

## **Bioactive Limonoids from the Leaves of *Azadirachta indica* (Neem)\***

Maria J. Gualtieri,<sup>†, ‡</sup> Nicola Malafronte,<sup>‡</sup> Antonio Vassallo,<sup>§</sup> Alessandra Braca,<sup>⊥</sup>  
Roberta Cotugno,<sup>‡</sup> Michele Vasaturo,<sup>‡</sup> Nunziatina De Tommasi,<sup>\*,‡</sup> and Fabrizio Dal  
Piaz<sup>‡</sup>

<sup>†</sup>Departamento de Farmacognosia y Medicamentos Organicos, Laboratorio de  
Medicamentos Organicos Sector Campo de Oro, detras del HULA Merida, 5101,  
Venezuela

<sup>‡</sup>Dipartimento di Farmacia, Università degli Studi di Salerno, via Ponte Don Melillo,  
84084 Fisciano (SA), Italy

<sup>§</sup>Dipartimento di Scienze, Università degli Studi della Basilicata, viale dell'Ateneo  
Lucano 10, 85100 Potenza, Italy

<sup>⊥</sup>Dipartimento di Farmacia, Università di Pisa, via Bonanno 33, 56126 Pisa, Italy

\*Dedicated to Prof. Dr. Otto Sticher of ETH-Zurich, Zurich, Switzerland, for his  
pioneering work in pharmacognosy and phytochemistry

**Figure S1.**  $^1\text{H}$  NMR spectrum of compound **1** ( $\text{CD}_3\text{OD}$ )

**Figure S2.** HSQC spectrum of compound **1** ( $\text{CD}_3\text{OD}$ )

**Figure S3.** HMBC spectrum of compound **1** ( $\text{CD}_3\text{OD}$ )

**Figure S4.** MS spectrum of compound **1** ( $\text{CD}_3\text{OD}$ )

**Figure S5.**  $^1\text{H}$  NMR spectrum of compound **2** ( $\text{CD}_3\text{OD}$ )

**Figure S6.** HSQC spectrum of compound **2** ( $\text{CD}_3\text{OD}$ )

**Figure S7.** HMBC spectrum of compound **2** ( $\text{CD}_3\text{OD}$ )

**Figure S8.**  $^1\text{H}$  NMR spectrum of compound **3** ( $\text{CD}_3\text{OD}$ )

**Figure S9.**  $^{13}\text{C}$  NMR spectrum of compound **3** ( $\text{CD}_3\text{OD}$ )

**Figure S10.** HMBC spectrum of compound **3** ( $\text{CD}_3\text{OD}$ )

**Figure S11.**  $^1\text{H}$  NMR spectrum of compound **4** ( $\text{CD}_3\text{OD}$ )

**Figure S12.** HSQC spectrum of compound **4** ( $\text{CD}_3\text{OD}$ )

**Figure S13.** HMBC spectrum of compound **4** ( $\text{CD}_3\text{OD}$ )

**Figure S14.**  $^1\text{H}$  NMR spectrum of compound **5** ( $\text{CD}_3\text{OD}$ )

**Figure S15.** HSQC spectrum of compound **5** ( $\text{CD}_3\text{OD}$ )

**Figure S16.** HMBC spectrum of compound **5** ( $\text{CD}_3\text{OD}$ )

**Figure S17.**  $^1\text{H}$  NMR spectrum of compound **6** ( $\text{CD}_3\text{OD}$ )

**Figure S18.** HSQC spectrum of compound **6** ( $\text{CD}_3\text{OD}$ )

**Figure S19.** HMBC spectrum of compound **6** ( $\text{CD}_3\text{OD}$ )

**Figure S20.**  $^1\text{H}$  NMR spectrum of compound **7** ( $\text{CD}_3\text{OD}$ )

**Figure S21.** HSQC spectrum of compound **7** ( $\text{CD}_3\text{OD}$ )

**Figure S22.** COSY spectrum of compound **7** ( $\text{CD}_3\text{OD}$ )

**Figure S23.** HMBC spectrum of compound **7** ( $\text{CD}_3\text{OD}$ )

**Figure S24.**  $^1\text{H}$  NMR spectrum of compound **8** ( $\text{CD}_3\text{OD}$ )

**Figure S25.** HSQC spectrum of compound **8** ( $\text{CD}_3\text{OD}$ )

**Figure S26.** HMBC spectrum of compound **8** ( $\text{CD}_3\text{OD}$ )

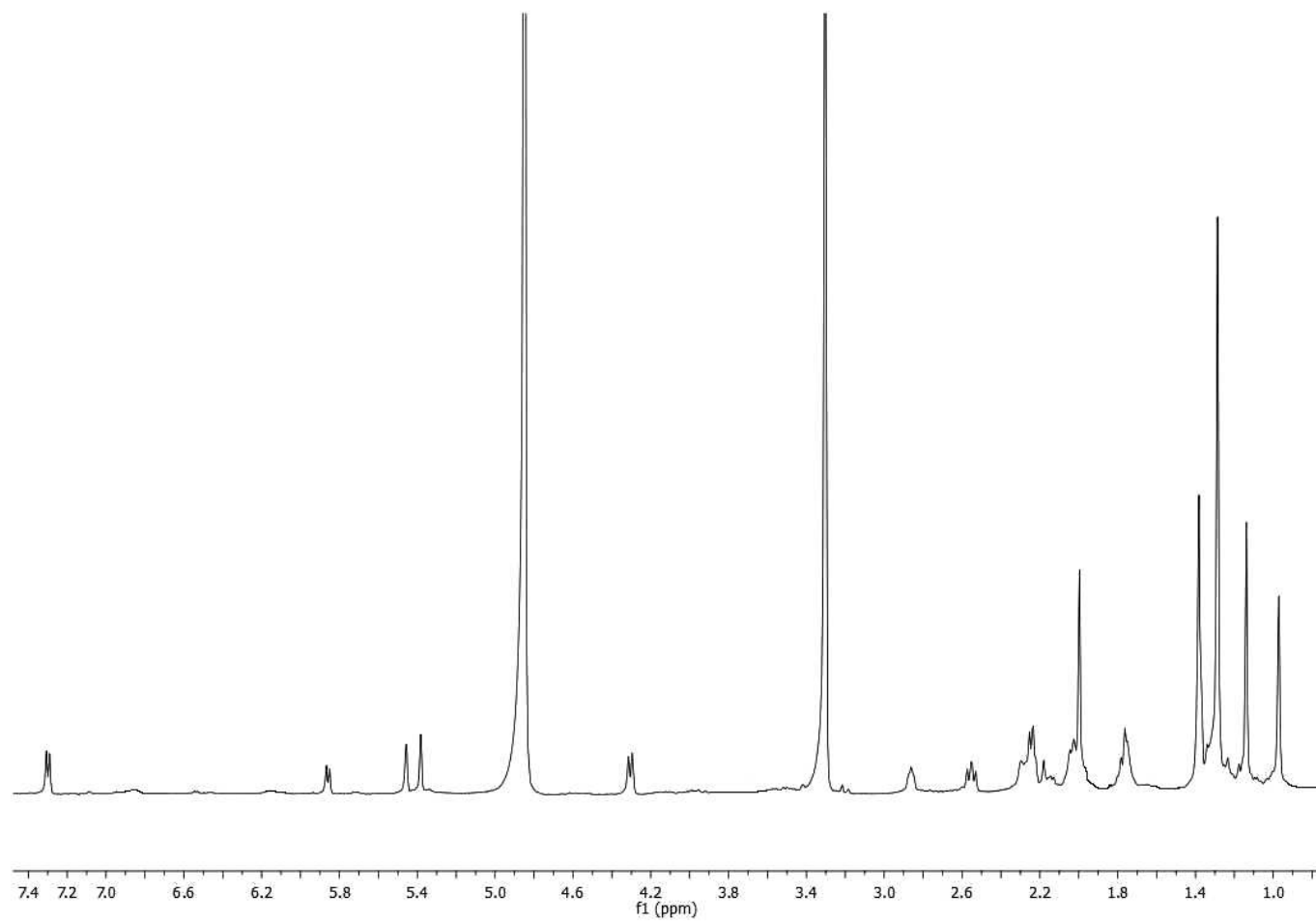
**Figure S27.**  $^1\text{H}$  NMR spectrum of compound **9** ( $\text{CD}_3\text{OD}$ )

**Figure S28.** 1D-TOCSY spectrum of compound **9** ( $\text{CD}_3\text{OD}$ )

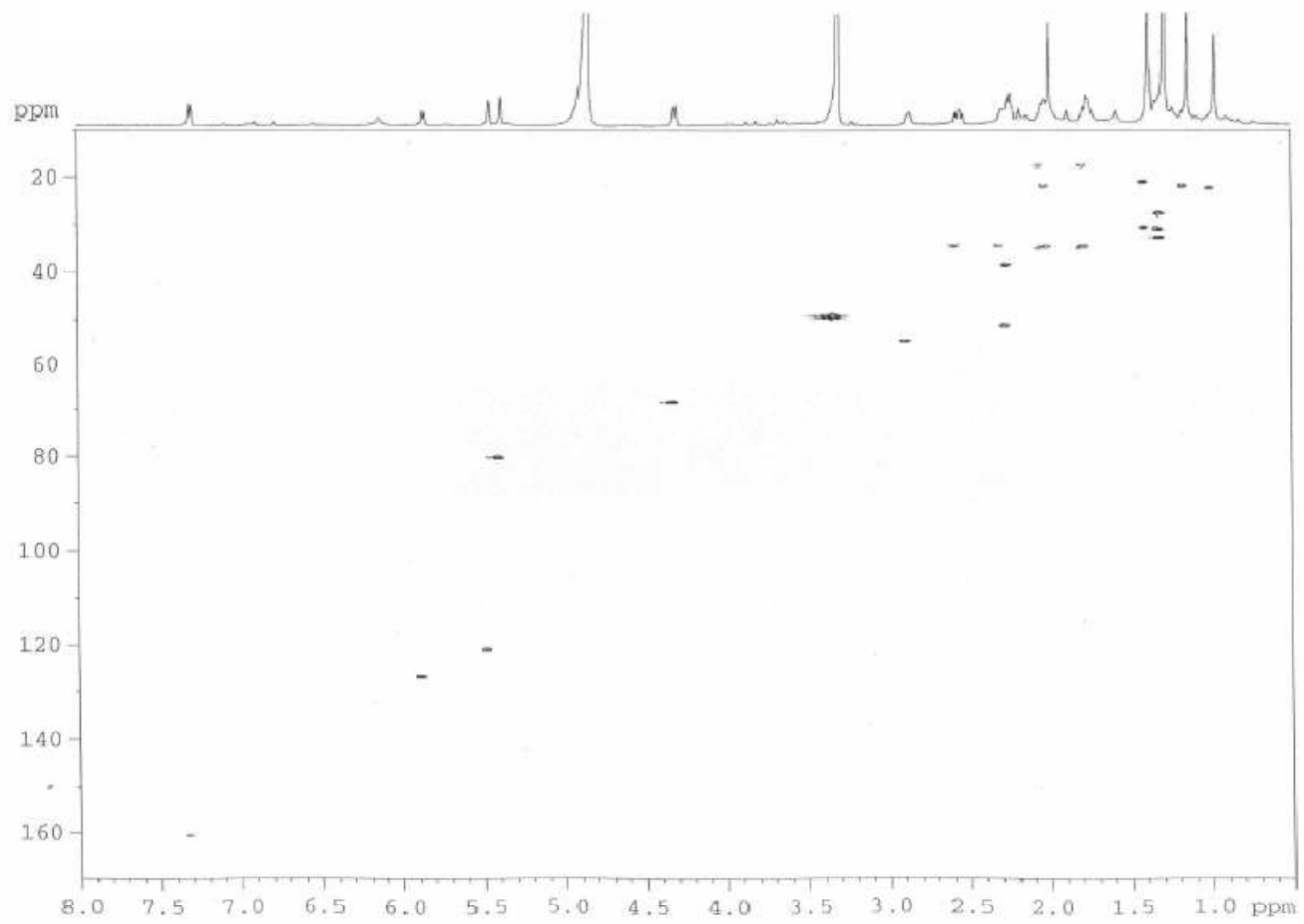
**Figure S29.** HSQC spectrum of compound **9** ( $\text{CD}_3\text{OD}$ )

