

One-Pot Synthesis and Evaluation of Antioxidative Stress and Anticancer Properties of an Active Chromone Derivative

Chirattikan Maicheen¹, Chokchaloemwat Churnthammakarn², Nichapat Pongsroypech², Thitiphong Khamkhenshorngphanuch^{2,3}, Jiraporn Ungwitayatorn⁴, Kanin Rangsangthong², Rathapon Asasutjarit^{2,3}, Sewan Theeramunkong^{*2,3}

1 Department of Pharmaceutical Chemistry, Faculty of Pharmaceutical Sciences,
Huachiew Chalermprakiet University, Samut Prakarn 10540, Thailand

2 Faculty of Pharmacy, Thammasat University, Pathumthani, 12120, Thailand

3 Thammasat University Research Unit in Drug, Health Product Development and
Application (DHP-DA), Department of Pharmaceutical Sciences, Faculty of Pharmacy,
Thammasat University, Pathumthani, 12120, Thailand.

4 Department of Pharmaceutical Chemistry, Faculty of Pharmacy, Mahidol University,
Bangkok 10400, Thailand

*Corresponding author, e-mail: sewan@tu.ac.th

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1. General Materials and Methods

All reactions, unless otherwise indicated, were carried out in single-neck, round-bottom flasks fitted with a rubber septum and equipped with a magnetic stir bar. Air or water-sensitive solvents were transferred via a syringe. When required, solvents were degassed by bubbling nitrogen through a needle. A rotary evaporator concentrated the resulting organic layers at 45-70 °C under reduced pressure. The progress of the reaction was monitored with thin-layer chromatography (TLC) using a silica gel 60 F254 aluminum sheet. Visualization was performed with a 254 / 365 nm UV light source (Camag, Switzerland), generally followed by immersion in potassium permanganate (KMnO₄) or iodine vapor, as appropriate. Upon completion of the reaction, the crude products were prepared with a suitable procedure and finally dried and purified by column chromatography to provide a pure product.

The FTIR spectrum of the compounds was obtained by the KBr technique using an FTIR spectrometer (IRTracer-100 Shimadzu, Japan). The NMR spectrum was performed using an Ascend TM600/Advance III HD, Bruker, Switzerland. The spectrum of compounds was taken from the 4000 cm⁻¹ to 650 cm⁻¹ wave number range. ESI-TOF-MS was performed using a time-of-flight mass spectrometer (micrOTOF-Q-II, BrukerDaltonics, Germany). The ESI system positive-ion mode generated 200-800 m/z. The optimized mass spectrometric conditions included a gas temperature of 350 °C and a drying gas flow rate of 10.0 L/min.

