Supporting information II:

Metallacarborane sulfamides: unconventional, specific, and highly selective inhibitors of carbonic anhydrase IX

Bohumír Grüner, Jiří Brynda, Viswanath Das, Václav Šícha, Jana Štěpánková, Jan Nekvinda, Josef Holub, Klára Pospíšilová, Milan Fábry, Petr Pachl, Vlastimil Král, Michael Kugler, Vlastimil Mašek, Martina Medvedíková, Stanislava Matějková, Alice Nová, Barbora Lišková, Soňa Gurská, Petr Džubák, Marián Hajdúch, Pavlína Řezáčová

Contents

HPLC purity traces of compounds **1**$^-$ to **9**$^-$ and ^{11}B, ^{1}H and ^{13}C NMR spectra of compounds **6**$^-$ and **8**$^-$ selected for detailed biological studies.

D-7000 HPLC System Manager Report

Compound 1-

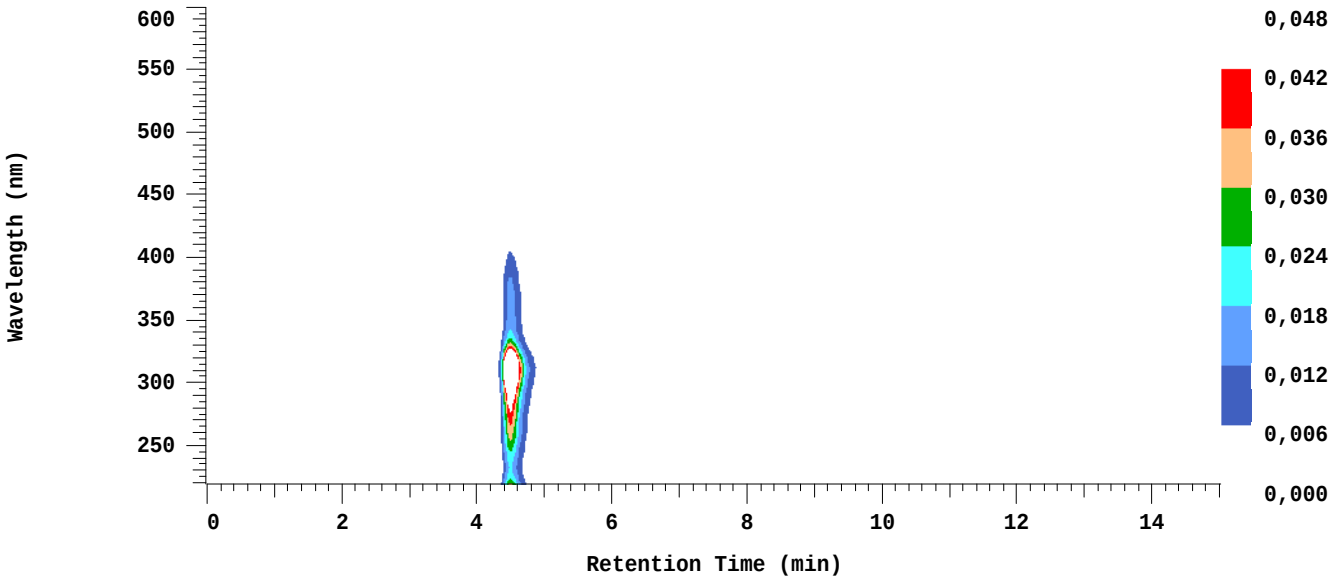
Data Path: C:\WIN32APP\HSM\Samples\DATA\0356 CB17
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB17
Injection from this vial: 1 of 1
Sample Description:

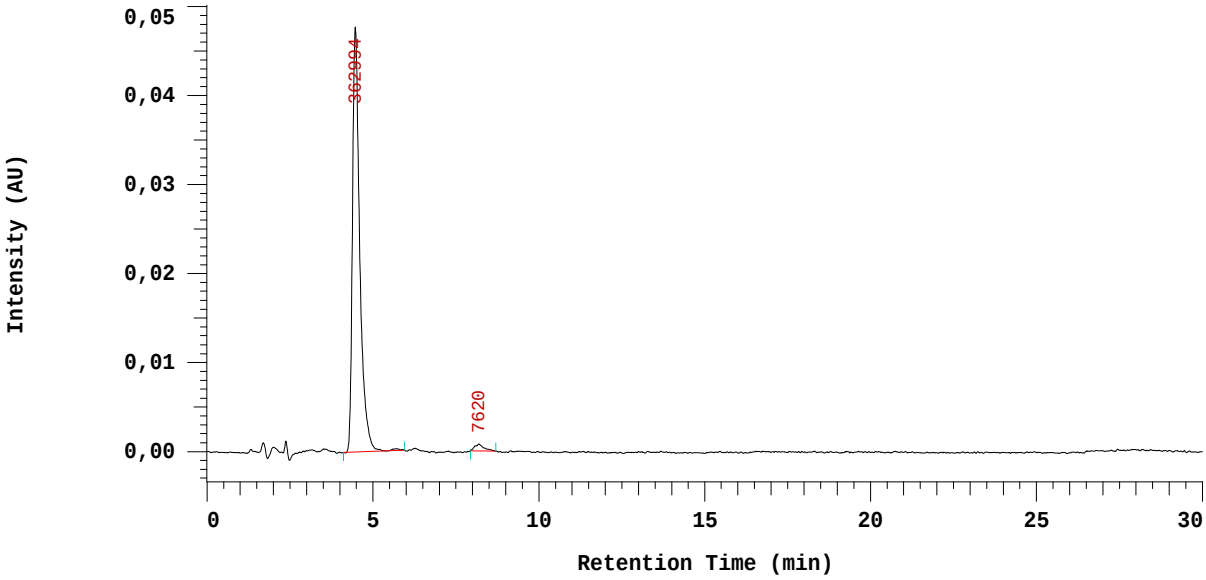
Series:0356 CB17
Vial Number: 5
Vial Type: UNK
Volume: 3,0 ul

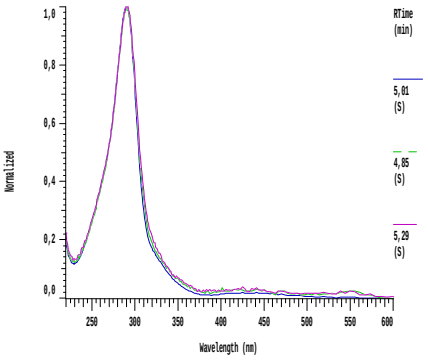
Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.048
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm





Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	4,29	51648	4,069		3285	0,9915	
2	5,01	1217673	95,931		59598	0,9997	
		1269321	100,000		62883		

Peak rejection level: 1000

D-7000 HPLC System Manager Report

Compound 2-

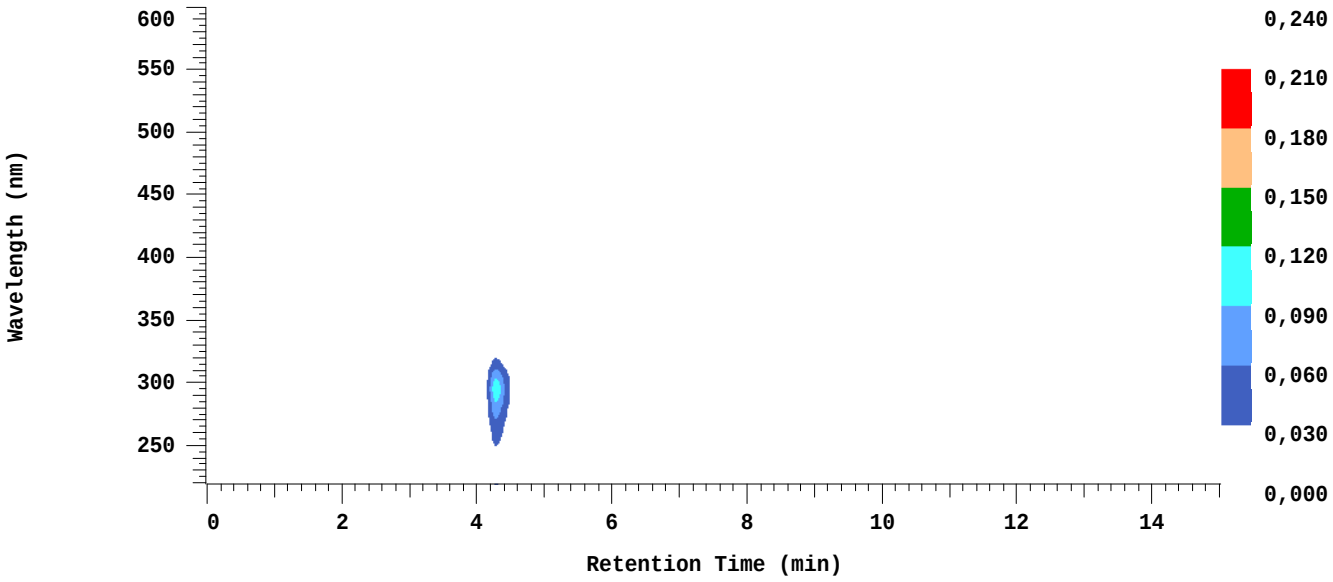
Data Path: C:\WIN32APP\HSM\Samples\DATA\0984 CB16
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB16
Injection from this vial: 1 of 1
Sample Description:

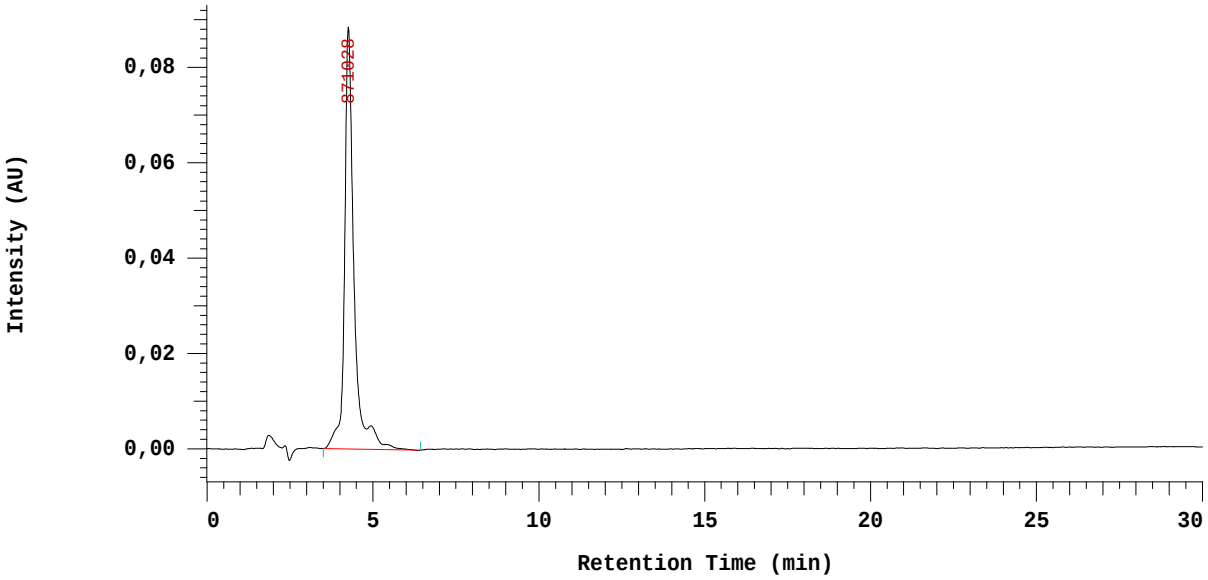
Series:0984 CB16
Vial Number: 13
Vial Type: UNK
Volume: 6,0 ul

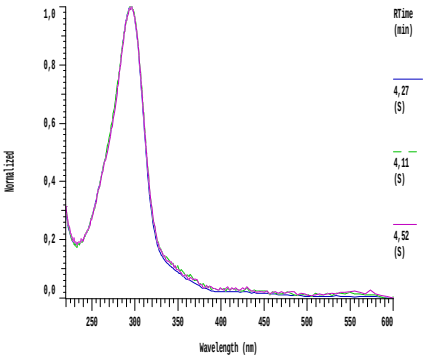
Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.240
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm





Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	4,27	871028	100,000		44222	0,9998	
		871028	100,000		44222		

Peak rejection level: 1000

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Compound 3-

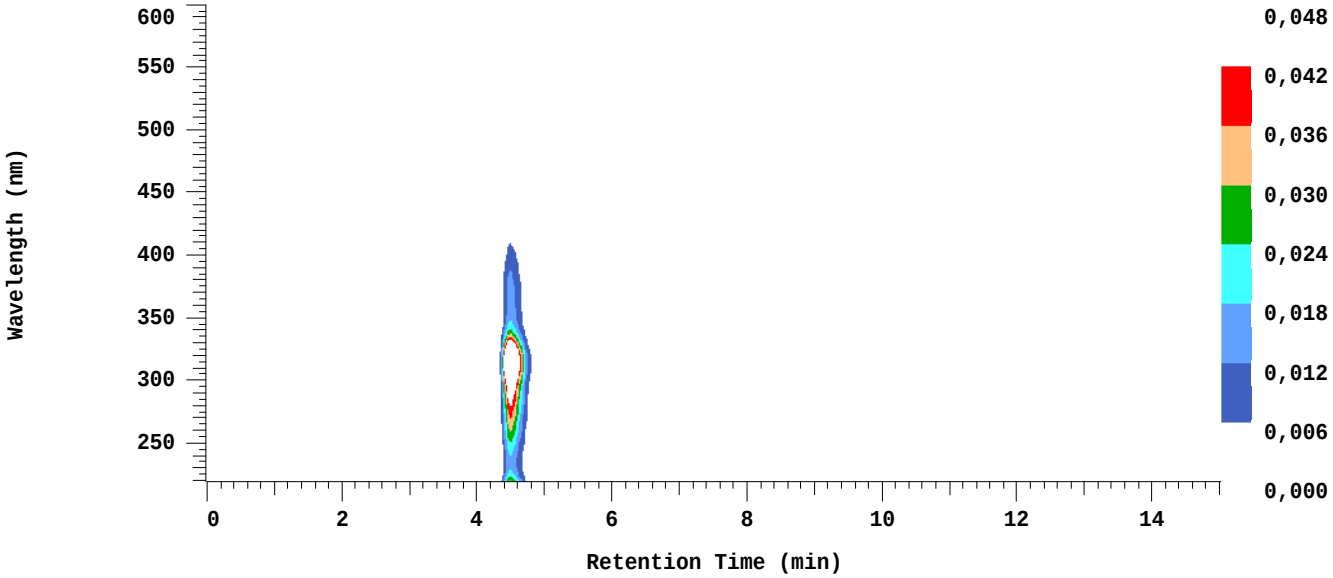
Data Path: C:\WIN32APP\HSM\Samples\DATA\0339 CB4
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB-4
Injection from this vial: 1 of 1
Sample Description:

