**Supporting Information**

# Gracilosulfates A-G, Monosulfated Polyoxygenated Steroids from the Marine Sponge *Haliclona gracilis*

**Larisa K. Shubina[1], Tatyana N. Makarieva[1,*], Vladimir A. Denisenko[1], Roman S. Popov[1], Sergey A. Dyshlovoy[1,2,3], Boris B. Grebnev[1], Pavel S. Dmitrenok[1], Gunhild von Amsberg[2,3], and Valentin A. Stonik[1]**

[1] G. B. Elyakov Pacific Institute of Bioorganic Chemistry, Far-Eastern Branch of the Russian Academy of Sciences, Prospect 100-let Vladivostoku 159, Vladivostok 690022, Russian Federation

[2] Laboratory of Experimental Oncology, Department of Oncology, Hematology and Bone Marrow Transplantation with Section Pneumology, Hubertus Wald-Tumorzentrum, University Medical Center Hamburg-Eppendorf, Hamburg, Germany

[3] Martini-Klinik, Prostate Cancer Center, University Hospital Hamburg-Eppendorf, Hamburg, Germany

[*] To whom correspondence should be addressed. Tel: 7 (4232) 31-11`-68. Fax: 7 (4232) 31-40-50. E-mail: makarieva@piboc.dvo.ru

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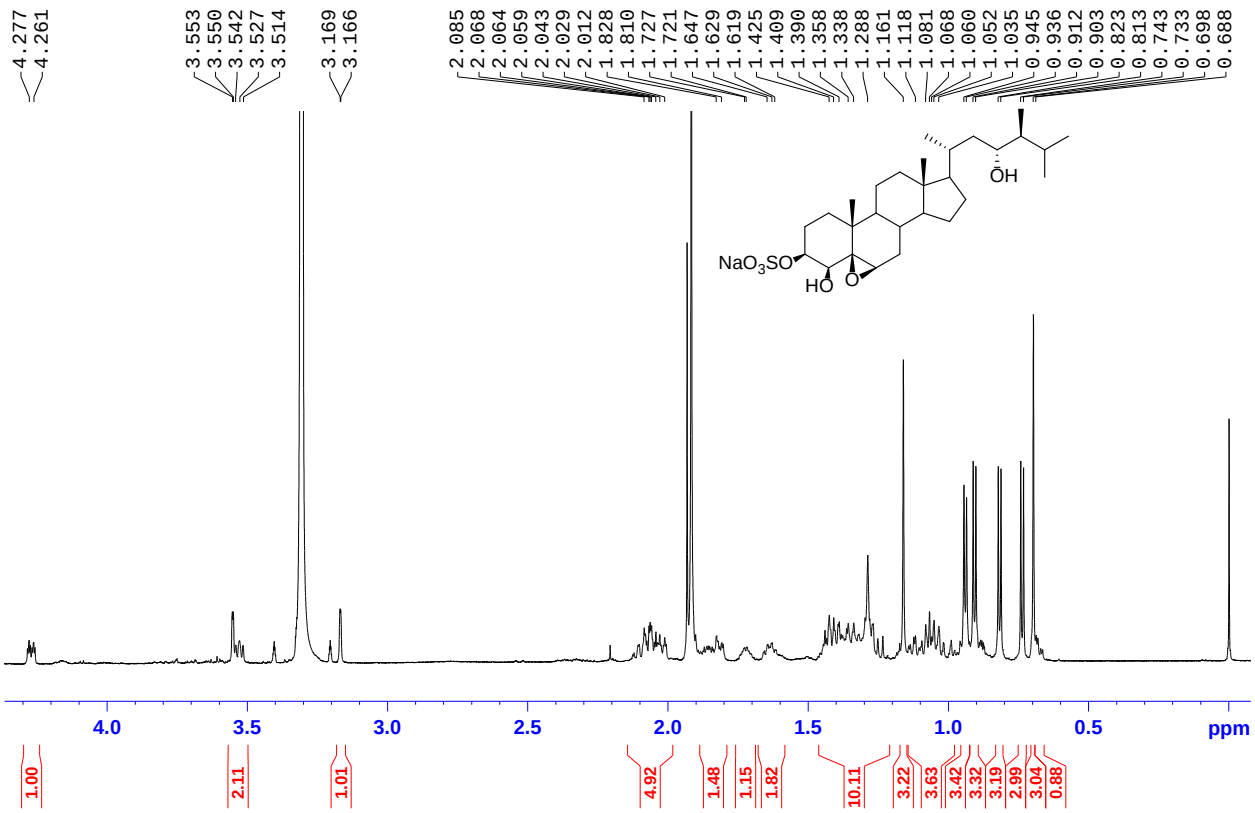

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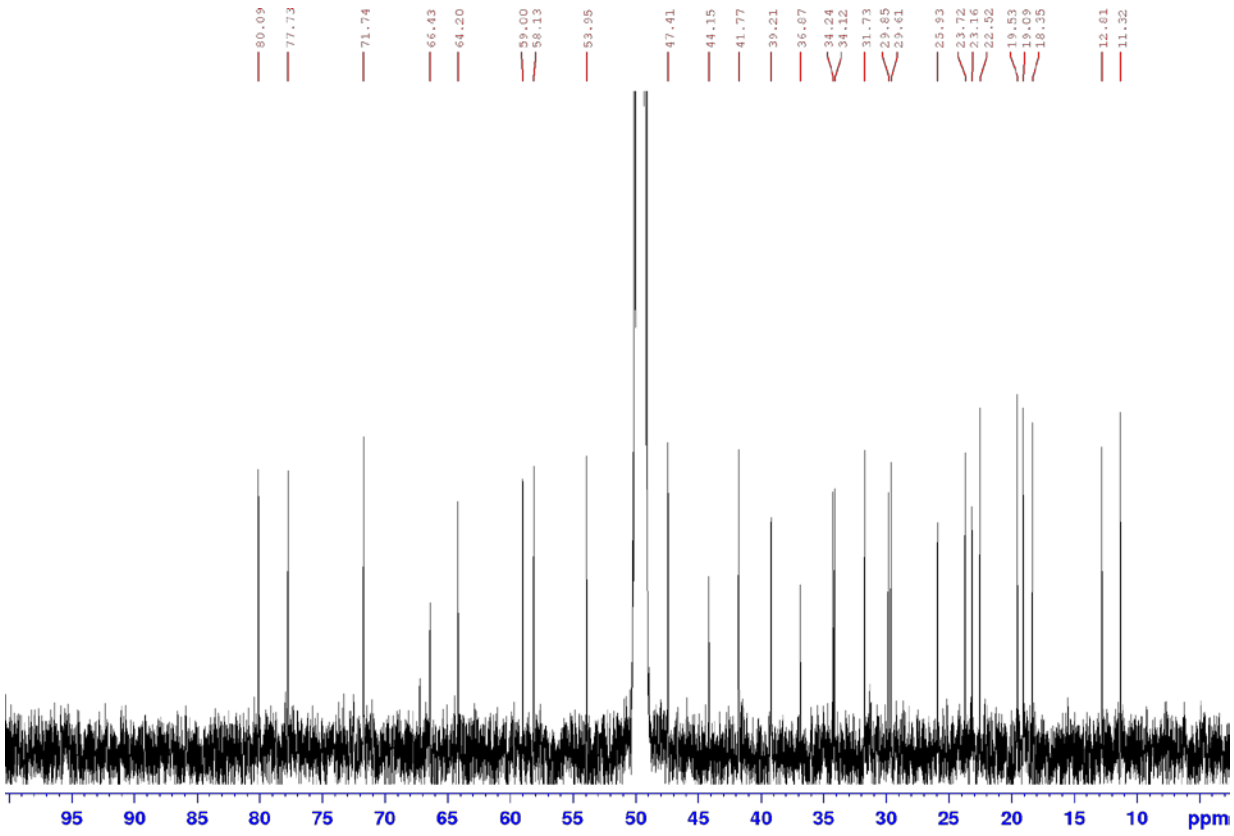


**Figure S3.** The COSY (700 MHz, $CD_3OD$) spectrum of compound **1**

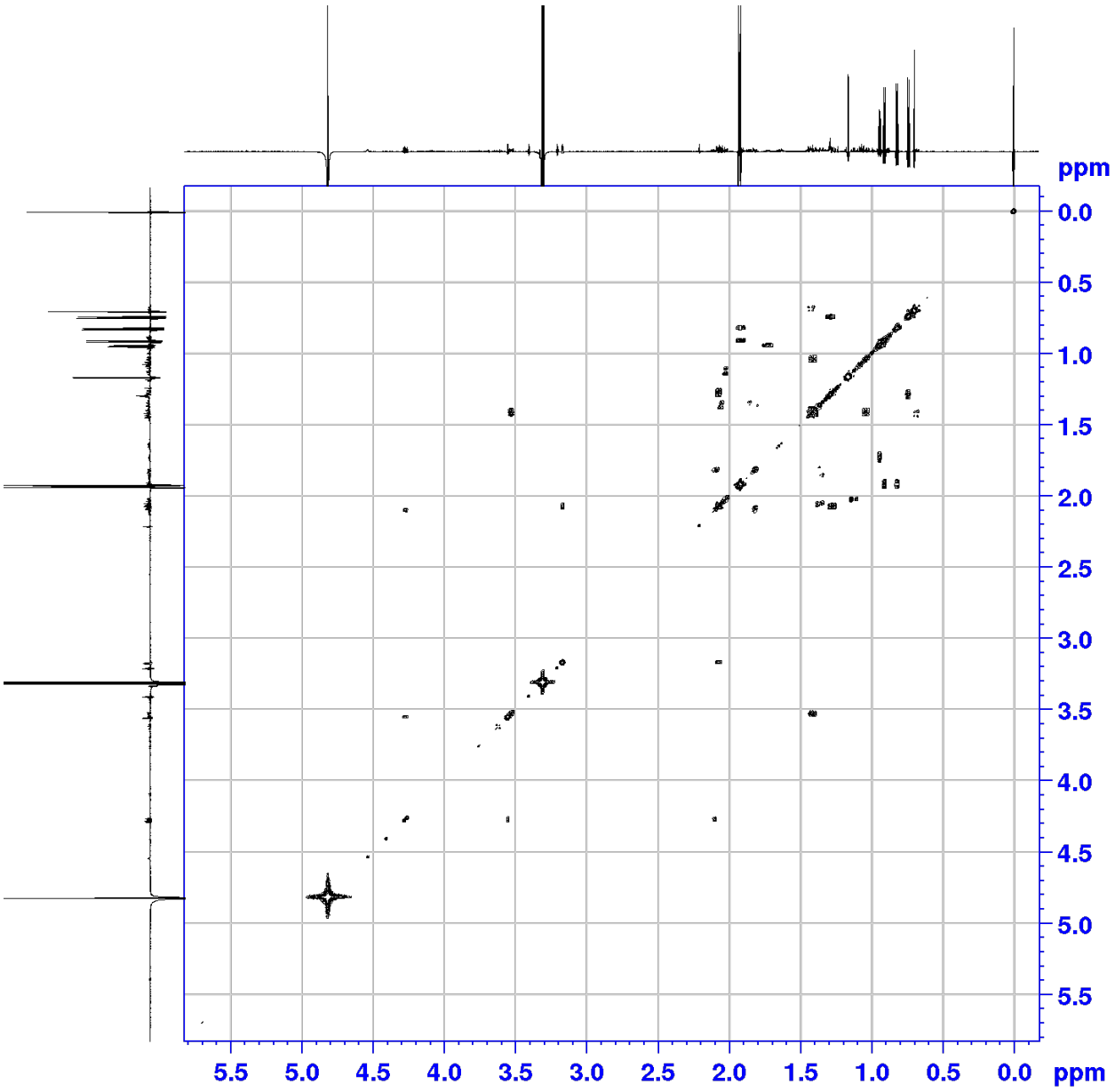


**Figure S4.** The HSQC (500/125 MHz, $CD_3OD$) spectrum of compound **1**

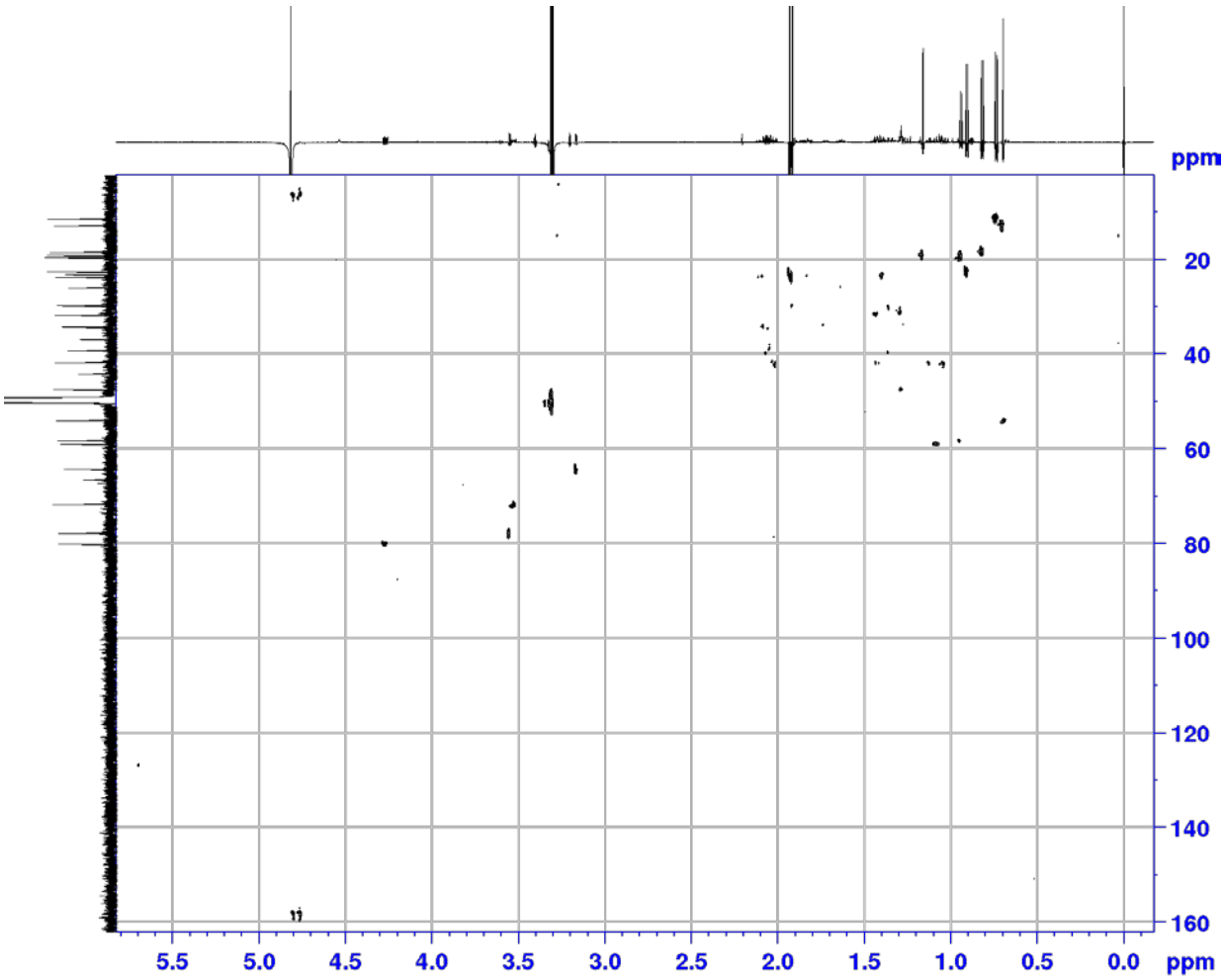
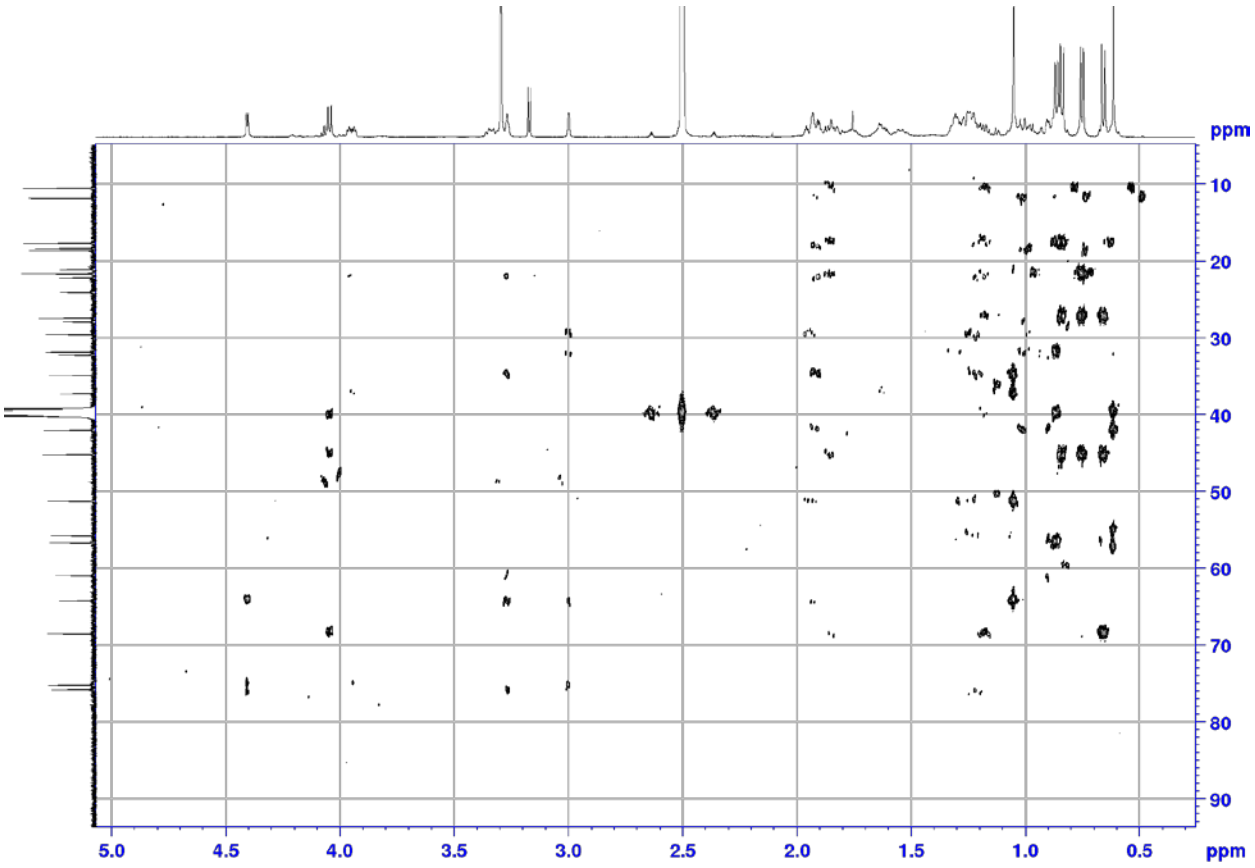


**Figure S5.** The HMBC (500/125 MHz, DMSO-$d_6$) spectrum of compound **1**



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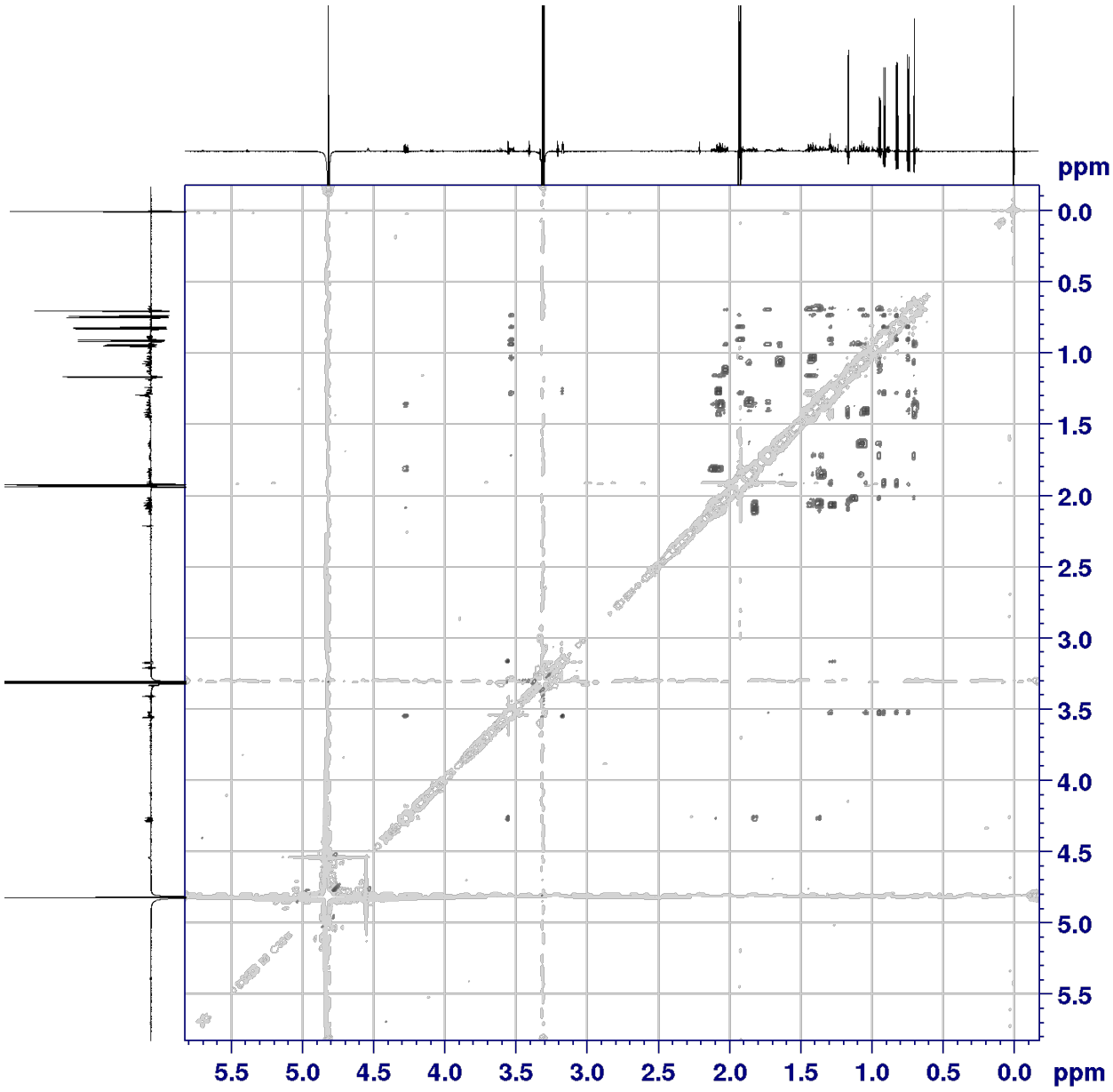


