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Primary amine-metal Lewis acid bifunctional catalysts: the application to asymmetric direct aldol reaction

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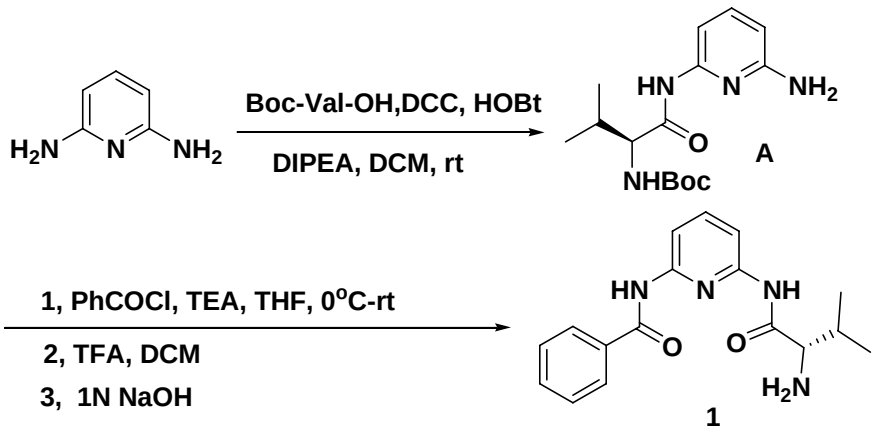
General: All NMR spectra were recorded on Bruker-500 or 300 MHz spectrometer. Optical rotation was measured on Rudolph Research Autopol III. Ee values were measured on chiral HPLC analysis using Gold Nouveau Chromatography system and the data was recorded on Shimadzu C-R6A Chromatopac integrator. Chiral AD-H and As-H column were purchased from Daicel Chemical Industries. Routine monitoring of the reaction was performed by TLC using precoated silica gel plates. Cyclohexanone was ACS reagent pure. THF was dried on Innovative Technology solvent purification system. All the other reagents were purchased from Acros or Aldrich and used directly.

Synthesis of the ligands

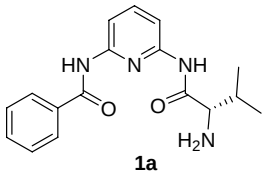


To a stirred solution of N-Boc-L-valine (2.17g, 10 mmol) in CH_2Cl_2 (100 mL) was added pyridine-2,6-diamine (10 mmol, 1.09g), DCC (2.3g, 10 mmol), HOBt (1.5 g, 10 mmol) and DIPEA (1.25 mL, 10 mmol) at 0 ^oC. This reaction mixture was stirred at room temperature for 24 h. The solution was filtered and washed with aqueous $NaHCO_3$. The organic phase was evaporated under reduced pressure and purified by column chromatography (silica gel) to give the pure product **A** (1.43 g, 46%). $[\alpha]_D^{25}$= -10.0 (c= 0.24, $CHCl_3$); ^1H NMR (300 MHz, $CDCl_3$) δ 0.95 (d, *J* = 6.9 Hz, 3H), 1.01 (d, *J* = 6.9 Hz, 3H), 1.47 (s, 9H), 2.22-2.28 (m, 1H), 4.10-4.17 (m, 1H), 4.35 (br, 2H), 5.12 (m, 1H), 6.27 (d, *J* =7.8 Hz, 1H), 7.28 (t, *J* =7.8 Hz, 1H), 7.57 (d, *J* =7.8 Hz, 1H), 8.12 (br, 1H); ^{13}C NMR (125Hz, $CDCl_3$) δ 24.61, 27.59, 30.69, 42.61, 57.08, 73.96, 121.97, 122.81, 129.38, 133.17, 143.26, 148.25, 214.79; MS (ESI) 331.2

$(M+Na)^+$; HRMS exact mass calcd for ($C_{15}H_{24}O_4N_3$+Na) requires m/z 331.1746, found m/z 331.1750.

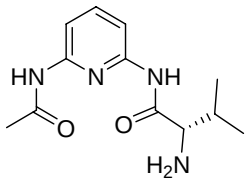
Product **A** (0.92 g, 3 mmol) was dissolved in THF (30 mL). The solution was cooled down to 0 ^oC and TEA (1 mL, 6.6 mmol) was added. Then to this solution Benzoyl Chloride (0.38 mL, 3.3 mmol) was added dropwise at 0 ^oC. After the solution was stirred at 0 ^oC for 30 min, the resulting solution was stirred at room temperature overnight. The solid was filtered off and solvent removed, the residue was purified through column chromatography on silica gel (eluent: Hexane Ethyl Acetate = 3:1) to give the product (1.11g, 90%).

The obtained N-Boc compound (1.11g) was dissolved into DCM (5 mL) and TFA (5 mL) and stirred at rt for 4h. The reaction mixtue was evaporated and dissolved in Ethyl Acetate. 1 N NaOH solution was used to tune pH to 9 and The mixture was extated with Ethyl Acetate. Then the solvent was evaporated to dryness to get the pure product **1a** (0.82g, 97%).



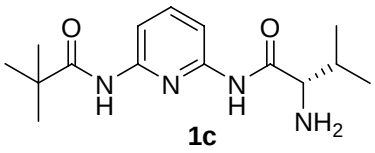
1a

(S)-N-(6-(2-amino-3-methylbutanamido)pyridin-2-yl)benzamide (1a) $[\alpha]_D^{25}$= +6.5 (c= 0.15, $CHCl_3$); ^1H NMR (500 MHz, DMSO) δ 0.87 (d, *J* = 7.0 Hz, 3H), 0.98 (d, *J* = 7.0 Hz, 3H), 2.15-2.17 (m, 1H), 3.42 (m, 1H), 7.51-7.61(m, 3H), 7.87-7.89 (m, 3H), 8.00-8.02 (m, 2H), 10.56 (s, 1H). ^{13}C NMR (125Hz, DMSO) δ 16.24, 19.11, 30.67, 60.16, 109.51, 109.86, 127.07, 128.49, 132.01, 133.87, 140.40, 149.05, 149.77, 165.68, 172.02. MS (ESI) 313.2 $(M+H)^+$; HRMS exact mass calcd for ($C_{17}H_{20}N_4O_2$+H) requires m/z 313.1664, found m/z 313.1653.



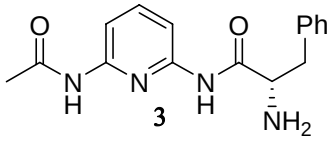
1b

(S)-N-(6-acetamidopyridin-2-yl)-2-amino-3-methylbutanamide(1b) $[\alpha]_D^{25}$= +37.0 (c= 0.12, $CHCl_3$); ^{1}H NMR (500 MHz, DMSO) δ 0.78 (d, *J* = 7.0 Hz, 3H), 0.92 (d, *J* = 7.0 Hz, 3H), 2.05-2.10 (m, 4H), 3.22 (m, 1H), 7.73-7.75(m, 3H), 10.29 (s, 1H). ^{13}C NMR (125Hz, DMSO) δ 16.94, 19.90, 24.36, 31.39, 60.49, 108.20, 109.17, 140.77, 149.89, 151.12, 169.69, 174.42. MS (ESI) 248.9 $(M-H)^-$; HRMS exact mass calcd for ($C_{12}H_{18}N_4O_2$+H) requires m/z 251.1508, found m/z 251.1500.



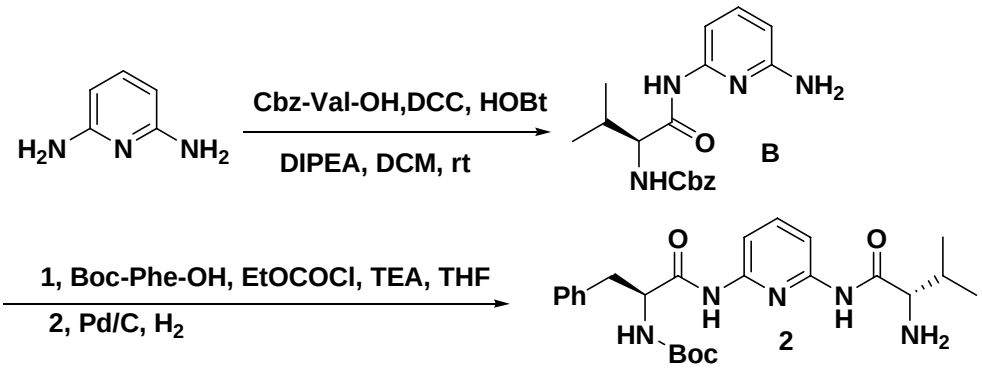
1c

(S)-2-amino-3-methyl-N-(6-pivalamidopyridin-2-yl)butanamide (1c) $[\alpha]_D^{25}$= +3.9 (c= 0.21, $CHCl_3$); ^{1}H NMR (500 MHz, CD_3OD) δ 1.04 (d, *J* = 6.5 Hz, 3H), 1.09 (d, *J* = 6.5 Hz, 3H), 1.29 (s, 9H), 3.34 (m, 1H), 7.68 (m, 1H), 7.73 (d, *J* = 8.0 Hz, 1H), 7.82 (d, *J* = 8.0 Hz, 1H). ^{13}C NMR (125Hz, CD_3OD) δ 15.89, 17.74, 36.15, 36.17 , 39.43, 56.07, 109.08, 109.23, 139.96, 149.44, 150.53, 169.23, 178.31. MS (ESI) 293.2 $(M+H)^+$; HRMS exact mass calcd for ($C_{15}H_{24}N_4O_2$+H) requires m/z 293.1977 found m/z 293.1976.



3

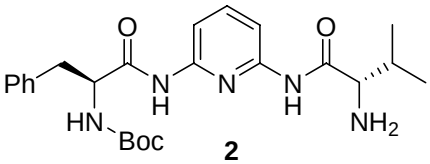
(S)-N-(6-acetamidopyridin-2-yl)-2-amino-3-phenylpropanamide (3) $[\alpha]_D^{25}$= -8.8 (c= 0.12, $CHCl_3$); ^{1}H NMR (500 MHz, DMSO) δ 2.06 (s, 3H), 2.71 (dd, *J* = 9.0, 13.5 Hz, 1H), 3.10 (dd, , *J* = 4.5, 9.0 Hz, 1H), 3.64 (m, 1H), 7.25-7.27 (m, 5H), 7.75-7.77 (m, 3H), 10.29 (s, 1H). ^{13}C NMR (125Hz, DMSO) δ 24.36, 40.51, 56.86, 108.20, 109.26, 126.75, 128.69, 129.76, 138.84, 140.79, 149.90, 151.12, 169.71, 174.12. MS (ESI) 321.2 $(M+Na)^+$; HRMS exact mass calcd for ($C_{16}H_{18}N_4O_2$+H) requires m/z 299.1508, found m/z 299.1500.



To a stirred solution of N-Cbz-L-valine (2.51g, 10 mmol) in CH_2Cl_2 (100 mL) was added pyridine-2,6-diamine (1.09 g, 10 mmol), DCC (2.3g, 10 mmol), HOBt (1.5 g, 10 mmol) and DIPEA (1.25 mL, 10 mmol) at 0^{o}C. This reaction mixture was stirred at room temperature for 24 h. The solution was filtered and washed with aqueous $NaHCO_3$. The organic phase was evaporated under reduced pressure and purified by column chromatography (silica gel) to give the pure product **B** (1.7 g, 49%).

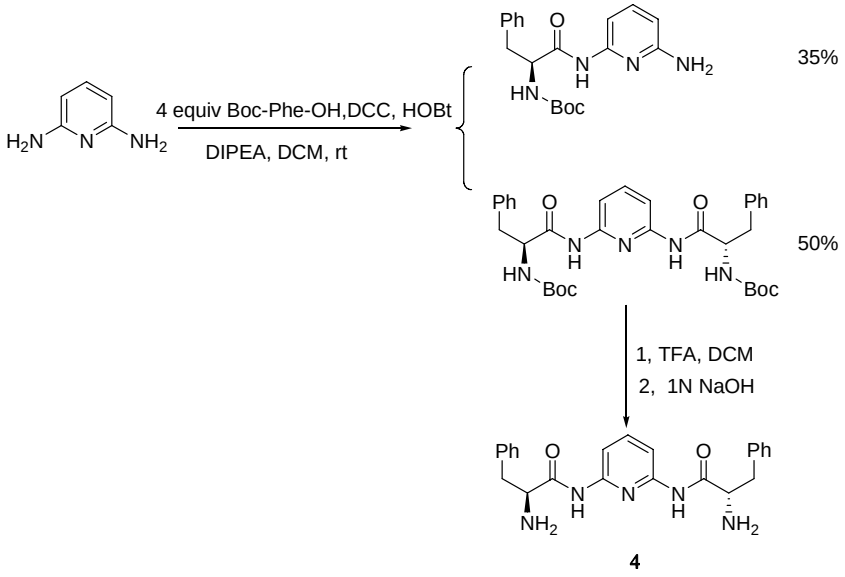
N-Boc-L-Phenylalanine (1.0 g, 2.9 mmol) was dissolved in THF (20 mL). The solution was cooled down to 0 ^{o}C. TEA (0.6 mL, 4.4 mmol) was added. Then to this solution ethylchloroformate (0.45 mL, 4.4 mmol) was added dropwise for 15 min. After the solution was stirred at 0 ^{o}C for 45 min, amine **B** (1.0 g, 2.9 mmol) was added slowly for 10 minutes in 10 mL THF solution at 0 ^{o}C. The resulting solution was stirred at room temperature for 16 h, and then refluxed for 3 h. After cooling down to room temperature, the solid was filtered off and solvent removed. The oily product was then dissolved in DCM. The mixture was washed with aqueous $NaHCO_3$ and dried with anhydrous Na_2SO_4. After removal of the solvent, the residue was purified through column chromatography on silica gel (eluent: Hexane: Ethyl Acetate = 2:1) to give the product (0.96 g, yield: 56%).[1]

The obtained compound (0.96 g), 10% Pd/C (200 mg) and methanol (30 mL) were mixed in a 100 mL flask. After stirring under hydrogen (1 atm) for 4 h, the solution was filtered on Celite to remove the Pd/C, and then evaporated to dryness to give the products **2** (0.74 g, yield: 99 %).



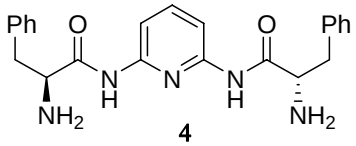
2

$[\alpha]_D^{25}$= -6.2 (c= 0.08, $CHCl_3$);^{1}H NMR (500 MHz, $CDCl_3$) δ 1.15 (d, *J* = 6.5 Hz, 3H), 1.28 (d, *J* = 6.5 Hz, 3H), 1.45 (s, 9H), 2.33 (m, 1H), 3.05-3.20 (m, 2H), 3.57-3.69 (m, 1H), 4.52 (m, 1H), 5.11 (m, 1H), 7.24-7.33 (m, 6H), 7.63-7.73 (m, 2H), 7.93 (m, 1H). ^{13}C NMR (125Hz, $CDCl_3$) δ 16.91, 19.45, 26.87, 27.88, 28.25, 28.84, 63.10, 78.70, 109.25, 110.01, 127.00, 128.55, 128.68, 129.20, 129.31, 129.72, 148.28, 149.05x, 170.24, 174.74. MS(ESI) 456.3 $(M+H)^+$; HRMS exact mass calcd for ($C_{24}H_{33}N_5O_4$+H) requires m/z 456.2611, found m/z 456.2604.



4

Procedure as synthesis of compound **1**.



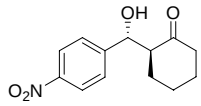
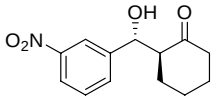
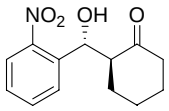
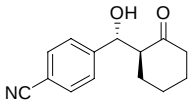
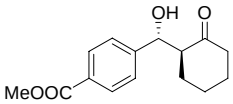
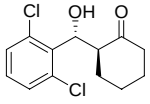
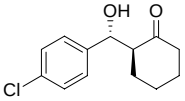
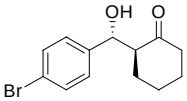
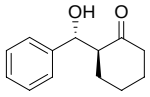
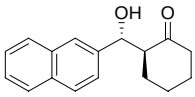
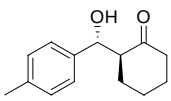
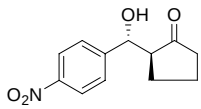
4

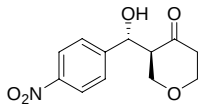
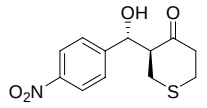
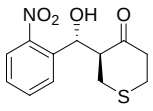
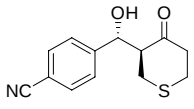
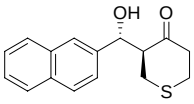
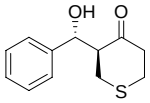
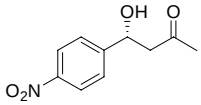
(2S,2'S)-N,N'-(pyridine-2,6-diyl)bis(2-amino-3-phenylpropanamide) (4) $[\alpha]_D^{25}$= -13.3 (c= 0.15, $CHCl_3$); ^{1}H NMR (500 MHz, CD_3OD) δ 2.89 (dd, *J* = 7.5, 13.5 Hz, 1H), 3.14 (dd, , *J* = 5.5, 13.5 Hz, 1H), 3.74 (t, *J* = 6.5Hz, 1H), 7.22-7.31 (m, 10H), 7.75-7.85 (m, 3H). ^{13}C NMR (125Hz, CD_3OD) δ 45.03, 61.73, 112.82, 124.01, 132.78, 134.01, 135.14, 143.62, 155.09, 179.18. MS (ESI) 404.3 $(M+H)^+$; HRMS

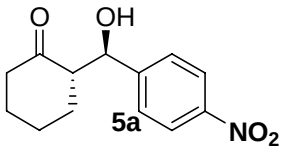
exact mass calcd for ($C_{23}H_{25}N_5O_2$+H) requires m/z 404.2086, found m/z 404.2079.

General procedure of the enantioselective aldol reaction: A mixture of $CuCl_2$ (5.4 mg, 0.04 mmol, 20 mol%), $AgSbF_6$ (27.5 mg, 0.08 mmol, 40 mol%), ligand **1c** (11.7 mg, 0.04 mmol, 20 mol%), and cyclohexanone (1 mL) was stirred at room temperature for 4 h. And then the aldehyde (0.2 mmol) was added. The resulting mixture was stirred for 12-48 h. After the reaction was completed (monitered by TLC), the reaction mixture was treated with saturated ammonium chloride solution, and extracted with ethyl acetate. After removal of the solvent, mixture ^{1}H NMR was taken to determine diastereoselectivity. The mixture was purified through column chromatography on silica gel (eluent: mixture of Hexane and ethyl acetate) to give the pure products. All aldol products are known compounds and their spectroscopic data are identical with those reported. The ee values were determined by chiral HPLC analysis. The HPLC conditions and retention time were collected in **Table 1** .

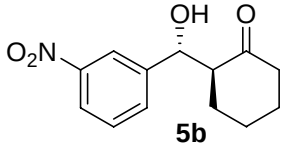
Table 1 HPLC Conditions and retention time

Compound	Eluent *i*-PrOH/Hexane	Flow rate (mL/min)	Column	Wave length (nm)	T(major) (min)	T(minor) (min)
	**20/80**	**1.0**	**AD-H**	**254**	**16.8**	**13.1**
	**20/80**	**1.0**	**AD-H**	**254**	**17.9**	**22.4**
	**20/80**	**0.8**	**AD-H**	**254**	**19.2**	**20.8**
	**20/80**	**0.9**	**AD-H**	**254**	**26.4**	**21.2**
	**20/80**	**1.0**	**AS-H**	**254**	**15.6**	**22.3**
	**10/90**	**1.0**	**AS-H**	**220**	**15.6**	**19.3**
	**10/90**	**1.0**	**AS-H**	**220**	**20.8**	**18.3**
	**10/90**	**1.0**	**AS-H**	**220**	**22.6**	**19.7**
	**5/95**	**0.5**	**AS-H**	**220**	**34.1**	**36.8**
	**10/90**	**0.7**	**AS-H**	**220**	**26.0**	**22.4**
	**10/90**	**0.7**	**AS-H**	**220**	**15.0**	**18.4**
	**5/95**	**0.7**	**AD-H**	**254**	**87.8**	**85.1**

	**20/80**	**1.0**	**AD-H**	**254**	**21.3**	**17.8**
	**20/80**	**1.0**	**AD-H**	**254**	**31.7**	**18.8**
	**20/80**	**0.7**	**AD-H**	**254**	**24.8**	**20.4**
	**20/80**	**0.7**	**AD-H**	**254**	**30.4**	**18.8**
	**20/80**	**0.7**	**AS-H**	**220**	**22.7**	**34.5**
	**20/80**	**0.7**	**AS-H**	**220**	**16.9**	**24.1**
	**30/70**	**1.0**	**AS-H**	**254**	**15.0**	**18.8**

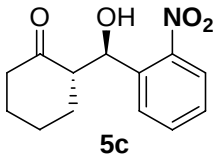


(S)-2-((R)-hydroxy(4-nitrophenyl)methyl)cyclohexanone (**5a**)[2] yield 90% ; dr >30/1; Ee 95% ; $[\alpha]_D^{25}$= +12.0 (c= 0.20, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.34-1.82 (m, 5H), 2.07 (m, 1H), 2.33-2.35 (m, 1H), 2.45-2.46 (m, 1H), 2.48-2.56 (m, 1H), 4.04 (s, 1H), 4.87 (dd, *J* = 3 Hz, 8.5 Hz, 1H), 7.48 (d, *J* = 8.5 Hz, 2H), 8.18 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.70, 27.63, 30.76, 42.68, 57.20, 74.02, 123.57, 127.87, 147.59, 148.37, 214.70.



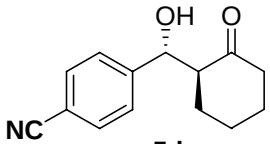
(S)-2-((R)-hydroxy(3-nitrophenyl)methyl)cyclohexanone (**5b**)[3] yield 90% ; dr 20/1; Ee 95% ; $[\alpha]_D^{25}$= +37.2 (c= 0.50, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ

1.35-1.39 (m, 1H), 1.54-1.67 (m, 4H), 2.08-2.09 (m, 1H), 2.35-2.38 (m, 1H), 2.46-2.49 (m, 1H), 2.61(m, 1H), 4.14 (br, 1H), 4.88 (d, *J* = 8.5 Hz, 1H), 7.49 (t, *J* = 8.0 Hz, 1H), 8.18 (d, *J* = 7.5 Hz, 2H), 8.12-8.19 (m, 1H), 8.20 (s, 1H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.61, 27.59, 30.69, 42.61, 57.08, 73.96, 121.97, 122.81, 129.26, 133.17, 143.26, 148.25, 214.79.



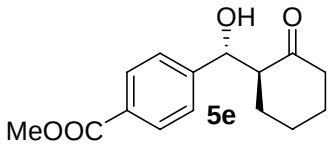
5c

(S)-2-((R)-hydroxy(2-nitrophenyl)methyl)cyclohexanone (**5c**)[3] yield 82% ; dr >30/1; Ee 94% ; $[\alpha]_D^{25}$= +29.1 (c= 0.31, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.55-1.84 (m, 5H), 2.05-2.06 (m, 1H), 2.31-2.33 (m, 1H), 2.41-2.44 (m, 1H), 2.72-2.74 (m, 1H), 4.15 (br, 1H), 5.42 (d, *J* = 7.0 Hz, 1H), 7.40-7.42 (m, 1H), 7.61 (m, 1H), 7.81 (m, 1H). 7.83 (m, 1H) ^{13}C NMR (125Hz, $CDCl_3$) δ 24.96, 27.73, 31.09, 42.80, 57.28, 69.76, 124.06, 128.37, 128.98, 133.04, 136.60, 148.72, 214.91



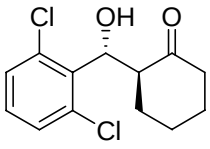
5d

4-((R)-hydroxy((S)-2-oxocyclohexyl)methyl)benzonitrile (**5d**)[3] yield 93% ; dr 12/1 ; Ee 92% ; $[\alpha]_D^{25}$= +26.2 (c= 0.41, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.34-1.85 (m, 5H), 2.10-2.14 (m, 1H), 2.36-2.39 (m, 1H), 2.49 (m, 1H), 2.51-2.58 (m, 1H), 4.07 (s, 1H), 4.85 (dd, *J* = 3 Hz, 8.5 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.68, 27.64, 30.73, 42.66, 57.14, 74.20, 111.69, 119.80, 127.78, 132.17, 146.41, 214.76.



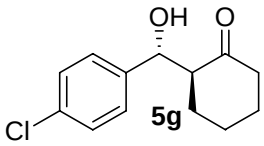
5e

methyl 4-((R)-hydroxy((S)-2-oxocyclohexyl)methyl)benzoate (5e)[3] yield 89% ; dr 9/1 ; Ee 94% ; $[\alpha]_D^{25}$= +14.6 (c= 0.27, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.29-1.33 (m, 1H), 1.51-1.79 (m, 4H) , 2.06-2.09 (m, 1H), 2.33-2.37 (m, 1H), 2.45-2.46 (m, 1H), 2.48-2.58 (m, 1H), 3.90 (s, 3H), 4.04 (s, 1H), 4.83 (d, *J* = 8.5 Hz, 1H), 7.38 (d, *J* = 8.5 Hz, 2H), 8.00 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.64, 27.66, 30.71, 42.62, 52.04, 57.24, 74.32, 126.97, 129.62, 129.66, 146.05, 166.81, 215.04.



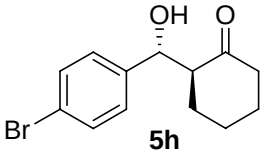
5f

(S)-2-((R)-(2,6-dichlorophenyl)(hydroxy)methyl)cyclohexanone (5f)[5] yield 98% ; dr 12/1 ; Ee 92% ; $[\alpha]_D^{25}$= -41.5 (c= 0.31, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.36-1.40 (m, 1H), 1.52-1.85 (m, 4H), 2.08-2.10 (m, 1H), 2.41-2.54 (m, 2H), 3.49-3.52 (m, 1H), 3.70 (br, 1H), 5.85 (d, *J* = 10.0 Hz, 1H), 7.17 (d, *J* = 8.0 Hz, 1H), 7.32-7.33 (m, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.69, 27.63, 29.86, 42.44, 53.65, 70.57, 129.34, 129.75, 134.73, 135.69, 214.39.



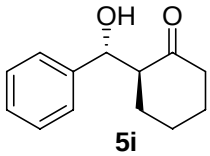
5g

(S)-2-((R)-(4-chlorophenyl)(hydroxy)methyl)cyclohexanone (5g)[3] yield 73% ; dr 6/1 ; Ee 88% ; $[\alpha]_D^{25}$= +22.2 (c= 0.20, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.29-1.33 (m, 1H), 1.55-1.81 (m, 4H), 2.09-2.58 (m, 3H), 4.04 (s, 1H), 4.78 (d, *J* = 8.0 Hz, 1H), 7.28 (d, *J* = 8.5 Hz, 2H), 7.34 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.72, 27.72, 30.76, 42.68, 57.38, 74.14, 128.39, 128.54, 138.59, 139.50, 215.29.



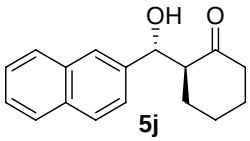
5h

(S)-2-((R)-(4-bromophenyl)(hydroxy)methyl)cyclohexanone (5h)[3] yield 60% ; dr 6/1 ; Ee 94% ; $[\alpha]_D^{25}$= +20.2 (c= 0.30, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.27-1.30 (m, 1H), 1.52-1.78 (m, 4H), 2.33-2.35 (m, 1H), 2.07-2.08 (m, 1H), 2.33-2.35 (m, 1H), 2.45-2.54 (m, 2H), 3.98 (s, 1H), 4.74 (d, *J* = 9.0 Hz, 1H), 7.18 (dd, *J* = 1.5, 6.5 Hz, 2H), 7.46 (dd, *J* = 2.0, 6.5 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.68, 27.69, 30.72, 42.63, 57.30, 74.15, 121.68, 128.71, 131.45, 140.00, 215.21.



5i

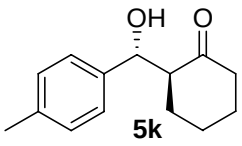
(S)-2-((R)-hydroxy(phenyl)methyl)cyclohexanone (5i)[3] yield 76% ; dr 10/1 ; Ee 86% ; $[\alpha]_D^{25}$= +20.9 (c= 0.31, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.28-1.31 (m, 1H), 1.52-1.77 (m, 4H), 2.35-2.36 (m, 1H), 2.06 (m, 1H), 2.35-2.36 (m, 1H), 2.46-2.47 (m, 1H), 2.49-2.62 (m, 1H), 3.94 (s, 1H), 4.78 (d, *J* = 8.5 Hz, 1H), 7.2-7.34 (m, 5H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.68, 27.77, 30.81, 42.63, 57.40, 74.71, 126.99, 127.85, 128.33, 140.92, 215.48.



5j

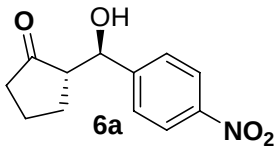
(S)-2-((R)-hydroxy(naphthalen-2-yl)methyl)cyclohexanone (5j)[5] yield 71% ; dr 12/1 ; Ee 90% ; $[\alpha]_D^{25}$= +7.3 (c= 0.30, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.32-1.35 (m, 1H), 1.50-1.76 (m, 4H), 2.06 (m, 1H), 2.36-2.38 (m, 1H), 2.48 (m, 1H), 2.72 (m, 1H), 2.49-2.62 (m, 1H), 4.08 (br, 1H), 4.96 (d, *J* = 9.0 Hz, 1H), 7.46-7.49 (m, 3H), 7.76 (s, 1H), 7.82-7.85 (m, 3H). ^{13}C NMR (125Hz, $CDCl_3$) δ 24.71, 27.80, 30.92, 42.71, 57.41, 74.92, 124.68, 125.95, 126.15, 126.26, 127.70, 127.99, 128.28,

133.17, 133.21, 138.38, 215.49



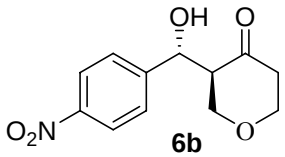
5k

(S)-2-((R)-hydroxy(p-tolyl)methyl)cyclohexanone (5k)[6] yield 75% ; dr 8/1 ; Ee 83% ; $[\alpha]_D^{25}$= +9.3 (c= 0.40, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.26-1.29 (m, 1H), 1.52-1.78 (m, 4H), 2.05-2.07 (m, 1H), 2.33-2.45 (m, 4H), 2.46-2.48 (m, 1H), 2.60 (m, 1H), 3.91 (br, 1H), 4.74 (d, *J* = 8.5 Hz, 1H), 7.14 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 7.0 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 21.11, 24.69, 27.79, 30.84, 42.64, 57.42, 74.50, 125.66, 126.89, 129.01, 137.50, 215.56.



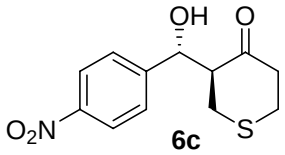
6a

(S)-2-((R)-hydroxy(4-nitrophenyl)methyl)cyclopentanone (**6a**)[2] yield 66% ; dr 3/1 ; Ee 86% ; $[\alpha]_D^{25}$= -60.0 (c= 0.40, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.60-1.79 (m, 3H), 1.97-2.49 (m, 5H), 2.99 (br, 0.18 H, -OH, syn), 4.77 (br, 1H, -OH, anti), 4.86 (d, *J* = 8.5 Hz, 0.81H, -CHOH, anti), 5.41 (s, 0.21H, -CHOH, syn), 7.54 (d, *J* = 8.5 Hz, 2H), 8.20 (d, *J* = 8.5 Hz, 2H).



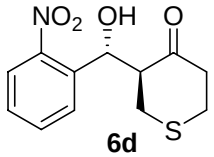
6b

(S)-3-((R)-hydroxy(4-nitrophenyl)methyl)-tetrahydropyran-4-one (**6b**)[4] yield 97% ; dr 10/1 ; Ee 87% ; $[\alpha]_D^{25}$= 1.7 (c= 0.20, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.50-2.54 (m, 1H), 2.64-2.66 (m, 1H), 2.87-2.90 (m, 1H), 3.42-3.47 (m, 1H), 3.69-3.75 (m, 2H), 4.16-4.19 (m, 1H), 4.98 (d, *J* = 8.0 Hz, 1H), 7.50 (d, *J* = 9.0 Hz, 2H), 8.20 (d, *J* = 9.0 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 42.81, 57.64, 68.33, 69.75, 71.30, 123.81, 127.46, 147.46, 147.76, 209.13.



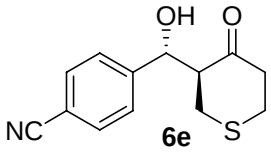
6c

(S)-3-((R)-hydroxy(4-nitrophenyl)methyl)-tetrahydrothiopyran-4-one (**6c**)[4] yield 82% ; dr 22/1 ; Ee 94% ; $[\alpha]_D^{25}$= 15.0 (c= 0.50, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.54-2.56 (m, 1H), 2.64-2.69 (m, 1H), 2.77-2.87 (m, 2H), 2.95-3.00 (m, 3H), 3.69 (br, 1H), 5.07 (d, *J* = 8.0 Hz, 1H), 7.55 (d, *J* = 8.5 Hz, 2H), 8.22-8.24 (m, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 30.80, 32.80, 44.70, 59.46, 73.15, 123.79, 126.72, 127.81, 147.74, 211.14.



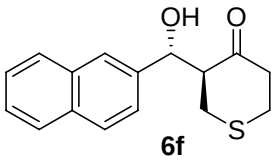
6d

(S)-3-((R)-hydroxy(2-nitrophenyl)methyl)-tetrahydrothiopyran-4-one (**6d**)[4] yield 76% ; dr 25/1 ; Ee 94% ; $[\alpha]_D^{25}$= -30.0 (c= 0.37, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.60-2.64 (m, 1H), 2.75-2.83 (m, 2H), 2.94-3.15 (m, 3H), 3.16-3.18 (m, 1H), 3.90 (br, 1H), 5.55 (d, *J* = 7.0 Hz, 1H), 7.46 (d, *J* = 7.5 Hz, 1H), 7.66 ((d, *J* = 7.5 Hz, 1H), 7.77 (d, *J* = 7.0 Hz, 1H), 7.89 (d, *J* = 7.0 Hz, 1H) . ^{13}C NMR (125Hz, $CDCl_3$) δ 30.78, 33.36, 45.16, 59.55, 69.37, 124.39, 128.85, 129.03, 133.48, 136.00, 148.59, 211.46.



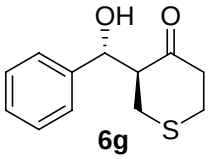
6e

4-((R)-hydroxy((S)-4-oxo-tetrahydro-2H-thiopyran-3-yl)methyl)benzonitrile(**6e**)[4] yield 86% ; dr 46/1 ; Ee 91% ; $[\alpha]_D^{25}$= +7.73 (c= 0.42, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.47-2.52 (m, 1H), 2.62-2.66 (m, 1H), 2.76-2.85 (m, 2H), 2.95-3.00 (m, 3H), 3.65 (br, 1H), 4.99 (d, *J* = 8.5 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.66 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 30.82, 32.82, 44.75, 59.44, 73.42, 112.17, 118.51, 127.68, 132.44, 145.68, 211.29.



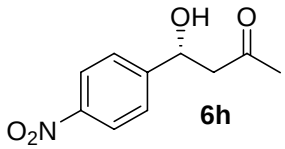
6f

(S)-3-((R)-hydroxy(naphthalen-2-yl)methyl)-tetrahydrothiopyran-4-one(**6f**)[7] yield 57% ; dr 34/1 ; Ee 87% ; $[\alpha]_D^{25}$= +23.4 (c= 0.12, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.53-2.56 (m, 2H), 2.82-2.99 (m, 4H), 3.11-3.16 (m, 1H), 5.18 (d, *J* = 8.5 Hz, 1H), 7.51-7.53 (m, 3H), 7.81 (s, 1H), 7.86-7.89 (m, 4H). ^{13}C NMR (125Hz, $CDCl_3$) δ 30.89, 32.99, 44.54, 59.63, 74.03, 124.26, 126.24, 126.36, 127.72, 127.99, 128.72, 133.11, 133.32, 137.58, 211.83.



6g

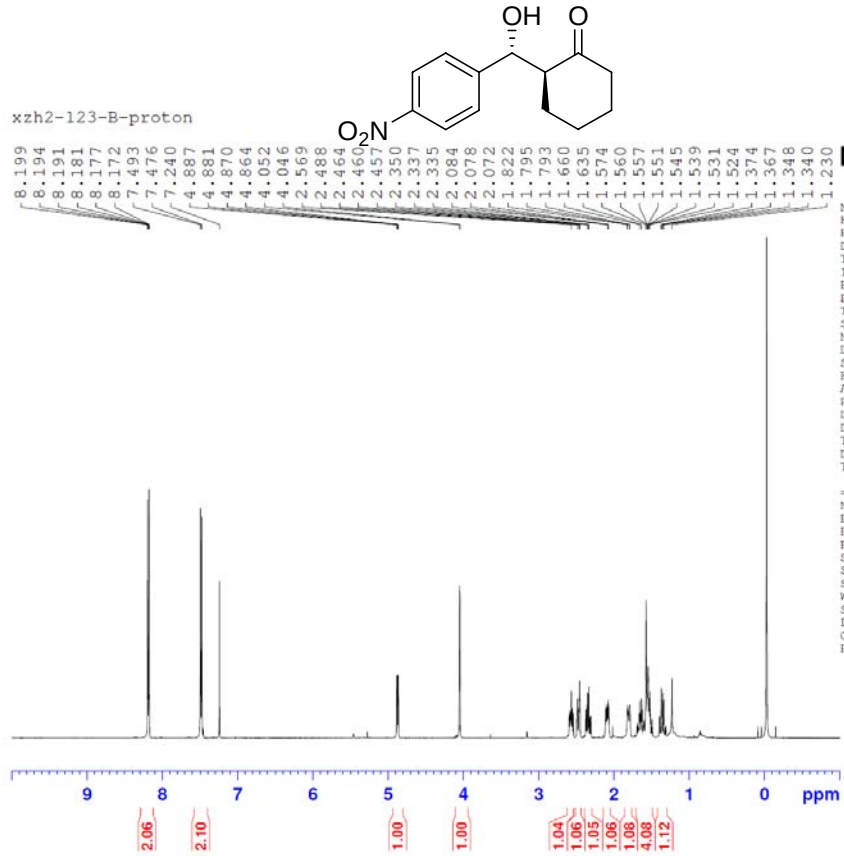
(S)-3-((R)-hydroxy(phenyl)methyl)-tetrahydrothiopyran-4-one(5r)[4] yield 68% ; dr 25/1 ; Ee 94% ; $[\alpha]_D^{25}$= +21.0 (c= 0.30, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.54-2.62 (m, 2H), 2.79-2.82 (m, 3H), 2.98-3.03 (m, 3H), 4.99 (d, *J* = 8.5 Hz, 1H), 7.34-7.38 (m, 5H). ^{13}C NMR (125Hz, $CDCl_3$) δ 30.88, 32.91, 44.46, 59.67, 73.82, 126.91, 128.32, 128.65, 140.24, 211.82, 211.82.

