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Self-condensation of 2-(ethoxalylmethyl)chromones into new derivatives of isotetronic acid

Vyacheslav Ya. Sosnovskikh, Daria A. Vetyugova, Alexander V. Safrygin, Oleg S. Eltsov and Pavel A. Slepukhin

IR spectra were recorded on a PerkinElmer Spectrum BX-II instrument with ATR accessory. ^{1}H and ^{13}C NMR spectra were acquired on a Bruker Avance II spectrometer (400 and 100 MHz, respectively) in DMSO-d_6 with TMS and residual solvent peaks as the references.

2,3-Diaminopyridinium 5-ethoxycarbonyl-4-(6-chloro-4-oxo-4H-chromen-2-yl)-5-[(6-chloro-4-oxo-4H-chromen-2-yl)methyl]-2-oxo-2,5-dihydrofuran-3-olate **4c**. This compound was prepared according to the procedure described for **4a**. Yield 79%, orange powder, mp 165–167 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.12 (t, *J* 7.1 Hz, 3H, Me-16), 3.68 (d, *J* 14.8 Hz, 1H, C*H*H-9), 3.83 (d, *J* 14.8 Hz, 1H, CH*H*-9), 4.17 (dq, *J* 11.4, 7.1 Hz, 1H, C*H*H-15), 4.21 (dq, *J* 11.4, 7.1 Hz, 1H, CH*H*-15), 5.76 (br s, 2H, NH_2), 6.17 (s, 1H, H-3), 6.73 (dd, *J* 7.6, 6.3 Hz, 1H, H-5 Py), 7.04 (d, *J* 7.6 Hz, 1H, H-4 Py), 7.05 (s, 1H, H-3′), 7.13 (d, *J* 8.9 Hz, 1H, H-8), 7.26 (d, *J* 6.1 Hz, 1H, H-6 Py), 7.37 (br s, 2H, NH_2), 7.48 (d, *J* 8.9 Hz, 1H, H-8′, *J* 8.8), 7.71 (dd, *J* 8.9, 2.7 Hz, 1H, H-7), 7.75 (dd, *J* 8.9, 2.7 Hz, 1H, H-7′), 7.85 (d, *J* 2.7 Hz, 1H, H-5), 7.86 (d, *J* 2.7 Hz, 1H, H-5′), 13.10 (br s, 1H, $N^{+}H$). ^{13}C NMR (100 MHz, DMSO-d_6) δ: 13.9, 61.8, 82.0, 100.1, 100.8, 109.9, 112.2, 113.6, 119.6, 119.7, 120.1, 122.5, 123.5, 123.8, 124.0, 125.3, 128.7, 129.8, 132.3, 133.3, 134.1, 144.7, 153.1, 154.2, 160.7, 162.6, 165.1, 168.7, 169.6, 173.5, 175.2. IR (ν/cm^{-1}): 3384, 3208, 1759, 1742, 1632, 1576, 1527. Found (%): C, 56.59; H, 3.40; N, 6.72. Calc. for $C_{31}H_{23}Cl_2N_3O_9 \cdot 0.33H_2O$ (%): C, 56.55; H, 3.62; N, 6.38.

‡*Ethyl 4-hydroxy-3-(6-methoxy-4-oxo-4*H-*chromen-2-yl)-2-[(6-methoxy-4-oxo-4*H-*chromen-2-yl)methyl]-5-oxo-2,5-dihydrofuran-2-carboxylate* **5b**. Yield 65%, yellow powder, mp 230–231 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.23 (t, *J* 7.1 Hz, 3H, Me-16), 3.76 (d, *J* 15.0 Hz, 1H, C*H*H-9), 3.82 (d, *J* 15.0 Hz, 1H, CH*H*-9), 3.84 (s, 3H, MeO), 3.91 (s, 3H, MeO), 4.27 (dq, *J* 10.7, 7.0 Hz, 1H, C*H*H-15), 4.34 (dq, *J* 10.7, 7.0 Hz, 1H, CH*H*-15), 6.15 (s, 1H, H-3), 6.85 (s, 1H, H-3′), 7.01 (d, *J* 9.1 Hz, 1H, H-8), 7.22 (dd, *J* 9.1, 3.1 Hz, 1H, H-7), 7.31 (d, *J* 3.1 Hz, 1H, H-5), 7.34 (dd, *J* 9.1, 3.1 Hz, H-7′), 7.42 (d, *J* 3.1 Hz, 1H, H-5′), 7.47 (d, *J* 9.1 Hz, 1H, H-8′), OH was not observed. ^{13}C NMR (100 MHz, DMSO-d_6) δ: 13.7, 38.2, 55.7, 55.8, 62.9, 82.8, 105.0,

105.1, 108.6, 112.0, 116.2, 119.1, 119.6, 123.3, 123.6, 123.9, 147.5, 149.5, 150.2, 153.8, 156.4, 156.6, 156.9, 162.5, 165.9, 166.8, 175.4, 176.0. IR (ν/cm^{-1}): 3098, 3006, 2962, 2939, 1781, 1751, 1652, 1612, 1563. HRMS (ESI): calcd for $C_{28}H_{23}O_{11}$ $[M+H]^+$ 535.1235, found 535.1227. Found (%): C, 61.38; H, 4.03. Calc. for $C_{28}H_{22}O_{11}\cdot 0.75H_2O$ (%): C, 61.37; H, 4.32.

*Ethyl 3-(6-chloro-4-oxo-4H-chromen-2-yl)-2-[(6-chloro-4-oxo-4*H*-chromen-2-yl)methyl]-4-hydroxy-5-oxo-2,5-dihydrofuran-2-carboxylate* **5c**. Yield 62%, brown powder, mp 244–246 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.24 (t, *J* 7.1 Hz, 3H, Me-16), 3.80 (d, *J* 15.1 Hz, 1H, C*H*H-9), 3.85 (d, *J* 15.1 Hz, 1H, CH*H*-9), 4.27 (dq, *J* 10.8, 7.1 Hz, 1H, C*H*H-15), 4.33 (dq, *J* 10.8, 7.1 Hz, 1H, CH*H*-15), 6.28 (s, 1H, H-3), 6.91 (s, 1H, H-3′), 7.17 (d, *J* 8.9 Hz, 1H, H-8), 7.58 (d, *J* 8.9 Hz, 1H, H-8′), 7.65 (dd, *J* 8.9, 2.6 Hz, 1H, H-7), 7.75 (dd, *J* 8.9, 2.6 Hz, 1H, H-7′), 7.88 (d, *J* 2.6 Hz, 1H, H-5), 7.97 (d, *J* 2.6 Hz, 1H, H-5′), OH was not observed. ^{13}C NMR (100 MHz, DMSO-d_6) δ: 13.7, 38.3, 62.9, 82.7, 108.8, 112.8, 114.7, 120.1, 120.5, 123.8, 123.9, 124.0, 124.4, 130.1, 130.2, 134.1, 134.3, 149.0, 153.4, 154.0, 157.3, 163.4, 165.9, 166.8, 174.5, 175.2. IR (ν/cm^{-1}): 3089, 2983, 2934, 1783, 1756, 1653, 1616, 1562. HRMS (ESI): calcd for $C_{26}H_{17}Cl_2O_9$ $[M+H]^+$ 543.0244, found 543.0232. Found (%): C, 56.53; H, 2.80. Calc. for $C_{26}H_{16}Cl_2O_9\cdot 0.5H_2O$ (%): C, 56.54; H, 3.10.

*Ethyl 3-(6-bromo-4-oxo-4H-chromen-2-yl)-2-[(6-bromo-4-oxo-4*H*-chromen-2-yl)methyl]-4-hydroxy-5-oxo-2,5-dihydrofuran-2-carboxylate* **5d**. Yield 72%, light green powder, mp 240–241 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.19 (t, *J* 7.1 Hz, 3H, Me-16), 3.83 (d, *J* 15.1 Hz, 1H, C*H*H-9), 3.88 (d, *J* 15.1 Hz, 1H, CH*H*-9), 4.25 (dq, *J* 10.8, 7.1 Hz, 1H, C*H*H-15), 4.32 (dq, *J* 10.8, 7.1 Hz, 1H, CH*H*-15), 6.32 (s, 1H, H-3), 6.89 (s, 1H, H-3′), 7.14 (d, *J* 8.9 Hz, 1H, H-8), 7.55 (d, *J* 8.9 Hz, 1H, H-8′), 7.84 (dd, *J* 8.9, 2.5 Hz, 1H, H-7), 7.94 (dd, *J* 8.9, 2.5 Hz, 1H, H-7′), 8.01 (d, *J* 2.5 Hz, 1H, H-5), 8.10 (d, *J* 2.5 Hz, 1H, H-5′), OH was not observed. ^{13}C NMR (100 MHz, DMSO-d_6) δ: 13.8, 38.4, 62.9, 82.7, 108.8, 112.9, 114.6, 118.0, 118.1, 120.3, 120.6, 124.4, 124.8, 126.9, 127.1, 136.9, 137.0, 149.1, 153.8, 154.4, 157.3, 163.4, 165.9, 166.8, 174.3, 175.1. IR (ν/cm^{-1}): 3086, 2978, 2932, 1781, 1754, 1652, 1619, 1558. HRMS (ESI): calcd for $C_{26}H_{17}Br_2O_9$ $[M+H]^+$ 632.9213, found 632.9229. Found (%): C, 48.72; H, 2.58. Calc. for $C_{26}H_{16}Br_2O_9\cdot 0.5H_2O$ (%): C, 48.70; H, 2.67.

