rkoch GROUP:peters SAMPLE:fk125-1-K1

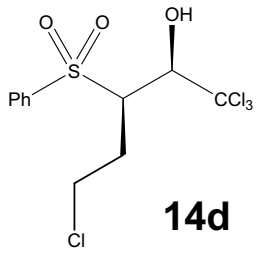


LOC ETHZ NMR Gemini 300 MHz Nr.3 11/12/06 19:15:39 USER:rkoch GROUP:peters SAMPLE:fk116-4-F5-8

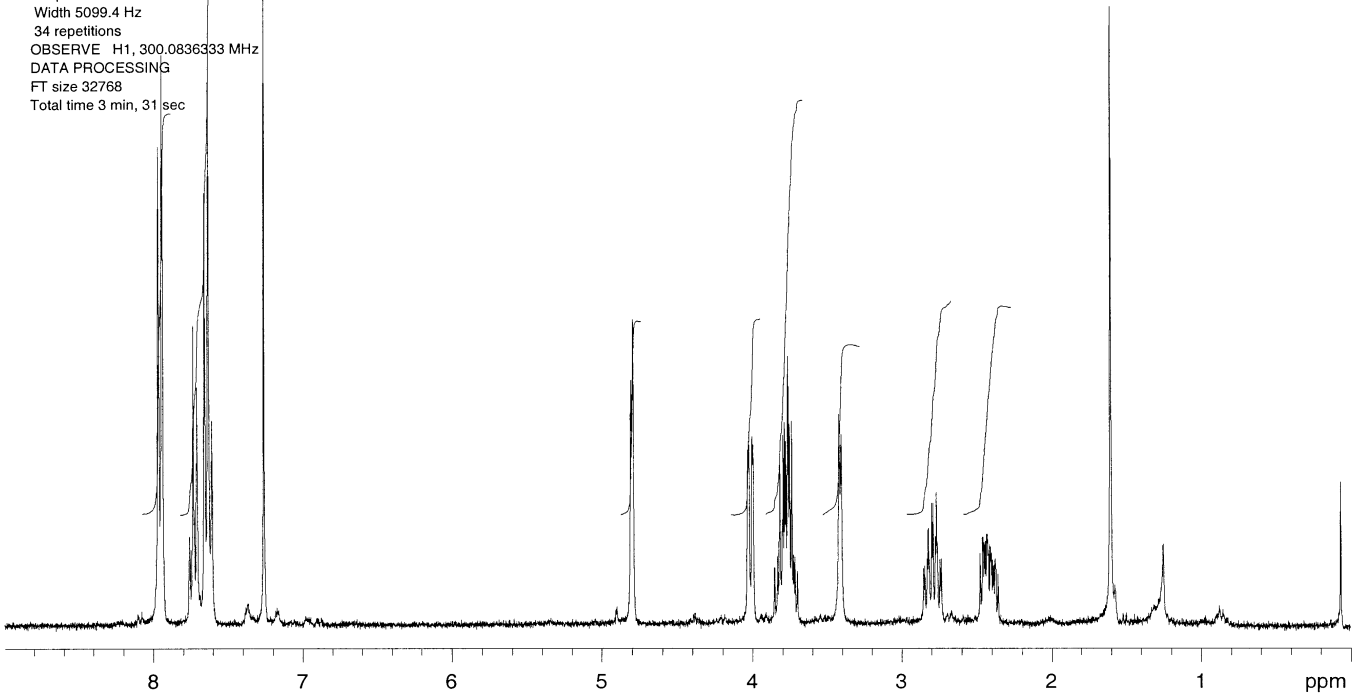
STANDARD 1H OBSERVE

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk116-4-F5-8
GEMINI-300BB "gem3oc"

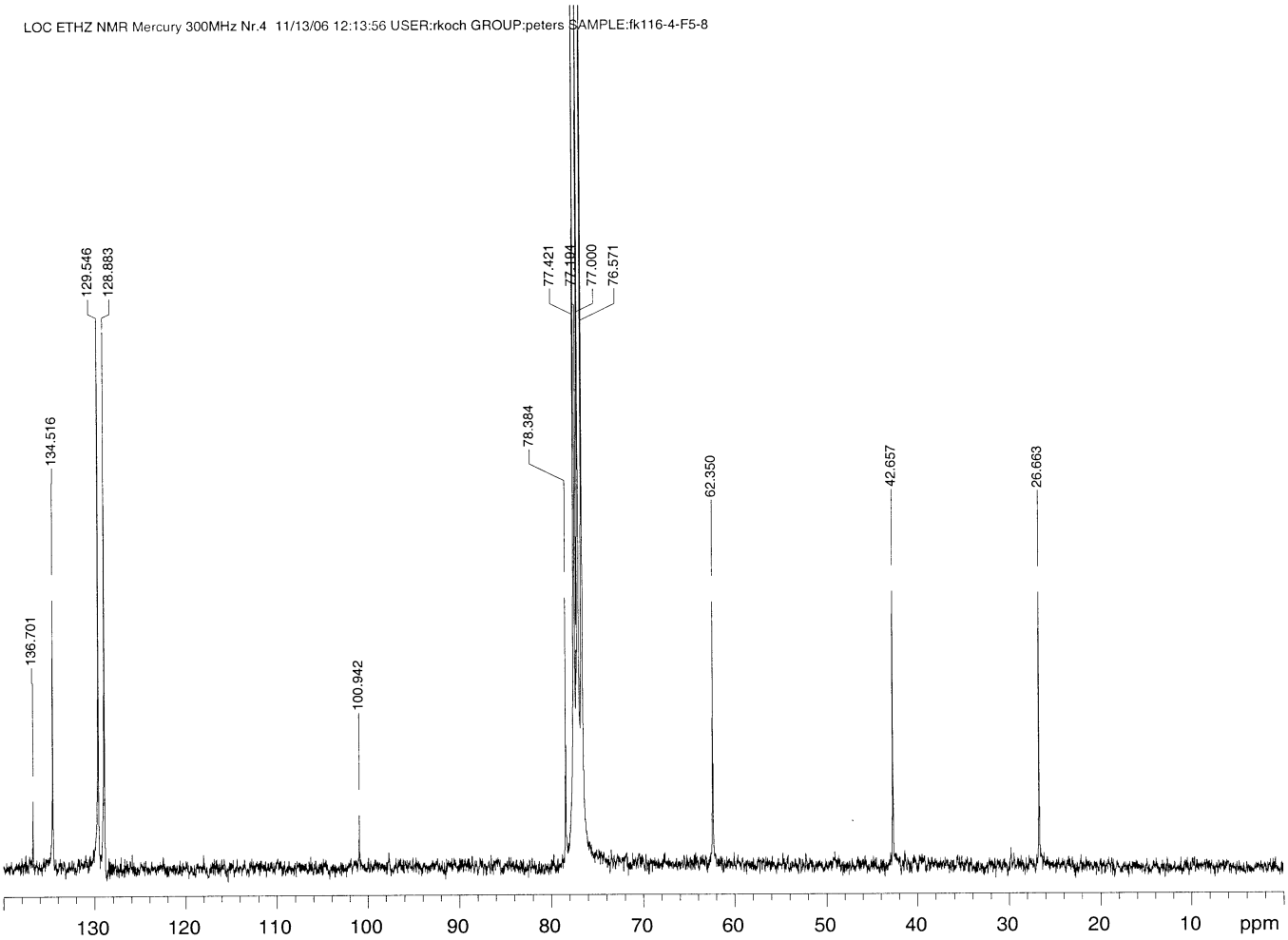
Pulse 29.5 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
34 repetitions
OBSERVE H1, 300.0836333 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec



14d



LOC ETHZ NMR Mercury 300MHz Nr.4 11/13/06 12:13:56 USER:rkoch GROUP:peters SAMPLE:fk116-4-F5-8



LOC ETHZ NMR Mercury-vx 300MHz Nr.5 11/14/06 21:13:46 USER:rkoch GROUP:peters SAMPLE:fk139m-FIV-VI

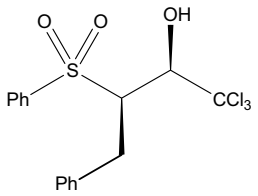
STANDARD 1H OBSERVE

Sample directory: fk139m-FIV-VI

Pulse Sequence: s2pul

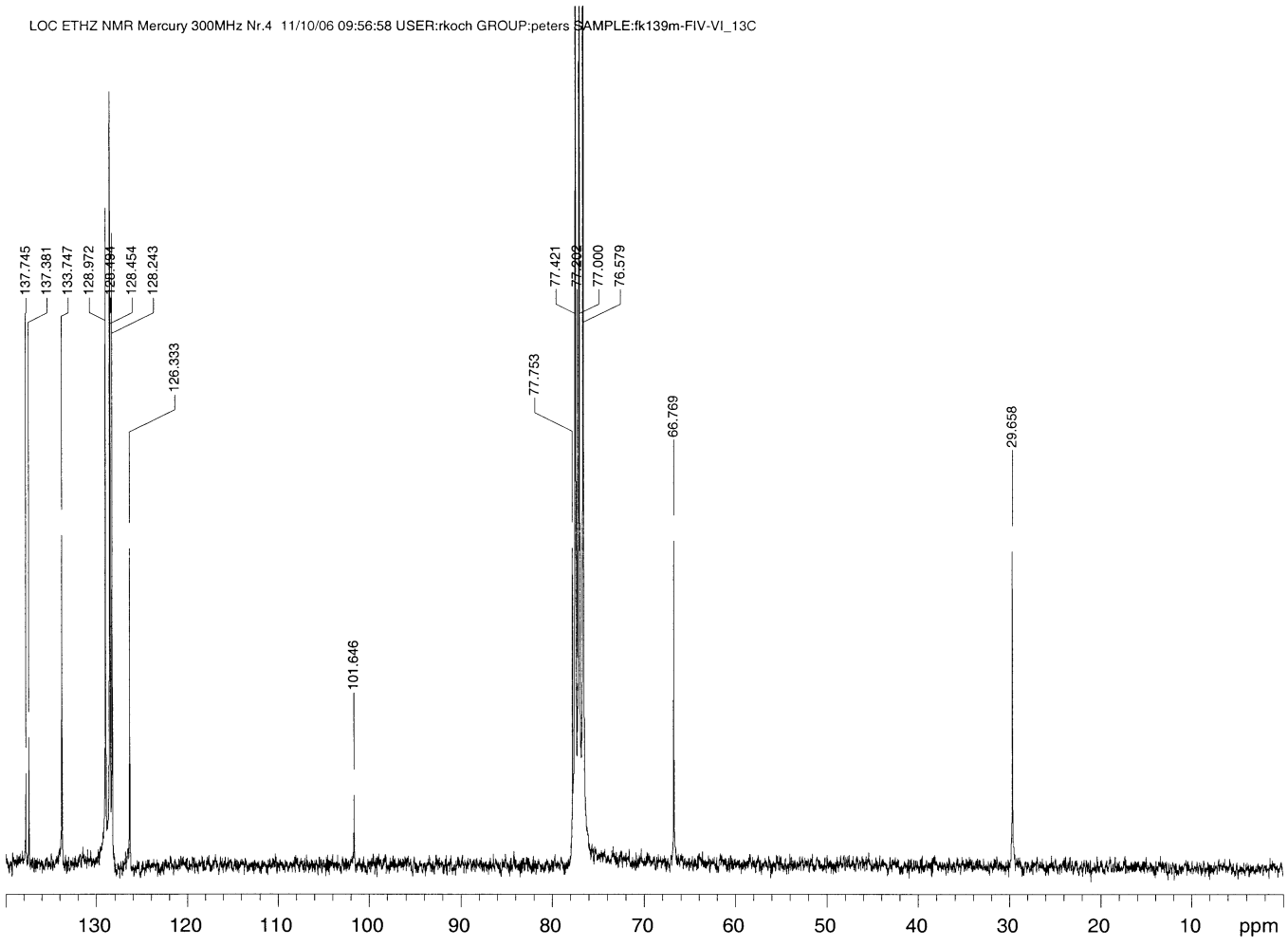
Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk139mix-FIV-VI
INOVA-500 "nmroc"

Relax. delay 1.000 sec
Pulse 56.2 degrees
Acq. time 1.998 sec
Width 4500.5 Hz
64 repetitions
OBSERVE H1, 299.7729181 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 19 sec



14e

LOC ETHZ NMR Mercury 300MHz Nr.4 11/10/06 09:56:58 USER:rkoch GROUP:peters SAMPLE:fk139m-FIV-VI_13C



LOC ETHZ NMR Mercury-vx 300MHz Nr.6 11/14/06 21:18:47 USER:rkoch GROUP:peters SAMPLE:f150-1-k1

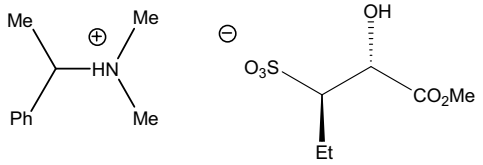
STANDARD 1H OBSERVE

Sample directory: f150-1-k1

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk150-1-k1
INOVA-500 "nmroc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 300.2230599 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec



16

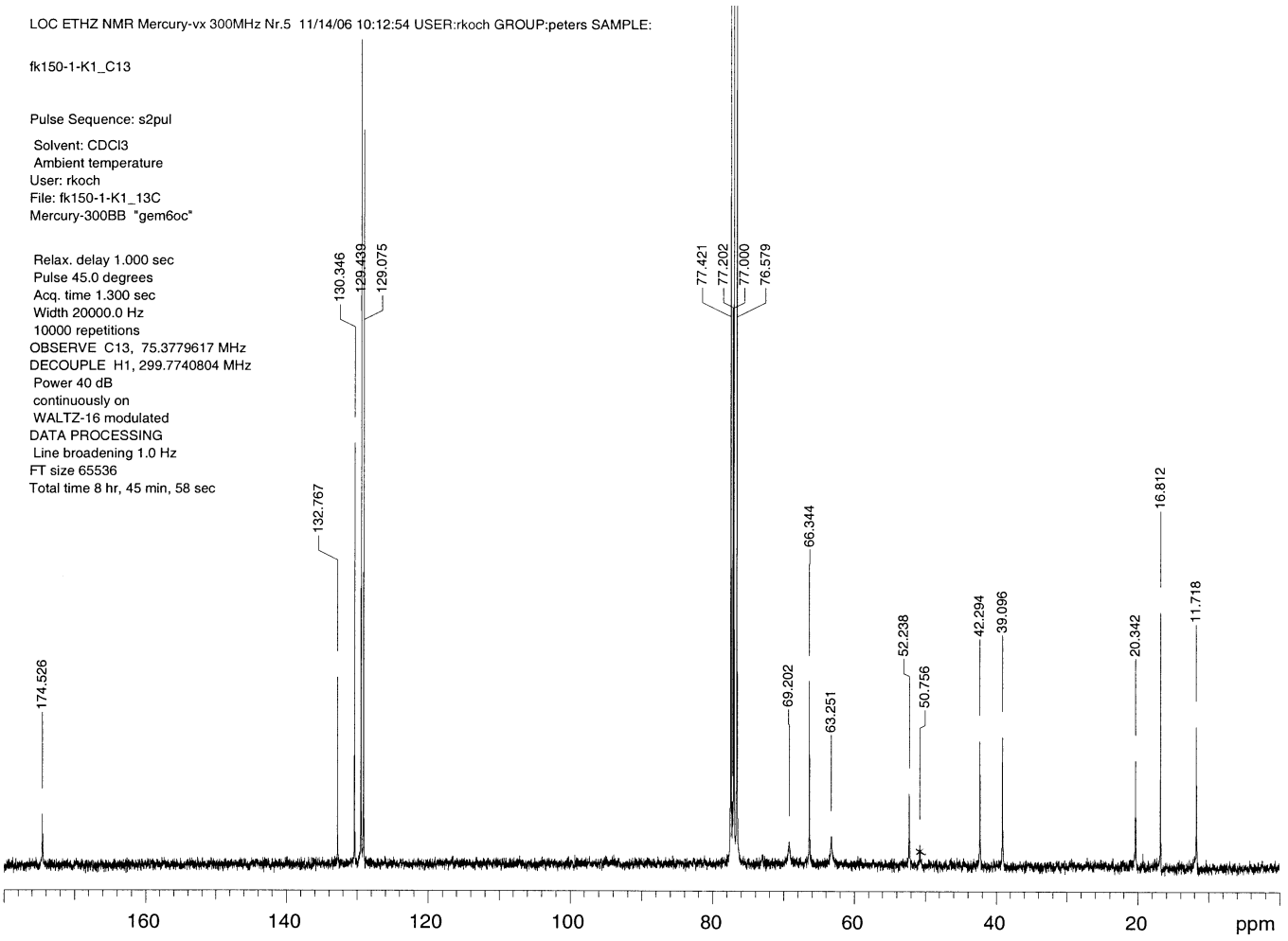
LOC ETHZ NMR Mercury-vx 300MHz Nr.5 11/14/06 10:12:54 USER:rkoch GROUP:peters SAMPLE:

fk150-1-K1_C13

Pulse Sequence: s2pul

Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk150-1-K1_13C
Mercury-300BB "gem6oc"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
Width 20000.0 Hz
10000 repetitions
OBSERVE C13, 75.3779617 MHz
DECOUPLE H1, 299.7740804 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 65536
Total time 8 hr, 45 min, 58 sec

