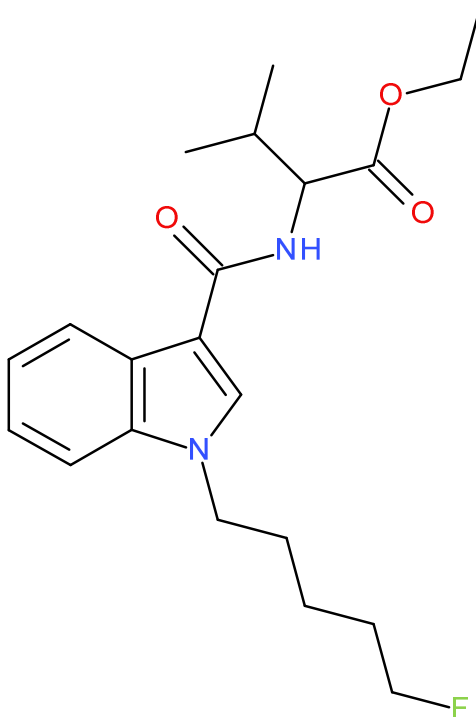


## 5F-EMB-PICA



ethyl 2-[[1-(5-fluoropentyl)indole-3-carbonyl]amino]-3-methyl-butanoate

Formula: C<sub>21</sub>H<sub>29</sub>FN<sub>2</sub>O<sub>3</sub>

Formula weight: 376.46

Chemical Abstracts No.: *n. a.*

Smiles code: CCOC(=O)C(NC(=O)c1cn(CCCCCF)c2ccccc12)C(C)C

InChi key: WPRKGJGOLPGBMG-UHFFFAOYSA-N

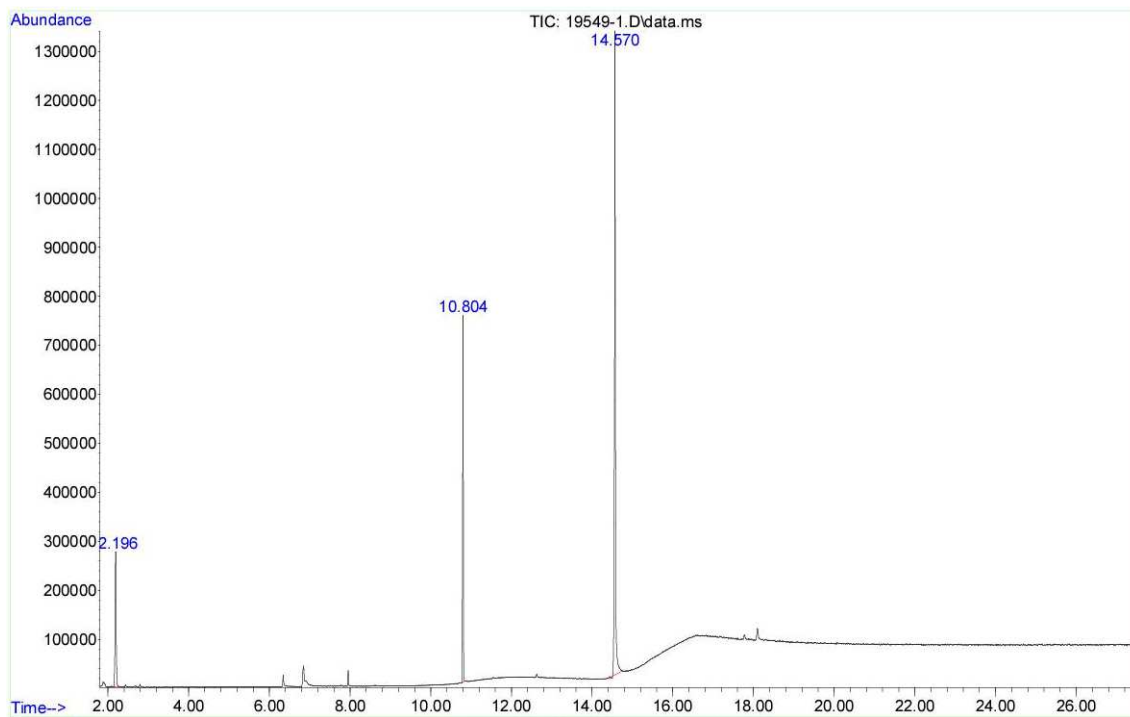
Other names:

The evidence was 95.29 grams orange colored solid material

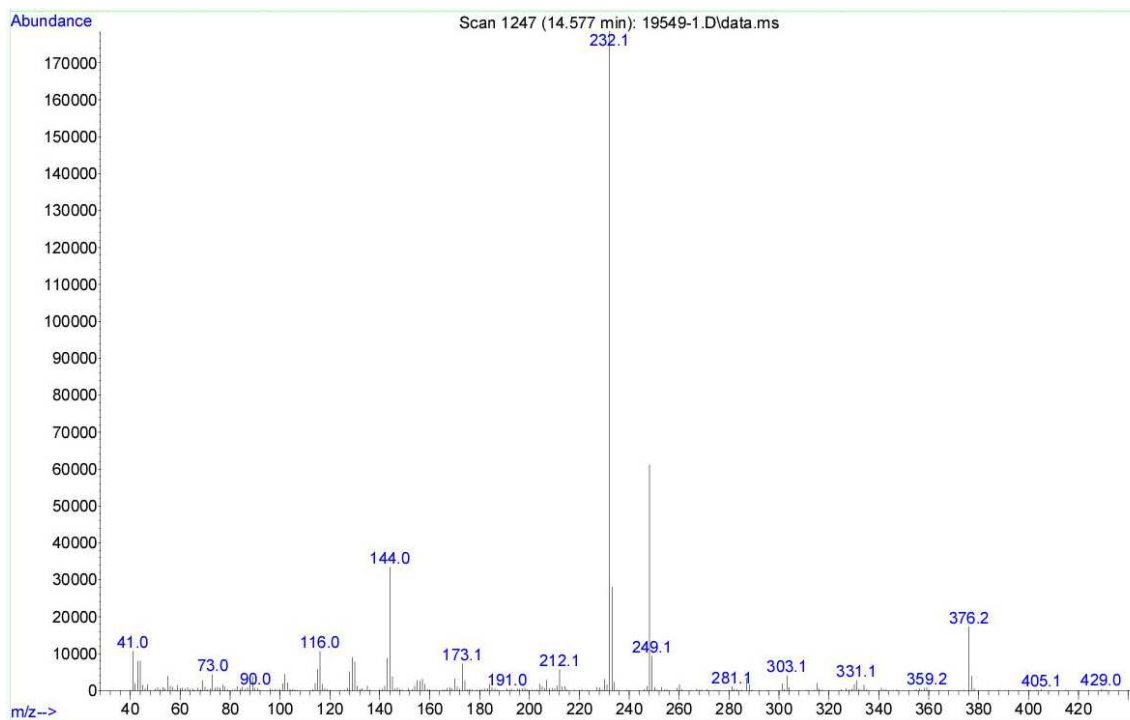
### GC-MS

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. One piece of blotter was extracted by 1 cm<sup>3</sup> methanol, the solution was concentrated and then 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 14.57 min retention time

