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Synthesis of daidzein derivatives for targeted drug delivery

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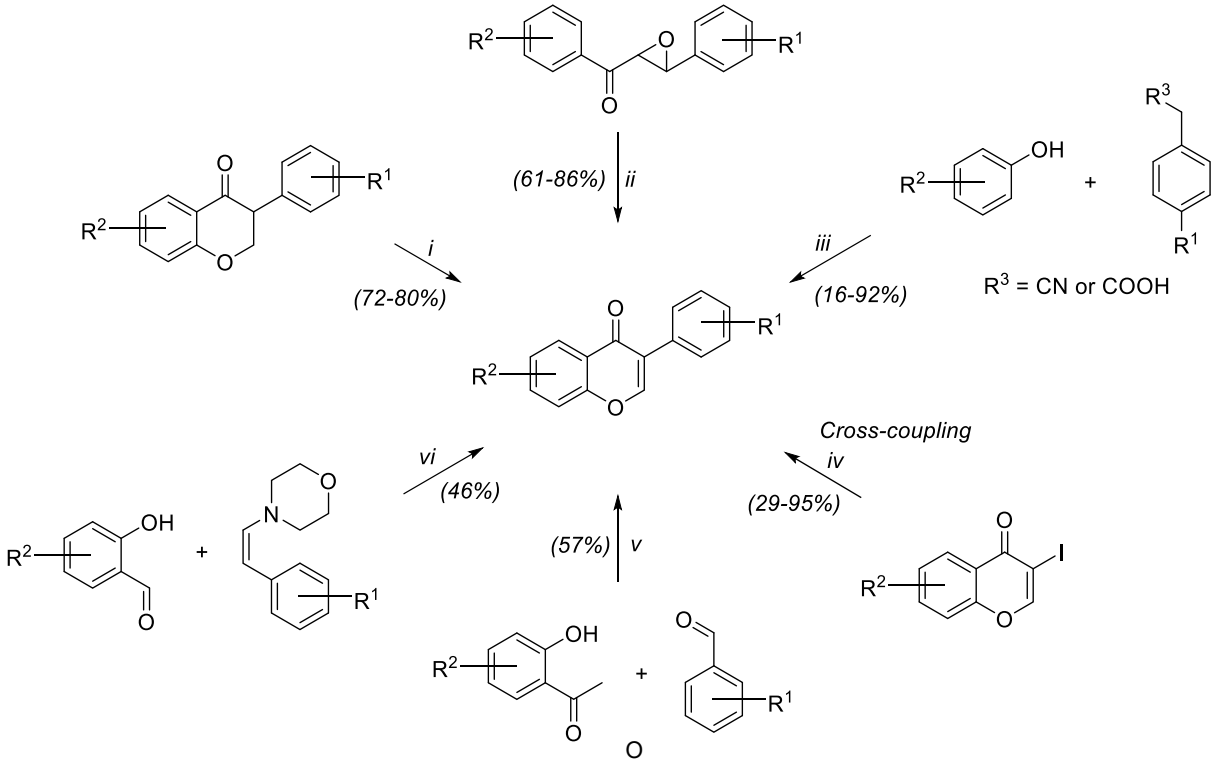
Scheme S1 Literature survey of approaches to the synthesis of isoflavonoids.



Reagents and conditions: i: $Tl(NO_3)_3$, MeOH, $CHCl_3$; ii: $BF_3{\cdot}Et_2O$, then HCl/AcOH; iii: R^3 = CN: HCl, $ZnCl_2$, H^+, then $POCl_3$, DMF, $BF_3{\cdot}Et_2O$, R^3 = COOH: $BF_3{\cdot}Et_2O$, then $POCl_3$, DMF; iv: $ArB(OH)_2$, $Pd(PPh_3)_4$, Na_2CO_3; v: 50% KOH, then $CH(OMe)_3$, $Tl(NO_3)_3$, then H^+; vi: benzene, reflux, CrO_3, Py.

Experimental section

General

2,4-Dihydroxyacetophenone, methyl bromoacetate, DMF-DMA, $Pd(OAc)_2$, PEG3350, 4-hydroxyphenylboronic acid, DIPEA, HBTU, trifluoroacetic acid were purchased in "Sigma-Aldrich" (Germany), abs. DMF, $KHCO_3$, P_2O_5, EtOAc, hexane, Na_2SO_4, I_2, abs. MeOH, Na_2SO_3, AcOH, CH_2Cl_2, HCl, *i*-PrOH, EtOH, NaOH were purchased in "Ruskhim" (Russia). All reagents and solvents were reagent grade and were used without further purification. Unless otherwise stated, the intermediate products were dried *in vacuo* over P_2O_5 and NaOH before use. The reactions completion was monitored by thin-layer chromatography using Merck Kieselgel-60 F_{254} pre-coated plates visualized under 254nm light. Melting points were determined using a Boethius instrument.

^1H, ^{13}C and ^1H–^{13}C HMBC NMR spectra were recorded on an AMX-300 instrument (operating frequency: 300.0 MHz for ^1H nuclei and 75.0 MHz for ^{13}C nuclei) in DMSO-d_6 at 27°C; The signal from the residual protons of the solvent was used as an internal standard. Chemical shifts (δ) were given in parts per million (ppm), the values of coupling constants (*J*) were measured in Hertz (Hz).

Mass spectra were registered using high-resolution mass spectrometer Bruker Daltonics micrOTOF-Q II with electrospray ionization (ESI HRMS). The measurements were carried out in positive and negative ion modes. Capillary voltage: 4500 V; mass scanning range m/z 50-3000; external calibration (Agilent Electrospray Calibrant Solution, USA); spray pressure: 0.4 bar; flow rate: 3 μl/min; nebulizer gas: nitrogen (6 ml/min); interface temperature: 180°C. Samples were injected into the spray chamber of the mass spectrometer after an Agilent 1260 high-performance liquid chromatograph system equipped with an Agilent Poroshell 120 EC-C18 column (3.0 × 50 mm; 2.7 μm) and a precolumn corresponding to its parameters; flow rate 0.2 ml/min; samples were injected *via* the autosampler into the HPLC chromatograph from a 1:1 acetonitrile-water solution (5 μl) and eluted in a concentration gradient of acetonitrile (0 → 70%) in water.

IR spectra were recorded on a Bruker ALPHA spectrometer (Bruker BioSpin GmbH) in a thin layer between KBr plates, in the region 4000–400 cm-1 (16 scans, resolution 2 cm-1).

Methyl 2-(4-acetyl-3-hydroxyphenoxy)acetate (2)

To a suspension of 2,4-dihydroxyacetophenone **1** (5.00 g, 0.033 mol) and $KHCO_3$ (5.00 g, 0.049 mol) in abs. DMF (50 ml), methyl bromoacetate (5.30 g, 3.28 ml, 0.035 mol) was added and this was heated at 80°C for 6 hours. Then approx. 35 ml of the solvent was evaporated on a rotary evaporator, and 100 ml of H_2O were added to the suspension. The formed precipitate was filtered

off, washed with water (2×50 ml) and dried *in vacuo* over P_2O_5. According to TLC data in the EtOAc:hexane (2:1) system, the product was homogeneous (Rf = 0.9). Compound **2** was obtained in 96% yield as a white powder (7.096 g). M.p. = 105-107°C. ^{1}H NMR (DMSO-d_6): 12.54 (s, 1H), 7.85 (d, J = 9.0 Hz, 1H), 6.55 (dd, J_1 = 9.0 Hz, J_2 = 2.5 Hz, 1H), 6.46 (d, J = 2.5 Hz, 1H), 4.91 (s, 2H), 3.71 (s, 3H), 2.57 (s, 3H). ^{13}C NMR (DMSO-d_6): 203.17, 168.56, 163.79, 163.74, 133.32, 114.32, 107.30, 107.30 101.57, 64.65, 51.89, 26.65. HRMS of $C_{11}H_{12}O_5$, *m/z*: calculated for $[M+H]^+$ 225.0757, found 225.0760.

Methyl 2-{4-[3-(dimethylamino)acryloyl]-3-hydroxyphenoxy}acetate (3)

To a solution of compound **2** (3.00 g, 0.013 mol) in abs. DMF (50 ml) heated to 70°C, dimethylformamide dimethyl acetal (1.645 g, 1.834 ml, 0.013 mol) was added dropwise over 30 minutes and stirred for 24 hours. 150 ml of H_2O were added to the reaction mixture and the product was extracted with EtOAc (3x50 ml). The combined organic phase was washed with H_2O (2×150 ml), dried over Na_2SO_4 and evaporated. Residue was crystallized from EtOAc:hexane. According to TLC data in the EtOAc – hexane (2:1) system, the product is homogeneous (R_f = 0.4). Compound **3** was obtained in 87% yield as a bright yellow powder (3.143 g). M.p. = 140-143°C. ^{1}H NMR (DMSO-d_6): 14.95 (s, 1H) 7.84 (d, J = 11.9 Hz, 1H), 7.83 (d, J = 2.6 Hz, 1H), 6.41 (dd, J = 12.0 Hz, J = 2.6 Hz, 1H), 6.31 (d, J = 2.6 Hz, 1H), 5.85 (d, J = 12 Hz, 1H), 4.83 (s, 2H), 3.71 (s, 3H), 3.18 (s, 3H), 2.97 (s, 3H), ^{13}C NMR (DMSO-d_6): 189.20, 168.84, 164.78, 161.87, 154.90, 130.29, 114.06, 105.94, 101.62, 89.00, 64.52, 51.83, 44.82, 37.30. HRMS of $C_{14}H_{17}NO_5$, *m/z*: calculated for $[M+H]^+$ 280.1179, found 280.1177.

Methyl 2-[(3-iodo-4-oxo-4*H*-chromen-7-yl)oxy]acetate (4)

A solution of compound **3** (3.000 g, 0.011 mol) and iodine (6.479 g, 0.026 mol) in methanol (40 ml) was stirred for 24 hours in the dark, then cooled to -20°C. The precipitate was filtered off, washed with cooled to -20°C methanol (15 ml), 2% Na_2SO_3 solution (15 ml), H_2O (15 ml) and dried over P_2O_5. According to TLC data in the EtOAc/cyclohexane (4:7) system, the product is homogeneous (Rf = 0.64). Compound **4** was obtained in 77% yield as a pale yellow solid (3.050 g). M.p. = 143-145°C. ^{1}H NMR (DMSO-d_6): 8.77 (s, 1H), 7.97 (d, J = 8.9 Hz, 1H), 7.19 (d, J = 2.5 Hz, 1H), 7.14 (dd, J_1 = 8.9 Hz, J_2 = 2.5 Hz, 1H), 4.99 (s, 2H), 3.72 (s, 3H). ^{13}C NMR (DMSO-d_6): 171.96, 168.43, 162.14, 158.71, 157.26, 127.19, 115.46, 101.64, 86.87, 65.05, 51.97. HRMS of $C_{12}H_9IO_5$, *m/z*: calculated for $[M+H]^+$ 360.9567, found 360.9566.

Methyl 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetate (5)

A mixture of Na_2CO_3 (2.230 g, 21 mmol), $Pd(OAc)_2$ (0.180 g, 0.8 mmol) and PEG 3350 (2.070 g) in abs. MeOH (60 ml) was heated in a water bath at 50°C until a black color appeared, then iodide **4** (3.027 g, 8.4 mmol) and 4-hydroxyphenylboronic acid (1.800 g, 13 mmol) were added and stirred for 3 hours. Water (100 ml) was added to the reaction mixture, the precipitate was filtered off, washed with H_2O (3x30 ml). The product was then isolated by boiling EtOAc in a Soxhlet extractor (50 ml) for 12 hours. The organic phase was evaporated and dried over P_2O_5. According to TLC data in the EtOAc:cyclohexane (4:7) system, the product is homogeneous (R_f = 0.5). Compound **5** was obtained in 86% yield as a white powder (2.36 g). M.p. = 205-208°C. ^{1}H NMR (DMSO-d_6): 9.52 (s, 1H), 8.37 (s, 1H), 8.04 (d, J = 8.9 Hz, 1H), 7.40 (d, J = 6.8 Hz, 2H), 7.18 (d, J = 2.4 Hz, 1H), 7.12 (dd, J = 8.9 Hz, J = 2.4 Hz, 1H), 6.82 (d, J = 6.8 Hz, 2H), 5.00 (s, 2H), 3.73 (s, 3H). ^{13}C NMR (DMSO-d_6): 174.68, 168.56, 161.84, 157.27, 157.15, 153.20, 130.07, 127.08, 123.75, 122.31, 118.15, 114.99, 114.77, 101.58, 65.03, 51.97. HRMS of $C_{18}H_{14}O_6$, *m/z*: calculated for $[M+H]^+$ 327.0865, found 327.0863.

2-{[3-(4-Hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetic acid (6)

A suspension of compound **5** (2.000 g, 6.13 mmol) in 50% acetic acid (15 ml) was refluxed for 8 hours. The precipitate was filtered off, washed with cold 50% acetic acid (10 ml), H_2O (3x50 ml) and dried over P_2O_5. According to TLC data in the system CH_2Cl_2/CH_3OH/AcOH (16:2:1), the product is homogeneous (R_f=0.5). Compound **6** was obtained in 93% yield as an off-white solid (1.78 g). M.p. = 285-287°C. ^{1}H NMR (DMSO-d_6): 13.12 (br. s., 1H), 9.50 (br. s., 1H), 8.36 (s, 1H), 8.04 (d, J = 8.9 Hz, 1H), 7.40 (d, J = 6.8 Hz, 2H), 7.11 (m, 2H), 6.82 (d, J = 6.8 Hz, 2H), 4.88 (s, 2H). ^{13}C NMR (DMSO-d_6): 174.71, 169.53, 162.07, 157.28, 157.18, 153.16, 130.10, 127.06, 123.76, 122.38, 118.02, 115.02, 114.77, 101.50, 65.01. HRMS of $C_{28}H_{34}N_2O_7$, *m/z:* calculated for $[M+H]^+$ 311.0550, found 311.0548. IR spectrum (cm^{-1}): 3073 (broad, medium, ν COOH, OH), 1730 (strong, ν C=O), 1624 (medium, ν C=C), 1252 (medium, ν C-O-C).