**Figure S30.** HMBC spectrum of compound **9** ( $\text{CD}_3\text{OD}$ )

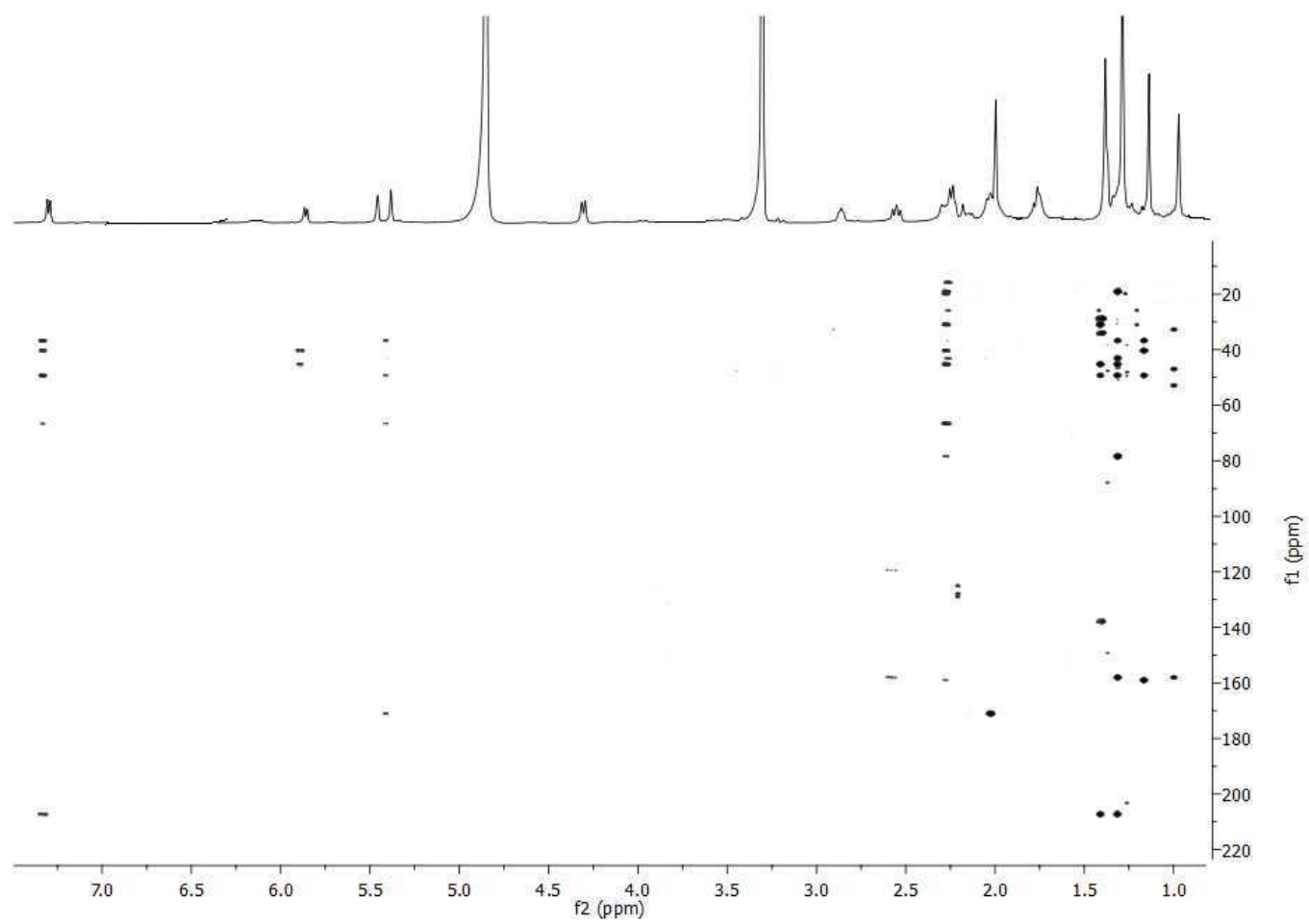
**Figure S1.**  $^1\text{H}$  NMR spectrum of compound **1** ( $\text{CD}_3\text{OD}$ )



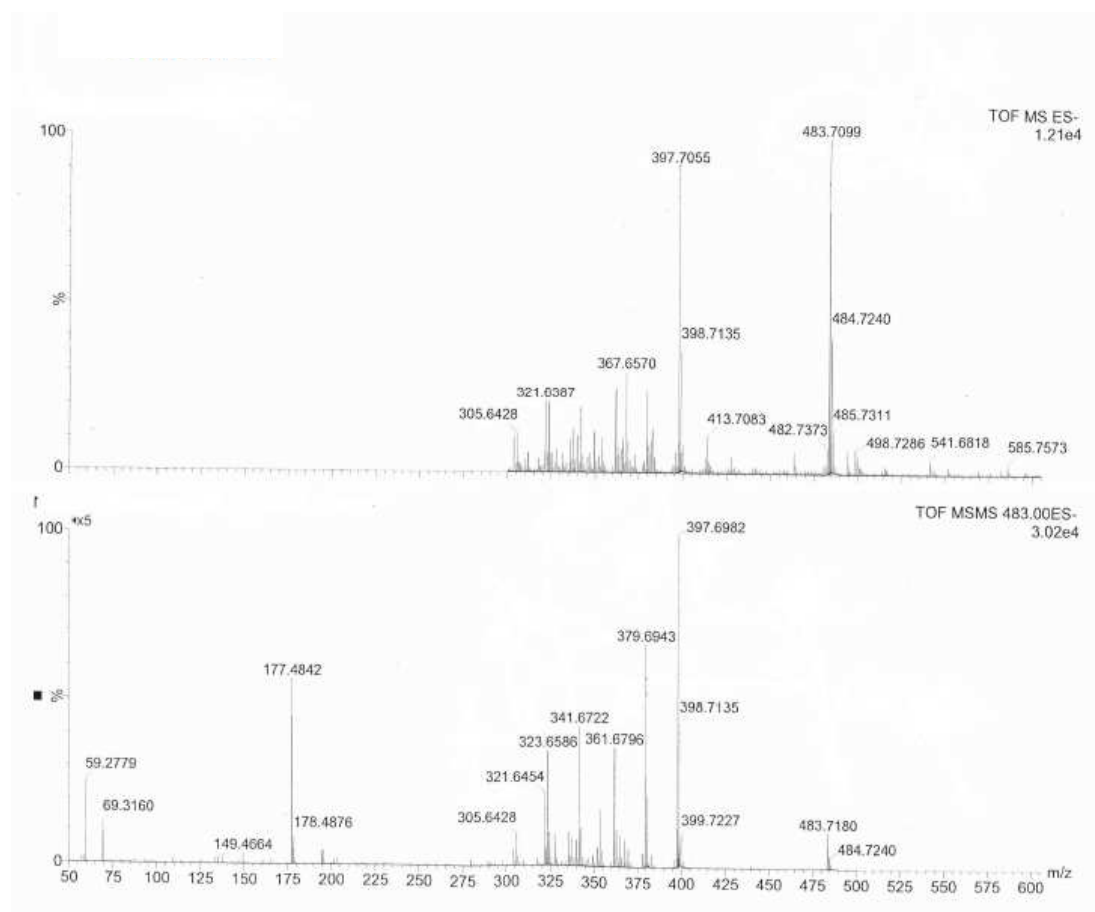
**Figure S2.** HSQC spectrum of compound **1** (CD<sub>3</sub>OD)



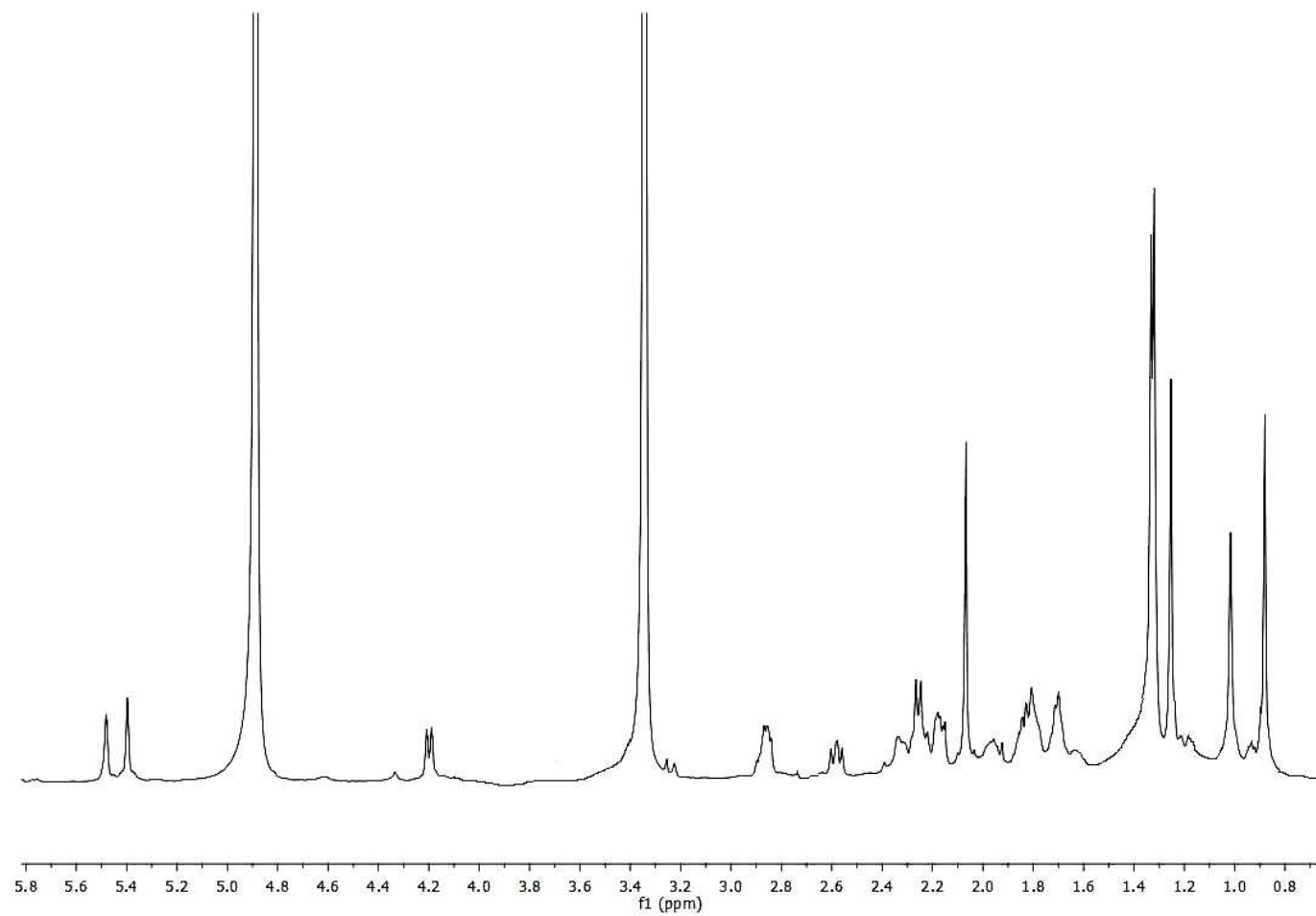
**Figure S3.** HMBC spectrum of compound **1** (CD<sub>3</sub>OD)



