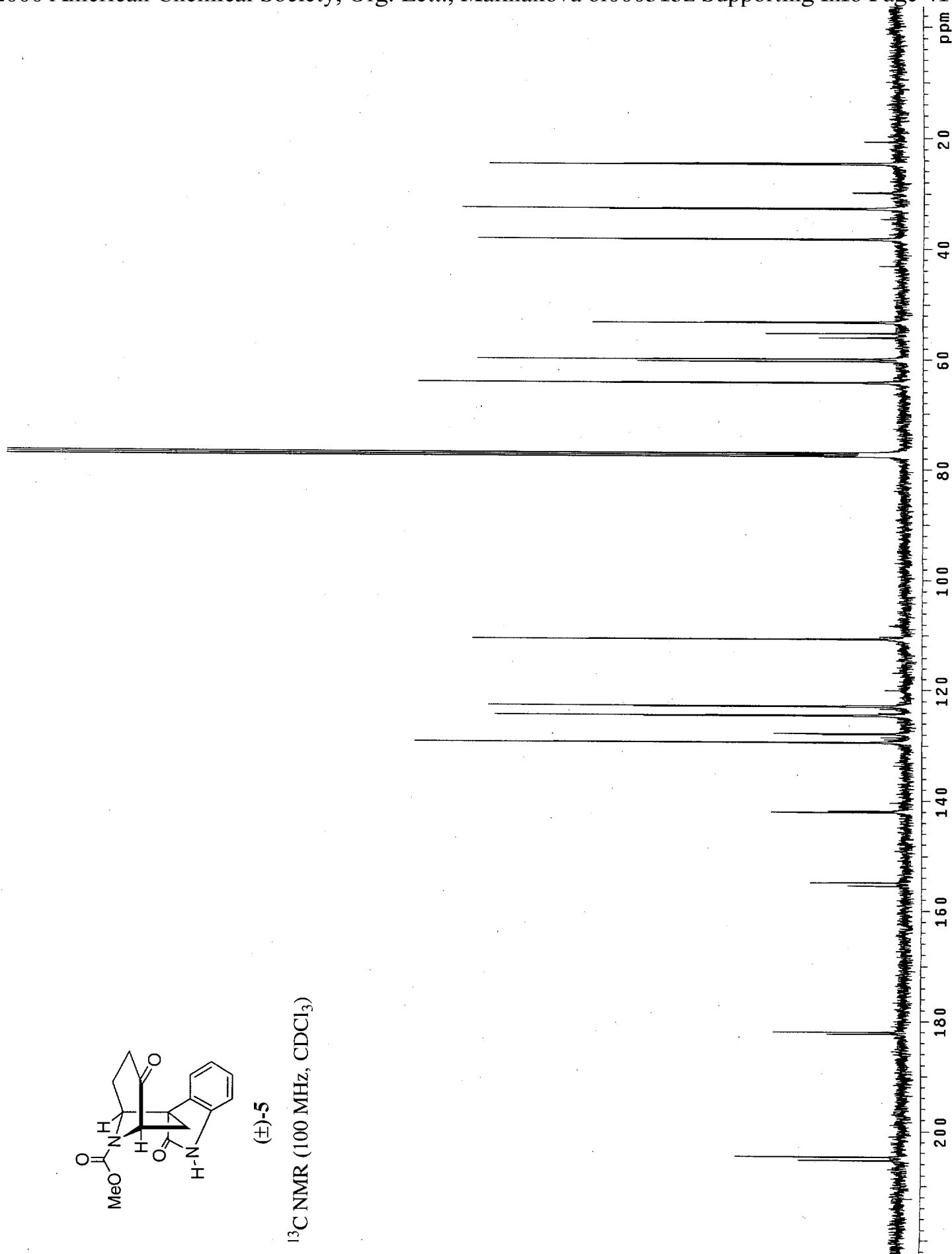
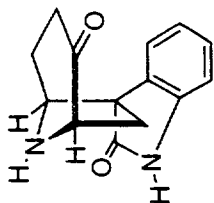


(±)-5

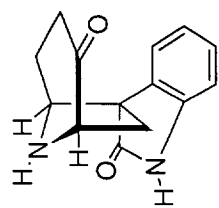
^{13}C NMR (100 MHz, CDCl_3)



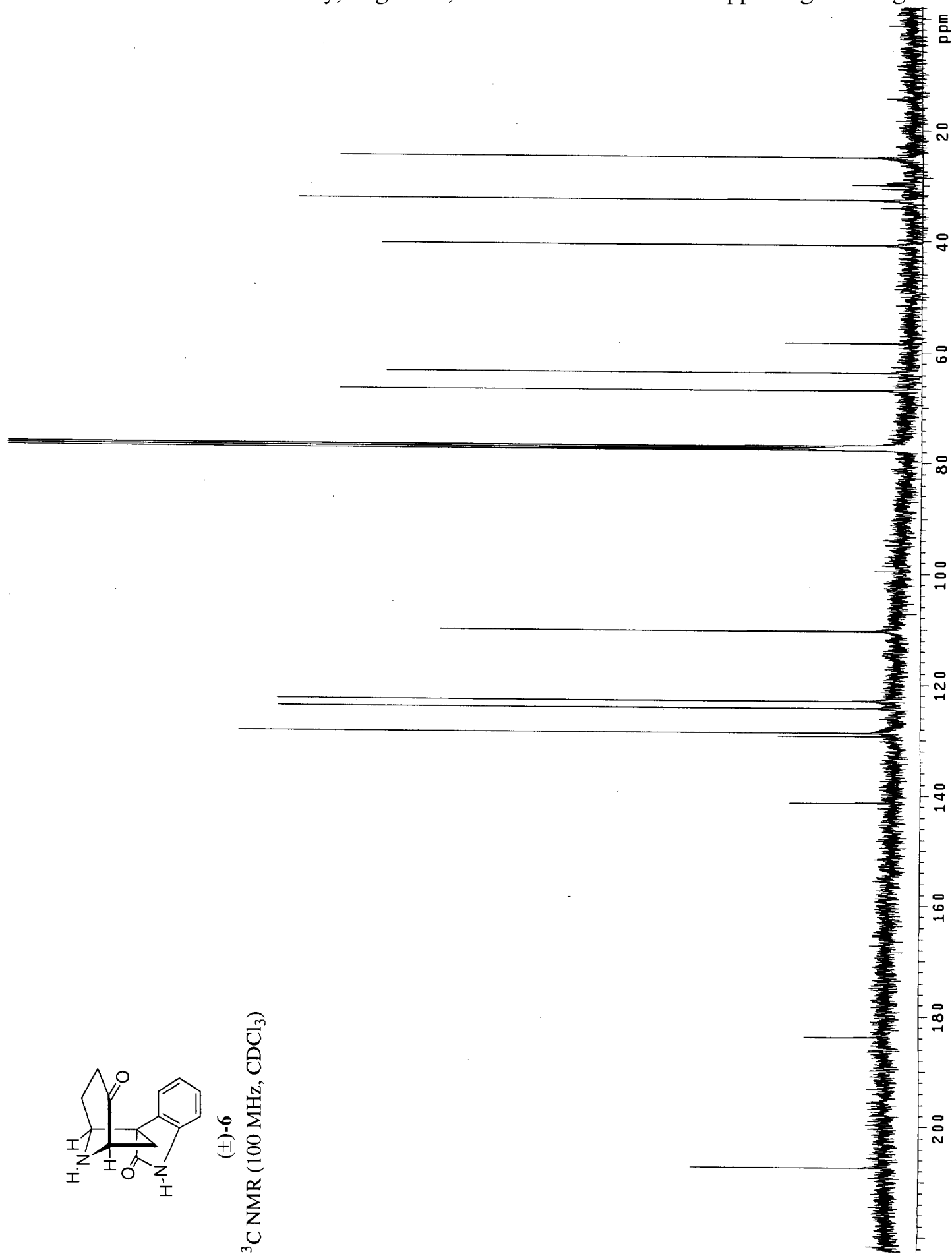


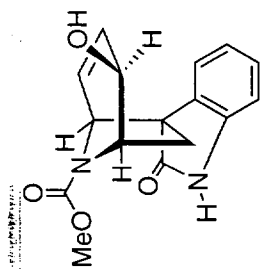
(±)-6
¹H NMR (400 MHz, CDCl₃)





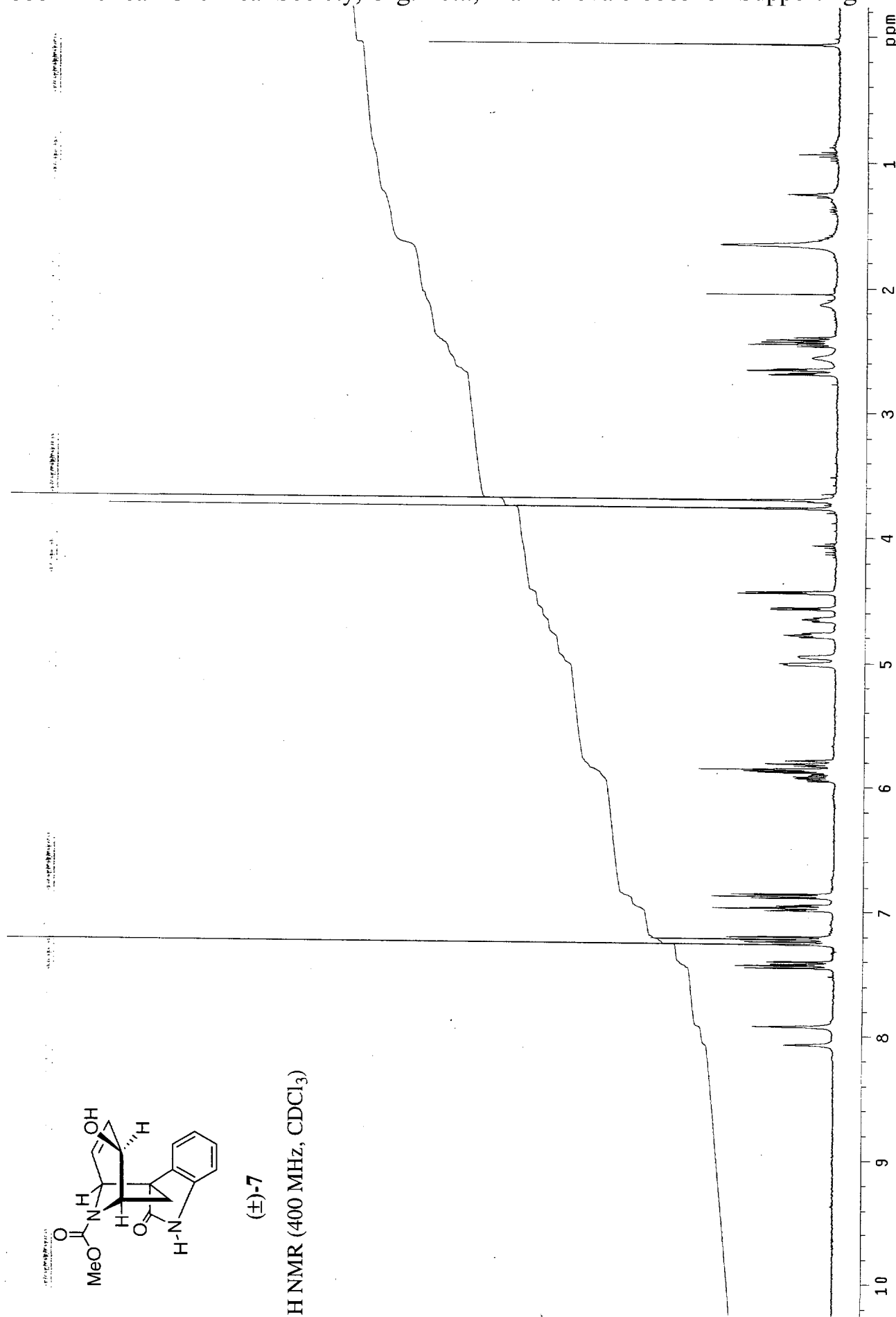
(±)-6
¹³C NMR (100 MHz, CDCl₃)

