SZEGEDI TUDOMÁNYEGYETEM
GYÓGYSZERANALITIKAI INTÉZET

NMR SPEKTROSZKÓPIAI SPEKTRUMGYŰJTEMÉNY

KÜLÖNÖS TEKINTETTEL AZ ÉLELMISZERIPARBAN ÉS TÁPLÁLÉK-KIEGÉSZÍTŐKBEN ELŐFORDULÓ VEGYÜLETEKRE

Dr. Dombi György

Készült
TÁMOP-4.1.1.C–12/1/KONV–2012-0014
Élelmiszerbiztonság és gasztronómia vonatkozású egyetemi együttműködés, DE–SZTE–EKF–NYME
pályázat keretében

Szeged
2015.

Tartalomjegyzék

I. Bevezetés

A mágneses magrezonancia spektroszkópia (Nuclear Magnetic Resonance Spectroscopy, NMR) az elmúlt ötven évben az analitikai azonosítás, koncentrációmeghatározás, eredetbizonyítás egyik legfontosabb eszközévé vált. Az ^{1}H ill. ^{13}C NMR spektrumok olyan információt hordoz, amit gyakran keverékek esetén is egyszerű értékelni. A modern többdimenziós spektroszkópiai módszerek bonyolult spektrumrészletek értékelését is lehetővé teszik.

Jelen spektrumgyűjtemény az élelmiszeriparban, élelmiszer-kiegészítőkben, -adalékanyagokban és a gyógyszerekben előforduló vegyületek nmr spektrumait tartalmazza. A gyűjtemény segítséget kíván nyújtani a spektrumok értékelésének elsajátításában, valamint referenciaanyag nélküli azonosítást kívánja elősegíteni.

A spektrumok a Gyógyszerésztudományi Kar által működtetett BRUKER AVANCE 500 ill ^{1}H-frekvenciájú nagyfeloldású nmr spektrométerein készültek. A mérési paraméterek a spektrumokon ill. a táblázatos összefoglalóban láthatók.

Az mérésekért és összeállításért köszönetemet fejezem ki Dr. Forgó Péternek. Dr. Hetényi Anasztáziának, Dr. Szakonyi gerdának és Dr. Kupihár Zoltánnak. A pénzügyi feltételeket a TÁMOP-4.1.1.C–12/1/KONV–2012-0014 pályázat biztosította.

Szeged, 2011. június 05.

Dr. Dombi György
egyetemi tanár

II. Rövidítések jegyzéke

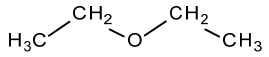
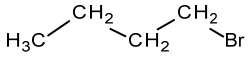
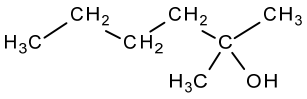
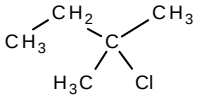
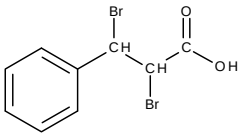
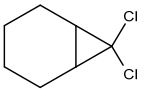
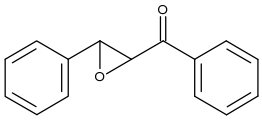
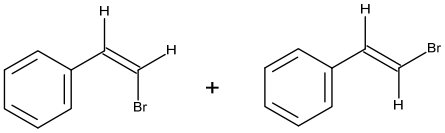
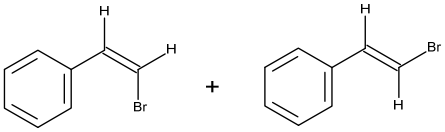
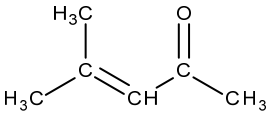
Oldószerek

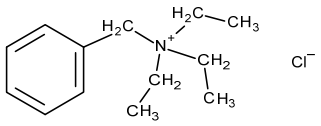
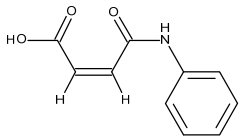
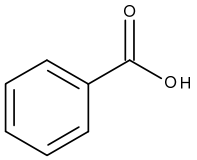
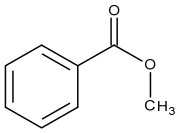
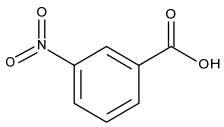
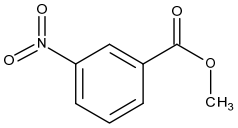
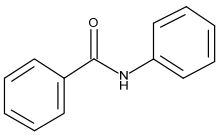
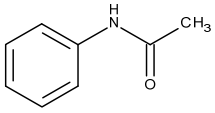
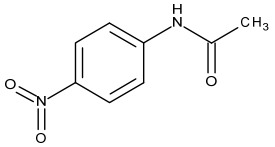
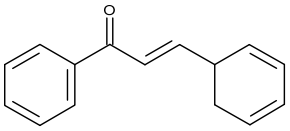
$CDCl_3$	Deutero-kloroform
DMSO	Hexadeutero-dimetilszulfoxid [$D_3C–S(O)–CD_3$]
MeOD	Perdeutero-metanol [$D_3C–OD$]

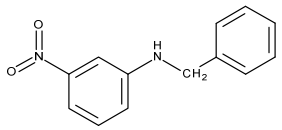
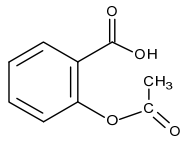
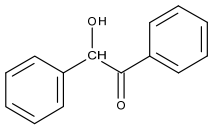
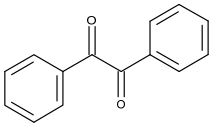
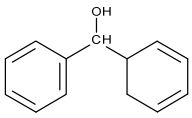
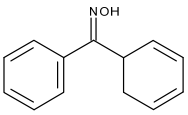
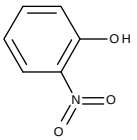
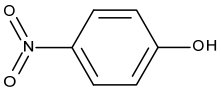
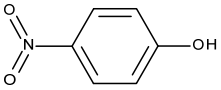
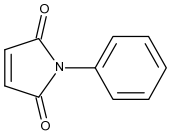
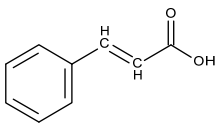
Spektrumok mérési jellemzői

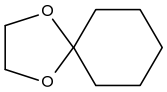
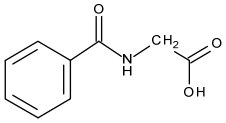
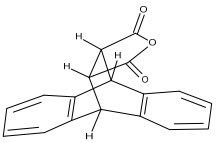
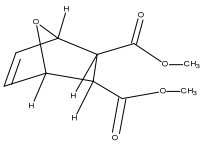
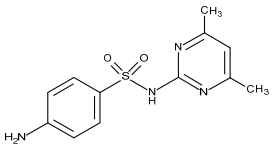
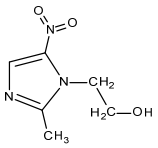
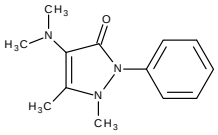
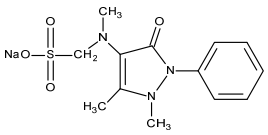
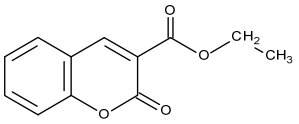
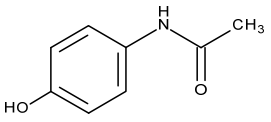
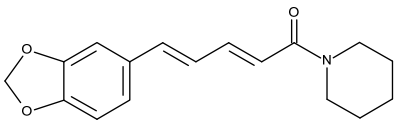
^1H	1-es tömegszámú hidrogén (proton) rezonancia spektrum.
^{13}C	13-as tömegszámú szénizotóp rezonancia spektrum. (1,1%-os természetes előfordulás és szélessávú ^1H lecsatolás mellett).
COSY	Correlation Spectroscopy: Olyan kétdimenziós nmr méréstechnika, amikor az azonos magok közötti csatolást térképezzük fel. (A magnetizáció átadás a J-csatoláson keresztül történik.)
JMOD	J-modulált spektrum: a ^{13}C jelek multiplicitását (metil: kvartett, metilén: triplett, metin: dublett, kvaterner: singlett) a teljesen ^1H lecsatolt ^{13}C spektrum jeleinek fázisában tükrözzük vissza SEFT (Spin-Echo Fourier Transform) technikát használva. A spektrumban a metil és metin jelek (páratlan hidrogénszám) pozitív, a metilén és kvaterner szenek (páros és 0 hidrogén) negatív fázisú jelek adnak.
HSQC	Heteronuclear Single Quantum Coherence: kétdimenziós ^1H,^{13}C korrelációt mutat. A spektrumban az F1 dimenzióban nincs szélesedés a homonukleáris ^1H-^1H csatolás miatt.A keresztcsúcsok az összetartozó ^{13}C és ^1H jeleket mutatják.
HMBC	Heteronuclear Multiple Bond Correlation: kétdimenziós ^1H,^{13}C korrelációt mutat $^2J(C,H)$ és $^3J(C,H)$ indirekt csatolás alapján
NOESY	Nuclear Overhauser Enhancement SpectroscopY: a NOE differencia spektrum kétdimenziós változata és olyan jelek között mutat korrelációr, melyet térbeli közelségben lévő magok közötti dipoláris kereszt-relaxáció okoz.
ROESY	Rotating frame Overhauser Enhancement SpectroscopY: kétdimenziós méréstechnika a NOE mérésére ún. spinlock feltételek mellett. Kiválóan alkalmas 1000-3000-es molekulatömegű minták mérésére, mert ilyenkor a NOESY keresztcsúcsok eltűnhetnek.

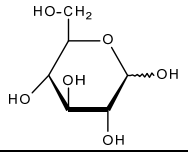
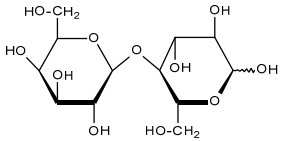
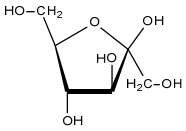
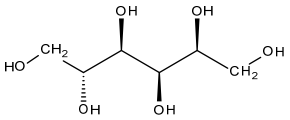
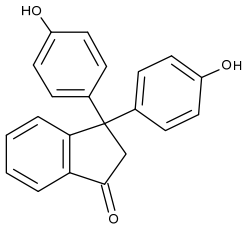
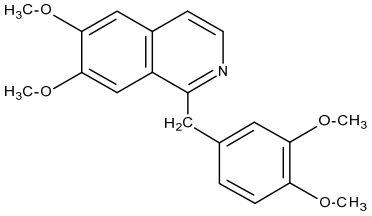
III. Összefoglaló táblázat

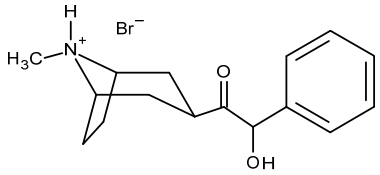
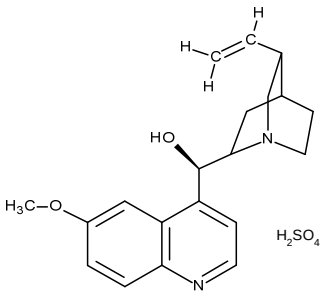
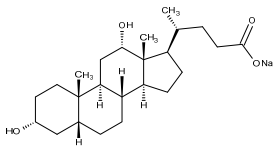
No.	Név (Oldószer)	Képlet	Spektrum
1.	Dietil-éter ($CDCl_3$)		^{1}H-NMR
2.			^{13}C JMOD
3.	1-Bróm-bután ($CDCl_3$)		^{1}H-NMR
4.			^{13}C JMOD
5.	2-Metil-2-hexanol (DMSO)		^{1}H-NMR
6.			^{13}C JMOD
7.	2-Klór-2-metil-bután ($CDCl_3$)		^{1}H-NMR
8.			^{13}C JMOD
9.	2,3-Dibróm-3-fenil propánsav (DMSO)		^{1}H-NMR
10.			^{13}C JMOD
11.	7,7-Diklór-biciklo-[4.1.0]-heptán ($CDCl_3$)		^{1}H-NMR
12.			^{13}C JMOD
13.	[2*RS*,3*SR*]-2,3-Epoxi-1,3-difenil-propán-1-on (DMSO)		^{1}H-NMR
14.			^{13}C JMOD
15.	Bróm-sztirol (*Z*-91%, *E*-9 %) ($CDCl_3$)		^{1}H-NMR
16.			^{13}C JMOD
17.	Bróm-sztirol *(Z*-25%, *E*-75 %) ($CDCl_3$)		^{1}H-NMR
18.			^{13}C JMOD
19.	Mezitil-oxid ($CDCl_3$)		^{1}H-NMR
20.			^{13}C JMOD

No.	Név (Oldószer)	Képlet	Spektrum
21.	Benzil-trietil-ammónium-klorid (CD_3OD)		^{1}H-NMR
22.			^{13}C-JMOD
23.	Maleanilidsav, maleinsav-monoanikid (DMSO)		^{1}H-NMR
24.			^{13}C-JMOD
25.	Benzoesav ($CDCl_3$)		^{1}H-NMR
26.			^{13}C JMOD
27.	Benzoesav (DMSO)		^{1}H-NMR
28.			^{13}C-JMOD
29.	Benzoesav-metilészter, metil-benzoát ($CDCl_3$)		^{1}H-NMR
30.			^{13}C-JMOD
31.	3-Nitro-benzoesav (DMSO)		^{1}H-NMR
32.			^{13}C-JMOD
33.	3-Nitro-benzoesav-metilészter ($CDCl_3$)		^{1}H-NMR
34.			^{13}C-JMOD
35.	Benzanilid (DMSO)		^{1}H-NMR
36.			^{13}C-JMOD
37.	Acetanilid ($CDCl_3$)		^{1}H-NMR
38.			^{13}C JMOD
39.	4-Nitro-acetanilid (DMSO)		^{1}H-NMR
40.			^{13}C-JMOD
41.	Benzilidén-acetofenon ($CDCl_3$)		^{1}H-NMR
42.			^{13}C-JMOD

No.	Név (Oldószer)	Képlet	Spektrum
43.	*N*-Benzil-3-nitro-anilin ($CDCl_3$)		^{1}H-NMR
44.			^{13}C-JMOD
45.	Acetil-szalicilsav (DMSO)		^{1}H-NMR
46.			^{13}C-JMOD
47.	Benzoin (DMSO)		^{1}H-NMR
48.			^{13}C-JMOD
49.	Benzil ($CDCl_3$)		^{1}H-NMR
50.			^{13}C-JMOD
51.	Benzhidrol (DMSO)		^{1}H-NMR
52.			^{13}C-JMOD
53.	Benzofenon-oxim (DMSO)		^{1}H-NMR
54.			^{13}C-JMOD
55.	*orto*-Nitro-fenol ($CDCl_3$)		^{1}H-NMR
56.			^{13}C-JMOD
57.	*para*-Nitro-fenol ($CDCl_3$)		^{1}H-NMR
58.			^{13}C-JMOD
59.	*para*-Nitro-fenol ($CDCl_3$)		^{1}H-NMR
60.			^{13}C-JMOD
61.	*N*-Fenil-maleimid (DMSO)		^{1}H-NMR
62.			^{13}C-JMOD
63.	Fahéjsav ($CDCl_3$)		^{1}H-NMR
64.			^{13}C-JMOD

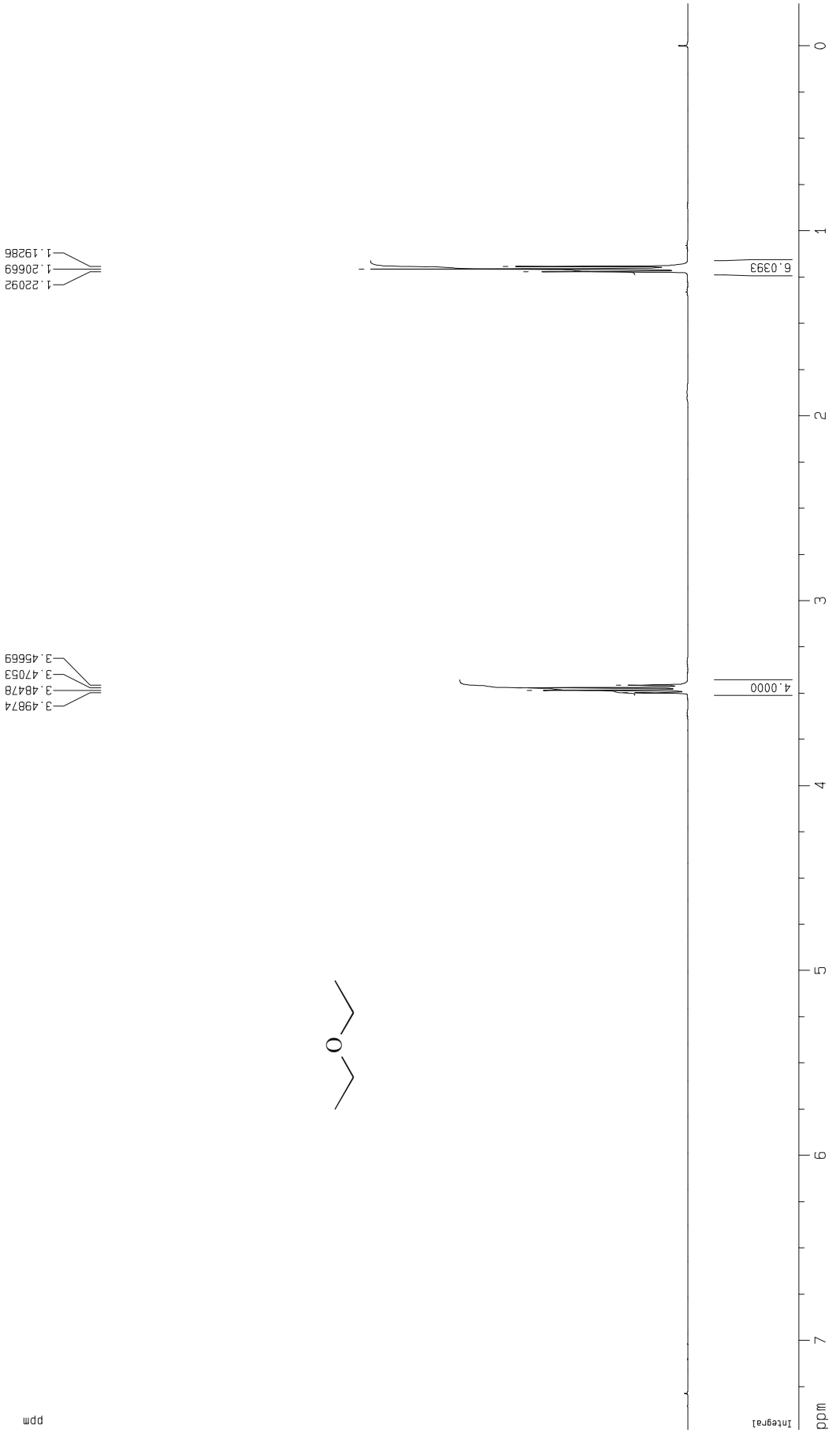
No.	Név (Oldószer)	Képlet	Spektrum
65.	1,4-Dioxaspiro[4,5]-dekán ($CDCl_3$)		^{1}H-NMR
66.			^{13}C-JMOD
67.	Hippursav, *N*-Benzoil-glicin (DMSO)		^{1}H-NMR
68.			^{13}C-JMOD
69.	*cisz*-9,10-Dihidro-9,10-etano-antracen-11,12-dikarbonsav-anhidrid ($CDCl_3$)		^{1}H-NMR
70.			^{13}C-JMOD
71.	7-Oxabiciklo[2.2.1]hept-5-én-2exo,3exo-disav-dimetil-észter ($CDCl_3$)		^{1}H-NMR
72.			^{13}C-JMOD
73.	Sulfadimidin (DMSO)		^{1}H-NMR
74.			^{13}C-JMOD
75.	Metrodinazol (DMSO)		^{1}H-NMR
76.			^{13}C-JMOD
77.	Aminofenazon (DMSO)		^{1}H-NMR
78.			^{13}C-JMOD
79.	Noramino-phenazon (CD_3OD)		^{1}H-NMR
80.			^{13}C-JMOD
81.	3-Karbetoxi-kumarin ($CDCl_3$)		^{1}H-NMR
82.			^{13}C-JMOD
83.	Paracetamol (DMSO)		^{1}H-NMR
84.			^{13}C-JMOD
85.	Piperin (DMSO)		^{1}H-NMR
86.			^{13}C-JMOD

No.	Név (Oldószer)	Képlet	Spektrum
87.	D-Glükóz (DMSO)		^{1}H-NMR
88.			^{13}C-JMOD
89.	Laktóz, ß-*D*-Galaktopiranozil-(1→4)-*D*-glükopiranóz (DMSO)		^{1}H-NMR
90.			^{13}C-JMOD
91.	D-(-) Fruktóz (DMSO)		^{1}H-NMR
92.			^{13}C-JMOD
93.	Szorbitol, hexán-1,2,3,4,5,6-hexol (DMSO)		^{1}H-NMR
94.			^{13}C-JMOD
95.	Fenolftalein (DMSO)		^{1}H
96.			^{13}C-JMOD
97.			^{1}H-^{1}H COSY
98.			^{1}H-^{13}C HSQC
99.			^{1}H-^{13}C HMBC
100.			^{1}H-^{1}H ROESY
101.	Papaverin ($CDCl_3$)		^{1}H-NMR
102.			^{13}C-JMOD
103.			^{1}H-^{1}H COSY
104.			^{1}H-^{13}C HSQC
105.			^{1}H-^{13}C HMBC
106.			^{1}H-^{1}H NOESY

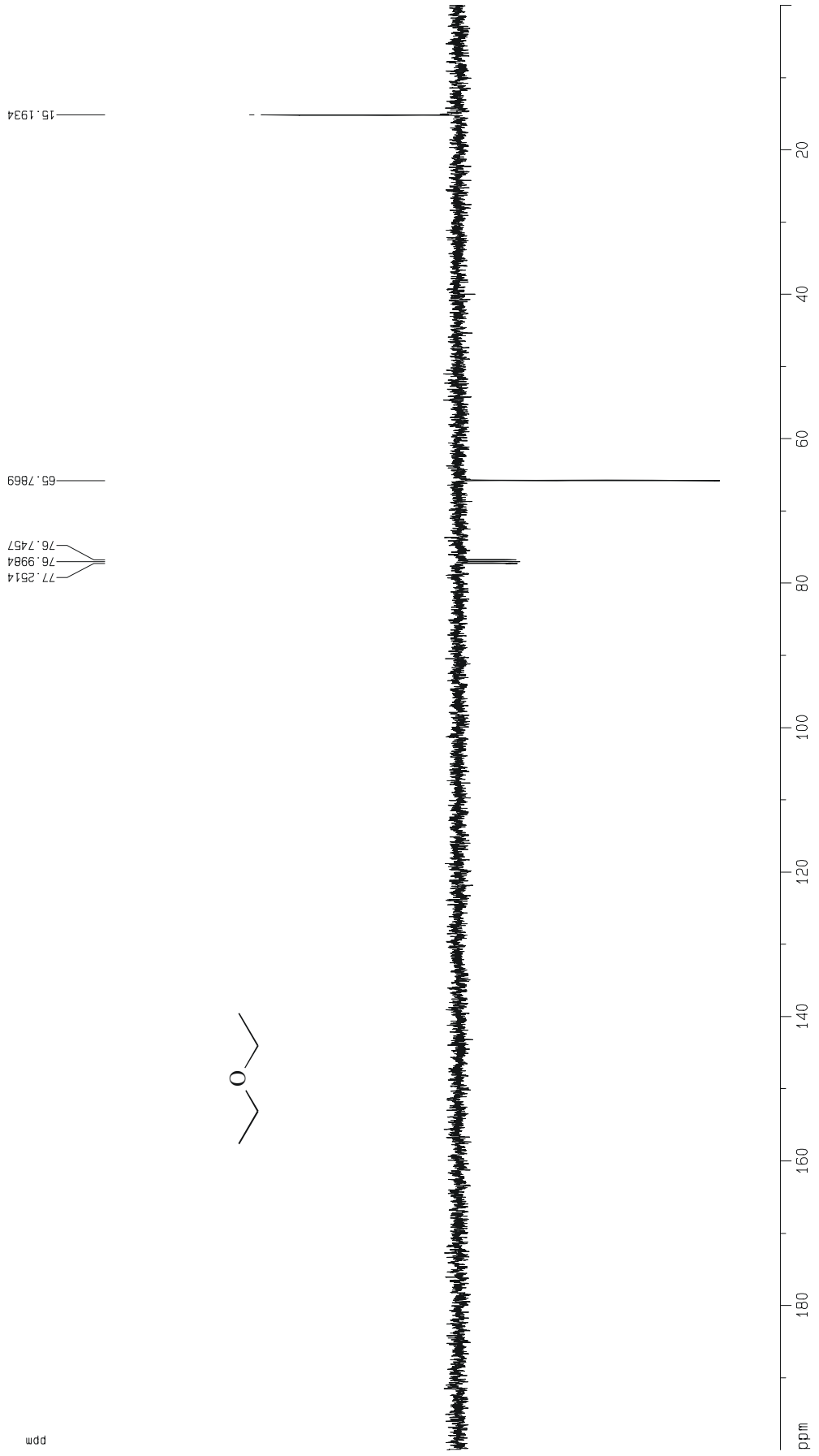
No.	Név (Oldószer)	Képlet	Spektrum
107.	Metil-homatropinium-bromid (DMSO)		^{1}H-NMR
108.			^{13}C-JMOD
109.			^{1}H-^{1}H COSY
110.			^{1}H-^{13}C HSQC
111.			^{1}H-^{13}C HMBC
112.			^{1}H-^{1}H NOESY
113.			^{1}H-^{1}H ROESY
114.	Kinin-szulfát (CD_3OD)		^{1}H-NMR
115.			^{13}C-JMOD
116.			^{1}H-^{1}H COSY
117.			^{1}H-^{13}C HSQC
118.			^{1}H-^{13}C HMBC
119.			^{1}H-^{1}H ROESY
120.	3α,12α-Dihidroxi-kólsav Na só, (CD_3OD)		^{1}H-NMR
121.			^{13}C-JMOD

IV. Spektrumgyűjtemény

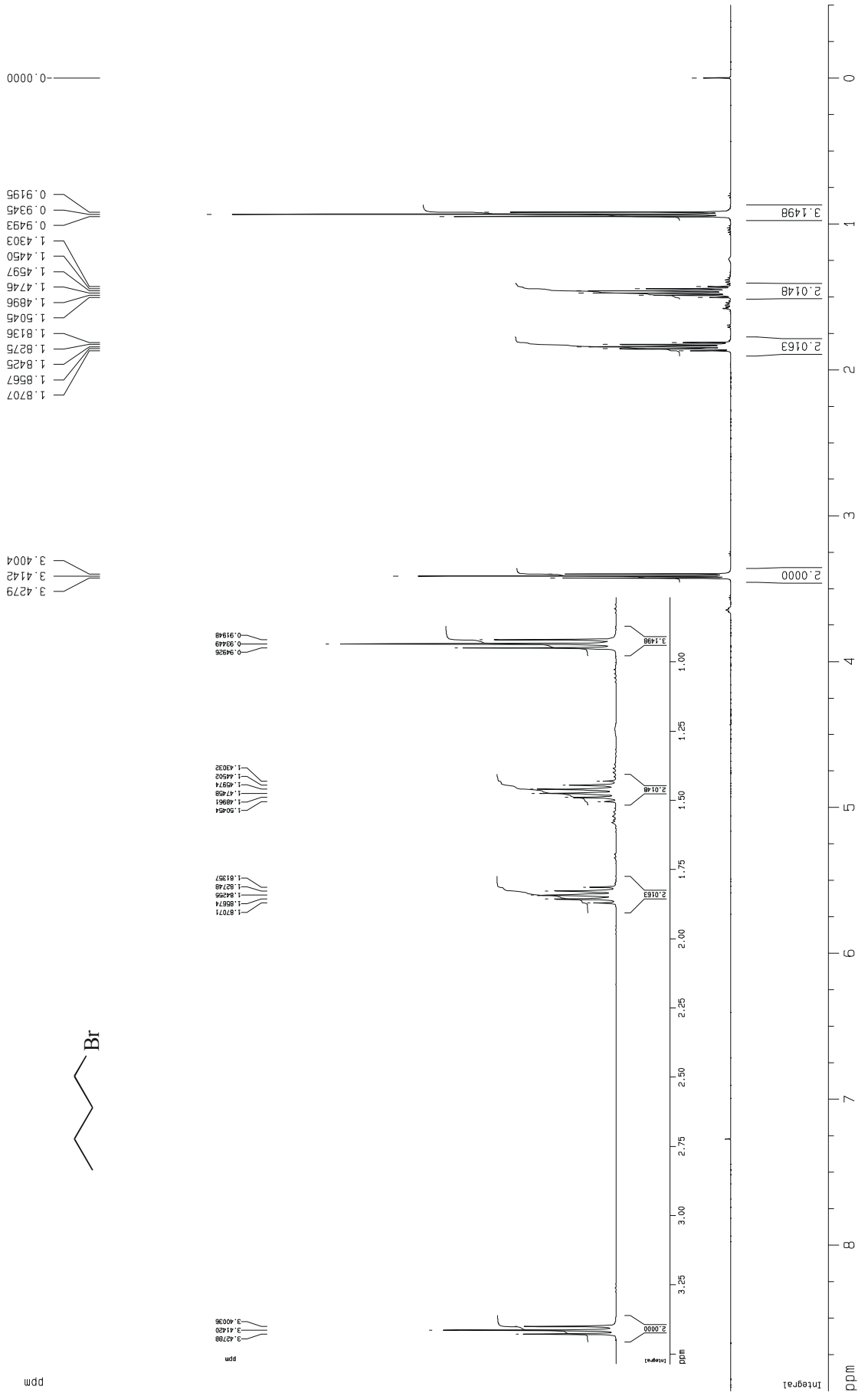
1. Dietil-éter ^{1}H-NMR spektrum ($CDCl_3$)



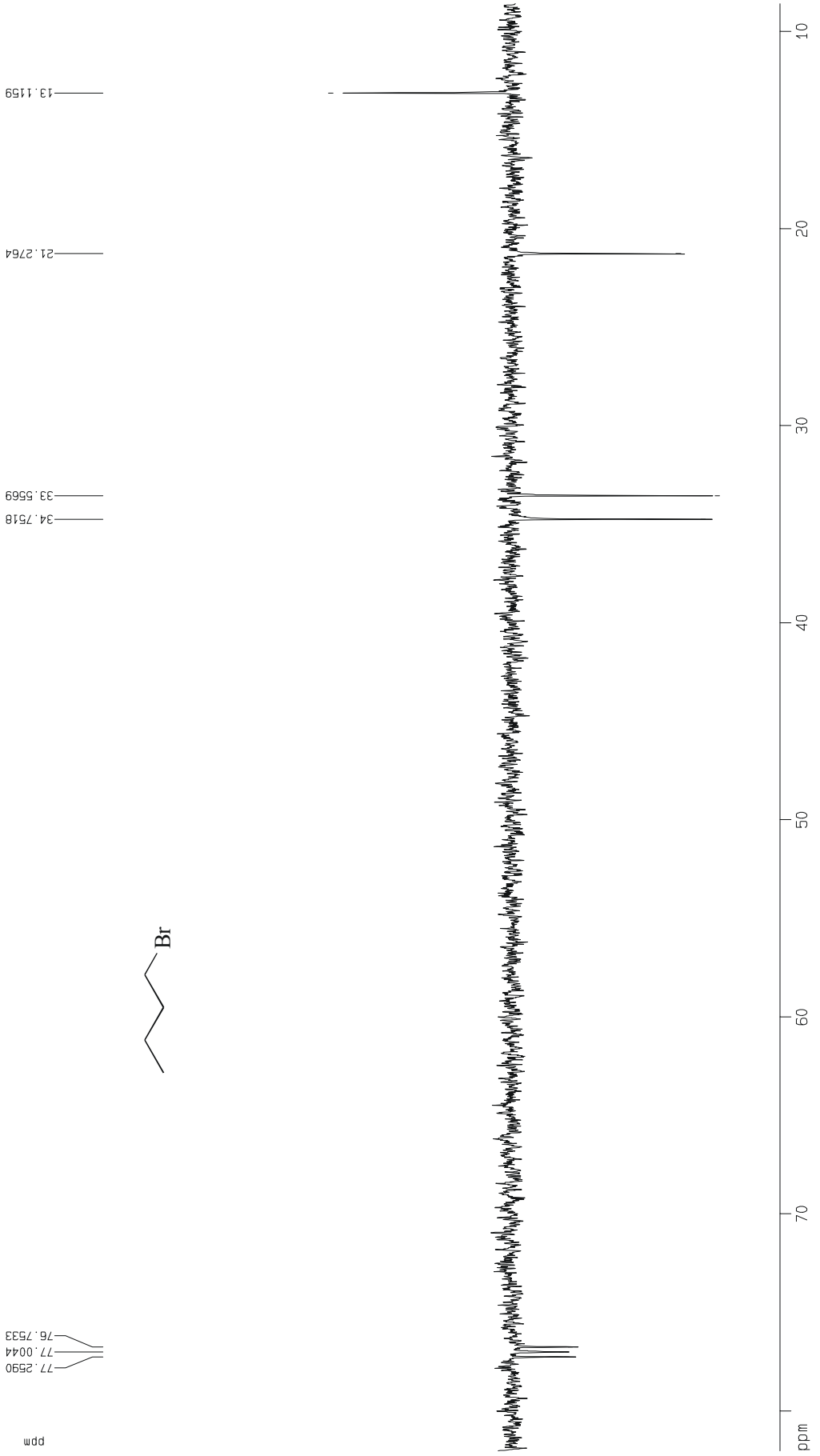
2. Dietil-éter ^{13}C-JMOD NMR pektrum ($CDCl_3$)



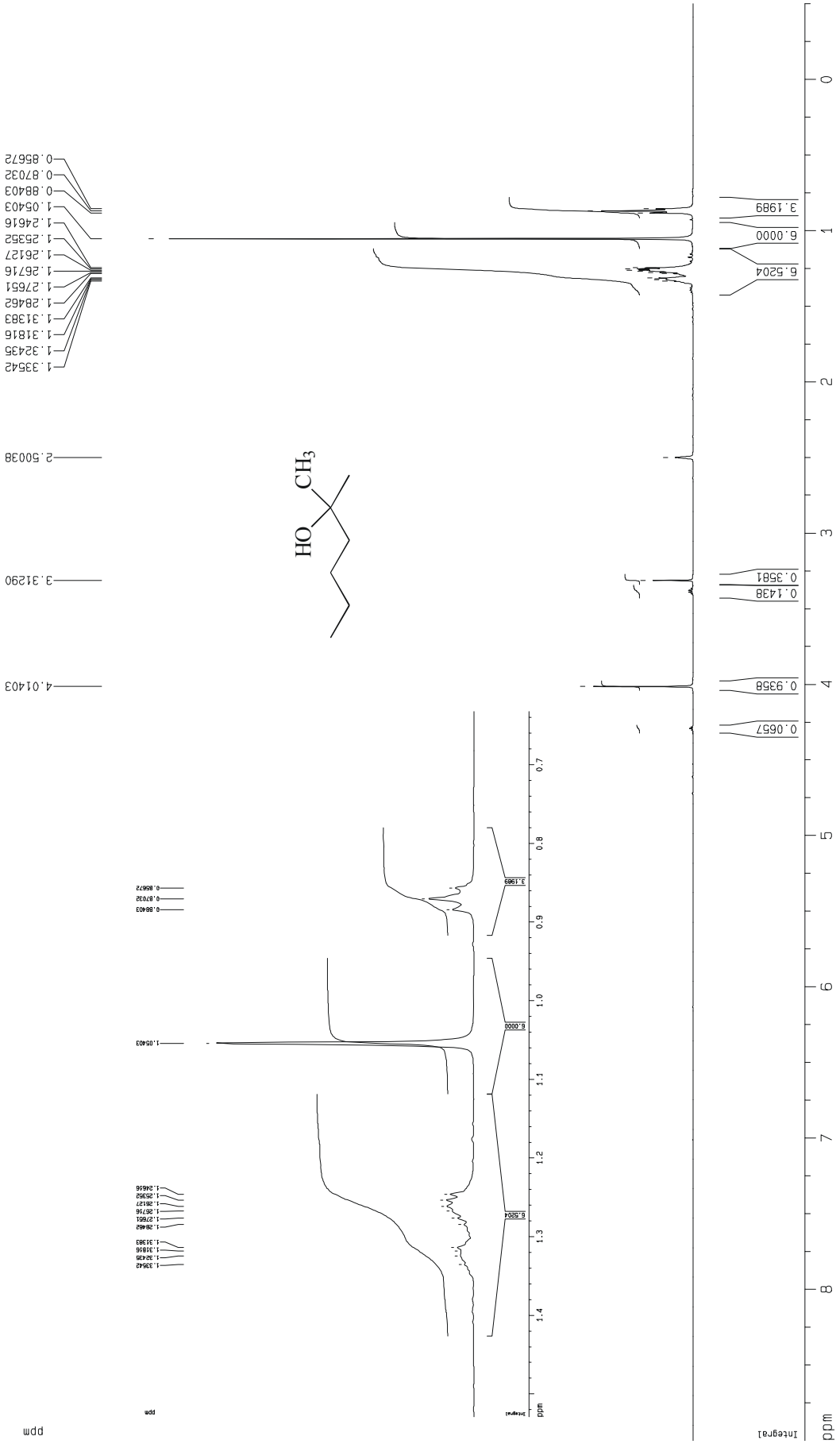
3. 1-Bróm-bután ^{1}H-NMR spektrum ($CDCl_3$)



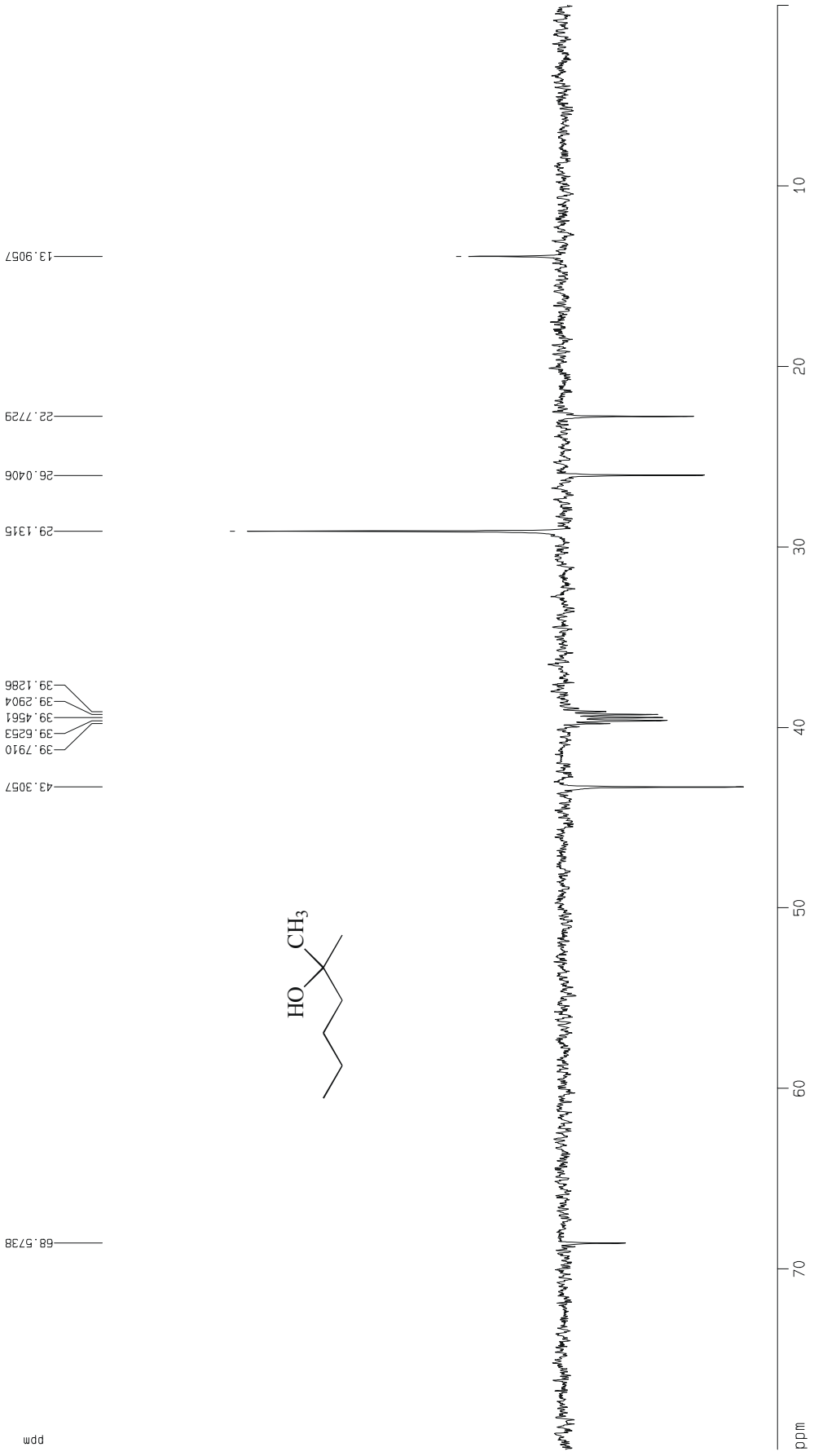
4. 1-Bróm-bután ^{13}C-JMOD NMR spektrum ($CDCl_3$)



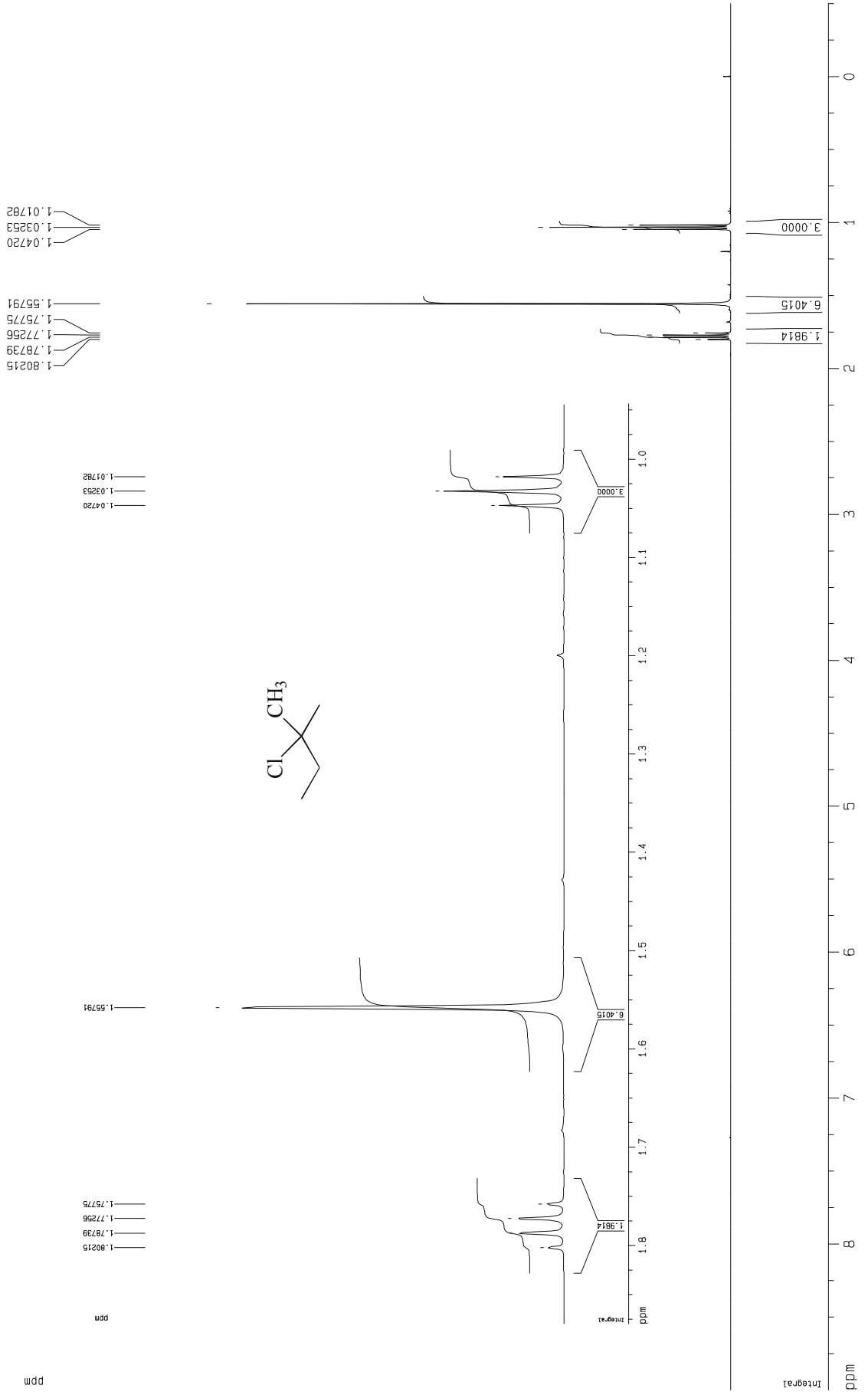
5. 2-Metil-2-hexanol ^{1}H-NMR spektrum (DMSO)



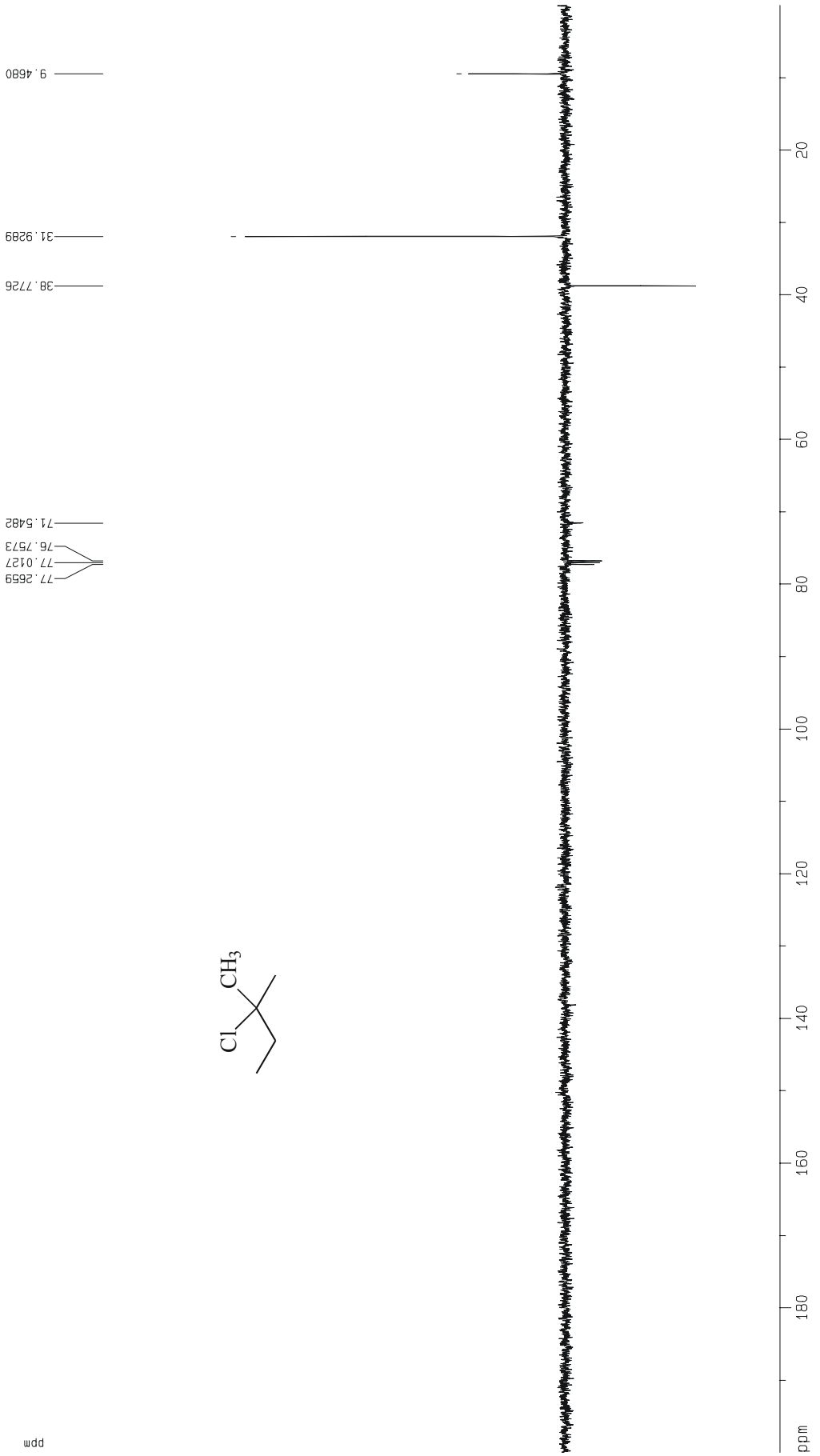
6. 2-Metil-2-hexanol ^{13}C-JMOD NMR spektrum (DMSO)



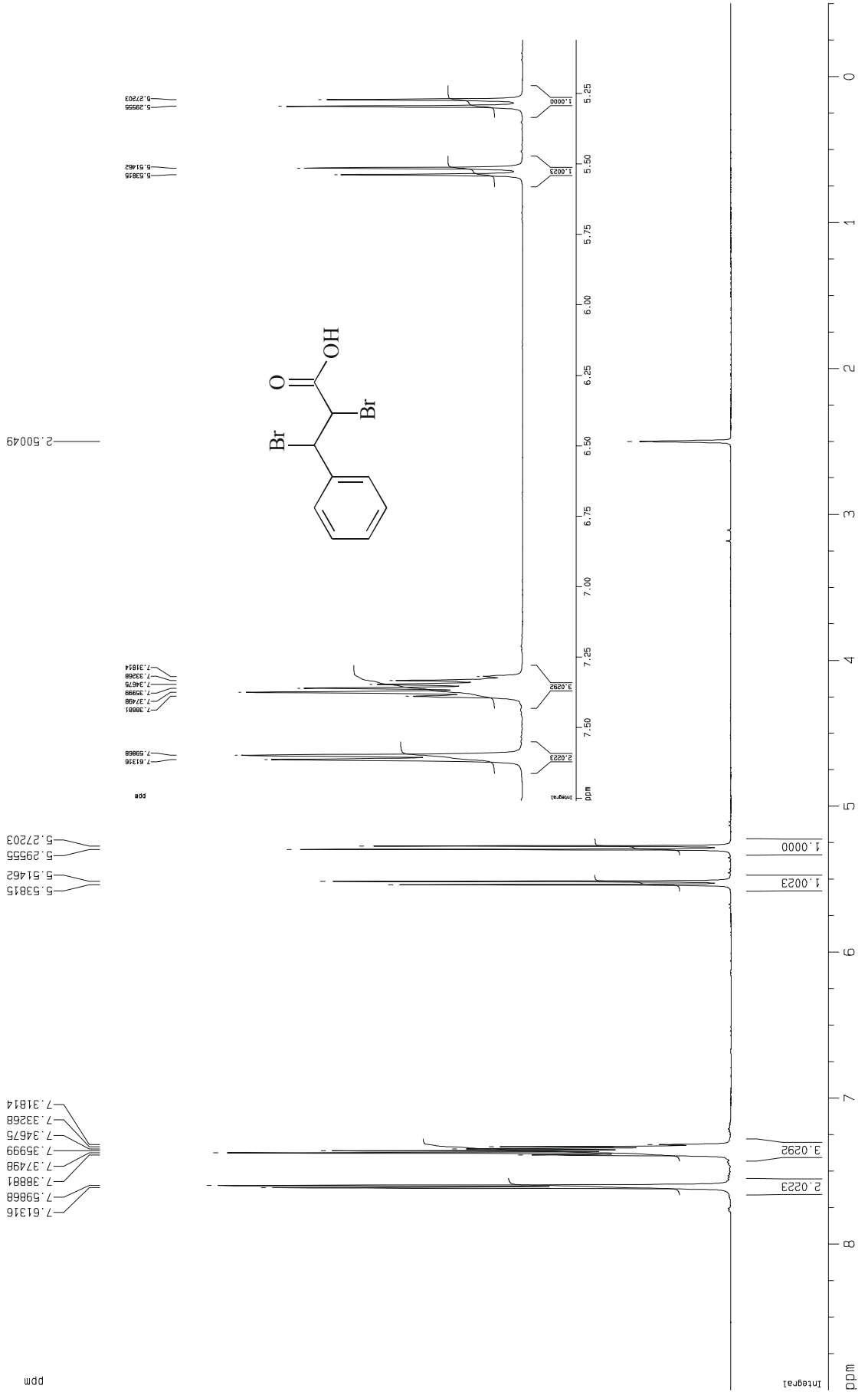
7. 2-Klór-2-metil-bután ^{1}H-NMR spektrum ($CDCl_3$)



8. 2-Klór-2-metil-bután ^{13}C-JMOD spektrum ($CDCl_3$)

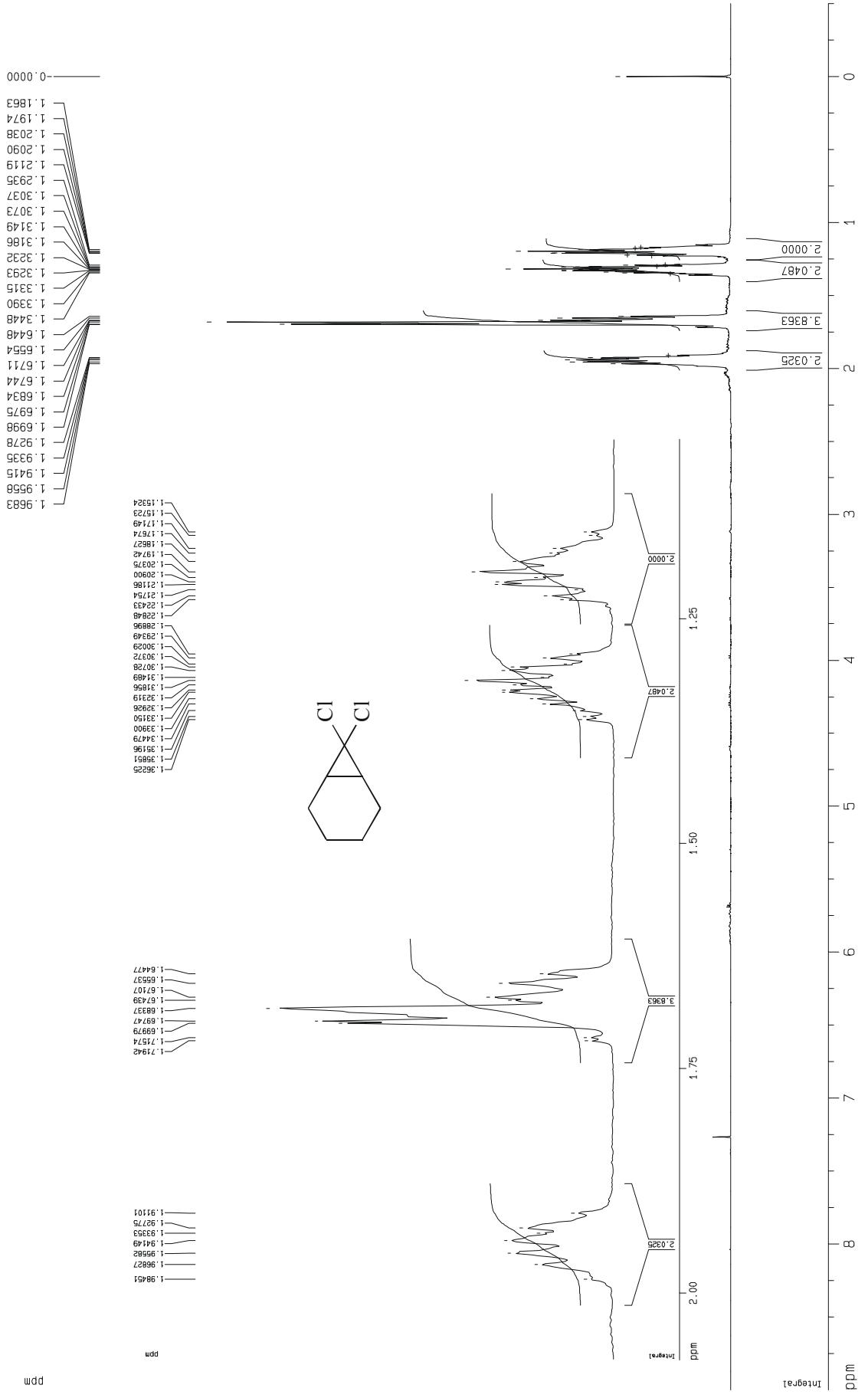


9. 2,3-Dibróm-fenil propánsav ^{1}H-NMR spektrum (DMSO)