***tert*-Butyl *N*-[6-(2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamido)-hexyl]carbamate (7)**

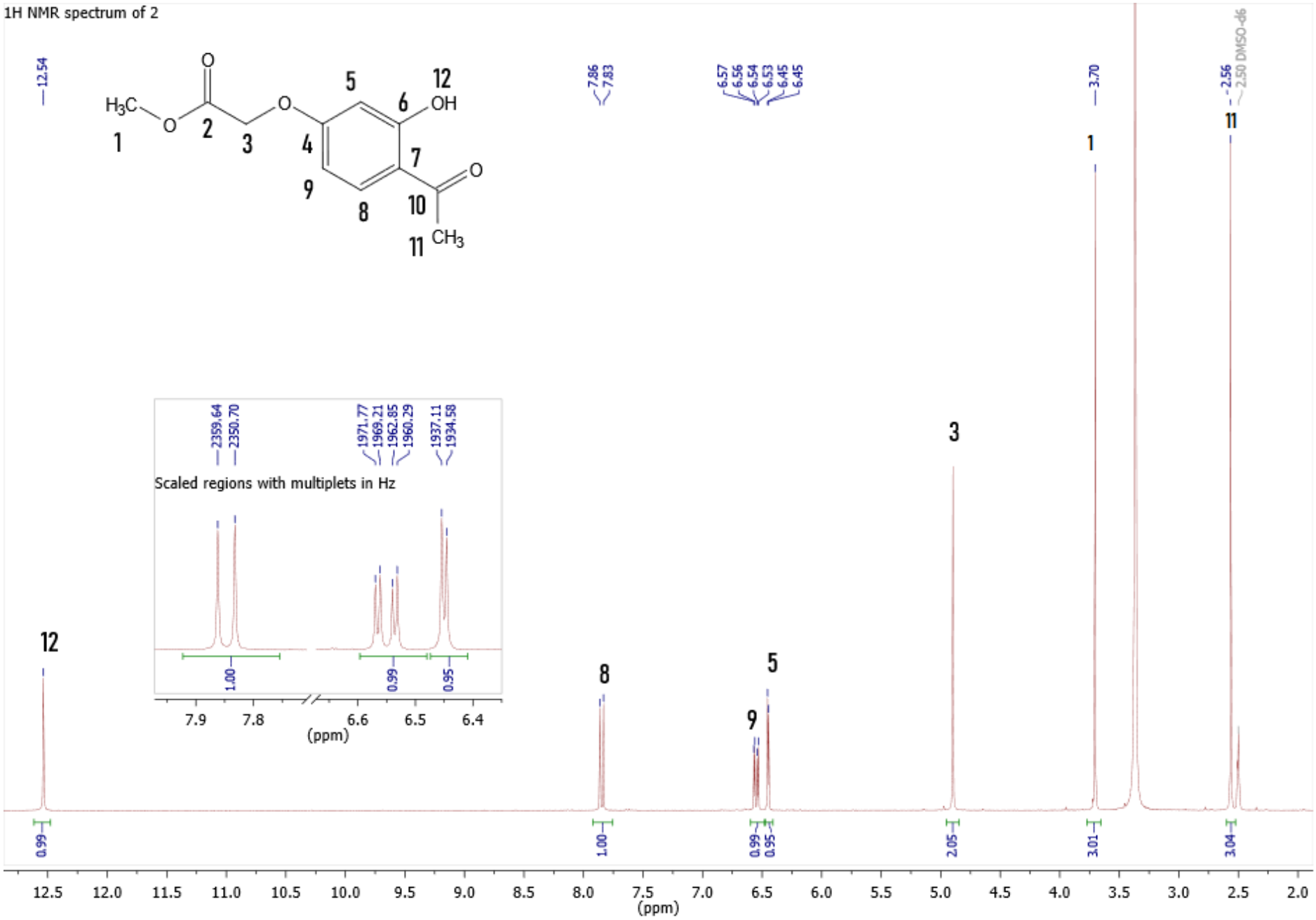
DIPEA (121 μl (0.091 g), 0.7 mmol) and *tert*-butyl *N*-(6-aminohexyl)carbamate (0.152 g, 0.7 mmol) were added to a cooled to 0°C solution of **6** (0.2 g, 0.64 mmol) in DMF (10 ml). Then, HBTU (0.267 g, 0.7 mmol) was added and stirred for 45 minutes. The resulting solution was evaporated, a 0.1% HCl solution (50 ml) was added to the residue, and extracted with EtOAc (3x50 ml). The combined organic phase was washed with 3% $KHCO_3$ solution (50 ml), H_2O (2x50 ml),

then dried over Na_2SO_4 and evaporated. According to TLC data in the $Pr^iOH/AcOH/H_2O$ (4:1:1) system, the product is homogeneous (R_f = 0.9). Compound **7** was obtained in 77% yield as a yellow oil (0.252 g). ^{1}H NMR (DMSO-*d*$_6$): 9,52 (s, 1H), 8.37 (s, 1H), 8.16 (t, *J* = 5.8 Hz, 1H), 8.06 (d, *J* = 8.8 Hz, 1H), 7.40 (d, *J* = 8.7 Hz, 2H), 7.14 (m, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 6.73 (t, *J* = 6.0 Hz 1H), 4.66 (s, 2H), 3.13 (q, *J* = 5.8 Hz, 2H), 2.89 (q, *J* = 6.0 Hz, 2H), 1.16-1.51 (m, 17H), ^{13}C NMR (DMSO-*d*$_6$): 174.65, 166.56, 161.98, 157.23, 155.54, 153.14, 131.15, 126.94, 123.74, 122.28, 118.00, 115.53, 114.95, 101.49, 77.26, 67.34, 39.40, 38.27, 29.42, 29.09, 29.01, 28.24, 26.02, 25.95. HRMS of $C_{11}H_{12}O_5$, *m/z:* calculated for $[M+NH_4]^+$ 528.2710 found 528.2707.

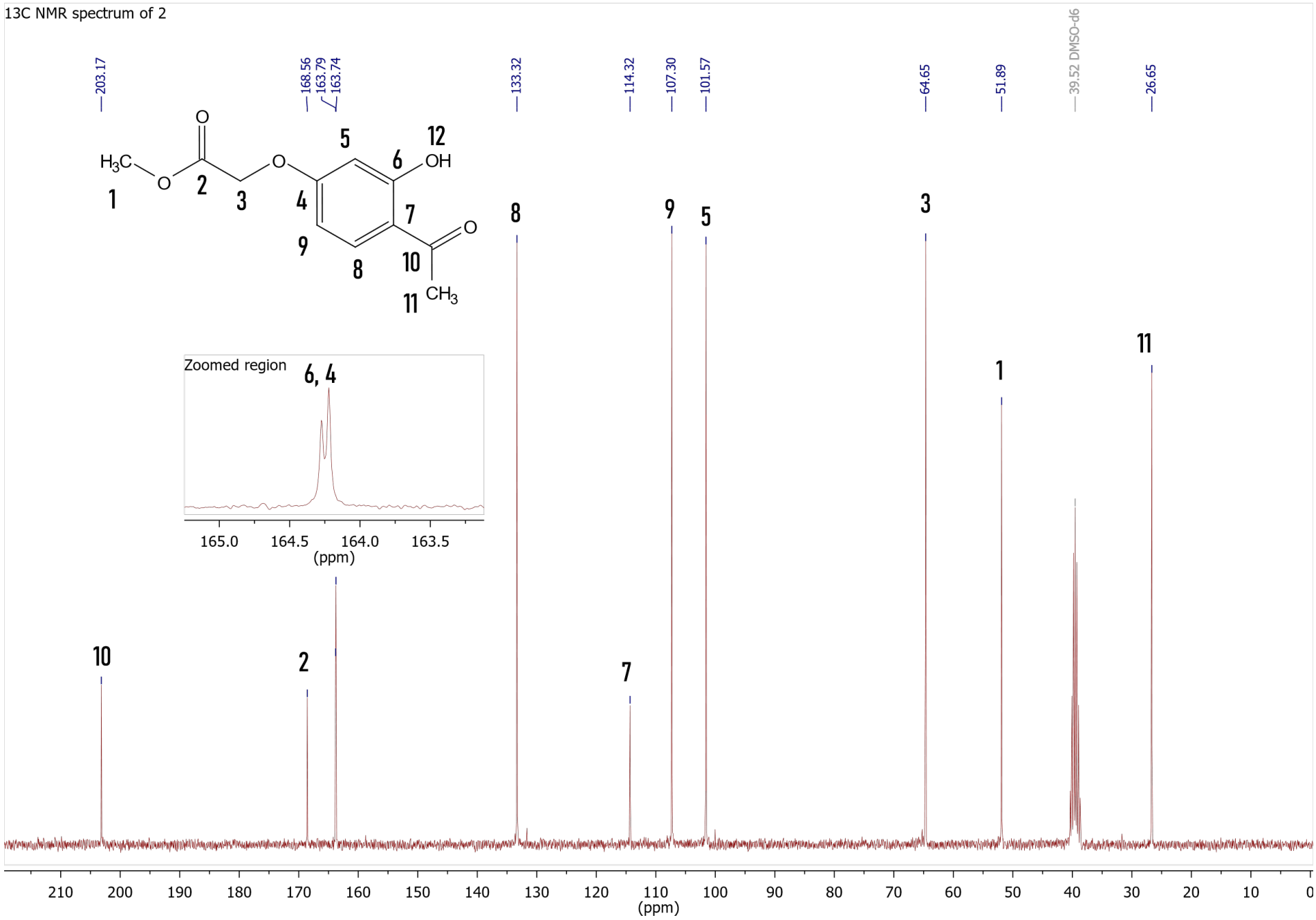
_N_-(6-Aminohexyl)-2-{[3-(4-hydroxyphenyl)-4-oxo-4H-chromen-7-yl]oxy}acetamide (8)

To a solution of compound **7** (0.200 g, 0.392 mmol) in CH_2Cl_2 (5 ml) upon cooling to 0°C CF_3COOH (5 ml) was slowly added dropwise, stirred for 30 minutes, evaporated and then re-evaporated with EtOH (2x5 ml). The residue was dried over P_2O_5/NaOH. According to TLC data in the Pr^iOH: $AcOH/H_2O$ (4:1:1) system, the product is homogeneous (R_f = 0.71). Compound **8** was obtained in 98% yield as a white powder (0.158 g). M.p. = 198-200°C. ^{1}H NMR (DMSO-*d*$_6$): 9.57 (br. s, 1H), 8.38 (s, 1H), 8.21 (t, *J* = 6.7 Hz, 1H), 8.06 (d, *J* = 9.0 Hz, 1H), 7.69 (m, 3H), 7.40 (d, *J* = 8.7 Hz, 2H), 7.14 (m, 2H), 6.82 (d, *J* = 8.7 Hz, 2H), 4.67 (s, 2H), 3.15 (q, *J* = 6.8 Hz, 2H), 2.77 (m, 2H), 1.59-1.20 (m, 8H). ^{13}C NMR (DMSO-*d*$_6$): 174.73, 166.69, 162.03, 157.35, 157.17, 153.20, 130.07, 127.01, 123.80, 122.28, 118.05, 115.03, 101.55, 67.35, 38.78, 38.25, 28.89, 26.95, 25.87, 25.50. HRMS of $C_{23}H_{26}N_2O_5$, *m/z:* calculated for $[M+H]^+$ 411.1916, found 411.1914. IR spectrum (cm^{-1}): 3417 (weak, ν NH), 3064 (broad, medium, ν COOH, OH), 2948 (broad, ν NH_2), 1670 (strong, ν C=O), 1628 (medium, ν C=C), 1190 (medium, ν C-O-C).

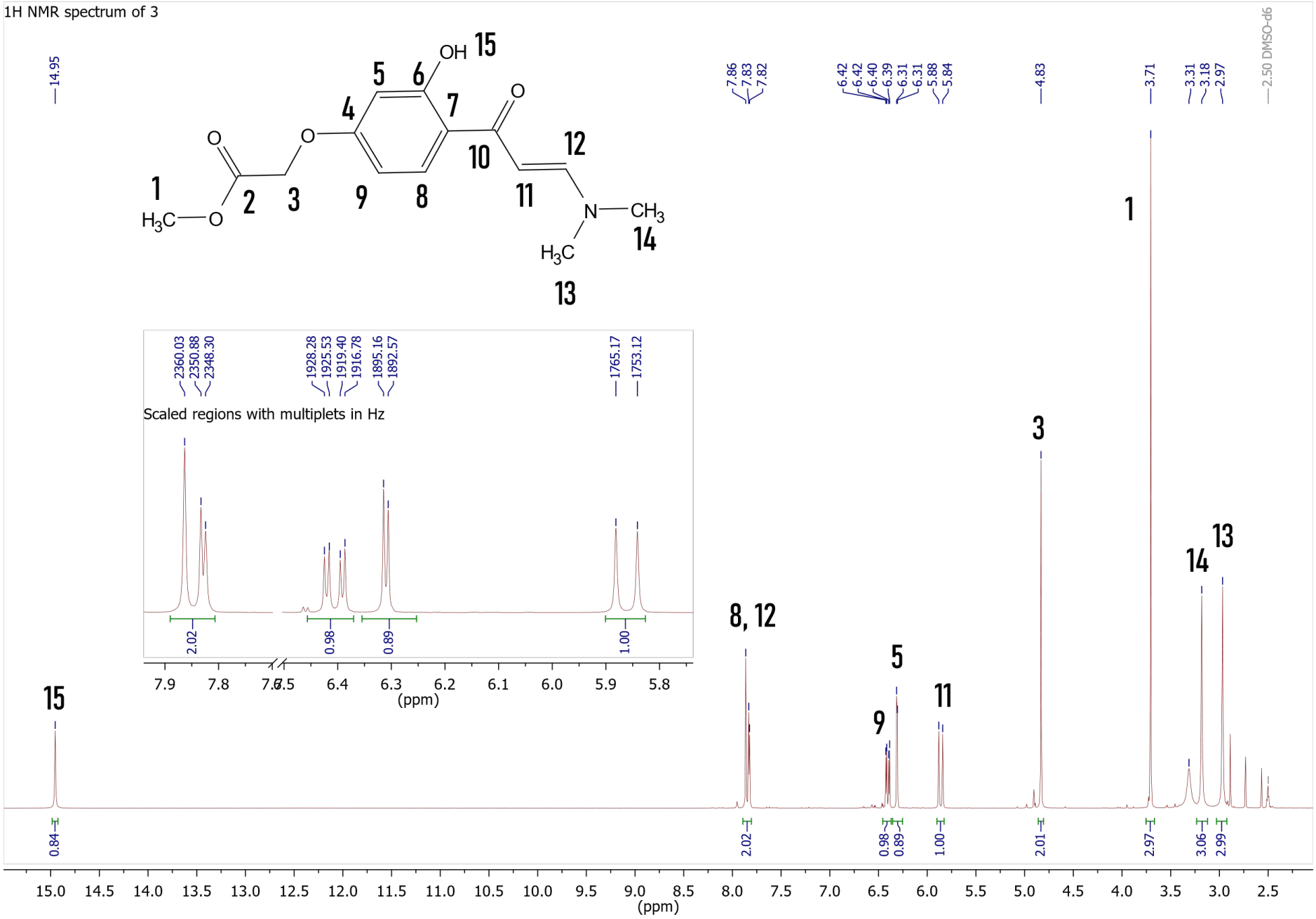
[1]H NMR spectrum of methyl 2-(4-acetyl-3-hydroxyphenoxy)acetate (**2**)



