Electronic Supplementary Material (ESI) for RSC Advances.

Supporting Information

A Green Catalystless Electrochemical Method for the Synthesis of Dicoumarols. Investigation of Electro-chemically Induced Cross-Dehydrogenative Coupling Reaction

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Contents

Apparatus and Reagents

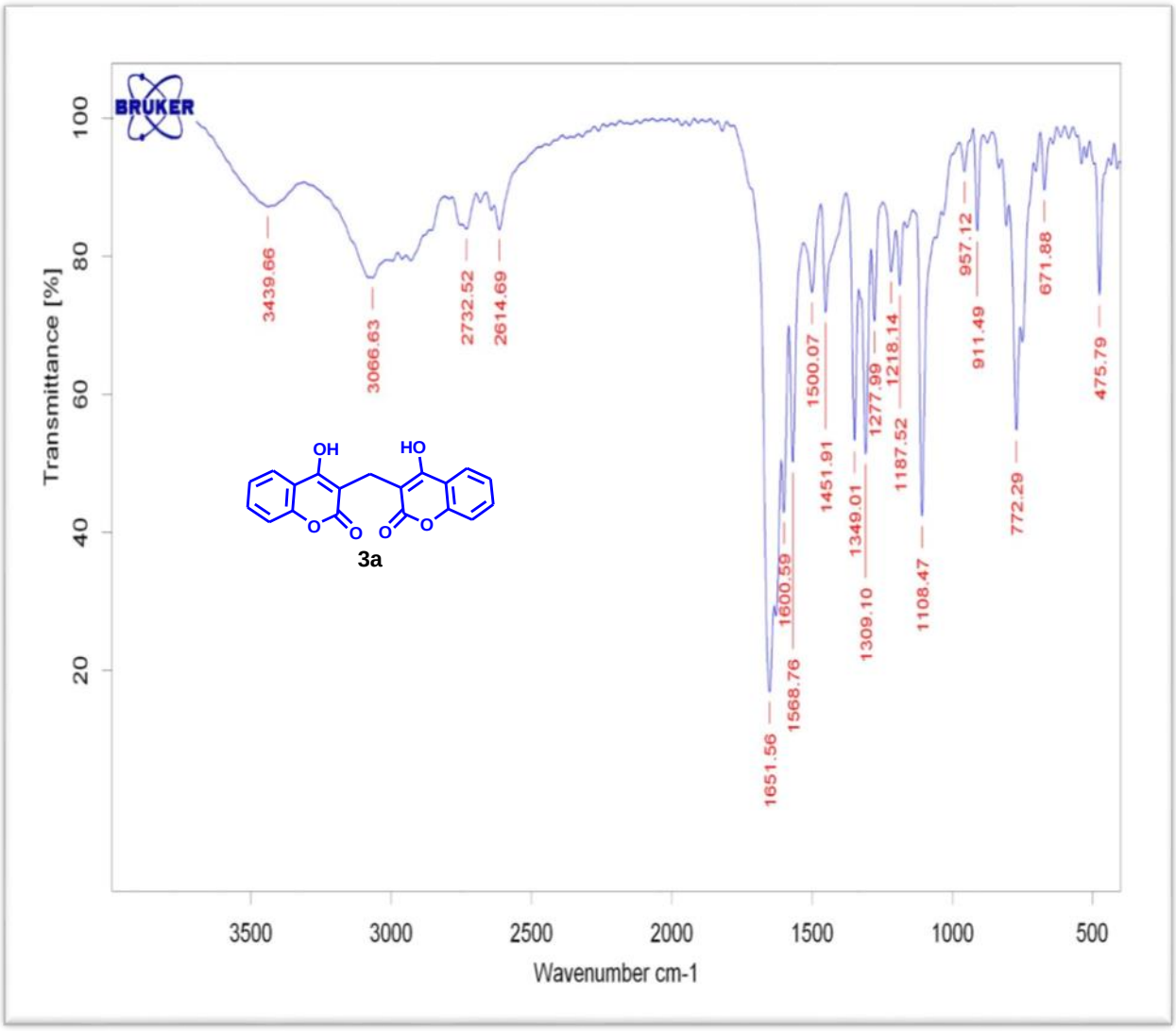
Cyclic voltammetry, controlled-potential coulometry and preparative electrolysis were performed using an Autolab model PGSTAT 30 potentiostat/galvanostat. The working electrode used in the voltammetry experiments was a glassy carbon disc (1.8 mm^2 area) and platinum wire was used as counter electrode. The working electrode used in controlled-potential coulometry and macroscale electrolysis was carbon plate (148 cm^2) and large steel gauze constitutes the counter electrode. The working electrode potential was measured versus Ag/AgCl (electrode from METROHM).

The products were characterized by Nuclear Magnetic Resonance, Fourier Transform Infrared Spectrometry, Mass Spectroscopy and Elemental analysis.