**Figure S7.** The HRESIMS and MS/MS spectra of compound **1**

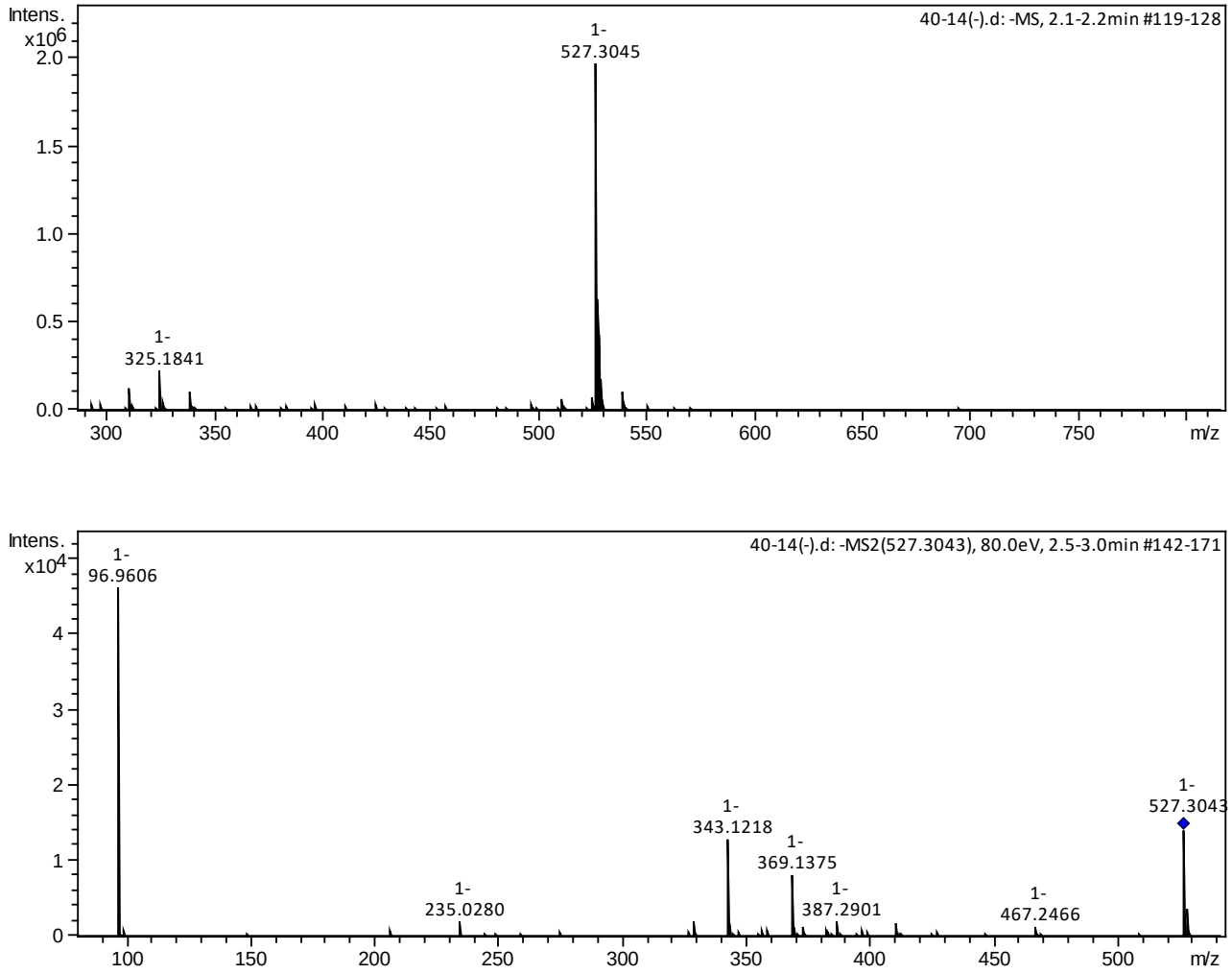
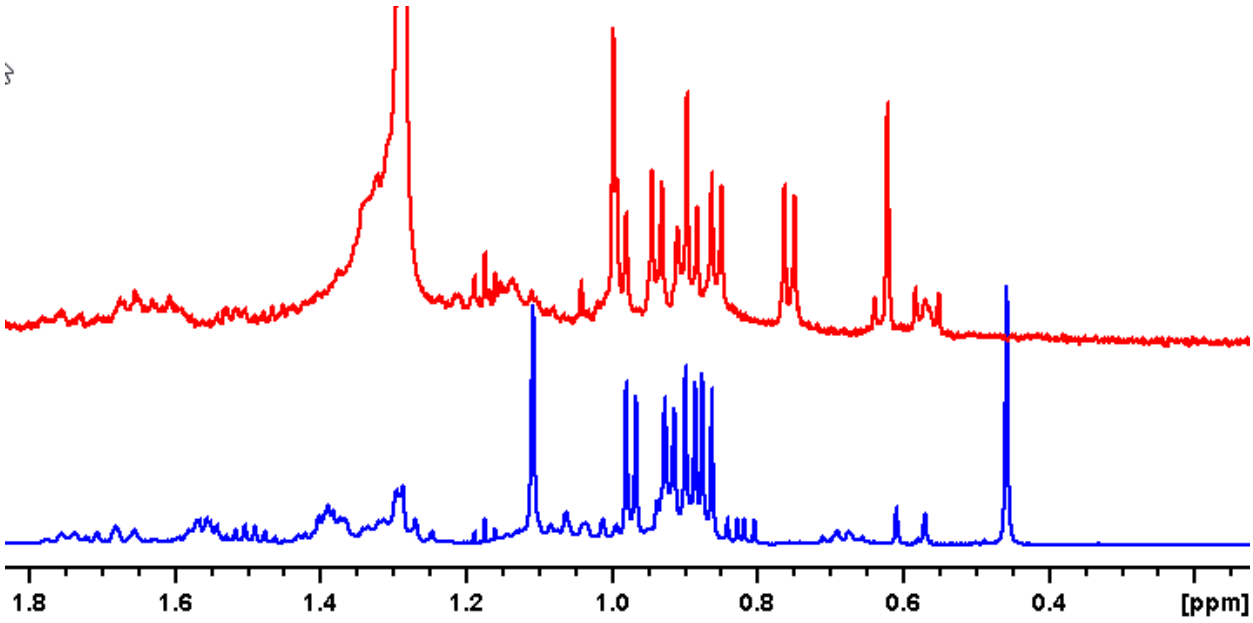


**Figure S8.** The $^{1}H$ NMR (700 MHz, $CD_3OD$) chemical shift differences between **1S** (red) and **1R** (blue)



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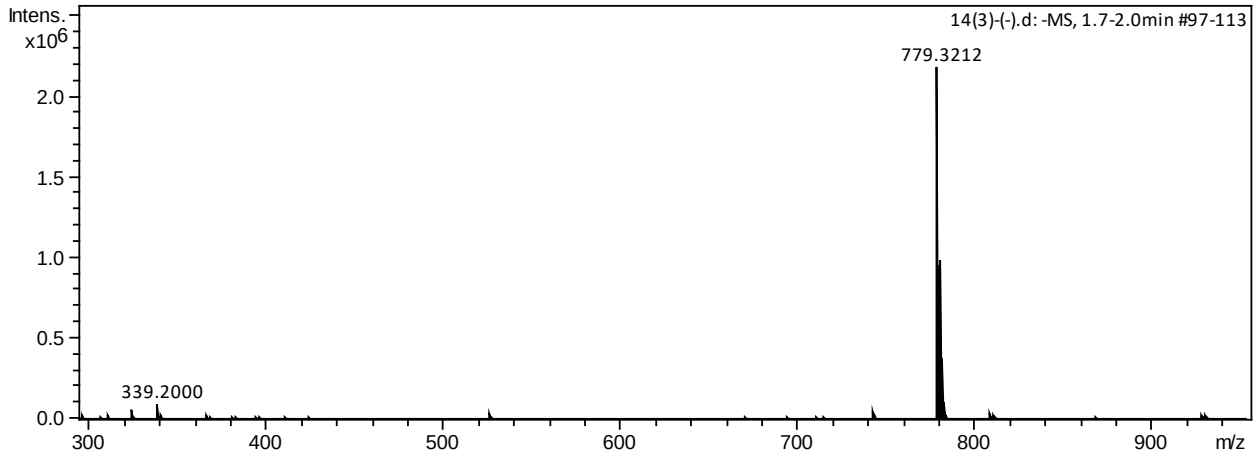

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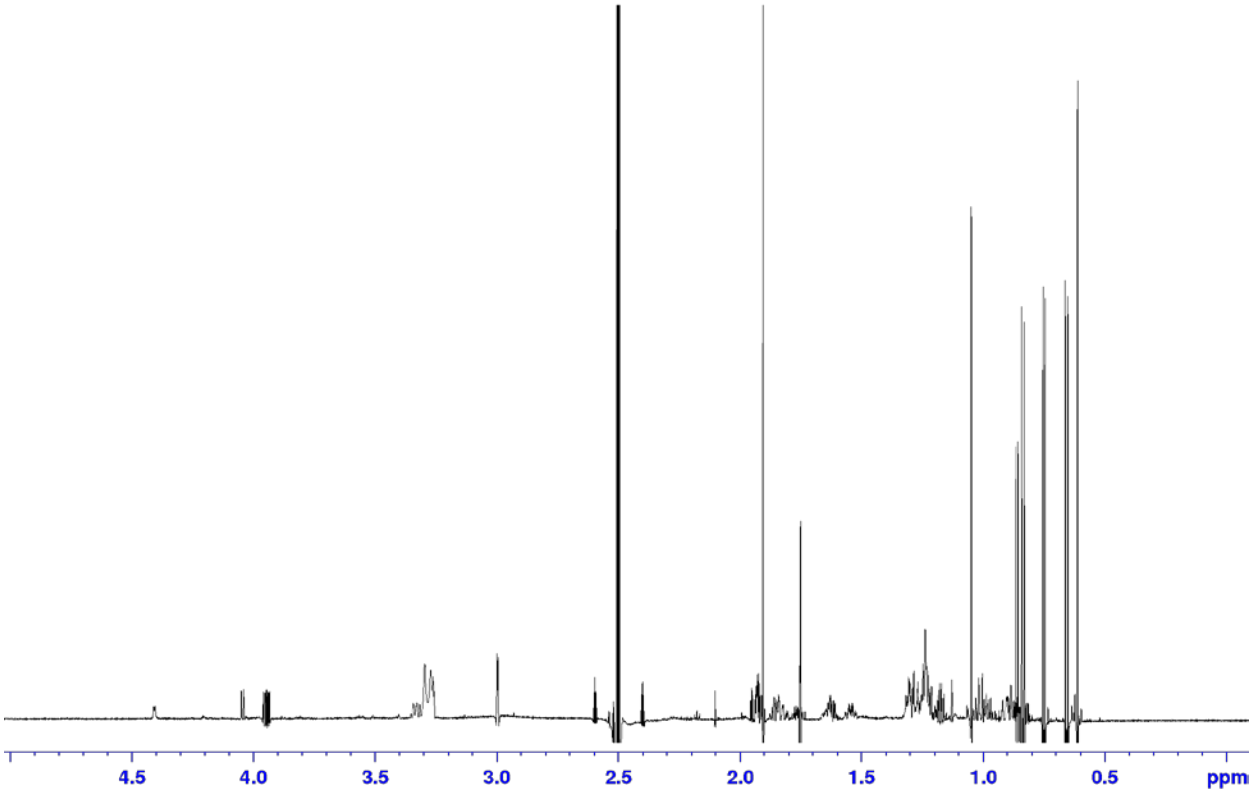

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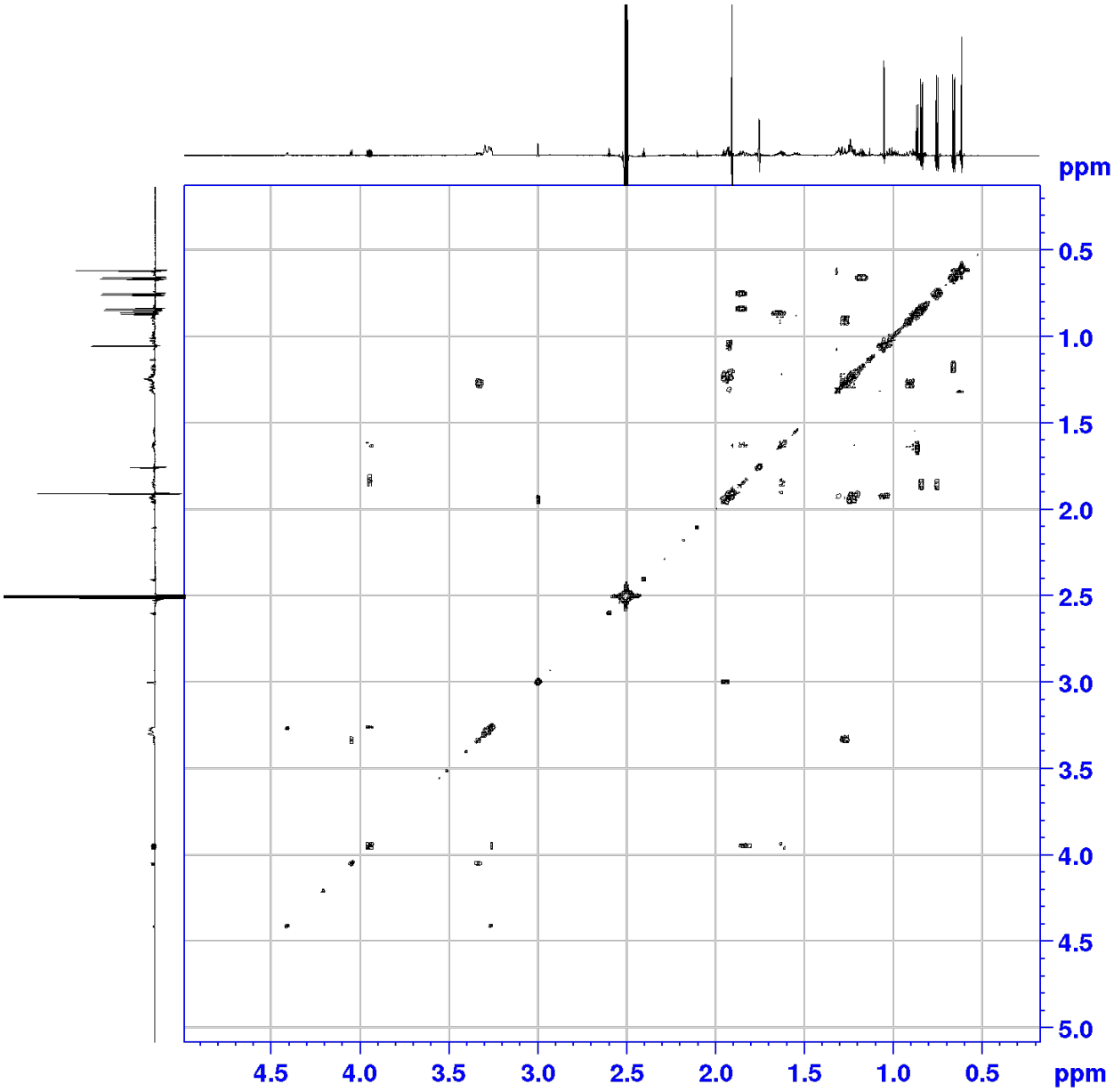

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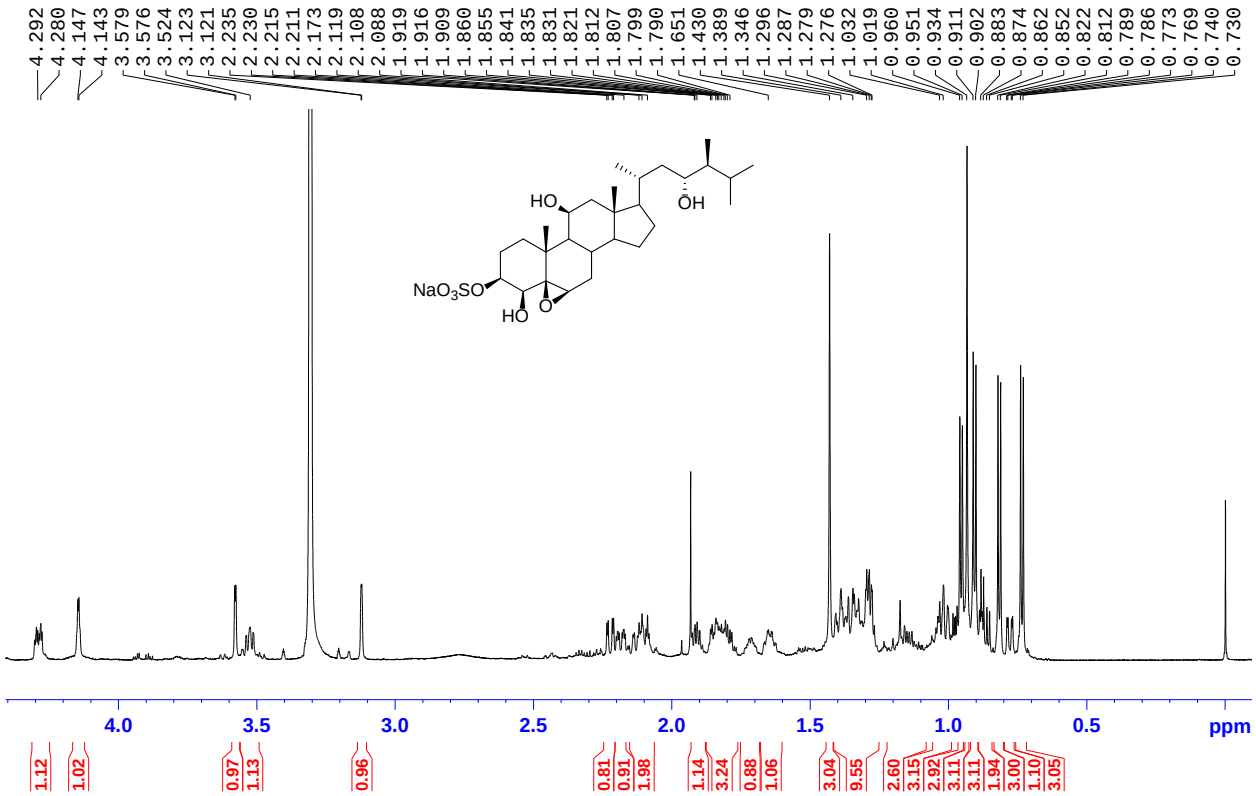

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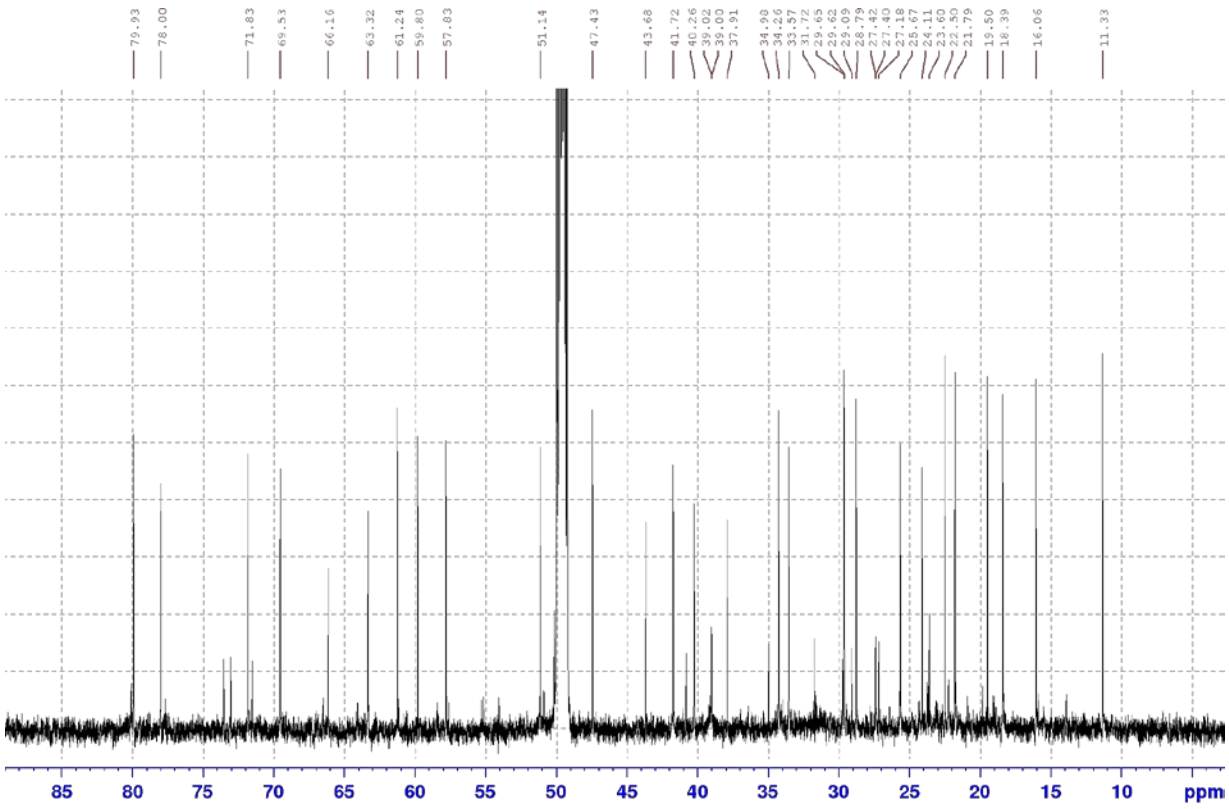

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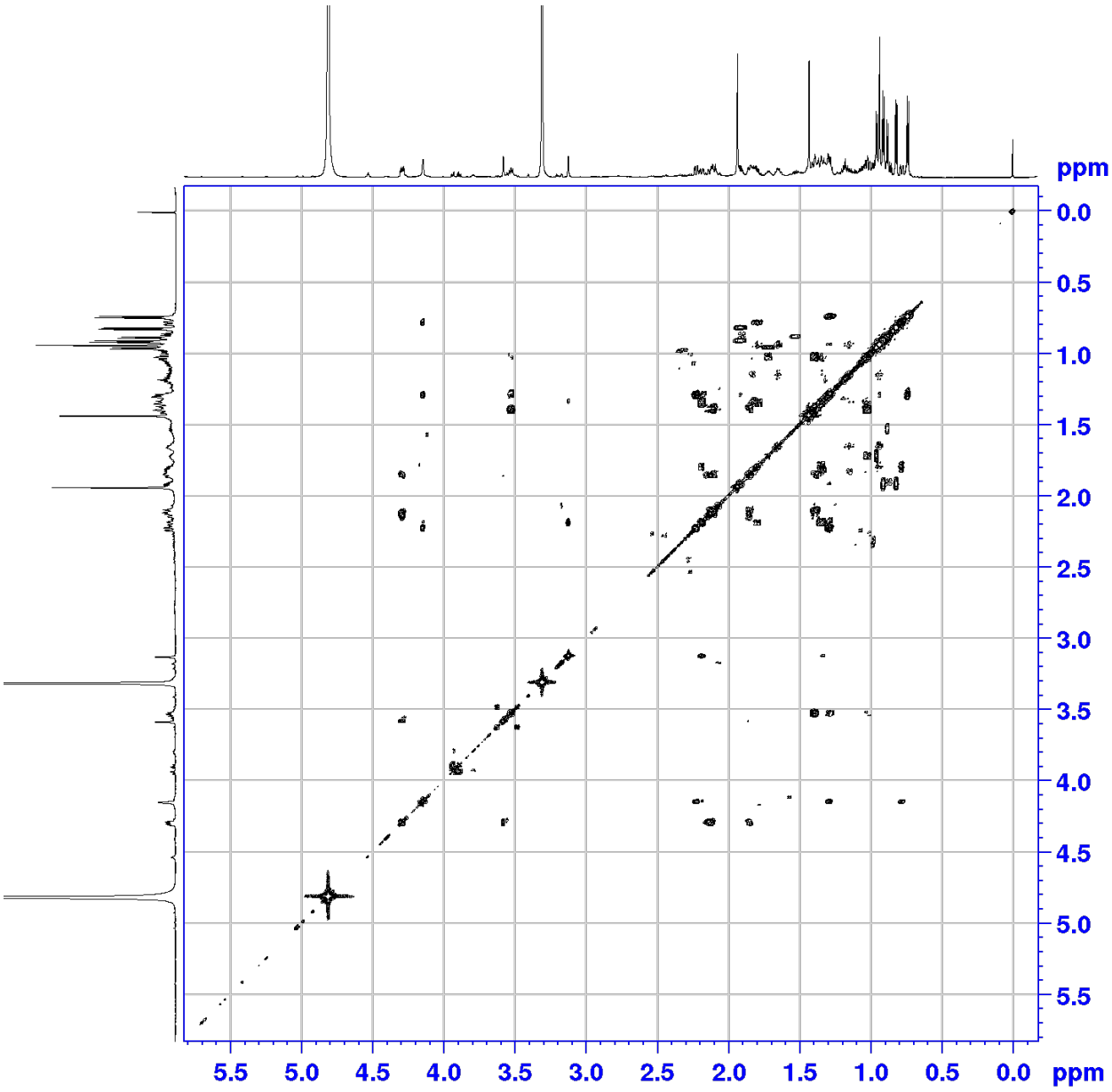