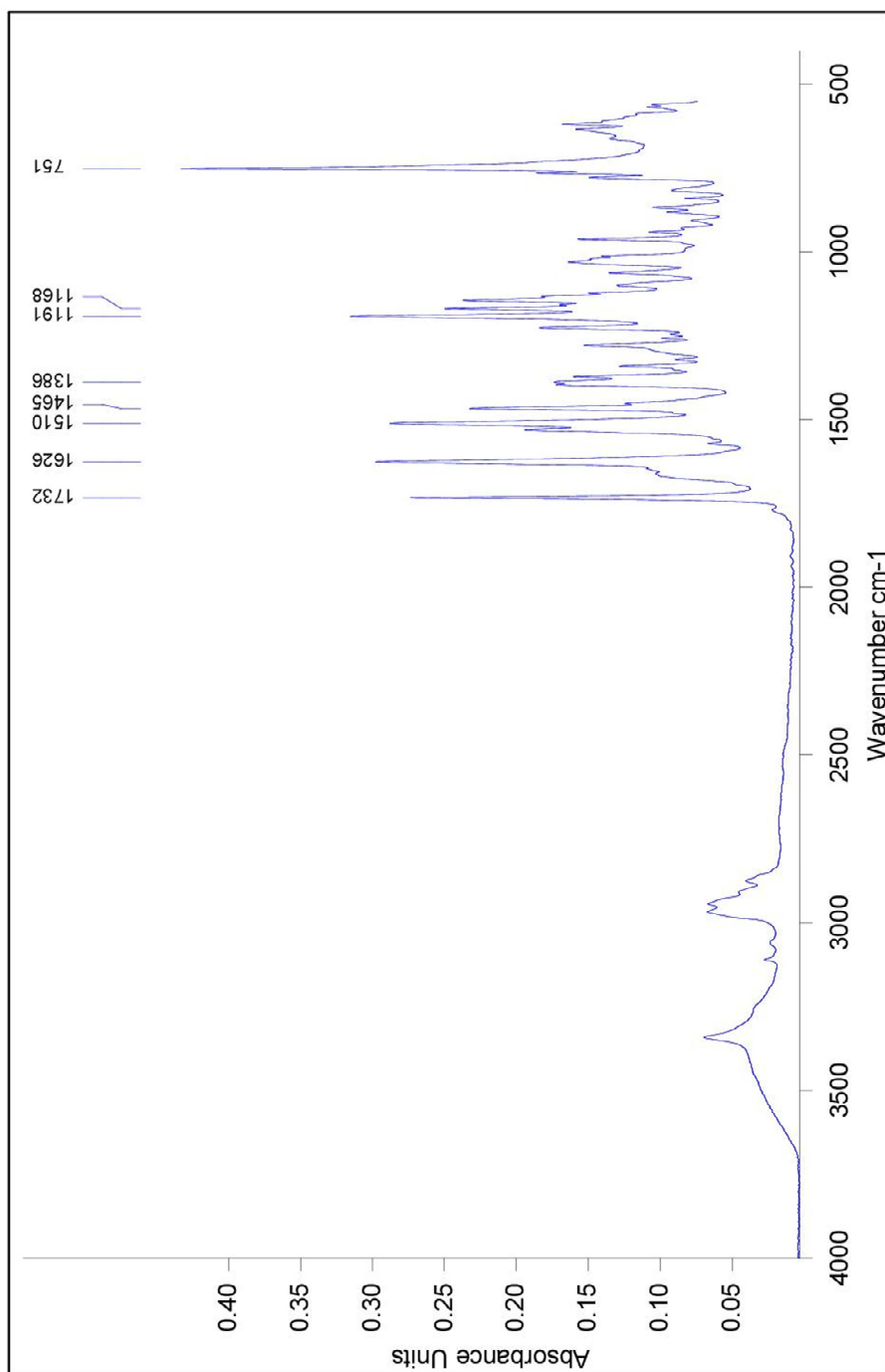


Agilent 6890N Network GC system set up with Agilent HP-5MS

## IR

The IR spectrum was recorded on a Thermo SCIENTIFIC Nicolet iS5 FT-IR spectrometer equipped with an iD5 ATR accessory, in absorbance mode. The digital resolution is  $4\text{ cm}^{-1}$ . The spectrometer was controlled, and the data were processed using Omnic 9 software package. The spectrum was off-line imported into Bruker OPUS software, and the output below was performed by OPUS 7.5 software.

### IR spectrum of the evidence



Thermo SCIENTIFIC Nicolet iS5 FT-IR spectrometer

## 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 °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. 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

