

Supplementary Information

Microbispora sp. LGMB259 Endophytic Actinomycete Isolated from *V. divergens* (Pantanal, Brazil)

Producing β -Carbolines and Indoles with Biological Activity

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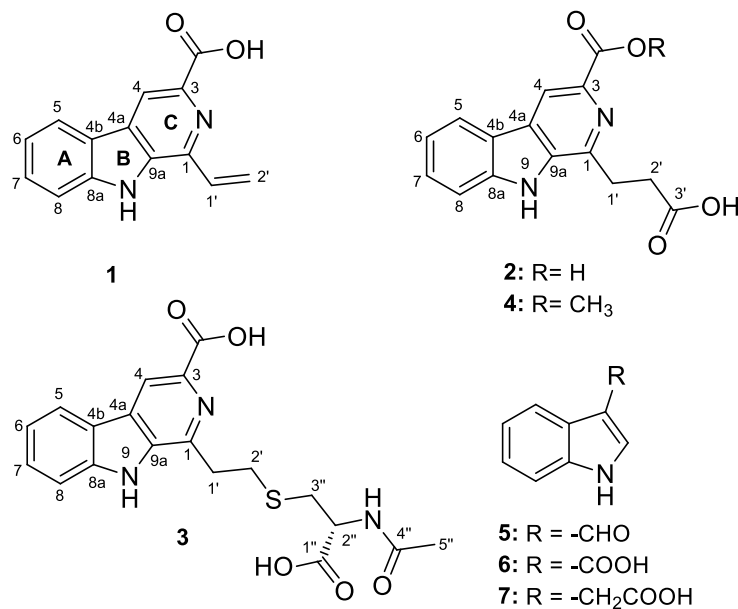


Figure S1: Chemical structure of compounds 1-7

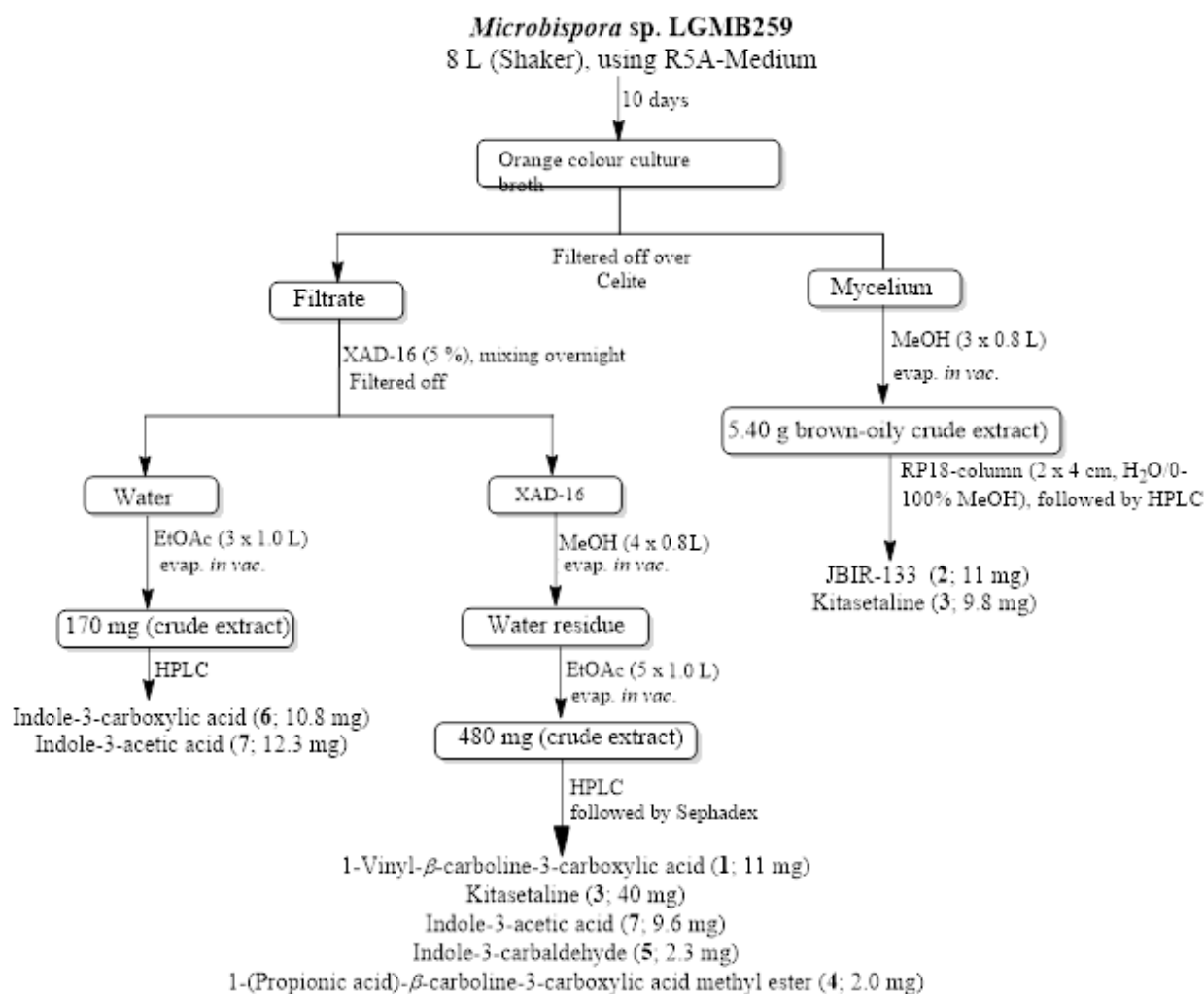


Figure S2: Work-up scheme of *Microbispora* sp. LGMB259 using R5A-medium

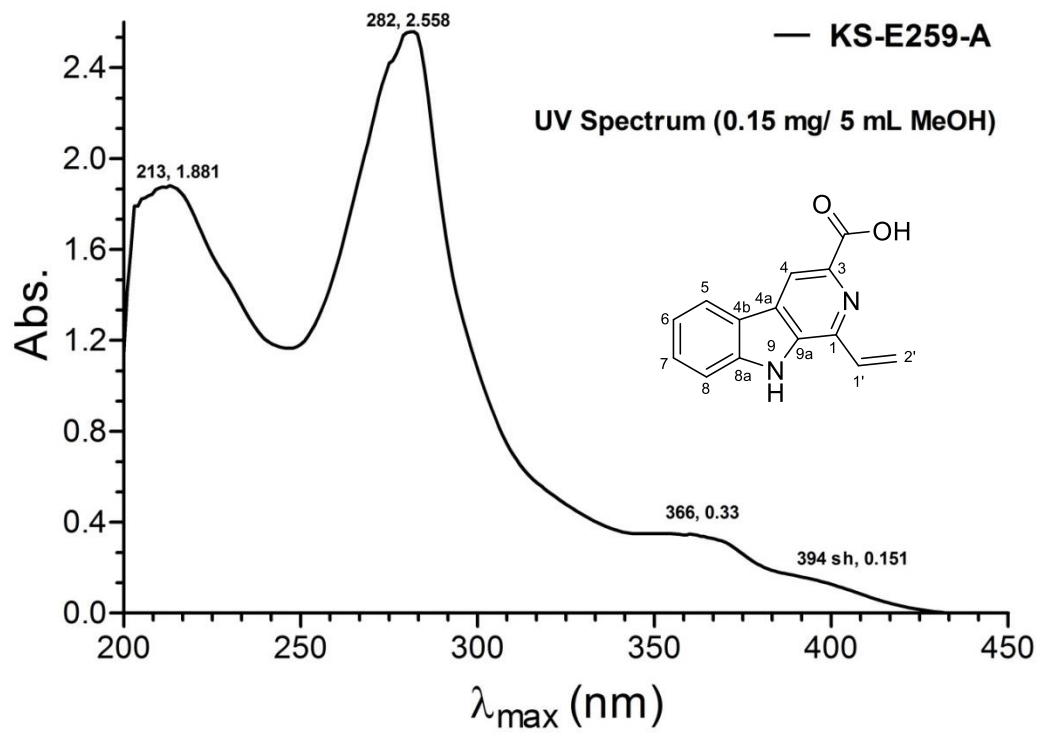


Figure S3: UV (MeOH) spectrum of 1-Vinyl- β -carboline-3-carboxylic acid (**1**)

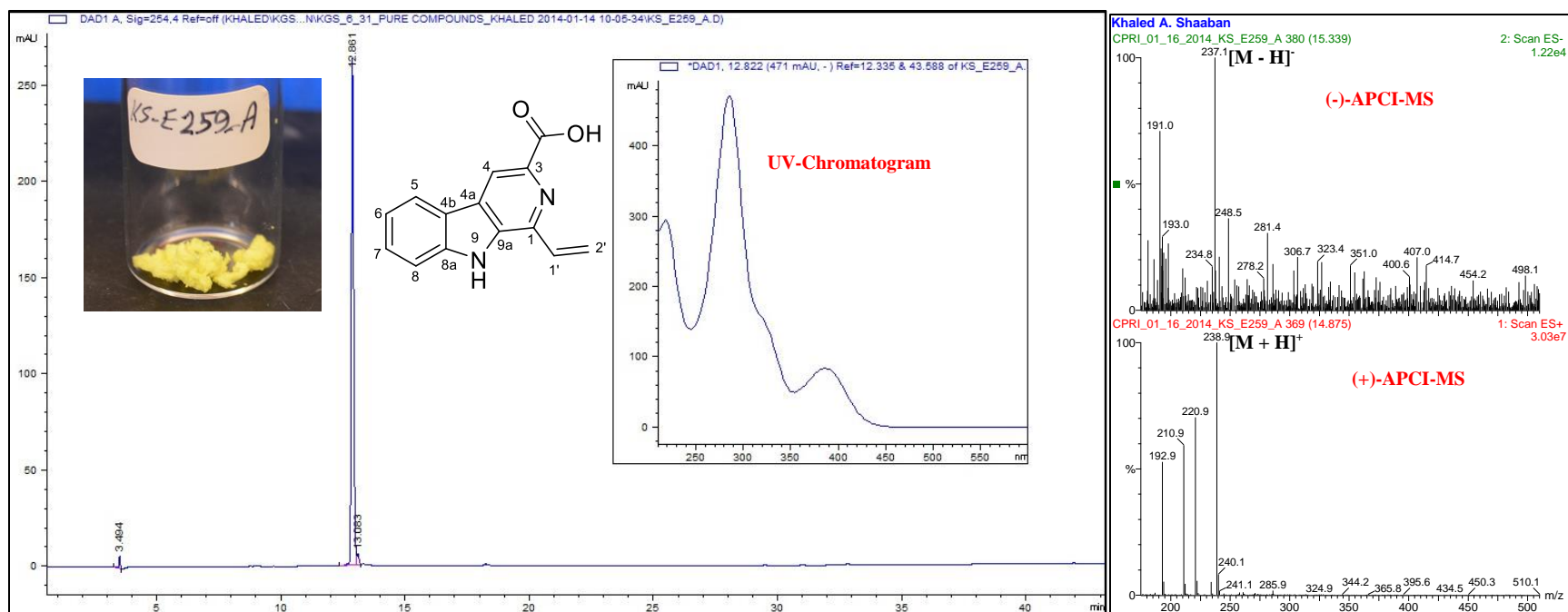


Figure S4: HPLC/UV/APCI-MS analyses of 1-Vinyl- β -carboline-3-carboxylic acid (**1**). HPLC-conditions: Detection wavelength 254 nm; solvent A: H₂O/0.1% TFA; solvent B: acetonitrile; flow rate: 1.0 mL min⁻¹; 0-35 min, 95-0% A (linear gradient); 35-40 min 0% A; 40-41 min 0-95% A (linear gradient); 41-45 min 95% A.

13-0533 #172-193 RT: 2.92-3.26 AV: 22 SB: 8 0.88-1.00 NL: 3.75E5

T: + c Full ms [40.00-750.00]

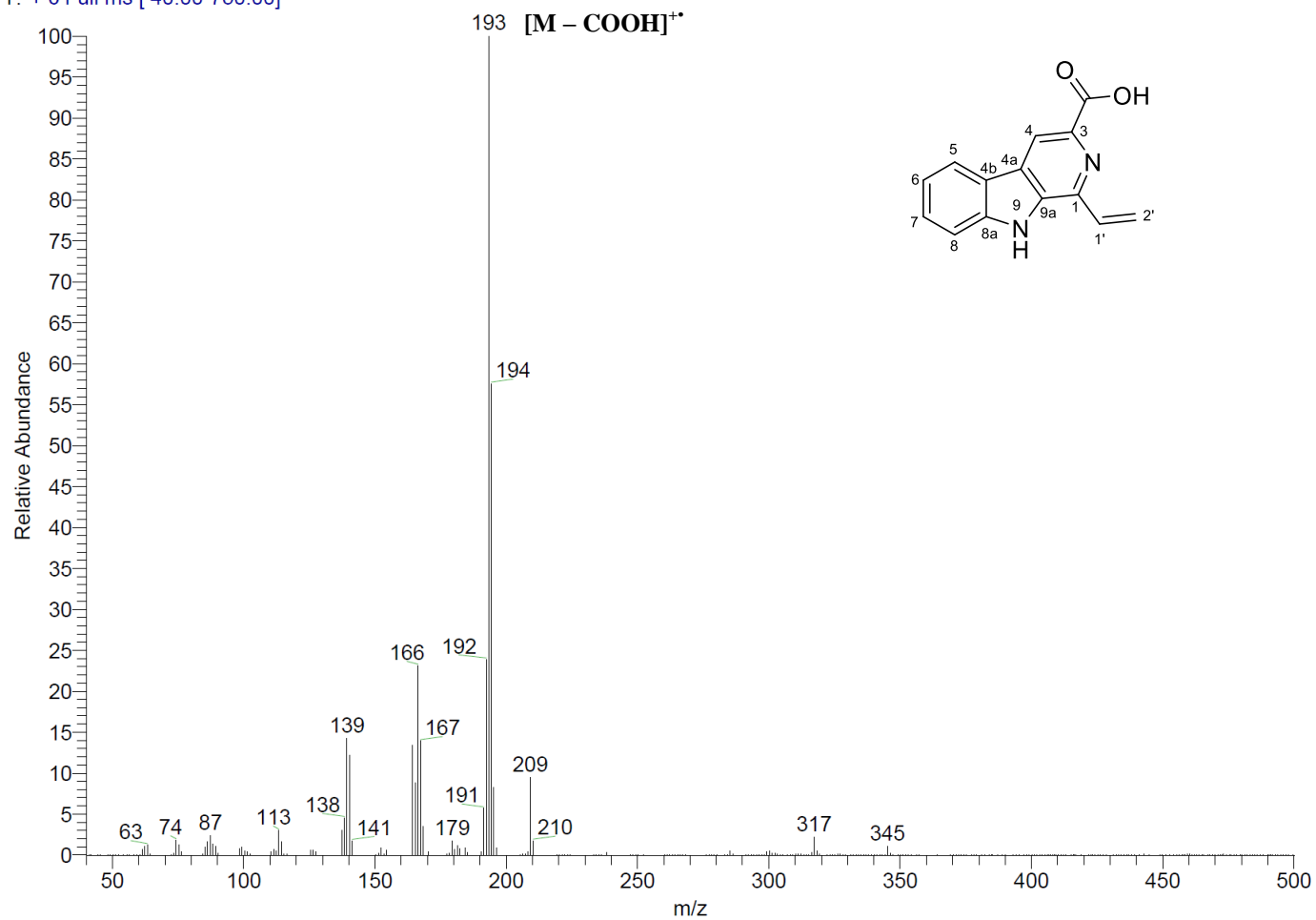
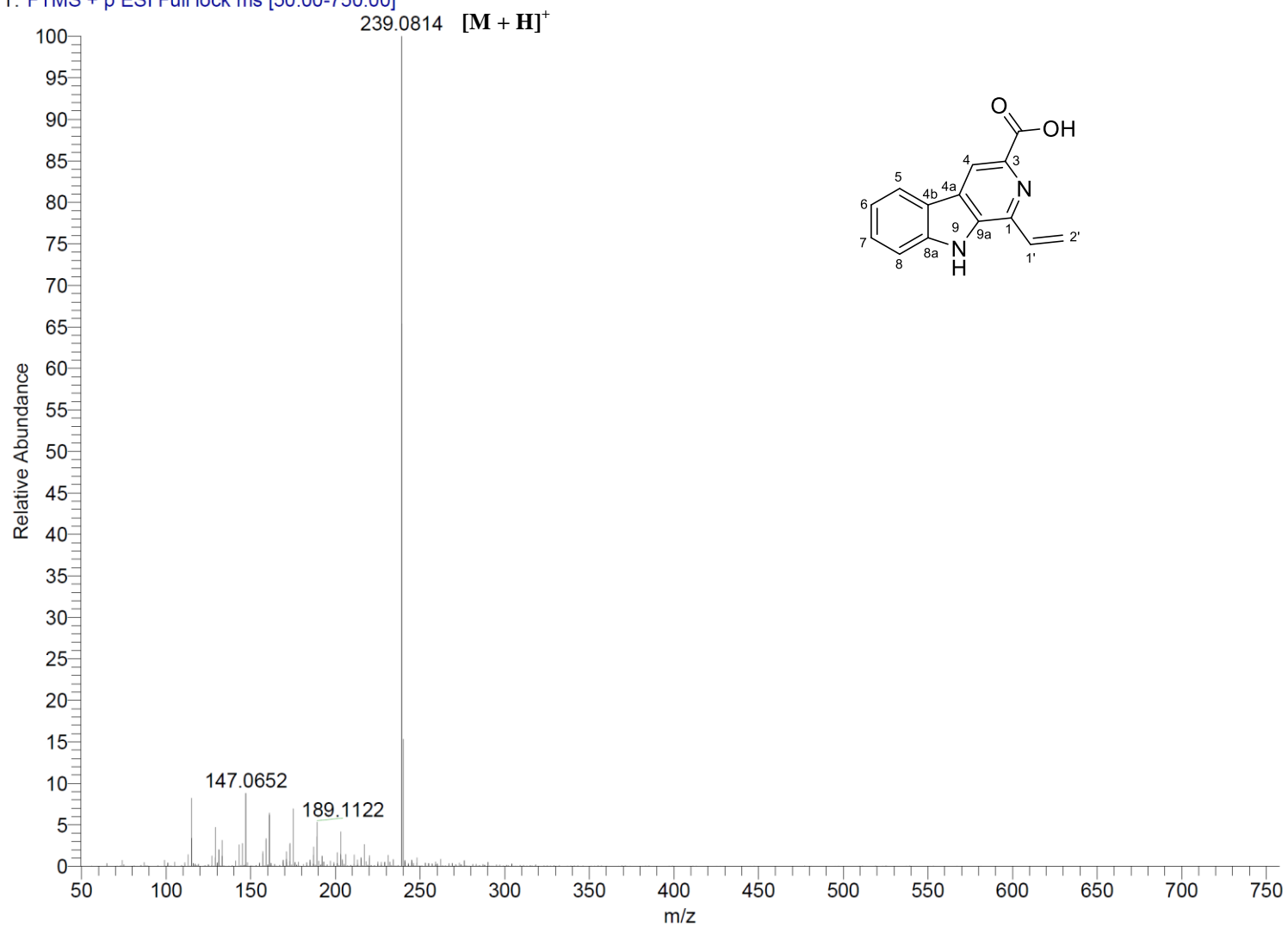


Figure S5: EI-MS spectrum of 1-Vinyl- β -carboline-3-carboxylic acid (1)

13-0533 #30-38 RT: 0.85-1.08 AV: 9 NL: 1.45E9

T: FTMS + p ESI Full lock ms [50.00-750.00]

**Figure S6:** (+)-HRESI-MS spectrum of 1-Vinyl- β -carboline-3-carboxylic acid (**1**)

13-0533 #72-80 RT: 2.06-2.29 AV: 9 NL: 2.71E7
T: FTMS - p ESI Full ms [100.00-750.00]