**Figure S4.** MS spectrum of compound **1**

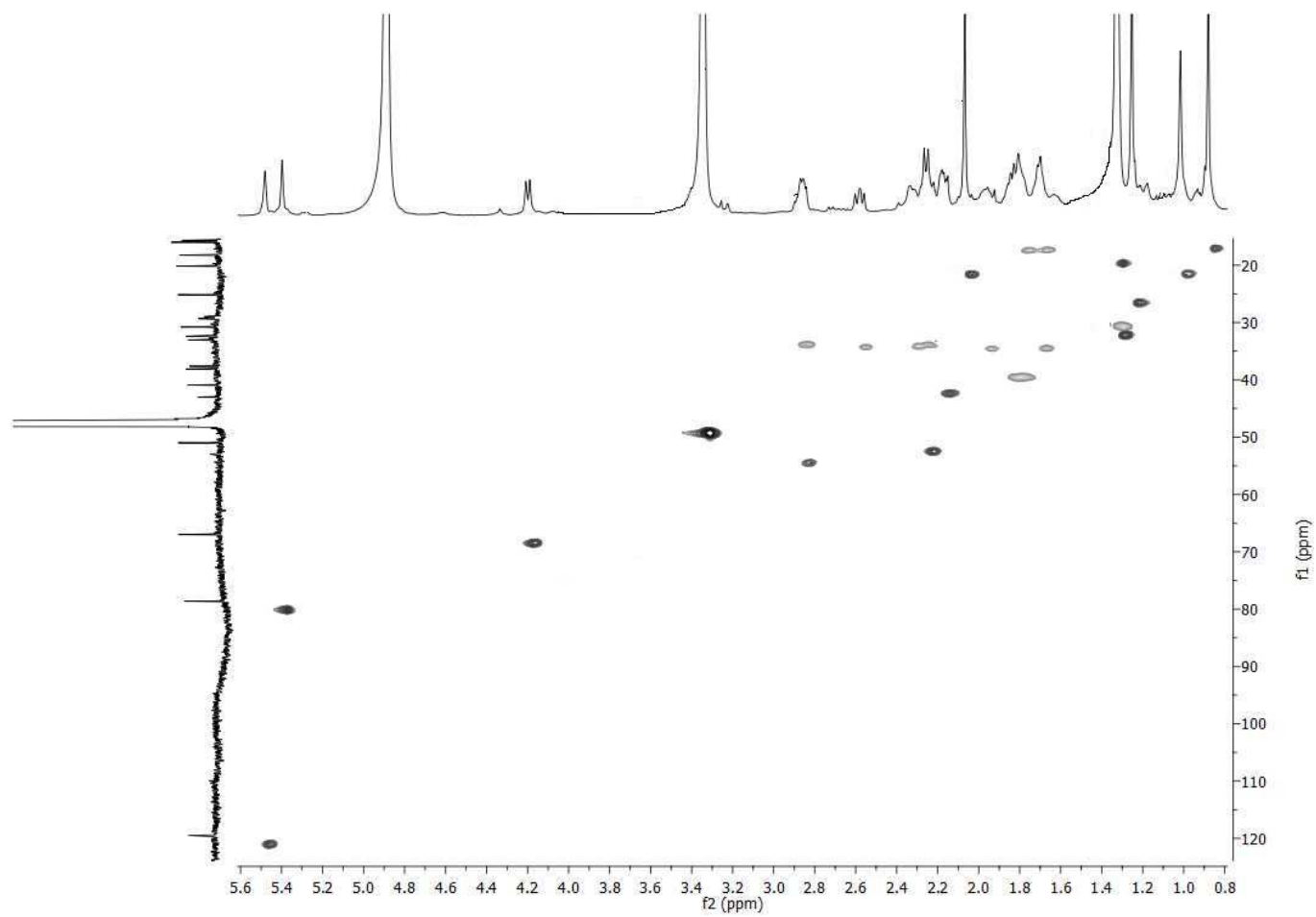


**Figure S5.**  $^1\text{H}$  NMR spectrum of compound **2** ( $\text{CD}_3\text{OD}$ )

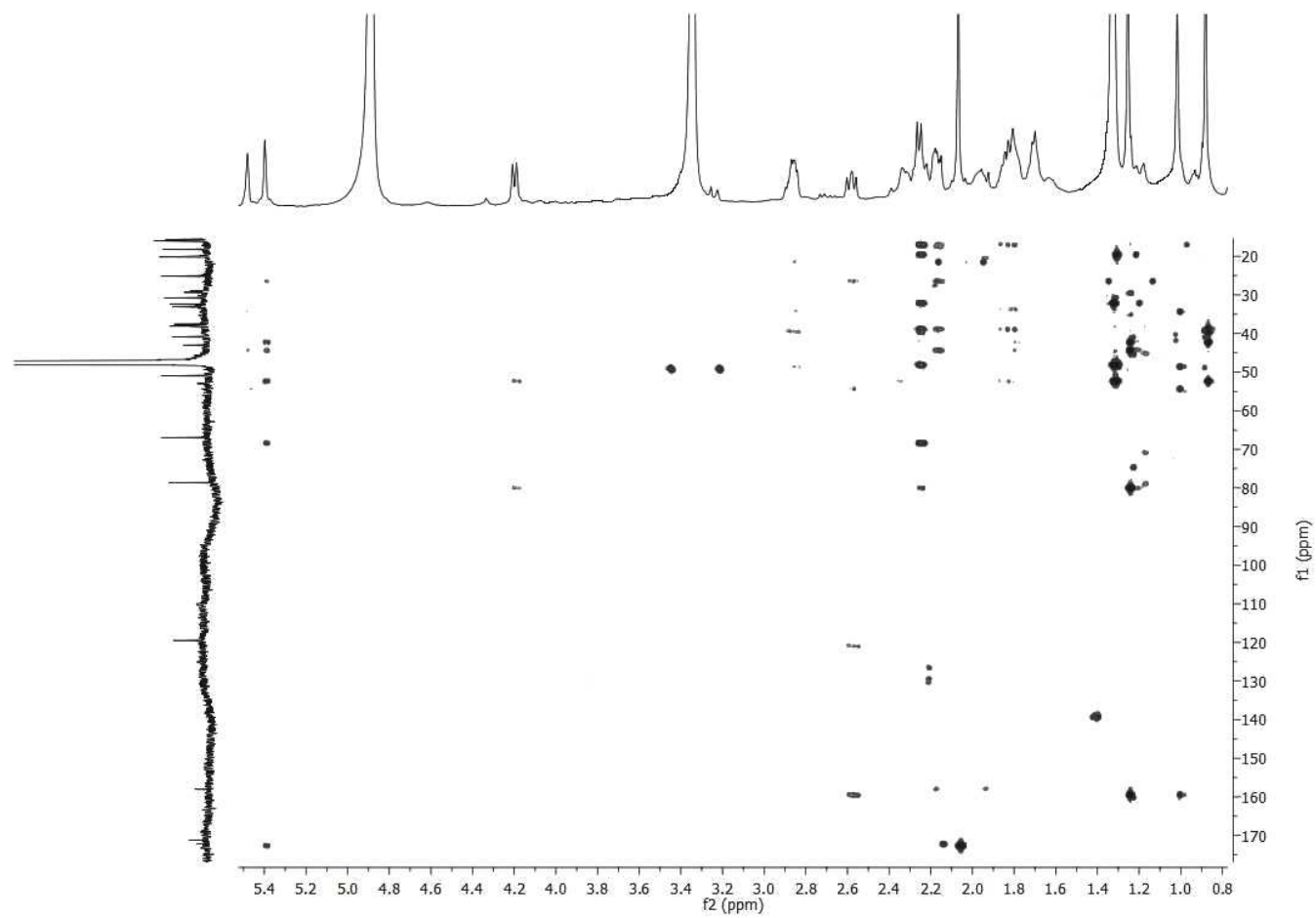




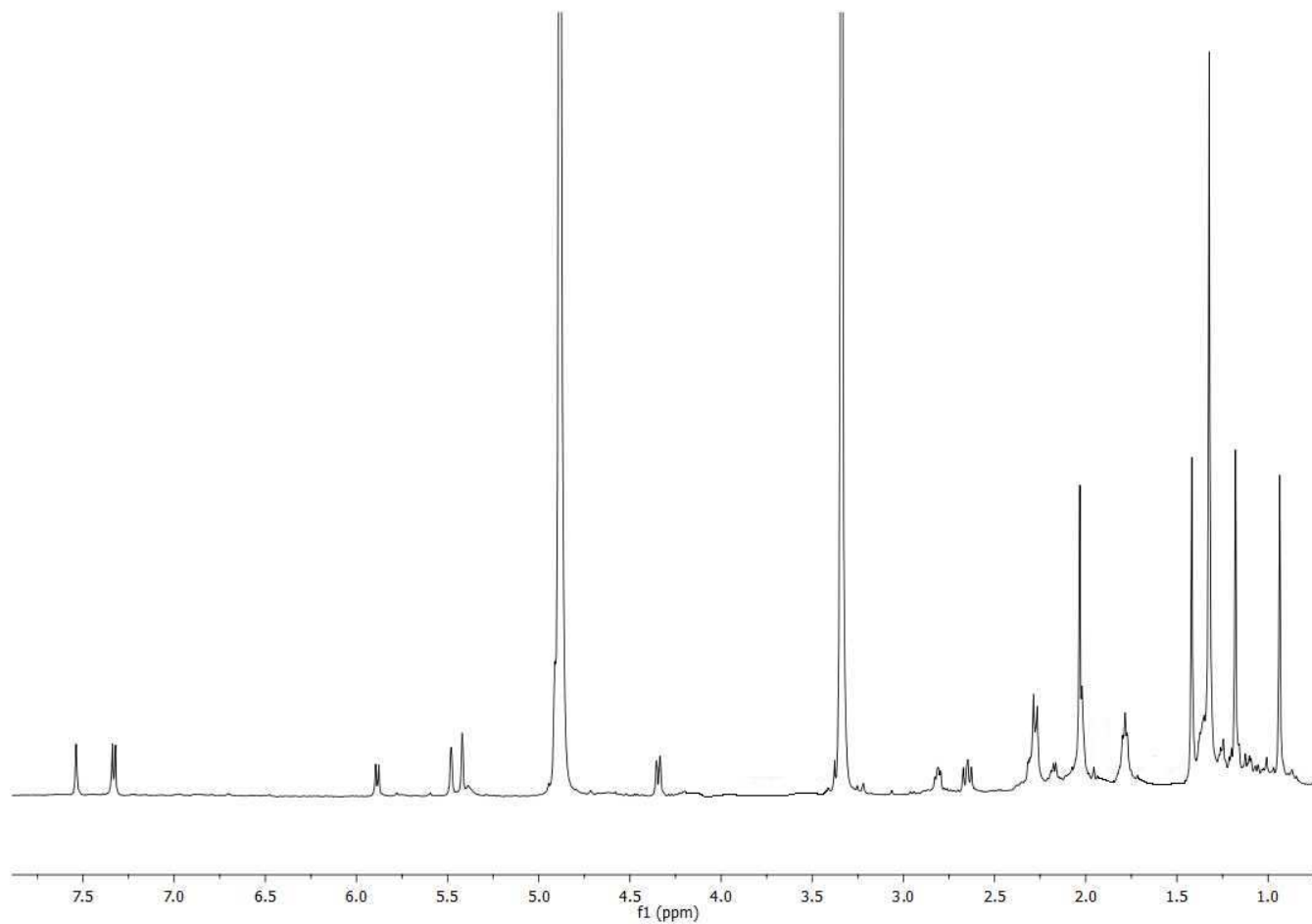
**Figure S6.** HSQC spectrum of compound **2** (CD<sub>3</sub>OD)



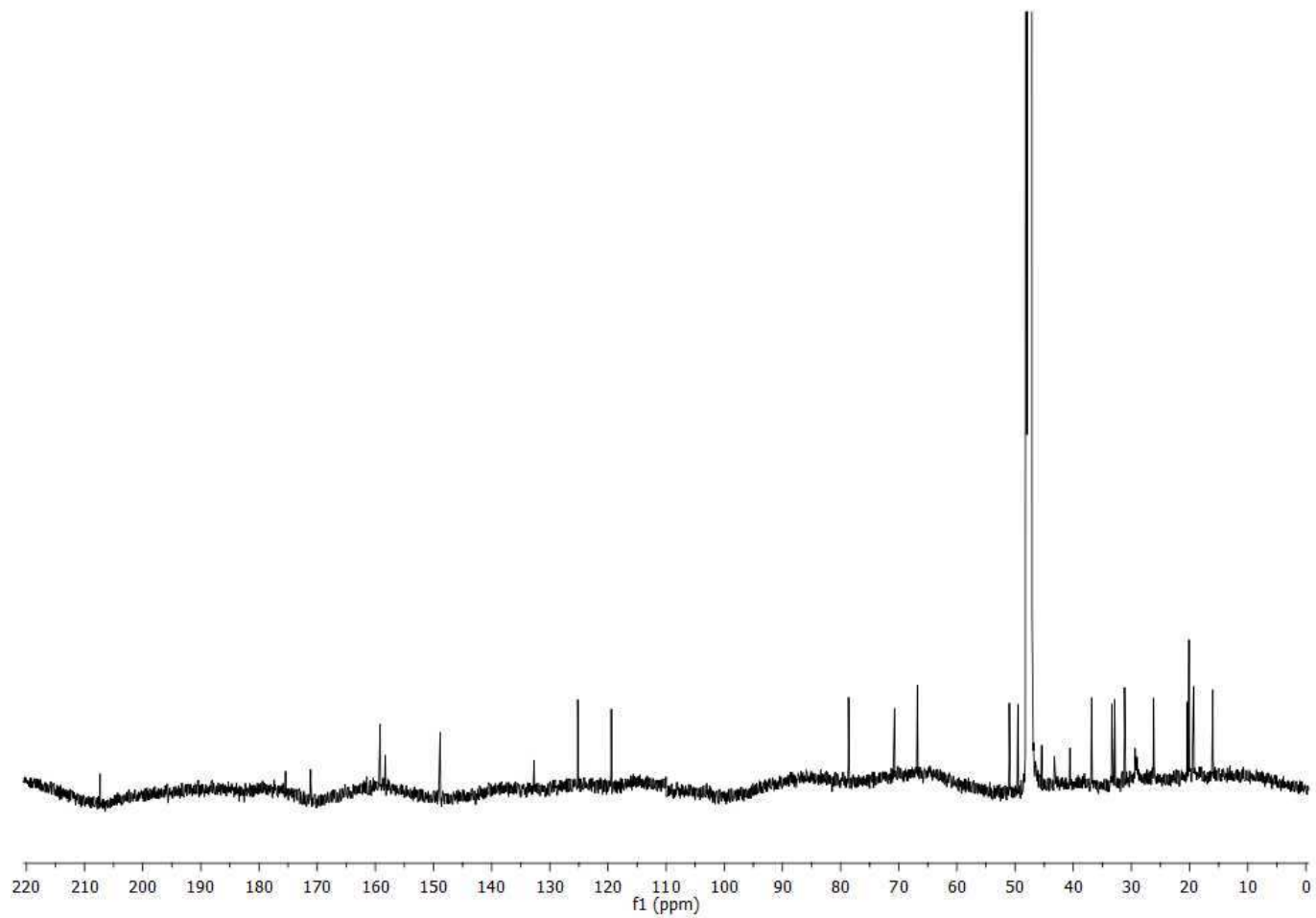
**Figure S7.** HMBC spectrum of compound **2** (CD<sub>3</sub>OD)



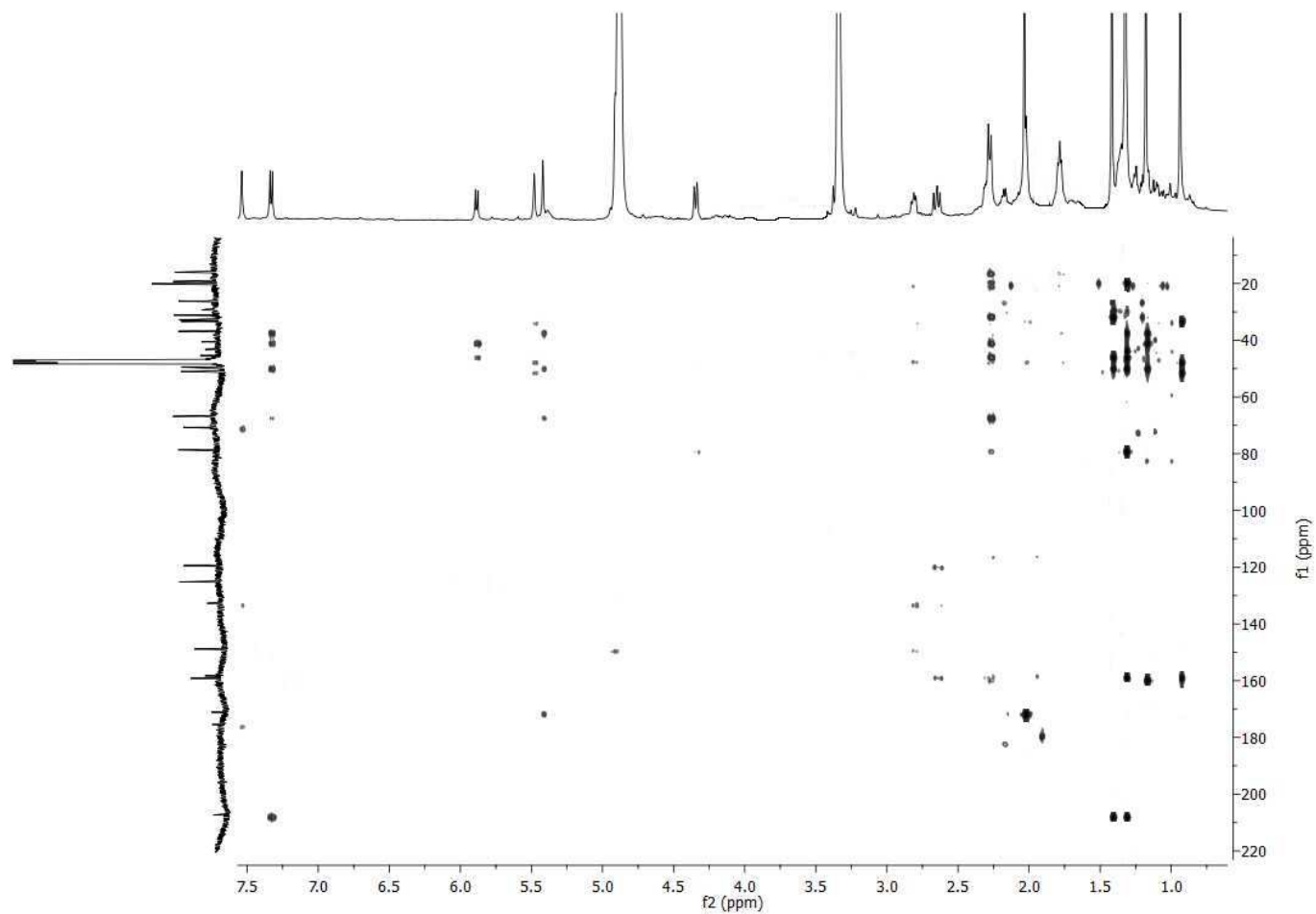
**Figure S8.**  $^1\text{H}$  NMR spectrum of compound **3** ( $\text{CD}_3\text{OD}$ )