2. ^1H -NMR spectrum of synthetic compounds

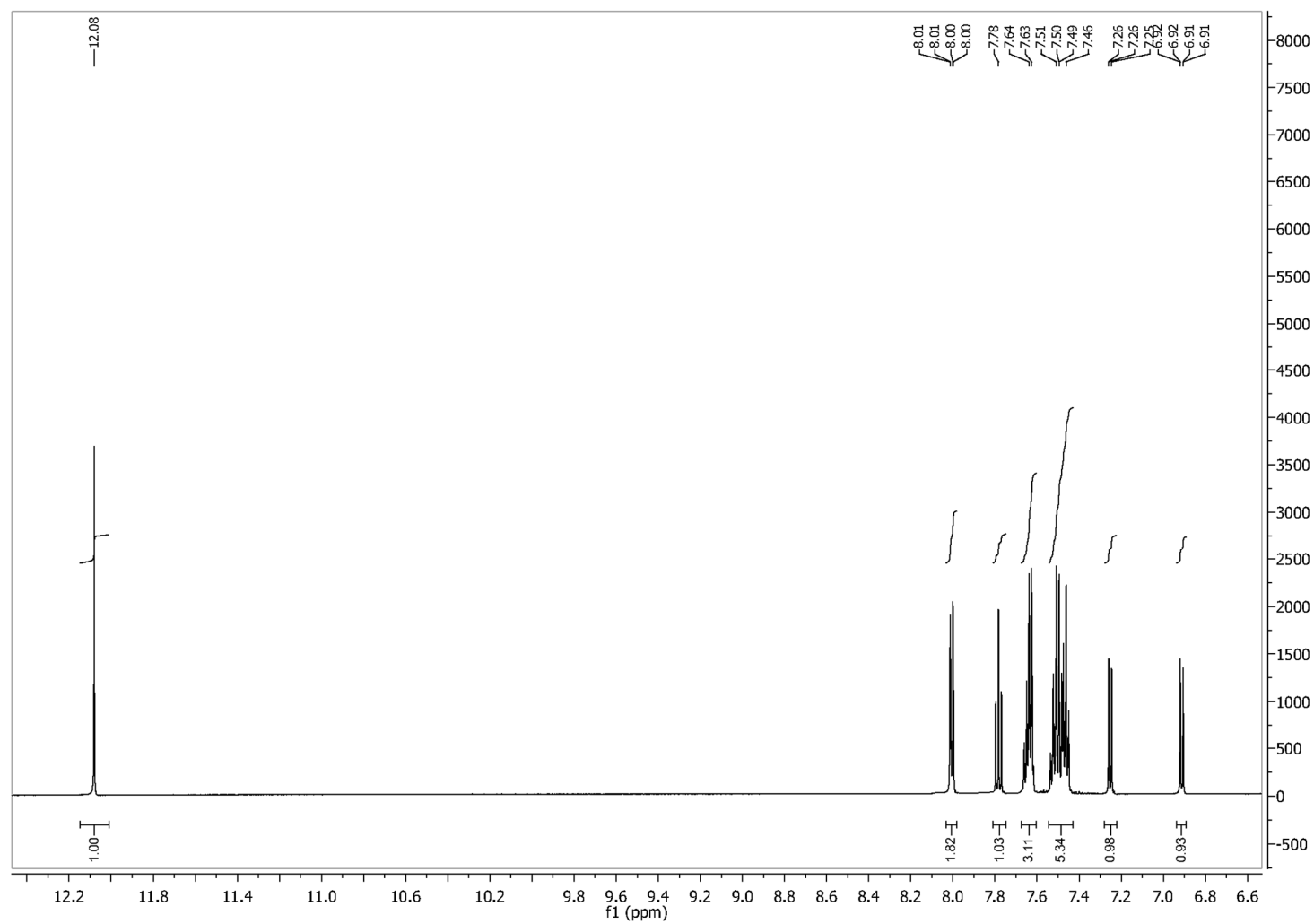


Figure S1 ^1H -NMR spectrum of compound 2a

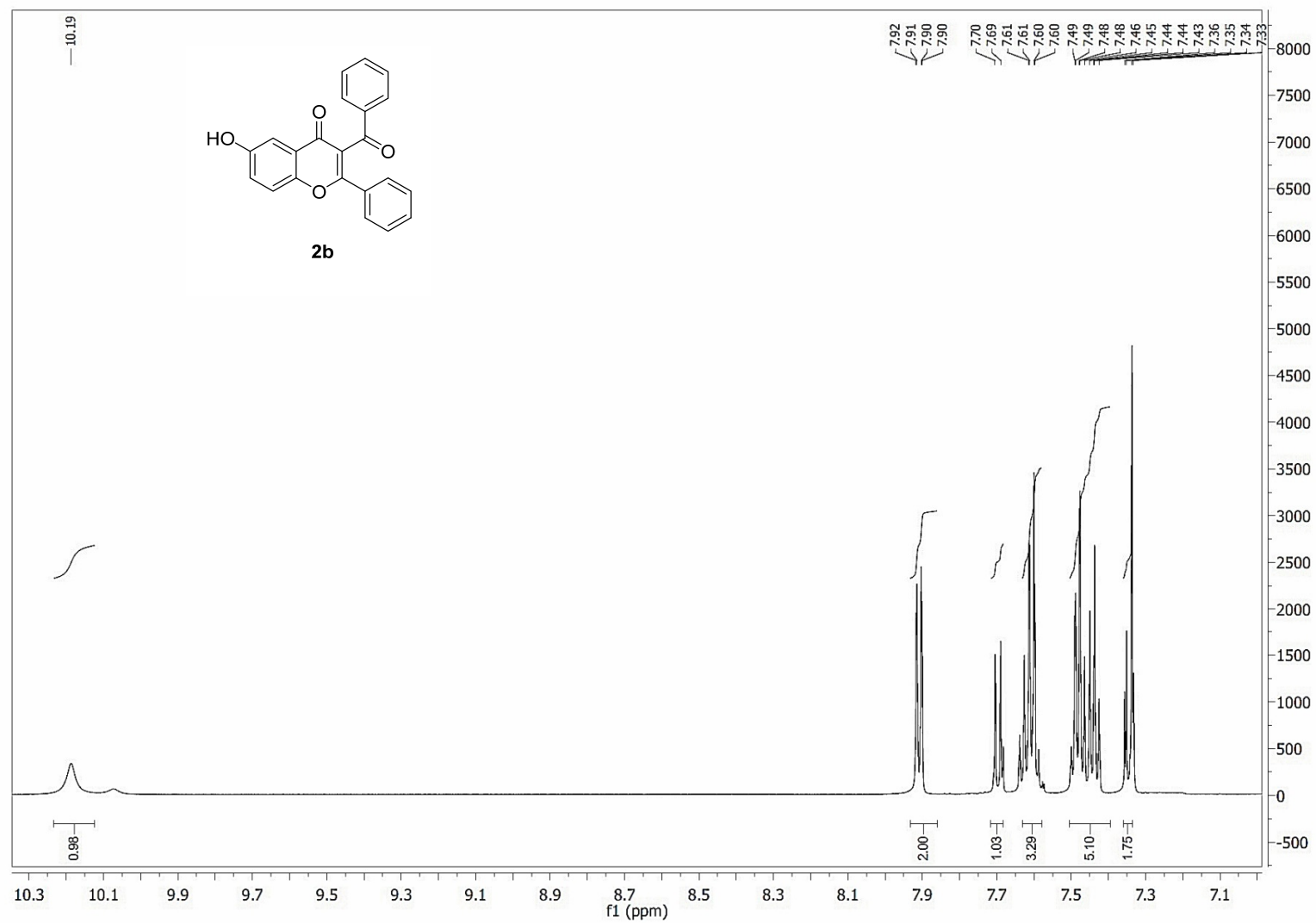


Figure S2 ^1H -NMR spectrum of compound **2b**

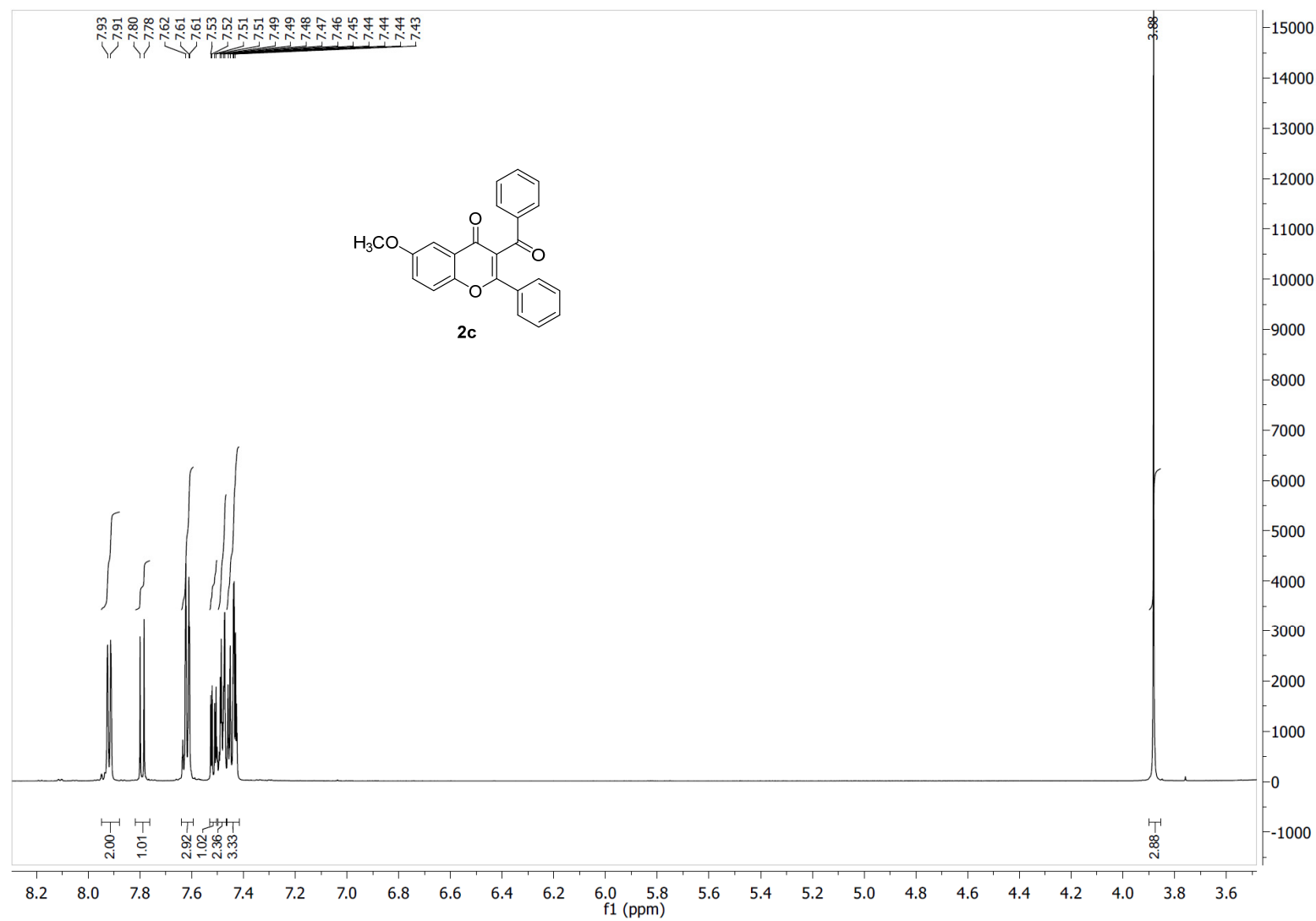


Figure S3 ^1H -NMR spectrum of compound **2c**

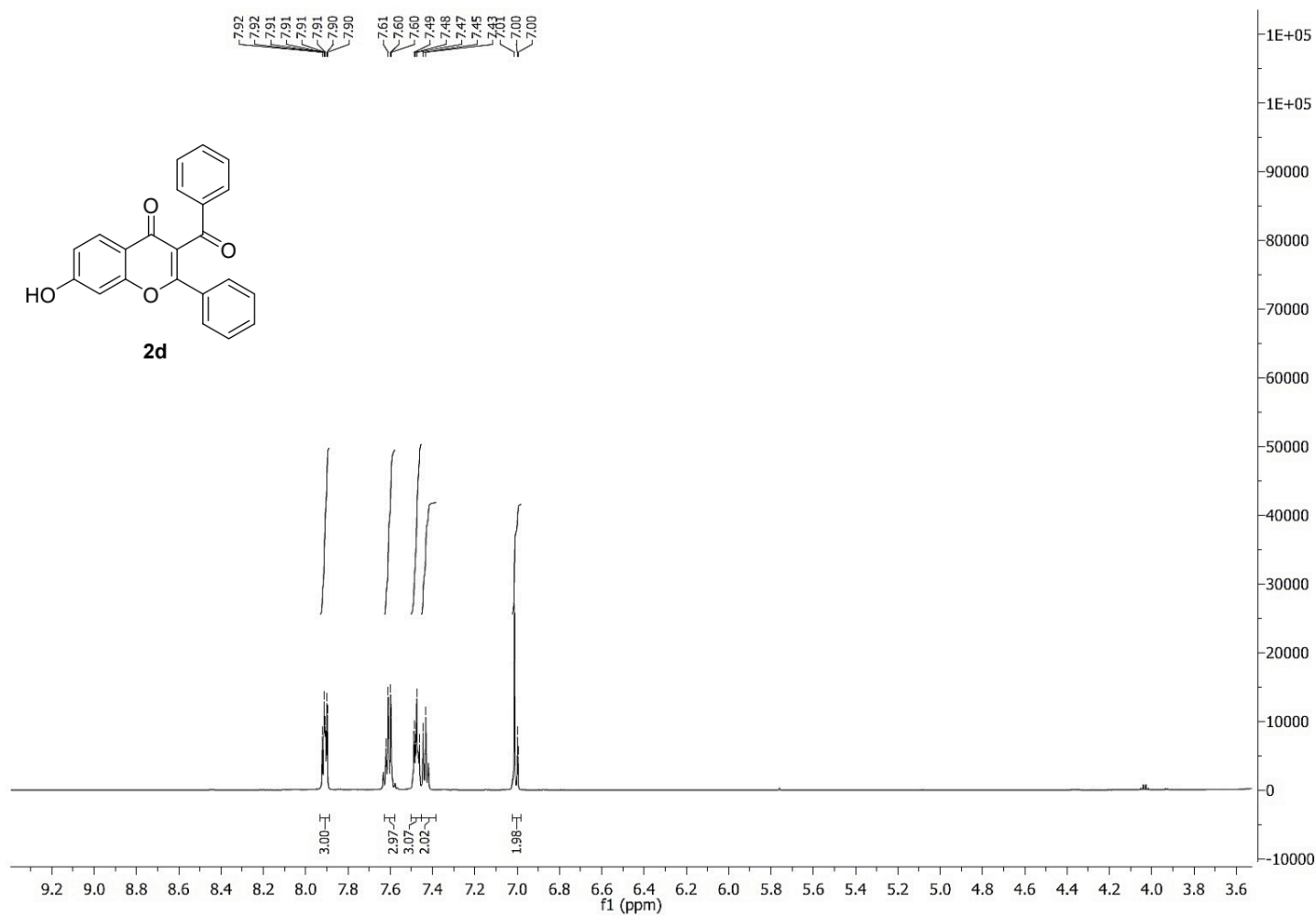


Figure S4 $^1\text{H-NMR}$ spectrum of compound **2d**

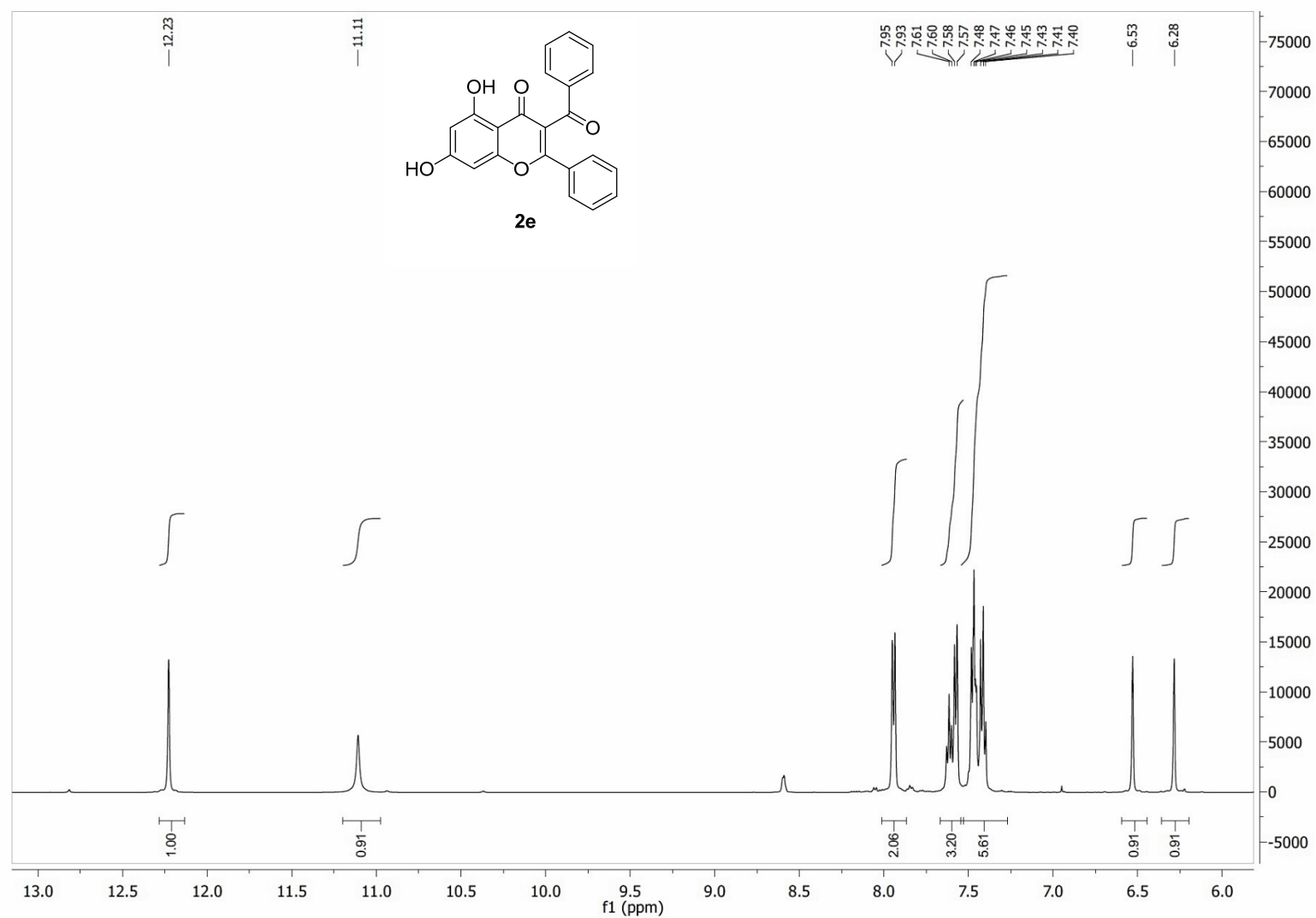


Figure S5 ^1H -NMR spectrum of compound **2e**

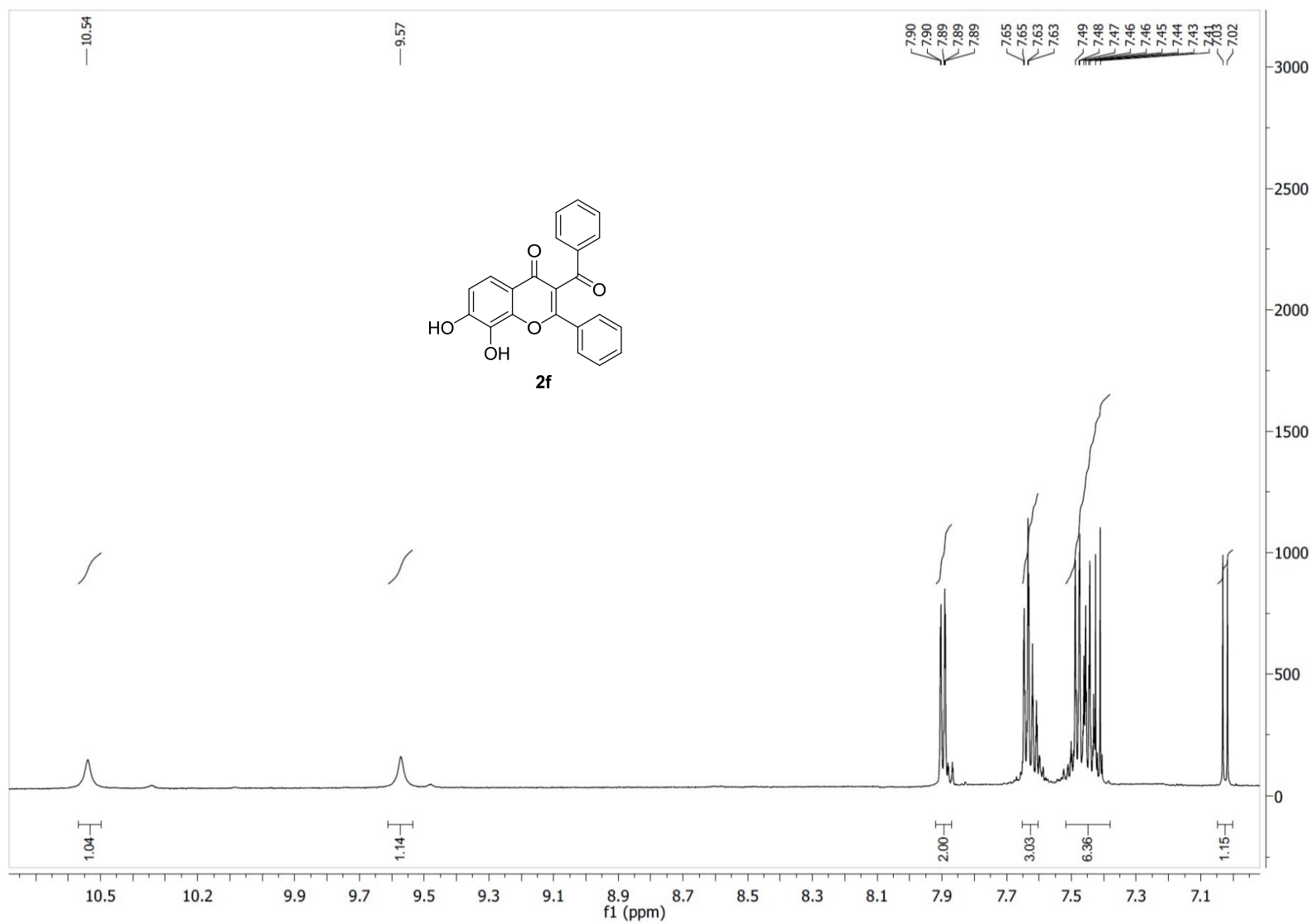