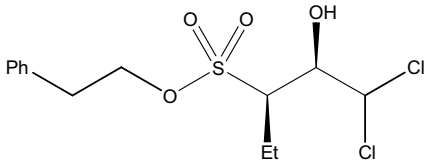


LOC ETHZ NMR Mercury-vx 300MHz Nr.6 11/12/06 19:17:15 USER:rkoch GROUP:peters SAMPLE:fk147m-F3-5

STANDARD 1H OBSERVE

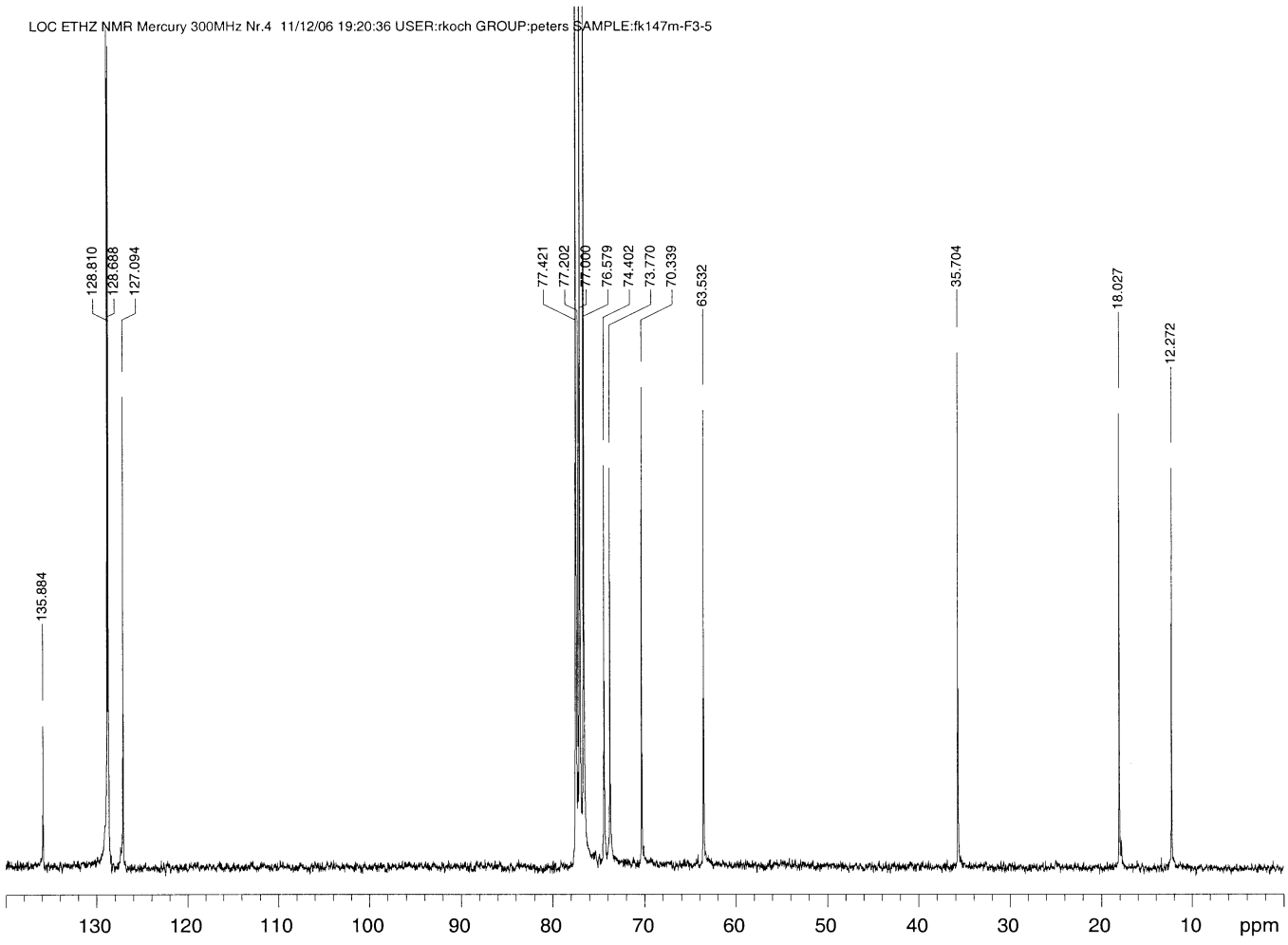
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk147m-F3-5
GEMINI-300BB "gem3oc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
64 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec



17

LOC ETHZ NMR Mercury 300MHz Nr.4 11/12/06 19:20:36 USER:rkoch GROUP:peters SAMPLE:fk147m-F3-5

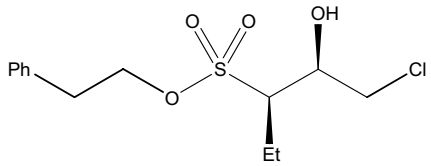


LOC ETHZ NMR Mercury-vx 300MHz Nr.6 11/12/06 19:18:18 USER:rkoch GROUP:peters SAMPLE:fk148m-F4-7

STANDARD 1H OBSERVE

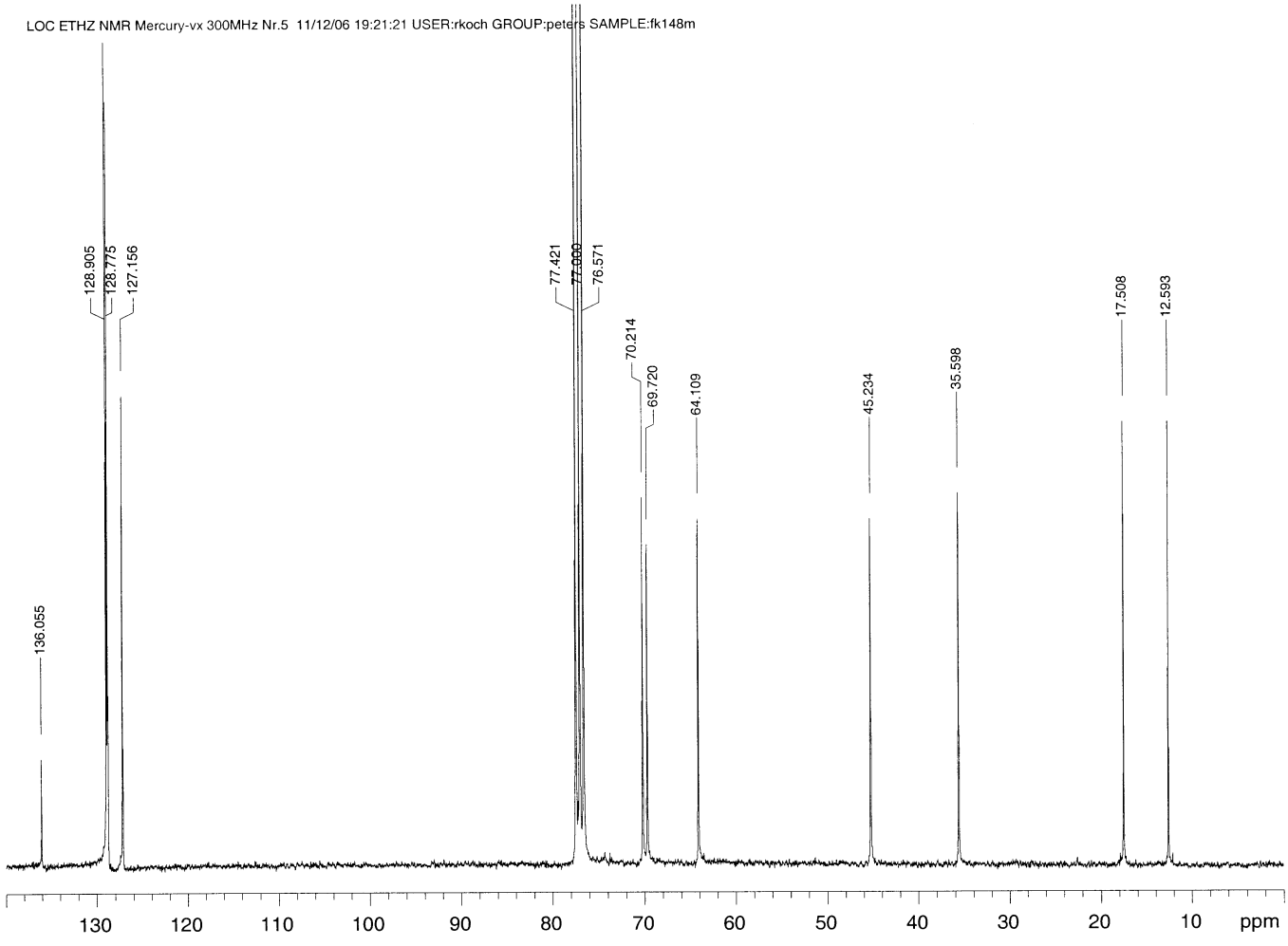
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
User: rkoch
File: fk148m-F4-8
GEMINI-300BB "gem3oc"

Pulse 30.0 degrees
Acq. time 3.138 sec
Width 5099.4 Hz
35 repetitions
OBSERVE H1, 300.2230602 MHz
DATA PROCESSING
FT size 32768
Total time 3 min, 31 sec



18

LOC ETHZ NMR Mercury-vx 300MHz Nr.5 11/12/06 19:21:21 USER:rkoch GROUP:peters SAMPLE:fk148m



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Current Data Parameters
NAME                  g1348
EXPNO                    1
PROCNO                   1

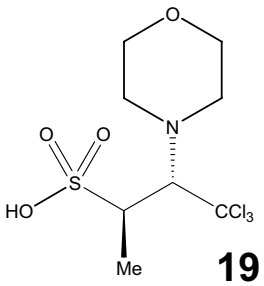
F2 - Acquisition Parameters
Date_              20061103
Time                  9.25
INSTRUM             DRX400
PROBHD     5 mm BBO BB-
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       1
DS                       0
SWH               6410.256 Hz
FIDRES           0.097813 Hz
AQ                5.1118579 sec
RG                   287.4
DW                  78.000 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec

============ CHANNEL f1 =============
NUC1                    1H
P1                    8.20 usec
PL1                  -4.00 dB
SFO1          400.1325608 MHz

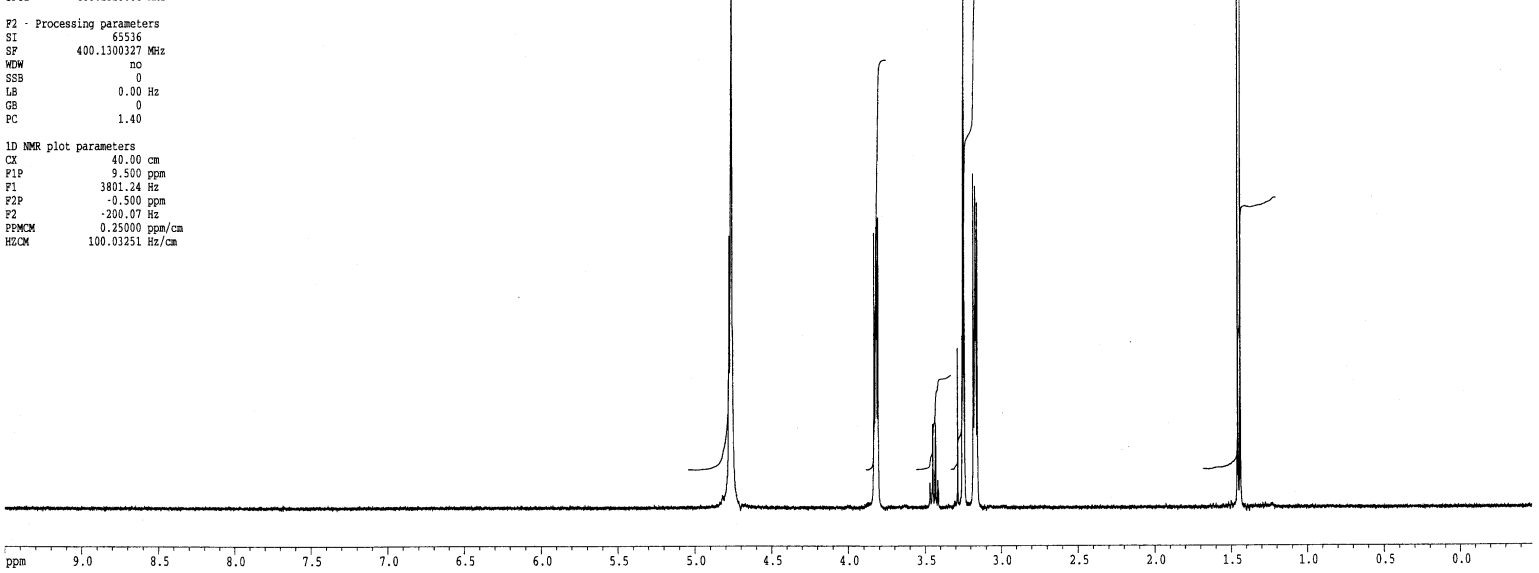
F2 - Processing parameters
SI                   65536
SF          400.1300327 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   40.00 cm
F1P                   9.500 ppm
F1                 3801.24 Hz
F2P                  -0.500 ppm
F2                 -200.07 Hz
PPMCM              0.25000 ppm/cm
HZCM             100.03251 Hz/cm
```

F.Koch/Peters FK052-1-K2 OPR:PZ
400 MHz 1H-NMR



19

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Current Data Parameters
NAME                  g1348
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_              20061103
Time                 10.40
INSTRUM             DRX400
PROBHD     5 mm BBO BB-
PULPROG            zg45_cpd
TD                   68804
SOLVENT              CDCl3
NS                    5821
DS                       0
SWH              26246.719 Hz
FIDRES           0.381471 Hz
AQ                1.3107662 sec
RG                    8192
DW                  19.050 usec
DE                   6.00 usec
TE                  300.0 K
D1             1.00000000 sec
d11            0.03000000 sec
d12            0.00002000 sec

============ CHANNEL f1 =============
NUC1                   13C
P1                    7.60 usec
PL1                   1.00 dB
SFO1          100.6241989 MHz

============ CHANNEL f2 =============
CPDPRG2            waltz16
NUC2                    1H
PCPD2                80.00 usec
PL2                  -4.00 dB
PL12                 18.00 dB
SFO2          400.1316005 MHz

F2 - Processing parameters
SI                   65536
SF          100.6126279 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   40.00 cm
F1P                 232.000 ppm
F1                 23342.13 Hz
F2P                  -8.000 ppm
F2                 -804.90 Hz
PPMCM              6.00000 ppm/cm
HZCM             603.67584 Hz/cm
```
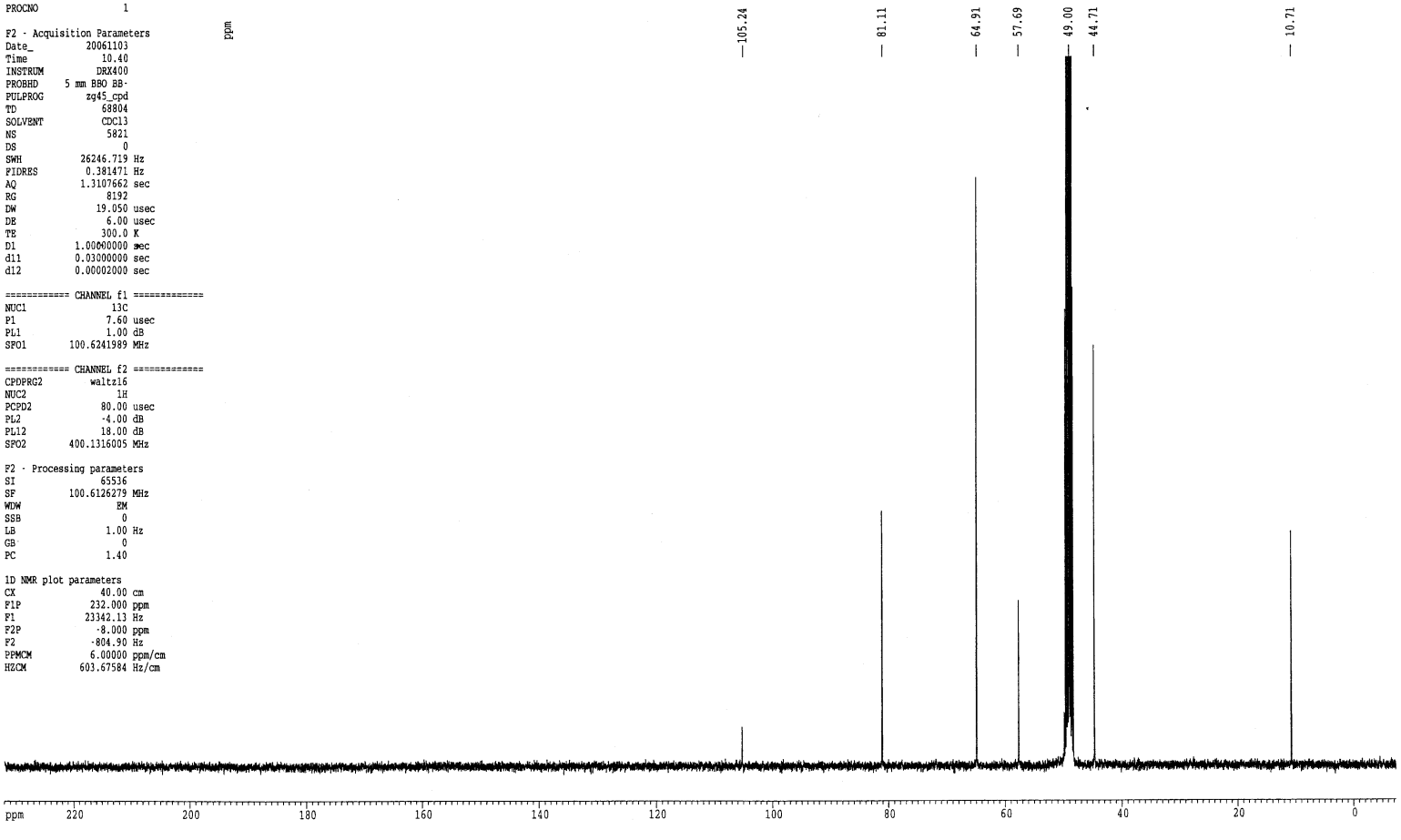
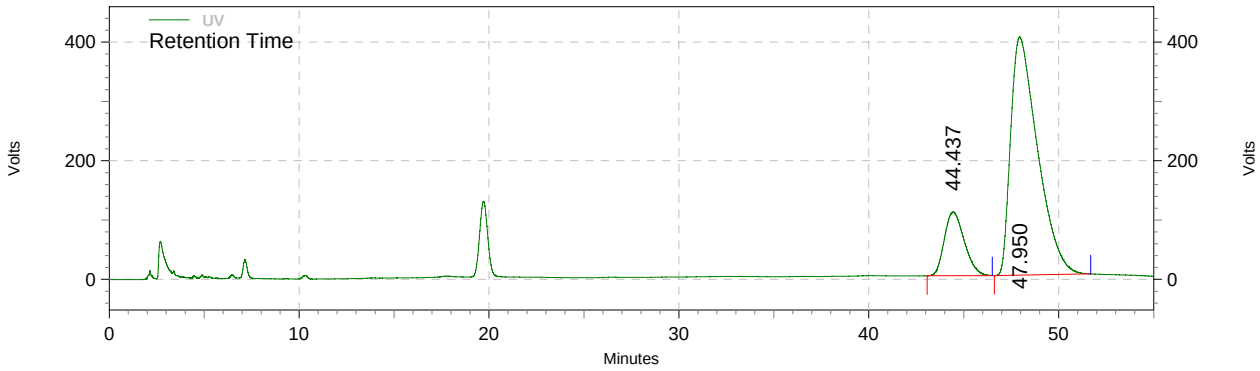