### Chemstation conditions

Method name: KABSEQ2\_5S.M

Run time: 27.40 minutes

Split mode: SPLIT

Split ratio: 5 : 1

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

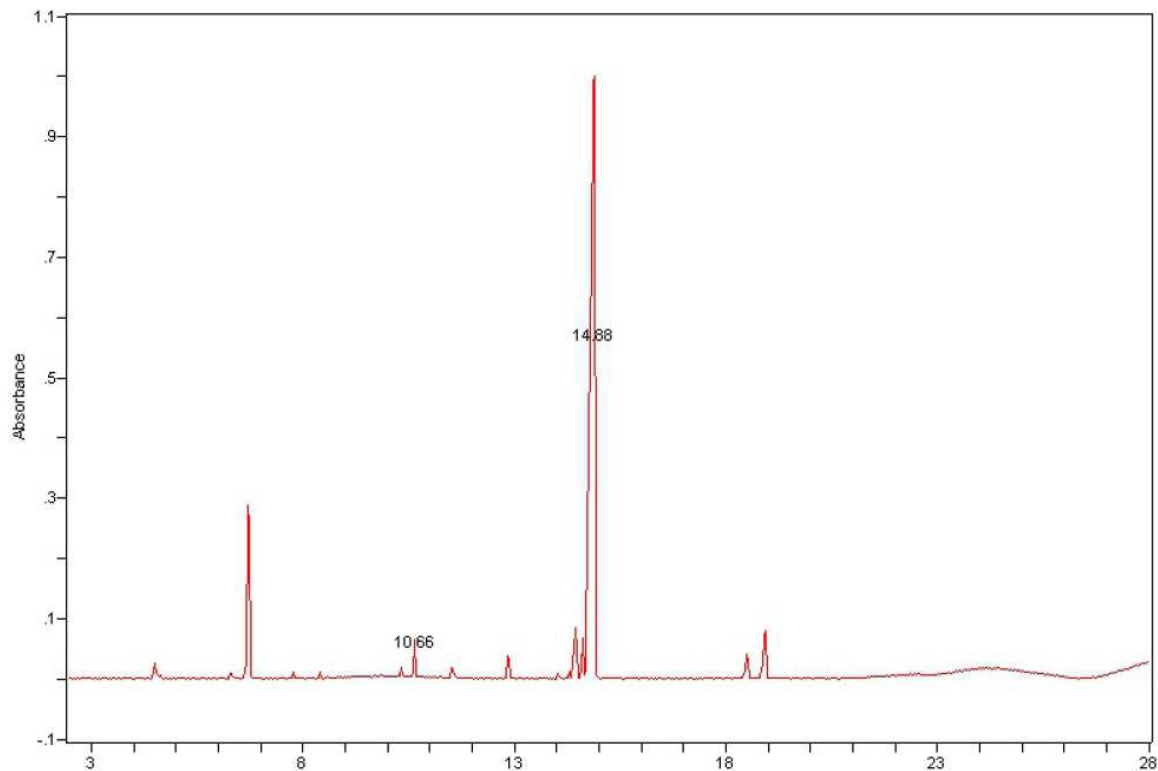
Injection port temperature (°C): 300

Injection volume (µL): 1

Gas pressure (psi): 24.10

### Chromatogram

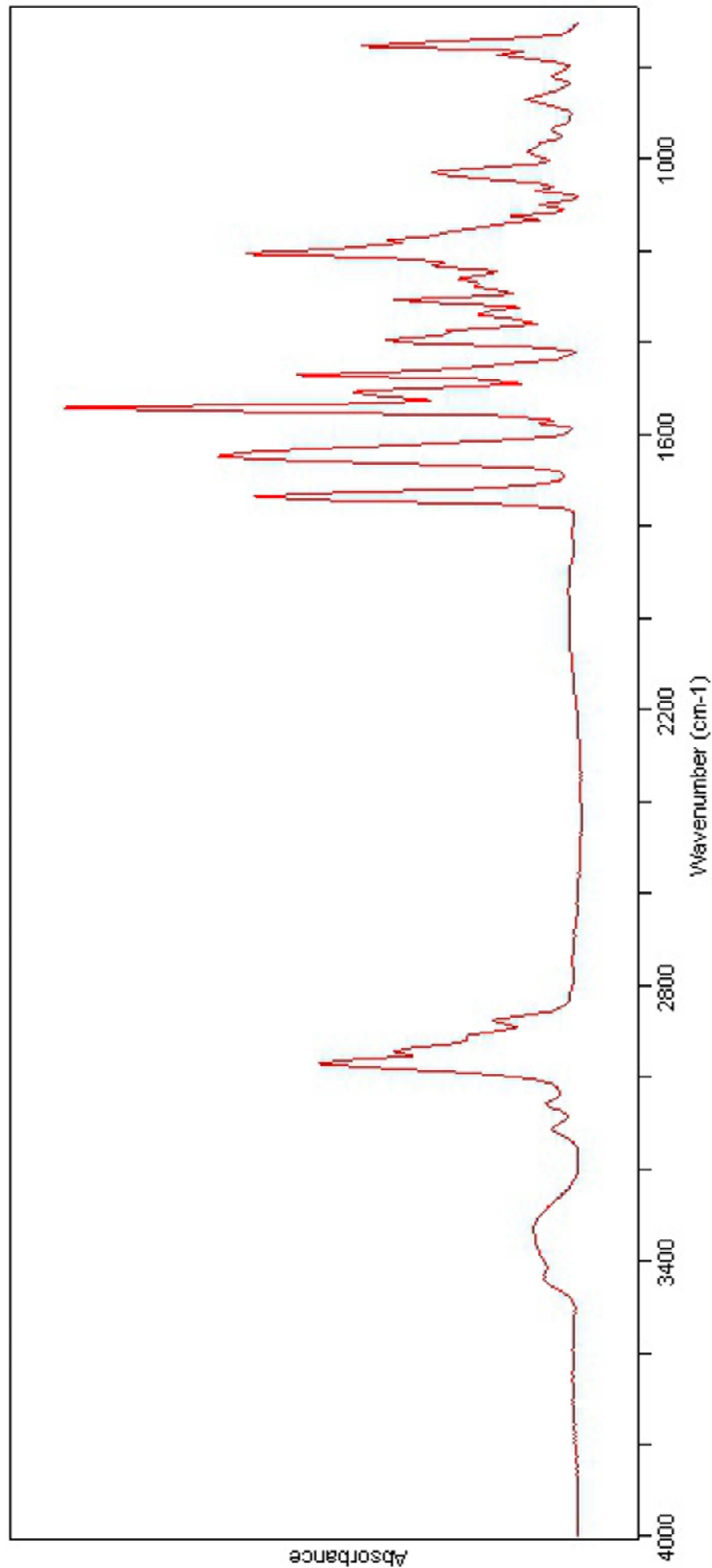
Data file: C:/Data/200715/20\_19549-1/reproc/20\_19549-1.Multifile.cgm



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

### IR spectrum of the reported component at 14.88 min retention time

**Peak 2**  
Peak name: 20\_19549-1 @ 14.88 minutes  
Chemist: BD  
Date analyzed:



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

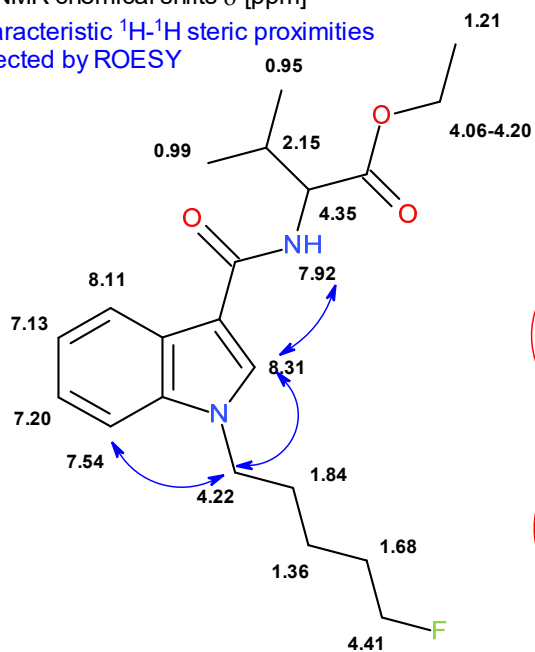
## NMR

The NMR spectra were recorded on a Bruker Avance Neo 400 NMR 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 ( $\delta$ ) are given in parts per million unit, referenced to tetramethylsilane ( $\delta_{\text{TMS}} = 0.00$  ppm). The determination of the structure was based on  $^1\text{H}$ , zqs-clip-COSY, zqs-TOCSY, zqs-easy-ROESY, DOSY difference, as well as  $^{13}\text{C}$ , multiplicity edited HSQC and HMBC spectra.

