Support Information

Azaphilones from the Red Sea Fungus *Aspergillus falconensis*

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Figure S1. UV spectrum of compound **1**.

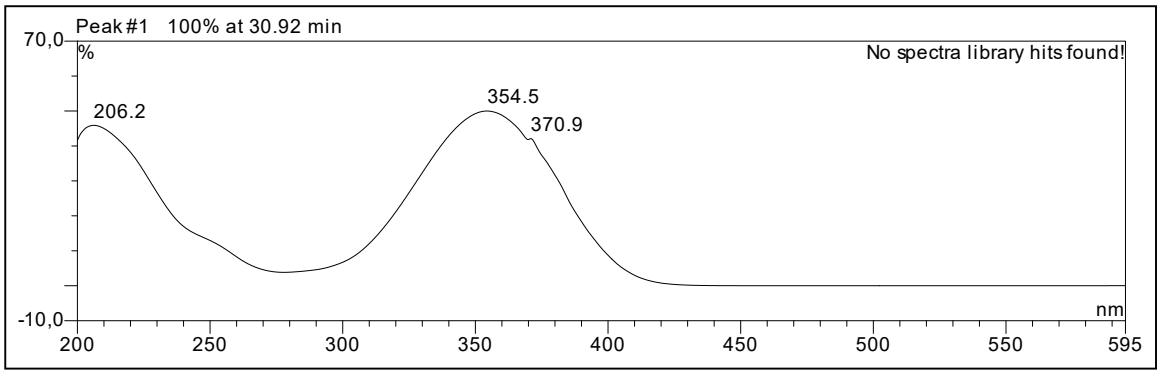

Figure S2. HRESIMS of compound **1**.

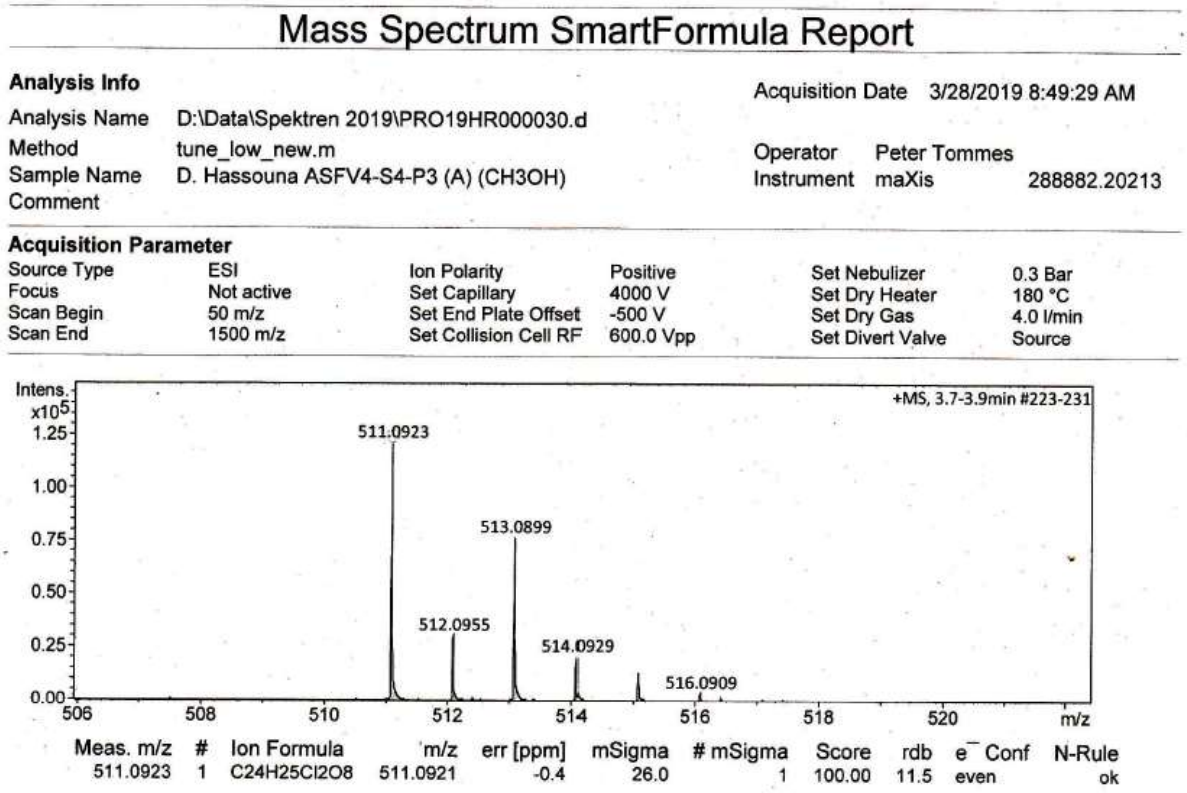

Figure S3. ^{1}H NMR (600 MHz, $CDCl_3$) spectrum of compound **1**.

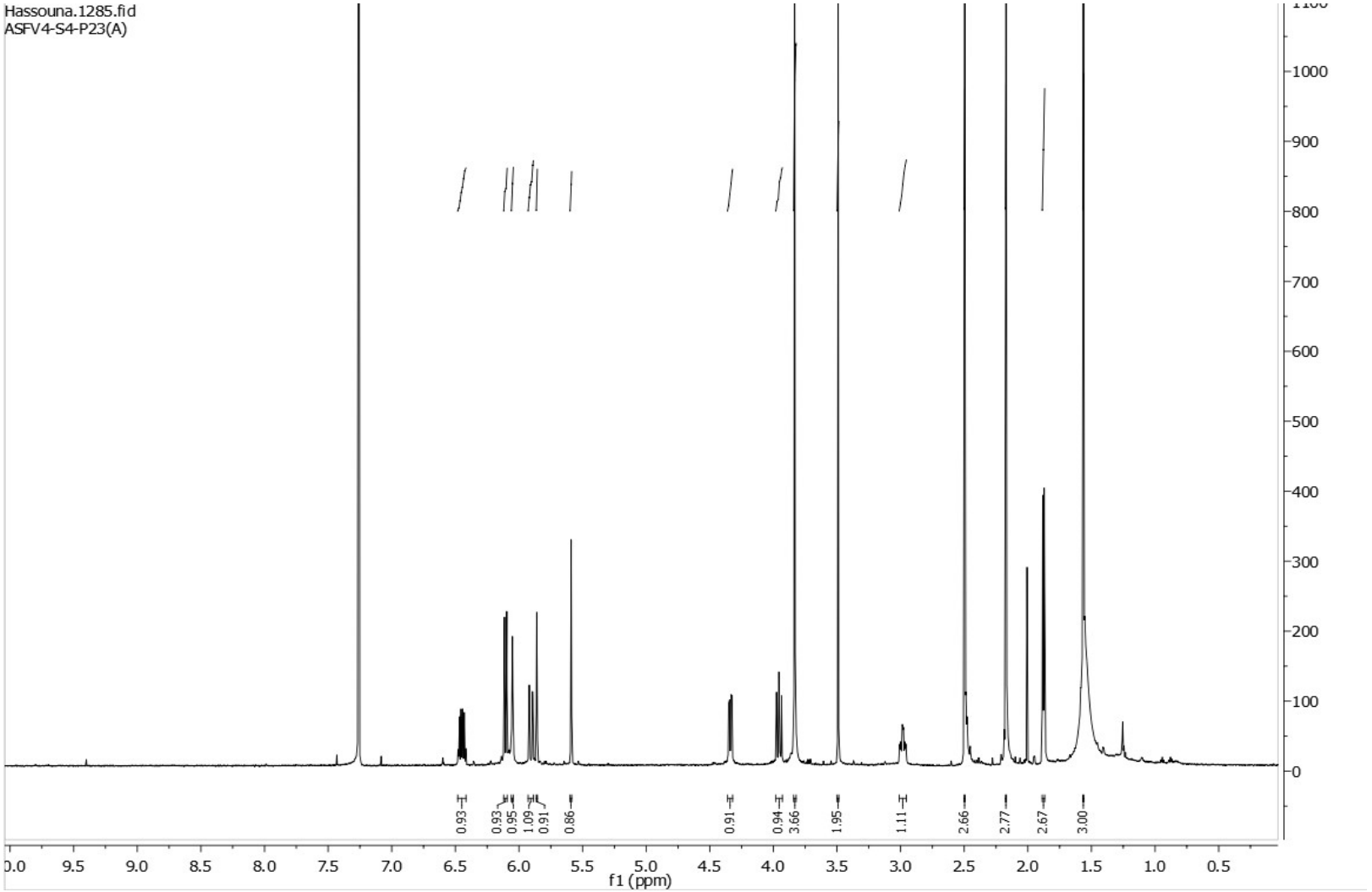

Figure S4. COSY (600 MHz, $CDCl_3$) spectrum of compound **1**.

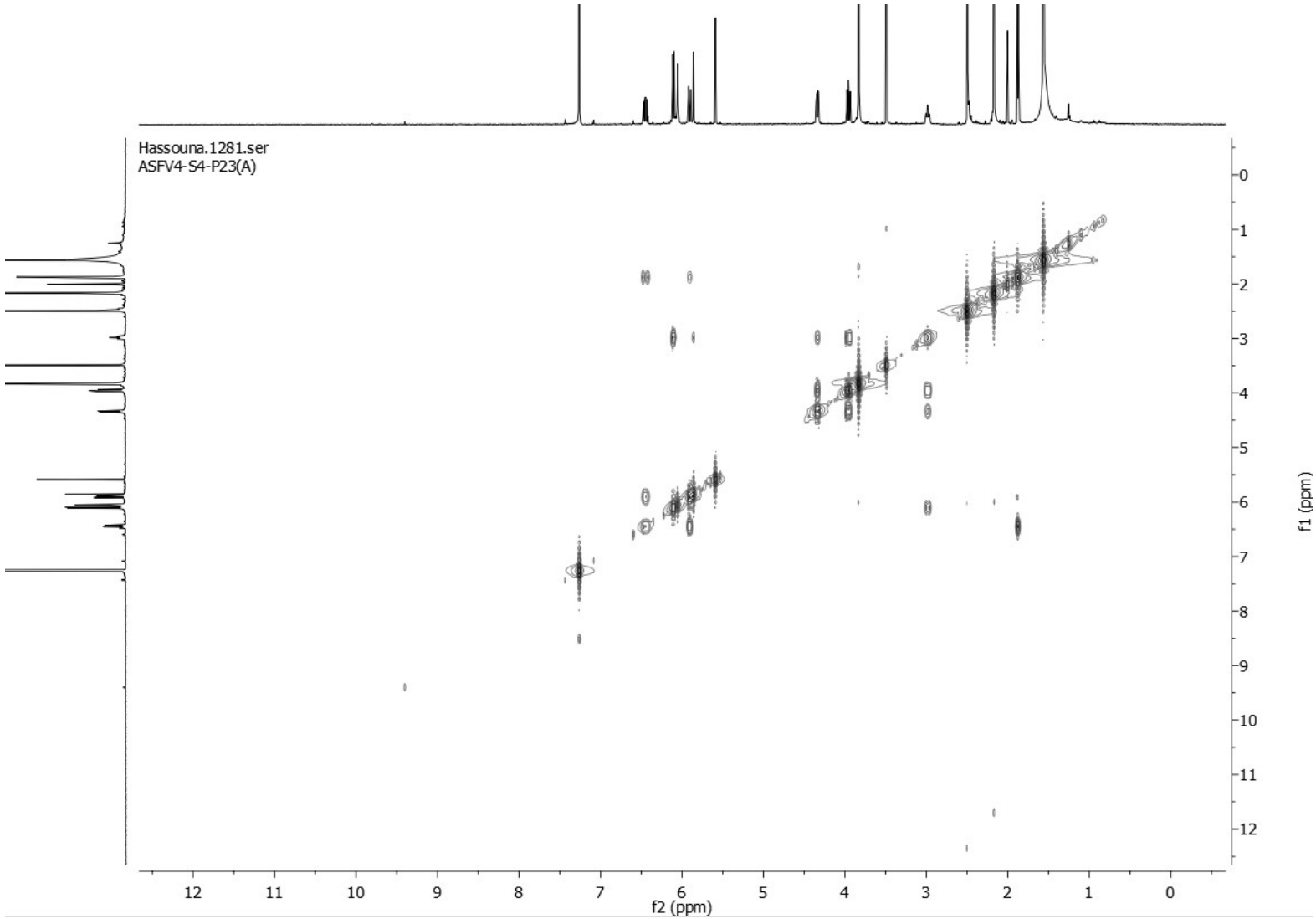

Figure S5. HSQC (600 MHz/150 MHz, $CDCl_3$) spectrum of compound **1**.

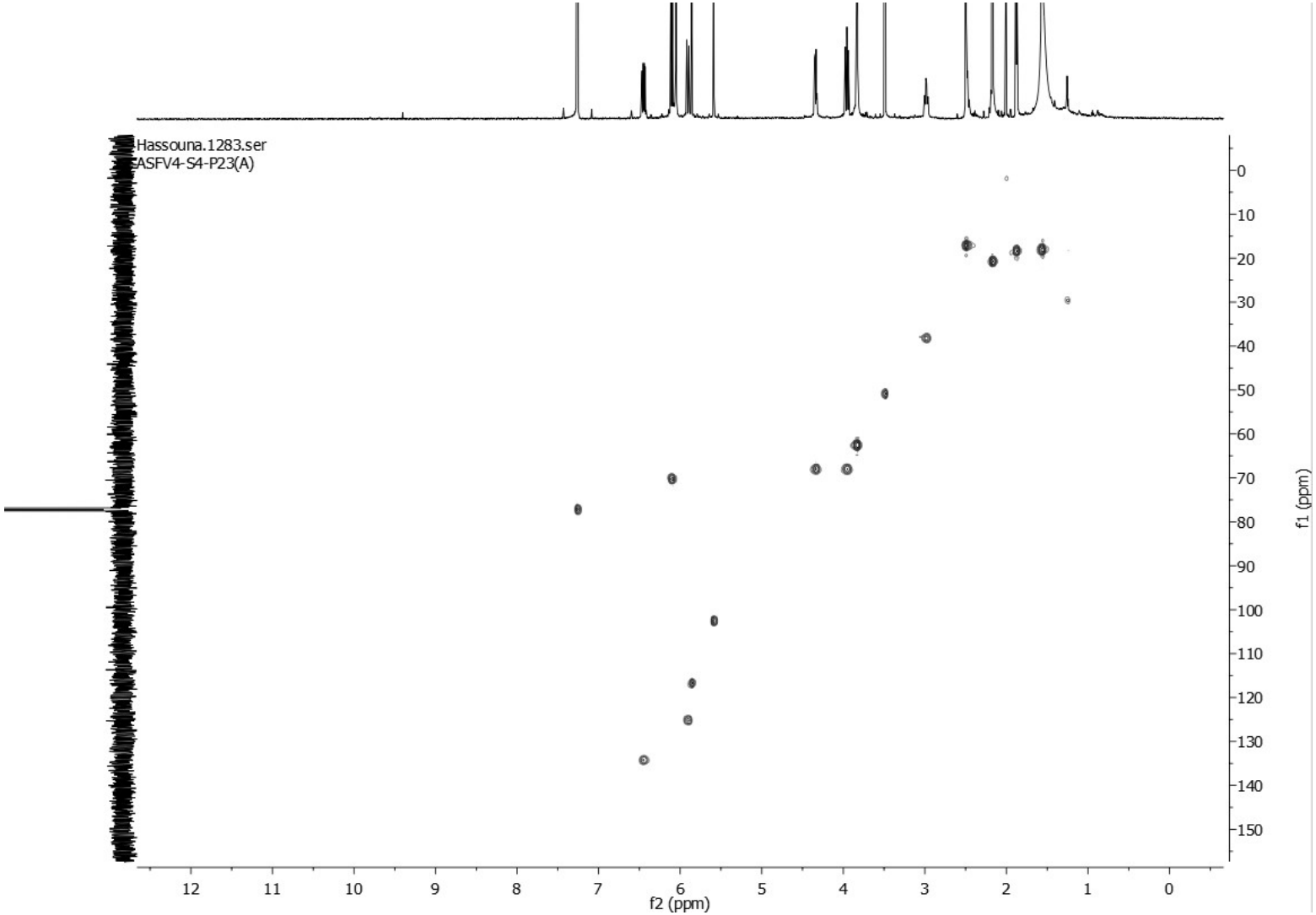

Figure S6. HMBC (600 MHz/150 MHz, $CDCl_3$) spectrum of compound **1**.

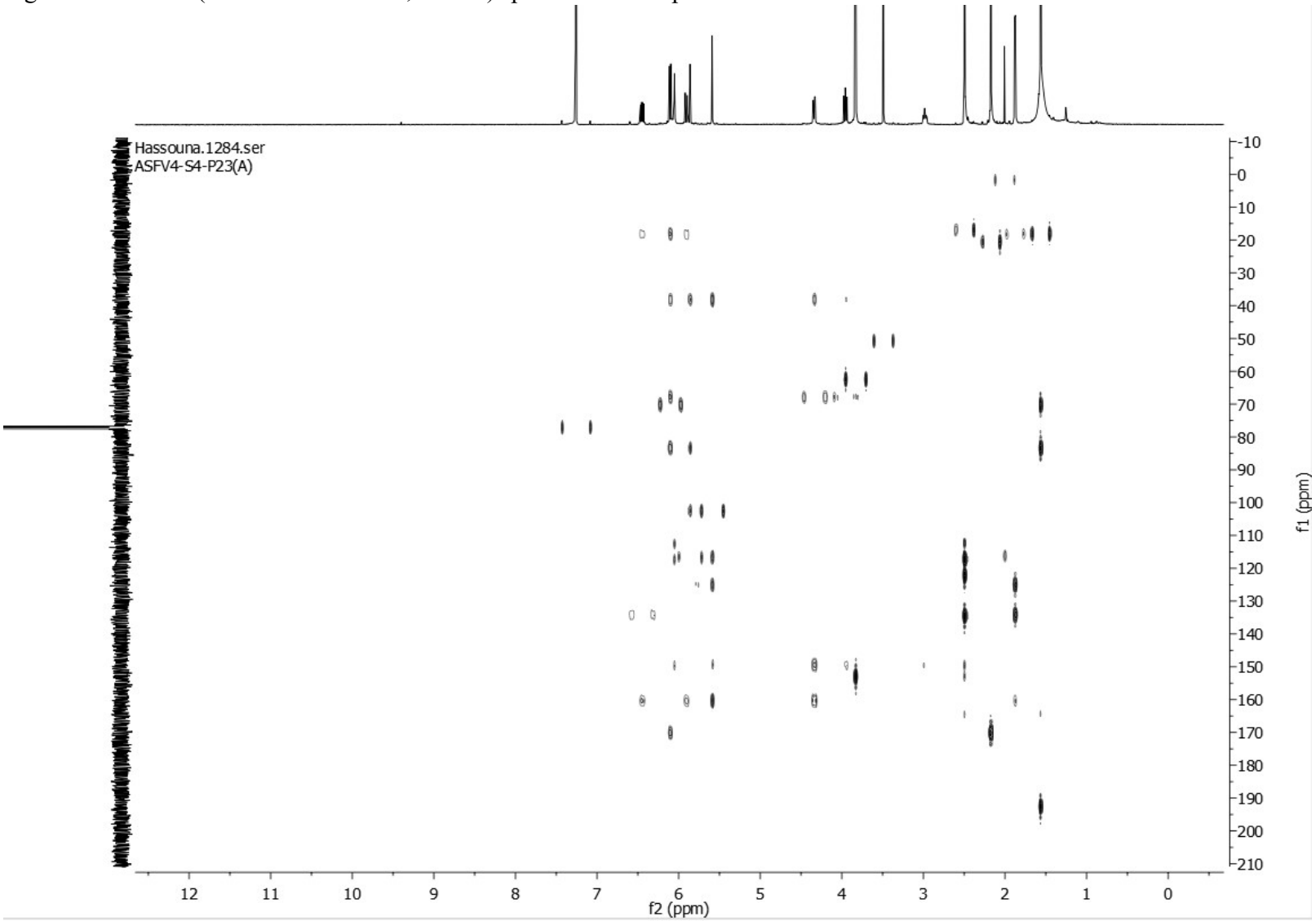

Figure S7. ROESY (600 MHz, $CDCl_3$) spectrum of compound **1**.