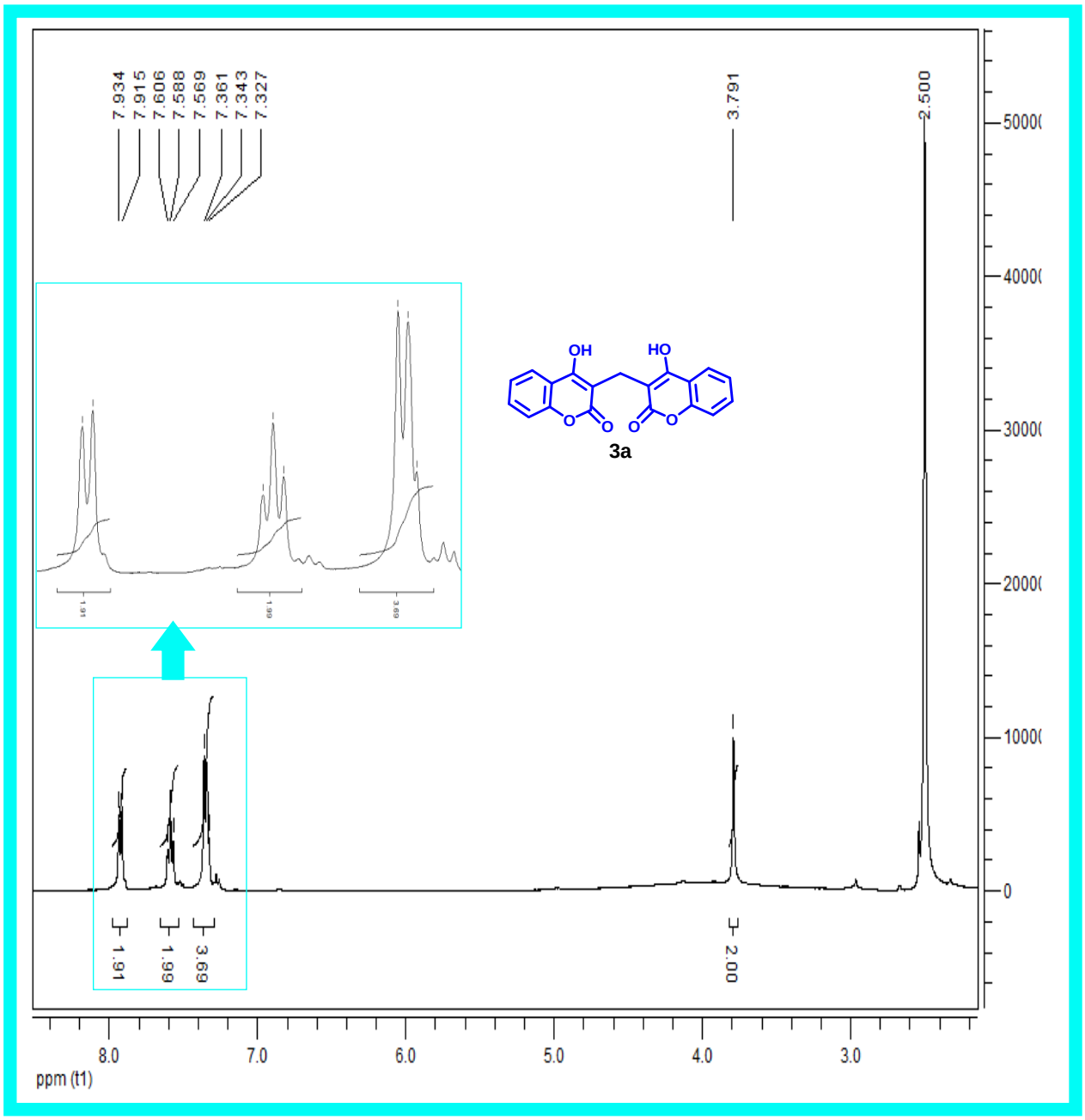
MS spectra were obtained by an Agilent Technology (HP) Mass Spectrometer, 5973 Network Mass Selective Detector model. The following parameters were employed for MS analysis in our study: ion Electron Impact (EI) 70eV and Ion source temperature 230ºC. ^{1}H NMR and ^{13}C NMR were performed using 400 MHz and 100 MHz Bruker instruments. Peak listings for all NMR spectra are given in ppm and referenced against the solvent (DMSO-d6) residual signal. FTIR analysis was done using VERTEX Bruker instrument. Spectra were collected by KBr pellet.

N,N,N',N'-tetramethyl-1,4-phenylenediamine, 4-hydroxycoumarin, 4-hydroxy-6-methyl-coumarin, 4-hydroxy-6-methylpyron, ethanol, phosphoric acid and phosphate salts were reagent-grade materials and obtained from commercial sources. These chemicals were used without further purification.

