Supporting information

Catalytic Asymmetric [3+2]-Cycloaddition for Stereodivergent Synthesis of Chiral Indolyl-pyrrolidines

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1-33 Yayoi, Inage, Chiba 263-8522, Japan.
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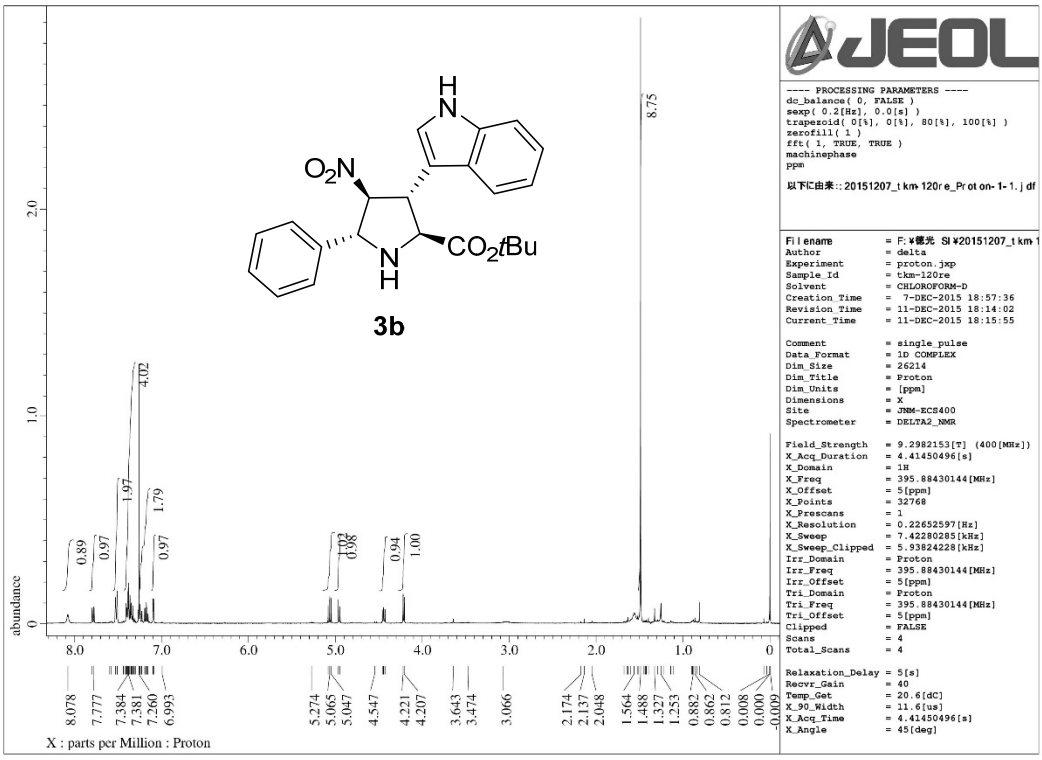
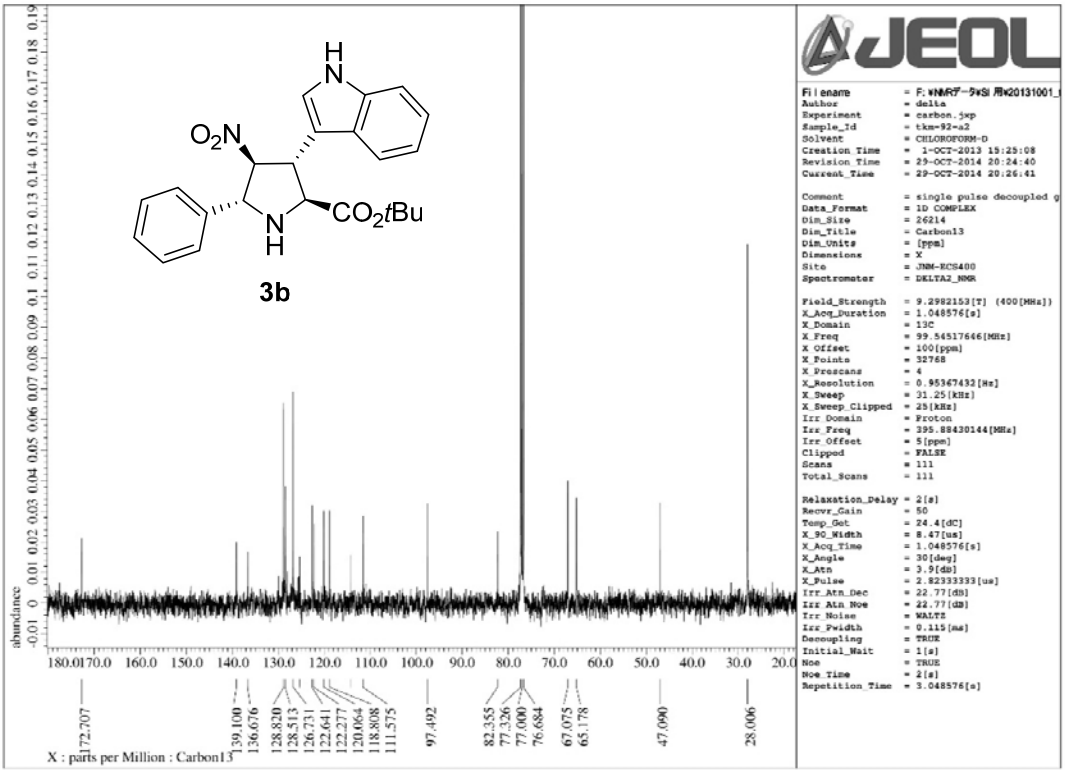
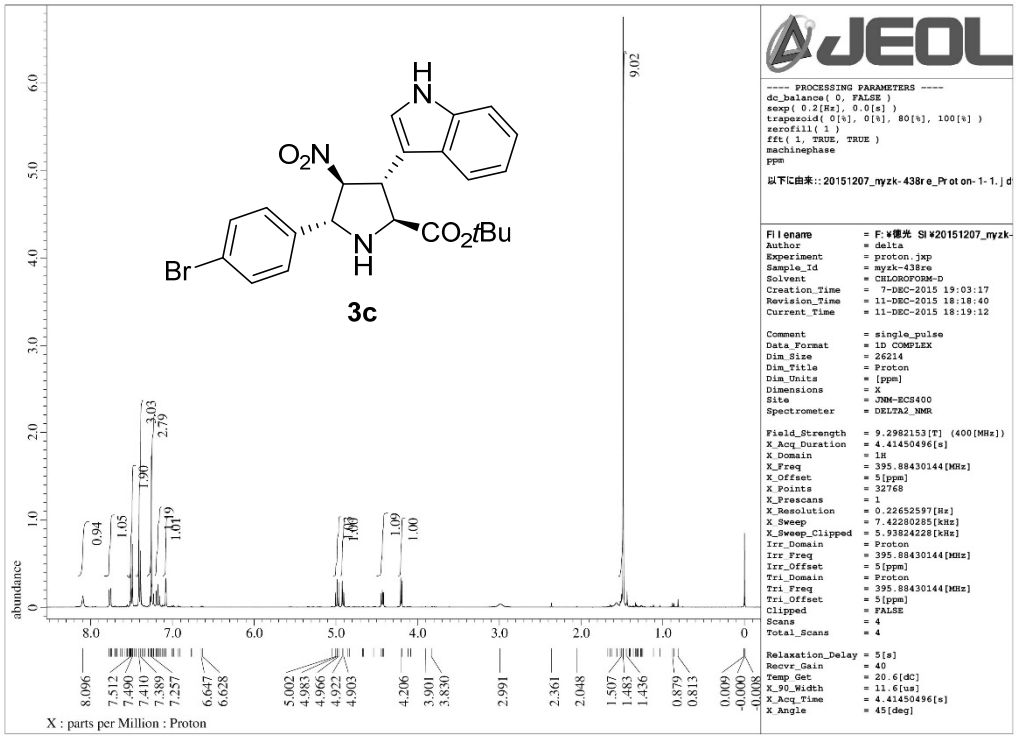
Contents