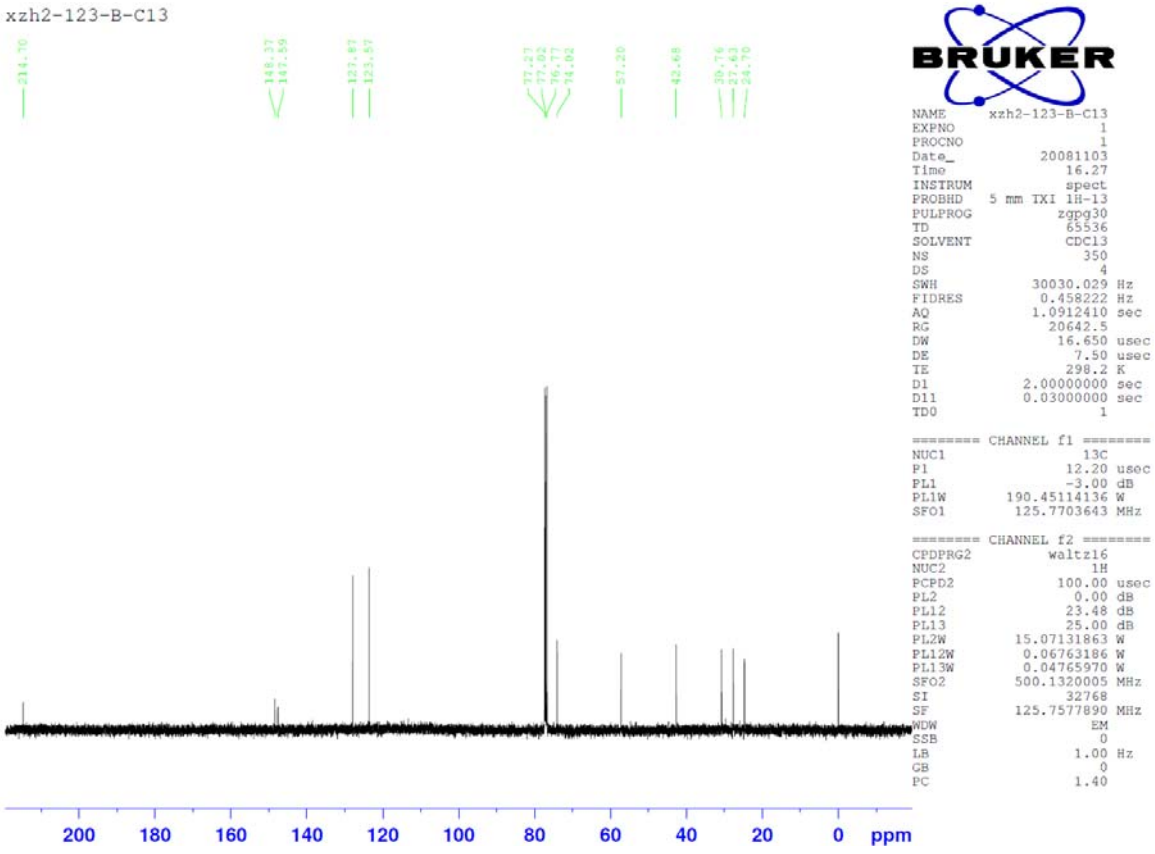


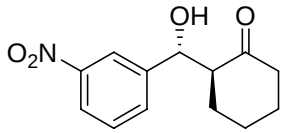
6h

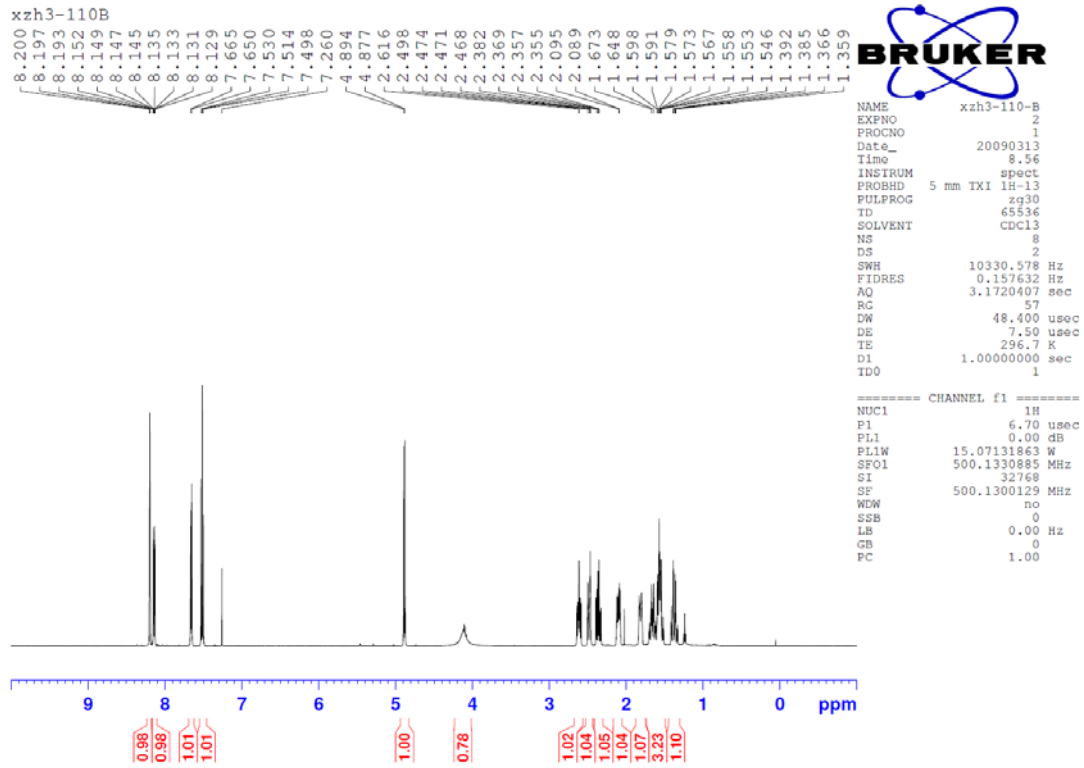
(R)-4-hydroxy-4-(4-nitrophenyl)butan-2-one (**5s**)[2] yield 84% ; Ee 72 % ; $[\alpha]_D^{25}$= 35.2 (c= 0.2, $CHCl_3$) ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.18 (s, 3H), 2.82 (m, 2H), 3.65 (s, 1H), 5.23 (t, *J* = 6 Hz, 1H), 7.50 (d, *J* = 8.5 Hz, 2H), 8.15 (d, *J* = 8.5 Hz, 2H). ^{13}C NMR (125Hz, $CDCl_3$) δ 30.70, 51.52, 68.91, 123.75, 126.43, 147.10, 150.06, 208.44.

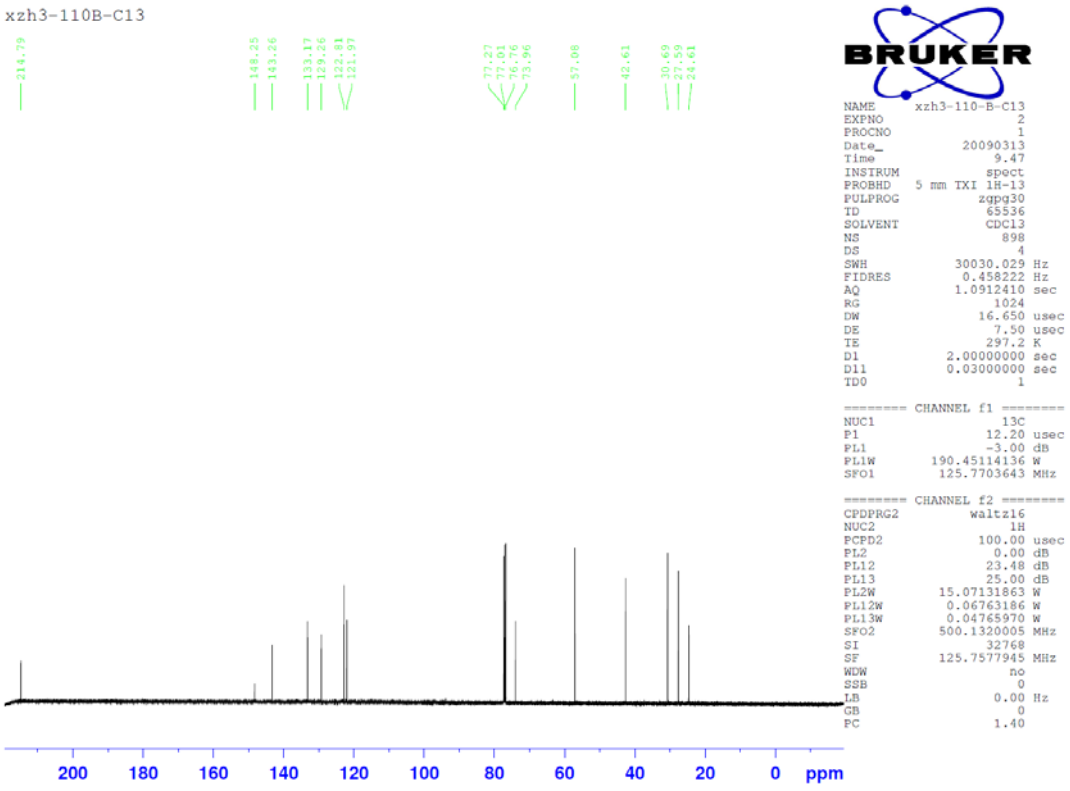
NMR Copy of the aldol products and ligands

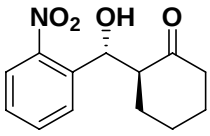


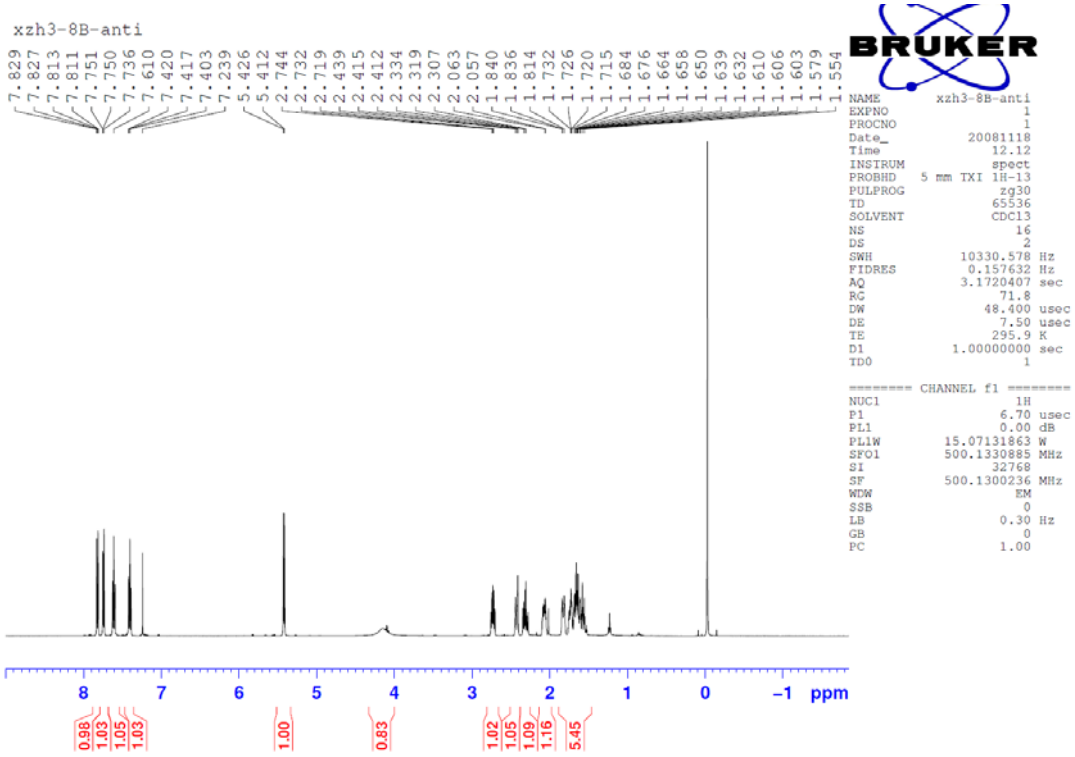



OH
O
O_2N


xzh3-110B
8.200 8.197 8.193 8.152 8.149 8.147 8.145 8.135 8.133 8.131 8.129 7.665 7.650 7.530 7.514 7.498 7.260 4.894 4.877 2.616 2.498 2.474 2.471 2.468 2.382 2.369 2.357 2.355 2.095 2.089 1.673 1.648 1.598 1.591 1.579 1.573 1.567 1.558 1.553 1.546 1.392 1.385 1.366 1.359
BRUKER
NAME xzh3-110-B
EXPNO 2
PROCNO 1
Date_ 20090313
Time 8.56
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 57
DW 48.400 usec
DE 7.50 usec
TE 296.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300129 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
0.98 0.98 1.01 1.01 1.00 0.78 1.02 1.04 1.05 1.04 1.07 3.23 1.10

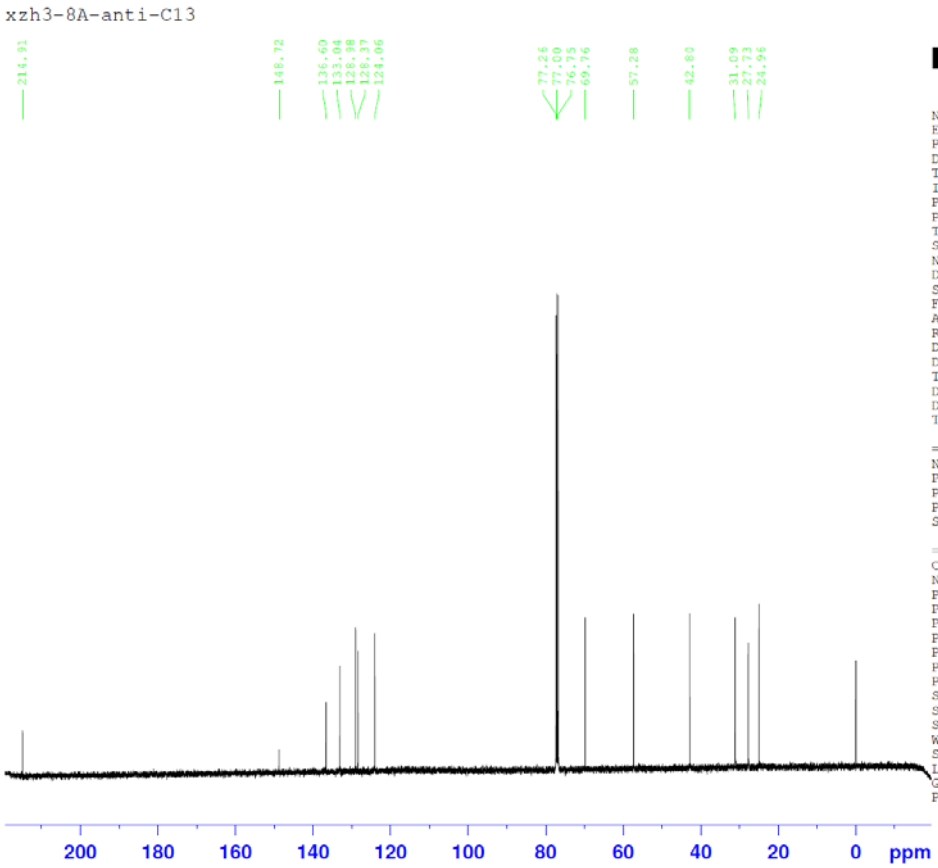

xzh3-110B-C13
214.79 148.25 143.26 133.17 129.26 122.81 121.97 77.27 77.01 76.76 73.96 57.08 42.61 30.69 27.59 24.61
BRUKER
NAME xzh3-110-B-C13
EXPNO 2
PROCNO 1
Date_ 20090313
Time 9.47
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 898
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1024
DW 16.650 usec
DE 7.50 usec
TE 297.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577945 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm





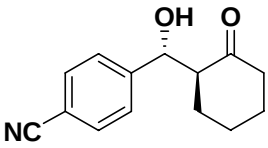
BRUKER

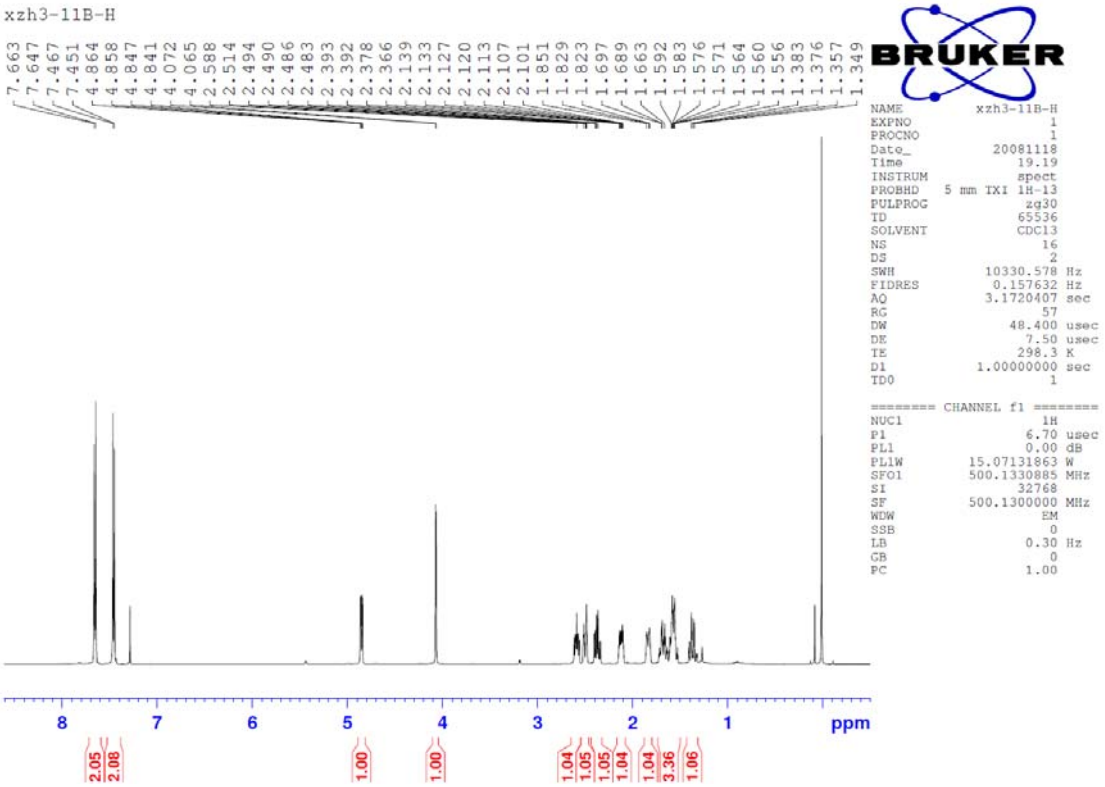
```
NAME          xzh3-8B-anti
EXPNO                    1
PROCNO                   1
Date_             20081118
Time                 12.12
INSTRUM              spect
PROBHD   5 mm TXI 1H-13
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                     16
DS                      2
SWH           10330.578 Hz
FIDRES         0.157632 Hz
AQ             3.1720407 sec
RG                  71.8
DW                48.400 usec
DE                 7.50 usec
TE                295.9 K
D1           1.00000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                   1H
P1                   6.70 usec
PL1                  0.00 dB
PL1W          15.07131863 W
SFO1         500.1330885 MHz
SI                  32768
SF           500.1300236 MHz
WDW                    EM
SSB                     0
LB                   0.30 Hz
GB                      0
PC                   1.00
```

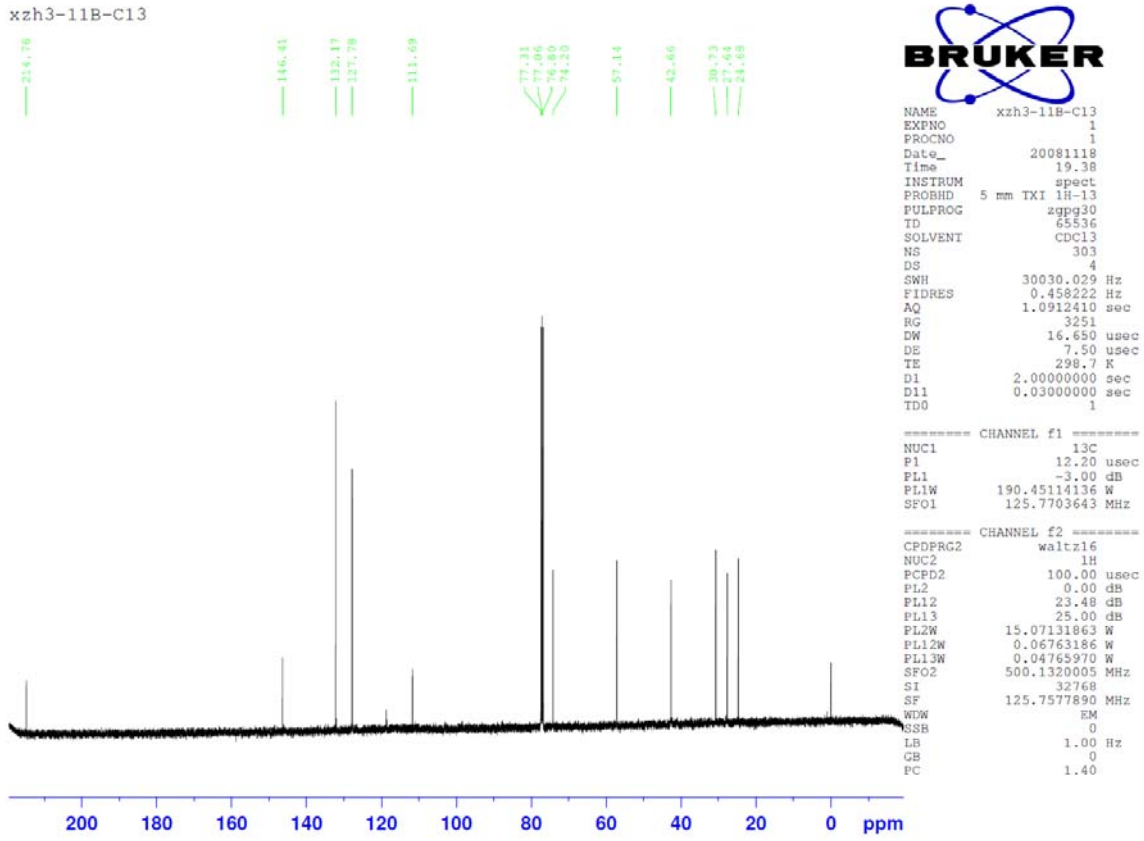


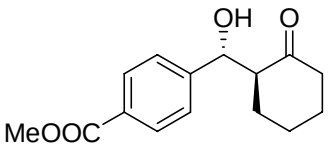
BRUKER

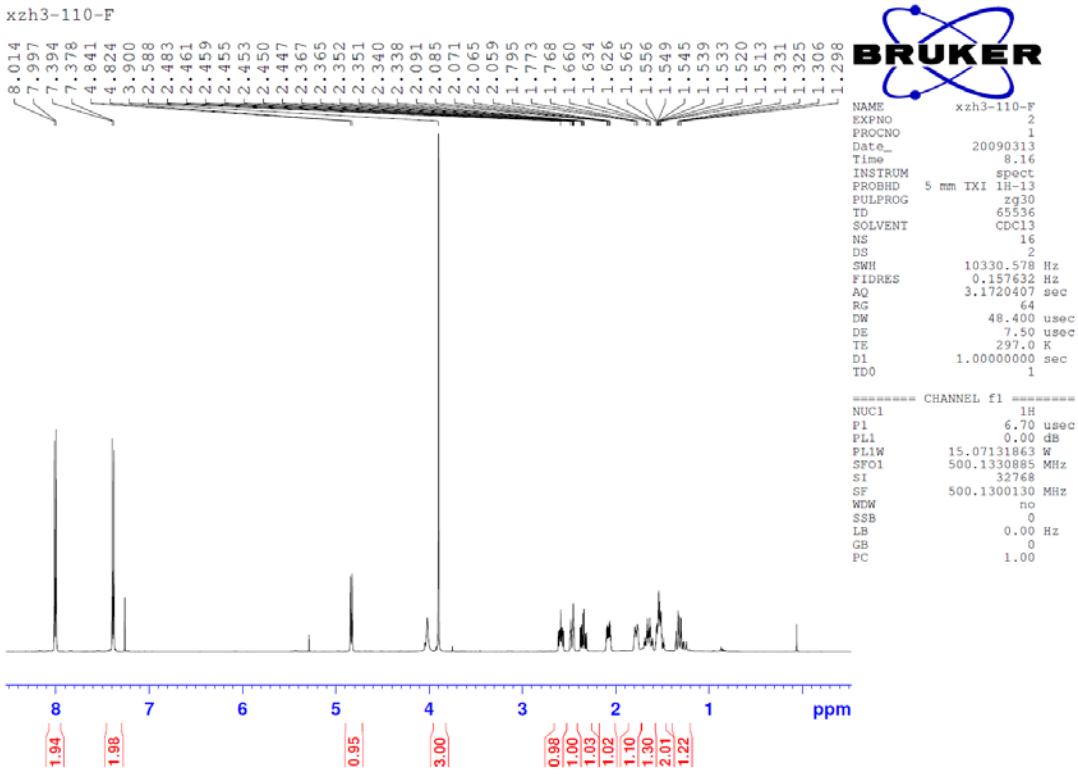
```
NAME          xzh3-8A-anti-c13
EXPNO                    1
PROCNO                   1
Date_             20081118
Time                 13.01
INSTRUM              spect
PROBHD   5 mm TXI 1H-13
PULPROG            zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    840
DS                      4
SWH           30030.029 Hz
FIDRES         0.458222 Hz
AQ             1.0912410 sec
RG                1149.4
DW                16.650 usec
DE                 7.50 usec
TE                296.0 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                     1

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.20 usec
PL1                 -3.00 dB
PL1W         190.45114136 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2              100.00 usec
PL2                  0.00 dB
PL12                23.48 dB
PL13                25.00 dB
PL2W          15.07131863 W
PL12W          0.06763186 W
PL13W          0.04765970 W
SFO2         500.1320005 MHz
SI                  32768
SF           125.7577935 MHz
WDW                    EM
SSB                     0
LB                   1.00 Hz
GB                      0
PC                   1.40
```

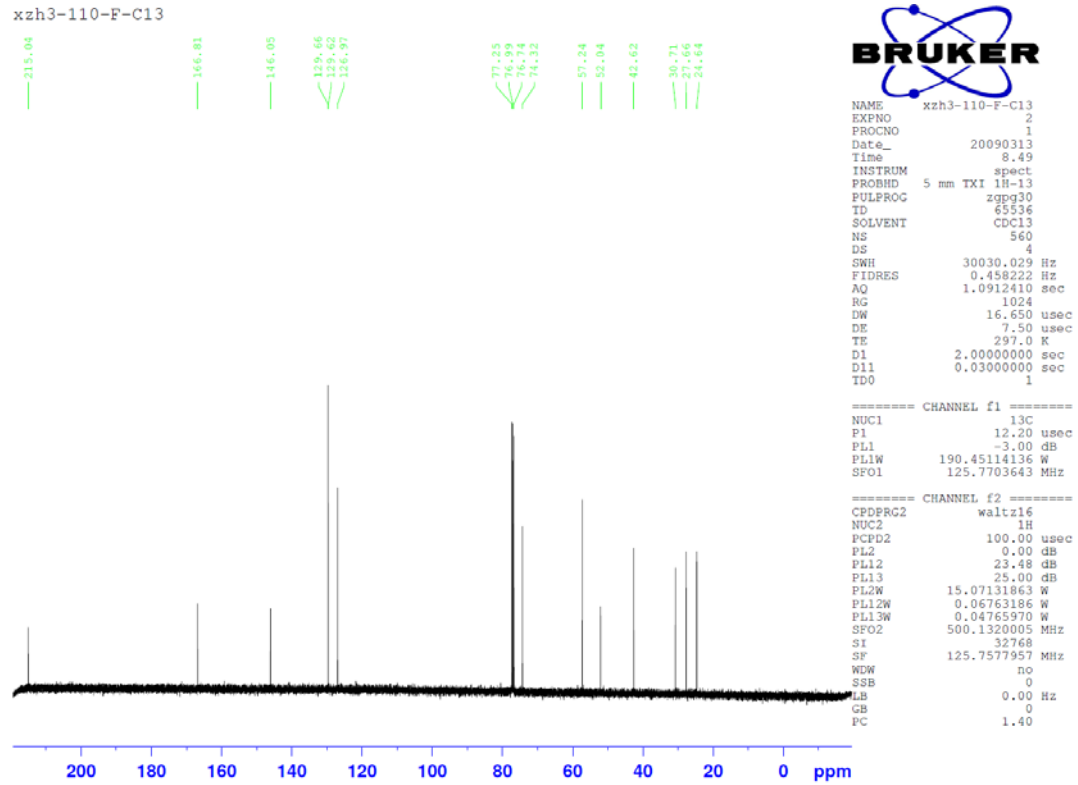

OH
O
NC

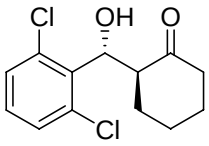

xzh3-11B-H
7.663 7.647 7.467 7.451 4.864 4.858 4.847 4.841 4.072 4.065 2.588 2.514 2.494 2.490 2.486 2.483 2.393 2.392 2.378 2.366 2.139 2.133 2.127 2.120 2.113 2.107 2.101 1.851 1.829 1.823 1.697 1.689 1.663 1.592 1.583 1.576 1.571 1.564 1.560 1.556 1.383 1.376 1.357 1.349
BRUKER
NAME xzh3-11B-H
EXPNO 1
PROCNO 1
Date_ 20081118
Time 19.19
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 57
DW 48.400 usec
DE 7.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8 7 6 5 4 3 2 1 ppm
2.05 2.08 1.00 1.00 1.04 1.05 1.05 1.04 1.04 3.36 1.06

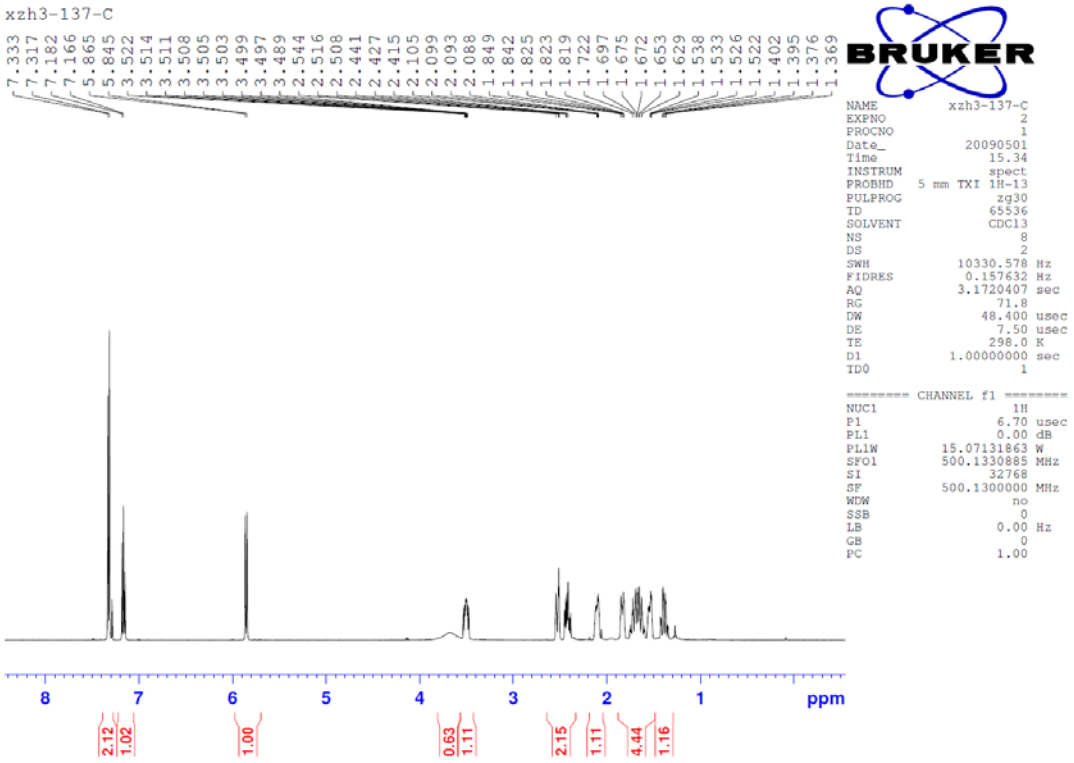

xzh3-11B-C13
214.76 146.41 132.17 127.78 111.69 77.31 77.06 76.80 74.20 57.14 42.46 30.73 27.44 24.68
BRUKER
NAME xzh3-11B-C13
EXPNO 1
PROCNO 1
Date_ 20081118
Time 19.38
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 303
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 3251
DW 16.650 usec
DE 7.50 usec
TE 298.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

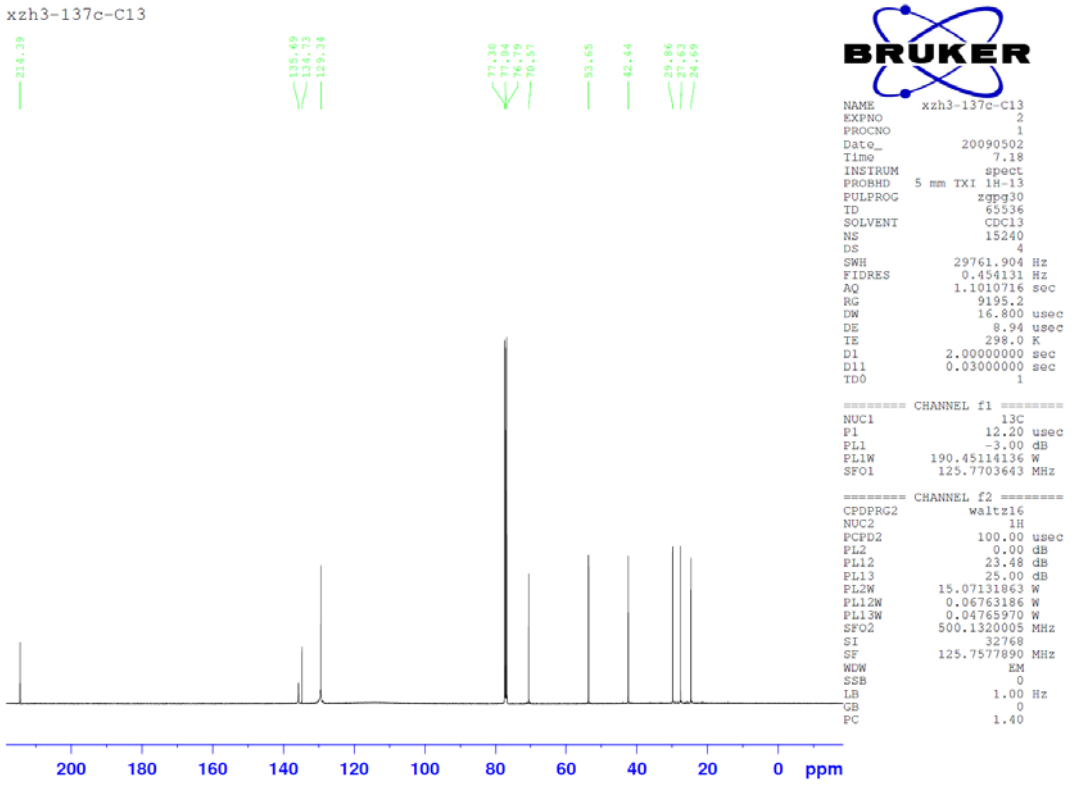

OH
O
MeOOC

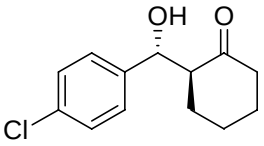

xzh3-110-F
8.014
7.997
7.394
7.378
4.841
4.824
3.900
2.588
2.483
2.461
2.459
2.455
2.453
2.450
2.447
2.367
2.365
2.352
2.351
2.340
2.338
2.091
2.085
2.071
2.065
2.059
1.795
1.773
1.768
1.660
1.634
1.626
1.565
1.556
1.549
1.545
1.539
1.533
1.520
1.513
1.331
1.325
1.306
1.298
BRUKER
NAME xzh3-110-F
EXPNO 2
PROCNO 1
Date_ 20090313
Time 8.16
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 7.50 usec
TE 297.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300130 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
ppm
1.94
1.98
0.95
3.00
0.98
1.00
1.03
1.02
1.10
1.30
2.01
1.22


xzh3-110-F-C13
215.04
166.81
146.05
129.66
129.62
126.97
77.25
76.99
76.74
74.12
57.24
52.04
42.62
30.71
27.66
24.64
BRUKER
NAME xzh3-110-F-C13
EXPNO 2
PROCNO 1
Date_ 20090313
Time 8.49
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 560
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1024
DW 16.650 usec
DE 7.50 usec
TE 297.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577957 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

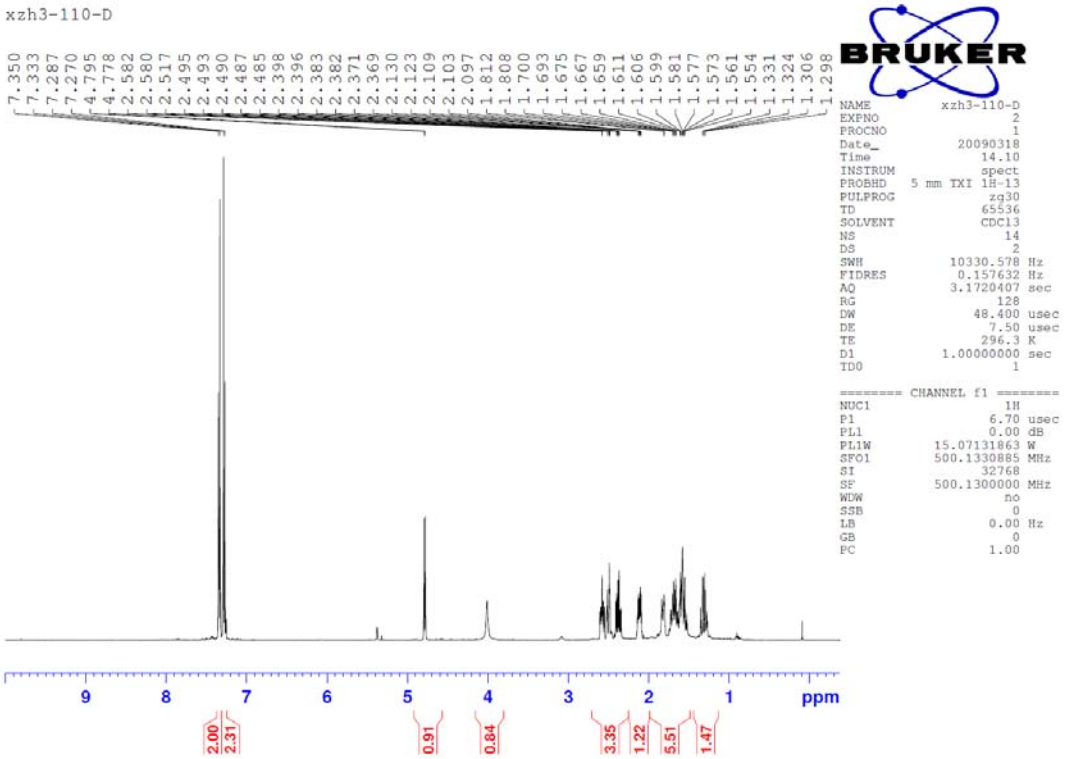

Cl
OH
O
Cl


xzh3-137-C
7.333
7.317
7.182
7.166
5.865
5.845
3.522
3.514
3.511
3.508
3.505
3.503
3.499
3.497
3.489
2.544
2.516
2.508
2.441
2.427
2.415
2.105
2.099
2.093
2.088
1.849
1.842
1.825
1.823
1.819
1.722
1.697
1.675
1.672
1.653
1.629
1.538
1.533
1.526
1.522
1.402
1.395
1.376
1.369
BRUKER
NAME xzh3-137-C
EXPNO 2
PROCNO 1
Date_ 20090501
Time 15.34
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 71.8
DW 48.400 usec
DE 7.50 usec
TE 298.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
ppm
2.12
1.02
1.00
0.63
1.11
2.15
1.11
4.44
1.16