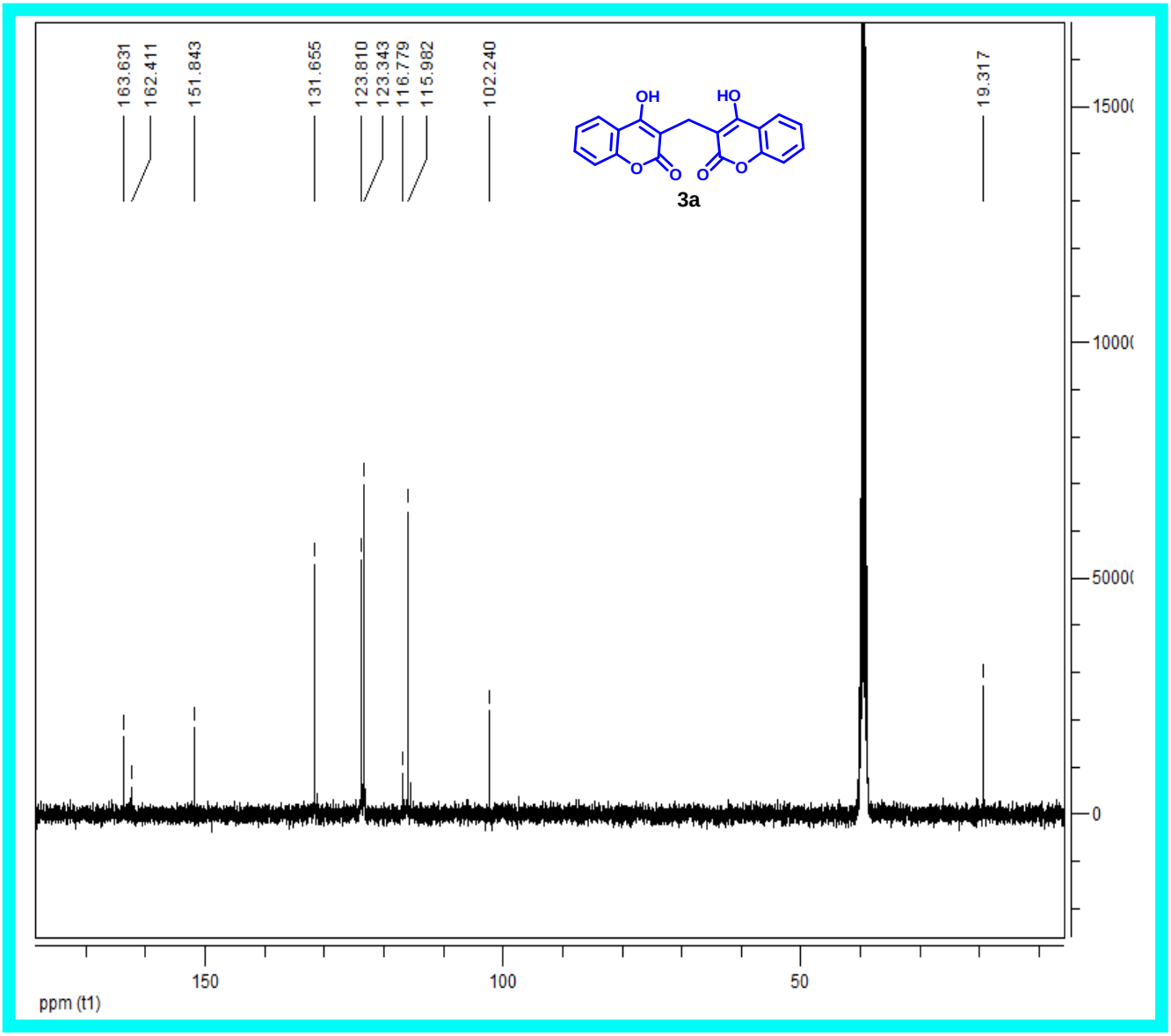
FTIR spectrum of **3a**



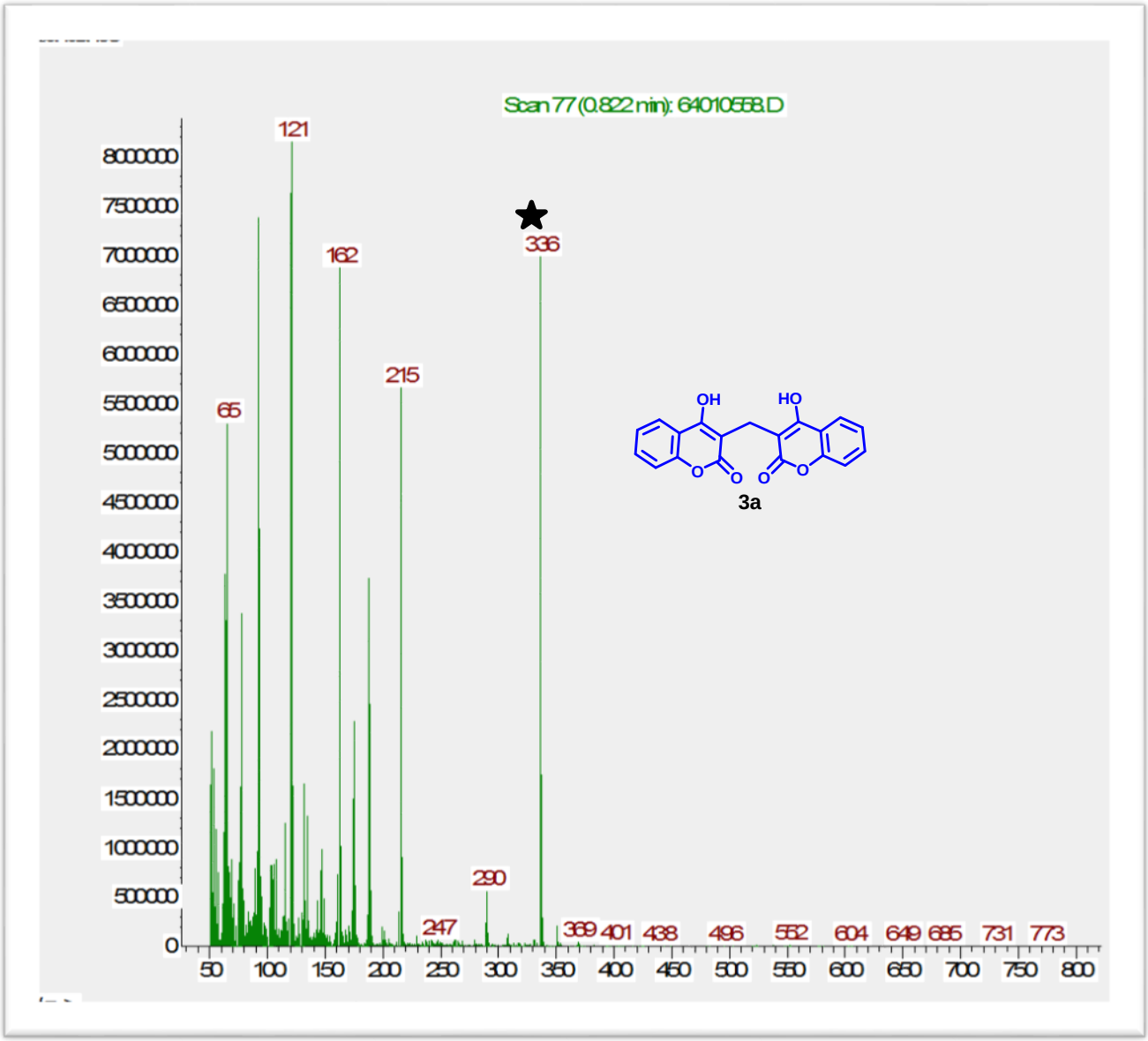
[1]H NMR spectrum of **3a**



