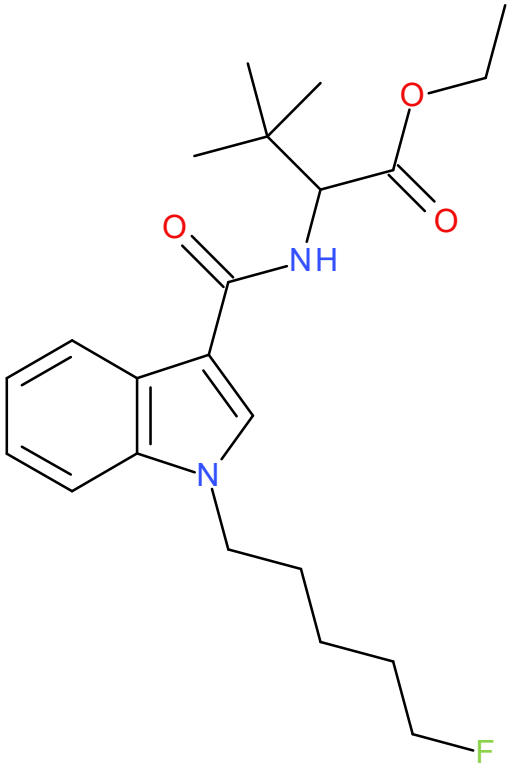
5F-EDMB-PICA



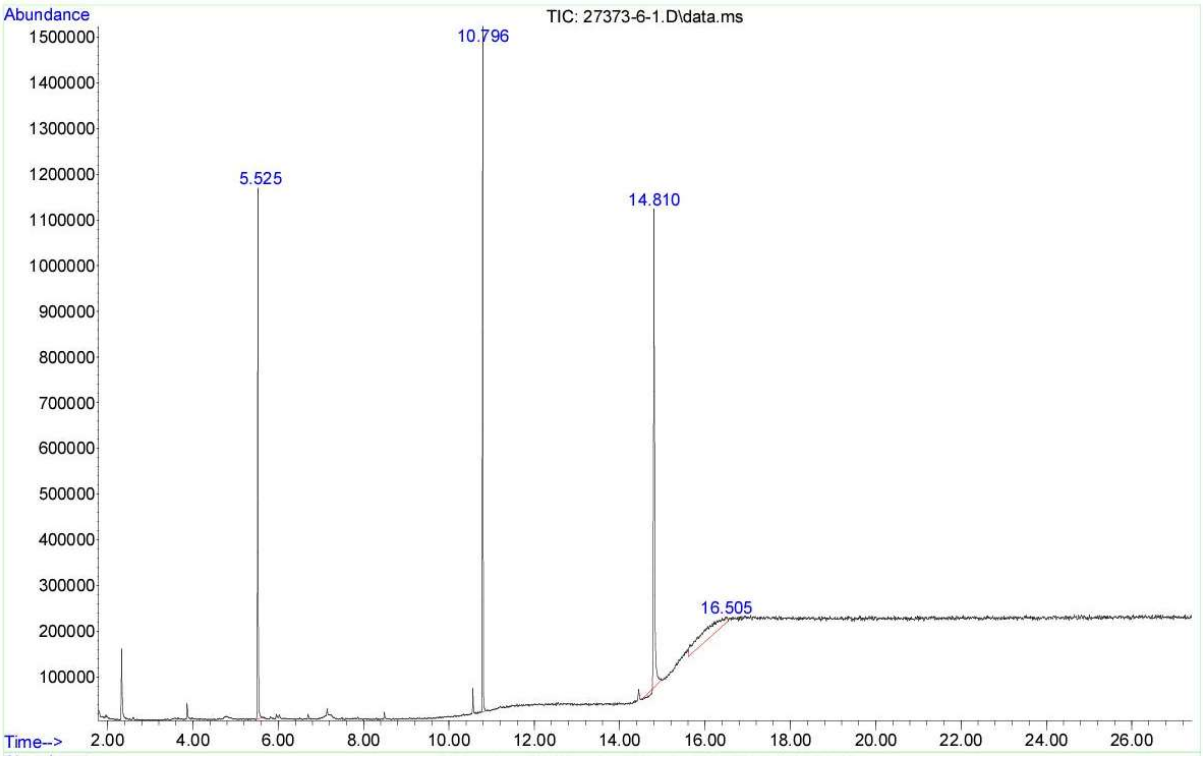
ethyl 2-[[1-(5-fluoropentyl)indole-3-carbonyl]amino]-3,3-dimethyl-butanoate
Formula: $C_{22}H_{31}FN_2O_3$
Formula weight: 390.23
Chemical Abstracts No.: *n. a.*
Smiles code: CCOC(=O)C(NC(=O)c1cn(CCCCCF)c2ccccc12)C(C)(C)C
InChi key: RNWBJOCYFGGMRJ-UHFFFAOYSA-N
Other names: 5-fluoro EDMB-2201, EDMB-5F-PICA

The first evidence was 2.053 grams brown herbal fractures (tabaco) distributed into 13 packets

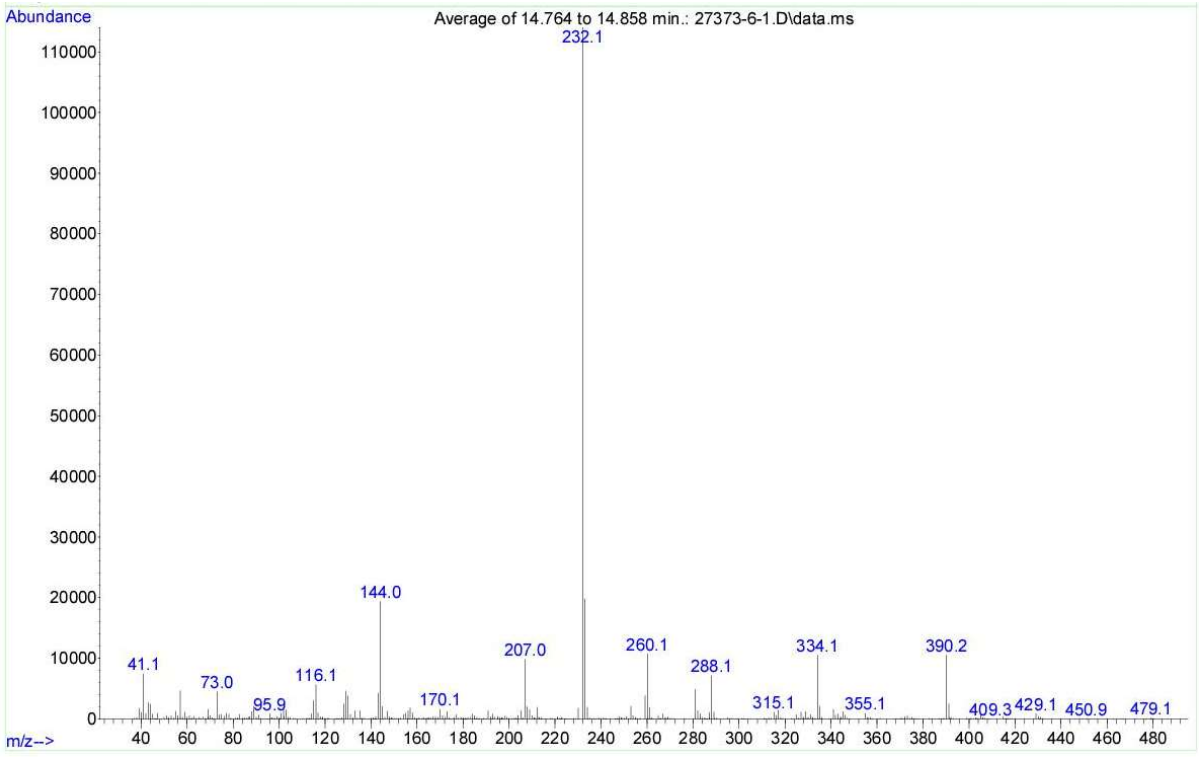
GC-MS

An Agilent 6890N Network GC system set up with Agilent HP-5MS (length: 30 m, diameter: 0.25 mm, film: 0.25 mm) coupled to an Agilent 5973 Network Mass Selective Detector (scan range m/z 35 – m/z 500) was used. The acetonic extract of the herbal leaves was injected. Samples were subjected to electron ionization (EI) mode. GC-MS conditions: HP-5MS column was temperature programmed from 100 ˚C (which was held for 2 minutes) to 280 ˚C at 20 ˚C/min, 280°C was held for 3 minutes, then to 315 ˚C at 25 ˚C/min, the temperature was stated at 315 ˚C for 12 minutes. The carrier gas was helium. Tribenzyl-amine was applied as an internal standard (locked to 10.8 minutes). Data handling was carried out with GC/MSD ChemStation software.

GC-MS total ion chromatogram



Mass spectrum at 14.80 min retention time



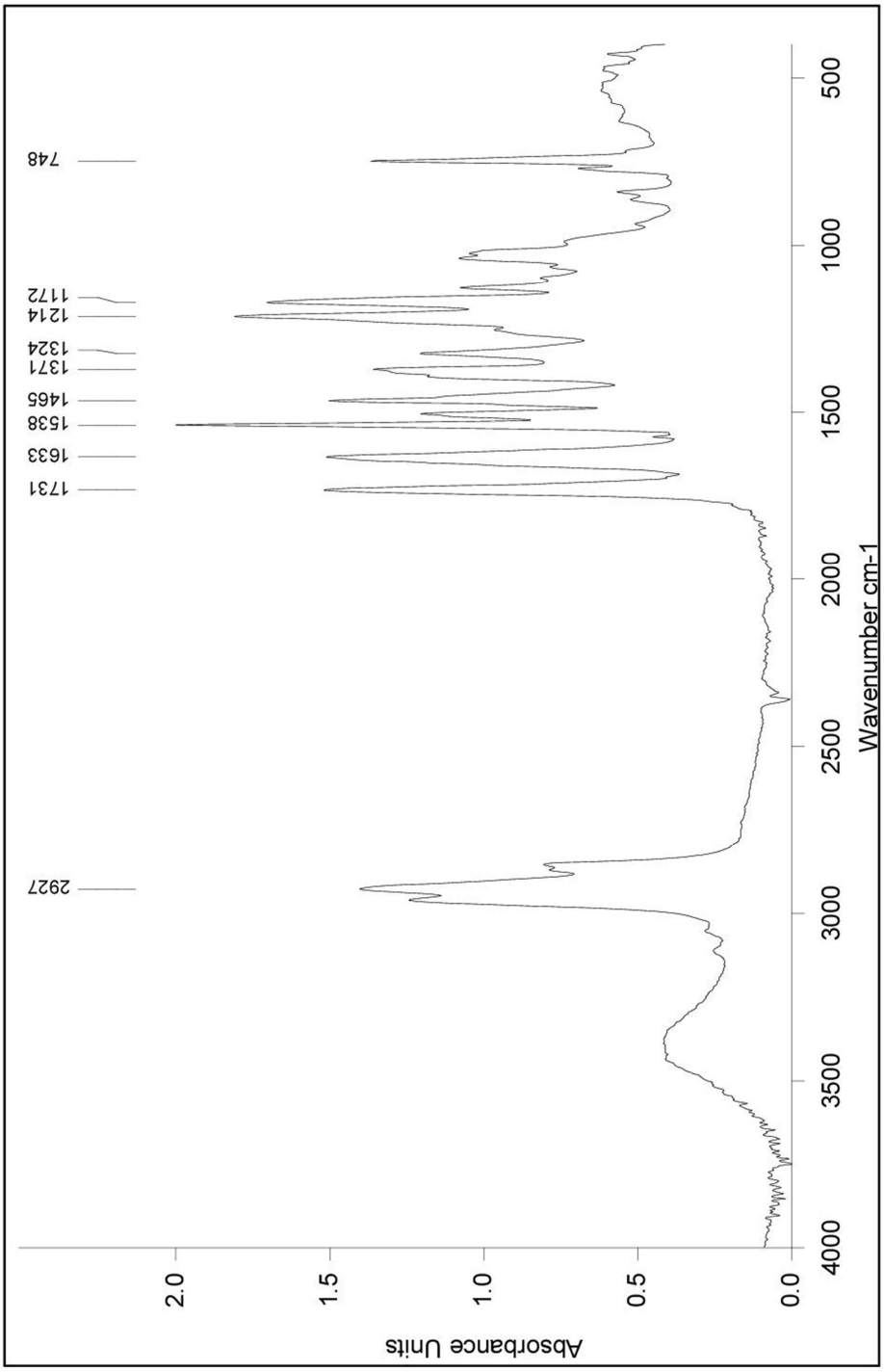
The component at 5.52 min retention time is identified as nicotine.