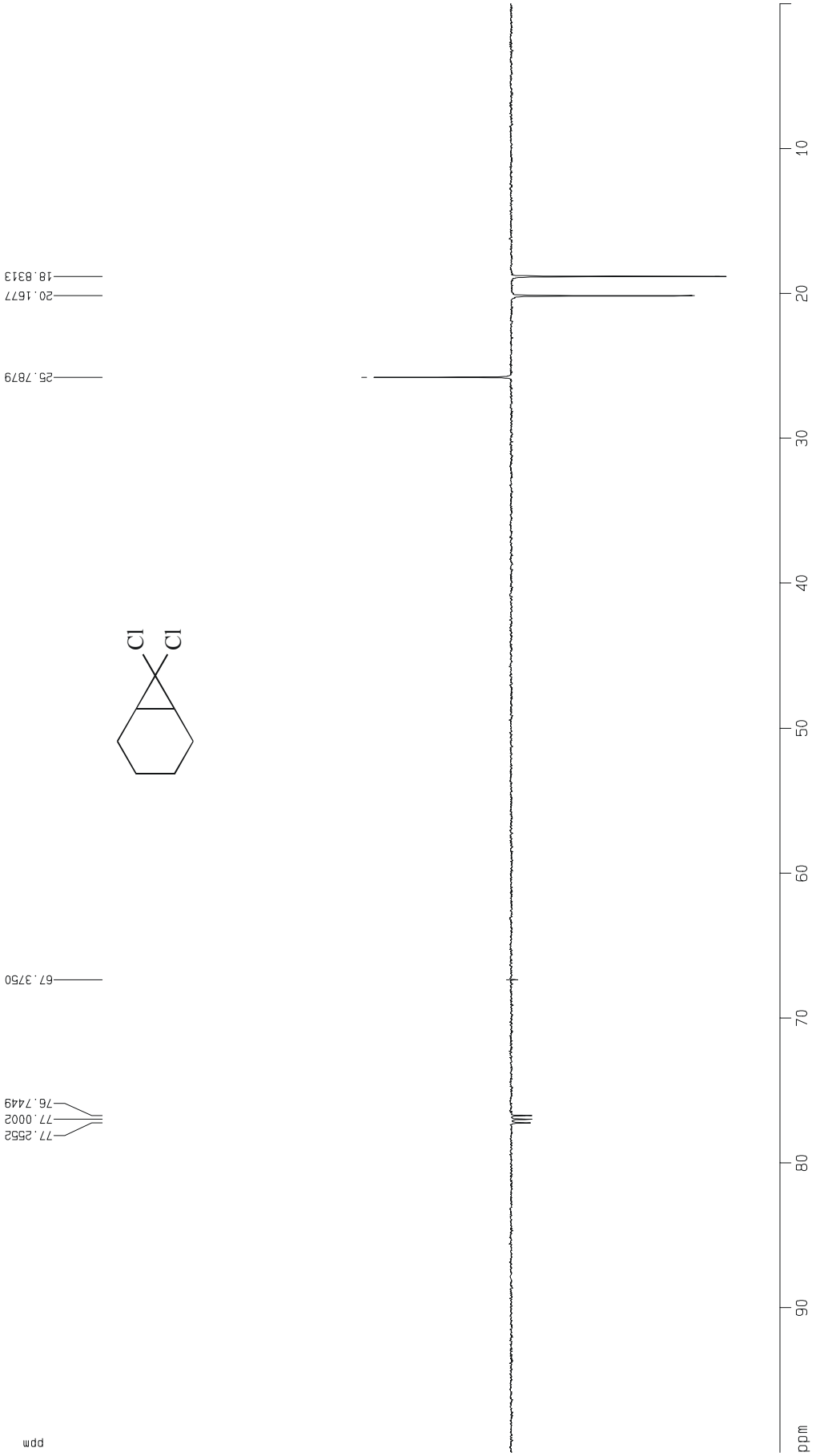


10. 2,3-Dibróm-fenil propánsav ^{13}C-JMOD NMR spektrum (DMSO)

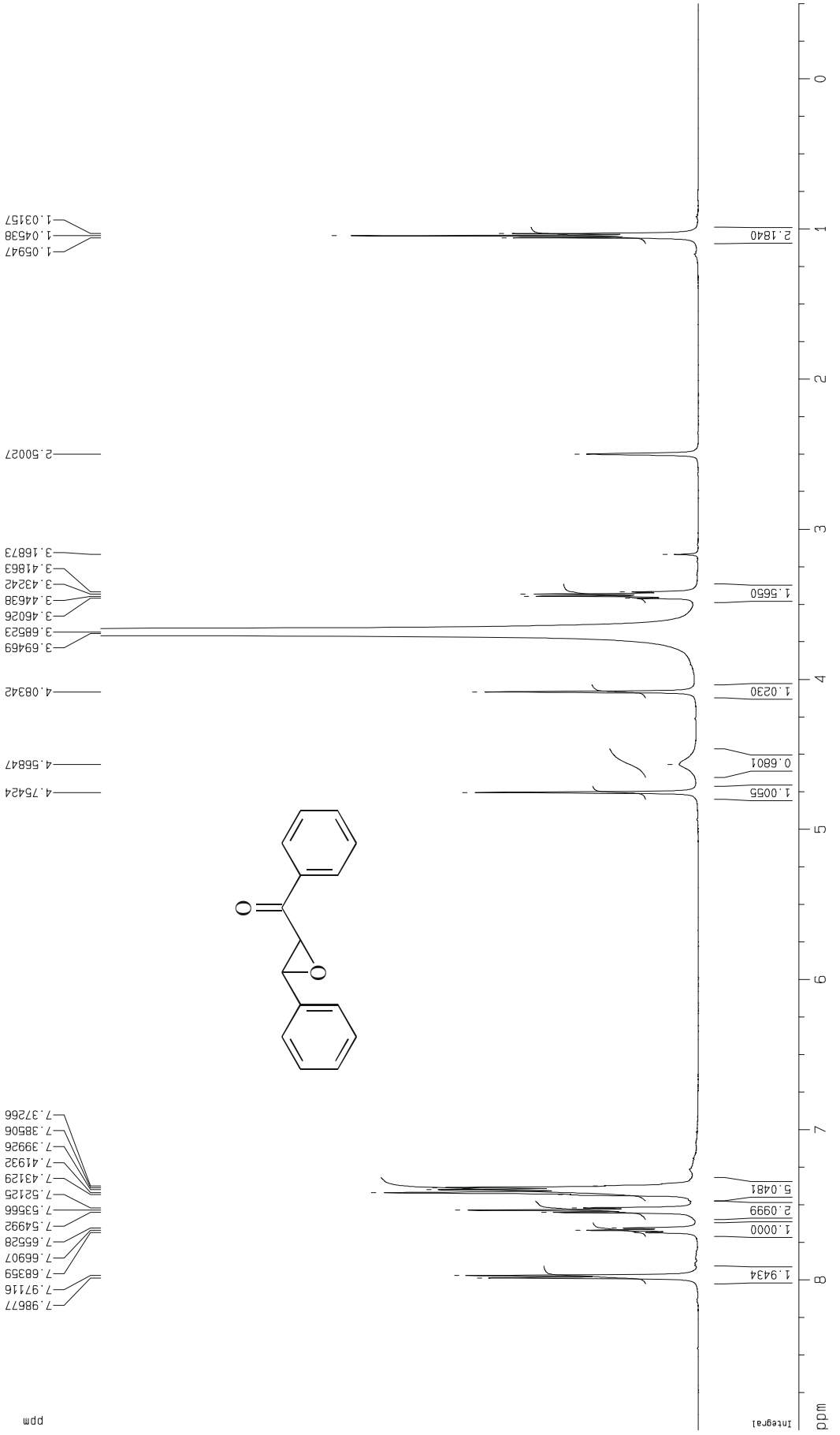
11. 7,7-Diklór-biciklo[4.1.0]heptán ^{1}H-NMR spektrum ($CDCl_3$)



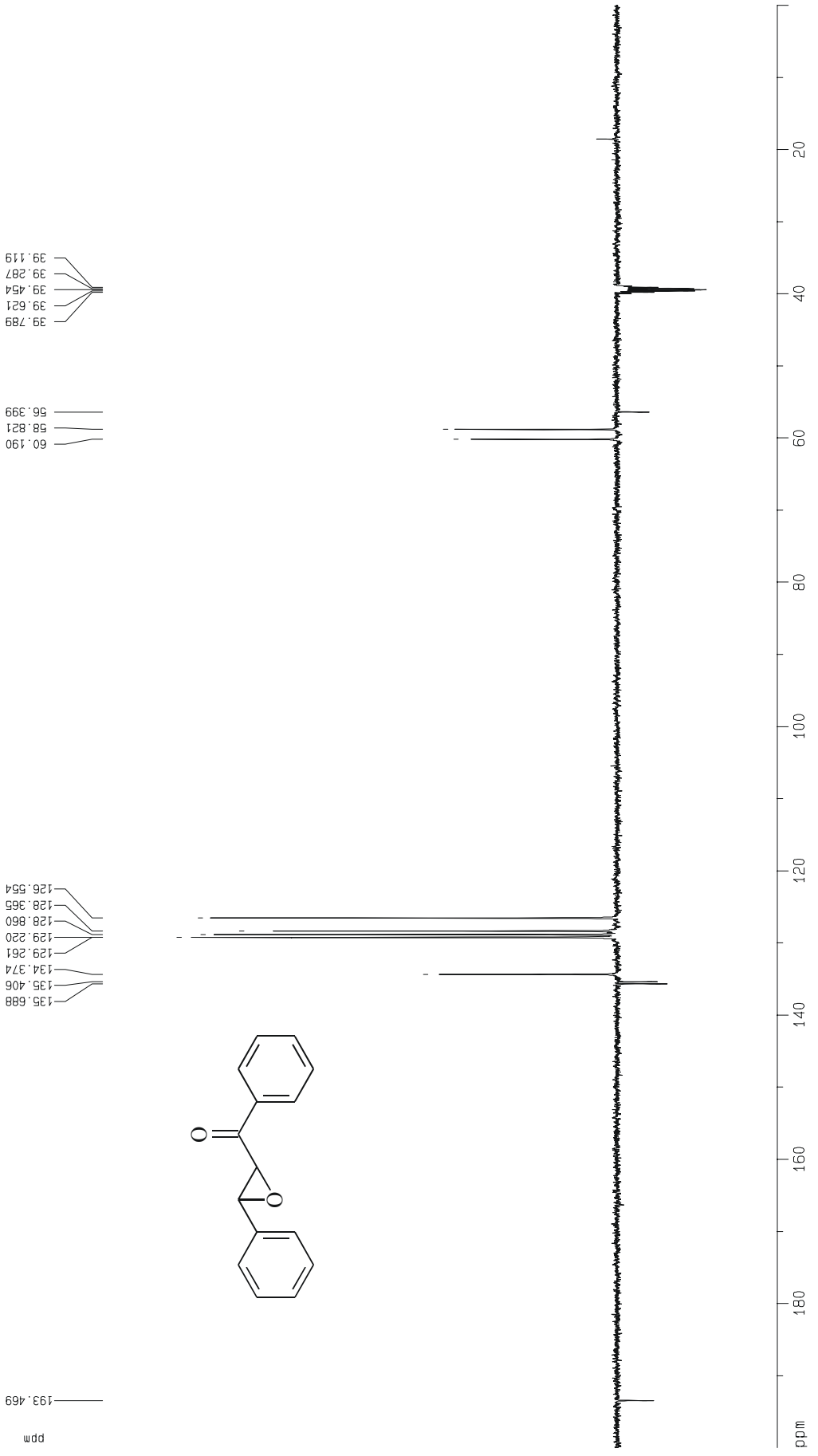
12. 7,7-Diklór-biciklo[4.1.0]heptán ^{13}C-JMOD NMR spektrum ($CDCl_3$)



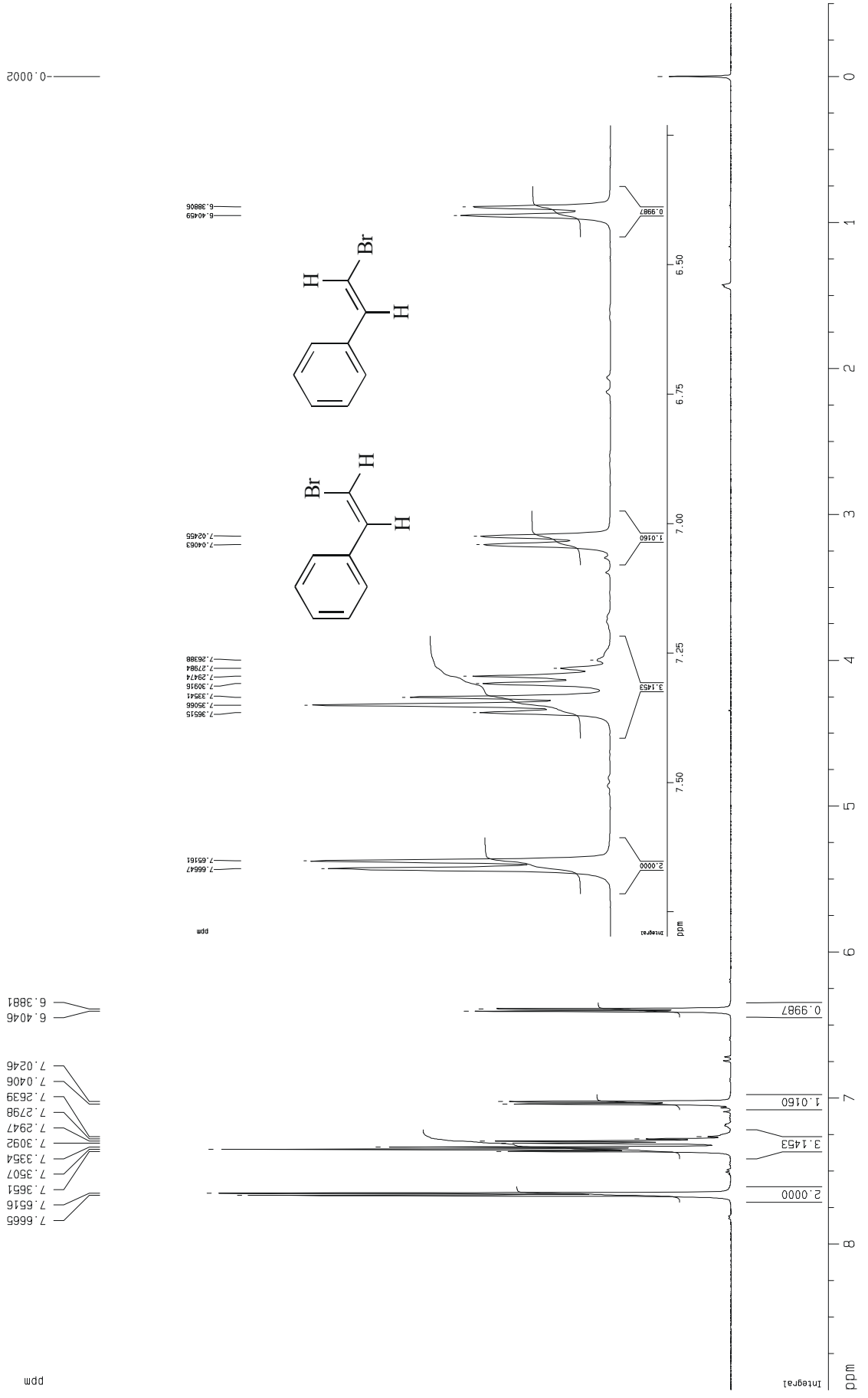
13. [2*RS*,3*SR*]-2,3-Epoxi-1,3-difenil-propán-1-on ^{1}H-NMR spektrum (DMSO)



14. [2*RS*,3*SR*]-2,3-Epoxi-1,3-difenil-propán-1-on ^{13}C-JMOD NMR spektrum (DMSO)

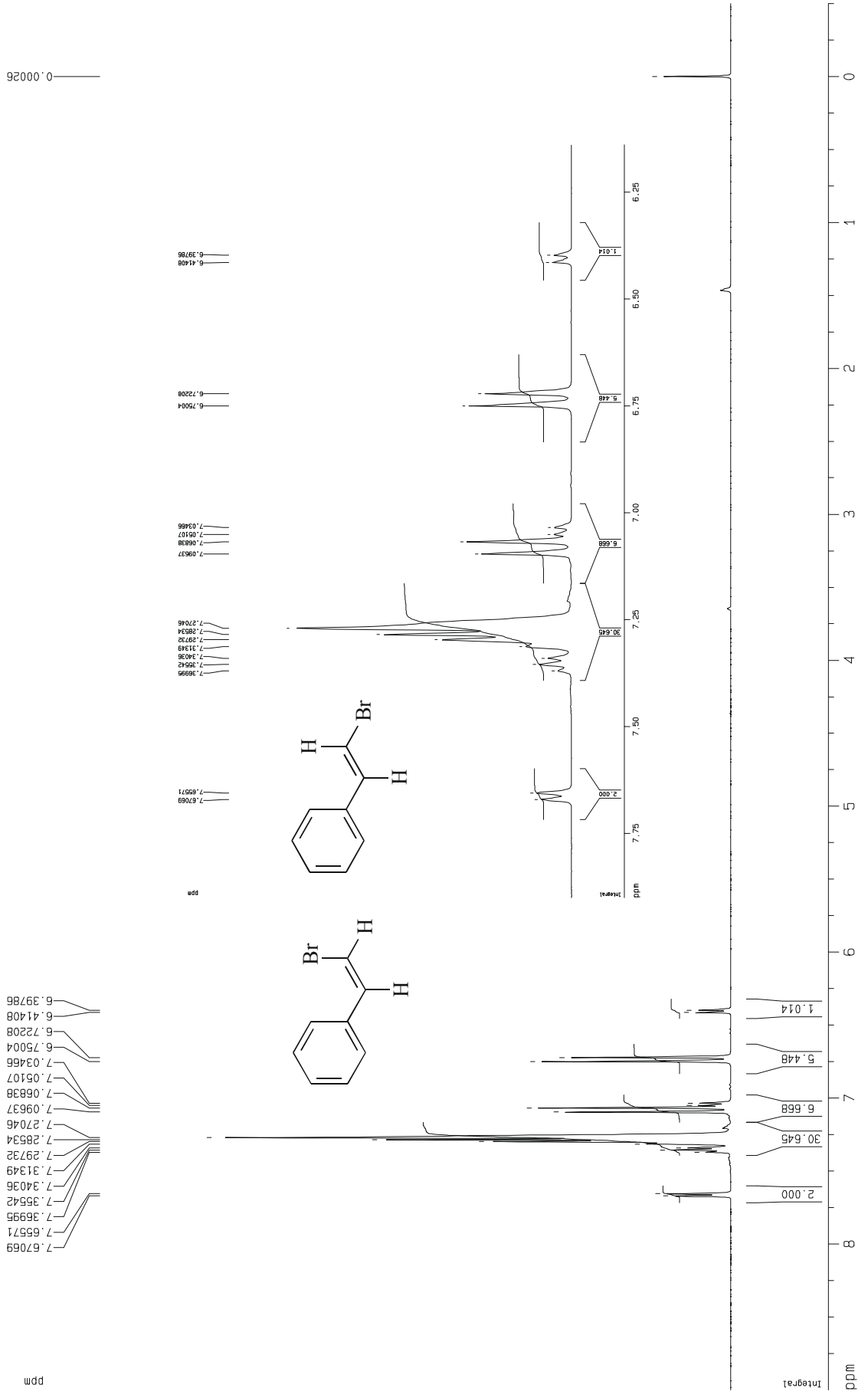


15. Bróm-sztirol (Z-91%, E-9 %) ^{1}H-NMR spektrum ($CDCl_3$)

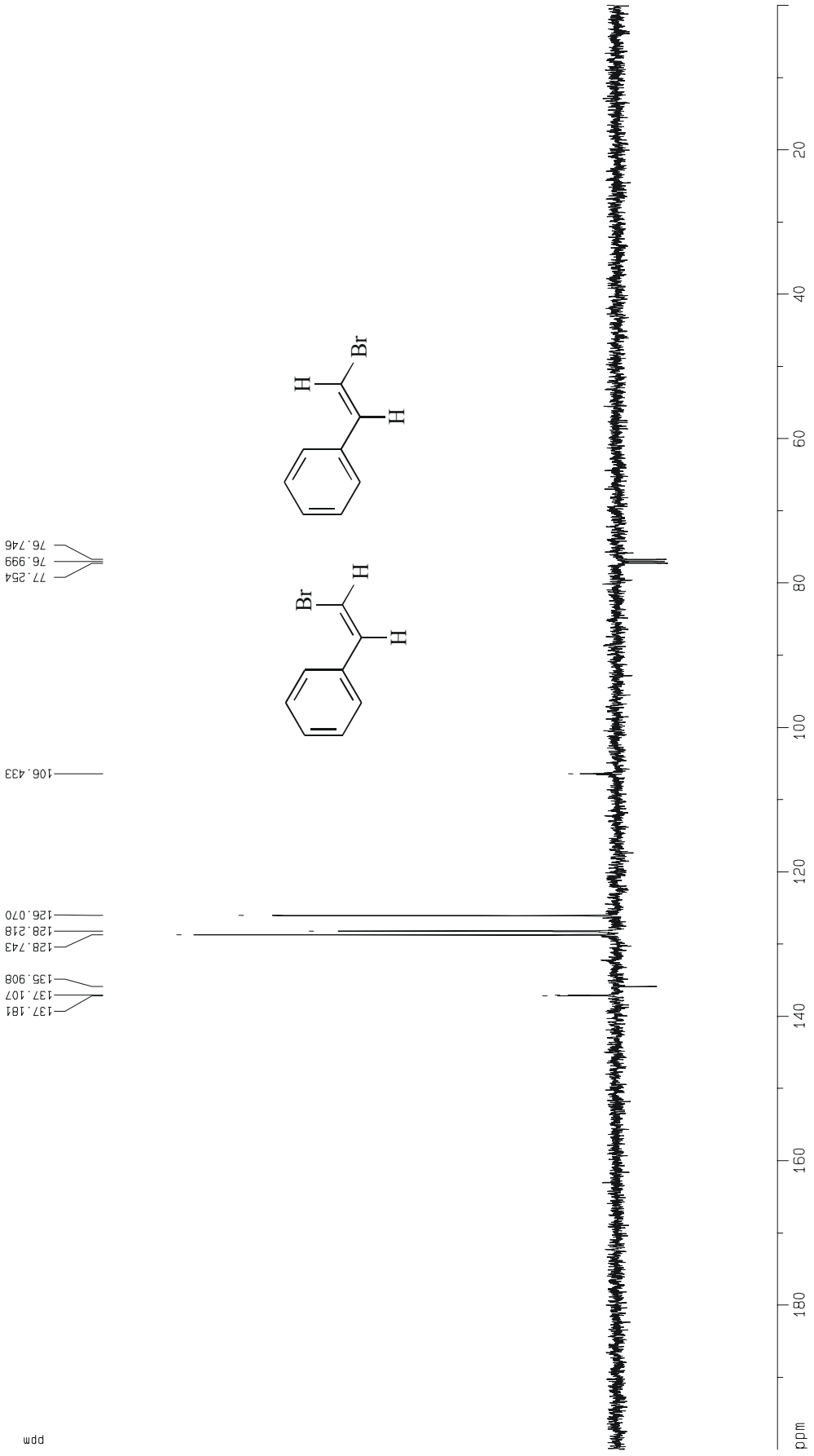


16. Bróm-sztirol (Z-91%, E-9 %) ^{13}C-JMOD NMR spektrum ($CDCl_3$)

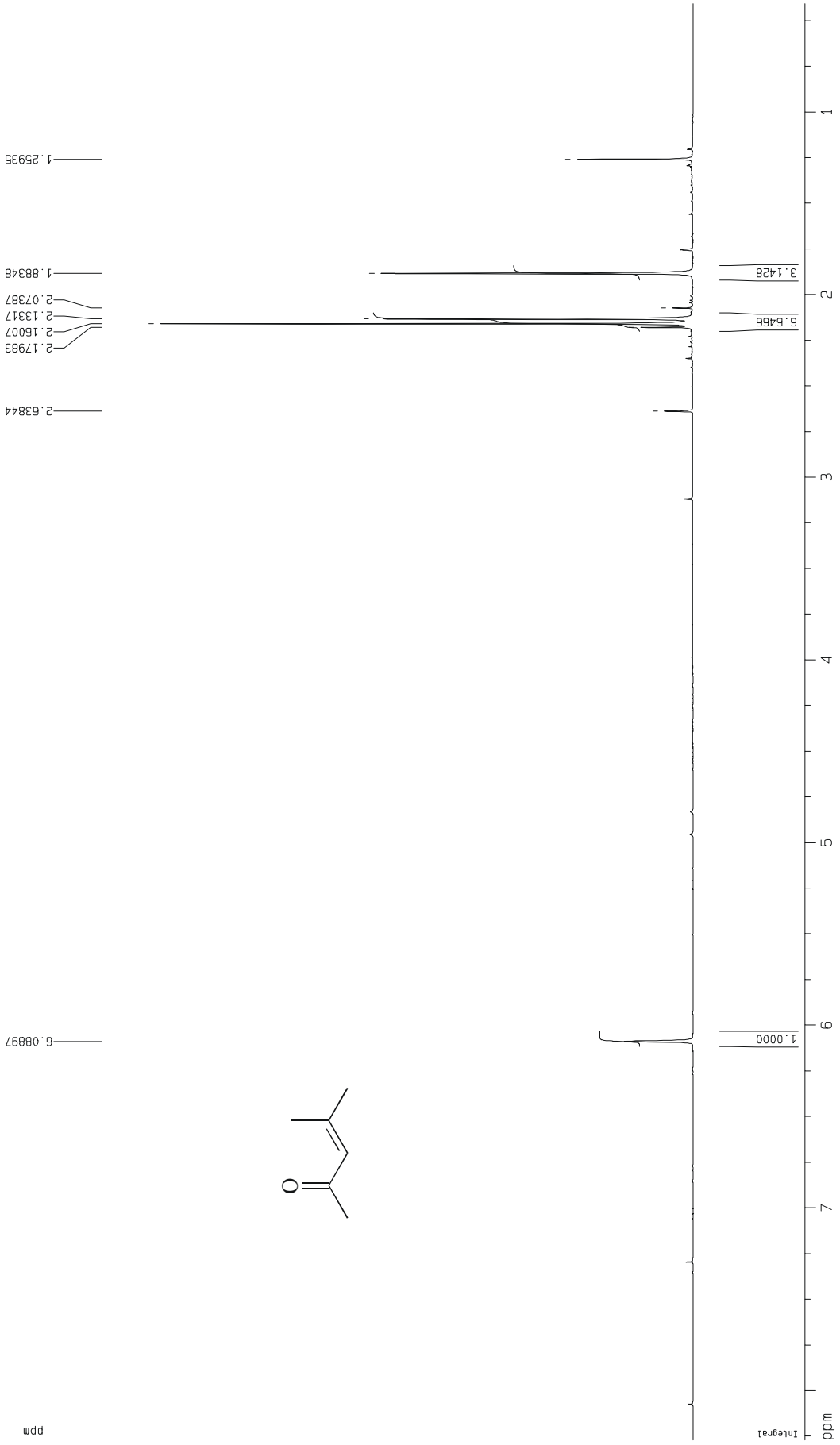
17. Bróm-sztirol (Z-25%, E-75 %) ^{1}H-NMR spektrum ($CDCl_3$)



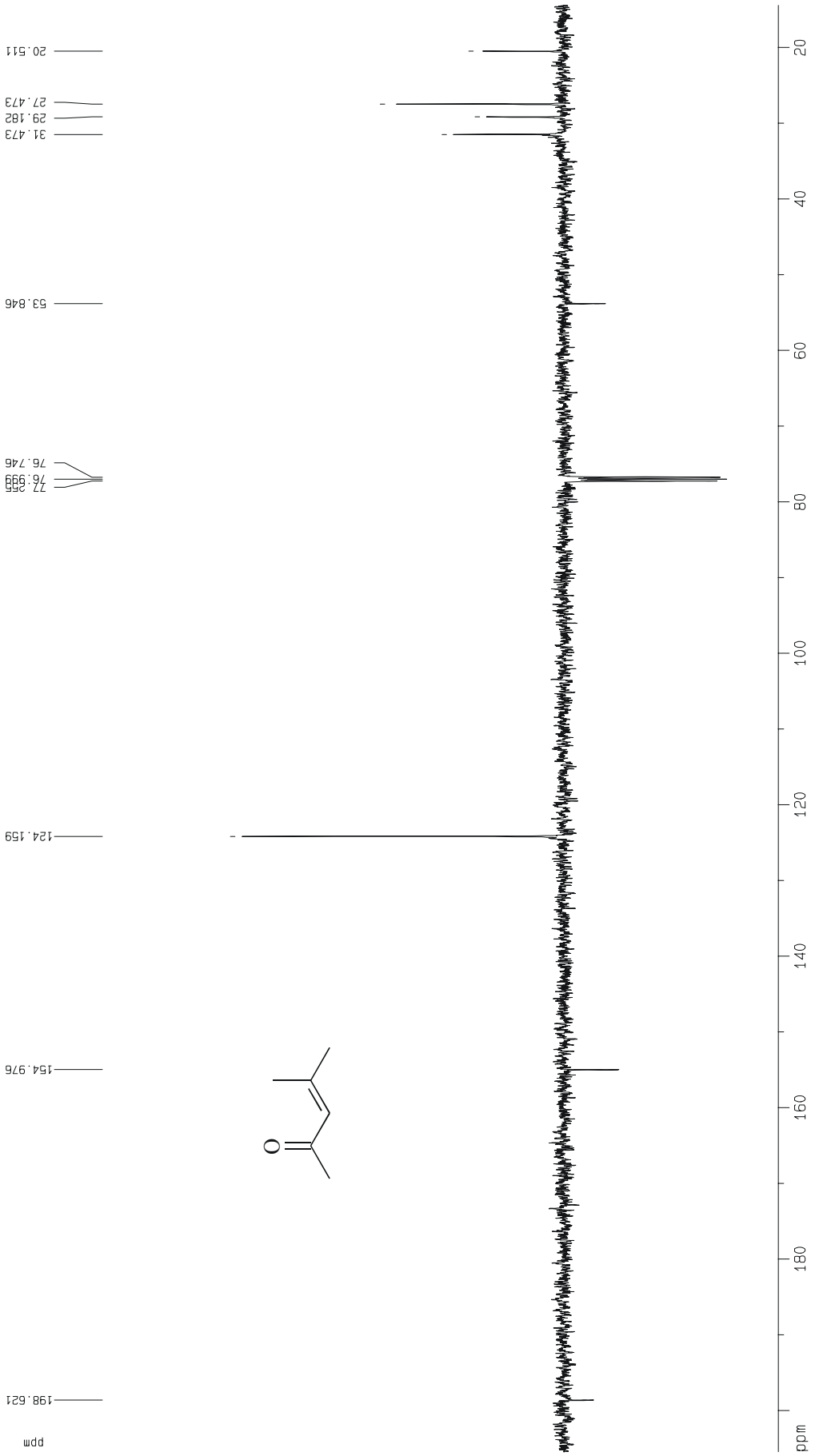
18. Bróm-sztirol (Z-91%, E-9 %) ^{13}C-JMOD NMR spektrum ($CDCl_3$)



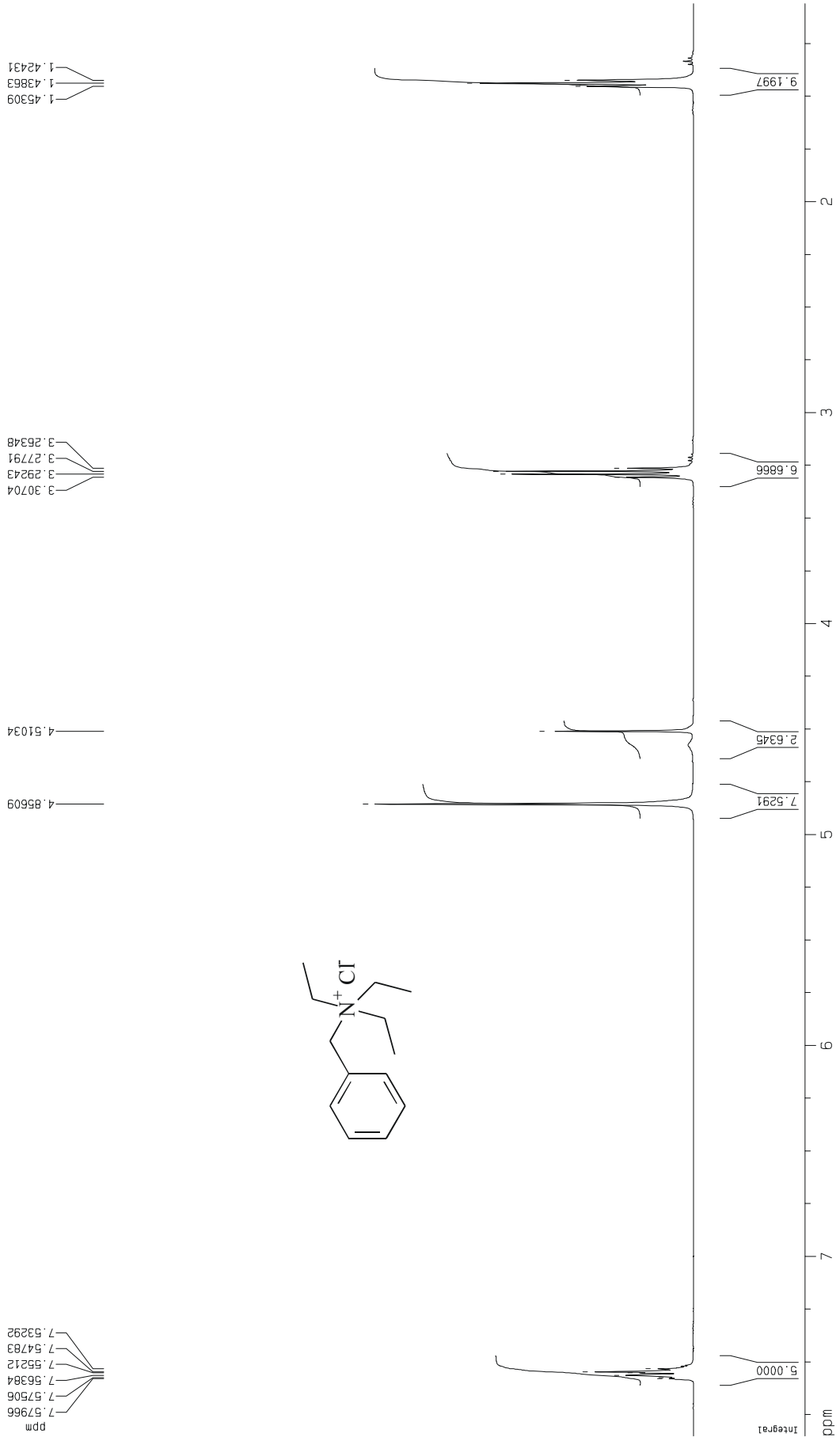
19. Mezitiloxid ^{1}H-NMR spektrum ($CDCl_3$)



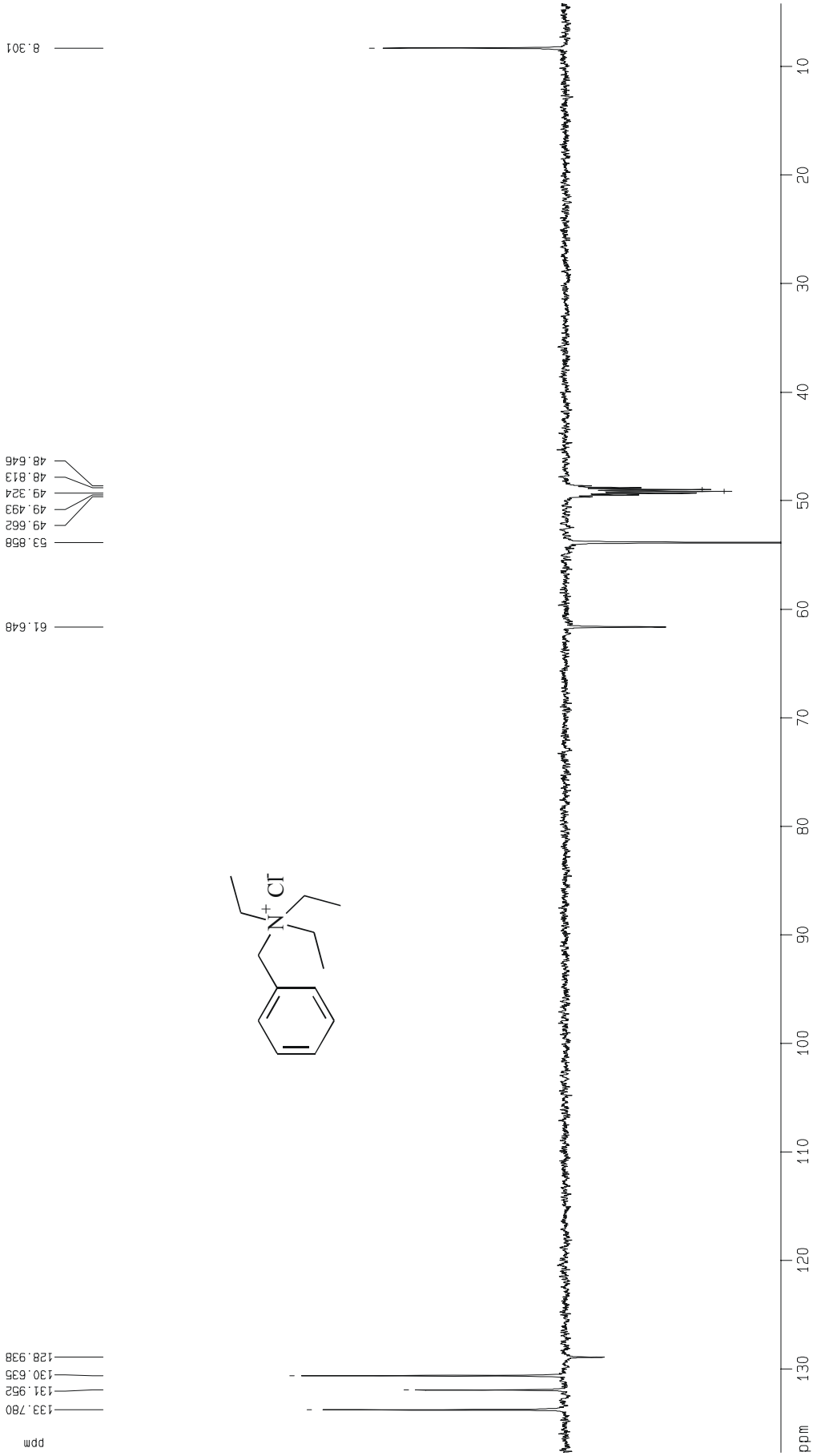
20. Mezitiloxid ^{13}C-JMOD NMR spektrum ($CDCl_3$)



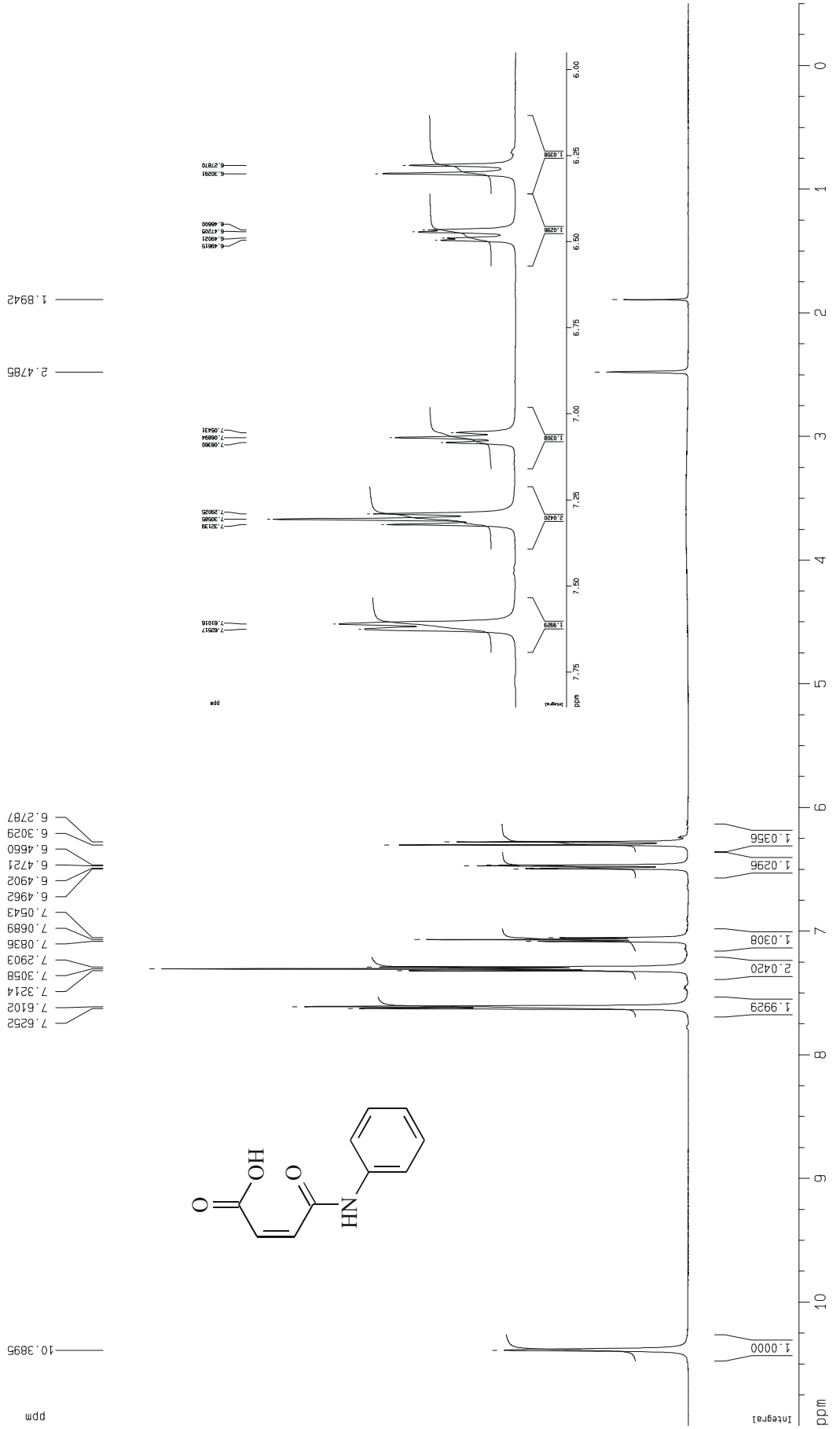
21. Benzil-trietil-ammónium-klorid ^{1}H-NMR spektrum (CD_3OD)



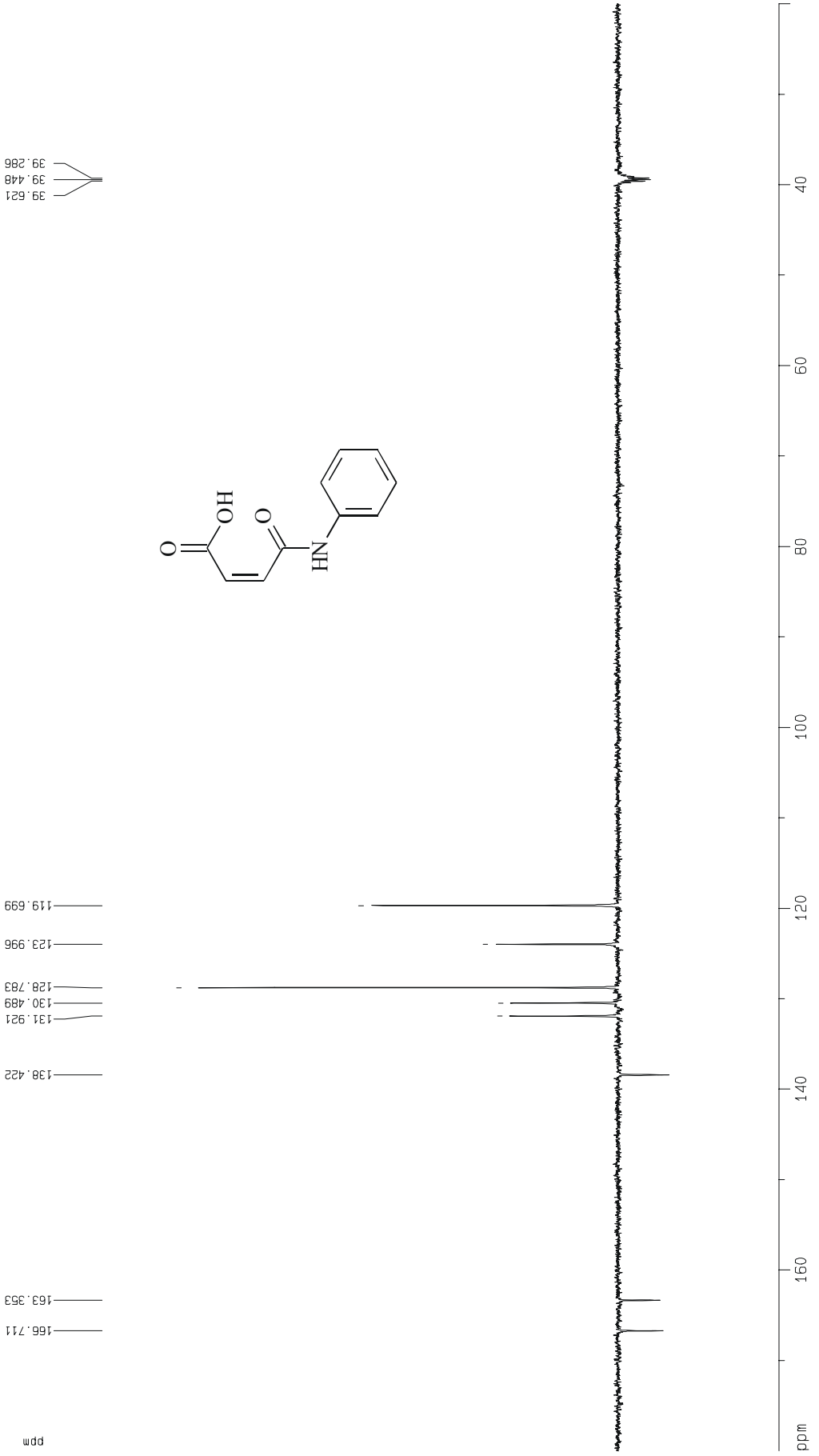
22. Benzil-trietil-ammónium-klorid ^{13}C-JMOD NMR spektrum (CD_3OD)



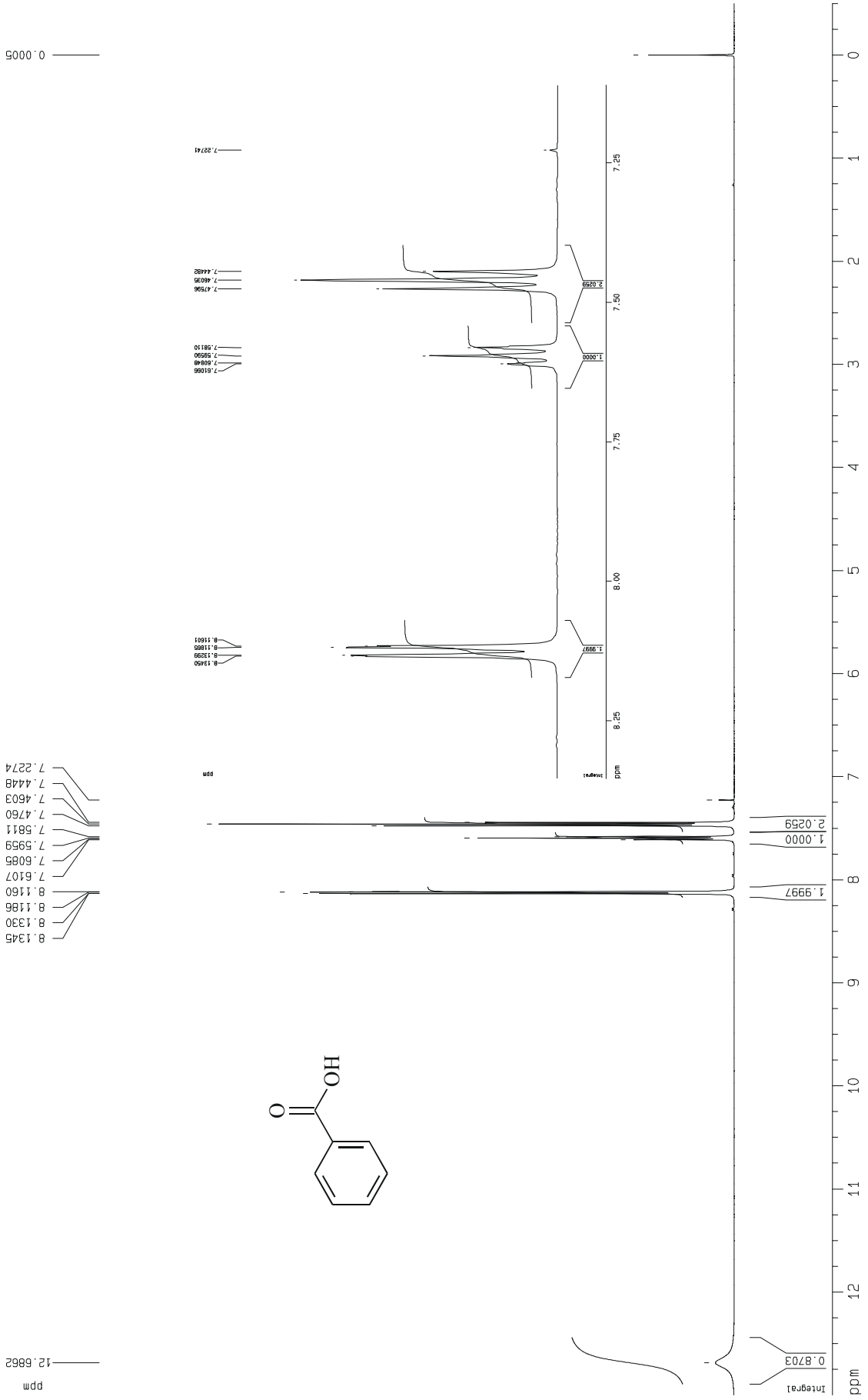
24. Maleanilidsav ^{1}H-NMR spektrum (DMSO)



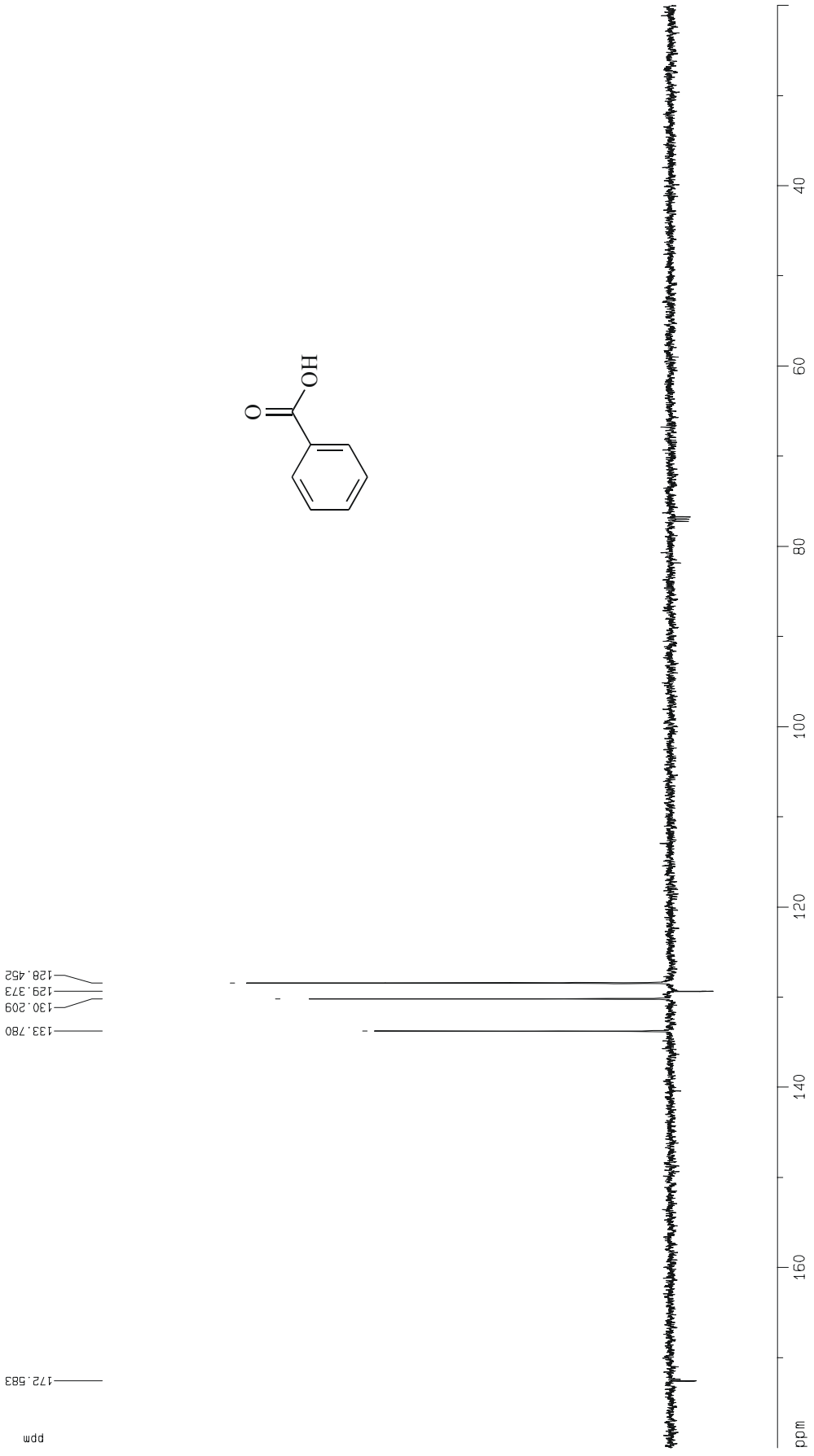
25. Maleanilidsav ^{13}C-JMOD NMR spektrum (DMSO)



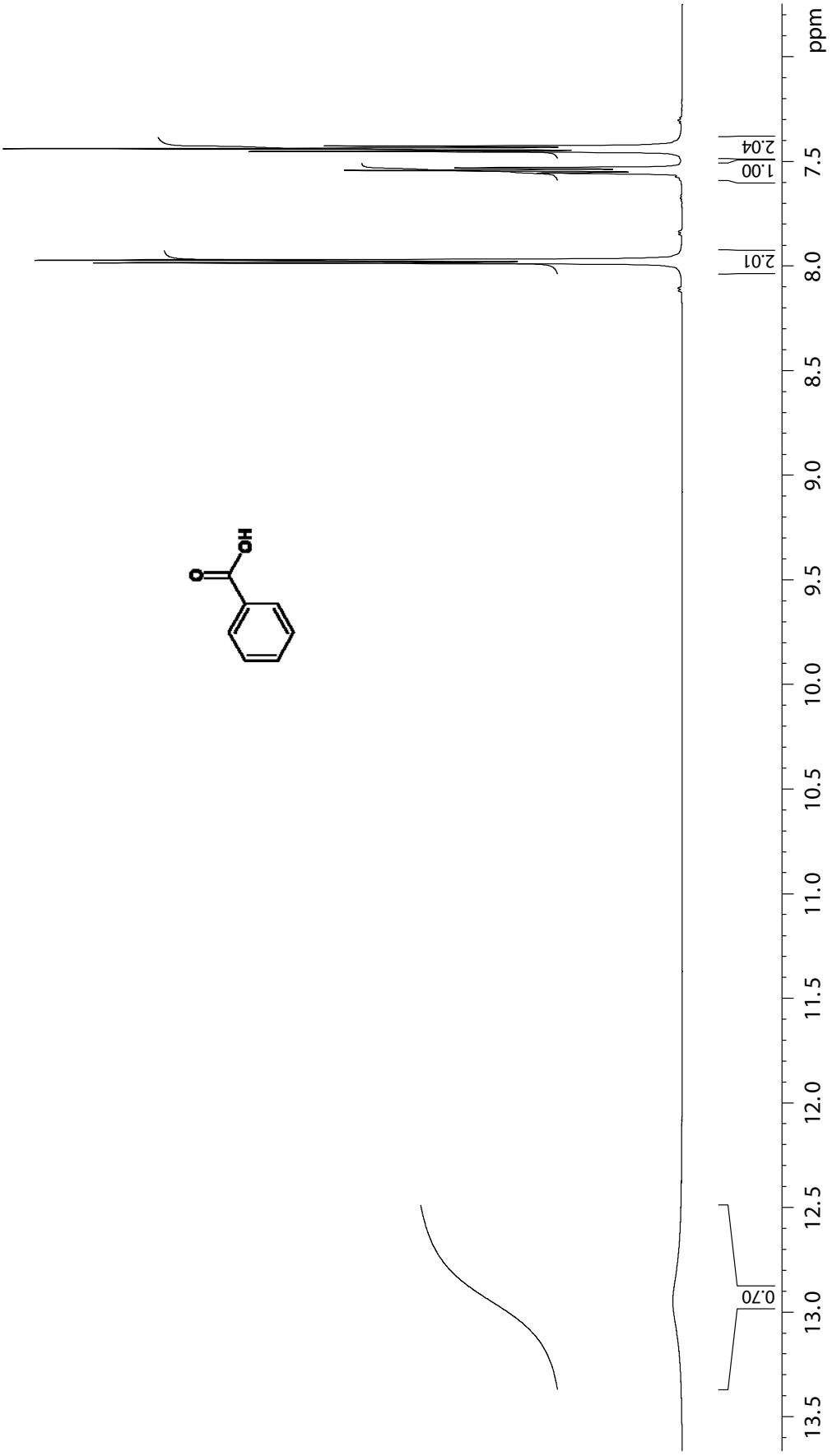
25. Benzoesav ^{1}H-NMR spektrum ($CDCl_3$)



