Supporting Information

Synthesis of Polysubstituted Tetrahydrofurans via Visible Light-Induced De Mayo Acetalization of Benzoylacetones

Ranadheer Reddy Indurmuddam,[a] Bor-Cherng Hong,*[a] and Su-Ying Chien[b]

[a] Department of Chemistry and Biochemistry, National Chung Cheng University, Chia-Yi, 621, Taiwan, R.O.C., and
[b] Instrumentation Center, National Taiwan University, Taipei, 106, Taiwan, R.O.C.
E-mail: chebch@ccu.edu.tw

SUPPORTING INFORMATION:

Contents:

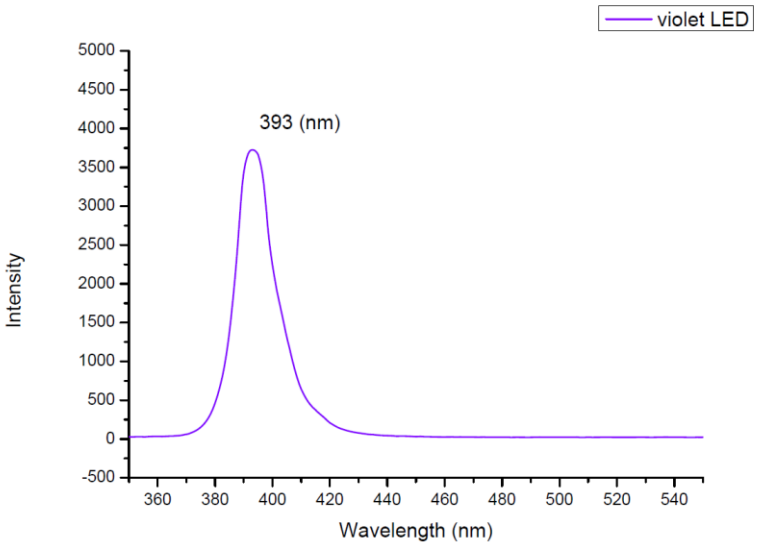
General Procedure.

All solvents were reagent grade. Catalyst thioxanthone was purchased from Aldrich Chemicals, Tokyo Chemical Industry Co., Ltd., Alfa Aesar, etc. Reactions were normally carried out under a nitrogen atmosphere in glassware or vial. Merck silica gel 60 (particle size 0.04-0.063 mm) was employed for flash chromatography. ^{1}H NMR spectra were obtained in $CDCl_3$ unless otherwise noted at 400 MHz (Bruker DPX-400, Bruker Ascend^{TM} 400**)** or 500 MHz (Varian-Unity INOVA-500).^{13}C NMR spectra were obtained at 125 MHz or 100 MHz. The melting point was recorded on a melting point apparatus (MPA100–Automated melting point system, Stanford Research Systems, Inc.) and is uncorrected. IR spectra were recorded on Bruker Alpha FT-IR spectrometer. ESI ionization time-of-flight mass (ESI-TOF HRMS) spectral data were collected on a JMS-T100LP 4G(JEOL) mass spectrometer equipped with the ESI source, detecting positive and negative ions. Typical measurement conditions are as follows: needle voltage: 2000 kV, orifice 1 voltage: 30 V, ring lens voltage: 10 V, spray temperature: 250 °C. EI-TOF mass spectral data were collected on a JMS-T200GC AccuTOF GCx-plus (JEOL) mass spectrometer. EI ionization time-of-flight mass (EI-TOF HRMS) spectral data were collected on a JMS-T200GC AccuTOF GCx-plus (JEOL) mass spectrometer equipped with the EI ion source and DIP sampling device. Typical measurement conditions are as follows: ionizing voltage: 70 eV, ionizing current: 300 μA, ion chamber temperature: 250 ℃, DIP temperature: 50 to 200 ℃ in 2 minutes. The single-crystal X-ray diffraction data of crystals were individually collected in-house on a Bruker D8 Venture diffractometer equipped with a Cu-target (Kα =1.54178 Å) or Mo-target (Kα = 0.71073 Å) microfocus X-ray generators and a

PHOTON-II CMOS detector. The temperature was adjusted with a nitrogen flow (Oxford Cryosystems). After collection, the data were integrated with the Bruker SAINT software package using a narrow-frame algorithm and were corrected for absorption effects using the Multi-Scan method (SADABS). Then, the molecular structure was solved and refined by the Bruker SHELXTL Software Package and the final anisotropic full-matrix least-squares method was used to refine on F2 with variables parameters to determine crystal structure. UV-Vis was recorded on HITACHI U-3310 Spectrophotometer, Fluorescence was recorded by HITACHI F-7000 Fluorescence Spectrophotometer.

For the UV lamps used in Table 1, entry 30: Regular ultraviolet (UV) blacklight CFL light bulb (365 nm, 40 W, 110 V, 50 Hz).

Violet LED lamps were purchased from https://honlychem.com; LED wavelength was measured by StellarNet EPP2000 Spectrometer (StellarNet, Inc.); LED optical power was measured by Optical Power Meter PM100A (thorlabs.com).



Violet LED

Power:

1 cm: 12 W

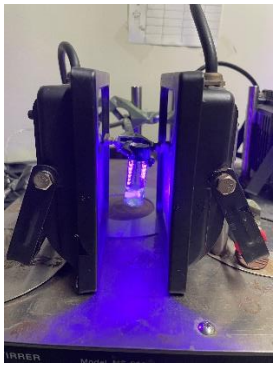
2 cm: 7.7 W

3 cm: 5.5 mW

4 cm: 3.95 mW

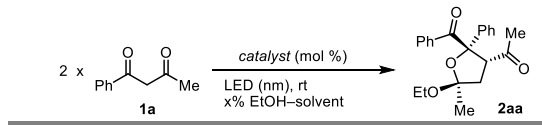
5 cm: 2.8 mW

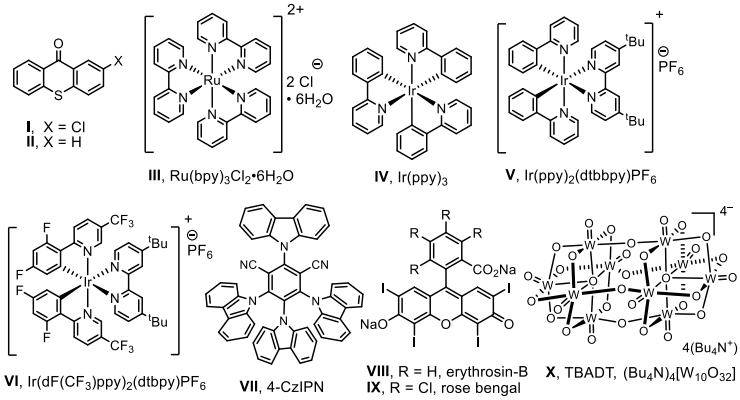
393 nm LED lamp



reaction set up

Table S1. Screening for the Conversion of **1a** to **2aa**[a]

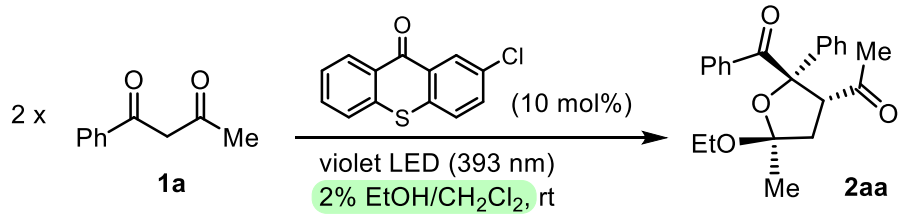




entry	catalyst (mol%)	LED light (nm)	solvent	time (h)	yield (%)[b]
1	**I** (10)	violet (393)	$CHCl_3$[c]	48	55 (51)
2	**I** (10)	violet (393)	CH_2Cl_2	48	0[d]
3	**I** (10)	violet (393)	EtOH	72	nr
4	**I** (10)	violet (393)	1% EtOH/CH_2Cl_2	48	50
5	**I** (10)	violet (393)	2% EtOH/CH_2Cl_2	48	85(76)[e]
6	**I** (10)	violet (393)	3% EtOH/CH_2Cl_2	48	71(64)
7	**I** (10)	violet (393)	4% EtOH/CH_2Cl_2	48	62
8	**I** (10)	violet (393)	5% EtOH/CH_2Cl_2	48	33
9	**I** (10)	violet (393)	10% EtOH/CH_2Cl_2	48	24
10	**I** (10)	violet (393)	50% EtOH/CH_2Cl_2	72	nr
11	**I** (10)	violet (393)	2% EtOH/CH_3CN	48	19
12	**I** (10)	violet (393)	2% EtOH/THF	48	nr
13	**I** (10)	violet (393)	2% EtOH/acetone	48	8
14	**I** (10)	violet (393)	2% EtOH/DMF	48	nr
15	**I** (10)	violet (393)	2% EtOH/$CDCl_3$	48	54
16[f]	**I** (10)	violet (393)	2% EtOH/CH_2Cl_2	48	61
17[g]	**I** (10)	violet (393)	2% EtOH/CH_2Cl_2	48	58
18[h]	**I** (10)	violet (393)	2% EtOH/CH_2Cl_2	48	73
19	**II** (10)	violet (393)	2% EtOH/CH_2Cl_2	48	41
20	**III** (2)	blue (450)	2% EtOH/CH_2Cl_2	72	nr
21	**IV** (2)	blue (450)	2% EtOH/CH_2Cl_2	48	11
22	**V** (2)	blue (450)	2% EtOH/CH_2Cl_2	48	20
23	**VI** (2)	blue (450)	2% EtOH/CH_2Cl_2	48	49
24	**VII** (2)	blue (450)	2% EtOH/CH_2Cl_2	48	55
25	**VIII** (2)	green (515)	2% EtOH/CH_2Cl_2	72	nr
26	**IX** (2)	green (515)	2% EtOH/CH_2Cl_2	72	nr
27	**X** (5)	violet (393)	2% EtOH/CH_2Cl_2	72	nr
28	**I** (10)	dark	2% EtOH/CH_2Cl_2	48	nr
29	-	violet (393)	2% EtOH/CH_2Cl_2	48	nr
30	-	UV (365)	2% EtOH/CH_2Cl_2	48	nr

[a] Under ambient temperature and LED irradiation, the reaction was conducted in a capped vial containing **1a** (0.1 M) and a catalyst (2−10 mol %) in a solvent. [b] The NMR yield was determined by ^{1}H NMR spectroscopy analysis using 1,3,5-trimethylbenzene as an internal standard, with the isolated yield of **2aa** provided in parentheses. [c]Commercial chloroform usually contains 0.5-1.0% ethanol as a stabilizer. [d] Complicated mixtures were observed. [e] dr>20:1 [f] A concentrated aqueous HCl solution (3:1 *v*/*v*, ~40 equiv of HCl) was added to the reaction mixture. [g] The reaction was carried out in 0.2 M of **1a**. [h]The reaction was carried out in 0.05 M of **1a**. nr = no reaction

Preparation of 2aa:



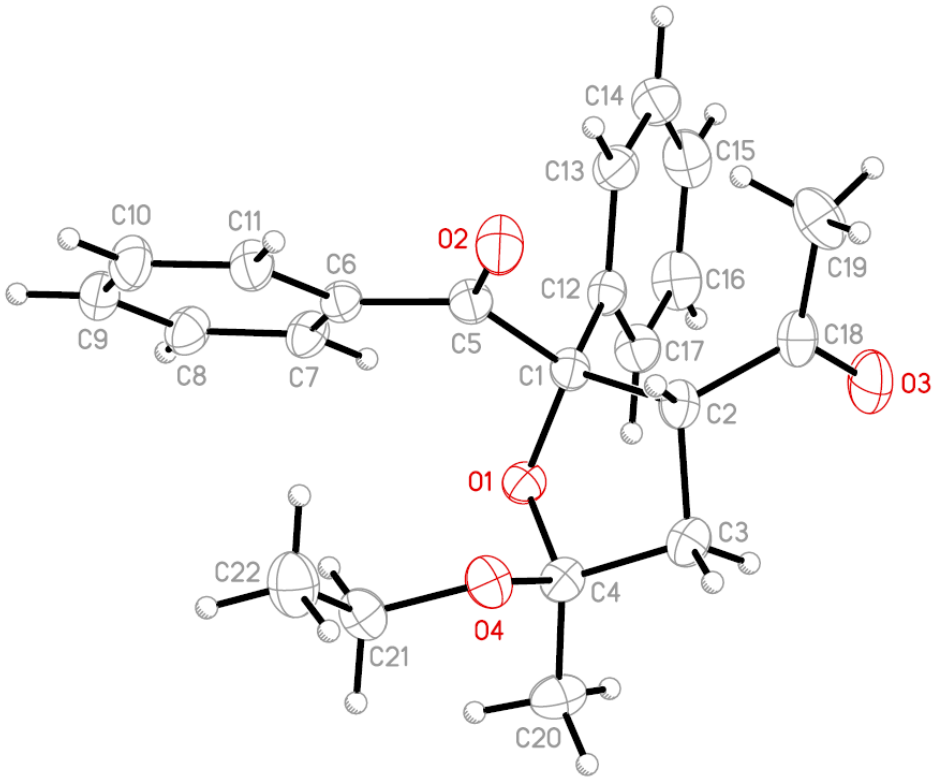
A magnetic stirring bar and 1-phenylbutane-1,3-dione (**1a**, 20 mg, 0.12 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.01 mmol, 0.1 equiv) in 2% ethanol/CH2Cl2 (1.2 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2aa** (R_f = 0.44 in 20% EtOAc–hexane; 16.5 mg, 76% yield) as a yellow solid.

Selected data for **2aa**: mp: 98-99 °C; IR (neat): 3064, 2981, 2933, 2886, 1712, 1683, 1592, 1446, 1357, 1314, 1227, 1177, 1106, 1034, 952, 868, 755, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.92 (dd, J = 8.5, 1.5 Hz, 2 H), 7.42 – 7.37 (m, 1 H), 7.31 – 7.19 (m, 7 H), 4.75 (t, J = 8.0 Hz, 1 H), 3.19 (dq, J = 8.8, 7.0 Hz, 1 H), 2.71 (dq, J = 8.8, 7.0 Hz, 1 H), 2.48 (dd, J = 13.0, 8.0 Hz, 1 H), 2.06 (dd, J = 13.0, 8.0 Hz, 1 H), 1.80 (s, 3 H), 1.67 (s, 3 H), 0.58 (t, J = 7.0 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.7 (C), 198.6 (C), 137.9 (C), 134.0 (C), 132.6 (CH), 131.1 (two CH), 128.6 (two CH), 128.3 (CH), 127.8 (two CH), 125.4 (two CH), 108.7 (C), 93.5 (C), 57.5 (CH_2), 57.3 (CH), 41.1 (CH_2), 31.2 (CH_3), 21.3 (CH_3), 14.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{22}H_{24}O_4$ 352.1675; found 352.1666.

Recrystallization of 2aa was performed as follows:

The compound (**2aa**, ~5 mg) in a screw-capped vial (4 mL vial) was dissolved in $CHCl_3$ (~0.5 mL) and diluted with *n*-hexane (~2.5 mL). The vial was covered with aluminum foil (having 4-5 holes on it) and placed in another vial (20 mL vial) filled with *n*-hexane (~8 mL). The 20 mL vial was closed gently with a screw cap and stands it for 5 days until complete evaporation of the solvent in the inner vial. The crystals formed were subjected to single-crystal X-ray analysis.

[1] Purchased from Combi-Blocks Inc.



Thermal ellipsoids draw at the 30% probability level

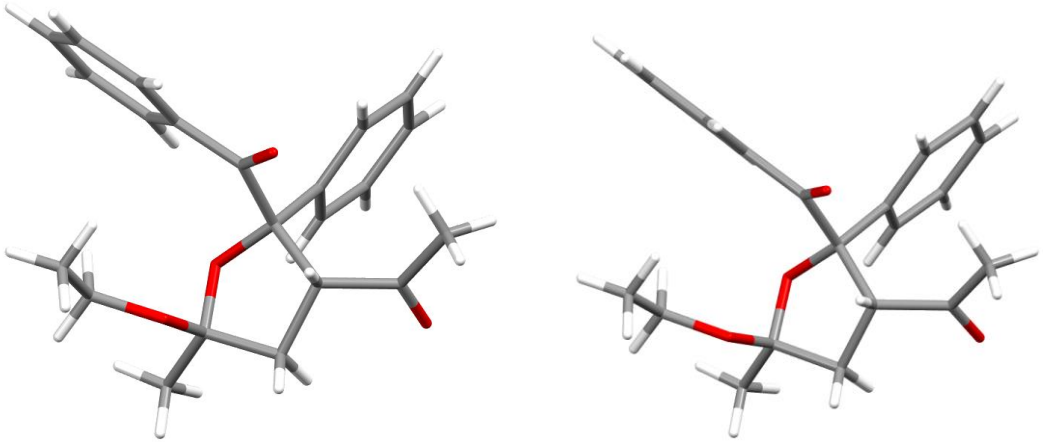


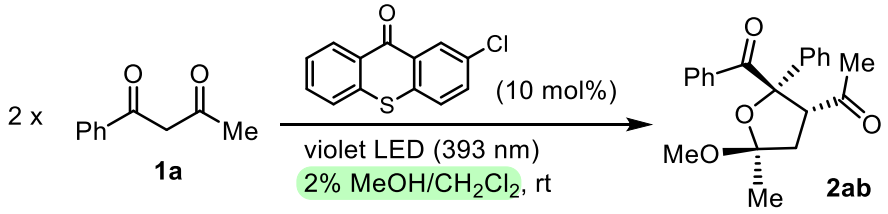
Figure S1. ORTEP and Stereo plots for X-ray crystal structures of **2aa (**ic22359).

CCDC 2382209 contains the supplementary crystallographic data for **2aa (**ic22359). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk

Table S2. Crystal data and structure refinement for **2aa (**ic22359).

Identification code	ic22359	
Empirical formula	C22 H24 O4	
Formula weight	352.41	
Temperature	200(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.3493(7) Å	= 111.527(3)°.
	b = 10.3182(8) Å	= 96.606(3)°.
	c = 12.4869(10) Å	= 104.002(3)°.
Volume	945.89(13) $Å^3$	
Z	2	
Density (calculated)	1.237 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	376	
Crystal size	0.239 x 0.207 x 0.134 mm^3	
Theta range for data collection	2.219 to 30.000°.	
Index ranges	-10<=h<=11, -14<=k<=14, -17<=l<=17	
Reflections collected	19902	
Independent reflections	5523 [R(int) = 0.0726]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9602 and 0.7947	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5523 / 0 / 238	
Goodness-of-fit on F^2	1.036	
Final R indices [I>2sigma(I)]	R1 = 0.0657, wR2 = 0.1580	
R indices (all data)	R1 = 0.1118, wR2 = 0.1925	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.422 and -0.203 $e.Å^{-3}$	

Preparation of 2ab:

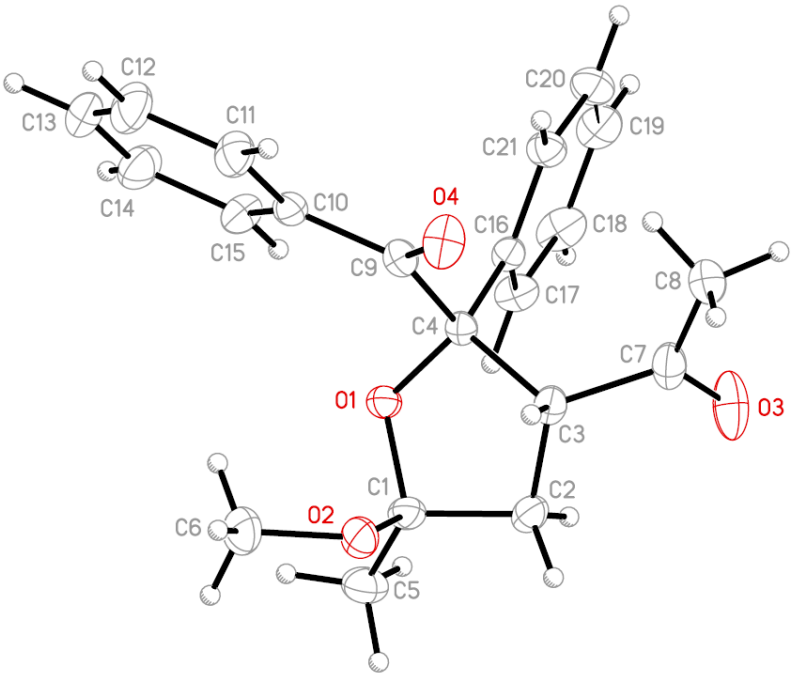


A magnetic stirring bar and 1-phenylbutane-1,3-dione (**1a**, 20 mg, 0.12 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.01 mmol, 0.1 equiv) in 2% Methanol/CH_2Cl_2 (1.2 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ab** (R_f = 0.37 in 15% EtOAc–hexane; 15.8 mg, 76% yield) as a yellow solid.

Selected data for **2ab**: mp: 121-122 °C; IR (neat): 3062, 2990, 2952, 2831, 1711, 1683, 1592, 1490, 1446, 1355, 1316, 1228, 1178, 1106, 1030, 872, 755, 700 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.87 – 7.83 (m, 2 H), 7.41 – 7.36 (m, 1 H), 7.30 – 7.24 (m, 7 H), 4.72 (t, J = 7.9 Hz, 1 H), 2.56 (s, 3 H), 2.49 (dd, J = 13.1, 7.9 Hz, 1 H), 2.04 (dd, J = 13.1, 7.9 Hz, 1 H), 1.76 (s, 3 H), 1.64 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.7 (C), 198.9 (C), 137.6 (C), 134.4 (C), 132.4 (CH), 131.0 (two CH), 128.7 (two CH), 128.4 (CH), 127.8 (two CH), 125.5 (two CH), 109.2 (C), 93.7 (C), 57.3 (CH), 49.6 (CH_3), 41.0 (CH_2), 31.2 (CH_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{22}O_4$ 338.1518; found 338.1510.

Recrystallization of 2ab was performed as follows:

The compound (**2ab**, ~5 mg) in a screw-capped vial (4 mL vial) was dissolved in CH_2Cl_2 (~0.5 mL) and diluted with *n*-hexane (~2.5 mL). The vial was covered with aluminum foil (having 4-5 holes on it) and placed in another vial (20 mL vial) filled with *n*-hexane (~8 mL). The 20 mL vial was closed gently with a screw cap and stands it for 5 days until complete evaporation of the solvent in the inner vial. The crystals formed were subjected to single-crystal X-ray analysis.



Thermal ellipsoids draw at the 30% probability level

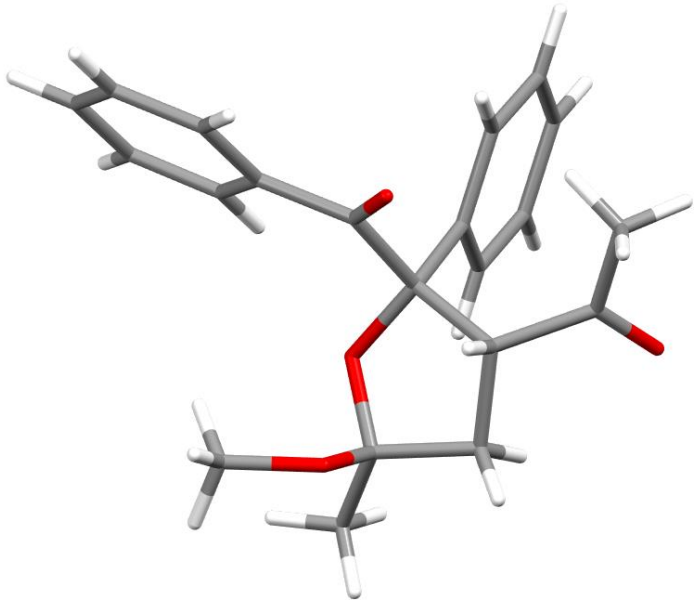


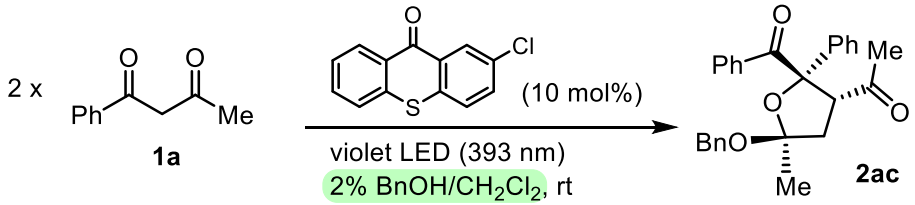
Figure S2. ORTEP and Stereo plots for X-ray crystal structures of **2ab** (ic22493).

CCDC 2382210 contains the supplementary crystallographic data for **2ab** (ic22493). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk

Table S3. Crystal data and structure refinement for **2ab (**ic22493).

Identification code	ic22493	
Empirical formula	C21 H22 O4	
Formula weight	338.38	
Temperature	200(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 17.4908(4) Å	α= 90°.
	b = 8.9534(2) Å	β= 108.5396(8)°.
	c = 24.3196(7) Å	γ = 90°.
Volume	3610.85(16) $Å^3$	
Z	8	
Density (calculated)	1.245 Mg/m^3	
Absorption coefficient	0.691 mm^{-1}	
F(000)	1440	
Crystal size	0.395 x 0.249 x 0.181 mm^3	
Theta range for data collection	3.834 to 74.425°.	
Index ranges	-21<=h<=21, -11<=k<=11, -30<=l<=29	
Reflections collected	39618	
Independent reflections	3684 [R(int) = 0.0246]	
Completeness to theta = 67.679°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9819 and 0.8673	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3684 / 0 / 229	
Goodness-of-fit on F^2	1.037	
Final R indices [I>2sigma(I)]	R1 = 0.0411, wR2 = 0.1069	
R indices (all data)	R1 = 0.0427, wR2 = 0.1083	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.202 and -0.263 $e.Å^{-3}$	

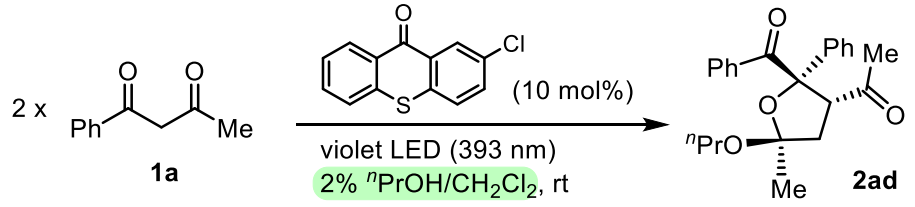
Preparation of 2ac:



A magnetic stirring bar and 1-phenylbutane-1,3-dione (**1a**, 20 mg, 0.12 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.01 mmol, 0.1 equiv) in 2% benzyl alcohol/CH_2Cl_2 (1.2 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ac** (R_f = 0.46 in 15% EtOAc–hexane; 20.6 mg, 81% yield) as a yellow liquid.

Selected data for **2ac**: IR (neat): 3064, 3030, 2991, 2949, 1711, 1681, 1595, 1447, 1357, 1314, 1225, 1176, 1108, 1025, 881, 753, 698 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.80 – 7.77 (m, 2 H), 7.37 – 7.24 (m, 6 H), 7.21 – 7.14 (m, 5 H), 6.87 – 6.84 (m, 2 H), 4.82 (dd, J = 8.8, 7.8 Hz, 1 H), 4.22 (d, J = 11.6 Hz, 1 H), 3.74 (d, J = 11.6 Hz, 1 H), 2.58 (dd, J = 13.0, 8.8 Hz, 1 H), 2.22 (dd, J = 13.0, 7.8 Hz, 1 H), 1.86 (s, 3 H), 1.78 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.4 (C), 198.4 (C), 138.2 (C), 137.9 (C), 133.9 (C), 132.5 (CH), 131.1 (two CH), 128.6 (two CH), 128.4 (CH), 127.9 (two CH), 127.7 (two CH), 126.9 (CH), 126.6 (two CH), 125.4 (two CH), 109.1 (C), 93.9 (C), 63.9 (CH_2), 57.3 (CH), 41.1 (CH_2), 31.2 (CH_3), 21.6 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{27}H_{26}O_4$ 414.1831; found 414.1823.

Preparation of 2ad:

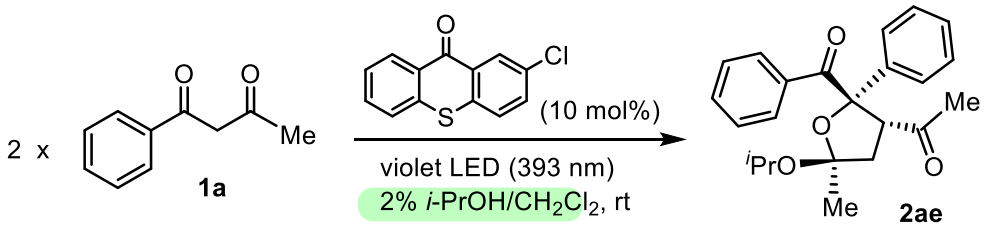


A magnetic stirring bar and 1-phenylbutane-1,3-dione (**1a**, 20 mg, 0.12 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.01 mmol, 0.1 equiv) in 2% *n*-propanol/CH_2Cl_2 (1.2 mL).

The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ad** (R_f = 0.42 in 15% EtOAc–hexane; 13.7 mg, 61% yield) as a pale-yellow solid.

Selected data for **2ad**: mp: 79-80 °C; IR (neat): 3064, 2963, 2877, 1712, 1683, 1593, 1447, 1357, 1314, 1227, 1177, 1106, 1028, 877, 755, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.95 – 7.90 (m, 2 H), 7.43 – 7.37 (m, 1 H), 7.31 – 7.21 (m, 7 H), 4.75 (t, *J* = 8.0 Hz, 1 H), 3.07 (td, *J* = 8.5, 6.4 Hz, 1 H), 2.61 (td, *J* = 8.5, 5.5 Hz, 1 H), 2.49 (dd, *J* = 12.9, 8.0 Hz, 1 H), 2.07 (dd, *J* = 12.9, 8.0 Hz, 1 H), 1.84 (s, 3 H), 1.68 (s, 3 H), 1.16 – 1.05 (m, 1 H), 0.93 – 0.83 (m, 1 H), 0.45 (t, *J* = 7.4 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): *δ* 207.6 (C), 198.5 (C), 138.0 (C), 134.0 (C), 132.5 (CH), 131.2 (two CH), 128.6 (two CH), 128.3 (CH), 127.8 (two CH), 125.4 (two CH), 108.7 (C), 93.5 (C), 63.7 (CH_2), 57.3 (CH), 41.0 (CH_2), 31.2 (CH_3), 22.4 (CH_2), 21.3 (CH_3), 10.0 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{23}H_{26}O_4$ 366.1831; found 366.1824.

Preparation of 2ae:

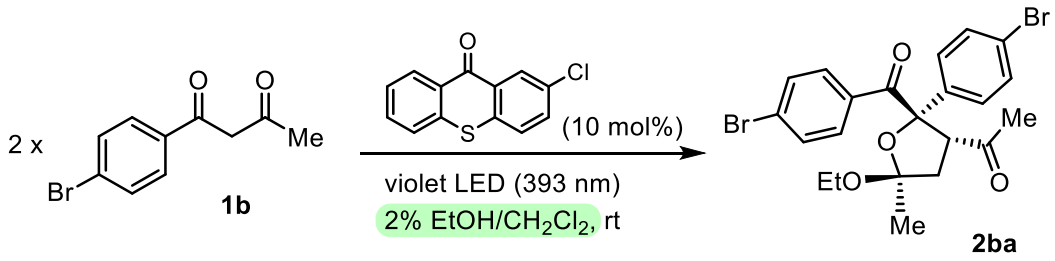


A magnetic stirring bar and 1-phenylbutane-1,3-dione (**1a**, 20 mg, 0.123 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.01 mmol, 0.1 equiv) in 2% *iso*-propanol/CH_2Cl_2 (1.2 mL). The vial was purged with argon and closed with a screw cap. The vial was stirred and irradiated with a violet LED (393 nm, 2x10 W) at r.t. for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ae** (R_f = 0.40 in 15% EtOAc–hexane; 9.9 mg, 44% yield) as a pale-yellow liquid. The diastereomeric ratio of the products was determined to be ca. 14:1 by ^1H NMR analysis.

Selected data for **2ae**: IR (neat): 3062, 2979, 2926, 1712, 1683, 1592, 1448, 1380, 1309, 1225, 1177, 1101, 989, 880, 756, 700 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$, for the major isomer): *δ* 7.95 – 7.92 (m, 2 H), 7.42 – 7.37 (m, 1 H), 7.28 – 7.23 (m, 4 H), 7.23

– 7.21 (m, 1 H), 7.20 – 7.16 (m, 2 H), 4.72 (dd, J = 11.2, 7.2 Hz, 1 H), 3.68 – 3.59 (m, 1 H), 2.44 (dd, J = 12.8, 11.2 Hz, 1 H), 2.04 (s, 3 H), 2.03 (dd, J = 12.8, 11.2 Hz, 1 H), 1.75 (s, 3 H), 0.98 (d, J = 6.0 Hz, 3 H), 0.55 (d, J = 6.0 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$, for the major isomer): δ 206.9 (C), 197.9 (C), 138.7 (C), 134.2 (C), 132.7 (CH), 131.4 (two CH), 128.6 (two CH), 128.2 (CH), 127.8 (two CH), 125.0 (two CH), 108.5 (C), 93.6 (C), 64.4 (CH), 57.0 (CH), 41.0 (CH_2), 31.3 (CH_3), 24.4 (CH_3), 23.4 (CH_3), 23.3 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{23}H_{26}O_4$ 366.1831; found 366.1823.

Preparation of 2ba:



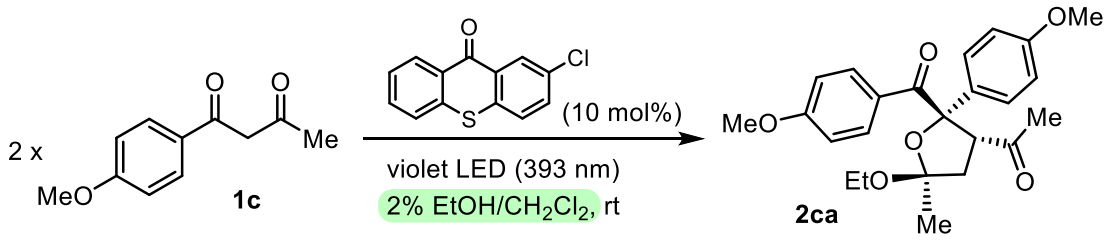
A magnetic stirring bar and 1-(4-bromophenyl)butane-1,3-dione (**1b**, 20 mg, 0.08 mmol)[2] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.0 mg, 0.008 mmol, 0.1 equiv) in 2% ethanol/CH_2Cl_2 (0.8 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5-10% EtOAc–hexane to afford **2ba** (R_f = 0.42 in 20% EtOAc–hexane; 13.1 mg, 62% yield) as a yellow solid.

Selected data for **2ba**: mp: 128-129 °C; IR (neat): 2979, 2933, 1711, 1684, 1582, 1484, 1394, 1225, 1176, 1105, 1069, 1007, 950, 813 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.79 – 7.76 (m, 2 H), 7.44 – 7.39 (m, 4 H), 7.10 (d, J = 8.0 Hz, 2 H), 4.73 (t, J = 8.5 Hz, 1 H), 3.23 (dq, J = 8.8, 7.0 Hz, 1 H), 2.74 (dq, J = 8.8, 7.0 Hz, 1 H), 2.43 (dd, J = 13.0, 8.5 Hz, 1 H), 2.07 (dd, J = 13.0, 8.5 Hz, 1 H), 1.86 (s, 3 H), 1.66 (s, 3 H), 0.62 (t, J = 7.0 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.2 (C), 197.1 (C), 136.8 (C), 132.6 (two CH), 132.4 (C), 131.9 (two CH), 131.3 (two CH), 128.2 (C), 127.1 (two CH), 122.8 (C), 109.0 (C), 92.9 (C), 57.6 (CH_2), 57.0 (CH), 41.2 (CH_2), 31.5 (CH_3), 21.2

[2] Compound **1b** was prepared according to literature procedures: (a) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244. (b) Yuan, Y.; Hou, W.; Zhang-Negrerie, D.; Zhao, K.; Du, Y. *Org. Lett.* **2014**, *16*, 5410 – 5413.

(CH_3), 14.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{22}H_{22}O_4Br_2$ 507.9885; found 507.9878.

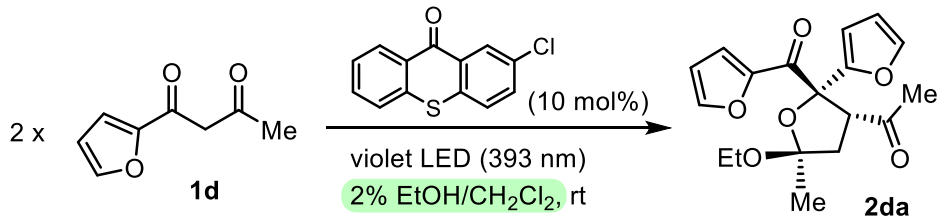
Preparation of 2ca:



A magnetic stirring bar and 1-(4-methoxyphenyl)butane-1,3-dione (20 mg, 0.10 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.6 mg, 0.01 mmol, 0.1 equiv) in 2% ethanol/CH_2Cl_2 (1.0 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ca** (R_f = 0.34 in 15% EtOAc–hexane; 15.7 mg, 73% yield) as a yellow liquid.

Selected data for **2ca**: IR (neat): 2973, 2933, 2841, 1712, 1673, 1600, 1510, 1307, 1252, 1176, 1109, 1030, 833 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.93 (d, J = 9.0 Hz, 2 H), 7.14 (d, J = 9.0 Hz, 2 H), 6.77 (d, J = 9.0 Hz, 2 H), 6.75 (d, J = 9.0 Hz, 2 H), 4.75 (t, J = 8.5 Hz, 1 H), 3.78 (s, 3 H), 3.73 (s, 3 H), 3.20 (dq, J = 8.8, 7.0 Hz, 1 H), 2.78 (dq, J = 8.8, 7.0 Hz, 1 H), 2.47 (dd, J = 12.9, 8.5 Hz, 1 H), 2.04 (dd, J = 12.9, 8.5 Hz, 1 H), 1.83 (s, 3 H), 1.65 (s, 3 H), 0.62 (t, J = 7.0 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.9 (C), 197.4 (C), 162.9 (C), 159.3 (C), 133.5 (two CH), 130.4 (C), 127.0 (C), 126.8 (two CH), 113.8 (two CH), 113.0 (two CH), 108.3 (C), 93.2 (C), 57.4 (CH_2), 57.0 (CH), 55.3 (CH_3), 55.1 (CH_3), 41.0 (CH_2), 31.2 (CH_3), 21.3 (CH_3), 14.6 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{24}H_{28}O_6$ 412.1886; found 412.1880.

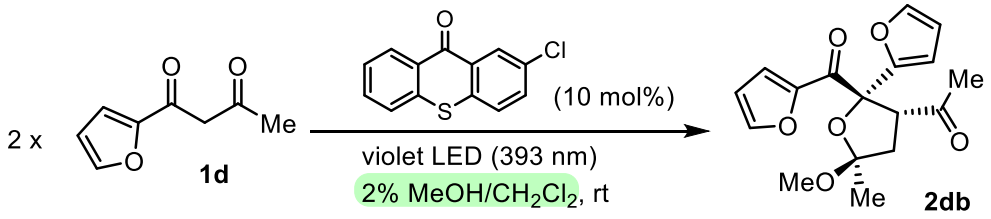
Preparation of 2da:



A magnetic stirring bar and 1-(furan-2-yl)butane-1,3-dione (**1d**, 20 mg, 0.13 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.2 mg, 0.01 mmol, 0.1 equiv) in 2% ethanol/CH2Cl2 (1.3 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5-10% EtOAc–hexane to afford **2da** (R_f = 0.33 in 15% EtOAc–hexane; 18.3 mg, 84% yield) as a pale-yellow liquid.

Selected data for **2da**: IR (neat): 2920, 2850, 1712, 1676, 1462, 1388, 1278, 1229, 1156, 1047, 1021, 906, 775, 593 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.60 (dd, J = 1.5, 0.5 Hz, 1 H), 7.26 – 7.25 (m, 2 H), 6.45 (dd, J = 3.6, 1.5 Hz, 1 H), 6.28 (dd, J = 3.4, 1.8 Hz, 1 H), 6.26 (dd, J = 3.4, 0.9 Hz, 1 H), 4.64 (dd, J = 10.7, 7.5 Hz, 1 H), 3.31 (dq, J = 8.8, 7.0 Hz, 1 H), 3.09 (dq, J = 8.8, 7.0 Hz, 1 H), 2.49 (dd, J = 12.5, 10.7 Hz, 1 H), 2.07 (dd, J = 12.5, 7.5 Hz, 1 H), 2.05 (s, 3 H), 1.60 (s, 3 H), 0.75 (t, J = 7.0 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.2 (C), 184.9 (C), 151.1 (C), 149.9 (C), 147.1 (CH), 142.9 (CH), 121.9 (CH), 112.0 (CH), 110.7 (CH), 108.5 (CH), 108.4 (C), 88.3 (C), 57.5 (CH_2), 55.2 (CH), 40.3 (CH_2), 30.3 (CH_3), 21.2 (CH_3), 14.6 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{18}H_{20}O_6$ 332.1260; found 332.1255.

Preparation of 2db:

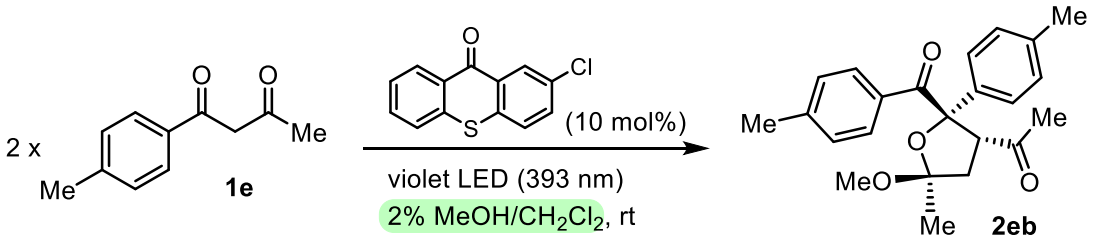


A magnetic stirring bar and 1-(furan-2-yl)butane-1,3-dione (**1d**, 20 mg, 0.13 mmol)[1] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9H-thioxanthen-9-one (3.2 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.3 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for

40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5-10% EtOAc–hexane to afford **2db** (R_f = 0.29 in 15% EtOAc–hexane; 18.2 mg, 87% yield) as a yellow liquid.

Selected data for **2db**: IR (neat): 3139, 2990, 2949, 2836, 1712, 1677, 1462, 1385, 1231, 1176, 1154, 1081, 1023, 861, 779 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.60 (dd, J = 1.7, 0.8 Hz, 1 H), 7.27 – 7.23 (m, 2 H), 6.45 (dd, J = 3.4, 1.7 Hz, 1 H), 6.28 (dd, J = 3.4, 1.8 Hz, 1 H), 6.25 (dd, J = 3.4, 0.8 Hz, 1 H), 4.59 (dd, J = 10.7, 7.5 Hz, 1 H), 2.90 (s, 3 H), 2.51 (dd, J = 12.8, 10.7 Hz, 1 H), 2.06 (dd, J = 12.8, 7.5 Hz, 1 H), 2.05 (s, 3H), 1.59 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.0 (C), 184.6 (C), 151.0 (C), 149.9 (C), 147.2 (CH), 143.0 (CH), 121.9 (CH), 112.0 (CH), 110.7 (CH), 108.6 (C), 108.5 (CH), 88.4 (C), 55.0 (CH), 49.4 (CH_3), 40.0 (CH_2), 30.3 (CH_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{17}H_{18}O_6$ 318.1103; found 318.1100.

Preparation of 2eb:



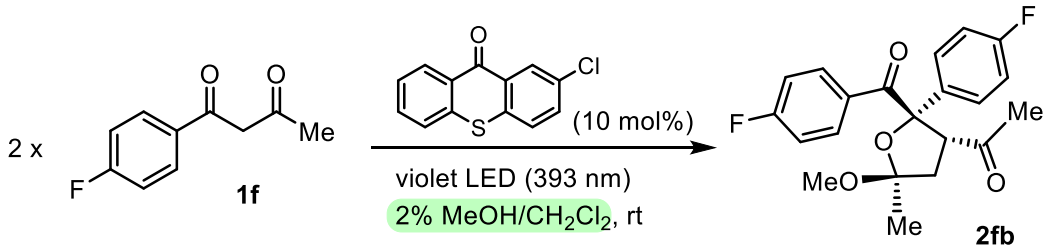
A magnetic stirring bar and 1-(*p*-tolyl)butane-1,3-dione (**1e**, 20 mg, 0.11 mmol)[3] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.8 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.1 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2eb** (R_f = 0.39 in 15% EtOAc–hexane; 15.6 mg, 75% yield) as a colorless liquid.

Selected data for **2eb**: IR (neat): 3031, 2990, 2953, 2831, 1712, 1680, 1606, 1449, 1355, 1317, 1229, 1181, 1108, 1033, 875, 817, 775 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.77 (d, J = 8.0 Hz, 2 H), 7.13 (d, J = 8.0 Hz, 2 H), 7.06 (d, J = 8.0 Hz, 4 H), 4.71 (t, J = 8.0 Hz, 1 H), 2.60 (s, 3 H), 2.49 (dd, J = 13.0, 8.0 Hz, 1 H), 2.29 (s, 3 H), 2.26 (s, 3

[3] Compound **1e** was prepared according to literature procedures: (a) Berti, F.; Bincoletto, S.; Donati, I.; Fontanive, G.; Fregonese, M.; Benedetti, F. *Org. Biomol. Chen.* **2011**, *9*, 1987 – 1999. (b) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244.

H), 2.02 (dd, J = 13.0, 8.0 Hz, 1 H), 1.78 (s, 3 H), 1.63 (s, 3 H);^{13}C NMR (125 MHz, $CDCl_3$): δ 207.8 (C), 198.5 (C), 143.1 (C), 138.0 (C), 134.9 (C), 131.9 (C), 131.1 (two CH), 129.3 (two CH), 128.5 (two CH), 125.4 (two CH), 108.8 (C), 93.6 (C), 57.1 (CH), 49.6 (CH_3), 40.8 (CH_2), 31.2 (CH_3), 21.6 (CH_3), 21.1 (CH_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{23}H_{26}O_4$ 366.1831; found 366.1823.

Preparation of 2fb:



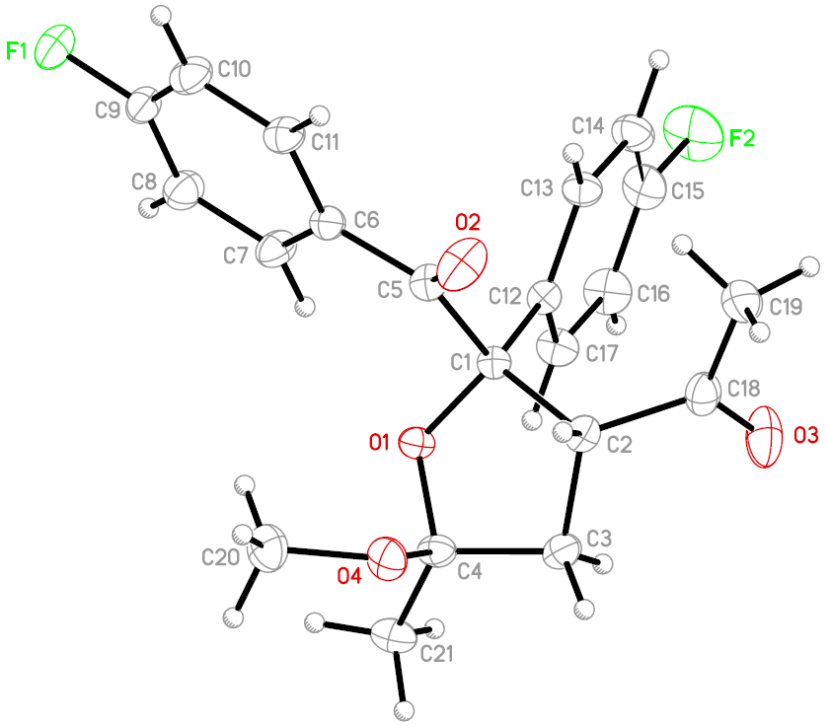
A magnetic stirring bar and 1-(4-fluorophenyl)butane-1,3-dione (**1f**, 20 mg, 0.11 mmol)[4] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9H-thioxanthen-9-one (2.7 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.1 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at room temperature for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2fb** (R_f = 0.34 in 15% EtOAc–hexane; 16.7 mg, 80% yield) as a yellow solid.

Selected data for **2fb**: m.p: 123-125 °C; IR (neat): 3076, 2993, 2957, 2833, 1714, 1684, 1599, 1507, 1358, 1232, 1160, 1106, 1035, 877, 838, 785 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.91 – 7.86 (m, 2 H), 7.27 – 7.21 (m, 2 H), 7.01 – 6.92 (m, 4 H), 4.70 (t, J = 8.0 Hz, 1 H), 2.60 (s, 3 H), 2.46 (dd, J = 13.1, 8.0 Hz, 1 H), 2.04 (dd, J = 13.1, 8.0 Hz, 1 H), 1.79 (s, 3 H), 1.63 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.4 (C), 197.0 (C), 165.1 (d, J = 255.3 Hz, C), 162.6 (d, J = 248.5 Hz, C), 133.6 (d, J = 9.2 Hz, two CH), 133.3 (d, J = 3.3 Hz, C), 130.5 (d, J = 3.0 Hz, C), 127.3 (d, J = 8.2 Hz, two CH), 115.8 (d, J = 21.6 Hz, two CH), 115.1 (d, J = 21.9 Hz, two CH), 109.3 (C), 93.1 (C), 57.0 (CH), 49.7 (CH_3), 41.0 (CH_2), 31.3 (CH_3), 20.5 (CH_3); ^{19}F NMR (470 MHz, $CDCl_3$): δ -105.1 – -105.2 (m), -112.9 – -113.0 (m); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{20}F_2O_4$ 374.1330; found 374.1327.

[4] Compound **1f** was prepared according to literature procedures: (a) Sun, X.; Lyu, Y.; Zhang-Negrerie, D.; Du, Y.; Zhao, K. *Org. Lett.* **2013**, *15*, 6222 – 6225. (b) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244.

Recrystallization of 2fb was performed as follows:

The compound (**2fb**, ~5 mg) in a screw-capped vial (4 mL vial) was dissolved in CH_2Cl_2 (~0.5 mL) and diluted with *n*-hexane (~2.5 mL). The vial was covered with aluminum foil (having 4-5 holes on it) and placed in another vial (20 mL vial) filled with *n*-hexane (~8 mL). The 20 mL vial was closed gently with a screw cap and stands it for 3 days until complete evaporation of the solvent in the inner vial. The crystals formed were subjected to single-crystal X-ray analysis.



Thermal ellipsoids draw at the 30% probability level

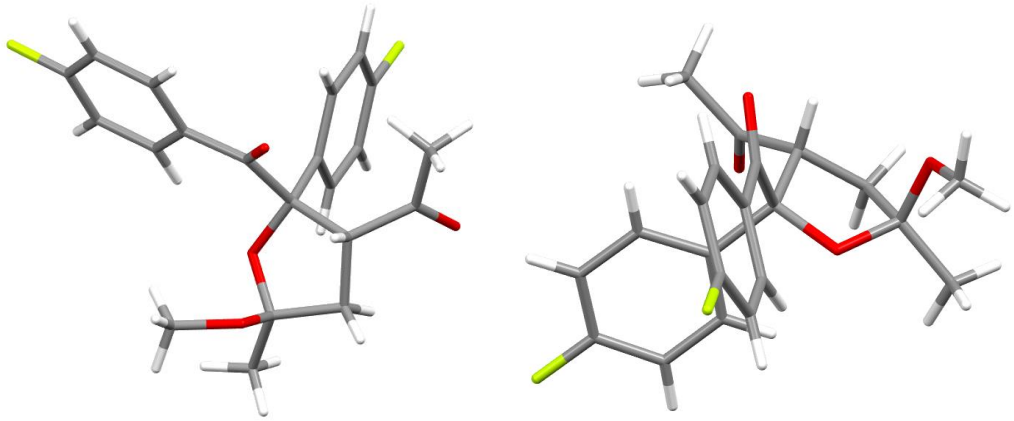


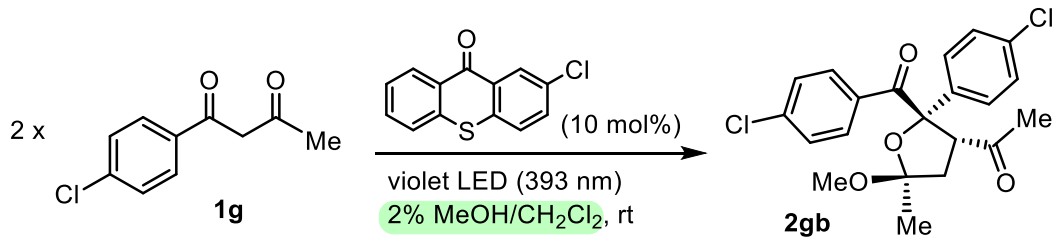
Figure S3. ORTEP and Stereo plots for X-ray crystal structures of **2fb (**ic22557).

CCDC 2382211 contains the supplementary crystallographic data for **2fb (**ic22557). These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk

Table S4. Crystal data and structure refinement for **2fb (**ic22557).

Identification code	ic22557	
Empirical formula	C21 H20 F2 O4	
Formula weight	374.37	
Temperature	200(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 17.2464(4) Å	= 90°.
	b = 9.1025(2) Å	= 109.7066(8)°.
	c = 25.1237(7) Å	= 90°.
Volume	3713.06(16) $Å^3$	
Z	8	
Density (calculated)	1.339 Mg/m^3	
Absorption coefficient	0.886 mm^{-1}	
F(000)	1568	
Crystal size	0.258 x 0.188 x 0.154 mm^3	
Theta range for data collection	3.737 to 74.599°.	
Index ranges	-21<=h<=21, -11<=k<=11, -31<=l<=31	
Reflections collected	53373	
Independent reflections	3790 [R(int) = 0.0301]	
Completeness to theta = 67.679°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9819 and 0.6840	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3790 / 0 / 247	
Goodness-of-fit on F^2	1.025	
Final R indices [I>2sigma(I)]	R1 = 0.0386, wR2 = 0.1039	
R indices (all data)	R1 = 0.0396, wR2 = 0.1050	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.231 and -0.222 $e.Å^{-3}$	

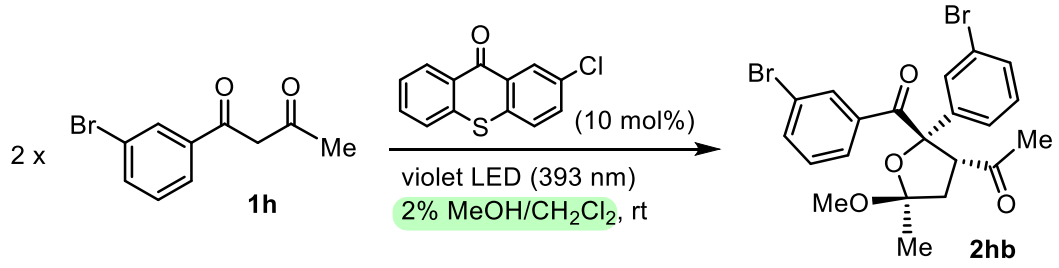
Preparation of 2gb:



A magnetic stirring bar and 1-(4-chlorophenyl)butane-1,3-dione (**1g**, 20 mg, 0.1 mmol)[5] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.8 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.0 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2gb** (R_f = 0.42 in 20% EtOAc–hexane; 16.2 mg, 78% yield) as a white solid.

Selected data for **2gb**: m.p: 151-152 °C; IR (neat): 3069, 2991, 2959, 2833, 1714, 1685, 1588, 1489, 1357, 1228, 1177, 1096, 1037, 872, 814, 749 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.81 – 7.77 (m, 2 H), 7.28 – 7.24 (m, 4 H), 7.21 – 7.17 (m, 2 H), 4.69 (t, J = 8.0 Hz, 1 H), 2.61 (s, 3 H), 2.45 (dd, J = 13.0, 8.0 Hz, 1 H), 2.05 (dd, J = 13.0, 8.0 Hz, 1 H), 1.81 (s, 3 H), 1.63 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.2 (C), 197.2 (C), 139.1 (C), 136.0 (C), 134.6 (C), 132.4 (C), 132.3 (two CH), 129.0 (two CH), 128.3 (two CH), 126.9 (two CH), 109.4 (C), 93.0 (C), 57.0 (CH), 49.8 (CH_3), 41.0 (CH_2), 31.4 (CH_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{20}O_4Cl_2$ 406.0739; found 406.0732.

Preparation of 2hb:

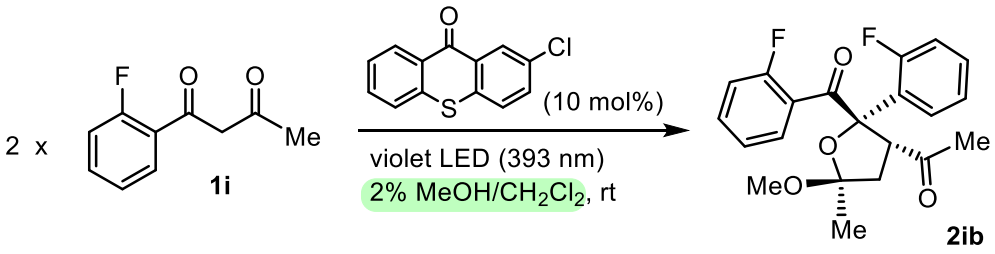


[5] Compound **1g** was prepared according to literature procedures: (a) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244. (b) Sun, Xi; Li, Pinhua; Zhang, Xiuli; Wang, Lei *Org. Lett.* **2014**, *16*, 2126 – 2129.

A magnetic stirring bar and 1-(3-bromophenyl)butane-1,3-dione (**1h**, 20 mg, 0.08 mmol)[6] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.0 mg, 0.008 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (0.8 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2hb** (R_f = 0.43 in 20% EtOAc–hexane; 14.4 mg, 70% yield) as a pale-yellow liquid.

Selected data for **2hb**: IR (neat): 3068, 2991, 2957, 2833, 1712, 1688, 1565, 1469, 1416, 1357, 1319, 1225, 1176, 1105, 1038, 863, 789, 707 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 8.05 (dd, J = 1.7, 1.7 Hz, 1 H), 7.71 – 7.68 (m, 1 H), 7.56 – 7.52 (m, 1 H), 7.45 (s, 1 H), 7.42 – 7.38 (m, 1 H), 7.19 – 7.12 (m, 3 H), 4.66 (t, J = 7.8 Hz, 1 H), 2.61 (s, 3 H), 2.44 (dd, J = 13.1, 7.7 Hz, 1 H), 2.04 (dd, J = 13.1, 8.0 Hz, 1 H), 1.82 (s, 3 H), 1.65 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.1 (C), 196.9 (C), 139.4 (C), 135.8 (C), 135.5 (CH), 133.4 (CH), 131.8 (CH), 130.3 (CH), 129.6 (CH), 129.5 (CH), 128.4 (CH), 123.9 (CH), 123.2 (C), 122.3 (C), 109.6 (C), 92.9 (C), 57.2 (CH), 49.8 (CH_3), 41.1 (CH_2), 31.4 (CH_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{20}O_4Br_2$ 493.9728; found 493.9724.

Preparation of 2ib:



A magnetic stirring bar and 1-(2-fluorophenyl)butane-1,3-dione (**1i**, 20 mg, 0.11 mmol)[7] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.7 mg, 0.011 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.1 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 48 h until the

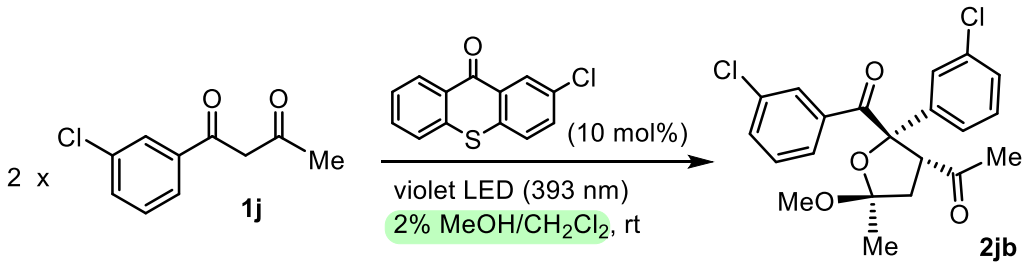
[6] Compound **1h** was prepared according to literature procedures: (a) Palani, P.; Arumugam, A.; Raja, D.; Muthu, K.; Senadi, G. C. *Chem. Commun.* **2023**, *59*, 11433 – 11436. (b) Rajasekar, S.; Anbarasan, P. *Chem. Asian J.* **2019**, *14*, 4563 – 4567.

[7] Compound **1i** was prepared according to literature procedures: (a) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244. (b) Marichev, K. O.; Wang, Y.; Carranco, A. M.; Garcia, E. C.; Yu, Z.-X.; Doyle, M. P. *Chem. Commun.* **2018**, *54*, 9513 – 9516.

completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ib** (R_f = 0.34 in 20% EtOAc–hexane; 13.7 mg, 66% yield) as a yellow liquid.

Selected data for **2ib**: IR (neat): 3078, 2994, 2955, 2833, 1708, 1609, 1486, 1450, 1227, 1105, 1030, 867, 761 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.68 (ddd, J = 7.7, 7.7, 1.8 Hz, 1 H), 7.48 (ddd, J = 7.9, 7.9, 1.8 Hz, 1 H), 7.39 – 7.33 (m, 1 H), 7.27 – 7.21 (m, 1 H), 7.15 (ddd, J = 7.6, 7.6, 1.2 Hz, 1 H), 7.04 (ddd, J = 11.2, 8.3, 1.2 Hz, 1 H), 6.98 – 6.94 (m, 1 H), 6.88 (ddd, J = 11.5, 8.3, 1.2 Hz, 1 H), 4.88 (dd, J = 9.2, 7.7 Hz, 1 H), 2.81 (s, 3 H), 2.47 (dd, J = 12.9, 9.2 Hz, 1 H), 2.14 (dd, J = 12.9, 7.7 Hz, 1 H), 1.98 (d, J = 1.1 Hz, 3 H), 1.64 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.1 (C), 194.3 (d, J = 2.8 Hz, C), 161.8 (d, J = 261.1 Hz, C), 159.0 (d, J = 247.4 Hz, C), 133.8 (d, J = 9.2 Hz, CH), 131.7 (CH), 130.4 (d, J = 8.5 Hz, CH), 128.3 (d, J = 3.5 Hz, CH), 125.9 (d, J = 11.6 Hz, C), 124.5 (d, J = 3.3 Hz, CH), 123.9 (d, J = 9.0 Hz, C), 123.2 (d, J = 4.0 Hz, CH), 116.9 (d, J = 22.8 Hz, CH), 115.9 (d, J = 22.1 Hz, CH), 109.2 (C), 91.31 (d, J = 2.2 Hz, C), 56.1 (CH), 49.3 (CH_3), 41.1 (CH_2), 30.9 (d, J = 2.8 Hz, CH_3), 20.6 (CH_3); ^{19}F NMR (470 MHz, $CDCl_3$): δ -109.33 – -109.41 (m), -109.81 – -109.89 (m); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{20}F_2O_4$ 374.1330; found 374.1323.

Preparation of 2jb:



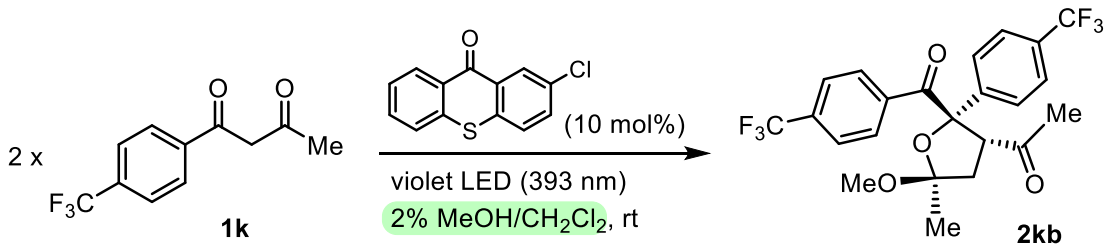
A magnetic stirring bar and 1-(3-chlorophenyl)butane-1,3-dione (**1j**, 20 mg, 0.1 mmol)[8] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.5 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.0 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2jb** (R_f = 0.40 in 20%

[8] Compound **1j** was prepared according to literature procedures: (a) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244. (b) He, J.-P.; Huang, G.-S.; Luo, N.; Zhan, Z.-Z.; Zhang, M.-M. *Org. Biomol. Chem.* **2020**, *18*, 9831 – 9835.

EtOAc–hexane; 15.3 mg, 74% yield) as a pale-yellow liquid.

Selected data for **2jb**: IR (neat): 3071, 2993, 2956, 2833, 1712, 1688, 1569, 1419, 1357, 1225, 1176, 1105, 1037, 863, 789, 717 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.88 (dd, J = 2.0, 2.0 Hz, 1 H), 7.69 – 7.66 (m, 1 H), 7.39 (ddd, J = 7.9, 2.0, 1.0 Hz, 1 H), 7.30 (brs, 1 H), 7.27 – 7.19 (m, 3 H), 7.12 (brd, J = 7.1 Hz, 1 H), 4.67 (t, J = 8.0 Hz, 1 H), 2.61 (s, 3 H), 2.44 (dd, J = 13.1, 8.0 Hz, 1 H), 2.05 (dd, J = 13.1, 8.0 Hz, 1 H), 1.82 (s, 3 H), 1.66 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.1 (C), 197.0 (C), 139.2 (C), 135.6 (C), 135.1 (C), 134.3 (C), 132.6 (CH), 130.5 (CH), 130.0 (CH), 129.24 (CH), 129.15 (CH), 128.9 (CH), 125.6 (CH), 123.5 (CH), 109.6 (C), 93.0 (C), 57.2 (CH), 49.8 (CH_3), 41.1 (CH_2), 31.5 (CH_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M-H]^+$ Calcd for $C_{21}H_{19}O_4Cl_2$ 405.0660; found 405.0655.

Preparation of 2kb:



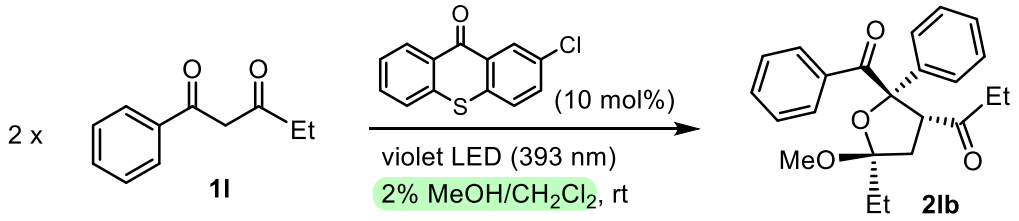
A magnetic stirring bar and 1-(4-(trifluoromethyl)phenyl)butane-1,3-dione (**1k**, 20 mg, 0.09 mmol)[9] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.7 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (0.9 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2kb** (R_f = 0.38 in 15% EtOAc–hexane; 16.3 mg, 79% yield) as a yellow liquid.

Selected data for **2kb**: IR (neat): 3075, 2994, 2957, 2837, 1714, 1694, 1411, 1326, 1229, 1171, 1129, 1069, 1016, 846 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.93 (d, J = 8.2 Hz, 2 H), 7.59 – 7.54 (m, 4 H), 7.42 (d, J = 7.9 Hz, 2 H), 4.73 (t, J = 8.0 Hz, 1 H), 2.58 (s, 3 H), 2.47 (dd, J = 13.1, 8.0 Hz, 1 H), 2.10 (dd, J = 13.1, 8.0 Hz, 1 H), 1.83 (s, 3 H), 1.66 (s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 206.9 (C), 197.3 (C), 141.1 (C), 136.8 (C), 134.0 (q, J = 32.7 Hz, two CH), 131.1 (two CH), 130.9 (q, J = 32.9 Hz, two

[9] Compound **1k** was prepared according to literature procedures: (a) An, Z.; Liu, Y.; Yan, R.; Zhao, P. *Adv. Synth. Catal.* **2021**, *363*, 3240 – 3244. (b) He, J.-P.; Huang, G.-S.; Luo, N.; Zhan, Z.-Z.; Zhang, M.-M. *Org. Biomol. Chem.* **2020**, *18*, 9831 – 9835.

CH), 125.88 (two CH), 125.87 (q, J = 3.7 Hz, C), 125.1 (q, *J* = 3.7 Hz, C), 123.7 (q, *J* = 272.4 Hz, C), 123.5 (q, *J* = 272.7 Hz, C), 109.8 (C), 93.2 (C), 57.2 (CH), 49.8 (CH_3), 41.2 (CH_2), 31.5 (CH_3), 20.4 (CH_3); ^{19}F NMR (470 MHz, $CDCl_3$): δ -62.79 (s), -63.32 (s); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{23}H_{20}F_6O_4$ 474.12603; found 474.1262.

Preparation of 2lb:

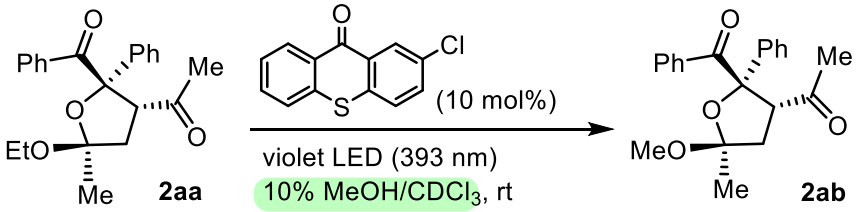


A magnetic stirring bar and 1-phenylpentane-1,3-dione (**1l**, 20 mg, 0.11 mmol)[10] were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (2.8 mg, 0.01 mmol, 0.1 equiv) in 2% methanol/CH_2Cl_2 (1.1 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2lb** (R_f = 0.44 in 20% EtOAc–hexane; 15.2 mg, 73% yield) as a yellow solid.

Selected data for **2lb**: mp: 70-72 °C; IR (neat): 3064, 2974, 2942, 2884, 1714, 1684, 1593, 1452, 1347, 1245, 1181, 1031, 878, 754, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.86 – 7.83 (m, 2 H), 7.40 – 7.36 (m, 1 H), 7.29 – 7.21 (m, 7 H), 4.72 (t, *J* = 8.1 Hz, 1 H), 2.52 (s, 3 H), 2.49 (dd, *J* = 13.0, 8.0 Hz, 1 H), 2.23 – 2.16 (m, 2 H), 2.11 – 2.04 (m, 1 H), 2.03 – 1.93 (m, 2 H), 0.95 (t, *J* = 7.5 Hz, 3 H), 0.55 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 210.3 (C), 198.9 (C), 137.9 (C), 134.4 (C), 132.4 (CH), 131.0 (two CH), 128.6 (two CH), 128.3 (CH), 127.8 (two CH), 125.5 (two CH), 111.8 (C), 93.6 (C), 55.5 (CH), 49.1 (CH_3), 38.3 (CH_2), 37.6 (CH_2), 26.1 (CH_2), 8.7 (CH_3), 7.1 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{23}H_{26}O_4$ 366.1831; found 366.1826.