Figure S6 ^1H -NMR spectrum of compound **2f**

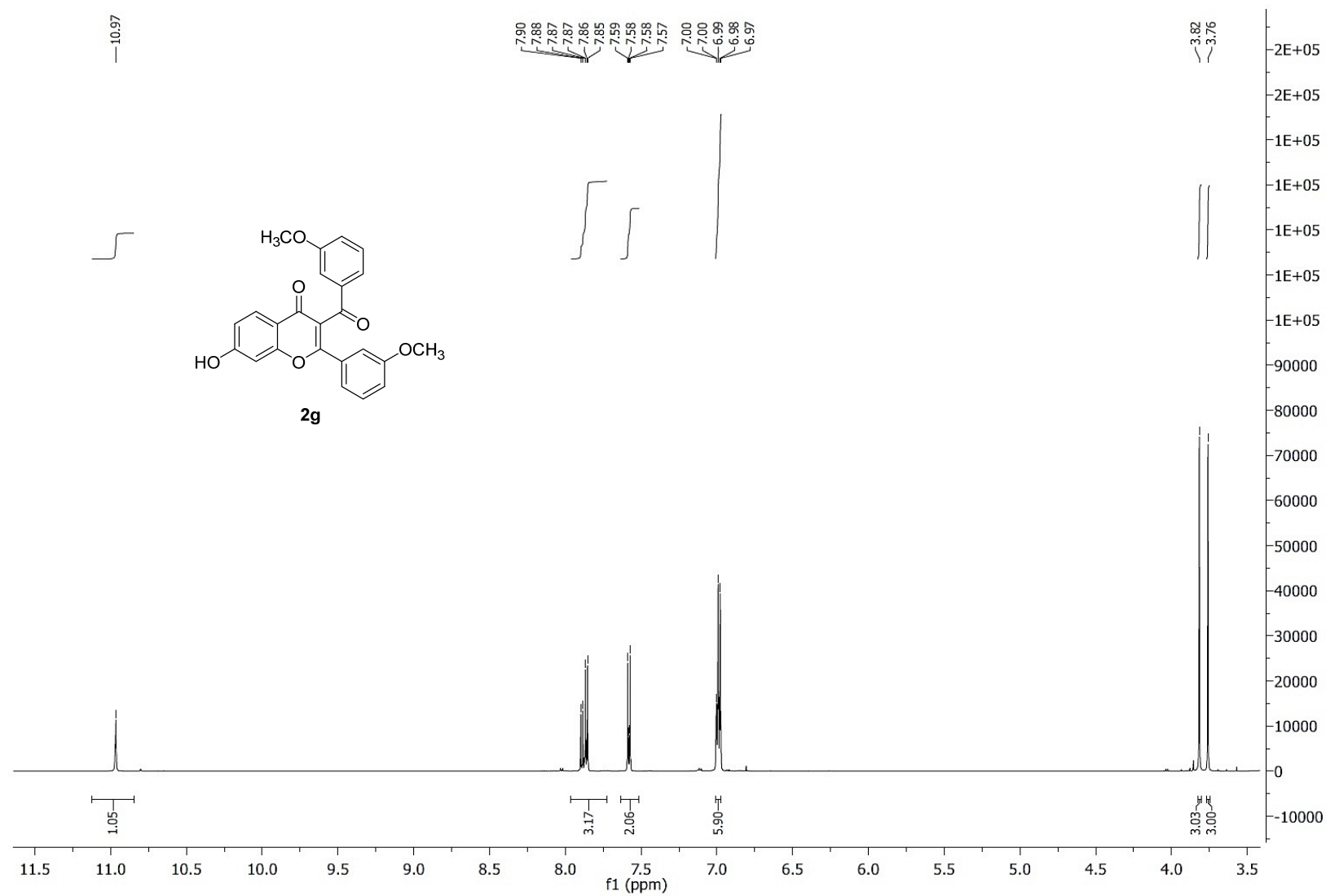


Figure S7 ^1H -NMR spectrum of compound **2g**

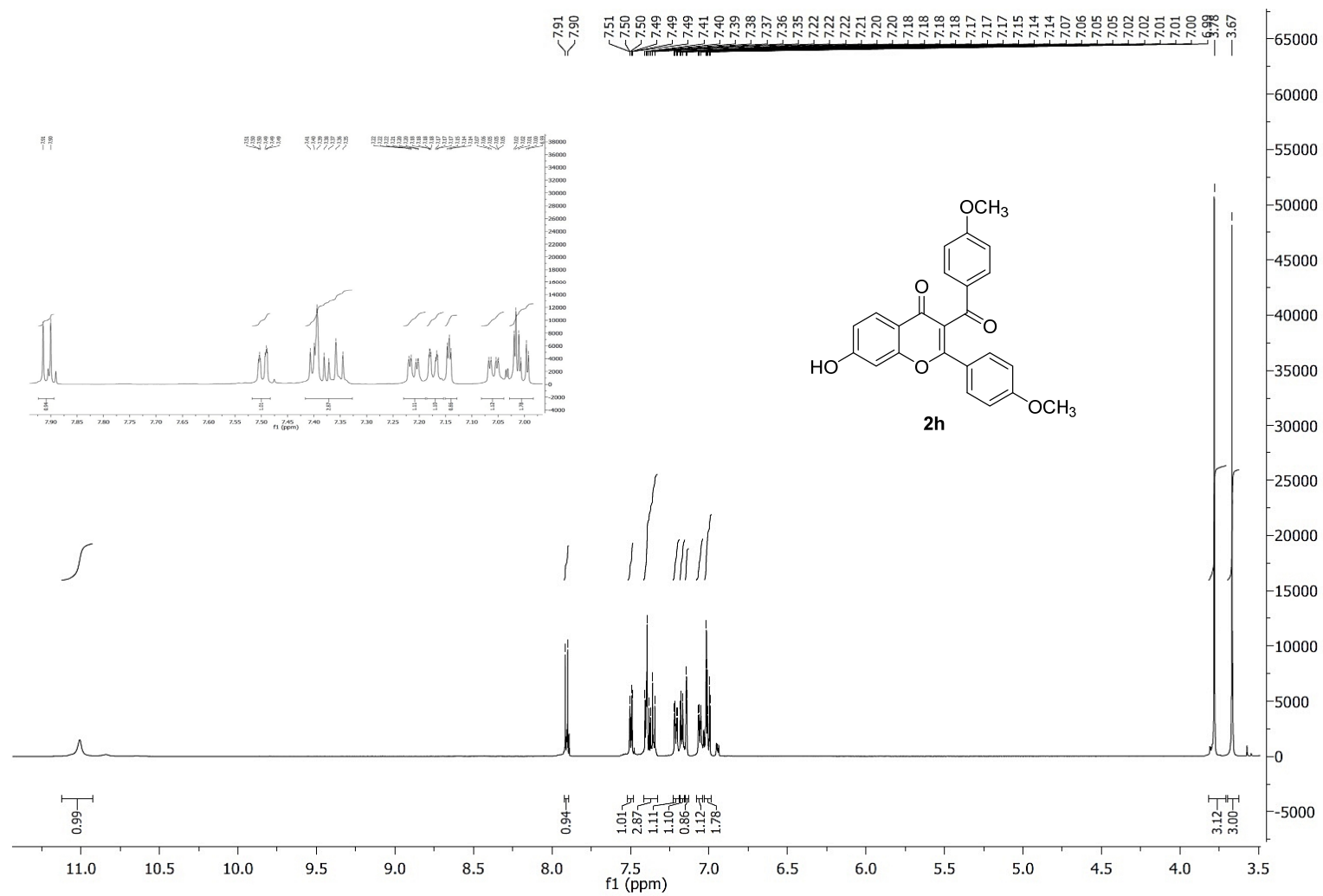


Figure S8 ^1H -NMR spectrum of compound **2h**

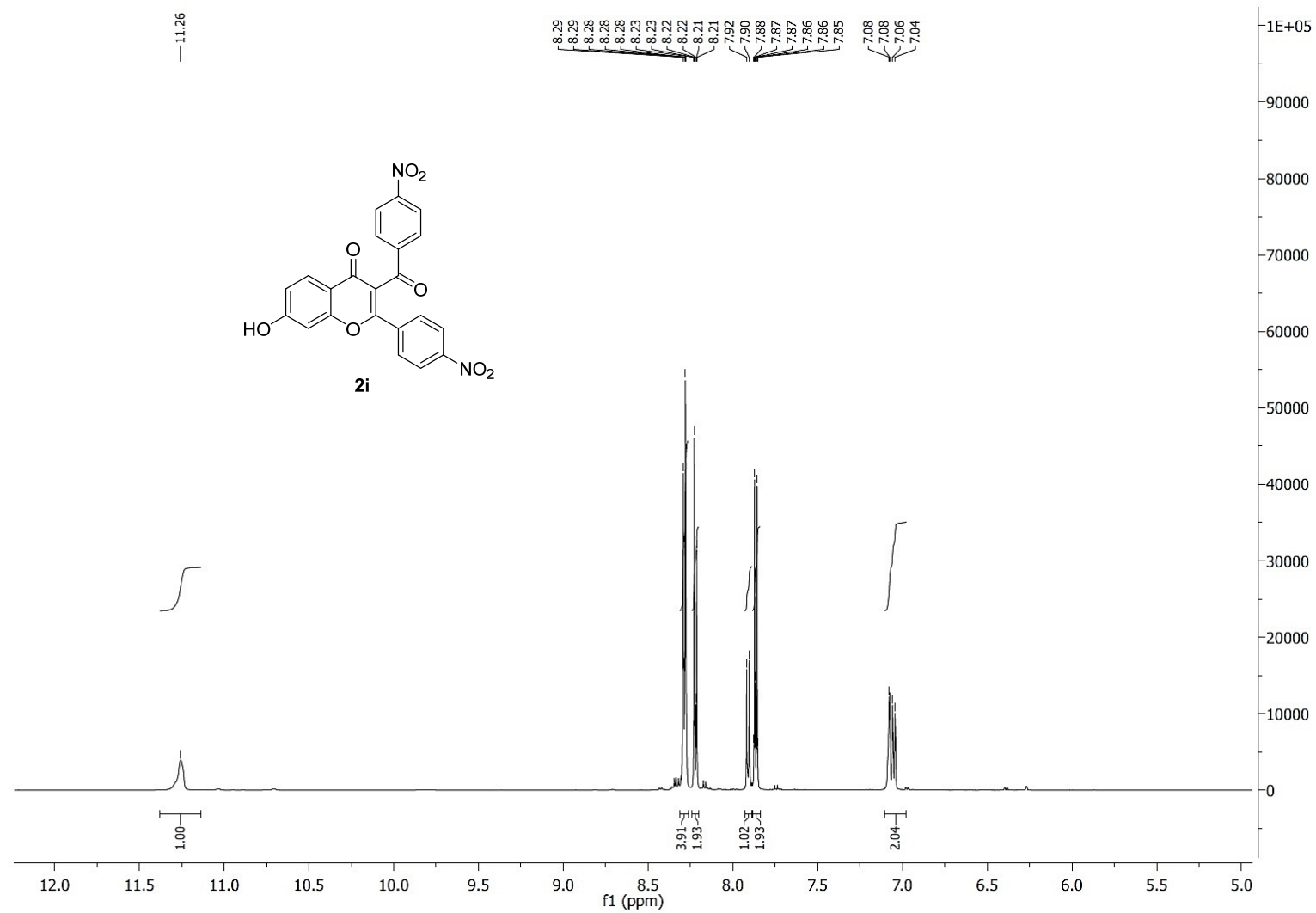


Figure S9 ^1H -NMR spectrum of compound **2i**

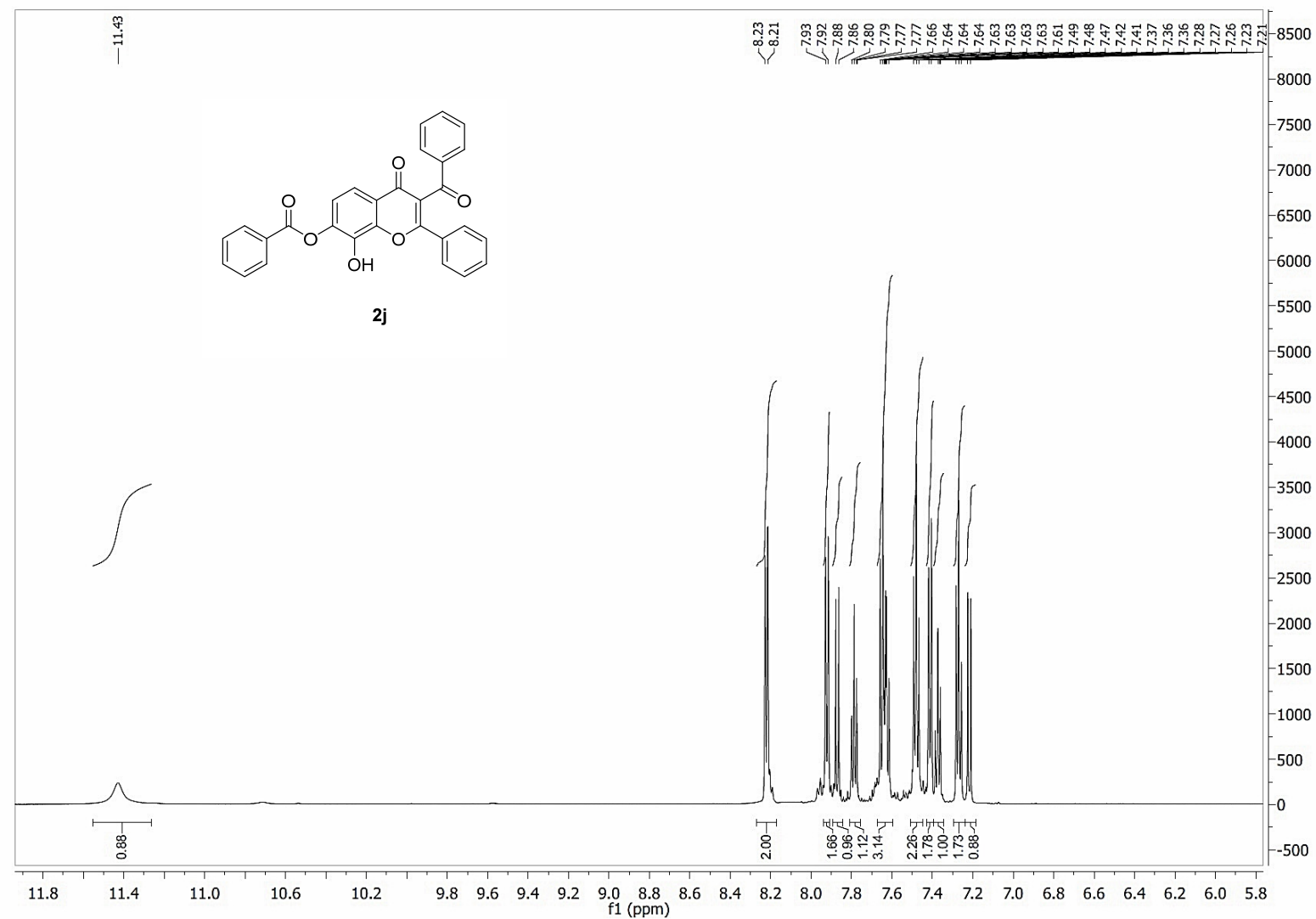


Figure S10 ^1H -NMR spectrum of compound **2j**

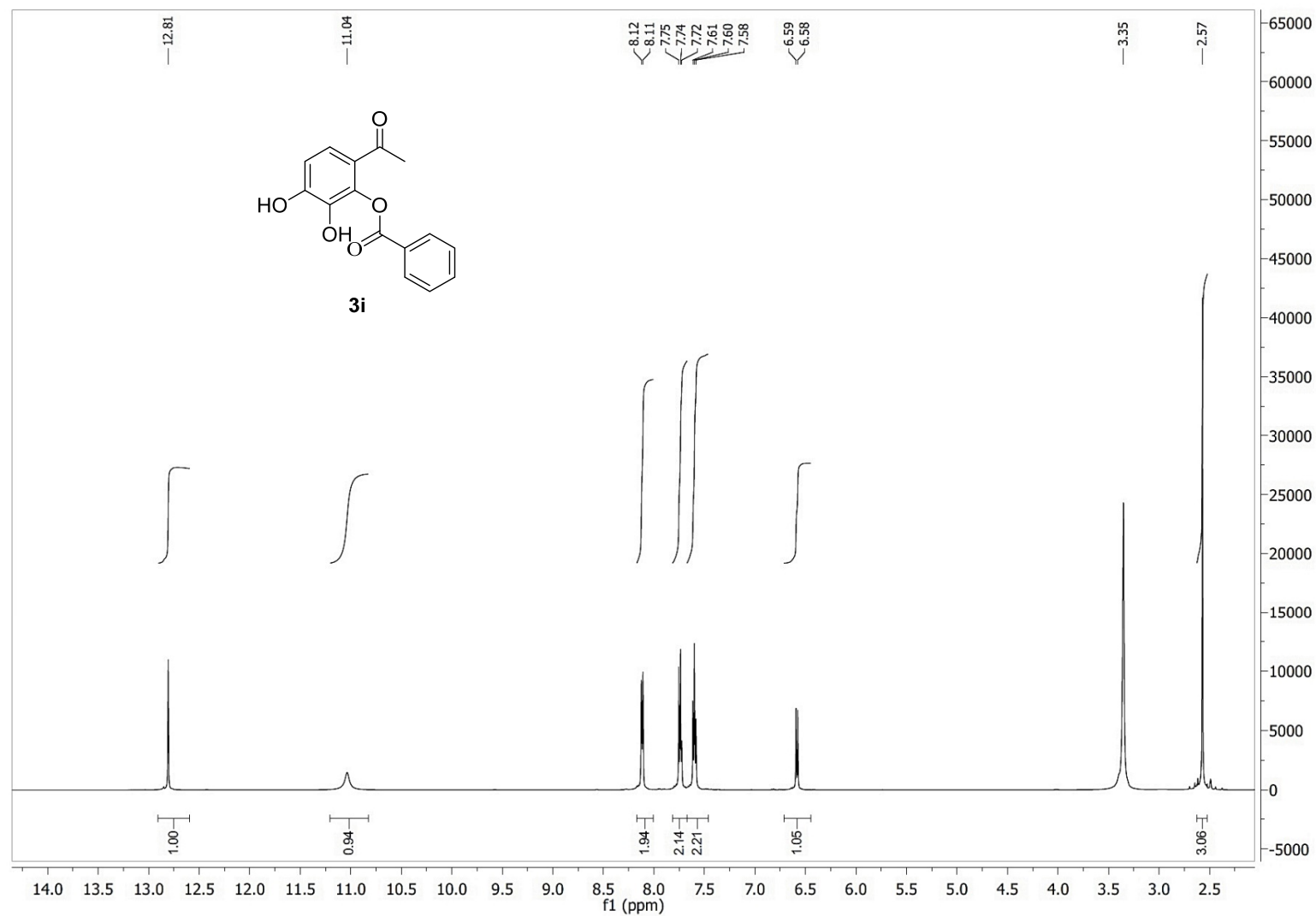


Figure S11 ¹H-NMR spectrum of compound **3i**

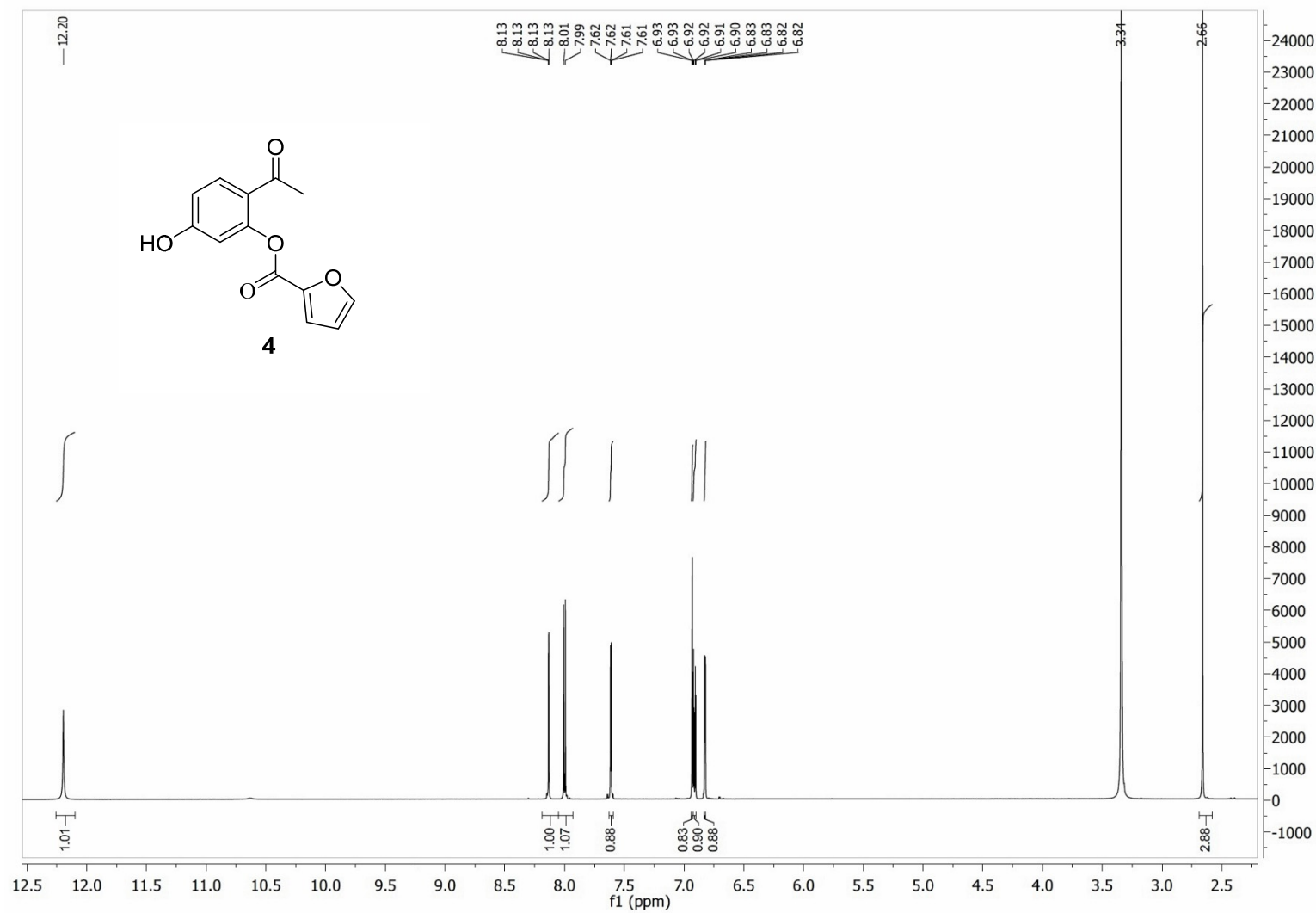