F.Koch/Peters FK052-1-K2 OPR:PZ
100MHz 13C-BB-NMR



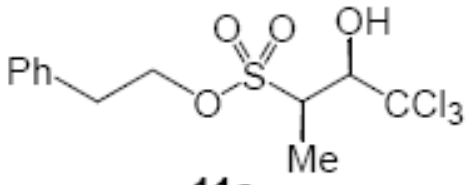
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK046-125rohkochSulfonatII.met3-23-2006 12-23-07 PM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 3/23/2006 12:24:54 PM
Printed: 11/12/2006 10:25:57 PM



UV Results

Retention Time	Area	Area %	Height	Height %
44.437	31286870	16.51	429552	21.10
47.950	158252911	83.49	1606157	78.90
Totals	189539781	100.00	2035709	100.00

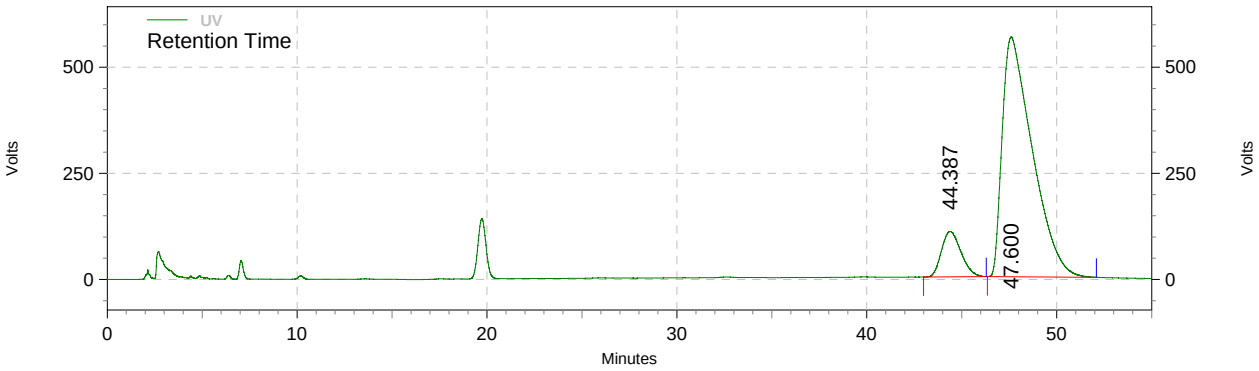


11a

M=Bi for formation of Sultone

Area % Report

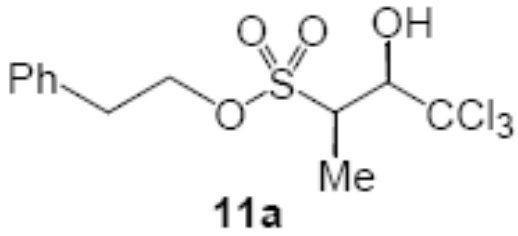
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK046-126rohkochSulfonatII.met3-23-2006 1-26-33 PM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 3/23/2006 1:27:22 PM
Printed: 11/12/2006 10:29:16 PM



UV Results

Retention Time	Area	Area %	Height	Height %
44.387	30525521	10.94	427610	15.91
47.600	248434683	89.06	2260136	84.09

Totals	278960204	100.00	2687746	100.00

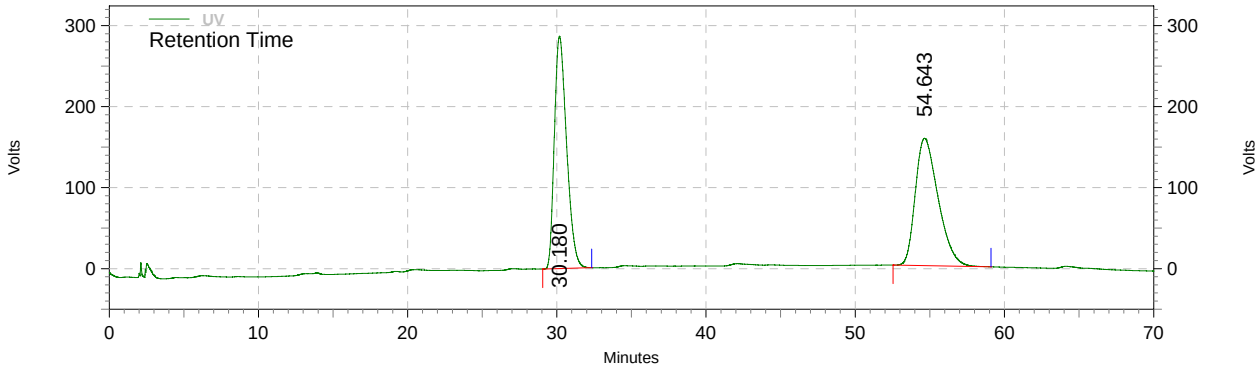


11a

M=In for formation of Sultone

Area % Report

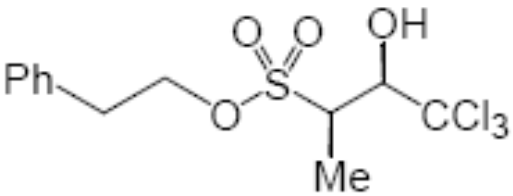
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK136-1-F11-21kochCl3-MeSulfon.met10-2-2006 6-05-47 PM171.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 10/2/2006 6:06:56 PM
Printed: 11/12/2006 10:31:49 PM



UV Results

Retention Time	Area	Area %	Height	Height %
30.180	65389496	49.42	1147389	64.61
54.643	66926212	50.58	628609	35.39

Totals	132315708	100.00	1775998	100.00

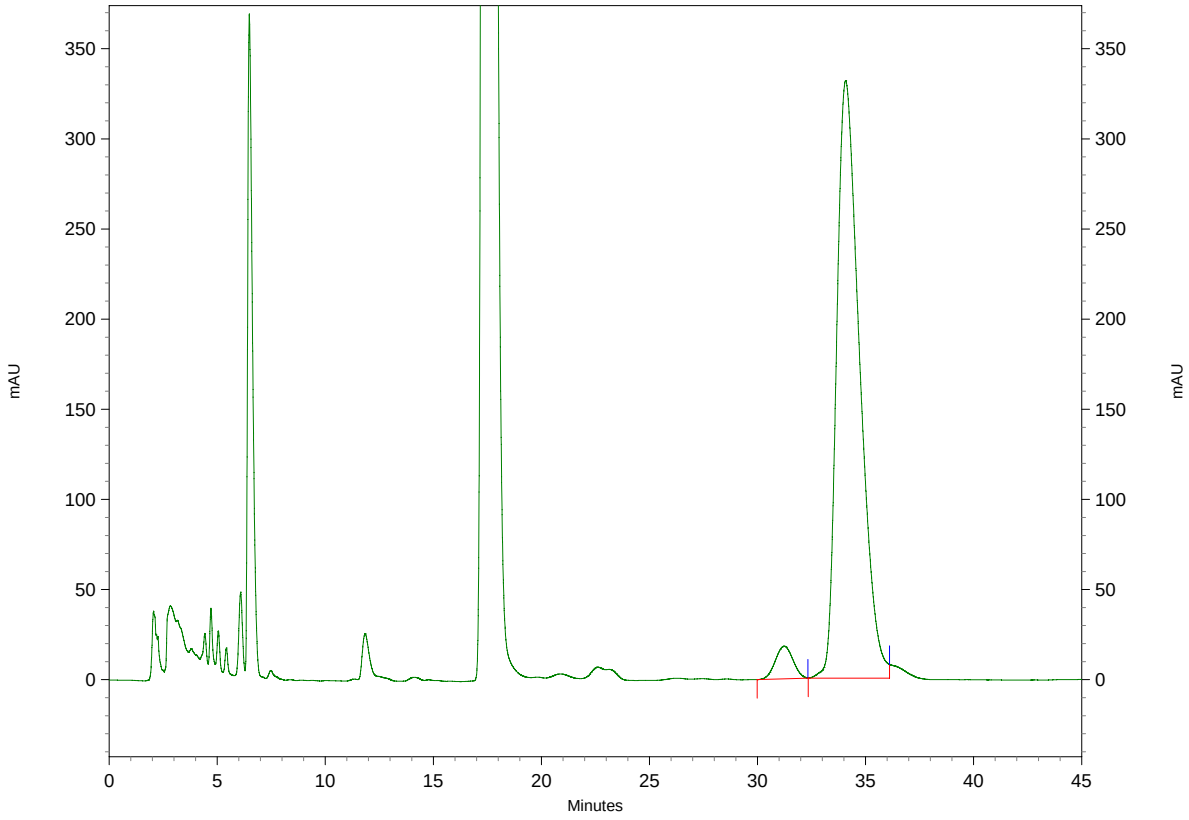


11a

racemate

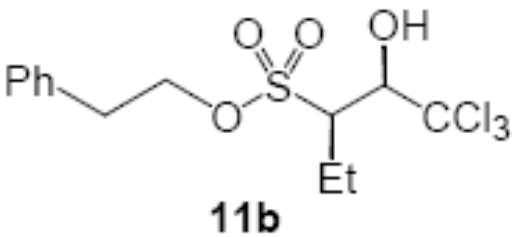
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK059-37rohkochCl3-EtSulfon.met1435-22-2006 10-02-10 PM
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 10:37:19 PM
Printed: 11/12/2006 11:03:12 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
31.237	4101890	4.022	72449	5.182
34.077	97880854	95.978	1325551	94.818
Totals	101982744	100.000	1398000	100.000

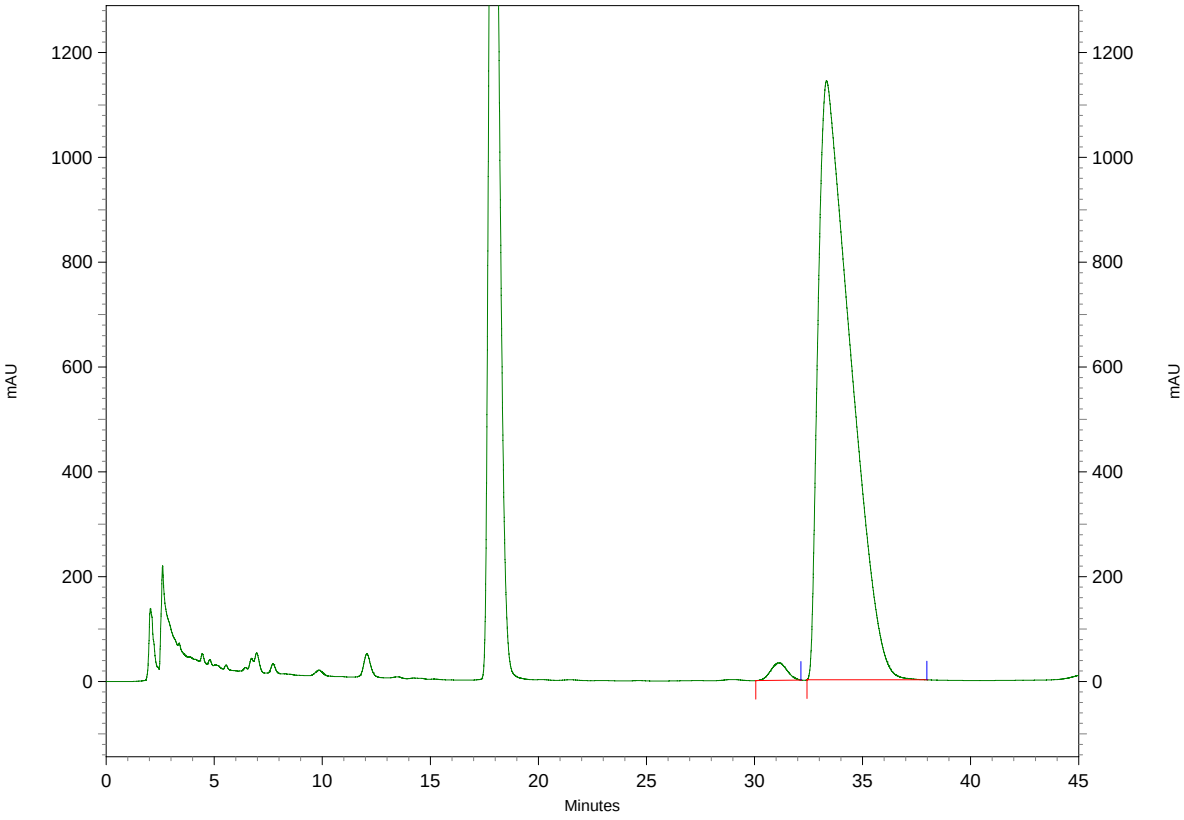


11b

M=Bi for formation of Sultone

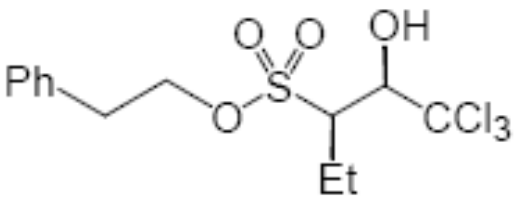
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK059-21rohkochCl3-EtSulfon.met5-9-2006 10-44-13 AM192.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 5/9/2006 12:45:14 PM
Printed: 11/12/2006 11:08:02 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
31.123	6979222	1.448	133092	2.828
33.337	474944178	98.552	4572831	97.172
Totals	481923400	100.000	4705923	100.000