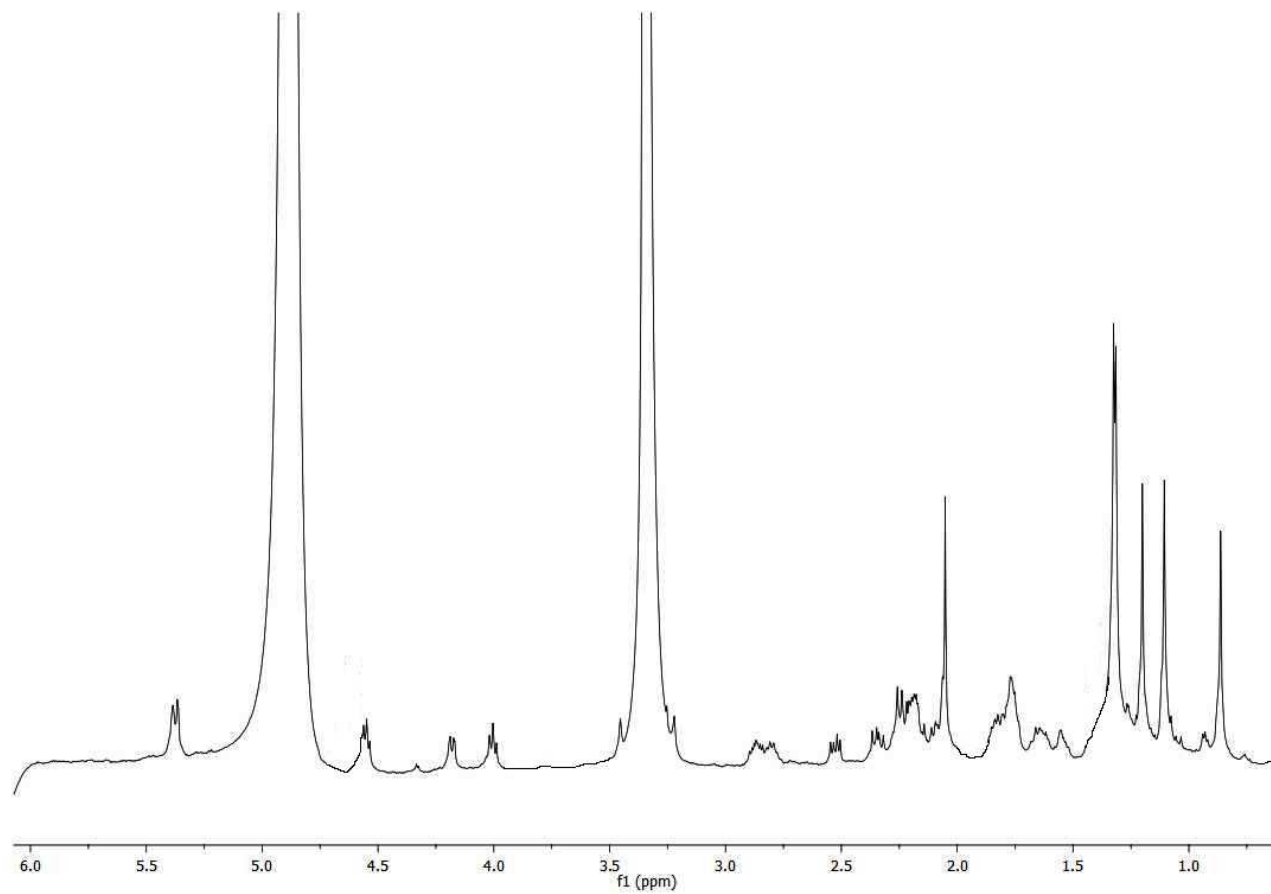
**Figure S9.**  $^{13}\text{C}$  NMR spectrum of compound **3** ( $\text{CD}_3\text{OD}$ )



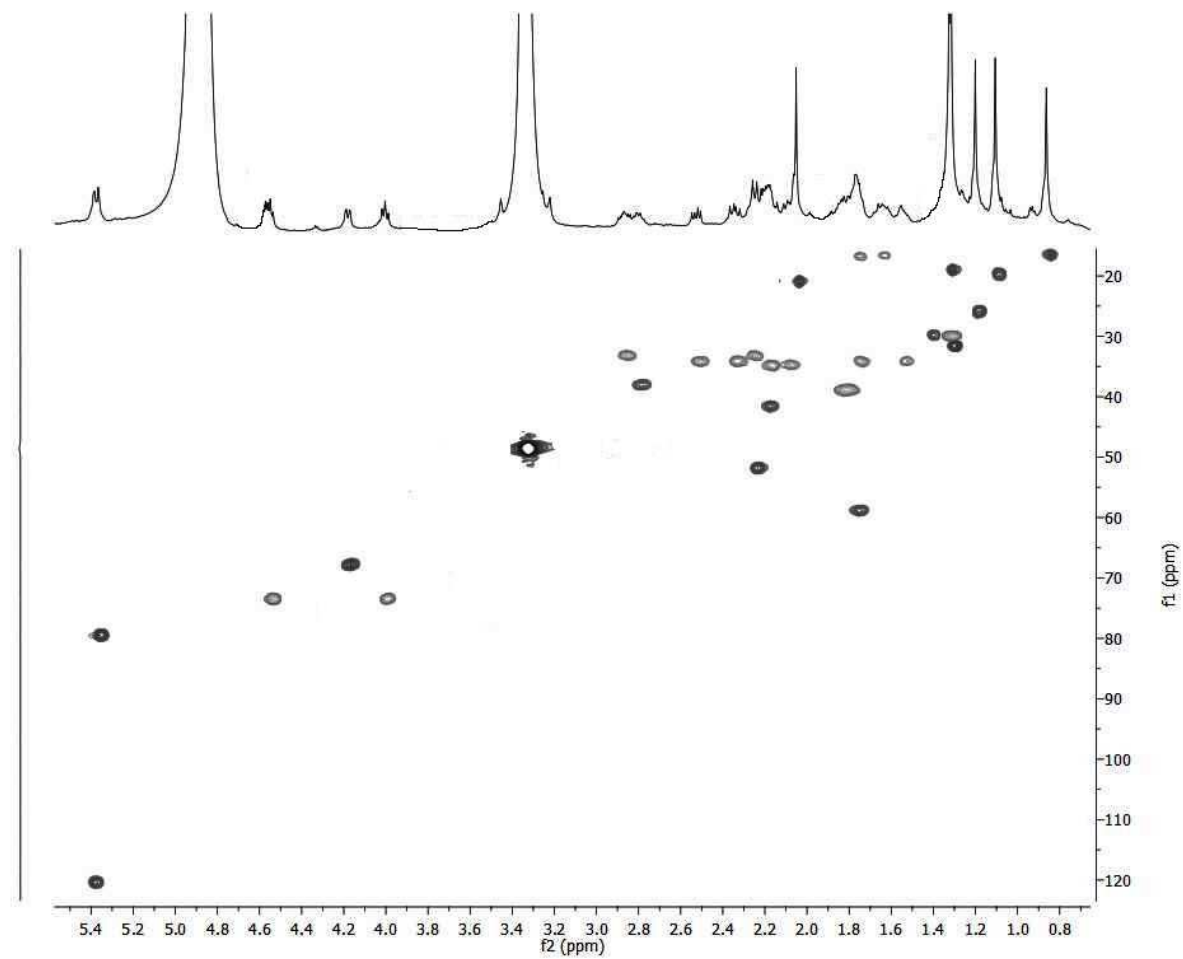
**Figure S10.** HMBC spectrum of compound **3** (CD<sub>3</sub>OD)



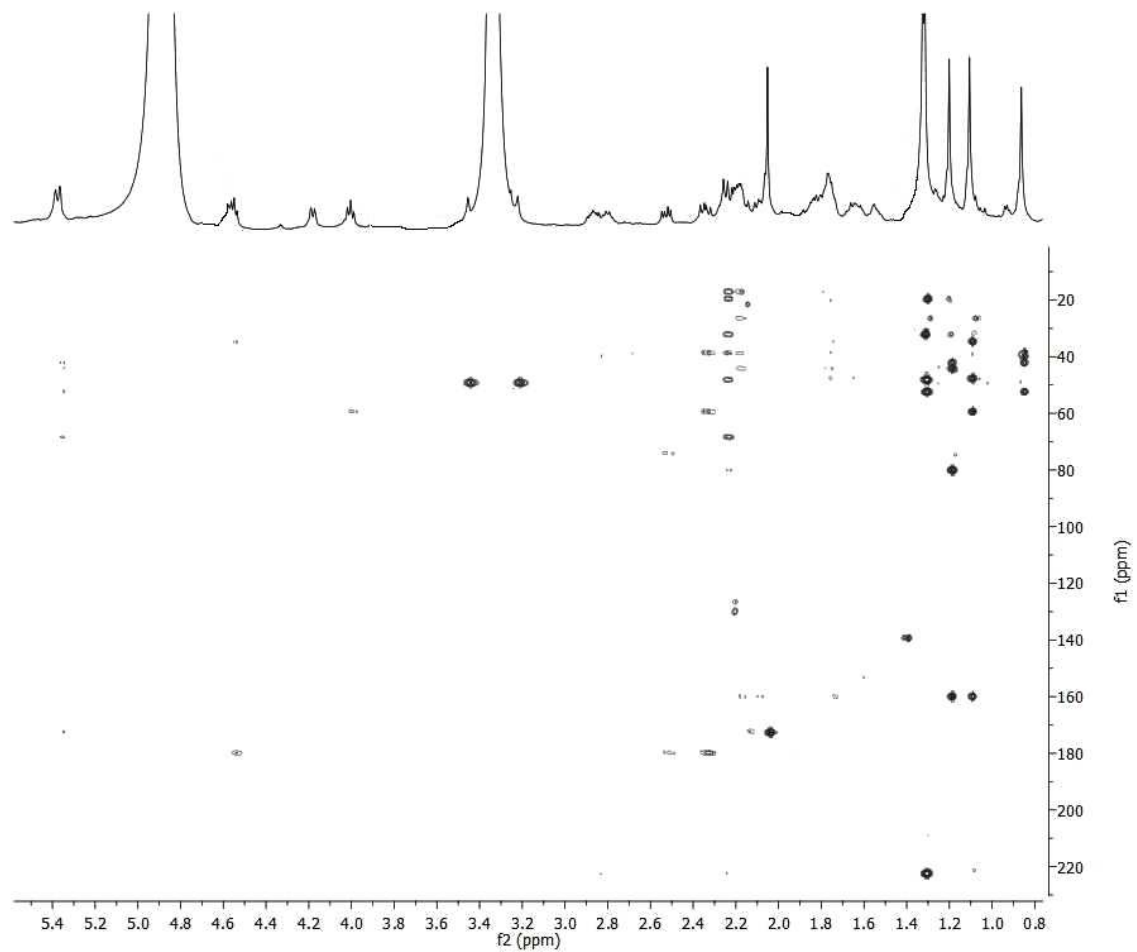
**Figure S11.**  $^1\text{H}$  NMR spectrum of compound **4** ( $\text{CD}_3\text{OD}$ )