Agilent 6890N Network GC system set up with Agilent HP-5MS

IR

The IR spectrum was recorded on a Bruker Tensor 27 IR spectrometer equipped with a Platinum ATR accessory, in absorbance mode. The evidence was extracted by acetone, the solution was dribbled onto the surface of the ATR accessory and and the solvent was evaporated. The digital resolution is 4 cm^{-1}. The spectrometer was controlled, and the data were processed using Opus 6.5 software package.

IR spectrum of the evidence



Bruker Tensor 27

GC-IRD

An Agilent 6890N Network GC system set up with Agilent HP-5MS (length: 30 m, diameter: 0.25 mm, film: 0.25 mm) coupled to a Dani DiscovIR FT-IR spectrometer was used. GC conditions: Split ratio: 1:5, HP-5MS column was temperature programmed from 100 ℃ (which was held for 2 minutes) to 280 ℃ at 20 ℃/min, 280°C was held for 3 minutes, then to 315 ℃ at 25 ℃/min, the temperature was stated at 315 ℃ for 12 minutes. The carrier gas was helium. Tribenzyl-amine was applied as an internal standard. IRD conditions: Deposition tip: 280 °C, Restrictor: 280 °C, Transfer Line: 280 °C, Disk temperature:- 40 °C, Dewar Cap temperature: 30 °C. Data handling was carried out with GRAMS software.

GC-IR chromatogram of the sized material

DiscovIR conditions

Peaks file: C:/Data/200812/20_27373-61/reproc/20_27373-61.Peaks.spc
Sample name: 20_27373-61
Chemist: BD
Date collected: 08/12/2020 10:17
Date analyzed:
Disk speed: 3 mm/min
Vial number: 2

Run conditions	Start	End
Transfer line temperature (°C):	280	280
Oven tempurature (°C):	280	280
Restrictor temperature (°C):	281	278
Disk temperature (°C):	-40	-40
Data collect (minutes):	2.50	28.00
Track pointer:	69747	133429
Chamber pressure (Torr):	2.800e-4	2.800e-4

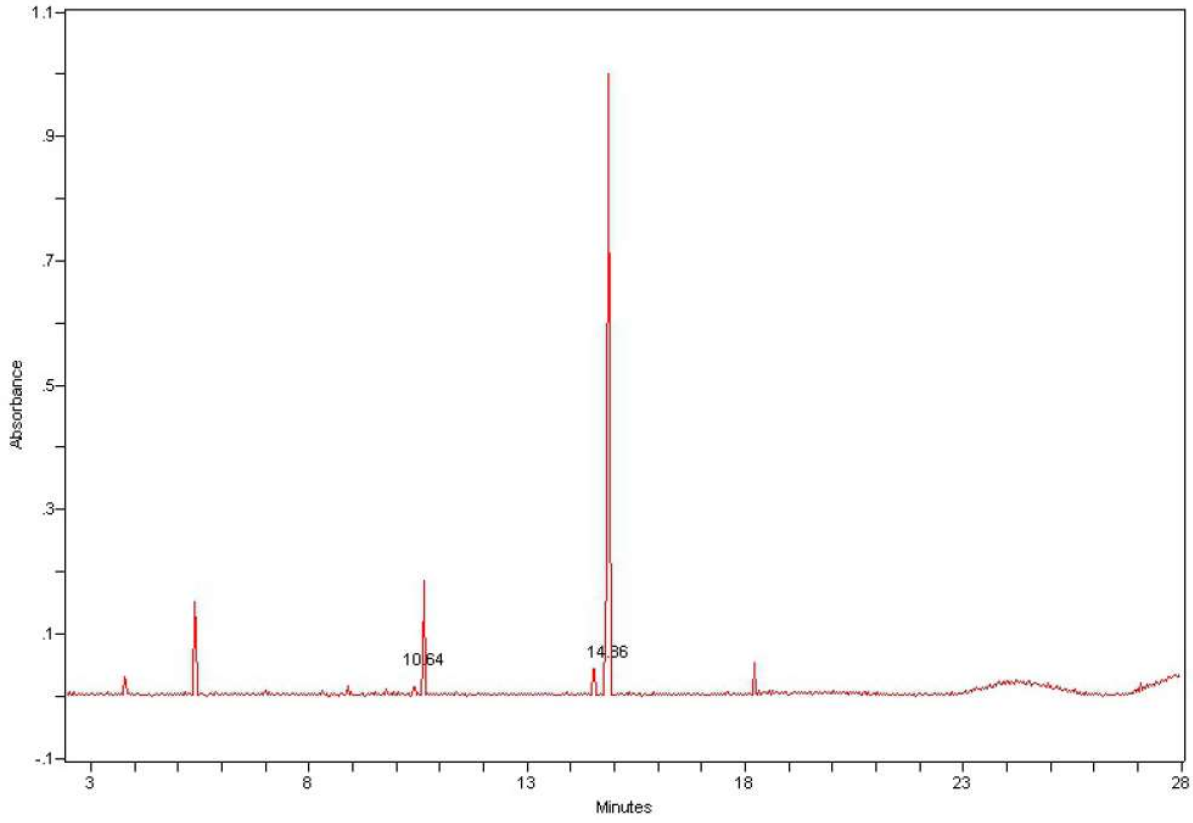
Comment: 27373/20 6.1. / MeOH (5F-EDMB-PICA)

Chemstation conditions

Method name: KABSEQ2_5S.M
Run time: 27.40 minutes
Split mode: SPLIT
Split ratio: 6 : 1
Injection port temperature (°C): 300
Injection volume (µL): 1
Gas pressure (psi): 24.10
GC column: HP-5ms Ultra Inert : 30 m x 250 µm x 0.25 µm film
Oven Temperature: 100°C for 2 min, then 20°C/min to 280°C, hold 3 min, then 25°C/min to 315°C, hold 12 min