Figure S12 ^1H -NMR spectrum of compound 4

3. ^{13}C -NMR spectrum of synthetic compounds

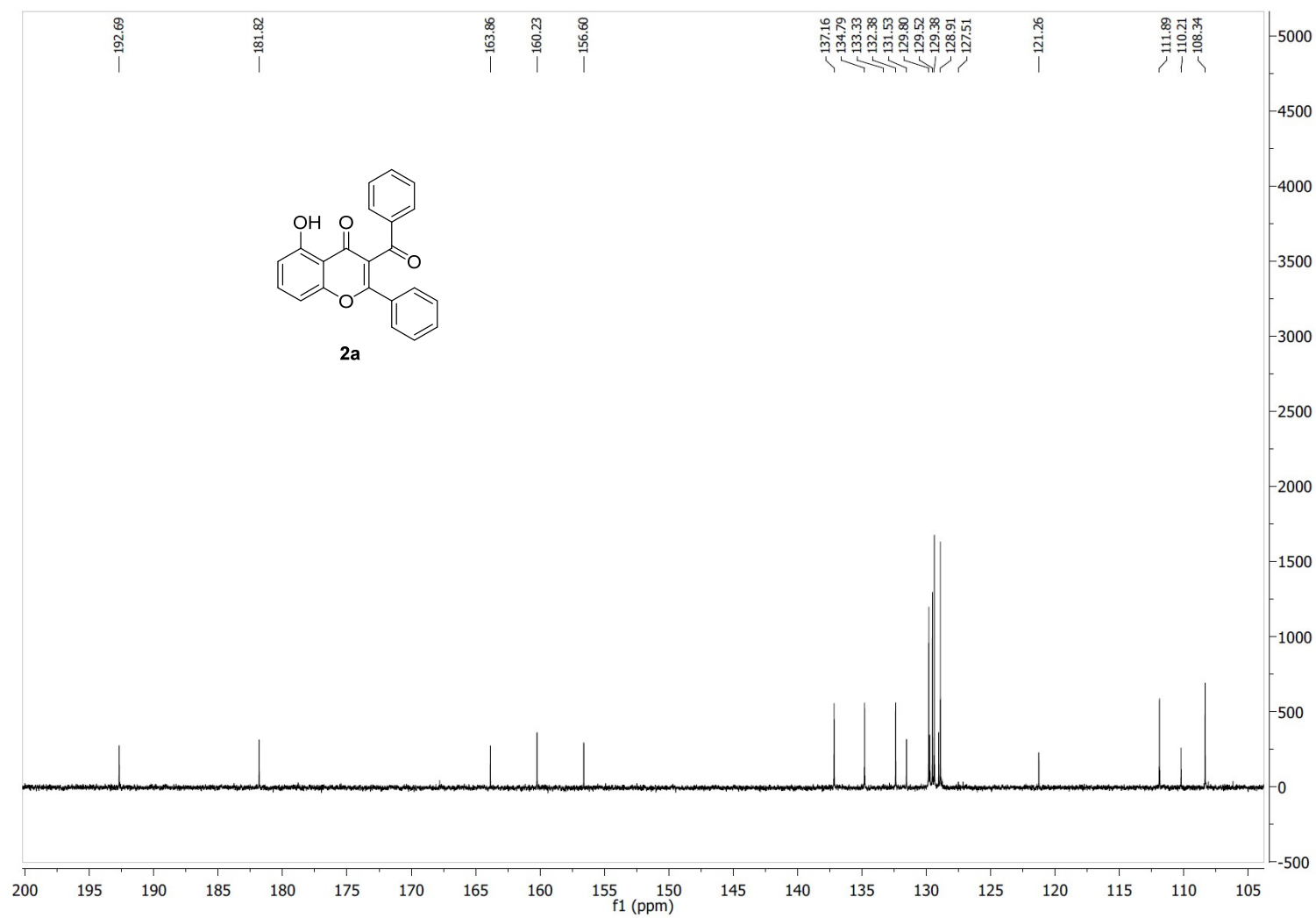


Figure S13 ^{13}C -NMR spectrum of compound **2a**

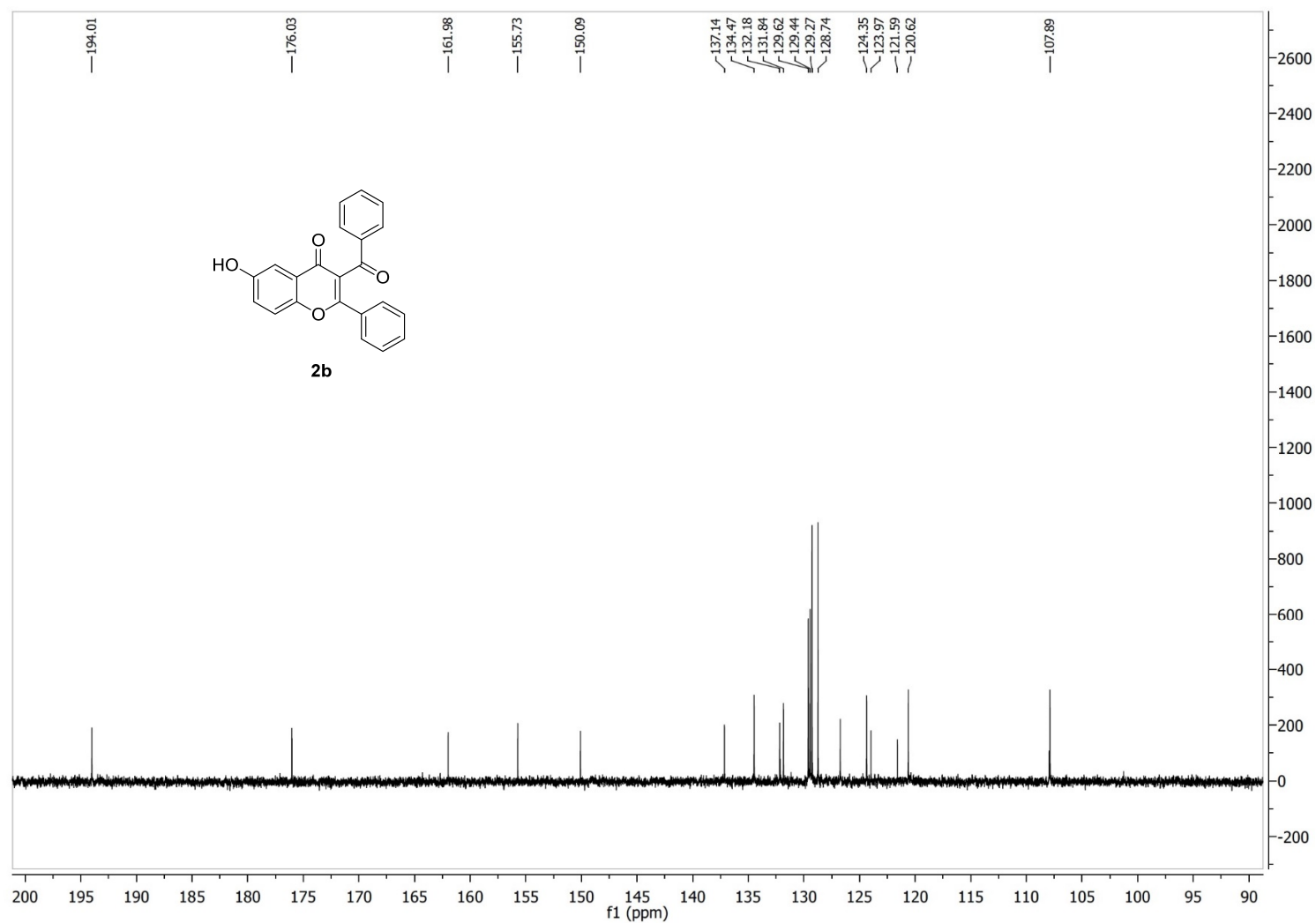


Figure S14 ^{13}C -NMR spectrum of compound **2b**

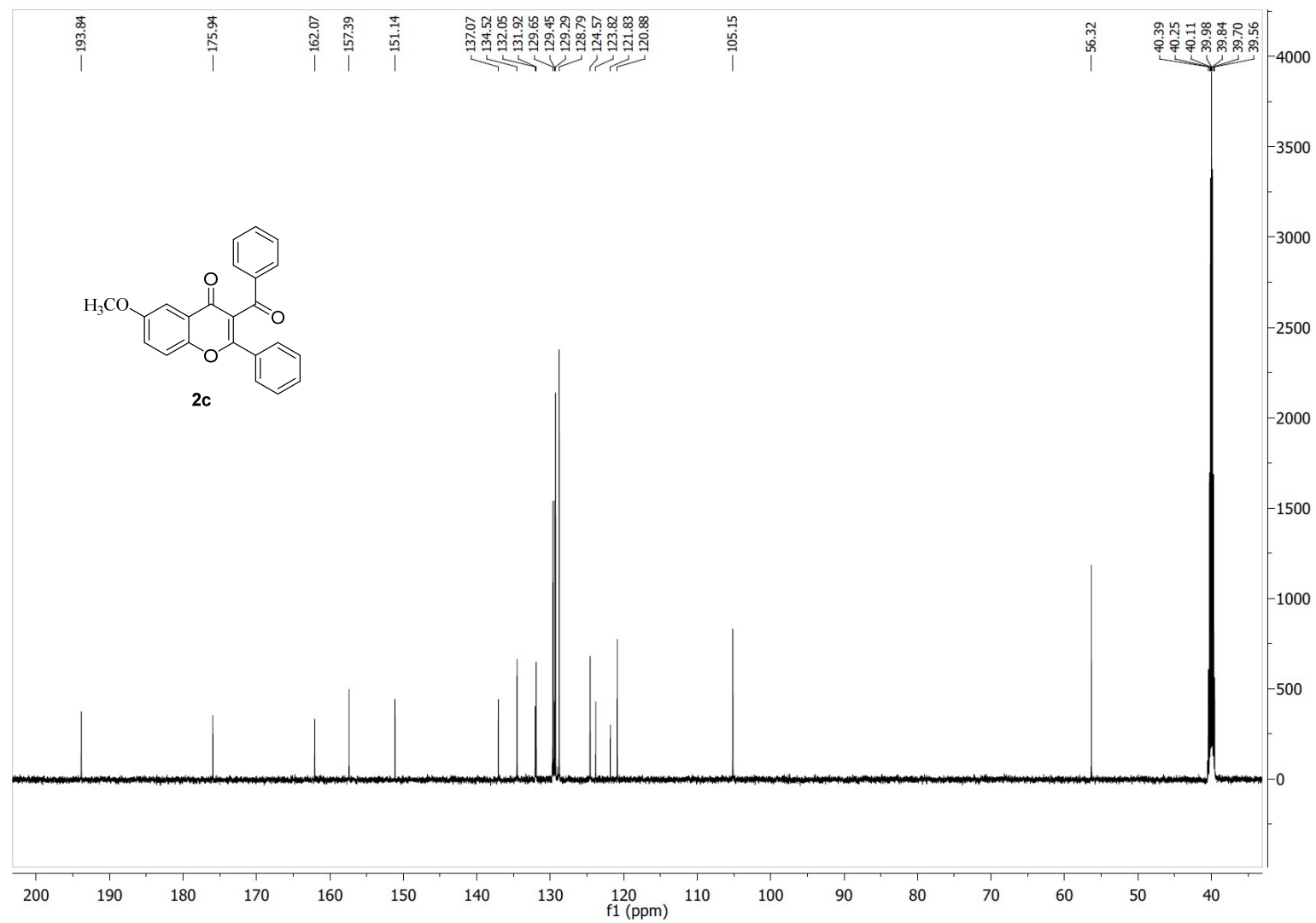


Figure S15 ^{13}C -NMR spectrum of compound **2c**

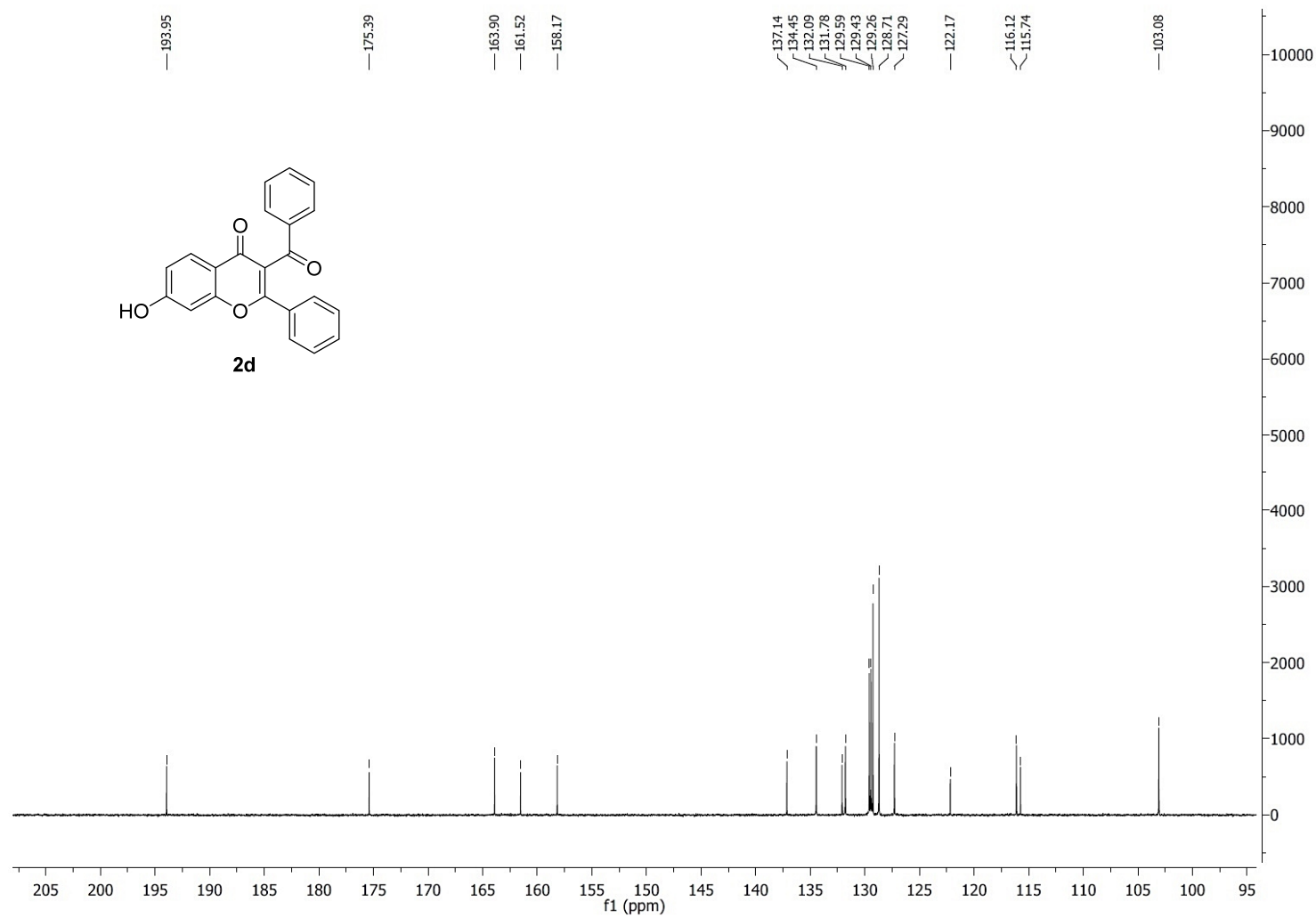


Figure S16 ^{13}C -NMR spectrum of compound **2d**

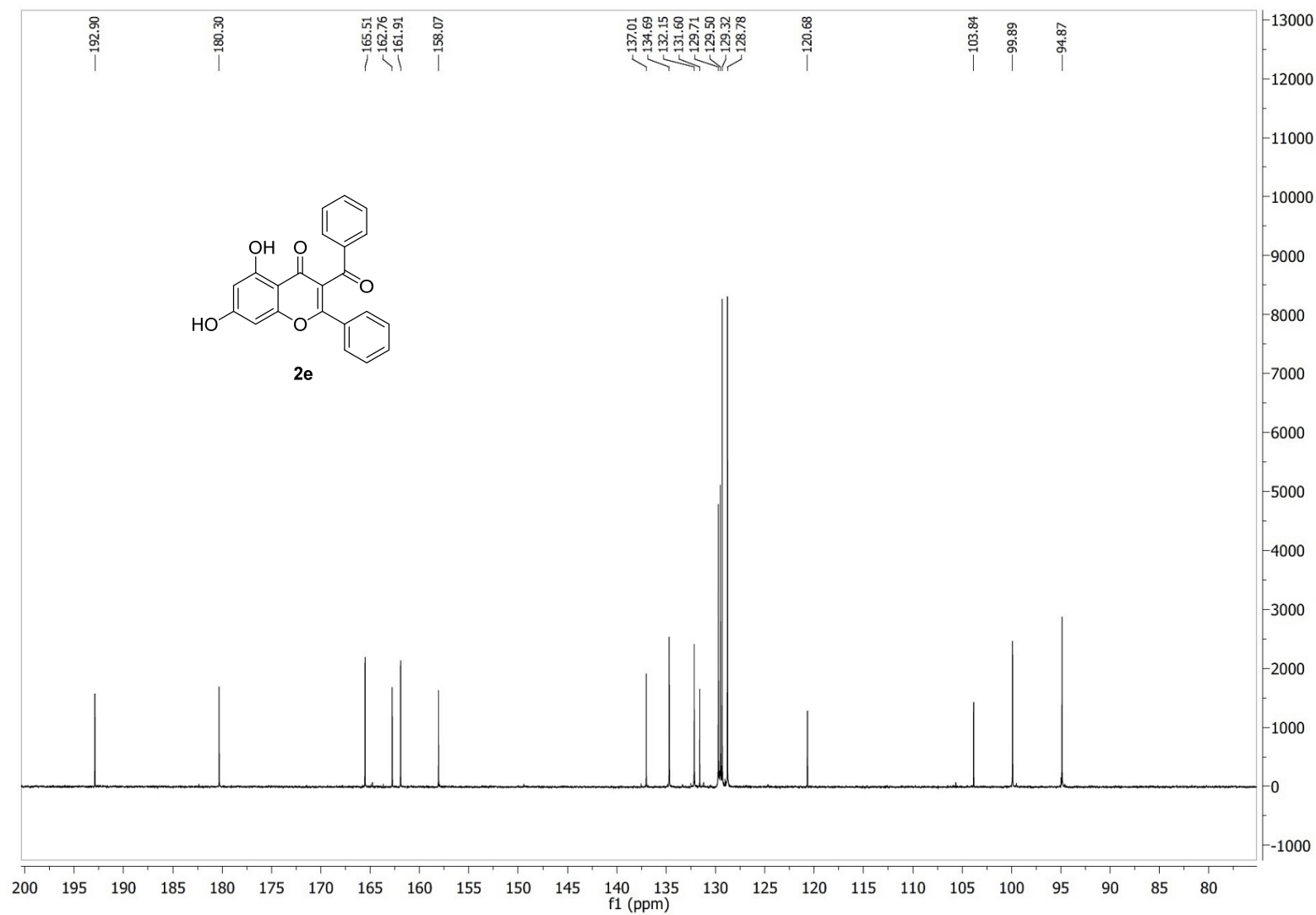


