

Supporting Information

One-pot Synthesis of Benzofused 8-Oxabicyclo[3.3.1]nonanes via GaCl₃-Mediated Cyclocondensation of *o*-Allylbenzaldehydes and 1,3-Dicarbonyl Synthons

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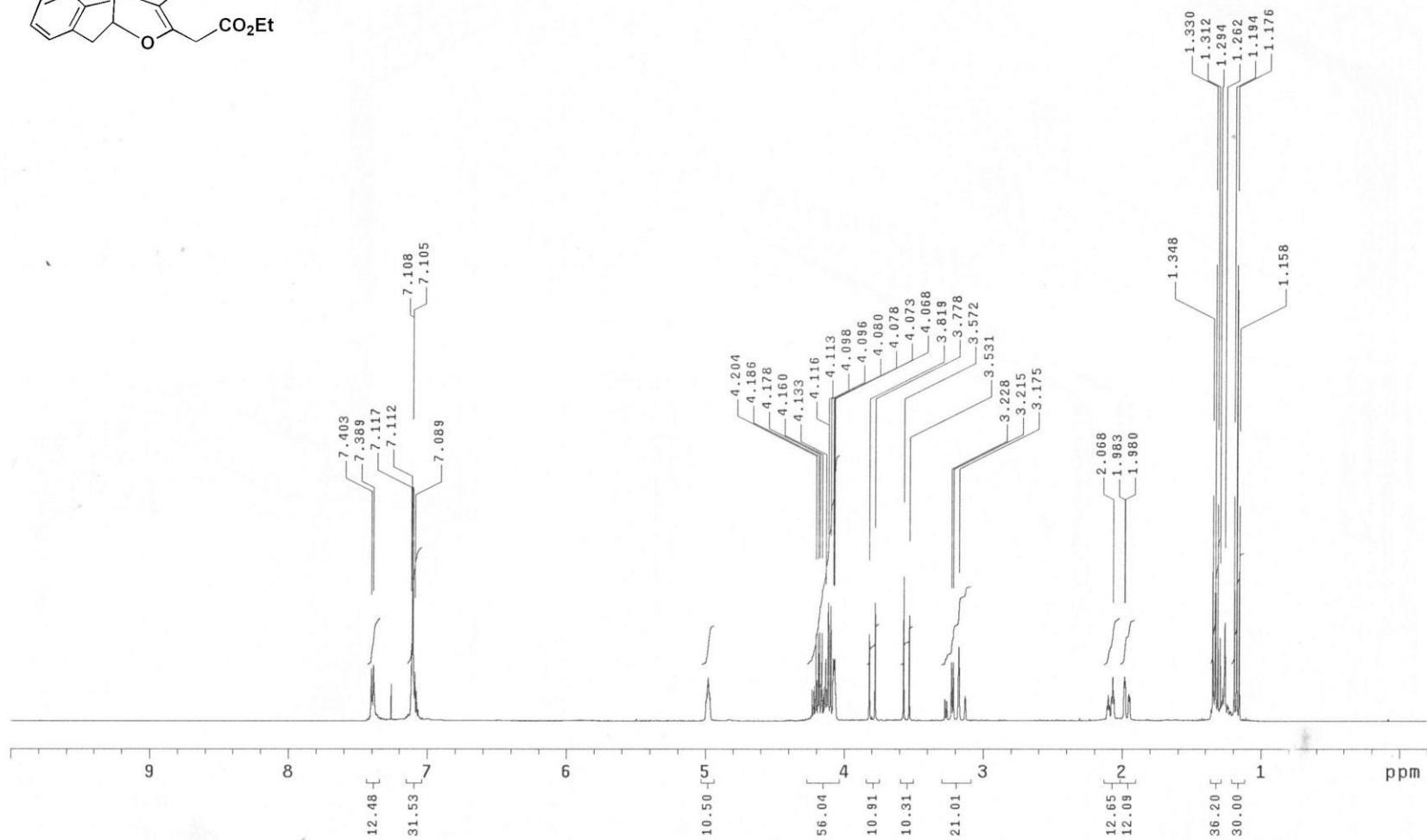
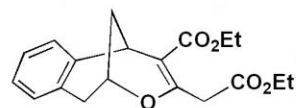
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Compound 6a (¹H-NMR spectral data)