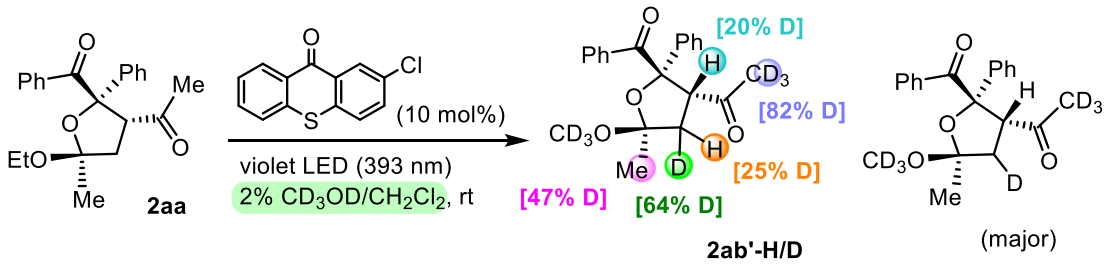
[10] Compound **1l** was prepared according to literature procedures: (a) Geng, H.; Zhou, L.; Wu, W.; Zhang, W.; Chen, J.; Hou, G.; Zou, Y.; Zhang, X. *Angew. Chem. Int. Ed.* **2009**, *48*, 6052 – 6054. (b) Geng, H.; Huang, K.; Sun, T.; Li, W.; Zhang, X.; Zhou, L.; Wu, W.; Zhang, X. *J. Org. Chem.* **2011**, *76*, 332 – 334.

Preparation of 2ab from 2aa:



A magnetic stirring bar and **2aa** (20 mg, 0.06 mmol) were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (1.4 mg, 0.006 mmol, 0.1 equiv) in 10% methanol/$CDCl_3$ (0.6 mL). The vial was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2x10 W) at r.t. for 48 h until the completion of the reaction, monitored by TLC as well as crude ^{1}H-NMR. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ab** (R_f = 0.37 in 15% EtOAc–hexane; 18 mg, 94% yield) as a yellow solid.

Preparation of 2ab'-H/D from 2aa:

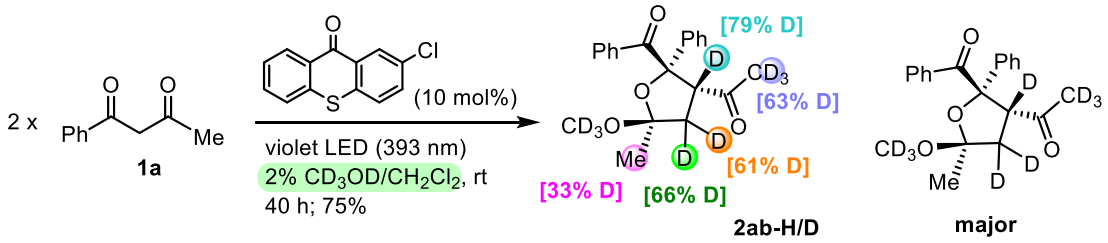


A magnetic stirring bar and **2aa** (10 mg, 0.03 mmol) were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (0.7 mg, 0.003 mmol, 0.1 equiv) in 2% CD_3OD/CH_2Cl_2 (0.4 mL). The solution was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ab'-H/D** (R_f = 0.36 in 15% EtOAc–hexane; 8.7 mg, 88% yield) as a yellow solid.

Selected data for **2ab'-H/D**: m.p.: 127-128 °C; IR (neat): 3065, 2915, 2848, 2224, 2069, 1691, 1446, 1327, 1239, 1173, 1024, 754, 697 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 – 7.82 (m, 2 H), 7.41 – 7.37 (m, 1 H), 7.31 – 7.24 (m, 7 H), 4.74 – 4.70 (m, 0.8

H), 2.53 – 2.46 (m, 0.75 H), 2.07 – 2.00 (m, 0.35 H), 1.64 (d, J = 0.8 Hz, 1.53 H); For major isomer: ^{1}H NMR (500 MHz, Chloroform-*d*): δ 7.87 – 7.82 (m, 2 H), 7.41 – 7.36 (m, 1 H), 7.31 – 7.24 (m, 7 H), 4.71 (d, J = 6.8 Hz, 1 H), 2.07 – 2.01 (m, 1 H), 1.64 (s, 3 H); ^{13}C NMR (126 MHz, $CDCl_3$): δ 207.9-207.8 (m, C), 198.9 (C), 137.6 (C), 134.4 (C), 132.4 (CH), 131.0 (two CH), 128.7 (two CH), 128.4 (CH), 127.8 (two CH), 125.5 (two CH), 109.1 (C), 93.7 (C), 57.3 (CH), 49.1-48.5 (m, CD_3), 40.7 (t, J = 20 Hz, CDH), 31.29 – 30.29 (m, CD_3), 20.6 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{15}O_4D_7$ 345.1957; found 345.1949.

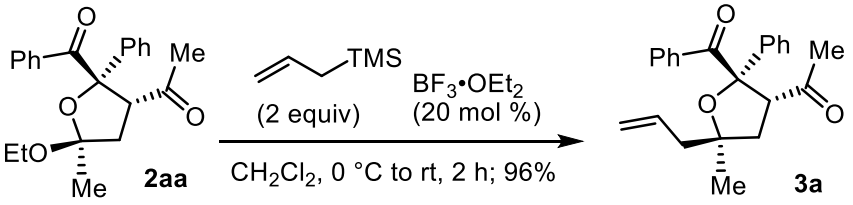
Preparation of 2ab-H/D:



A magnetic stirring bar and 1-phenylbutane-1,3-dione (1a, 20 mg, 0.12 mmol) were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.012 mmol, 0.1 equiv) in 2% CD_3OD/CH_2Cl_2 (1.2 mL). The vial was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 40 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2ab-H/D** (R_f = 0.36 in 15% EtOAc–hexane; 16.1 mg, 75% yield) as a yellow solid.

Selected data for **2ab-H/D**: m.p.: 128-129 °C; IR (neat): 3064, 2990, 2933, 2221, 2071, 1682, 1592, 1446, 1244, 1180, 1093, 1025, 751, 700 cm^{-1}; ^{1}H NMR (500 MHz, $CDCl_3$): δ 7.87 – 7.82 (m, 2 H), 7.41 – 7.37 (m, 1 H), 7.31 – 7.24 (m, 7 H), 4.74 – 4.70 (m, 0.21 H), 2.53 – 2.46 (m, 0.39 H), 2.07 – 2.00 (m, 0.34 H), 1.77 – 1.75 (d, J = 2.9 Hz, 0.26 H), 1.64 (d, J = 0.8 Hz, 2.02 H); For major isomer: ^{1}H NMR (500 MHz, Chloroform-*d*): δ 7.87 – 7.82 (m, 2 H), 7.41 – 7.36 (m, 1 H), 7.31 – 7.24 (m, 7 H), 1.64 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 208.0 – 207.6 (m, C), 198.9 (C), 137.6 (C), 134.4 (C), 132.4 (CH), 131.0 (two CH), 128.7 (two CH), 128.4 (CH), 127.8 (two CH), 125.5 (two CH), 109.1 (C), 93.6 (d, J = 6 Hz, C), 57.3 – 57.1 (m, CD), 49.3 – 48.2 (m, CD_3), 41.14 – 40.28 (m, CD_2), 31.3 – 30.3 (m, CD_3), 20.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{13}O_4D_9$ 347.2083; found 347.2080.

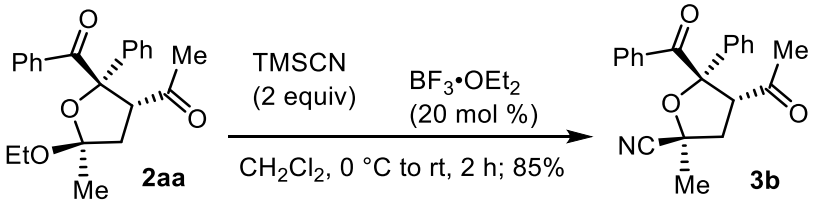
Preparation of 3a from 2aa:



A solution of **2aa** (20 mg, 0.06 mmol) in CH_2Cl_2 (1.1 mL) was cooled to 0°C. Allyl trimethylsilane (13 mg, 0.11 mmol, 2.0 equiv) and boron trifluoride etherate (1.6 mg, 0.01 mmol, 0.2 equiv) were then added sequentially. The reaction mixture was stirred at room temperature for 2 h. After completion, the reaction was quenched with saturated aqueous $NaHCO_3$ solution and extracted with CH_2Cl_2 (2×5 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo* to give a residue. The crude product was purified by flash column chromatography with 5% EtOAc–hexane as eluent to afford **3a** (R_f = 0.38 in 15% EtOAc–hexane; 18.9 mg, 96%) as a pale-yellow oil.

Selected data for **3a**: IR (neat): 3066, 2974, 2932, 1712, 1678, 1593, 1446, 1357, 1242, 1178, 1042, 918, 755, 701 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.92 – 7.88 (m, 2 H), 7.42 – 7.37 (m, 1 H), 7.31 – 7.19 (m, 7 H), 5.41 – 5.32 (m, 1 H), 4.85 – 4.74 (m, 2 H), 4.64 (t, J = 8.0 Hz, 1 H), 2.35 (dd, J = 13.0, 8.0 Hz, 1 H), 2.07 – 2.00 (m, 1 H), 1.98 – 1.90 (m, 2 H), 1.79 (s, 3 H), 1.53 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 207.3 (C), 199.8 (C), 138.8 (C), 134.2 (C), 133.8 (CH), 132.7 (CH), 131.4 (two CH), 128.6 (two CH), 128.2 (CH), 127.9 (two CH), 125.5 (two CH), 117.9 (CH_2), 94.0 (C), 85.6 (C), 58.0 (CH), 45.7 (CH_2), 39.0 (CH_2), 31.2 (CH_3), 25.6 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{23}H_{24}O_3$ 348.1725; found 348.1723.

Preparation of 3b from 2aa:

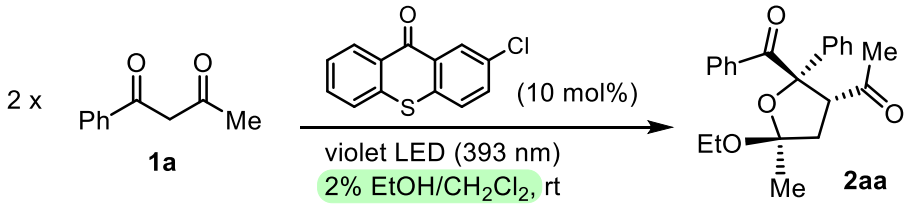


A solution of **2aa** (20 mg, 0.06 mmol) in CH_2Cl_2 (1.1 mL) was cooled to 0°C. trimethylsilyl cyanide (11.3 mg, 0.11 mmol, 2.0 equiv) and boron trifluoride etherate (1.6 mg, 0.01 mmol, 0.2 equiv) were then added sequentially. The reaction mixture was stirred at room temperature for 2 h. After completion, the reaction was quenched with saturated aqueous $NaHCO_3$ solution and extracted with CH_2Cl_2 (2×5 mL). The combined organic layers were dried over Na_2SO_4, and concentrated *in vacuo* to give a

residue. The crude product was purified by flash column chromatography with 5-10% EtOAc–hexane as eluent to afford **3b** (R_f = 0.32 in 15% EtOAc–hexane; 16.1 mg, 85% yield) as a pale-yellow oil.

Selected data for **3b**: IR (neat): 3065, 2990, 2928, 2857, 1714, 1681, 1593, 1446, 1360, 1242, 1178, 1112, 1033, 898, 754, 704 cm^{-1}; ^1H NMR (500 MHz, $CDCl_3$): δ 7.88 – 7.84 (m, 2 H), 7.47 – 7.42 (m, 1 H), 7.34 – 7.23 (m, 7 H), 4.76 (t, J = 7.5 Hz, 1 H), 2.62 (dd, J = 13.4, 7.5 Hz, 1 H), 2.46 (dd, J = 13.4, 7.5 Hz, 1 H), 1.91 (s, 3 H), 1.78 (s, 3 H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 206.0 (C), 197.5 (C), 136.4 (C), 133.6 (C), 133.3 (CH), 131.2 (two CH), 129.01 (CH), 128.95 (two CH), 128.1 (two CH), 125.3 (two CH), 120.3 (C), 95.5 (C), 75.9 (C), 57.1 (CH), 40.6 (CH_2), 31.1 (CH_3), 25.5 (CH_3); HRMS (GC-EI-TOF) *m/z*: $[M]^+$ Calcd for $C_{21}H_{19}NO_3$ 333.1365; found 333.1360.

Preparation of 2aa (1.0 mmol scale):

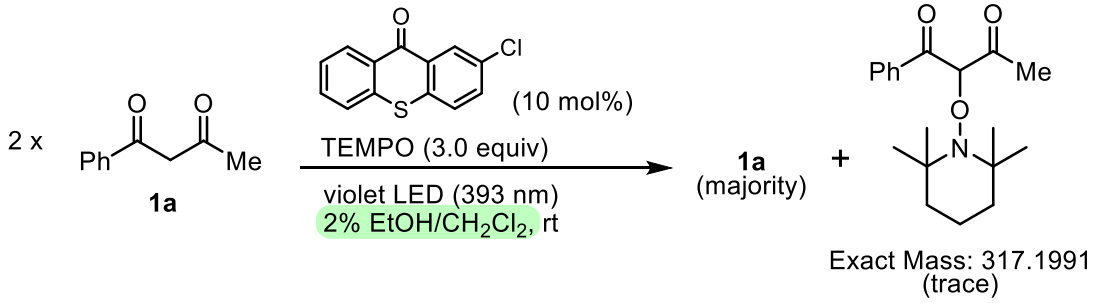


A magnetic stirring bar and 1-phenylbutane-1,3-dione (**1a**, 162.2 mg, 1.0 mmol) were placed in a 12-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (24.7 mg, 0.1 mmol, 0.1 equiv) in 2% ethanol/CH_2Cl_2 (10 mL). The vial was purged with argon and closed with a screw cap. The solution was stirred and irradiated with the violet LEDs (393 nm, 2 x 10 W) at rt for 48 h until the completion of the reaction, monitored by TLC. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. The crude product was purified by column chromatography with 5% EtOAc–hexane to afford **2aa** (R_f = 0.44 in 20% EtOAc–hexane; 126.8 mg, 72% yield) as a yellow solid.

The light on/off experiment:

A 7-mL sample vial was charged with a magnetic stirring bar and compound **1a** (100 mg, 0.62 mmol), followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (**I**, 15.2 mg, 0.06 mmol, 0.1 equiv) and 2% CH_3OH/CH_2Cl_2 (6.2 mL). The vial was purged with argon and sealed with a screw cap. The resulting solution was subjected to stirring and irradiated with violet LEDs (393 nm, 2 x 10 W) at room temperature. After the specified reaction time, an aliquot (~50 μL) of the reaction mixture was withdrawn, diluted with $CDCl_3$, and analyzed by ^1H NMR, with 1,3,5-trimethylbenzene employed as an internal standard.

Control experiment (radical trapping reaction with TEMPO):



A magnetic stirring bar and **1a** (20 mg, 0.12 mmol) were placed in a 4-mL sample vial, followed by the addition of 2-chloro-9*H*-thioxanthen-9-one (3.0 mg, 0.01 mmol, 0.1 equiv) and TEMPO (57.8 mg, 0.37 mmol, 3.0 equiv) in 2% EtOH/CH_2Cl_2 (1.2 mL). The vial was purged with argon and closed with a screw cap. The solution was stirred and irradiated with a violet LED (393 nm, 2 x 10 W) at rt for 48 h. The reaction was monitored by TLC, showing that approximately 80-90% of the starting compound **1a** remained unreacted. No detectable production of **2aa** was observed, and the reaction appeared to be inhibited, with the majority of **1a** remaining unreacted. After the irradiation, the reaction solution was concentrated *in vacuo* to give a residue. HRMS (GC-EI-TOF) analysis of the crude reaction mixture revealed an ion at *m*/*z* $[M]^+$ 317.1984, consistent with the molecular formula $C_{19}H_{27}NO_3$, which gives a calculated value of *m*/*z* 317.1991. The molecular weight of **1a** ($C_{10}H_{10}O_2$) minus one hydrogen atom plus TEMPO, *i.e.,* **1a**-TEMPO with the loss of a hydrogen atom, corresponds to this mass-to-charge ratio.

Figure S4. Light-on/light-off intermittent experiment of **1a**

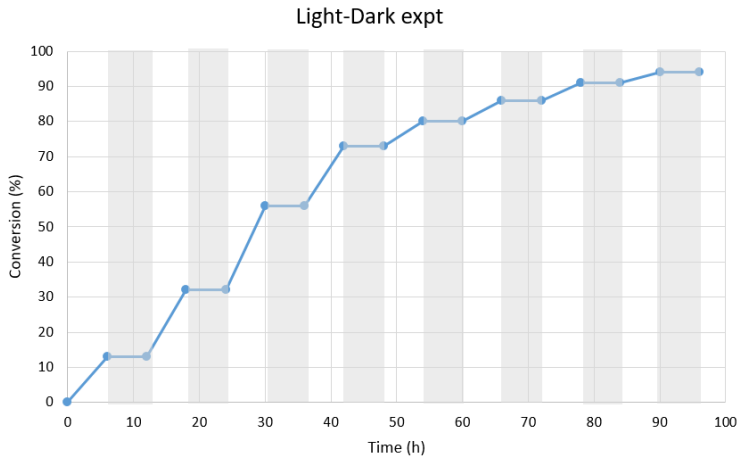


Figure S5. Reaction Progress:

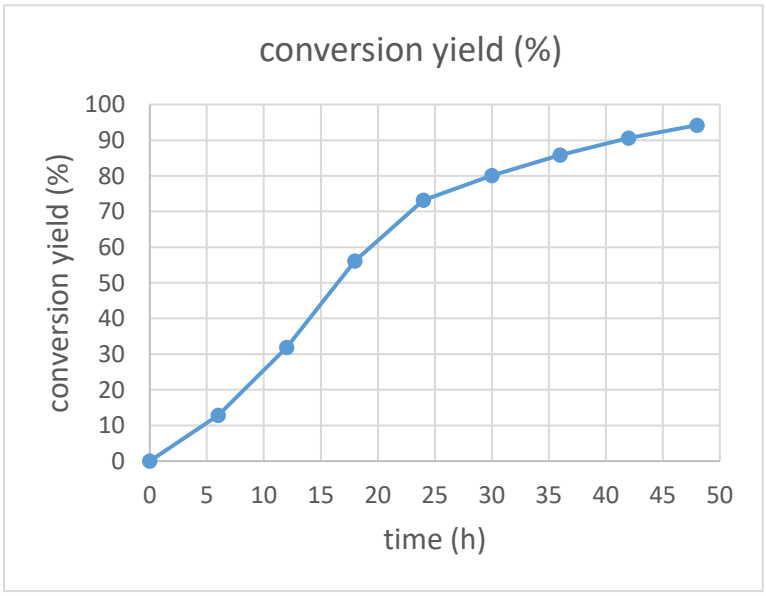


Figure S6. UV-Vis spectra of **1a** (**IRR-140**) in CH_2Cl_2.

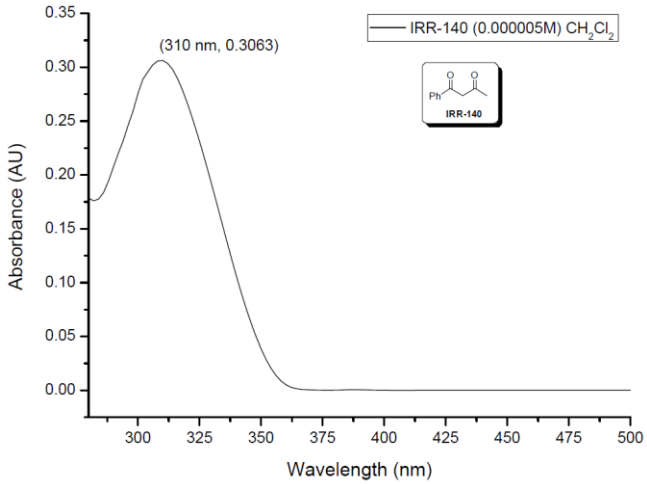


Figure S7. Emission spectra of **1a** (**IRR-140**) in CH_2Cl_2 (irradiated at 390 nm).

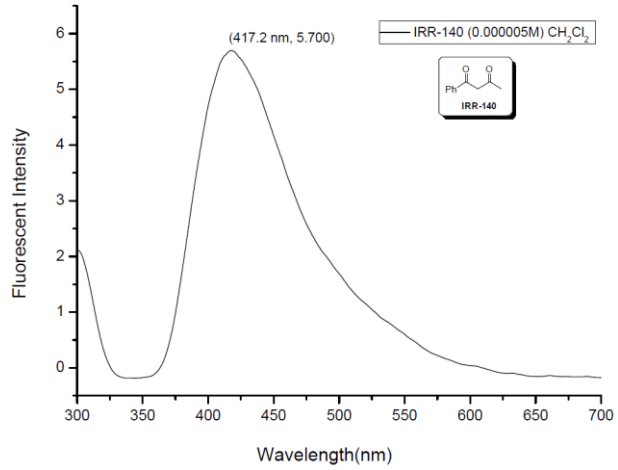


Figure S8. Emission spectra of **I** (**2-CITX**) in CH_2Cl_2 (irradiated at 390 nm).

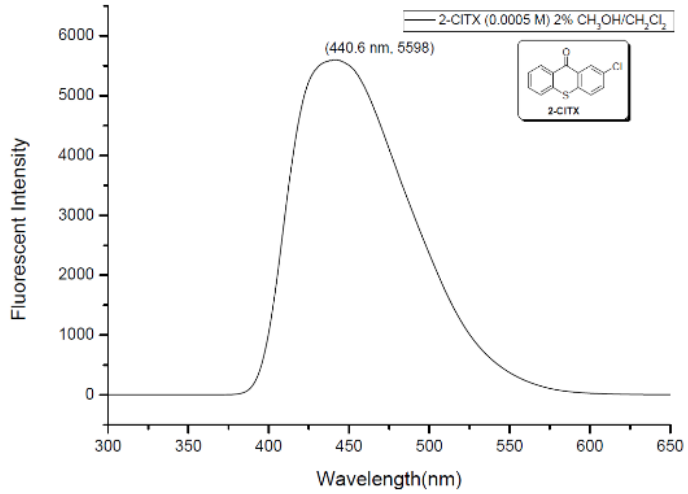


Figure S9. UV-Vis of **I** (**2-CITX**) in CH_2Cl_2.

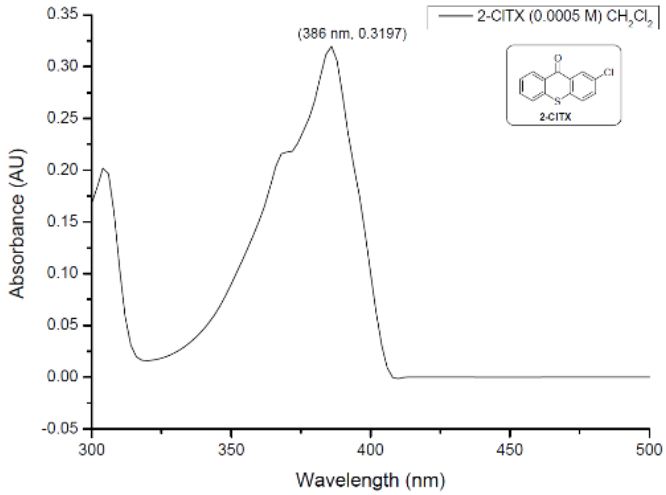


Figure S10. Stern-Volmer quenching of catalyst **I** (**2-ClTX**) with **1a** (**IRR-140**).

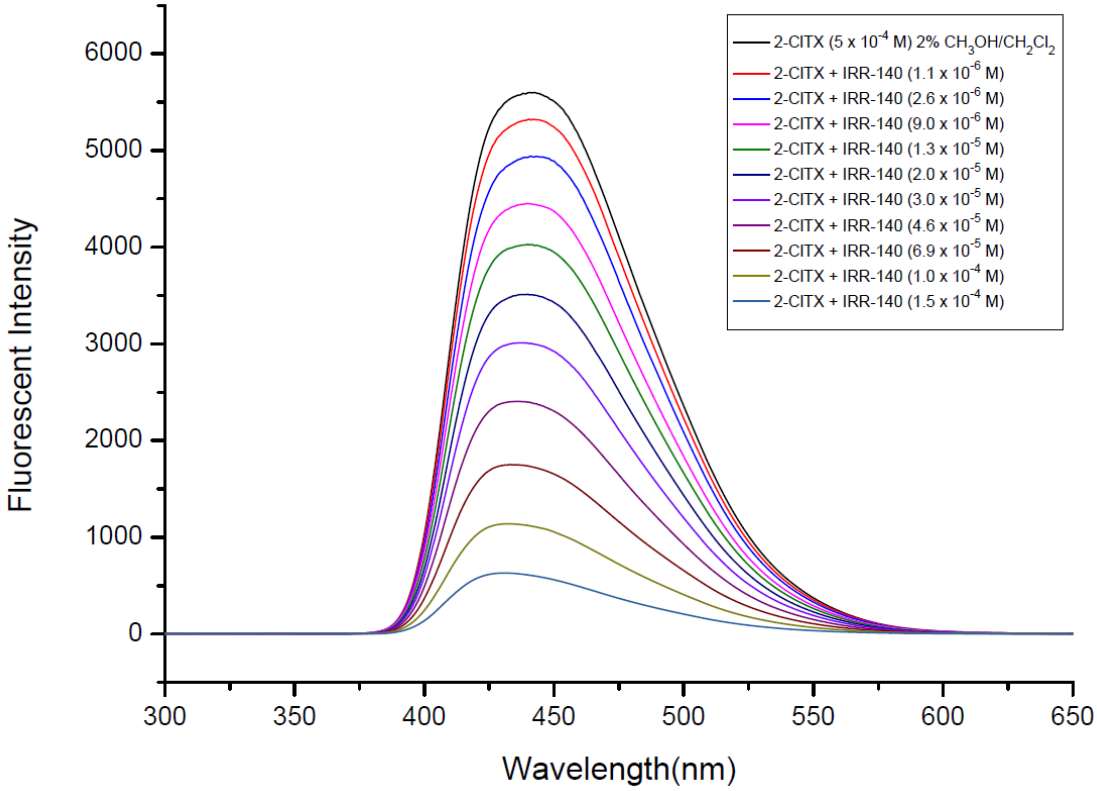
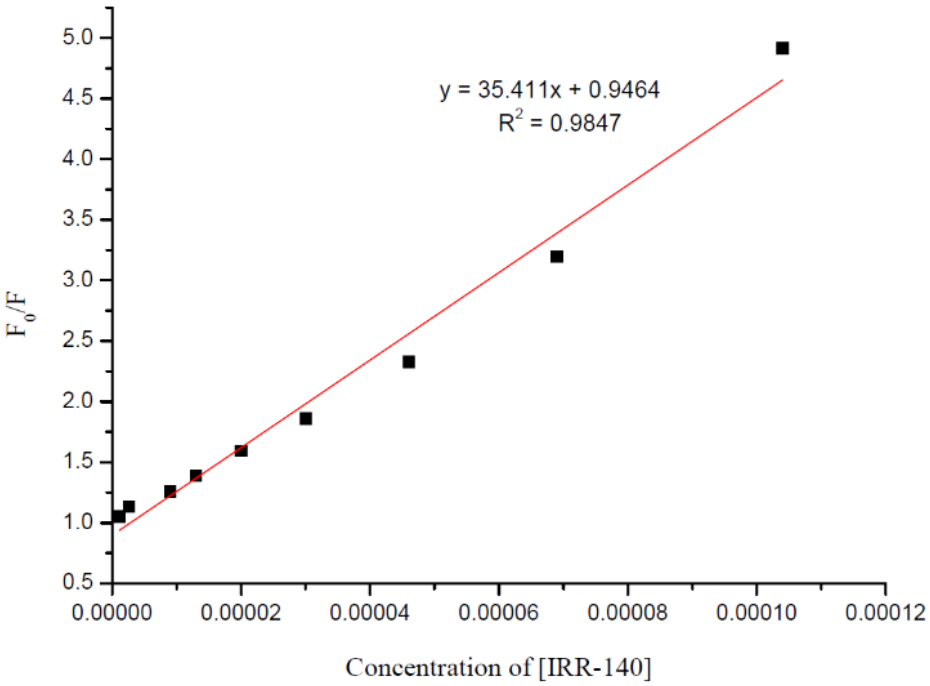
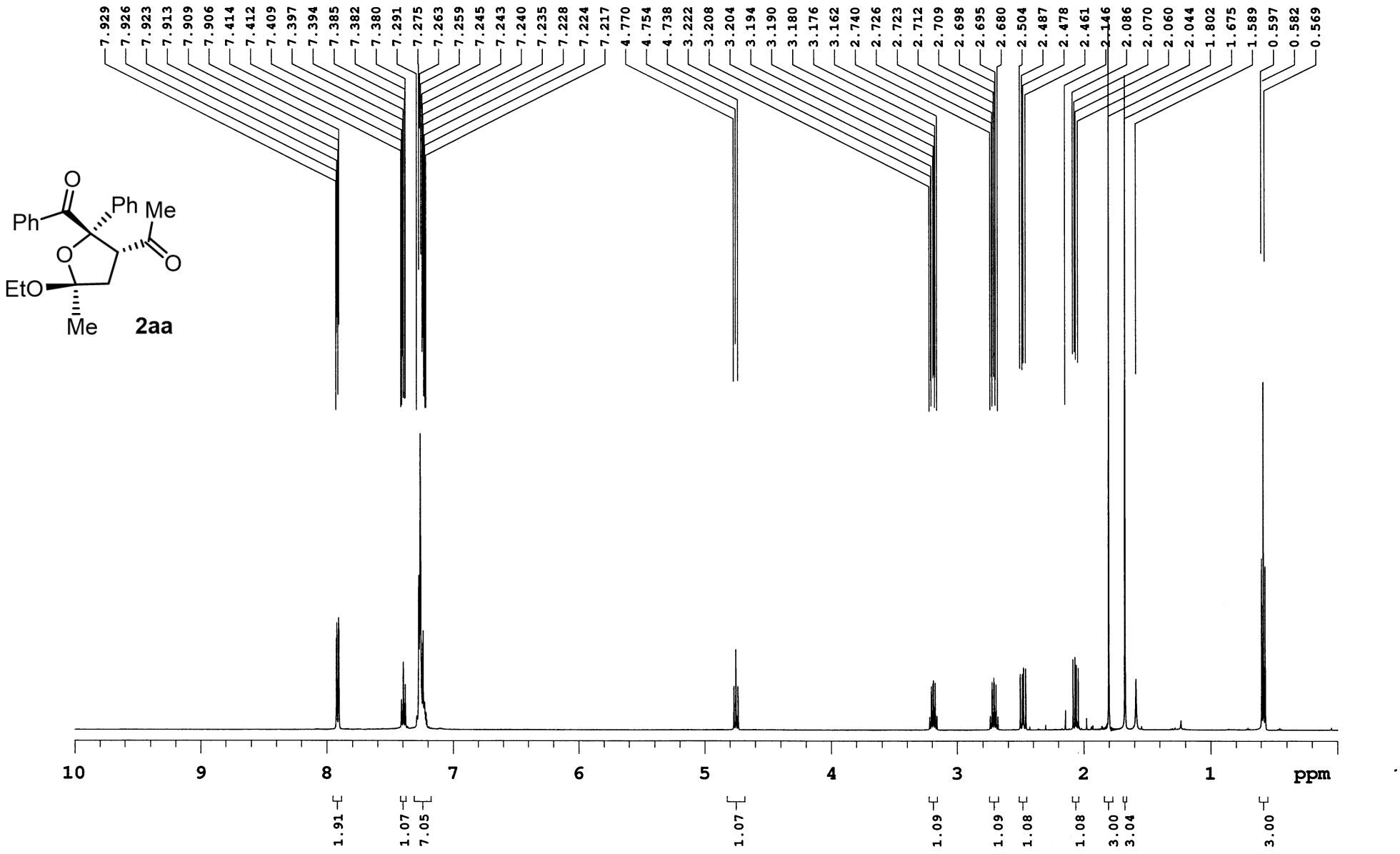


Figure S11. Fluorescence quenching (Stern-Volmer) plot of **1a** (**IRR-140**) and **I** (**2-ClTX**) in 2% CH_3OH/CH_2Cl_2.



IRR-02-137

Sample Name	**IRR-02-137**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-01**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-137/PROTON_03

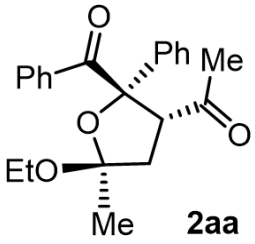
1H NMR (500 MHz, CDCl3) of compound 2aa

Plot date 2023-11-02

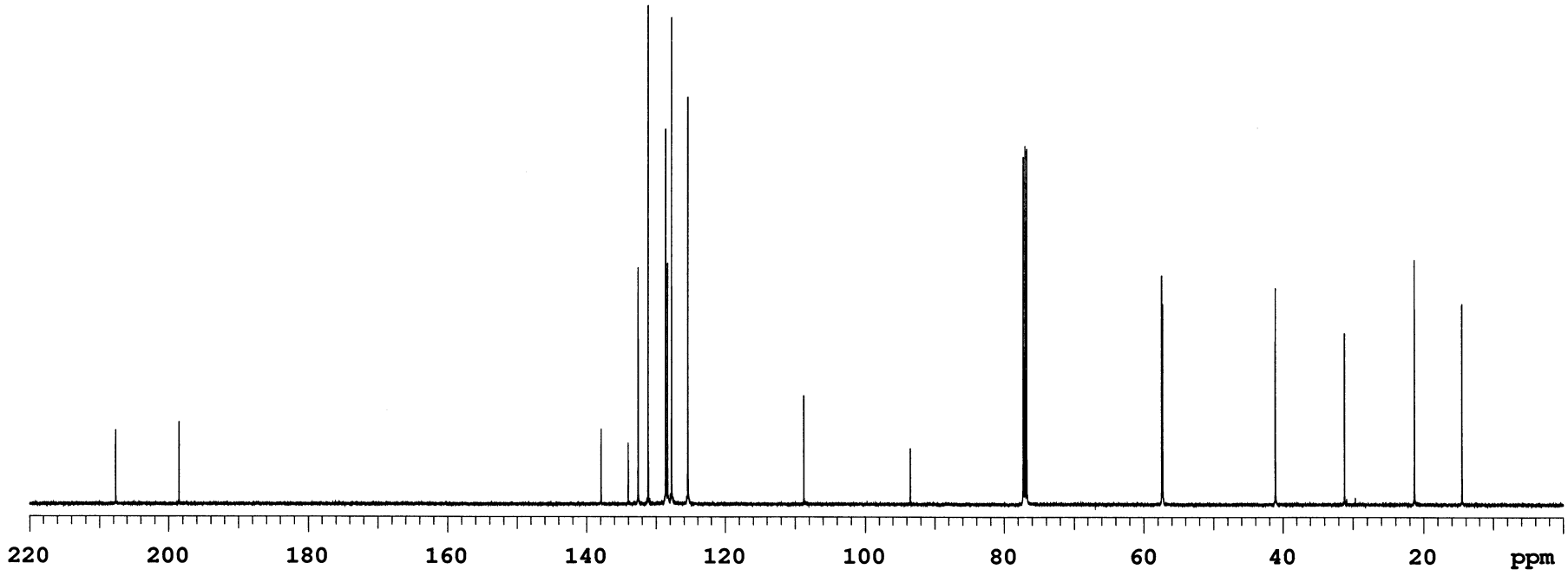
IRR-02-137

Sample Name	**IRR-02-137**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-01**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

207.662
198.549
137.931
134.008
132.586
131.141
128.615
128.340
127.768
125.424
108.745
93.545
77.254
77.000
76.746
57.464
57.317
41.137
31.195
21.332
14.513



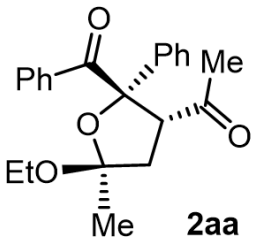
2aa



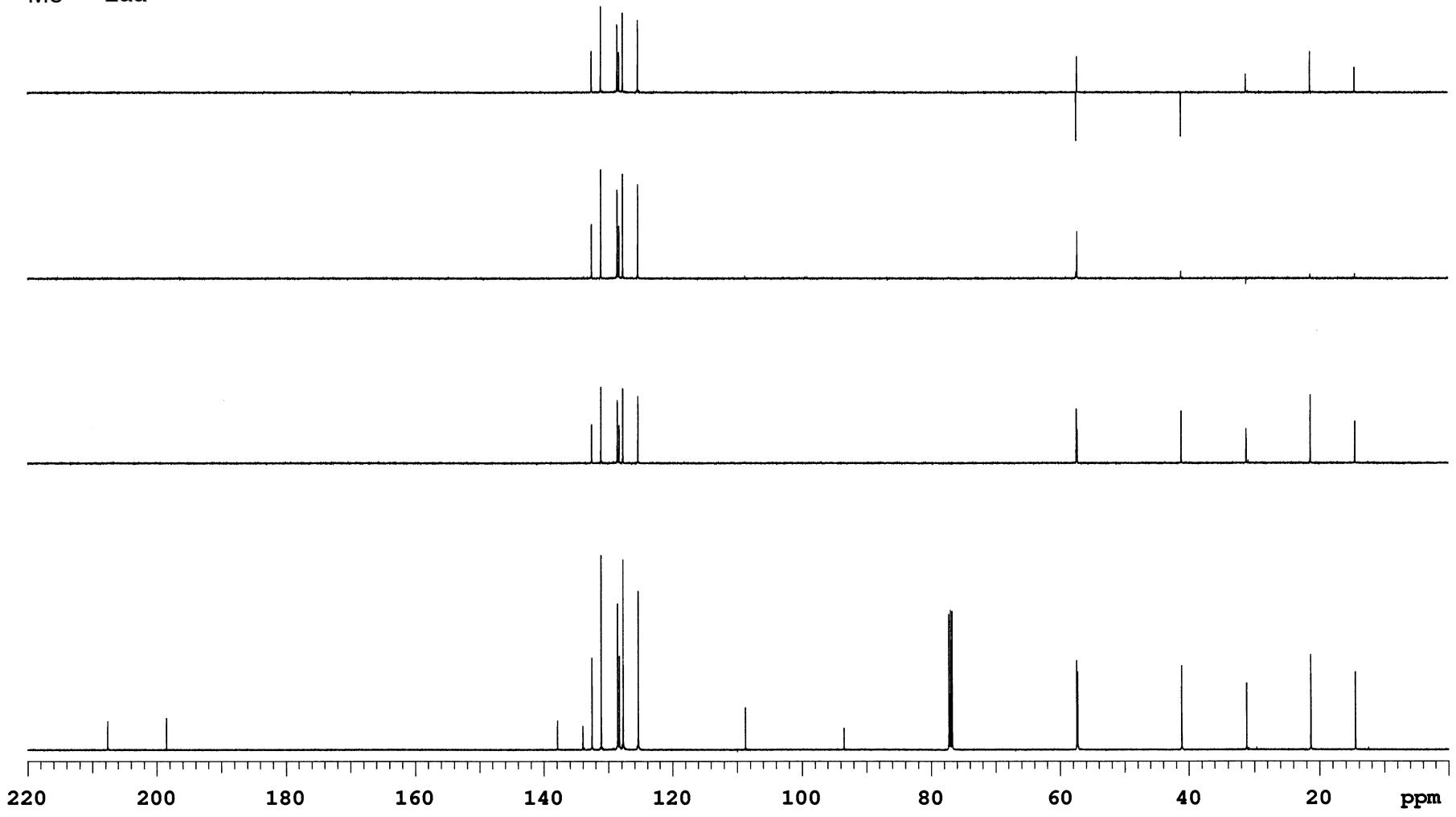
13C NMR (125 MHz, CDCl3) of compound 2aa

IRR-02-137

Sample Name	**IRR-02-137**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-02**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



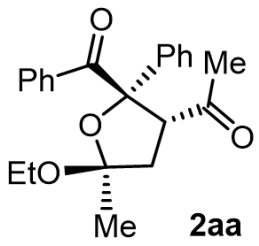
2aa



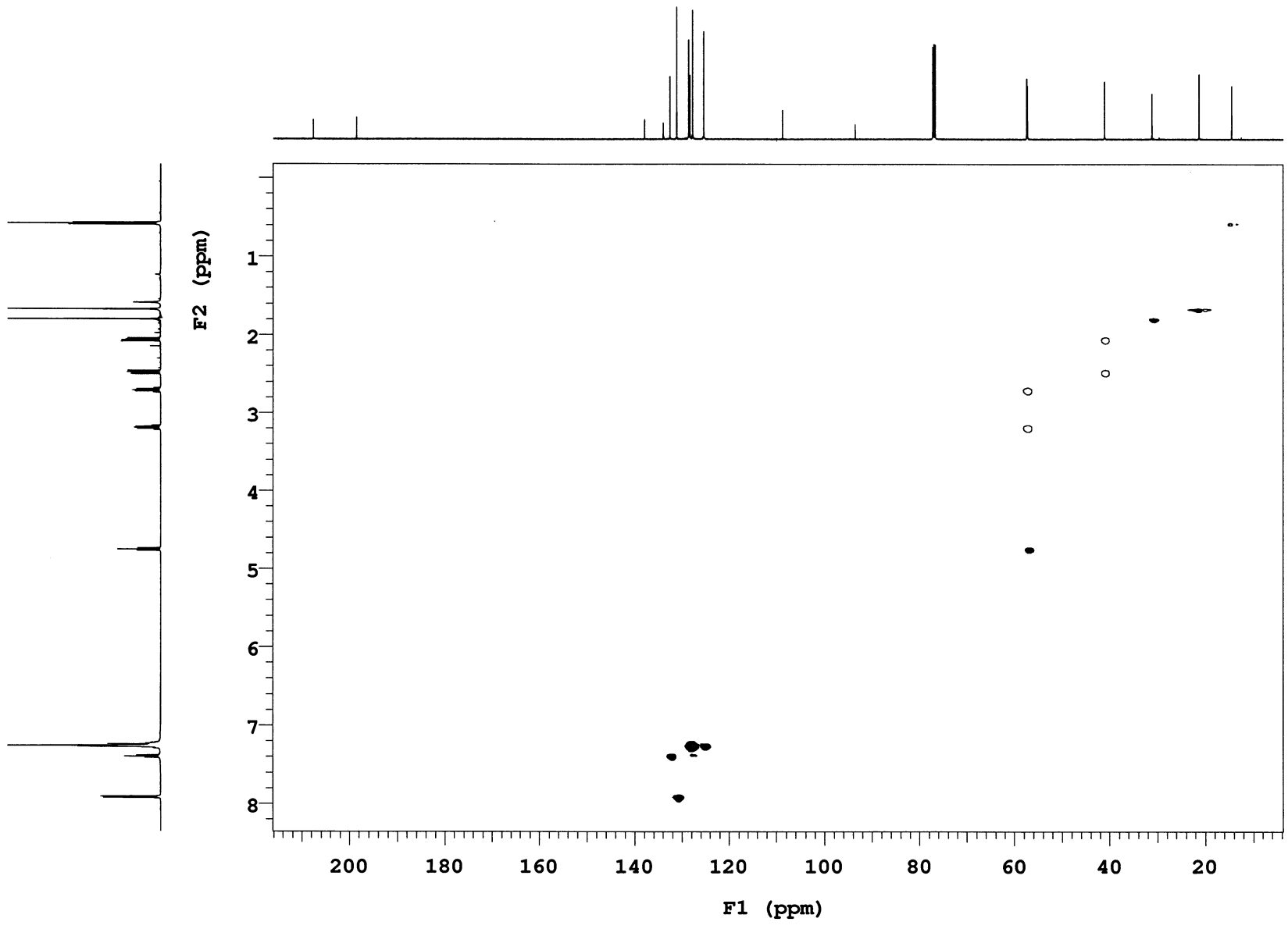
DEPT of compound 2aa

IRR-02-137

Sample Name **IRR-02-137**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2023-11-02**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



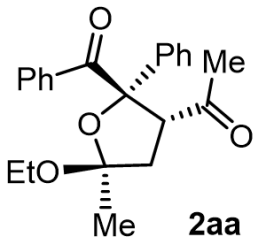
2aa



HSQC of compound 2aa

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-137/gHSQC_01

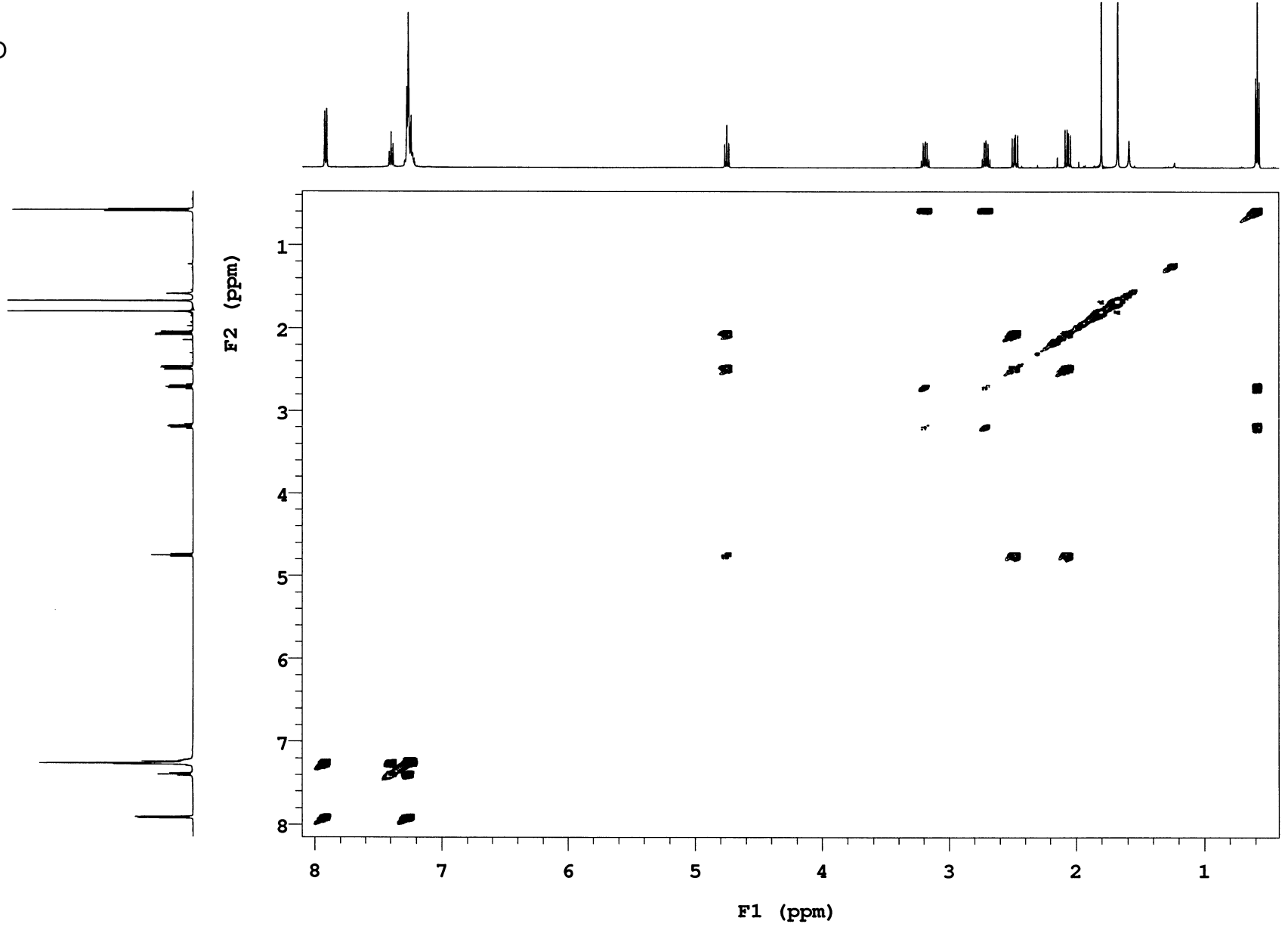
Plot date 2023-11-02



2aa

IRR-02-137

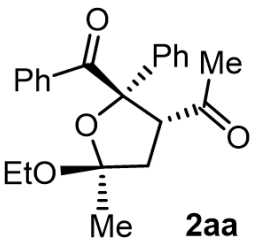
Sample Name	**IRR-02-137**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-02**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



COSY of compound 2aa

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-137/gCOSY_01

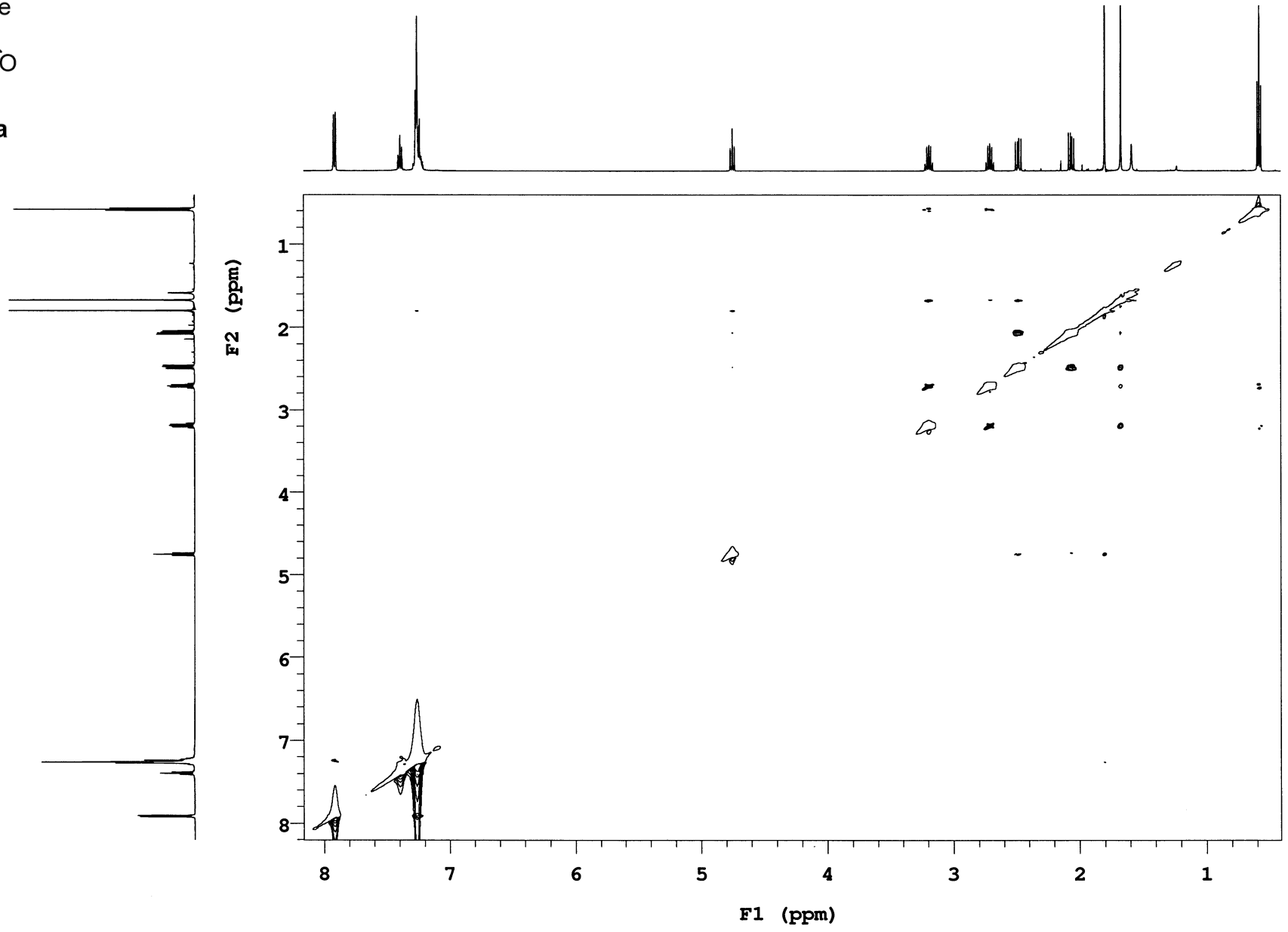
Plot date 2023-11-02



2aa

IRR-02-137

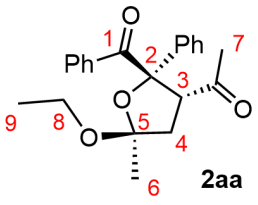
Sample Name	**IRR-02-137**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-02**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2aa

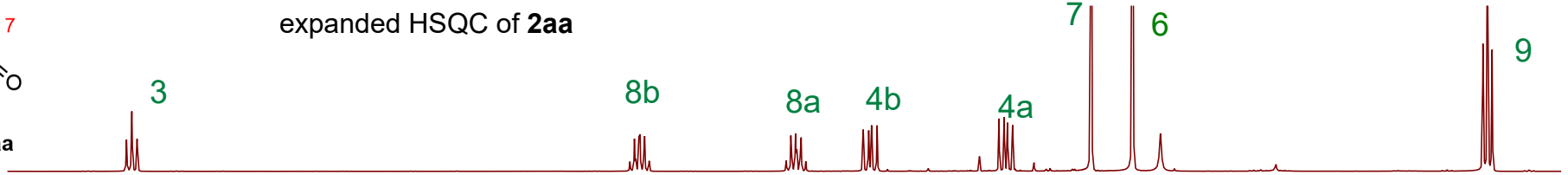
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-137/NOESY_01

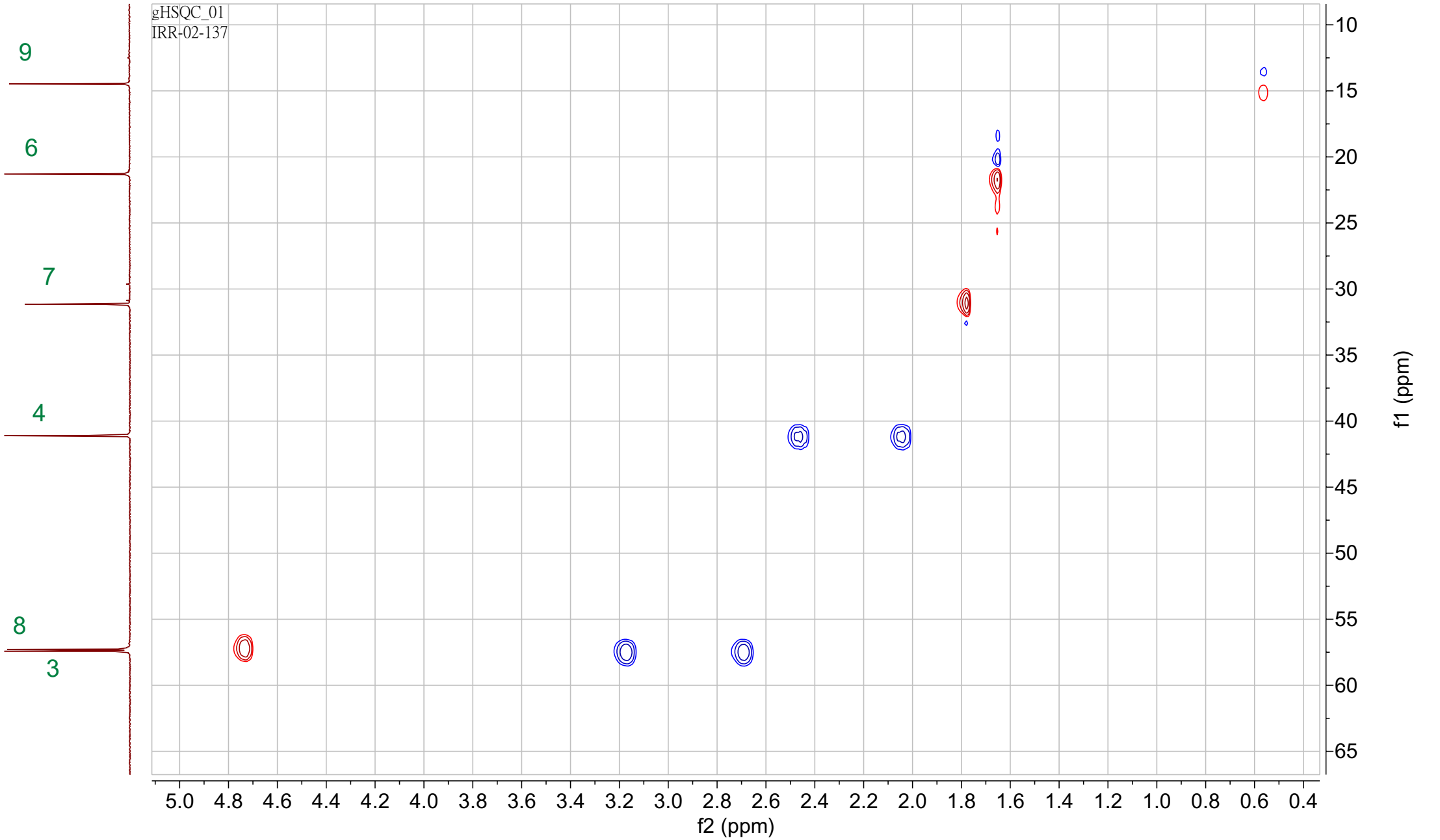
Plot date 2023-11-02



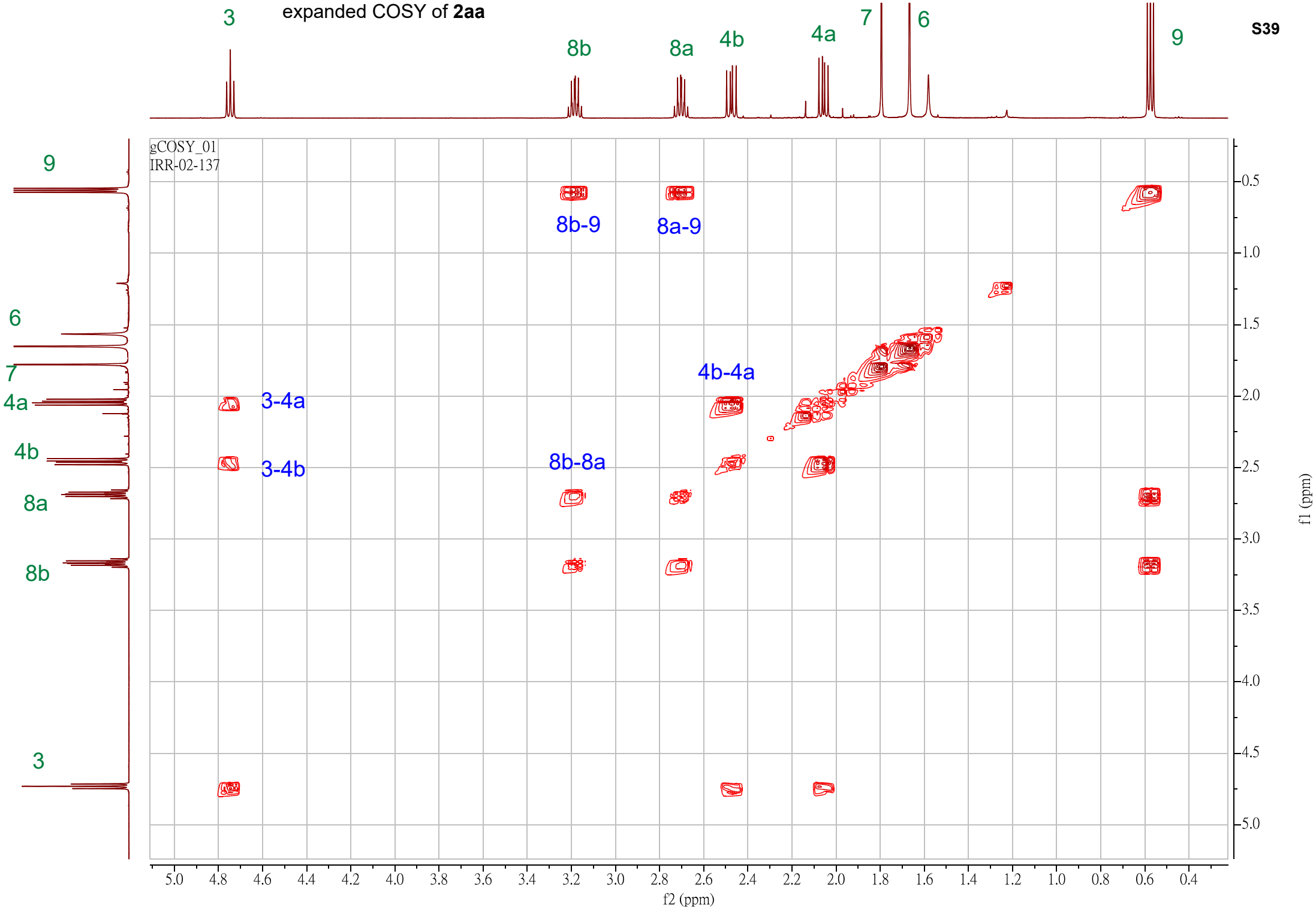
2aa

expanded HSQC of **2aa**

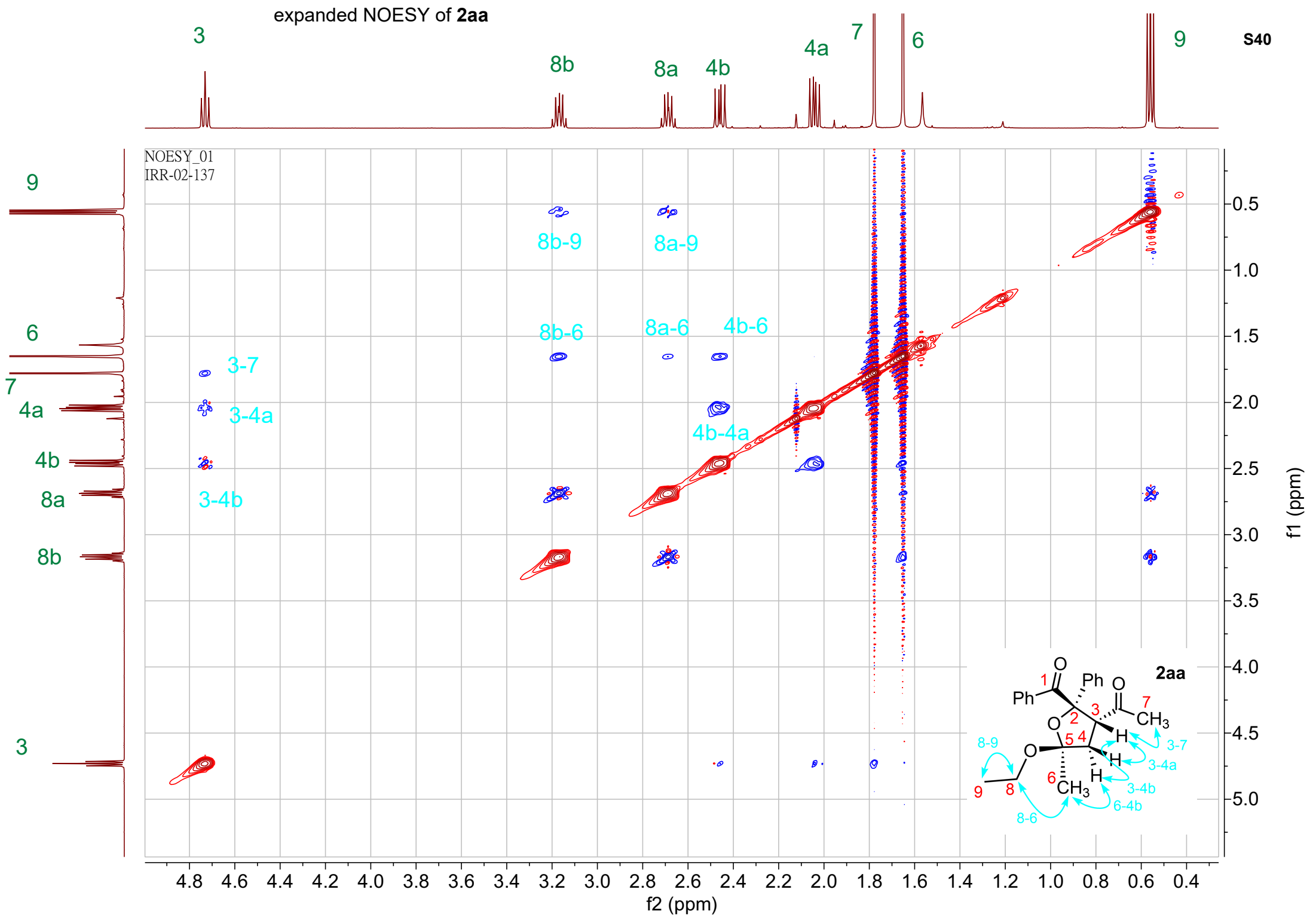


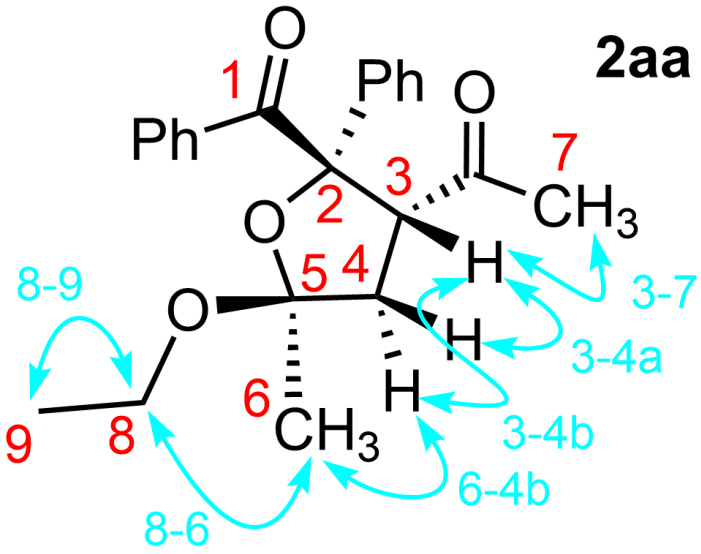


expanded COSY of **2aa**

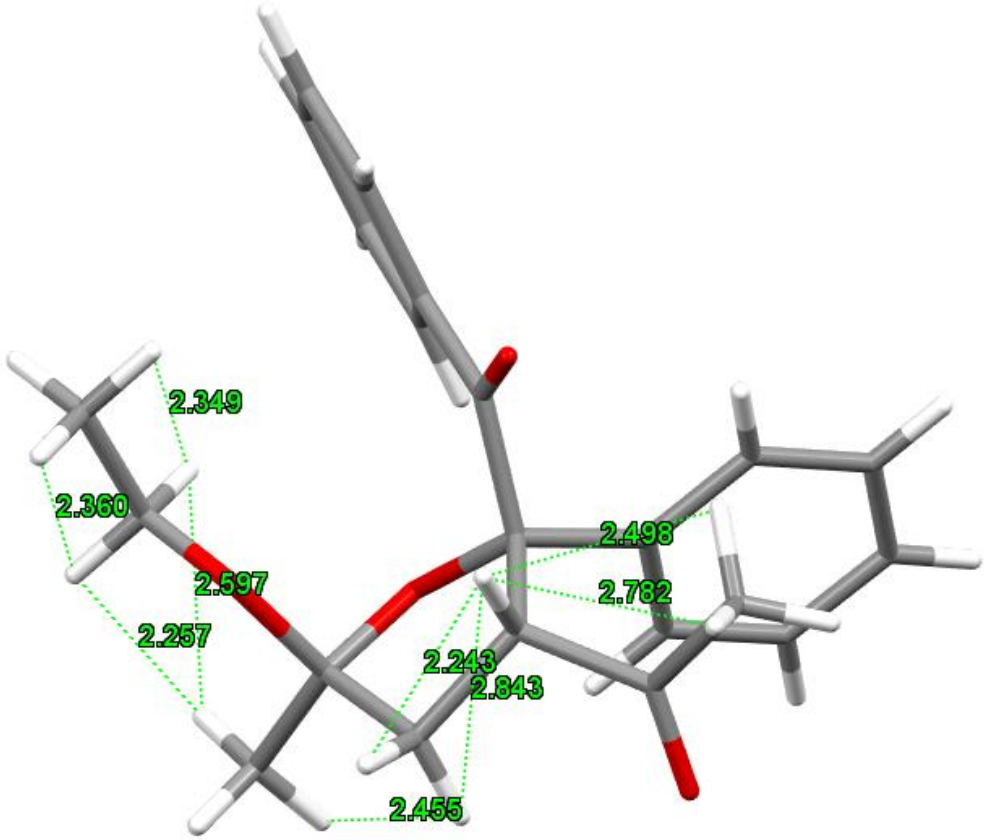


expanded NOESY of **2aa**





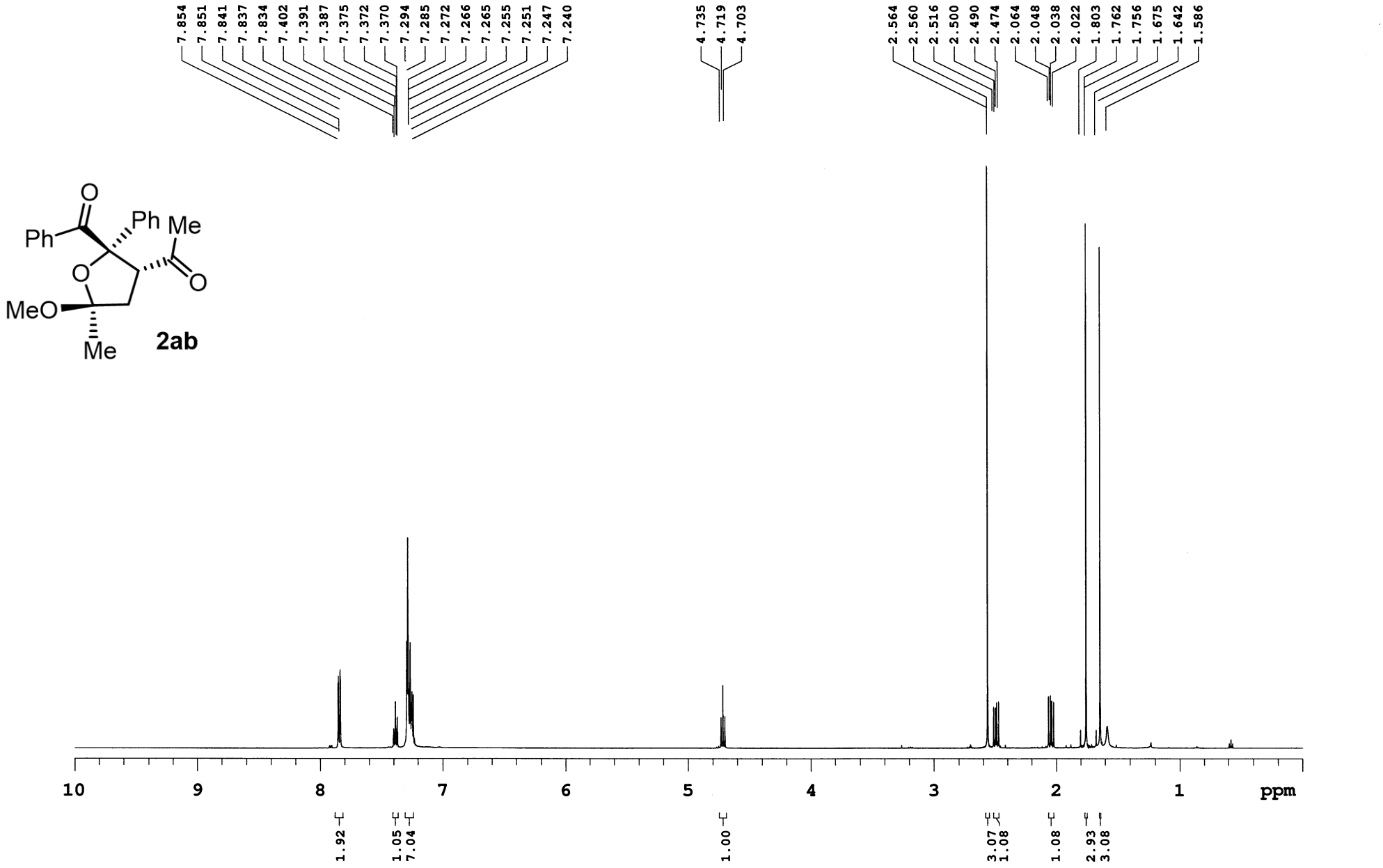
Analysis of Data from the NOESY Spectrum of **2aa**