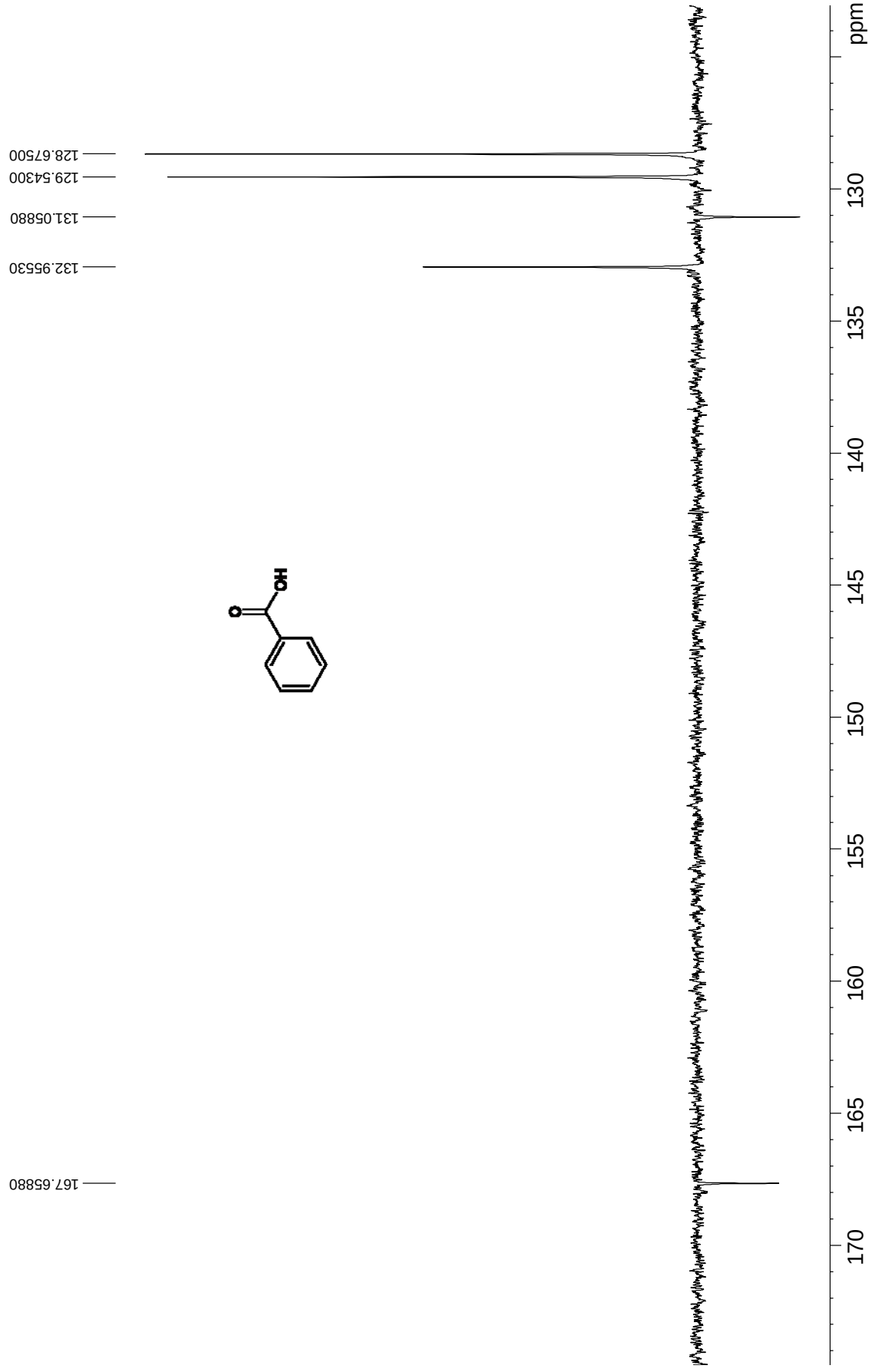
26. Benzoesav ^{13}C JMOD NMR spektrum ($CDCl_3$)



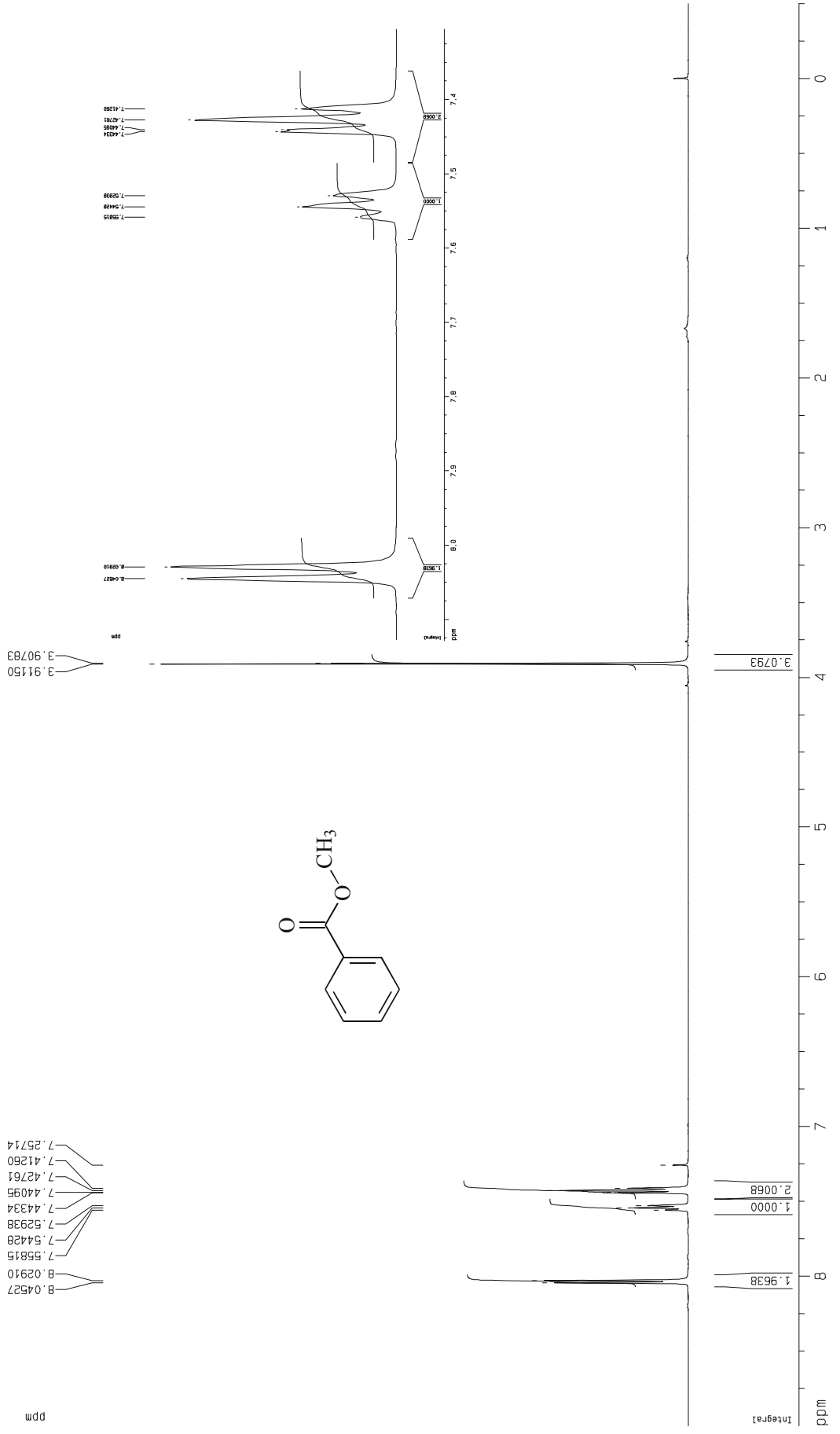
27. Benzoesav ^{1}H-NMR spektrum (DMSO)



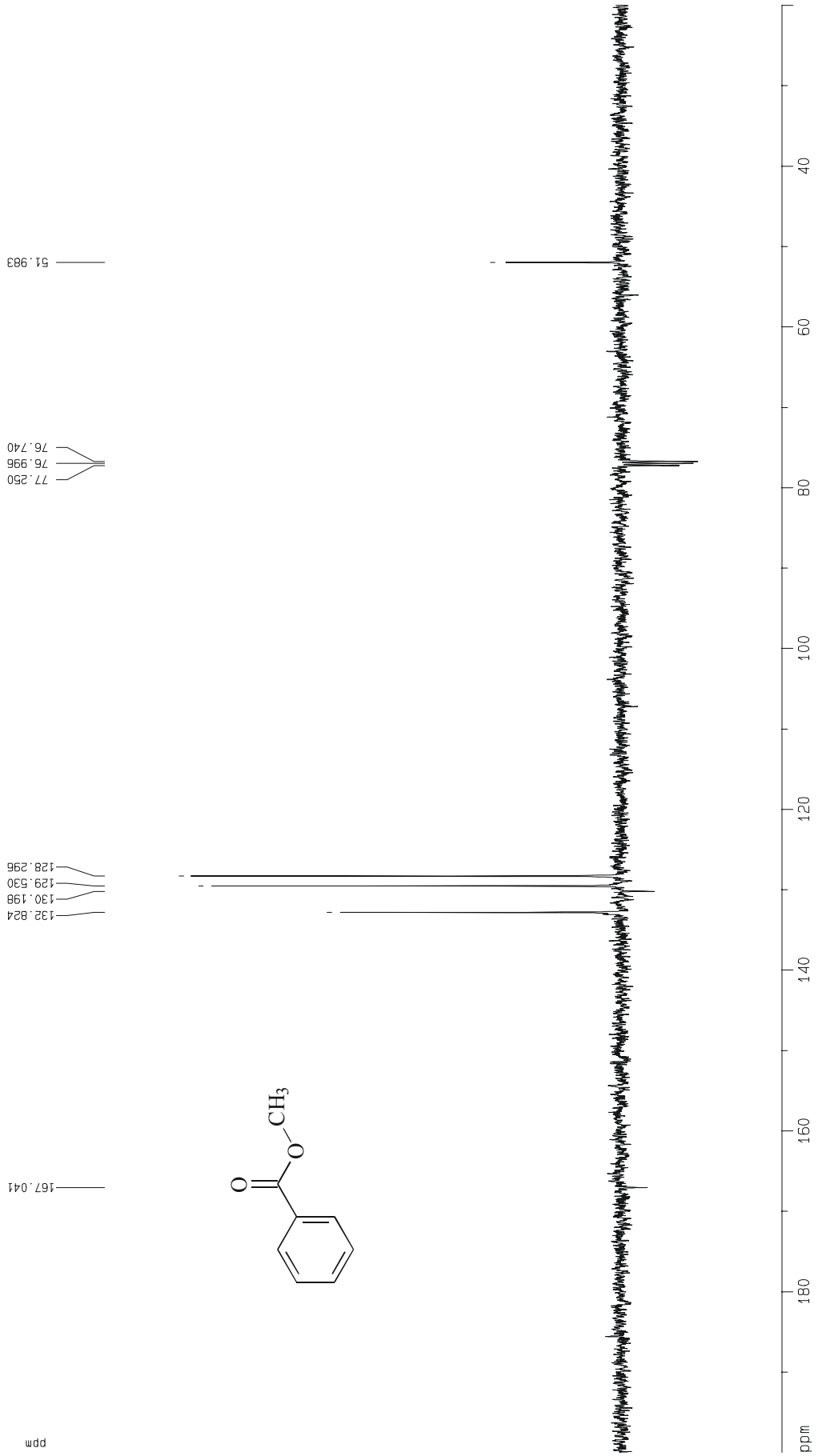
28. Benzoesav ^{13}C-JMOD NMR (DMSO)



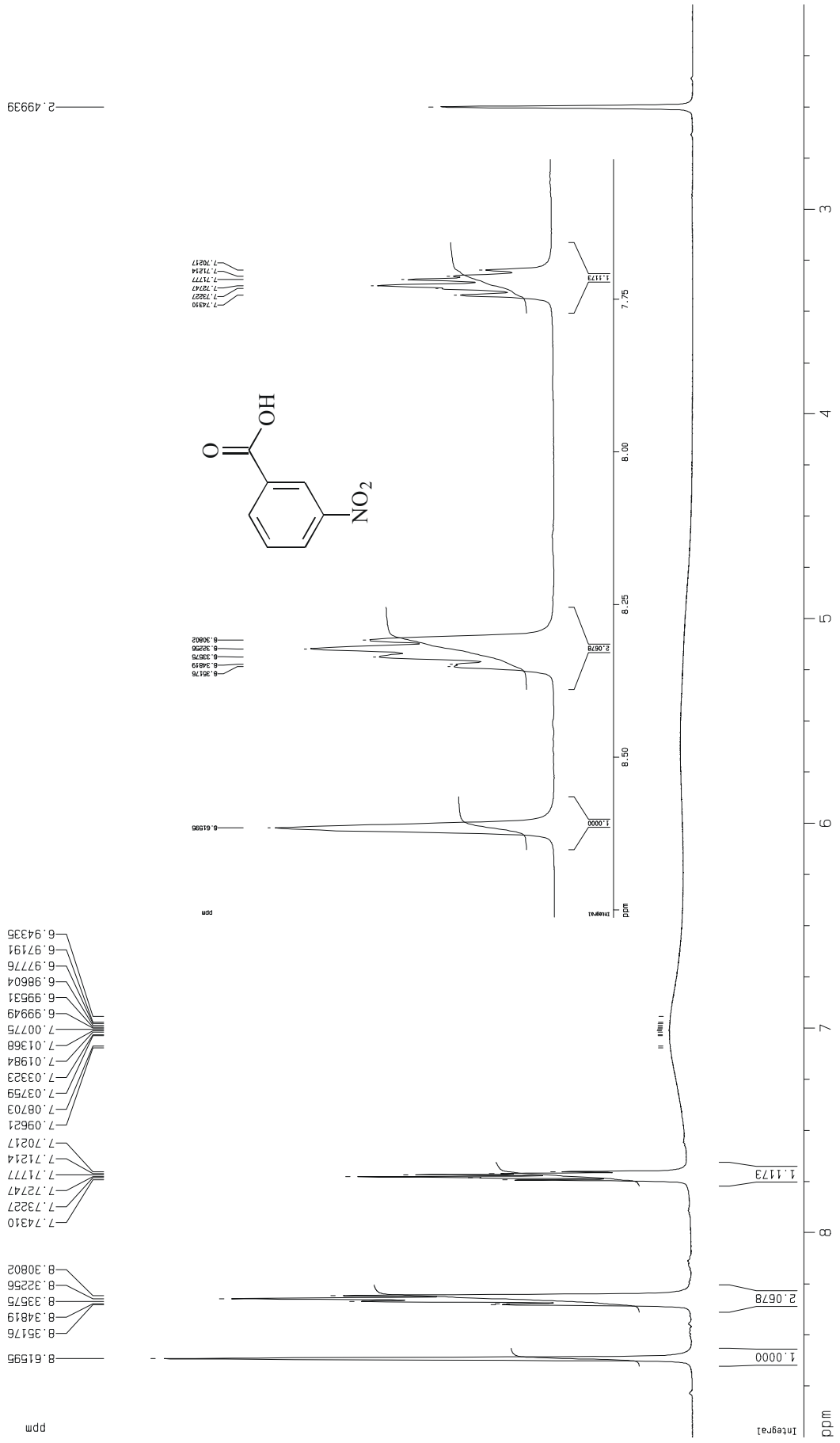
29. Benzoesav-metilészter ^{1}H-NMR spektrum ($CDCl_3$)



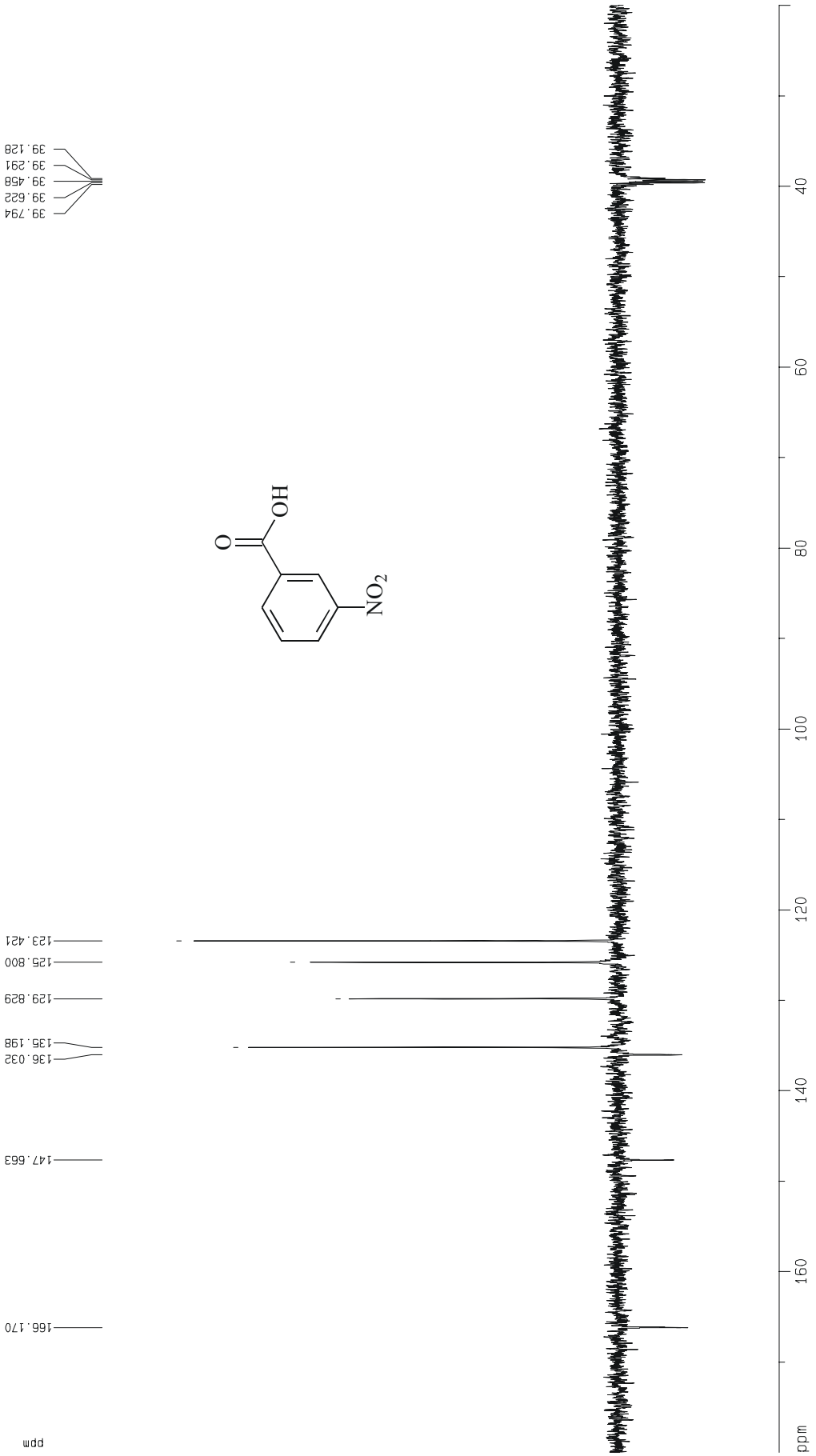
30. Benzoesav-metilészter ^{13}C-JMOD NMR spektrum ($CDCl_3$)



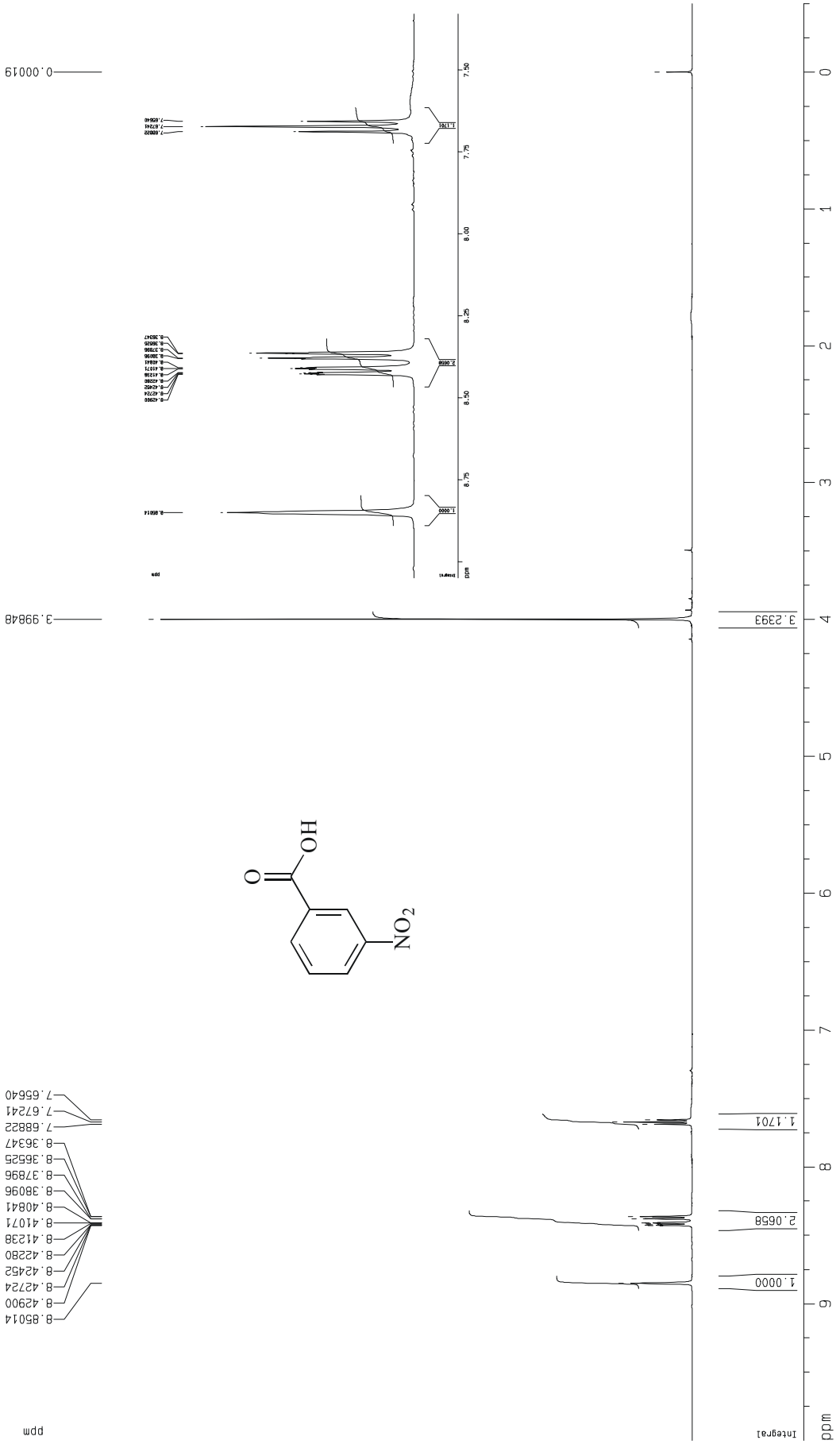
32. 3-Nitro-benzoesav ^{1}H-NMR spektrum (DMSO)



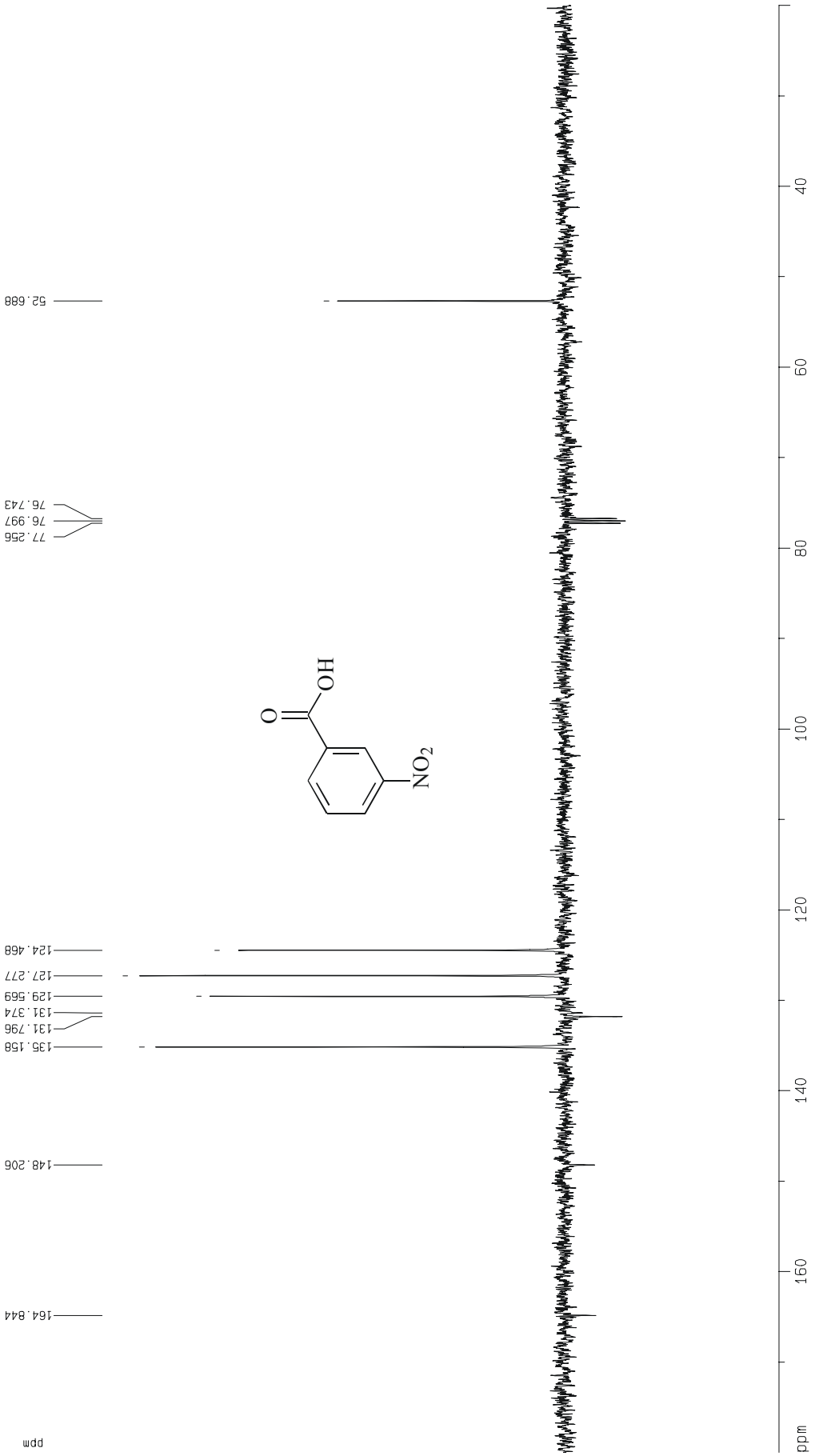
33. 3-Nitro-benzoesav ^{13}C-JMOD NMR spektrum (DMSO)



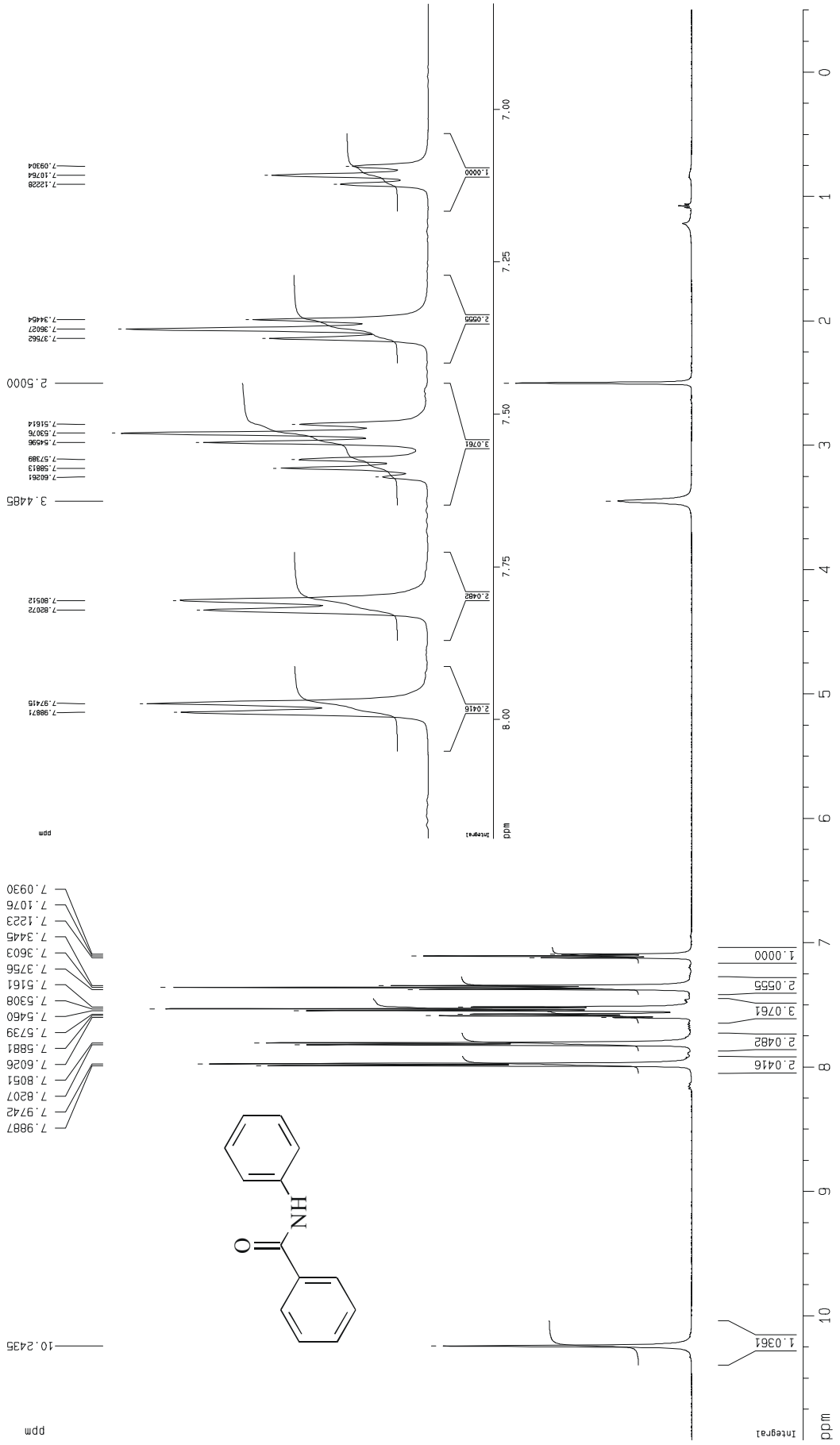
34. 3-Nitro-benzoesav-metilészter ^{1}H-NMR spektrum ($CDCl_3$)



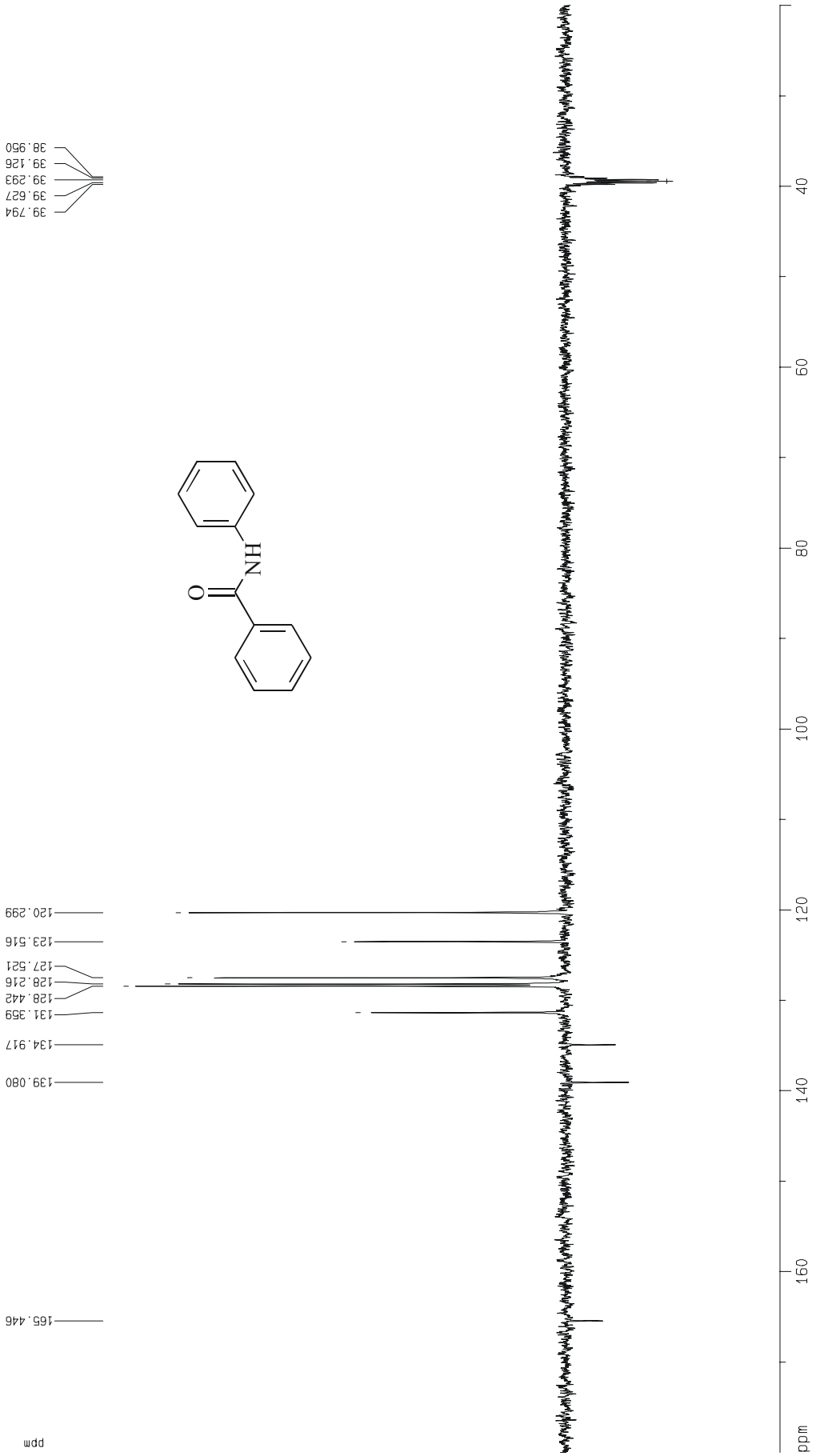
35. 3-Nitro-benzoesav-metilészter ^{13}C-JMOD NMR spektrum ($CDCl_3$)



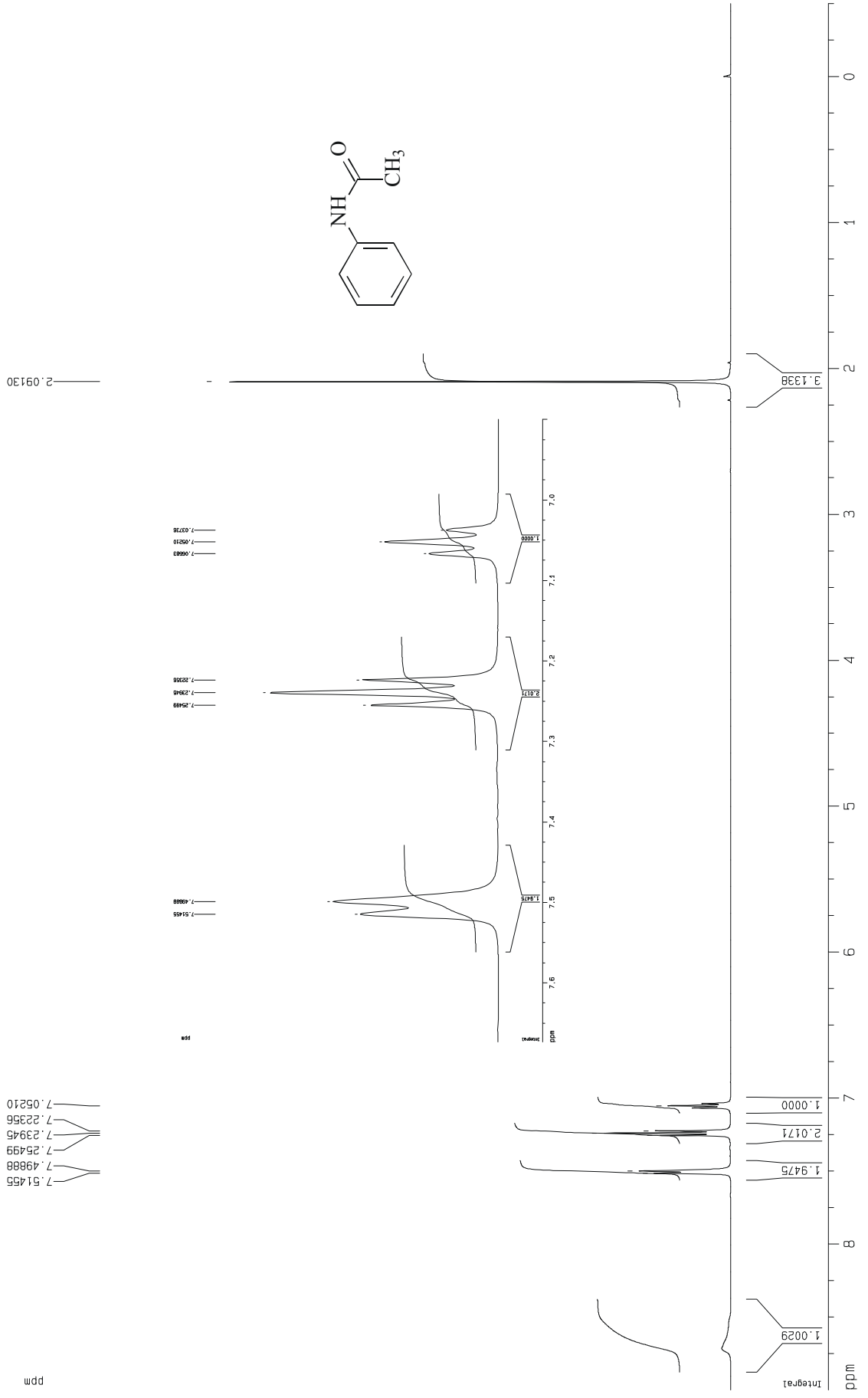
35. Benzanilid ^{1}H-NMR spektrum (DMSO)



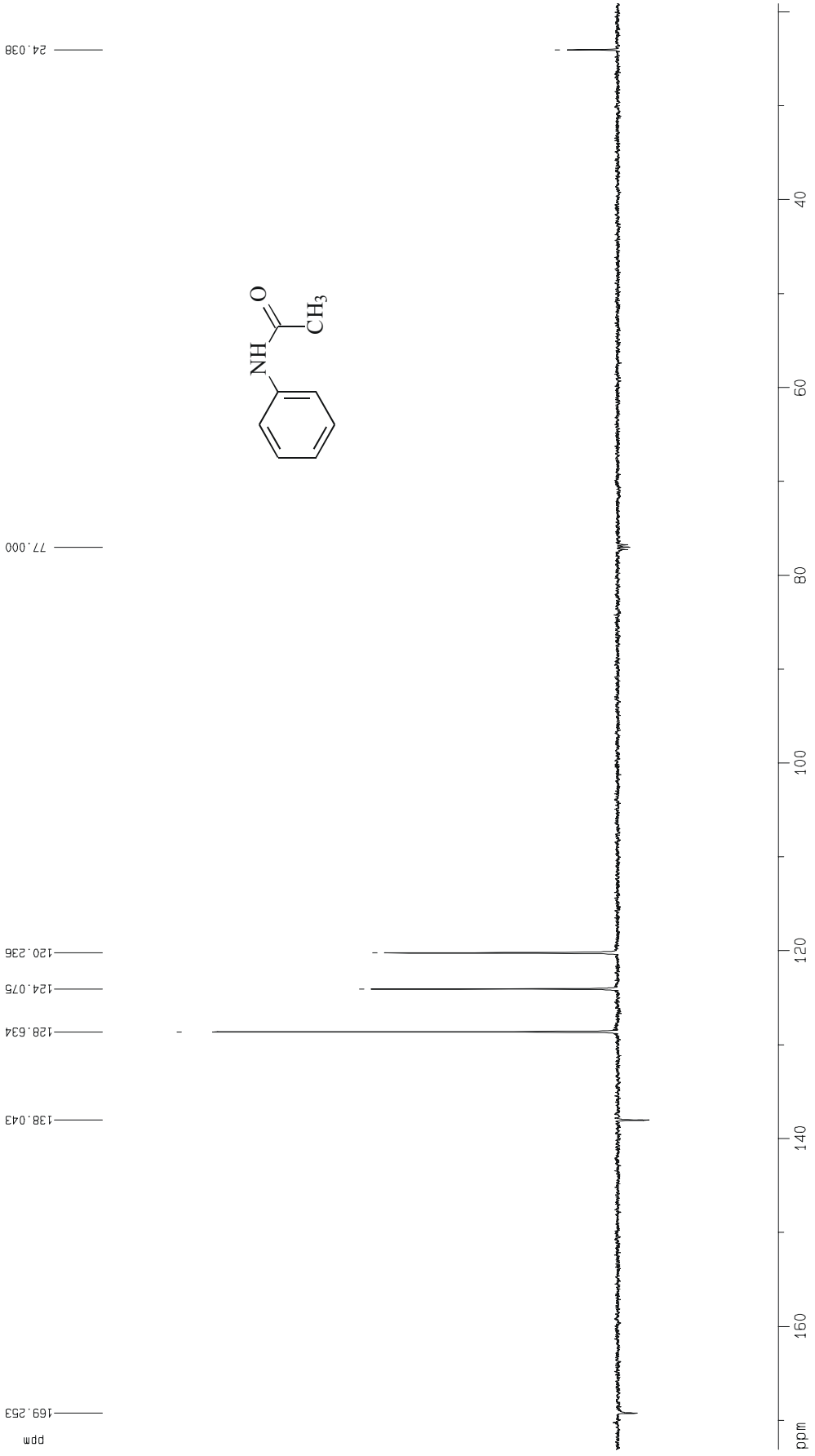
36. Benzanilid ^{13}C-JMOD NMR spektrum (DMSO)



37. Acetanilid ^{1}H-NMR spektrum ($CDCl_3$)



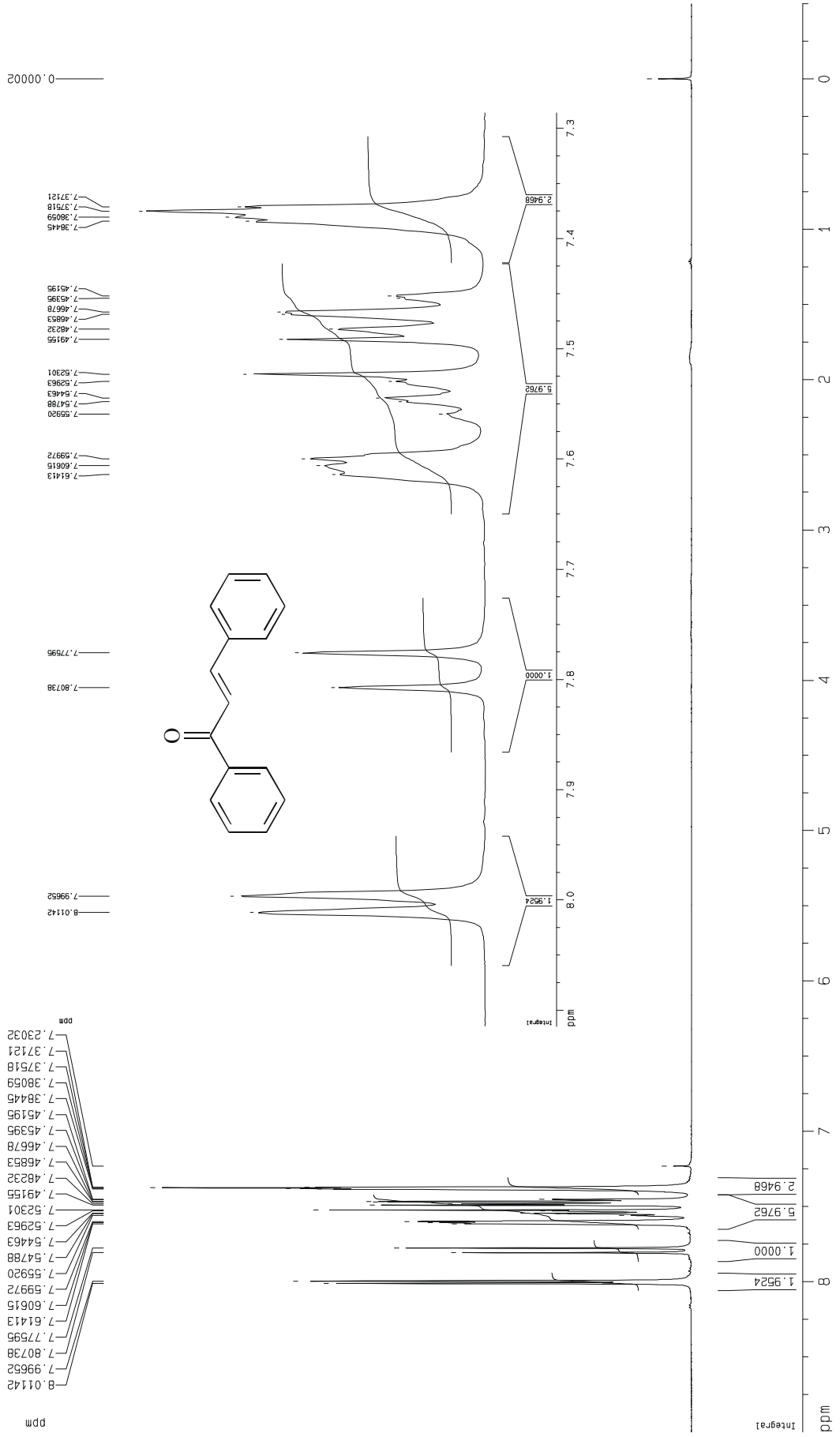
38. Acetanilid ^{13}C-JMOD NMR spektrum ($CDCl_3$)



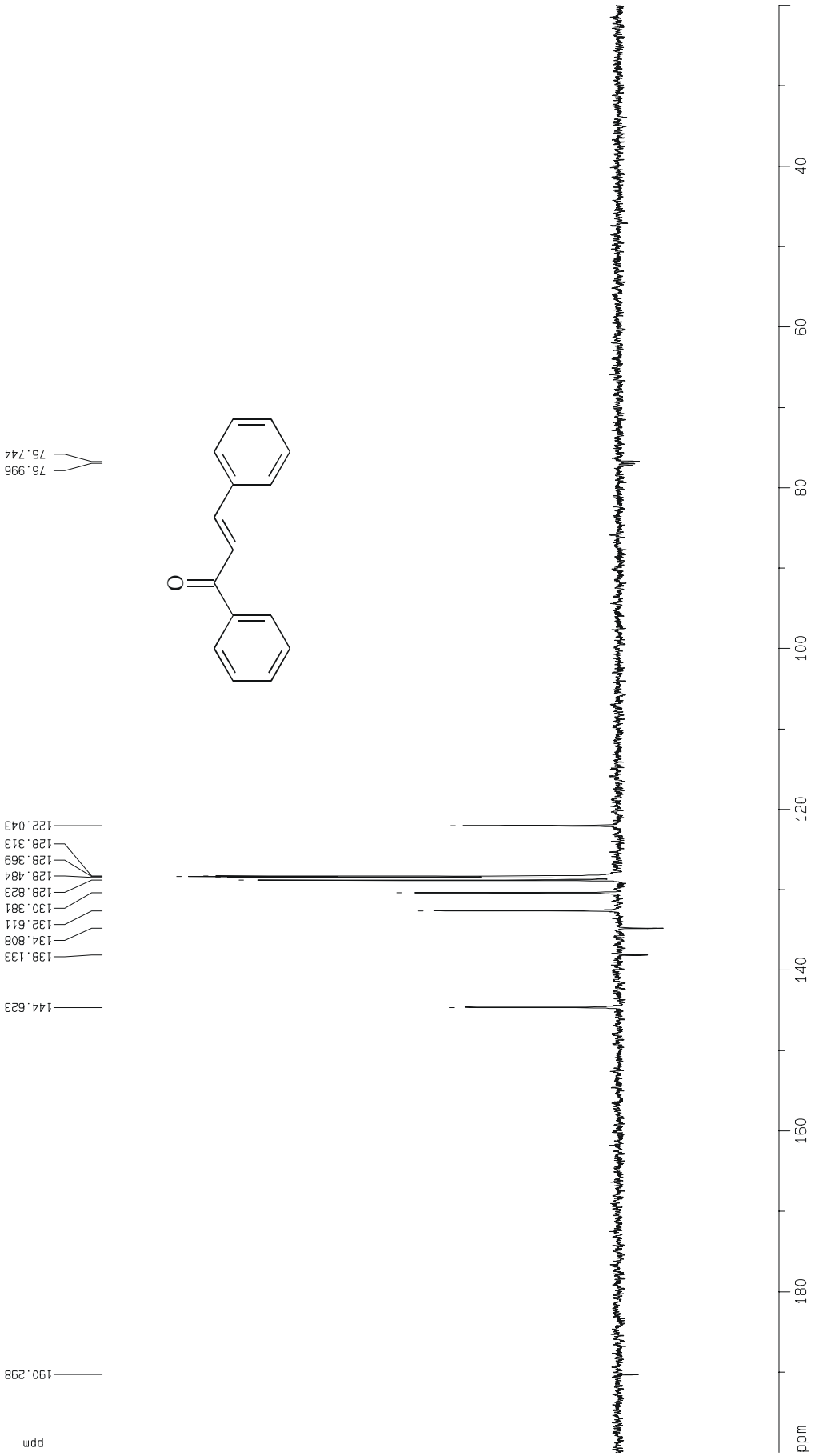
39. 4-Nitro-acetanilid ^{1}H-NMR spektrum (DMSO)

40. 4-Nitro-acetanilid ^{13}C-JMOD NMR spektrum (DMSO)

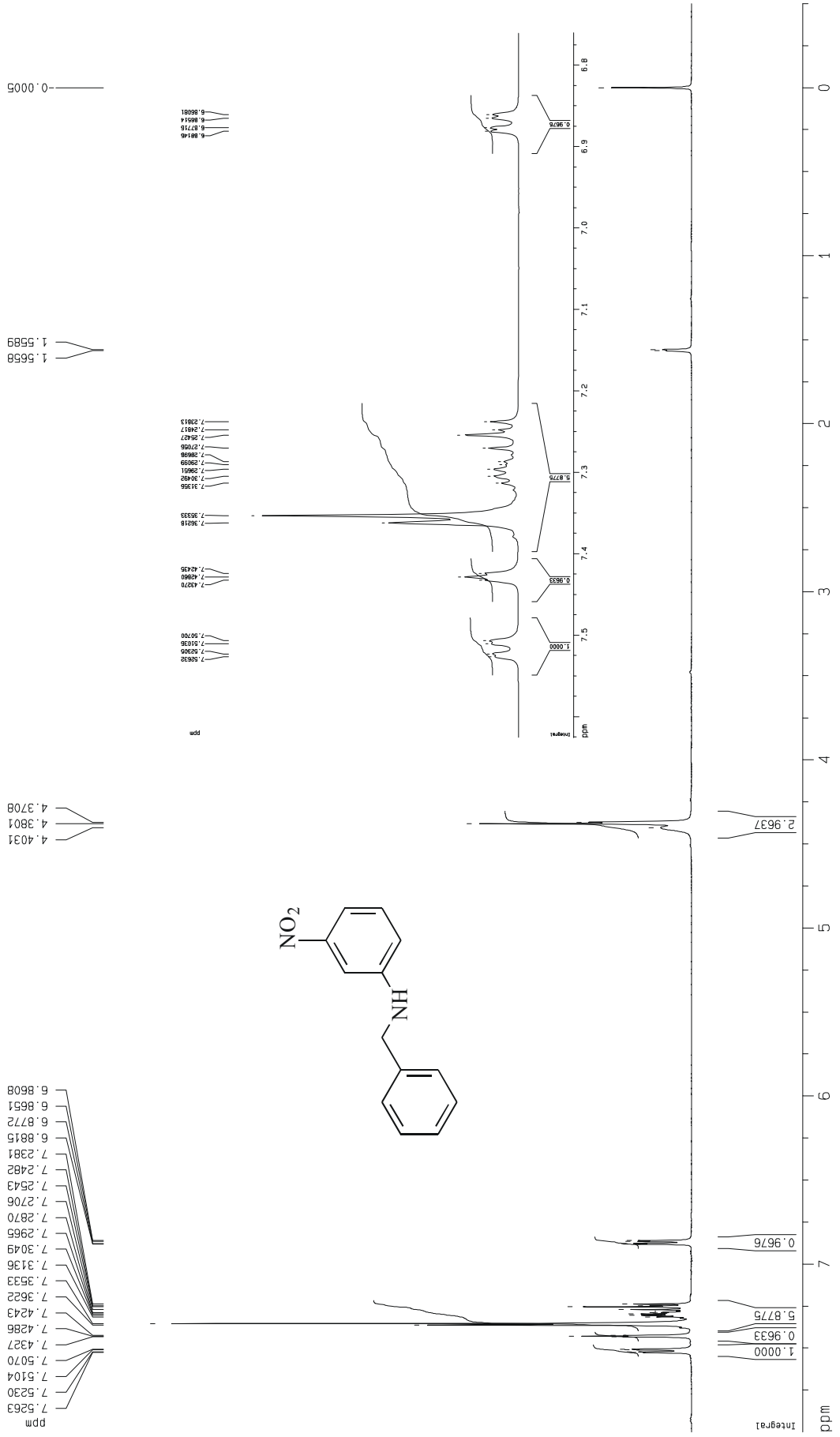
41. Benzilidén-acetofenon ^{1}H-NMR spektrum ($CDCl_3$)



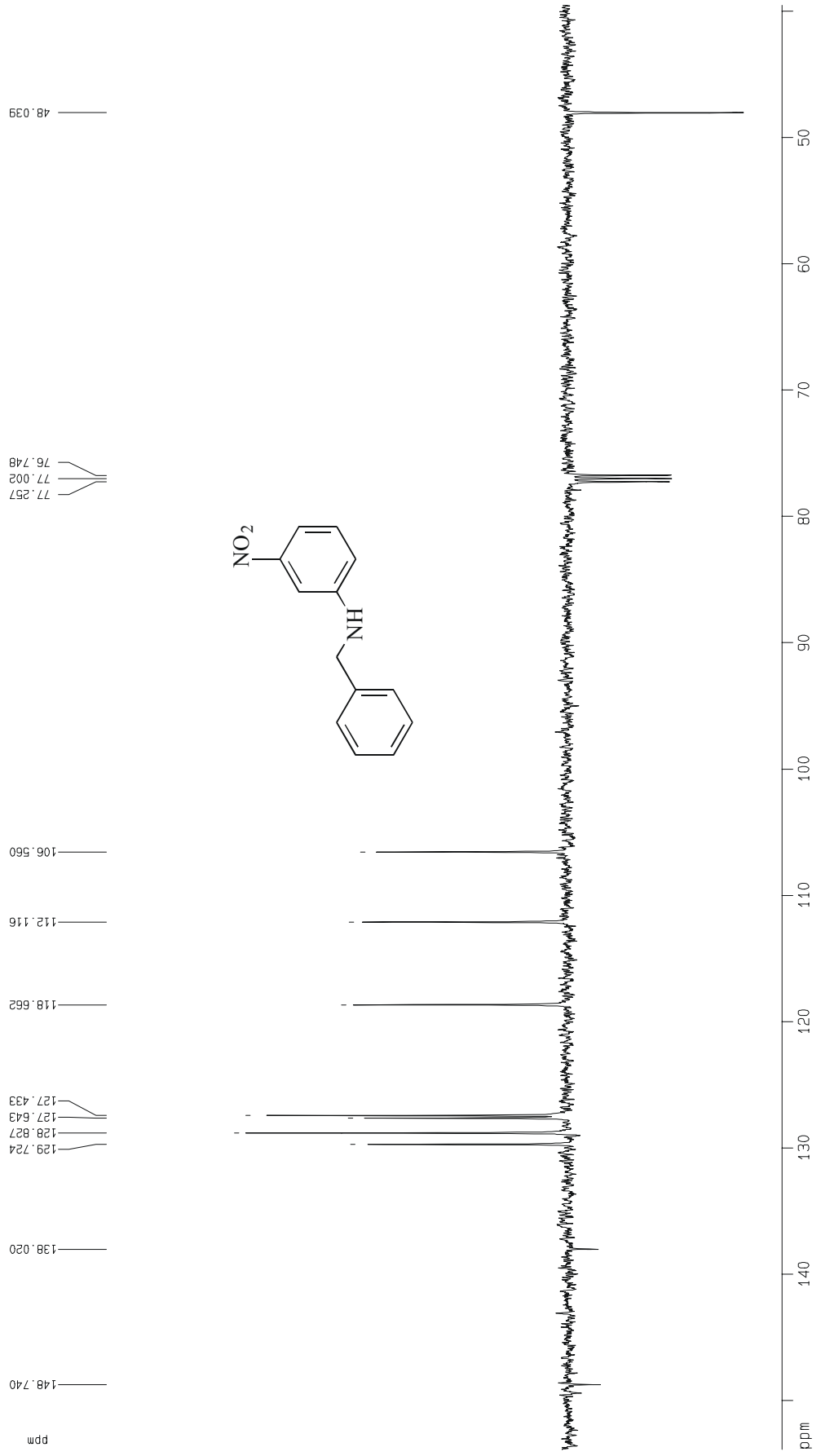
42. Benzilidén-acetofenon ^{13}C-JMOD NMR spektrum ($CDCl_3$)