**Figure S12.** HSQC spectrum of compound **4** (CD<sub>3</sub>OD)

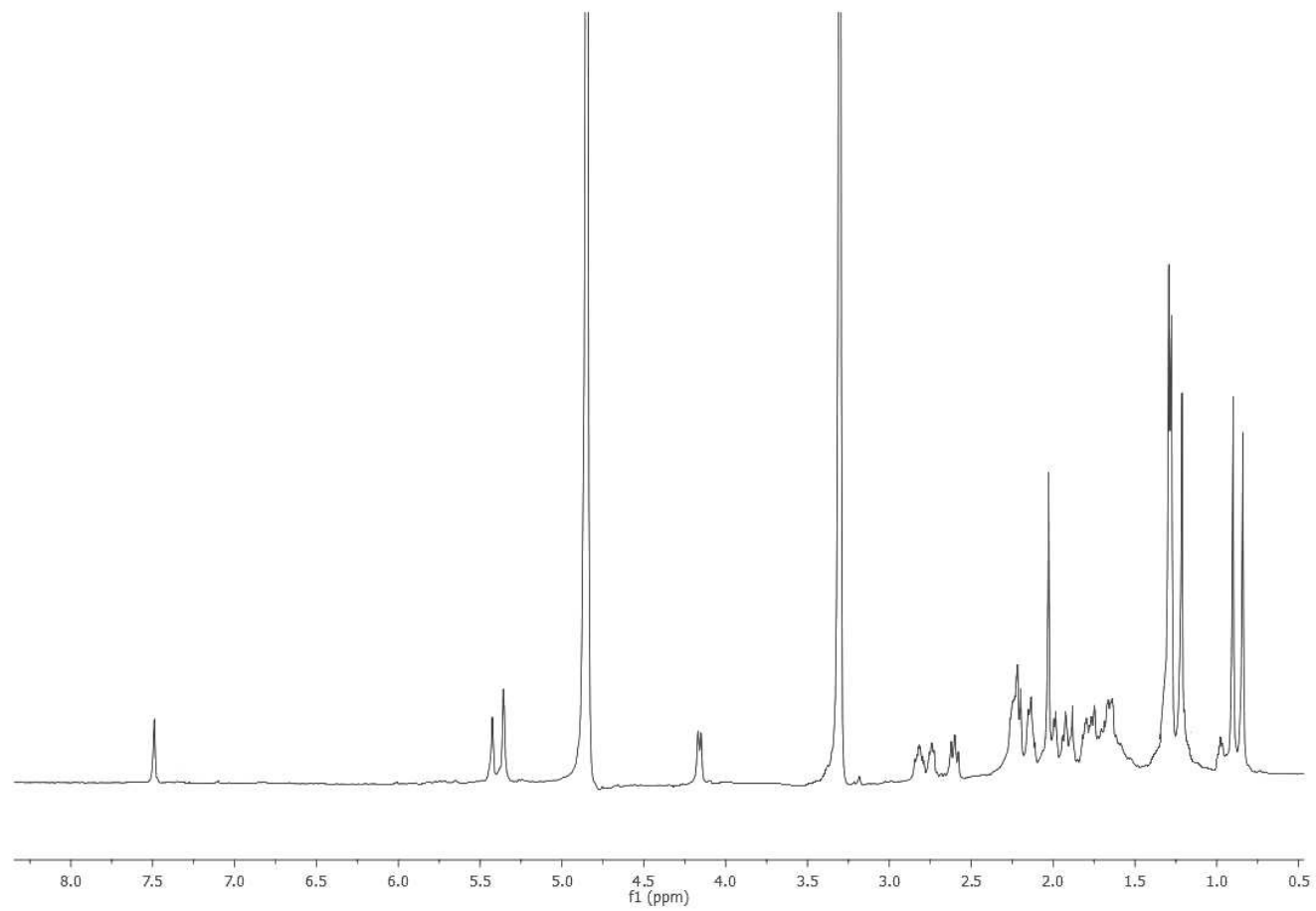


**Figure S13.** HMBC spectrum of compound **4** (CD<sub>3</sub>OD)

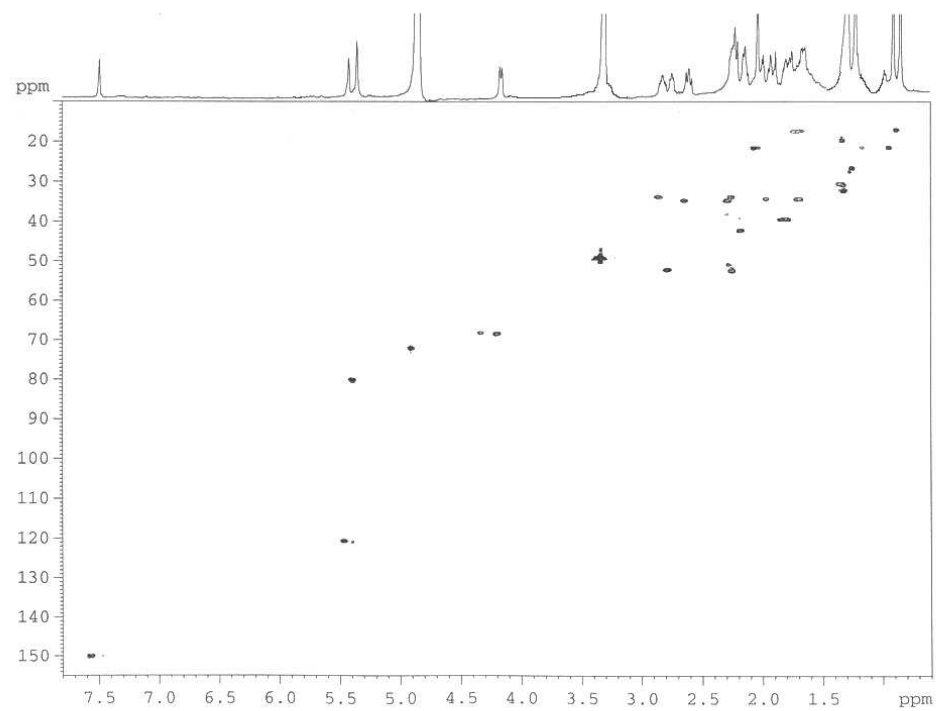




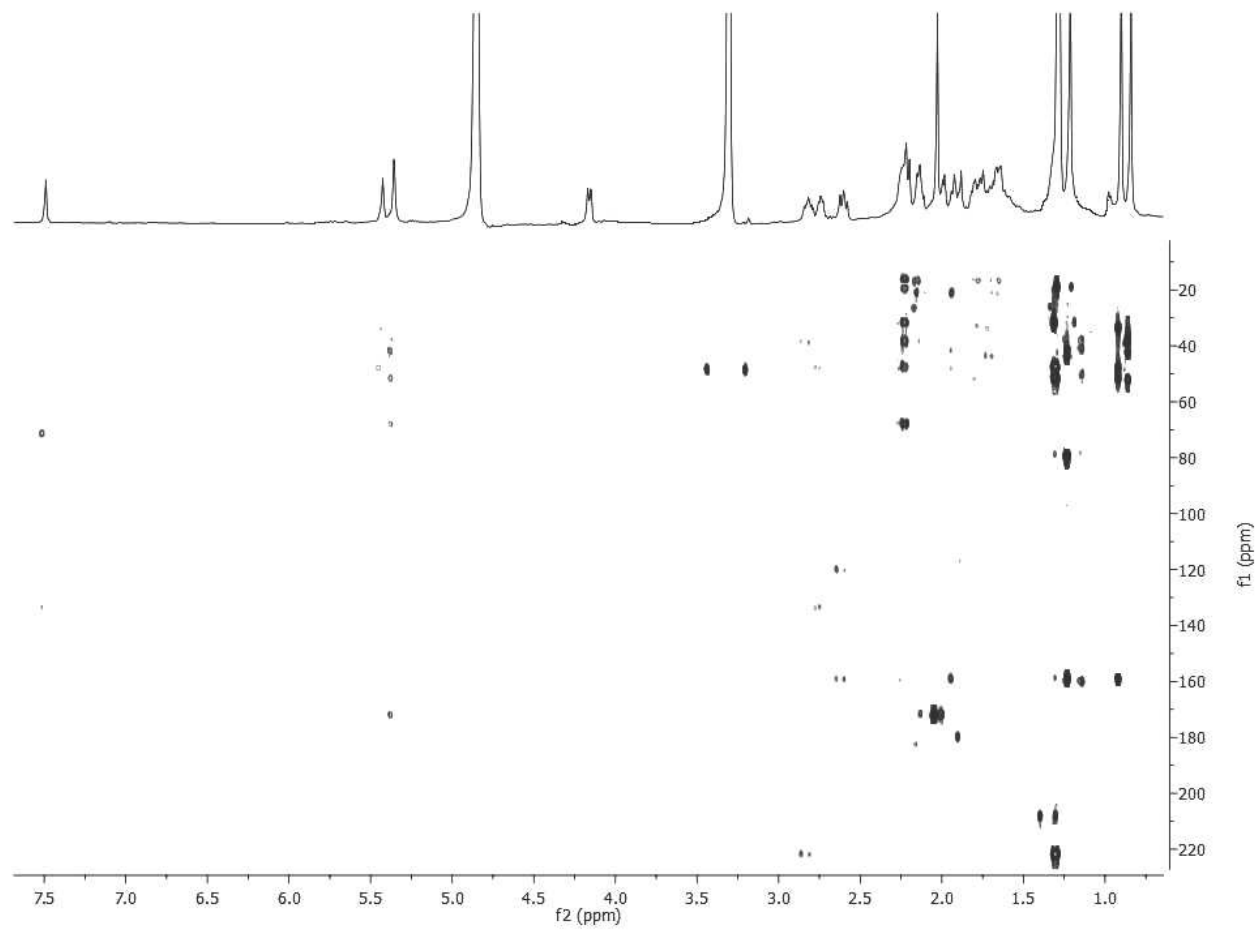
**Figure S14.**  $^1\text{H}$  NMR spectrum of compound **5** ( $\text{CD}_3\text{OD}$ )



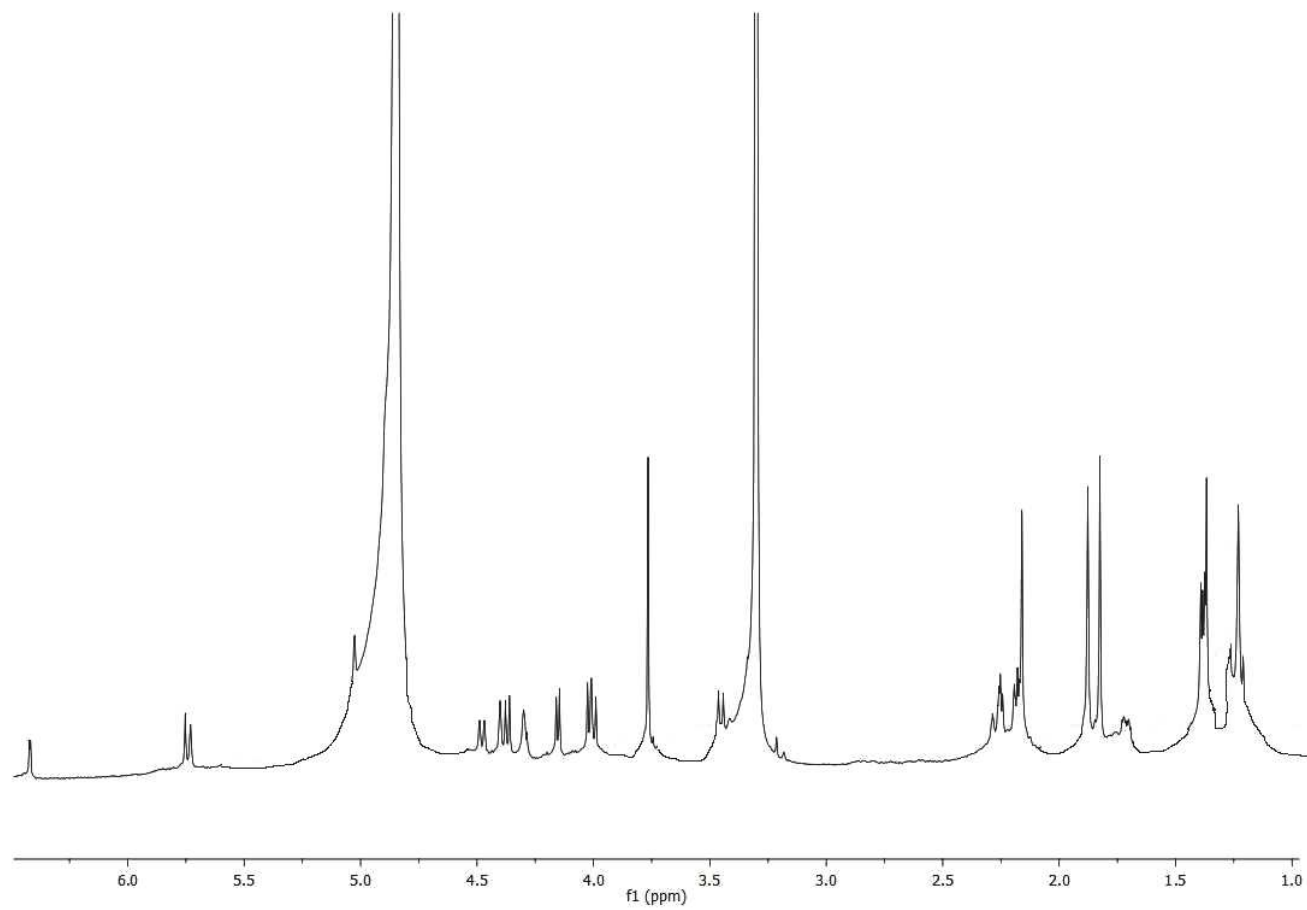
**Figure S15.** HSQC spectrum of compound **5** (CD<sub>3</sub>OD)



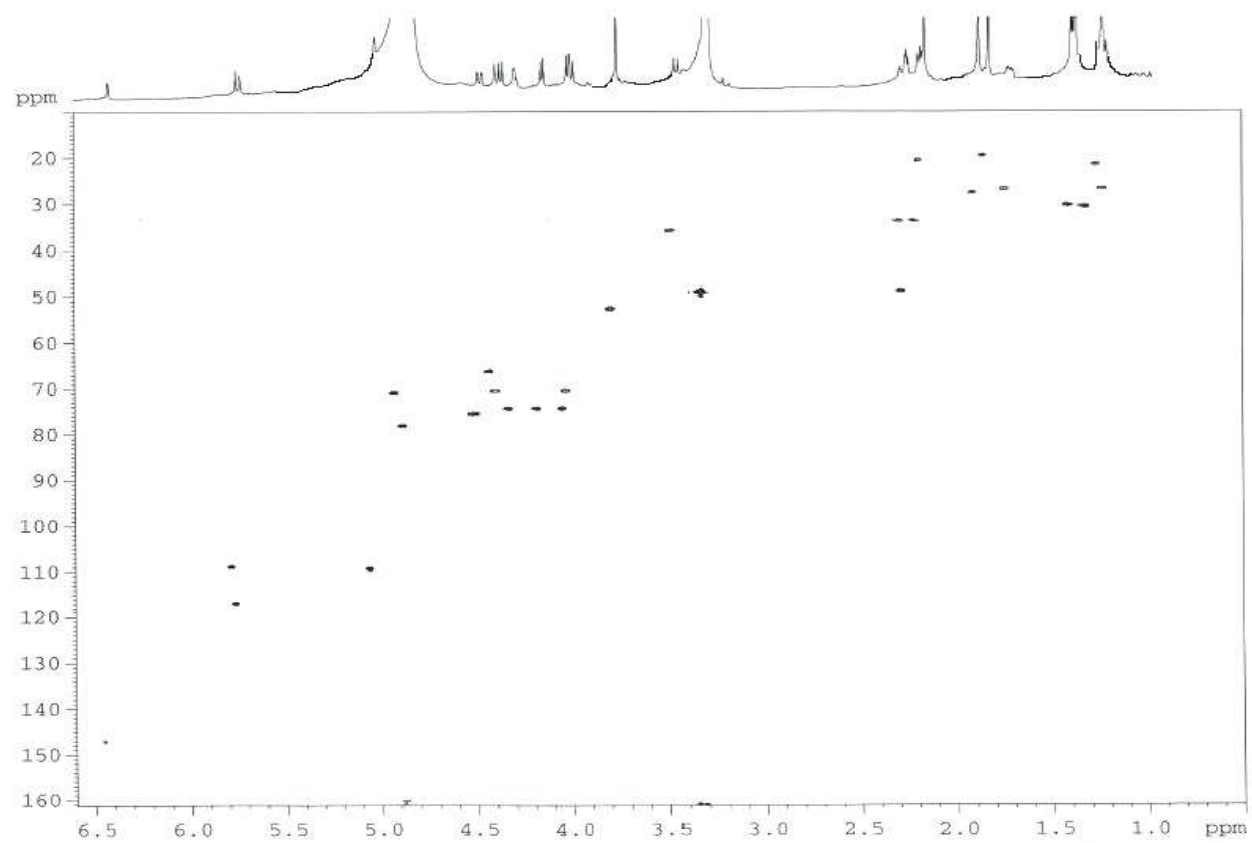
**Figure S16.** HMBC spectrum of compound **5** (CD<sub>3</sub>OD)