Atom distances measured from X-ray data of **2aa**

IRR-02-217

Sample Name	**IRR-02-217**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-20**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-217/PROTON_03

1H NMR (500 MHz, CDCl3) of compound 2ab

Plot date 2024-02-21

IRR-02-217

Sample Name	**IRR-02-217**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-20**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

207.727
198.862
137.612
134.430
132.387
130.969
128.676
128.415
127.829
125.494
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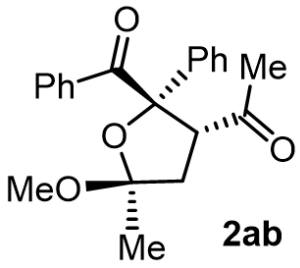
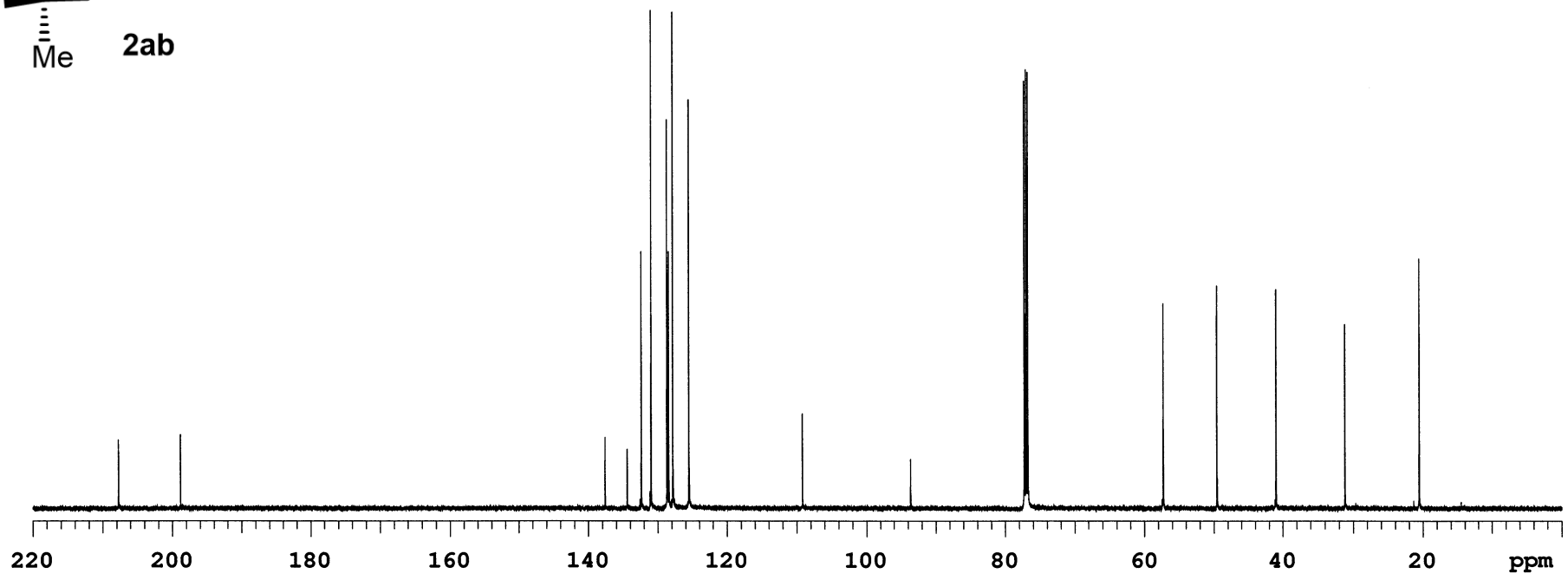
57.288
49.554
40.996
31.194
20.542



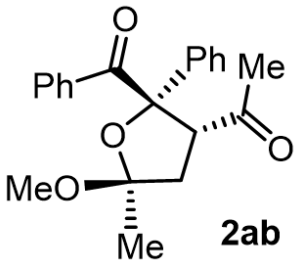
2ab



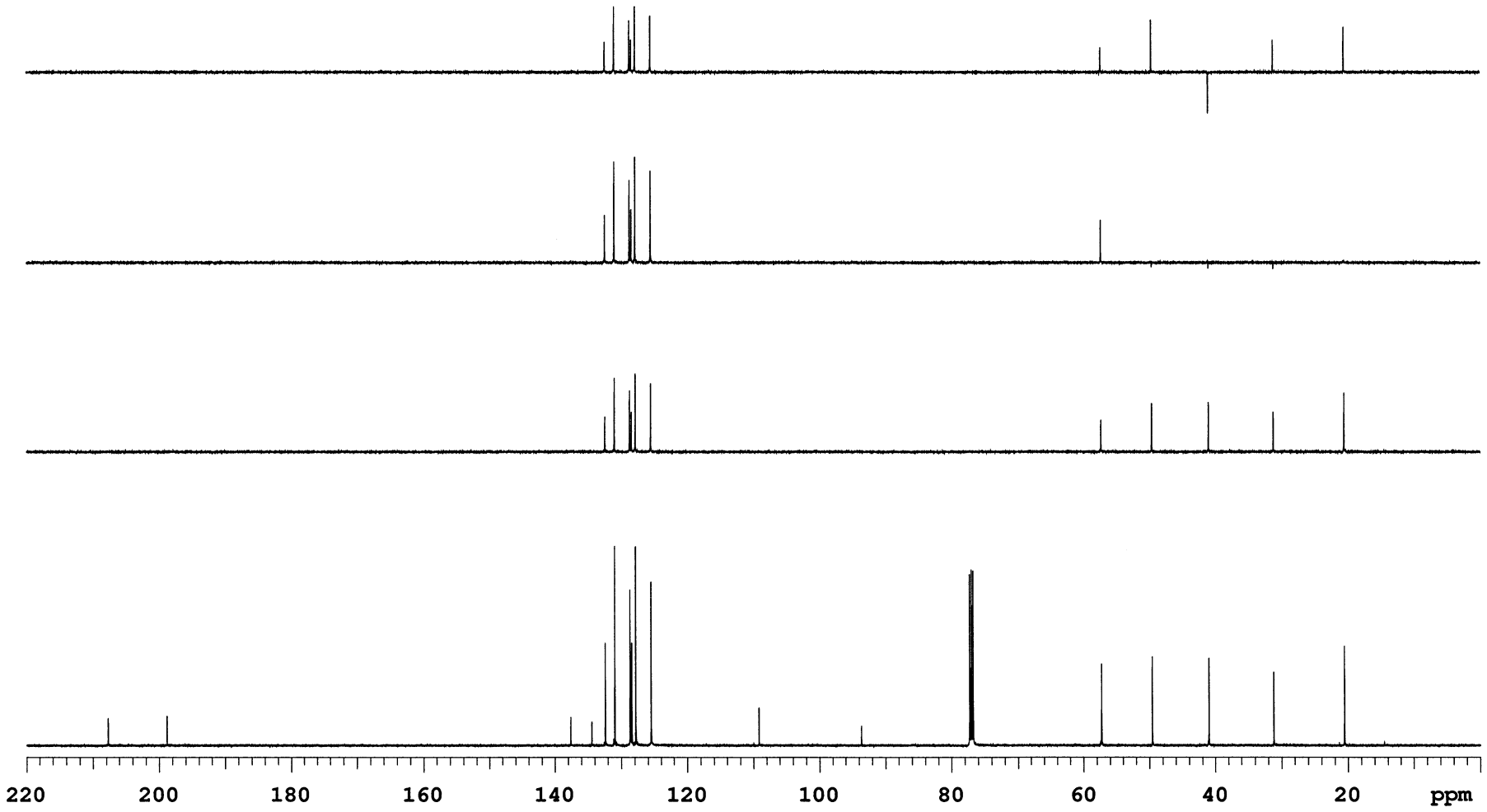
13C NMR (125 MHz, CDCl3) of compound 2ab

IRR-02-217

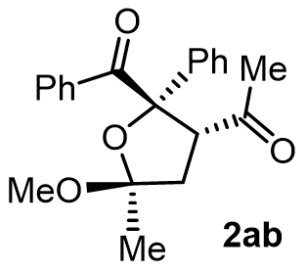
Sample Name	**IRR-02-217**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2ab



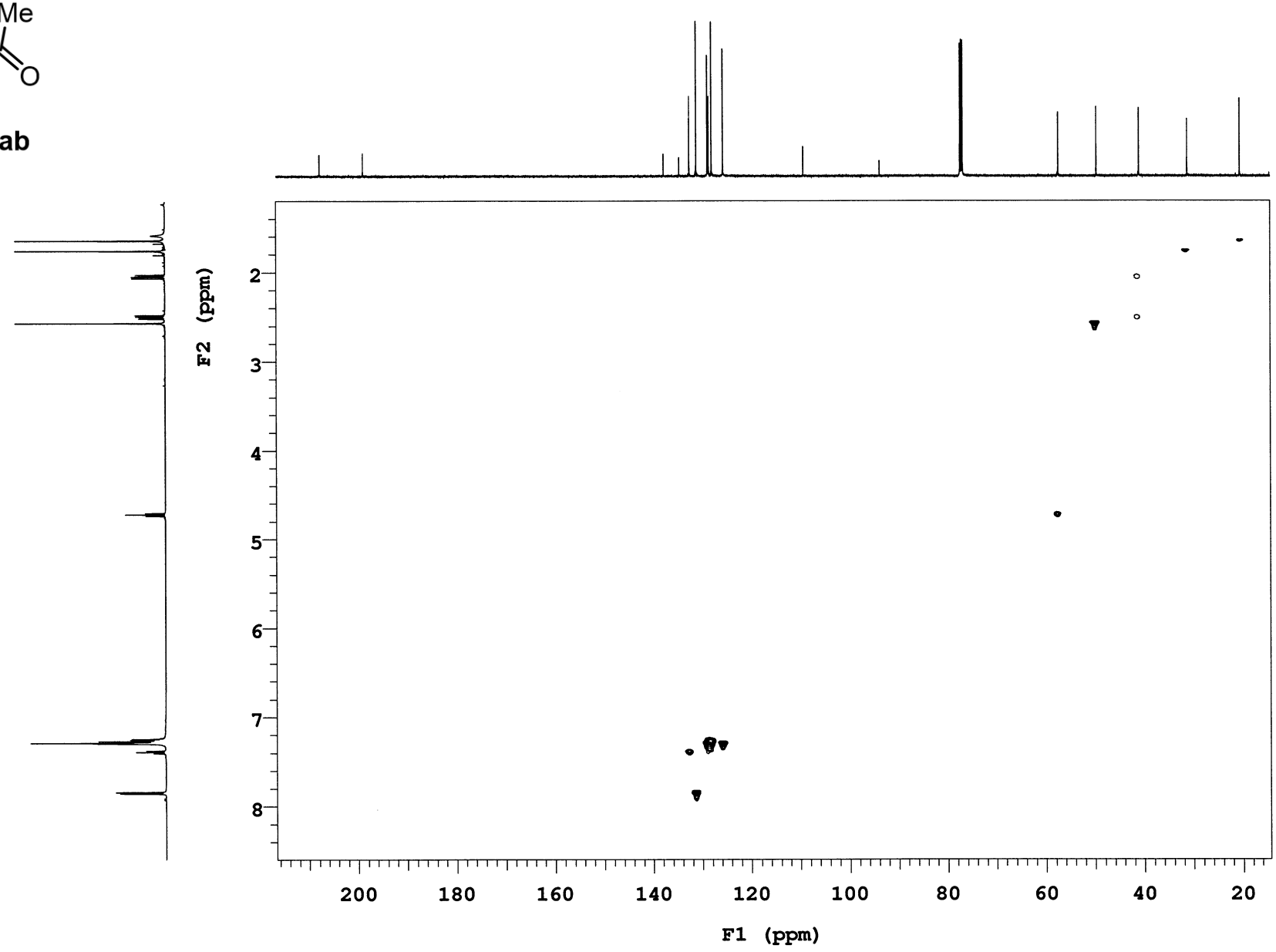
DEPT of compound 2ab



2ab

IRR-02-217

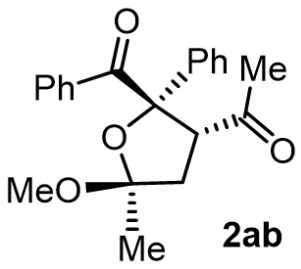
Sample Name	**IRR-02-217**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2ab

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-217/gHSQC_01

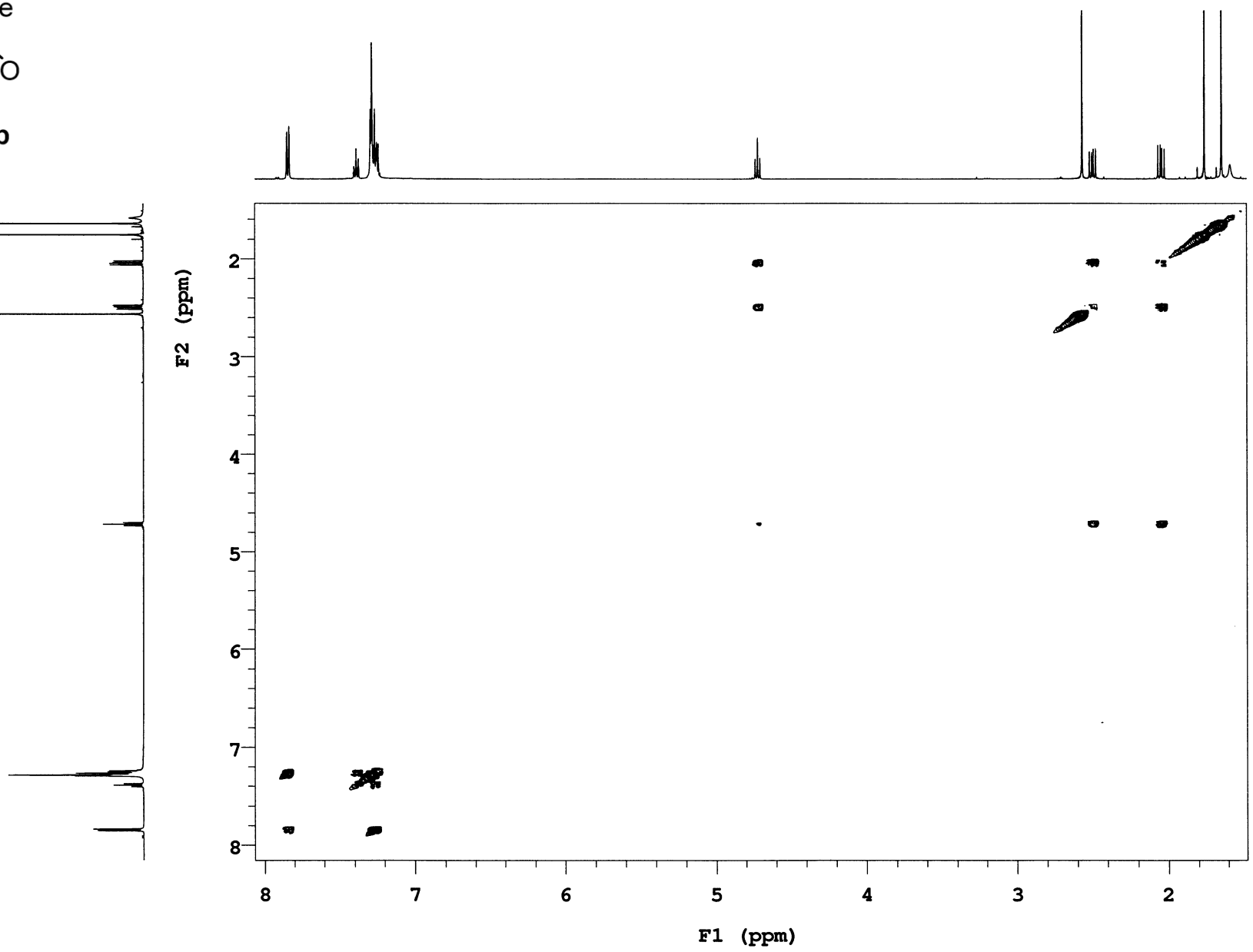
Plot date 2024-02-21



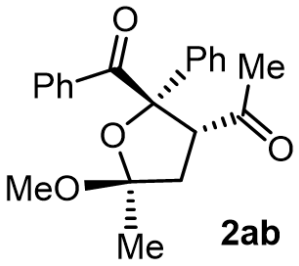
2ab

IRR-02-217

Sample Name	**IRR-02-217**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



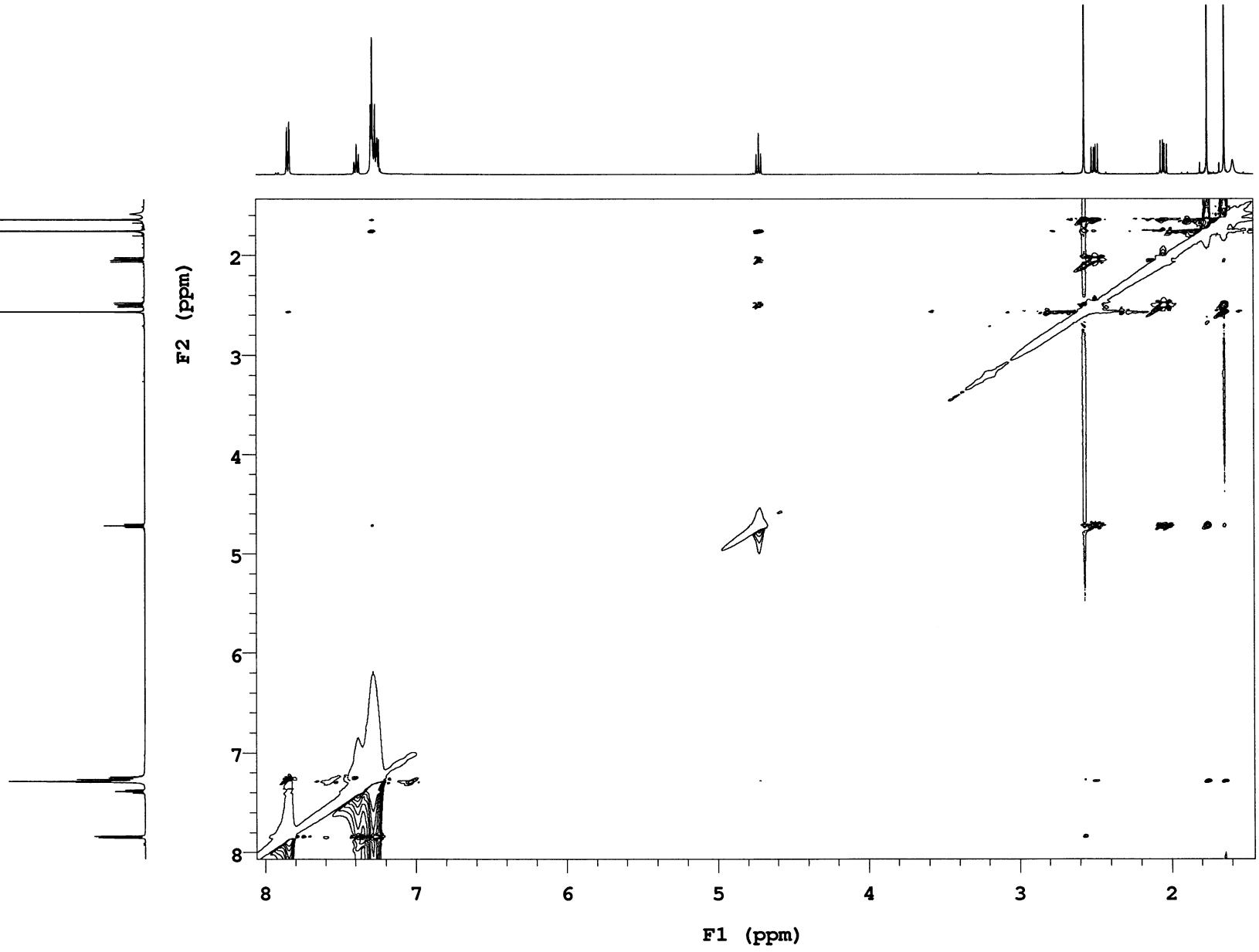
COSY of compound 2ab



2ab

IRR-02-217

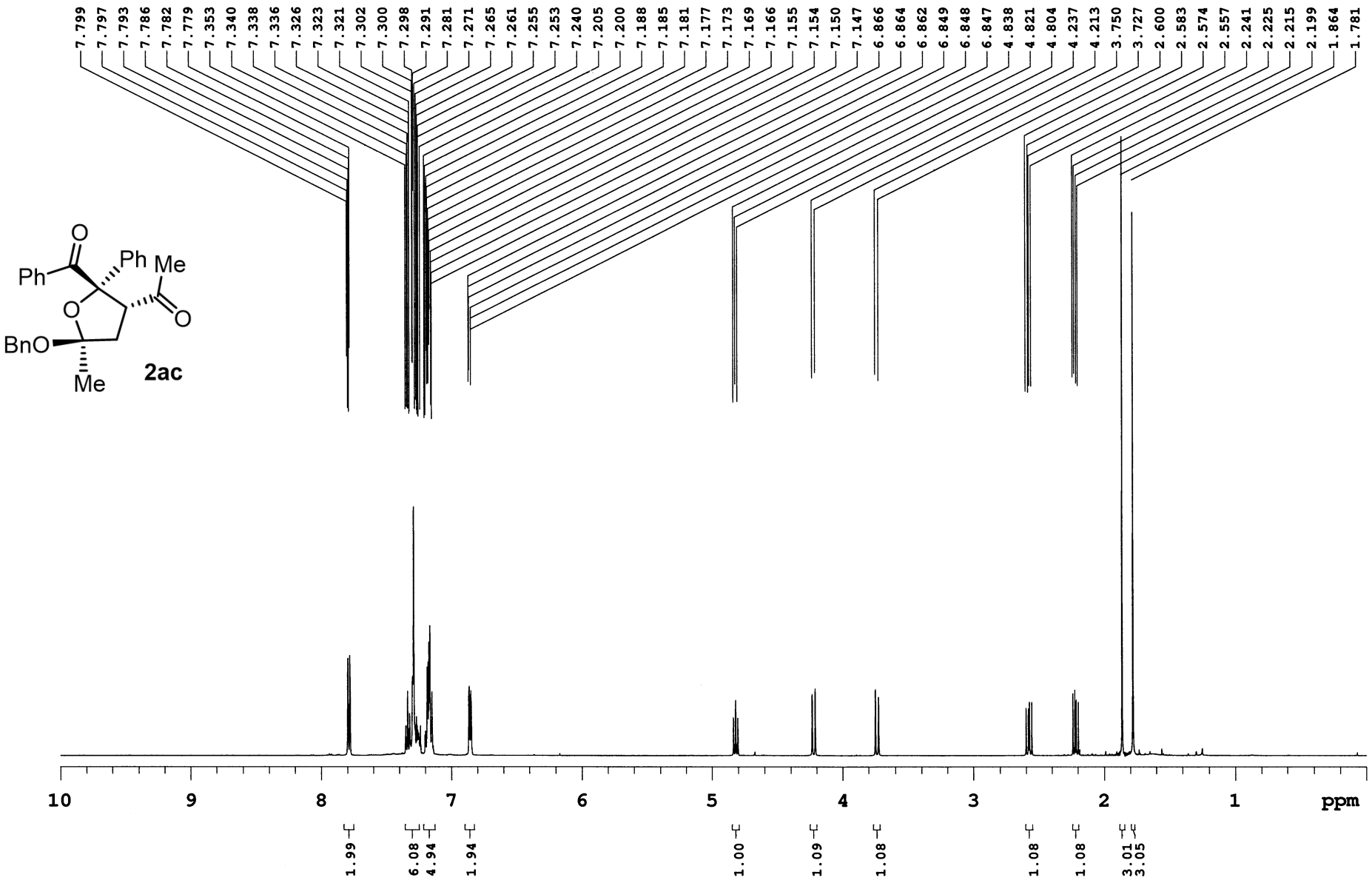
Sample Name	**IRR-02-217**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2ab

IRR-02-220

Sample Name	**IRR-02-220**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-05**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



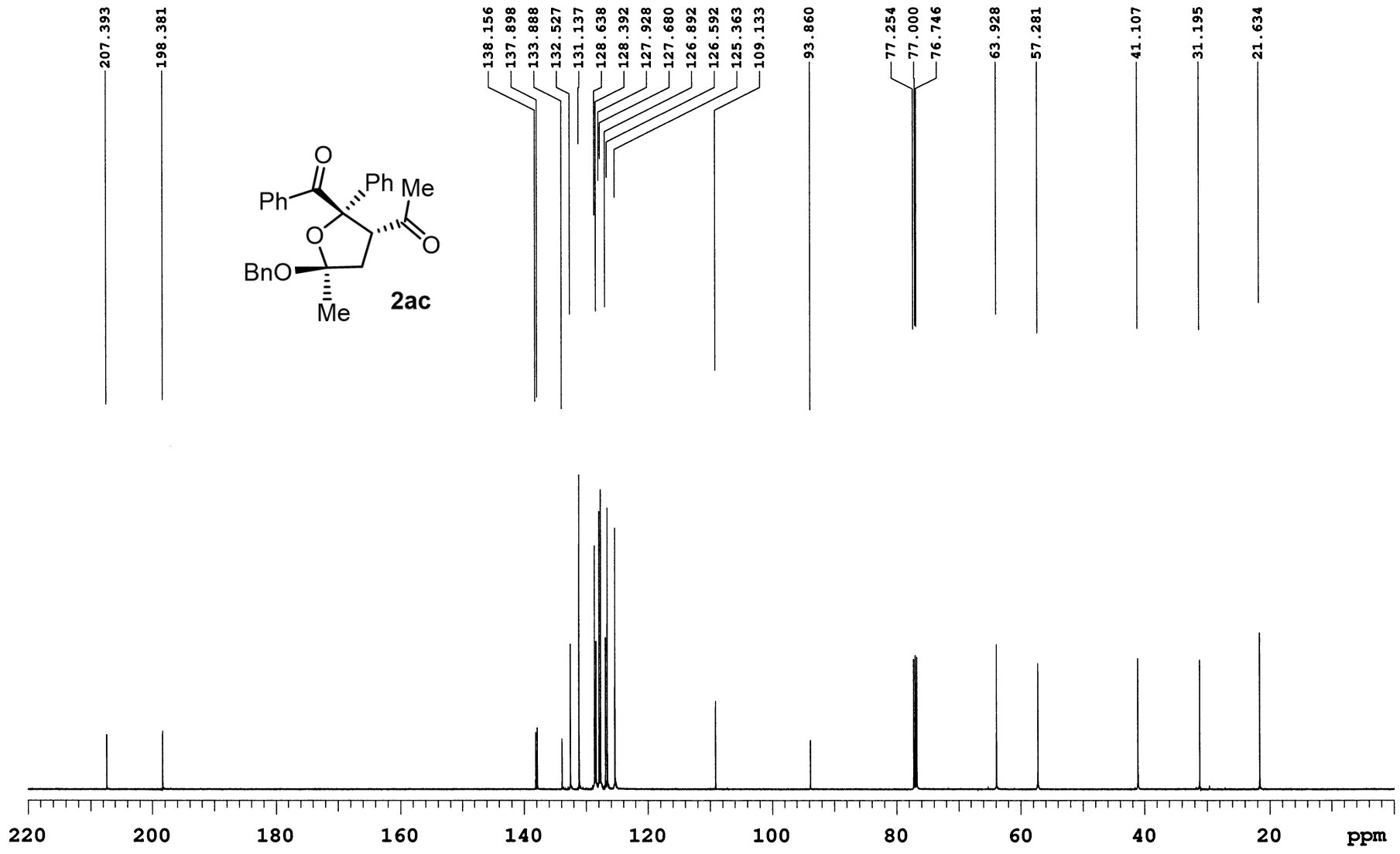
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-220/PROTON_03

1H NMR (500 MHz, CDCl3) of compound 2ac

Plot date 2024-02-06

IRR-02-220

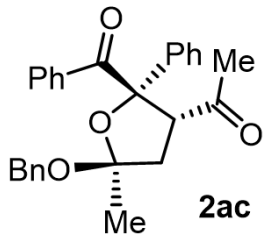
Sample Name	**IRR-02-220**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-05**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



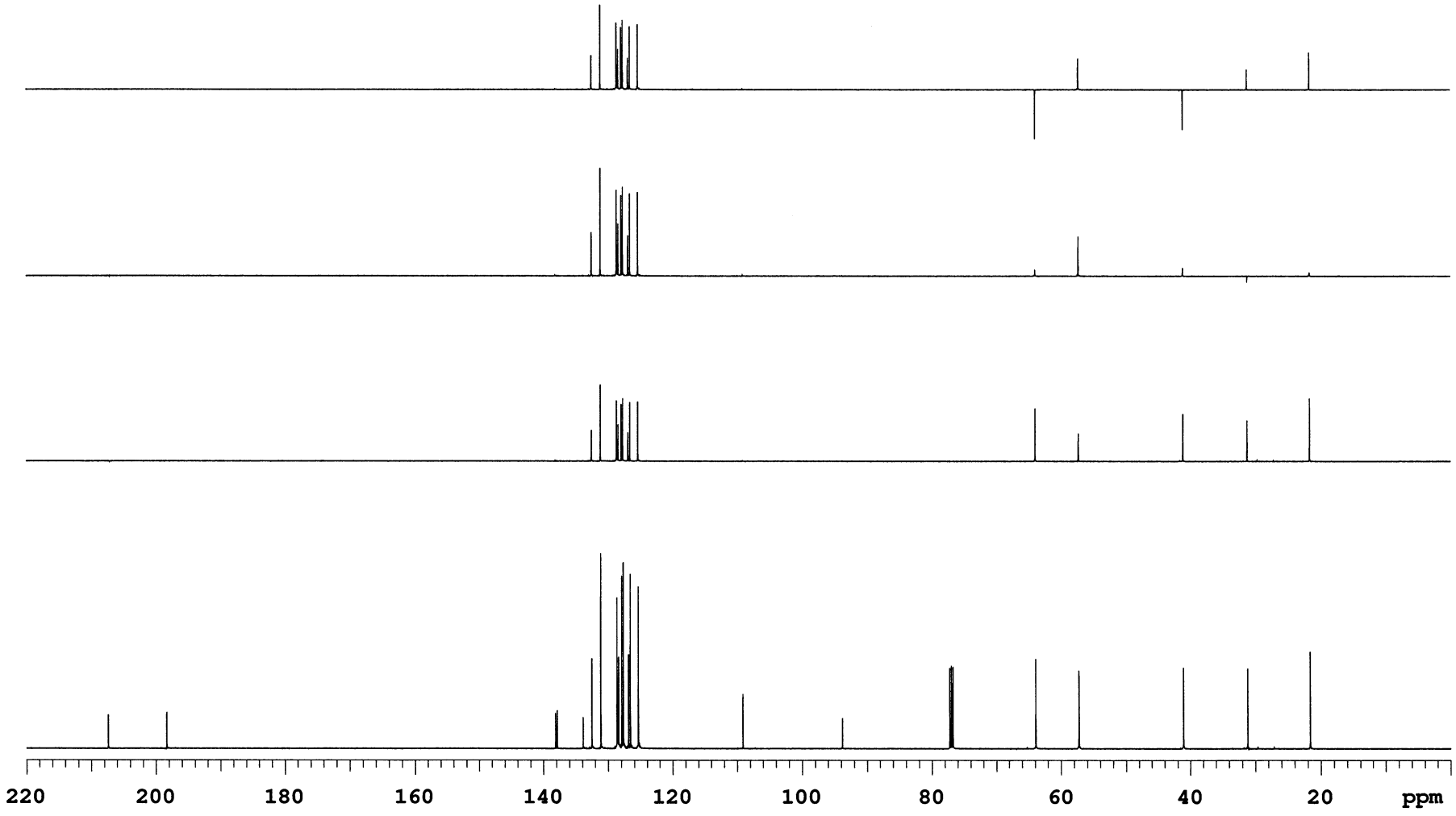
13C NMR (125 MHz, CDCl3) of compound 2ac

IRR-02-220

Sample Name	**IRR-02-220**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-05**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



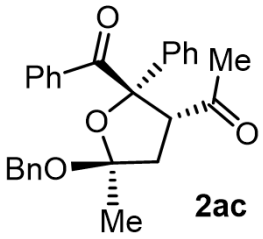
2ac



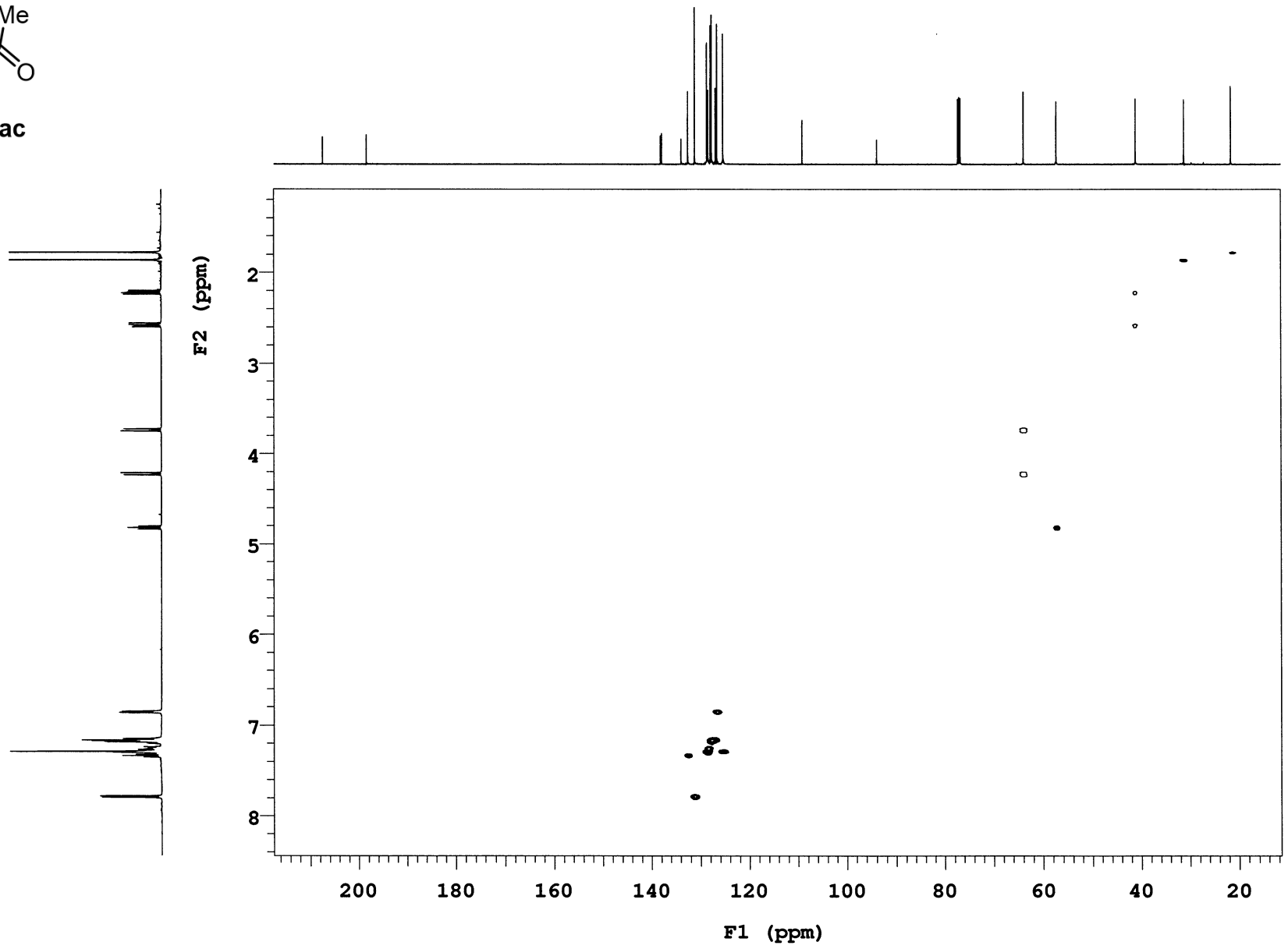
DEPT of compound 2ac

IRR-02-220

Sample Name	**IRR-02-220**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-06**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



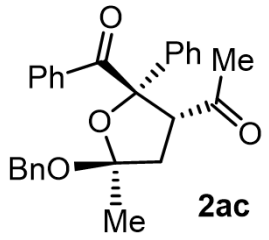
2ac



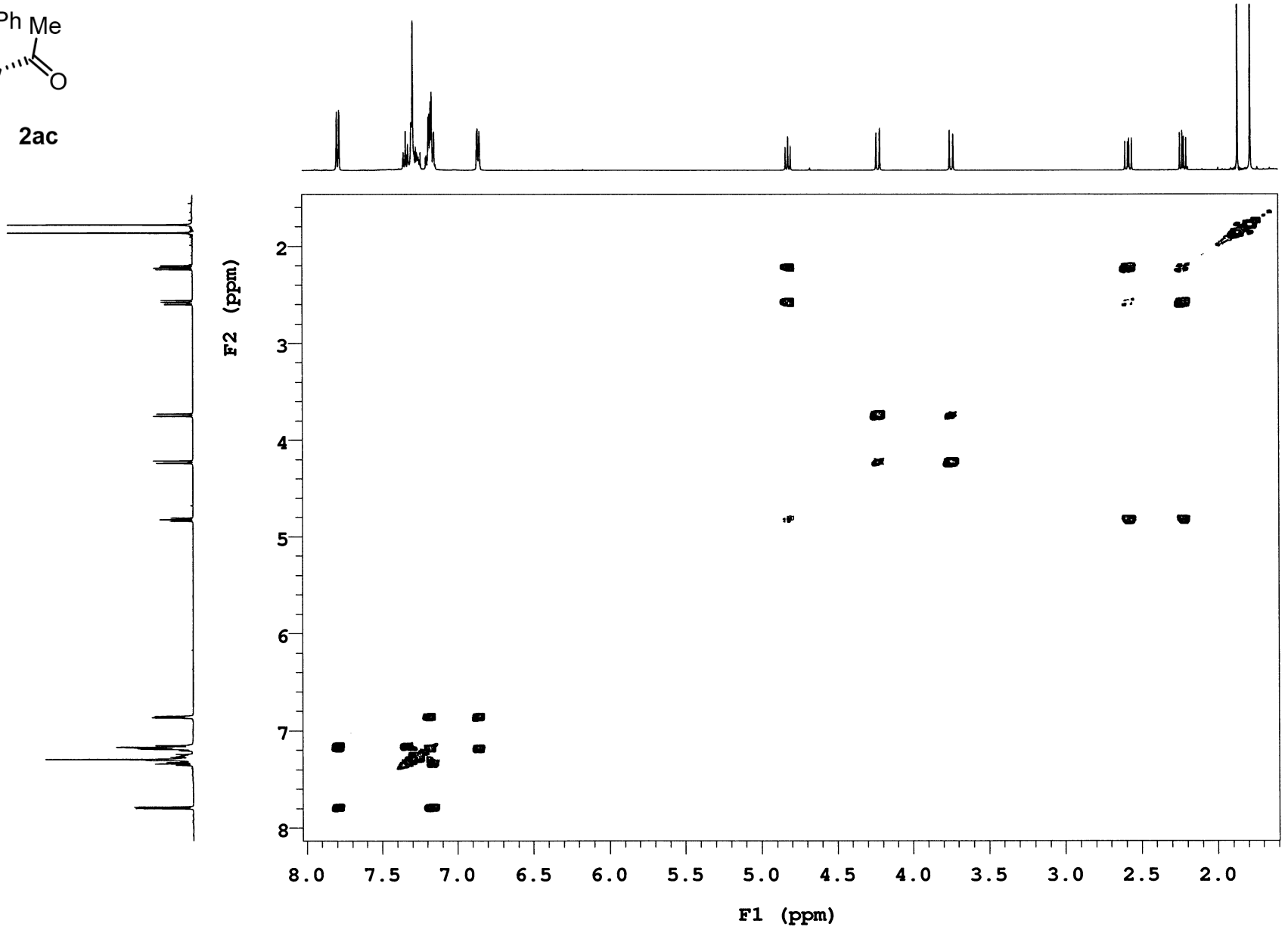
HSQC of compound 2ac

IRR-02-220

Sample Name	**IRR-02-220**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-06**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



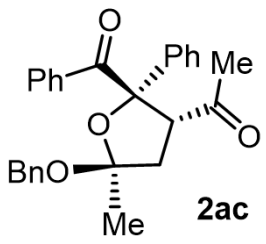
2ac



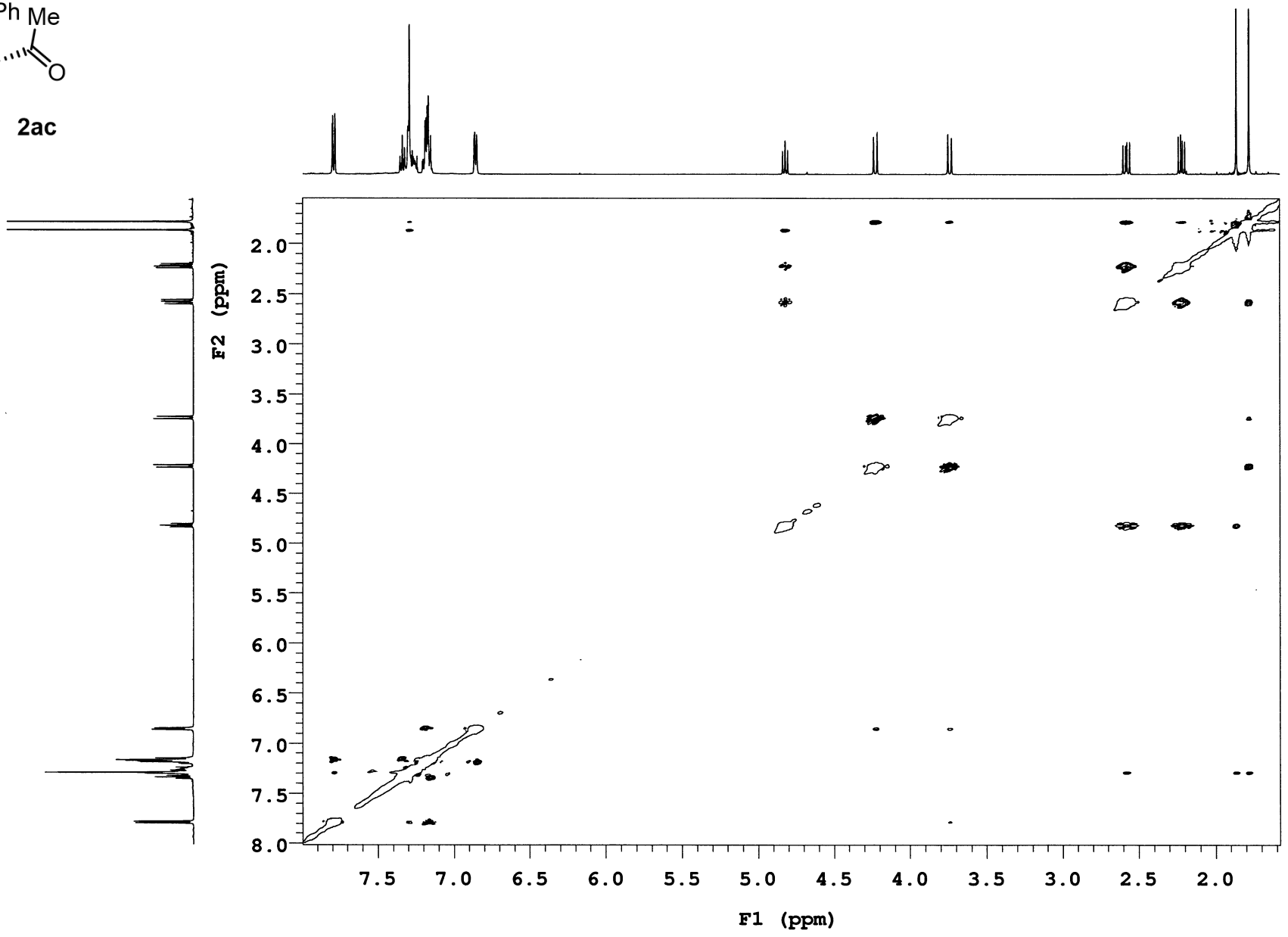
COSY of compound 2ac

IRR-02-220

Sample Name	**IRR-02-220**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-06**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



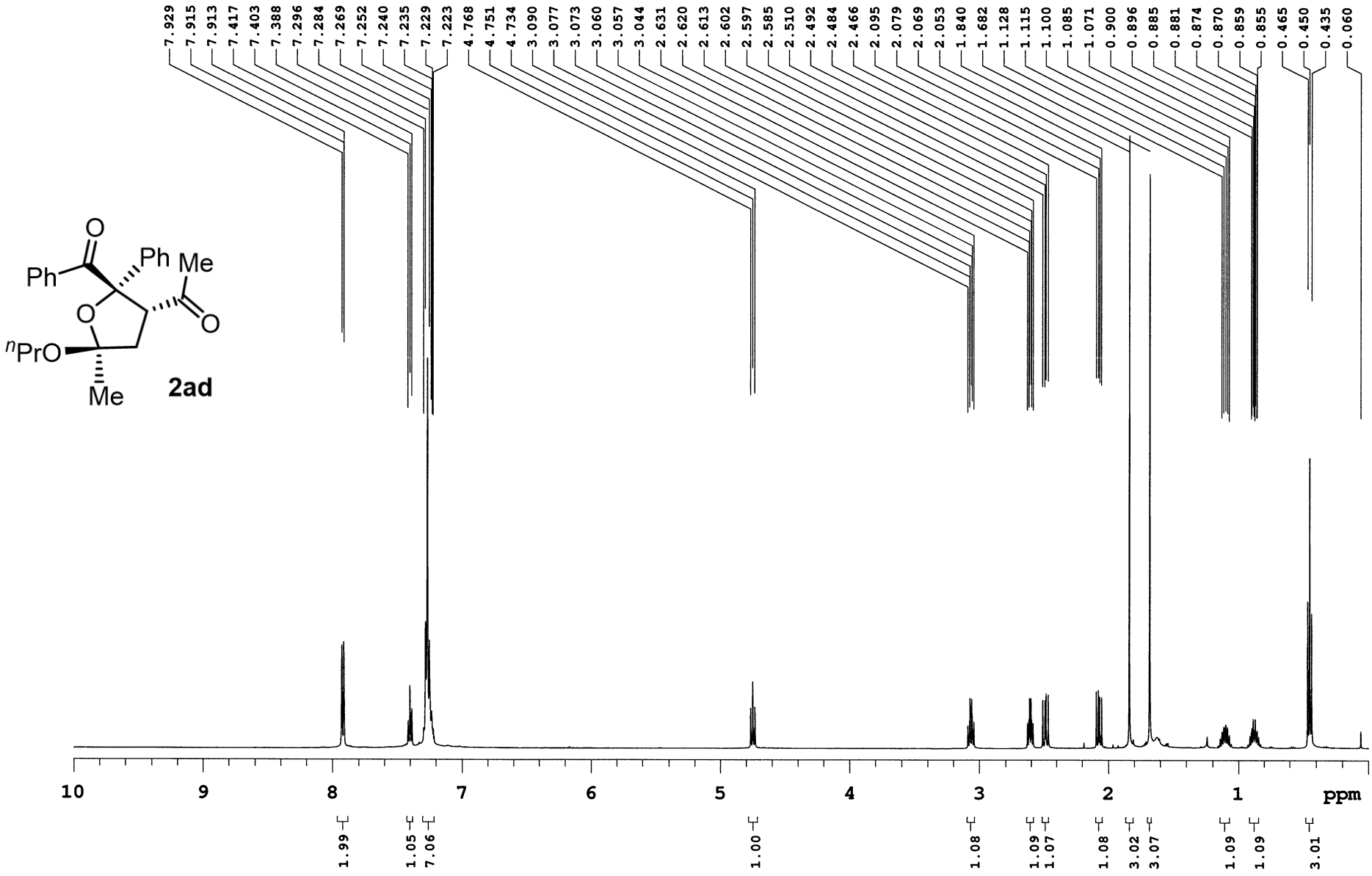
2ac



NOESY of compound 2ac

IRR-02-218

Sample Name	**IRR-02-218**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-218/PROTON_03

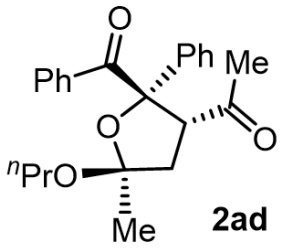
1H NMR (500 MHz, CDCl3) of compound 2ad

Plot date 2024-02-22

IRR-02-218

Sample Name	**IRR-02-218**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

207.574
198.541
138.017
134.025
132.538
131.181
128.596
128.323
127.770
125.403
108.690
93.543
77.256
77.000
76.746
63.672
57.330
41.023
31.190
22.395
21.345
9.961

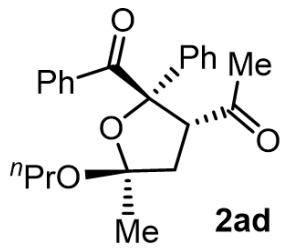


2ad

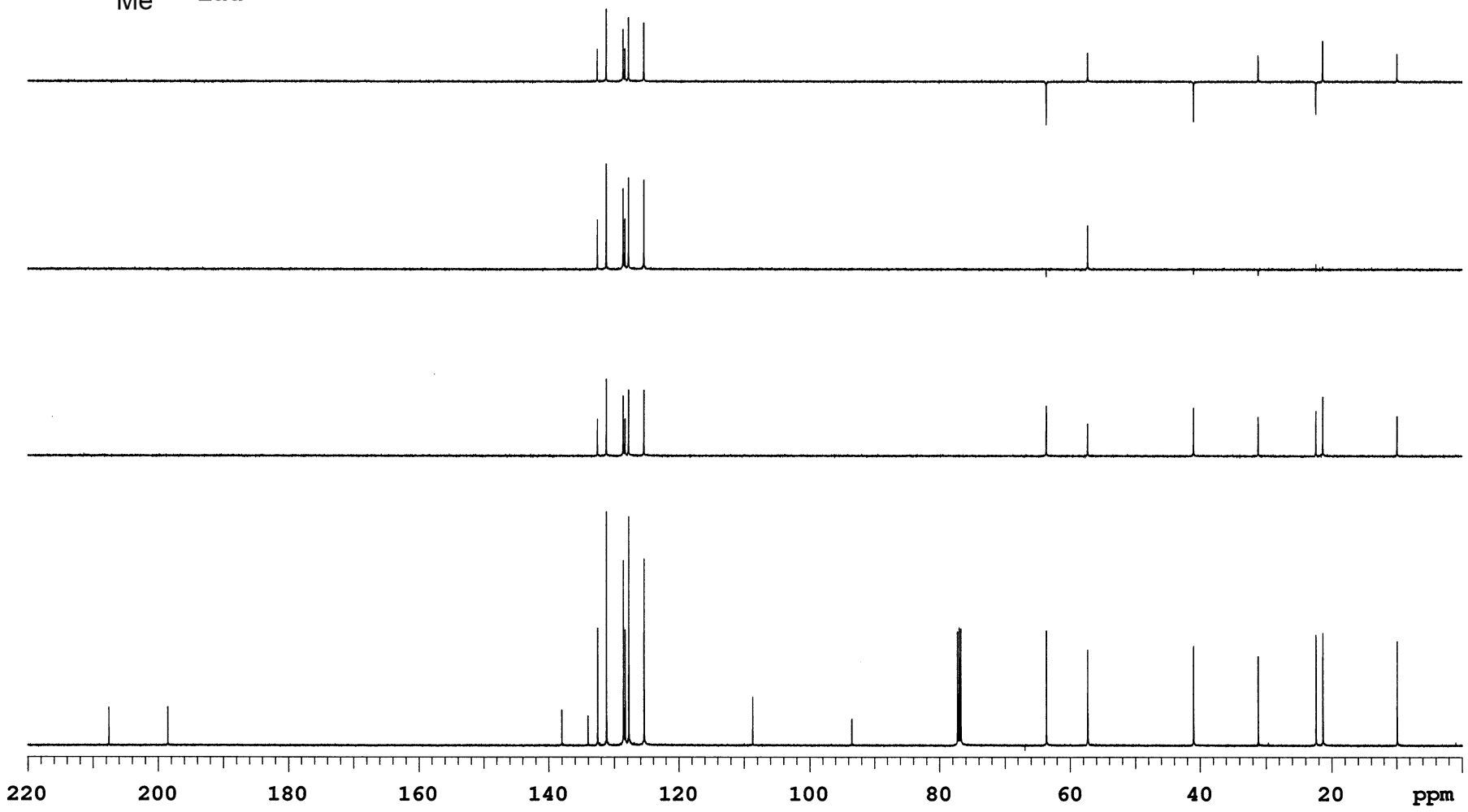
13C NMR (125 MHz, CDCl3) of compound 2ad

IRR-02-218

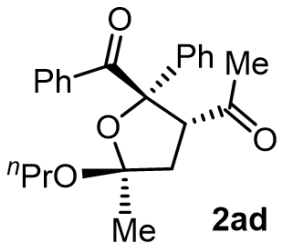
Sample Name	**IRR-02-218**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2ad



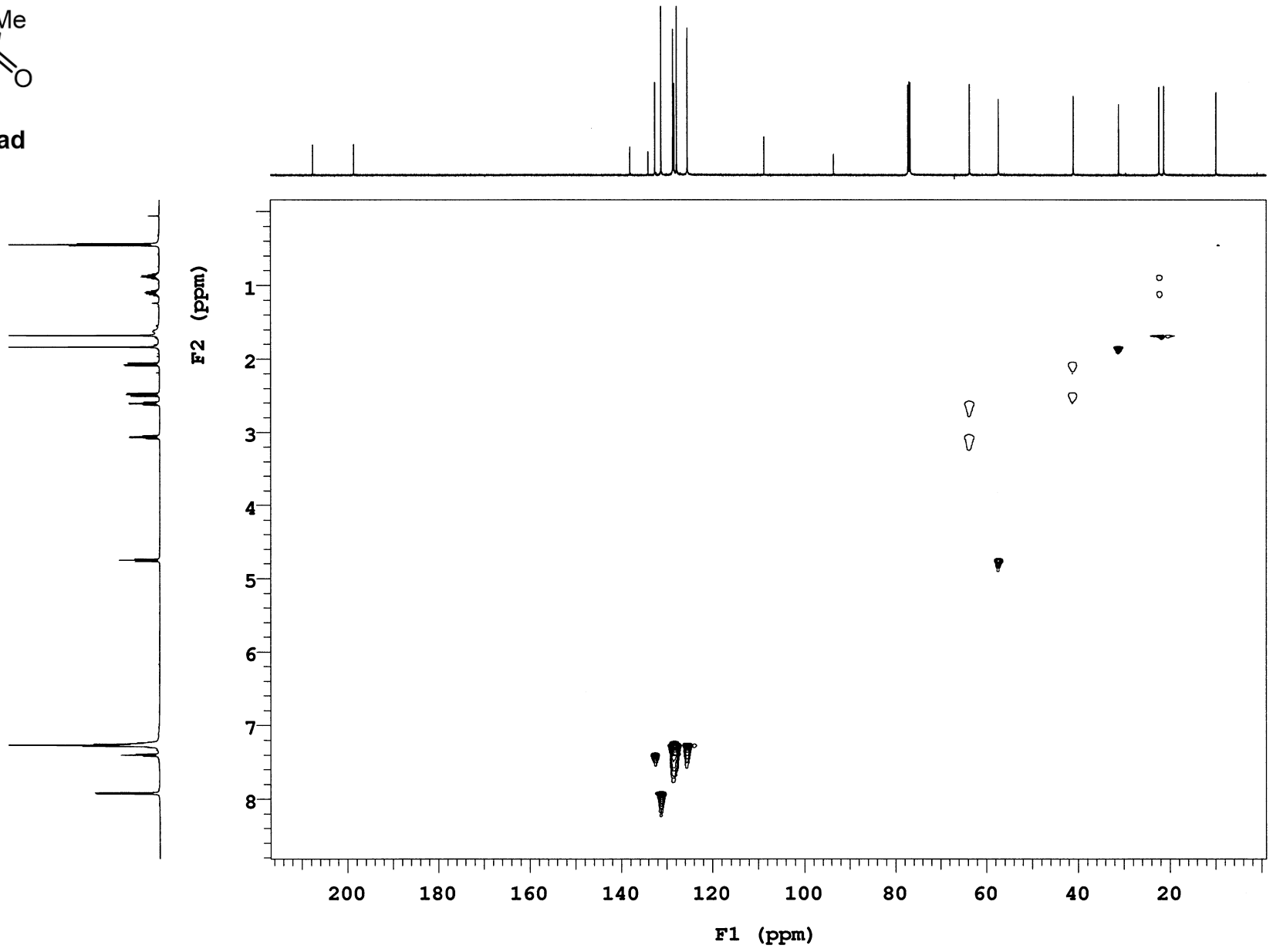
DEPT of compound 2ad



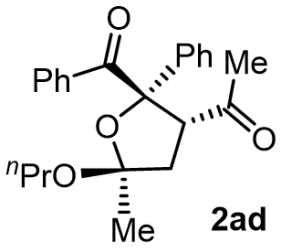
2ad

IRR-02-218

Sample Name	**IRR-02-218**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



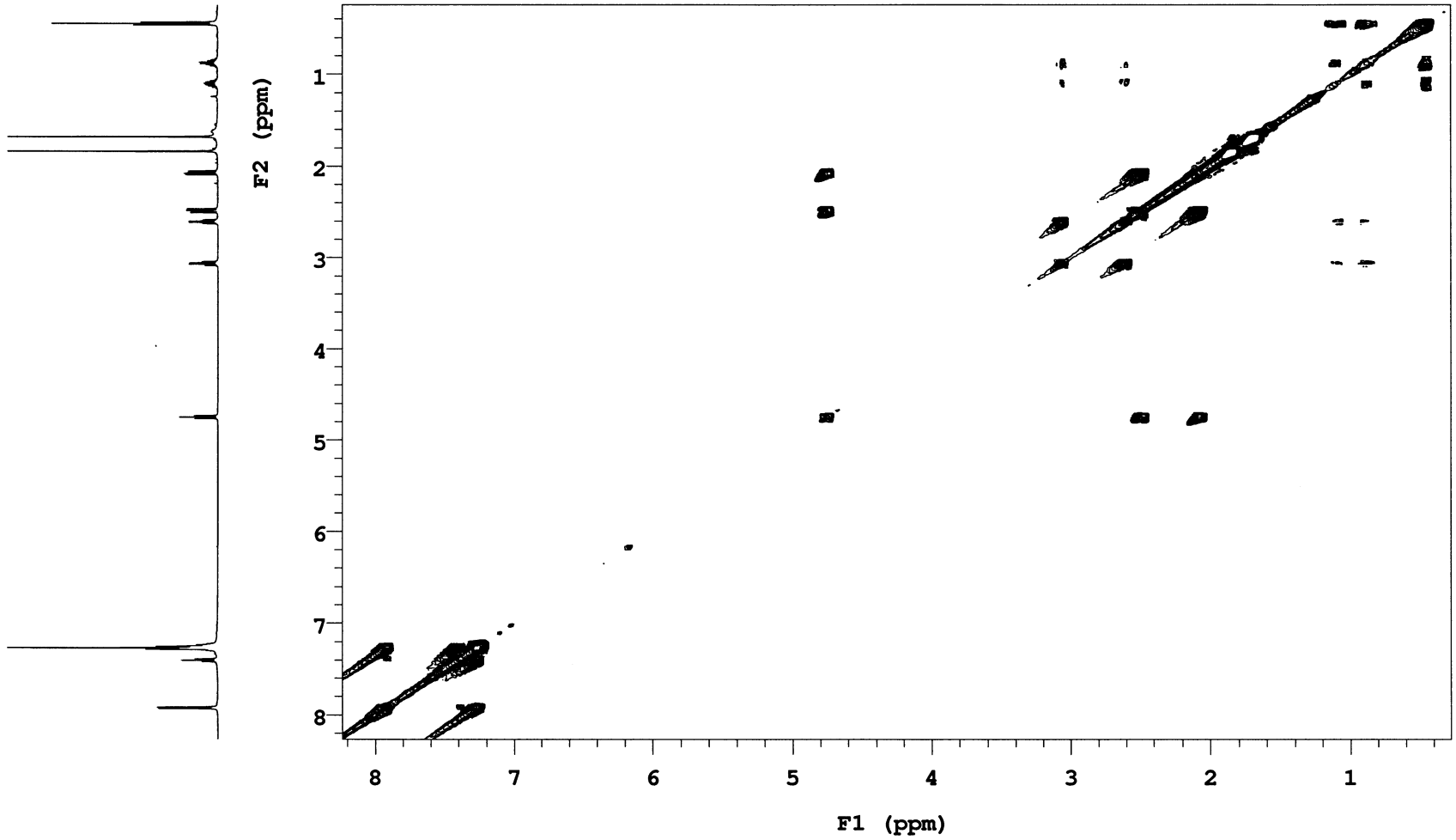
HSQC of compound 2ad



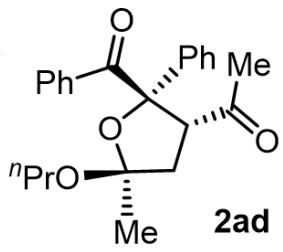
2ad

IRR-02-218

Sample Name	**IRR-02-218**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



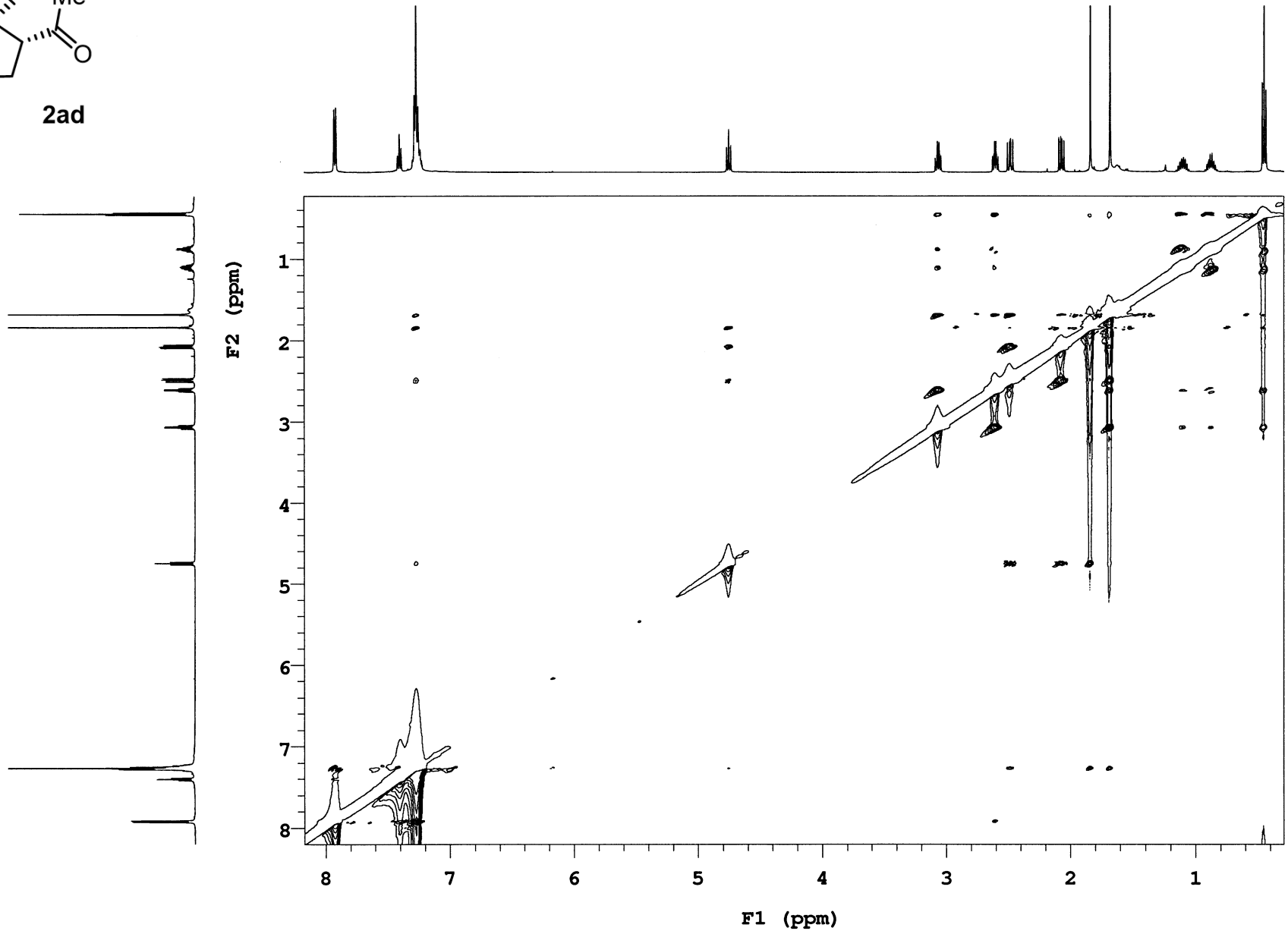
COSY of compound 2ad



2ad

IRR-02-218

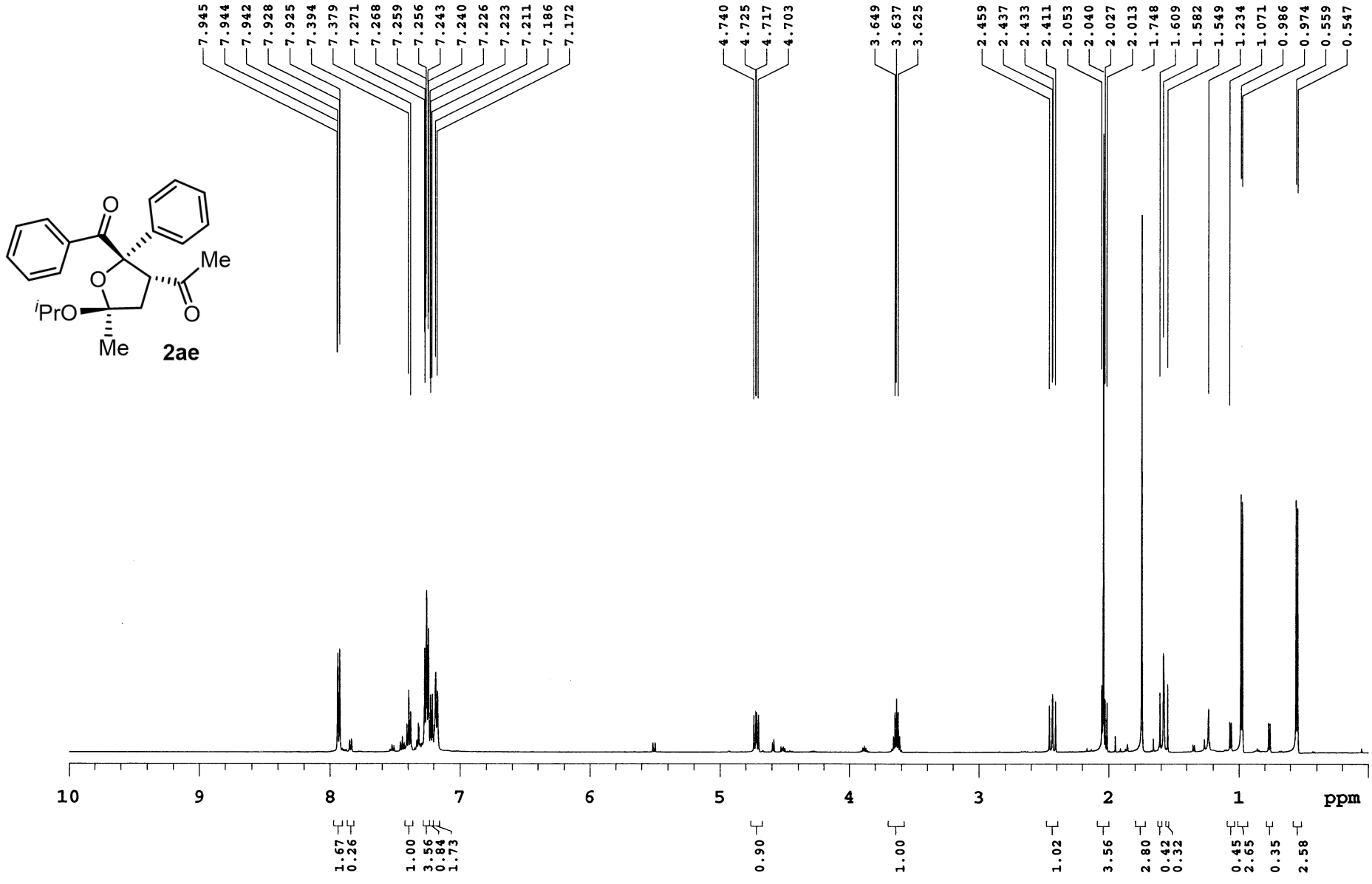
Sample Name	**IRR-02-218**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2ad

IRR-02-219

Sample Name **IRR-02-219**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-09-24**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