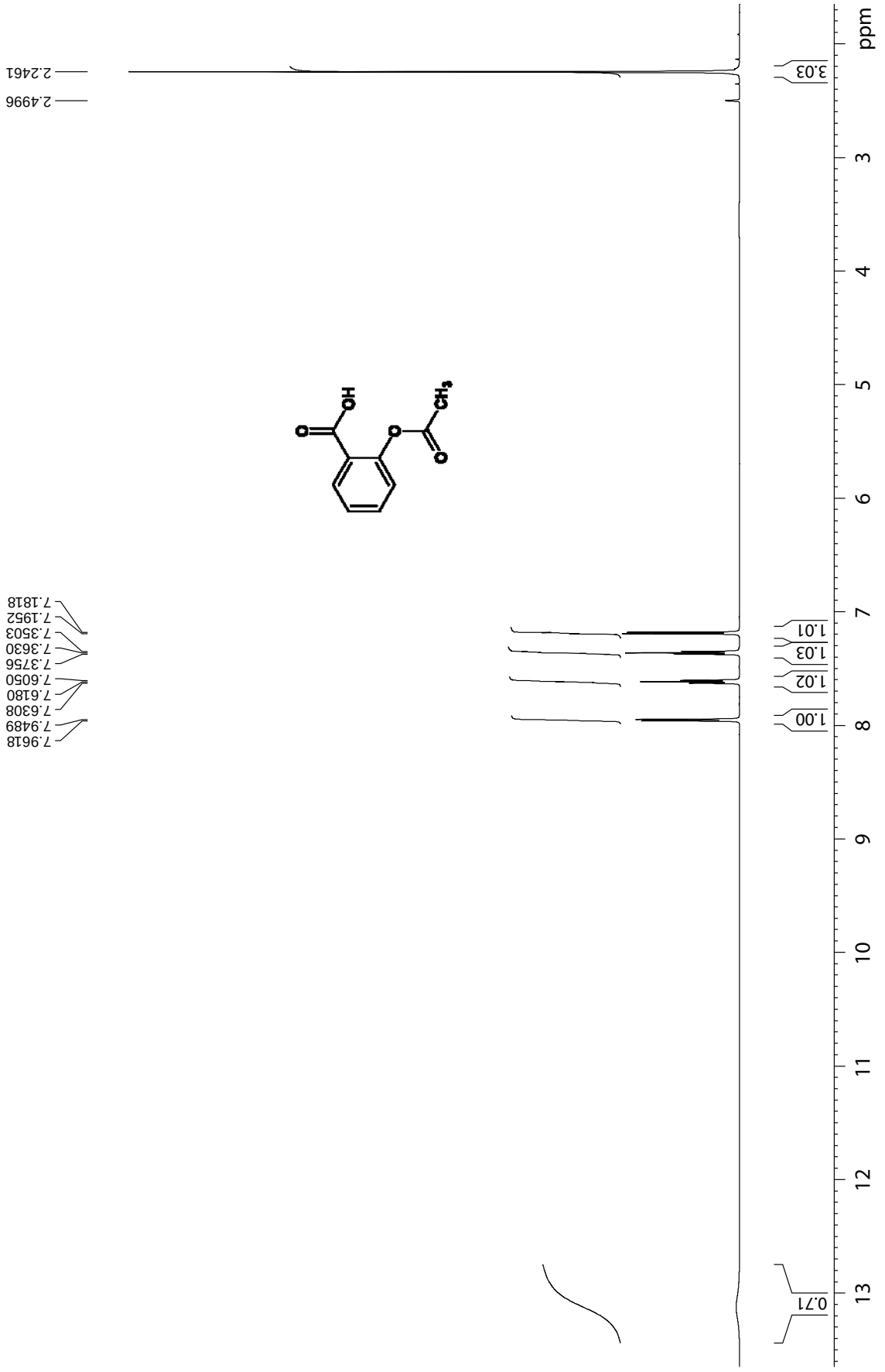
43. *N*-Benzil-3-nitro-anilin ^{1}H-NMR spektrum ($CDCl_3$)



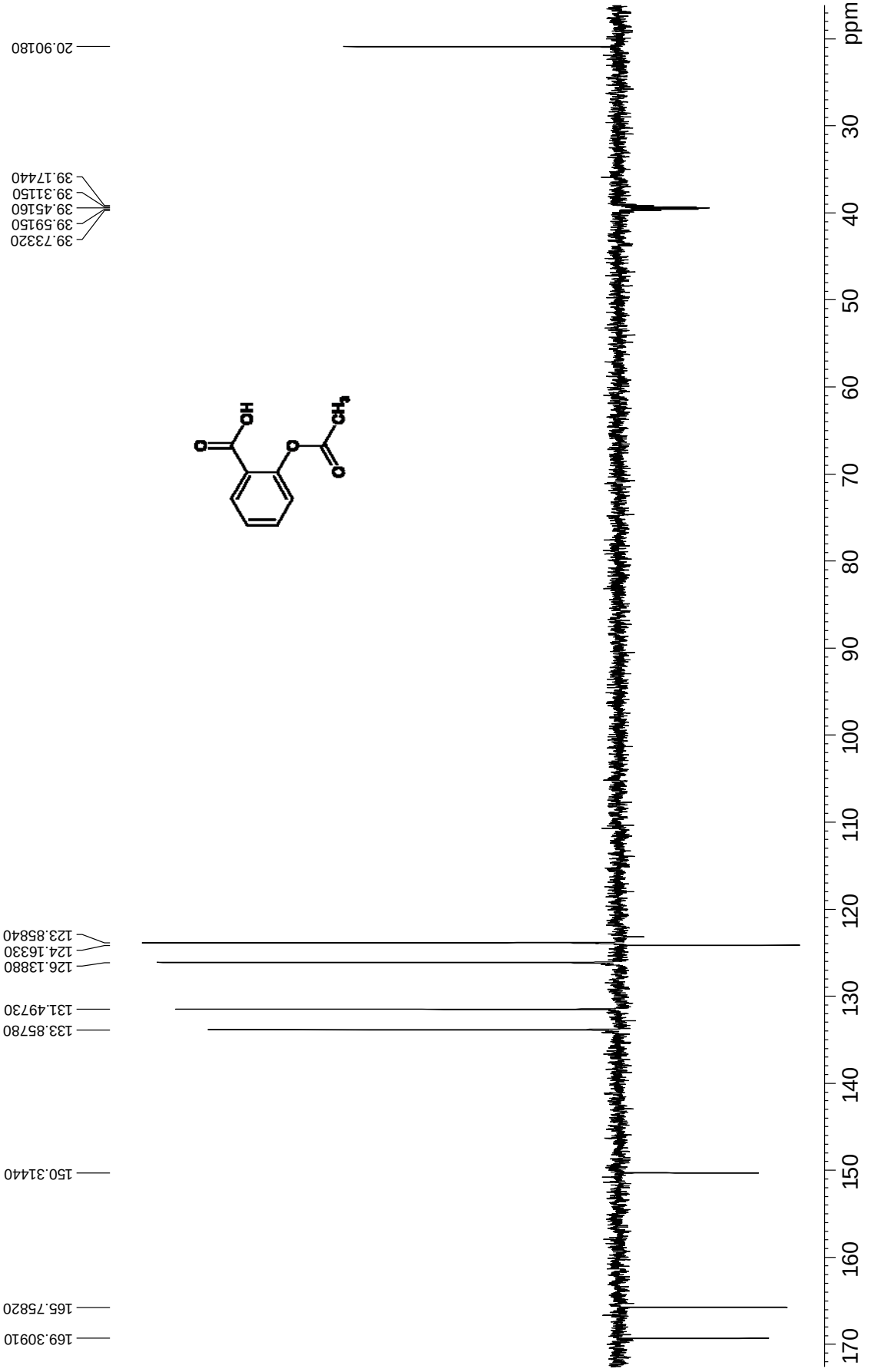
44. *N*-benzil-3-nitro-anilin ^{13}C-JMOD NMR spektrum ($CDCl_3$)



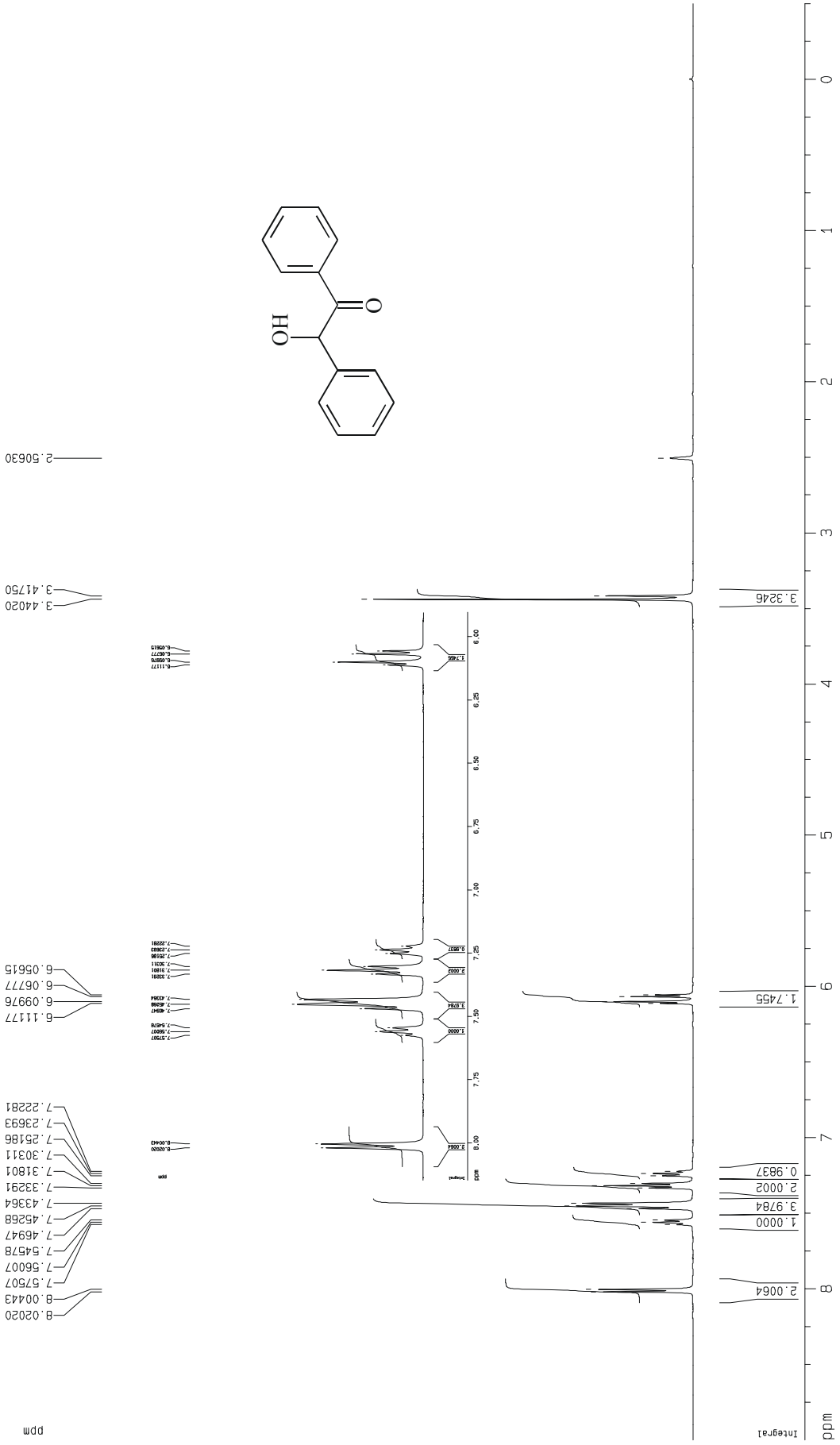
45. Acetil-szalicilsav ^{1}H-NMR spektrum (DMSO)



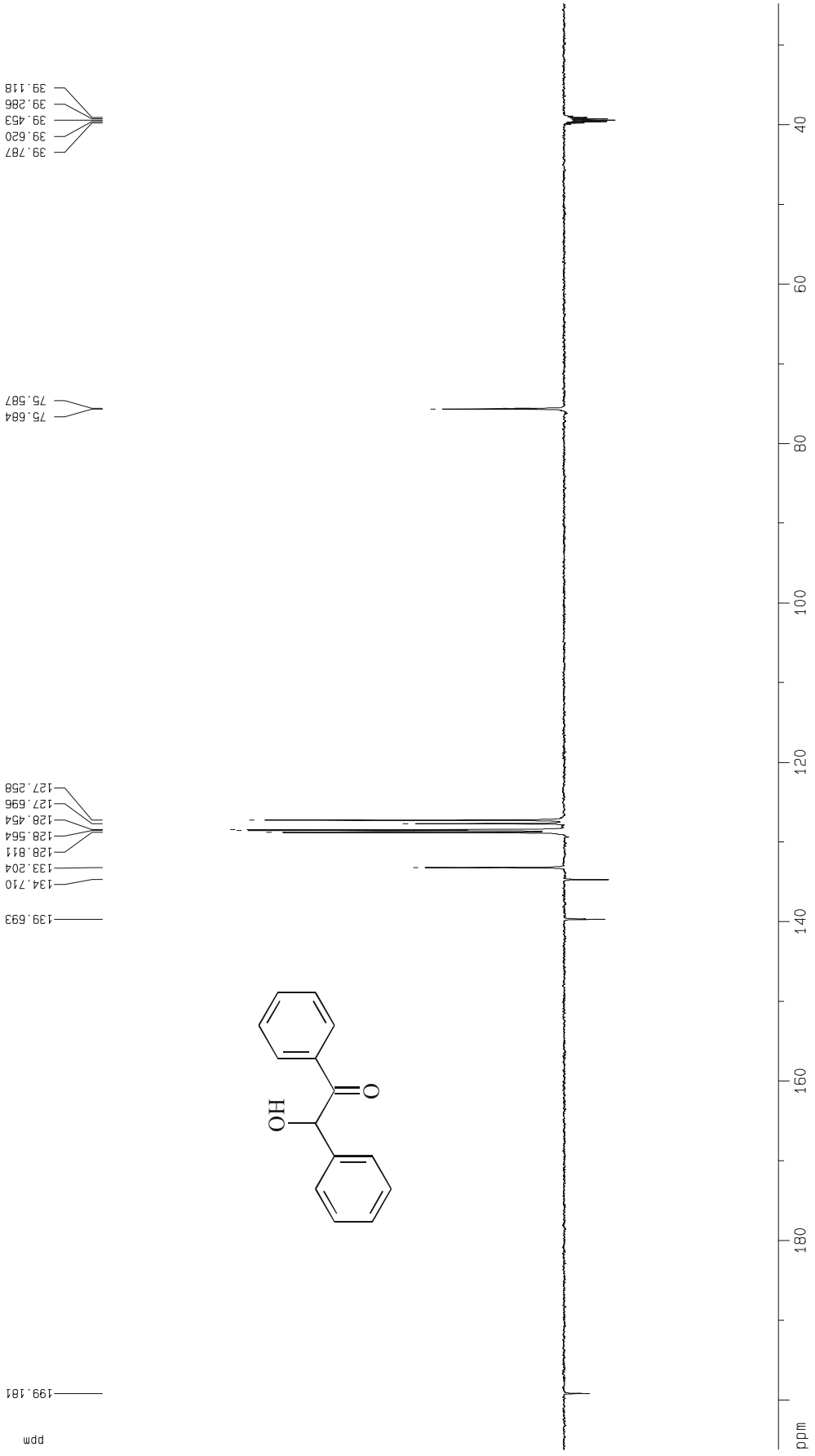
46. Acetil-szalicilsav ^{13}C-JMOD NMR spektrum (DMSO)



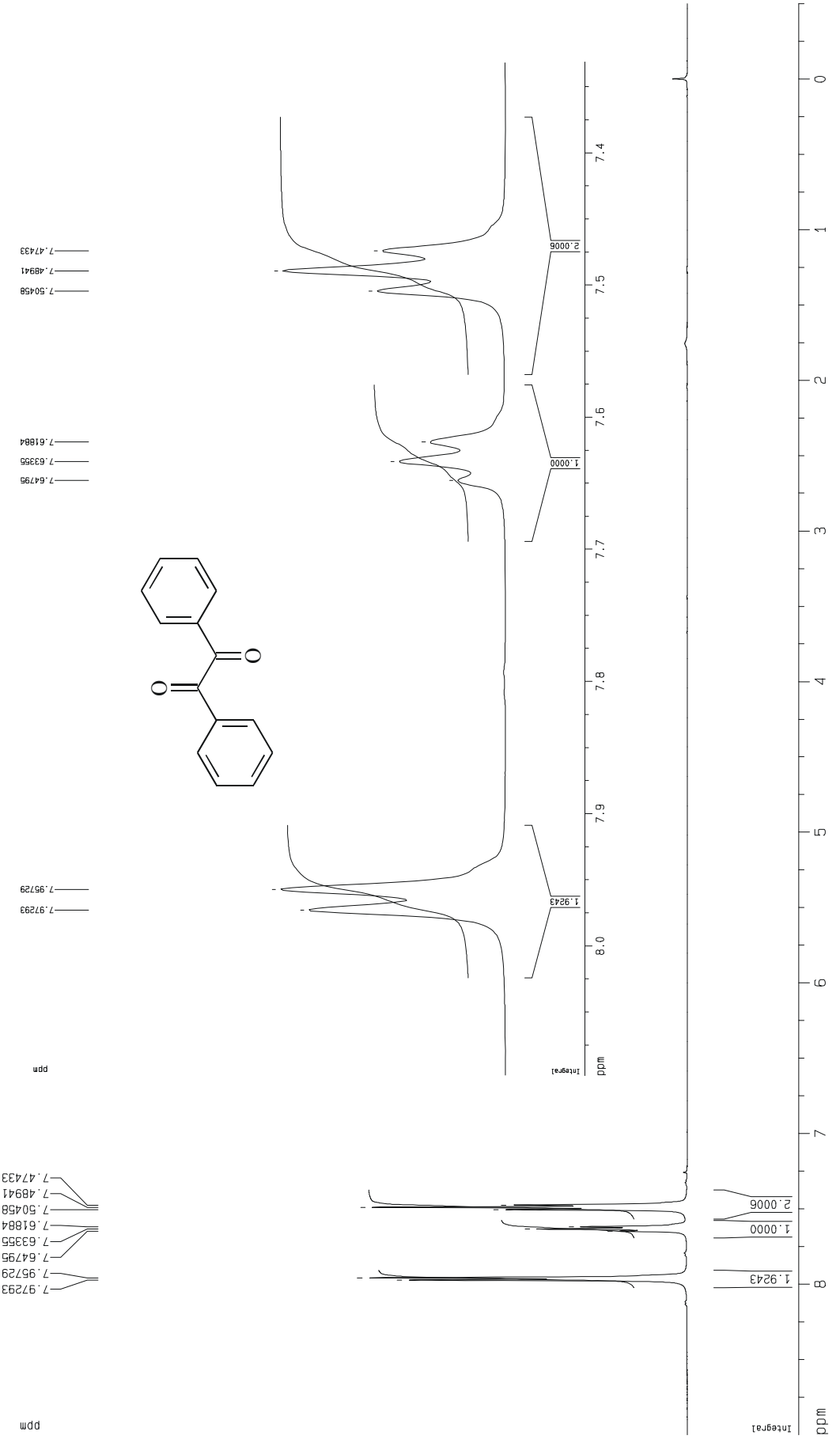
47. Benzoin ^{1}H-NMR spektrum (DMSO)



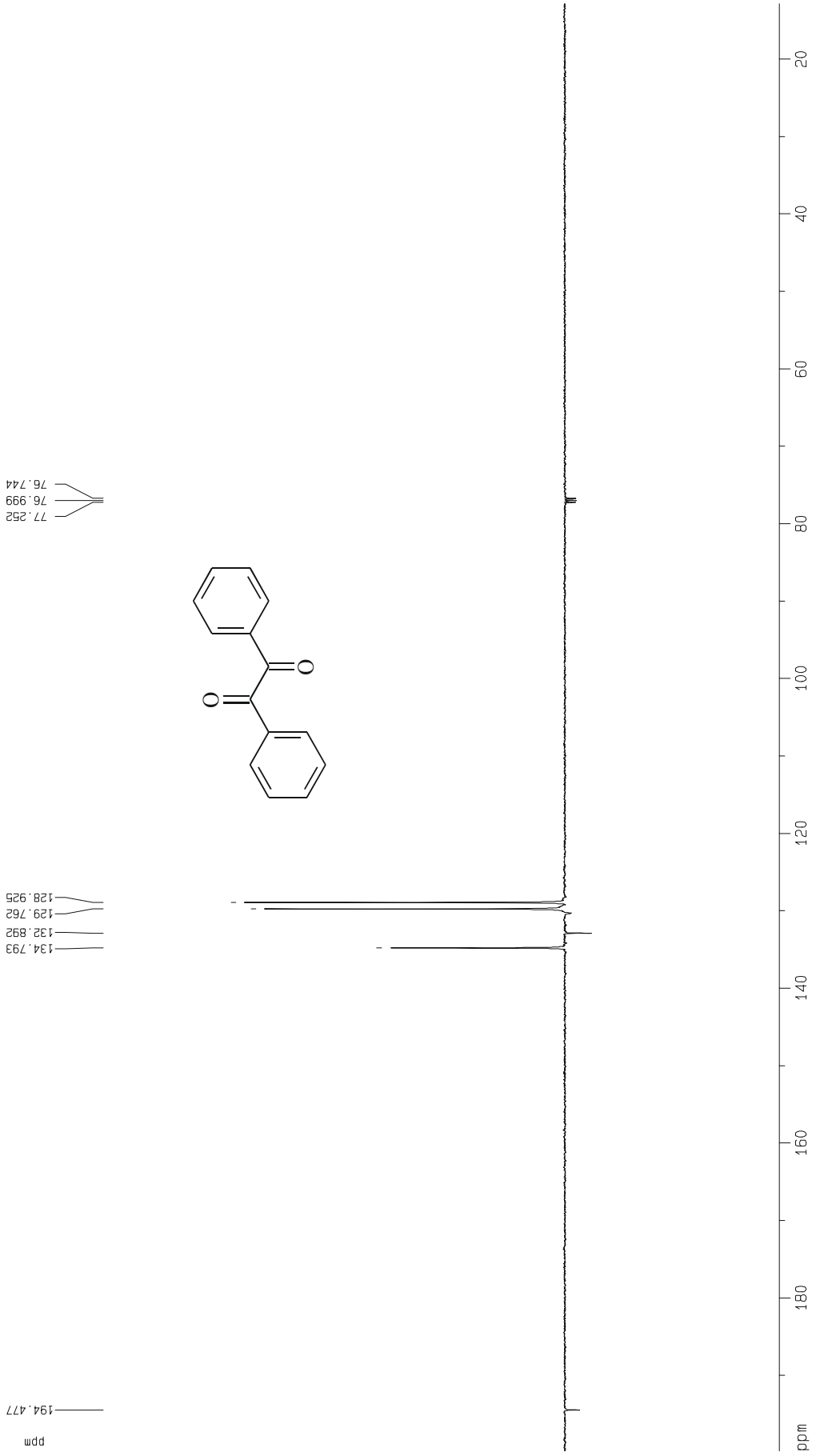
48. Benzoin ^{13}C-JMOD NMR spektrum (DMSO)



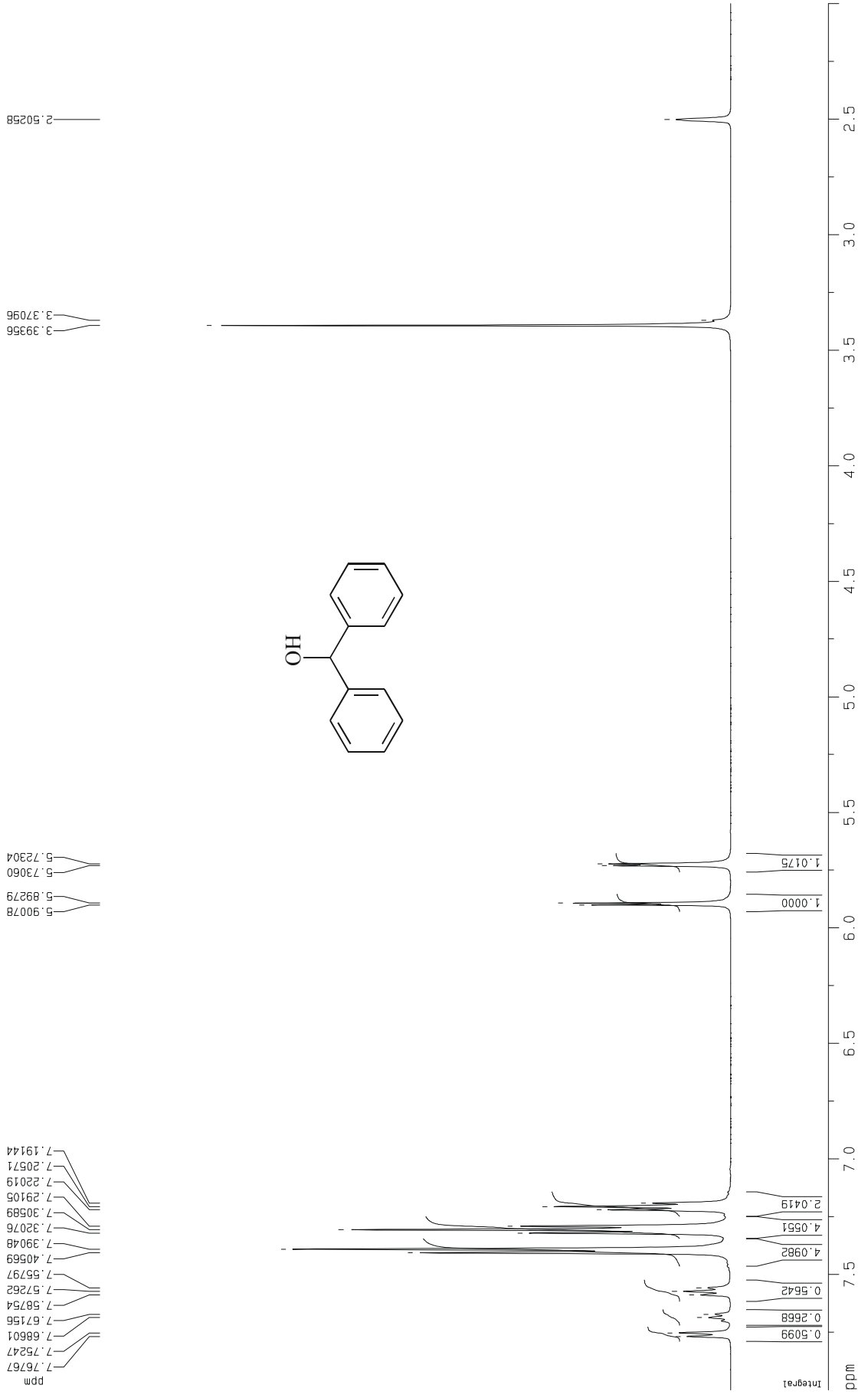
49. Benzil ^{1}H-NMR spektrum ($CDCl_3$)



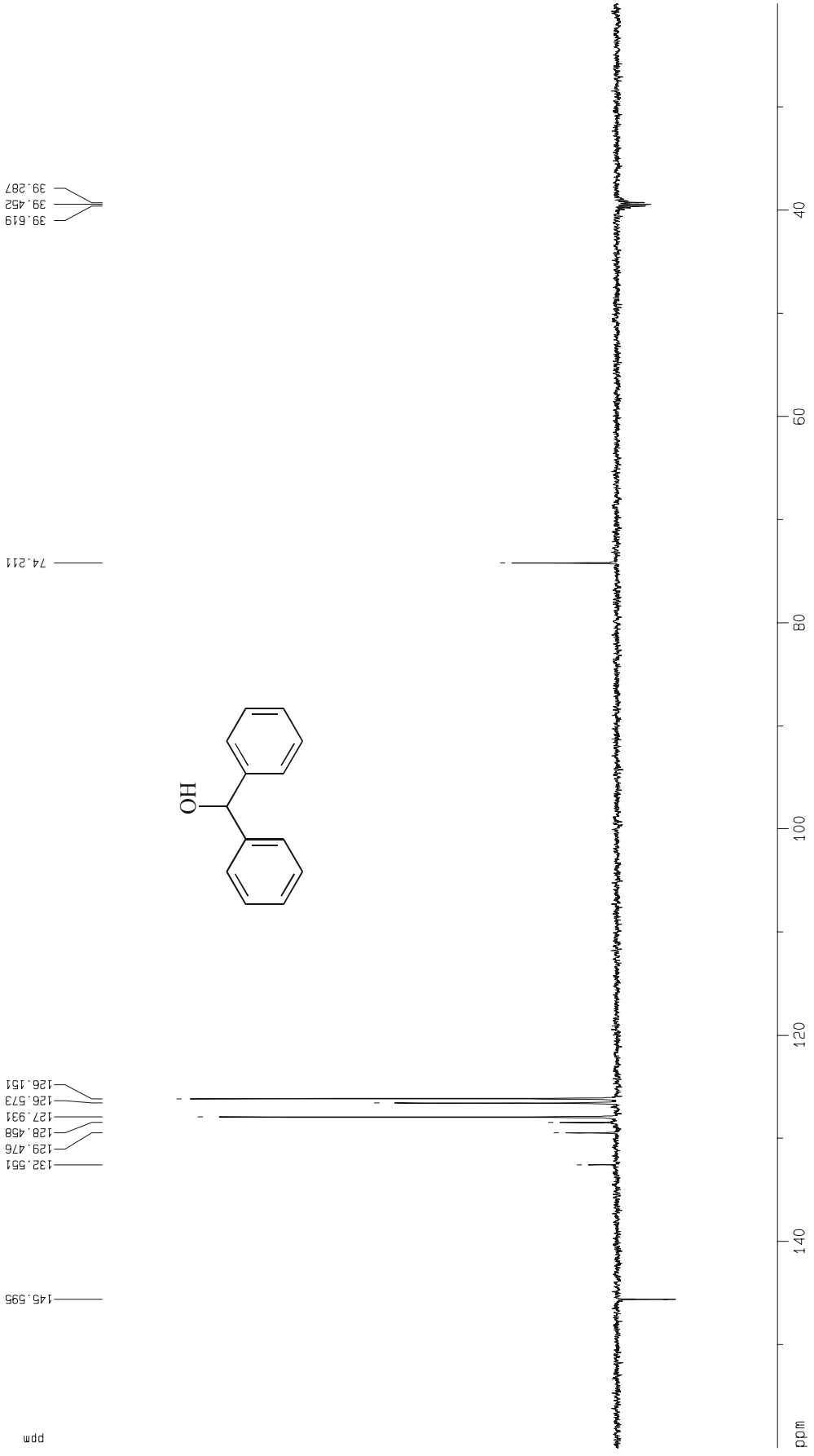
50. Benzil ^{13}C-JMOD NMR spektrum ($CDCl_3$)



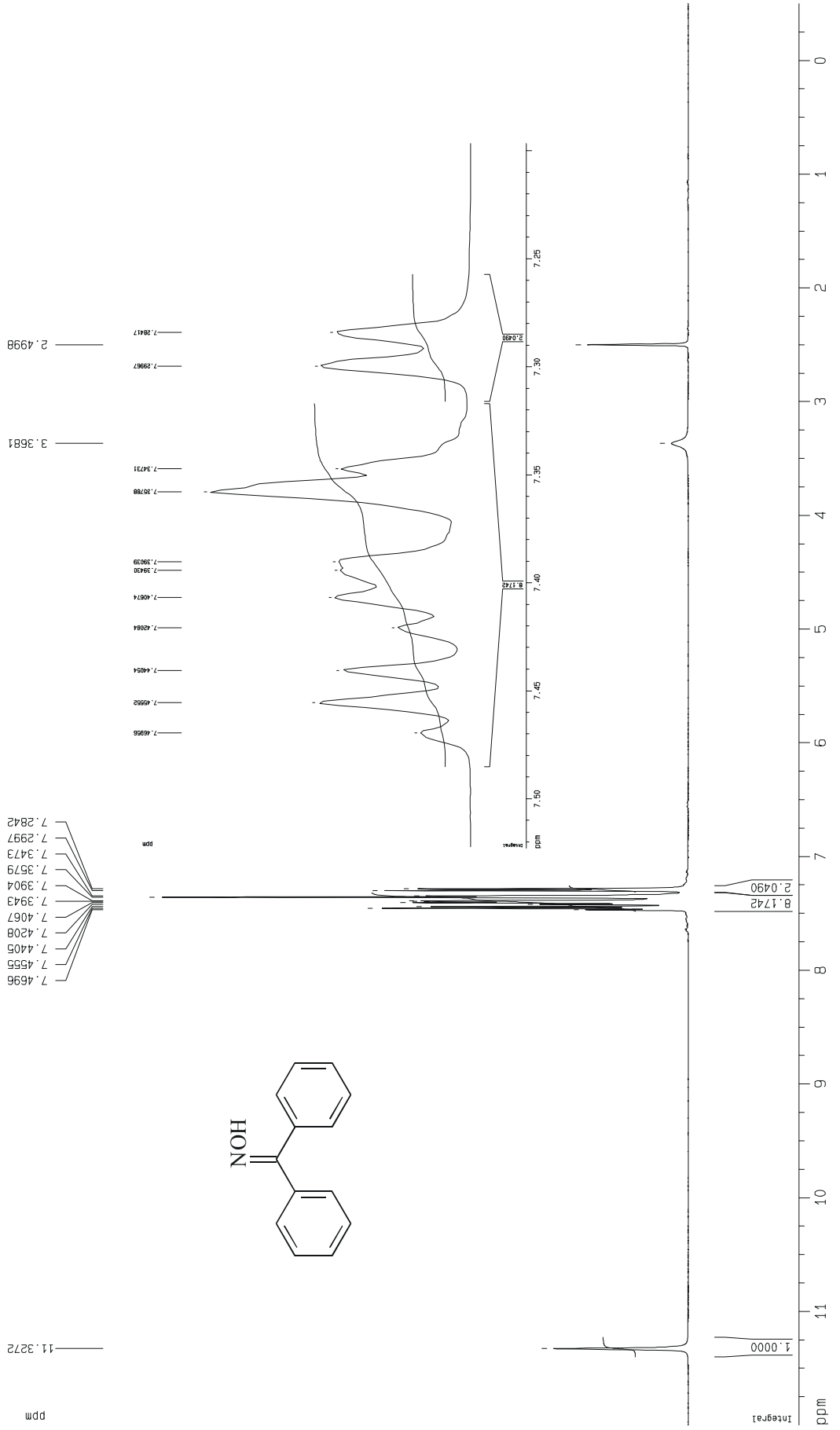
51. Benzhidrol ^{1}H-NMR spektrum (DMSO)



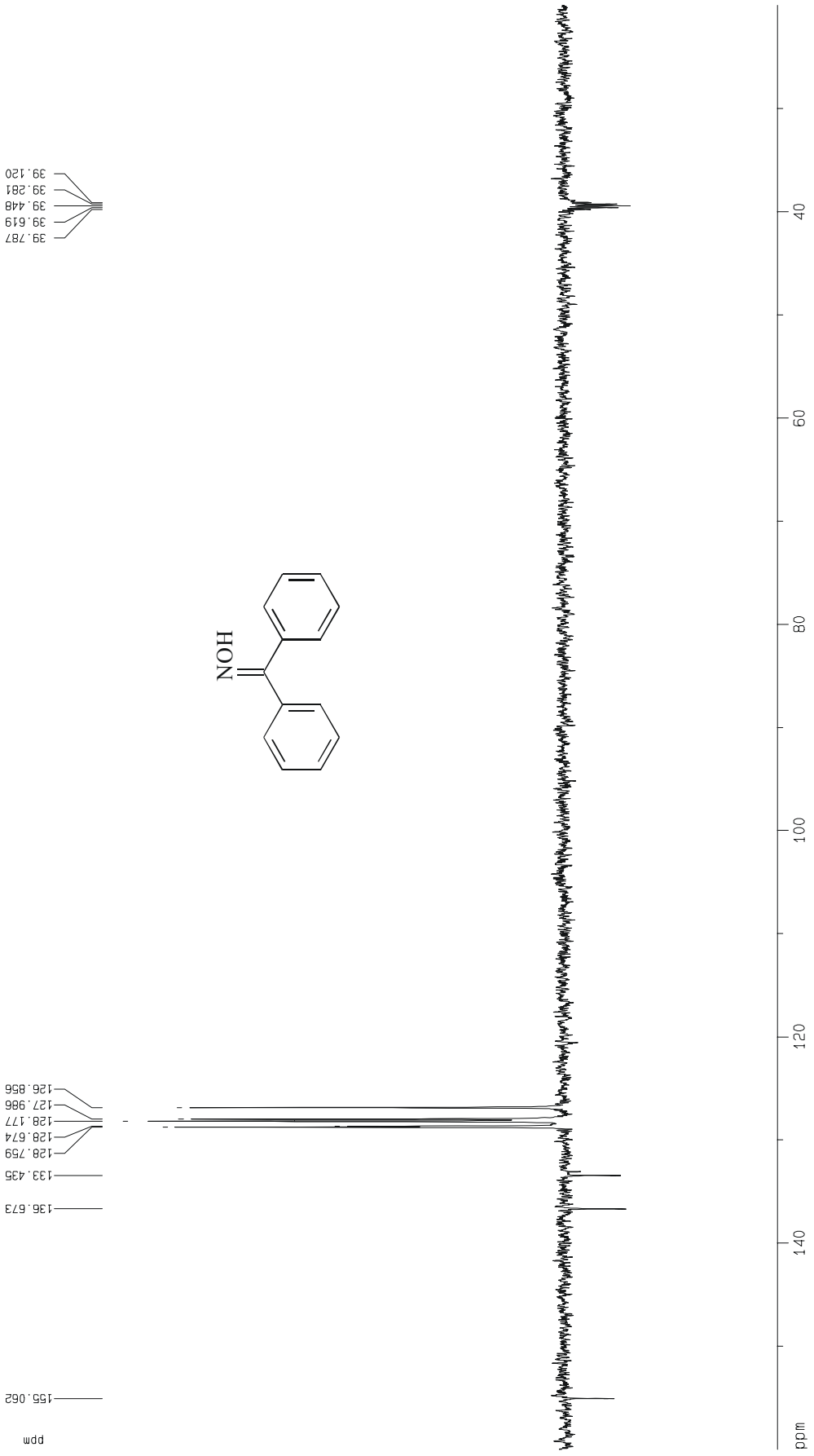
52. Benzhidrol ^{13}C-JMOD NMR spektrum (DMSO)



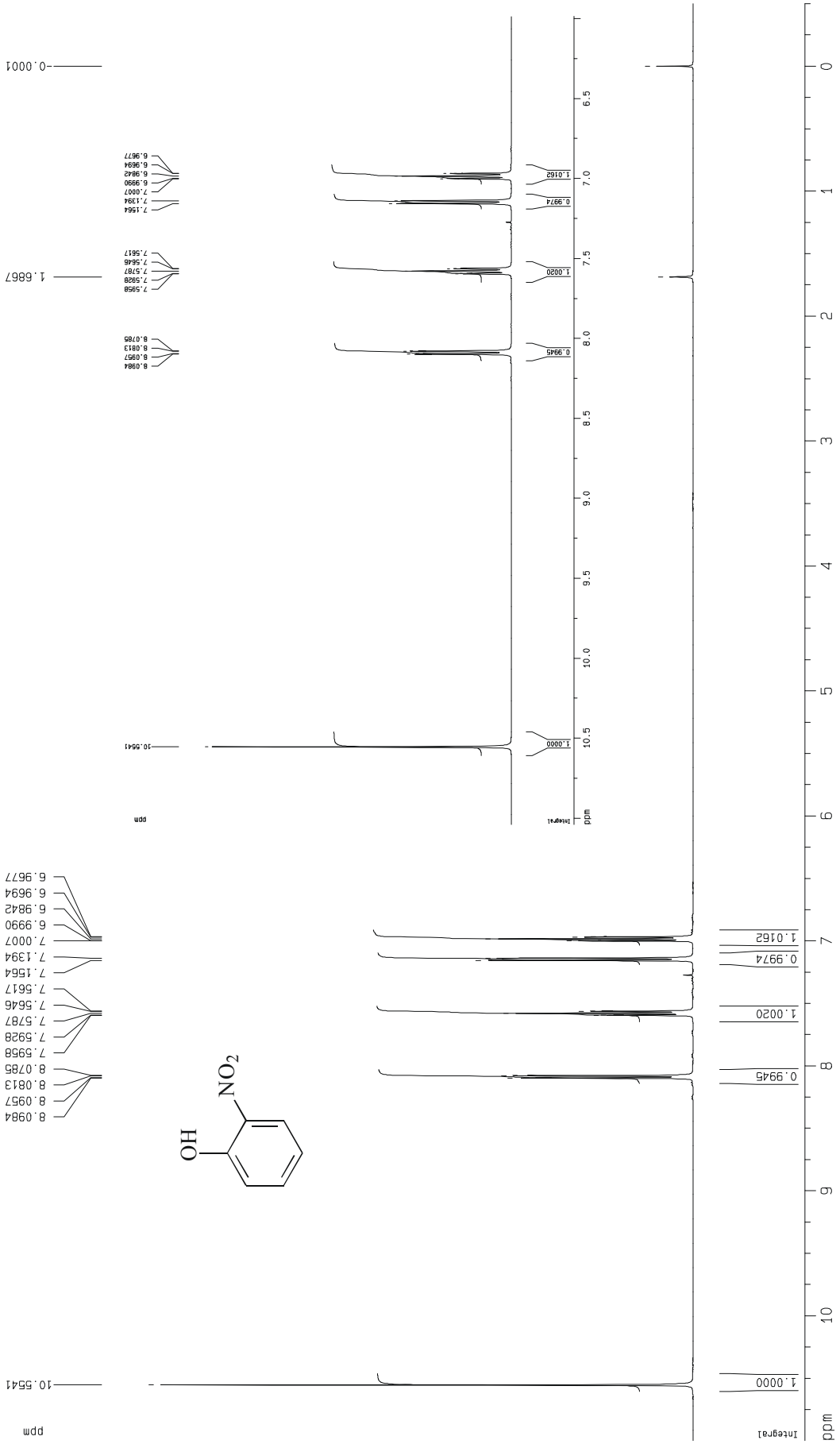
53. Benzofenon-oxim ^{1}H-NMR spektrum (DMSO)



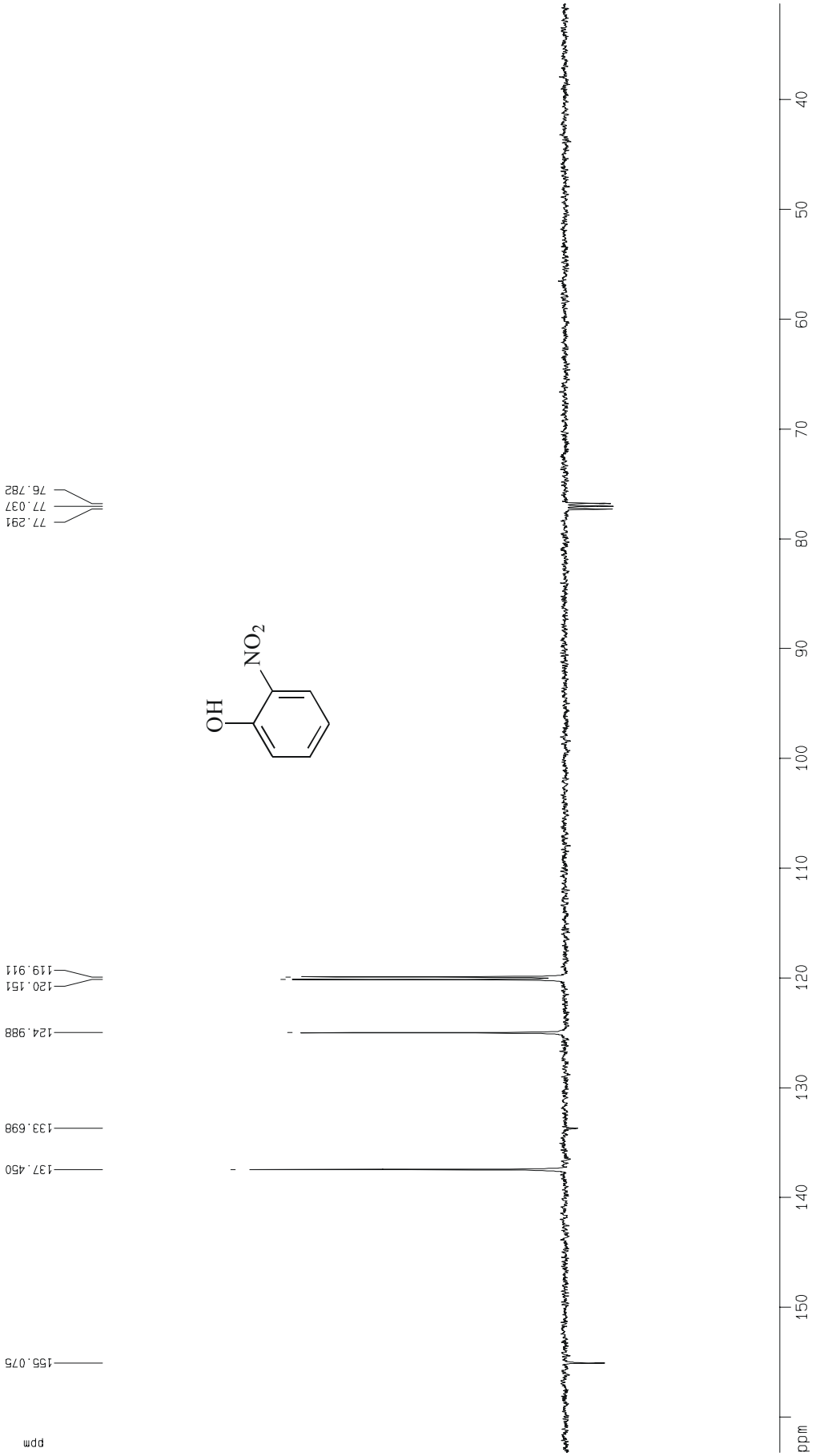
54. Benzofenon-oxim ^{13}C-JMOD NMR spektrum (DMSO)



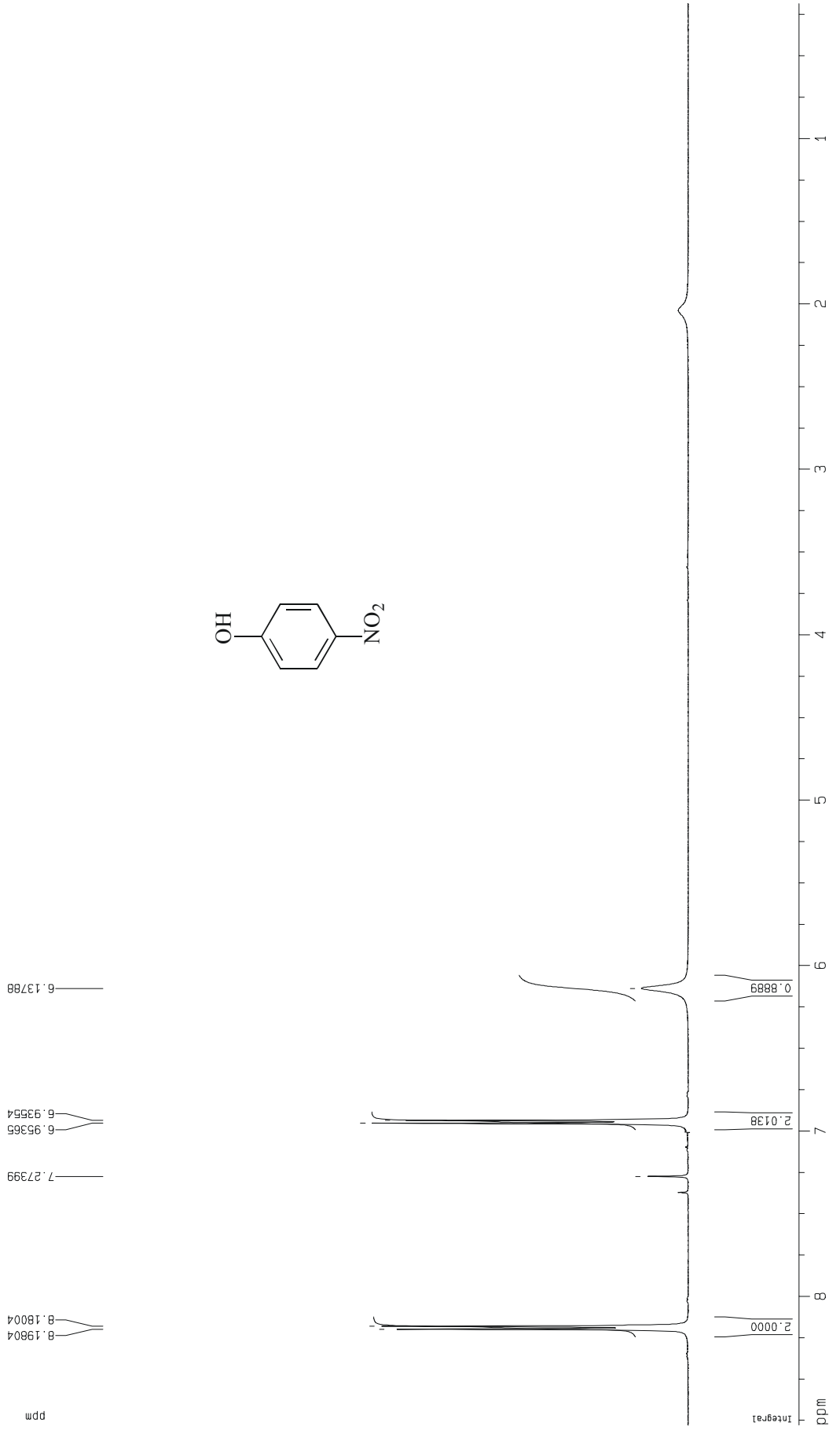
55. *orto*-Nitro-fenol ^{1}H-NMR spektrum ($CDCl_3$)



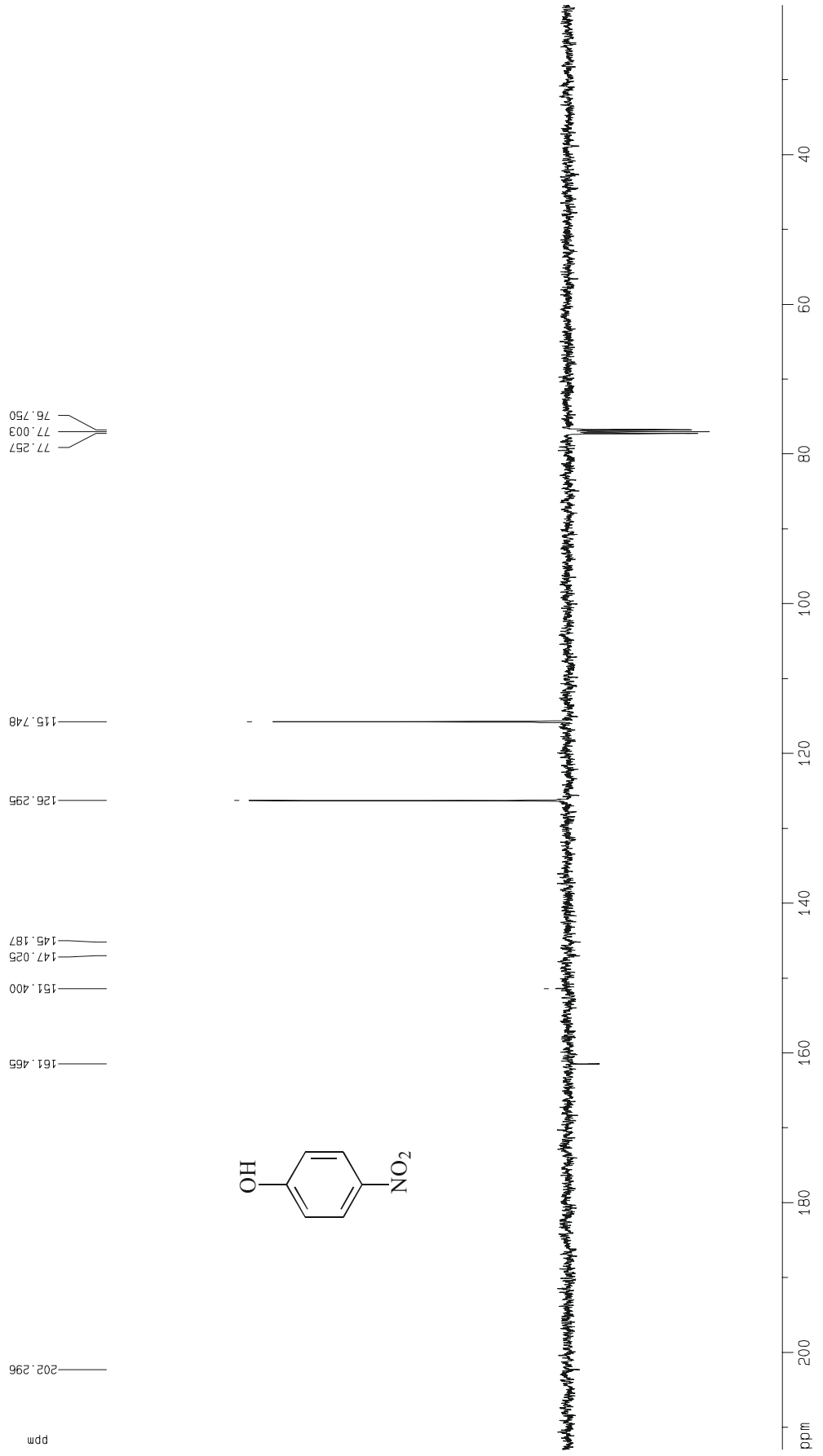
56. *orto*-Nitro-fenol ^{13}C-JMOD NMR spektrum ($CDCl_3$)



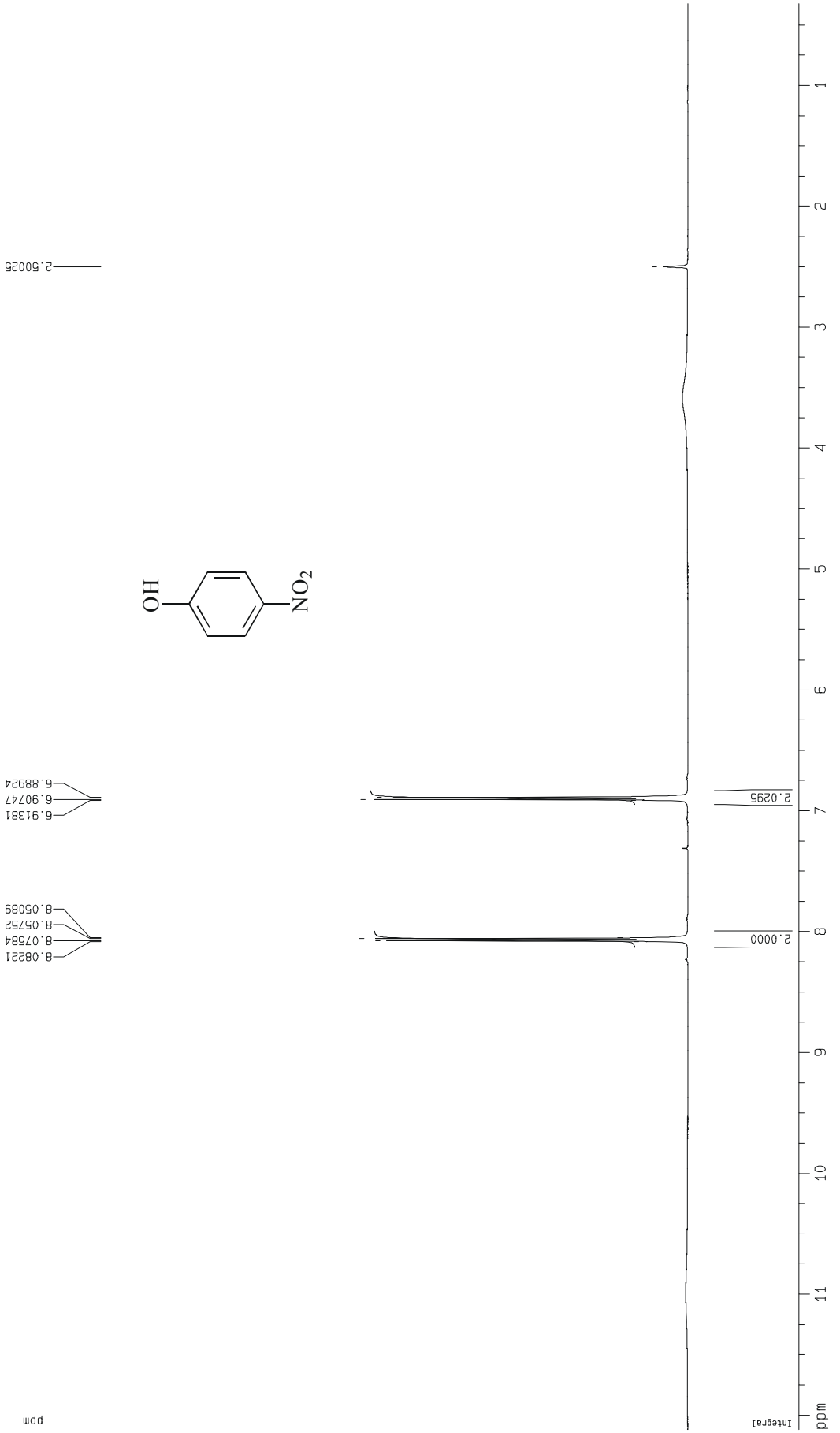
57. *para*-Nitro-fenol ^{1}H-NMR spektrum ($CDCl_3$)