1. ^{1}H-NMR and ^{13}C-NMR spectra



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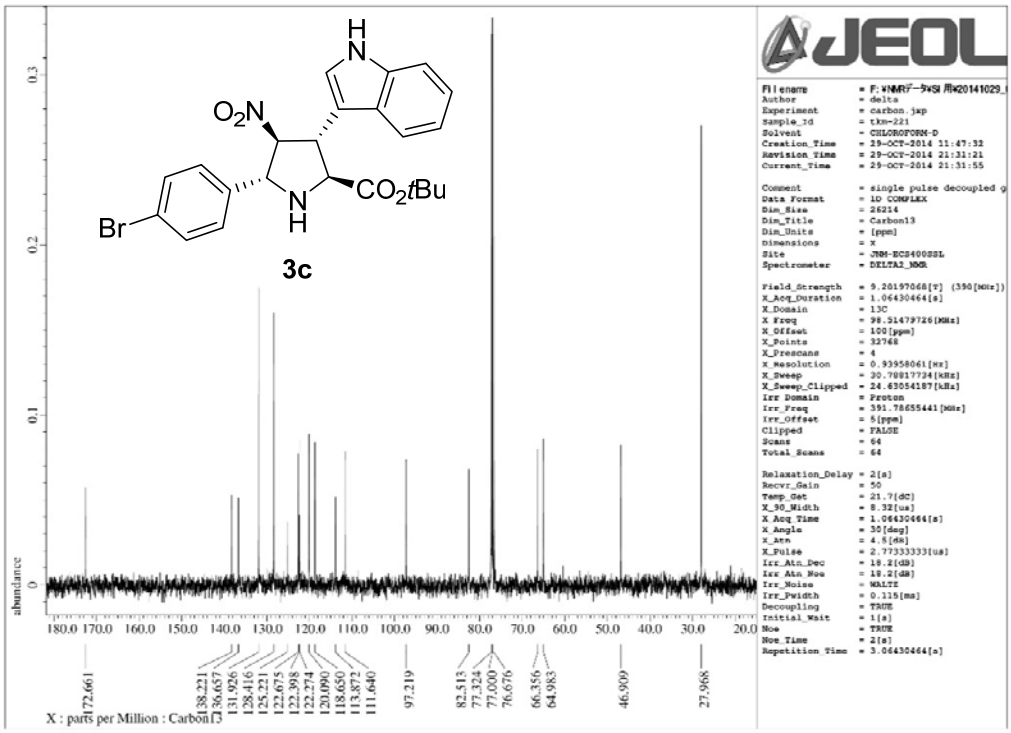
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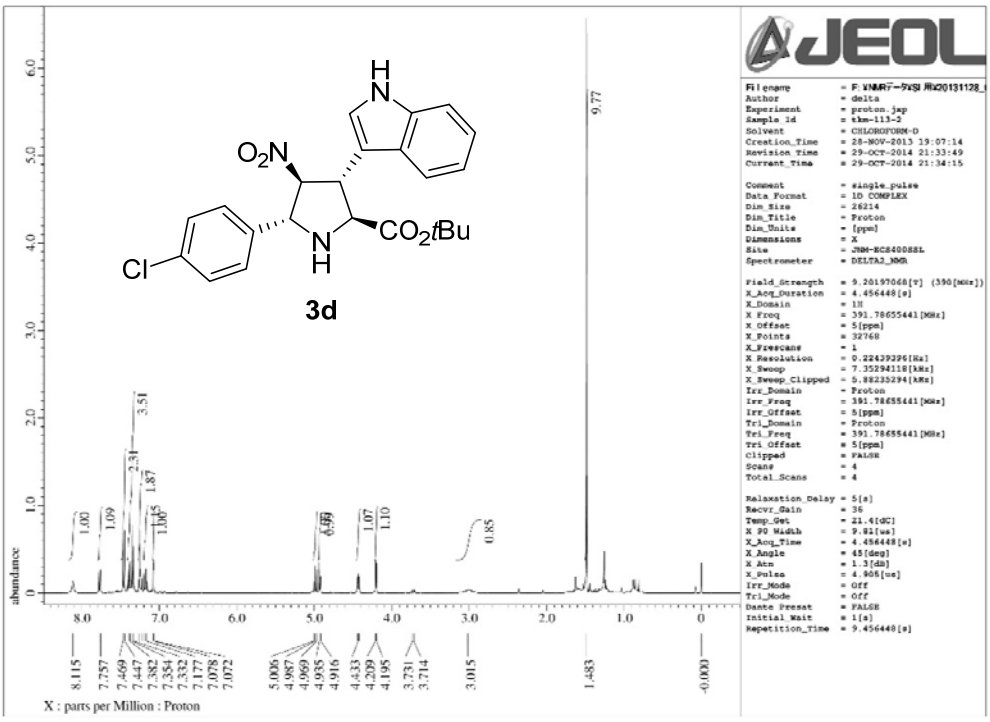
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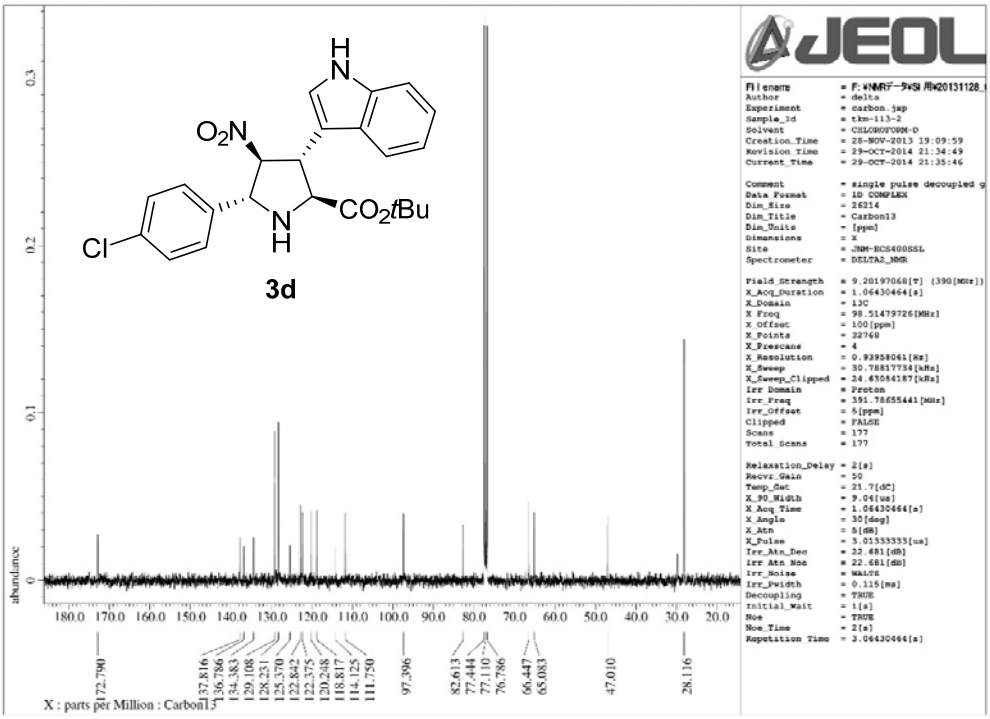
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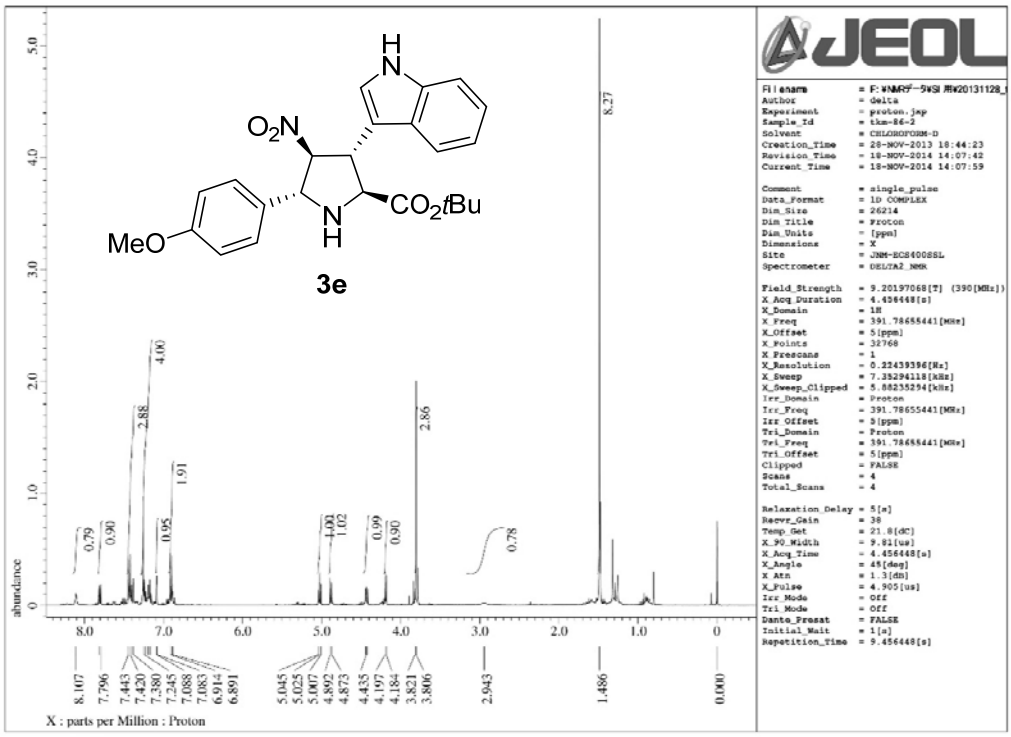
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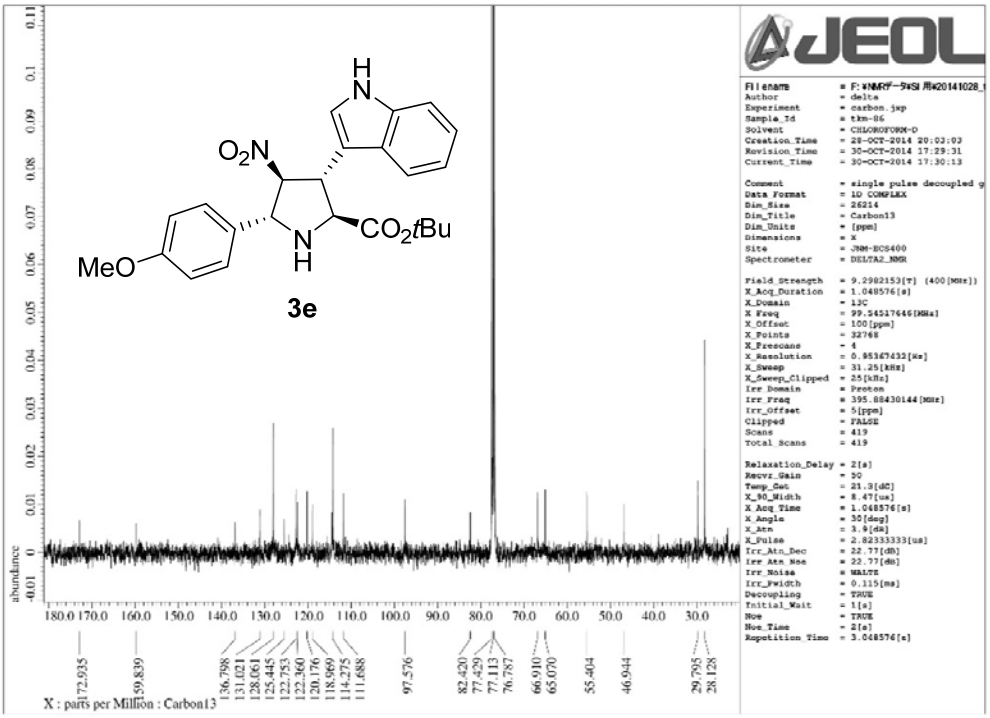
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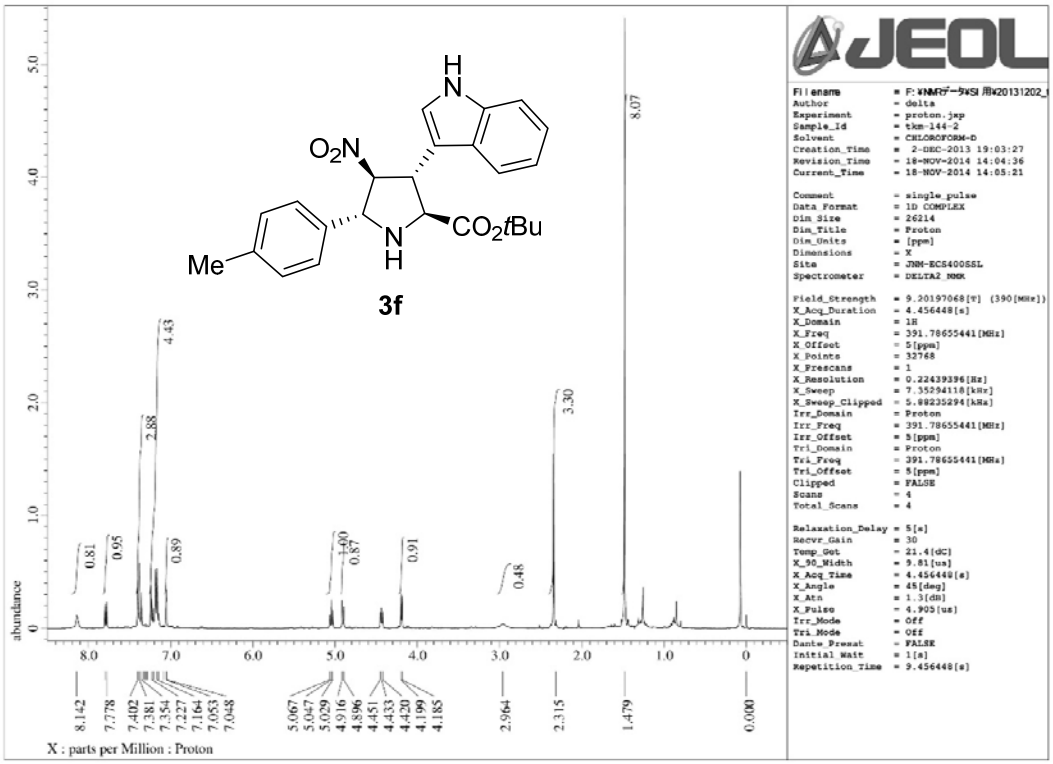
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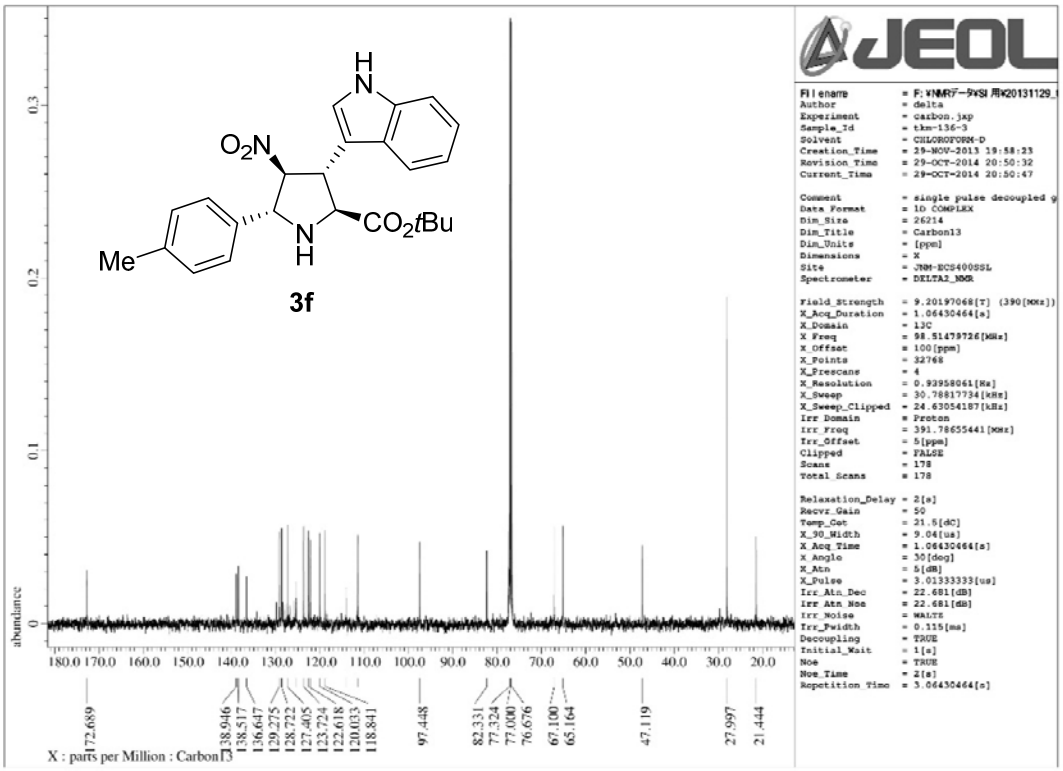
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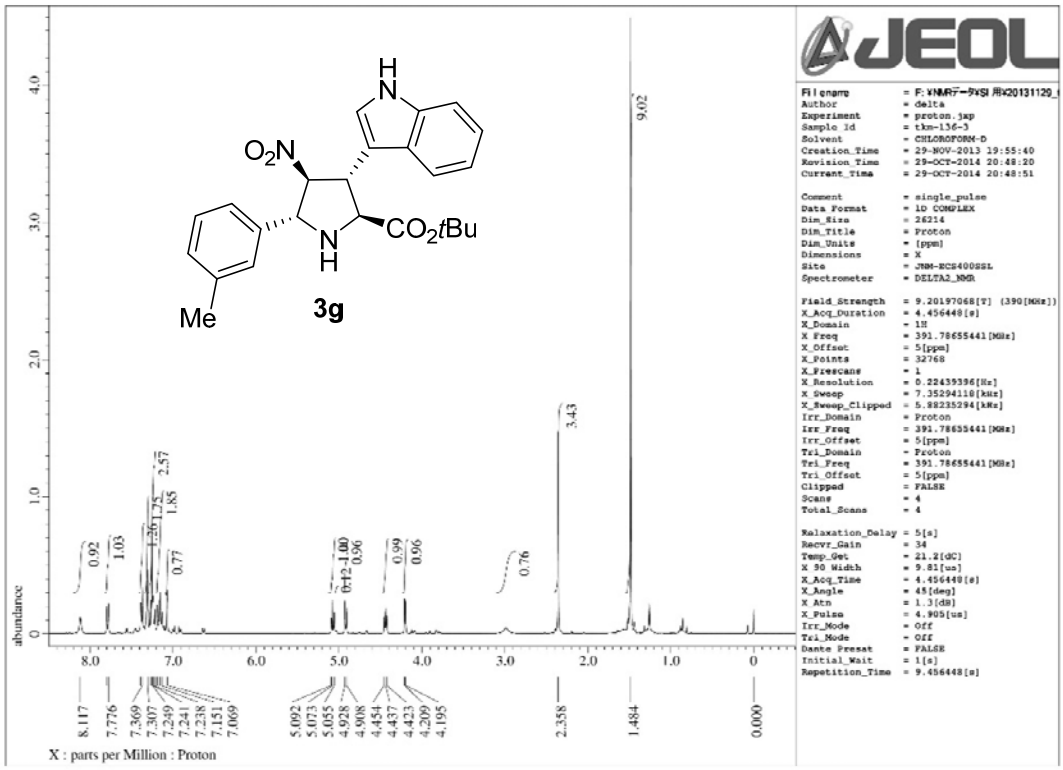
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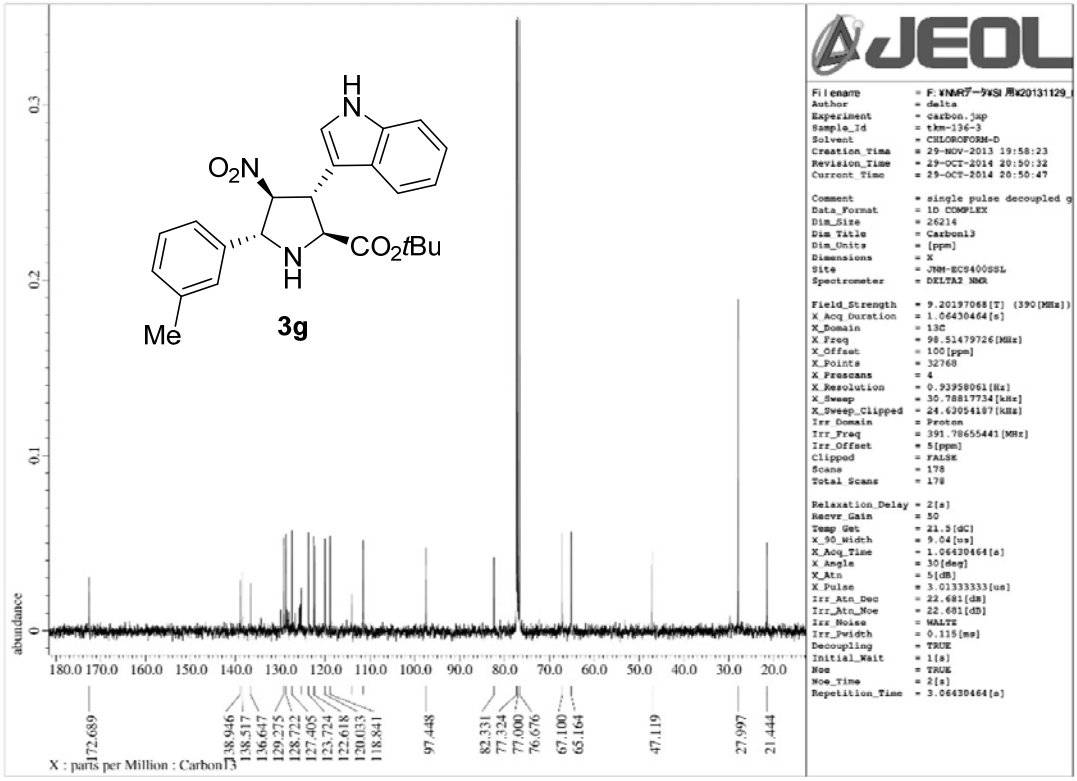
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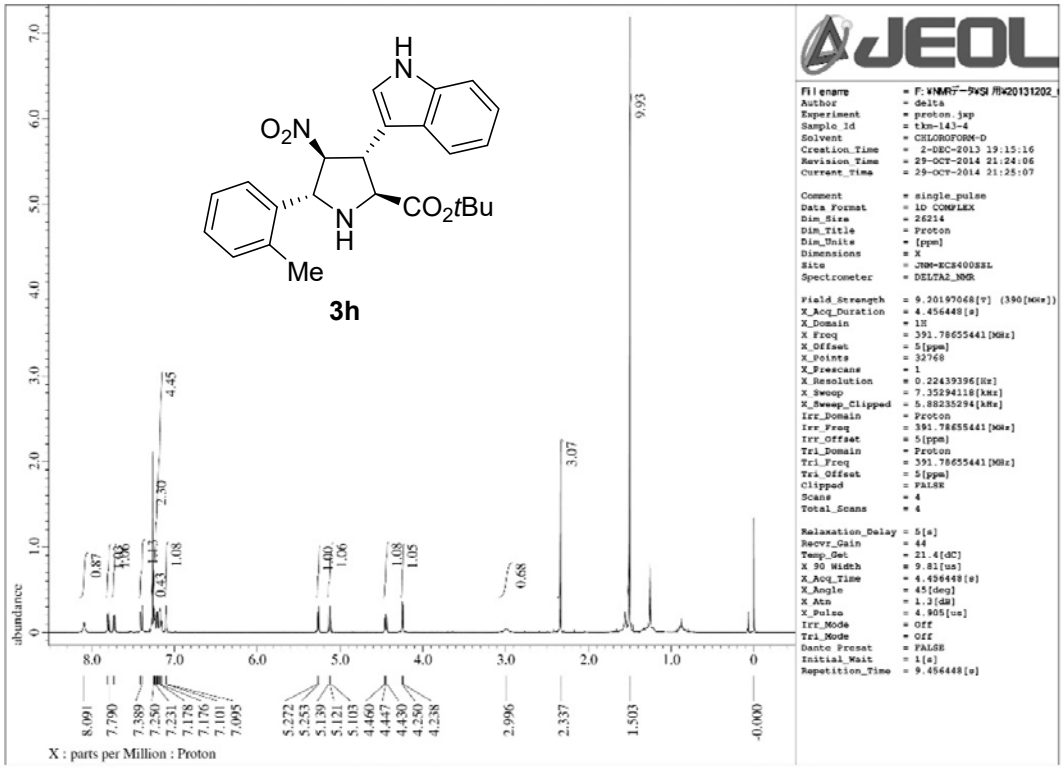
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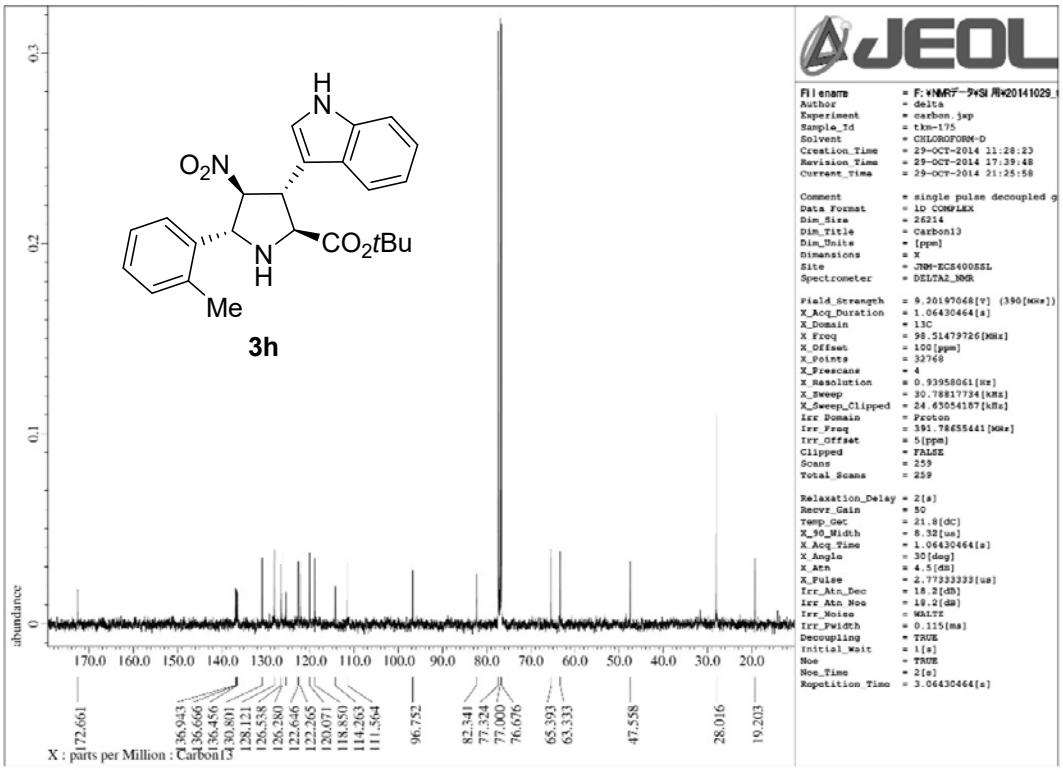
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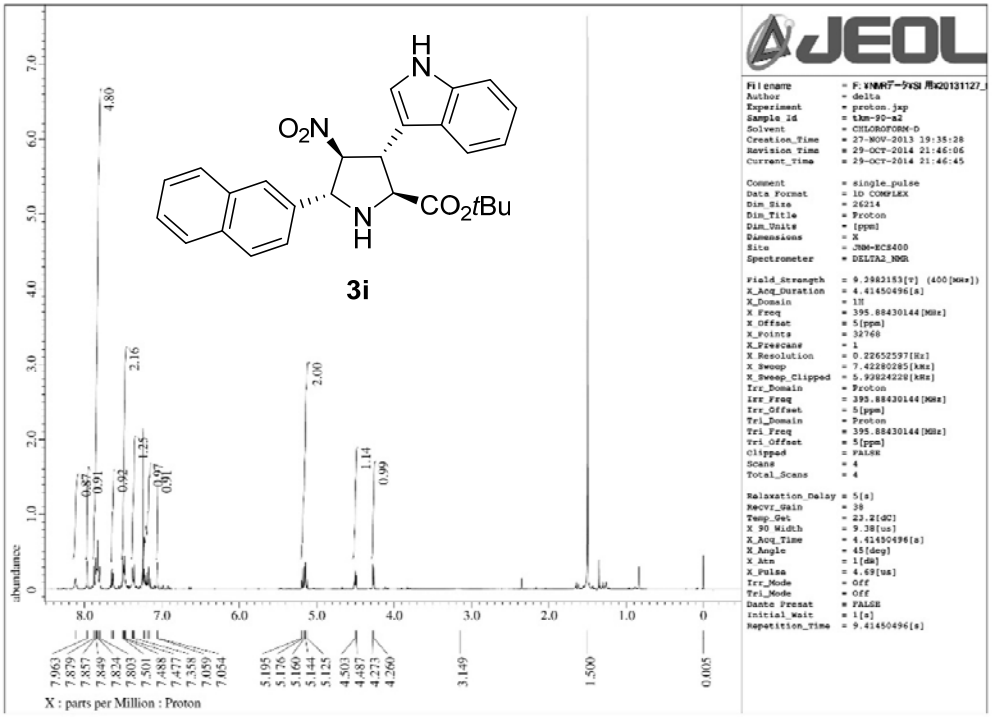
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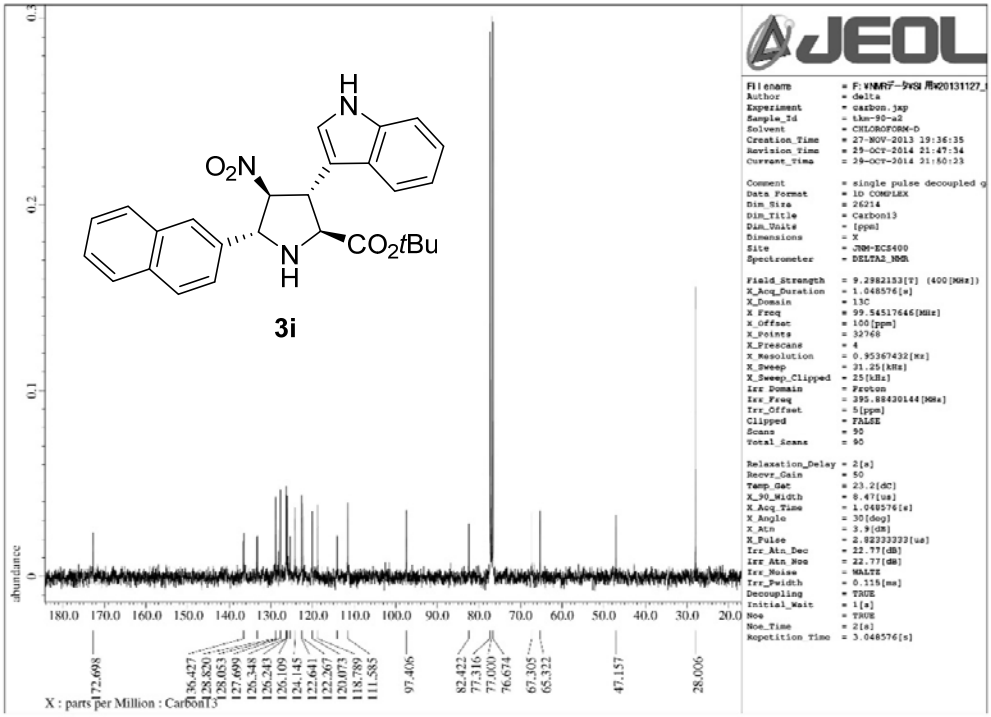
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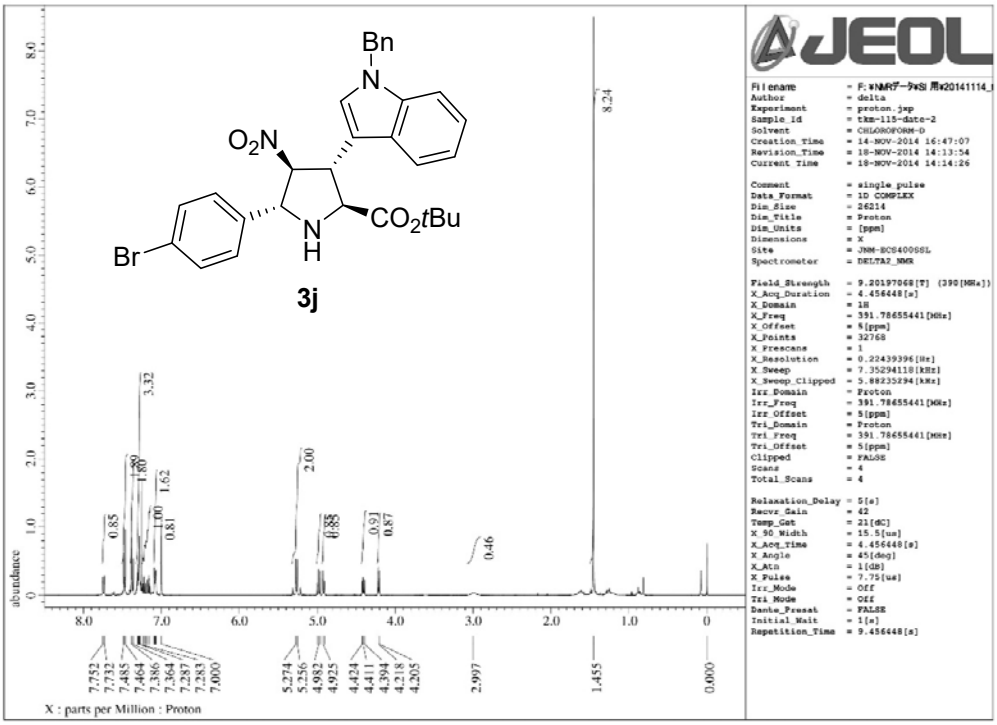
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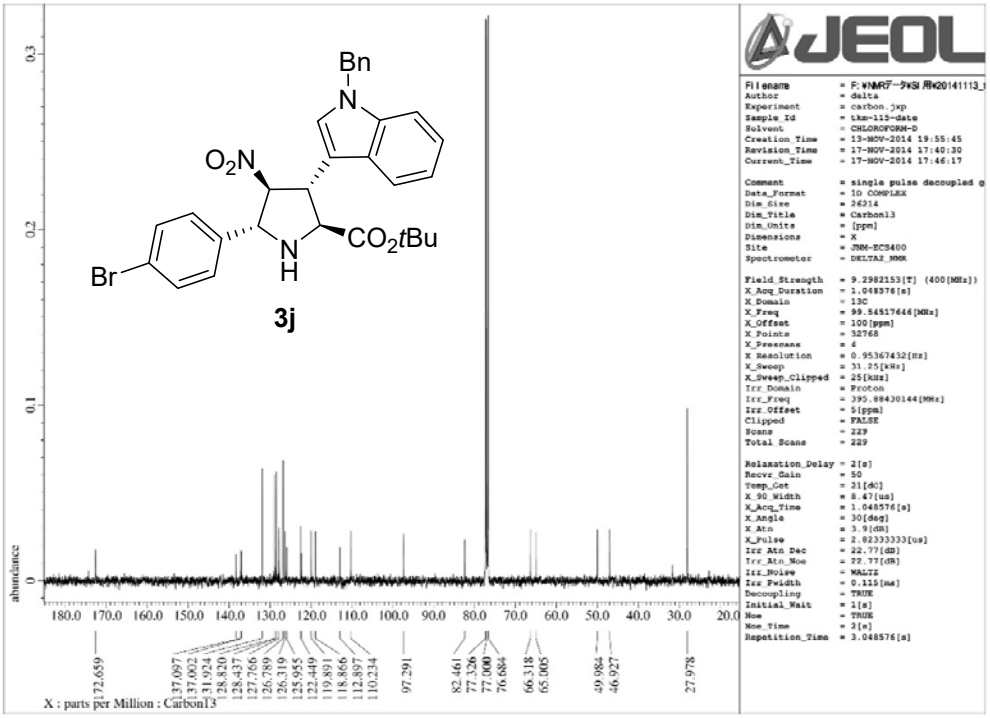
O2N
CO2tBu
N
H
Br
3j
JEOL
X : parts per Million : Proton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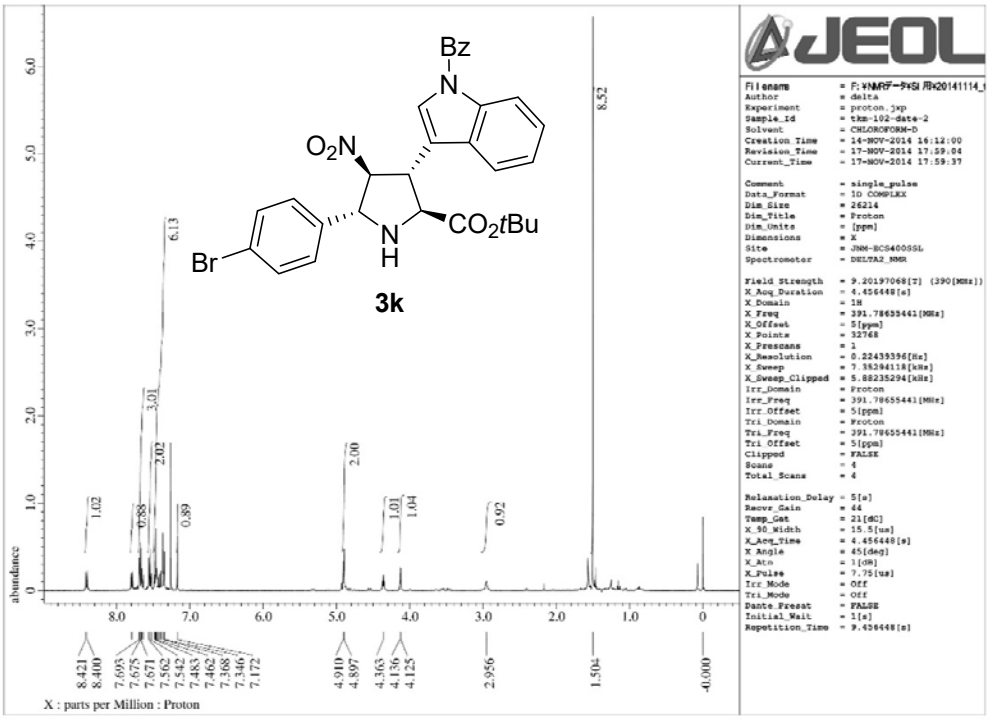
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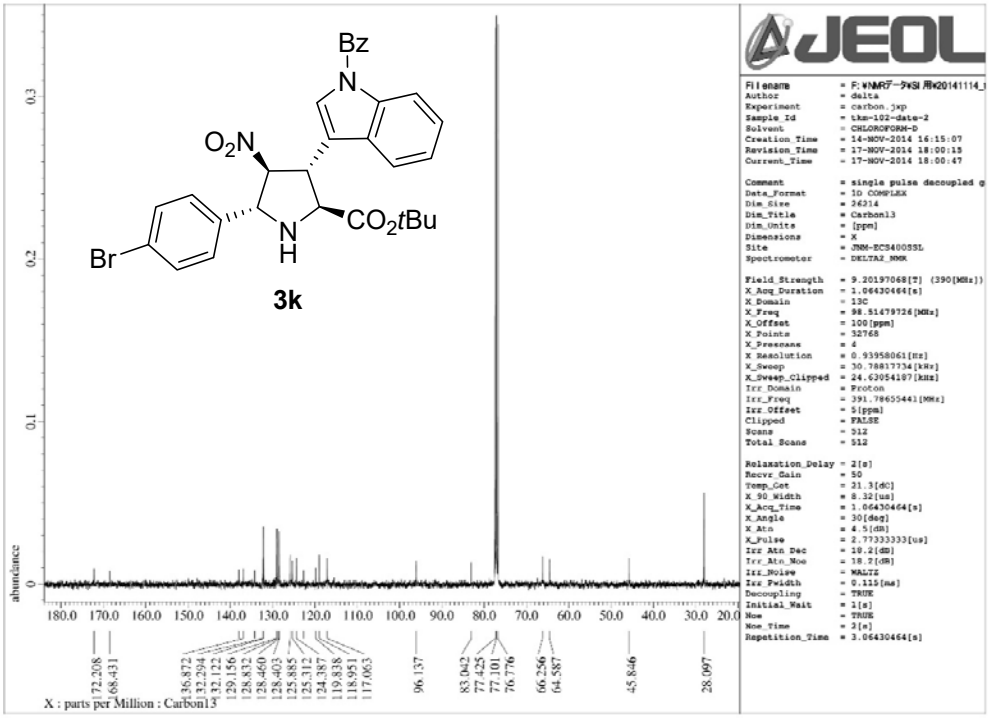
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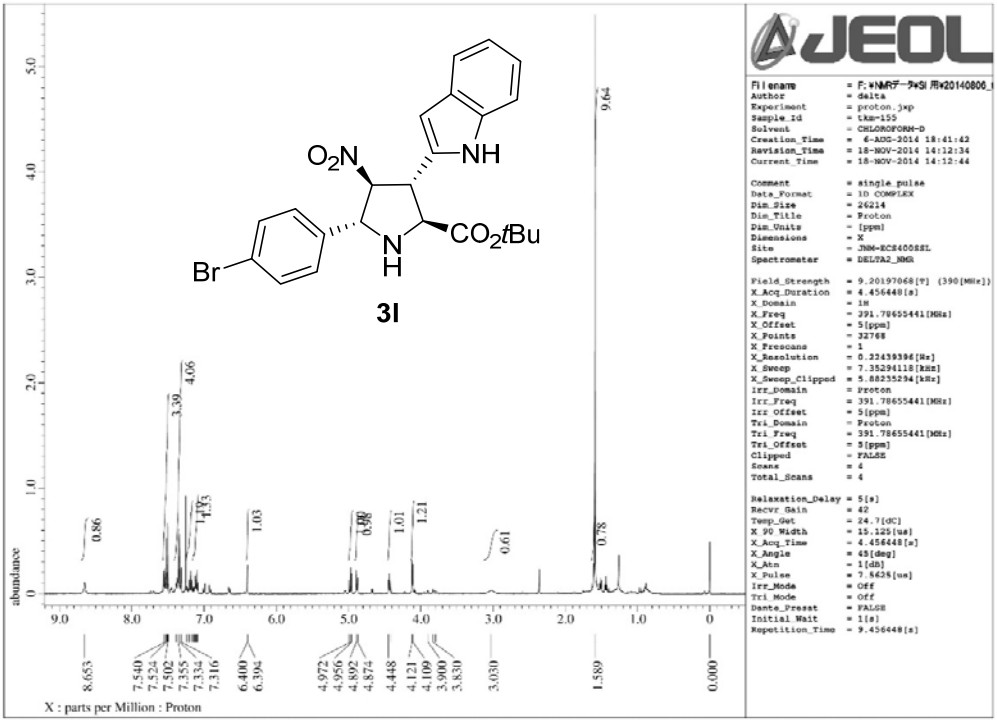
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Br
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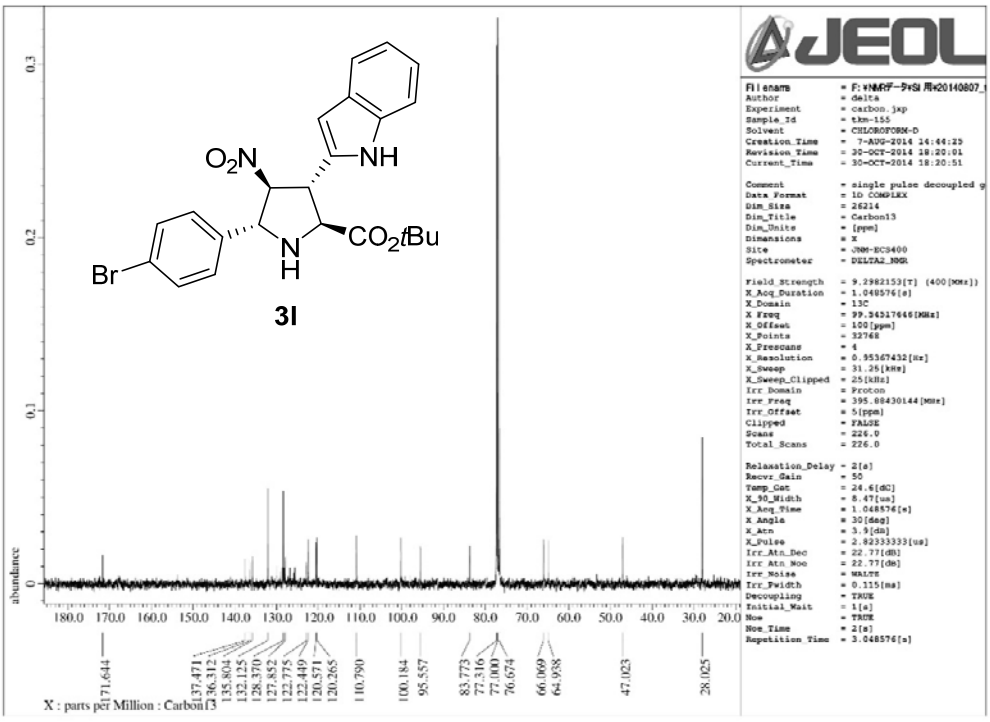
abundance
X : parts per Million : Proton
JEOL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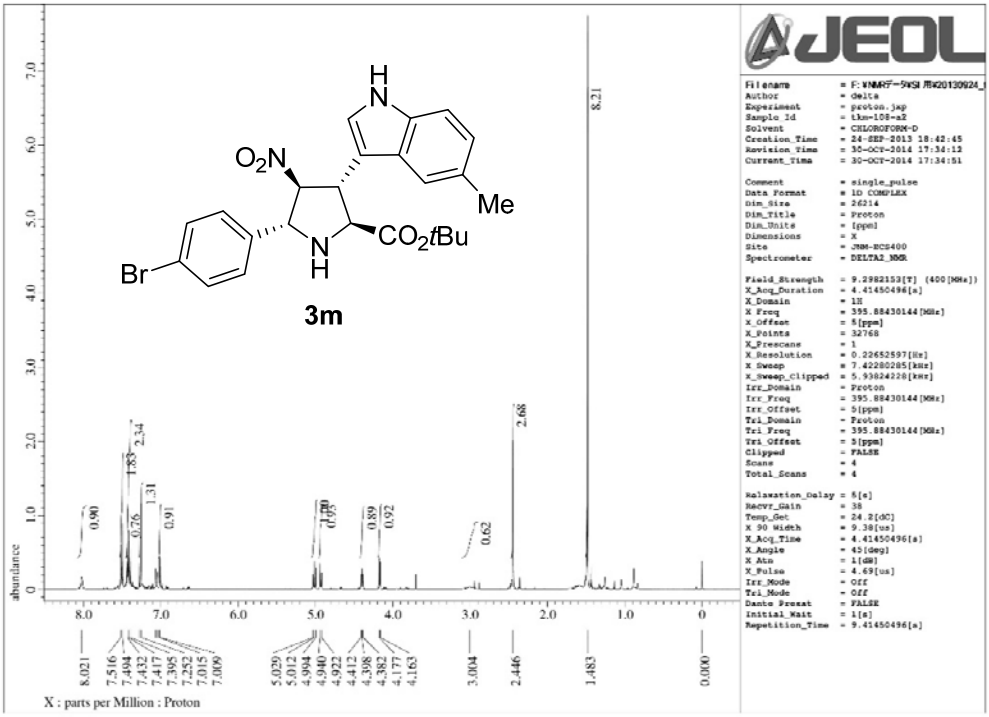
abundance
X : parts per Million : Carbon13
JEOL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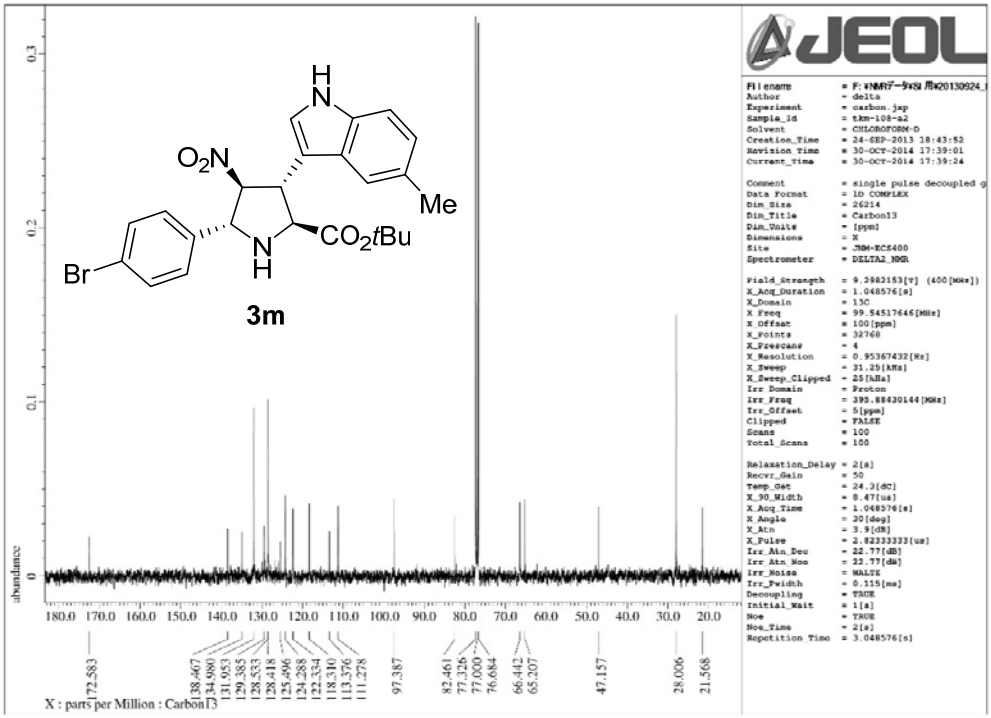
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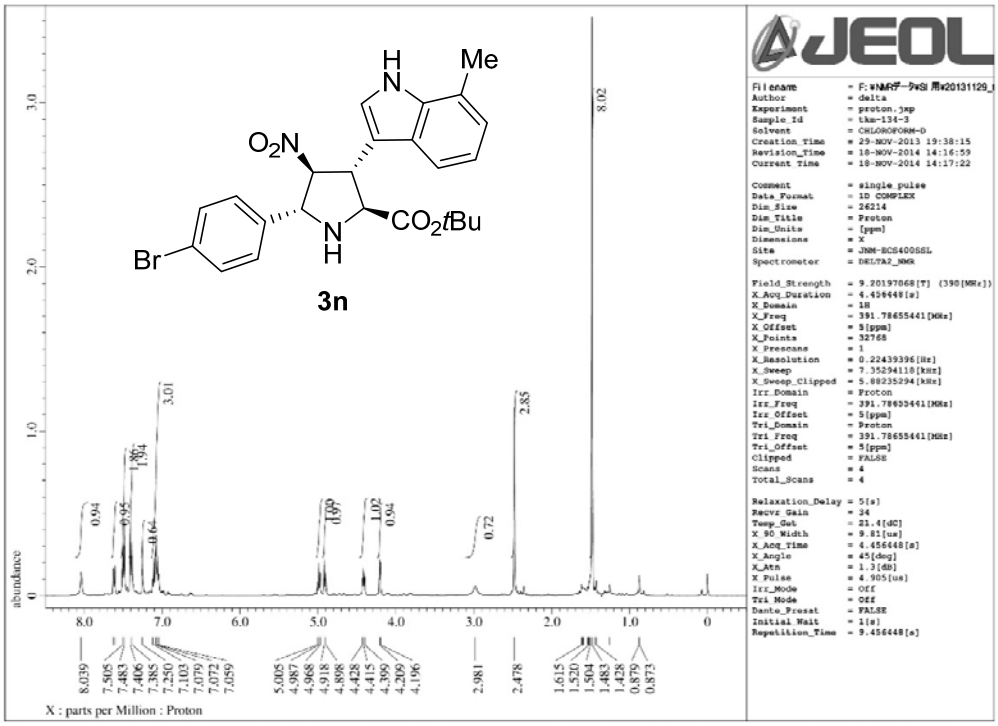
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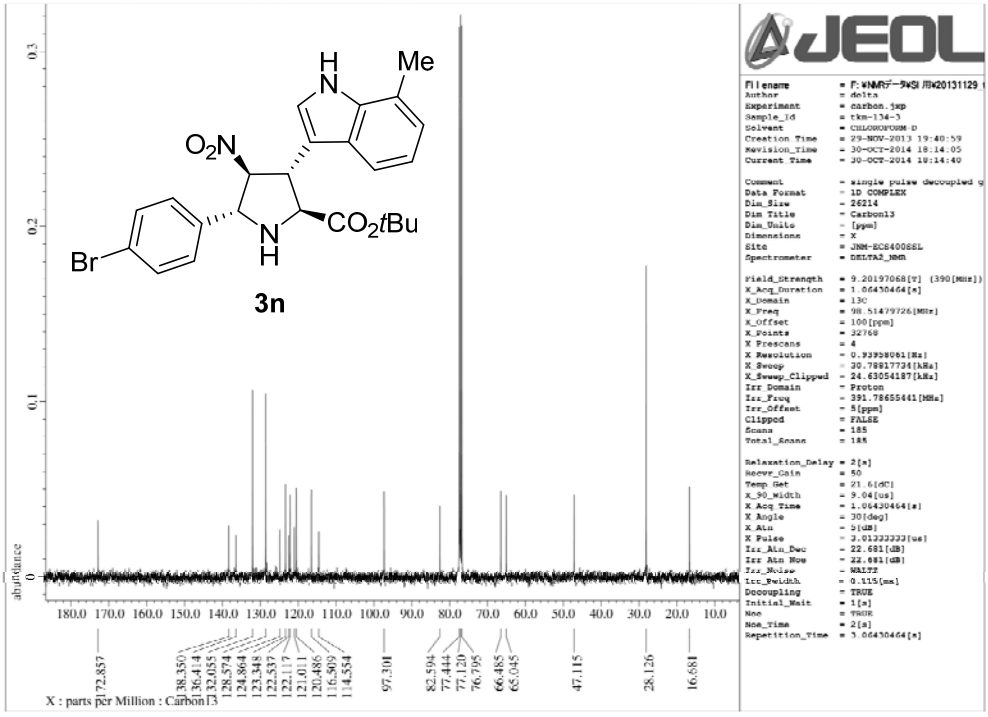
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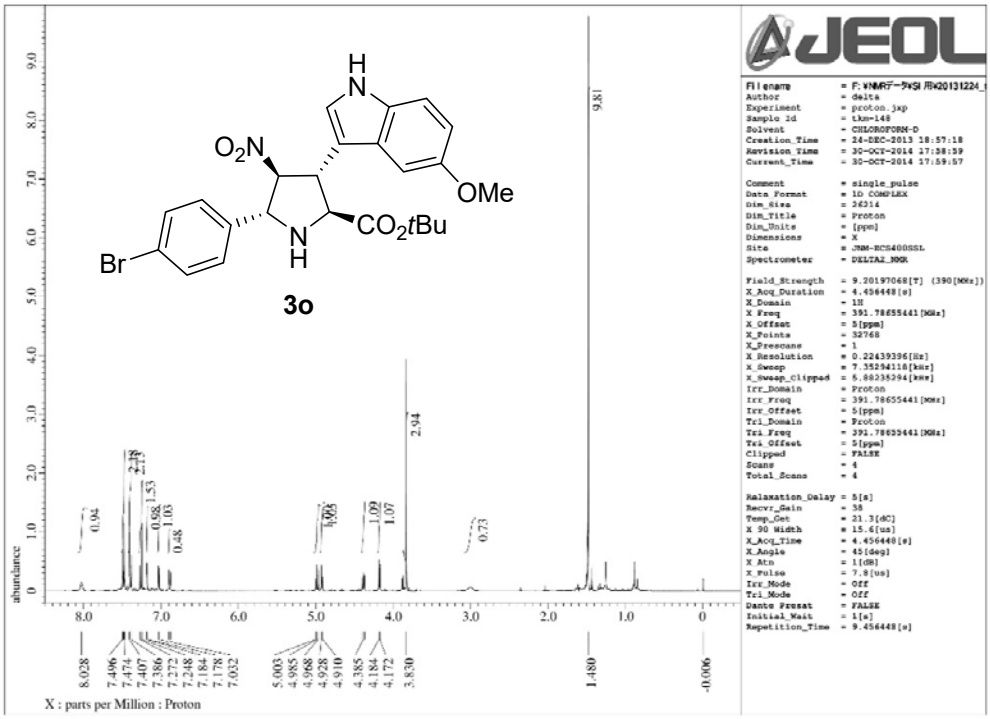
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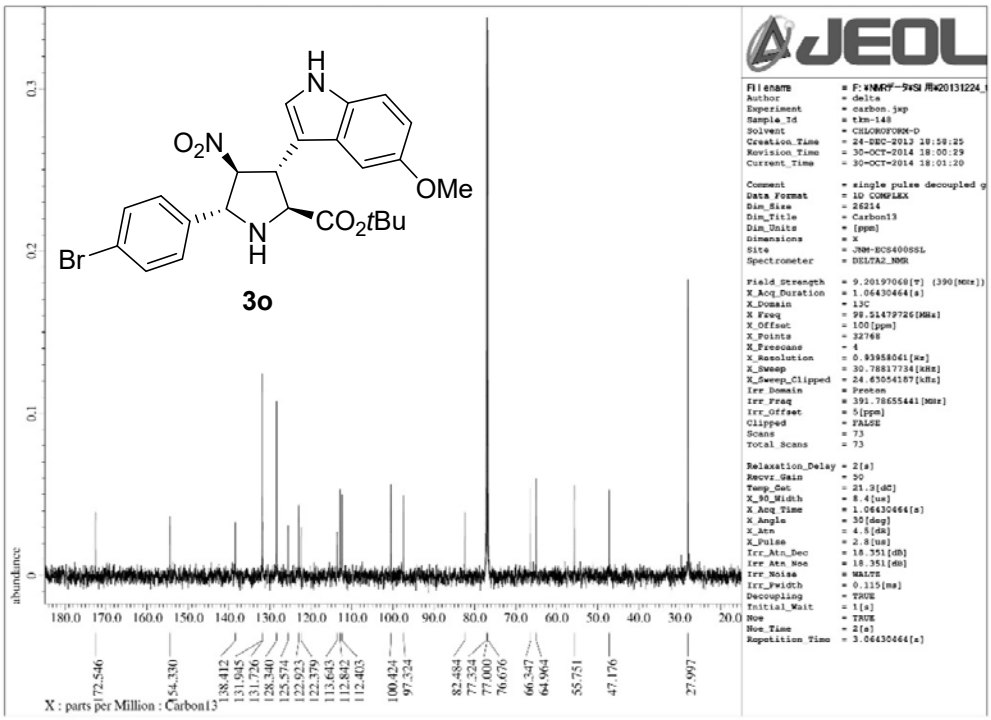
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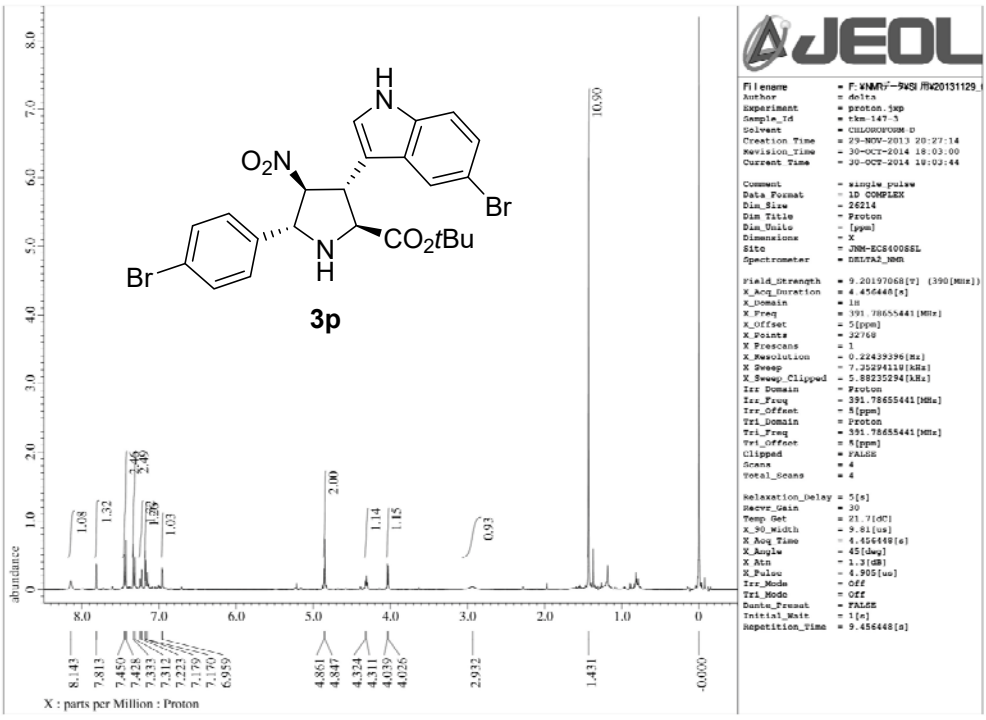
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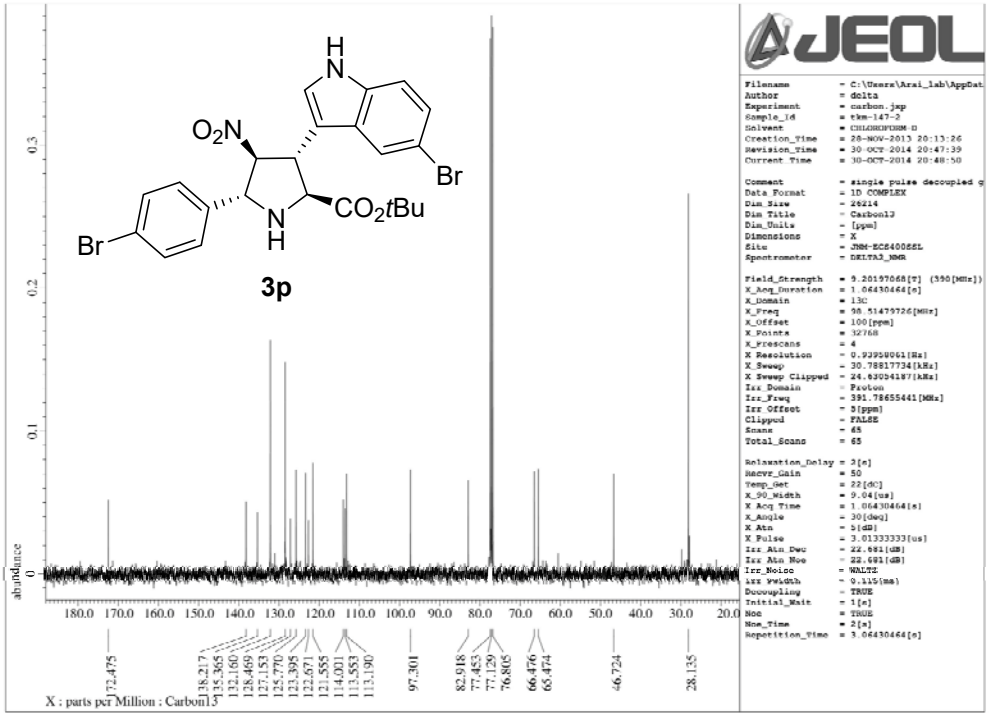
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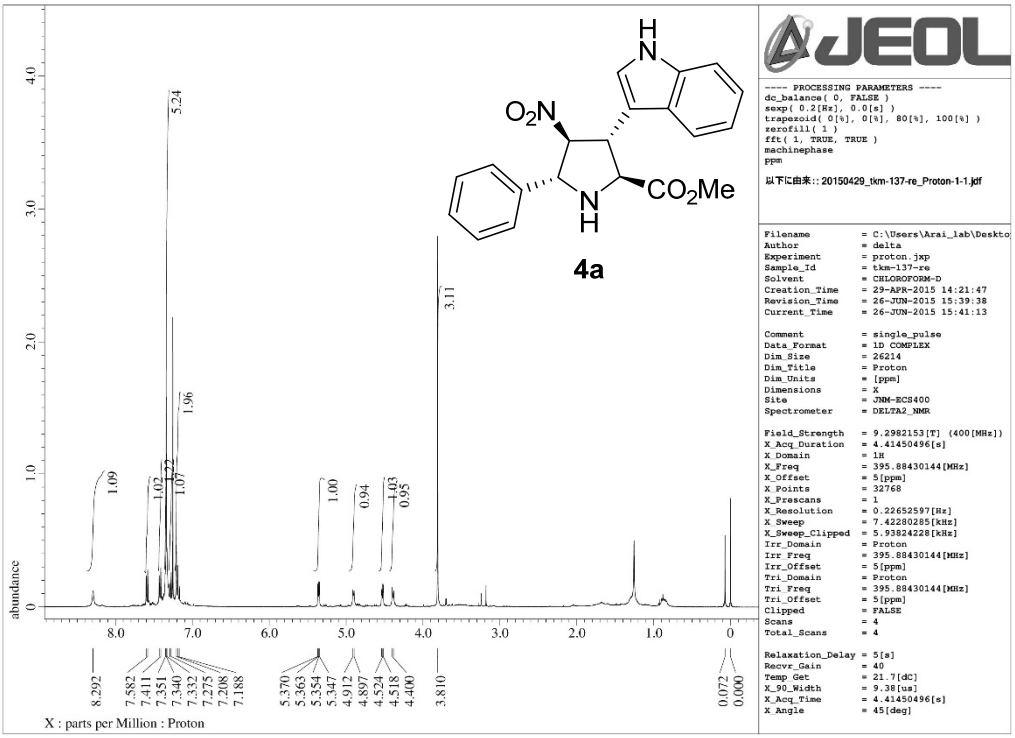
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X : parts per Million : Proton
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zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
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Experiment = proton.jxp
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Dimensions = X
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X_Sweep_Clipped = 5.93824228[kHz]