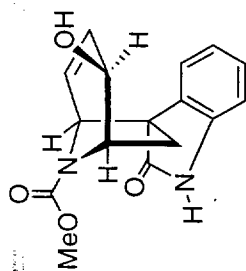




(±)-7

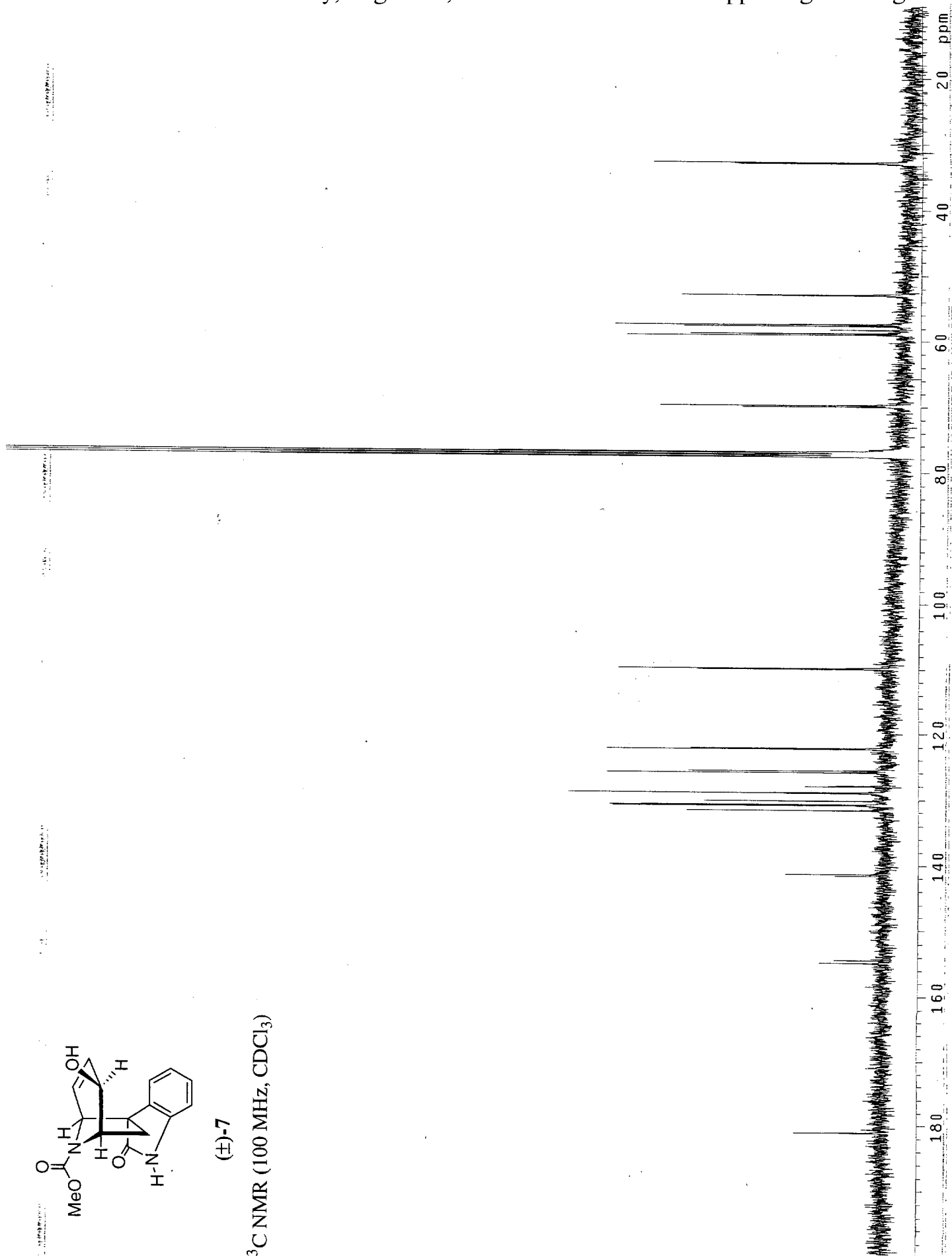
¹H NMR (400 MHz, CDCl₃)

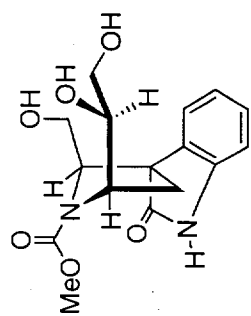




(±)-7

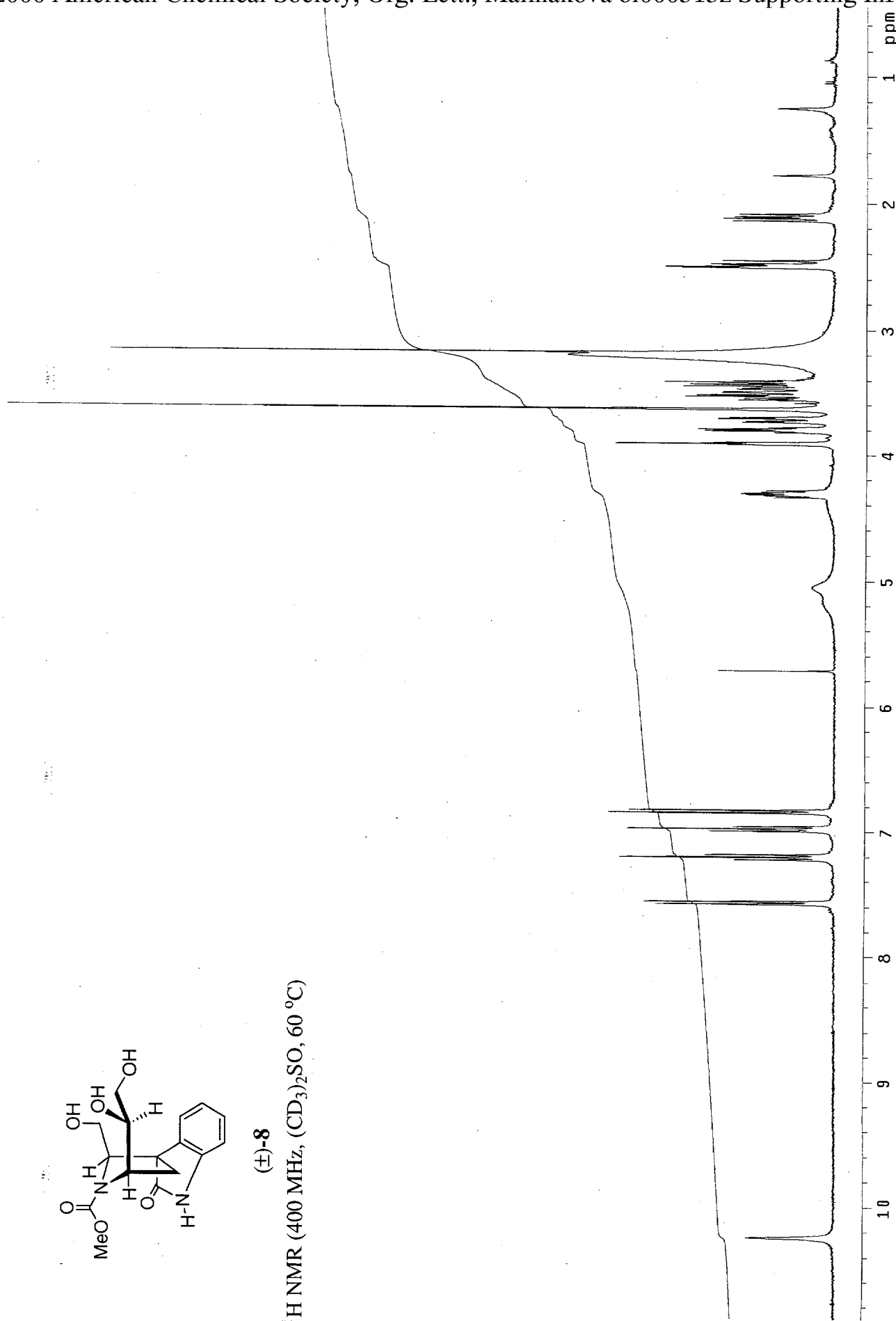
¹³C NMR (100 MHz, CDCl₃)

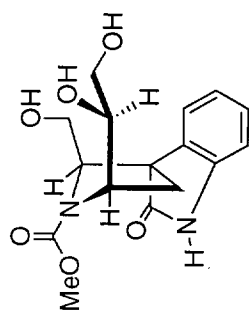




(±)-8

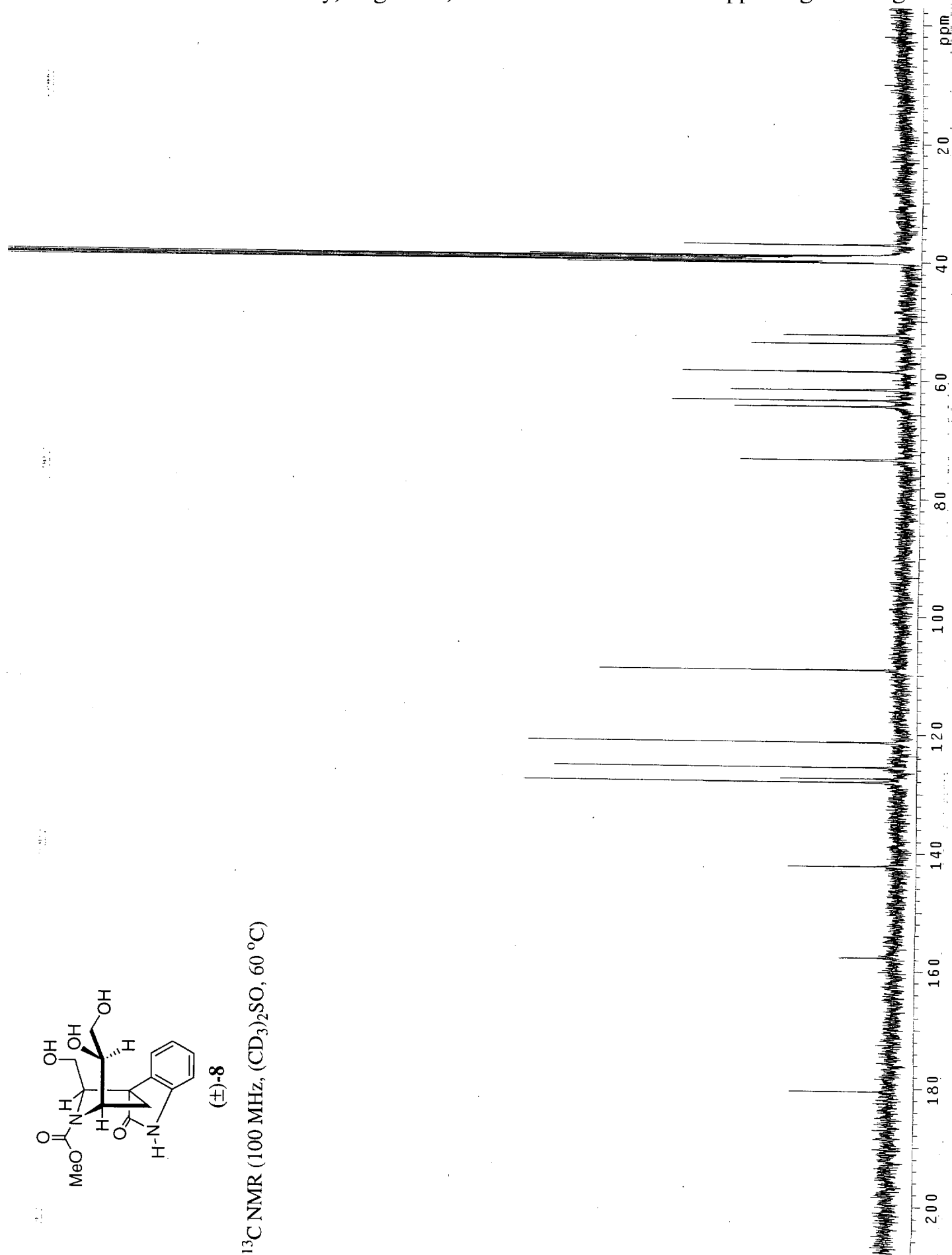
¹H NMR (400 MHz, (CD₃)₂SO, 60 °C)





