Supplementary Information

Electrochemical and mechanochemical synthesis of dihydrofuro[3,2-*c*]chromenones *via* intramolecular C_{sp3}-H *cross*-dehydrogenative oxygenation within warfarin framework: an efficient and straightforward dual approach

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1. General.

All chemicals (analytical grade) except starting warfarin analogues were purchased from reputed companies and used without further purification. All the starting warfarin derivatives used in this present study were synthesized as per the previous reported method.[1] ^{1}H, ^{13}C and ^{19}F NMR spectra were collected at 400, 100 and 376 MHz, respectively, on a Bruker DRX spectrometer using $CDCl_3$ as solvent. Chemical shifts were reported in δ (ppm), relative to the internal standard, TMS. The signals observed are described as s (singlet), d (doublet), t (triplet), and m (multiplet). Coupling constants are reported as *J* value in Hz. Structural assignments were made with additional information from gCOSY, gHSQC, and gHMBC experiments. Mass spectrometry was obtained using a Bruker maXis Impact (Q-TOF) high-resolution mass spectrometer. X-ray single crystallographic data were collected on X′Calibur CCD area-detector diffractometer. The melting points were recorded on a Chemiline CL-725 melting point apparatus and are uncorrected. Thin Layer Chromatography (TLC) was performed using silica gel 60 F_{254} (Merck) plates. The 'DC Variable Power Supply Unit' has been designed and assembled with standard individual electrical parts purchased from reputed companies at the end of the present investigators. Graphite, Pt, Ag, Zn, and Cu plate-electrodes have been used for performing electrochemical cell reactions. A pictorial view (Figure S1) of the designed electrochemical set-up is shown below. A PM 100, Retsch GmbH, Germany, ball-milling apparatus was used for all reactions.

A pictorial view of the electrochemical experimental set-up

Figure S1a: Pictorial view (Front-View) of the experimental set-up for the model reaction showing the designed '*DC Variable Power Supply*' unit tagged with *Voltmeter* and *Ammeter*, and an undivided electrochemical cell containing a pair of graphite plates (cathode and anode) and the reaction mixture within a glass vessel placed on a magnetic stirrer with constant stirring at ambient conditions.

A pictorial view of the mechanochemical experimental set-up

Figure S1b: Pictorial view PM 100, Retsch GmbH, Germany, ball-milling apparatus

2. General procedure for the synthesis of functionalized dihydrofuro[3,2-*c*]chromenones (2)

2.1 In Electrochemical cell

An oven-dried glass vessel was charged with warfarin analogues (**1**; 0.1 mmol), 4.5 mL of 0.07 M KI electrolyte solution in DMSO, and a magnetic stir bar in a sequential manner. The reaction vessel was then capped with a pair of graphite plate electrodes (both the cathode and anode are of the size in a dimension of 0.7 cm × 0.7 cm × 0.2 cm) placed at a 0.5 cm distance constructing an undivided electrochemical cell. Now, an uninterrupted flow of DC current (in mA) was maintained with a constant voltage of 7.0 V through the reaction mixture with stirring at ambient temperature for a stipulated time frame of 20-40 min. The progress of the reaction was monitored by TLC. On completion of the reaction, 20 mL of a mixture of ethyl acetate and saturated aqueous sodium thiosulfate solution in a proportion of 3:1 (v/v) was added to the resulting mixture and shaken well in a separating funnel. The organic layer was separated and dried over anhydrous sodium sulphate. The solvent was then removed under reduced pressure to obtain a white crude mass, which was then subjected to column chromatographic purification using EtOAc-hexane mixtures as eluents, to have pure products of dihydrofuro[3,2-*c*]chromenones **2** (**2a** – **2t**). The structure of each compound was confirmed by spectral studies including ^{1}H-NMR, ^{13}C-NMR, DEPT-135, ^{19}F NMR, 2D-NMR and HRMS. Further structural confirmation was done by single X-ray crystallographic studies with a representative entry, 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**).

2.2 In Ball-milling

A starting warfarin analogue (**1**; 0.2 mmol) mixed with I_2-DMSO (1:1 molar ratio) (1.0 equiv) and neutral alumina (1.5 g) used as the surface was subjected to ball-milling at 550 rpm using a 25 mL stainless steel jar with seven balls (10 mm in diameter) of the same material for 25-55 min. The ball-milling operation was performed using inverted rotation direction, with an interval of 5 min and taking a break of 10 sec. On completion of the reaction (monitored by TLC), 20 mL of ethyl acetate and saturated aqueous sodium thiosulfate solution in a proportion of 3:1 (v/v) was added to the resulting mixture and shaken well in a separating funnel. The organic layer was separated and dried over anhydrous sodium sulphate. The solvent was then removed under reduced pressure to obtain a white crude mass, which was then subjected to column chromatographic purification using EtOAc-hexane mixtures as eluents, to have pure products of dihydrofuro[3,2-*c*]chromenones **2** (**2-1** – **2-29**).

3. Scanned copies of ^{1}H NMR, ^{13}C NMR, DEPT-135, ^{19}F NMR, 2D-NMR (for model compound 2-6, along with showing the corresponding homo- and hetero-nuclear interactions in Table S1) and HRMS spectra for all the synthesized dihydrofuro[3,2-*c*]chromenones 2 (2-1–2-29) (Figure S2 – S130)

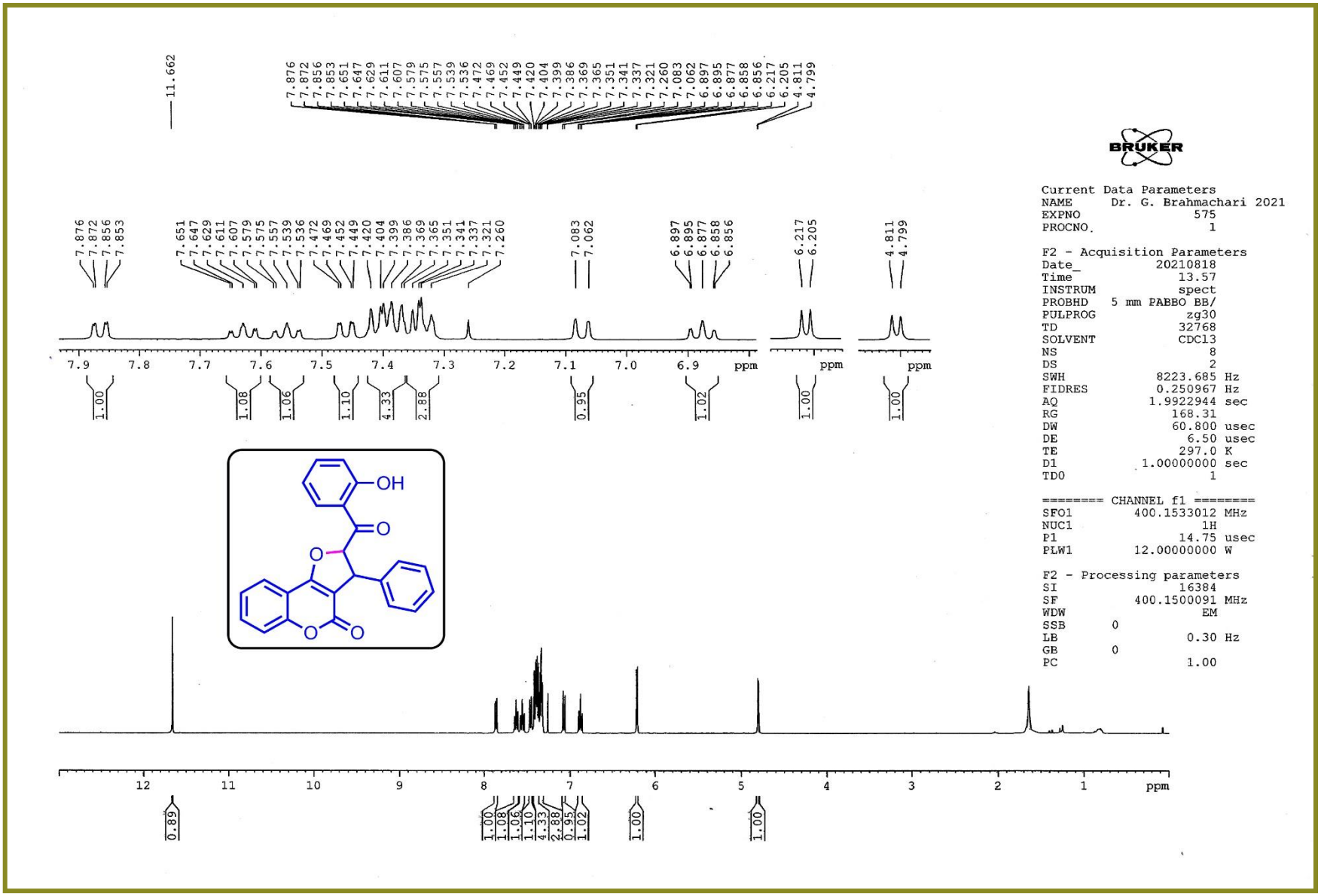


Figure S2. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**)

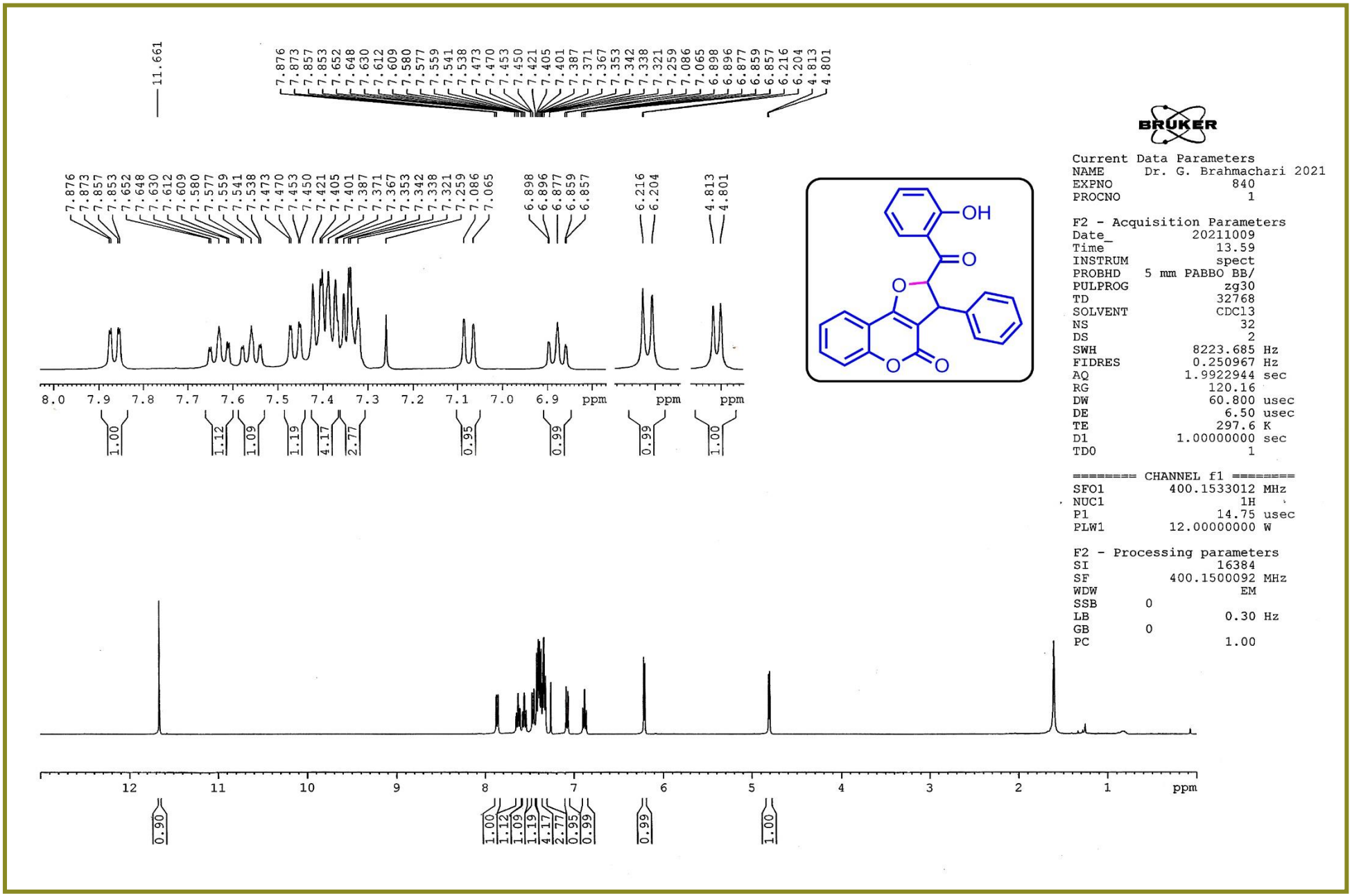


Figure S3. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**) [**Larger Scale**]

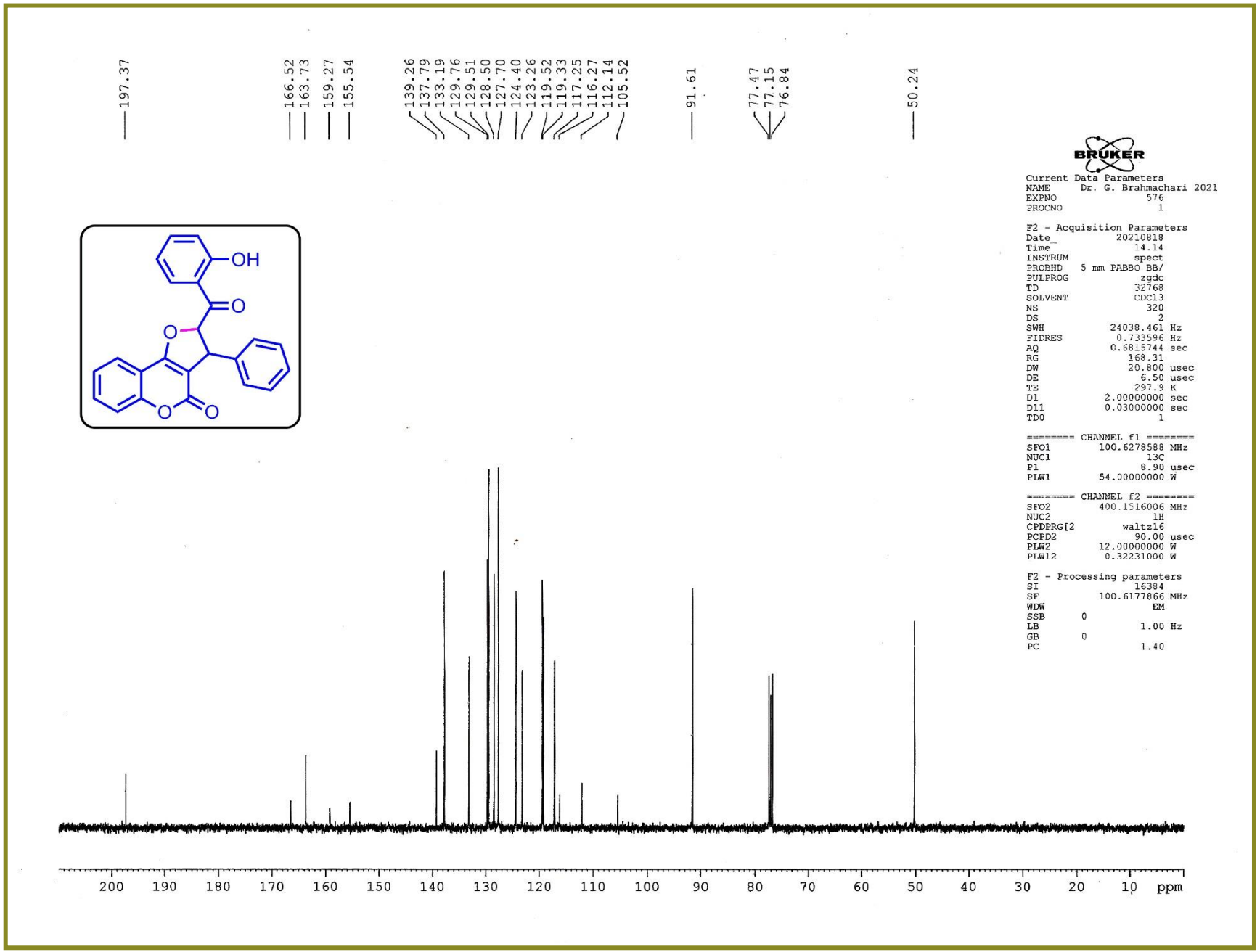


Figure S4. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**)

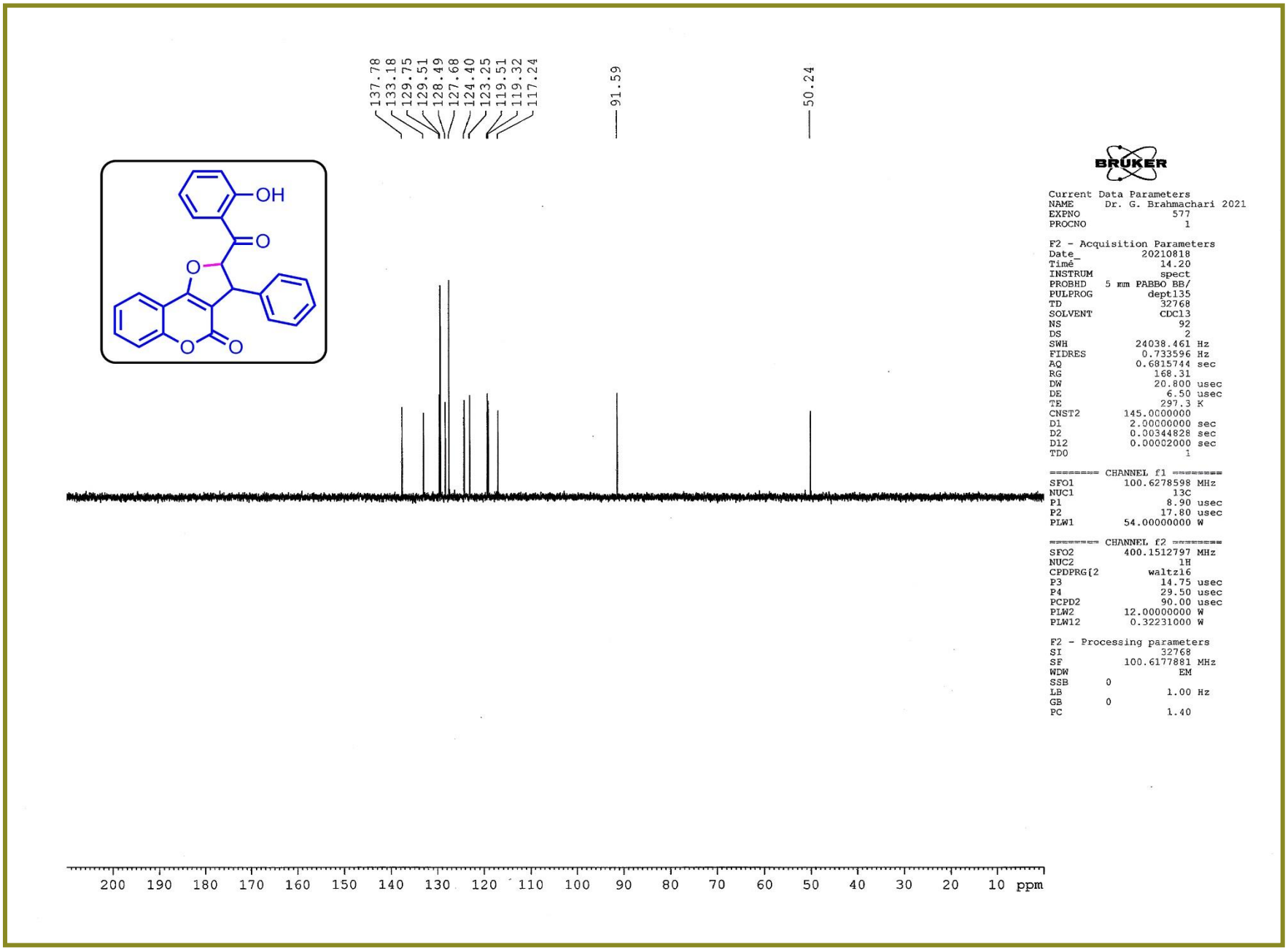


Figure S5. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**)

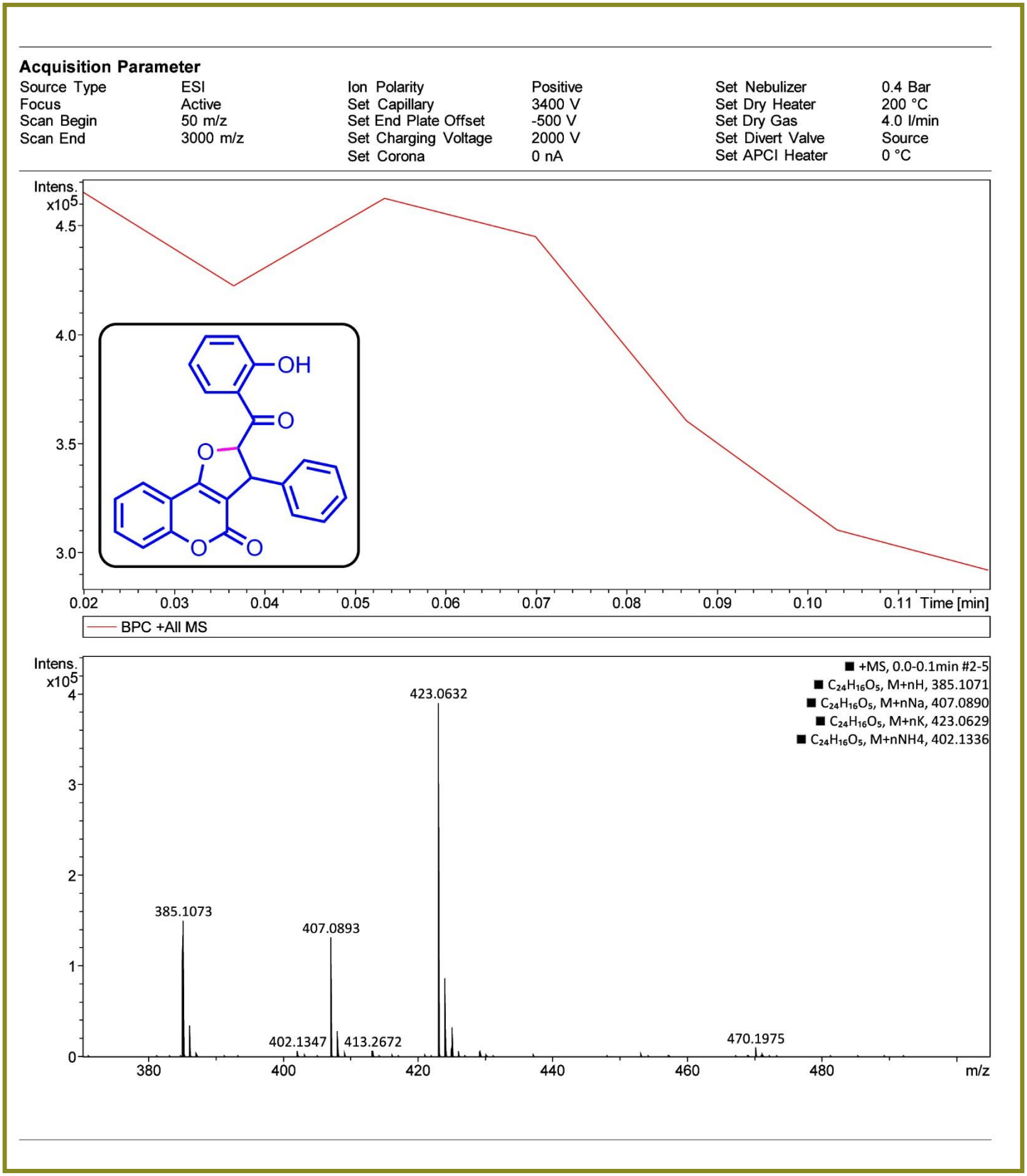


Figure S6. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**)

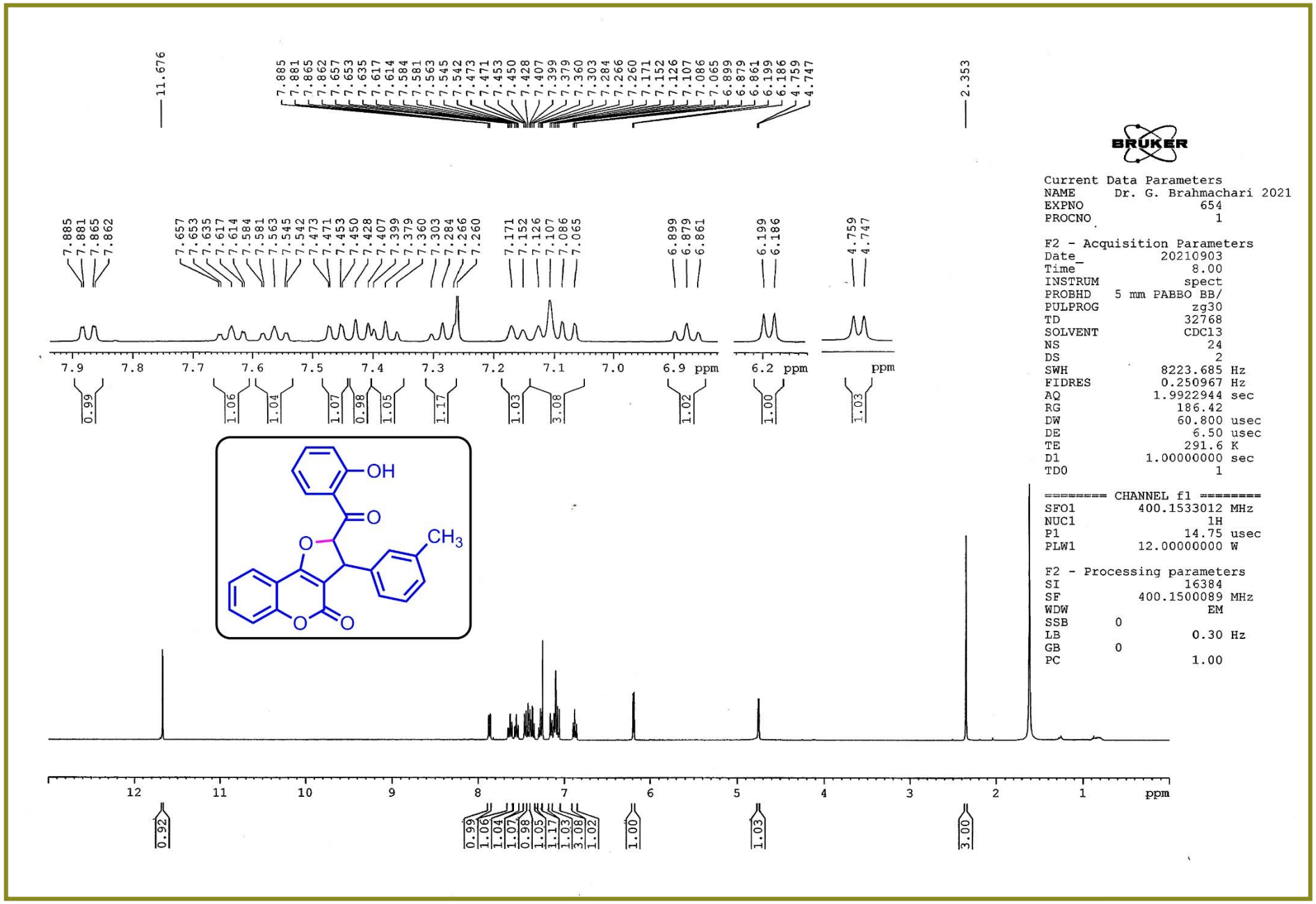


Figure S7. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(*m*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-2**)

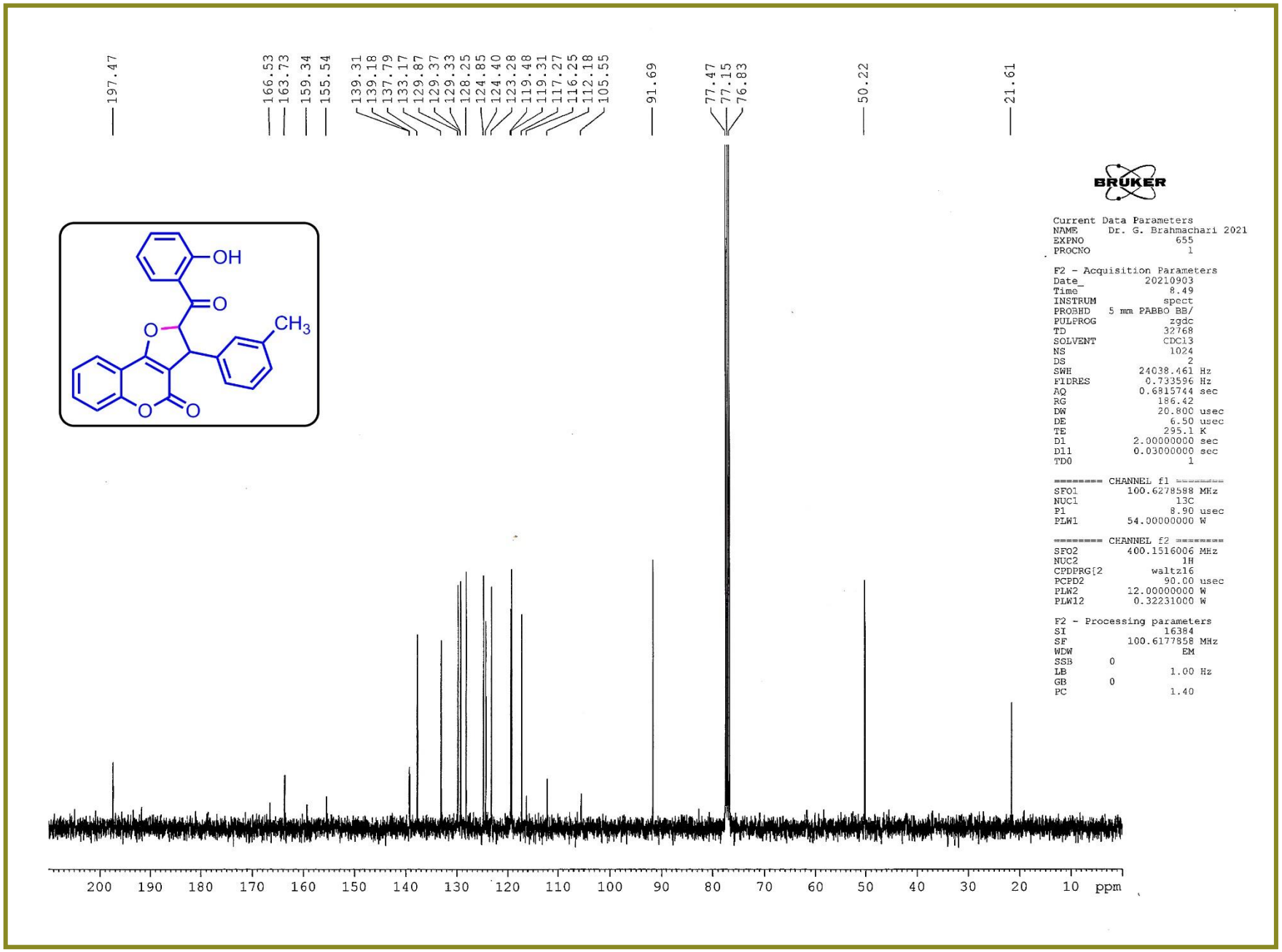


Figure S8. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(*m*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-2**)

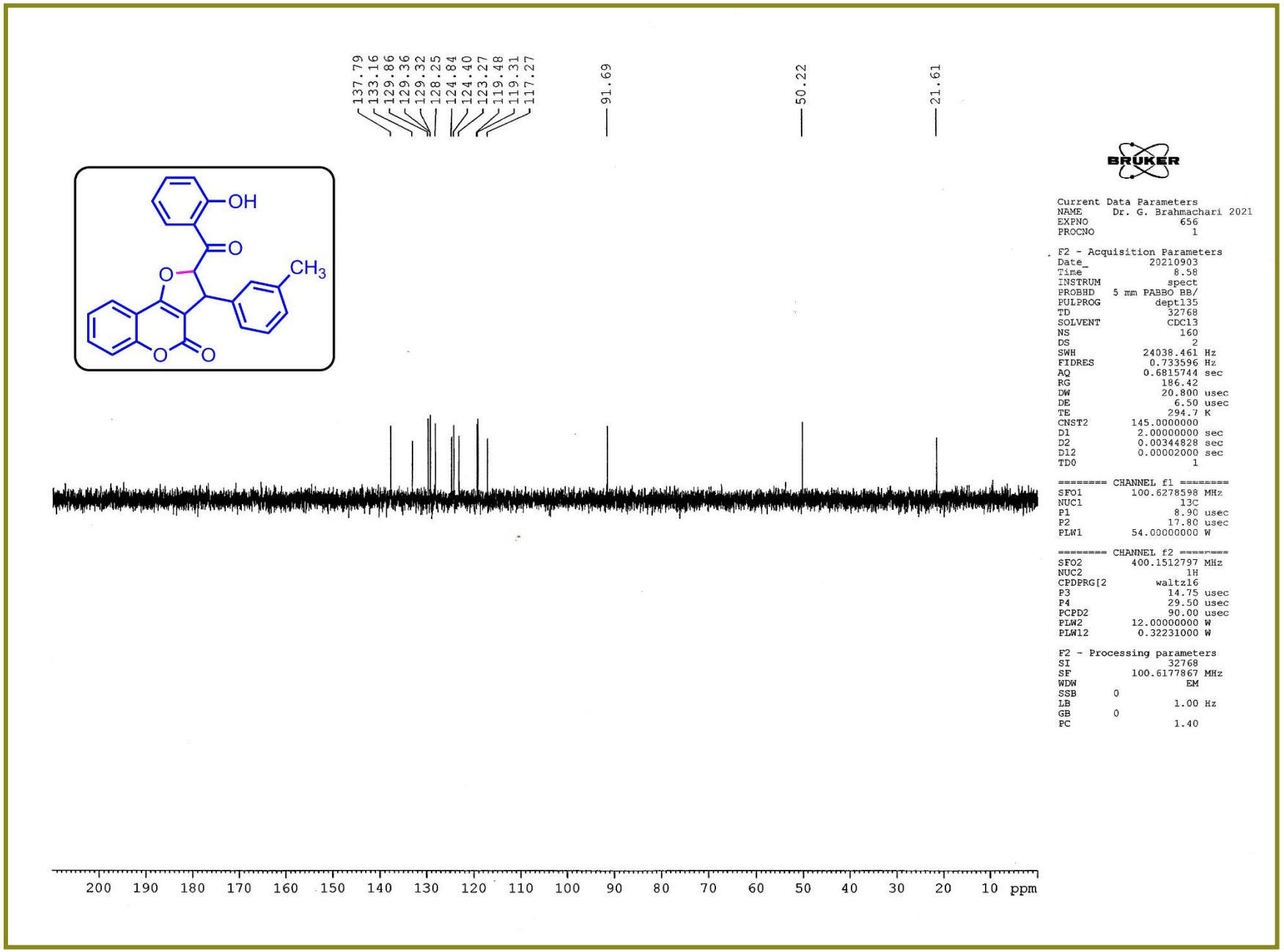


Figure S9. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(*m*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-2**)

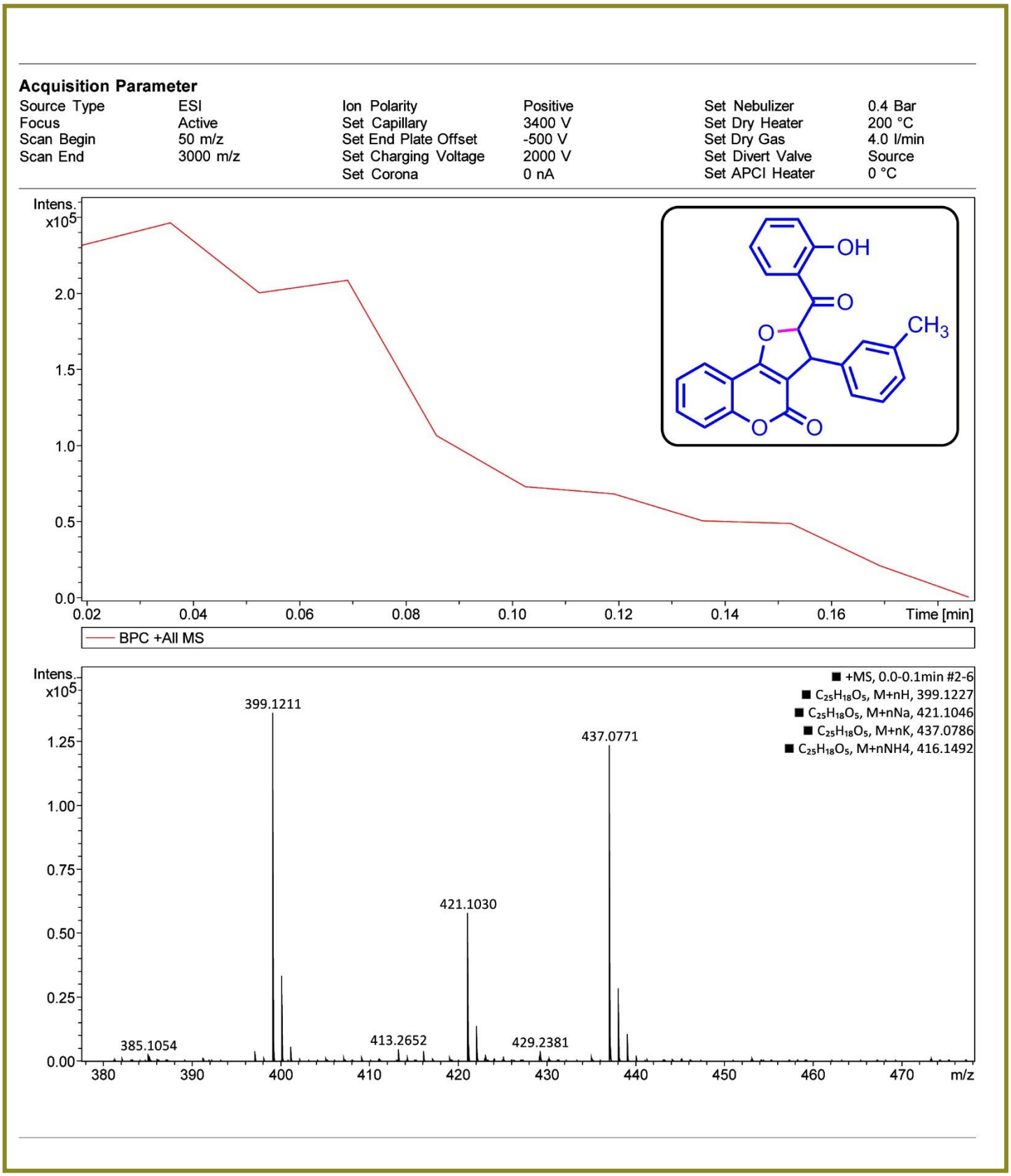


Figure S10. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(*m*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-2**)

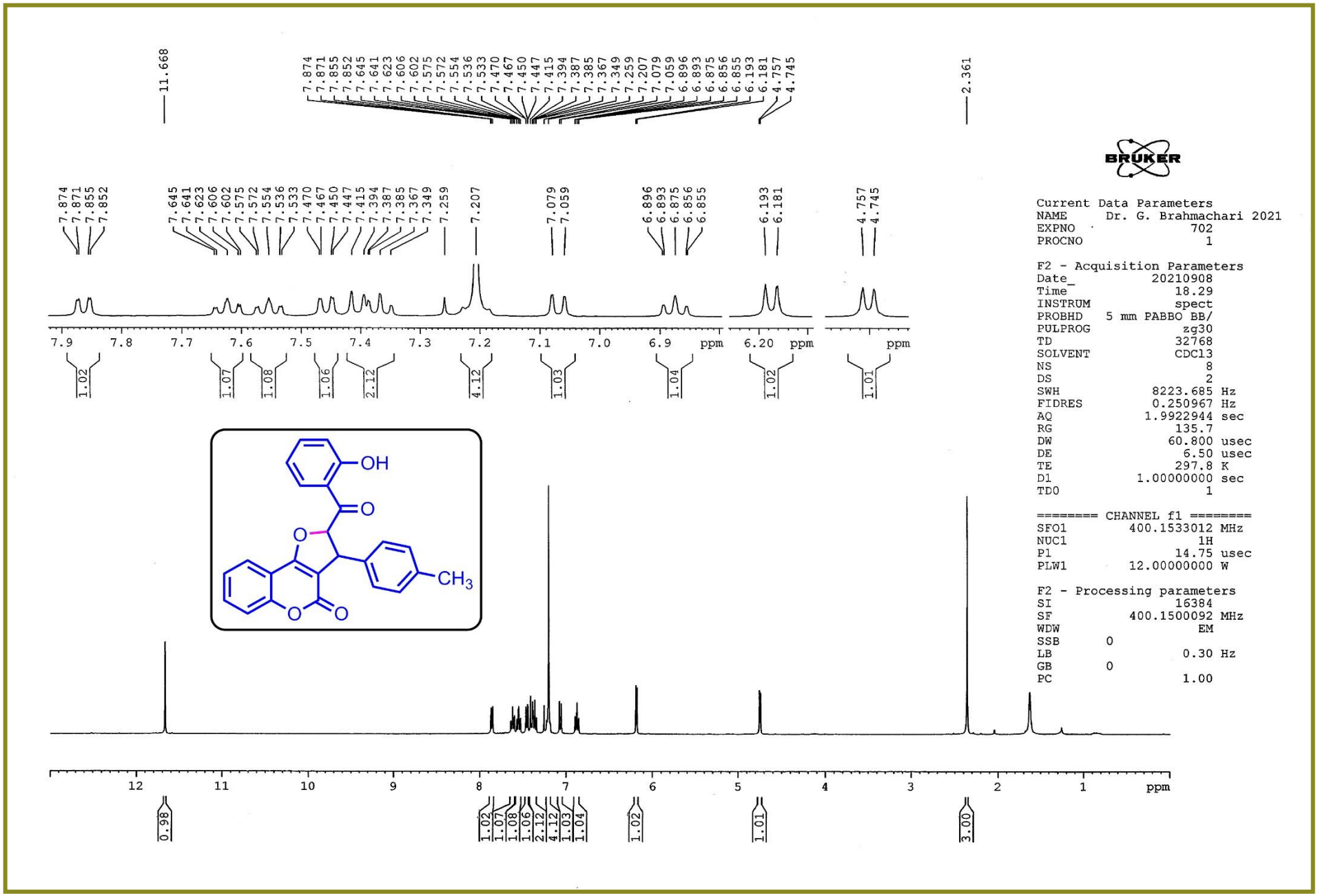


Figure S11. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(*p*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-3**)

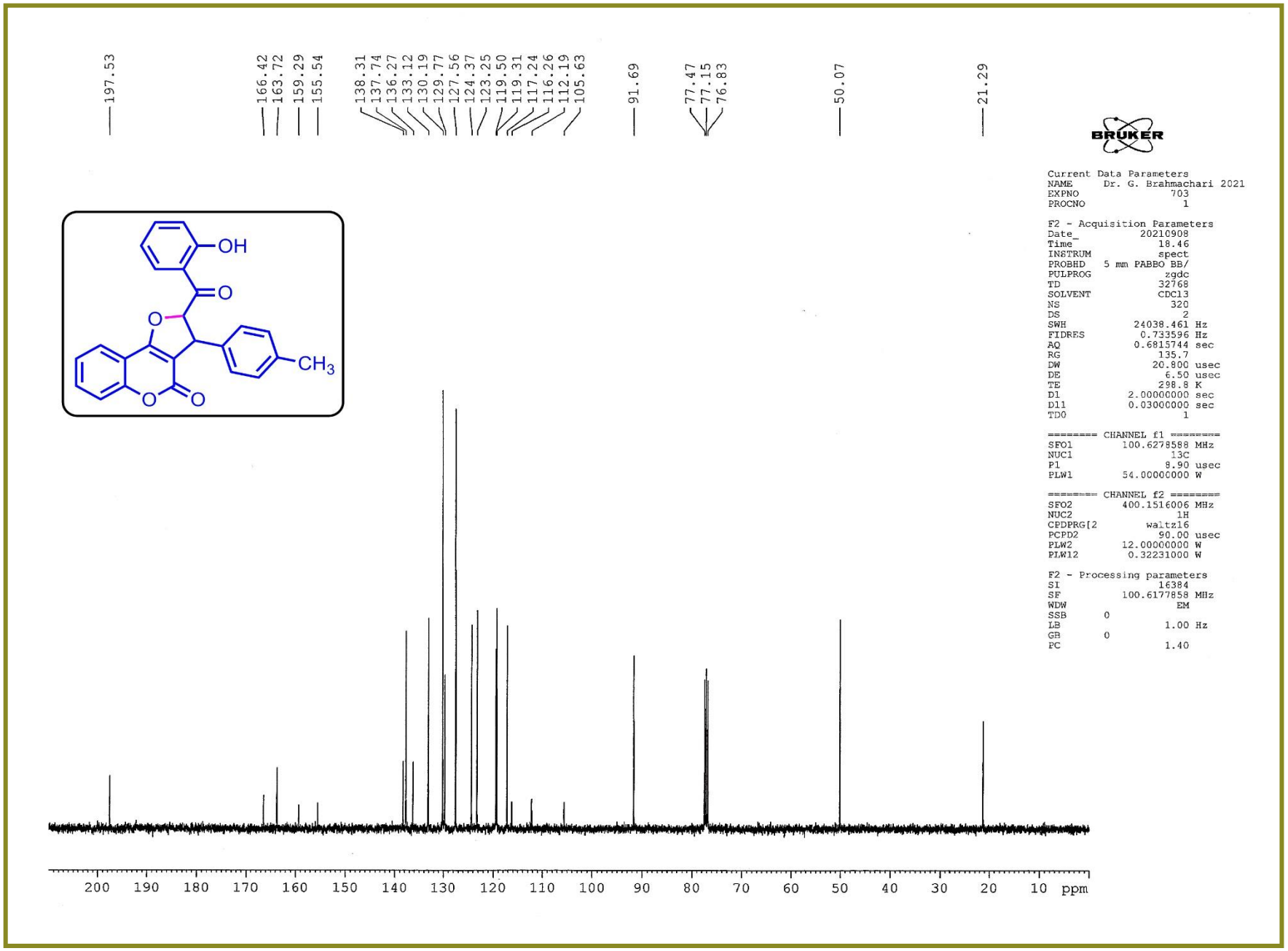


Figure S12. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(*p*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-3**)

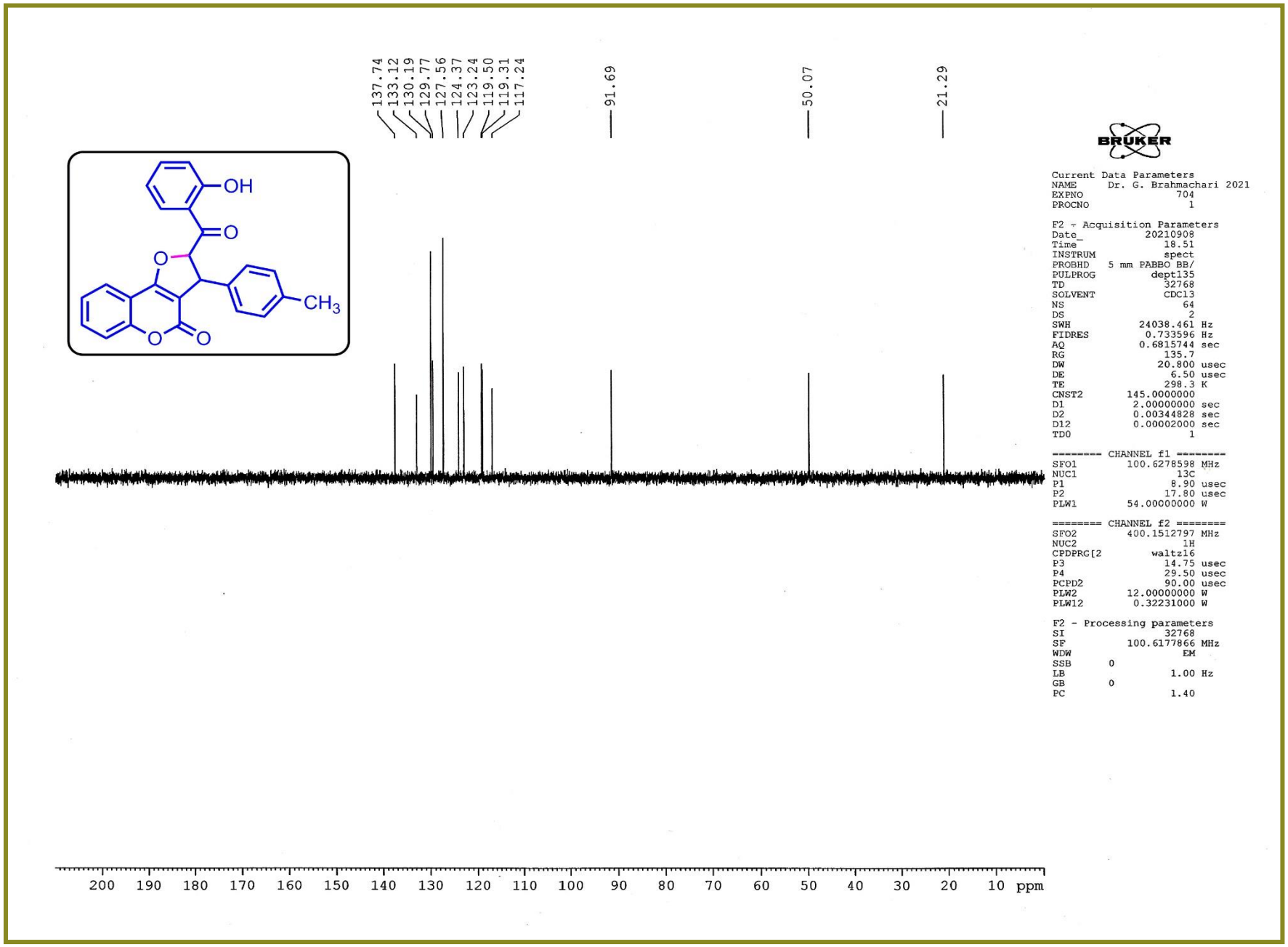
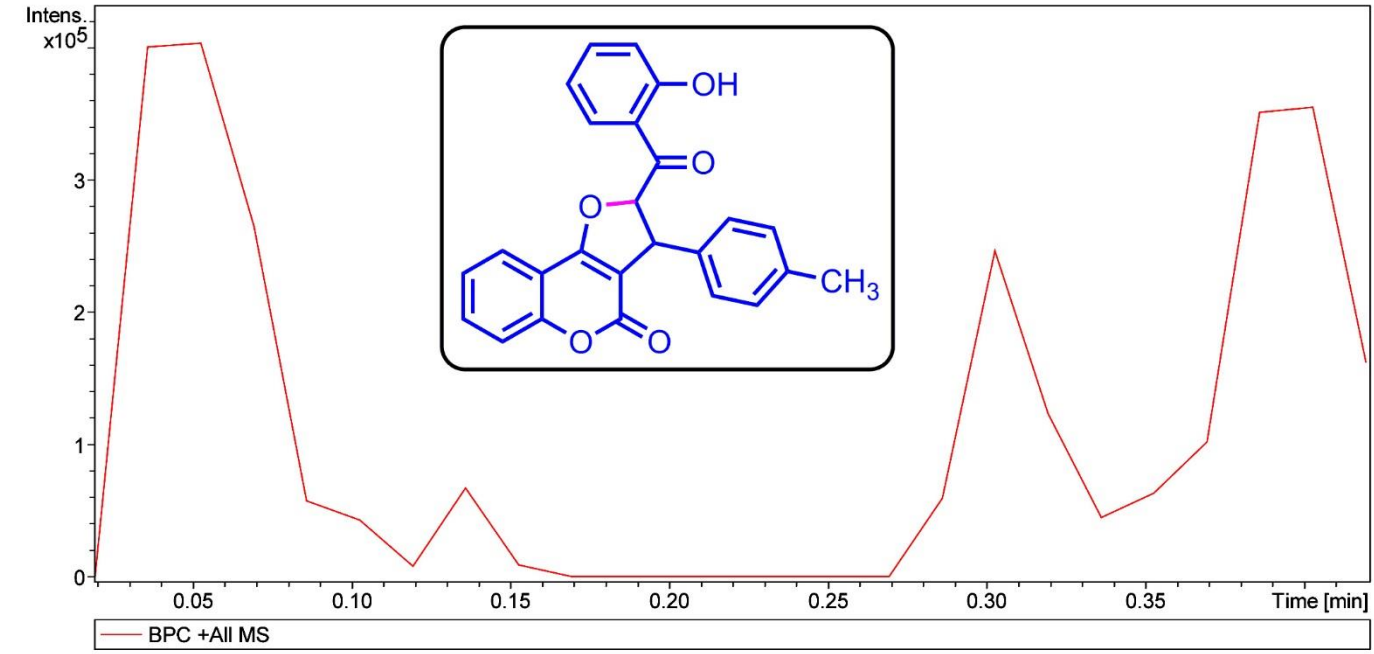


Figure S13. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(*p*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-3**)

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	0.4 Bar
Focus	Active	Set Capillary	3400 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



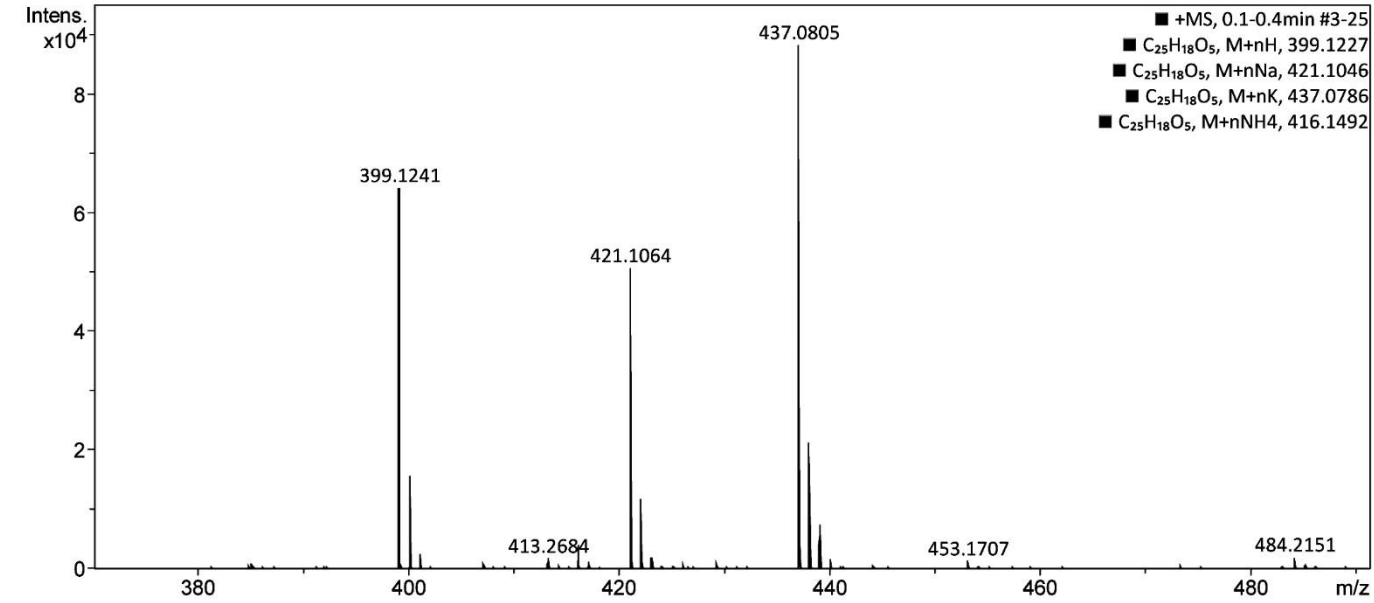


Figure S14. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(*p*-tolyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-3**)

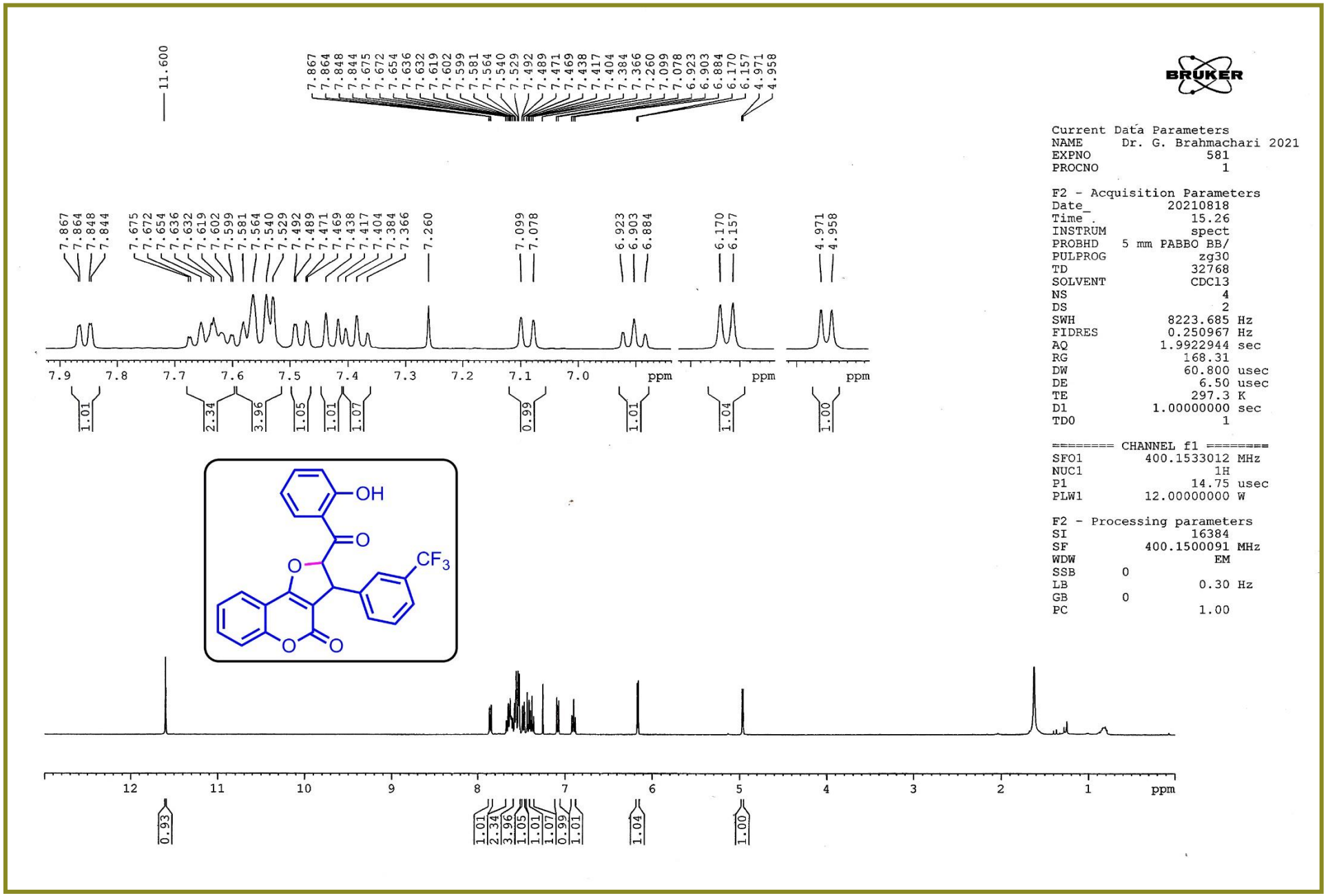


Figure S15. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-4**)

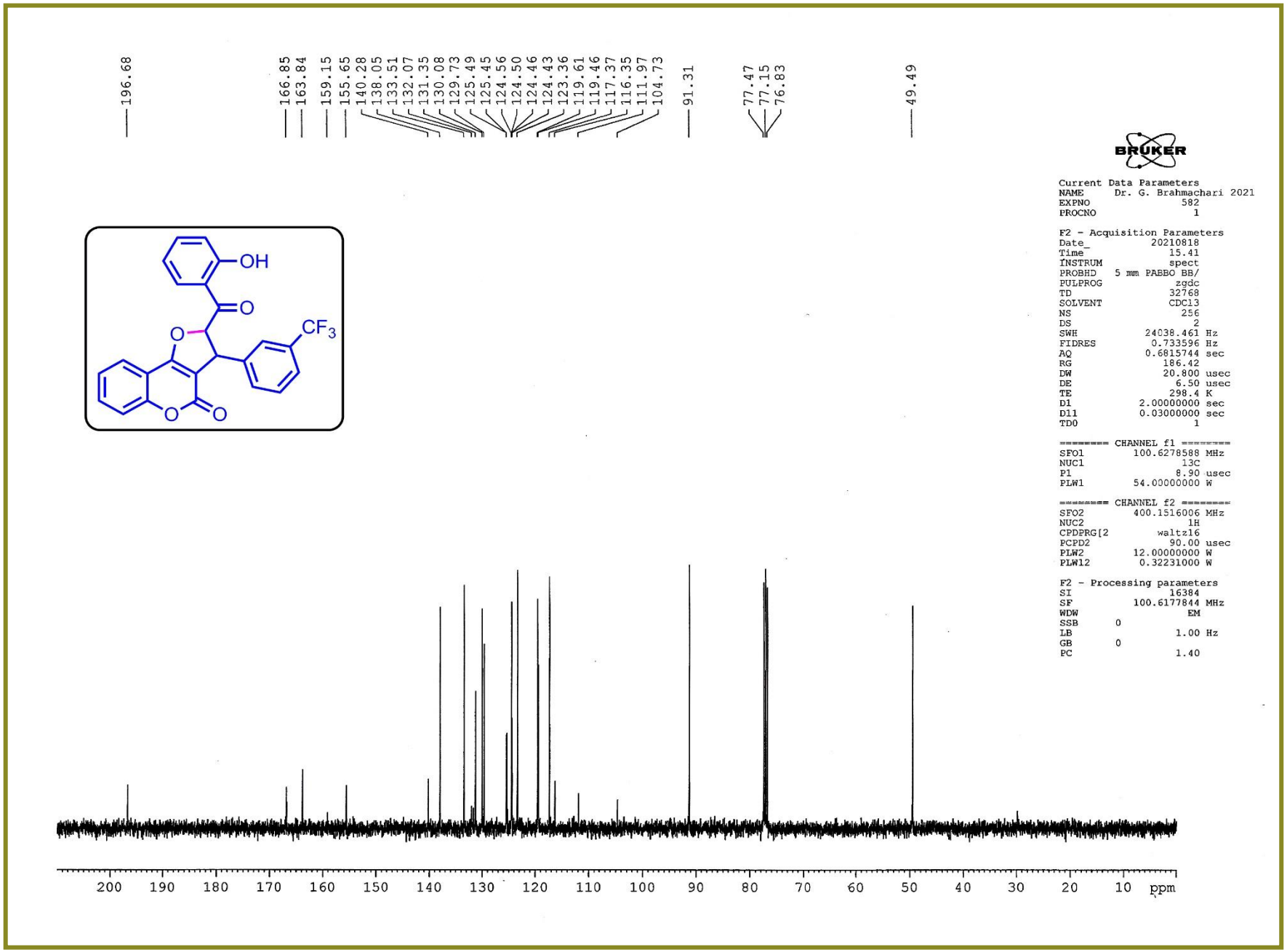


Figure S16. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-4**)

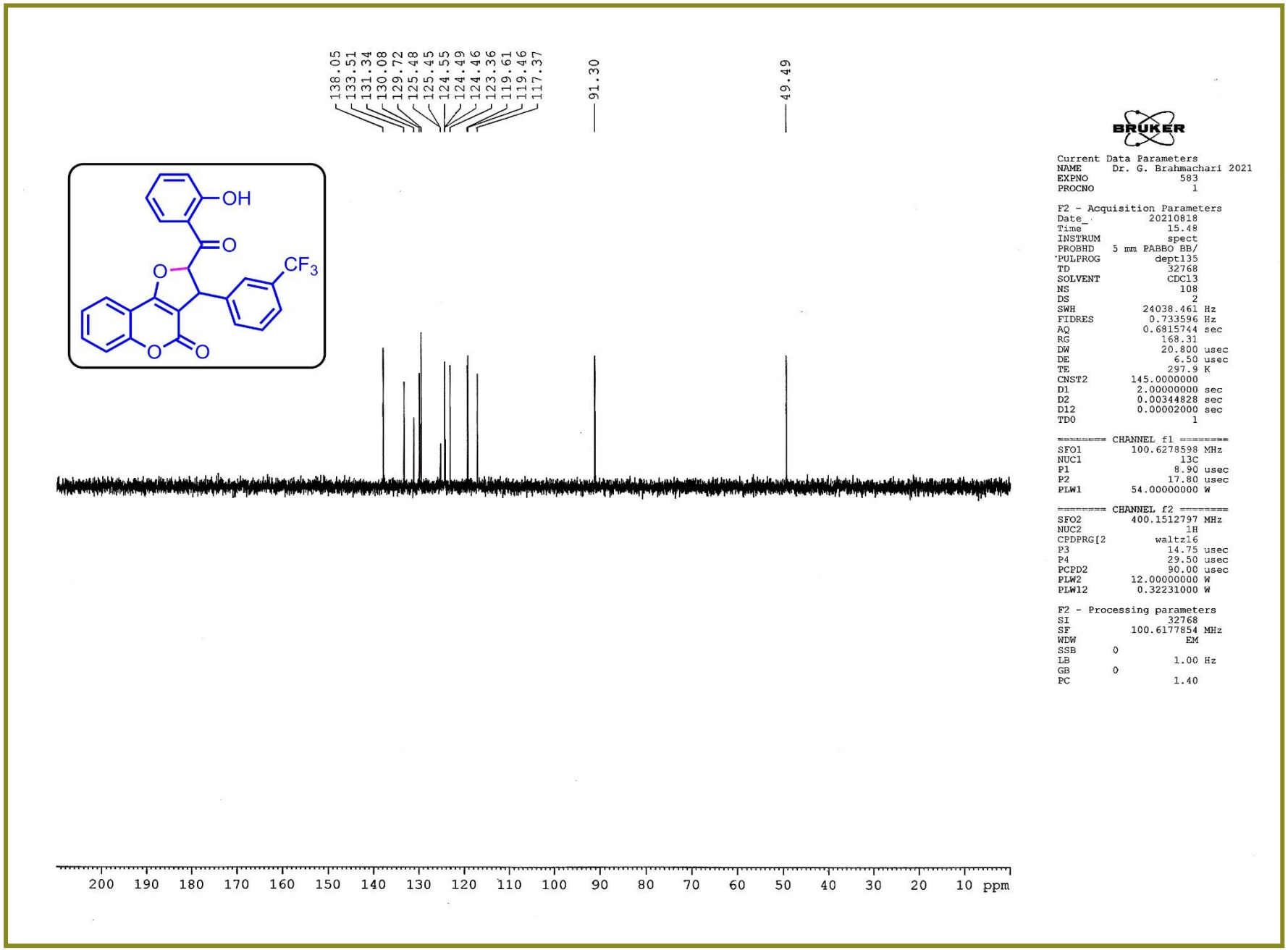


Figure S17. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-4**)