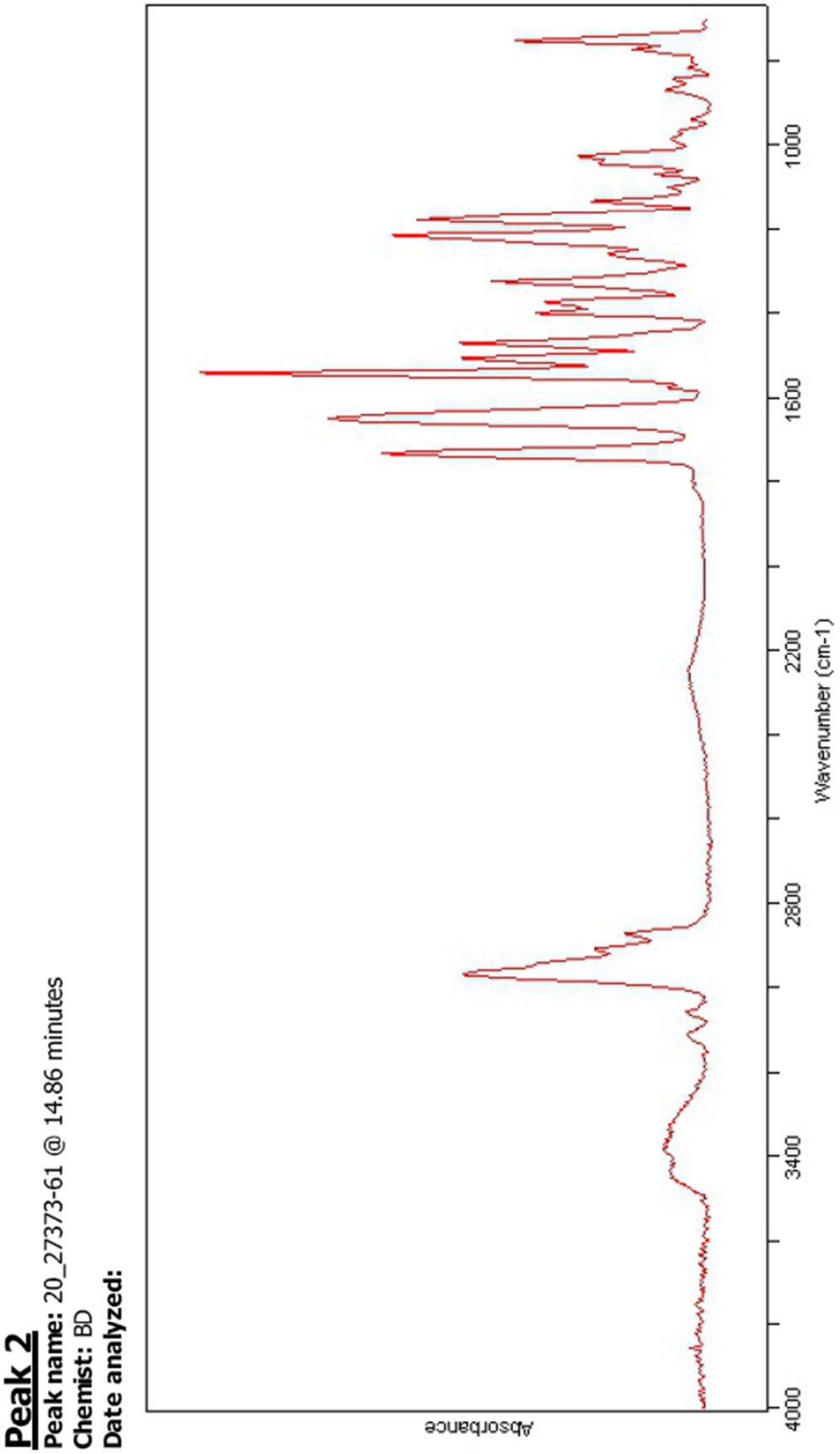
Chromatogram

Data file: C:/Data/200812/20_27373-61/reproc/20_27373-61.Multifile.cgm



Agilent 6890N Network GC system coupled to Dani DiscovIR FT-IR spectrometer

IR spectrum of the reported component at 14.86 min retention time



Agilent 6890N Network GC system coupled to Dani DiscovIR FT-IR spectrometer

NMR

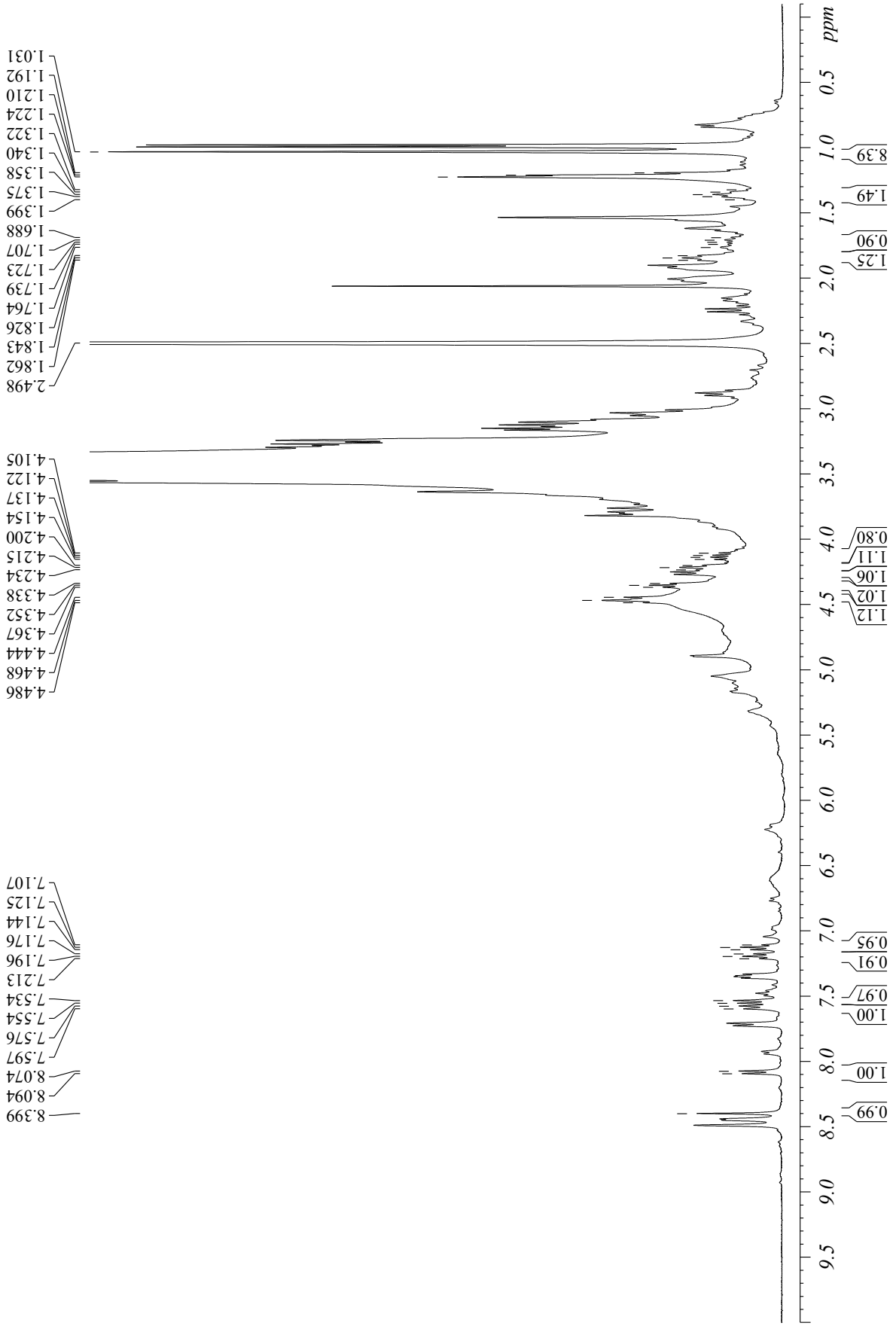
The NMR spectra were recorded on a Bruker Avance Neo 400 NMR spectrometer operating at 9.4 Tesla magnetic field, equipped with Prodigy BBO-H&F-D-05 Z-gradient probe. The spectra were recorded at 25°C in DMSO-d_6 solution. The spectrometer was controlled, and the data were processed using TopSpin 4.0 software package. Chemical shifts (δ) are given in parts per million unit, referenced to tetramethylsilane (δ_{TMS} = 0.00 ppm). The determination of the structure was based on ^{1}H, ^{13}C, DEPT-135, multiplicity edited HSQC and HMBC spectra.

The evidence herbal was extracted by DMSO, the extractum was filtered and measured. On the ^{1}H-NMR spectra, only the signals of the reported component are integrated, the peaks of the signals of the reported component and the solvent DMSO are picked. In case of the ^{13}C-NMR spectra, only the peaks of the reported component and the solvent DMSO are picked. The signals of the matrix are not marked.

In case of the 2D spectra, the cross-peaks of the reported compound are marked by red ovals, the cross-peaks of the matrix are not marked.

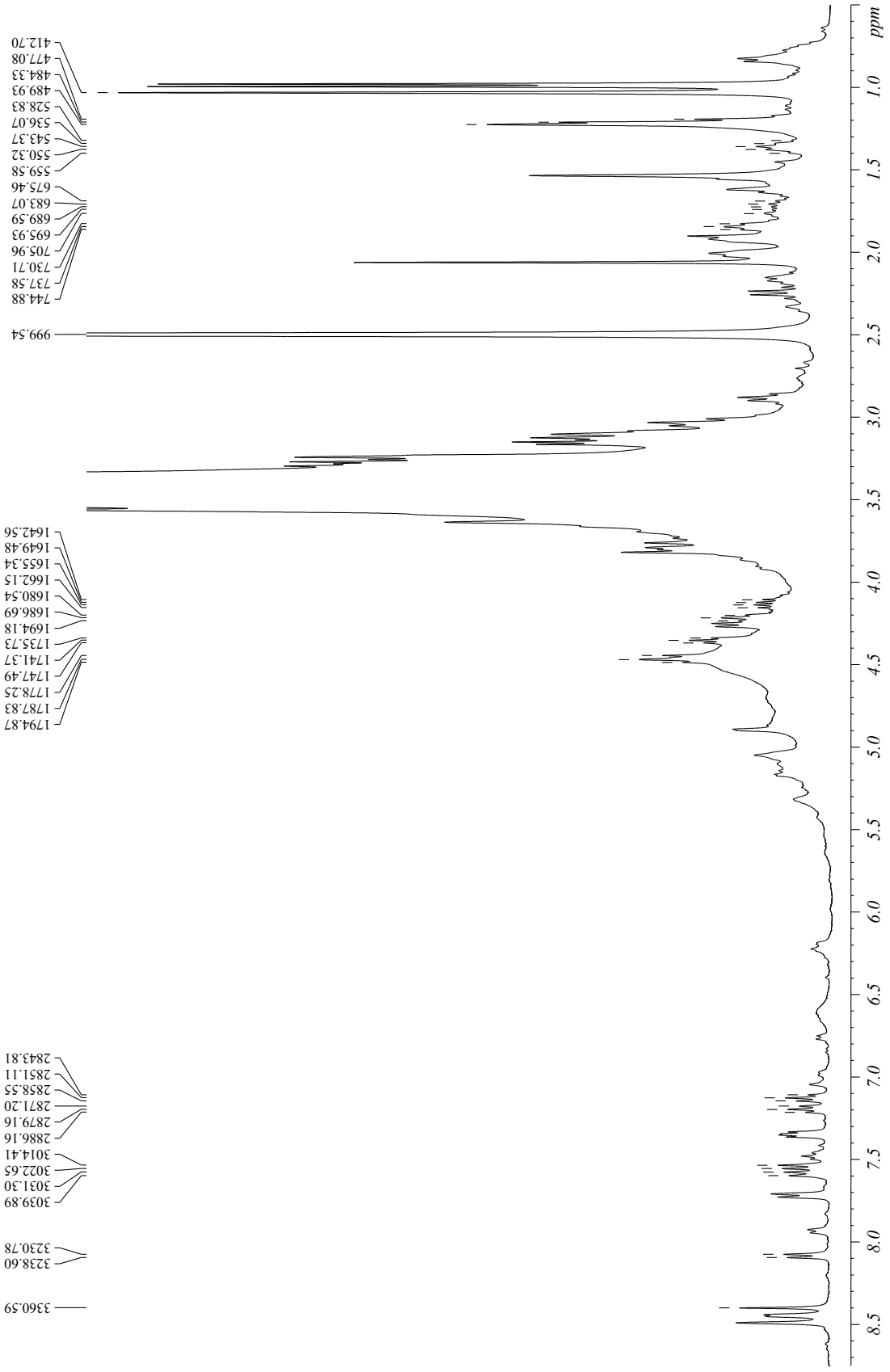
There were nicotine, glicerol, propyleneglycol, glucose identified as components of the matrix extracted from the tabaco.

^{1}H-NMR spectrum (overview)



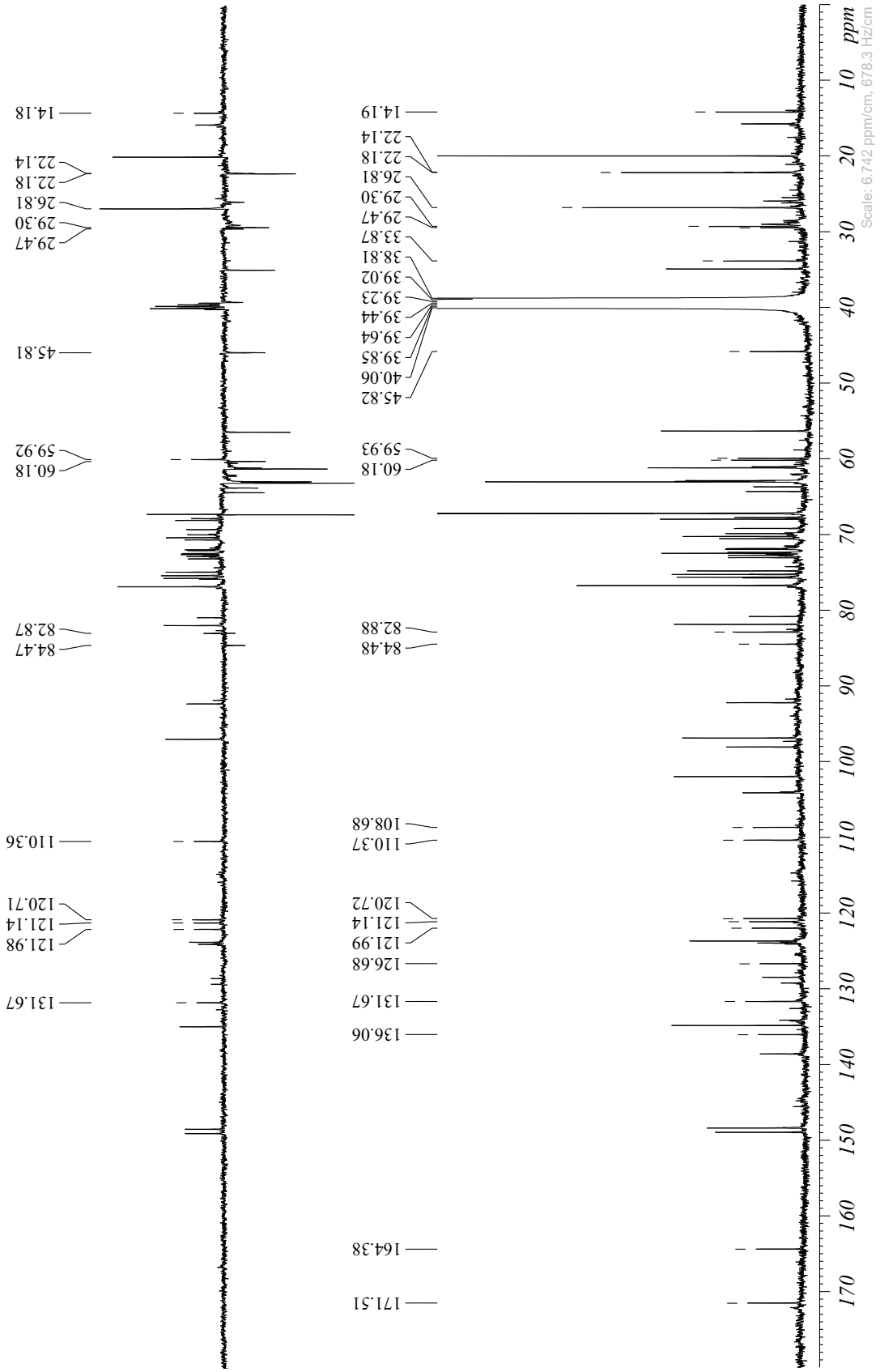
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