Ethyl 3-(5,7-dimethyl-4-oxo-4H-chromen-2-yl)-2-[(5,7-dimethyl-4-oxo-4H-chromen-2-yl)methyl]-4-hydroxy-5-oxo-2,5-dihydrofuran-2-carboxylate **5e**. Yield 83%, yellow powder, mp 212–215 ºC. ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.15 (t, *J* 7.1 Hz, 3H, Me-16), 2.27 (s, 3H, Me), 2.39 (s, 3H, Me), 2.59 (s, 3H, Me), 2.70 (s, 3H, Me), 3.70 (d, *J* 15.0 Hz, 1H, C*H*H-9), 3.81 (d, *J* 15.0 Hz, 1H, CH*H*-9), 4.25 (dq, *J* 10.8, 7.1 Hz, 1H, C*H*H-15), 4.31 (dq, *J* 10.8, 7.1 Hz, 1H, CH*H*-15), 6.11 (s, 1H, H-3), 6.66 (s, 1H, H-8), 6.68 (s, 1H, H-3′), 6.94 (s, 1H, H-8′), 7.06 (s, 1H, H-6), 7.12 (s, 1H, H-6′), OH was not observed. ^{13}C NMR (100 MHz, DMSO-d_6) δ: 13.7, 20.9,

20.9, 21.8, 21.9, 37.9, 62.9, 82.8, 111.0, 113.9, 115.2, 115.5, 118.8, 119.1, 128.9, 129.3, 139.3, 139.4, 143.6, 144.1, 146.6, 154.5, 156.2, 156.9, 160.5, 165.9, 166.6, 177.8, 178.2, one C was not observed. IR (ν/cm^{-1}): 2978, 2934, 1787, 1753, 1682, 1626, 1606, 1561. Found (%): C, 65.39; H, 4.67. Calc. for $C_{30}H_{26}O_9 \cdot H_2O$ (%): C, 65.69; H, 5.15.

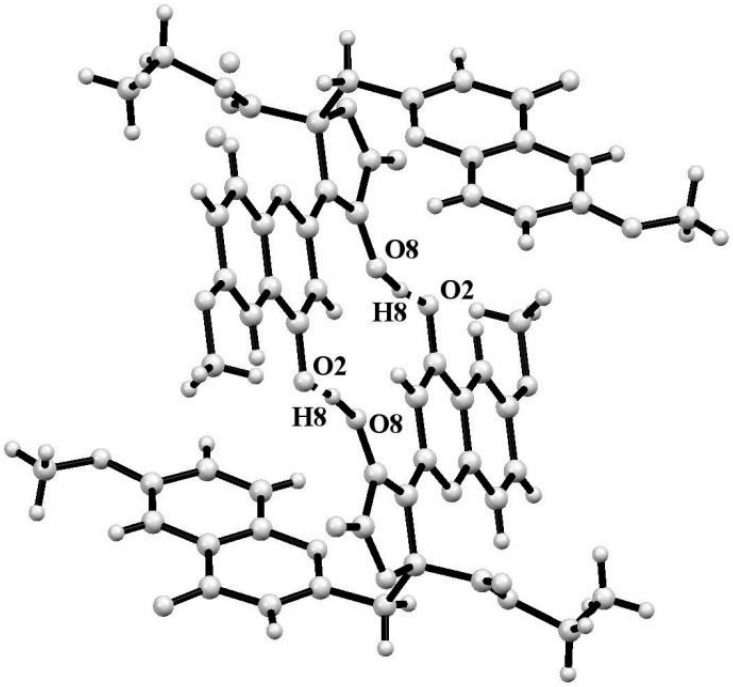


Figure S1 Intermolecular H-bonds in the crystals of compound **5b**.

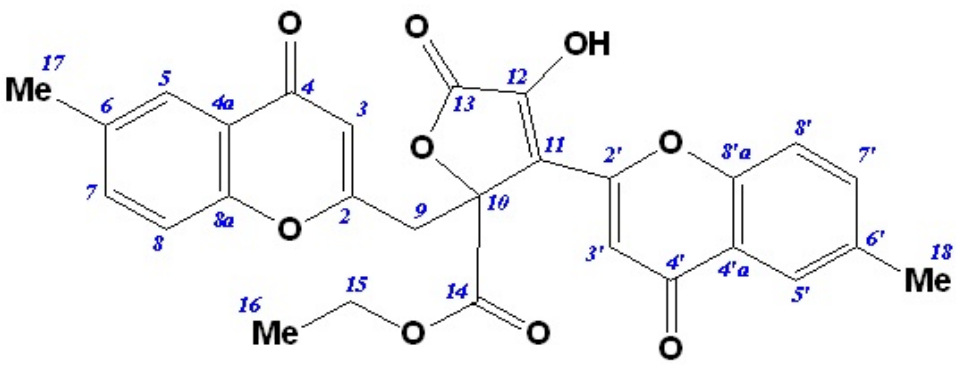
Single-crystal X-ray diffraction analysis was performed on an Xcalibur 3 automatic diffractometer using the standard procedure (MoK irradiation, graphite monochromator, ω-scanning with 1º step, 295(2) K). An empirical adjustment for absorption was introduced. The structure was solved and refined using the OLEX2 software package.[1] The structure was solved by the direct method using ShelXS program[2] and refined by full-matrix least squares method against F^2 using ShelXL program in anisotropic approximation for non-hydrogen atoms. The hydrogen atoms of C–H bonds were placed in geometrically calculated positions and included in the refinement in the "riding" model. The hydrogens of the O–H bonds were refined independently in isotropic approximation. The disordered solvent was excluded from the structure model using the SQWEEZE procedure.

References

1 O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Crystallogr*., 2009, **42**, 339.

2 G. M. Sheldrick, *Acta Crystallogr*., 2008, **A64**, 112.

^{1}H NMR



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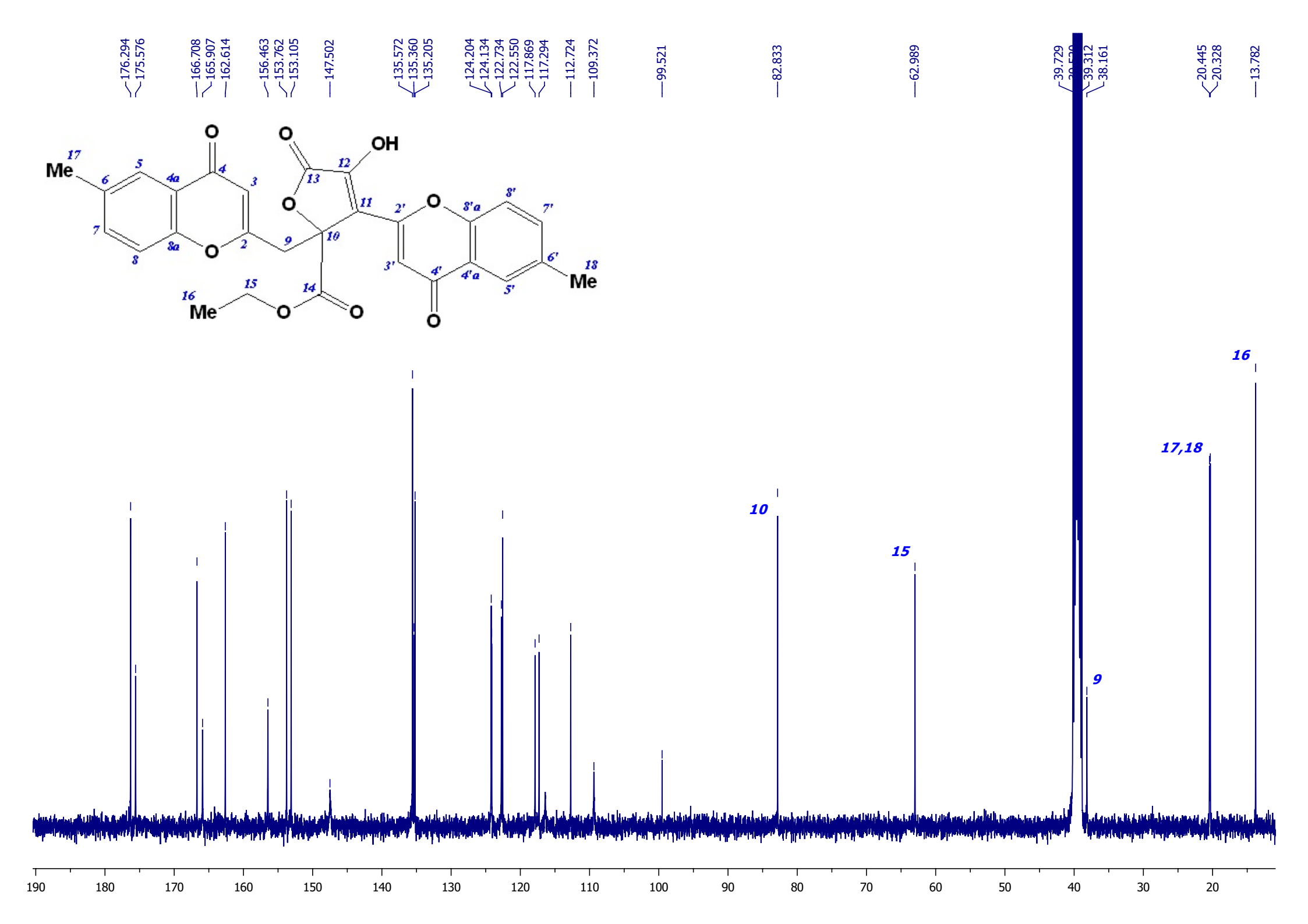
5' 5 7' 7,8' 8 3' 3 15 9 18 17 16