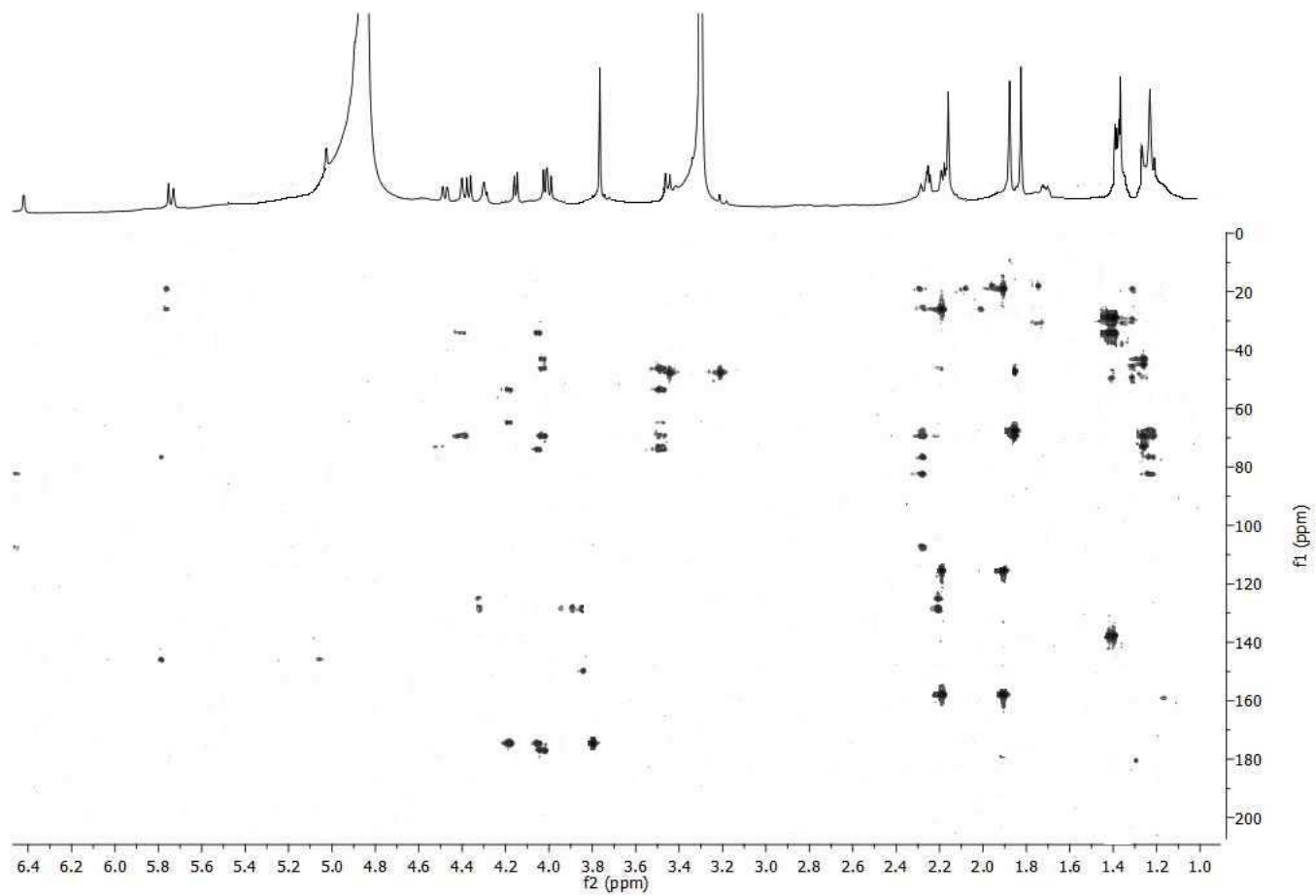
**Figure S17.**  $^1\text{H}$  NMR spectrum of compound **6** ( $\text{CD}_3\text{OD}$ )



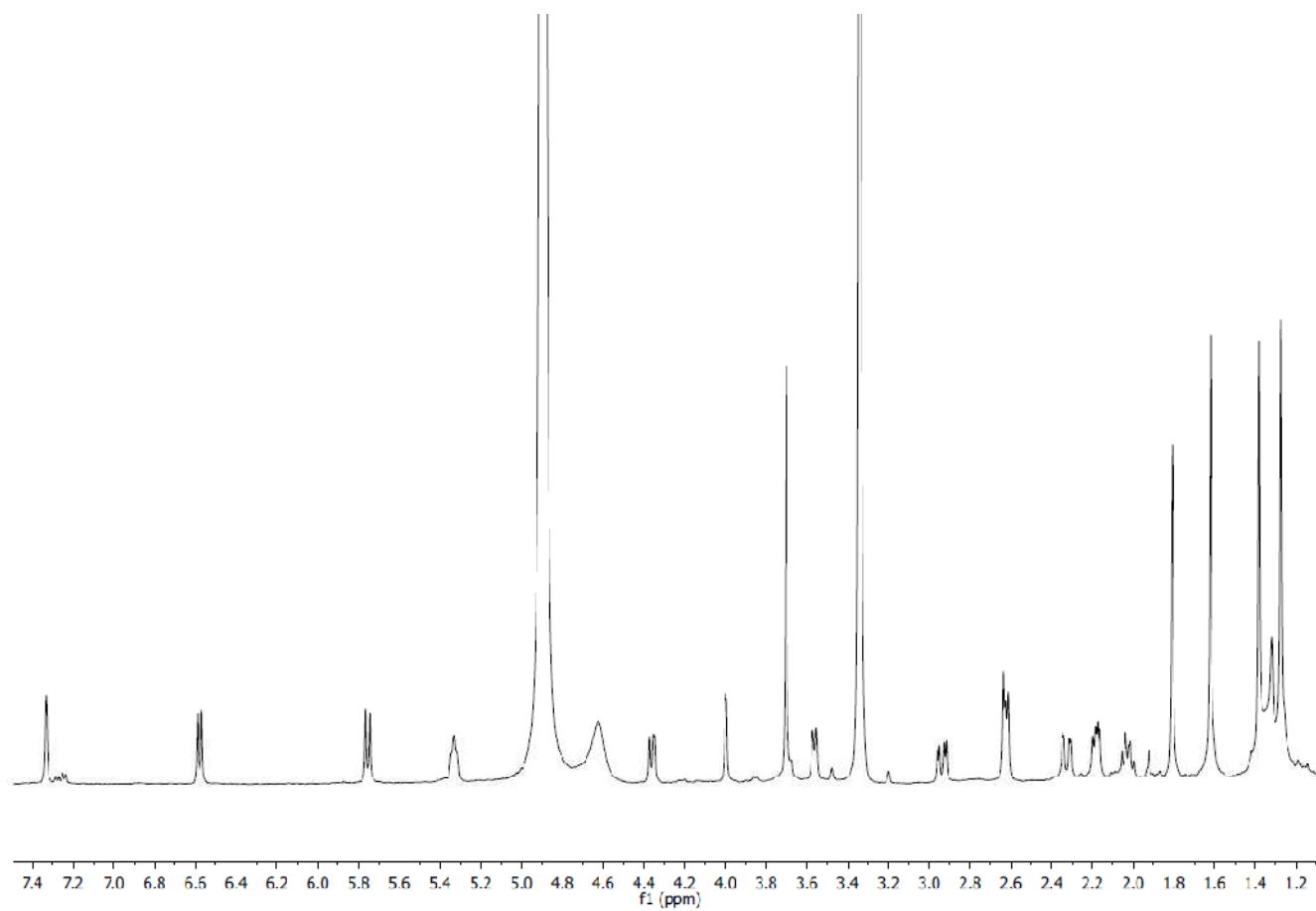
**Figure S18.** HSQC spectrum of compound **6** (CD<sub>3</sub>OD)



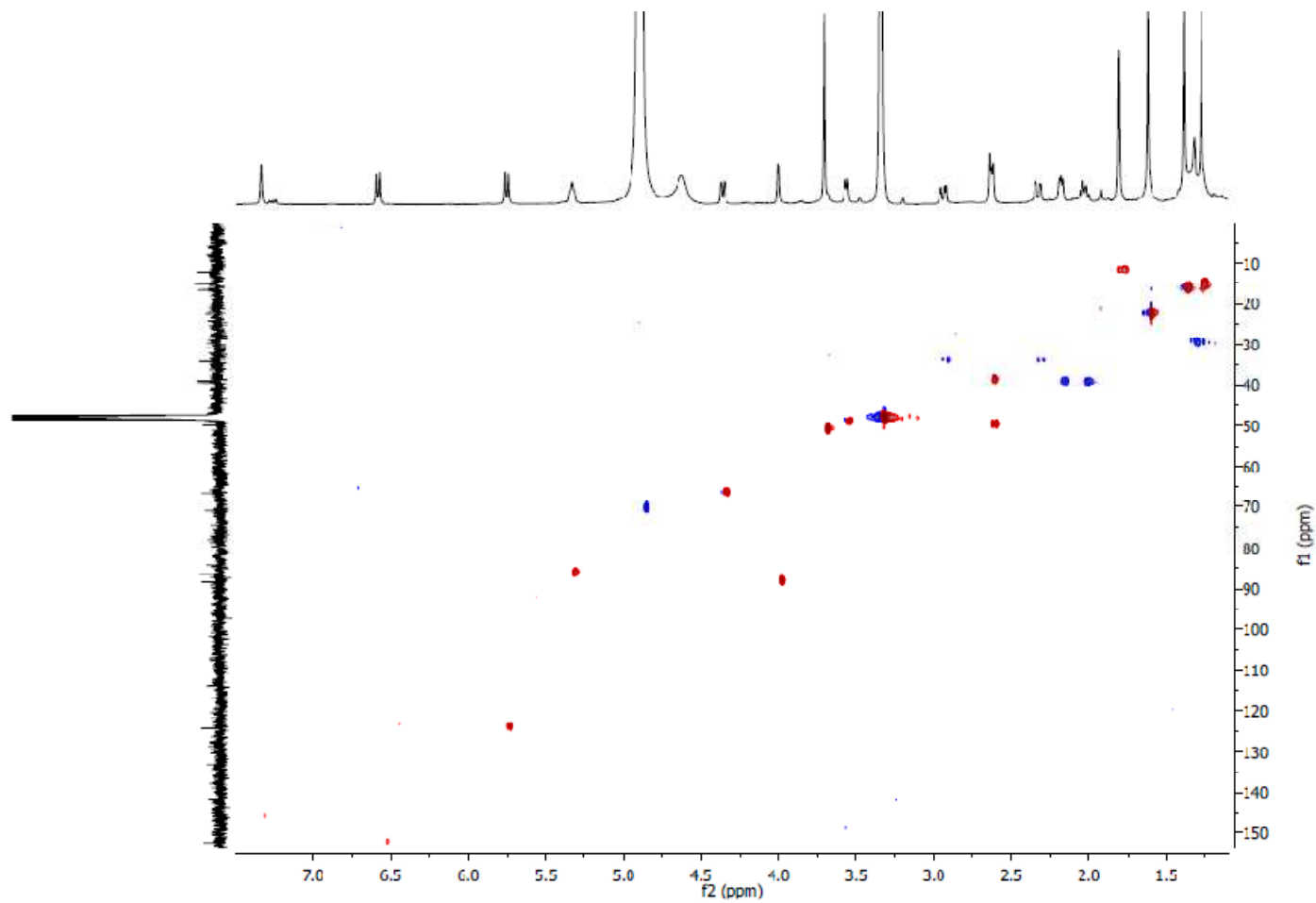
**Figure S19.** HMBC spectrum of compound **6** (CD<sub>3</sub>OD)