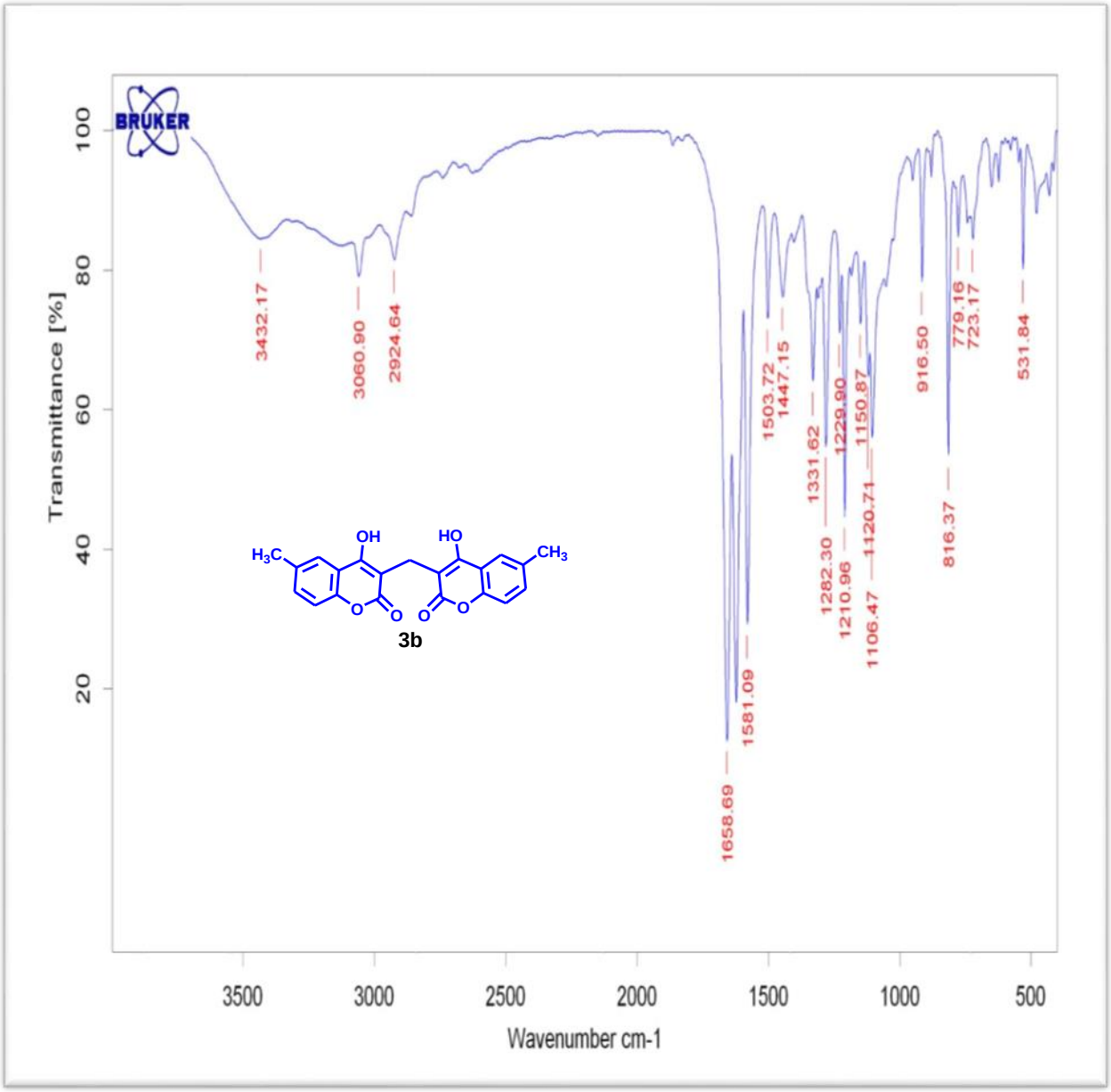
^{13}C NMR spectrum of **3a**



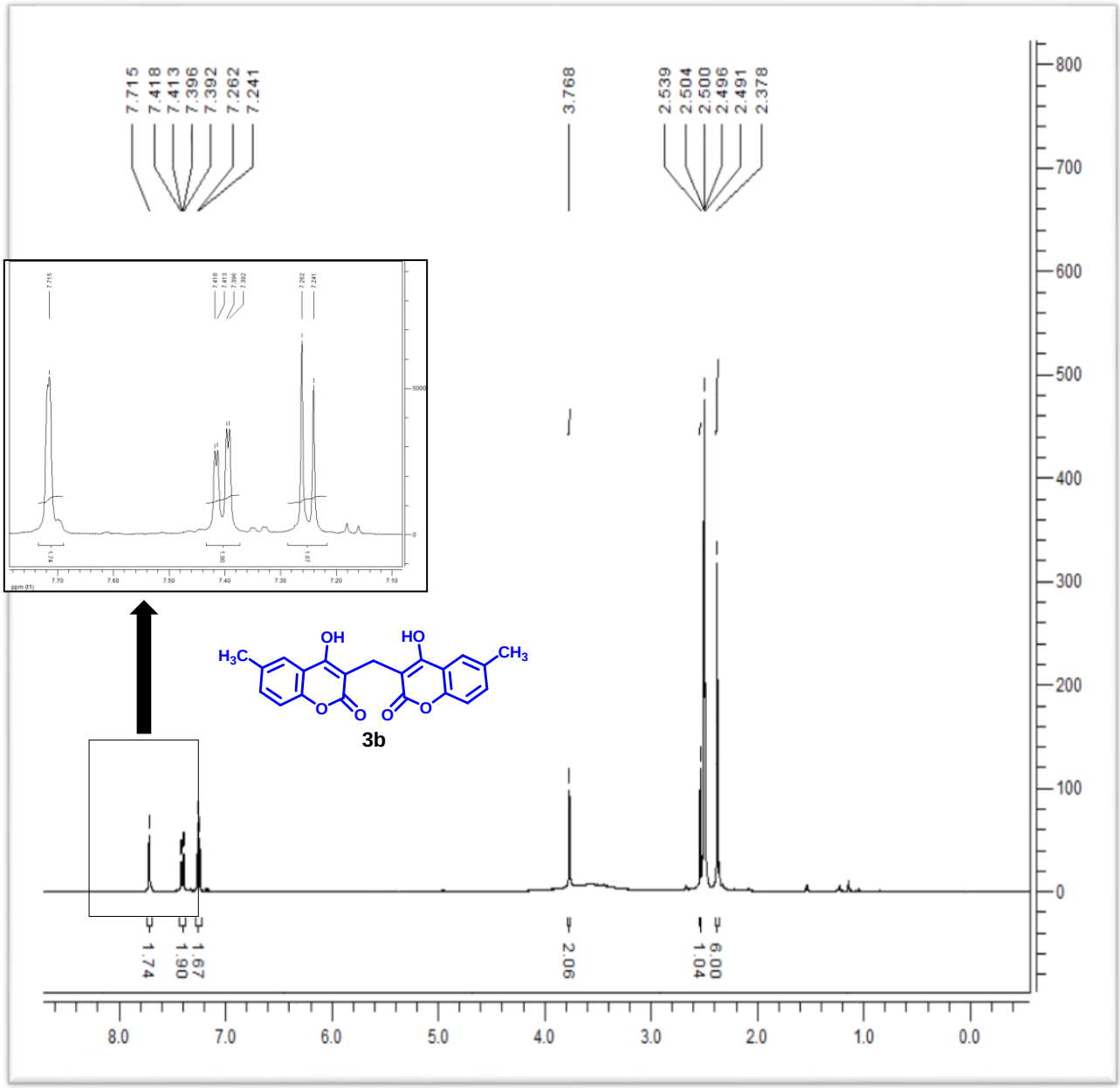
MS spectrum of **3a**



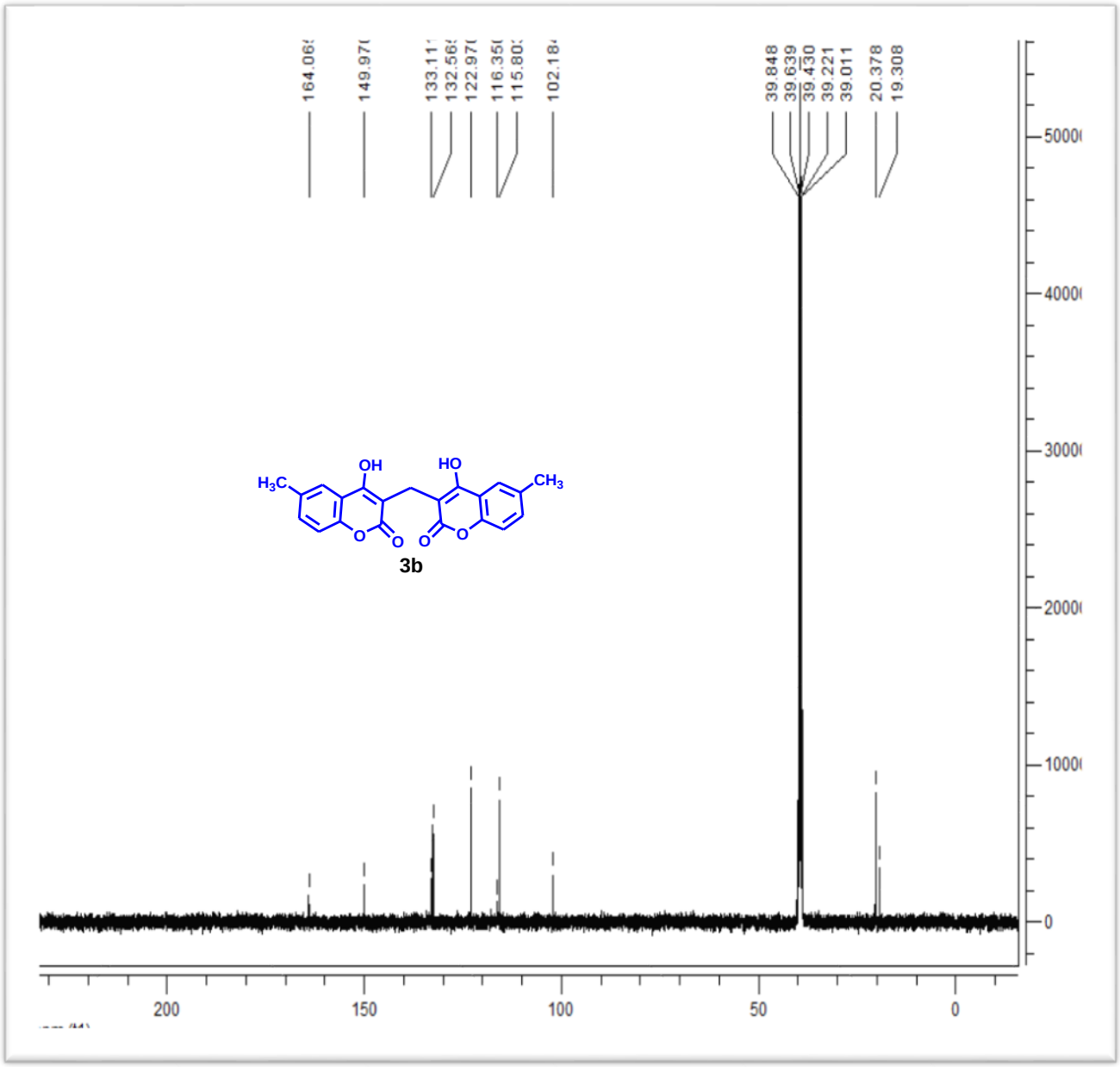