## Interpretation of the NMR spectra

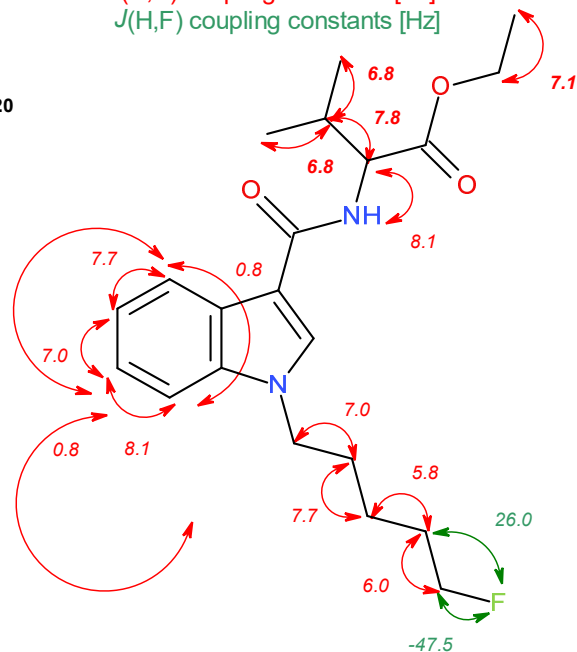
$^1\text{H-NMR}$  chemical shifts  $\delta$  [ppm]

Characteristic  $^1\text{H-}^1\text{H}$  steric proximities detected by ROESY



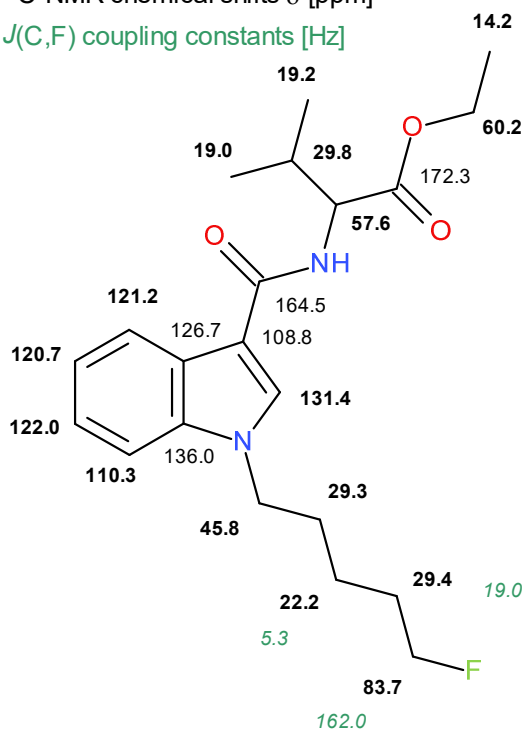
$J(\text{H,H})$  coupling constants [Hz]

$J(\text{H,F})$  coupling constants [Hz]



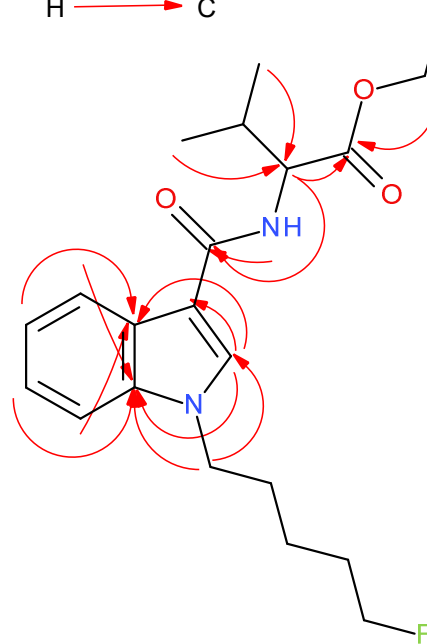
$^{13}\text{C-NMR}$  chemical shifts  $\delta$  [ppm]

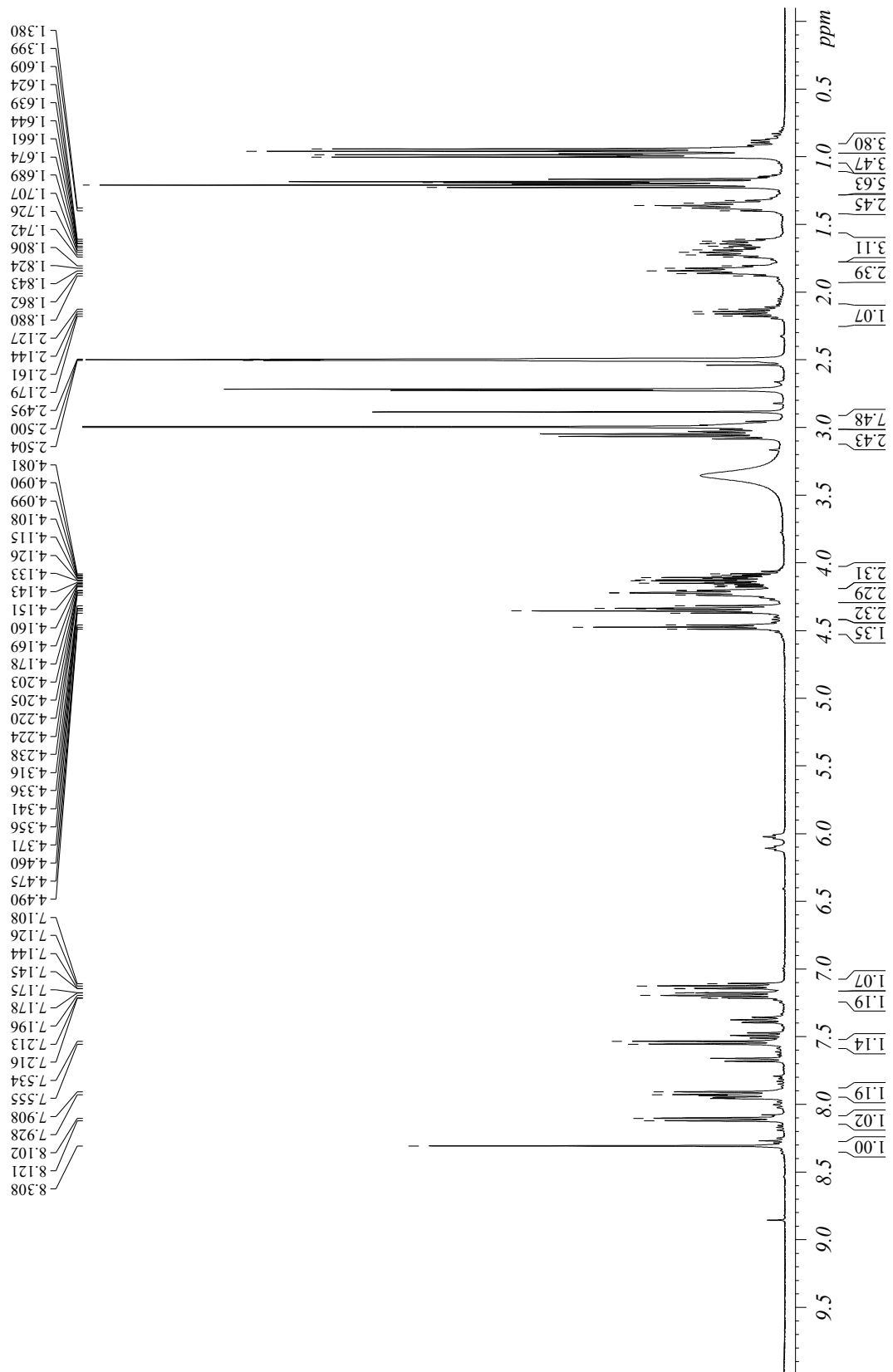
$J(\text{C,F})$  coupling constants [Hz]