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Tri_Offset = 5[ppm]
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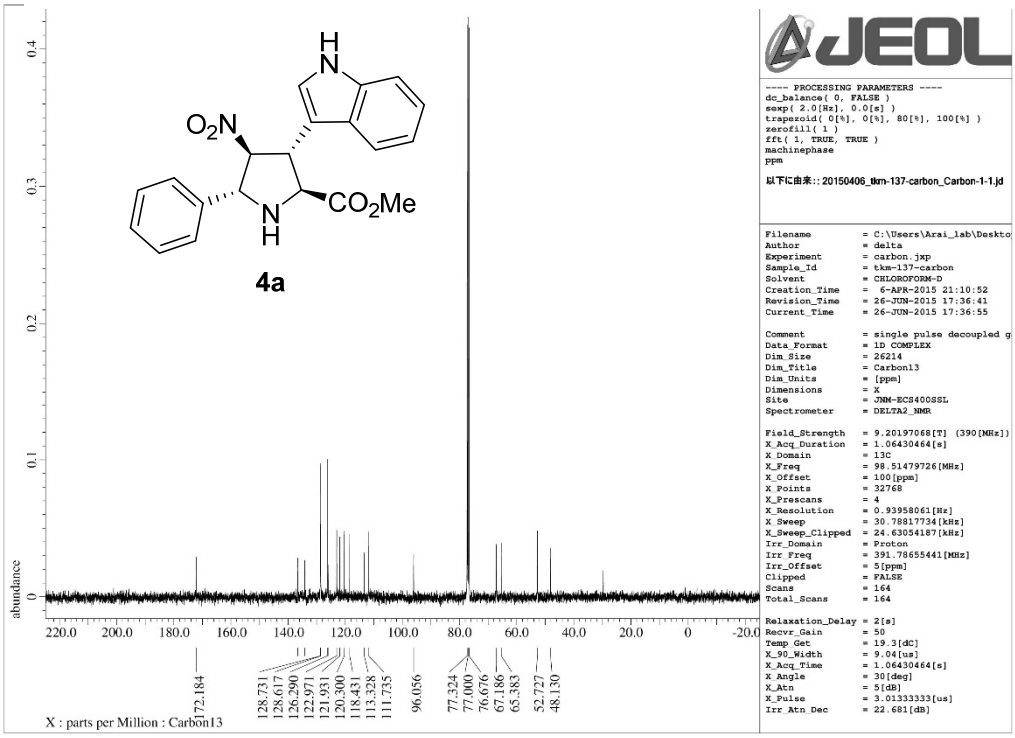
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X : parts per Million : Carbon13
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machinephase
ppm
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X_Sweep_Clipped = 24.63054187[kHz]
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Irr_Freq = 391.78655441[MHz]
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X_Acq_Time = 1.06430464[s]
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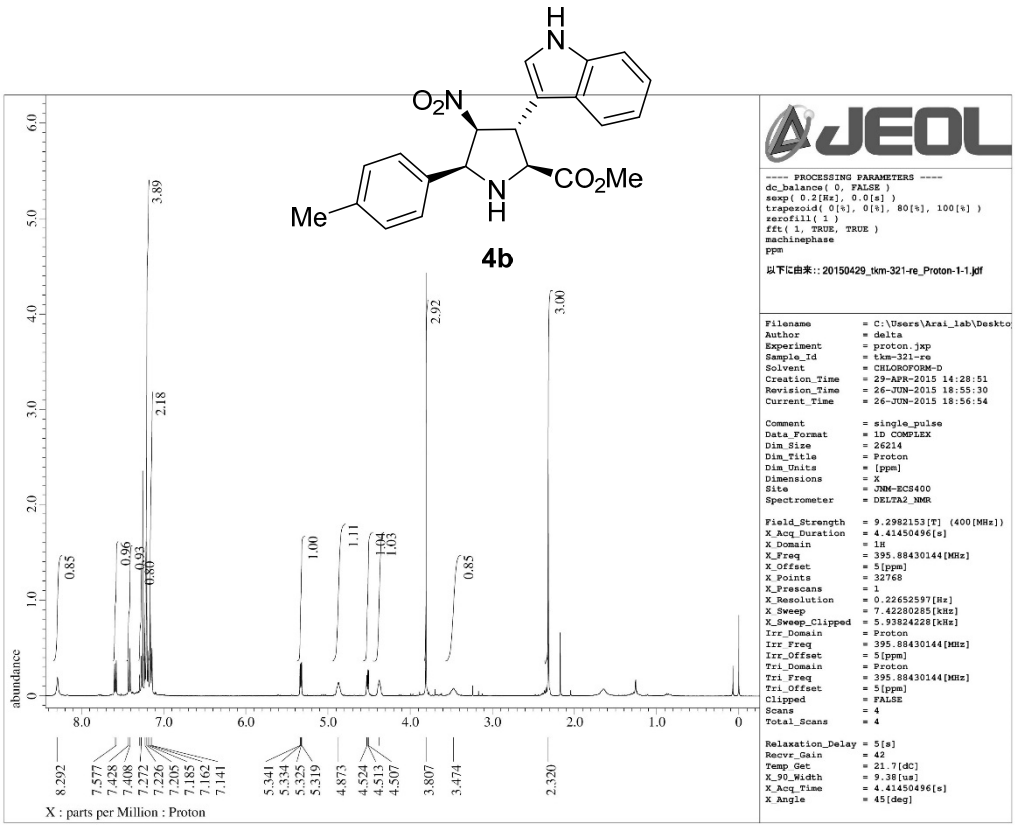
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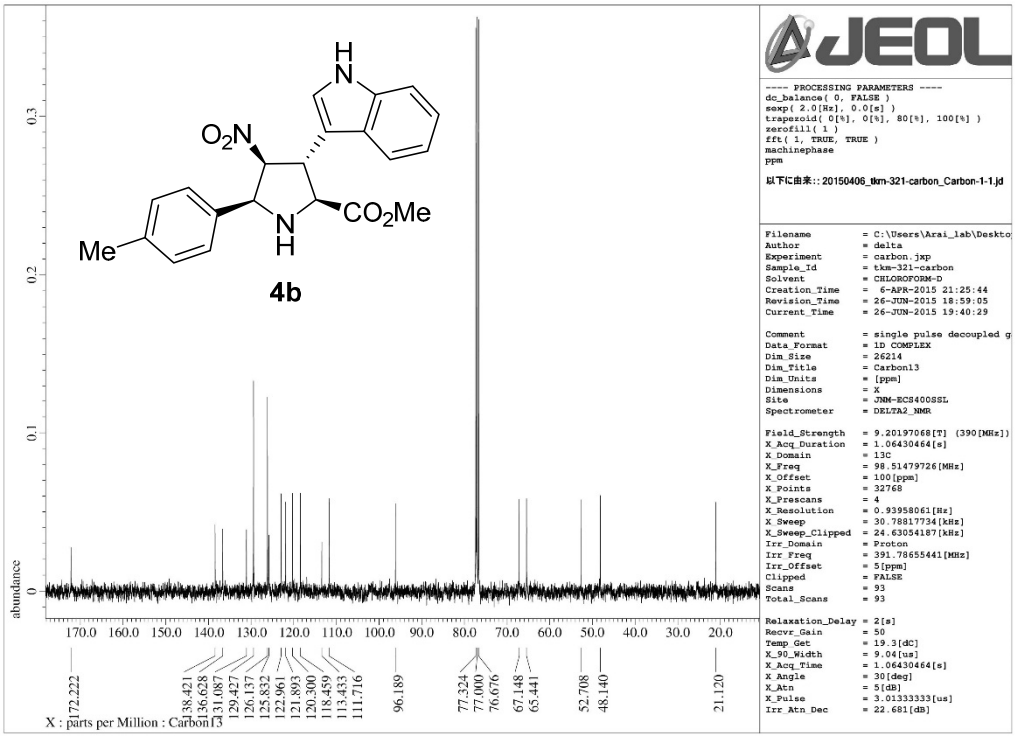
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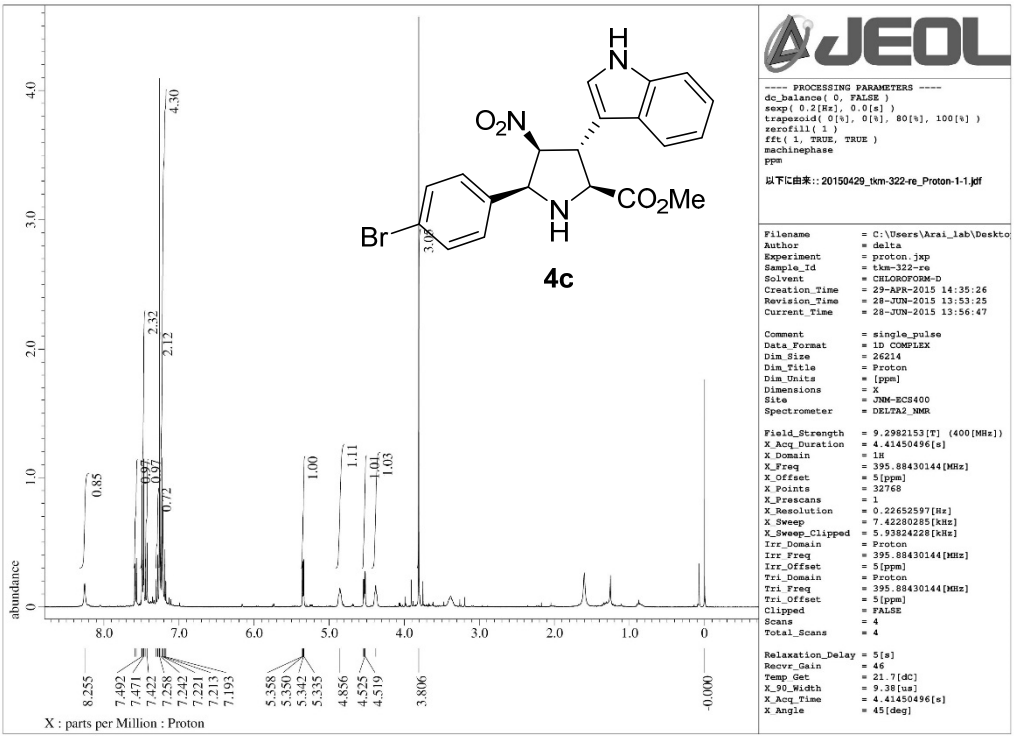
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abundance
X : parts per Million : Proton
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X_Acq_Duration = 4.41450496[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.22652597[Hz]
X_Sweep = 7.42280285[kHz]
X_Sweep_Clipped = 5.93824228[kHz]
Irr_Domain = Proton
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
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Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
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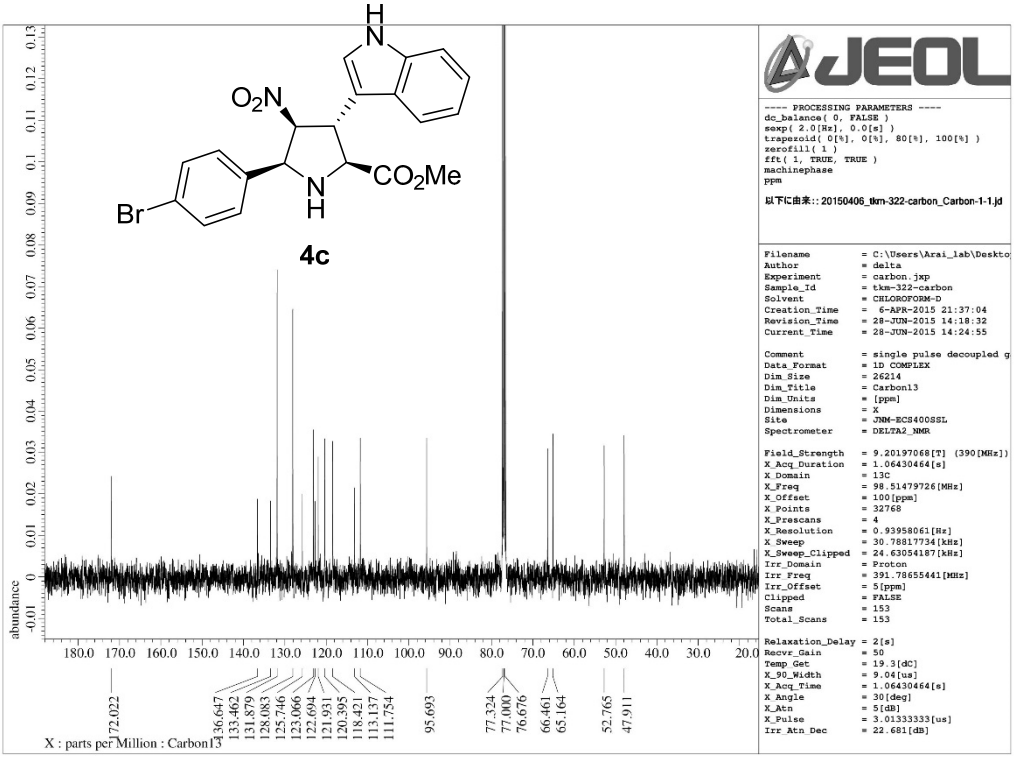
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abundance
X : parts per Million : Carbon13
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trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
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X_Freq = 98.51479726[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.93958061[Hz]
X_Sweep = 30.78817734[kHz]
X_Sweep_Clipped = 24.63054187[kHz]
Irr_Domain = Proton
Irr_Freq = 391.78655441[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 153
Total_Scans = 153
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.3[dC]
X_90_Width = 9.04[us]
X_Acq_Time = 1.06430464[s]
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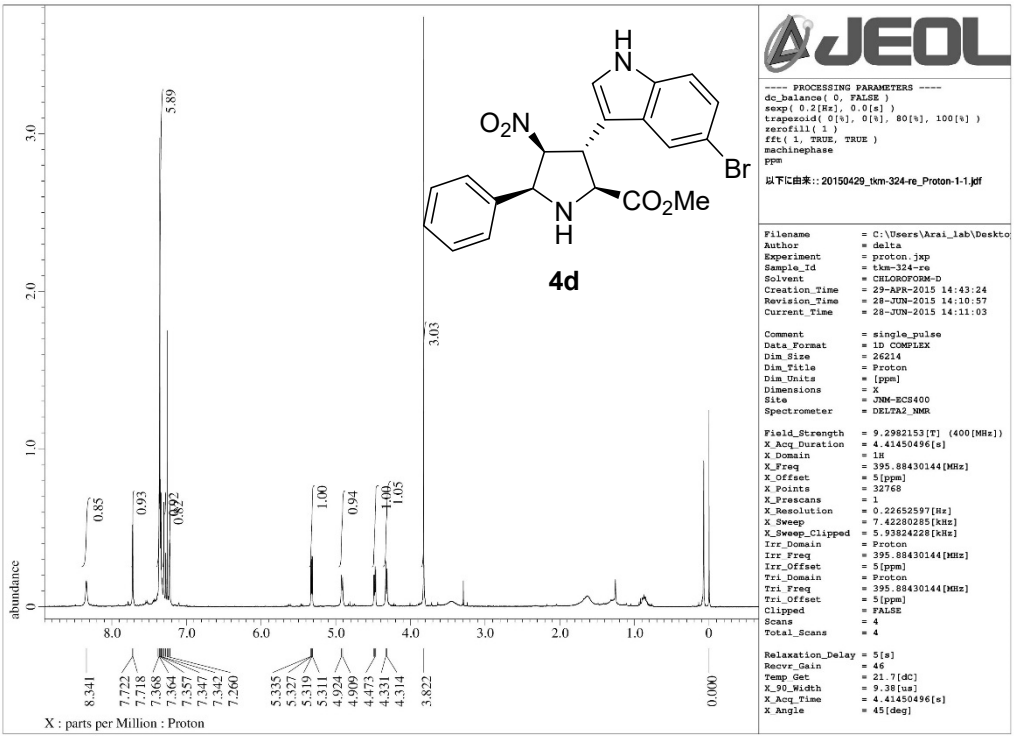
X_Atn = 5[dB]
X_Pulse = 3.01333333[us]
Irr_Atn_Dec = 22.681[dB]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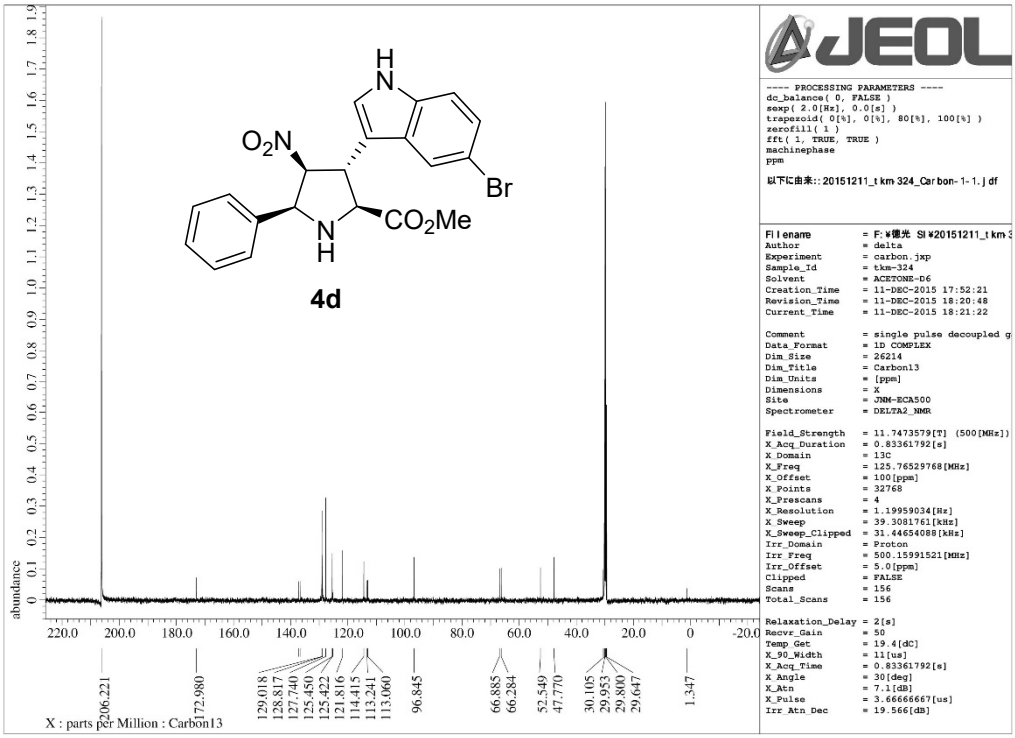
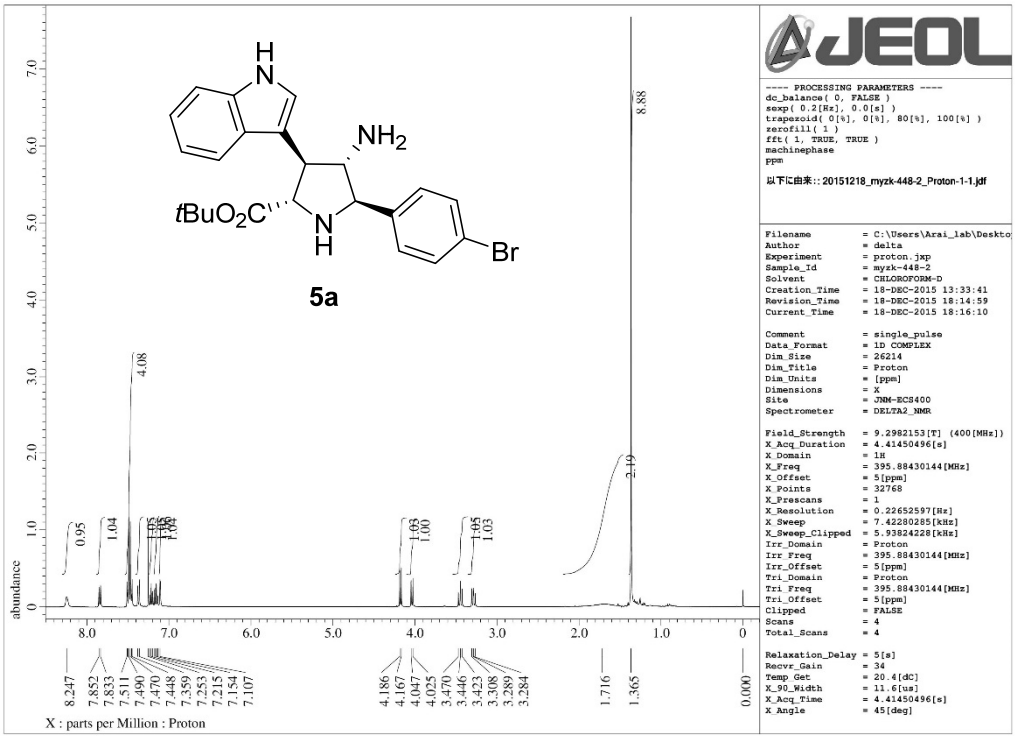
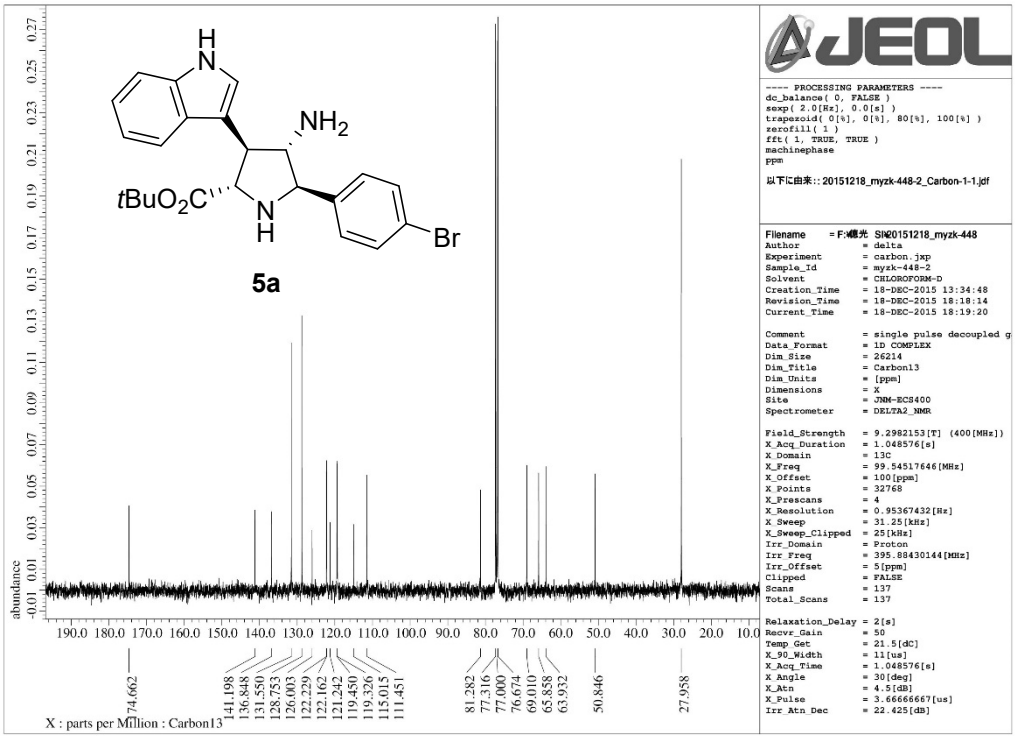
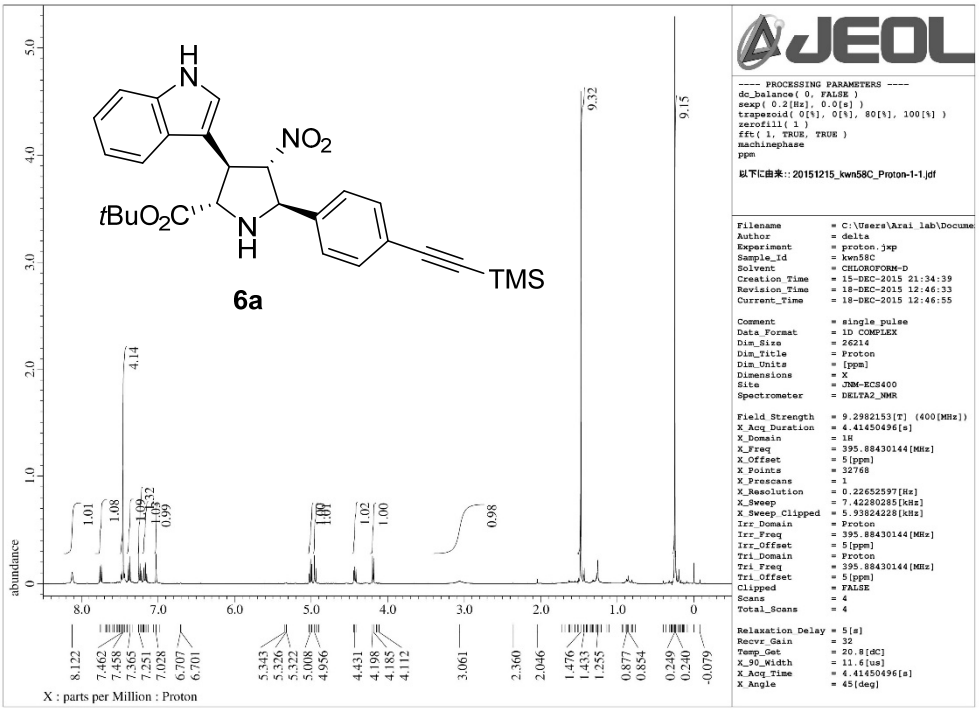
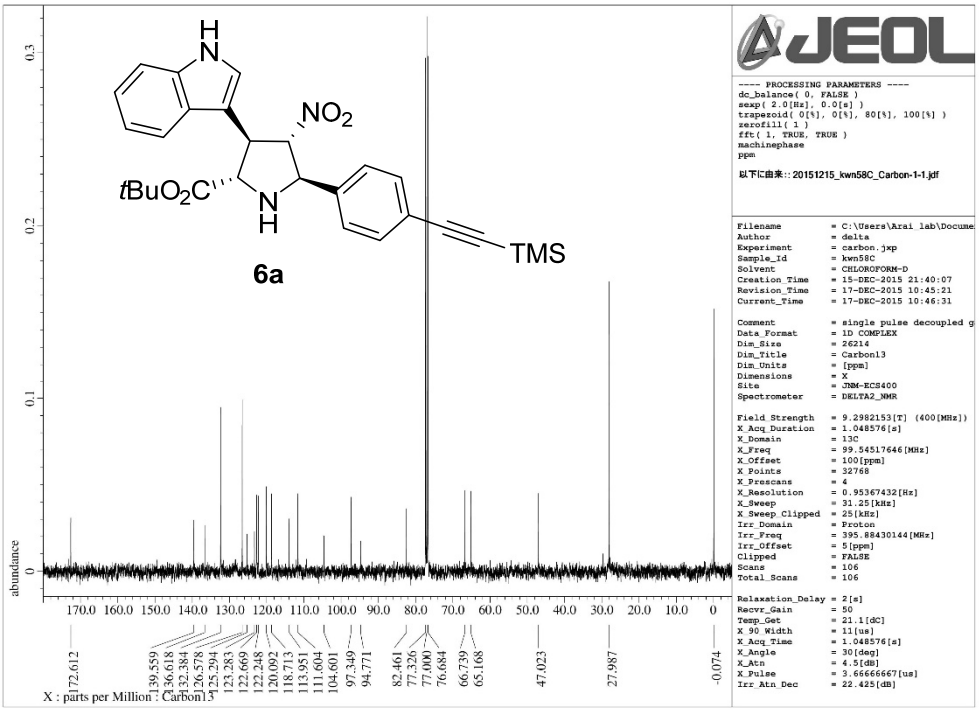
abundance
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 20150429_tkm-324-re_Proton-1-1.jdf
Filename = C:\Users\Arai_lab\Deskto
Author = delta
Experiment = proton.jxp
Sample_Id = tkm-324-re
Solvent = CHLOROFORM-D
Creation_Time = 29-APR-2015 14:43:24
Revision_Time = 28-JUN-2015 14:10:57
Current_Time = 28-JUN-2015 14:11:03
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 4.41450496[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.22652597[Hz]
X_Sweep = 7.42280285[kHz]
X_Sweep_Clipped = 5.93824228[kHz]
Irr_Domain = Proton
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 4
Total_Scans = 4
Relaxation_Delay = 5[s]
Recvr_Gain = 46
Temp_Get = 21.7[dC]
X_90_Width = 9.38[us]
X_Acq_Time = 4.41450496[s]
X_Angle = 45[deg]