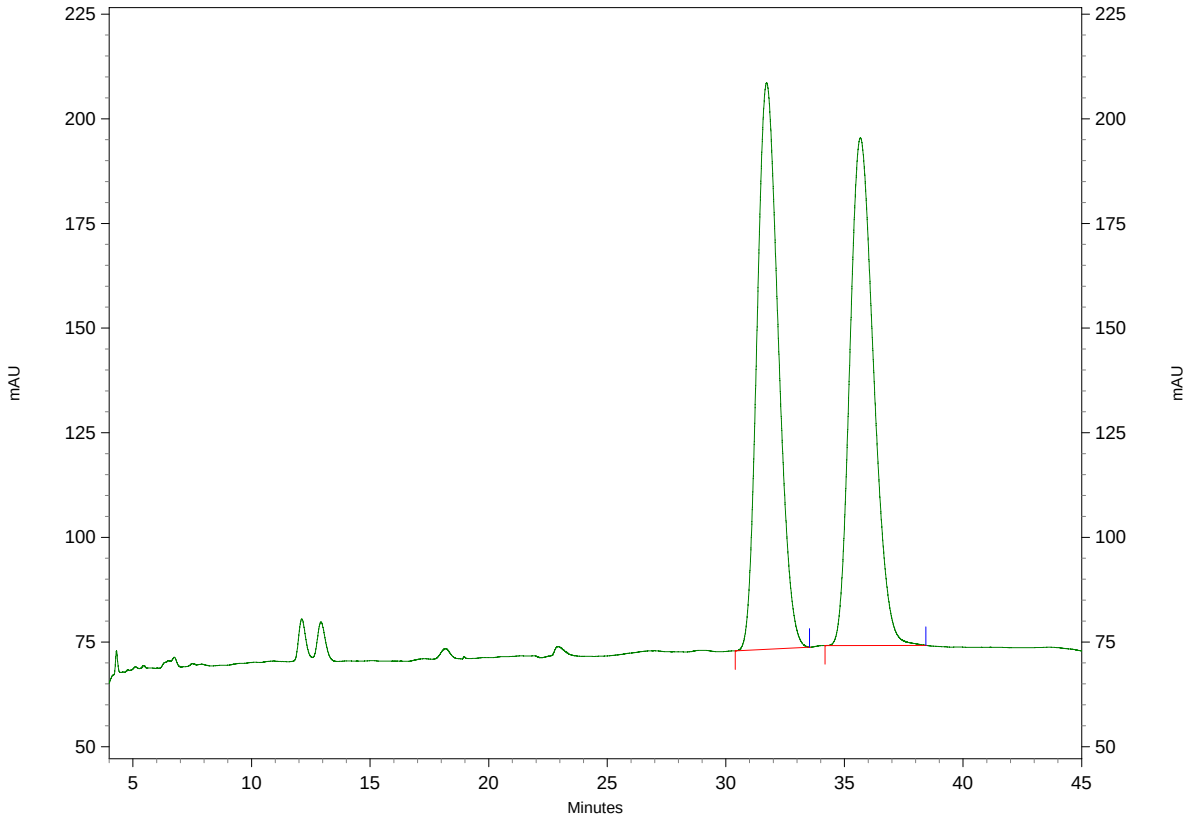


11b

M=In for formation of Sultone

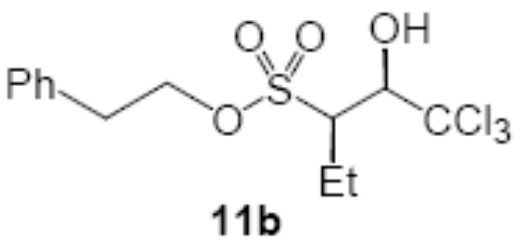
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK061-2kochCl3-EtSulfon.met4-27-2006 3-45-48 PM194.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 4/27/2006 5:49:54 PM
Printed: 11/12/2006 11:09:08 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
31.717	33730703	49.978	541485	52.740
35.673	33760255	50.022	485220	47.260
Totals	67490958	100.000	1026705	100.000

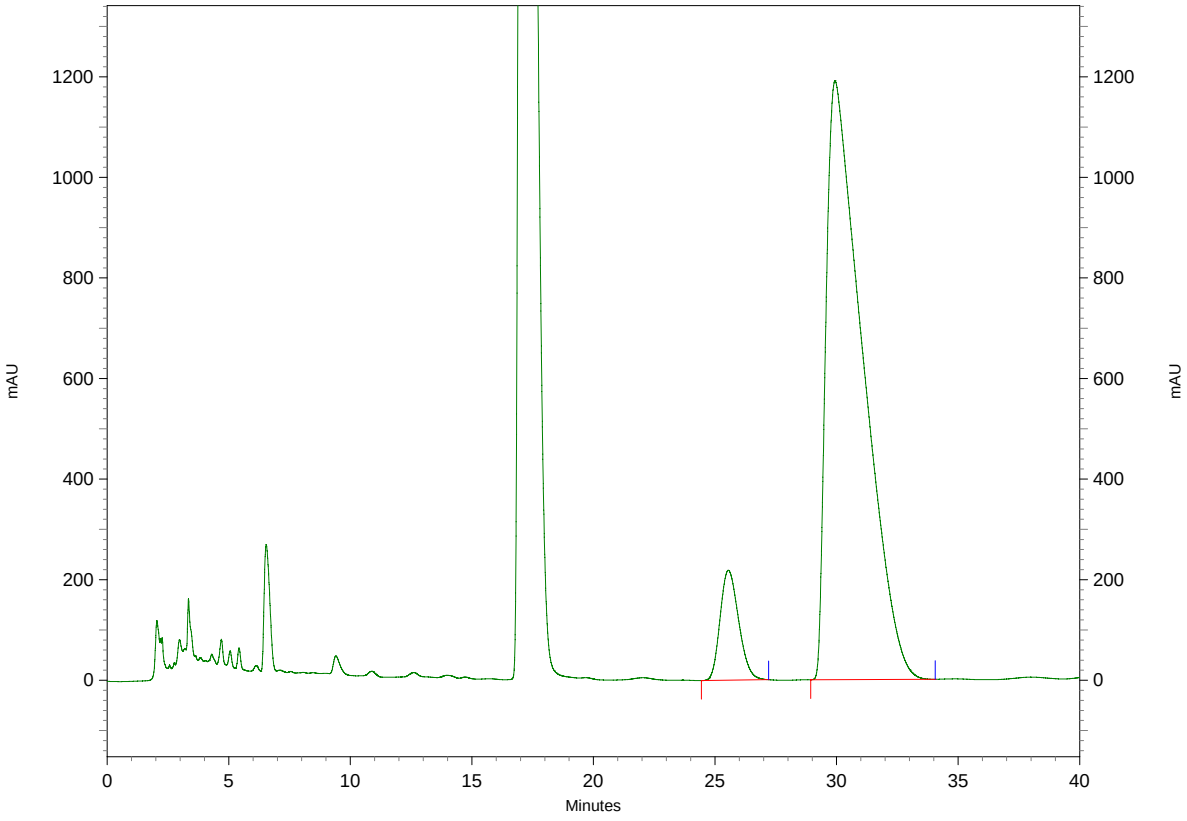


11b

racemate

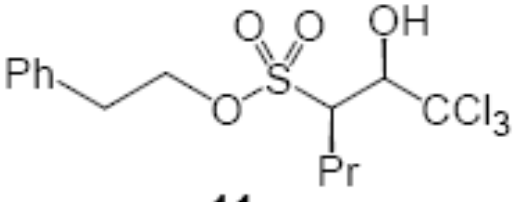
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK103-02rohkochSulfonatPr.met5-30-2006 8-09-17 PM182
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\CH2Cl_PhSulfonatEt.met
Acquired: 5/31/2006 9:05:21 AM
Printed: 11/13/2006 9:34:38 AM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
25.550	46416107	8.520	874766	15.512
29.937	498376147	91.480	4764520	84.488
Totals	544792254	100.000	5639286	100.000

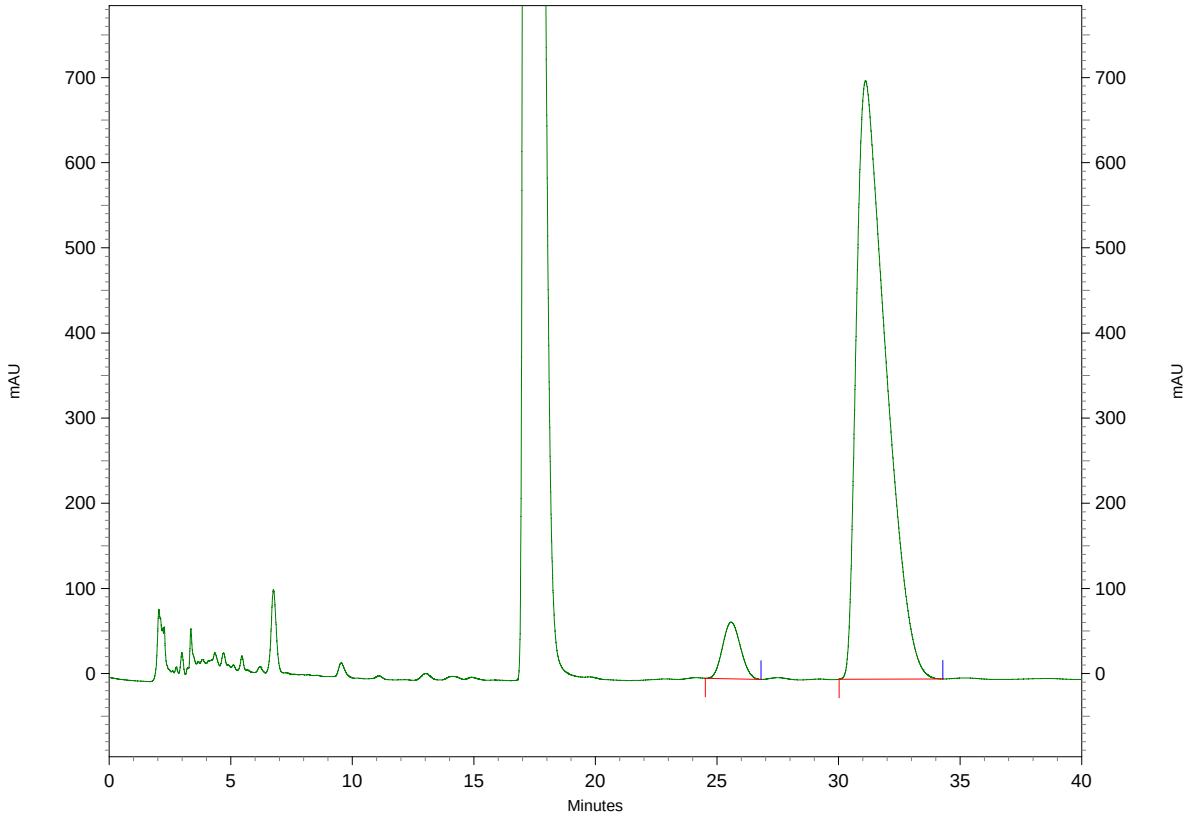


11c

M=Bi for formation of Sultone

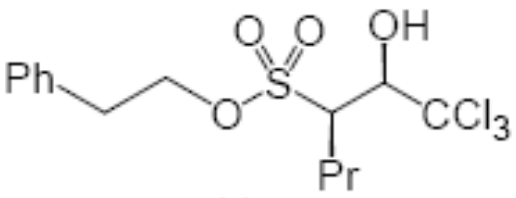
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK103-03rohkochSulfonatPr.met5-30-2006 8-50-35 PM183
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\CH2Cl_PhSulfonatEt.met
Acquired: 5/31/2006 9:05:56 AM
Printed: 11/13/2006 9:35:37 AM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
25.583	13644888	5.461	266175	8.649
31.110	236228152	94.539	2811307	91.351
Totals	249873040	100.000	3077482	100.000

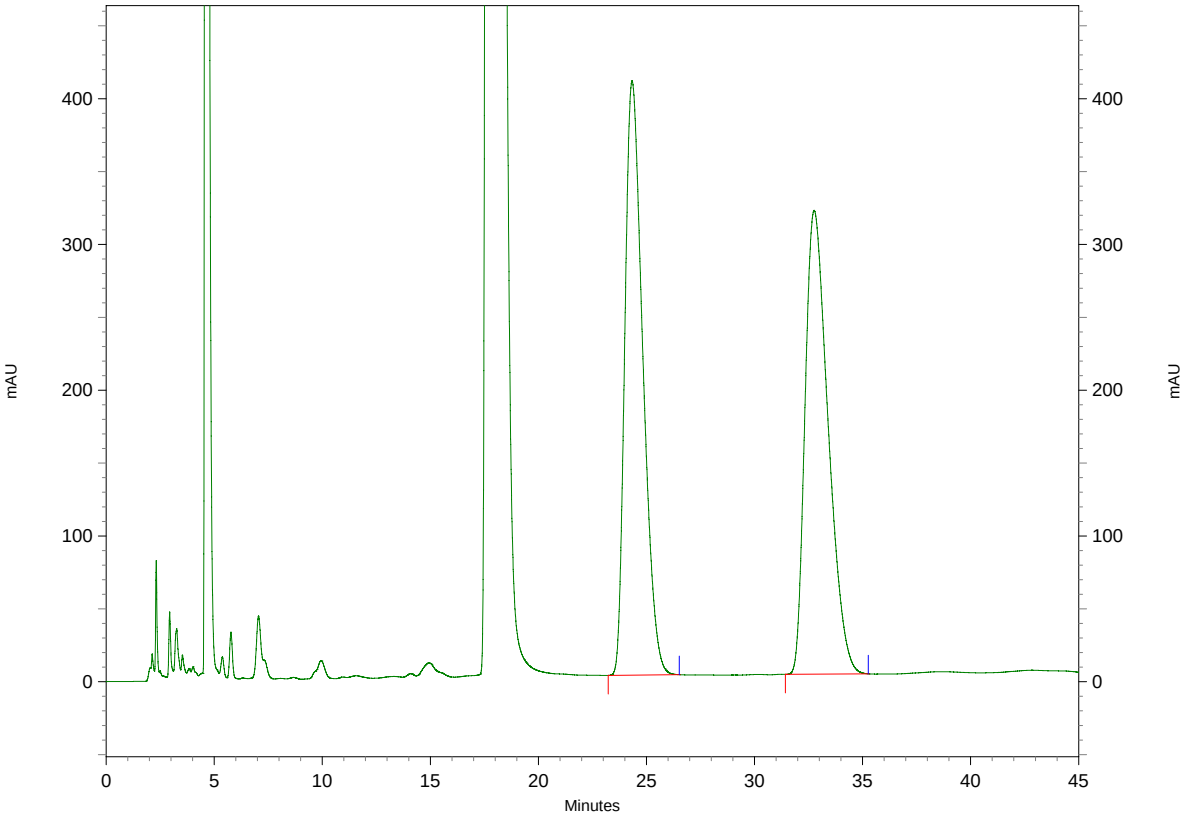


11c

M=In for formation of Sultone

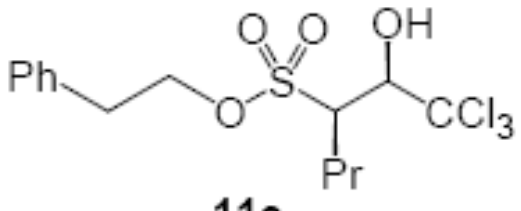
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK076-01rohkochSulfonatPr.met5-29-2006 4-02-54 PM181.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 5/29/2006 4:53:41 PM
Printed: 11/14/2006 6:38:32 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
24.333	94284307	49.994	1631279	56.168
32.763	94307131	50.006	1273031	43.832
Totals	188591438	100.000	2904310	100.000