0.99 0.97 1.06 2.11 1.02 1.05 1.00 2.04 2.03 3.04 3.02 3.00

8.0 7.8 7.6 7.4 7.2 7.0 6.8 6.6 6.4 6.2 6.0 4.4 4.2 4.0 3.8 3.6 2.8 2.6 2.4 2.2 1.4 1.2 1.0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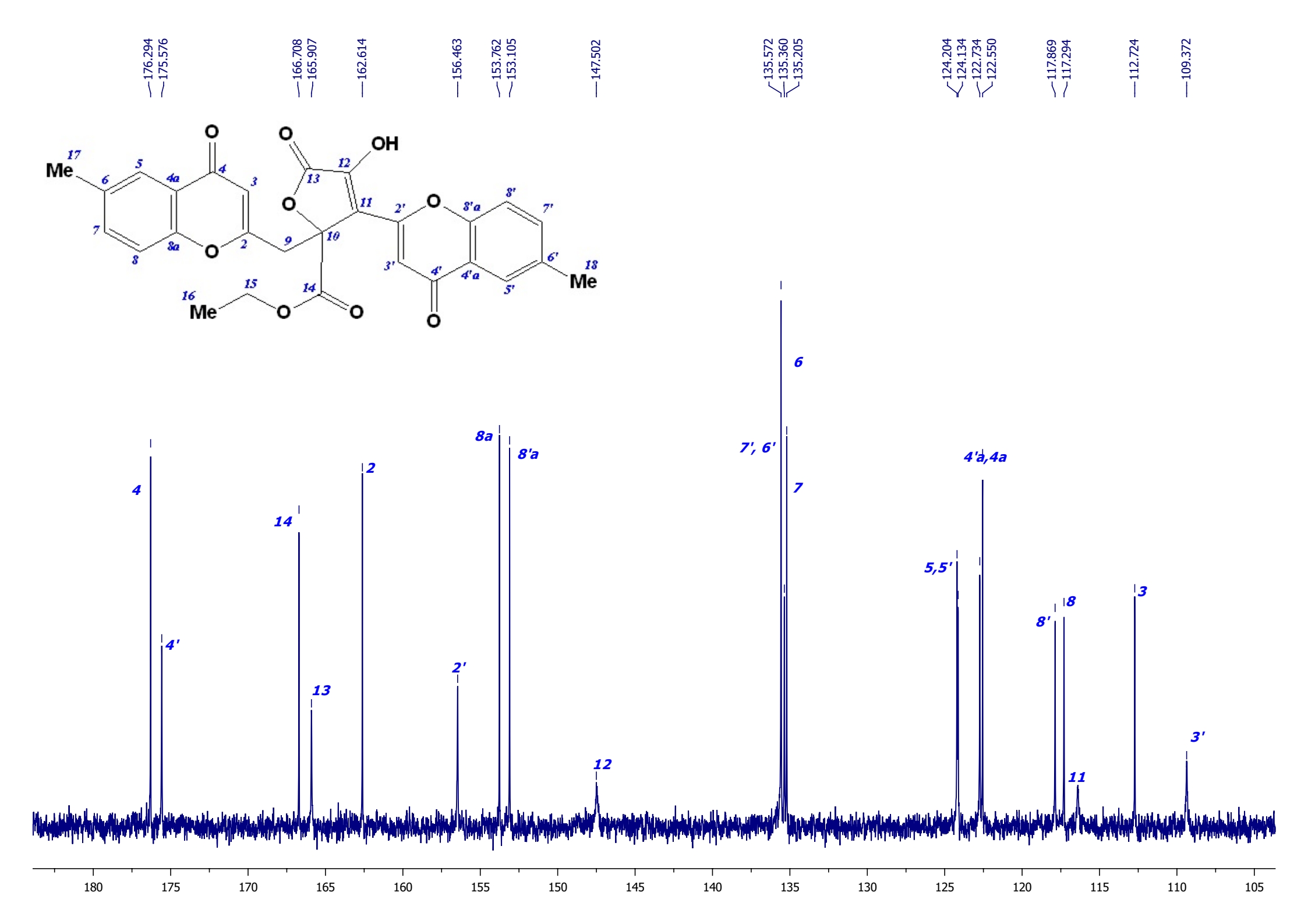
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122.550
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7'
8'
8'a
4
4'
14
13
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2'
8a
8'a
12
7', 6'
6
7
4'a,4a
5,5'
8'
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11
3'
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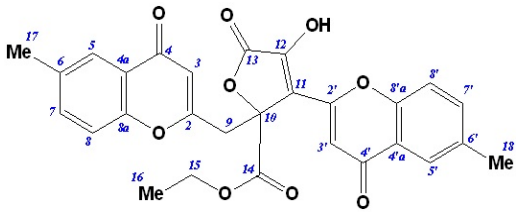
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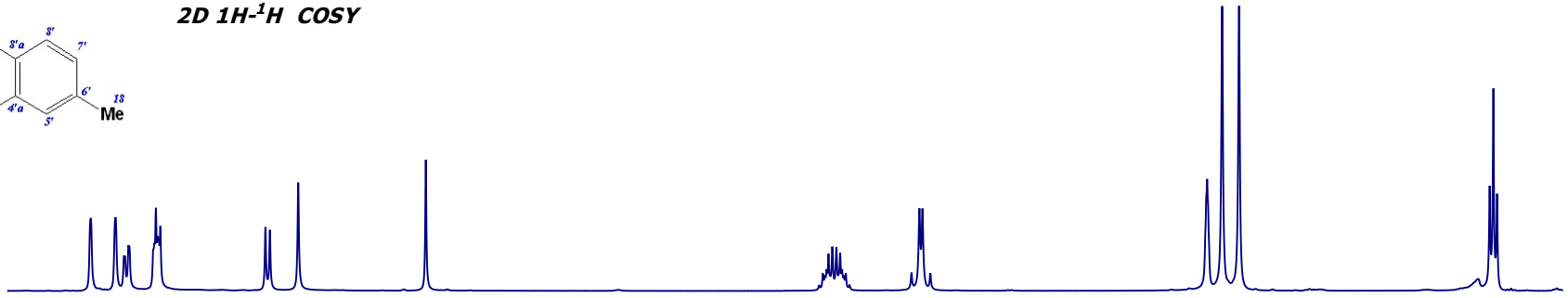
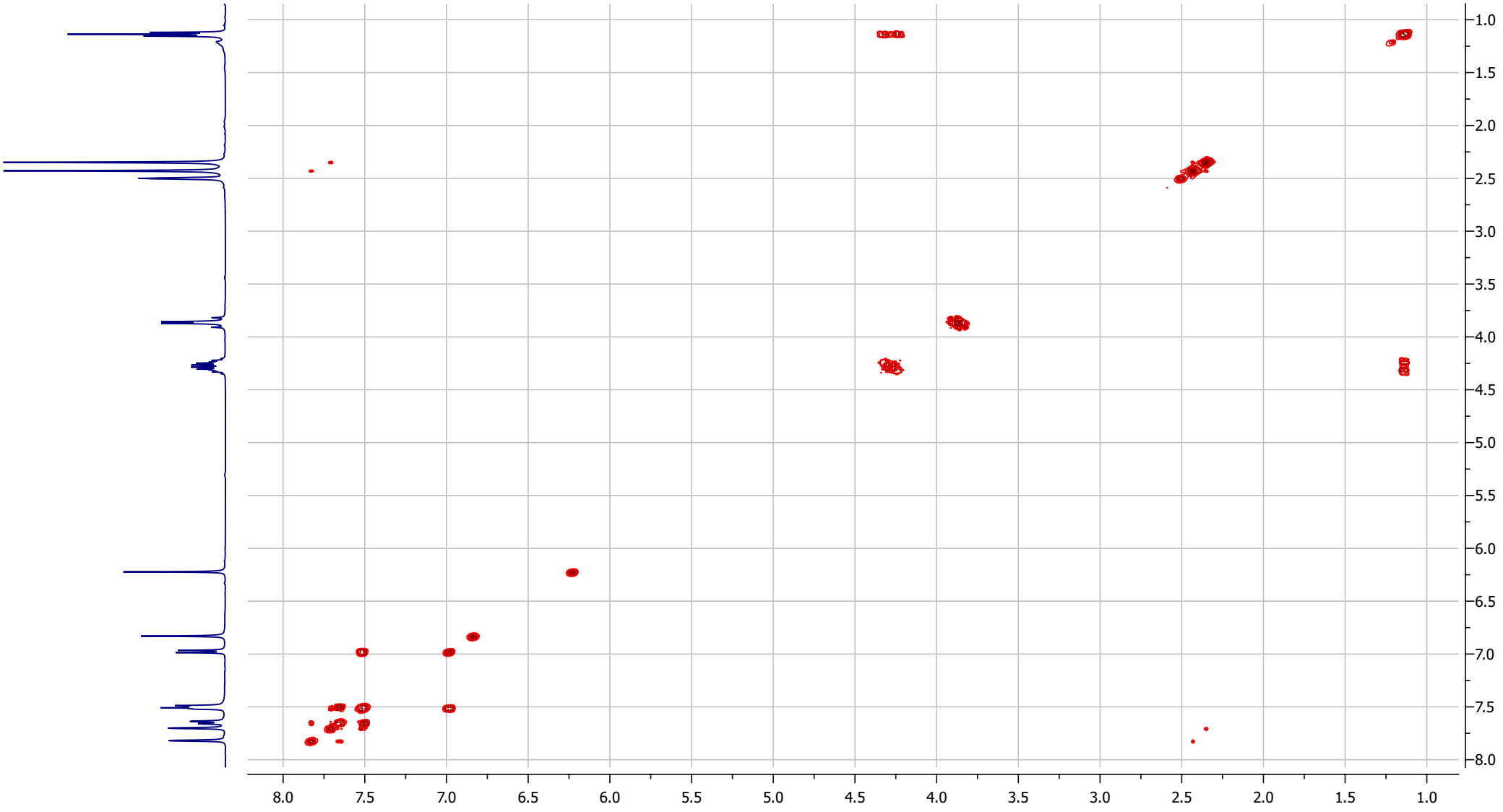
2D 1H-^{1}H COSY