4d
abundance
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来:: 20151211_t km 324_Car bon-1-1. j df
Fi l ename = F: ¥環光 Si ¥20151211_t km 3
Author = delta
Experiment = carbon.jxp
Sample_Id = tkm-324
Solvent = ACETONE-D6
Creation_Time = 11-DEC-2015 17:52:21
Revision_Time = 11-DEC-2015 18:20:48
Current_Time = 11-DEC-2015 18:21:22
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA500
Spectrometer = DELTA2_NMR
Field_Strength = 11.7473579[T] (500[MHz])
X_Acq_Duration = 0.83361792[s]
X_Domain = 13C
X_Freq = 125.76529768[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.19959034[Hz]
X_Sweep = 39.3081761[kHz]
X_Sweep_Clipped = 31.44654088[kHz]
Irr_Domain = Proton
Irr_Freq = 500.15991521[MHz]
Irr_Offset = 5.0[ppm]
Clipped = FALSE
Scans = 156
Total_Scans = 156
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 19.4[dC]
X_90_Width = 11[us]
X_Acq_Time = 0.83361792[s]
X_Angle = 30[deg]
X_Atn = 7.1[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 19.566[dB]


5a
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来：: 20151218_myzk-448-2_Proton-1-1.jdf
Filename = C:\Users\Arai_lab\Deskto
Author = delta
Experiment = proton.jxp
Sample_Id = myzk-448-2
Solvent = CHLOROFORM-D
Creation_Time = 18-DEC-2015 13:33:41
Revision_Time = 18-DEC-2015 18:14:59
Current_Time = 18-DEC-2015 18:16:10
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 4.41450496[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.22652597[Hz]
X_Sweep = 7.42280285[kHz]
X_Sweep_Clipped = 5.93824228[kHz]
Irr_Domain = Proton
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 4
Total_Scans = 4
Relaxation_Delay = 5[s]
Recvr_Gain = 34
Temp_Get = 20.4[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 4.41450496[s]
X_Angle = 45[deg]

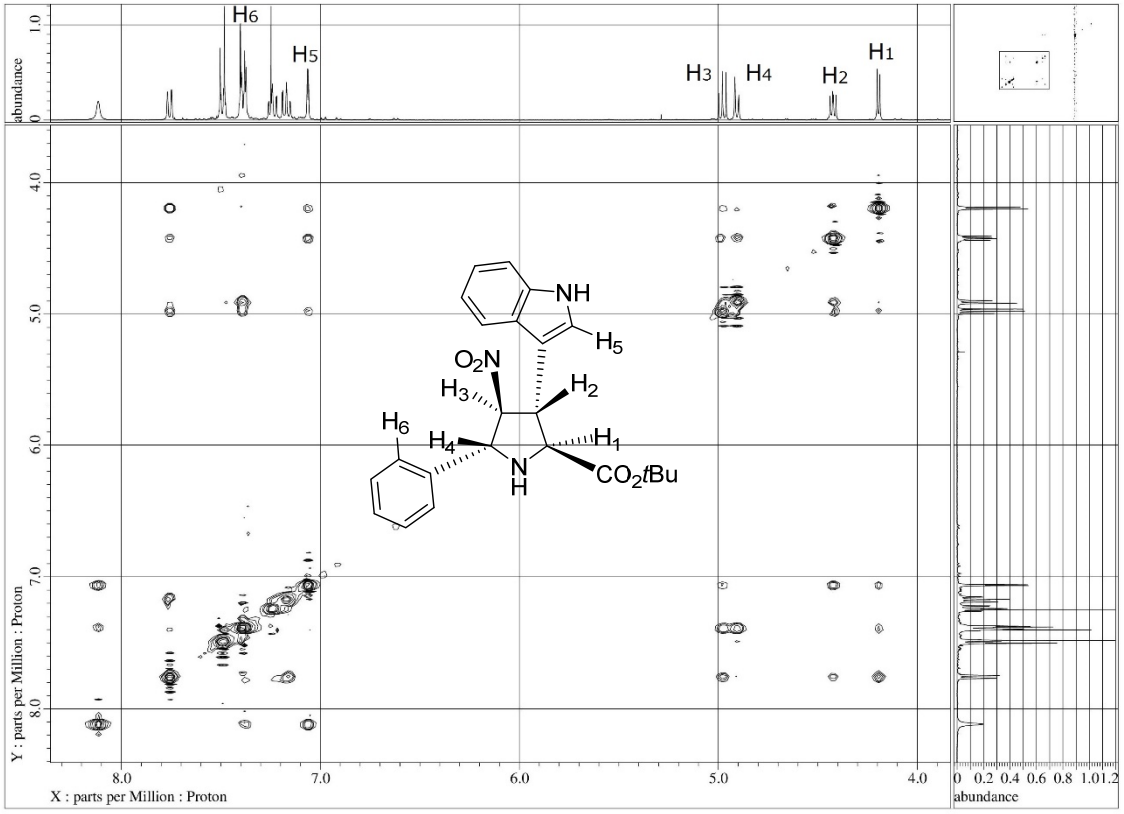

5a
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来：: 20151218_myzk-448-2_Carbon-1-1.jdf
Filename = F:\構光 Sh\20151218_myzk-448
Author = delta
Experiment = carbon.jxp
Sample_Id = myzk-448-2
Solvent = CHLOROFORM-D
Creation_Time = 18-DEC-2015 13:34:48
Revision_Time = 18-DEC-2015 18:18:14
Current_Time = 18-DEC-2015 18:19:20
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 1.048576[s]
X_Domain = 13C
X_Freq = 99.54517646[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95367432[Hz]
X_Sweep = 31.25[kHz]
X_Sweep_Clipped = 25[kHz]
Irr_Domain = Proton
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 137
Total_Scans = 137
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.5[dC]
X_90_Width = 11[us]
X_Acq_Time = 1.048576[s]
X_Angle = 30[deg]
X_Atn = 4.5[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 22.425[dB]