Figure S17 ^{13}C -NMR spectrum of compound **2e**

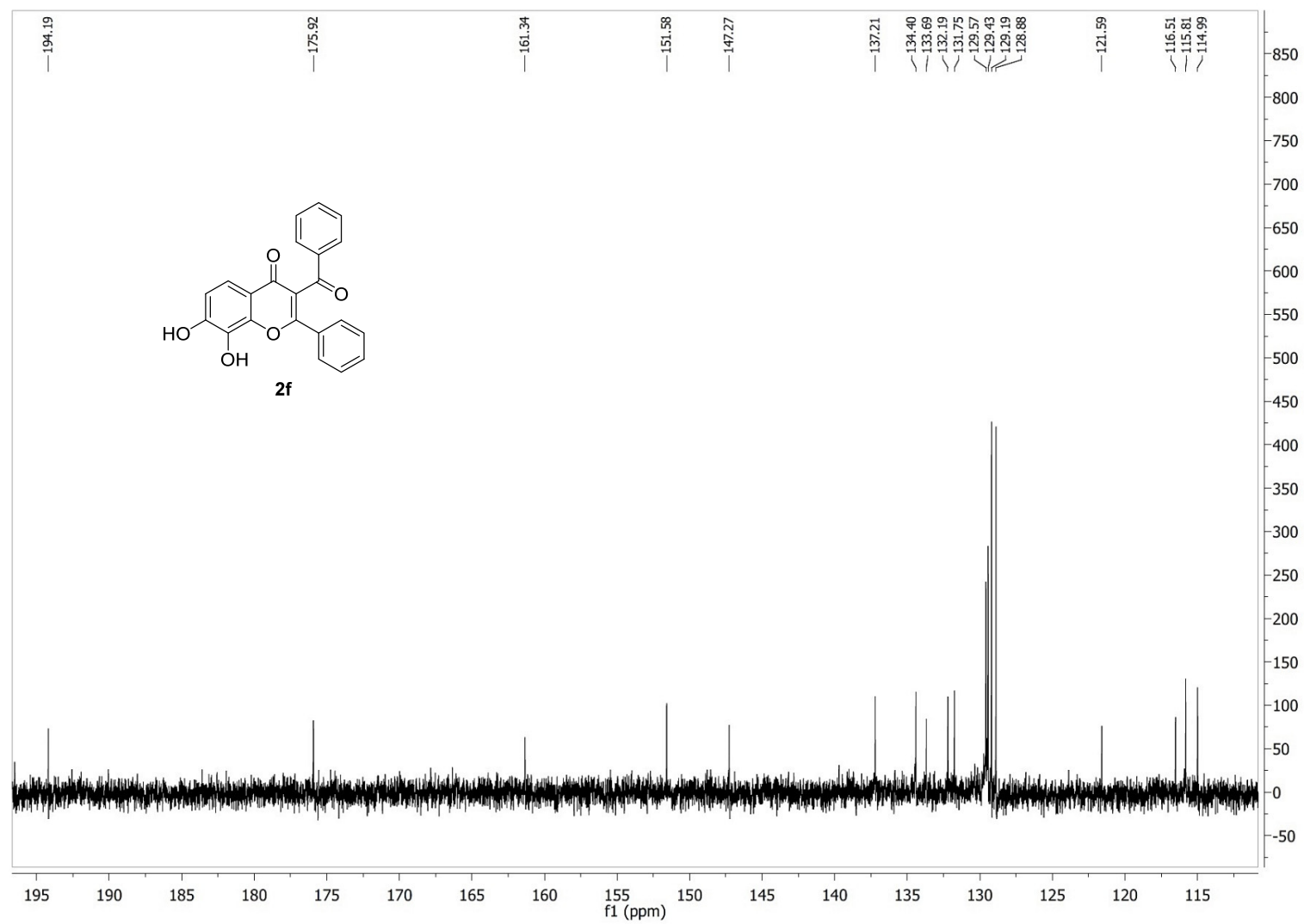


Figure S18 ^{13}C -NMR spectrum of compound **2f**

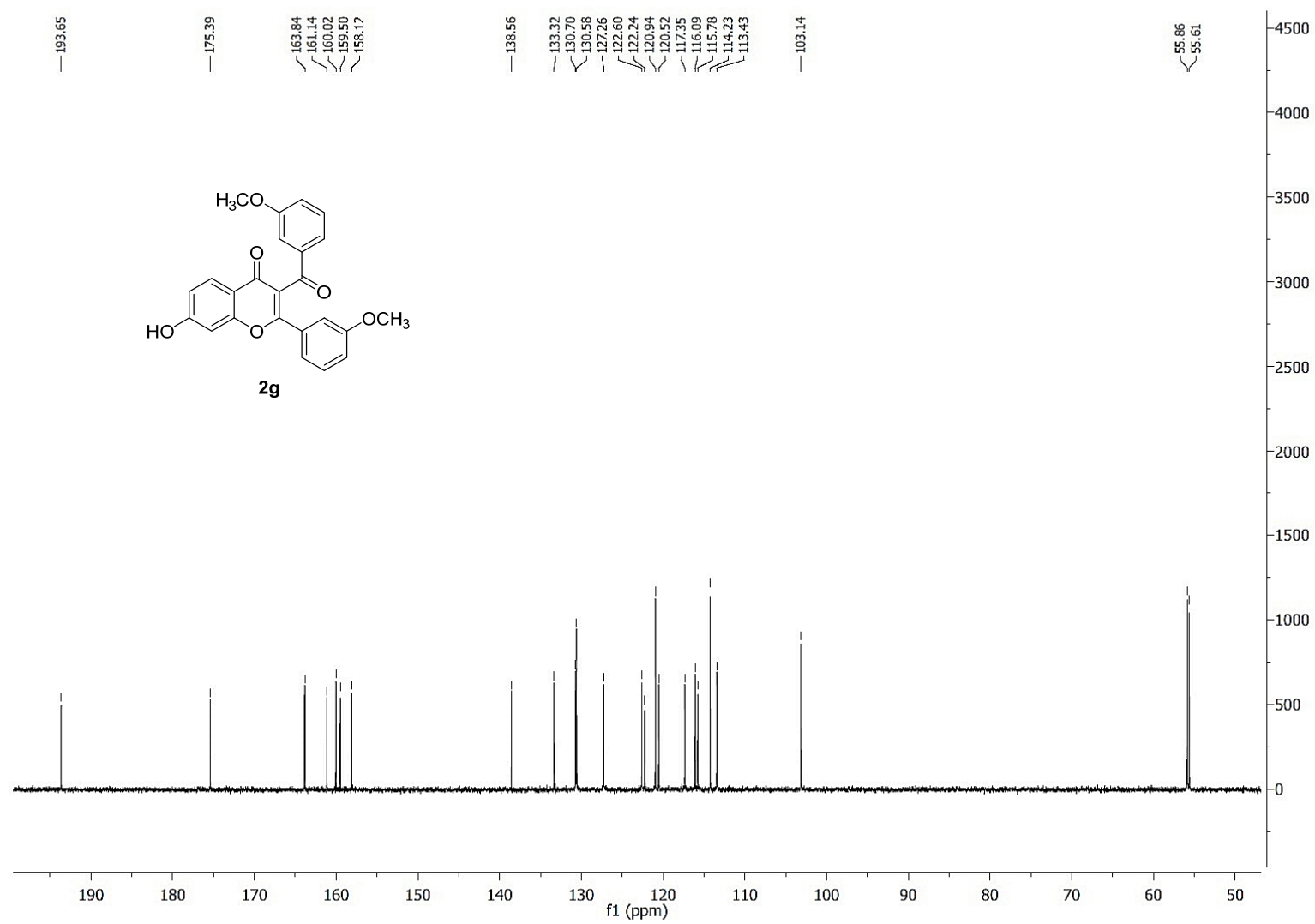


Figure S19 ¹³C-NMR spectrum of compound **2g**

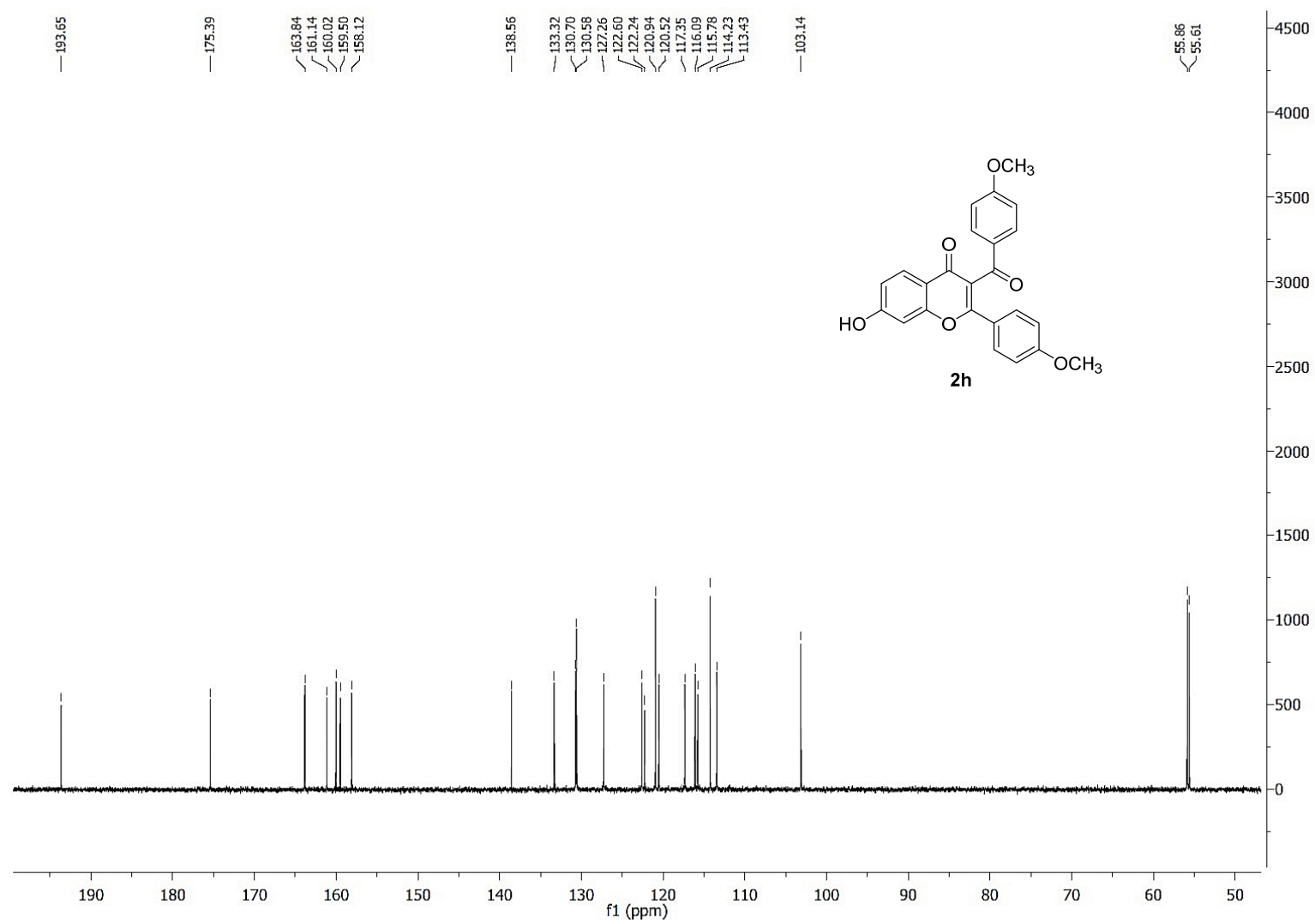


Figure S20 ^{13}C -NMR spectrum of compound **2h**

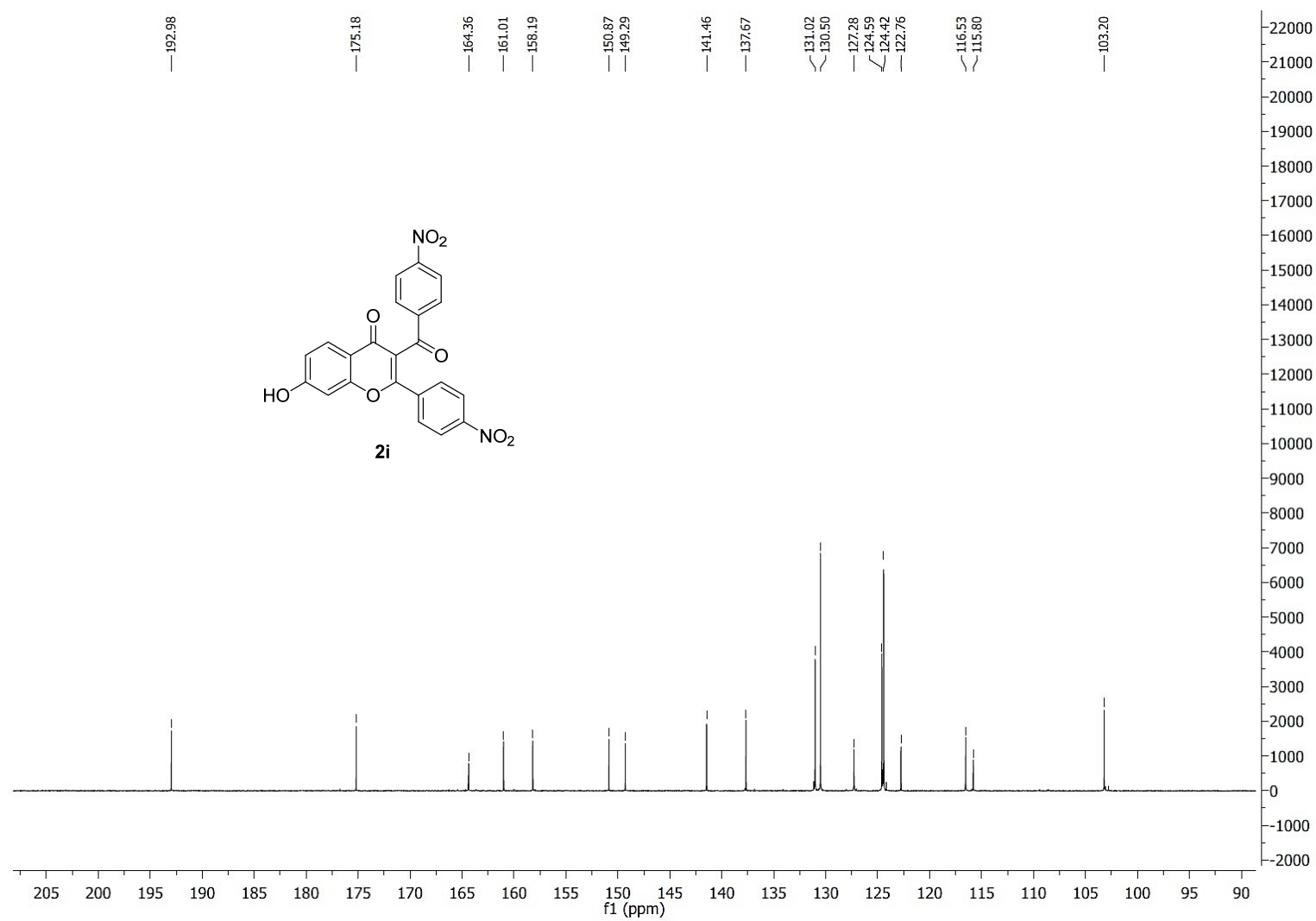


Figure S21 ^{13}C -NMR spectrum of compound **2i**

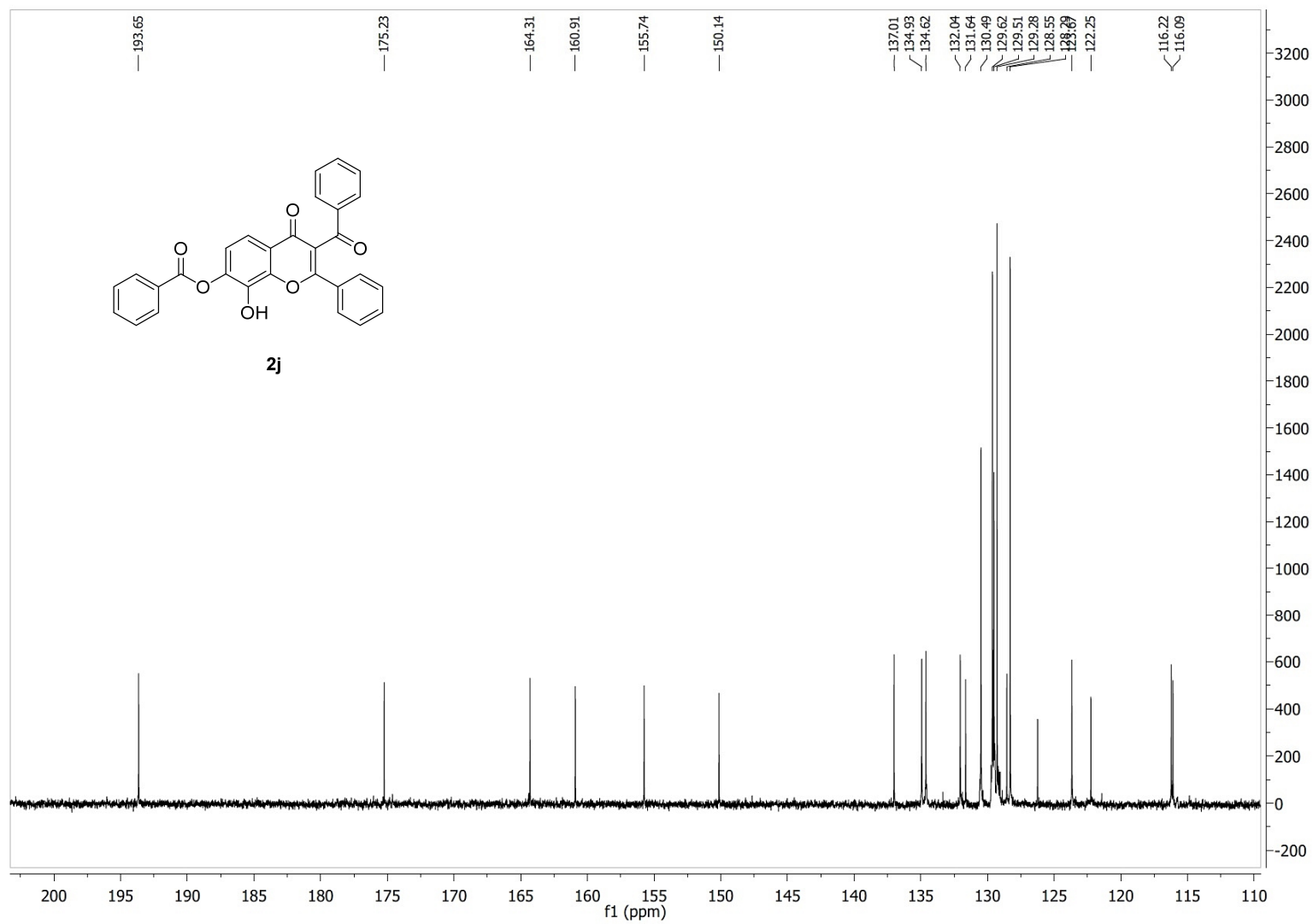


Figure S22 ^{13}C -NMR spectrum of compound **2j**

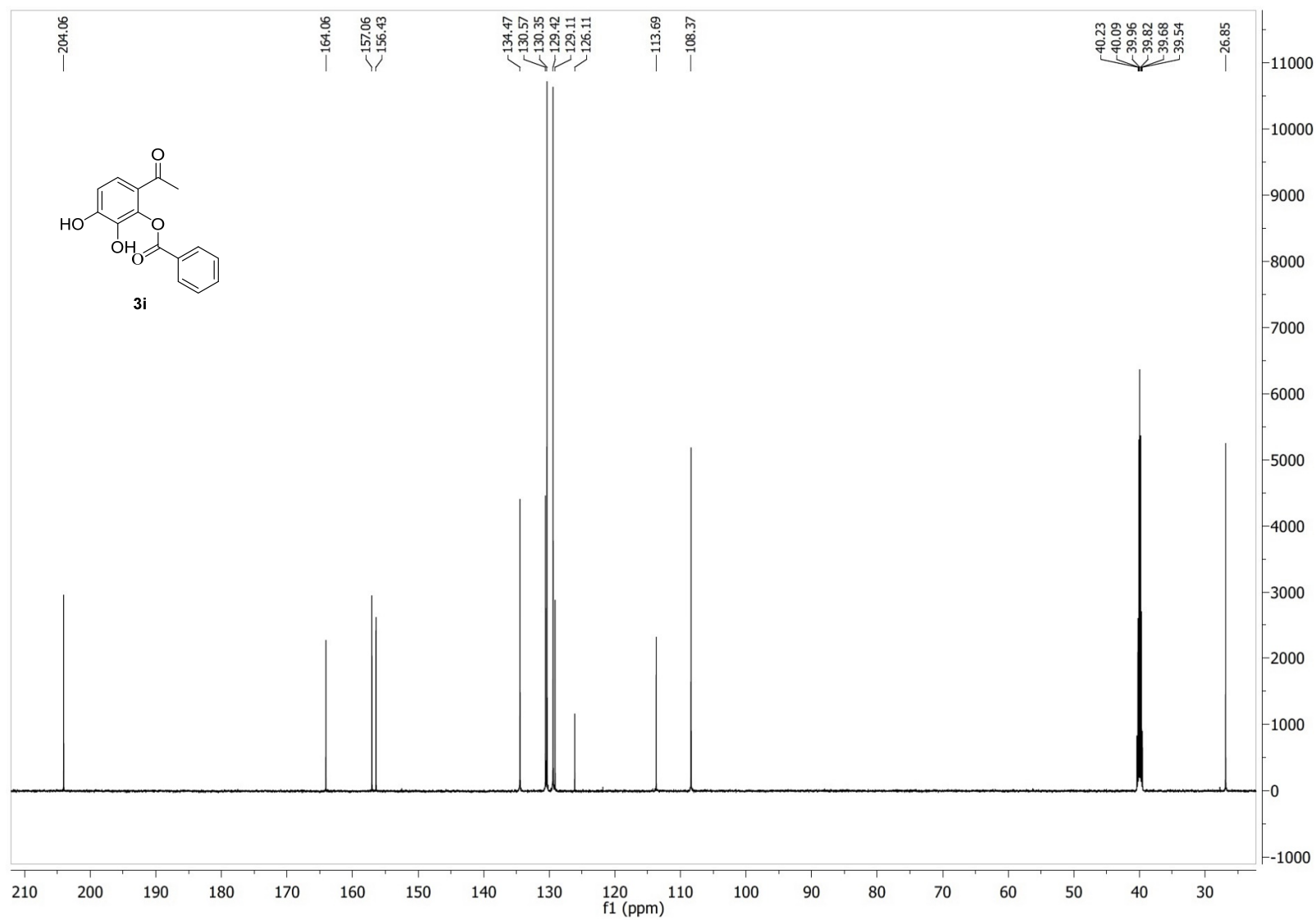