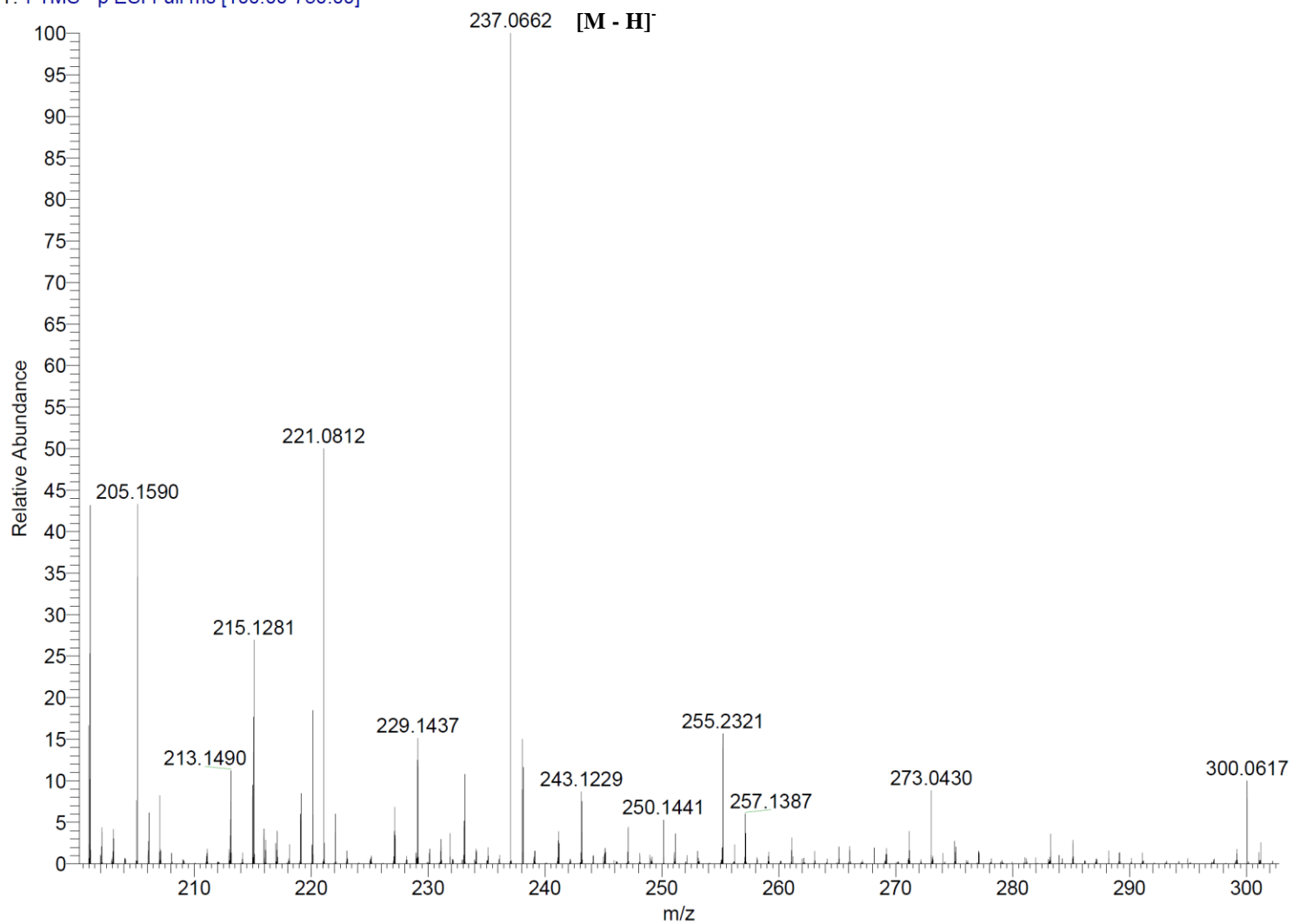


Figure S7: (-)-HRESI-MS spectrum of 1-Vinyl- β -carboline-3-carboxylic acid (1)

KS_E259_A_1HNMR_DMSO_01_16_2014
500 MHz, DMSO-d6, nt=32
Khaled A. Shaaban

Sample: khaled_A_Shaaban
File: xp

Pulse Sequence: s2pul

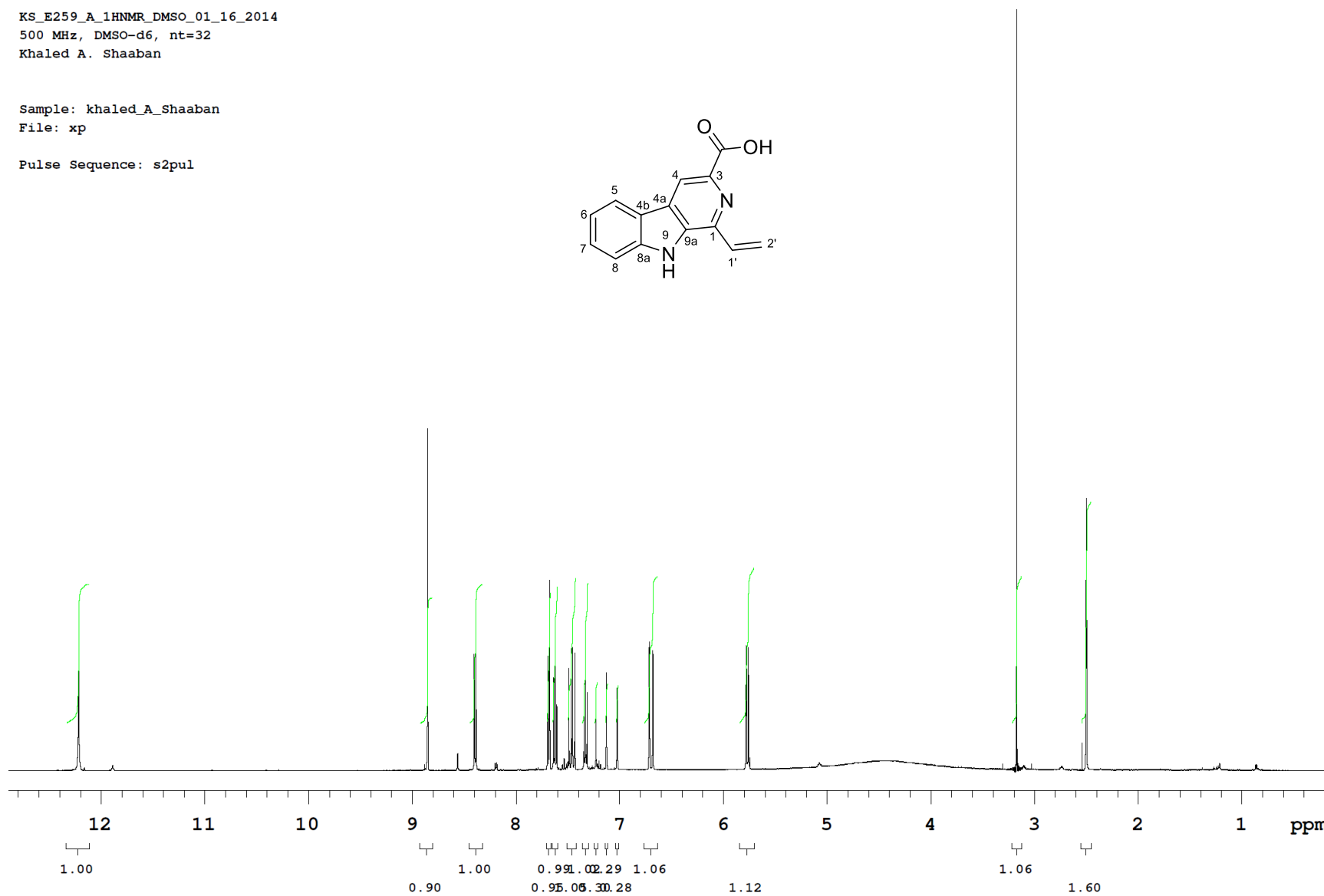
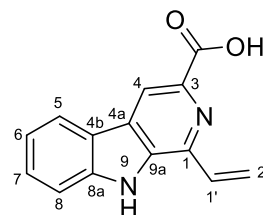


Figure S8: ¹H NMR spectrum (DMSO-*d*₆, 500 MHz) of 1-Vinyl-β-carboline-3-carboxylic acid (1)

KS_E259_A_13CNMR_DMSO_01_23_2014
125 MHz, DMSO-d6, 15 hrs
Khaled A. Shaaban

Sample: khaled_A_Shaaban
File: xp

Pulse Sequence: s2pul

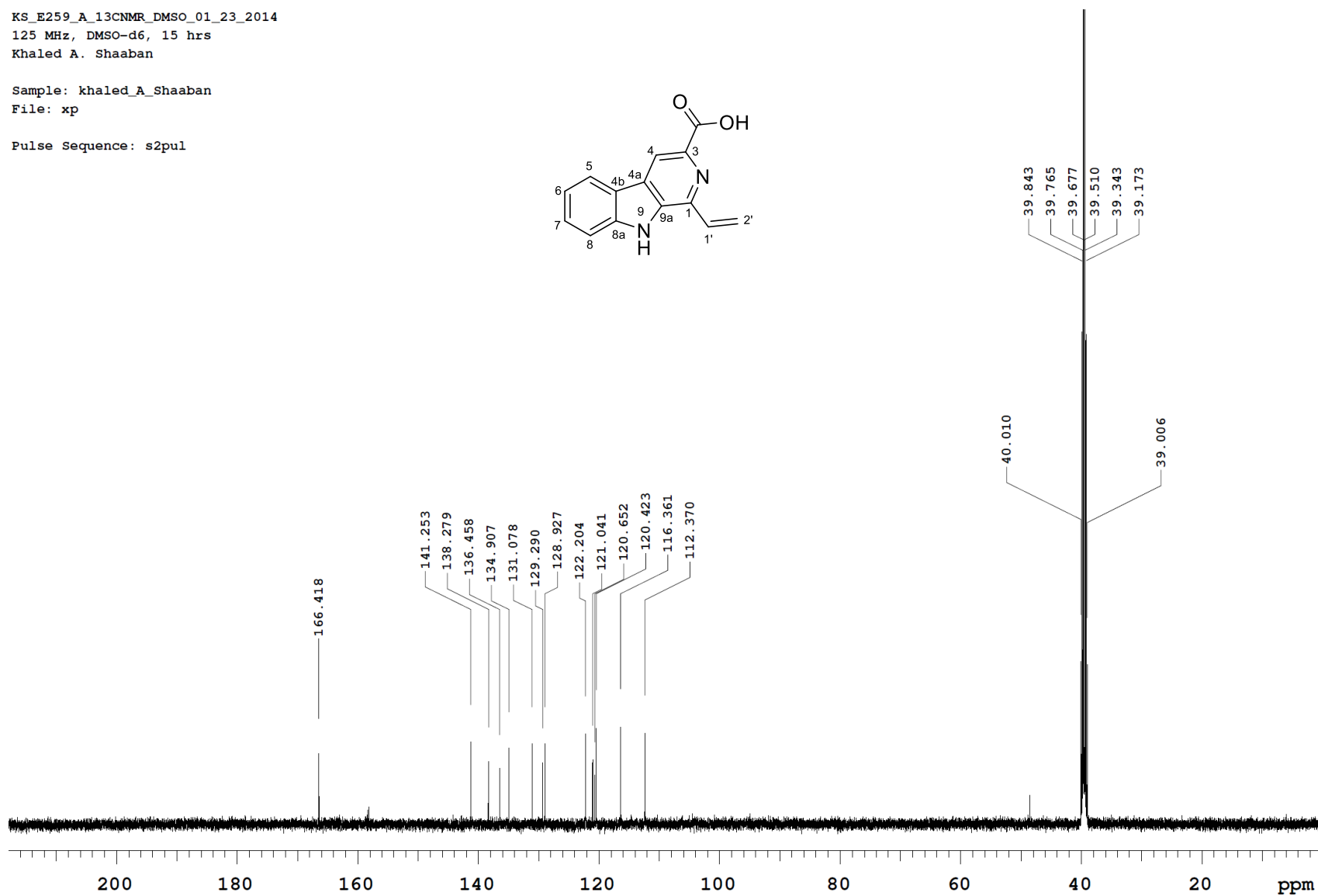


Figure S9: ¹³C NMR spectrum (DMSO-d₆, 125 MHz) of 1-Vinyl-β-carboline-3-carboxylic acid (1)

KS_E259_A_gCOSY_DMSO_01_16_2014
500 MHz, DMSO-d₆, 60 mins
Khaled A. Shaaban

Sample: khaled_A_Shaaban
File: xp

Pulse Sequence: gCOSY

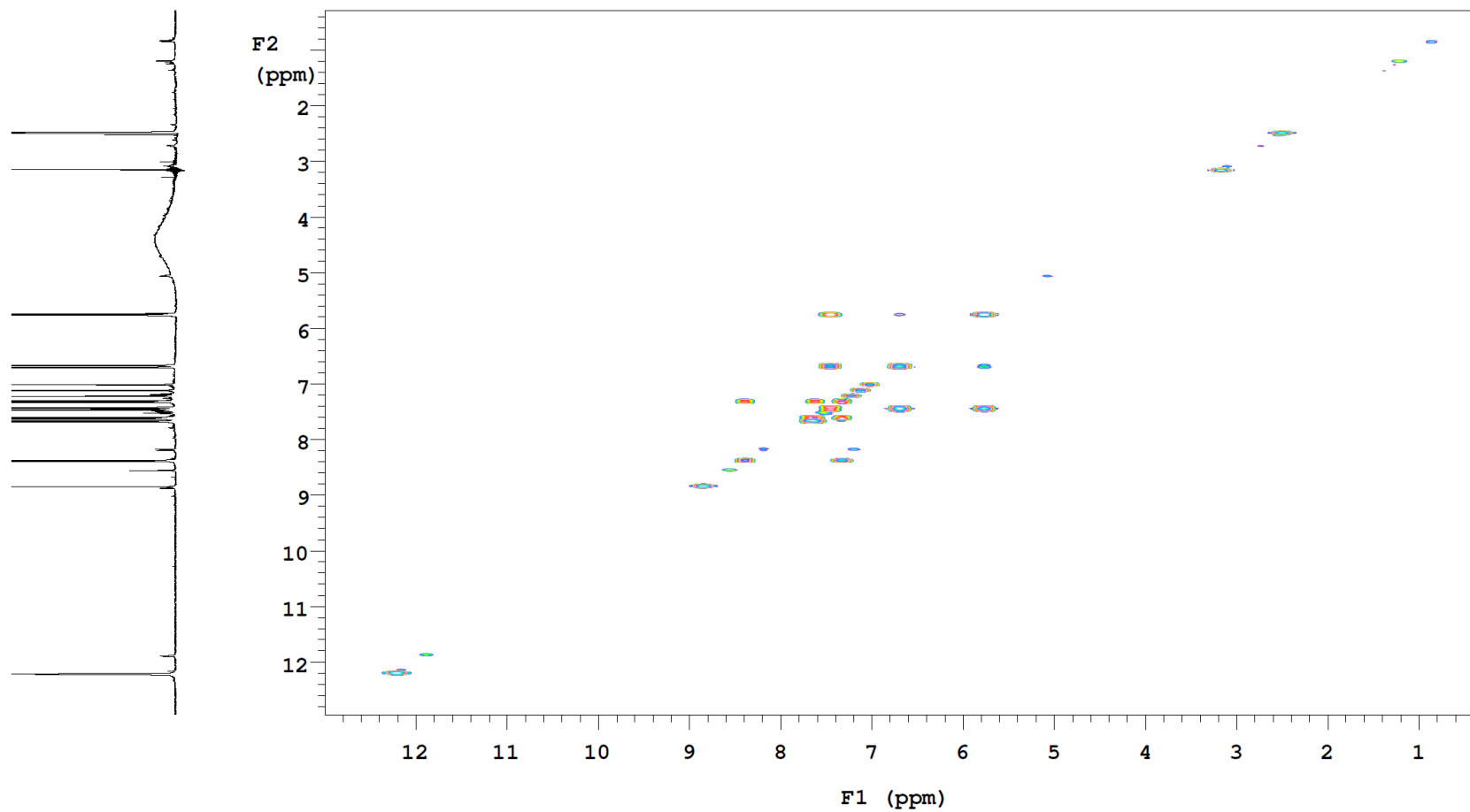
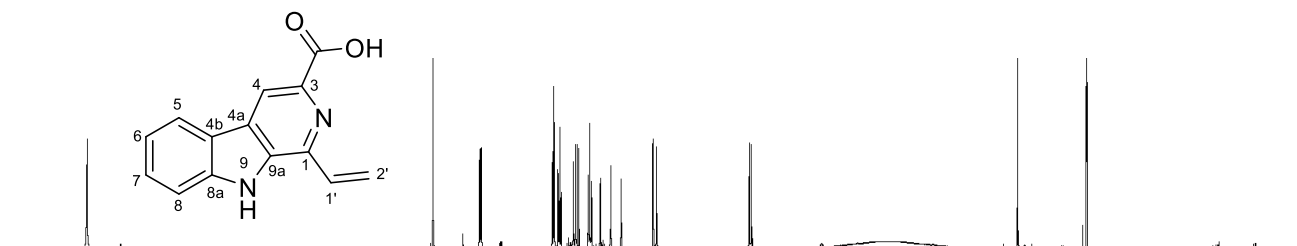
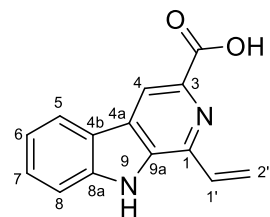


Figure S10: ¹H-¹H COSY spectrum (DMSO-*d*₆, 500 MHz) of 1-Vinyl-β-carboline-3-carboxylic acid (**1**)

KS_E259_A_gHSQC_DMSO_01_24_2014
500 MHz, DMSO-d6, 3 hrs
Khaled A. Shaaban



Sample: Khaled_A_Shaaban
File: xp

Pulse Sequence: gHSQC

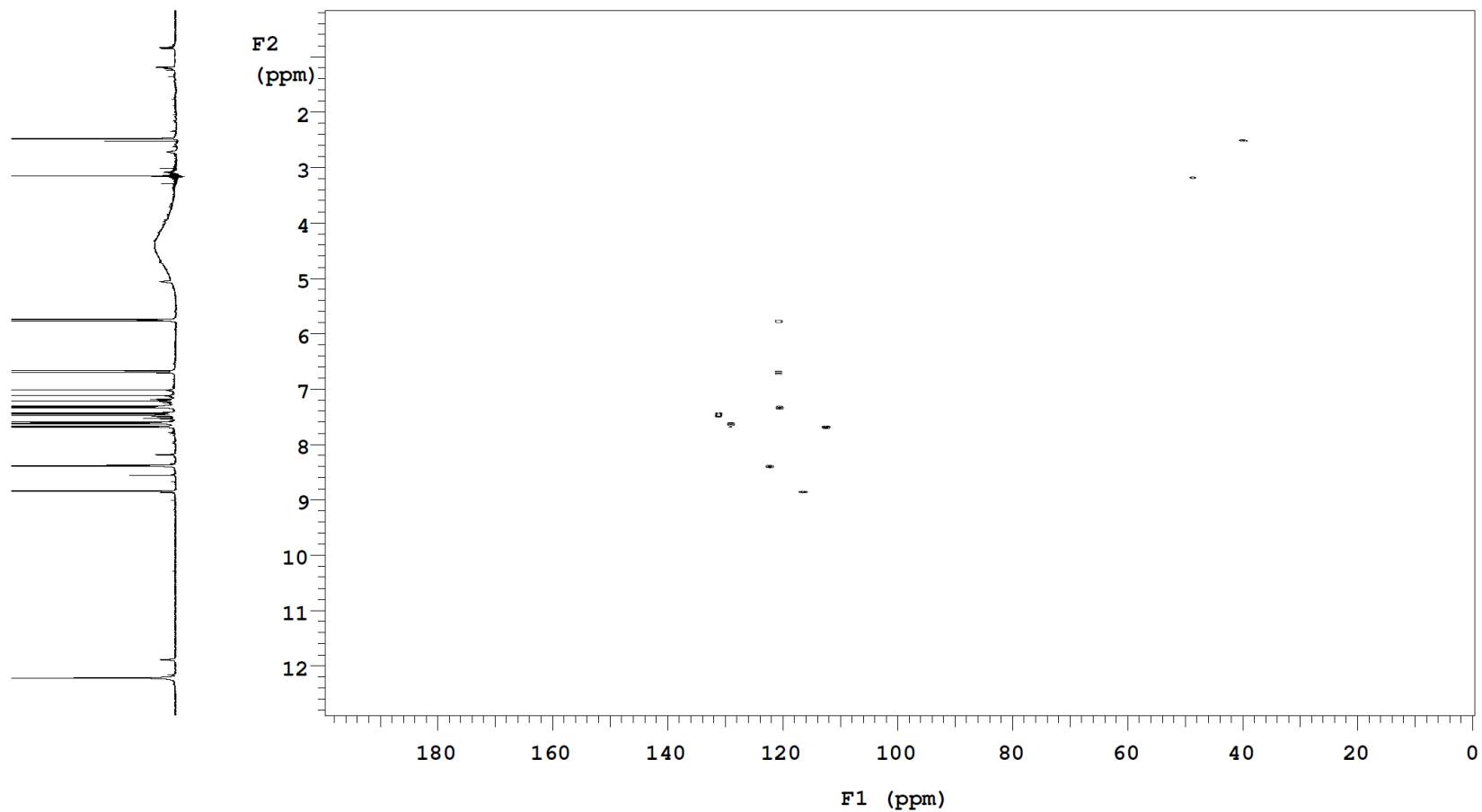


Figure S11: HSQC spectrum (DMSO- d_6 , 500 MHz) of 1-Vinyl- β -carboline-3-carboxylic acid (1)