FTIR spectrum of **3b**



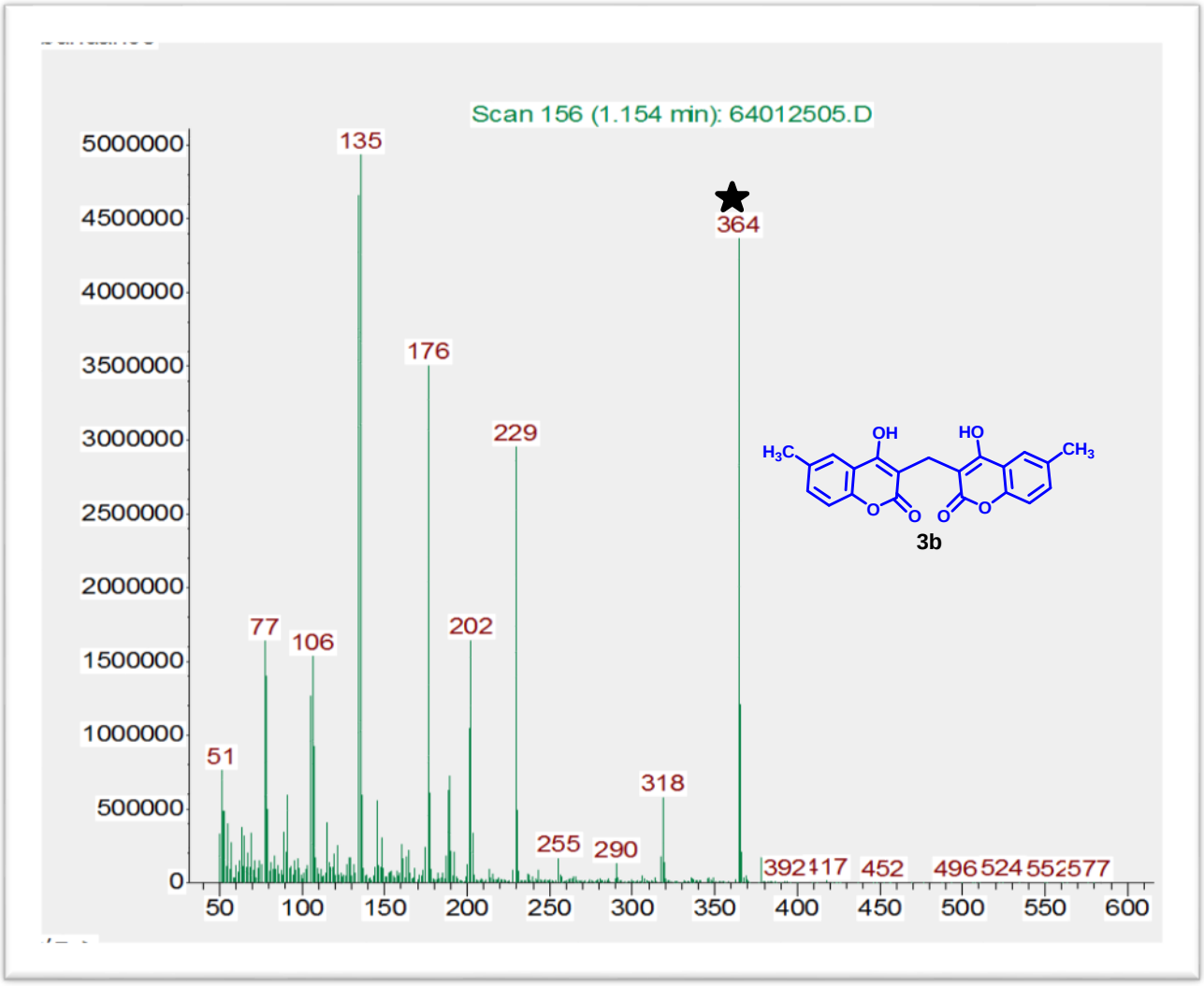
[1]H NMR spectrum of **3b**



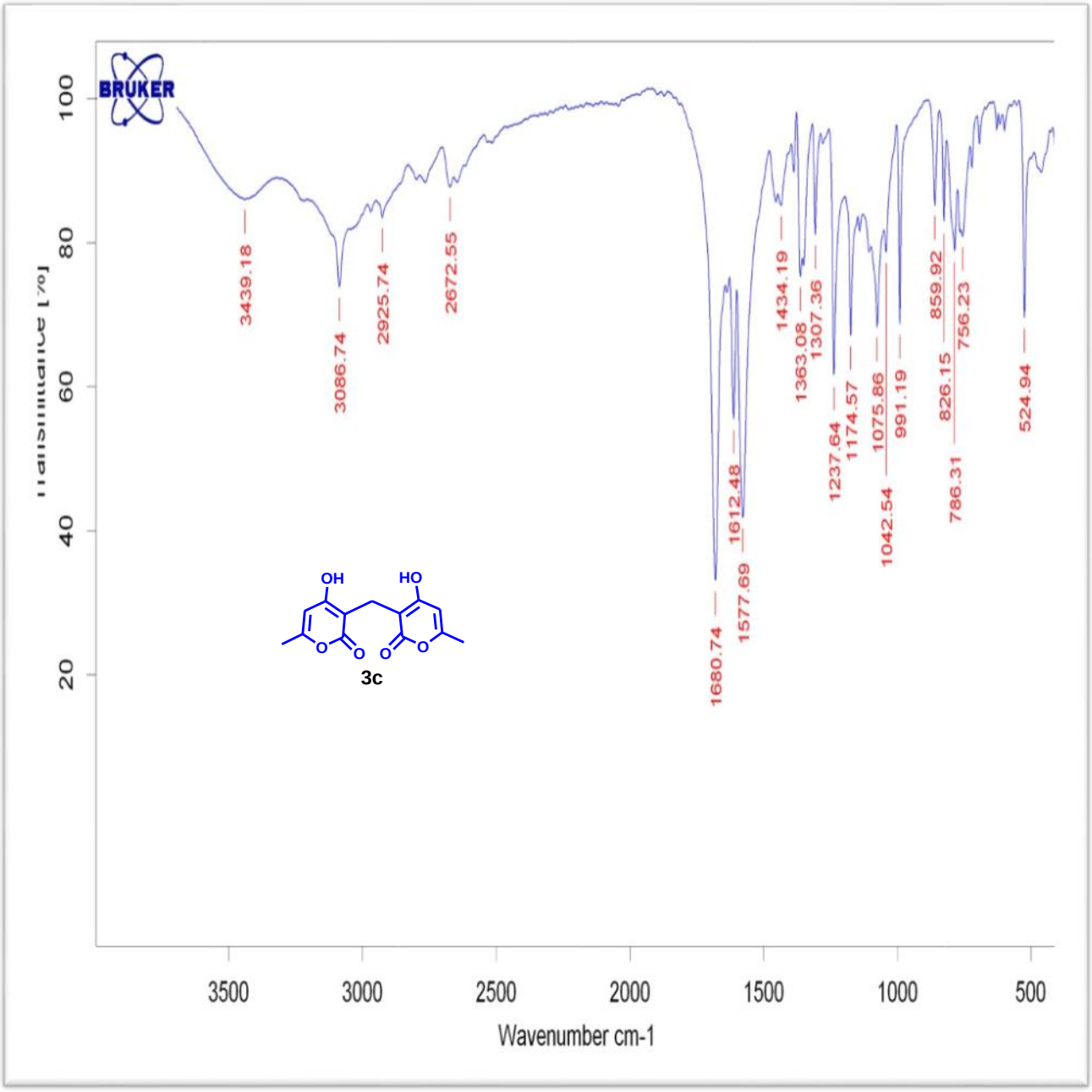