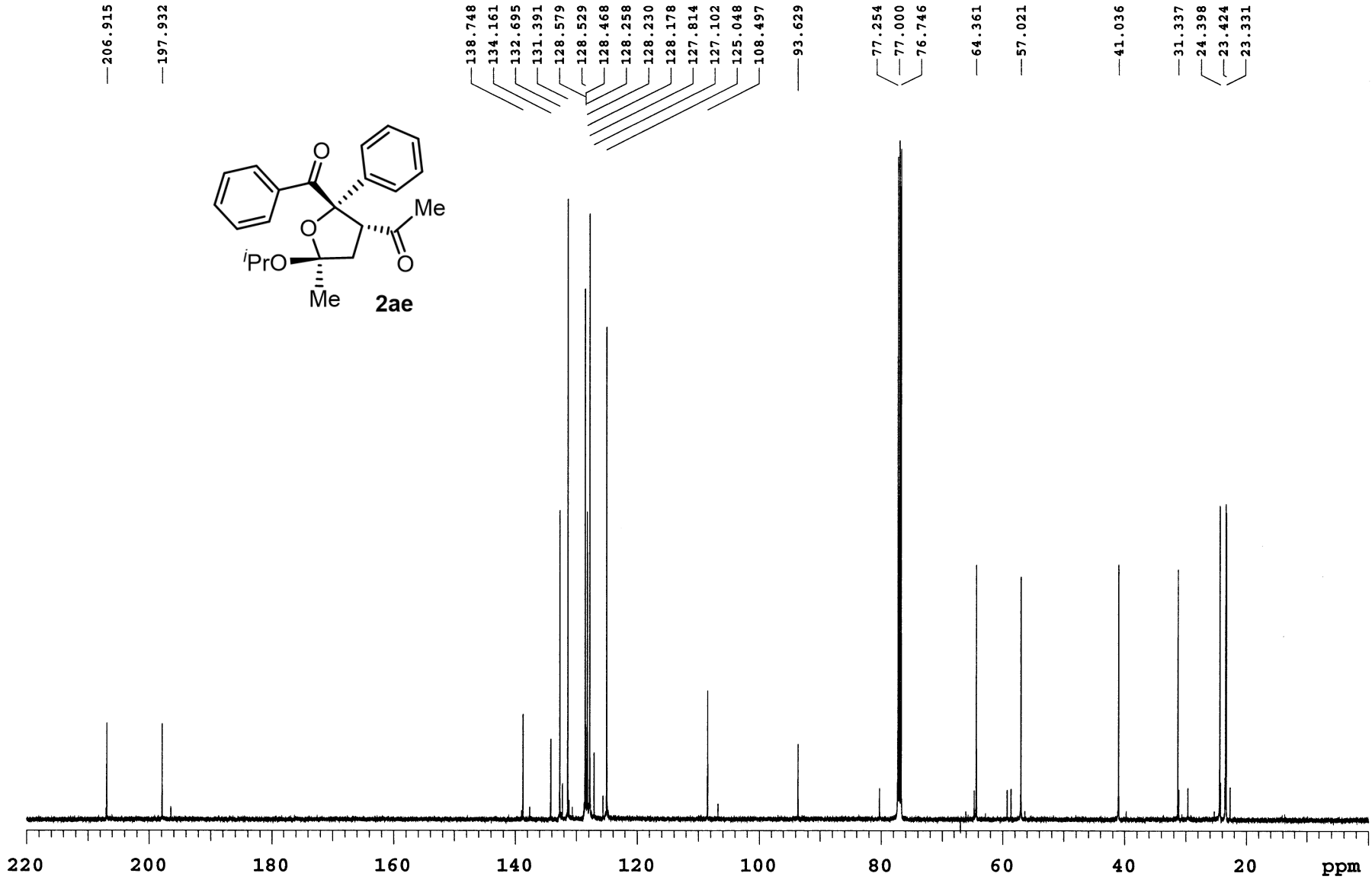
1H NMR (500 MHz, CDCl3) of compound 2ae

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-219/PROTON_04

Plot date 2024-09-25

IRR-02-219

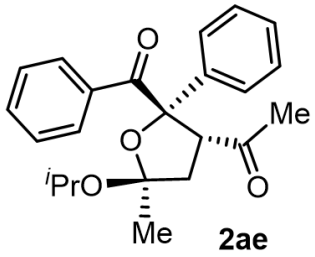
Sample Name	**IRR-02-219**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



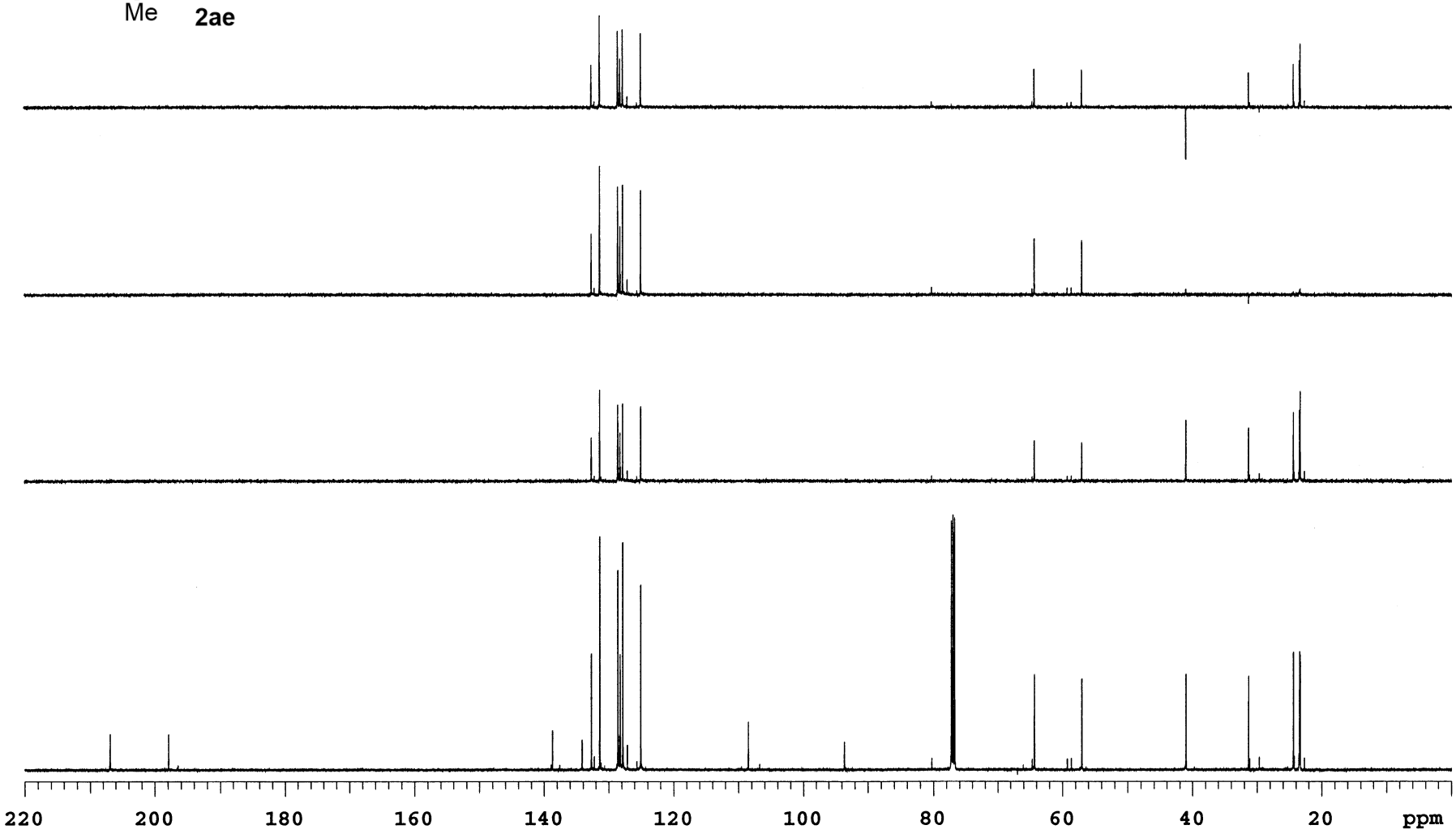
13C NMR (125 MHz, CDCl3) of compound 2ae

IRR-02-219

Sample Name **IRR-02-219**	Pulse sequence **DEPT**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-09-25**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



2ae



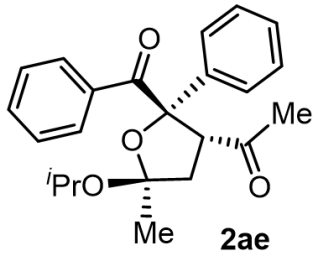
DEPT of compound 2ae

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-219/DEPT_01

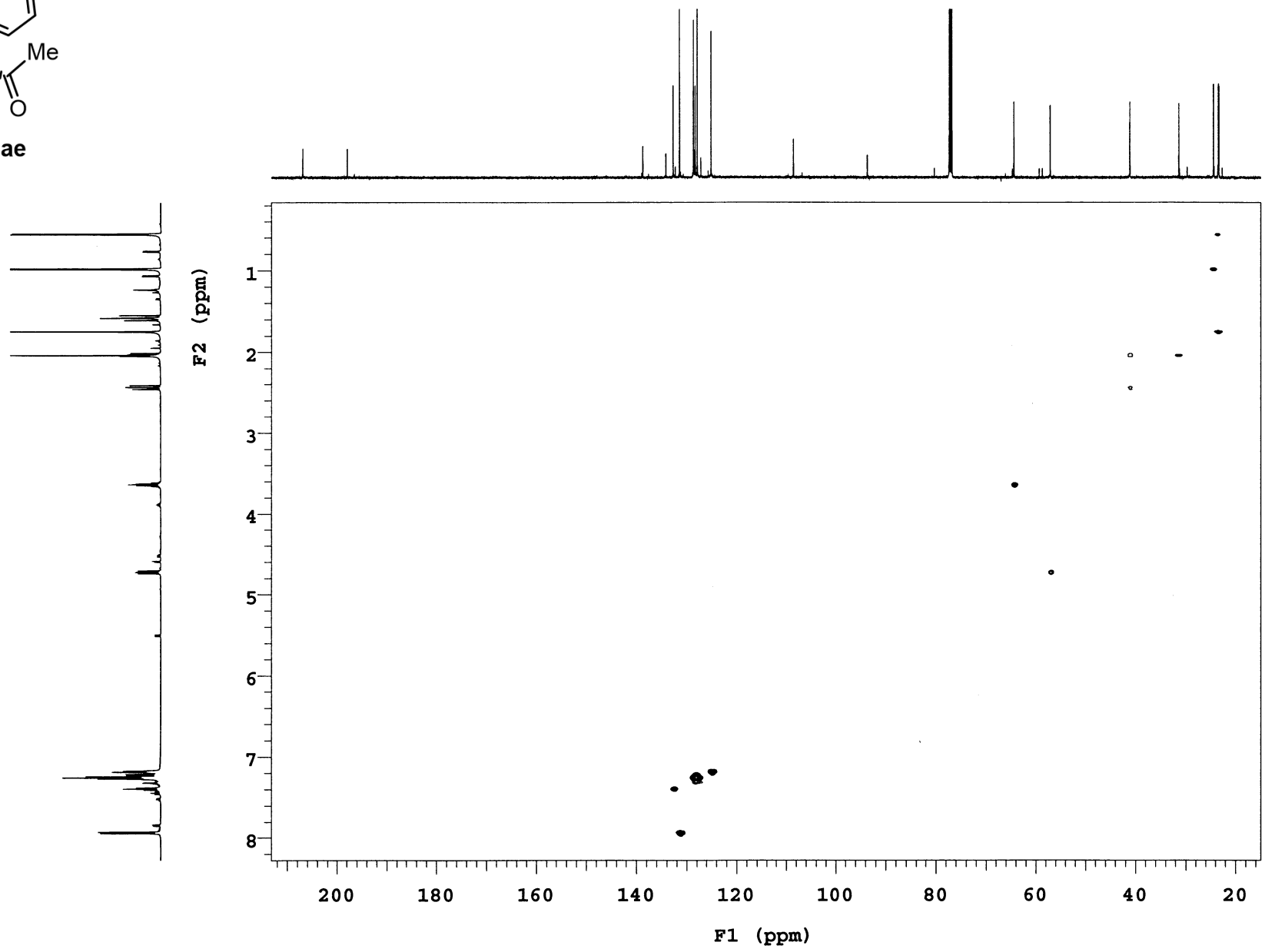
Plot date 2024-09-25

IRR-02-219

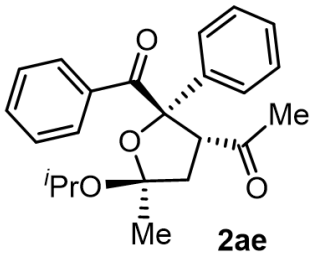
Sample Name	**IRR-02-219**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-25**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2ae



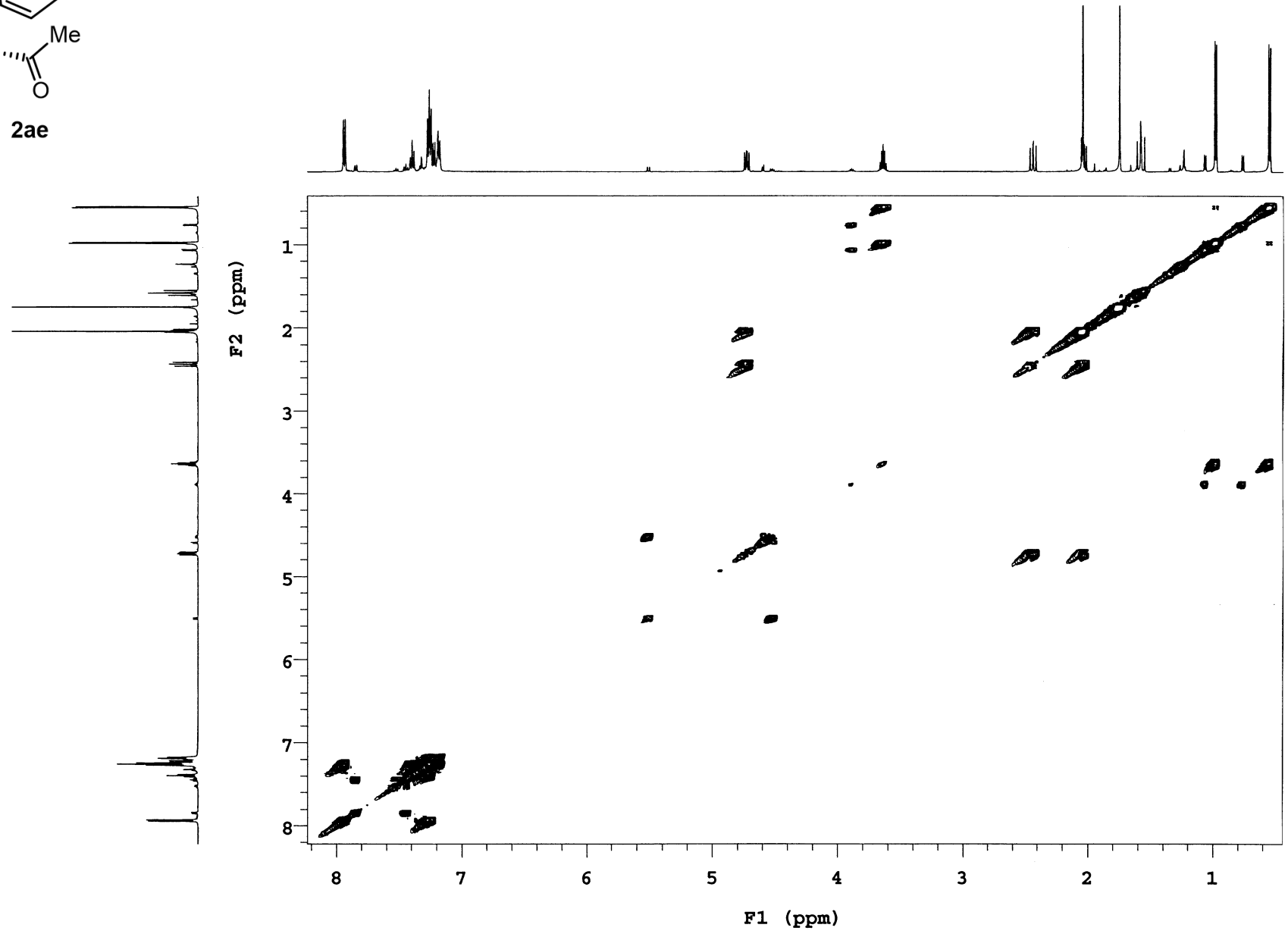
HSQC of compound 2ae



2ae

IRR-02-219

Sample Name	IRR-02-219	Pulse sequence	gCOSY	Temperature	25	Study owner	vnmr2
Date collected	2024-09-25	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



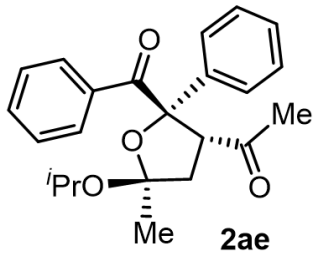
COSY of compound 2ae

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-219/gCOSY_01

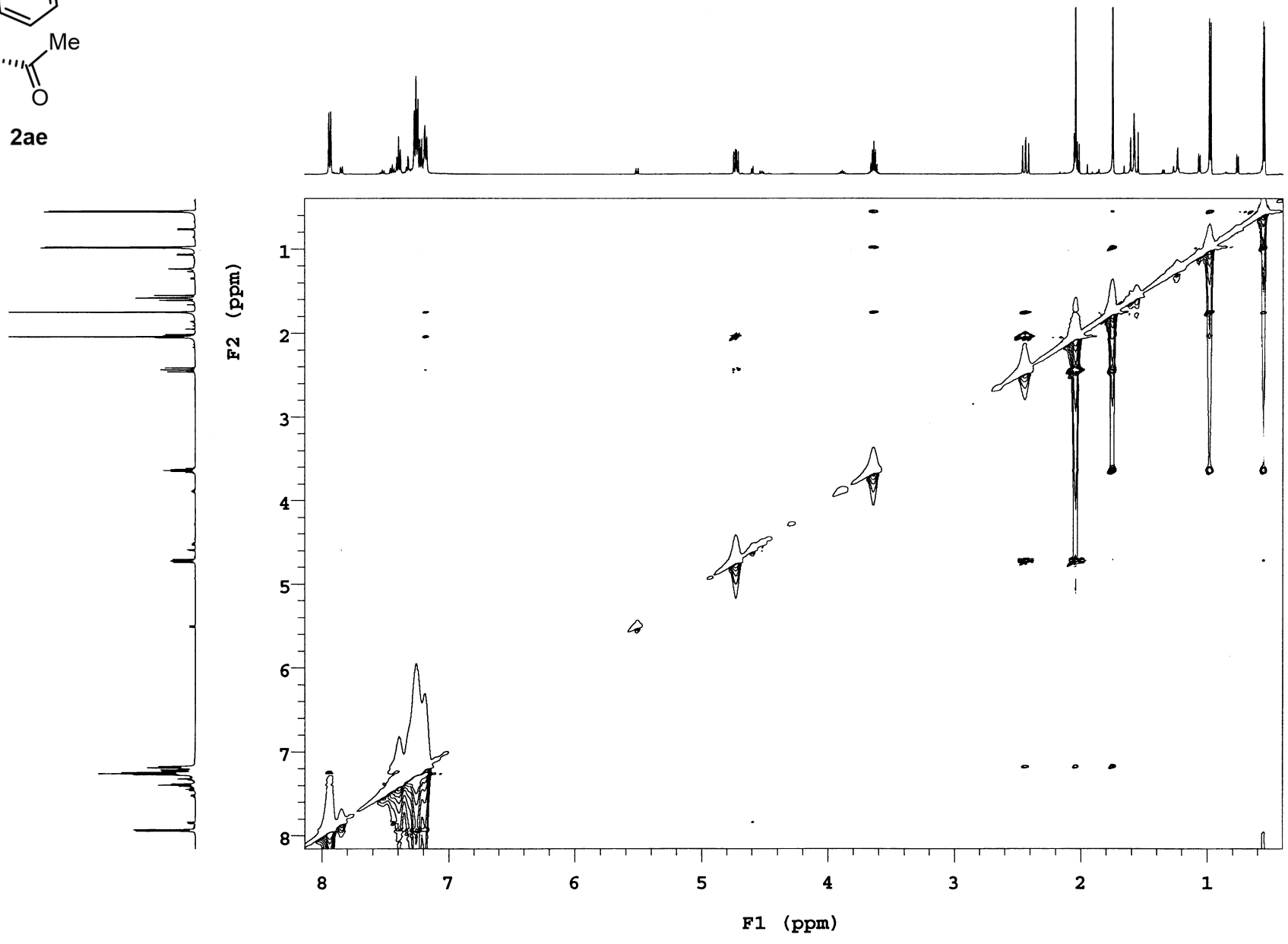
Plot date 2024-09-25

IRR-02-219

Sample Name	**IRR-02-219**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-25**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



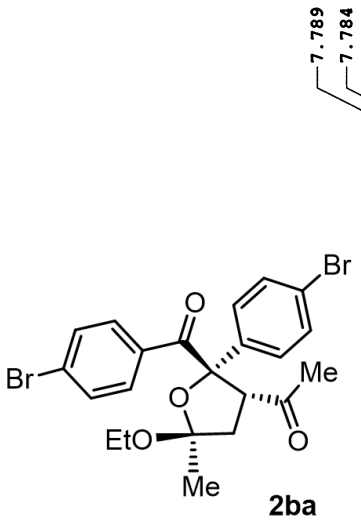
2ae



NOESY of compound 2ae

IRR-02-156

Sample Name	**IRR-02-156**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2ba

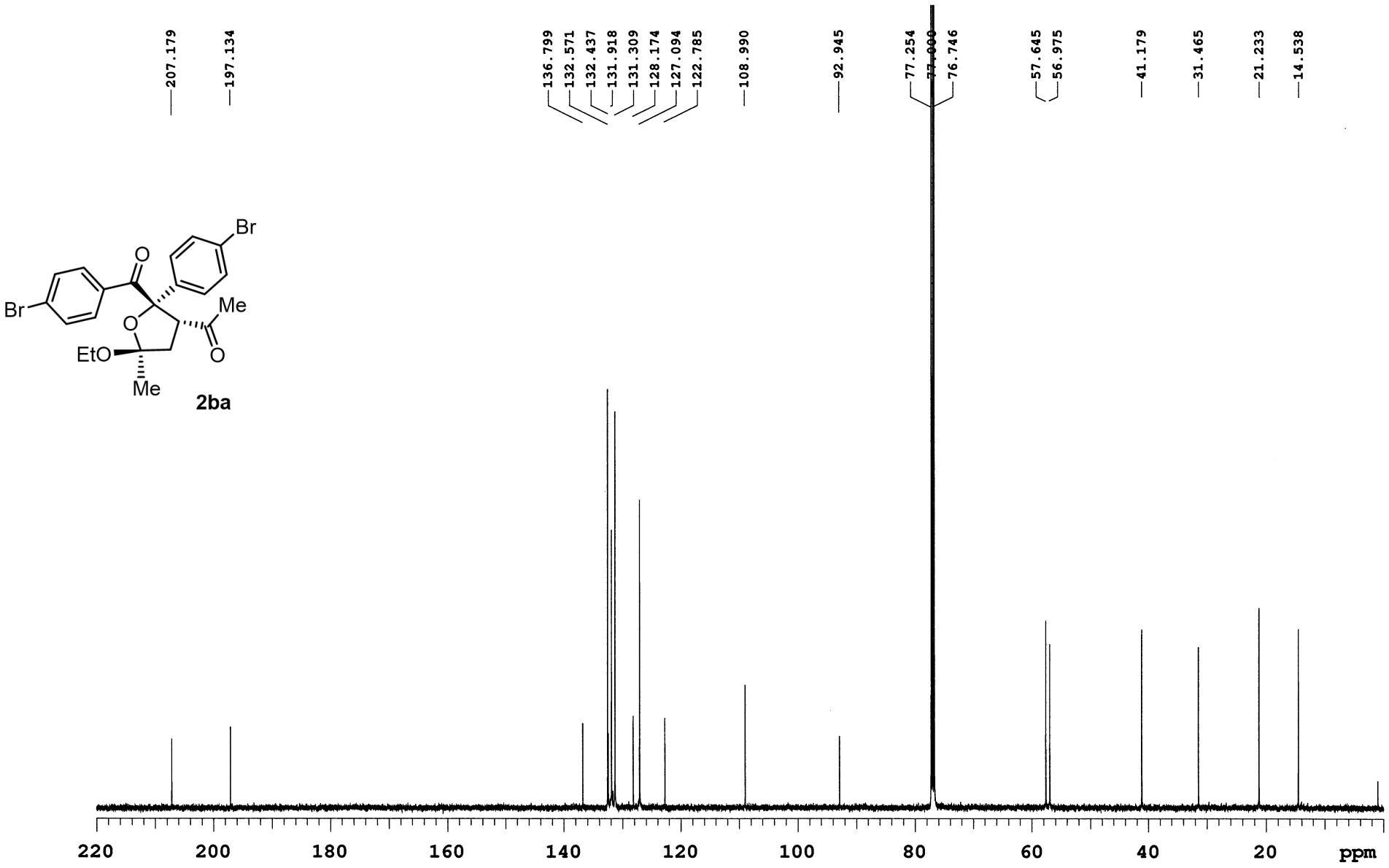
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-156/PROTON_10

1H NMR (500 MHz, CDCl3) of compound 2ba

Plot date 2023-11-29

IRR-02-156

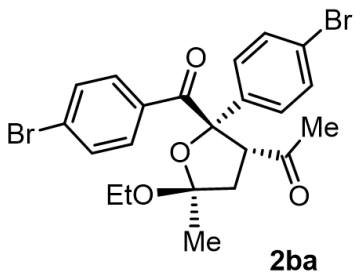
Sample Name	**IRR-02-156**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



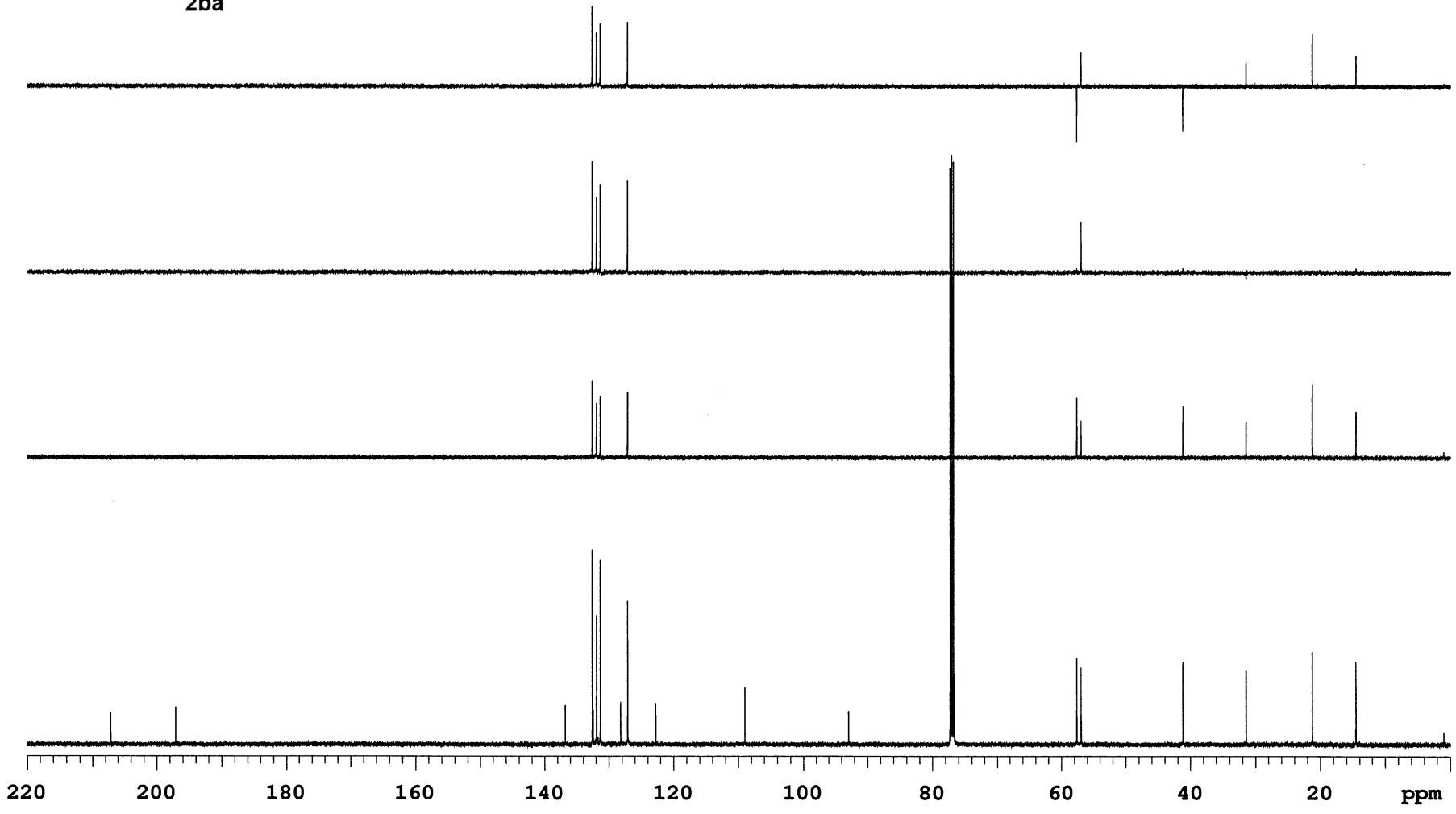
13C NMR (125 MHz, CDCl3) of compound 2ba

IRR-02-156

Sample Name	**IRR-02-156**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



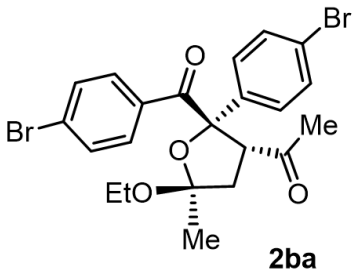
2ba



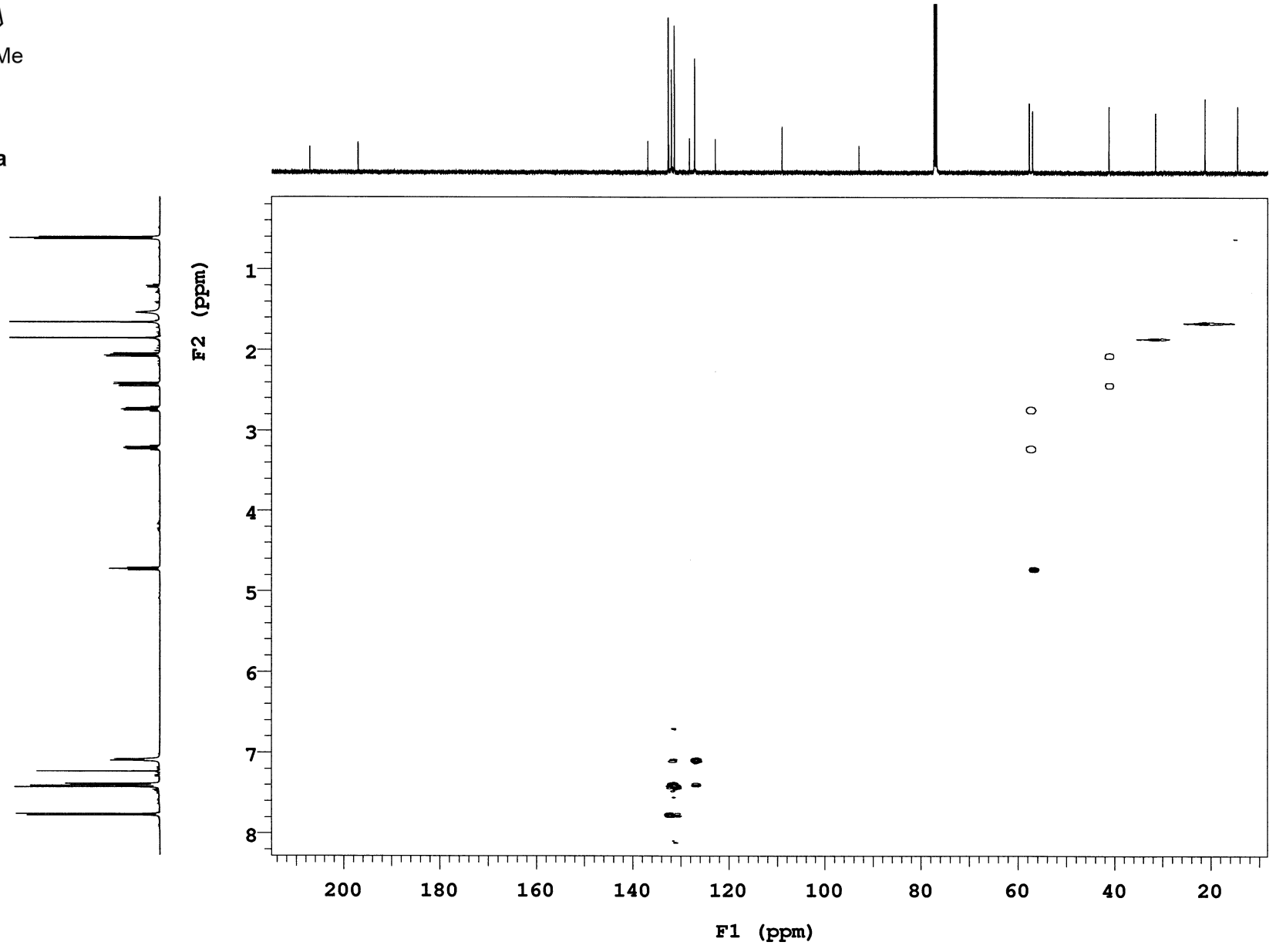
DEPT of compound 2ba

IRR-02-156

Sample Name	**IRR-02-156**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-29**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



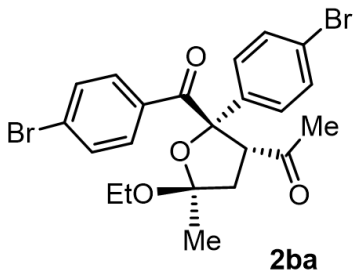
2ba



HSQC of compound 2ba

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-156/gHSQC_03

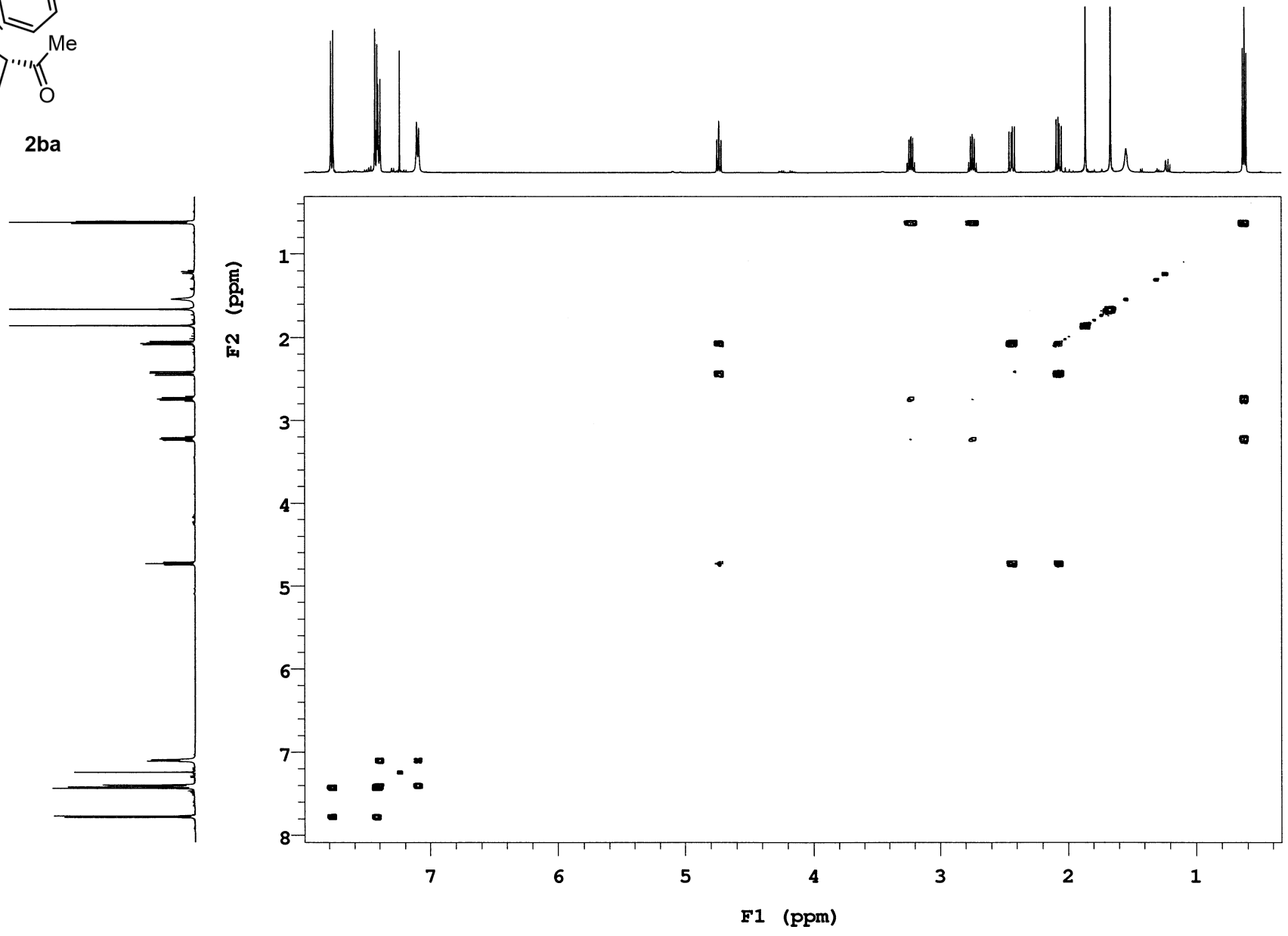
Plot date 2023-11-29



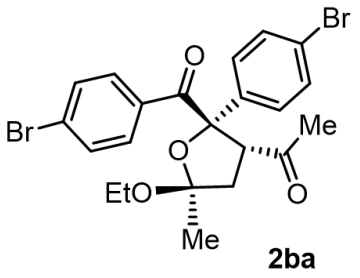
2ba

IRR-02-156

Sample Name	**IRR-02-156**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-29**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



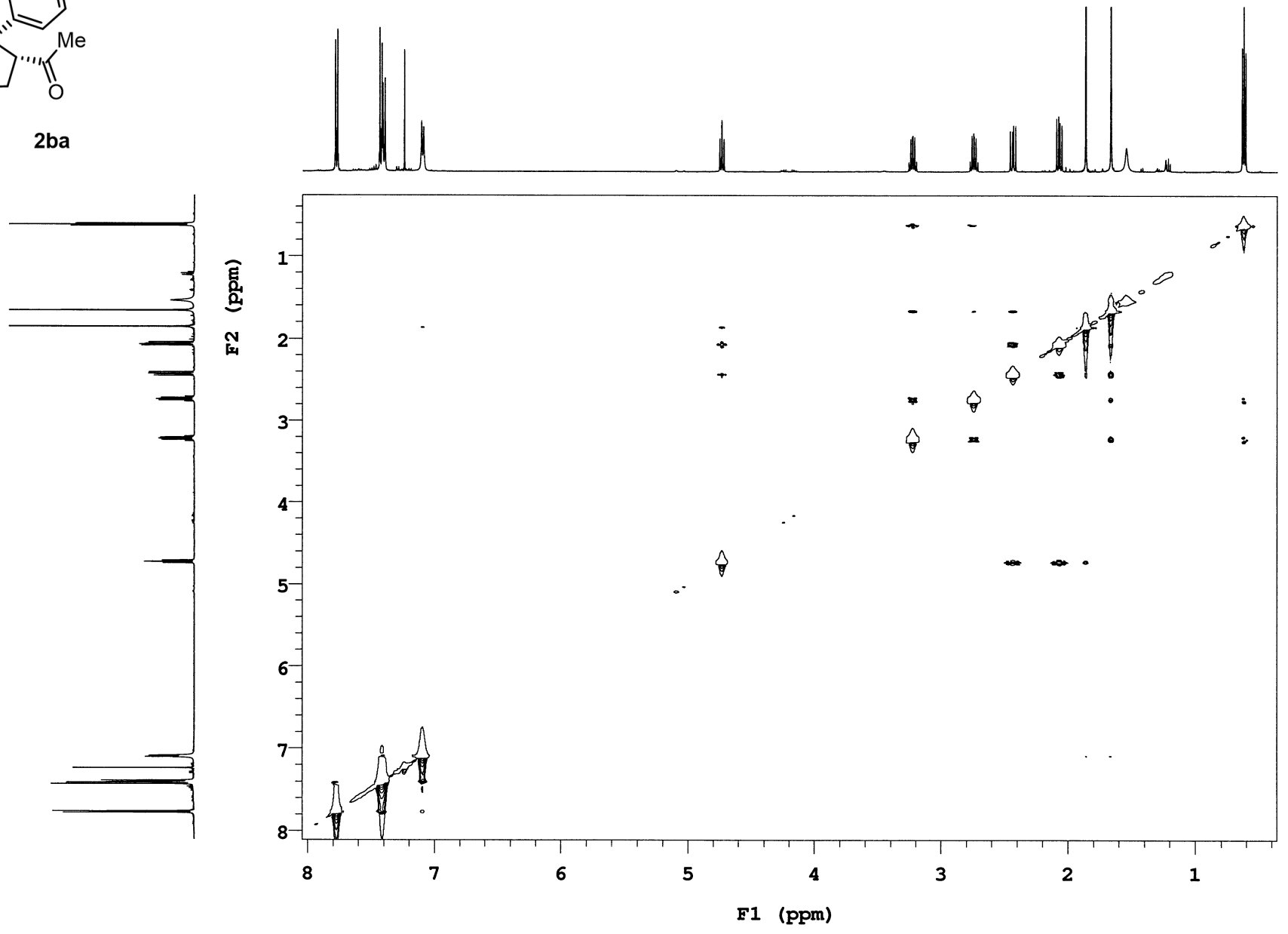
COSY of compound 2ba



2ba

IRR-02-156

Sample Name	**IRR-02-156**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2023-11-29**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



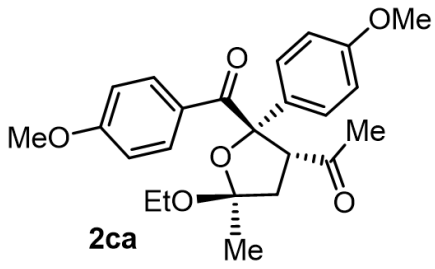
NOESY of compound 2ba

IRR-120-N

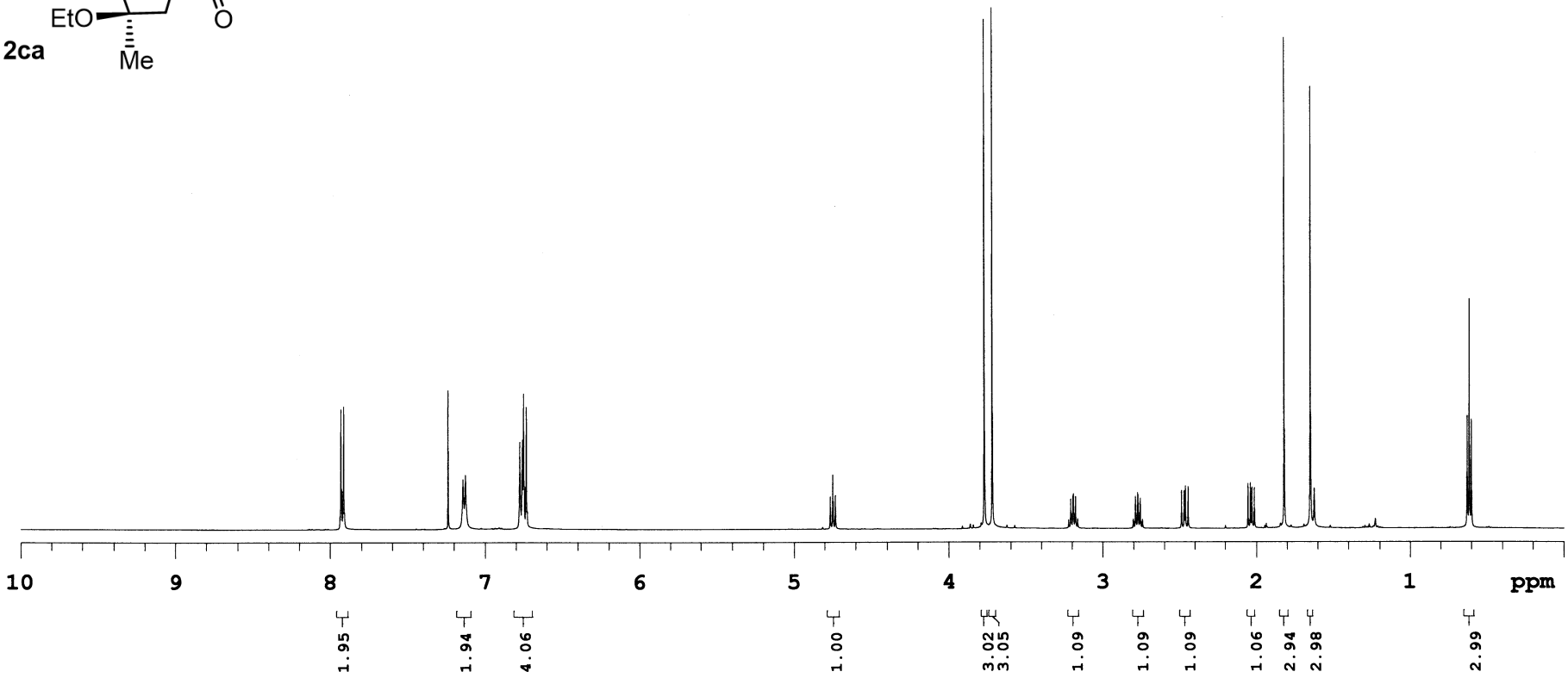
Sample Name	**IRR-120-N**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-07-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

7.938 7.932 7.929 7.918 7.915 7.909 7.240 7.144 7.127 6.777 6.759 6.752 6.734 6.729

4.767 4.750 4.733 3.773 3.721 3.224 3.210 3.196 3.192 3.179 3.165 2.803 2.789 2.774 2.771 2.757 2.743 2.489 2.471 2.463 2.445 2.055 2.039 2.029 2.014 1.820 1.649 1.624 1.228 0.630 0.616 0.602



2ca



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-120-N/PROTON_04

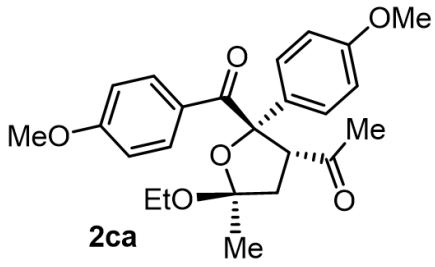
1H NMR (500 MHz, CDCl3) of compound 2ca

Plot date 2024-07-09

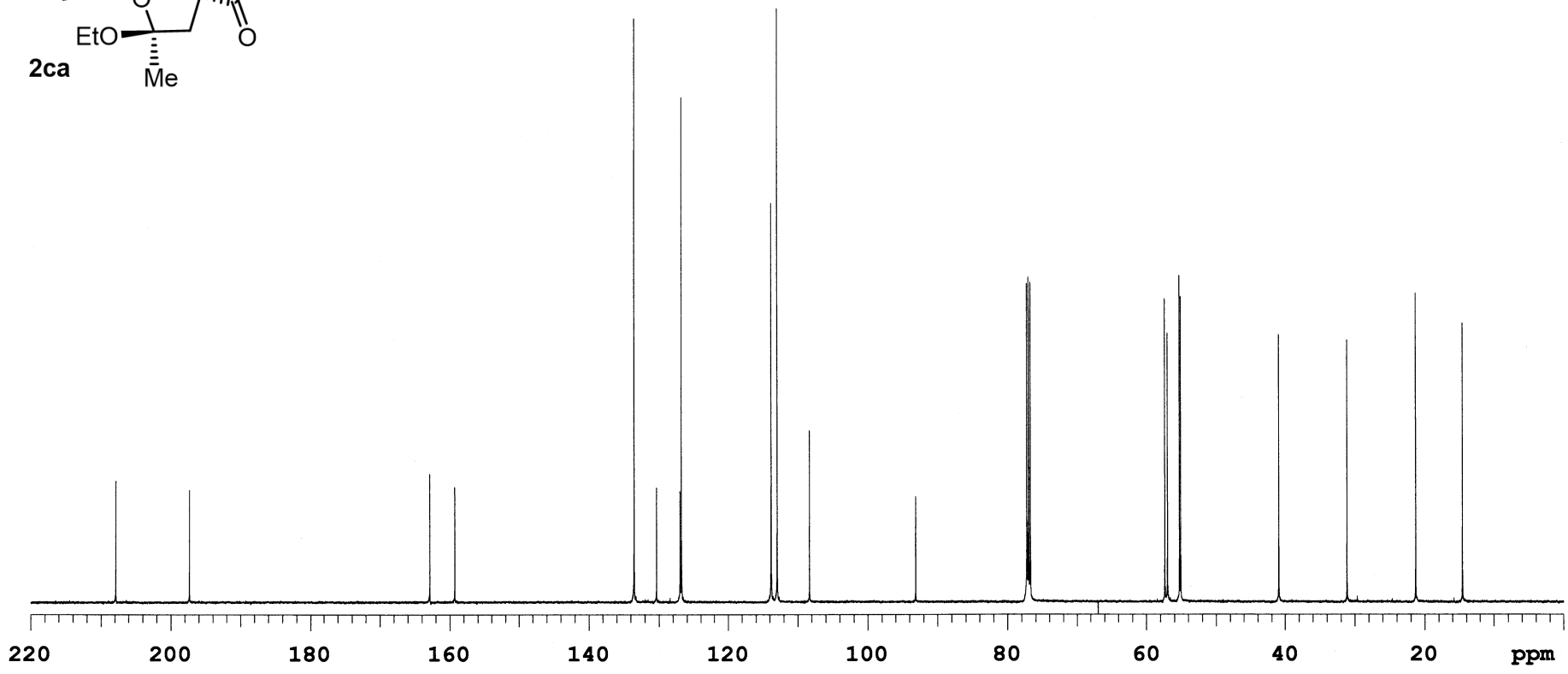
IRR-120-N

Sample Name	**IRR-120-N**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-07-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

207.872, 197.377, 162.875, 159.296, 133.514, 130.330, 126.991, 126.781, 113.825, 112.962, 108.320, 93.165, 77.254, 77.000, 76.746, 57.372, 56.987, 55.278, 55.089, 40.937, 31.150, 21.294, 14.546



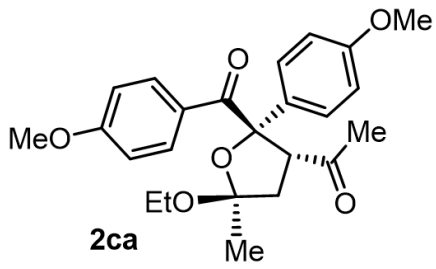
2ca



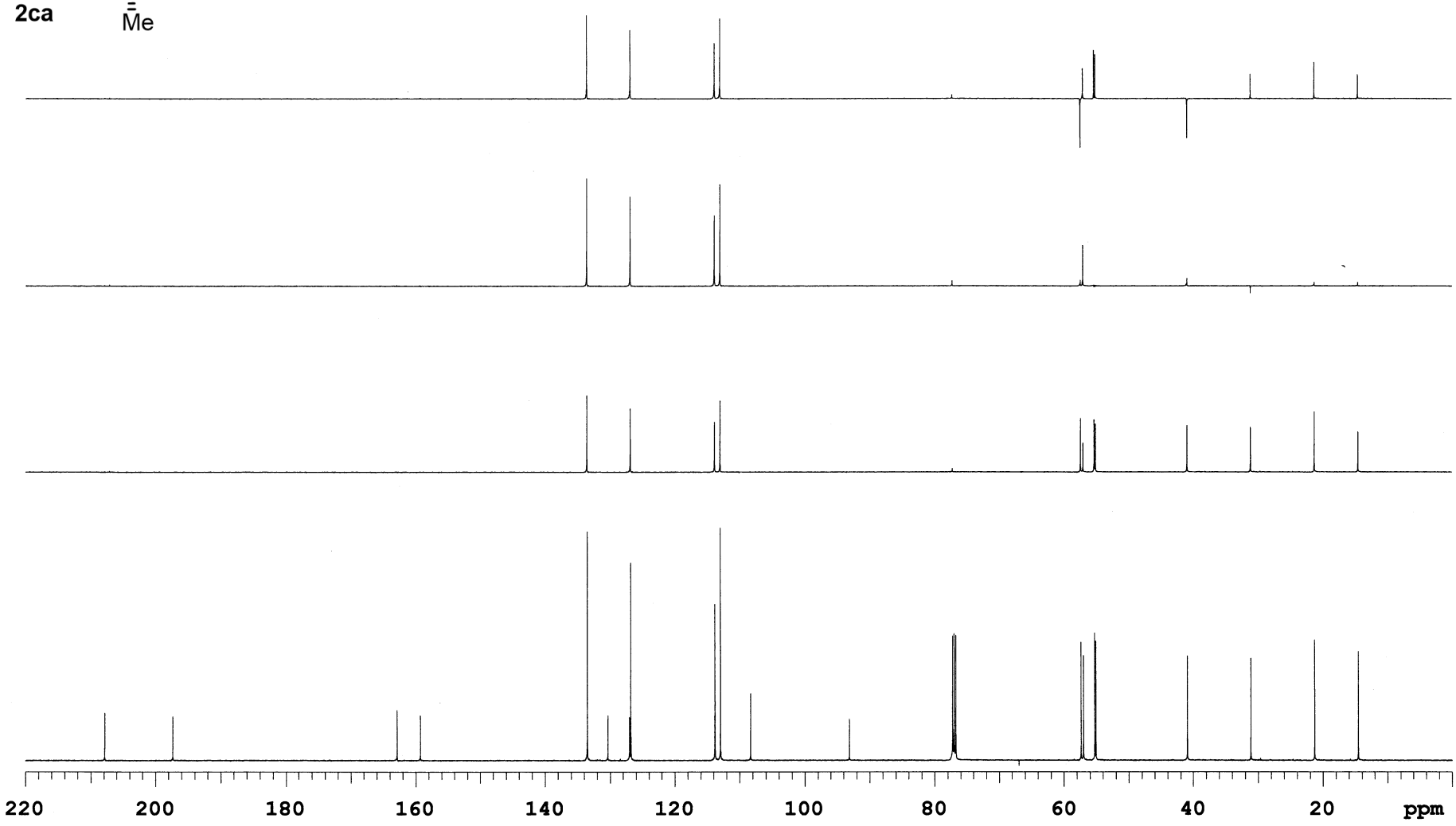
13C NMR (125 MHz, CDCl3) of compound 2ca

IRR-120-N

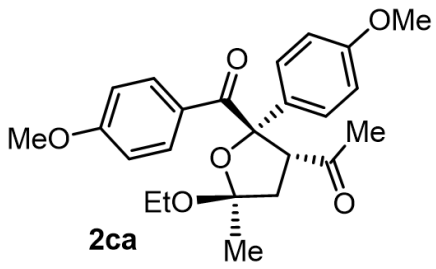
Sample Name **IRR-120-N**	Pulse sequence **DEPT**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-07-09**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



2ca

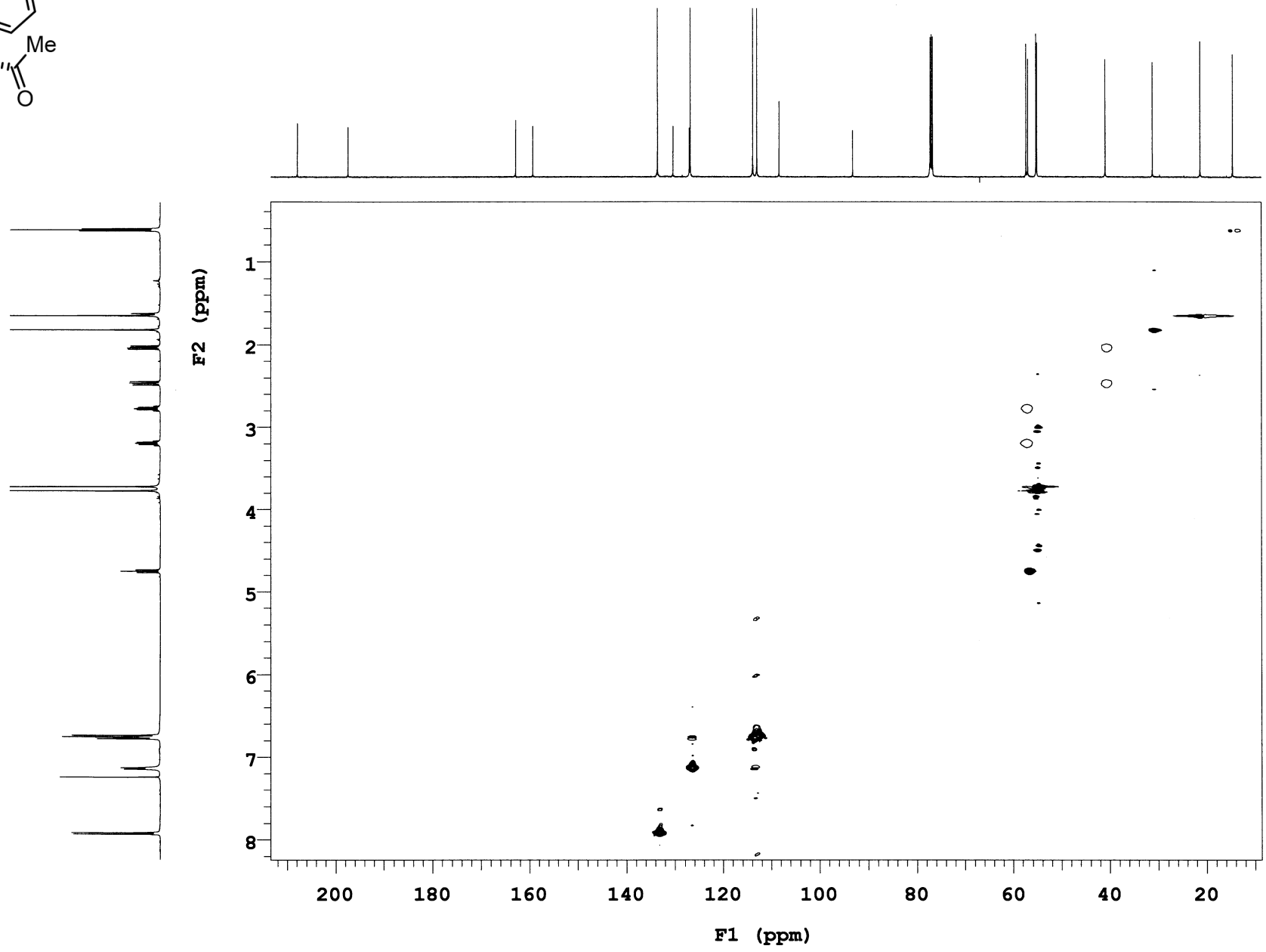


DEPT of compound 2ca

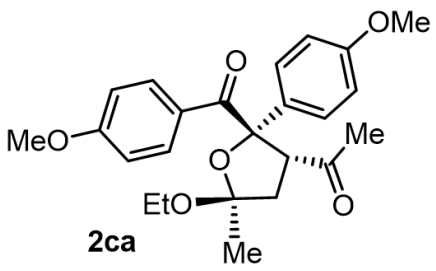


IRR-120-N

Sample Name **IRR-120-N**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-08-28**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

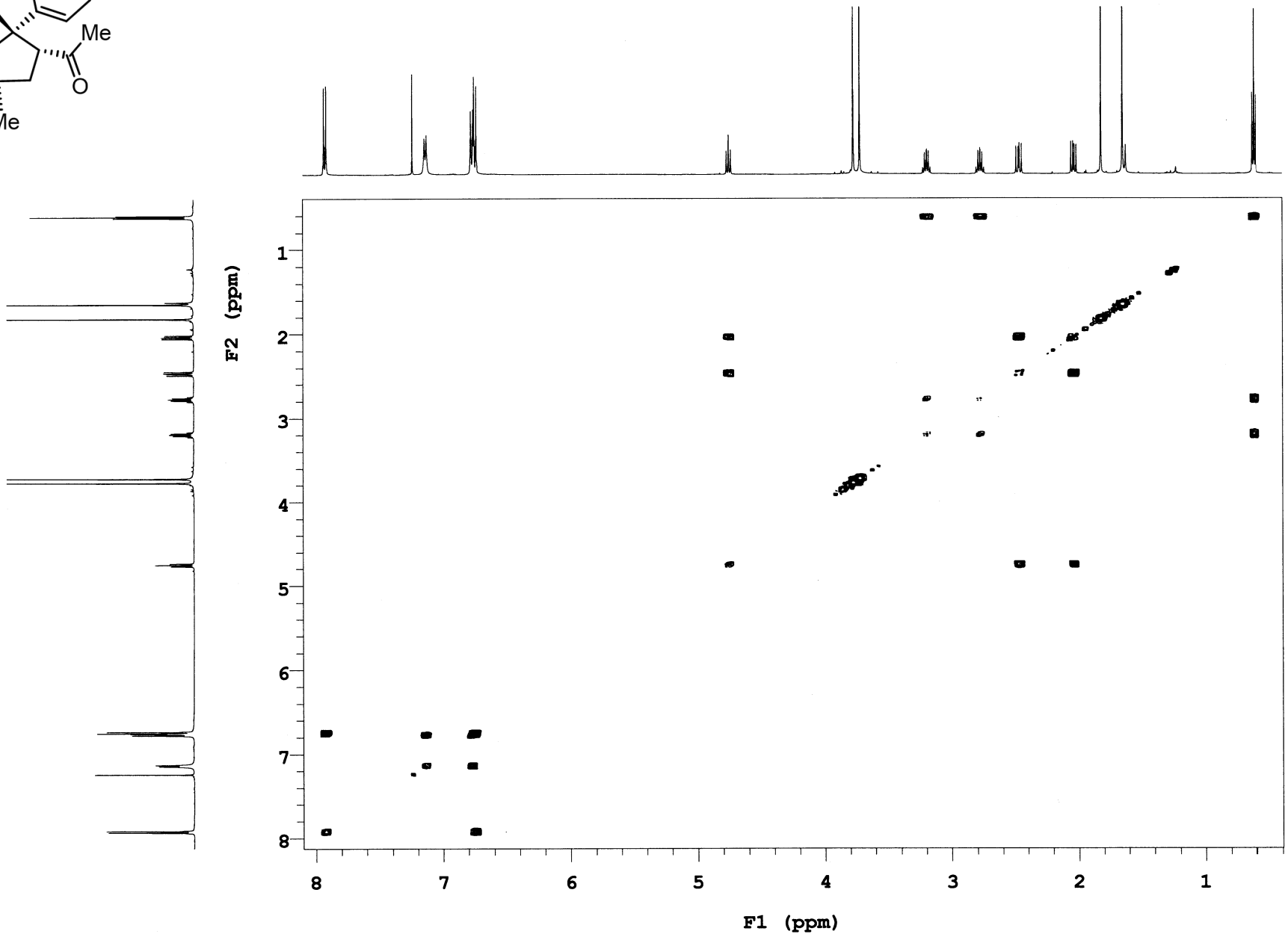


HSQC of compound 2ca

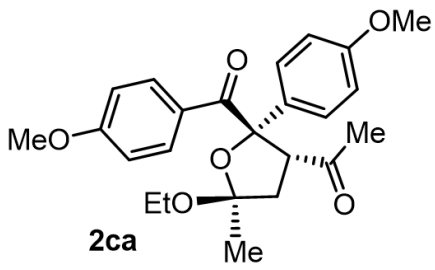


IRR-120-N

Sample Name	**IRR-120-N**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-08-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

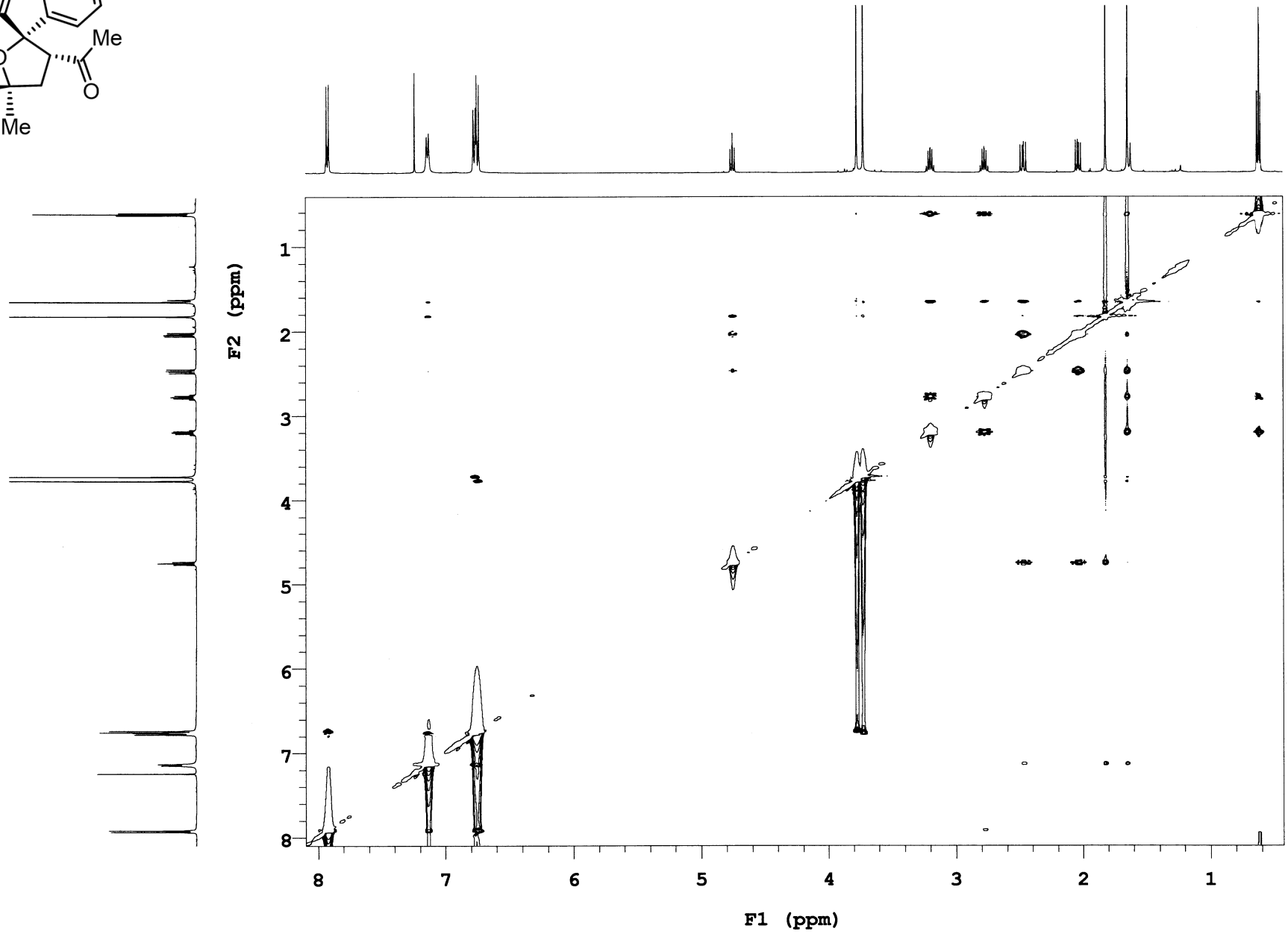


COSY of compound 2ca



IRR-120-N

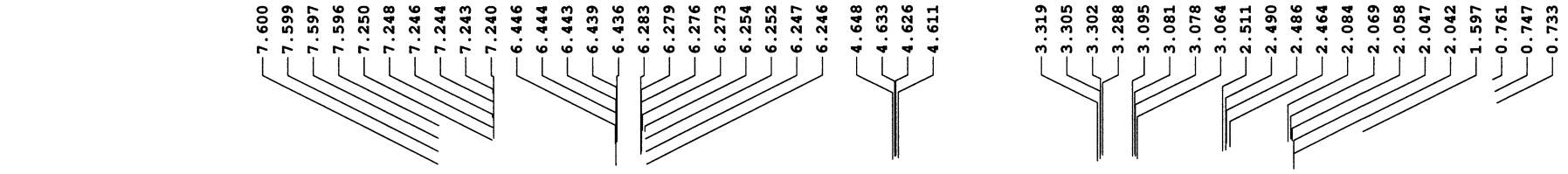
Sample Name	**IRR-120-N**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-08-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

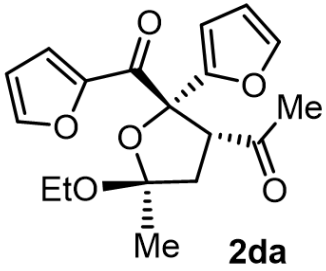


NOESY of compound 2ca

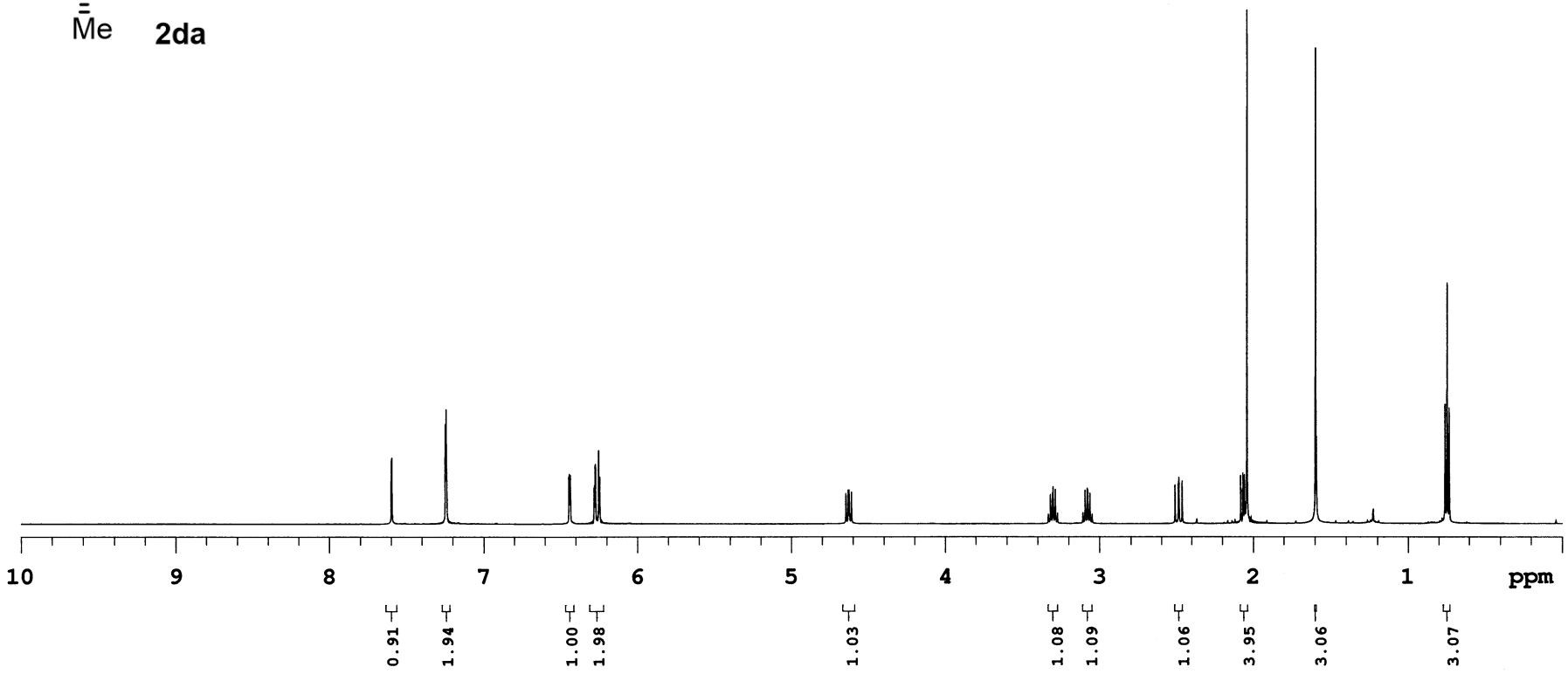
IRR-02-213-P

Sample Name	**IRR-02-213-P**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-06**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**