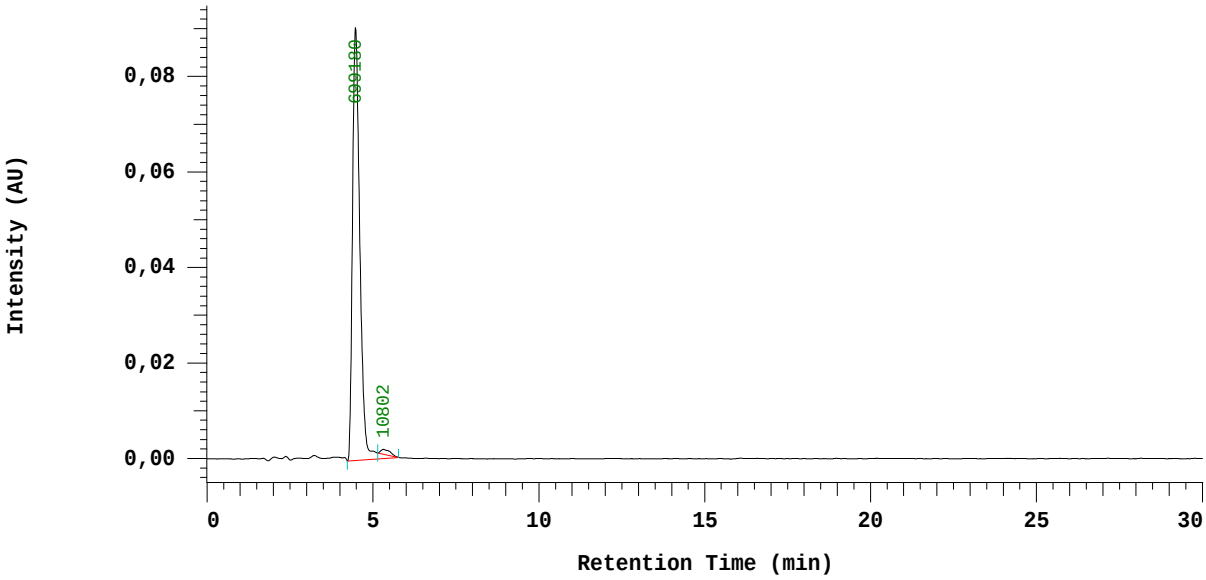
Series:0339 CB4
Vial Number: 12
Vial Type: UNK
Volume: 3,0 ul

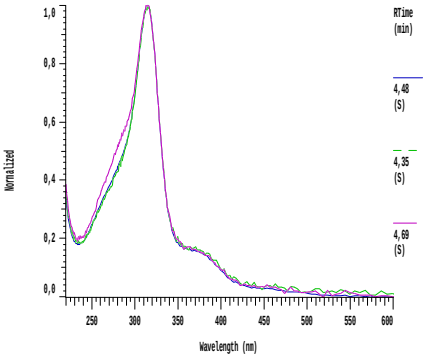
Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.048
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 312 nm





Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 312 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	4,48	699180	98,478		45301	0,9980	
2	5,32	10802	1,522		509		
		709982	100,000		45810		

Peak rejection level: 1000

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Compound 4

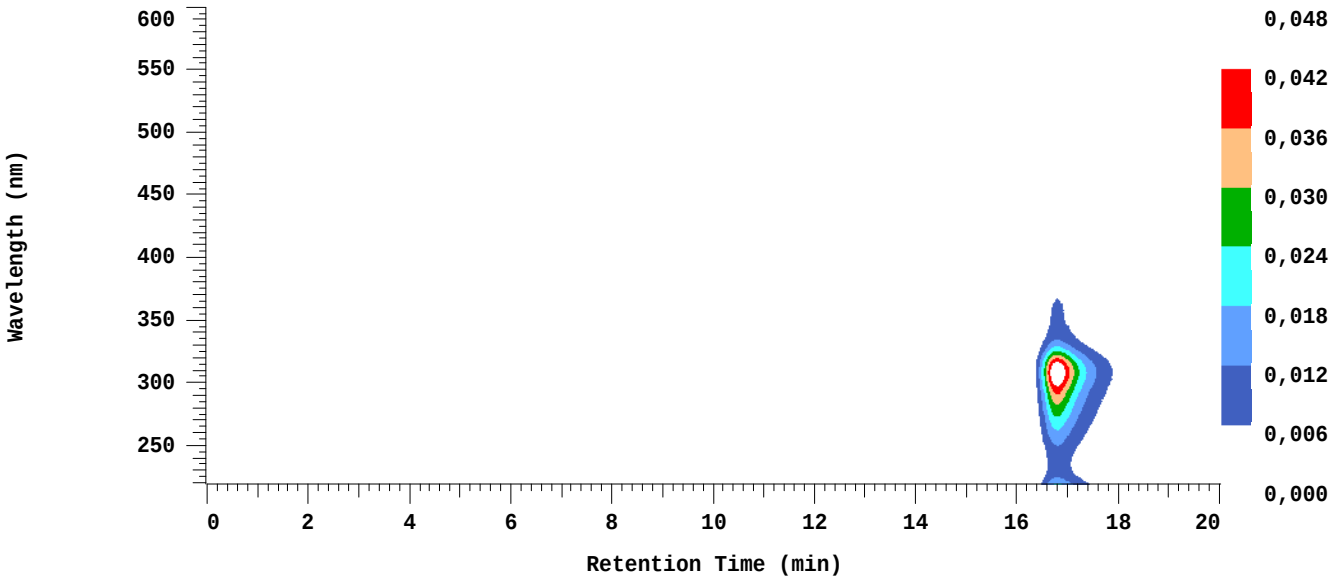
Data Path: C:\WIN32APP\HSM\Samples\DATA\0977 CB15
Processing Method: COSAN-ACCN-new 7100

System(acquisition): Sys 1
Application: Samples
Sample Name: CB15
Injection from this vial: 1 of 1
Sample Description:

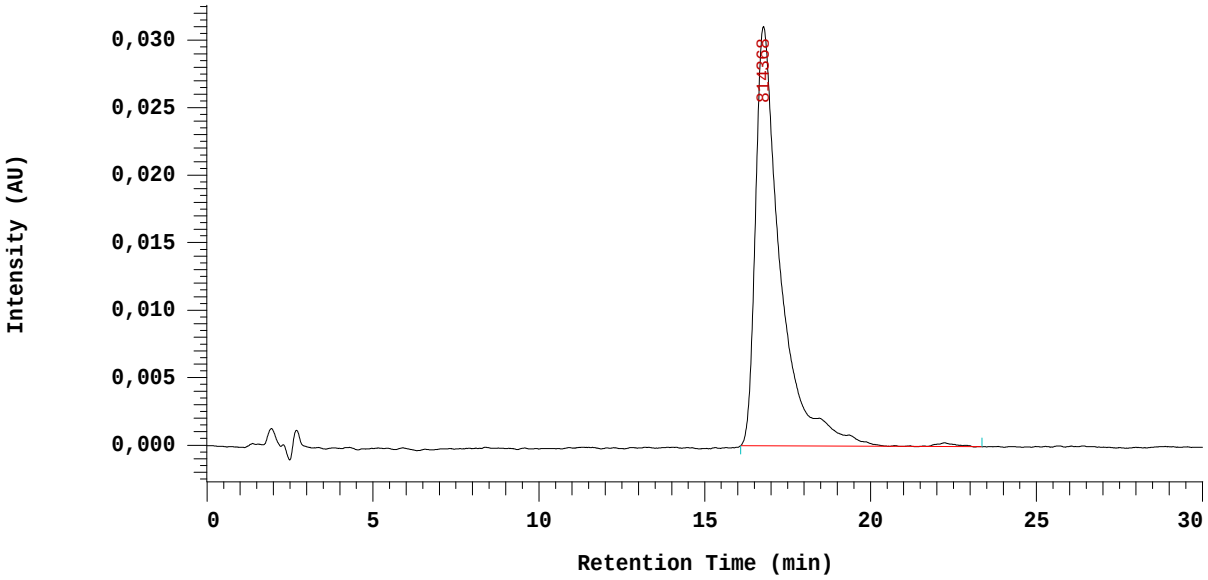
Series:0977 CB15
Vial Number: 10
Vial Type: UNK
Volume: 3,0 ul

Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.048
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm



Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	16,77	814368	100,000		15520	0,9995	
		814368	100,000		15520		

Peak rejection level: 1000

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Compound 5-

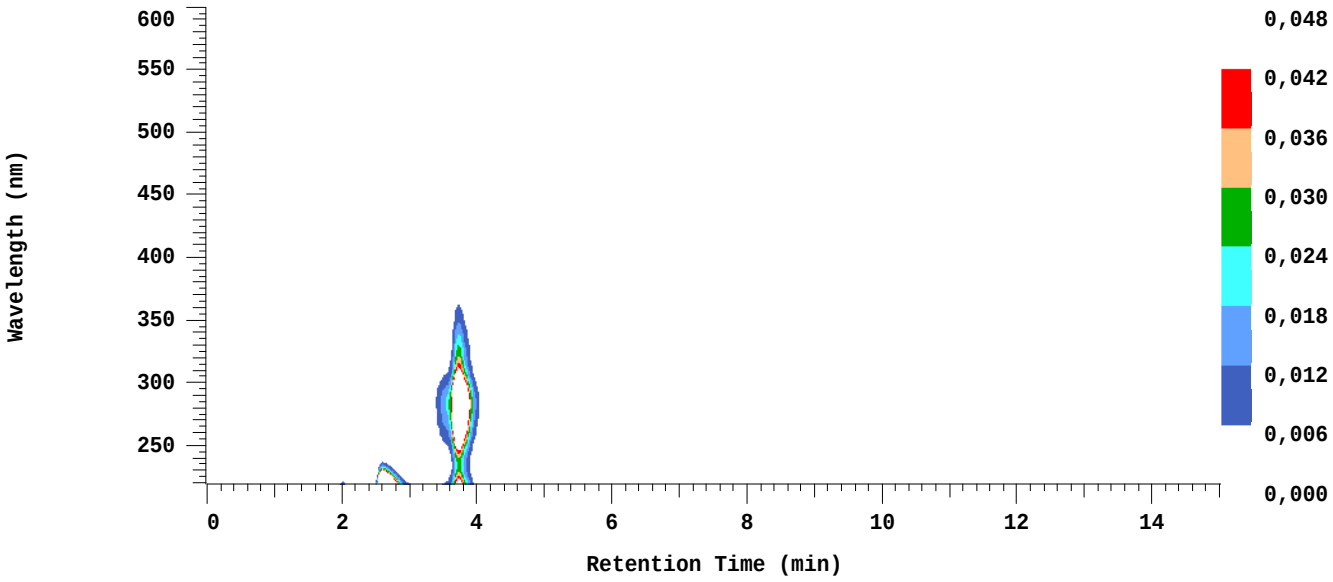
Data Path: C:\Win32App\HSM\samples\DATA\0297\
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB5 stock solution
Injection from this vial: 1 of 1
Sample Description:

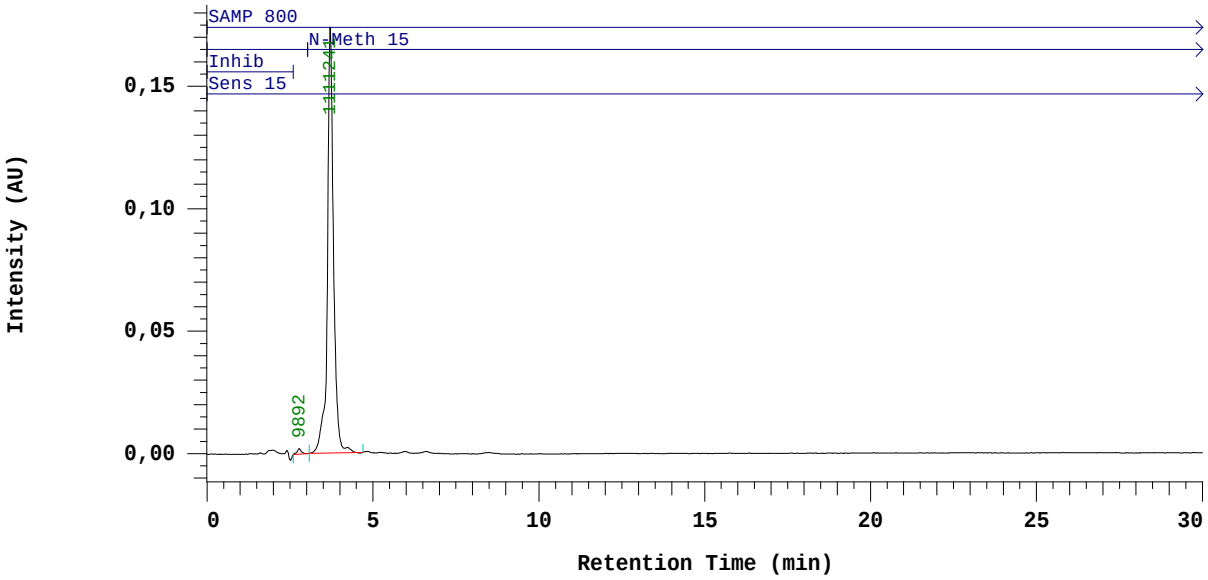
Series:0297
Vial Number: 3
Vial Type: UNK
Volume: 5,0 ul

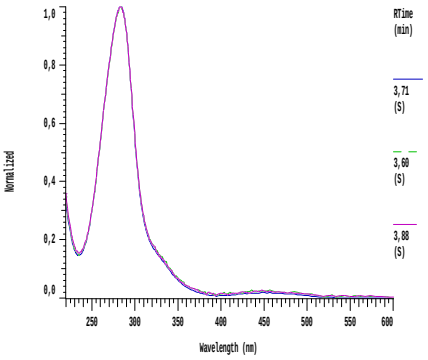
Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.048
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm





Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	2,79	9892	0,882		1113	0,9371	
2	3,71	1111241	99,118		86906	1,0000	
		1121133	100,000		88019		

Peak rejection level: 1000

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Compound 6-

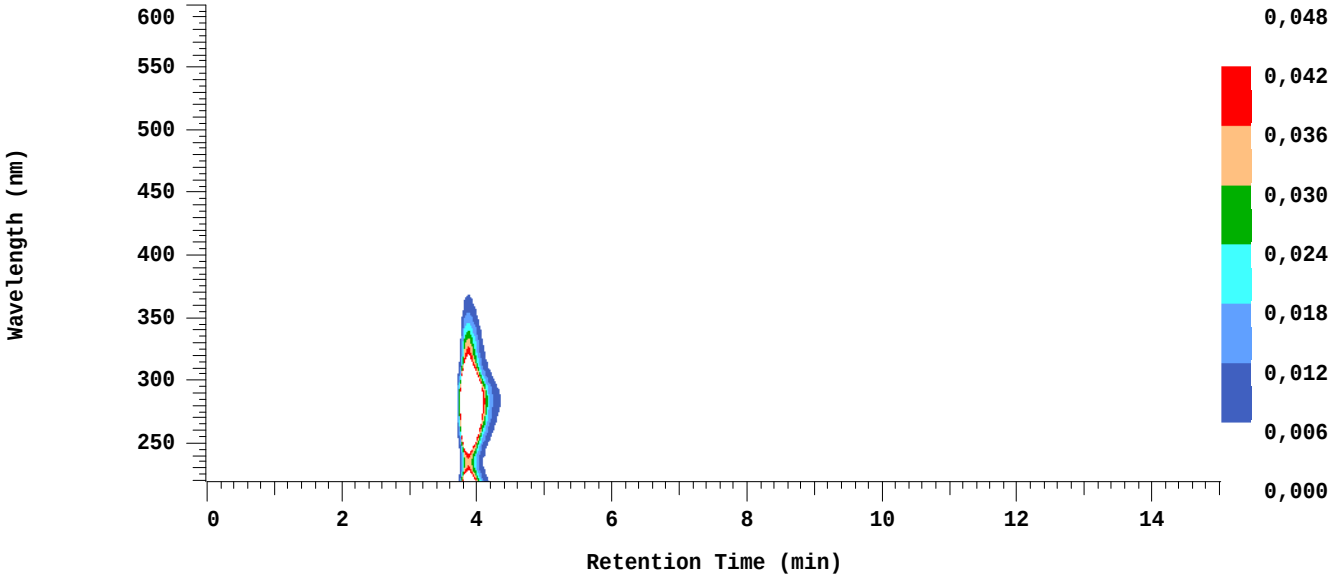
Data Path: C:\WIN32APP\HSM\Samples\DATA\0979 CB30
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB30Na main
Injection from this vial: 1 of 1
Sample Description:

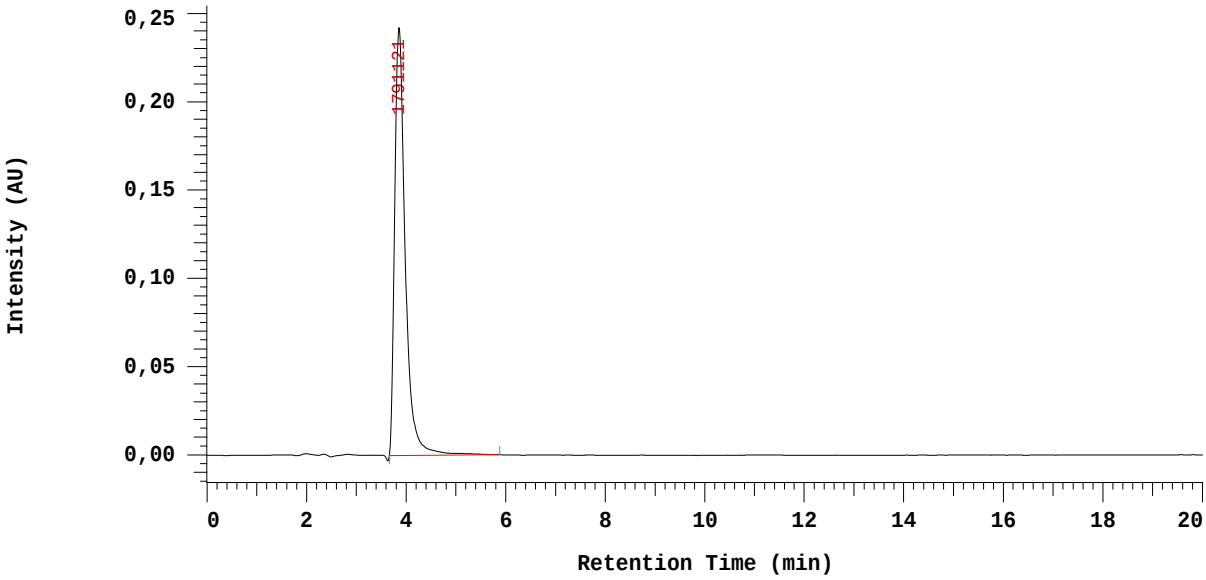
Series:0979 CB30
Vial Number: 17
Vial Type: UNK
Volume: 3,0 ul

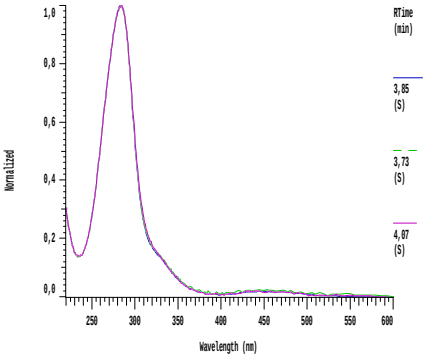
Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.048
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm





Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	3,85	1791121	100,000		121056	0,9999	
		1791121	100,000		121056		

Peak rejection level: 1000

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Compound 7-

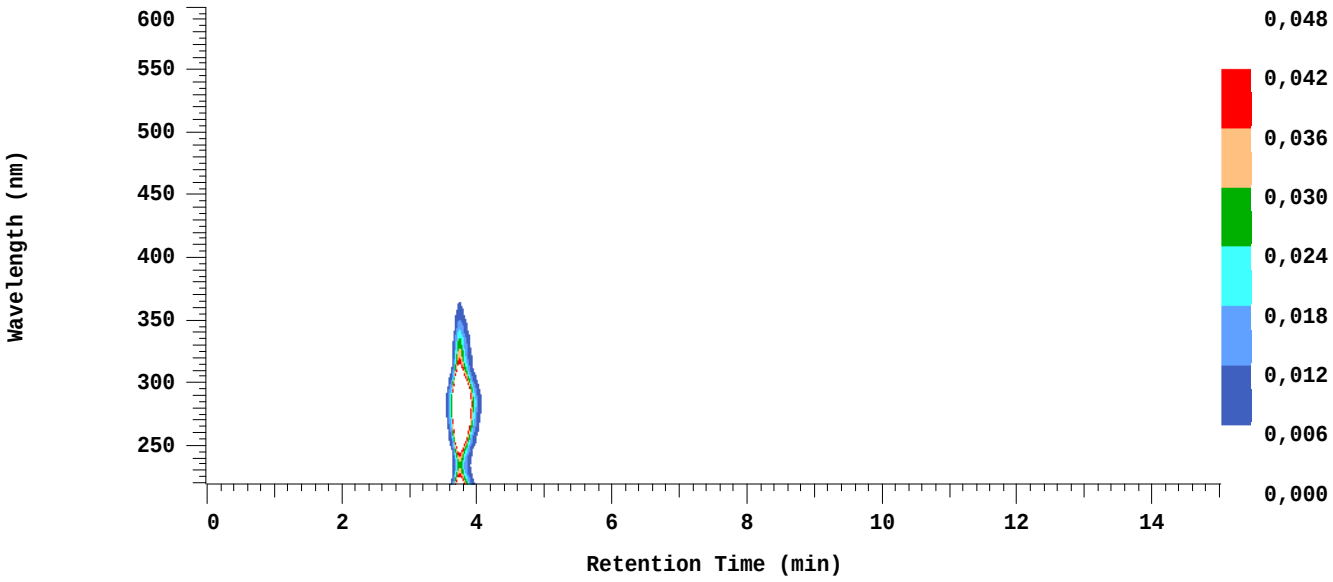
Data Path: C:\Win32App\HSM\samples\DATA\0356\
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB29
Injection from this vial: 1 of 1
Sample Description:

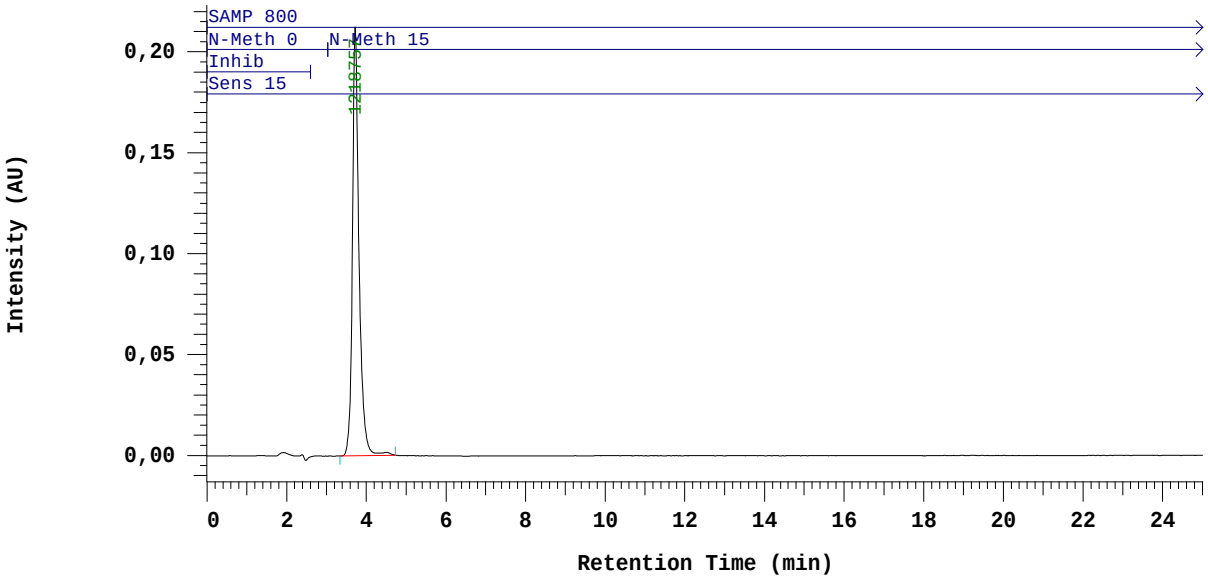
Series:0356
Vial Number: 19
Vial Type: UNK
Volume: 3,0 ul

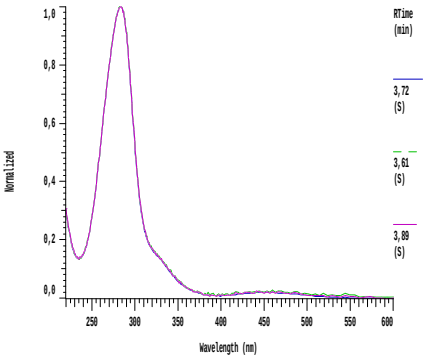
Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.048
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm





Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	3,73	1218757	100,000		106156	1,0000	
		1218757	100,000		106156		

Peak rejection level: 1000

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Compound 8-

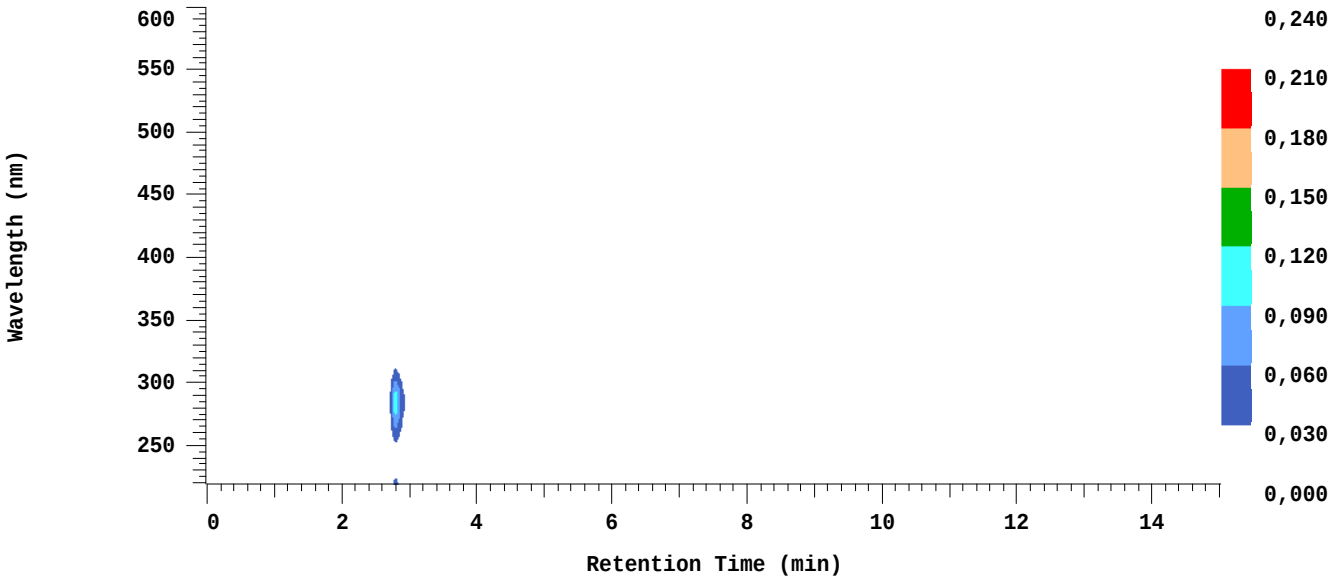
Data Path: C:\WIN32APP\HSM\Samples\DATA\0982 CB31
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB31Na main
Injection from this vial: 1 of 1
Sample Description:

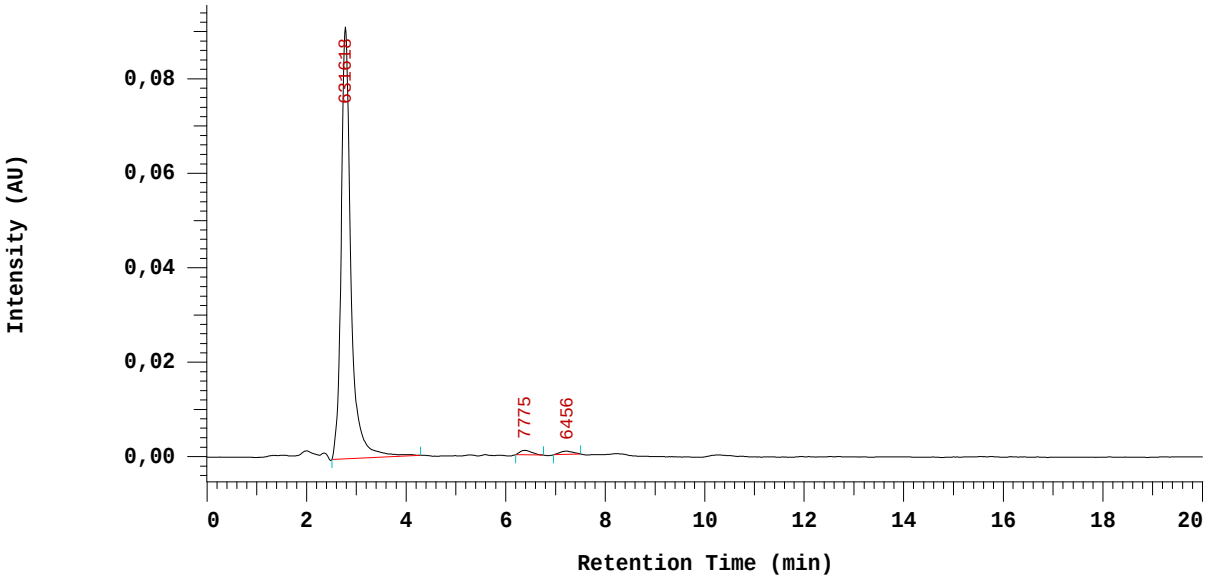
Series:0982 CB31
Vial Number: 18
Vial Type: UNK
Volume: 3,0 ul

Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.240
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm



Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	2,79	631618	97,796		45696	0,9999	
2	6,39	7775	1,204		477		
3	7,24	6456	1,000		342		
		645849	100,000		46515		