YNB720

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jul 21 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6a (¹³C-NMR spectral data)

YNB720

Pulse Sequence: s2pu1

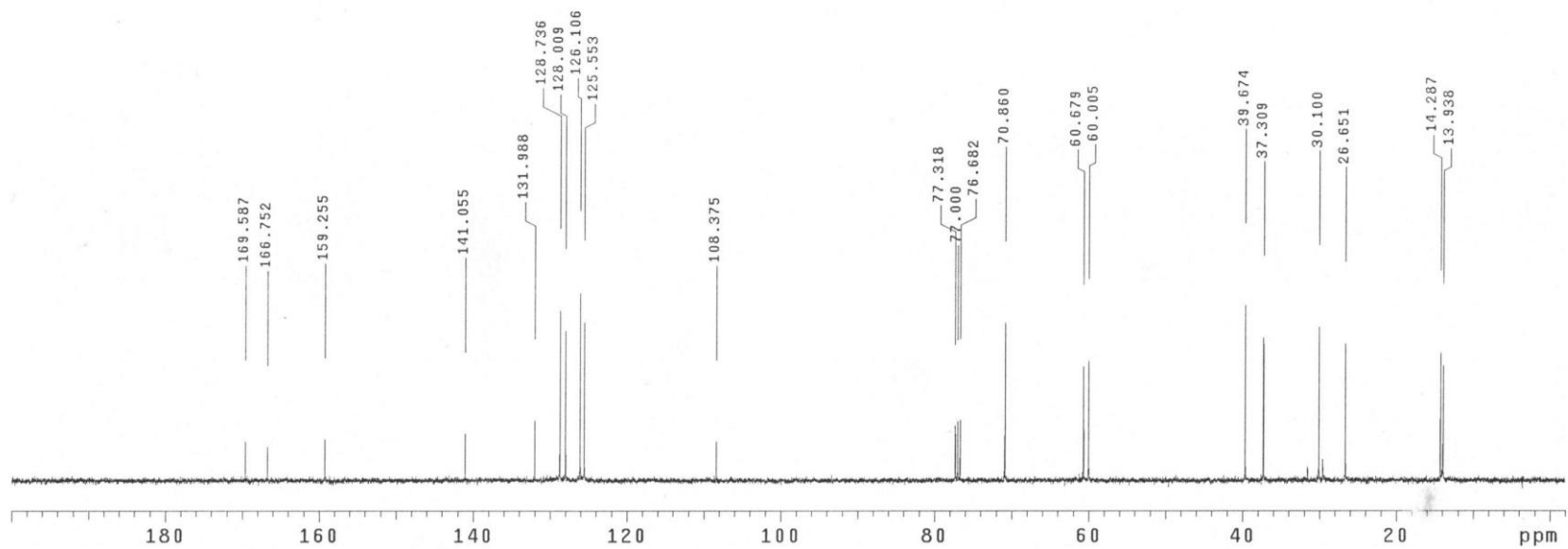
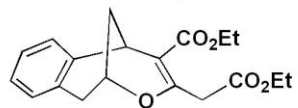
UNITYplus-400 "unity400"