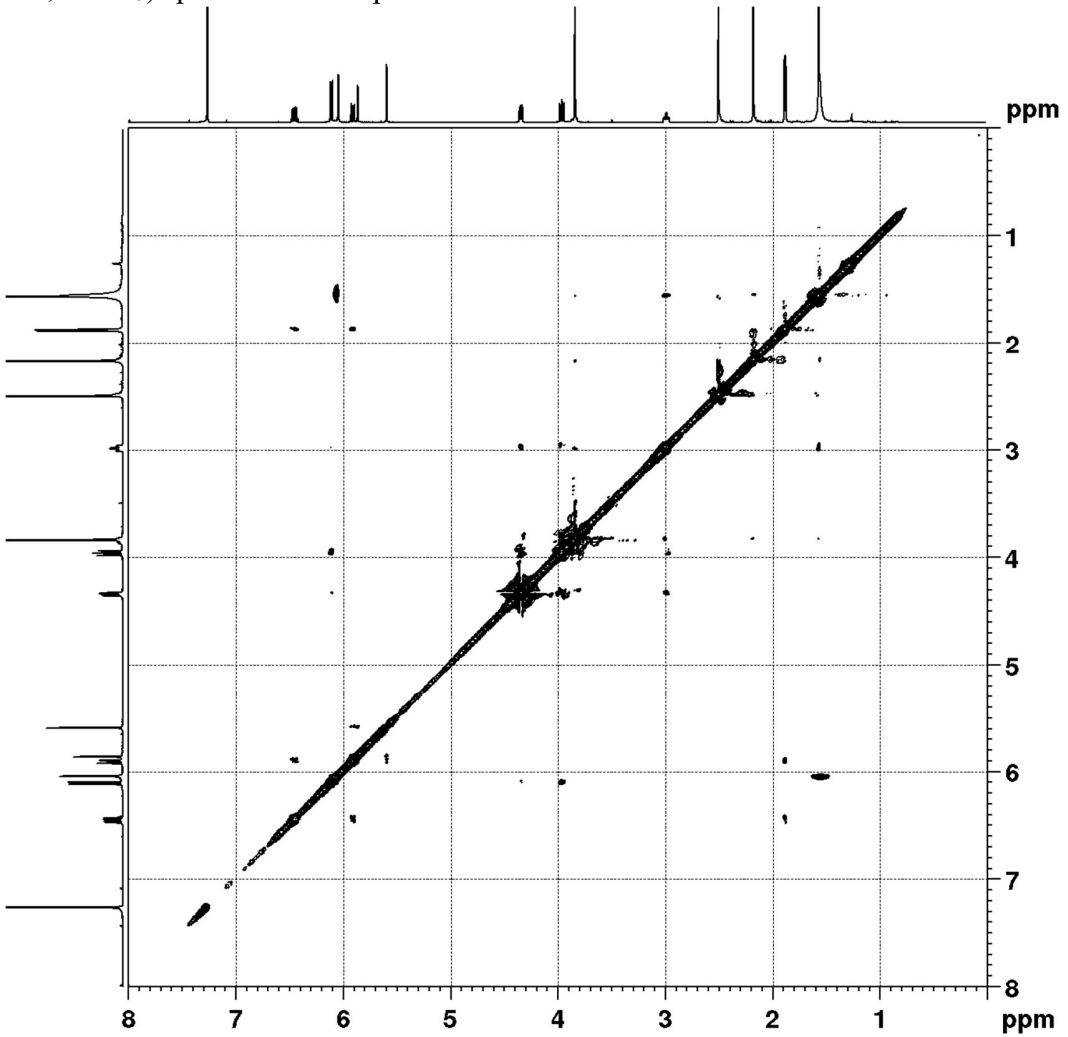

Figure S8. UV spectrum of compound **2**.

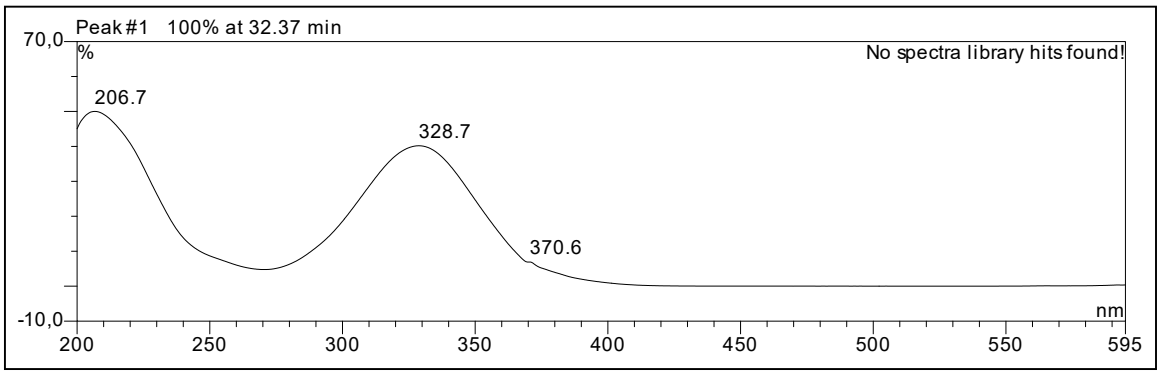

Figure S9. HRESIMS of compound **2**.

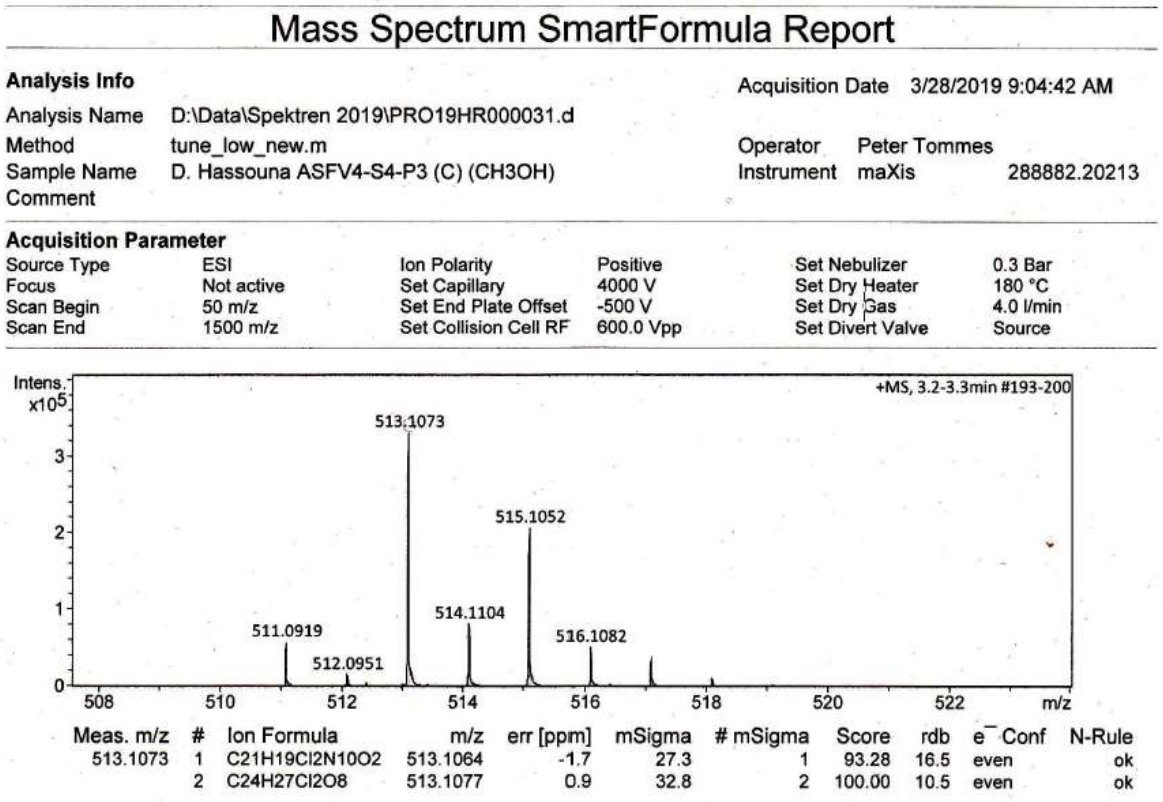

Figure S10. ^{1}H NMR (600 MHz, $CDCl_3$) spectrum of compound **2**.

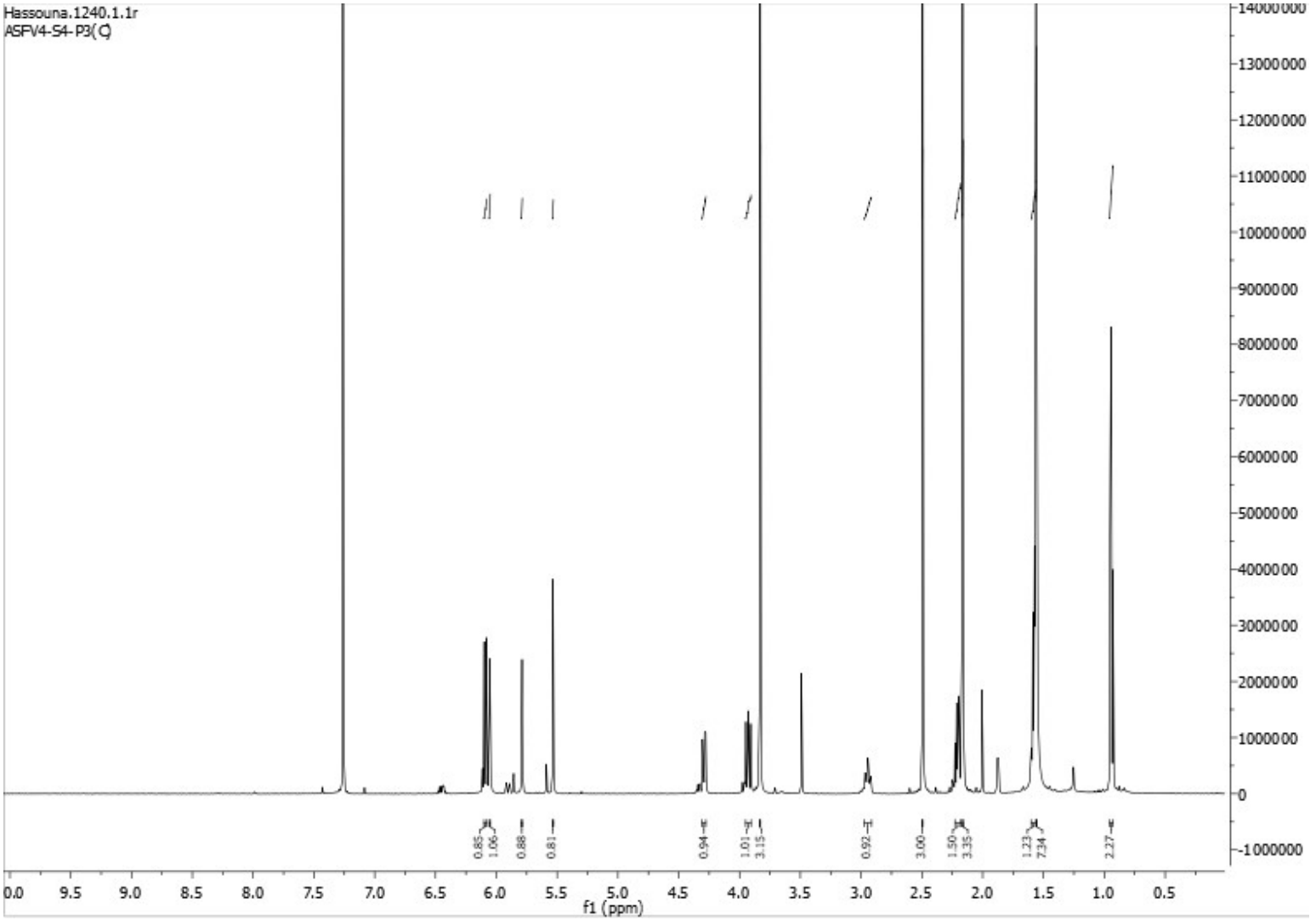

Figure S11. COSY (600 MHz, $CDCl_3$) spectrum of compound **2**.

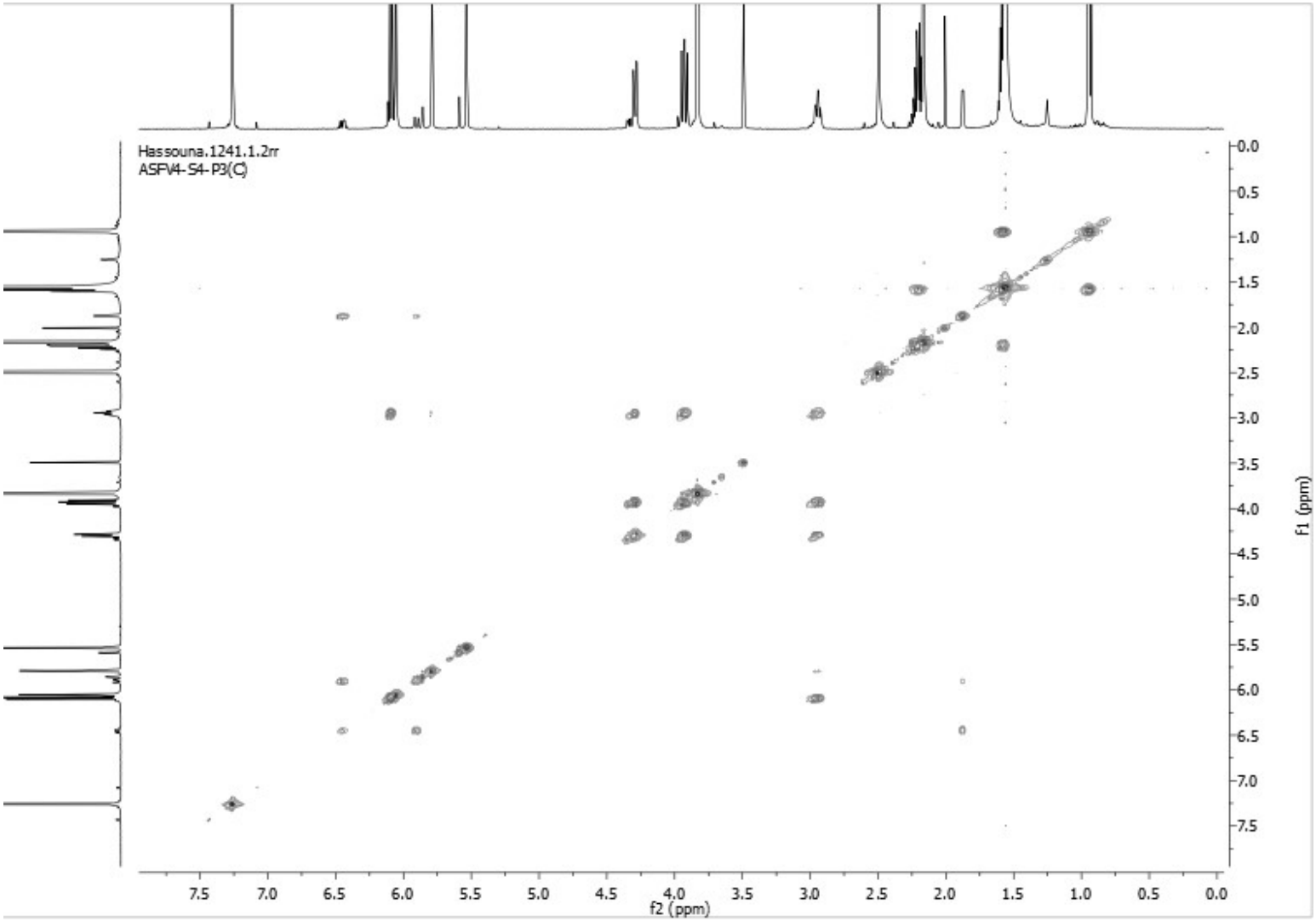

Figure S12. HSQC (600 MHz/150 MHz, $CDCl_3$) spectrum of compound **2**.

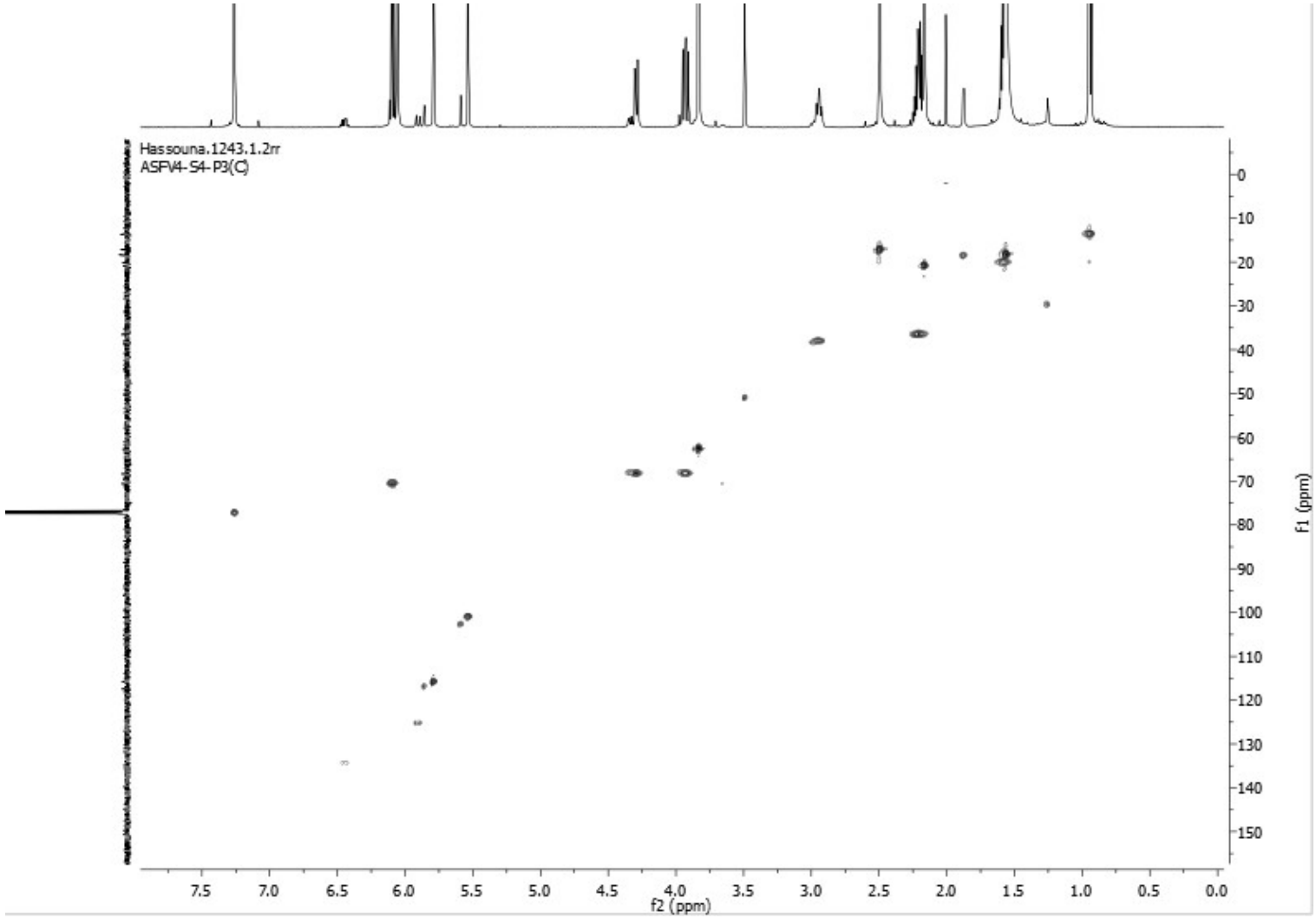

Figure S13. HMBC (600 MHz/150 MHz, $CDCl_3$) spectrum of compound **2**.