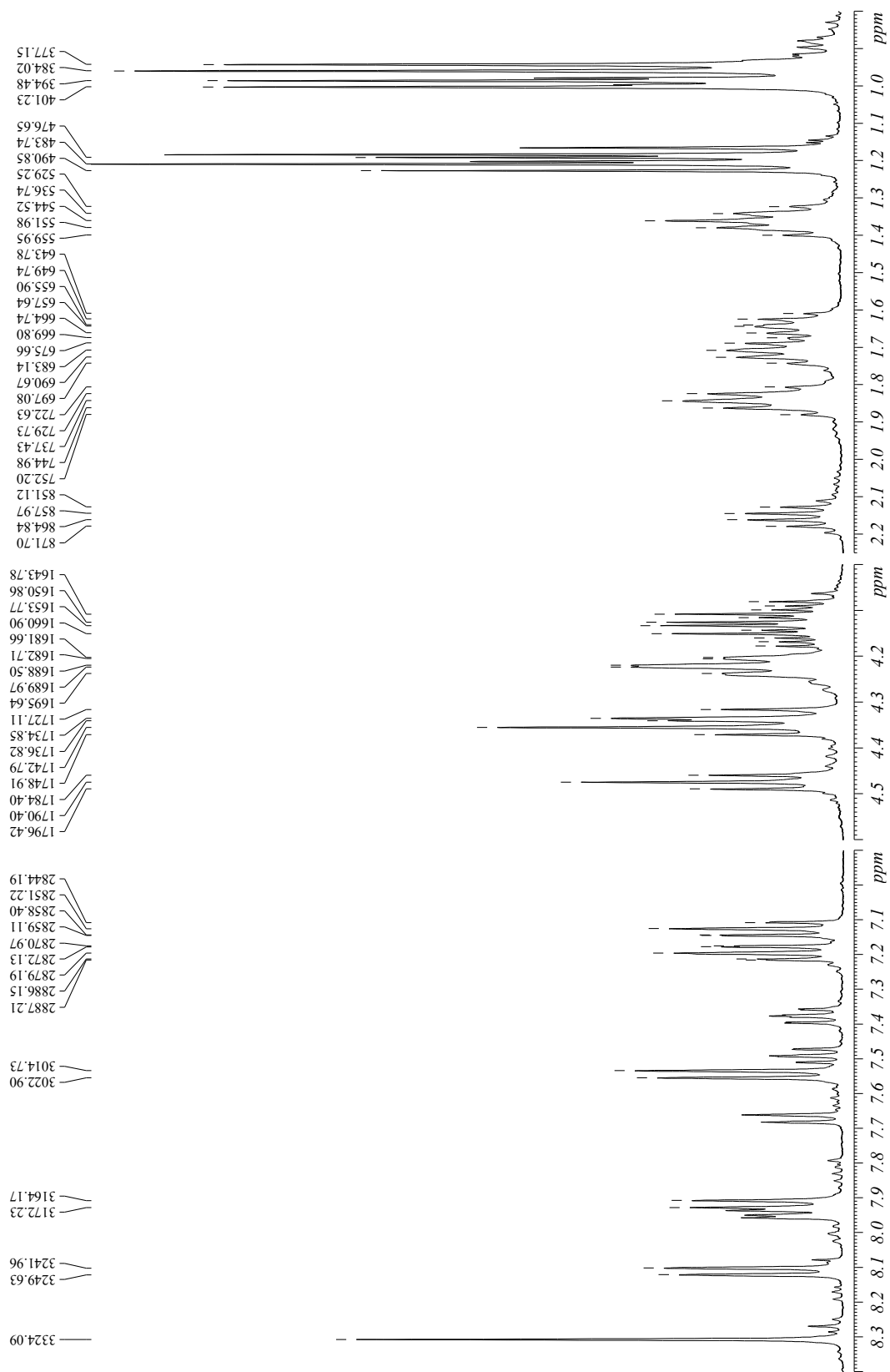
Characteristic heteronuclear long-range couplings detected by HMBC experiment