KS_E259_A_gHMBC_DMSO_01_24_2014
500 MHz, DMSO-d₆, 12 hrs
Khaled A. Shaaban

Sample: khaled_A_Shaaban
File: xp

Pulse Sequence: gHMBC

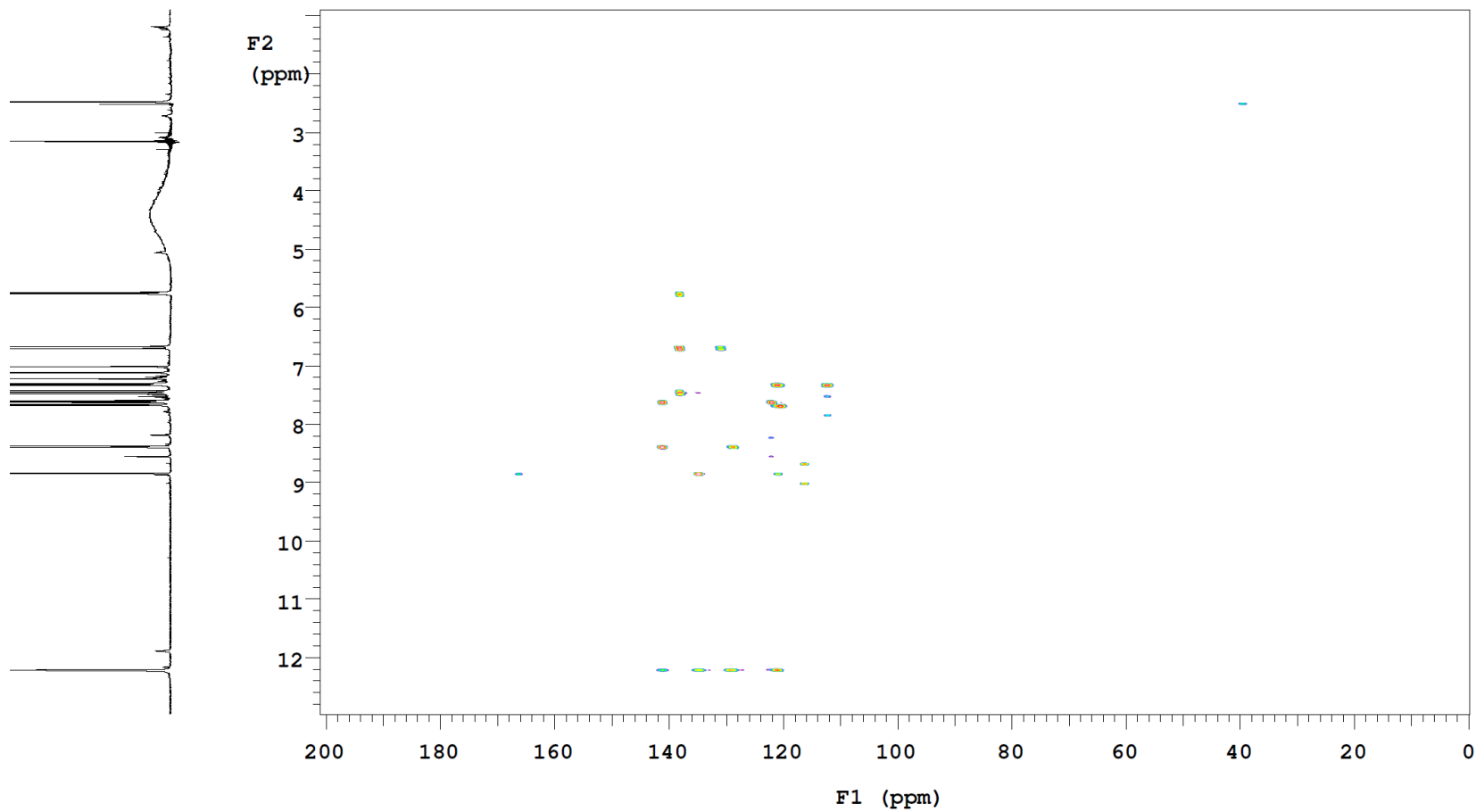
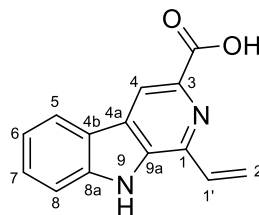


Figure S12: HMBC spectrum (DMSO-*d*₆, 500 MHz) of 1-Vinyl- β -carboline-3-carboxylic acid (1)

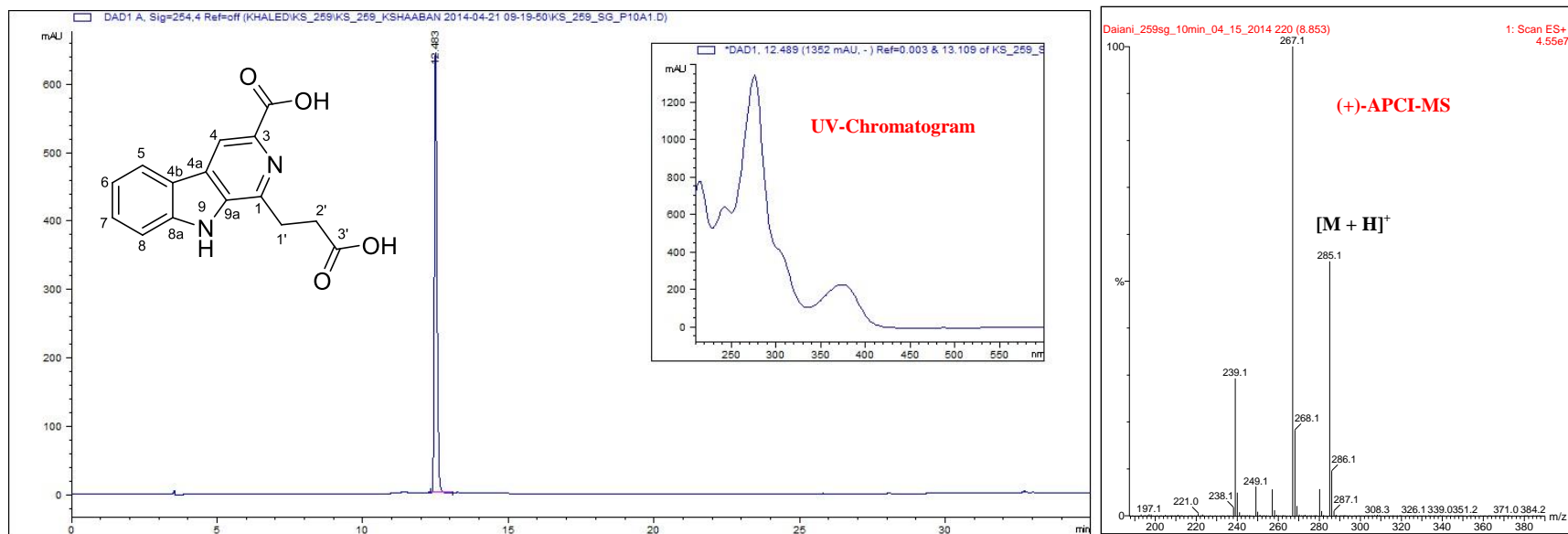


Figure S13: HPLC/UV/APCI-MS analyses of JBIR-133 (**2**). HPLC-conditions: Detection wavelength 254 nm; solvent A: H₂O/0.1% Formic acid; solvent B: acetonitrile; flow rate: 1.0 mL min⁻¹; 0-35 min, 95-0% A (linear gradient); 35-40 min 0-95% A (linear gradient).

Sample: Khaled_A_Shaaban
File: xp

Pulse Sequence: s2pul

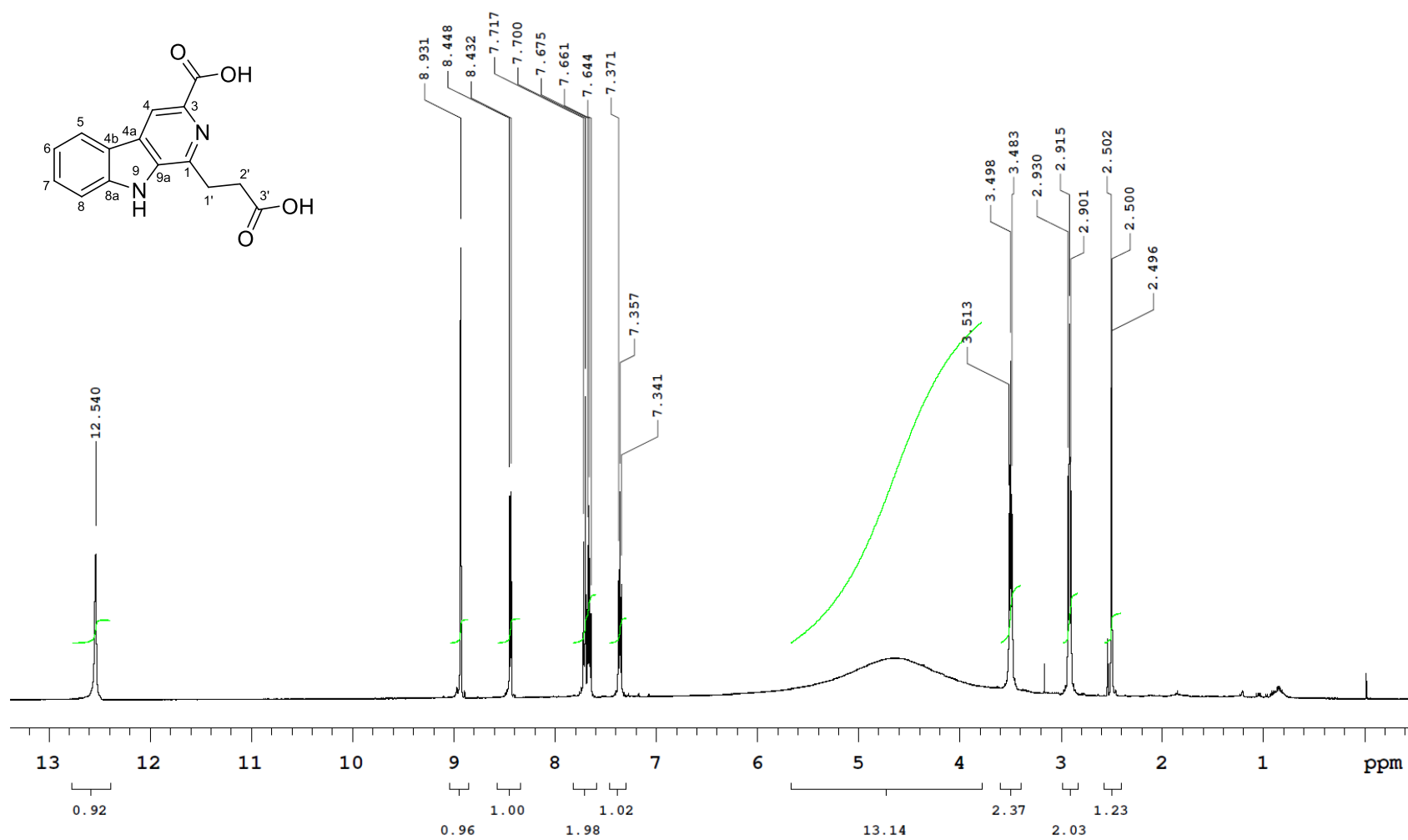


Figure S14: ¹H NMR spectrum (DMSO-d₆, 500 MHz) of JBIR-133 (2)

Sample: Khaled_A_Shaaban
File: xp
Pulse Sequence: s2pul

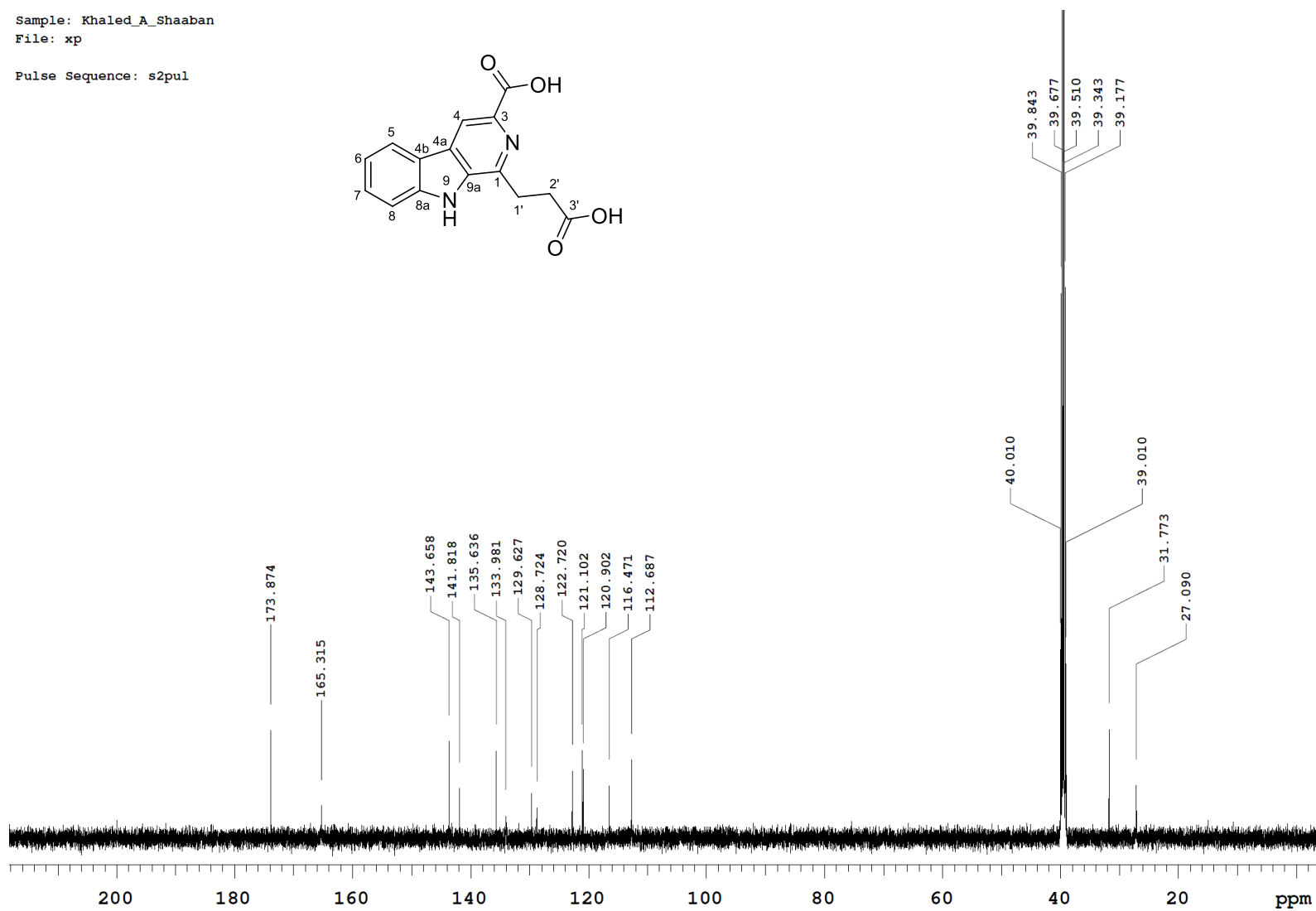
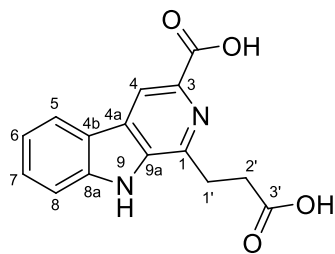


Figure S15: ¹³C NMR spectrum (DMSO-*d*₆, 125 MHz) of JBIR-133 (2)

KS_259_SG_F10A_gCOSY_DMSO_04_23_2014
500 MHz, DMSO-d6, time=80 mins
Khaled A. Shaaban

Sample: Khaled_A_Shaaban
File: xp

Pulse Sequence: gCOSY

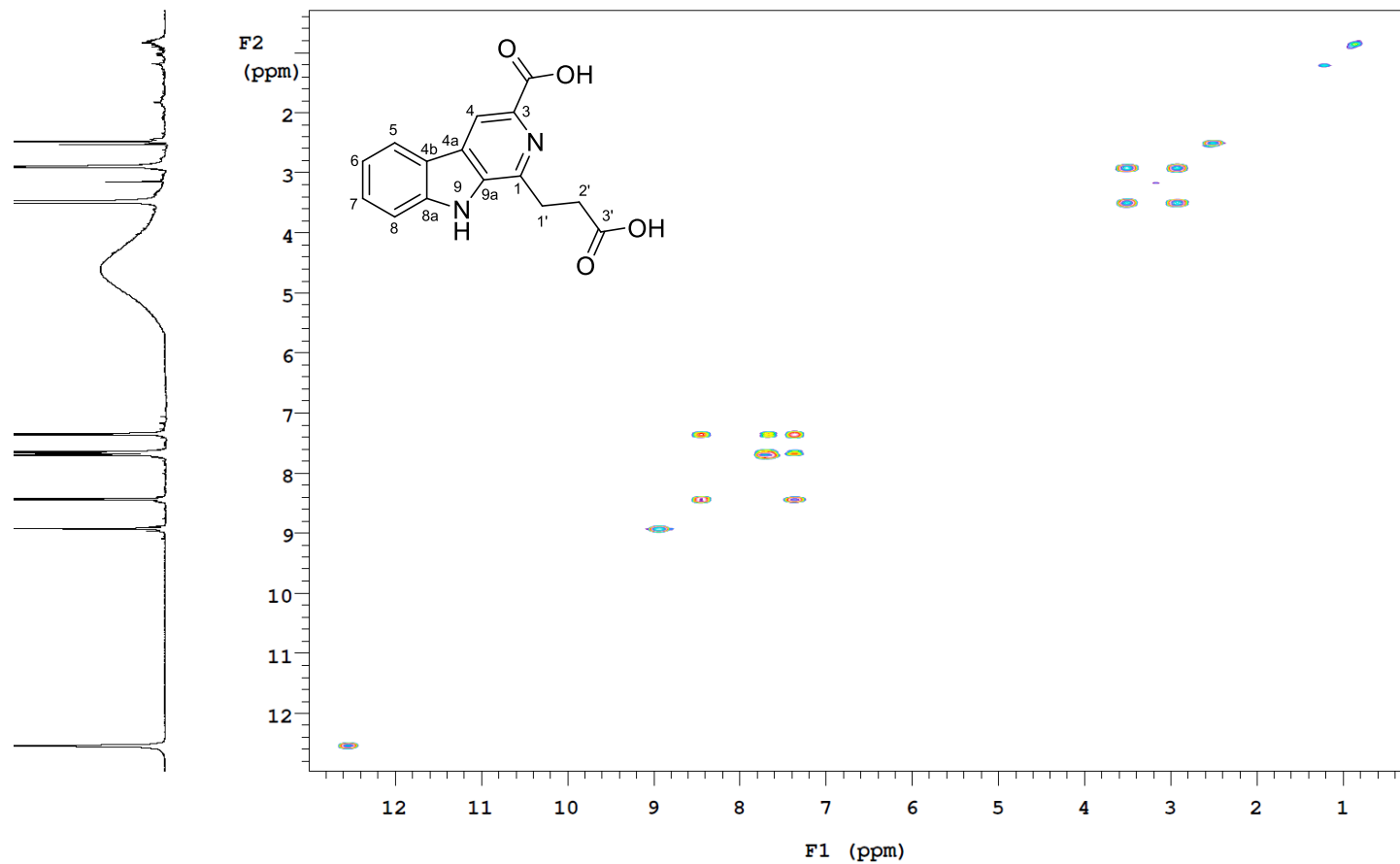


Figure S16: ^1H - ^1H COSY spectrum (DMSO- d_6 , 500 MHz) of JBIR-133 (2)