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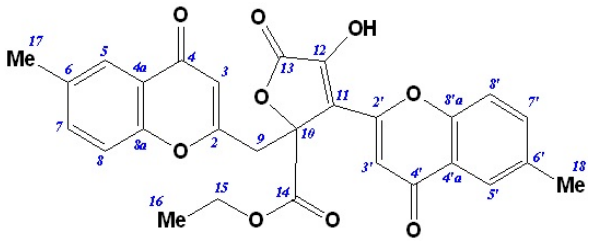

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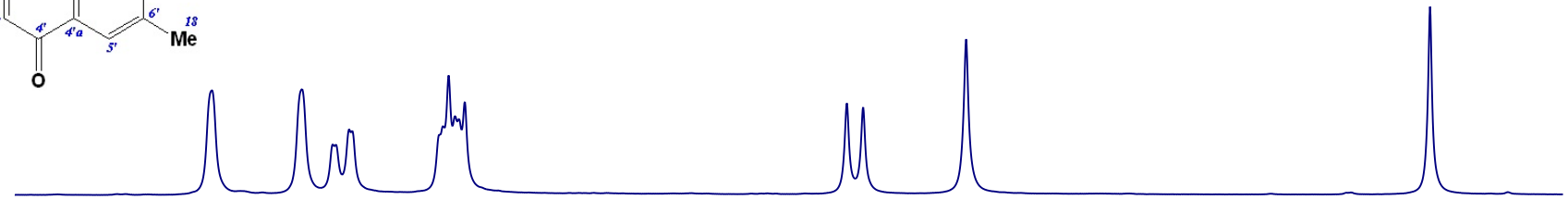