2da



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-213-P/PROTON_03

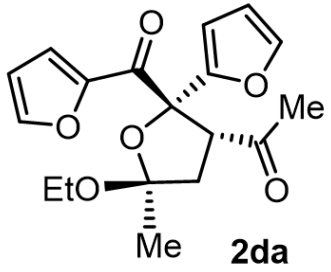
1H NMR (500 MHz, CDCl3) of compound 2da

Plot date 2024-03-07

IRR-02-213-P

Sample Name	**IRR-02-213-P**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-06**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

206.202 184.881 151.038 149.911 147.071 142.919 121.907 111.996 110.727 108.520 108.364 88.326 77.256 77.000 76.746 57.451 55.187 40.270 30.272 21.183 14.555

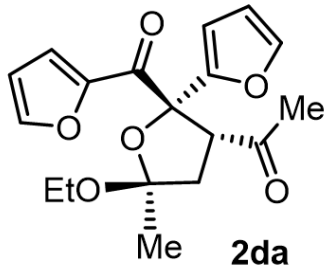


2da

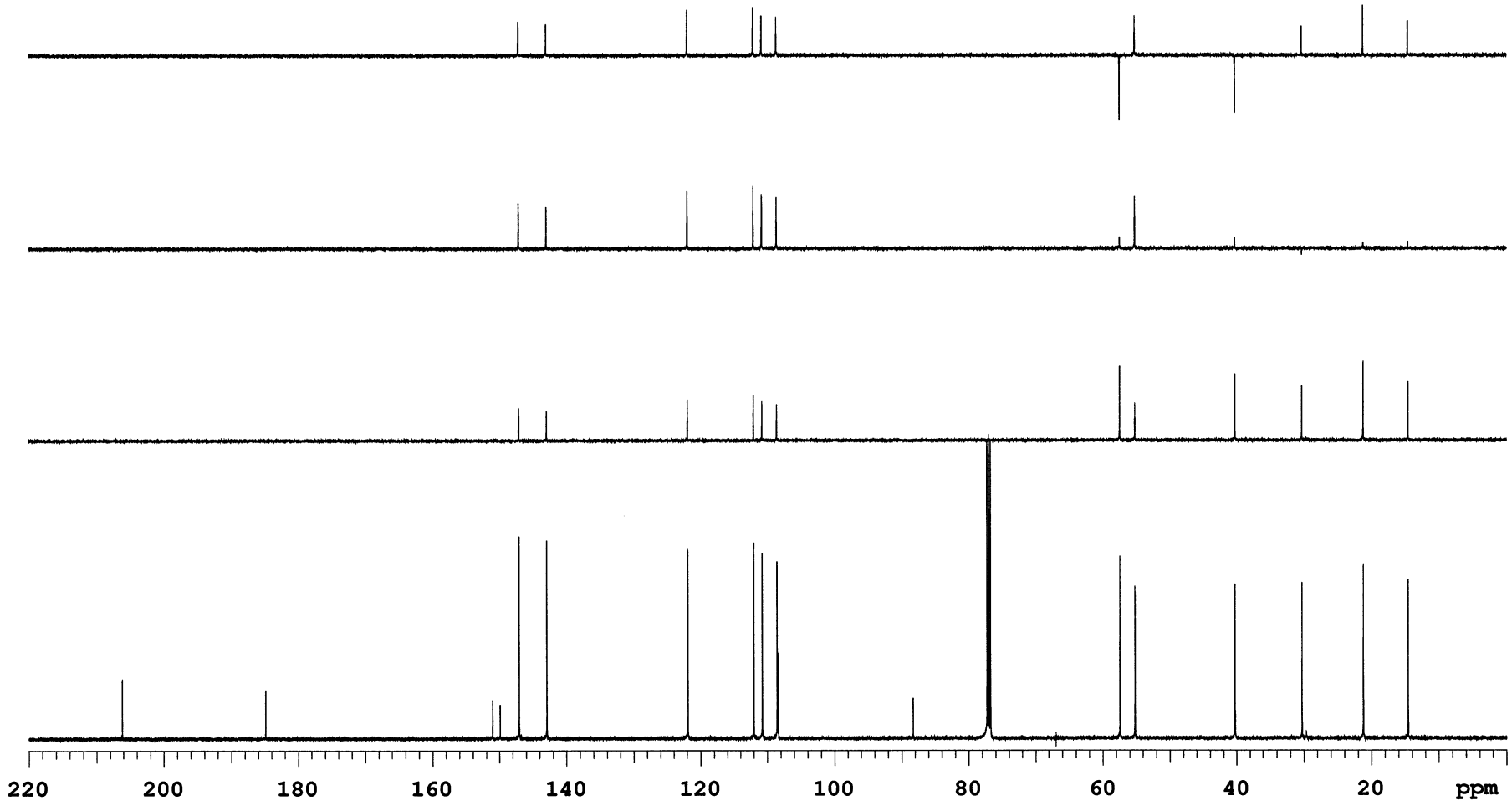
13C NMR (125 MHz, CDCl3) of compound 2da

IRR-02-213-P

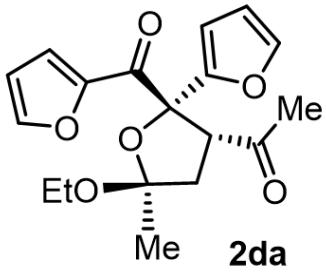
Sample Name	**IRR-02-213-P**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-07**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2da



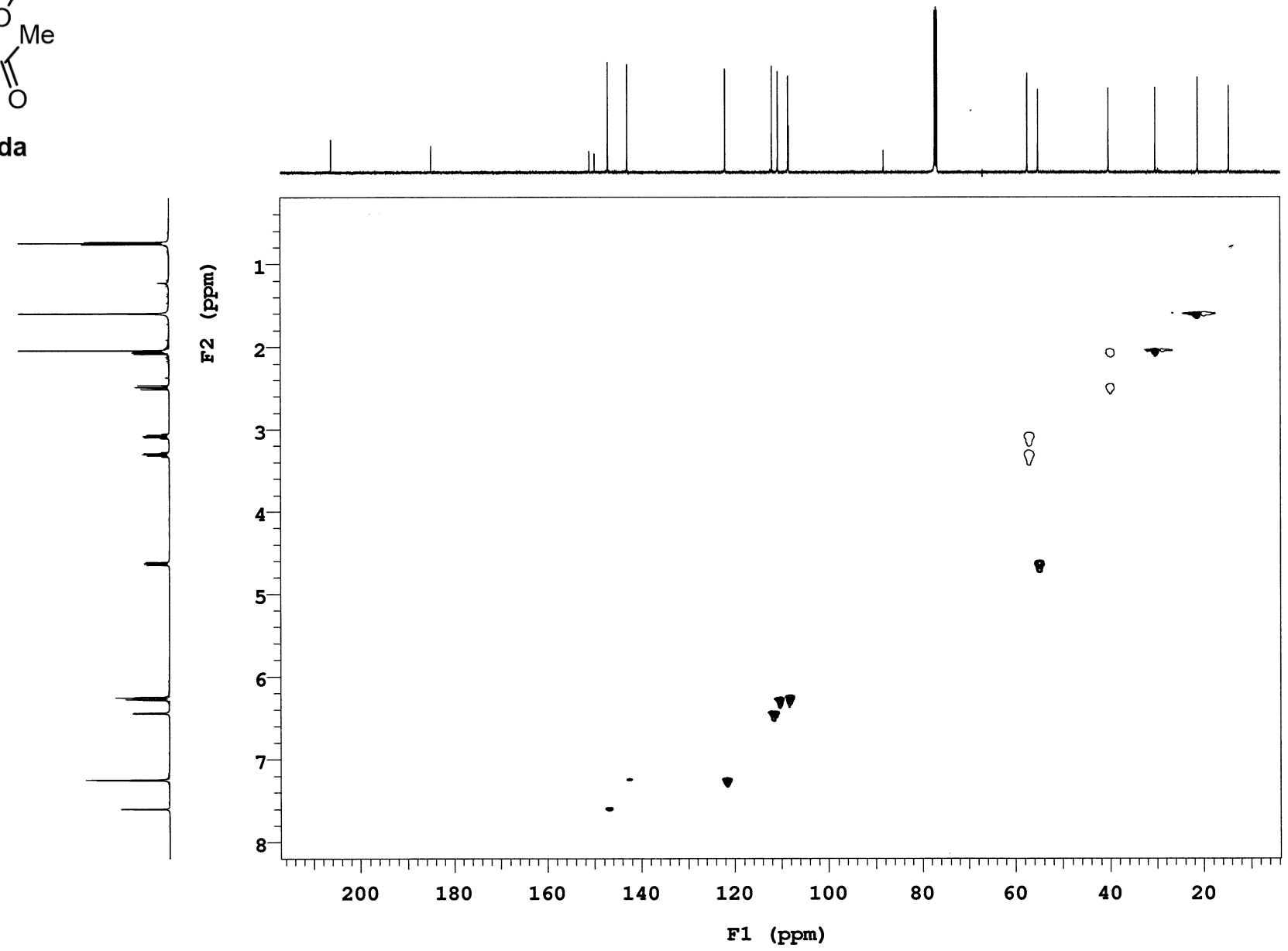
DEPT of compound 2da



2da

IRR-02-213-P

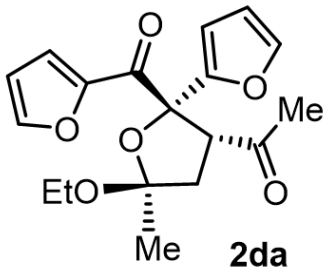
Sample Name	**IRR-02-213-P**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-07**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2da

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-213-P/gHSQC_01

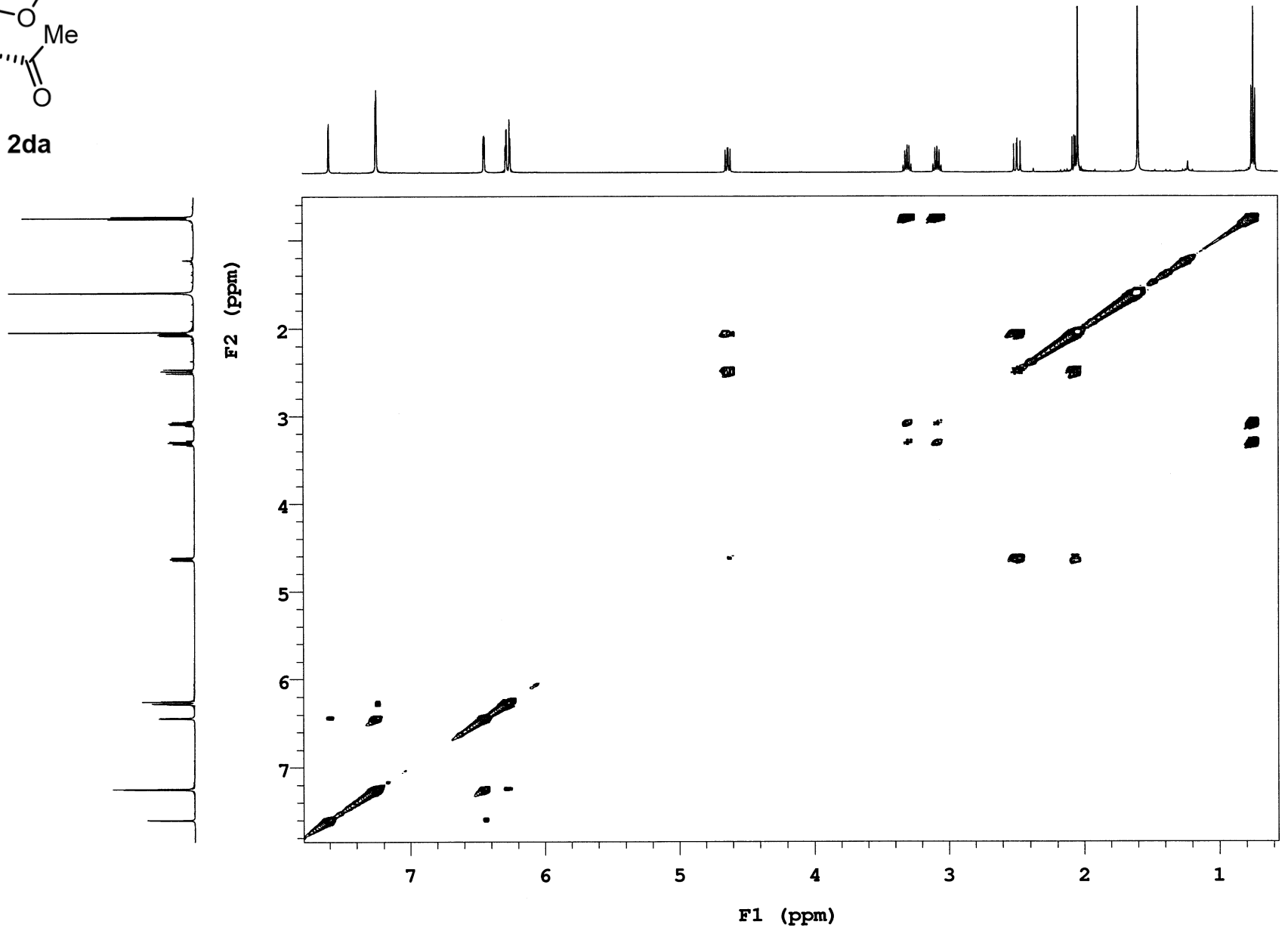
Plot date 2024-03-07



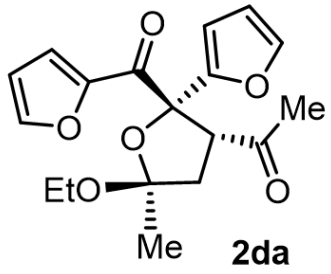
2da

IRR-02-213-P

Sample Name	IRR-02-213-P	Pulse sequence	gCOSY	Temperature	25	Study owner	vnmr2
Date collected	2024-03-07	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



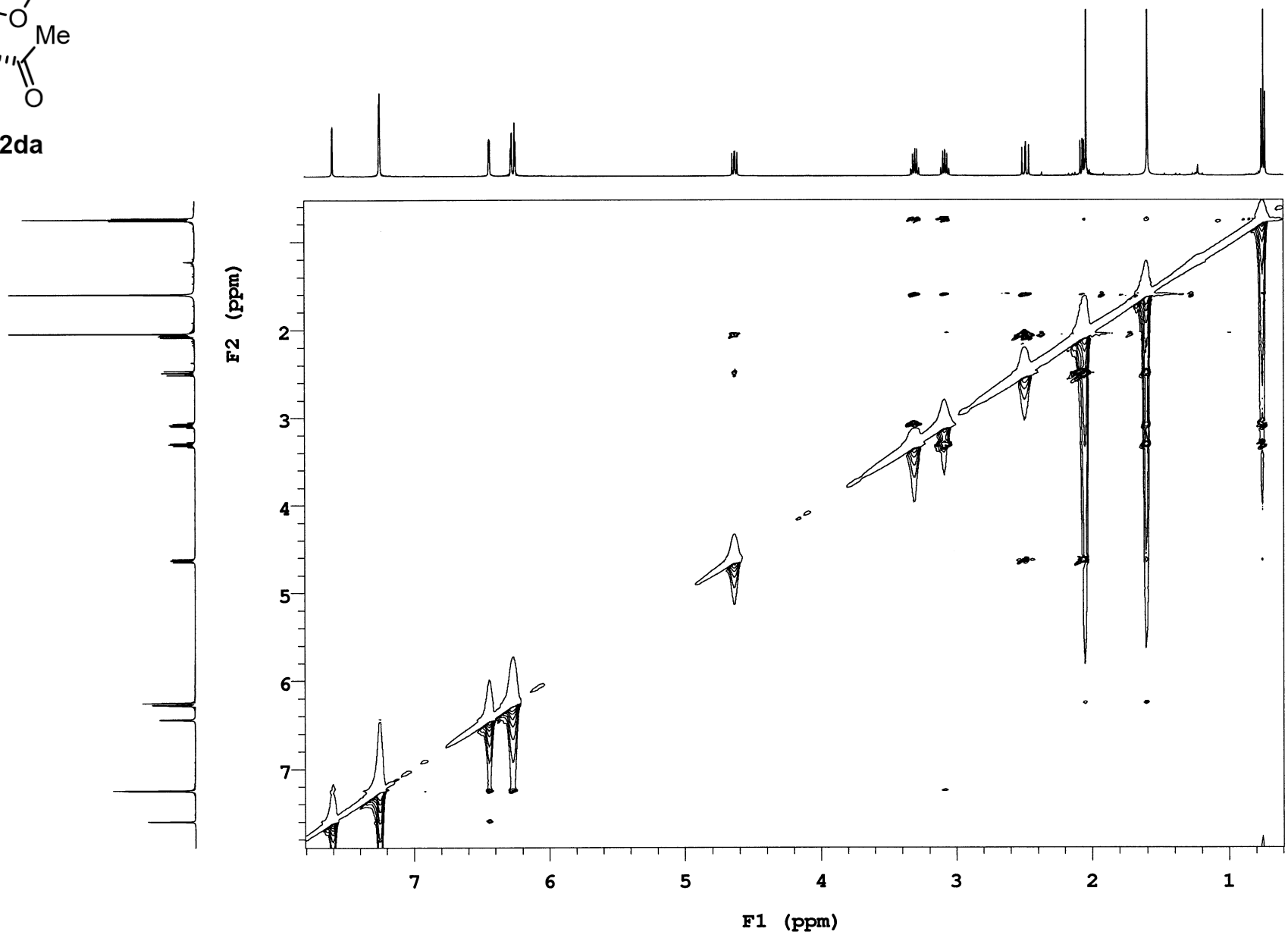
COSY of compound 2da



2da

IRR-02-213-P

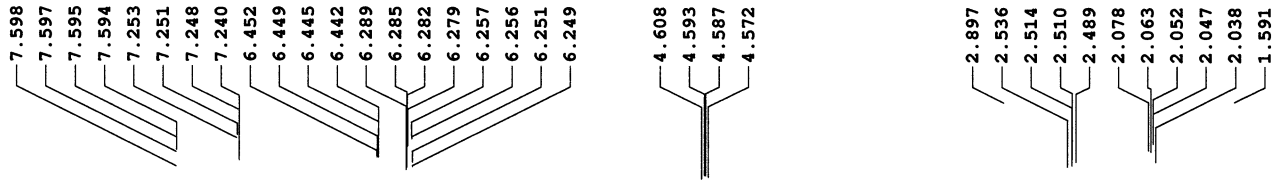
Sample Name	**IRR-02-213-P**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-07**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

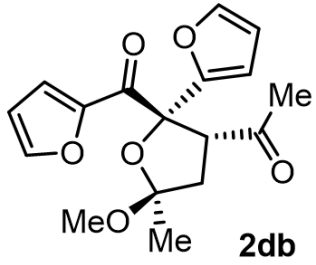


NOESY of compound 2da

IRR-02-229

Sample Name	**IRR-02-229**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-27**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**





2db

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-229/PROTON_04

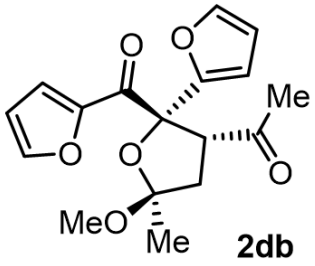
1H NMR (500 MHz, CDCl3) of compound 2db

Plot date 2024-02-29

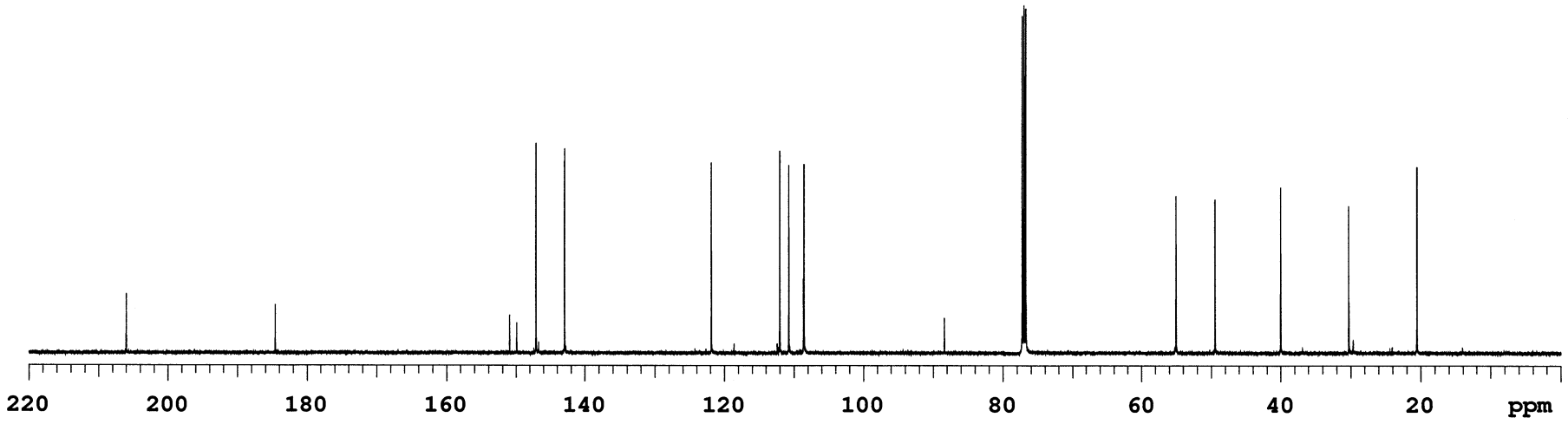
IRR-02-229

Sample Name	**IRR-02-229**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-27**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

206.035
184.583
150.959
149.934
147.153
142.951
121.921
112.034
110.738
108.642
108.547
88.419
77.254
77.000
76.746
55.034
49.447
40.022
30.304
20.532



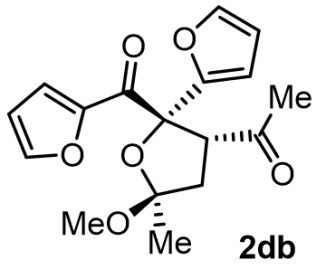
2db



13C NMR (125 MHz, CDCl3) of compound 2db

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-229/CARBON_02

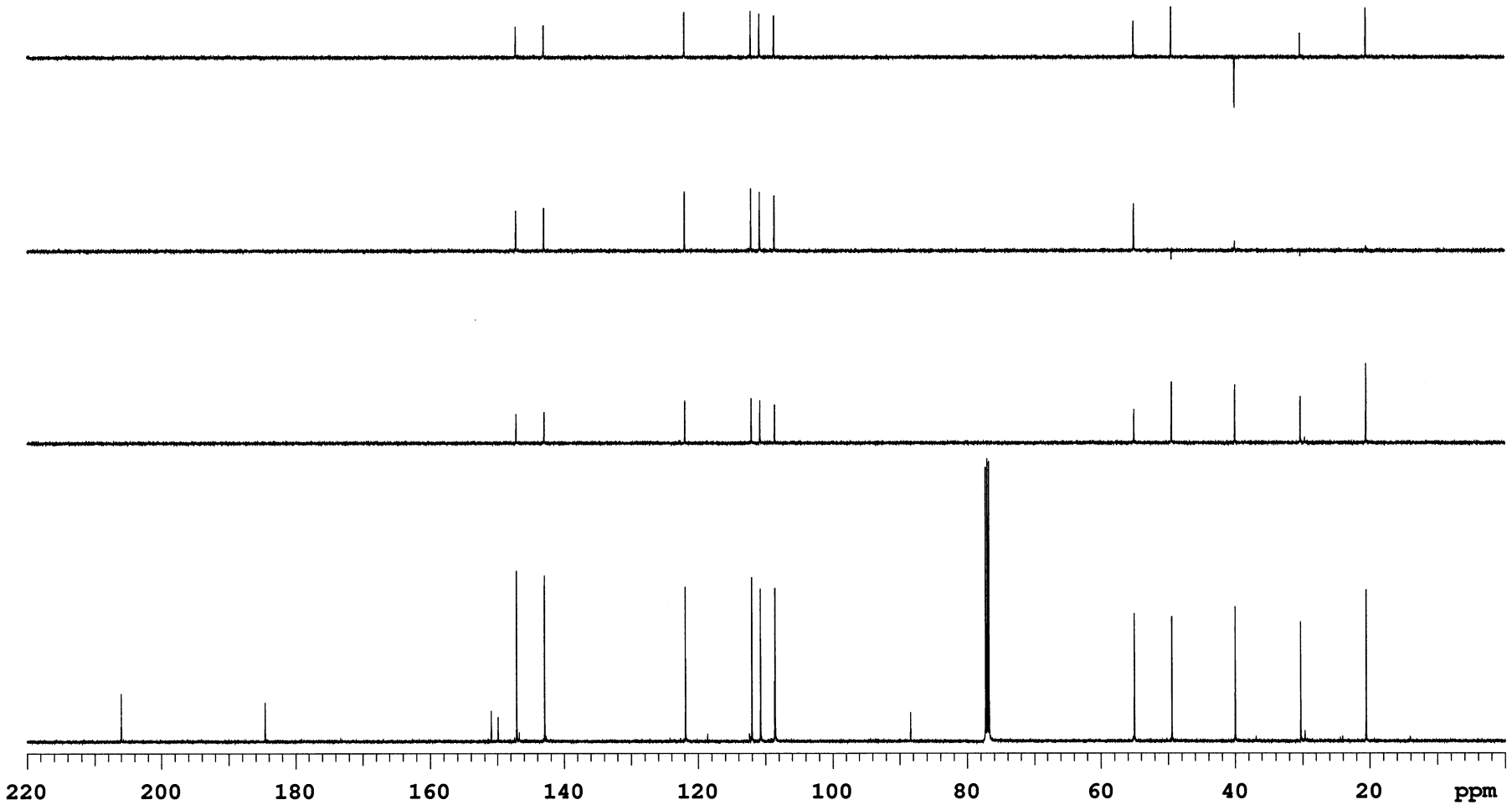
Plot date 2024-03-01



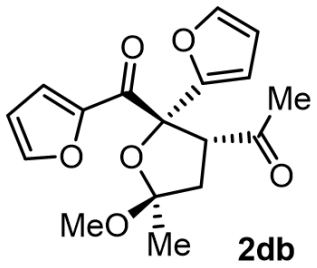
2db

IRR-02-229

Sample Name	**IRR-02-229**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



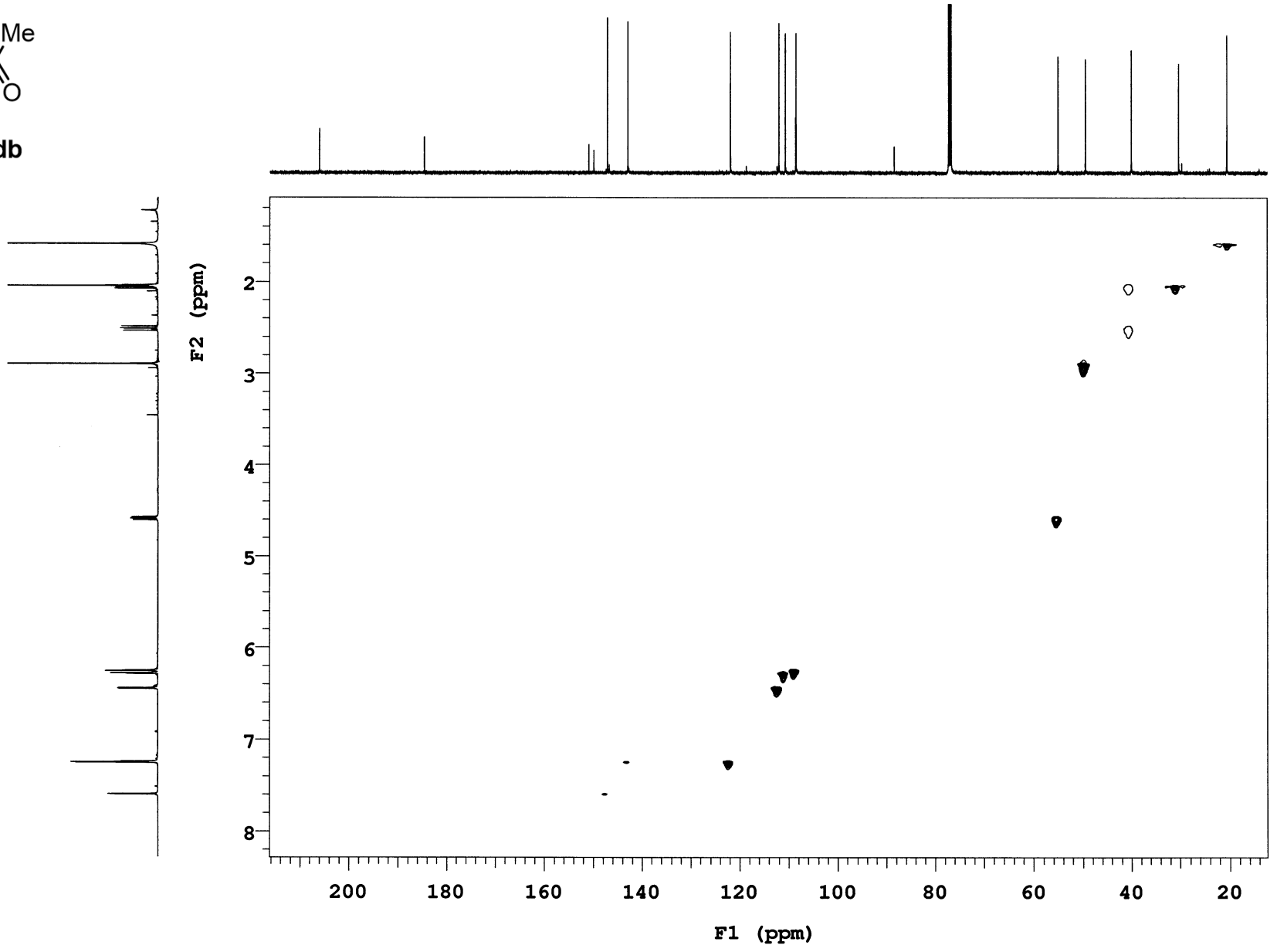
DEPT of compound 2db



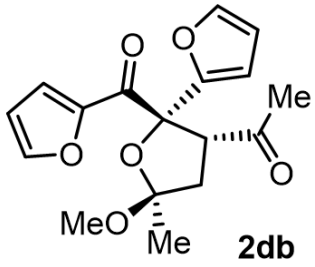
2db

IRR-02-229

Sample Name	**IRR-02-229**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



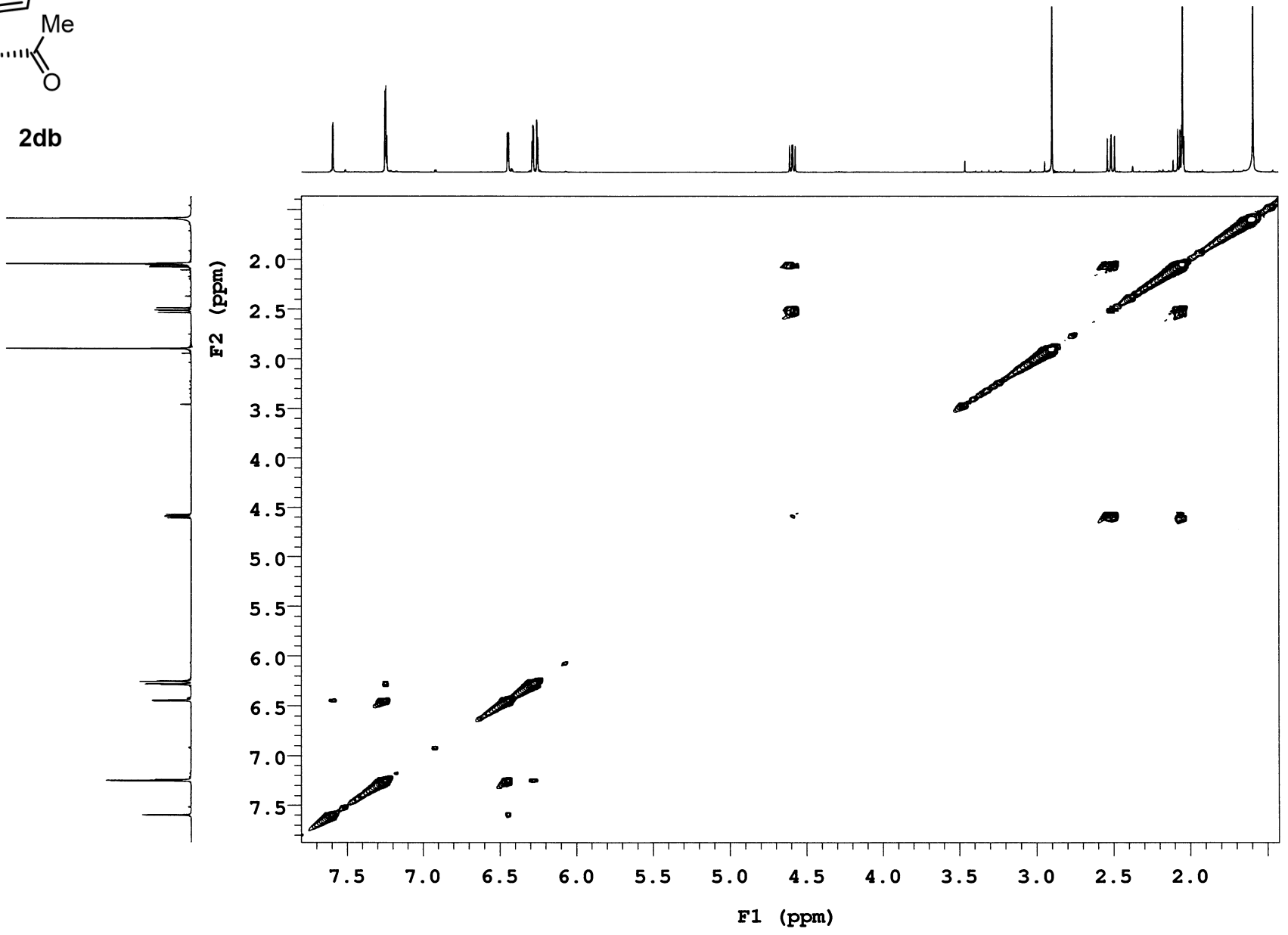
HSQC of compound 2db



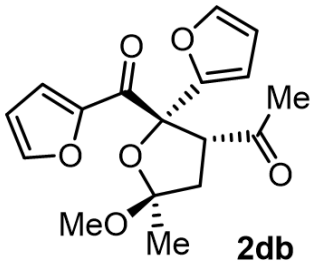
2db

IRR-02-229

Sample Name	**IRR-02-229**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



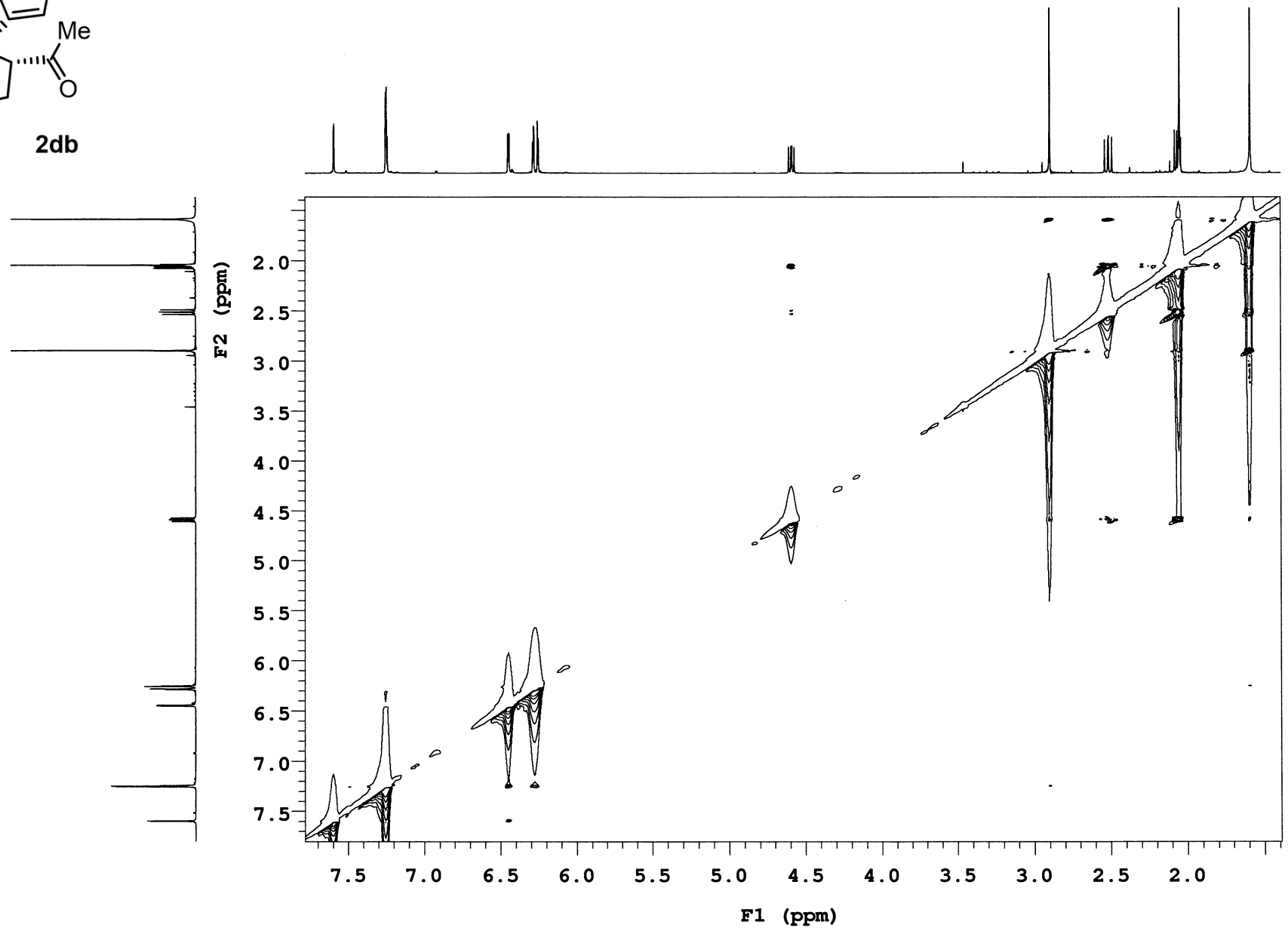
COSY of compound 2db



2db

IRR-02-229

Sample Name	**IRR-02-229**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-28**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



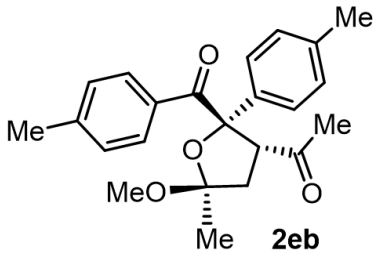
NOESY of compound 2db

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-02-229/NOESY_01

Plot date 2024-02-29

IRR-03-008

Sample Name **IRR-03-008**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-02-26**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

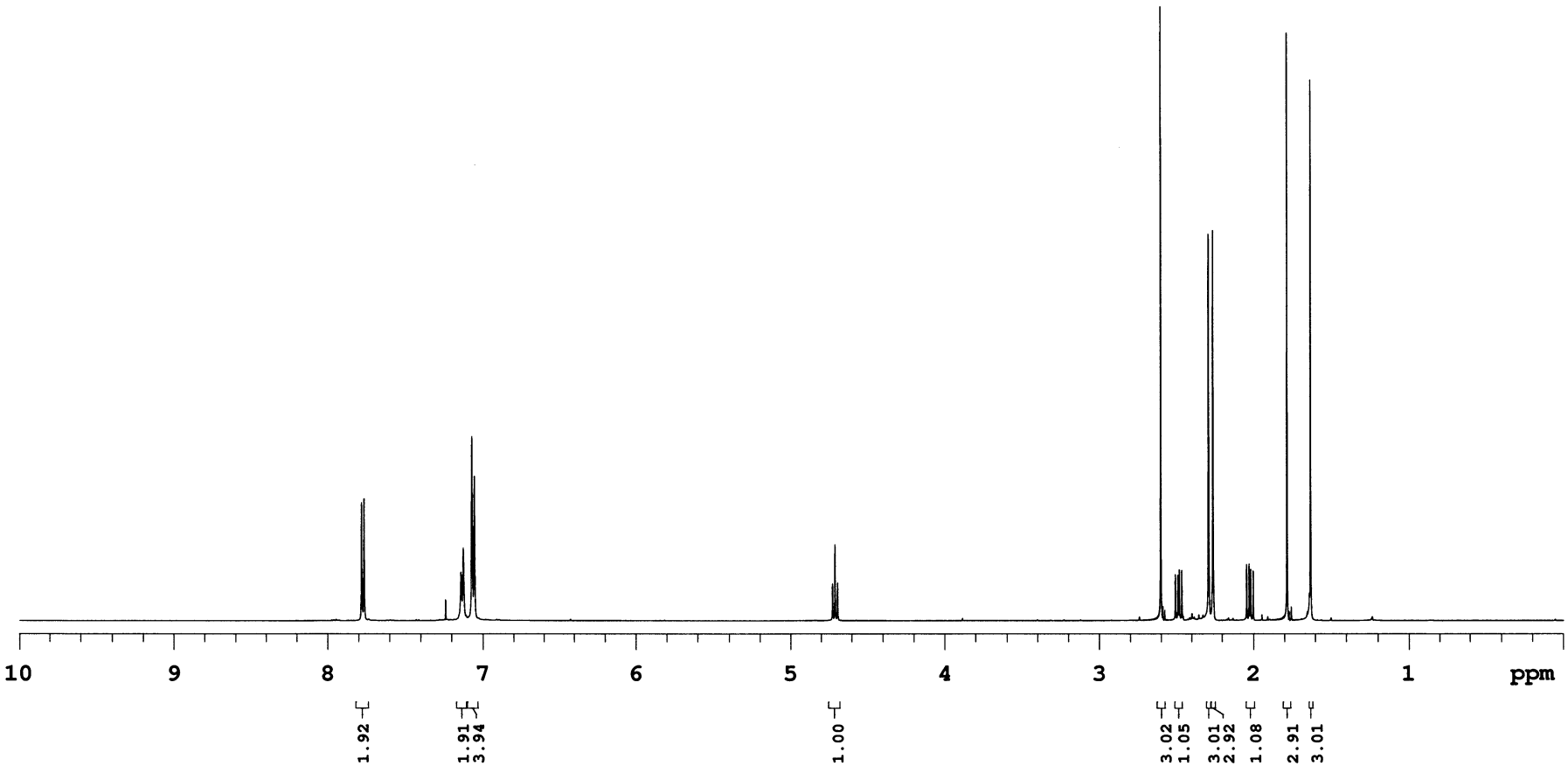


2eb

7.781 7.764 7.240 7.141 7.125 7.070 7.054

4.727 4.710 4.694

2.601 2.507 2.490 2.481 2.464 2.290 2.261 2.044 2.028 2.018 2.002 1.783 1.638 1.629



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-008/PROTON_03

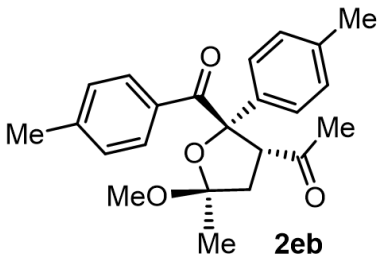
1H NMR (500 MHz, CDCl3) of compound 2eb

Plot date 2024-02-27

IRR-03-008

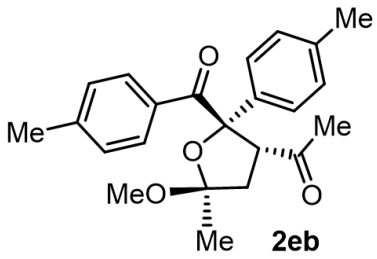
Sample Name	**IRR-03-008**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-26**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

207.795 198.516 143.077 138.017 134.892 131.859 131.129 129.262 128.516 125.384 108.839 93.571 77.254 77.000 76.746 57.099 49.557 40.830 31.190 21.561 21.063 20.504



2eb

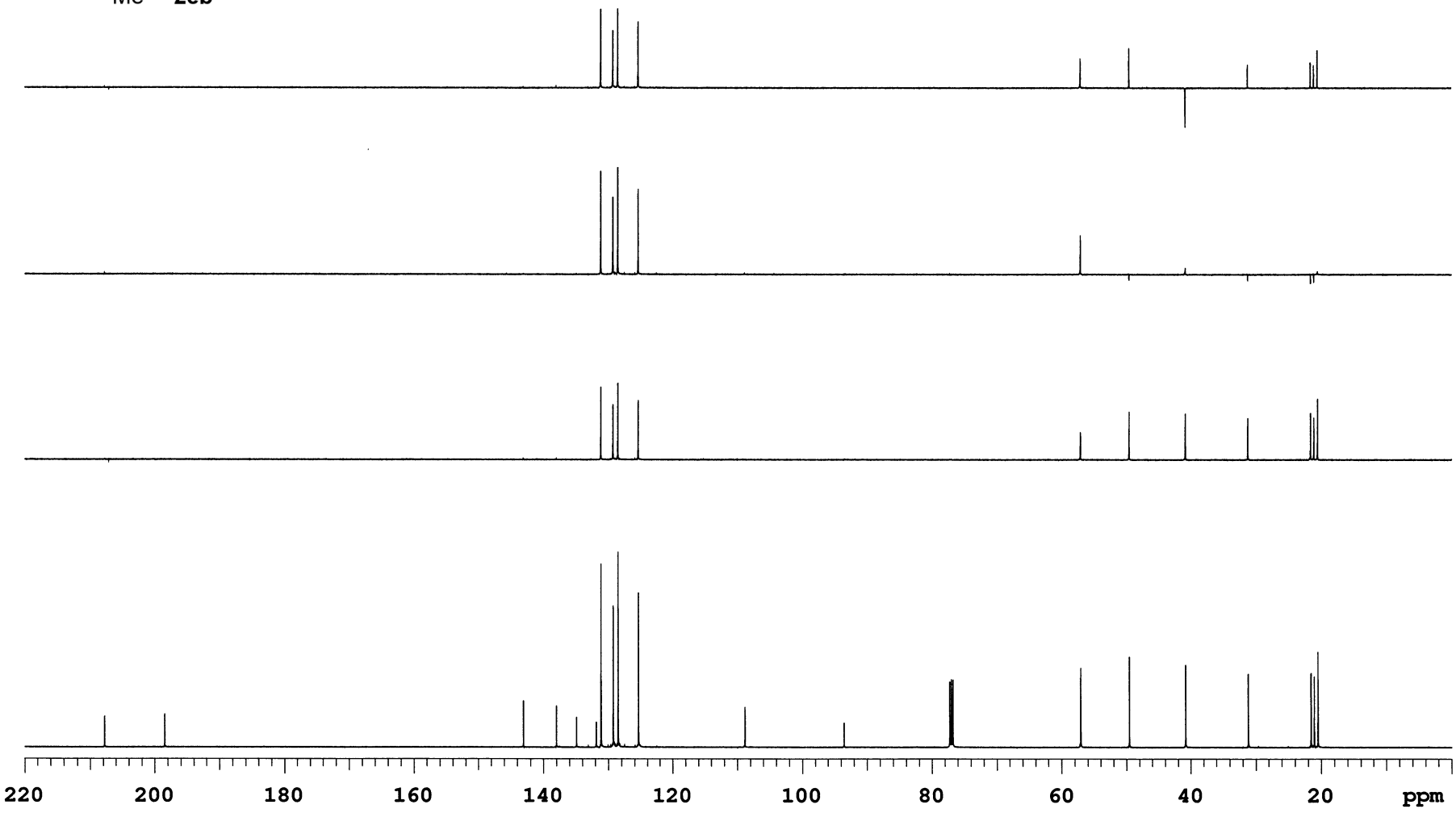
13C NMR (125 MHz, CDCl3) of compound 2eb



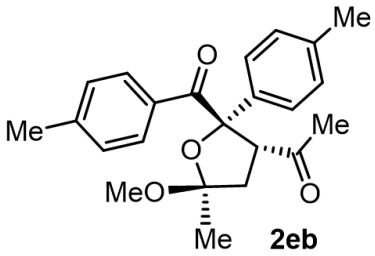
2eb

IRR-03-008

Sample Name **IRR-03-008**	Pulse sequence **DEPT**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-02-27**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

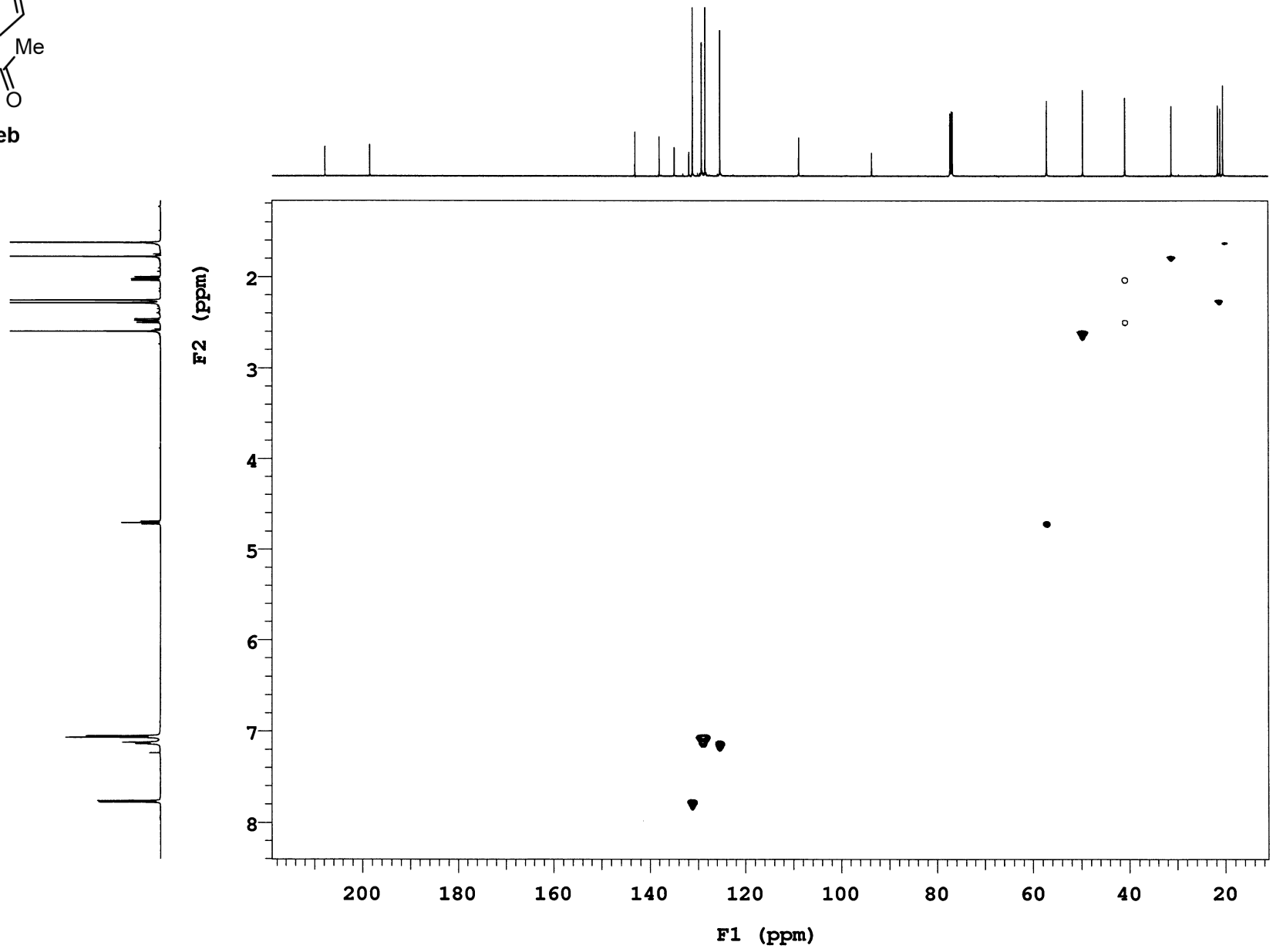


DEPT of compound 2eb

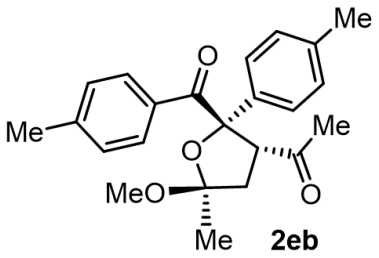


IRR-03-008

Sample Name **IRR-03-008**	Pulse sequence **gHSQC**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-02-27**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

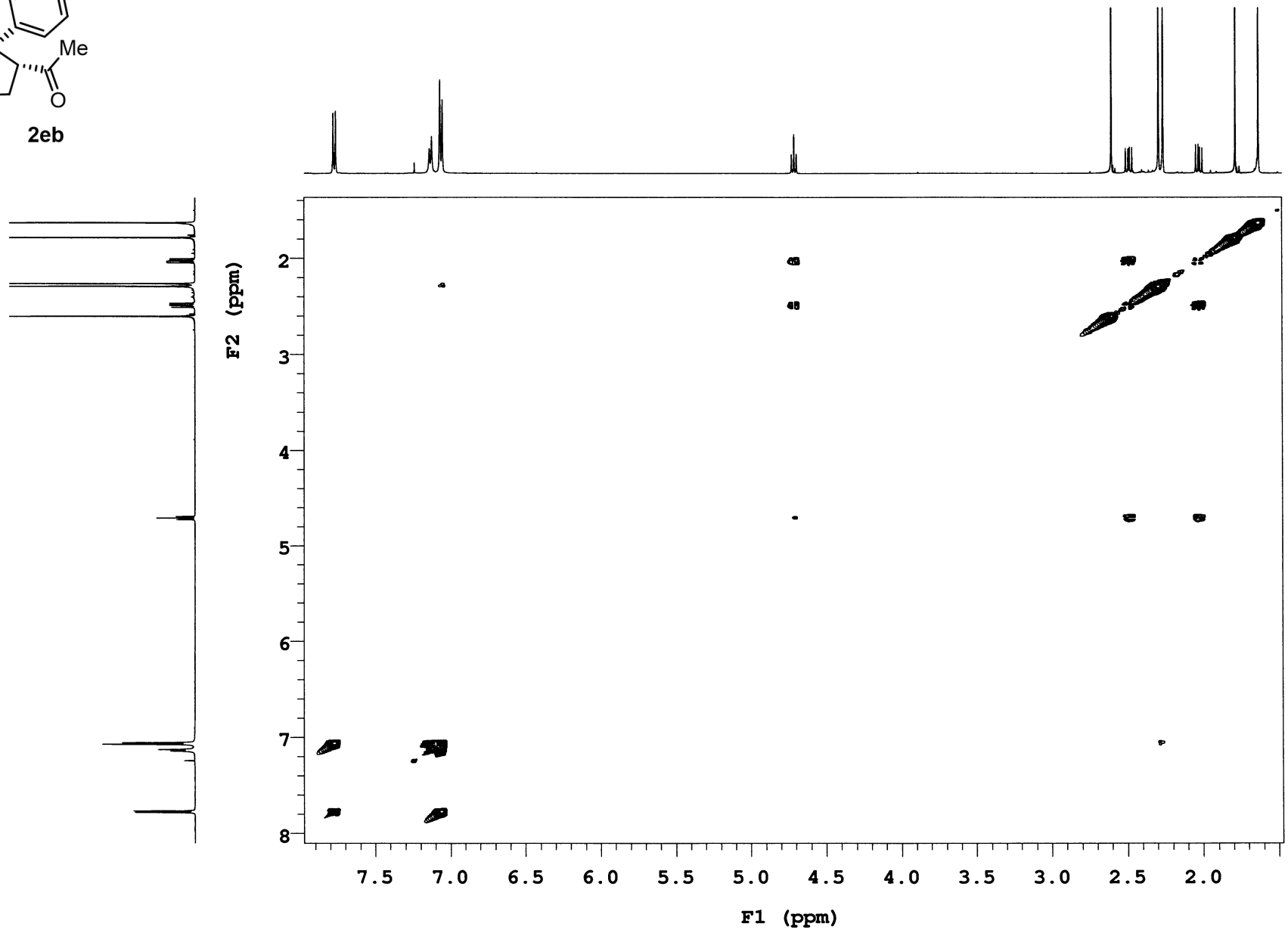


HSQC of compound 2eb



IRR-03-008

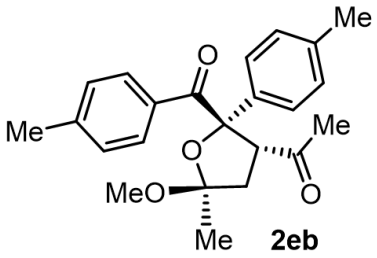
Sample Name **IRR-03-008**	Pulse sequence **gCOSY**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-02-27**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



COSY of compound 2eb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-008/gCOSY_01

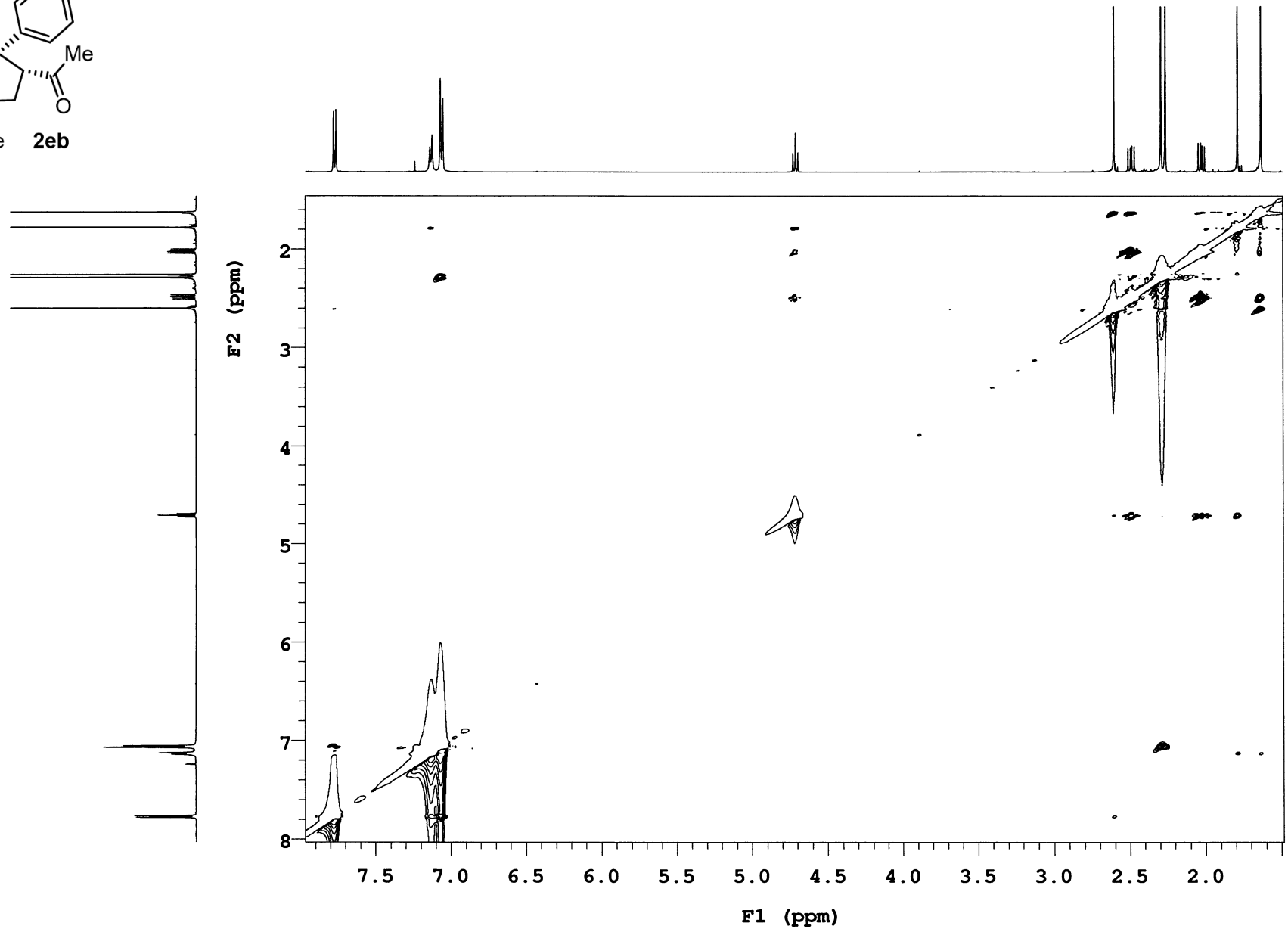
Plot date 2024-02-27



2eb

IRR-03-008

Sample Name	**IRR-03-008**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-27**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



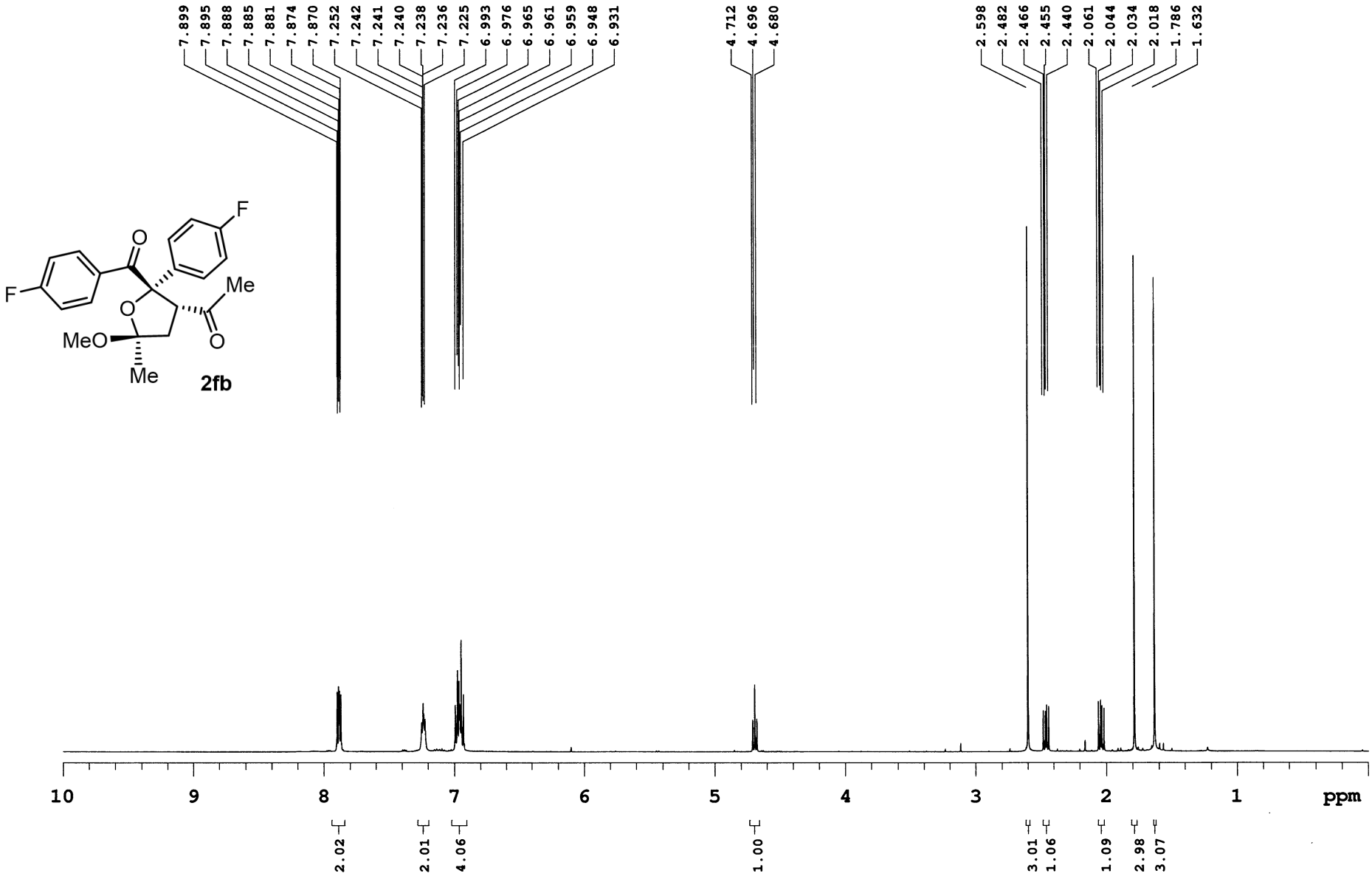
NOESY of compound 2eb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-008/NOESY_01

Plot date 2024-02-27

IRR-03-009

Sample Name **IRR-03-009**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-02-23**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



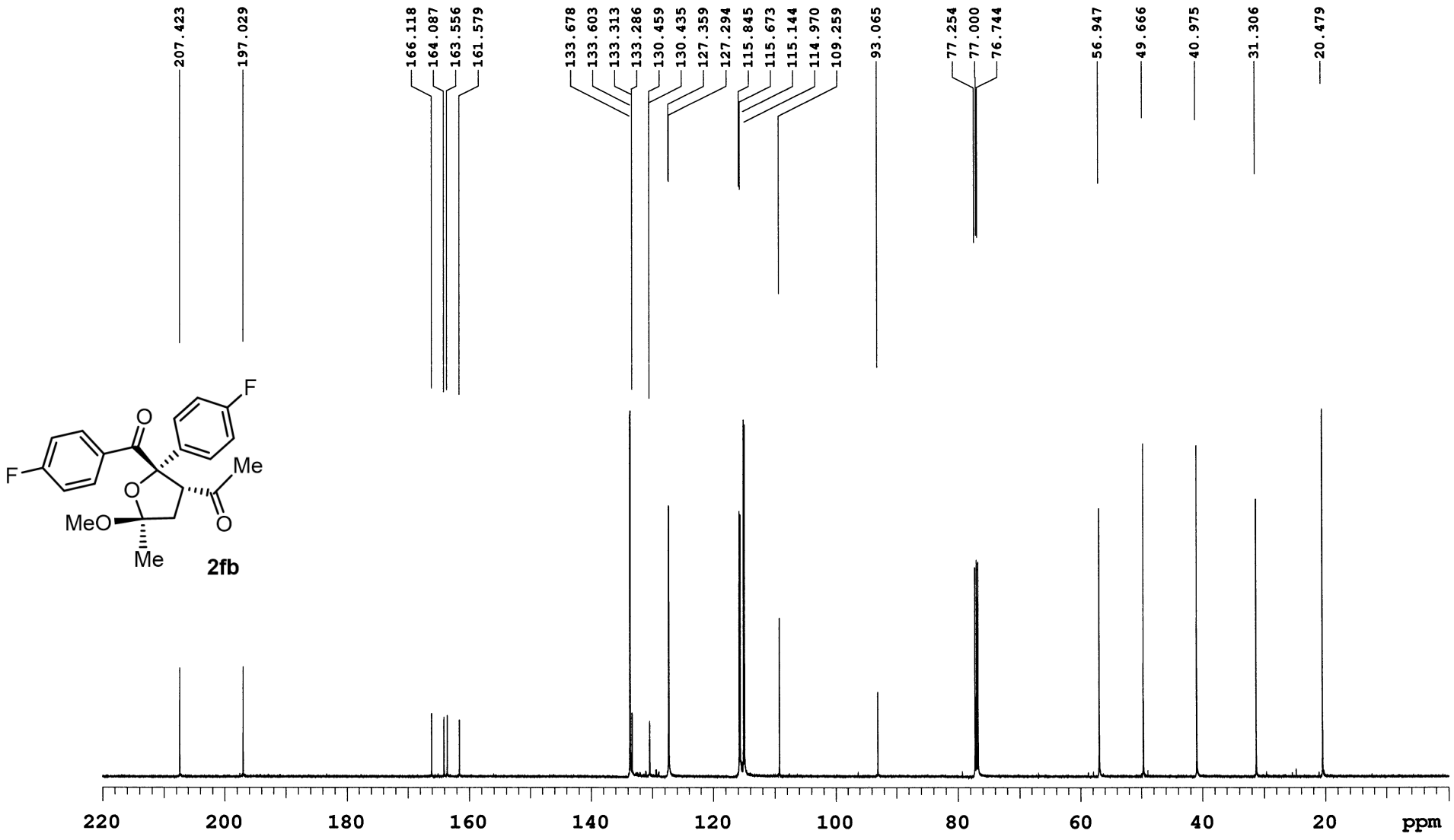
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-009/PROTON_04

1H NMR (500 MHz, CDCl3) of compound 2fb

Plot date 2024-02-26

IRR-03-009

Sample Name	**IRR-03-009**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-23**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



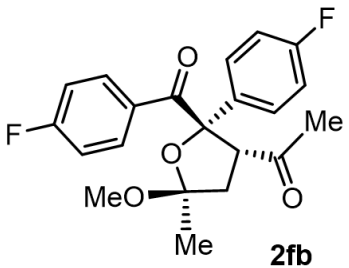
13C NMR (125 MHz, CDCl3) of compound 2fb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-009/CARBON_03

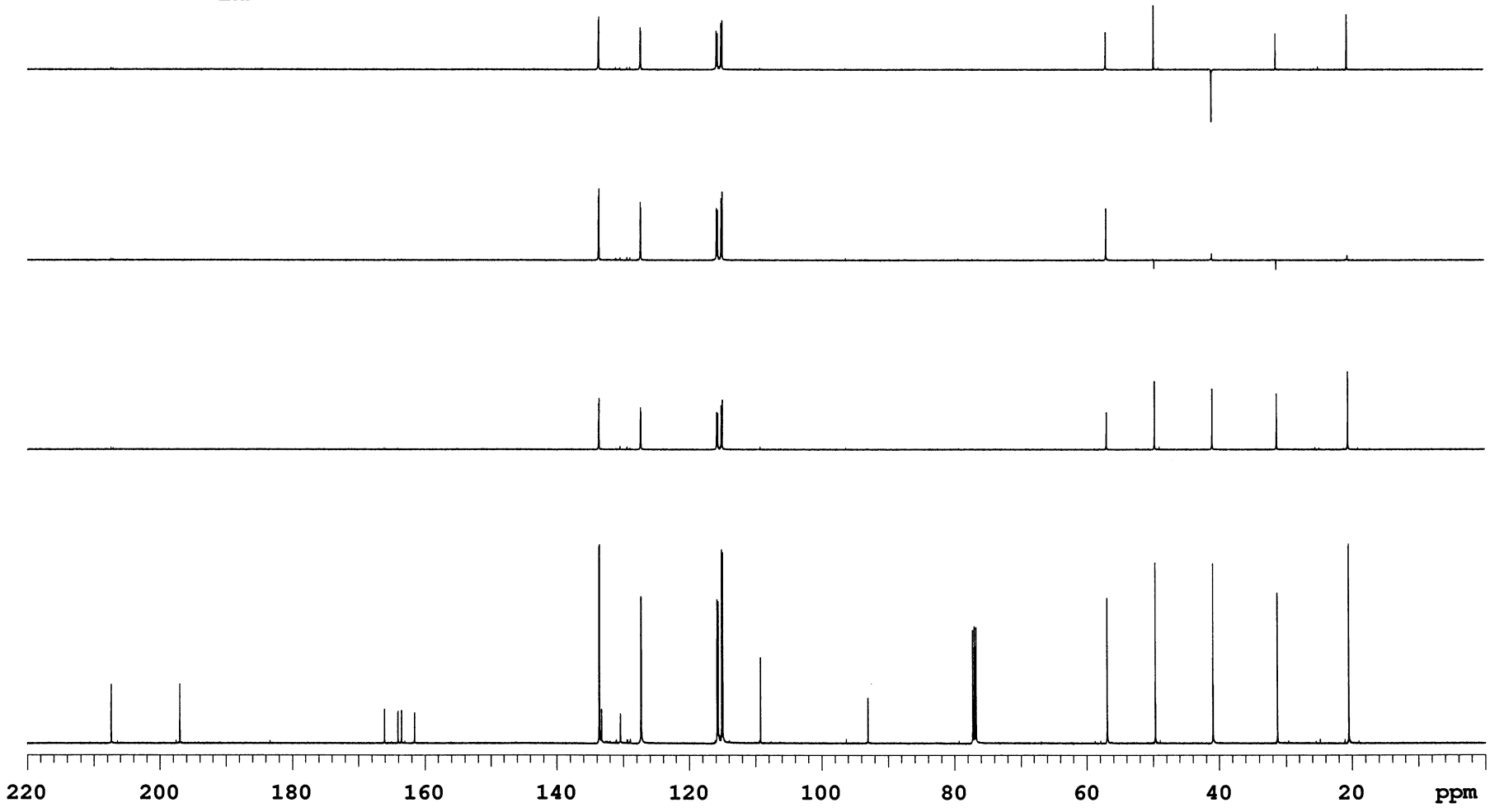
Plot date 2024-02-26

IRR-03-009

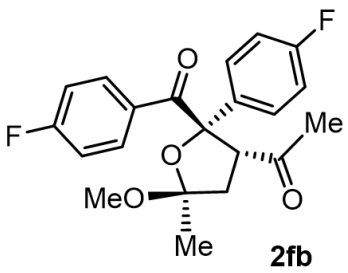
Sample Name	**IRR-03-009**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2fb