Sample: Khaled_A_Shaaban

File: xp

Pulse Sequence: gHSQC

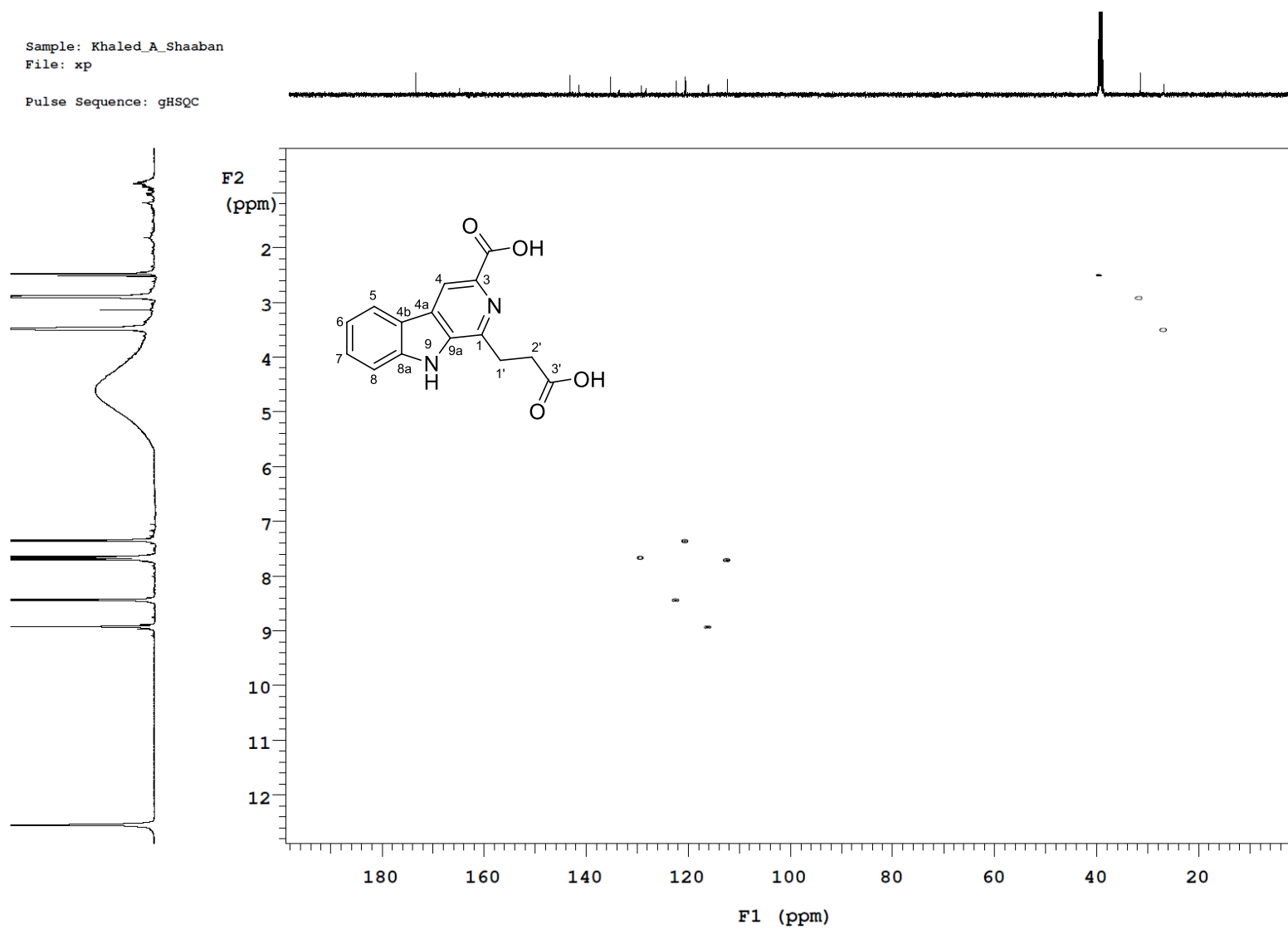


Figure S17: HSQC spectrum (DMSO-*d*₆, 500 MHz) of JBIR-133 (2)

Sample: Khaled_A_Shaaban
File: xp
Pulse Sequence: gHMBC

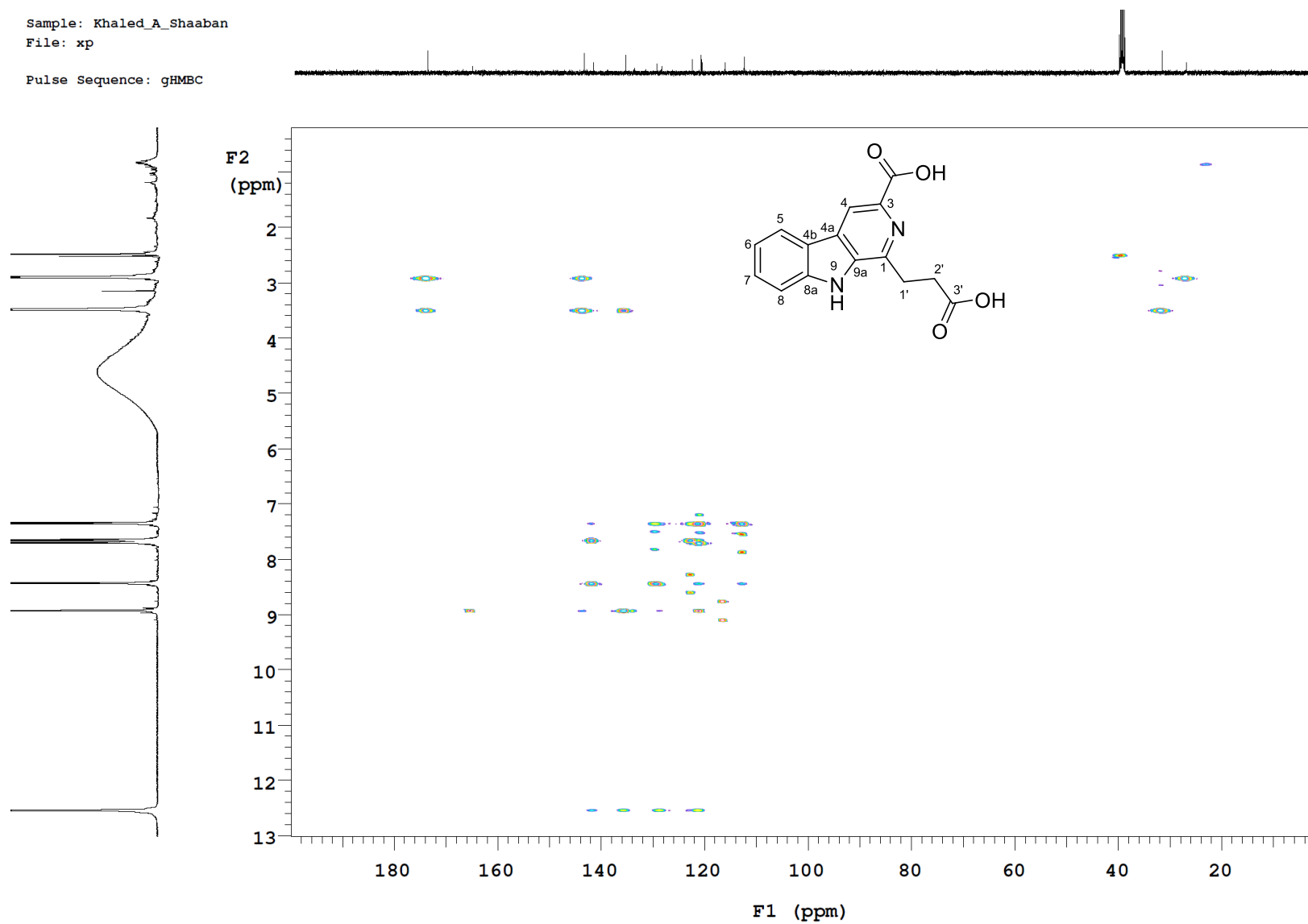


Figure S18: HMBC spectrum (DMSO- d_6 , 500 MHz) of JBIR-133 (2)

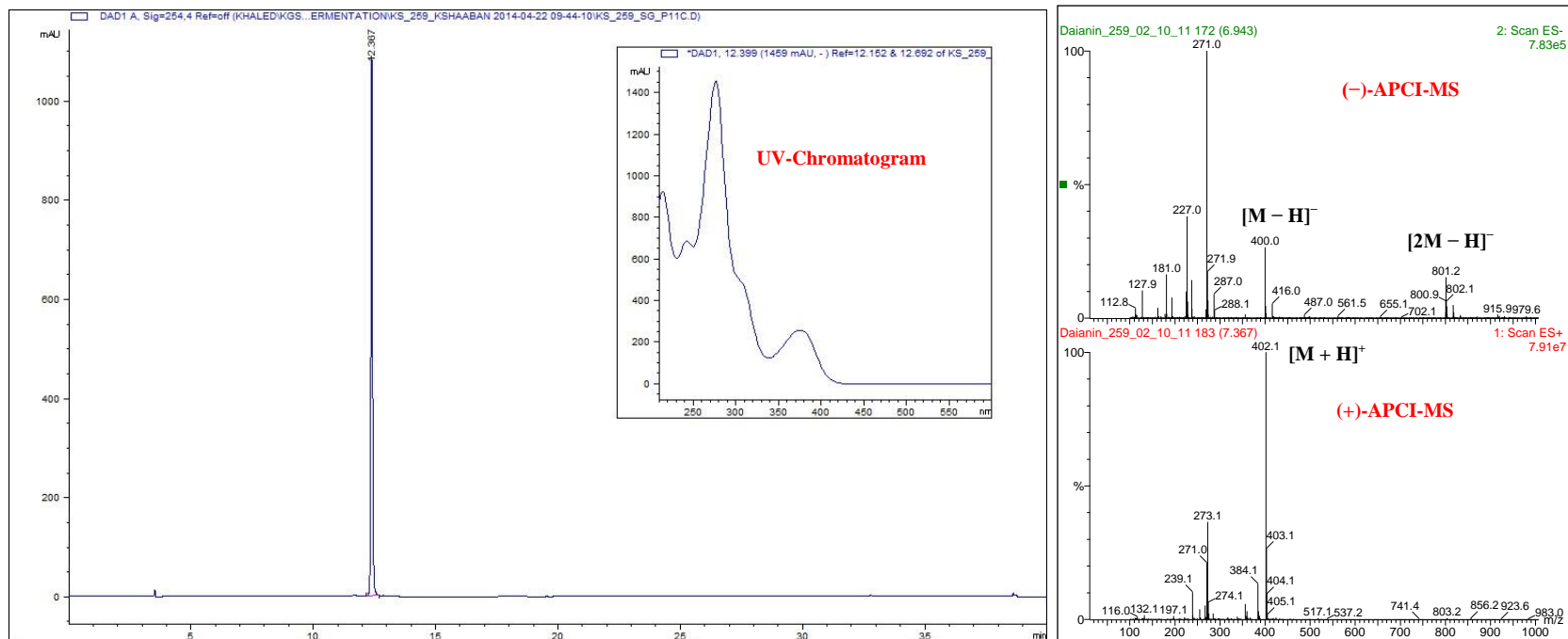
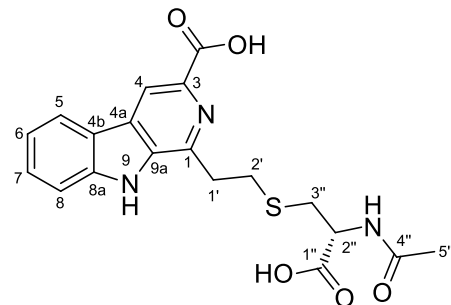


Figure S19: HPLC/UV/APCI-MS analyses of Kitasetaline (**3**). HPLC-conditions: Detection wavelength 254 nm; solvent A: H₂O/0.1% Formic acid; solvent B: acetonitrile; flow rate: 1.0 mL min⁻¹; 0-35 min, 95-0% A (linear gradient); 35-40 min 0-95% A (linear gradient).

Sample: Khaled_A_Shaaban

File: xp

Pulse Sequence: s2pul

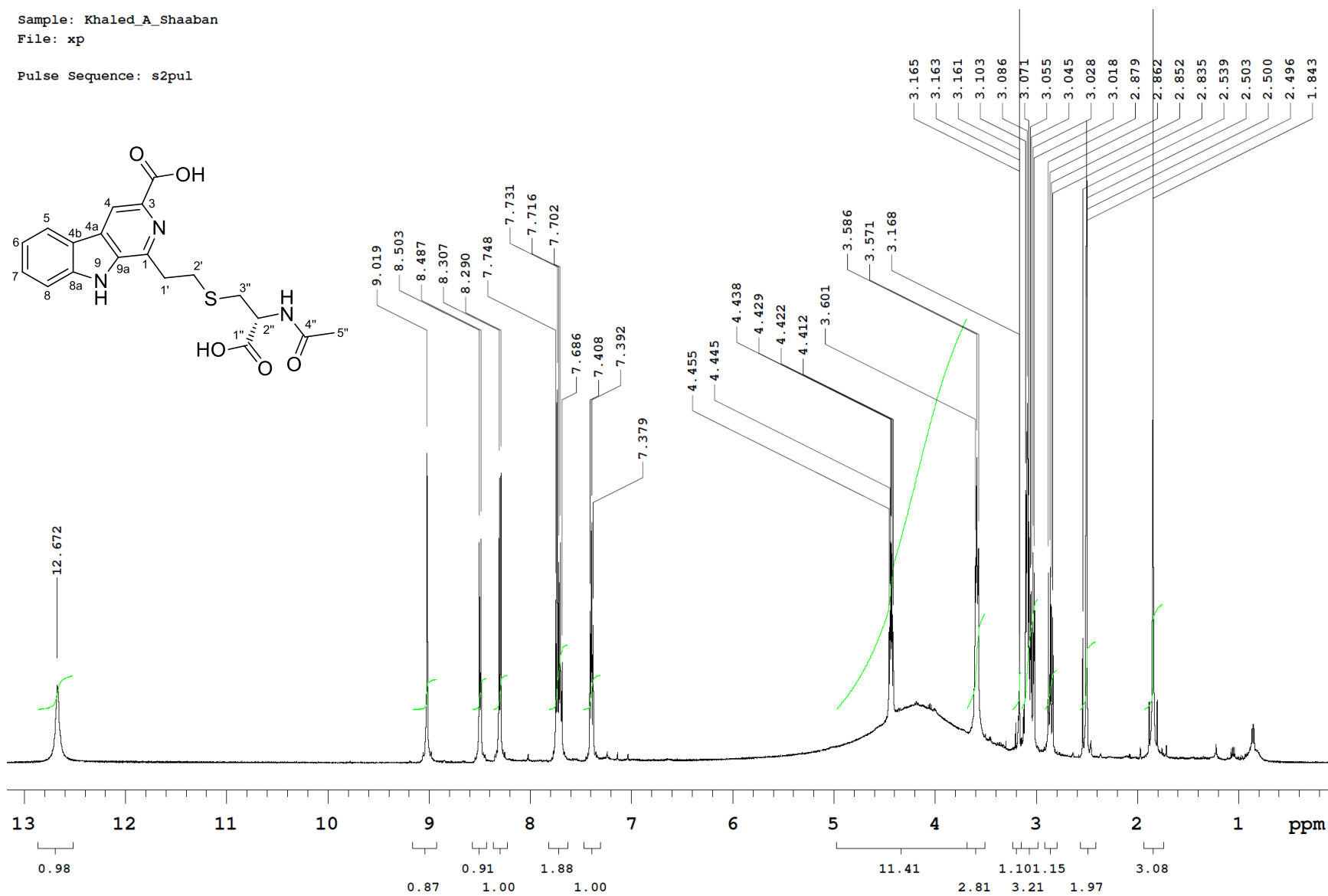


Figure S20: ¹H NMR spectrum (DMSO-*d*₆, 500 MHz) of Kitasetaline (3)

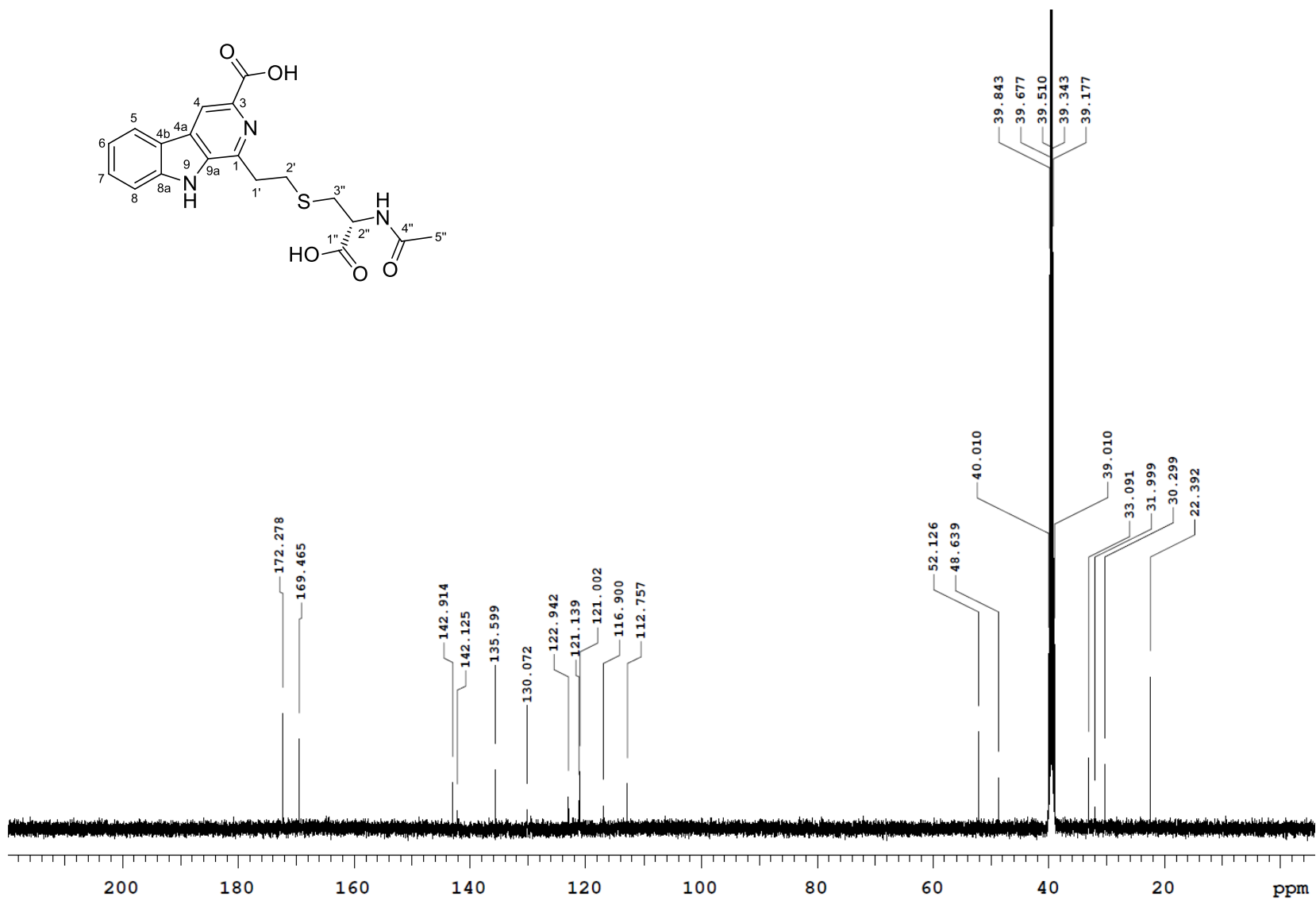


Figure S21: ^{13}C NMR spectrum (DMSO- d_6 , 125 MHz) of Kitasetaline (3)