^{1}H-NMR spectrum (with Hz list)



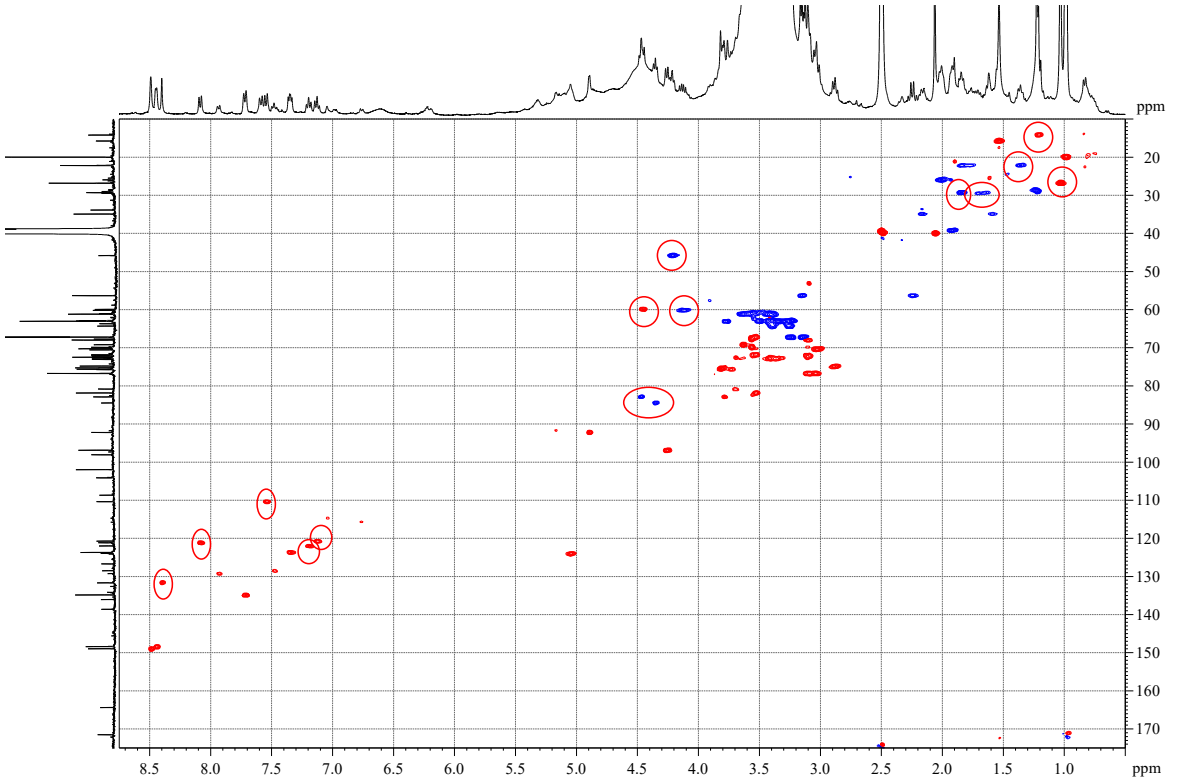
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

^{13}C-NMR and DEPT-135 spectra

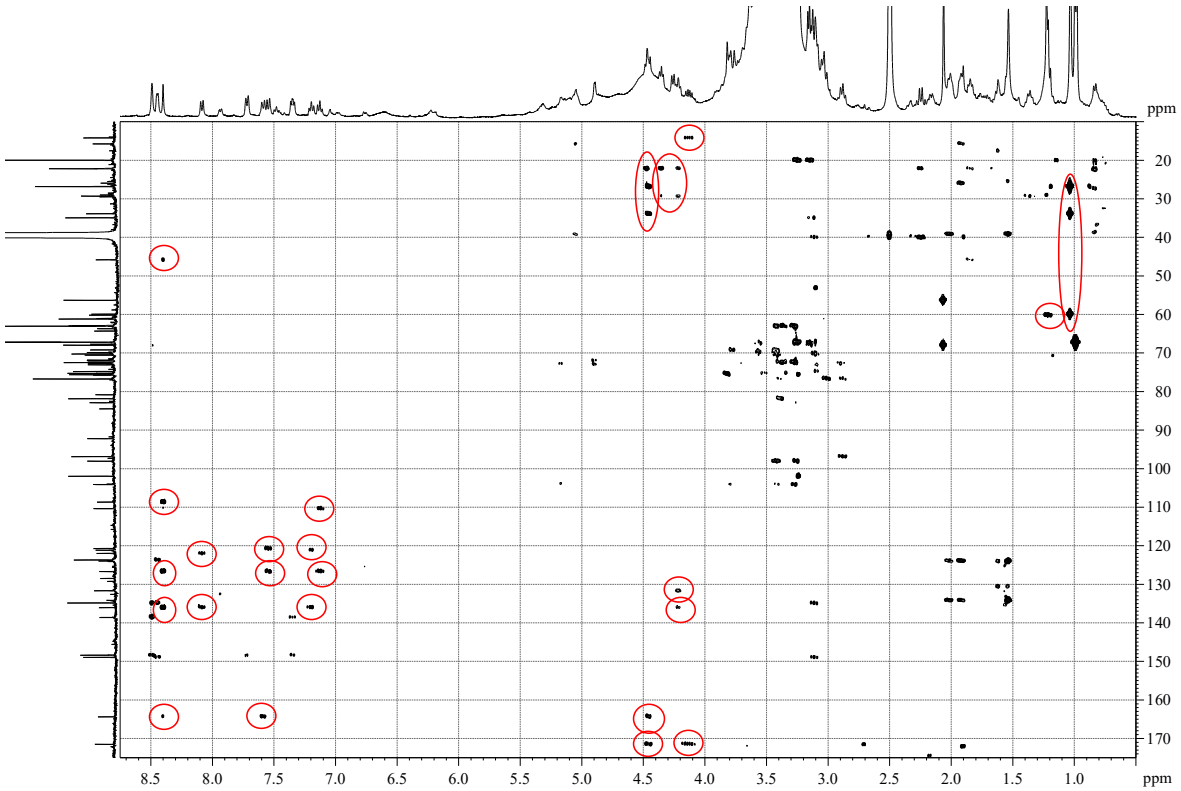


Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

ed-HSQC



HMBC



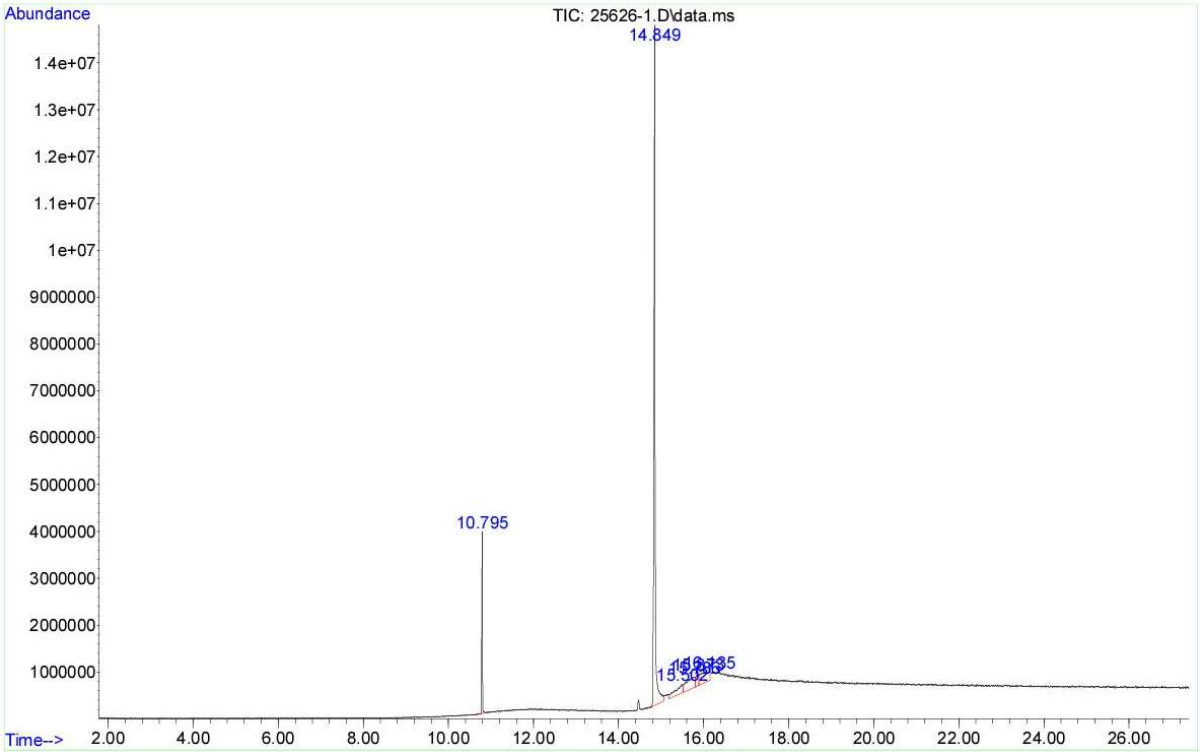
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

The second evidence was 22.66 grams light yellow coloured powder, chemical material.