Peak rejection level: 1000

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Compound 9-

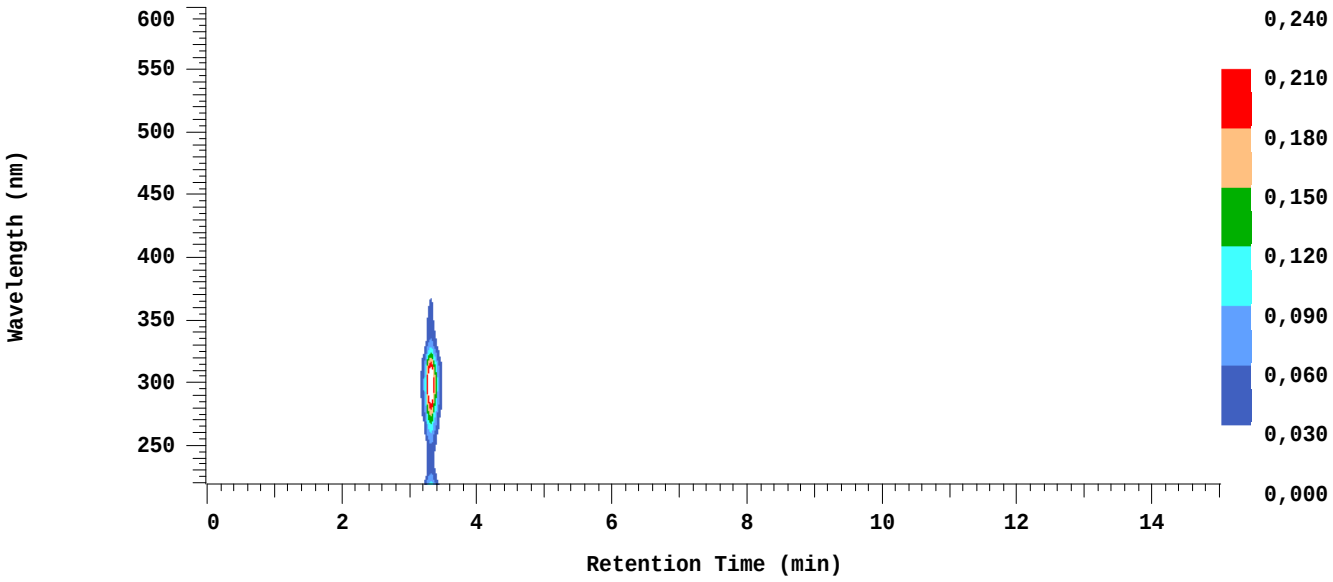
Data Path: C:\WIN32APP\HSM\Samples\DATA\0971
Processing Method: COSANS-ACCN-new 7100-short

System(acquisition): Sys 1
Application: Samples
Sample Name: CB52
Injection from this vial: 1 of 1
Sample Description:

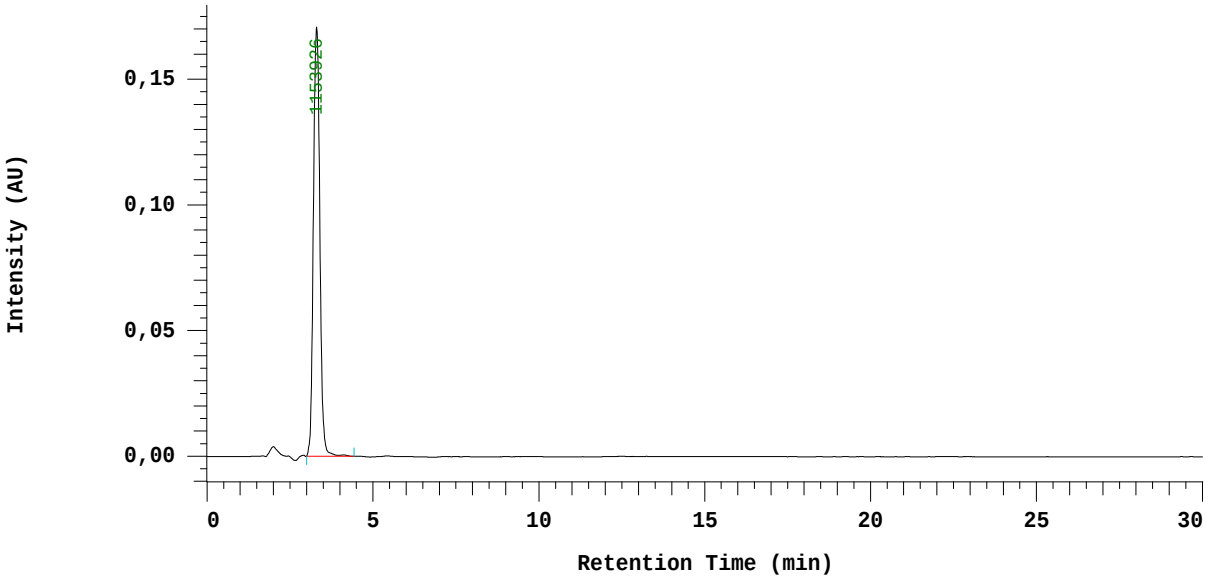
Series:0971 CB52
Vial Number: 9
Vial Type: UNK
Volume: 3,0 ul

Absorbance Mode: NORMAL(2.0 AU)
Spectral Bandwidth: 4 nm

Absorbance Scale: 0.240
Spectral Interval: 800 ms



Chrom Type: Fixed WL Chromatogram, 285 nm



Acquisition Method: COSANS-ACCN-new 7100-short
Column Type: 123 Developed by: Gruner
Pump A Type: L-7100
Solvent A: 58% ACCN-H2O, 4.5 Mm HA
Method Description: Determination of substituted COSANS

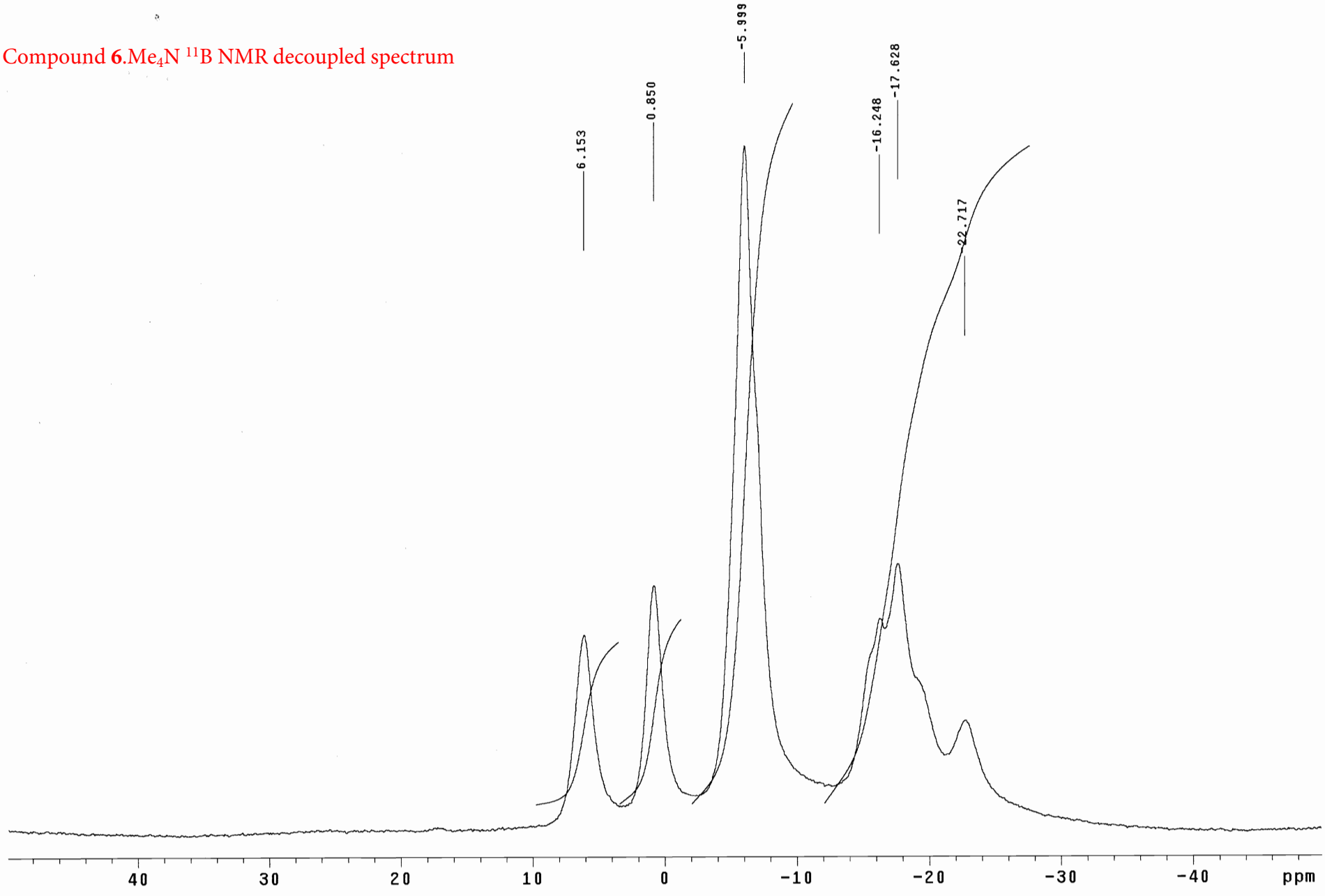
Chrom Type: Fixed WL Chromatogram, 285 nm

Peak Quantitation: AREA
Calculation Method: AREA%

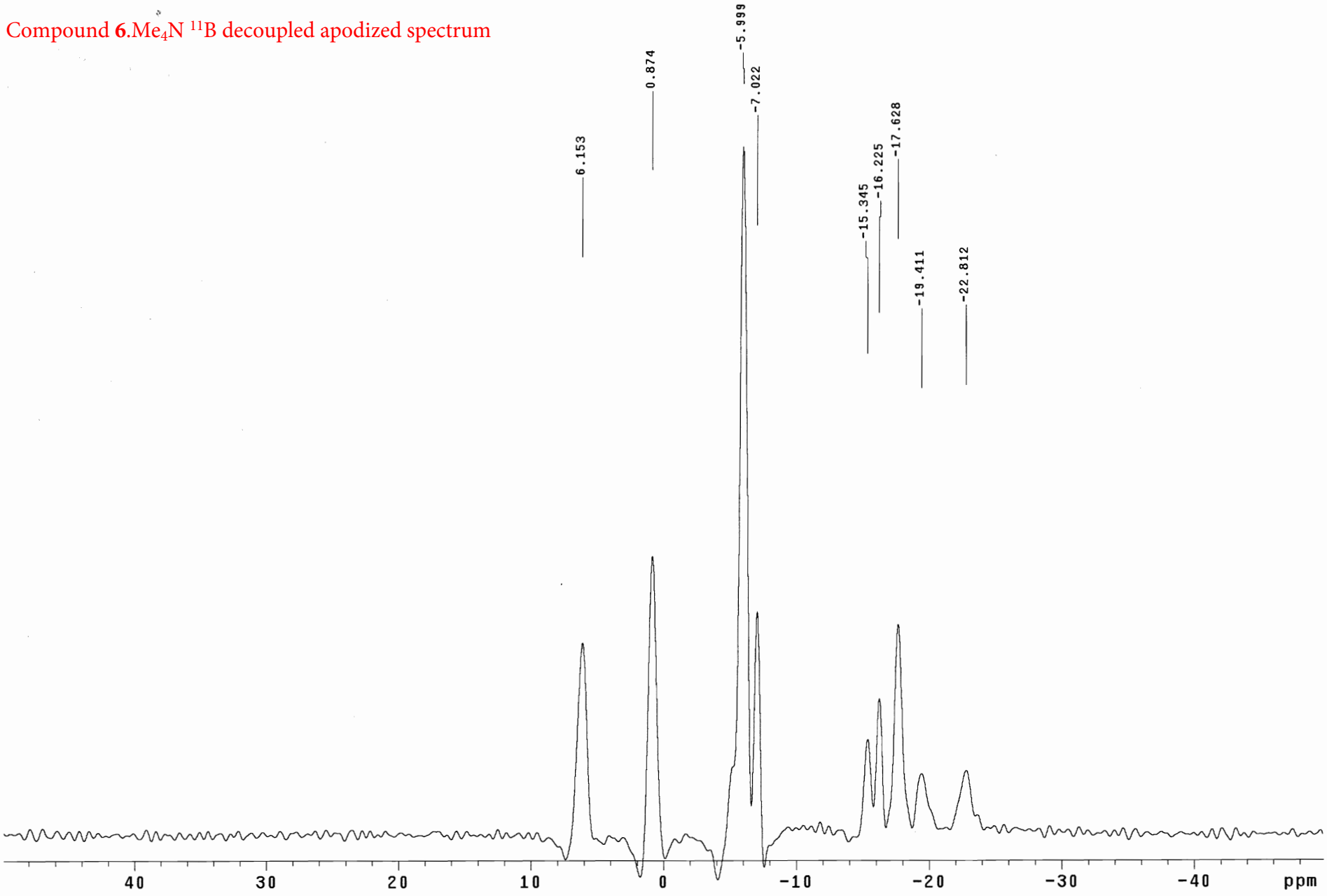
No.	RT	Area	Area %	Name	Height	Purity	R-Factor
1	3,31	1153926	100,000		85379		
		1153926	100,000		85379		