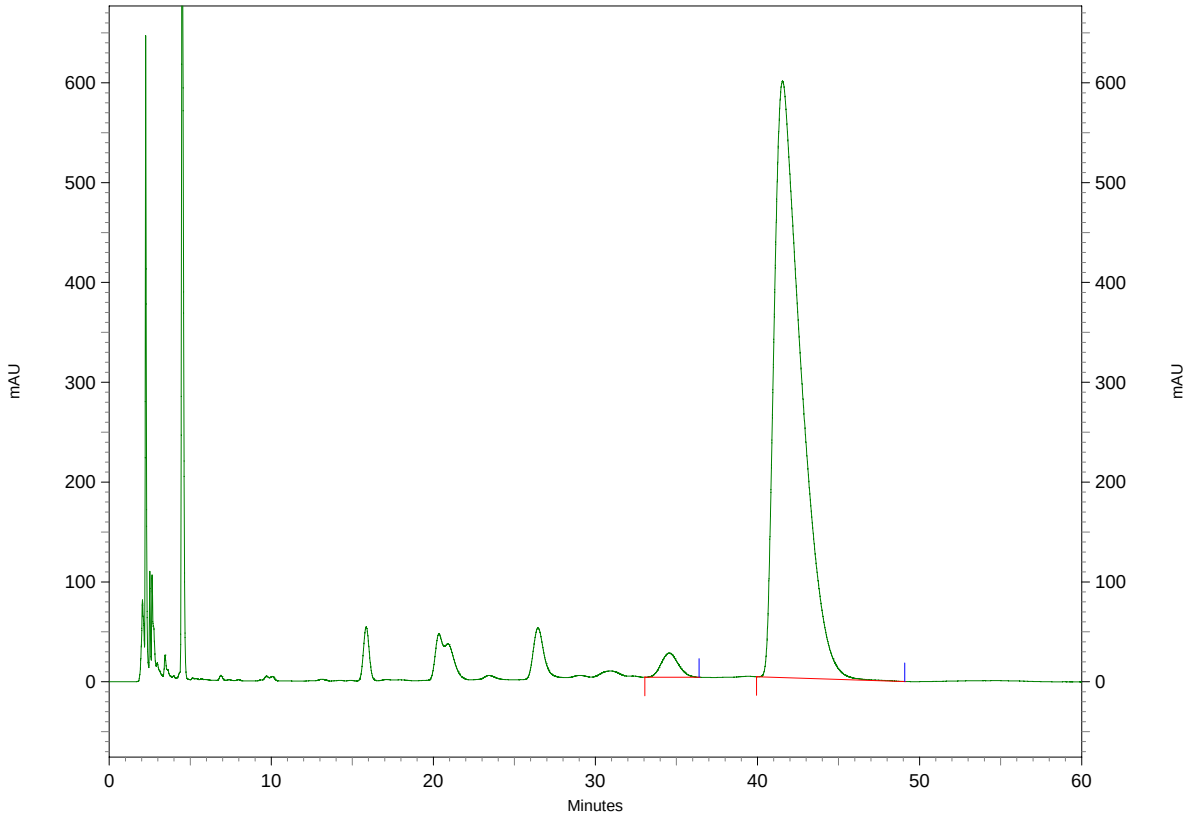


11c

racemate

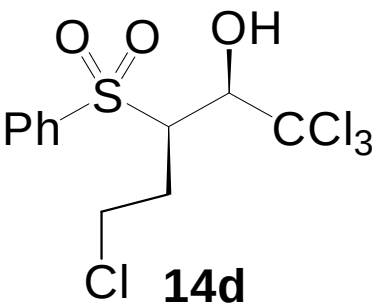
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK116-01rohkochCl3-ClEtSulfon.met 6-14-2006 1-03-59 PM178.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 10:57:32 PM
Printed: 11/12/2006 11:01:20 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
34.547	7114474	2.523	96703	3.888
41.543	274824950	97.477	2390415	96.112
Totals	281939424	100.000	2487118	100.000

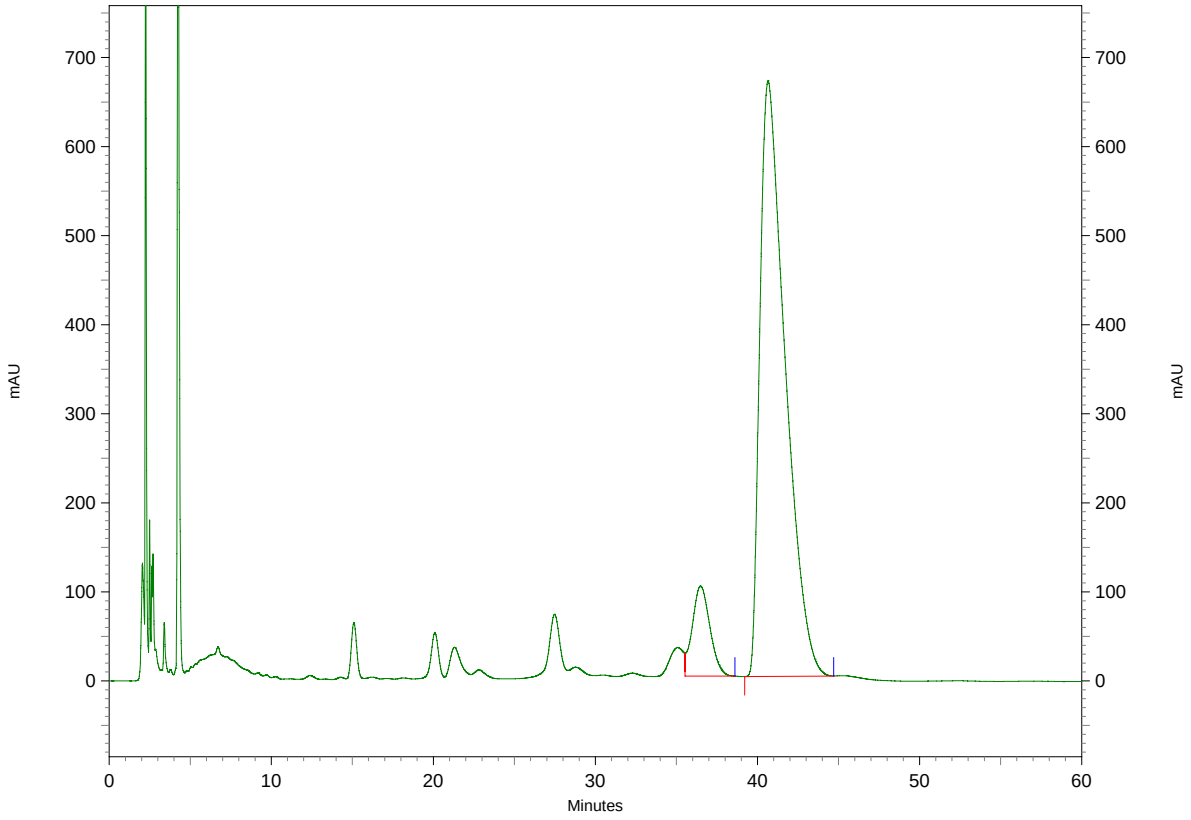


14d

M=Bi for formation of Sultone

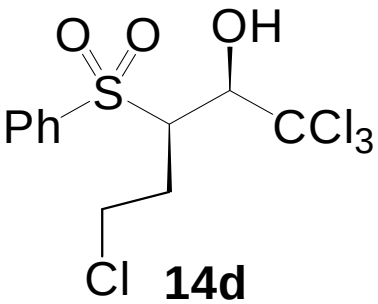
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK116-02rohkochCl3-ClEtSulfon.met 6-14-2006 2-05-19 PM179.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 11:11:31 PM
Printed: 11/12/2006 11:11:37 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
36.487	30718722	9.565	404962	13.144
40.653	290436381	90.435	2676034	86.856
Totals	321155103	100.000	3080996	100.000

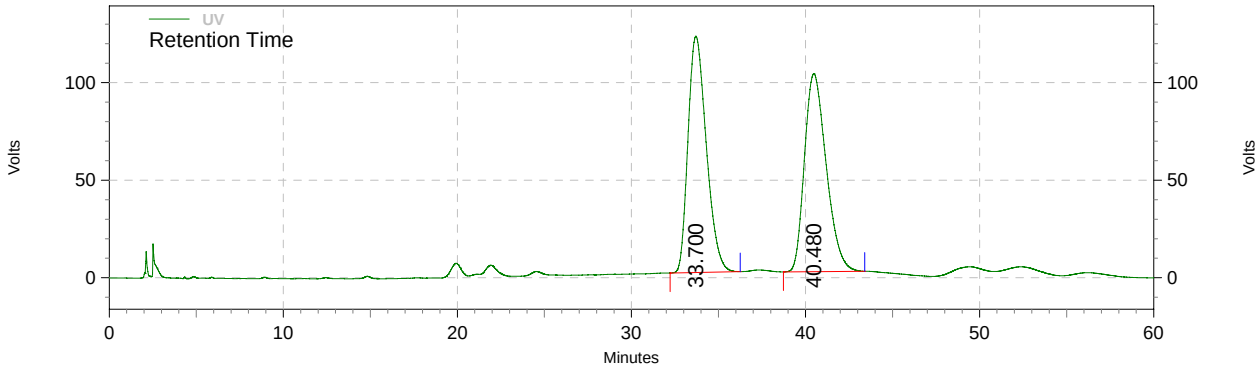


14d

M=In for formation of Sultone

Area % Report

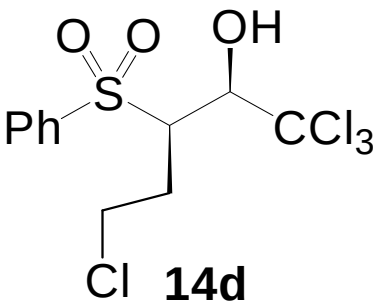
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK113-2-F6-9kochCl3-ClEtSulfon.met10-4-2006 11-37-27 AM171.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 10/4/2006 11:38:38 AM
Printed: 11/12/2006 11:12:28 PM



UV Results

Retention Time	Area	Area %	Height	Height %
33.700	35386963	50.07	484437	54.42
40.480	35290658	49.93	405740	45.58

Totals	70677621	100.00	890177	100.00

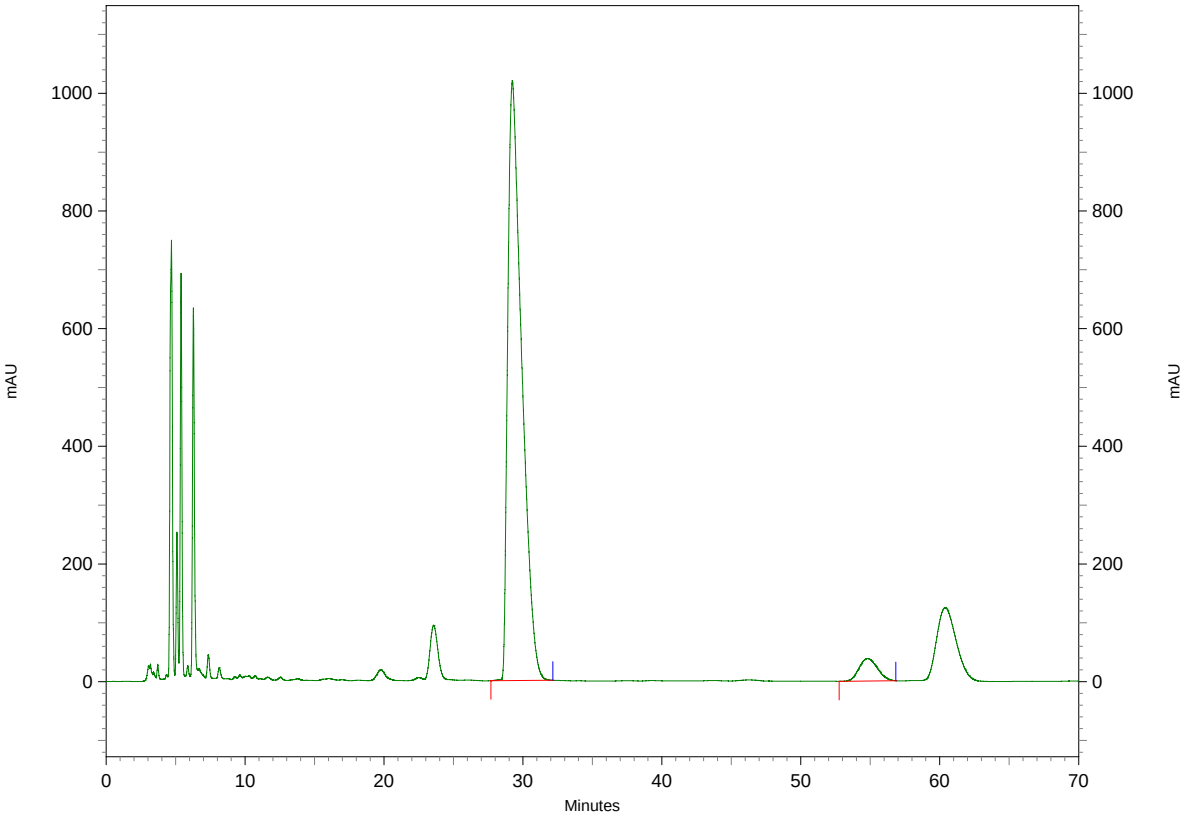


14d

racemate

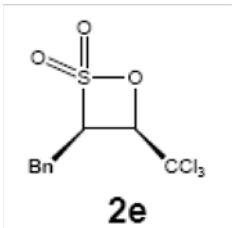
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK102-1rohkochSulton_Bn.met6-6-2006 6-58-55 PM173.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 11:18:43 PM
Printed: 11/12/2006 11:19:15 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
29.233	285472087	95.307	4078305	96.384
54.803	14056974	4.693	152993	3.616
Totals	299529061	100.000	4231298	100.000