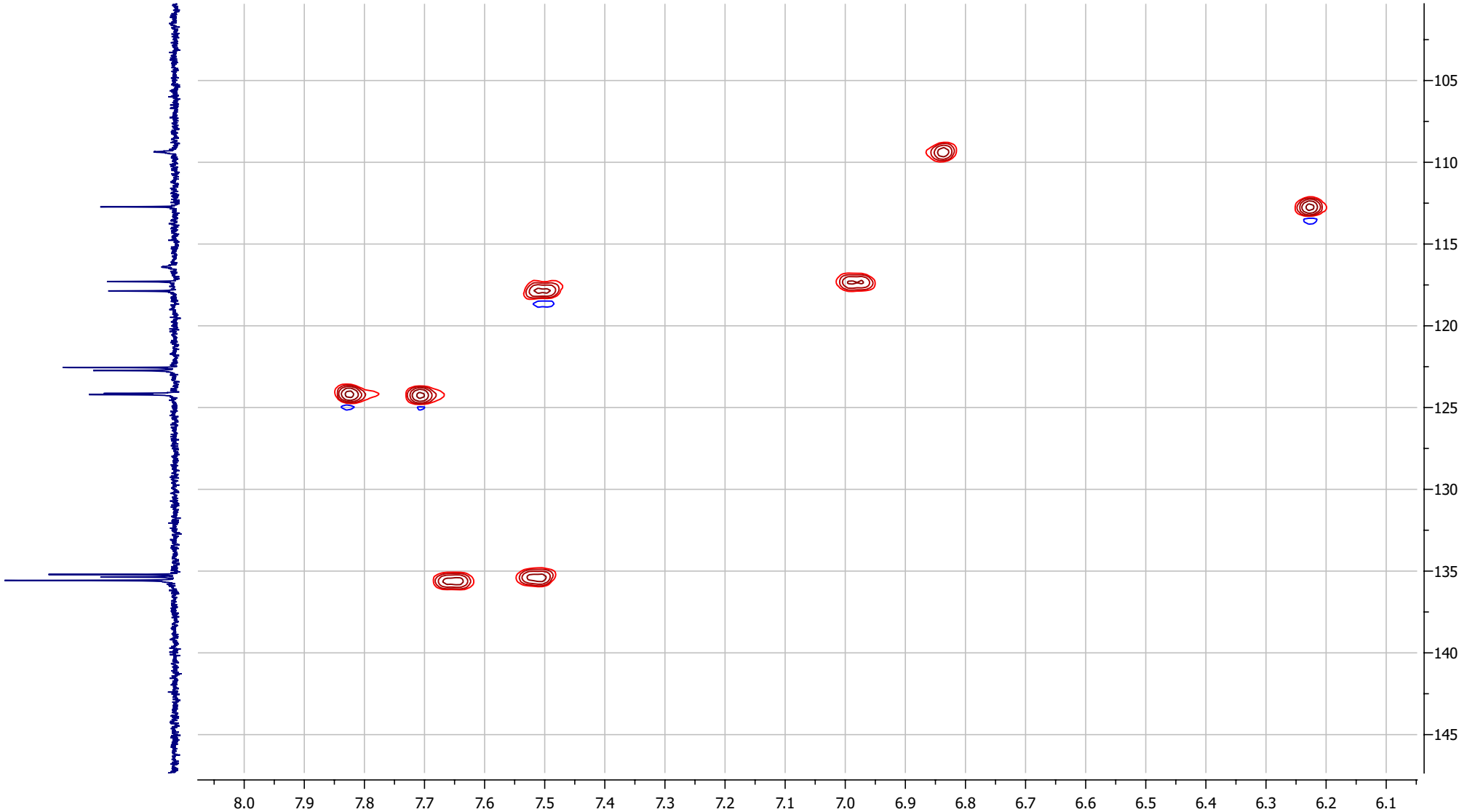

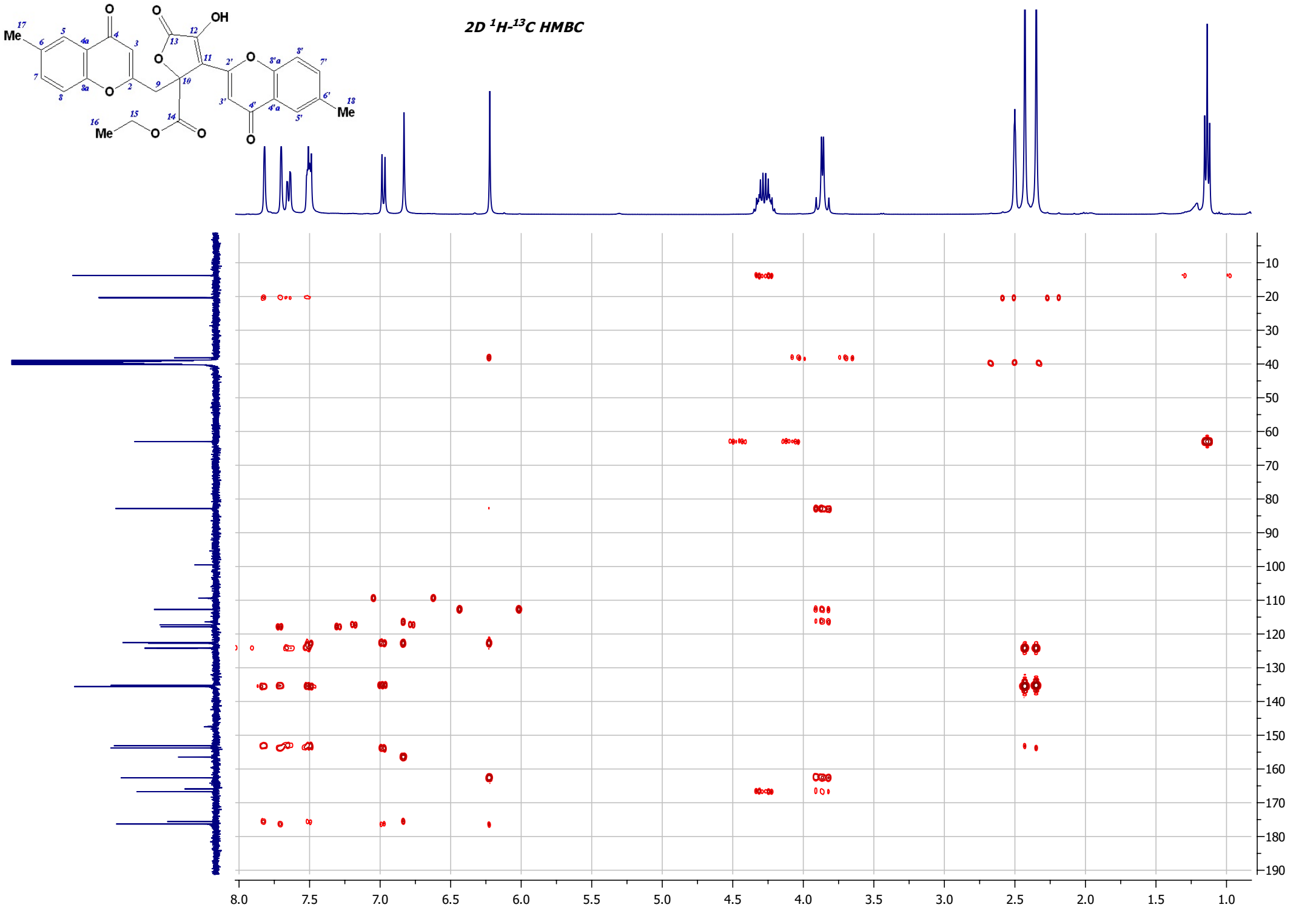
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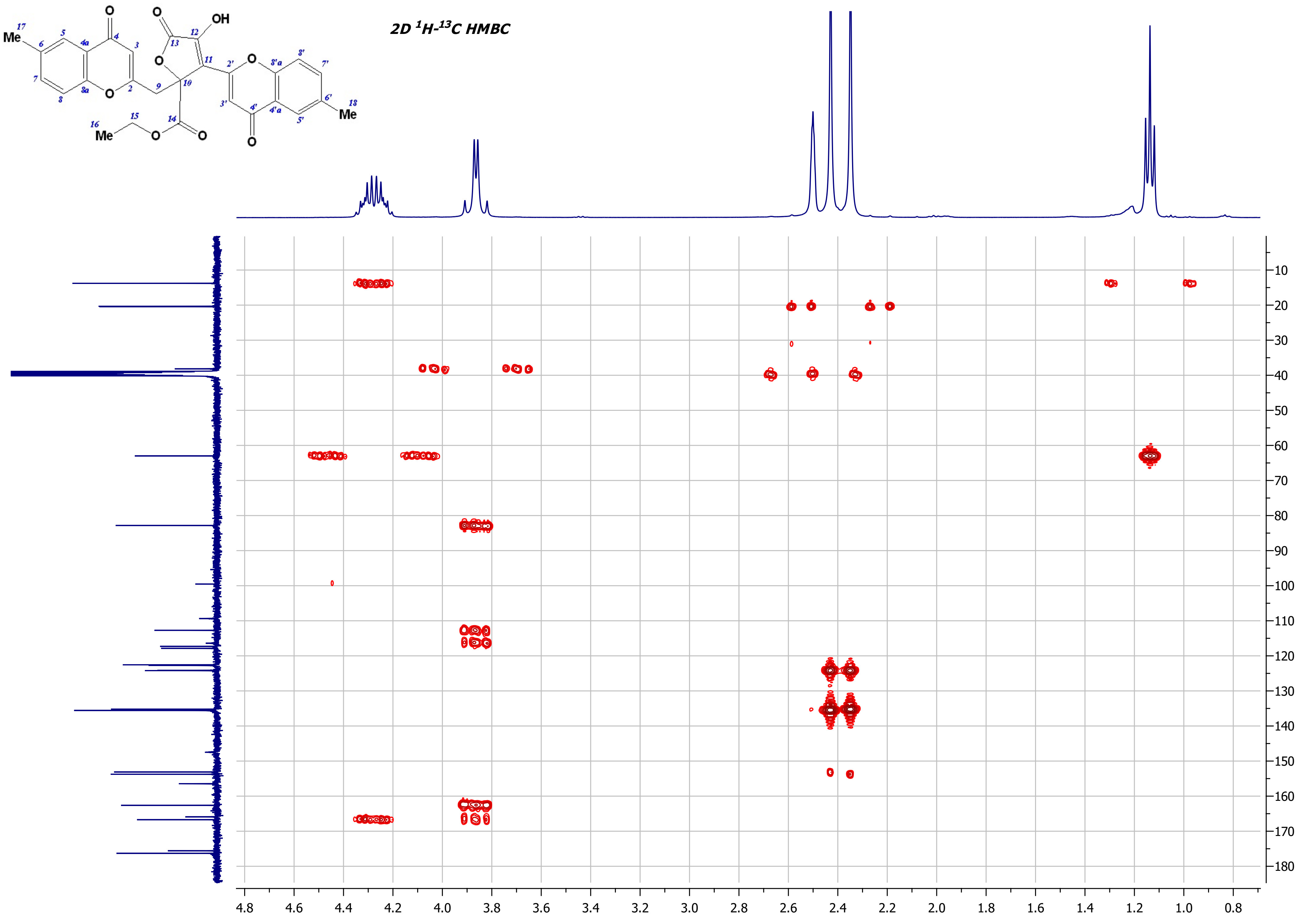