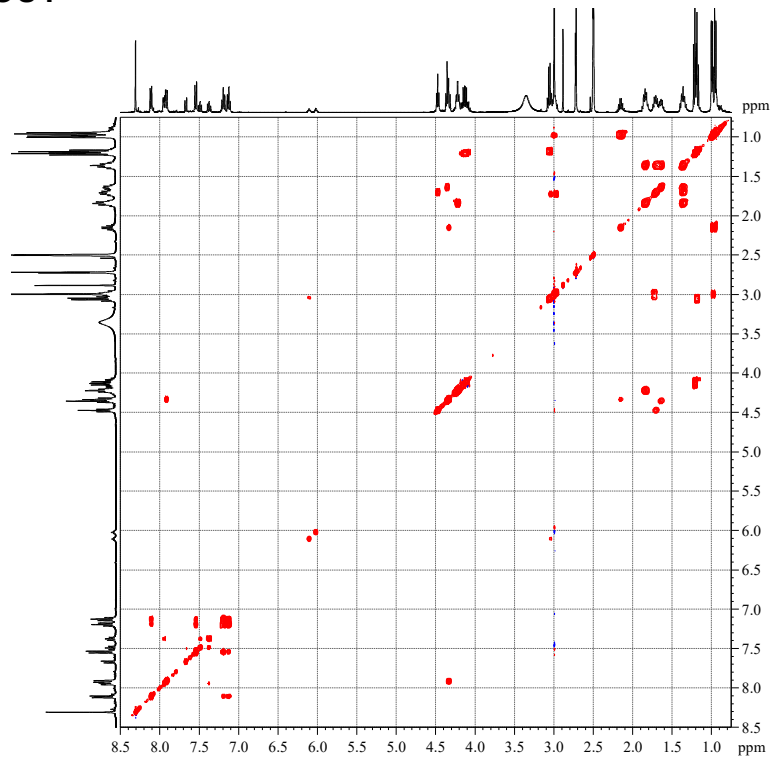
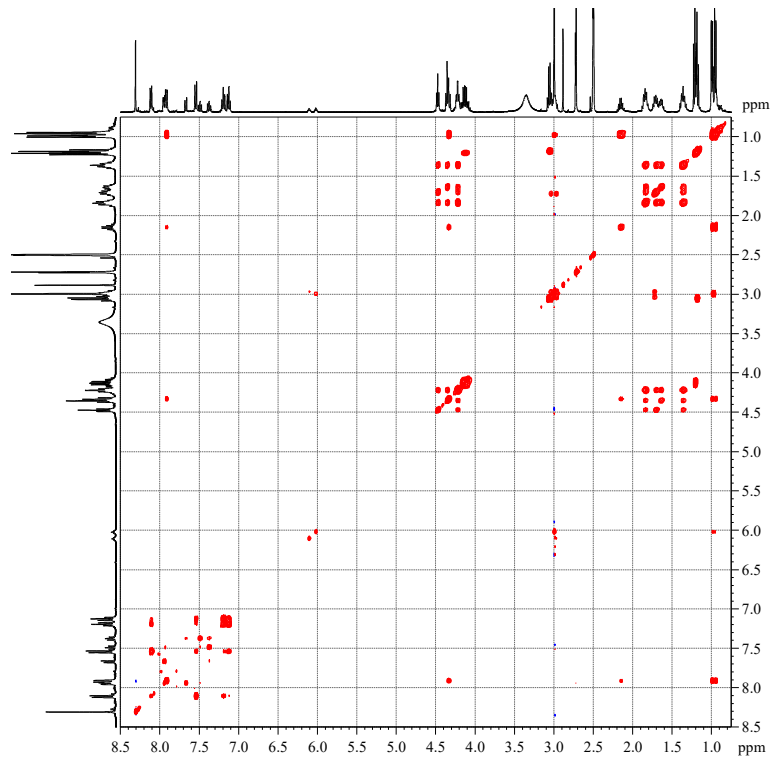
H  $\rightarrow$  C

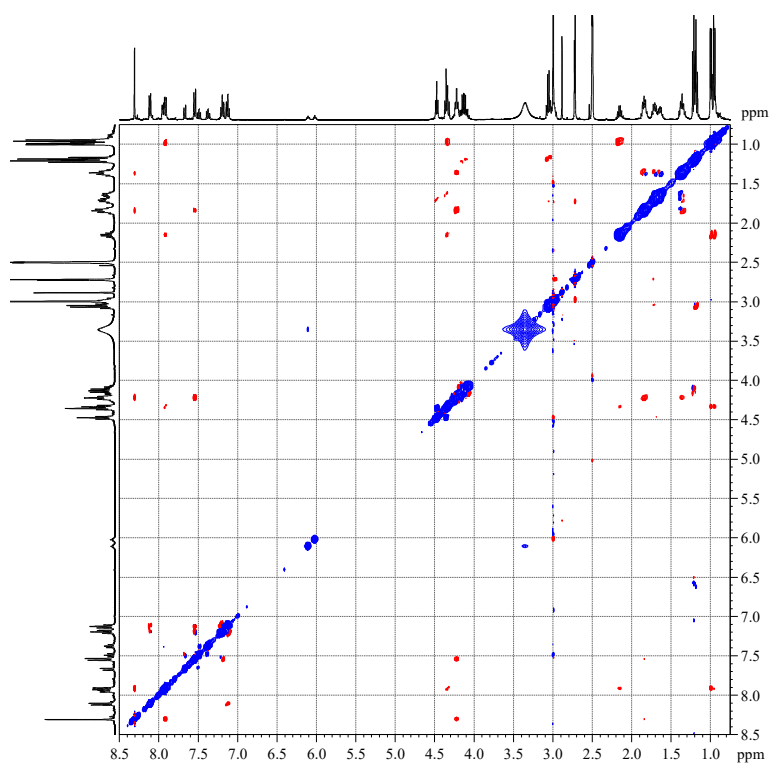
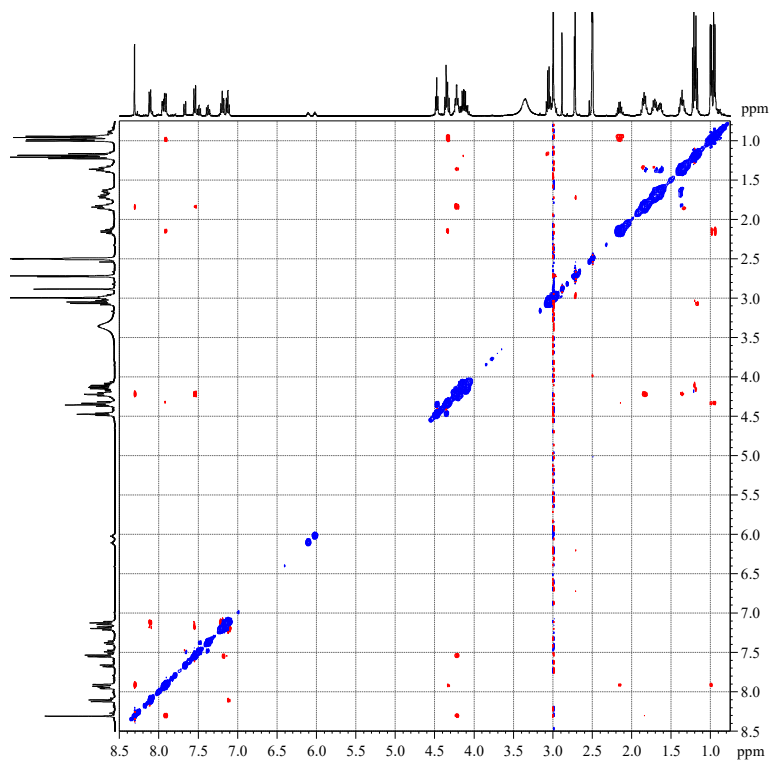