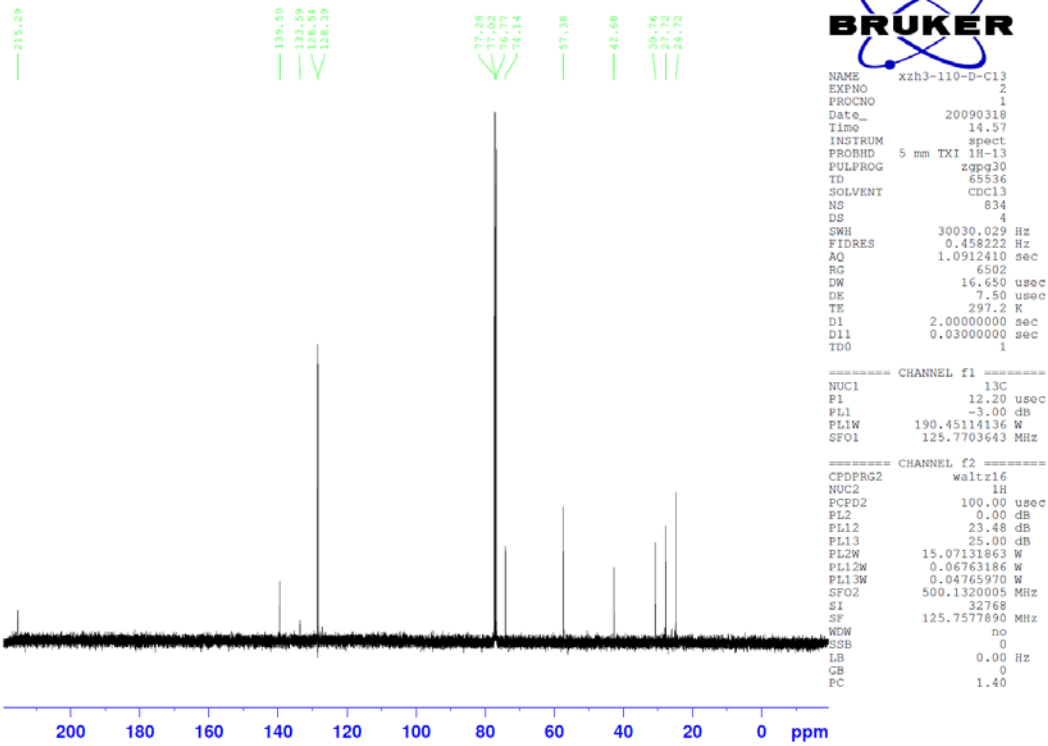

xzh3-137c-C13
214.39
135.69
134.73
129.34
77.30
77.04
76.79
70.57
53.65
42.44
29.86
27.63
24.69
BRUKER
NAME xzh3-137c-C13
EXPNO 2
PROCNO 1
Date_ 20090502
Time 7.18
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 15240
DS 4
SWH 29761.904 Hz
FIDRES 0.454131 Hz
AQ 1.1010716 sec
RG 9195.2
DW 16.800 usec
DE 8.94 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

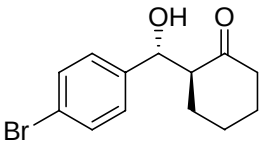


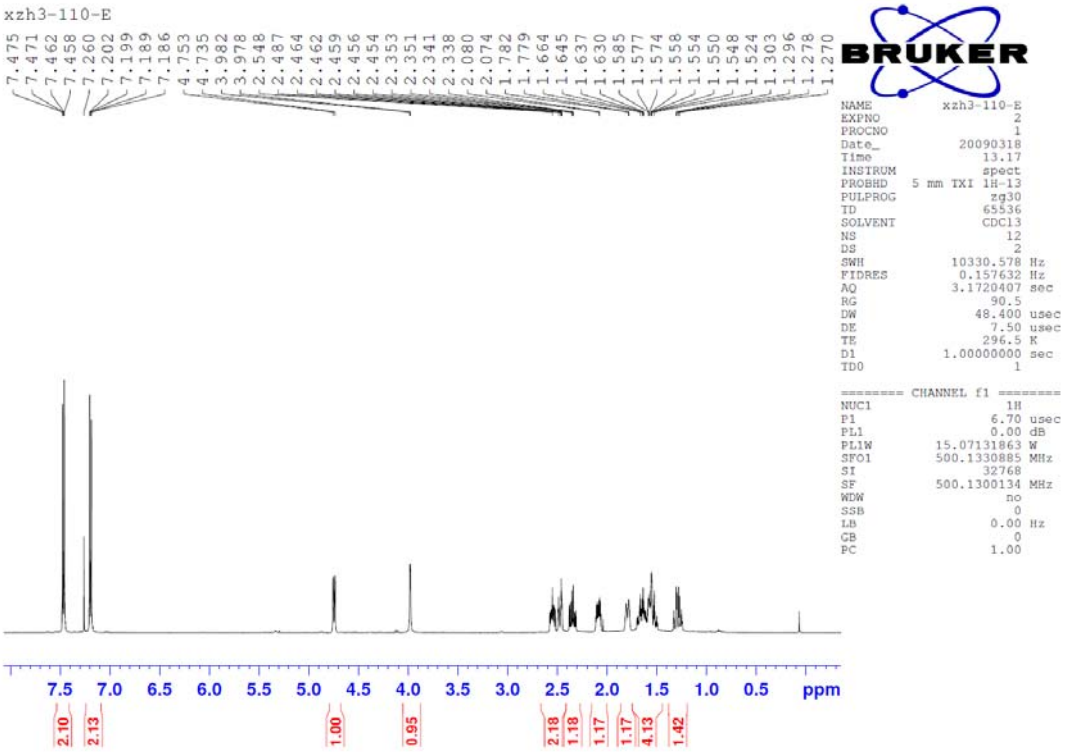
xzh3-110-D

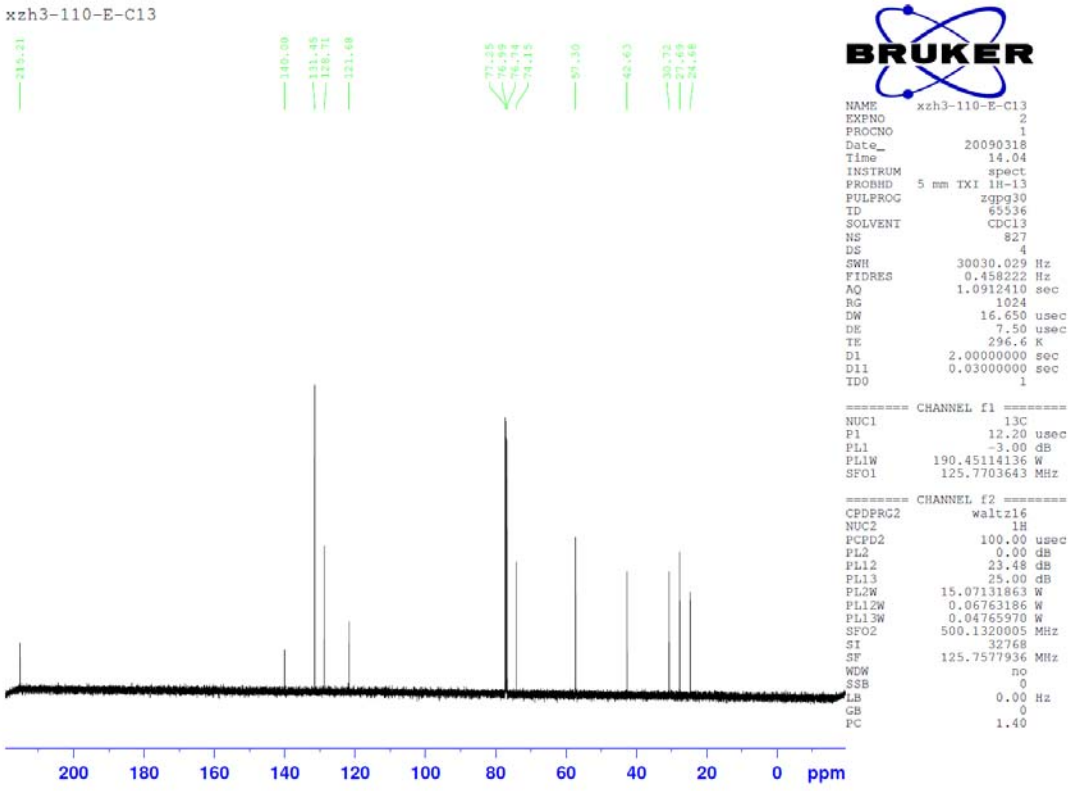


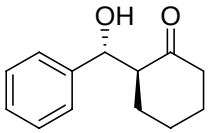
xzh3-110-D-C13

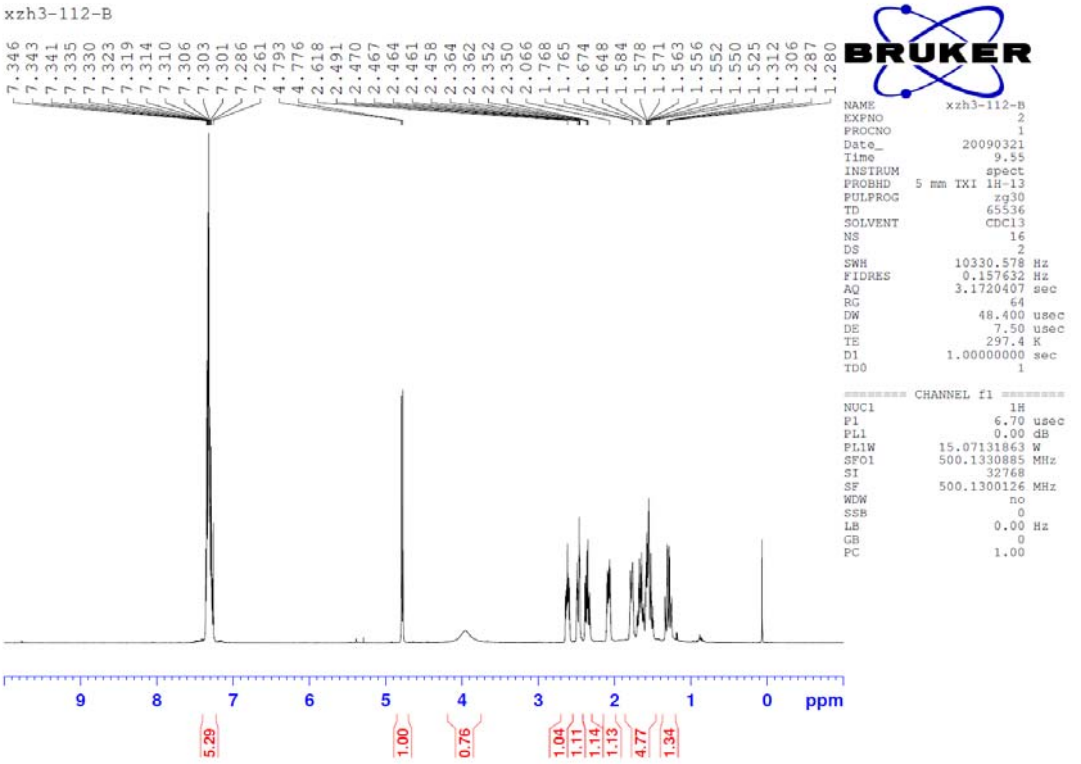


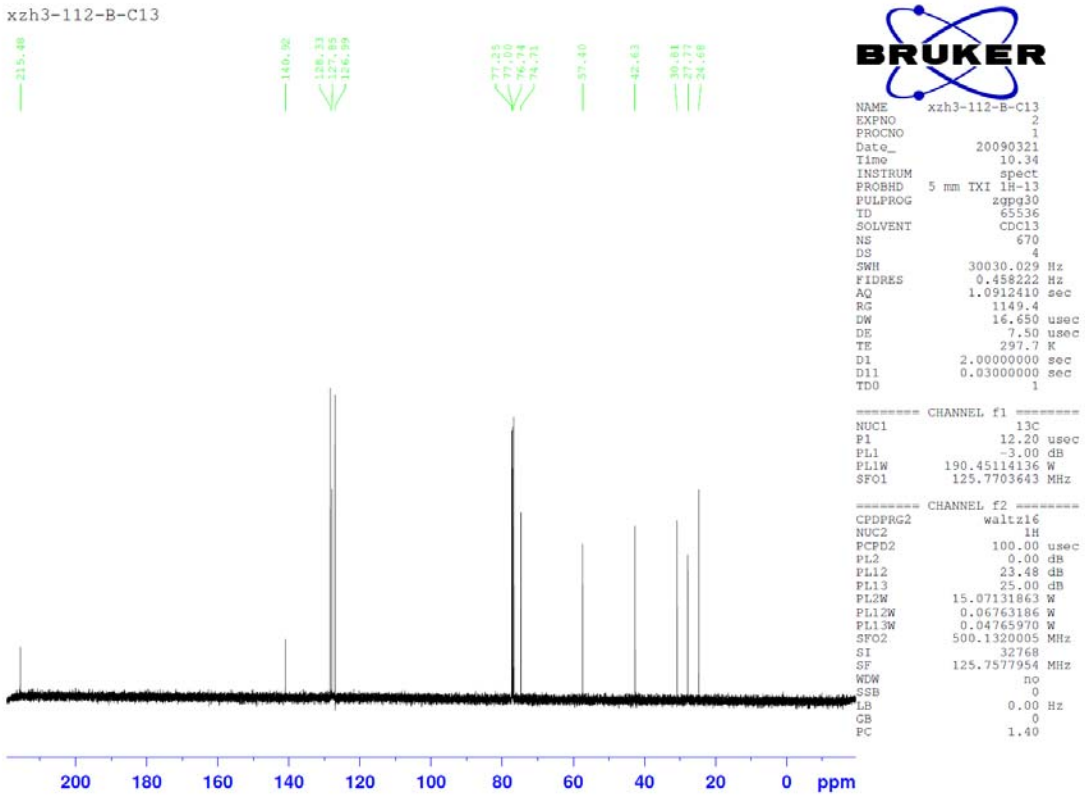

OH
O
Br

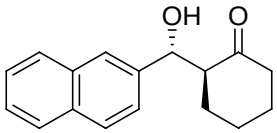

xzh3-110-E
7.475 7.471 7.462 7.458 7.260 7.202 7.199 7.189 7.186 4.753 4.735 3.982 3.978 2.548 2.487 2.464 2.462 2.459 2.456 2.454 2.353 2.351 2.341 2.338 2.080 2.074 1.782 1.779 1.664 1.645 1.637 1.630 1.585 1.577 1.574 1.558 1.554 1.550 1.548 1.524 1.303 1.296 1.278 1.270
BRUKER
NAME xzh3-110-E
EXPNO 2
PROCNO 1
Date_ 20090318
Time 13.17
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 12
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 90.5
DW 48.400 usec
DE 7.50 usec
TE 296.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300134 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.10 2.13 1.00 0.95 2.18 1.18 1.17 1.17 4.13 1.42

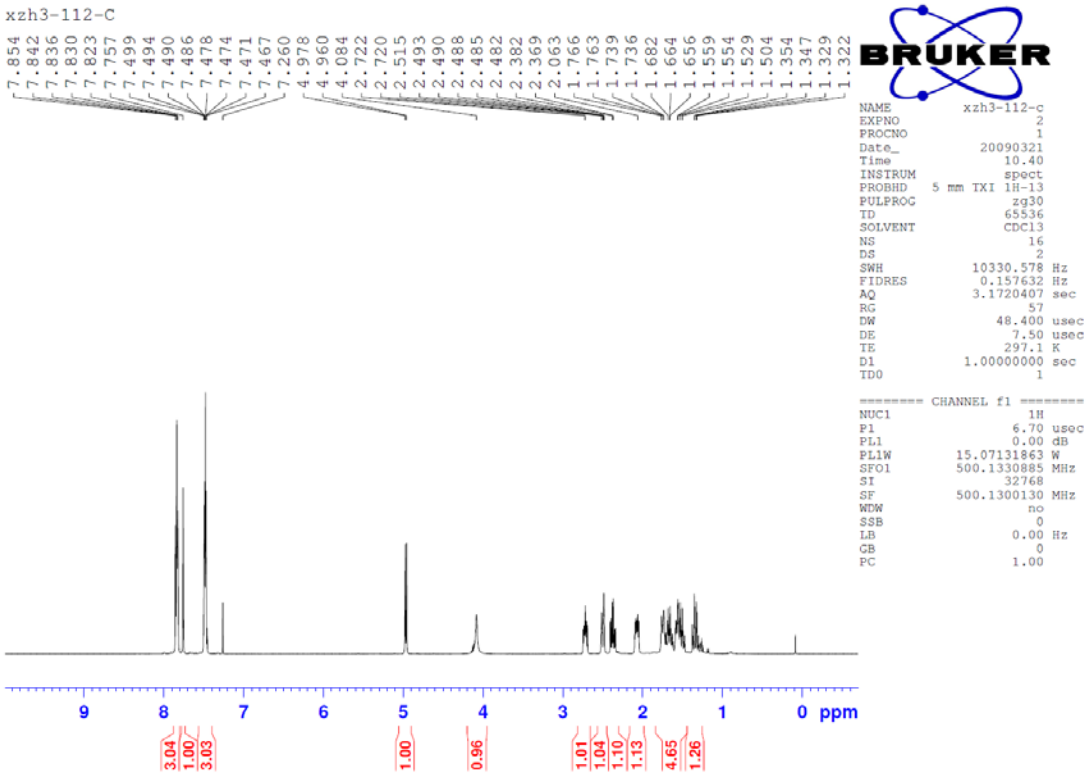

xzh3-110-E-C13
215.21 140.09 131.45 128.71 121.68 77.25 76.94 76.74 74.13 57.30 43.63 30.72 27.69 24.68
BRUKER
NAME xzh3-110-E-C13
EXPNO 2
PROCNO 1
Date_ 20090318
Time 14.04
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 827
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1024
DW 16.650 usec
DE 7.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577936 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

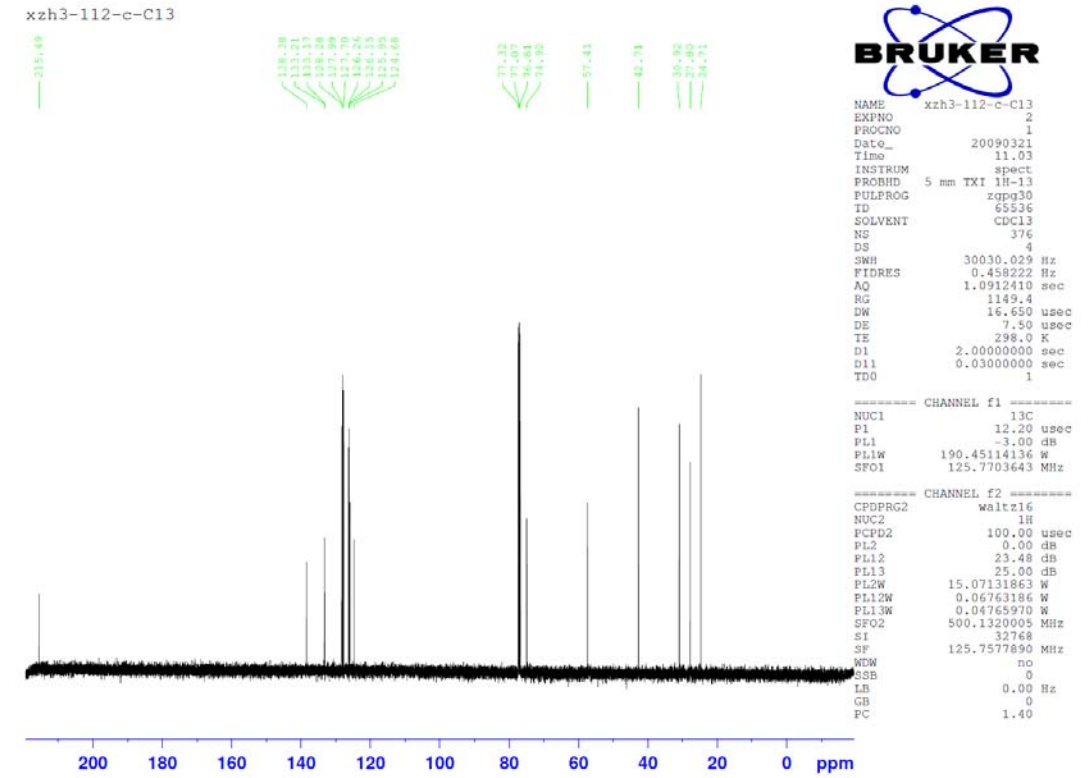

OH
O

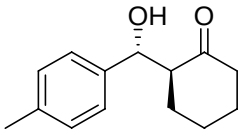

xzh3-112-B
7.346
7.343
7.341
7.335
7.330
7.323
7.319
7.314
7.310
7.306
7.303
7.301
7.286
7.261
4.793
4.776
2.618
2.491
2.470
2.467
2.464
2.461
2.458
2.364
2.362
2.352
2.350
2.066
1.768
1.765
1.674
1.648
1.584
1.578
1.571
1.563
1.556
1.552
1.550
1.525
1.312
1.306
1.287
1.280
BRUKER
NAME xzh3-112-B
EXPNO 2
PROCNO 1
Date_ 20090321
Time 9.55
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 7.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300126 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9
8
7
6
5
4
3
2
1
0
ppm
5.29
1.00
0.76
1.04
1.11
1.14
1.13
4.77
1.34

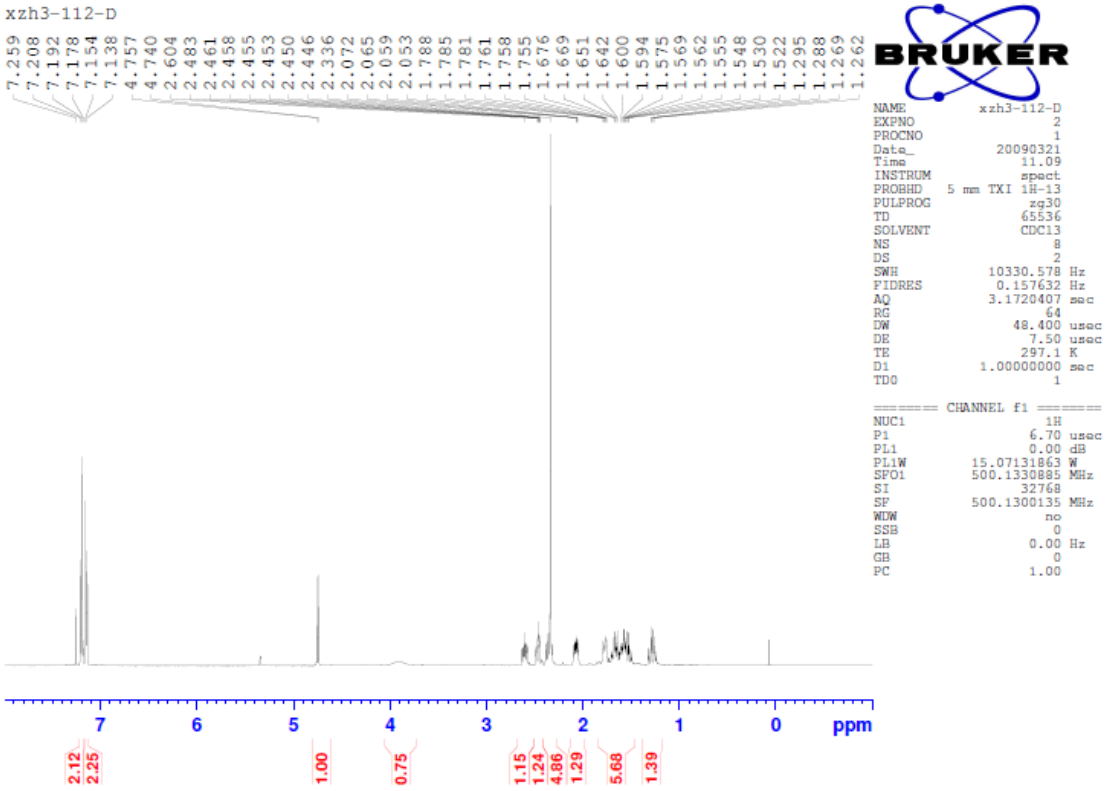

xzh3-112-B-C13
215.48
140.92
128.33
127.85
126.89
77.25
77.00
76.74
74.71
57.40
42.63
30.81
27.77
24.68
BRUKER
NAME xzh3-112-B-C13
EXPNO 2
PROCNO 1
Date_ 20090321
Time 10.34
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 670
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1149.4
DW 16.650 usec
DE 7.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577954 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

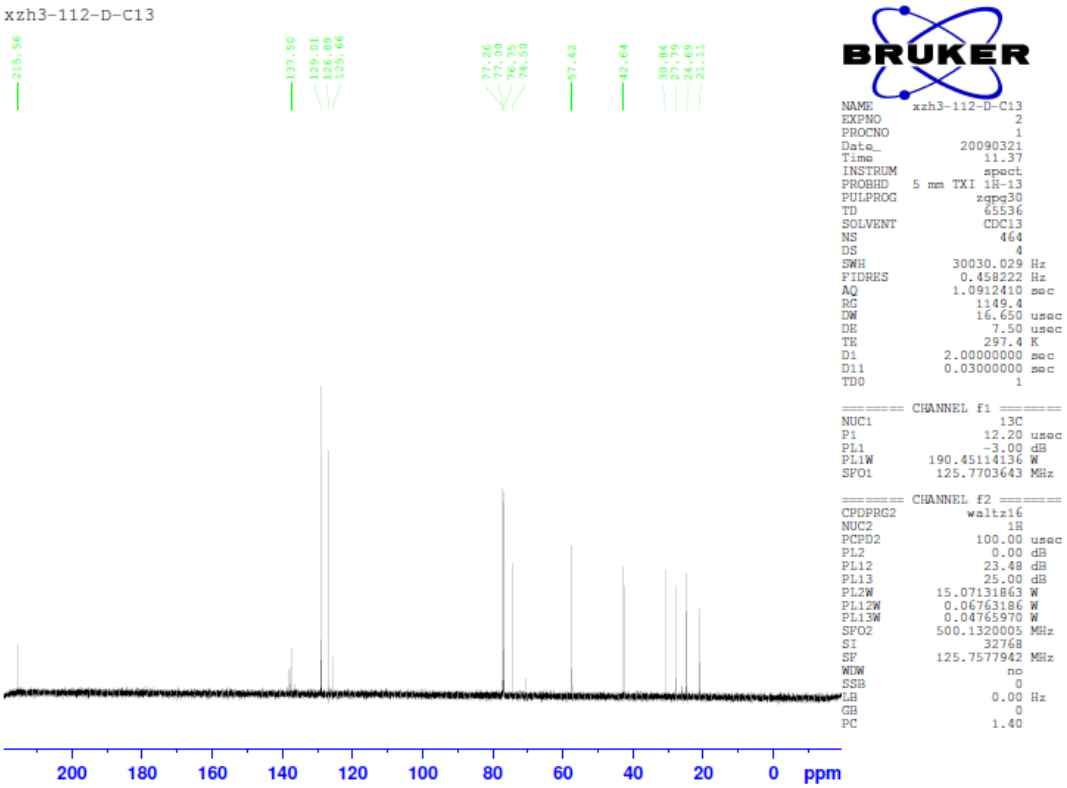

OH
O

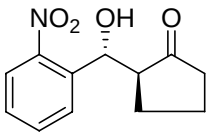

xzh3-112-C
7.854 7.842 7.836 7.830 7.823 7.757 7.499 7.494 7.490 7.486 7.478 7.474 7.471 7.467 7.260 4.978 4.960 4.084 2.722 2.720 2.515 2.493 2.490 2.488 2.485 2.482 2.382 2.369 2.063 1.766 1.763 1.739 1.736 1.682 1.664 1.656 1.559 1.554 1.529 1.504 1.354 1.347 1.329 1.322
BRUKER
NAME xzh3-112-c
EXPNO 2
PROCNO 1
Date_ 20090321
Time 10.40
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 57
DW 48.400 usec
DE 7.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300130 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
3.04 1.00 3.03 1.00 0.96 1.01 1.04 1.10 1.13 4.65 1.26


xzh3-112-c-C13
BRUKER
NAME xzh3-112-c-C13
EXPNO 2
PROCNO 1
Date_ 20090321
Time 11.03
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 376
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1149.4
DW 16.650 usec
DE 7.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm

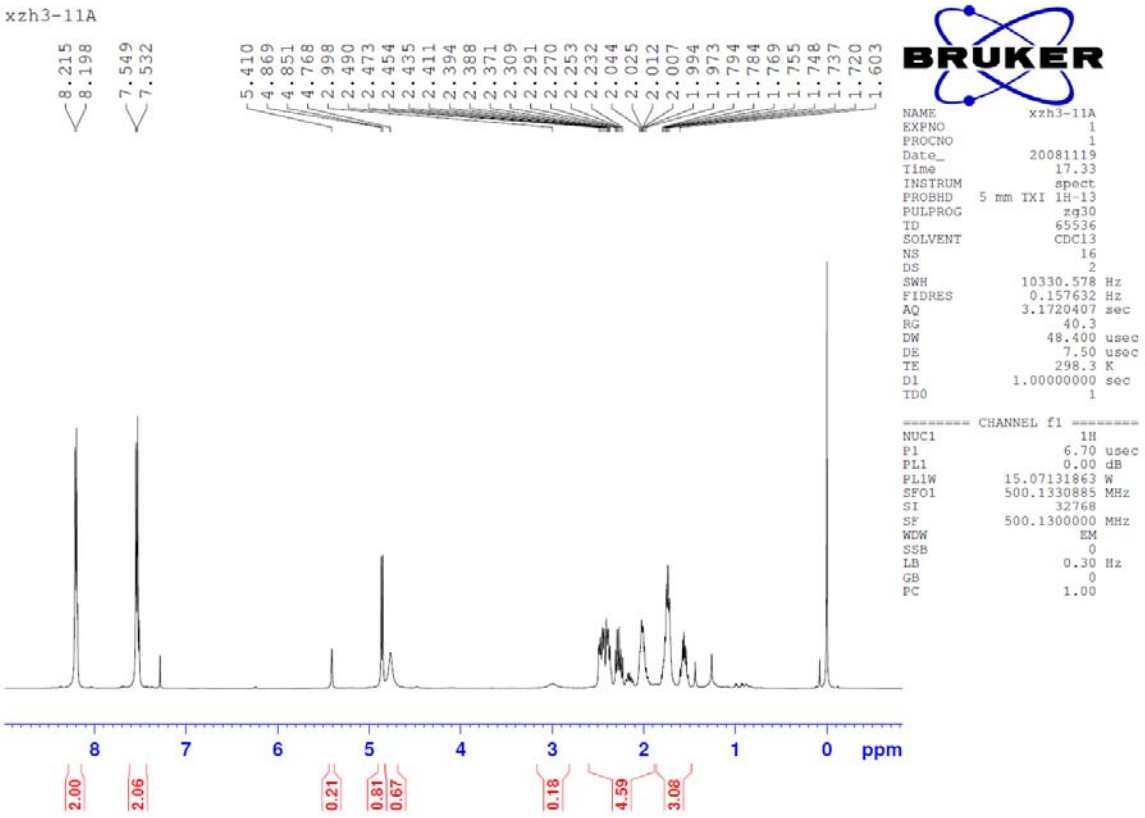

OH
O

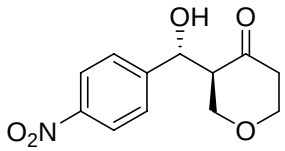

xzh3-112-D
7.259
7.208
7.192
7.178
7.154
7.138
4.757
4.740
2.604
2.483
2.461
2.458
2.455
2.453
2.450
2.446
2.336
2.072
2.065
2.059
2.053
1.788
1.785
1.781
1.761
1.758
1.755
1.676
1.669
1.651
1.642
1.600
1.594
1.575
1.569
1.562
1.555
1.548
1.530
1.522
1.295
1.288
1.269
1.262
BRUKER
NAME xzh3-112-D
EXPNO 2
PROCNO 1
Date_ 20090321
Time 11.09
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 64
DW 48.400 usec
DE 7.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
====== CHANNEL f1 ======
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300135 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
7
6
5
4
3
2
1
0
ppm
2.12
2.25
1.00
0.75
1.15
1.24
4.86
1.29
5.68
1.39

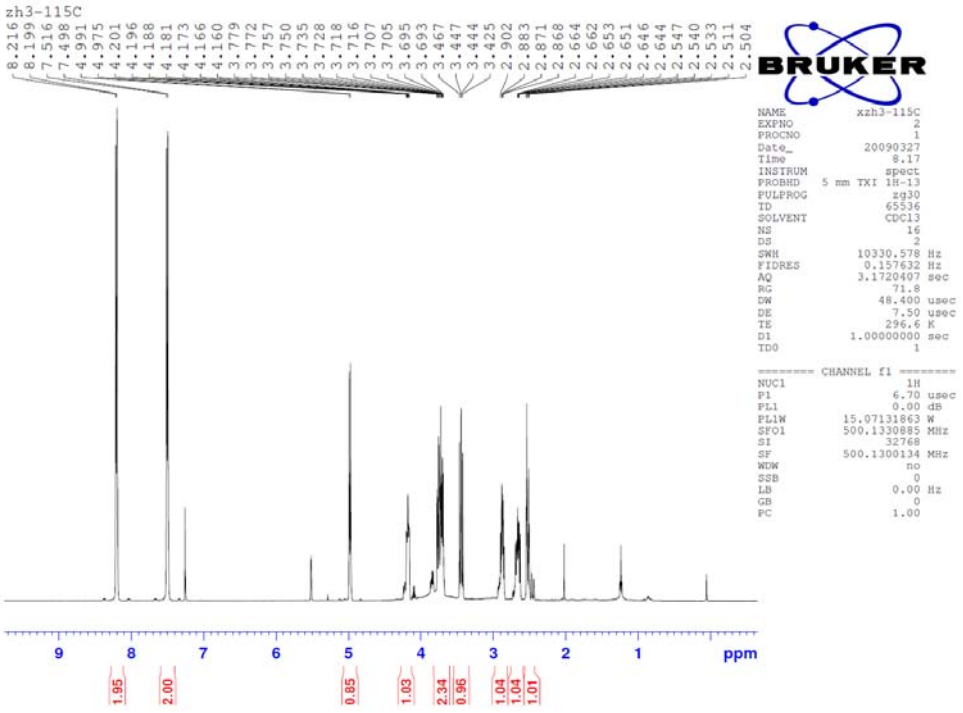

xzh3-112-D-C13
215.56
137.50
129.01
126.89
125.66
77.26
77.00
76.75
74.50
57.42
42.64
39.84
27.79
24.69
21.11
BRUKER
NAME xzh3-112-D-C13
EXPNO 2
PROCNO 1
Date_ 20090321
Time 11.37
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 464
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1149.4
DW 16.650 usec
DE 7.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
====== CHANNEL f1 ======
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
====== CHANNEL f2 ======
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577942 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
0
ppm

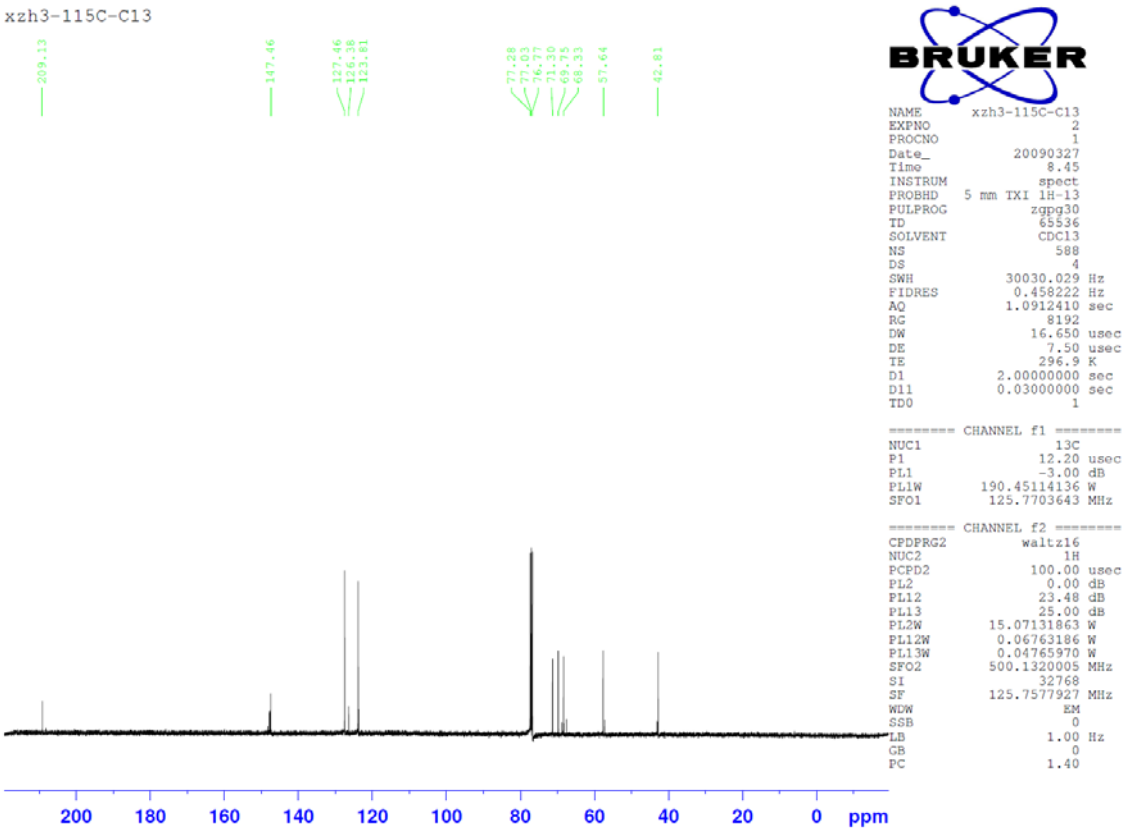

NO2
OH
O

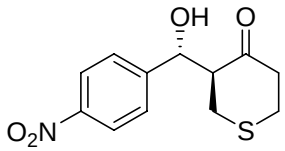
anti/syn = 3/1

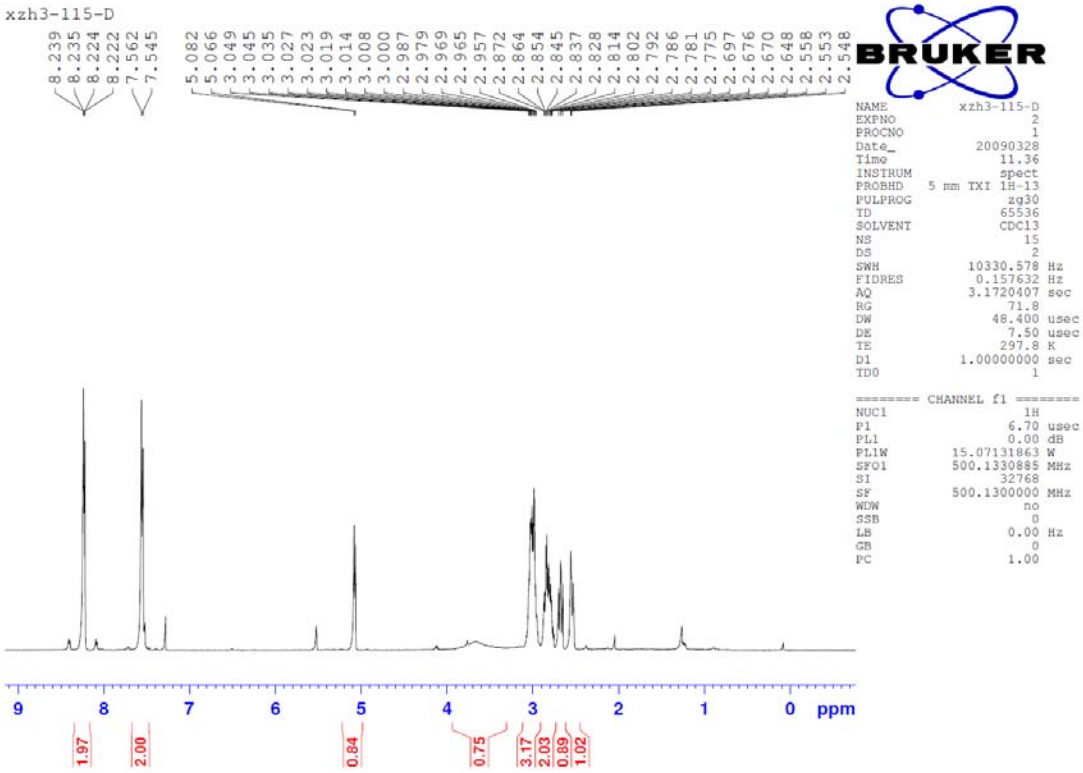

xzh3-11A
8.215
8.198
7.549
7.532
5.410
4.869
4.851
4.768
2.998
2.490
2.473
2.454
2.435
2.411
2.394
2.388
2.371
2.309
2.291
2.270
2.253
2.232
2.044
2.025
2.012
2.007
1.994
1.973
1.794
1.784
1.769
1.755
1.748
1.737
1.720
1.603
BRUKER
NAME xzh3-11A
EXPNO 1
PROCNO 1
Date_ 20081119
Time 17.33
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 40.3
DW 48.400 usec
DE 7.50 usec
TE 298.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8
7
6
5
4
3
2
1
0
ppm
2.00
2.06
0.21
0.81
0.67
0.18
4.59
3.08