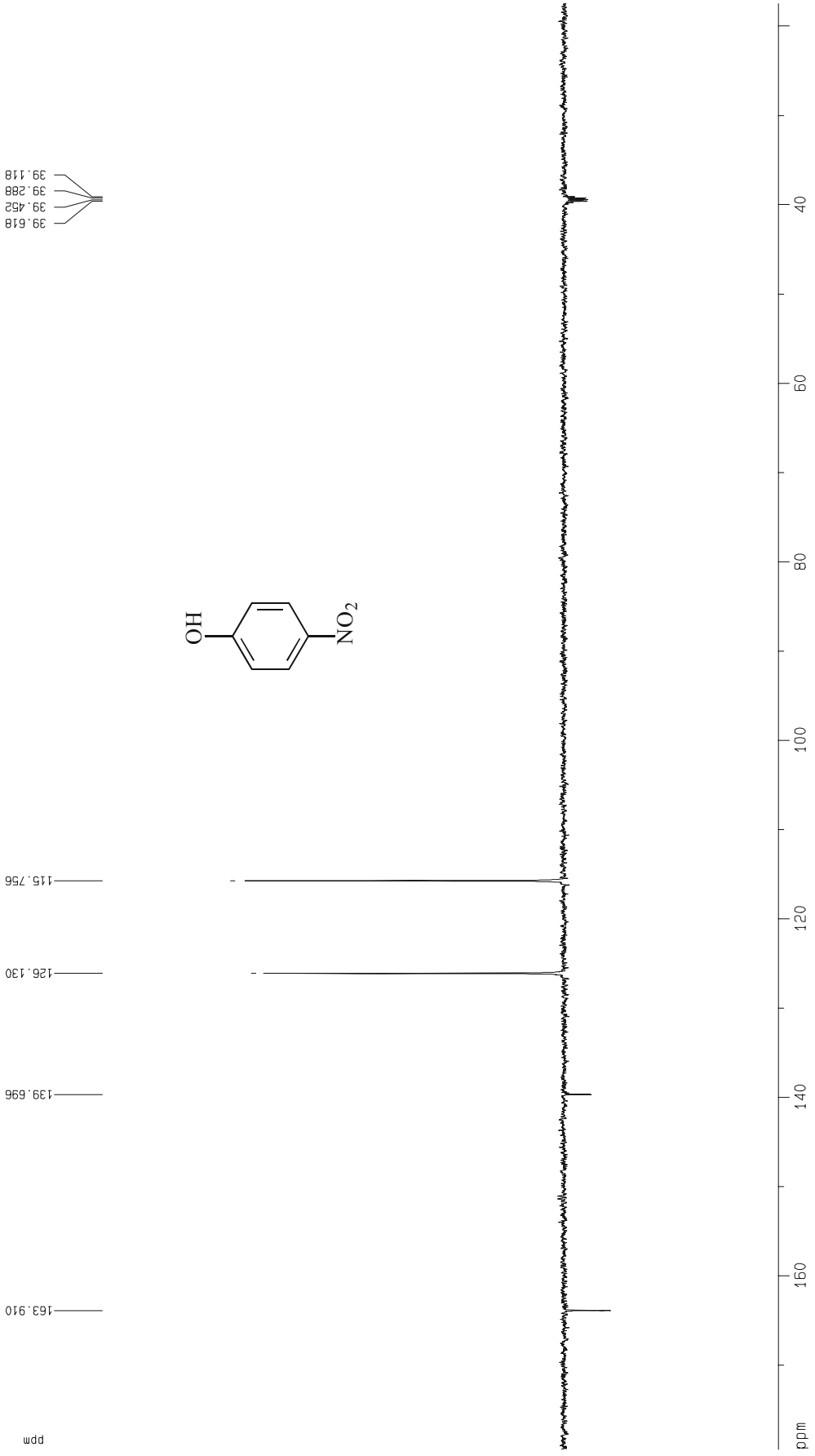
58. *para*-Nitro-fenol ^{13}C-JMOD spektrum ($CDCl_3$)



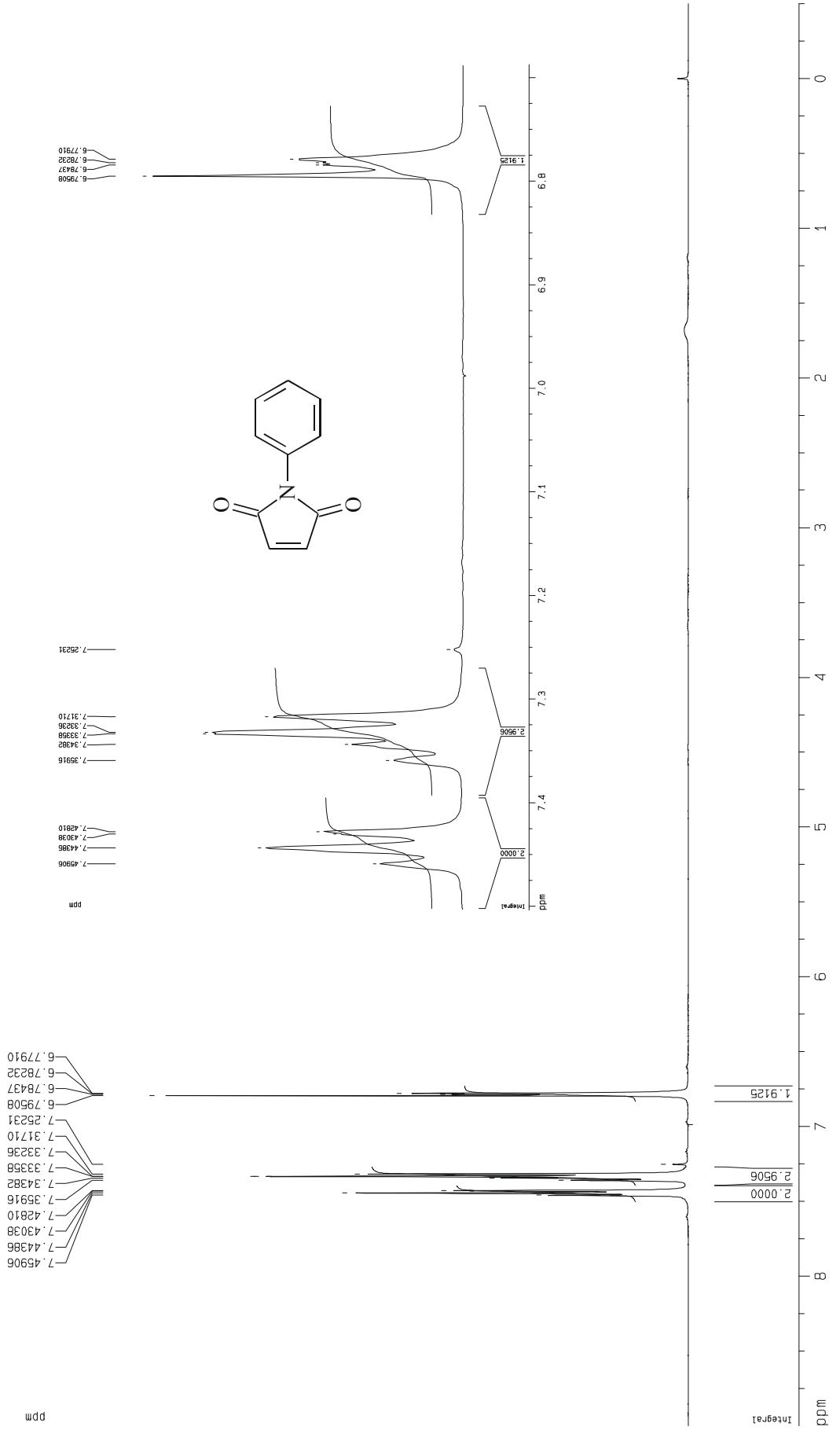
59. *para*-Nitro-fenol ^{1}H-NMR spektrum (DMSO)



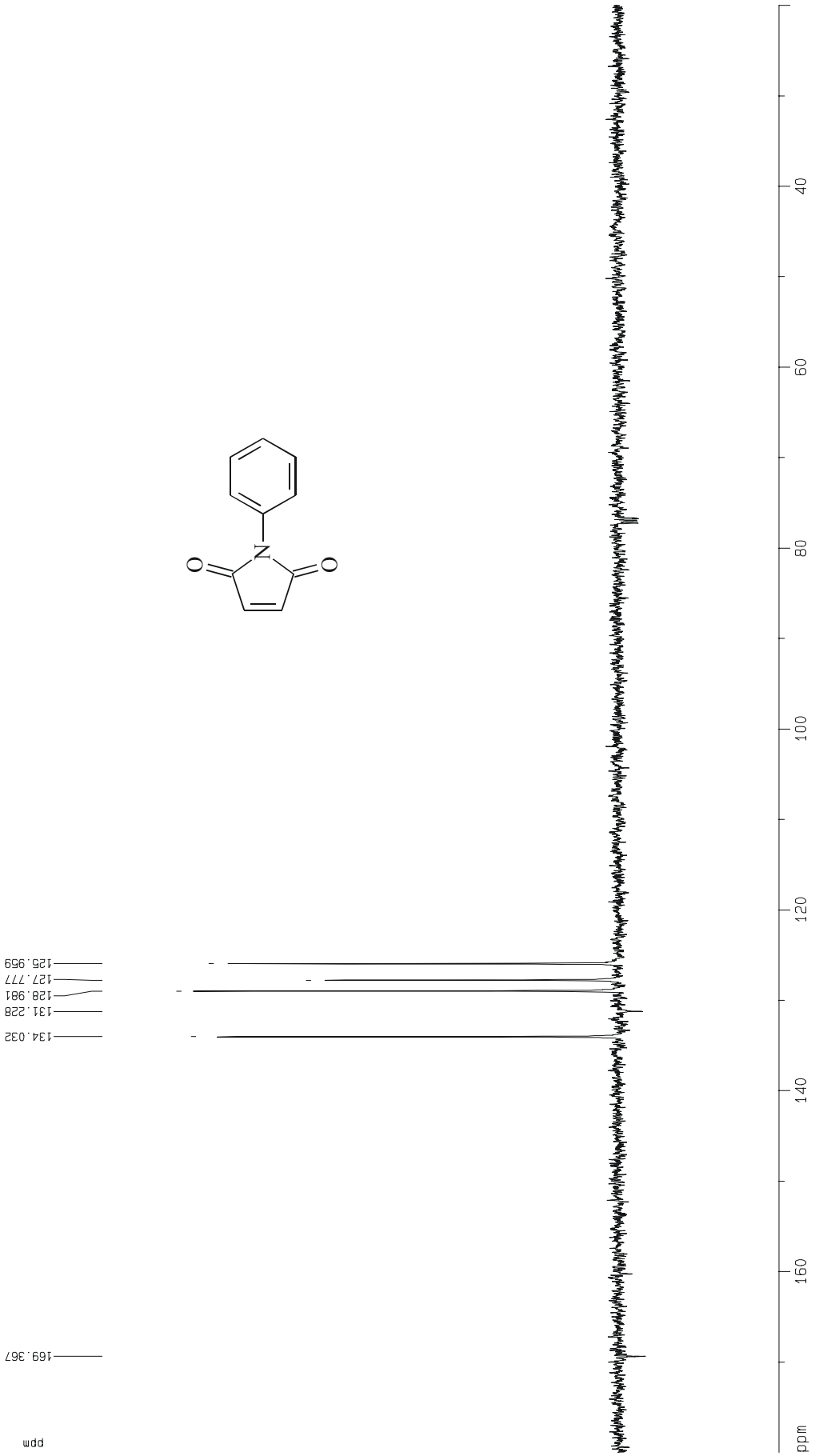
60. *para*-Nitro-fenol ^{13}C-JMOD spektrum (DMSO)



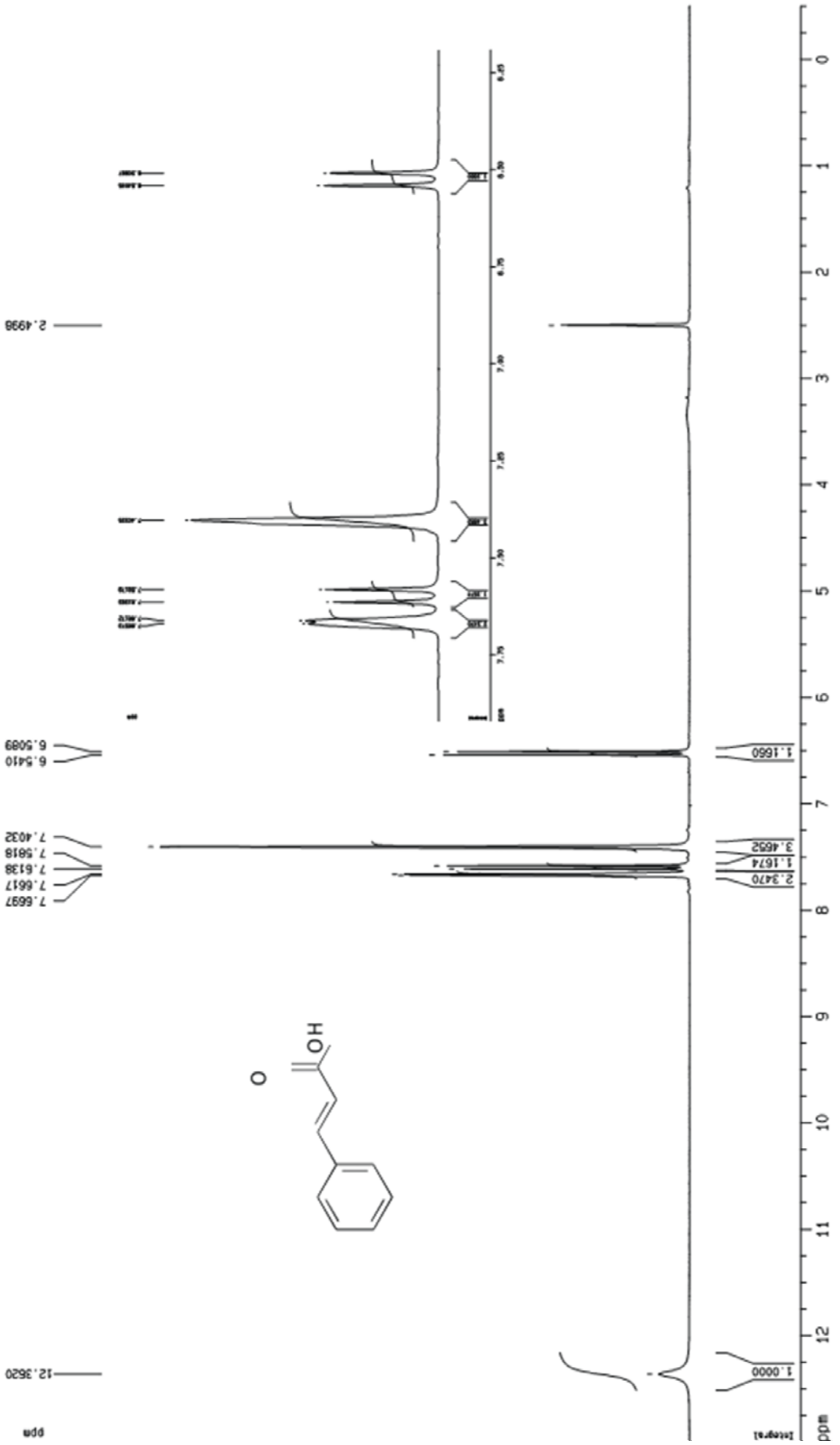
61. *N*-Fenil-maleimid ^{1}H-NMR spektrum (DMSO)



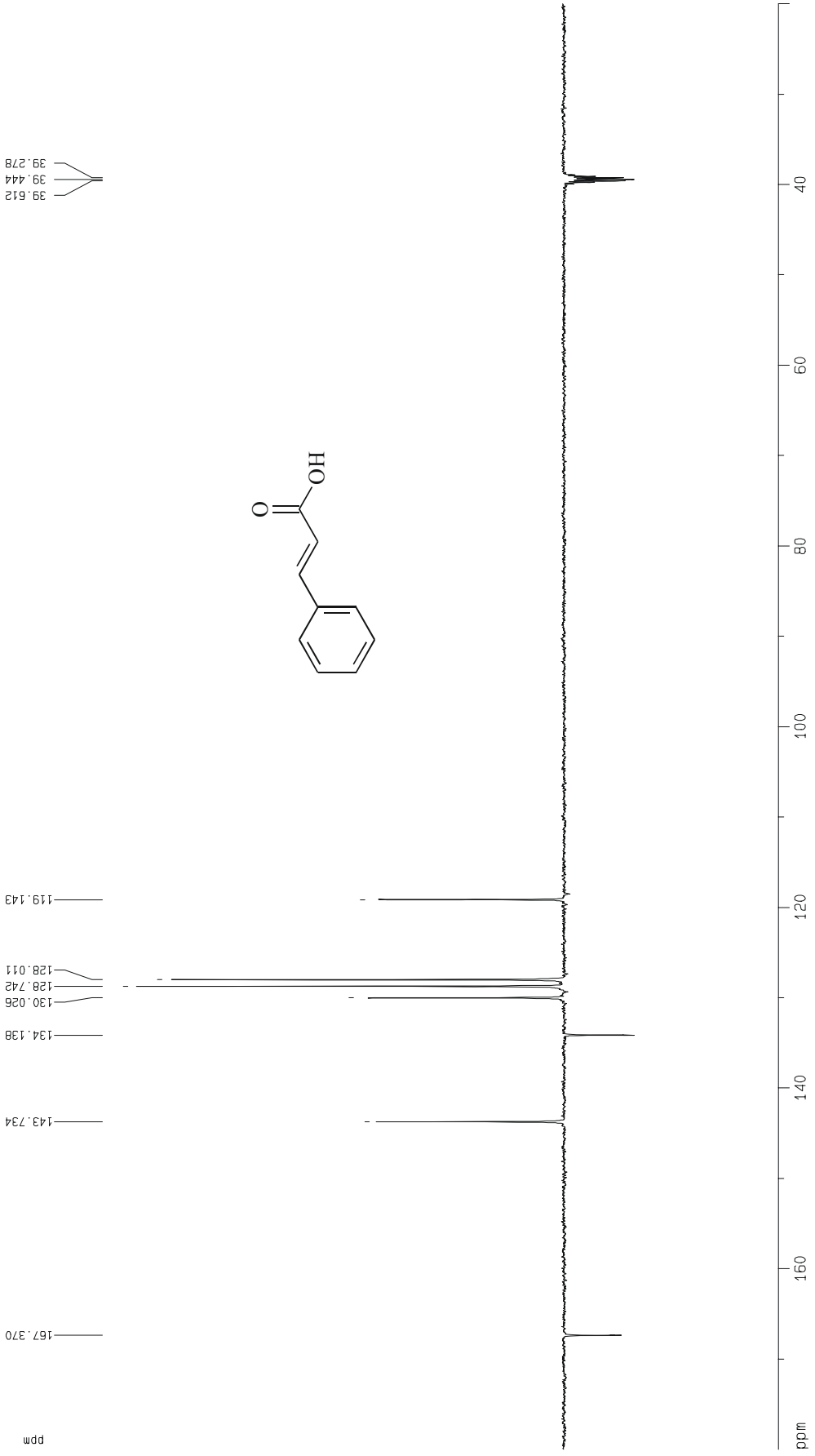
62. *N*-Fenil-maleimid ^{13}C-JMOD NMR spektrum (DMSO)



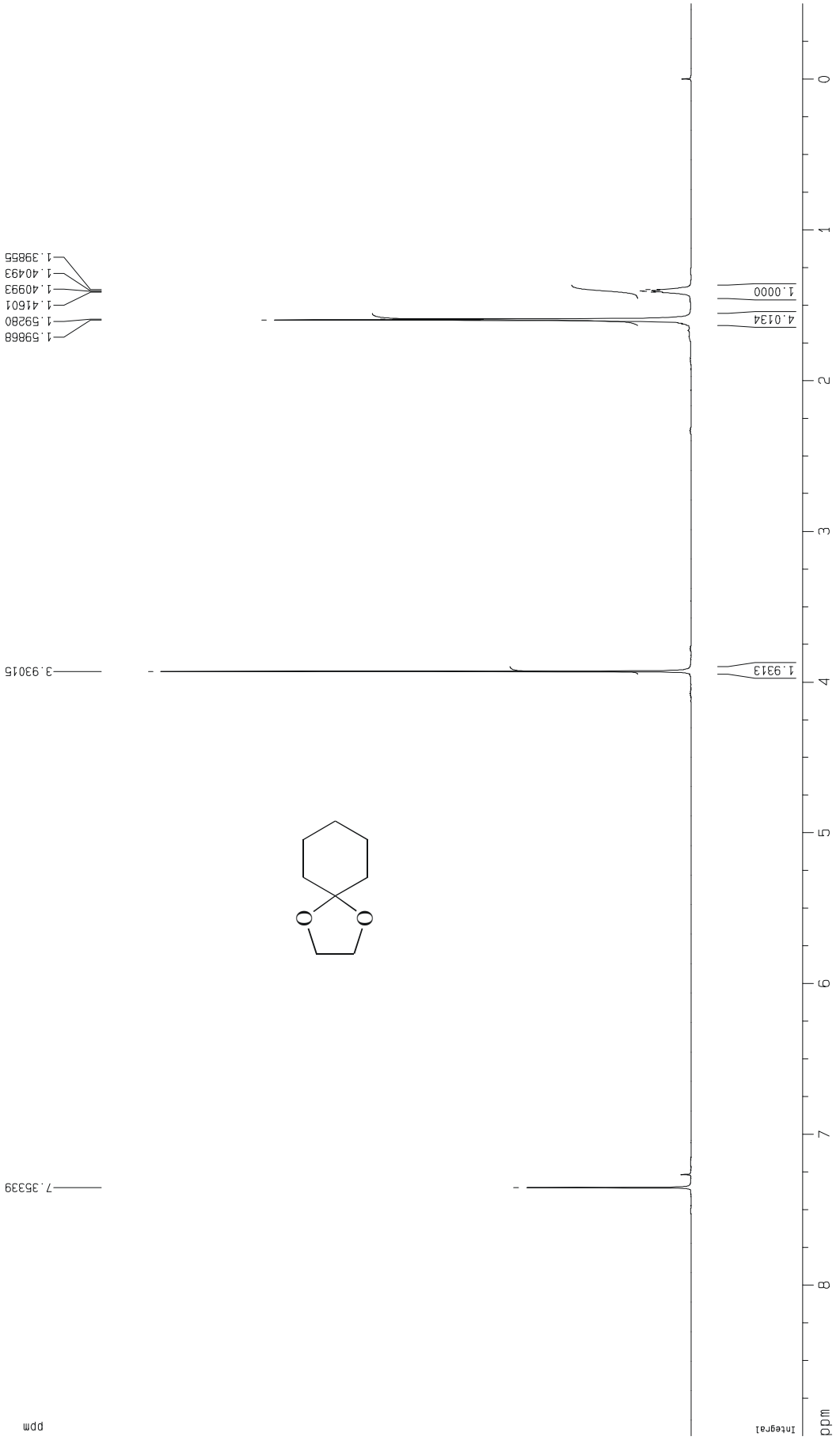
63. Fahéjsav ^{1}H-NMR spektrum ($CDCl_3$)



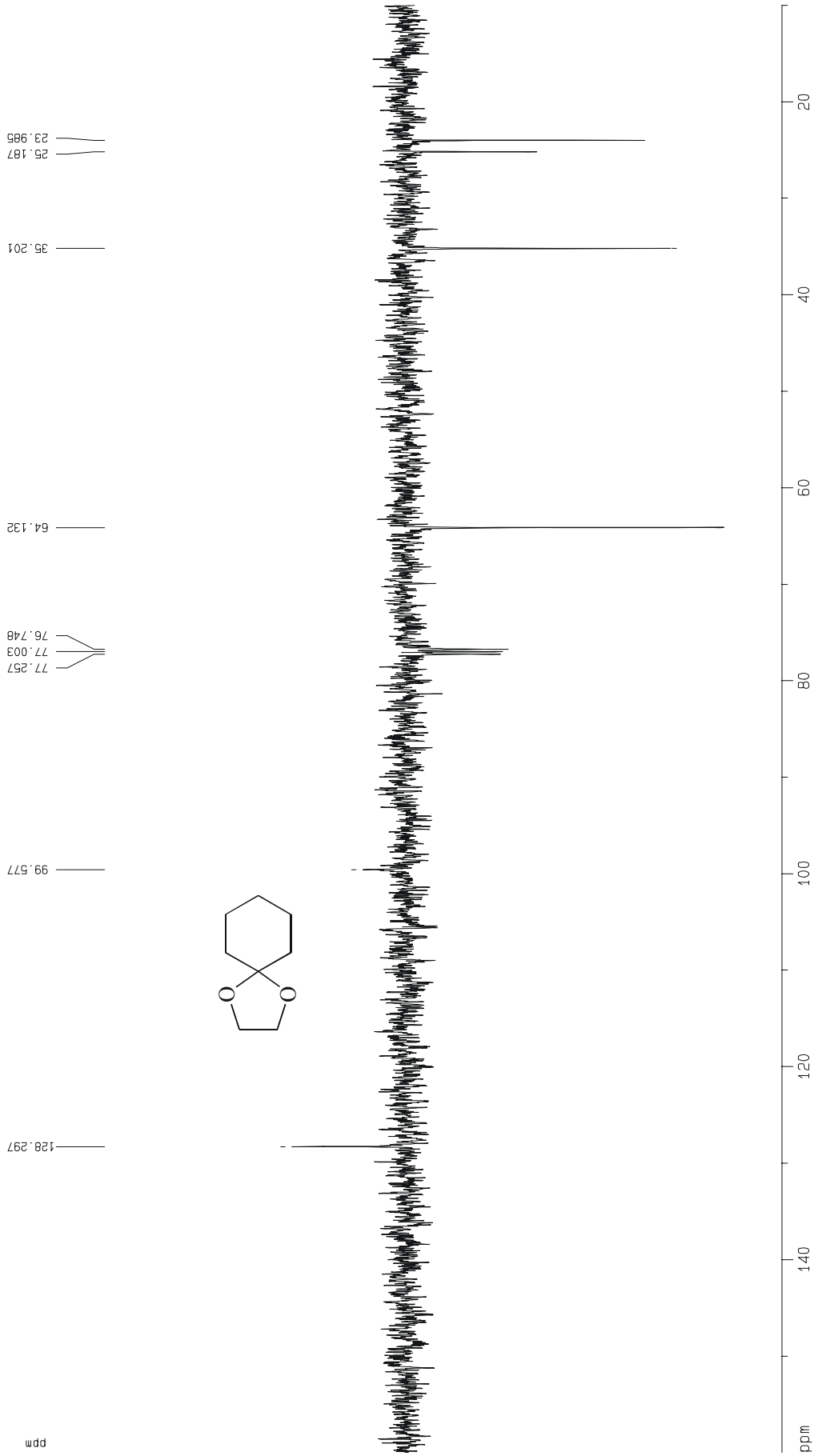
64. Fahéjsav ^{13}C-JMOD NMR spektrum ($CDCl_3$)



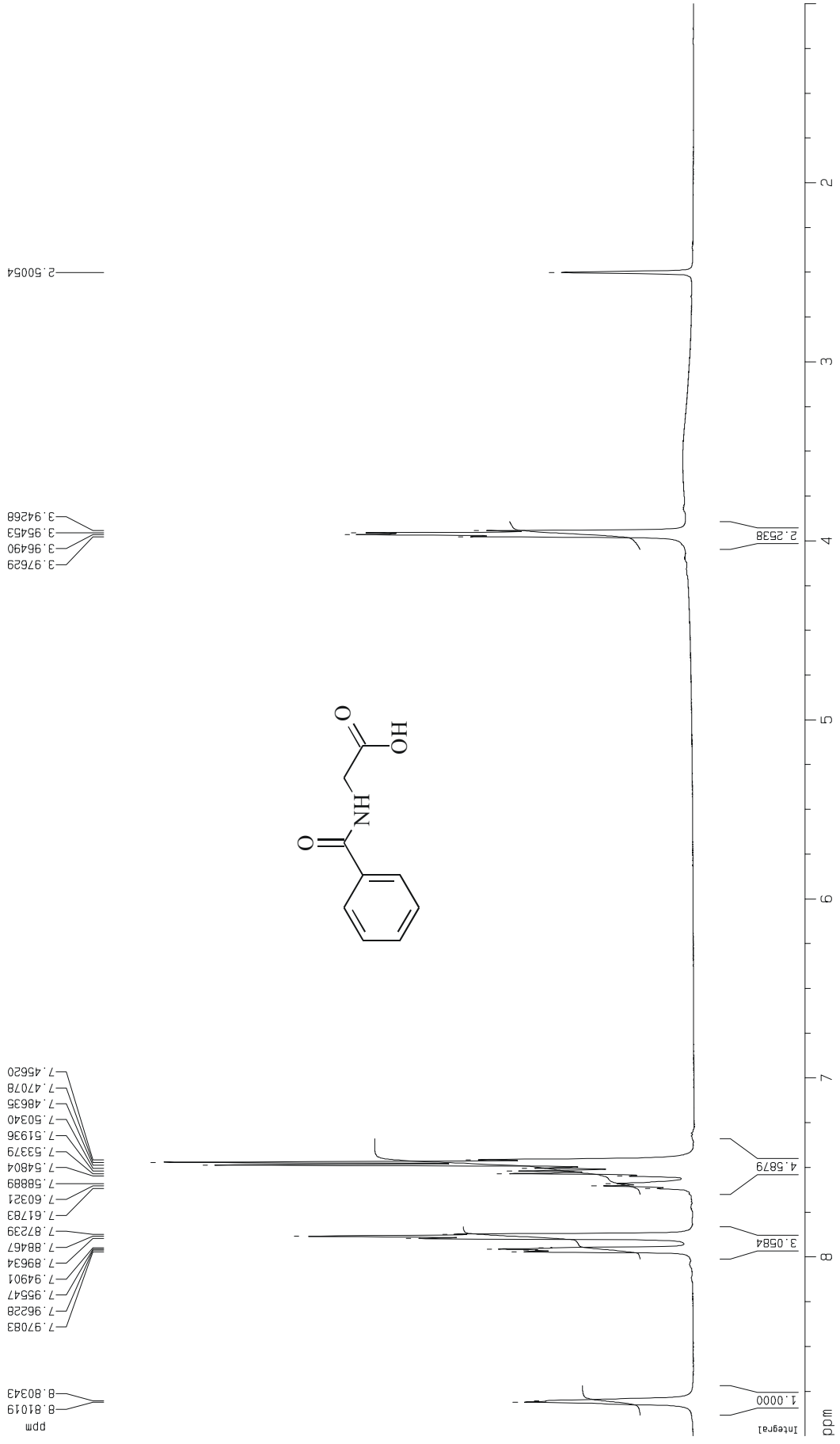
65. 1,4-Dioxaspiro[4,5]dekán ^{1}H-NMR spektrum ($CDCl_3$)



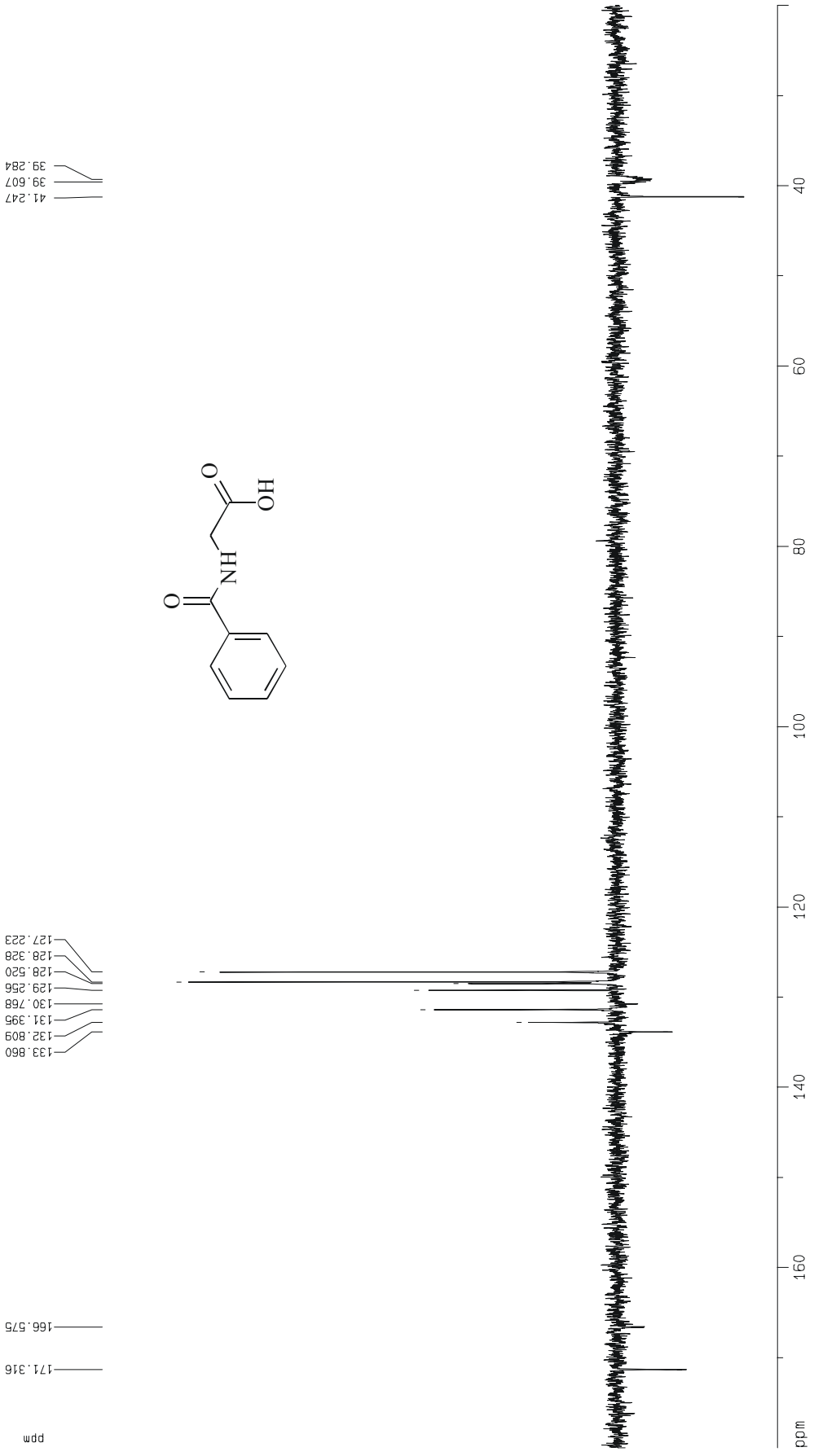
66. 1,4-Dioxaspiro[4,5]dekán ^{13}C-JMOD NMR spektrum ($CDCl_3$)



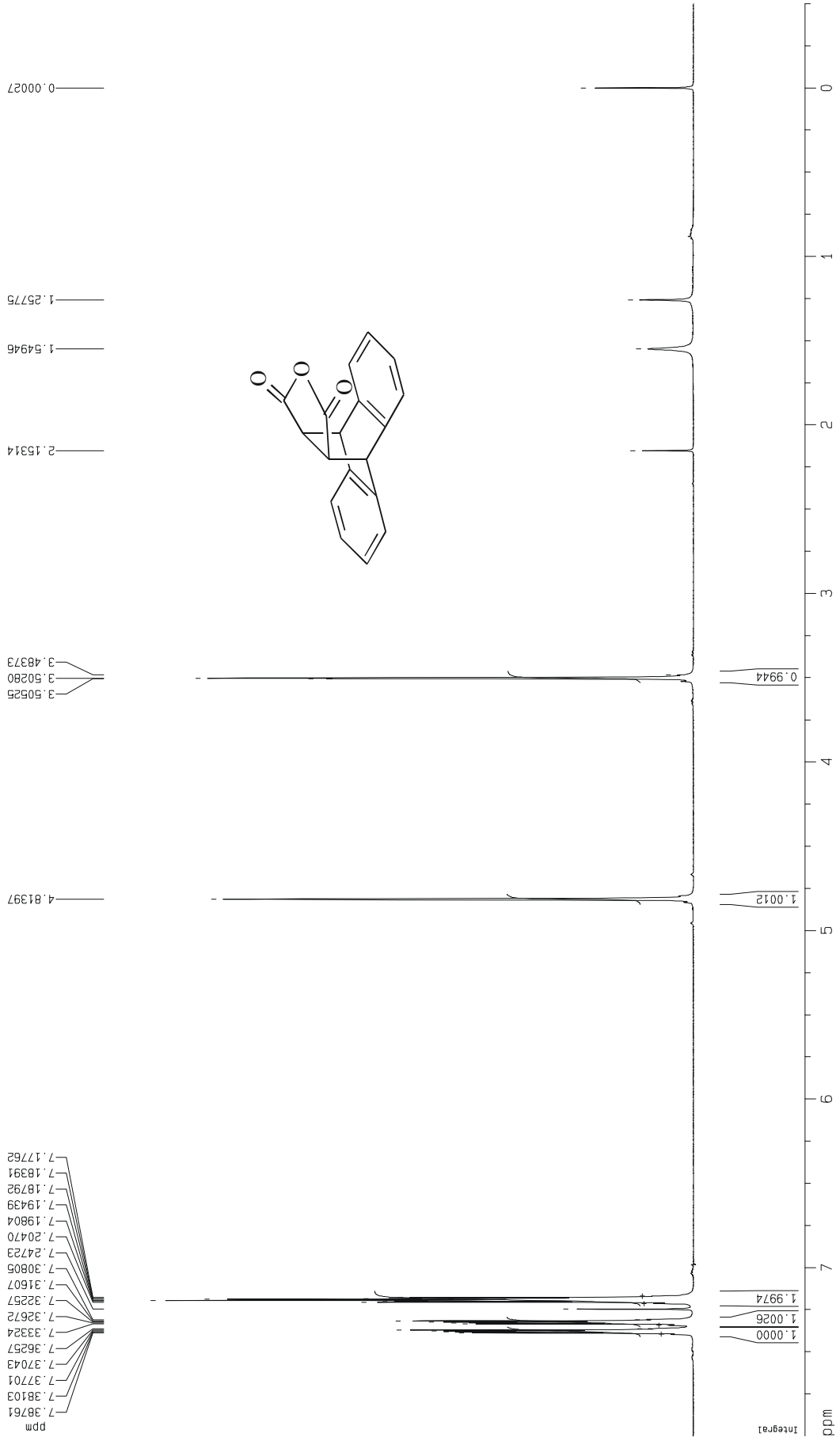
67. Hippursav ^{1}H-NMR spektrum (DMSO)



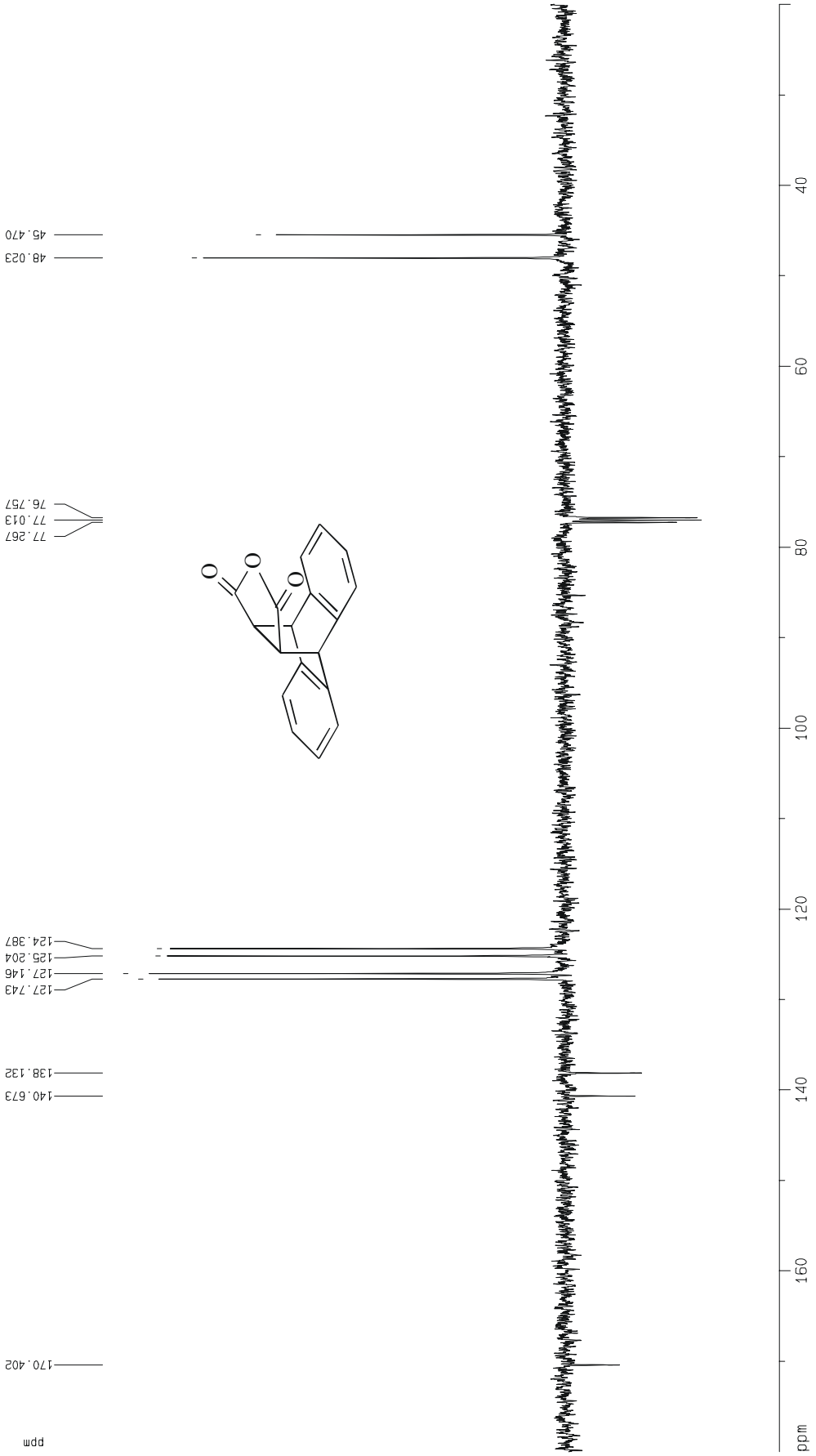
68. Hippursav ^{13}C-JMOD NMR spektrum (DMSO)



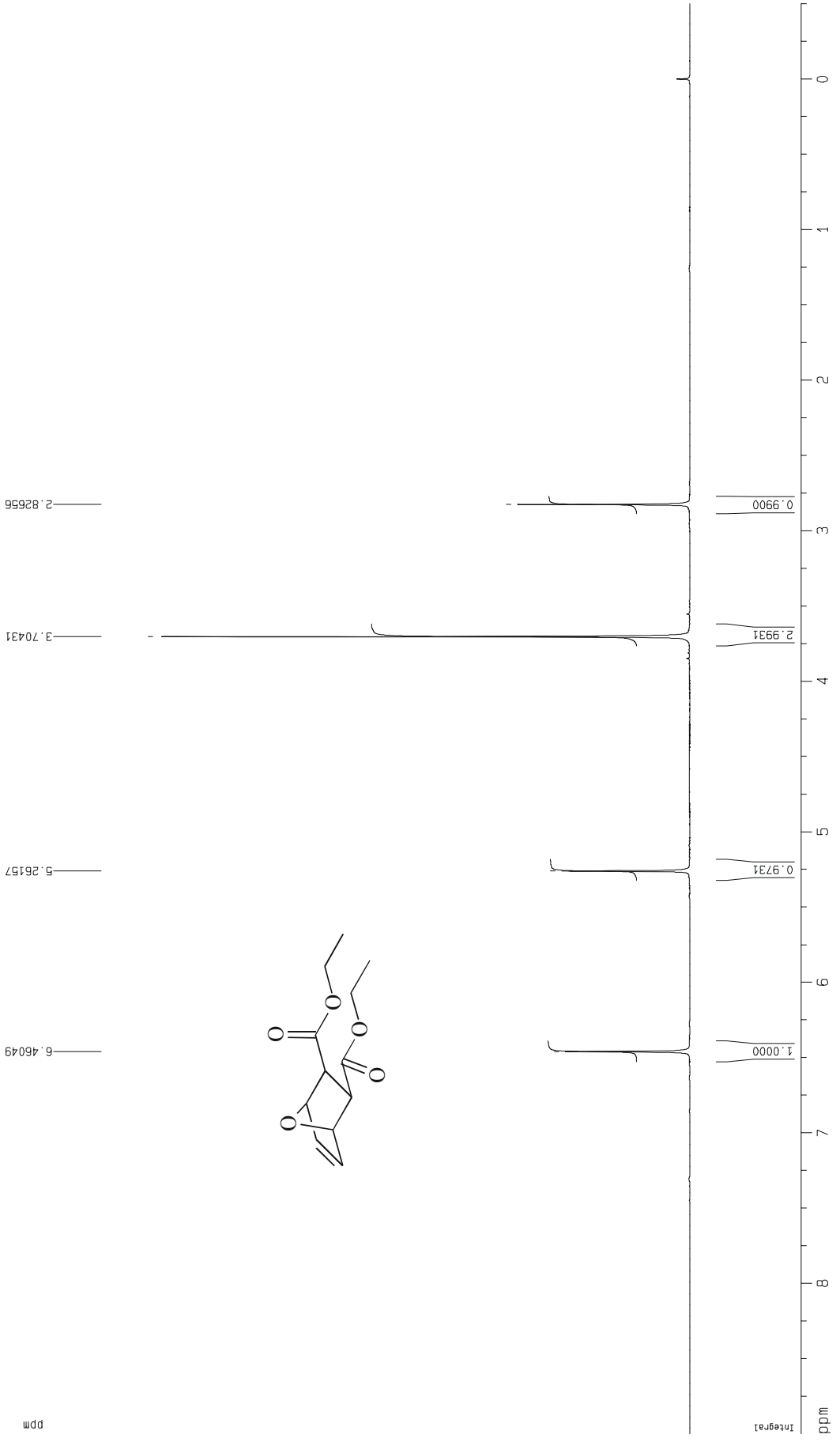
69. *cisz*-9,10-Dihidro-9,10-etano-antracen-11,12-dikarbonsav-anhidrid ^{1}H-NMR spektrum ($CDCl_3$)



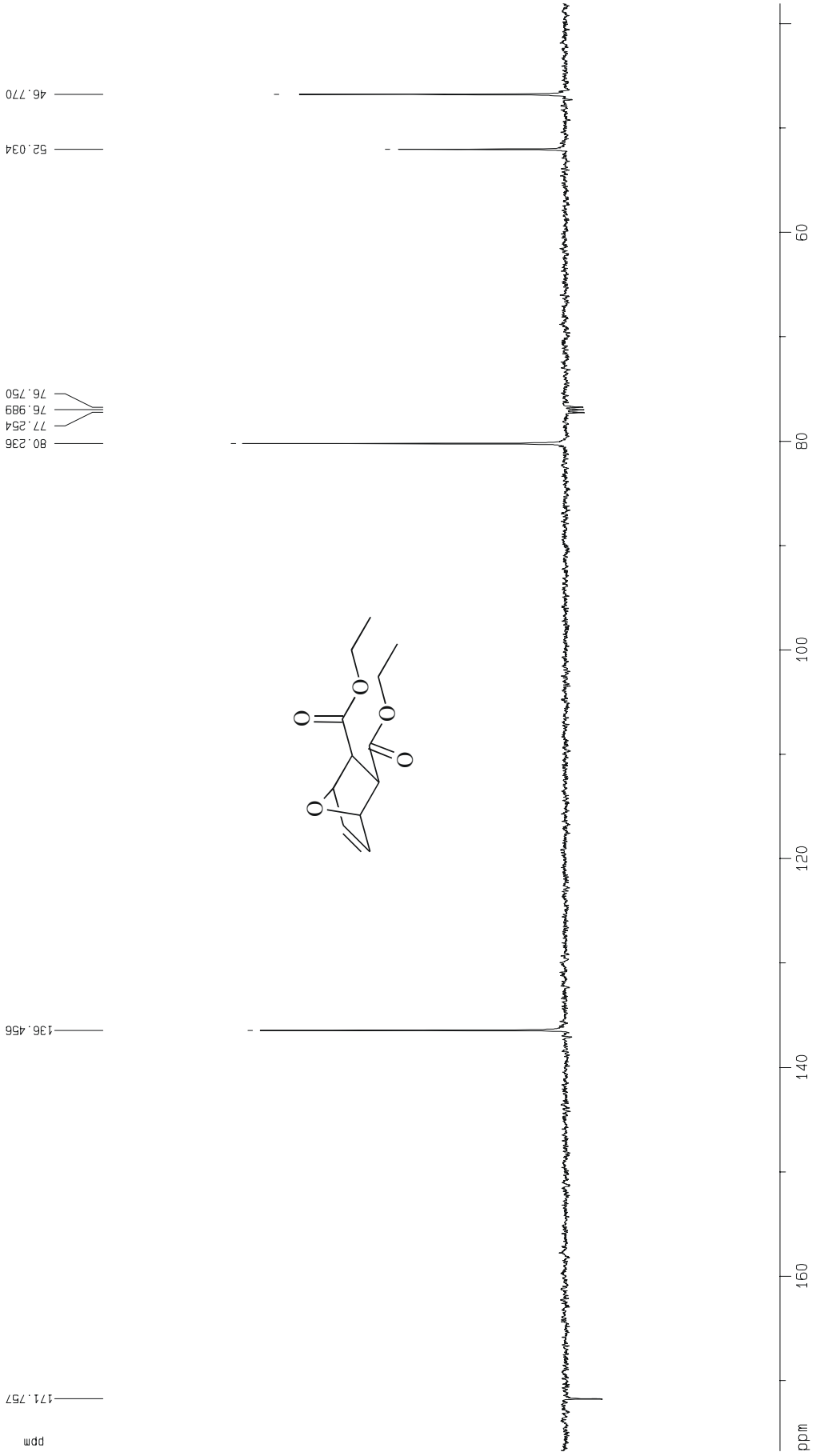
70. *cisz*-9,10-Dihidro-9,10-etano-antracen-11,12-dikarbonsav-anhidrid ^{13}C-JMOD NMR spektrum ($CDCl_3$)



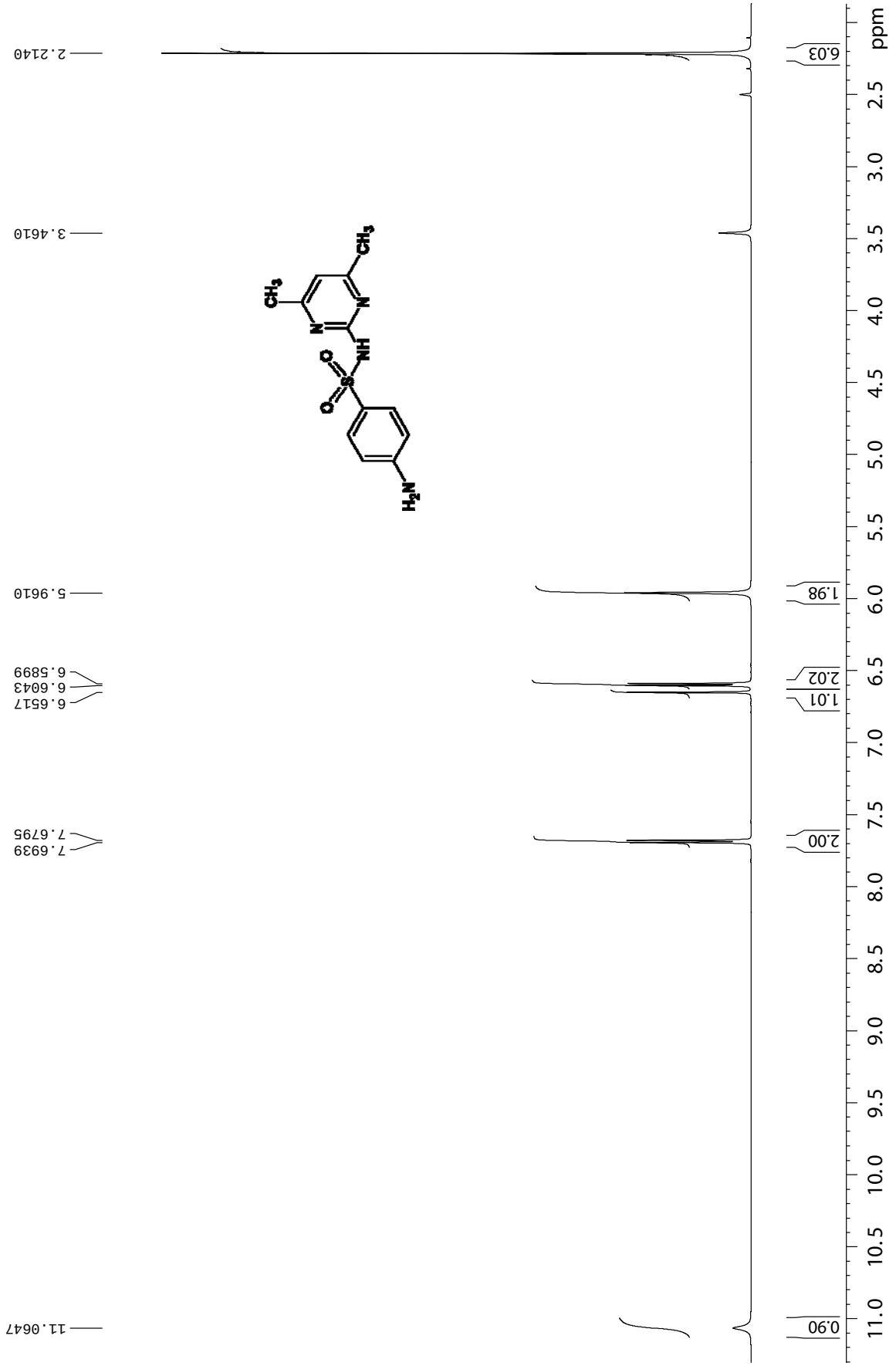
71.-7-oxabiciklo[2.2.1]hept-5-én-2*exo*,3*exo*-disav-dimetil-észter ^{1}H-NMR spektrum ($CDCl_3$)



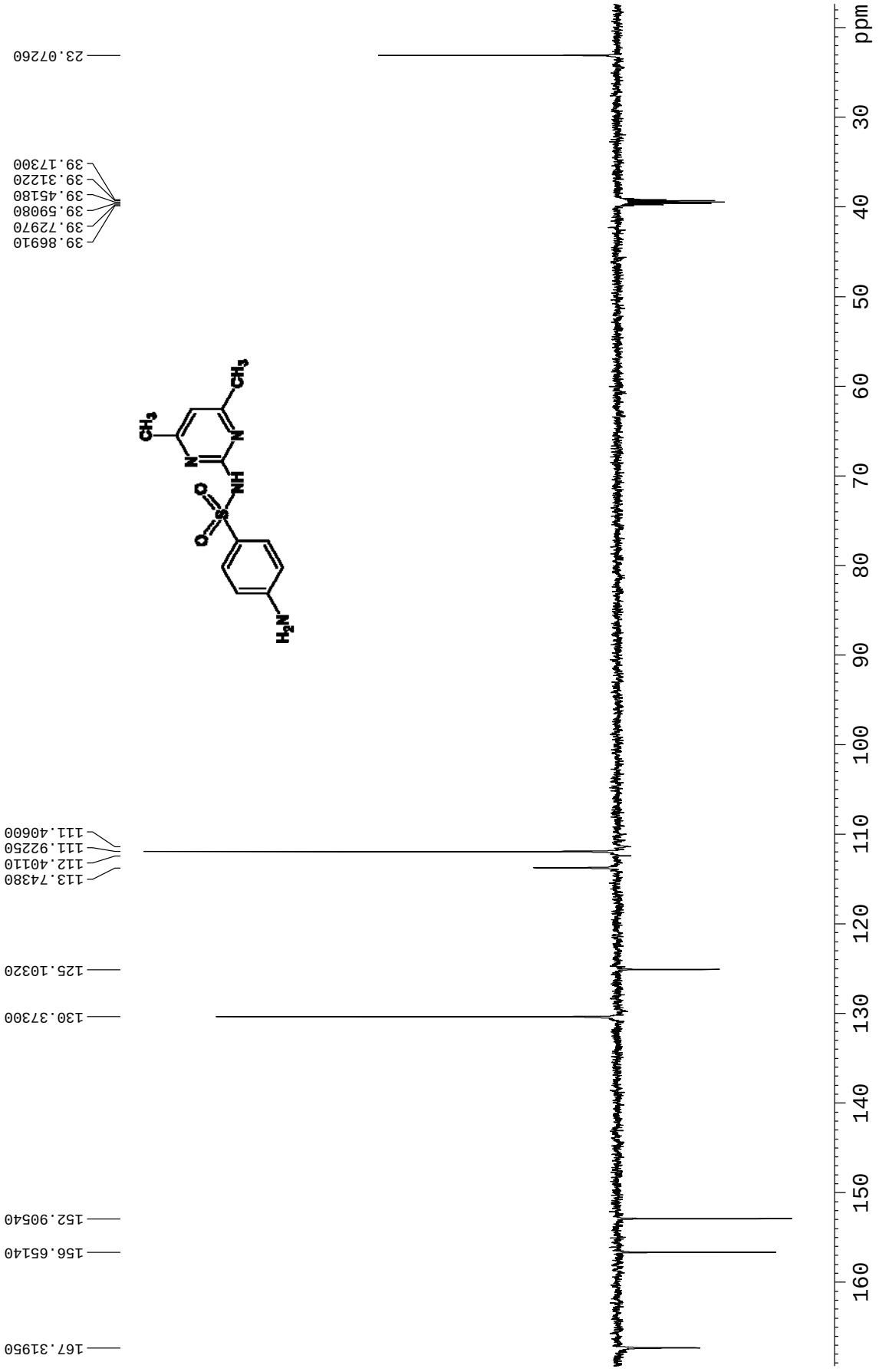
72. 7-oxabiciklo[2.2.1]hept-5-én-2*exo*,3*exo*-disav-dimetil-észter ^{13}C-JMOD spektrum NMR ($CDCl_3$)