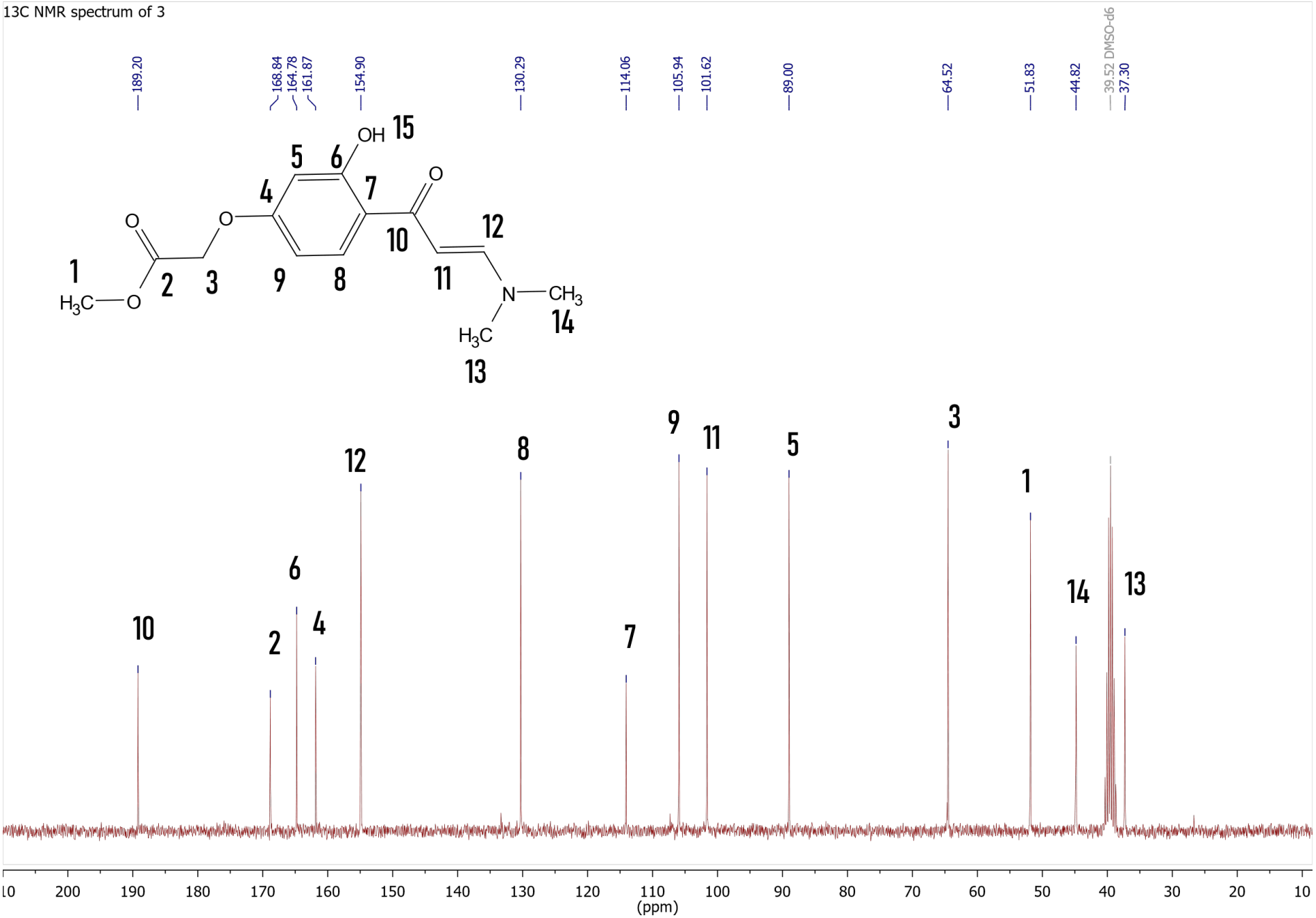
^{13}C NMR spectrum of methyl 2-(4-acetyl-3-hydroxyphenoxy)acetate (**2**)



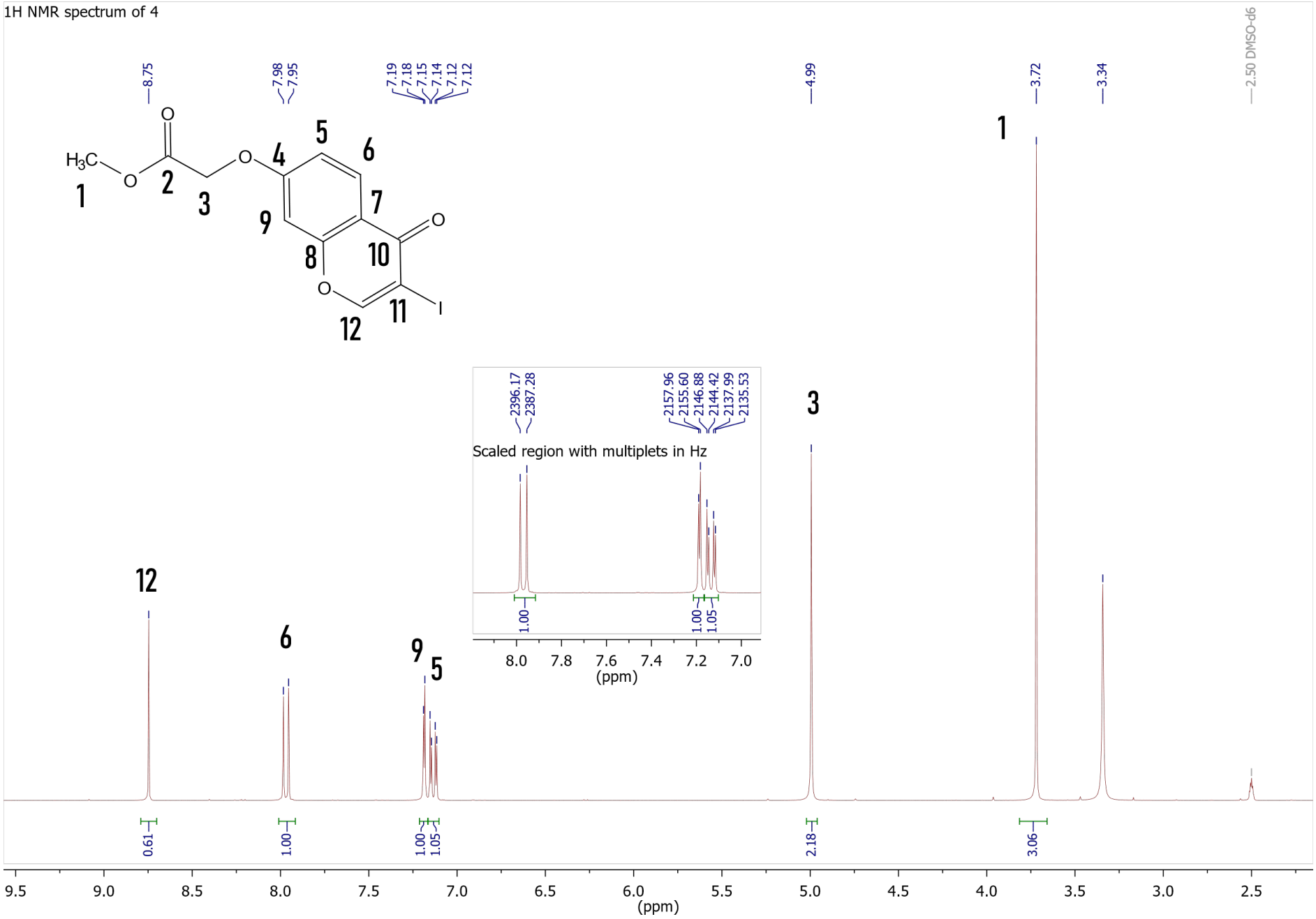
[1]H NMR spectrum of methyl 2-{4-[3-(dimethylamino)acryloyl]-3-hydroxyphenoxy}acetate (**3**)



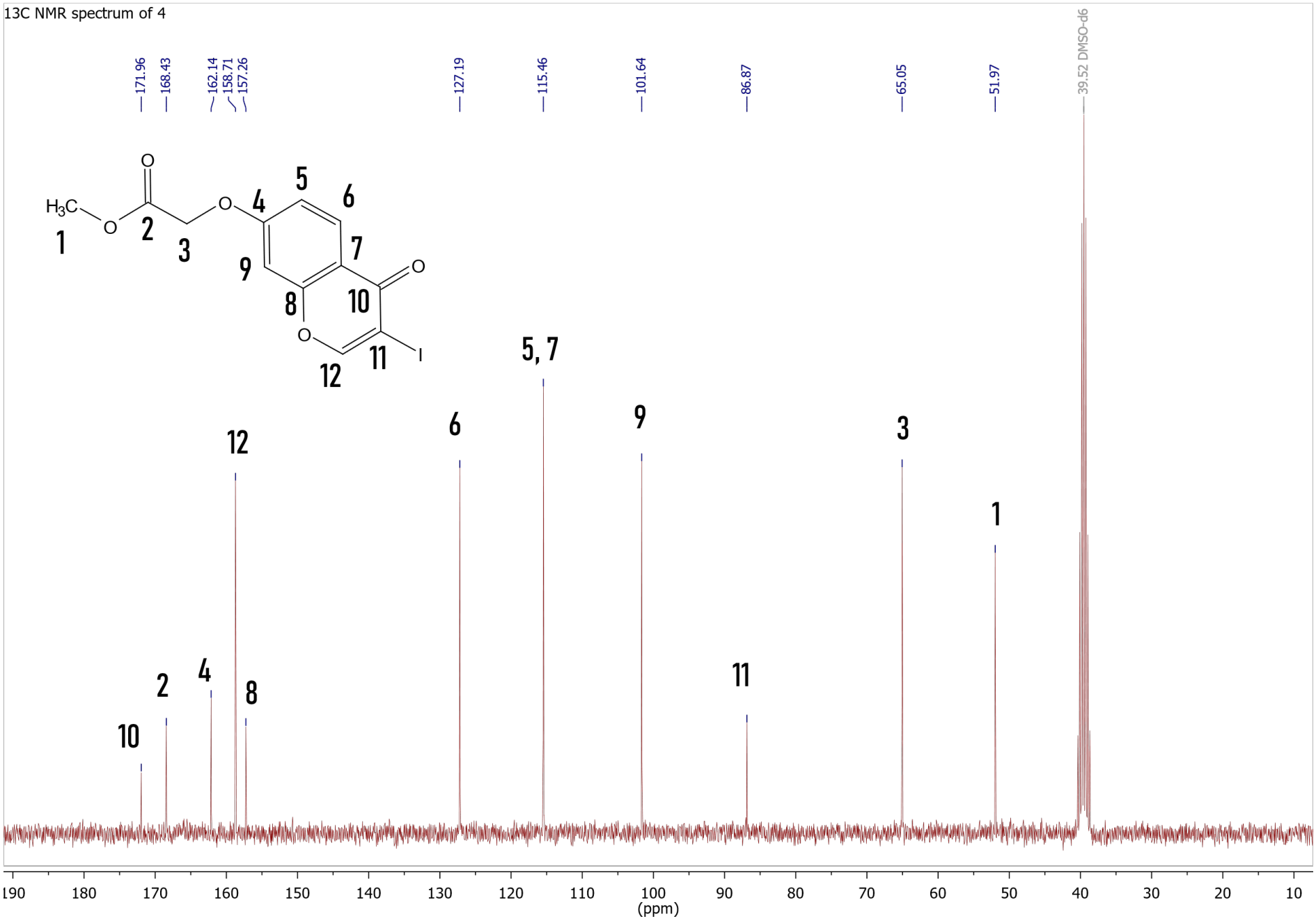
^{13}C NMR spectrum of methyl 2-{4-[3-(dimethylamino)acryloyl]-3-hydroxyphenoxy}acetate (**3**)



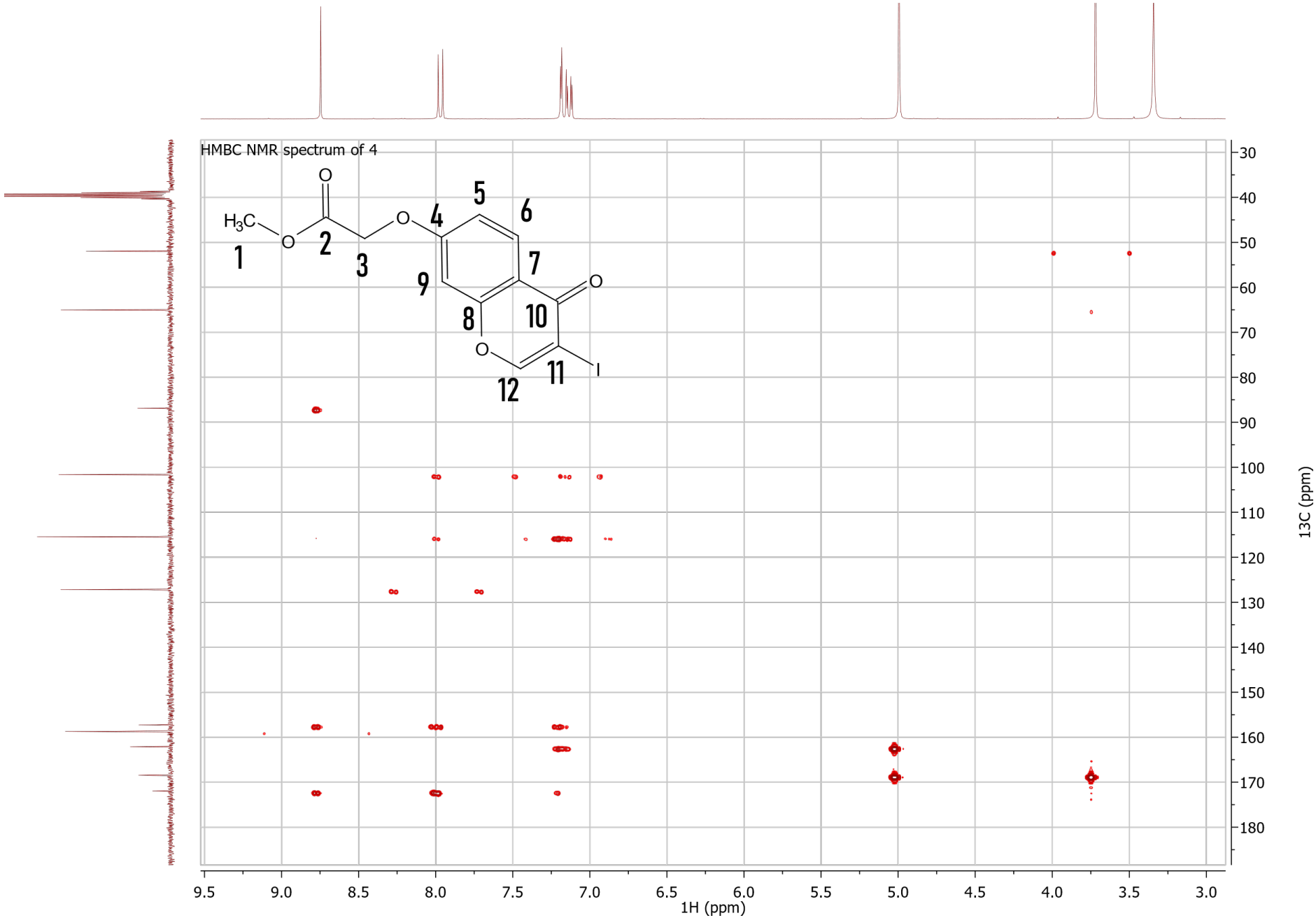
[1]H NMR spectrum of methyl 2-[(3-iodo-4-oxo-4*H*-chromen-7-yl)oxy]acetate (**4**)