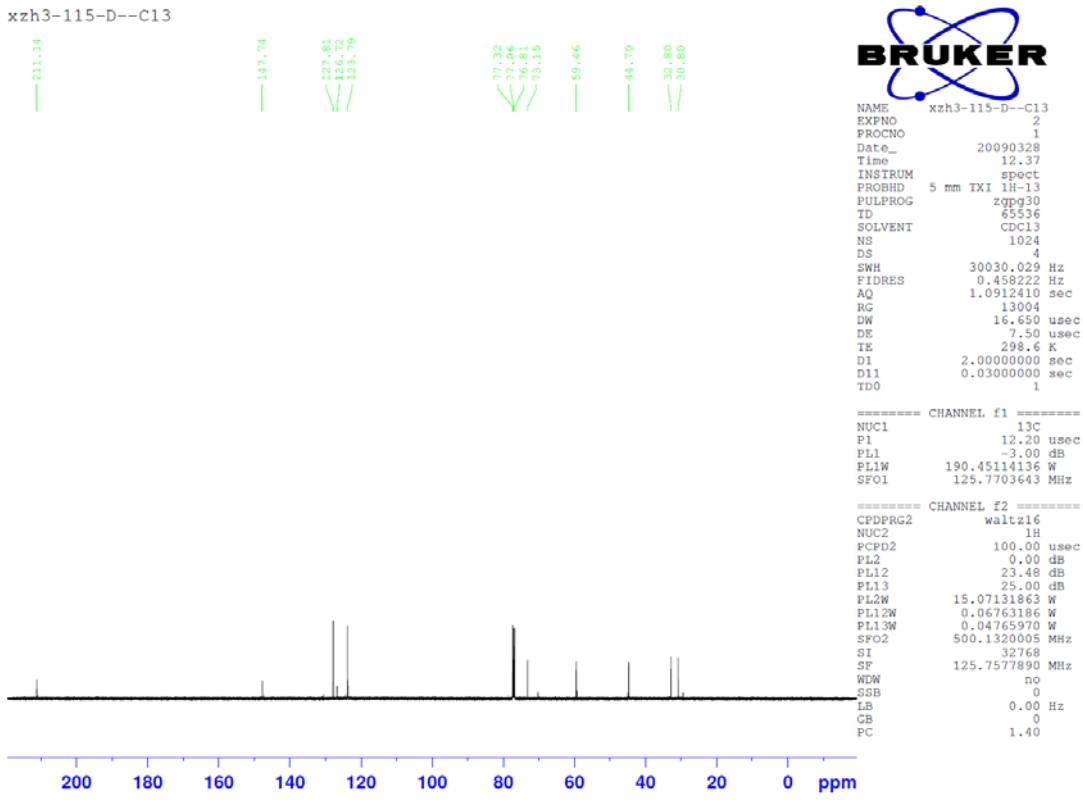

OH
O
O_2N
O

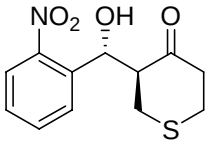

zh3-115C
8.216 8.199 7.516 7.498 4.991 4.975 4.201 4.196 4.188 4.181 4.173 4.166 4.160 3.779 3.772 3.757 3.750 3.735 3.728 3.718 3.716 3.707 3.705 3.695 3.693 3.467 3.447 3.444 3.425 2.902 2.883 2.871 2.868 2.664 2.662 2.653 2.651 2.646 2.644 2.547 2.540 2.533 2.511 2.504
BRUKER
NAME xzh3-115C
EXPNO 2
PROCNO 1
Date_ 20090327
Time 8.17
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 71.8
DW 48.400 usec
DE 7.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300134 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 ppm
1.95 2.00 0.85 1.03 2.34 0.96 1.04 1.04 1.01


xzh3-115C-C13
209.13 147.46 127.46 126.38 123.81 77.28 77.03 76.77 71.30 69.75 68.33 57.64 42.81
BRUKER
NAME xzh3-115C-C13
EXPNO 2
PROCNO 1
Date_ 20090327
Time 8.45
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 588
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 7.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577927 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm


OH
O
O_2N
S


xzh3-115-D
8.239 8.235 8.224 8.222 7.562 7.545
5.082 5.066 3.049 3.045 3.035 3.027 3.023 3.019 3.014 3.008 3.000 2.987 2.979 2.969 2.965 2.957 2.872 2.864 2.854 2.845 2.837 2.828 2.814 2.802 2.792 2.786 2.781 2.775 2.697 2.676 2.670 2.648 2.558 2.553 2.548
BRUKER
NAME xzh3-115-D
EXPNO 2
PROCNO 1
Date_ 20090328
Time 11.36
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 15
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 71.8
DW 48.400 usec
DE 7.50 usec
TE 297.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
9 8 7 6 5 4 3 2 1 0 ppm
1.97 2.00 0.84 0.75 3.17 2.03 0.89 1.02

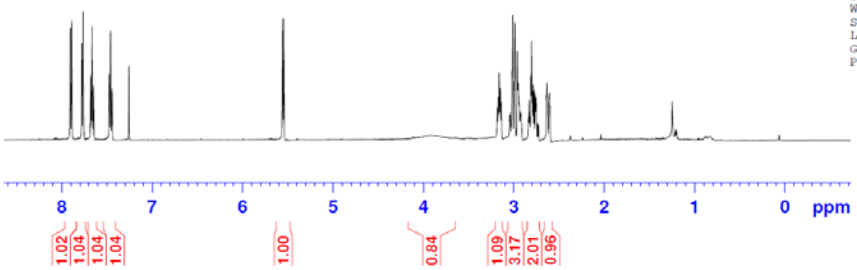

xzh3-115-D--C13
211.14
147.74
127.81
126.72
123.79
77.32
77.06
76.81
73.15
59.46
44.70
32.80
30.80
BRUKER
NAME xzh3-115-D--C13
EXPNO 2
PROCNO 1
Date_ 20090328
Time 12.37
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 1024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 13004
DW 16.650 usec
DE 7.50 usec
TE 298.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



xzh3-150A-anti

7.892 7.780 7.764 7.681 7.666 7.651 7.477 7.462 7.446 7.260

5.559 5.545 3.184 3.172 3.162 3.149 3.139 3.014 2.998 2.990 2.987 2.963 2.955 2.950 2.940 2.831 2.812 2.804 2.796 2.787 2.776 2.764 2.754 2.641 2.633 2.628 2.614 2.606 2.600

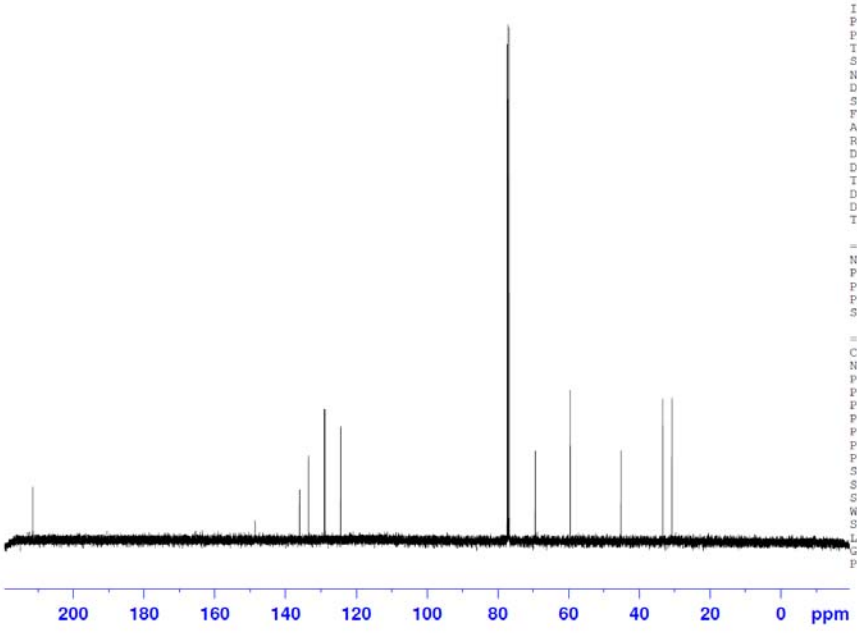


BRUKER

NAME	xzh3-150A-anti
EXPNO	2
PROCNO	1
Date_	20090512
Time	11.13
INSTRUM	spect
PROBHD	5 mm TXI 1H-13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	128
DW	48.400 usec
DE	7.50 usec
TE	296.2 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	6.70 usec
PL1	0.00 dB
PL1W	15.07131863 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300137 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

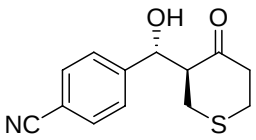
xzh3-150A-anti-C13

211.46 148.59 136.08 133.48 129.01 128.85 124.39 69.37 59.55 45.16 33.26 30.78

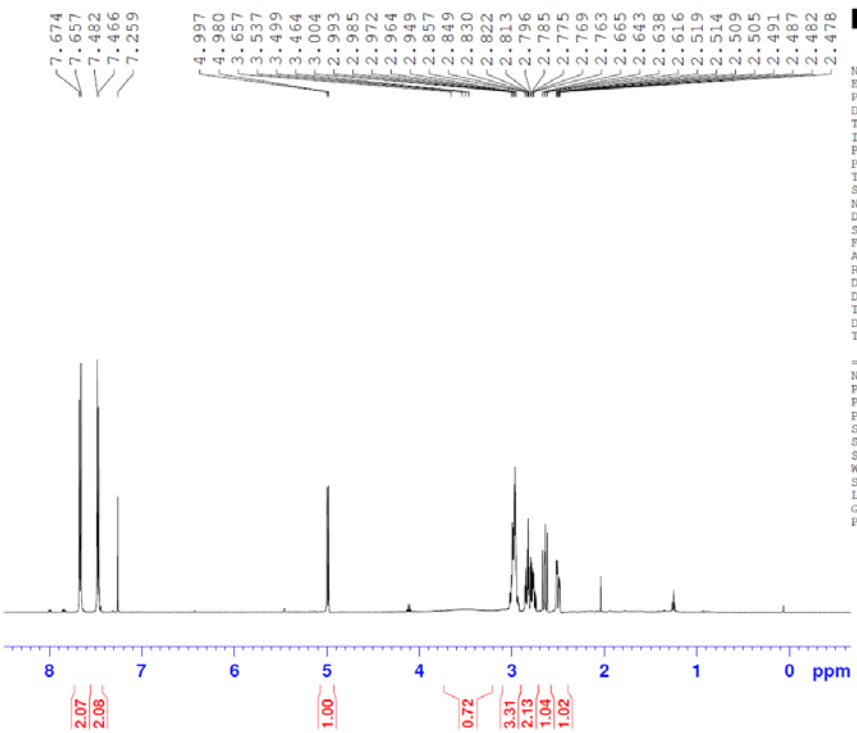


BRUKER

NAME	xzh3-150A-anti-C13
EXPNO	2
PROCNO	1
Date_	20090512
Time	12.20
INSTRUM	spect
PROBHD	5 mm TXI 1H-13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1195
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	1290.2
DW	16.650 usec
DE	7.50 usec
TE	296.9 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.20 usec
PL1	-3.00 dB
PL1W	190.45114136 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	23.48 dB
PL13	25.00 dB
PL2W	15.07131863 W
PL12W	0.06763186 W
PL13W	0.04765970 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577890 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40

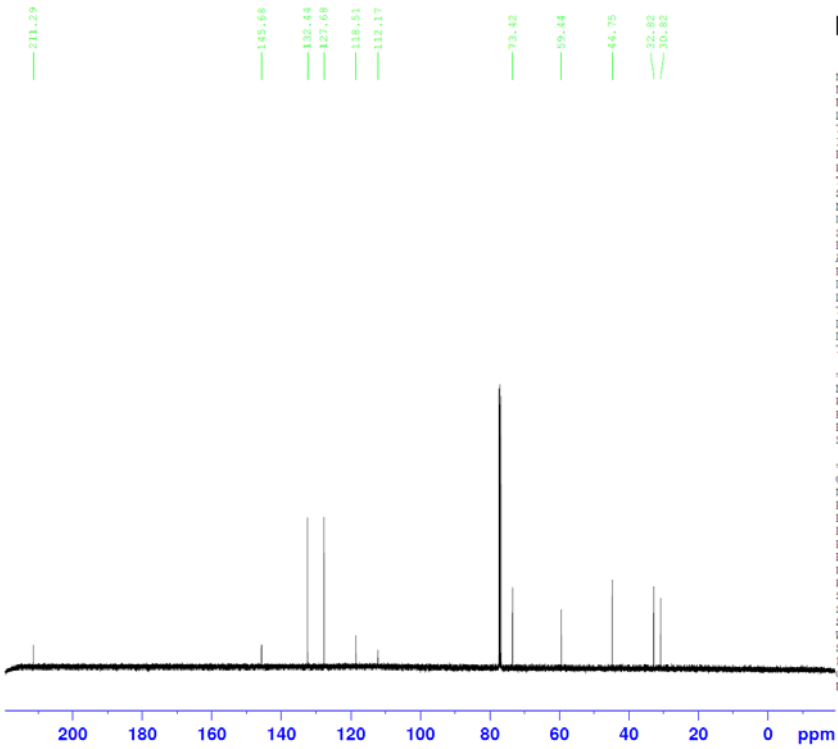


xzh3-150B-H

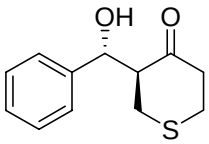


```
NAME                xzh3-150B-H
EXPNO                         2
PROCNO                        1
Date_                  20090512
Time                      12.26
INSTRUM                   spect
PROBHD    5 mm TXI 1H-13
PULPROG                    zg30
TD                        65536
SOLVENT                   CDCl3
NS                            8
DS                            2
SWH                10330.578 Hz
FIDRES             0.157632 Hz
AQ                 3.1720407 sec
RG                       161.3
DW                    48.400 usec
DE                     7.50 usec
TE                     296.4 K
D1                 1.00000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                        1H
P1                       6.70 usec
PL1                      0.00 dB
PL1W              15.07131863 W
SFO1             500.1330885 MHz
SI                        32768
SF               500.1300136 MHz
WDW                          no
SSB                           0
LB                       0.00 Hz
GB                            0
PC                       1.00
```

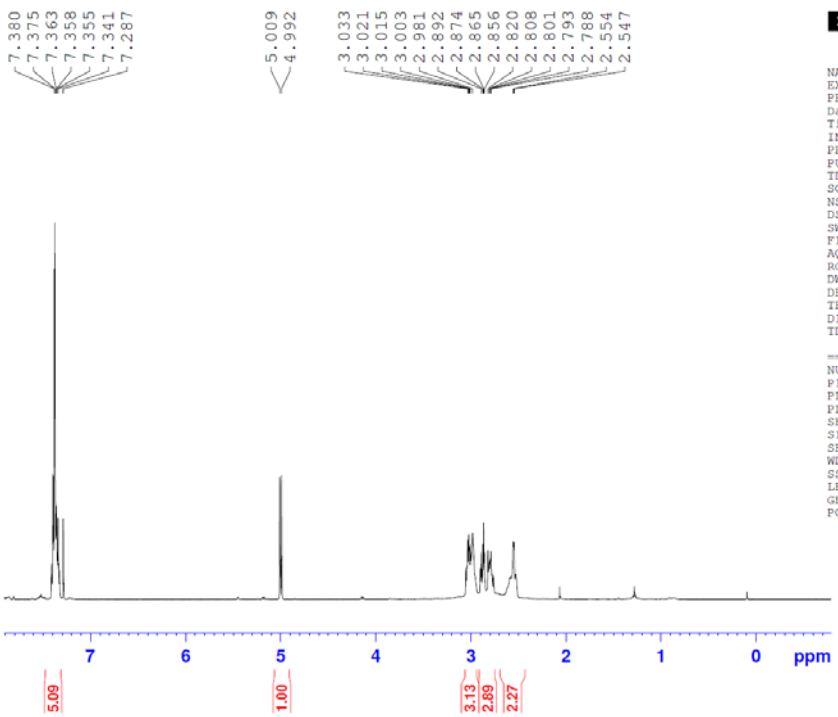
xzh3-150B-c13



```
NAME              xzh3-150B-c13
EXPNO                         2
PROCNO                        1
Date_                  20090512
Time                      14.00
INSTRUM                   spect
PROBHD    5 mm TXI 1H-13
PULPROG                 zgpg30
TD                        65536
SOLVENT                   CDCl3
NS                         1698
DS                            4
SWH                30030.029 Hz
FIDRES             0.458222 Hz
AQ                 1.0912410 sec
RG                       812.7
DW                    16.650 usec
DE                     7.50 usec
TE                     296.4 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                           1

======== CHANNEL f1 ========
NUC1                       13C
P1                      12.20 usec
PL1                     -3.00 dB
PL1W             190.45114136 W
SFO1             125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                 100.00 usec
PL2                      0.00 dB
PL12                    23.48 dB
PL13                    25.00 dB
PL2W              15.07131863 W
PL12W              0.06763186 W
PL13W              0.04765970 W
SFO2             500.1320005 MHz
SI                        32768
SF               125.7577890 MHz
WDW                          no
SSB                           0
LB                       0.00 Hz
GB                            0
PC                       1.40
```



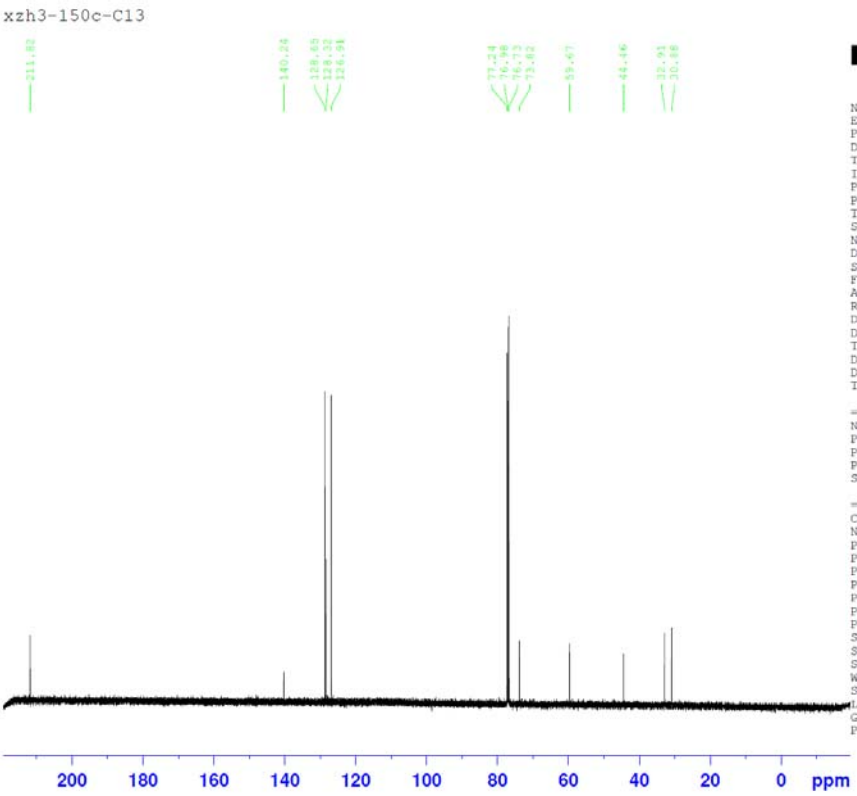
xzh3-150c-H



BRUKER

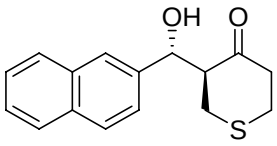
```
NAME            xzh3-150c-H
EXPNO                     2
PROCNO                    1
Date_              20090514
Time                  13.30
INSTRUM               spect
PROBHD    5 mm TXI 1H-13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                        8
DS                        2
SWH               10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1720407 sec
RG                      90.5
DW                   48.400 usec
DE                    7.50 usec
TE                   296.6 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    6.70 usec
PL1                   0.00 dB
PL1W             15.07131863 W
SFO1             500.1330885 MHz
SI                      32768
SF               500.1300000 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                     1.00
```

xzh3-150c-C13

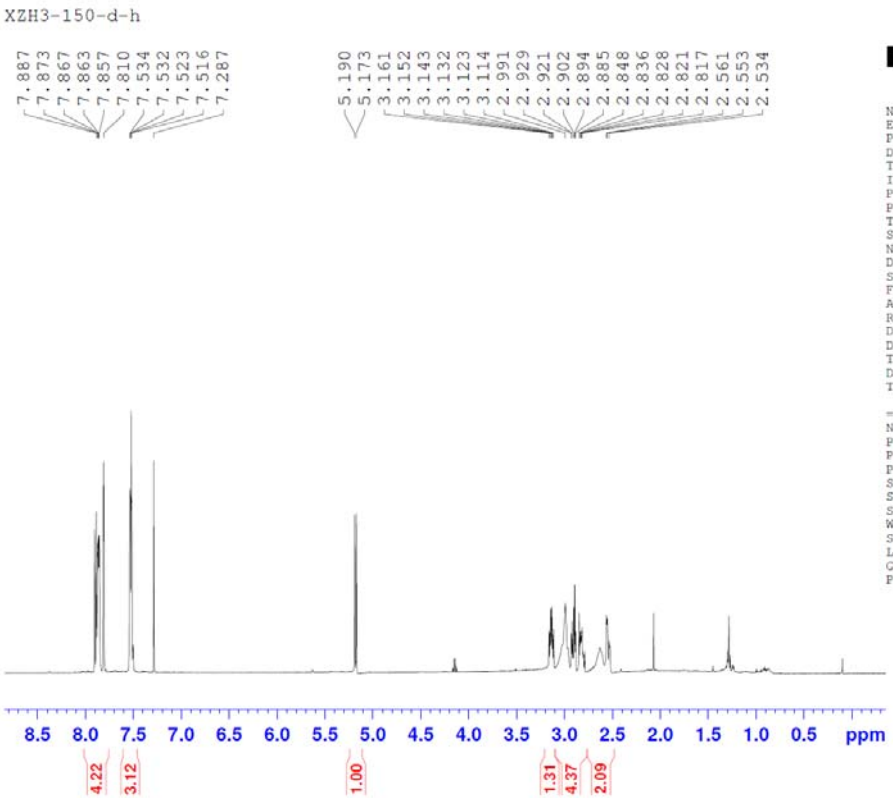


BRUKER

```
NAME          xzh3-150c-C13
EXPNO                     2
PROCNO                    1
Date_              20090514
Time                  14.48
INSTRUM               spect
PROBHD    5 mm TXI 1H-13
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                     1395
DS                        4
SWH               30030.029 Hz
FIDRES            0.458222 Hz
AQ                1.0912410 sec
RG                    812.7
DW                   16.650 usec
DE                    7.50 usec
TE                   297.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                   12.20 usec
PL1                  -3.00 dB
PL1W            190.45114136 W
SFO1             125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   0.00 dB
PL12                 23.48 dB
PL13                 25.00 dB
PL2W             15.07131863 W
PL12W             0.06763186 W
PL13W             0.04765970 W
SFO2             500.1320005 MHz
SI                      32768
SF               125.7577966 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                     1.40
```



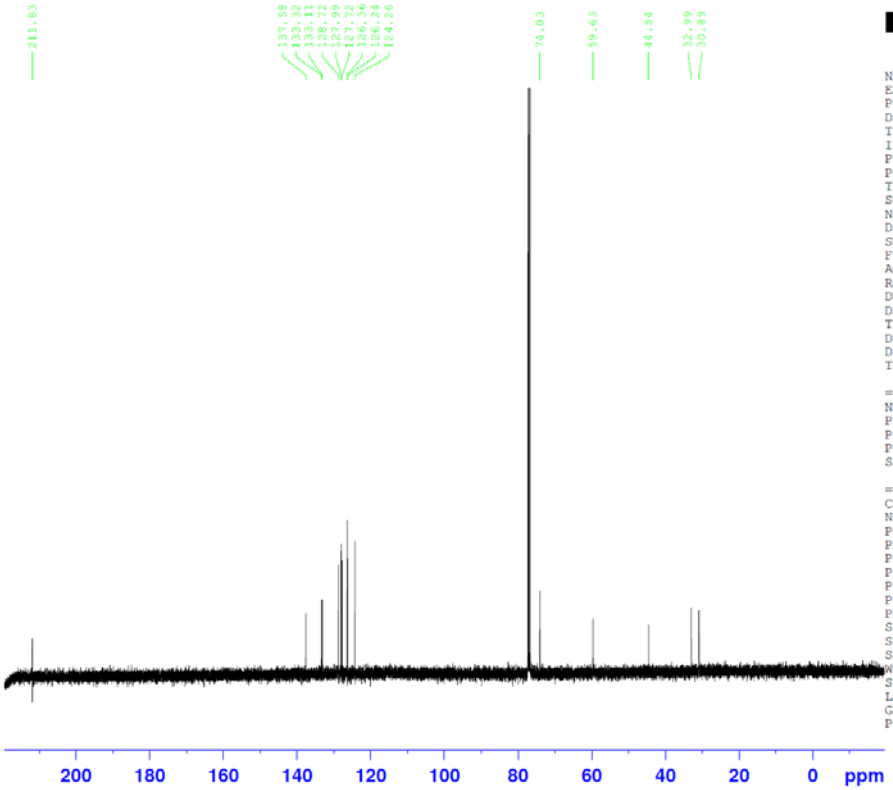
XZH3-150-d-h



BRUKER

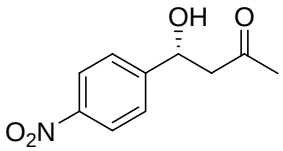
NAME	XZH3-150-d-h
EXPNO	2
PROCNO	1
Date_	20090514
Time	15.35
INSTRUM	spect
PROBHD	5 mm TXI 1H-13
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	161.3
DW	48.400 usec
DE	7.50 usec
TE	296.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	6.70 usec
PL1	0.00 dB
PL1W	15.07131863 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00

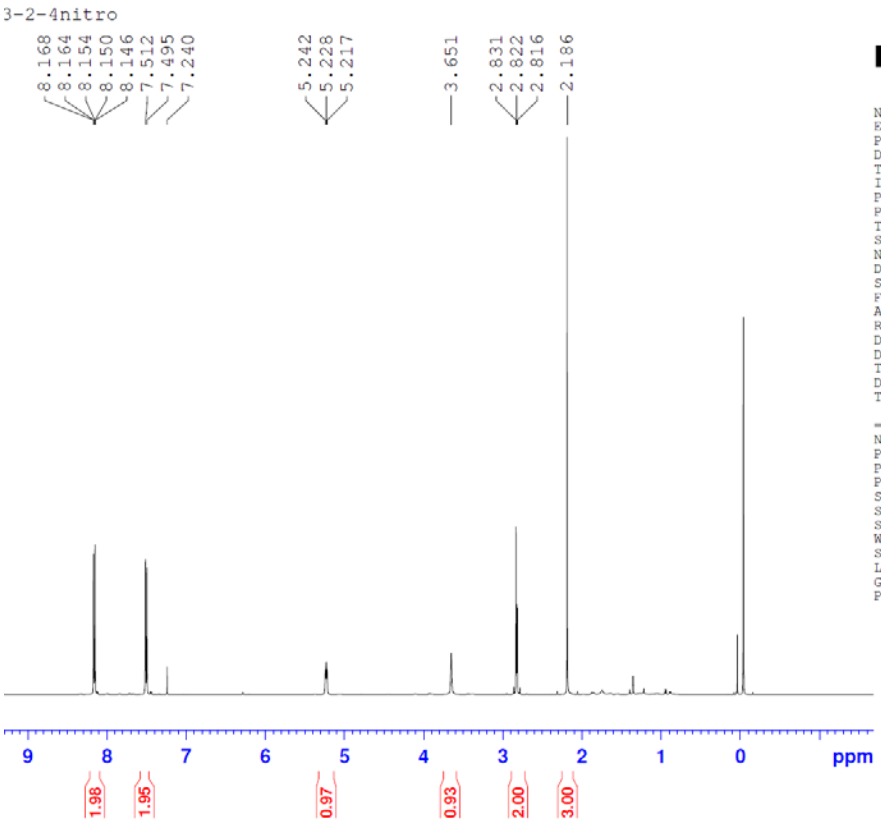
XZH3-150-d-C13



BRUKER

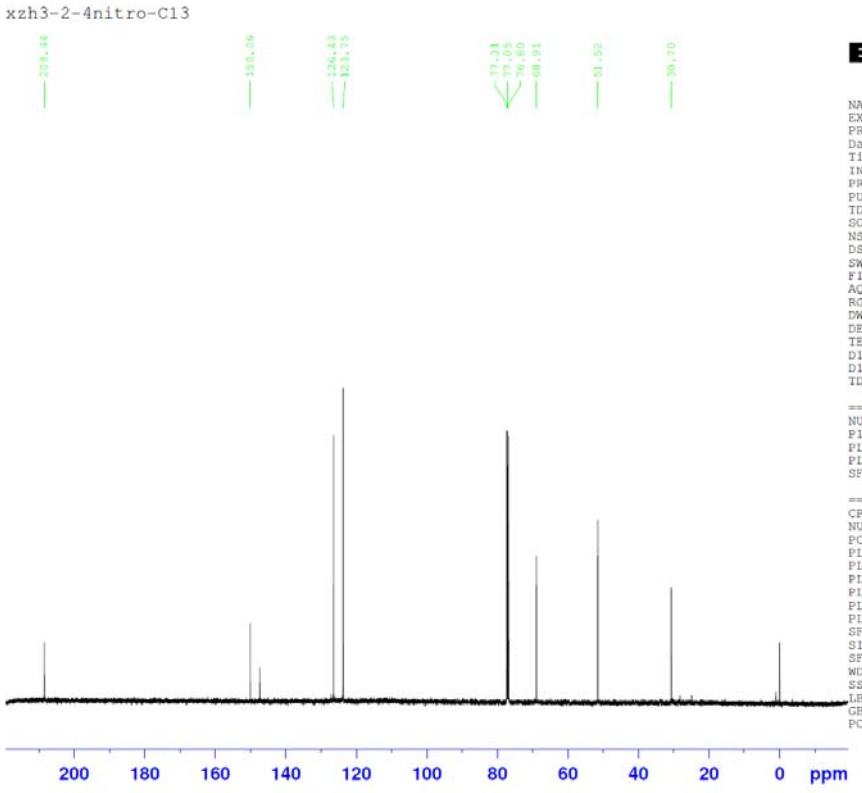
NAME	XZH3-150-d-C13
EXPNO	2
PROCNO	1
Date_	20090514
Time	17.25
INSTRUM	spect
PROBHD	5 mm TXI 1H-13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	2024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	812.7
DW	16.650 usec
DE	7.50 usec
TE	296.8 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.20 usec
PL1	-3.00 dB
PL1W	190.45114136 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	23.48 dB
PL13	25.00 dB
PL2W	15.07131863 W
PL12W	0.06763186 W
PL13W	0.04765970 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7577924 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40





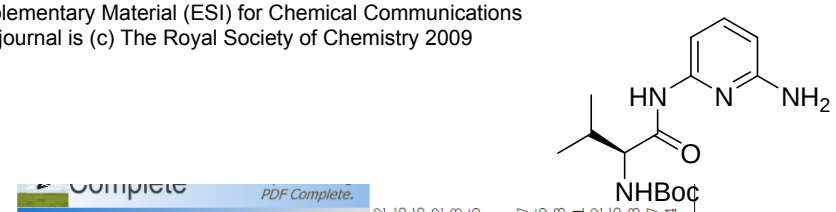
BRUKER

```
NAME          xzh3-2-4nitro
EXPNO                     1
PROCNO                    1
Date_              20081103
Time                  14.59
INSTRUM               spect
PROBHD   5 mm TXI 1H-13
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH               10330.578 Hz
FIDRES            0.157632 Hz
AQ                3.1720407 sec
RG                       57
DW                  48.400 usec
DE                   7.50 usec
TE                   297.9 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                   6.70 usec
PL1                  0.00 dB
PL1W          15.07131063 W
SFO1          500.1330885 MHz
SI                    32768
SF            500.1300230 MHz
WDW                     EM
SSB                      0
LB                   0.30 Hz
GB                       0
PC                    1.00
```



BRUKER

```
NAME       xzh3-2-4nitro-C13
EXPNO                     1
PROCNO                    1
Date_              20081103
Time                  15.32
INSTRUM               spect
PROBHD   5 mm TXI 1H-13
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      500
DS                        4
SWH               30030.029 Hz
FIDRES            0.458222 Hz
AQ                1.0912410 sec
RG                   7298.2
DW                  16.650 usec
DE                   7.50 usec
TE                   298.4 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                    13C
P1                   12.20 usec
PL1                  -3.00 dB
PL1W         190.45114136 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2               100.00 usec
PL2                  0.00 dB
PL12                23.48 dB
PL13                25.00 dB
PL2W          15.07131863 W
PL12W          0.06763186 W
PL13W          0.04765970 W
SFO2          500.1320005 MHz
SI                    32768
SF            125.7577890 MHz
WDW                     EM
SSB                      0
LB                   1.00 Hz
GB                       0
PC                    1.40
```



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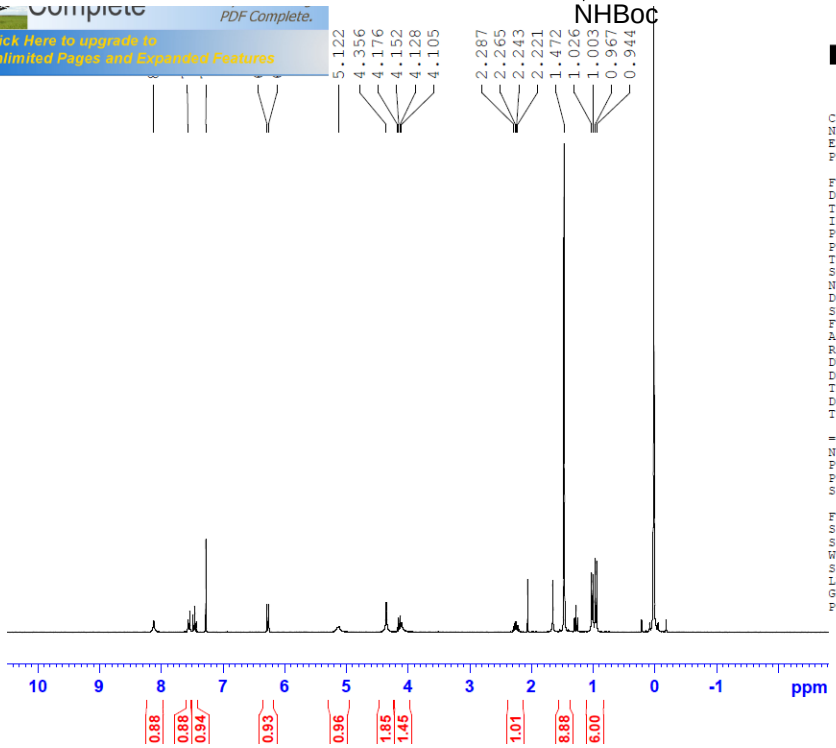


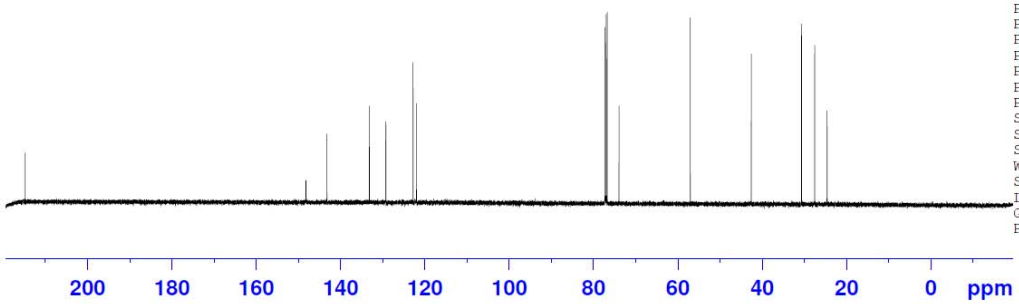
BRUKER

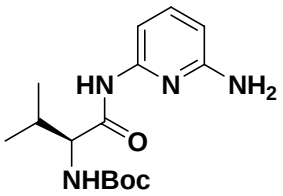
```
Current Data Parameters
NAME                 xzh3-10
EXPNO                      3
PROCNO                     2

F2 - Acquisition Parameters
Date_               20081116
Time                    9.14
INSTRUM                spect
PROBHD    5 mm Multinucl
PULPROG                zg30
TD                    65536
SOLVENT               CDCl3
NS                       16
DS                        2
SWH                6188.119 Hz
FIDRES            0.094423 Hz
AQ                5.2954397 sec
RG                    645.1
DW                   80.800 usec
DE                    6.00 usec
TE                   300.0 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     1H
P1                    14.10 usec
PL1                    3.00 dB
SFO1           300.1318534 MHz

F2 - Processing parameters
SI                    32768
SF               300.1300000 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

xzh3-110B-C13

BRUKER