Peak rejection level: 1000

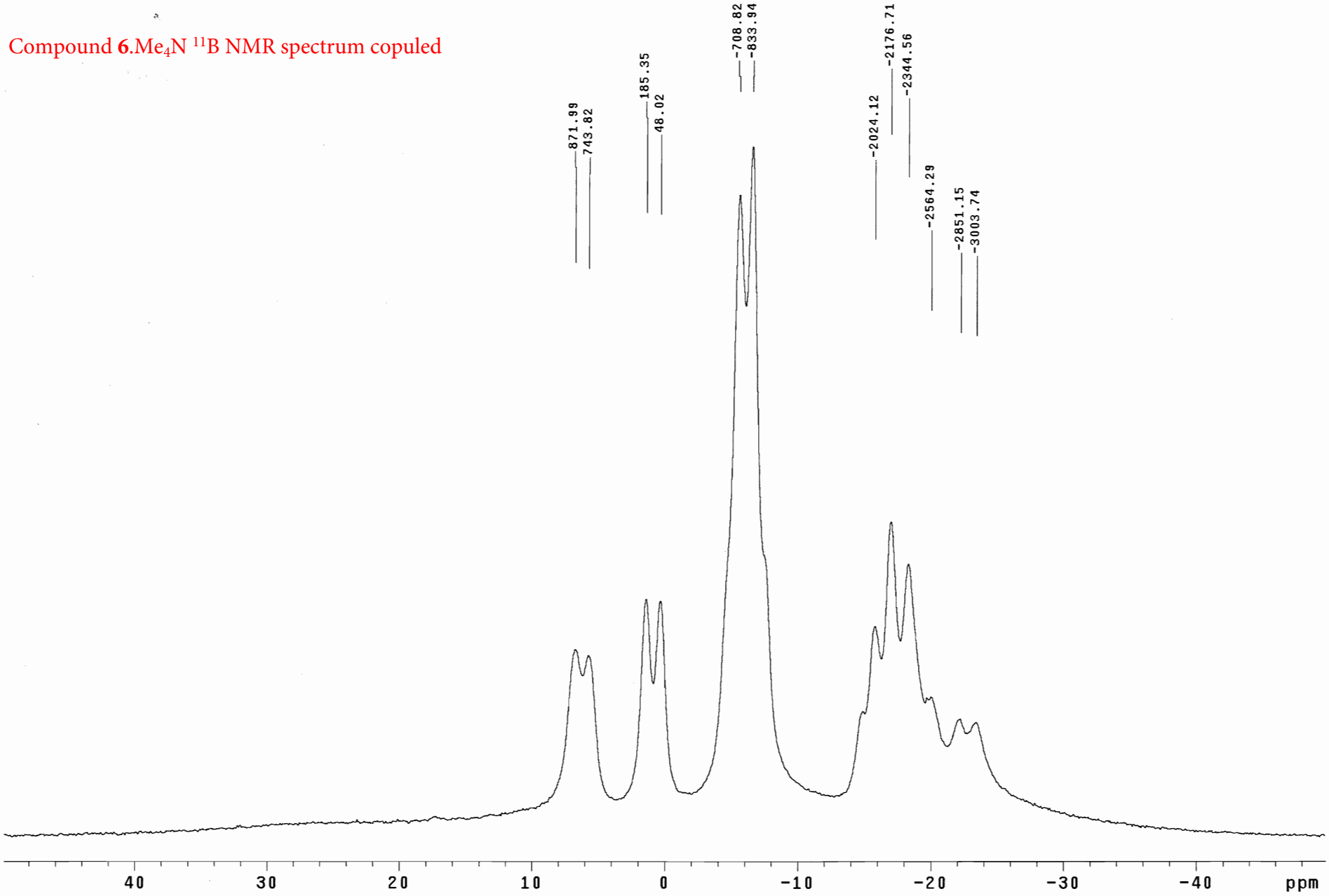
Compound **6**.Me_4N ^{11}B NMR decoupled spectrum



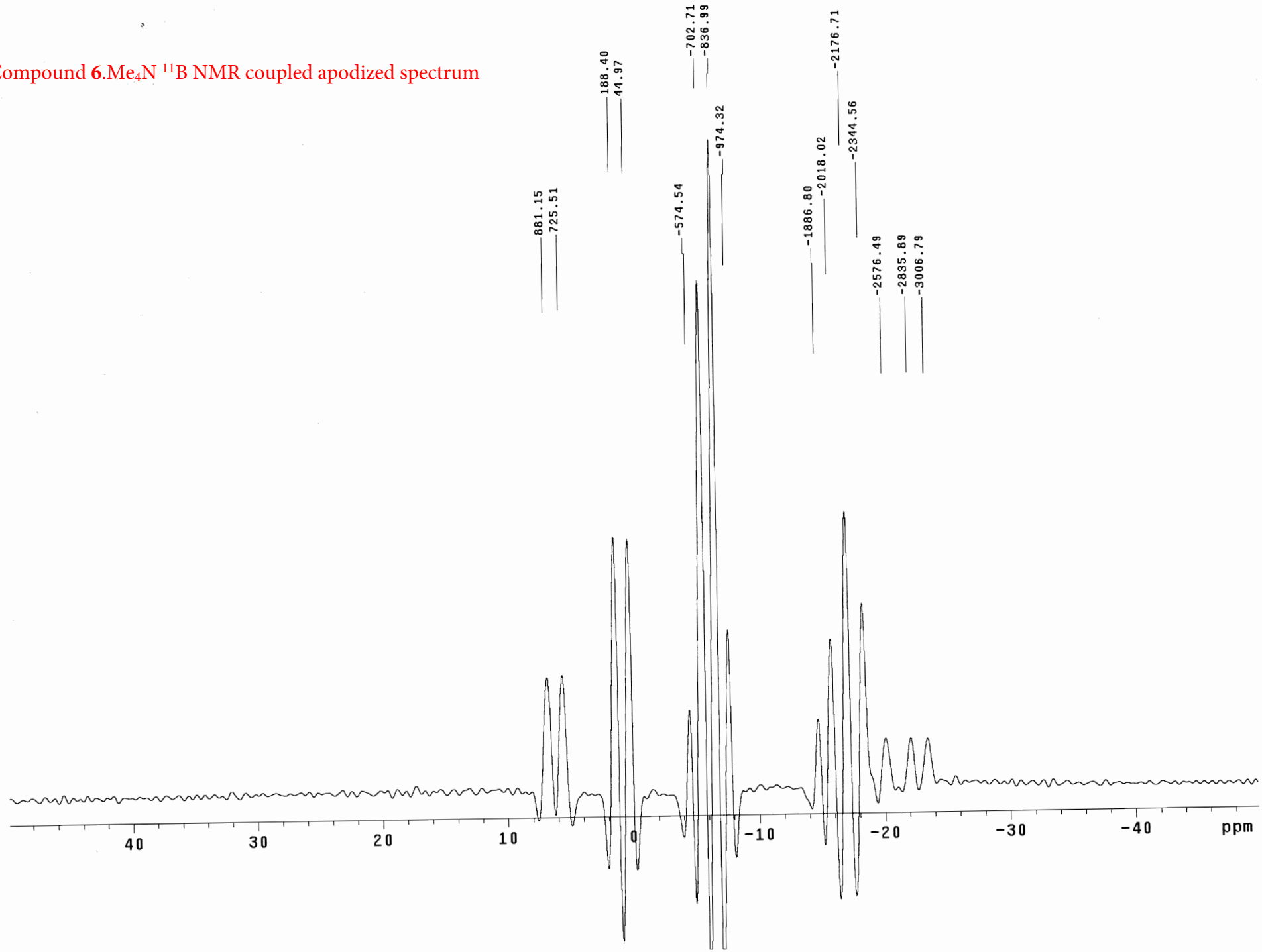
Compound **6**.Me_4N ^{11}B decoupled apodized spectrum



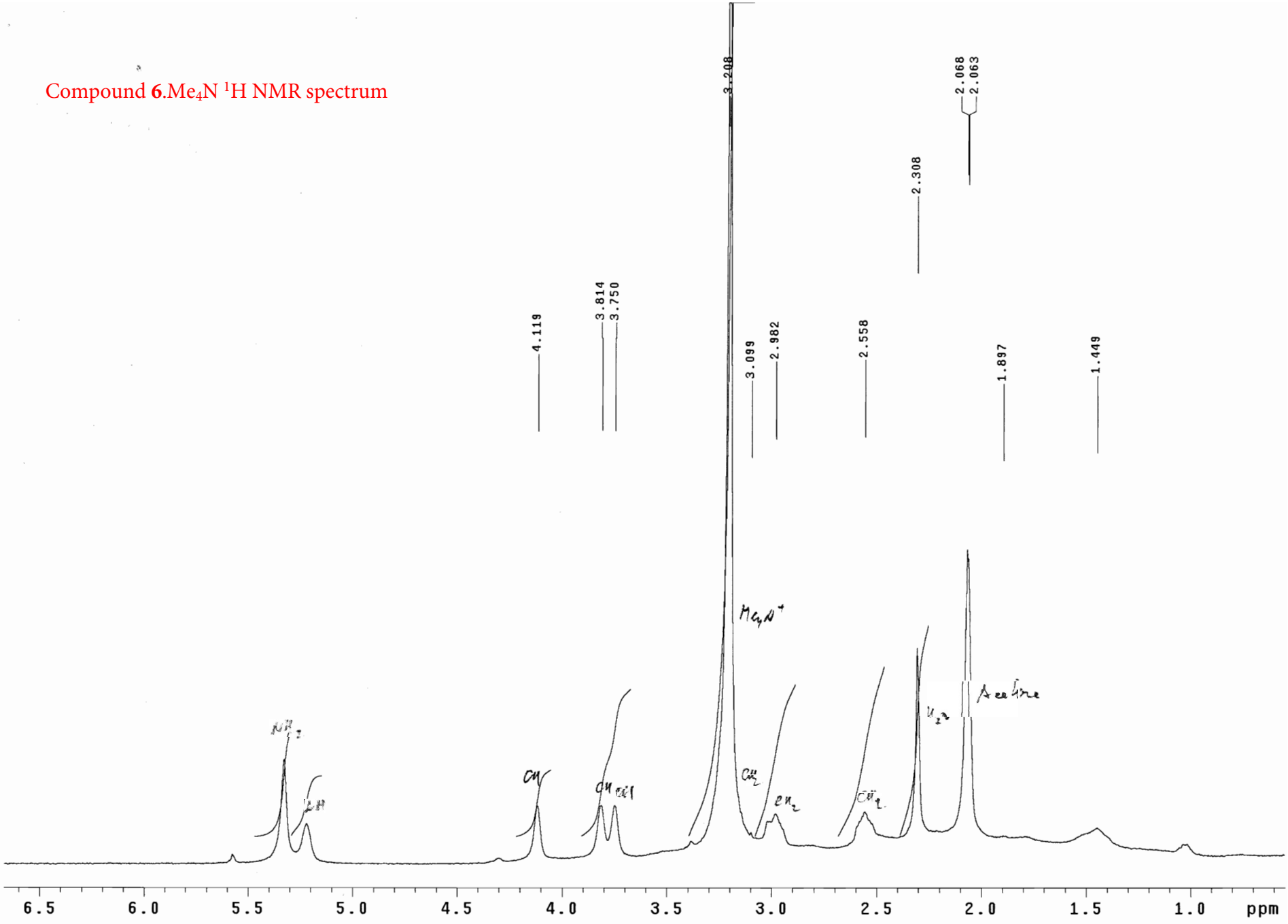
Compound **6**.Me_4N ^{11}B NMR spectrum copuled



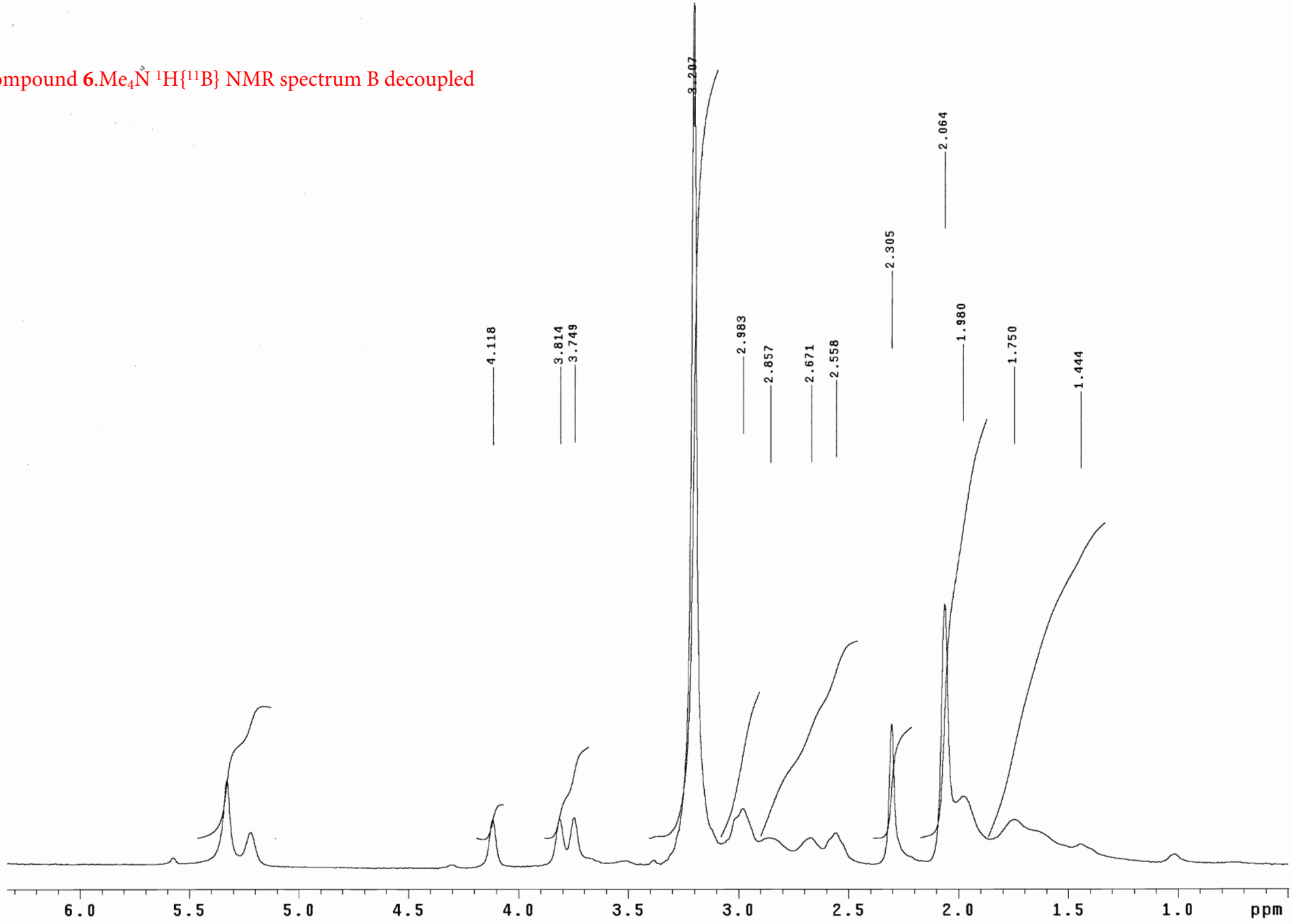
Compound **6**.Me_4N ^{11}B NMR coupled apodized spectrum