Date: Jul 21 2022

Solvent: CDCl₃

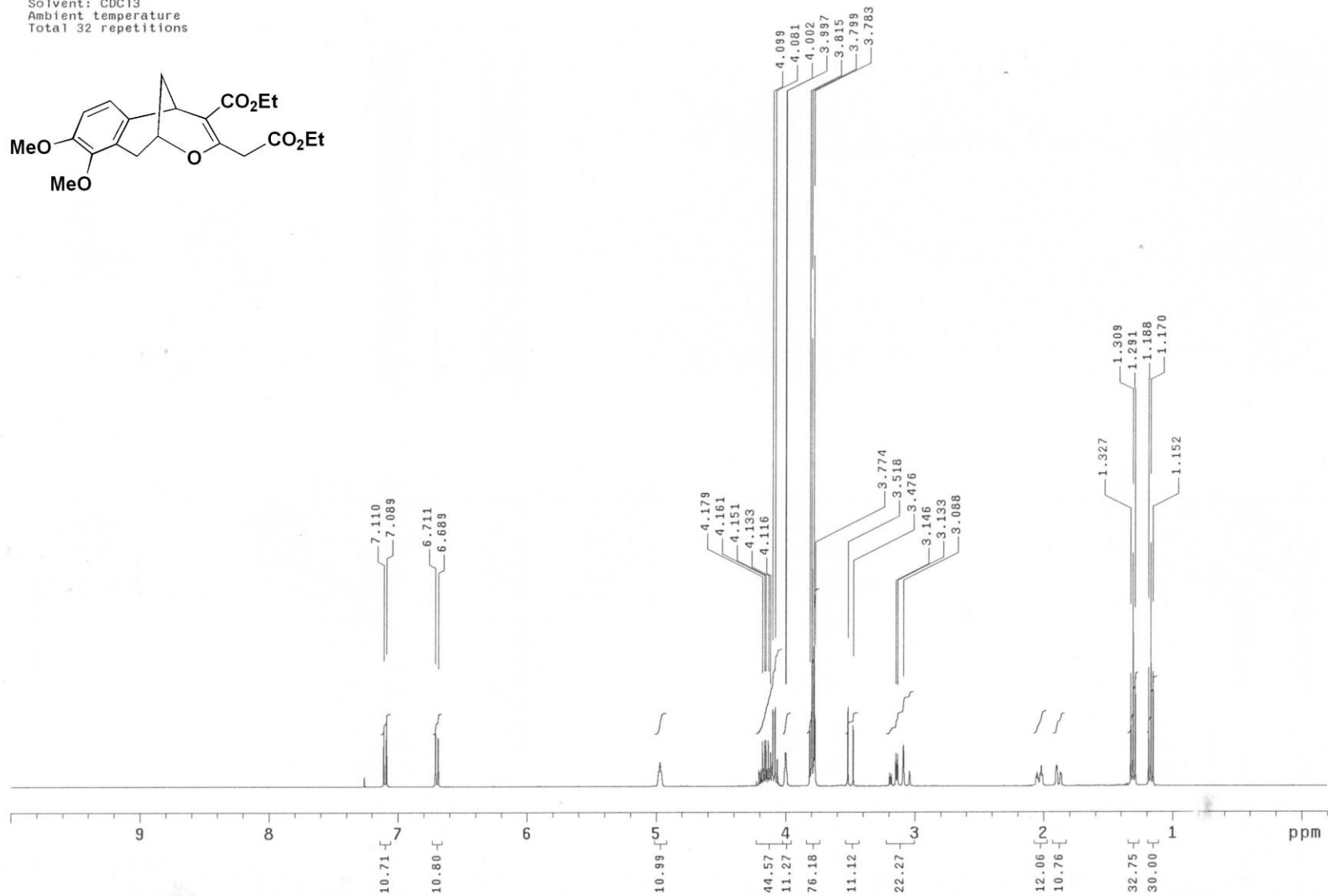
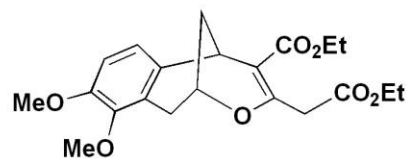
Ambient temperature