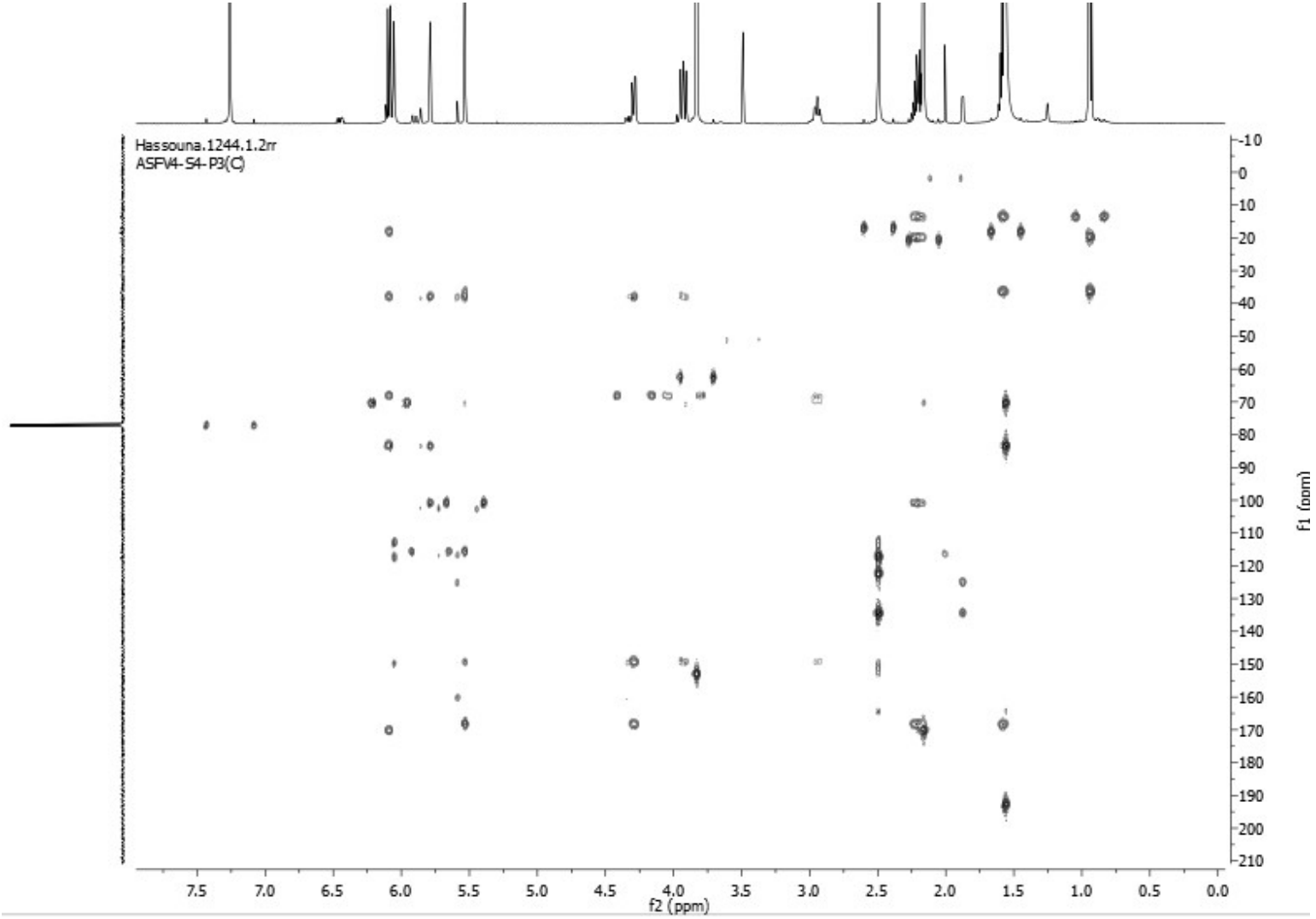

Figure S14. ROESY (600 MHz, $CDCl_3$) spectrum of compound **2**.

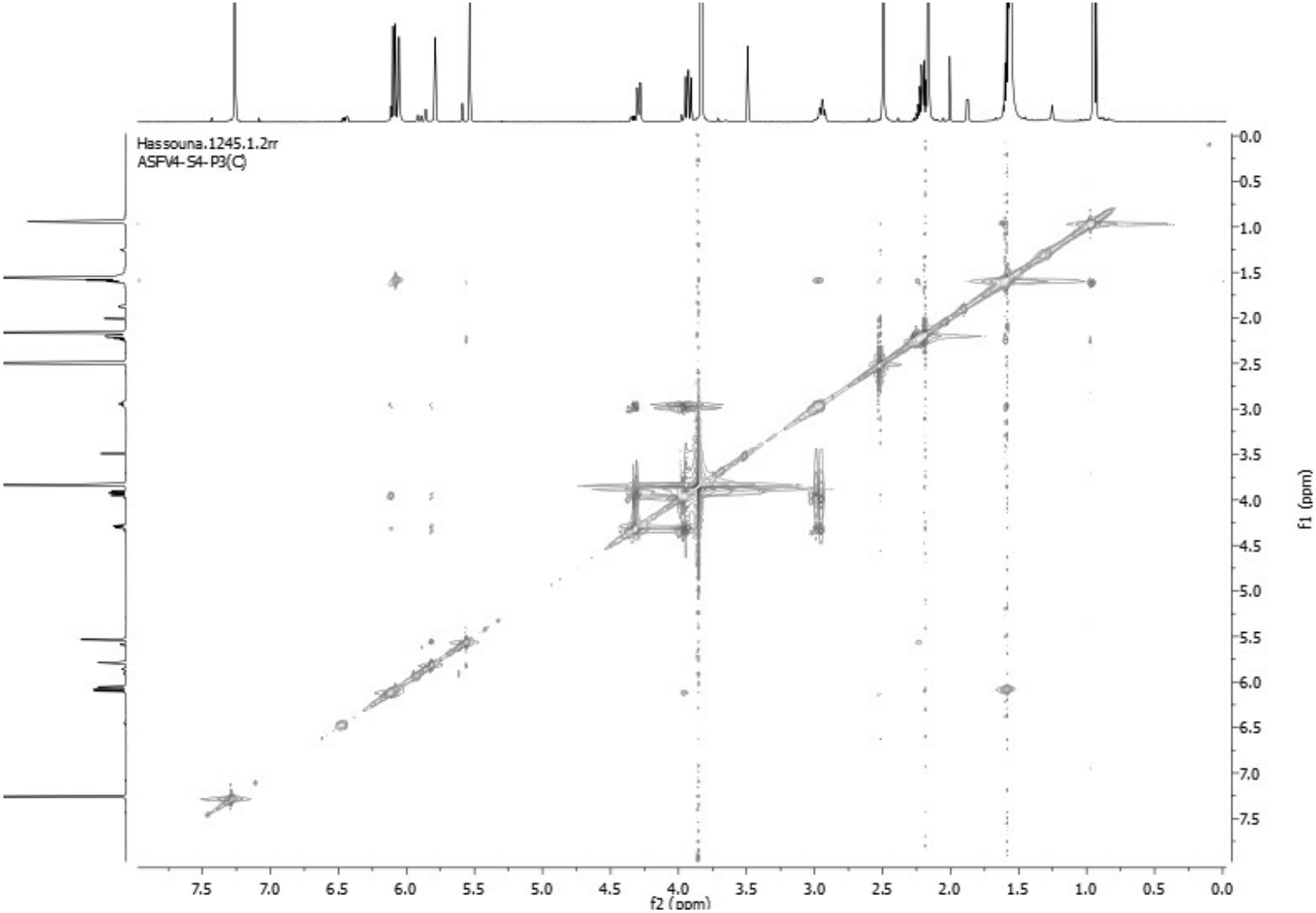

Figure S15. UV spectrum of compound **7**.

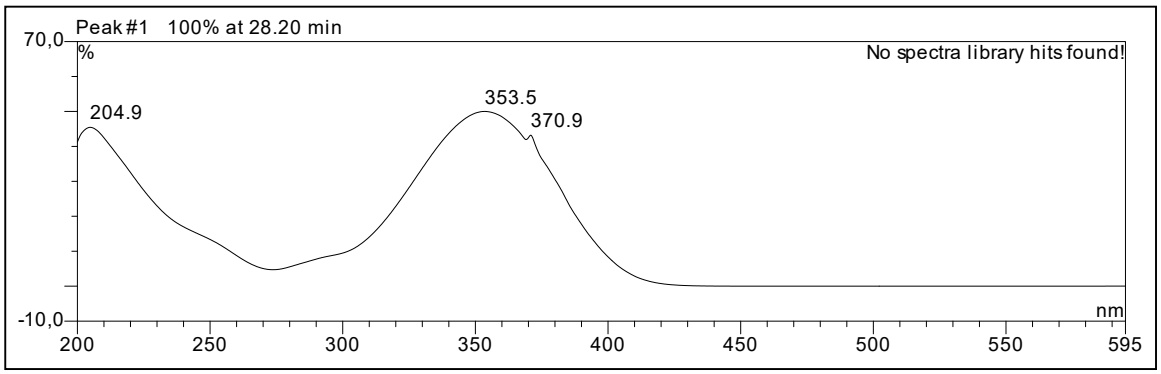

Figure S16. HRESIMS of compound **7**.

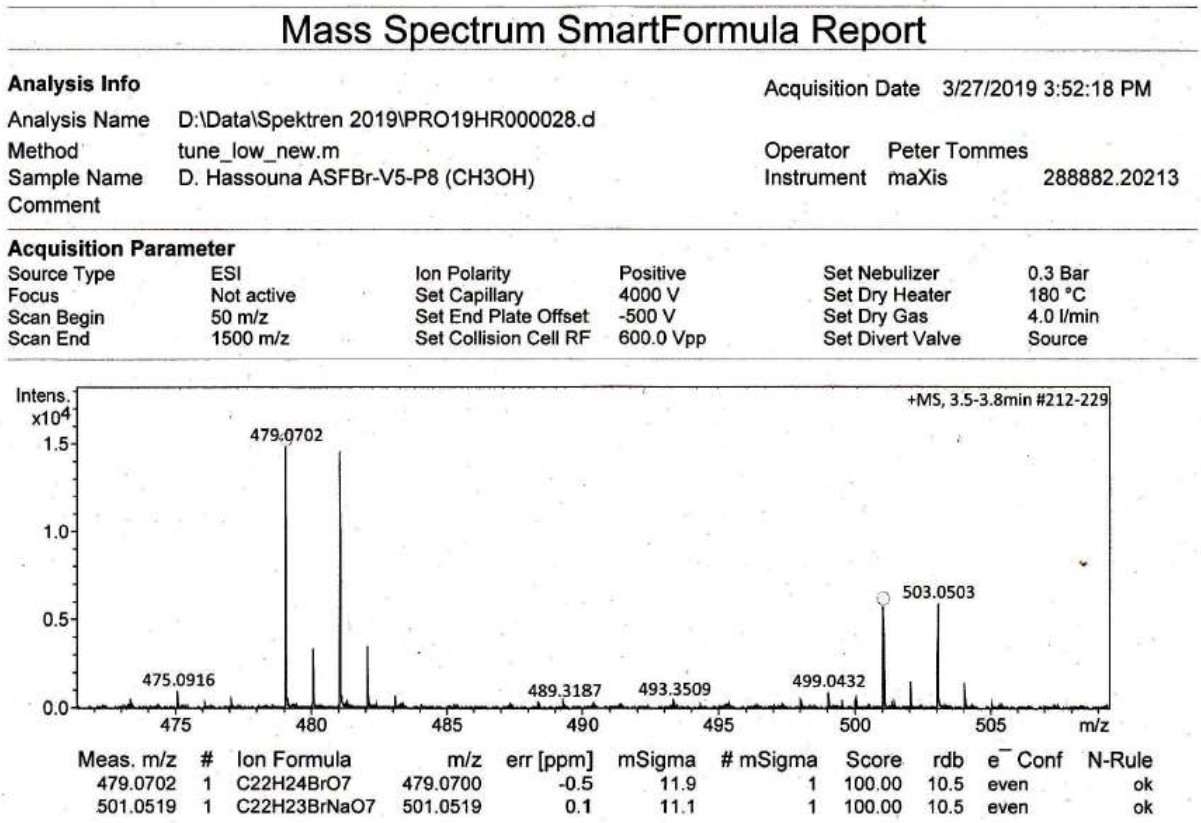

Figure S17. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of compound **7**.

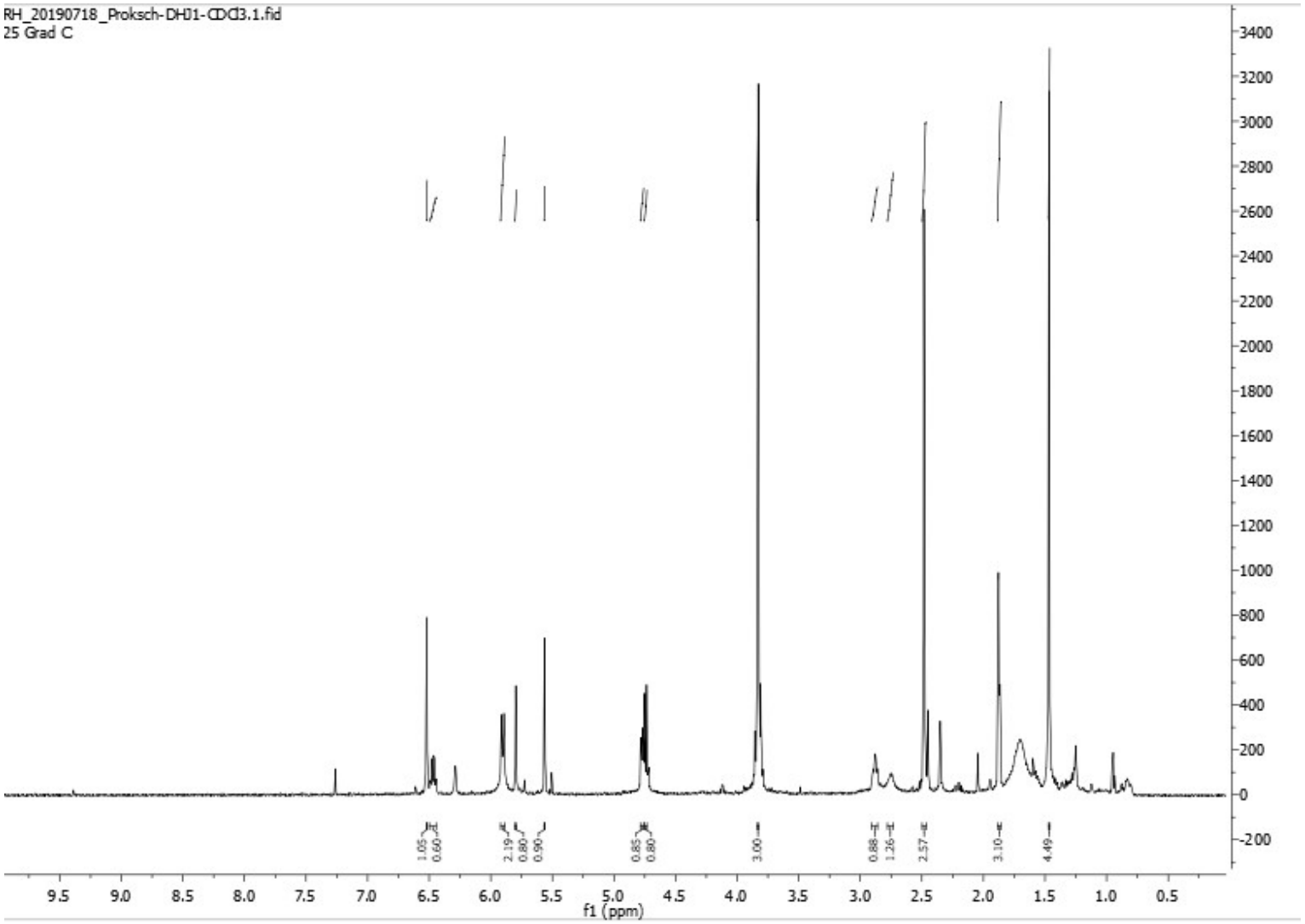

Figure S18. ^{13}C NMR (175 MHz, $CDCl_3$) spectrum of compound **7**.

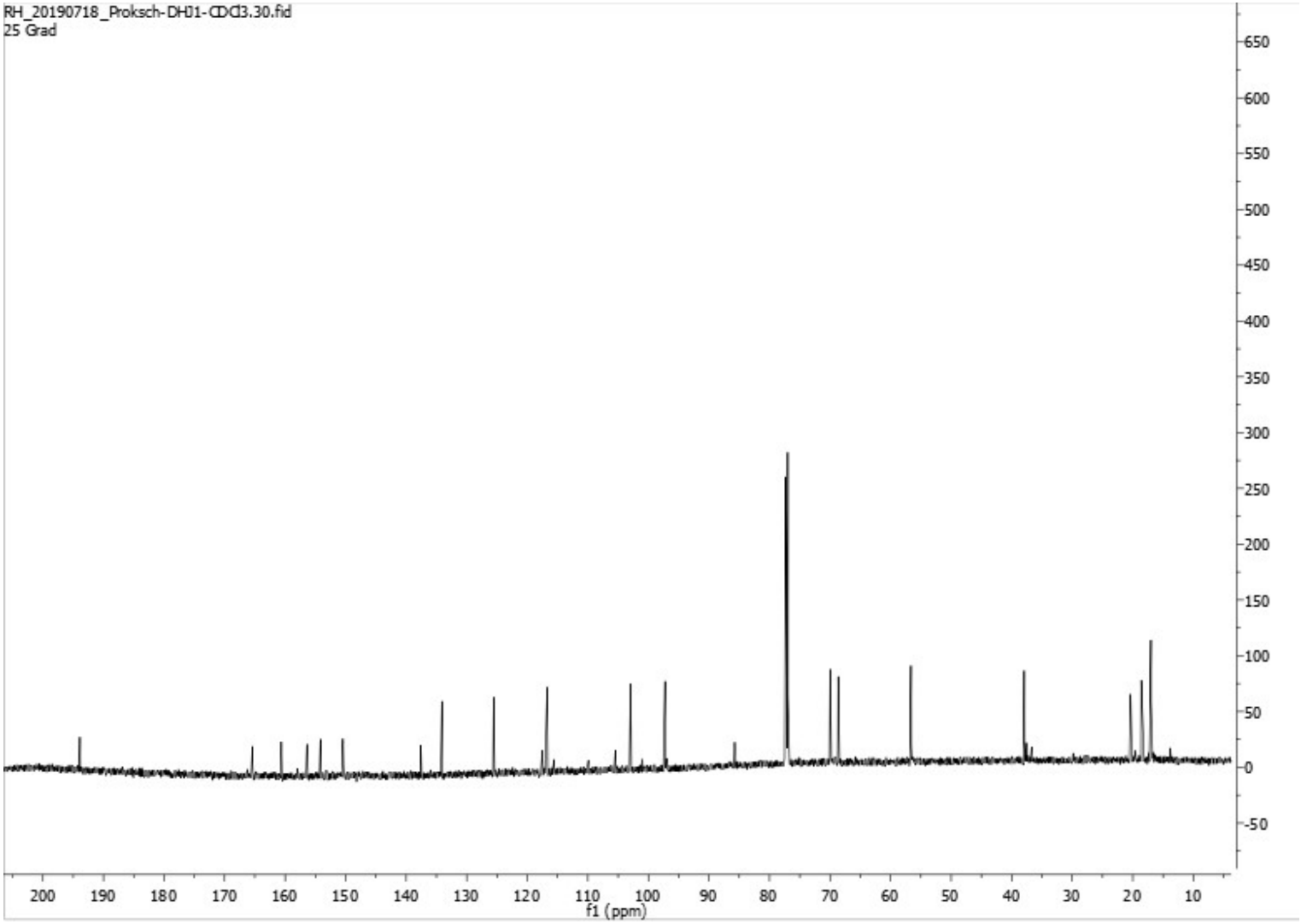

Figure S19. COSY (700 MHz, $CDCl_3$) spectrum of compound **7**.