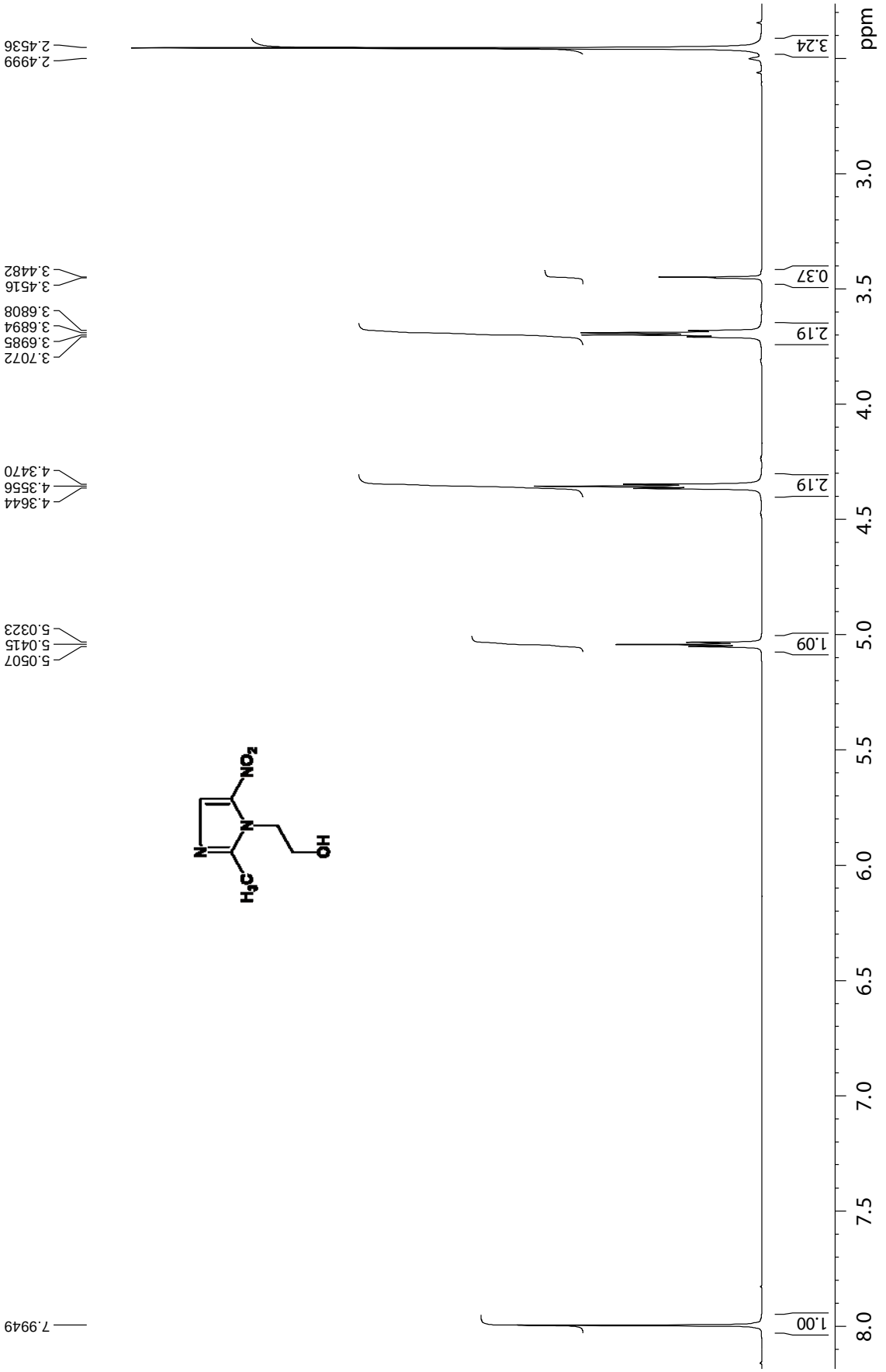
73. Sulfadimidin ^{1}H NMR (DMSO)



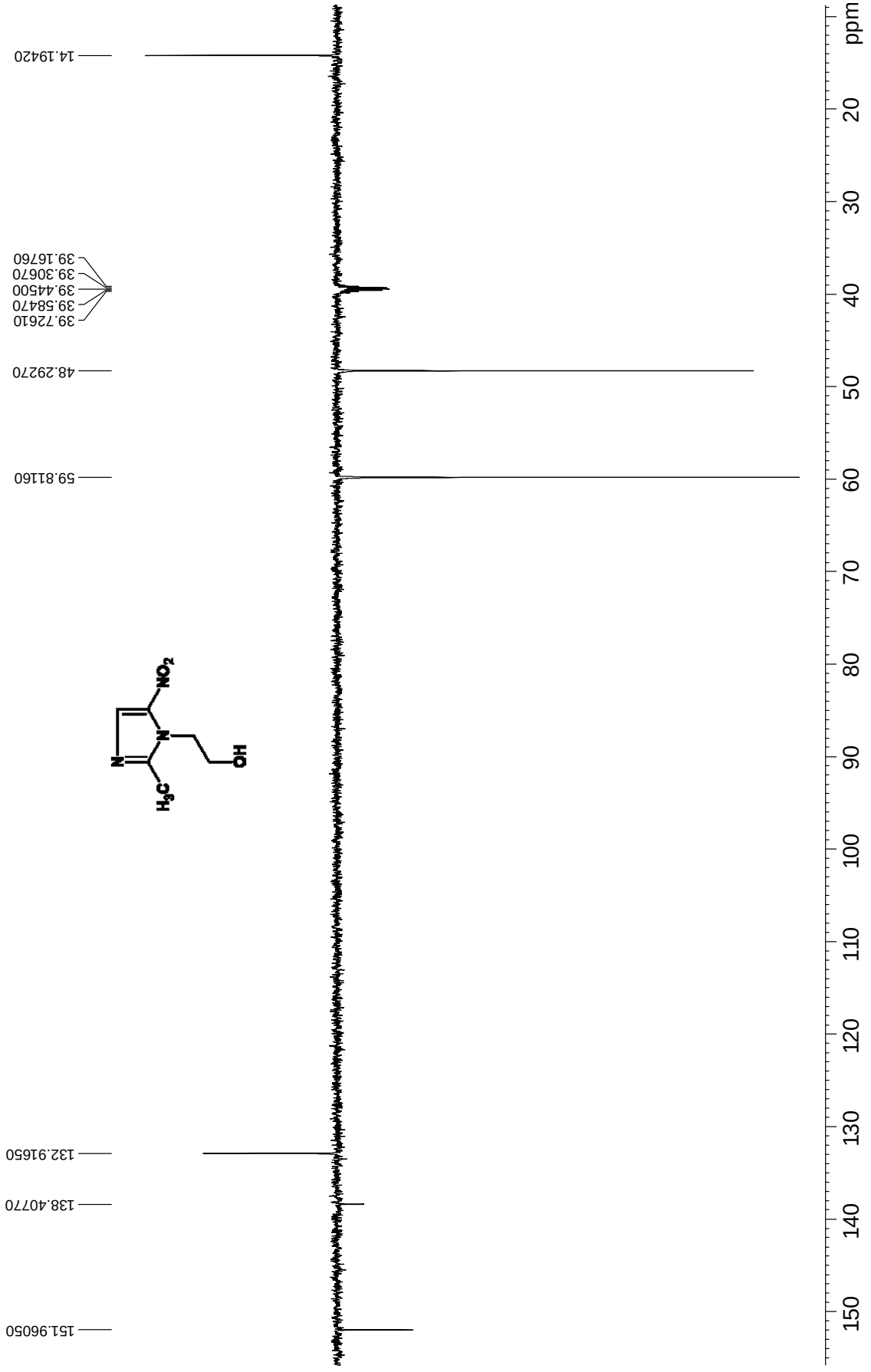
74. Sulfadimidin ^{13}C-JMOD NMR spektrum (DMSO)



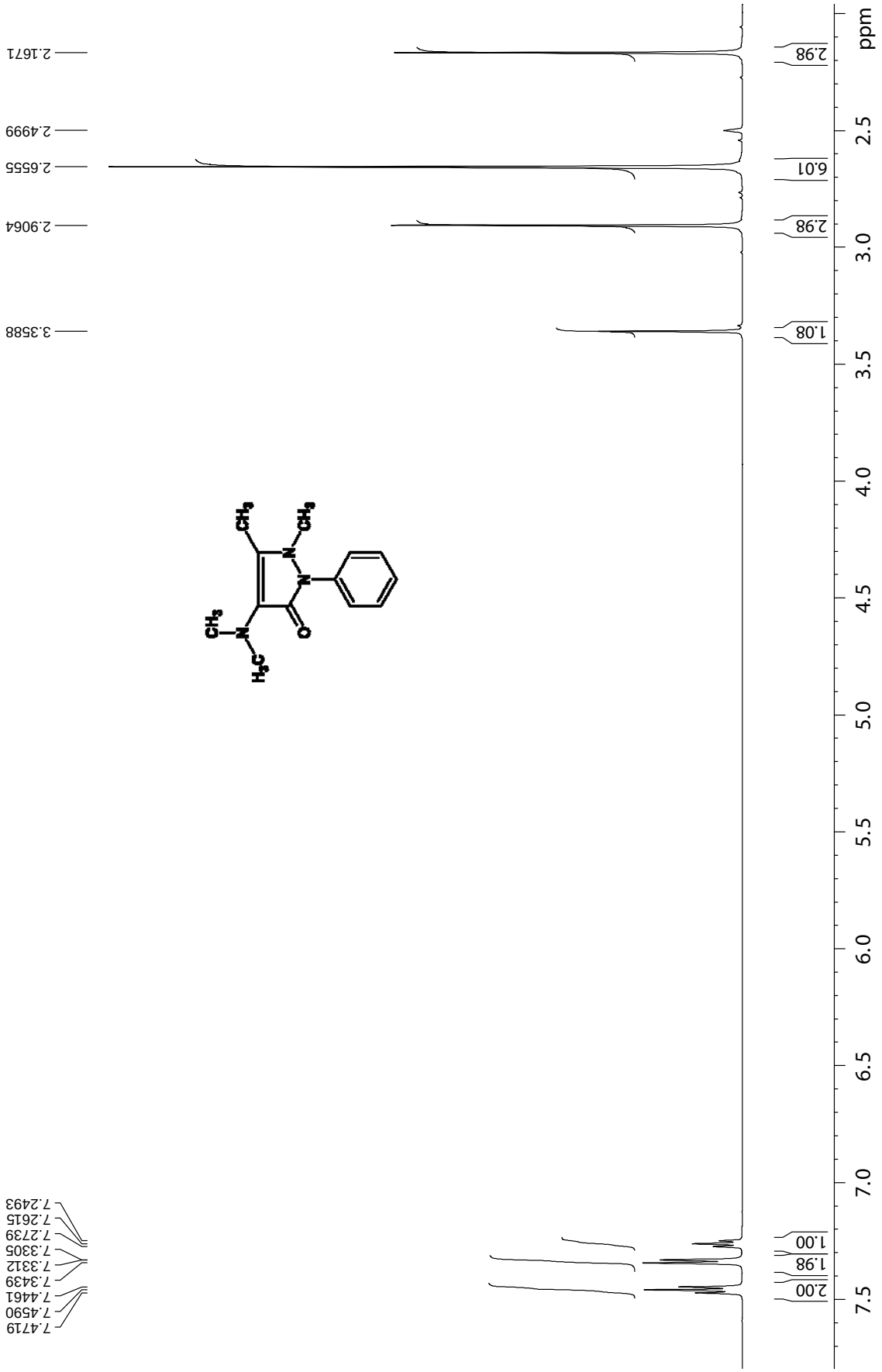
75. Metrodinazol ^{1}H, NMR spektrum (DMSO)



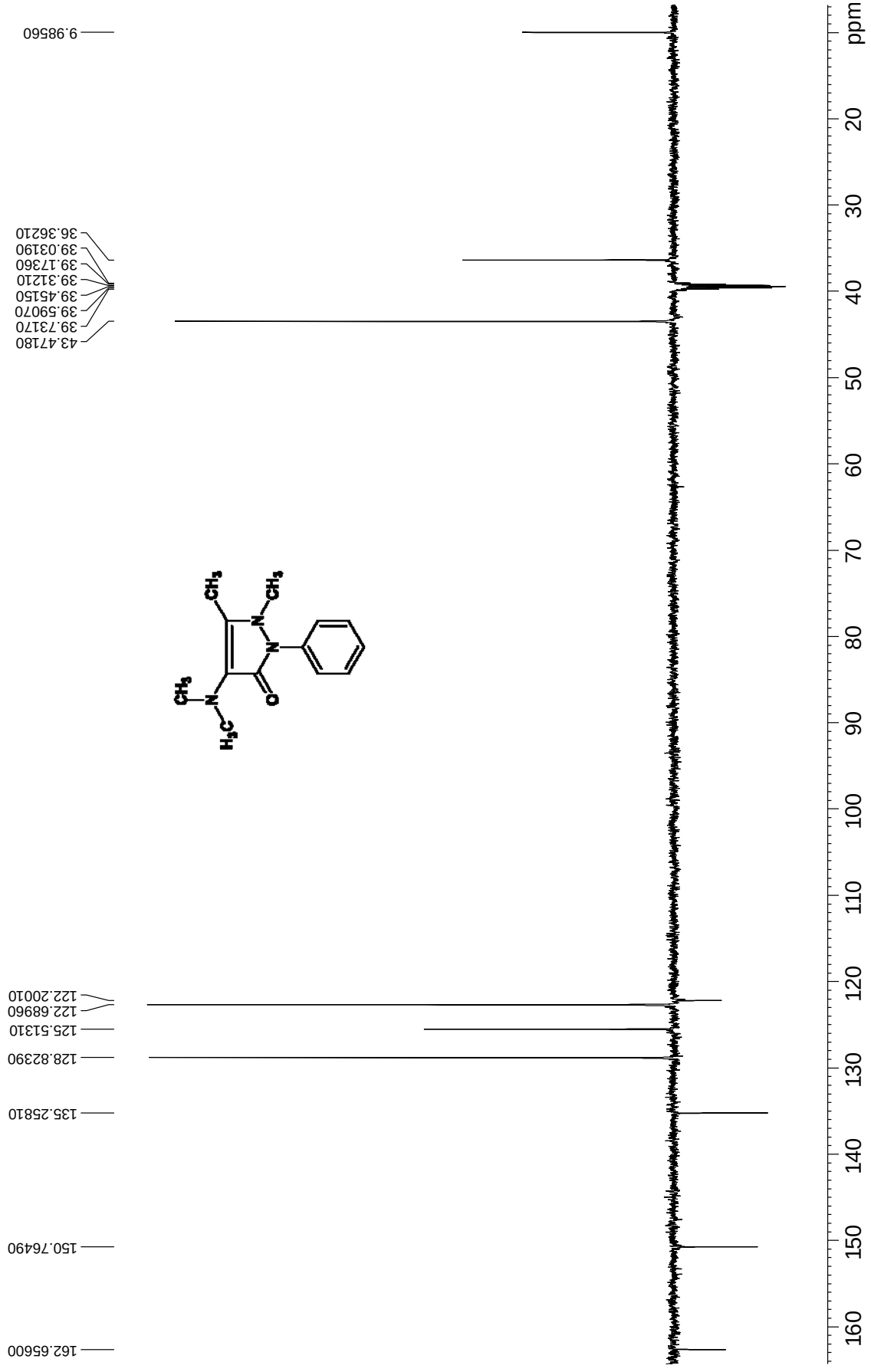
76. Metrodinazol ^{13}C-JMOD NMR spektrum (DMSO)



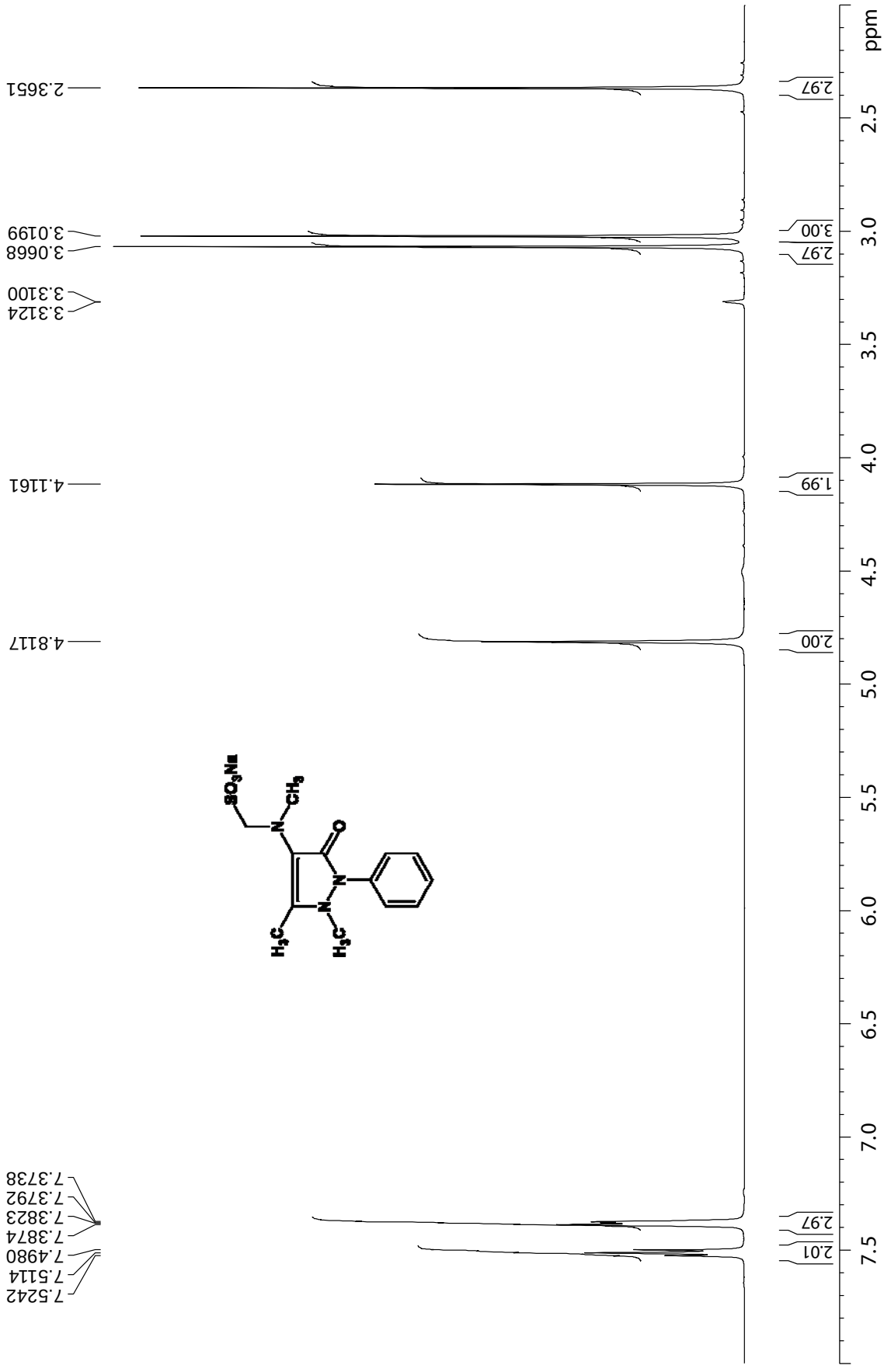
77. Aminofenazon ^{1}H NMR spektrum (DMSO)



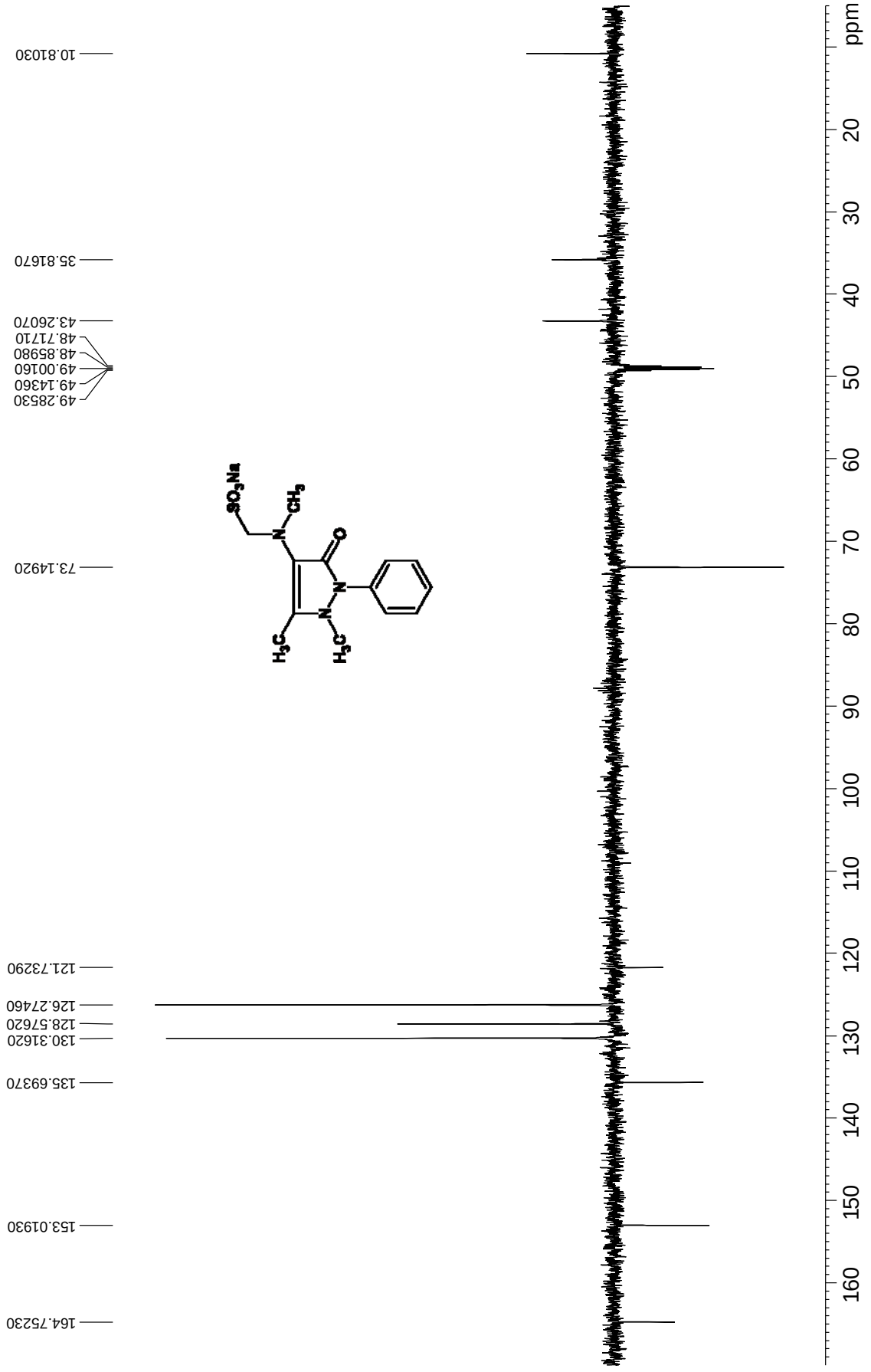
78. Aminofenazon ^{13}C-JMOD NMR spektrum (DMSO)



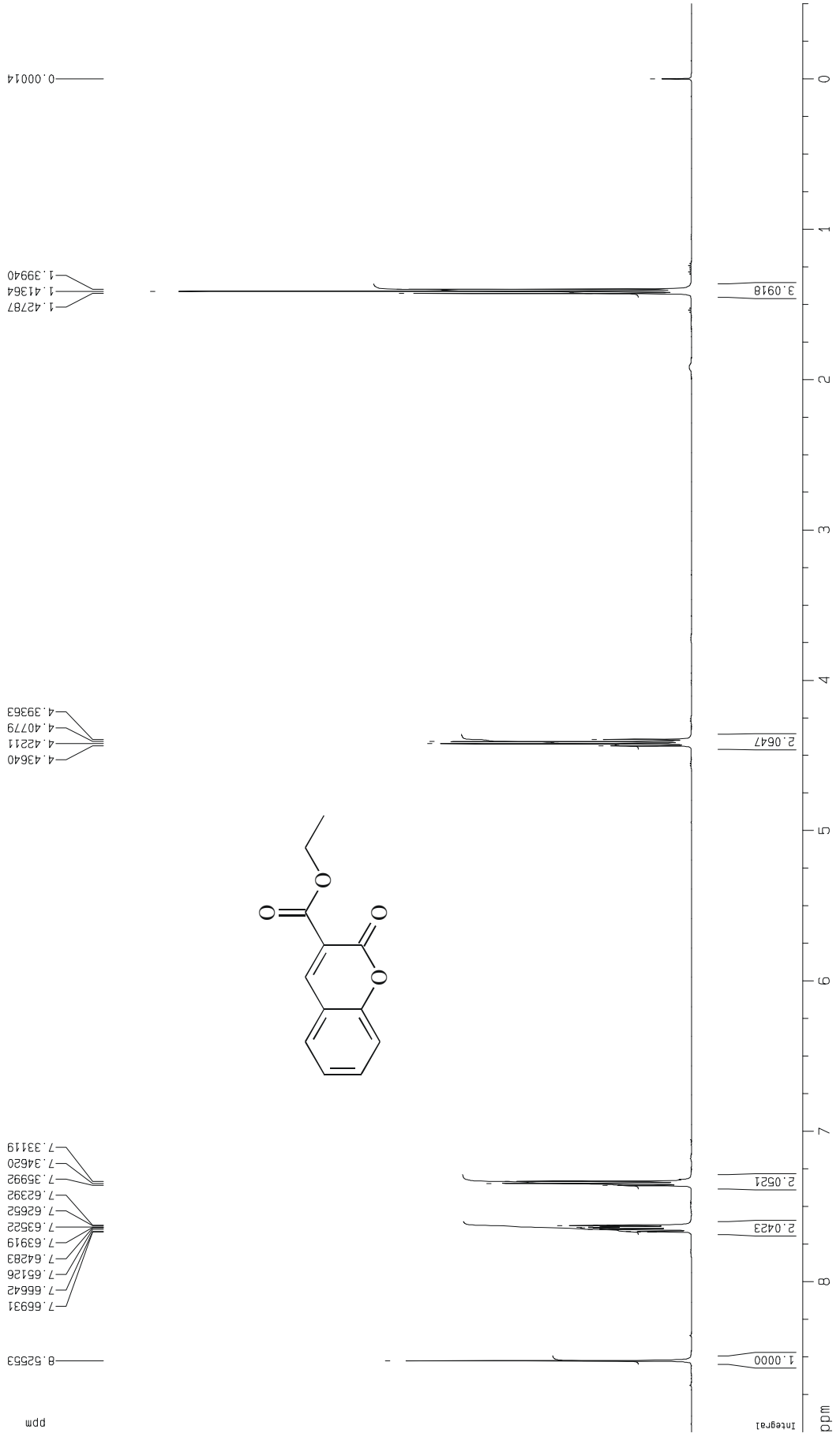
79. Noramino-fenazon ^{1}H NMR spektrum (MeOD)



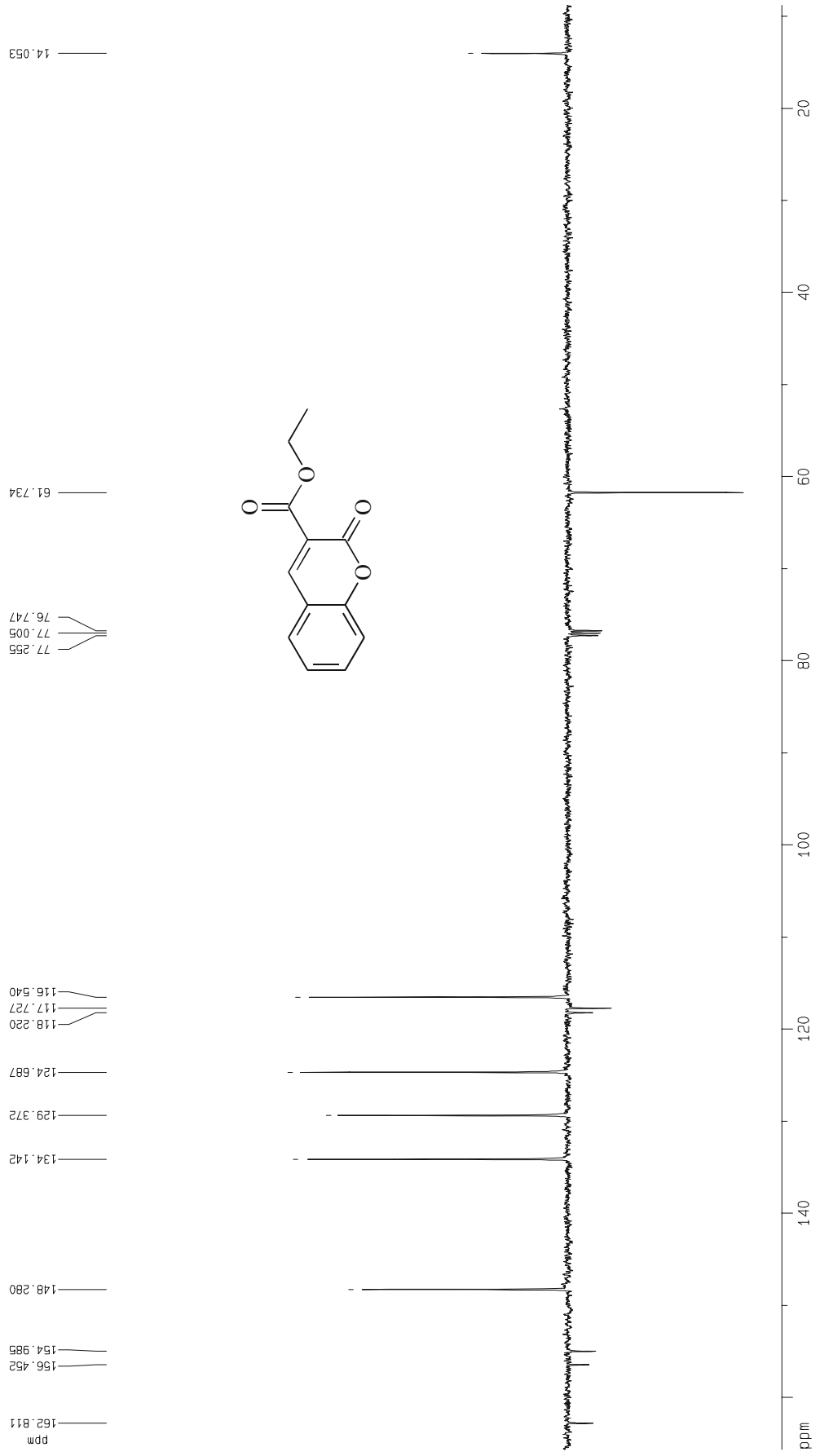
80. Noramino-fenazon ^{13}C-JMOD NMR spektrum (MeOD)



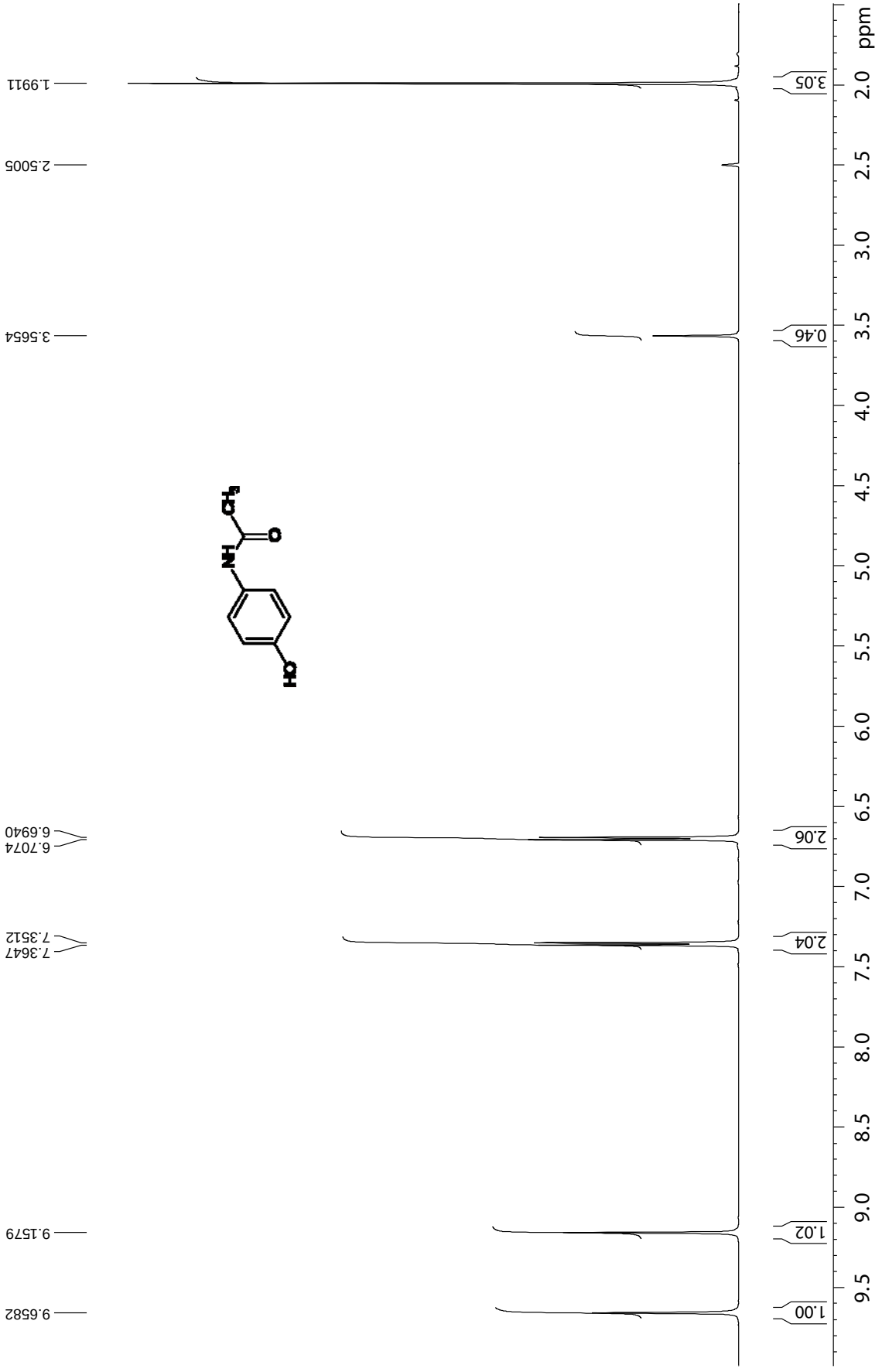
81. 3-Karbetoxi-kumarin ^{1}H-NMR spektrum ($CDCl_3$)



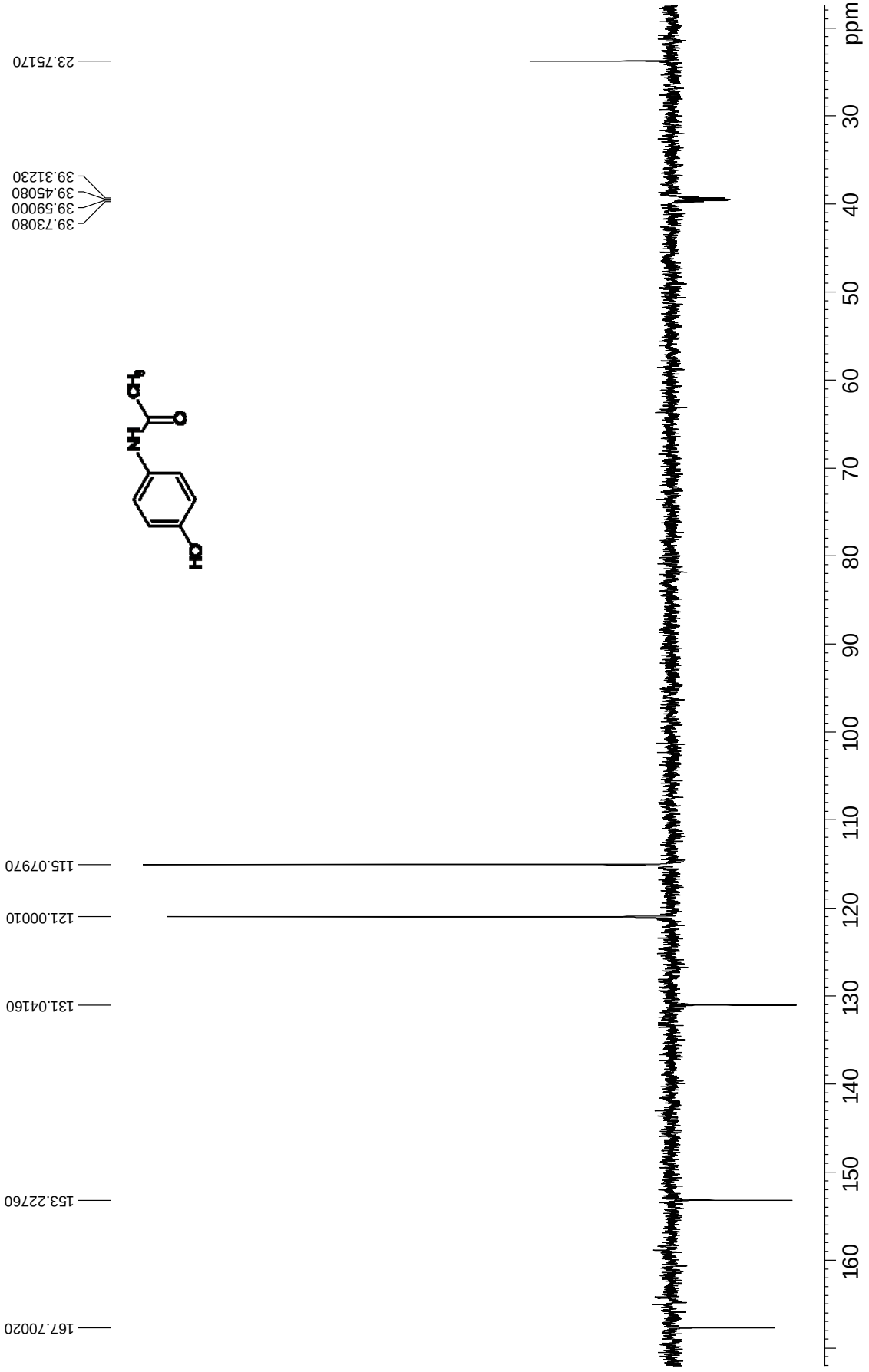
82. 3-Karbetoxi-kumarin ^{13}C-JMOD NMR spektrum ($CDCl_3$)



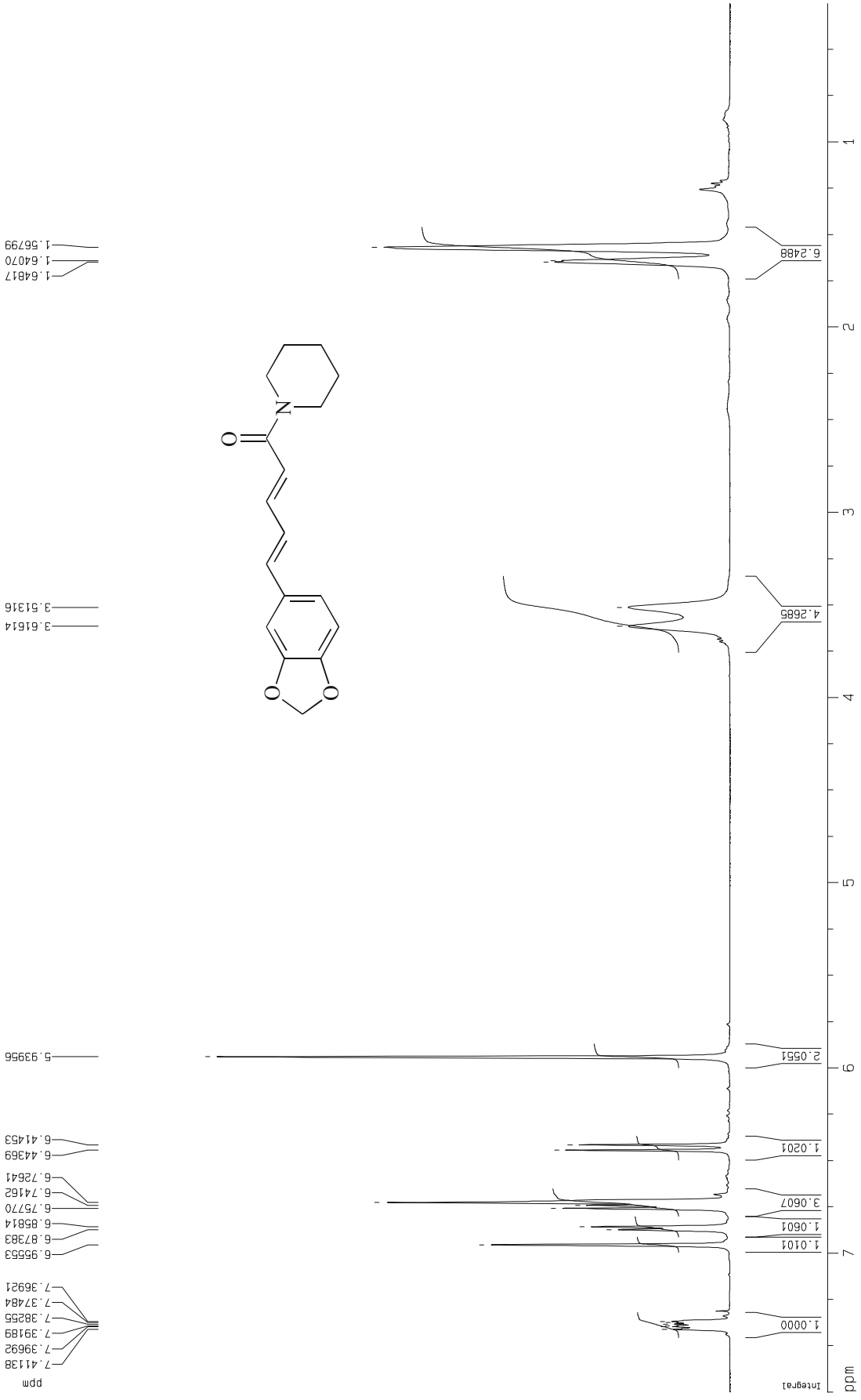
83. Paracetamol ^{1}H-NMR spektrum (DMSO)



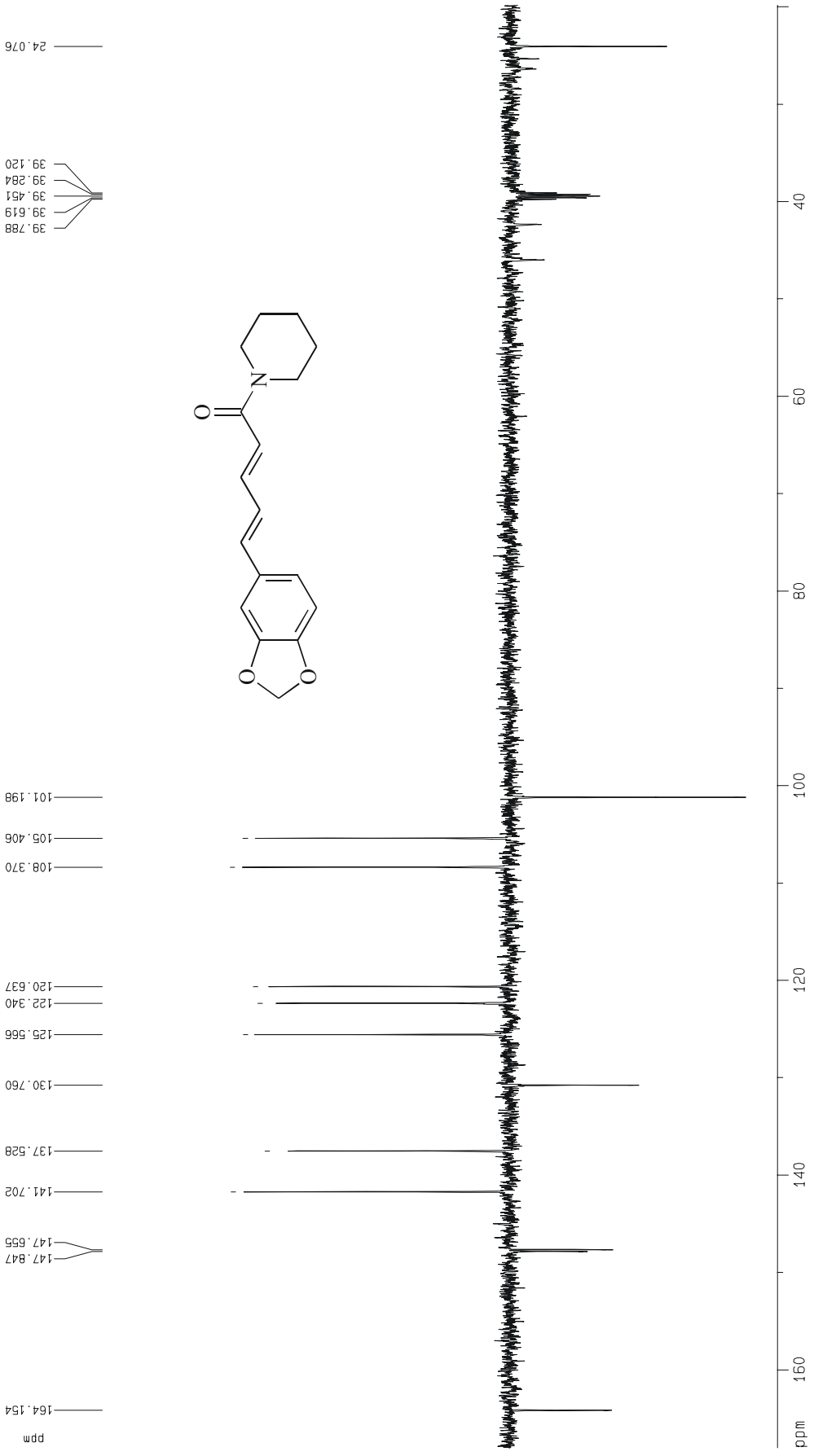
84. Paracetamol ^{13}C-JMOD NMR spektrum (DMSO)



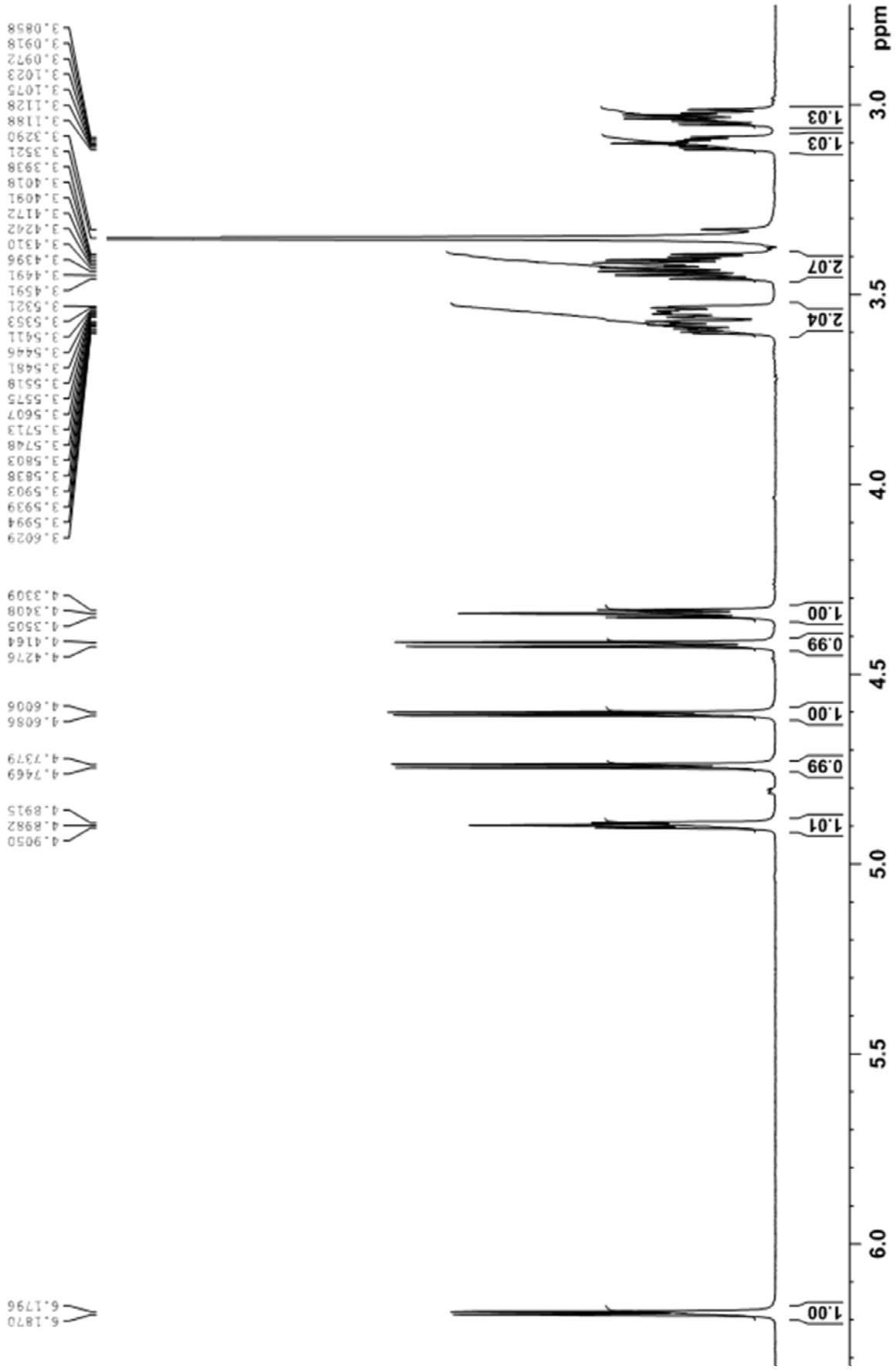
85. Piperin ^{1}H-NMR spektrum (DMSO)



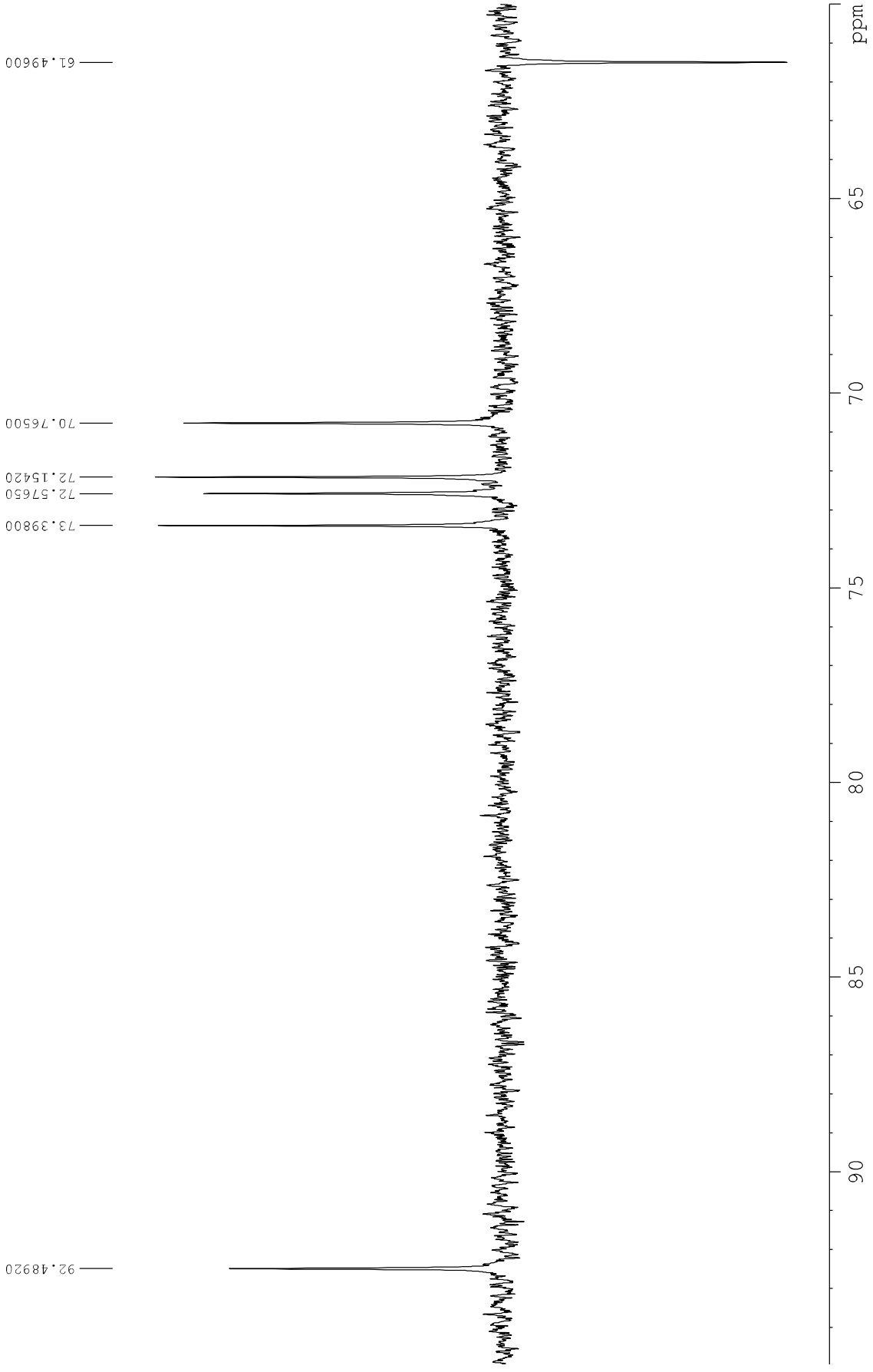
86. Piperin ^{13}C-JMOD NMR spektrum (DMSO)



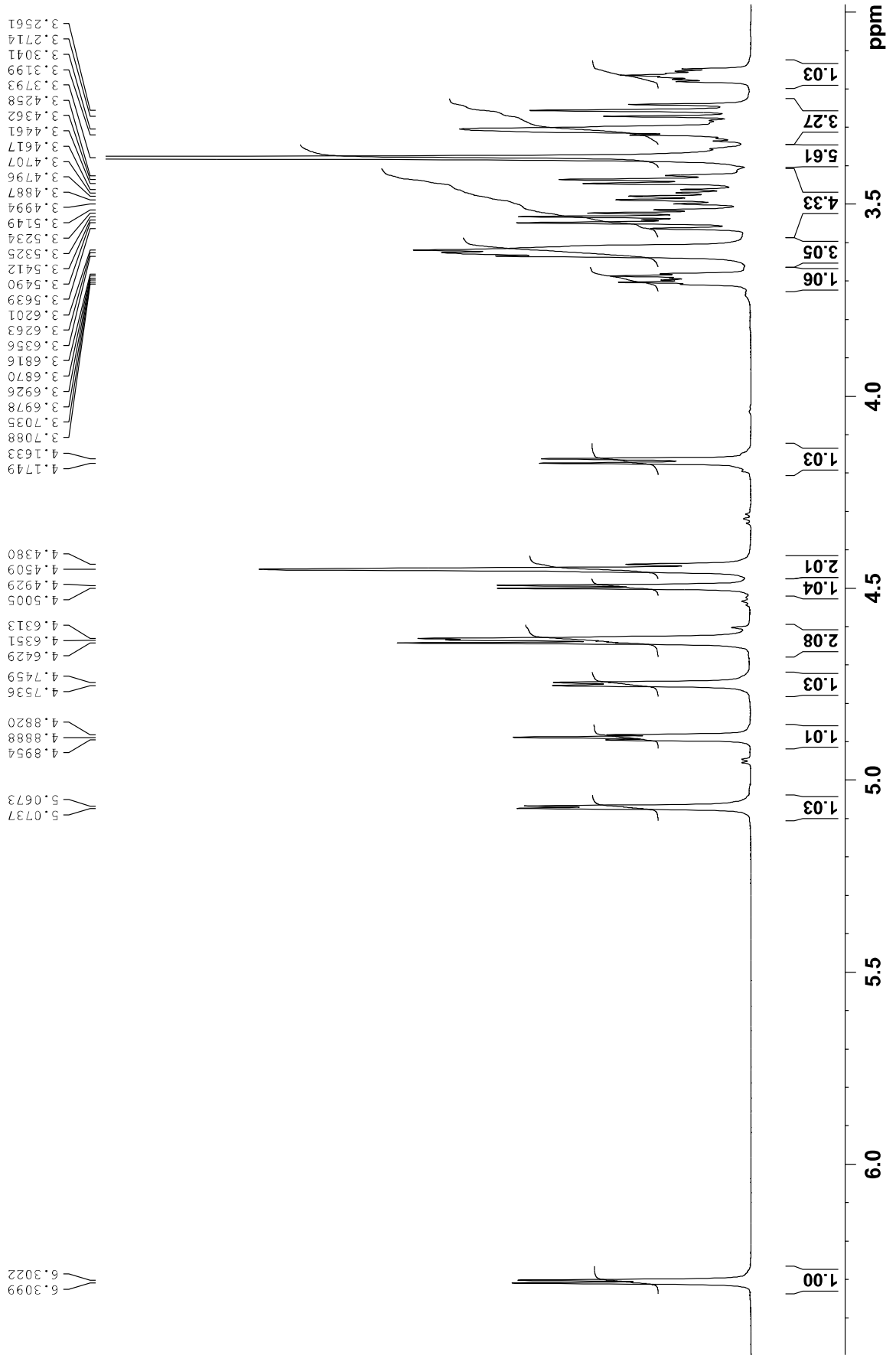
87. *D*-(+)-Glükóz ^{1}H NMR spektrum (DMSO)