Sample: Khaled_A_Shaaban

File: xp

Pulse Sequence: gCOSY

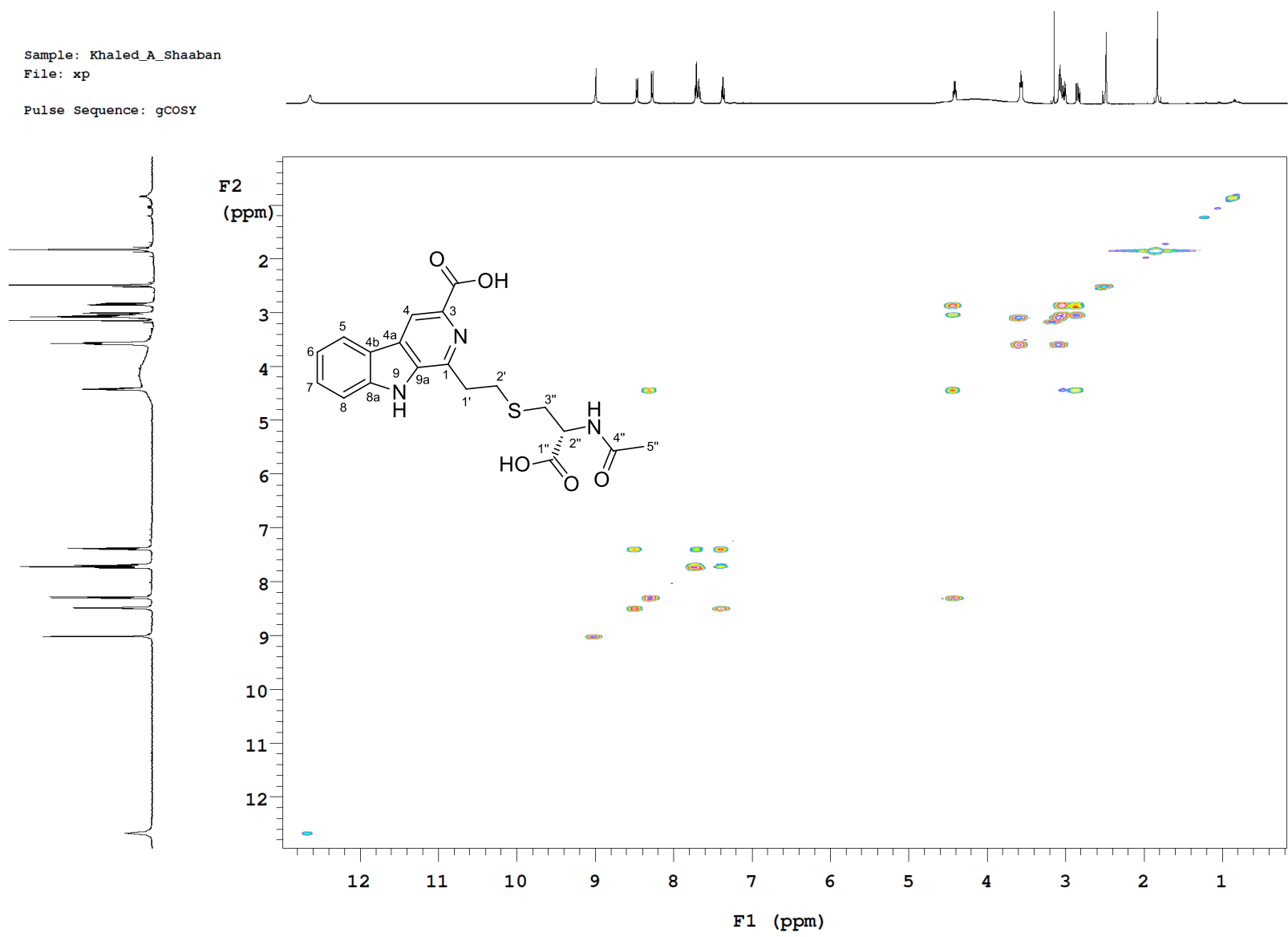


Figure S22: ^1H - ^1H COSY spectrum (DMSO- d_6 , 500 MHz) of Kitasetaline (**3**)

Sample: Khaled_A_Shaaban

File: xp

Pulse Sequence: gHSQC

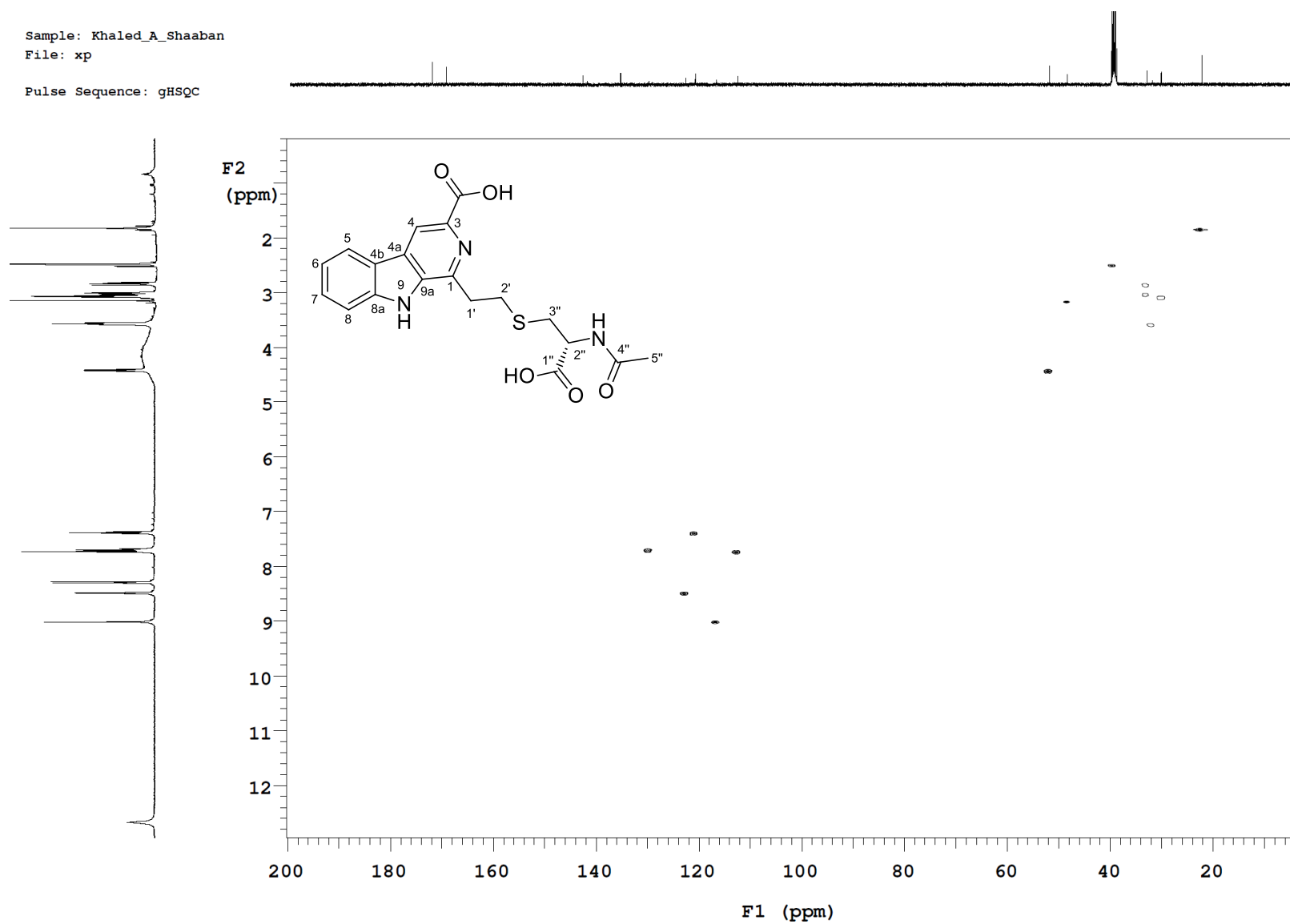


Figure S23: HSQC spectrum (DMSO-*d*₆, 500 MHz) of Kitasetaline (3)

Sample: Khaled_A_Shaaban

File: xp

Pulse Sequence: gHMBC

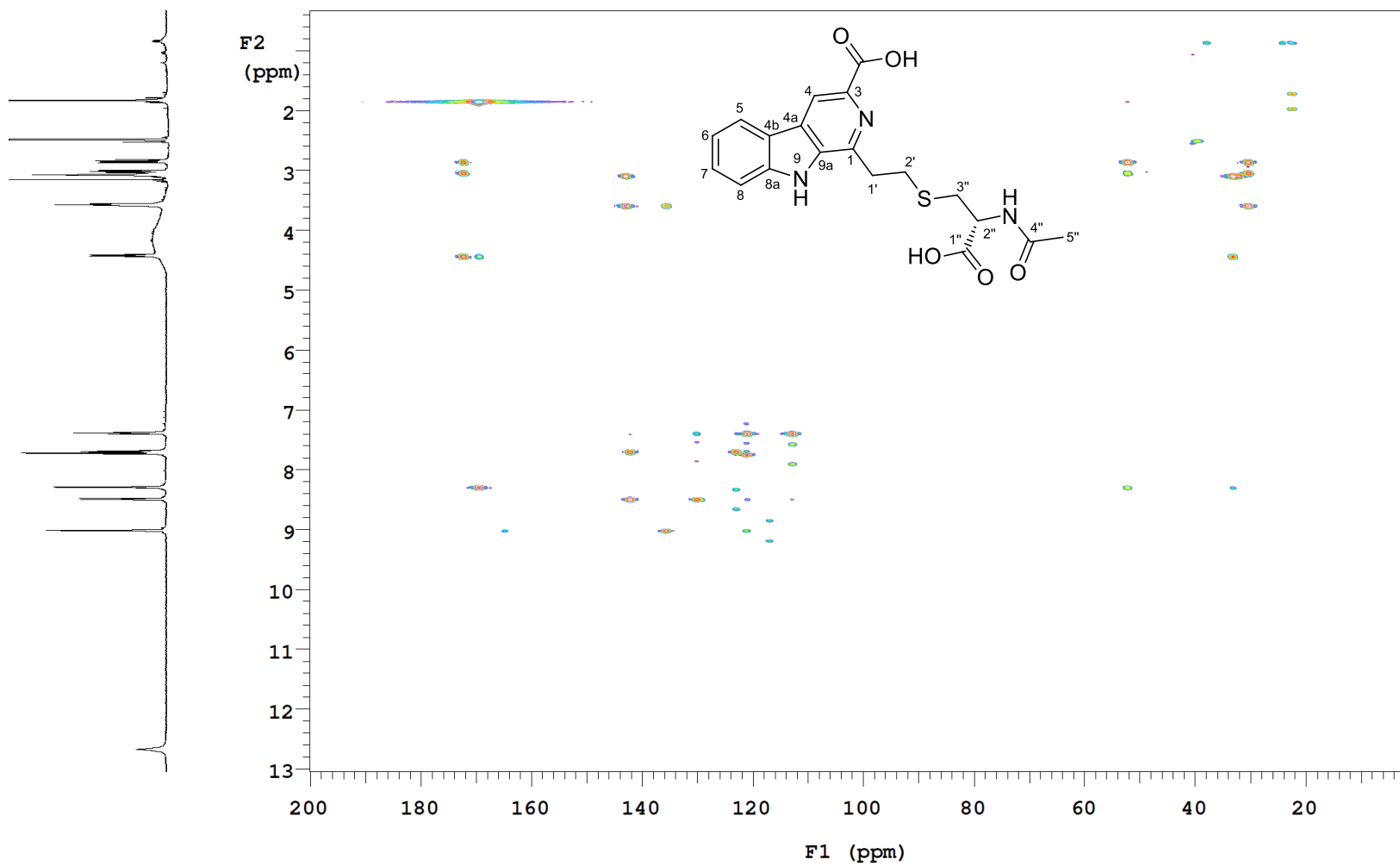


Figure S24: HMBC spectrum (DMSO- d_6 , 500 MHz) of Kitasetaline (3)

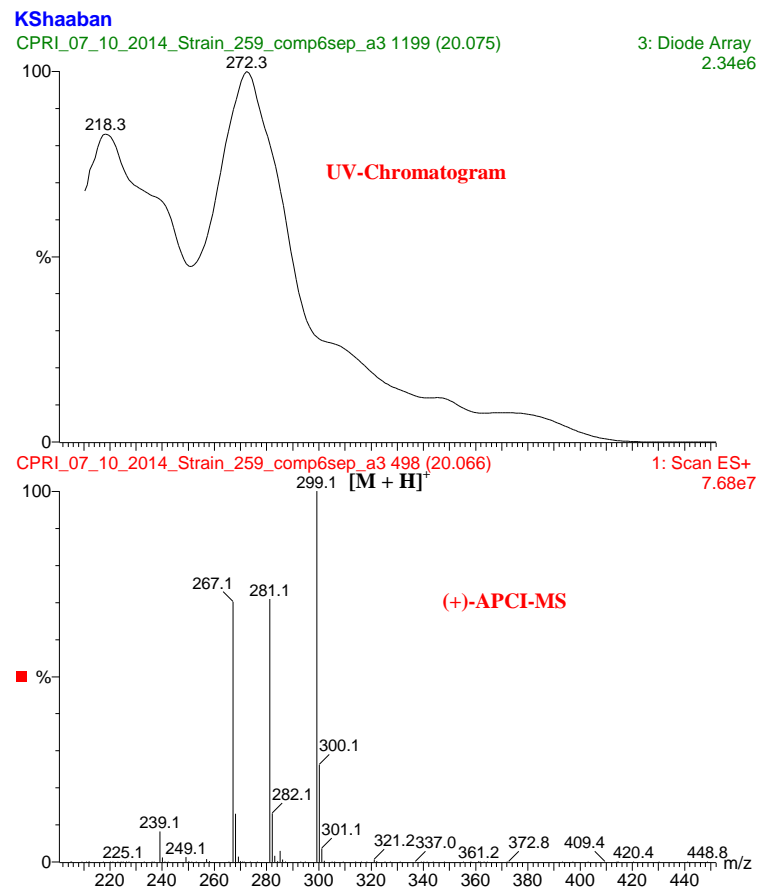
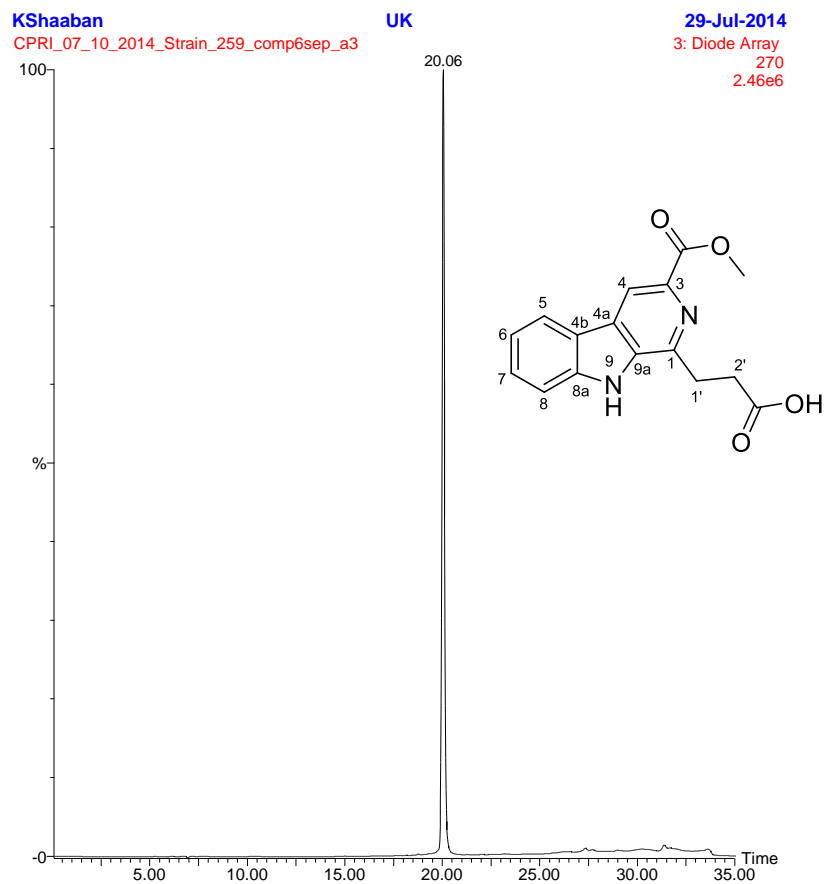


Figure S25: HPLC/UV/APCI-MS analyses of 1-(Propionic acid)- β -carboline-3-carboxylic acid methyl ester (**4**). HPLC-conditions: Detection wavelength 270 nm; solvent A: H₂O/0.1% Formic acid; solvent B: acetonitrile; flow rate: 0.5 mL min⁻¹; 0-4 min, 90% A; 4-22 min, 90-0% A (linear gradient); 22-27 min 0% A; 27-35 min 0-90% A (linear gradient).