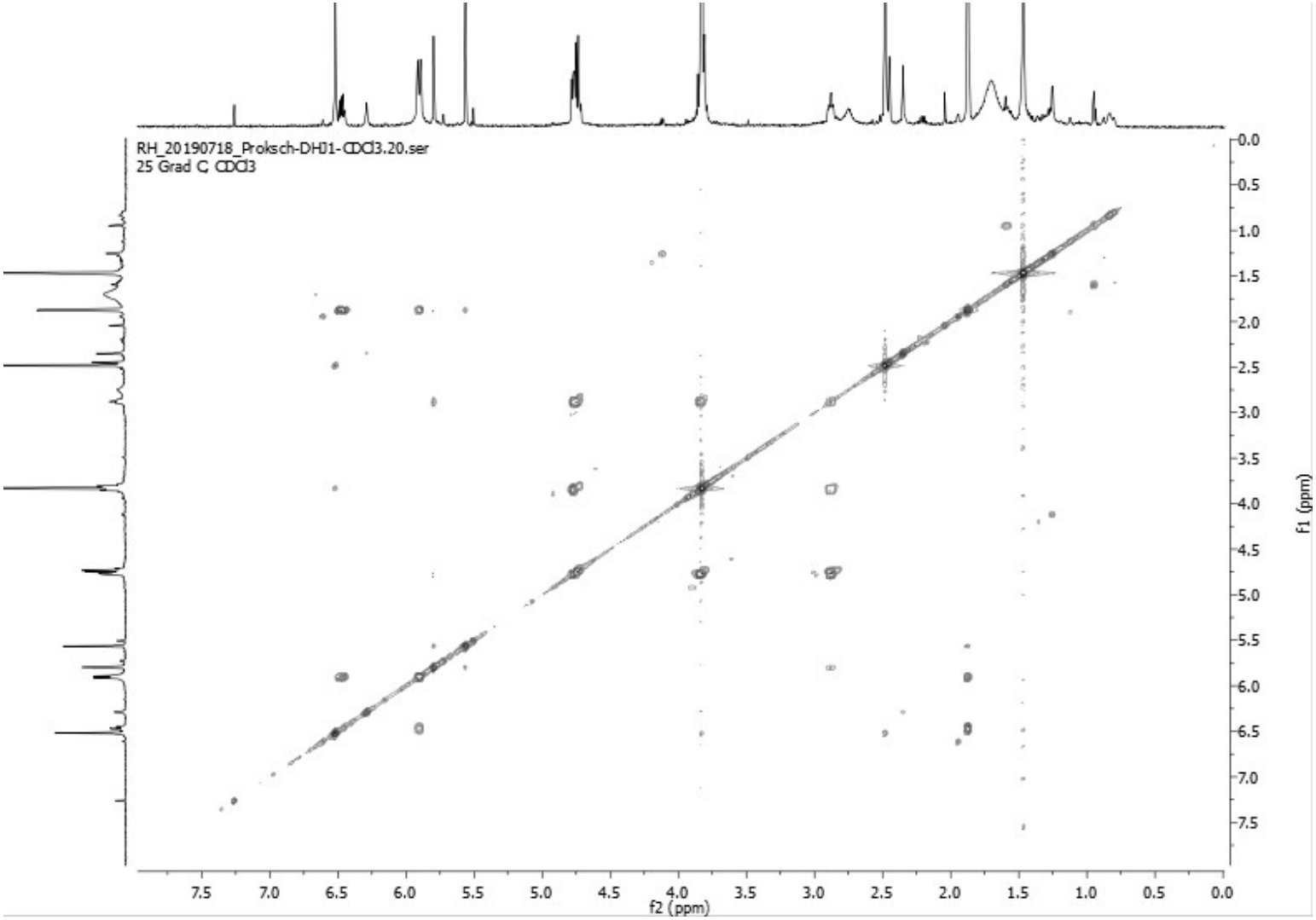

Figure S20. HSQC (700 MHz/175 MHz, $CDCl_3$) spectrum of compound **7**.

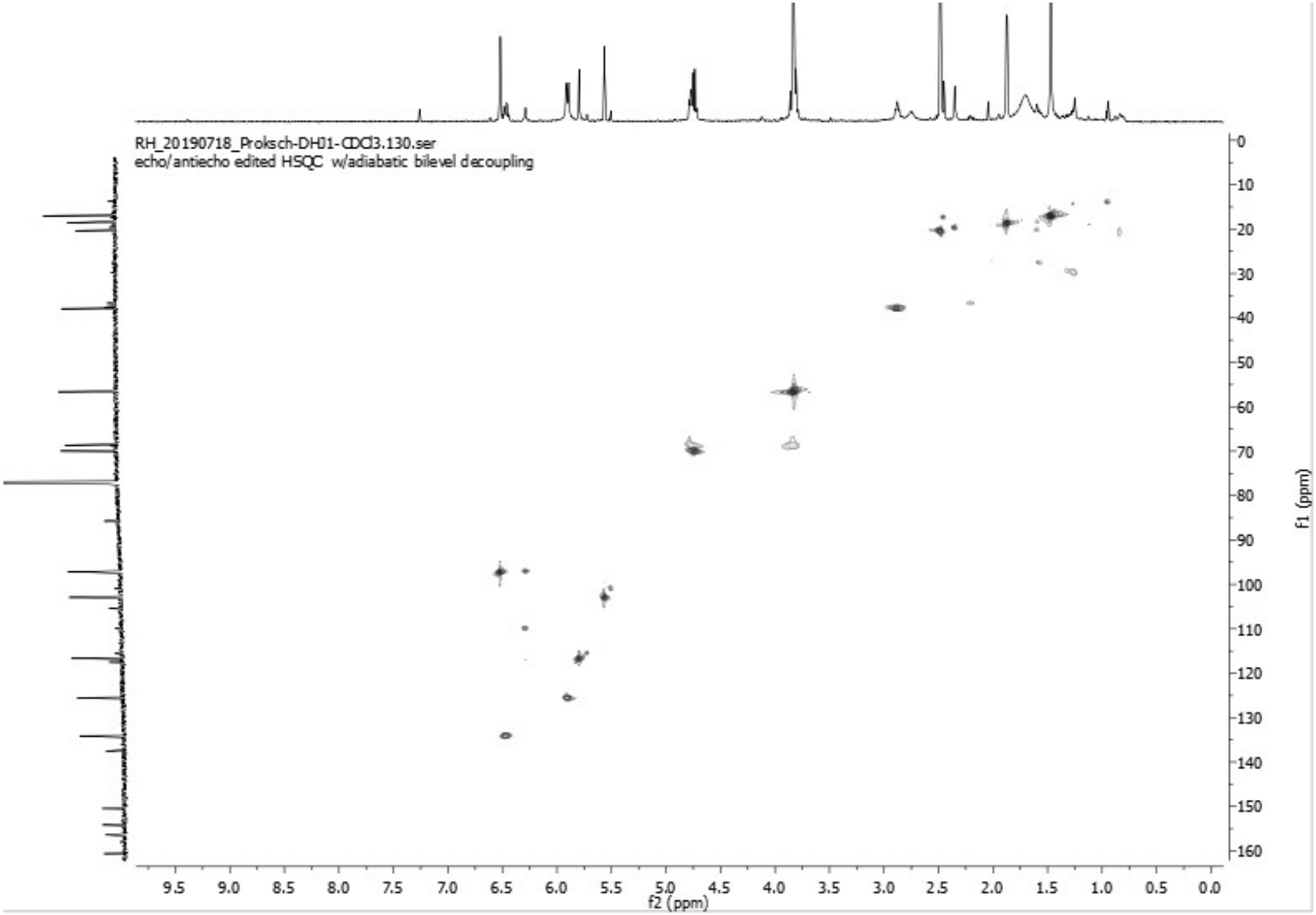

Figure S21. HMBC (700 MHz/175 MHz, $CDCl_3$) spectrum of compound **7**.

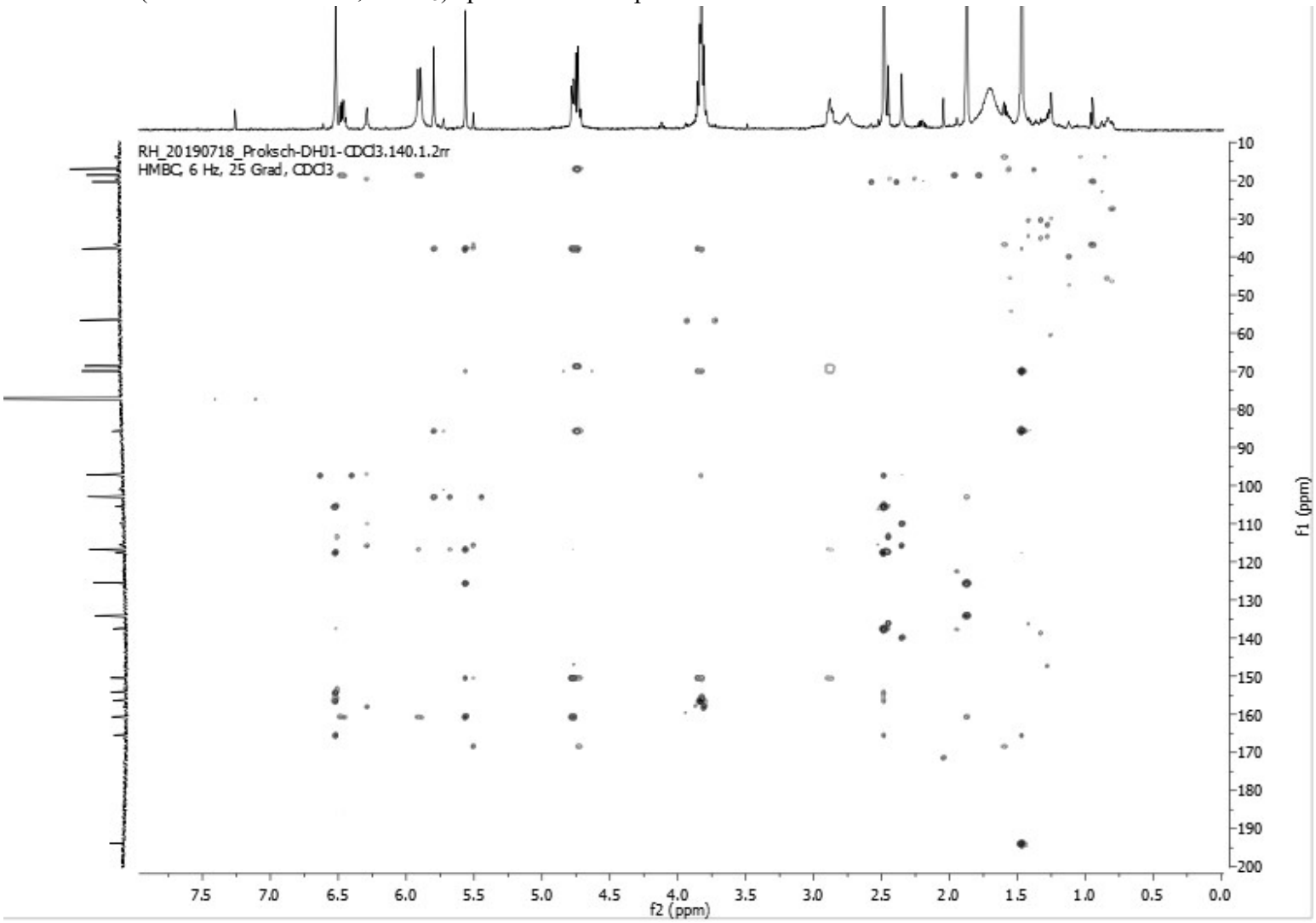

Figure S22. ROESY (700 MHz/175 MHz, $CDCl_3$) spectrum of compound **7**.

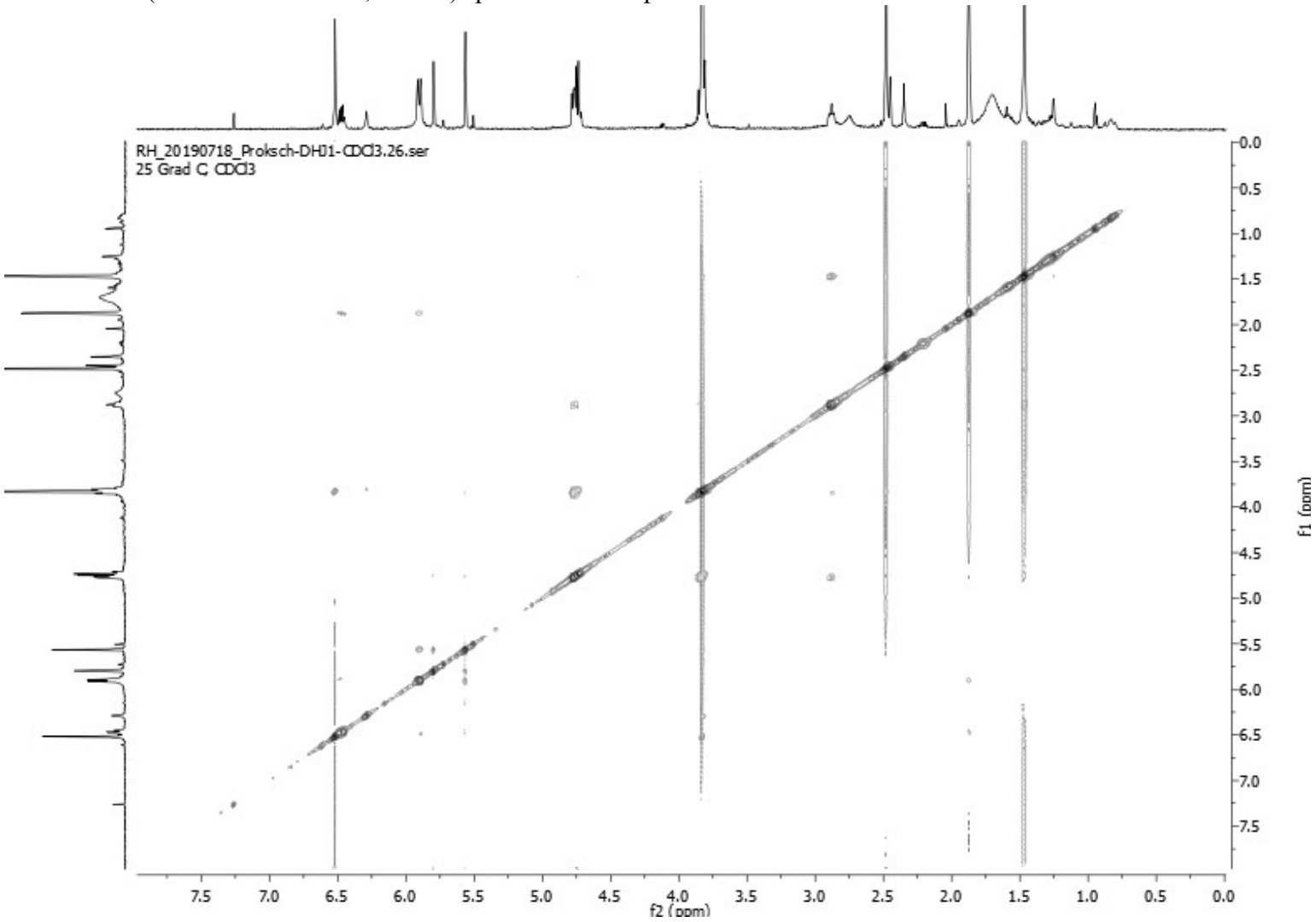

Figure S23. UV spectrum of compound **8**.

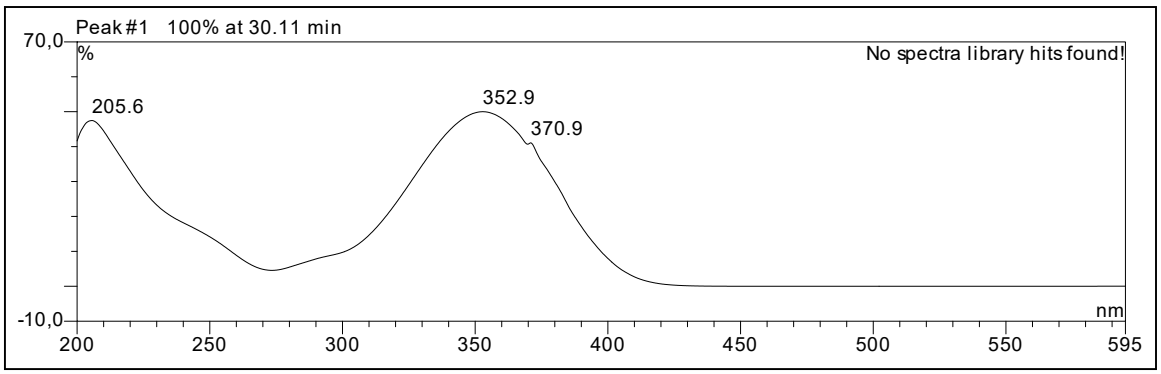

Figure S24. HRESIMS of compound **8**.

Mass Spectrum SmartFormula Report

Analysis Info

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Sample Name	Hassouna ASFBr-V5-P10 II (CH3OH)	Instrument	maXis 288882.20213
Comment	25 ul in 1 ml		

Acquisition Parameter

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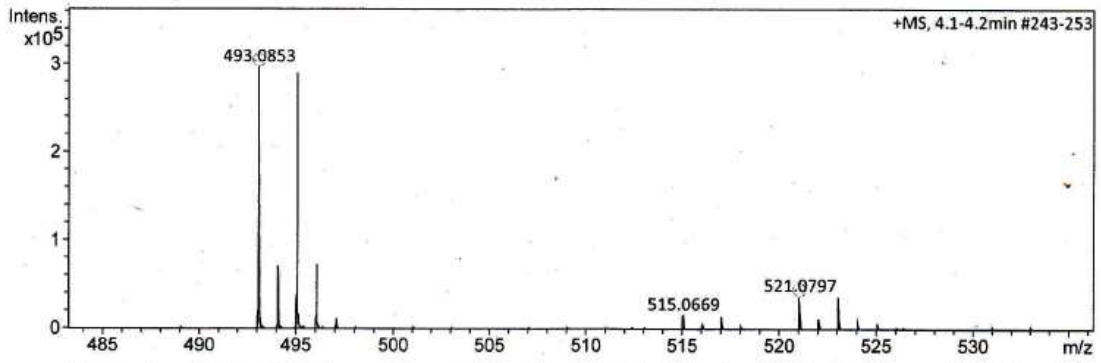
Scan End	1500 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Source



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493.0853	1	C20H18BrN10O	493.0843	-2.0	12.1	1	74.41	16.5	even	ok
	2	C23H26BrO7	493.0856	0.8	12.7	2	100.00	10.5	even	ok
521.0797	1	C24H26BrO8	521.0806	1.6	48.1	1	98.38	11.5	even	ok
	2	C21H18BrN10O2	521.0792	-1.0	52.4	2	100.00	17.5	even	ok

Figure S25. ^{1}H NMR (600 MHz, $CDCl_3$) spectrum of compound **8**.