```
NAME            xzh3-110-B-C13
EXPNO                        2
PROCNO                       1
Date_                 20090313
Time                      9.47
INSTRUM                  spect
PROBHD    5 mm TXI 1H-13
PULPROG               zgpg30
TD                        65536
SOLVENT                 CDCl3
NS                          898
DS                            4
SWH                30030.029 Hz
FIDRES              0.458222 Hz
AQ                  1.0912410 sec
RG                       1024
DW                     16.650 usec
DE                      7.50 usec
TE                     297.2 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                      12.20 usec
PL1                     -3.00 dB
PL1W           190.45114136 W
SFO1            125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                  100.00 usec
PL2                      0.00 dB
PL12                    23.48 dB
PL13                    25.00 dB
PL2W           15.07131863 W
PL12W           0.06763186 W
PL13W           0.04765970 W
SFO2           500.1320005 MHz
SI                       32768
SF              125.7577945 MHz
WDW                         no
SSB                          0
LB                      0.00 Hz
GB                           0
PC                      1.40
```





Chemical Formula: $C_{15}H_{24}N_4O_3$
Exact Mass: 308.18
Molecular Weight: 308.38
m/z: 308.18 (100.0%), 309.19 (16.6%), 310.19 (2.2%), 309.18 (1.5%)
Elemental Analysis: C, 58.42; H, 7.84; N, 18.17; O, 15.56

ESI: M+Na

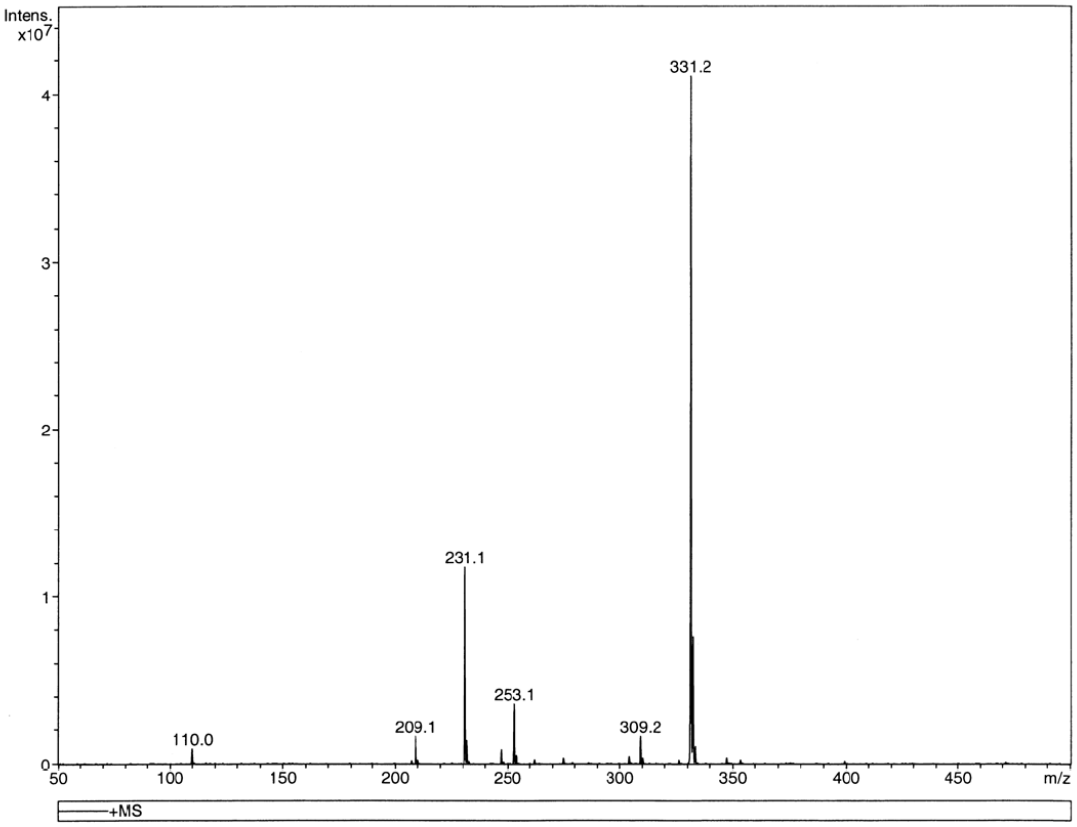
Display Report

Analysis Info

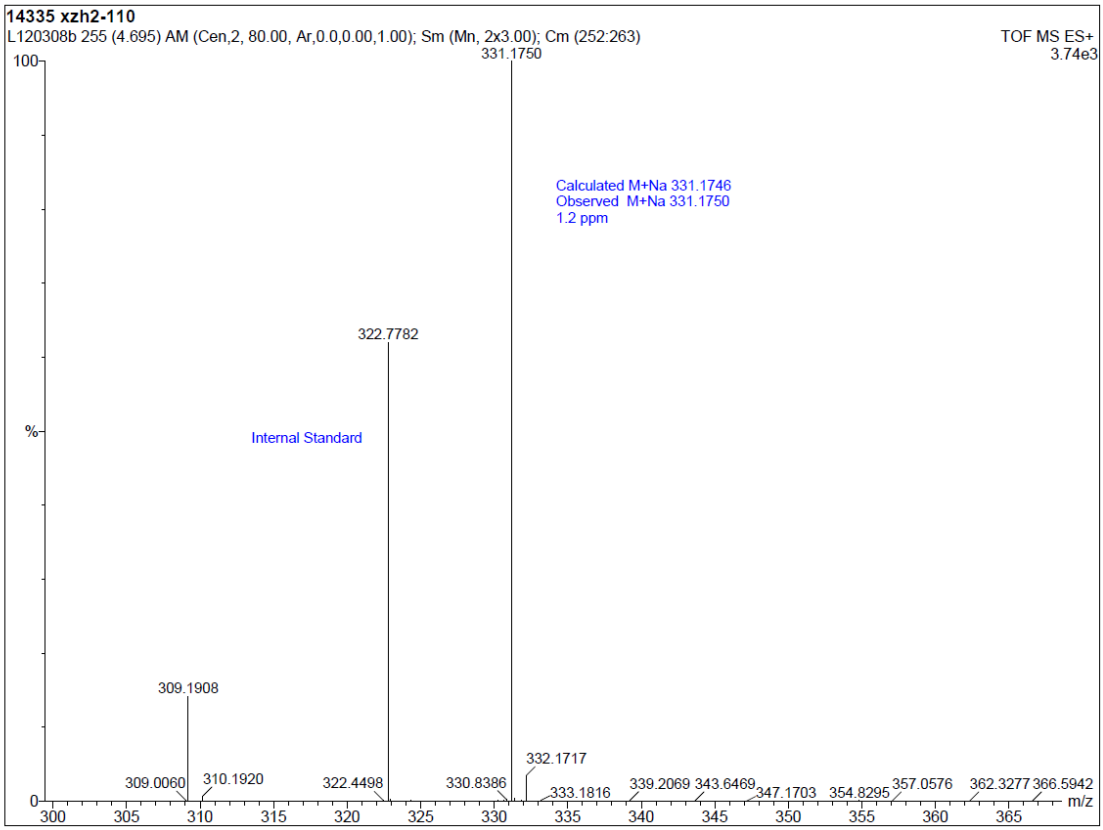
Analysis Name	XZH-1100.d	Acquisition Date	10/27/08 14:04:20
Method	NOVAKS.M	Operator	Administrator
Sample Name	XZH2-110	Instrument	Esquire-LC_00137
Comment	Diluted 1/100 in MEOH		

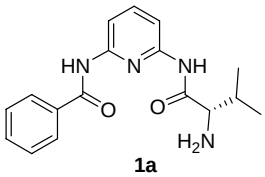
Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Normal	Scan Begin	50.00 m/z	Scan End	500.00 m/z
Capillary Exit	105.0 Volt	Skim 1	32.7 Volt	Trap Drive	35.4
Accumulation Time	95 μs	Averages	20 Spectra	Auto MS/MS	Off

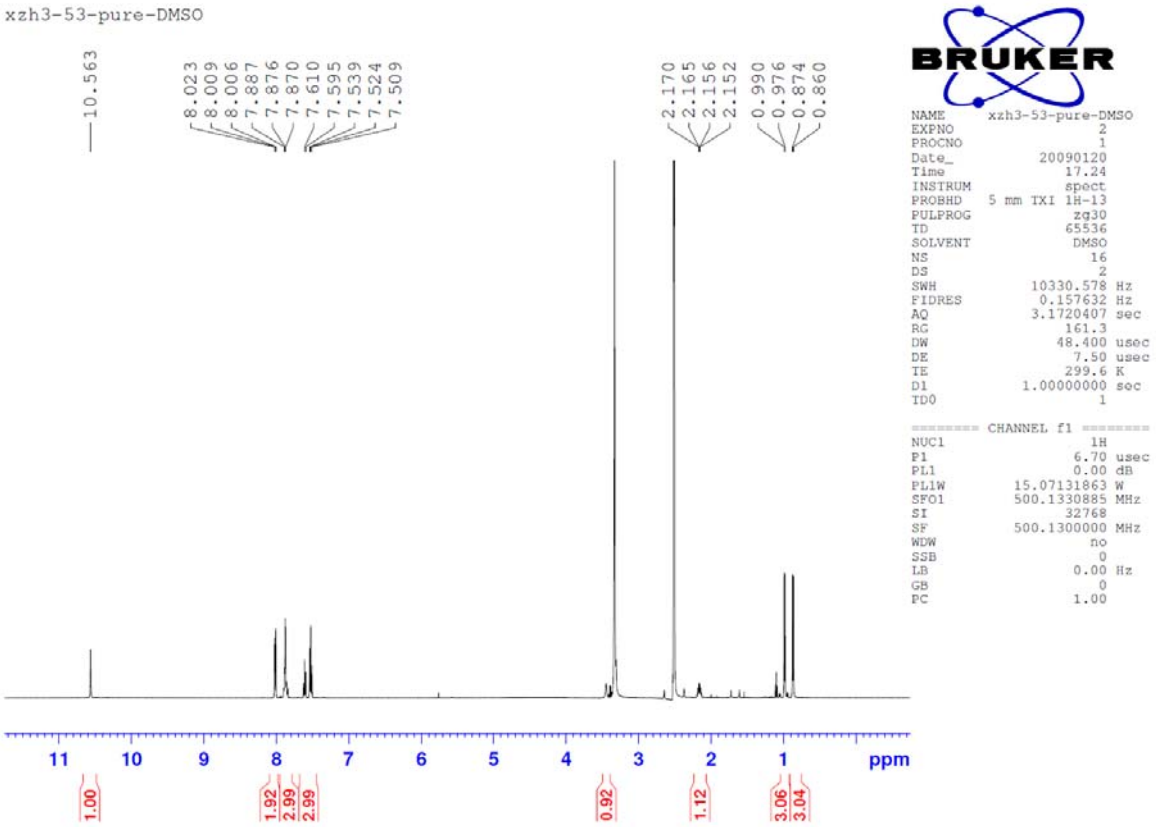


HRMS


14335 xzh2-110
L120308b 255 (4.695) AM (Cen,2, 80.00, Ar,0.0,0.00,1.00); Sm (Mn, 2x3.00); Cm (252:263)
TOF MS ES+
3.74e3
331.1750
Calculated M+Na 331.1746
Observed M+Na 331.1750
1.2 ppm
322.7782
Internal Standard
309.1908
309.0060
310.1920
322.4498
330.8386
332.1717
333.1816
339.2069
343.6469
347.1703
354.8295
357.0576
362.3277
366.5942
100
%
0
m/z
300
305
310
315
320
325
330
335
340
345
350
355
360
365

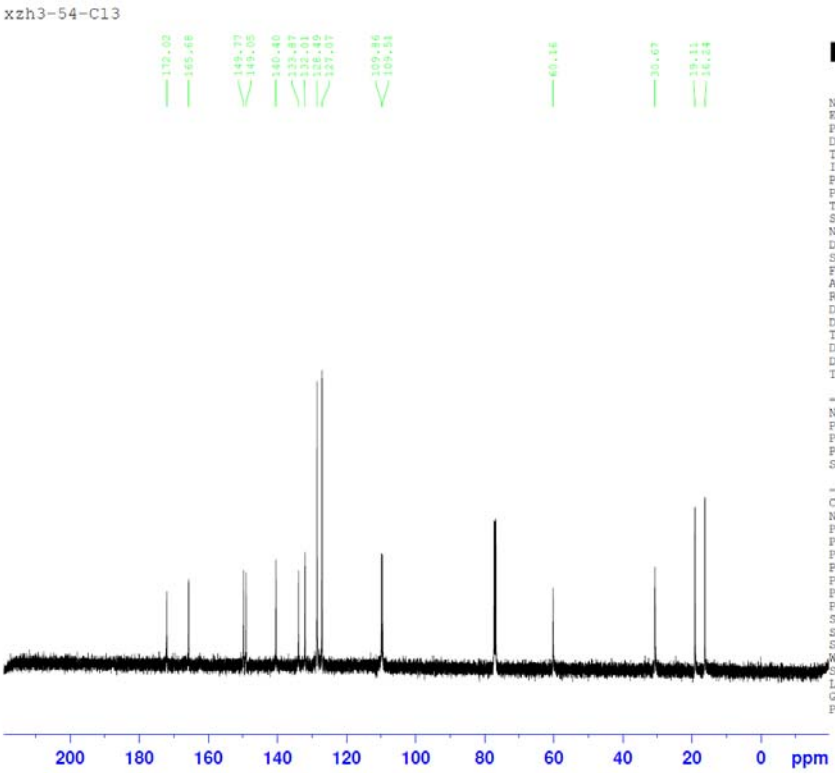


1a



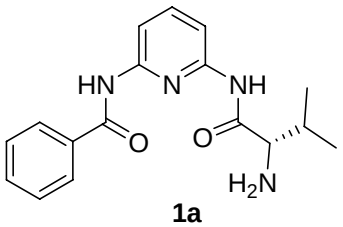
BRUKER

NAME	xzh3-53-pure-DMSO
EXPNO	2
PROCNO	1
Date_	20090120
Time	17.24
INSTRUM	spect
PROBHD	5 mm TXI 1H-13
PULPROG	zg30
TD	65536
SOLVENT	DMSO
NS	16
DS	2
SWH	10330.578 Hz
FIDRES	0.157632 Hz
AQ	3.1720407 sec
RG	161.3
DW	48.400 usec
DE	7.50 usec
TE	299.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	6.70 usec
PL1	0.00 dB
PL1W	15.07131863 W
SFO1	500.1330885 MHz
SI	32768
SF	500.1300000 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.00



BRUKER

NAME	xzh3-54-C13
EXPNO	2
PROCNO	1
Date_	20090410
Time	15.30
INSTRUM	spect
PROBHD	5 mm TXI 1H-13
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	30030.029 Hz
FIDRES	0.458222 Hz
AQ	1.0912410 sec
RG	1024
DW	16.650 usec
DE	7.50 usec
TE	297.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	12.20 usec
PL1	-3.00 dB
PL1W	190.45114136 W
SFO1	125.7703643 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	0.00 dB
PL12	23.48 dB
PL13	25.00 dB
PL2W	15.07131863 W
PL12W	0.06763186 W
PL13W	0.04765970 W
SFO2	500.1320005 MHz
SI	32768
SF	125.7578135 MHz
WDW	no
SSB	0
LB	0.00 Hz
GB	0
PC	1.40



1a

Chemical Formula: $C_{17}H_{20}N_4O_2$
Exact Mass: 312.16
Molecular Weight: 312.37
m/z: 312.16 (100.0%), 313.16 (20.2%), 314.17 (1.6%)
Elemental Analysis: C, 65.37; H, 6.45; N, 17.94; O, 10.24

ESI: [M+1] 313.2

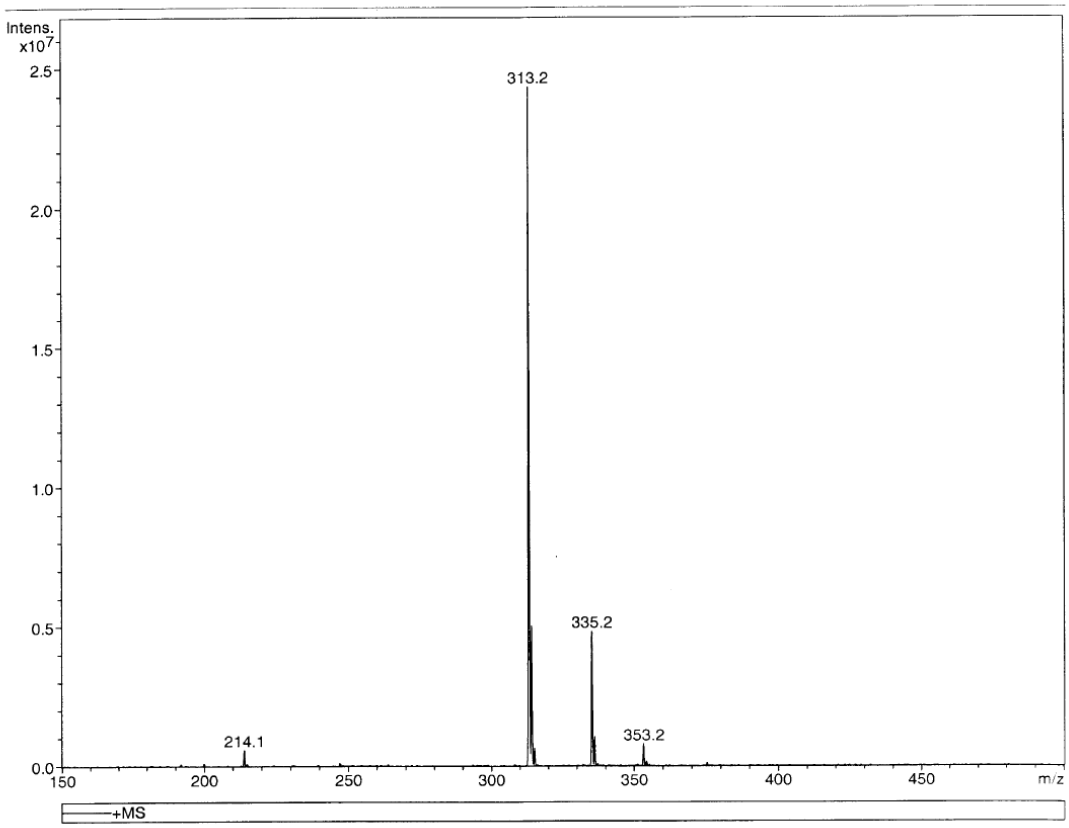
Display Report

Analysis Info

Analysis Name	xzh3-530.d	Acquisition Date	01/21/09 13:36:51
Method	NOVAKS.M	Operator	Administrator
Sample Name	xzh3-53	Instrument	Esquire-LC_00137
Comment	Diluted 1/100 in MEOH		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Normal	Scan Begin	150.00 m/z	Scan End	500.00 m/z
Capillary Exit	79.2 Volt	Skim 1	12.7 Volt	Trap Drive	29.6
Accumulation Time	192 μs	Averages	20 Spectra	Auto MS/MS	Off

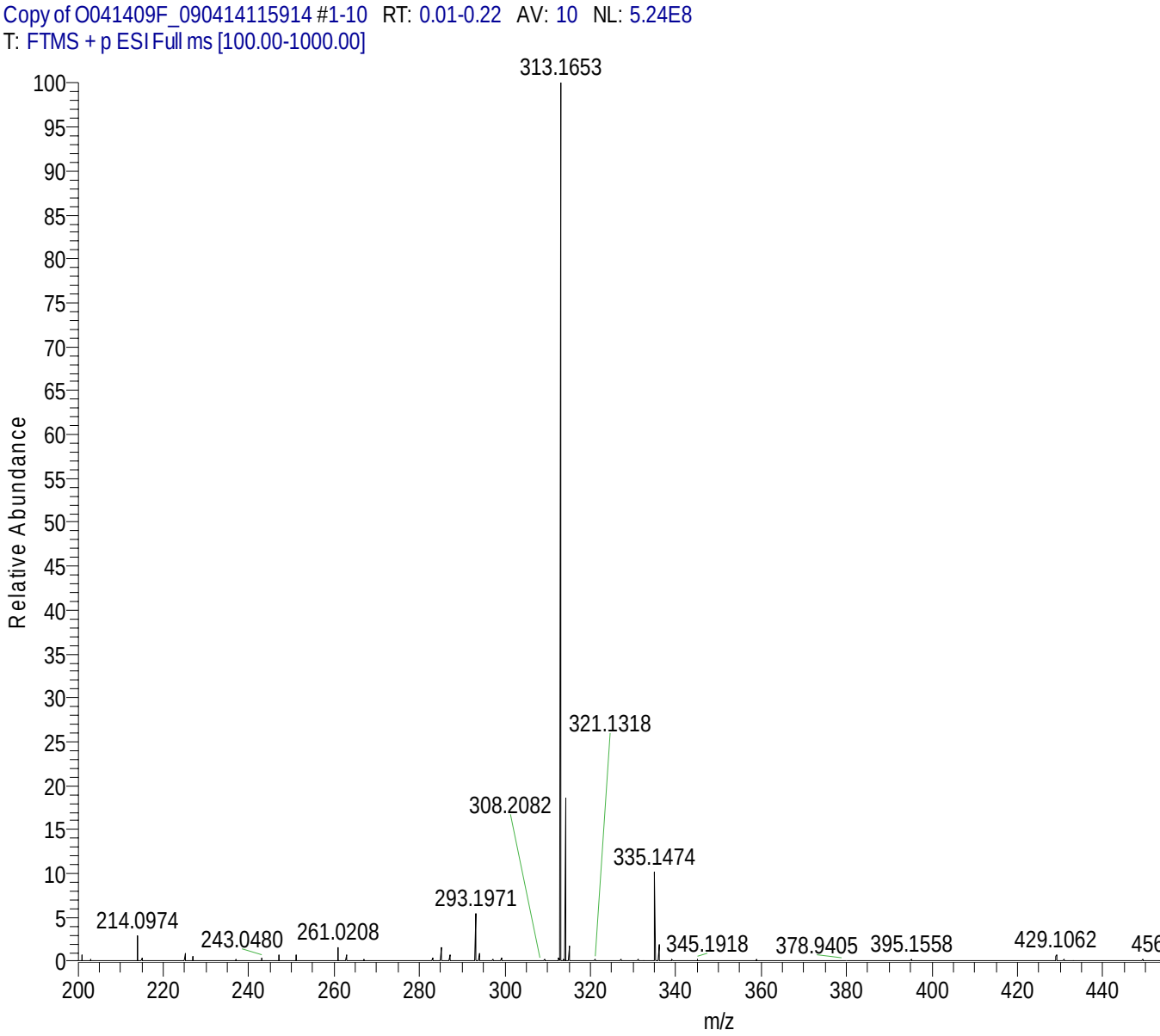


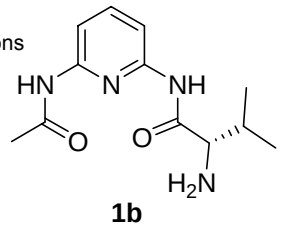
HRMS

Calculated mass H+ 313.1664

Measured mass 313.1653

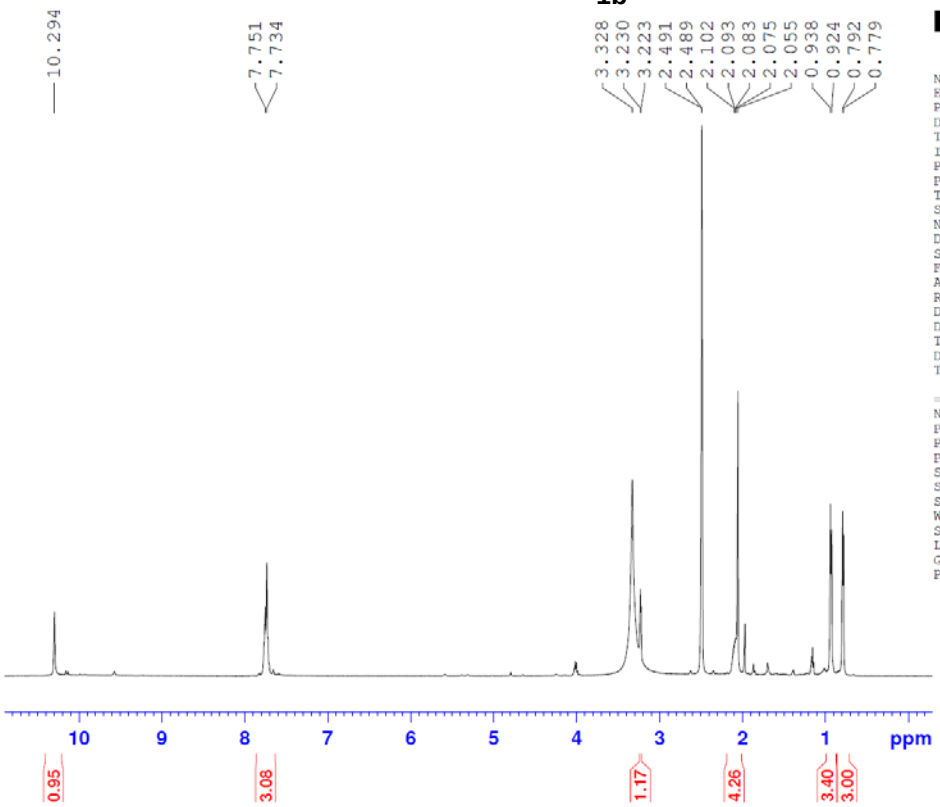
3.5 ppm





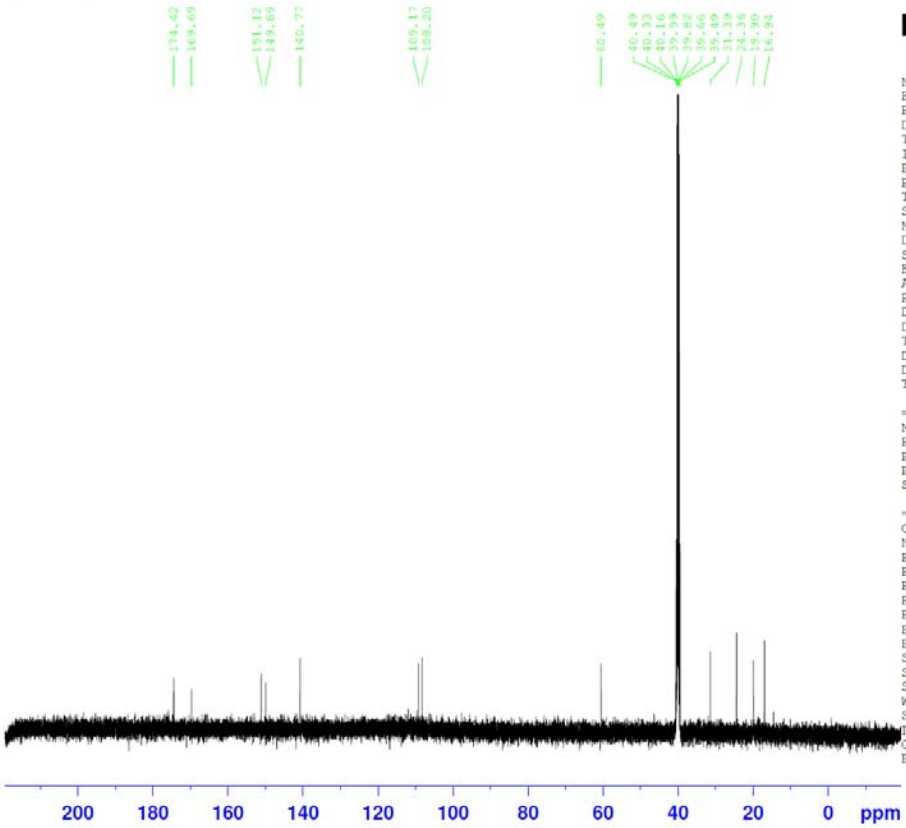
1b

xzh3-97p

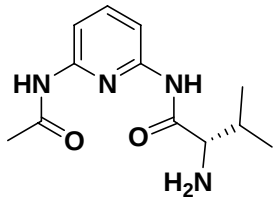



```
NAME                xzh3-97p
EXPNO                      2
PROCNO                     1
Date_               20090304
Time                   10.06
INSTRUM                spect
PROBHD    5 mm TXI 1H-13
PULPROG                zg30
TD                    65536
SOLVENT                DMSO
NS                       16
DS                        2
SWH             10330.578 Hz
FIDRES          0.157632 Hz
AQ              3.1720407 sec
RG                    161.3
DW                 48.400 usec
DE                  7.50 usec
TE                  297.7 K
D1              1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                  6.70 usec
PL1                 0.00 dB
PL1W          15.07131863 W
SFO1          500.1330885 MHz
SI                     32768
SF            500.1300090 MHz
WDW                       no
SSB                        0
LB                  0.00 Hz
GB                         0
PC                      1.00
```

xzh3-97p-C13




```
NAME             xzh3-97p-C13
EXPNO                       2
PROCNO                      1
Date_                20090304
Time                    11.32
INSTRUM                 spect
PROBHD     5 mm TXI 1H-13
PULPROG              zgpg30
TD                     65536
SOLVENT                DMSO
NS                      1516
DS                         4
SWH             30030.029 Hz
FIDRES          0.458222 Hz
AQ              1.0912410 sec
RG                    1149.4
DW                 16.650 usec
DE                  7.50 usec
TE                  298.5 K
D1              2.00000000 sec
D11             0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                  12.20 usec
PL1                 -3.00 dB
PL1W          190.45114136 W
SFO1          125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2              100.00 usec
PL2                 0.00 dB
PL12                23.48 dB
PL13                25.00 dB
PL2W          15.07131863 W
PL12W          0.06763186 W
PL13W          0.04765970 W
SFO2          500.1320005 MHz
SI                     32768
SF            125.7577890 MHz
WDW                       no
SSB                        0
LB                  0.00 Hz
GB                         0
PC                      1.40
```



1b

Chemical Formula: $C_{12}H_{18}N_4O_2$
Exact Mass: 250.14
Molecular Weight: 250.3
m/z: 250.14 (100.0%), 251.15 (13.3%), 251.14 (1.5%), 252.15 (1.2%)
Elemental Analysis: C, 57.58; H, 7.25; N, 22.38; O, 12.78

ESI: $[M-H]^-$ 248.9

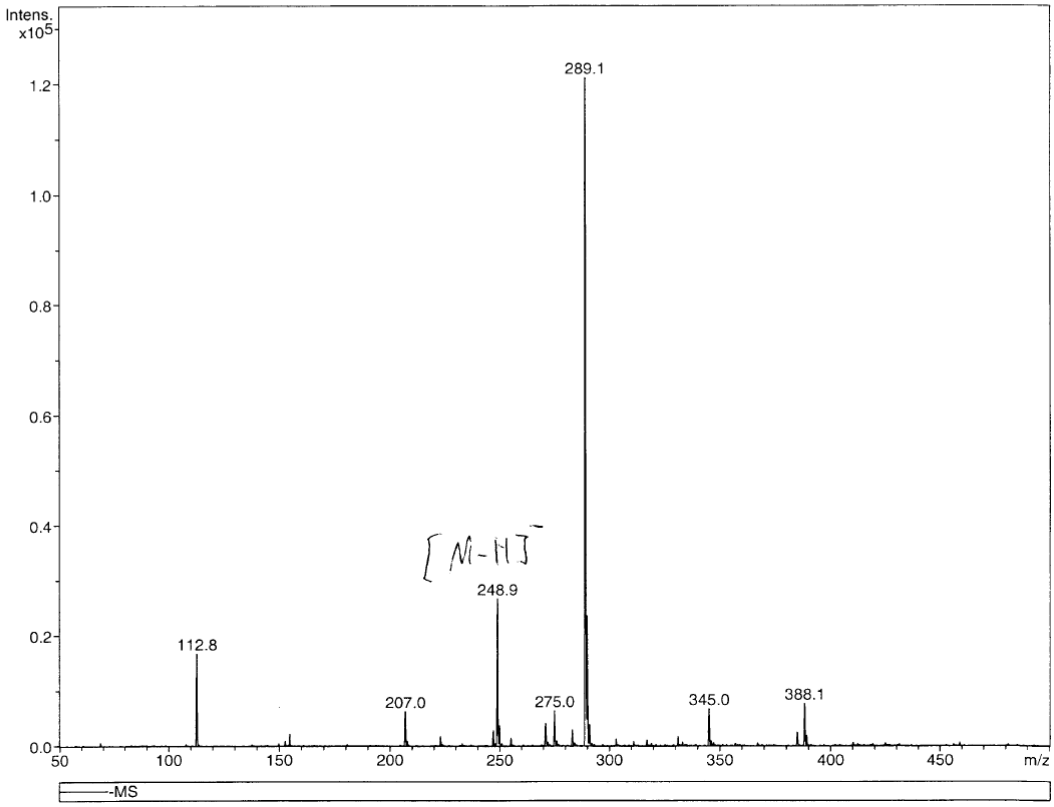
Display Report

Analysis Info

Analysis Name	XZH3-973.d	Acquisition Date	03/10/09 12:23:30
Method	XQ Default.ms	Operator	Administrator
Sample Name	XZH3-97	Instrument	Esquire-LC_00137
Comment	Diluted 1/100 in MEOH		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Negative	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Normal	Scan Begin	50.00 m/z	Scan End	500.00 m/z
Capillary Exit	-88.0 Volt	Skim 1	-19.9 Volt	Trap Drive	41.9
Accumulation Time	27191 μs	Averages	20 Spectra	Auto MS/MS	Off

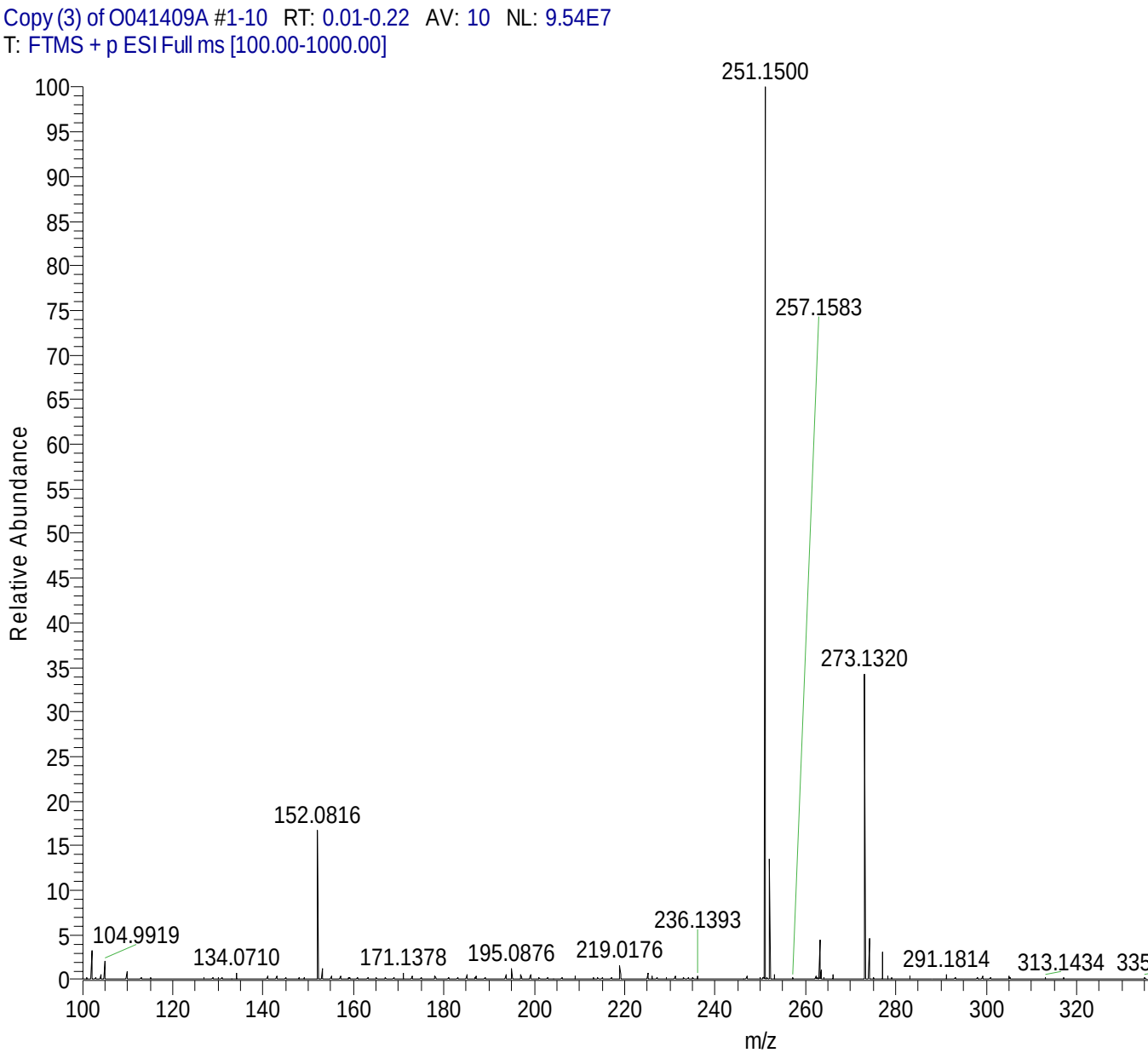


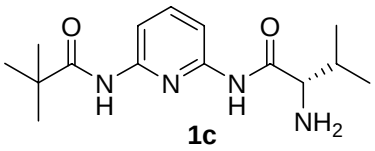
HRMS

Calculated mass H+ 251.1508

Measured mass 251.1500

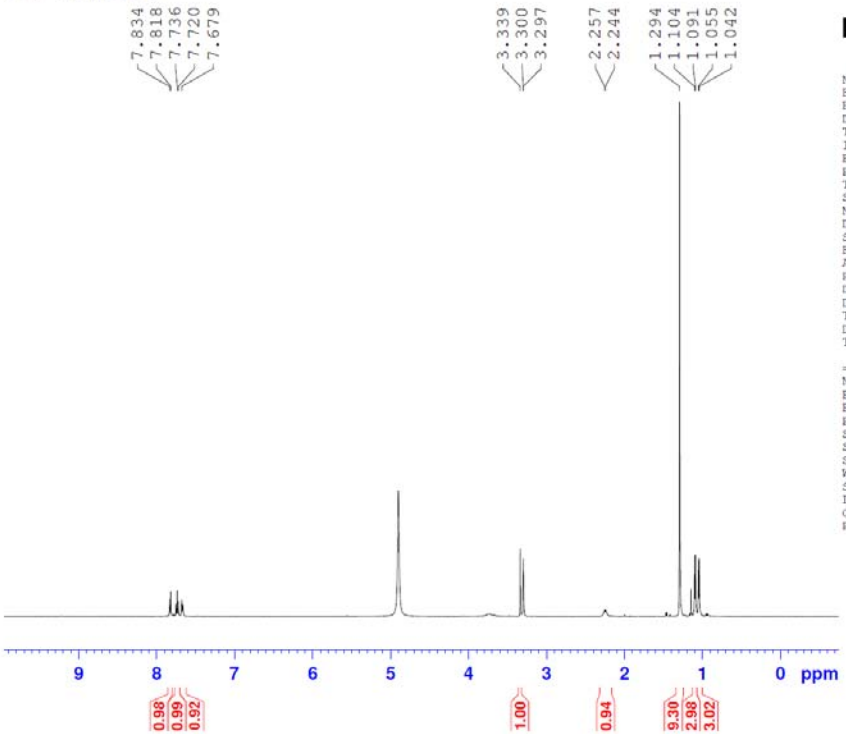
3.18 ppm





1c

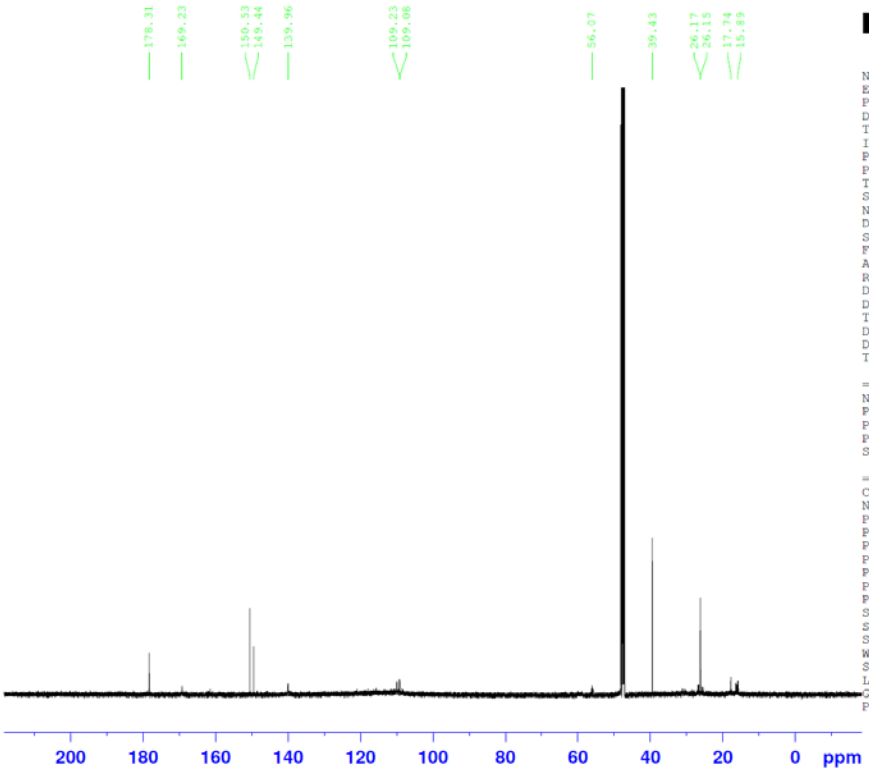
xzh3-98-MEOH-H



BRUKER

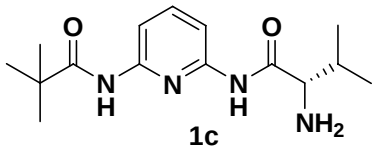
```
NAME          xzh3-98-MEOH-H
EXPNO                      2
PROCNO                     1
Date_               20090411
Time                   10.15
INSTRUM                spect
PROBHD   5 mm TXI 1H-13
PULPROG                zg30
TD                    65536
SOLVENT                MeOD
NS                        8
DS                        2
SWH              10330.578 Hz
FIDRES               0.157632 Hz
AQ                3.1720407 sec
RG                      114
DW                   48.400 usec
DE                    7.50 usec
TE                   297.1 K
D1                1.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    6.70 usec
PL1                   0.00 dB
PL1W         15.07131863 W
SFO1         500.1330885 MHz
SI                    32768
SF           500.1300155 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    1.00
```

xzh3-98-MEOH--C13-1



BRUKER