Total 320 repetitions



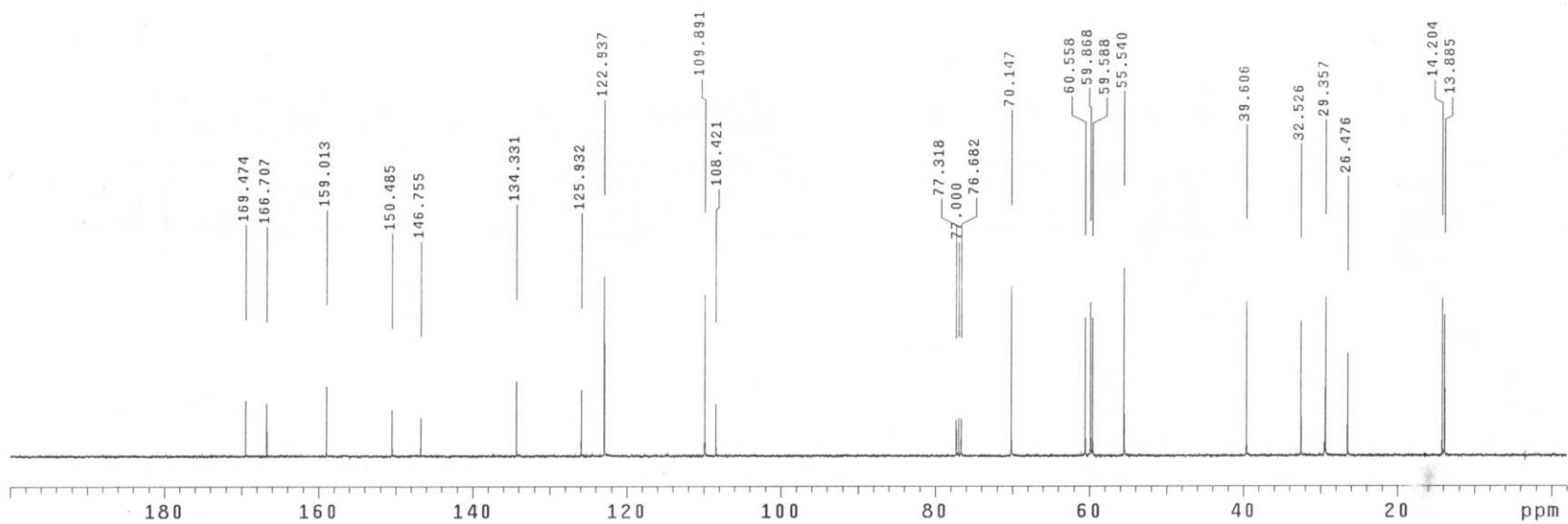
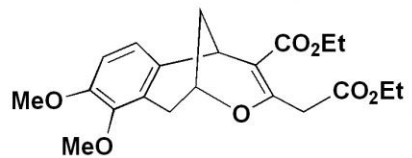
Compound 6b (¹H-NMR spectral data)

QYA607
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jun 7 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6b (¹³C-NMR spectral data)

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jun 7 2022
Solvent: CDCl₃
Ambient temperature
Total 560 repetitions



Compound 6c (¹H-NMR spectral data)

YNB705

Pulse Sequence: s2pu1

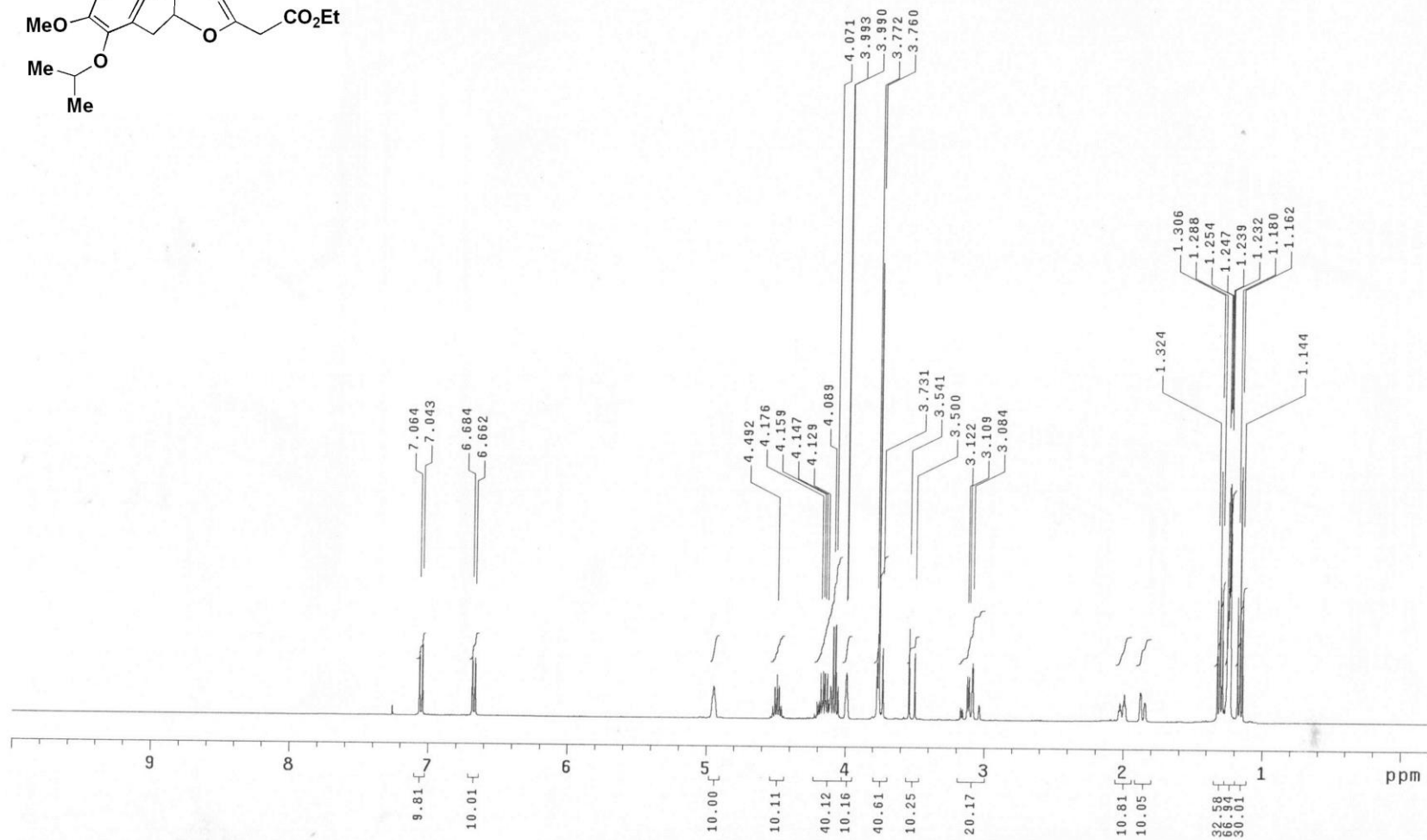
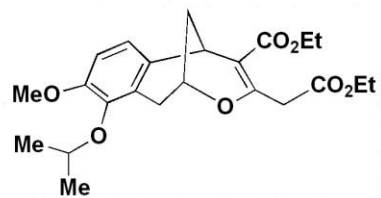
UNITYplus-400 "unity400"