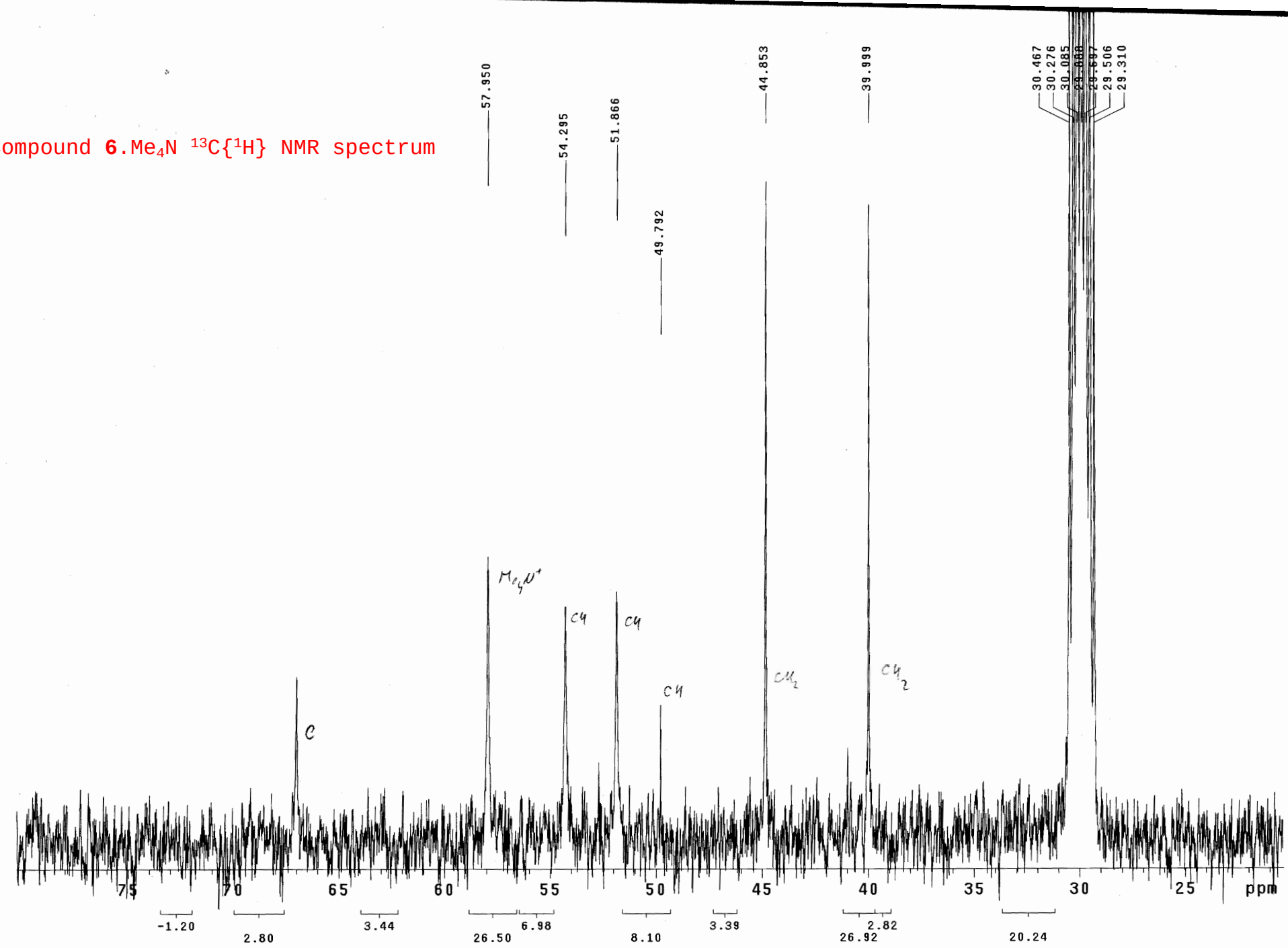
Compound **6**.Me_4N ^1H NMR spectrum



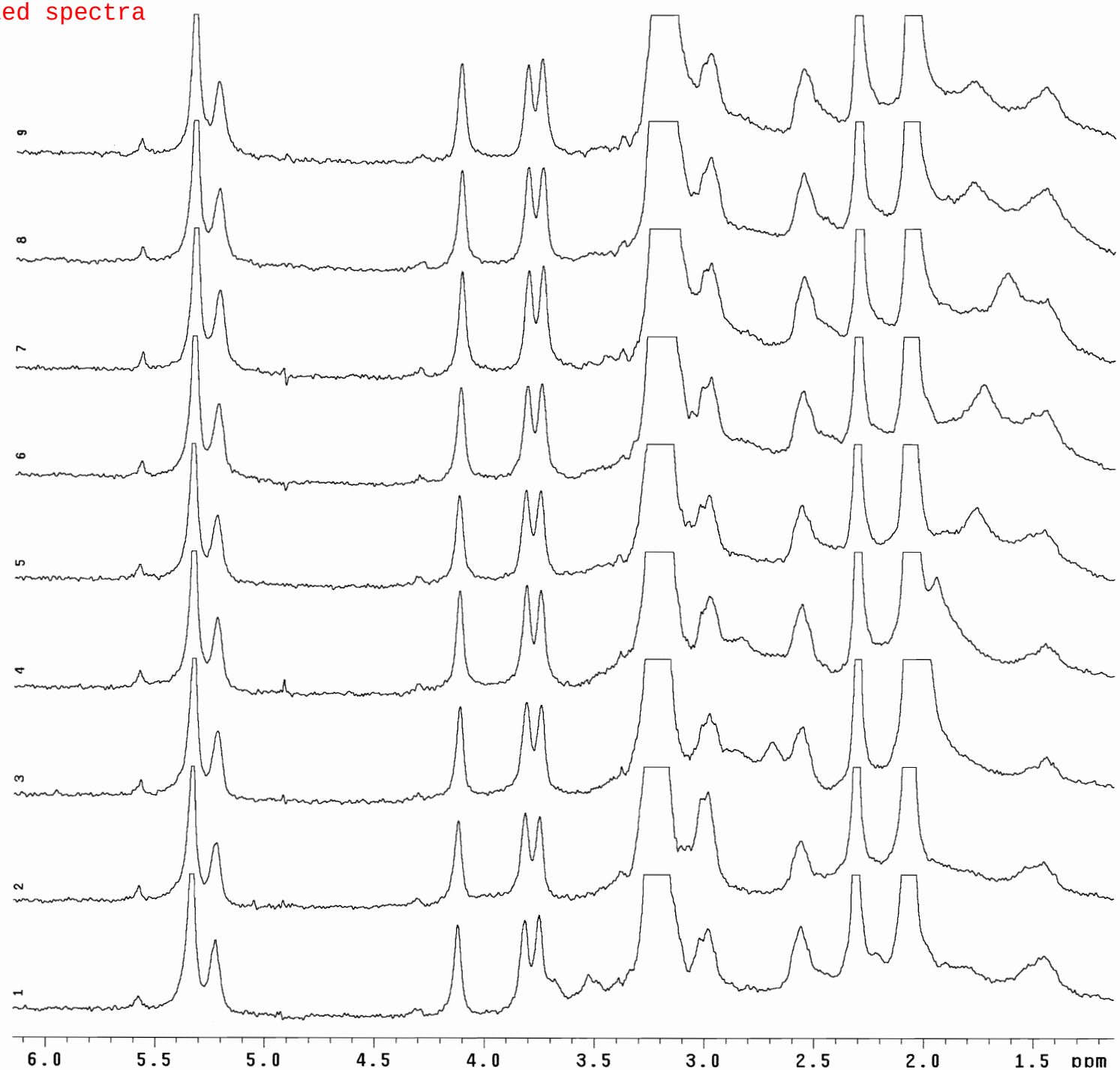
Compound **6**.$Me_4\overset{\oplus}{N}$ $^1H\{^{11}B\}$ NMR spectrum B decoupled



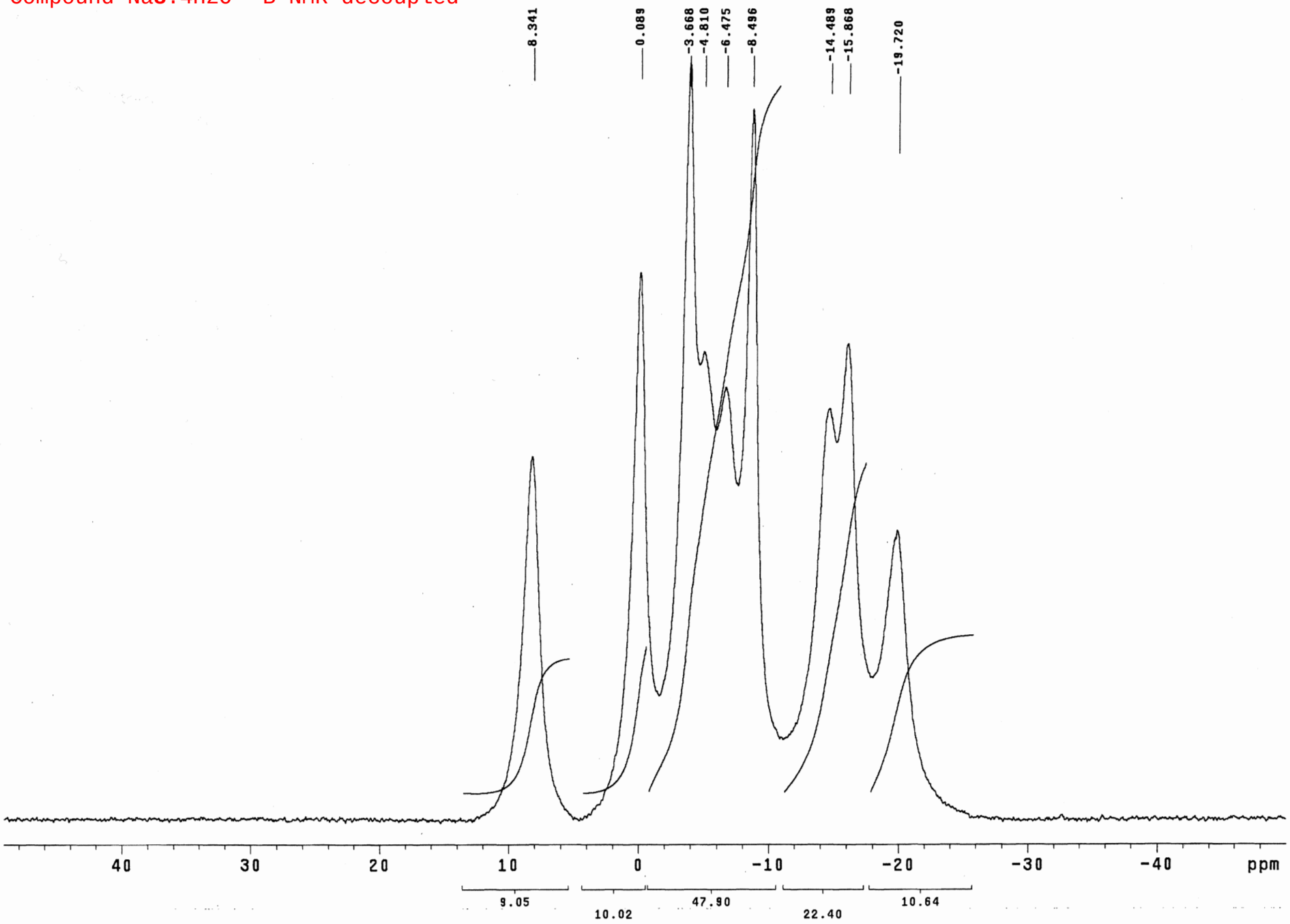
Compound **6**.Me_4N $^{13}C\{^1H\}$ NMR spectrum



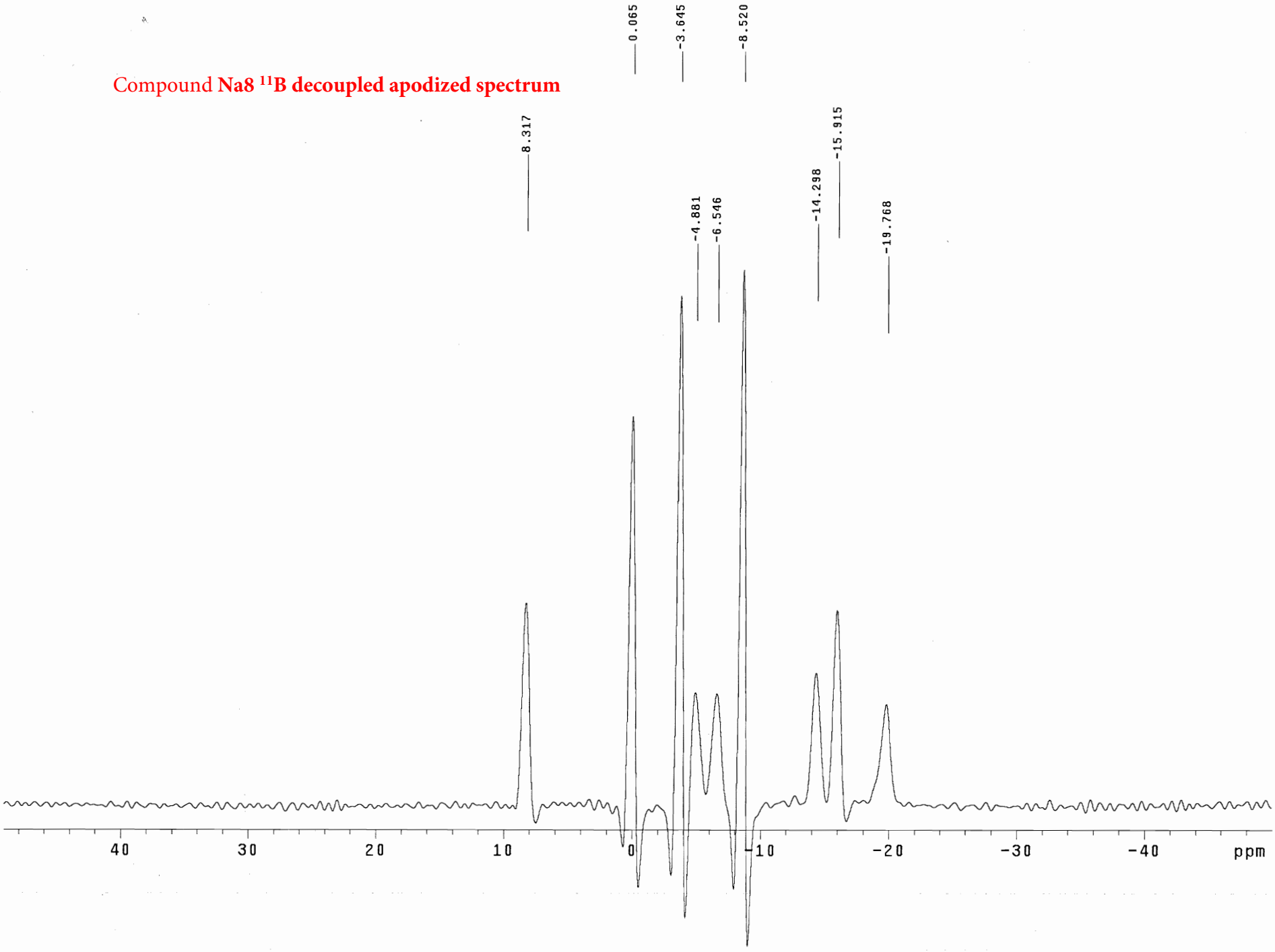
Compound **6**.Me_4N $^1H\{^{11}B\}$ NMR
array of selectively decoupled spectra