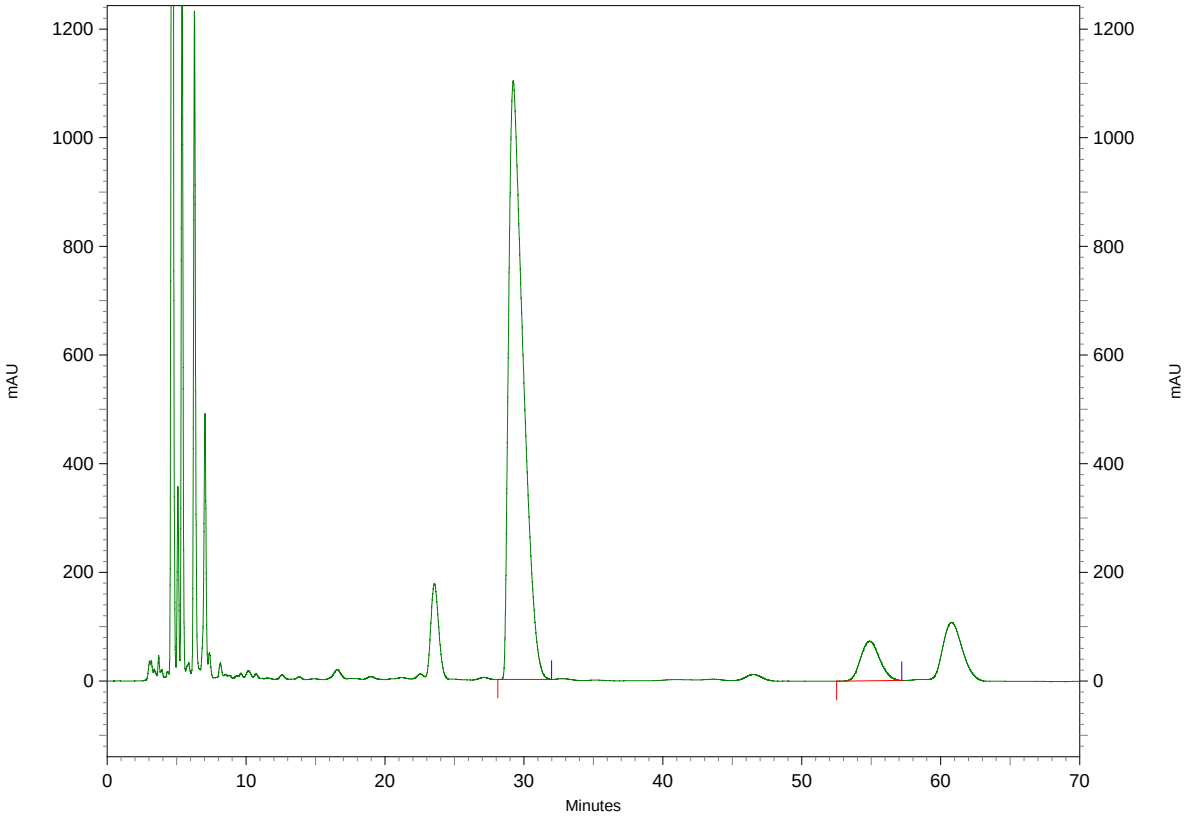


2e

M=Bi for formation of β-Sultone

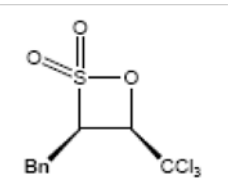
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK102-2rohkochSulton_Bn.met6-6-2006 8-10-13 PM174.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 11:20:37 PM
Printed: 11/12/2006 11:20:50 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
29.227	319520081	92.004	4409057	93.797
54.870	27767889	7.996	291591	6.203
Totals	347287970	100.000	4700648	100.000

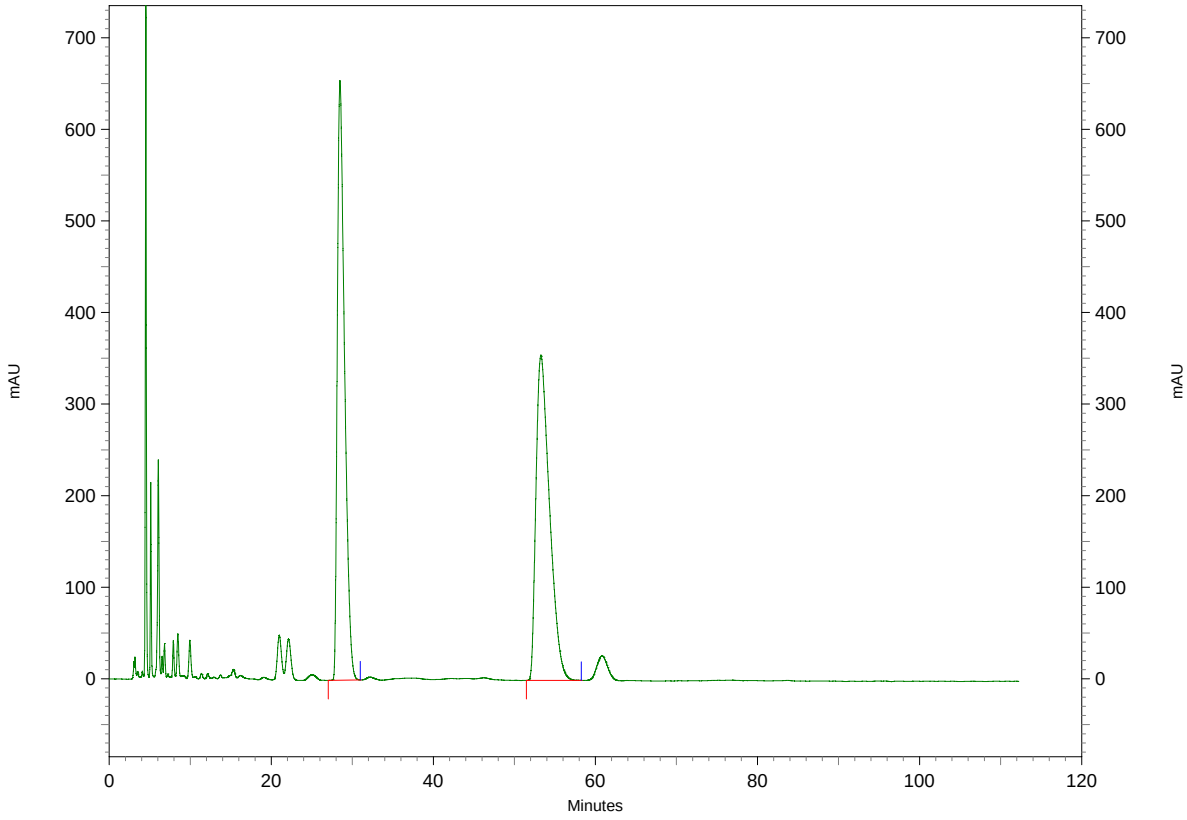


2e

M=In for formation of β-Sultone

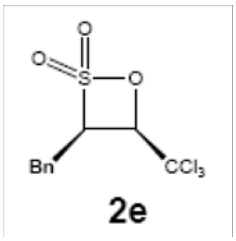
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK099-1rohkochSulton_Bn.met6-6-2006 5-05-22 PM172.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 11:16:37 PM
Printed: 11/12/2006 11:17:02 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
28.473	163546579	49.791	2618492	64.826
53.273	164919798	50.209	1420765	35.174
Totals	328466377	100.000	4039257	100.000

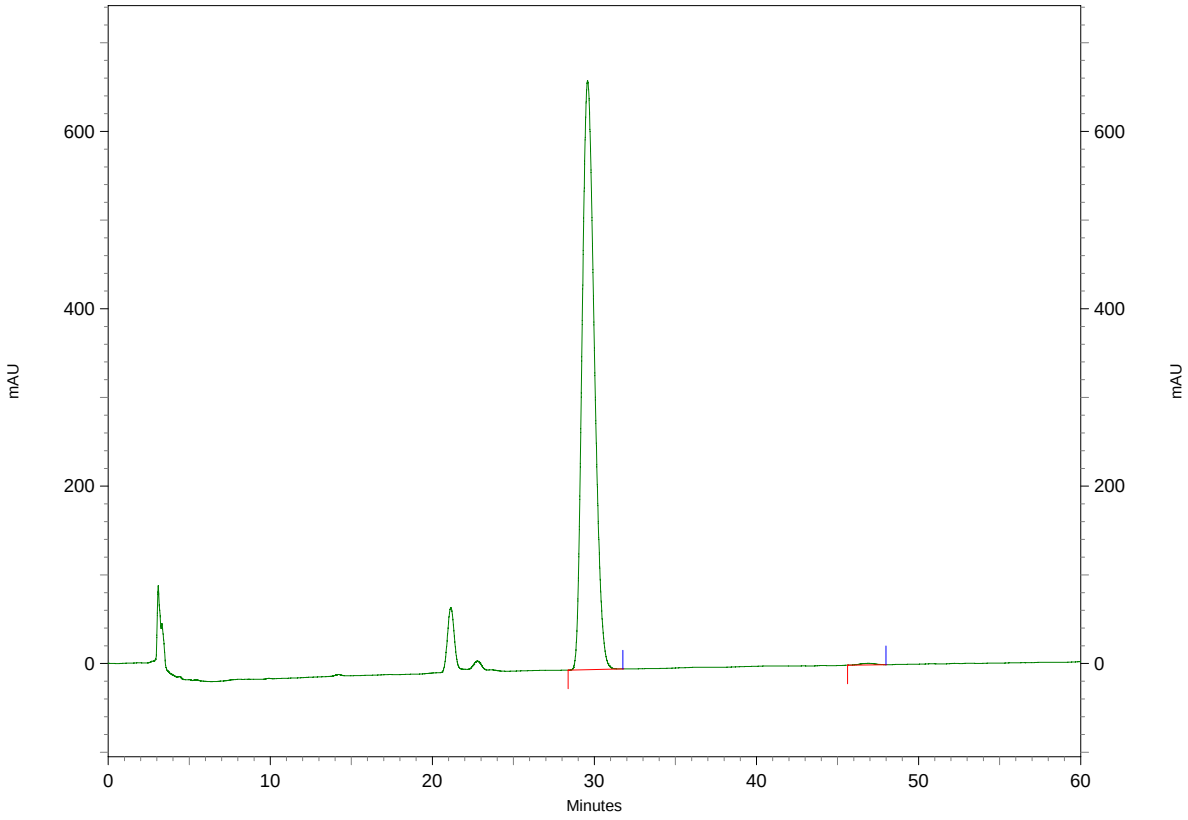


2e

racemate

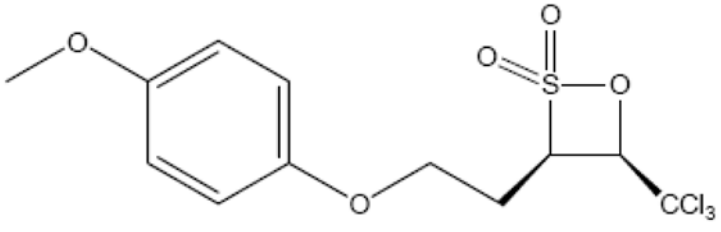
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK106-01rohwirklichkochSulton_MeOPhOEt_Ethanol.met6-16-2006 3-01-01 PM173.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 11/12/2006 11:23:05 PM
Printed: 11/12/2006 11:23:28 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
29.580	138978774	99.690	2656343	99.746
46.917	431601	0.310	6760	0.254
Totals	139410375	100.000	2663103	100.000

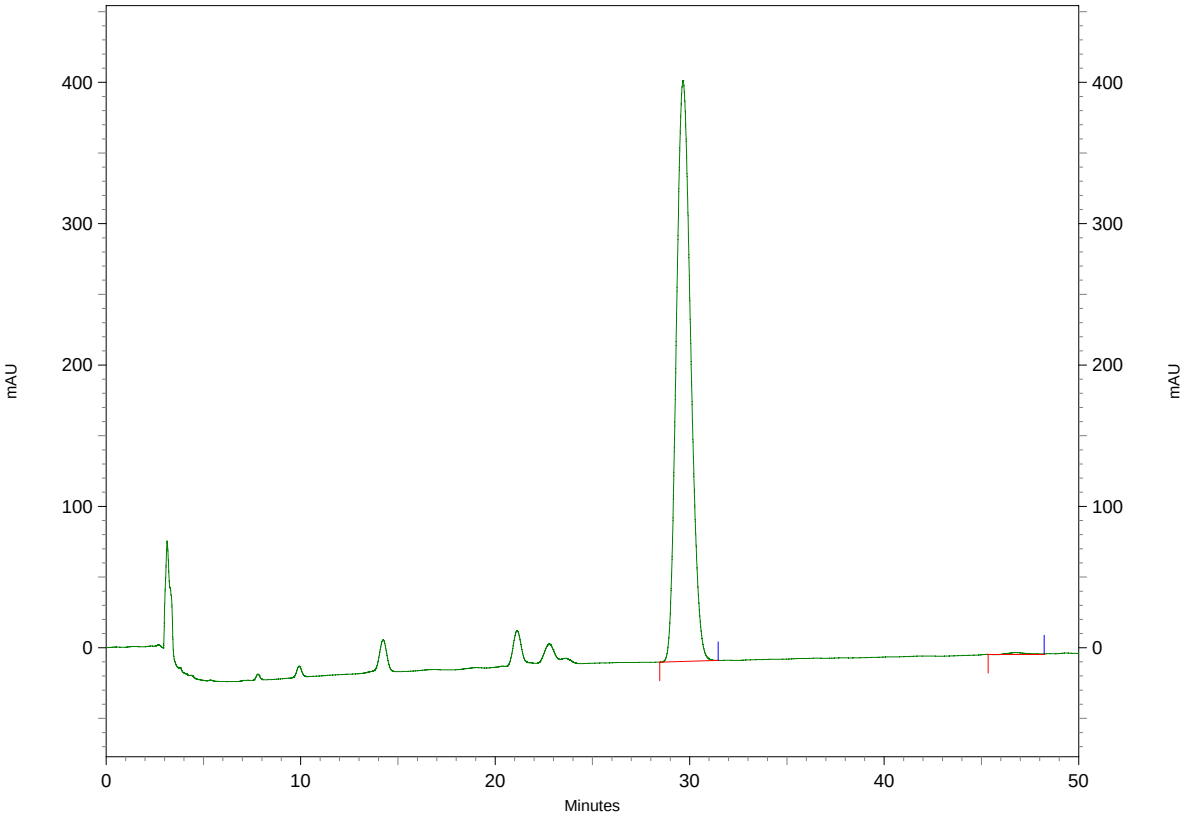


2f

M=Bi for formation of β-Sultone

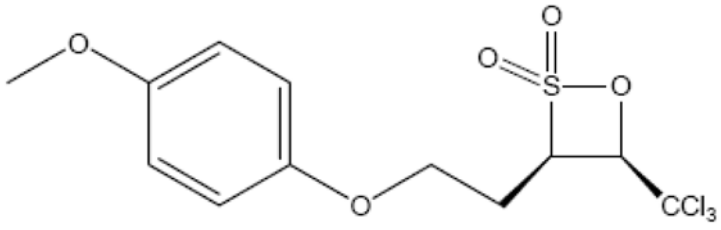
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK106-02rohkochSulton_MeOPhOEt_Ethanol.met6-16-2006 2-07-44 PM171.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Cl3-BnSulfon.met
Acquired: 6/16/2006 3:04:59 PM
Printed: 11/12/2006 11:24:51 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
29.657	83820121	99.577	1643625	99.698
46.777	356376	0.423	4972	0.302
Totals	84176497	100.000	1648597	100.000

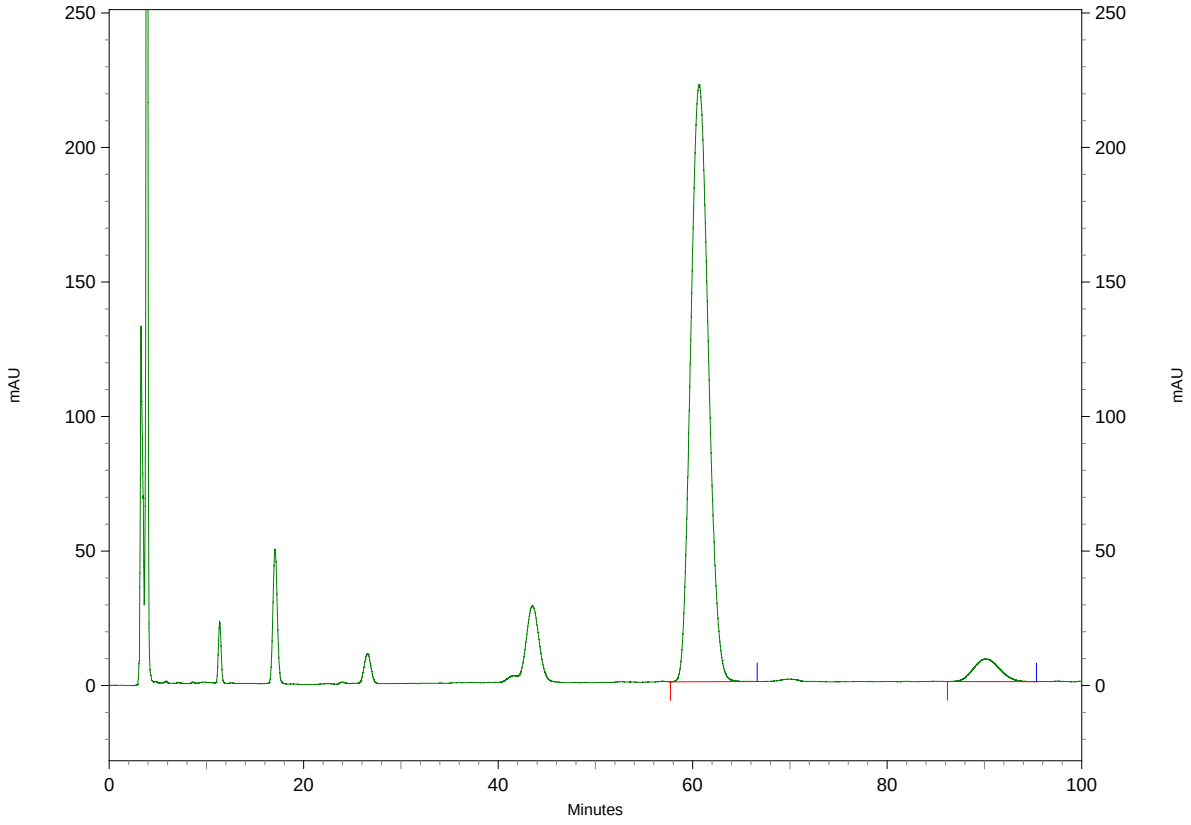


2f

M=In for formation of β-Sultone

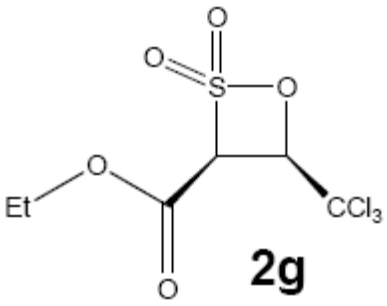
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK130-8-F7-14kochSulton_MeOPhOEt_Ethanol.met9-11-2006 11-36-03 AM172.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\CH2Cl_PhSulfonatEt.met
Acquired: 11/13/2006 10:21:23 AM
Printed: 11/13/2006 10:28:15 AM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
60.673	112279046	94.652	887661	96.344
90.133	6343608	5.348	33680	3.656
Totals	118622654	100.000	921341	100.000