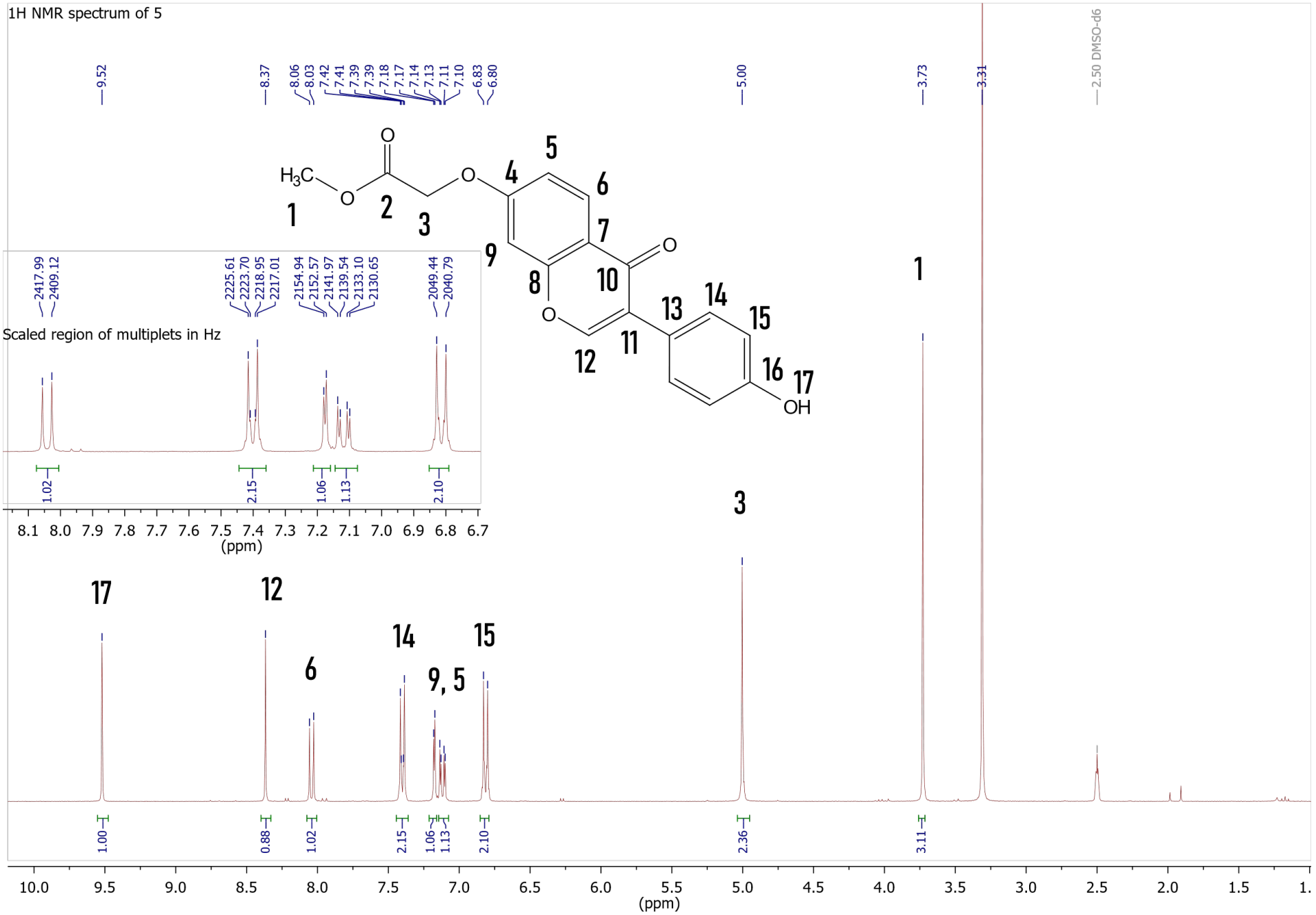
^{13}C NMR spectrum of methyl 2-[(3-iodo-4-oxo-4*H*-chromen-7-yl)oxy]acetate (**4**)



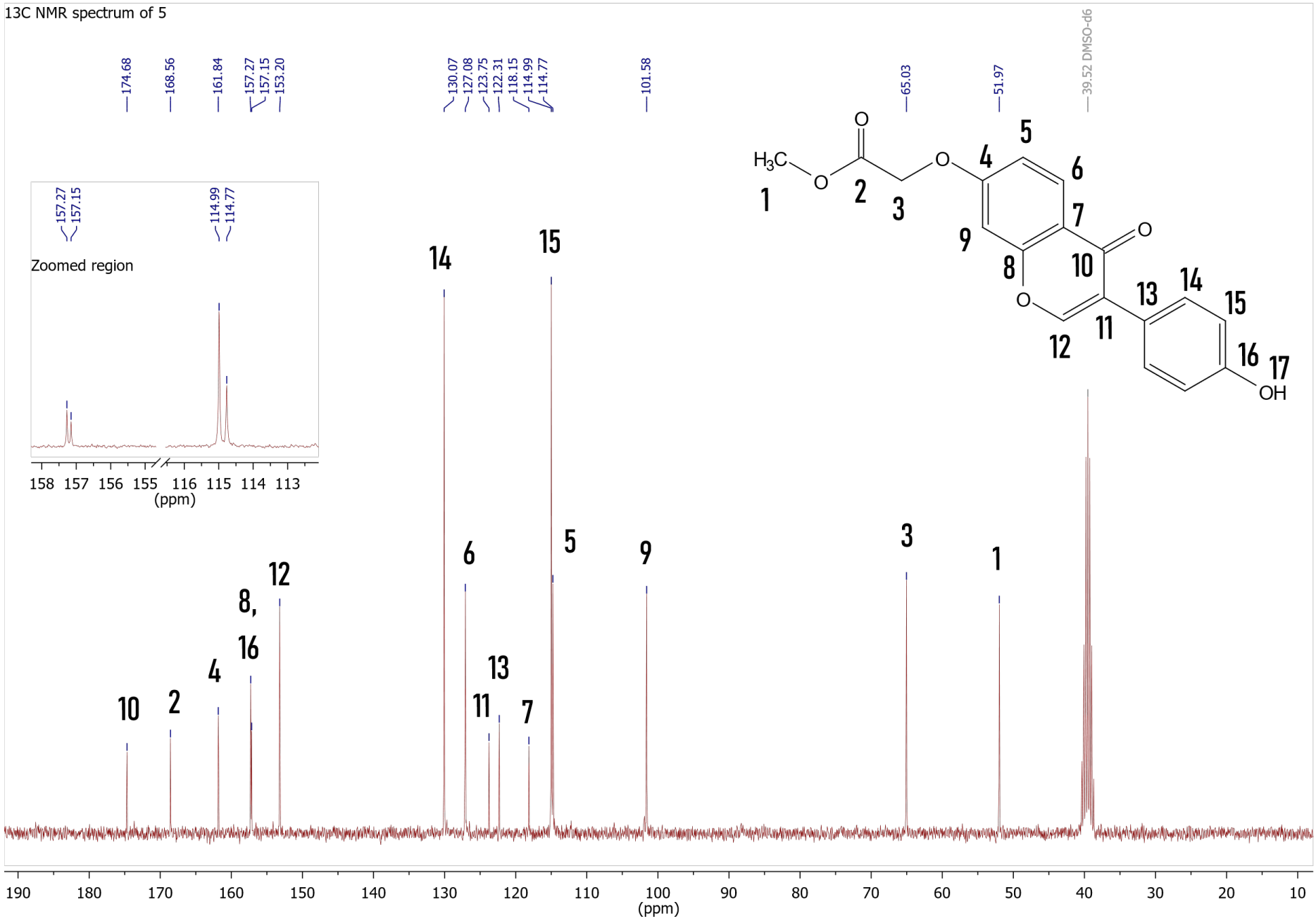
HMBC NMR spectrum of methyl 2-[(3-iodo-4-oxo-4*H*-chromen-7-yl)oxy]acetate (**4**)



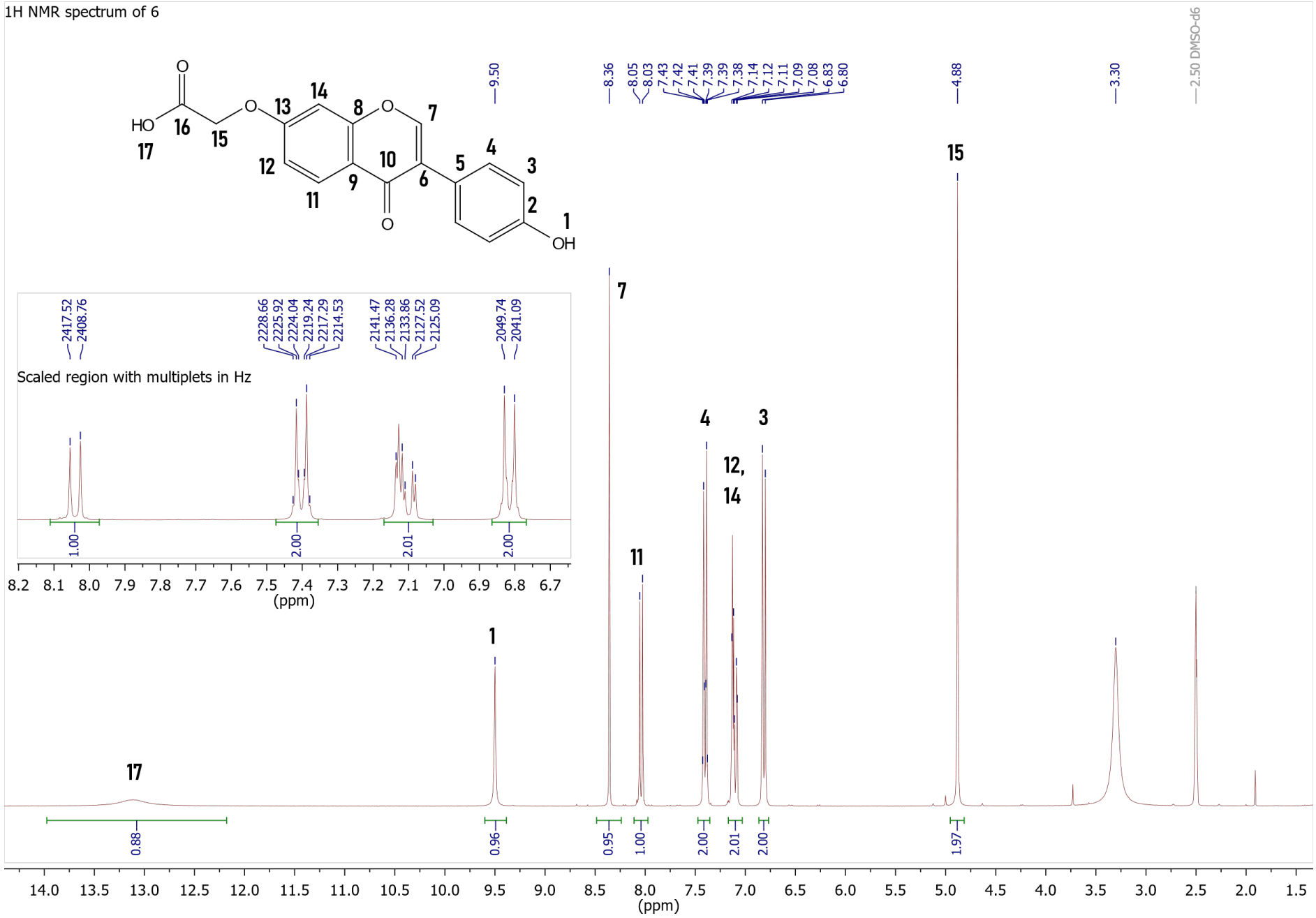
[1]H NMR spectrum of methyl 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetate (**5**)