Date: Jul 6 2022

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 6c (¹³C-NMR spectral data)

YNB705

Pulse Sequence: s2pu1

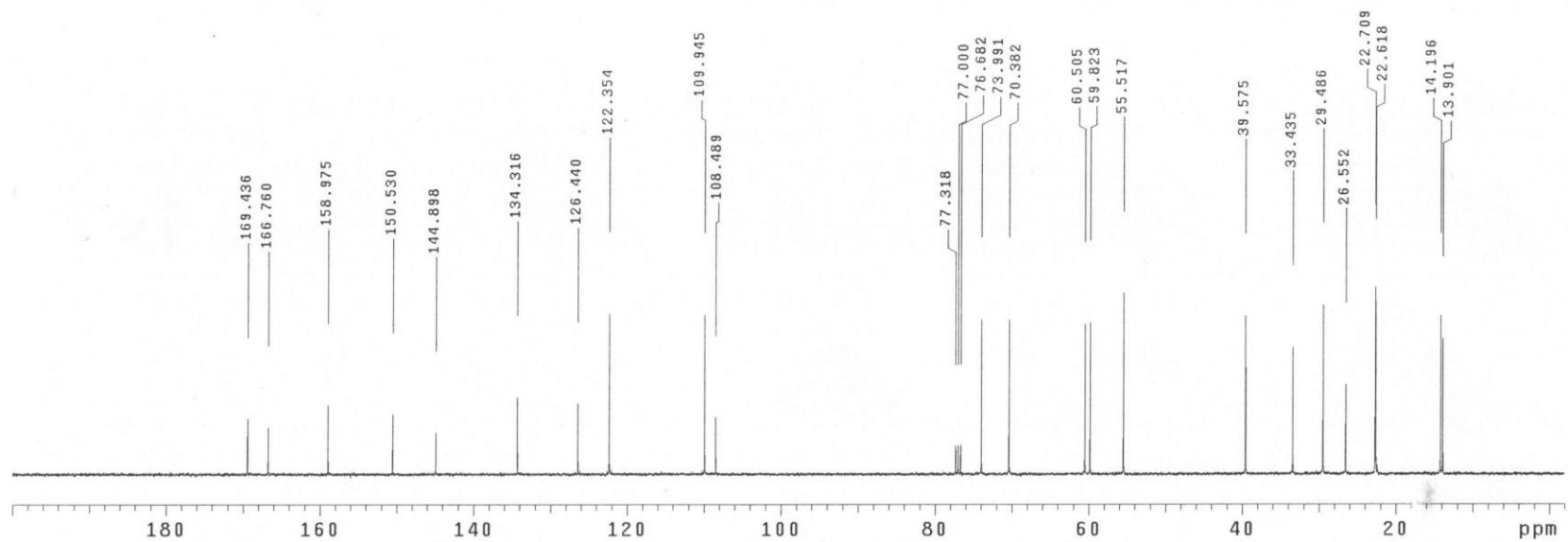
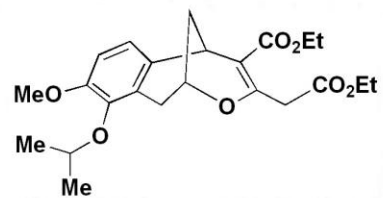
UNITYplus-400 "unity400"