(±)-8

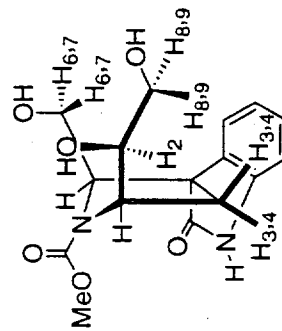
¹³C NMR (100 MHz, (CD₃)₂SO, 60 °C)



STANDARD CARBON PARAMETERS

Pulse Sequence: COSY
 Solvent: dmsd
 Temp. 60.0 C / 333.1 K
 File: cosy.0151
 INOVA-600 "1600data"
 PULSE SEQUENCE: COSY
 Relax. delay 2.000 sec
 Acq. time 0.144 sec
 Width 7120.6 Hz
 2D Width 7120.6 Hz
 32 repetitions
 312 increments
 OBSERVE H1, 599.7537500 MHz
 DATA PROCESSING
 Sq. sine bell 0.072 sec
 F1 DATA PROCESSING
 Sq. sine bell 0.018 sec
 FT size 2048 x 2048
 Total time 9 hr, 57 min, 59 sec

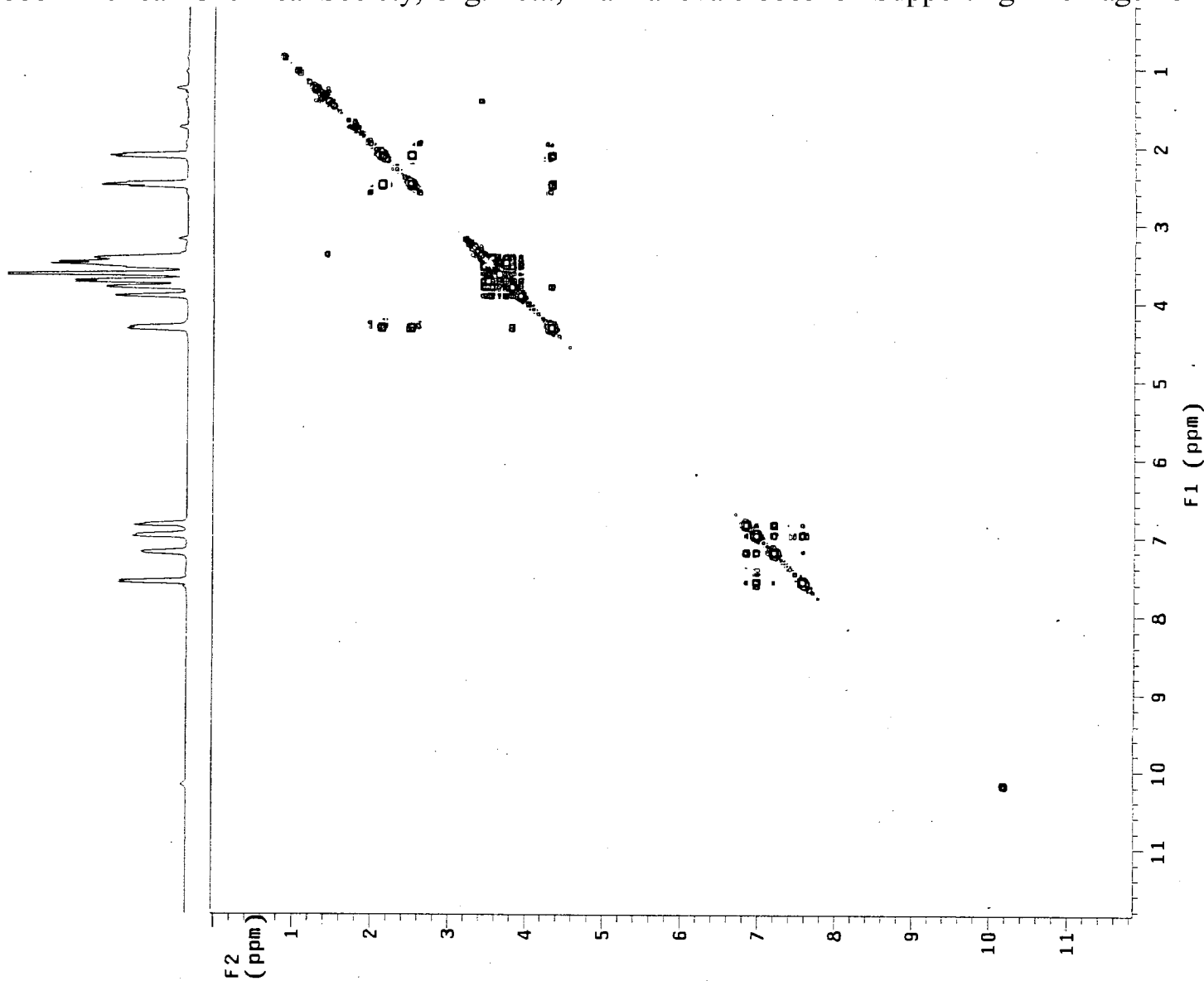
COSY 2D NMR



(±)-8

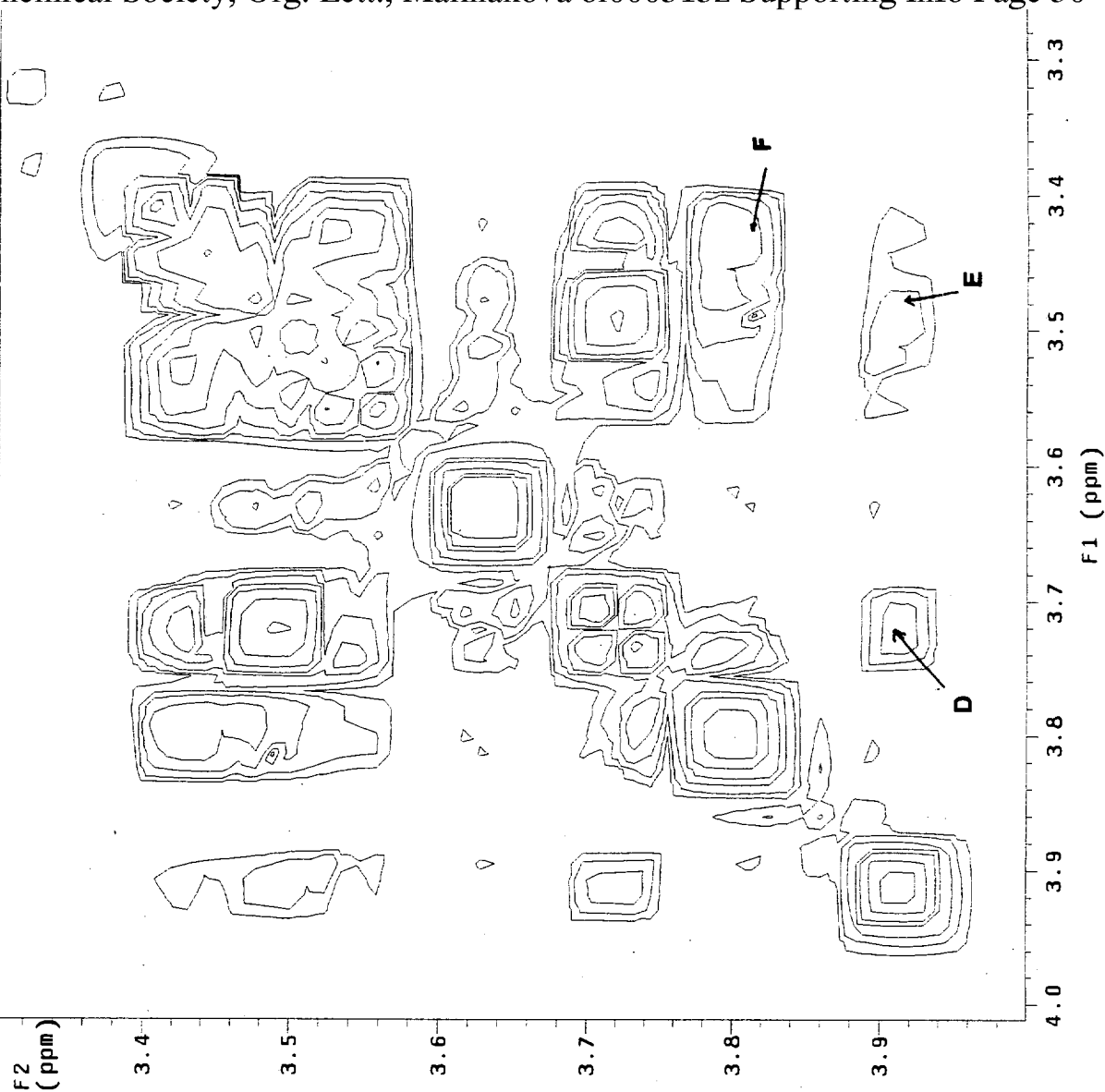
Important ¹H NMR signal assignments
 derived from COSY :

- H₂ 3.81 ppm
- H_{8,9} 3.43 ppm, 3.55 ppm
- H_{6,7} 3.49 ppm, 3.72 ppm
- H_{3,4} 2.10 ppm, 2.50 ppm

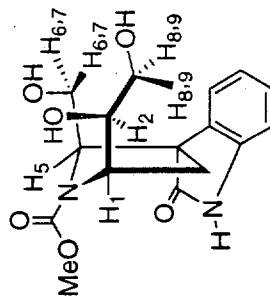


STANDARD CARBON PARAMETERS

Pulse Sequence: COSY
 Solvent: dmsd
 Temp. 60.0 C / 333.1 K
 File: cosy.0151
 INOVA-600 "1600data"
 PULSE SEQUENCE: COSY
 Relax. delay 2.000 sec
 Acq. time 0.144 sec
 Width 7120.6 Hz
 20 Width 7120.6 Hz
 32 repetitions
 512 increments
 OBSERVE HI, 599.7537500 MHZ
 DATA PROCESSING
 Sq. sine bell 0.072 sec
 F1 DATA PROCESSING
 Sq. sine bell 0.018 sec
 FT size 2048 x 2048
 Total time 9 hr, 57 min, 59 sec