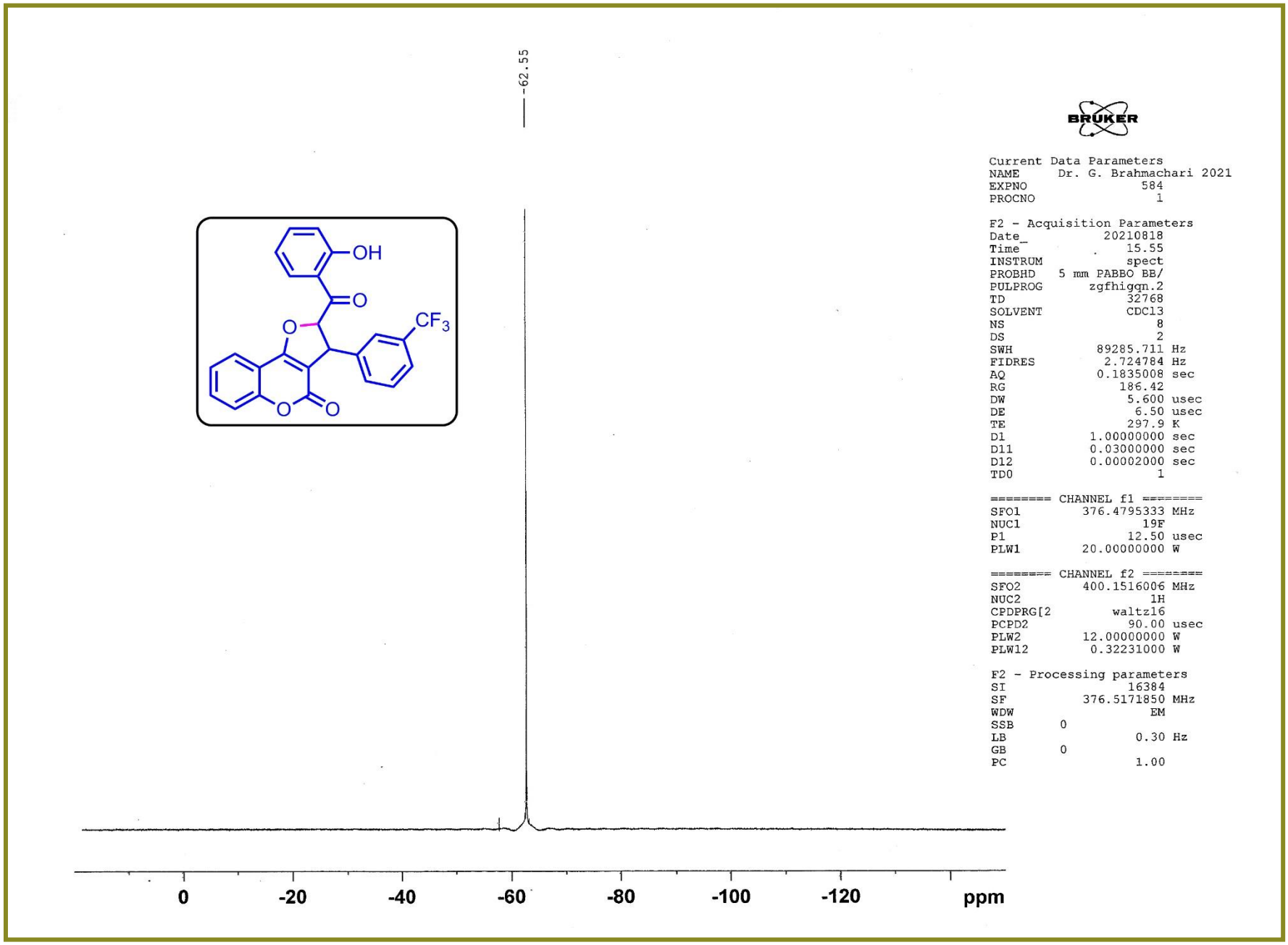


Figure S18. ^{19}F NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-4**)

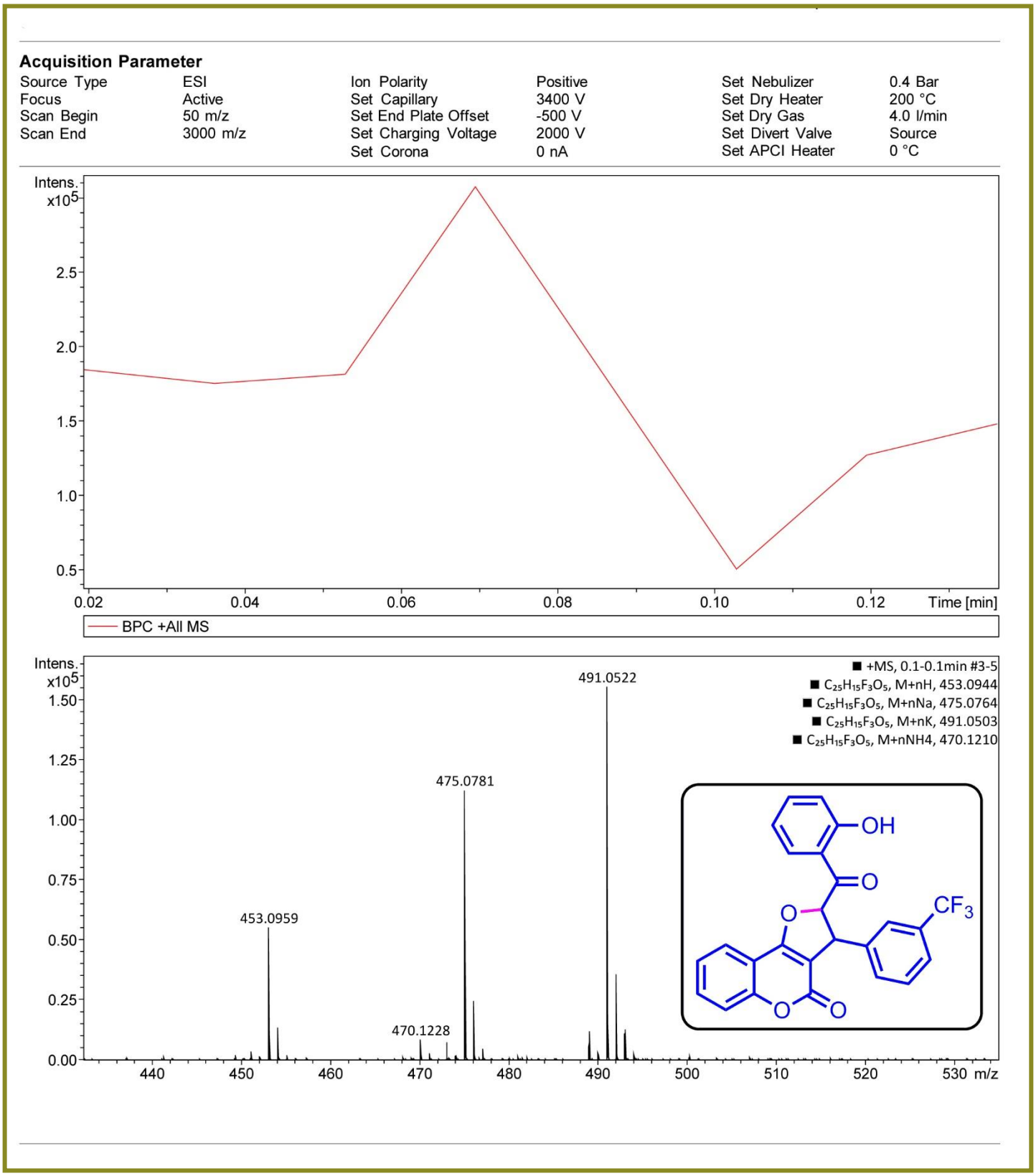


Figure S19. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-4**)

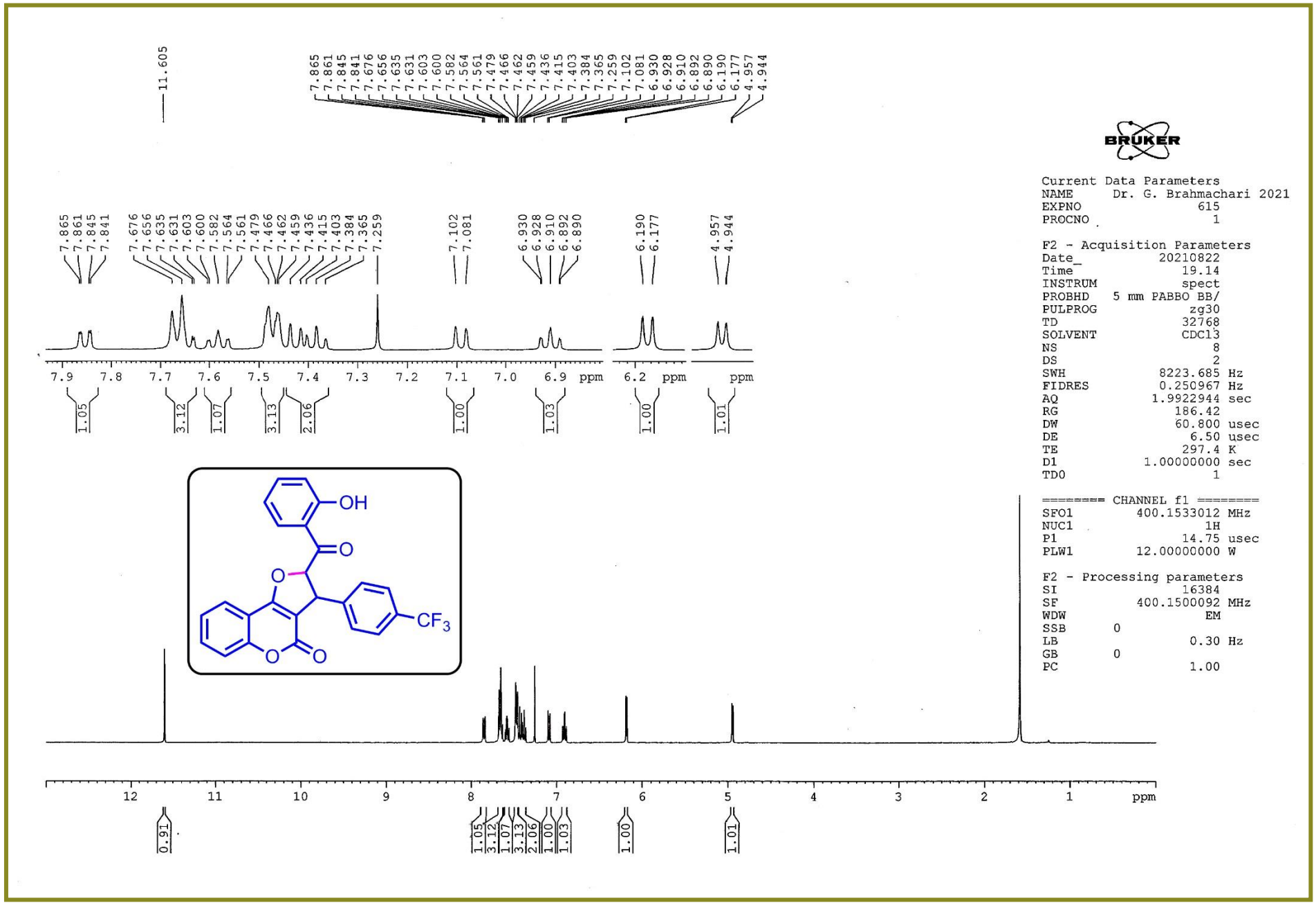


Figure S20. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-5**)

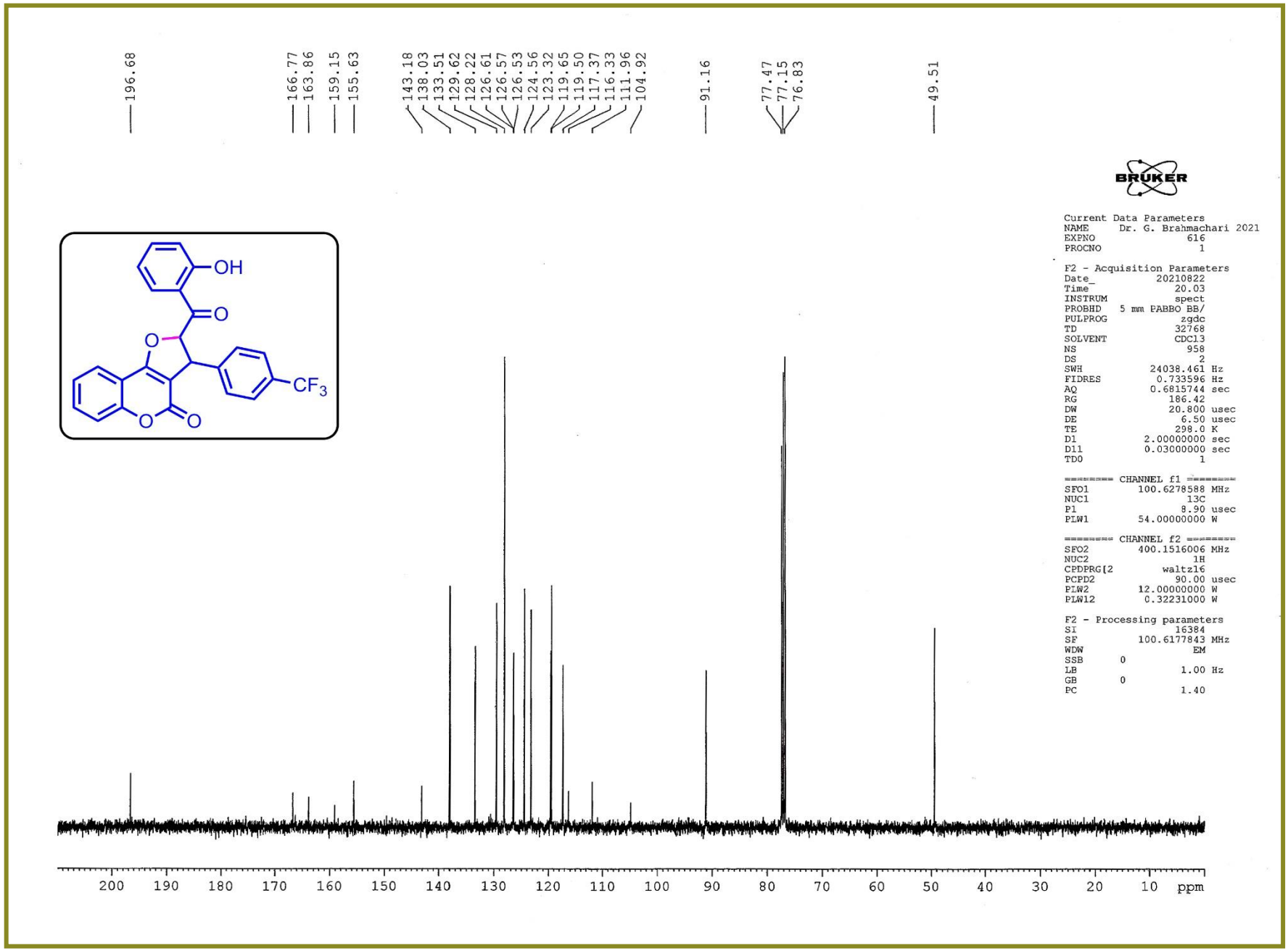


Figure S21. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-5**)

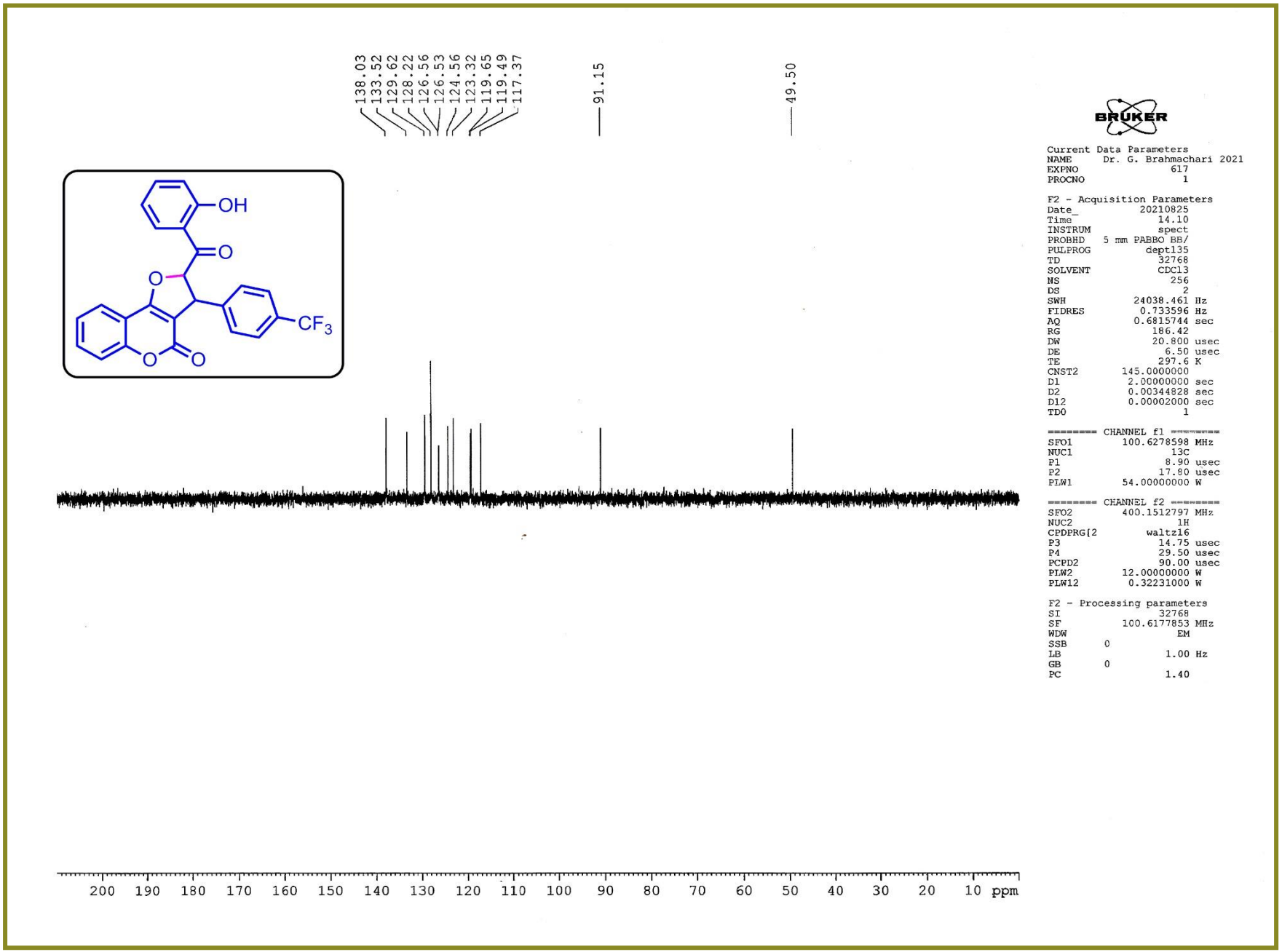


Figure S22. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-5**)

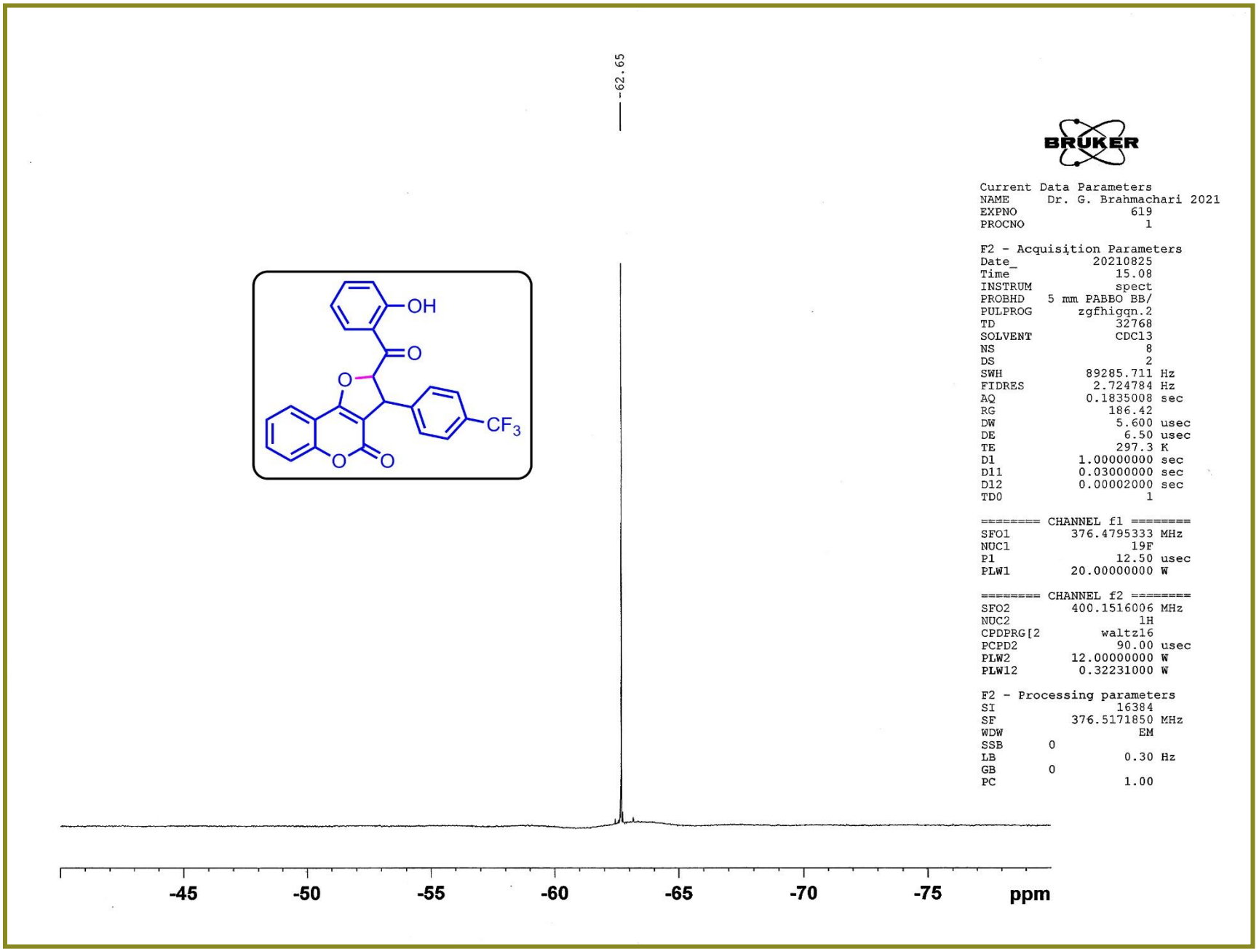


Figure S23. ^{19}F NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-5**)

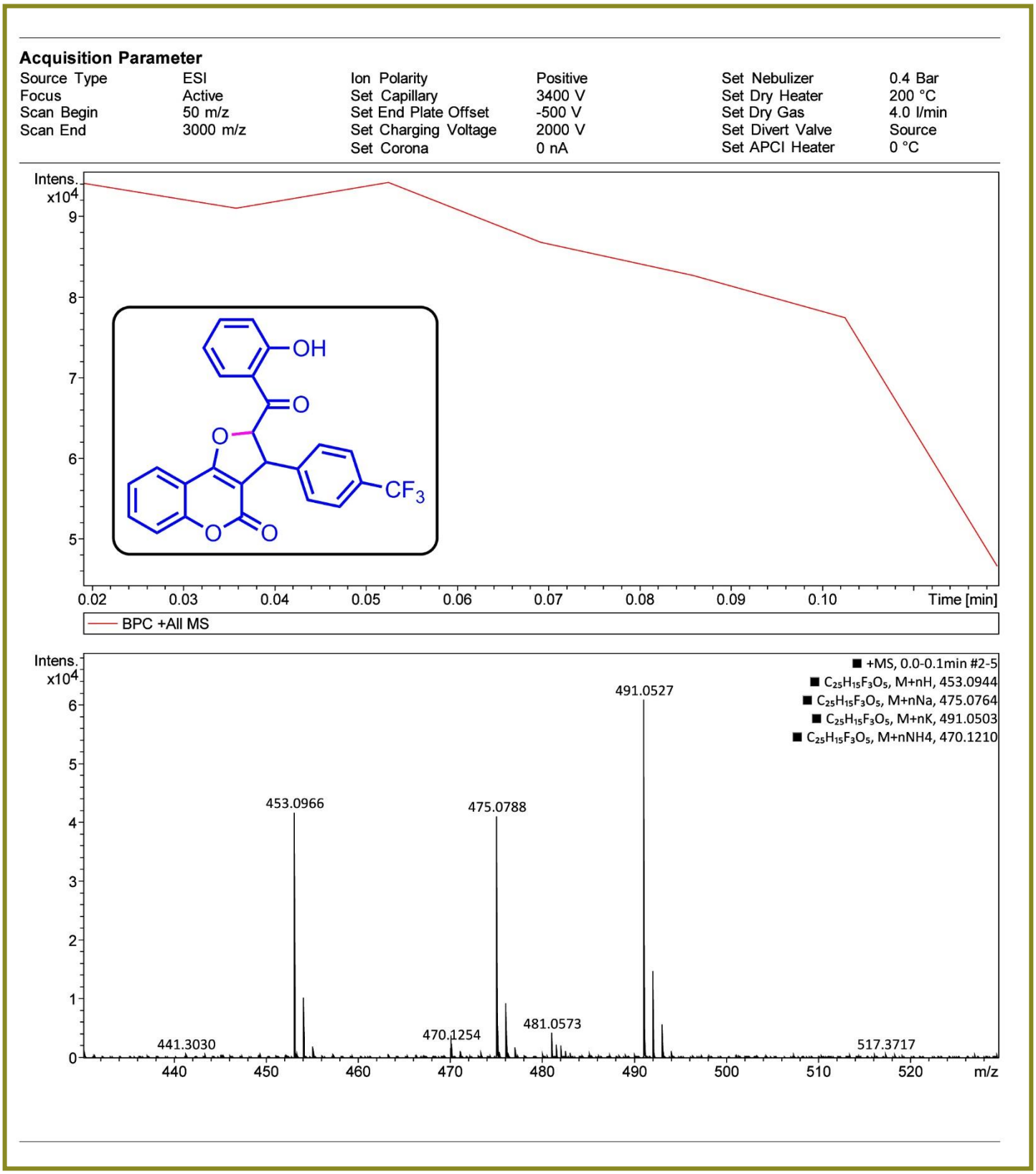


Figure S24. High-resolution Mass spectra of 22-(2-hydroxybenzoyl)-3-(4-(trifluoromethyl)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-5**)

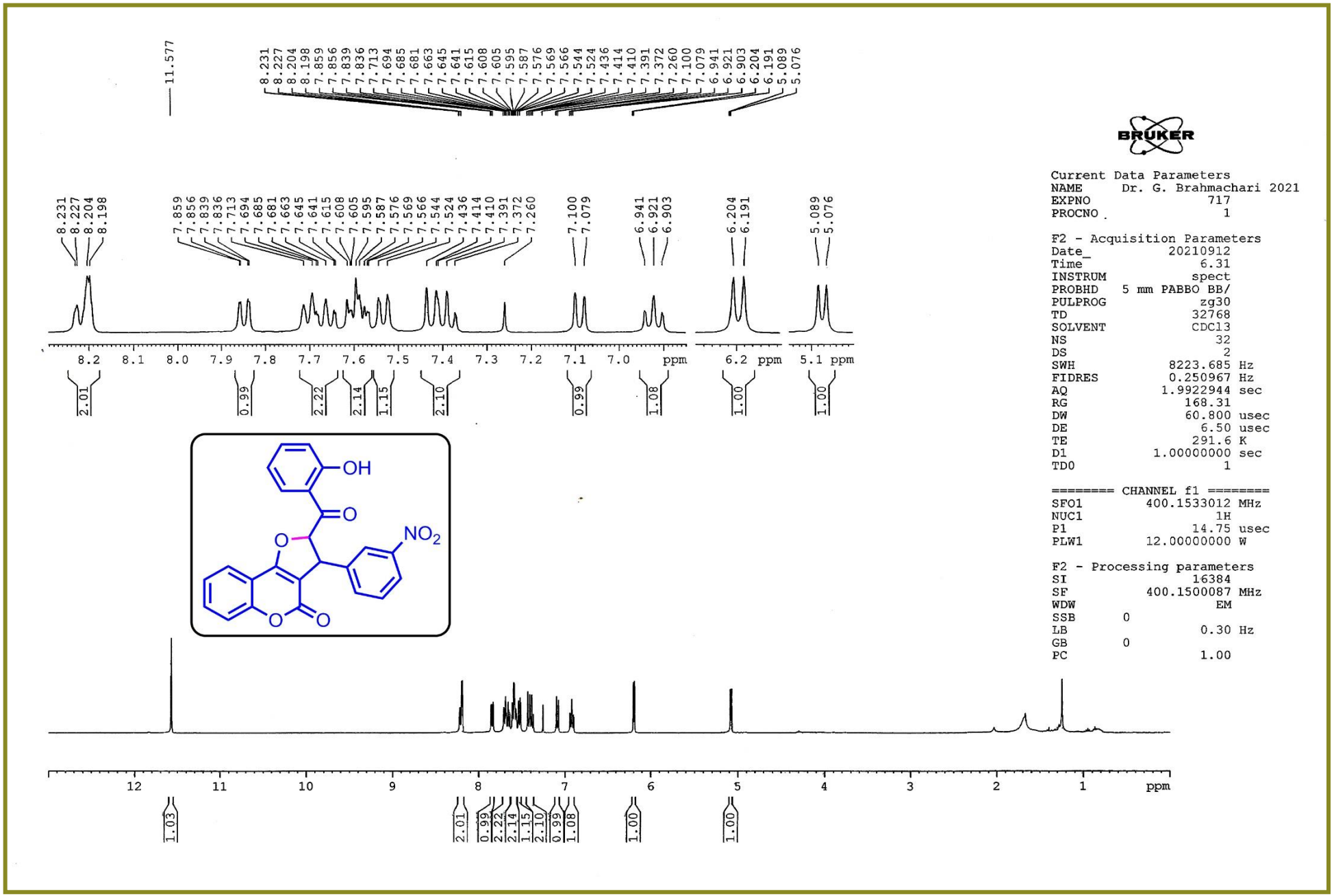


Figure S25. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

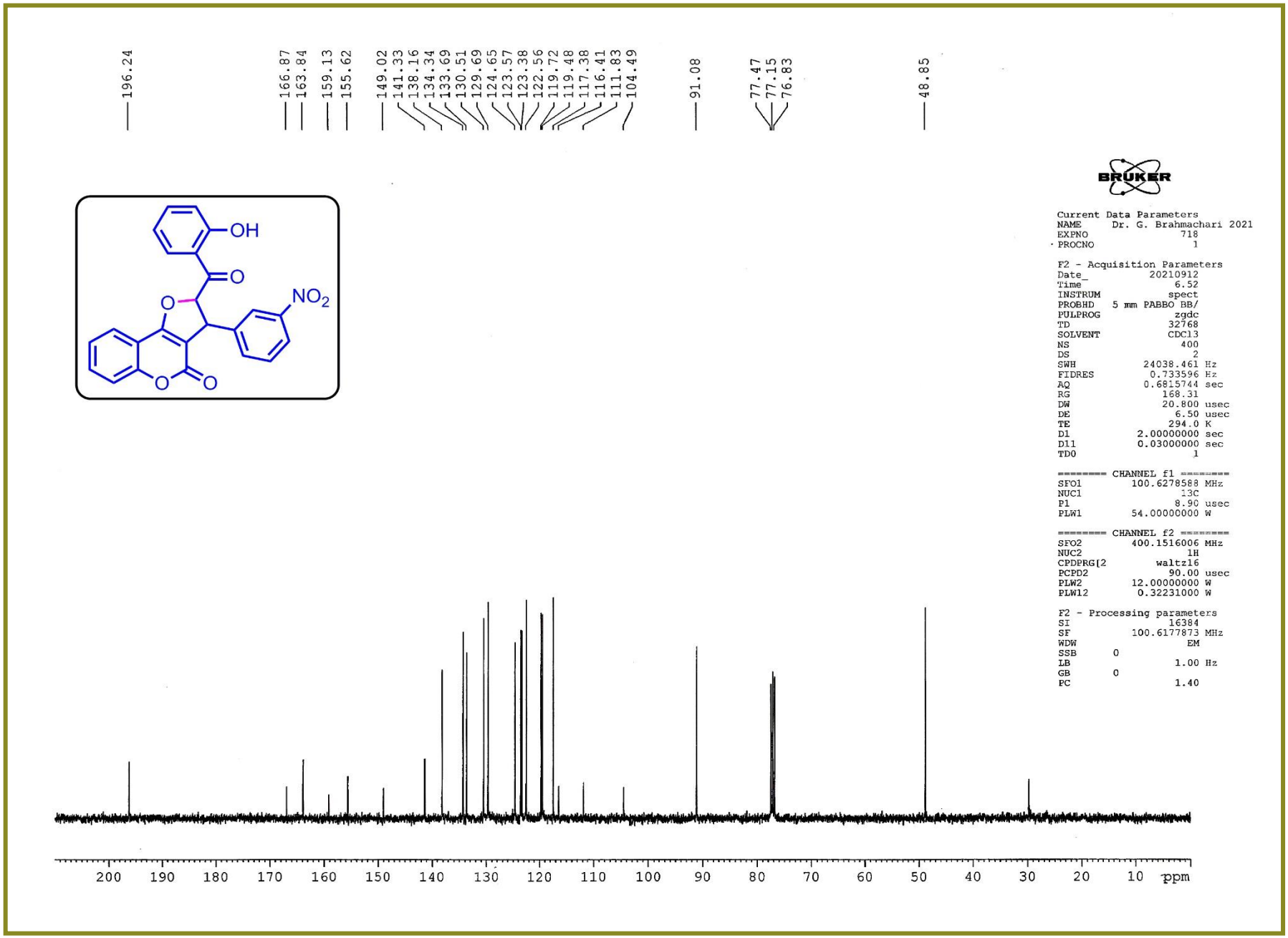


Figure S26. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

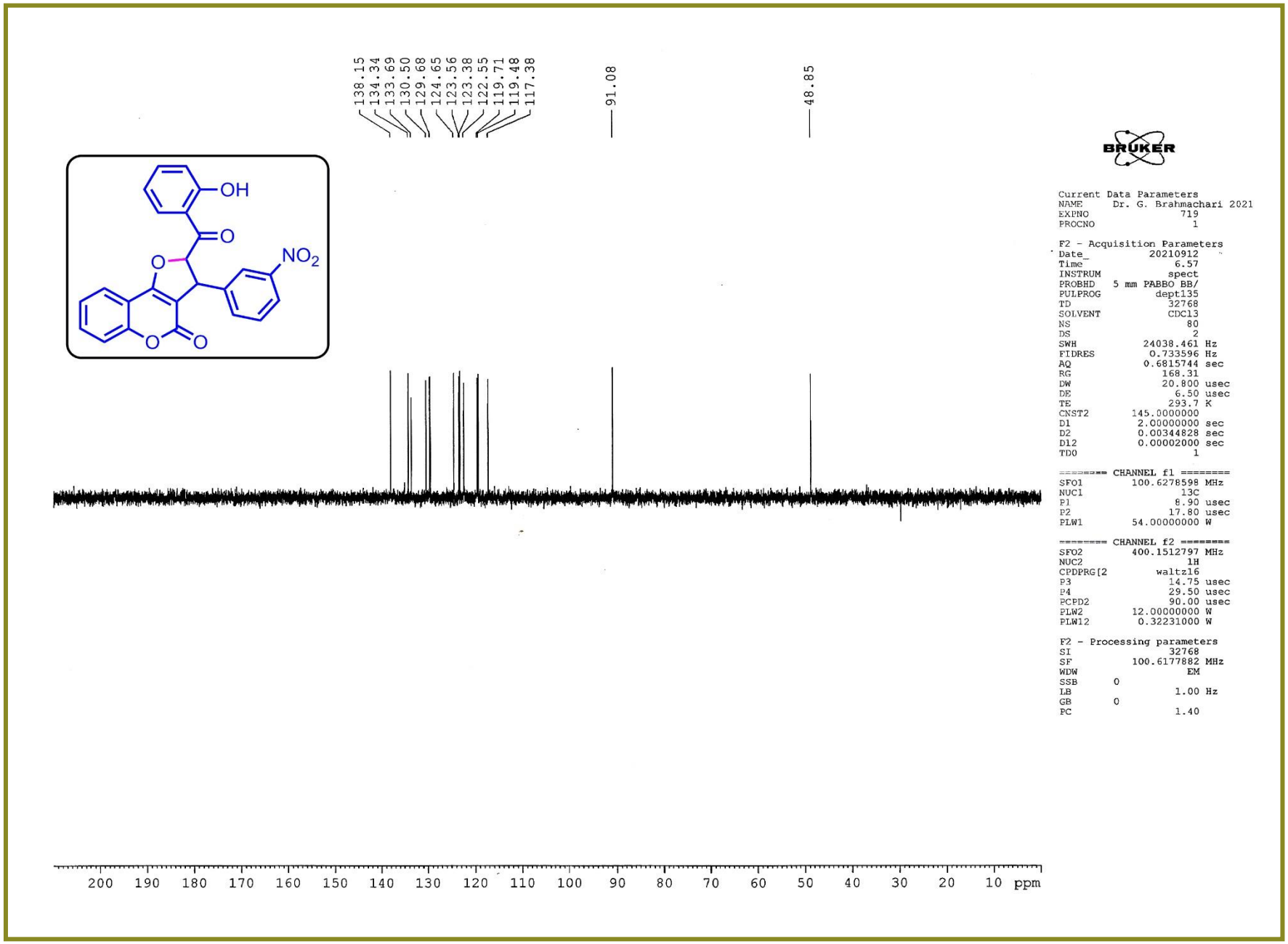


Figure S27. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

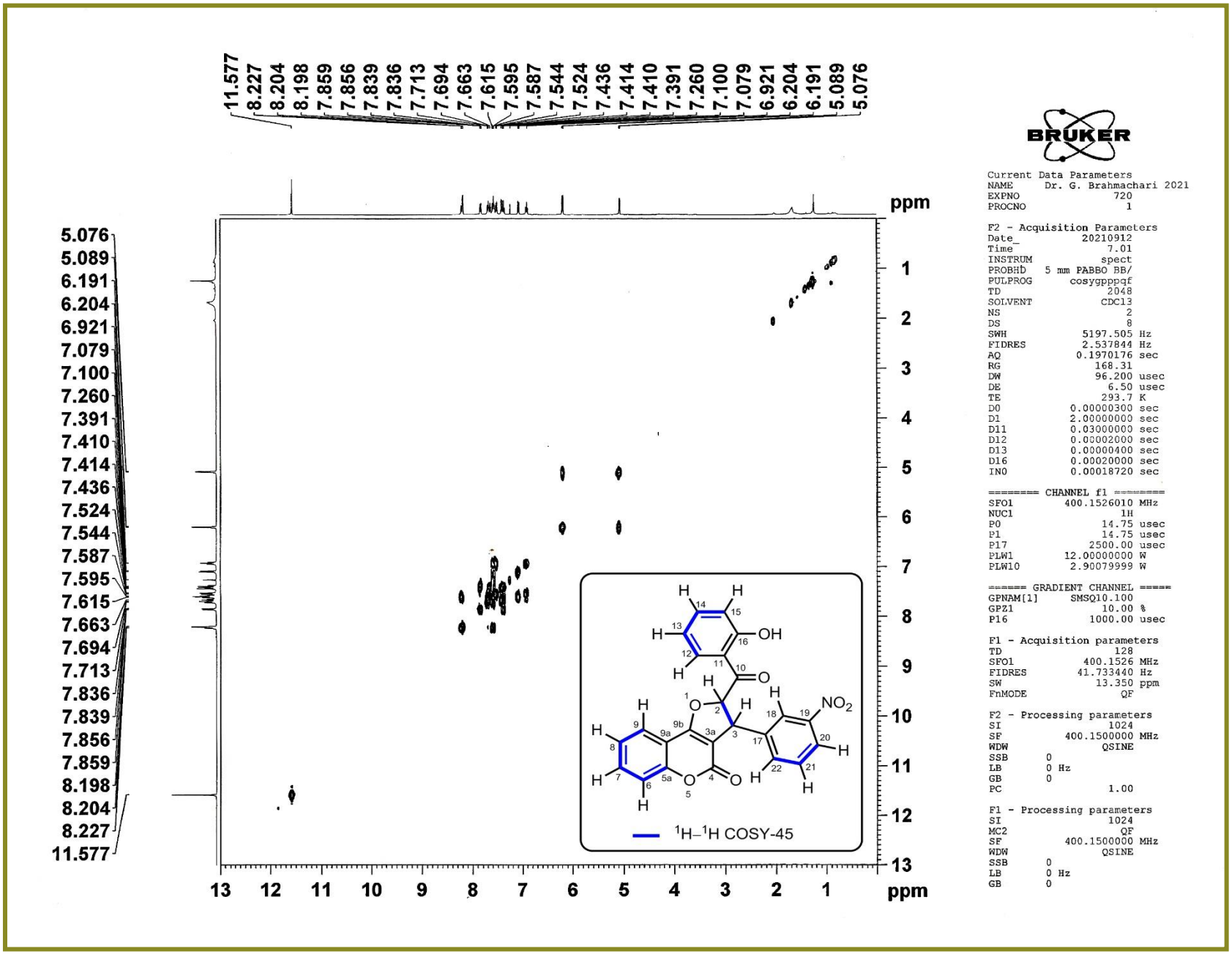


Figure S28. ^{1}H–^{1}H COSY-45 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

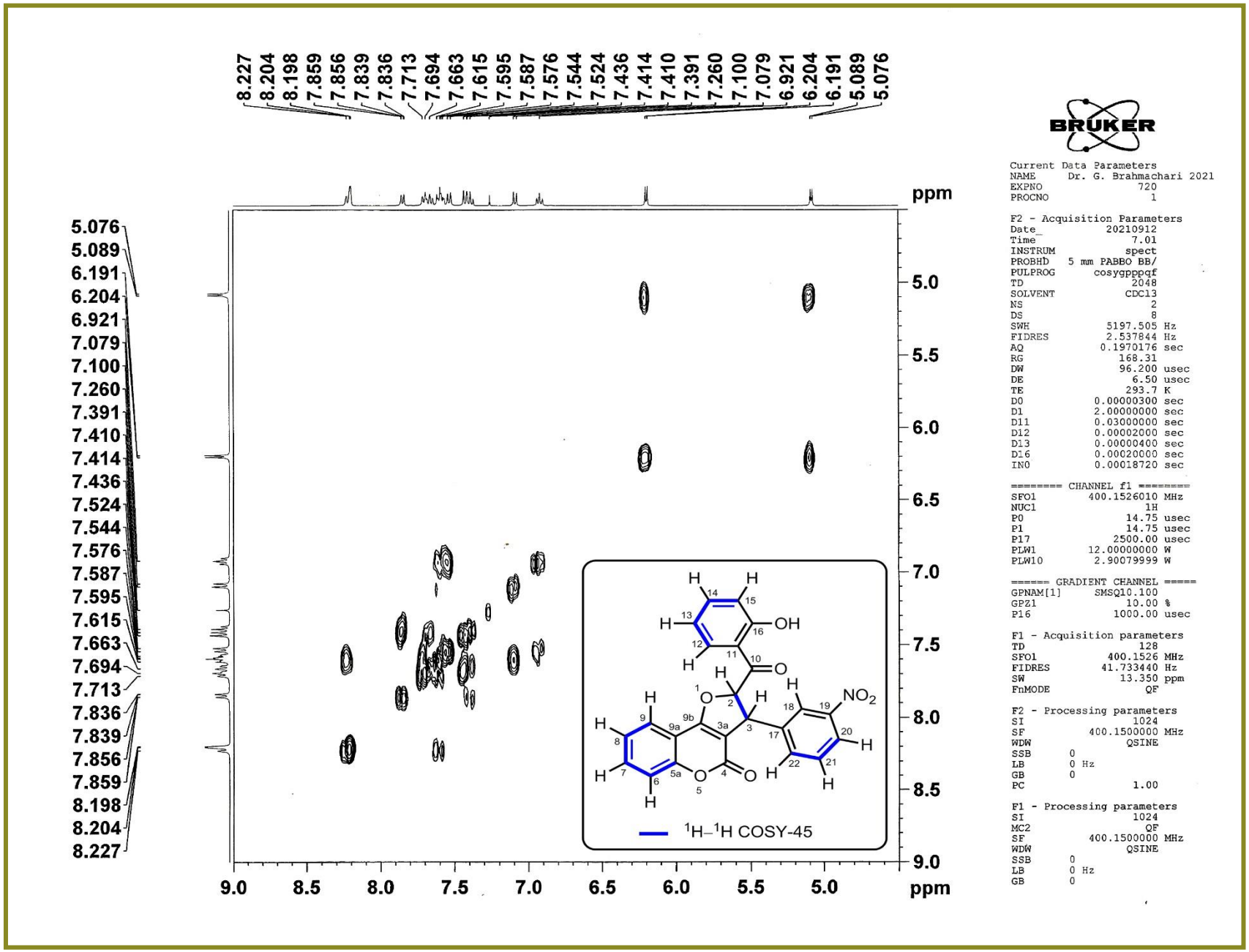


Figure S28a. ^{1}H–^{1}H COSY 45 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**) [Extended-1]

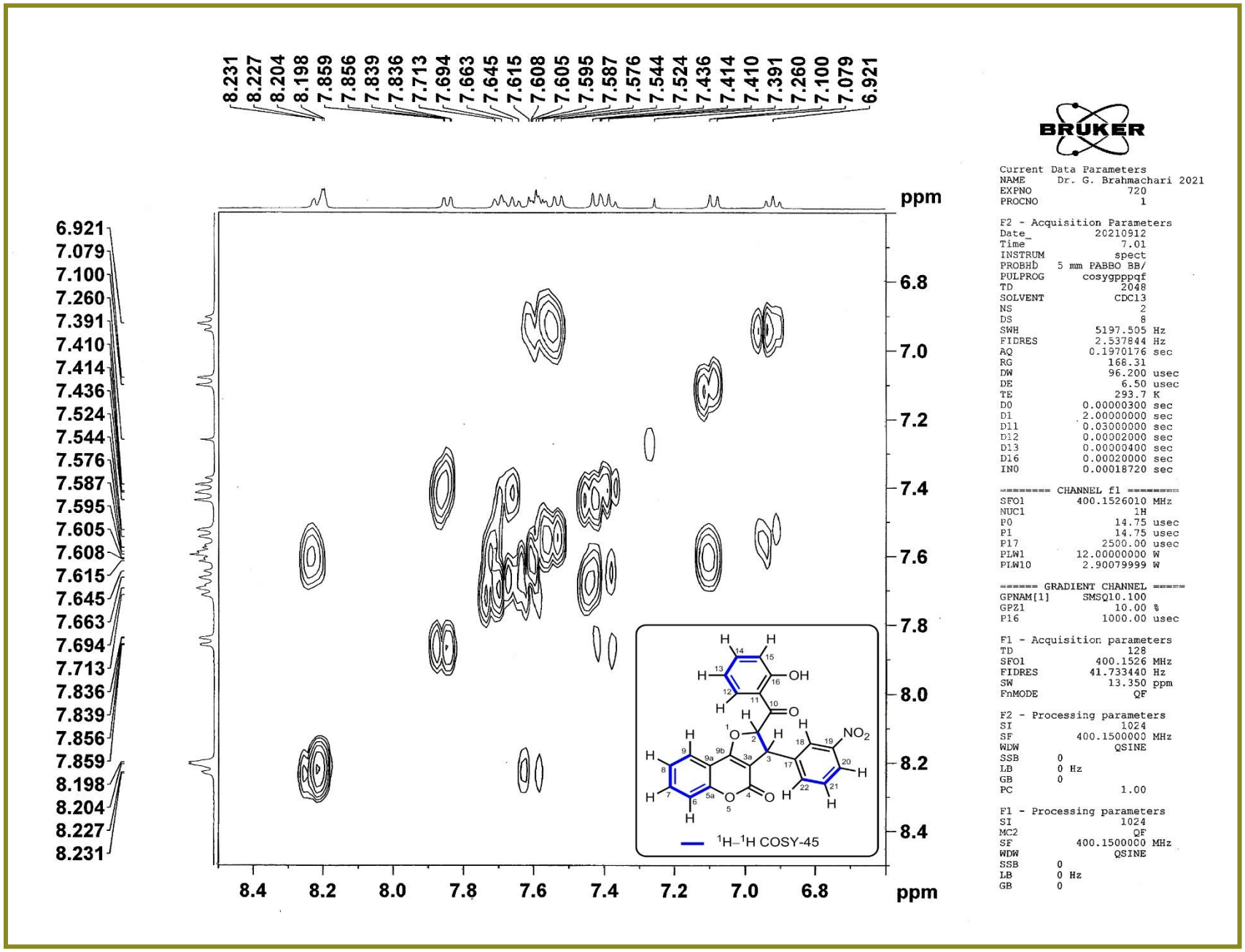


Figure S28b. ^{1}H–^{1}H COSY 45 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**) [Extended-2]

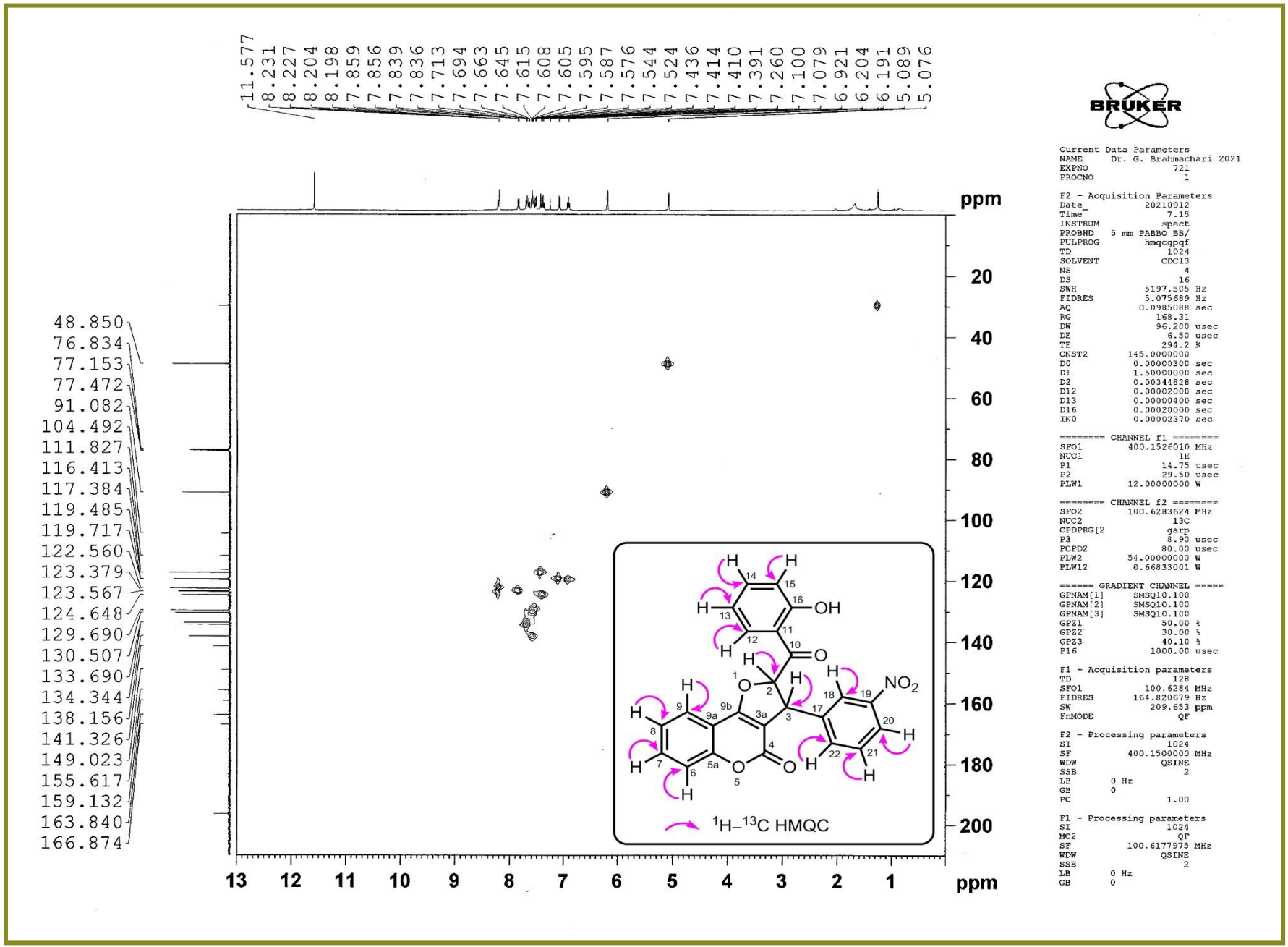


Figure S29. ^{1}H–^{13}C HMQC NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

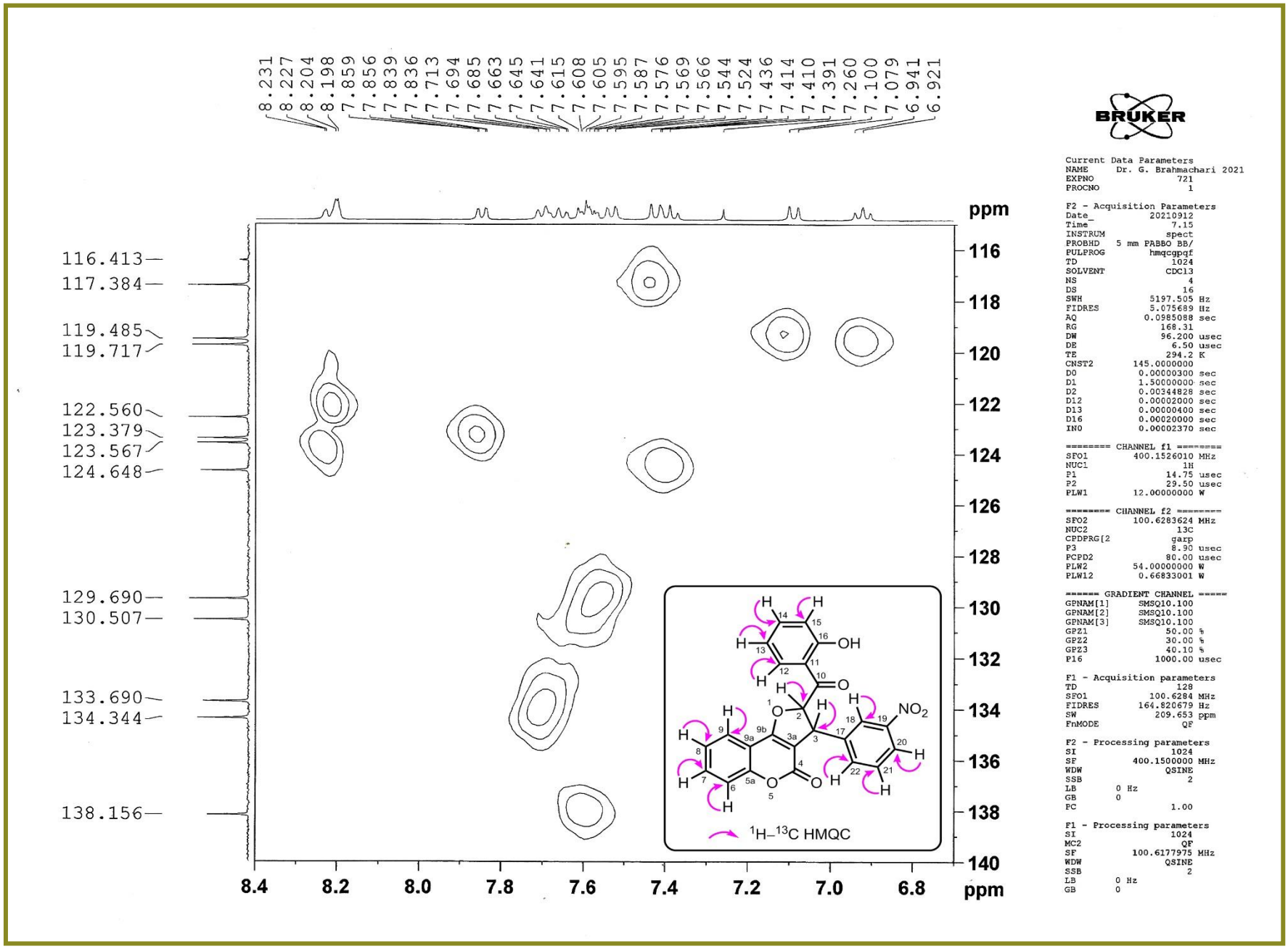


Figure S29a. ^{1}H–^{13}C HMQC NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**) [Extended-1]

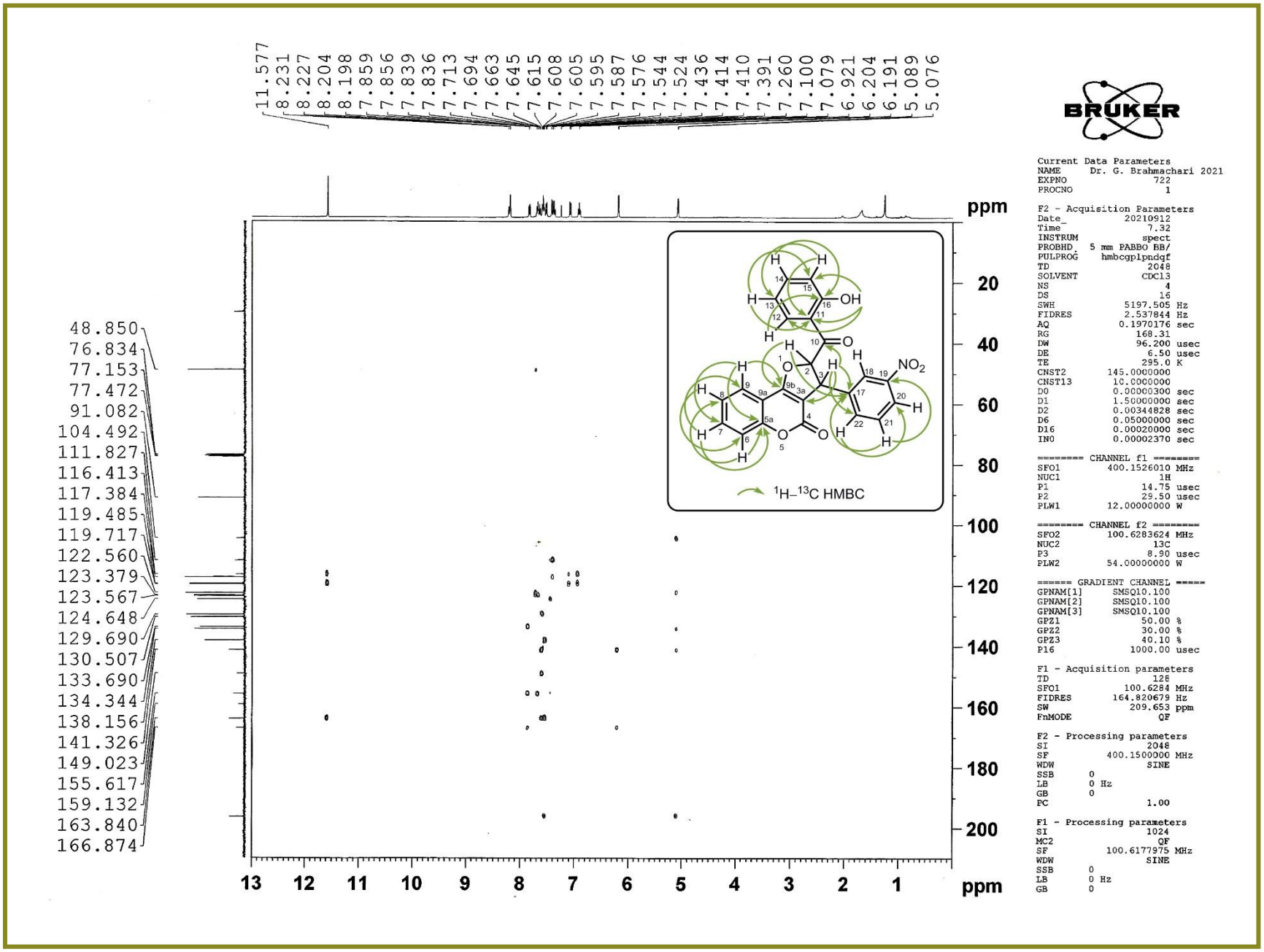


Figure S30. $^{1}H–^{13}C$ HMBC NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

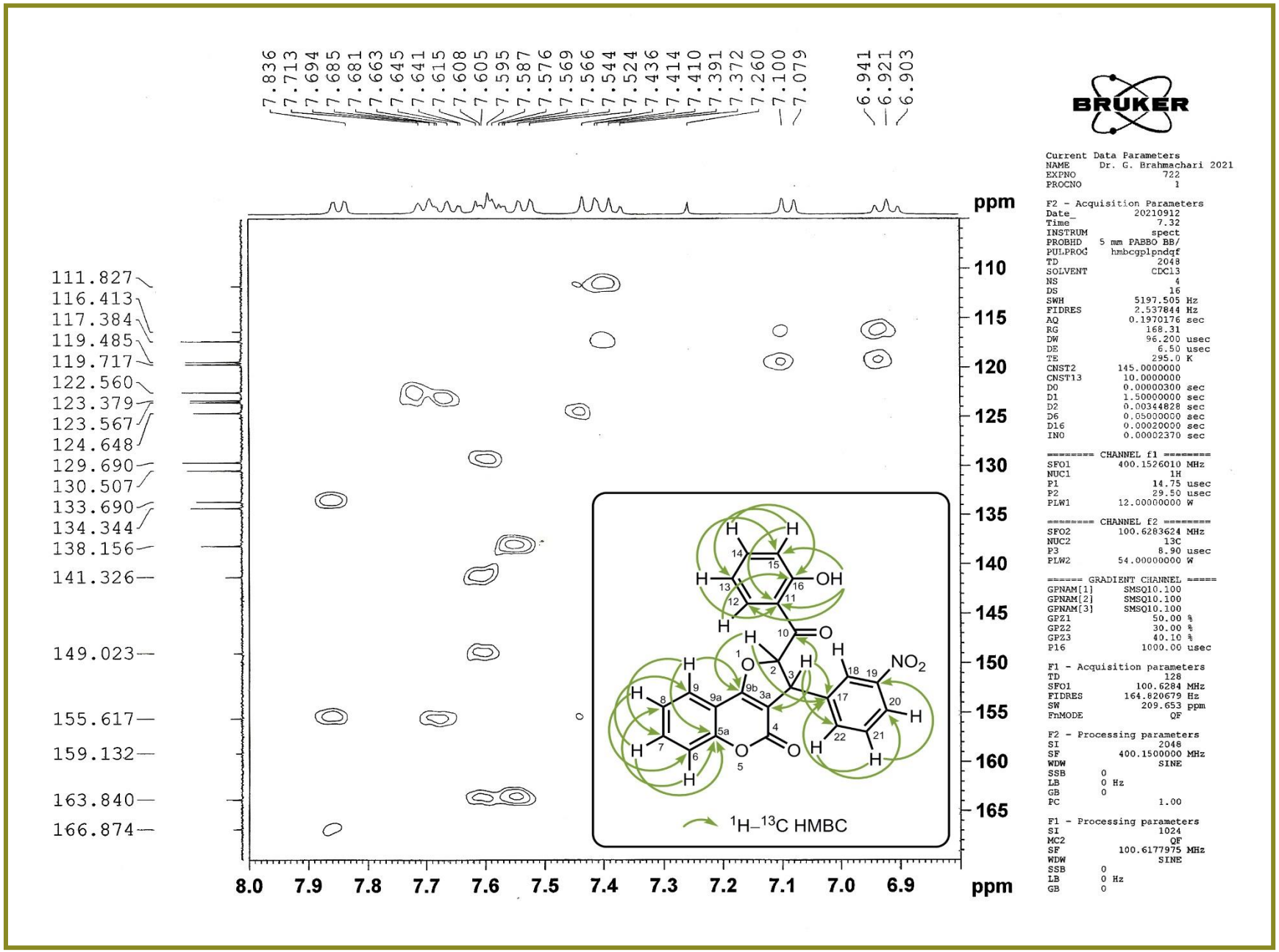


Figure S30a. ^{1}H–^{13}C HMBC NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**) [Extended-1]