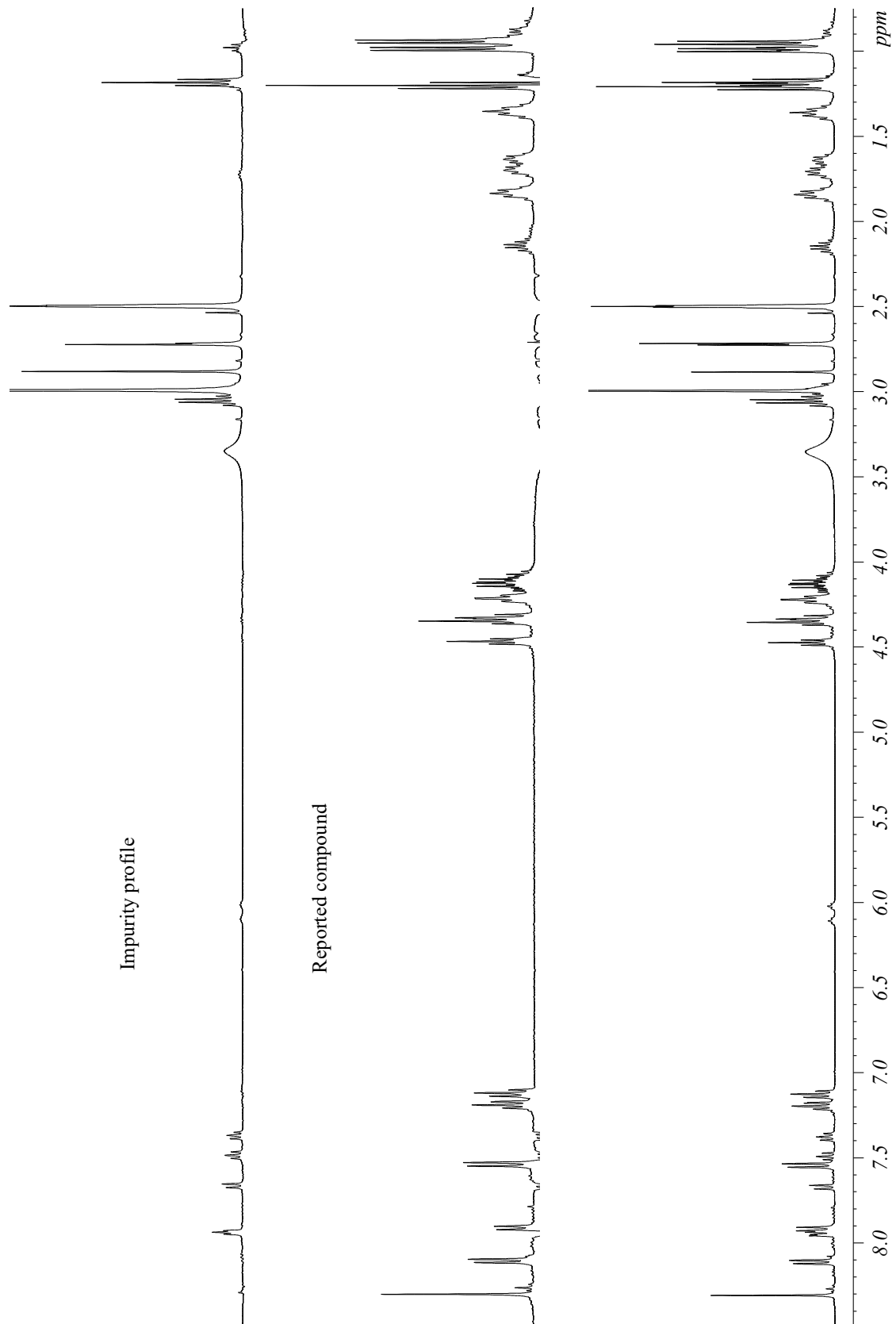
**$^1\text{H-NMR}$  spectrum (overview)**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent:  $\text{DMSO-}d_6$