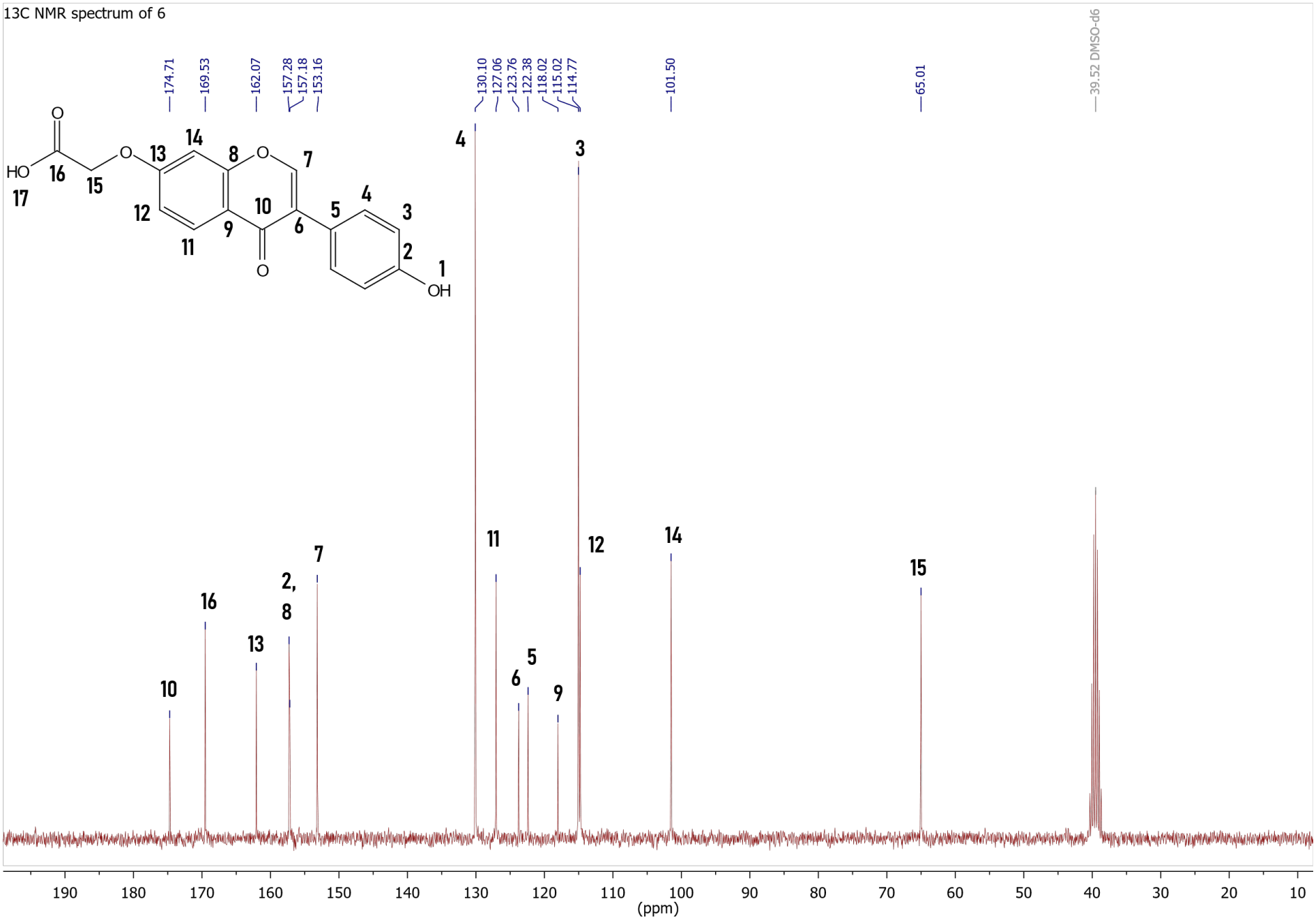
^{13}C NMR spectrum of methyl 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetate (**5**)



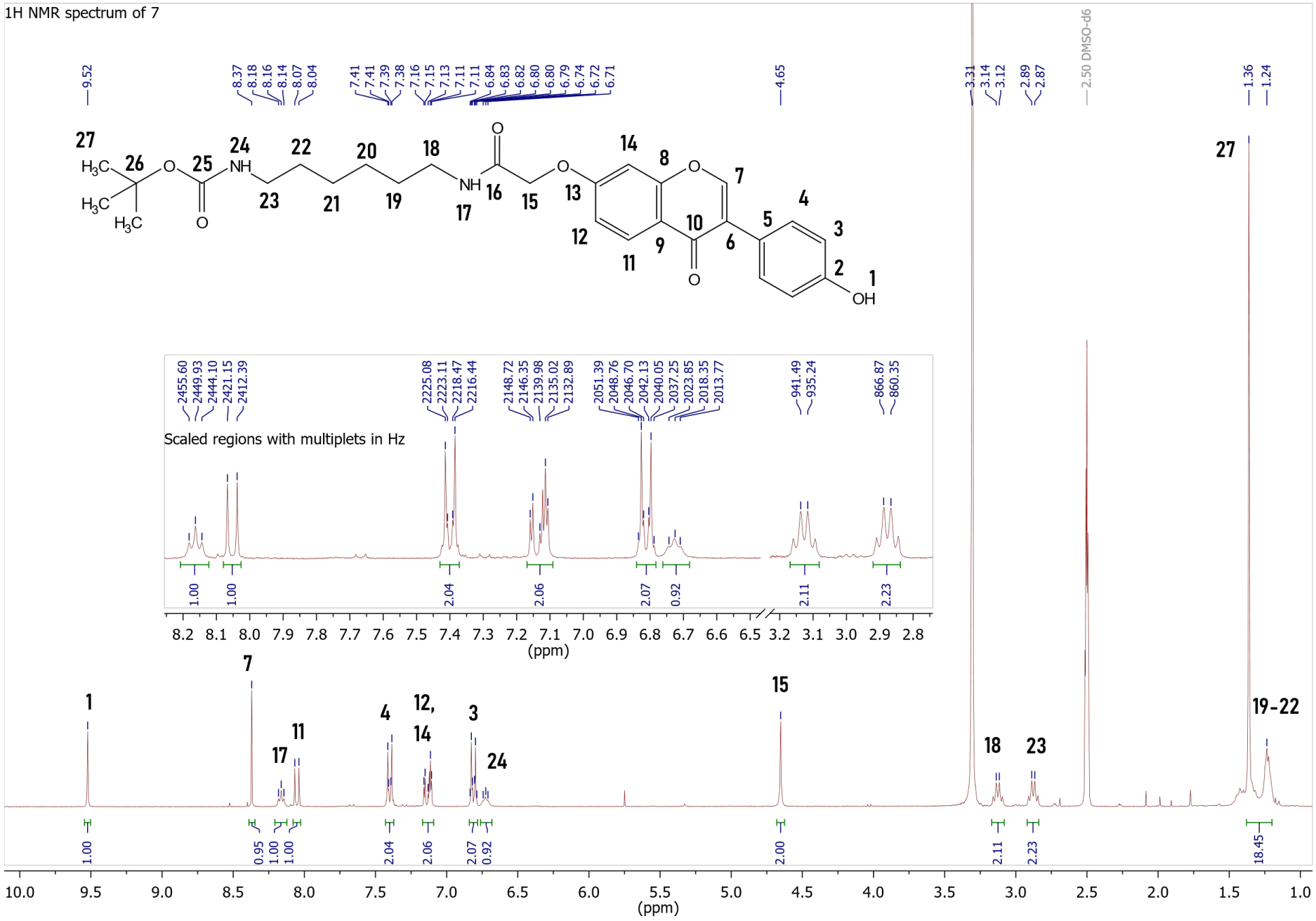
^{1}H NMR spectrum of 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetic acid (**6**)



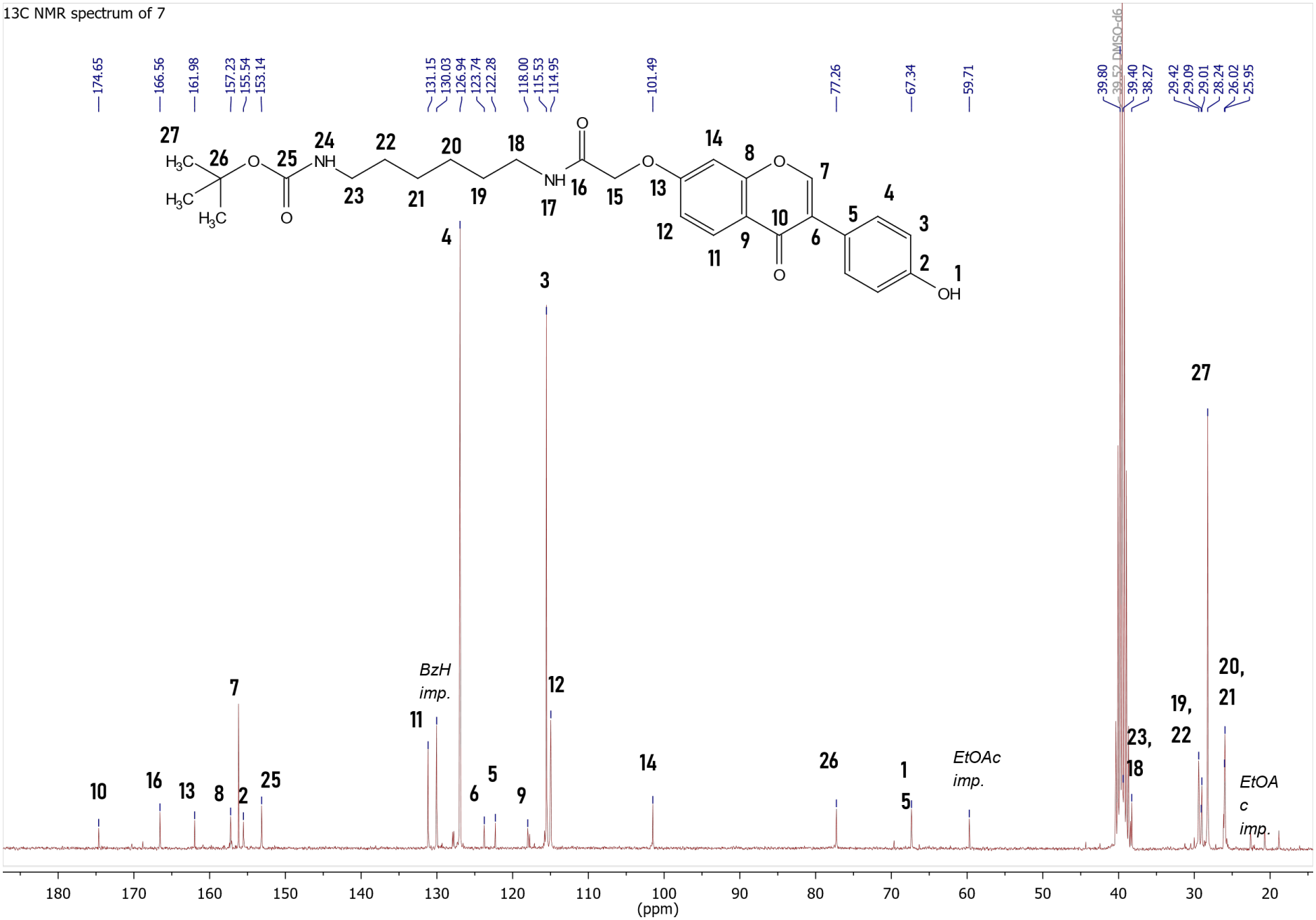
^{13}C NMR spectrum of 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetic acid (**6**)



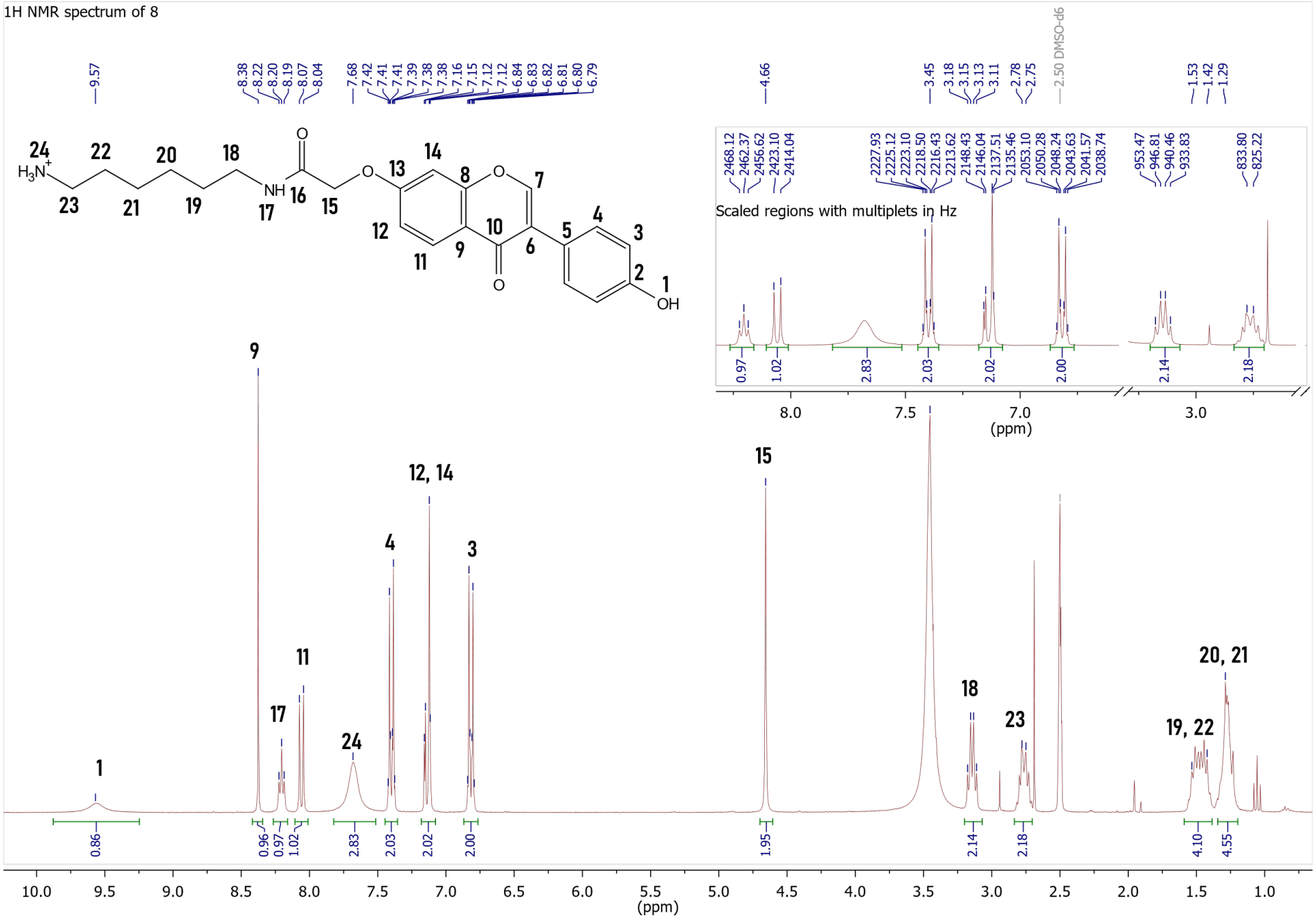
[1]H NMR spectrum of *N*-[6-(2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamido)hexyl]carbamate (**7**)