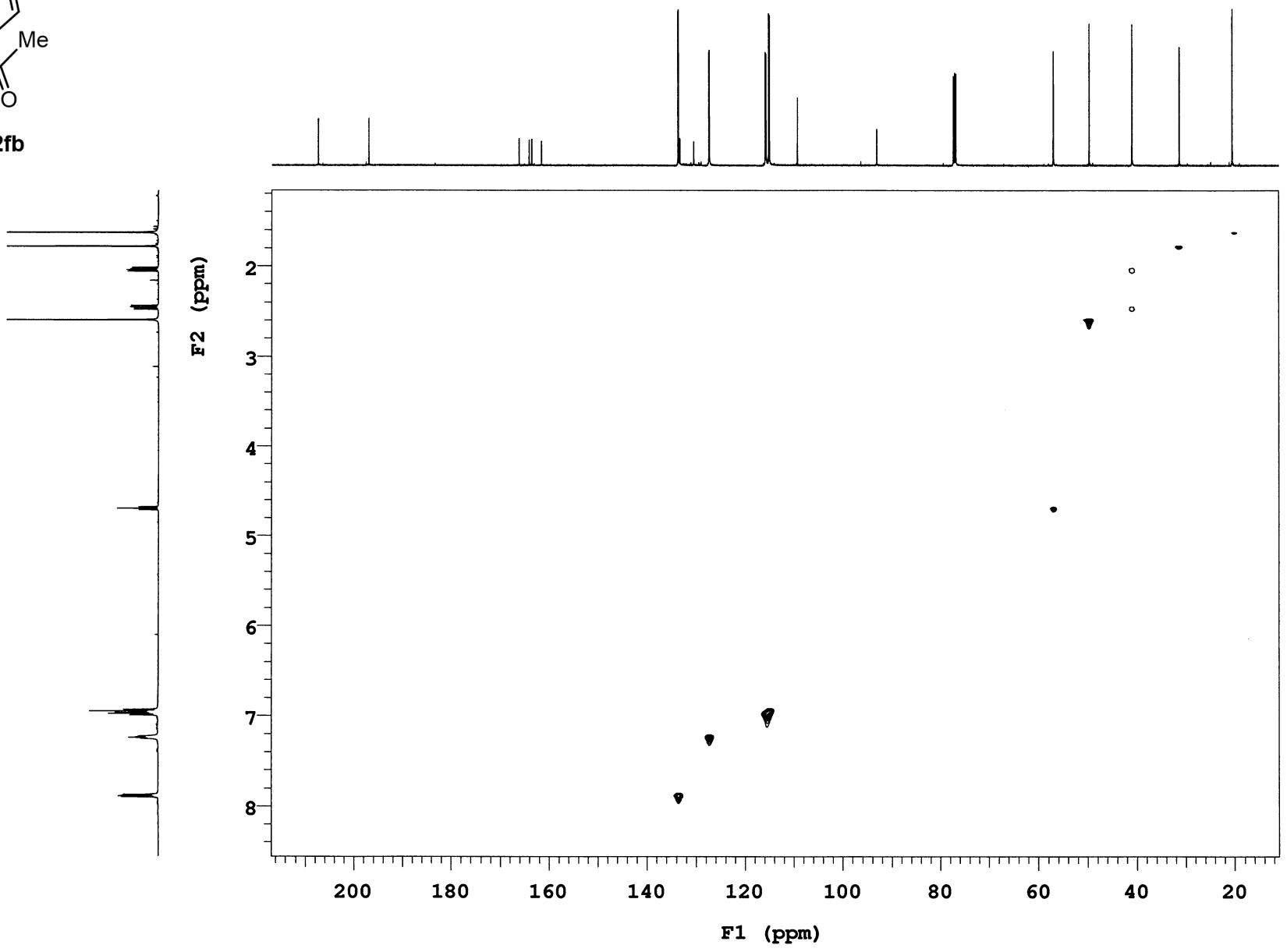
DEPT of compound 2fb



2fb

IRR-03-009

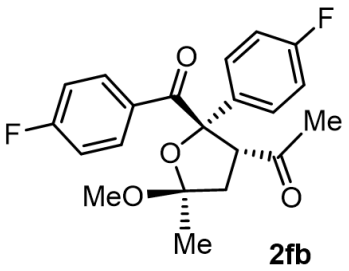
Sample Name	**IRR-03-009**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2fb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-009/gHSQC_01

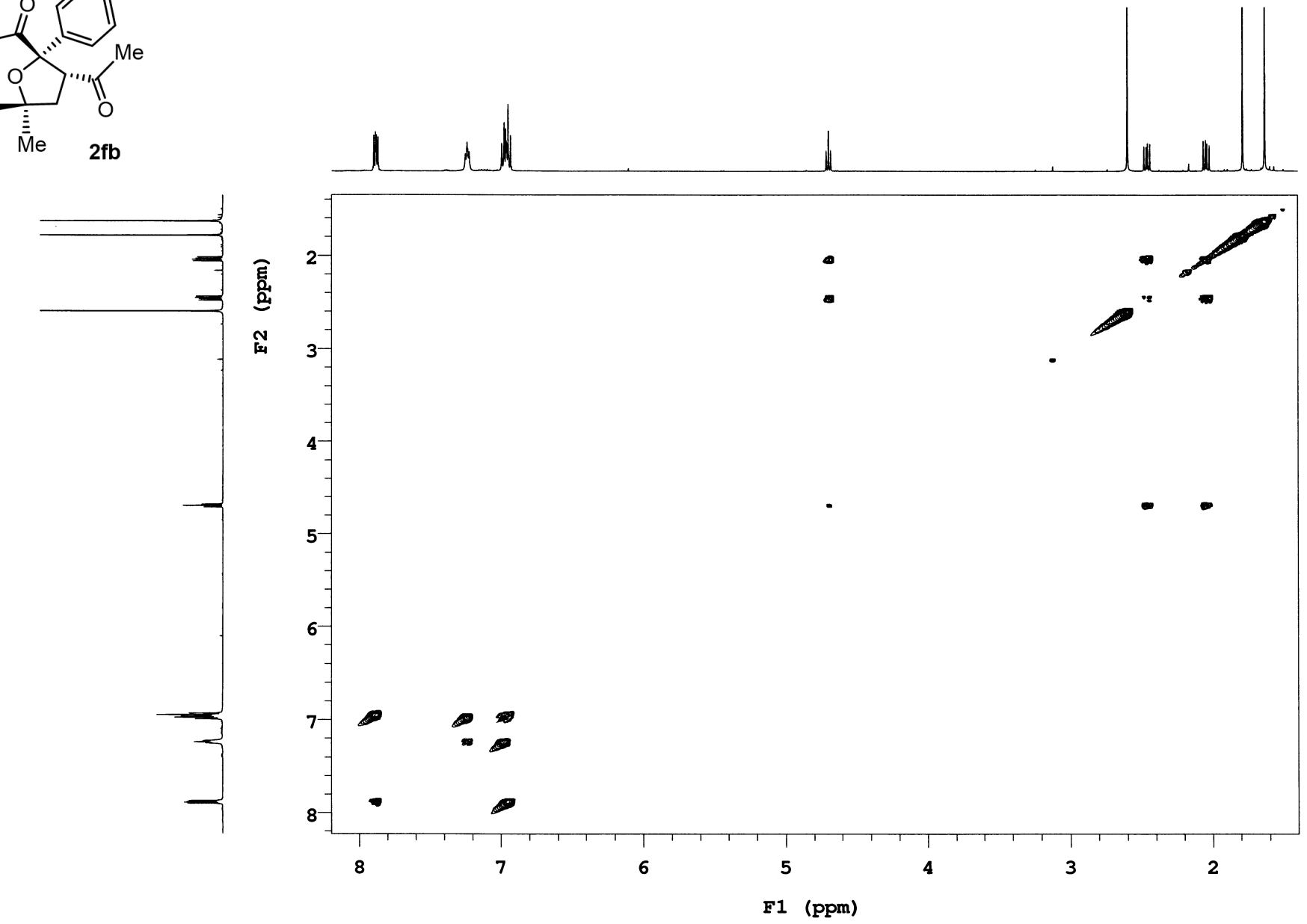
Plot date 2024-02-26



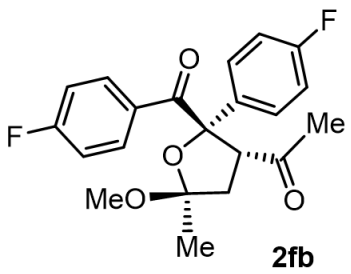
2fb

IRR-03-009

Sample Name	**IRR-03-009**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



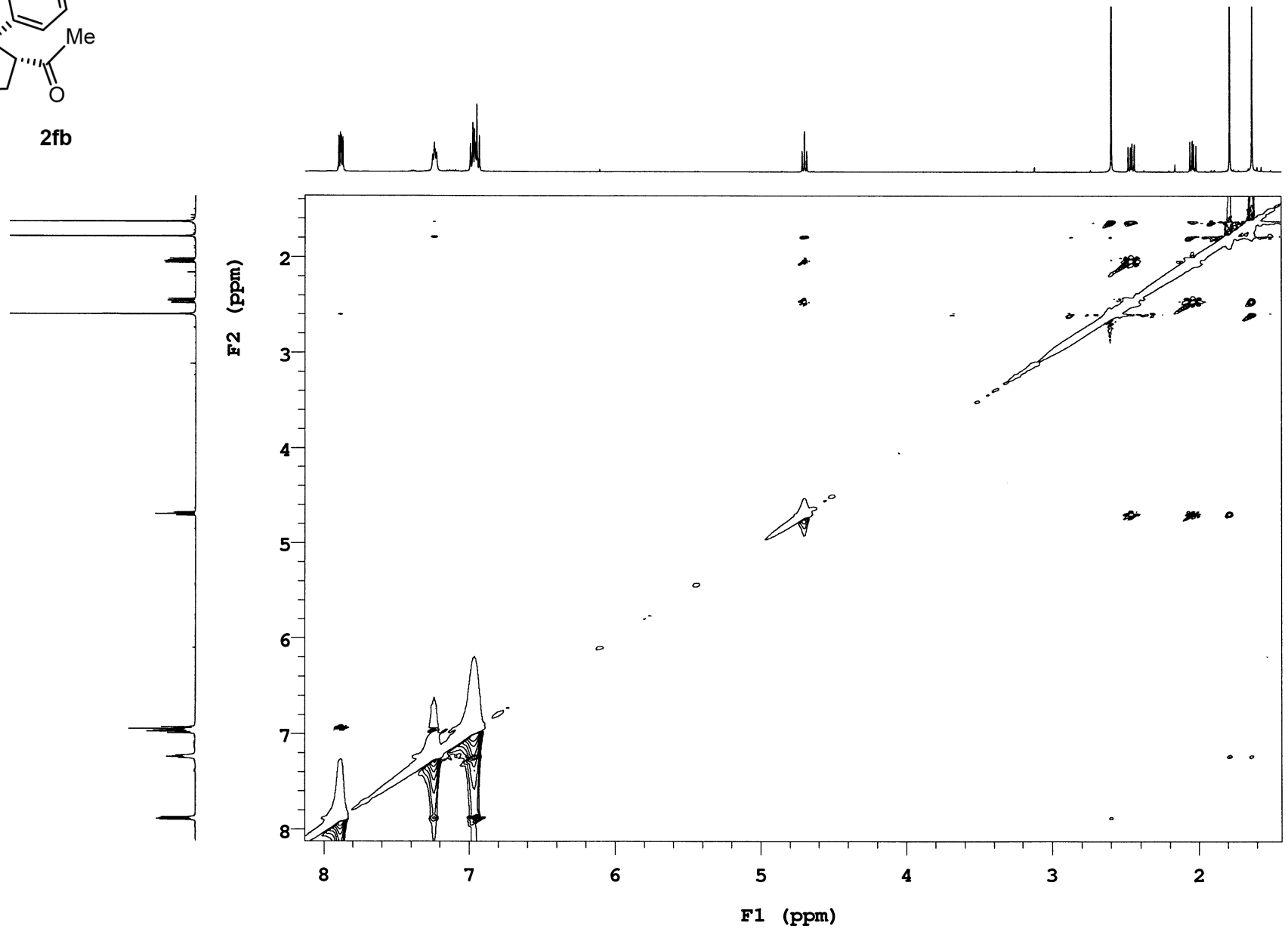
COSY of compound 2fb



2fb

IRR-03-009

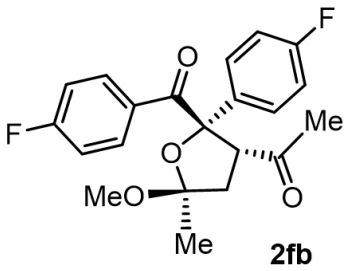
Sample Name	**IRR-03-009**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2fb

IRR-03-009

Sample Name	**IRR-03-009**	Pulse sequence	**FLUORINE**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-02-23**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2fb

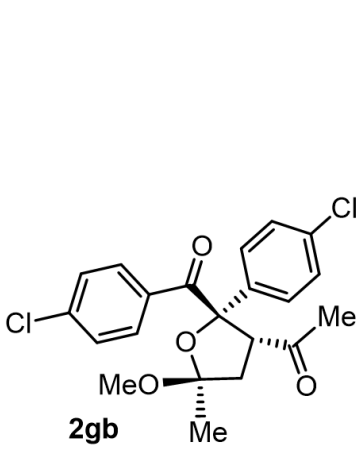
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-009/FLUORINE_03

19F NMR (471 MHz, CDCl3) of compound 2fb

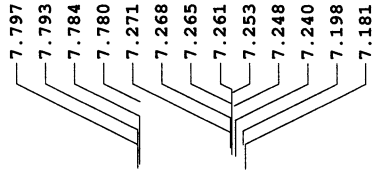
Plot date 2024-02-26

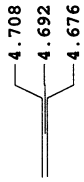
IRR-03-075

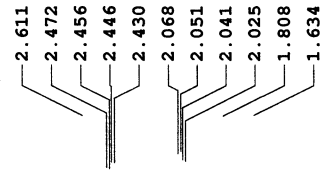
Sample Name	**IRR-03-075**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-03**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

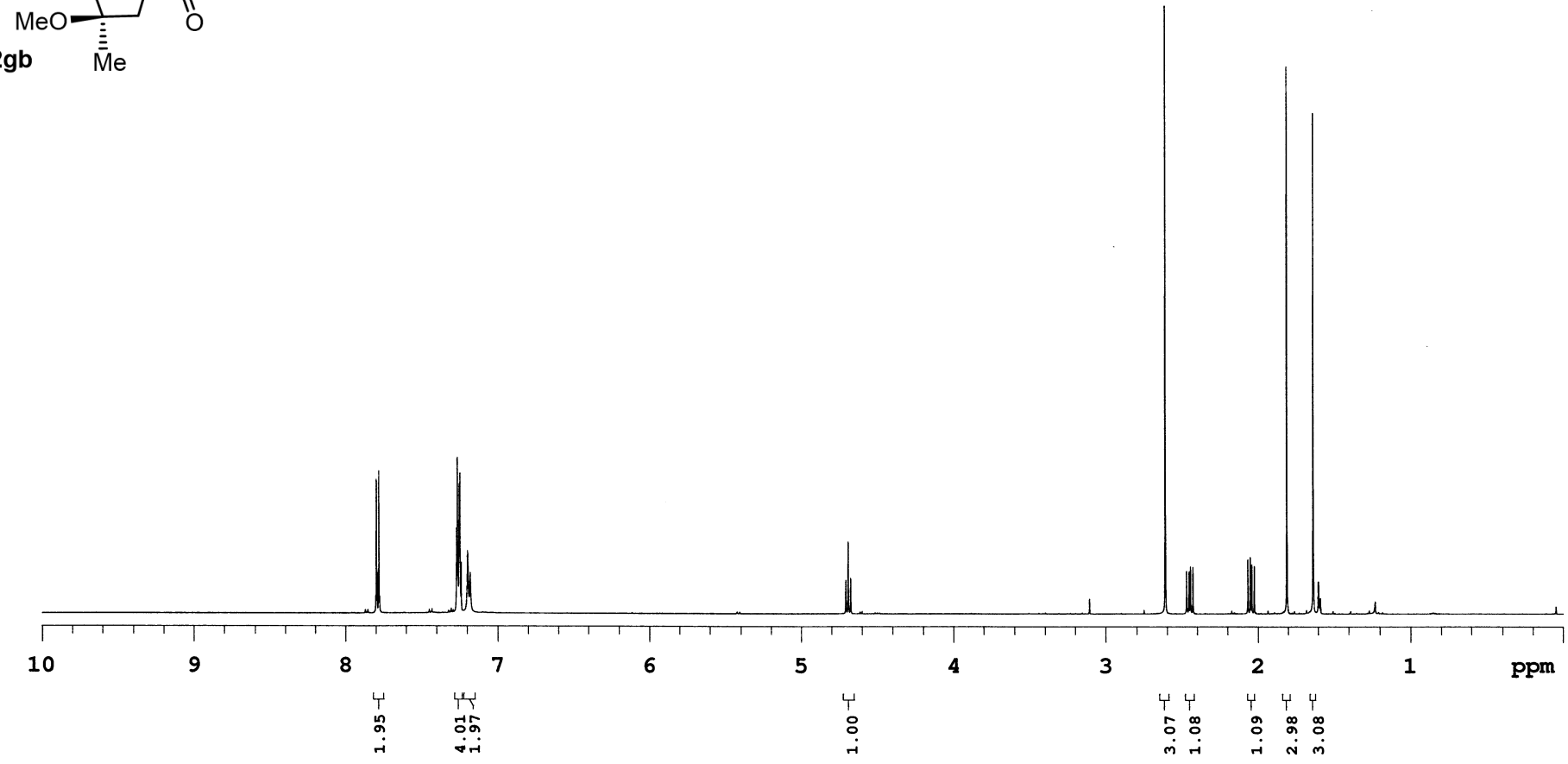


2gb









Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-075/PROTON_03

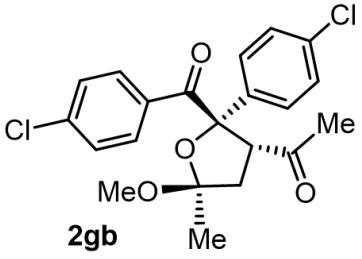
1H NMR (500 MHz, CDCl3) of compound 2gb

Plot date 2024-06-04

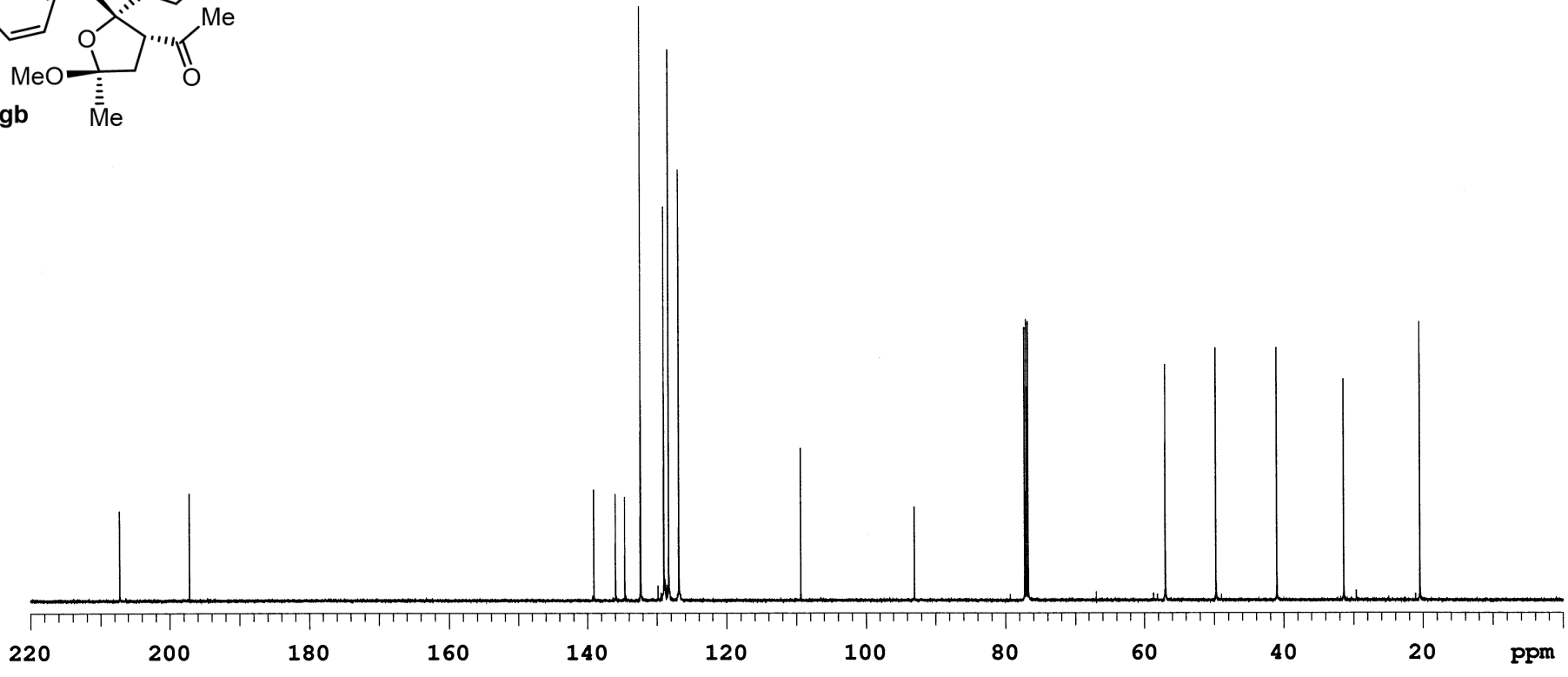
IRR-03-075

Sample Name	IRR-03-075	Pulse sequence	CARBON	Temperature	25	Study owner	vnmr2
Date collected	2024-06-05	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2

207.240
197.205
139.088
135.953
134.609
132.361
132.315
128.993
128.312
126.855
109.375
93.027
77.254
77.000
76.746
56.979
49.764
41.032
31.426
20.462



2gb



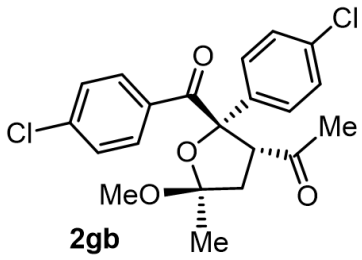
13C NMR (125 MHz, CDCl3) of compound 2gb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-075/CARBON_04

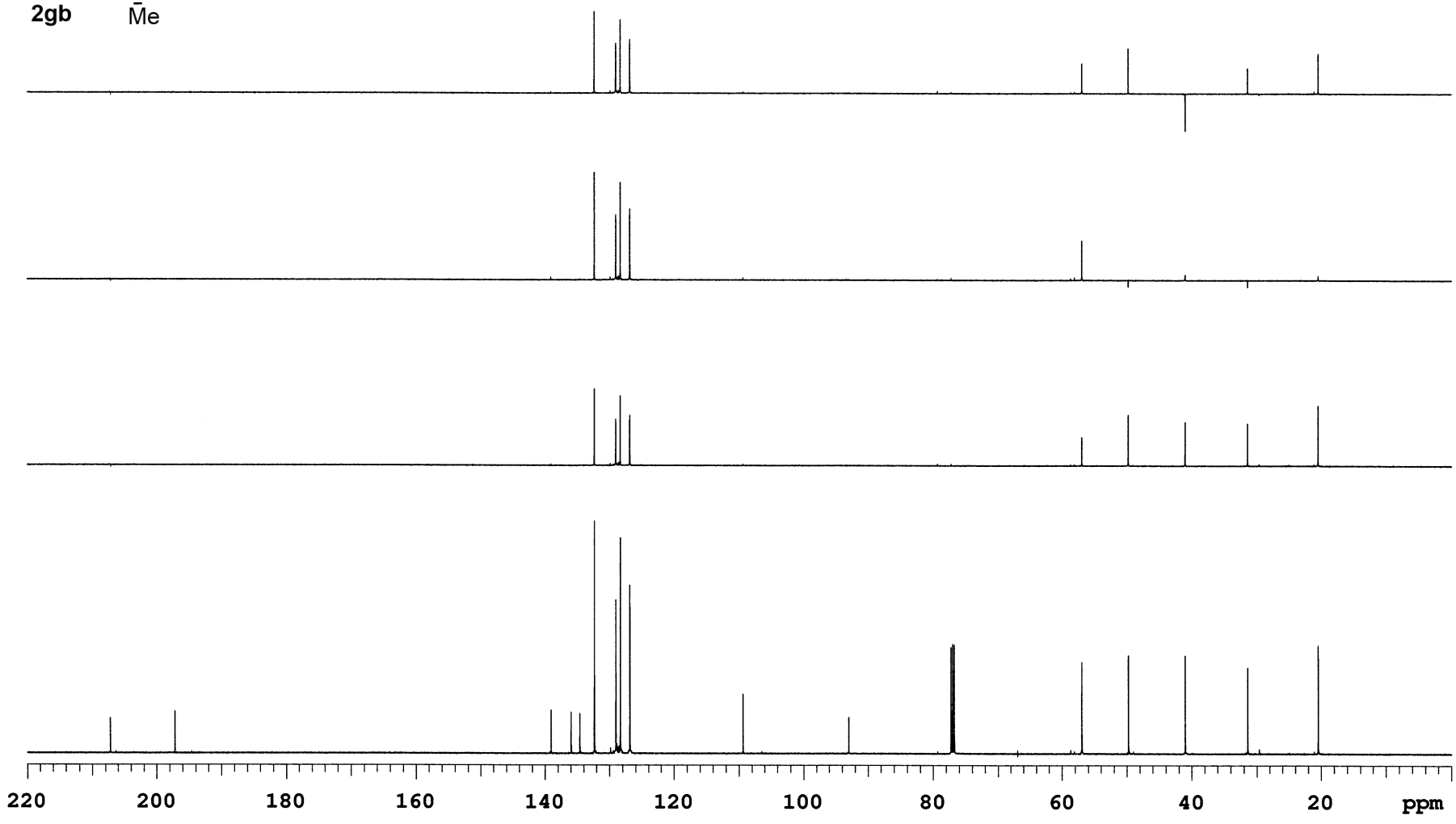
Plot date 2024-06-05

IRR-03-075

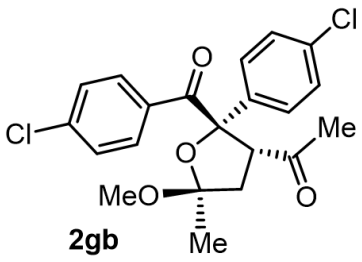
Sample Name	**IRR-03-075**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-03**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2gb



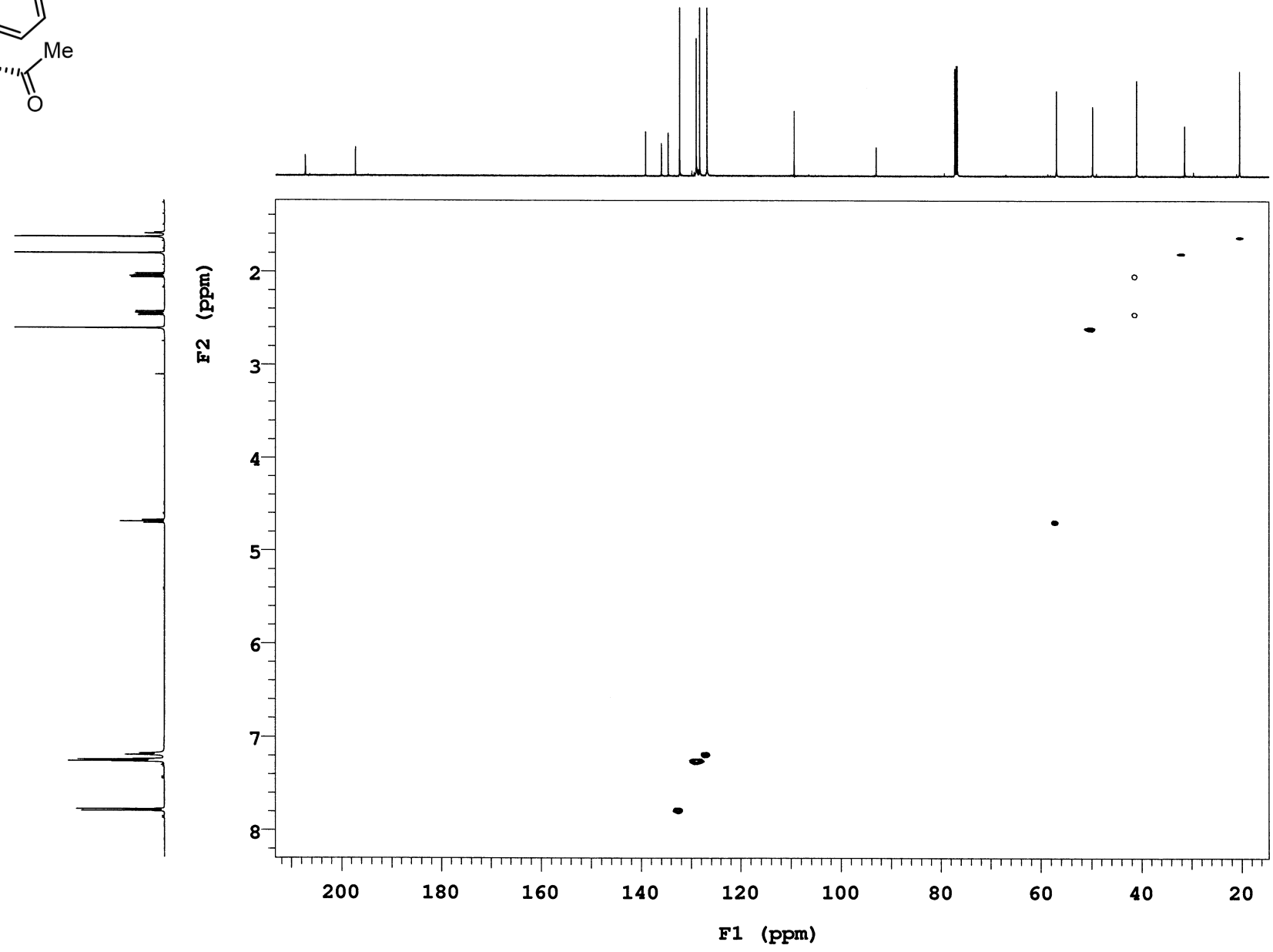
DEPT of compound 2gb



2gb

IRR-03-075

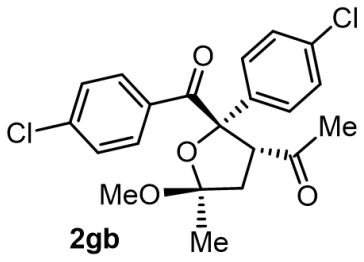
Sample Name	**IRR-03-075**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-04**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2gb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-075/gHSQC_01

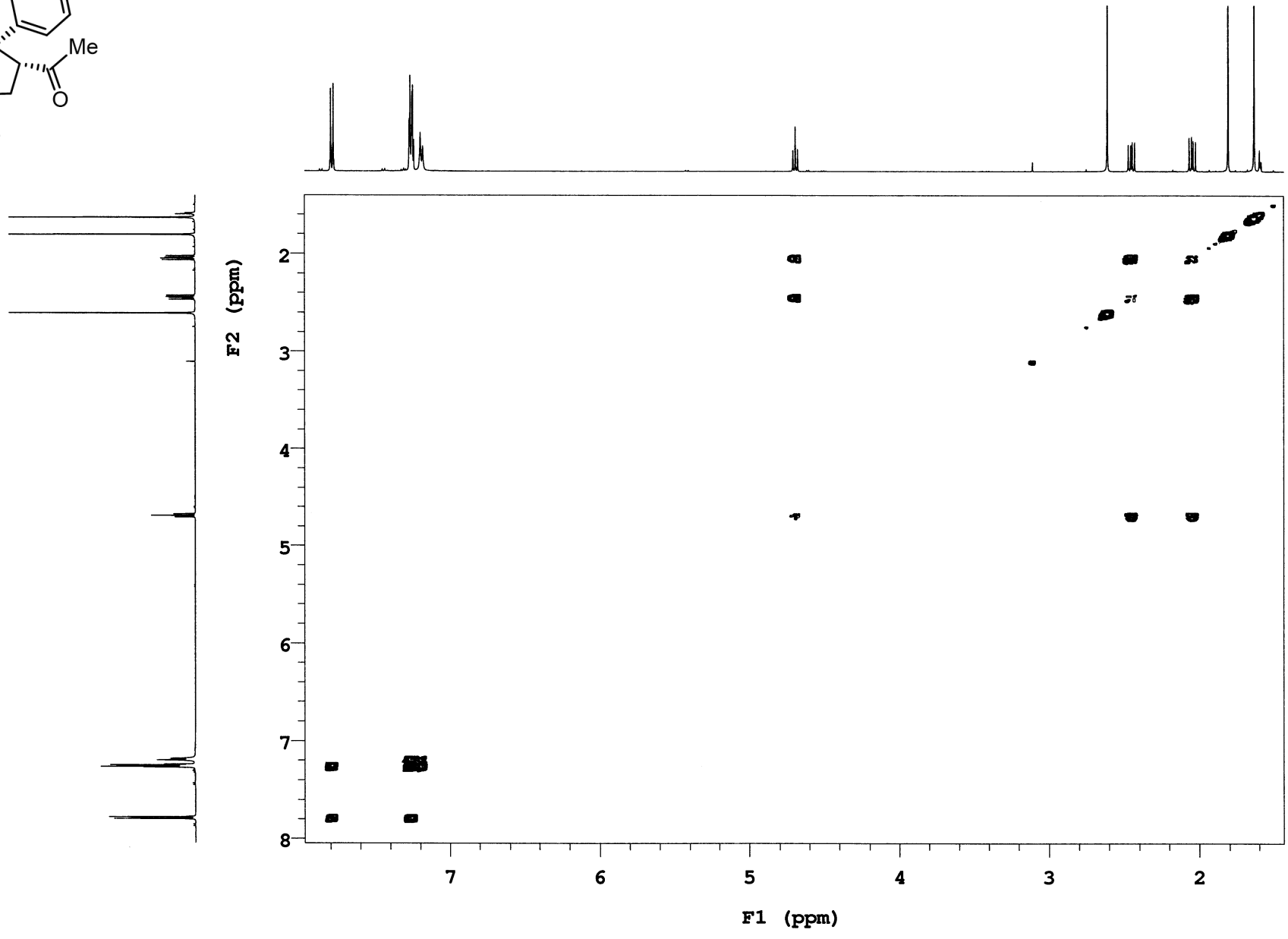
Plot date 2024-06-04



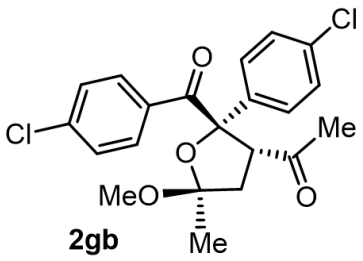
2gb

IRR-03-075

Sample Name	**IRR-03-075**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-04**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



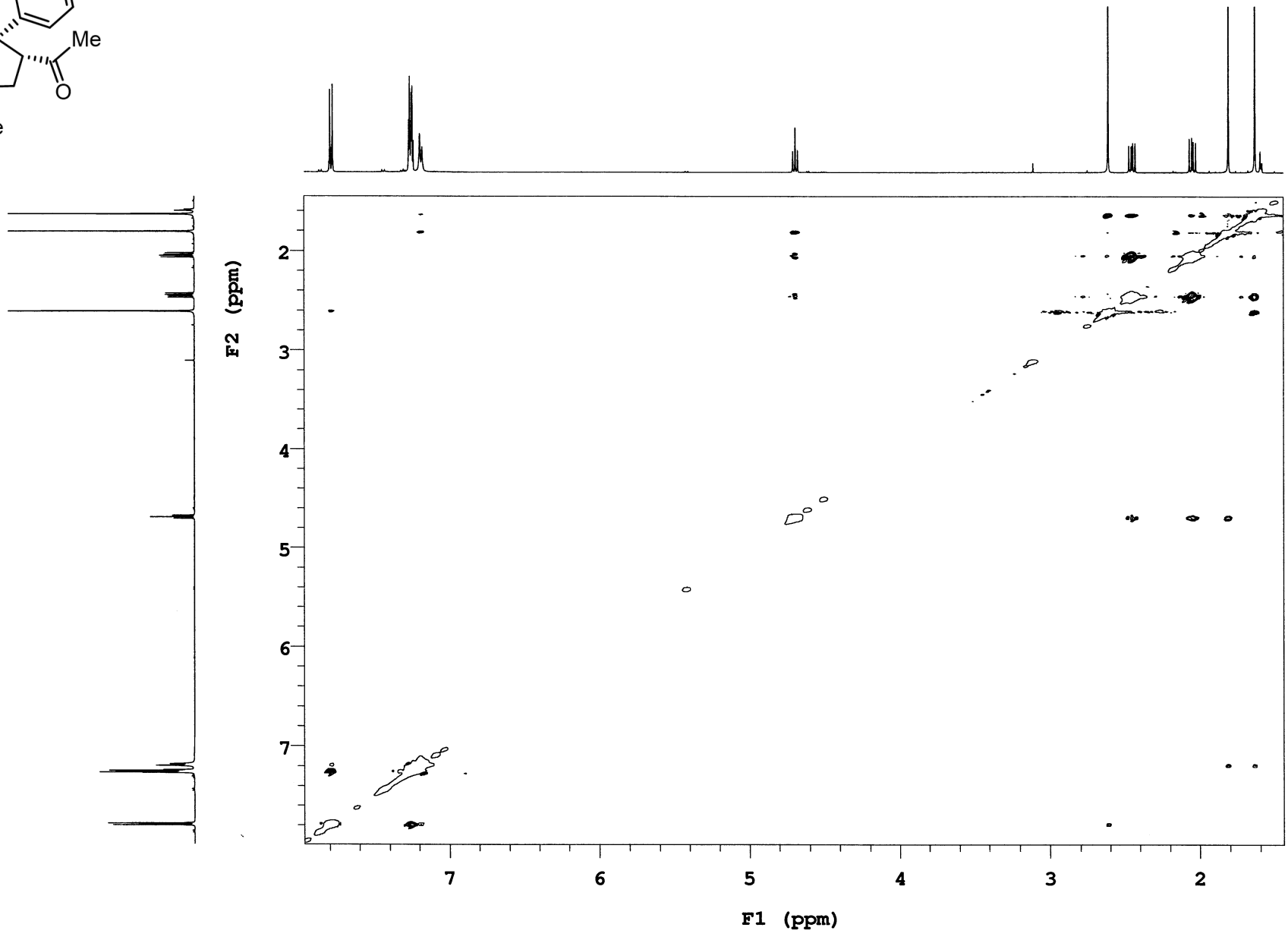
COSY of compound 2gb



2gb

IRR-03-075

Sample Name	**IRR-03-075**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-04**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2gb

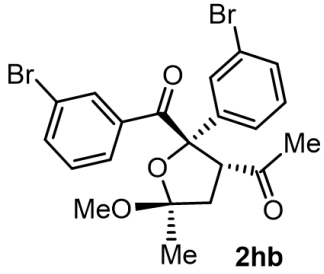
IRR-03-077

Sample Name	**IRR-03-077**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-16**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

8.049 8.045 8.042 7.707 7.706 7.705 7.692 7.691 7.689 7.548 7.546 7.544 7.542 7.532 7.530 7.528 7.526 7.446 7.410 7.406 7.397 7.392 7.388 7.240 7.173 7.160 7.155 7.139 7.124

4.678 4.662 4.647

2.608 2.458 2.442 2.432 2.416 2.062 2.046 2.036 2.020 1.817 1.650 1.605



2hb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-077/PROTON_03

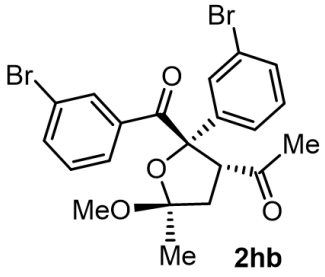
1H NMR (500 MHz, CDCl3) of compound 2hb

Plot date 2024-06-17

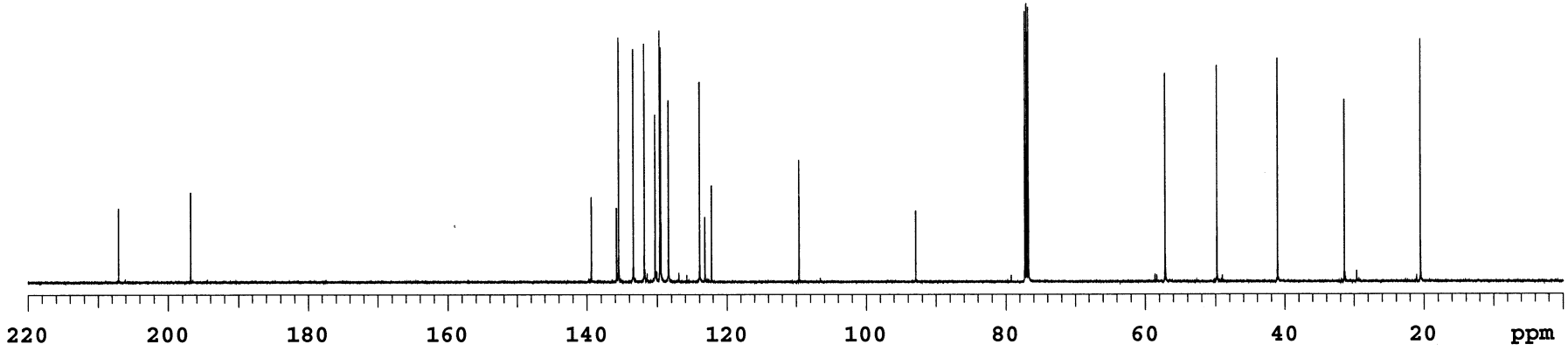
IRR-03-077

Sample Name **IRR-03-077**	Pulse sequence **CARBON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-06-16**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

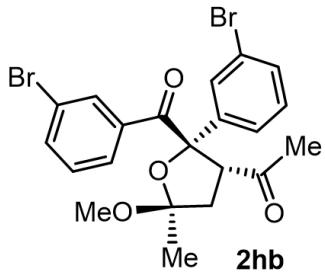
207.051
196.854
139.385
135.804
135.507
133.401
131.830
130.289
129.618
129.465
128.384
123.944
123.190
122.251
109.602
92.941
77.254
77.000
76.744
57.170
49.802
41.080
31.438
20.500



2hb



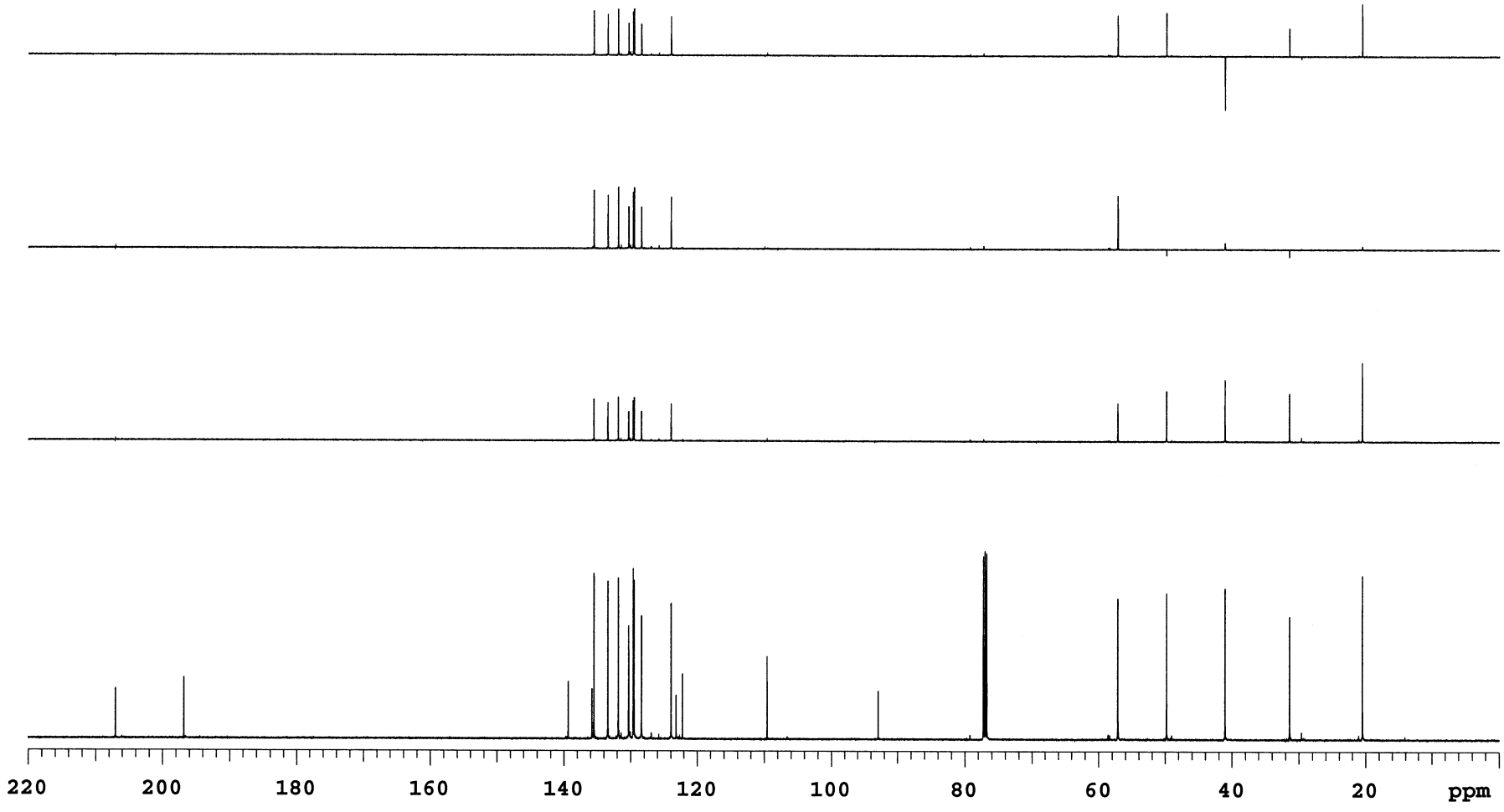
13C NMR (125 MHz, CDCl3) of compound 2hb



2hb

IRR-03-077

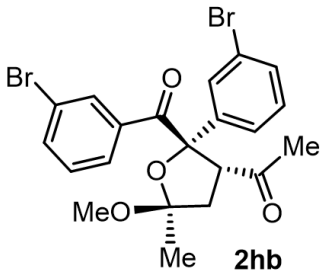
Sample Name	**IRR-03-077**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-17**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



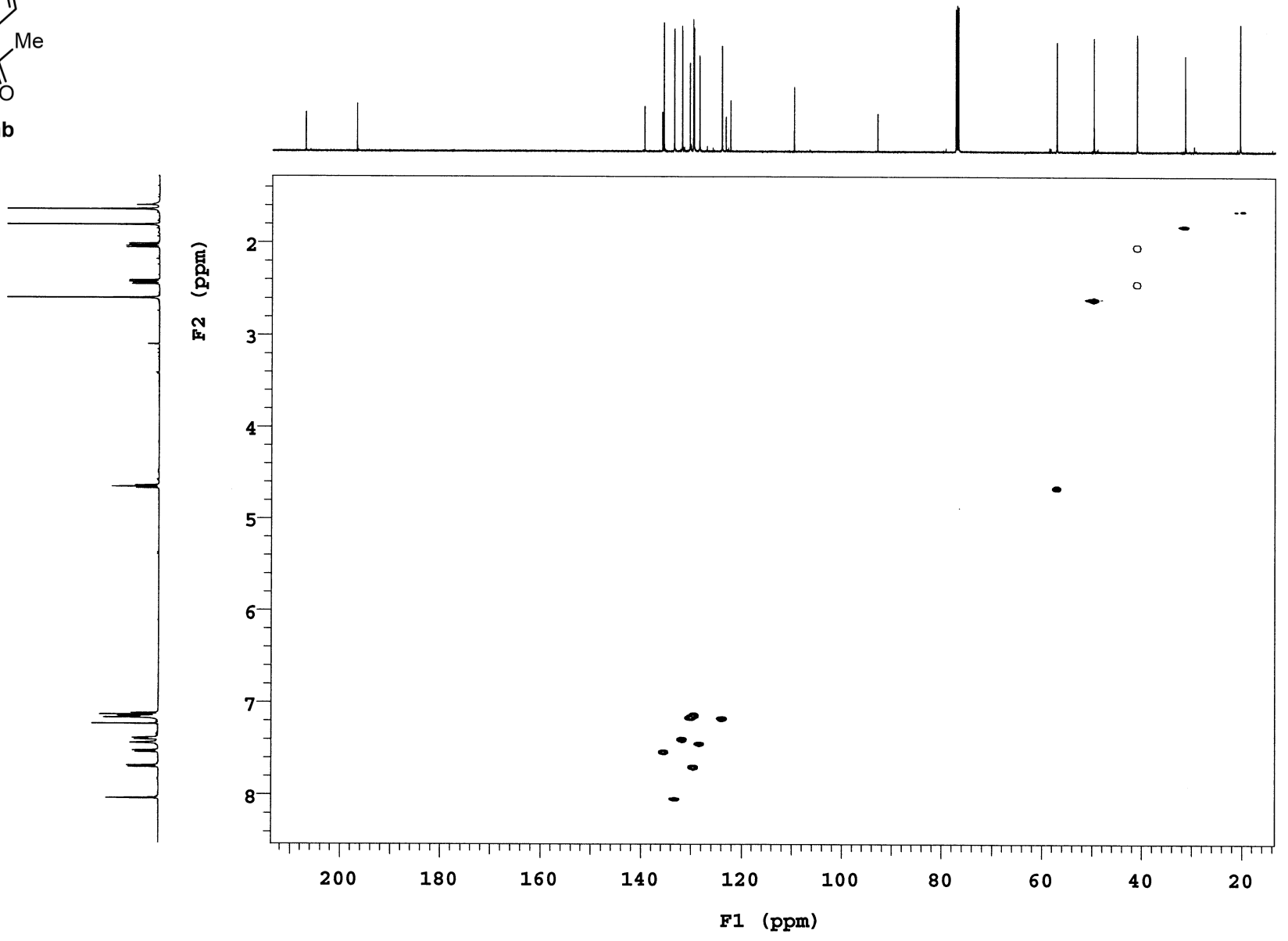
DEPT of compound 2hb

IRR-03-077

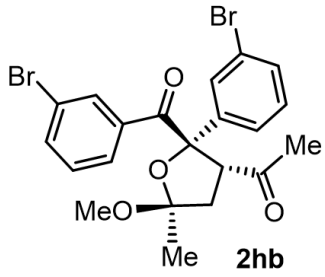
Sample Name	**IRR-03-077**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-17**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2hb



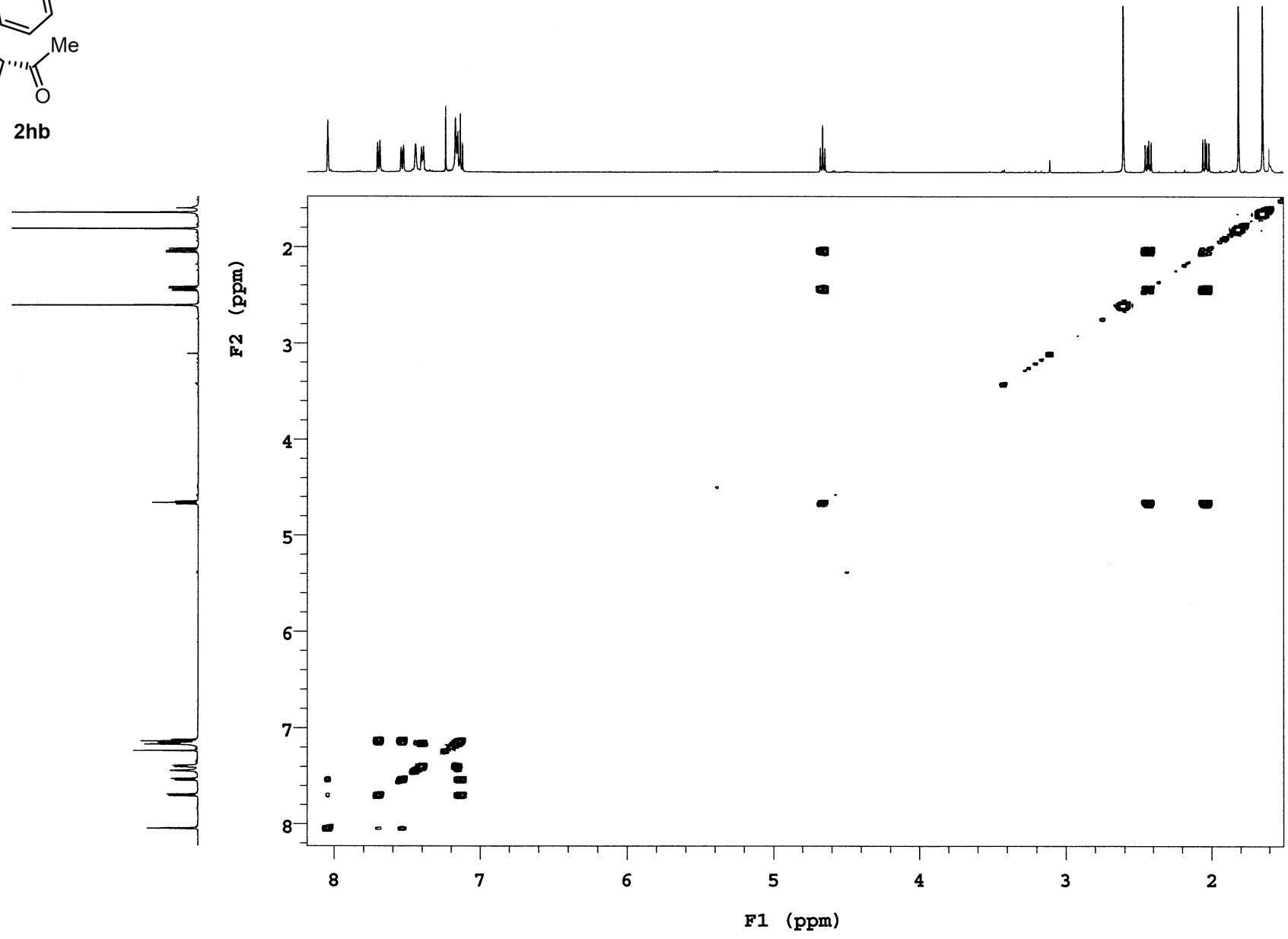
HSQC of compound 2hb



2hb

IRR-03-077

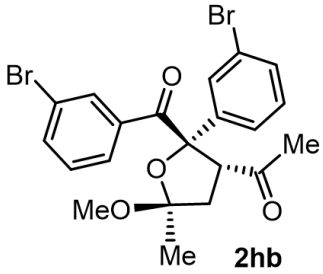
Sample Name	**IRR-03-077**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-17**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



COSY of compound 2hb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-077/gCOSY_01

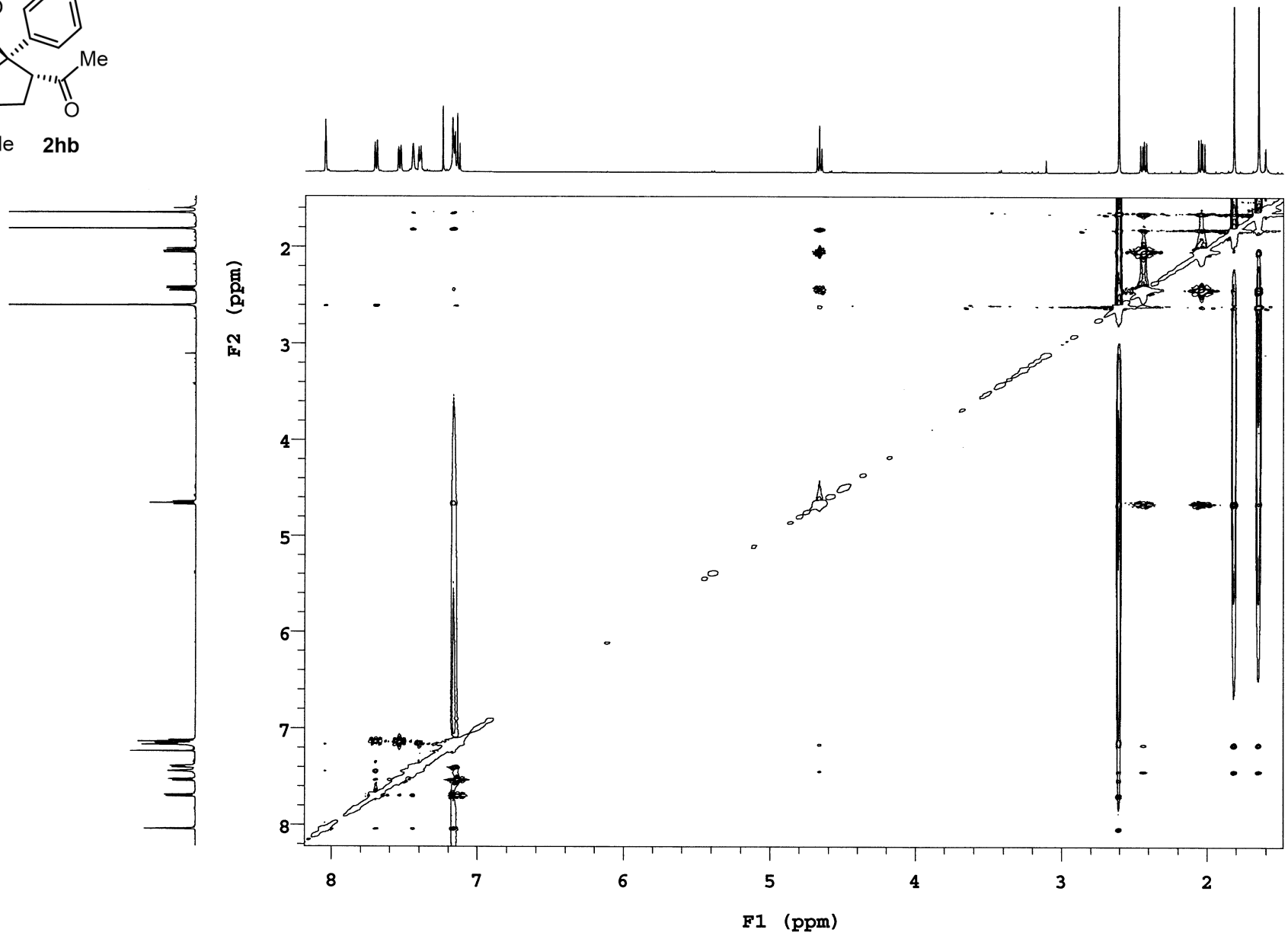
Plot date 2024-06-17



2hb

IRR-03-077

Sample Name	**IRR-03-077**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-17**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



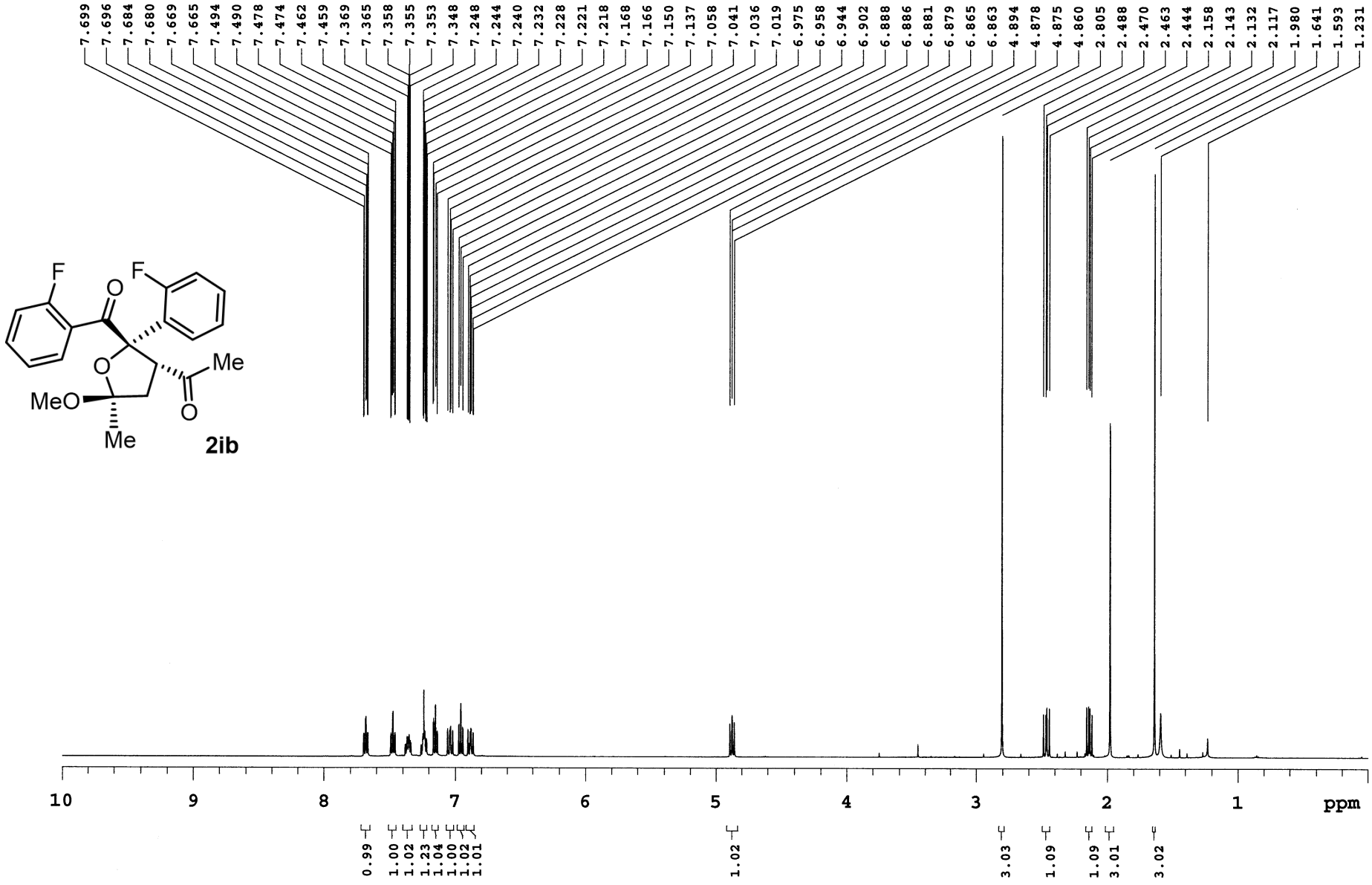
NOESY of compound 2hb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-077/NOESY_01

Plot date 2024-06-17

IRR-03-085

Sample Name **IRR-03-085**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-06-21**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



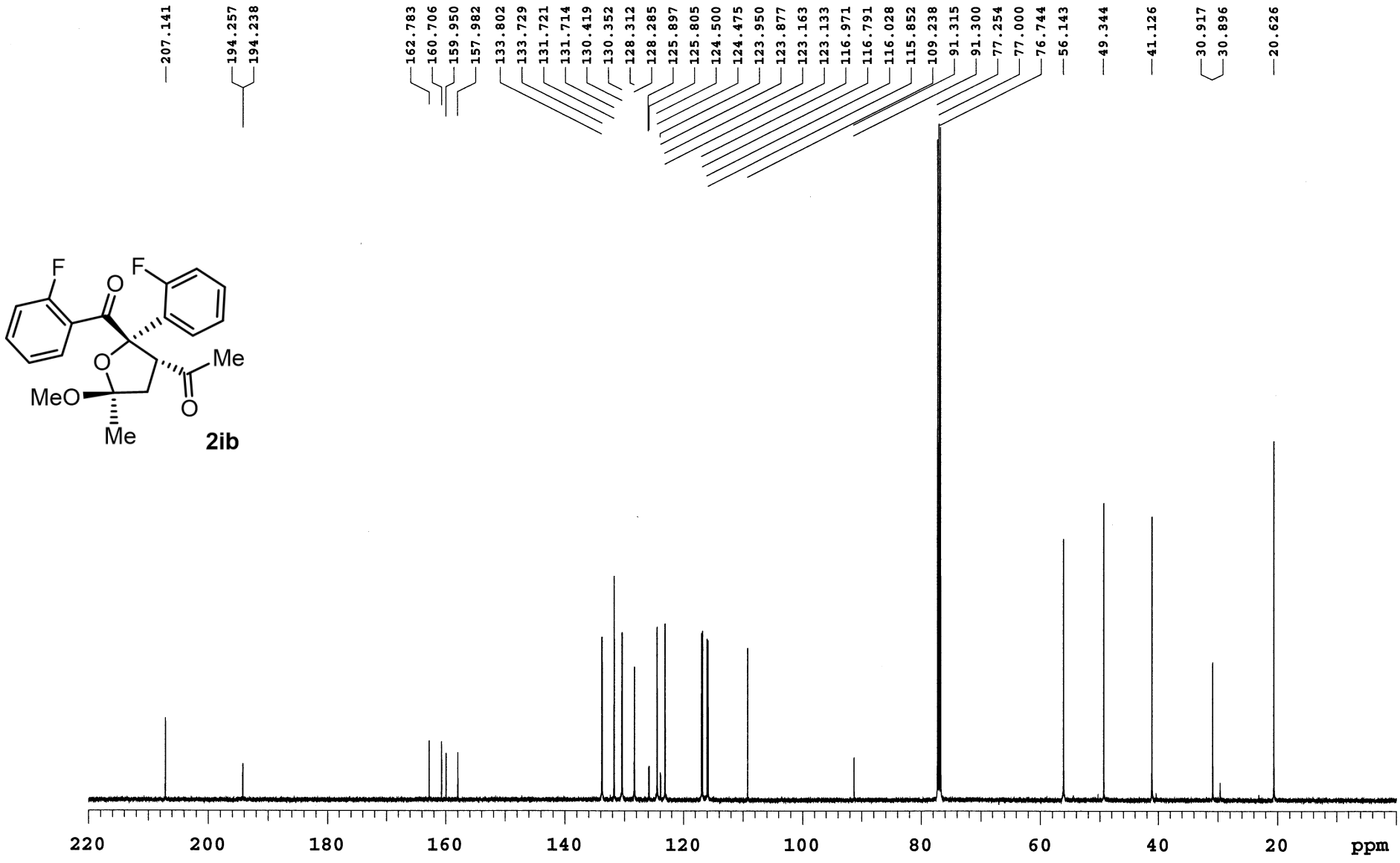
1H NMR (500 MHz, CDCl3) of compound 2ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-085/PROTON_03

Plot date 2024-06-25

IRR-03-085

Sample Name	IRR-03-085	Pulse sequence	CARBON	Temperature	25	Study owner	vnmr2
Date collected	2024-06-21	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



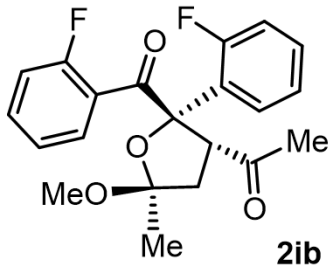
13C NMR (125 MHz, CDCl3) of compound 2ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-085/CARBON_02

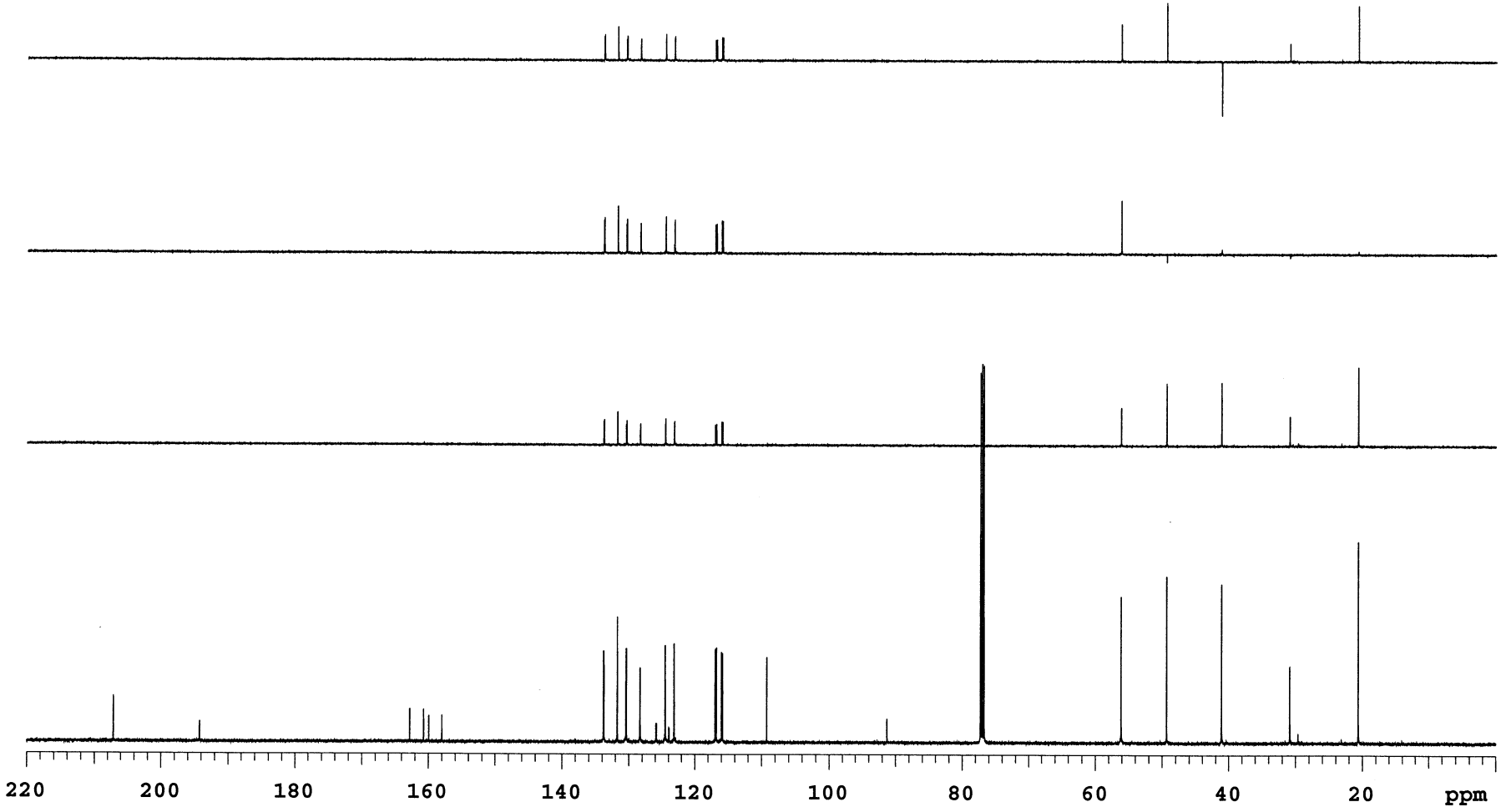
Plot date 2024-06-25

IRR-03-085

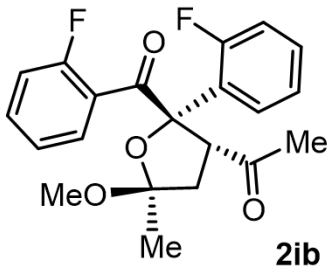
Sample Name	**IRR-03-085**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2ib