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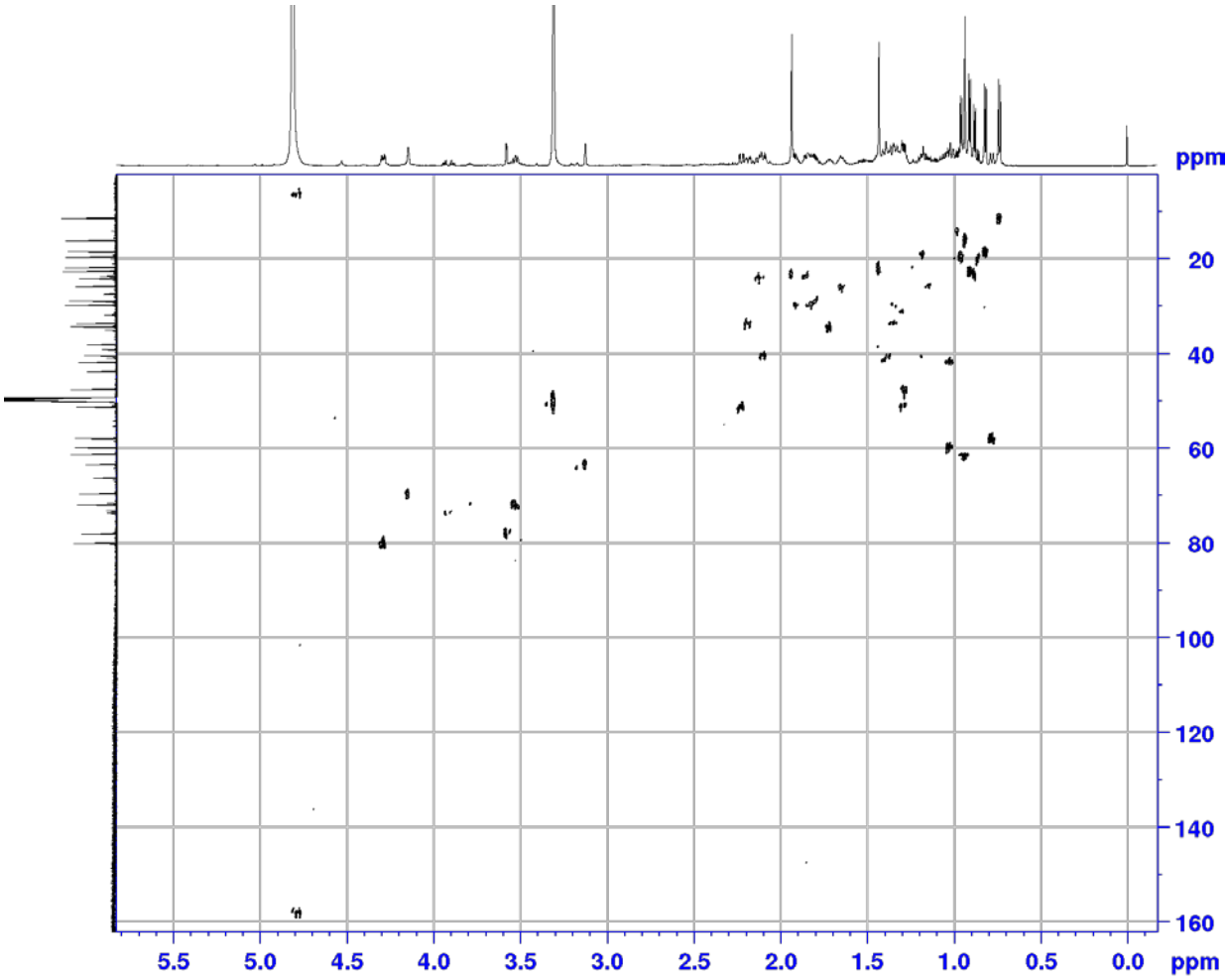

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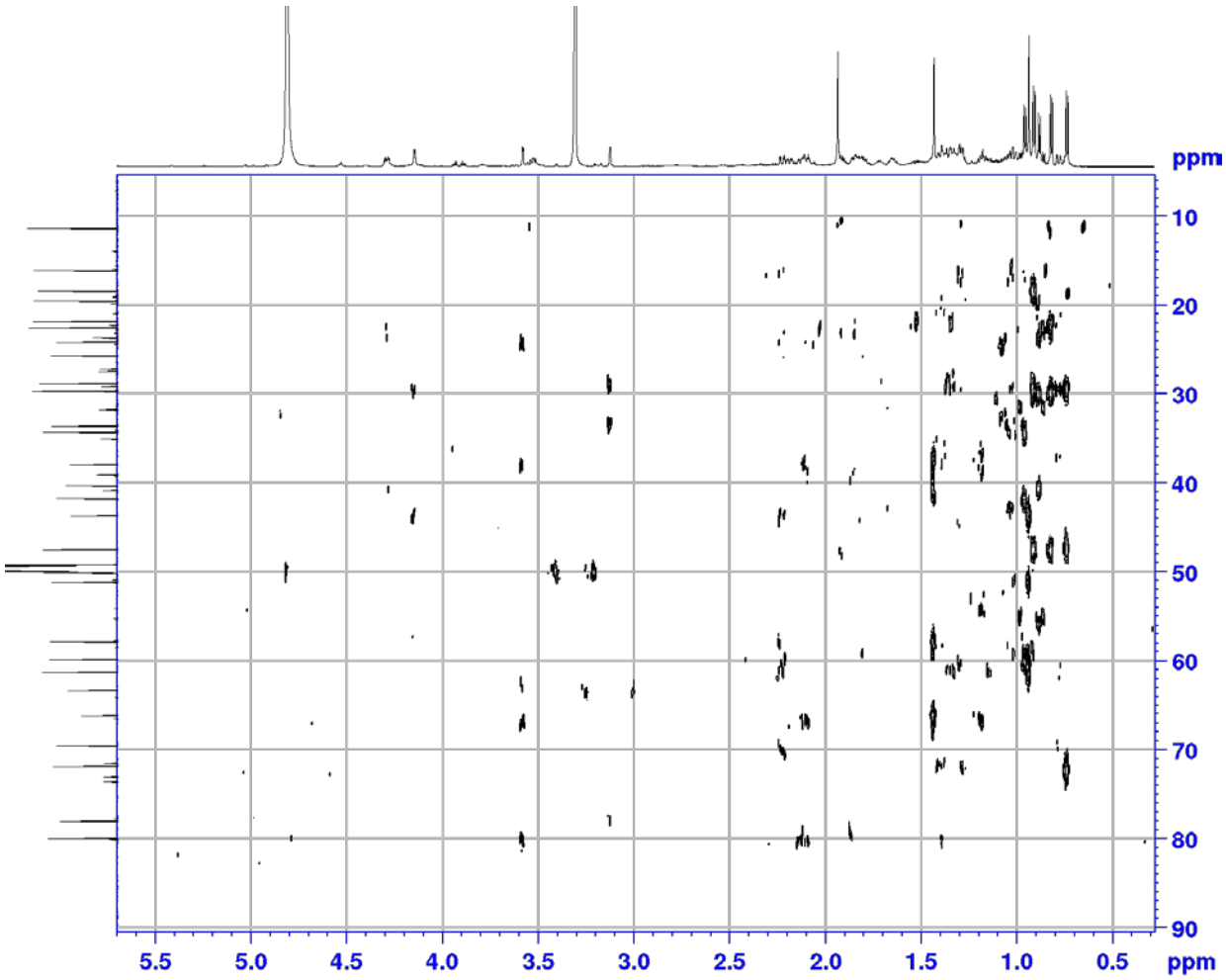

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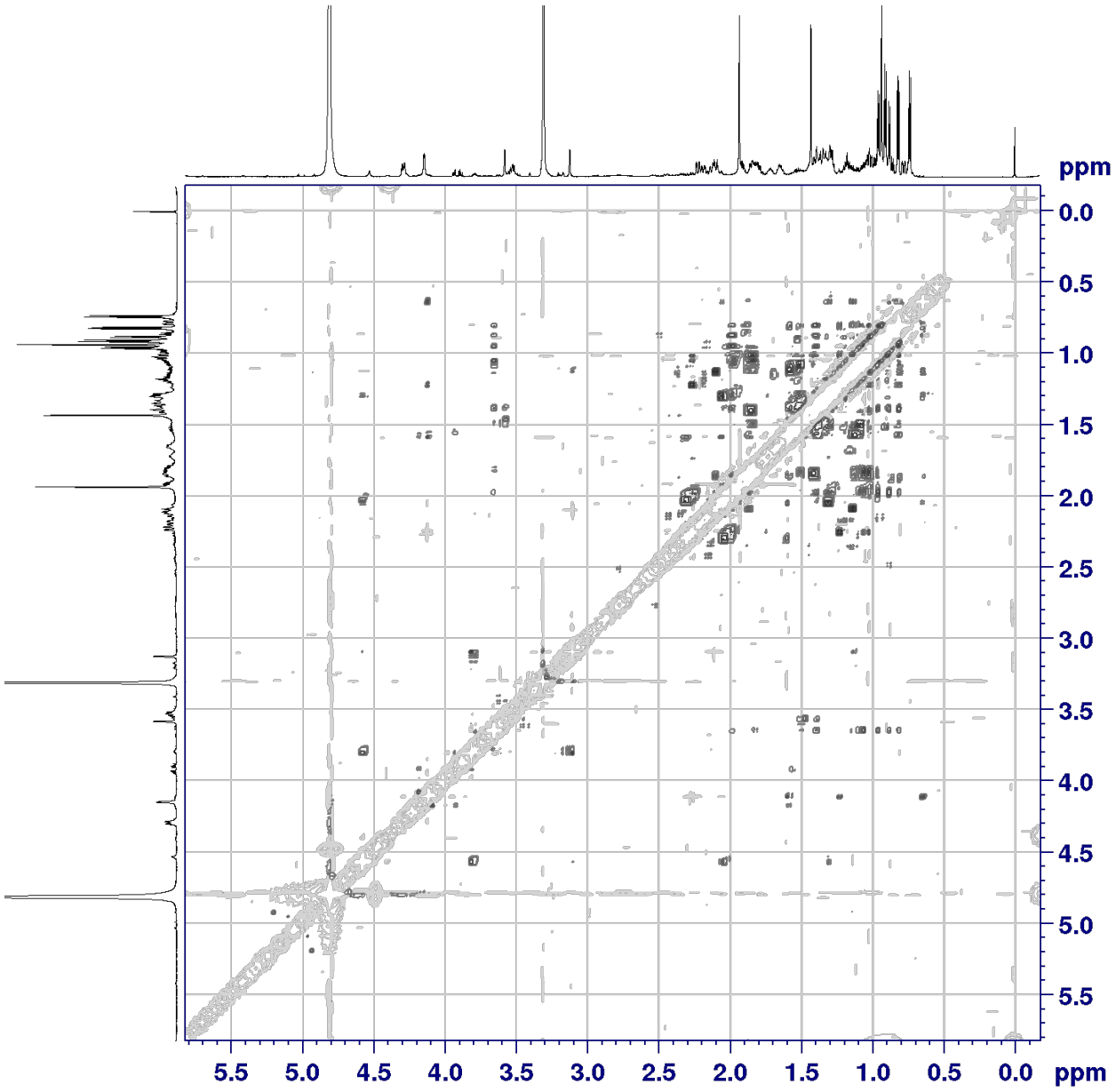

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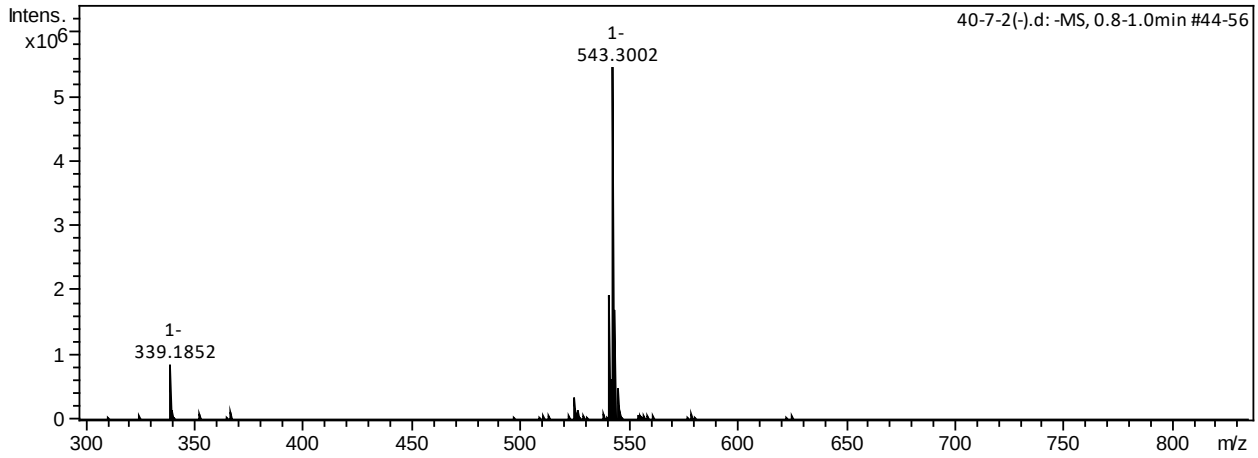

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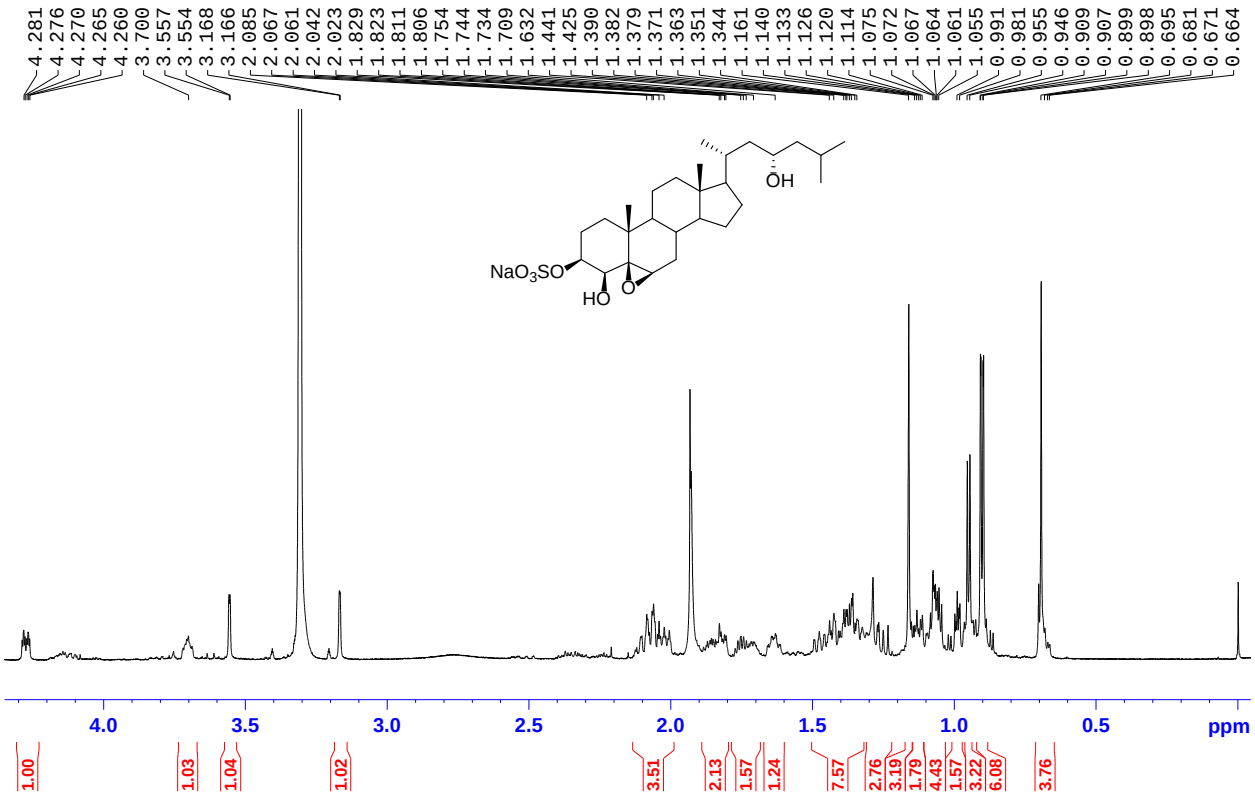

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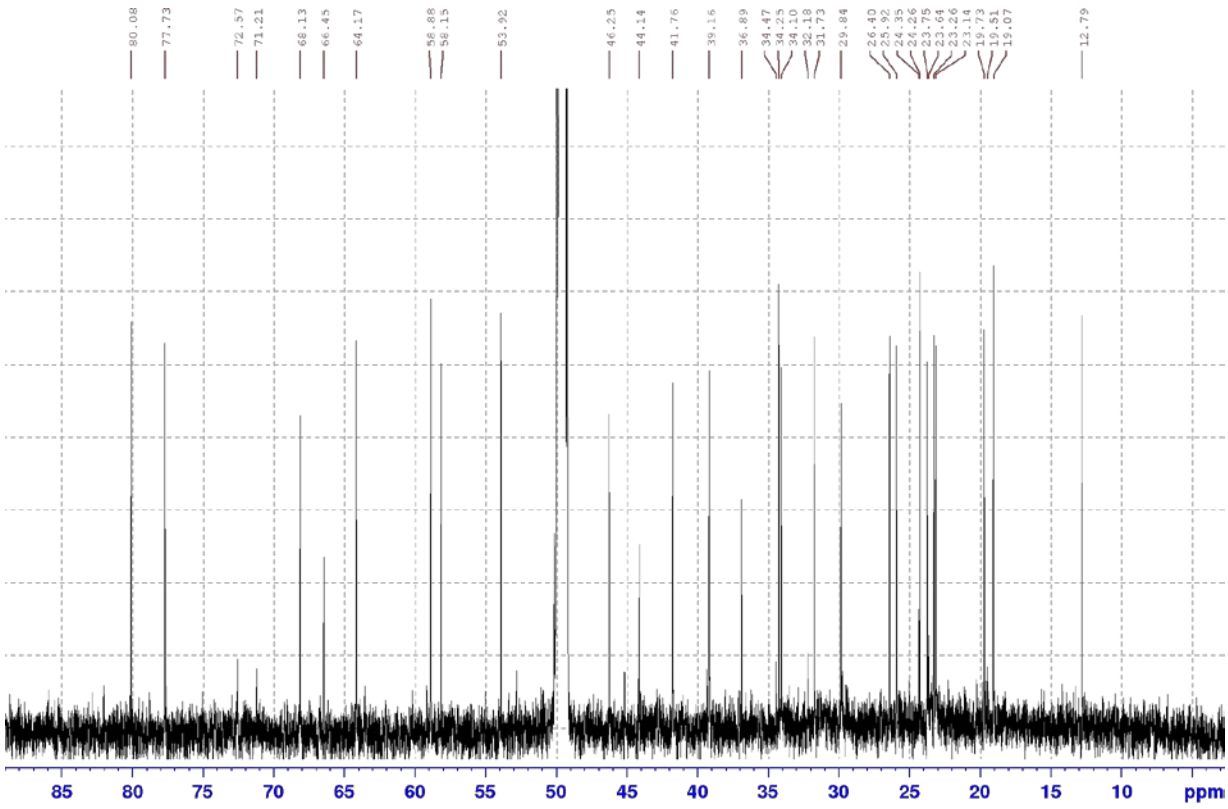

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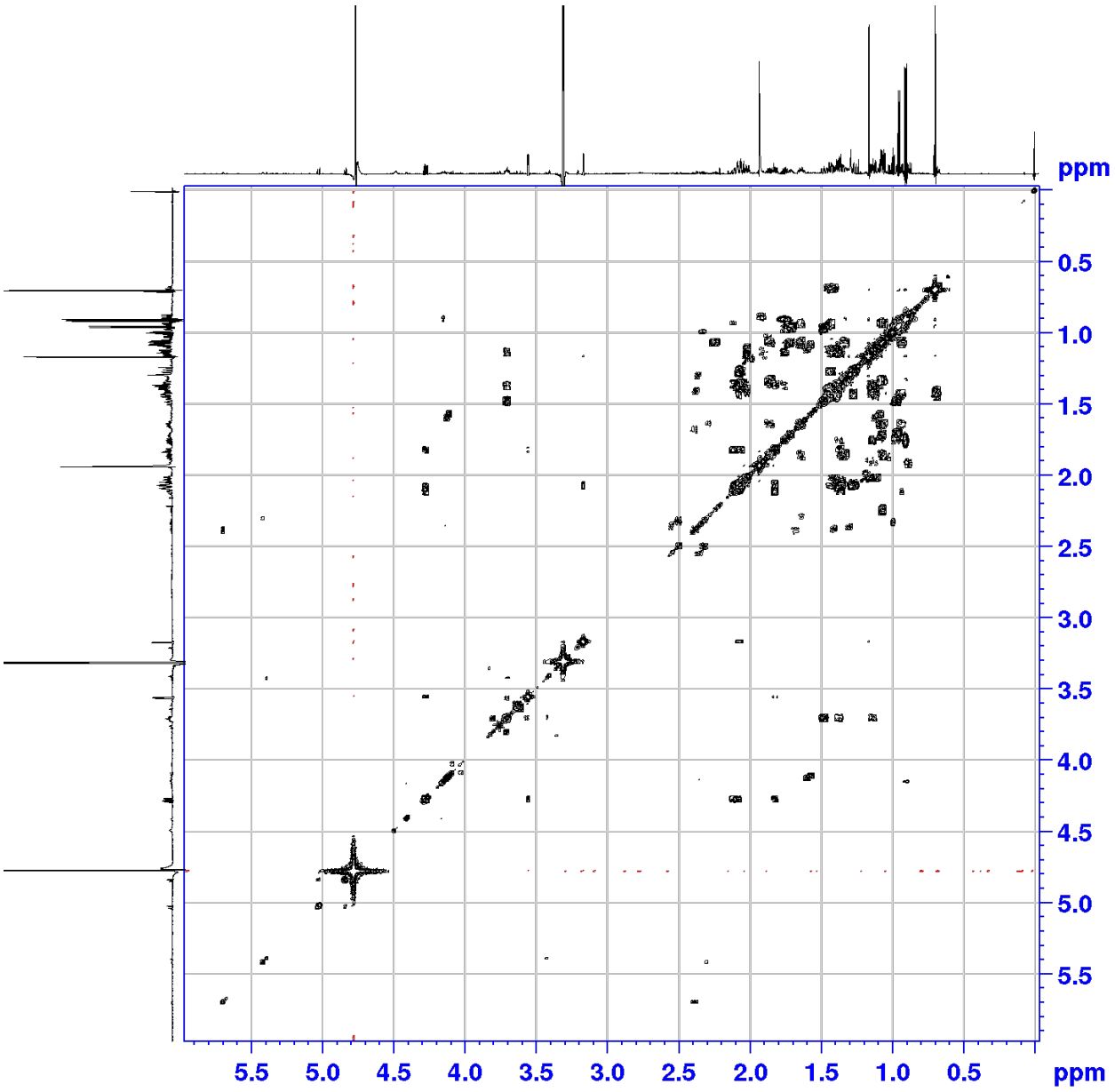