Figure S23 ¹³C-NMR spectrum of compound **3i**

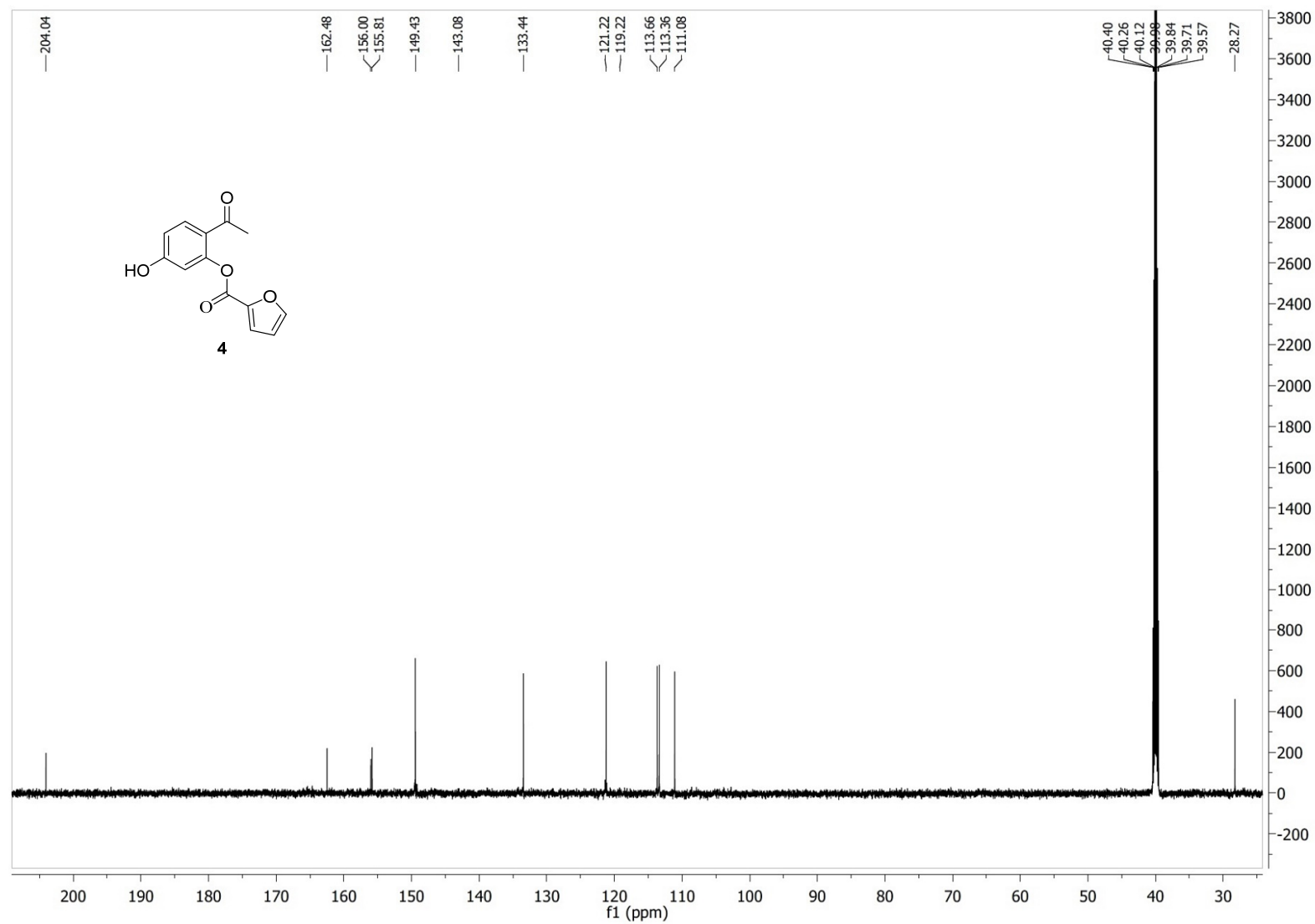


Figure S24 ^{13}C -NMR spectrum of compound 4

4. LCMS spectrum of synthetic compounds

Mass spectroscopy condition

The LC–ESI–MS/QTOF-MS system consists of HPLC unit 1260 infinity series (Agilent Technologies, Waldbronn, Germany) coupled with an ultra-high-definition accurate mass spectrometer (Agilent Technologies, Singapore). One microliter was injected to the system at a flow rate of 0.5 mL/min. The column temperature was maintained at 35 °C. The mobile phase consisted of A (0.1% formic acid in type I water, v/v) and B (0.1% formic acid in acetonitrile, v/v). The isocratic elution was 50% of mobile phase A. The dual electrospray ionization (ESI) source was operated in positive modes. The ESI condition was as follows: drying gas (gas N₂) temperature, 350 °C; gas flow rate, 10 L/min; nebulizer pressure, 30 psig; mass range, 200–800m/z; scan rate, 4 spectra/s; capillary voltage, 3500 V; skimmer voltage, 65 V; octapole RFV, 750 V; and fragment voltage, 50 V.