COSY 2D NMR



(±)-8

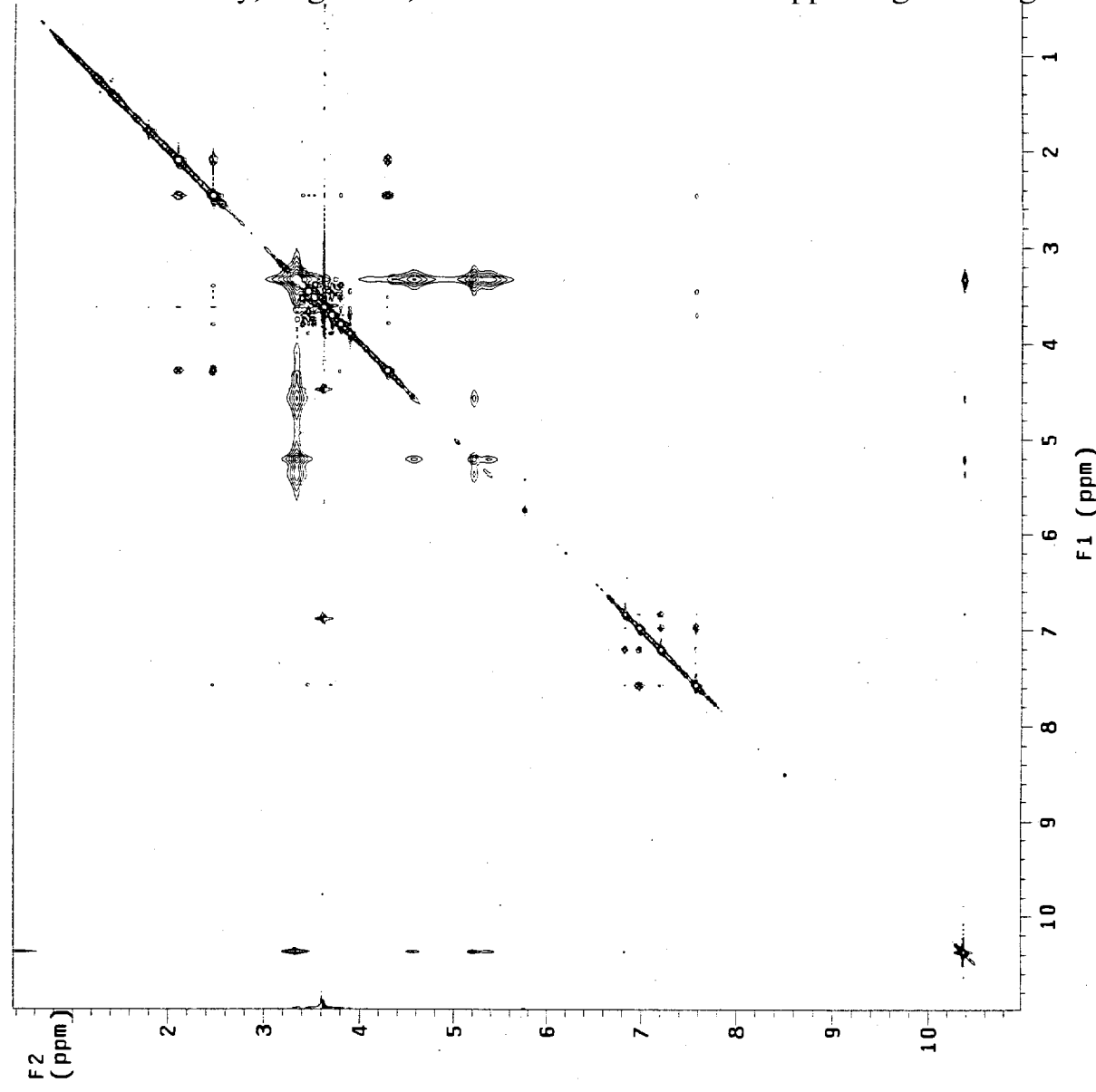
D (H₅ 3.90 ppm - H_{6,7} 3.72 ppm)

E (H₅ 3.90 ppm - H_{6,7} 3.49 ppm)

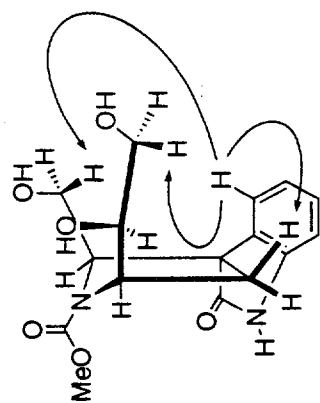
F (H₂ 3.81 ppm - H_{8,9} 3.43 ppm)

STANDARD CARBON PARAMETERS

Pulse Sequence: noesy
 Solvent: dms0
 Temp. 25.0 C / 298.1 K
 File: NOE12.data
 INOVA-600 "1600data"
 PULSE SEQUENCE: noesy
 Relax. delay 0.800 sec
 Mixing 0.200 sec
 Acq. time 0.161 sec
 2D Width 6362.3 Hz
 48 repetitions
 2 x 512 increments
 OBSERVE HI, 599.7537486 MHz
 DATA PROCESSING
 Line broadening 5.0 Hz
 Gauss window 0.080 sec
 center at 0.018 sec
 Sine bell 0.115 sec
 Shifted by -0.066 sec
 F1 DATA PROCESSING
 Sine bell 0.063 sec
 Shifted by -0.063 sec
 FT size 2048 x 2048
 Total time 16 hr, 33 min, 55 sec



NOESY 2D NMR

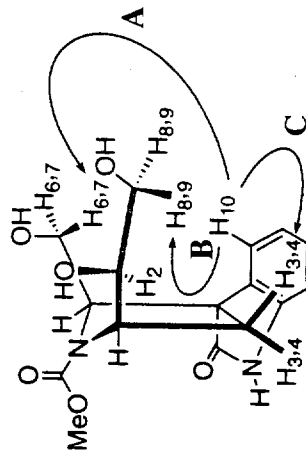
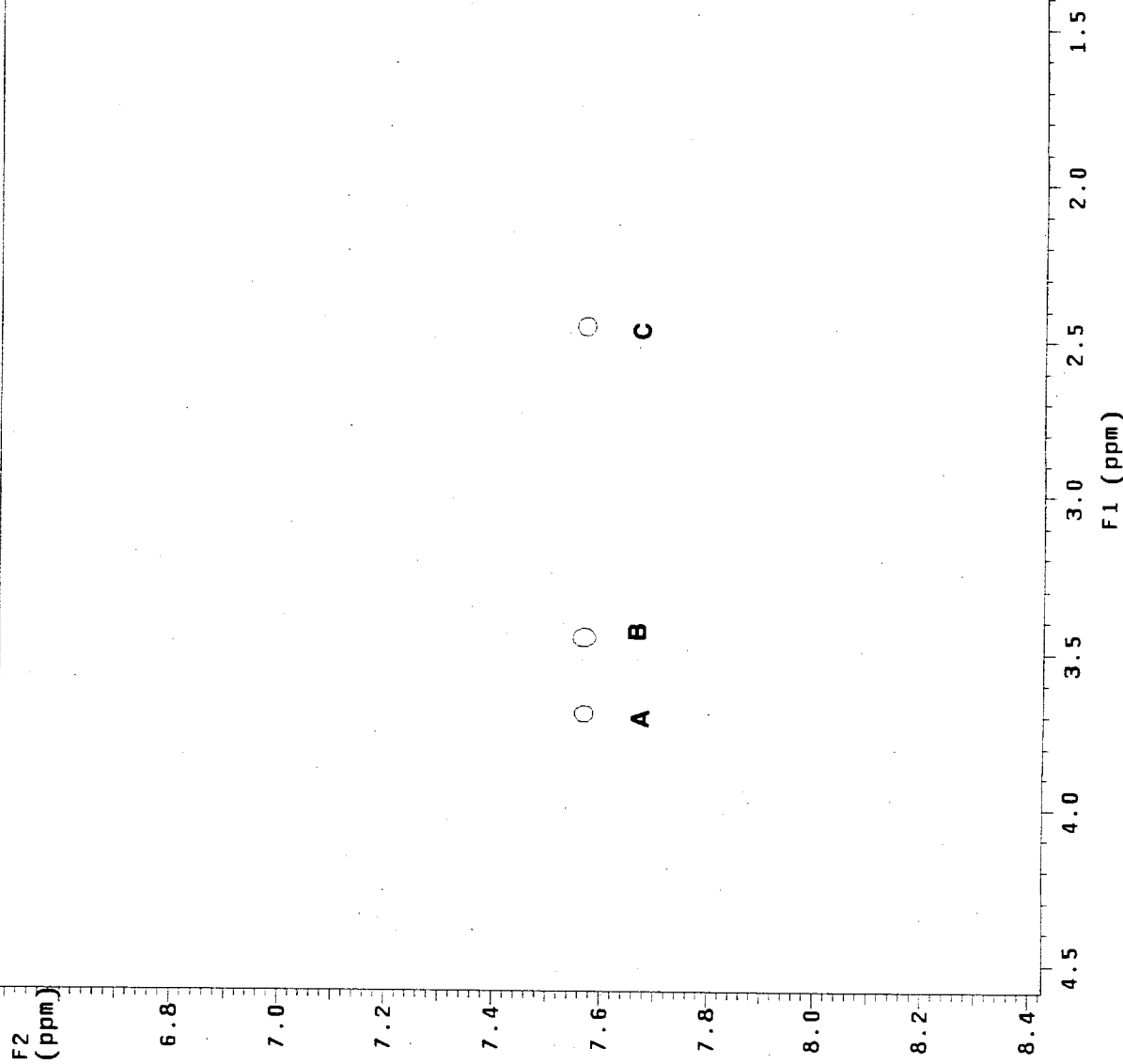


(±)-8

STANDARD CARBON PARAMETERS

Pulse Sequence: noesy
 Solvent: dms0
 Temp: 25.0 C / 298.1 K
 File: NOE12.data
 INDVA-600 "f600data"
 PULSE SEQUENCE: noesy
 Relax. delay 0.800 sec
 Mixing 0.200 sec
 Acq. time 0.161 sec
 Width 6362.3 Hz
 2D Width 6362.3 Hz
 48 repetitions
 2 x 512 increments
 OBSERVE H1, 599.7537486 MHZ
 DATA PROCESSING
 Line broadening 5.0 Hz
 Gauss window 0.080 sec
 center at 0.018 sec
 Sine bell 0.115 sec
 Shifted by -0.066 sec
 F1 DATA PROCESSING
 Sine bell 0.063 sec
 Shifted by -0.063 sec
 FT size 2048 x 2048
 Total time 16 hr, 33 min, 55 sec

NOESY 2D NMR



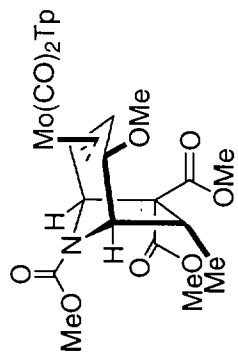
(±)-8

Correlations detected:

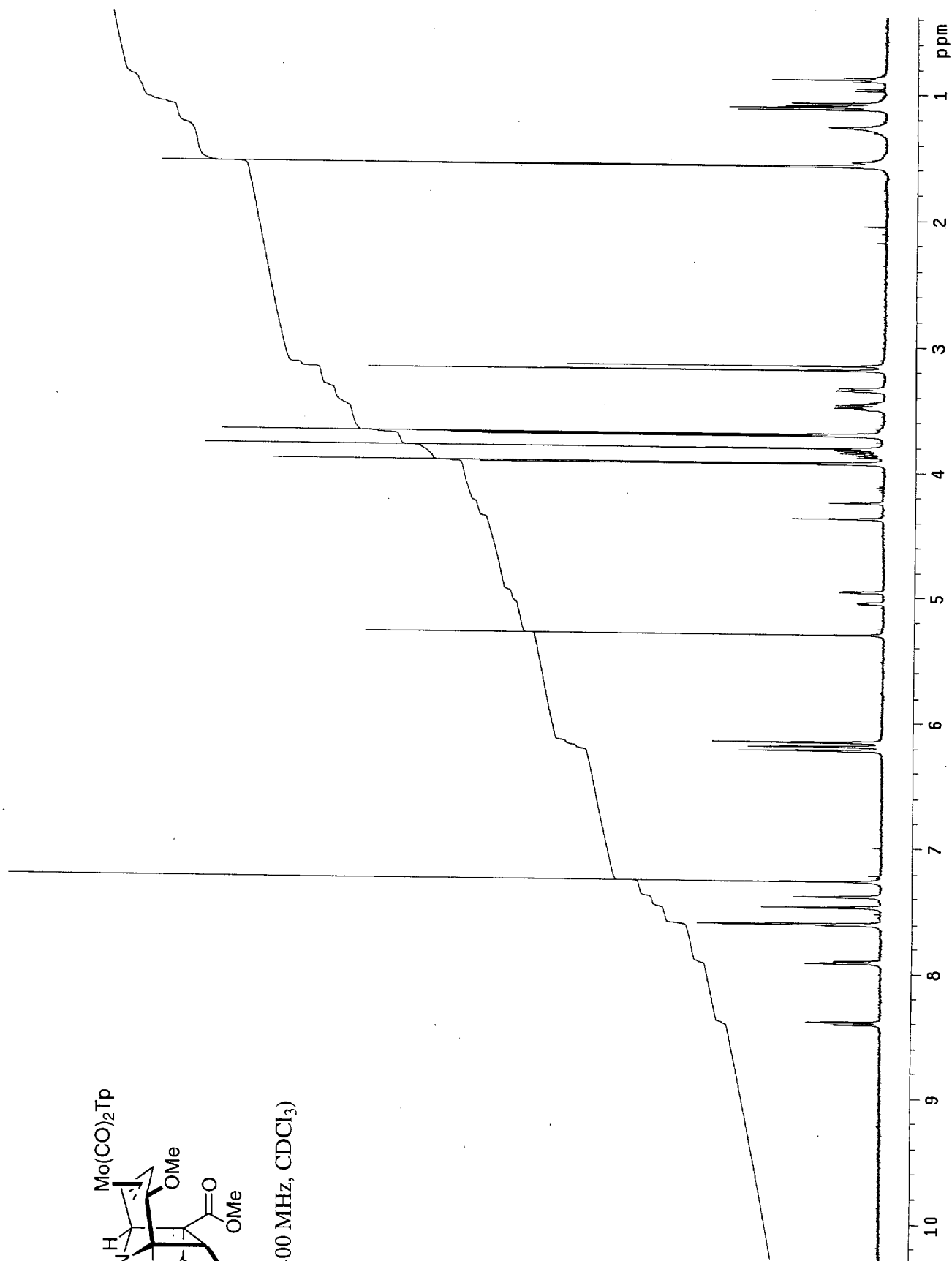
A: 7.58 ppm - 3.70 ppm

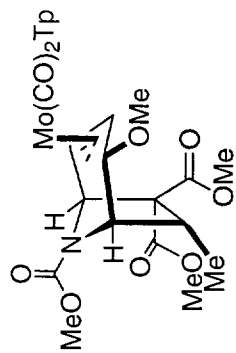
B: 7.58 ppm - 3.43 ppm

C: 7.58 ppm - 2.44 ppm

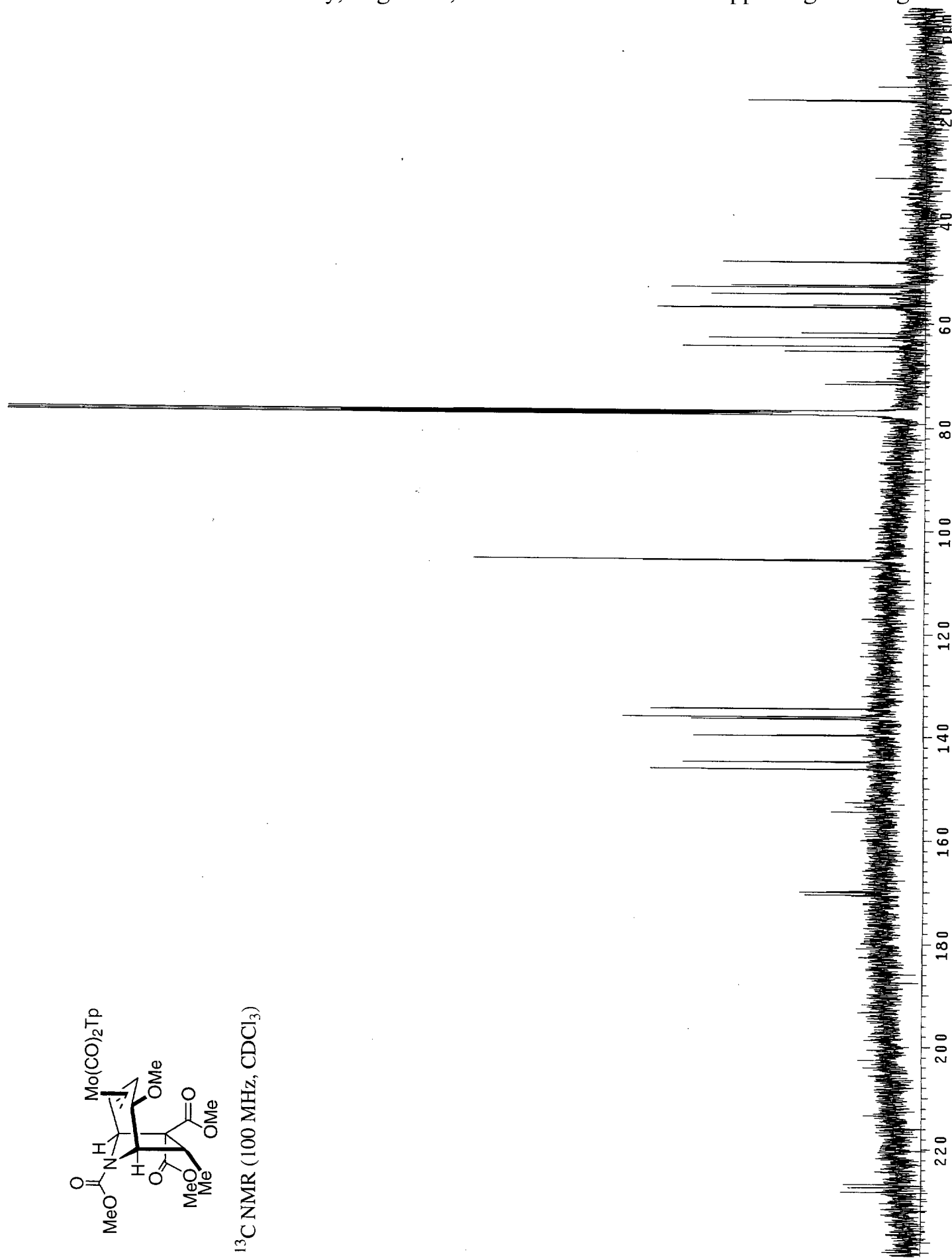


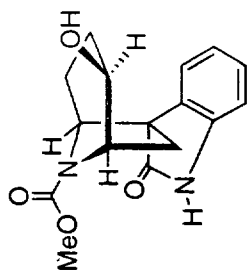
¹H NMR (400 MHz, CDCl₃)





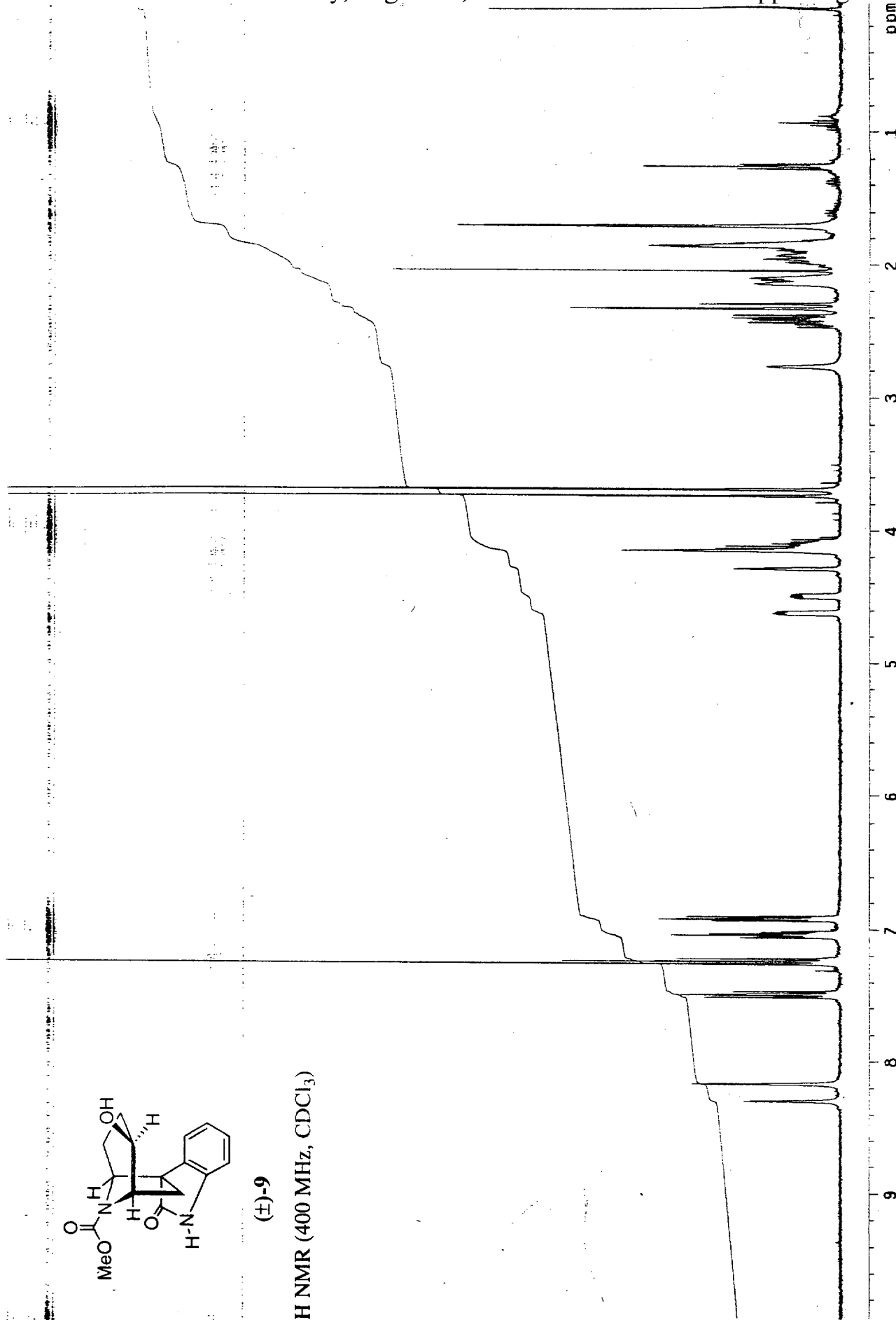
^{13}C NMR (100 MHz, CDCl_3)