^{13}C NMR spectrum of **3b**



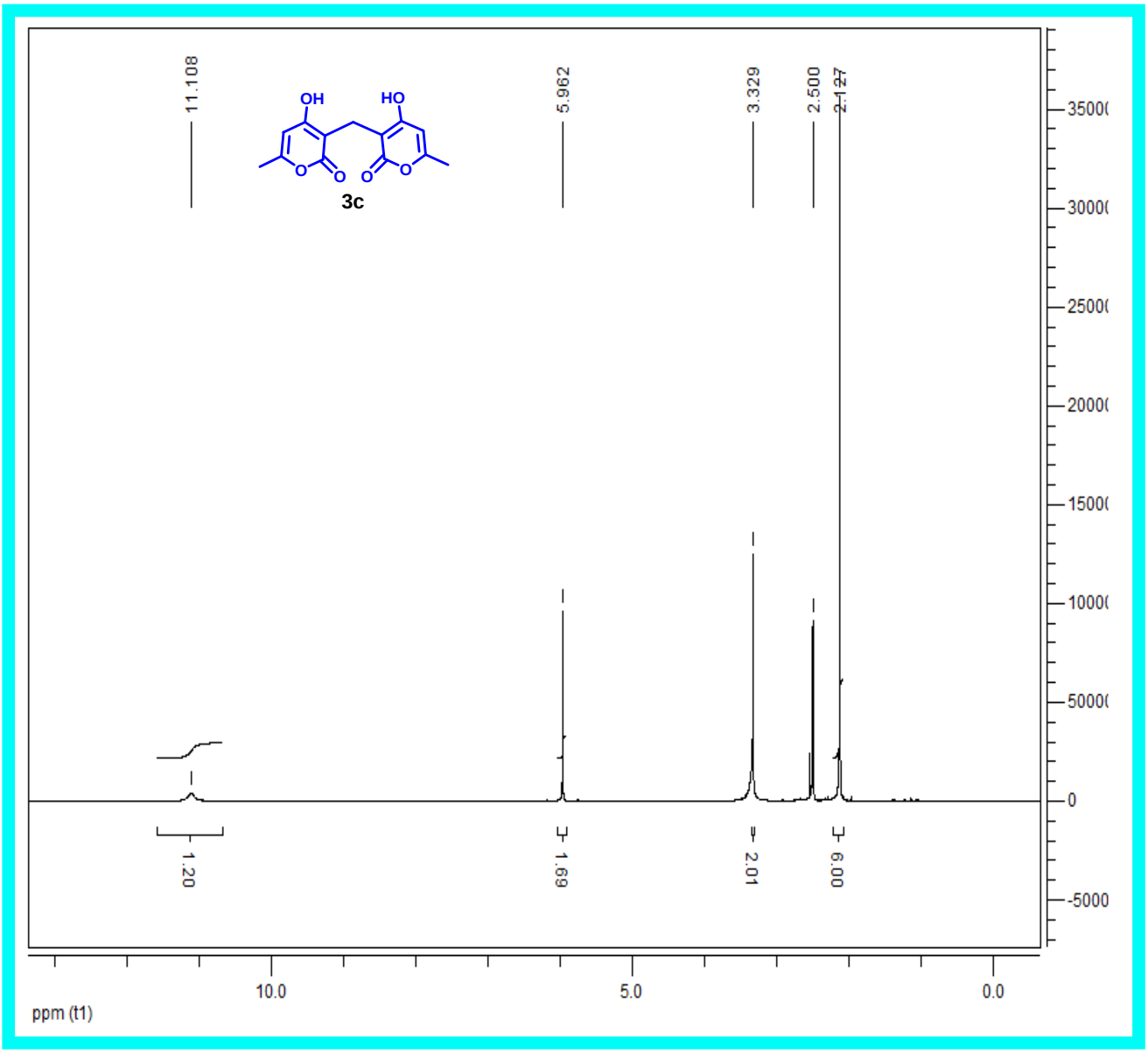
MS spectrum of **3b**



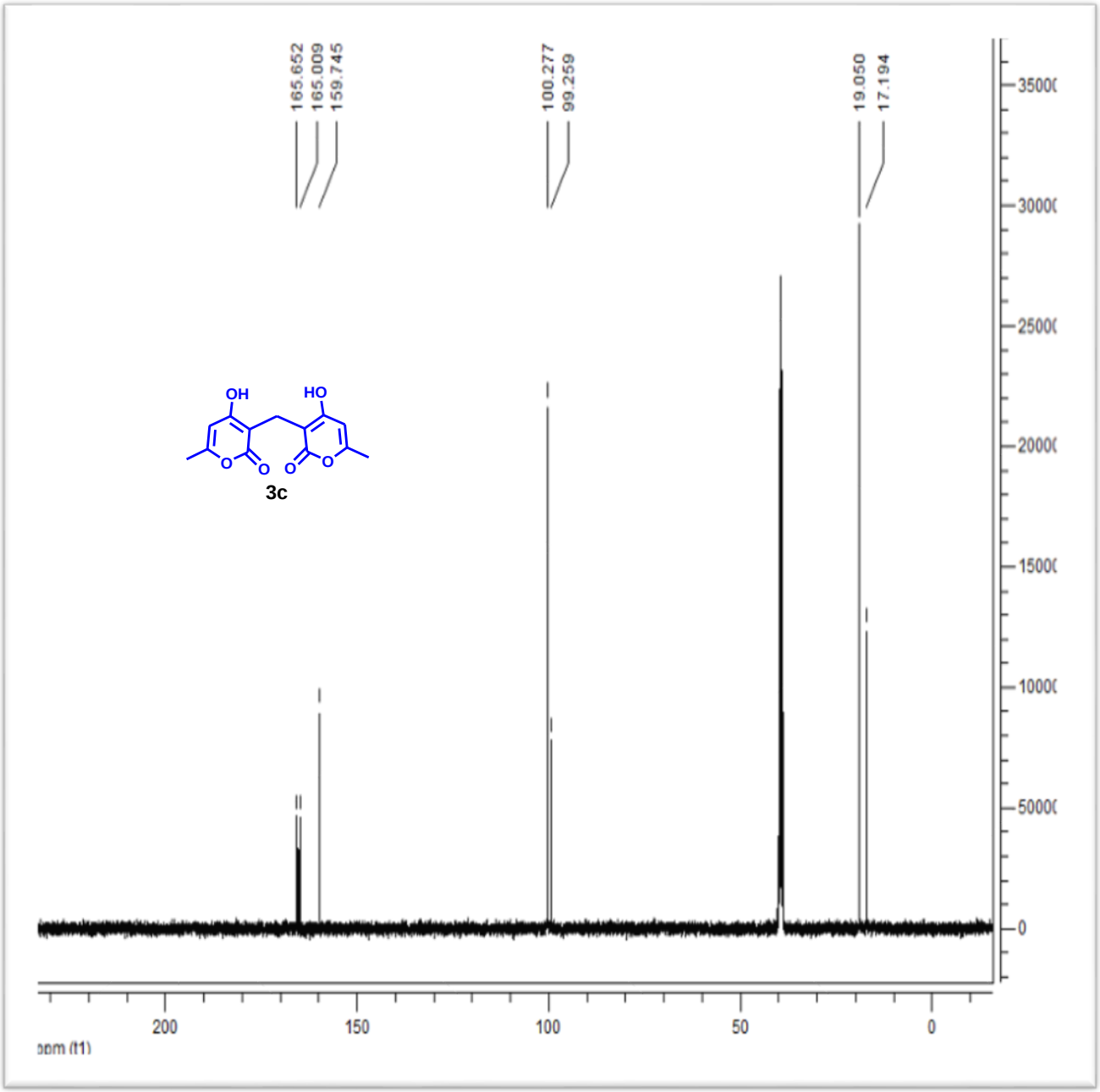
FTIR spectrum of **3c**