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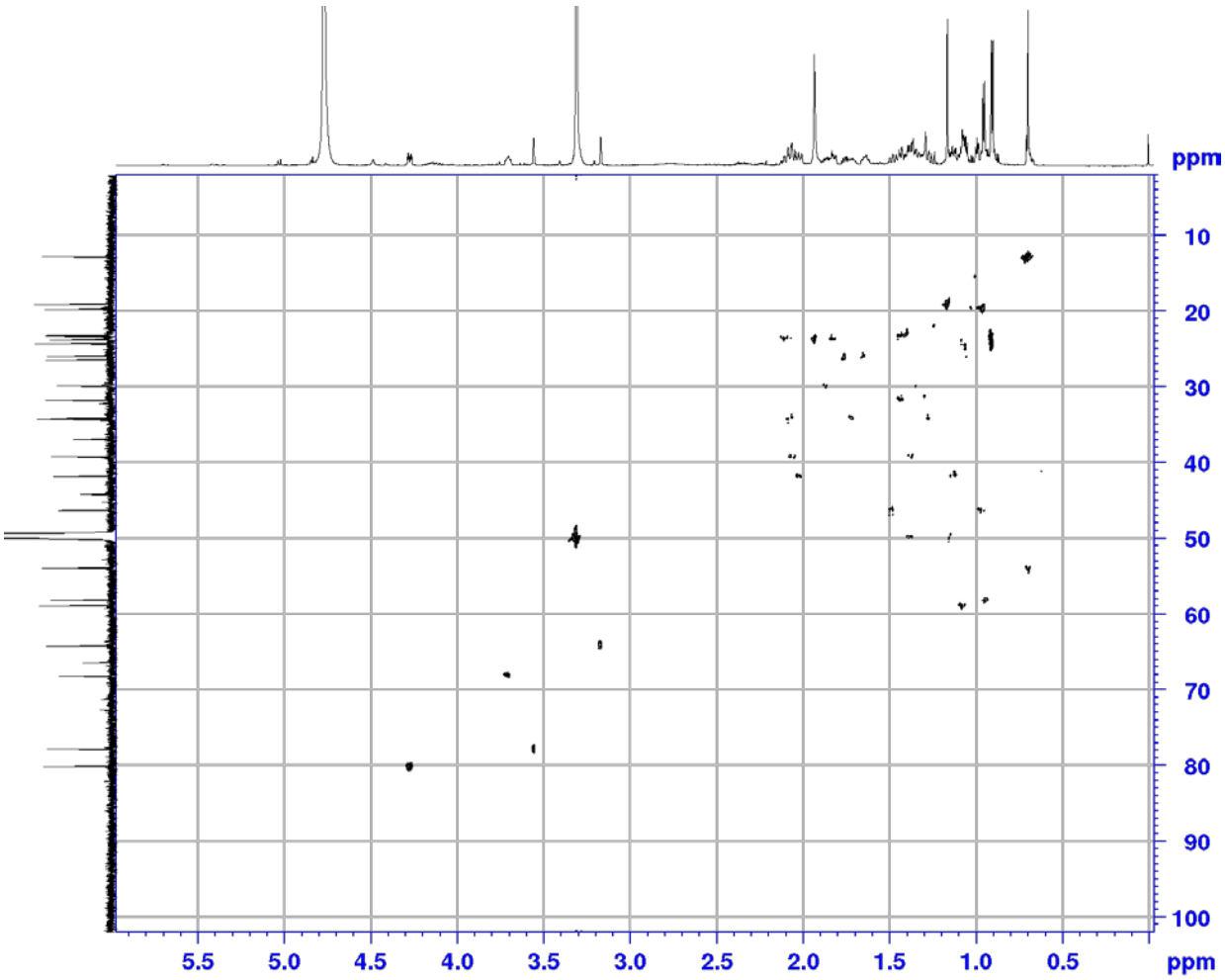

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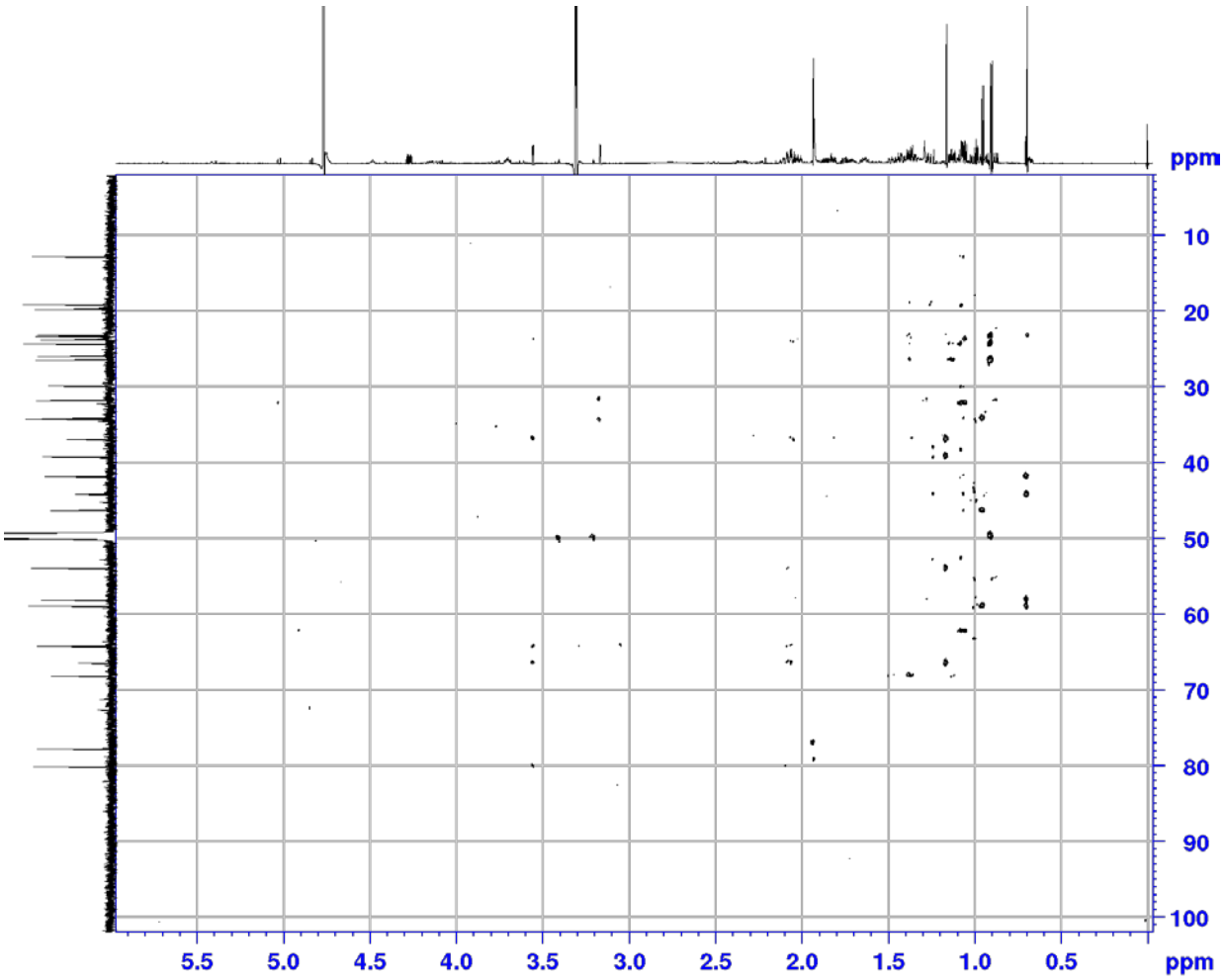

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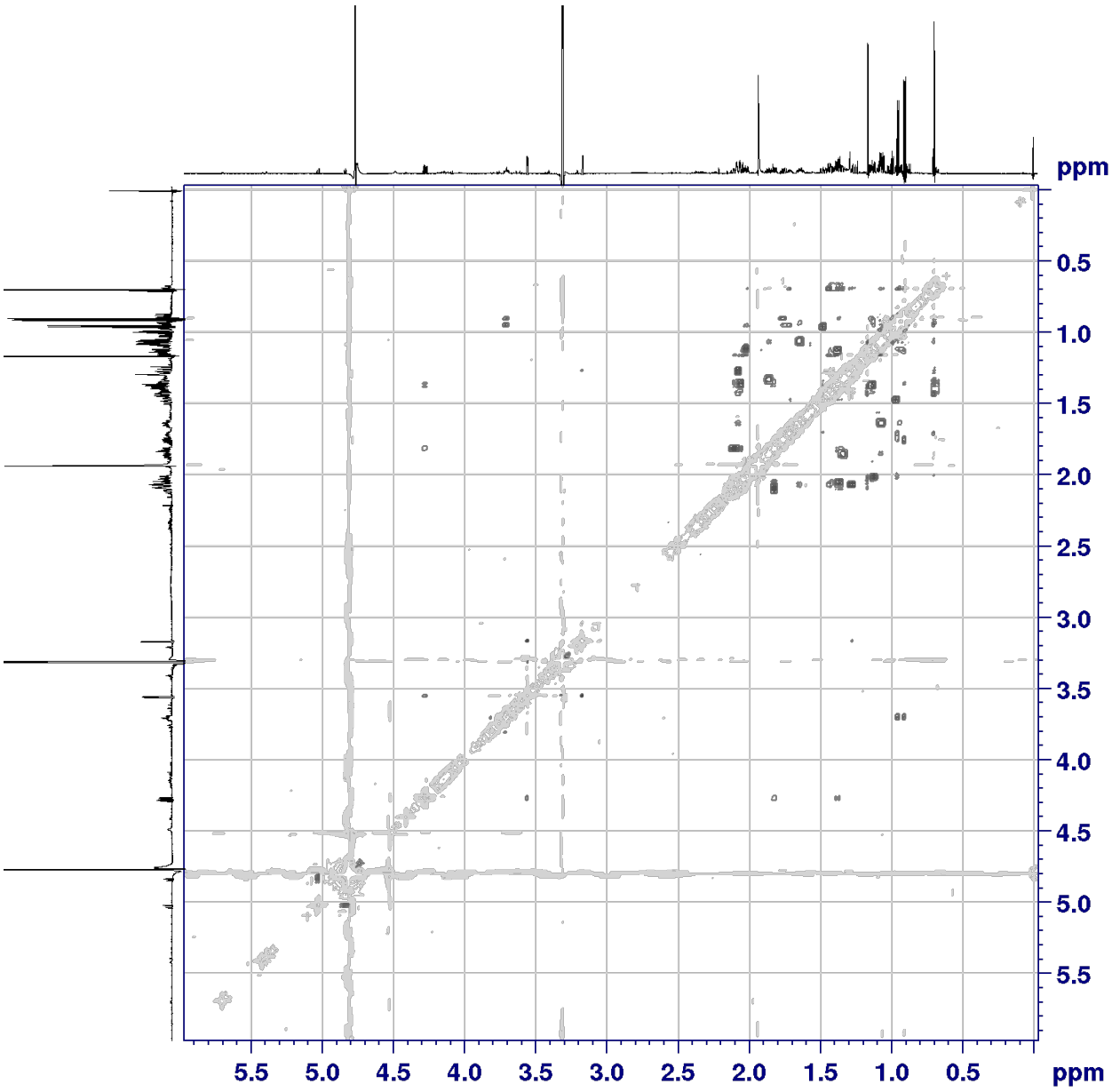

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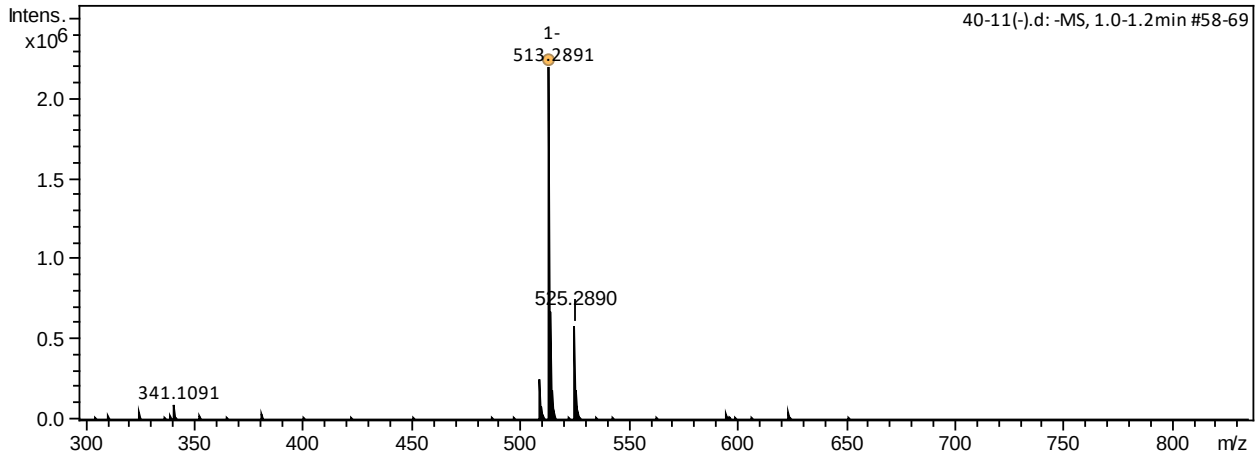
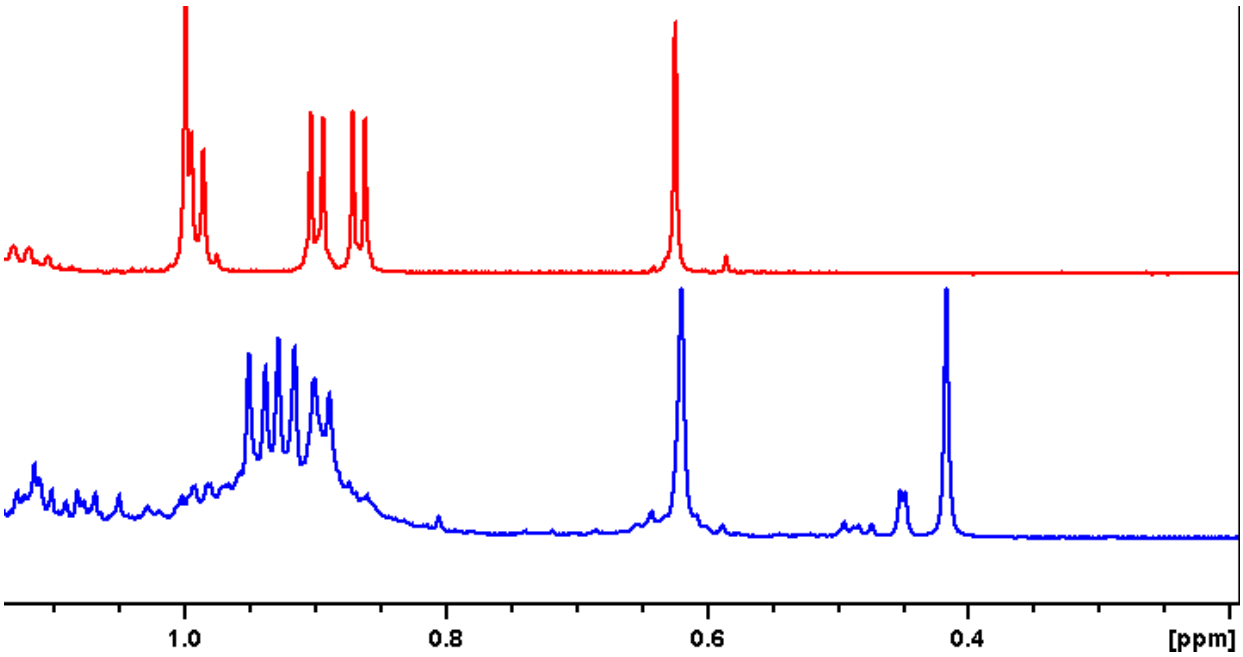


**Figure S26.** $^{1}$H NMR chemical shift differences between **3S** (red) and **3R** (blue)



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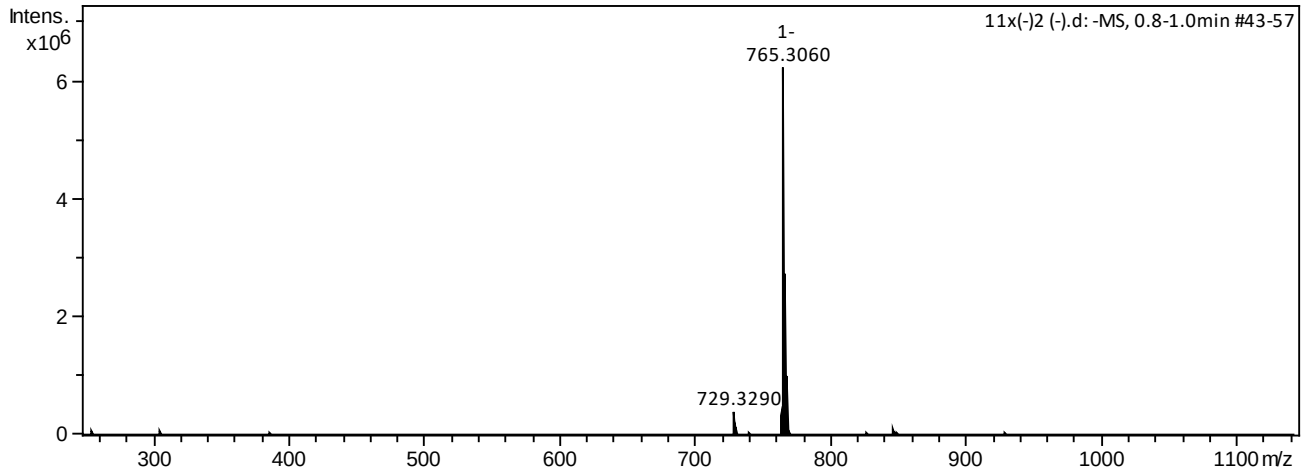

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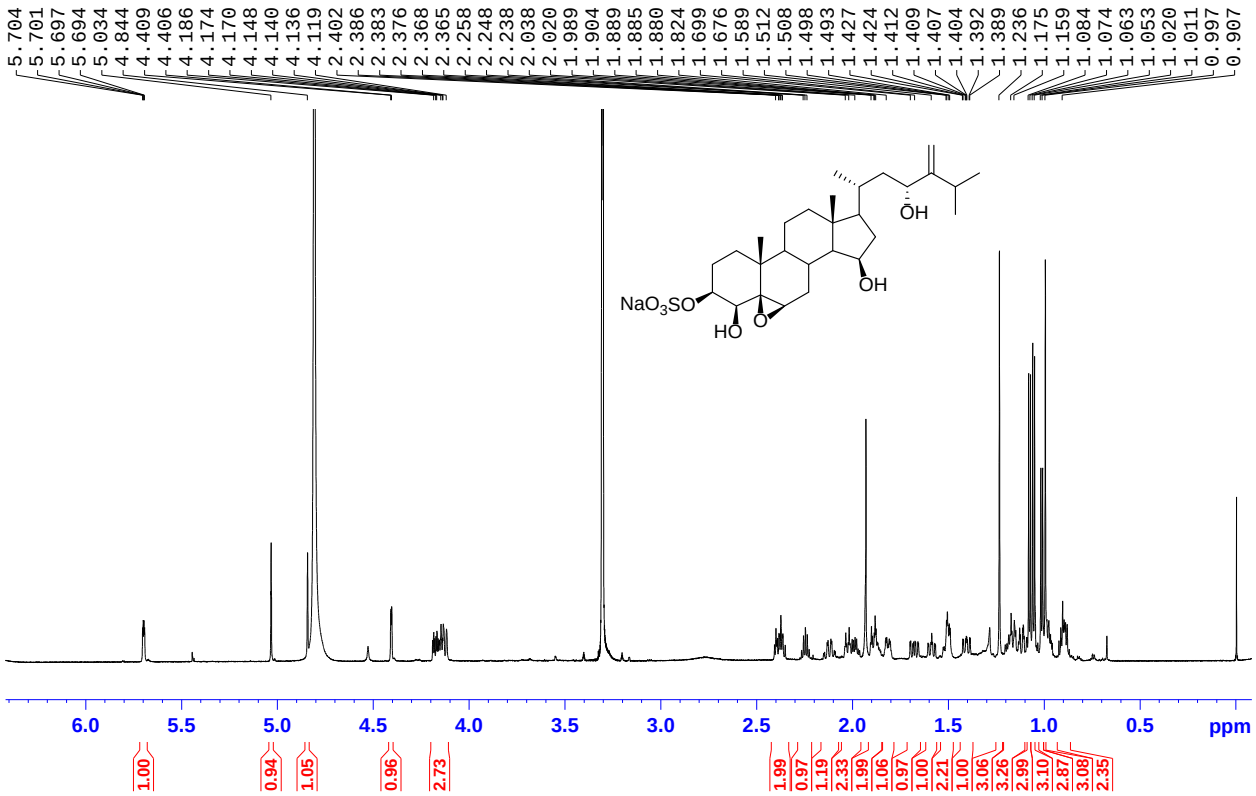

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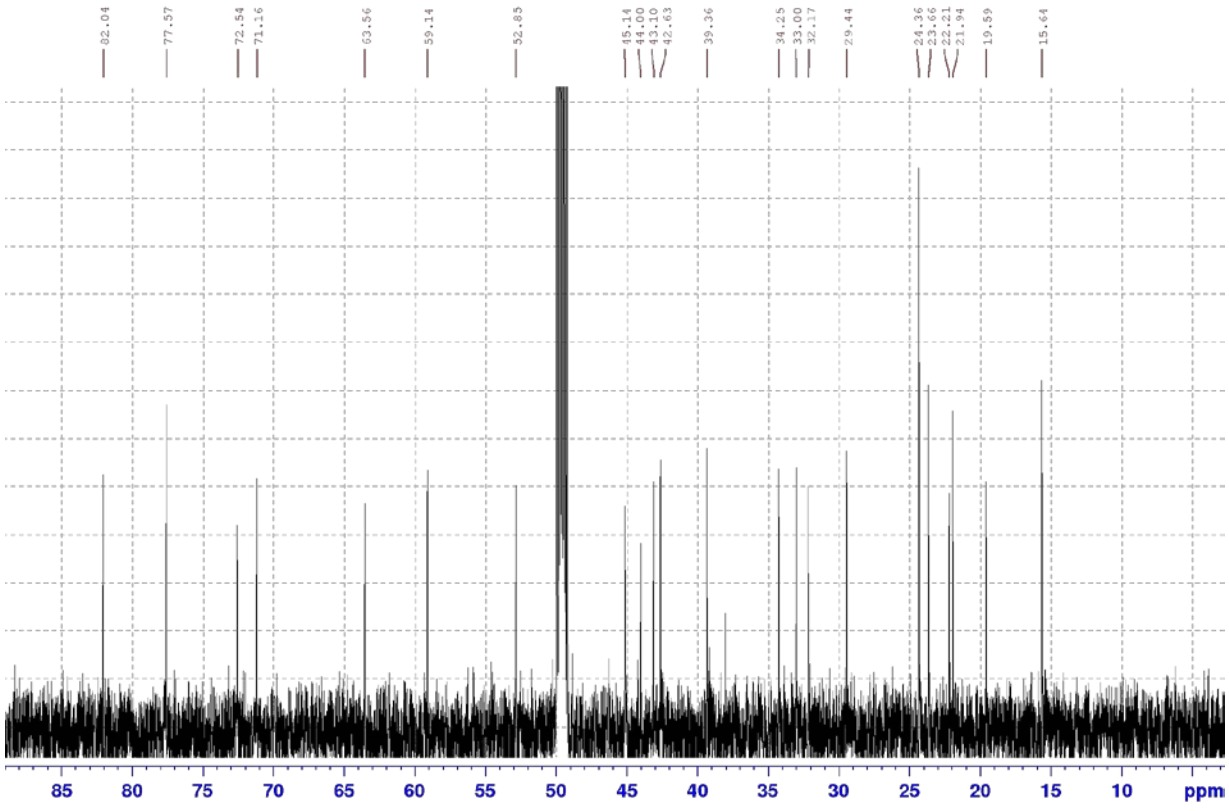