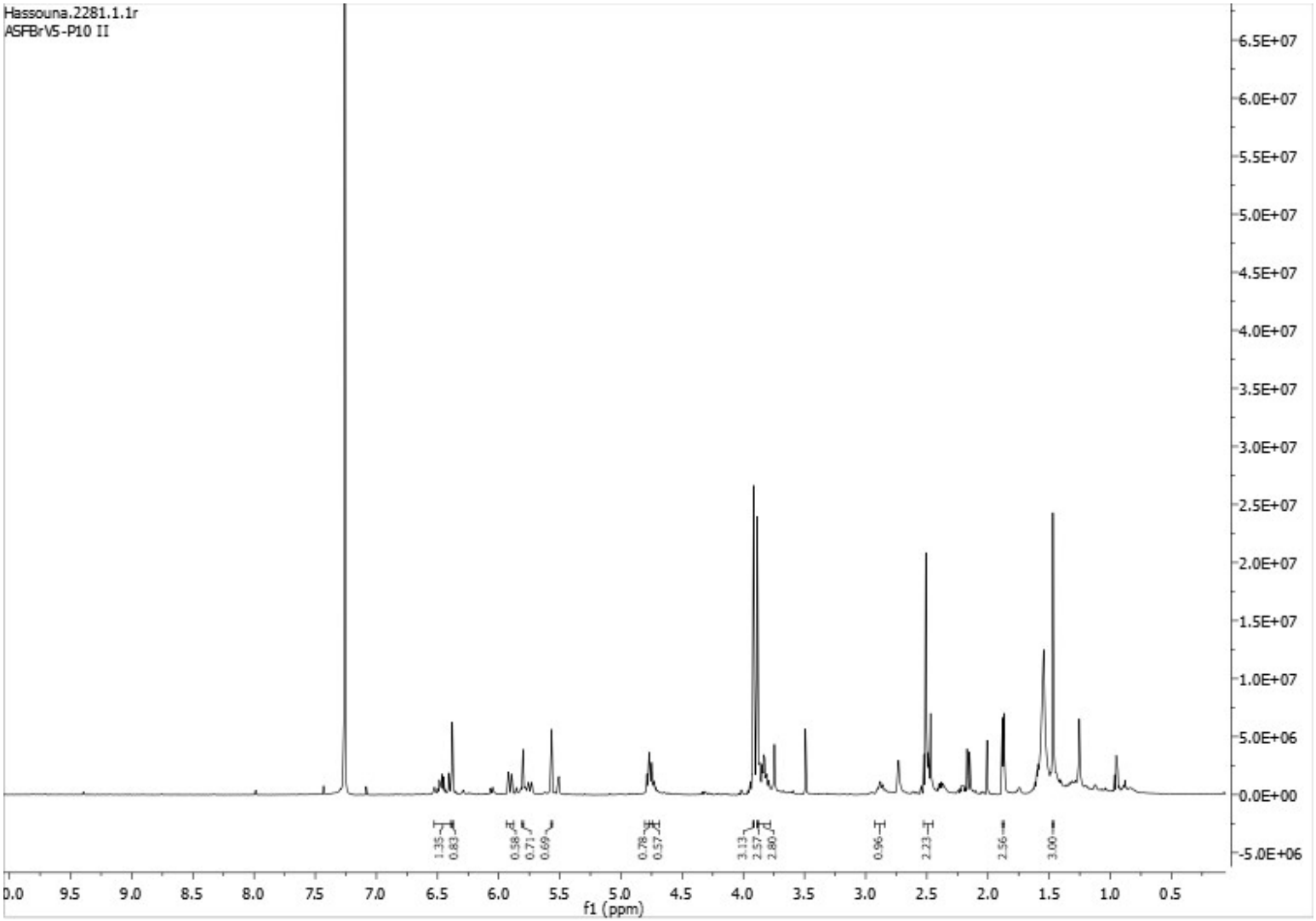

Figure S26. COSY (600 MHz, $CDCl_3$) spectrum of compound **8**.

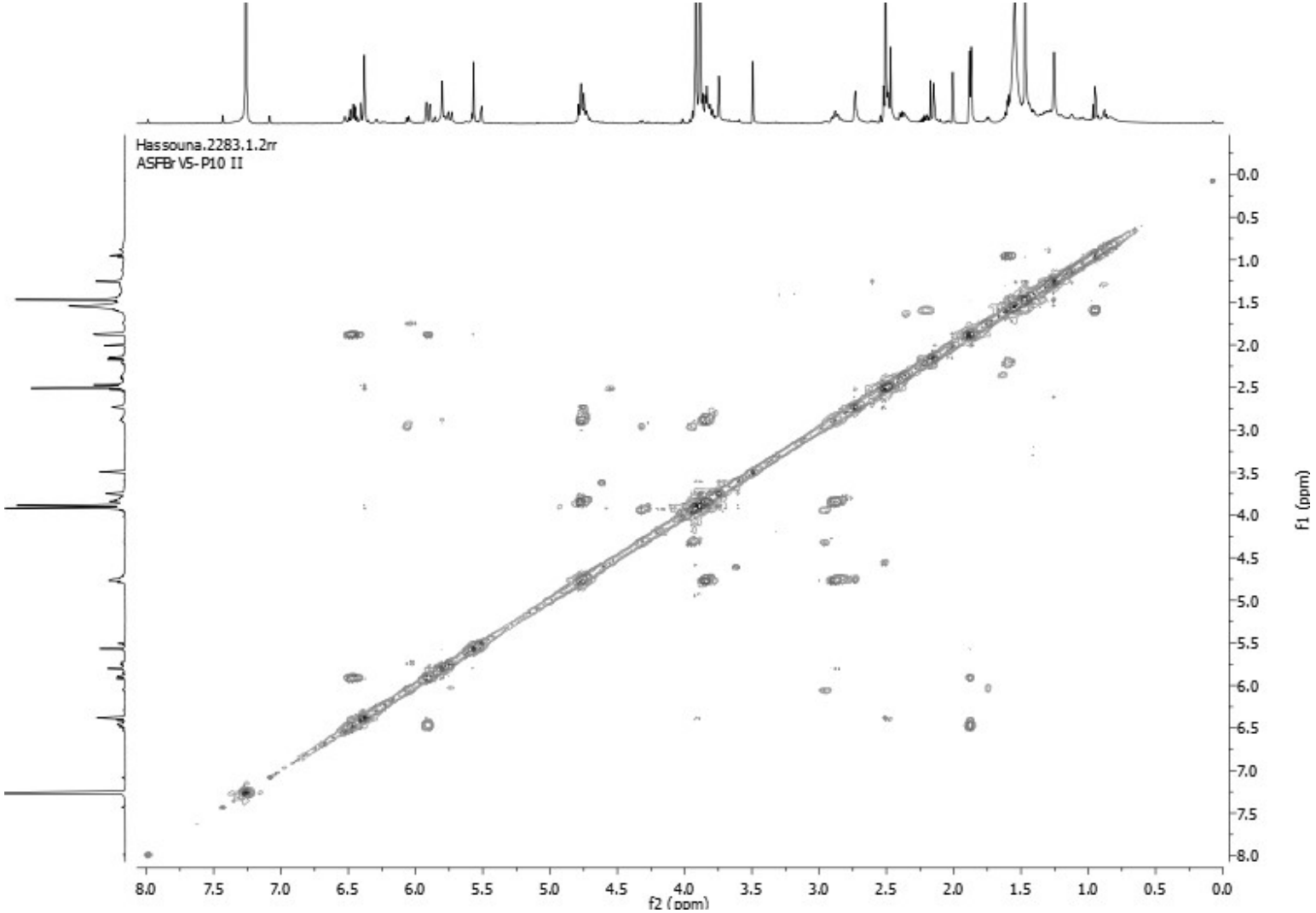

Figure S27. HSQC (600 MHz/150 MHz, $CDCl_3$) spectrum of compound **8**.

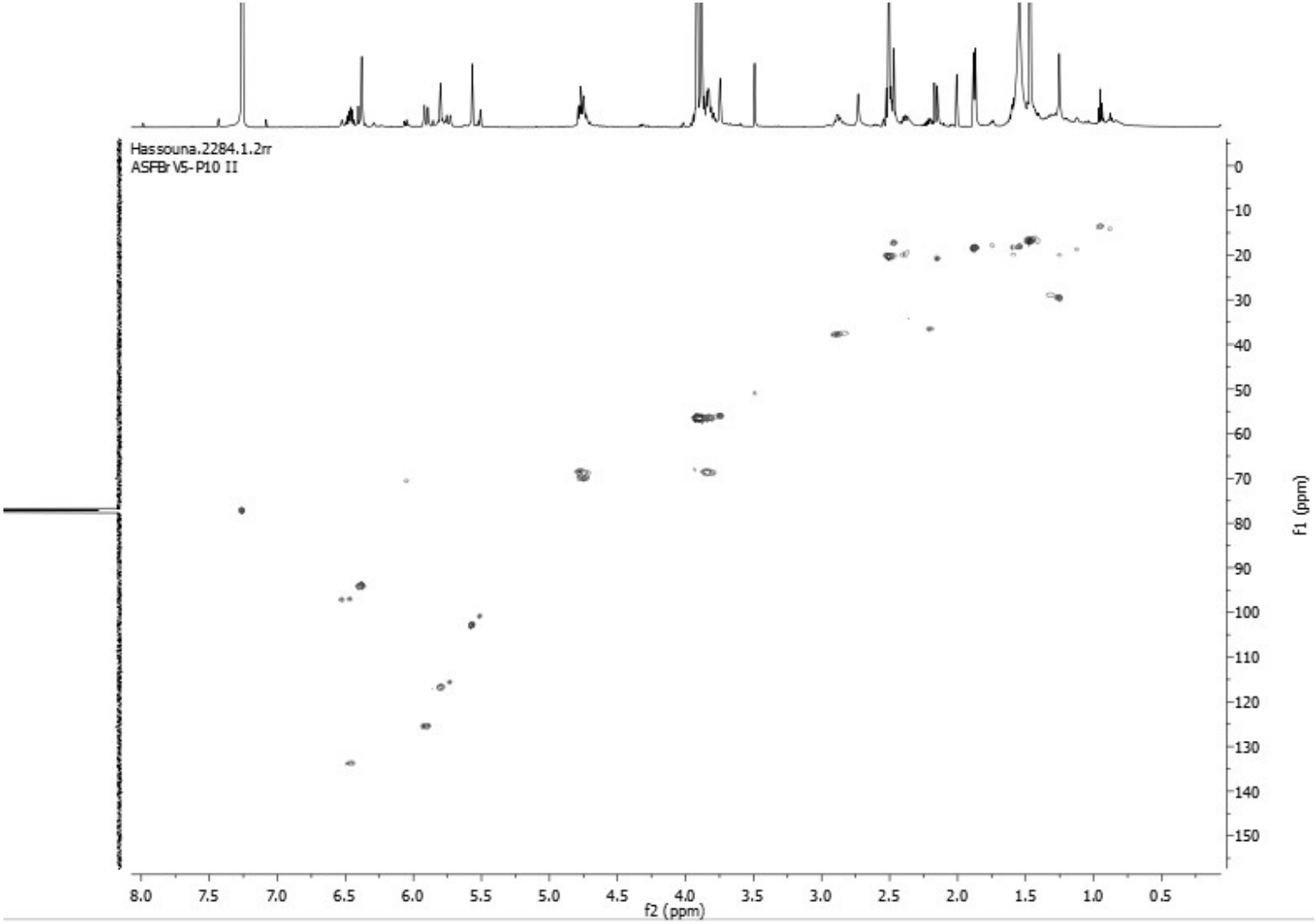

Figure S28. HMBC (600 MHz/150 MHz, $CDCl_3$) spectrum of compound **8**.

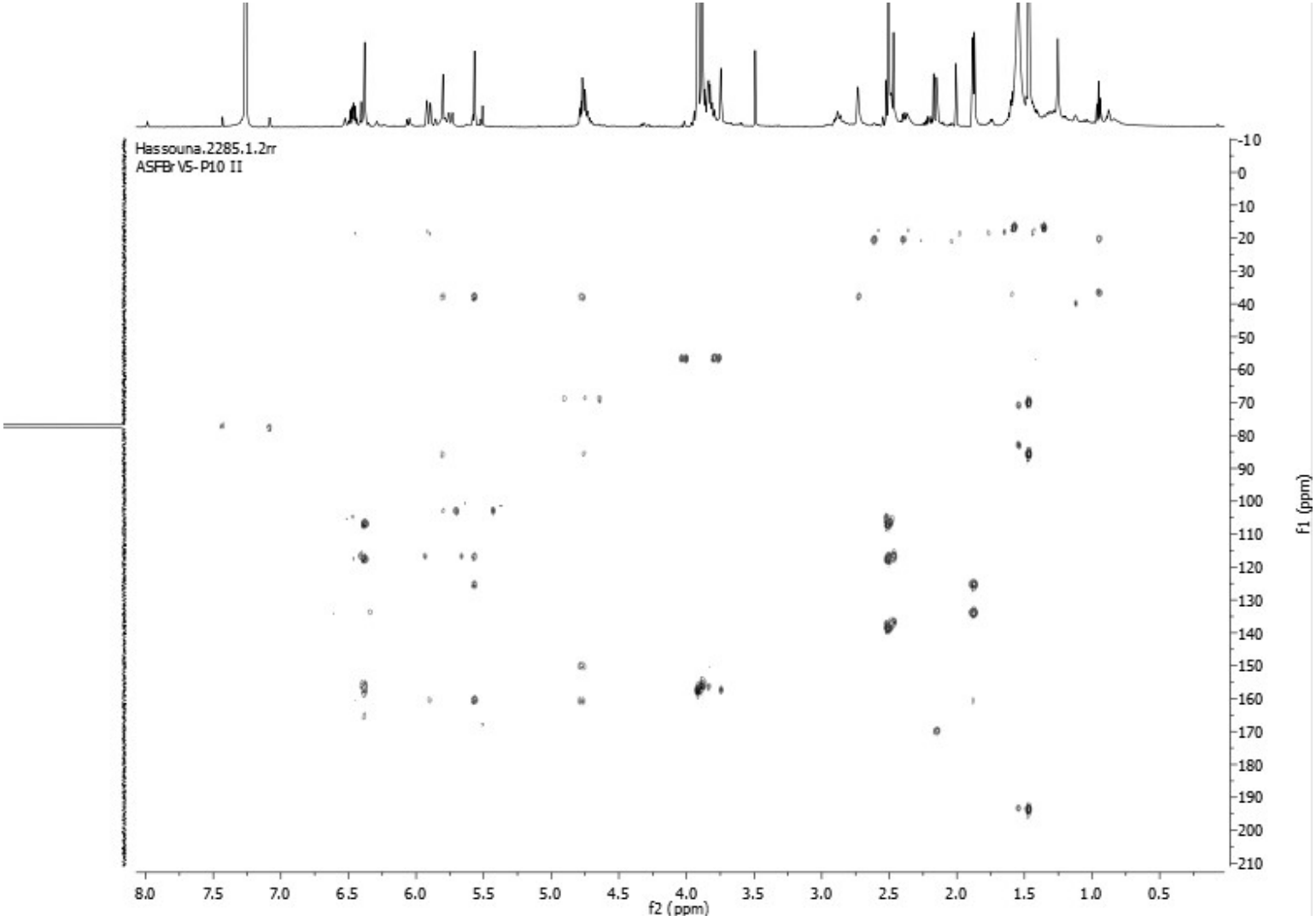

Figure S29. ROESY (600 MHz, $CDCl_3$) spectrum of compound **8**.

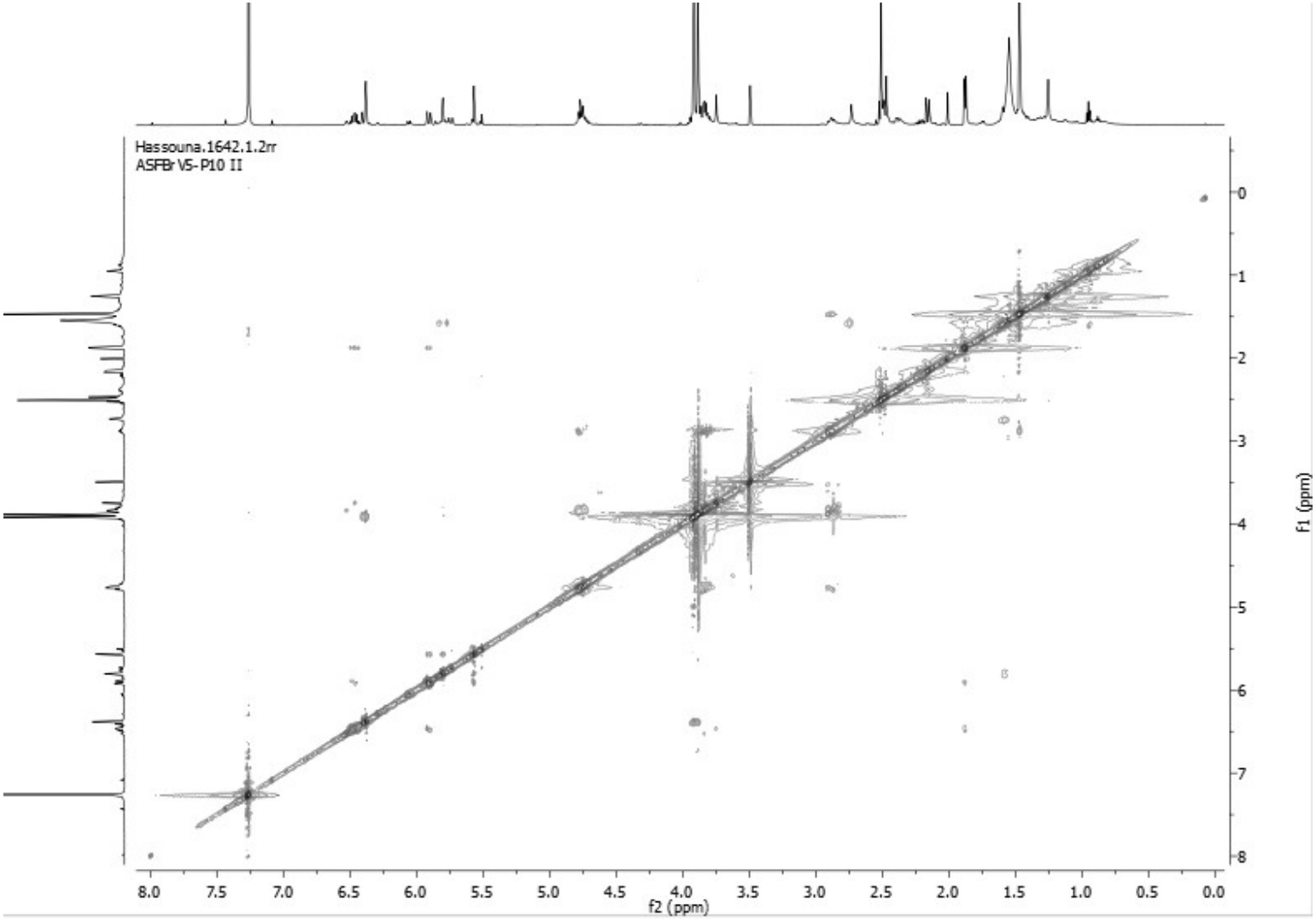

Figure S30. UV spectrum of compound **9**.