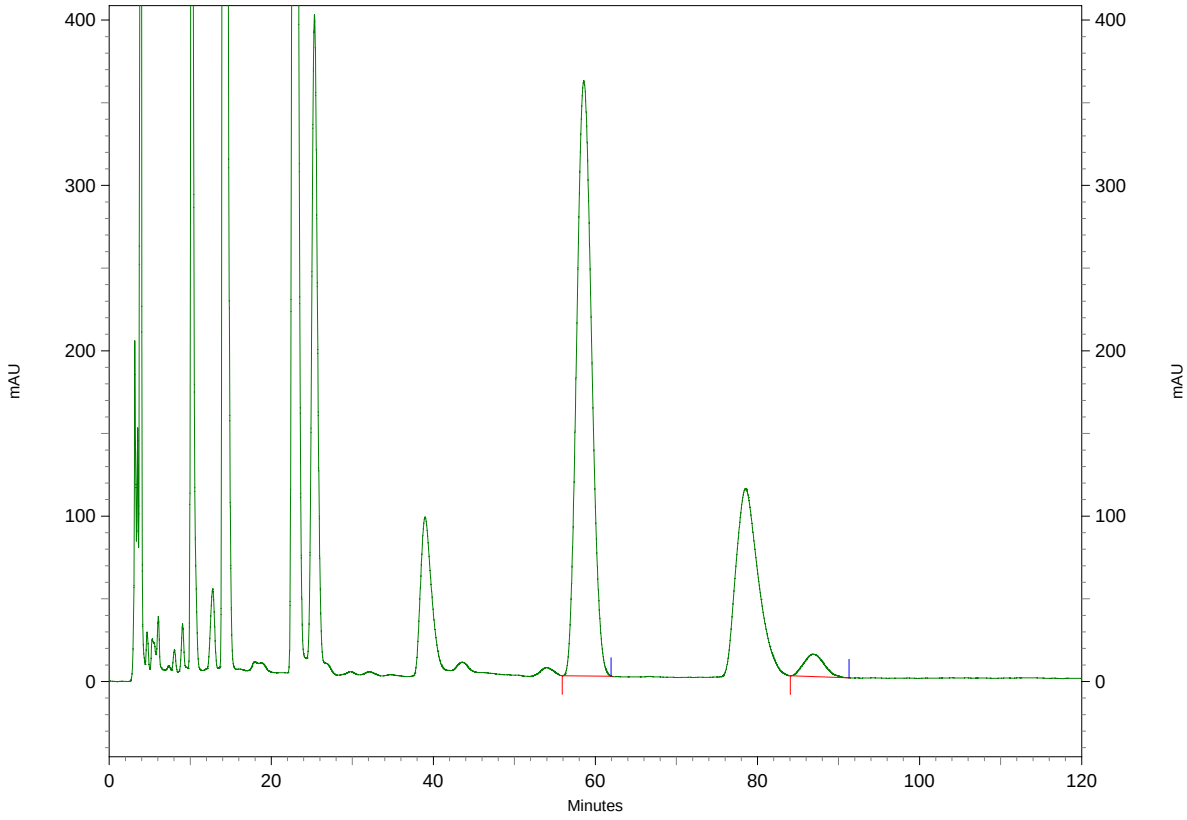


2g

M=Bi for formation of β-Sultone

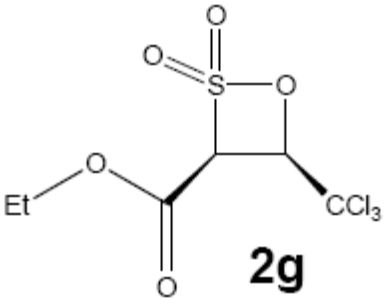
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK130-2rohkochSulton_MeOPhOEt_Ethanol.met8-21-2006 9-29-04 PM174.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\CH2Cl_PhSulfonatEt.met
Acquired: 8/22/2006 9:26:58 AM
Printed: 11/13/2006 10:26:46 AM



UV Results

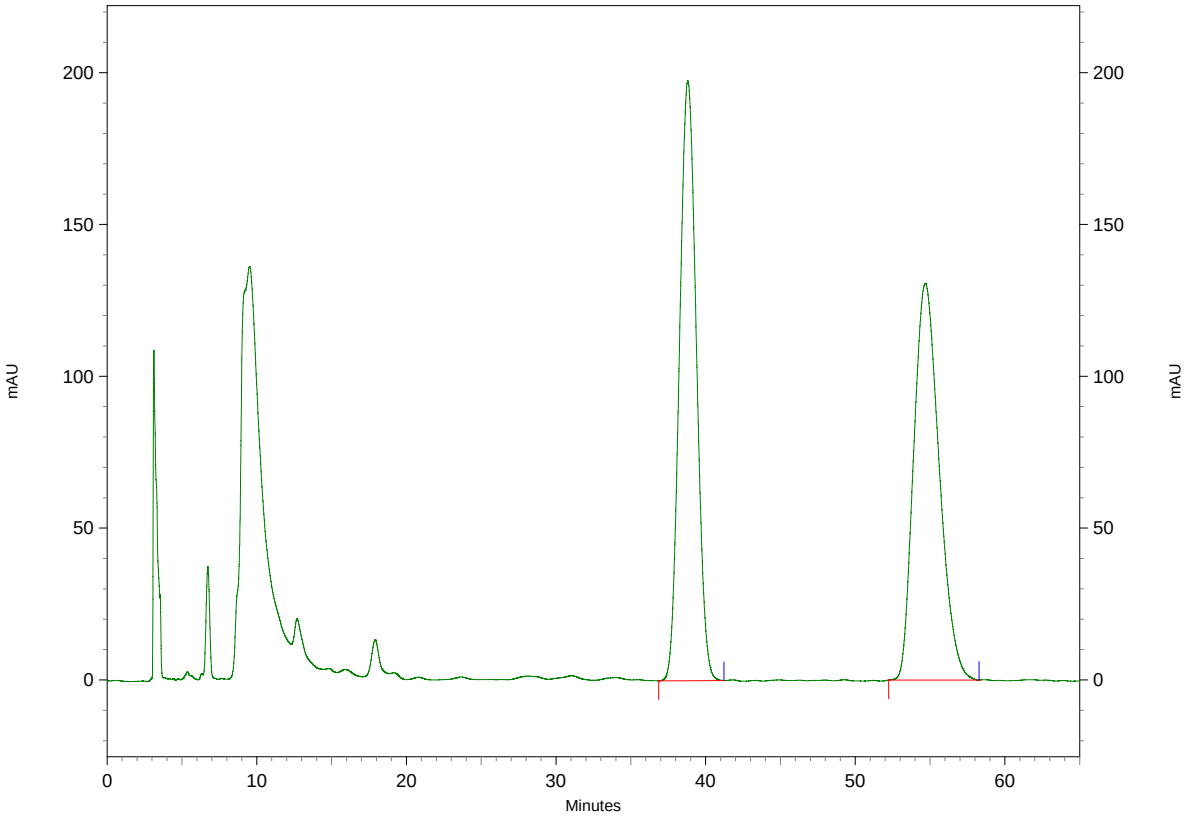
Retention Time	Area	Area Percent	Height	Height Percent
58.573	183635485	95.052	1439813	96.365
86.860	9559332	4.948	54306	3.635
Totals	193194817	100.000	1494119	100.000



M=In for formation of β-Sultone

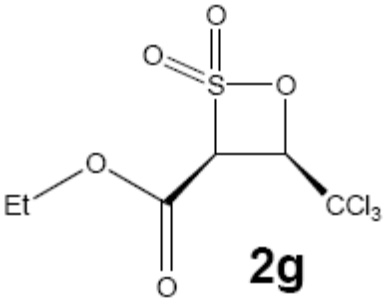
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK134-1-K1kochSulton_MeOPhOEt_Ethanol.met8-21-2006 1-28-42 PM172.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\CH2Cl_PhSulfonatEt.met
Acquired: 8/21/2006 3:17:38 PM
Printed: 11/13/2006 10:18:05 AM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
38.810	61829506	50.011	790410	60.190
54.680	61802665	49.989	522776	39.810
Totals	123632171	100.000	1313186	100.000

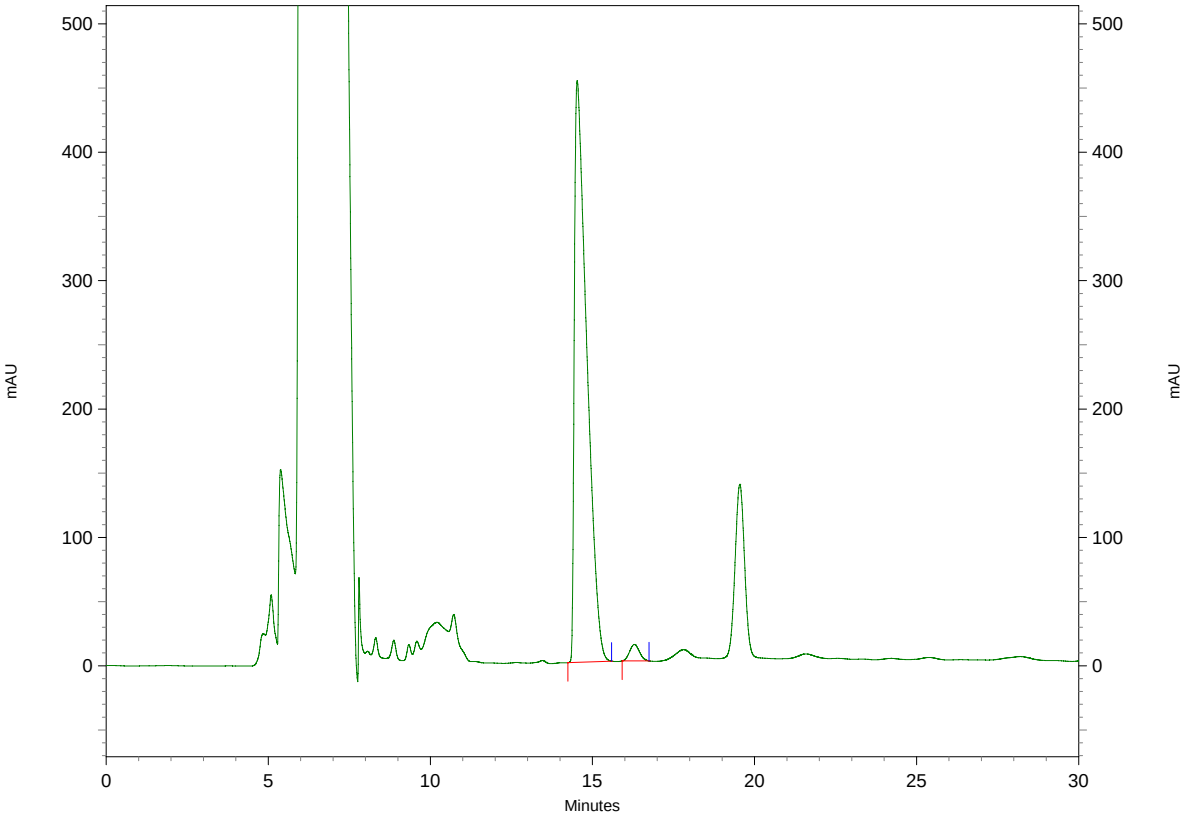


2g

racemate

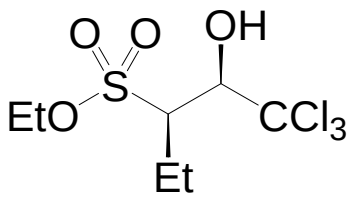
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK121-4F1-5kochEtOSulfonat_EtOH.met10-26-2006 3-49-55 PM190.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 10/26/2006 5:19:32 PM
Printed: 11/13/2006 9:26:46 PM



UV Results

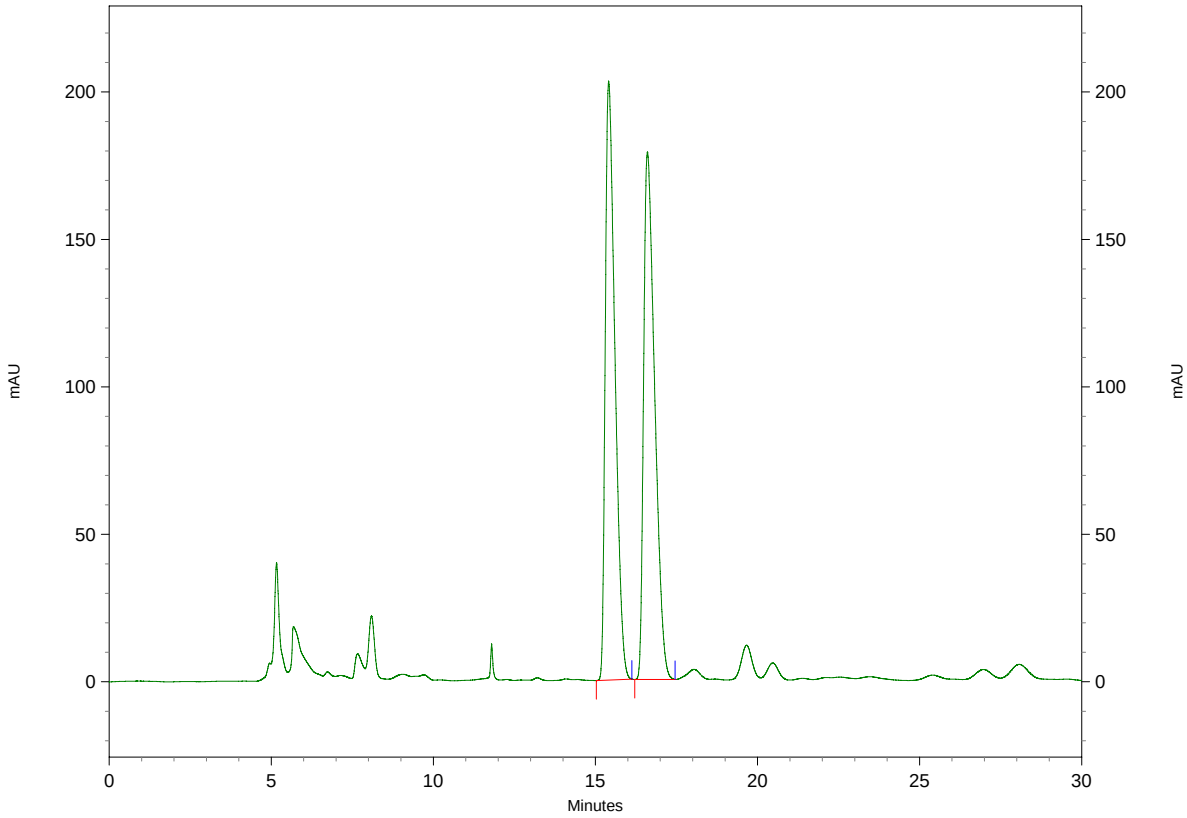
Retention Time	Area	Area Percent	Height	Height Percent
14.530	48211907	97.801	1812244	97.249
16.293	1083867	2.199	51268	2.751
Totals	49295774	100.000	1863512	100.000