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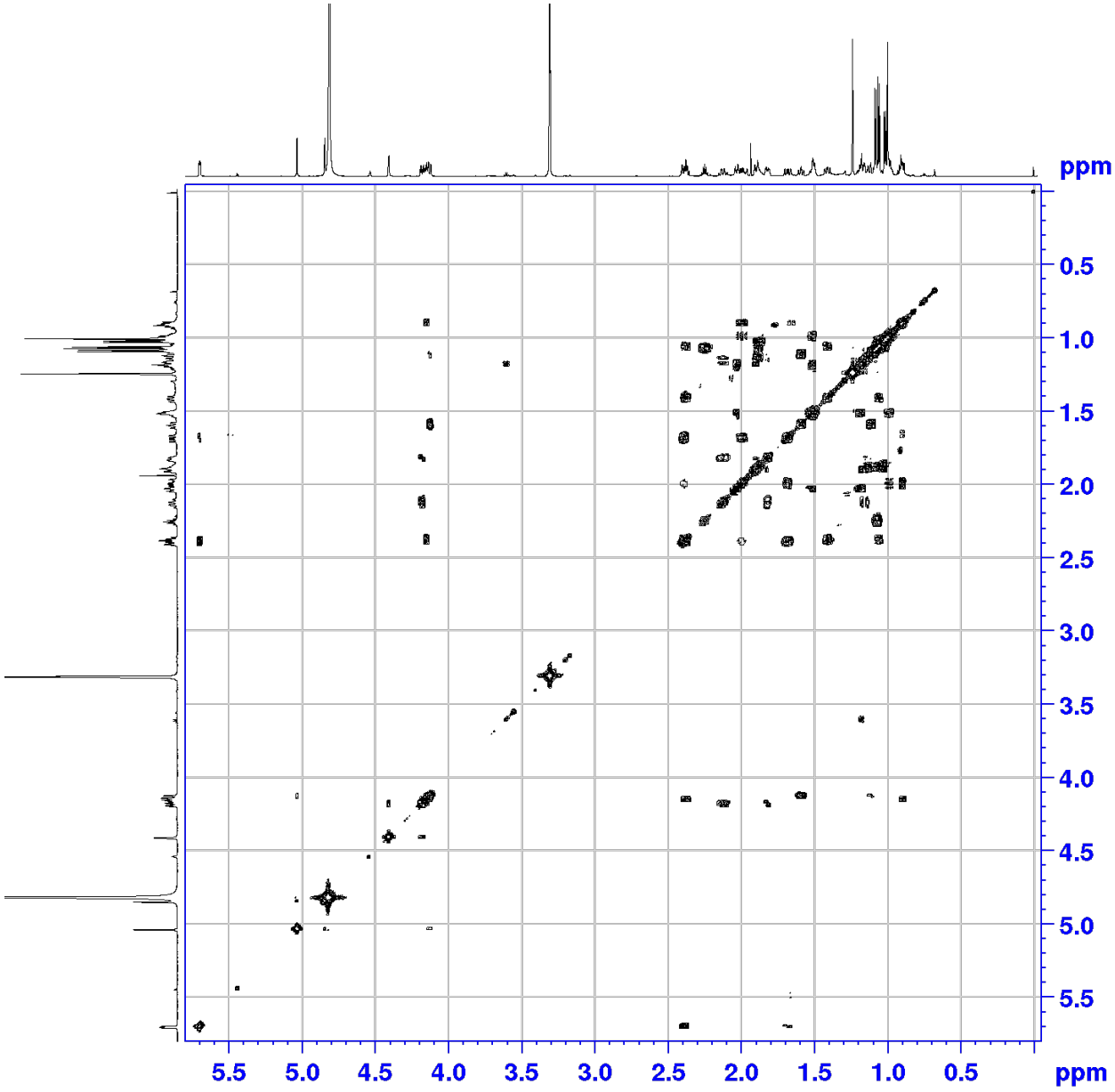

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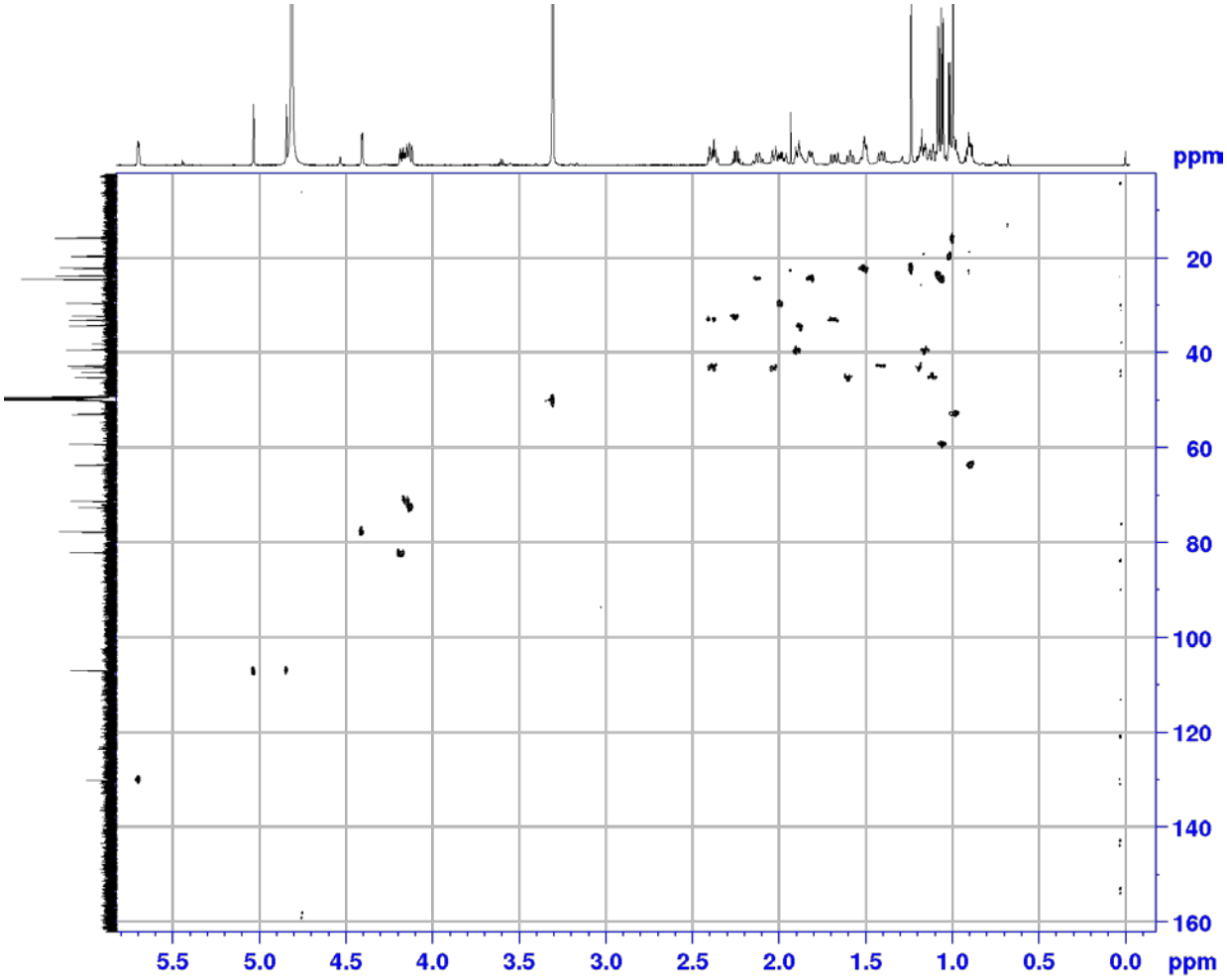

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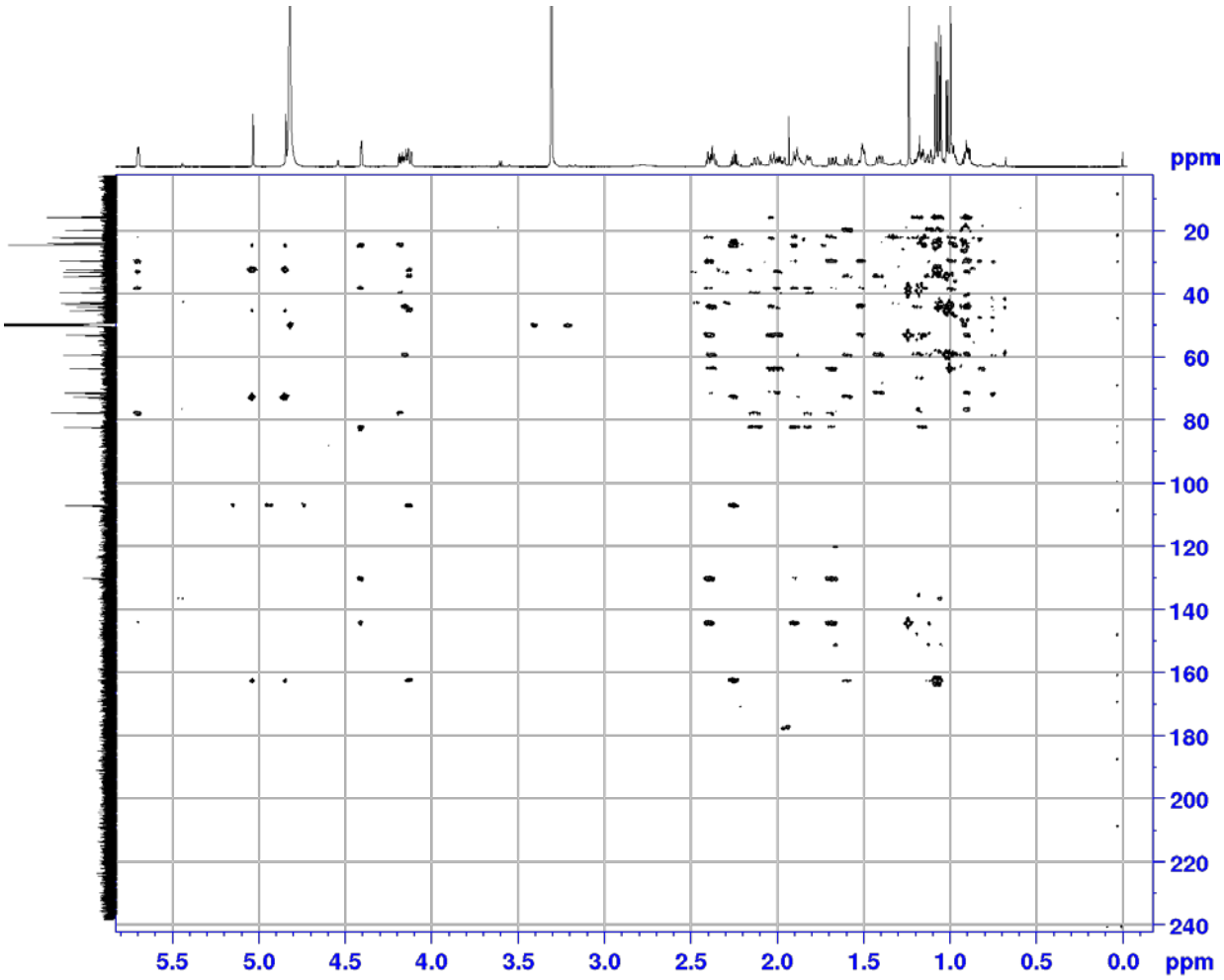
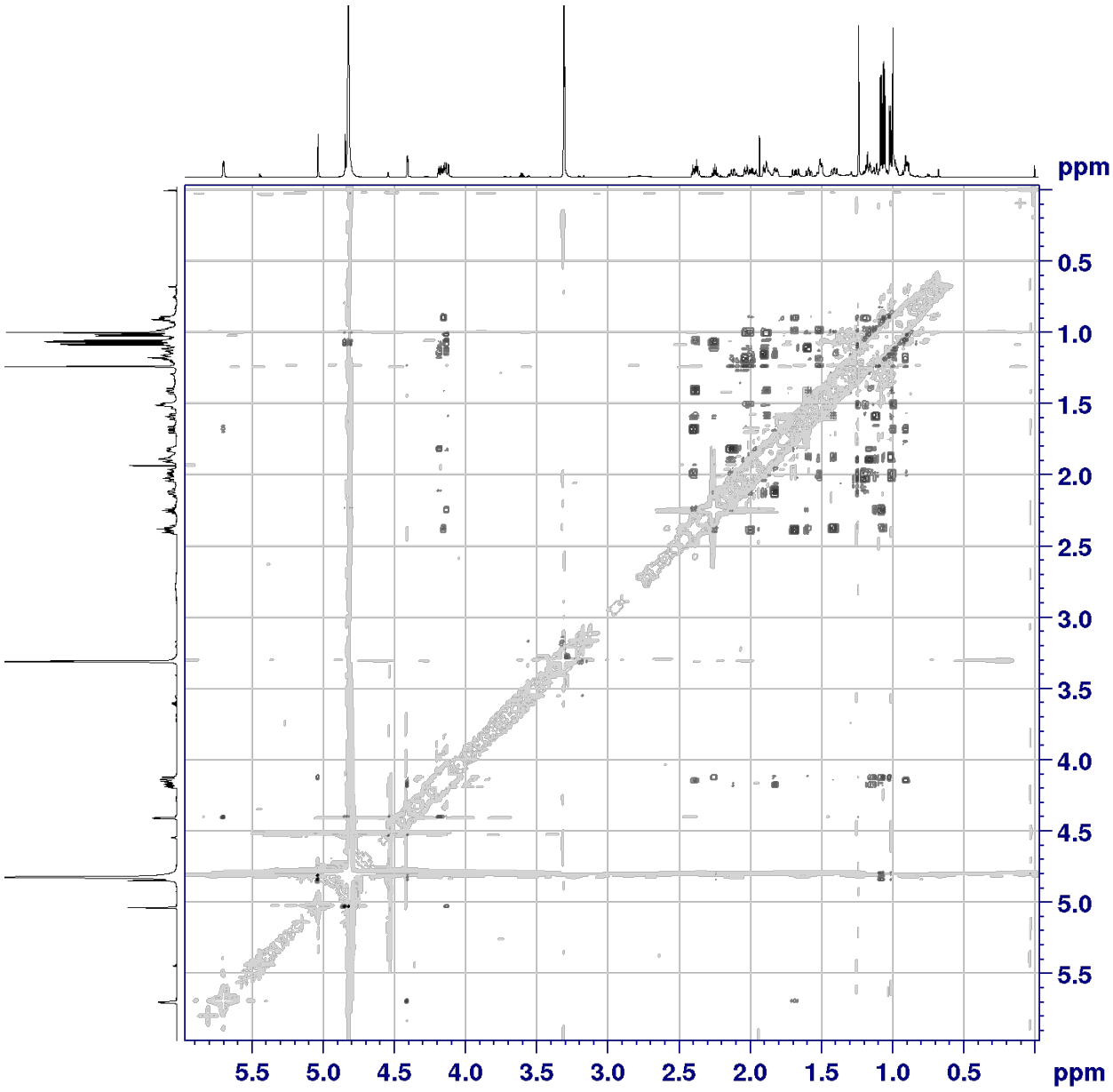


**Figure S33.** The NOESY (700 MHz, $CD_3OD$) spectrum of compound **4**



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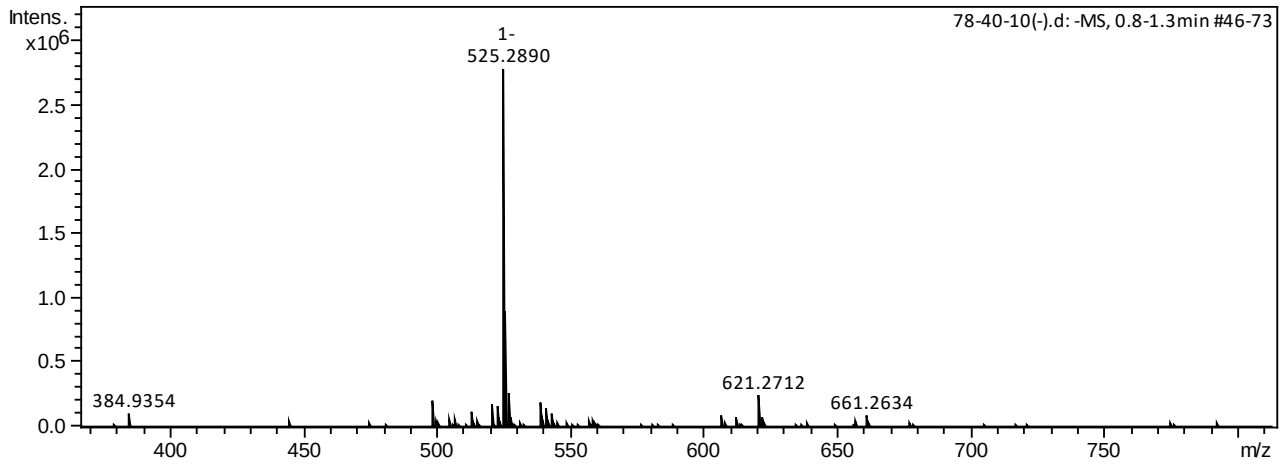