GC-MS

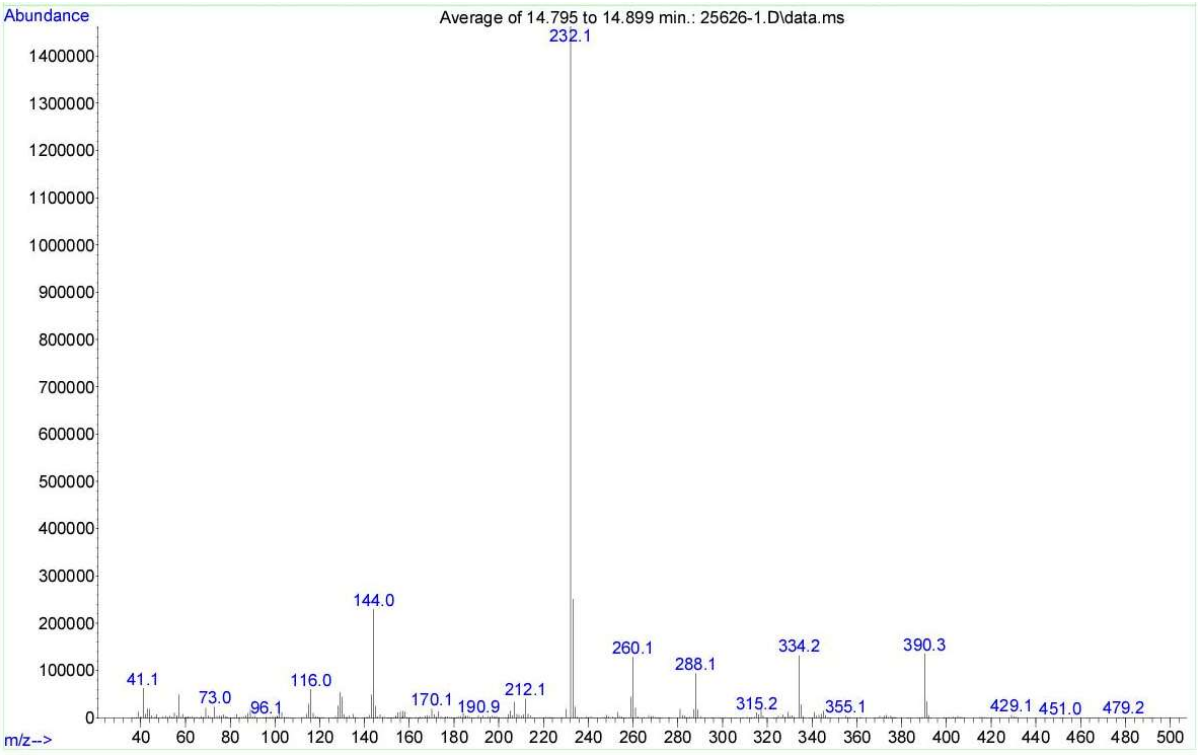
An Agilent 6890N Network GC system set up with Agilent HP-5MS (length: 30 m, diameter: 0.25 mm, film: 0.25 mm) coupled to an Agilent 5973 Network Mass Selective Detector (scan range m/z 35 – m/z 500) was used. The methanolic solution of the evidence was injected. Samples were subjected to electron ionization (EI) mode. GC-MS conditions: HP-5MS column was temperature programmed from 100 ˚C (which was held for 2 minutes) to 280 ˚C at 20 ˚C/min, 280°C was held for 3 minutes, then to 315 ˚C at 25 ˚C/min, the temperature was stated at 315 ˚C for 12 minutes. The carrier gas was helium. Tribenzyl-amine was applied as an internal standard (locked to 10.8 minutes). Data handling was carried out with GC/MSD ChemStation software.

GC-MS total ion chromatogram

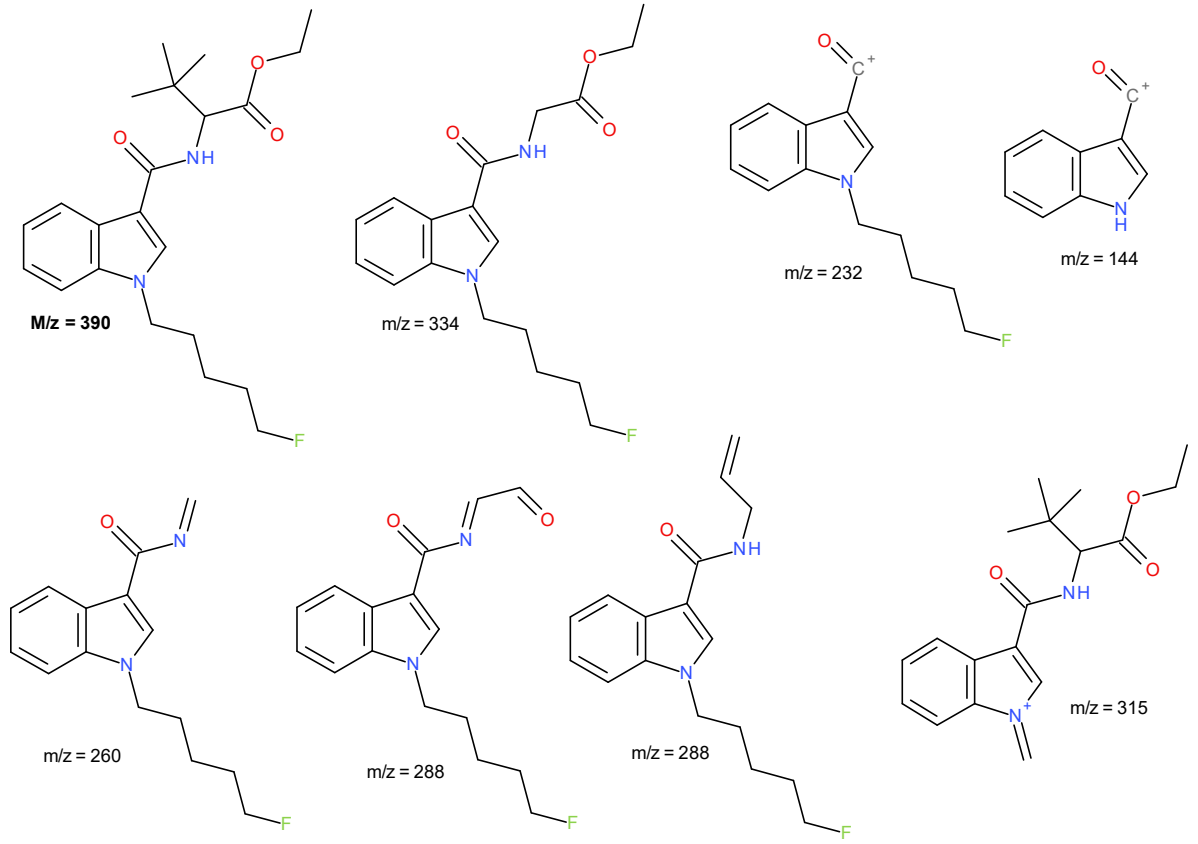


Agilent 6890N Network GC system set up with Agilent HP-5MS

Mass spectrum at 14.85 min retention time



Fragmentation of the compound 5F-EDMB-PICA

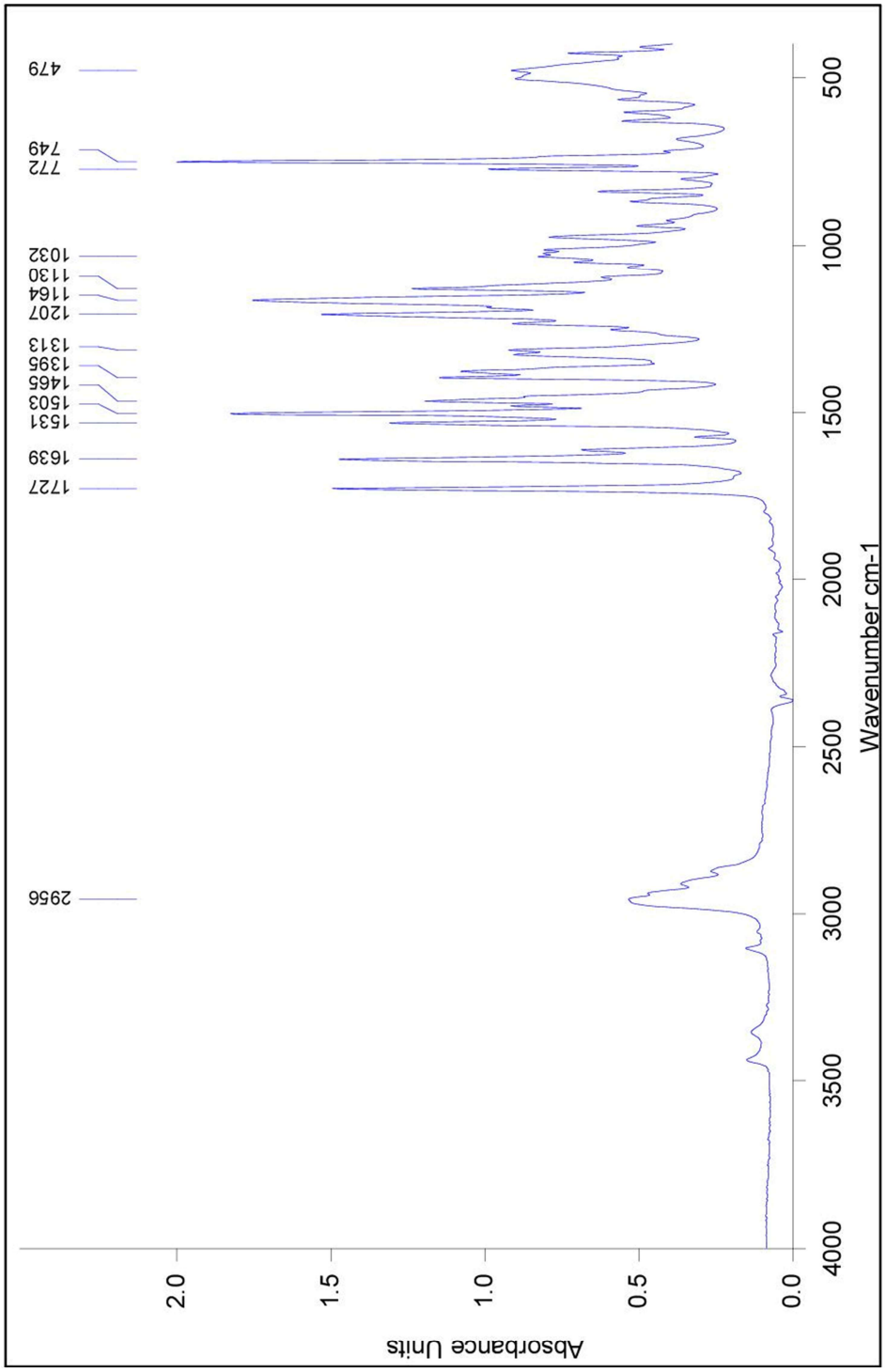


Agilent 6890N Network GC system set up with Agilent HP-5MS

IR

The IR spectrum was recorded on a Bruker Tensor 27 IR spectrometer equipped with a Platinum ATR accessory, in absorbance mode. The evidence was neasured without any sample preparation. The digital resolution is 4 cm^{-1}. The spectrometer was controlled, and the data were processed using Opus 6.5 software package.