6a
TMS
X : parts per Million : Proton
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(0.2[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来：: 20151215_kwn58C_Proton-1-1.jdf
Filename = C:\Users\Arai lab\Docume
Author = delta
Experiment = proton.jxp
Sample_Id = kwn58C
Solvent = CHLOROFORM-D
Creation_Time = 15-DEC-2015 21:34:39
Revision_Time = 18-DEC-2015 12:46:33
Current_Time = 18-DEC-2015 12:46:55
Comment = single_pulse
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Proton
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 4.41450496[s]
X_Domain = 1H
X_Freq = 395.88430144[MHz]
X_Offset = 5[ppm]
X_Points = 32768
X_Prescans = 1
X_Resolution = 0.22652597[Hz]
X_Sweep = 7.42280285[kHz]
X_Sweep_Clipped = 5.93824228[kHz]
Irr_Domain = Proton
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Tri_Domain = Proton
Tri_Freq = 395.88430144[MHz]
Tri_Offset = 5[ppm]
Clipped = FALSE
Scans = 4
Total_Scans = 4
Relaxation_Delay = 5[s]
Recvr_Gain = 32
Temp_Get = 20.8[dC]
X_90_Width = 11.6[us]
X_Acq_Time = 4.41450496[s]
X_Angle = 45[deg]

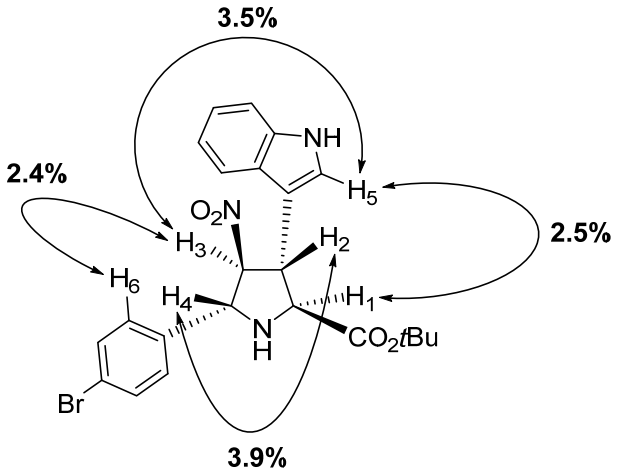

6a
TMS
X : parts per Million : Carbon13
---- PROCESSING PARAMETERS ----
dc_balance(0, FALSE)
sexp(2.0[Hz], 0.0[s])
trapezoid(0[%], 0[%], 80[%], 100[%])
zerofill(1)
fft(1, TRUE, TRUE)
machinephase
ppm
以下に由来：: 20151215_kwn58C_Carbon-1-1.jdf
Filename = C:\Users\Arai lab\Docume
Author = delta
Experiment = carbon.jxp
Sample_Id = kwn58C
Solvent = CHLOROFORM-D
Creation_Time = 15-DEC-2015 21:40:07
Revision_Time = 17-DEC-2015 10:45:21
Current_Time = 17-DEC-2015 10:46:31
Comment = single pulse decoupled g
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECS400
Spectrometer = DELTA2_NMR
Field_Strength = 9.2982153[T] (400[MHz])
X_Acq_Duration = 1.048576[s]
X_Domain = 13C
X_Freq = 99.54517646[MHz]
X_Offset = 100[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 0.95367432[Hz]
X_Sweep = 31.25[kHz]
X_Sweep_Clipped = 25[kHz]
Irr_Domain = Proton
Irr_Freq = 395.88430144[MHz]
Irr_Offset = 5[ppm]
Clipped = FALSE
Scans = 106
Total_Scans = 106
Relaxation_Delay = 2[s]
Recvr_Gain = 50
Temp_Get = 21.1[dC]
X_90_Width = 11[us]
X_Acq_Time = 1.048576[s]
X_Angle = 30[deg]
X_Atn = 4.5[dB]
X_Pulse = 3.66666667[us]
Irr_Atn_Dec = 22.425[dB]

2. Relative configuration of *exo'*-, and *endo*-products

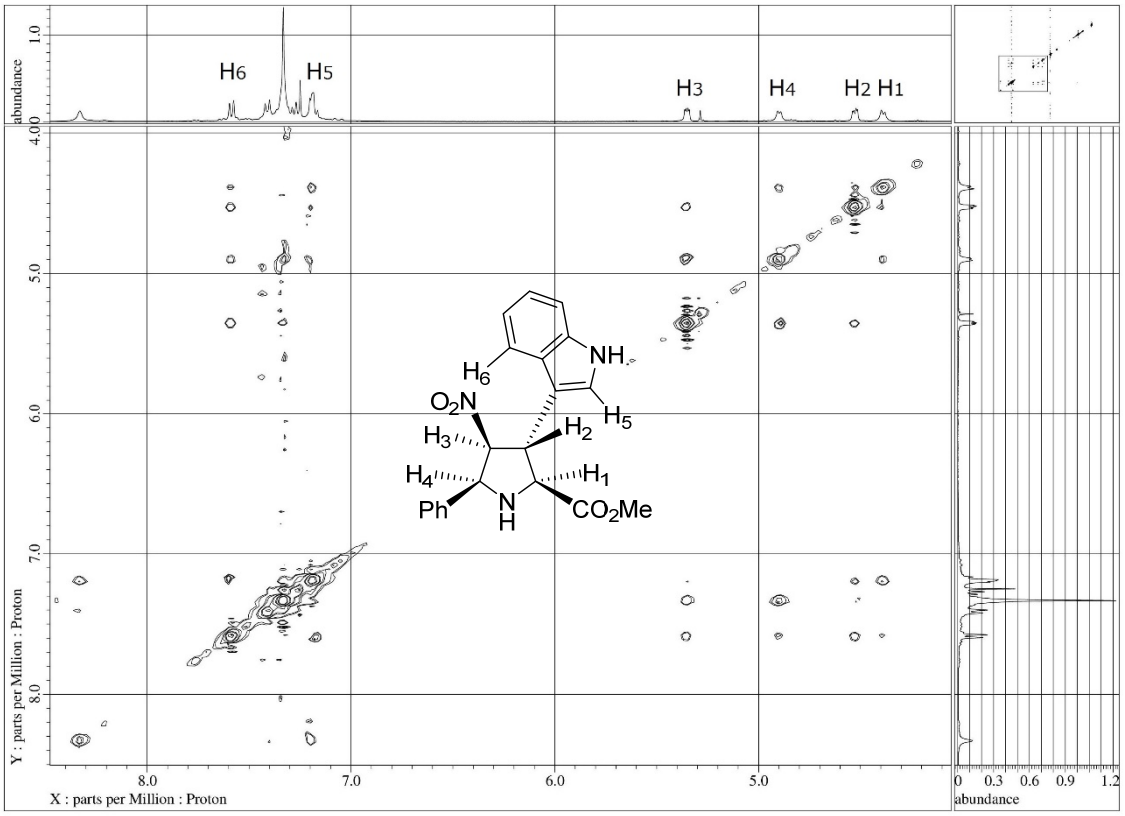
NOESY spectrum of *exo'*-product **3c**



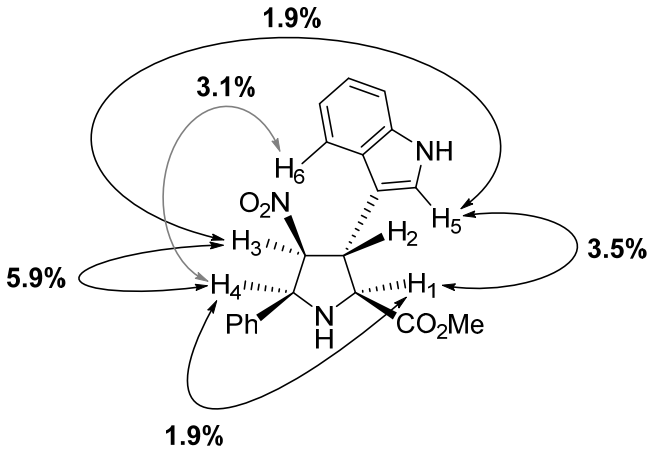
nOe experiment on *exo'*-product **3c**



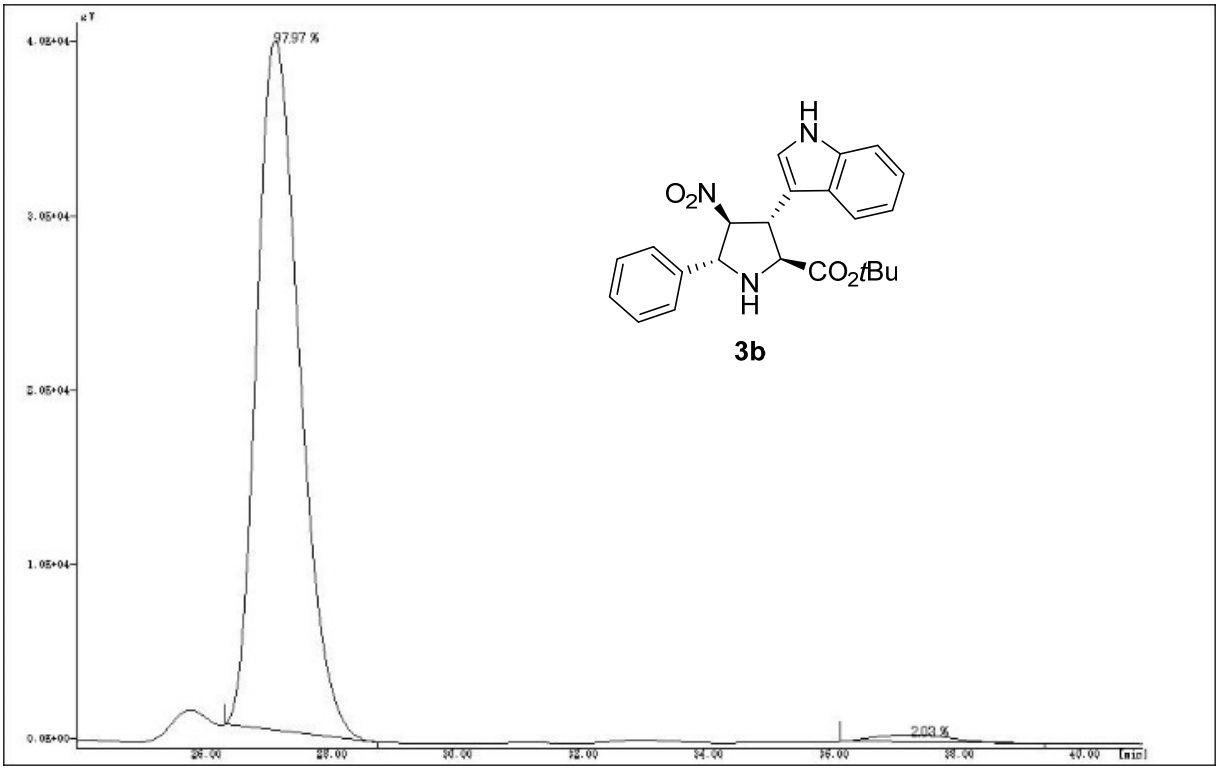
NOESY spectrum of *endo*-product **4a**

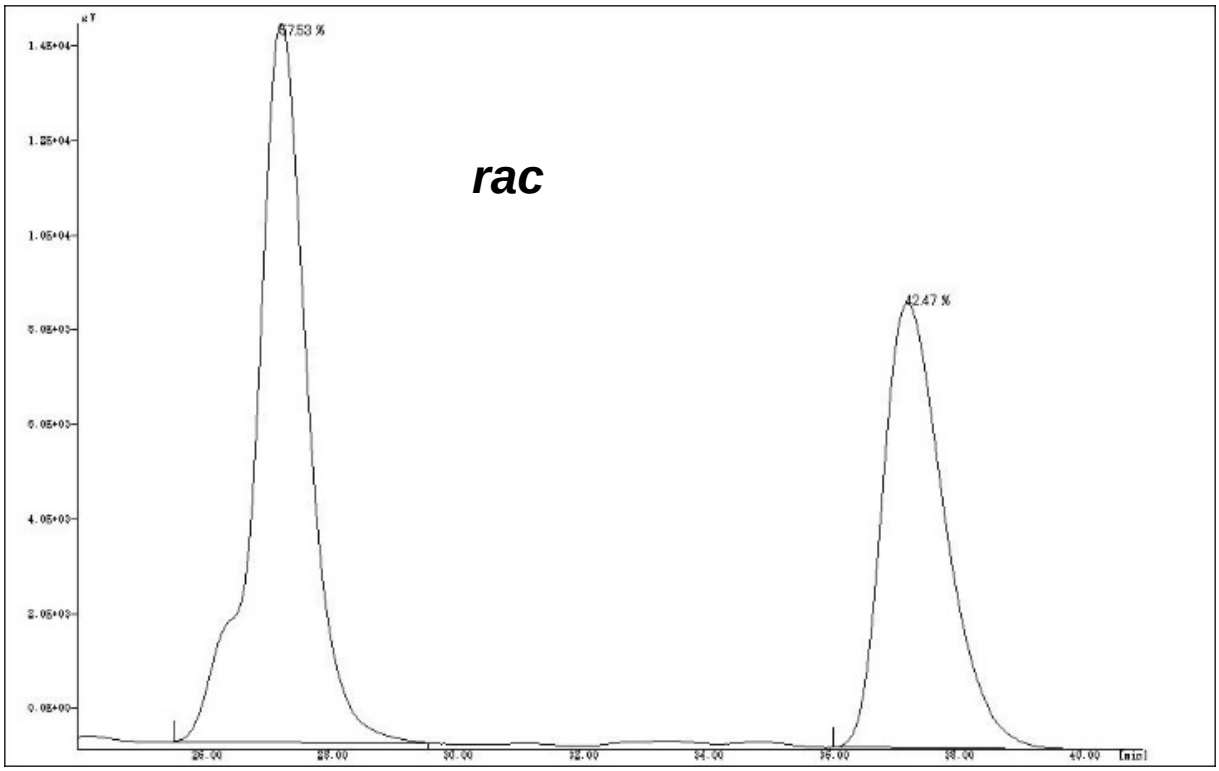


nOe experiment on *endo*-product **4a**

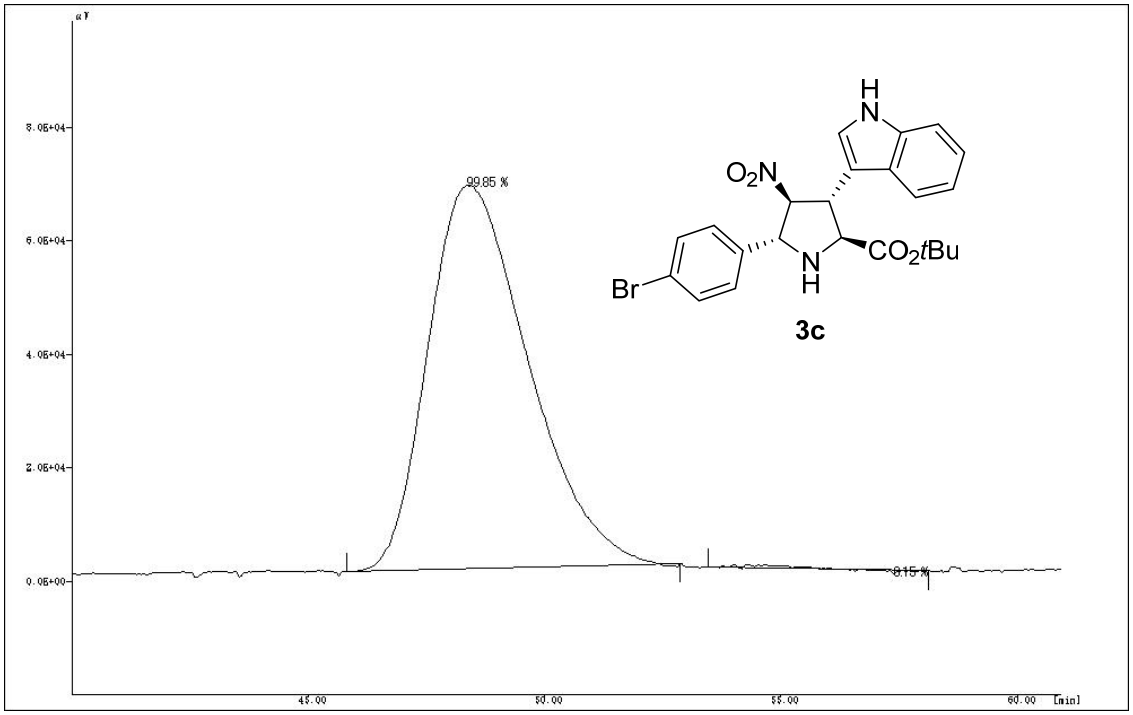


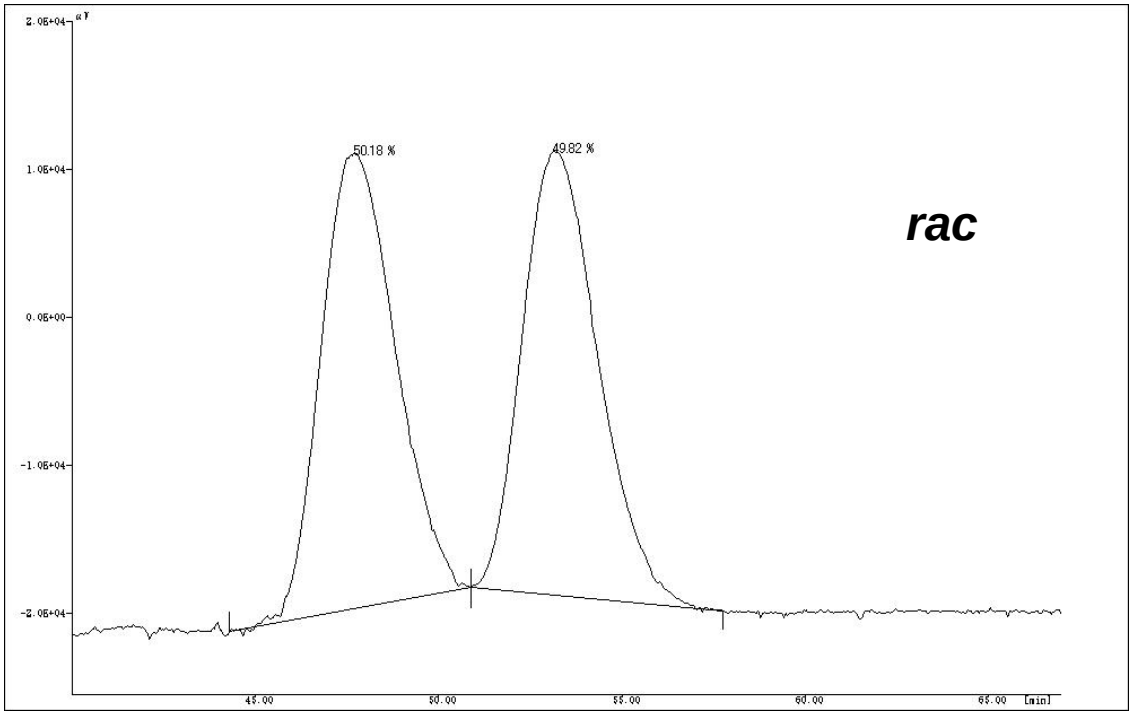
3.HPLC spectra



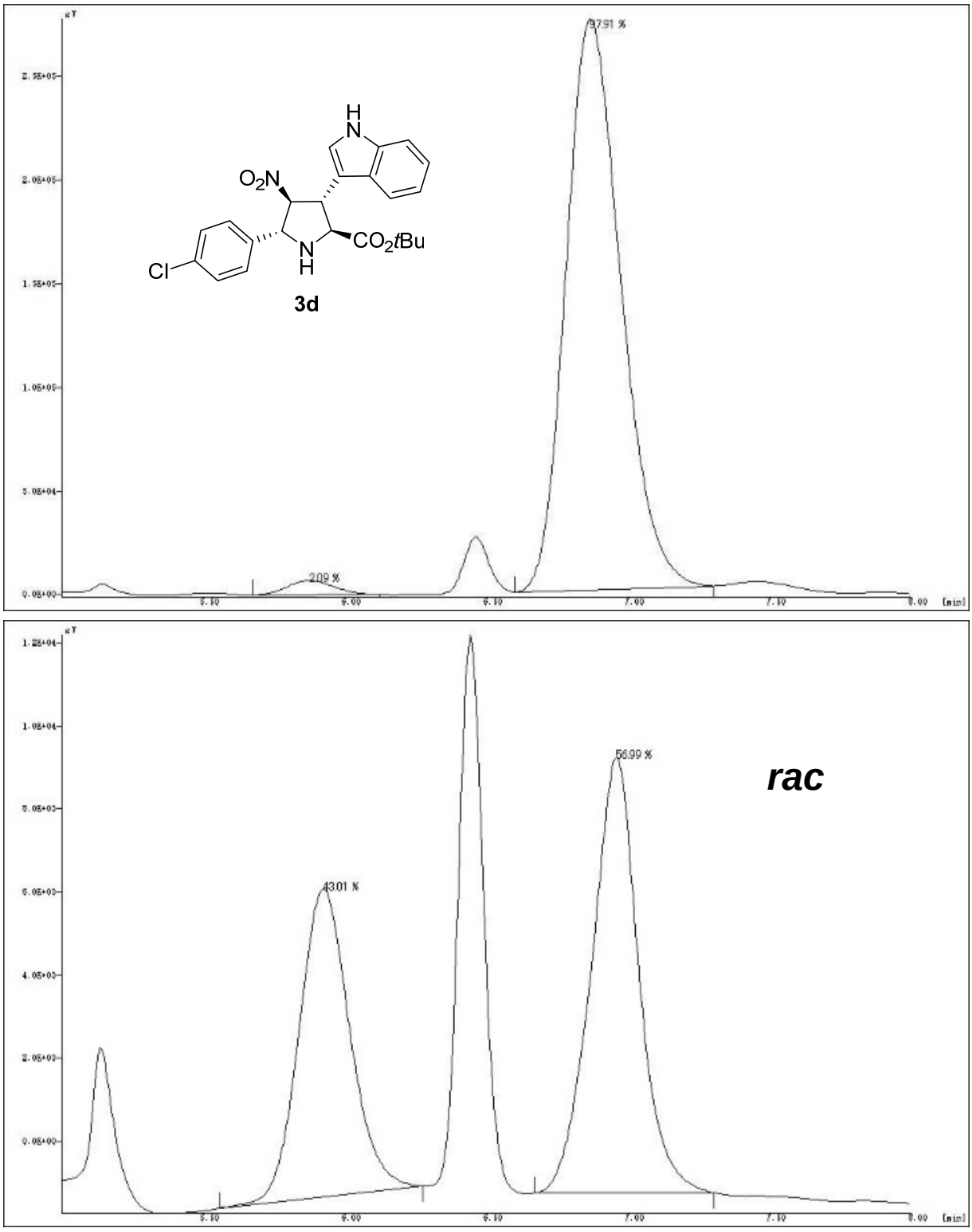


Chiralpack AD-H column (90:10 hexane: 2-propanol, 1.0 mL/min, 254 nm)

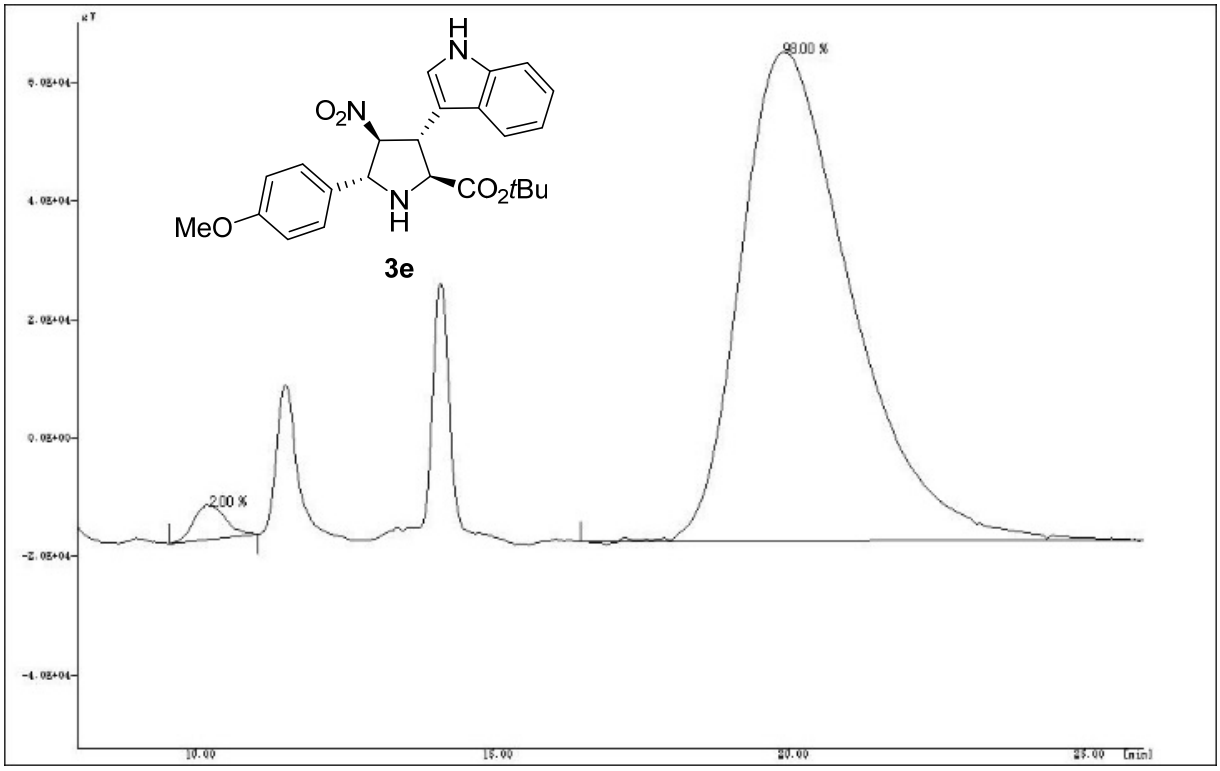


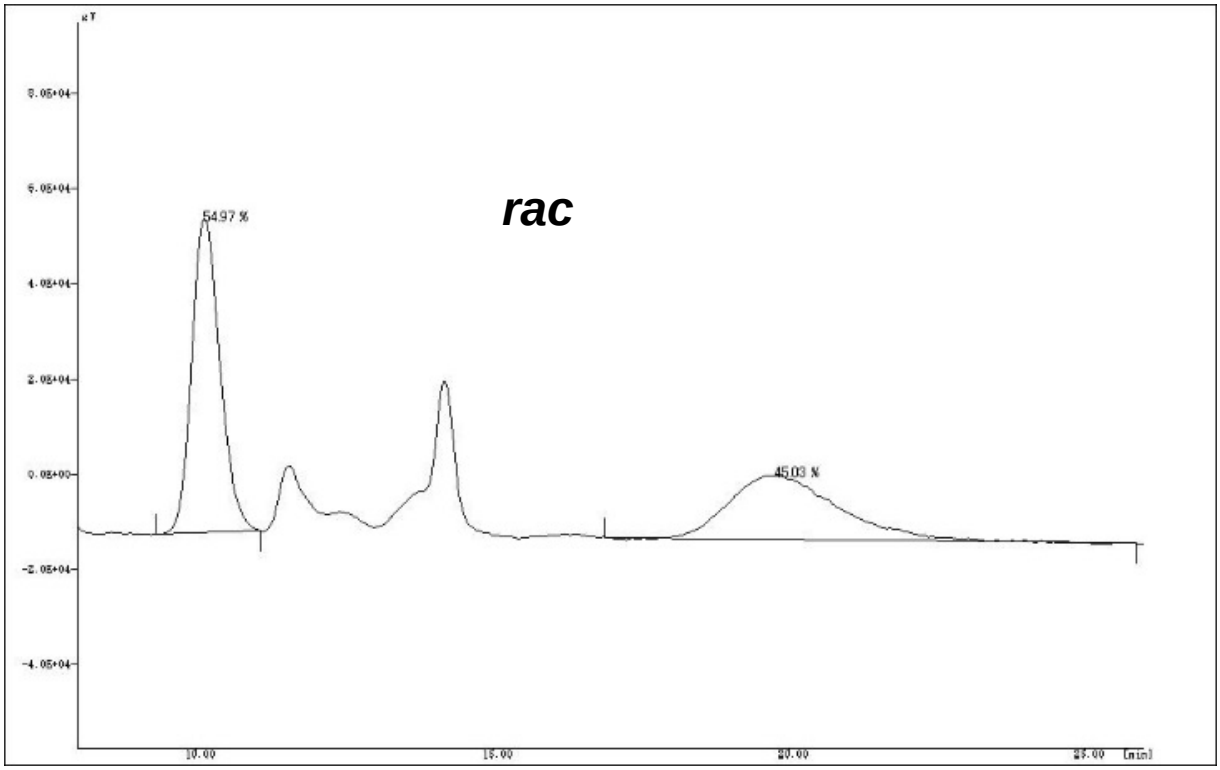


Chiralpack OD-H column (90:10 hexane: 2-propanol, 1.0 mL/min, 254 nm)