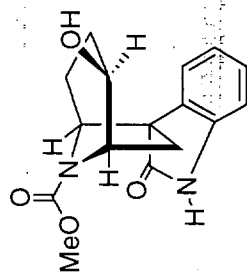




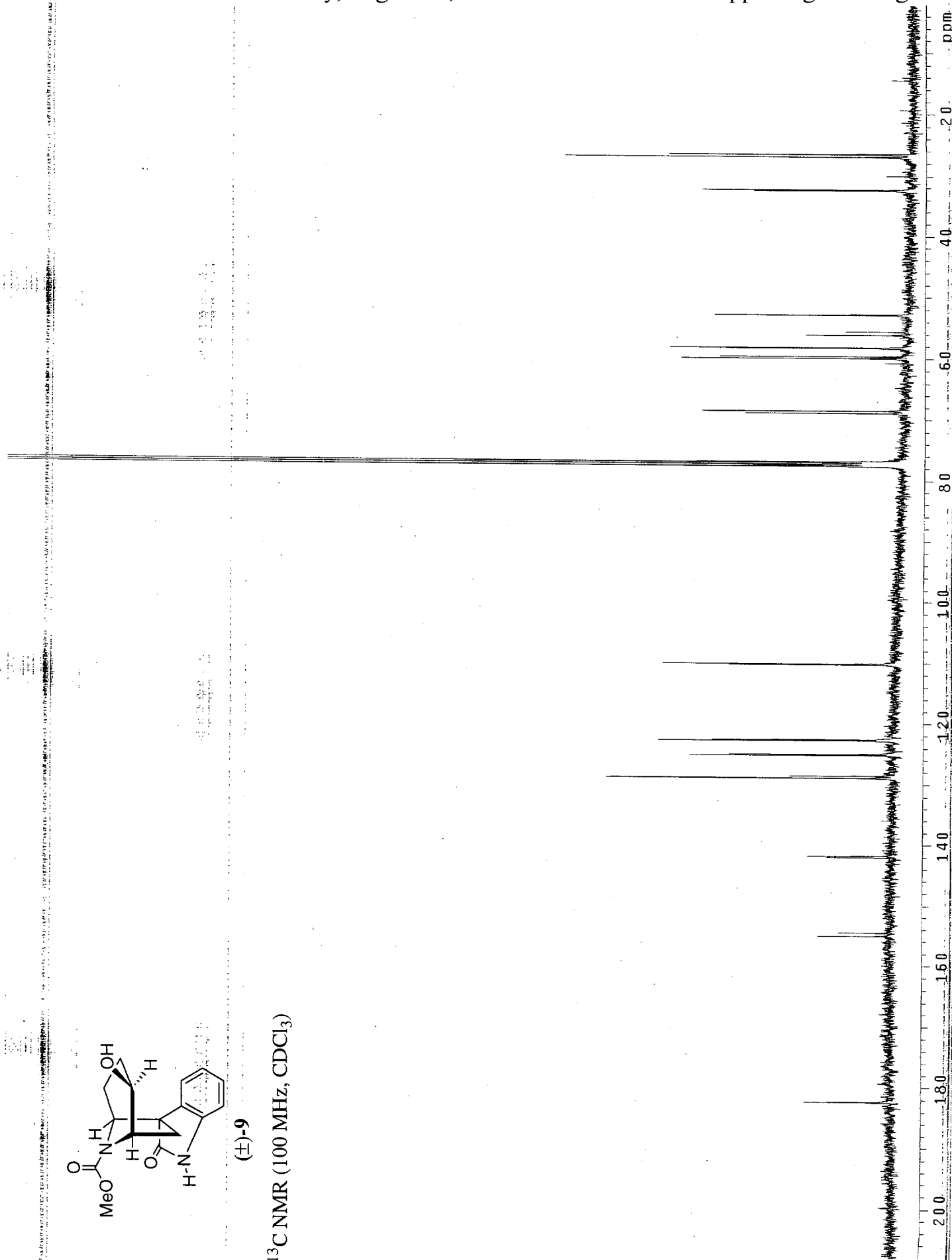
(±)-9

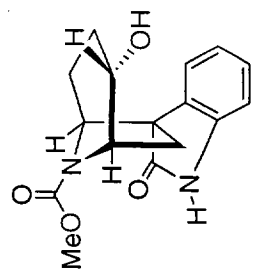
¹H NMR (400 MHz, CDCl₃)





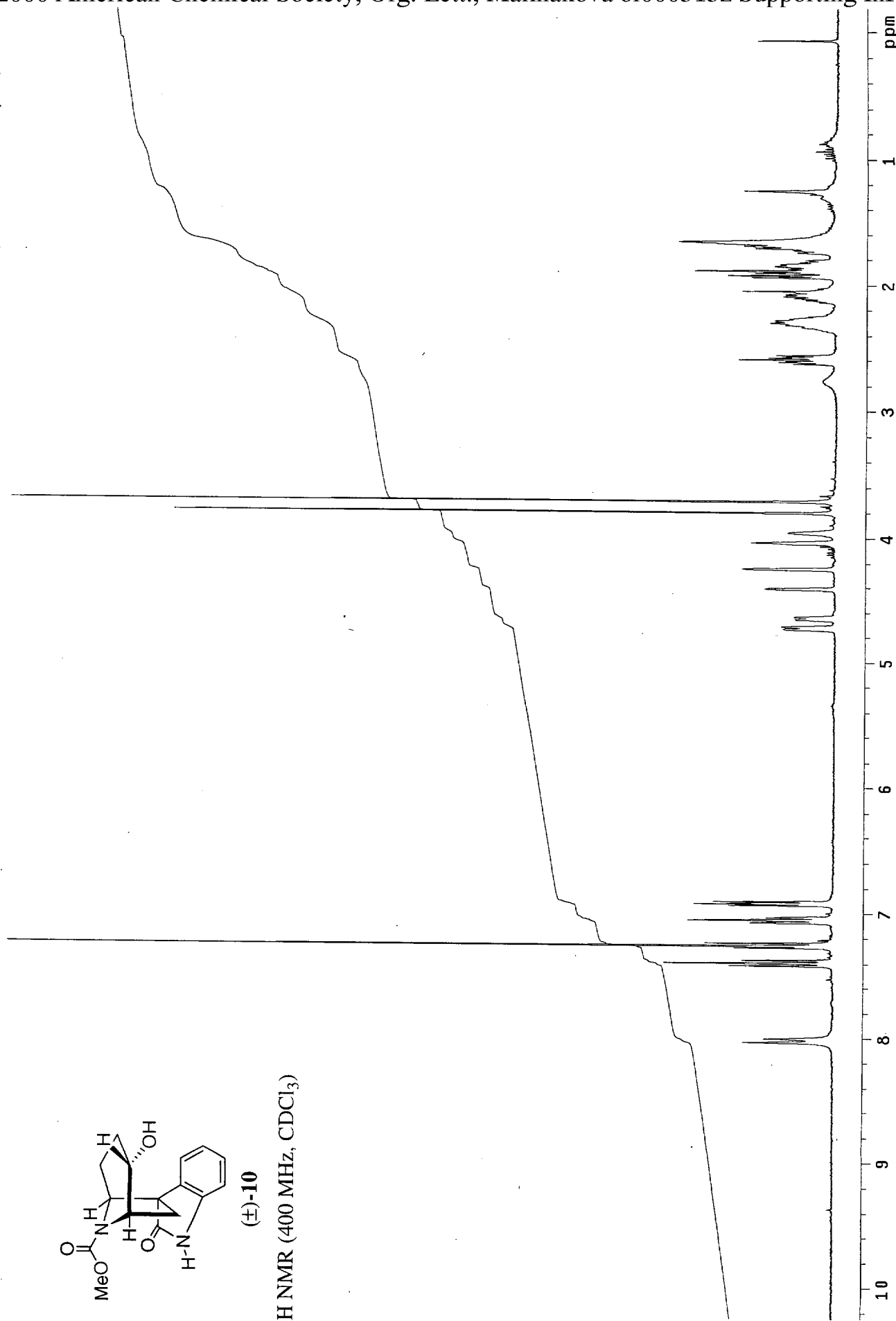
¹³C NMR (100 MHz, CDCl₃)

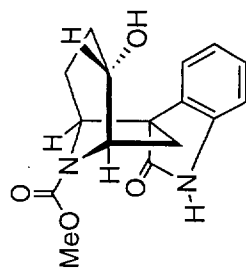




(±)-**10**

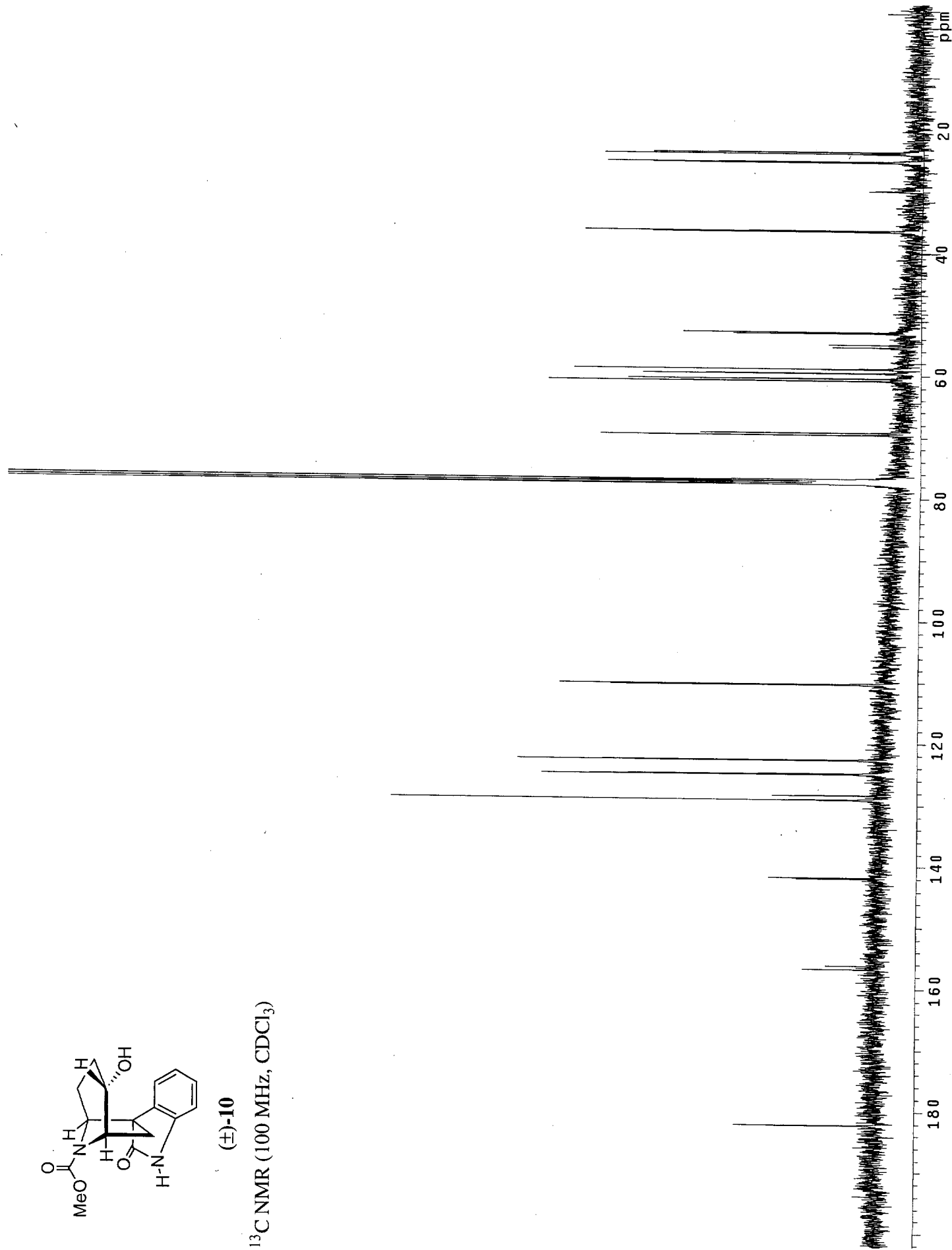
¹H NMR (400 MHz, CDCl₃)