**Figure S20.**  $^1\text{H}$  NMR spectrum of compound **7** ( $\text{CD}_3\text{OD}$ )

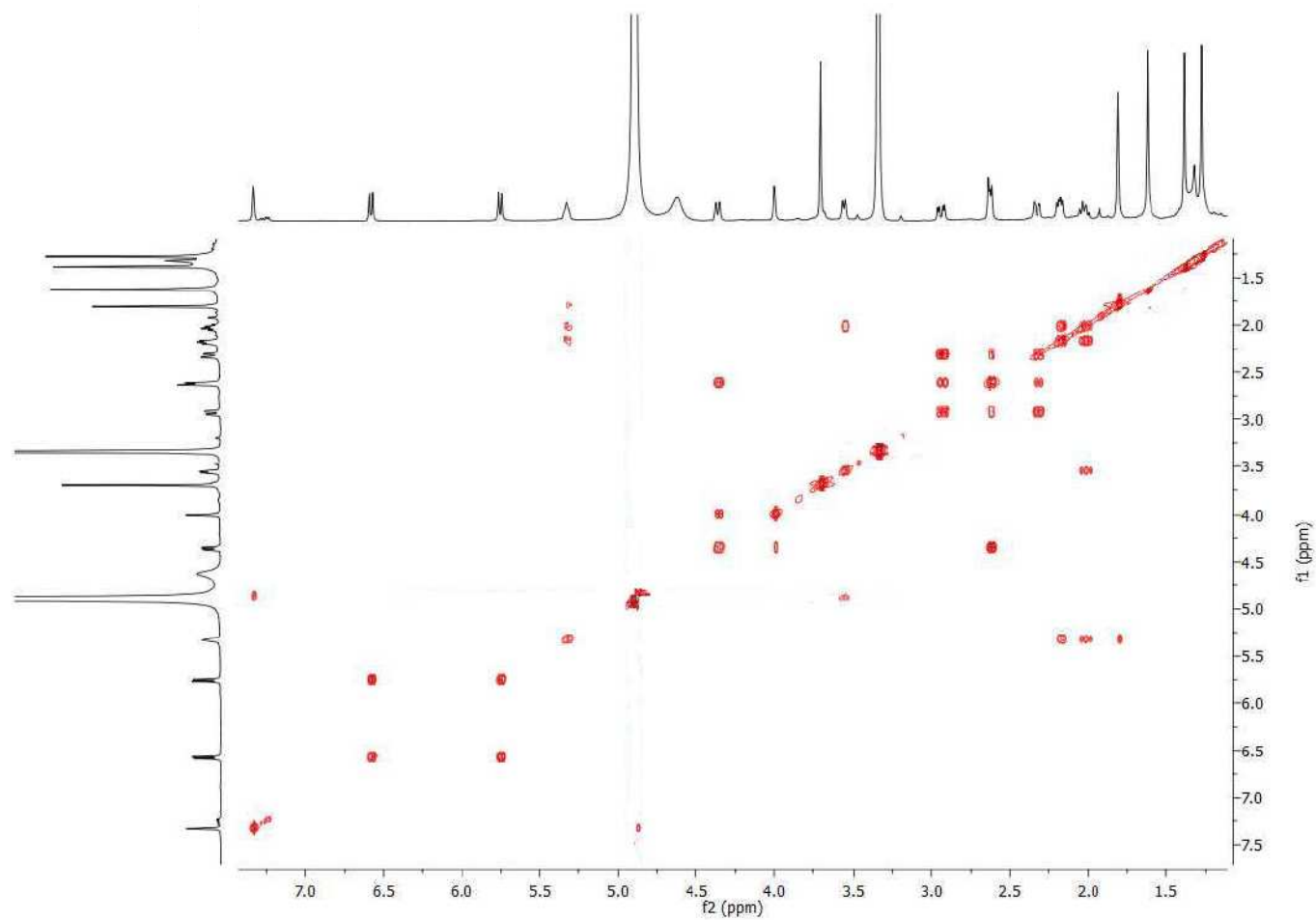


**Figure S21.** HSQC spectrum of compound **7** (CD<sub>3</sub>OD)

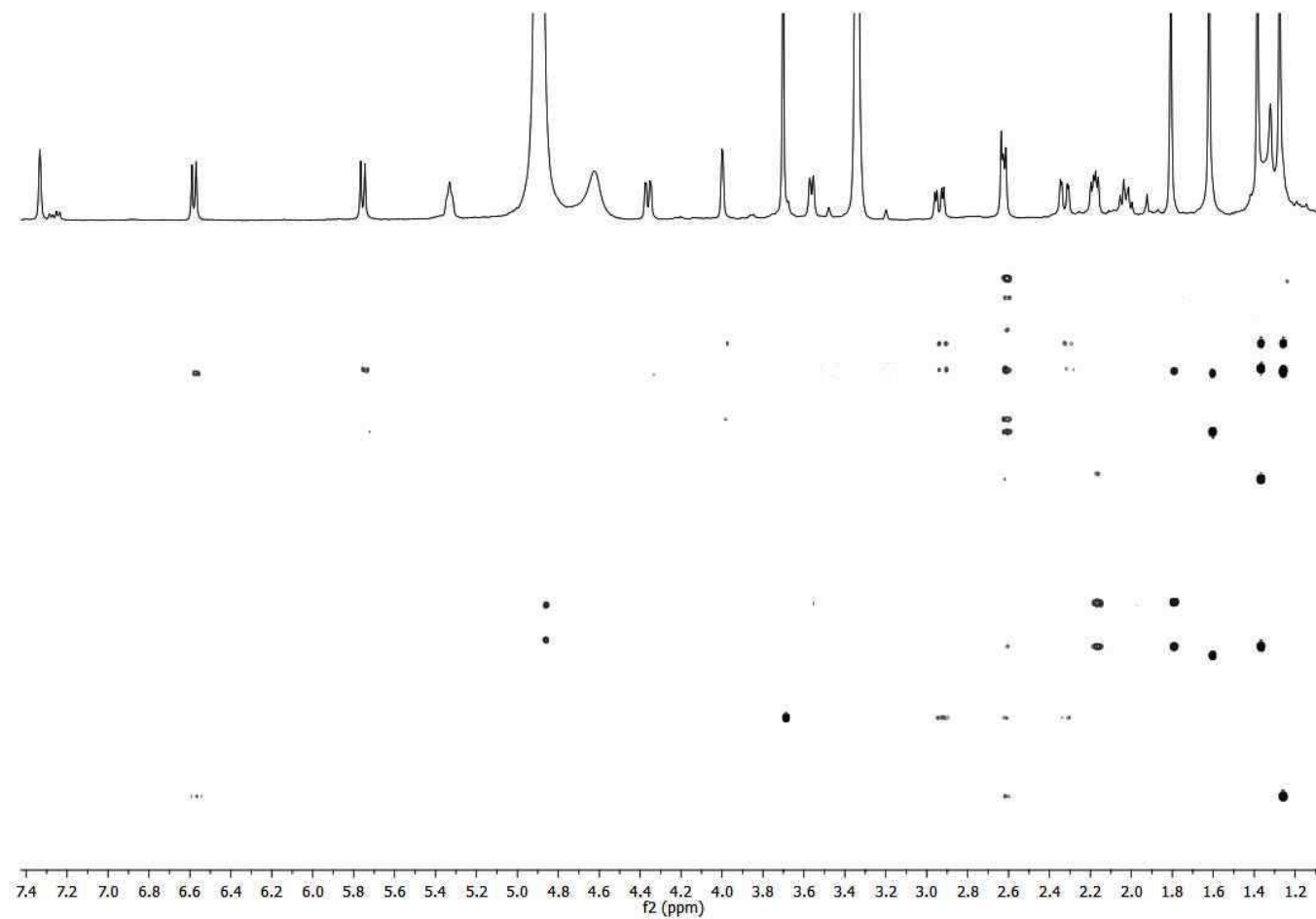




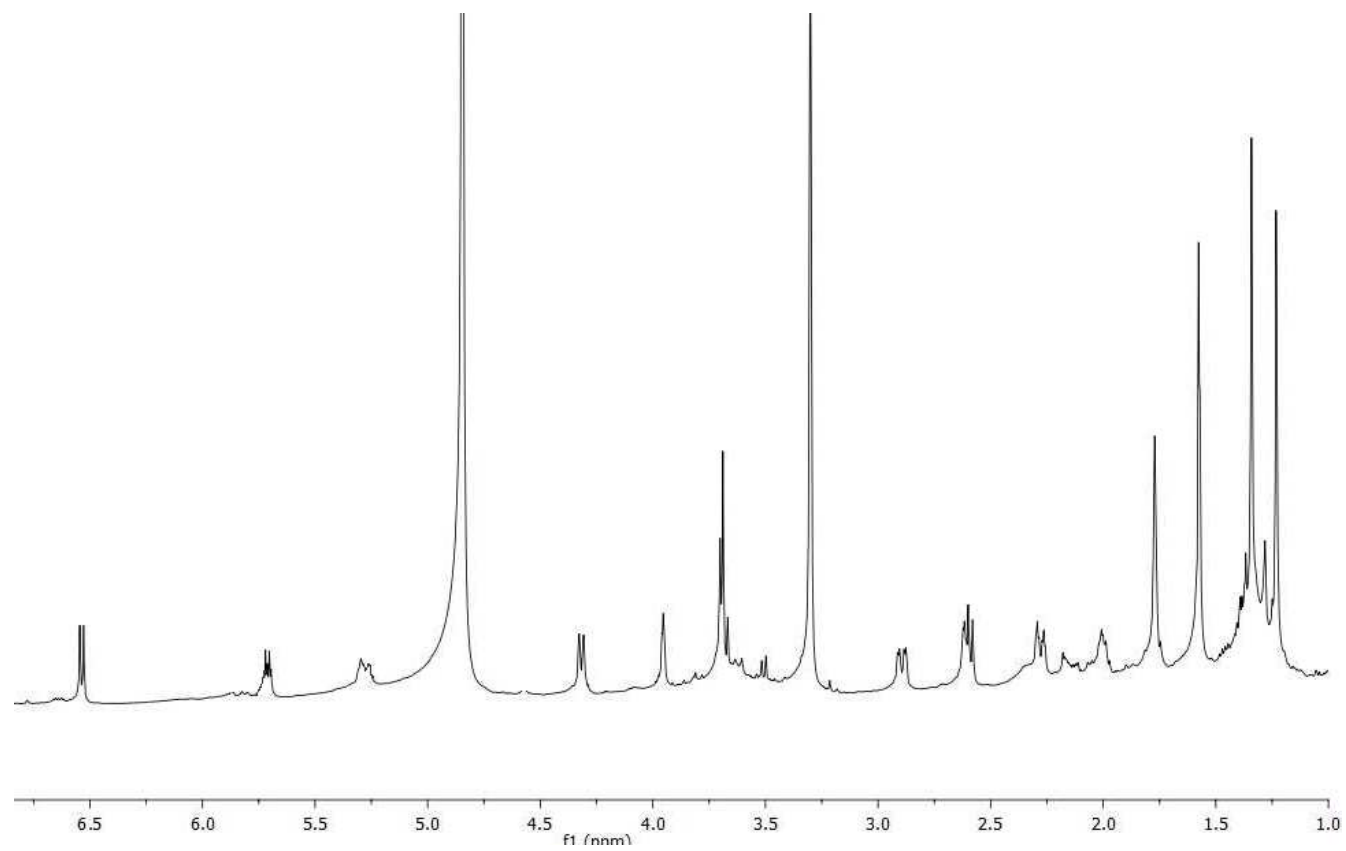
**Figure S22.** COSY spectrum of compound **7** (CD<sub>3</sub>OD)



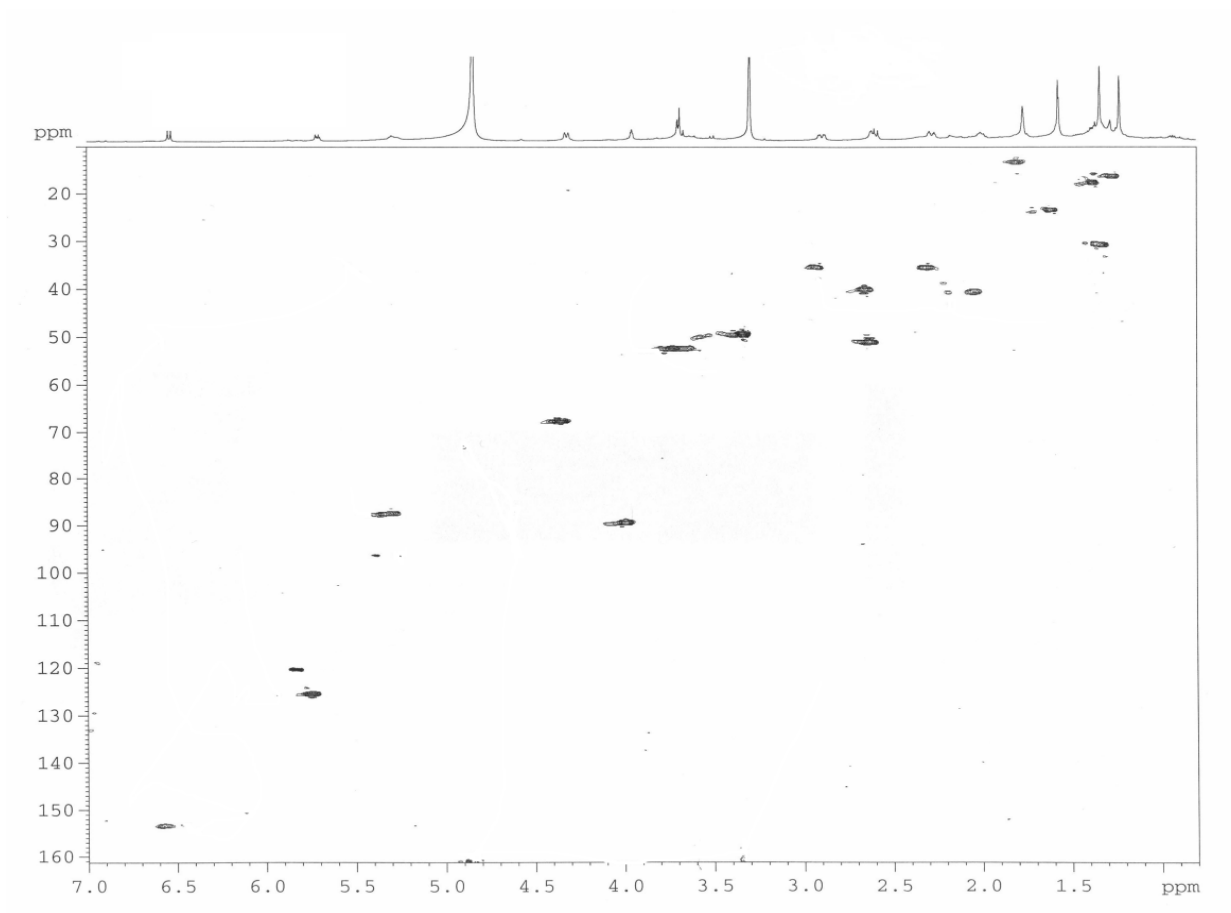
**Figure S23.** HMBC spectrum of compound **7** (CD<sub>3</sub>OD)