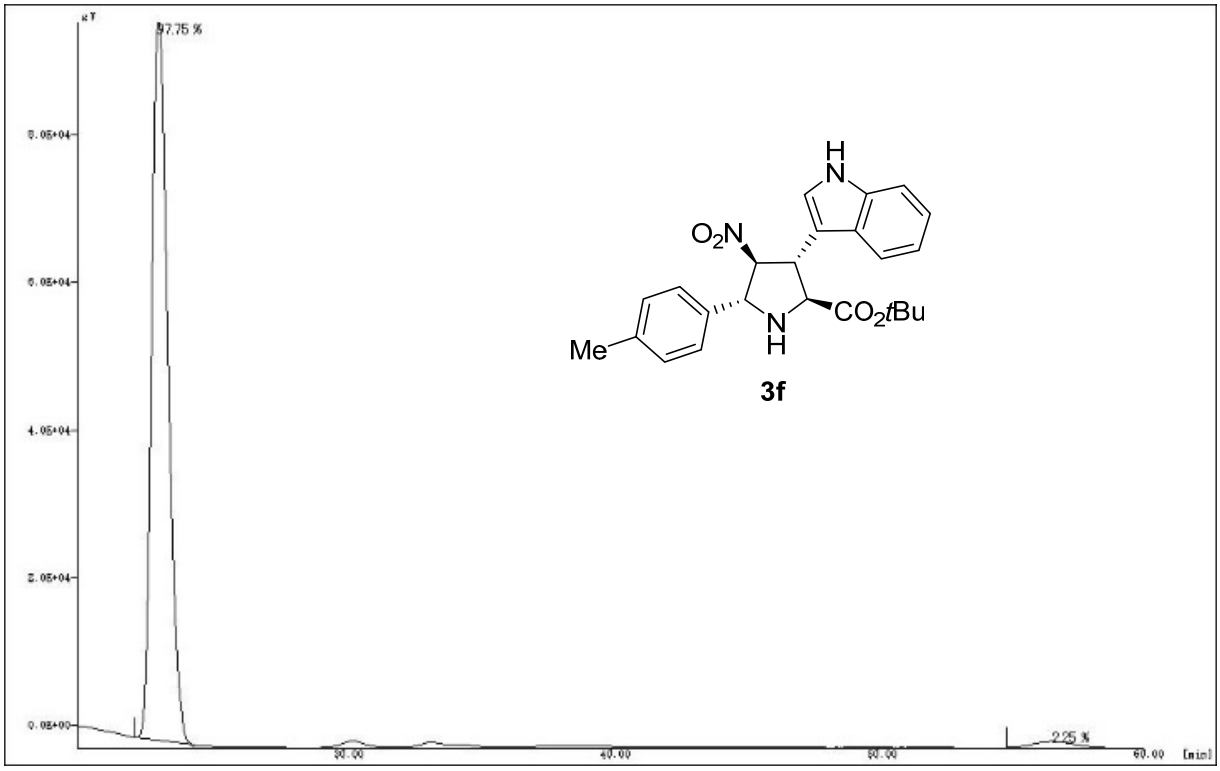


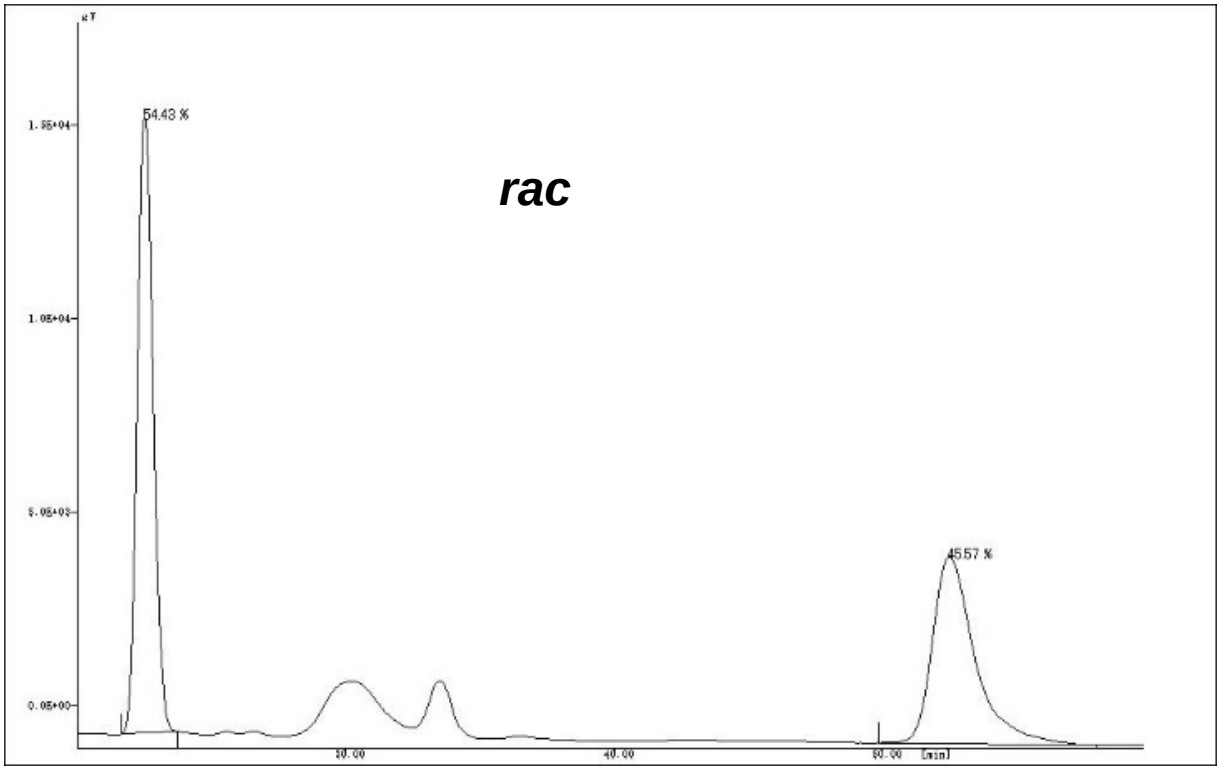
Chiralpack IC-3 column (90:10 hexane: 2-propanol, 1.0 mL/min, 254 nm)



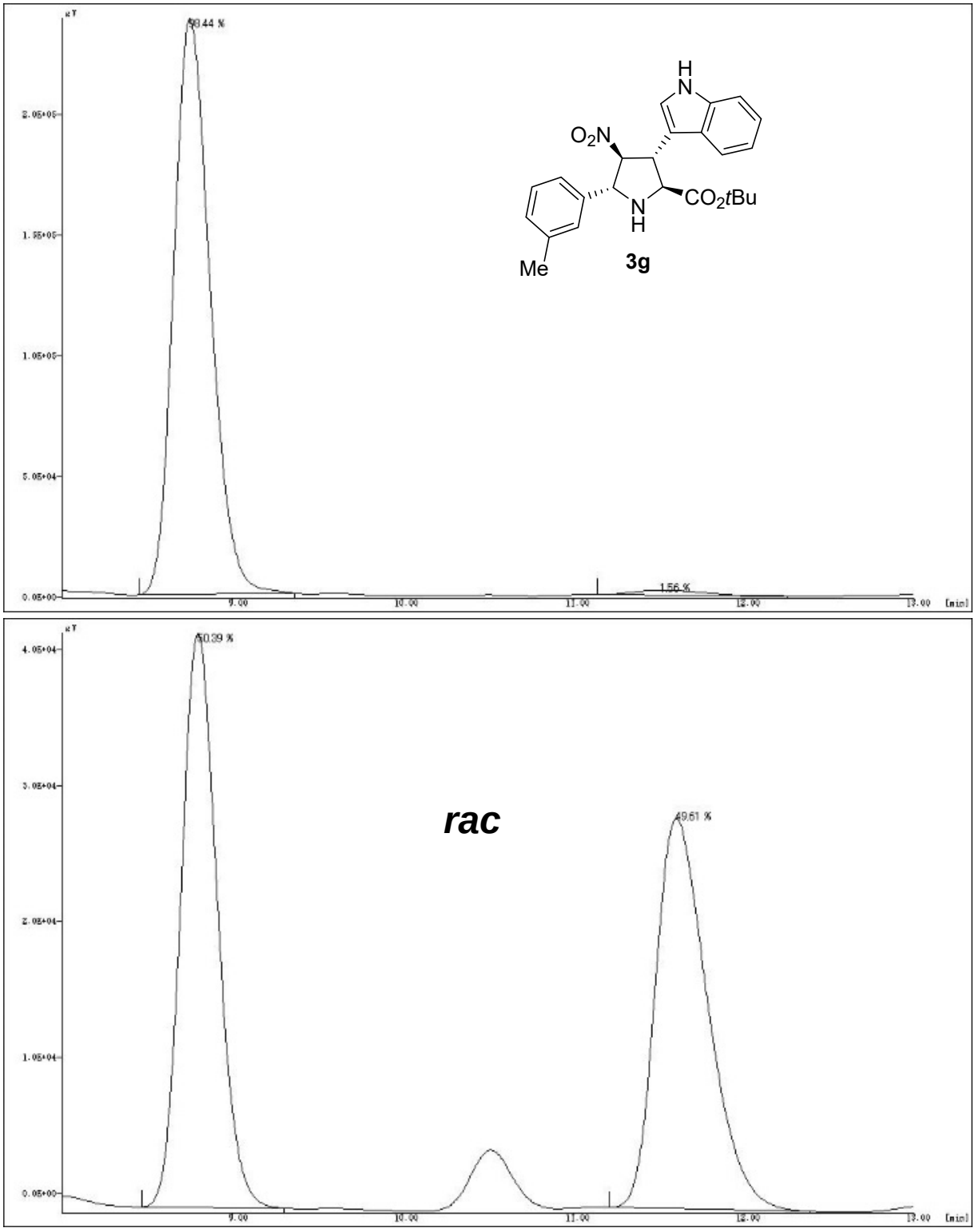


Chiralpack AS-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)

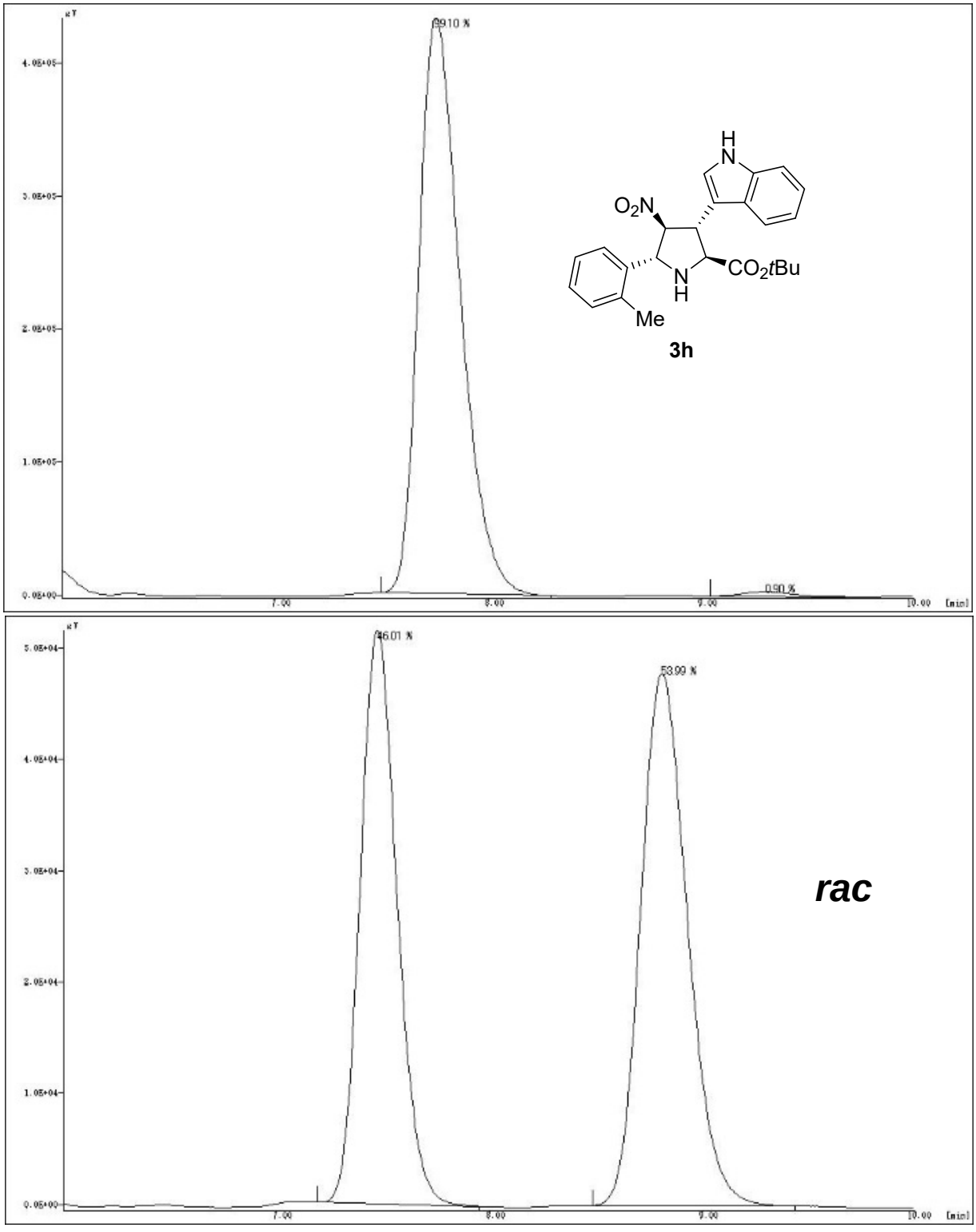




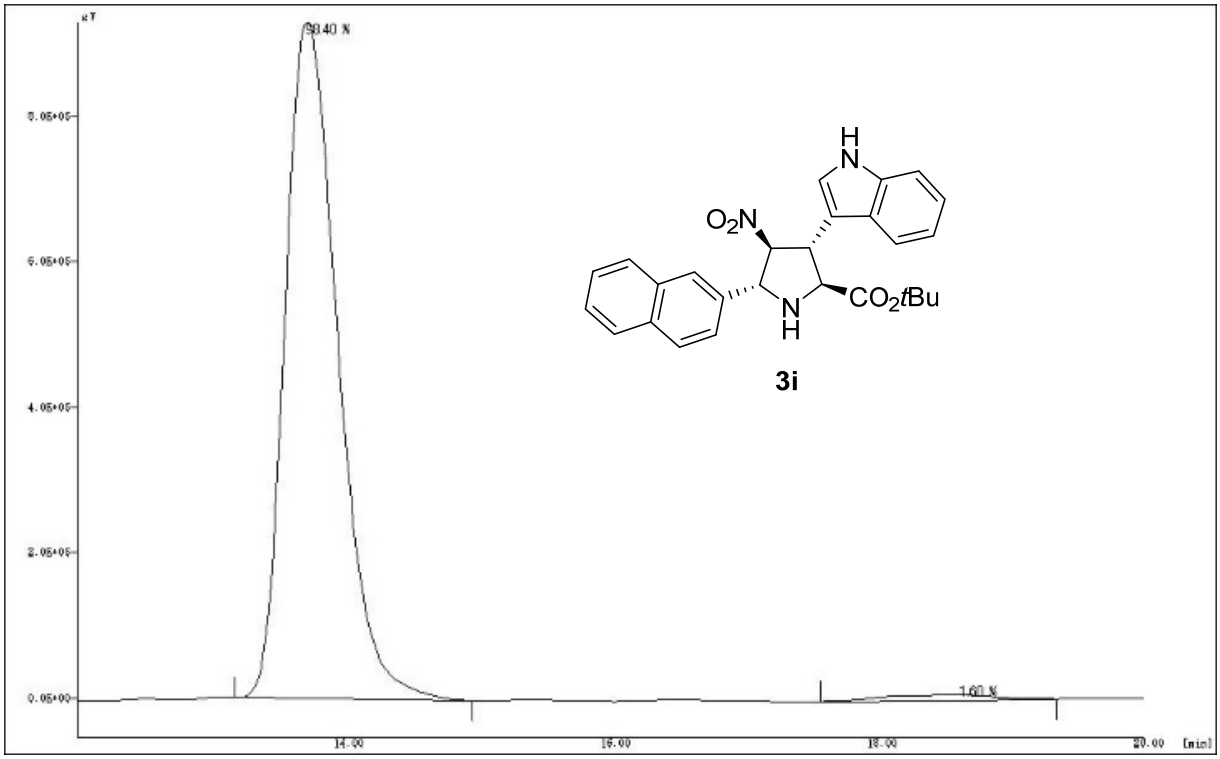
Chiralpack AD-H column (90:10 hexane: 2-propanol, 1.0 mL/min, 254 nm)



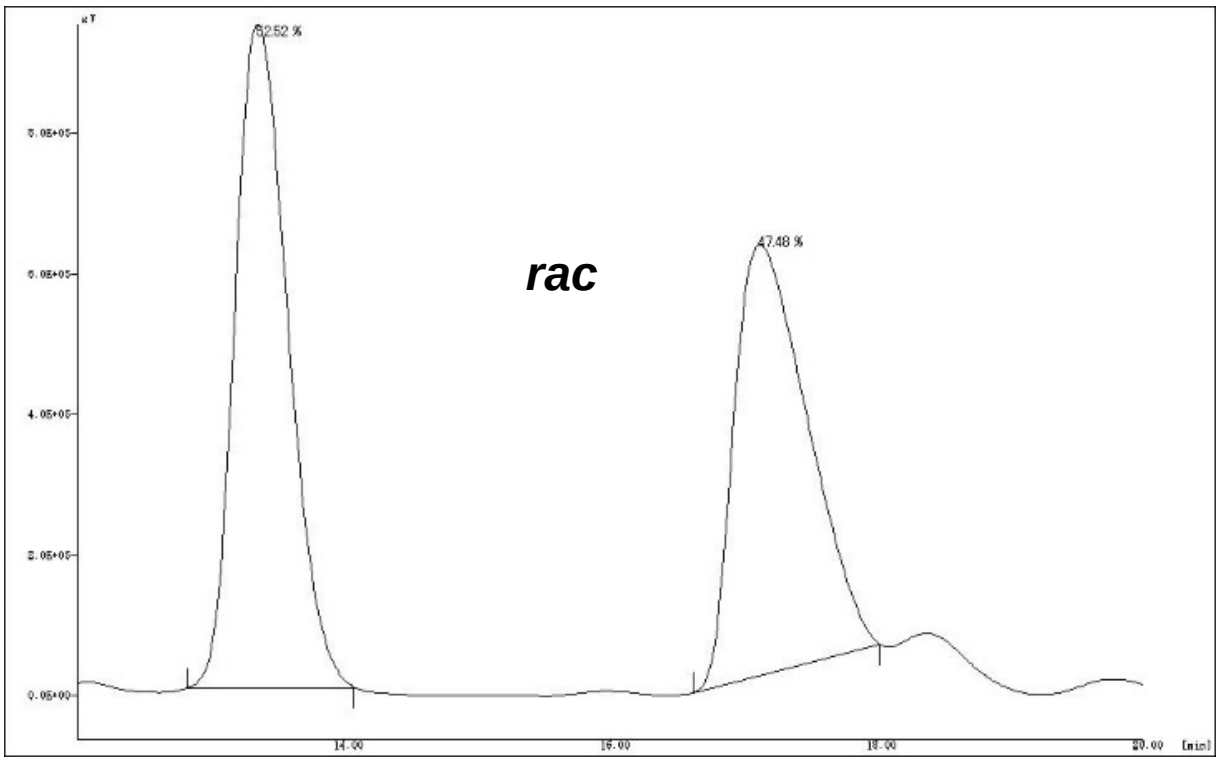
Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)



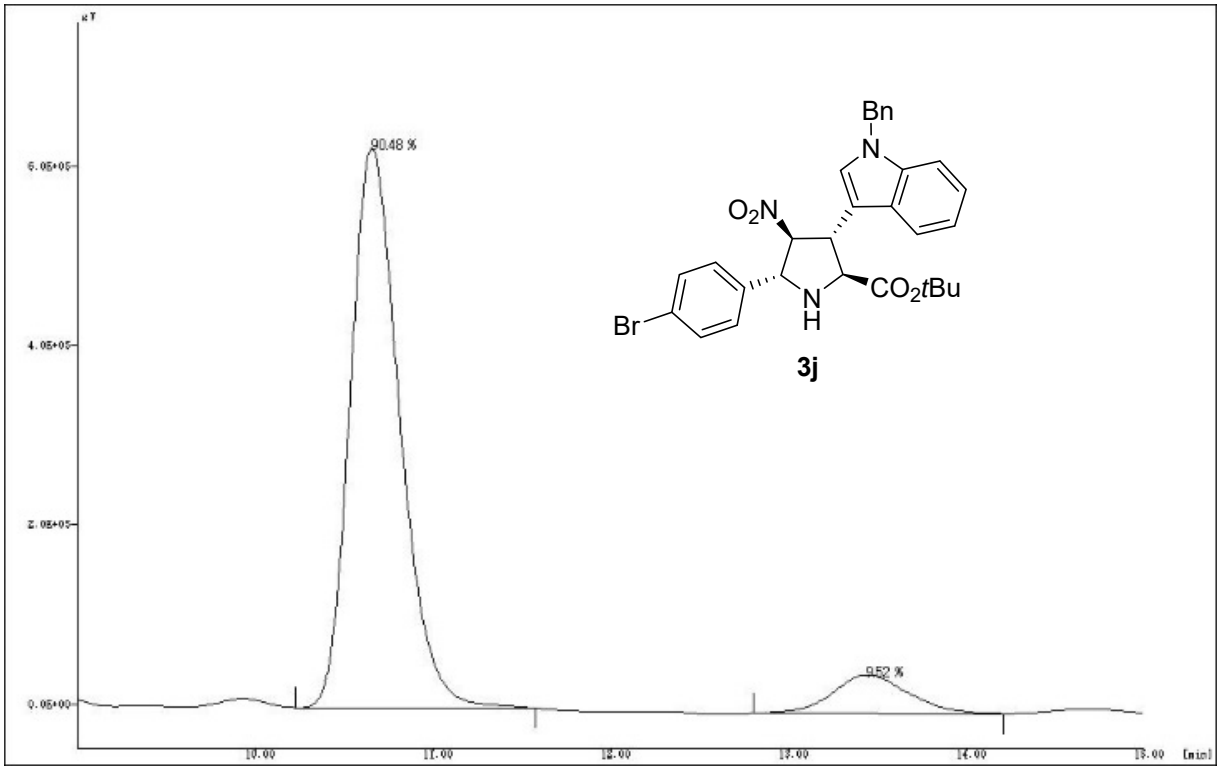
Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm）