```
NAME          xzh3-98-MEOH--C13-1
EXPNO                      2
PROCNO                     1
Date_               20090413
Time                   7.50
INSTRUM                spect
PROBHD   5 mm TXI 1H-13
PULPROG             zgpg30
TD                    65536
SOLVENT                MeOD
NS                    15024
DS                        4
SWH              29761.904 Hz
FIDRES               0.454131 Hz
AQ                1.1010716 sec
RG                   11585.2
DW                   16.800 usec
DE                    8.94 usec
TE                   297.7 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                     13C
P1                   12.20 usec
PL1                  -3.00 dB
PL1W        190.45114136 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                   0.00 dB
PL12                 23.48 dB
PL13                 25.00 dB
PL2W         15.07131863 W
PL12W         0.06763186 W
PL13W         0.04765970 W
SFO2         500.1320005 MHz
SI                    32768
SF           125.7577890 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    1.40
```



1c

Chemical Formula: $C_{15}H_{24}N_4O_2$
Exact Mass: 292.19
Molecular Weight: 292.38
m/z: 292.19 (100.0%), 293.19 (17.8%), 294.20 (1.3%)
Elemental Analysis: C, 61.62; H, 8.27; N, 19.16; O, 10.94

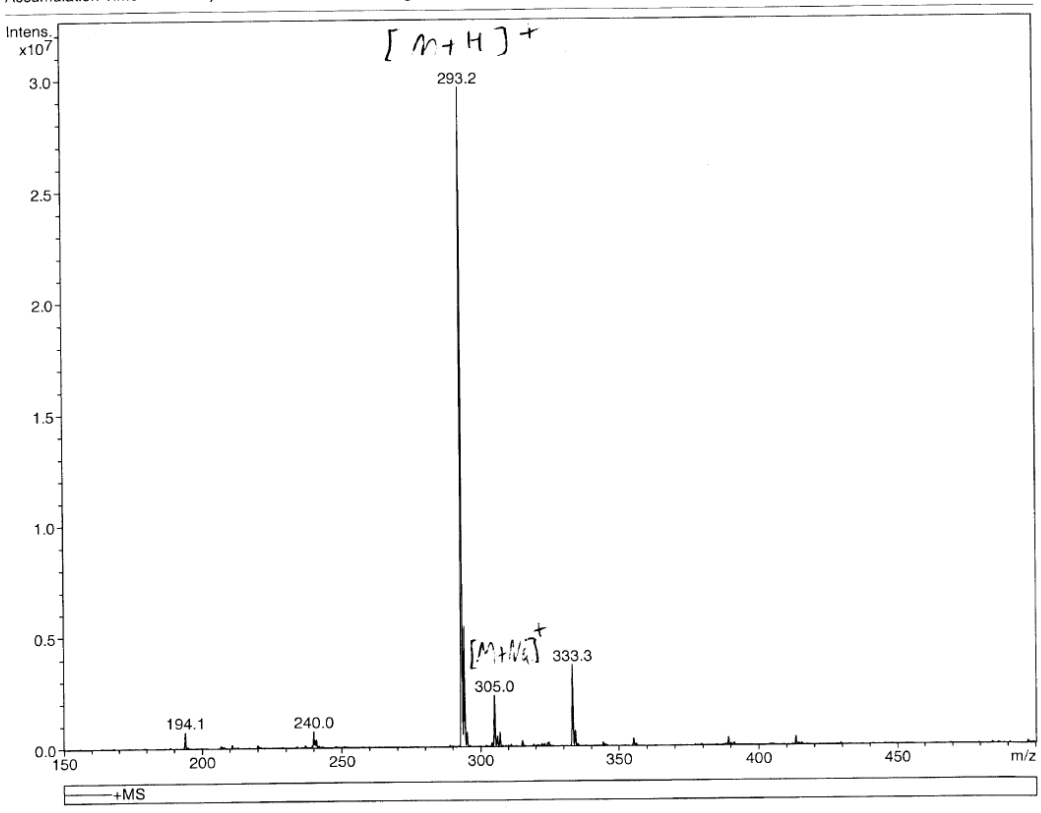
ESI [M+H] 293.2

Analysis Info

Analysis Name	XZh3-980.d	Acquisition Date	03/10/09 13:16:00
Method	XQ Default.ms	Operator	Administrator
Sample Name	XZH3-98	Instrument	Esquire-LC_00137
Comment	Diluted 1/100 in MEOH.		

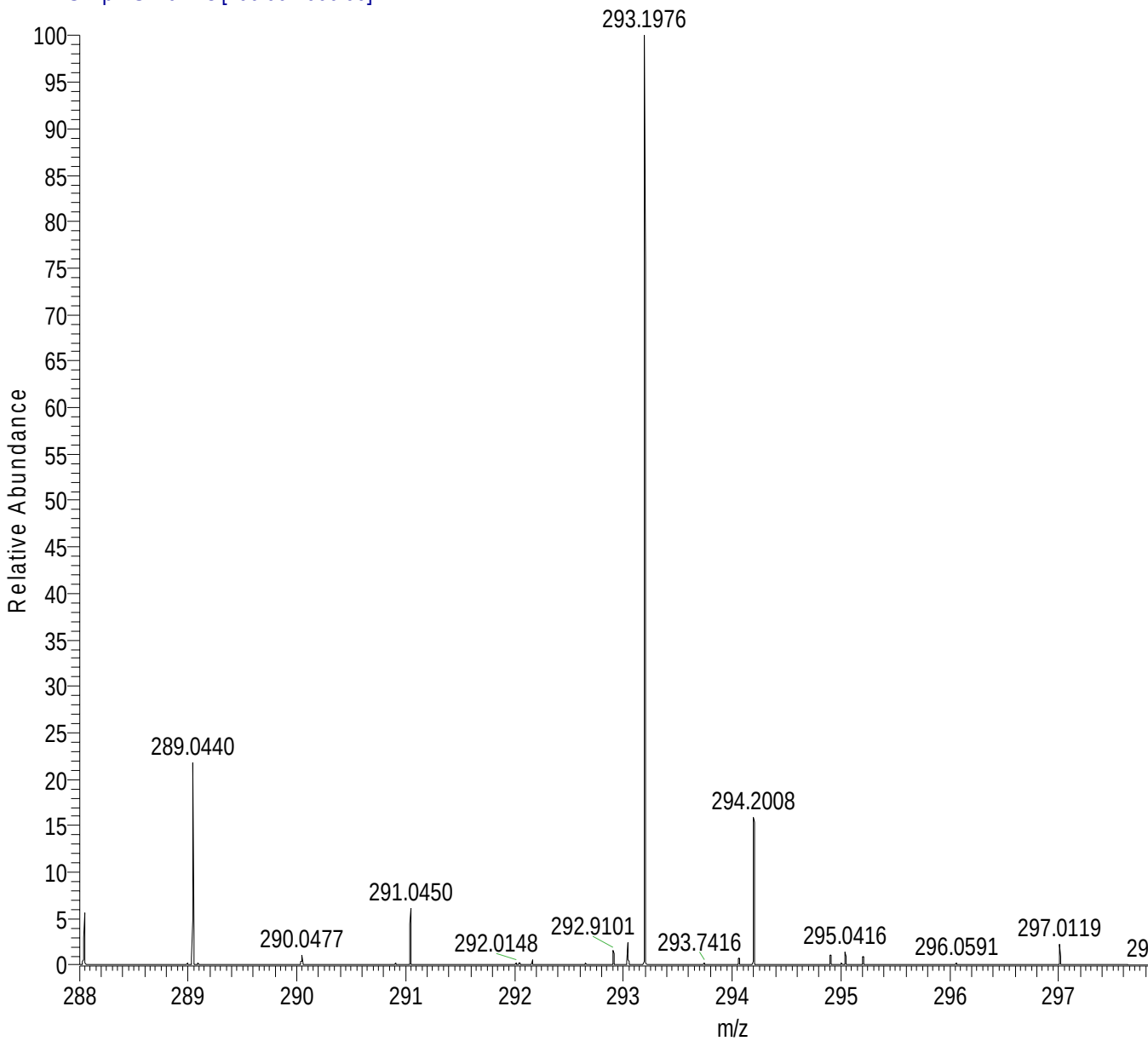
Acquisition Parameter

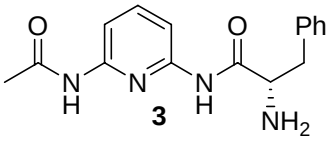
Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Normal	Scan Begin	150.00 m/z	Scan End	500.00 m/z
Capillary Exit	82.2 Volt	Skim 1	15.2 Volt	Trap Drive	30.7
Accumulation Time	140 μs	Averages	20 Spectra	Auto MS/MS	Off



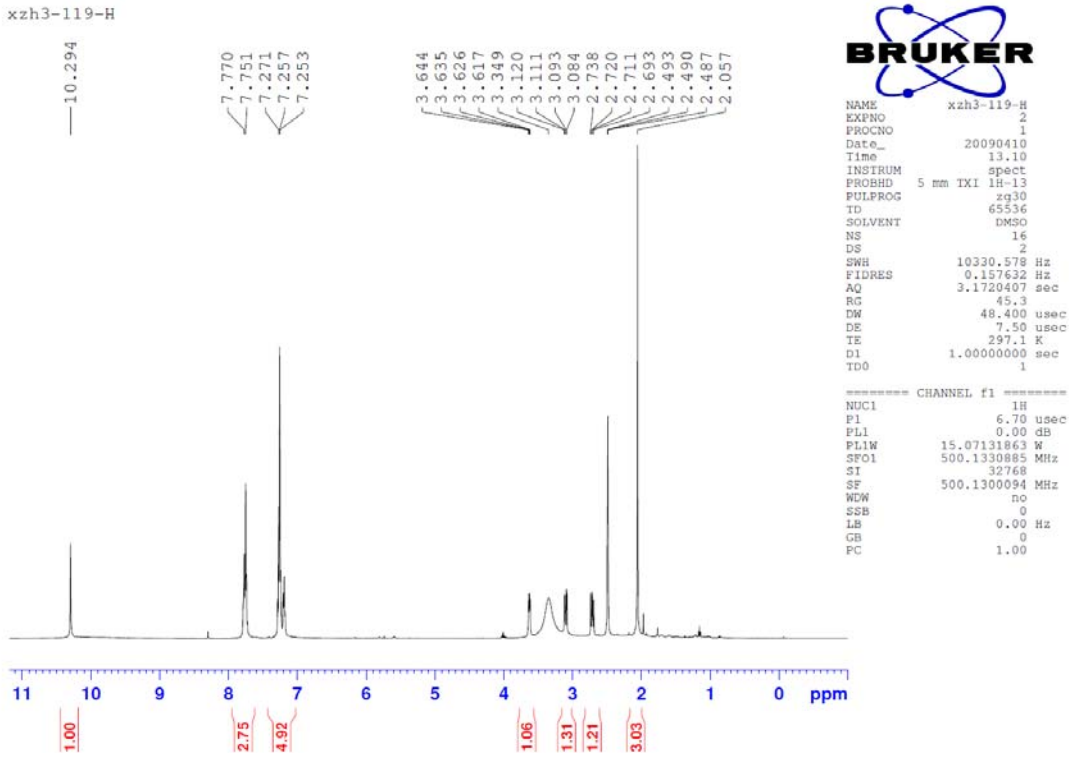
HRMS

Calculated mass H+ 293.1977
Measured mass 293.1976
0.3 ppm

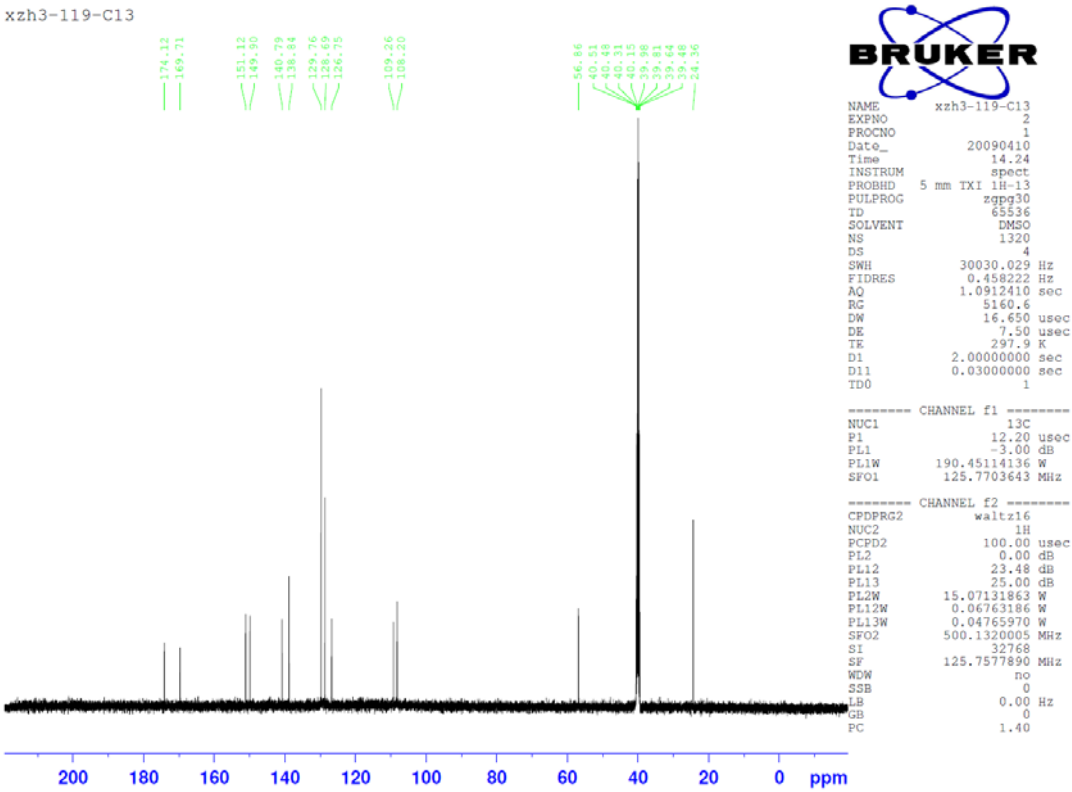




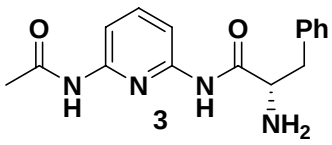
3




```
NAME            xzh3-119-H
EXPNO                    2
PROCNO                   1
Date_             20090410
Time                 13.10
INSTRUM              spect
PROBHD   5 mm TXI 1H-13
PULPROG               zg30
TD                   65536
SOLVENT               DMSO
NS                      16
DS                       2
SWH              10330.578 Hz
FIDRES           0.157632 Hz
AQ               3.1720407 sec
RG                    45.3
DW                  48.400 usec
DE                    7.50 usec
TE                   297.1 K
D1             1.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                    6.70 usec
PL1                   0.00 dB
PL1W         15.07131863 W
SFO1         500.1330885 MHz
SI                   32768
SF           500.1300094 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
```