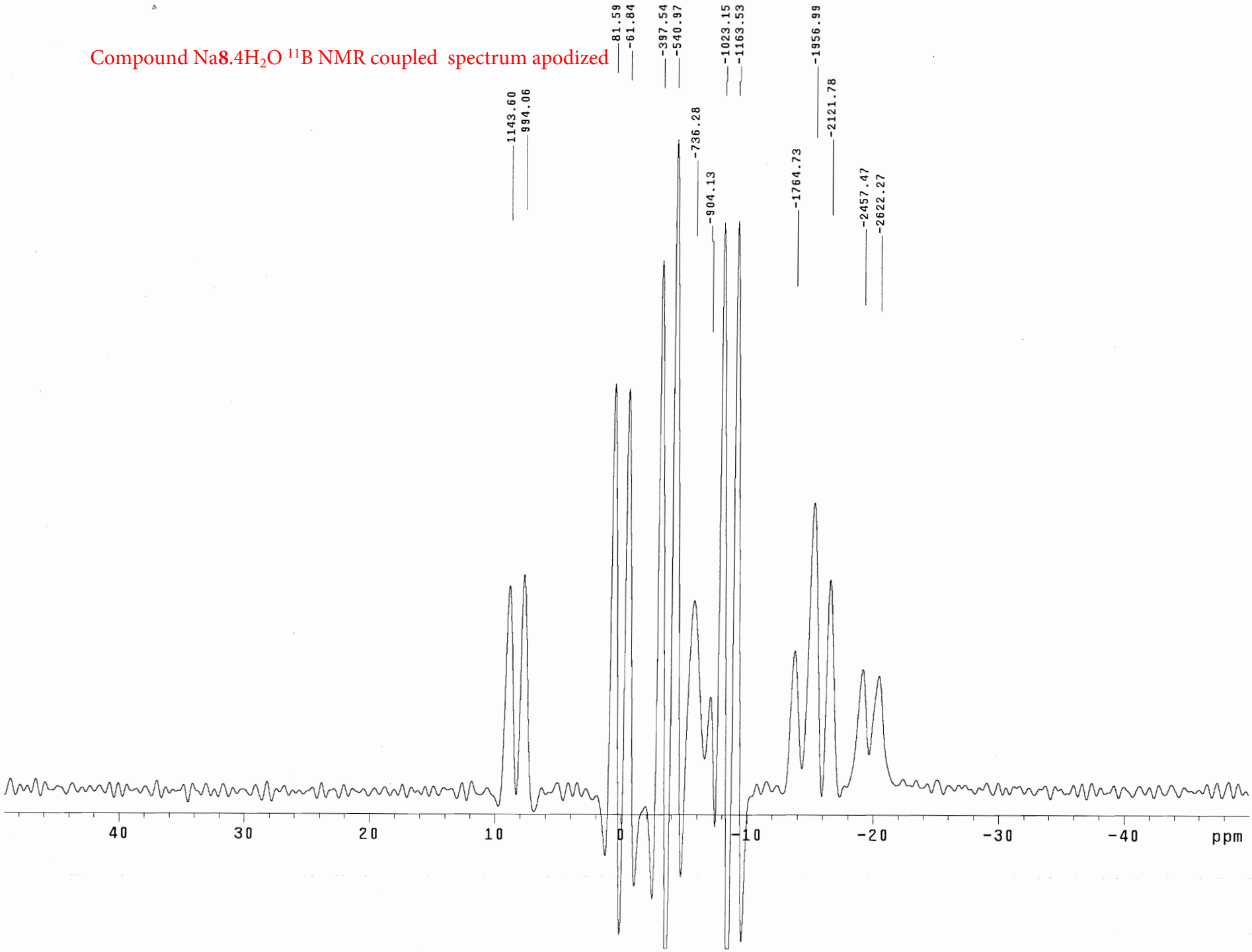
Compound Na**8**.4H2O ^{11}B NMR decoupled



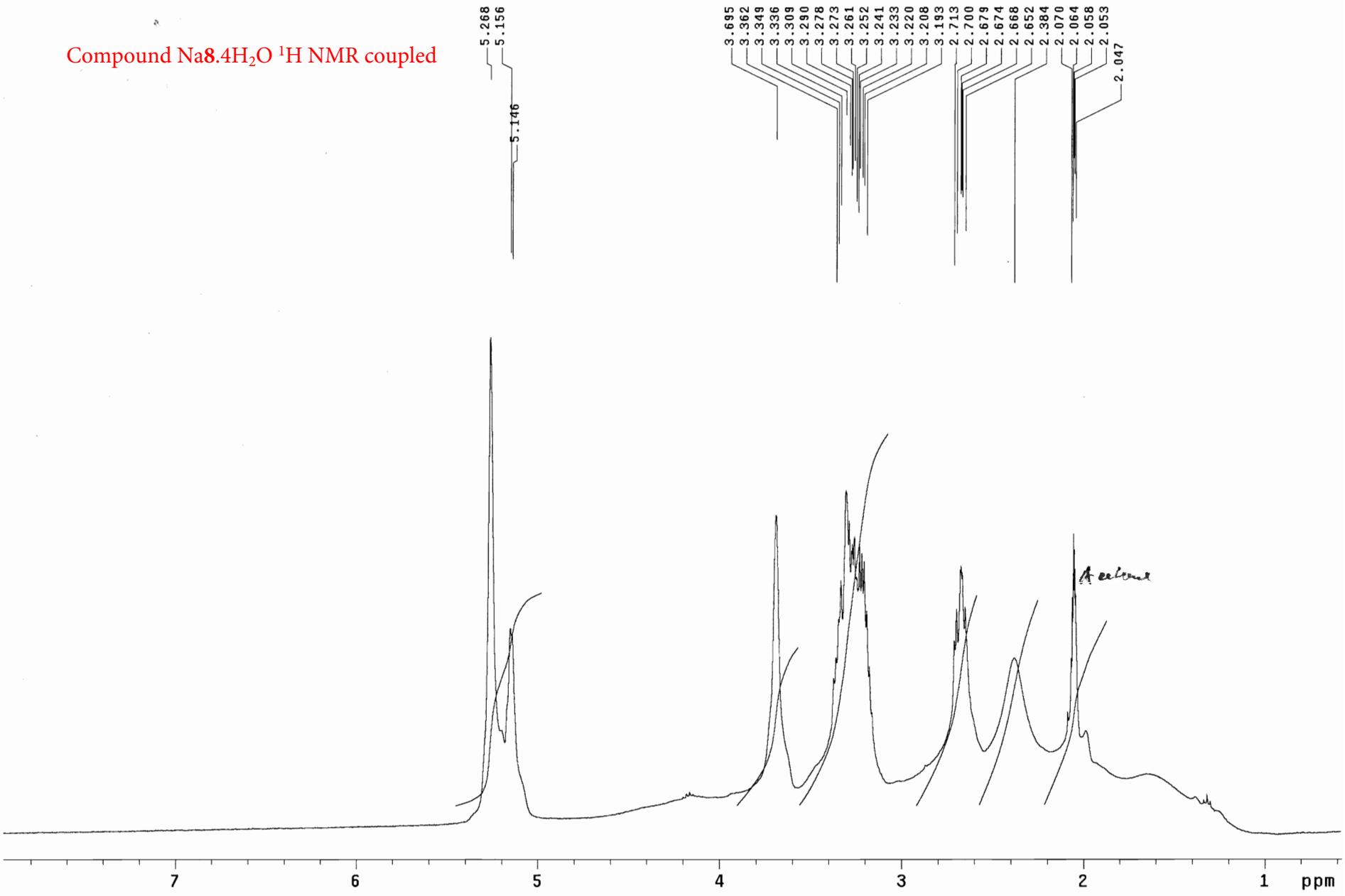
Compound **Na8 ^{11}B decoupled apodized spectrum**



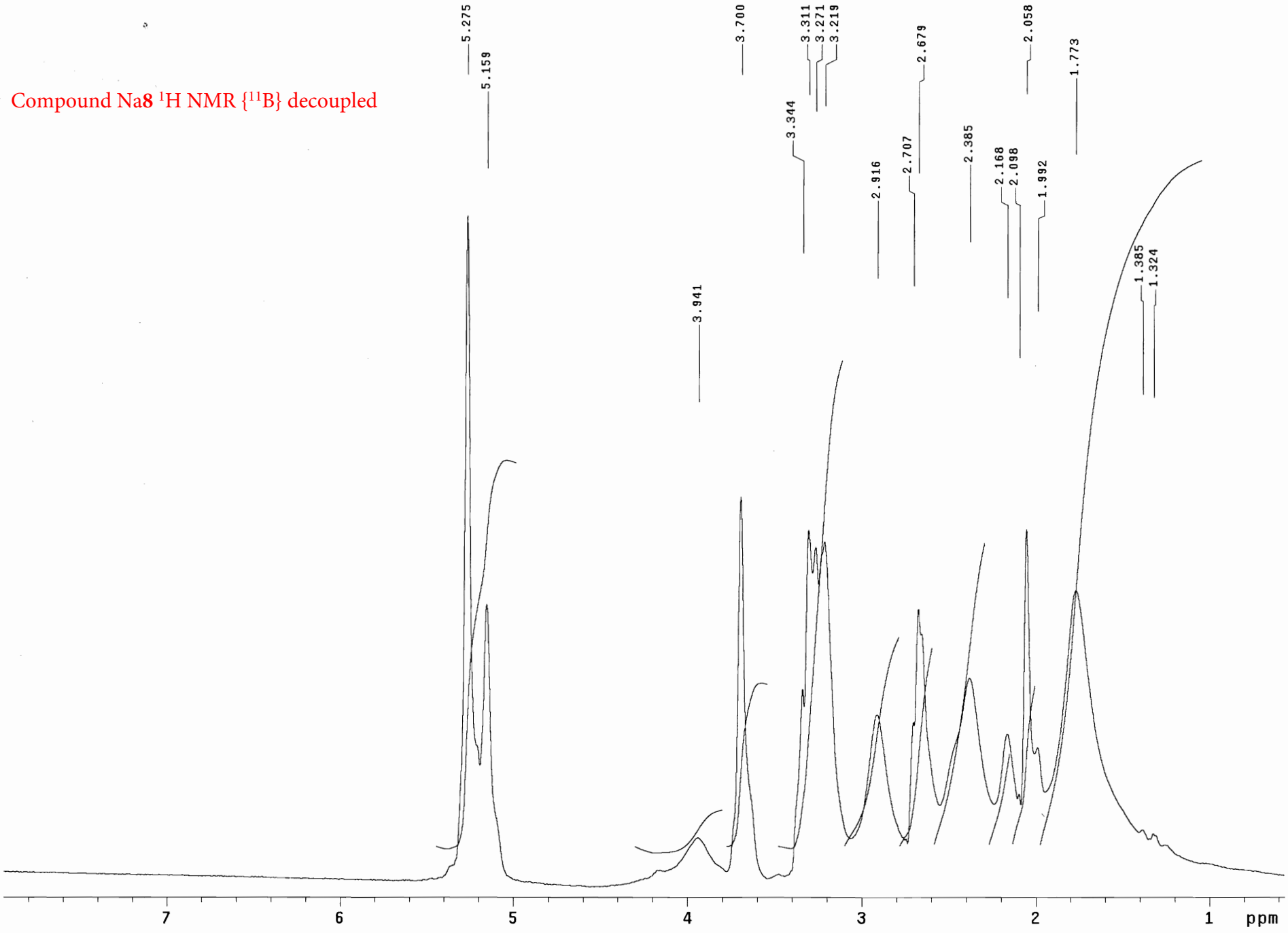
Compound Na**8**.4H_2O ^{11}B NMR coupled spectrum apodized



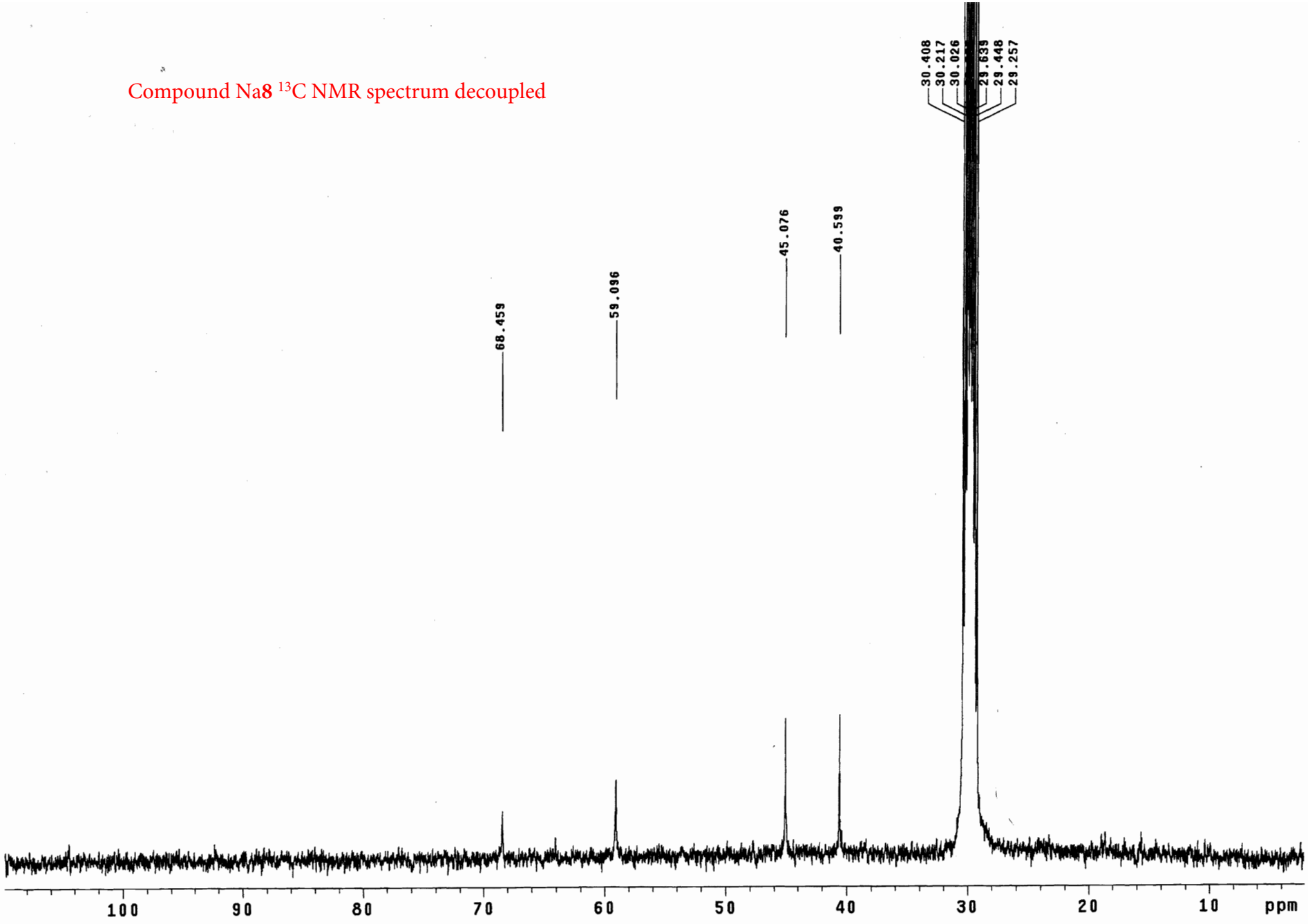
Compound Na**8**.4H_2O ^{1}H NMR coupled