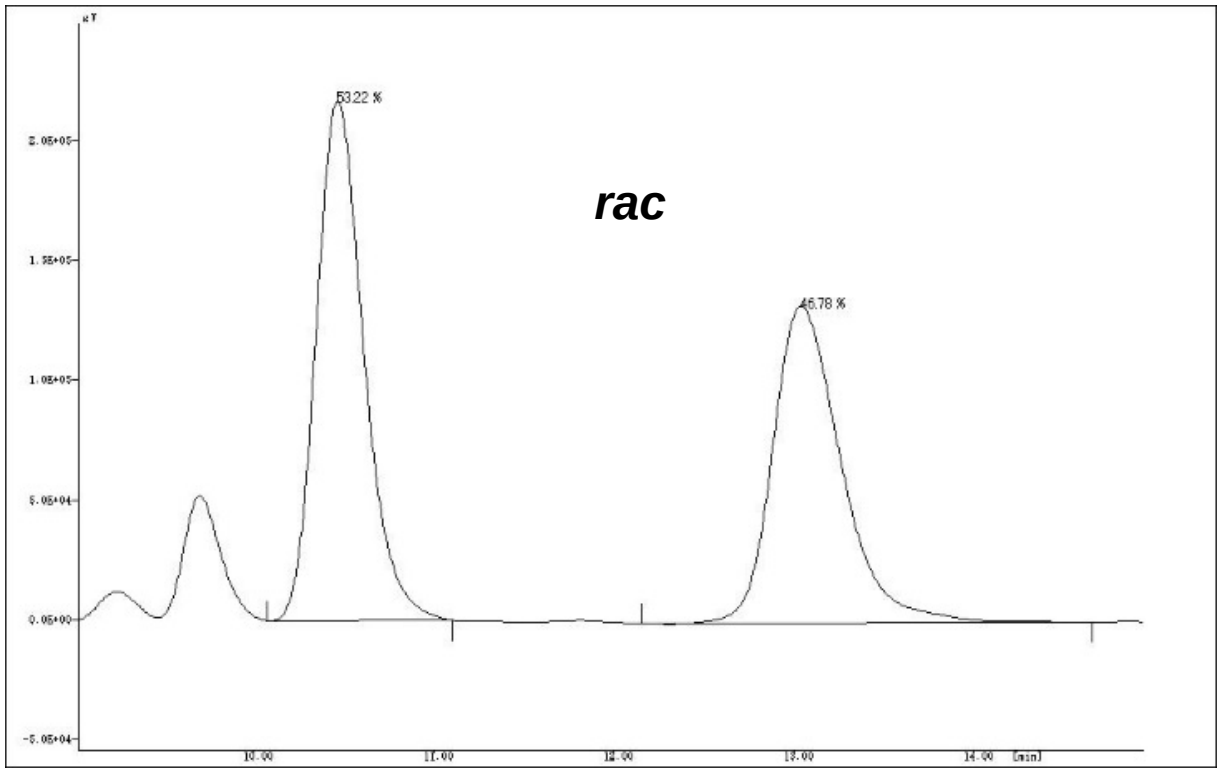


3i

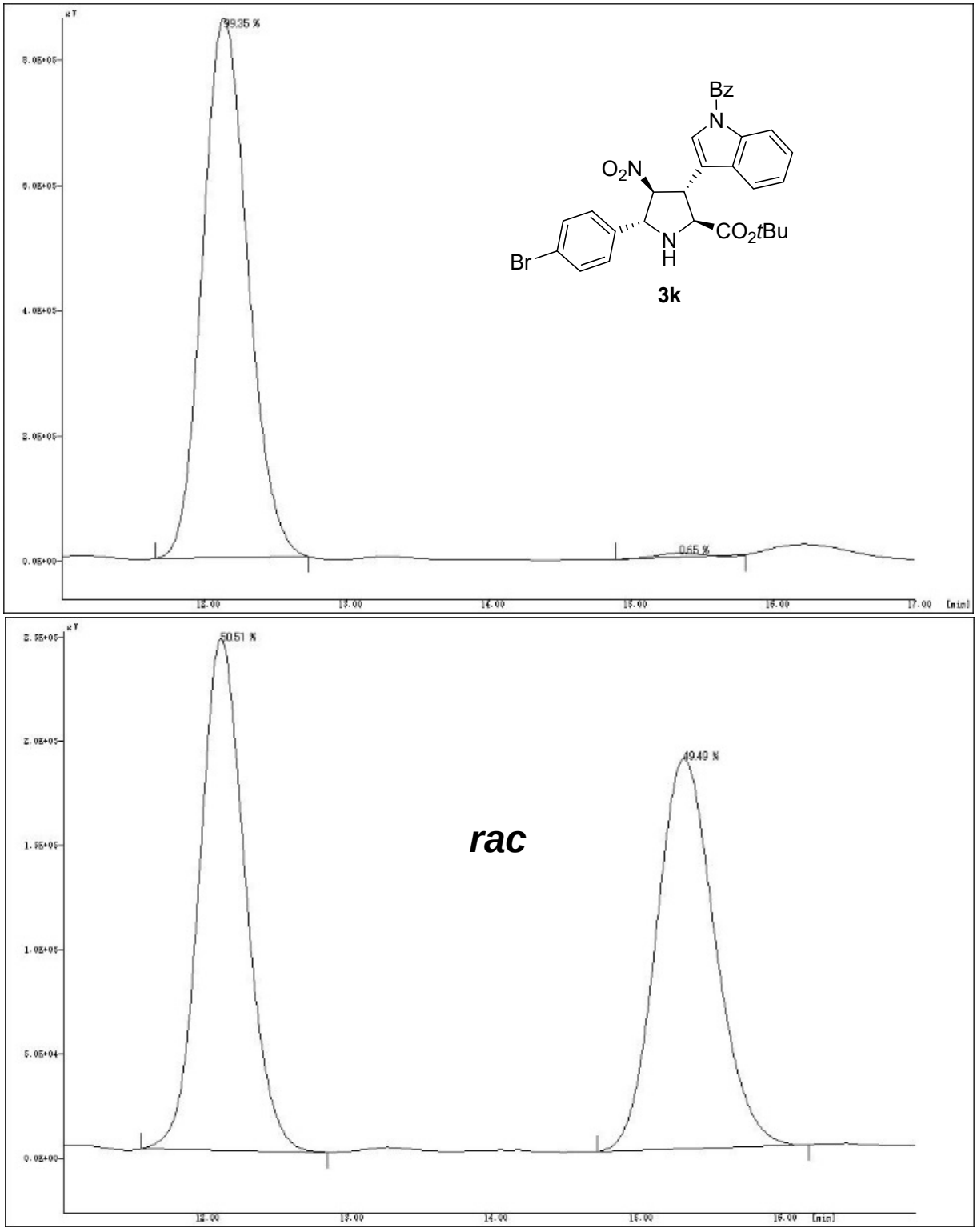


Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm）

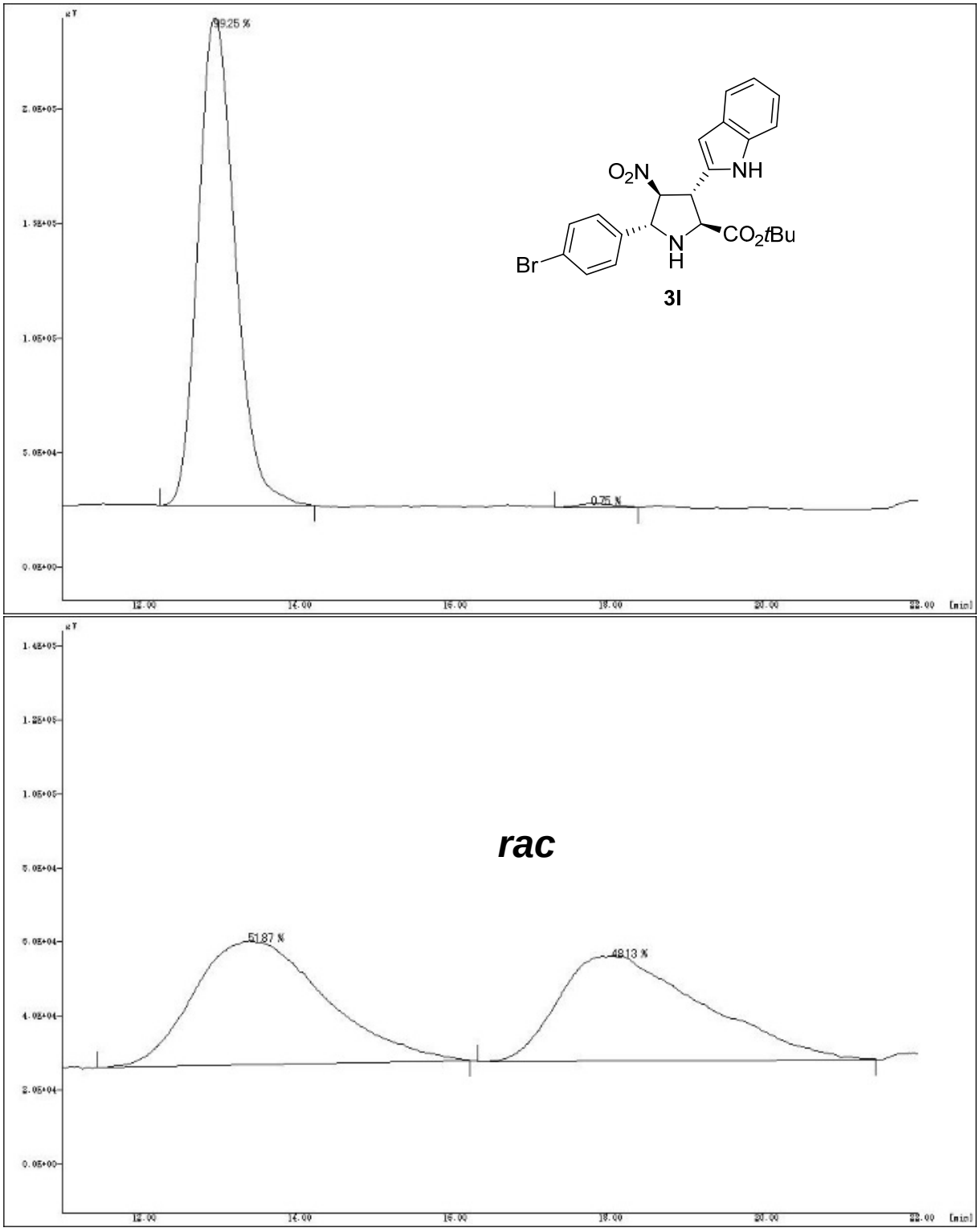




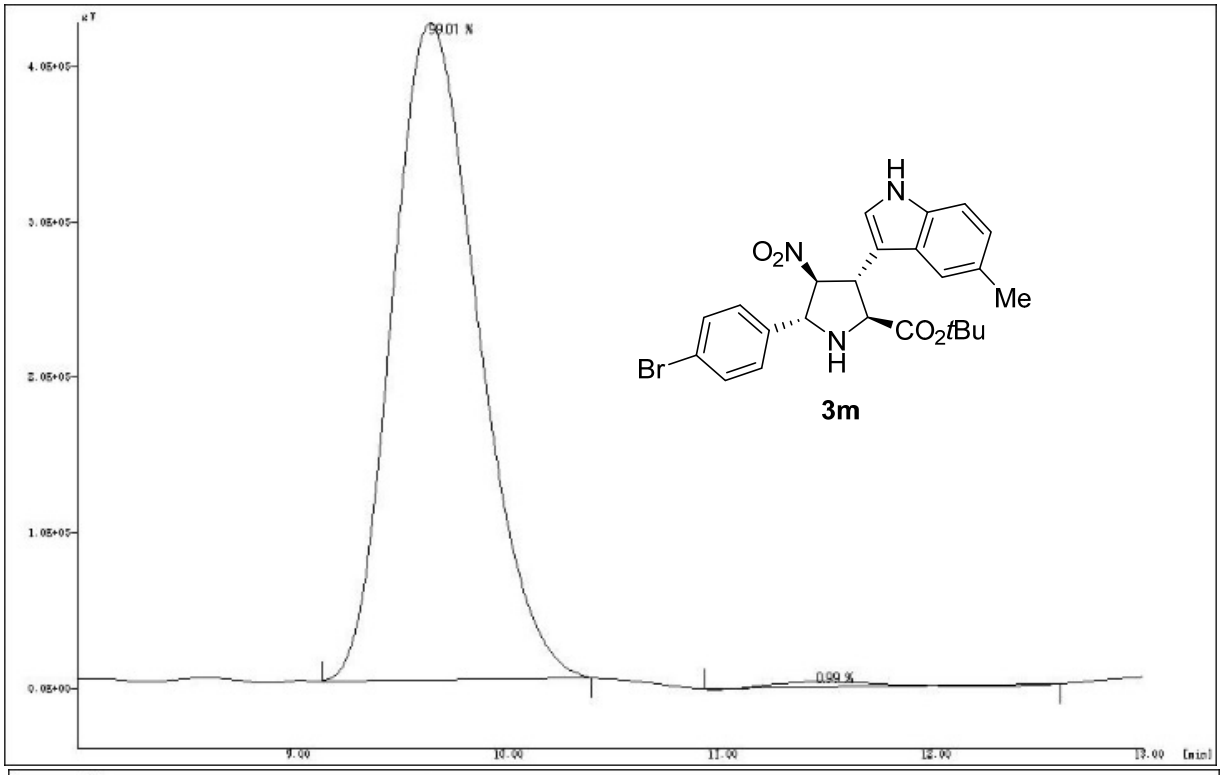
Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)

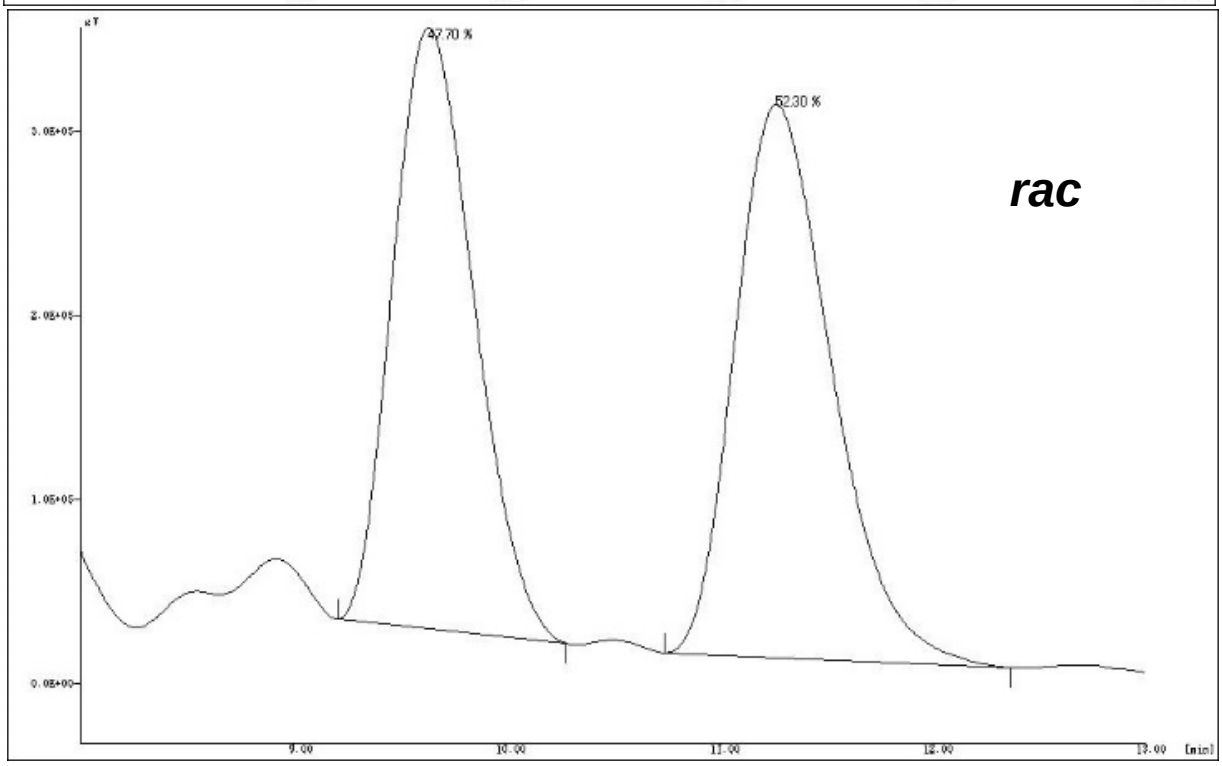


Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)

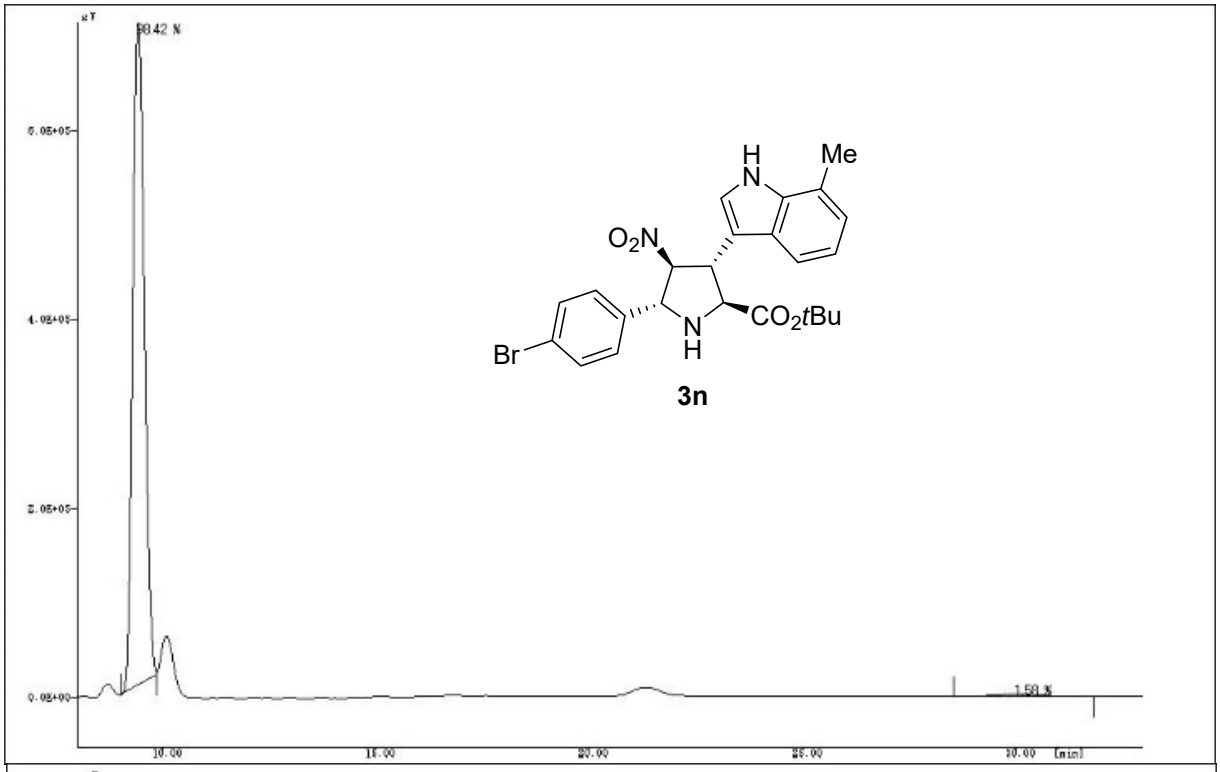


Chiralpack AD-H column (95:5 hexane: 2-propanol, 0.7 mL/min, 254 nm)

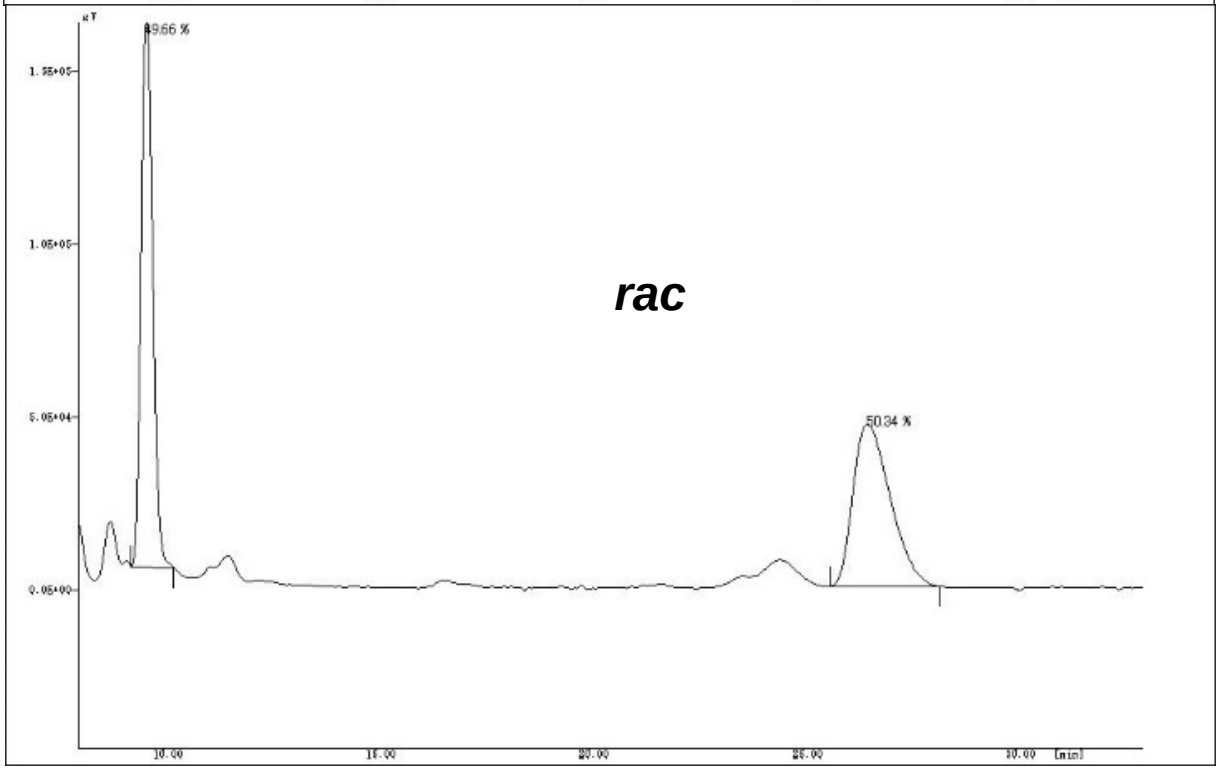




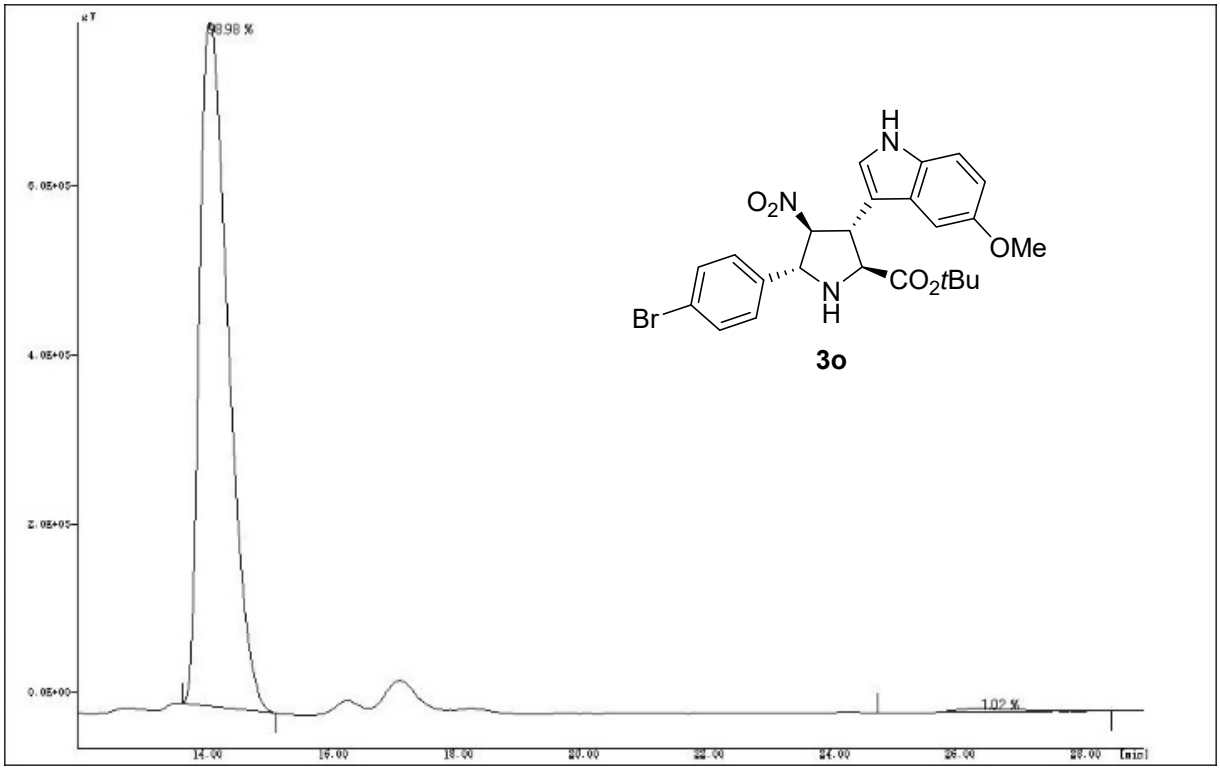
Chiralpack OD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm）