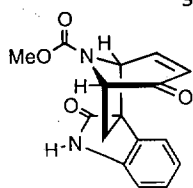
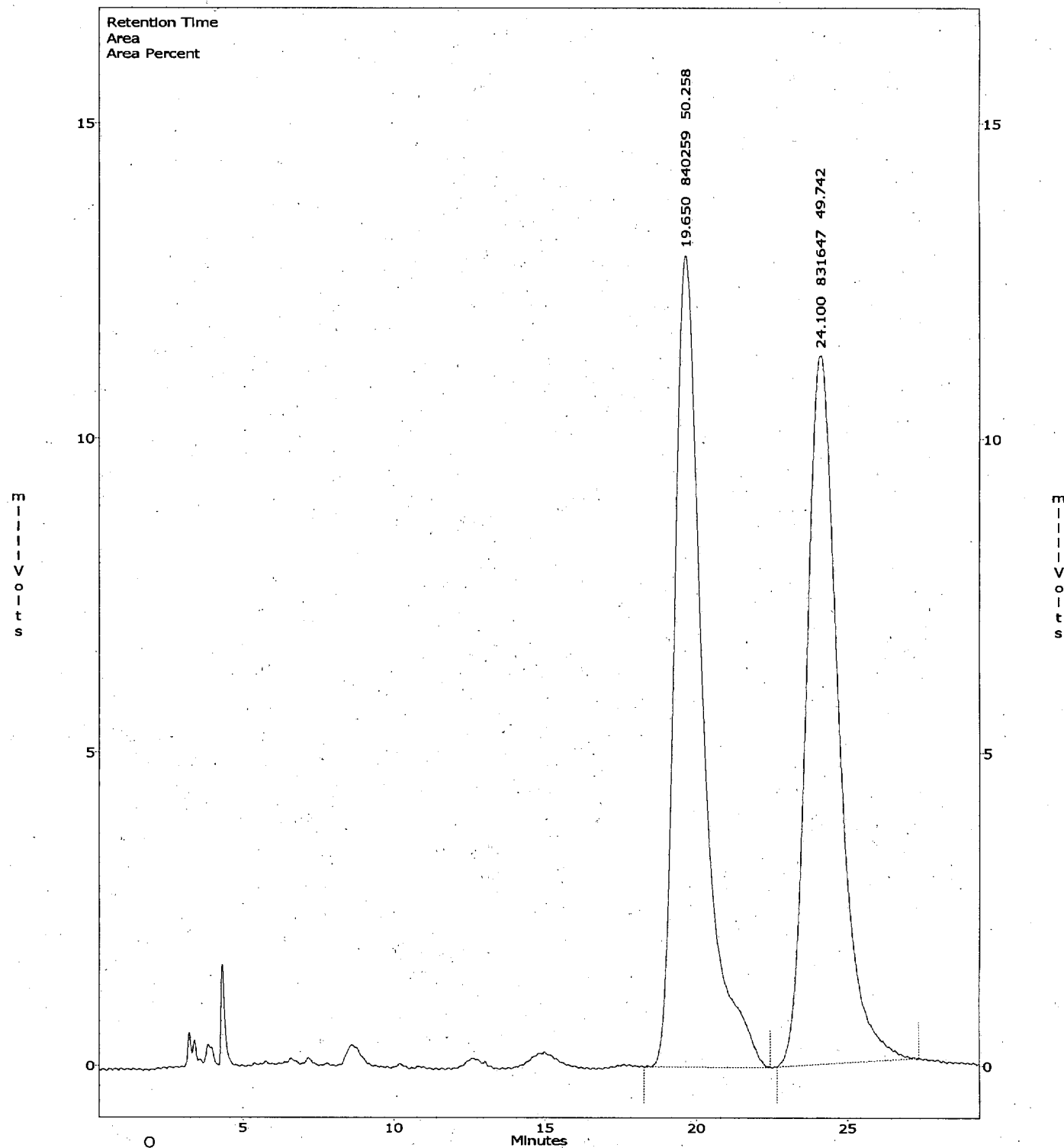


(±)-10

¹³C NMR (100 MHz, CDCl₃)

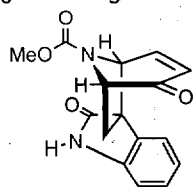
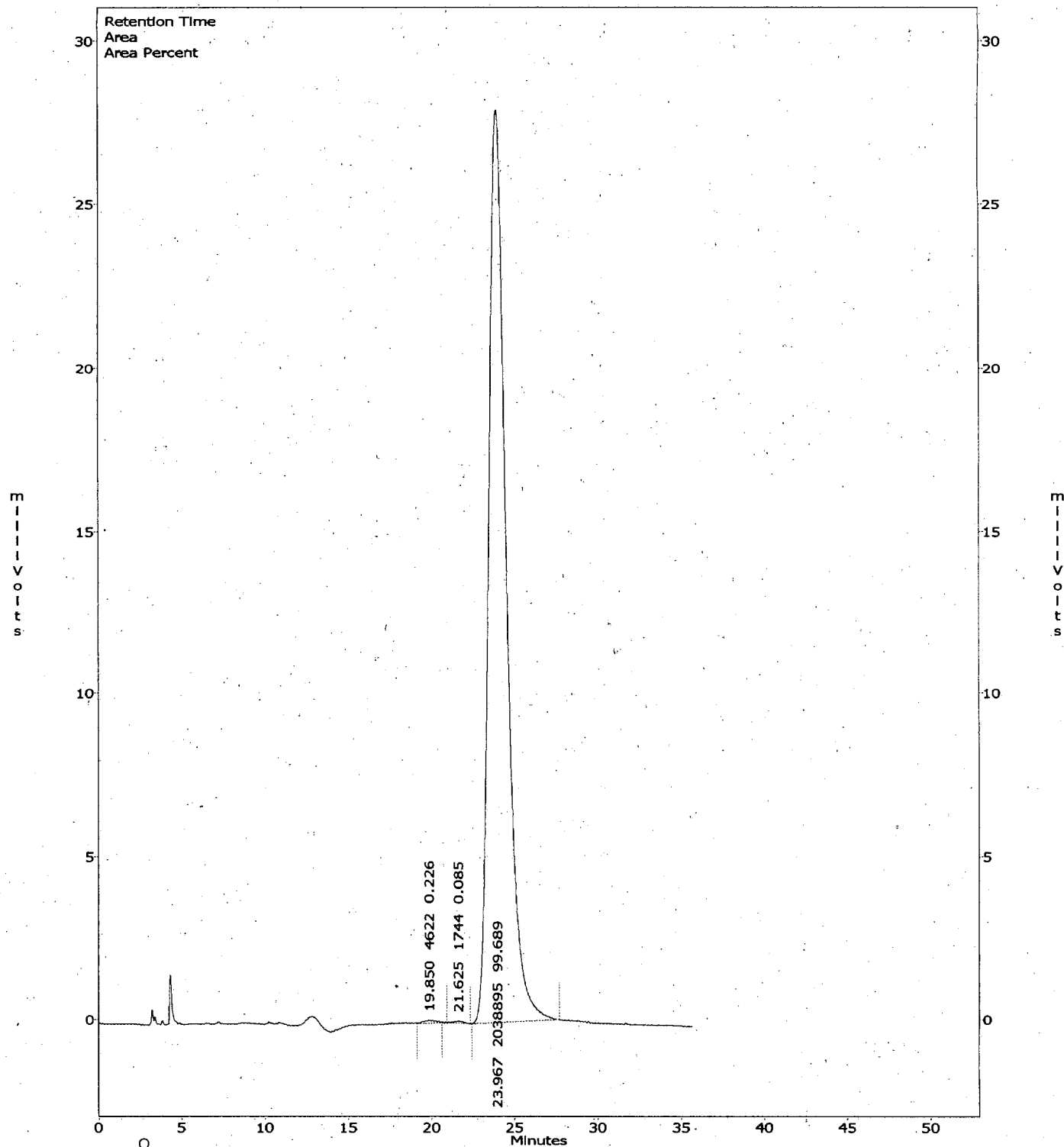


c:\class-vp\chrom\helena\helena.197, Channel A



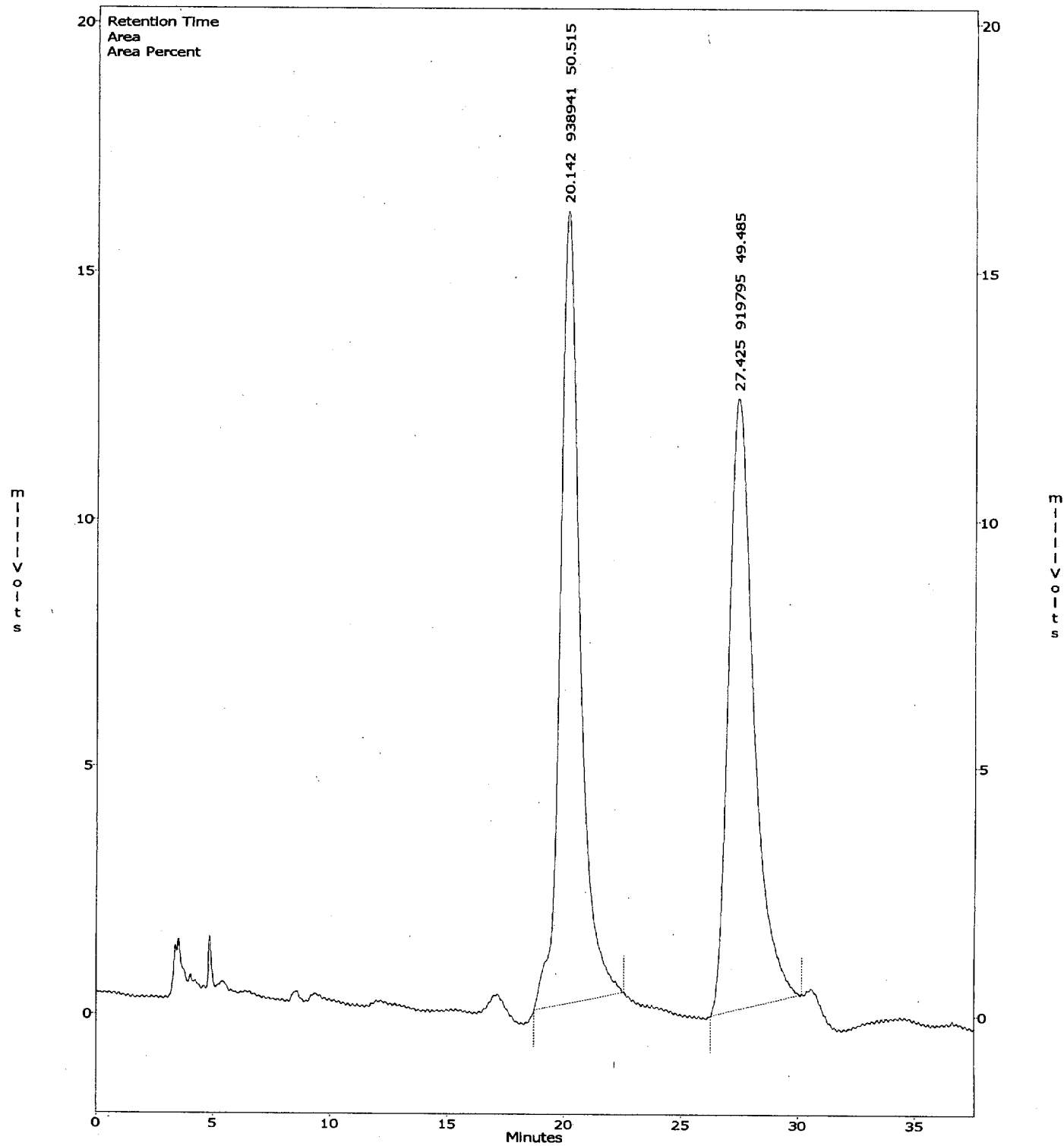
(±)-4 CHIRALPAK AD, 10/90 i-PrOH/Hexane, 1 mL/min, 230 nm