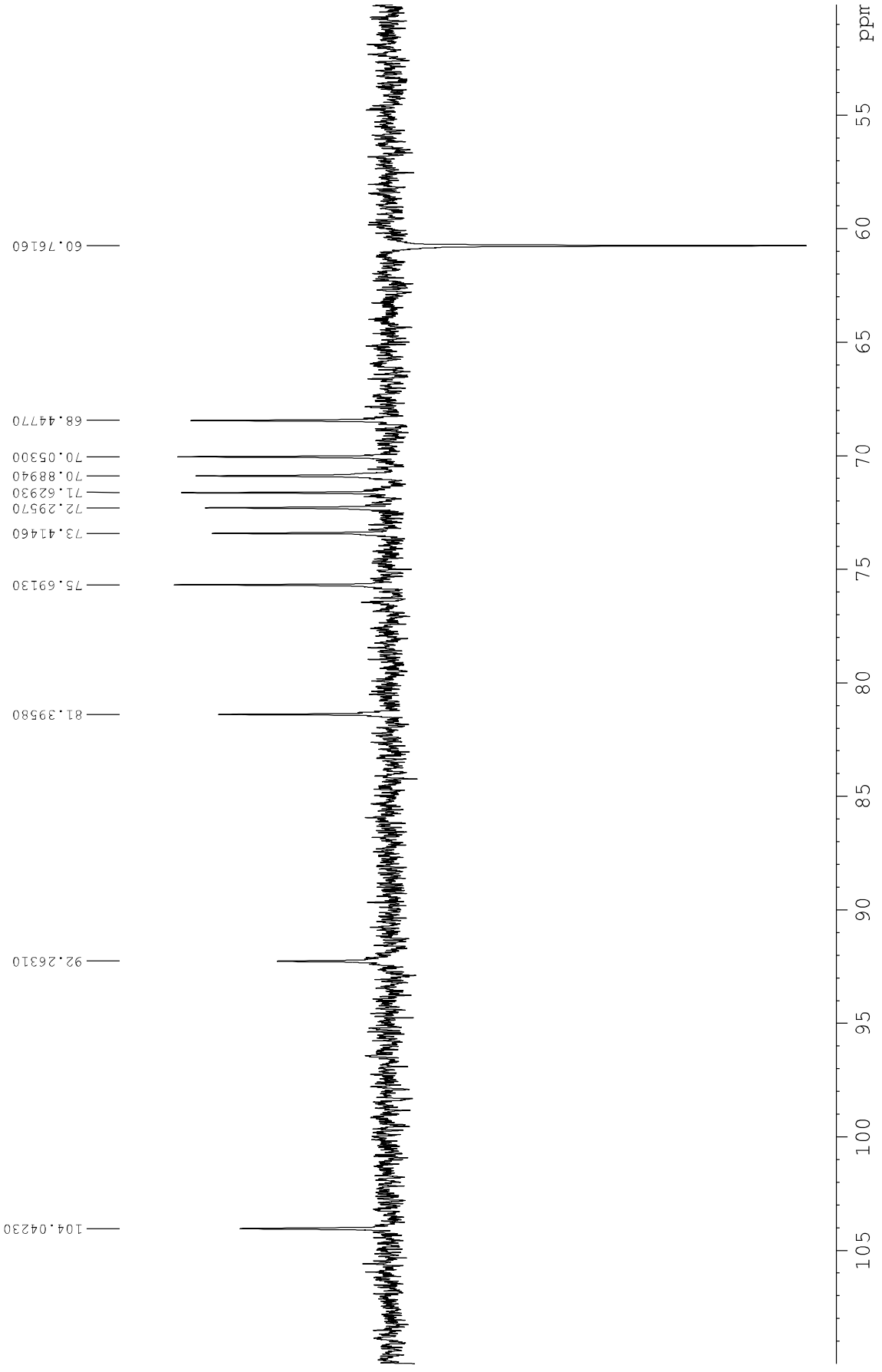
88. *D*-(+)-Glükóz ^{13}C-JMOD NMR spektrum (DMSO)



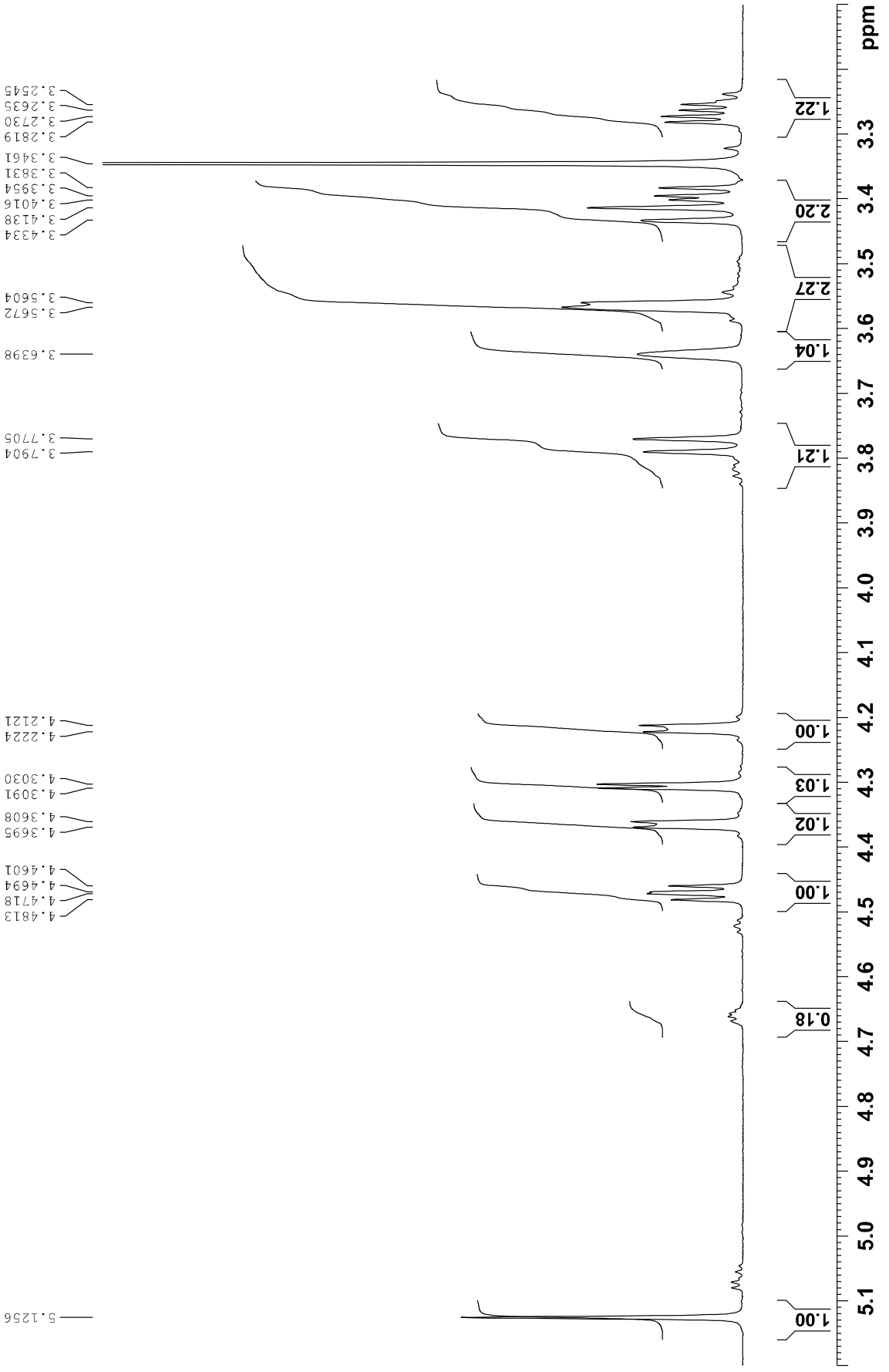
89. Laktóz ^{1}H NMR spektrum (DMSO)



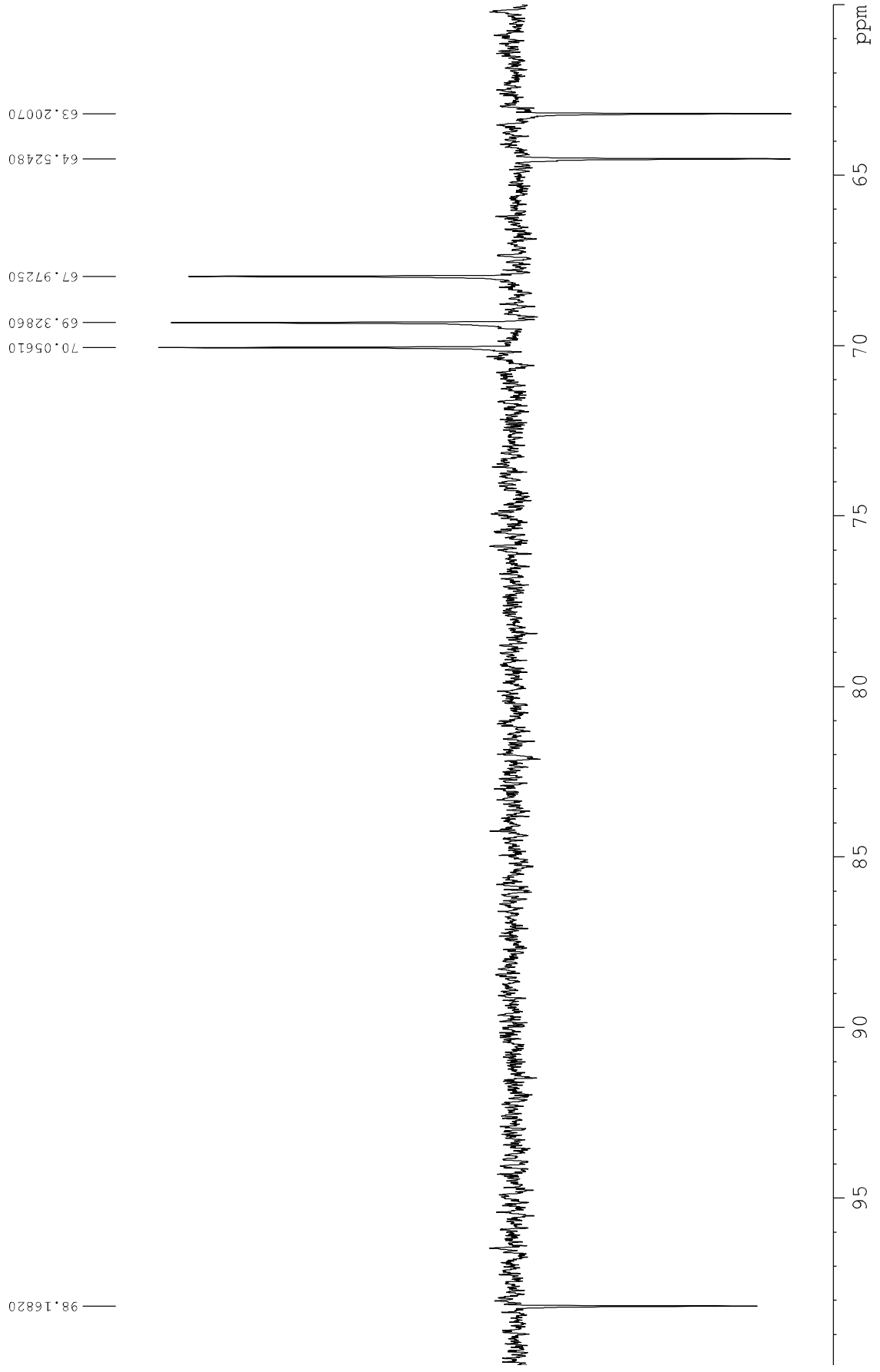
90. Laktóz ^{13}C-JMOD NMR spektrum (DMSO)



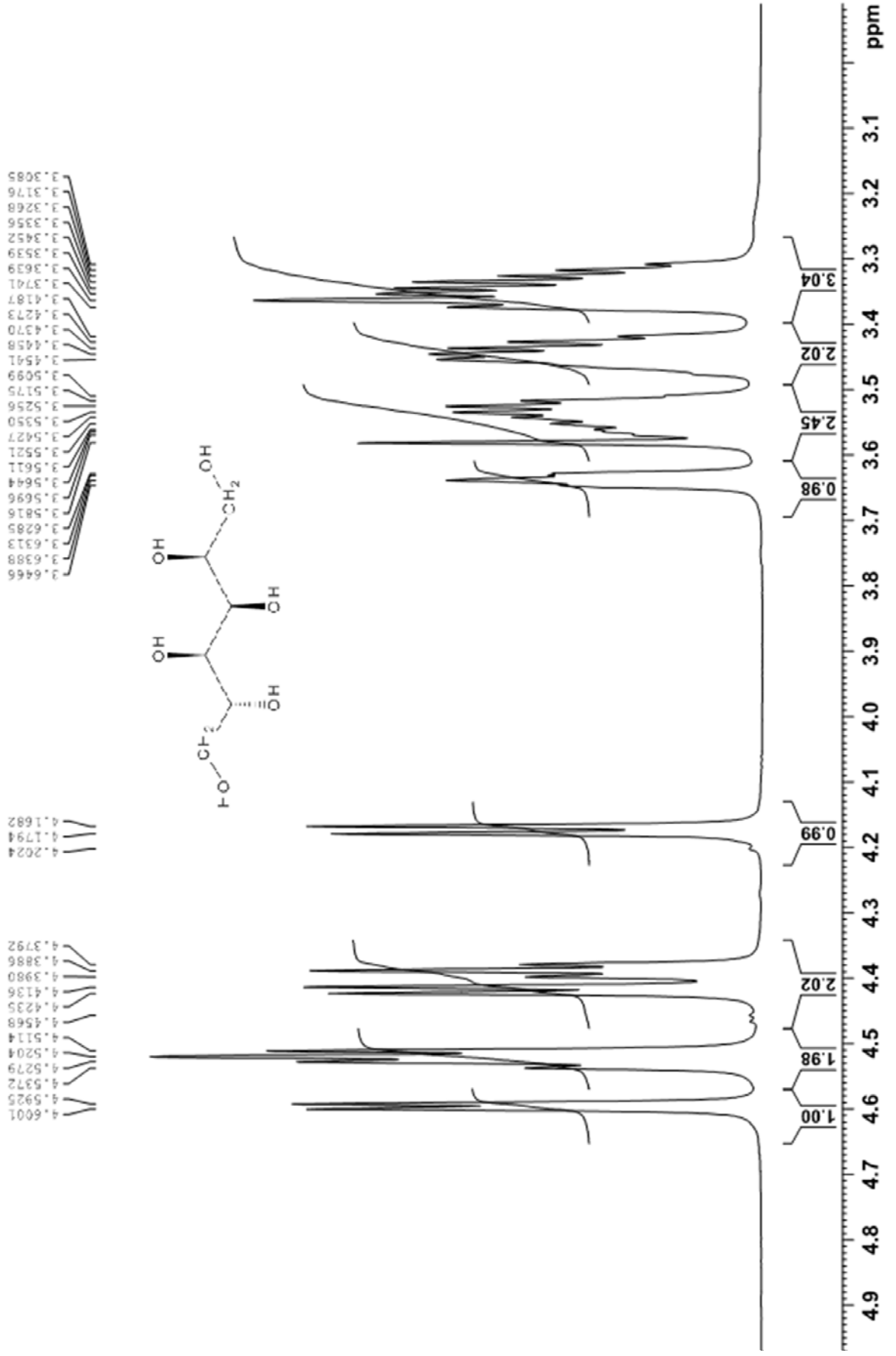
91. *D*-(-) Fruktóz ^{1}H NMR spektrum (DMSO)



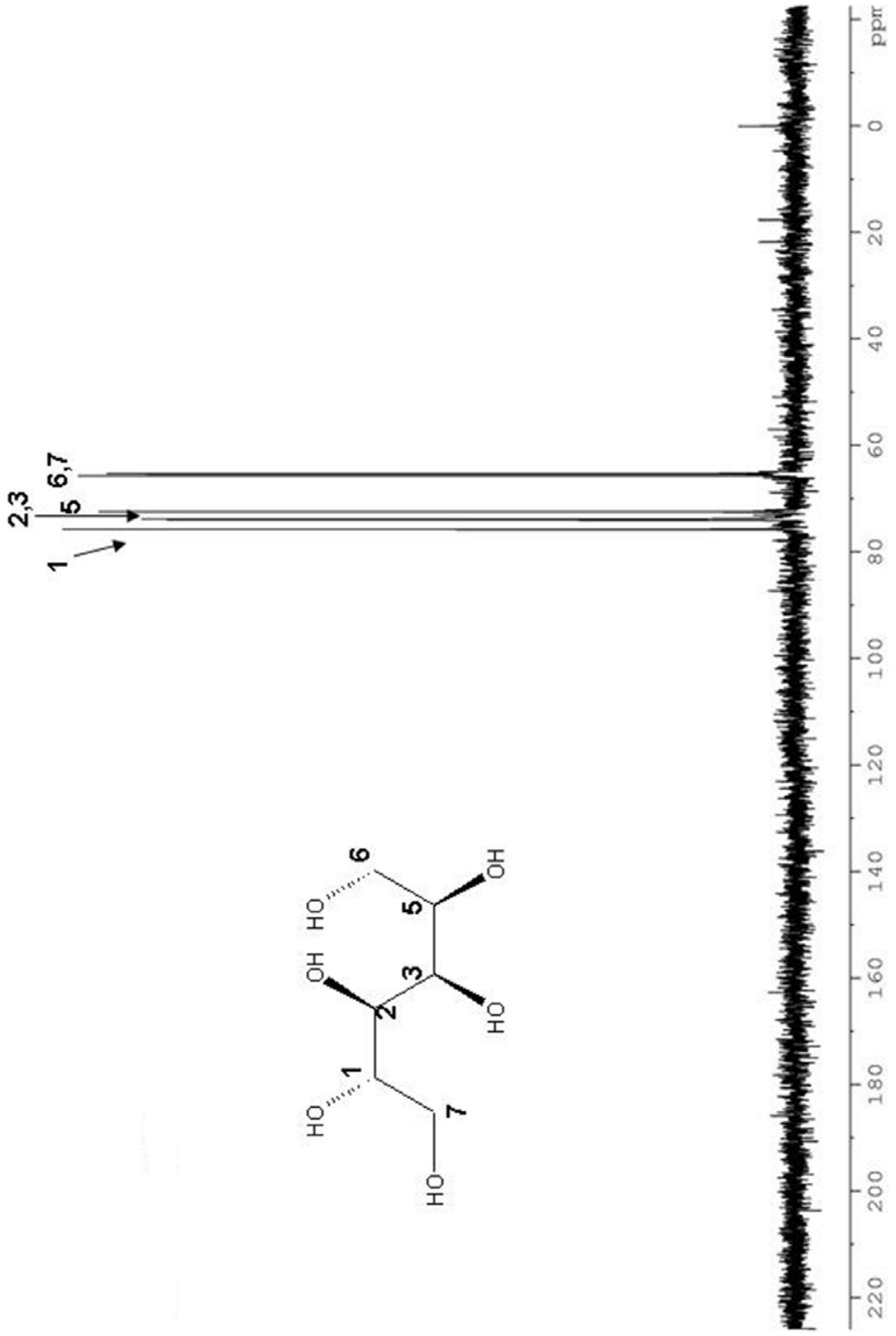
92. *D*-(-) Fruktóz ^{13}C-JMOD NMR (DMSO)



93. Szorbitol ^{1}H NMR spektrum (DMSO)

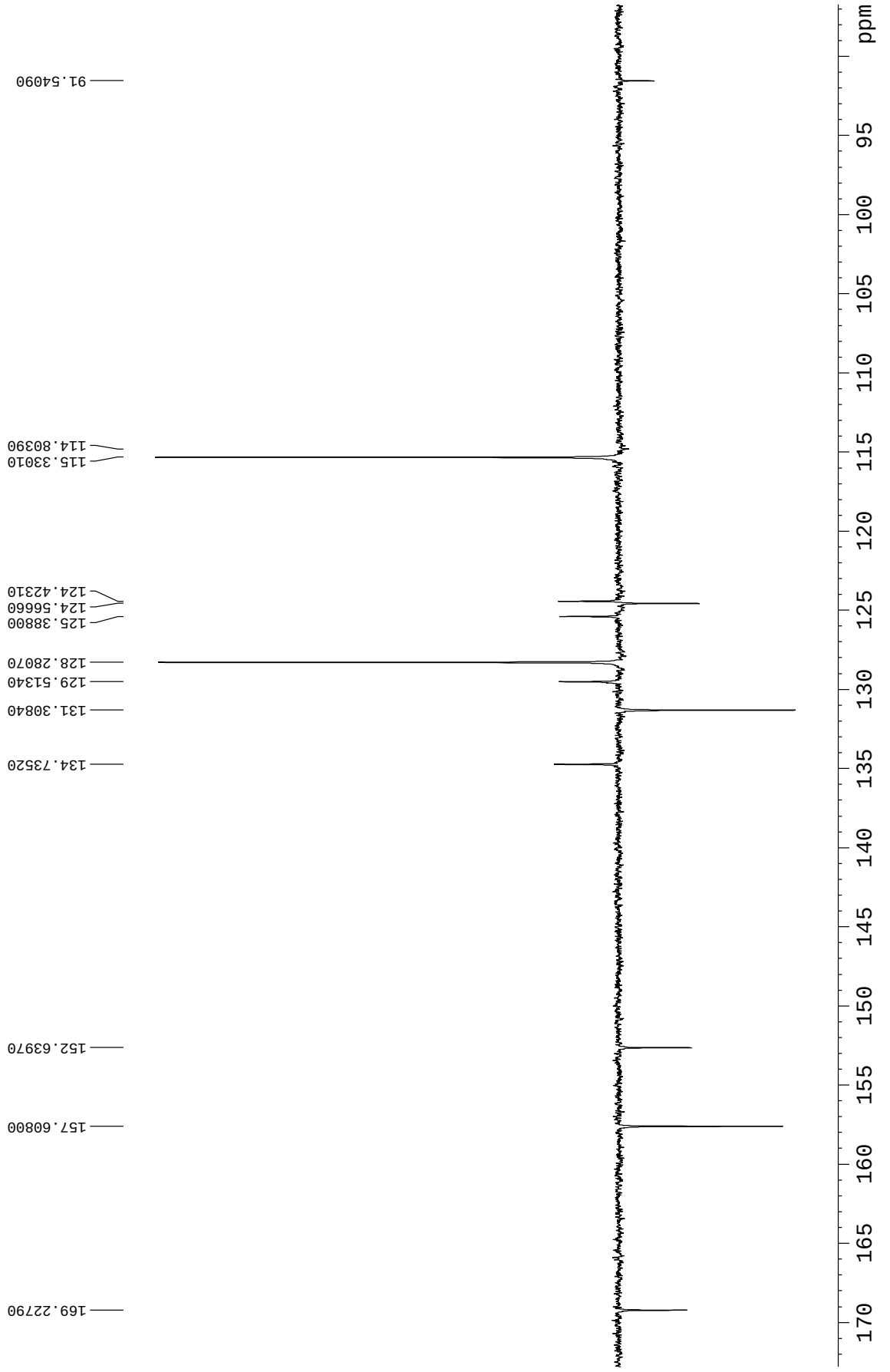


94. Szorbitol ^{13}C-JMOD NMR spektrum (DMSO)

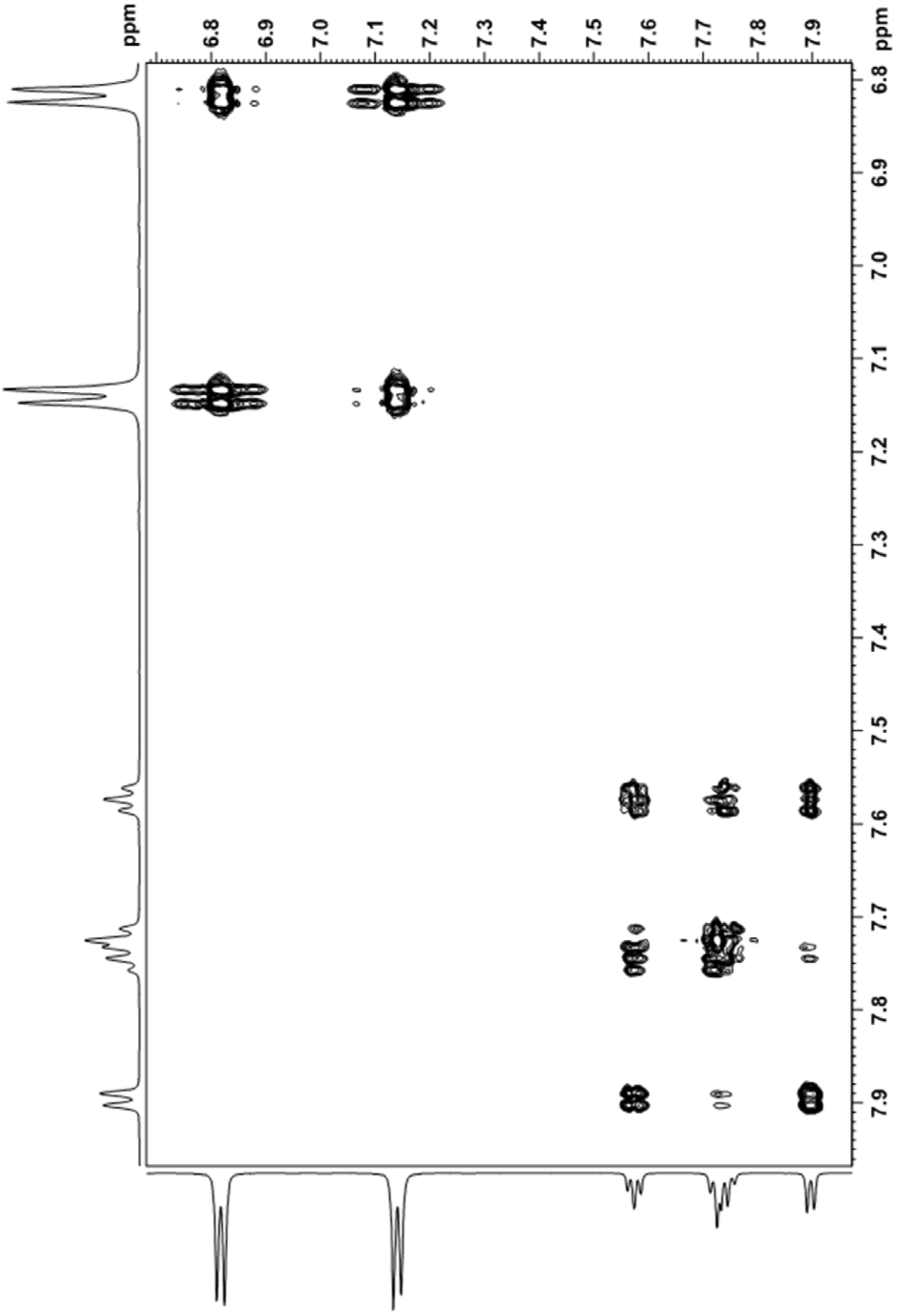


95. Fenolftalein ^{1}H NMR spektrum (DMSO)

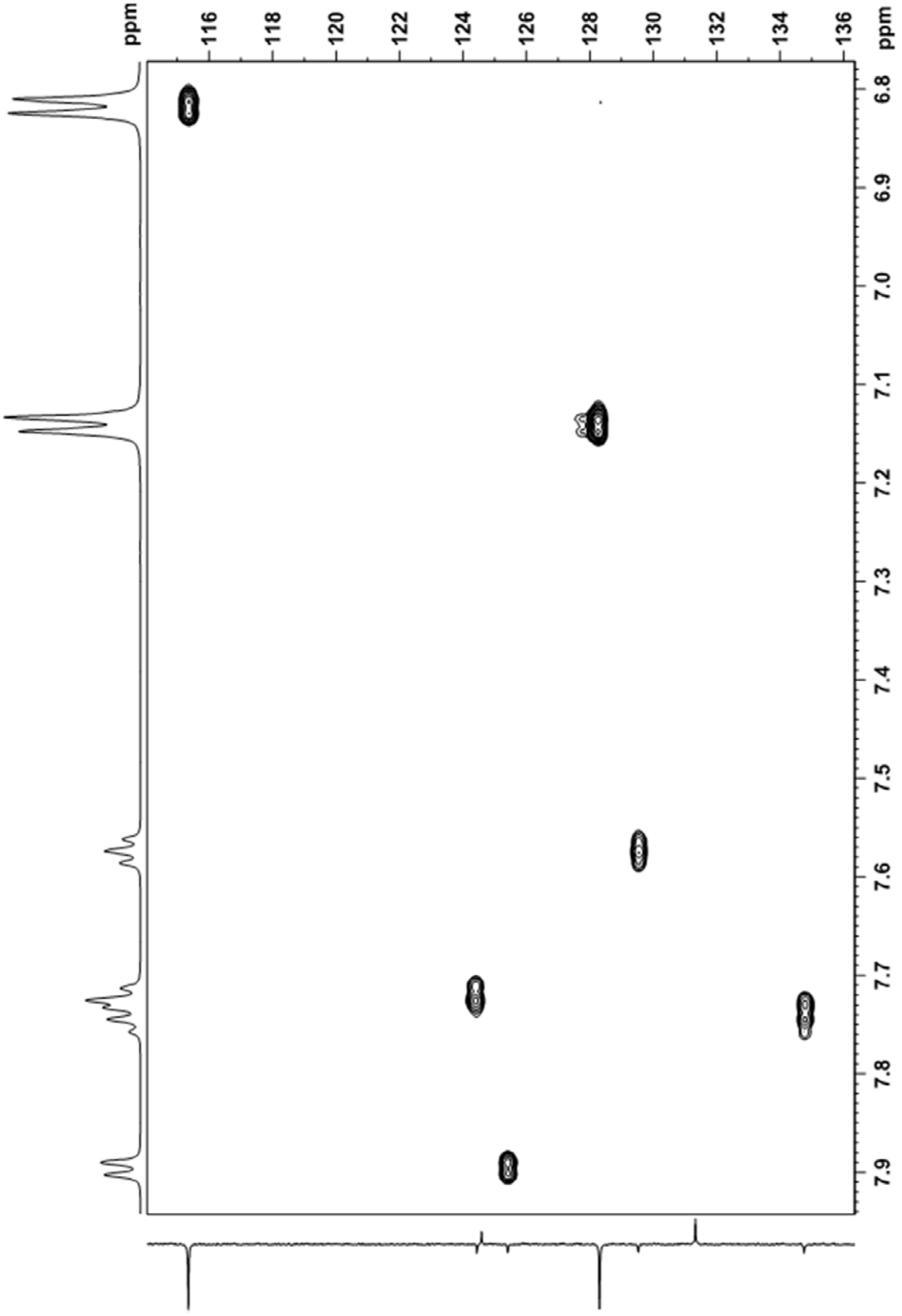
96. Fenolftalein ^{13}C-JMOD NMR spektrum (DMSO)



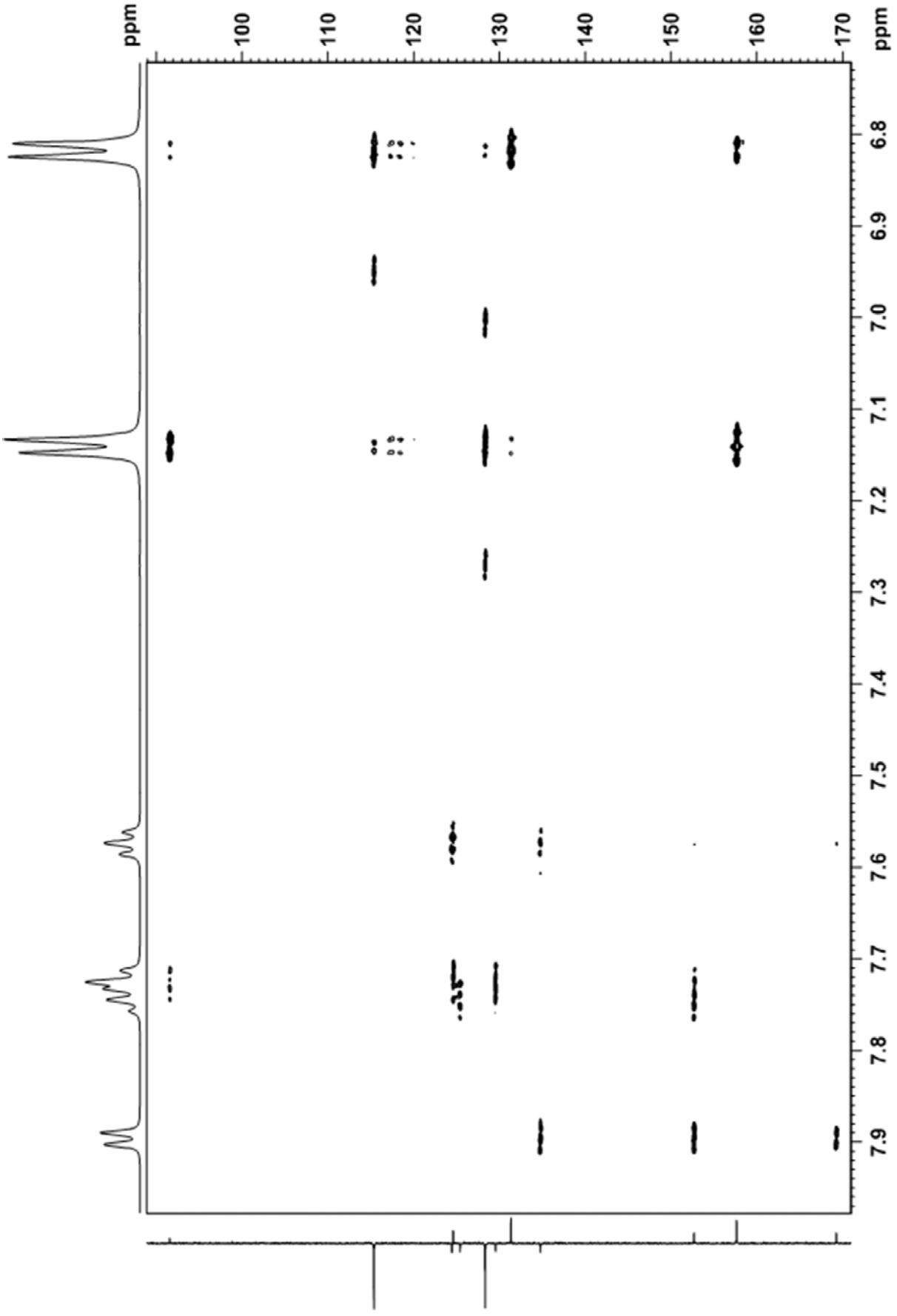
97. Fenolftalein ^{1}H-^{1}H COSY-NMR spektrum (DMSO)



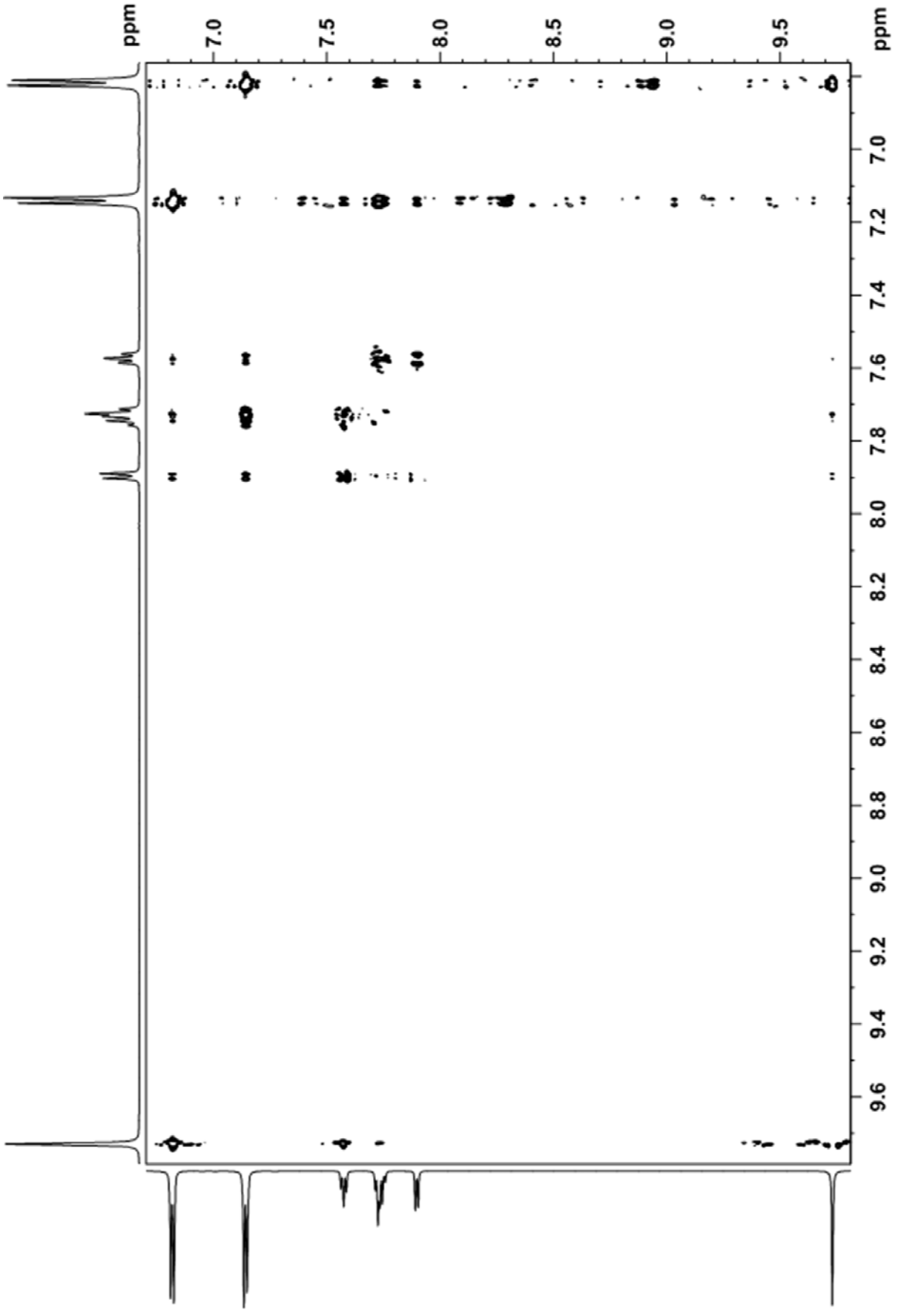
98. Fenolftalein ^{1}H-^{13}C HSQC-NMR spektrum (DMSO)



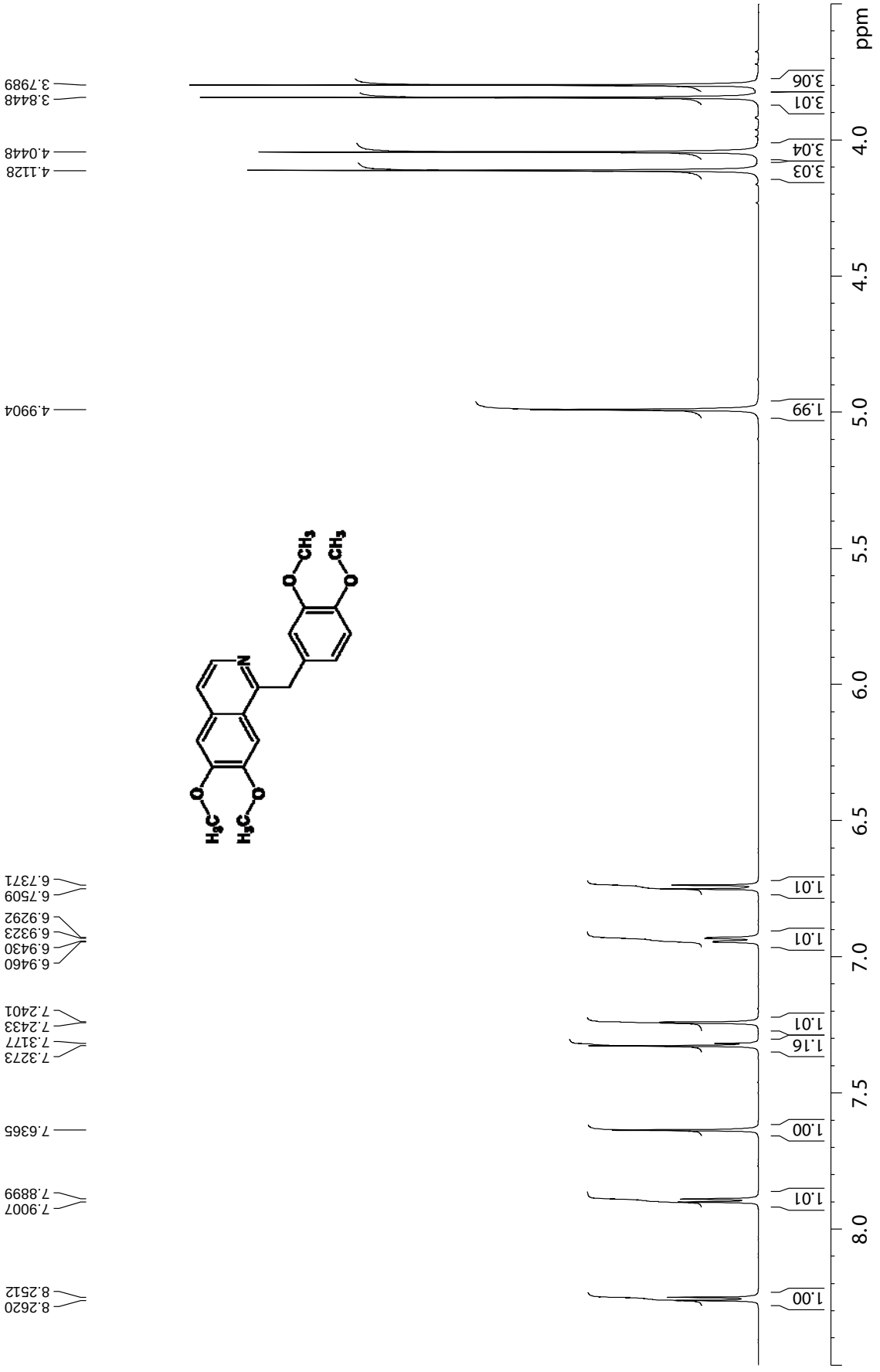
99. Fenolftalein ^{1}H-^{13}C HMBC-NMR spektrum (DMSO)



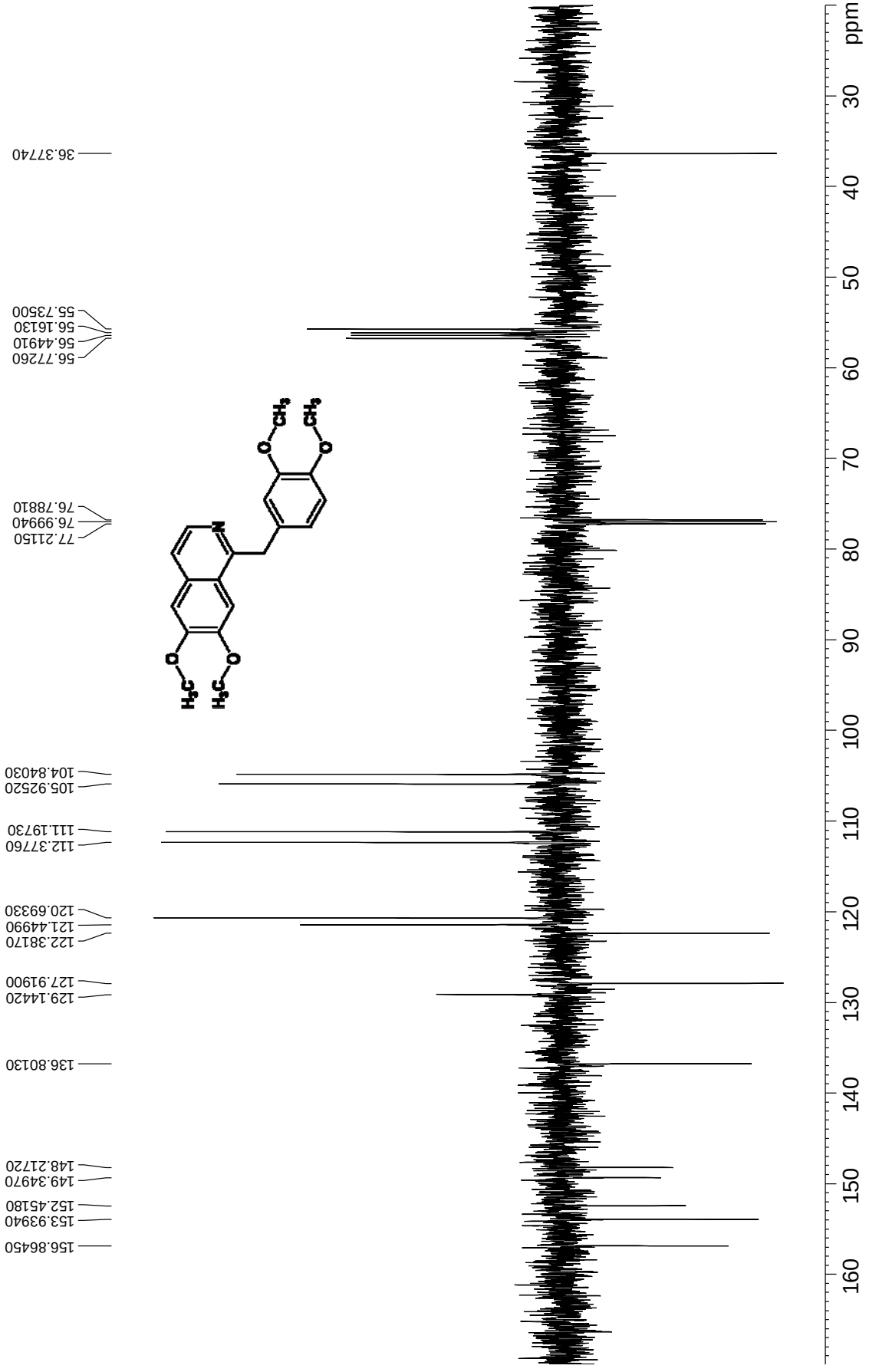
100. Fenolftalein ^{1}H-^{1}H ROESY-NMR spektrum (DMSO)



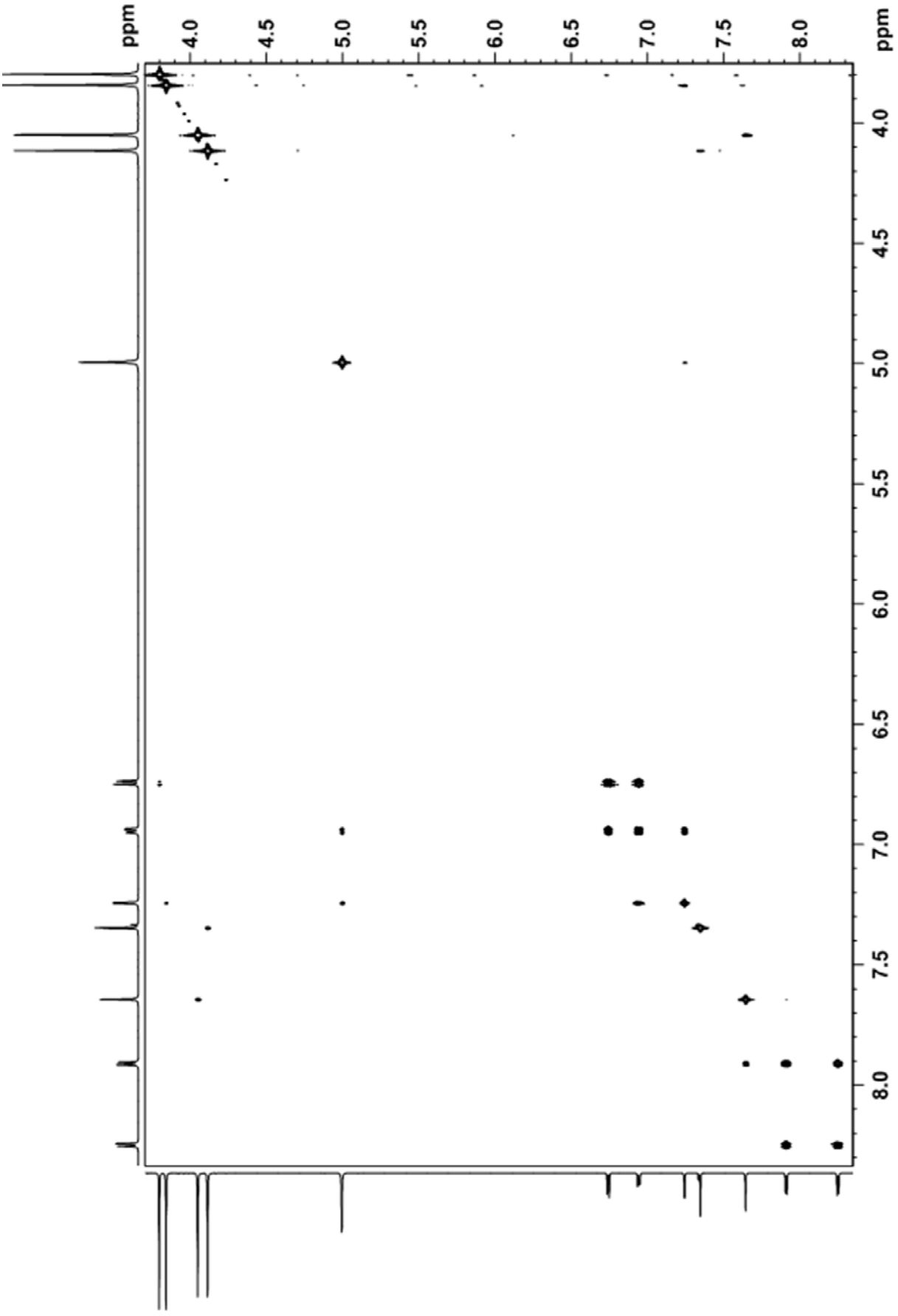
101. Papaverin ^{1}H-NMR spektrum ($CDCl_3$)



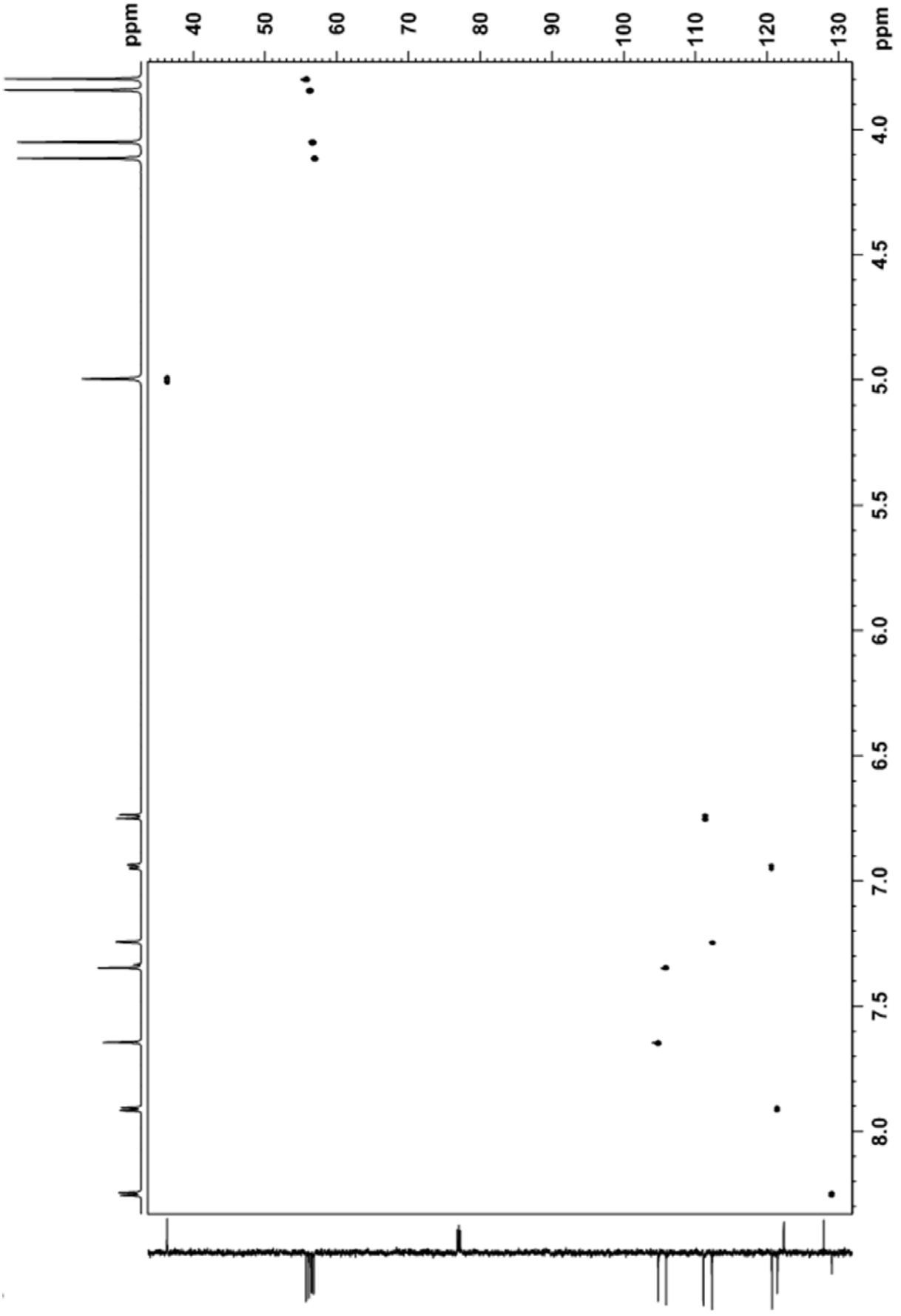
102. Papaverin ^{13}C-JMOD NMR spektrum ($CDCl_3$)



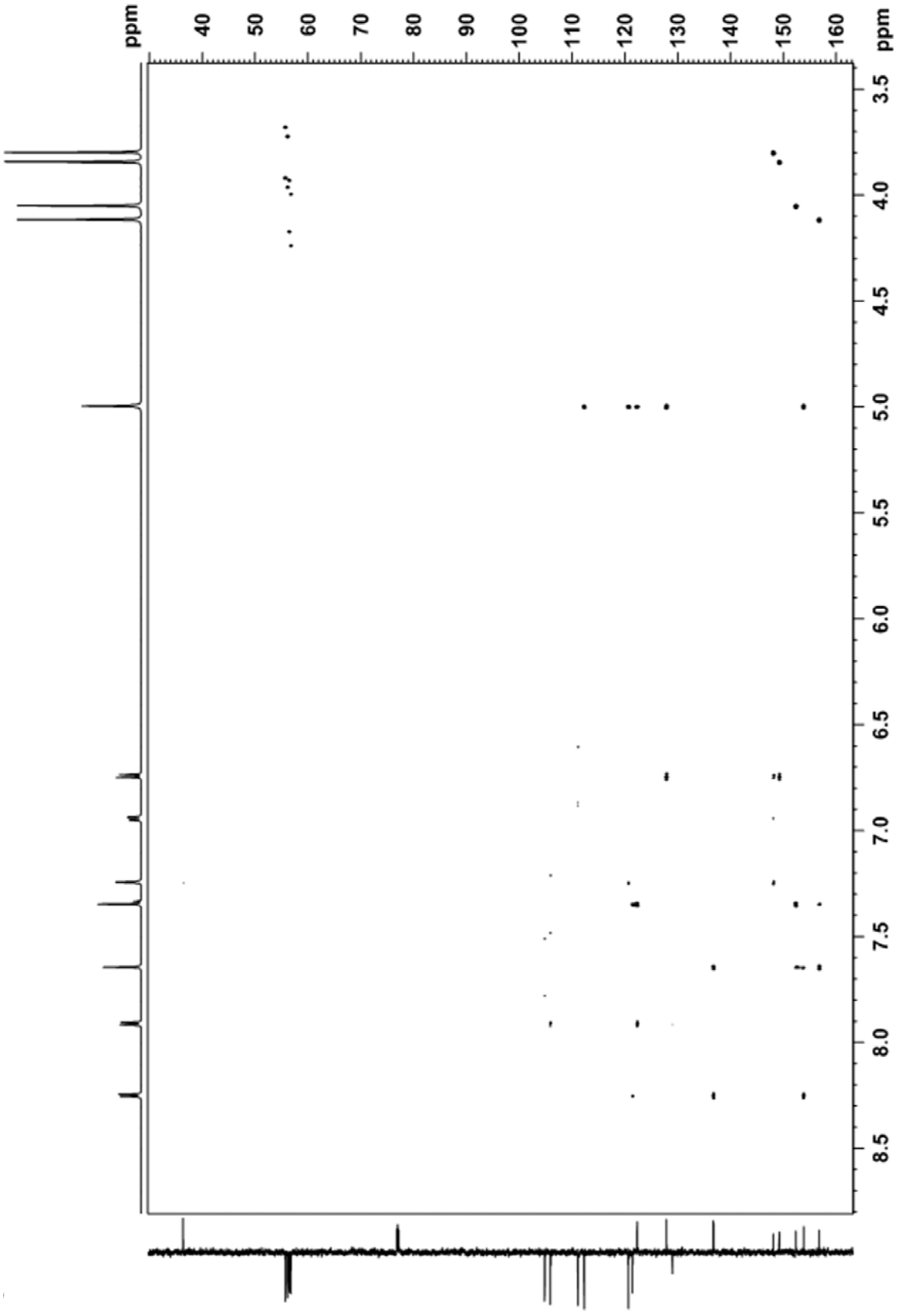
103. Papaverin ^{1}H-^{1}H COSY-NMR spektrum ($CDCl_3$)