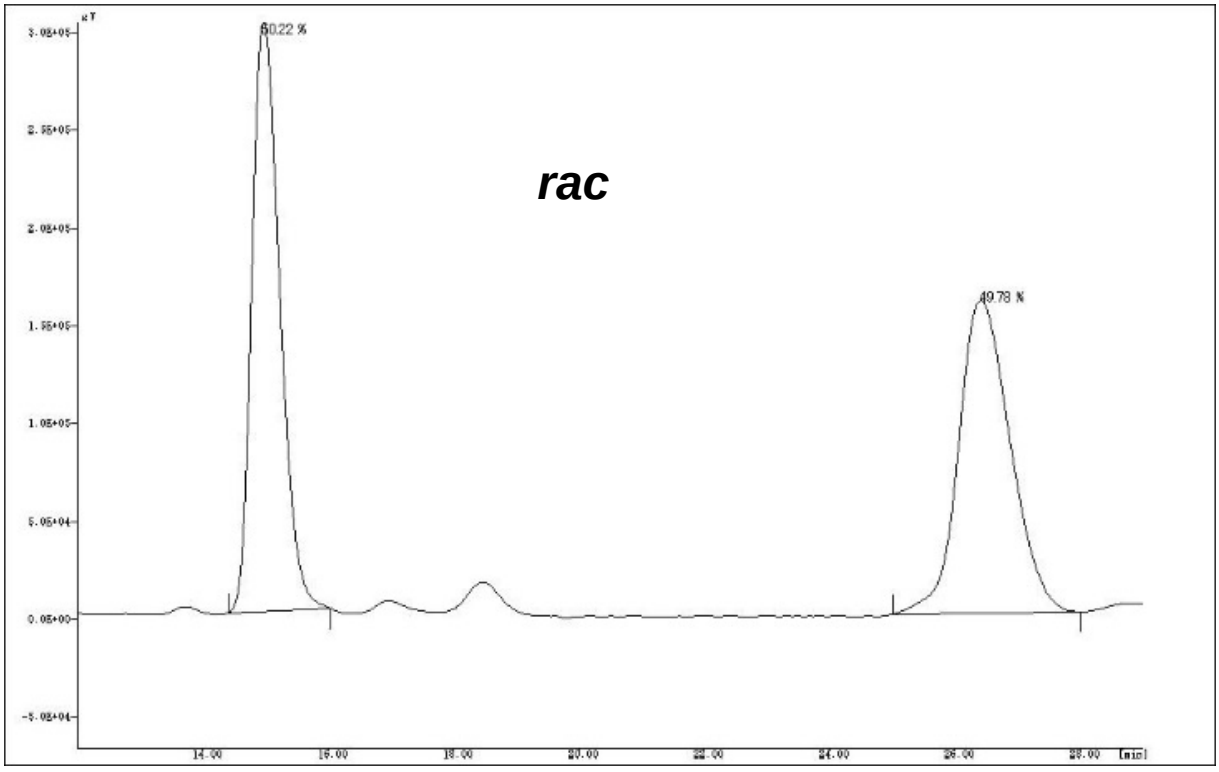


3n

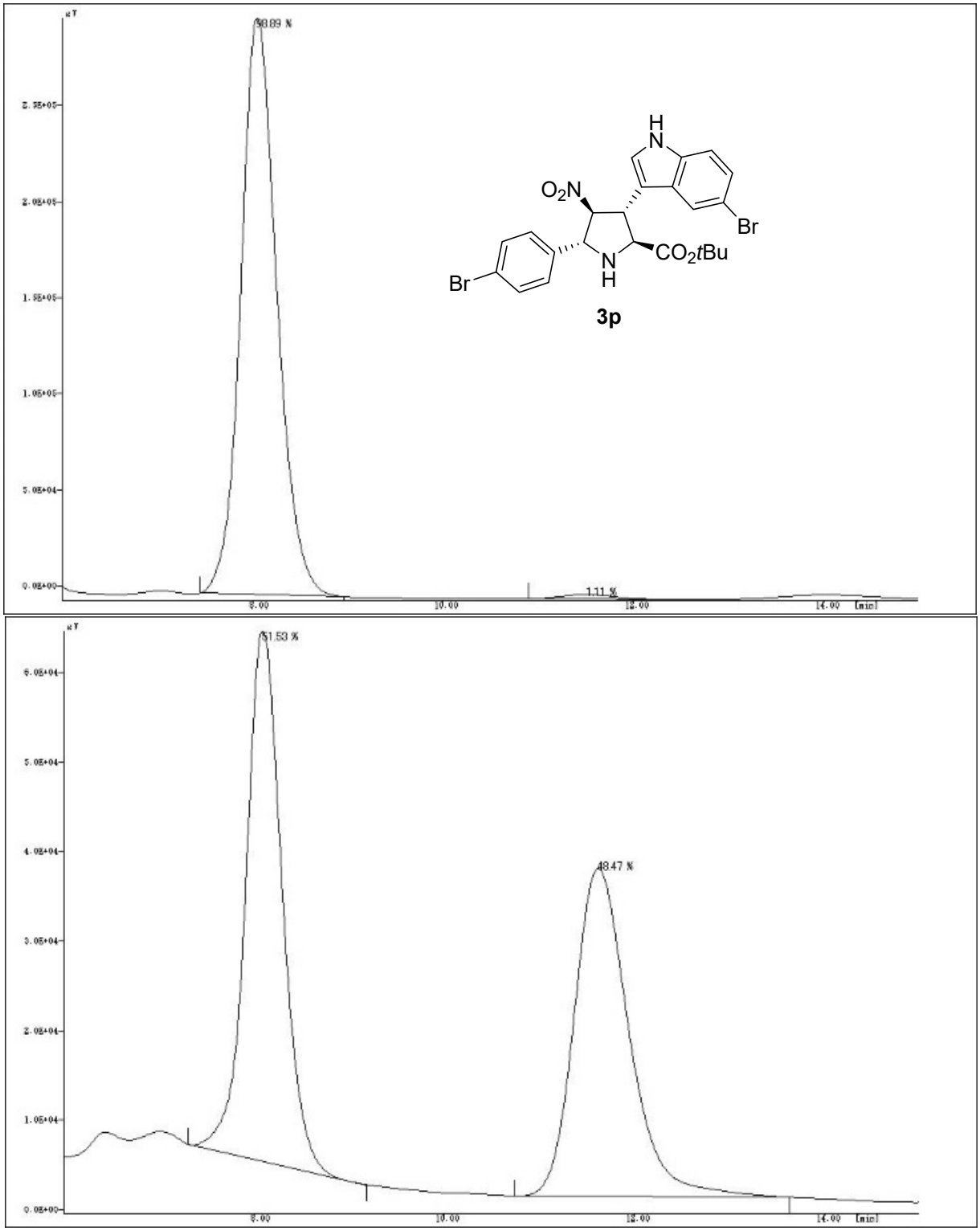


Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm）

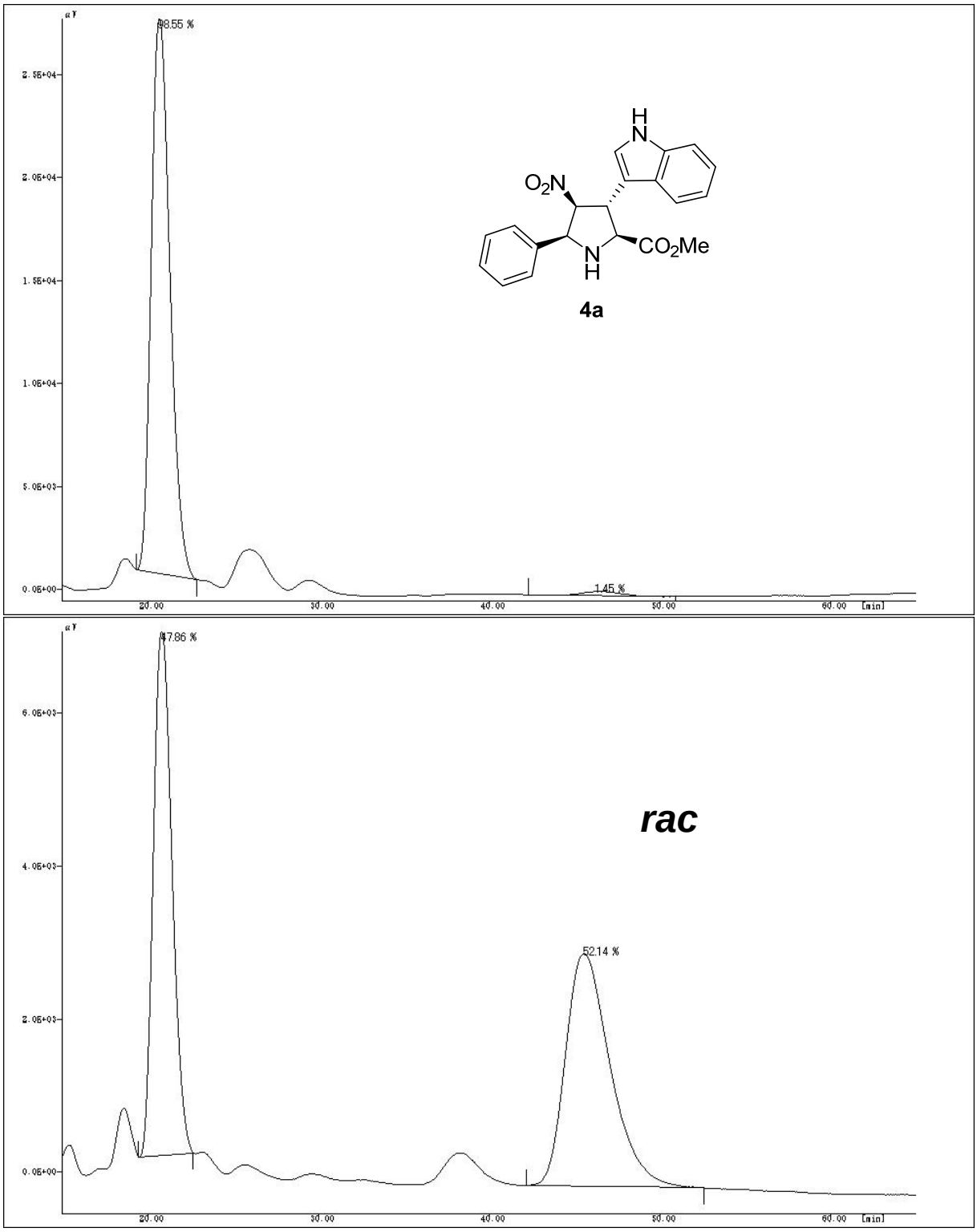




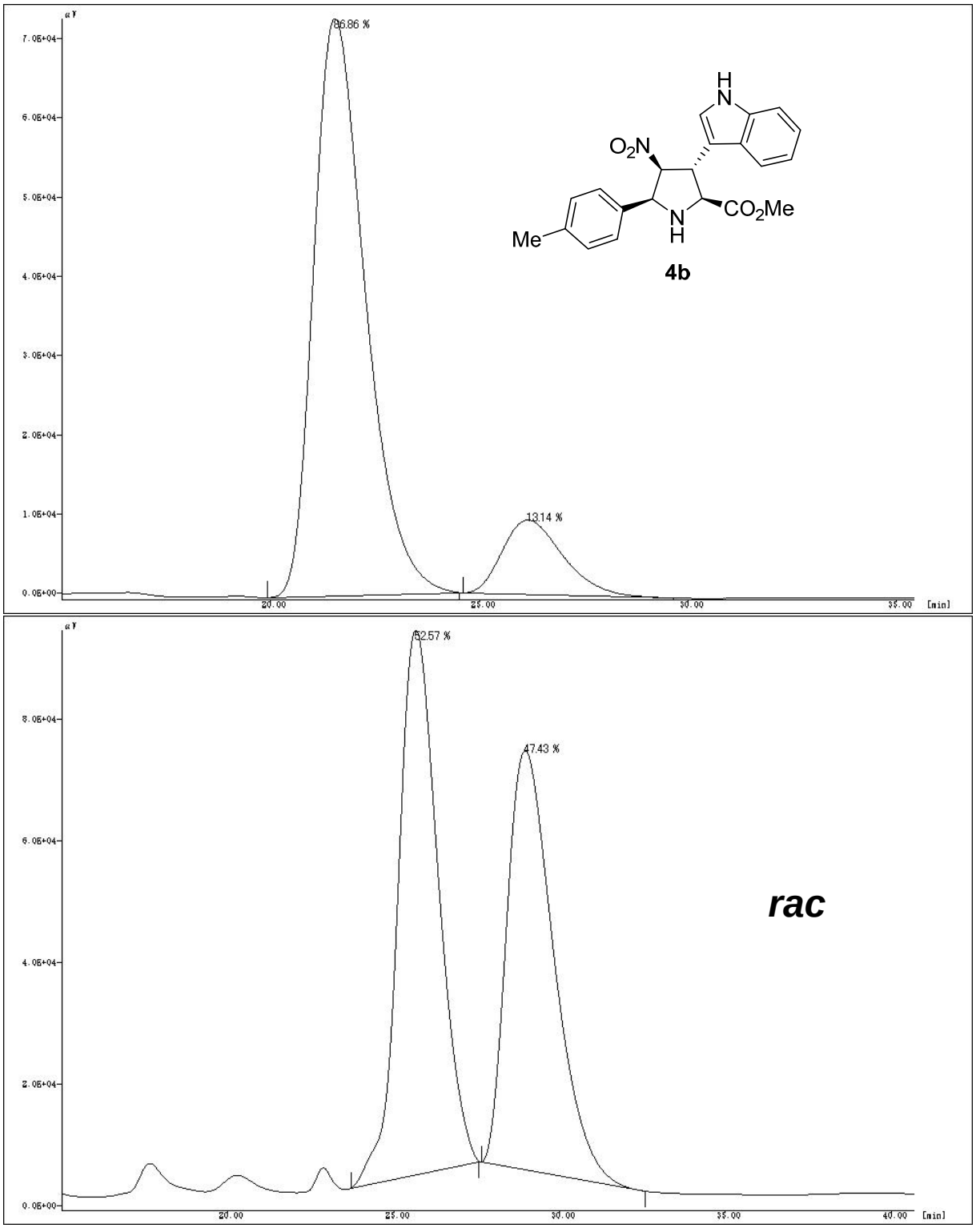
Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)



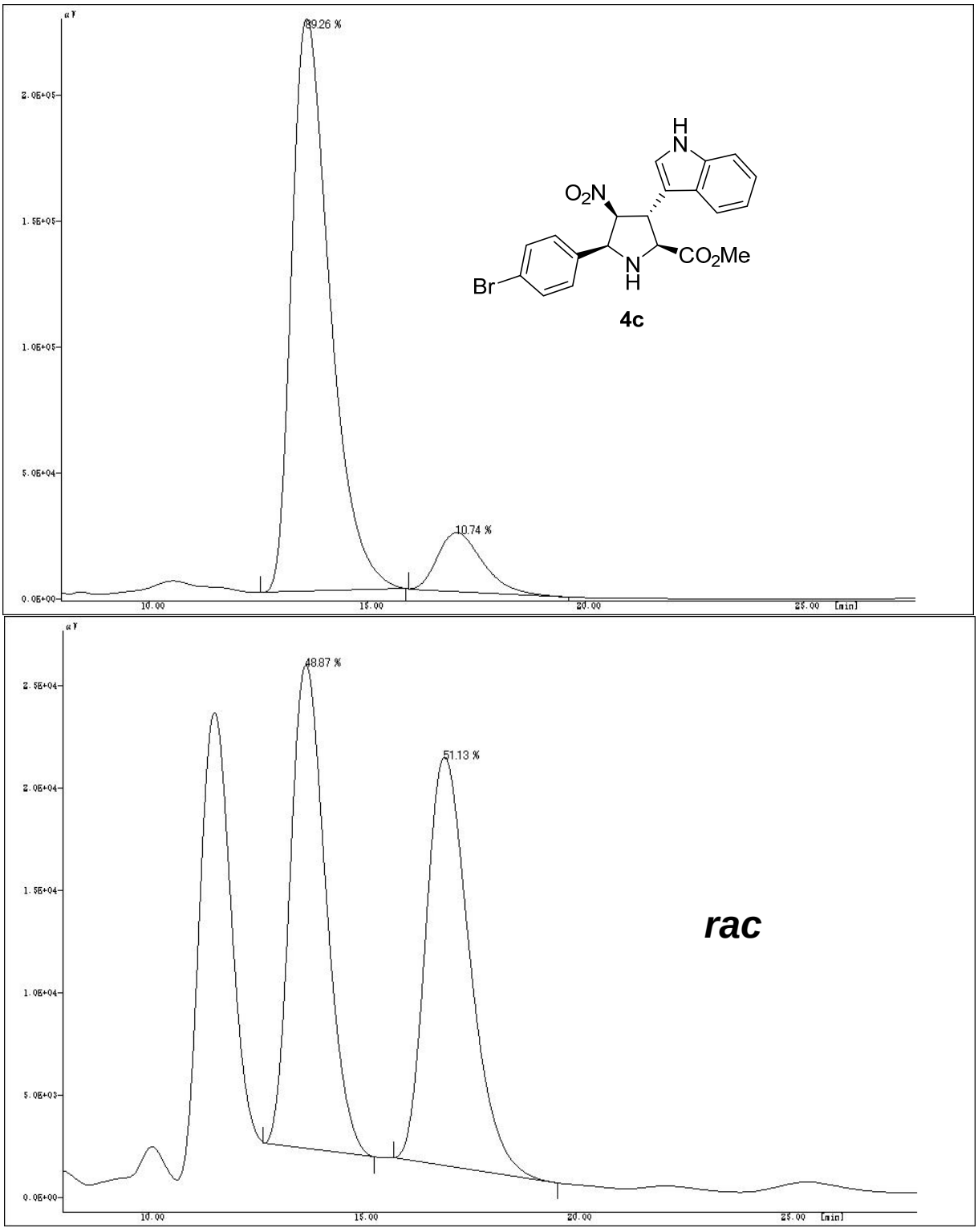
Chiralpack OD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)



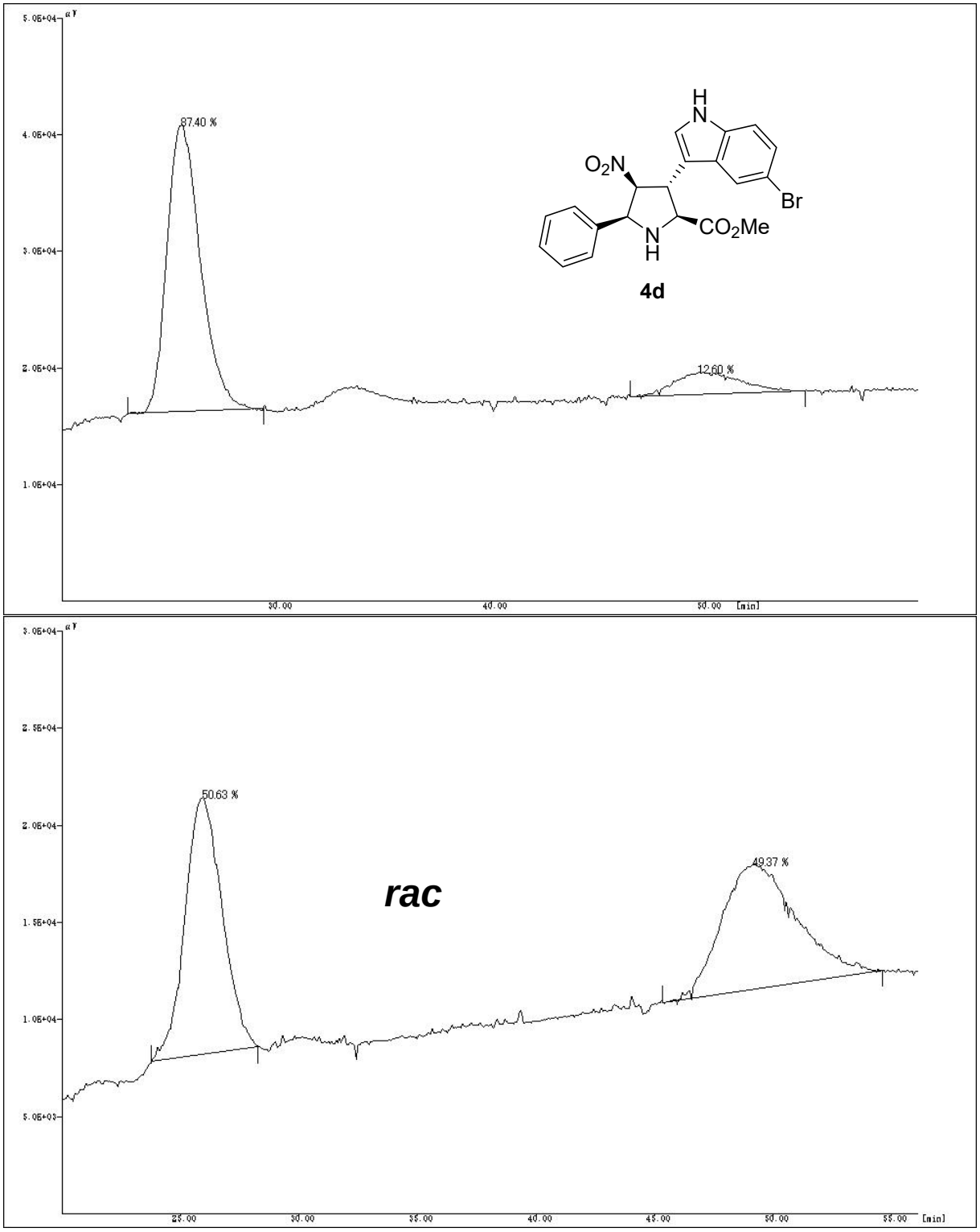
Chiralpack OD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)



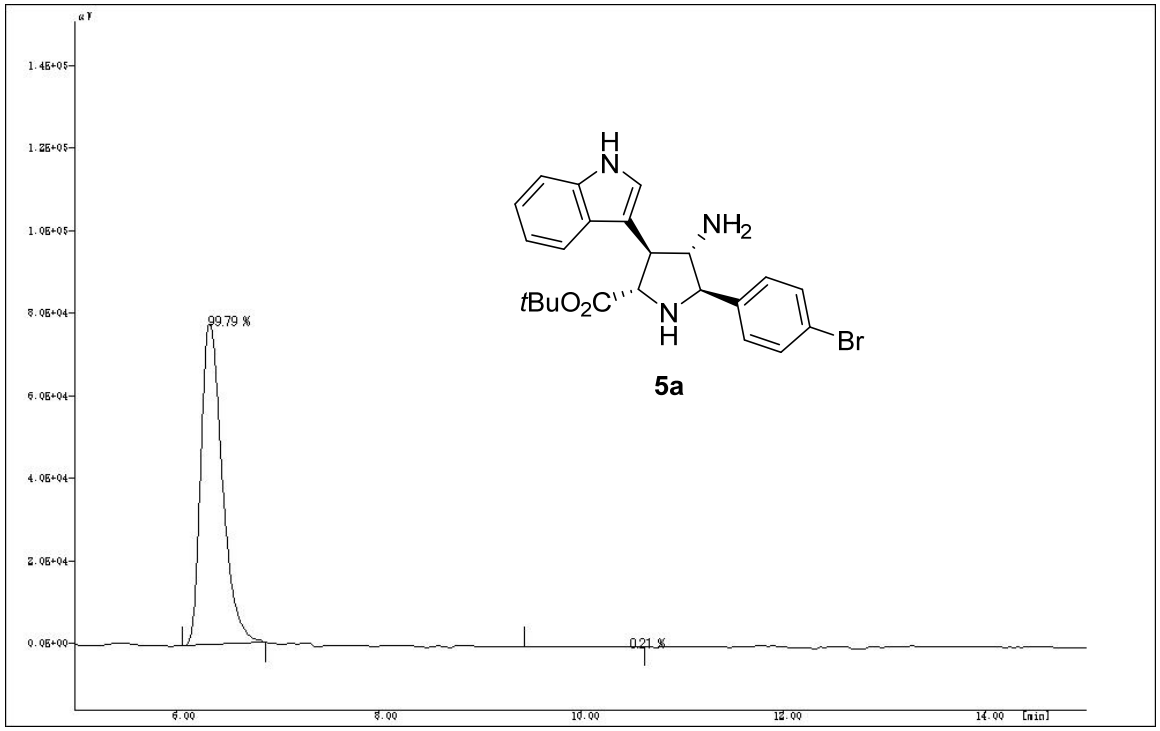
Chiralpack OD-H column (70:30 hexane: 2-propanol, 0.7 mL/min, 254 nm)

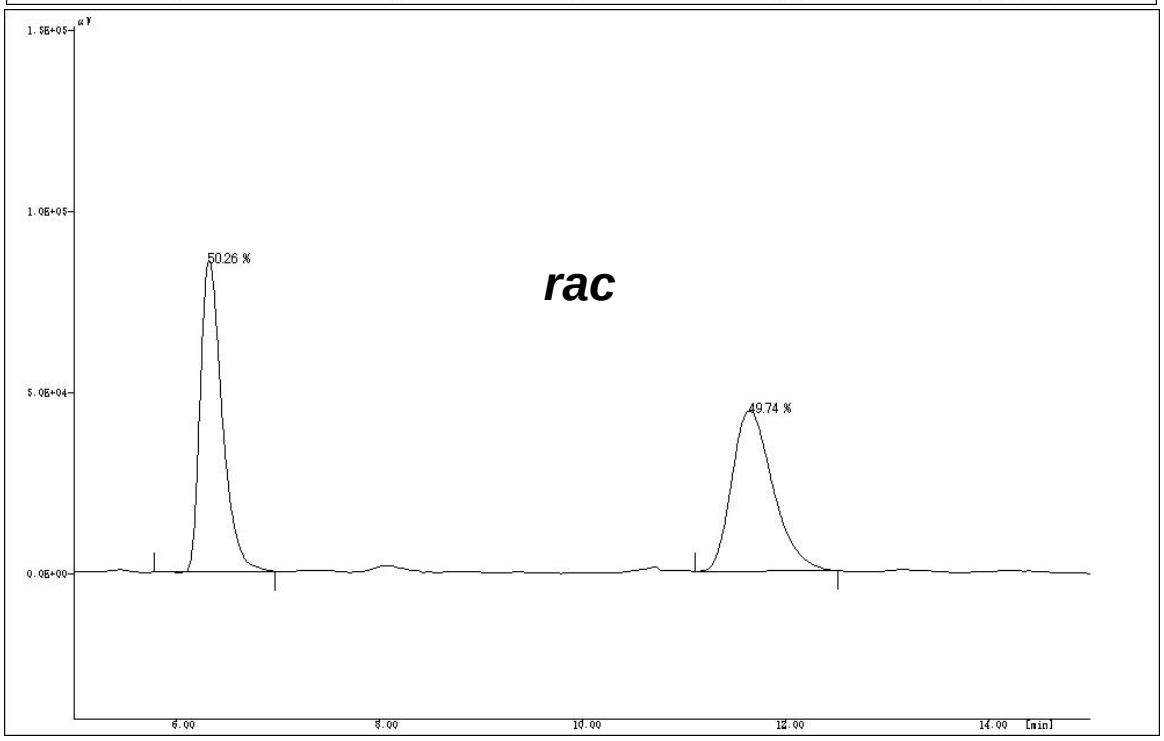


Chiralpack OD-H column (70:30 hexane: 2-propanol, 1.0 mL/min, 254 nm)

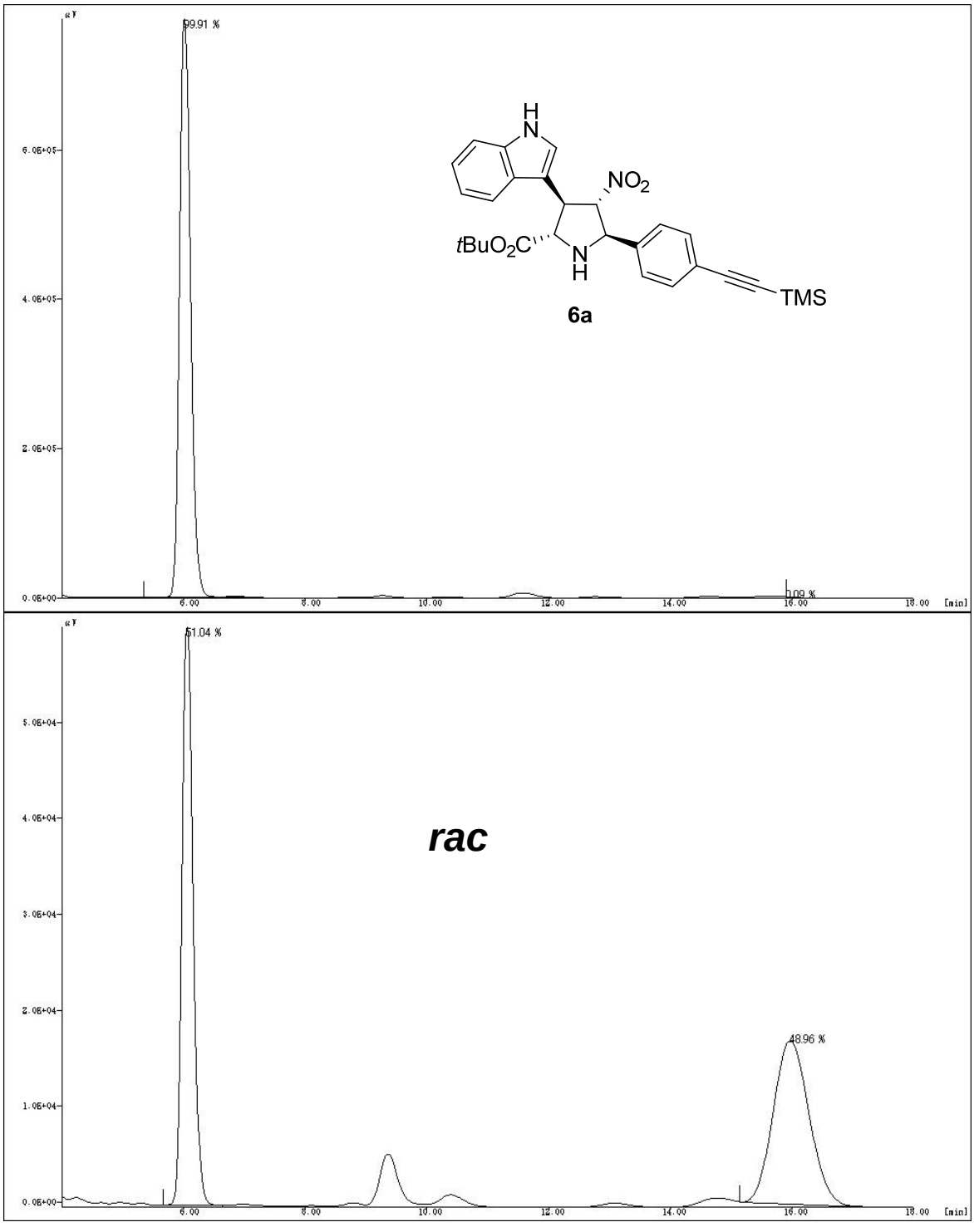


Chiralpack OD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)





Chiralpack AD-H column (70:30 hexane: 2-propanol, 1.0 mL/min, 254 nm)



Chiralpack AD-H column (80:20 hexane: 2-propanol, 1.0 mL/min, 254 nm)