IR spectrum of the evidence



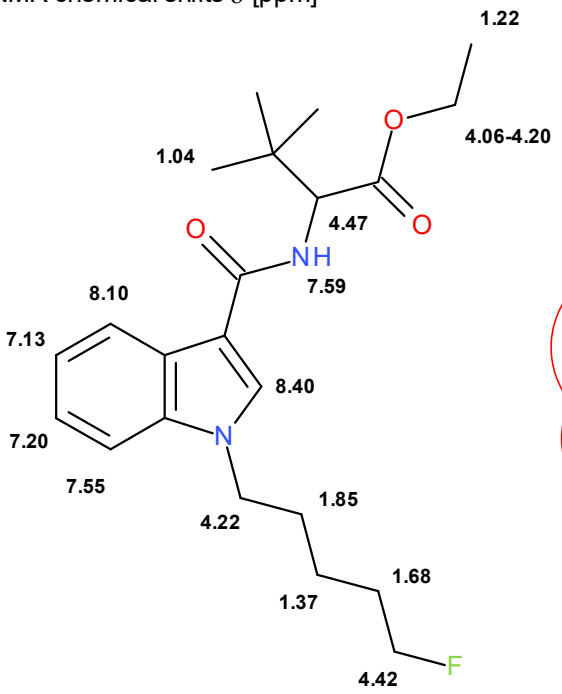
Bruker Tensor 27

NMR

The NMR spectra were recorded on a Bruker Avance Neo 400 NMR spectrometer operating at 9.4 Tesla magnetic field, equipped with Prodigy BBO-H&F-D-05 Z-gradient probe. The spectra were recorded at 25°C in DMSO-d_6 solution. The spectrometer was controlled, and the data were processed using TopSpin 4.0 software package. Chemical shifts (δ) are given in parts per million unit, referenced to tetramethylsilane (δ_{TMS} = 0.00 ppm). The determination of the structure was based on ^{1}H, zqs-clip-COSY, zqs-TOCSY, zqs-easy-ROESY as well as ^{13}C, DEPT-135, multiplicity edited HSQC, double edited HSQC-zqs-clip-COSY and magnitude mode HMBC spectra.

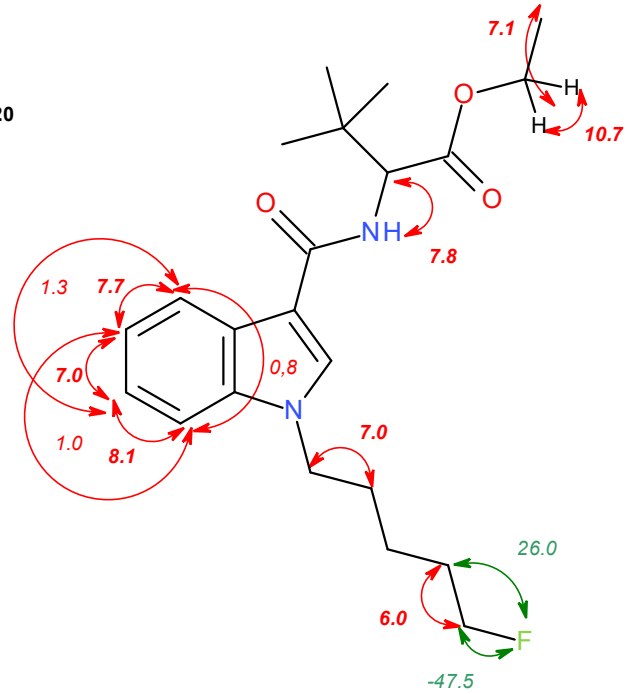
Interpretation of the NMR spectra

^{1}H-NMR chemical shifts δ [ppm]



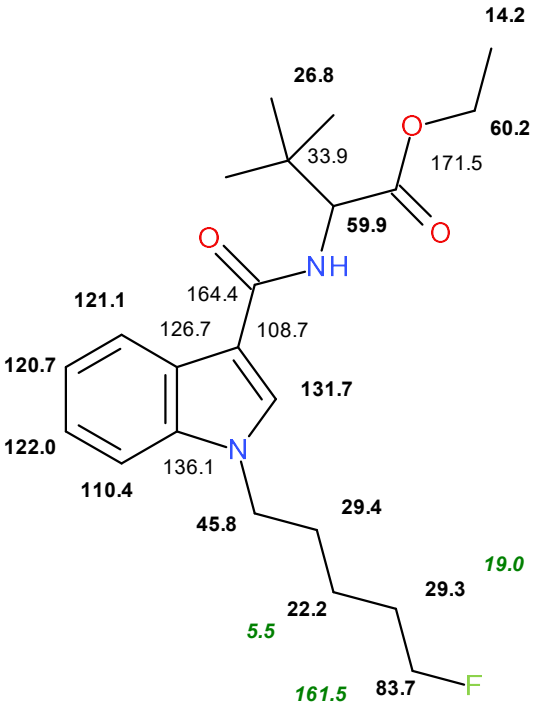
J(H,H) coupling constants [Hz]

J(H,F) coupling constants [Hz]

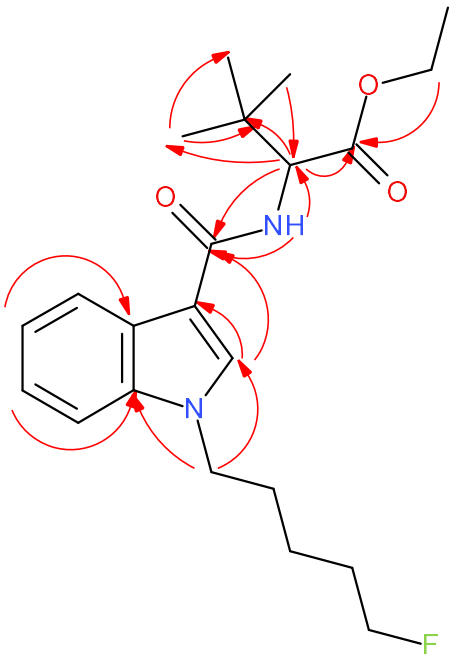


^{13}C-NMR chemical shifts [ppm]

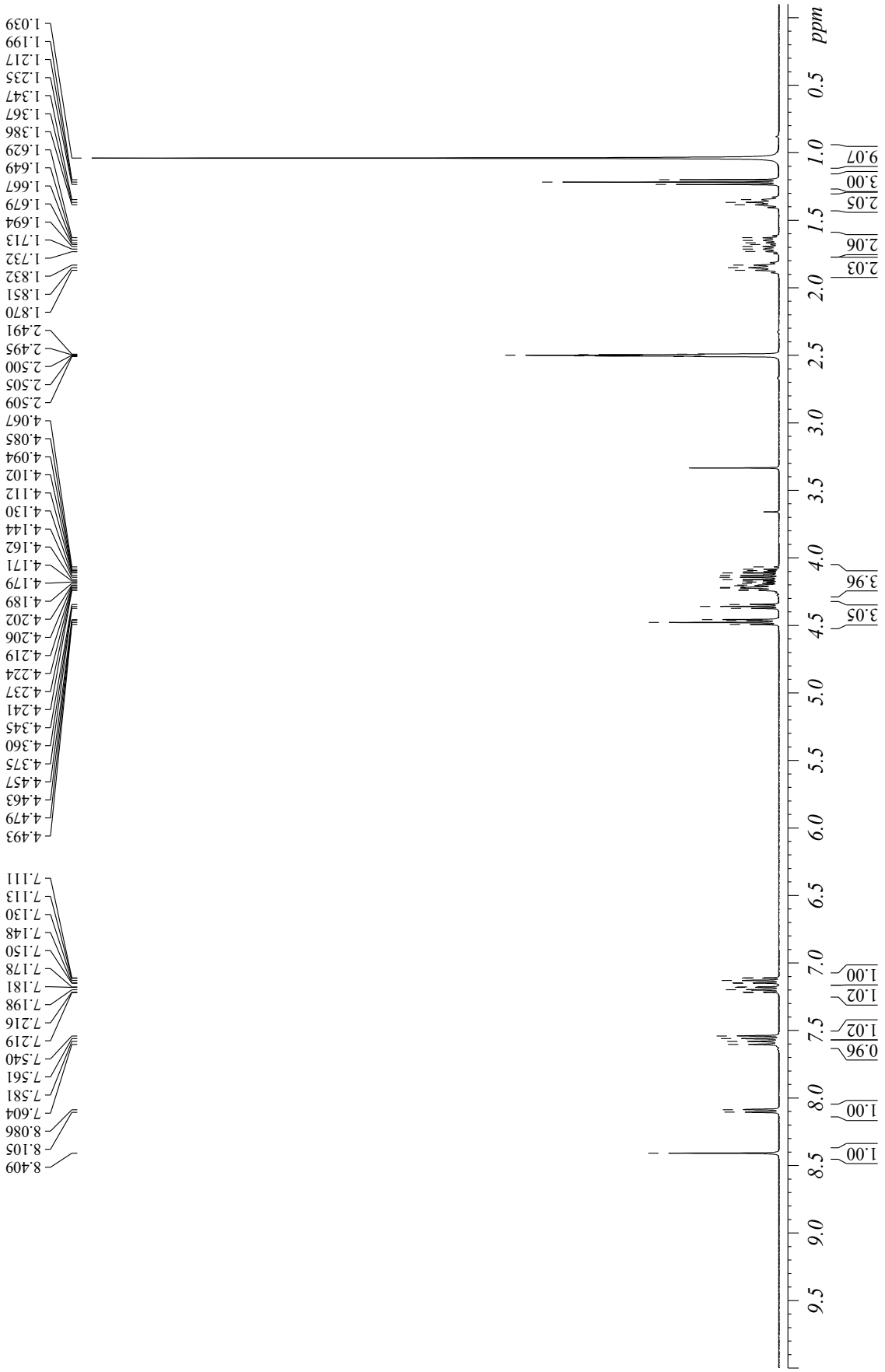
J(C,F) coupling constants [*Hz*]