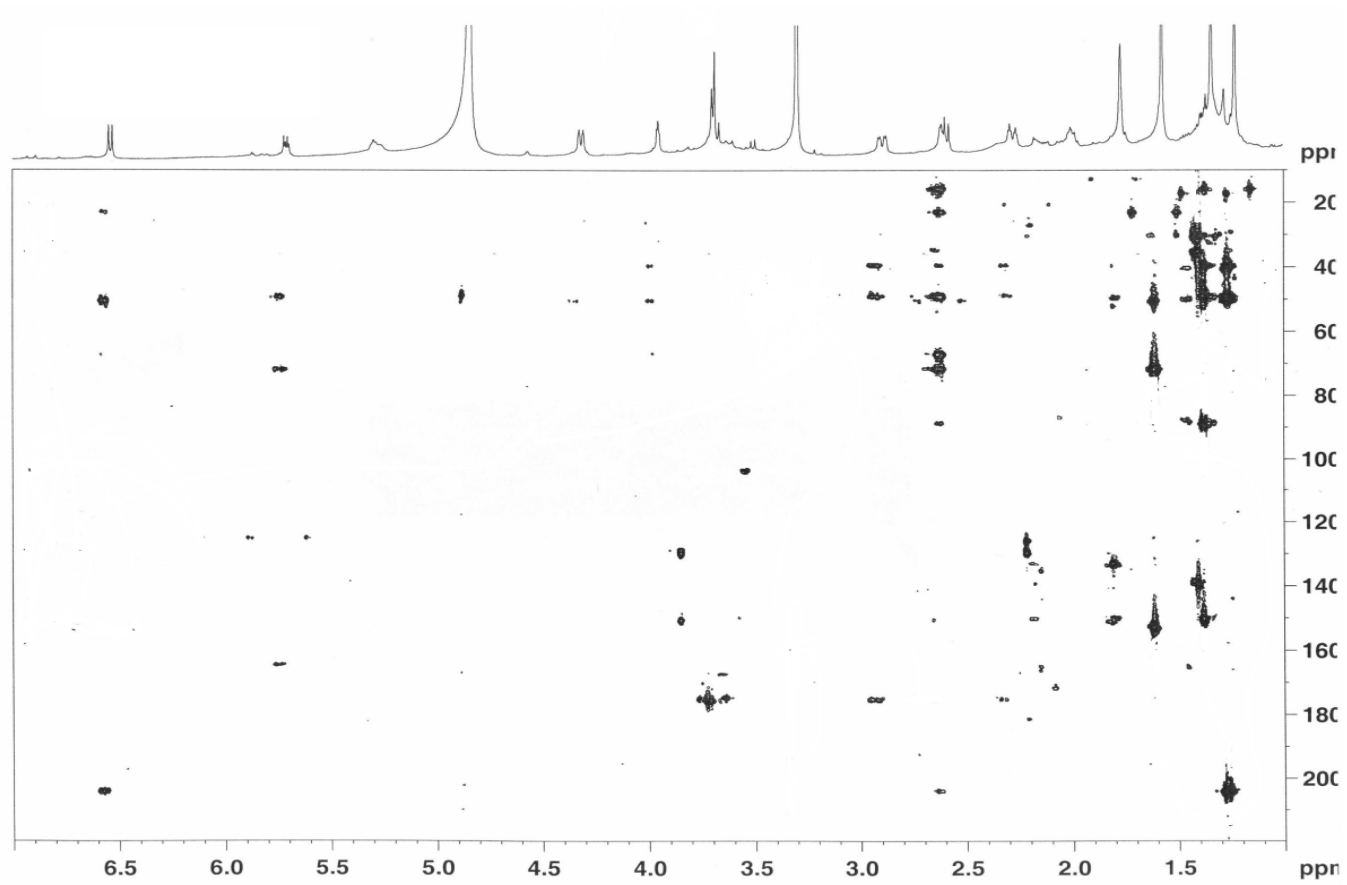
**Figure S24.**  $^1\text{H}$ NMR spectrum of compound **8** ( $\text{CD}_3\text{OD}$ )



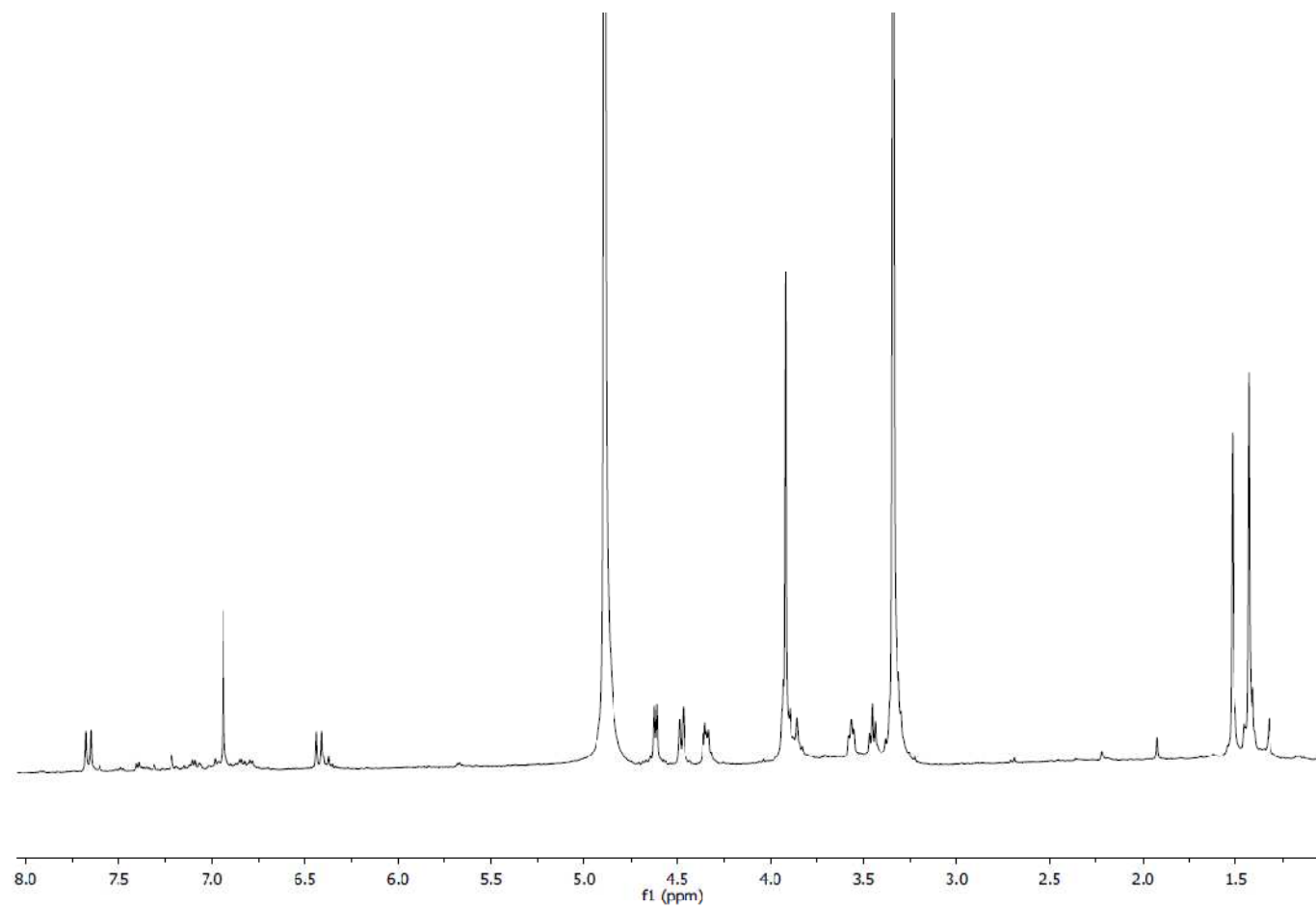
**Figure S25.** HSQC spectrum of compound **8** (CD<sub>3</sub>OD)



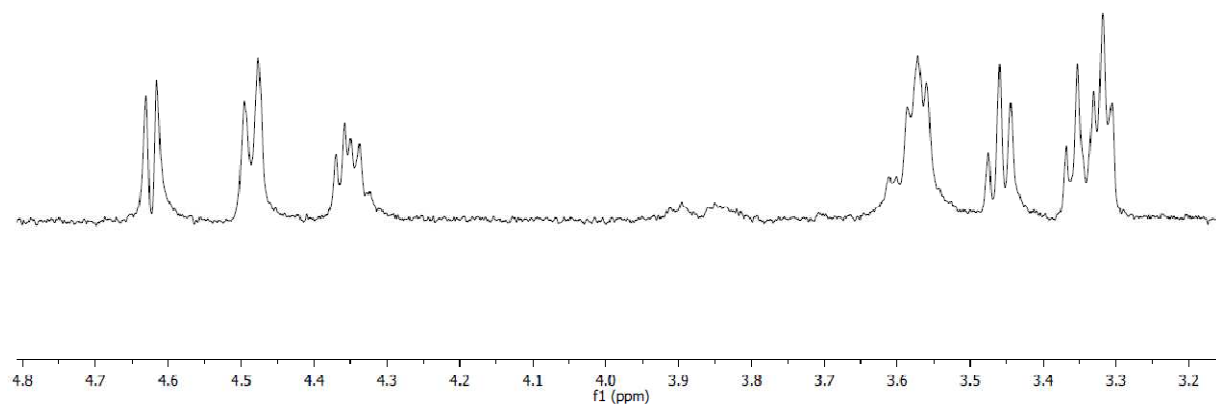
**Figure S26.** HMBC spectrum of compound **8** (CD<sub>3</sub>OD)



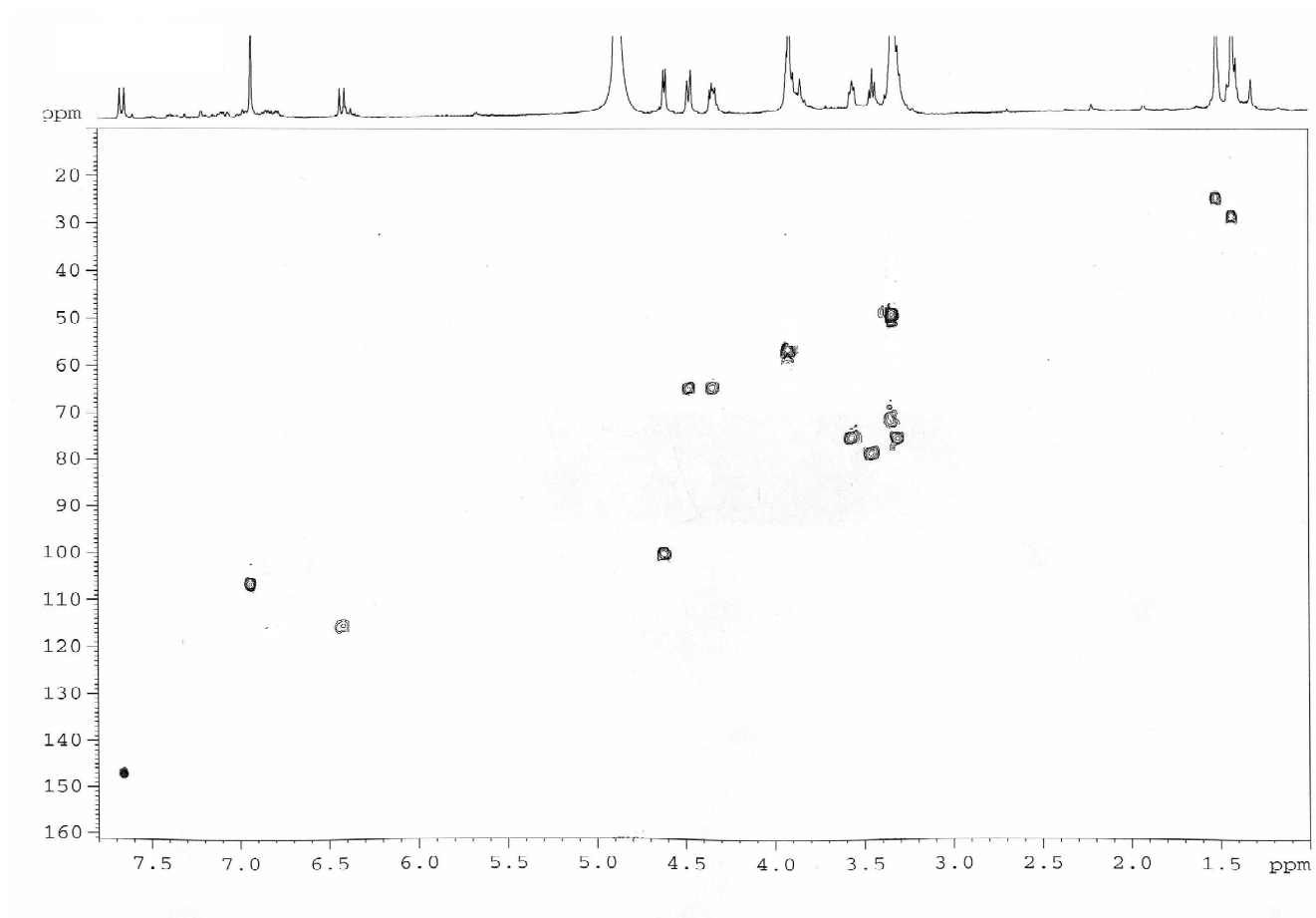
**Figure S27.**  $^1\text{H}$  NMR spectrum of compound **9** ( $\text{CD}_3\text{OD}$ )



**Figure S28.** 1D TOCSY spectrum of compound **9** (CD<sub>3</sub>OD)



**Figure S29.** HSQC spectrum of compound **9** (CD<sub>3</sub>OD)





**Figure S30.** HMBC spectrum of compound **9** (CD<sub>3</sub>OD)