**Figure S35.** $^{1}$H NMR chemical shift differences between **4S** (red) and **4R** (blue)

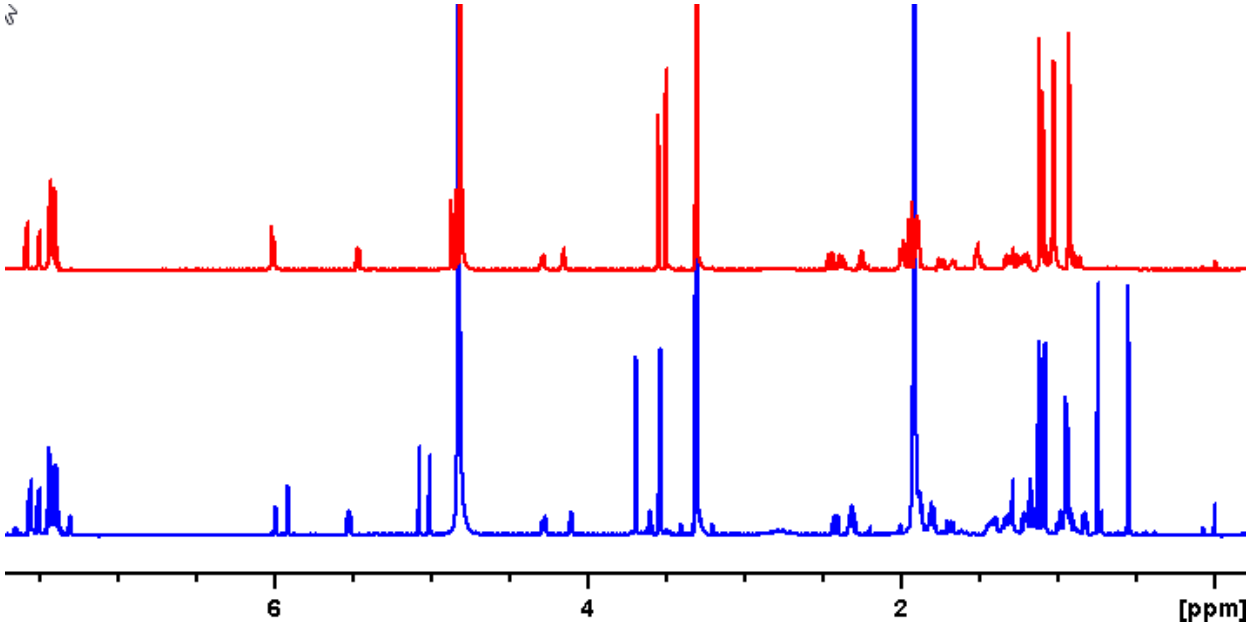


**Figure S36.** The HRESIMS spectrum of compound **4R** (**4S**)

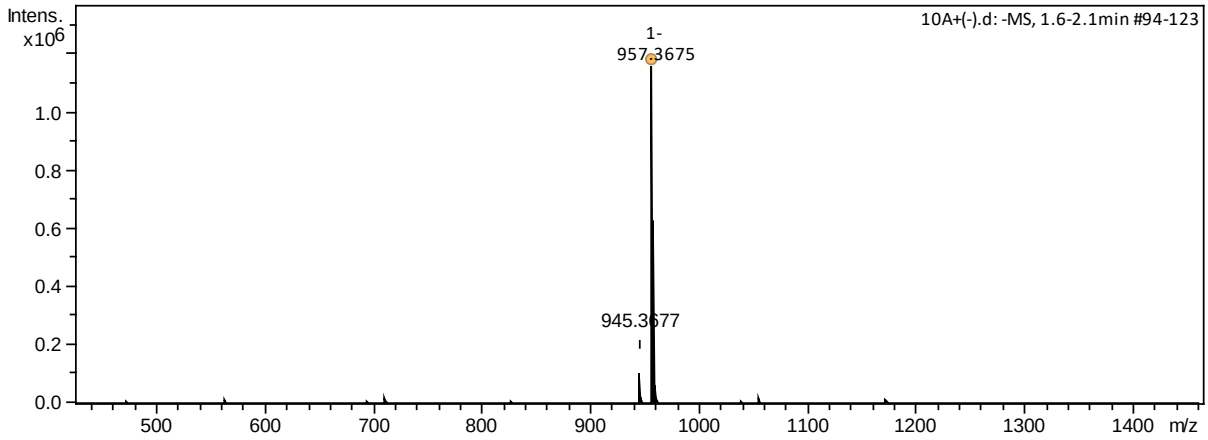


**Figure S37.** The $^{1}H$ NMR (500 MHz, $CD_3OD$) spectrum of compound **5**

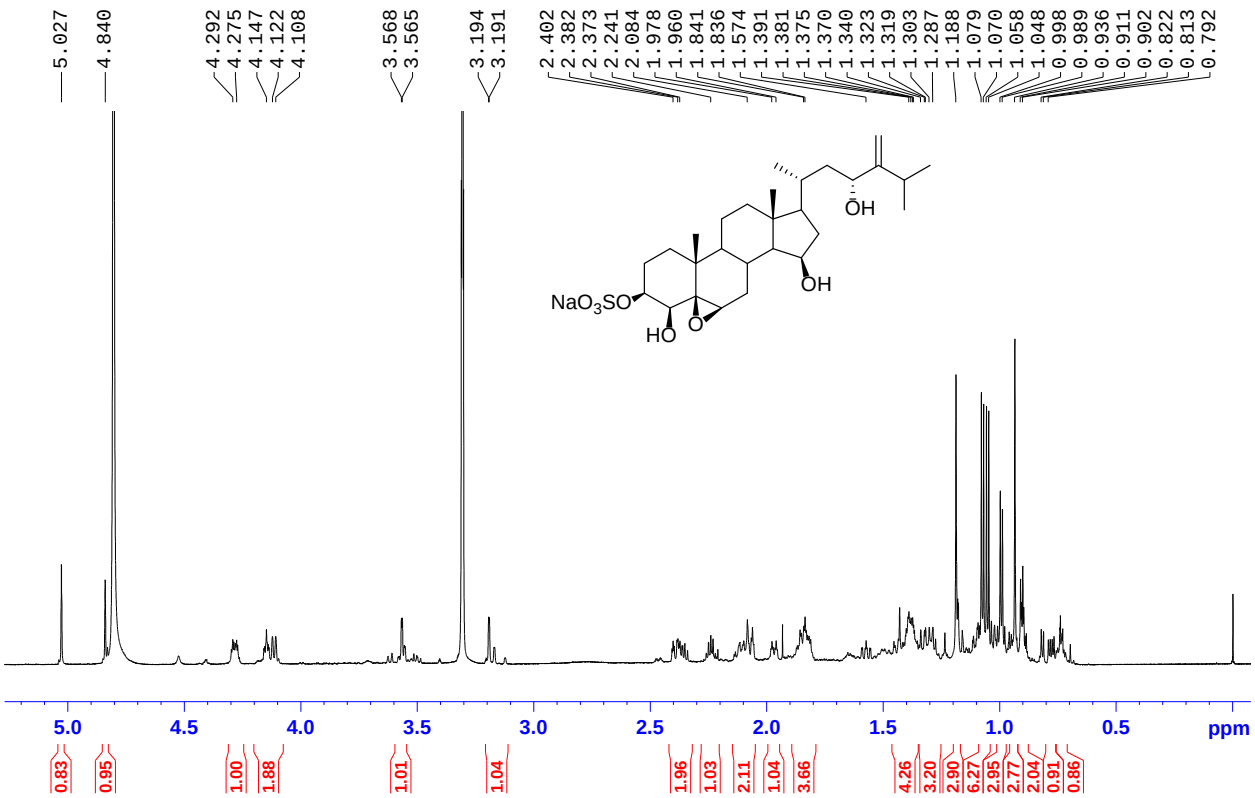
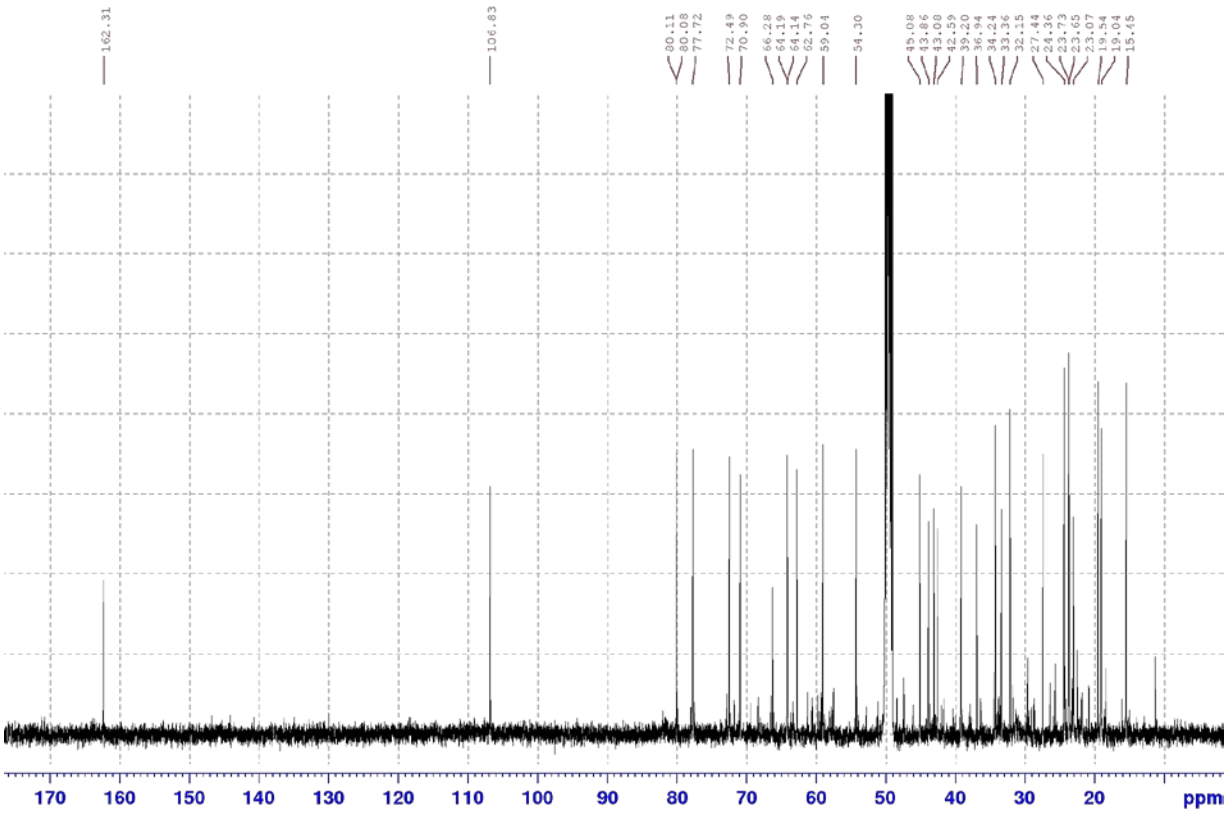


**Figure S38.** The $^{13}C$ NMR (125 MHz, $CD_3OD$) spectrum of compound **5**



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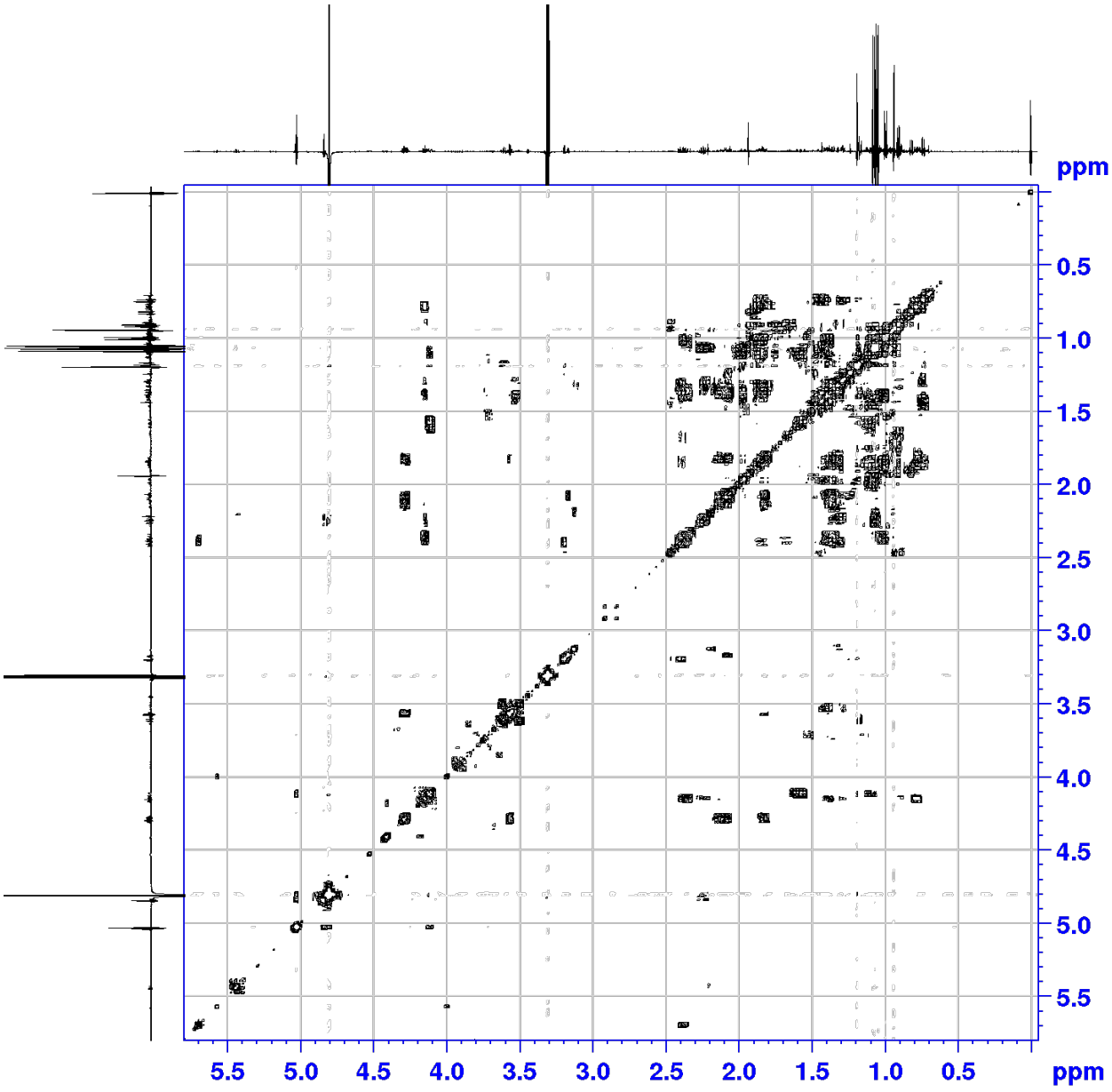
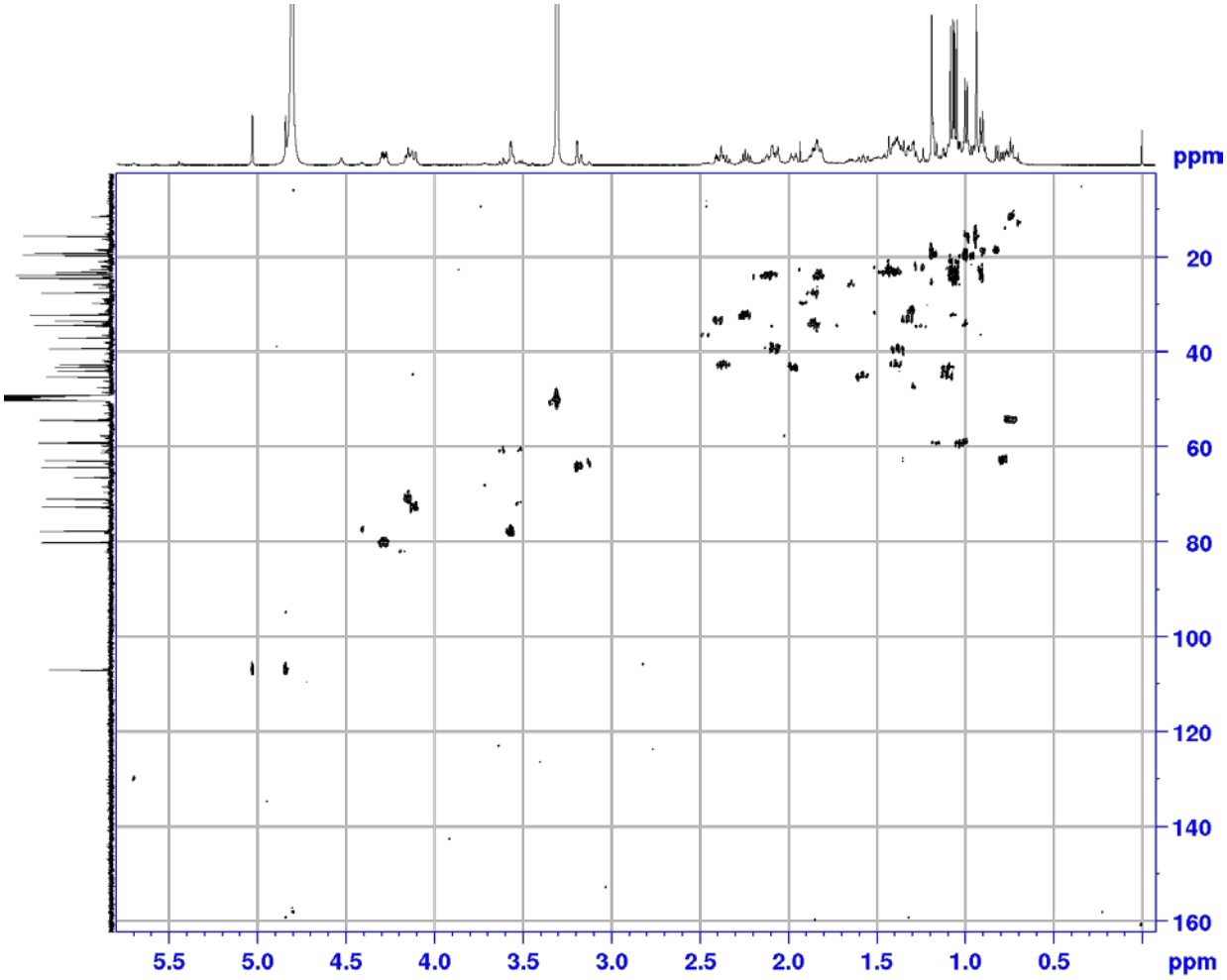


**Figure S40.** The HSQC (500/125 MHz, $CD_3OD$) spectrum of compound **5**



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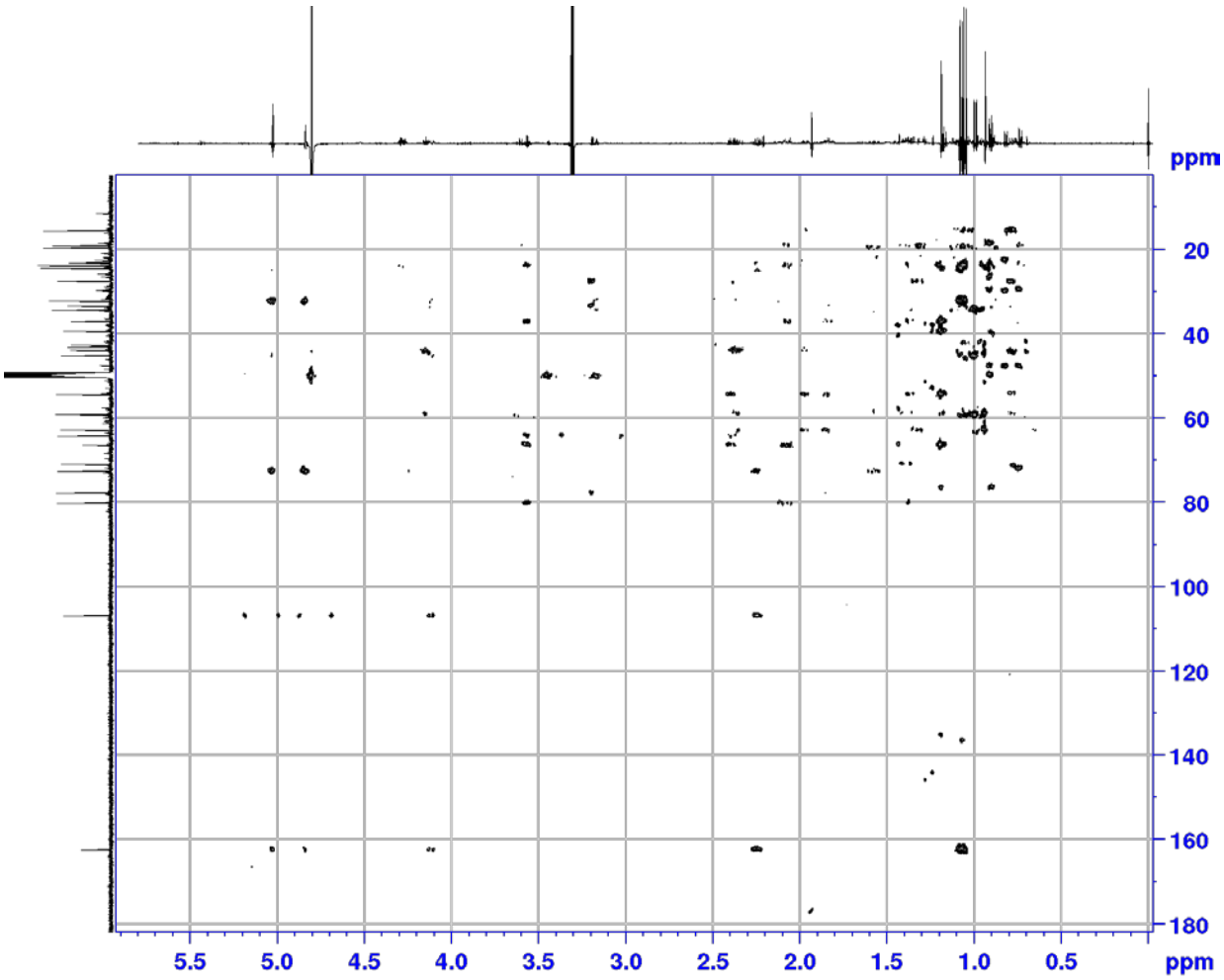


**Figure S42.** The NOESY (500 MHz, $CD_3OD$) spectrum of compound **5**

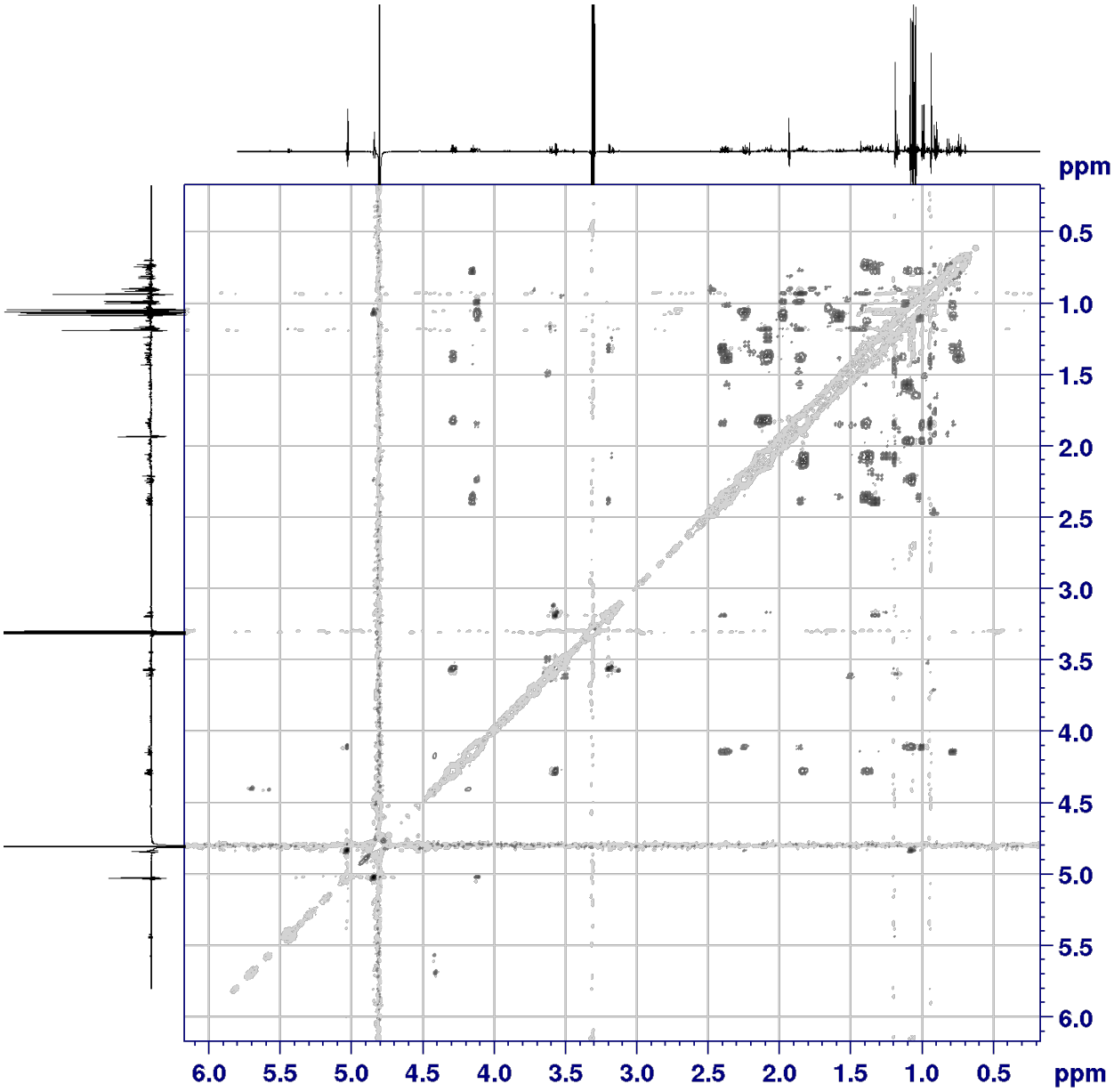


**Figure S43.** The HRESIMS spectrum of compound **5**

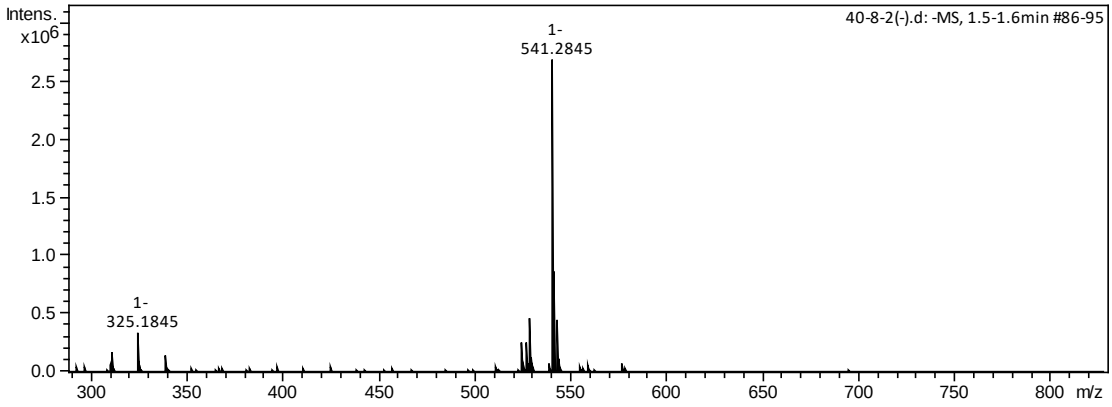
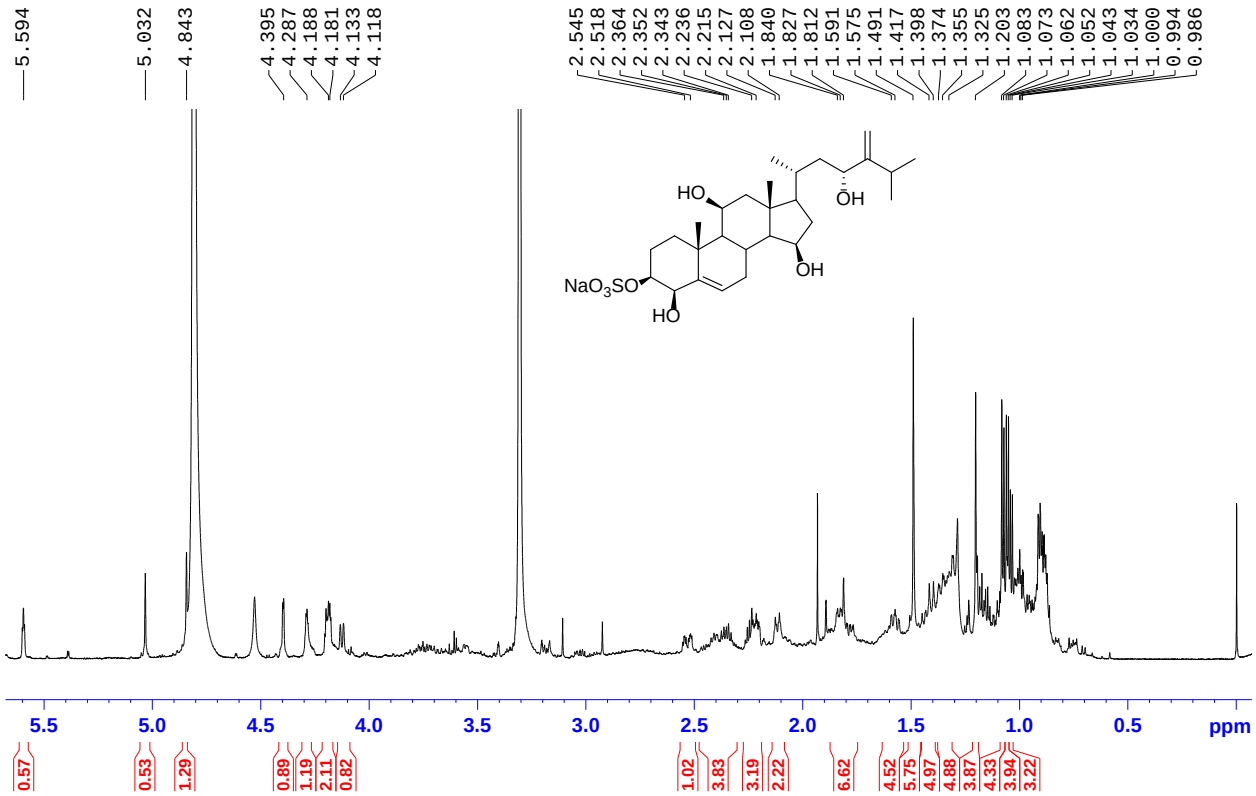