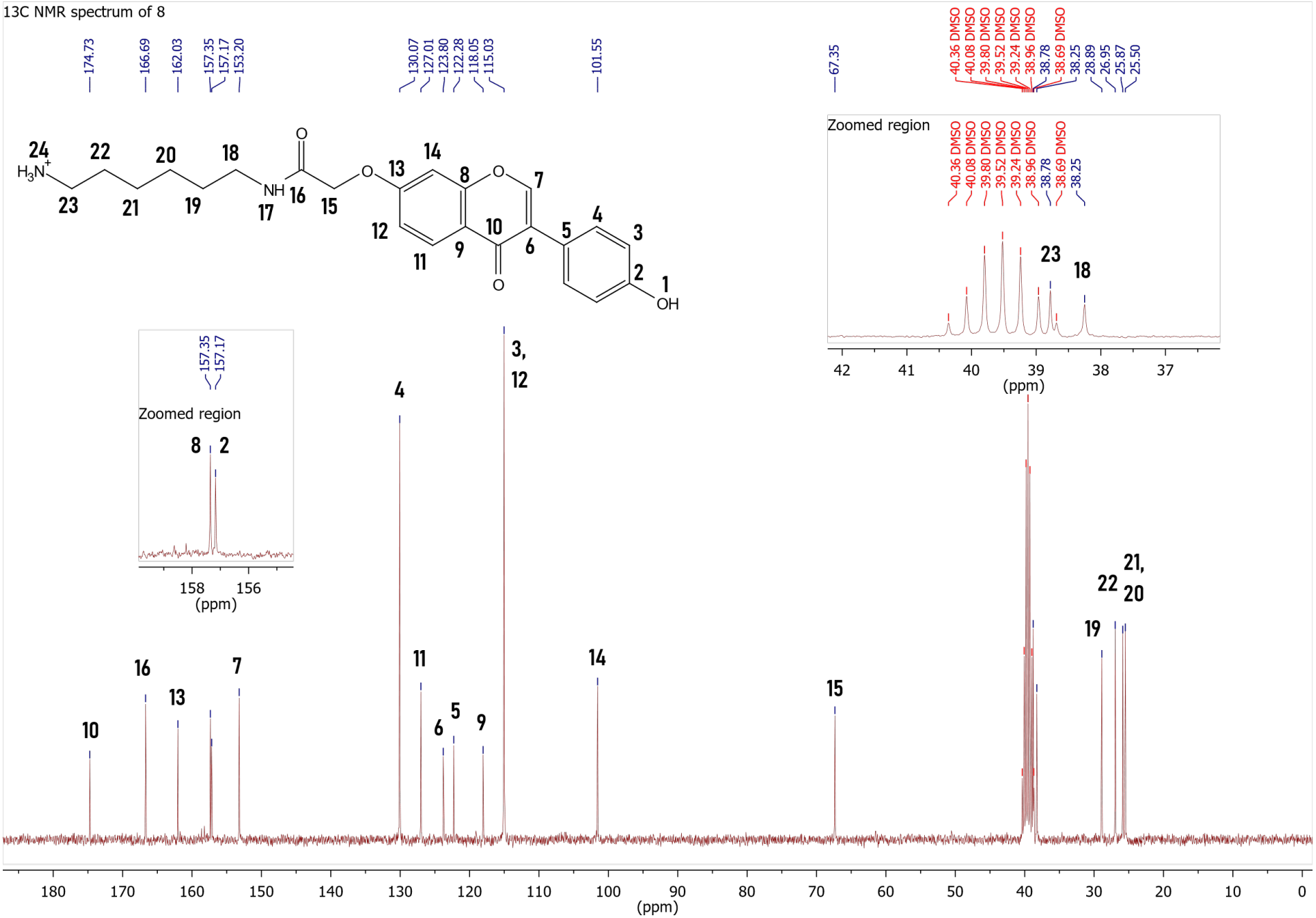
^{13}C NMR spectrum of *N*-[6-(2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamido)hexyl]carbamate (**7**)



[1]H NMR spectrum of *N*-(6-aminohexyl)-2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamide (**8**)



^{13}C NMR spectrum of *N*-(6-aminohexyl)-2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamide (**8**)



HPLC chromatogram and HRMS spectrum of methyl 2-(4-acetyl-3-hydroxyphenoxy)acetate (**2**)

Analysis Info

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Comment			

Acquisition Parameter

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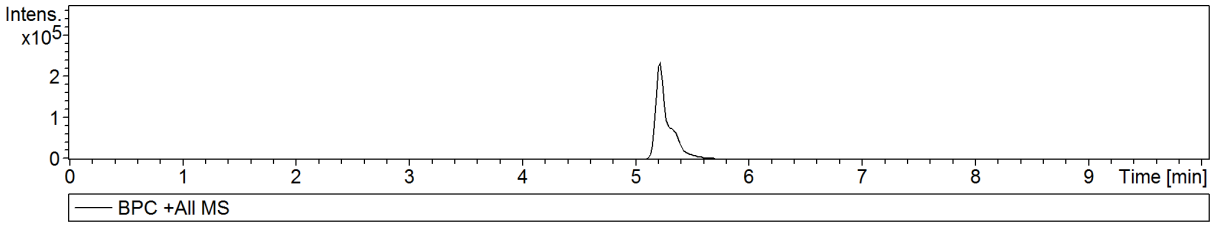

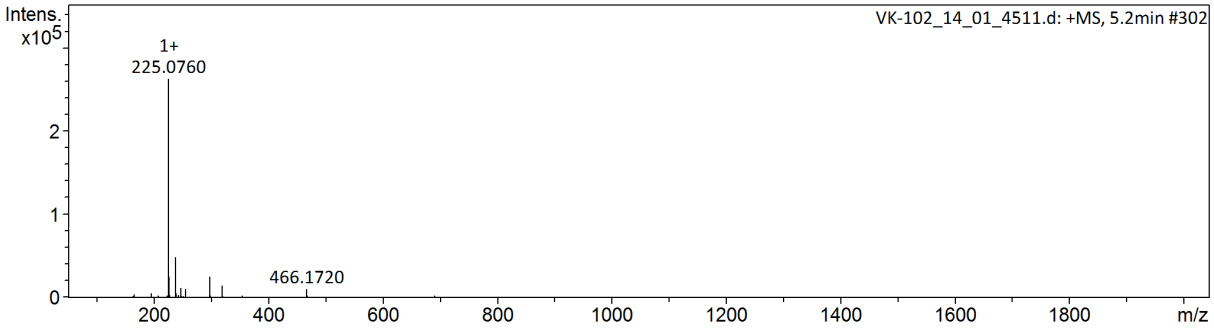

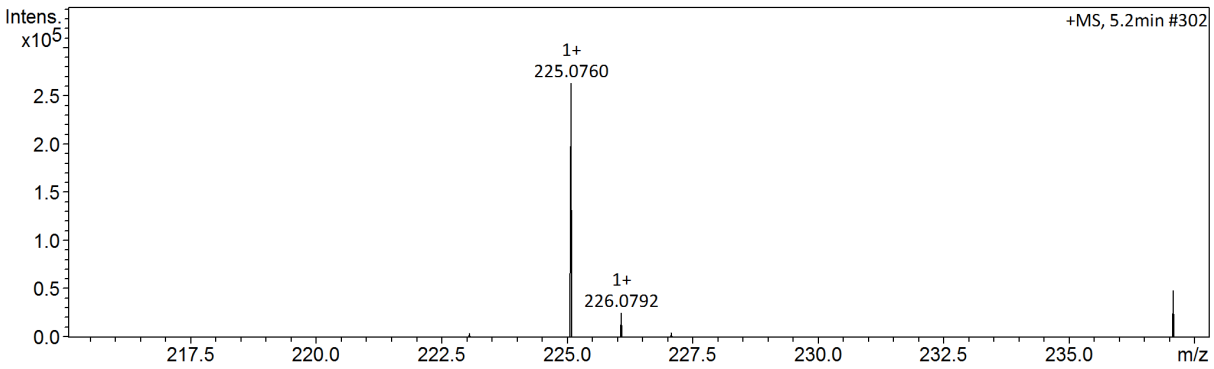

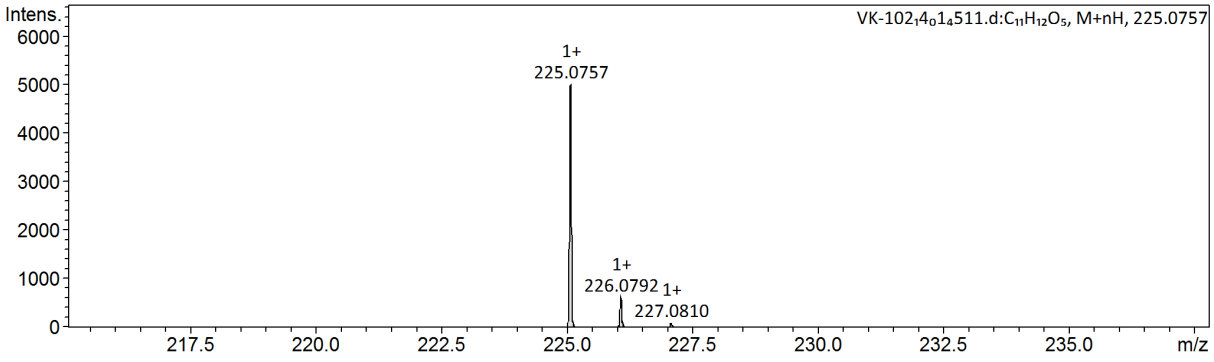

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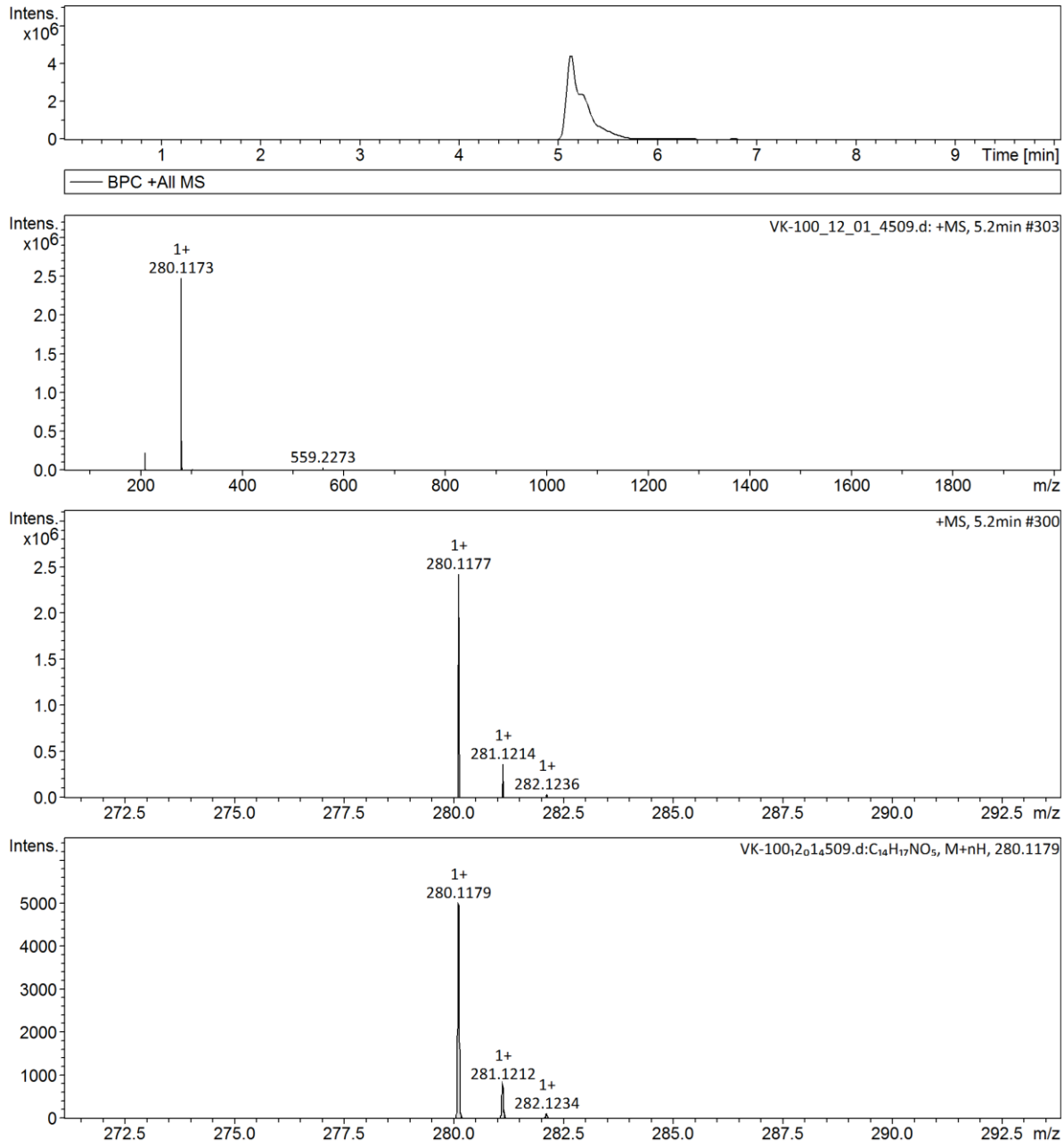
HPLC chromatogram and HRMS spectrum of methyl 2-{4-[3-(dimethylamino)acryloyl]-3-hydroxyphenoxy}acetate (**3**)

Analysis Info

Analysis Name	D:\Data\VK-100_12_01_4509.d	Acquisition Date	5/29/2024 5:21:19 PM
Method	la_2.2_small2.m	Operator	BDAL@DE
Sample Name	VK-100	Instrument	compact 8255754.20088
Comment			

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 C



VK-100_12_01_4509.d

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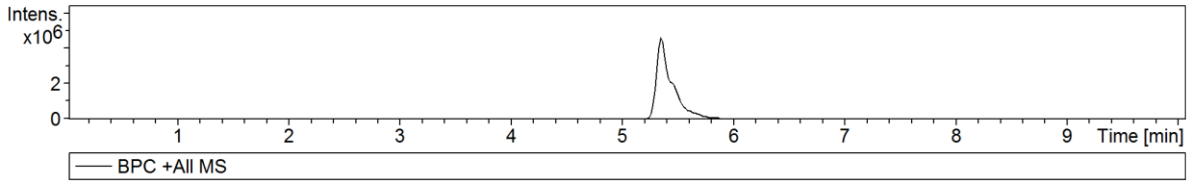
HPLC chromatogram and HRMS spectrum of methyl 2-[(3-iodo-4-oxo-4*H*-chromen-7-yl)oxy]acetate (**4**)