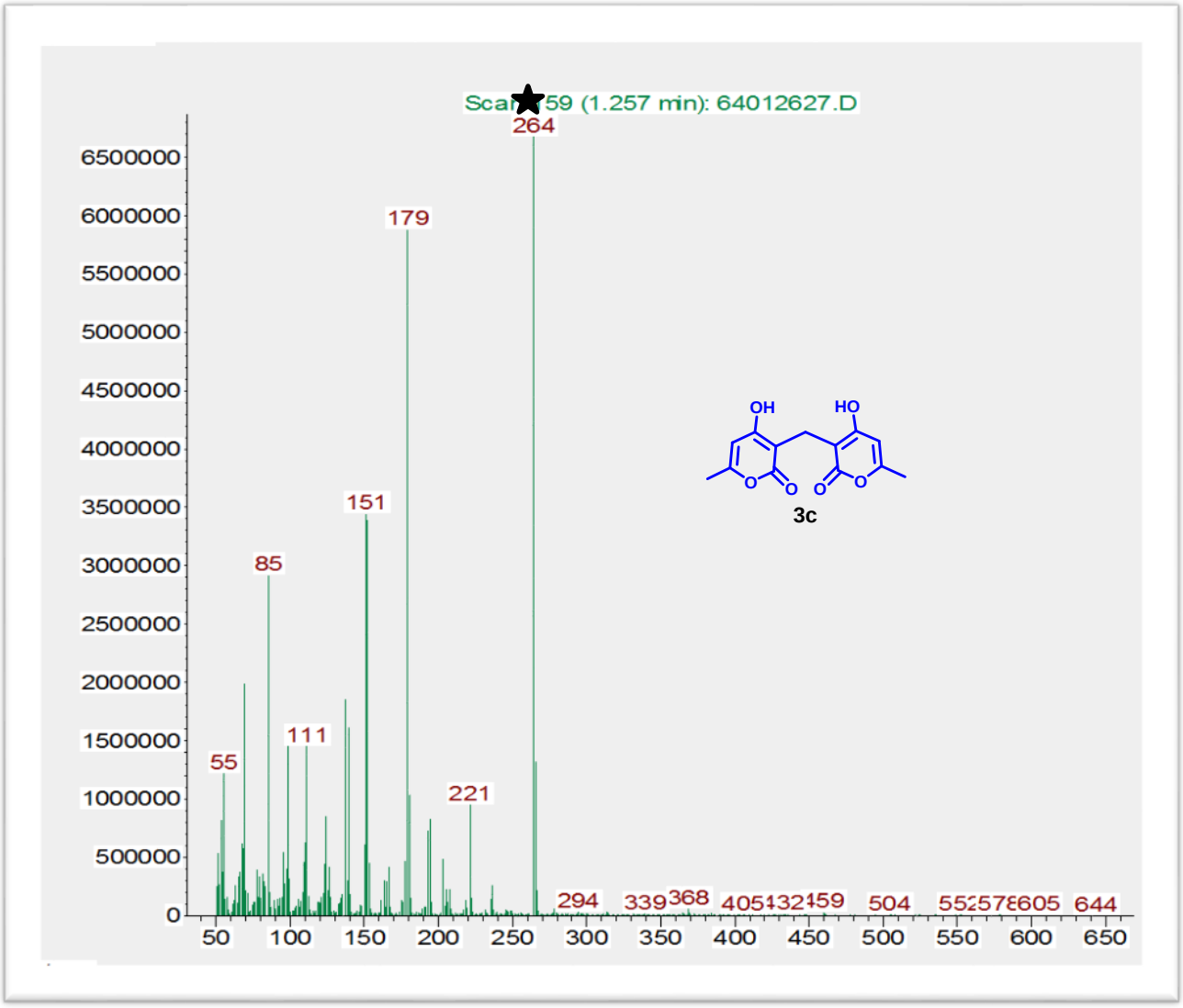
^{1}H NMR spectrum of **3c**



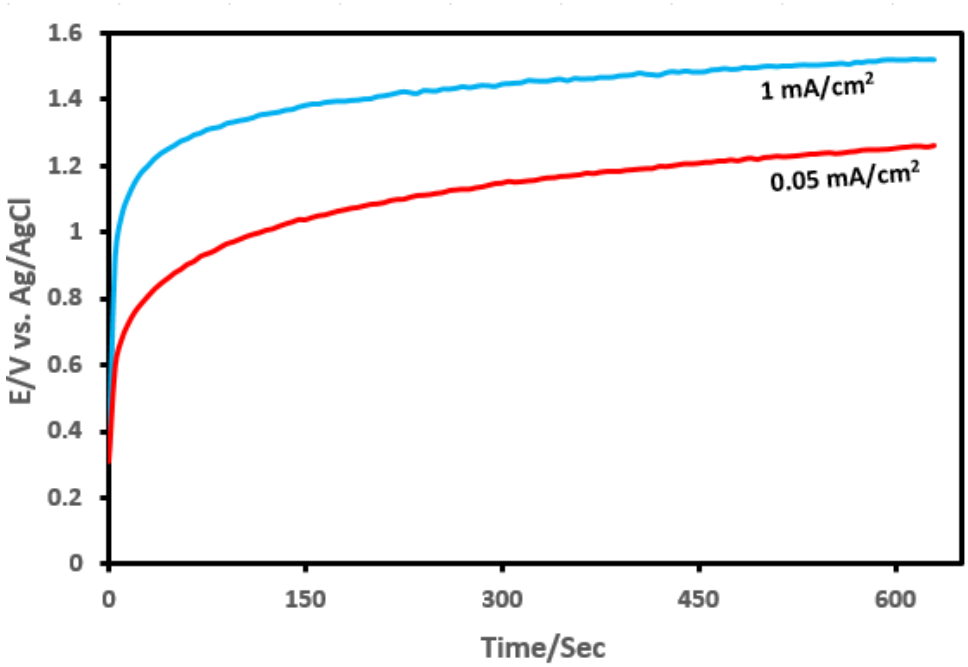
^{13}C NMR spectrum of **3c**



MS spectrum of **3c**



❖ Potential *vs.* Time



Voltage vs time graph for galvanostatic study in optimum current density (1 mA/cm^2) and 0.05 mA/cm^2