c:\class-vp\chrom\helena\helena.198, Channel A

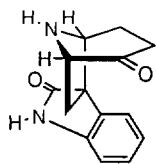
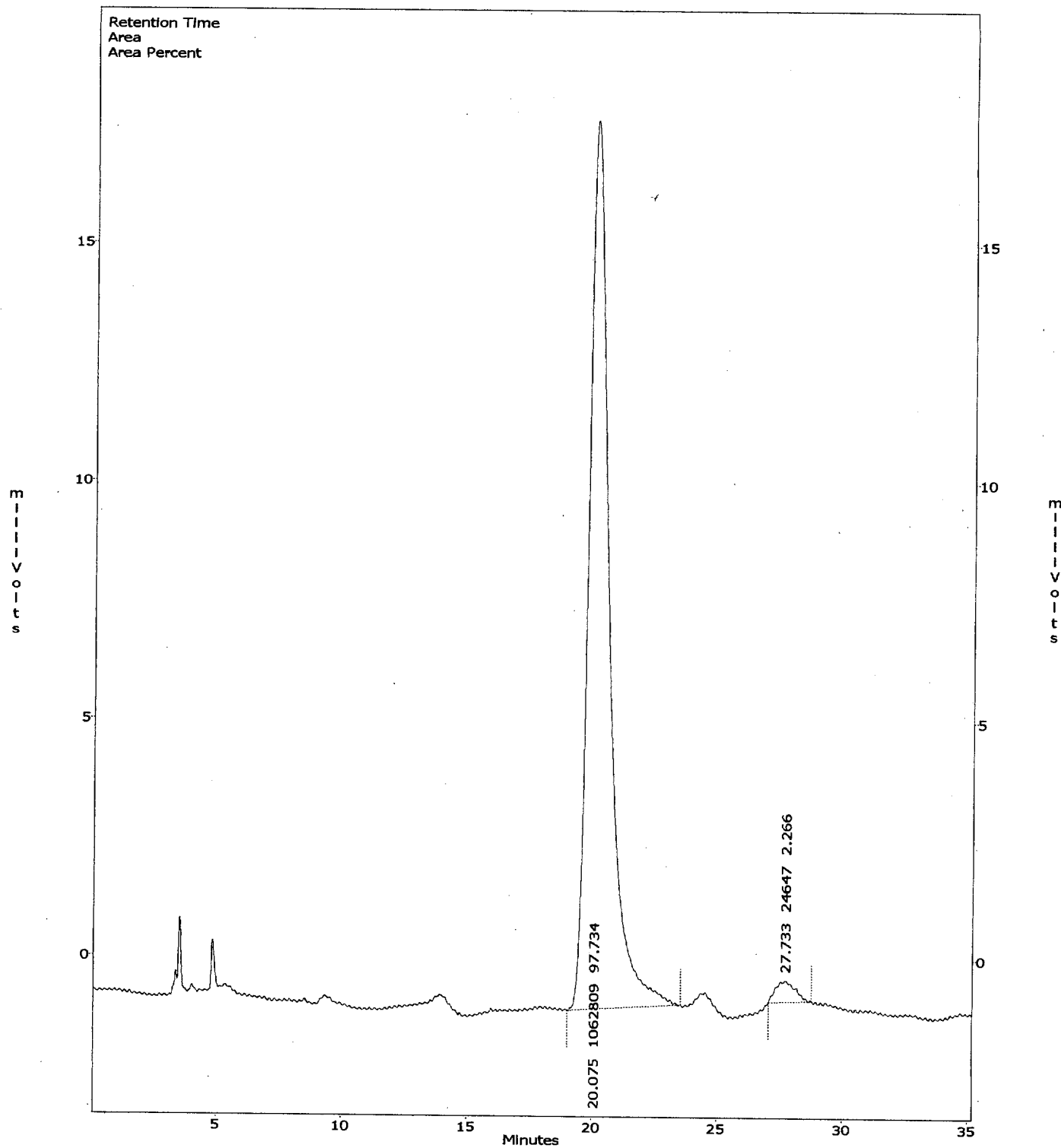


(+)-4 CHIRALPAK AD, 10/90 i-PrOH/Hexane, 1 mL/min, 230 nm

c:\class-vp\chrom\helena\helena.133, Channel A

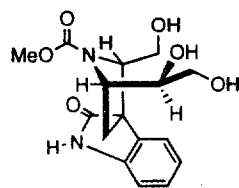
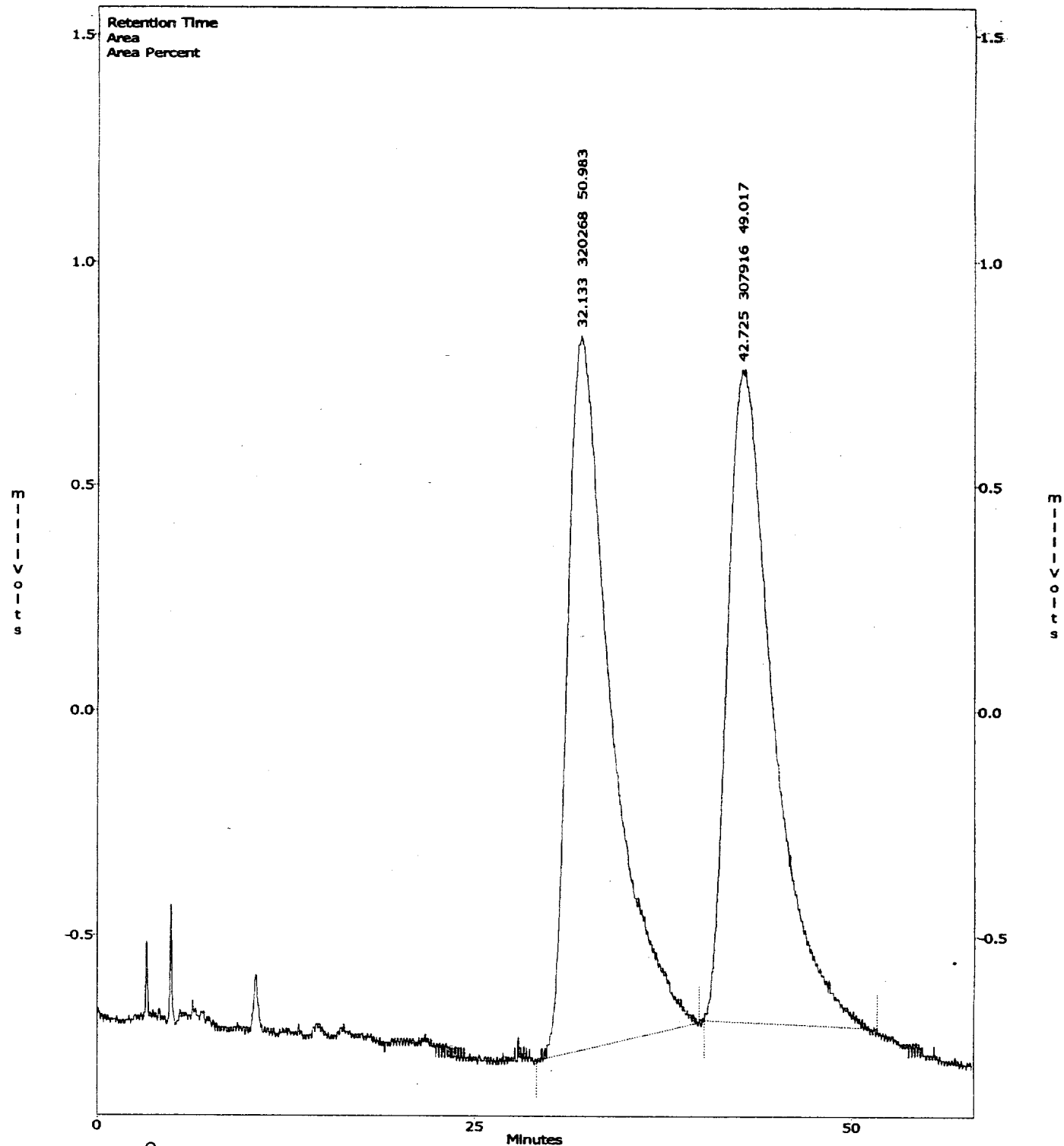


c:\class-vp\chrom\helena\helena.135, Channel A



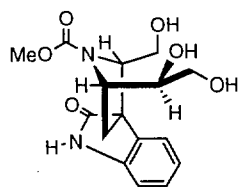
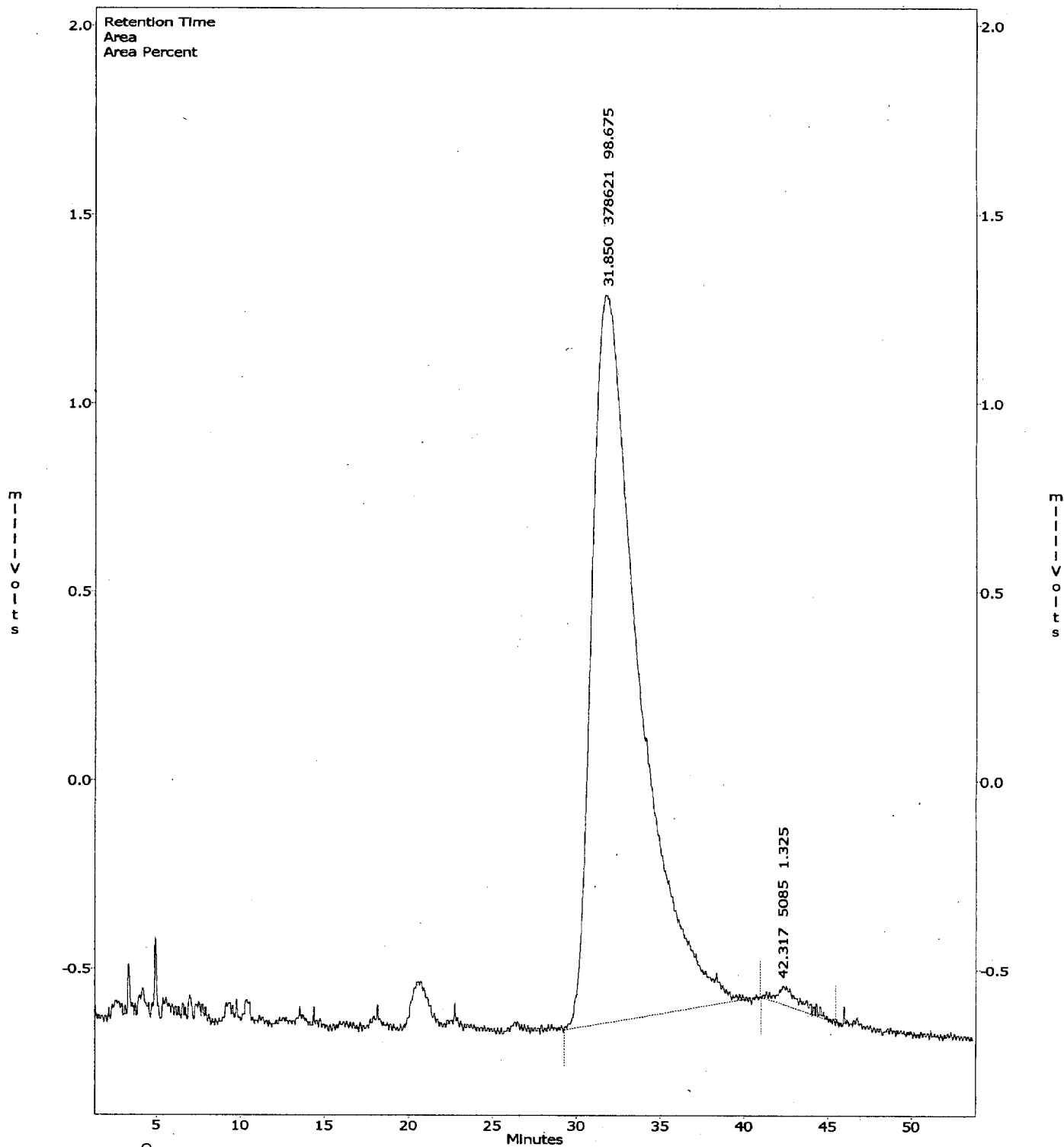
(-)-6 CHIRALPAK AD, 10/90 i-PrOH/Hexane, 1 mL/min, 230 nm

c:\class-vp\chrom\helena\helena.216, Channel A



(±)-8 CHIRALPAK AD, 10/90 i-PrOH/Hexane, 1 mL/min, 260 nm

c:\class-vp\chrom\helena\helena.215, Channel A



(-)-8 CHIRALPAK AD, 10/90 i-PrOH/Hexane, 1 mL/min, 260 nm