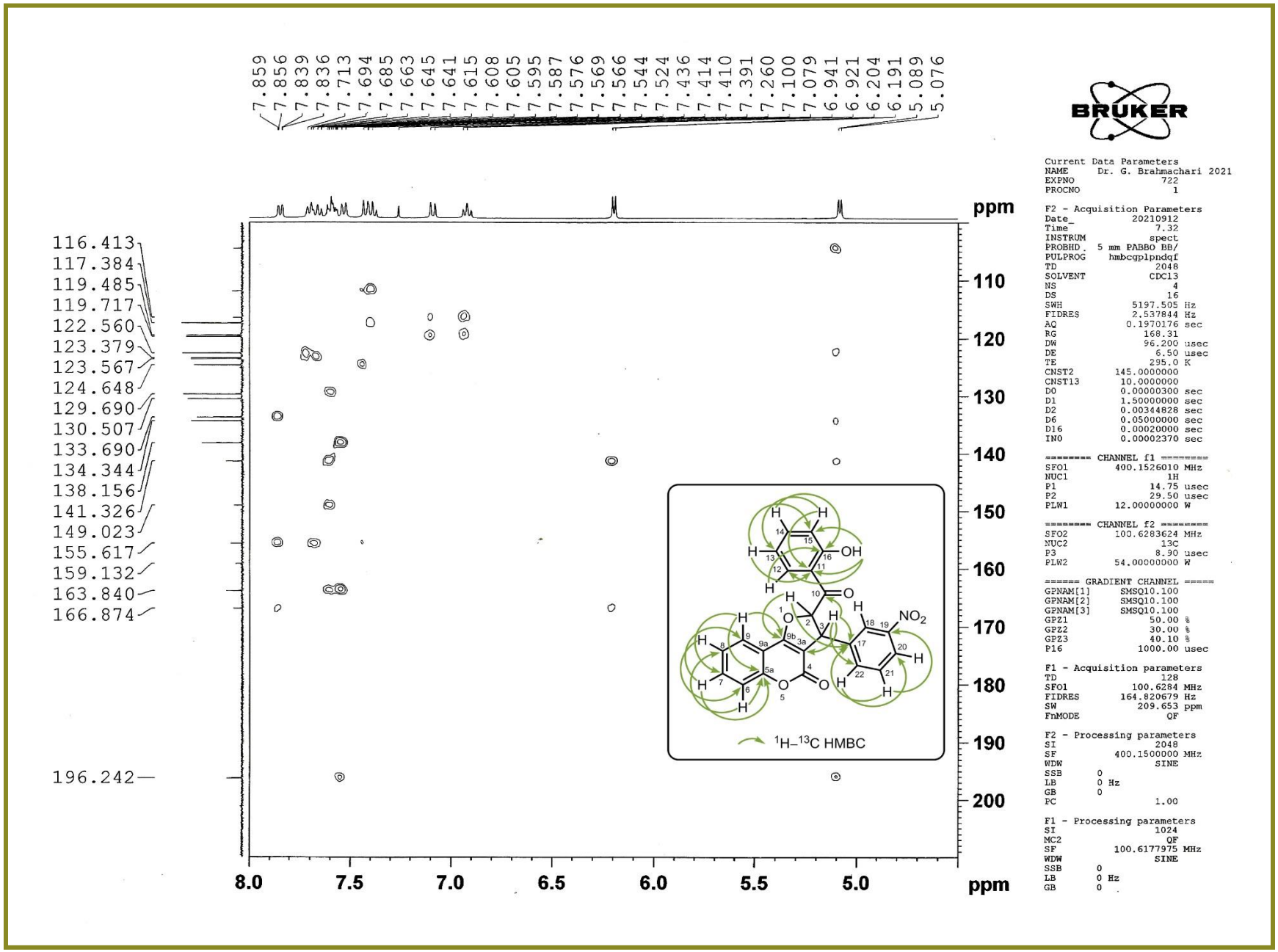
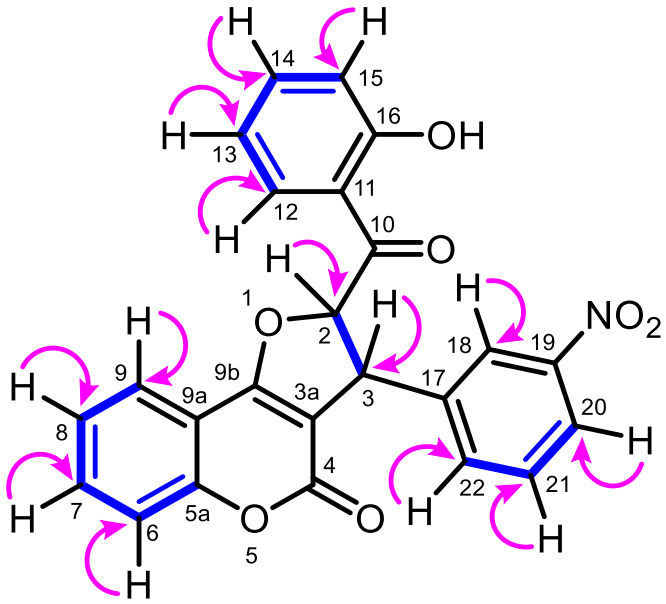
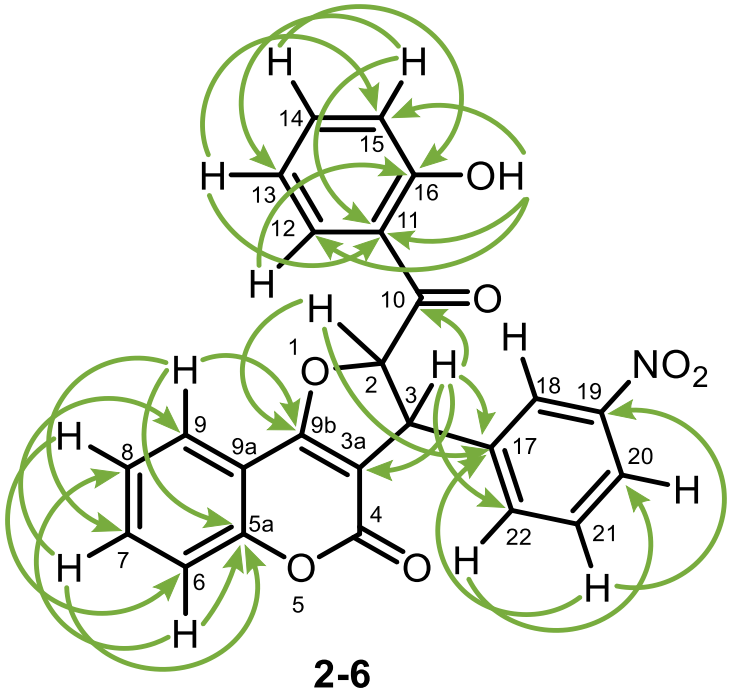
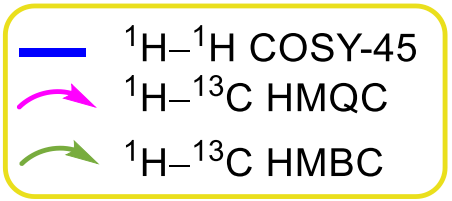


Figure S30b. ^{1}H–^{13}C HMBC NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**) [Extended-2]

Table S1. 2D-NMR properties of the representative compound, 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**) showing the corresponding homo- and hetero-nuclear interactions







Carbon	^{1}H (ppm/δ)	^{13}C (ppm/δ)	DEPT-135	^{1}H-^{1}H COSY-45	^{1}H-^{13}C HMQC	^{1}H-^{13}C HMBC
C-2	6.19 (d, 1H, *J* = 5.2 Hz, ArCOC*H*<)	91.08	CH	δ 6.19 (H-2) *vs* δ 5.08 (H-3)	δ 6.19 (H-2) *vs* δ 91.08 (C-2)	δ 6.19 (H-2) *vs* δ 141.33 (C-17) and 166.87 (C-9b)
C-3	5.08 (d, 1H, *J* = 5.2 Hz, ArC*H*<)	48.85	CH	δ 5.08 (H-3) *vs* δ 6.19 (H-2)	δ 5.08 (H-3) *vs* δ 48.85 (C-3)	δ 5.08 (H-3) *vs* δ 104.49 (C-3a), 134.34 (C-22), 141.33 (C-17) and 196.24 (C-10)
C-3a	–	104.49	C	–	–	–
C-4	–	159.62	C	–	–	–

C-5a	–	155.62	C	–	–	–
C-6	7.44-7.37 (m, 2H, Ar-H)	117.38	CH	δ 7.44-7.37 (H-6) *vs* δ 7.71-7.64 (H-7)	δ 7.44-7.37 (H-6) *vs* δ 117.38 (C-6)	δ 7.44-7.37 (H-6) *vs* δ 124.65 (C-8) and 155.62 (C-5a)
C-7	7.71-7.64 (m, 2H, Ar-H)	133.69	CH	δ 7.71-7.64 (H-7) *vs* δ 7.44-7.37 (H-6 and H-8)	δ 7.71-7.64 (H-7) *vs* δ 133.69 (C-7)	δ 7.71-7.64 (H-7) *vs* δ 123.38 (C-9) and 155.62 (C-5a)
C-8	7.44-7.37 (m, 2H, Ar-H)	124.65	CH	δ 7.44-7.37 (H-8) *vs* δ 7.71-7.64 (H-7) and δ 7.85 (H-9)	δ 7.44-7.37 (H-8) *vs* δ 124.65 (C-8)	δ 7.44-7.37 (H-8) *vs* δ 111.83 (C-9a) and 117.38 (C-6)
C-9	7.85 (dd, 1H, *J* = 8.0 & 1.2 Hz, Ar-H)	123.38	CH	δ 7.85 (H-9) *vs* δ 7.44-7.37 (H-8)	δ 7.85 (H-9) *vs* δ 123.38 (C-9)	δ 7.85 (H-9) *vs* δ 133.69 (C-7), 155.62 (C-5a) and 166.87 (C-9b)
C-9a	–	111.83	C	–	–	–
C-9b	–	166.87	C	–	–	–
C-10	–	196.24	C	–	–	–
C-11	–	116.15	C	–	–	–
C-12	7.62-7.57 (m, 2H, Ar-H)	129.69	CH	δ 7.62-7.57 (H-12) *vs* δ 7.09 (H-13)	δ 7.62-7.57 (H-12) *vs* δ 129.69 (C-12)	δ 7.62-7.57 (H-12) *vs* δ 163.84 (C-16)
C-13	7.09 (d, 1H, *J* = 8.4 Hz, Ar-H)	119.49	CH	δ 7.09 (H-13) *vs* δ 7.62-7.57 (H-12) and 7.53 (H-14)	δ 7.09 (H-13) *vs* δ 119.49 (C-13)	δ 7.09 (H-13) *vs* δ 116.15 (C-11) and 119.72 (C-15)
C-14	7.53 (d, 1H, *J* = 8.0 Hz, Ar-H)	138.16	CH	δ 7.53 (H-14) *vs* δ 7.09 (H-13) and 6.94-6.90 (H-15)	δ 7.53 (H-14) *vs* δ 138.16 (C-14)	δ 7.53 (H-14) *vs* δ 163.84 (C-16)
C-15	6.94-6.90 (m, 1H, Ar-H)	119.72	CH	δ 6.94-6.90 (H-15) *vs* δ 7.53 (H-14)	δ 6.94-6.90 (H-15) *vs* δ 119.72 (C-15)	δ 6.94-6.90 (H-15) *vs* δ 116.15 (C-11) and 119.49 (C-13)
C-16	–	163.84	C	–	–	–
C-17	–	141.33	C	–	–	–
C-18	8.23-8.19 (m, 2H, Ar-H)	123.57	CH	–	δ 8.23-8.19 (H-18) *vs* δ 124.65 (C-8)	–
C-19	–	149.02	C	–	–	–

C-20	8.23-8.19 (m, 2H, Ar-H)	122.56	CH	δ 8.23-8.19 (H-20) *vs* δ 7.62-7.57 (H-21)	δ 8.23-8.19 (H-20) *vs* δ 122.56 (C-20)	–
C-21	7.62-7.57 (m, 2H, Ar-H)	130.51	CH	δ 7.62-7.57 (H-21) *vs* δ 8.23-8.19 (H-20) and 7.71-7.64 (H-22)	δ 7.62-7.57 (H-21) *vs* δ 130.51 (C-21)	δ 7.62-7.57 (H-21) *vs* δ 141.33 (C-17) and 149.02 (C-19)
C-22	7.71-7.64 (m, 2H, Ar-H)	134.34	CH	δ 7.71-7.64 (H-22) *vs* δ 7.62-7.57 (H-21)	δ 7.71-7.64 (H-22) *vs* δ 134.34 (C-22)	δ 7.71-7.64 (H-22) *vs* δ 122.56 (C-20)
Ar-O*H*	11.58 (s, 1H, Ar-O*H*)	–	–	–	–	δ 11.58 (Ar-O*H*) *vs* δ 116.15 (C-11), 119.72 (C-15) and 163.84 (C-16)

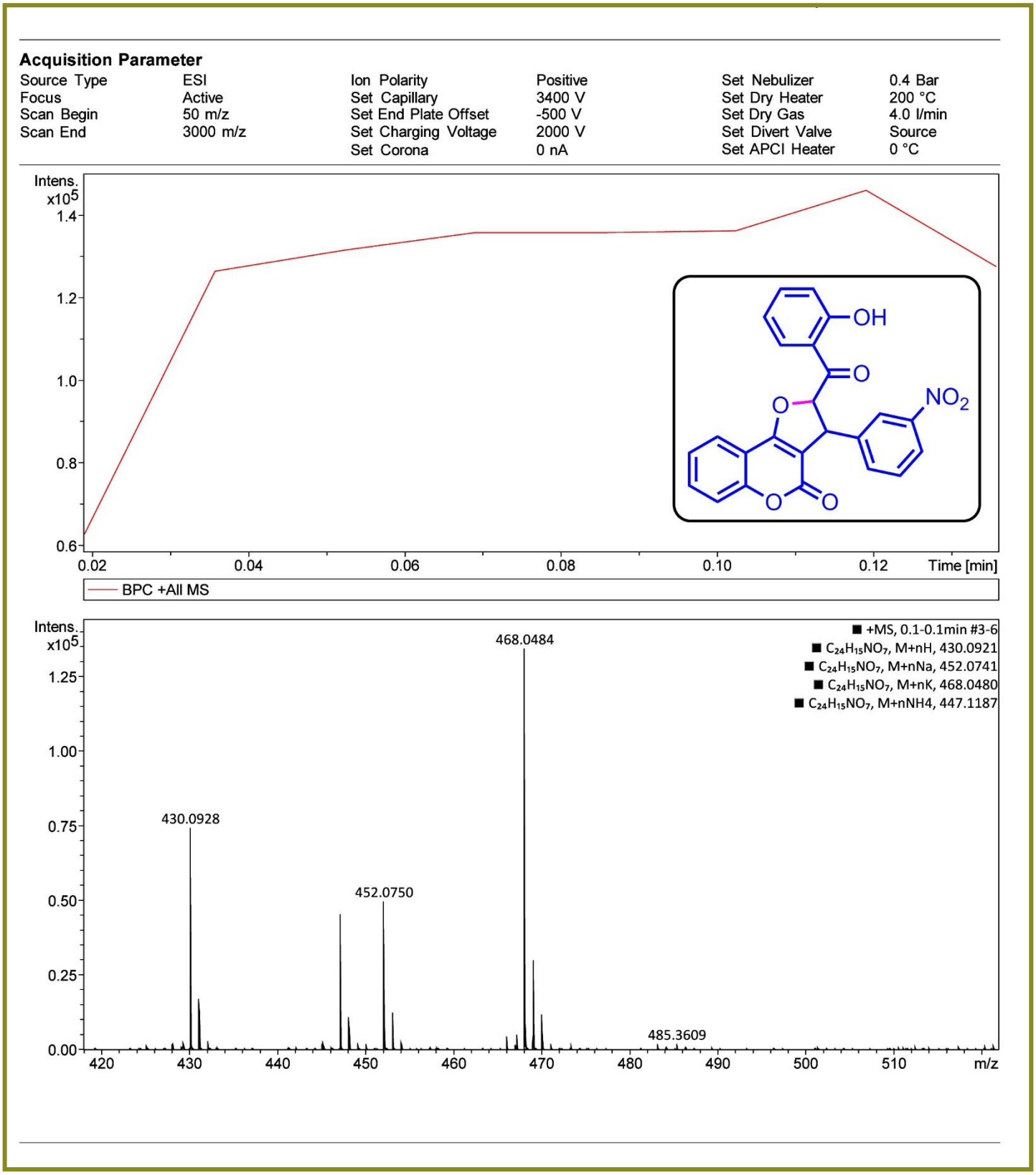


Figure S31. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(3-nitrophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-6**)

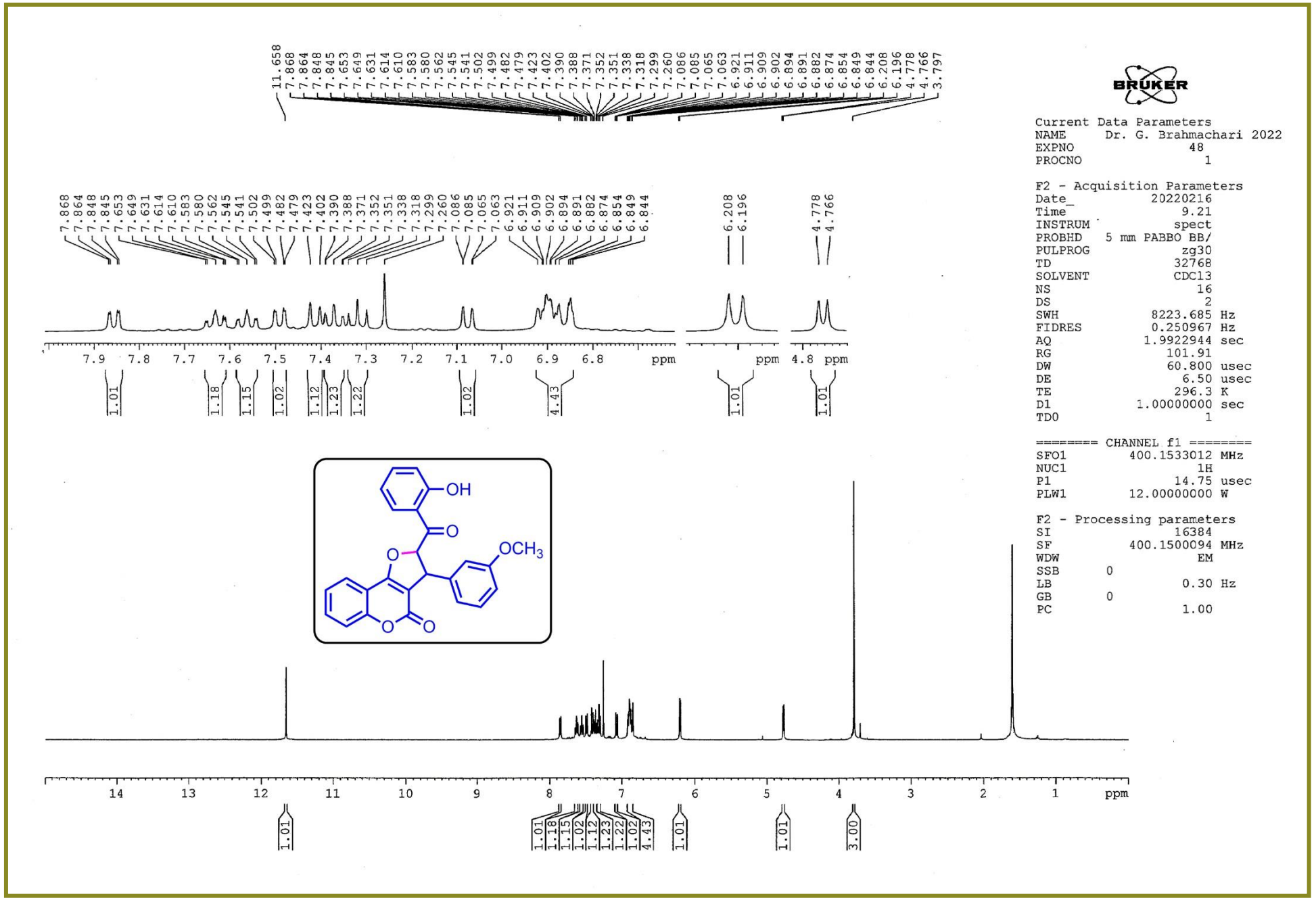


Figure S32. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-7**)

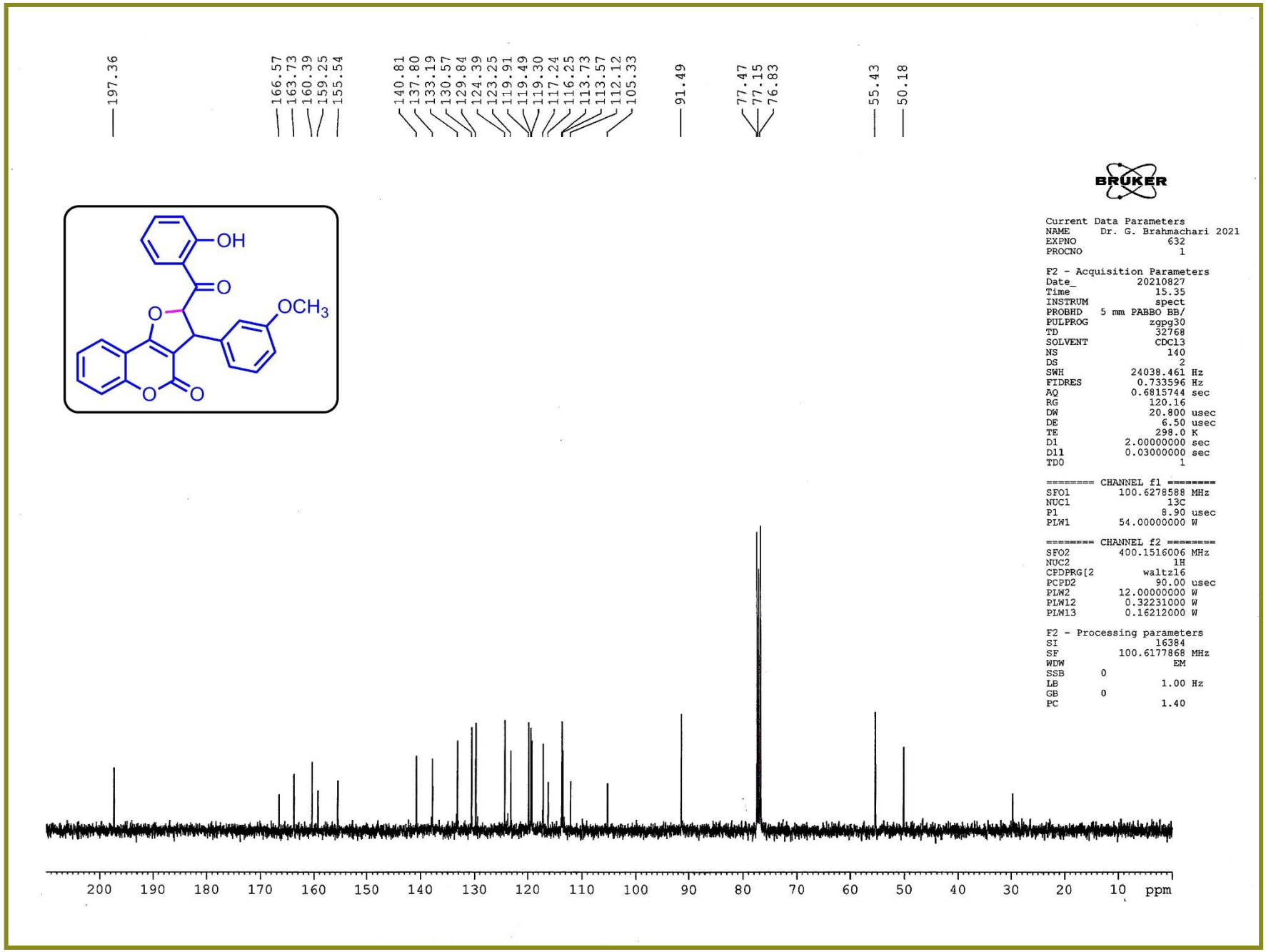


Figure S33. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-7**)

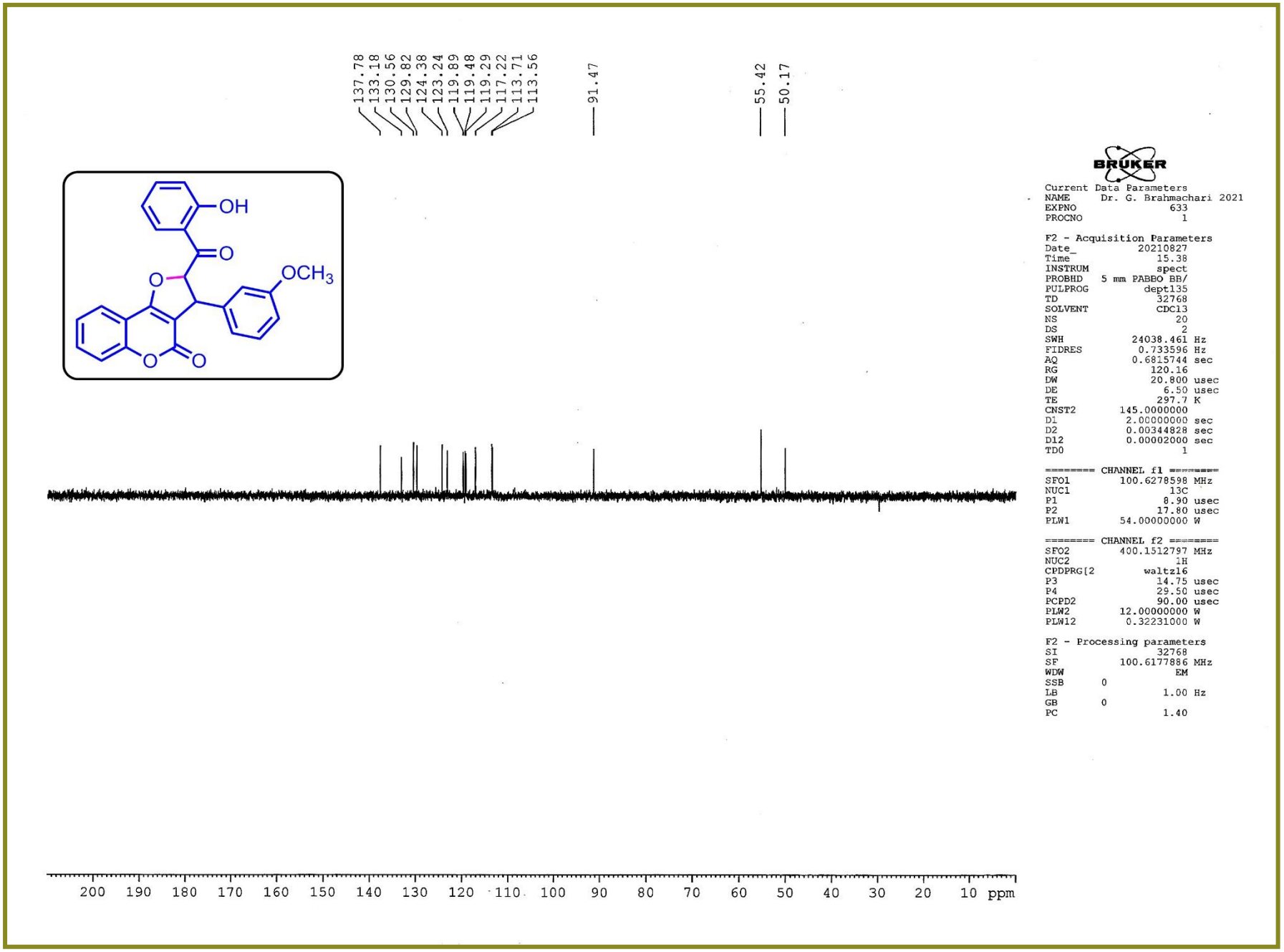


Figure S34. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-7**)

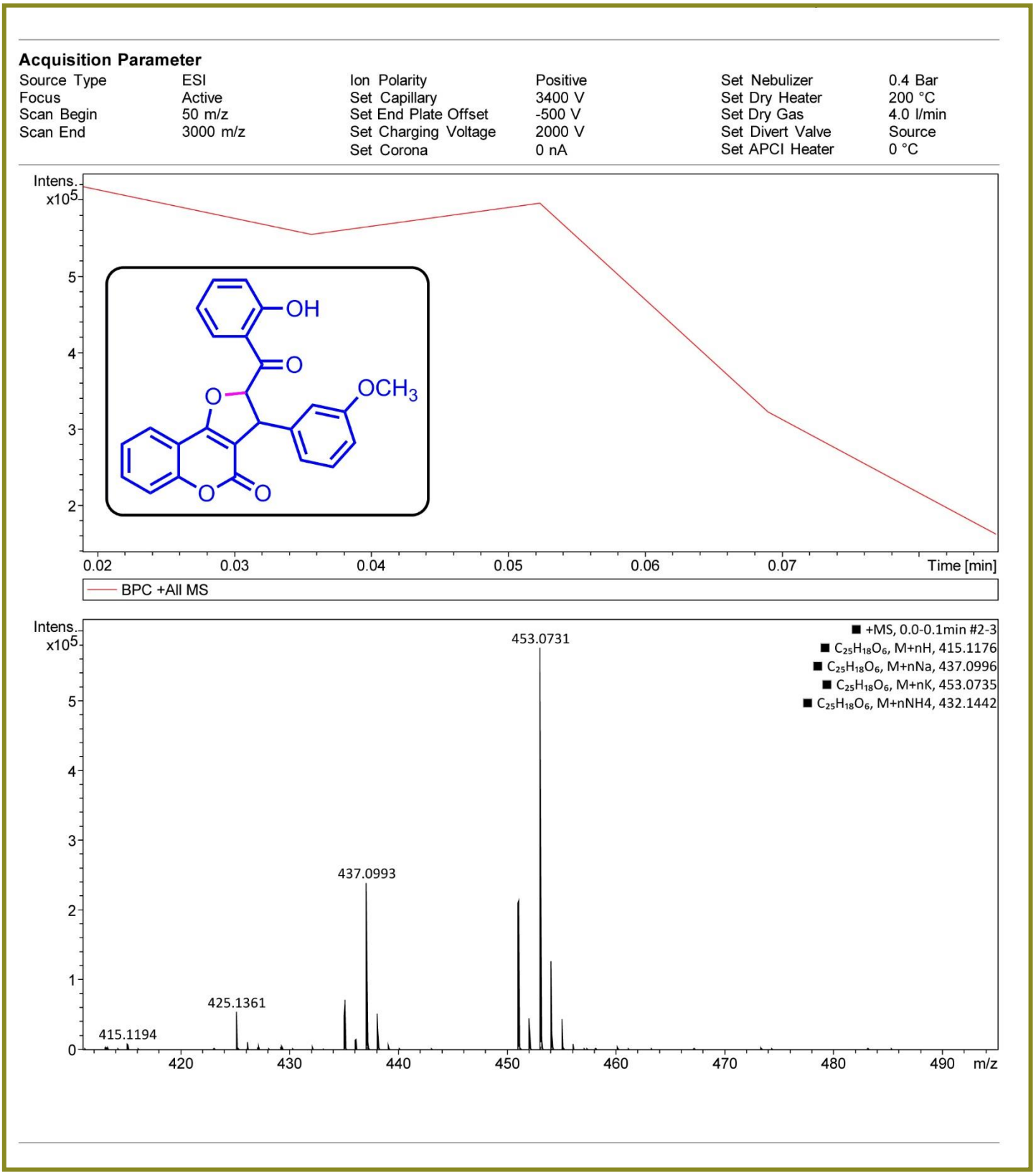


Figure S35. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(3-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-7**)

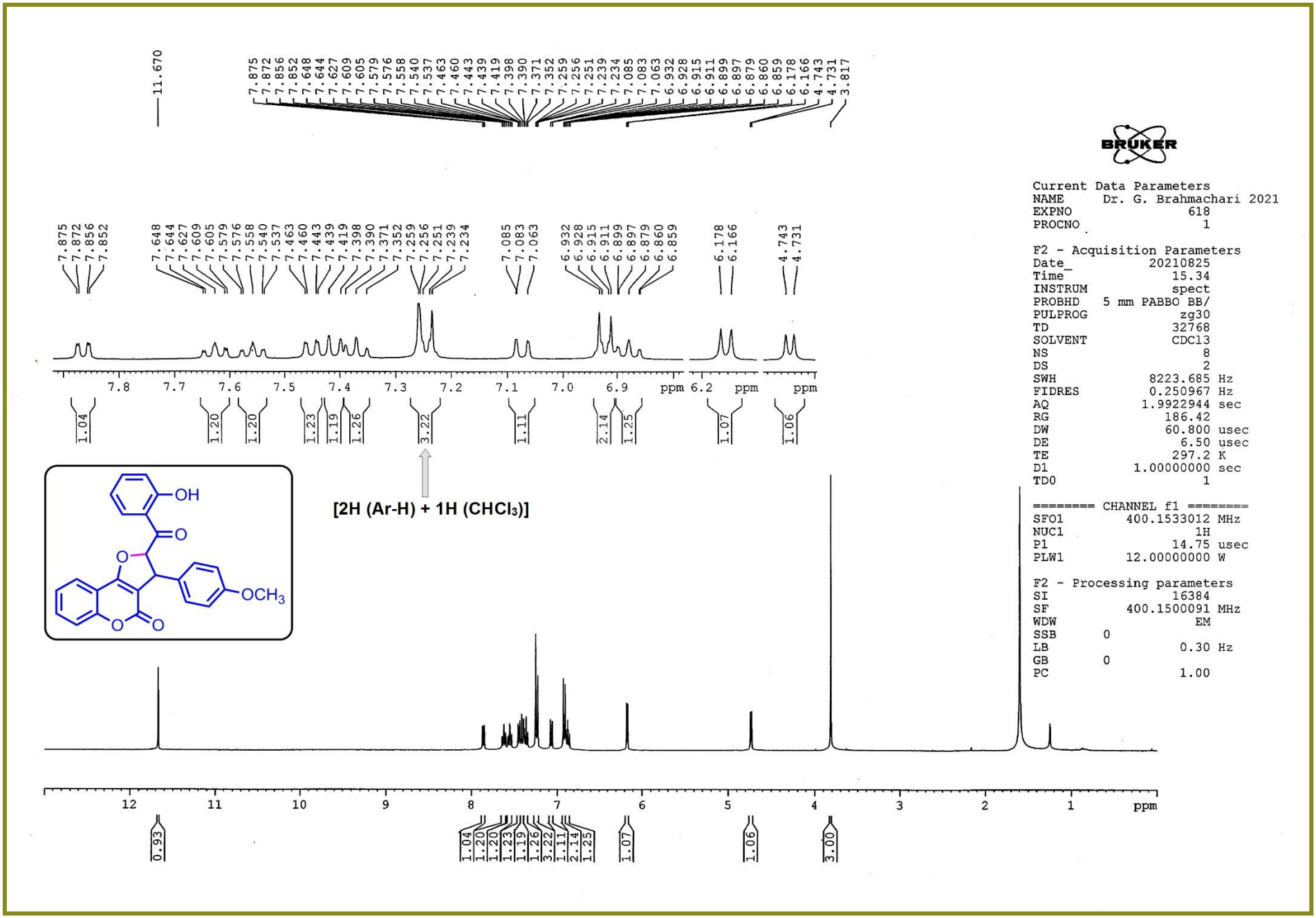


Figure S36. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-8**)

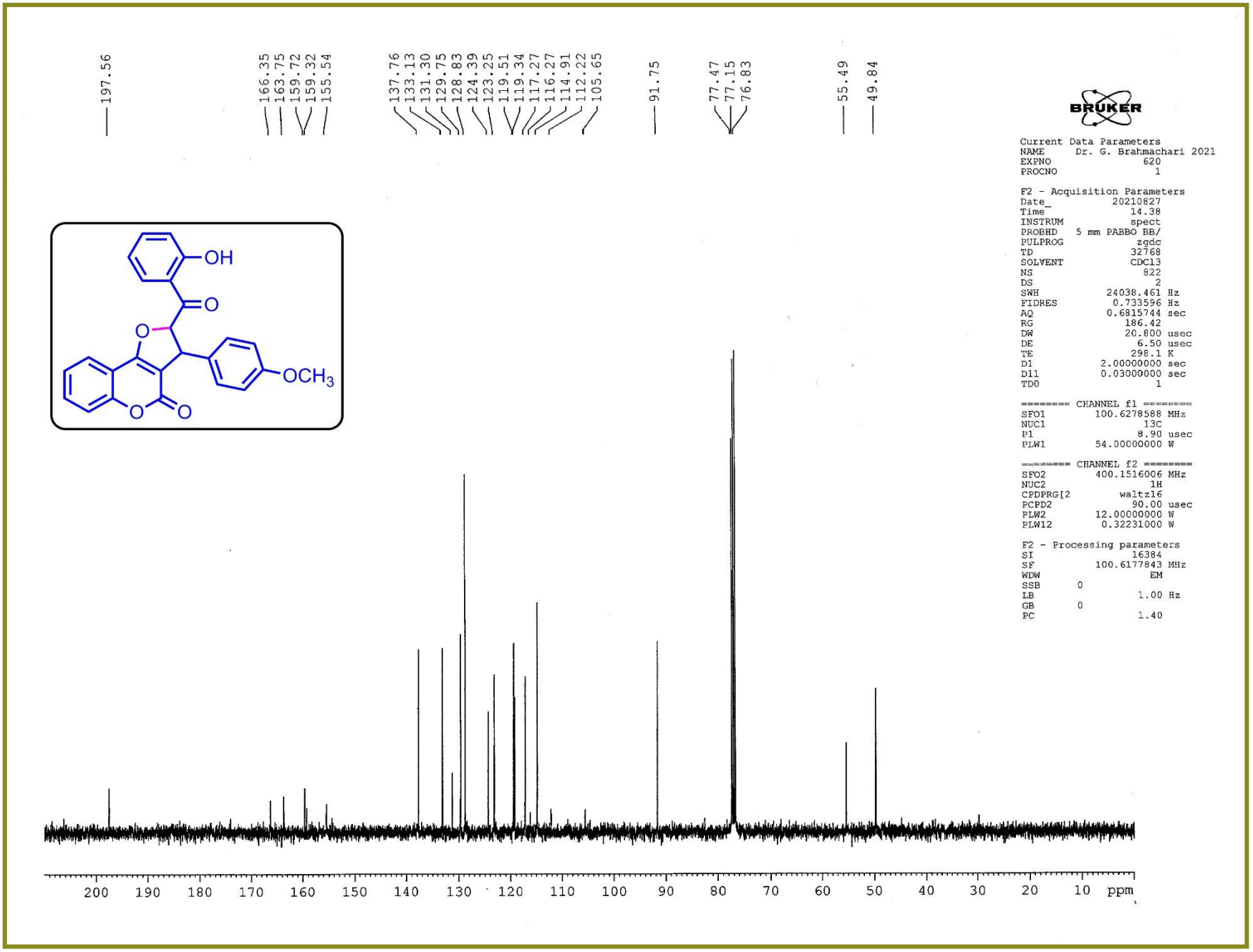


Figure S37. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-8**)

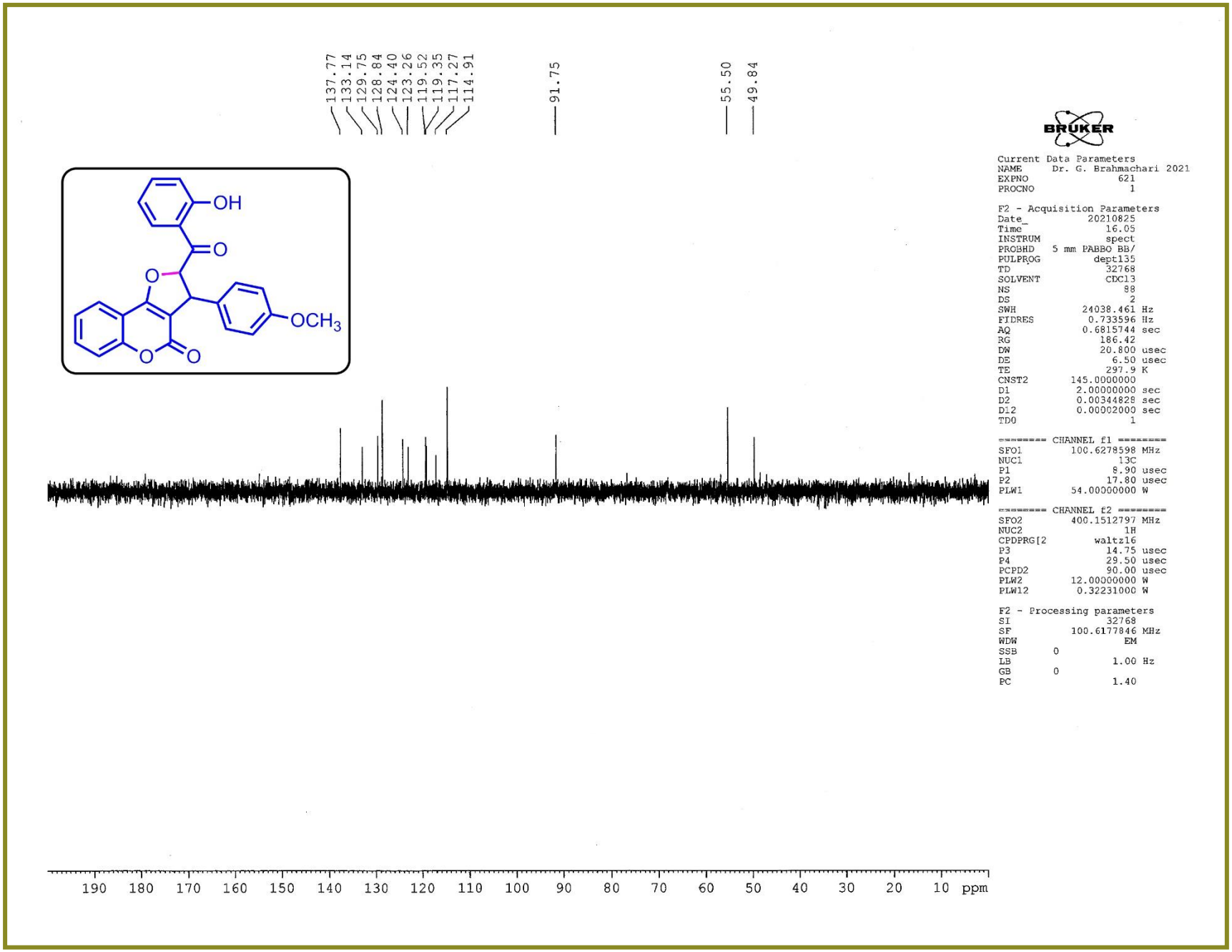


Figure S38. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-8**)

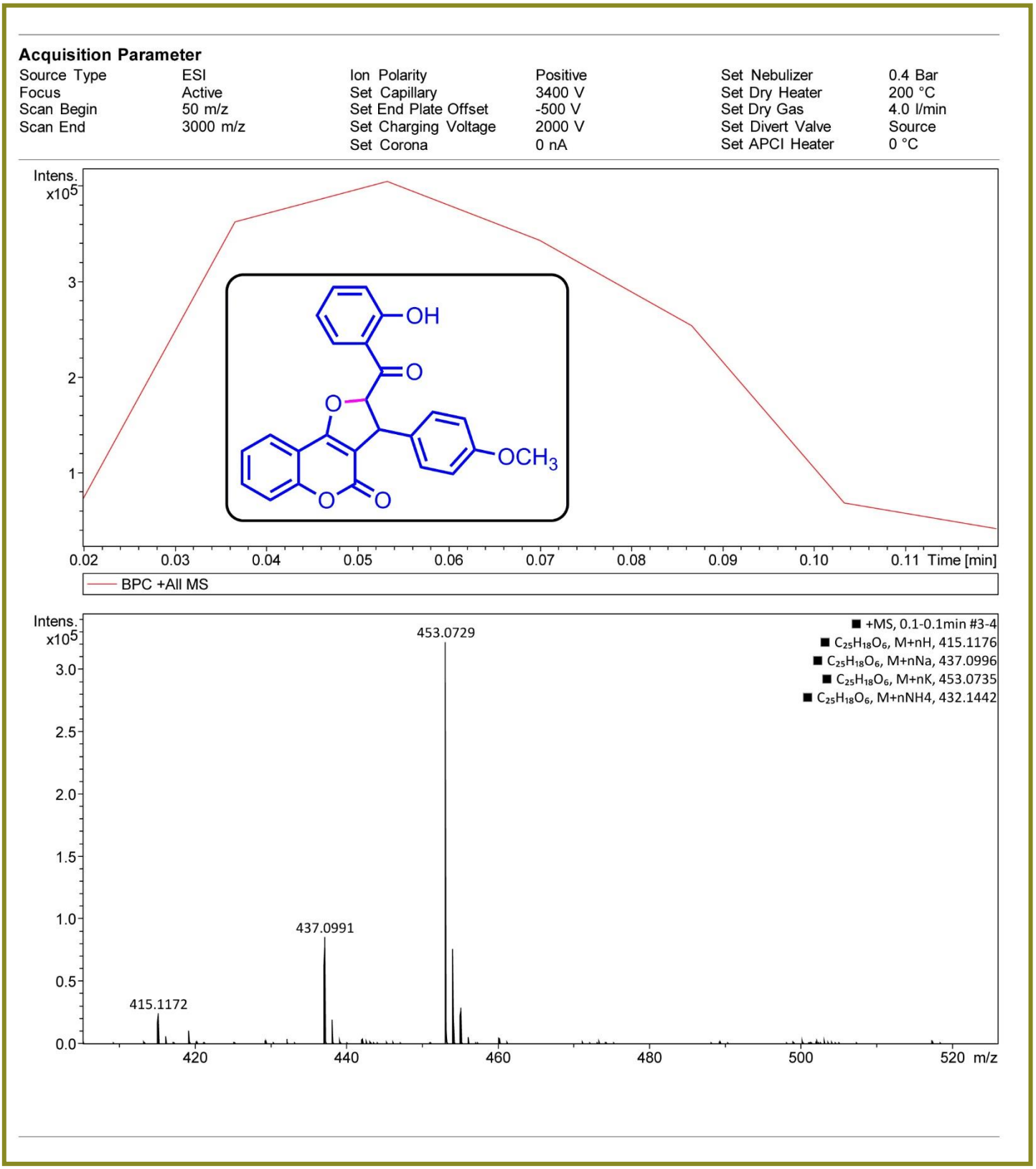


Figure S39. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(4-methoxyphenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-8**)

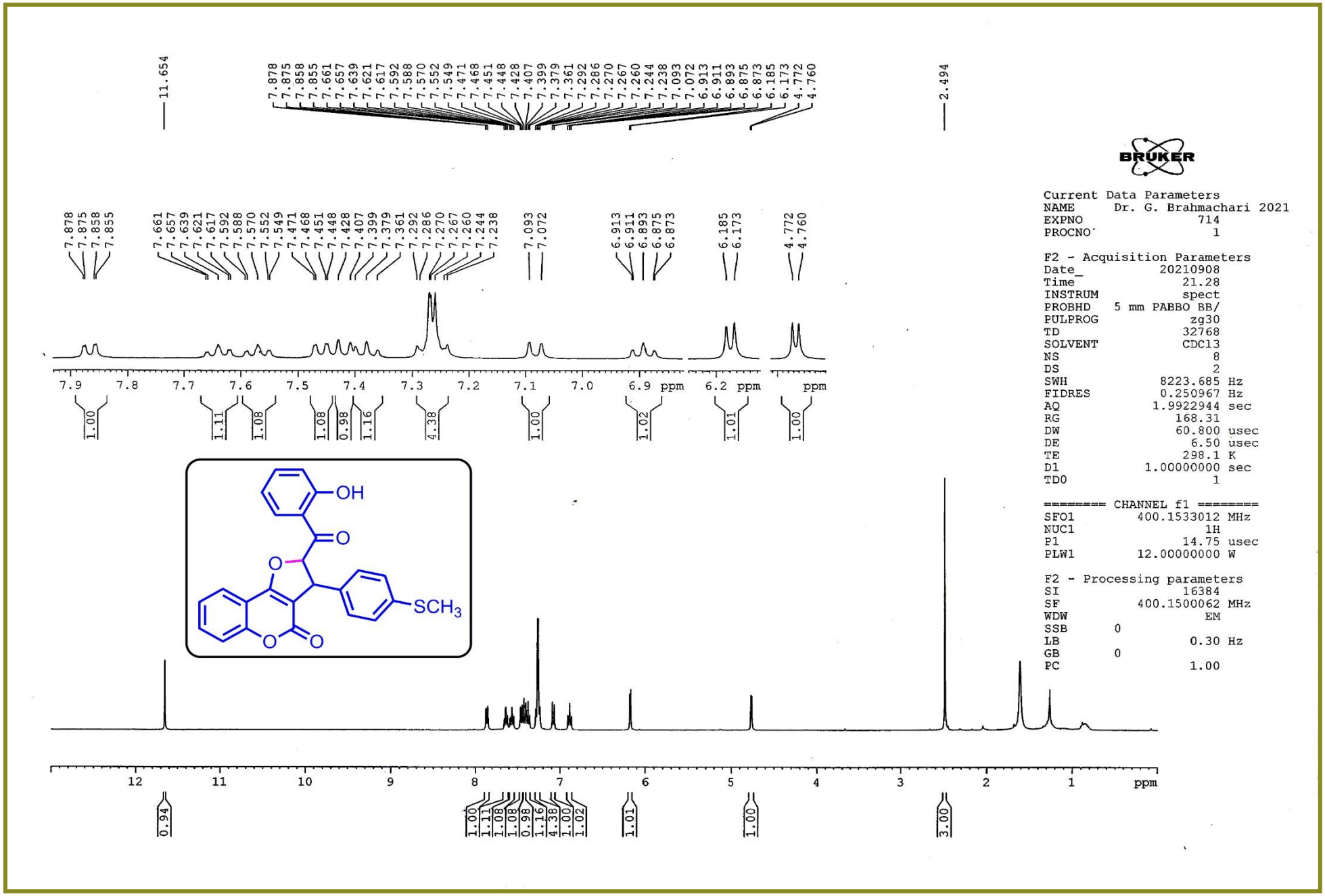


Figure S40. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(methylthio)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-9**)

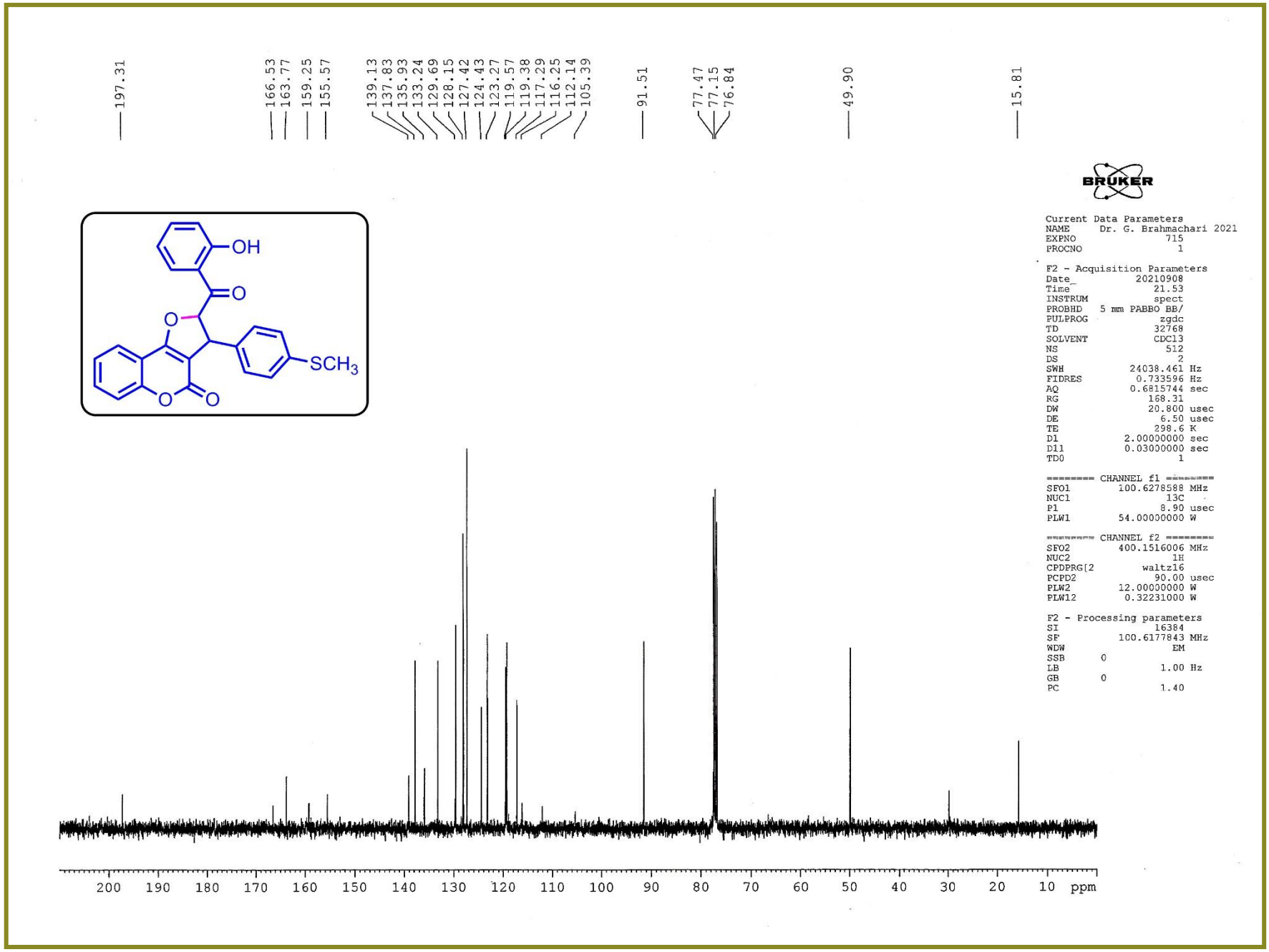


Figure S41. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(methylthio)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-9**)

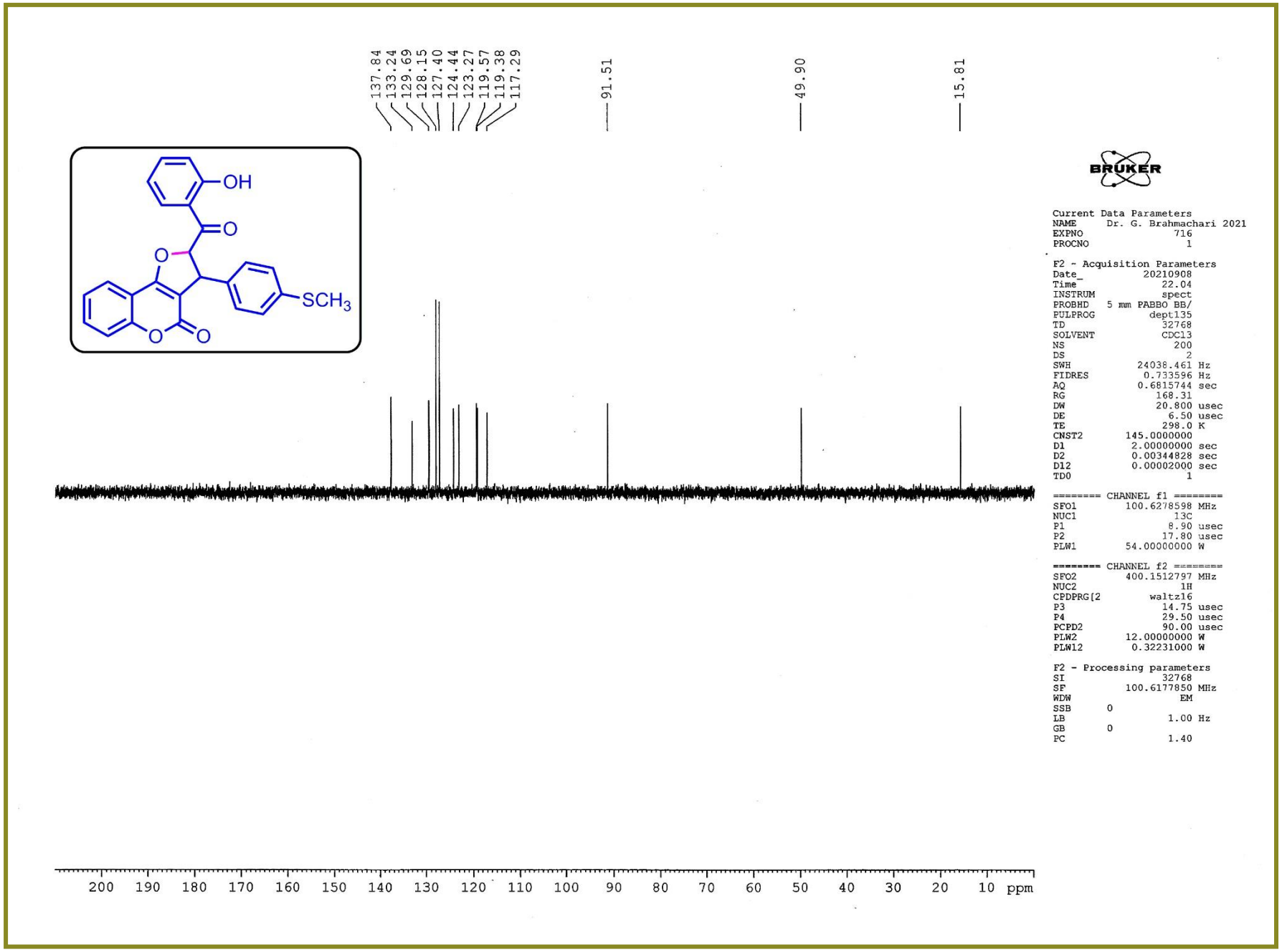


Figure S42. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(methylthio)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-9**)

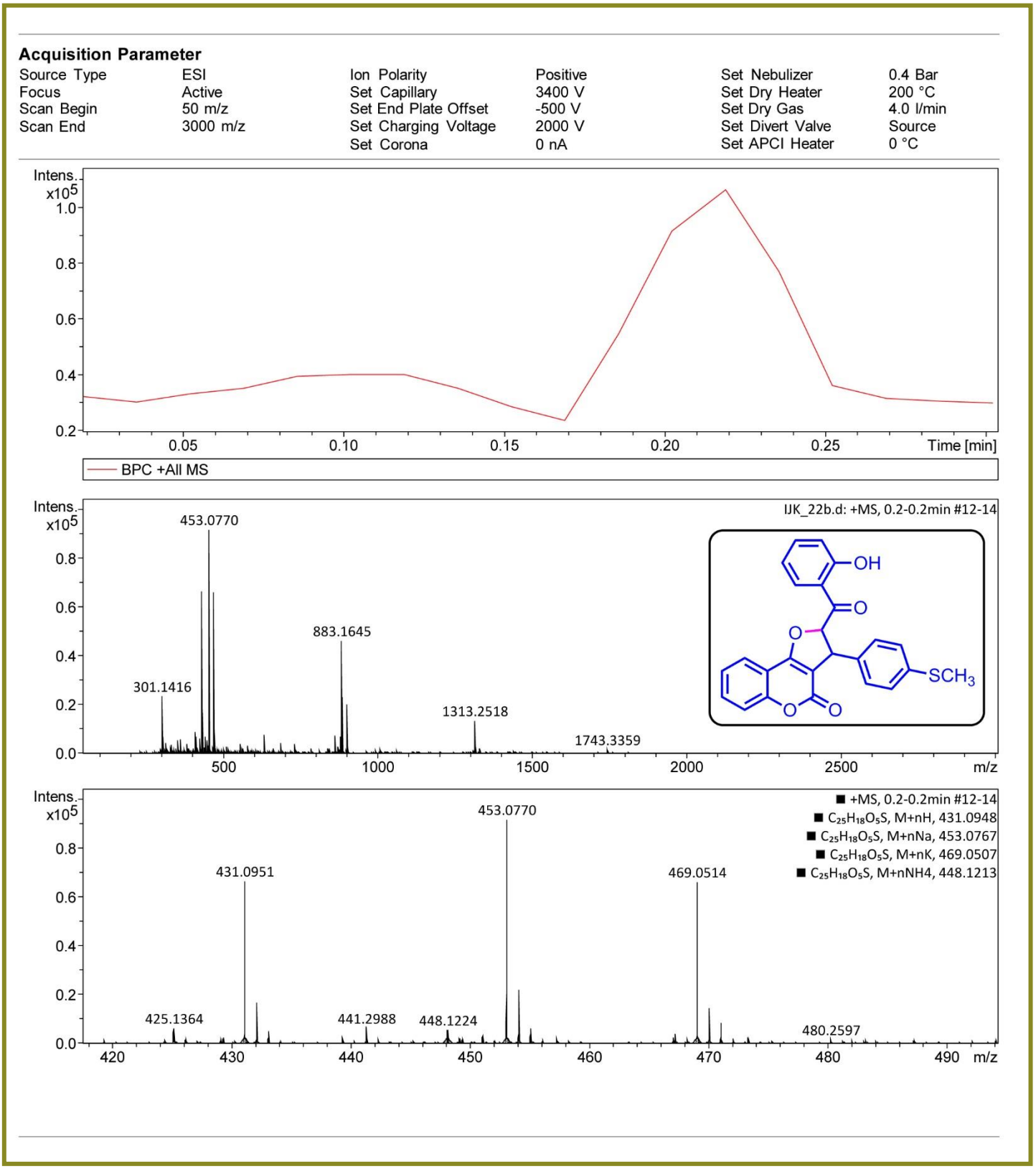


Figure S43. High-resolution Mass spectra 2-(2-hydroxybenzoyl)-3-(4-(methylthio)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-9**)

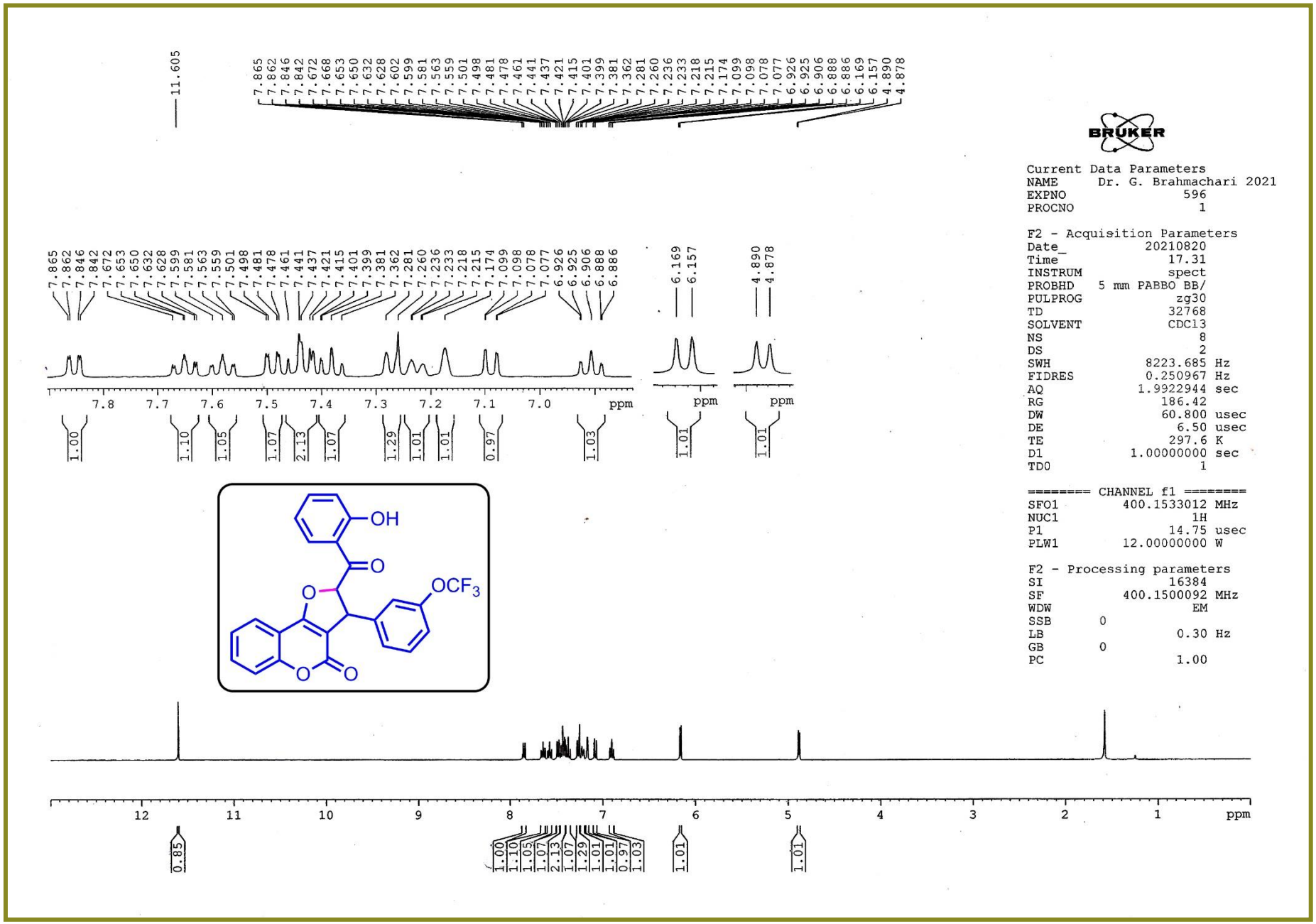


Figure S44. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-10**)