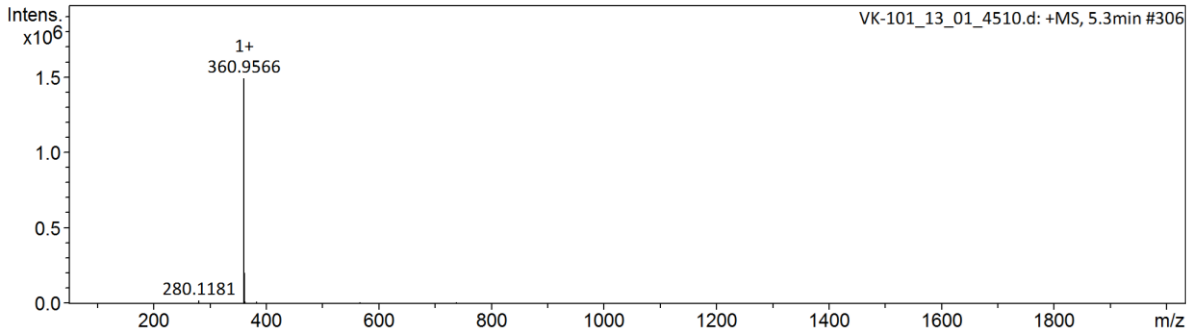
Analysis Info

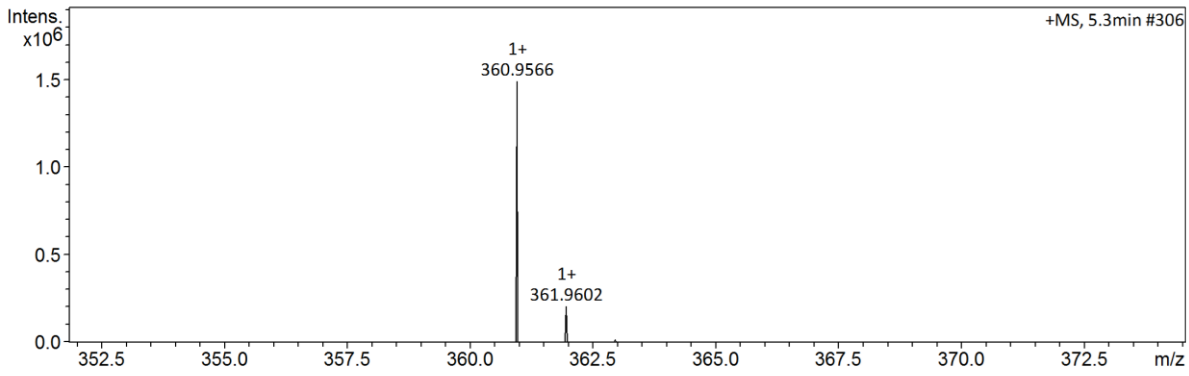
Analysis Name	D:\Data\VK-101_13_01_4510.d	Acquisition Date	5/29/2024 5:32:51 PM
Method	la_2.2_small2.m	Operator	BDAL@DE
Sample Name	VK-101	Instrument	compact 8255754.20088
Comment			

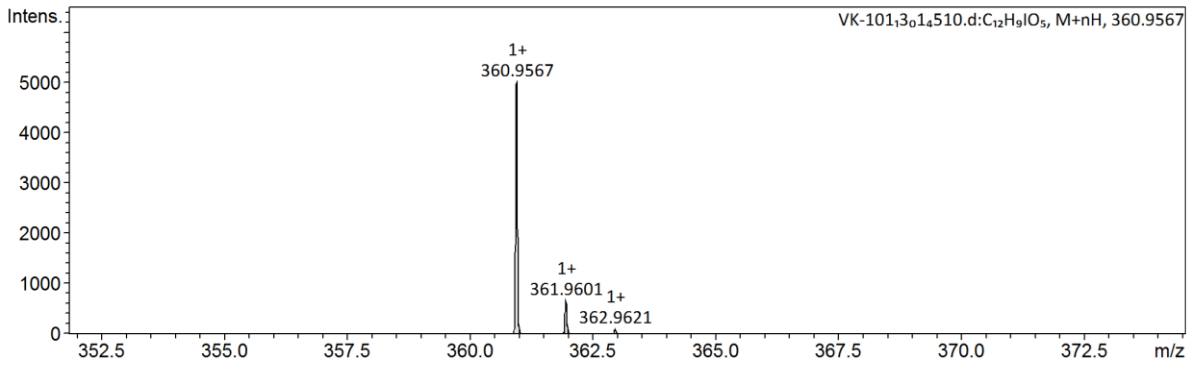
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 C









VK-101_13_01_4510.d

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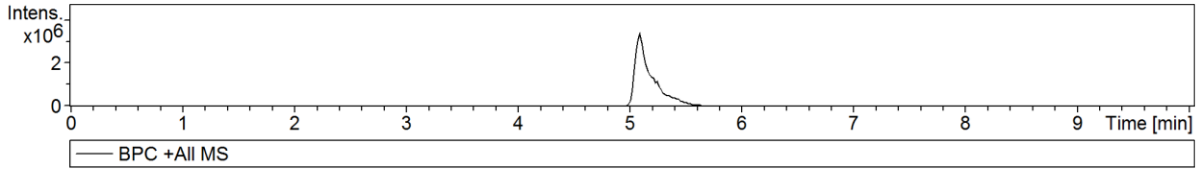
HPLC chromatogram and HRMS spectrum of methyl 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetate (**5**)

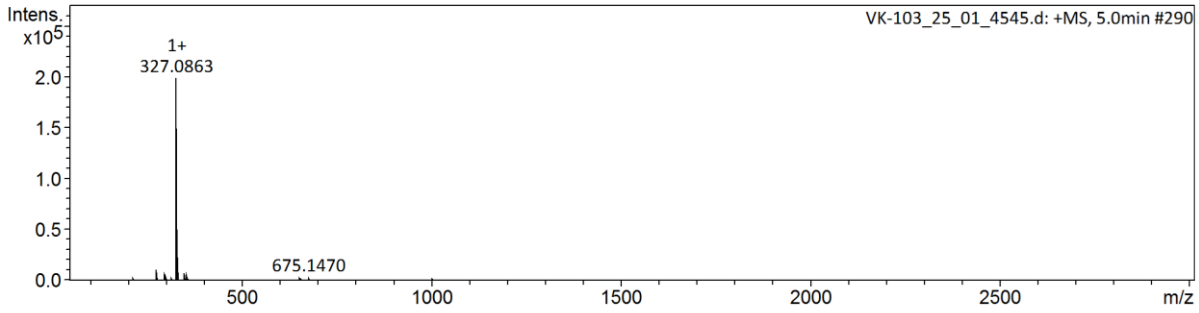
Analysis Info

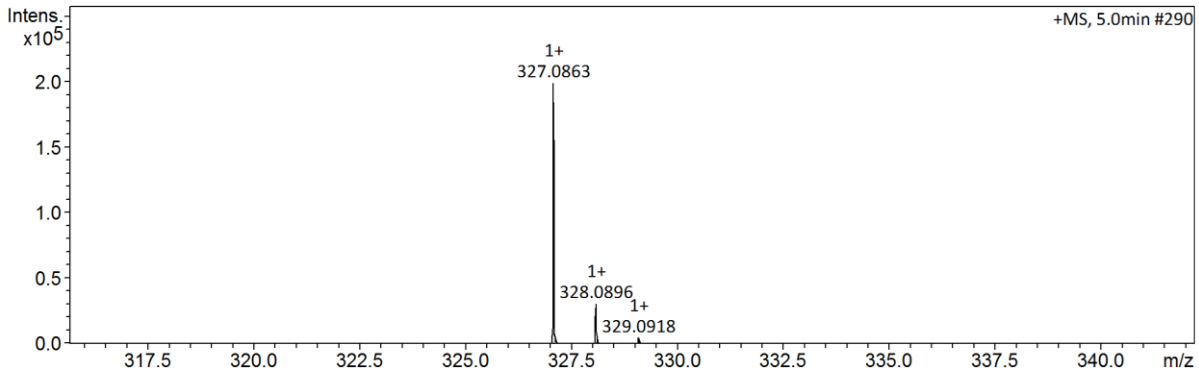
Analysis Name	D:\Data\VK-103_25_01_4545.d	Acquisition Date	6/4/2024 2:12:59 PM
Method	la_2.2_small2.m	Operator	BDAL@DE
Sample Name	VK-103	Instrument	compact 8255754.20088
Comment			

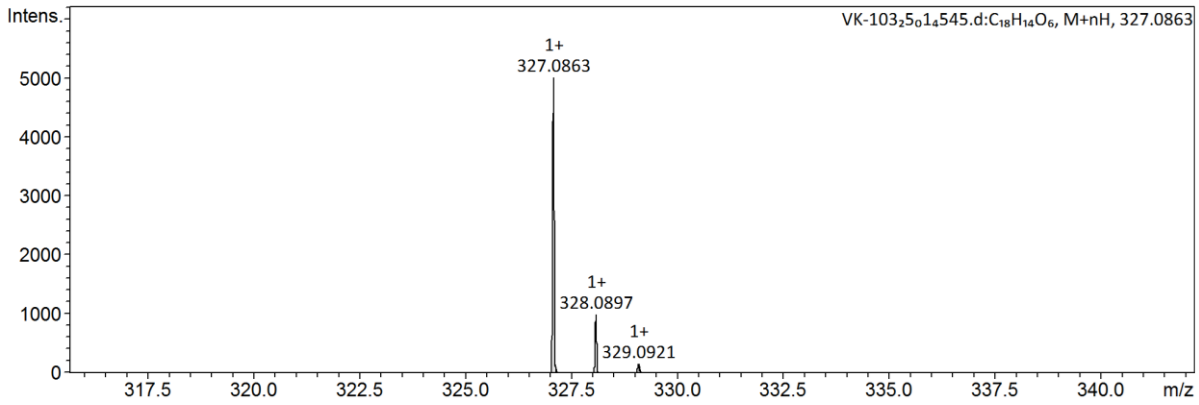
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 C









VK-103_25_01_4545.d

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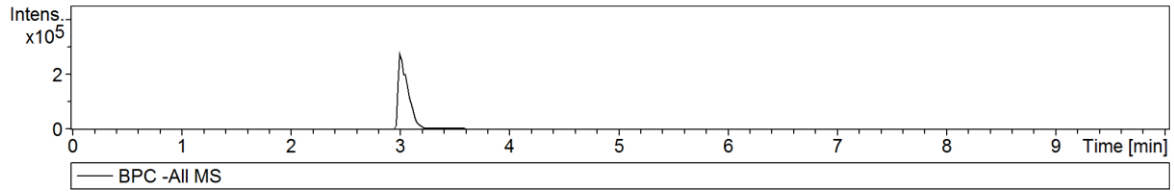
HPLC chromatogram and HRMS spectrum of 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetic acid (**6**)

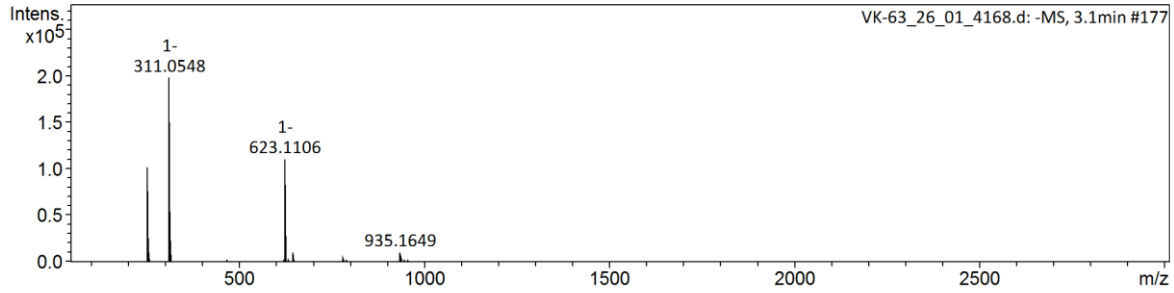
Analysis Info

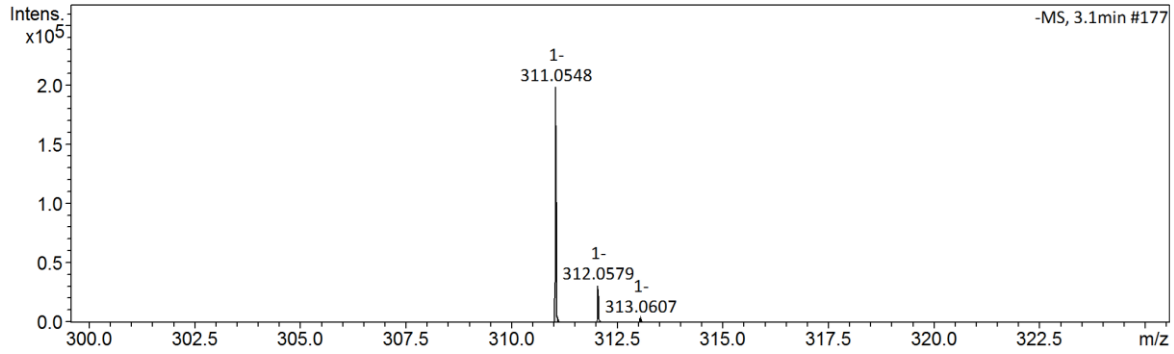
Analysis Name	D:\Data\VK-63_26_01_4168.d	Acquisition Date	6/2/2023 8:12:18 PM
Method	mg_02_neg-pico.m	Operator	BDAL@DE
Sample Name	VK-63	Instrument	compact 8255754.20088
Comment			

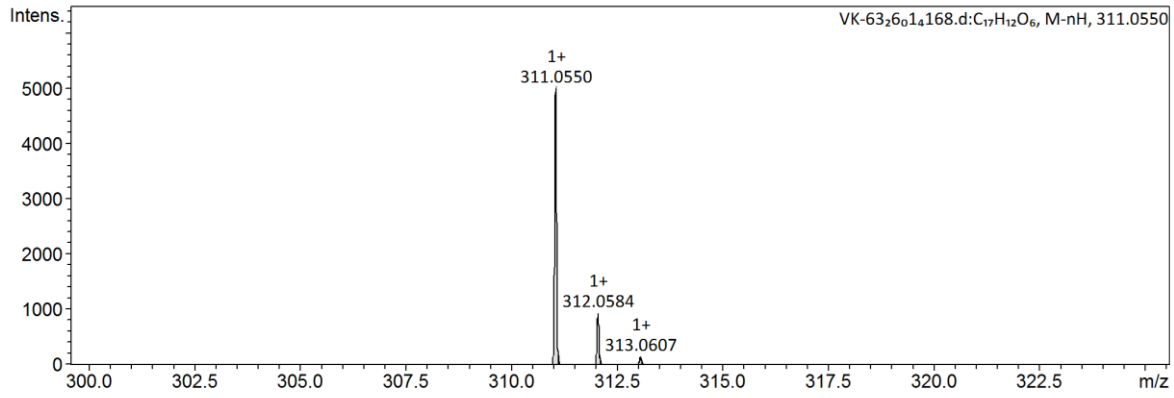
Acquisition Parameter

Source Type	ESI	Ion Polarity	Negative	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3700 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 °C