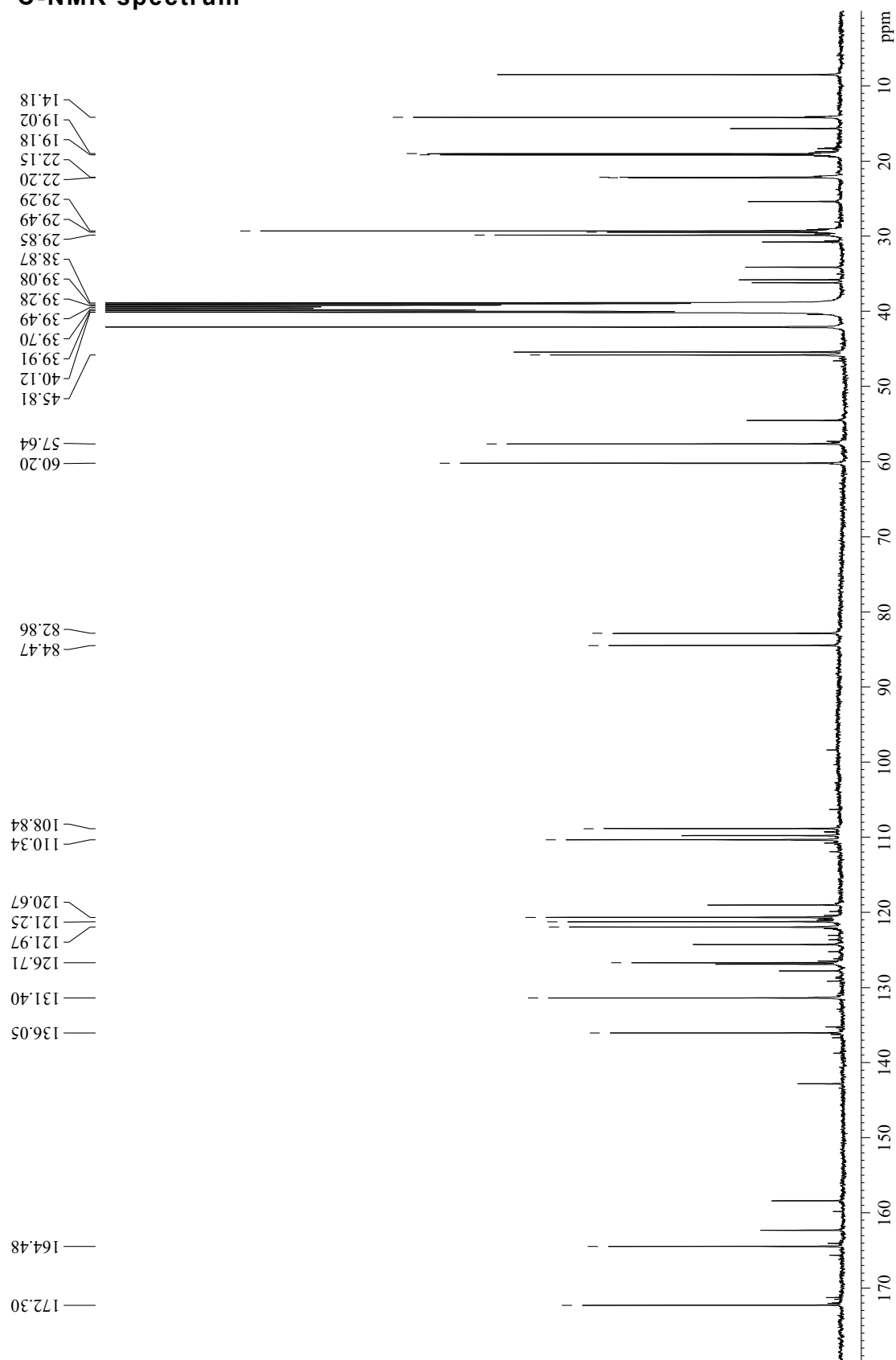


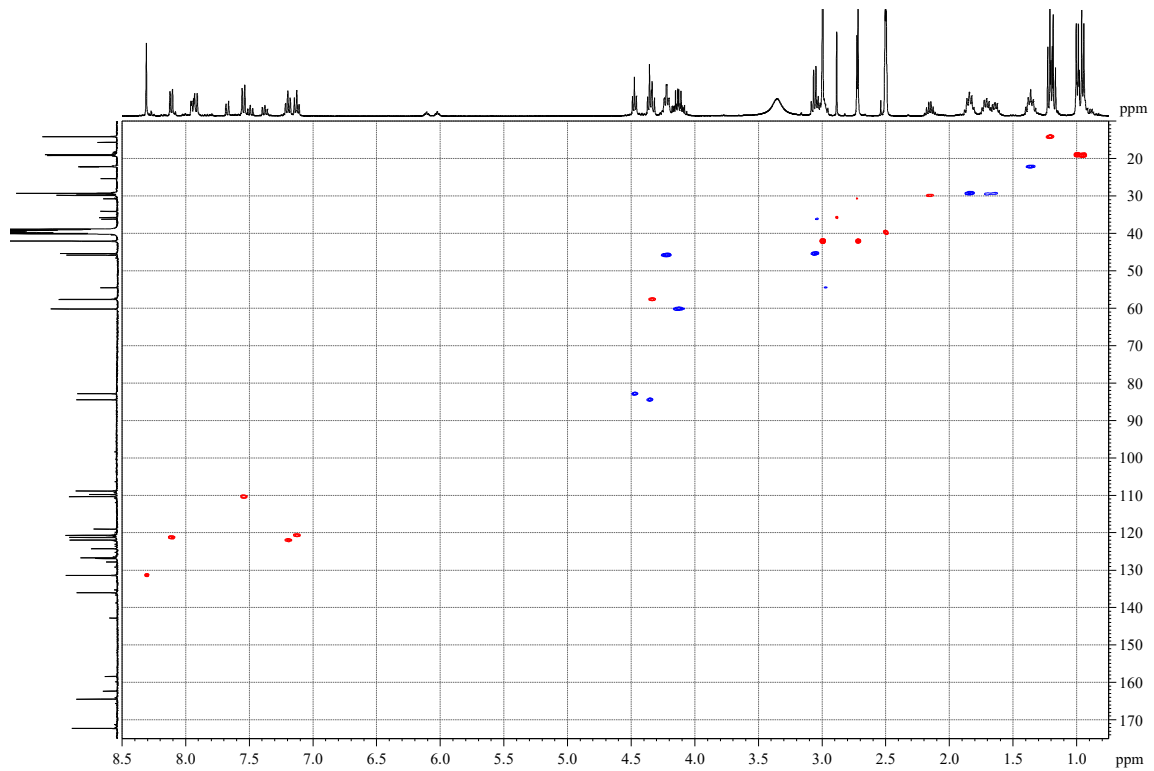
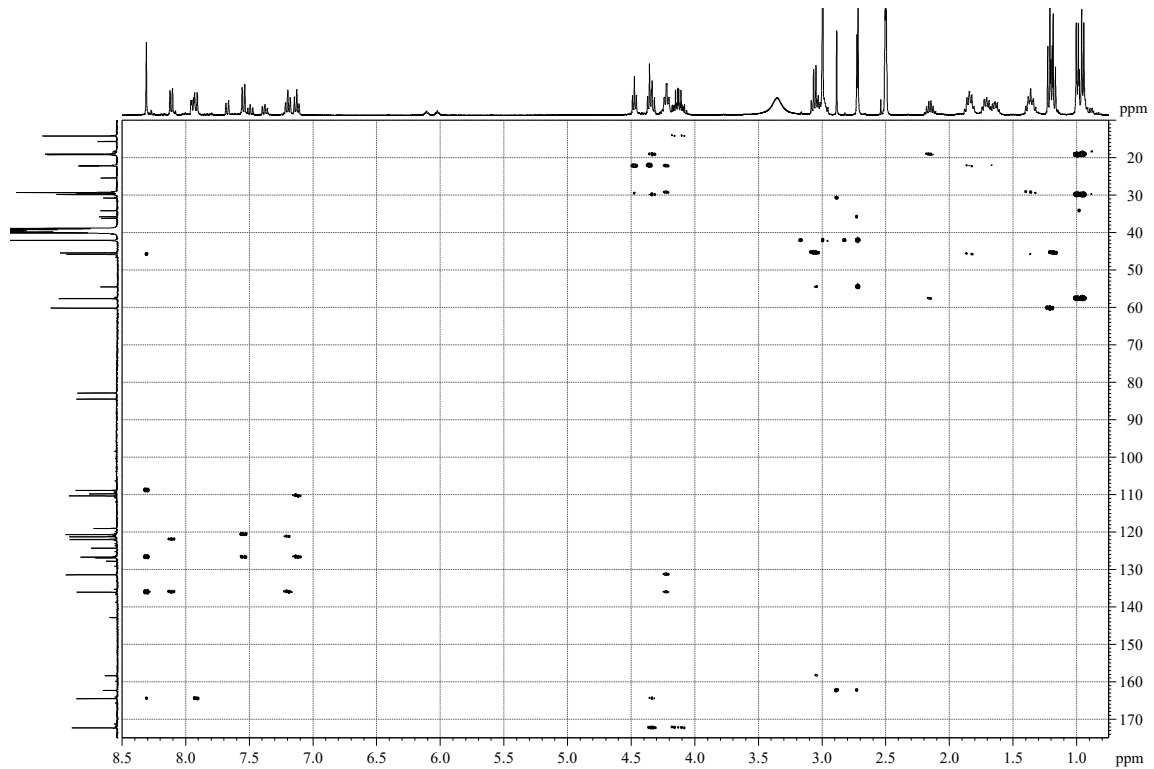
**$^1\text{H-NMR}$  spectrum (characteristic sections)**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent:  $\text{DMSO-}d_6$

**zqs-clip-COSY****zqs-TOCSY**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-*d*<sub>6</sub>

**zqs-NOESY****zqs-easy-ROESY**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-*d*<sub>6</sub>

**DOSY difference spectrum**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO- $d_6$

**$^{13}\text{C}$ -NMR spectrum**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent:  $\text{DMSO-}d_6$

**ed-HSQC****HMBC**Bruker AVANCE NEO 400, CryoProbe Prodigy; solvent: DMSO-*d*<sub>6</sub>