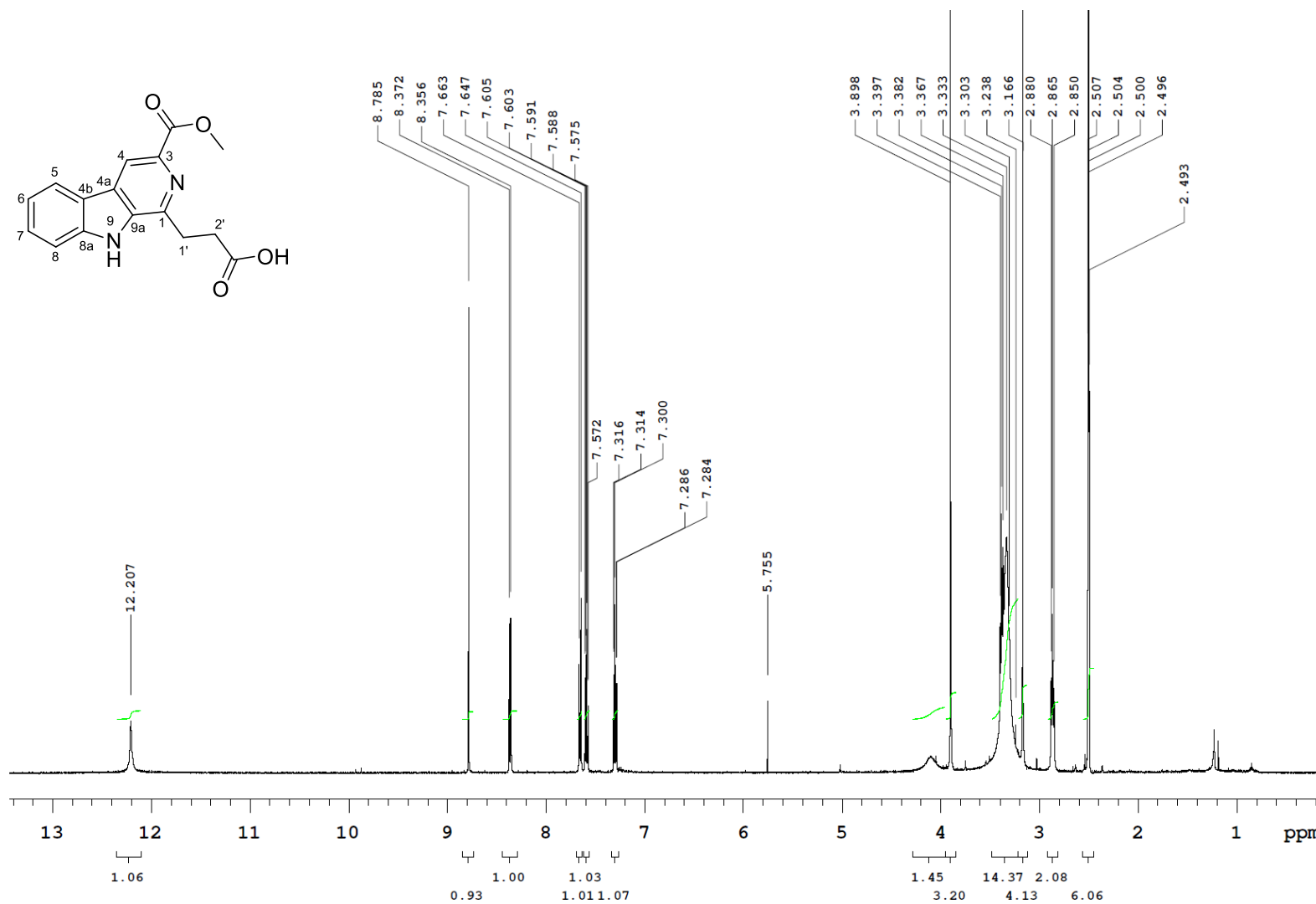


Figure S26: ¹H NMR spectrum (DMSO-*d*₆, 500 MHz) of 1-(Propionic acid)-β-carboline-3-carboxylic acid methyl ester (4)

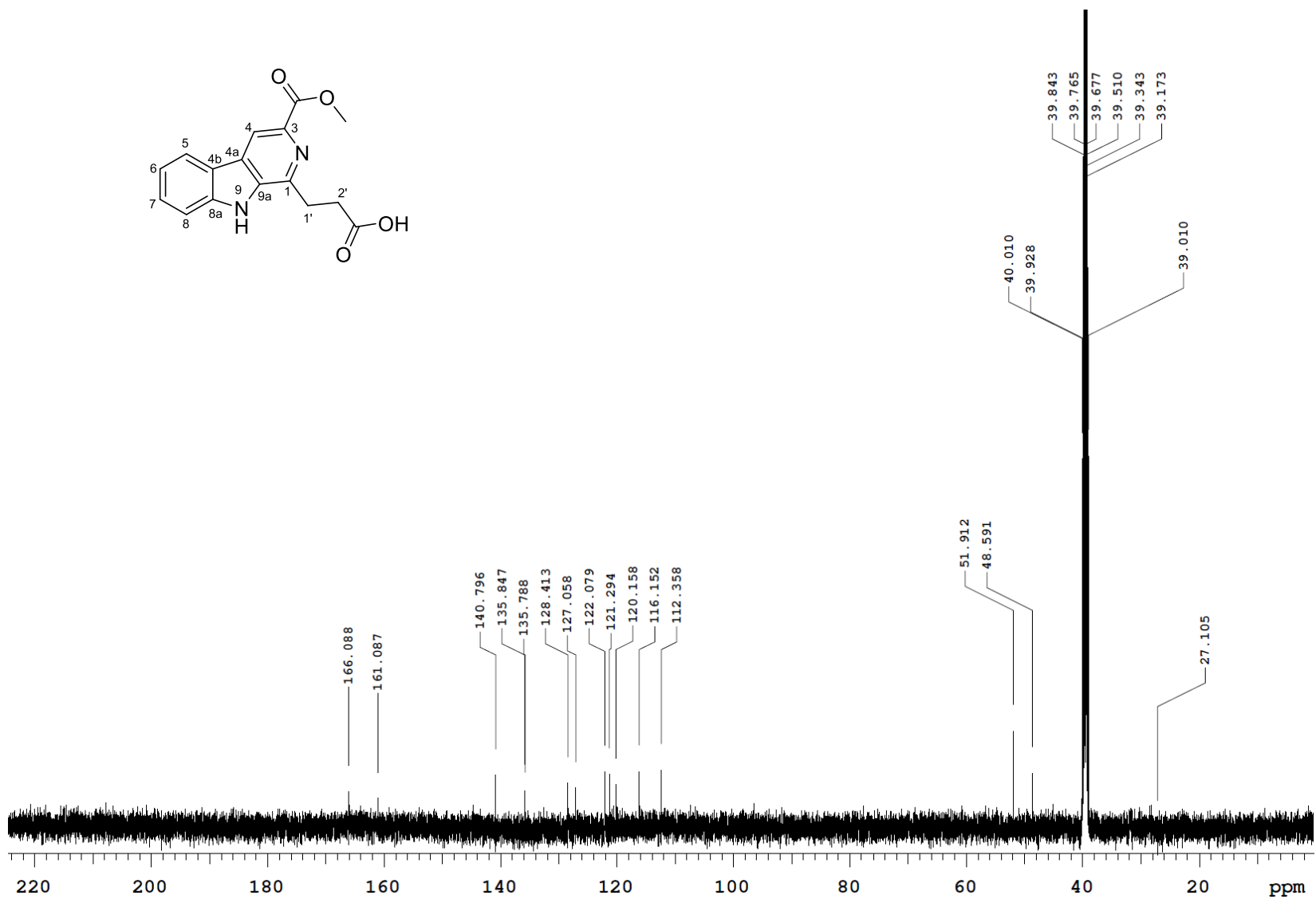


Figure S27: ¹³C NMR spectrum (DMSO-d₆, 125 MHz) of 1-(Propionic acid)-β-carboline-3-carboxylic acid methyl ester (4)

KS_259_6A3_gCOSY_DMSO_08_12_2014
500 MHz, DMSO-d6, 80 min
Khaled A. Shaaban
Sample: khaled_A_Shaaban
File: xp
Pulse Sequence: gCOSY

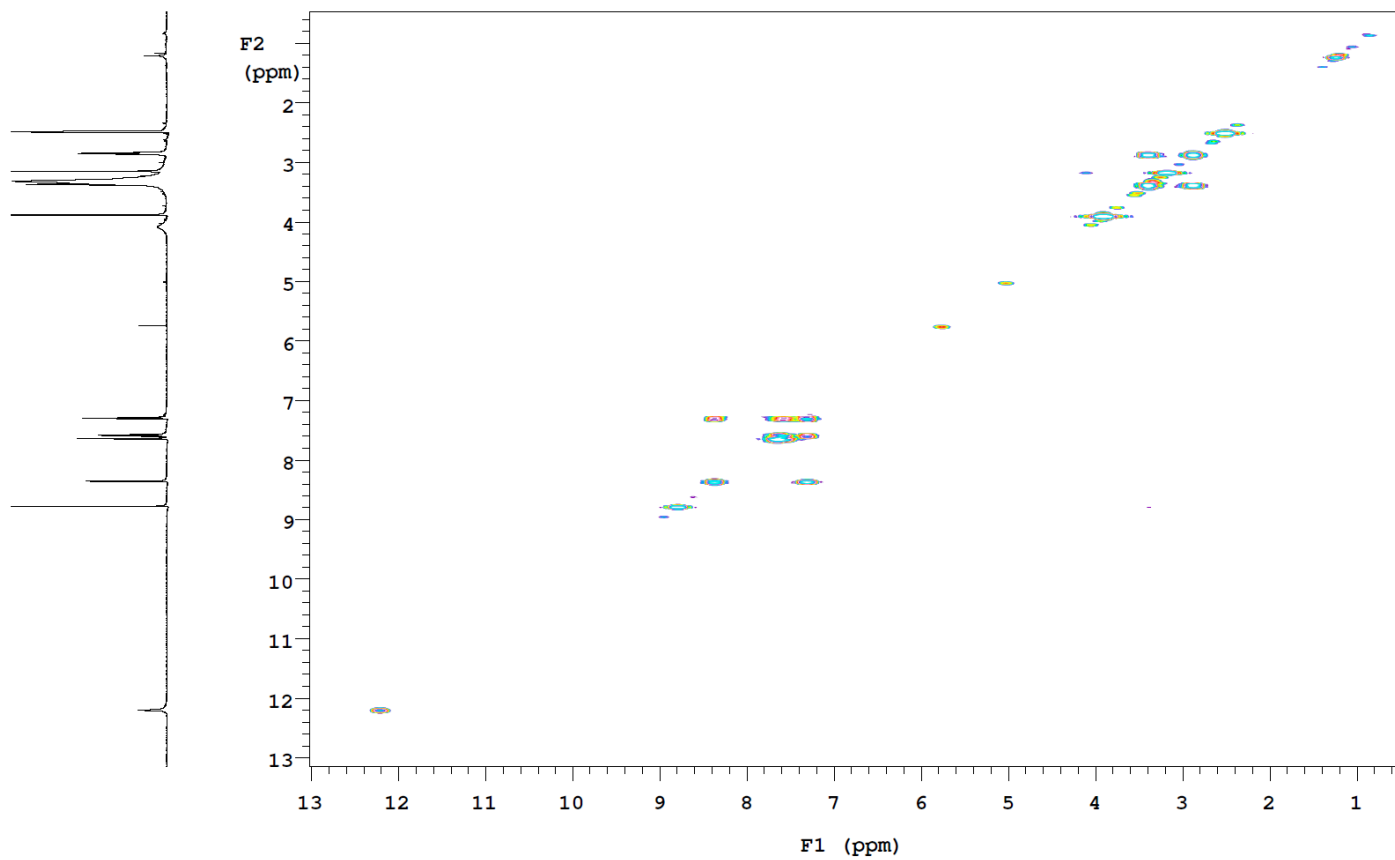
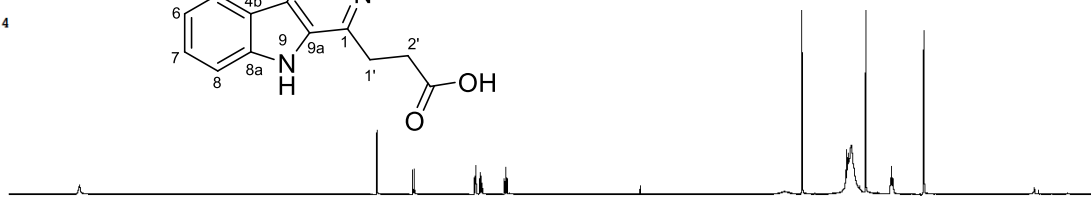
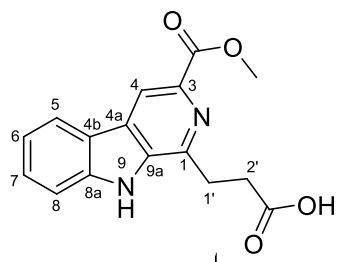


Figure S28: ^1H - ^1H COSY spectrum (DMSO- d_6 , 500 MHz) of 1-(Propionic acid)- β -carboline-3-carboxylic acid methyl ester (4)

Sample: khaled_A_Shaaban
File: xp
Pulse Sequence: gHSQC

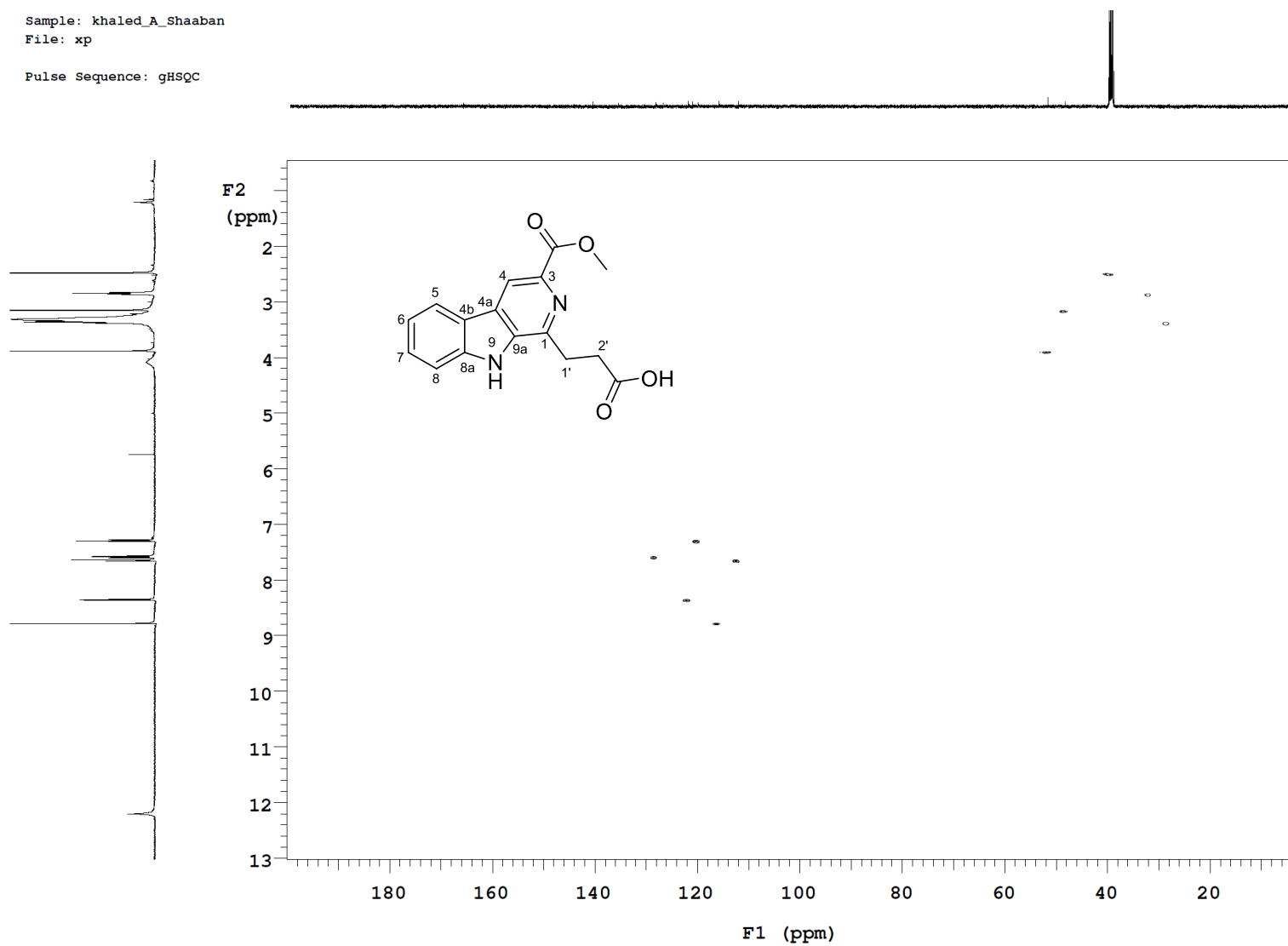


Figure S29: HSQC spectrum (DMSO-*d*₆, 500 MHz) of 1-(Propionic acid)-β-carboline-3-carboxylic acid methyl ester (**4**)

Sample: khaled_A_Shaaban
File: xp
Pulse Sequence: gHMBC

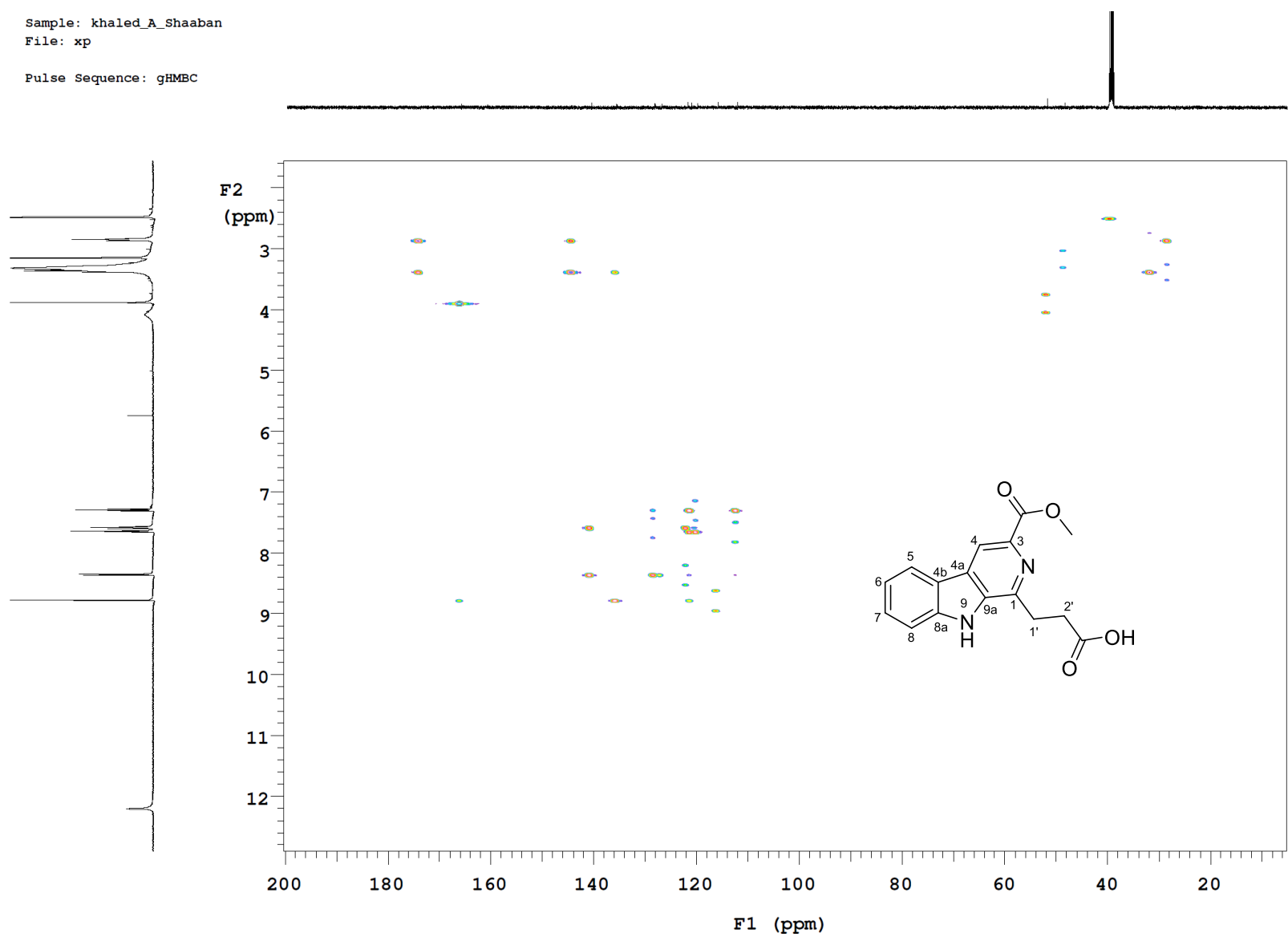


Figure S30: HMBC spectrum (DMSO-*d*₆, 500 MHz) of 1-(Propionic acid)-β-carboline-3-carboxylic acid methyl ester (4)