VK-63_26_01_4168.d
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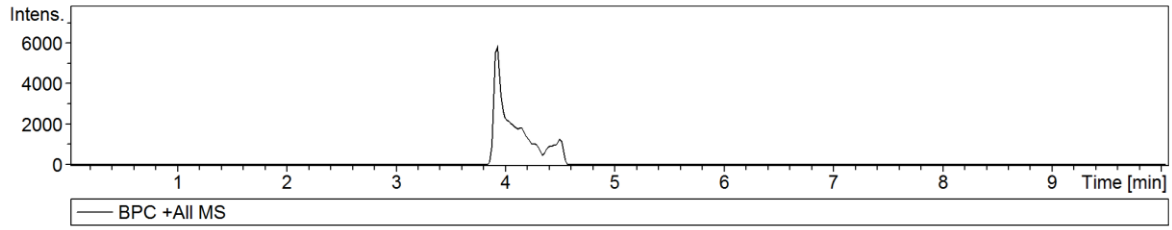
HPLC chromatogram and HRMS spectrum of *tert*-butyl *N*-[6-(2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamido)-hexyl]carbamate (**7**)

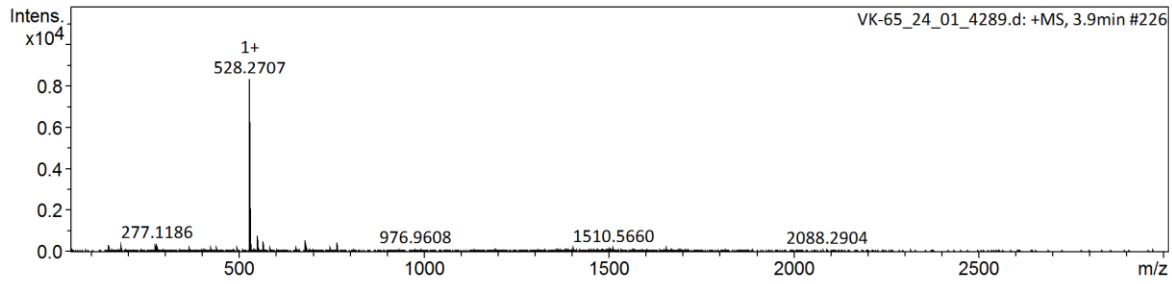
Analysis Info

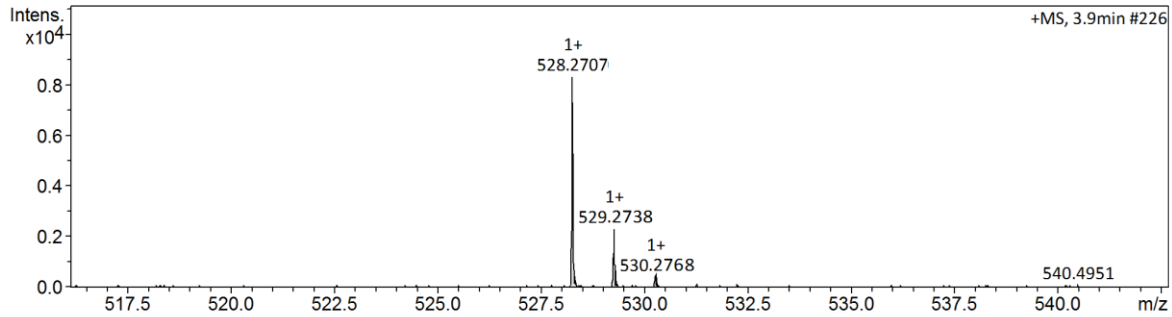
Analysis Name	D:\Data\VK-65_24_01_4289.d	Acquisition Date	10/2/2023 10:54:41 PM
Method	la_2.2_small2.m	Operator	BDAL@DE
Sample Name	VK-65	Instrument	compact 8255754.20088
Comment			

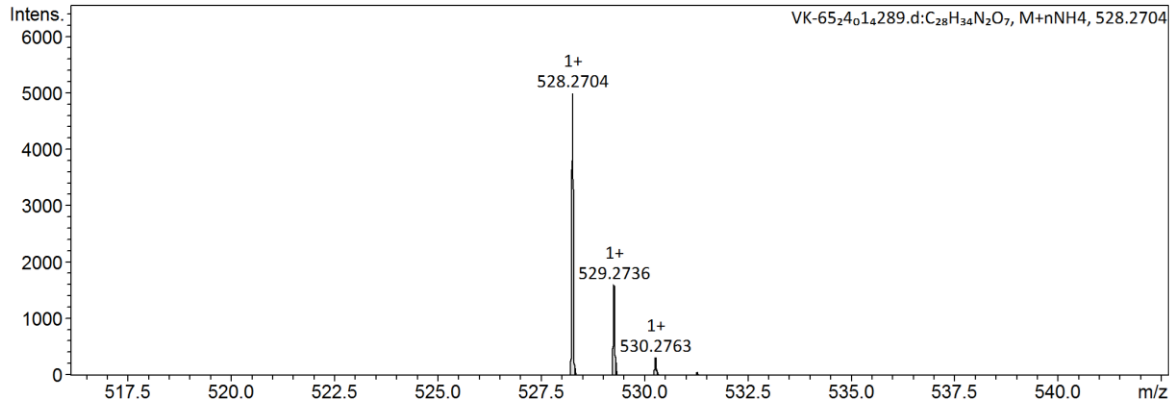
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 C









VK-65_24_01_4289.d

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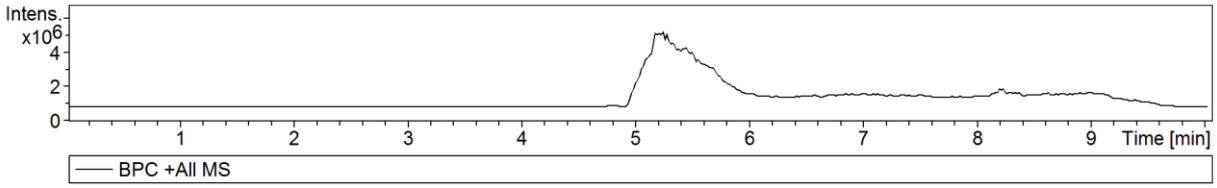
HPLC chromatogram and HRMS spectrum of *N*-(6-aminohexyl)-2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamide (**8**)

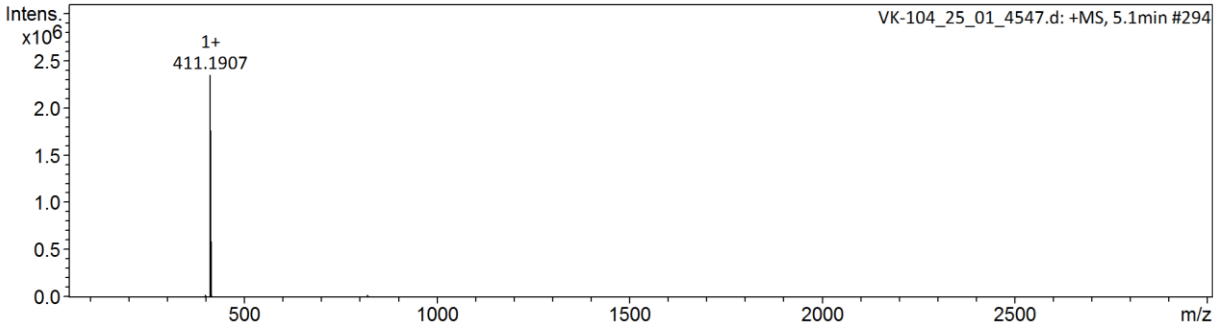
Analysis Info

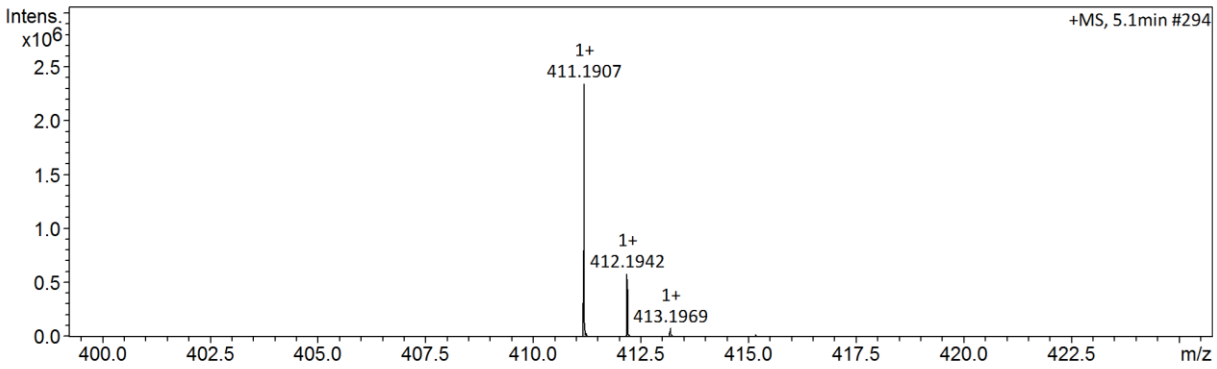
Analysis Name	D:\Data\VK-104_25_01_4547.d	Acquisition Date	6/5/2024 1:06:58 PM
Method	la_2.2_small2.m	Operator	BDAL@DE
Sample Name	VK-104	Instrument	compact 8255754.20088
Comment			

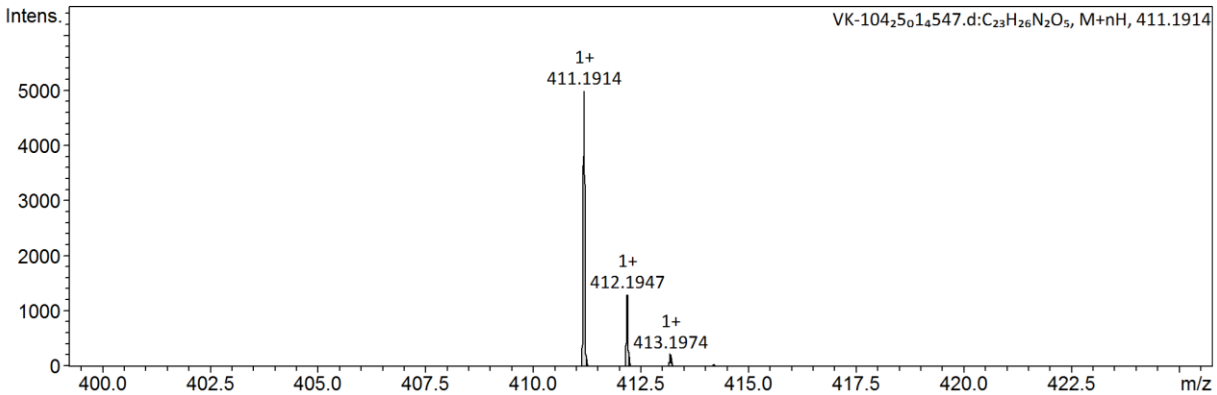
Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	180 C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	4.0 l/min
Scan End	3000 m/z	Set Charging Voltage	2000 V	Set Divert Valve	Source
		Set Corona	0 nA	Set APCI Heater	0 C









VK-104_25_01_4547.d

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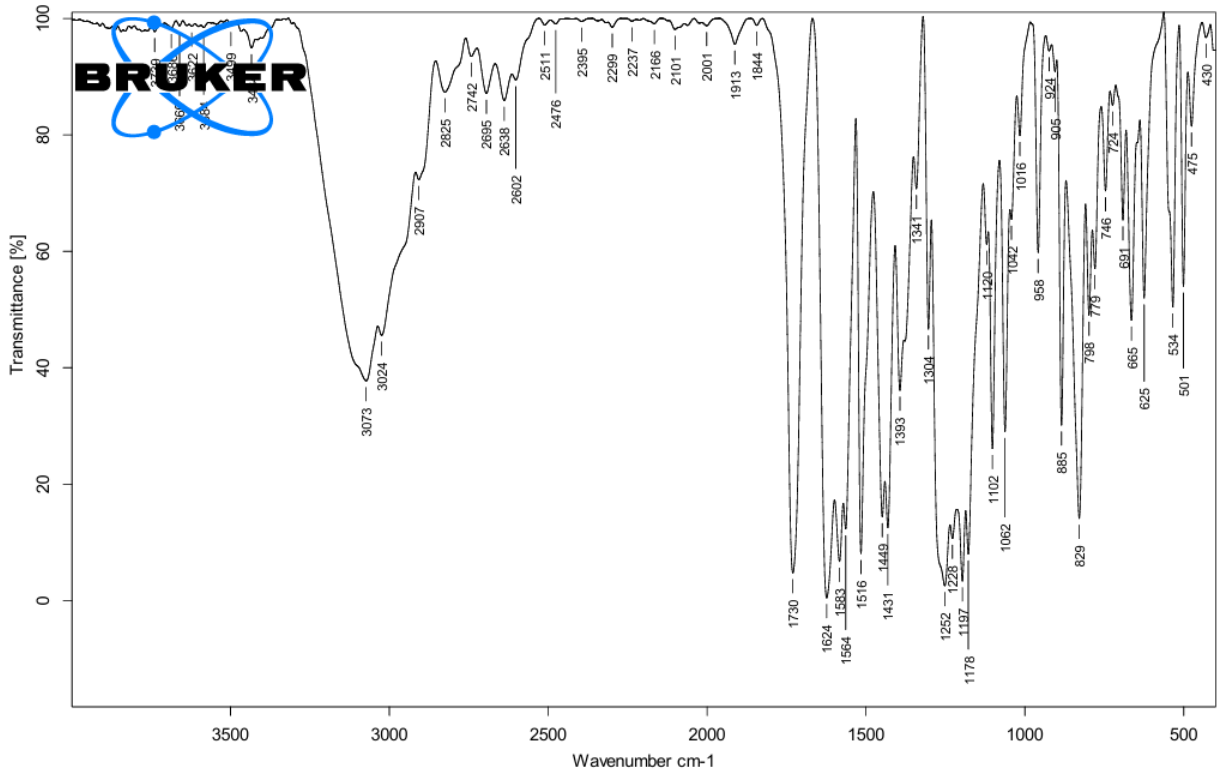
FTIR spectrum of 2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetic acid (**6**)



FTIR spectrum of *N*-(6-aminohexyl)-2-{[3-(4-hydroxyphenyl)-4-oxo-4*H*-chromen-7-yl]oxy}acetamide (**8**)