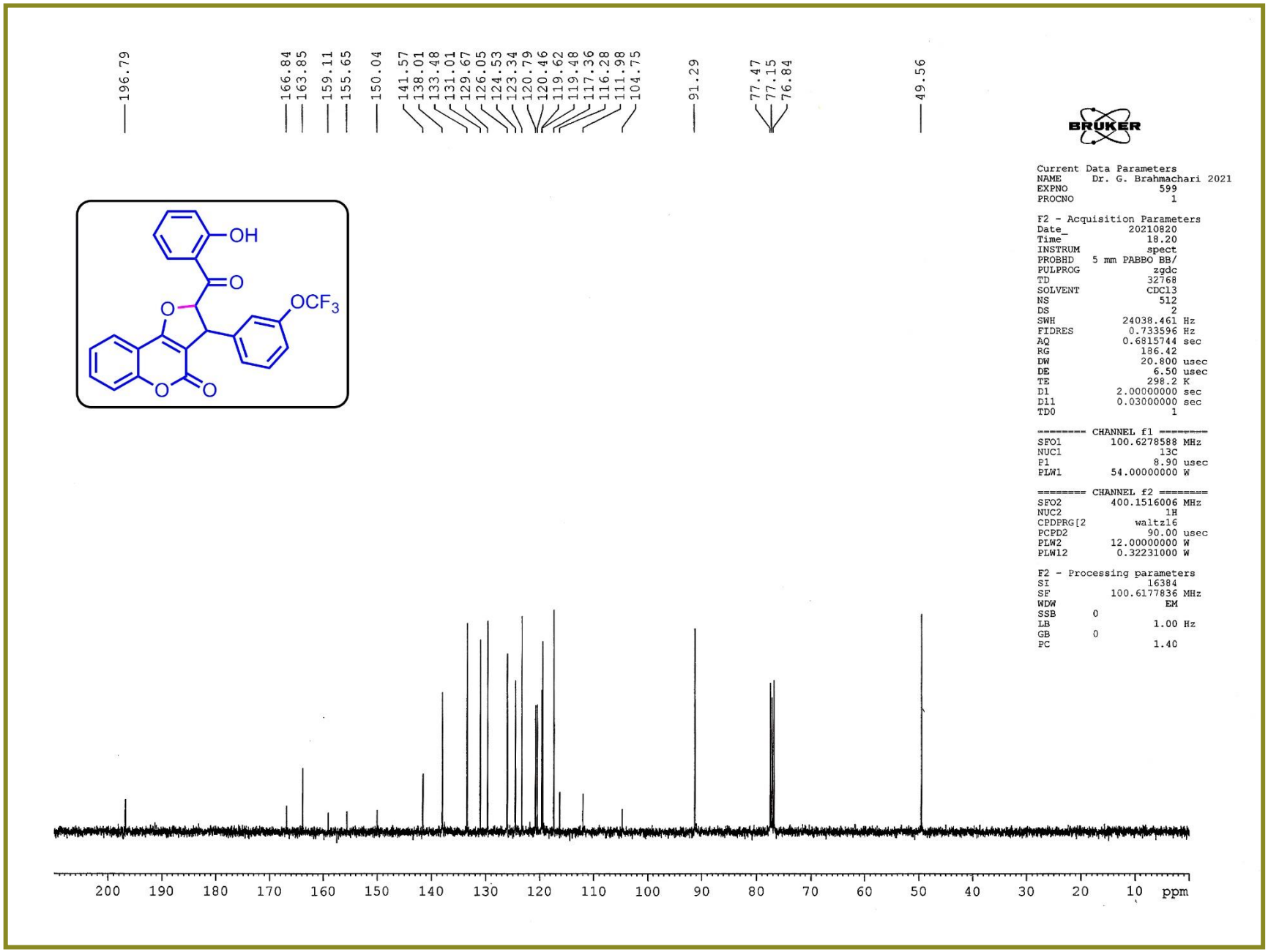


Figure S45. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-10**)

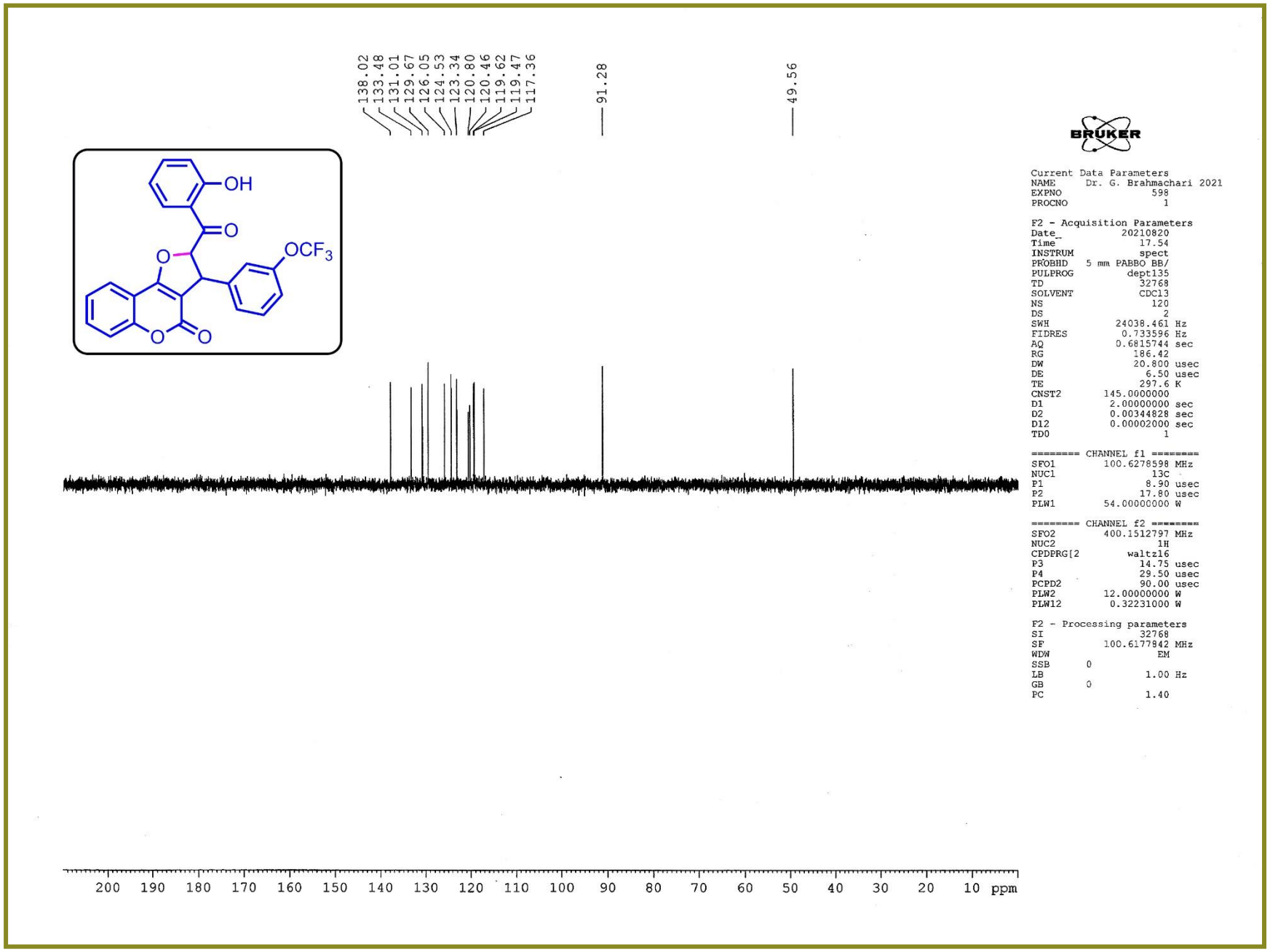


Figure S46. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-10**)

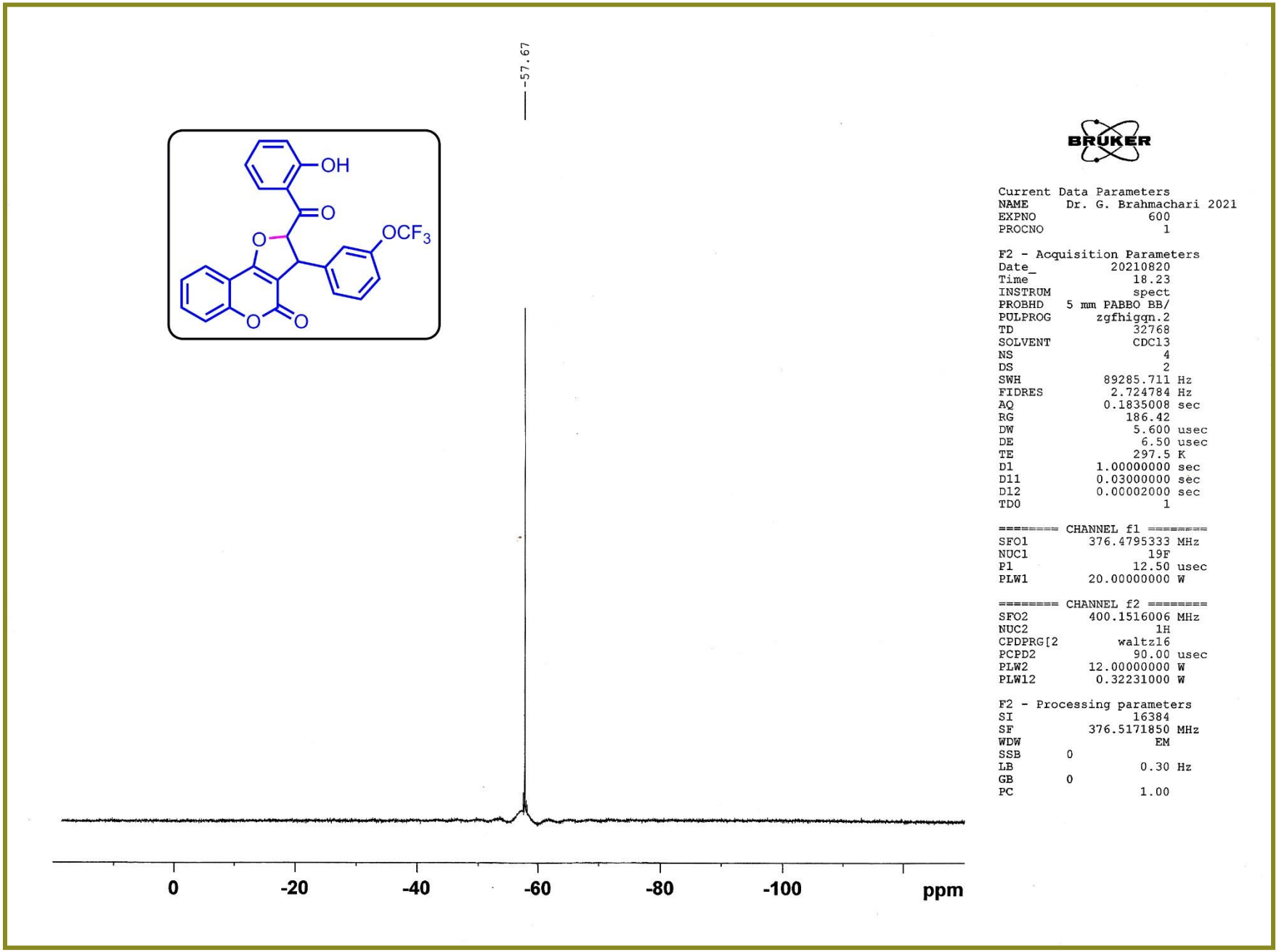


Figure S47. ^{19}F NMR spectrum of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-10**)

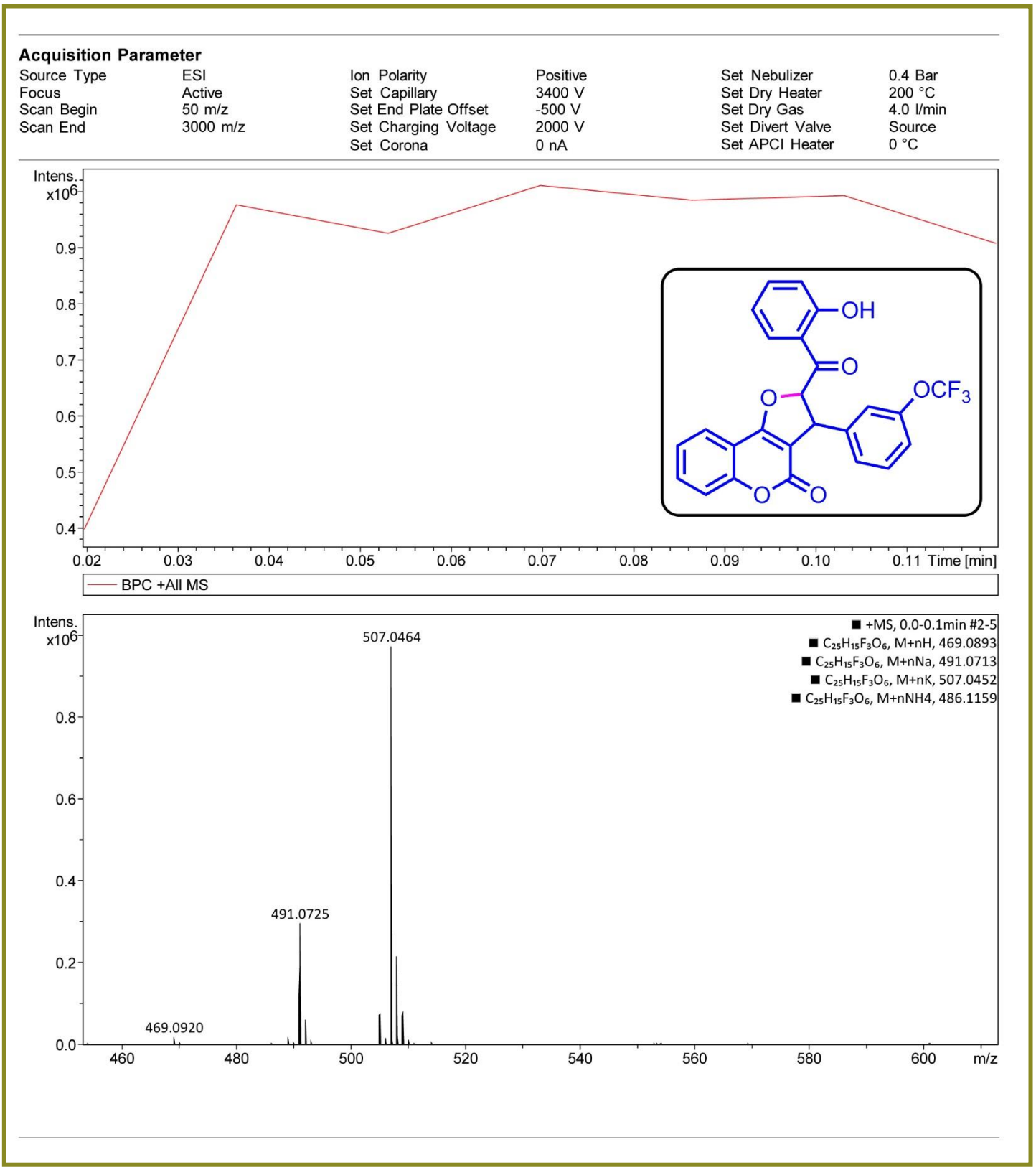


Figure S48. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(3-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-10**)

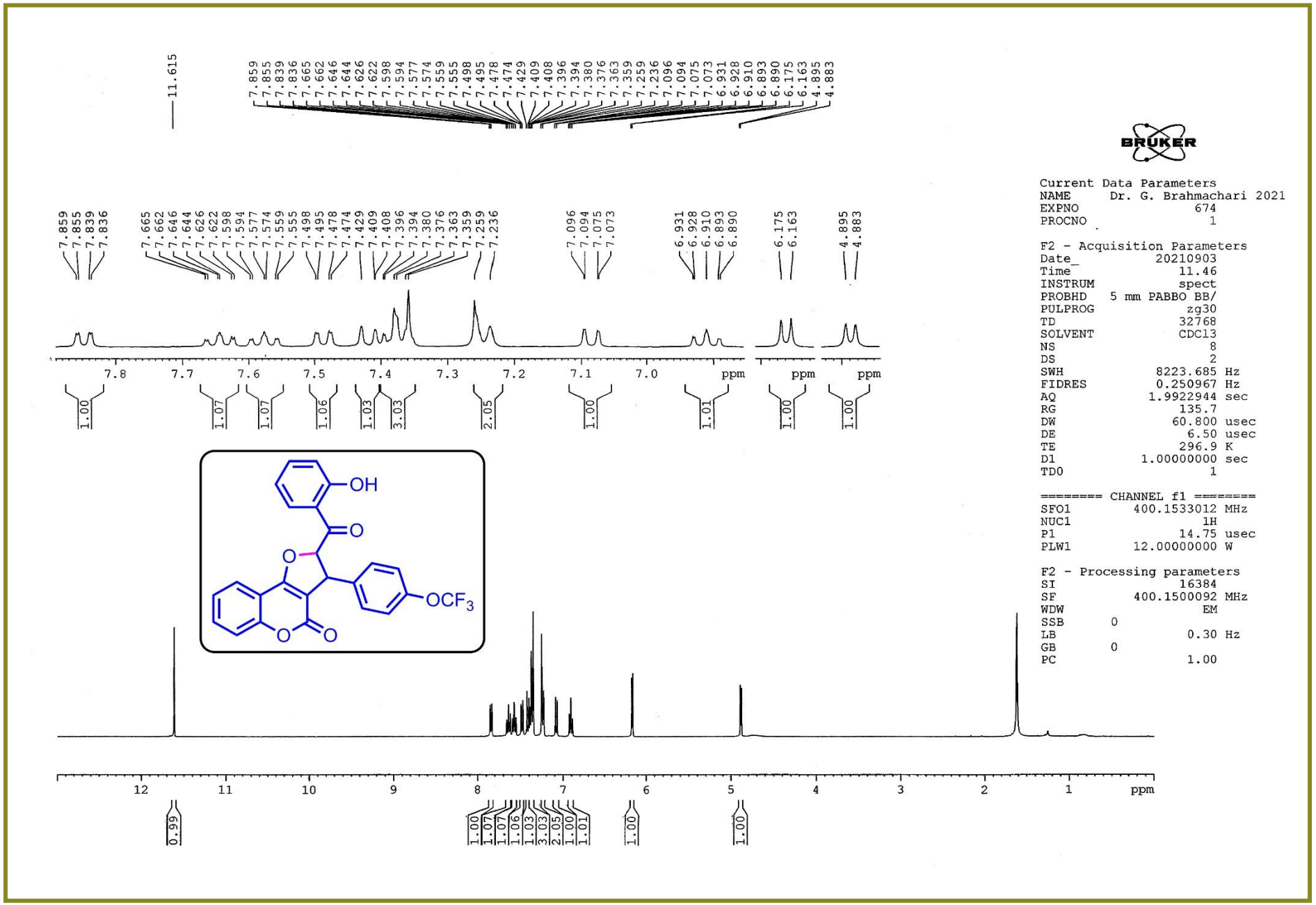


Figure S49. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-11**)

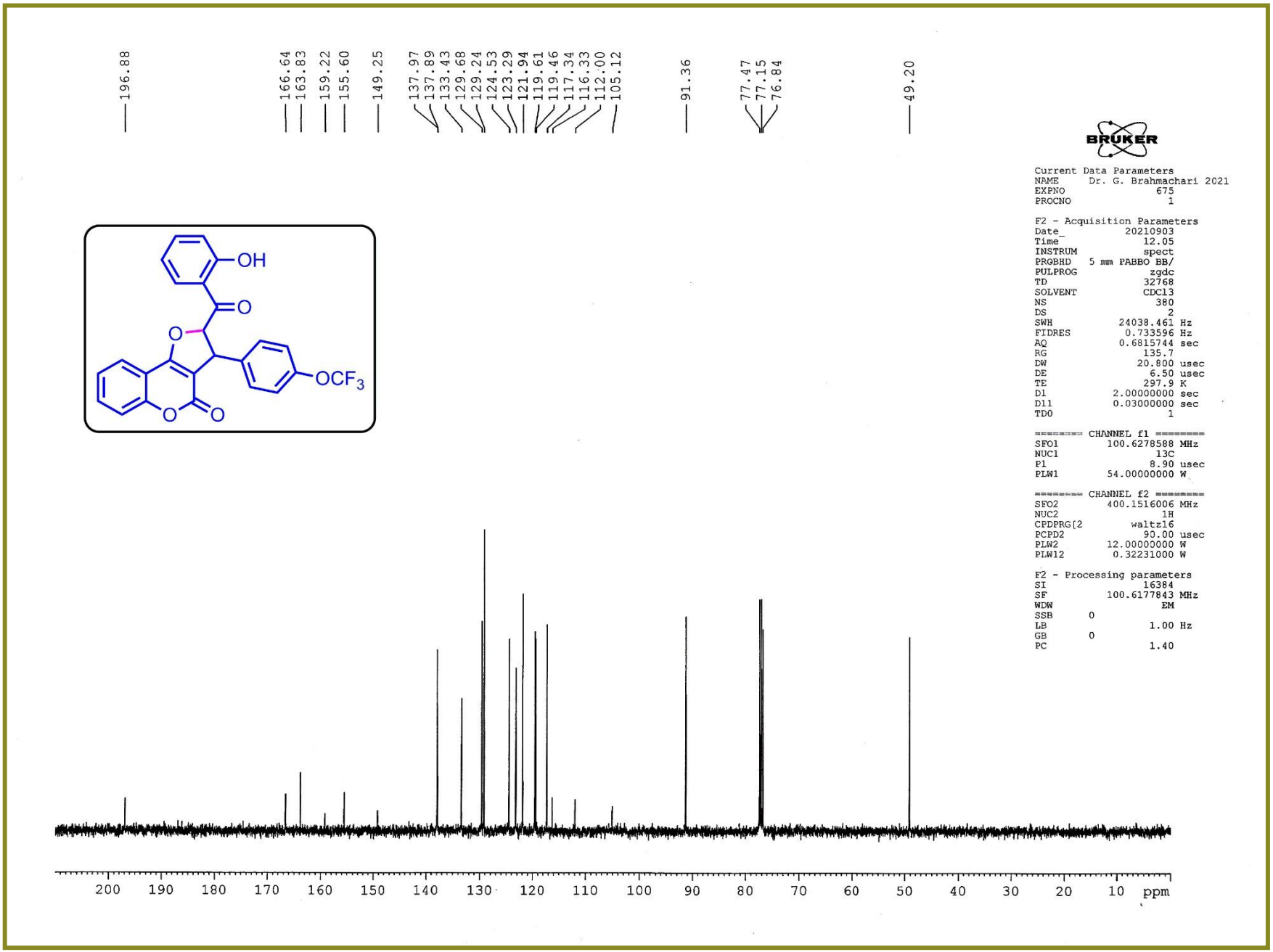


Figure S50. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-11**)

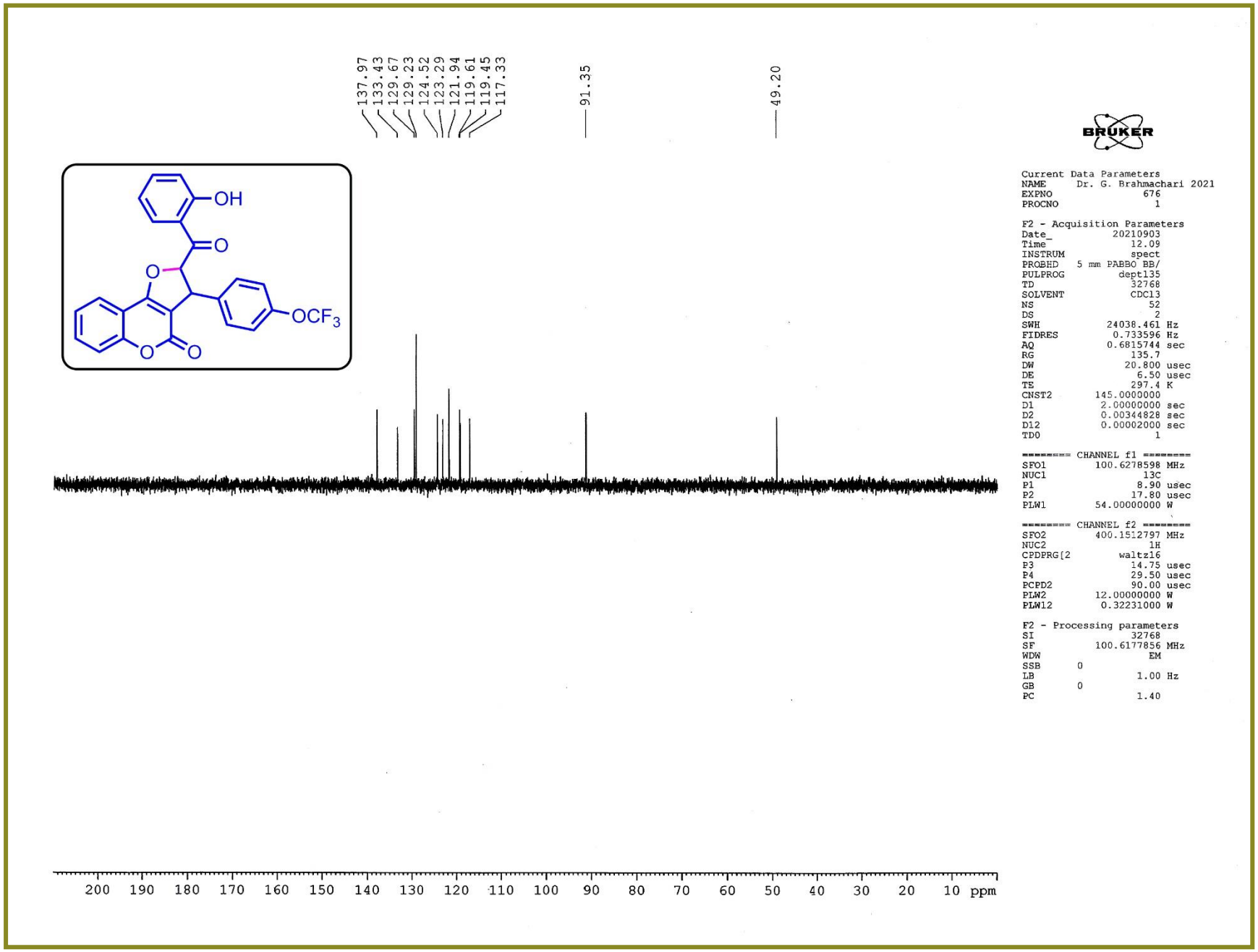


Figure S51. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-11**)

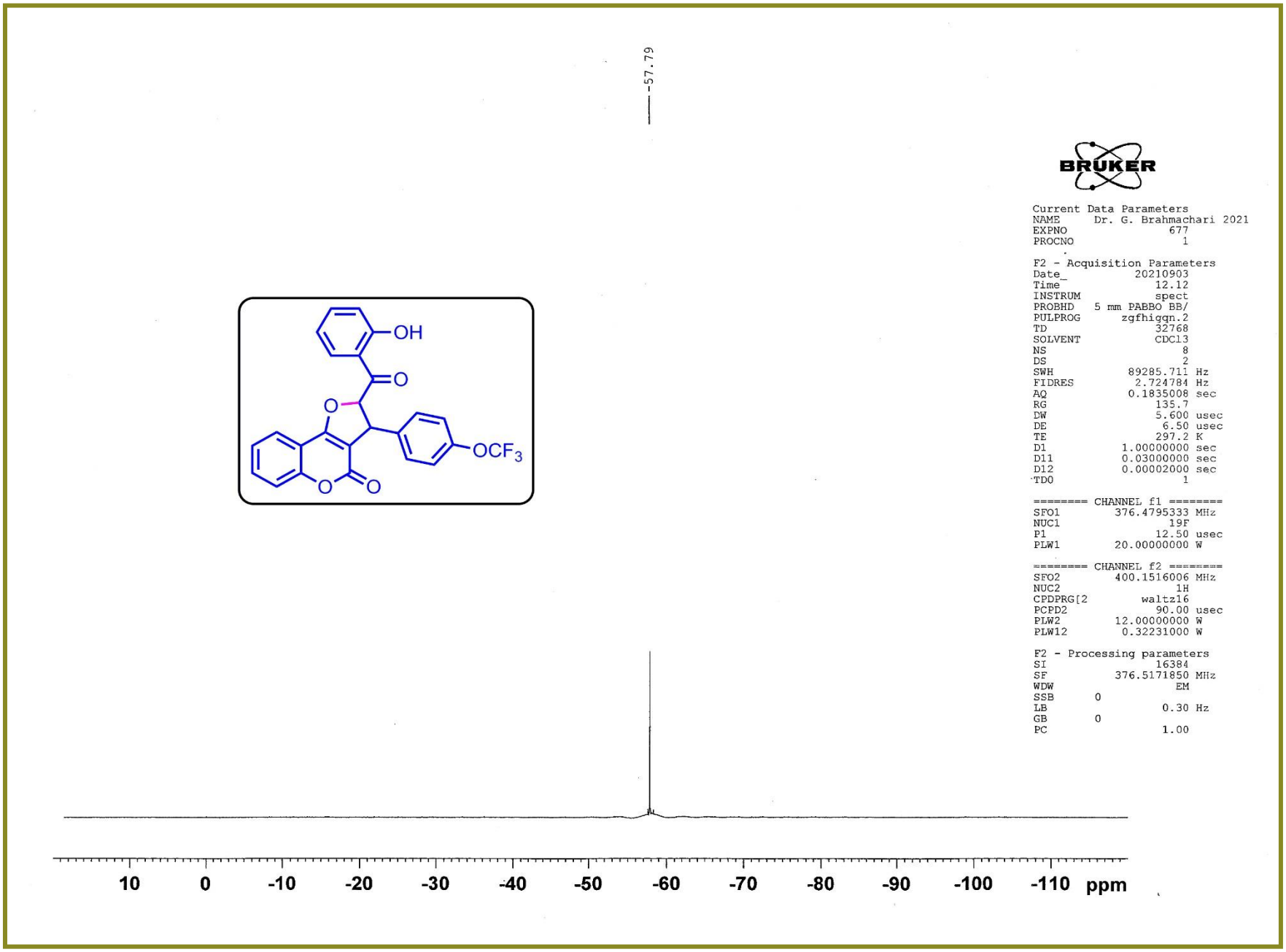


Figure S52. ^{19}F NMR spectrum of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-11**)

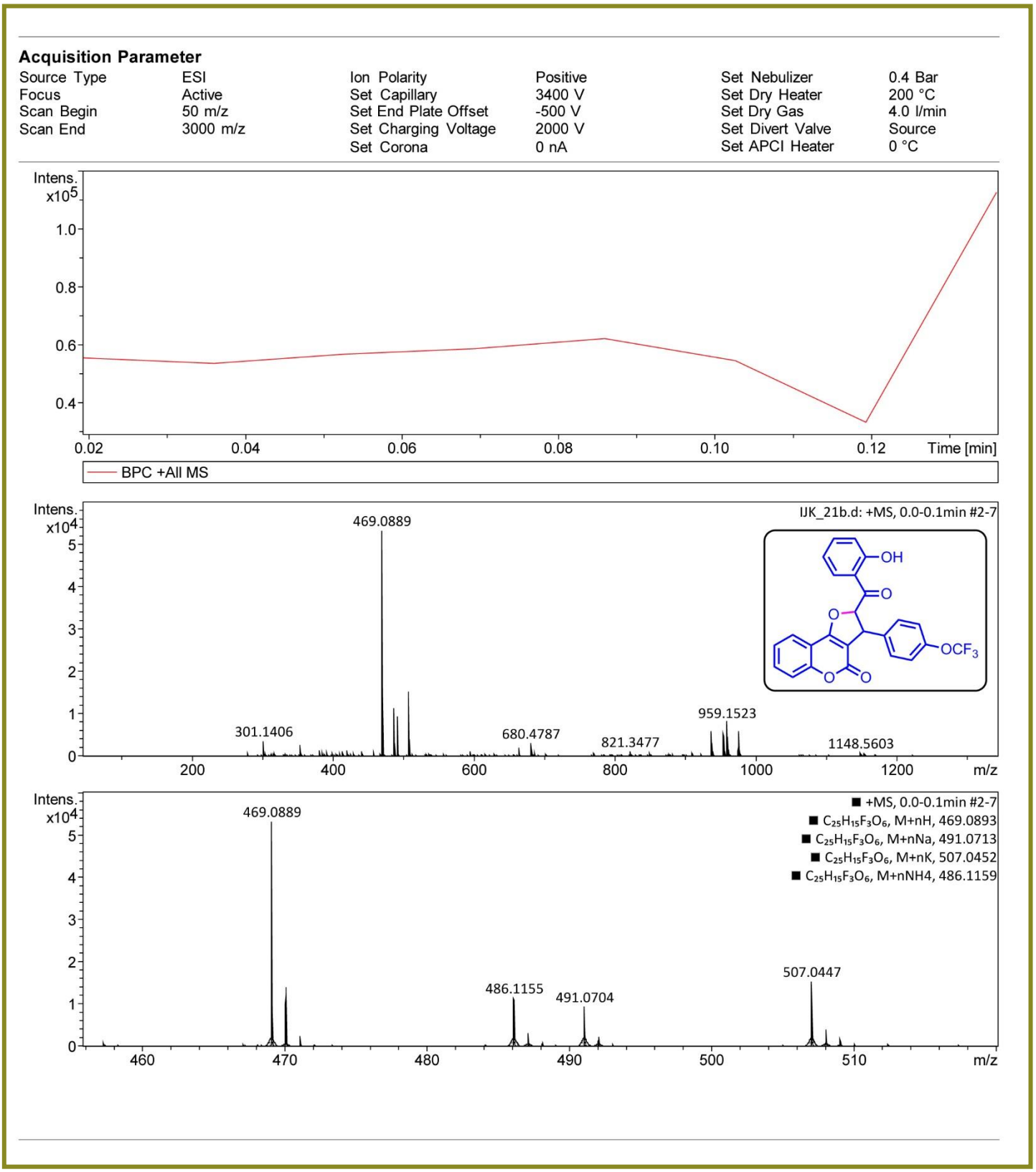


Figure S53. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(4-(trifluoromethoxy)phenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-11**)

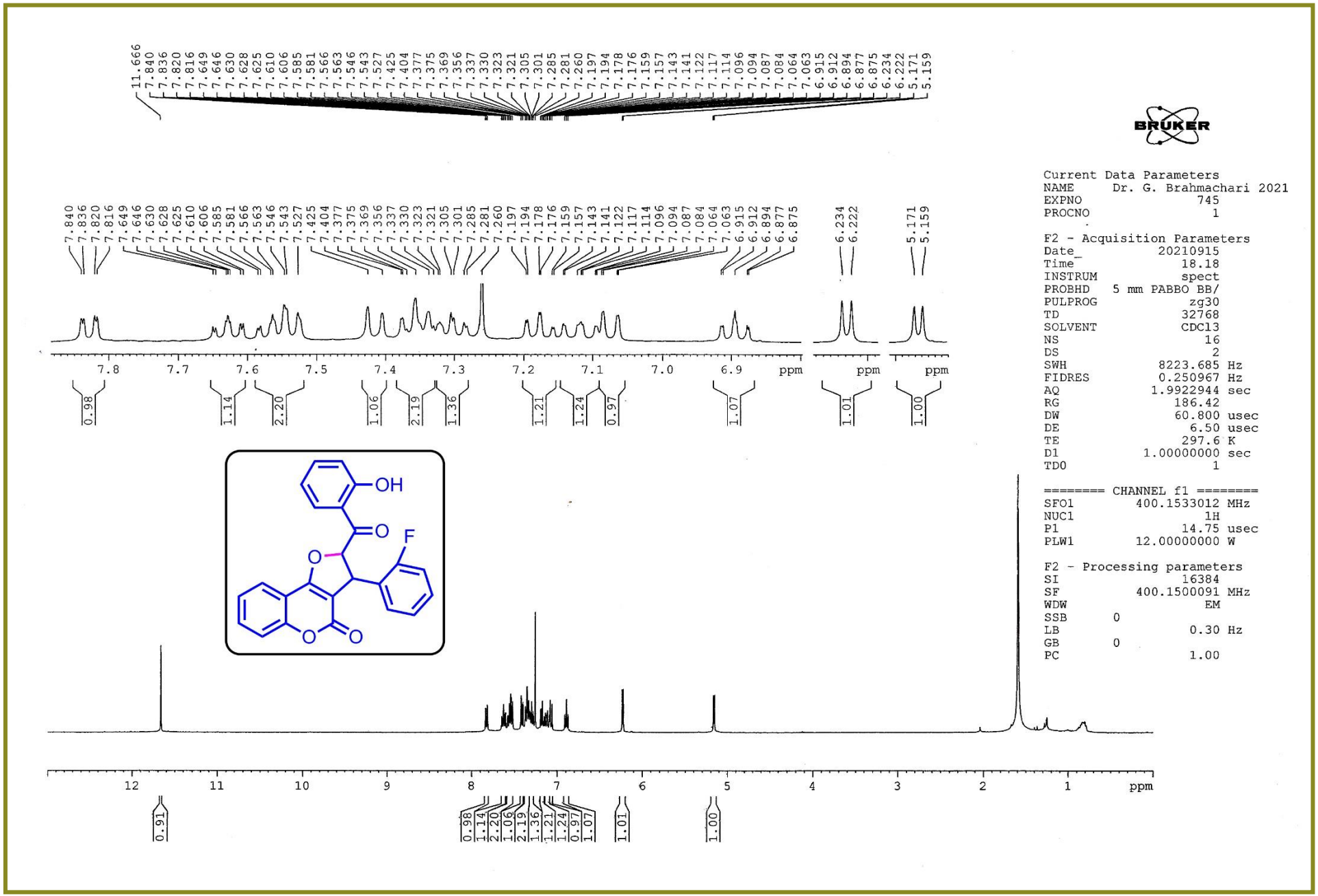


Figure S54. ^{1}H-NMR spectrum of 3-(2-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-12**)

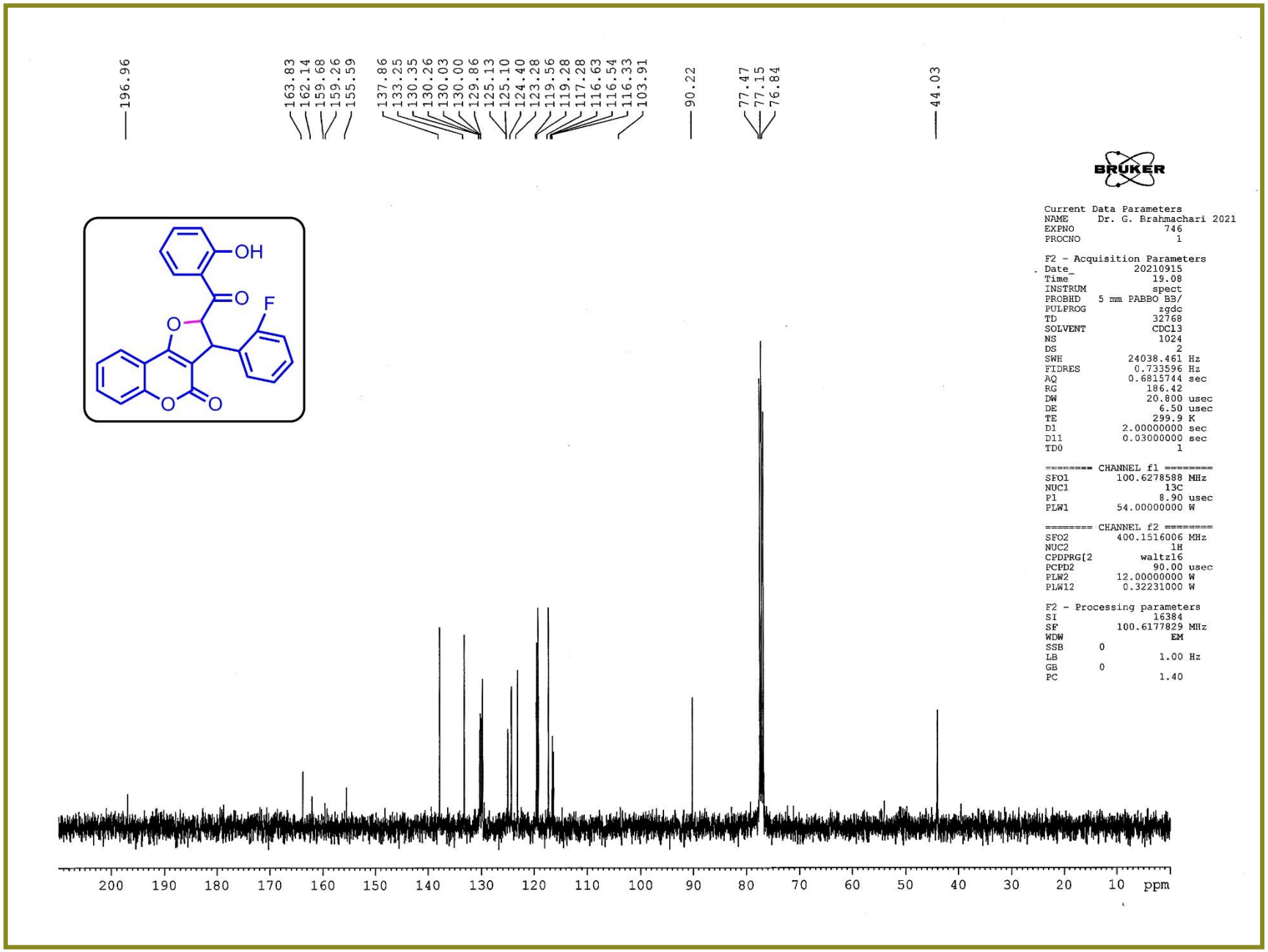


Figure S55. ^{13}C-NMR spectrum of 3-(2-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-12**)

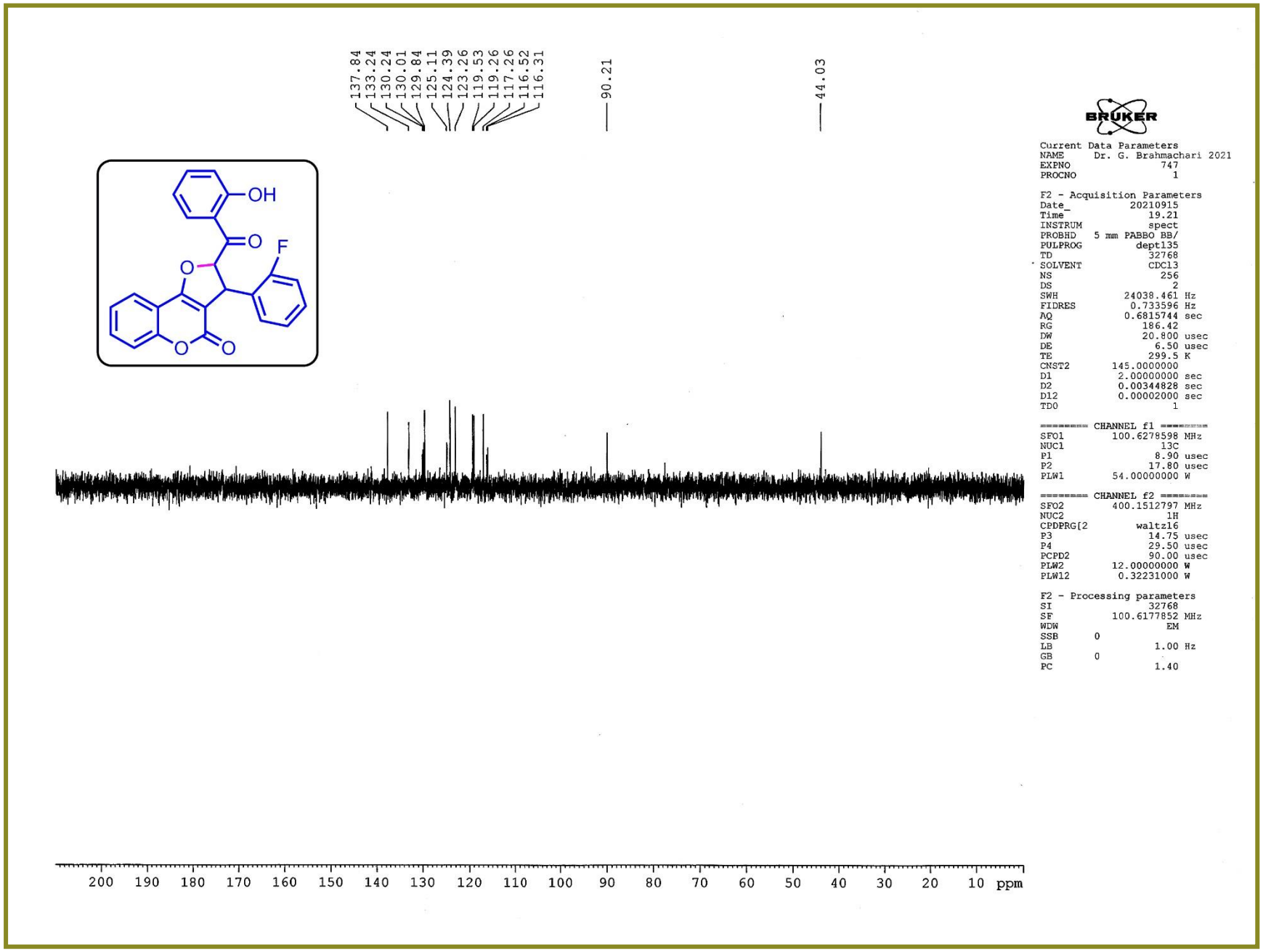


Figure S56. DEPT-135 NMR spectrum of 3-(2-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-12**)

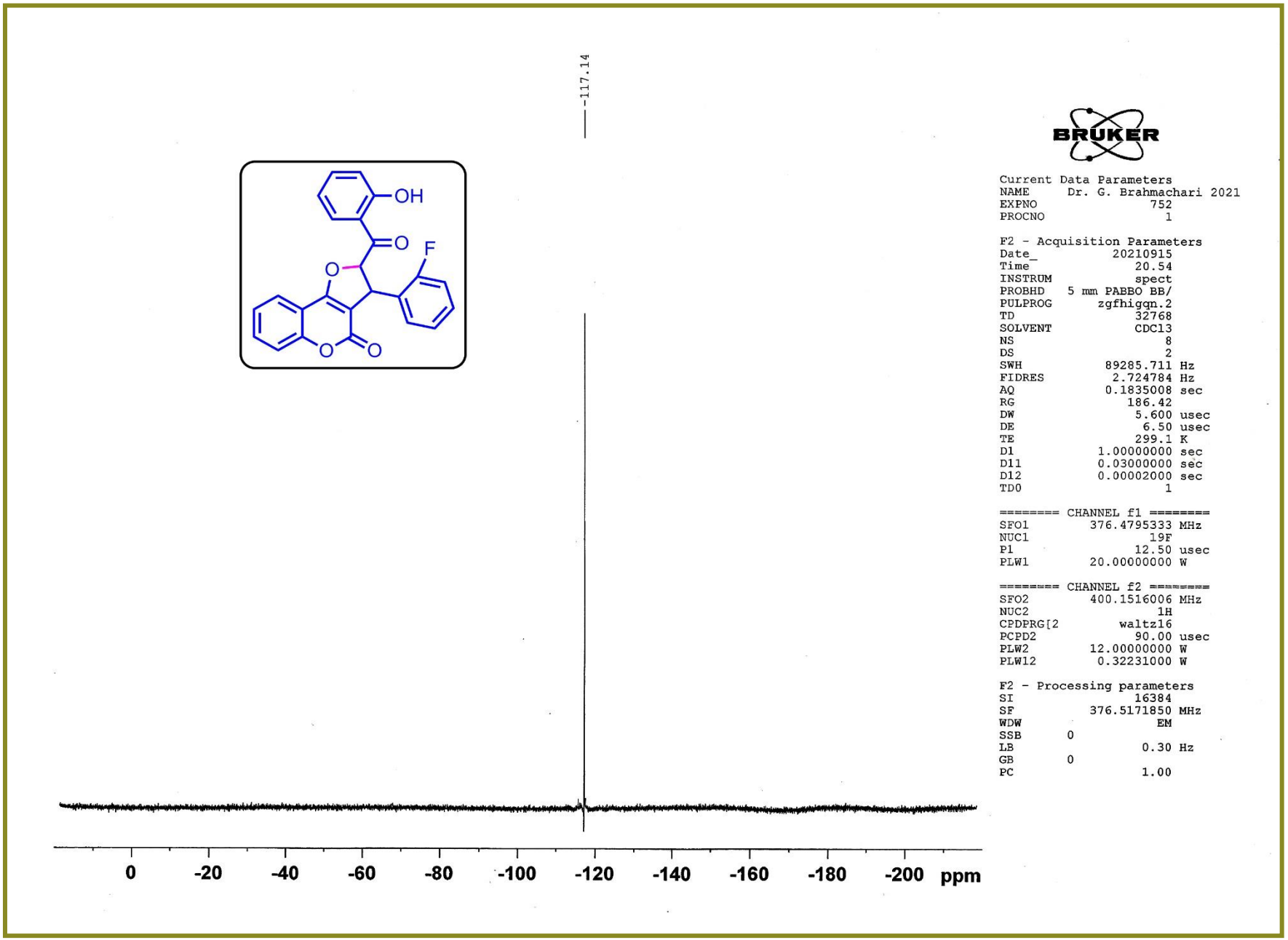


Figure S57. ^{19}F NMR spectrum of 3-(2-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-12**)

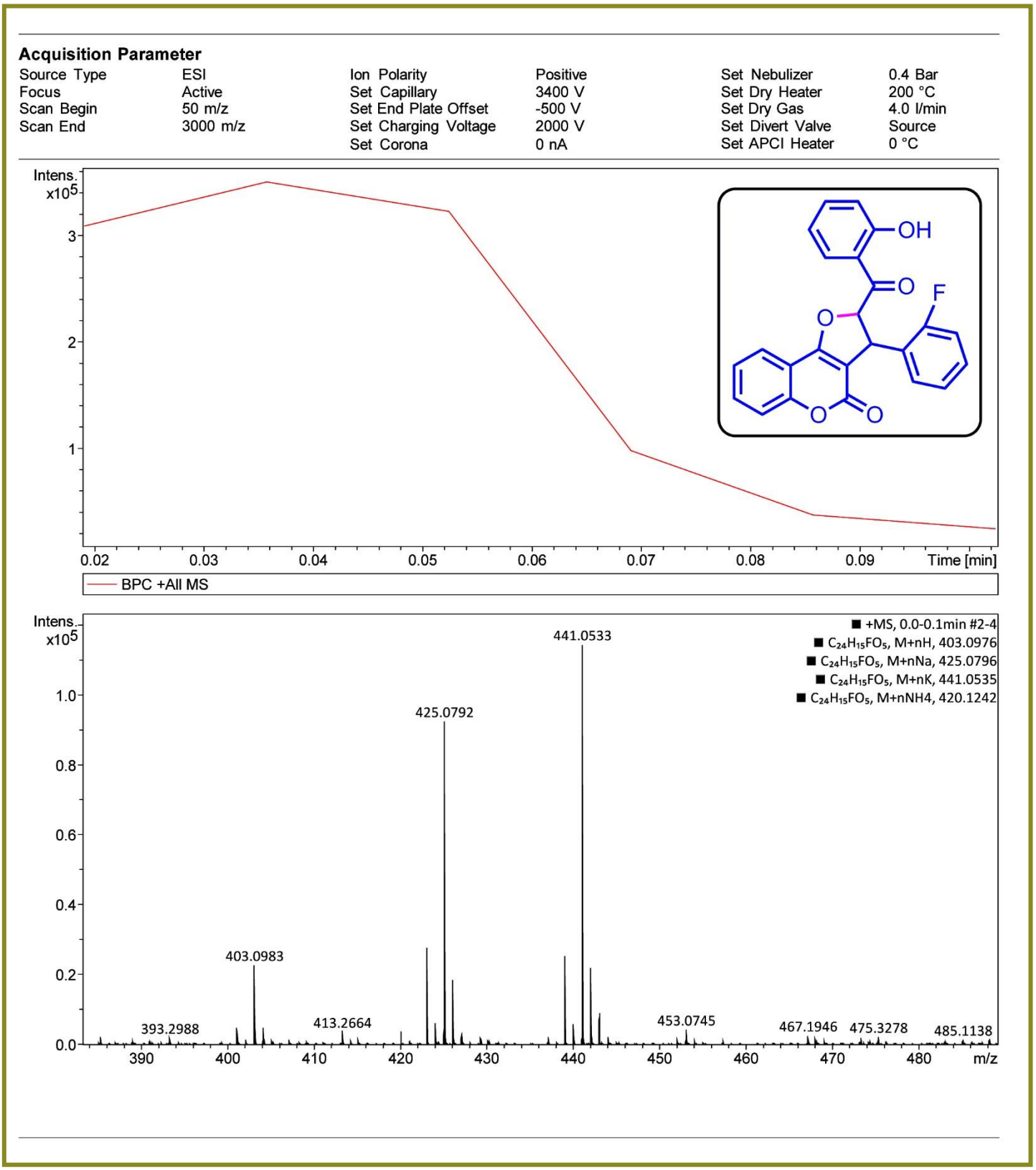


Figure S58. High-resolution Mass spectra of 3-(2-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-12**)

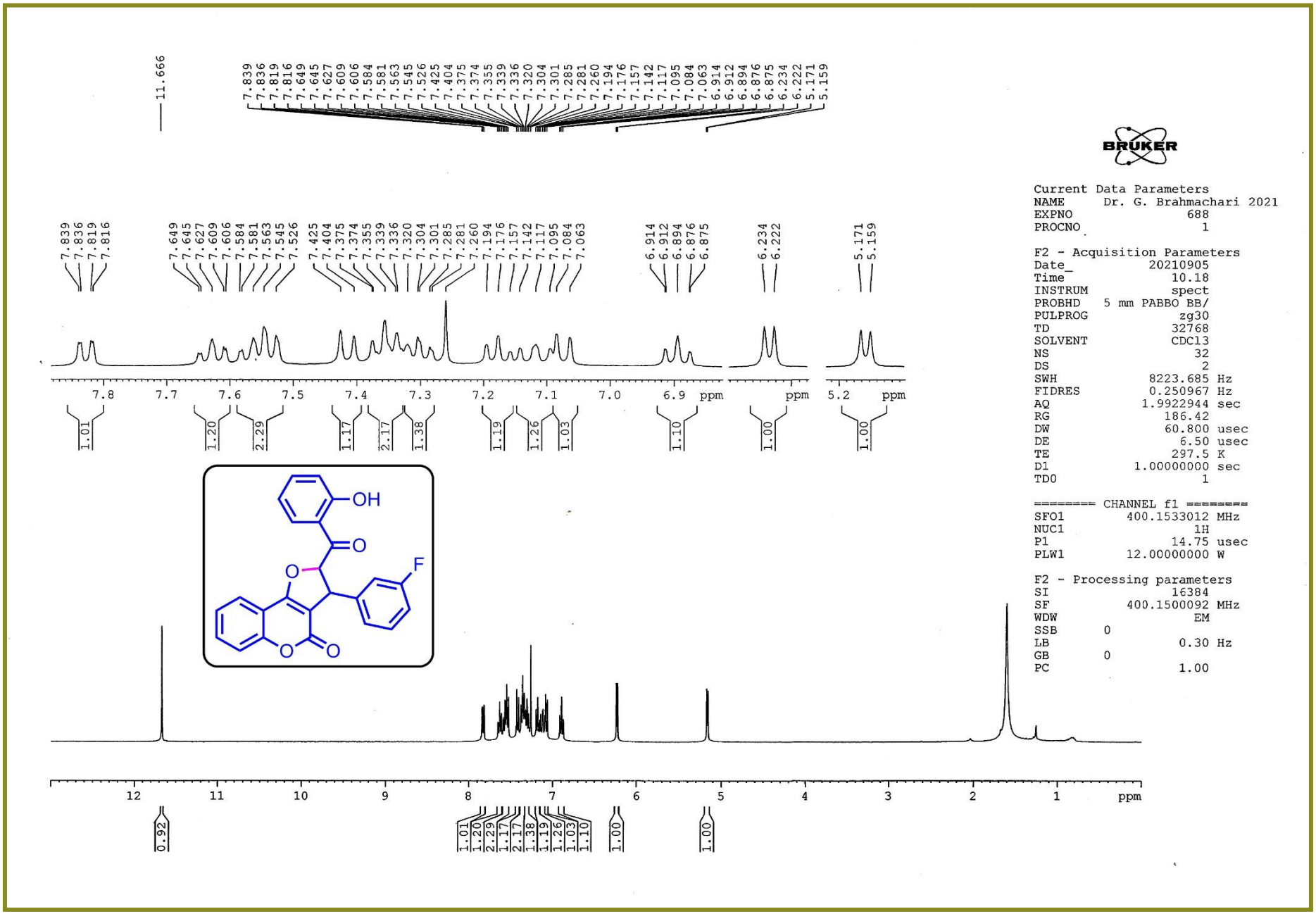


Figure S59. ^{1}H-NMR spectrum of 3-(3-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-13**)

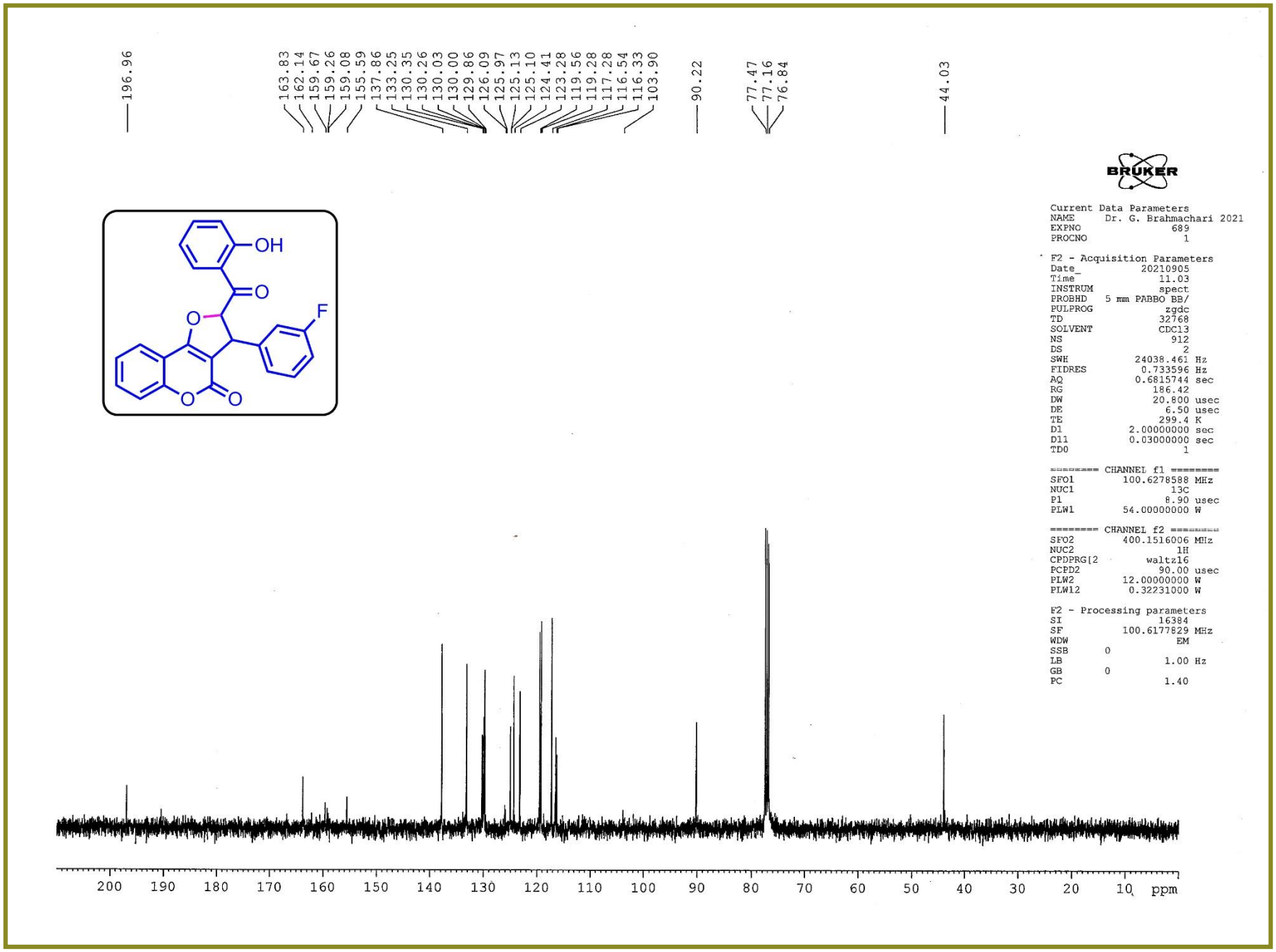


Figure S60. ^{13}C-NMR spectrum of 3-(3-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-13**)

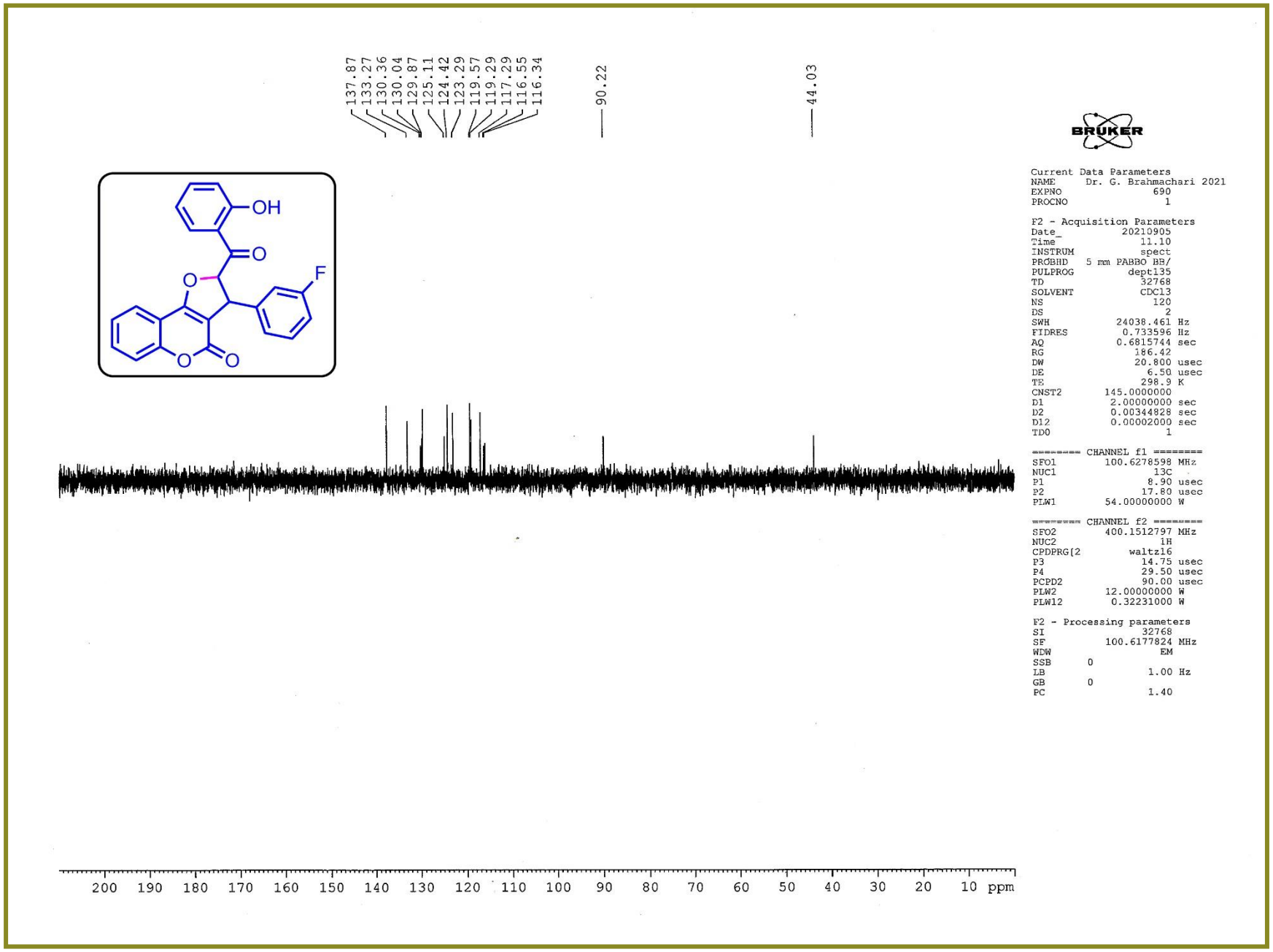


Figure S61. DEPT-135 NMR spectrum of 3-(3-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-13**)

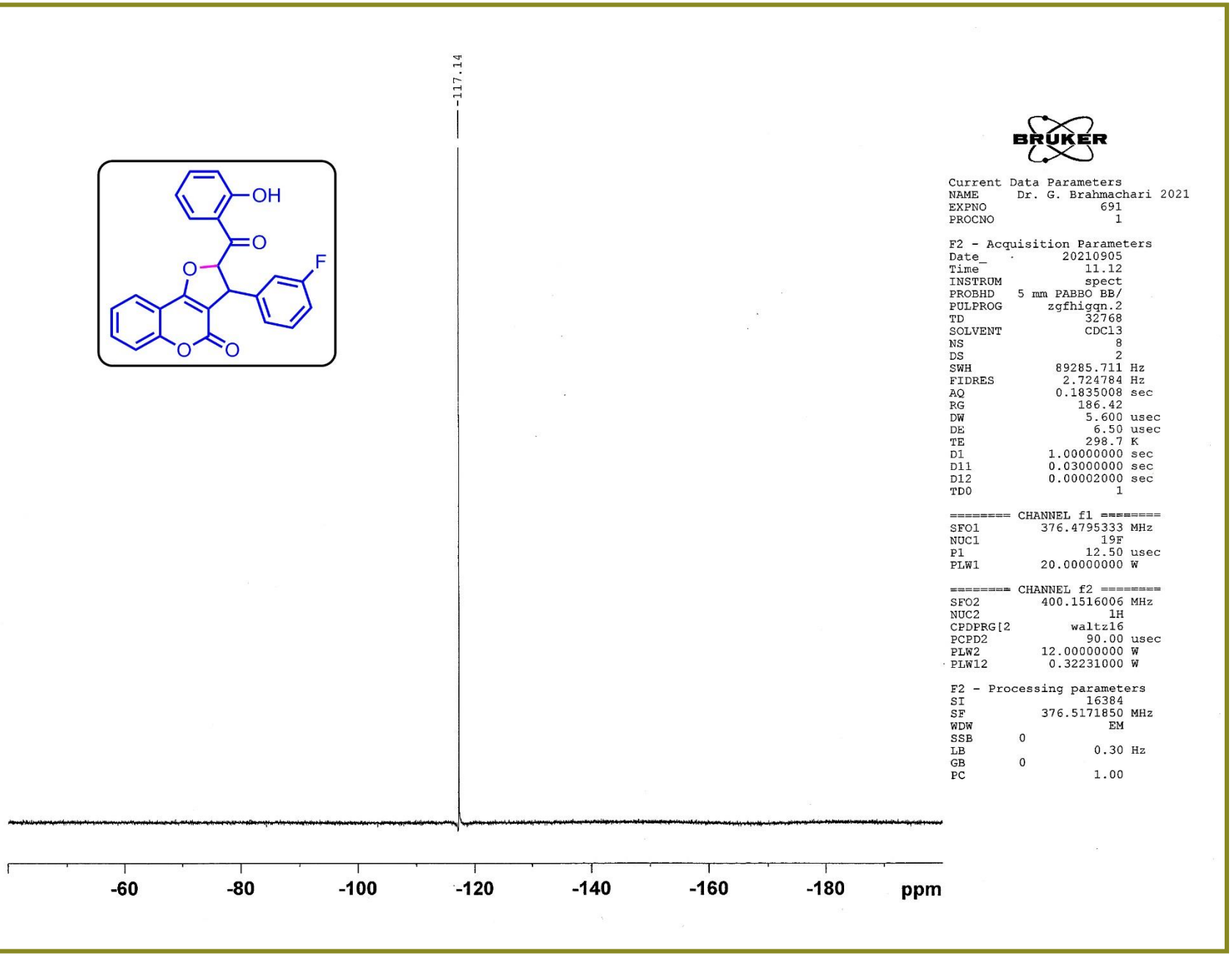


Figure S62. ^{19}F NMR spectrum of 3-(3-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-13**)