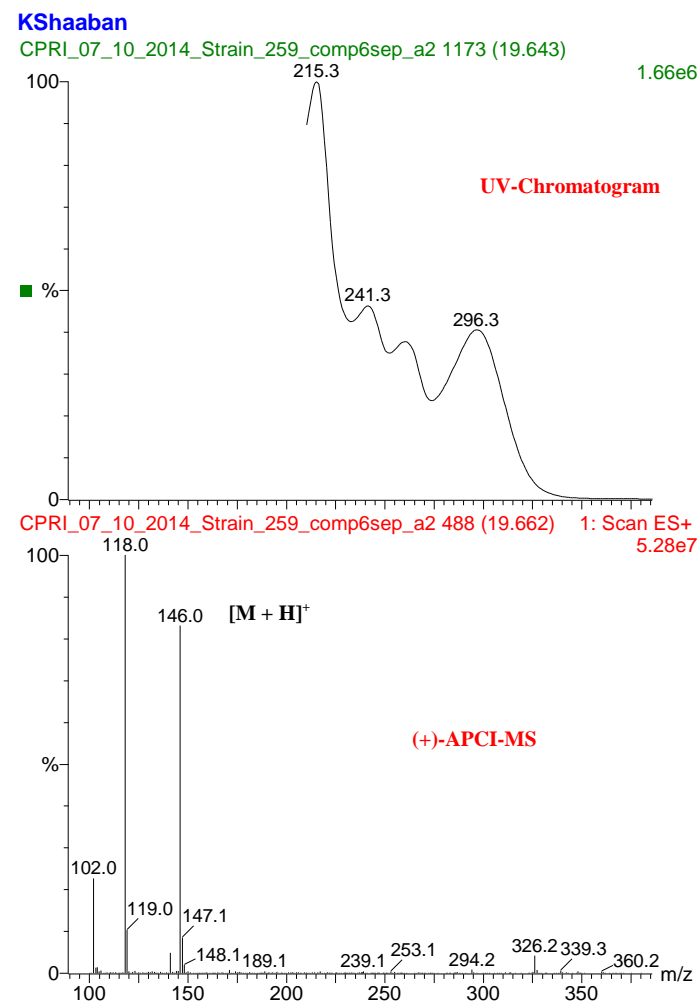
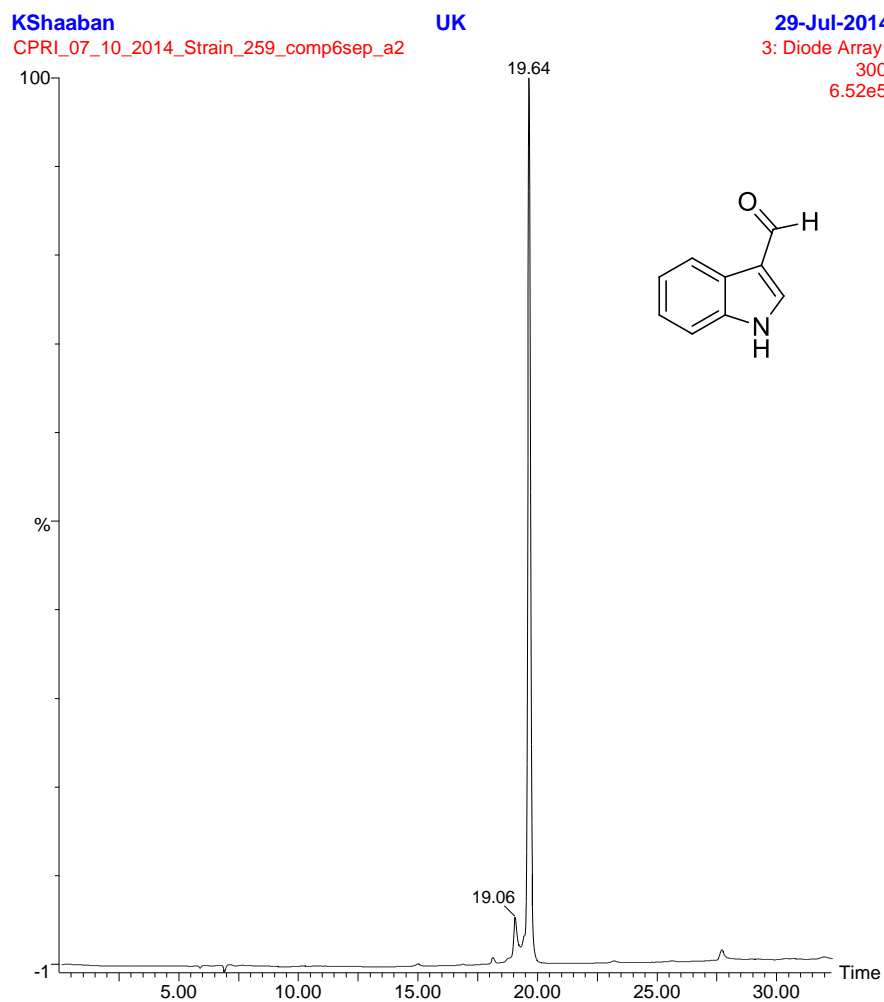


Figure S31: HPLC/UV/APCI-MS analyses of Indole-3-carbaldehyde (**5**). HPLC-conditions: Detection wavelength 270 nm; solvent A: H₂O/0.1% Formic acid; solvent B: acetonitrile; flow rate: 0.5 mL min⁻¹; 0-4 min, 90% A; 4-22 min, 90-0% A (linear gradient); 22-27 min 0% A; 27-35 min 0-90% A (linear gradient).

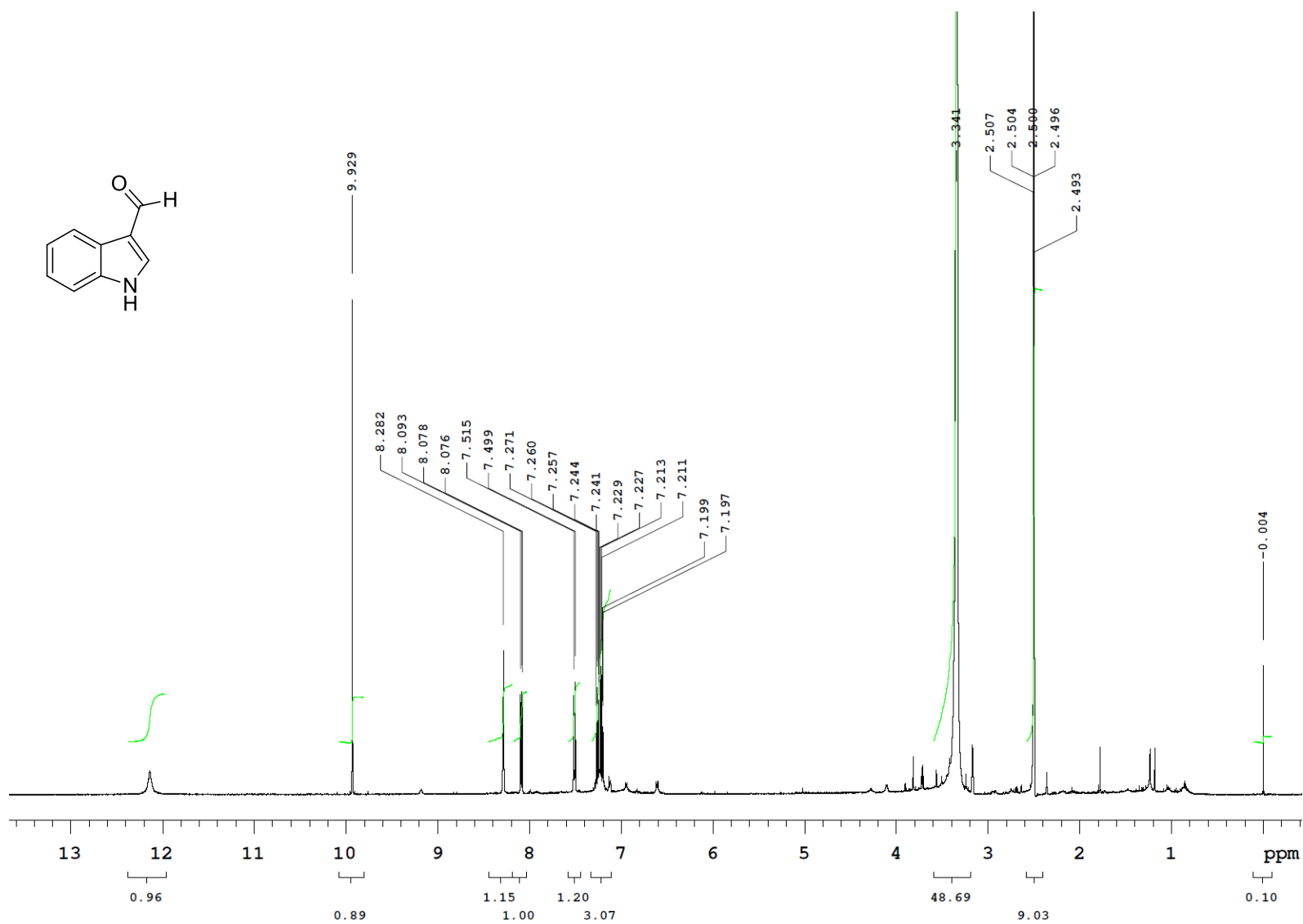


Figure S32: ¹H NMR spectrum (DMSO-*d*₆, 500 MHz) of Indole-3-carbaldehyde (5)

Pulse Sequence: gHSQC

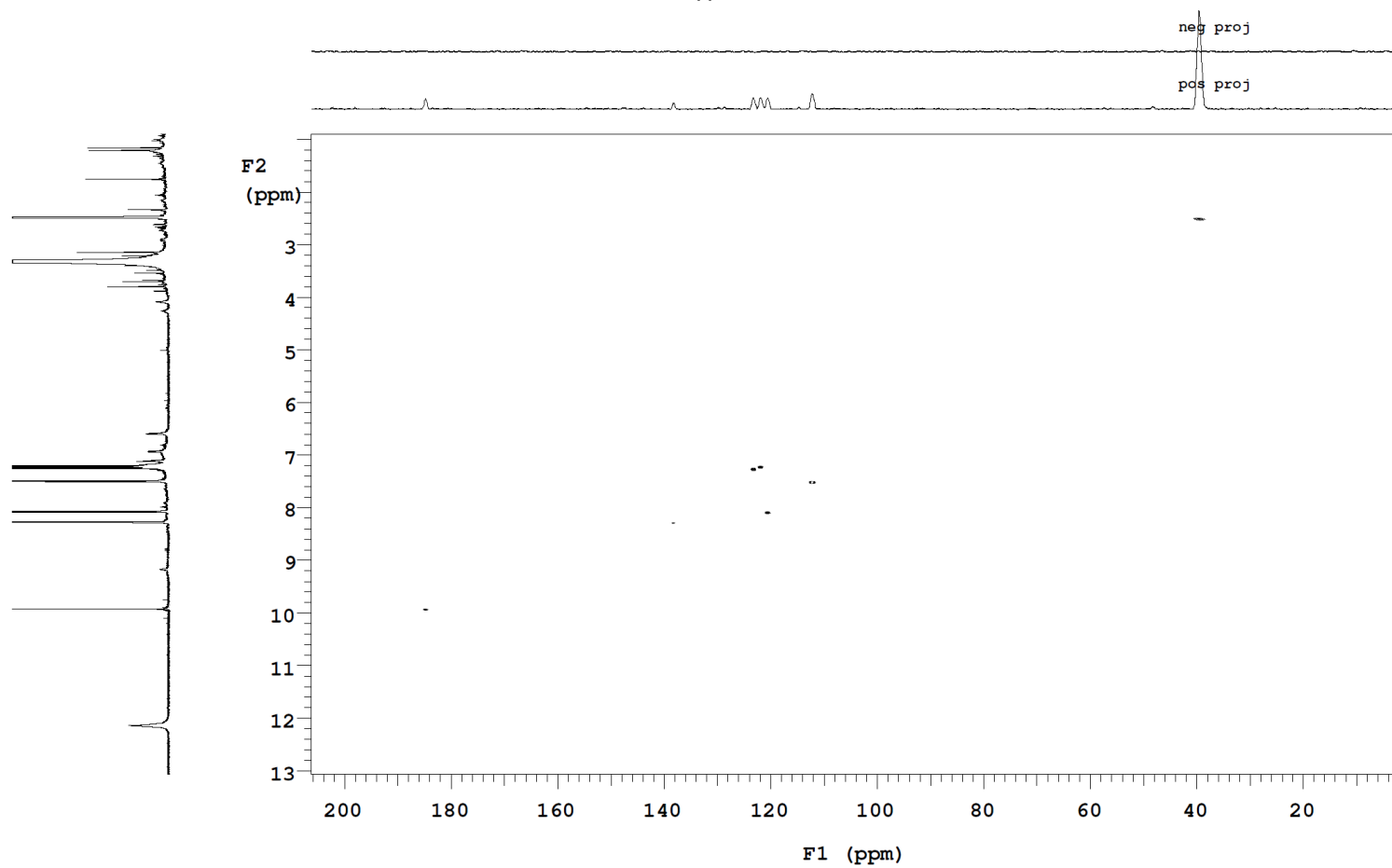
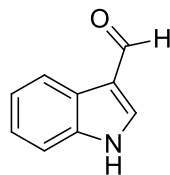


Figure S33: HSQC spectrum (DMSO- d_6 , 500 MHz) of Indole-3-carbaldehyde (5)

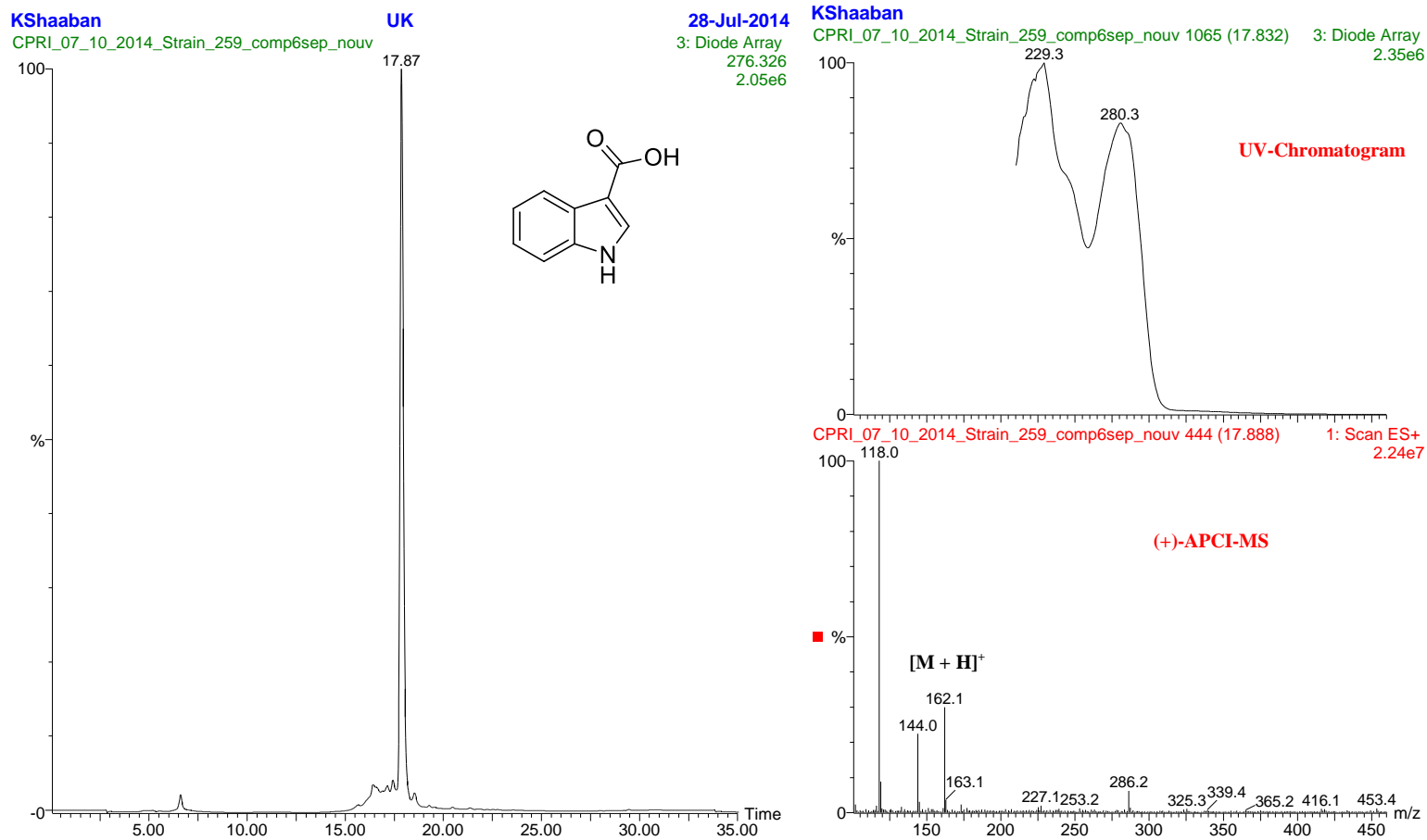


Figure S34: HPLC/UV/APCI-MS analyses of Indole-3-carboxylic acid (**6**). HPLC-conditions: Detection wavelength 270 nm; solvent A: H₂O/0.1% Formic acid; solvent B: acetonitrile; flow rate: 0.5 mL min⁻¹; 0-4 min, 90% A; 4-22 min, 90-0% A (linear gradient); 22-27 min 0% A; 27-35 min 0-90% A (linear gradient).