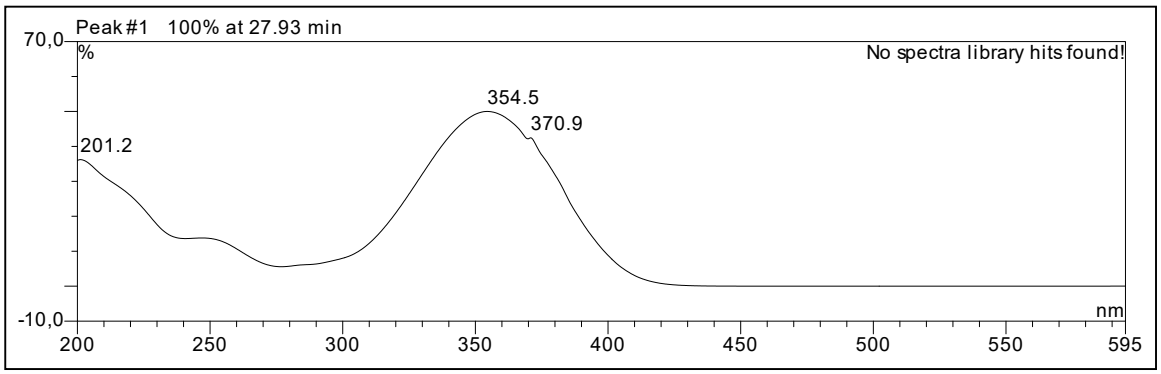

Figure S31. HRESIMS of compound **9**.

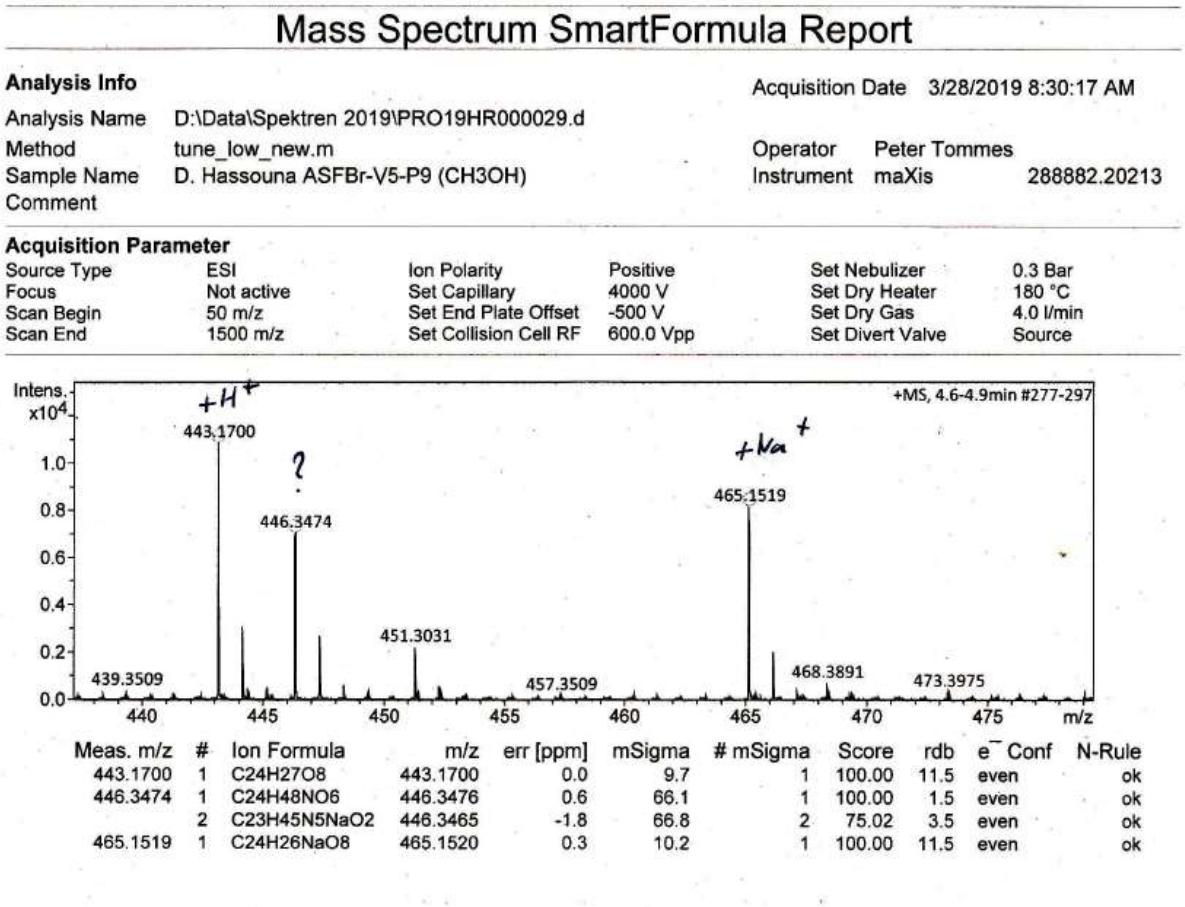

Figure S32. ^{1}H NMR (700 MHz, $CDCl_3$) spectrum of compound **9**.

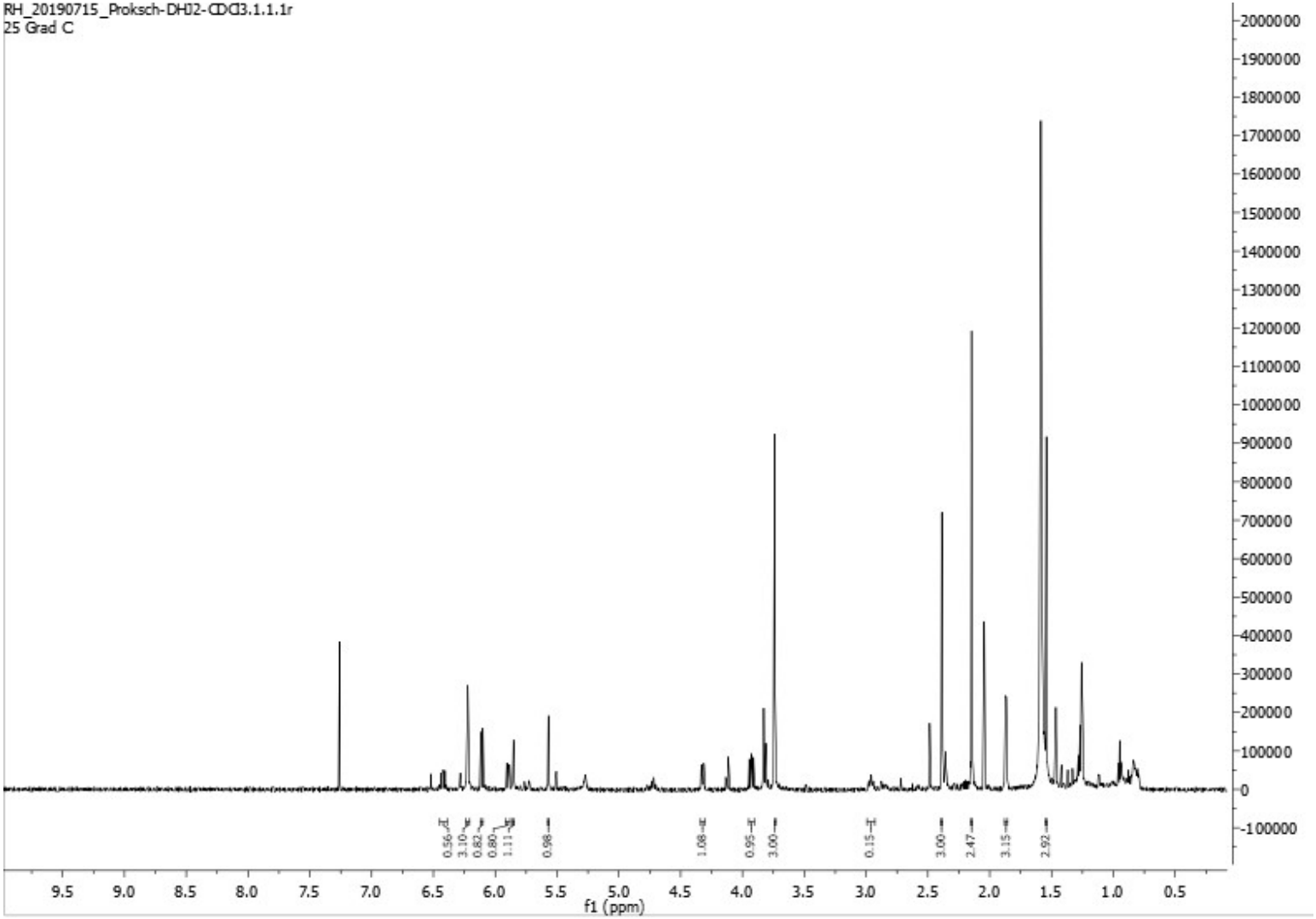

Figure S33. ^{13}C NMR (175 MHz, $CDCl_3$) spectrum of compound **9**.

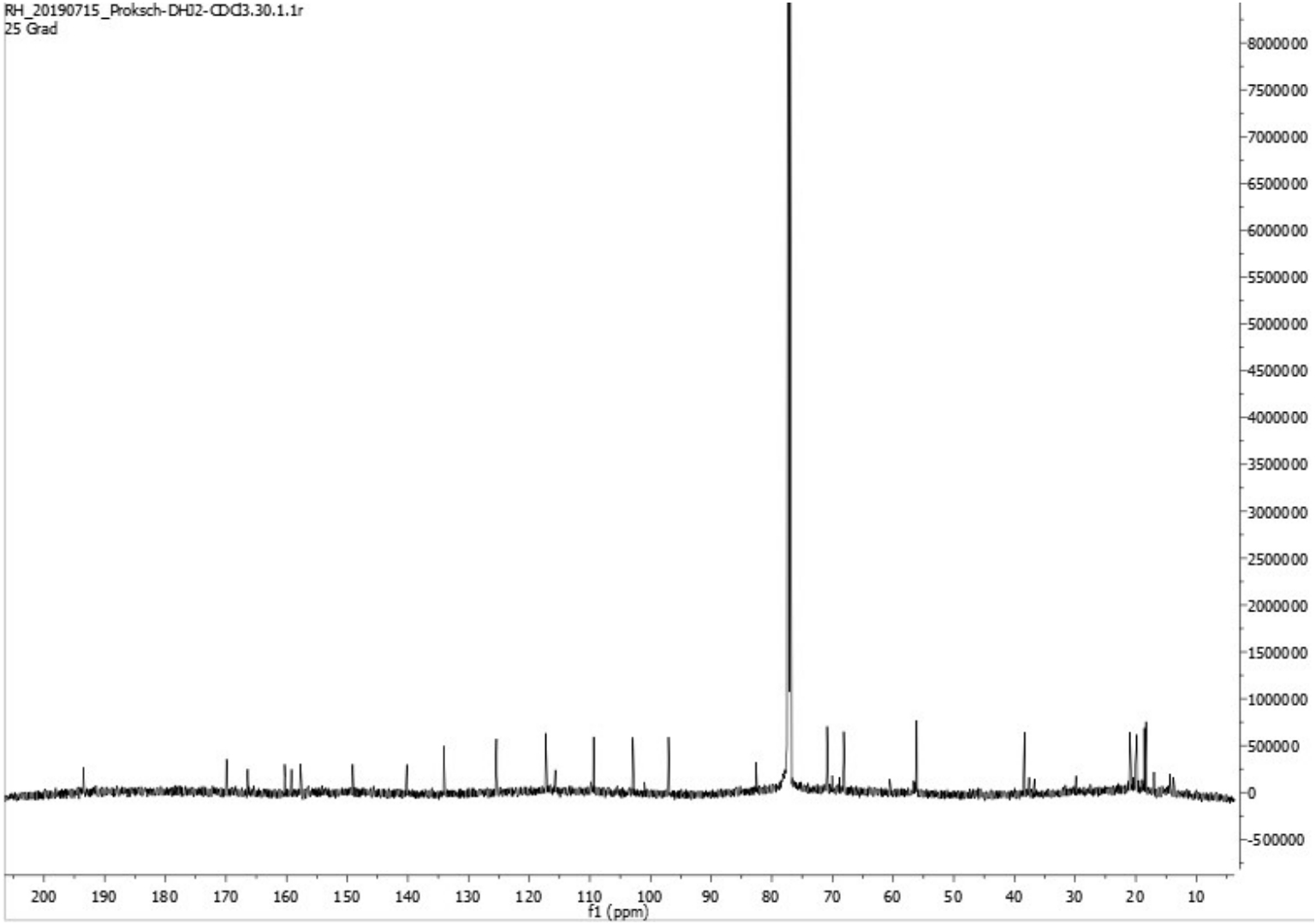

Figure S34. COSY (700 MHz, $CDCl_3$) spectrum of compound **9**.

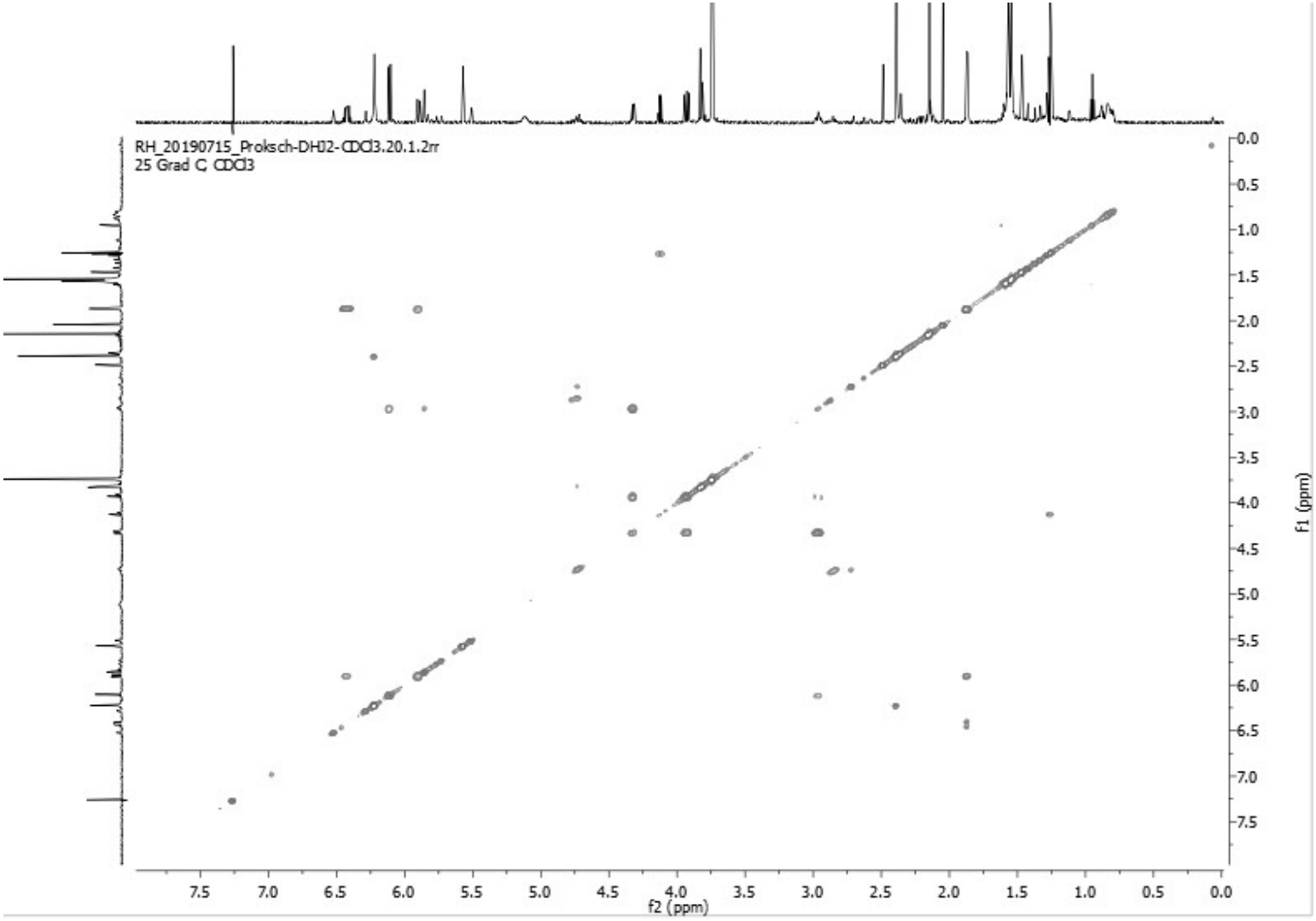

Figure S35. HSQC (700 MHz/175 MHz, $CDCl_3$) spectrum of compound **9**.

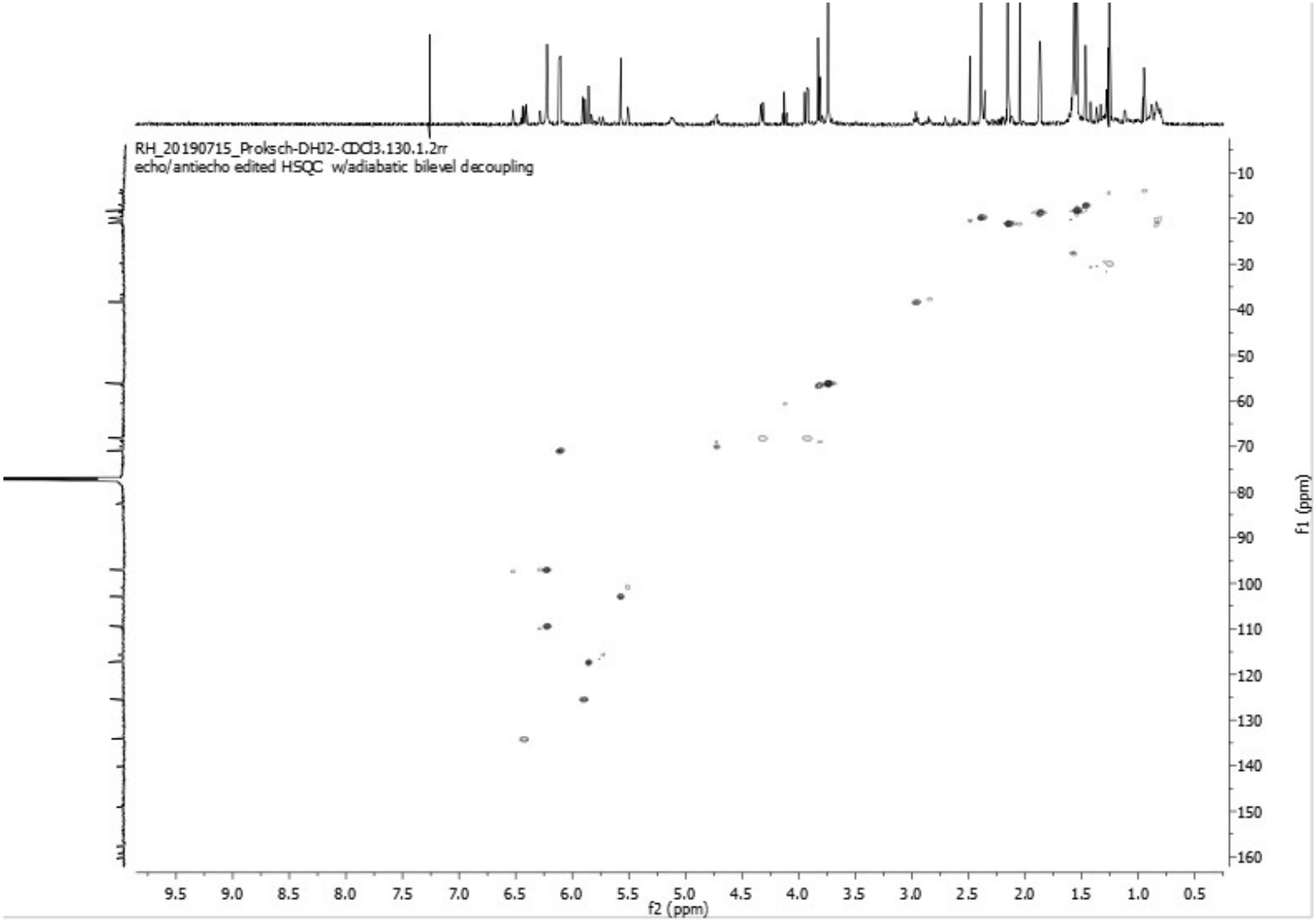

Figure S36. HMBC (700 MHz/175 MHz, $CDCl_3$) spectrum of compound **9**.