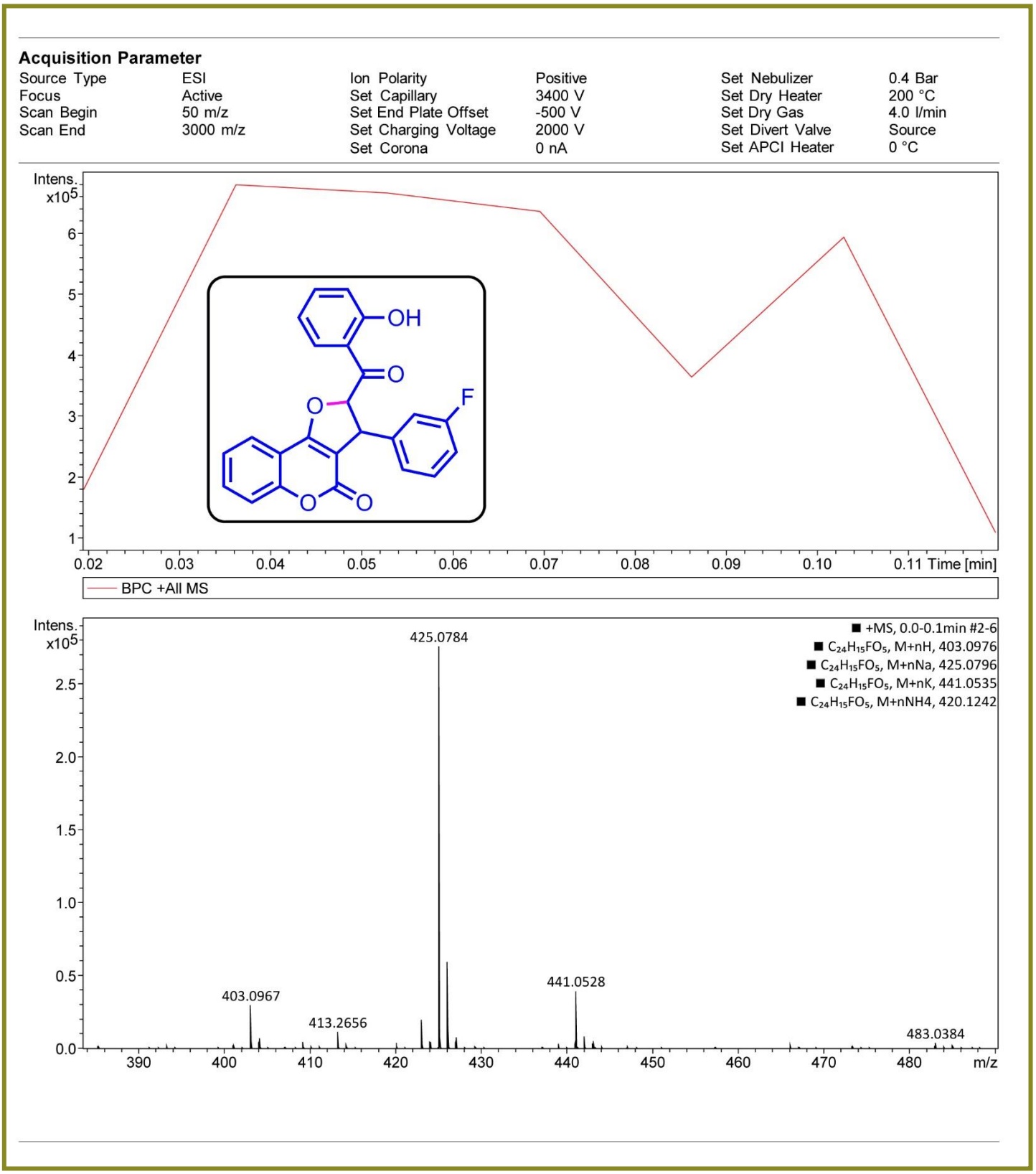


Figure S63. High-resolution Mass spectra of 3-(3-fluorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-13**)

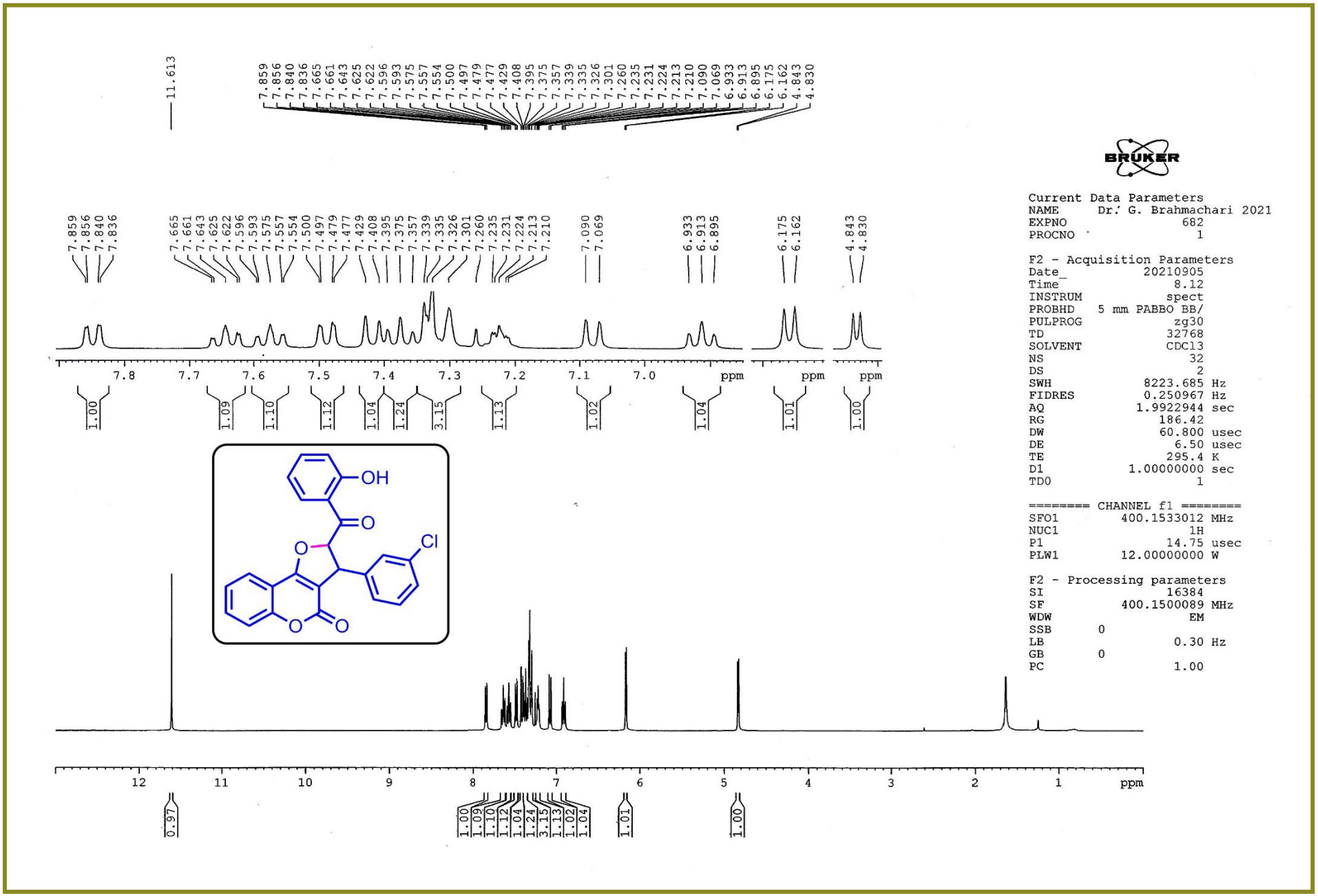


Figure S64. ^{1}H-NMR spectrum of 3-(3-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-14**)

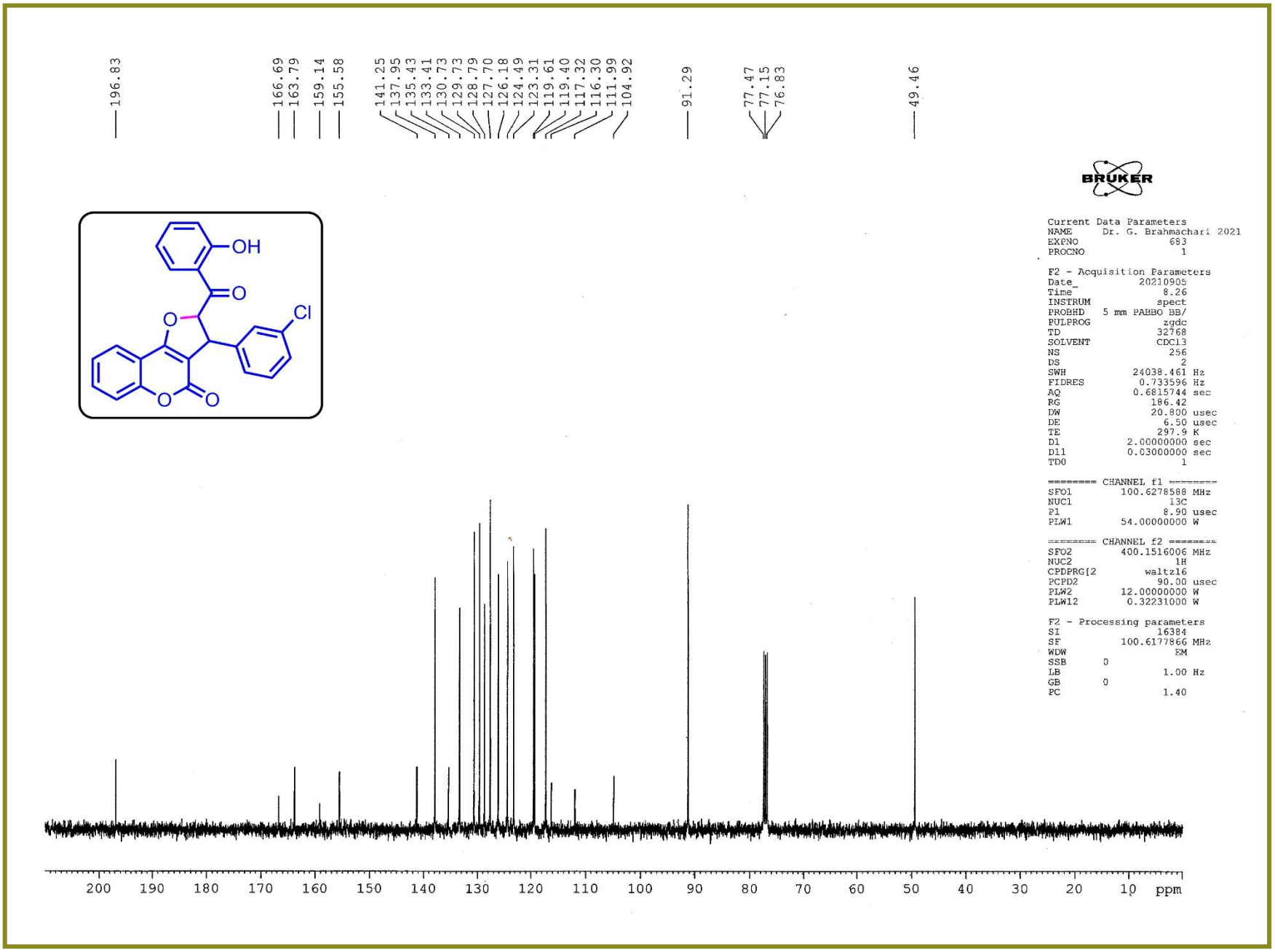


Figure S65. ^{13}C-NMR spectrum of 3-(3-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-14**)

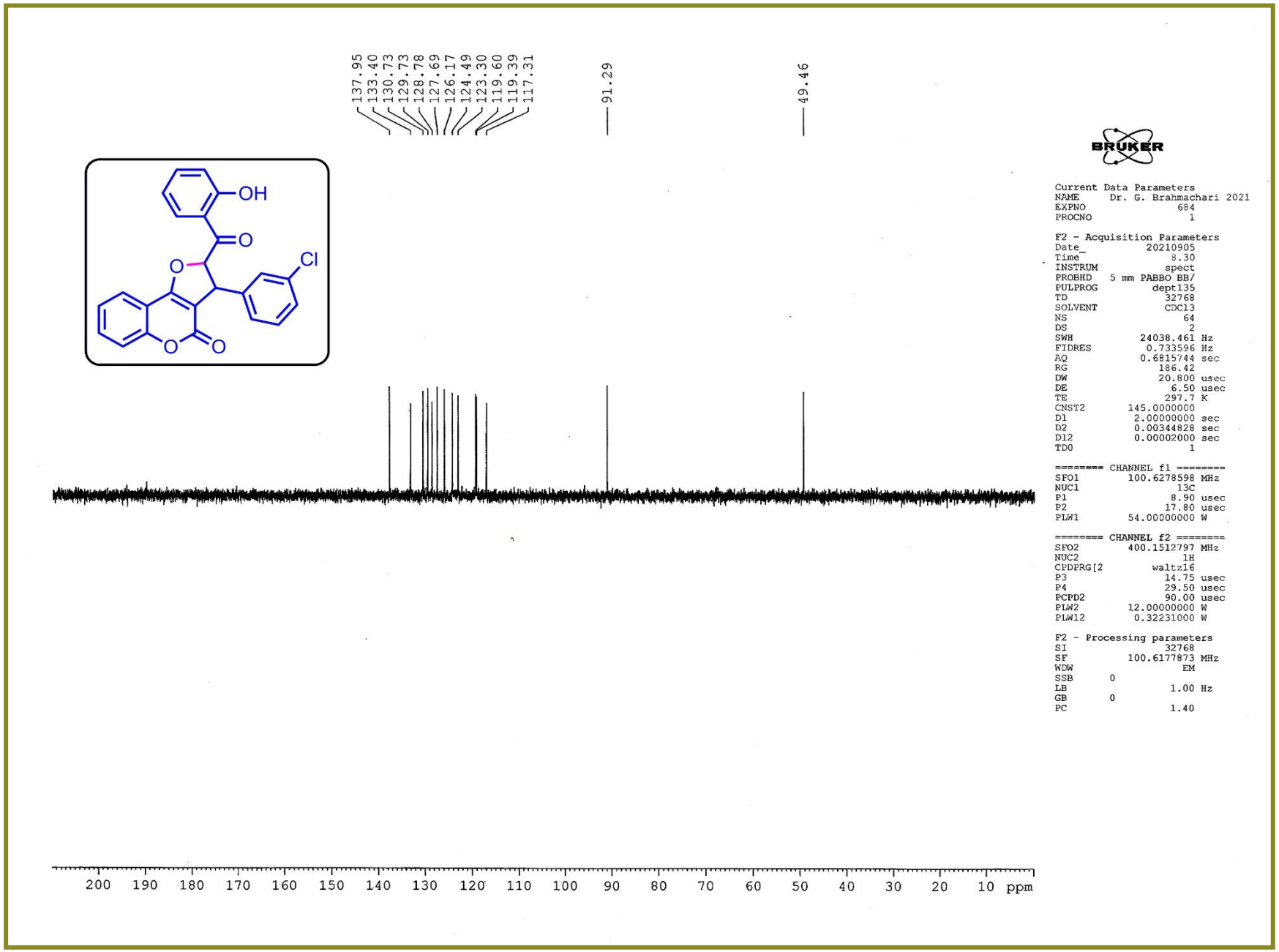


Figure S66. DEPT-135 NMR spectrum of 3-(3-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-14**)

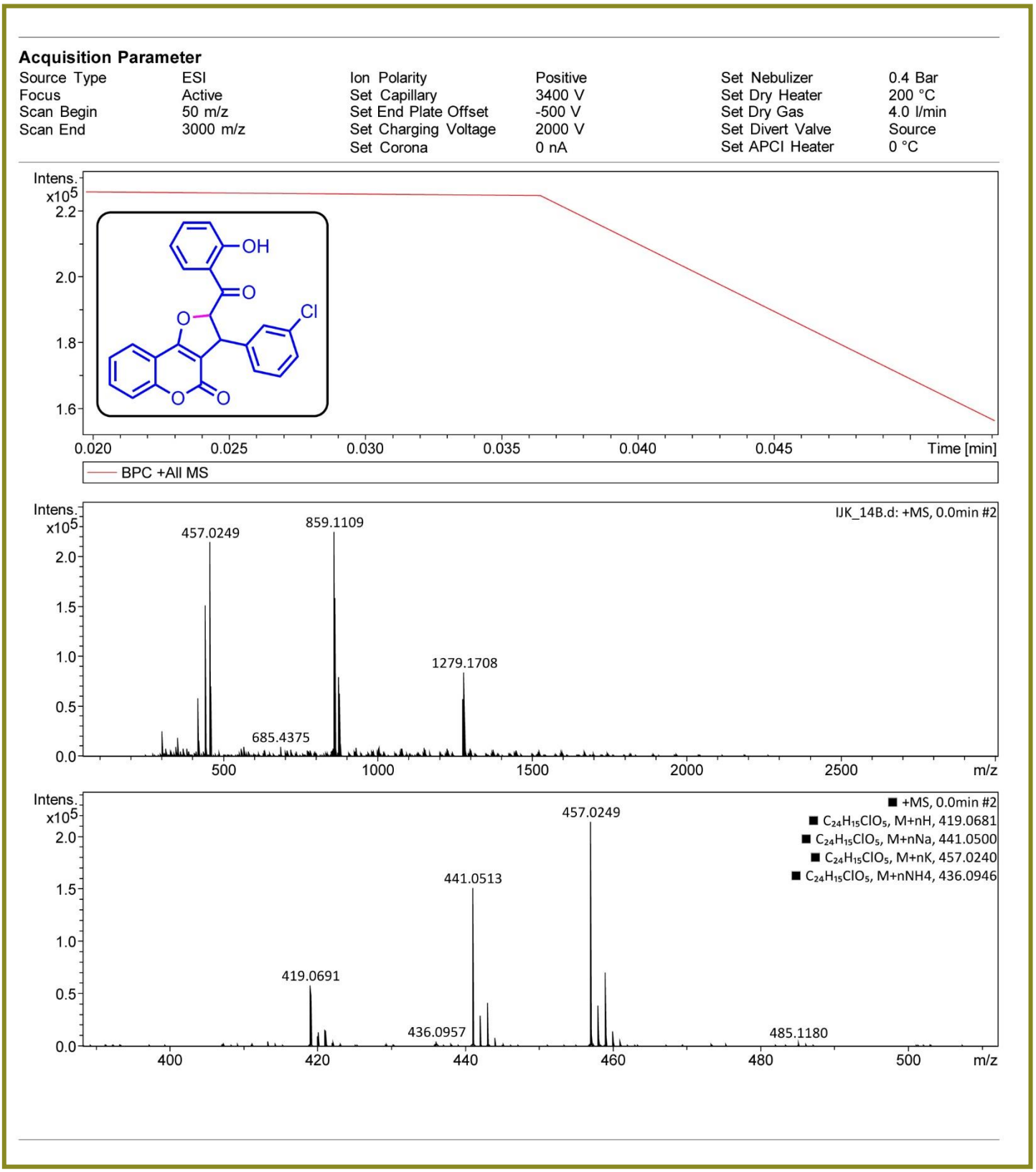


Figure S67. High-resolution Mass spectra of 3-(3-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-14**)

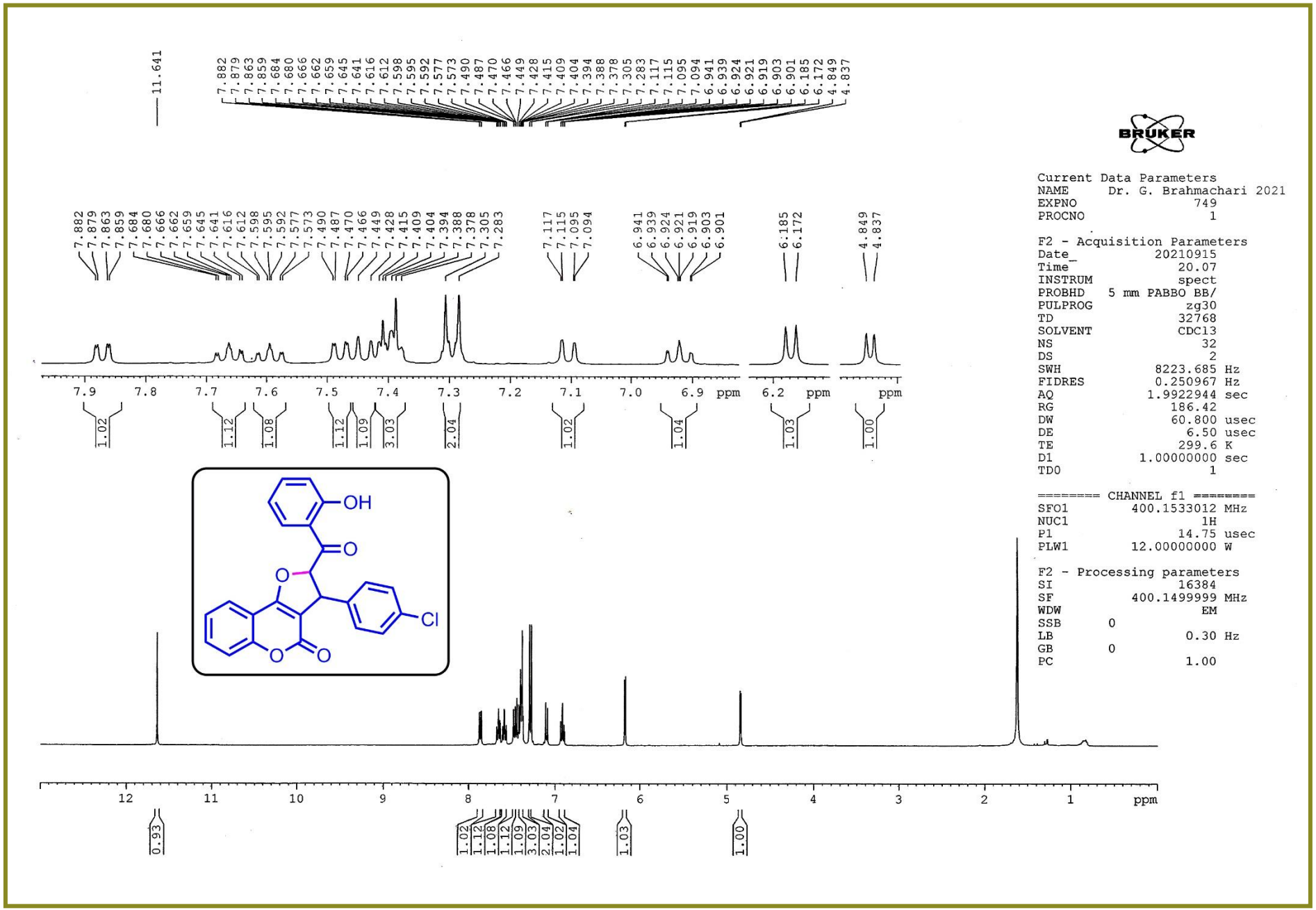


Figure S68. ^{1}H-NMR spectrum of 3-(4-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-15**)

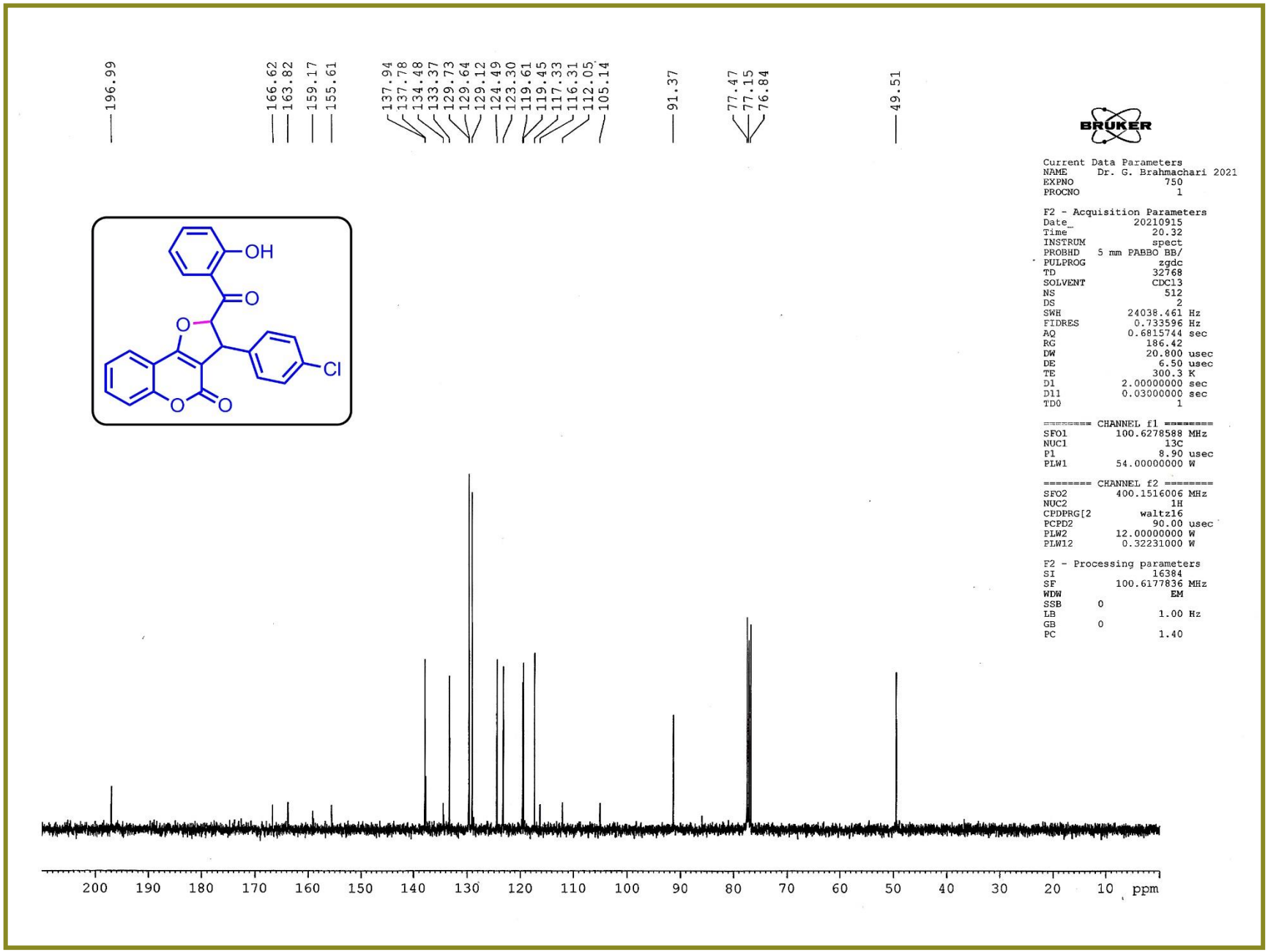


Figure S69. ^{13}C-NMR spectrum of 3-(4-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-15**)

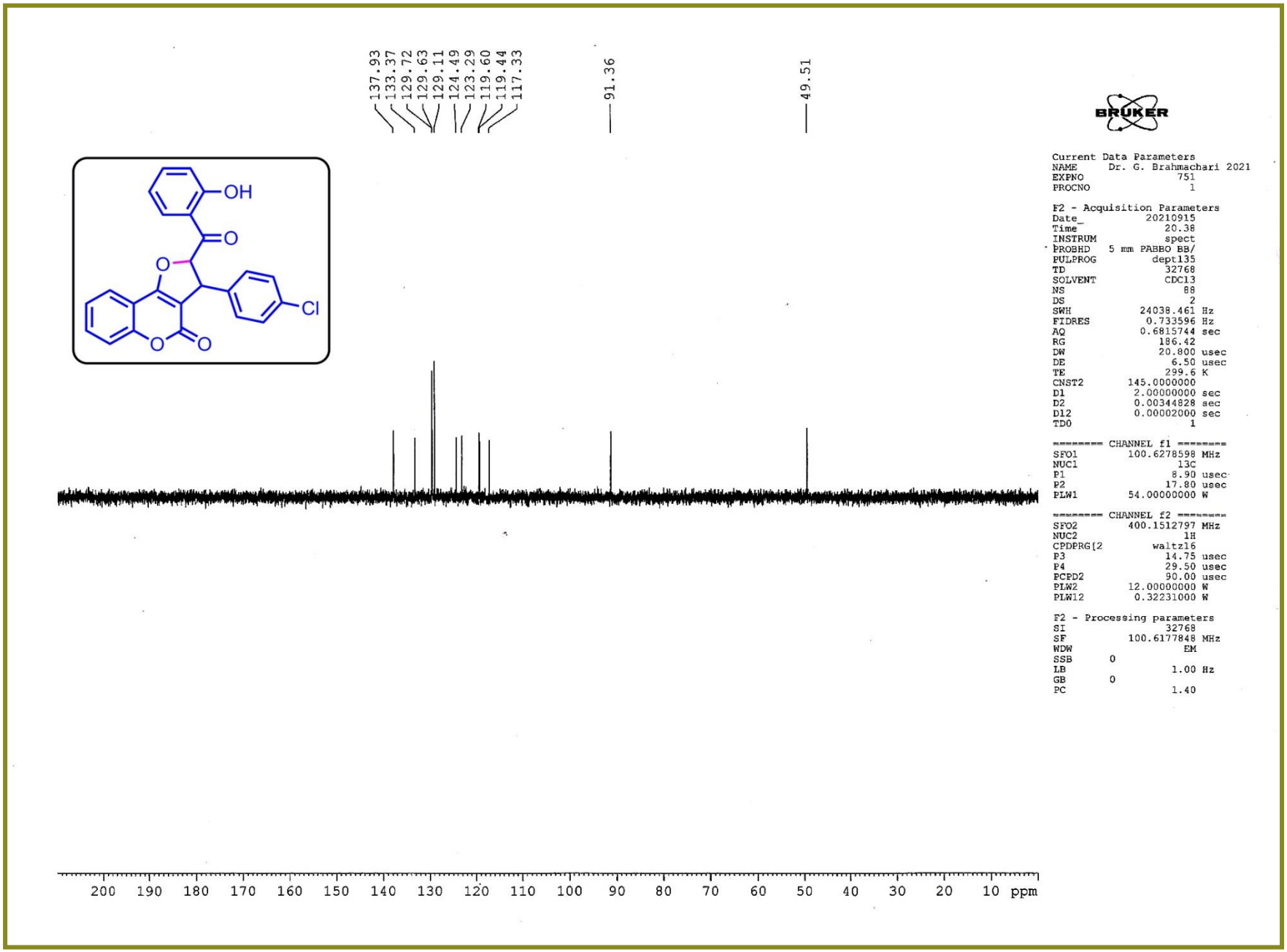


Figure S70. DEPT-135 NMR spectrum of 3-(4-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-15**)

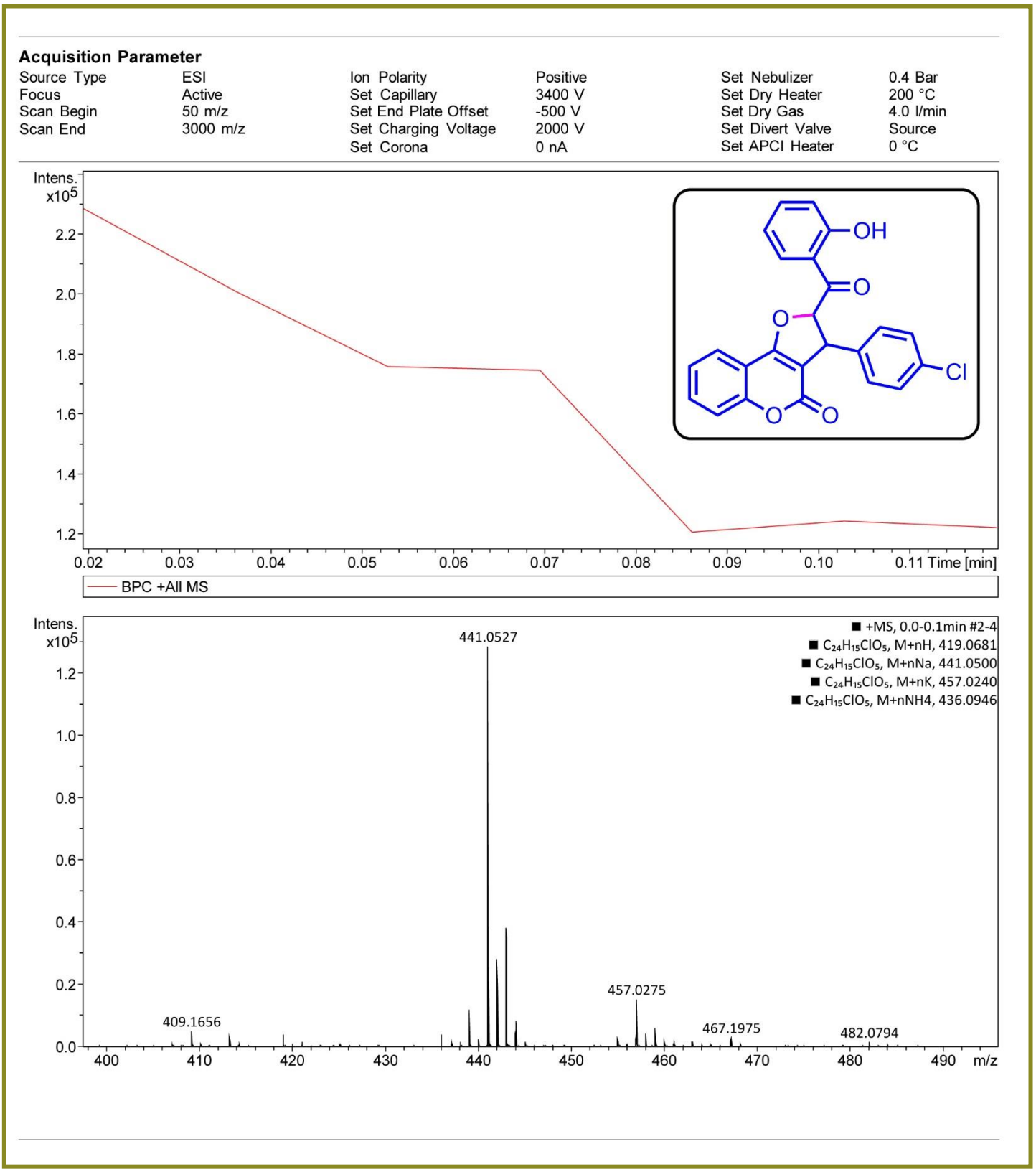


Figure S71. High-resolution Mass spectra of 3-(4-chlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-15**)

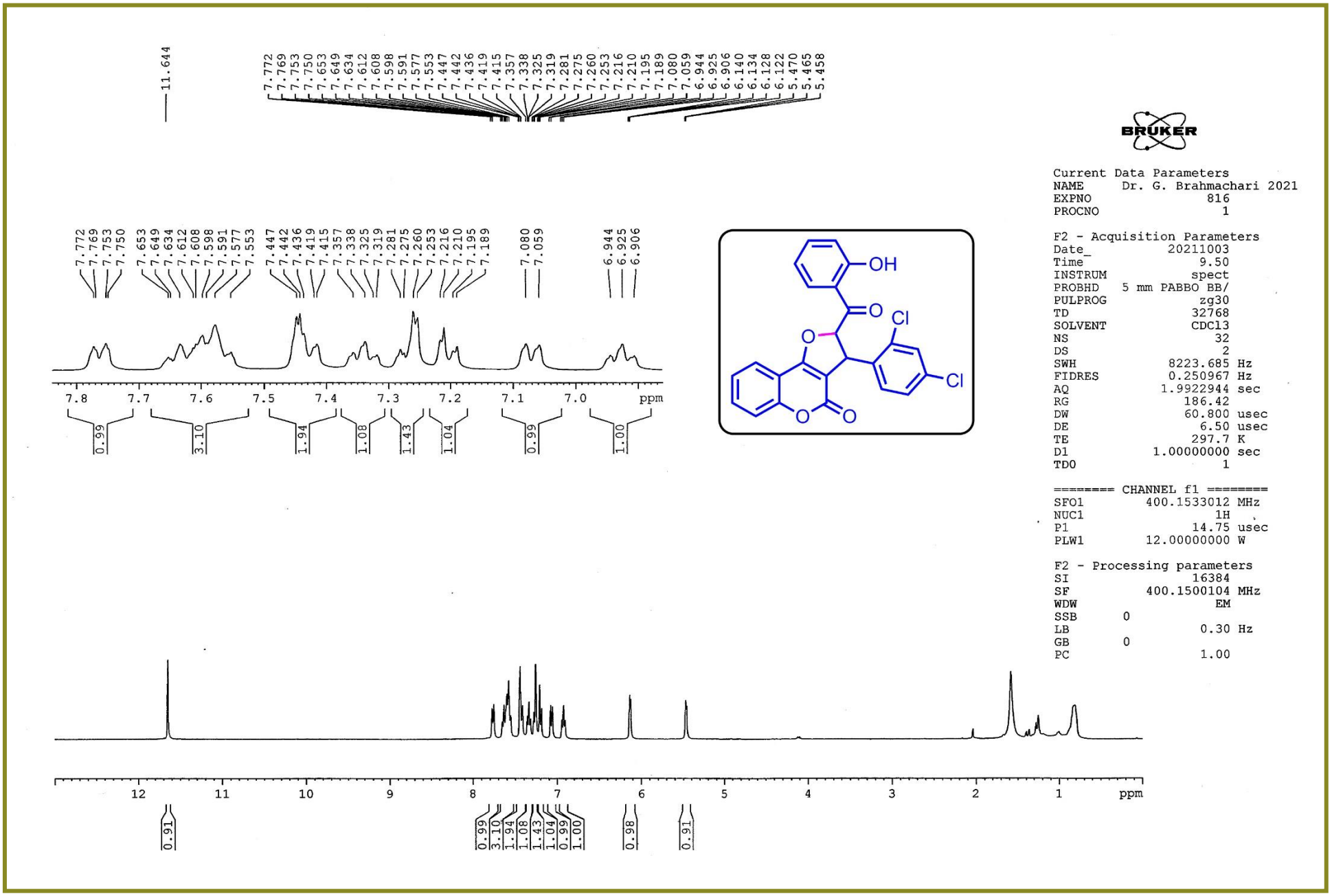


Figure S72. ^{1}H-NMR spectrum of 3-(2,4-dichlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-16**)

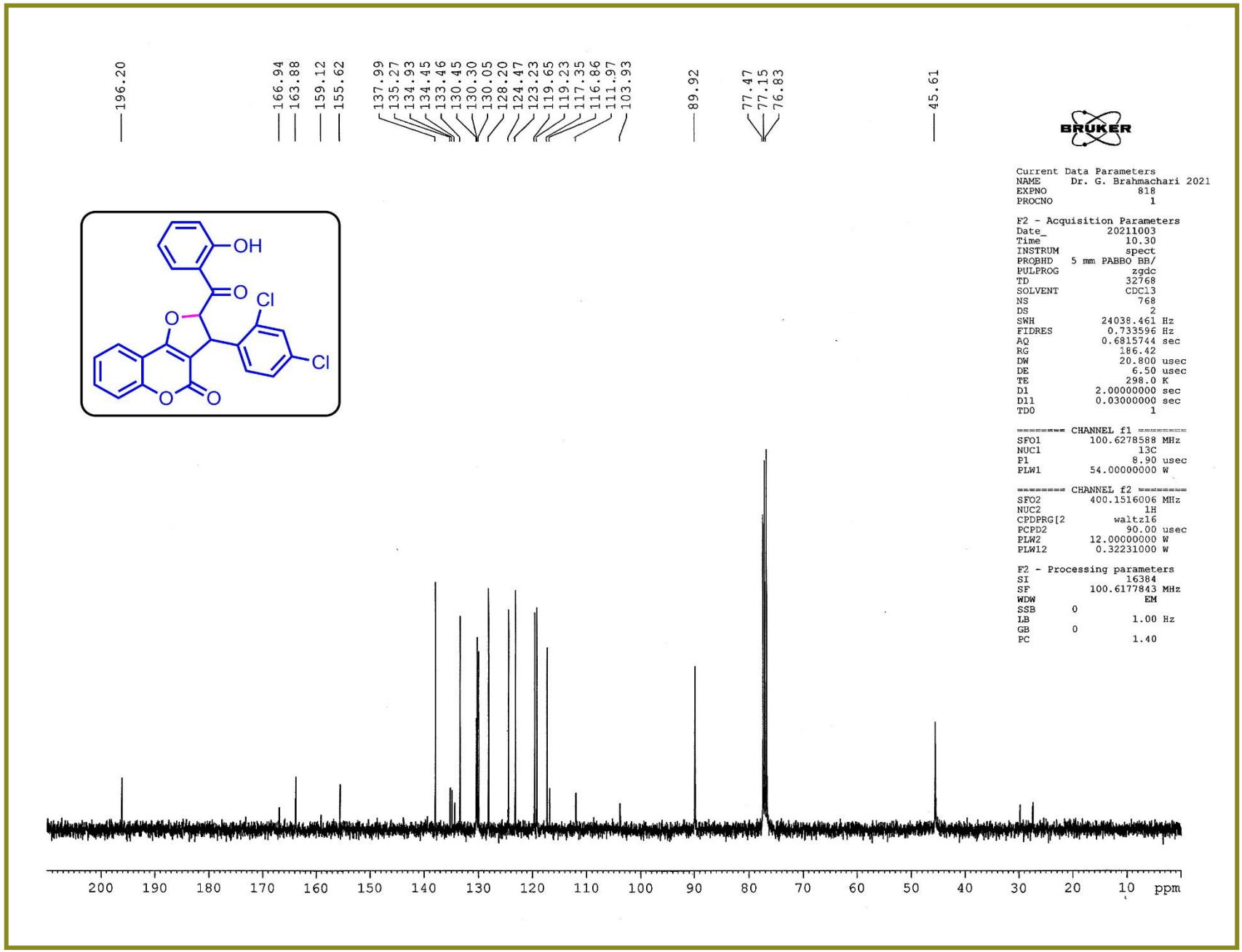


Figure S73. ^{13}C-NMR spectrum of 3-(2,4-dichlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-16**)

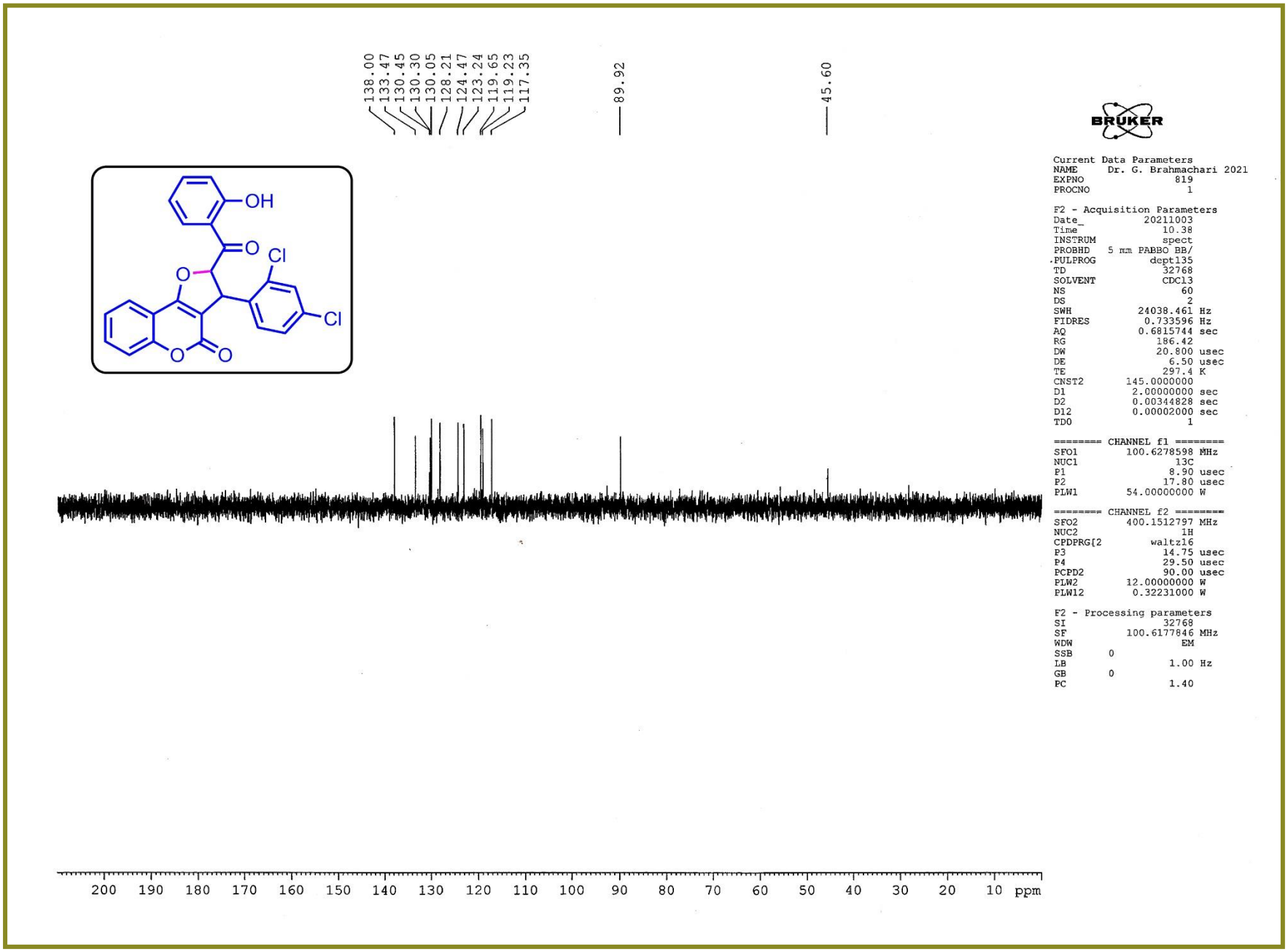


Figure S74. DEPT-135 NMR spectrum of 3-(2,4-dichlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-16**)

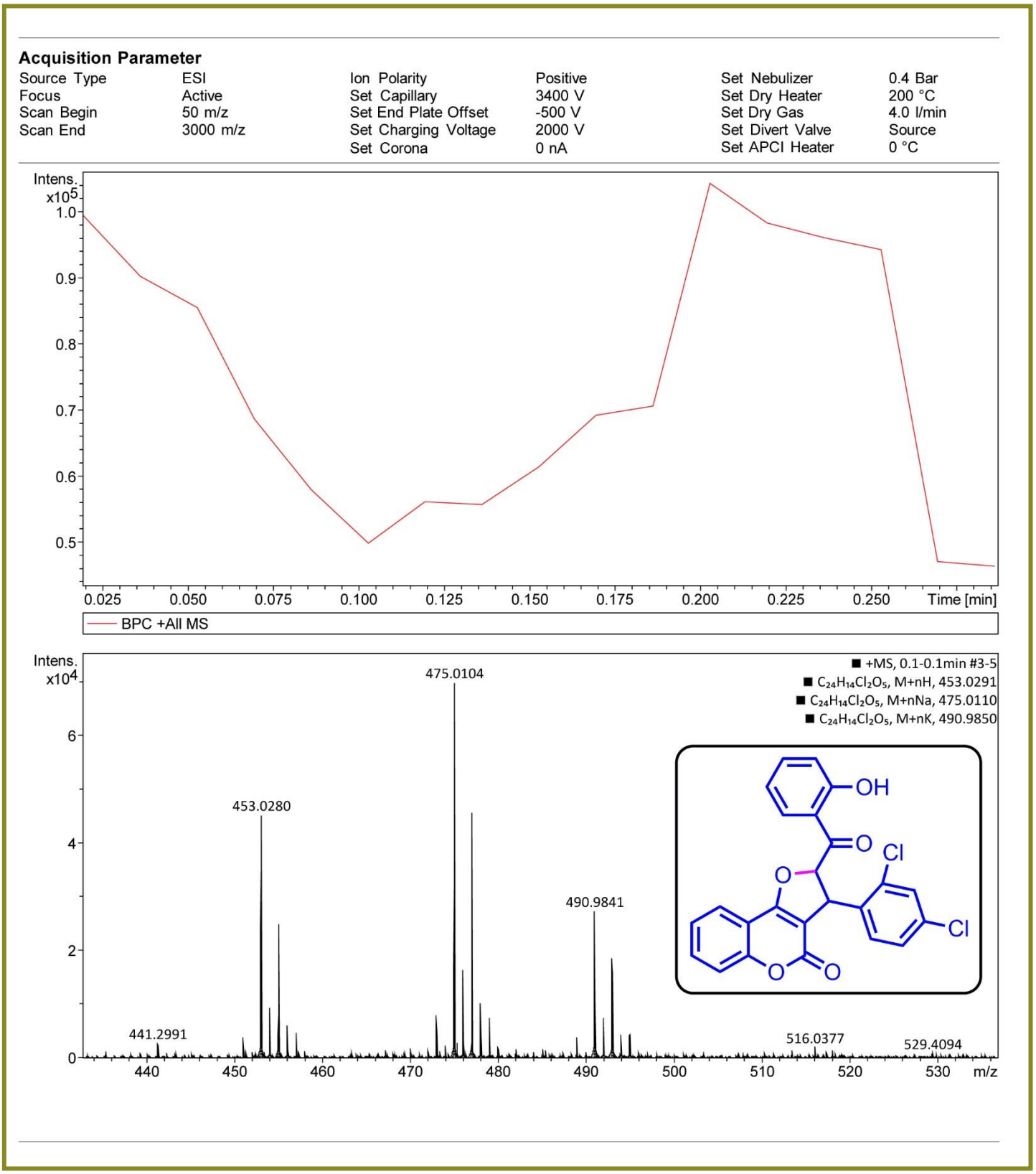


Figure S75. High-resolution Mass spectra of 3-(2,4-dichlorophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-16**)

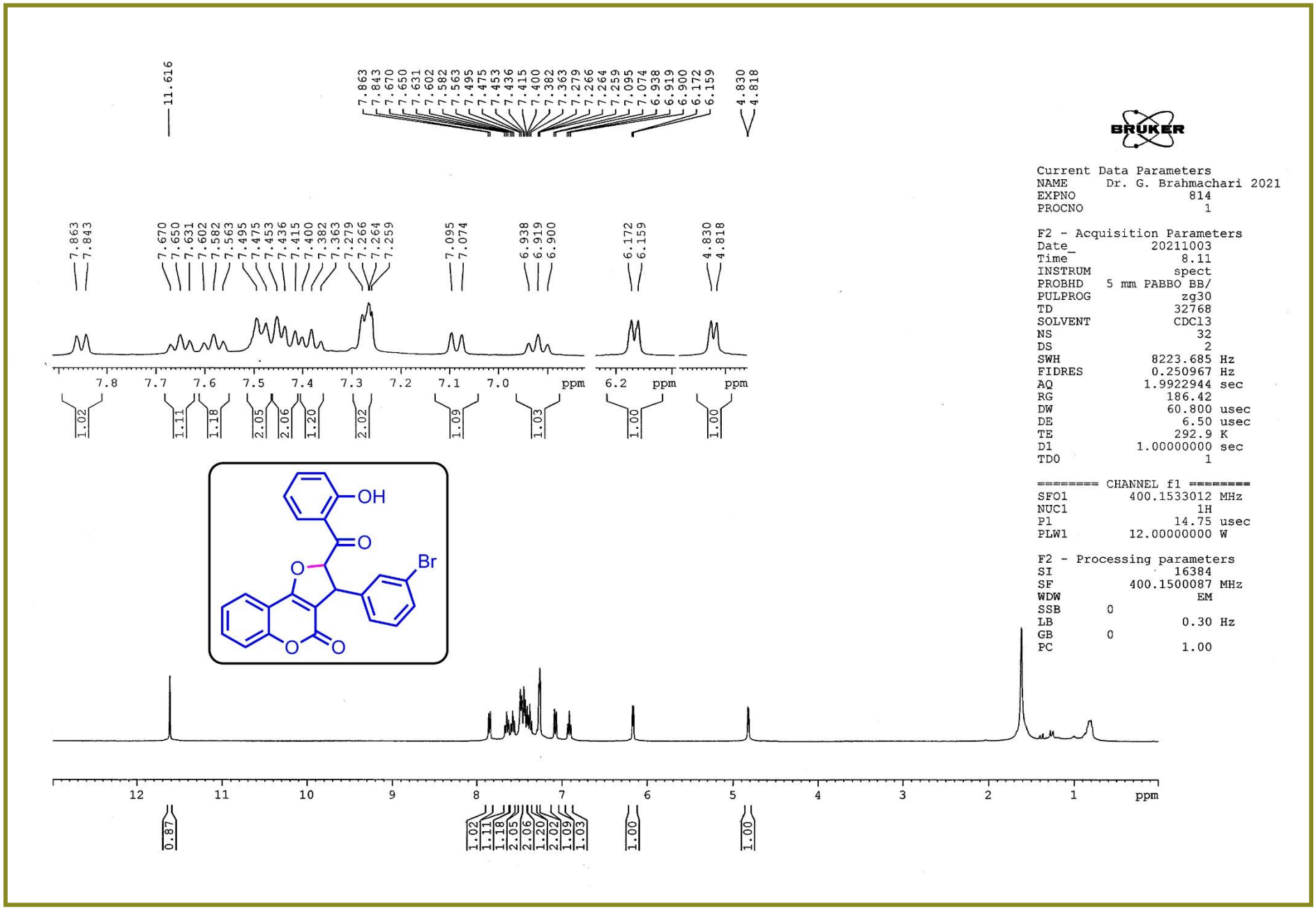


Figure S76. ^{1}H-NMR spectrum of 3-(3-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-17**)

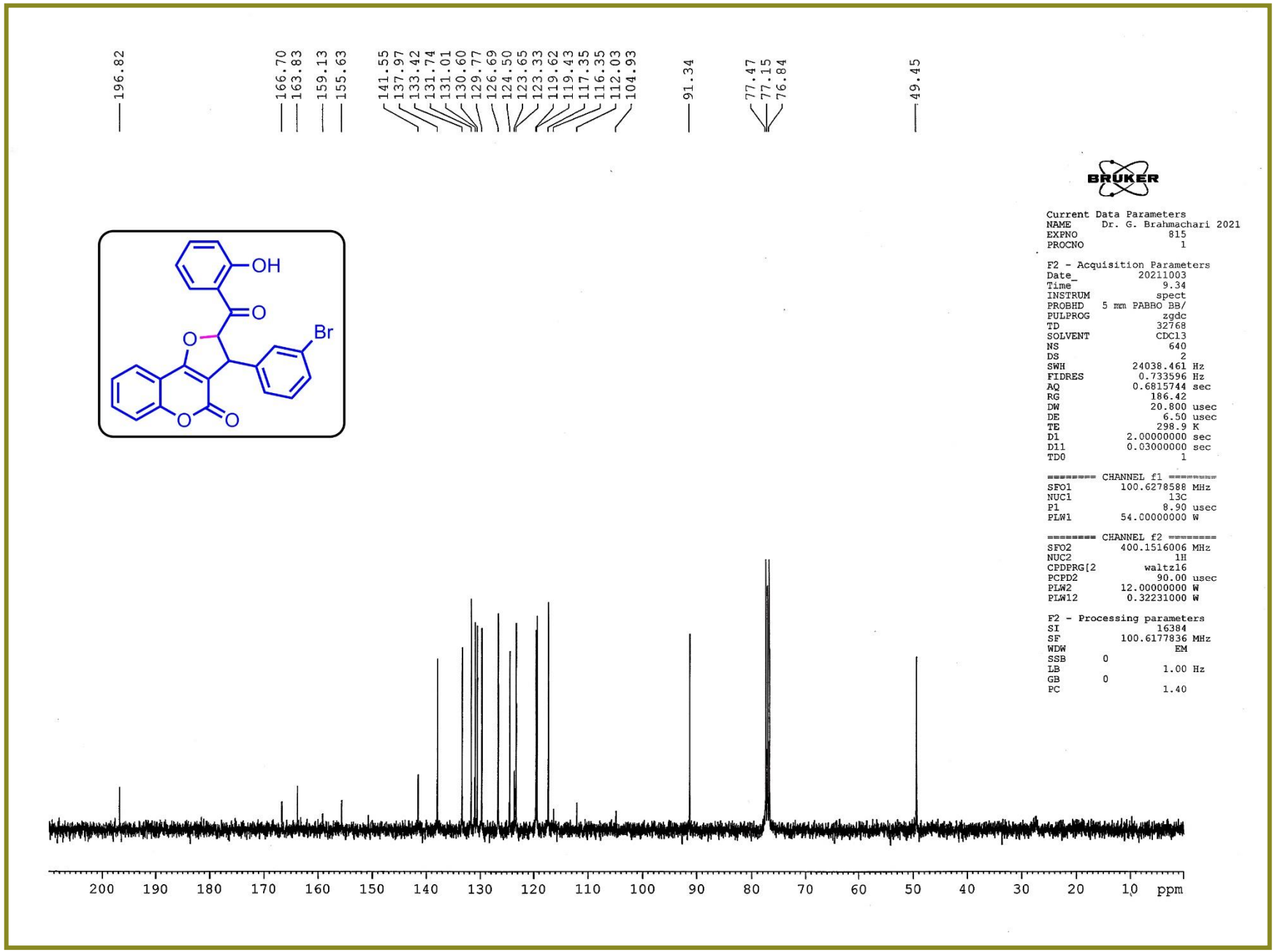


Figure S77. ^{13}C-NMR spectrum of 3-(3-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-17**)

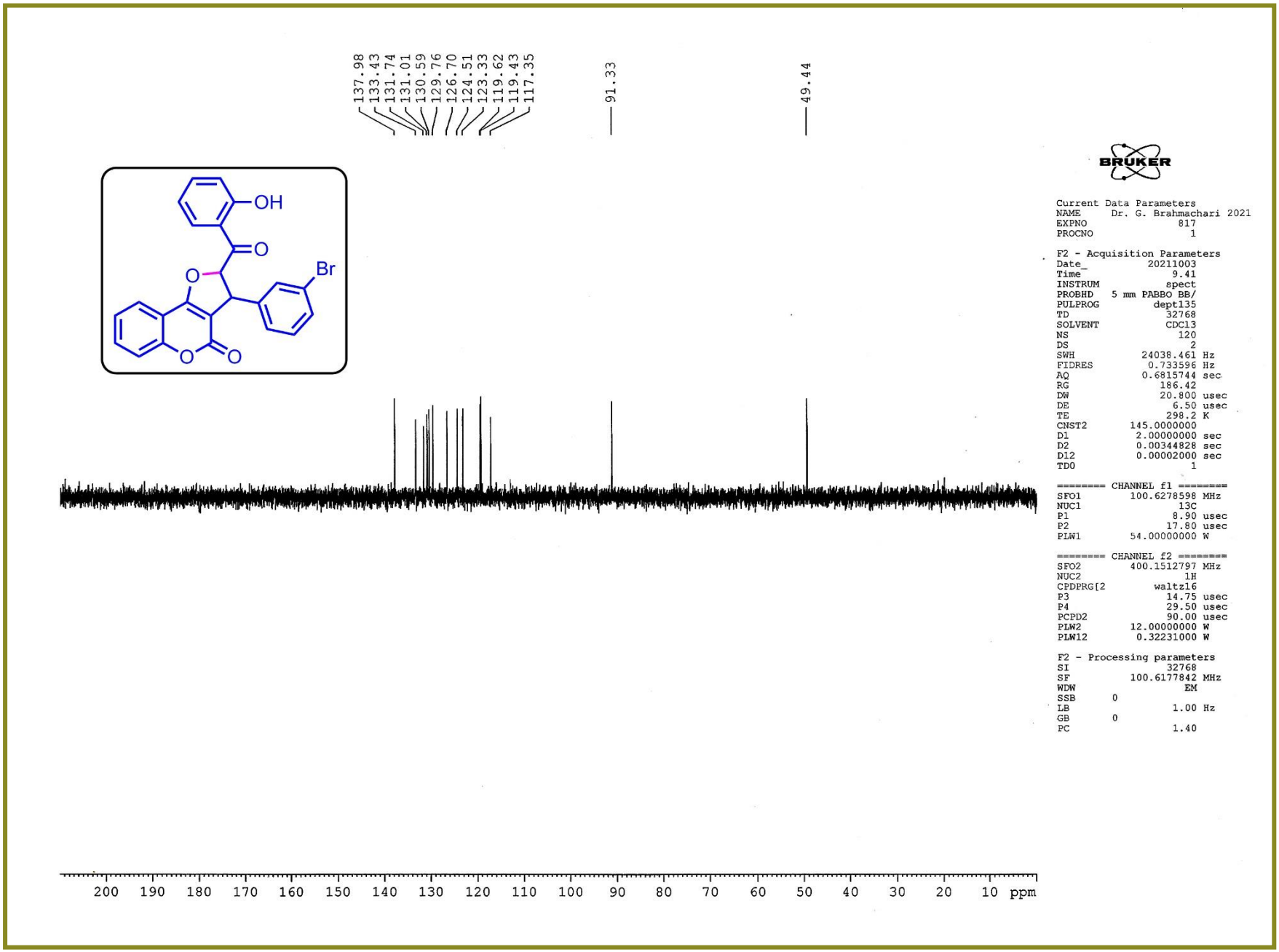


Figure S78. DEPT-135 NMR spectrum of 3-(3-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-17**)

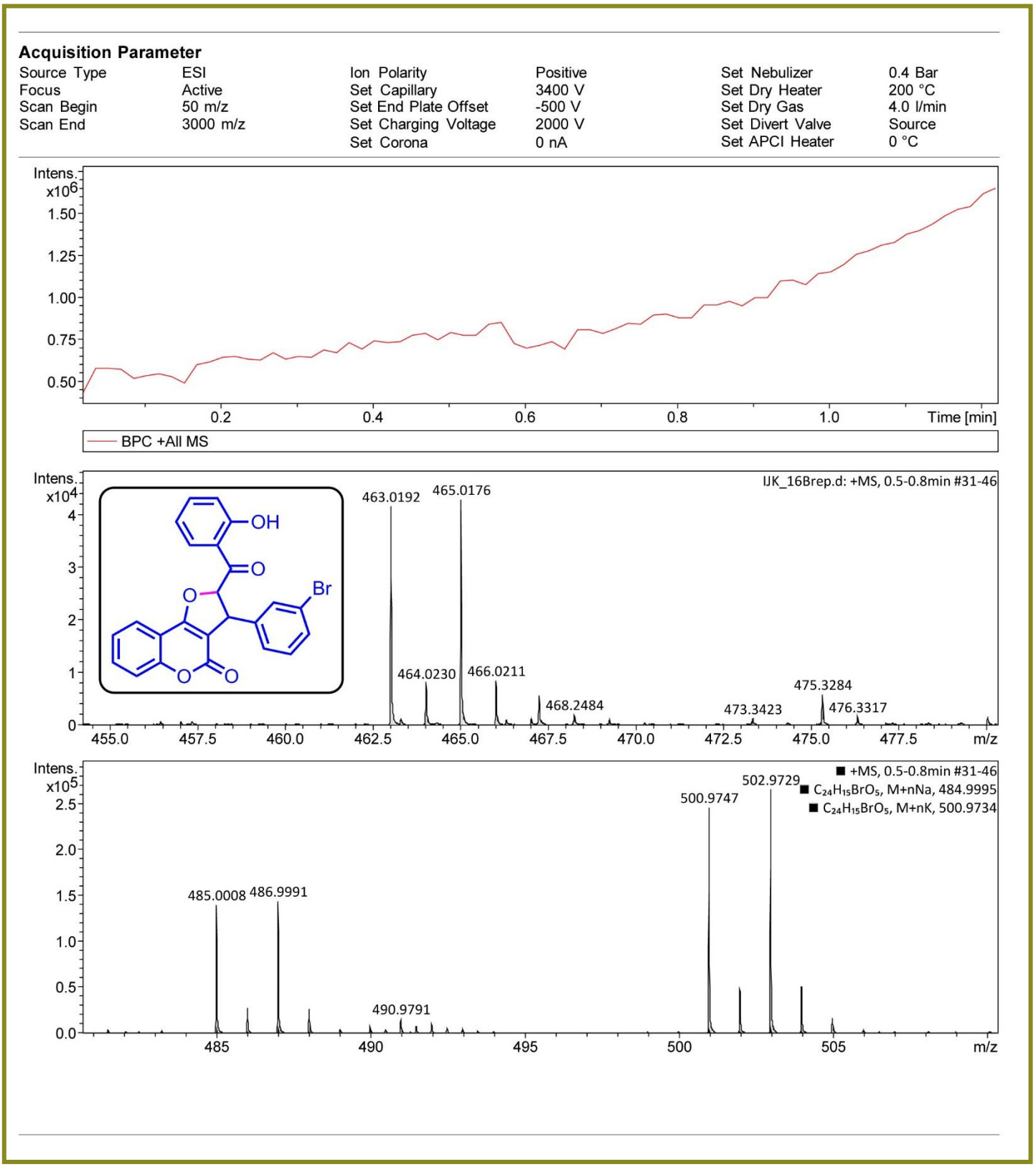


Figure S79. High-resolution Mass spectra of 3-(3-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-17**)