Characterictic heteronuclear long-range couplings detected by HMBC H → C

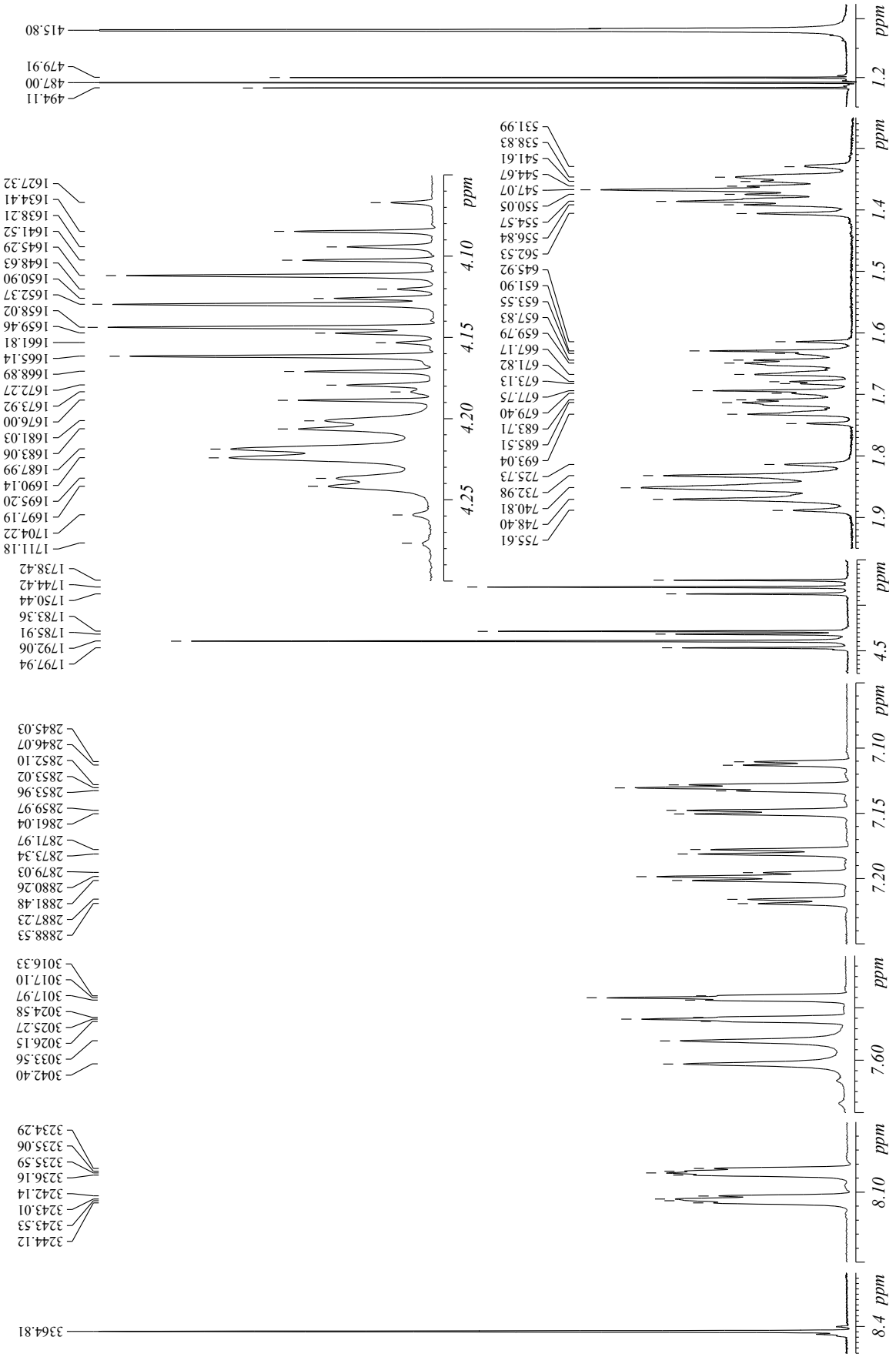


^{1}H-NMR spectrum (overview)



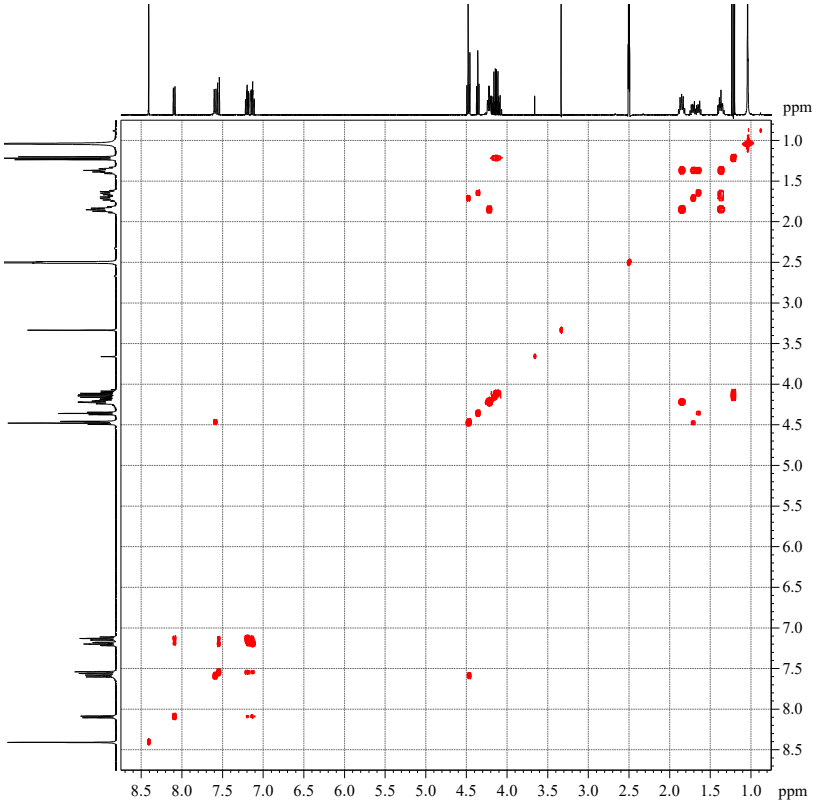
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

^{1}H-NMR spectrum (characteristic sections)

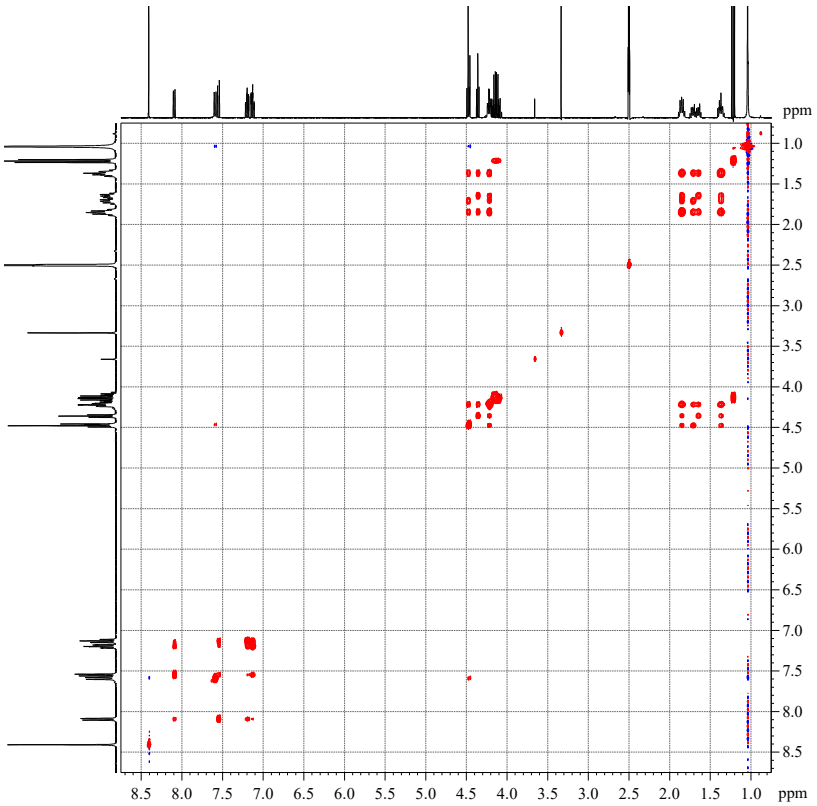


Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

zqs-clip-COSY spectrum

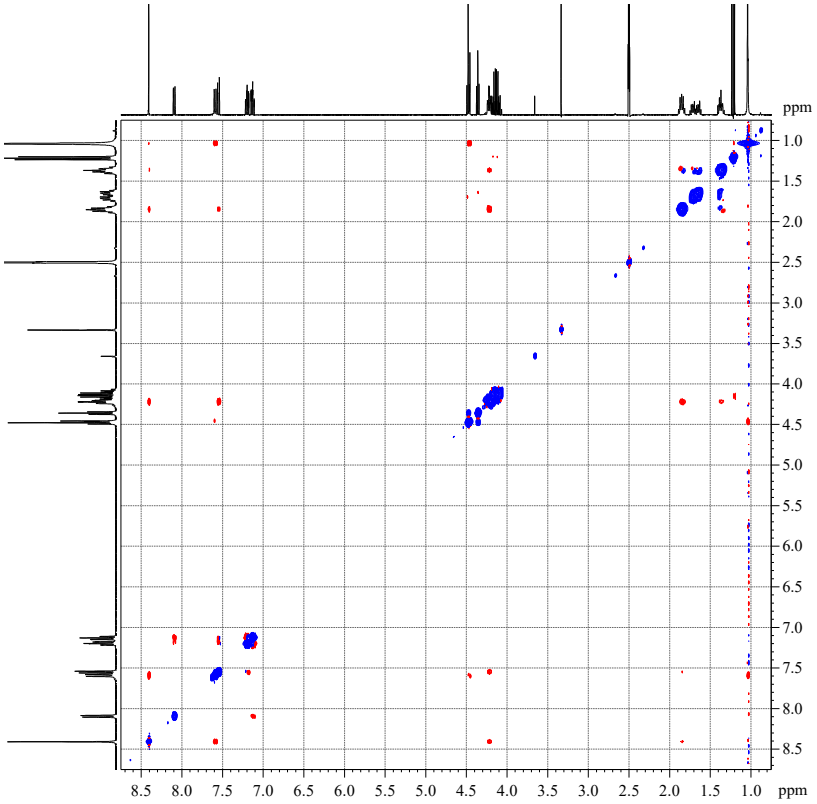


zqs-TOCSY spectrum



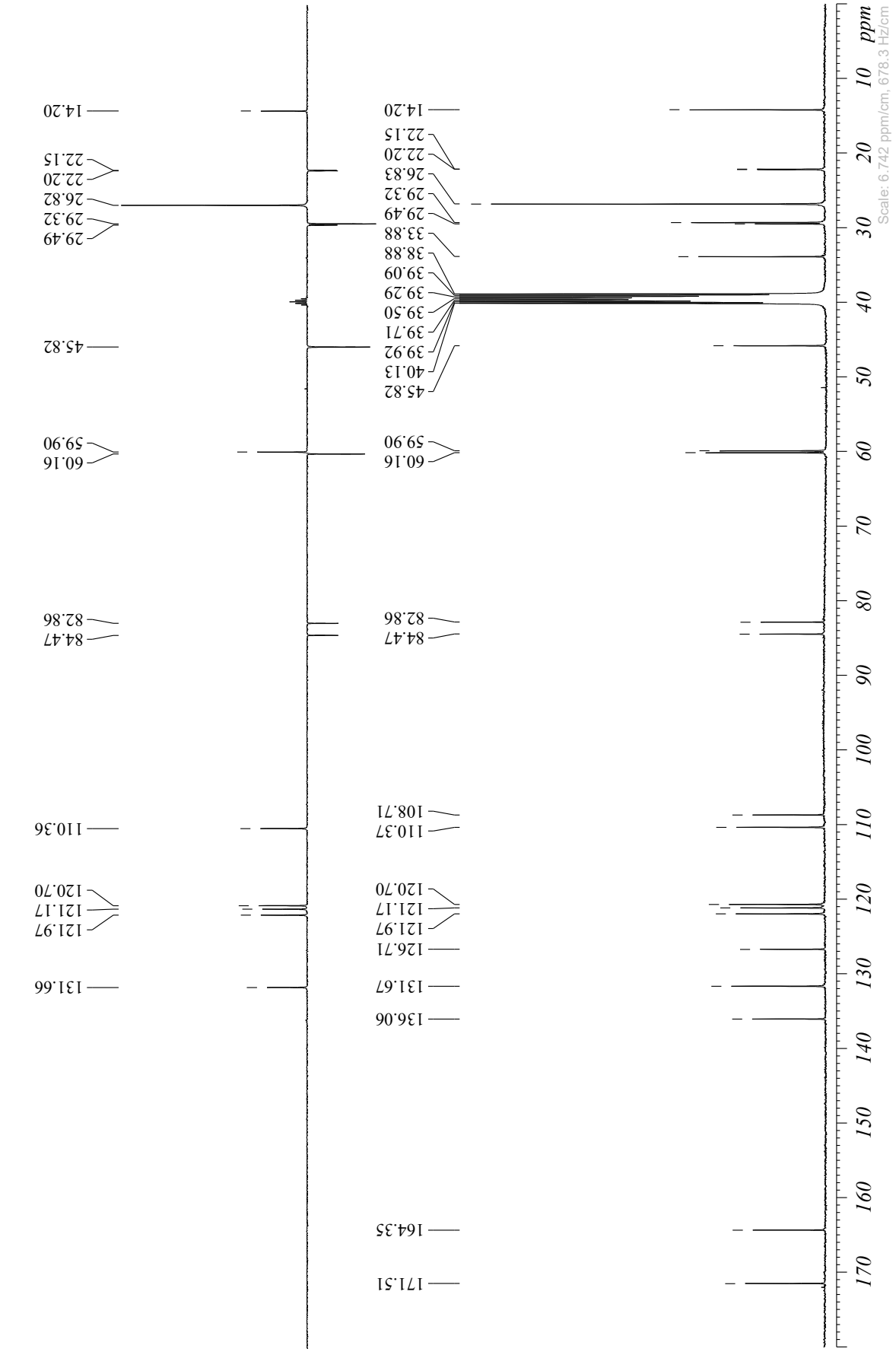
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