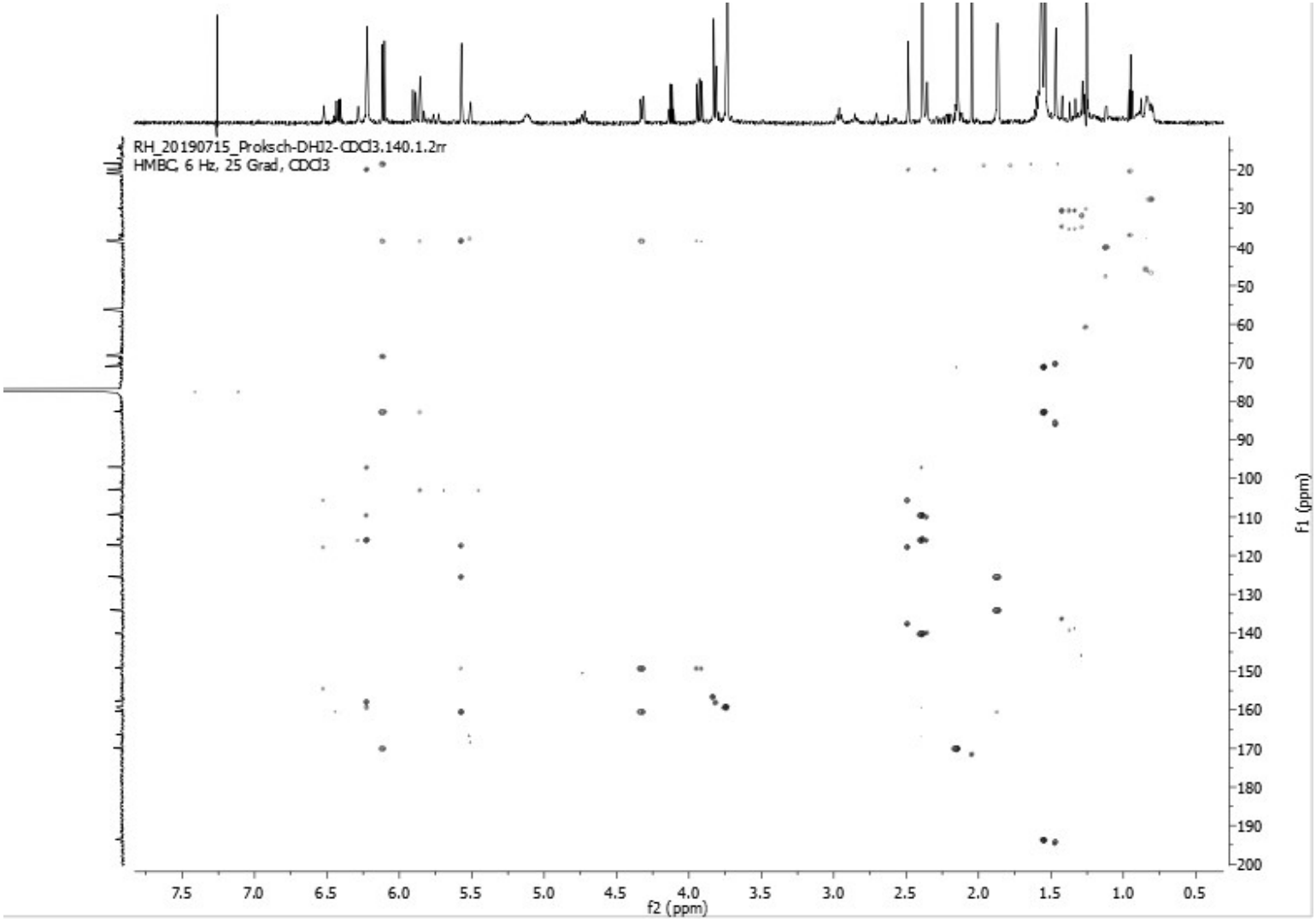

Figure S37. ROESY (700 MHz, $CDCl_3$) spectrum of compound **9**.

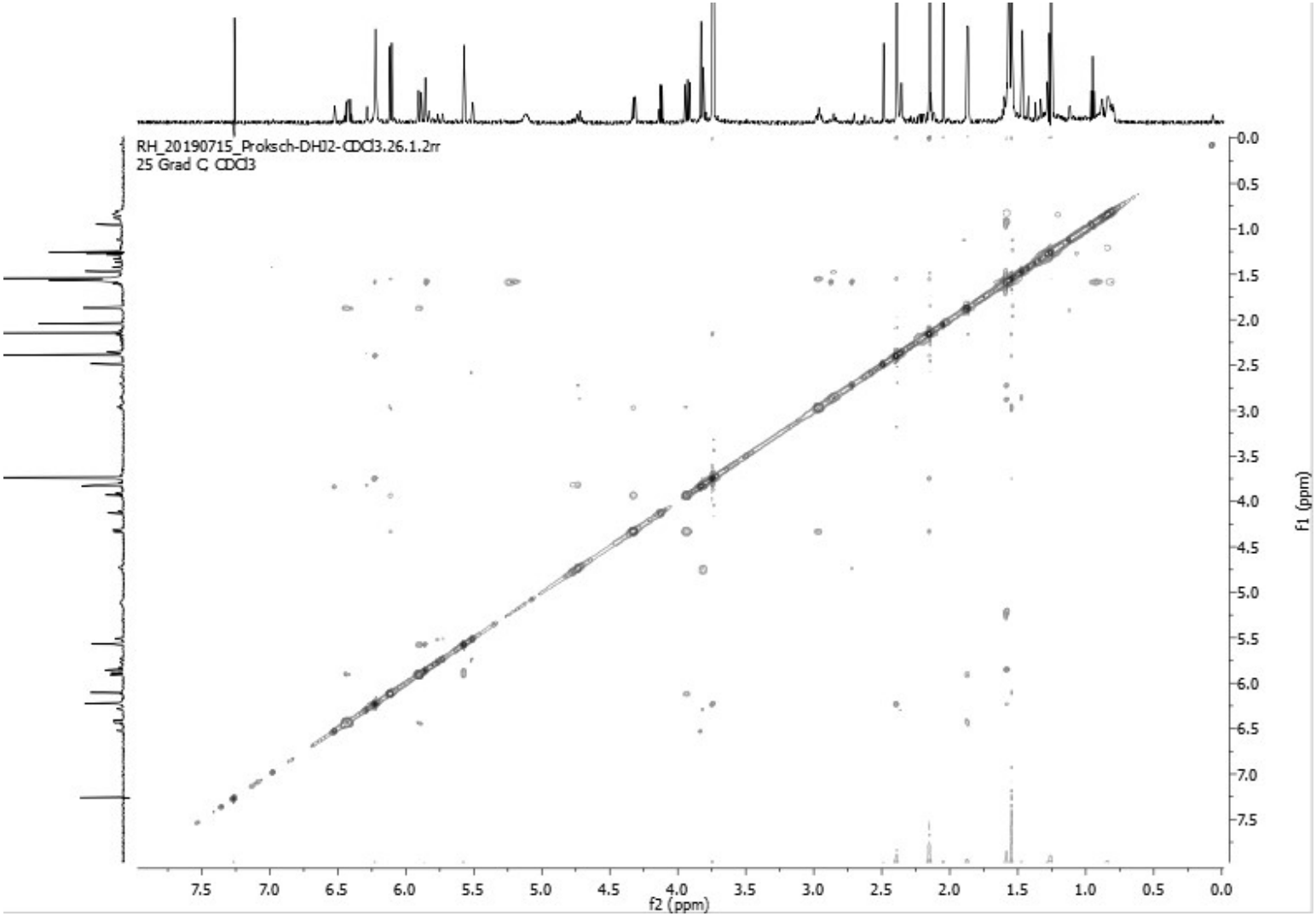

Figure S38. Section of the packing diagram over an extended unit cell of compound **5** (50% thermal elipsoids) determined by single-crystal x-ray diffraction. Hydrogen bonds are shown in dashed yellow lines.

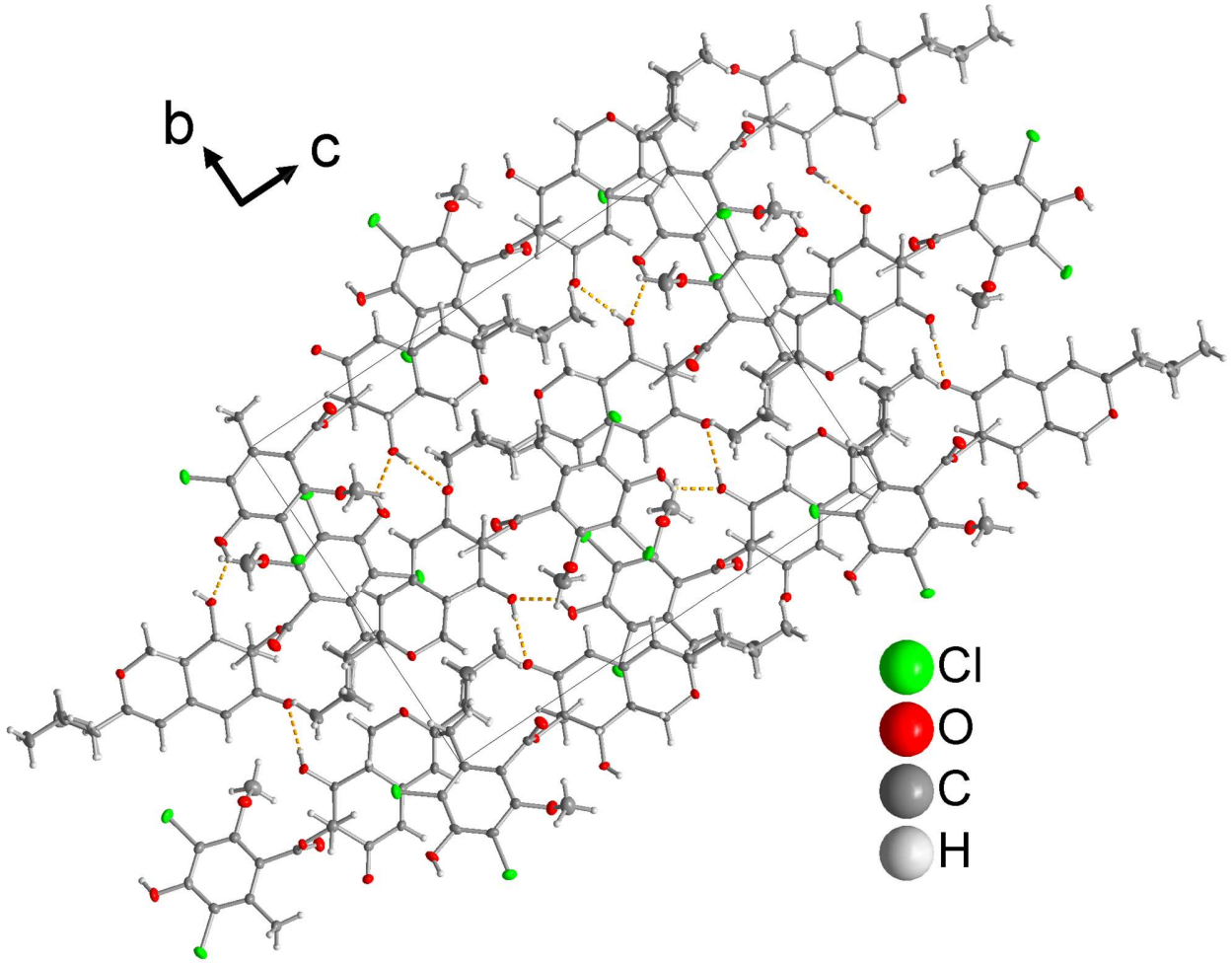

Figure S39. NFκB inhibitory potential of the compounds **1**, **3, 4**, **5**, **6**, **7**, **8**, **9** and **11**. Quantification of NFκB-dependent luciferase activity was performed by NFκB inhibition assay. In short, NFκB-MDA-MB-231 cells were pre-treated with the twofold serial diluted compound starting with 400 µM (**3**, **6**, **11**) or 200 µM to 0.78 µM (**1**, **4**, **5**, **7**, **9**, **11**) or left untreated (**Neg**). TNFα incubation induced NFκB activation, with untreated cells showing the maximal NFκB activity (**TNFα**). For RLU normalization, the RLU at the lowest concentration (0.78 µM) in each individual experiment was set as 100 %. Each data point represents the mean of at least three independent experiments. After the logarithmic transformation of the compound concentration in molar, nonlinear regression analysis without curve fitting was applied for data illustration using GraphPad Prism (GraphPad Software, San Diego, USA; Version 8.1.2). (M) Compound concentration in molar.

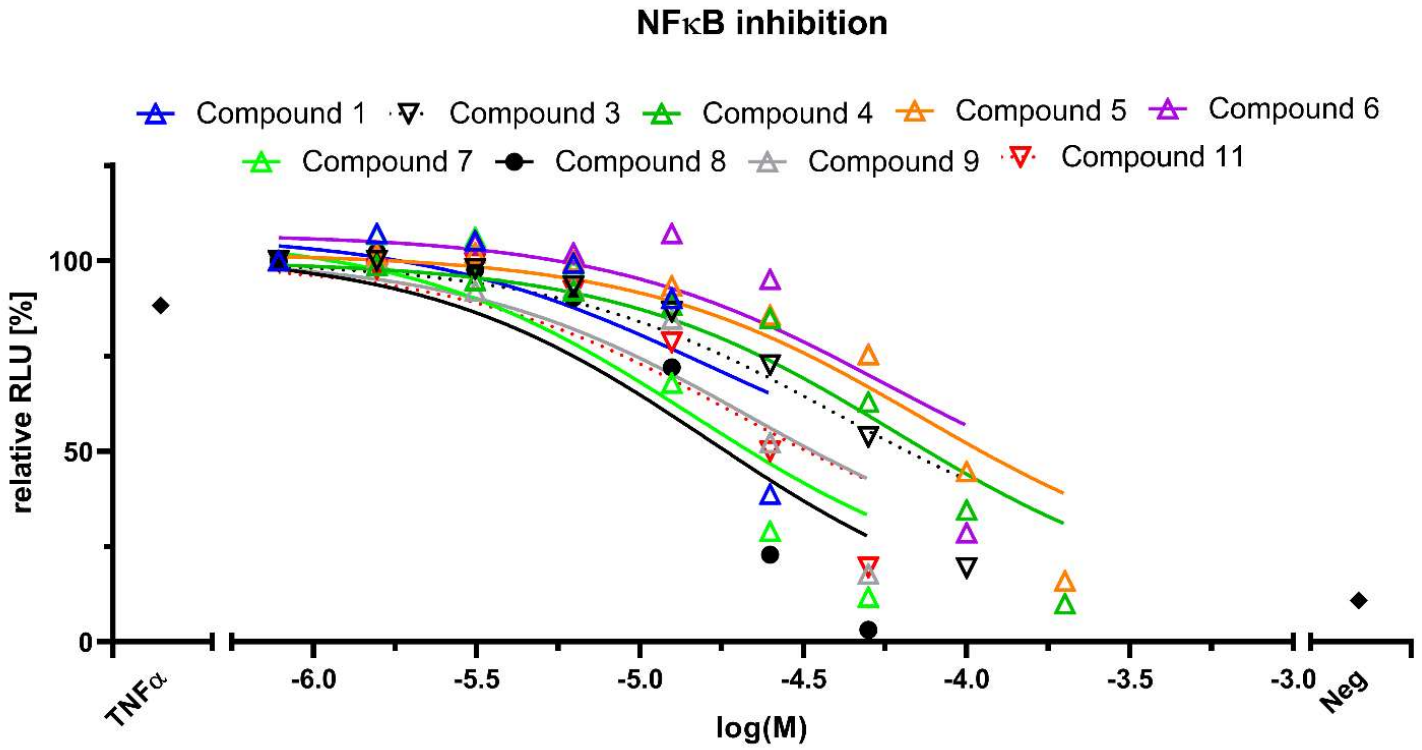

Figure S40. Potency in NFκB inhibition versus cytotoxicity. The scatterplot displays the pIC50 for the cell viability plotted against the pIC50 for the NFκB inhibition assay for the compounds **1**, **3**, **4**, **5**, **6**, **7**, **8**, **9** and **11**. The pIC_{50} value is determined as the negative decadic logarithm of the IC_{50}, which was calculated by nonlinear regression analysis without curve fitting using GraphPad Prism (GraphPad Software, San Diego, USA; Version 8.1.2). Compounds above the dotted line show greater potency in NFκB inhibition vs. cell viability and vice versa.

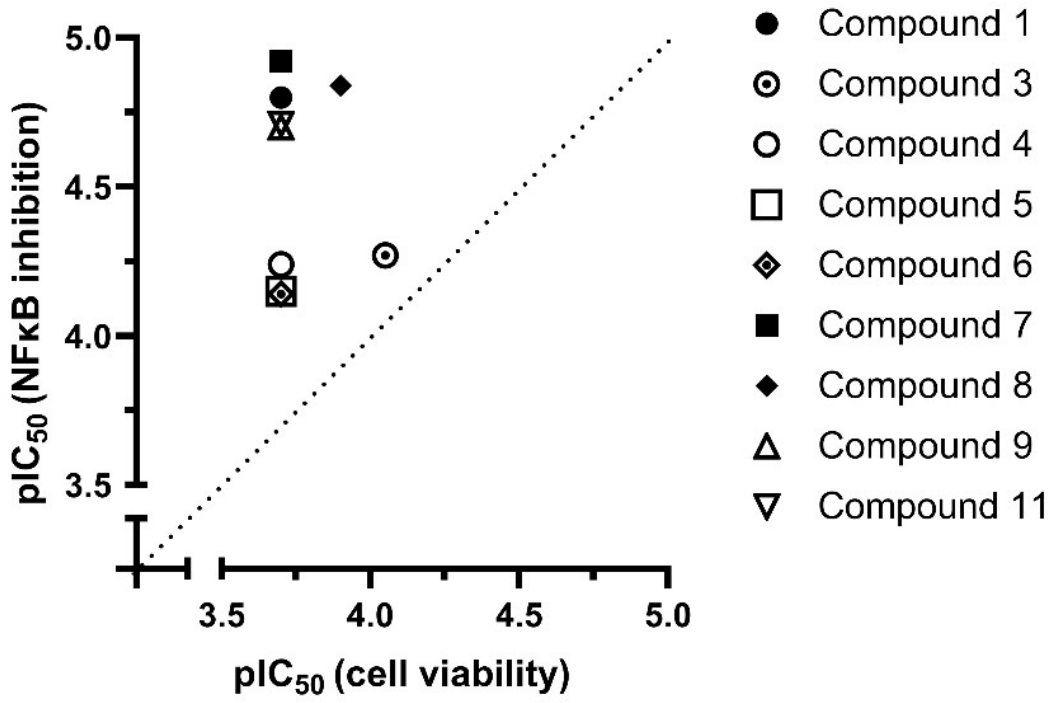

Table S1: Crystal data for compound **5**.