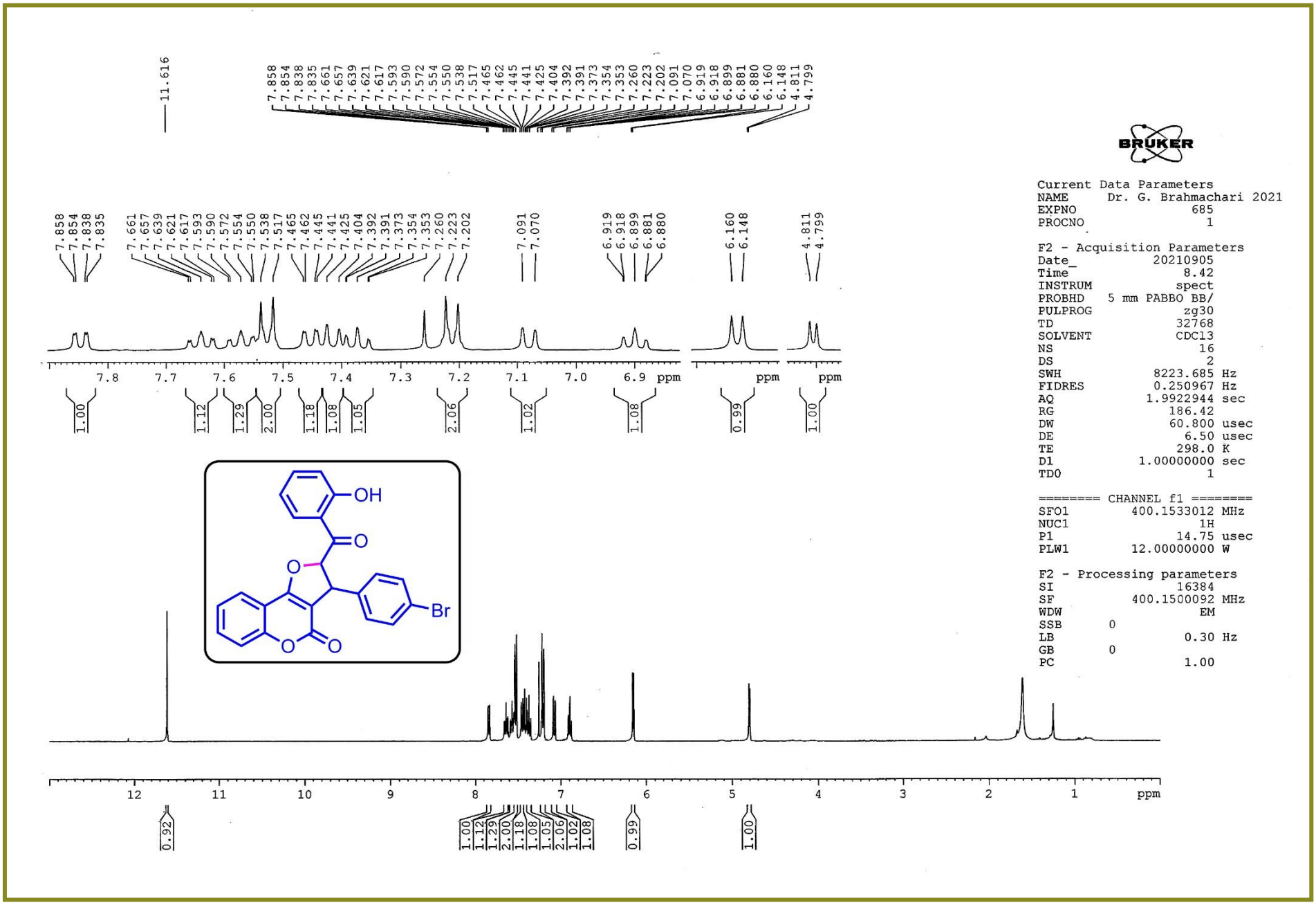


Figure S80. ^{1}H-NMR spectrum of 3-(4-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-18**)

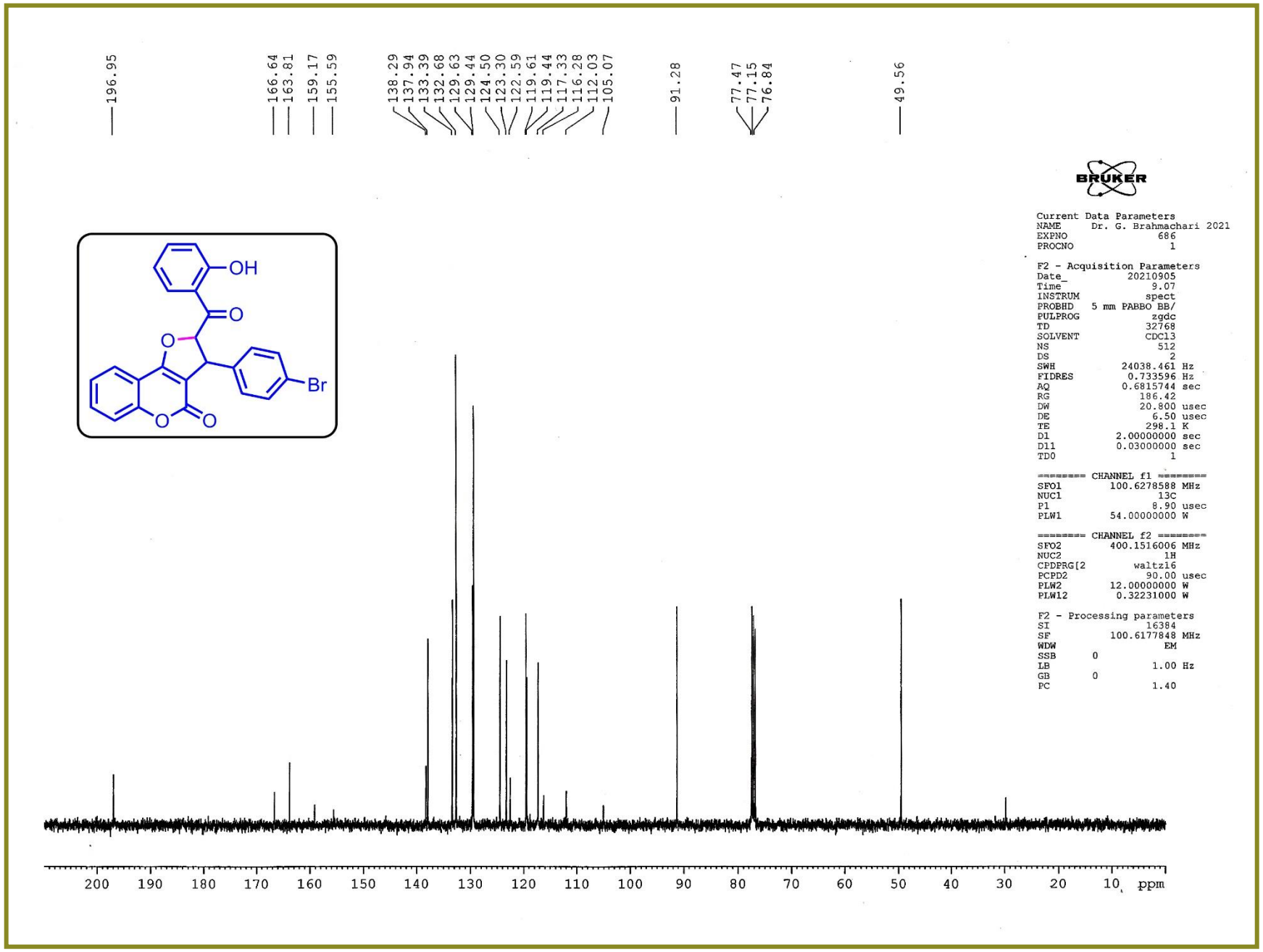


Figure S81. ^{13}C-NMR spectrum of 3-(4-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-18**)

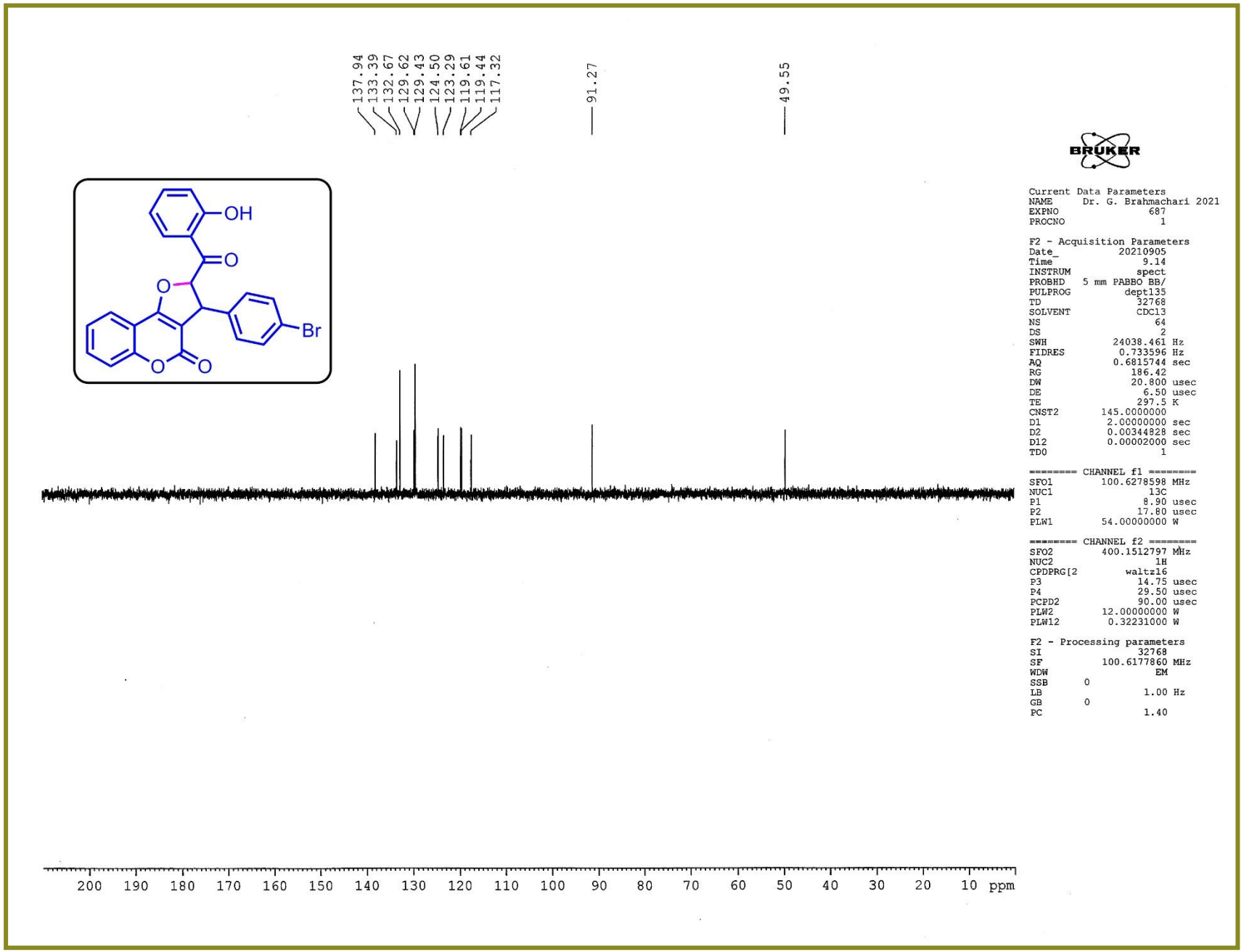


Figure S82. DEPT-135 NMR spectrum of 3-(4-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-18**)

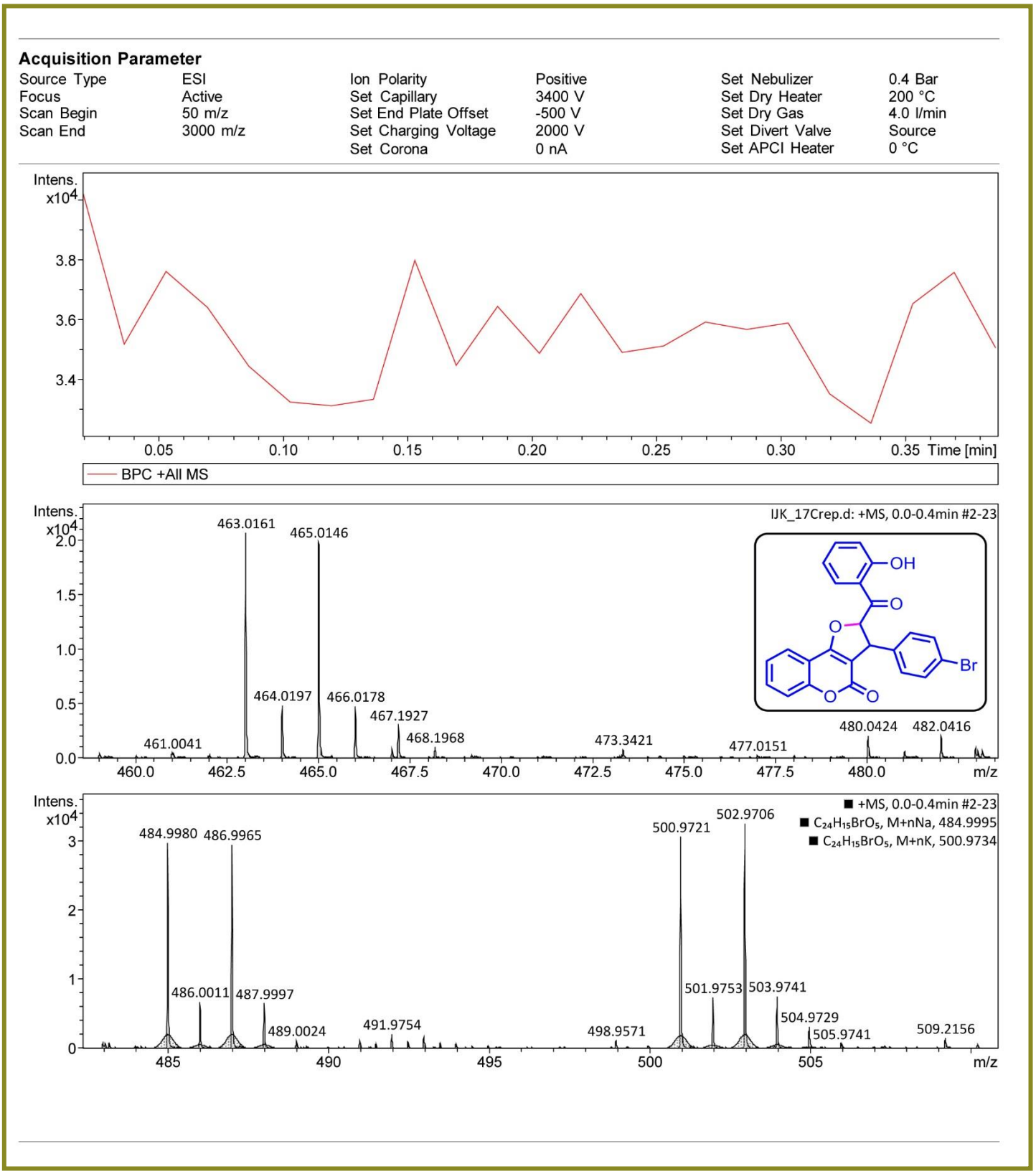


Figure S83. High-resolution Mass spectra of 3-(4-bromophenyl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-18**)

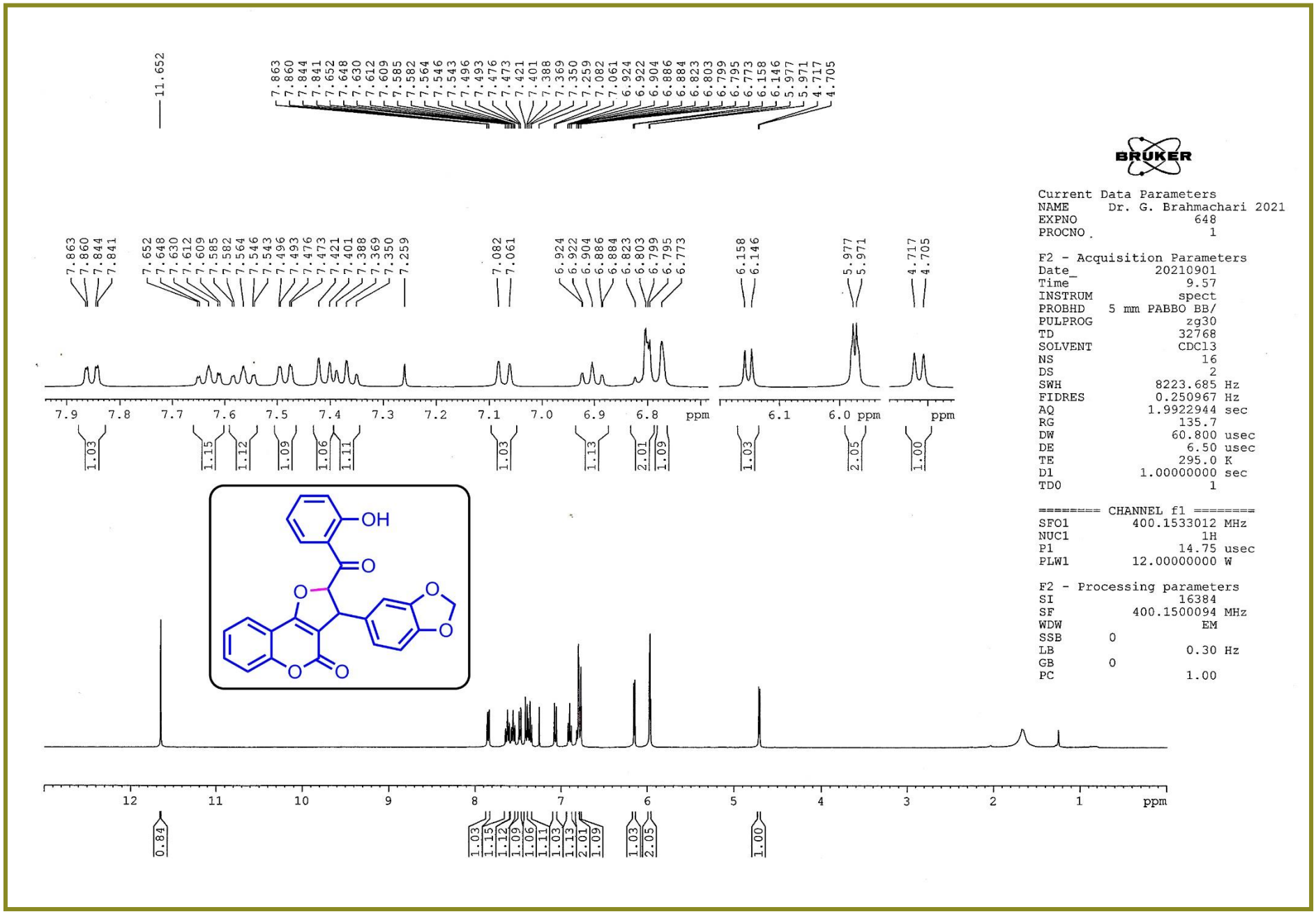


Figure S84. ^{1}H-NMR spectrum of 3-(benzo[*d*][1,3]dioxol-5-yl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-19**)

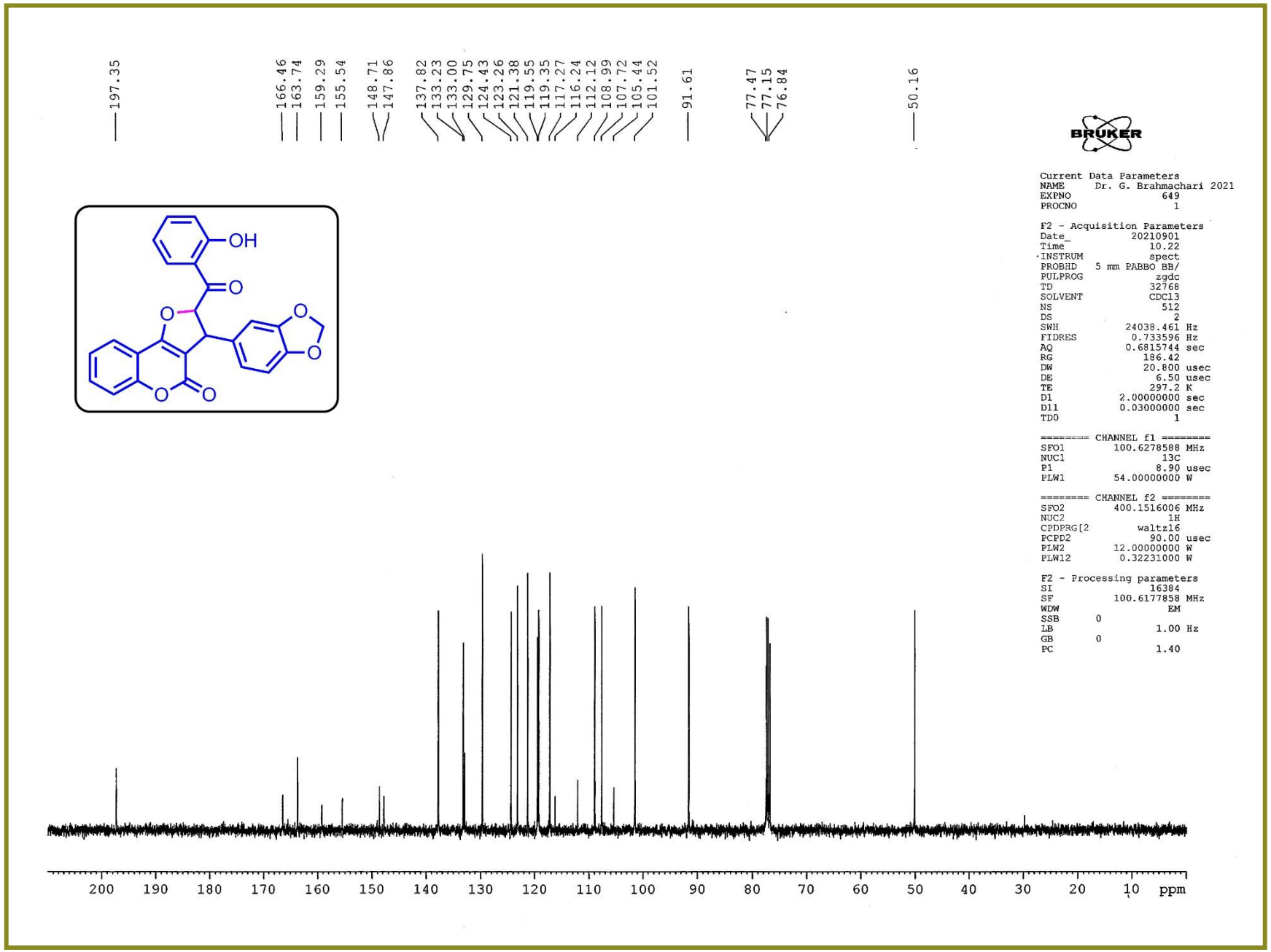


Figure S85. ^{13}C-NMR spectrum of 3-(benzo[*d*][1,3]dioxol-5-yl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-19**)

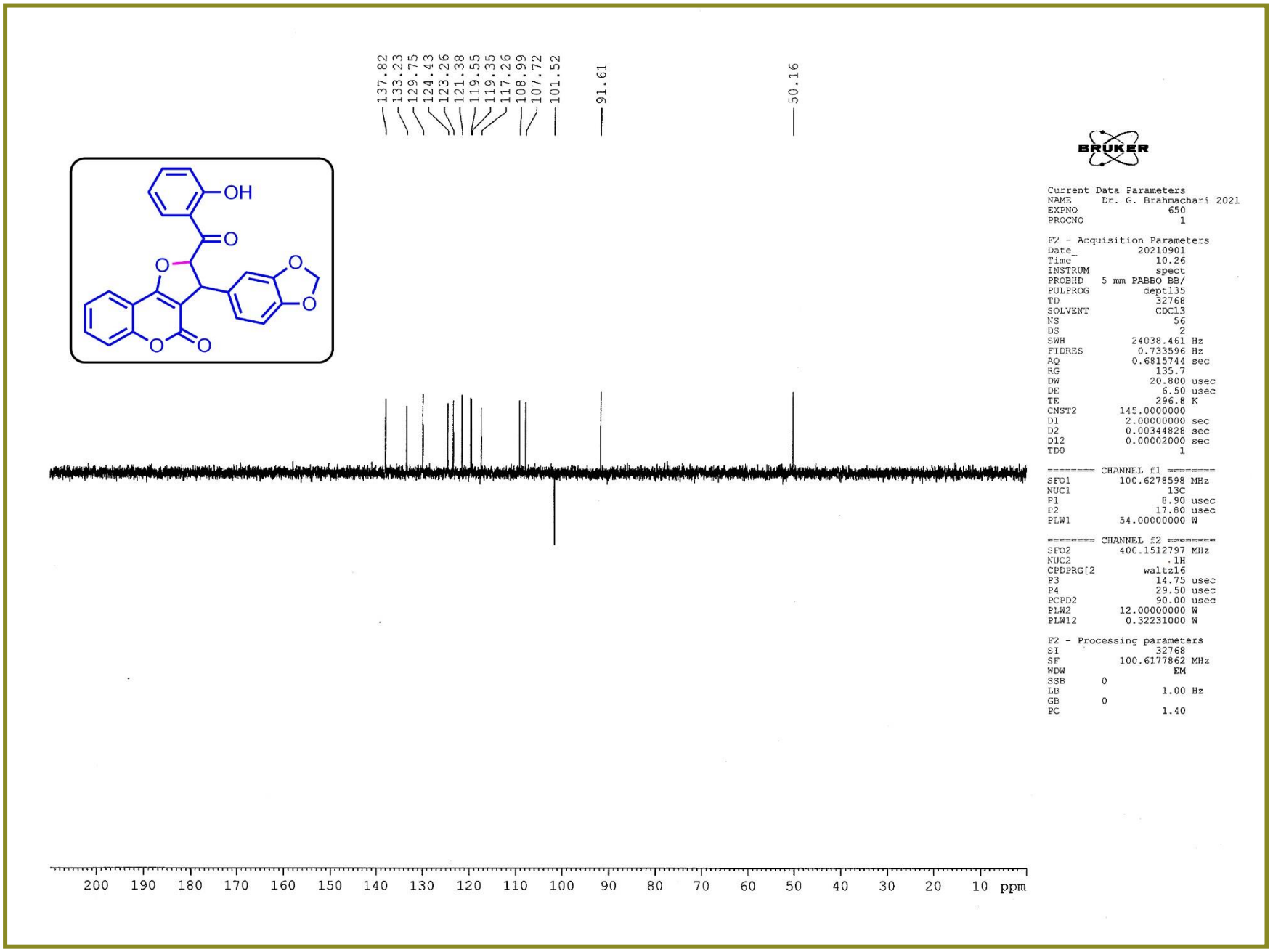


Figure S86. DEPT-135 NMR spectrum of 3-(benzo[*d*][1,3]dioxol-5-yl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-19**)

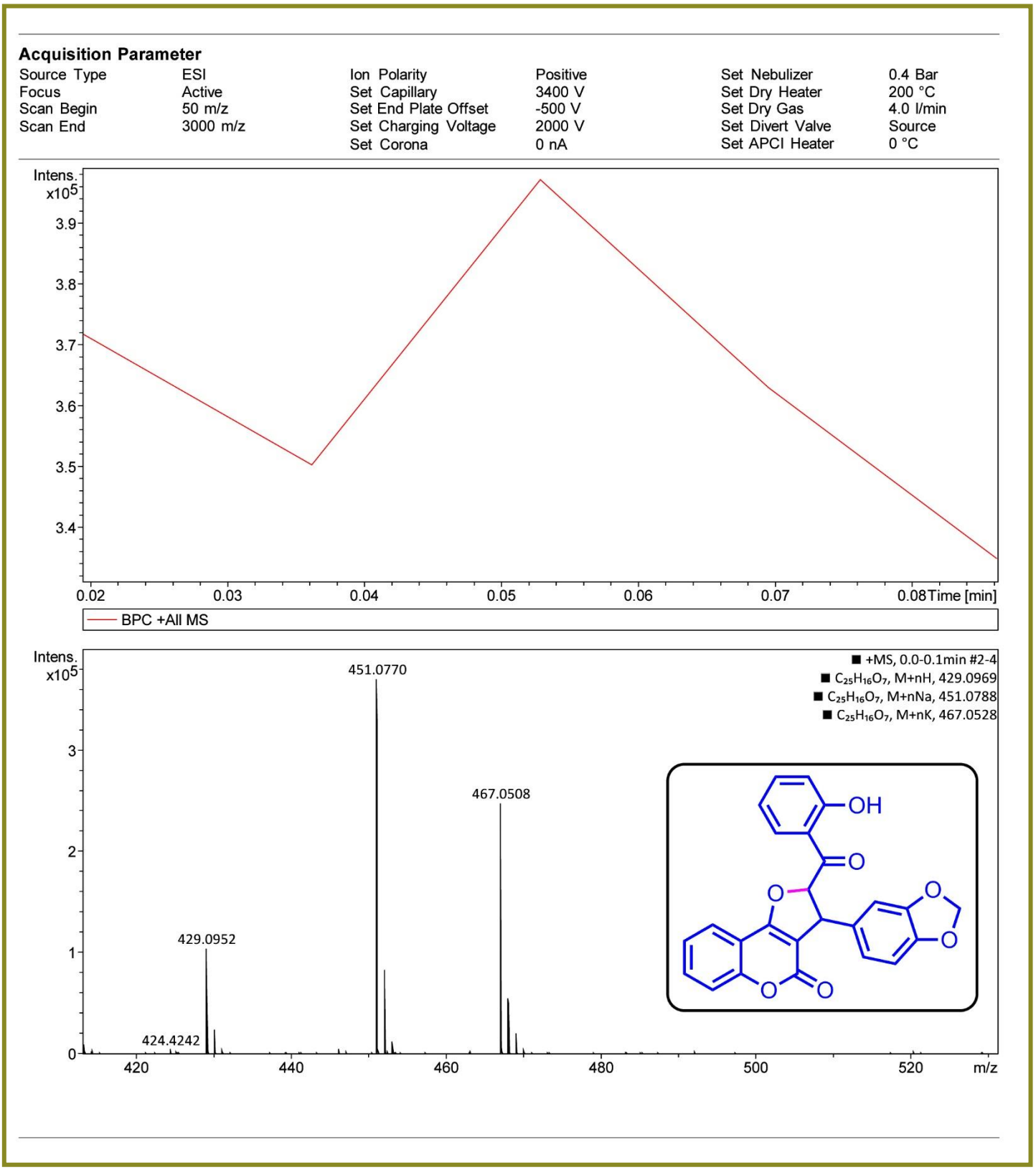


Figure S87. High-resolution Mass spectra of 3-(benzo[*d*][1,3]dioxol-5-yl)-2-(2-hydroxybenzoyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-19**)

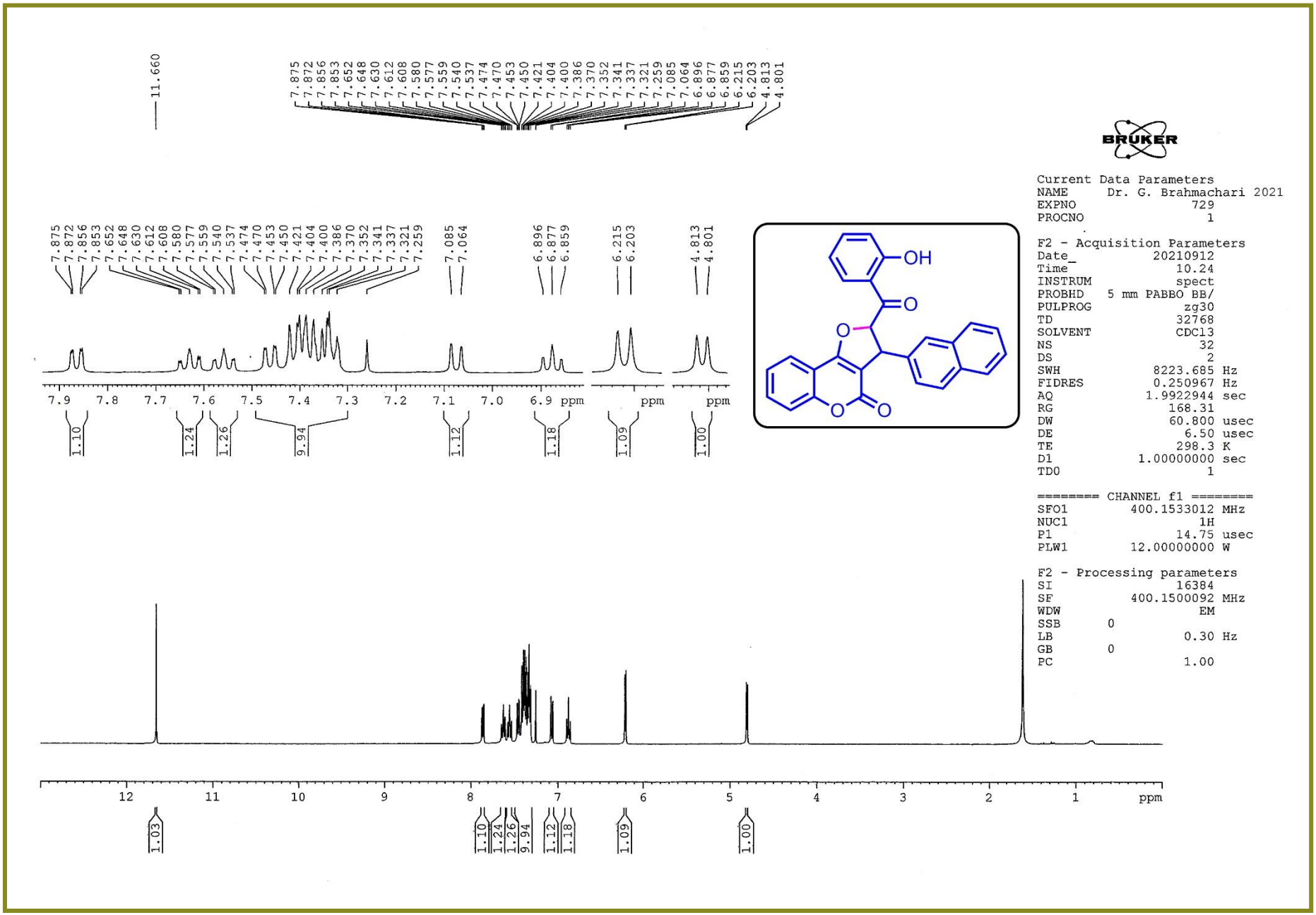


Figure S88. ^{1}H-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(naphthalen-2-yl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-20**)

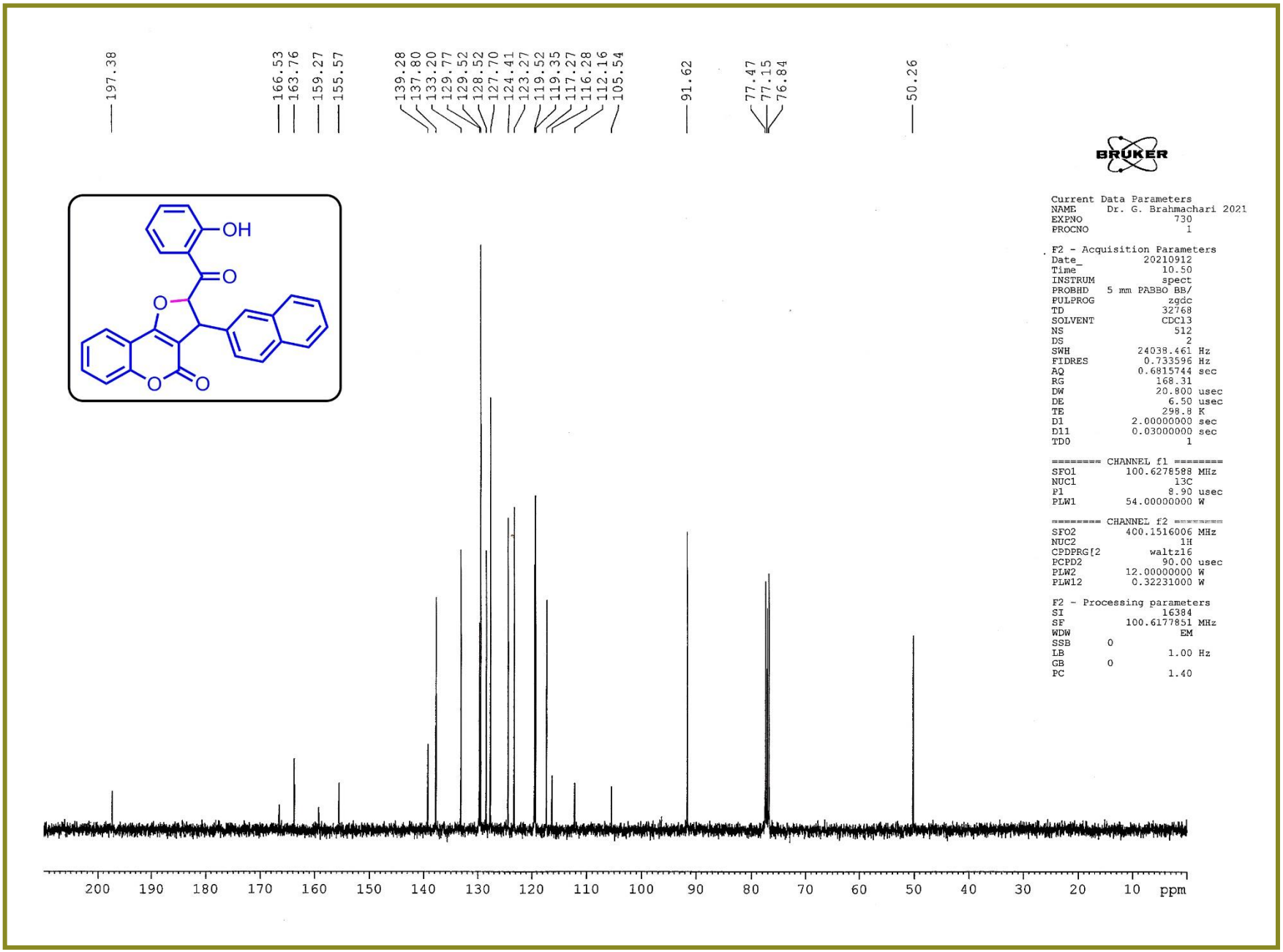


Figure S89. ^{13}C-NMR spectrum of 2-(2-hydroxybenzoyl)-3-(naphthalen-2-yl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-20**)

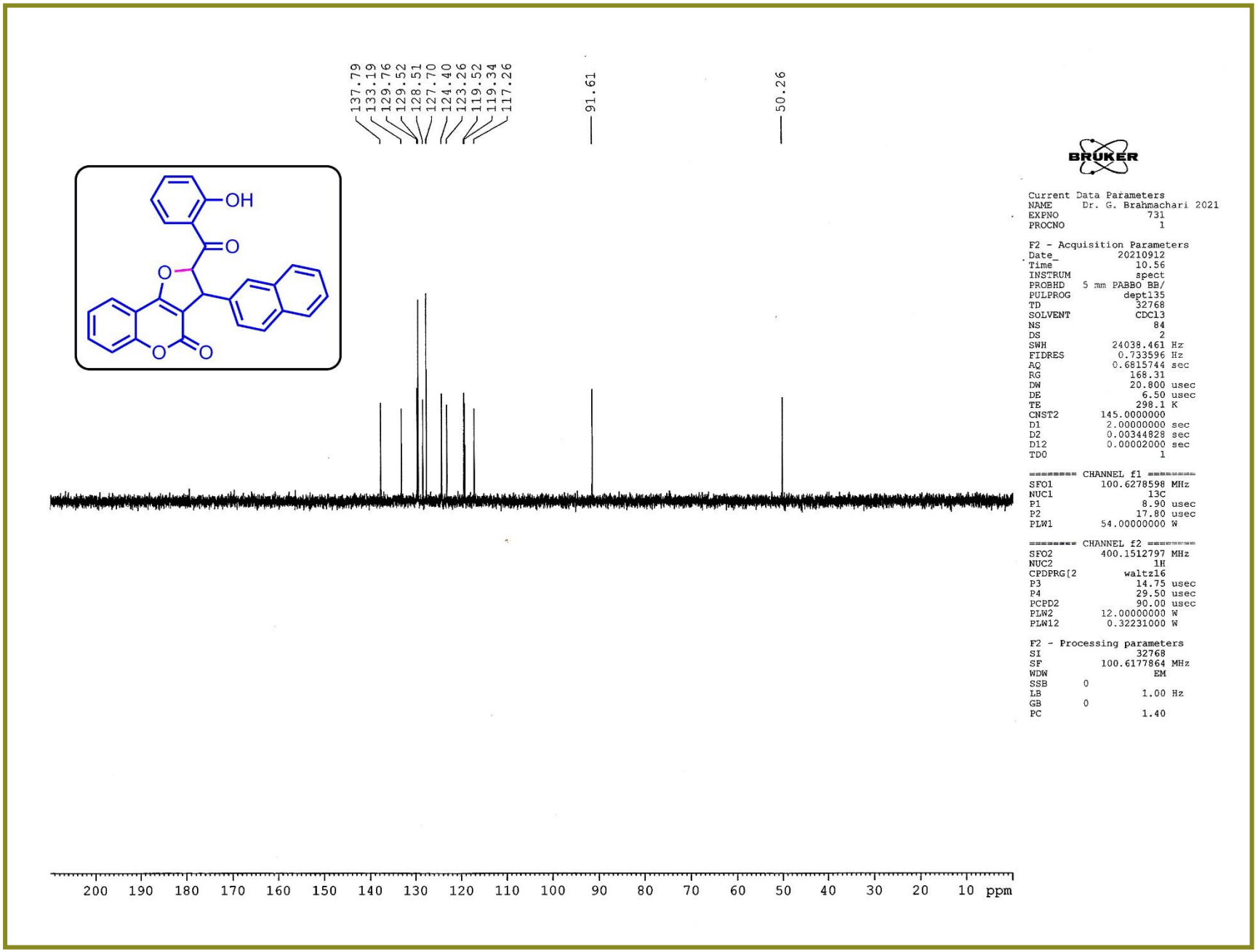


Figure S90. DEPT-135 NMR spectrum of 2-(2-hydroxybenzoyl)-3-(naphthalen-2-yl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-20**)

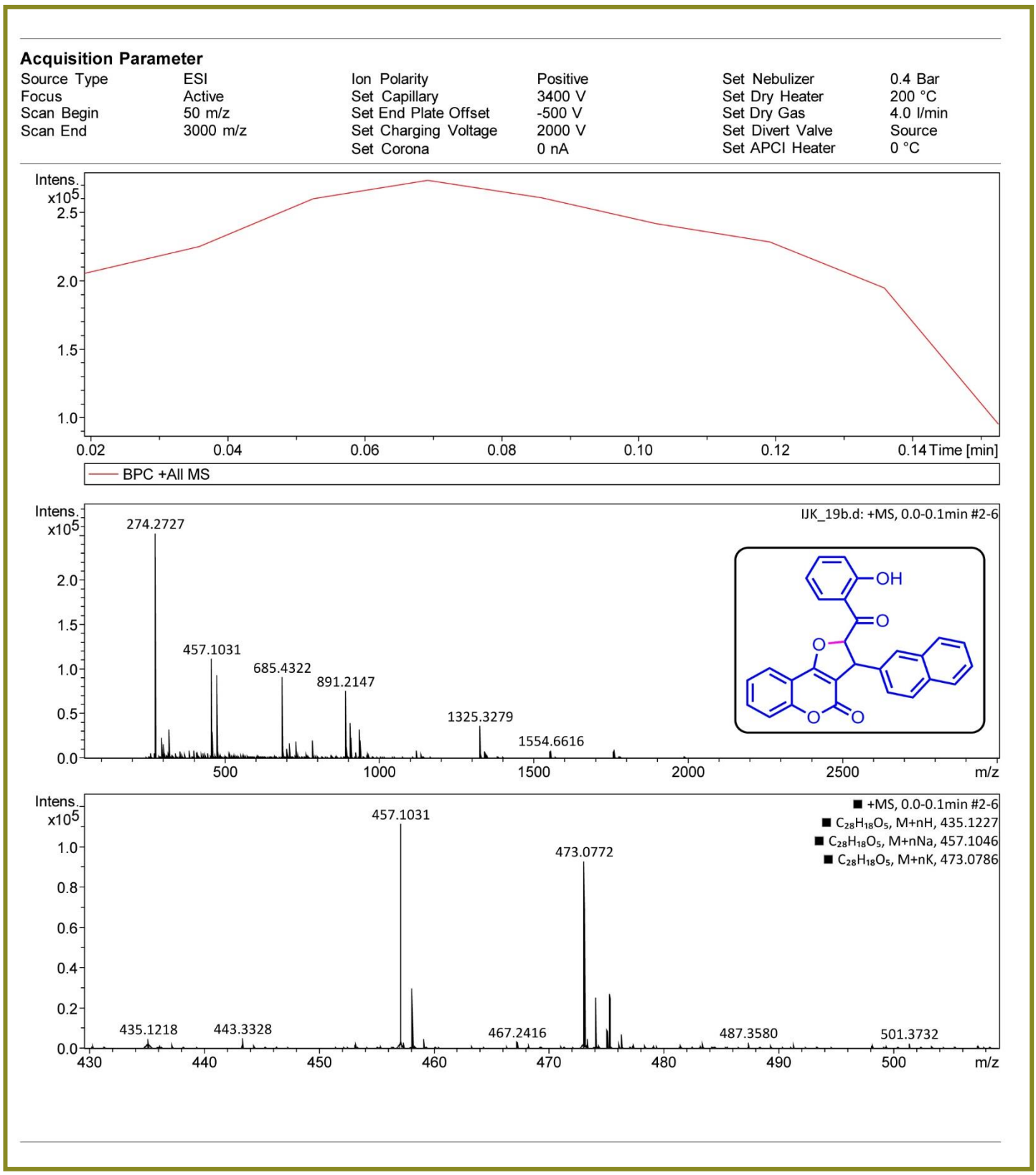


Figure S91. High-resolution Mass spectra of 2-(2-hydroxybenzoyl)-3-(naphthalen-2-yl)-2,3-dihydro-4*H*-furo[3,2-*c*] chromen-4-one (**2-20**)

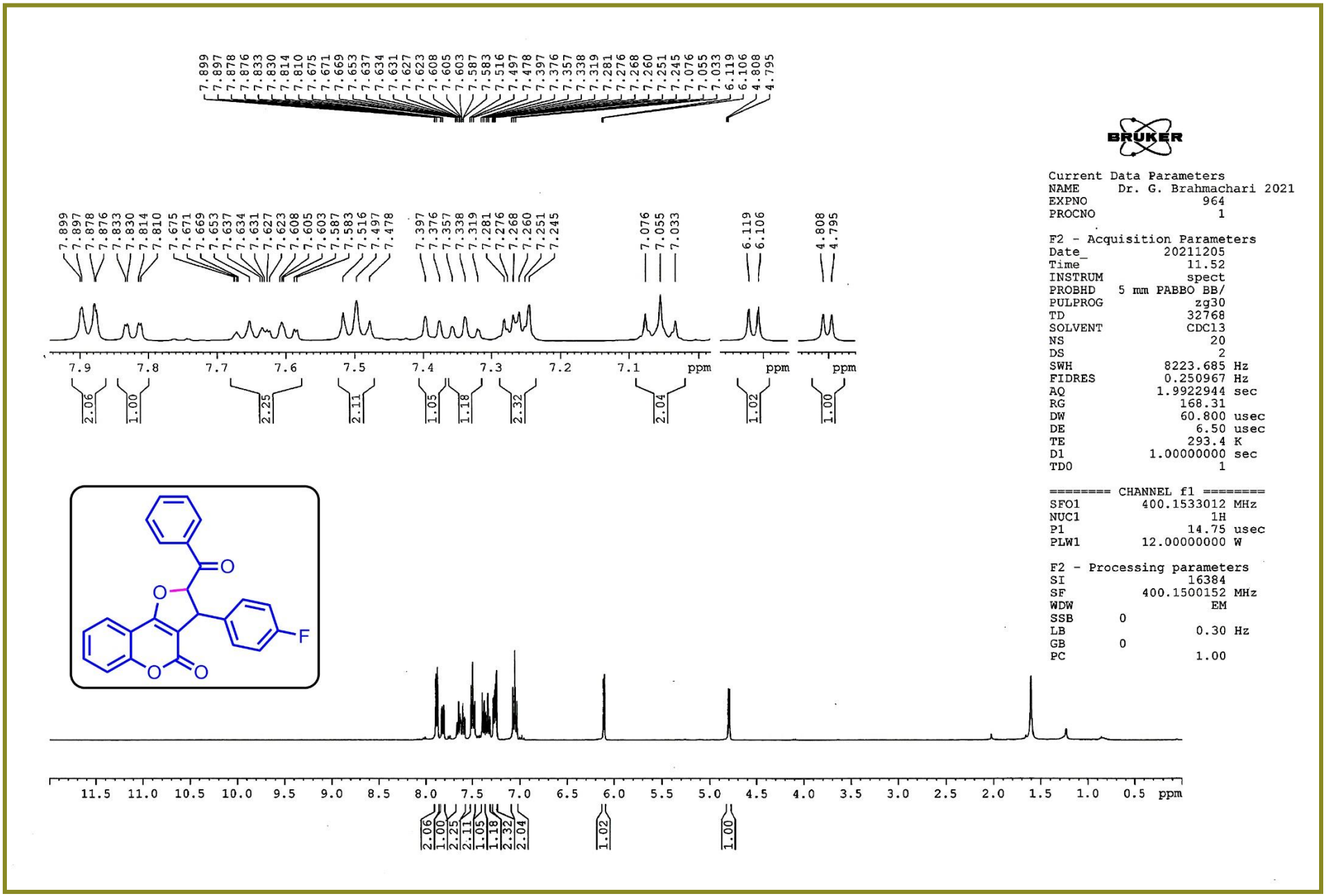


Figure S92. ^{1}H-NMR spectrum of 2-benzoyl-3-(4-fluorophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-21**)

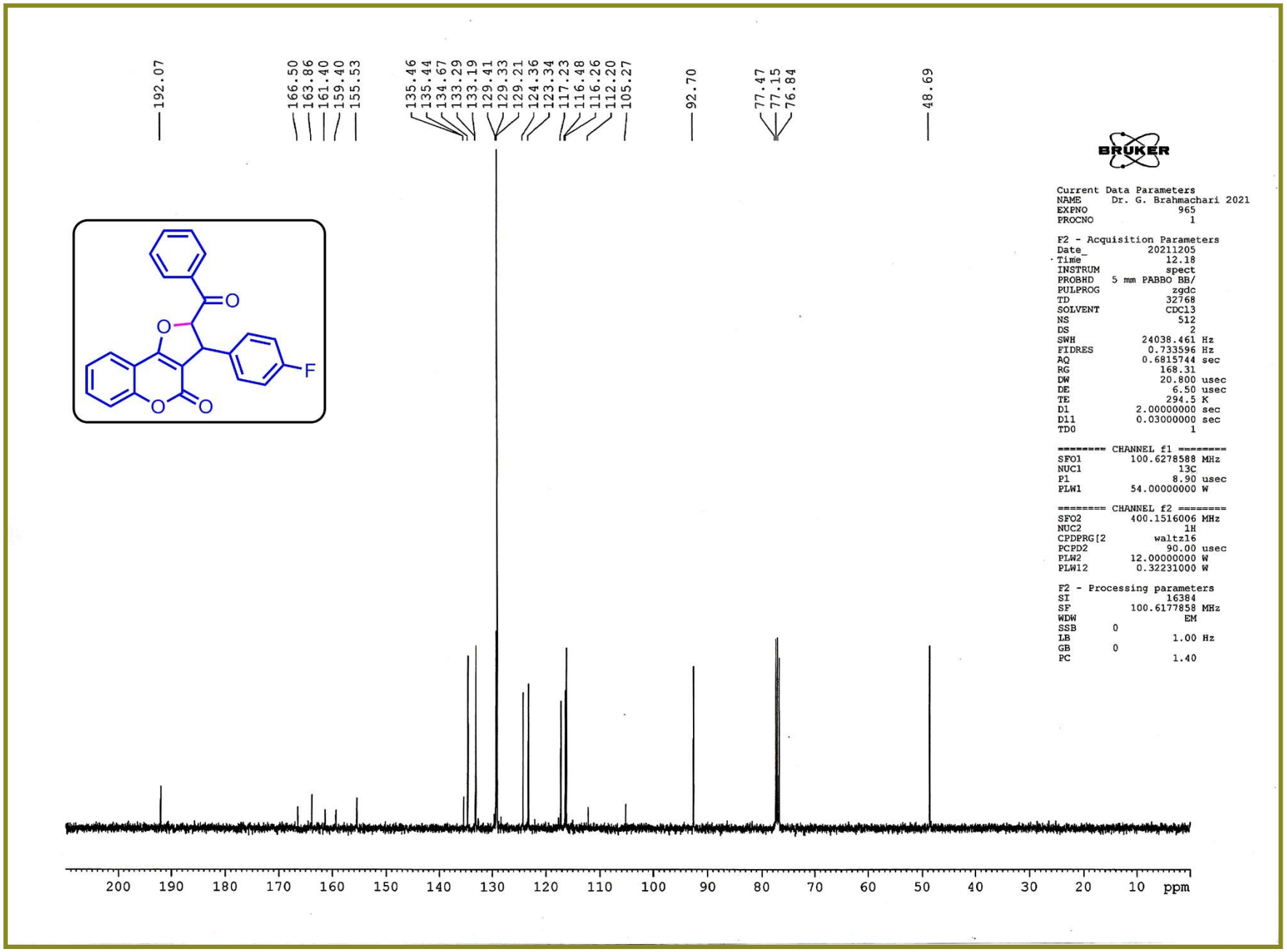


Figure S93. ^{13}C-NMR spectrum of 2-benzoyl-3-(4-fluorophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-21**)

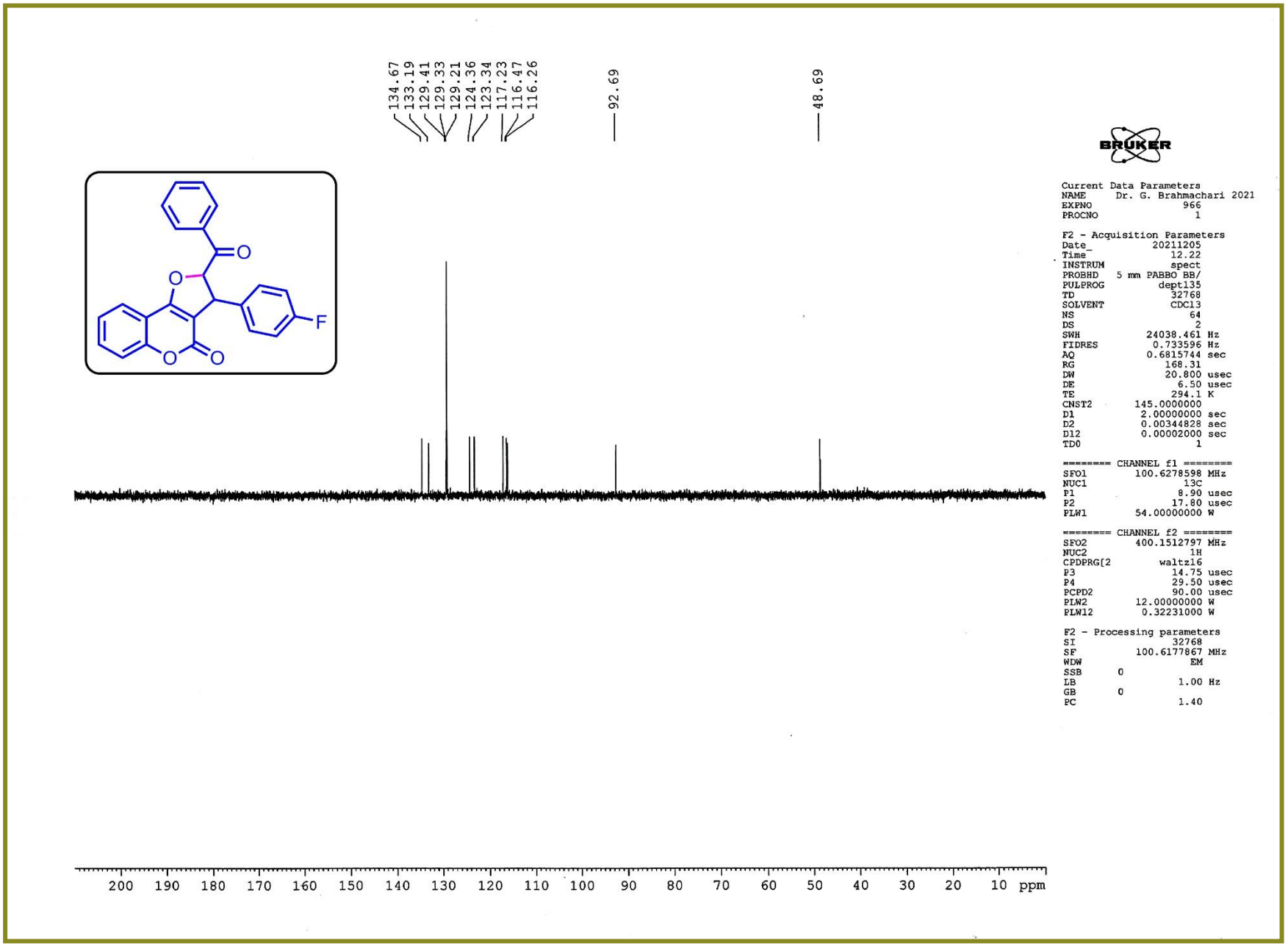


Figure S94. DEPT-135 NMR spectrum of 2-benzoyl-3-(4-fluorophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-21**)

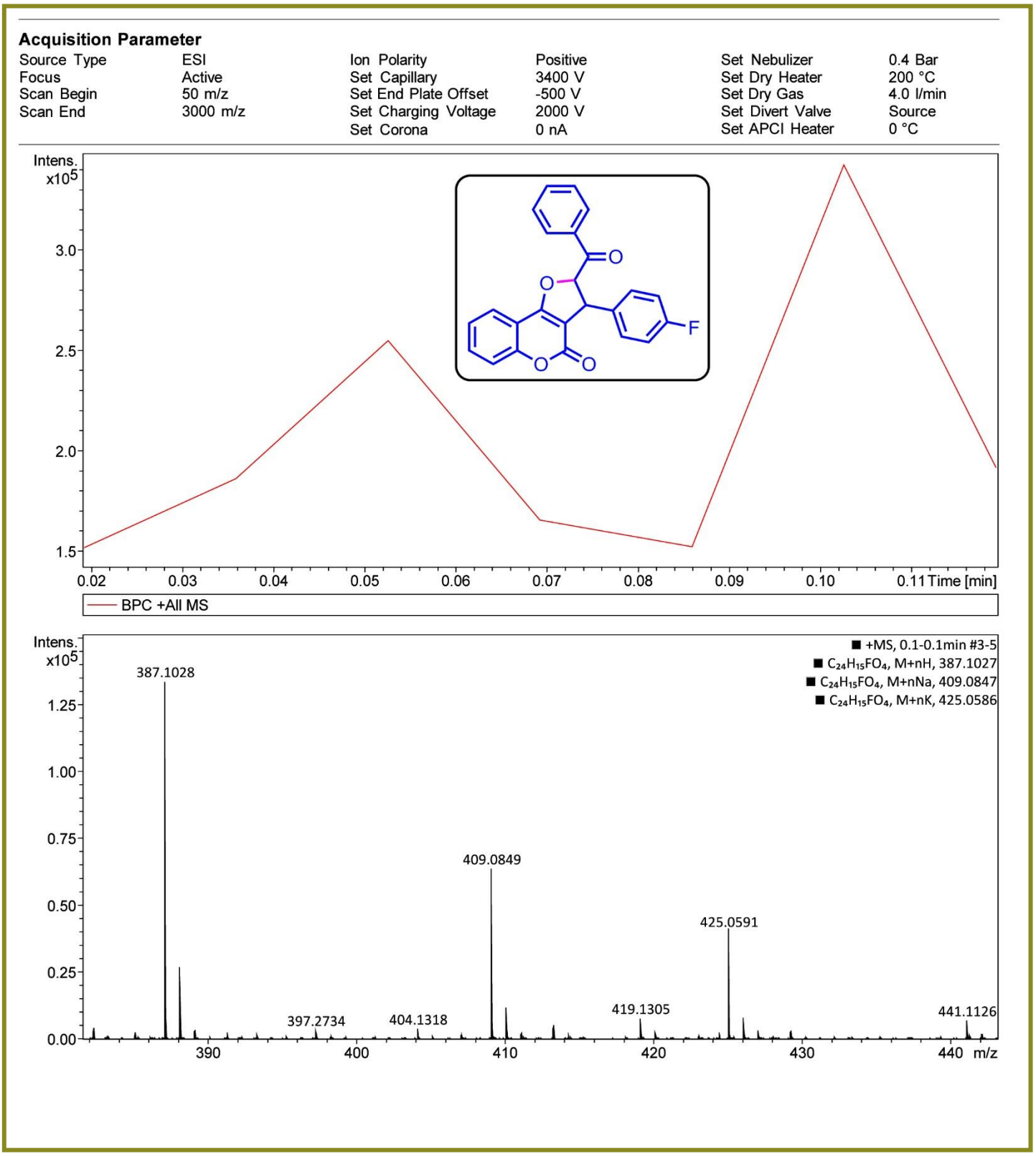


Figure S95. High-resolution Mass spectra of 2-benzoyl-3-(4-fluorophenyl)-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-21**)

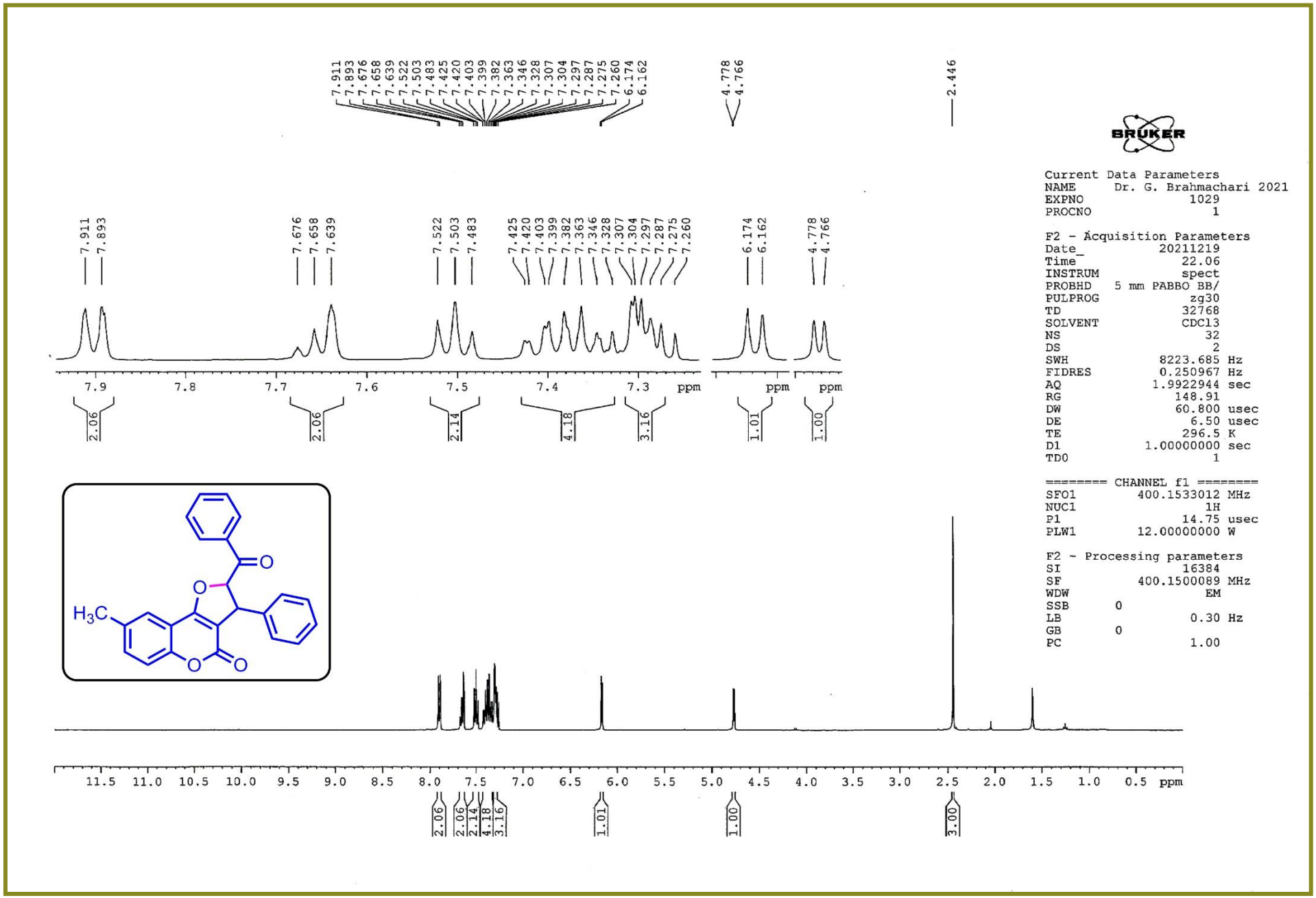


Figure S96. ^{1}H-NMR spectrum of 2-benzoyl-8-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-22**)

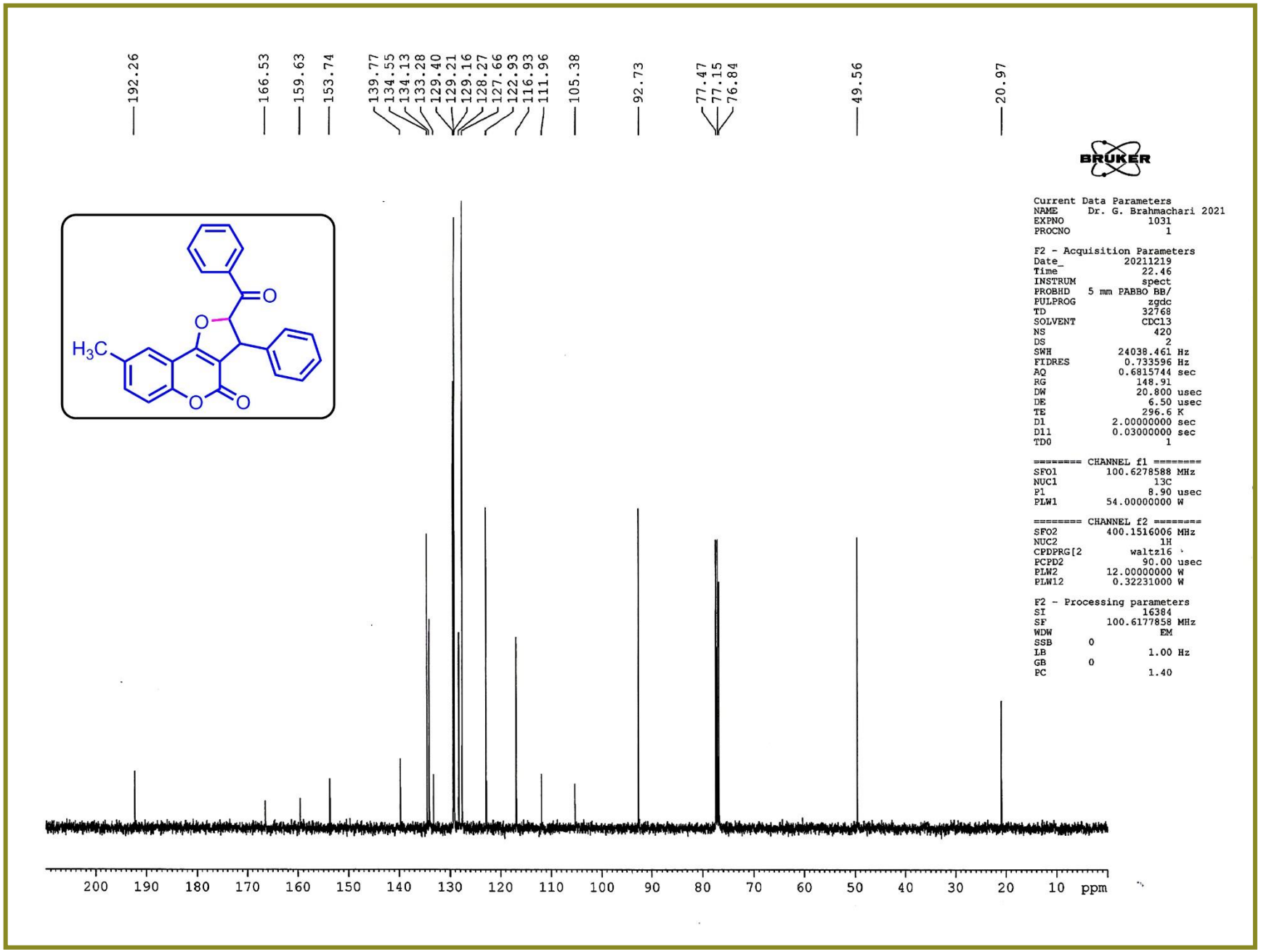


Figure S97. ^{13}C-NMR spectrum of 2-benzoyl-8-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-22**)