```
NAME            xzh3-119-C13
EXPNO                    2
PROCNO                   1
Date_             20090410
Time                 14.24
INSTRUM              spect
PROBHD   5 mm TXI 1H-13
PULPROG             zgpg30
TD                   65536
SOLVENT               DMSO
NS                    1320
DS                       4
SWH              30030.029 Hz
FIDRES           0.458222 Hz
AQ               1.0912410 sec
RG                  5160.6
DW                  16.650 usec
DE                    7.50 usec
TE                   297.9 K
D1             2.00000000 sec
D11            0.03000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                   13C
P1                   12.20 usec
PL1                  -3.00 dB
PL1W        190.45114136 W
SFO1         125.7703643 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2               100.00 usec
PL2                   0.00 dB
PL12                 23.48 dB
PL13                 25.00 dB
PL2W         15.07131863 W
PL12W         0.06763186 W
PL13W         0.04765970 W
SFO2         500.1320005 MHz
SI                   32768
SF           125.7577890 MHz
WDW                     no
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.40
```



3

Chemical Formula: $C_{16}H_{18}N_4O_2$
Exact Mass: 298.14
Molecular Weight: 298.34
m/z: 298.14 (100.0%), 299.15 (17.6%), 300.15 (1.9%), 299.14 (1.5%)
Elemental Analysis: C, 64.41; H, 6.08; N, 18.78; O, 10.73

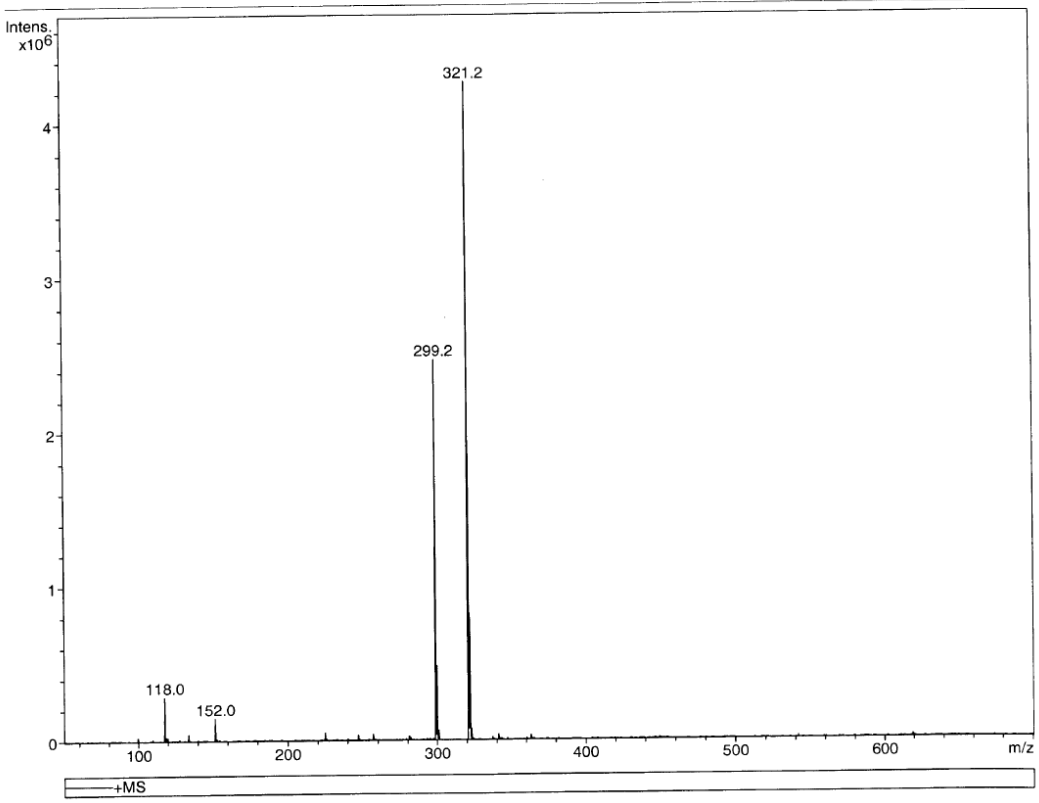
ESI [M+H] 299.2 [M+Na] 321.2

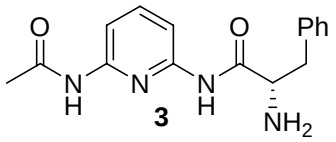
Analysis Info

Analysis Name	XZH-1190.d	Acquisition Date	04/01/09 11:28:35
Method	XQ Default.ms	Operator	Administrator
Sample Name	XZH3-119	Instrument	Esquire-LC_00137
Comment	Diluted 1/100 in MEOH.		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Normal	Scan Begin	50.00 m/z	Scan End	700.00 m/z
Capillary Exit	82.2 Volt	Skim 1	15.2 Volt	Trap Drive	30.7
Accumulation Time	768 µs	Averages	20 Spectra	Auto MS/MS	Off

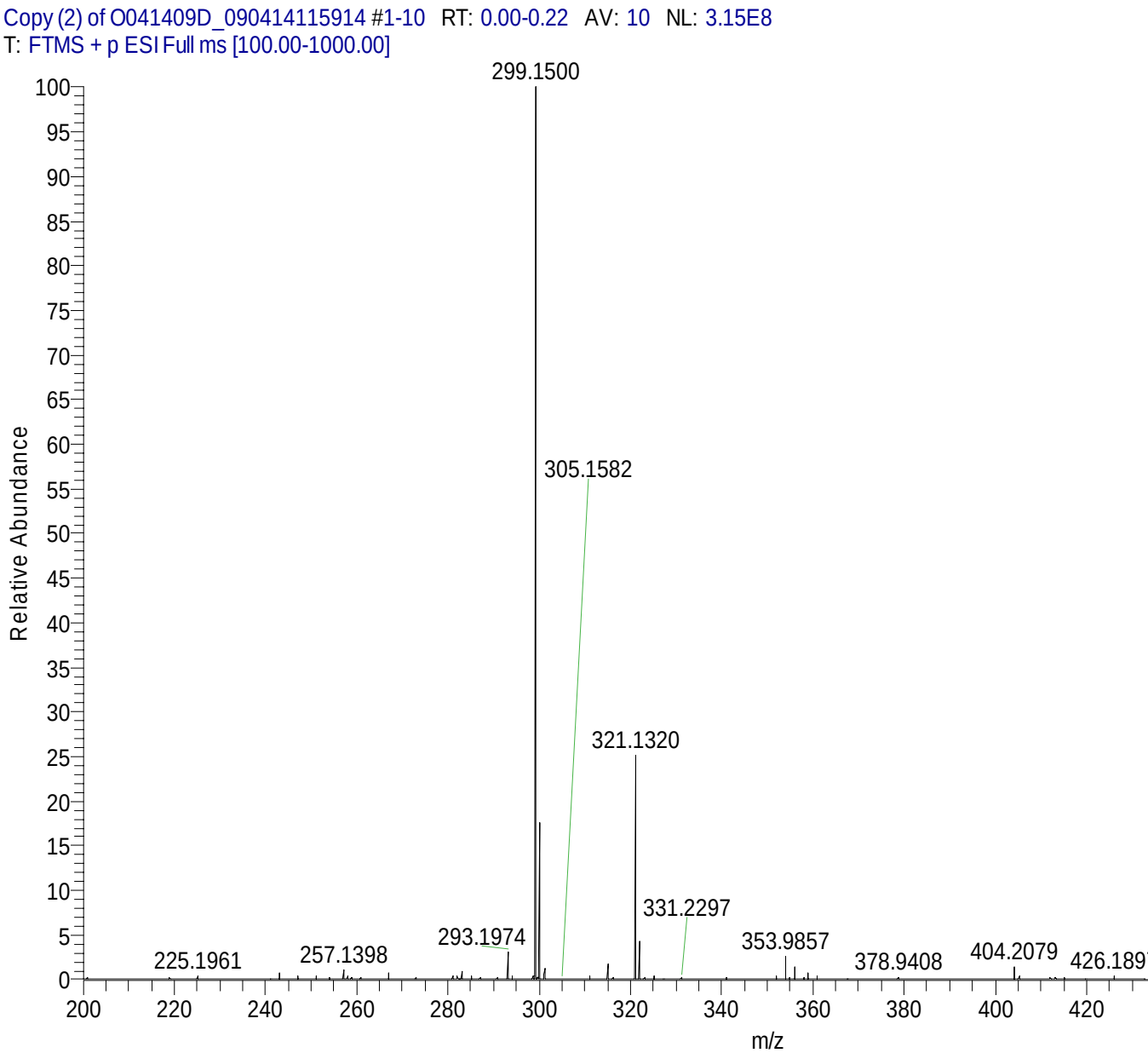


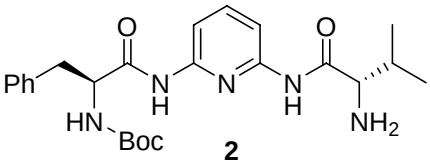


3

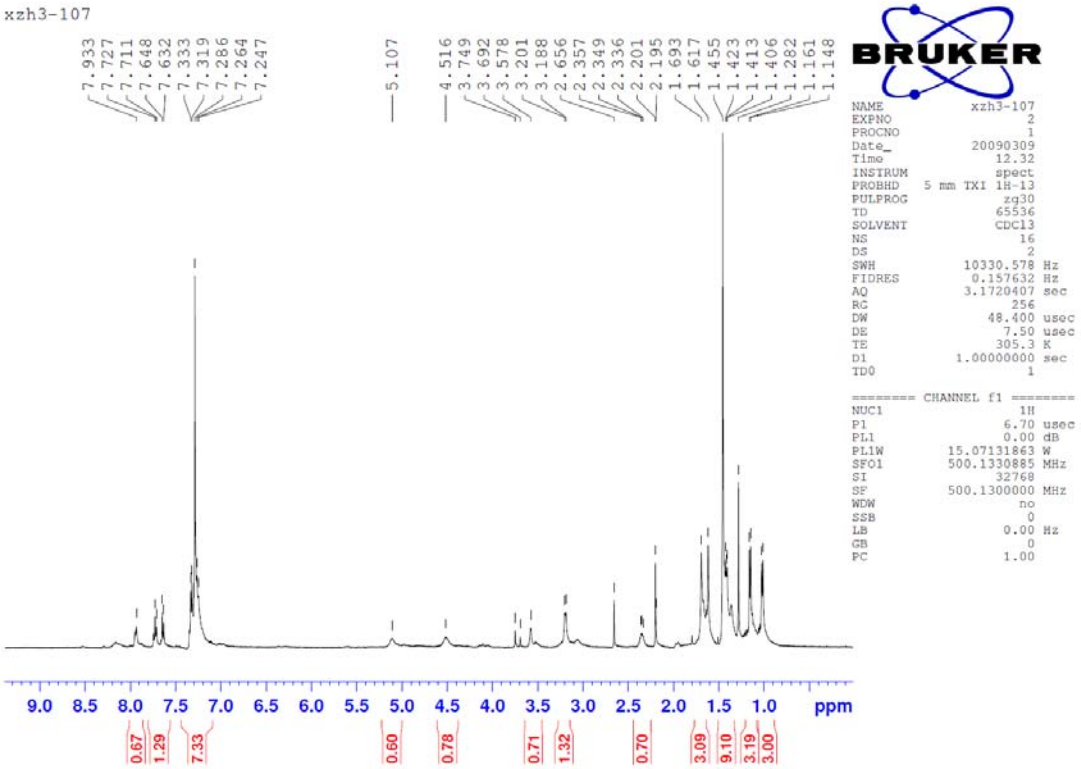
HRMS

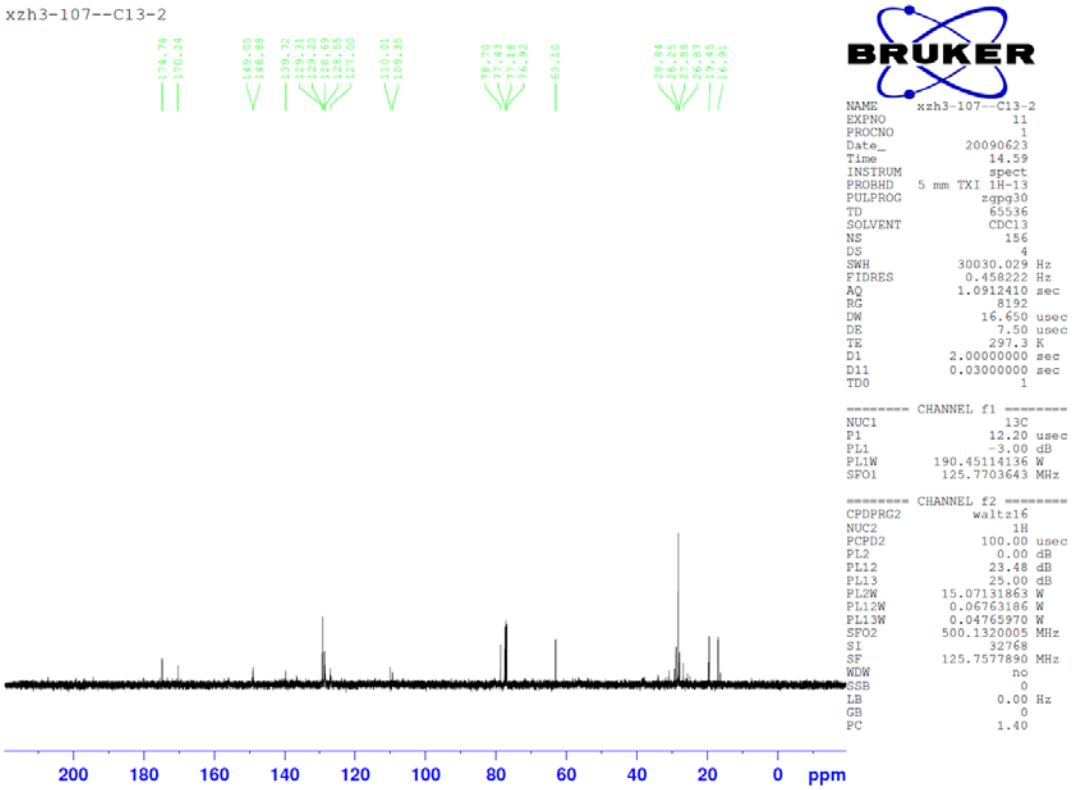
Calculated mass H+ 299.1508
Measured mass 299.1500
2.7 ppm

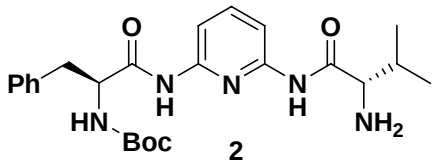



Ph
O
N
H
N
N
H
O
HN
Boc
NH2

2


xzh3-107
7.933
7.727
7.711
7.648
7.632
7.333
7.319
7.286
7.264
7.247
5.107
4.516
3.749
3.692
3.578
3.201
3.188
2.656
2.357
2.349
2.336
2.201
2.195
1.693
1.617
1.455
1.423
1.413
1.406
1.282
1.161
1.148
BRUKER
NAME xzh3-107
EXPNO 2
PROCNO 1
Date_ 20090309
Time 12.32
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 256
DW 48.400 usec
DE 7.50 usec
TE 305.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
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 ppm
0.67
1.29
7.33
0.60
0.78
0.71
1.32
0.70
3.09
9.10
3.19
3.00


xzh3-107--C13-2
174.74
170.24
149.05
148.88
139.72
129.31
129.20
128.69
128.58
127.00
110.01
109.35
78.70
77.43
77.18
76.92
63.10
28.84
28.25
27.88
26.87
19.45
16.91
BRUKER
NAME xzh3-107--C13-2
EXPNO 11
PROCNO 1
Date_ 20090623
Time 14.59
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 156
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 8192
DW 16.650 usec
DE 7.50 usec
TE 297.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 0 ppm



2

Chemical Formula: $C_{24}H_{33}N_5O_4$
Exact Mass: 455.25
Molecular Weight: 455.55
m/z: 455.25 (100.0%), 456.26 (26.5%), 457.26 (4.2%), 456.25 (1.8%)
Elemental Analysis: C, 63.28; H, 7.30; N, 15.37; O, 14.05

ESI : [M+H] 456.3

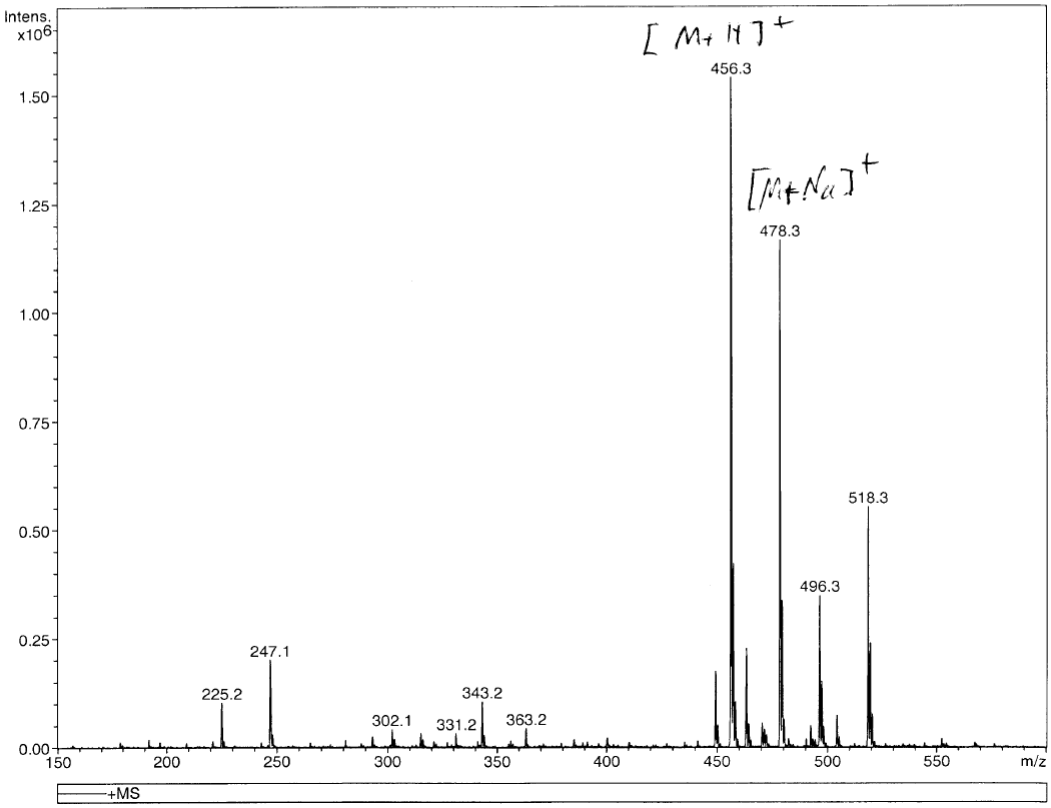
Display Report

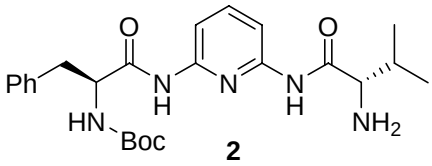
Analysis Info

Analysis Name	XZ3-1070.d	Acquisition Date	03/10/09 14:47:03
Method	XQ Default.ms	Operator	Administrator
Sample Name	XZH3-107	Instrument	Esquire-LC_00137
Comment	Diluted 1/100 in MEOH.		

Acquisition Parameter

Ion Source Type	ESI	Ion Polarity	Positive	Alternating Ion Polarity	n/a
Mass Range Mode	Std/Normal	Scan Begin	150.00 m/z	Scan End	600.00 m/z
Capillary Exit	83.6 Volt	Skim 1	16.3 Volt	Trap Drive	35.4
Accumulation Time	851 µs	Averages	20 Spectra	Auto MS/MS	Off

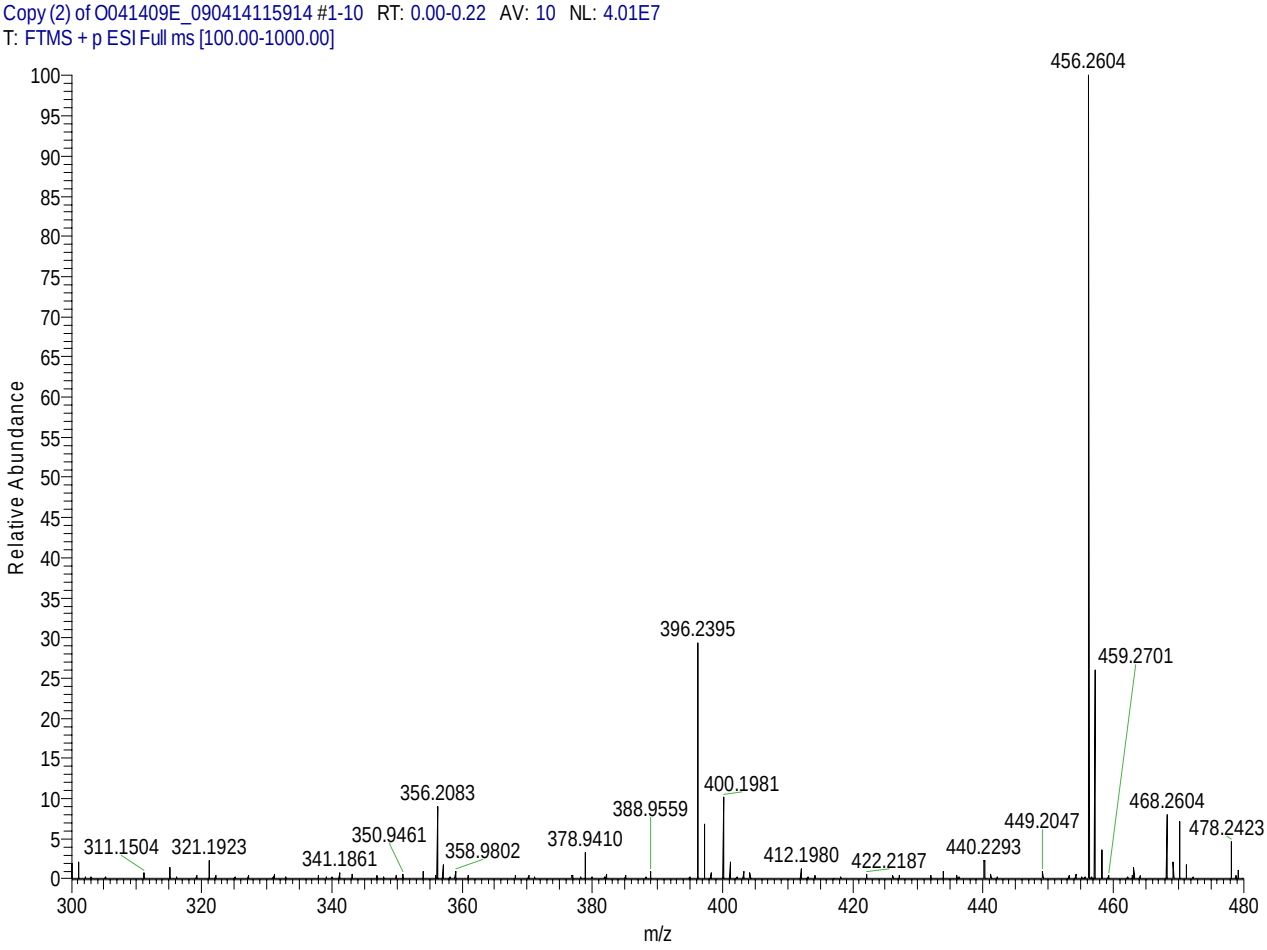


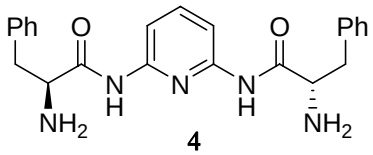


2

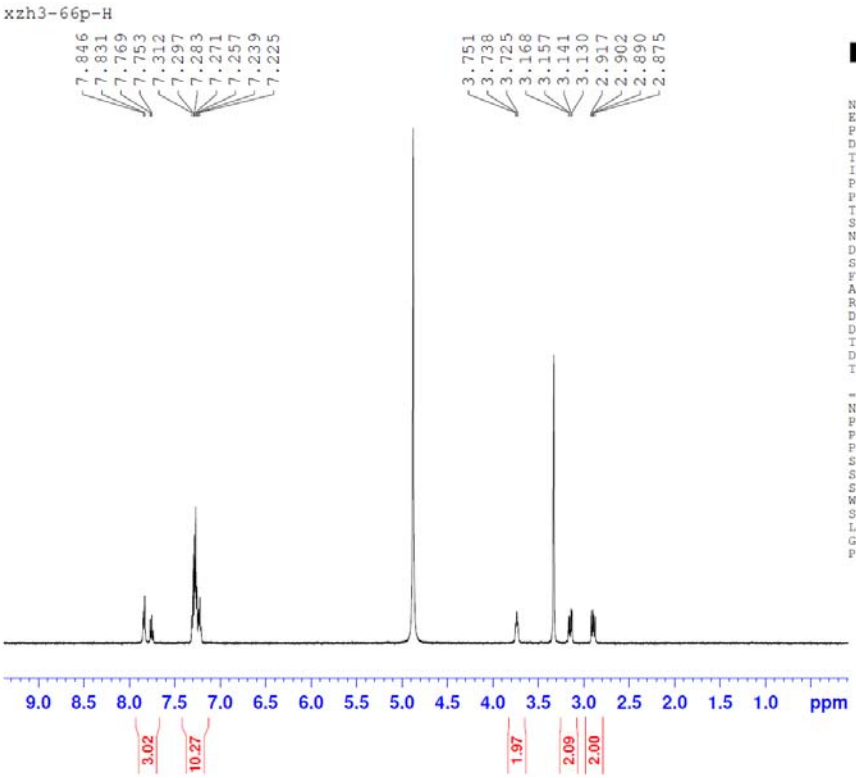
HRMS

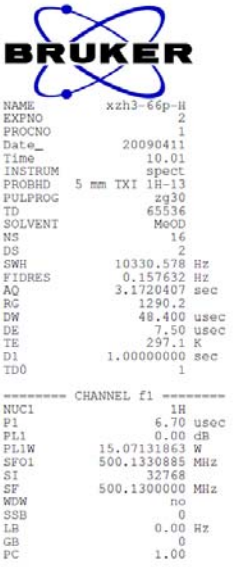
Calculated mass H+ 456.2611
Measured mass 456.2604
1.5 ppm

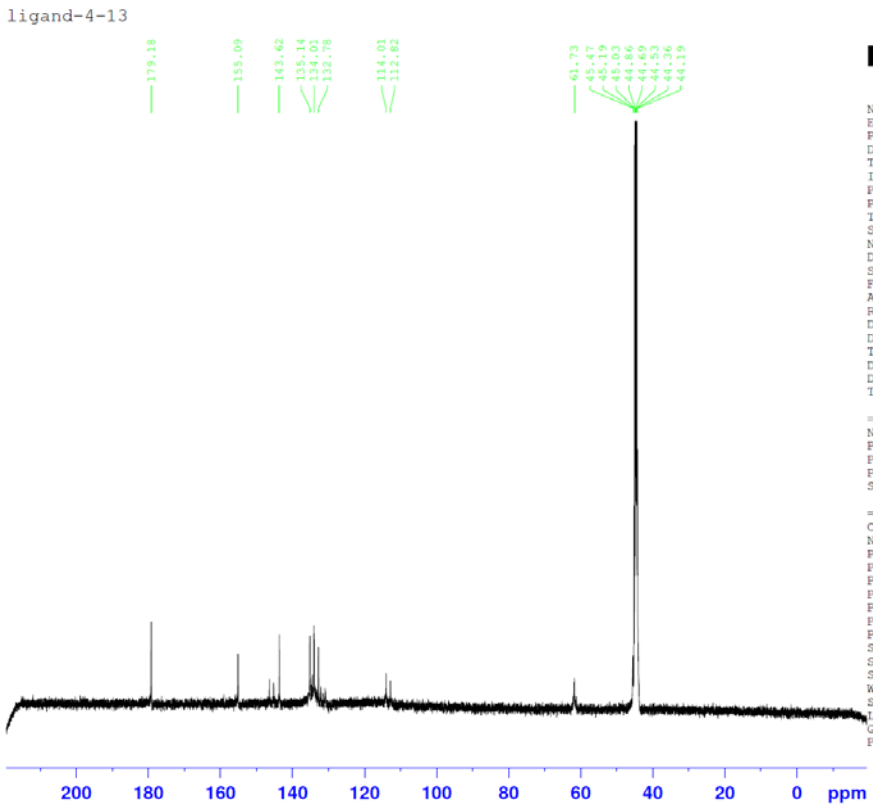


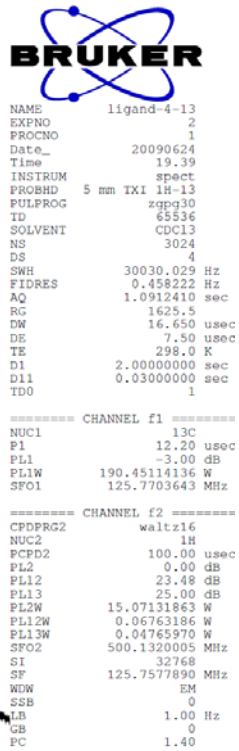

Ph
O
N
H
N
N
H
O
Ph
NH2
NH2

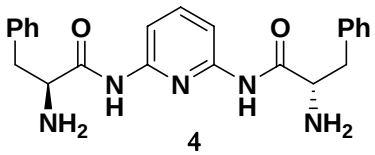
4


xzh3-66p-H
7.846
7.831
7.769
7.753
7.312
7.297
7.283
7.271
7.257
7.239
7.225
3.751
3.738
3.725
3.168
3.157
3.141
3.130
2.917
2.902
2.890
2.875
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 ppm
3.02
10.27
1.97
2.09
2.00


BRUKER
NAME xzh3-66p-H
EXPNO 2
PROCNO 1
Date_ 20090411
Time 10.01
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zg30
TD 65536
SOLVENT MeOD
NS 16
DS 2
SWH 10330.578 Hz
FIDRES 0.157632 Hz
AQ 3.1720407 sec
RG 1290.2
DW 48.400 usec
DE 7.50 usec
TE 297.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 6.70 usec
PL1 0.00 dB
PL1W 15.07131863 W
SFO1 500.1330885 MHz
SI 32768
SF 500.1300000 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00


ligand-4-13
179.18
155.09
143.62
135.14
134.01
132.78
114.01
112.82
61.73
45.47
45.19
45.03
44.86
44.69
44.53
44.36
44.19
200 180 160 140 120 100 80 60 40 20 0 ppm

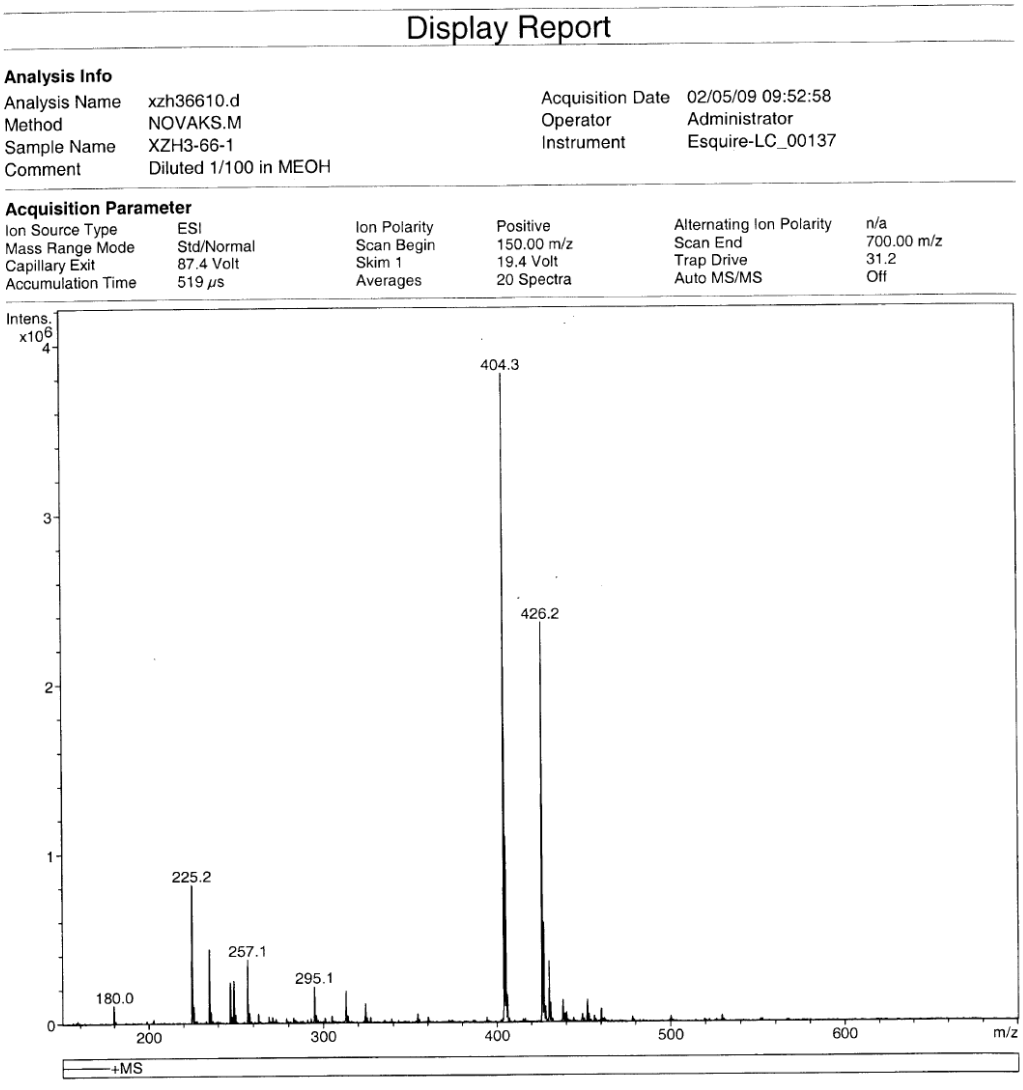

BRUKER
NAME ligand-4-13
EXPNO 2
PROCNO 1
Date_ 20090624
Time 19.39
INSTRUM spect
PROBHD 5 mm TXI 1H-13
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 3024
DS 4
SWH 30030.029 Hz
FIDRES 0.458222 Hz
AQ 1.0912410 sec
RG 1625.5
DW 16.650 usec
DE 7.50 usec
TE 298.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 12.20 usec
PL1 -3.00 dB
PL1W 190.45114136 W
SFO1 125.7703643 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 0.00 dB
PL12 23.48 dB
PL13 25.00 dB
PL2W 15.07131863 W
PL12W 0.06763186 W
PL13W 0.04765970 W
SFO2 500.1320005 MHz
SI 32768
SF 125.7577890 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

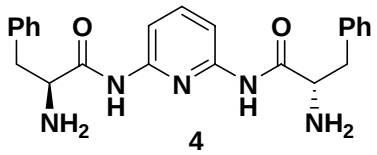


4

Chemical Formula: $C_{23}H_{25}N_5O_2$
Exact Mass: 403.2
Molecular Weight: 403.48
m/z: 403.20 (100.0%), 404.20 (26.7%), 405.21 (3.5%)
Elemental Analysis: C, 68.47; H, 6.25; N, 17.36; O, 7.93

ESI : $[M+H]^+$ 404.3

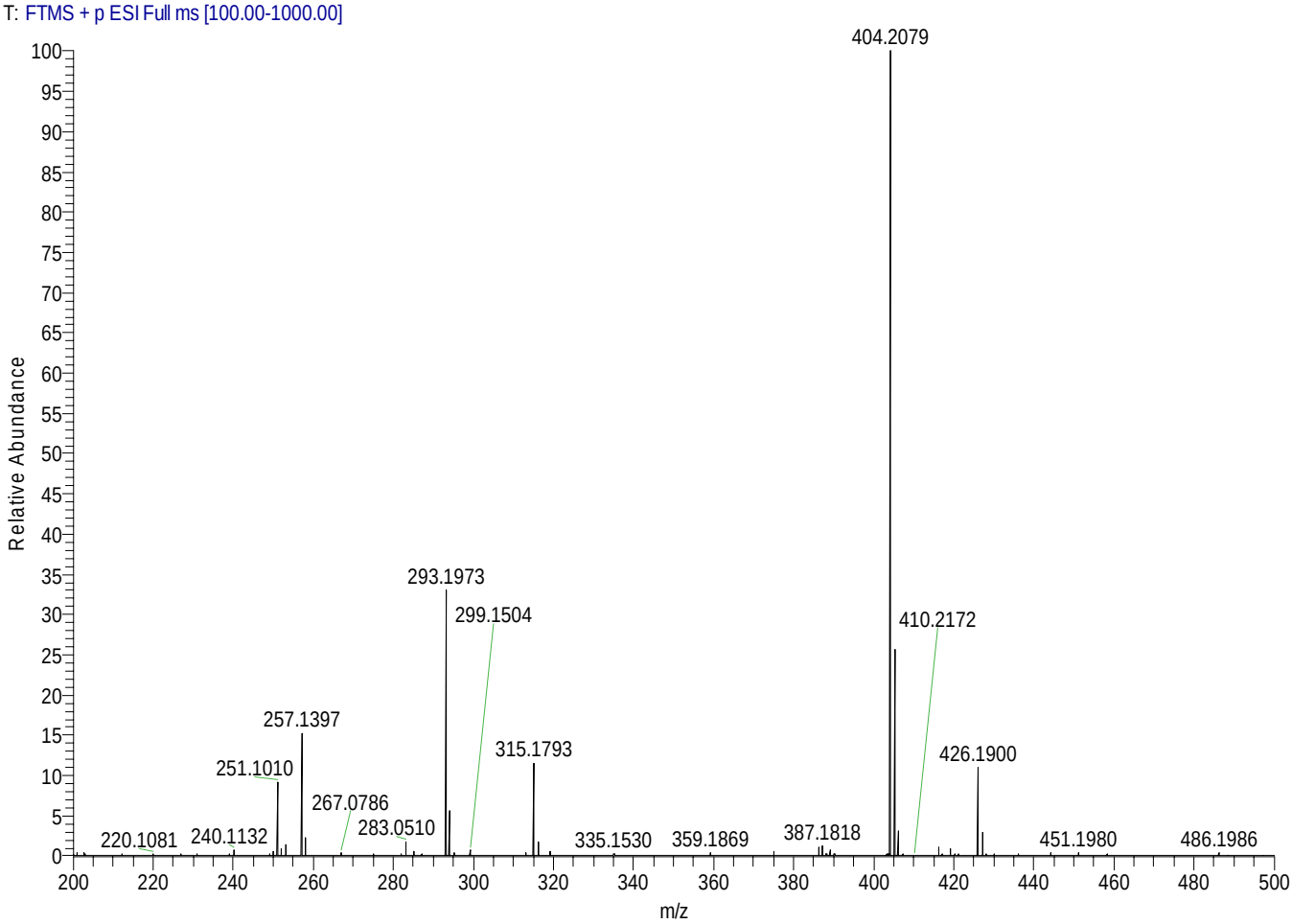




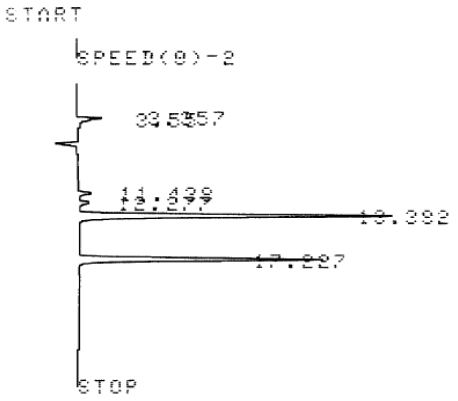
4

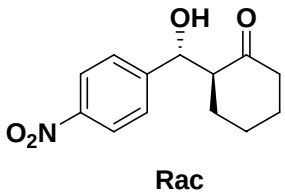
HRMS

Calculated mass H+ 404.2086
Measured mass 404.2079
1.7 ppm



HPLC spectra for the aldol products

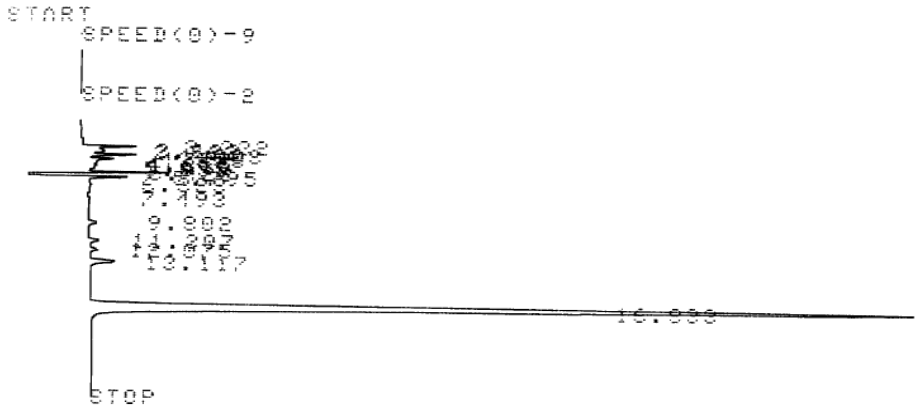


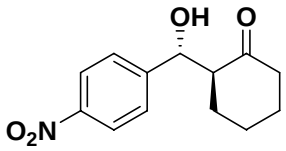


Rac

CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 668

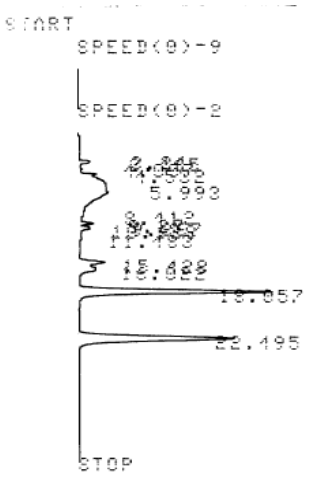
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	13.392	211929			49.9463	
2	17.227	212376			50.0537	
	TOTAL	424296			100	

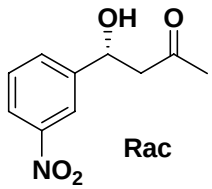




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 656

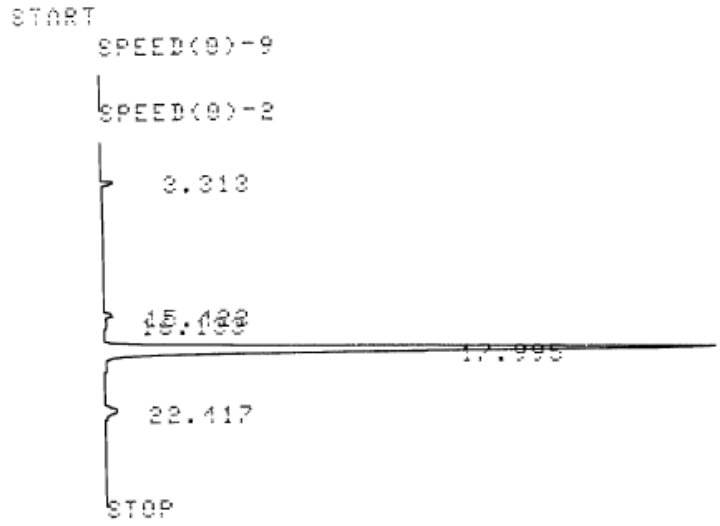
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.332	19999			1.9392	
2	3.622	11968	V		1.1597	
3	3.763	20010	V		1.9402	
4	4.008	42382	V		4.1018	
5	4.339	53533	V		5.1908	
6	4.655	19592	V		1.8997	
7	4.815	49870	V		4.8356	
8	5.005	53769	V		5.2155	
9	5.429	56375	V		5.4664	
10	5.795	49934	E		4.7449	
11	13.117	16132			1.5643	
12	16.933	638905			61.9599	
	TOTAL	1031309			100	

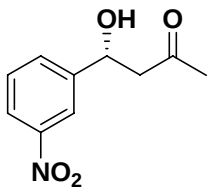




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 687

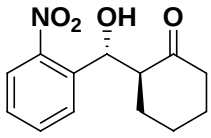
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	5.993	147740	V		25.6636	
2	9.27	11499	V		1.9974	
3	15.429	22791			3.9591	
4	16.022	22750	V		3.9519	
5	18.057	185944			32.2927	
6	22.495	185053			32.1453	
	TOTAL	575679			100	



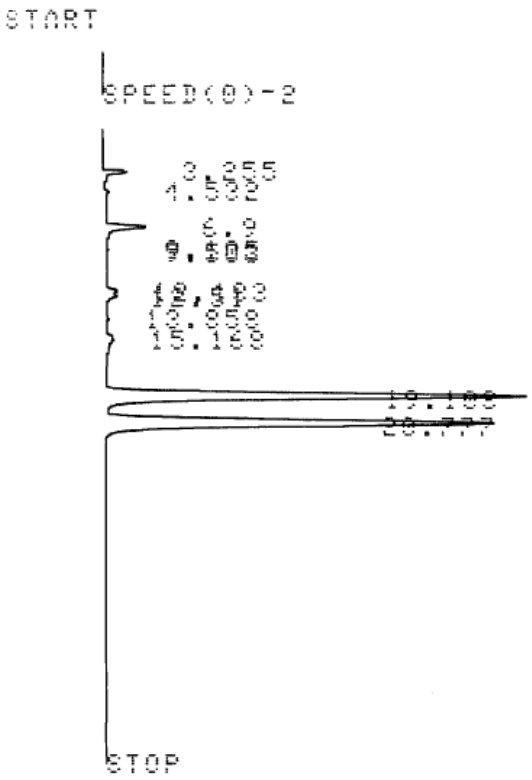


CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 689

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	17.995	471461			97.3467	
2	22.417	12850			2.6532	
	TOTAL	484311			100	

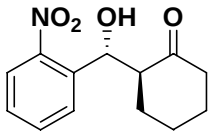


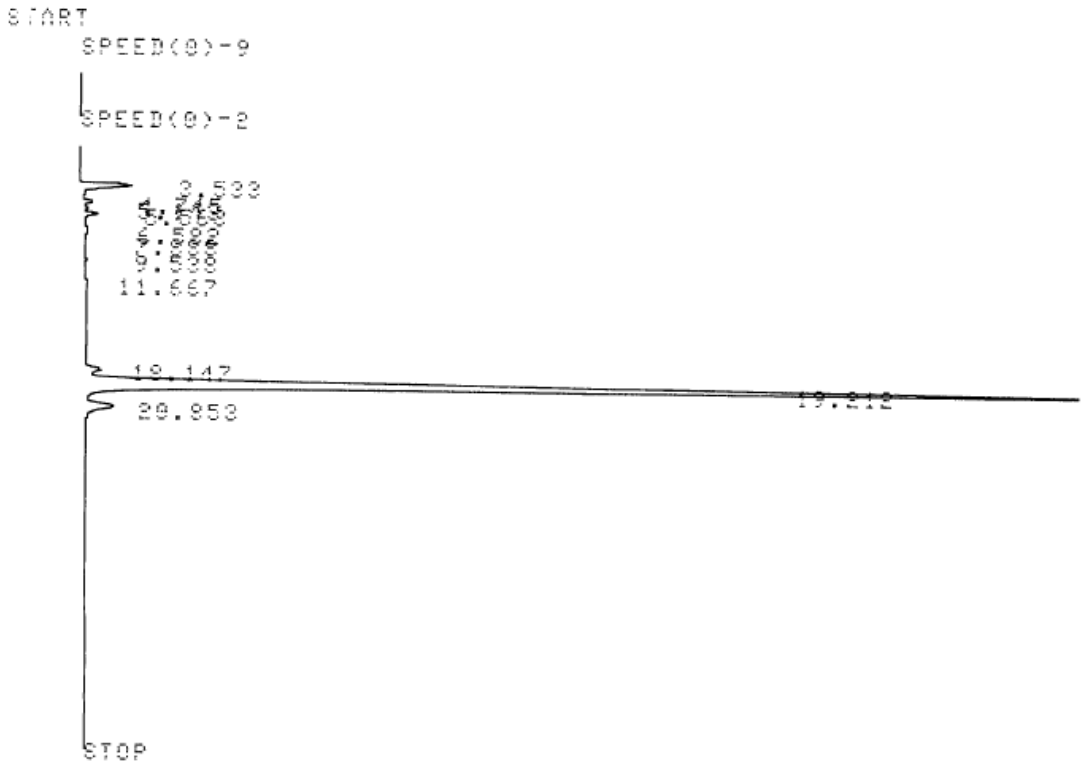
Rac



CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 670

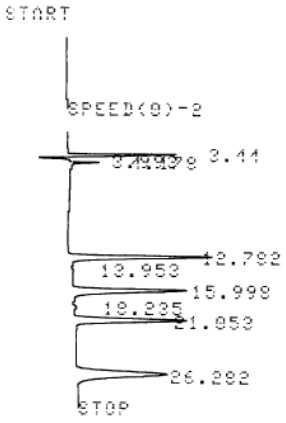
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	6.9	24011			3.549	
2	19.109	326167			48.2038	
3	20.777	326463	V		48.2473	
	TOTAL	676643			100	

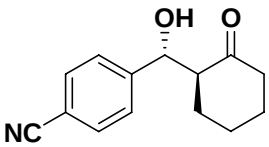




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 669

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.533	36752			3.4775	
2	18.147	16656			1.576	
3	19.212	972709	V		92.0379	
4	20.853	30739	V		2.9085	
	TOTAL	1056856			100	

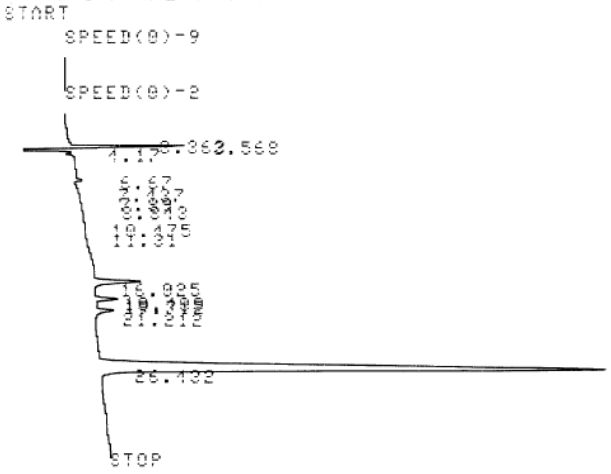


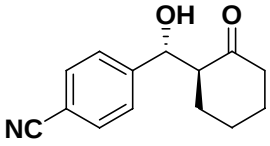


Rac

CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 708

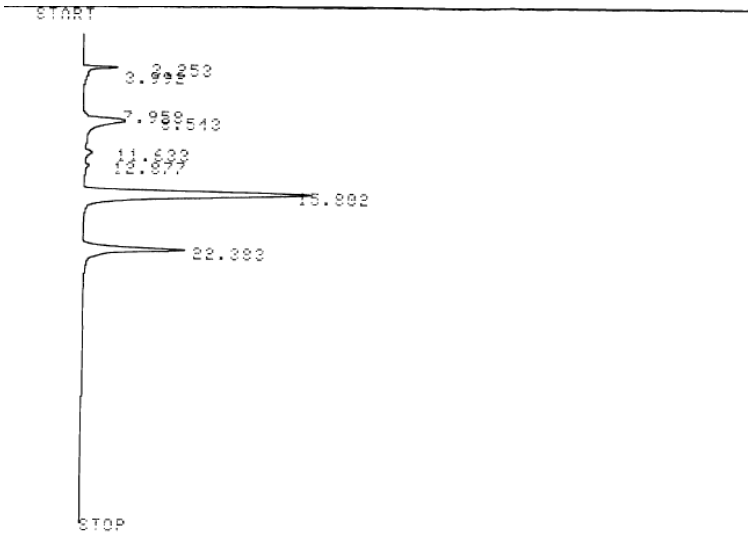
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.44	59975			10.4146	
2	3.993	12367			2.1511	
3	4.179	13935	V		2.4239	
4	12.782	109446			19.062	
5	15.998	112635			19.5916	
6	21.053	133318			23.1992	
7	26.282	134339			23.3666	
	TOTAL	574916			100	

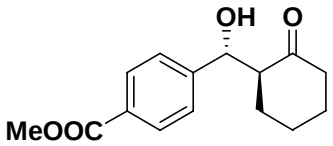




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 707

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.568	77995	V		6.7236	
2	4.17	22133			1.9092	
3	10.475	20390	V		1.759	
4	11.31	13665	V		1.1781	
5	16.025	152124	V		13.1155	
6	19.408	58219	V		5.0193	
7	19.712	20603	V		1.7763	
8	21.212	32226	V		2.7784	
9	26.432	762534	V		65.7426	
	TOTAL	1159879			100	



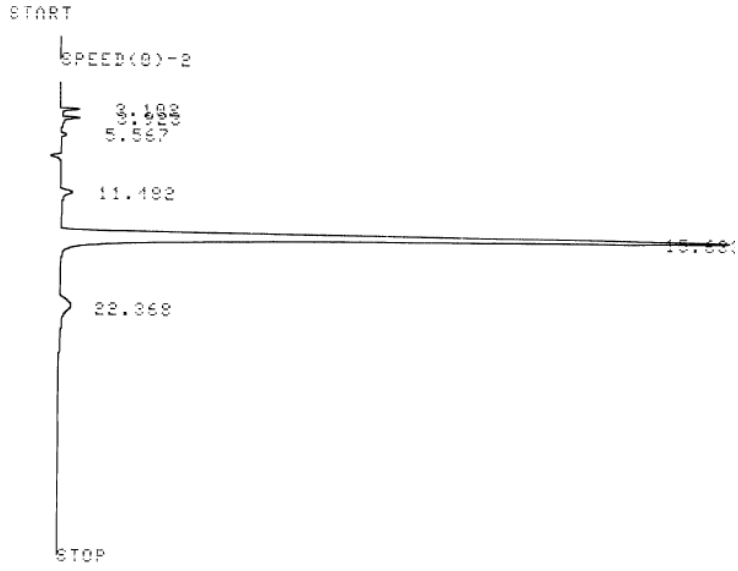


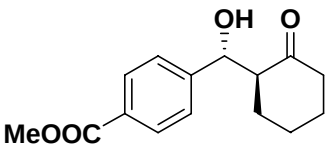
rac

CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 679

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.253	21175			2.0307	
2	8.543	95956	V		9.2433	
3	11.633	10270			0.9849	
4	15.902	463386			44.4393	
5	22.383	461951			44.3017	
	TOTAL	1042739			100	

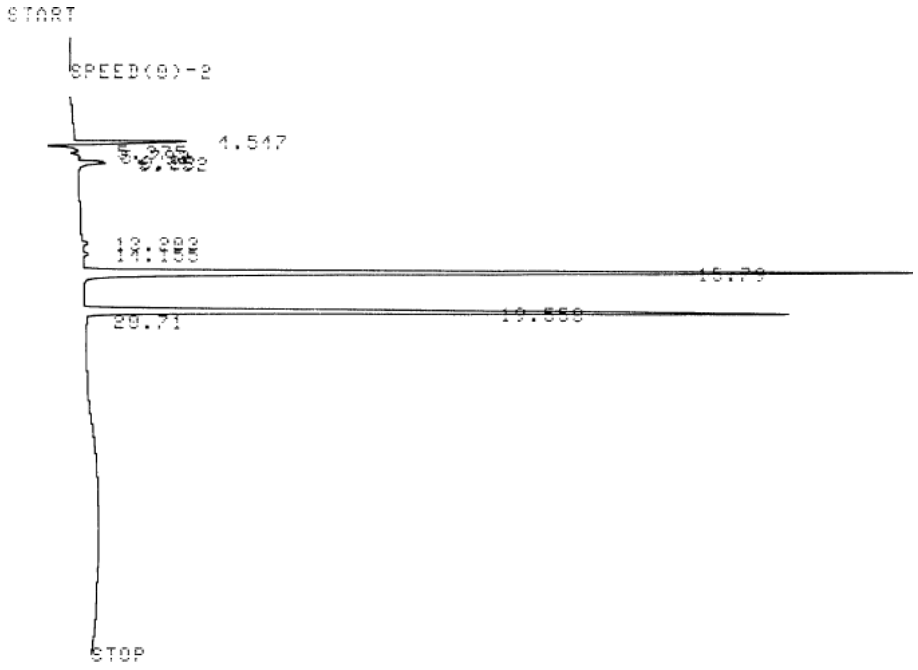


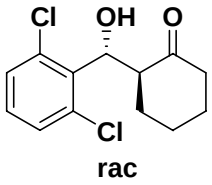


CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 678

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.923	10599			0.7645	
2	11.492	13911			1.0043	
3	15.633	1306357			94.3141	
4	22.368	54256			3.9171	
	TOTAL	1385113			100	

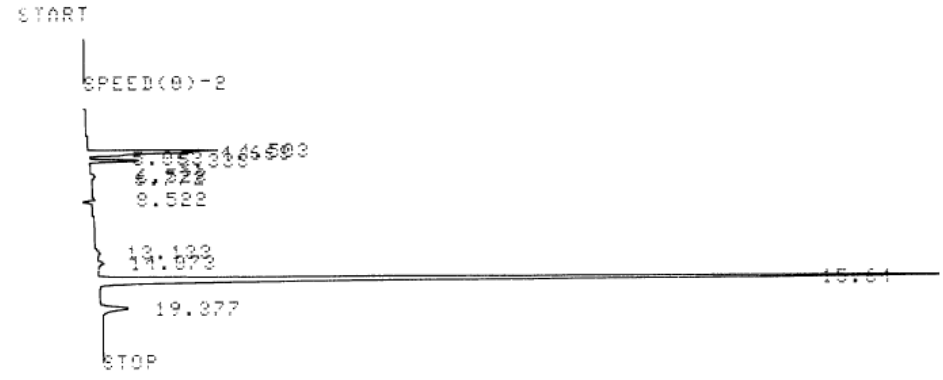


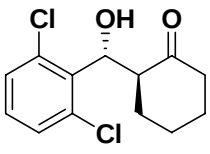


rac

CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 710

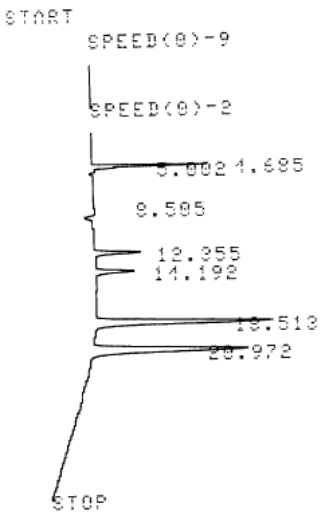