**Figure S44.** The $^{1}H$ NMR (700 MHz, $CD_3OD$) spectrum of compound **6**



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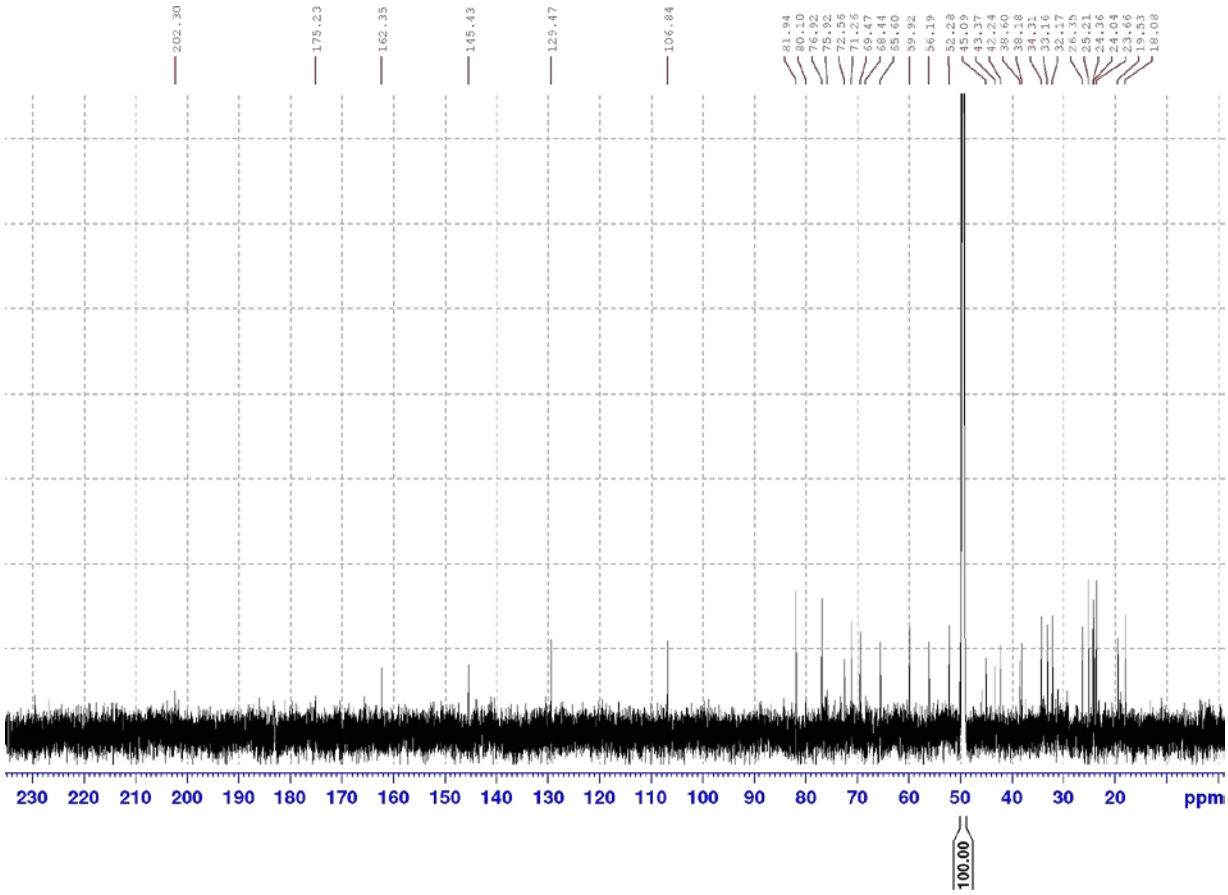

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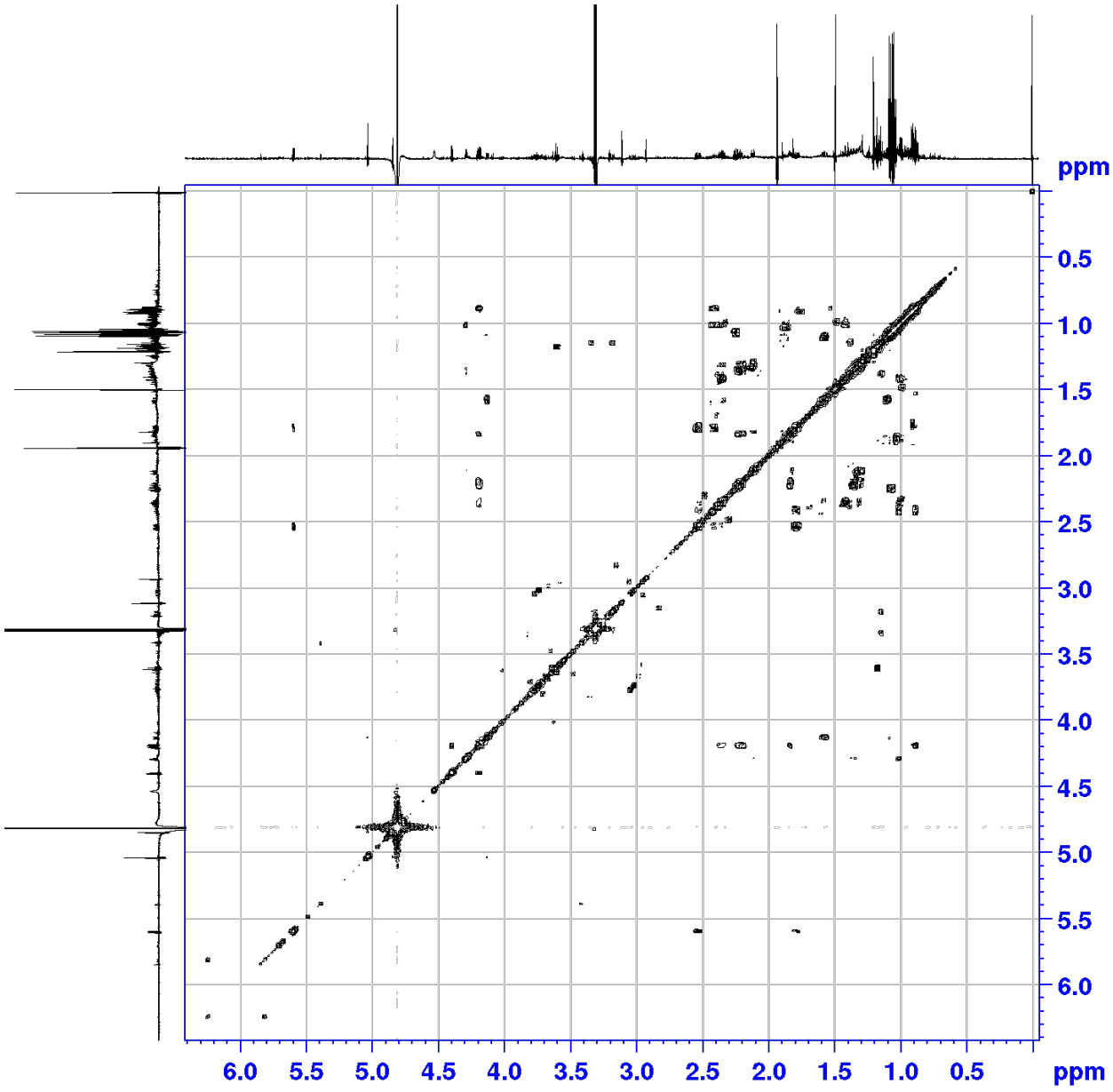