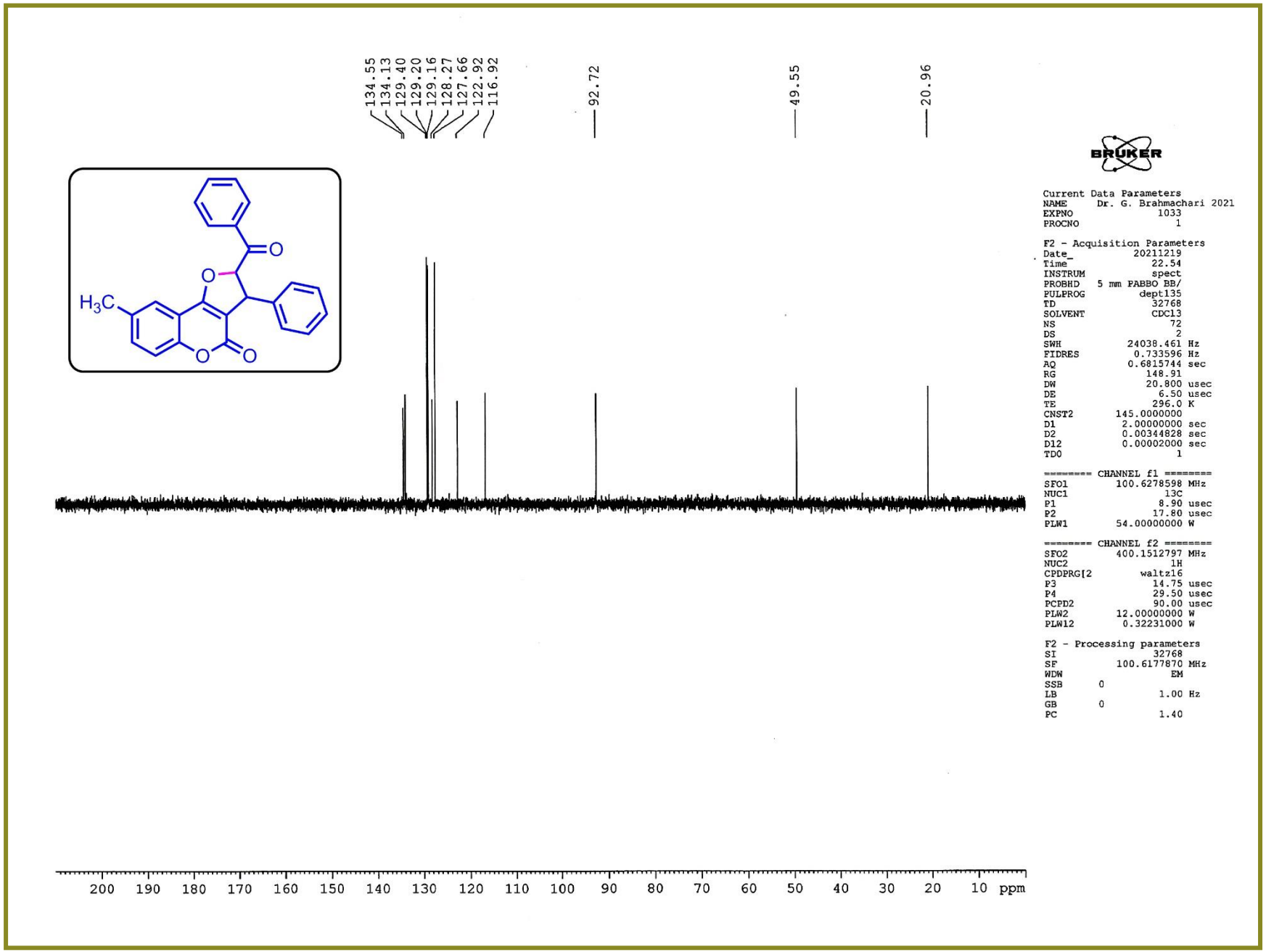


Figure S98. DEPT-135 NMR spectrum of 2-benzoyl-8-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-22**)

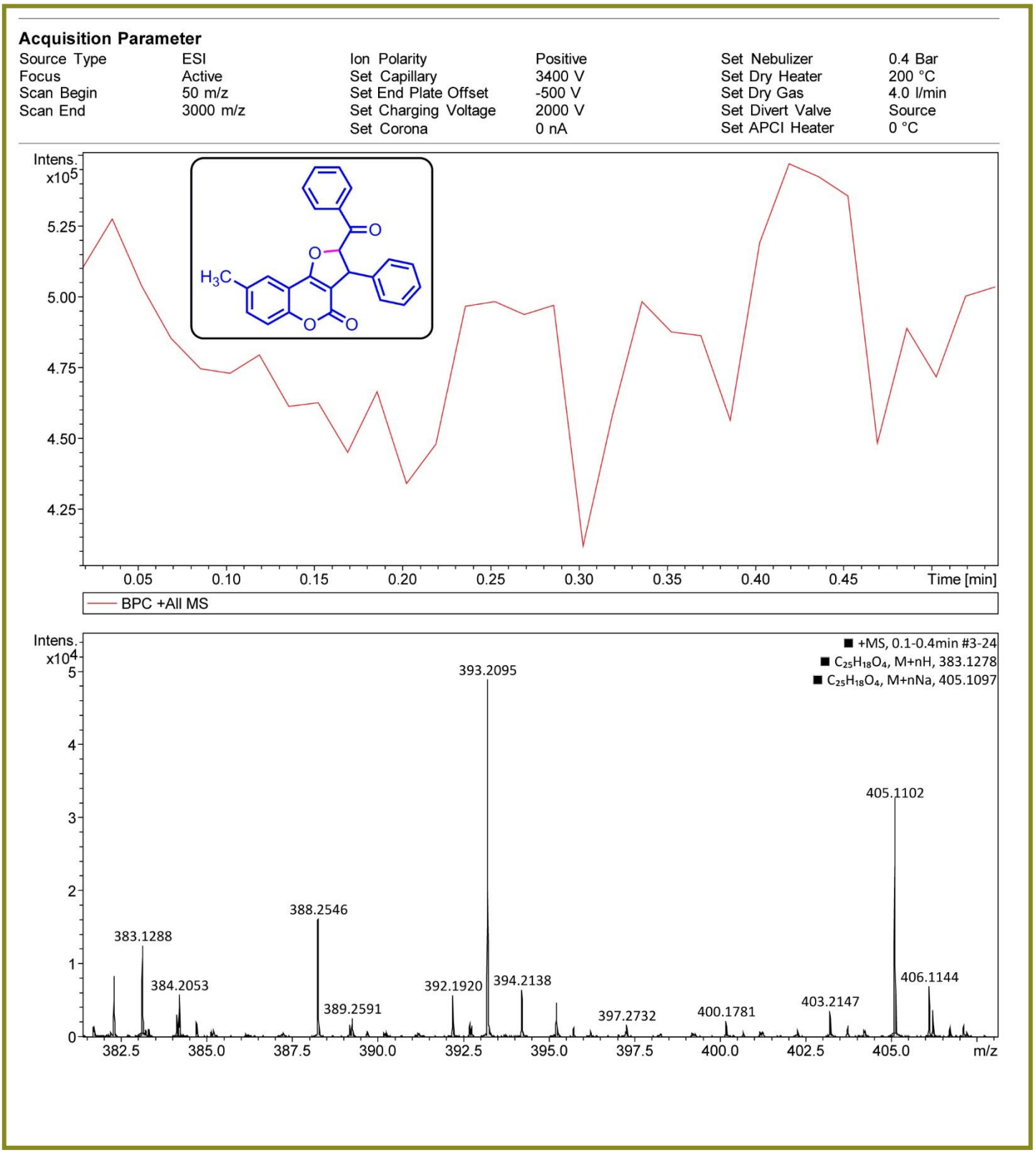


Figure S99. High-resolution Mass spectra of 2-benzoyl-8-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-22**)

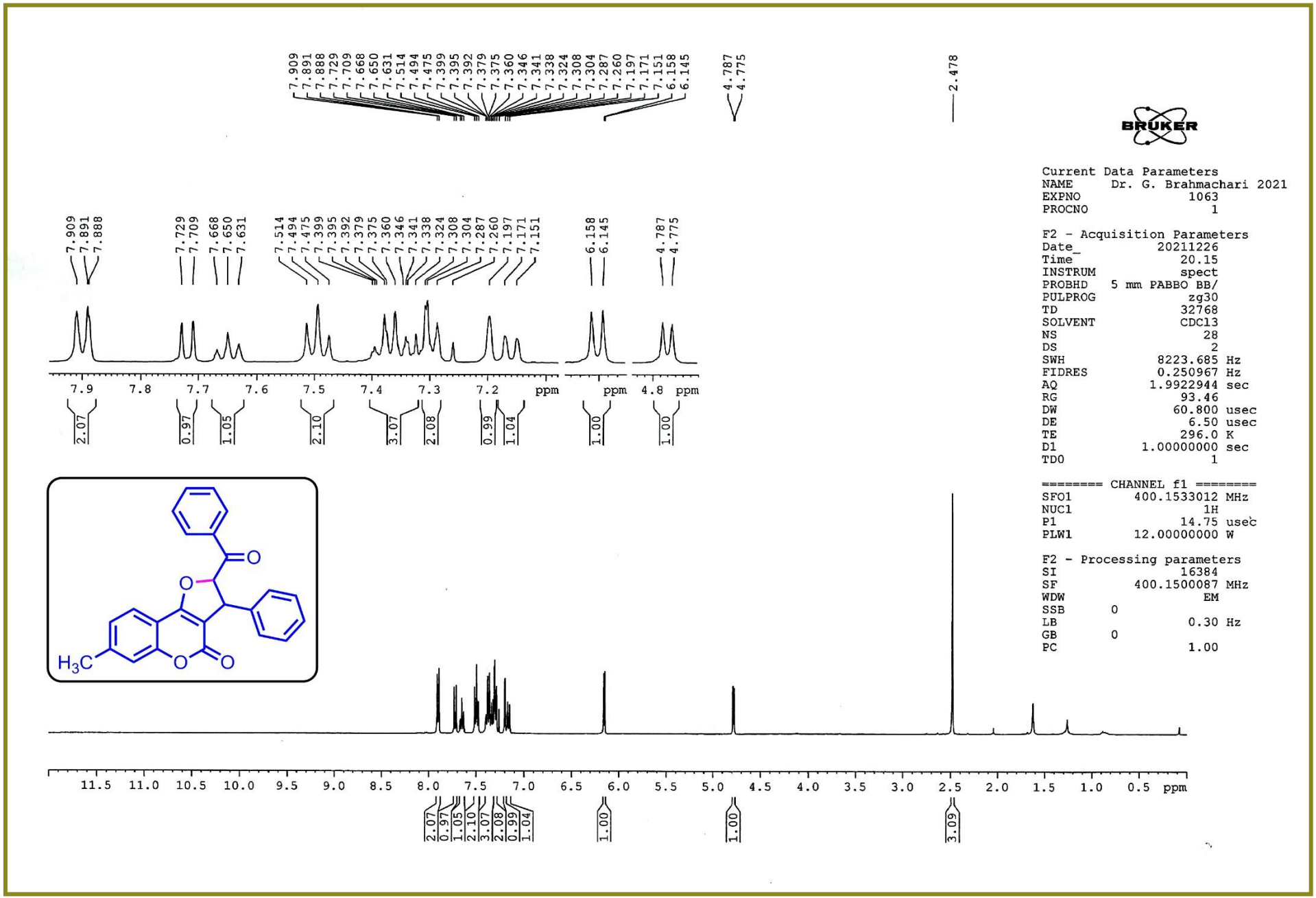


Figure S100. ^{1}H-NMR spectrum of 2-benzoyl-7-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-23**)

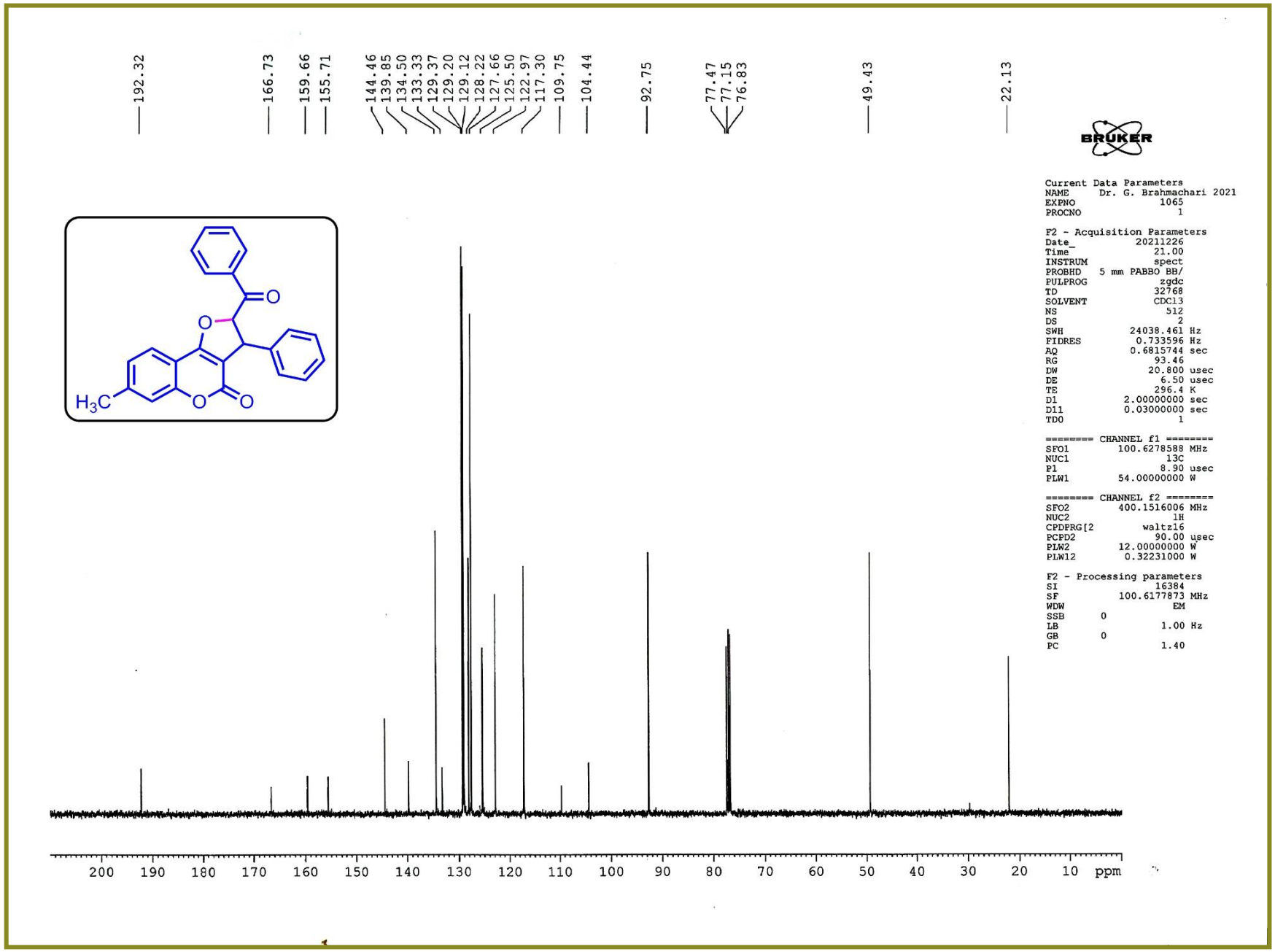


Figure S101. ^{13}C-NMR spectrum of 2-benzoyl-7-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-23**)

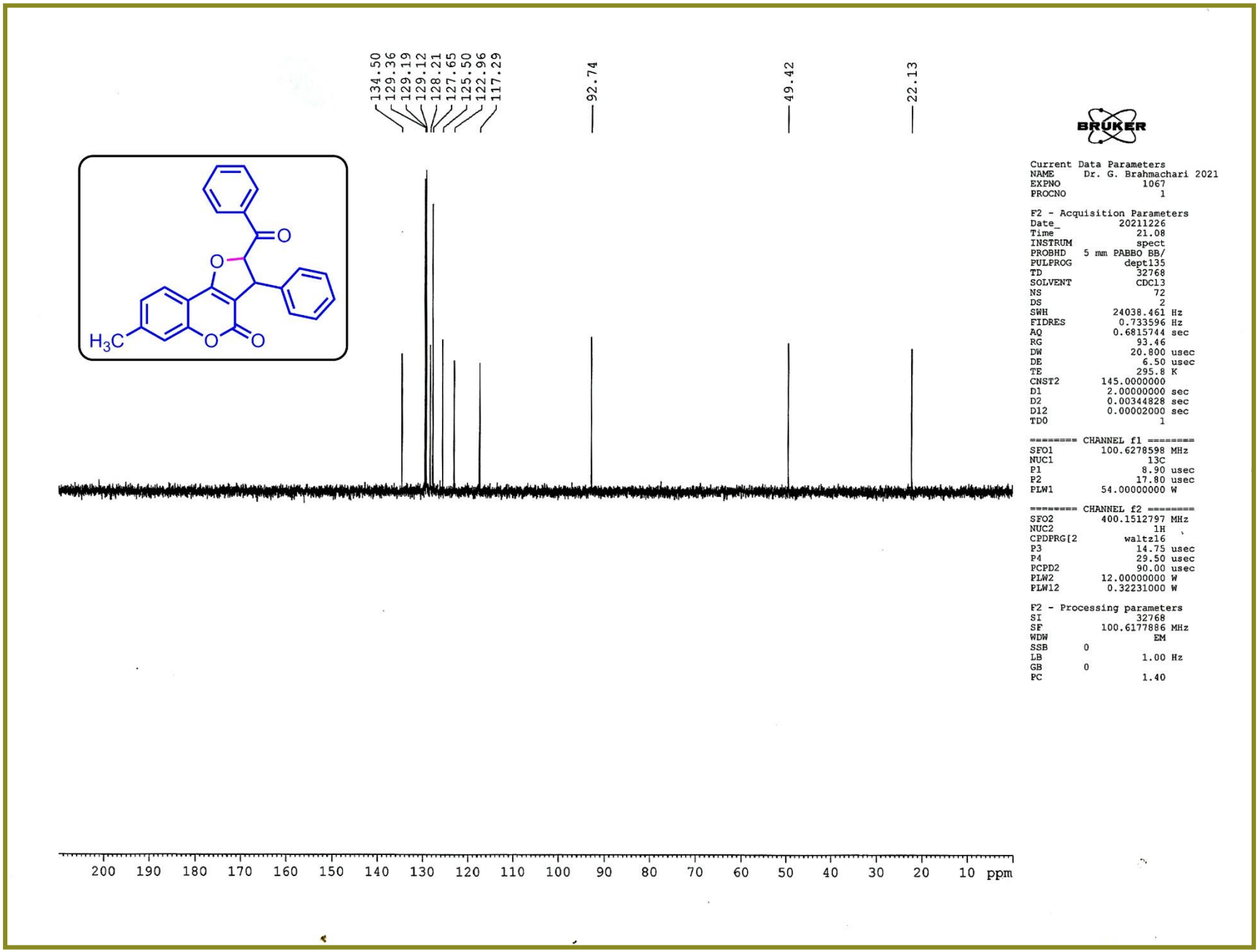


Figure S102. DEPT-135 NMR spectrum of 2-benzoyl-7-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-23**)

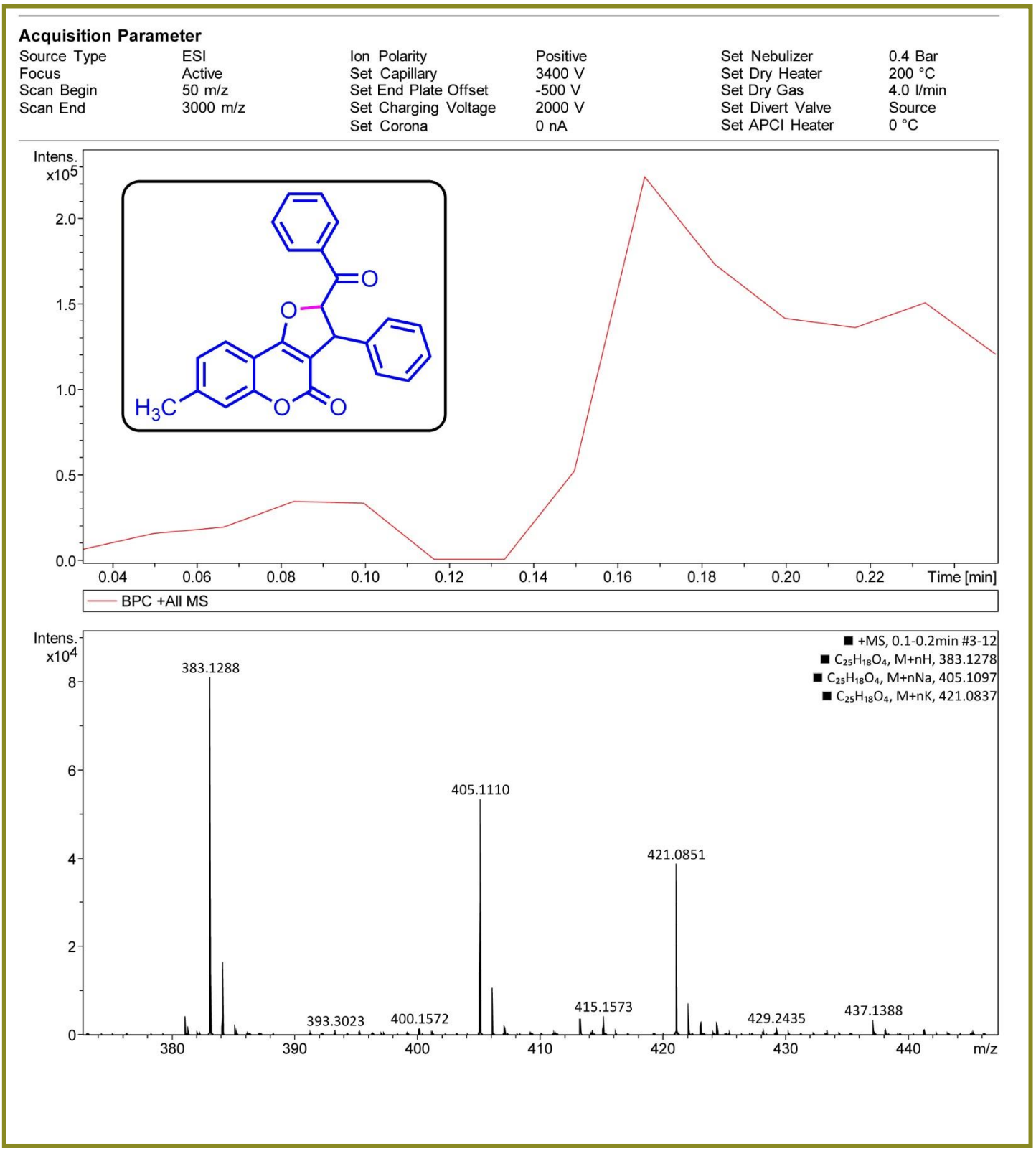


Figure S103. High-resolution Mass spectra of 2-benzoyl-7-methyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-23**)

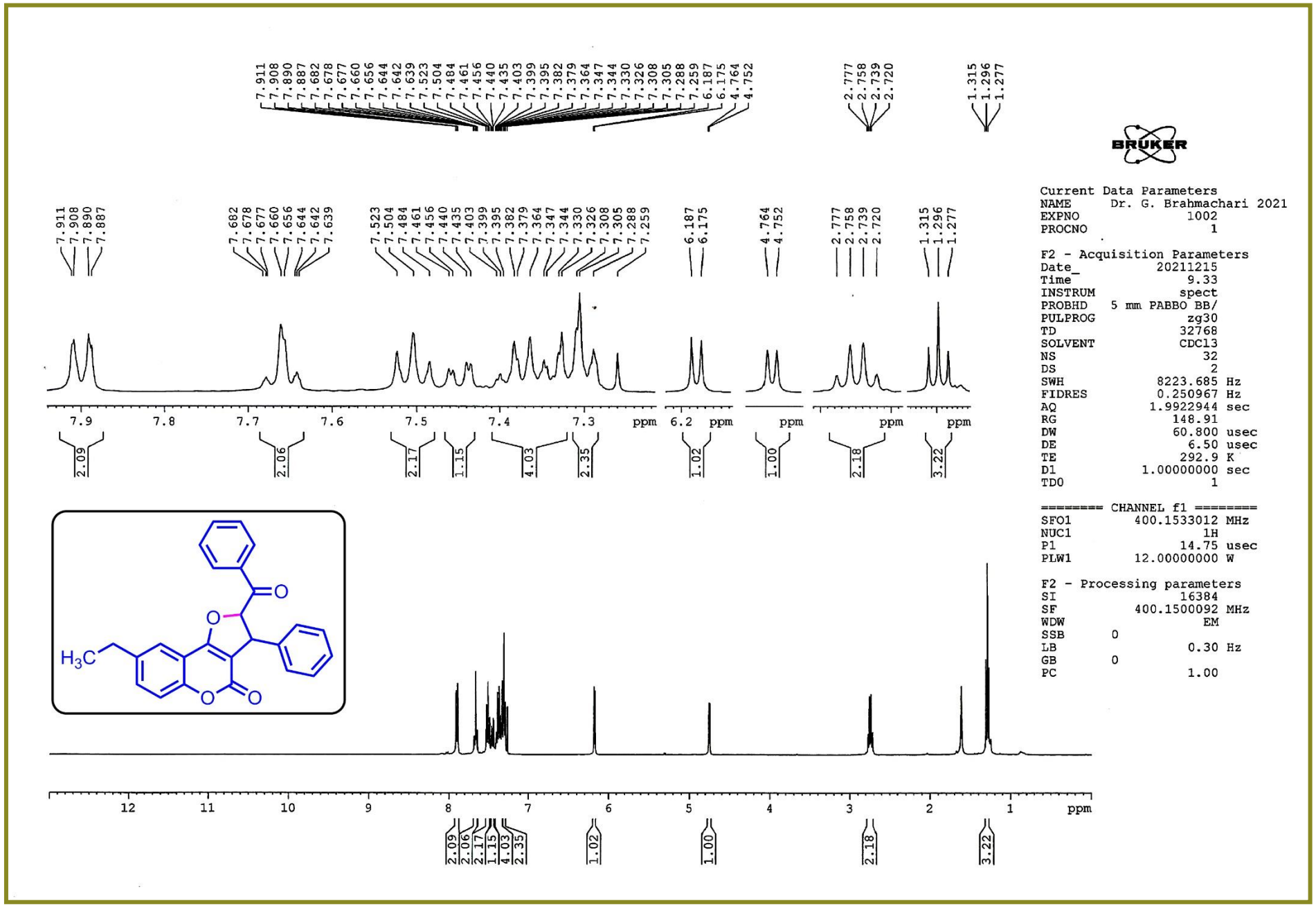


Figure S104. ^{1}H-NMR spectrum of 2-benzoyl-8-ethyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-24**)

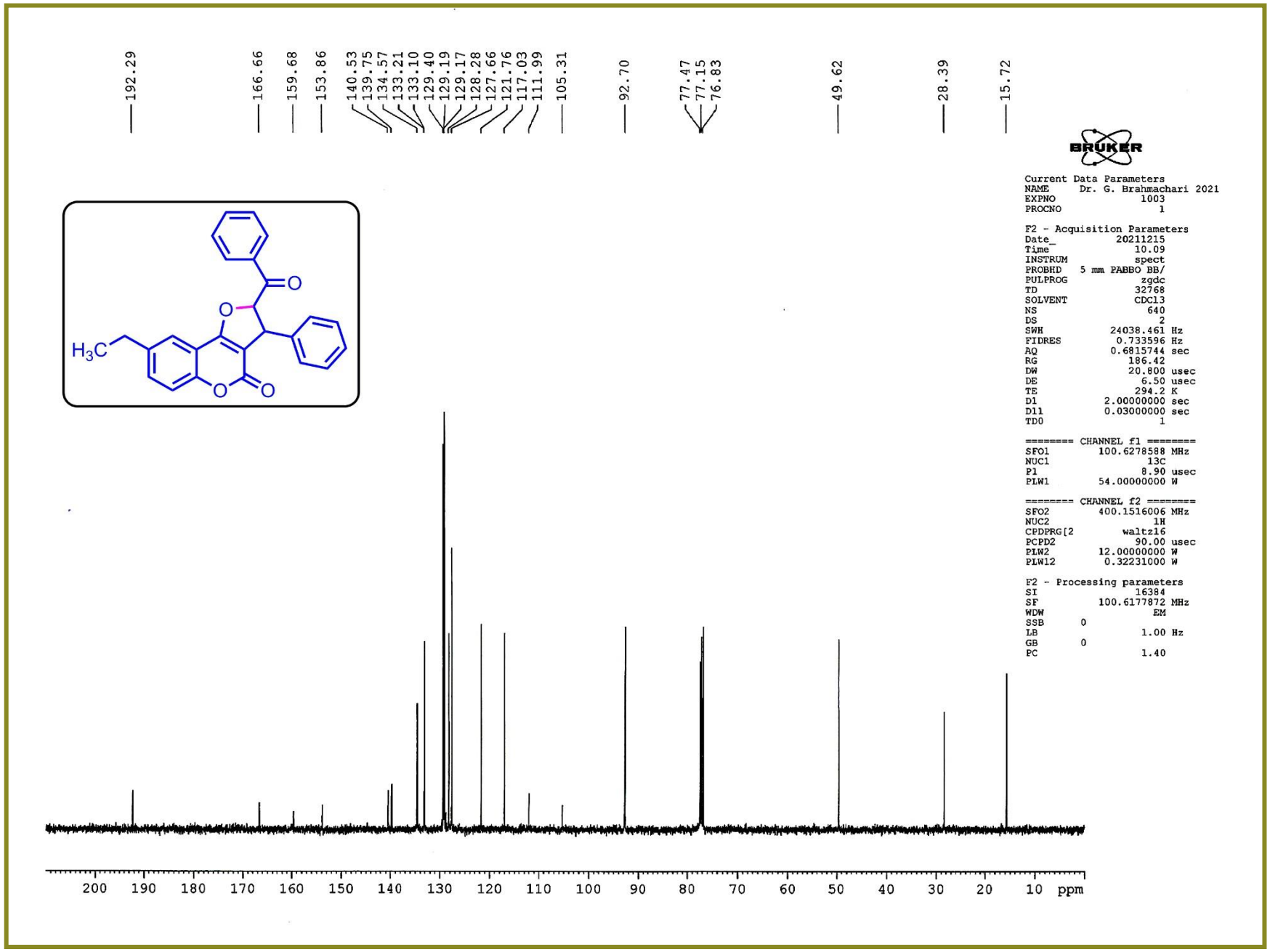


Figure S105. ^{13}C-NMR spectrum of 2-benzoyl-8-ethyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-24**)

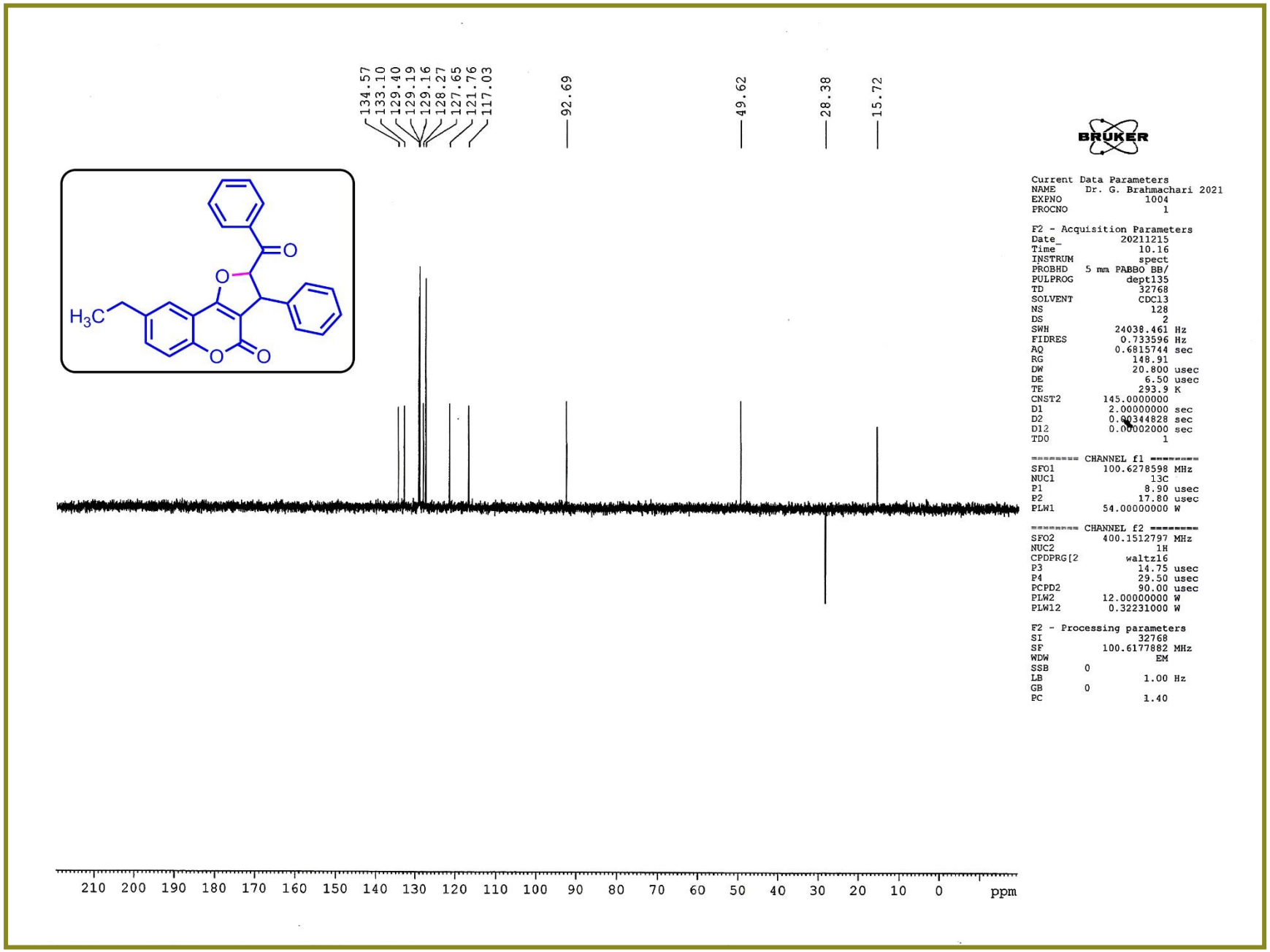


Figure S106. DEPT-135 NMR spectrum of 2-benzoyl-8-ethyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-24**)

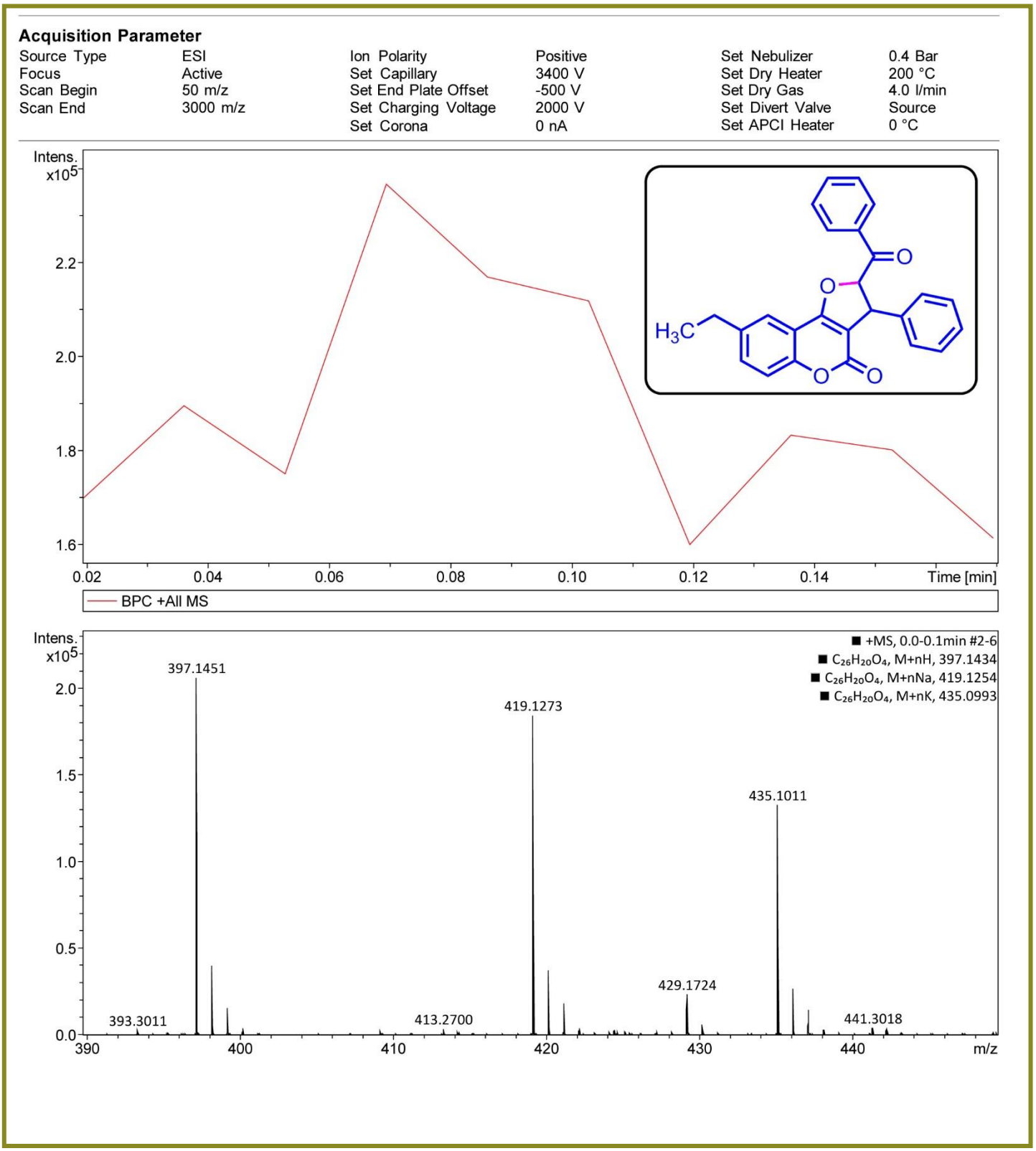


Figure S107. High-resolution Mass spectra of 2-benzoyl-8-ethyl-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-24**)

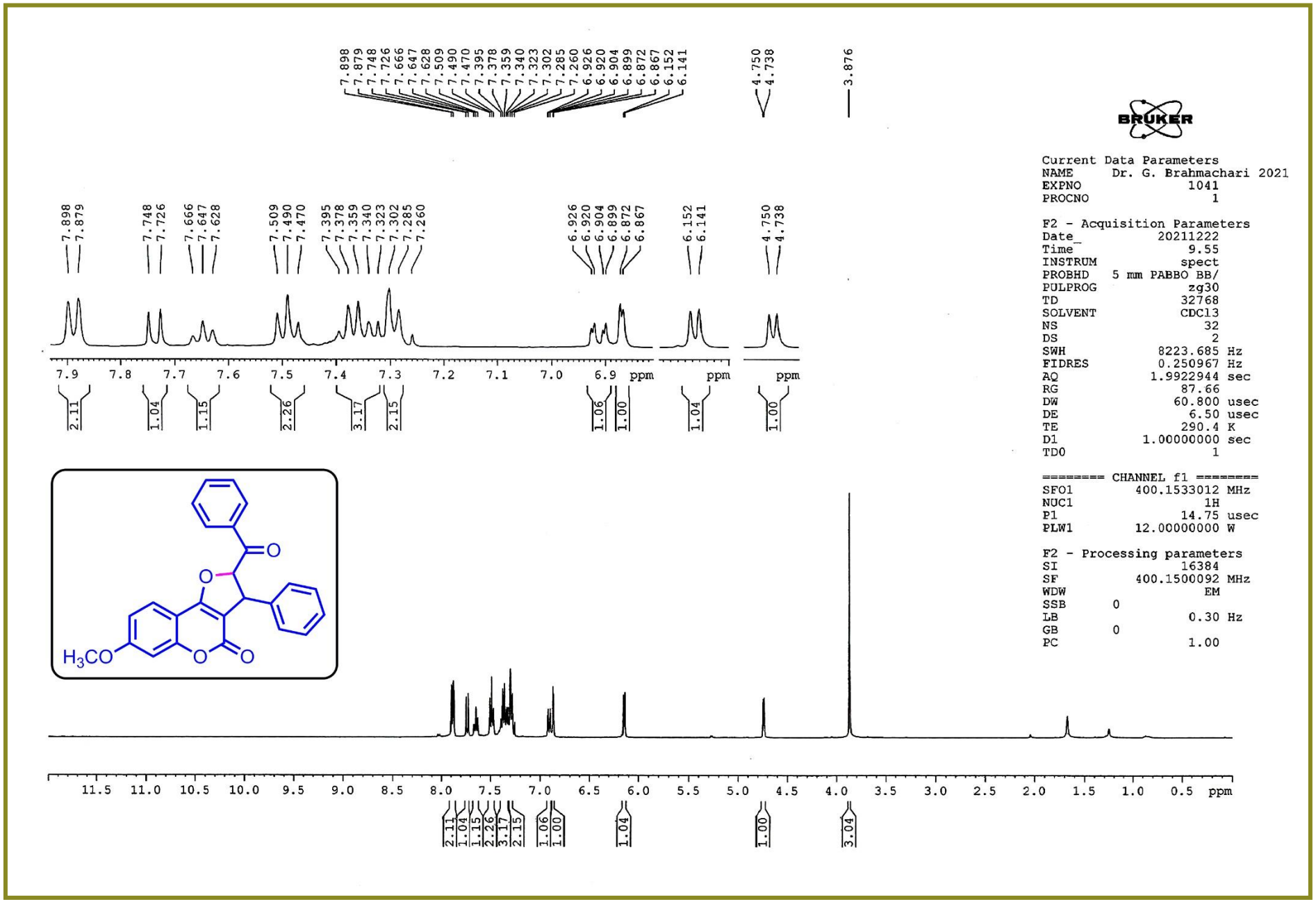


Figure S108. ^{1}H-NMR spectrum of 2-benzoyl-7-methoxy-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-25**)

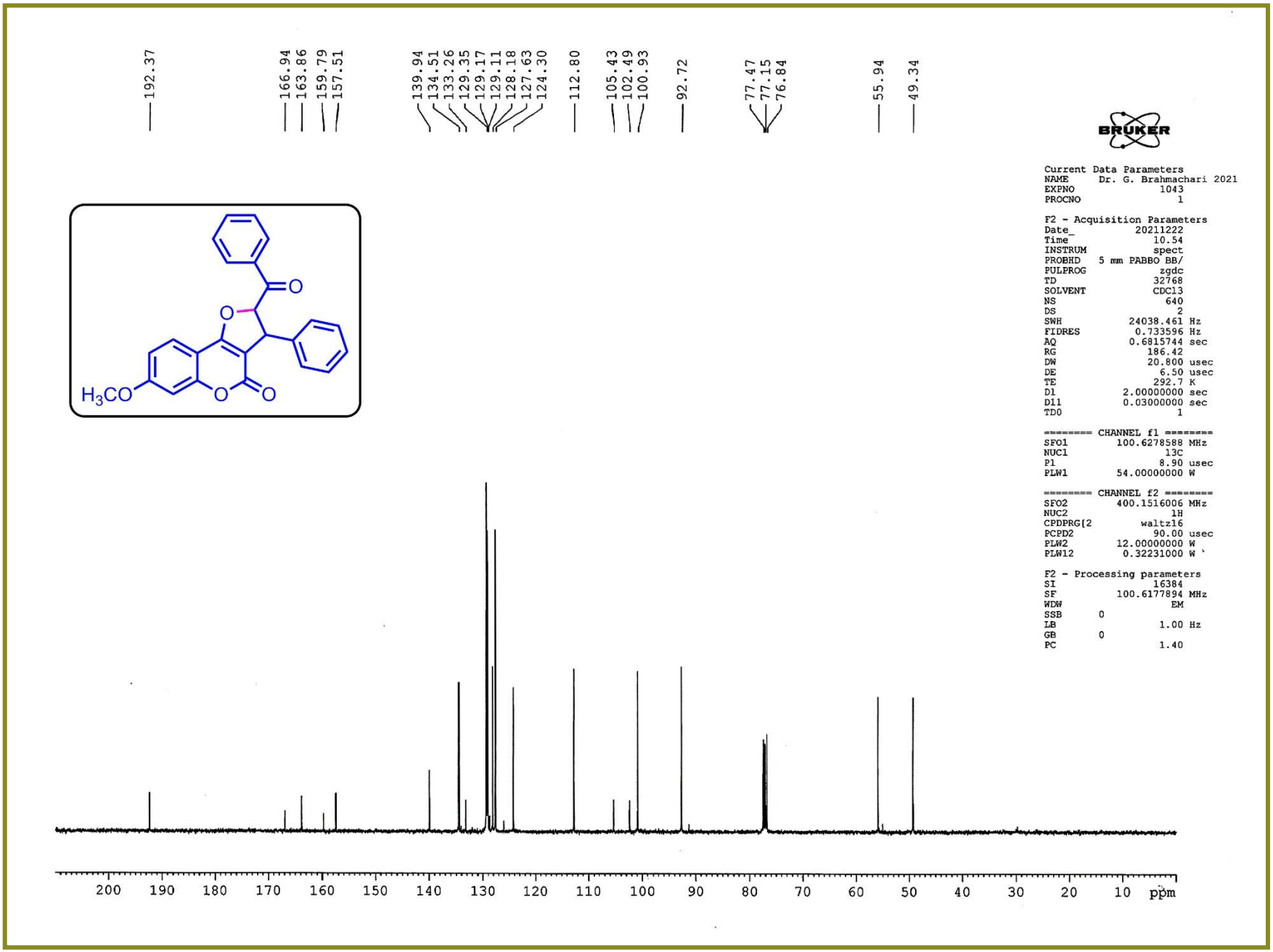


Figure S109. ^{13}C-NMR spectrum of 2-benzoyl-7-methoxy-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-25**)

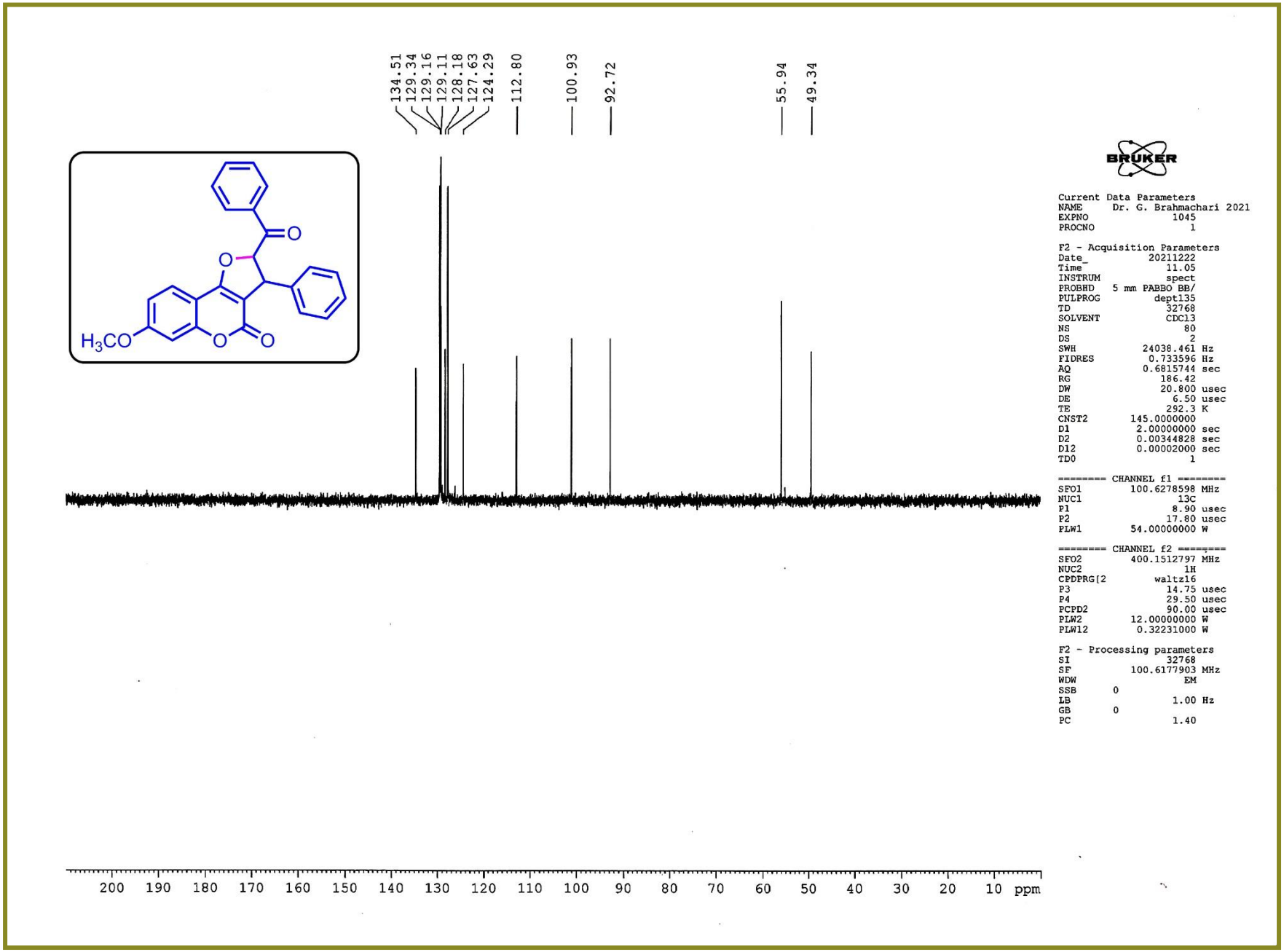


Figure S110. DEPT-135 NMR spectrum of 2-benzoyl-7-methoxy-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-25**)

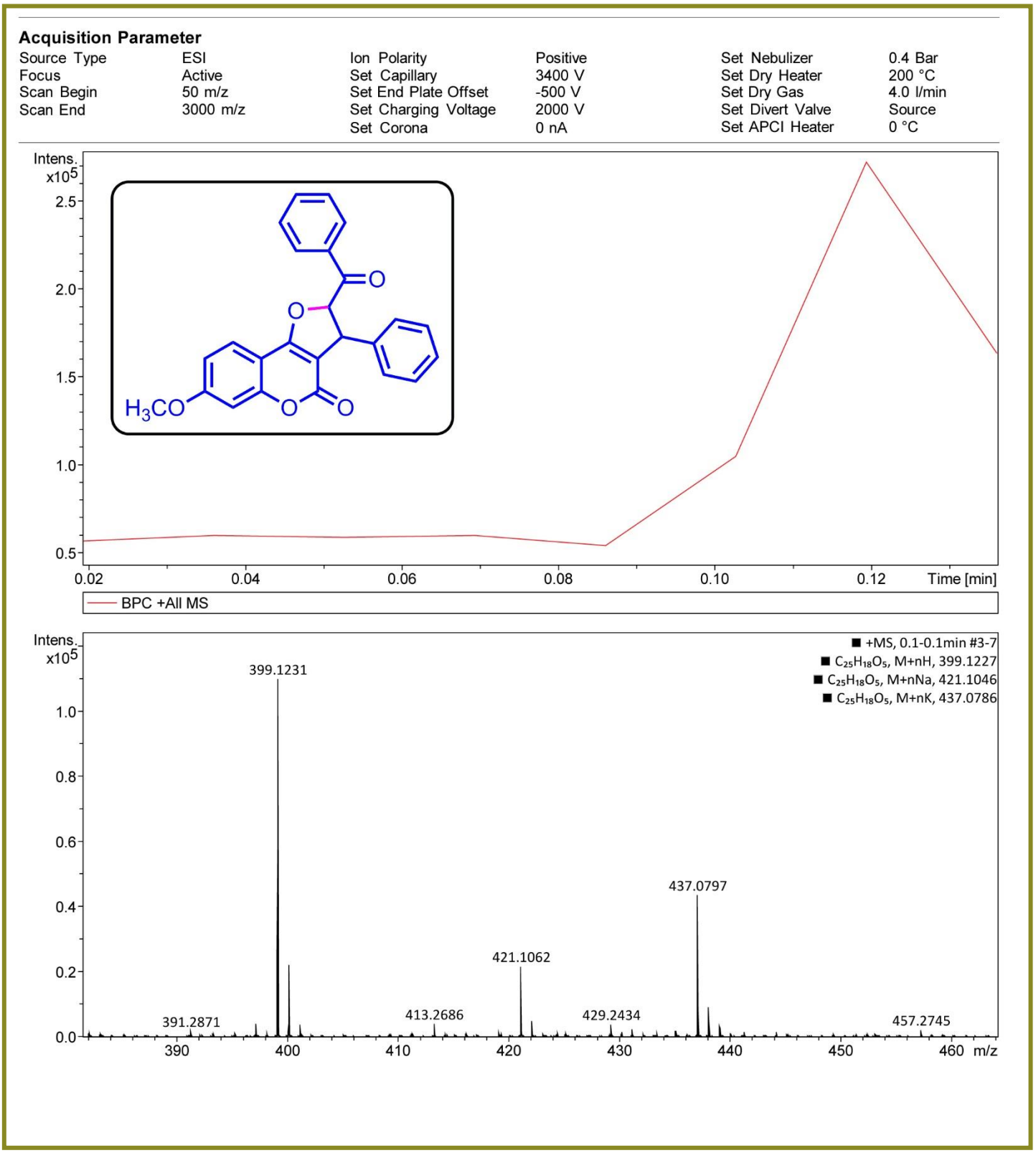


Figure S111. High-resolution Mass spectra of 2-benzoyl-7-methoxy-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-25**)

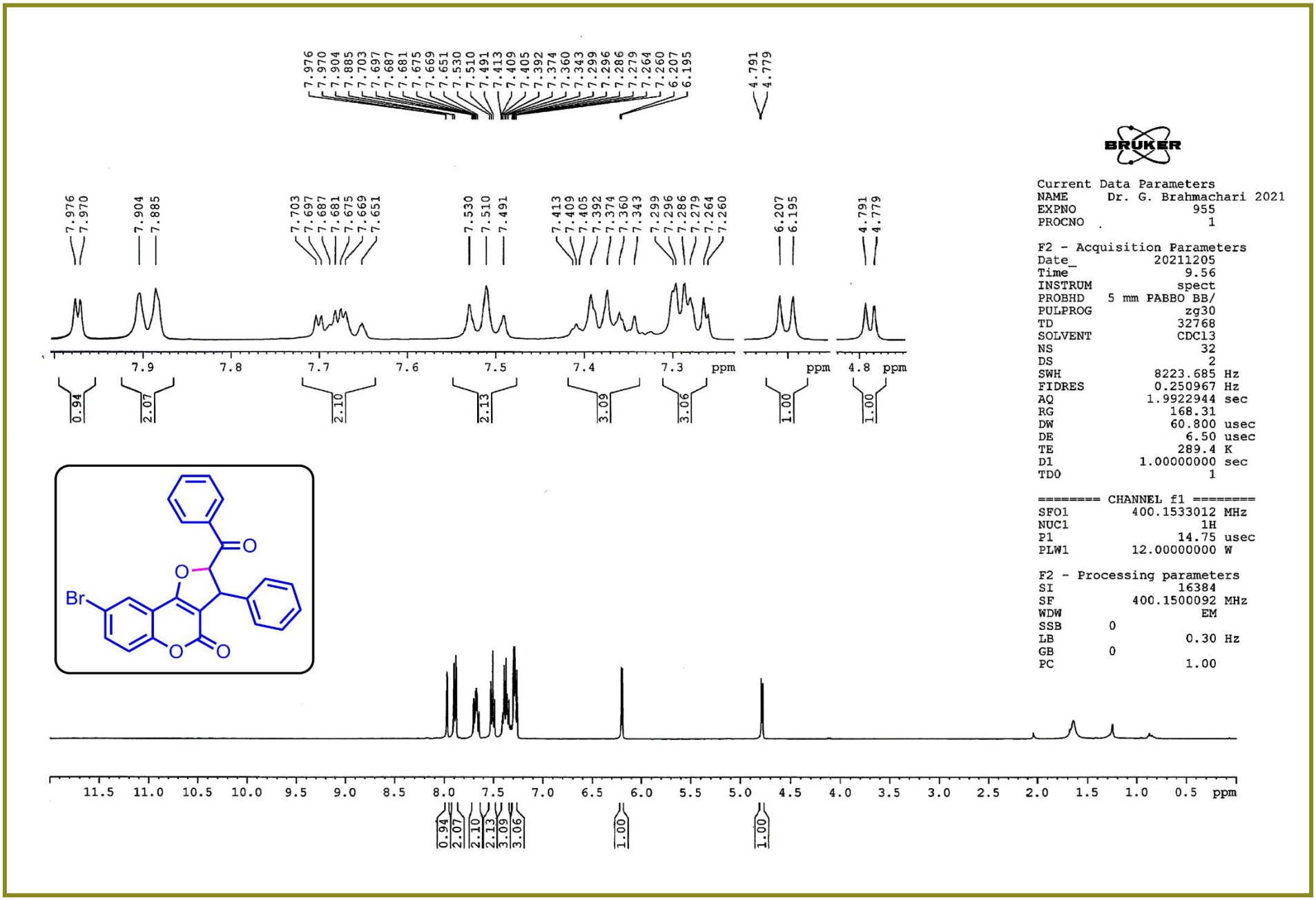


Figure S112. ^{1}H-NMR spectrum of 2-benzoyl-8-bromo-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-26**)

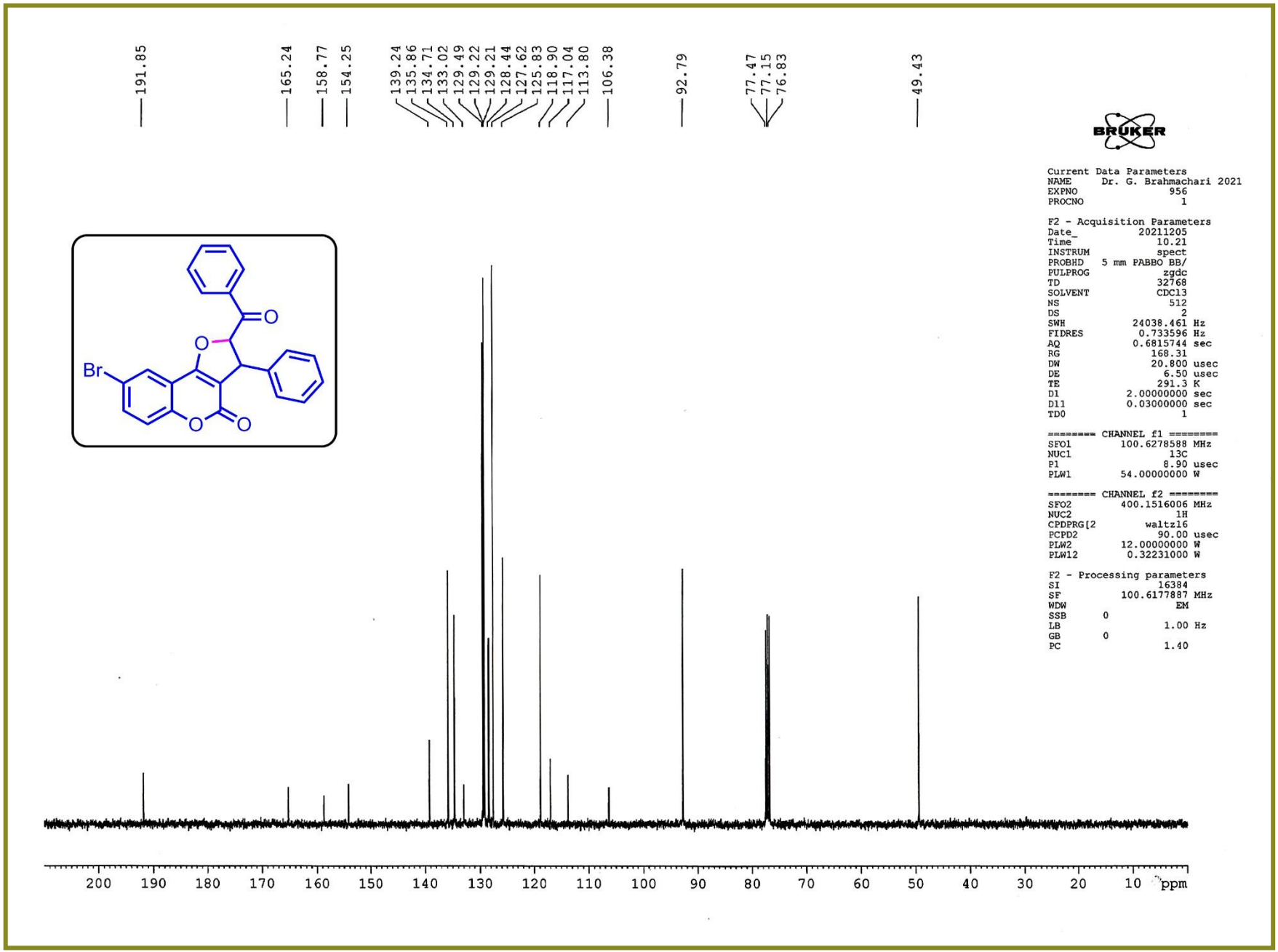


Figure S113. ^{13}C-NMR spectrum of 2-benzoyl-8-bromo-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-26**)

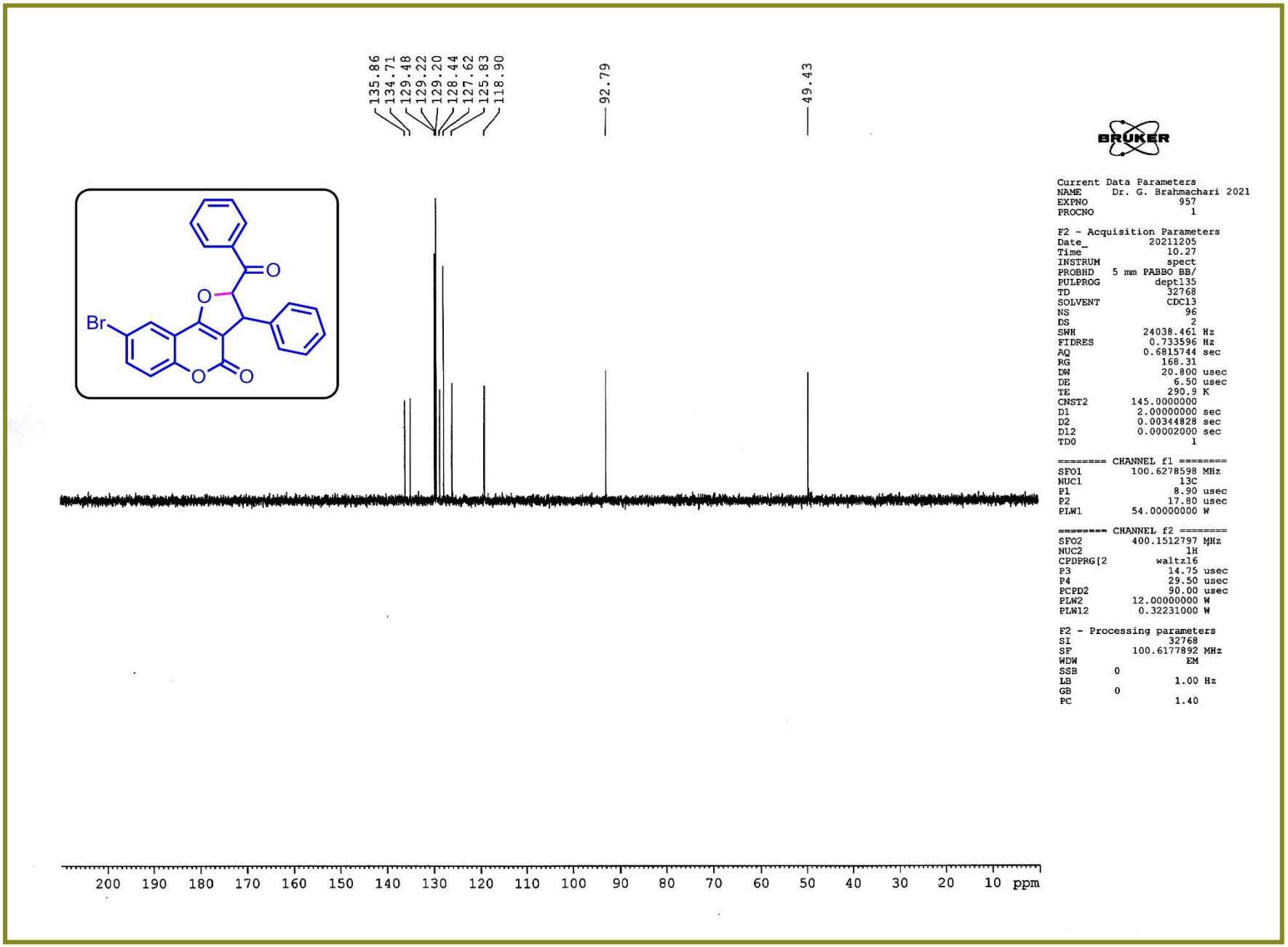


Figure S114. DEPT-135 NMR spectrum of 2-benzoyl-8-bromo-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-26**)