Date: Jul 6 2022

Solvent: CDCl₃

Ambient temperature

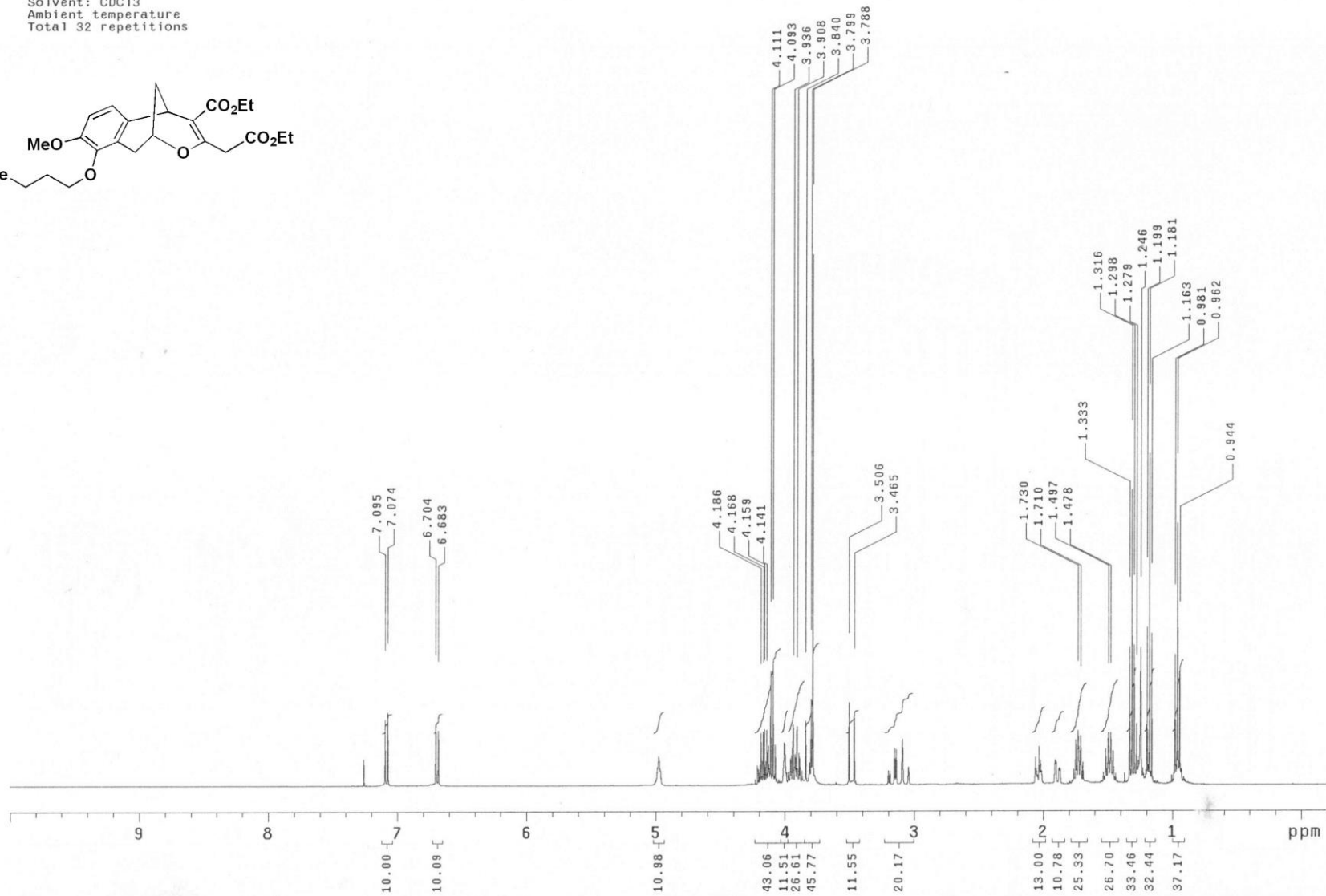
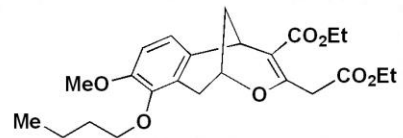
Total 112 repetitions