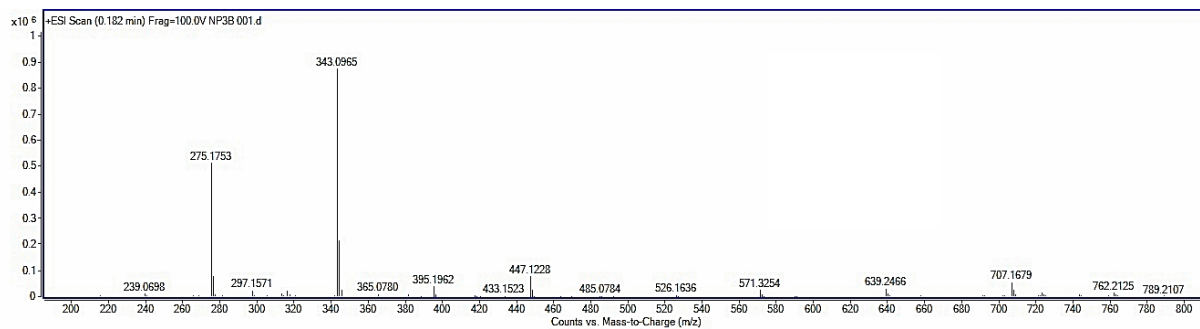


Figure S25 Mass spectrum (MS-ESI) of compound **2a**

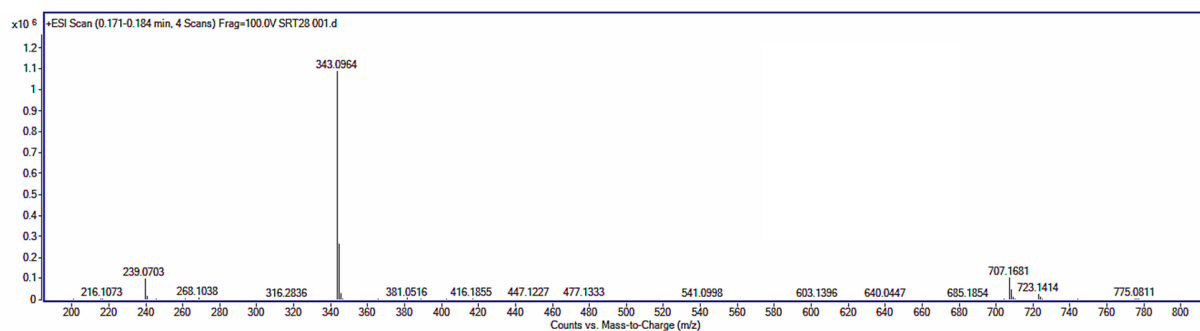


Figure S26 Mass spectrum (MS-ESI) of compound **2b**

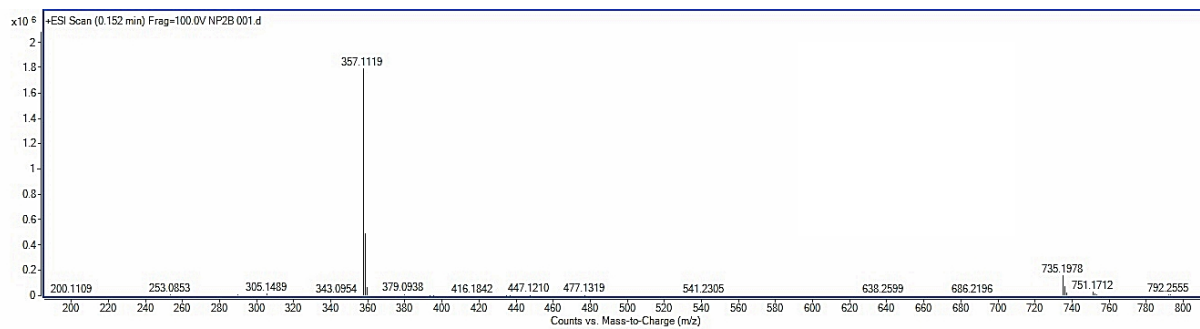


Figure S27 Mass spectrum (MS-ESI) of compound **2c**

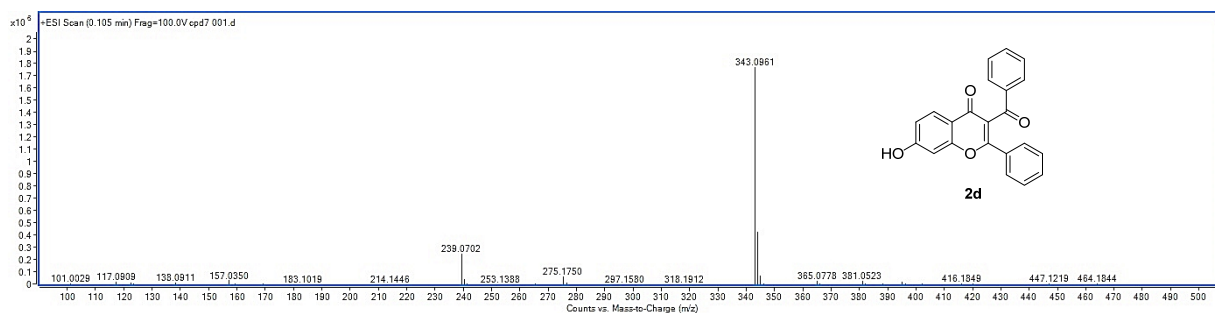


Figure S28 Mass spectrum (MS-ESI) of compound **2d**

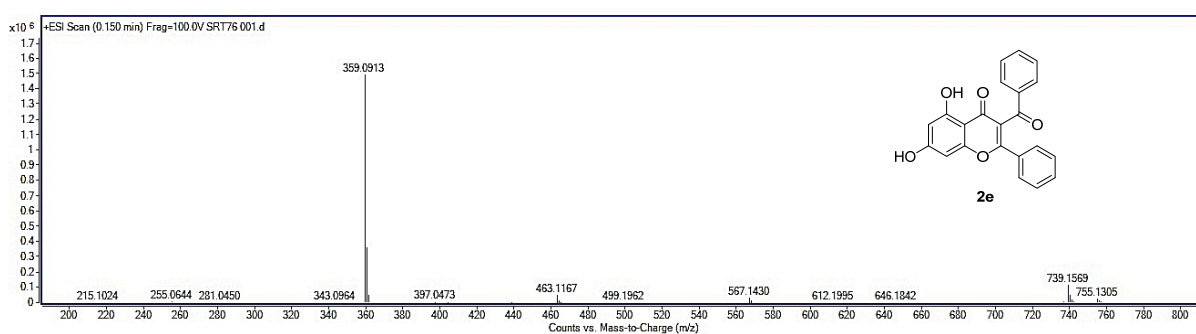


Figure S29 Mass spectrum (MS-ESI) of compound **2e**

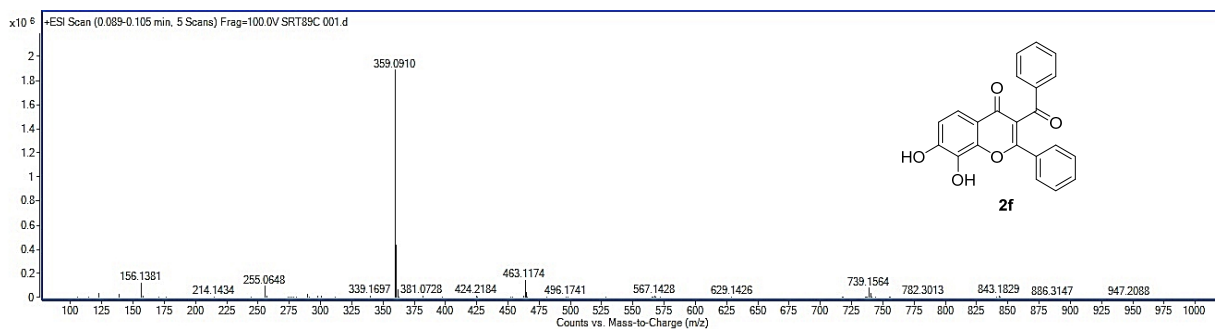


Figure S30 Mass spectrum (MS-ESI) of compound **2f**

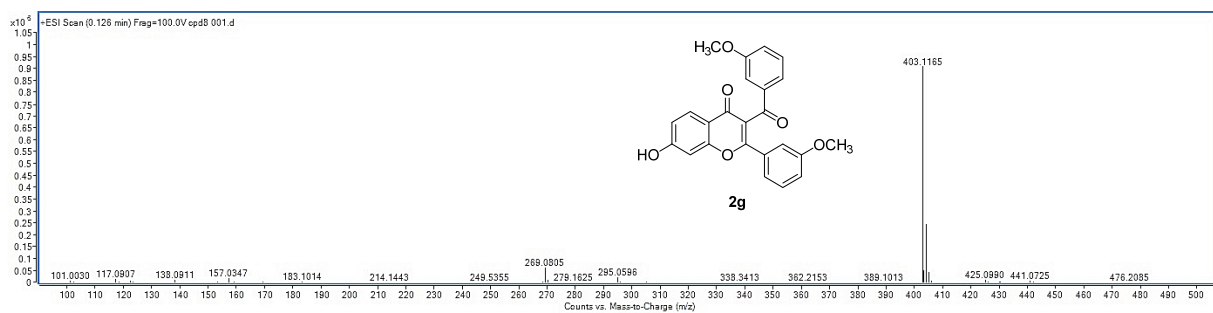


Figure S31 Mass spectrum (MS-ESI) of compound **2g**

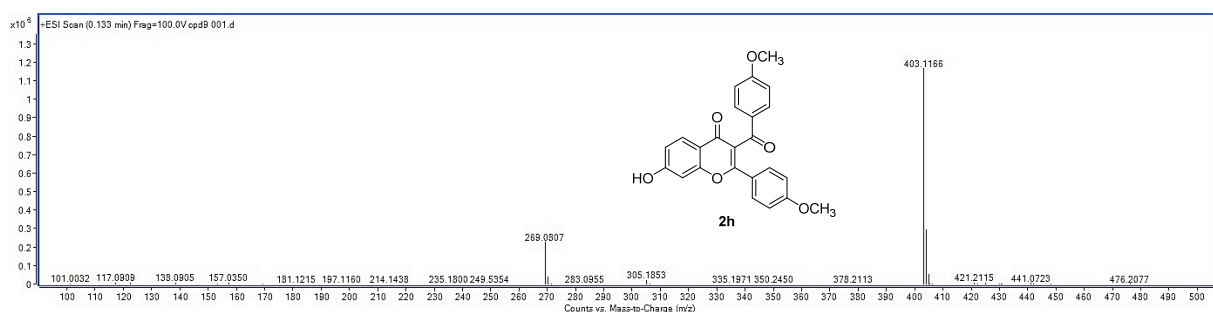


Figure S32 Mass spectrum (MS-ESI) of compound **2h**

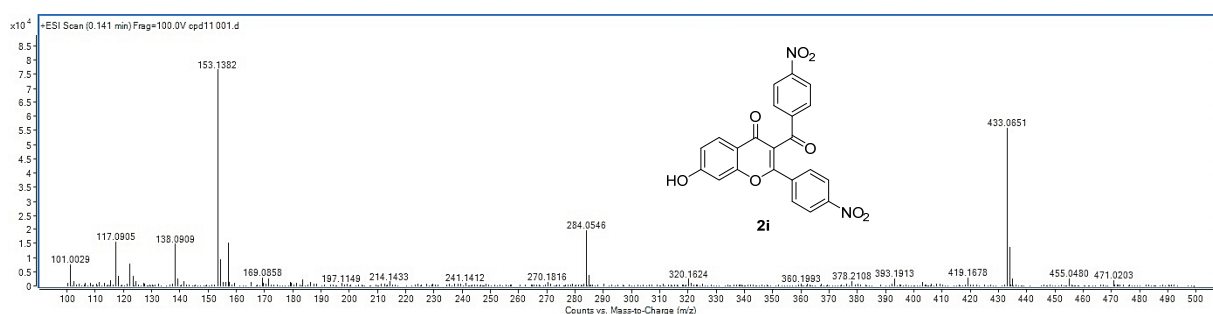


Figure S33 Mass spectrum (MS-ESI) of compound **2i**

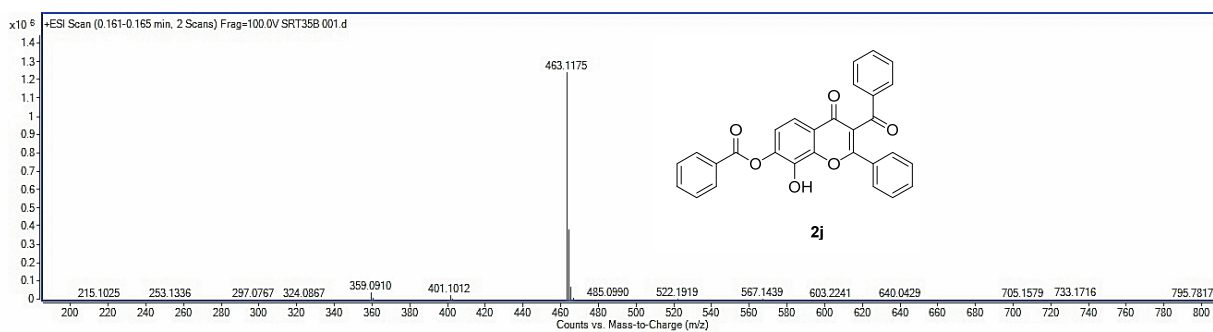


Figure S34 Mass spectrum (MS-ESI) of compound **2j**

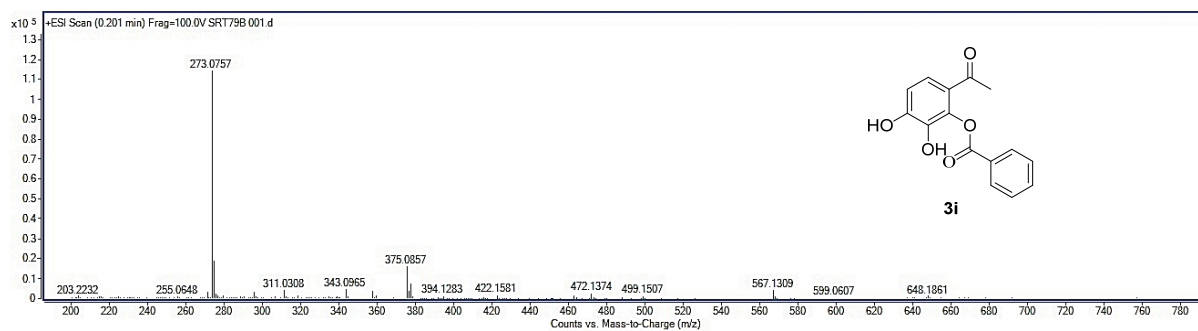


Figure S35 Mass spectrum (MS-ESI) of compound **3i**

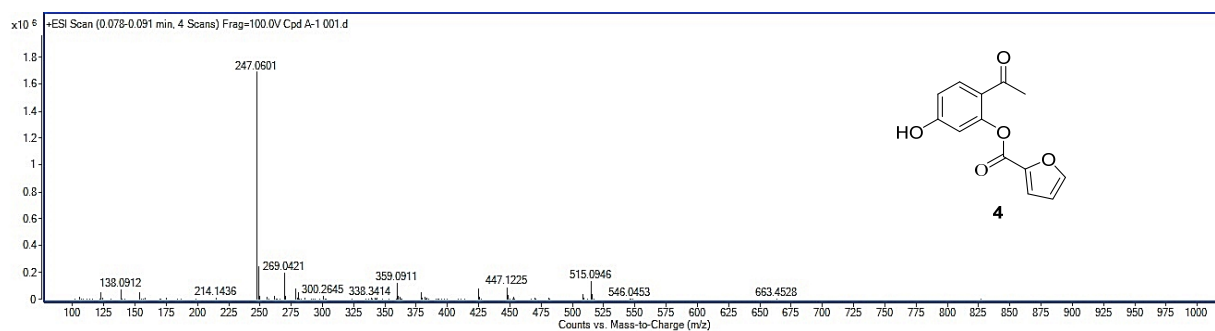


Figure S36 Mass spectrum (MS-ESI) of compound **4**