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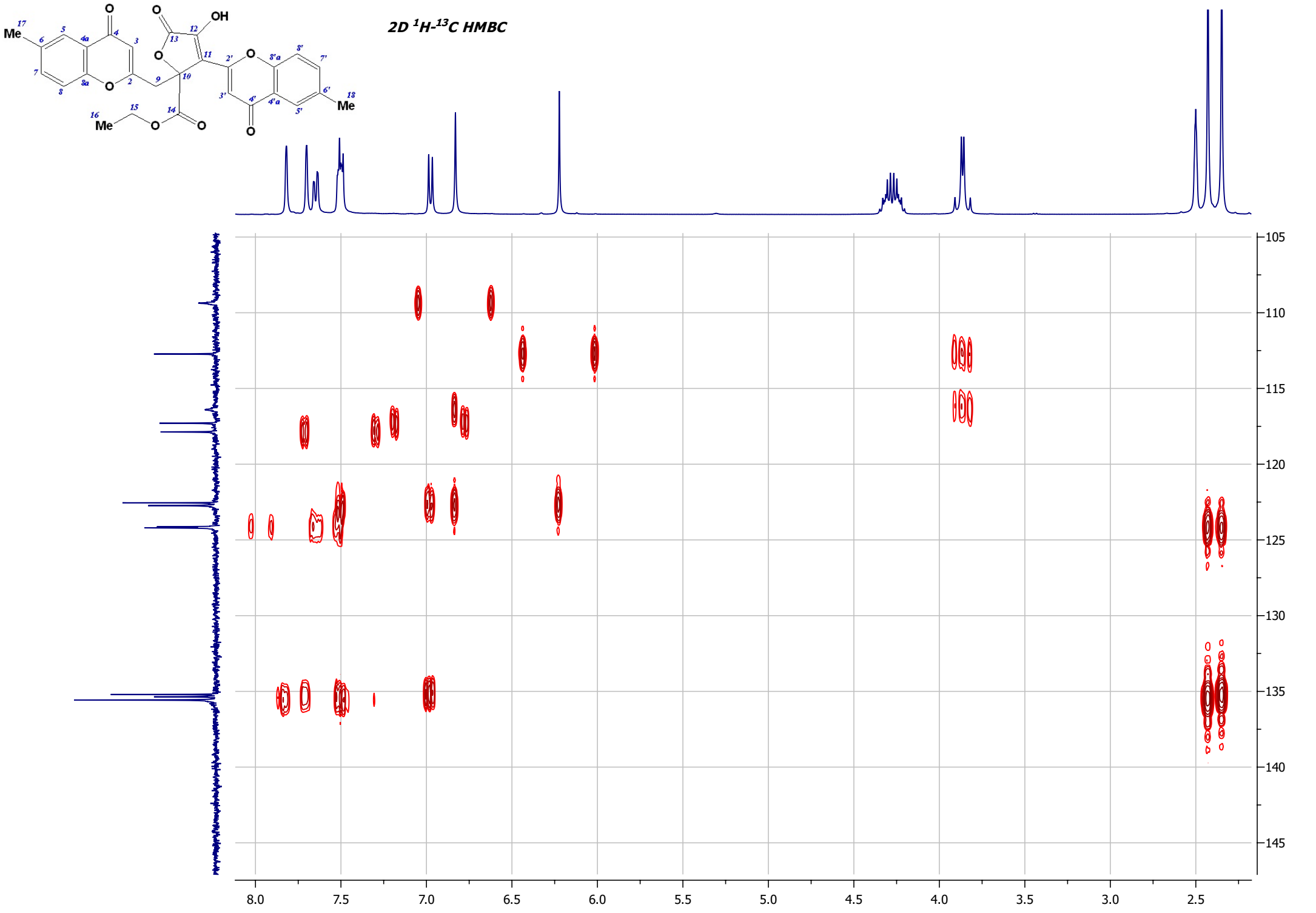
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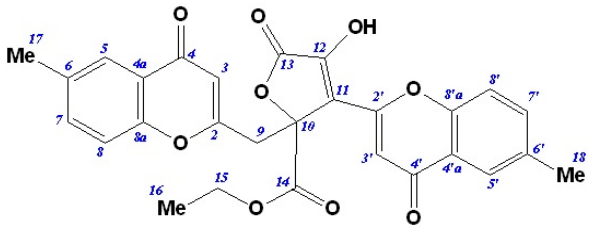
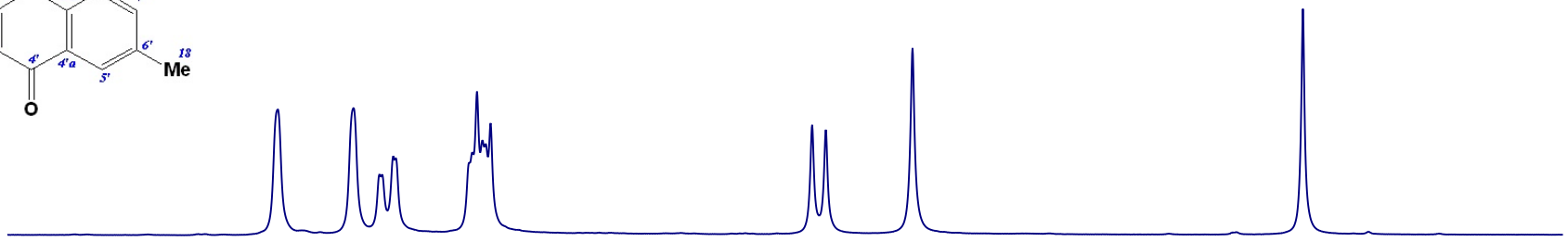


2D ^{1}H-^{13}C HMBC



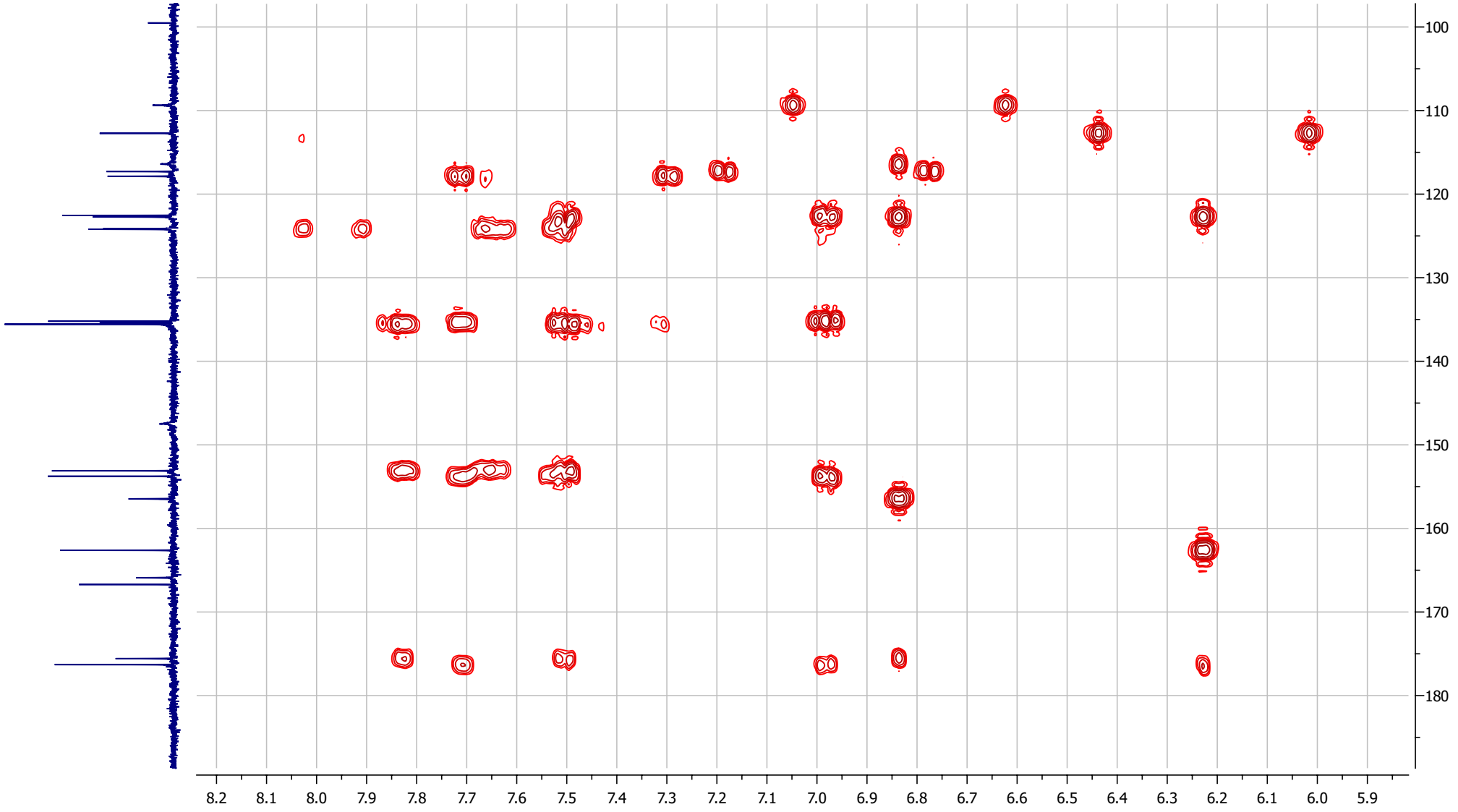


Table S1 Crystal data and structure refinement

Empirical formula	$C_{28}H_{22}O_{11}$
Formula weight	534.46
Temperature/K	295(2)
Crystal system	orthorhombic
Space group	Pbca
a/Å	24.5265(18)
b/Å	9.0278(7)
c/Å	25.1989(14)
α/°	90.00
β/°	90.00
γ/°	90.00
Volume/Å^3	5579.5(7)
Z	8
ρ_{calc}mg/mm^3	1.272
m/mm^{-1}	0.099
F(000)	2224.0
Crystal size/mm^3	0.35 × 0.15 × 0.03
2Θ range for data collection	5.6 to 56.6°
Index ranges	$-32 \le h \le 28$, $-12 \le k \le 12$, $-31 \le l \le 24$
Reflections collected	19685
Independent reflections	6334[R(int) = 0.0647]
Data/restraints/parameters	6334/24/375
Goodness-of-fit on F^2	1.005
Final R indexes [I>=2σ (I)]	R_1 = 0.0569, wR_2 = 0.1095
Final R indexes [all data]	R_1 = 0.1763, wR_2 = 0.1180
Largest diff. peak/hole / e Å^{-3}	0.24/-0.22