Sample: khaled_A_Shaaban

File: xp

Pulse Sequence: s2pul

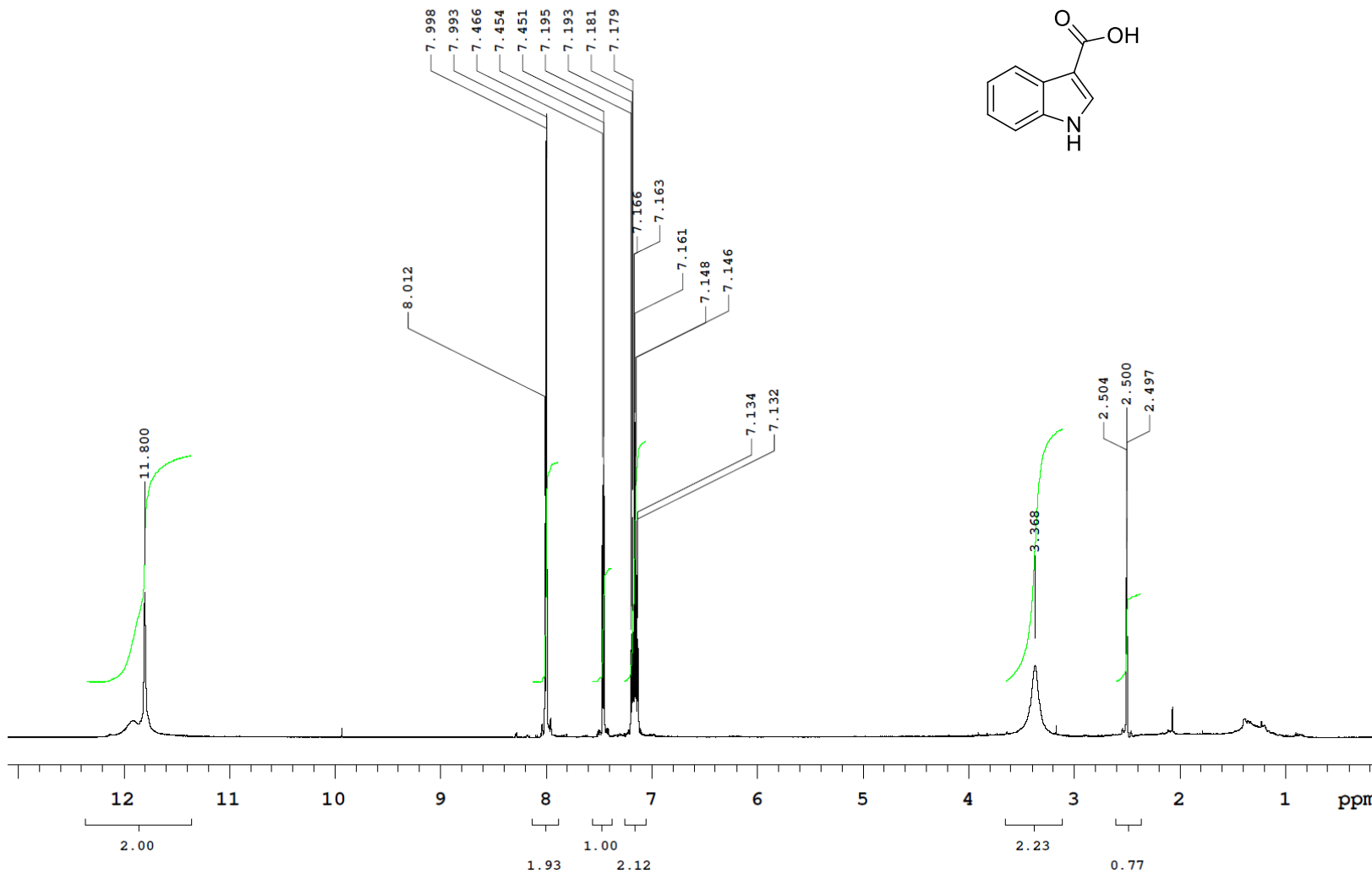


Figure S35: ^1H NMR spectrum ($\text{DMSO-}d_6$, 500 MHz) of Indole-3-carboxylic acid (6)

Sample: khaled_A_Shaaban

File: xp

Pulse Sequence: s2pul

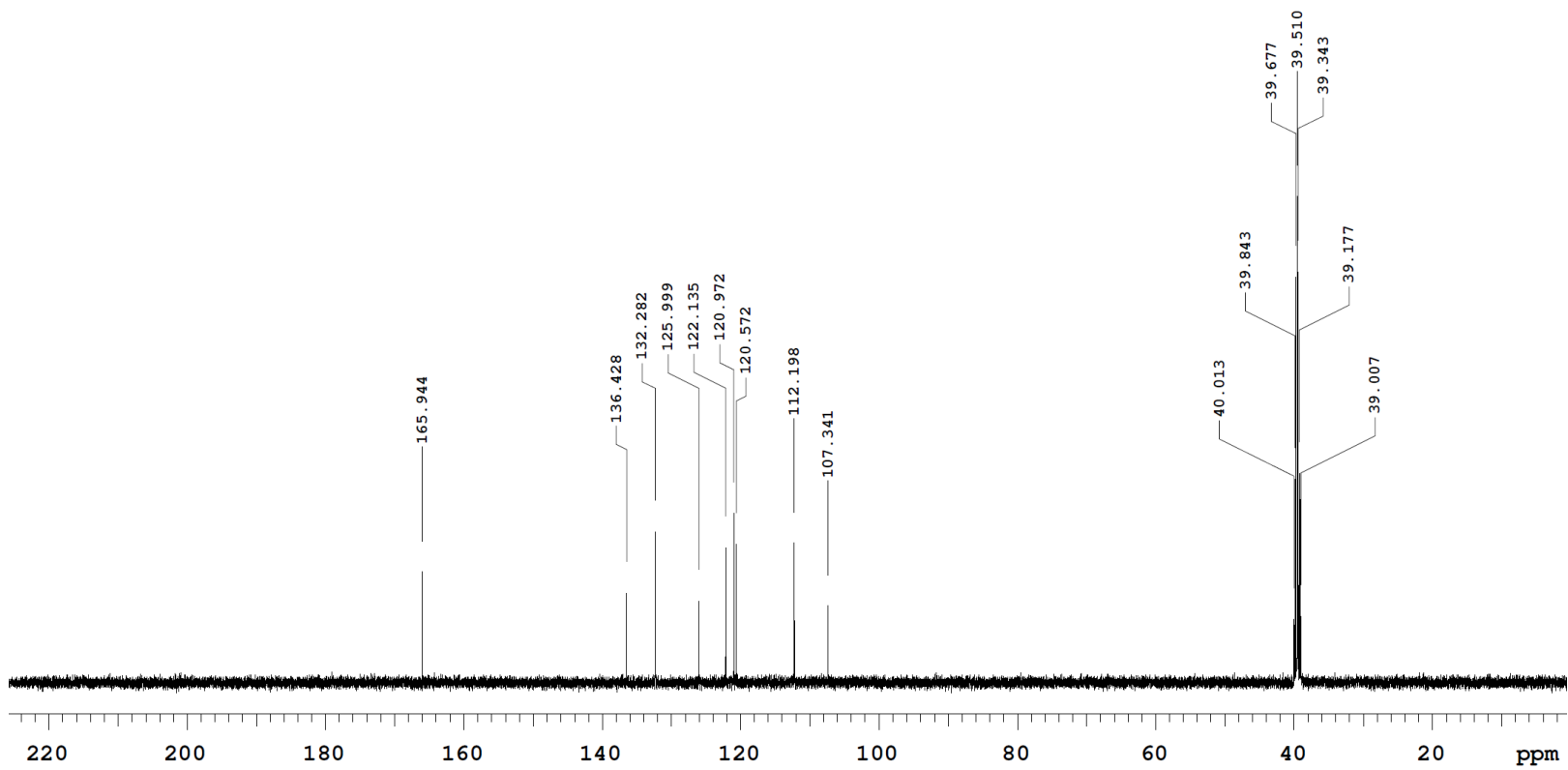
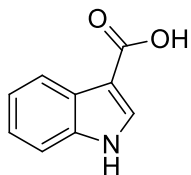


Figure S36: ¹³C NMR spectrum (DMSO-*d*₆, 125 MHz) of Indole-3-carboxylic acid (**6**)

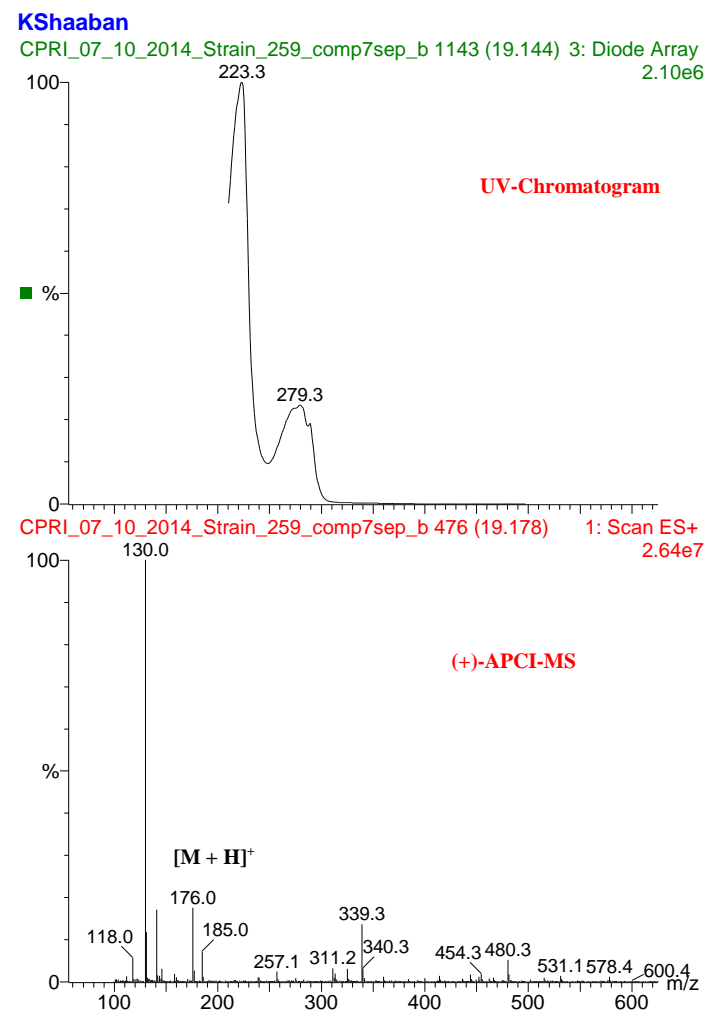
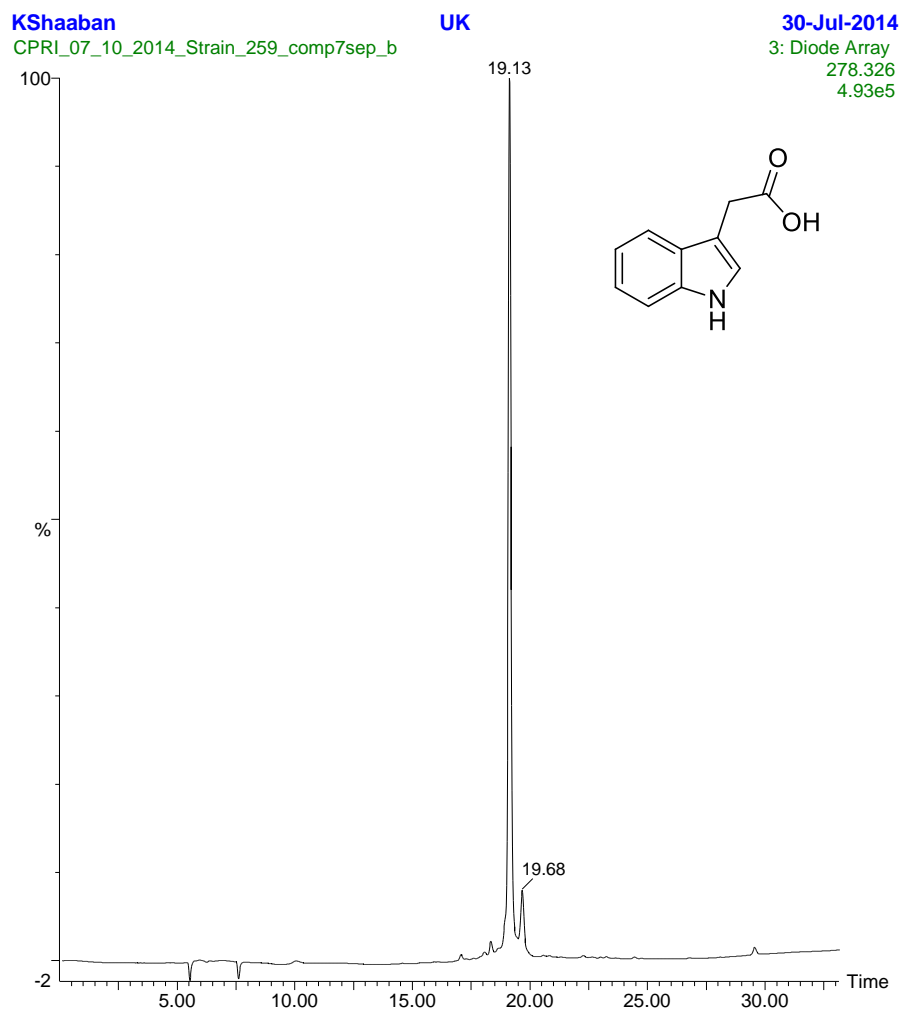


Figure S37: HPLC/UV/APCI-MS analyses of Indole-3-acetic acid (**7**). HPLC-conditions: Detection wavelength 270 nm; solvent A: H₂O/0.1% Formic acid; solvent B: acetonitrile; flow rate: 0.5 mL min⁻¹; 0-4 min, 90% A; 4-22 min, 90-0% A (linear gradient); 22-27 min 0% A; 27-35 min 0-90% A (linear gradient).

Sample: khaled_A_Shaaban
File: xp
Pulse Sequence: s2pul

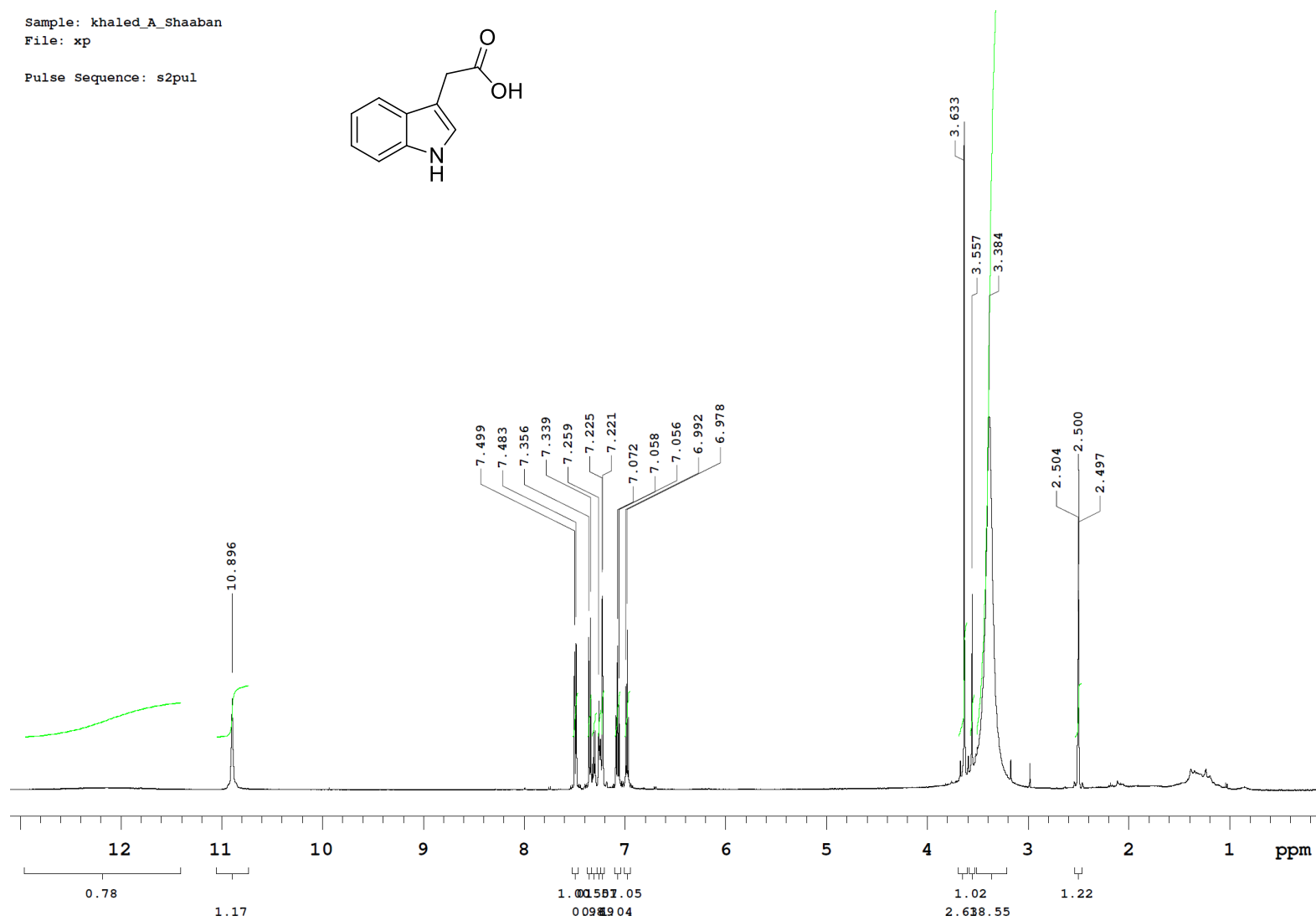
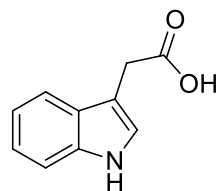


Figure S38: ¹H NMR spectrum (DMSO-*d*₆, 500 MHz) of Indole-3-acetic acid (7)

Sample: khaled_A_Shaaban
File: xp

Pulse Sequence: s2pul

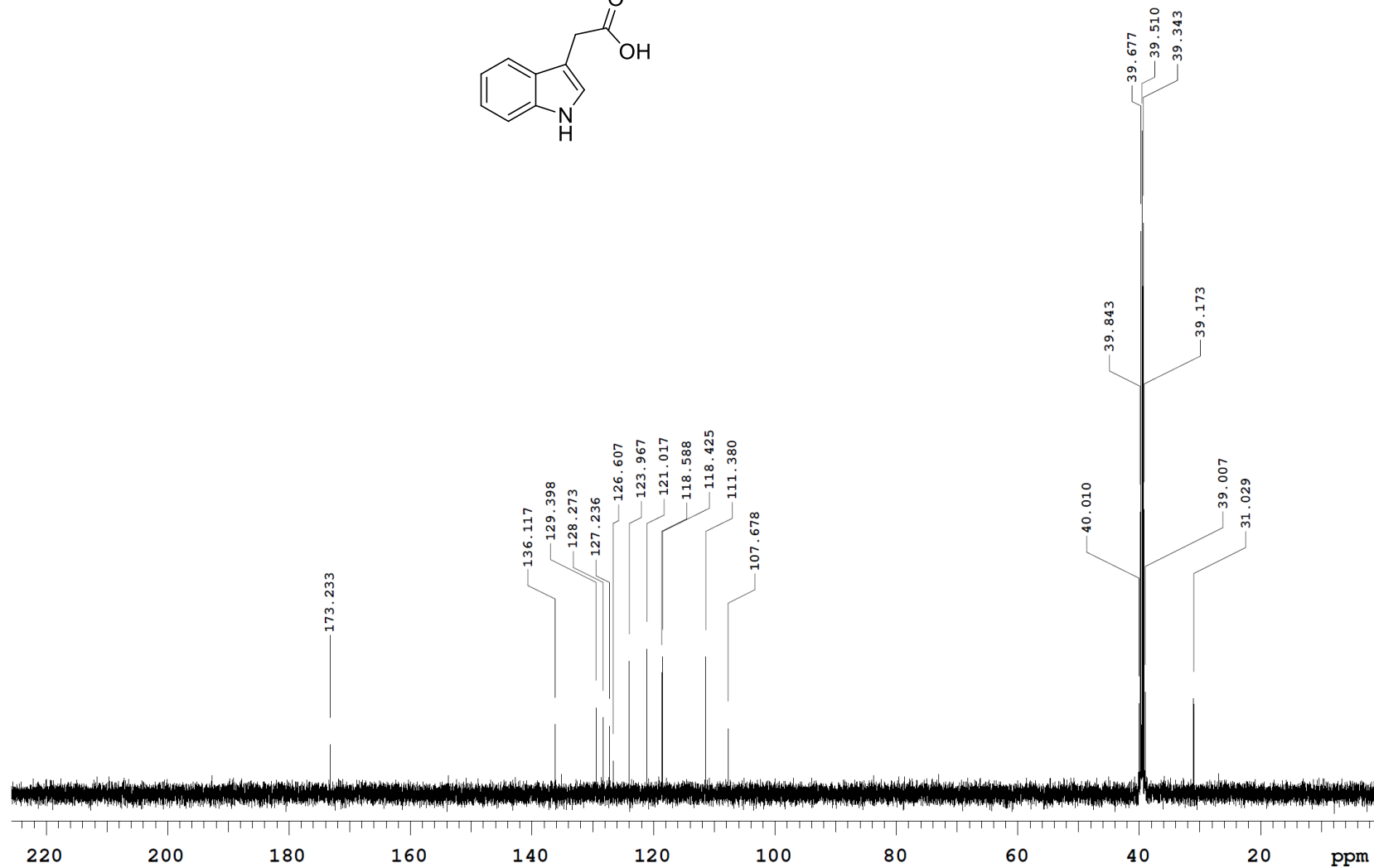
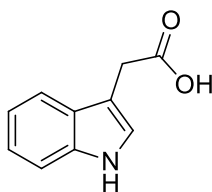


Figure S39: ¹³C NMR spectrum (DMSO-*d*₆, 125 MHz) of Indole-3-acetic acid (7)