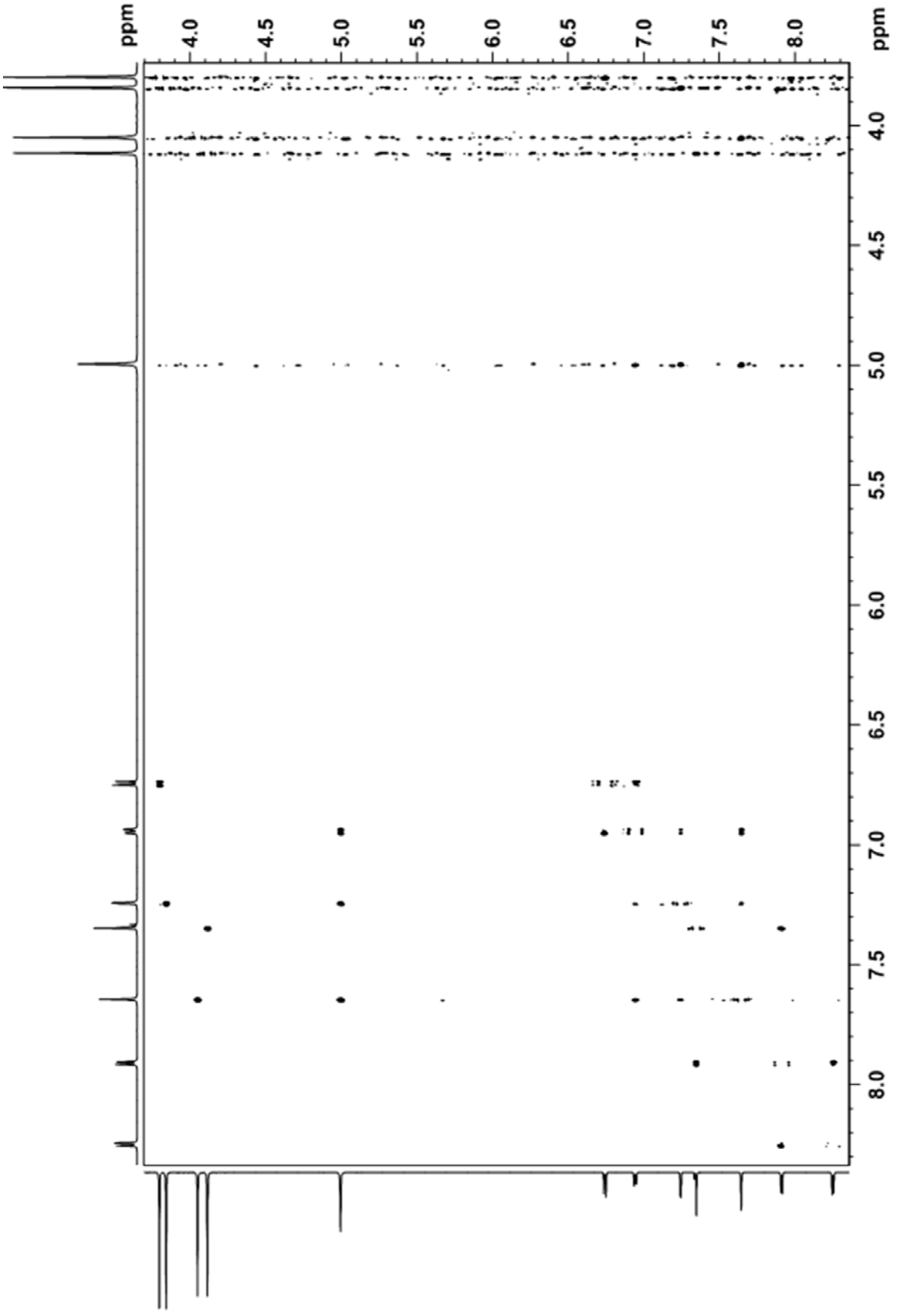
104. Papaverin ^{1}H-^{13}C HSQC-NMR spektrum ($CDCl_3$)



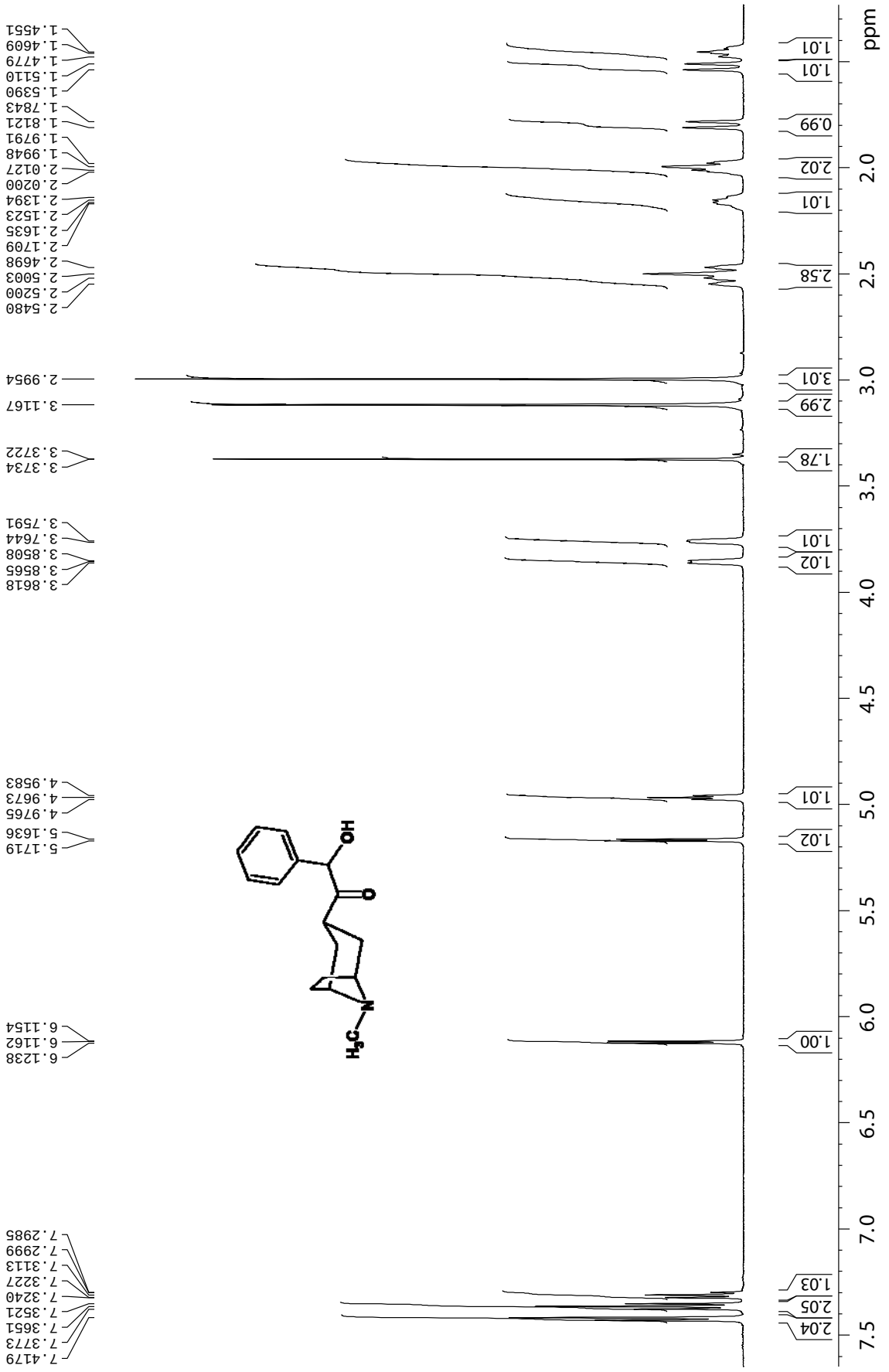
105. Papaverin ^{1}H-^{13}C HMBC-NMR spektrum ($CDCl_3$)



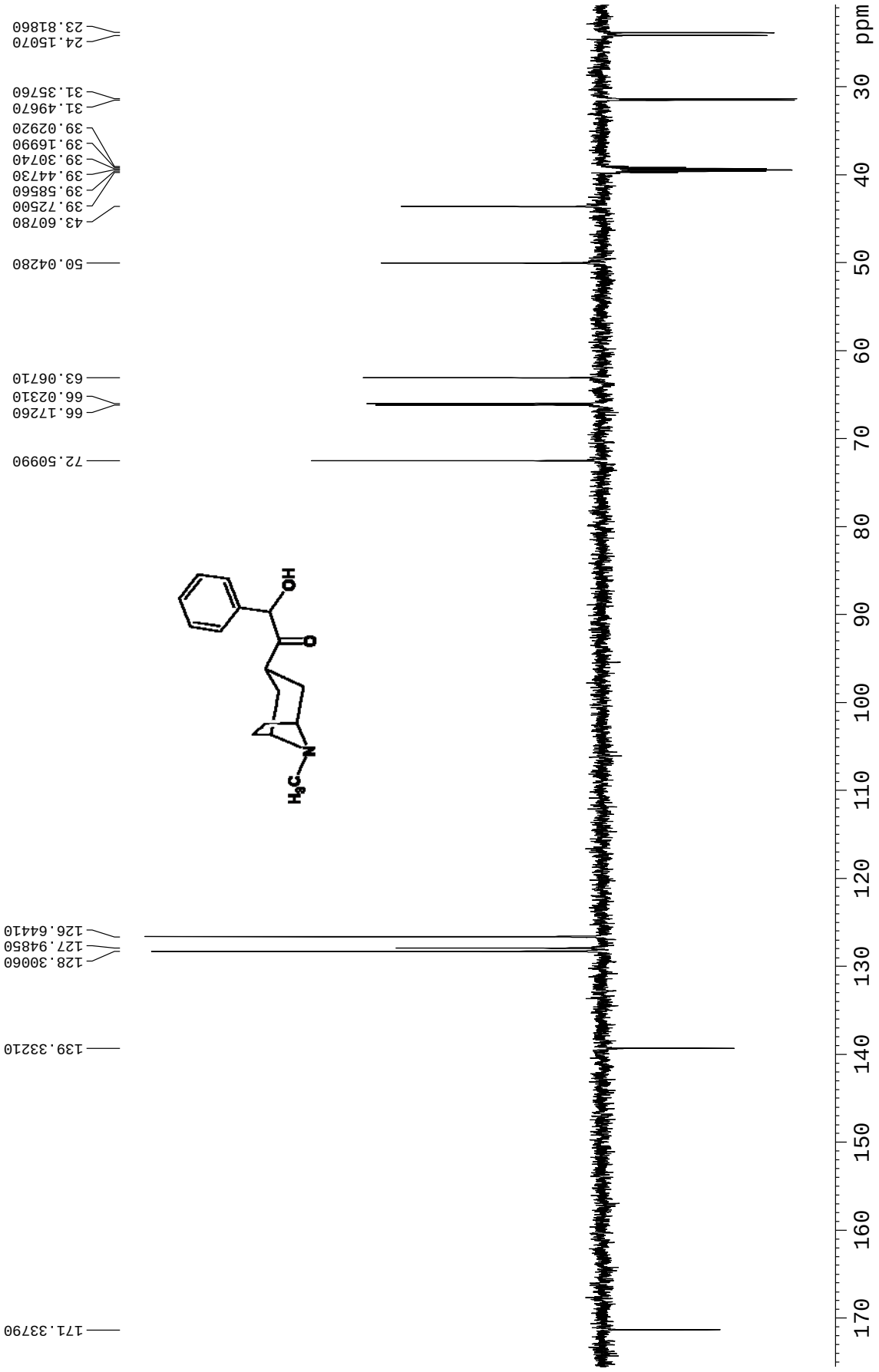
106. Papaverin ^{1}H-^{1}H NOESY-NMR spektrum ($CDCl_3$)



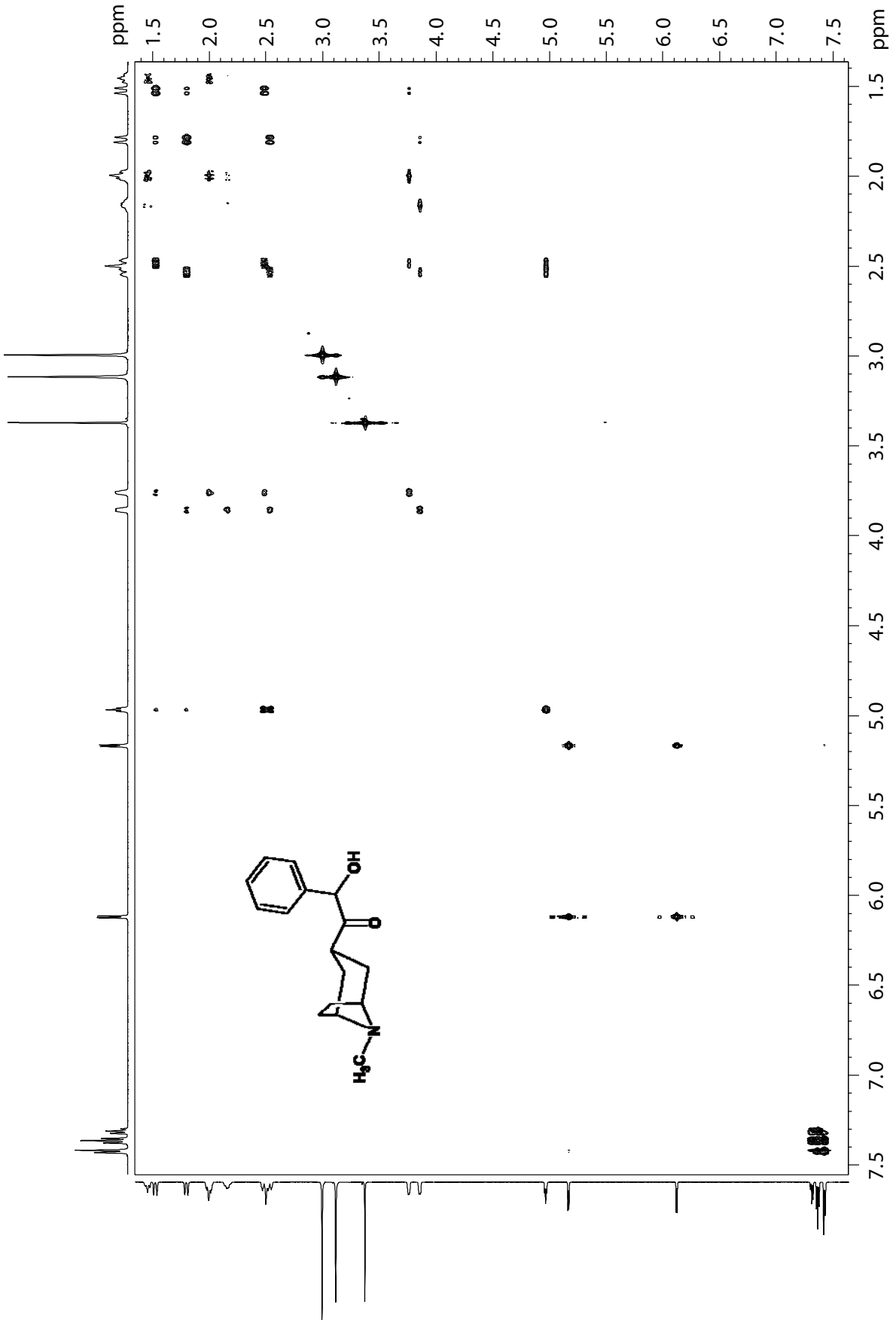
107. Metil-homatropinium-bromid ^{1}H-NMR spektrum (DMSO)



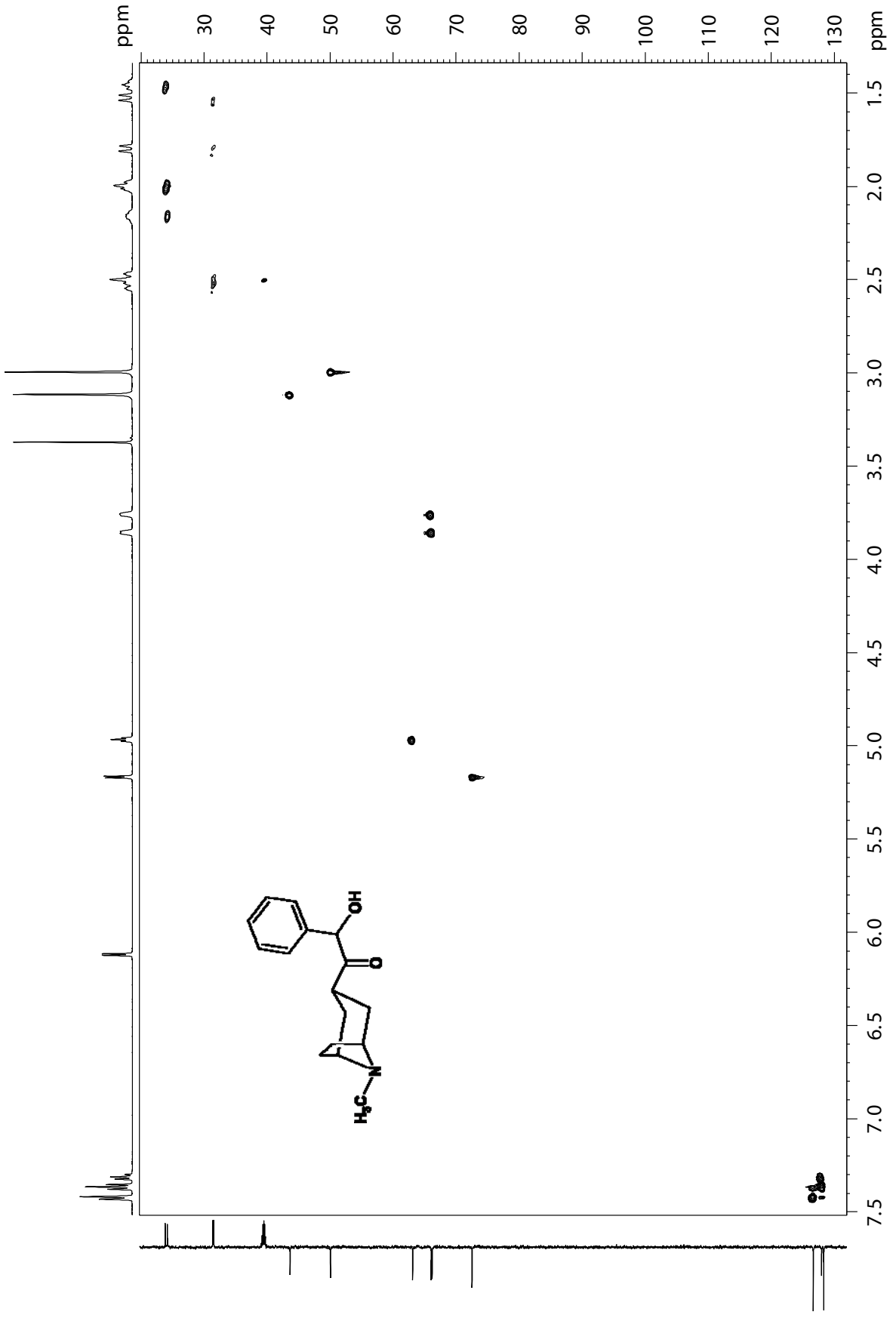
108. Metil-homatropinium-bromid ^{13}C-JMOD NMR spektrum (DMSO)



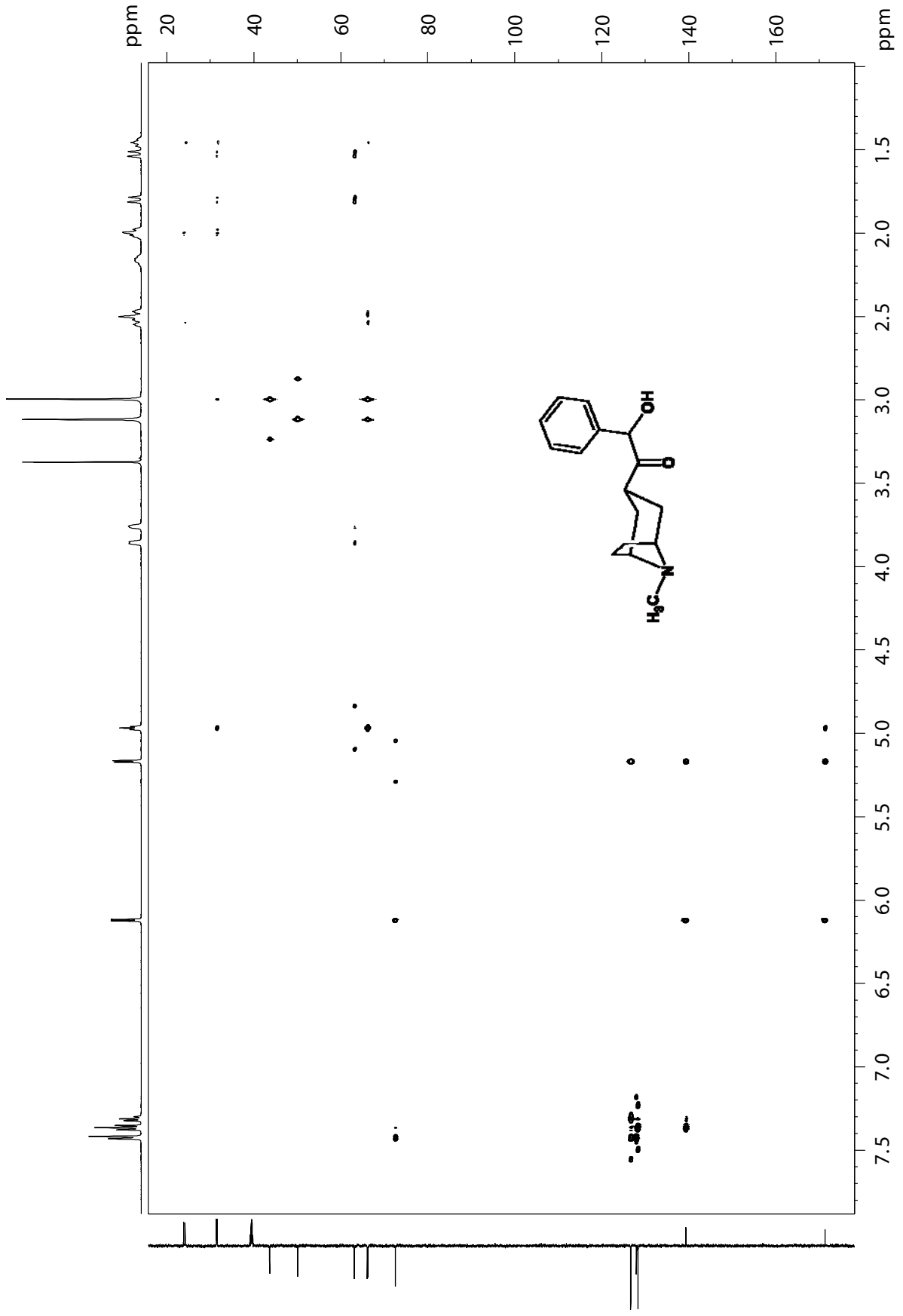
109. Metil-homatropinium-bromid ^{1}H-^{1}H COSY-NMR spektrum (DMSO)



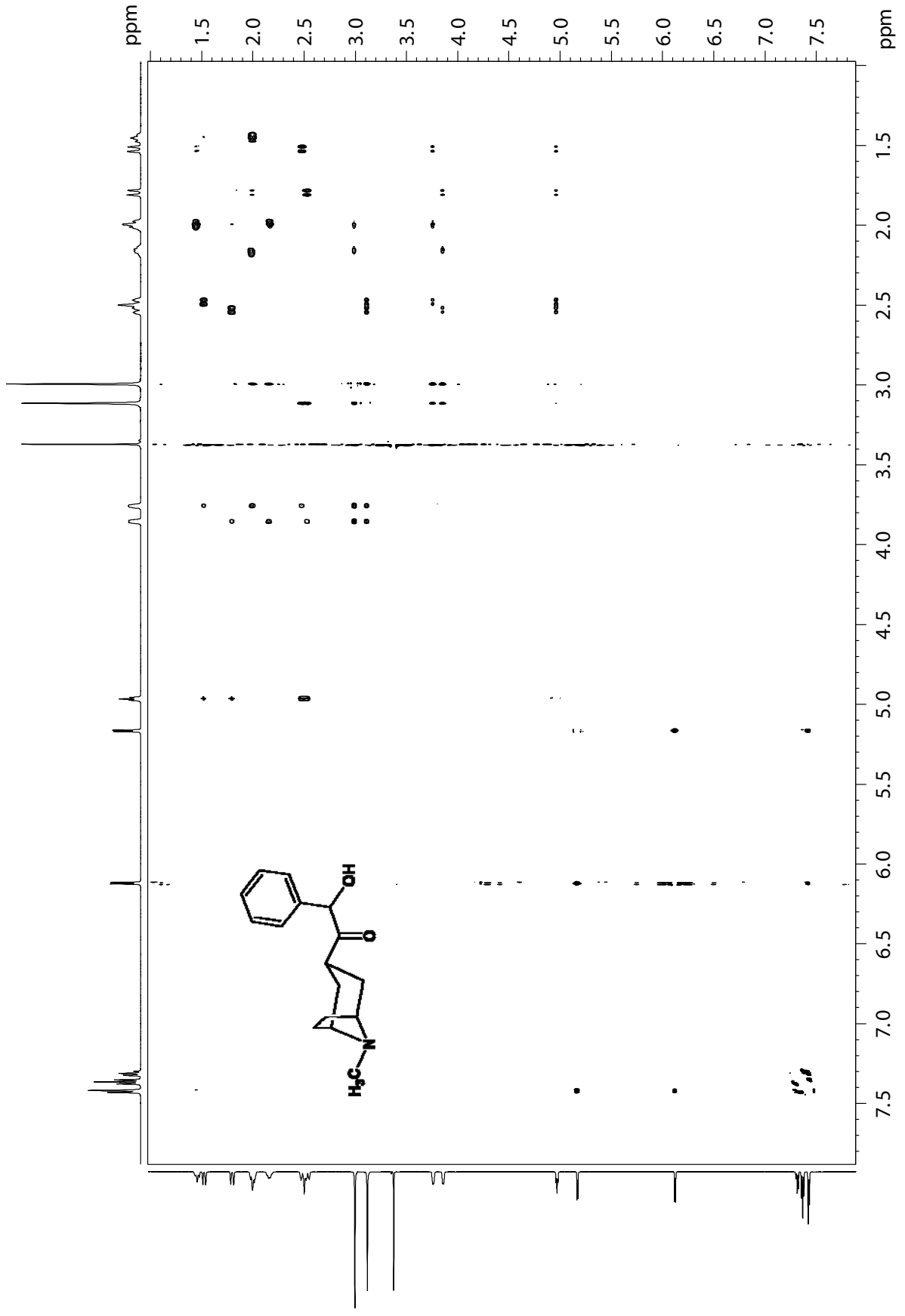
110. Metil-homatropinium-bromid ^{1}H-^{13}C HSQC-NMR spektrum (DMSO)



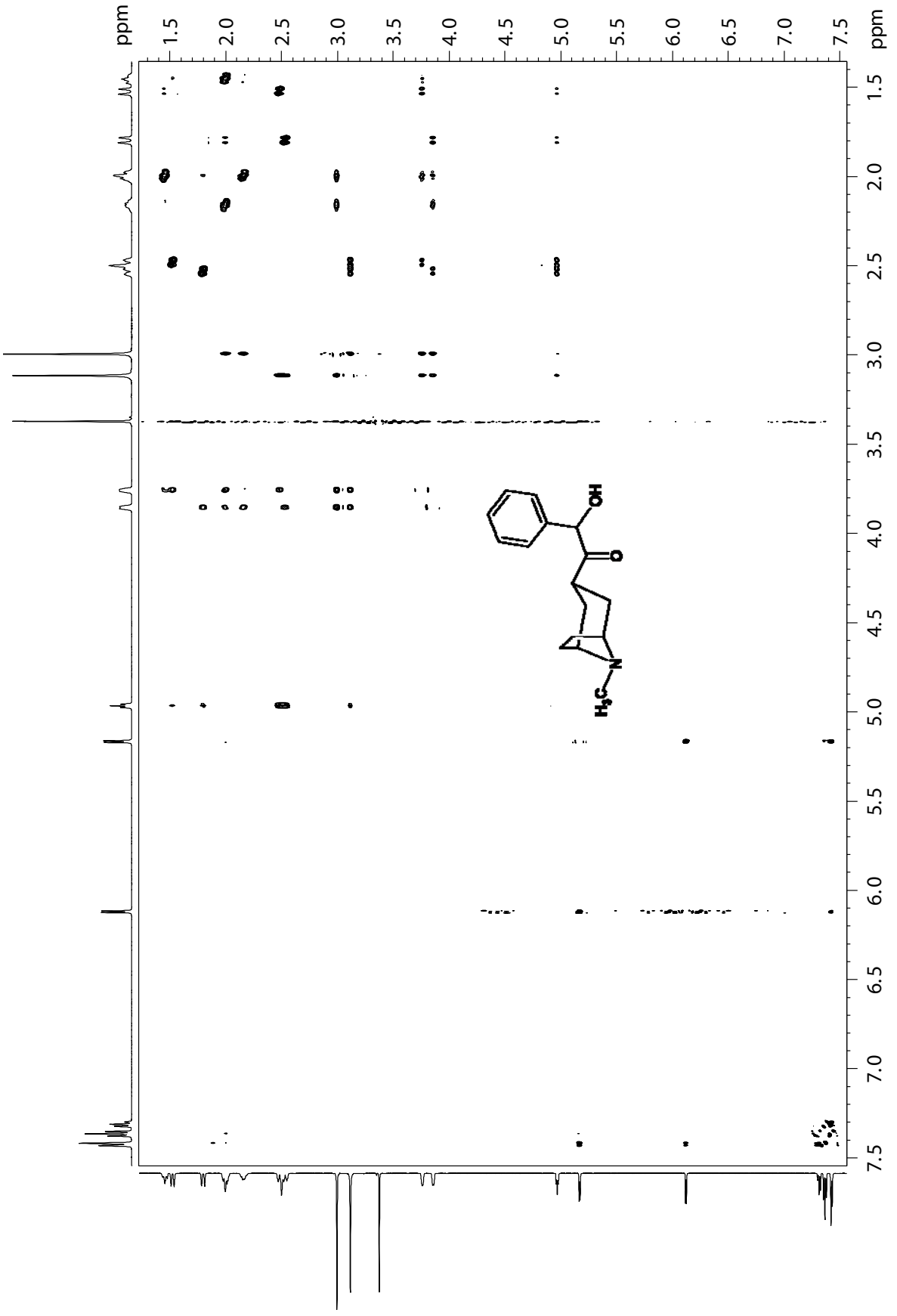
111. Metil-homatropinium-bromid ^{1}H-^{13}C HMBC-NMR spektrum (DMSO)



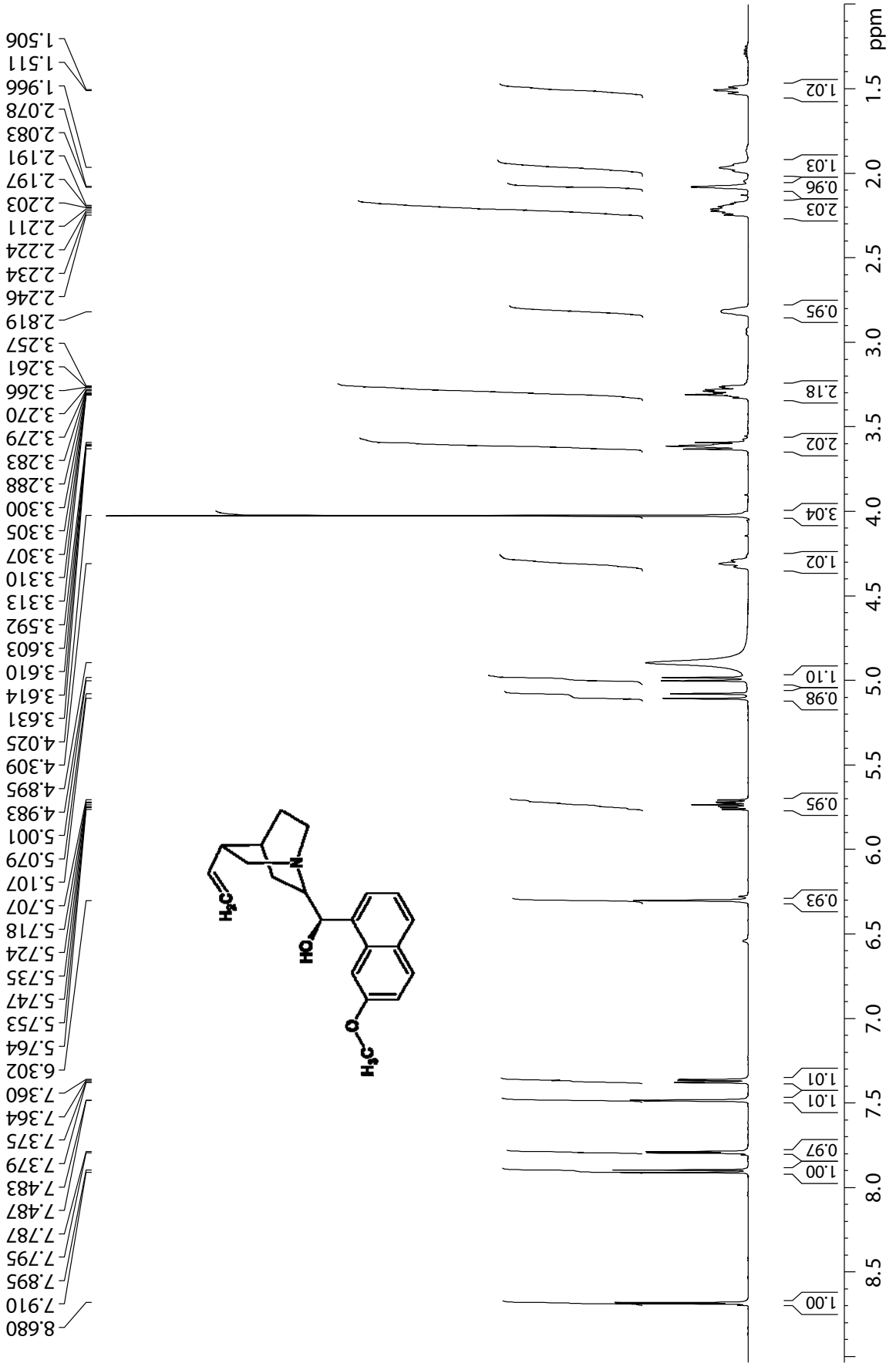
112. Metil-homatropinium-bromid ^{1}H-^{1}H NOESY-NMR spektrum (DMSO)



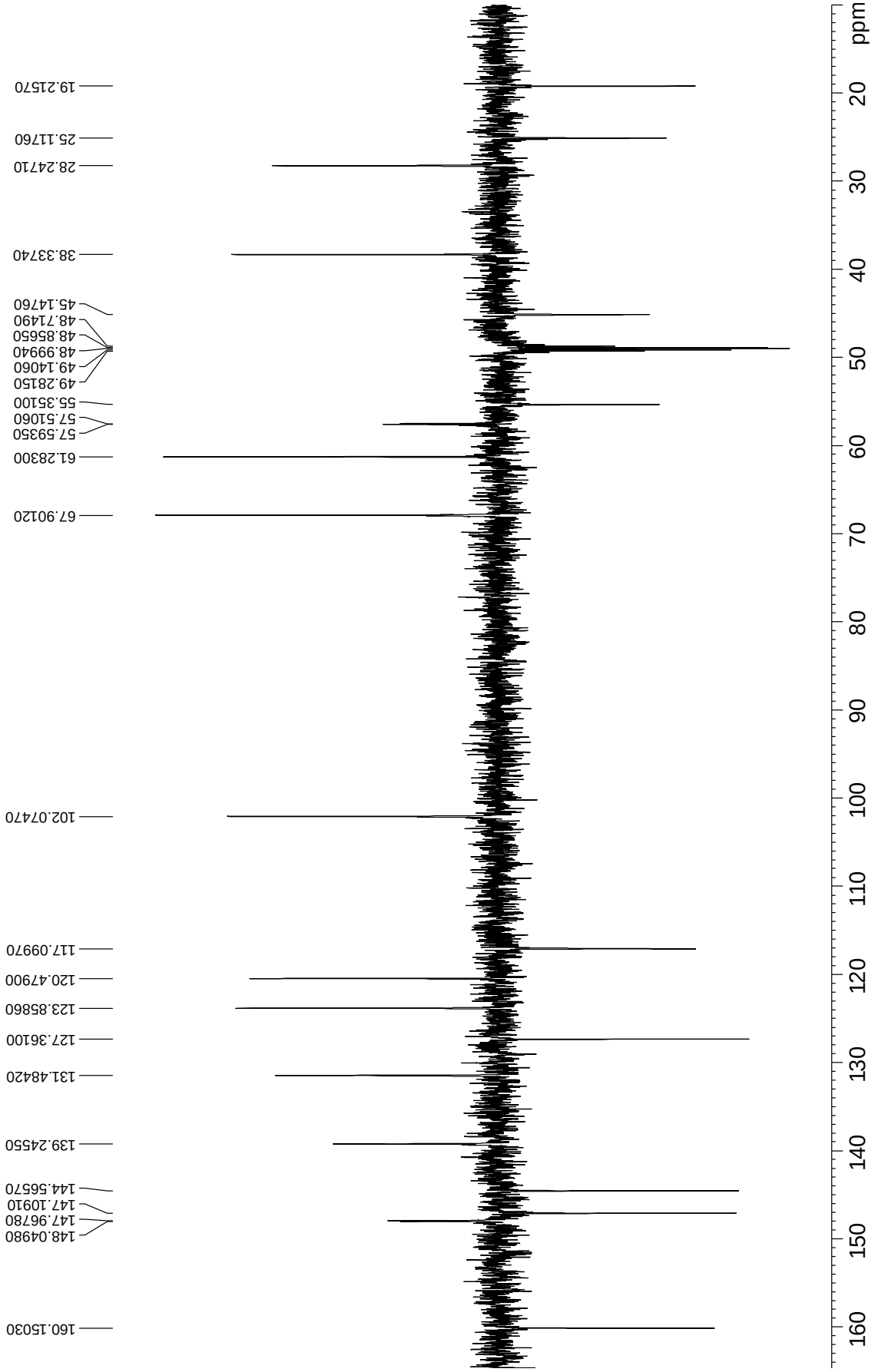
113. Metil-homatropinium-bromid ^{1}H-^{1}H ROESY-NMR spektrum (DMSO)



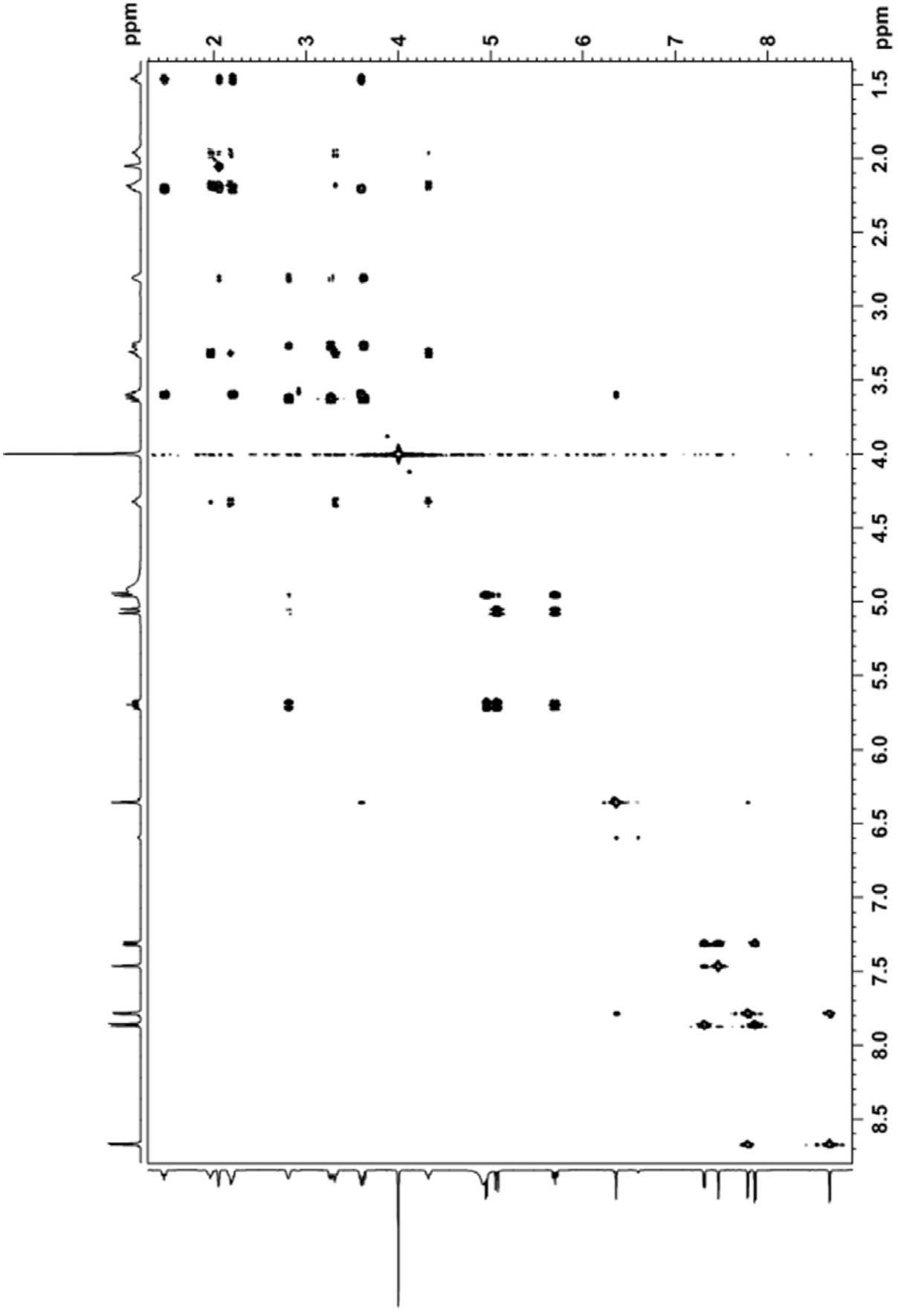
114. Kinin-szulfát ^{1}H-NMR spektrum (MeOD)



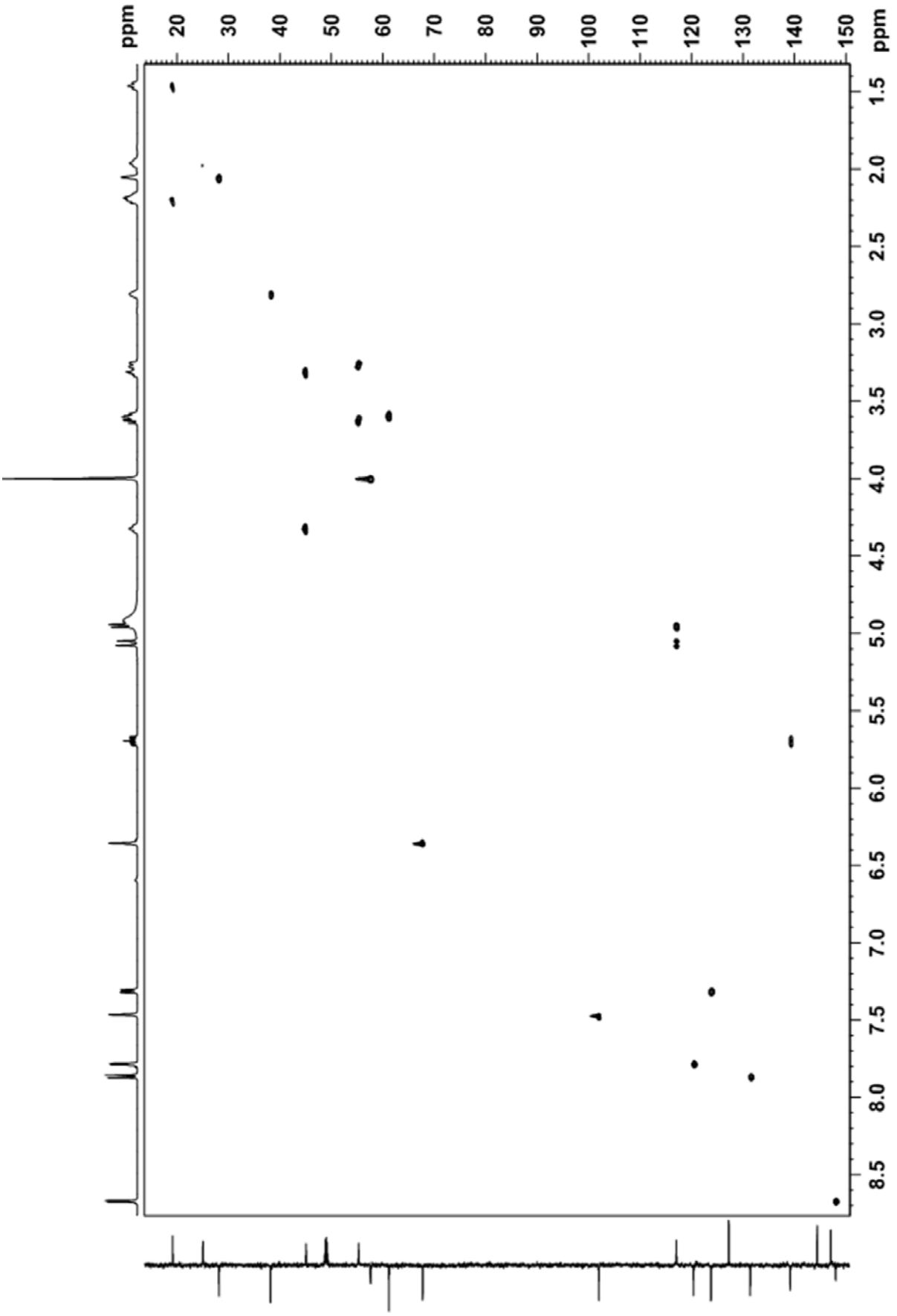
115. Kinin-szulfát ^{13}C-JMOD NMR spektrum (MeOD)



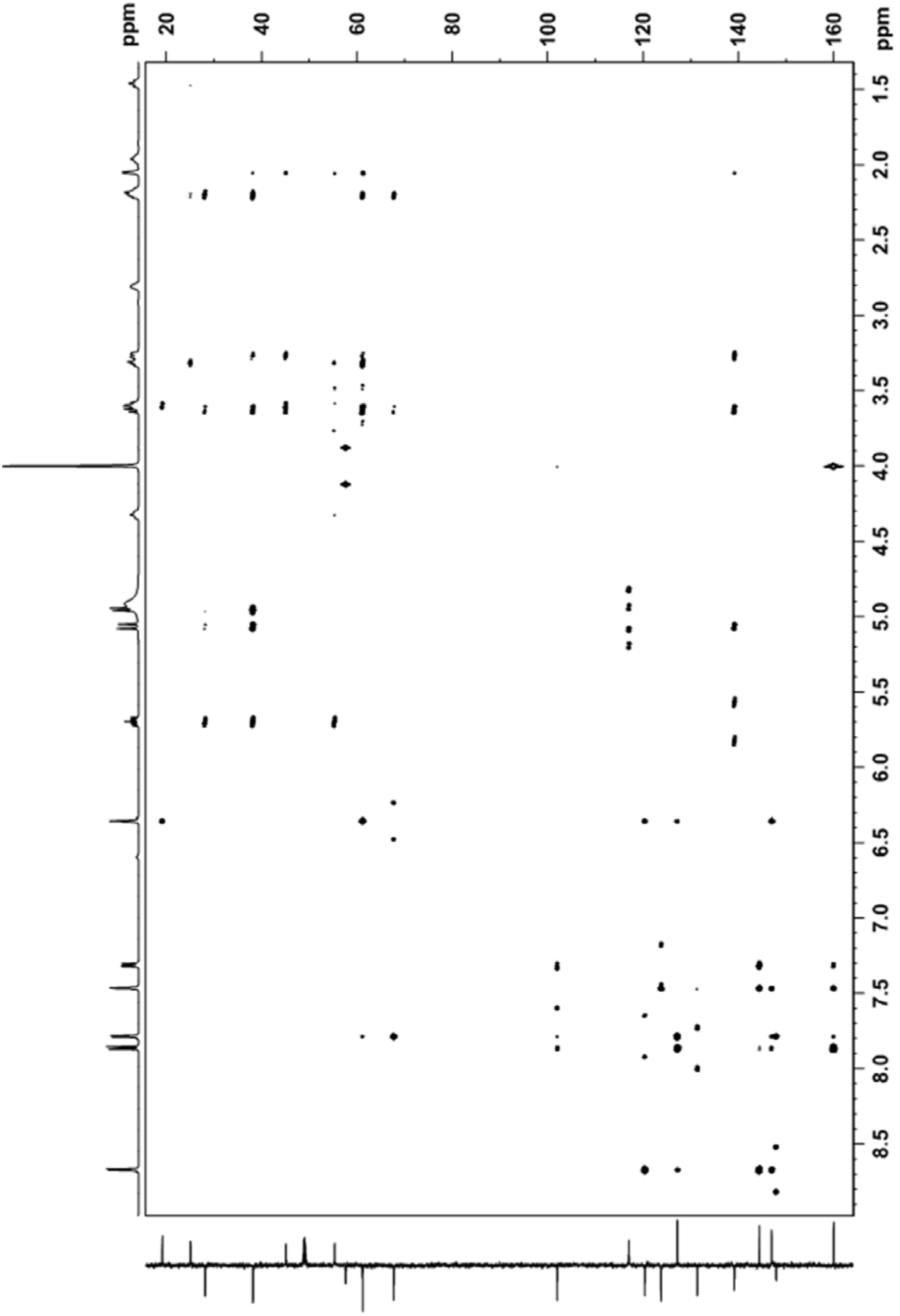
116. Kinin-szulfát ^{1}H-^{1}H COSY-NMR spektrum (MeOD)



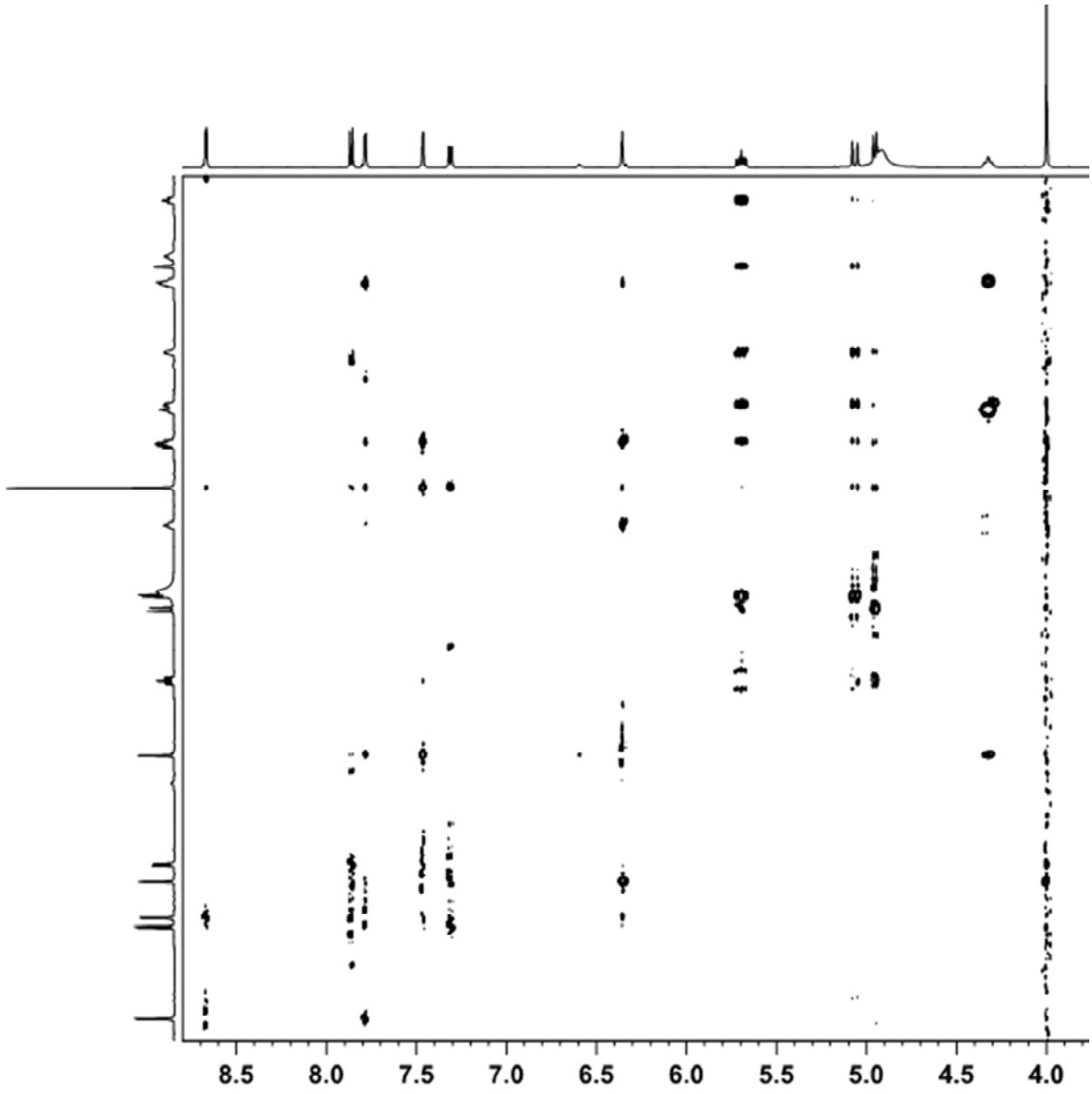
117. Kininszulfát ^{1}H-^{13}C HSQC-NMR spektrum (MeOD)



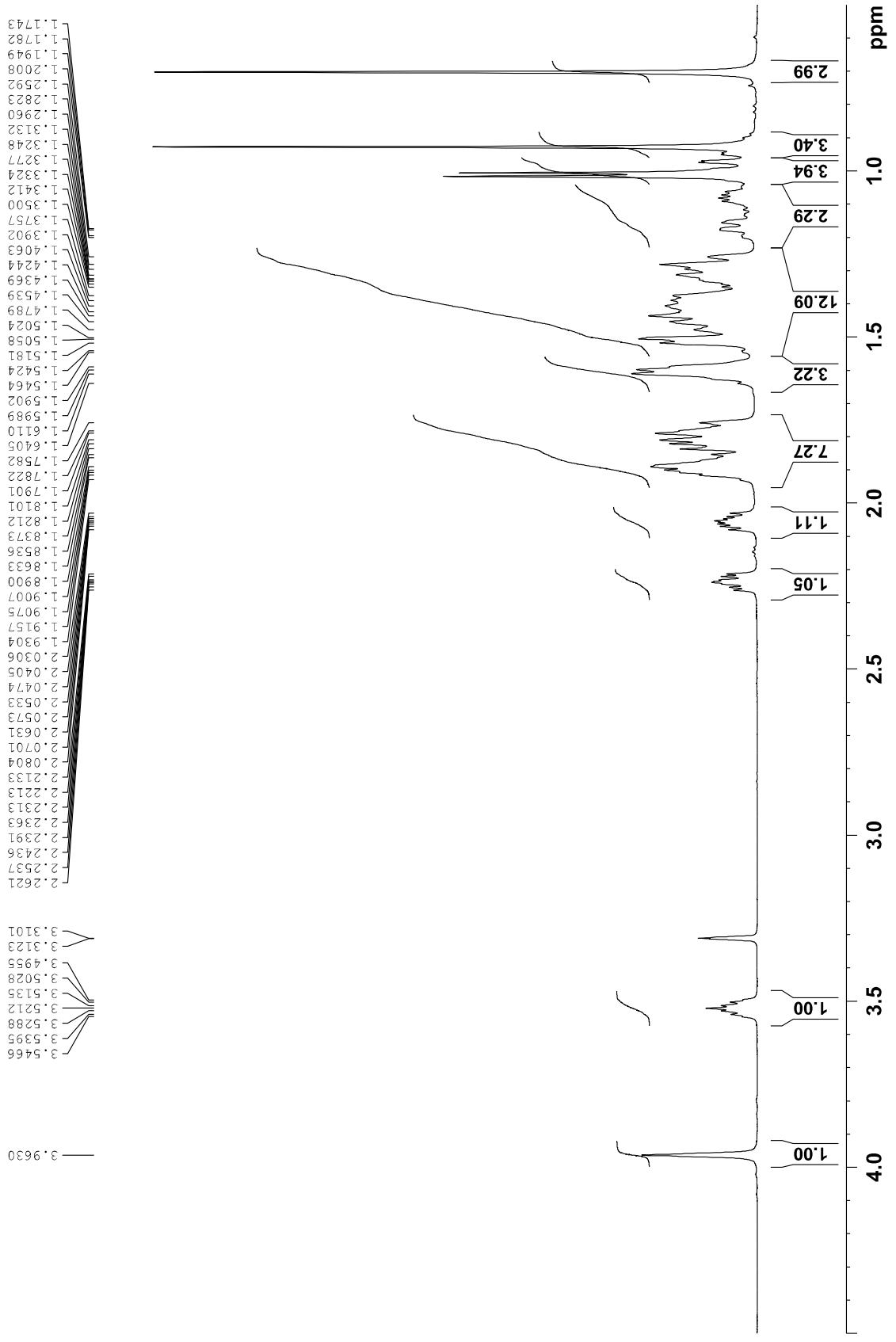
118. Kinin-szulfát ^{1}H-^{13}C HMBC-NMR spektrum (MeOD)



119. Kinin-szulfát ^{1}H-^{1}H ROESY-NMR spektrum (MeOD)



120. 3α,12α-dihidroxi kolsav Na só, ^{1}H-NMR spektrum (MeOD)



121. 3α,12α-dihidroxi-kolsav Na só, ^{13}C-JMOD NMR spektrum (MeOD)