**Figure S47.** The HSQC (700/175 MHz, $CD_3OD$) spectrum of compound **6**

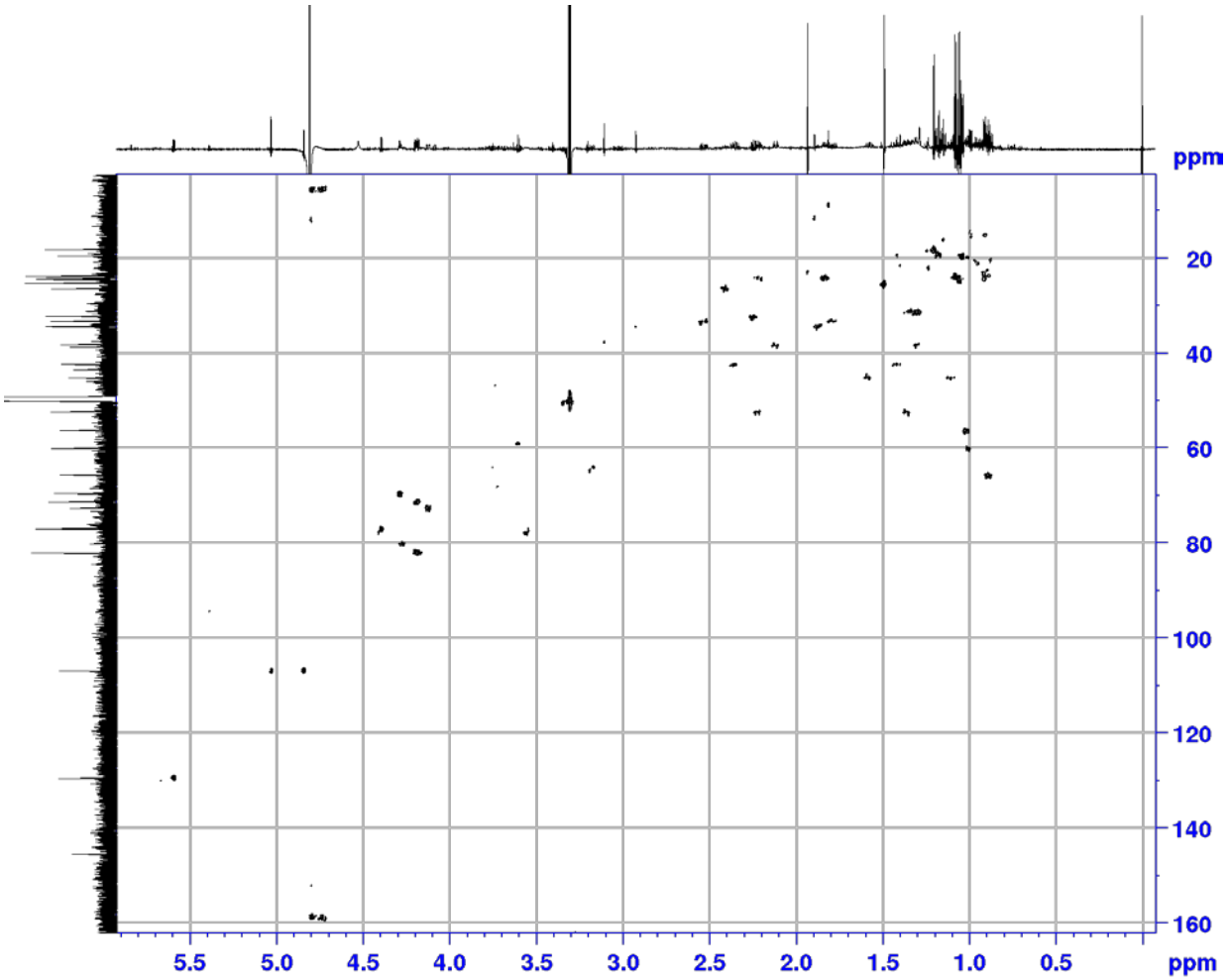

**Figure S48.** The HMBC (700/175 MHz, $CD_3OD$) spectrum of compound **6**

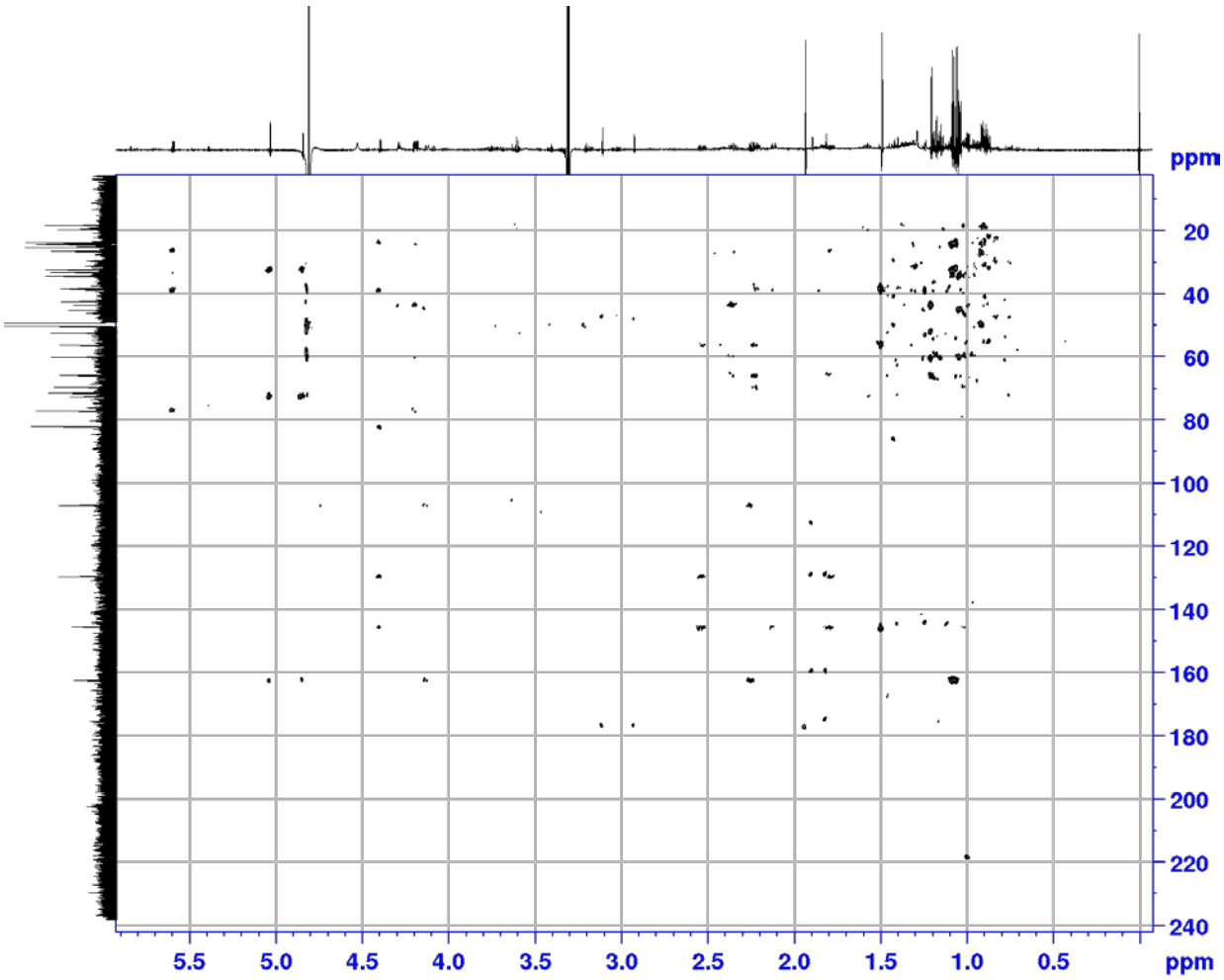

**Figure S49.** The NOESY (700 MHz, $CD_3OD$) spectrum of compound **6**

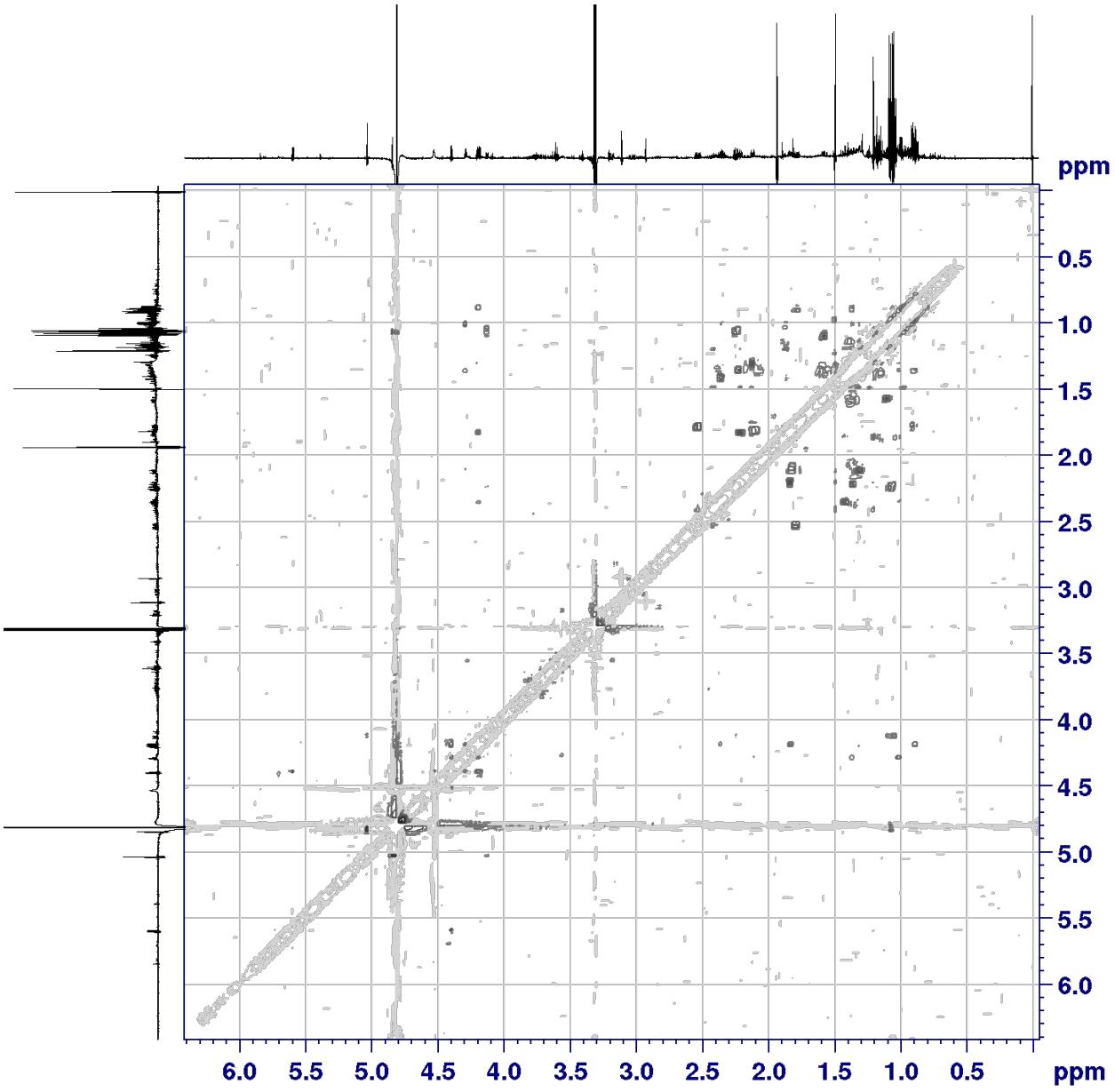

**Figure S50.** The HRESIMS spectrum of compound **6**

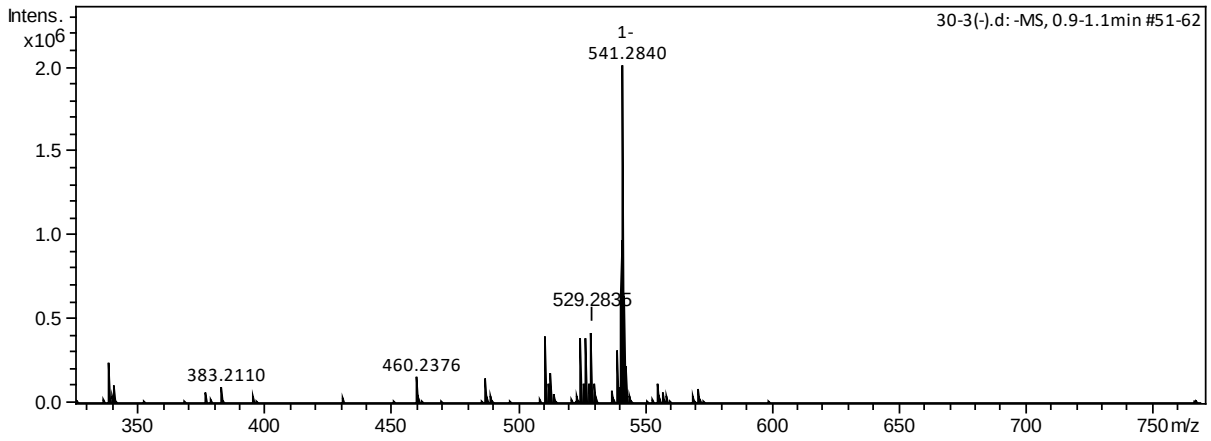

**Figure S51.** The $^{1}H$ NMR (700 MHz, $CD_3OD$) spectrum of compound **7**

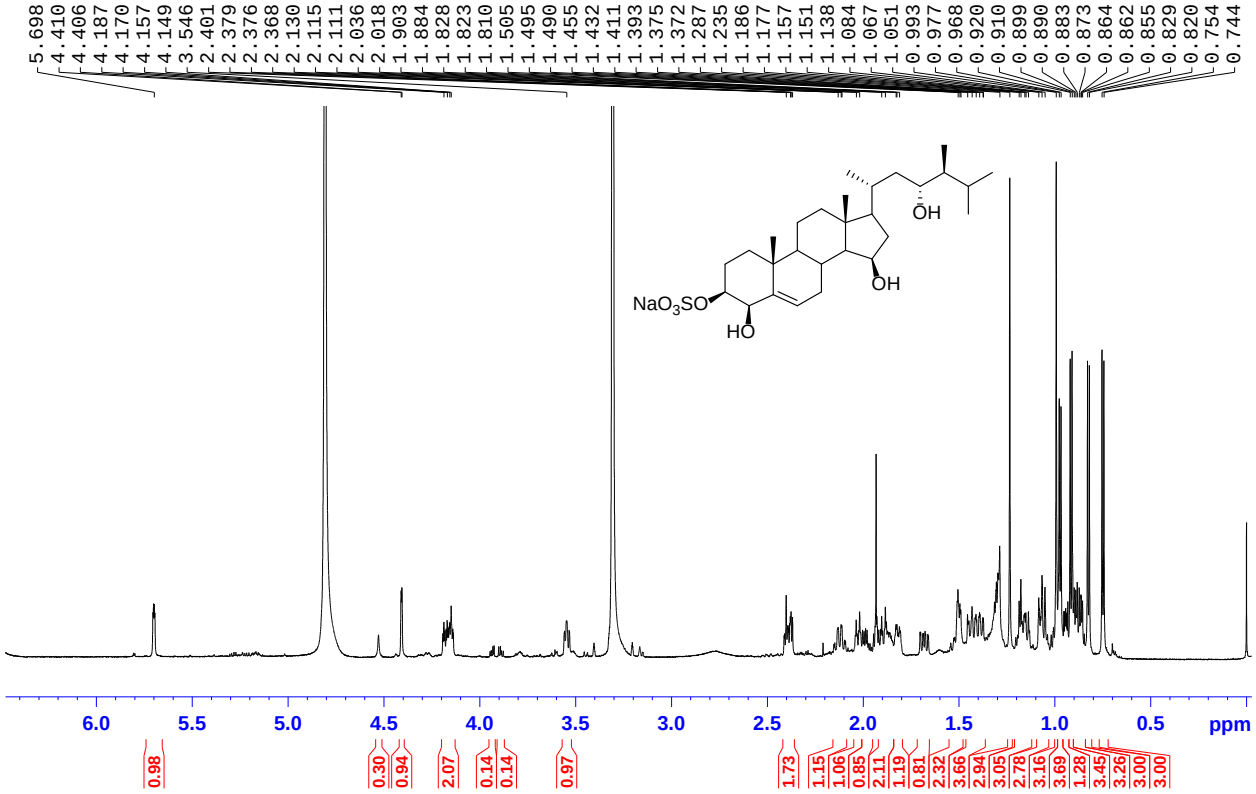

**Figure S52.** The $^{13}C$ NMR (175 MHz, $CD_3OD$) spectrum of compound **7**

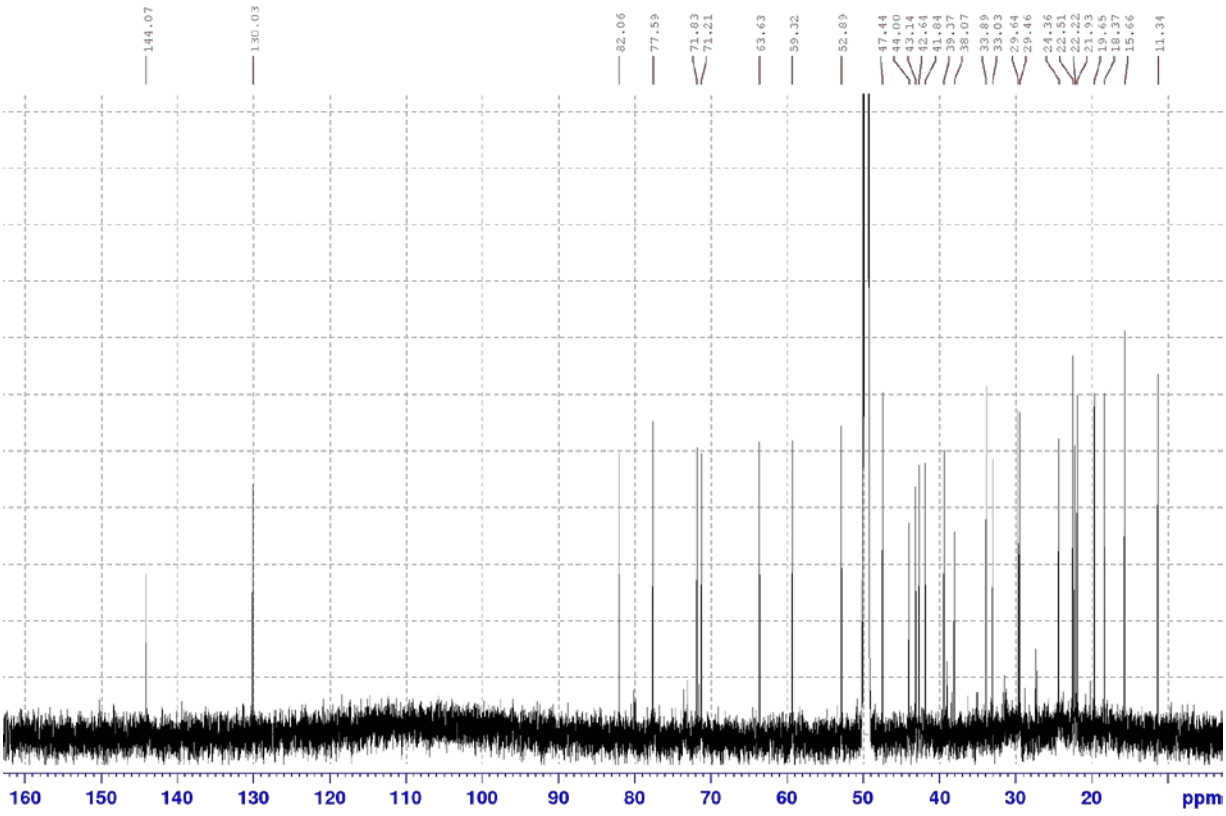

**Figure S53.** The COSY (700 MHz, $CD_3OD$) spectrum of compound **7**

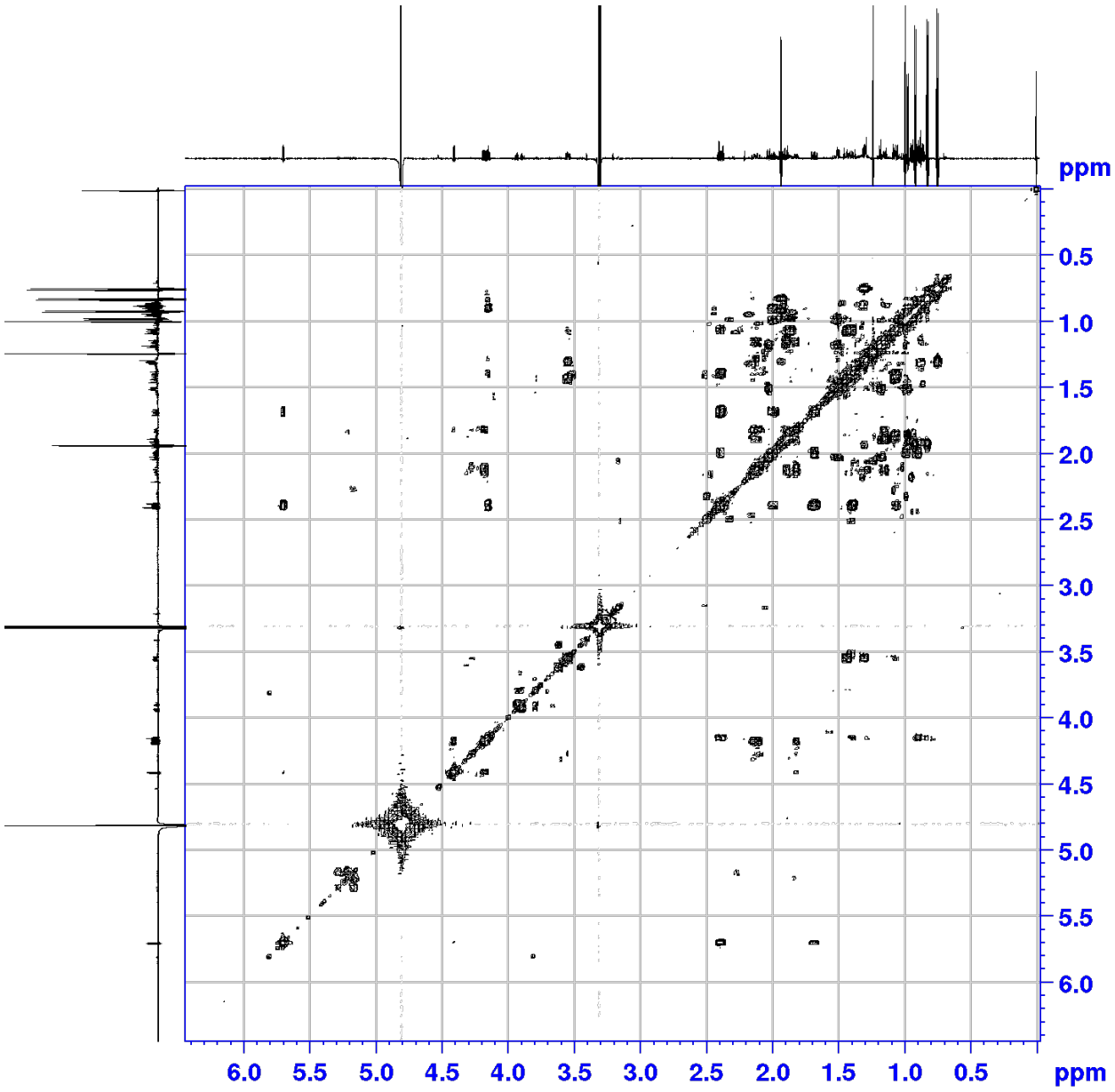

**Figure S54.** The HSQC (700/175 MHz, $CD_3OD$) spectrum of compound **7**

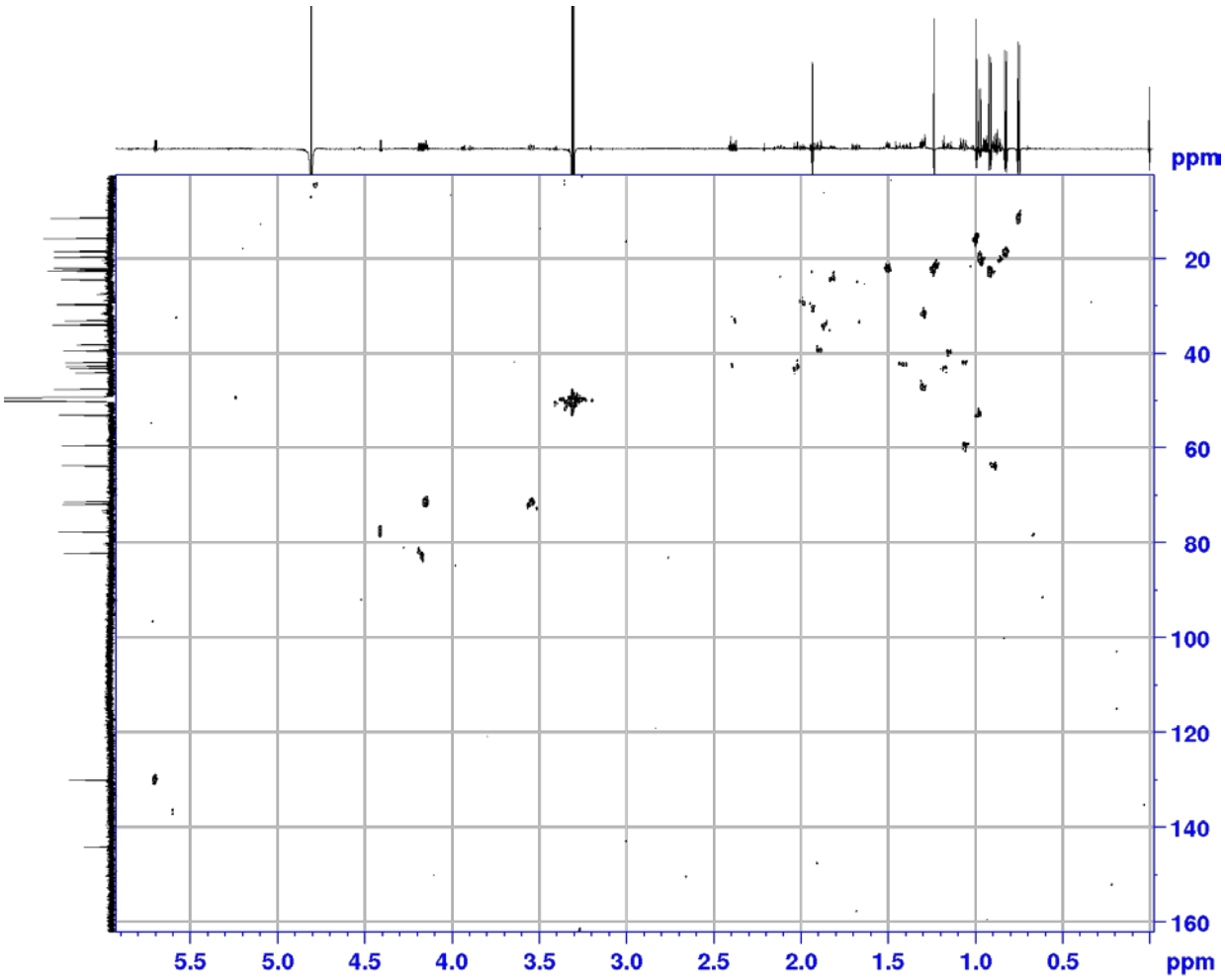

**Figure S55.** The HMBC (700/175 MHz, $CD_3OD$) spectrum of compound **7**

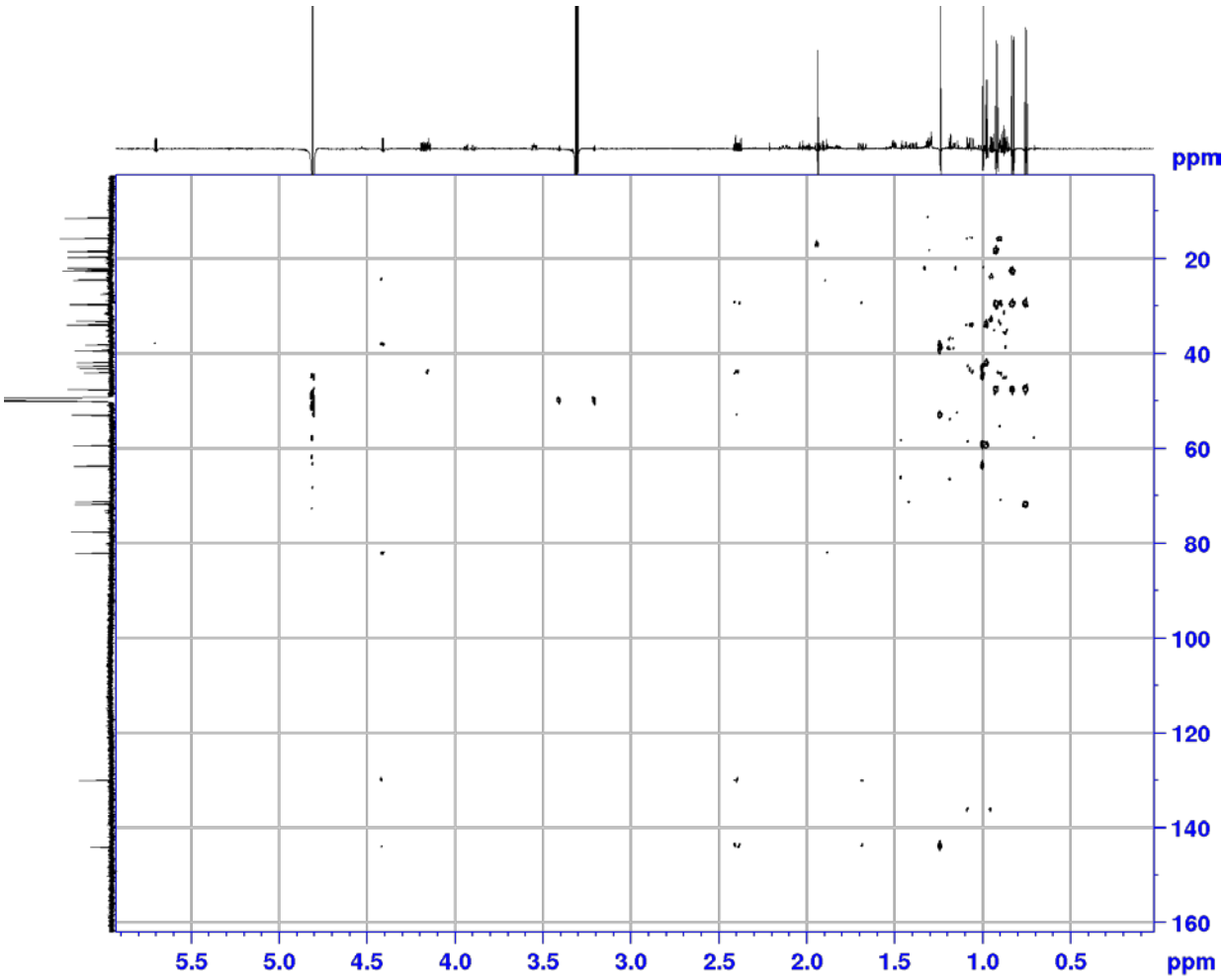

**Figure S56.** The NOESY (700 MHz, $CD_3OD$) spectrum of compound **7**

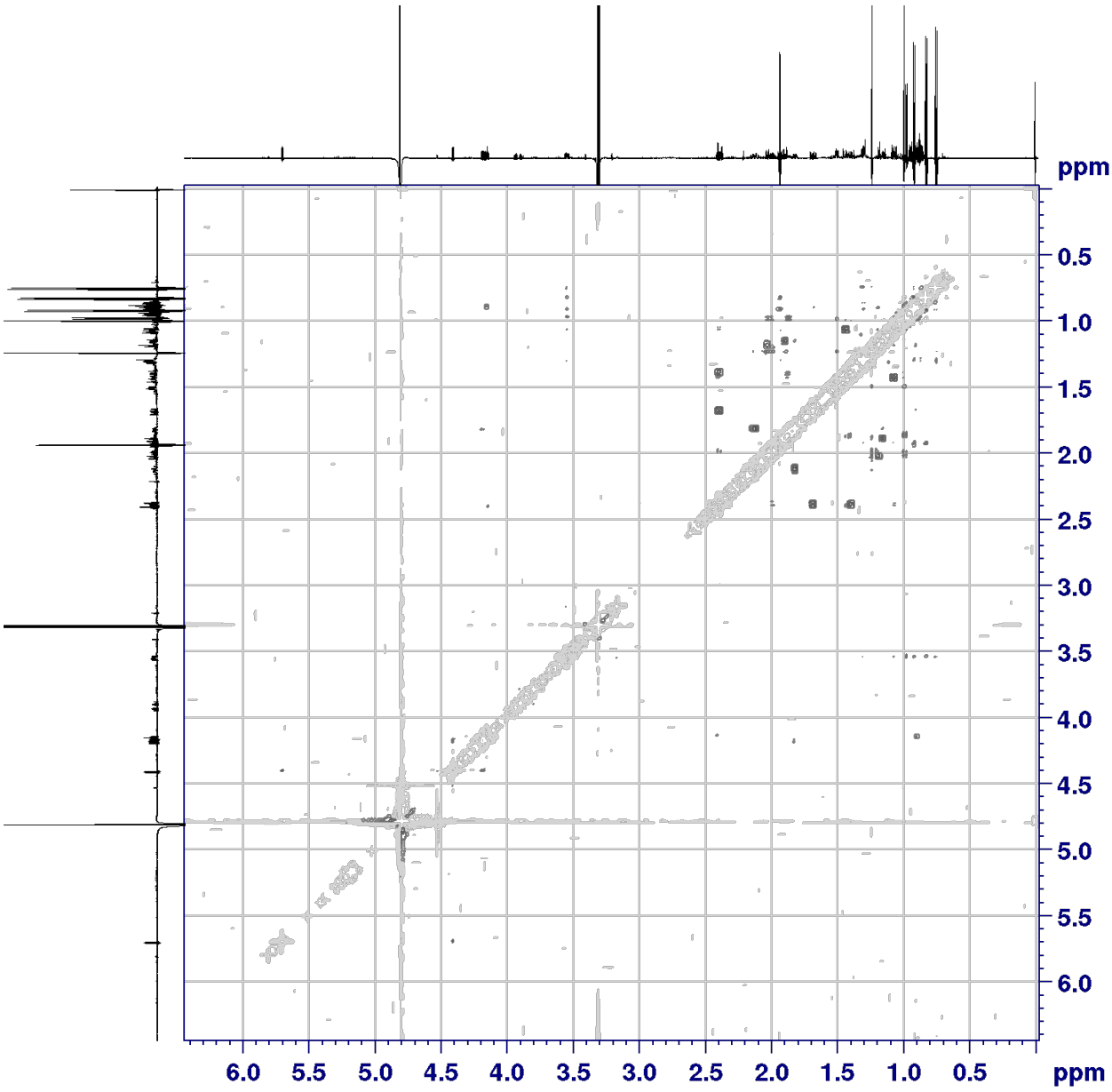

**Figure S57.** The HRESIMS spectrum of compound **7**

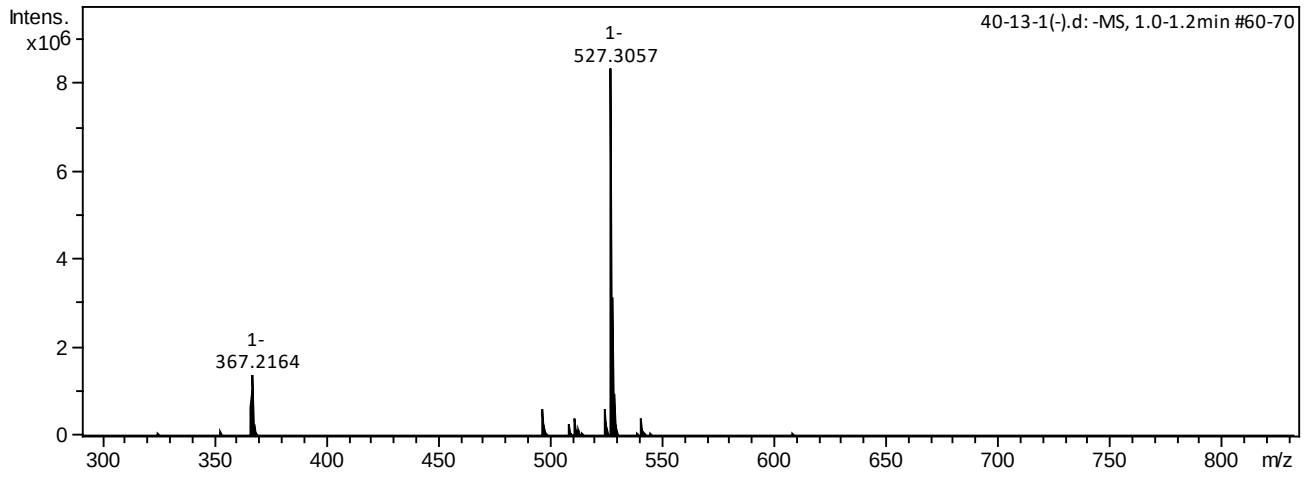

**Figure S58.** Photo of the sponge *Haliclona gracilis*.