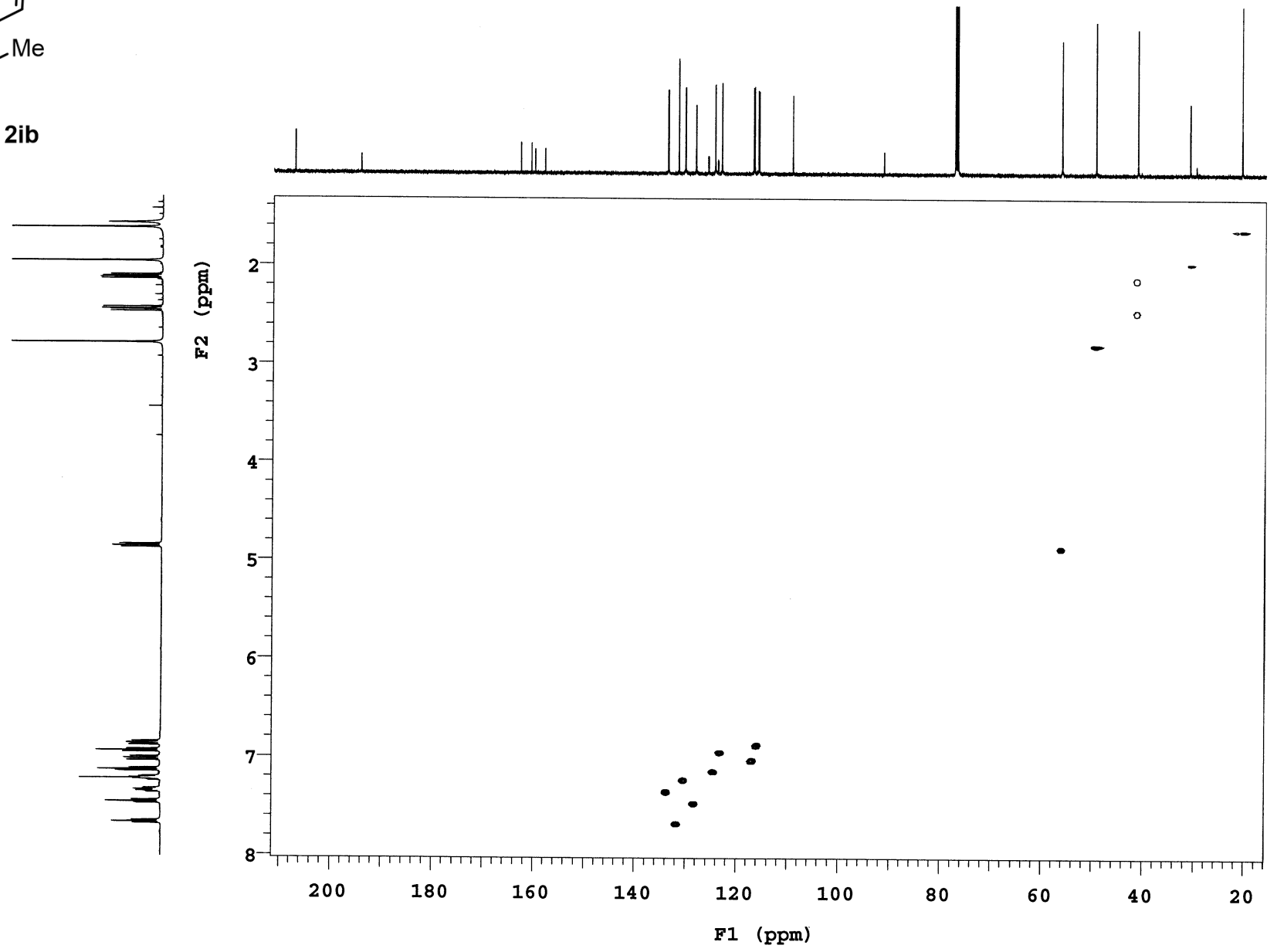
DEPT of compound 2ib



2ib

IRR-03-085

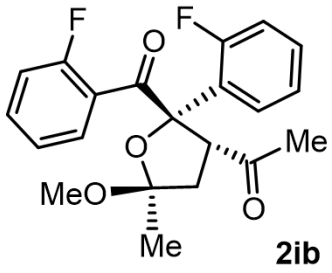
Sample Name	**IRR-03-085**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-085/gHSQC_01

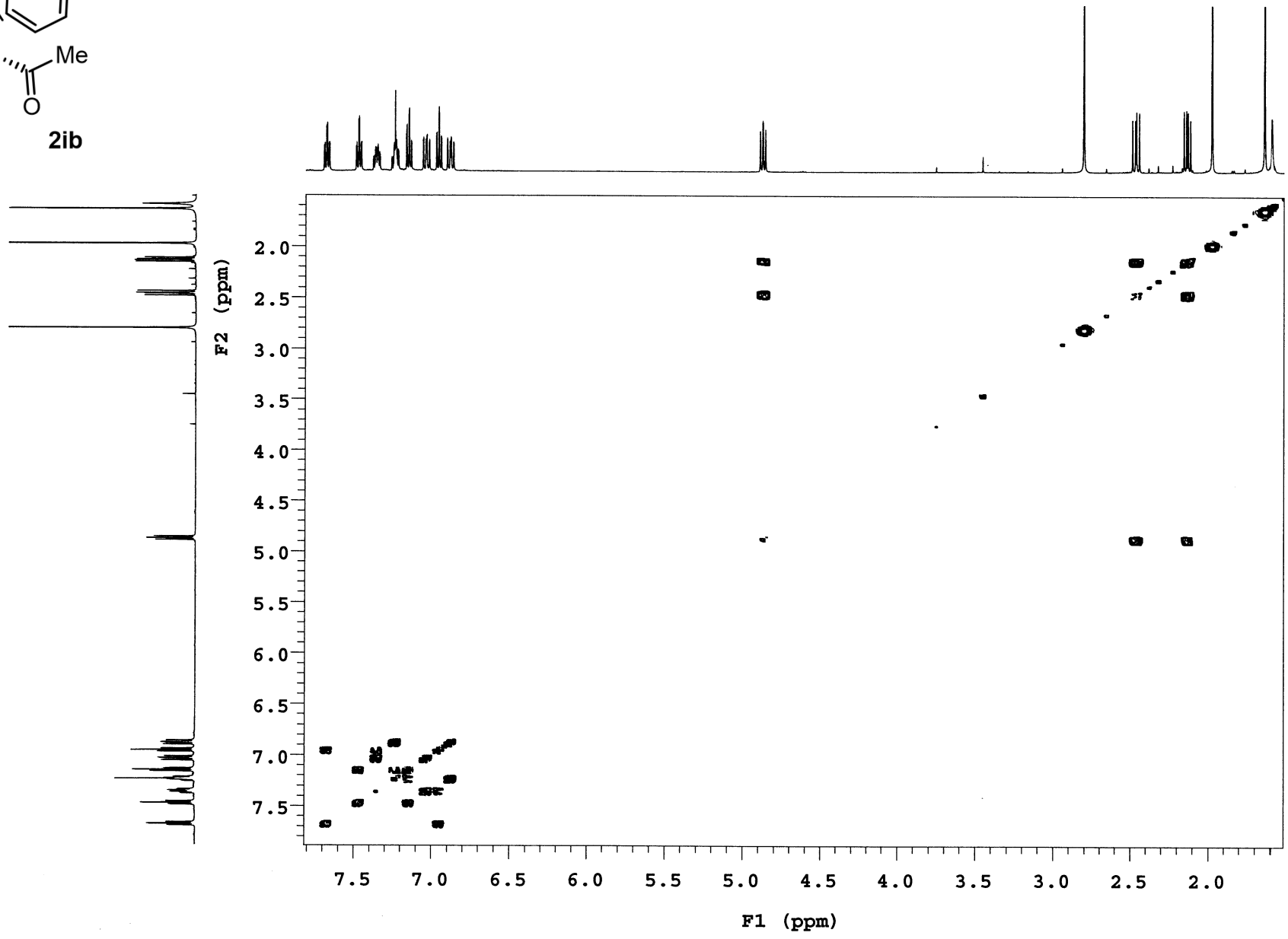
Plot date 2024-06-25



2ib

IRR-03-085

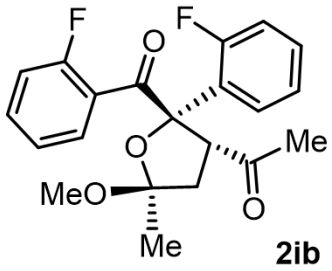
Sample Name	**IRR-03-085**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



COSY of compound 2ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-085/gCOSY_01

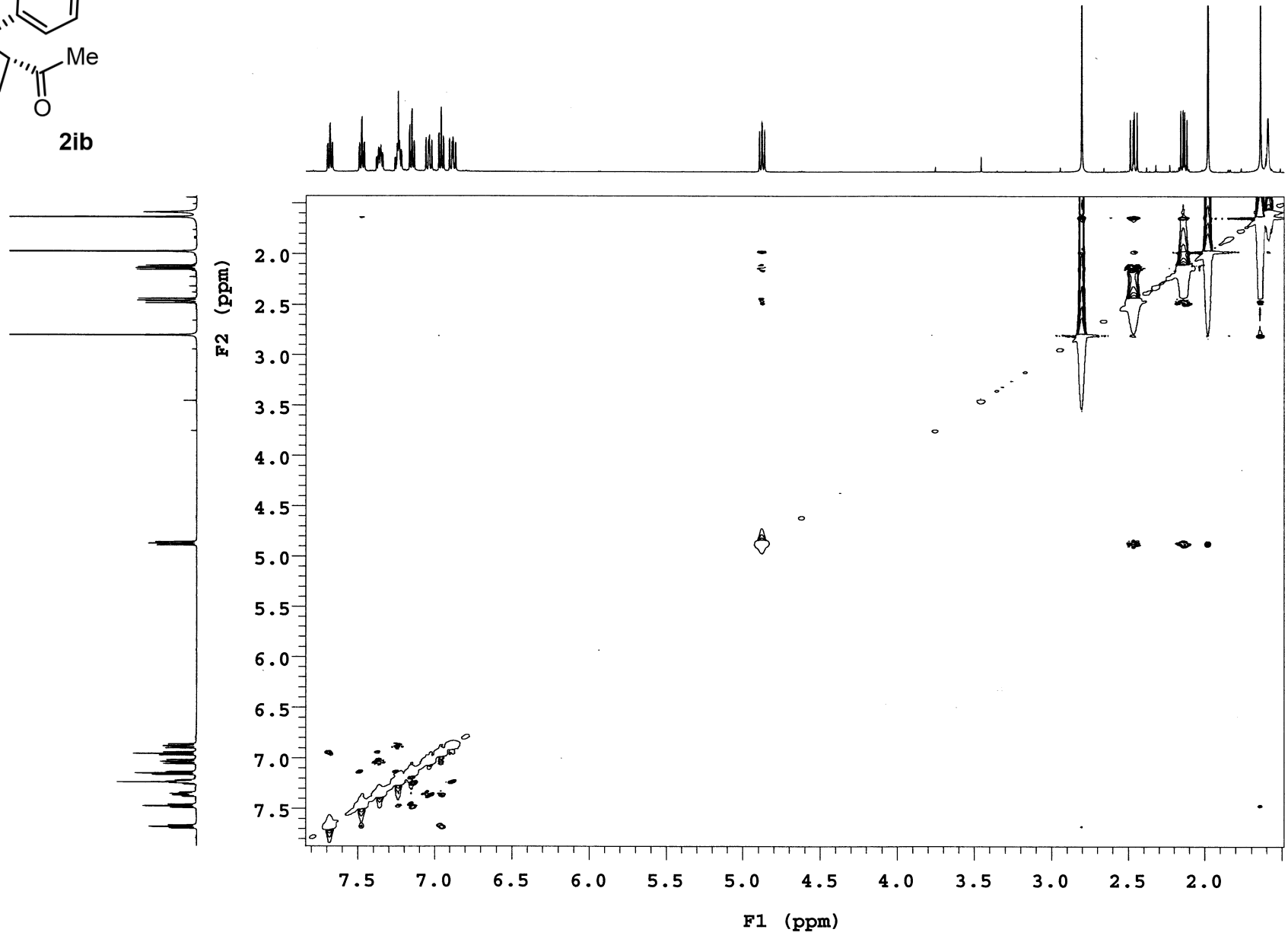
Plot date 2024-06-25



2ib

IRR-03-085

Sample Name	**IRR-03-085**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-22**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



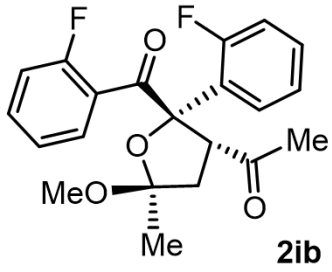
NOESY of compound 2ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-085/NOESY_01

Plot date 2024-06-25

IRR-03-085

Sample Name	**IRR-03-085**	Pulse sequence	**FLUORINE**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-24**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2ib

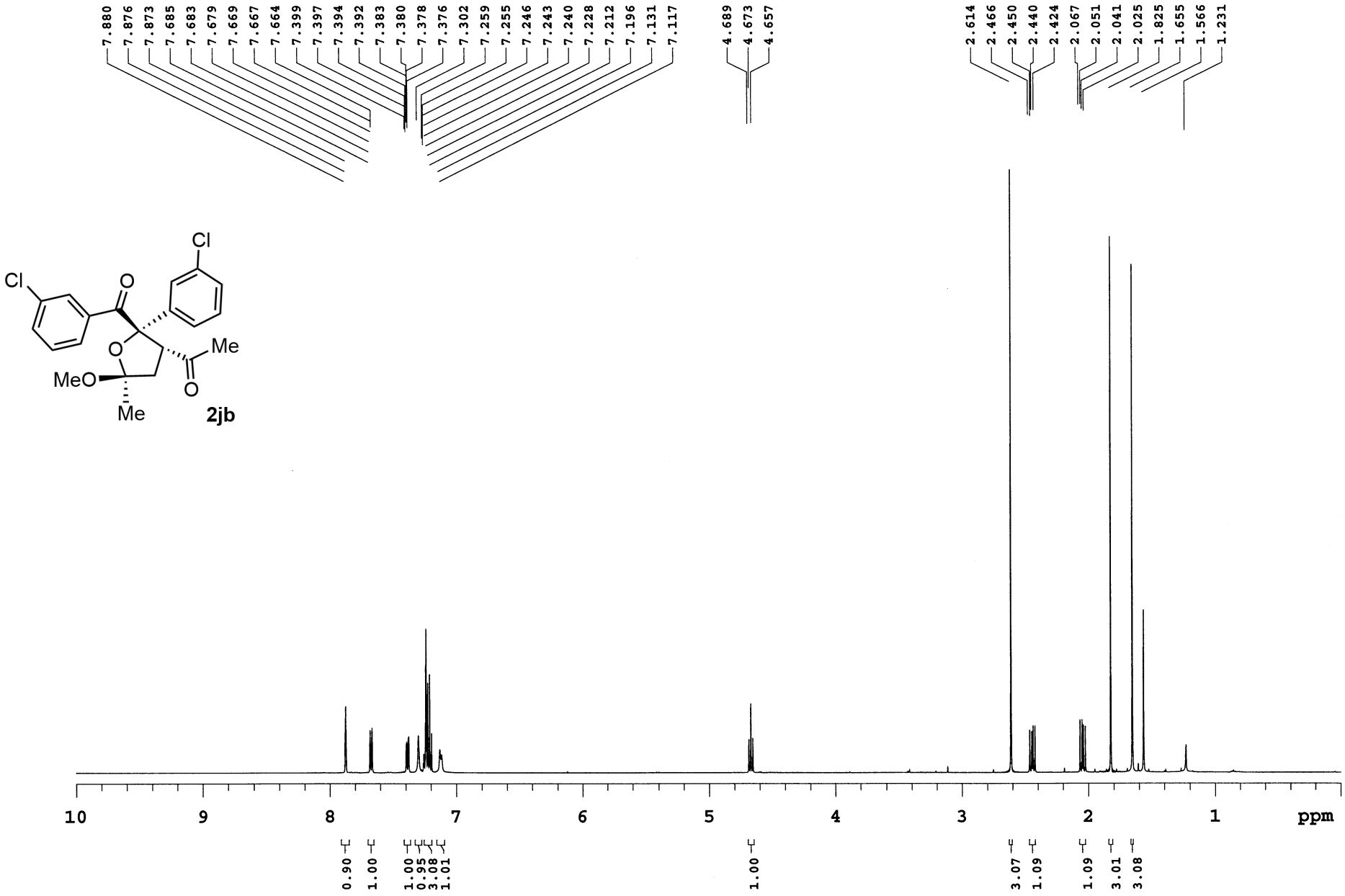
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-085/FLUORINE_04

19F NMR (470 MHz, CDCl3) of compound 2ib

Plot date 2024-06-25

IRR-03-086

Sample Name **IRR-03-086**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-06-20**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-086/PROTON_03

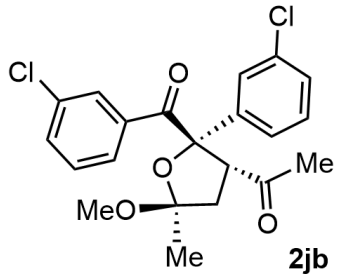
1H NMR (500 MHz, CDCl3) of compound 2jb

Plot date 2024-06-21

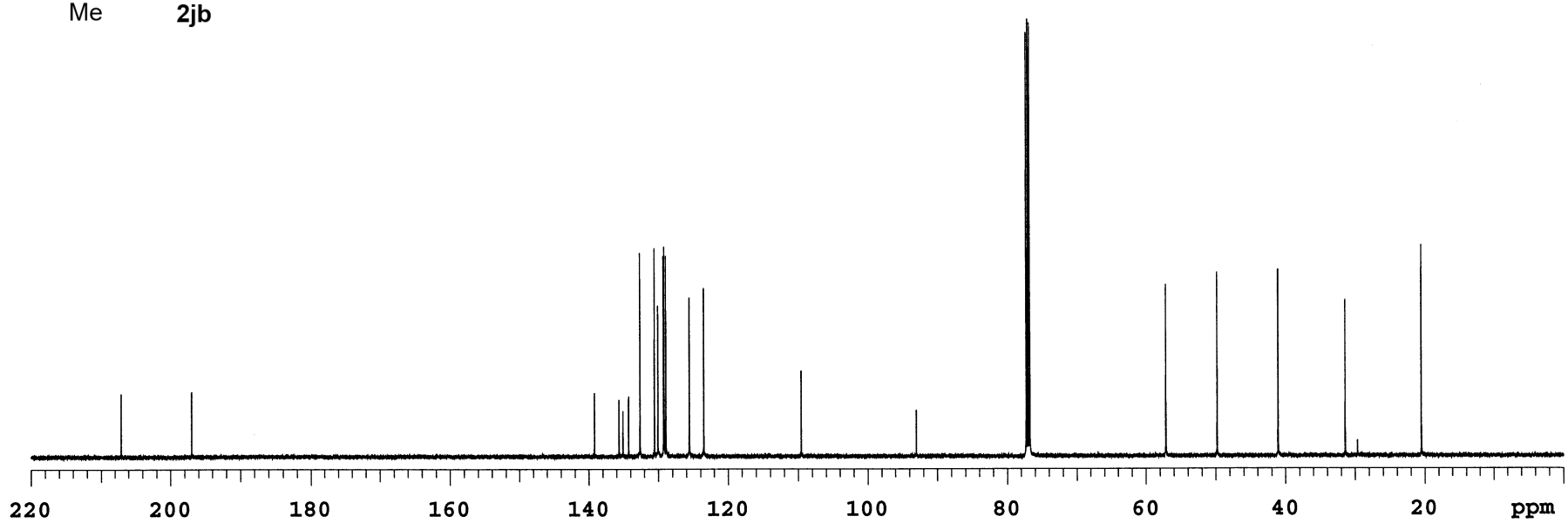
IRR-03-086

Sample Name	**IRR-03-086**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-20**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

207.095
197.002
139.217
135.648
135.081
134.260
132.620
130.536
130.049
129.243
129.154
128.904
125.574
123.503
109.591
93.048
77.254
77.000
76.746
57.161
49.798
41.087
31.451
20.500



2jb



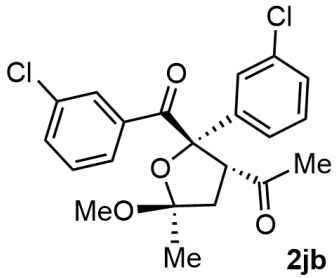
13C NMR (125 MHz, CDCl3) of compound 2jb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-086/CARBON_02

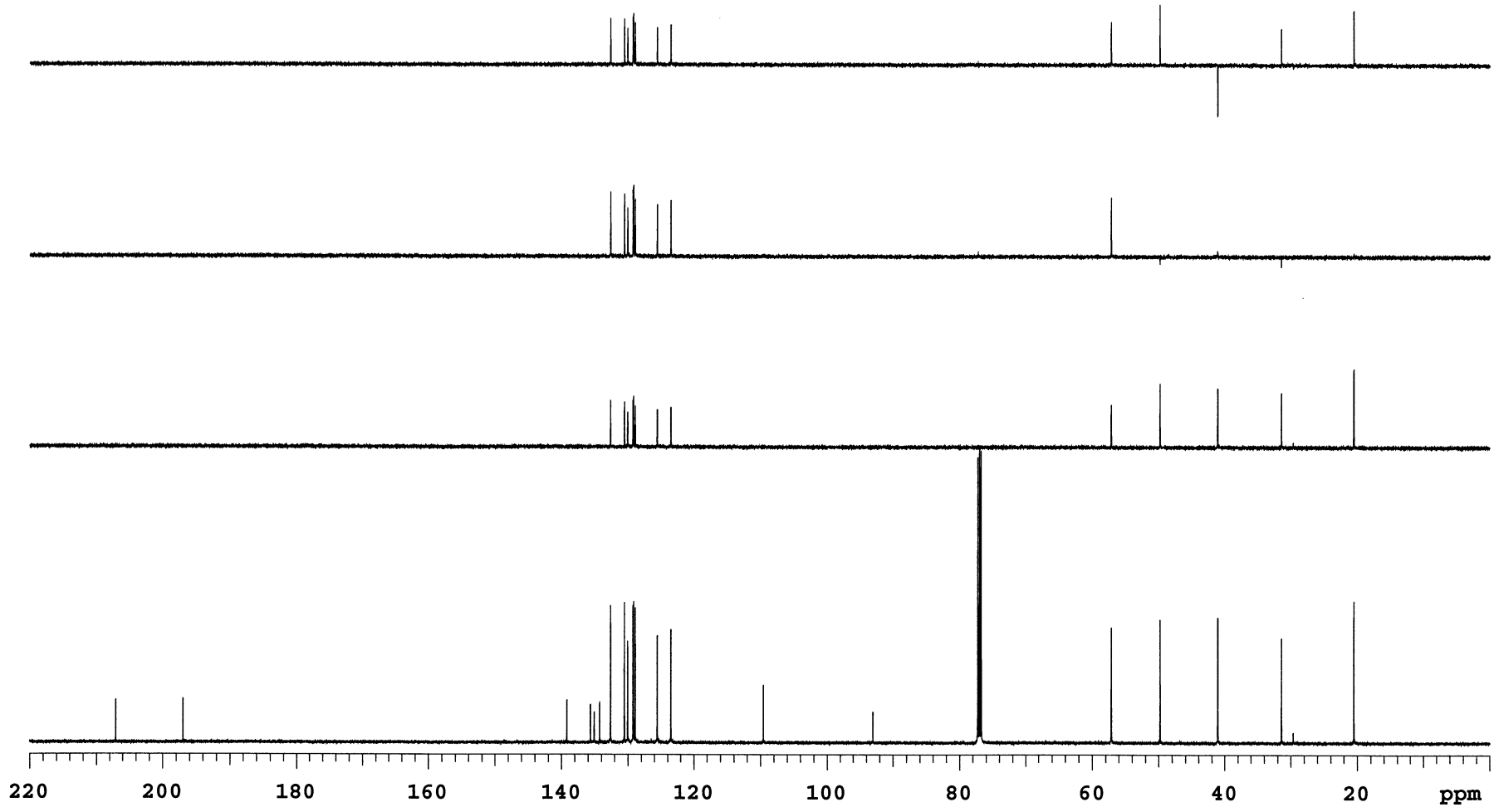
Plot date 2024-06-21

IRR-03-086

Sample Name	**IRR-03-086**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



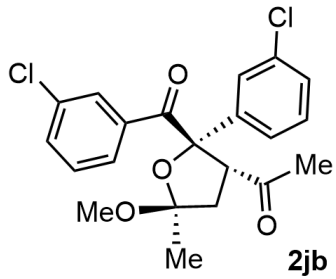
2jb



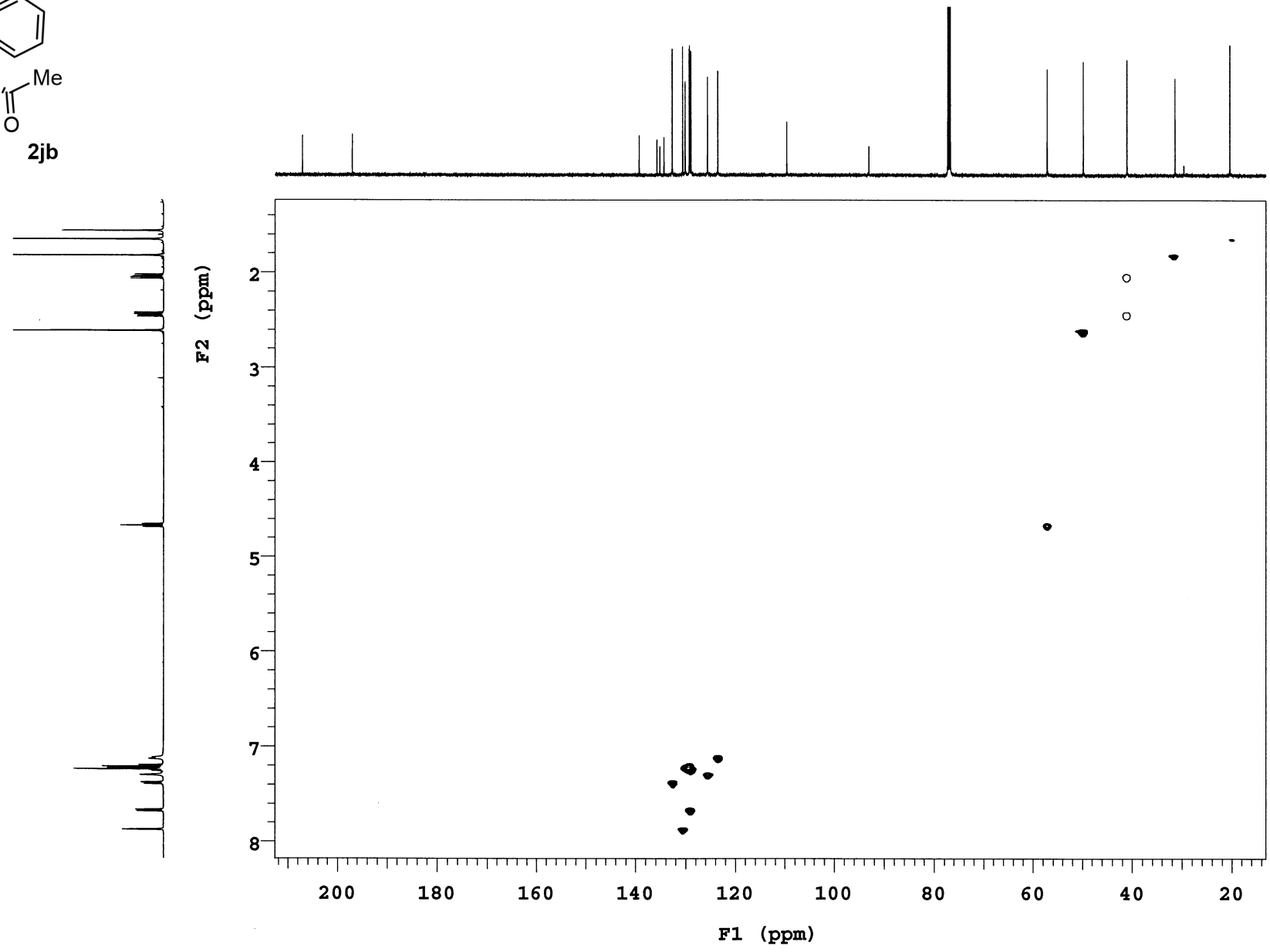
DEPT of compound 2jb

IRR-03-086

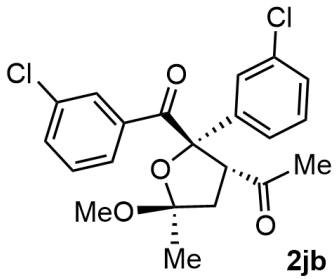
Sample Name	IRR-03-086	Pulse sequence	gHSQC	Temperature	25	Study owner	vnmr2
Date collected	2024-06-21	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



2jb



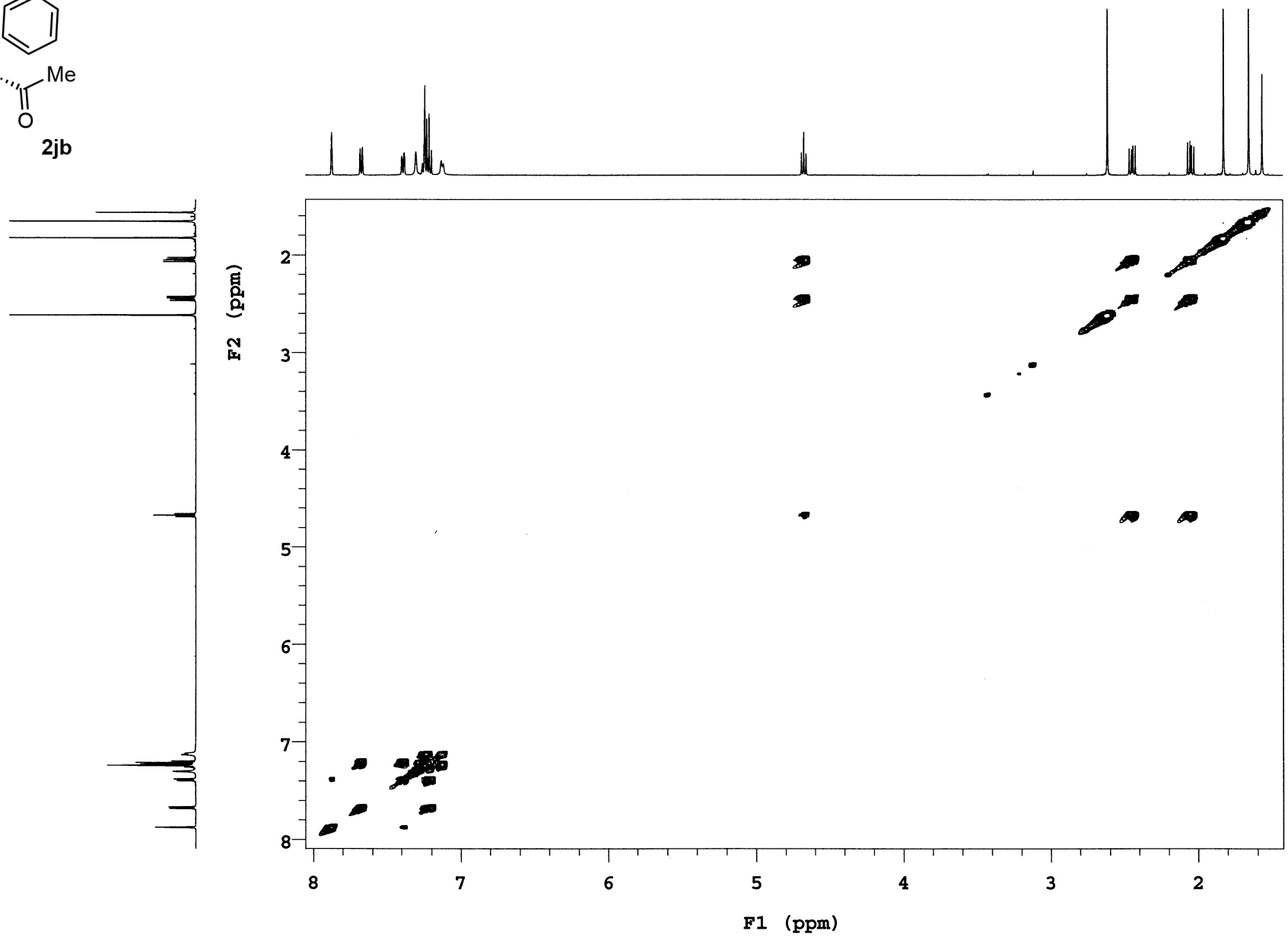
HSQC of compound 2jb



2jb

IRR-03-086

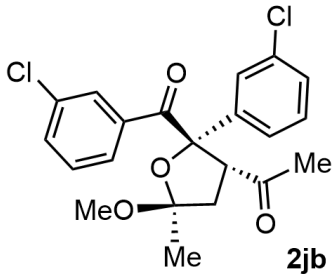
Sample Name	IRR-03-086	Pulse sequence	gCOSY	Temperature	25	Study owner	vnmr2
Date collected	2024-06-21	Solvent	cdcl3	Spectrometer	Agilent-NMR-inova500	Operator	vnmr2



COSY of compound 2jb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-086/gCOSY_01

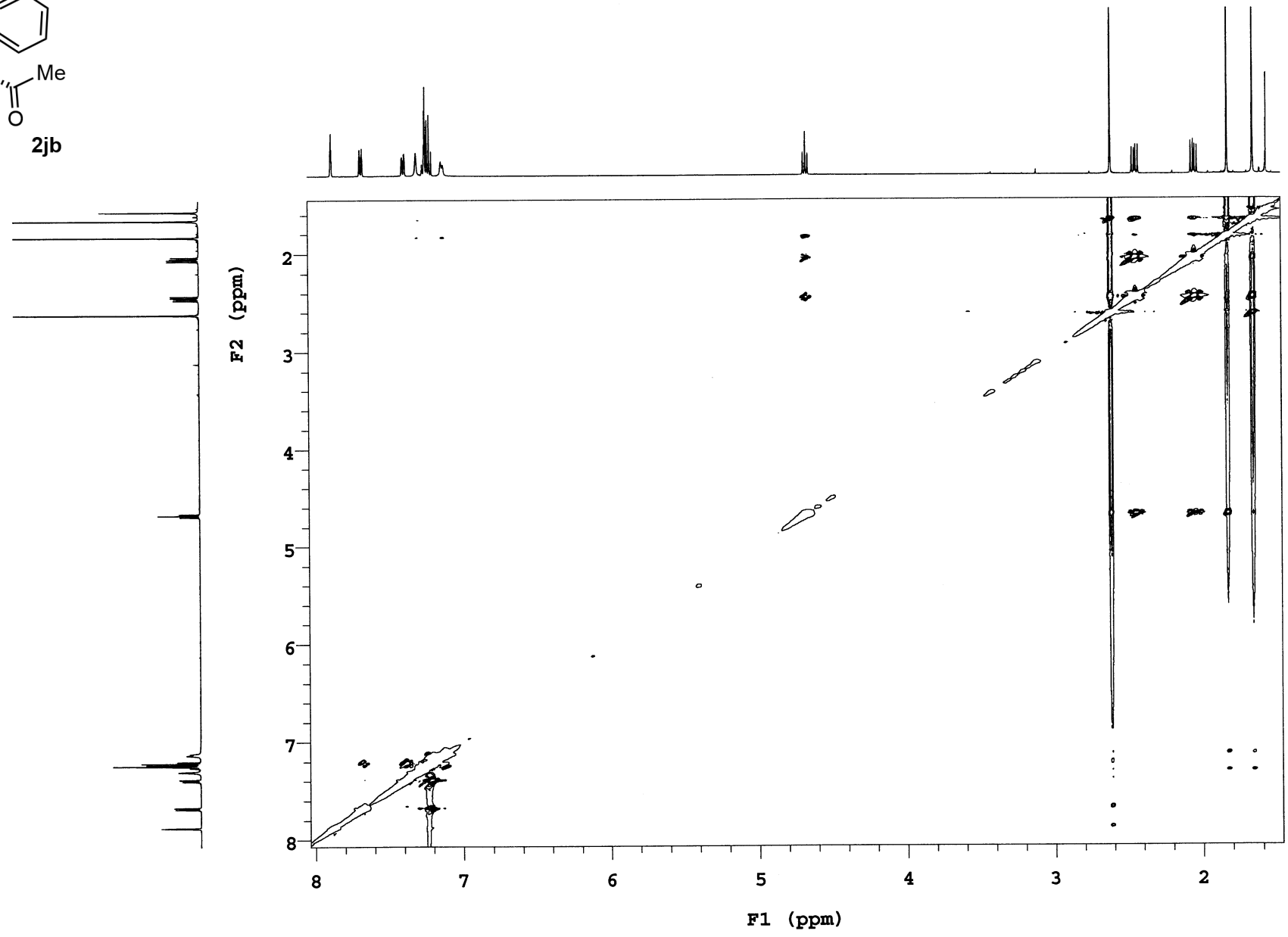
Plot date 2024-06-21



2jb

IRR-03-086

Sample Name	**IRR-03-086**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-21**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



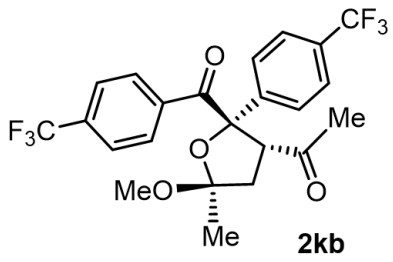
NOESY of compound 2jb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-086/NOESY_01

Plot date 2024-06-21

IRR-03-021

Sample Name	**IRR-03-021**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



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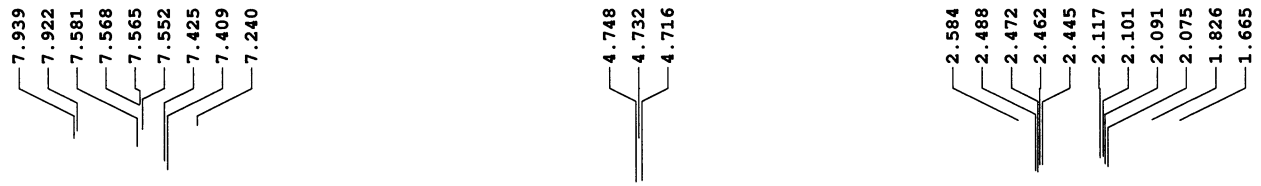
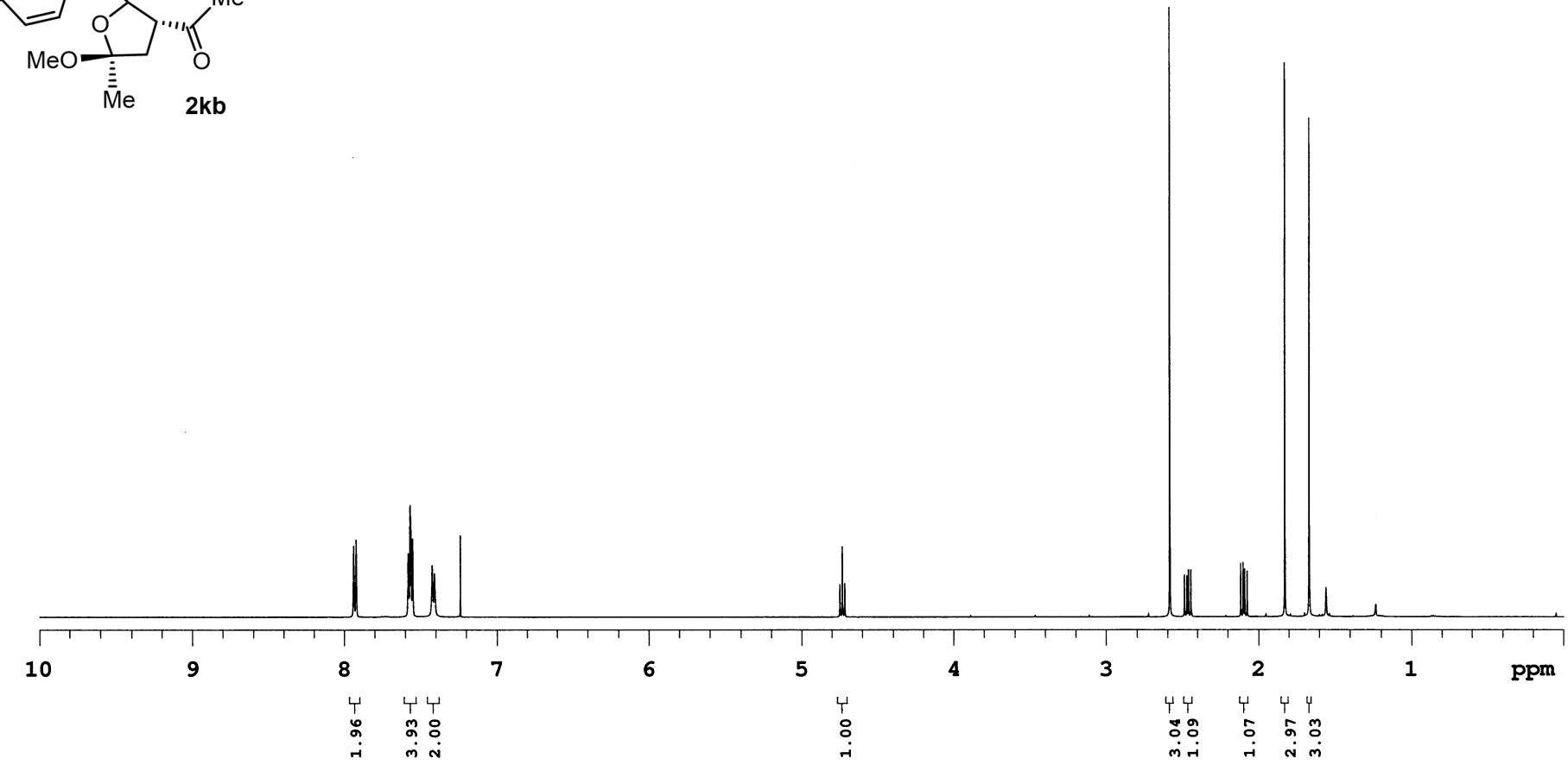




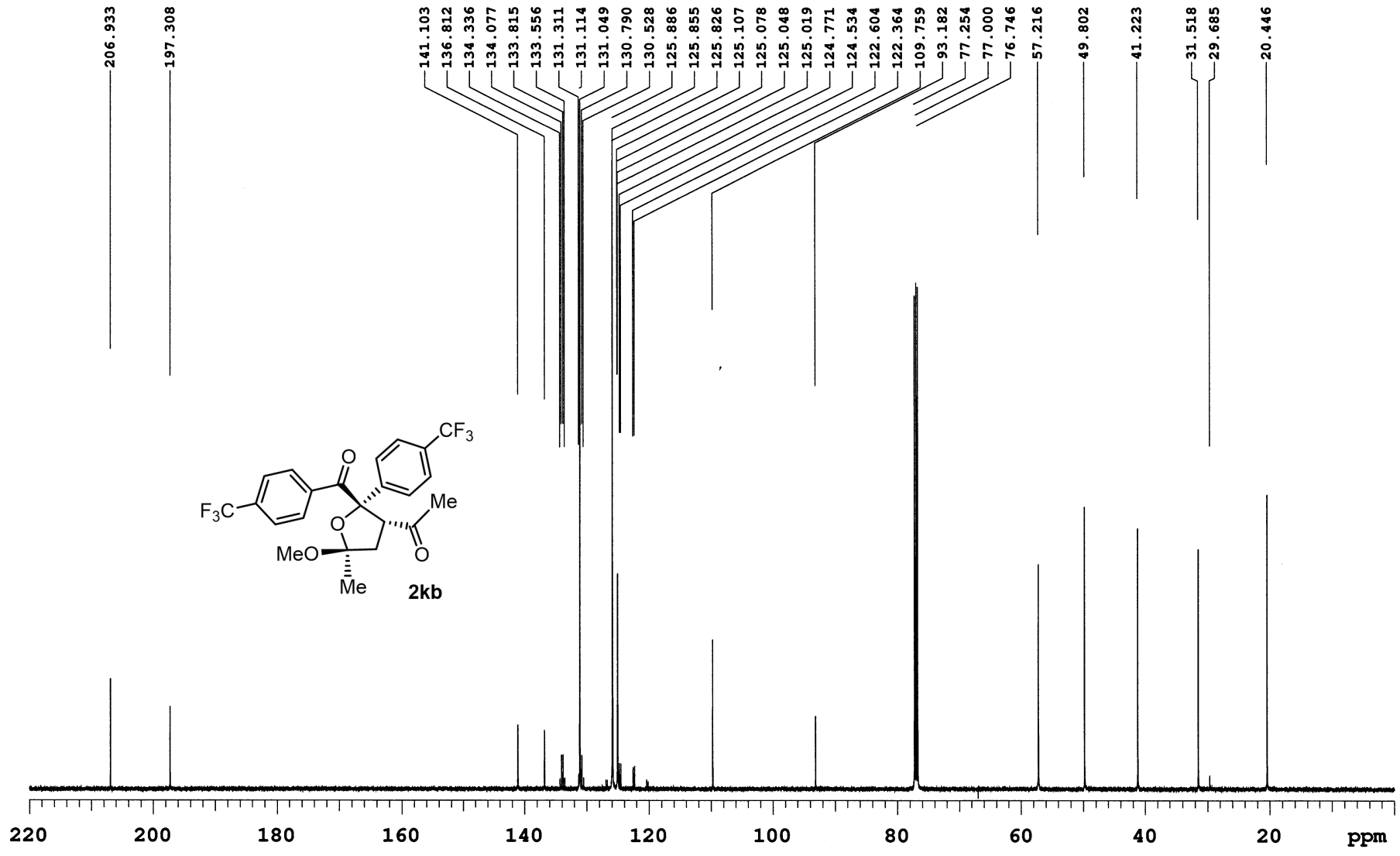
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-021/PROTON_03

1H NMR (500 MHz, CDCl3) of compound 2kb

Plot date 2024-03-11

IRR-03-021

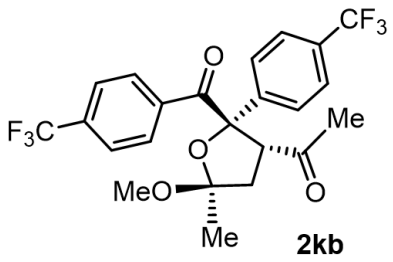
Sample Name	**IRR-03-021**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



13C NMR (125 MHz, CDCl3) of compound 2kb

IRR-03-021

Sample Name	**IRR-03-021**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



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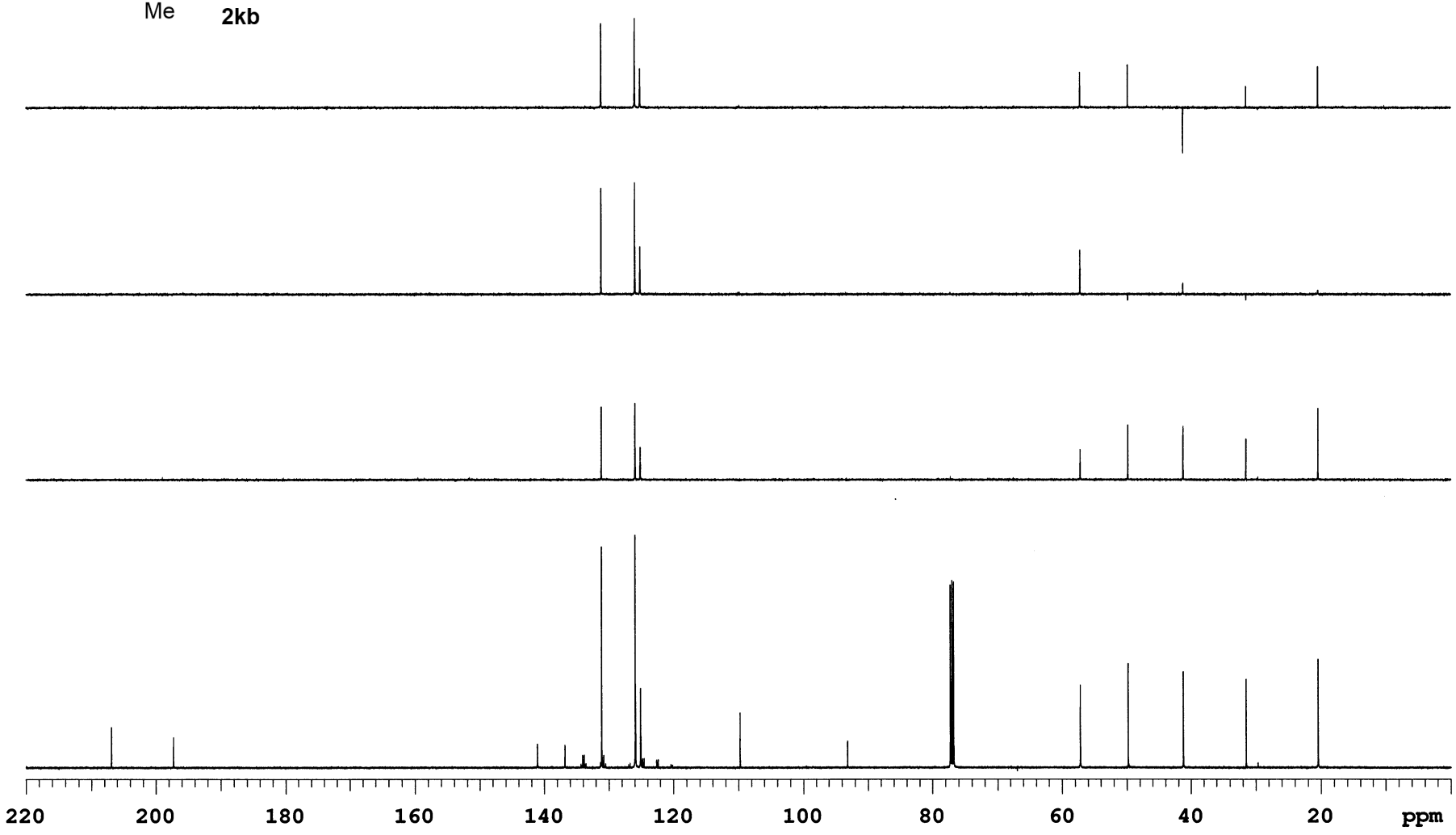
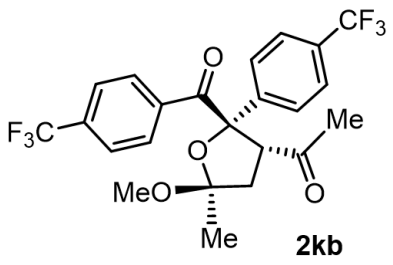


DEPT of compound 2kb



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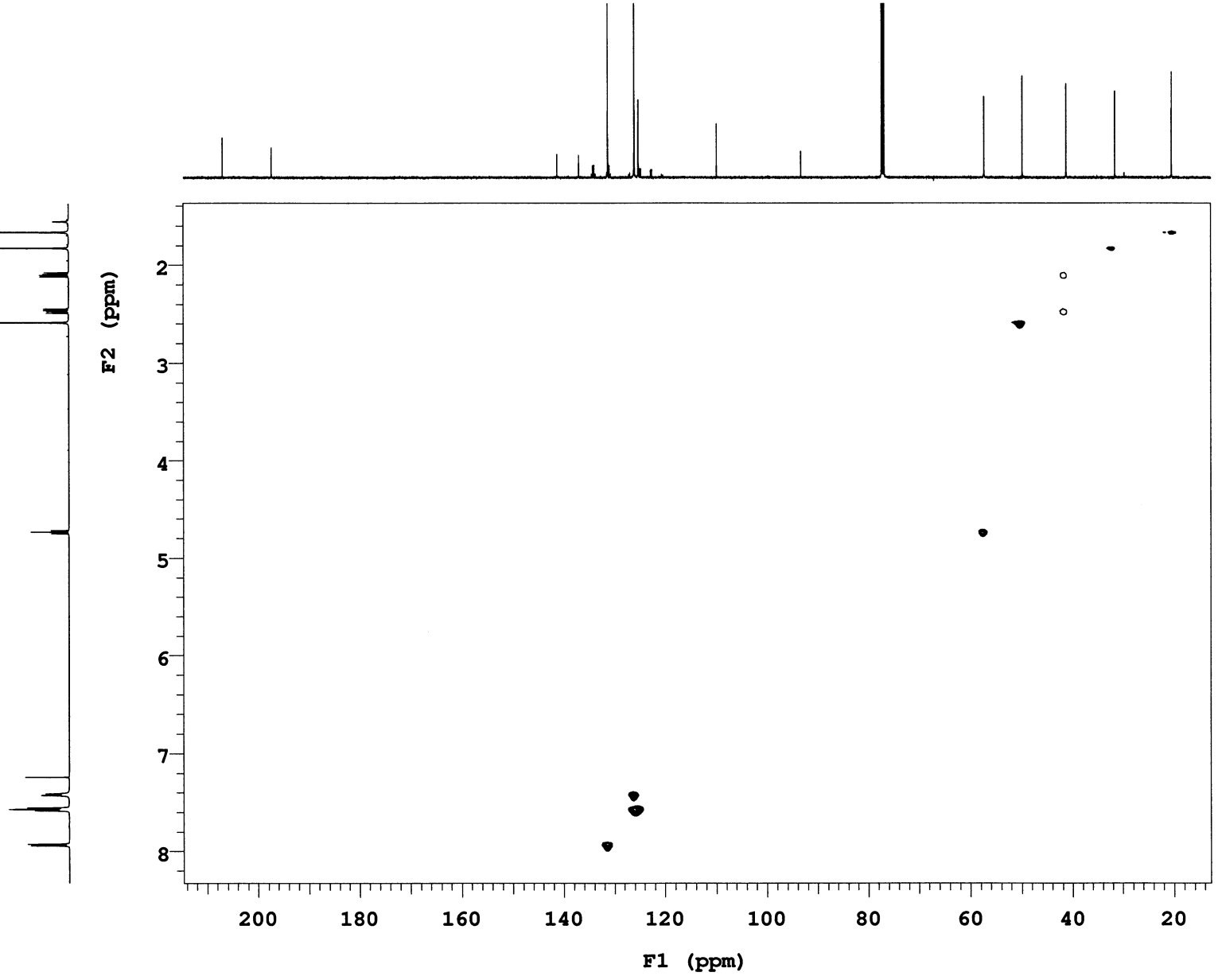
IRR-03-021

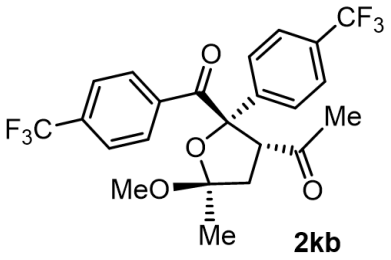
Sample Name	**IRR-03-021**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-09**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2kb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-021/gHSQC_01

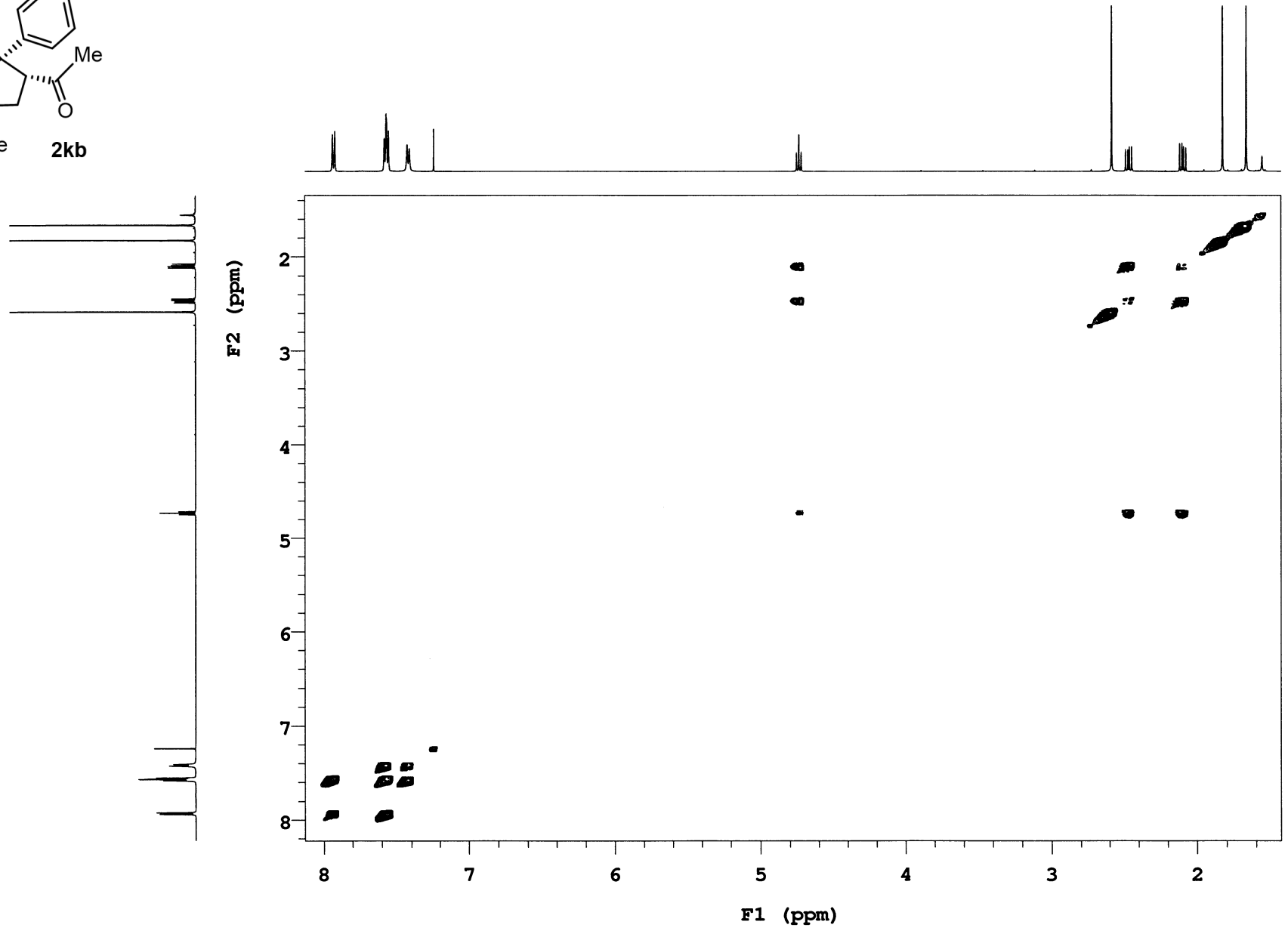
Plot date 2024-03-11



2kb

IRR-03-021

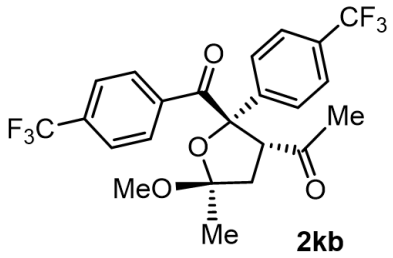
Sample Name	**IRR-03-021**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-09**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



COSY of compound 2kb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-021/gCOSY_01

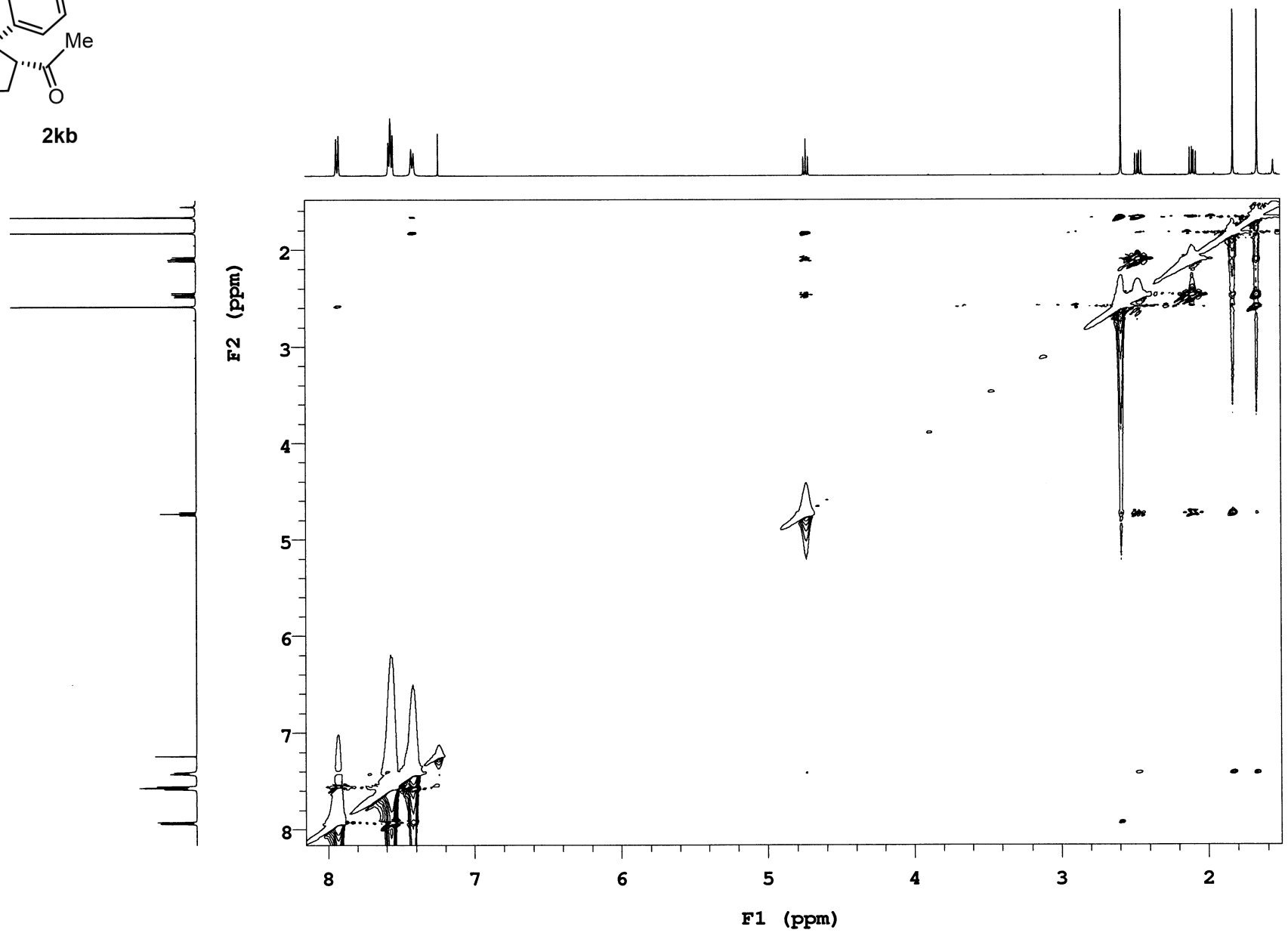
Plot date 2024-03-11



2kb

IRR-03-021

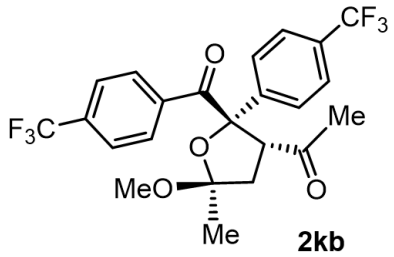
Sample Name	**IRR-03-021**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-09**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2kb

IRR-03-021

Sample Name	**IRR-03-021**	Pulse sequence	**FLUORINE**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-03-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



2kb

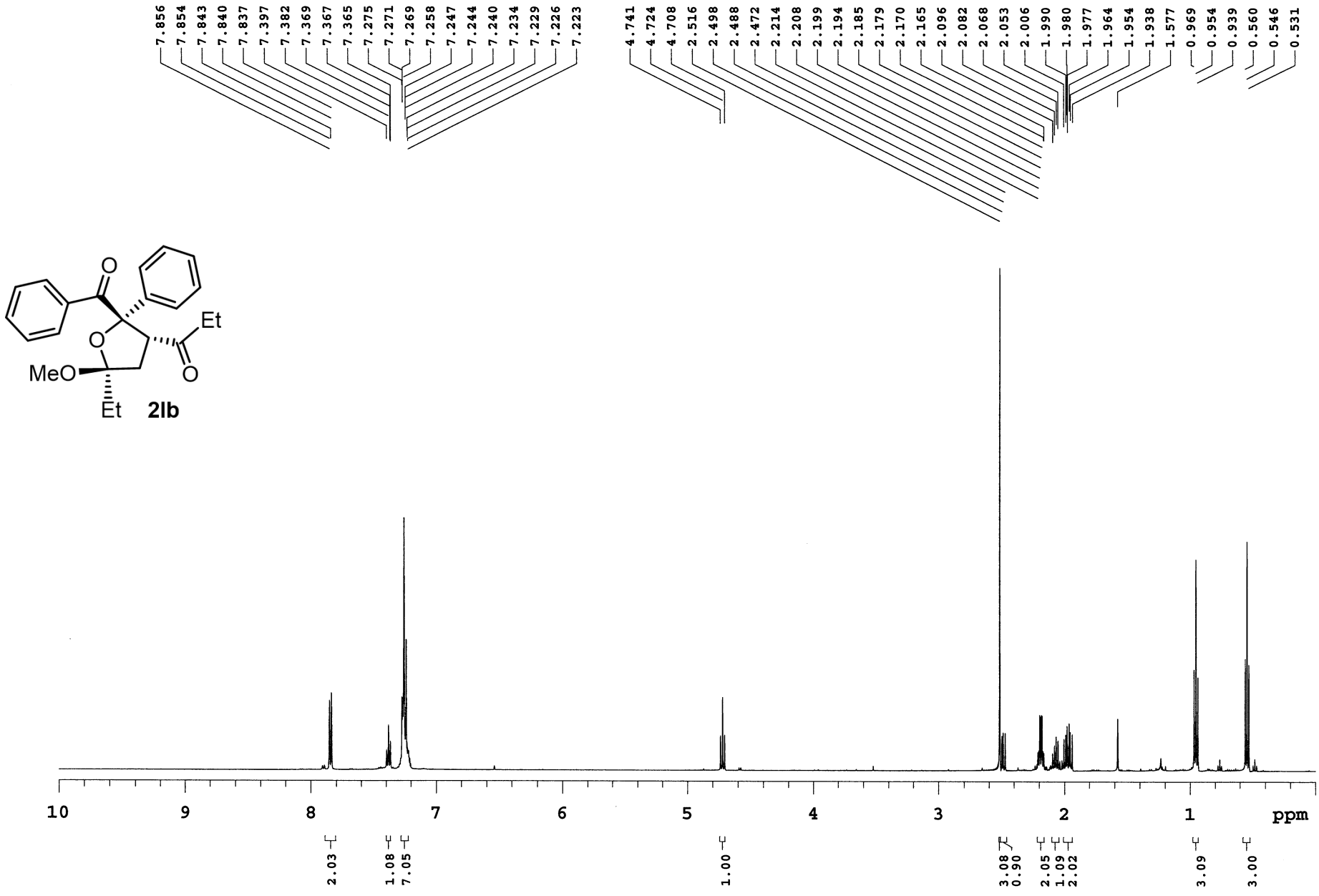
Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-021/FLUORINE_02

Plot date 2024-03-11

19F NMR (470 MHz, CDCl3) of compound 2kb

IRR-03-074

Sample Name **IRR-03-074**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-06-15**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**



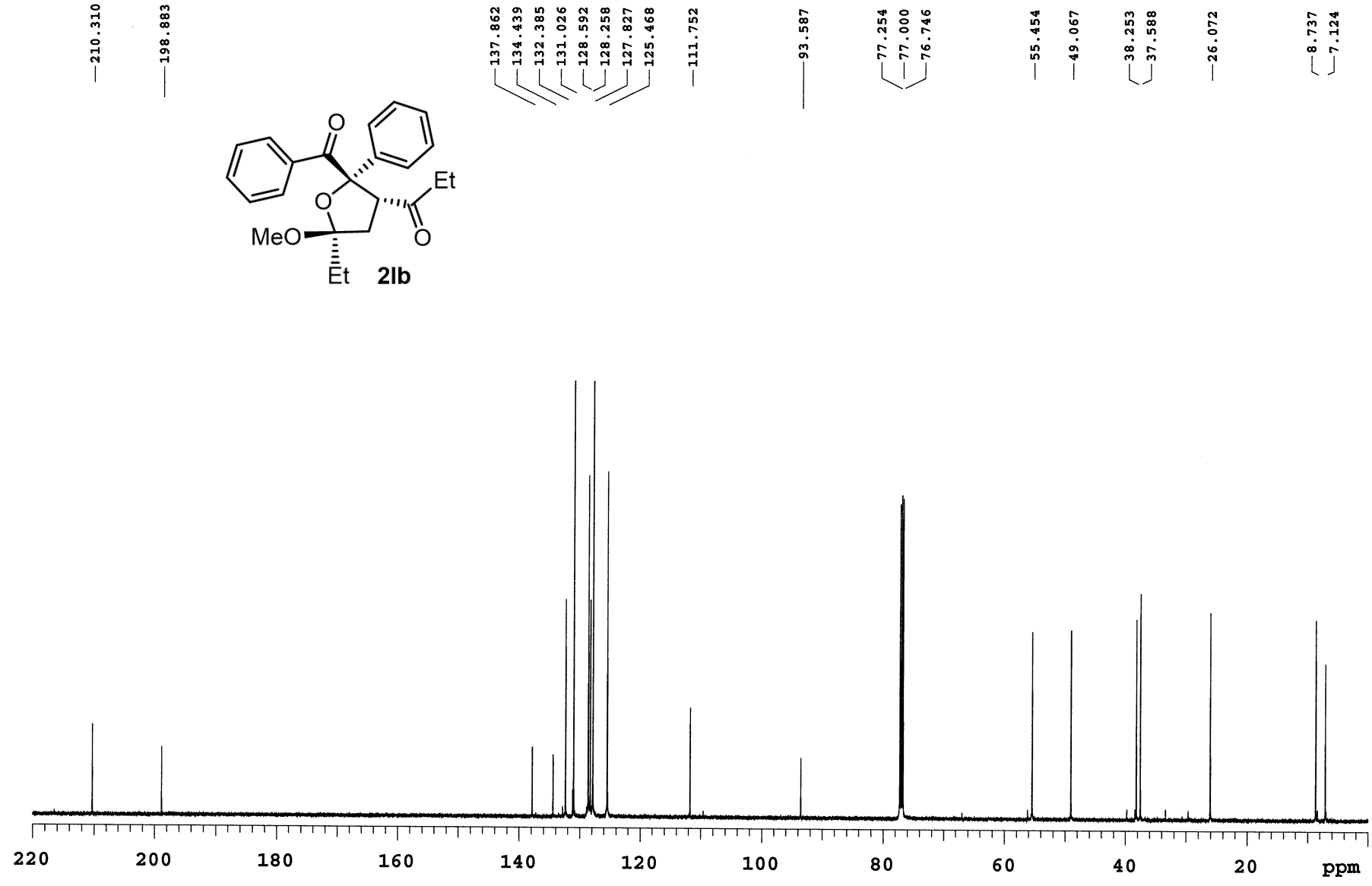
1H NMR (500 MHz, CDCl3) of compound 2lb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-074/PROTON_03