Table S2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) . U_{eq} is defined as 1/3 of the trace of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
O1	4172.2(8)	2028(2)	1188.8(6)	39.5(6)
C1	7808.6(18)	1059(5)	1385.9(17)	97.5(14)
O2	4566.1(10)	1641(2)	-372.7(8)	61.8(7)
C2	4412.8(13)	3154(3)	937.8(10)	37.1(8)
O6	7341.6(13)	271(3)	1246.7(11)	87.5(9)
O3	3731.3(11)	-3255(2)	232.4(9)	73.8(8)
C3	4550.0(13)	3053(3)	410.7(10)	43.2(9)
O4	6548.6(11)	4443(3)	2425.5(9)	73.9(8)
C4	4451.0(14)	1778(3)	118.6(11)	45.5(9)
O5	5303.7(10)	2061(2)	1810.3(7)	48.2(6)
C5	4095.7(14)	-795(3)	147.7(11)	46.9(9)

C6	3855.5(14)	-1900(3)	426.1(13)	50.1(9)
O7	4574.9(9)	6003(2)	1976.4(7)	46.8(6)
C7	3719.2(15)	-1681(3)	962.3(13)	58.0(11)
O8	4991.9(10)	5985(2)	648.3(7)	57.8(7)
C8	3828.1(13)	-380(3)	1210.2(12)	49.2(9)
C9	4073.5(13)	733(3)	924.9(11)	39.0(8)
O9	5022.2(11)	7842(2)	1578.4(7)	66.6(8)
C10	4202.9(13)	550(3)	392.6(11)	39.3(8)
C11	4812.1(14)	6661(4)	1556.5(11)	44.9(9)
C12	4776.1(13)	5641(3)	1098.8(11)	39.2(8)
C13	4513.0(12)	4414(3)	1257.5(9)	31.2(8)
C14	4366.7(14)	4572(3)	1834.8(10)	34.7(8)
C15	3754.8(17)	4679(4)	1904.5(15)	50.9(10)
C16	2960(2)	3606(6)	2247(2)	124(2)
C17	2715(2)	2582(7)	1945(3)	176(3)
C18	4620.2(14)	3470(3)	2215.1(10)	43.6(9)
C19	5212.2(16)	3253(3)	2124.9(11)	40.7(9)
C20	5619.6(17)	4062(3)	2321.9(11)	48.8(9)
C21	6174.8(18)	3731(3)	2225.2(13)	50.5(10)
C22	6257.6(16)	2477(3)	1870.2(12)	43.7(8)
C23	6780.2(17)	2017(4)	1730.1(12)	54.3(10)
C24	6855.1(19)	814(4)	1409.8(13)	60.6(10)
C25	6400.2(19)	35(4)	1237.5(14)	62.8(11)
C26	5893.8(17)	458(3)	1369.5(12)	53(1)
C27	5830.0(17)	1675(3)	1687.5(11)	44.2(9)
C28	3870.4(18)	-3540(4)	-307.8(14)	87.8(14)
O10	3552(4)	5923(8)	1908(3)	72(2)
O11	3538(6)	3511(14)	2040(4)	73(3)
O10A	3464(4)	5342(10)	1610(3)	92(3)
O11A	3552(6)	3764(14)	2259(3)	69(3)

Table S3 Anisotropic Displacement Parameters ($Å^2×10^3$) . The Anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U_{11}+...+2hka×b×U_{12}]$

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O1	50.2(16)	41.8(12)	26.5(11)	3.1(10)	0.3(10)	-15.1(11)
C1	70(4)	112(4)	110(3)	-18(3)	10(3)	11(3)
O2	99(2)	58.4(15)	27.8(12)	-10.3(11)	16.2(13)	-38.8(14)
C2	42(2)	42.0(18)	27.4(18)	6.9(15)	-1.5(16)	-11.5(16)
O6	65(2)	92(2)	106(2)	-31.8(16)	-1.1(19)	12.5(18)
O3	109(2)	43.3(14)	69.0(16)	-1.8(12)	-7.2(16)	-21.1(14)
C3	61(3)	40.3(18)	28.1(17)	-1.0(14)	-0.8(17)	-21.6(17)
O4	66(2)	78.5(18)	76.8(17)	-29.9(14)	-7.5(15)	-18.8(15)
C4	59(3)	47.5(19)	29.5(18)	2.4(16)	0.4(17)	-19.0(18)
O5	62(2)	46.6(13)	35.9(12)	-5.1(10)	-5.8(12)	-6.8(12)
C5	62(3)	37.7(18)	40.9(18)	-2.5(15)	-0.5(18)	-10.7(17)
C6	60(3)	34.4(18)	56(2)	-1.5(17)	-12(2)	-12.6(18)

O7	70.5(18)	45.7(12)	24.1(10)	-1.3(9)	11.3(11)	-11.1(12)
C7	75(3)	43(2)	56(2)	17.1(18)	-9(2)	-16.3(19)
O8	92(2)	54.7(14)	26.2(11)	-5(1)	16.6(12)	-30.4(13)
C8	61(3)	52(2)	34.1(18)	10.5(17)	-5.9(18)	-18.2(18)
C9	43(2)	40.7(19)	33.7(19)	4.5(15)	-4.8(17)	-9.3(16)
O9	105(2)	57.0(15)	37.8(12)	-11.4(12)	10.3(13)	-35.4(15)
C10	44(2)	39.2(18)	34.8(19)	1.7(15)	-2.9(16)	-5.8(16)
C11	58(3)	45.6(19)	31.3(18)	-1.4(17)	4.2(18)	-12.6(18)
C12	49(3)	47.7(19)	20.7(16)	-2.8(15)	0.7(17)	-9.3(16)
C13	40(2)	33.6(16)	20.4(15)	1.0(13)	0.9(15)	-9.1(15)
C14	41(2)	38.2(17)	25.5(17)	1.3(14)	4.3(16)	-9.2(16)
C15	60(3)	53(3)	40(2)	11(2)	10(2)	5(2)
C16	55(4)	137(5)	180(6)	42(4)	16(4)	-31(4)
C17	44(4)	216(7)	268(8)	-64(6)	19(5)	-32(4)
C18	57(3)	52.7(19)	21.6(15)	5.2(14)	0.7(17)	-10.1(18)
C19	57(3)	41.7(19)	23.0(16)	5.3(15)	-1.1(18)	-1.8(19)
C20	68(3)	41(2)	36.8(19)	-5.7(15)	-7(2)	-3(2)
C21	58(3)	47(2)	47(2)	-7.6(17)	-4(2)	-9(2)
C22	46(3)	46.1(19)	38.8(19)	-2.5(16)	-4(2)	-6.2(19)
C23	55(3)	59(2)	49(2)	-5.6(18)	-15(2)	-6(2)
C24	62(3)	61(2)	59(2)	-7(2)	2(2)	10(2)
C25	75(4)	54(2)	60(2)	-17.0(18)	-9(3)	2(2)
C26	61(3)	51(2)	46(2)	-11.7(17)	-5(2)	-9(2)
C27	50(3)	45(2)	36.9(19)	-0.7(15)	-2(2)	-2(2)
C28	125(4)	60(2)	78(3)	-24(2)	5(3)	-20(2)
O10	73(5)	70(5)	74(5)	18(3)	21(4)	11(4)
O11	42(5)	70(4)	108(7)	-13(5)	26(6)	-20(3)
O10A	64(5)	113(6)	99(6)	56(5)	14(5)	14(5)
O11A	68(6)	84(6)	56(5)	25(4)	11(5)	-14(5)

Table S4 Bond Lengths.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
O1	C2	1.334(3)	O9	C11	1.186(3)
O1	C9	1.367(3)	C11	C12	1.479(4)
C1	O6	1.393(4)	C12	C13	1.343(4)
O2	C4	1.276(3)	C13	C14	1.505(3)
C2	C3	1.373(3)	C14	C15	1.514(5)
C2	C13	1.415(3)	C14	C18	1.515(4)
O6	C24	1.354(4)	C15	O10	1.229(8)
O3	C6	1.352(3)	C15	O11	1.230(13)
O3	C28	1.427(4)	C15	O10A	1.191(8)
C3	C4	1.388(4)	C15	O11A	1.314(12)
O4	C21	1.228(4)	C16	C17	1.340(6)
C4	C10	1.441(4)	C16	O11	1.512(14)
O5	C19	1.355(3)	C16	O11A	1.458(15)
O5	C27	1.372(4)	C18	C19	1.483(4)
C5	C6	1.355(4)	C19	C20	1.334(4)

C5	C10	1.387(4)	C20	C21	1.415(4)
C6	C7	1.406(4)	C21	C22	1.457(4)
O7	C11	1.346(3)	C22	C23	1.393(4)
O7	C14	1.434(3)	C22	C27	1.355(4)
C7	C8	1.357(4)	C23	C24	1.366(4)
O8	C12	1.290(3)	C24	C25	1.389(5)
C8	C9	1.374(4)	C25	C26	1.341(5)
C9	C10	1.388(4)	C26	C27	1.369(4)

Table S5 Bond Angles .