zqs-easy-ROESY spectrum



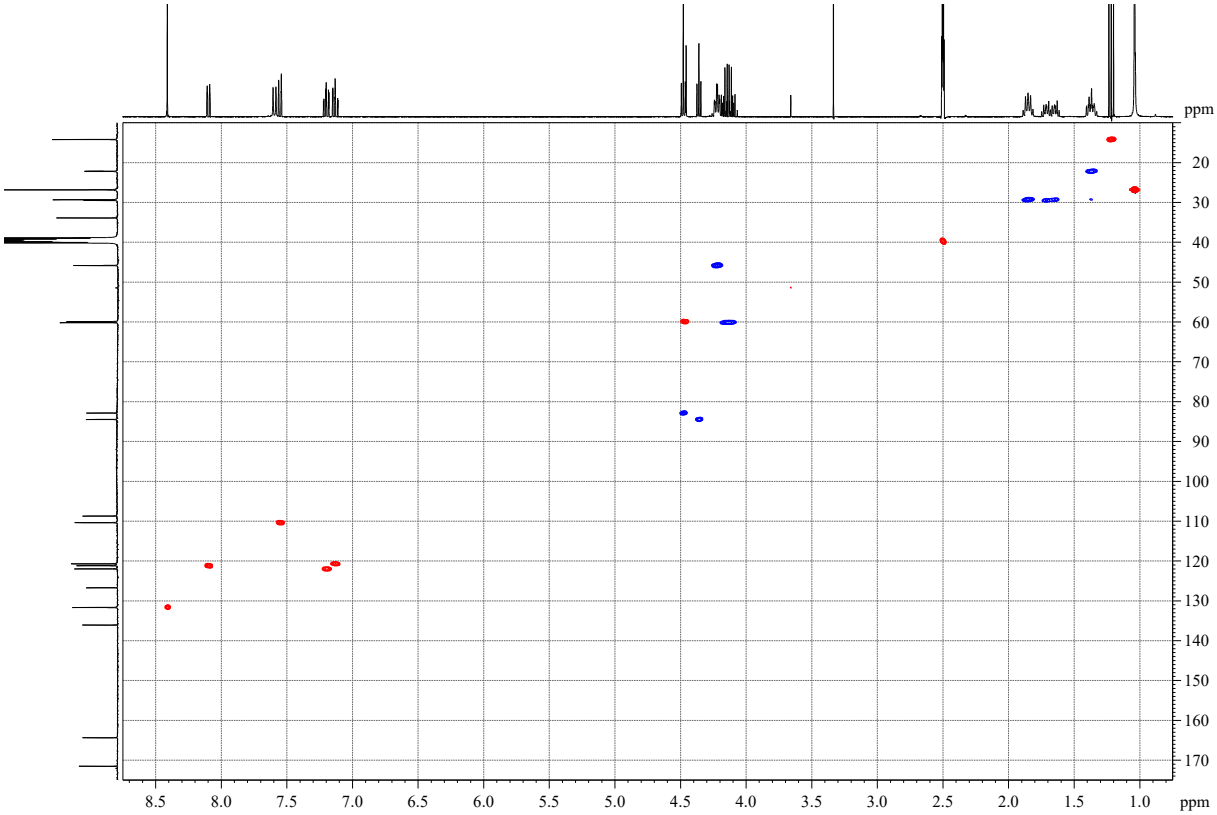
Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

^{13}C-NMR and DEPT-135 spectra

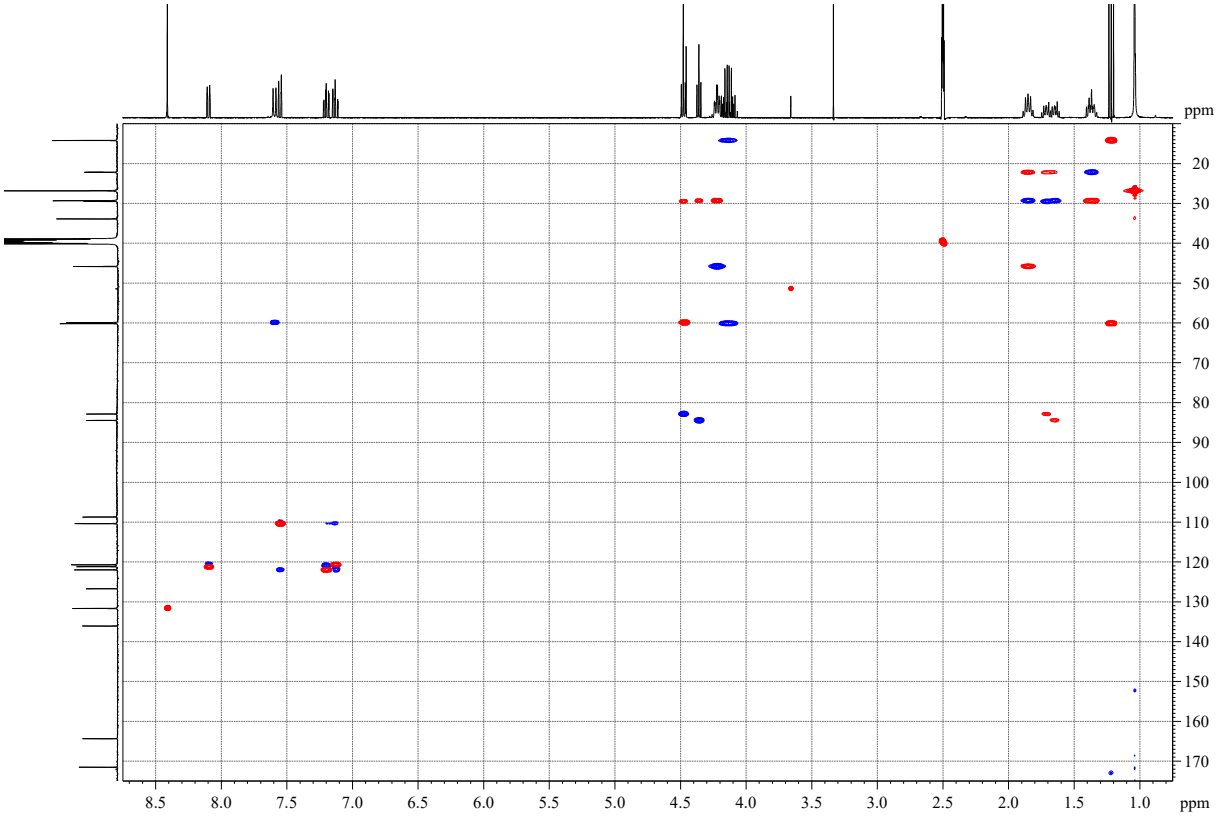


Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

multiplicity edited HSQC spectrum

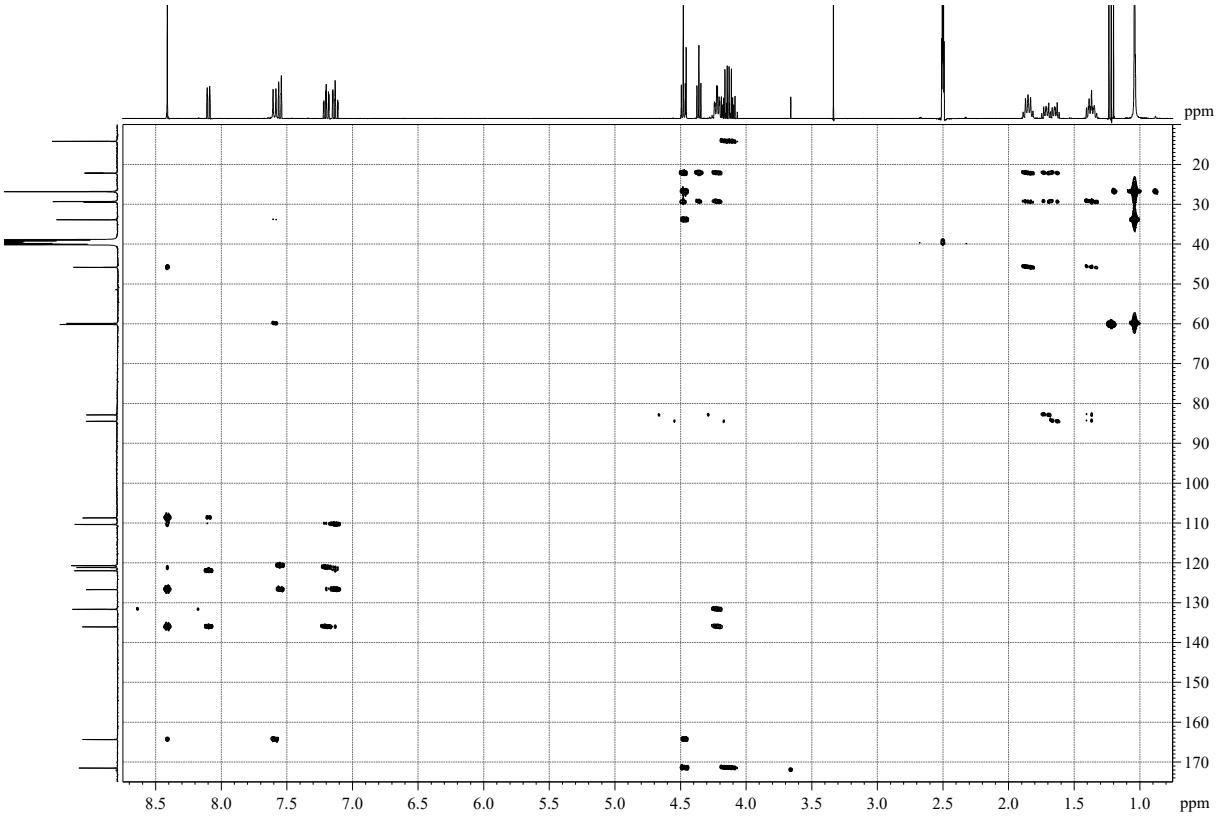


double edited ed-HSQC-zqs-clip-COSY spectrum



Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

magnitude mode HMBC spectrum



Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-d_6

Reference: https://www.caymanchem.com/product/30725/5-fluoro-edmb-pica