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.547	70668			4.5365	
2	5.075	29717			1.9178	
3	5.792	17500	V		1.1284	
4	6.29	20679	V		1.3275	
5	6.452	23924	V		1.5294	
6	15.79	698905			44.8673	
7	19.558	696422			44.7079	
	TOTAL	1557714			100	

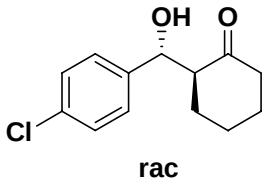




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 712

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.503	41947			1.9297	
2	4.668	52105	SV		6.0137	
3	5.333	25378	V		2.929	
4	15.61	719553			82.9311	
5	19.377	29562			3.2964	
	TOTAL	866446			100	



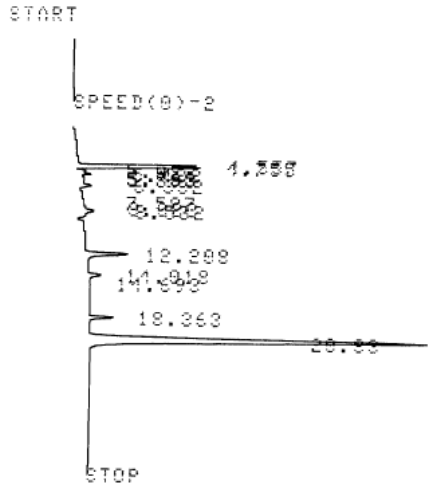


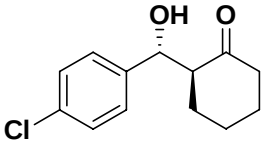
rac

CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 701

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.695	51369			11.9924	
2	12.355	29052			6.7822	
3	14.192	28574			6.6707	
4	18.513	159207			37.1674	
5	20.972	160149			37.3873	
	TOTAL	428351			100	

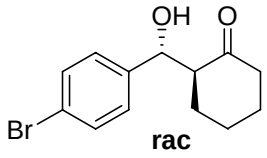




CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 700

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.557	38250			7.3458	
2	4.755	51485	V		9.8876	
3	8.532	17355	V		3.333	
4	12.208	28311			5.4371	
5	14.019	10003			1.9211	
6	18.363	23598			4.5319	
7	20.83	351701			67.5134	
	TOTAL	520704			100	



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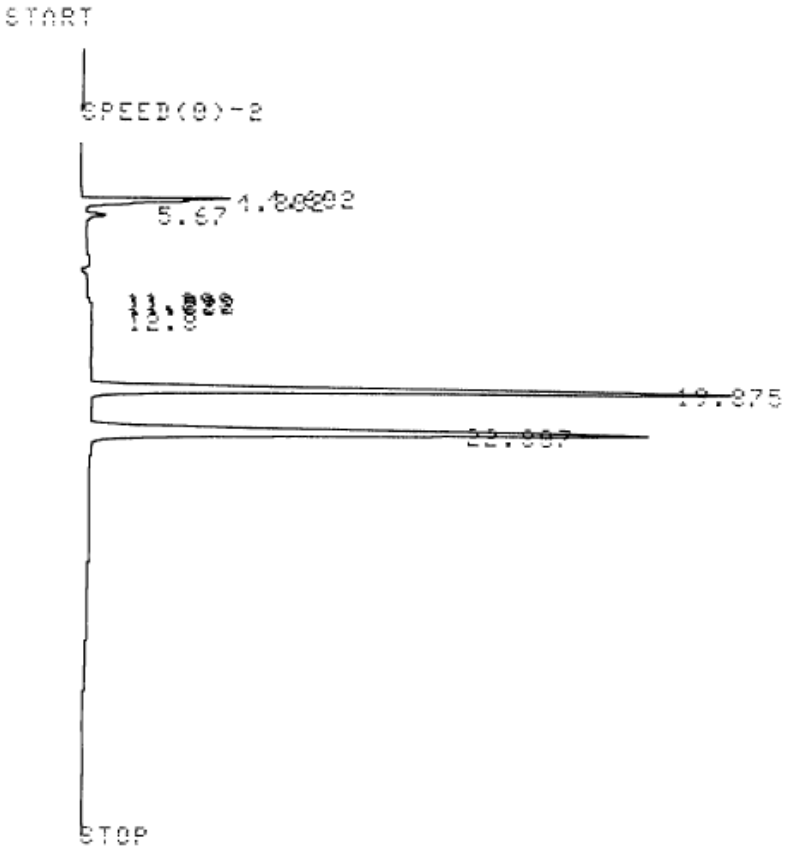
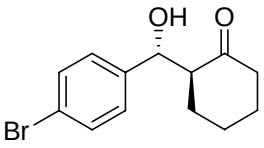
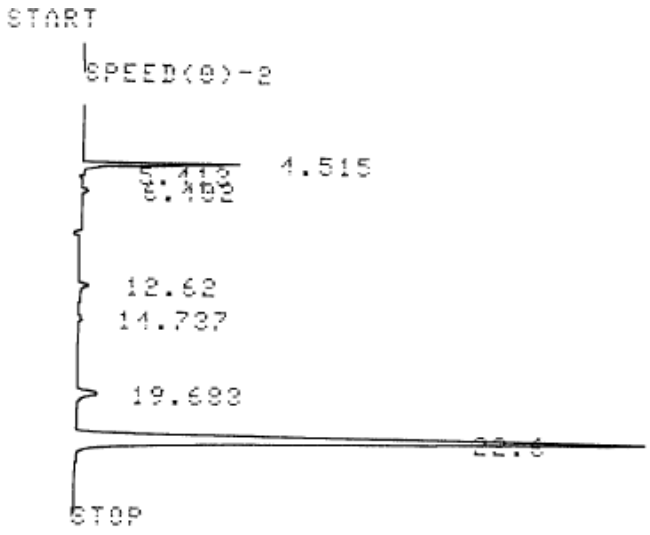


CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 702

FILE 0
METHOD 11

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.602	13149			1.0005	
2	4.802	10323	V		0.7925	
3	19.975	493750			45.0691	
4	22.807	499704			46.3296	
	TOTAL	1976426			100	

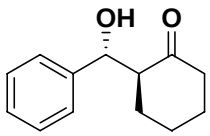




CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 704

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.515	71376			12.3619	
2	19.693	14683			2.5429	
3	22.6	491324			85.0951	
	TOTAL	577383			100	



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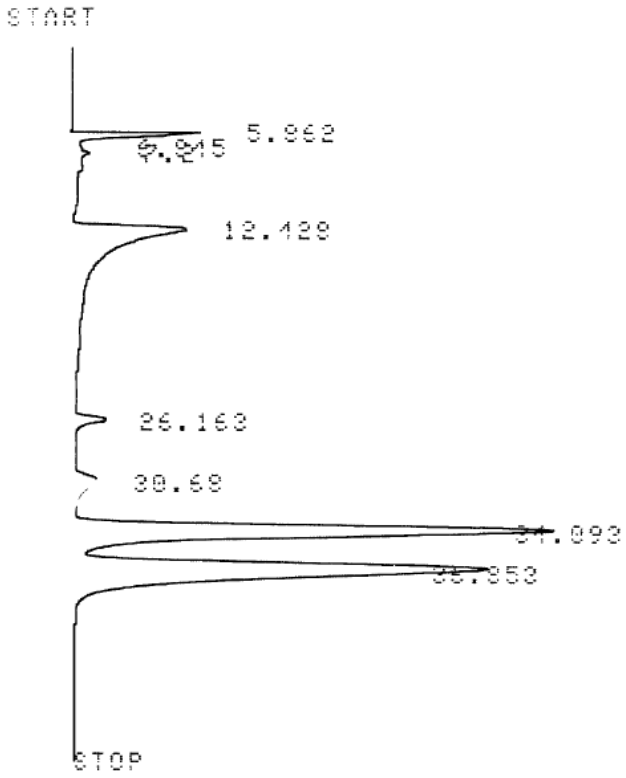
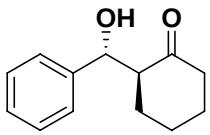
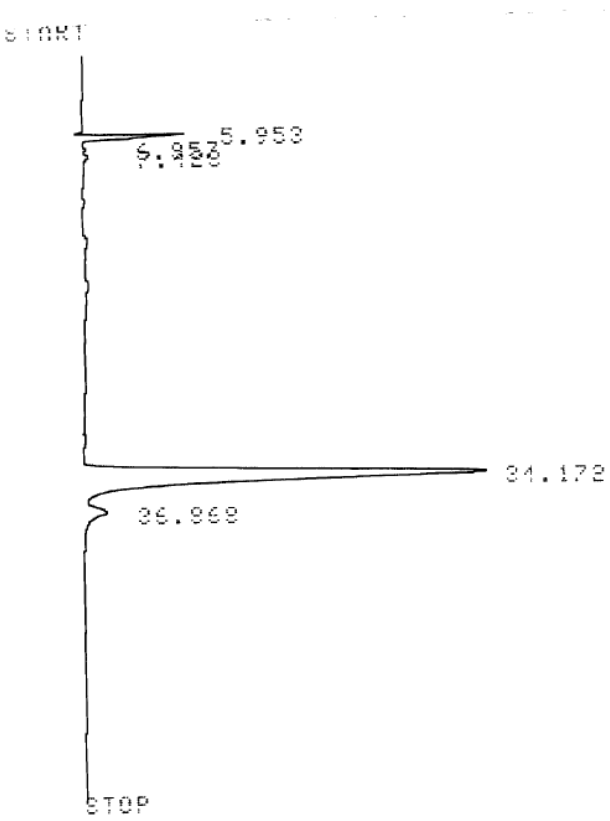


CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 730

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	5.962	90316			3.6093	
2	12.429	291039			12.6247	
3	26.163	49050			2.2031	
4	30.69	51929			2.3293	
5	34.093	999474	V		39.5525	
6	36.953	993351	V		39.6818	
	TOTAL	2226091			100	

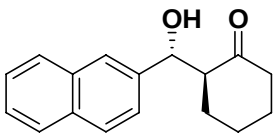




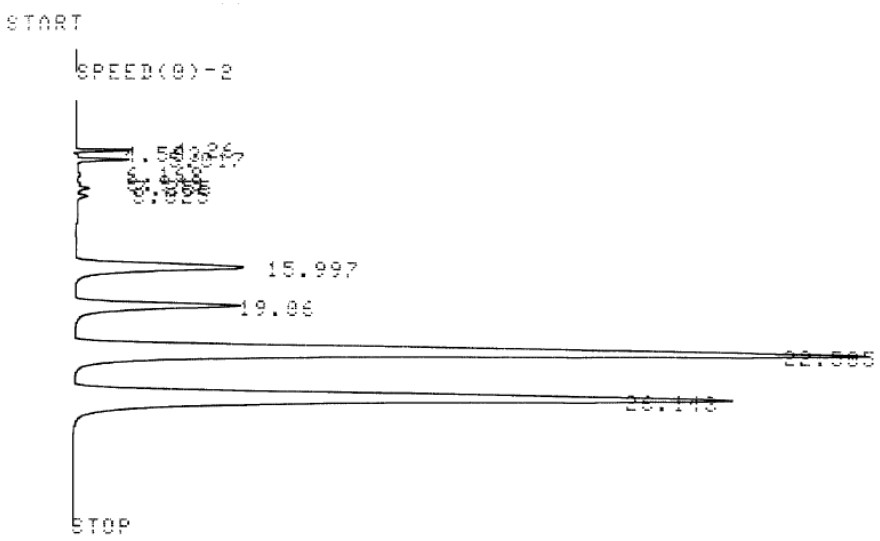
CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 729

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	5.953	56092			6.7365	
2	34.172	724203			86.9744	
3	36.969	52367	V		6.2892	
	TOTAL	832662			100	

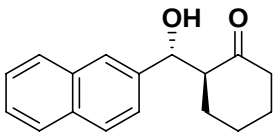


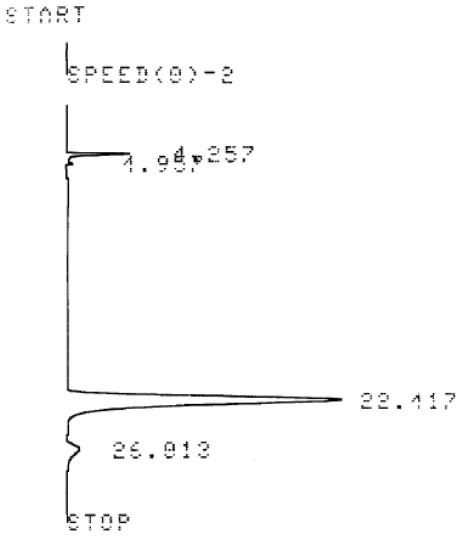
rac



CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 719

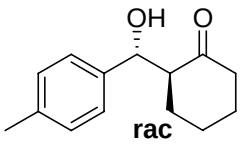
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.26	22557			0.7065	
2	5.017	24131	V		0.7559	
3	15.997	239326			7.4963	
4	19.06	248276			7.7767	
5	22.505	1331213			41.6972	
6	26.143	1327073	V		41.5675	
	TOTAL	3192576			100	

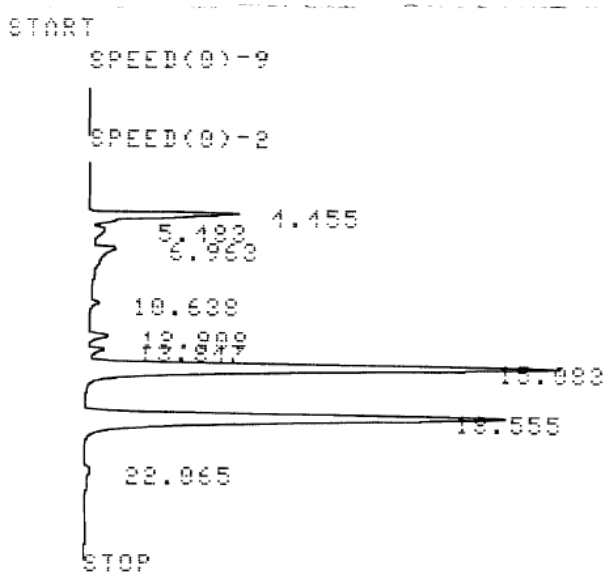




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 720

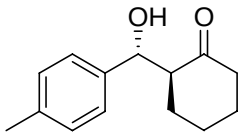
P<NO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.257	23115			4.9699	
2	22.417	419224			90.1159	
3	26.013	22967			4.9154	
	TOTAL	465206			100	

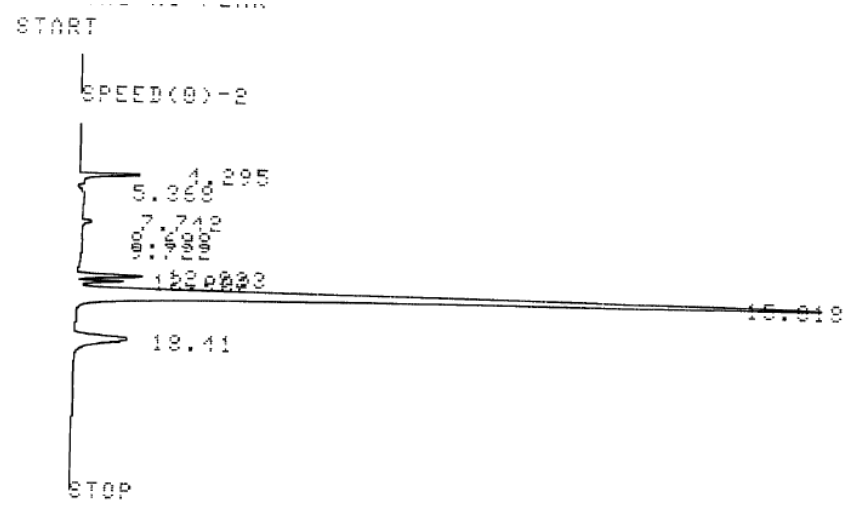




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 723

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.455	119907			9.4995	
2	5.493	13219	V		1.0559	
3	6.963	34891			2.7863	
4	12.909	16297			1.3019	
5	13.947	16019	V		1.2796	
6	15.093	521967	V		41.935	
7	18.555	527569			42.1429	
	TOTAL	1251959			100	

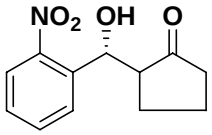




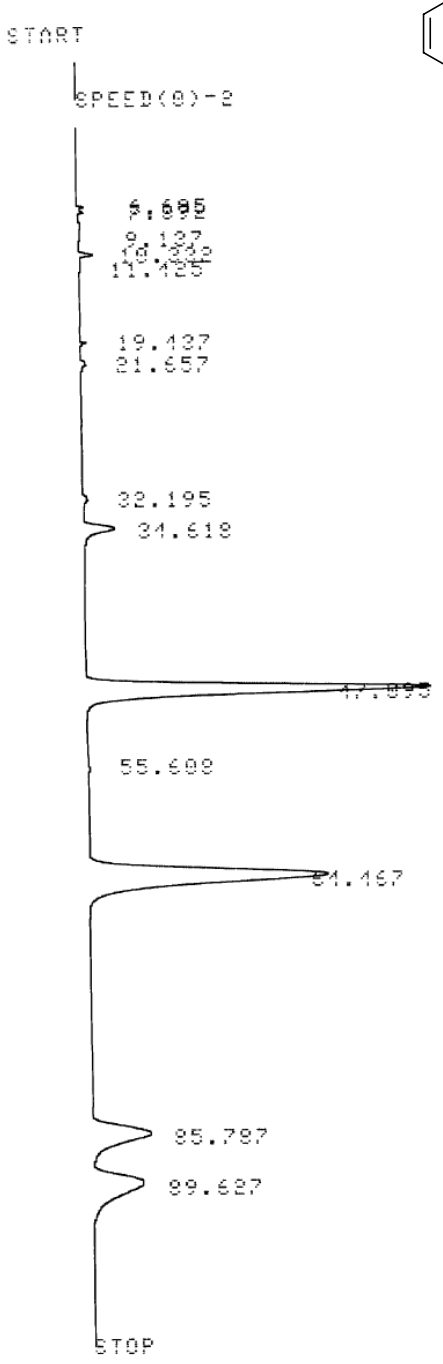
CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 724

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	1.295	28546			2.9225	
2	12.823	57201			5.8565	
3	13.902	45167	V		4.6242	
4	15.018	773995	V		79.2424	
5	18.41	71735			7.3443	
	TOTAL	976743			100	

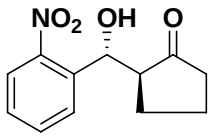


Rac



CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 769

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	34.618	44124			2.526	
2	47.095	643104			36.8327	
3	64.167	663279			37.9706	
4	85.787	196757			11.2637	
5	89.627	199261	V		11.407	
	TOTAL	1746925			100	



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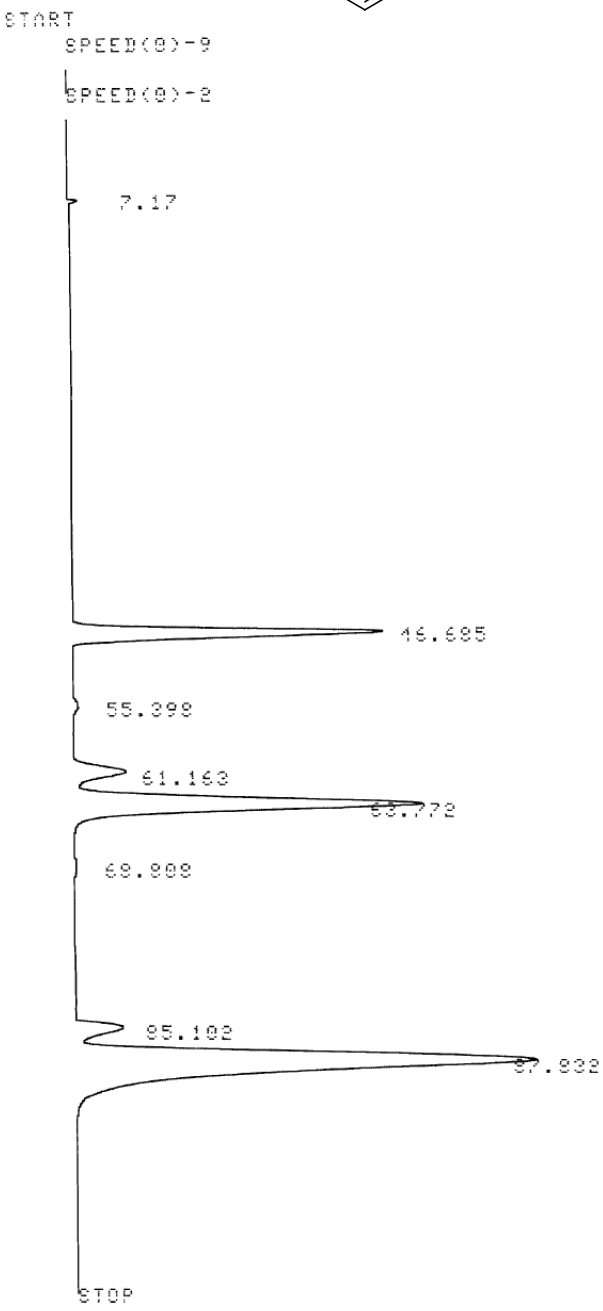
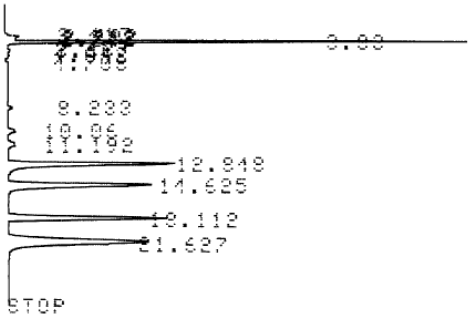


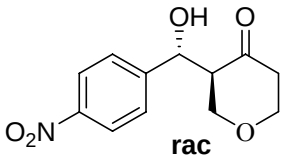
CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 767

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	46.685	619529			15.9314	
2	55.399	10959			0.2816	
3	61.163	135691			3.4891	
4	63.772	977931	V		25.1478	
5	69.809	11799	V		0.3031	
6	85.102	178504			4.5916	
7	87.832	1962339	V		50.4622	
	TOTAL	3888734			100	

START

SPEED(0)-2



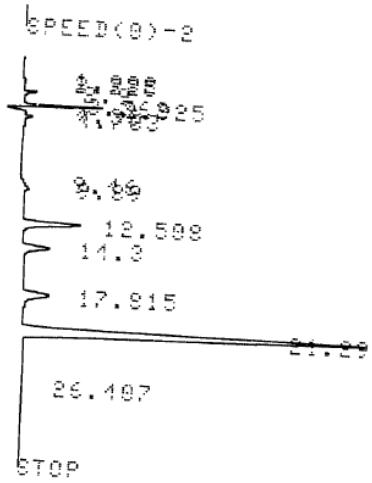


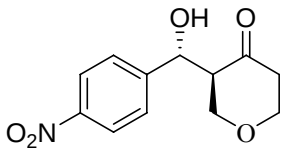
CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 749

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.03	138959	V		19.102	
2	12.948	125922			17.3918	
3	14.625	119452			16.5114	
4	18.112	170701			23.5953	
5	21.627	176519			24.3994	
	TOTAL	723451			100	

START

SPEED(0)-2

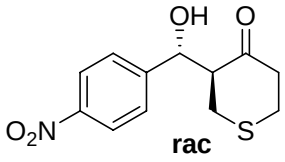




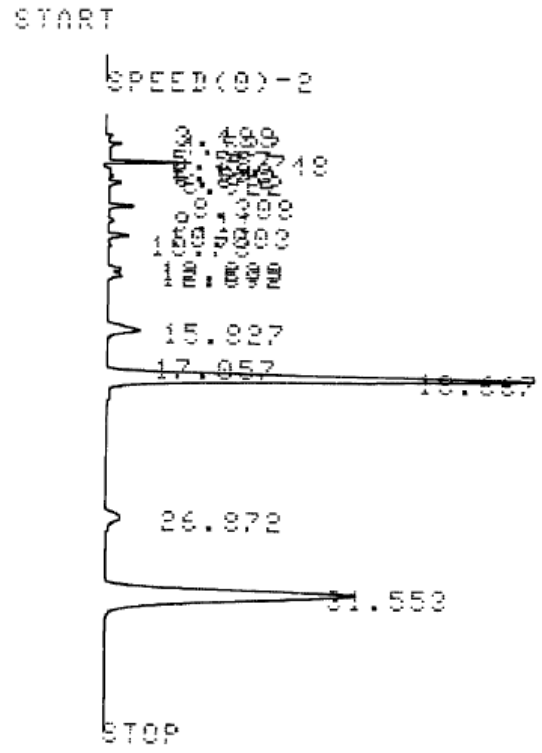
CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 747

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	3.925	20999	V		4.149	
2	12.508	42363			8.4149	
3	14.3	21435			4.2579	
4	17.915	27215			5.4119	
5	21.29	391509			77.7665	
	TOTAL	503449			100	

START



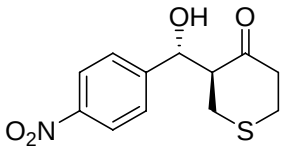
rac

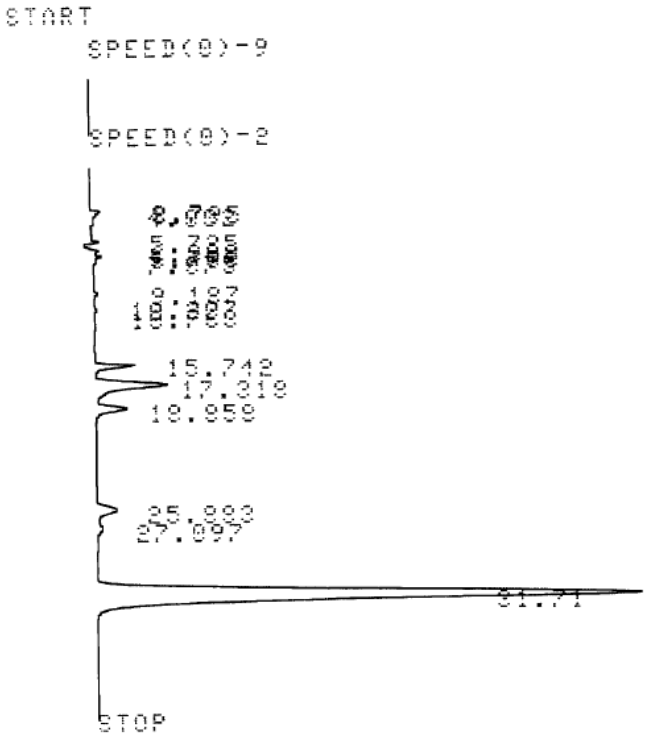


CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 753

FILE 0
METHOD 11

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	5.749	17655	V		2.5534	
2	15.927	26467			3.8279	
3	19.667	312731	V		45.2312	
4	26.972	17706			2.5609	
5	31.553	316847			45.8266	
	TOTAL	691405			100	

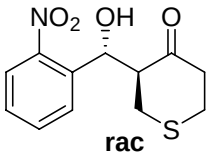




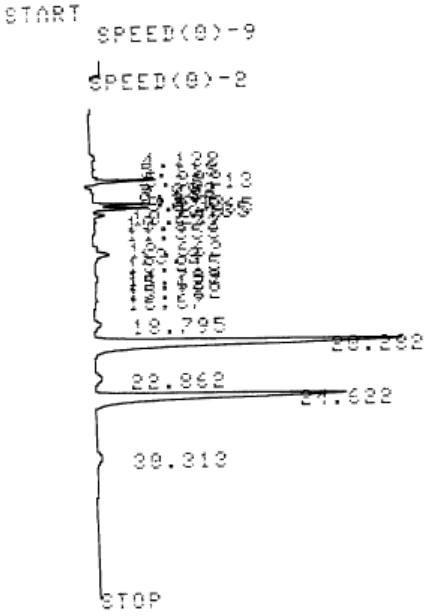
CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 754

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	15.742	39206			4.0566	
2	17.319	77402	V		8.2193	
3	18.959	26947	V		2.8611	
4	25.883	24076			2.5564	
5	31.71	775196			82.3076	
	TOTAL	941916			100	

T: 18.8 min / 31.7 min anti products (15.7, 17.3 syn products)



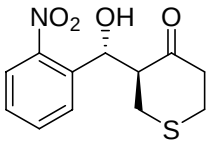
rac



CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 862

FILE 0
METHOD 41

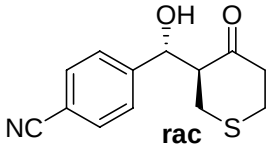
PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	6.13	29005	V		3.9952	
2	8.965	30695	V		1.1222	
3	9.135	35000	V		1.7014	
4	13.2	14559	V		1.955	
5	20.282	306737			41.1929	
6	22.862	10687			1.1352	
7	24.622	307535			41.2999	
8	30.313	10113			1.3993	
	TOTAL	744639			100	



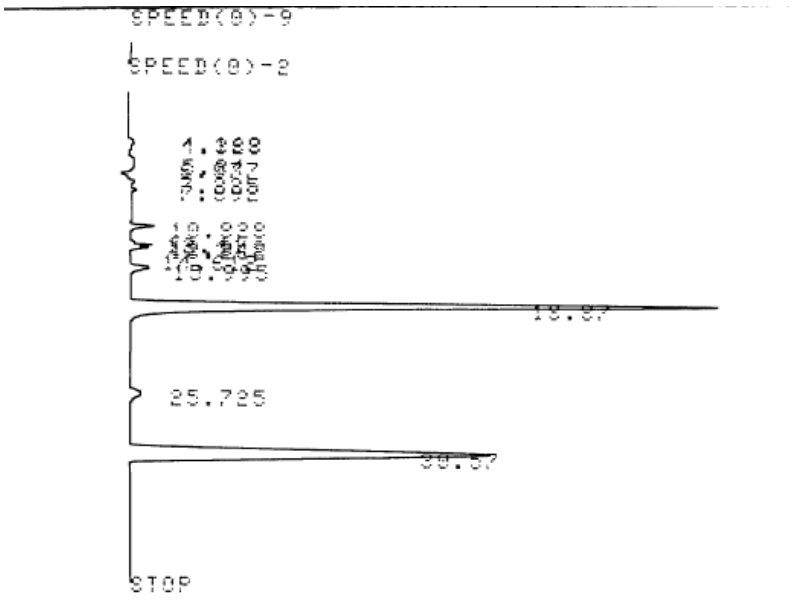
CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 867

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	6.079	28156	V		2.2099	
2	10.375	20709			1.6254	
3	20.457	35582			2.7991	
4	24.872	1189519			93.3656	
	TOTAL	1274076			100	

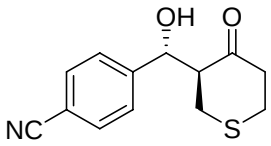


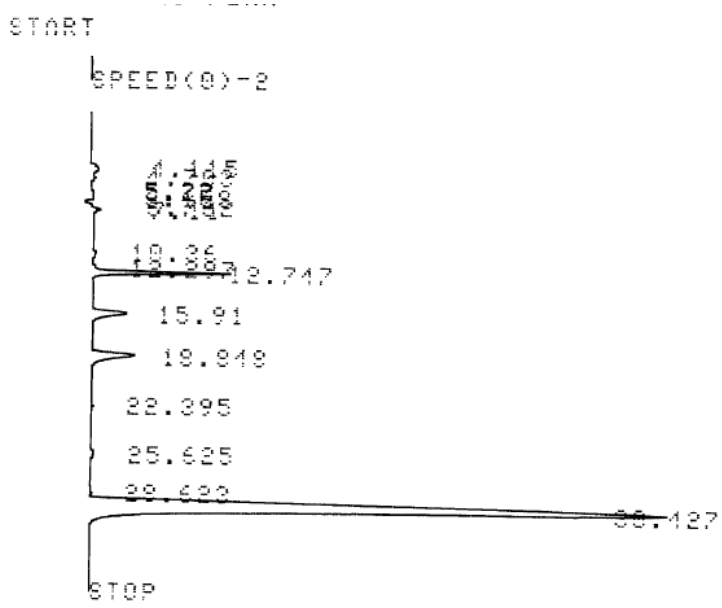
rac



CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 969

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	5.94	13791			1.0939	
2	7.225	12900			1.0233	
3	10.928	13885			1.1014	
4	12.942	12535	V		0.9943	
5	13.262	14420	V		1.1439	
6	15.995	17112			1.3573	
7	19.97	590209			46.8231	
8	25.725	15772			1.251	
9	30.57	590066			46.8119	
	TOTAL	1260699			100	

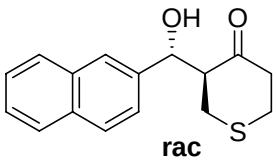




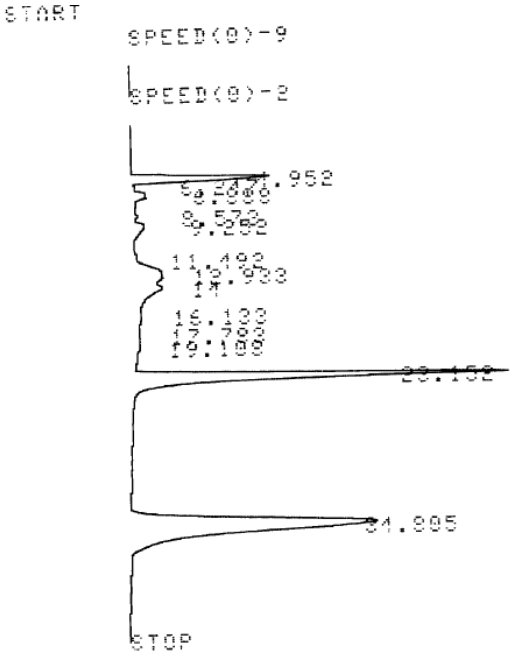
CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 869

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	12.747	69792	V		7.1226	
2	15.91	26104			2.664	
3	19.949	41224			4.207	
4	30.427	842749	V		86.0063	
	TOTAL	979869			100	



rac

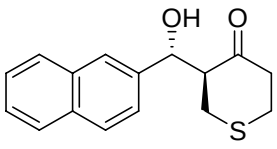


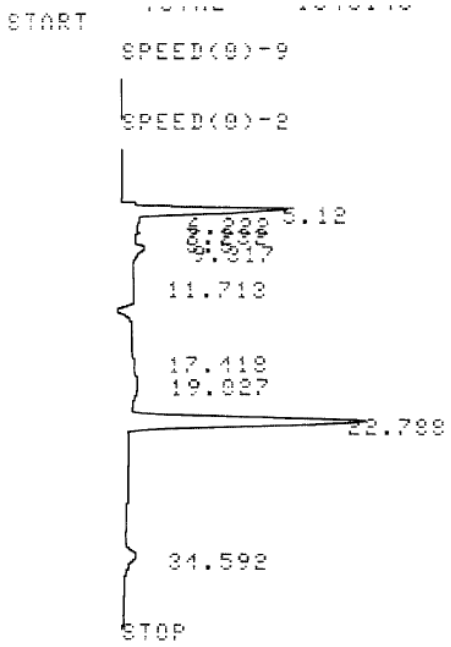
CHROMATOPAC C-R6A
SAMPLE NO 0
REPORT NO 10

FILE 0
METHOD 11

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.952	136651			8.2912	
2	6.999	20371	V		1.236	
3	9.252	22009	V		1.3353	
4	12.933	86029	V		5.2279	
5	11	68170	V		4.1362	
6	16.133	19759	V		1.1989	
7	17.793	10401	V		0.631	
8	23.152	636253			38.6042	
9	31.905	648204			39.3293	
	TOTAL	1648145			100	

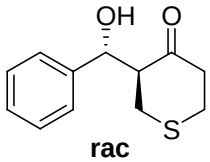
START



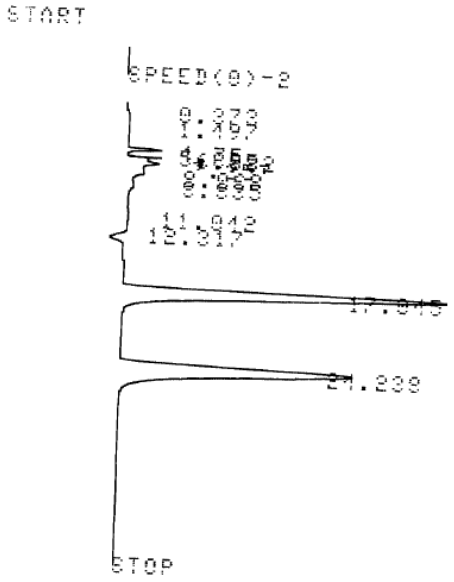


CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 11

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	5.12	170724			23.7926	
2	6.222	22517	V		3.1367	
3	7.232	19232	V		2.6791	
4	8.6	16307	V		2.2717	
5	9.317	24006	V		3.3442	
6	19.027	17073			2.3784	
7	22.799	419612			58.4446	
8	34.592	29390			4.0929	
	TOTAL	717861			100	



rac

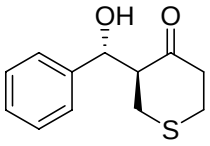


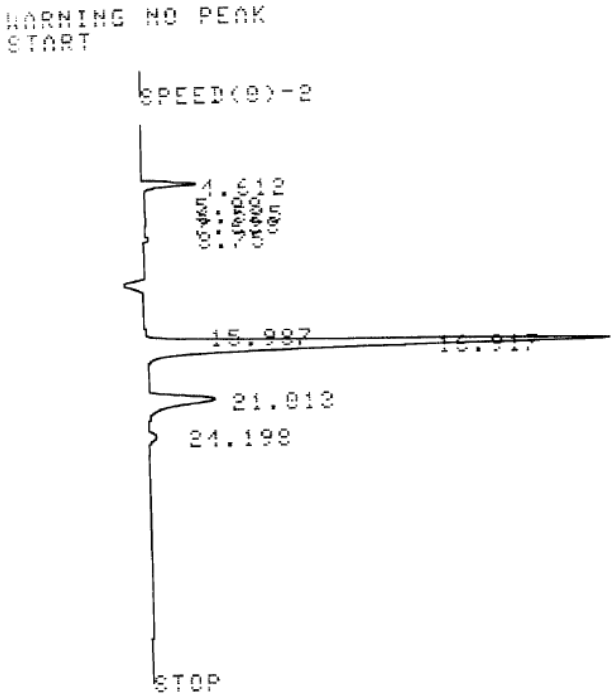
CHROMATOPAC C-R6A

SAMPLE NO 0
REPORT NO 16

FILE 0
METHOD 41

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.75	27791			2.6137	
2	6.582	25094	V		2.36	
3	7.357	11726	V		1.2065	
4	8.969	36160	V		3.4008	
5	9.935	37417	V		3.5219	
6	11.042	57559	V		5.4134	
7	12.317	13902	V		1.3074	
8	17.045	413619			38.9084	
9	24.239	406978			38.2759	
	TOTAL	1063273			100	

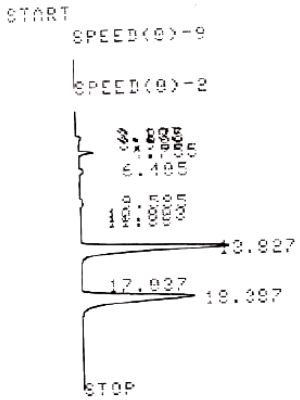


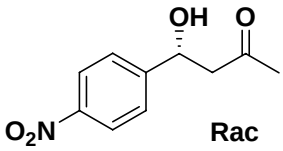


CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 14

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.612	36660			5.2517	
2	16.917	540191	V		77.427	
3	21.013	105291			15.0932	
4	24.198	15923			2.282	
	TOTAL	698066			100	

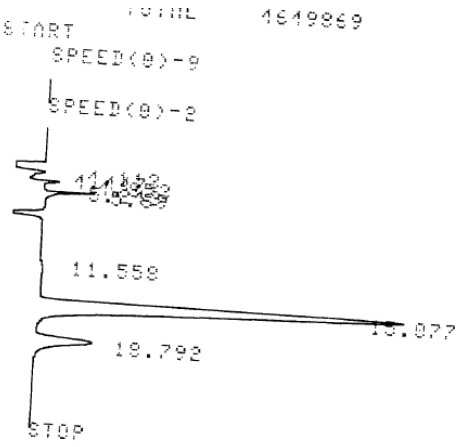
T: 16.9 min and 24.1 min

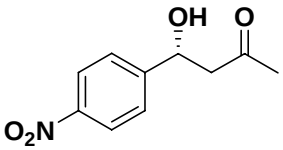




CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 393

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	12.927	446044			50.0505	
2	18.387	445143	V		49.9495	
	TOTAL	891187			100	





CHROMATOPAC C-R6A FILE 0
SAMPLE NO 0 METHOD 41
REPORT NO 600

PKNO	TIME	AREA	MK	IDNO	CONC	NAME
1	4.113	45574				
2	4.447	12423	V		1.4329	
3	4.953	35569	V		1.4909	
4	5.243	16825	V		4.21	
5	5.69	36438	V		2.0056	
6	15.077	590488			4.3427	
7	18.792	101565			70.3998	
	TOTAL	838971			12.1961	
					100	

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