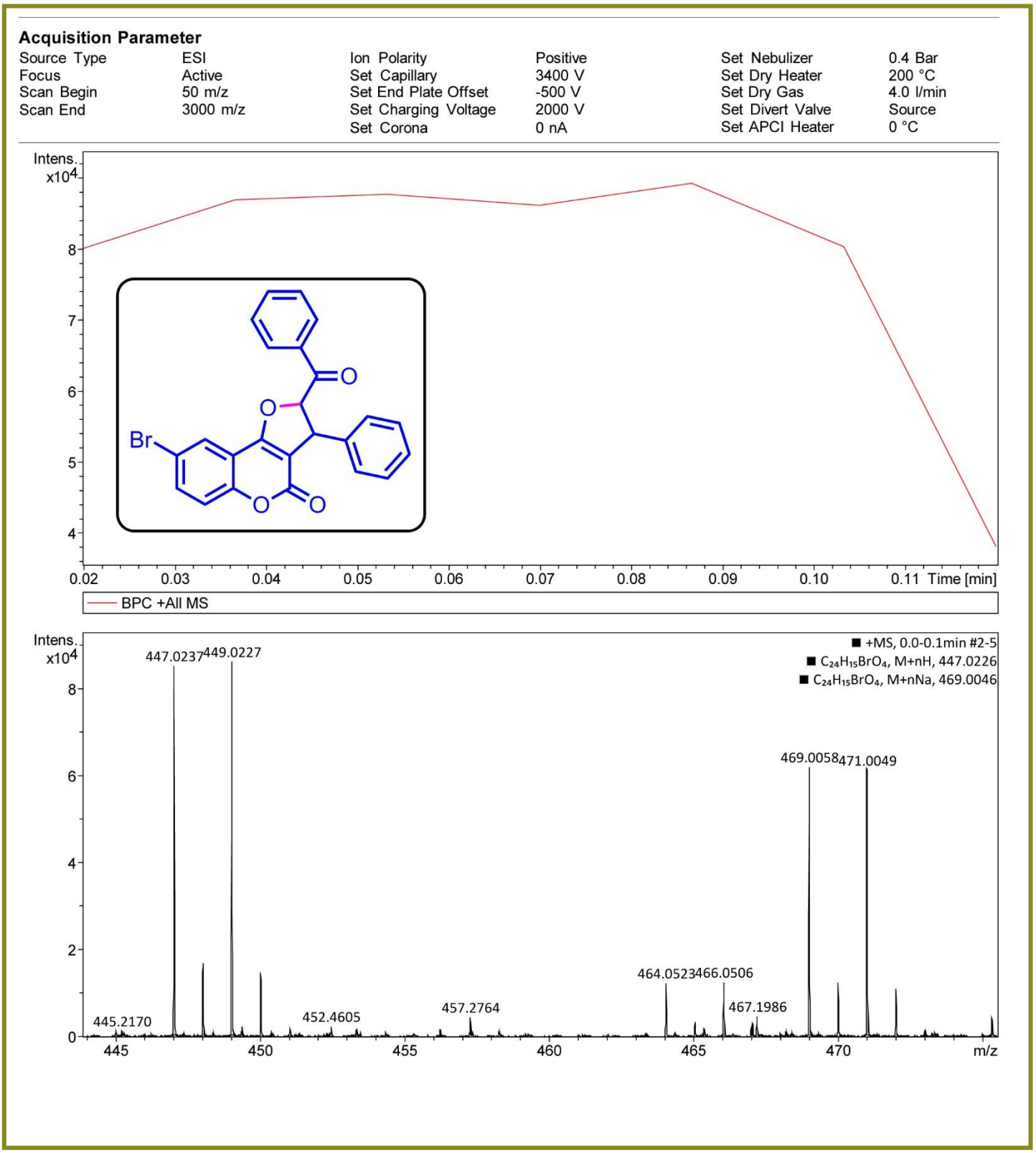


Figure S115. High-resolution Mass spectra of 2-benzoyl-8-bromo-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-26**)

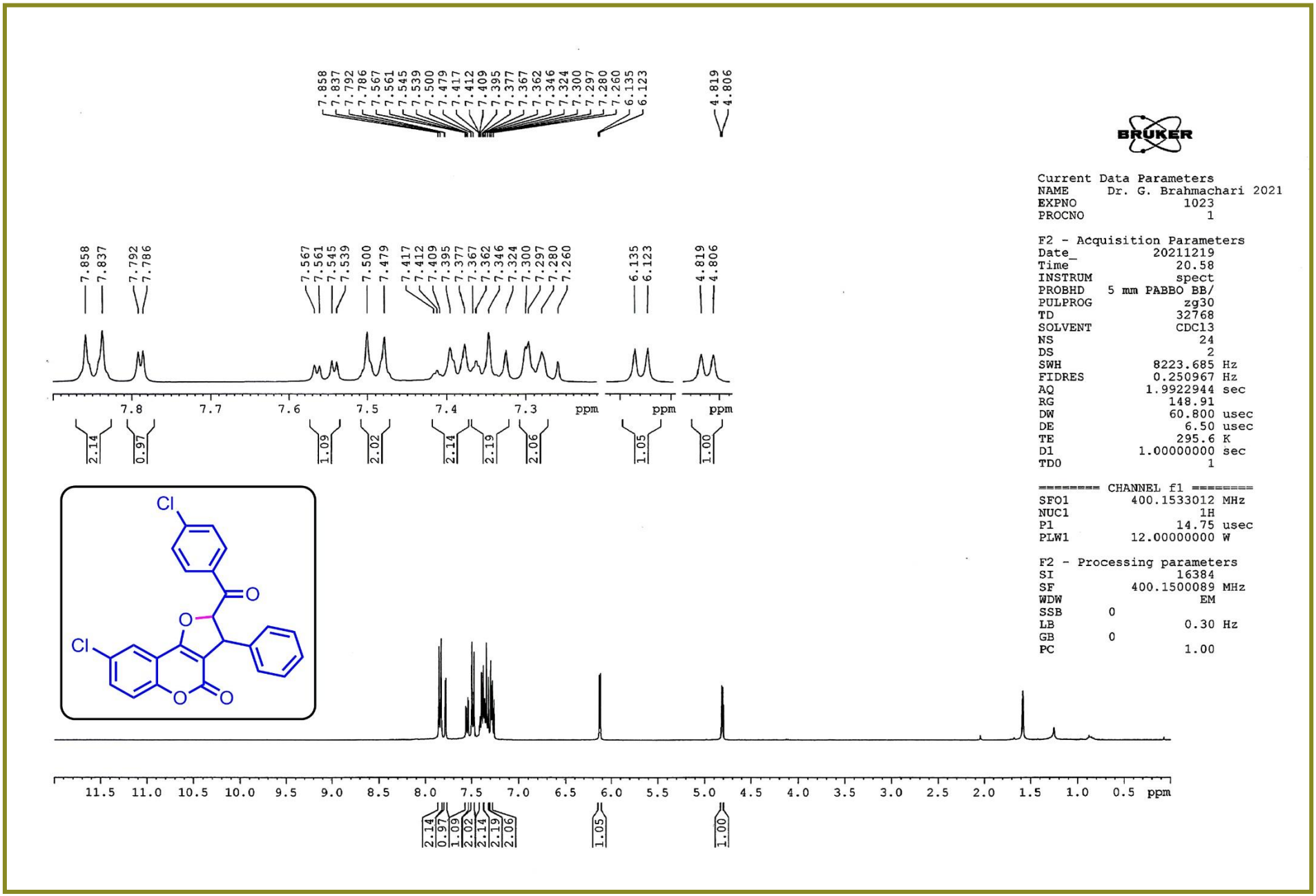


Figure S116. ^{1}H-NMR spectrum of 8-chloro-2-(4-chlorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-27**)

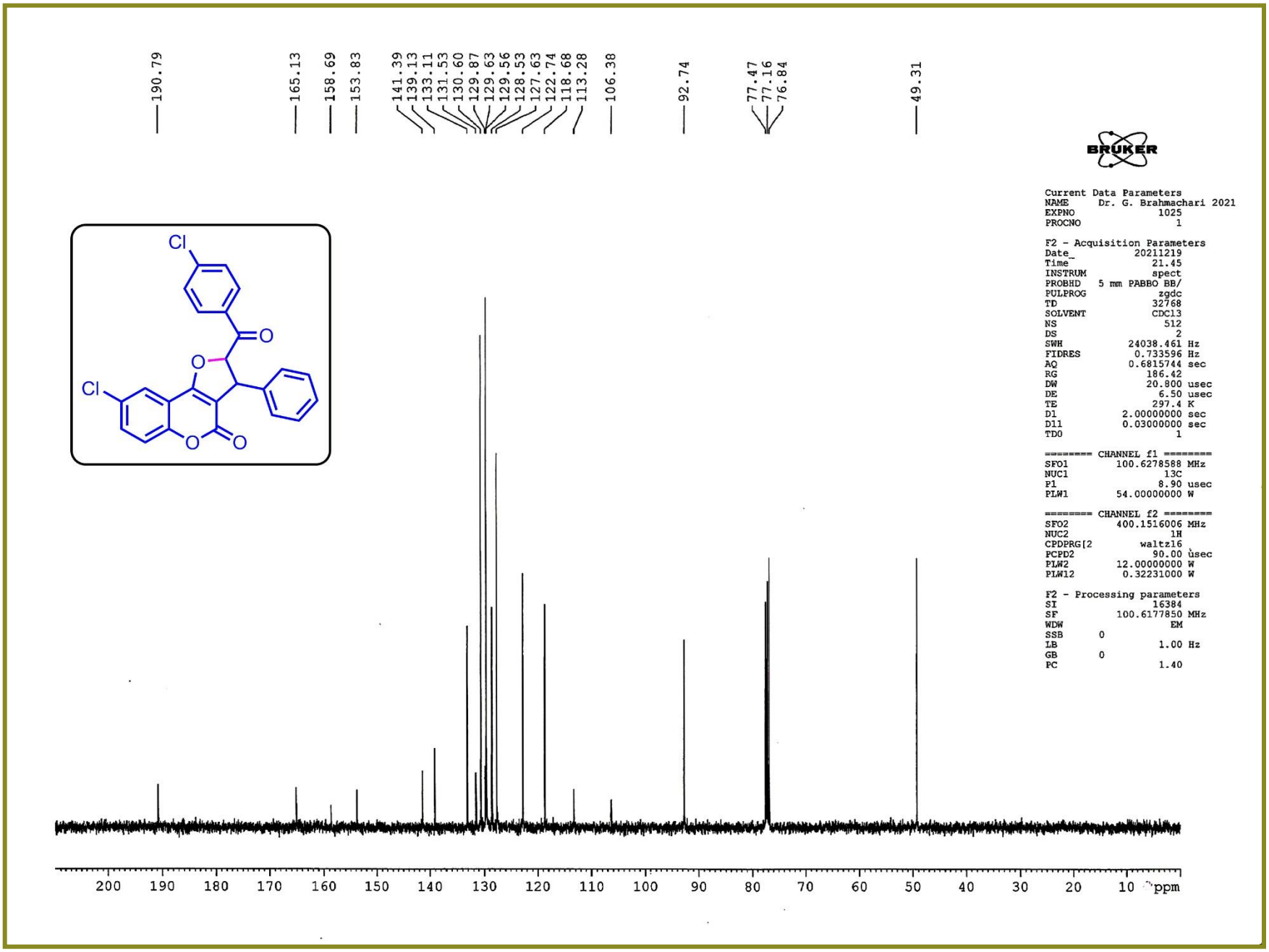


Figure S117. ^{13}C-NMR spectrum of 8-chloro-2-(4-chlorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-27**)

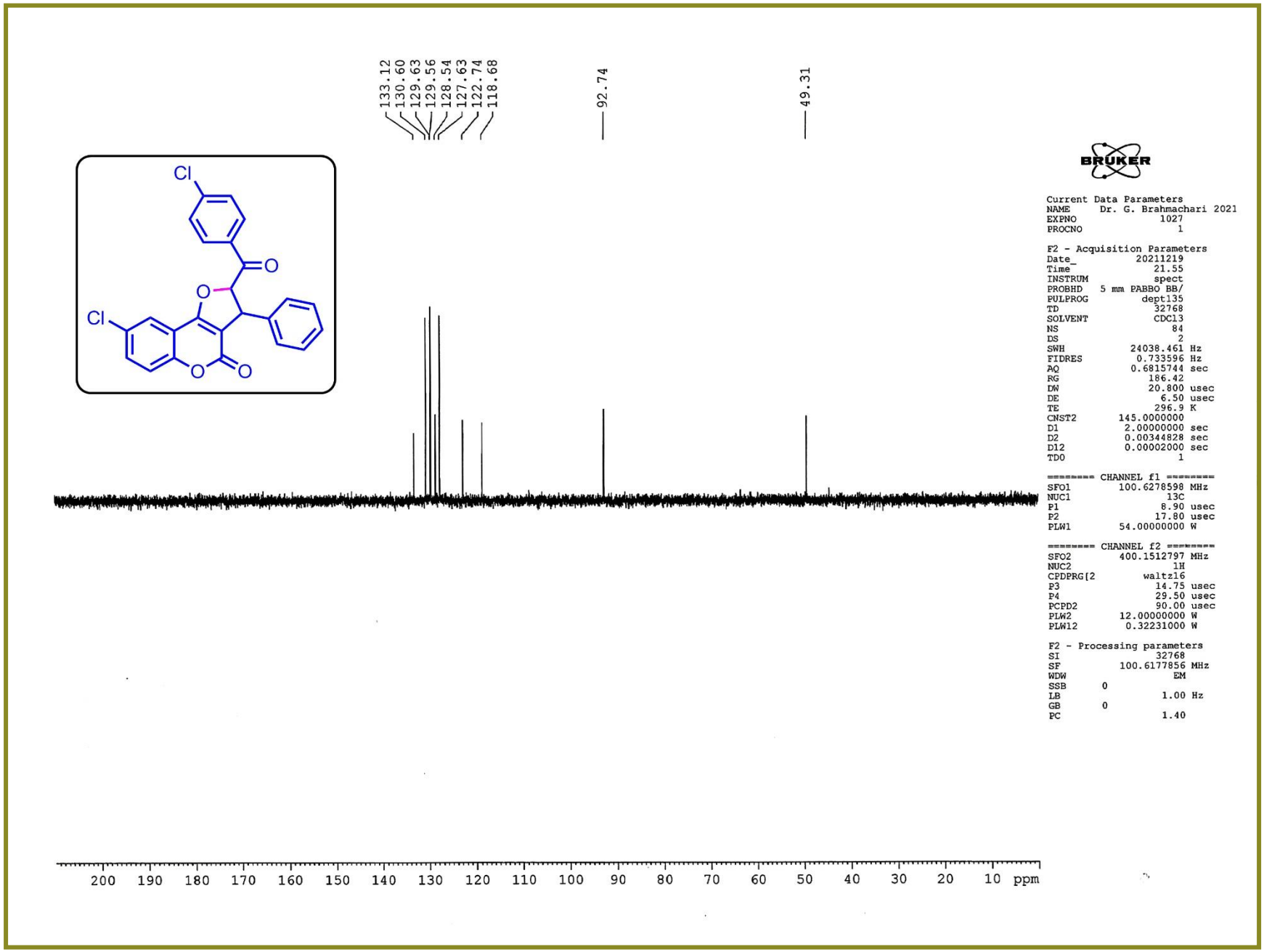


Figure S118. DEPT-135 NMR spectrum of 8-chloro-2-(4-chlorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-27**)

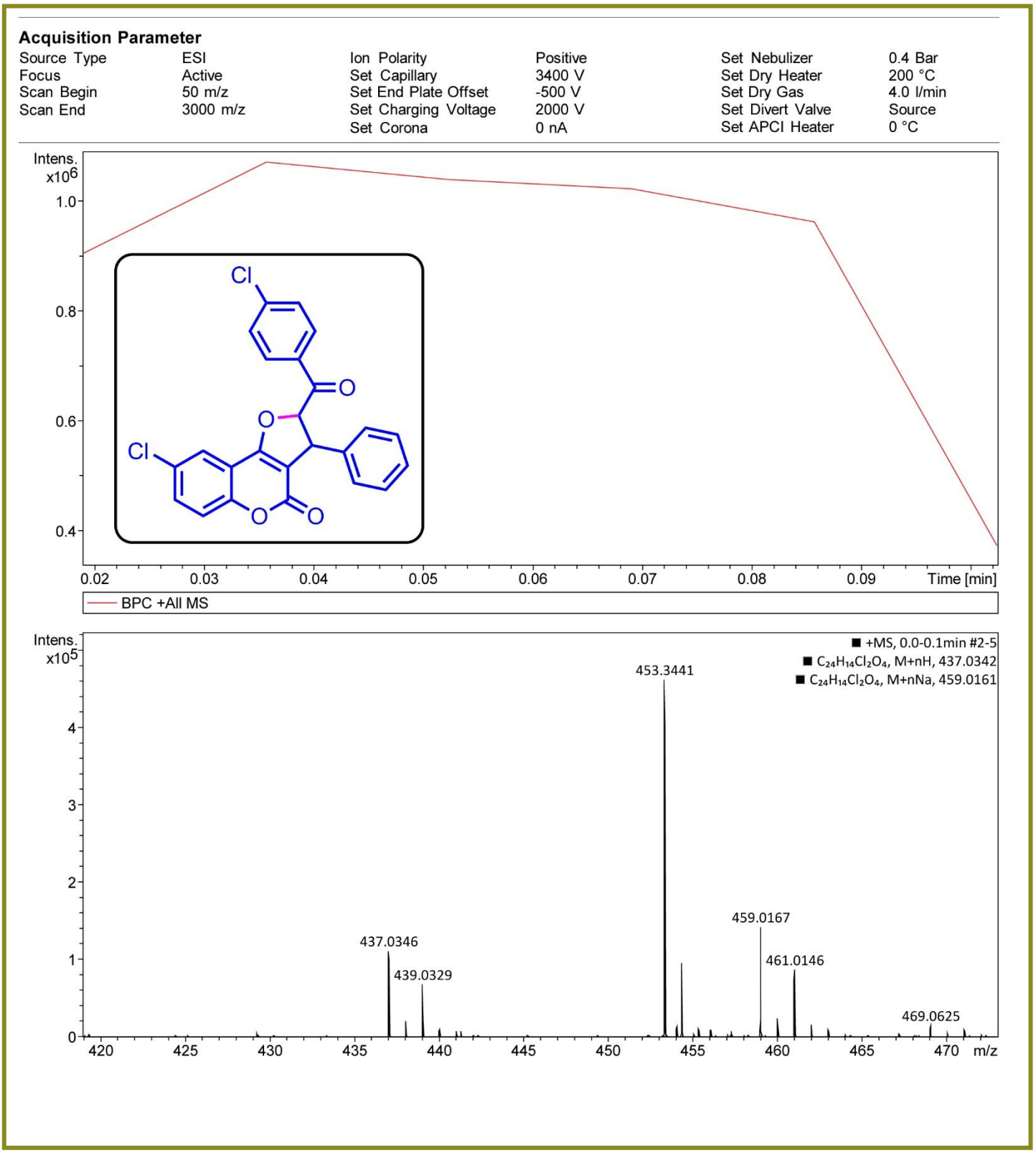


Figure S119. High-resolution Mass spectra of 8-chloro-2-(4-chlorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*] chromen-4-one (**2-27**)

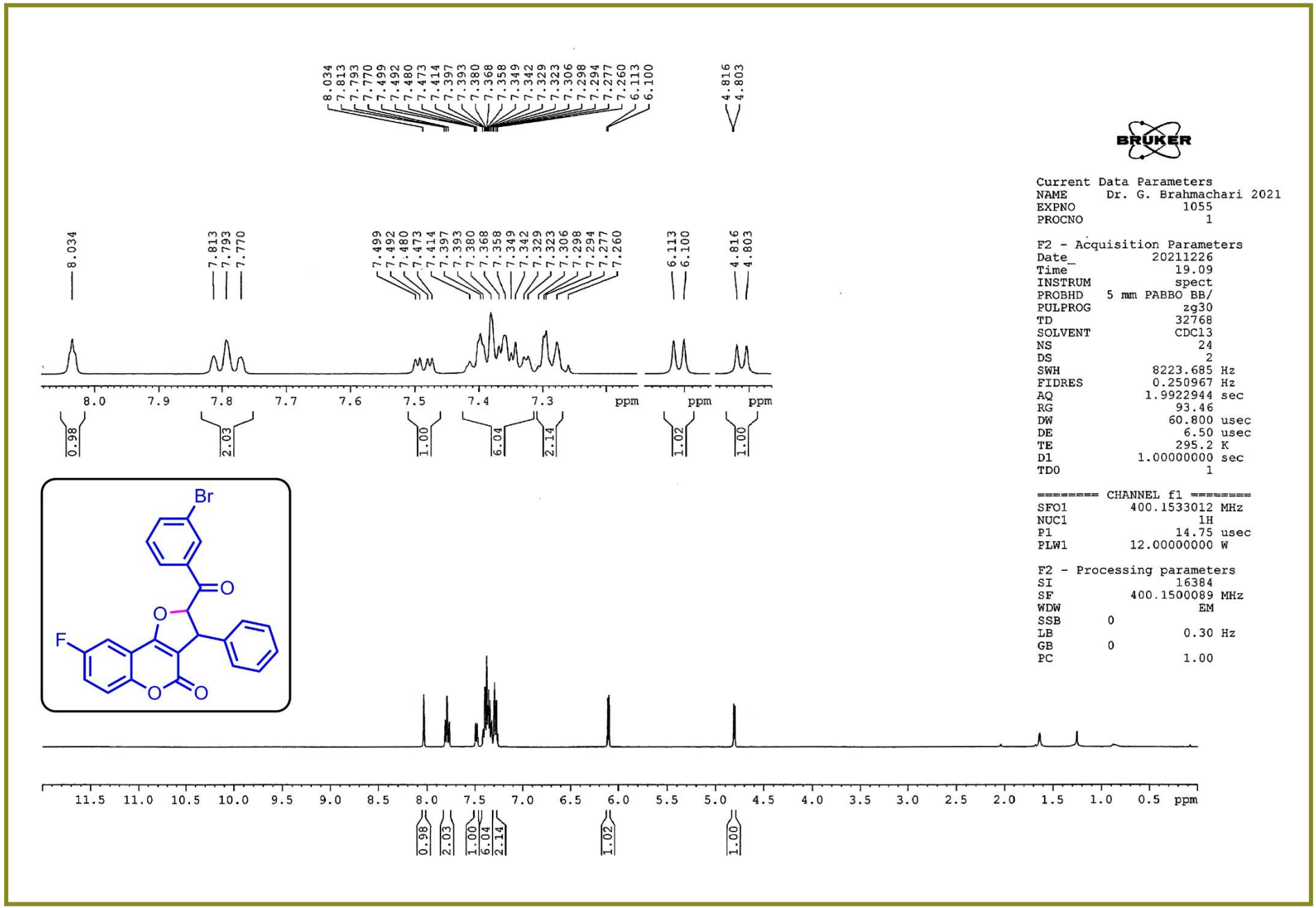


Figure S120. ^{1}H-NMR spectrum of 2-(3-bromobenzoyl)-8-fluoro-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-28**)

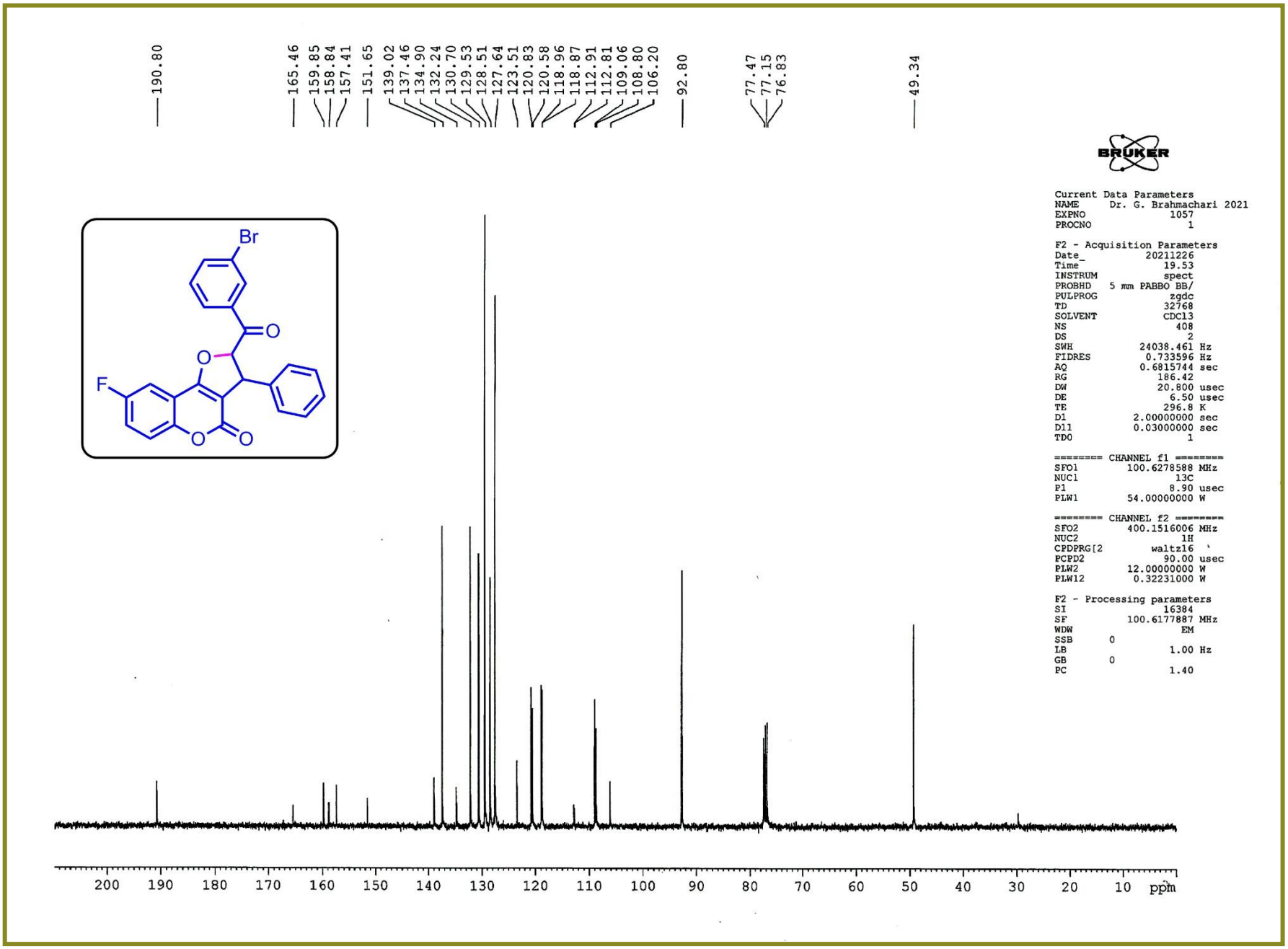


Figure S121. ^{13}C-NMR spectrum of 2-(3-bromobenzoyl)-8-fluoro-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-28**)

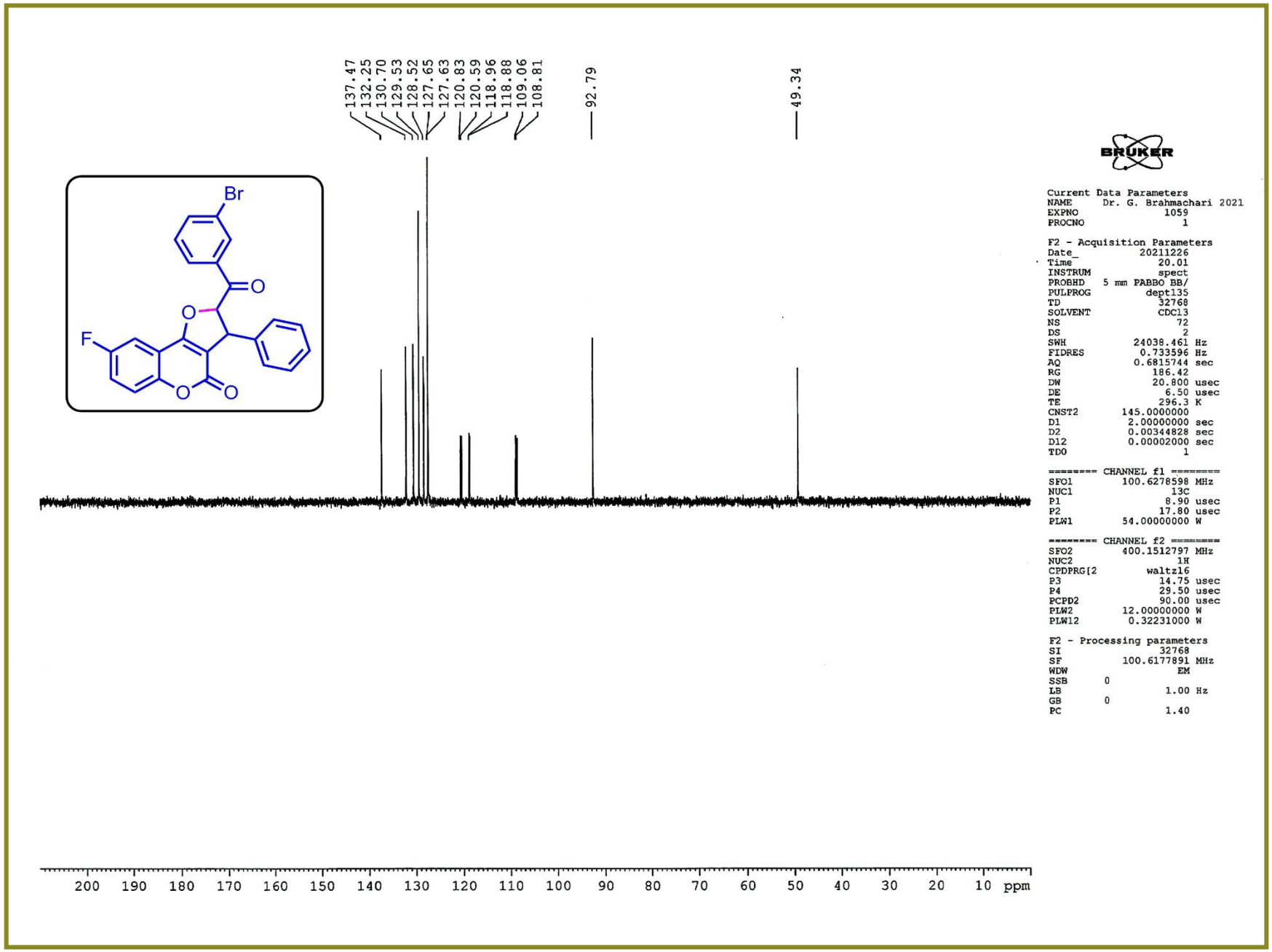


Figure S122. DEPT-135 NMR spectrum of 2-(3-bromobenzoyl)-8-fluoro-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-28**)

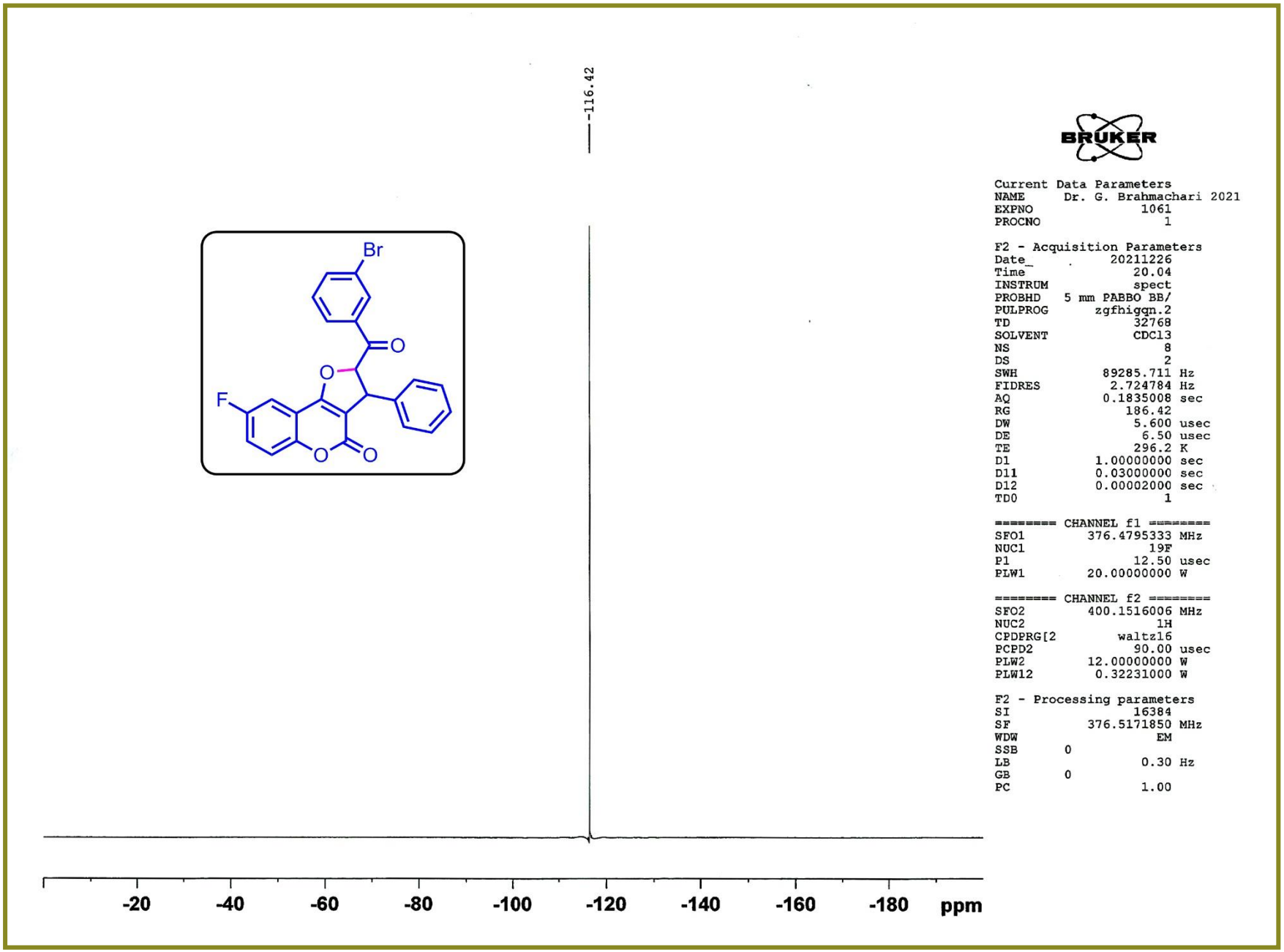


Figure S123. ^{19}F NMR spectrum of 2-(3-bromobenzoyl)-8-fluoro-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-28**)

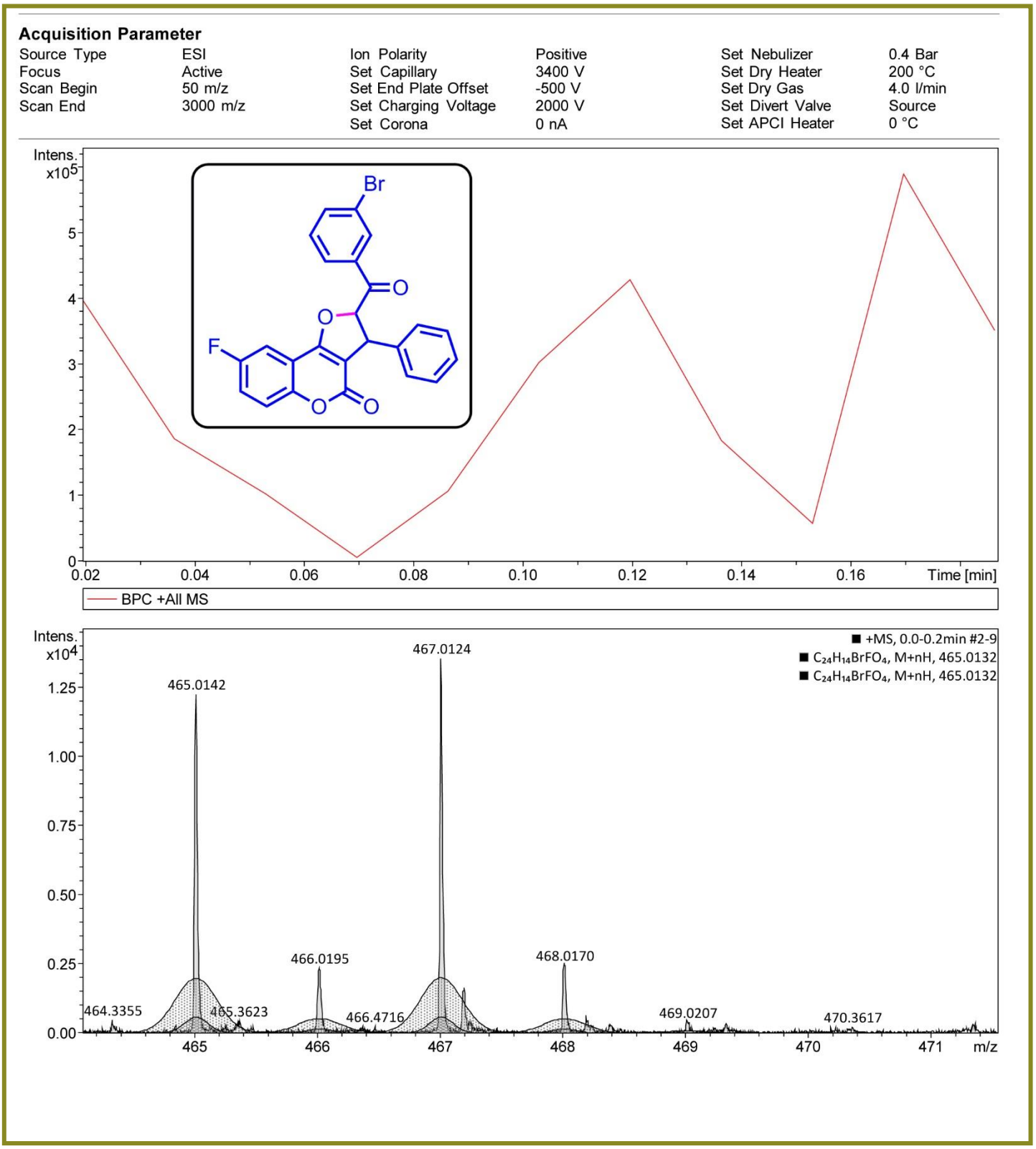


Figure S124. High-resolution Mass spectra of 2-(3-bromobenzoyl)-8-fluoro-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*] chromen-4-one (**2-28**)

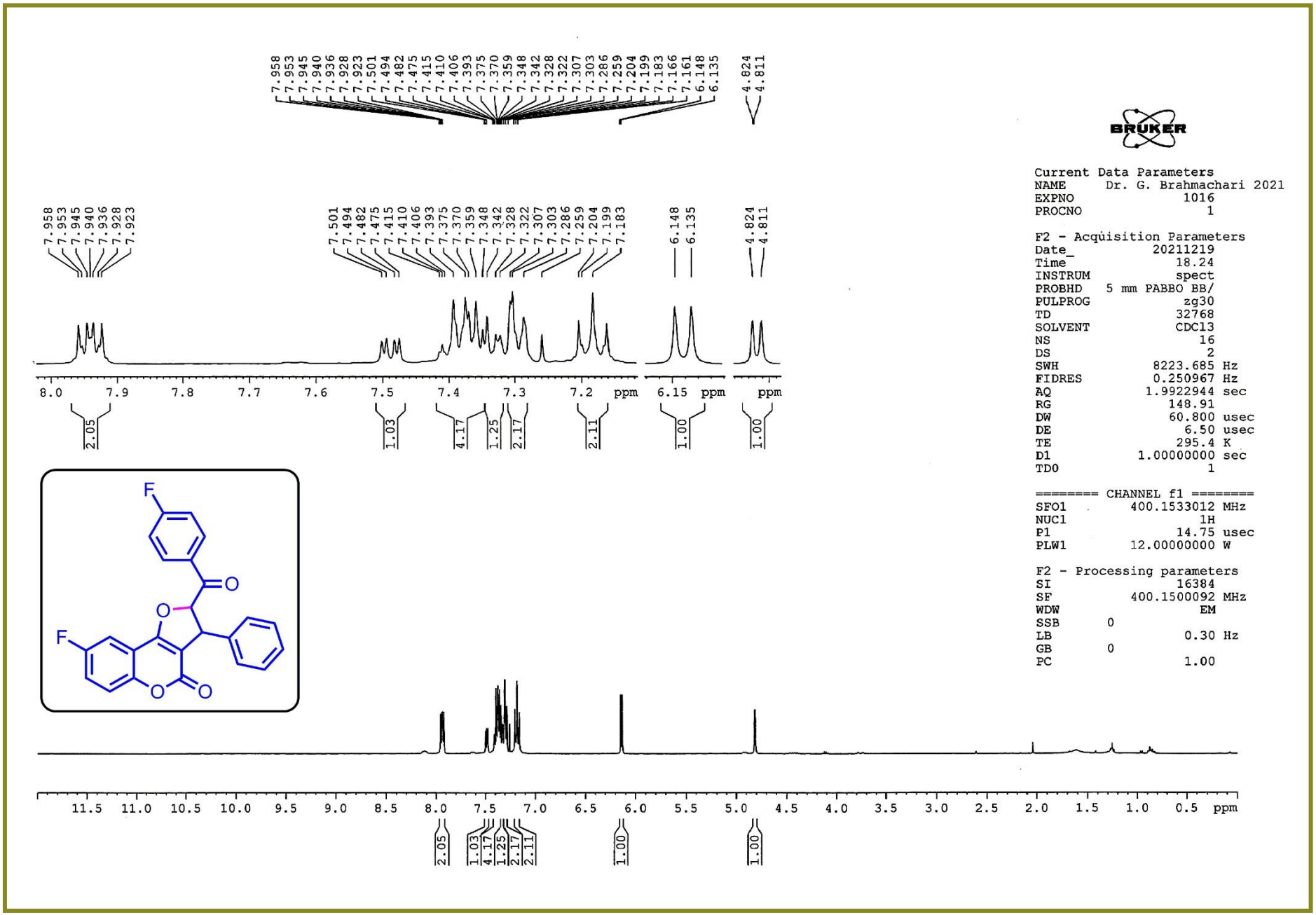


Figure S125. ^{1}H-NMR spectrum of 8-fluoro-2-(4-fluorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-29**)

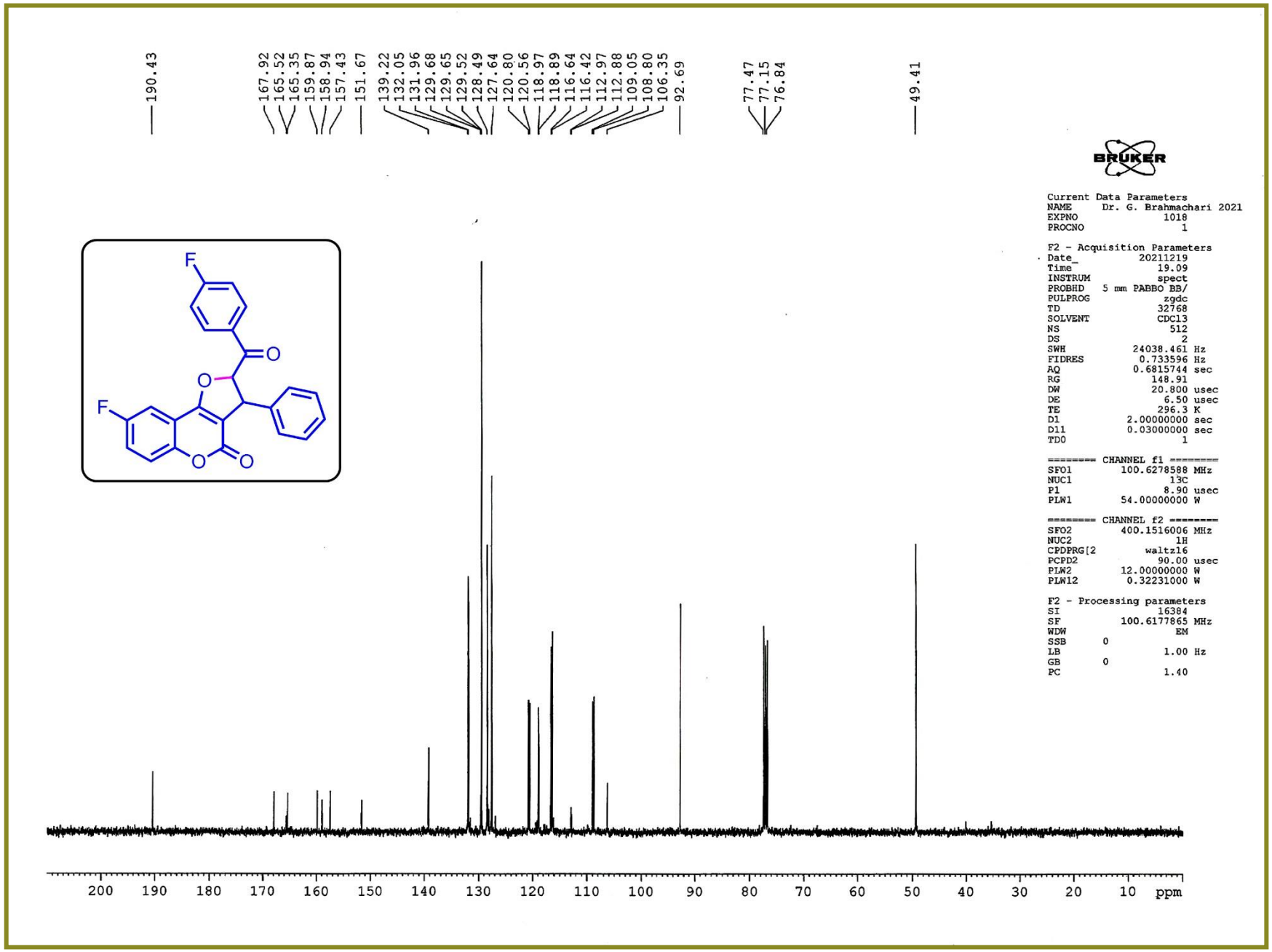


Figure S126. ^{13}C-NMR spectrum of 8-fluoro-2-(4-fluorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-29**)

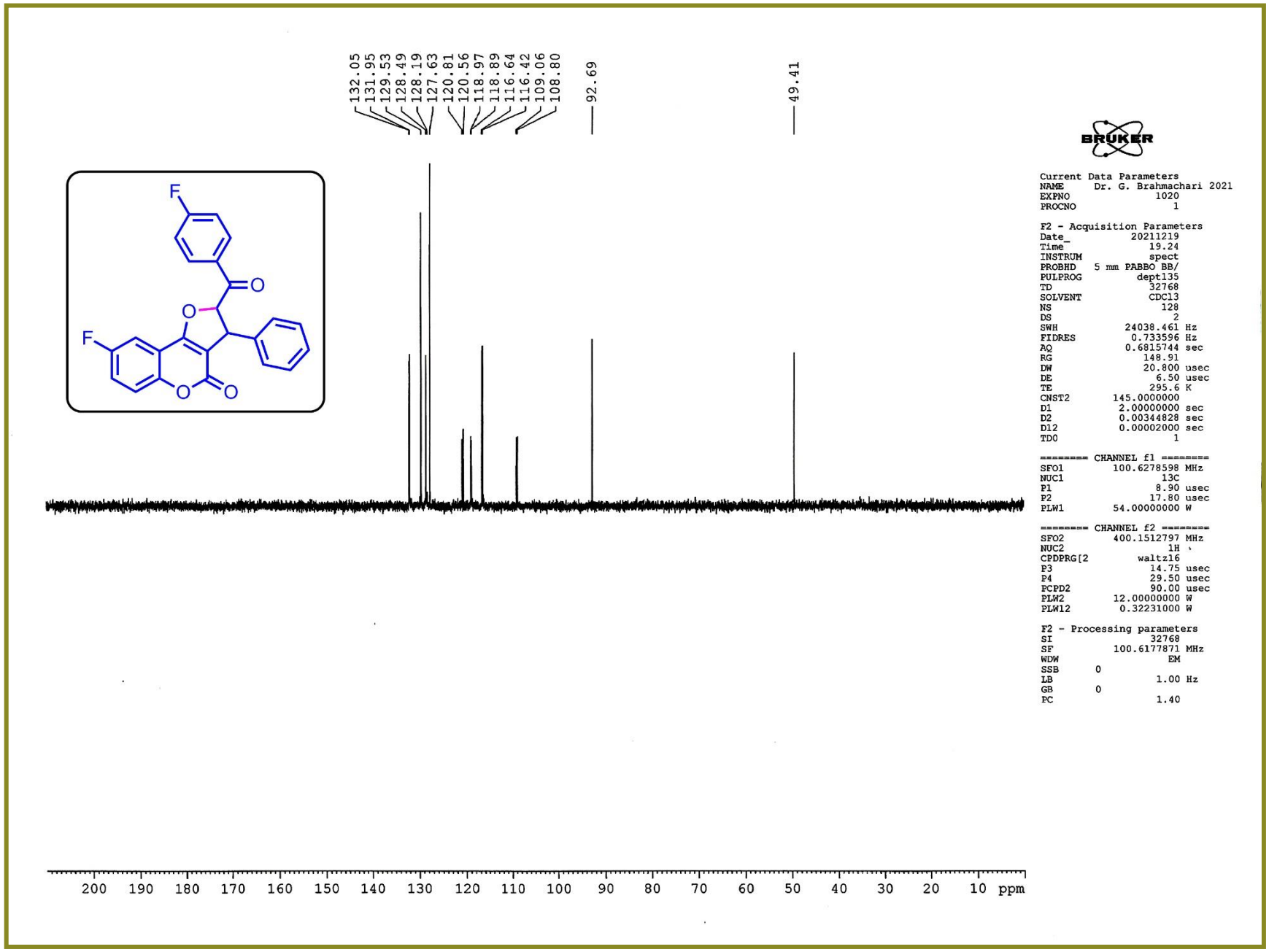


Figure S127. DEPT-135 NMR spectrum of 8-fluoro-2-(4-fluorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-29**)

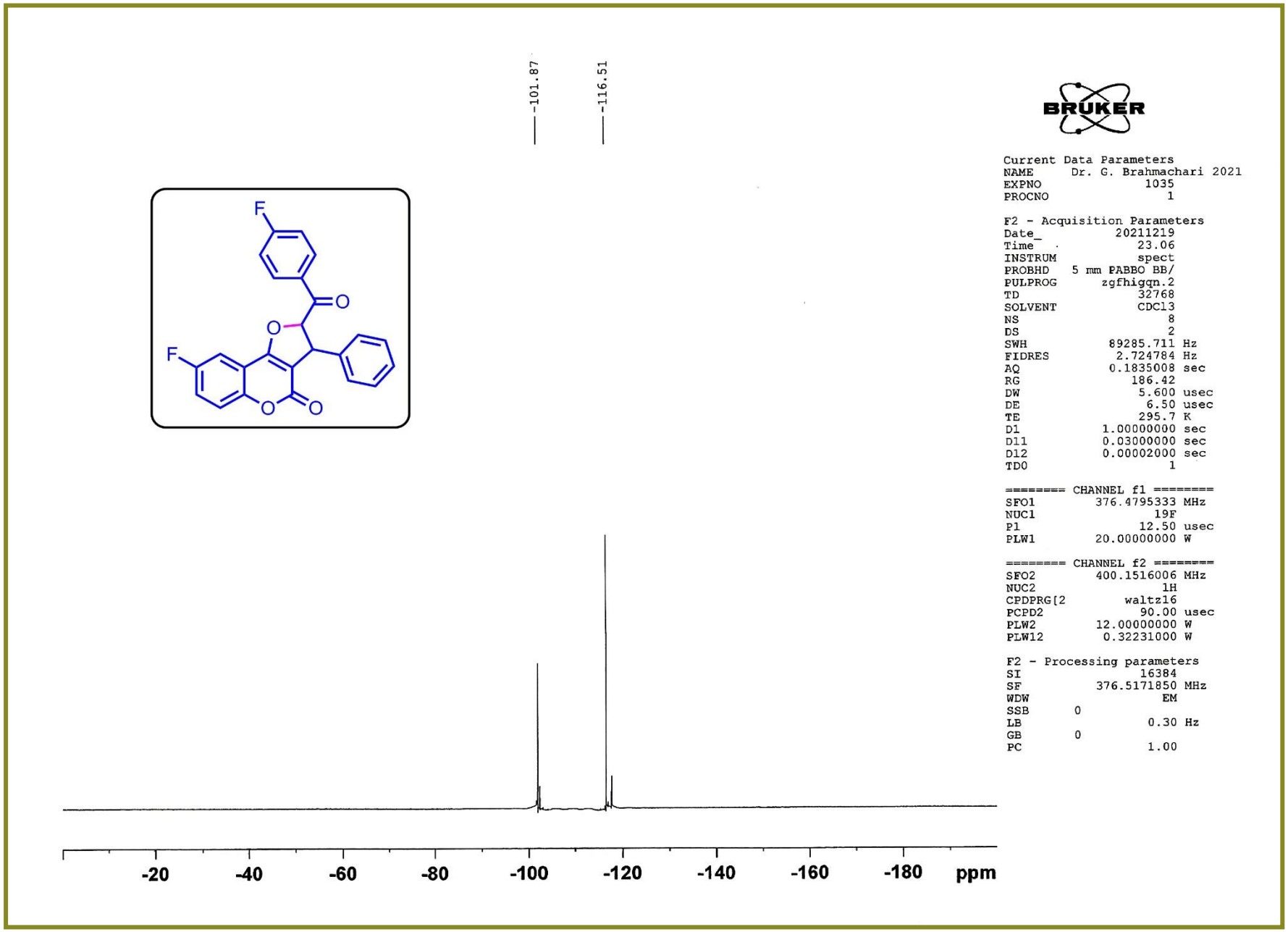


Figure S128. ^{19}F NMR spectrum of 8-fluoro-2-(4-fluorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-29**)

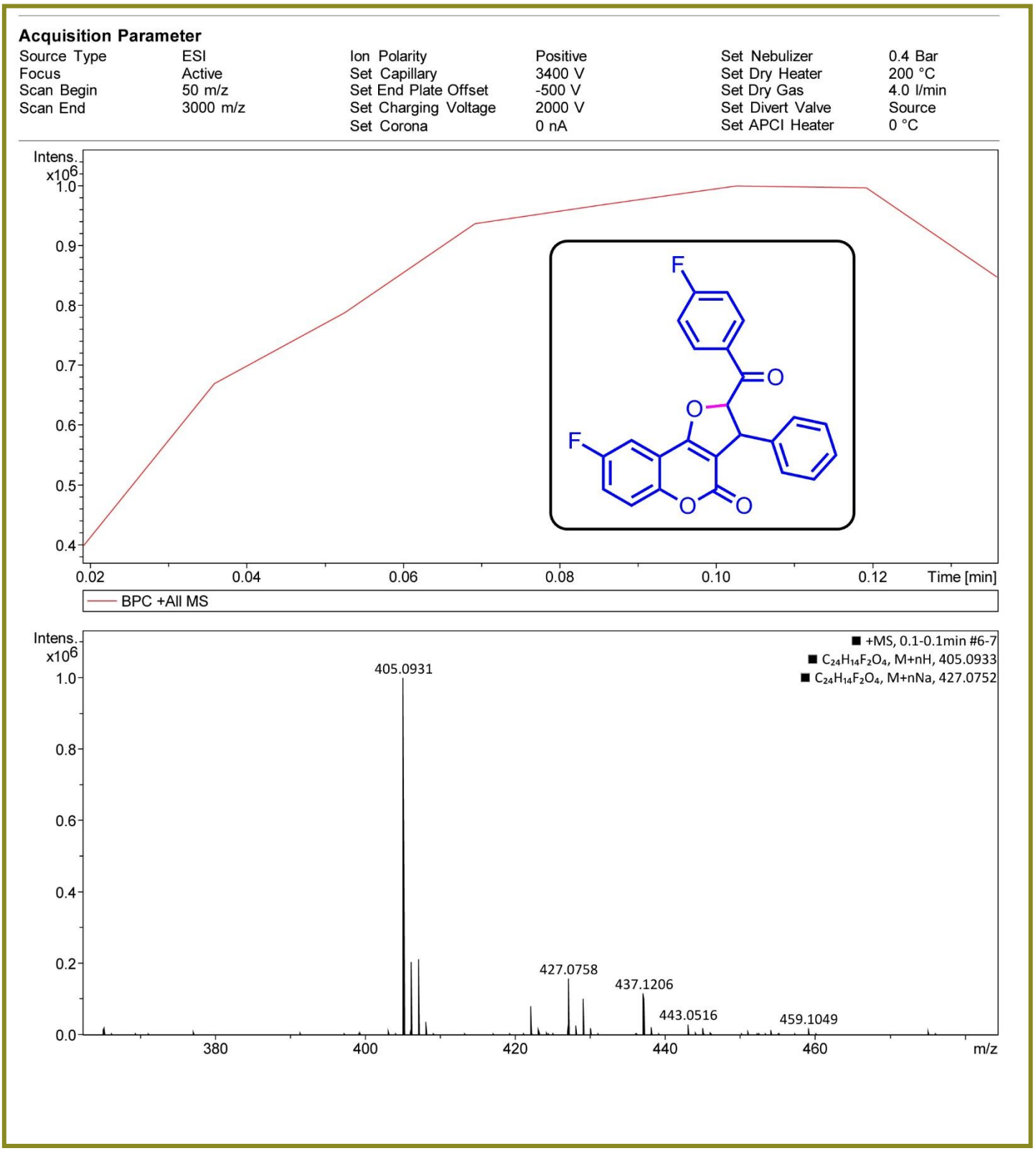


Figure S129. High-resolution Mass spectra of 8-fluoro-2-(4-fluorobenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*] chromen-4-one (**2-29**)

4. Single X-ray crystal structure analysis of 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (2-1)

*4.1 Preparation of single crystals of compound **2-1***

For preparing single crystals of compound **2-1**, 30 mg of the sample was dissolved in 5 mL of chloroform, and the solution was left for 3 days for slow evaporation at ambient temperature to yield colourless block-shaped crystals.

CCDC 2115681 (Compound **2-1**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from *The Cambridge Crystallographic Data Centre via* www.ccdc.cam.ac.uk/data_request/cif

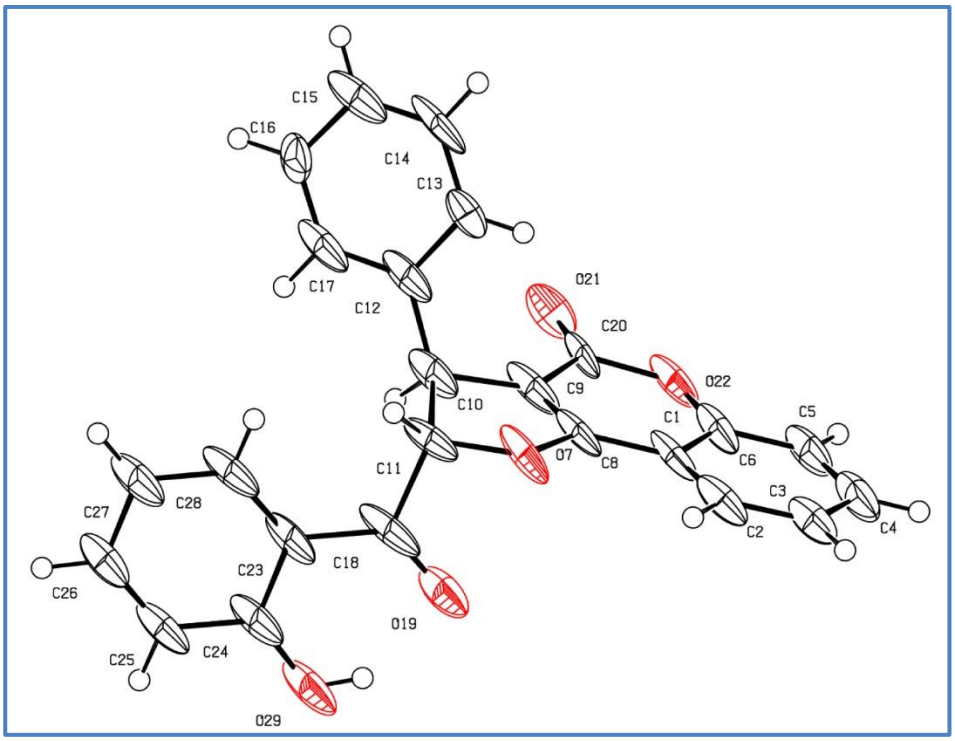


Fig. 130a *ORTEP* view of the molecule, showing the atom-labelling scheme
Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

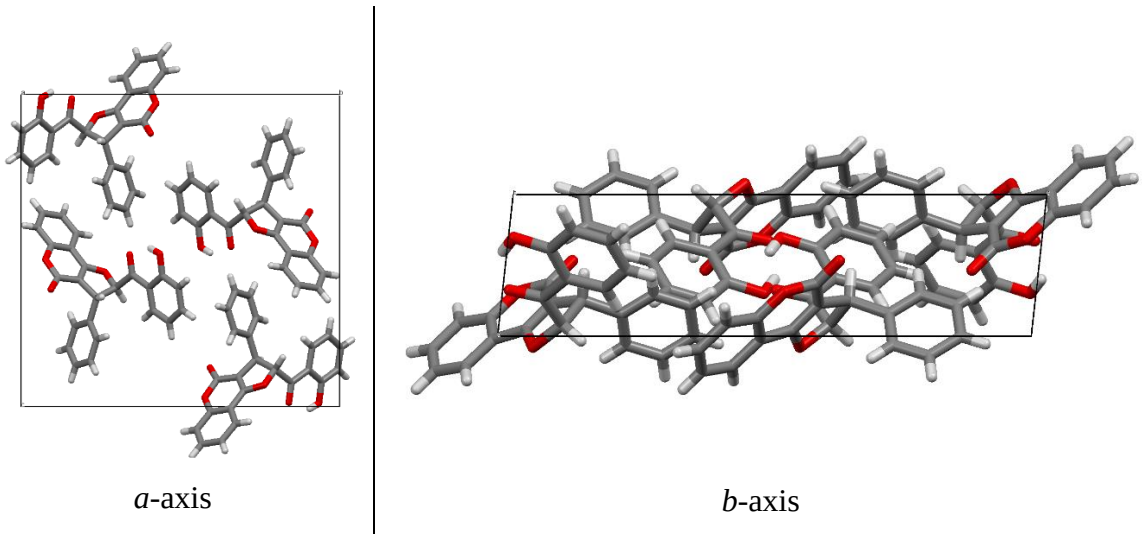


Fig. 130b. The packing arrangement of molecules viewed down the *a*-axis and *b*-axis.

Table S2. Crystal data and structure refinement for 2-(2-hydroxybenzoyl)-3-phenyl-2,3-dihydro-4*H*-furo[3,2-*c*]chromen-4-one (**2-1**)

CCDC Number	2115681	
Empirical formula	$C_{24}H_{16}O_5$	
Formula weight	384.37	
Temperature	150.00(7) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2_1/c	
Unit cell dimensions	a = 4.9980 (5) Å	α= 90°
	b = 19.0007(14) Å	β= 96.275(11)°
	c = 18.7257(19) Å	γ = 90°
Volume	1767.6 (3) $Å^3$	
Z	4	
Density (calculated)	1.444 g/cm^3	
Absorption coefficient	0.101 mm^{-1}	
F(000)	800.0	
Crystal size	0.314 × 0.254 × 0.156 mm^3	
Crystal shape (colour)	Block (colourless)	
Theta range for data collection	4.38 to 57.04°	
Index ranges	-6<=h<=6, -24<=k<=19, -13<=l<=24	
Reflections collected	5401	
Independent reflections	3658 [R_{int} = 0.1695, R_{sigma} = 0.1841]	
Completeness to theta = 28.640°	71.9 %	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3658 / 0 / 263	
Goodness-of-fit on F^2	0.934	
Final R indices [I>= 2σ (I)]	R_1 = 0.0982, wR_2 = 0.2102	
R indices (all data)	R_1 = 0.1489, wR_2 = 0.2541	
Largest diff. peak and hole	0.38 and -0.46 e.$Å^{-3}$	
Scan mode	ω scan	
Reflections observed (I > 2σ(I))	1585	
Structure determination	Direct methods	
No. of parameters refined	263	
Final residual electron density	0.38 and -0.46 e.$Å^{-3}$	
Software for geometry calculation	WinGX [2]	
Software for geometrical calculation	PARST [3]	
Software for molecular plotting	PLATON [4], Ortep3 [5]	
Software for structure solution	SHELXS-97 [6]	
Software for refinement	SHELXL-97 [7]	

5. References

1. (a) G. Brahmachari, M. Mandal and I. Karmakar, Synthesis, 2022, 54, 451-464; (b) O. Talhi, J. A. Fernandes, D. C. G. A. Pinto, F. A. A. Paz, A. M. S. Silva, *J. Mol. Struct*., 2015, **1094**, 13-21.
2. L. J. Farrugia, *J. Appl. Crystallogr*., 1999, **32**, 837.
3. M. Nardelli, *J. Appl. Crystallogr*., 1995, **28**, 659.
4. A. L. Spek, *Acta Crystallogr*. *D*., 2009, **65**, 148.
5. L. J. Farrugia, *J Appl Crystallogr*., 1997, **30**, 565.
6. G. M. Sheldrick, SHELXS97, Program for the solution of the crystal structure, University of Gottingen, Germany, 1997.
7. G. M. Sheldrick, SHELXL97, University of Göttingen, Germany, 1997.