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
C2	O1	C9	119.9(2)	C13	C14	C18	116.8(3)
O1	C2	C3	121.1(2)	C15	C14	C18	112.0(3)
O1	C2	C13	114.8(2)	O10	C15	C14	117.5(5)
C3	C2	C13	124.1(3)	O10	C15	O11	127.3(8)
C24	O6	C1	117.6(3)	O10	C15	O11A	114.6(7)
C6	O3	C28	117.0(2)	O11	C15	C14	114.0(7)
C2	C3	C4	121.7(3)	O11	C15	O11A	26.9(6)
O2	C4	C3	123.8(3)	O10A	C15	C14	123.6(5)
O2	C4	C10	118.9(3)	O10A	C15	O10	45.7(4)
C3	C4	C10	117.3(3)	O10A	C15	O11	110.2(9)
C19	O5	C27	119.3(3)	O10A	C15	O11A	120.8(8)
C6	C5	C10	119.8(3)	O11A	C15	C14	114.5(7)
O3	C6	C5	125.3(3)	C17	C16	O11	100.7(6)
O3	C6	C7	114.9(3)	C17	C16	O11A	121.7(6)
C5	C6	C7	119.8(3)	O11A	C16	O11	23.1(5)
C11	O7	C14	110.8(2)	C19	C18	C14	113.1(2)
C8	C7	C6	121.2(3)	O5	C19	C18	110.9(3)
C7	C8	C9	118.6(3)	C20	C19	O5	121.9(3)
O1	C9	C8	116.6(3)	C20	C19	C18	127.2(3)
O1	C9	C10	122.1(2)	C19	C20	C21	122.8(3)
C8	C9	C10	121.2(3)	O4	C21	C20	122.5(3)
C5	C10	C4	122.7(3)	O4	C21	C22	123.7(4)
C5	C10	C9	119.4(3)	C20	C21	C22	113.8(3)
C9	C10	C4	117.9(3)	C23	C22	C21	121.0(3)
O7	C11	C12	108.2(3)	C27	C22	C21	121.1(4)
O9	C11	O7	123.2(3)	C27	C22	C23	117.8(3)
O9	C11	C12	128.5(3)	C24	C23	C22	120.7(4)
O8	C12	C11	120.8(3)	O6	C24	C23	125.9(4)
O8	C12	C13	131.1(3)	O6	C24	C25	115.5(4)
C13	C12	C11	108.1(2)	C23	C24	C25	118.6(4)
C2	C13	C14	125.8(2)	C26	C25	C24	121.5(4)
C12	C13	C2	125.2(2)	C25	C26	C27	118.7(4)
C12	C13	C14	108.9(2)	C22	C27	O5	121.0(3)
O7	C14	C13	103.9(2)	C22	C27	C26	122.6(4)
O7	C14	C15	105.5(3)	C26	C27	O5	116.3(3)
O7	C14	C18	106.8(2)	C15	O11	C16	116.9(10)

C13	C14	C15	110.8(3)	C15	O11A	C16	115.2(9)

Table S6 Torsion Angles.

A	B	C	D	Angle/°
O1	C2	C3	C4	-0.8(5)
O1	C2	C13	C12	176.2(3)
O1	C2	C13	C14	-0.8(4)
O1	C9	C10	C4	0.3(5)
O1	C9	C10	C5	179.6(3)
C1	O6	C24	C23	-4.6(5)
C1	O6	C24	C25	176.7(3)
O2	C4	C10	C5	1.7(5)
O2	C4	C10	C9	-179.0(3)
C2	O1	C9	C8	179.5(3)
C2	O1	C9	C10	-1.7(4)
C2	C3	C4	O2	179.2(3)
C2	C3	C4	C10	-0.7(5)
C2	C13	C14	O7	177.4(3)
C2	C13	C14	C15	-69.8(4)
C2	C13	C14	C18	60.1(4)
O6	C24	C25	C26	-179.8(3)
O3	C6	C7	C8	179.0(3)
C3	C2	C13	C12	-3.4(5)
C3	C2	C13	C14	179.6(3)
C3	C4	C10	C5	-178.4(3)
C3	C4	C10	C9	0.9(5)
O4	C21	C22	C23	-0.8(5)
O4	C21	C22	C27	175.8(3)
O5	C19	C20	C21	-0.9(4)
C5	C6	C7	C8	-0.6(5)
C6	C5	C10	C4	-179.2(3)
C6	C5	C10	C9	1.5(5)
C6	C7	C8	C9	0.4(5)
O7	C11	C12	O8	-177.5(3)
O7	C11	C12	C13	0.7(4)
O7	C14	C15	O10	20.0(6)
O7	C14	C15	O11	-148.3(6)
O7	C14	C15	O10A	73.2(7)
O7	C14	C15	O11A	-118.7(6)
O7	C14	C18	C19	-69.8(3)
C7	C8	C9	O1	179.5(3)
C7	C8	C9	C10	0.8(5)
O8	C12	C13	C2	0.1(6)
O8	C12	C13	C14	177.5(3)
C8	C9	C10	C4	178.9(3)
C8	C9	C10	C5	-1.7(5)
C9	O1	C2	C3	2.0(4)

C9	O1	C2	C13	-177.6(3)
O9	C11	C12	O8	0.3(6)
O9	C11	C12	C13	178.6(4)
C10	C5	C6	O3	-179.9(3)
C10	C5	C6	C7	-0.4(5)
C11	O7	C14	C13	0.5(3)
C11	O7	C14	C15	-116.1(3)
C11	O7	C14	C18	124.5(3)
C11	C12	C13	C2	-177.8(3)
C11	C12	C13	C14	-0.4(4)
C12	C13	C14	O7	0.0(3)
C12	C13	C14	C15	112.9(3)
C12	C13	C14	C18	-117.3(3)
C13	C2	C3	C4	178.8(3)
C13	C14	C15	O10	-91.9(6)
C13	C14	C15	O11	99.8(6)
C13	C14	C15	O10A	-38.7(8)
C13	C14	C15	O11A	129.4(6)
C13	C14	C18	C19	46.0(4)
C14	O7	C11	O9	-178.7(3)
C14	O7	C11	C12	-0.7(3)
C14	C15	O11	C16	165.4(6)
C14	C15	O11A	C16	-166.6(6)
C14	C18	C19	O5	-95.3(3)
C14	C18	C19	C20	85.8(4)
C15	C14	C18	C19	175.2(3)
C17	C16	O11	C15	128.4(8)
C17	C16	O11A	C15	89.6(10)
C18	C14	C15	O10	135.8(5)
C18	C14	C15	O11	-32.5(6)
C18	C14	C15	O10A	-171.1(7)
C18	C14	C15	O11A	-3.0(6)
C18	C19	C20	C21	177.8(3)
C19	O5	C27	C22	-0.9(4)
C19	O5	C27	C26	179.8(2)
C19	C20	C21	O4	-176.9(3)
C19	C20	C21	C22	3.0(4)
C20	C21	C22	C23	179.3(3)
C20	C21	C22	C27	-4.1(4)
C21	C22	C23	C24	178.0(3)
C21	C22	C27	O5	3.2(4)
C21	C22	C27	C26	-177.5(3)
C22	C23	C24	O6	179.7(3)
C22	C23	C24	C25	-1.6(5)
C23	C22	C27	O5	179.9(3)
C23	C22	C27	C26	-0.8(4)
C23	C24	C25	C26	1.4(5)
C24	C25	C26	C27	-0.9(5)

C25	C26	C27	O5	179.9(3)
C25	C26	C27	C22	0.6(5)
C27	O5	C19	C18	-179.3(2)
C27	O5	C19	C20	-0.3(4)
C27	C22	C23	C24	1.3(4)
C28	O3	C6	C5	0.6(5)
C28	O3	C6	C7	-179.0(3)
O10	C15	O11	C16	-1.5(13)
O10	C15	O11A	C16	53.4(10)
O11	C15	O11A	C16	-71(2)
O11	C16	O11A	C15	62.7(19)
O10A	C15	O11	C16	-50.6(10)
O10A	C15	O11A	C16	1.8(12)
O11A	C15	O11	C16	68(2)
O11A	C16	O11	C15	-75(2)

Table S7 Hydrogen Atom Coordinates (Å×10^4) and Isotropic Displacement Parameters (Å^2×10^3).

Atom	*x*	*y*	*z*	U(eq)
H1A	8124	561	1248	146
H1B	7834	1118	1765	146
H1C	7789	2040	1240	146
H3	4713	3860	245	52
H5	4188	-937	-207	56
H7	3551	-2441	1150	70
H8A	3739	-244	1566	59
H16A	2940	3355	2621	149
H16B	2805	4582	2191	149
H17A	2329	2600	2010	264
H17B	2856	1620	2031	264
H17C	2783	2792	1577	264
H18A	4564	3812	2576	52
H18B	4436	2525	2178	52
H20	5535	4878	2532	59
H23	7081	2534	1856	65
H25	6448	-799	1026	75
H26	5592	-66	1248	64
H28A	3784	-4550	-394	132
H28B	4253	-3375	-359	132
H28C	3667	-2889	-535	132
H8	5193(16)	7290(50)	477(14)	122(14)