Plot date 2024-06-17

IRR-03-074

Sample Name	**IRR-03-074**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-15**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



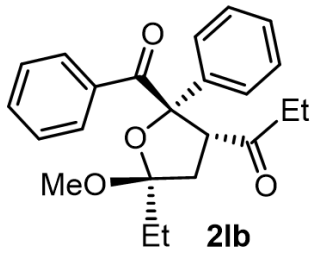
13C NMR (125 MHz, CDCl3) of compound 2Ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-074/CARBON_02

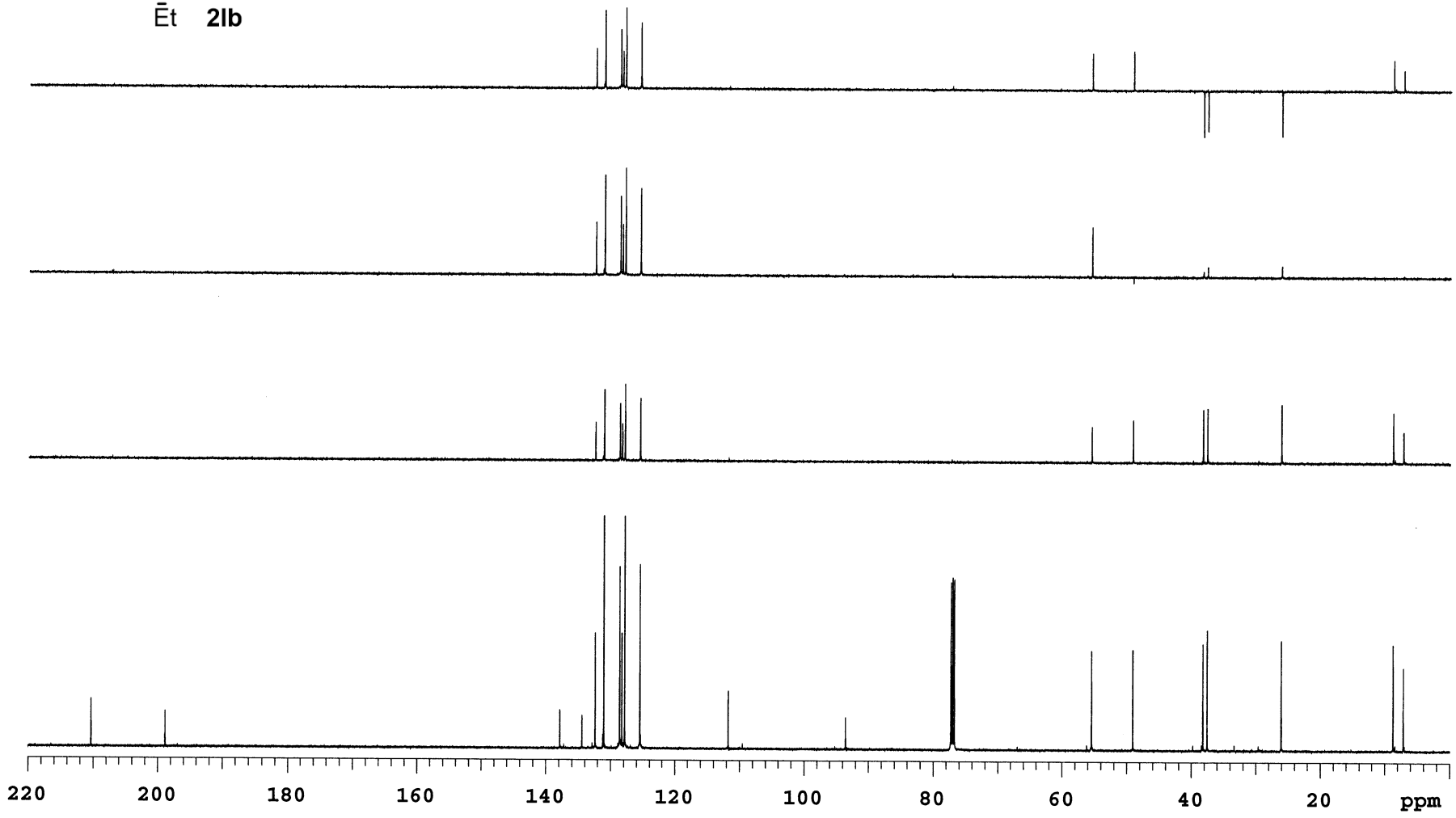
Plot date 2024-06-17

IRR-03-074

Sample Name	**IRR-03-074**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-16**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



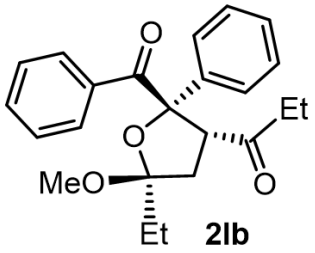
2lb



DEPT of compound 2lb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-074/DEPT_01

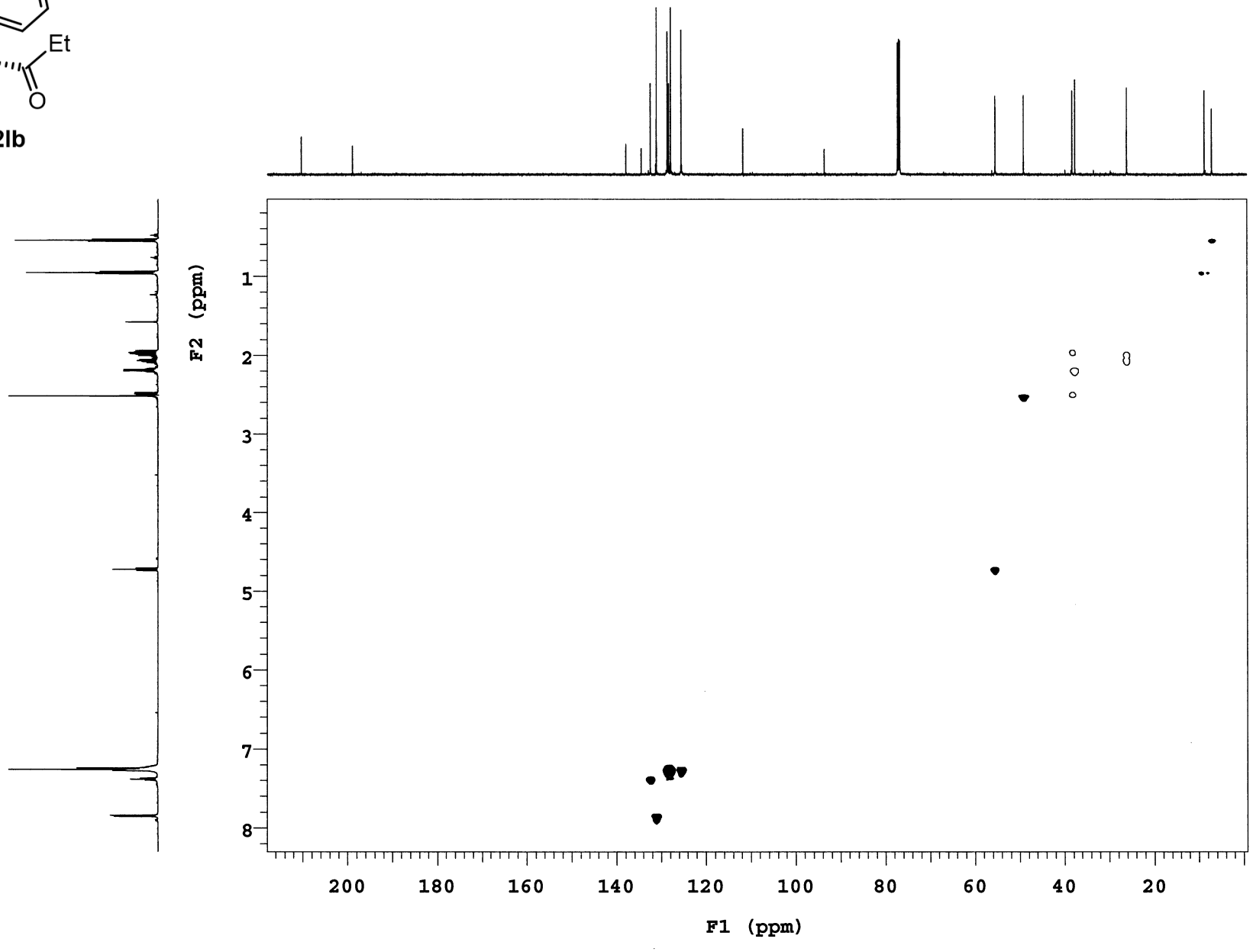
Plot date 2024-06-17



2Ib

IRR-03-074

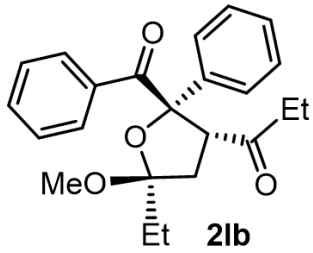
Sample Name	**IRR-03-074**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-16**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



HSQC of compound 2Ib

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-074/gHSQC_01

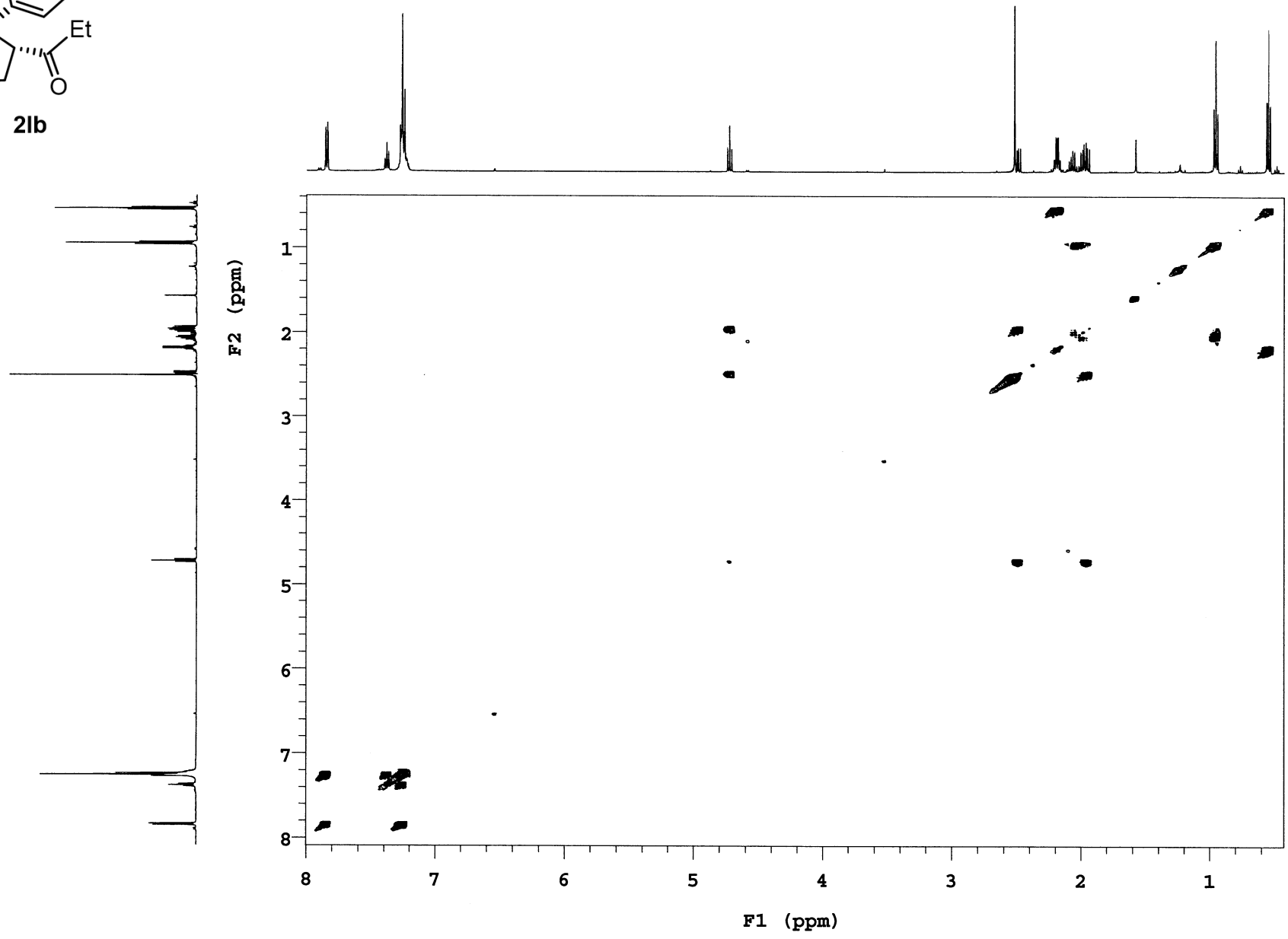
Plot date 2024-06-17



2lb

IRR-03-074

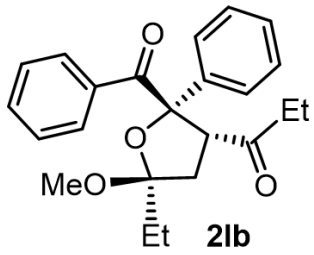
Sample Name	**IRR-03-074**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-16**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



COSY of compound 2lb

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-074/gCOSY_01

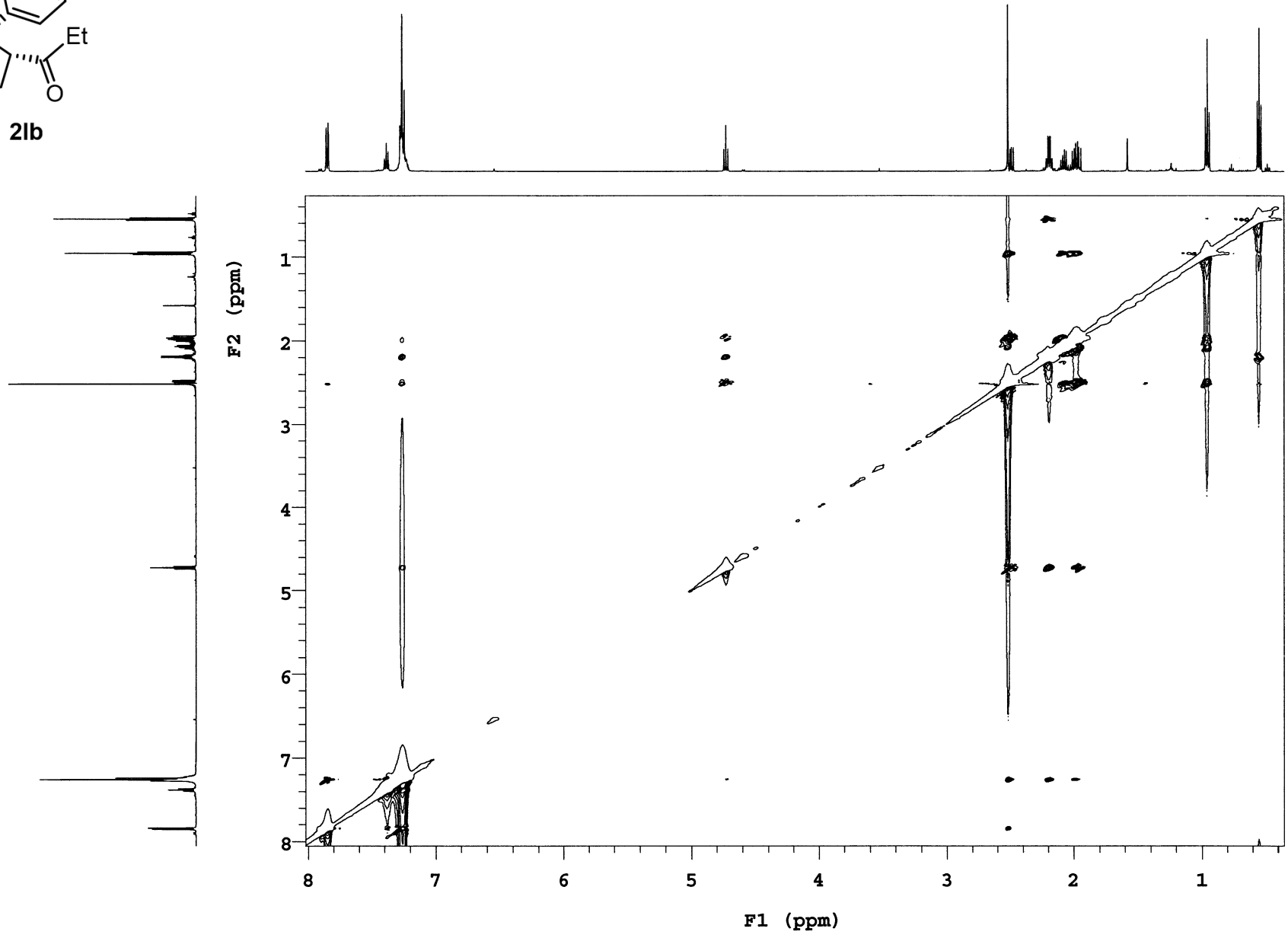
Plot date 2024-06-17



2lb

IRR-03-074

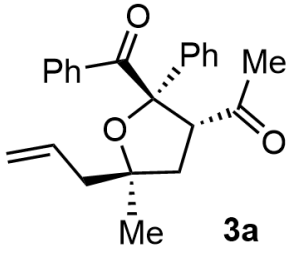
Sample Name	**IRR-03-074**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-06-16**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



NOESY of compound 2lb

IRR-03-116

Sample Name **IRR-03-116**	Pulse sequence **PROTON**	Temperature **25**	Study owner **vnmr2**
Date collected **2024-09-07**	Solvent **cdcl3**	Spectrometer **Agilent-NMR-inova500**	Operator **vnmr2**

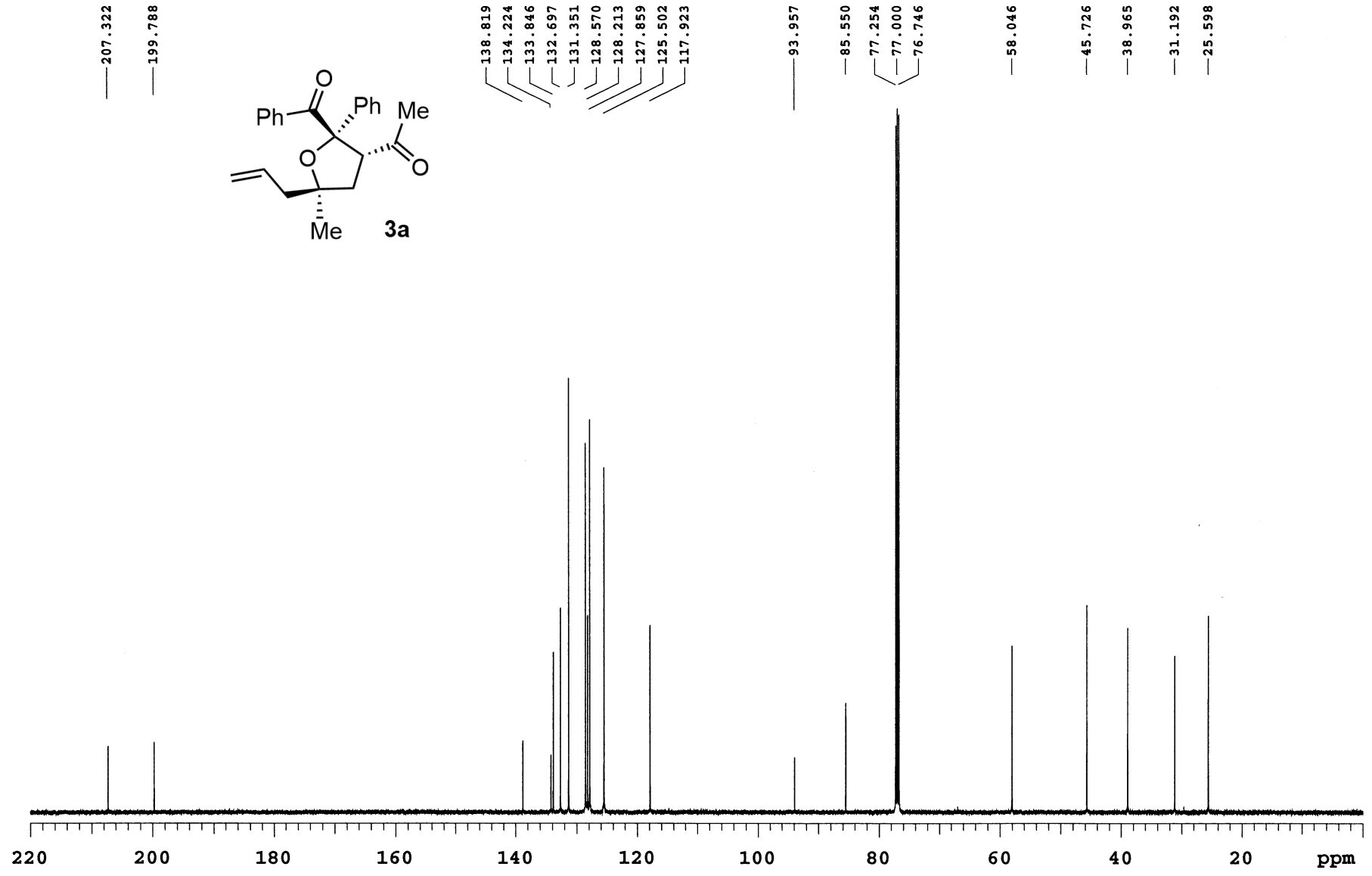


3a

1H NMR (500 MHz, CDCl3) of compound 3a

IRR-03-116

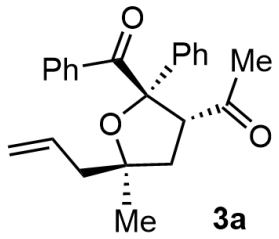
Sample Name	**IRR-03-116**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-07**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



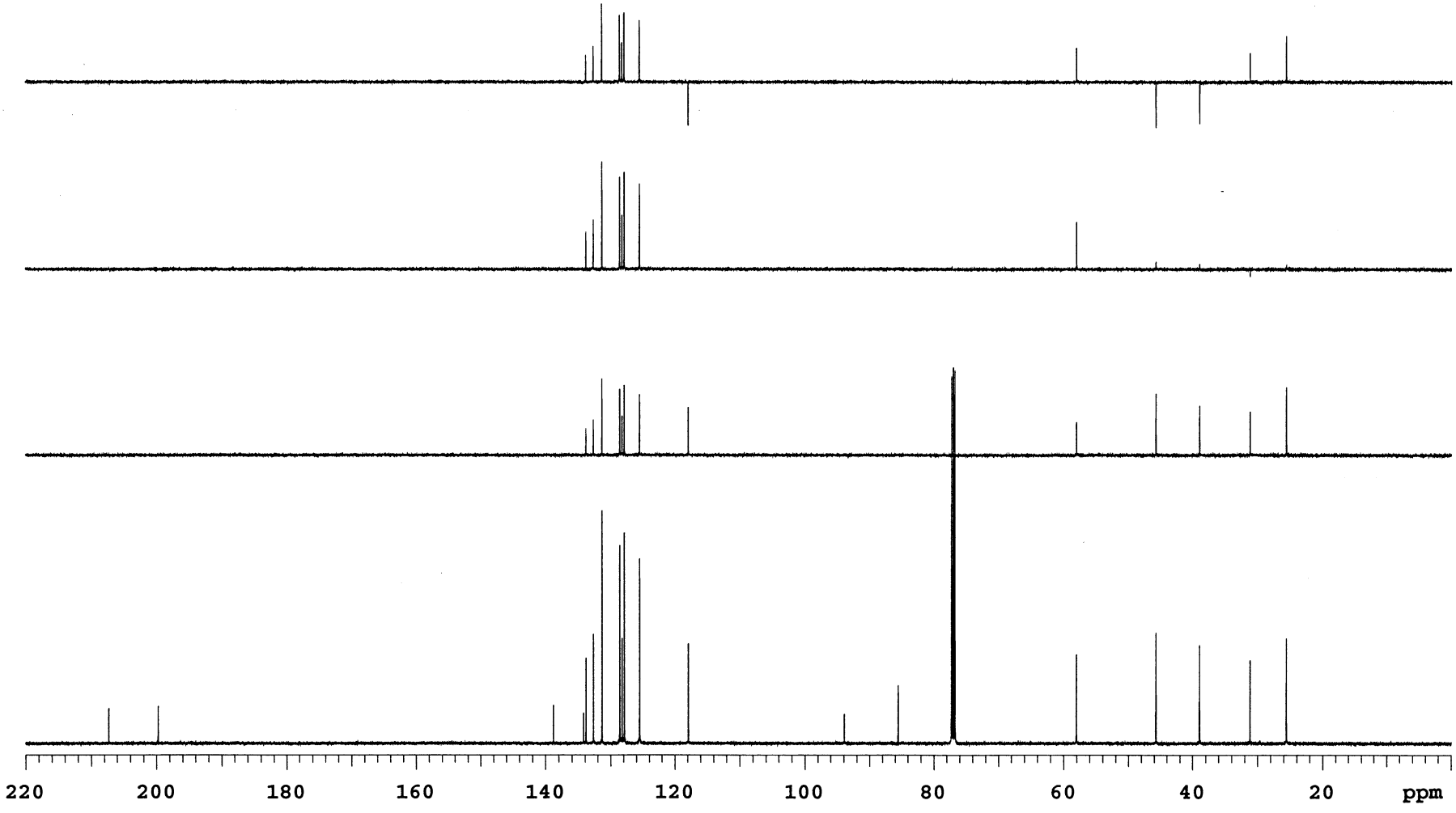
13C NMR (125 MHz, CDCl3) of compound 3a

IRR-03-116

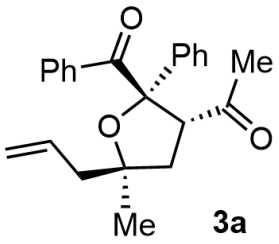
Sample Name	**IRR-03-116**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



3a



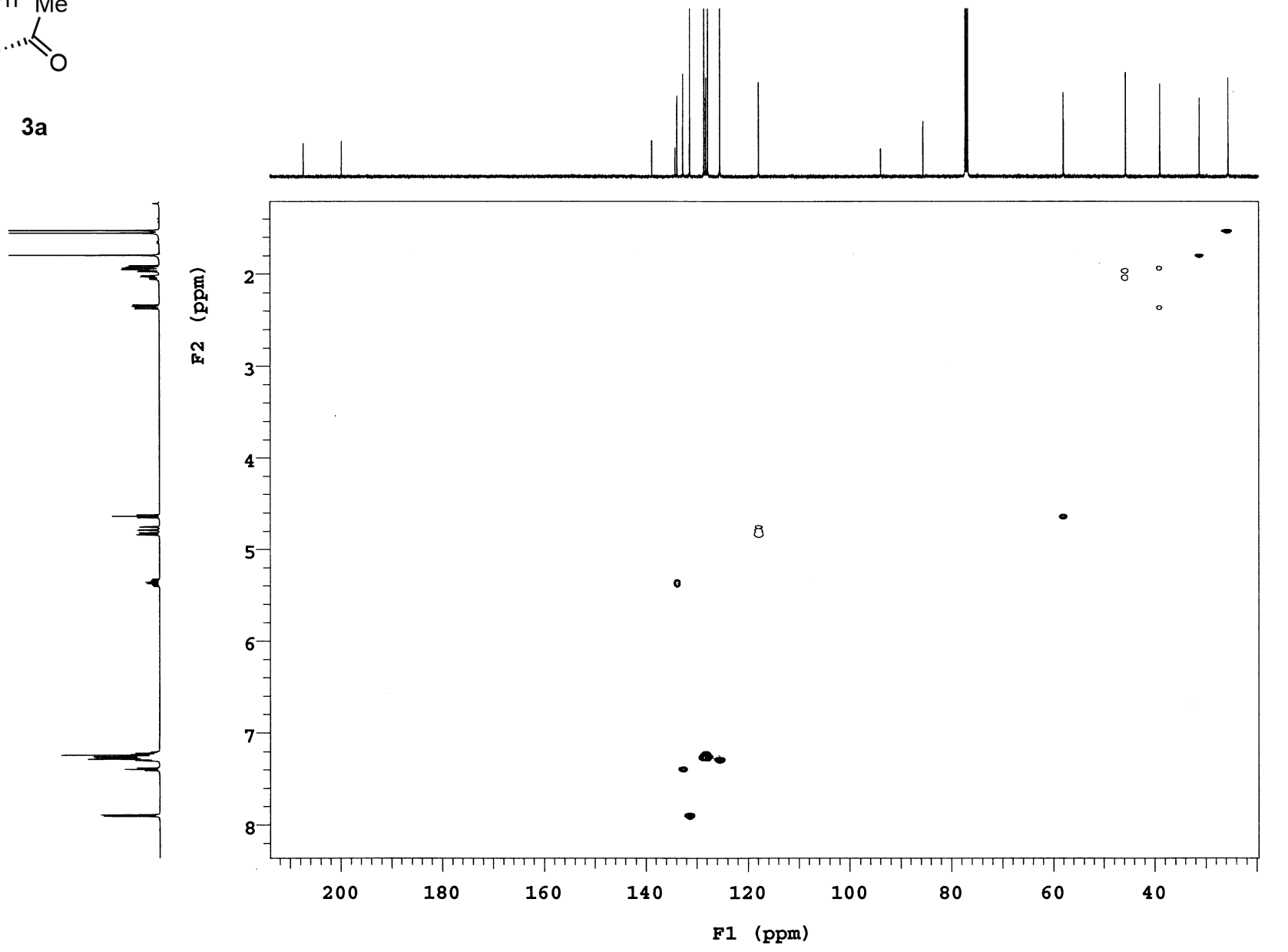
DEPT of compound 3a



3a

IRR-03-116

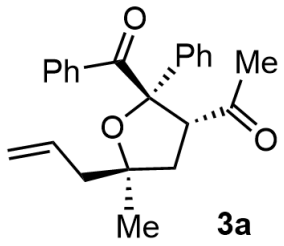
Sample Name	**IRR-03-116**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



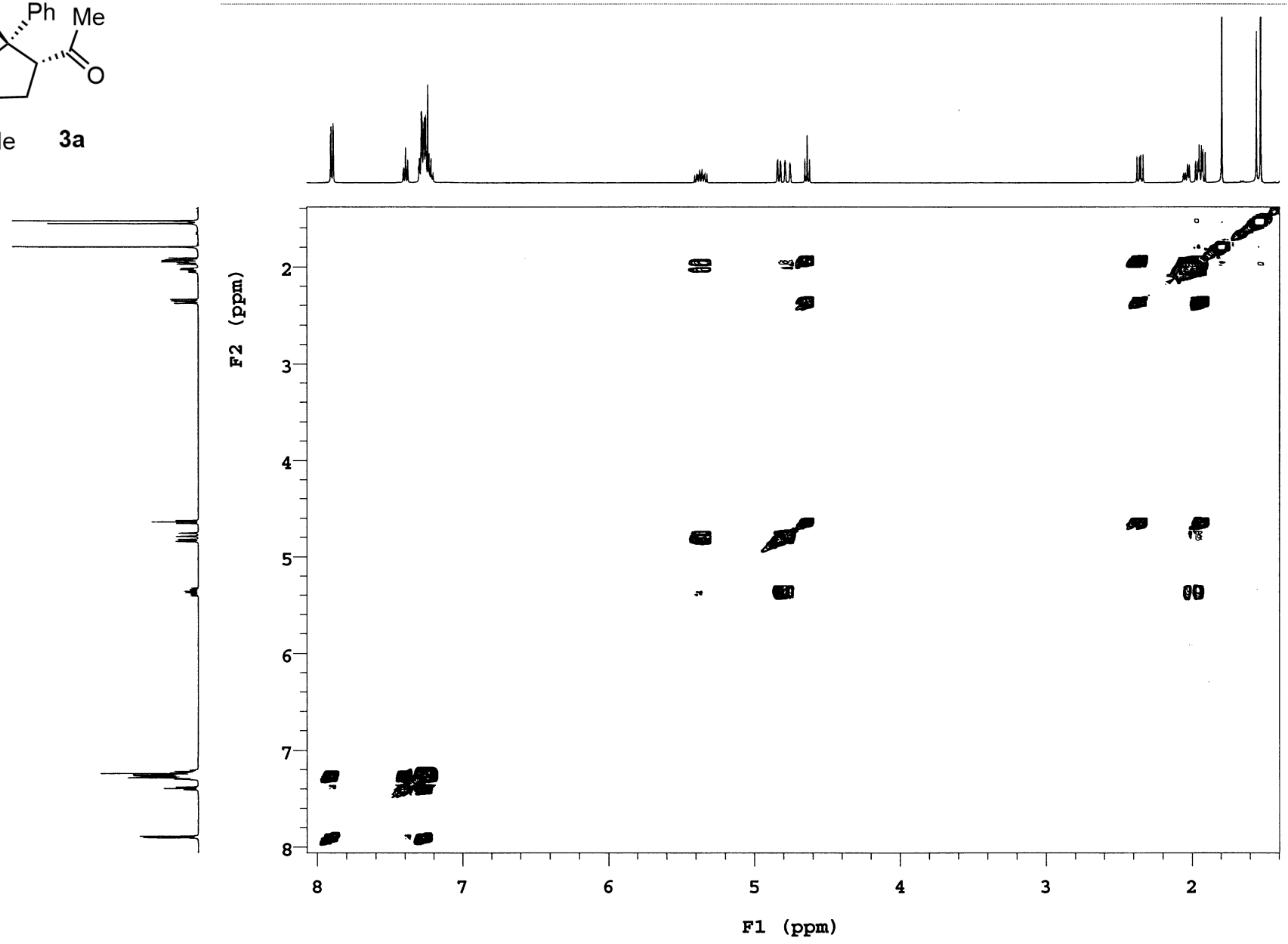
HSQC of compound 3a

IRR-03-116

Sample Name	**IRR-03-116**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



3a



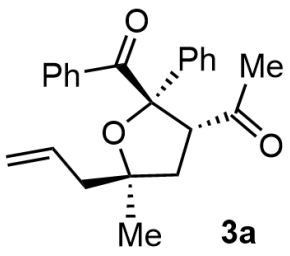
COSY of compound 3a

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-116/gCOSY_01

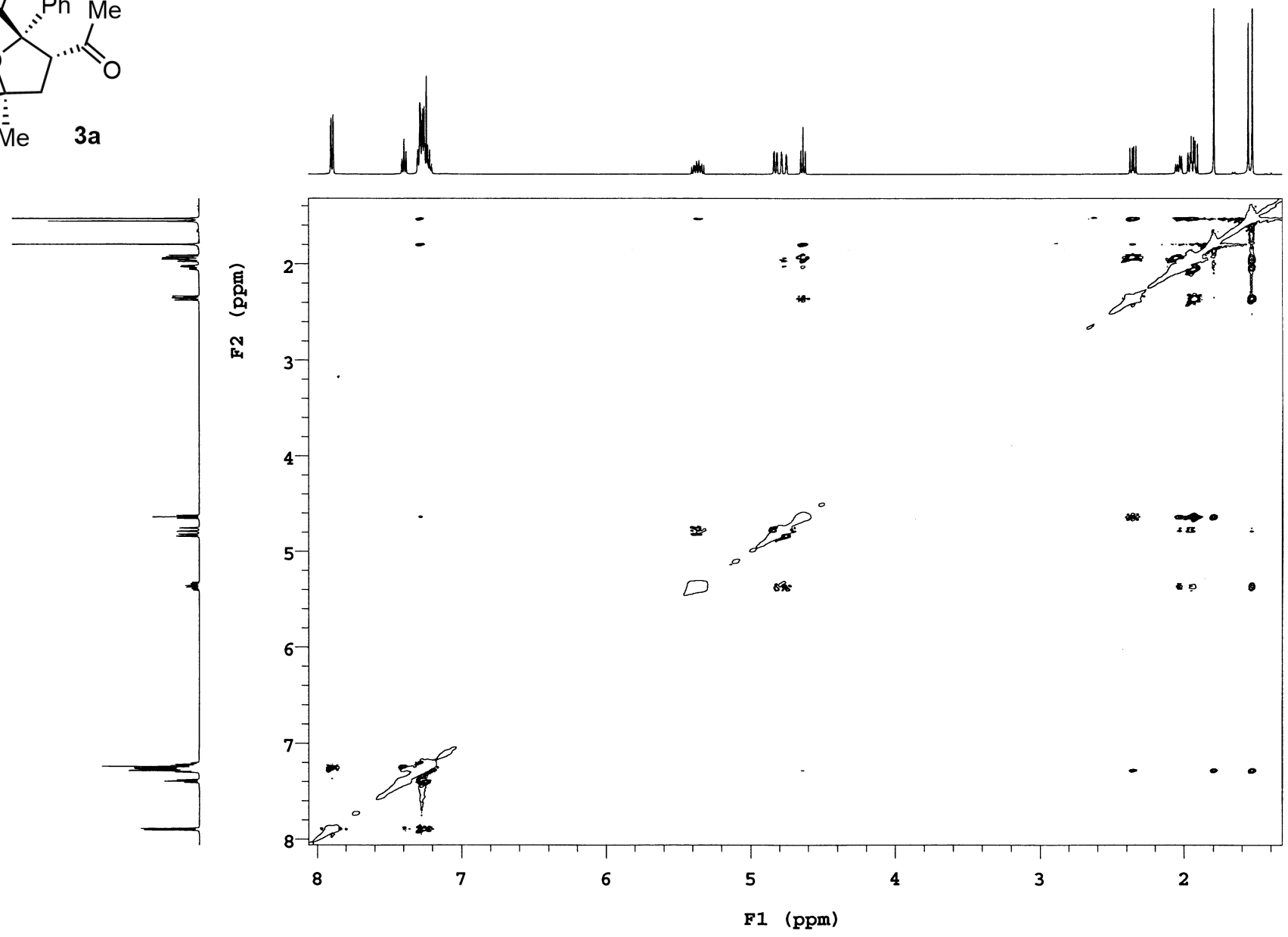
Plot date 2024-09-09

IRR-03-116

Sample Name	**IRR-03-116**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-08**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



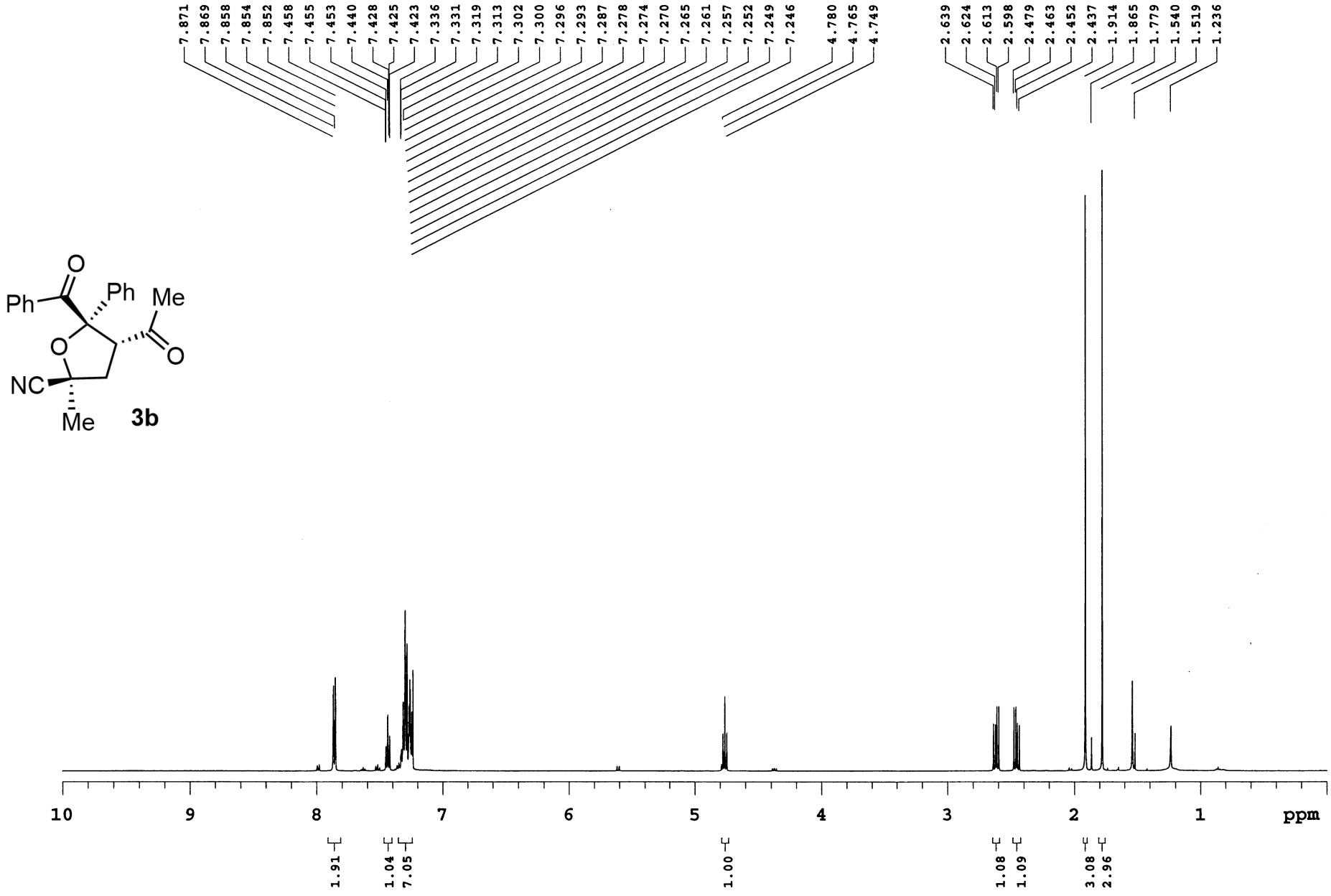
3a



NOESY of compound 3a

IRR-03-109

Sample Name	**IRR-03-109**	Pulse sequence	**PROTON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-10**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-109/PROTON_05

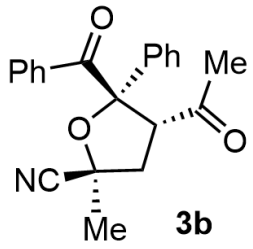
1H NMR (500 MHz, CDCl3) of compound 3b

Plot date 2024-09-11

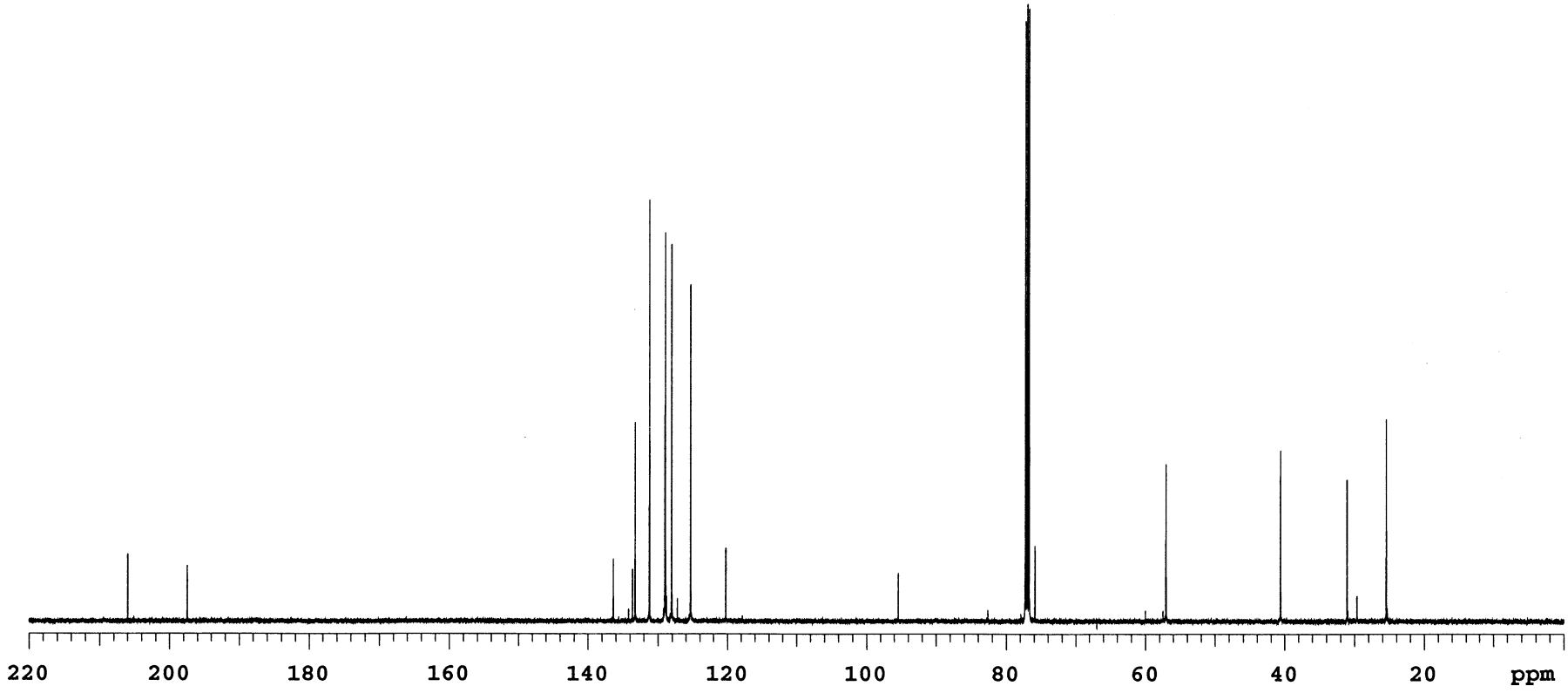
IRR-03-109

Sample Name	**IRR-03-109**	Pulse sequence	**CARBON**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-10**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**

206.014
197.489
136.385
133.636
133.262
131.202
129.005
128.953
128.054
125.324
120.264
95.467
77.254
77.000
76.746
75.899
57.082
40.631
31.115
25.472



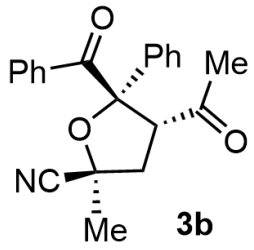
3b



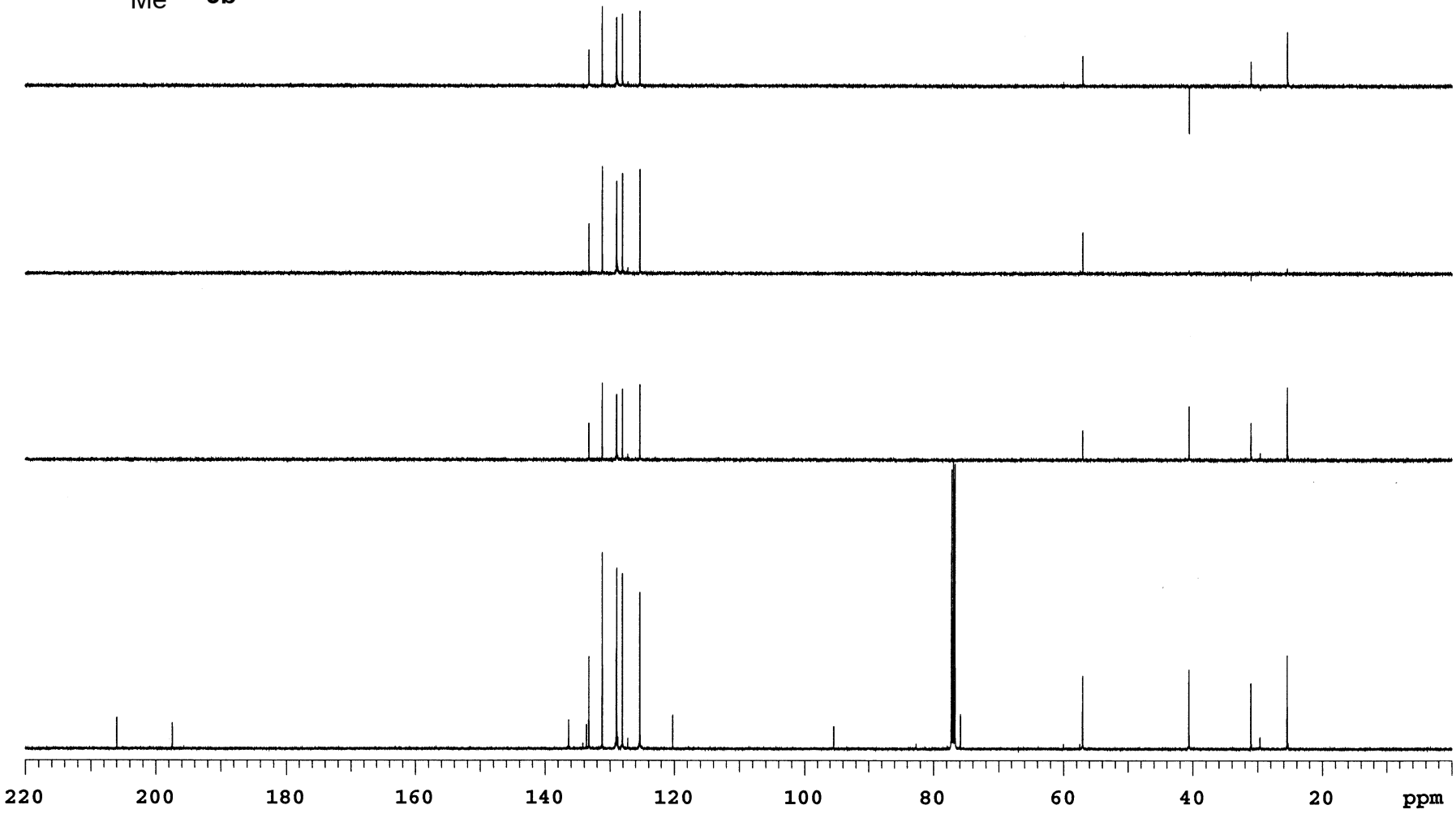
13C NMR (125 MHz, CDCl3) of compound 3b

IRR-03-109

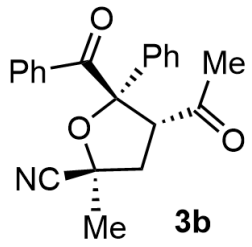
Sample Name	**IRR-03-109**	Pulse sequence	**DEPT**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-11**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



3b



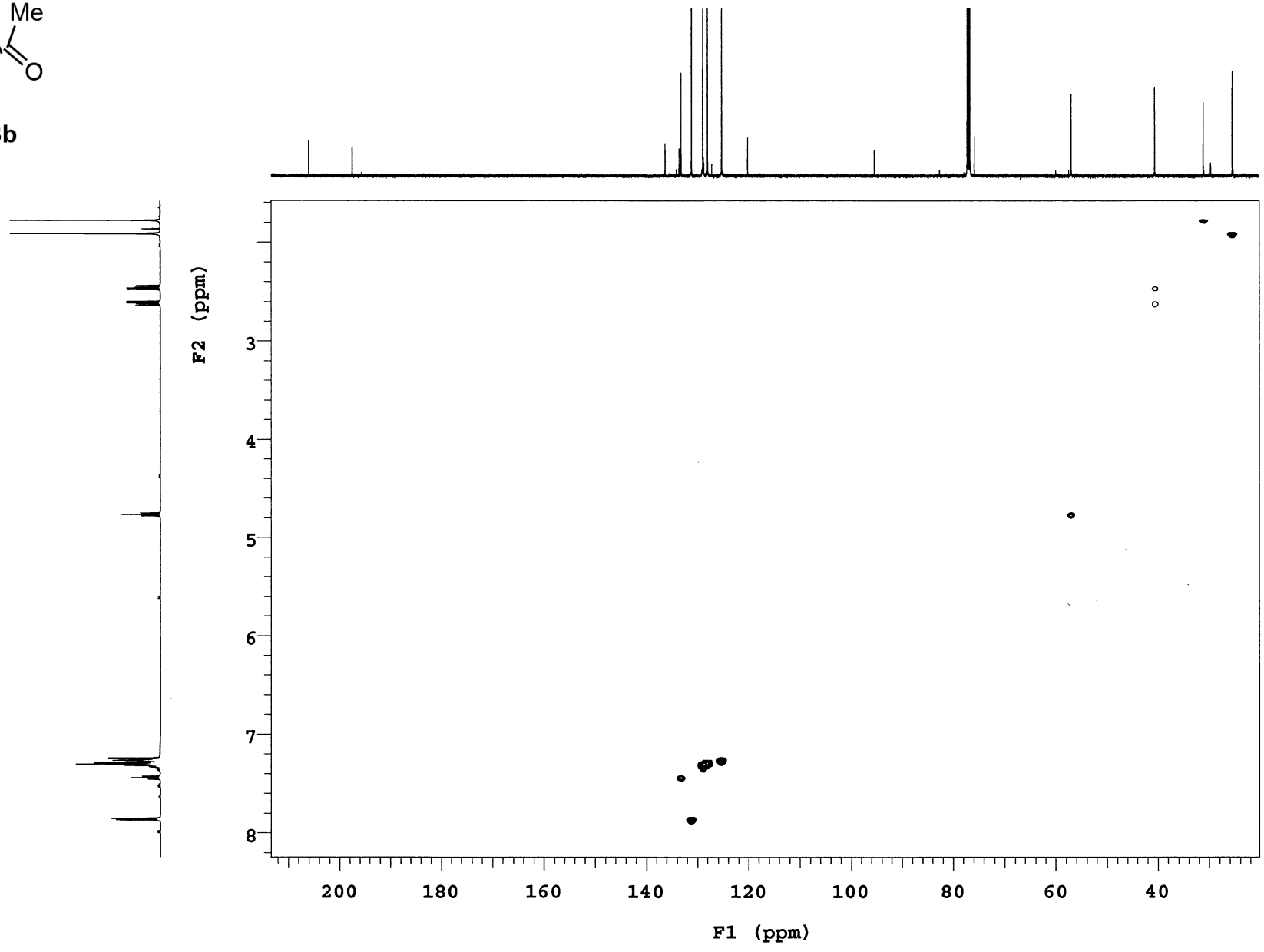
DEPT of compound 3b



3b

IRR-03-109

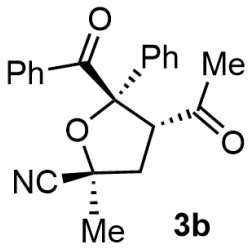
Sample Name	**IRR-03-109**	Pulse sequence	**gHSQC**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-11**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



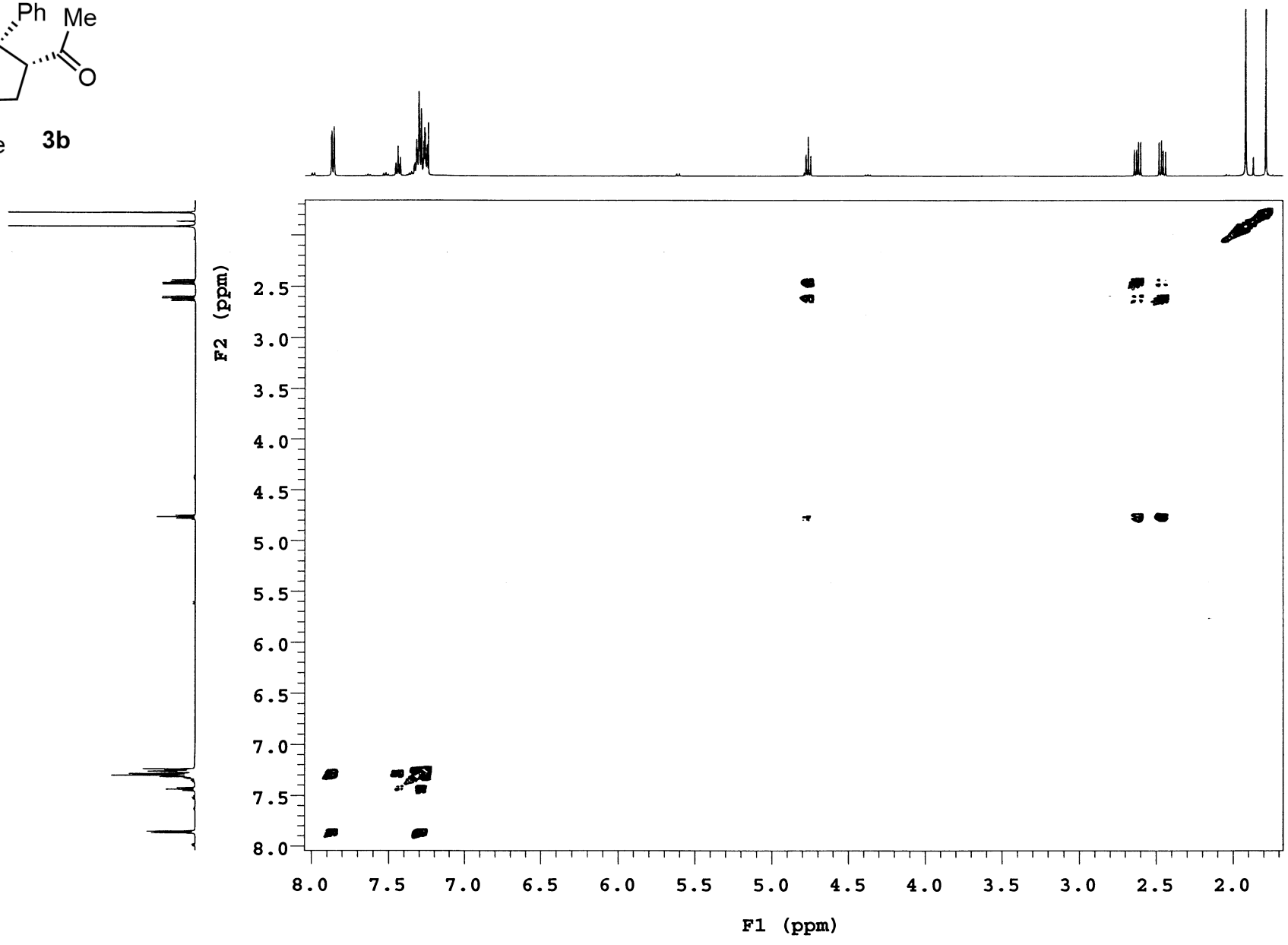
HSQC of compound 3b

IRR-03-109

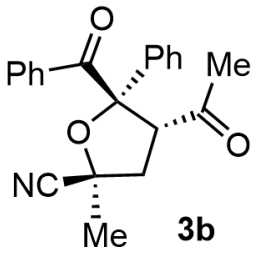
Sample Name	**IRR-03-109**	Pulse sequence	**gCOSY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-11**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



3b



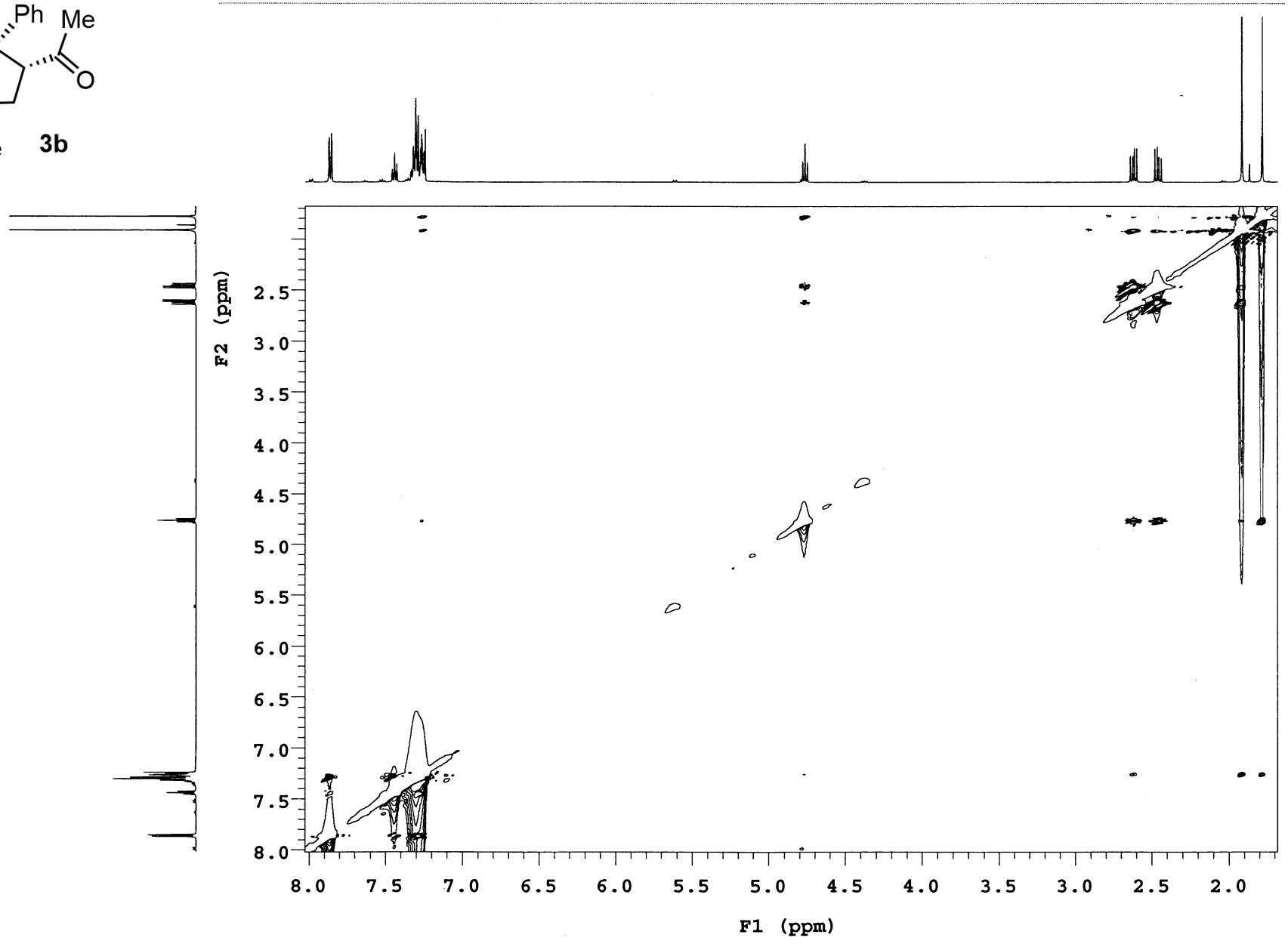
COSY of compound 3b



3b

IRR-03-109

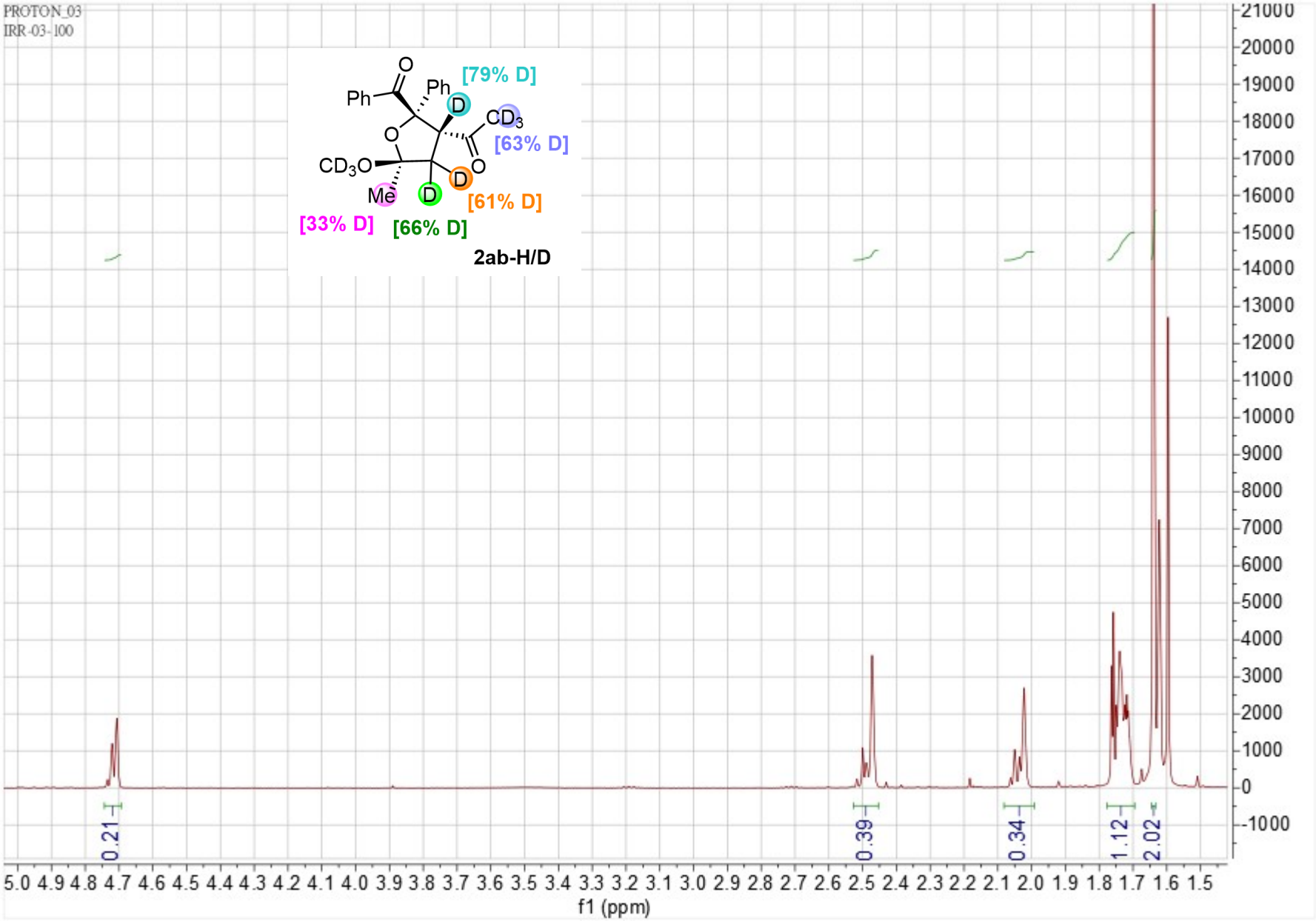
Sample Name	**IRR-03-109**	Pulse sequence	**NOESY**	Temperature	**25**	Study owner	**vnmr2**
Date collected	**2024-09-11**	Solvent	**cdcl3**	Spectrometer	**Agilent-NMR-inova500**	Operator	**vnmr2**



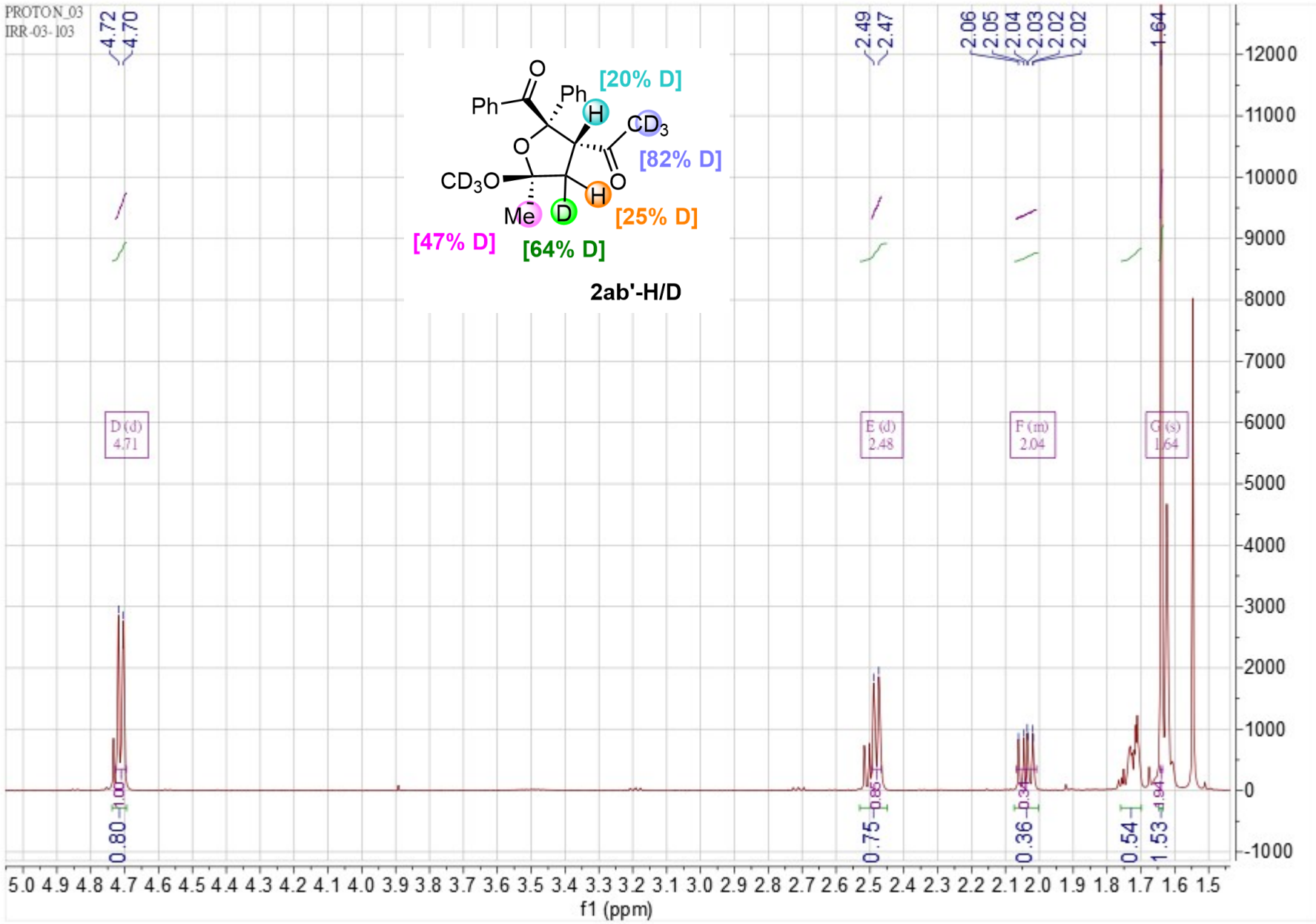
NOESY of compound 3b

Data file /home/vnmr2/vnmrsys/data/511/IRR/IRR-03-109/NOESY_01

Plot date 2024-09-11



Expansion of 1H NMR of **2ab-H/D**



Expansion of 1H NMR of **2ab'-H/D**