12

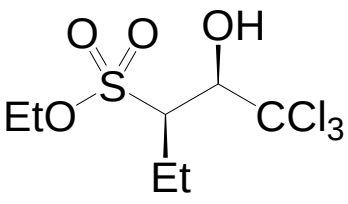
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK135-2F5-10kochEtOSulfonat_EtOH.met10-16-2006 6-31-08 PM189.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 10/16/2006 7:39:15 PM
Printed: 11/13/2006 9:21:12 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
15.407	16922766	49.924	812477	53.170
16.603	16974494	50.076	715586	46.830
Totals	33897260	100.000	1528063	100.000

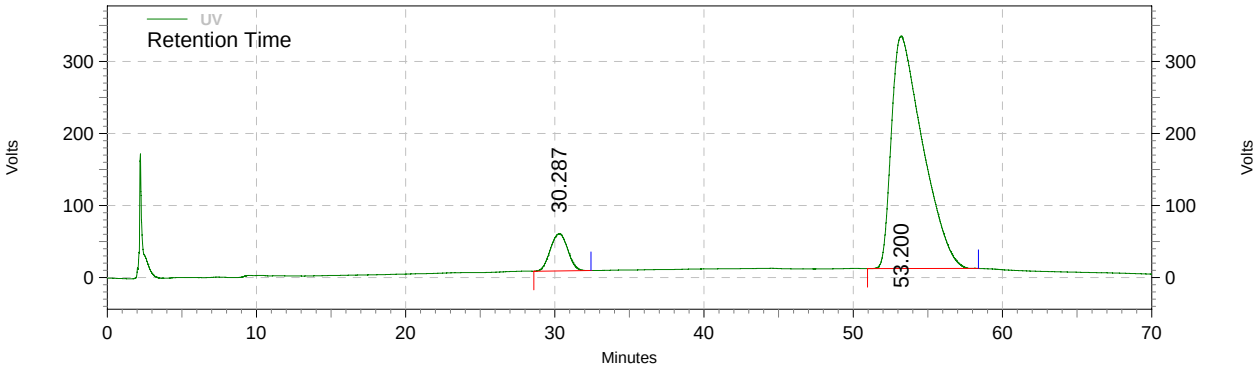


racemate

12

Area % Report

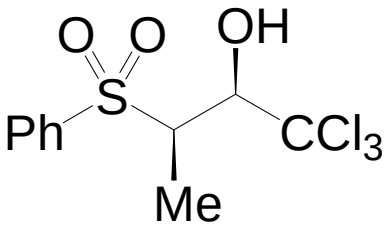
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK040-3-F16-21kochCl3-MeSulfon.met10-2-2006 8-28-24 PM171.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 10/2/2006 8:30:17 PM
Printed: 11/13/2006 9:29:03 PM



UV Results

Retention Time	Area	Area %	Height	Height %
30.287	16723095	8.18	205900	13.76
53.200	187728928	91.82	1290068	86.24

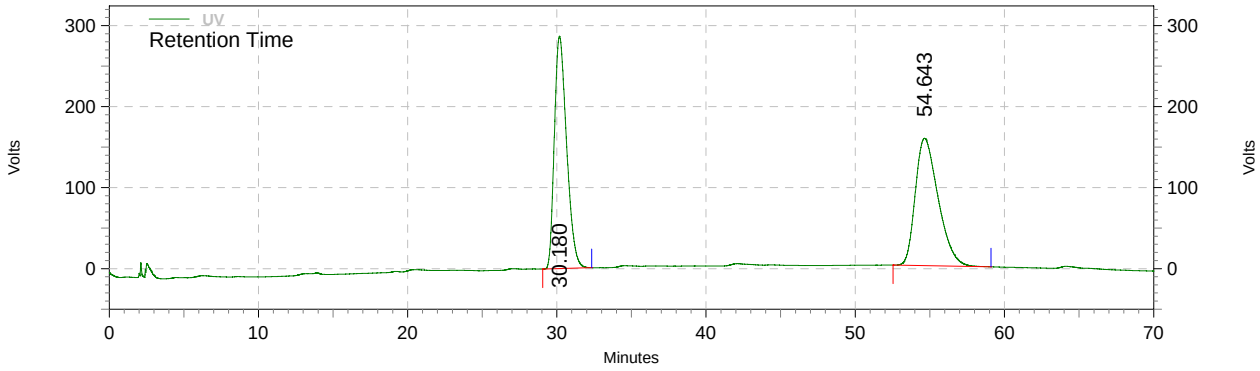
Totals	204452023	100.00	1495968	100.00



14a

Area % Report

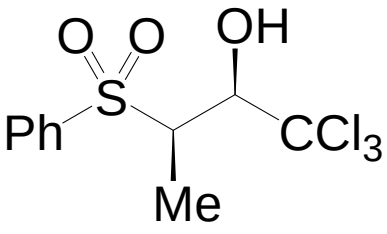
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK136-1-F11-21kochCl3-MeSulfon.met10-2-2006 6-05-47 PM171.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 10/2/2006 6:06:56 PM
Printed: 11/13/2006 9:30:08 PM



UV Results

Retention Time	Area	Area %	Height	Height %
30.180	65389496	49.42	1147389	64.61
54.643	66926212	50.58	628609	35.39

Totals	132315708	100.00	1775998	100.00



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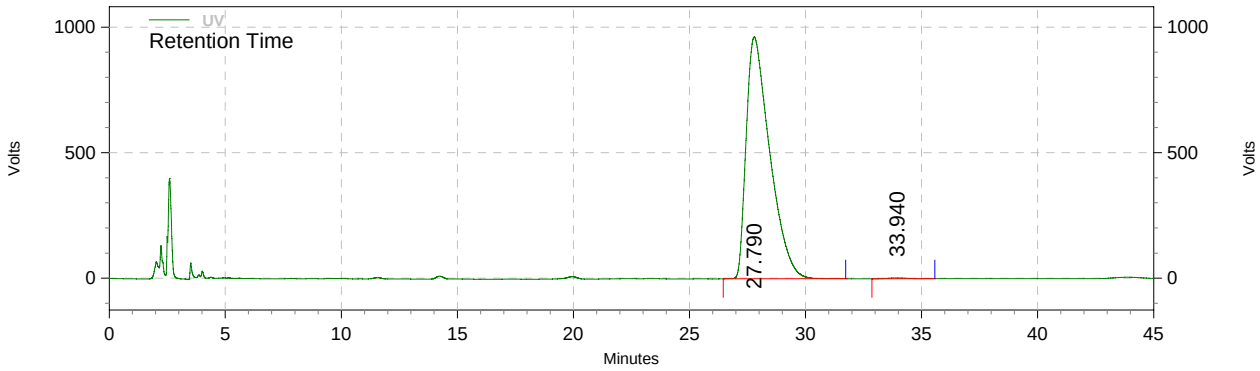
14a

Area % Report

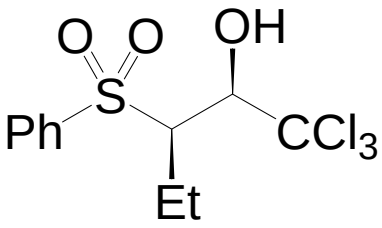
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK125-2rohkochCl3-EtSulfon.met9-28-2006 11-27-13 AM171.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 9/28/2006 11:28:13 AM
Printed: 11/13/2006 9:31:28 PM



UV Results

Retention Time	Area	Area %	Height	Height %
27.790	271532077	99.75	3850228	99.68
33.940	672767	0.25	12286	0.32

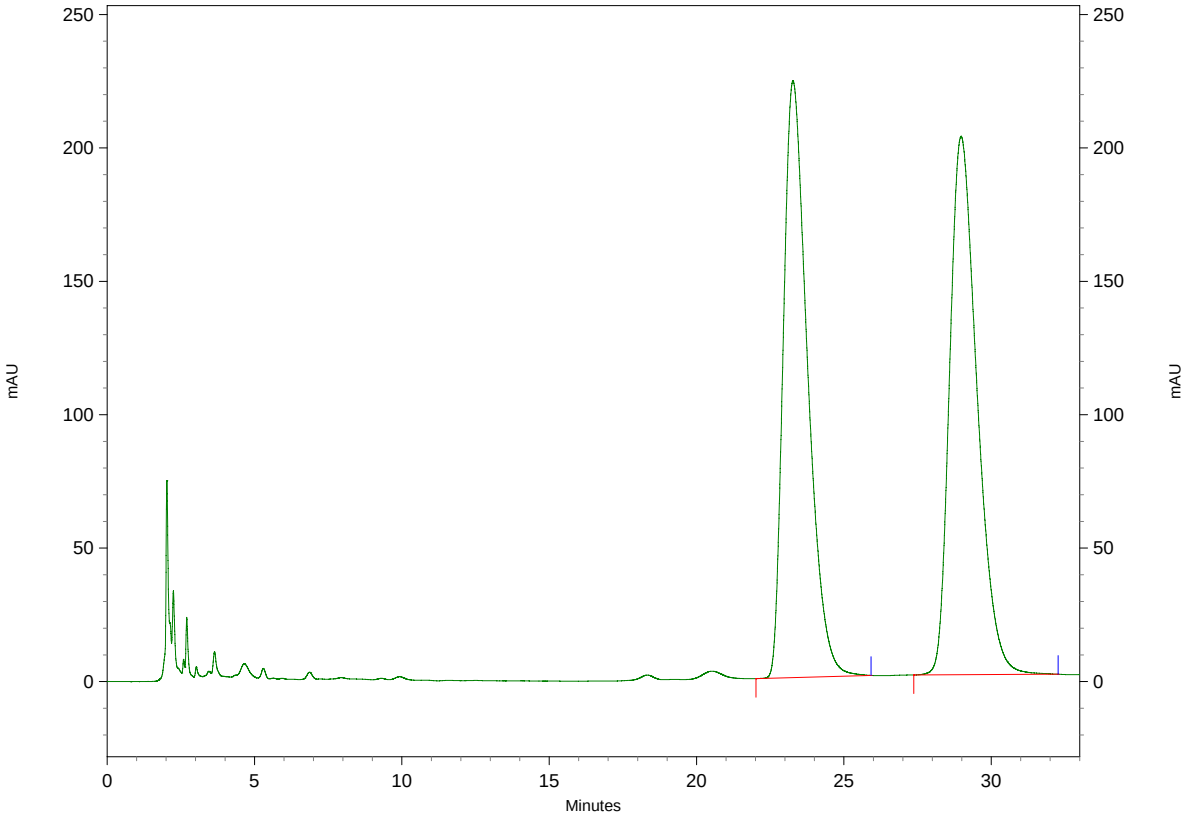
Totals	272204844	100.00	3862514	100.00



14b

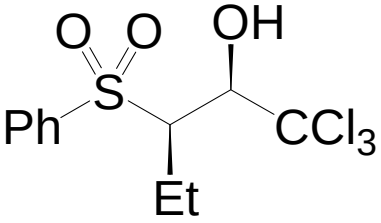
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK073-2washkochCl3-EtSulfon.met4-24-2006 1-02-03 PM.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 4/24/2006 1:40:48 PM
Printed: 11/13/2006 9:33:08 PM



UV Results

Retention Time	Area	Area Percent	Height	Height Percent
23.270	51068529	49.451	894895	52.572
28.977	52202340	50.549	807326	47.428
Totals	103270869	100.000	1702221	100.000



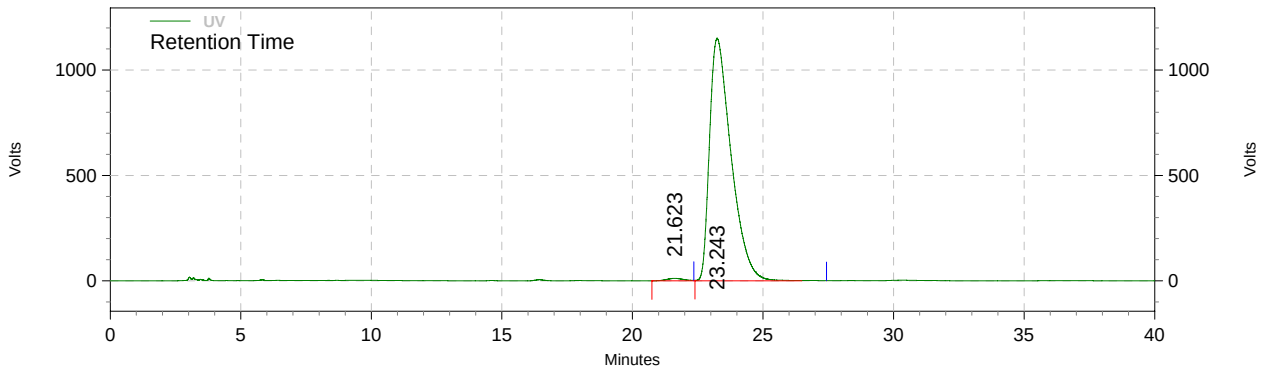
racemate

14b

Area % Report

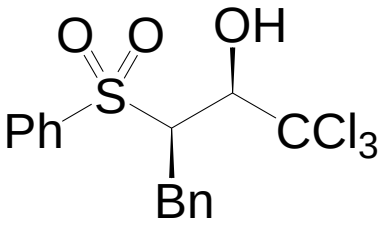
Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK143-1-F4-12kochCl3-BnSulfon.met11-1-2006 5-47-28 PM190.dat

Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 11/1/2006 5:48:29 PM
Printed: 11/13/2006 9:33:55 PM



UV Results

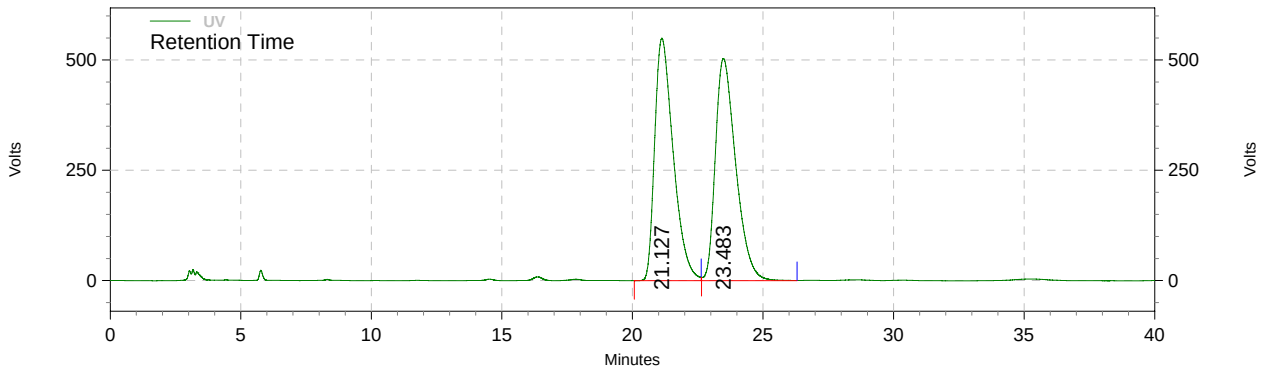
Retention Time	Area	Area %	Height	Height %
21.623	2114660	0.79	46795	1.01
23.243	264640279	99.21	4605493	98.99
Totals	266754939	100.00	4652288	100.00



14e

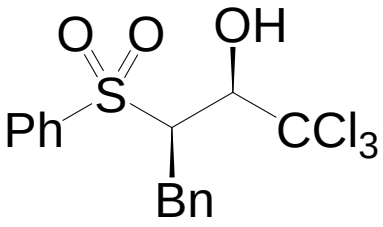
Area % Report

Data File: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Data\FK139-4-F11-20kochCl3-BnSulfon.met11-1-2006 5-01-19 PM190.dat
Method: C:\EZChrom Elite\Enterprise\Projects\SulfonatII\Method\Sulton_EtGlyMeOPhO.met
Acquired: 11/1/2006 5:02:18 PM
Printed: 11/13/2006 9:35:10 PM



UV Results

Retention Time	Area	Area %	Height	Height %
21.127	108703196	49.82	2198158	52.17
23.483	109483957	50.18	2015231	47.83
Totals	218187153	100.00	4213389	100.00



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14e