CCDC number	1976223
Empirical formula	$C_{22}H_{24}C_2O_7$
M [g mol^{-1}]	471.31
Crystal size [mm^3]	0.3 x 0.3 x 0.3
Temperature [K]	140
θ range [°] (completeness)	3.9 – 65.9 (0.99)
h; k; l range	±9; -14 – 16; -21 – 22
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
a [Å]	8.1520(5)
b [Å]	14.1088(9)
c [Å]	18.7346(12)
α [°]	90.0
β [°]	90.0
γ [°]	90.0
V [Å^3]	2154.8(2)
Z	4
Dcalc [mg m^{-3}]	1.453
μ (Cu Kα) [mm^{-1}]	3.08
F(000)	984
Max./min. transmission	0.753 / 0.600
Reflections collected	26106
Independent reflect. (Rint)	3718
Data/restraints/parameters	3718 / 311 / 0
Max./min. Δρ [eÅ^{-3}] [a]	-0.22 / 0.43
R_1/wR_2 [I>2σ(I)] [b]	0.026 / 0.069
R_1/wR_2 [all data] [b]	0.026 / 0.069
Goodness-of-fit on F^2 [c]	1.03
Flack parameter [d]	0.016(5)

[a] Largest difference peak and hole; [b] $R_1 = [\sum(||F_o| - |F_c||)/\sum|F_o|]$; $wR_2 = [\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$; [c] Goodness-of-fit $= [\sum[w(F_o^2 - F_c^2)^2]/(n - p)]^{1/2}$; [d] Absolute structure parameter.

Table S2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2) for compound **5**.

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl1	0.24321 (8)	0.81549 (4)	1.04217 (3)	0.03219 (17)	
O1	0.5589 (4)	0.69409 (13)	0.53721 (10)	0.0493 (7)	
O1"	0.2929 (2)	0.53104 (14)	0.82263 (9)	0.0280 (4)	
C1"	0.3988 (3)	0.55041 (16)	0.86460 (12)	0.0197 (5)	
C1A	0.5101 (5)	0.70018 (19)	0.61155 (13)	0.0388 (8)	
H1AA	0.389878	0.691728	0.615360	0.047*	
H1AB	0.537897	0.763740	0.630398	0.047*	
Cl2	0.39219 (8)	0.46808 (5)	1.13798 (3)	0.02943 (16)	
O2	0.5498 (2)	0.38313 (11)	0.78063 (9)	0.0257 (4)	
O2"	0.3218 (2)	0.73236 (13)	0.89865 (10)	0.0303 (4)	
C2"	0.3698 (3)	0.57644 (17)	0.94185 (12)	0.0205 (5)	
O3	0.5598 (2)	0.55473 (11)	0.84990 (8)	0.0181 (3)	
O3"	0.2951 (2)	0.66247 (14)	1.15017 (9)	0.0262 (4)	
C3	0.5456 (5)	0.6063 (2)	0.50865 (14)	0.0372 (7)	
C3"	0.3220 (3)	0.66943 (17)	0.95431 (13)	0.0221 (5)	
C3'	0.4783 (5)	0.6862 (3)	0.31316 (14)	0.0468 (9)	
H3'A	0.402981	0.733835	0.293872	0.056*	
H3'B	0.451852	0.623986	0.292861	0.056*	
H3'C	0.591316	0.703135	0.300684	0.056*	
H3"O	0.245 (5)	0.711 (3)	1.155 (2)	0.045 (10)*	
O4	0.6083 (2)	0.71764 (12)	0.76564 (9)	0.0224 (4)	
C4	0.5357 (3)	0.52728 (18)	0.54849 (12)	0.0253 (5)	
H4	0.514439	0.468460	0.525616	0.030*	
C4"	0.2934 (3)	0.69915 (17)	1.02408 (14)	0.0226 (5)	
C4A	0.5564 (3)	0.52915 (17)	0.62462 (12)	0.0193 (5)	
H4O	0.557 (5)	0.760 (3)	0.752 (2)	0.048 (11)*	
C5	0.5467 (3)	0.45072 (16)	0.66601 (12)	0.0198 (5)	
H5	0.528414	0.391237	0.643617	0.024*	
C5"	0.3139 (3)	0.63701 (18)	1.08111 (12)	0.0197 (5)	

C6	0.5628 (3)	0.45355 (16)	0.74267 (12)	0.0182 (5)	
C6"	0.3654 (3)	0.54475 (17)	1.06651 (12)	0.0198 (5)	
C7	0.6131 (3)	0.54889 (16)	0.77595 (11)	0.0179 (5)	
C7"	0.3956 (3)	0.51319 (16)	0.99741 (12)	0.0203 (5)	
C8	0.5449 (3)	0.63334 (15)	0.73403 (12)	0.0175 (5)	
H8	0.422487	0.633599	0.737541	0.021*	
C8"	0.1640 (4)	0.7509 (2)	0.86995 (18)	0.0431 (7)	
H8"A	0.172803	0.799089	0.832466	0.065*	
H8"B	0.091529	0.773985	0.907890	0.065*	
H8"C	0.118724	0.692472	0.849679	0.065*	
C8A	0.5954 (3)	0.62553 (16)	0.65544 (12)	0.0218 (5)	
H8A	0.716405	0.636047	0.651994	0.026*	
C9	0.7995 (3)	0.55032 (18)	0.78099 (13)	0.0242 (5)	
H9A	0.846684	0.543759	0.733123	0.036*	
H9B	0.836613	0.497662	0.811049	0.036*	
H9C	0.835308	0.610455	0.802039	0.036*	
C9"	0.4537 (4)	0.41479 (18)	0.98344 (13)	0.0278 (6)	
H9"A	0.458174	0.403747	0.931836	0.042*	
H9"B	0.377677	0.369501	1.005353	0.042*	
H9"C	0.563290	0.406388	1.003953	0.042*	
C1'A	0.5597 (12)	0.6085 (3)	0.42905 (19)	0.0314 (18)	0.775 (19)
H1'A	0.676302	0.618190	0.416185	0.038*	0.775 (19)
H1'B	0.526074	0.545947	0.410056	0.038*	0.775 (19)
C2'A	0.4610 (8)	0.6823 (5)	0.3940 (3)	0.0348 (14)	0.775 (19)
H2'A	0.492628	0.744633	0.413960	0.042*	0.775 (19)
H2'B	0.344101	0.671564	0.405934	0.042*	0.775 (19)
C1'B	0.467 (3)	0.6112 (11)	0.4296 (7)	0.027 (4)	0.225 (19)
H1'C	0.479037	0.549208	0.405513	0.033*	0.225 (19)
H1'D	0.348780	0.626511	0.432761	0.033*	0.225 (19)
C2'B	0.553 (7)	0.6847 (19)	0.3890 (14)	0.087 (13)	0.225 (19)
H2'C	0.671573	0.670387	0.386669	0.104*	0.225 (19)
H2'D	0.538411	0.747193	0.412247	0.104*	0.225 (19)

Table S3. Atomic displacement parameters (Å^2) for compound **5**.

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0442 (4)	0.0200 (3)	0.0323 (3)	0.0081 (3)	0.0045 (3)	-0.0070 (2)
O1	0.120 (2)	0.0177 (9)	0.0104 (8)	-0.0031 (12)	0.0016 (11)	0.0000 (7)
O1"	0.0299 (9)	0.0345 (10)	0.0196 (8)	-0.0048 (8)	0.0009 (7)	-0.0044 (8)
C1"	0.0300 (12)	0.0145 (11)	0.0147 (10)	-0.0003 (10)	0.0004 (10)	-0.0010 (9)
C1A	0.088 (2)	0.0173 (13)	0.0114 (11)	0.0047 (14)	-0.0014 (13)	-0.0024 (10)
Cl2	0.0390 (3)	0.0322 (3)	0.0171 (3)	0.0051 (3)	-0.0005 (2)	0.0059 (2)
O2	0.0435 (10)	0.0141 (8)	0.0197 (8)	-0.0005 (7)	-0.0004 (8)	0.0014 (7)
O2"	0.0374 (10)	0.0263 (10)	0.0271 (10)	0.0031 (8)	0.0031 (8)	0.0060 (8)
C2"	0.0271 (11)	0.0190 (11)	0.0155 (11)	-0.0006 (9)	0.0041 (10)	-0.0026 (9)
O3	0.0259 (8)	0.0174 (8)	0.0110 (7)	0.0009 (6)	0.0014 (6)	-0.0010 (6)
O3"	0.0345 (10)	0.0285 (10)	0.0158 (9)	0.0020 (8)	0.0052 (7)	-0.0067 (7)
C3	0.073 (2)	0.0224 (13)	0.0163 (12)	0.0002 (14)	0.0015 (13)	-0.0033 (10)
C3"	0.0278 (12)	0.0189 (12)	0.0194 (12)	0.0008 (10)	0.0027 (10)	0.0010 (10)
C3'	0.074 (2)	0.0469 (19)	0.0195 (14)	0.0284 (17)	-0.0023 (14)	0.0035 (13)
O4	0.0379 (9)	0.0118 (8)	0.0175 (8)	0.0005 (8)	-0.0054 (7)	-0.0028 (7)
C4	0.0429 (14)	0.0179 (12)	0.0152 (11)	0.0005 (11)	0.0006 (10)	-0.0049 (9)
C4"	0.0264 (12)	0.0159 (11)	0.0255 (12)	0.0019 (9)	0.0045 (10)	-0.0049 (10)
C4A	0.0237 (11)	0.0166 (11)	0.0175 (11)	0.0009 (9)	0.0029 (9)	-0.0036 (9)
C5	0.0282 (11)	0.0142 (11)	0.0171 (11)	0.0004 (10)	0.0015 (9)	-0.0036 (9)
C5"	0.0197 (10)	0.0243 (12)	0.0152 (11)	-0.0002 (9)	0.0035 (9)	-0.0053 (10)
C6	0.0219 (11)	0.0144 (11)	0.0182 (11)	0.0023 (9)	0.0026 (9)	-0.0016 (9)
C6"	0.0218 (11)	0.0215 (12)	0.0162 (11)	0.0013 (9)	0.0009 (9)	0.0020 (9)
C7	0.0265 (11)	0.0155 (11)	0.0116 (10)	0.0013 (9)	0.0013 (9)	-0.0022 (9)
C7"	0.0250 (11)	0.0179 (11)	0.0180 (11)	0.0008 (9)	0.0023 (9)	-0.0013 (9)
C8	0.0263 (11)	0.0121 (10)	0.0143 (10)	-0.0005 (9)	-0.0004 (9)	-0.0028 (9)
C8"	0.0405 (16)	0.0493 (18)	0.0396 (17)	0.0110 (14)	0.0018 (14)	0.0076 (14)
C8A	0.0376 (13)	0.0140 (11)	0.0138 (11)	-0.0037 (10)	0.0030 (10)	-0.0021 (9)
C9	0.0245 (12)	0.0245 (12)	0.0235 (12)	0.0019 (10)	-0.0018 (10)	-0.0021 (10)
C9"	0.0462 (15)	0.0183 (12)	0.0190 (12)	0.0030 (12)	0.0069 (11)	-0.0019 (10)
C1'A	0.054 (5)	0.0257 (19)	0.0148 (18)	0.009 (2)	0.000 (2)	-0.0013 (13)
C2'A	0.049 (3)	0.041 (3)	0.014 (2)	0.014 (3)	-0.002 (2)	-0.0002 (19)
C1'B	0.013 (9)	0.053 (9)	0.016 (6)	-0.002 (6)	-0.002 (6)	-0.013 (6)
C2'B	0.18 (4)	0.040 (11)	0.038 (11)	-0.02 (2)	0.04 (2)	0.009 (9)

Table S4. Geometric parameters (Å, º) for compound **5**.

Cl1—C4"	1.725 (2)	C4A—C5	1.354 (3)
O1—C3	1.353 (3)	C4A—C8A	1.511 (3)
O1—C1A	1.451 (3)	C5—C6	1.443 (3)
O1"—C1"	1.199 (3)	C5—H5	0.9500
C1"—O3	1.342 (3)	C5"—C6"	1.395 (4)
C1"—C2"	1.512 (3)	C6—C7	1.538 (3)
C1A—C8A	1.506 (4)	C6"—C7"	1.391 (3)
C1A—H1AA	0.9900	C7—C9	1.523 (3)
C1A—H1AB	0.9900	C7—C8	1.532 (3)
Cl2—C6"	1.735 (2)	C7"—C9"	1.490 (3)
O2—C6	1.226 (3)	C8—C8A	1.533 (3)
O2"—C3"	1.370 (3)	C8—H8	1.0000
O2"—C8"	1.419 (4)	C8"—H8"A	0.9800
C2"—C7"	1.387 (3)	C8"—H8"B	0.9800
C2"—C3"	1.388 (3)	C8"—H8"C	0.9800
O3—C7	1.454 (3)	C8A—H8A	1.0000
O3"—C5"	1.351 (3)	C9—H9A	0.9800
O3"—H3"O	0.80 (4)	C9—H9B	0.9800
C3—C4	1.344 (4)	C9—H9C	0.9800
C3—C1'A	1.496 (4)	C9"—H9"A	0.9800
C3—C1'B	1.614 (14)	C9"—H9"B	0.9800
C3"—C4"	1.392 (4)	C9"—H9"C	0.9800
C3'—C2'A	1.523 (6)	C1'A—C2'A	1.470 (8)
C3'—C2'B	1.55 (4)	C1'A—H1'A	0.9900
C3'—H3'A	0.9800	C1'A—H1'B	0.9900
C3'—H3'B	0.9800	C2'A—H2'A	0.9900
C3'—H3'C	0.9800	C2'A—H2'B	0.9900
O4—C8	1.426 (3)	C1'B—C2'B	1.46 (4)
O4—H4O	0.77 (4)	C1'B—H1'C	0.9900
C4—C4A	1.436 (3)	C1'B—H1'D	0.9900
C4—H4	0.9500	C2'B—H2'C	0.9900
C4"—C5"	1.392 (4)	C2'B—H2'D	0.9900
C3—O1—C1A	114.3 (2)	C2"—C7"—C6"	117.7 (2)
O1"—C1"—O3	125.4 (2)	C2"—C7"—C9"	121.1 (2)
O1"—C1"—C2"	124.8 (2)	C6"—C7"—C9"	121.2 (2)

O3—C1"—C2"	109.79 (19)	O4—C8—C7	107.72 (18)
O1—C1A—C8A	110.9 (2)	O4—C8—C8A	111.21 (19)
O1—C1A—H1AA	109.5	C7—C8—C8A	109.82 (18)
C8A—C1A—H1AA	109.5	O4—C8—H8	109.3
O1—C1A—H1AB	109.5	C7—C8—H8	109.3
C8A—C1A—H1AB	109.5	C8A—C8—H8	109.3
H1AA—C1A—H1AB	108.1	O2"—C8"—H8"A	109.5
C3"—O2"—C8"	114.1 (2)	O2"—C8"—H8"B	109.5
C7"—C2"—C3"	121.6 (2)	H8"A—C8"—H8"B	109.5
C7"—C2"—C1"	122.6 (2)	O2"—C8"—H8"C	109.5
C3"—C2"—C1"	115.8 (2)	H8"A—C8"—H8"C	109.5
C1"—O3—C7	119.03 (17)	H8"B—C8"—H8"C	109.5
C5"—O3"—H3"O	113 (3)	C1A—C8A—C4A	108.9 (2)
C4—C3—O1	123.0 (2)	C1A—C8A—C8	110.5 (2)
C4—C3—C1'A	125.1 (3)	C4A—C8A—C8	112.05 (19)
O1—C3—C1'A	111.7 (3)	C1A—C8A—H8A	108.4
C4—C3—C1'B	121.4 (6)	C4A—C8A—H8A	108.4
O1—C3—C1'B	110.8 (6)	C8—C8A—H8A	108.4
O2"—C3"—C2"	119.0 (2)	C7—C9—H9A	109.5
O2"—C3"—C4"	121.3 (2)	C7—C9—H9B	109.5
C2"—C3"—C4"	119.3 (2)	H9A—C9—H9B	109.5
C2'A—C3'—H3'A	109.5	C7—C9—H9C	109.5
C2'A—C3'—H3'B	109.5	H9A—C9—H9C	109.5
H3'A—C3'—H3'B	109.5	H9B—C9—H9C	109.5
C2'A—C3'—H3'C	109.5	C7"—C9"—H9"A	109.5
H3'A—C3'—H3'C	109.5	C7"—C9"—H9"B	109.5
H3'B—C3'—H3'C	109.5	H9"A—C9"—H9"B	109.5
C8—O4—H4O	108 (3)	C7"—C9"—H9"C	109.5
C3—C4—C4A	121.9 (2)	H9"A—C9"—H9"C	109.5
C3—C4—H4	119.0	H9"B—C9"—H9"C	109.5
C4A—C4—H4	119.0	C2'A—C1'A—C3	114.6 (5)
C5"—C4"—C3"	120.7 (2)	C2'A—C1'A—H1'A	108.6
C5"—C4"—Cl1	118.49 (19)	C3—C1'A—H1'A	108.6
C3"—C4"—Cl1	120.71 (19)	C2'A—C1'A—H1'B	108.6
C5—C4A—C4	123.2 (2)	C3—C1'A—H1'B	108.6
C5—C4A—C8A	121.9 (2)	H1'A—C1'A—H1'B	107.6
C4—C4A—C8A	114.9 (2)	C1'A—C2'A—C3'	114.7 (5)

C4A—C5—C6	122.8 (2)	C1'A—C2'A—H2'A	108.6
C4A—C5—H5	118.6	C3'—C2'A—H2'A	108.6
C6—C5—H5	118.6	C1'A—C2'A—H2'B	108.6
O3"—C5"—C4"	123.6 (2)	C3'—C2'A—H2'B	108.6
O3"—C5"—C6"	118.0 (2)	H2'A—C2'A—H2'B	107.6
C4"—C5"—C6"	118.3 (2)	C2'B—C1'B—C3	109 (2)
O2—C6—C5	123.2 (2)	C2'B—C1'B—H1'C	110.0
O2—C6—C7	119.8 (2)	C3—C1'B—H1'C	110.0
C5—C6—C7	116.9 (2)	C2'B—C1'B—H1'D	110.0
C7"—C6"—C5"	122.3 (2)	C3—C1'B—H1'D	110.0
C7"—C6"—Cl2	119.76 (18)	H1'C—C1'B—H1'D	108.4
C5"—C6"—Cl2	117.92 (18)	C1'B—C2'B—C3'	107 (3)
O3—C7—C9	103.80 (18)	C1'B—C2'B—H2'C	110.2
O3—C7—C8	109.62 (17)	C3'—C2'B—H2'C	110.2
C9—C7—C8	112.6 (2)	C1'B—C2'B—H2'D	110.2
O3—C7—C6	110.85 (18)	C3'—C2'B—H2'D	110.2
C9—C7—C6	107.64 (19)	H2'C—C2'B—H2'D	108.5
C8—C7—C6	112.06 (18)		

Table S5. Hydrogen-bond geometry (Å, º) for compound **5**.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3"—H3"*O*···Cl1	0.80 (4)	2.58 (4)	2.989 (2)	113 (3)
O3"—H3"*O*···O4^{i}	0.80 (4)	2.11 (4)	2.769 (2)	139 (3)
O4—H4*O*···O2^{ii}	0.77 (4)	2.04 (4)	2.804 (2)	171 (4)

Symmetry codes: (i) *x*-1/2, -*y*+3/2, -*z*+2; (ii) -*x*+1, *y*+1/2, -*z*+3/2.