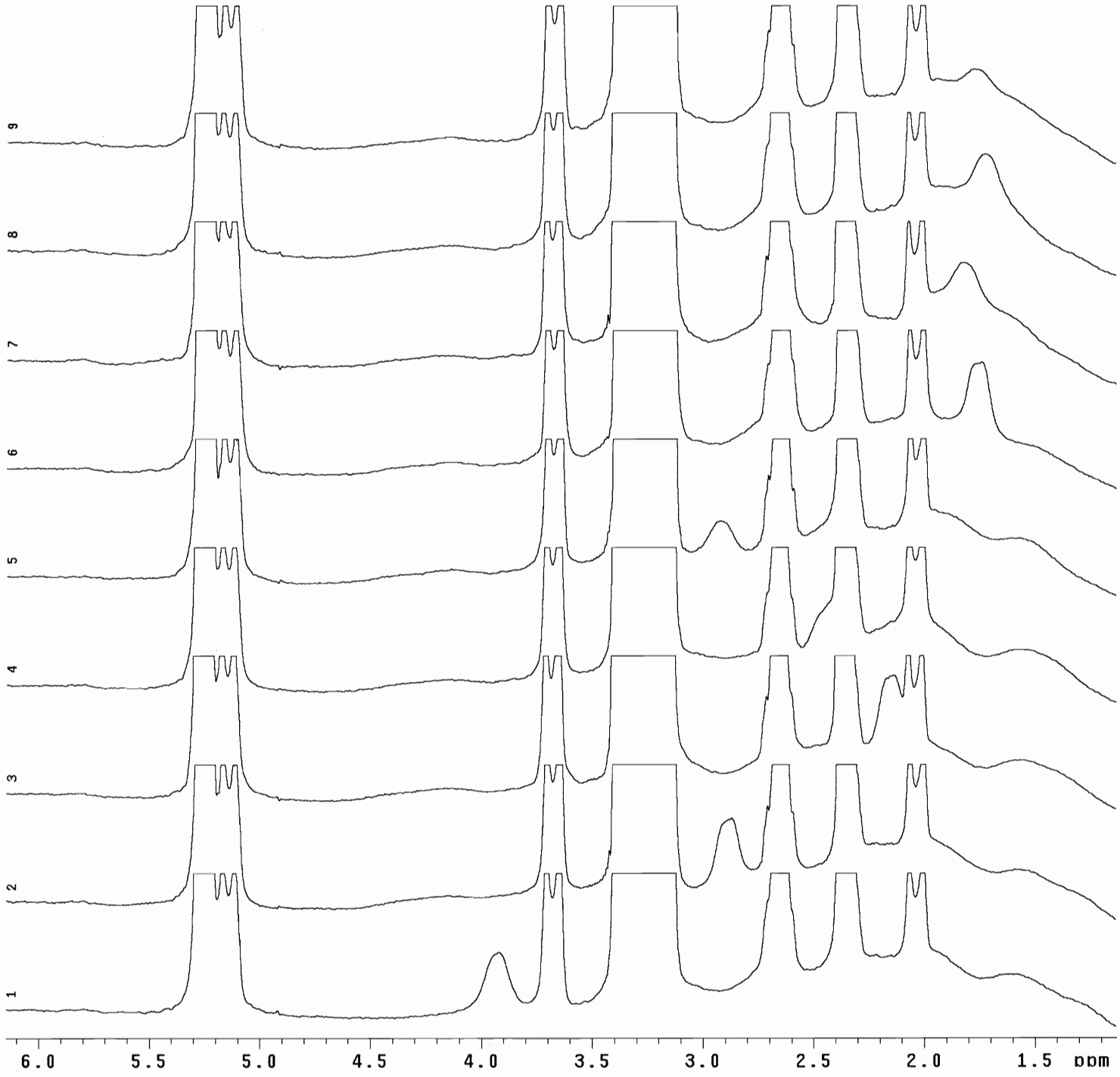
Compound Na**8** ^{1}H NMR {^{11}B} decoupled



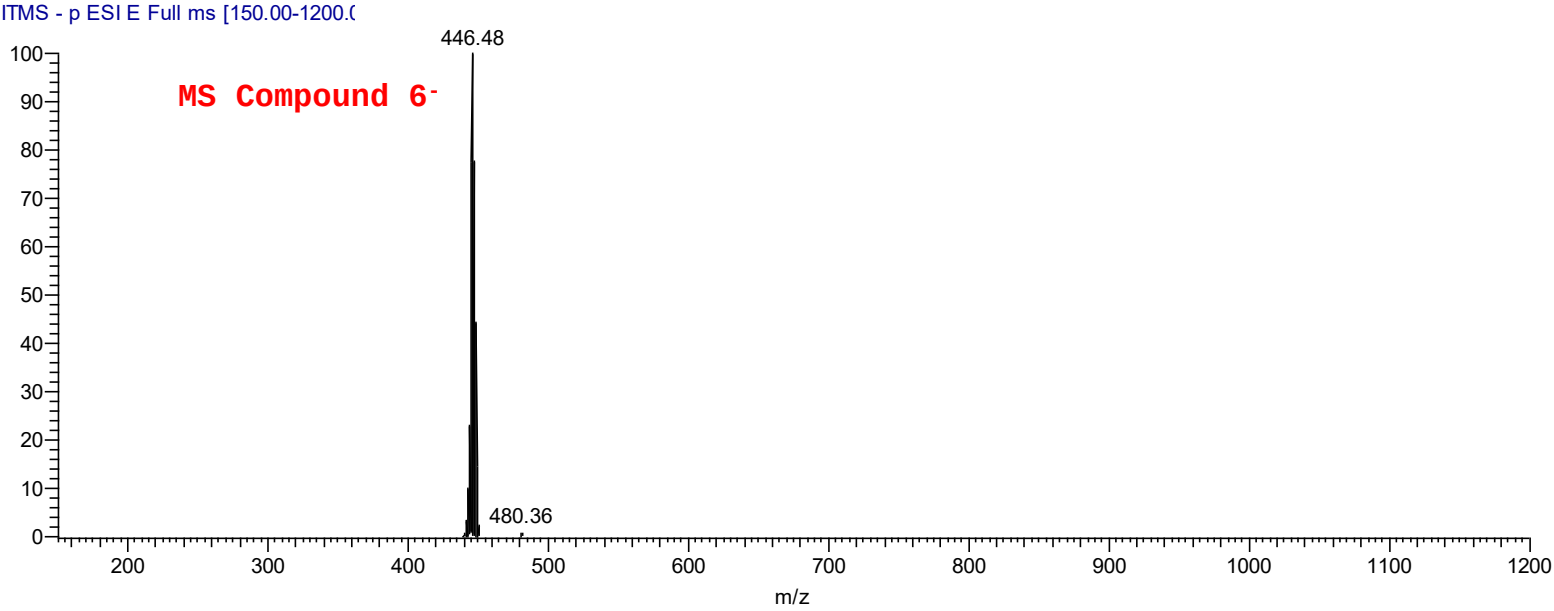
Compound Na**8** ^{13}C NMR spectrum decoupled

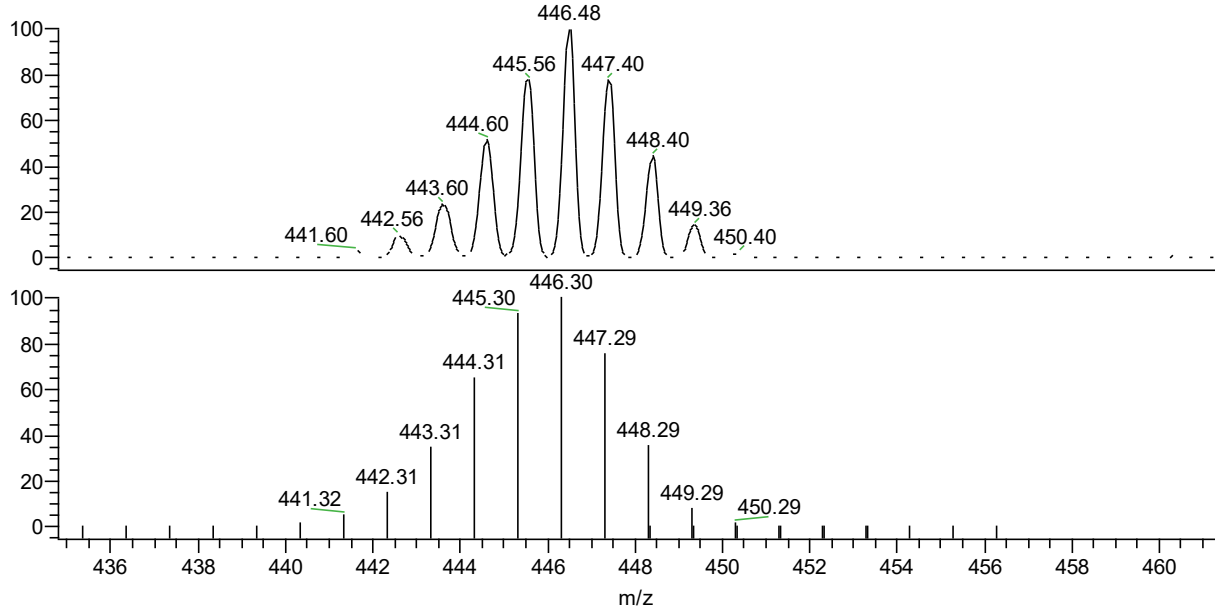


Compound Na8 $^{1}H\{^{11}B\}$ array of ^{11}B selectively decoupled NMR spectra



ESI C4B18H21CoC2H4NHSO2NH2 CB30_2 RT: 0.00 AV: 1 NL: 2.74E4
T: ITMS - p ESI E Full ms [150.00-1200.0

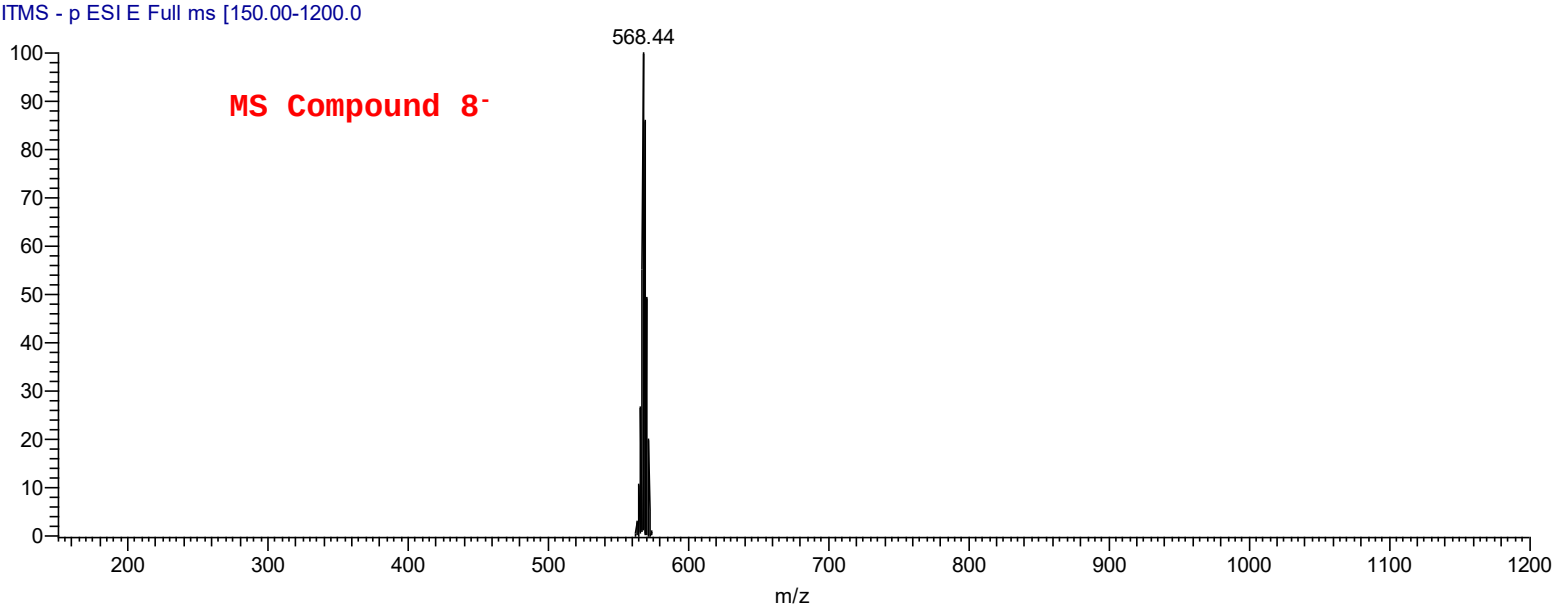


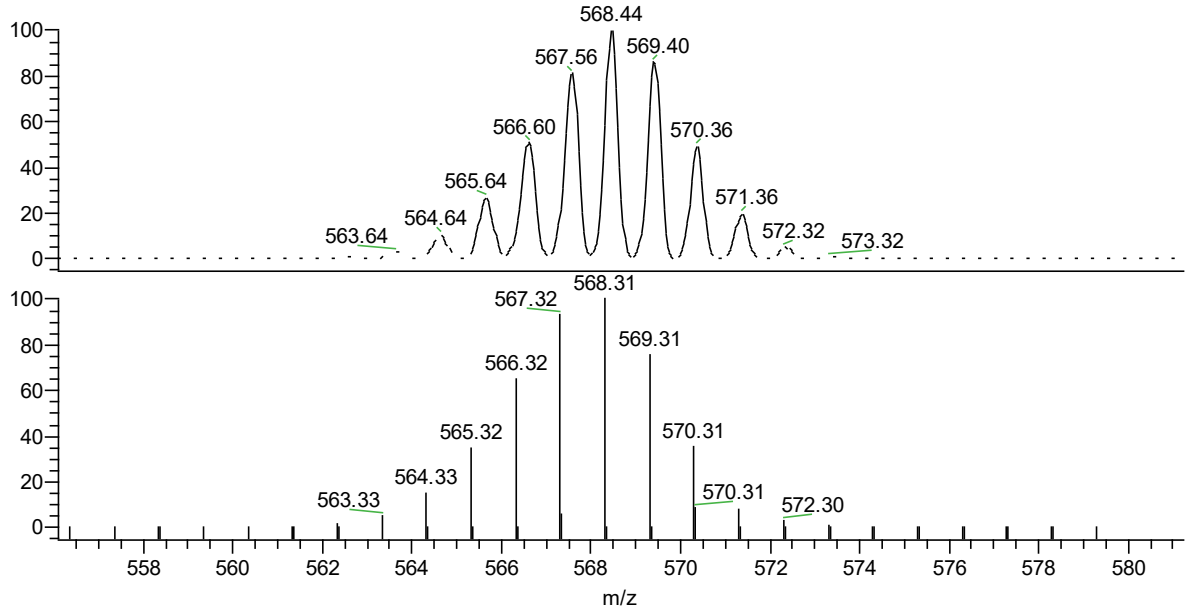


NL:
2.74E4
ESI
C4B18H21CoC2H4NHSO2NH2
CB30_2_190515121102#1 RT:
0.00 AV: 1 T: ITMS - p ESI E
Full ms [150.00-1200.00]

NL:
2.02E5
$C_4B_{18}H_{21}CoC_2H_4NHSO_2NH_2$:
$C_6B_{18}H_{28}Co_1N_2S_1O_2$
pa Chrg 1

ESI C4B18H20Co(C2H4NHSO2NH2)2 CB31_ AV: 1 NL: 7.43E4
T: ITMS - p ESI E Full ms [150.00-1200.0





NL:
7.43E4
ESI
C4B18H20Co(C2H4NHSO2NH2)2
CB31_ RT: 0.00
AV: 1 T: ITMS - p ESI E Full ms
[150.00-1200.00]

NL:
1.85E5

$C_4B_{18}H_{20}Co(C_2H_4NHSO_2NH_2)_2$:
$C_8B_{18}H_{34}Co_1O_4S_2N_4$
pa Chrg 1