Compound 6d (¹H-NMR spectral data)

YNB708

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jul 8 2022
Solvent: CDC13
Ambient temperature
Total 32 repetitions



Compound 6d (¹³C-NMR spectral data)

YNB708

Pulse Sequence: s2pu1

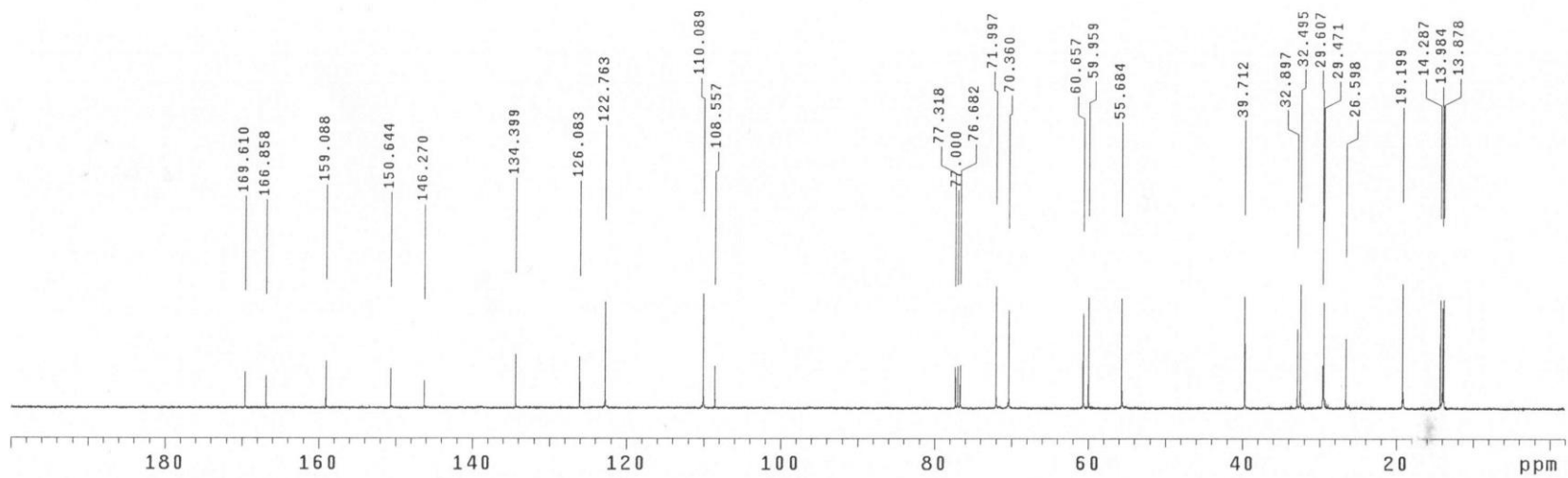
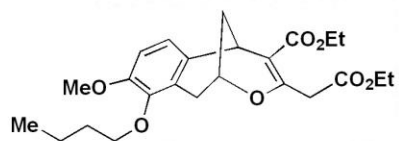
UNITYplus-400 "unity400"

Date: Jul 8 2022

Solvent: CDCl₃

Ambient temperature

Total 2752 repetitions



Compound 6e (¹H-NMR spectral data)

YNB716

Pulse Sequence: s2pu1

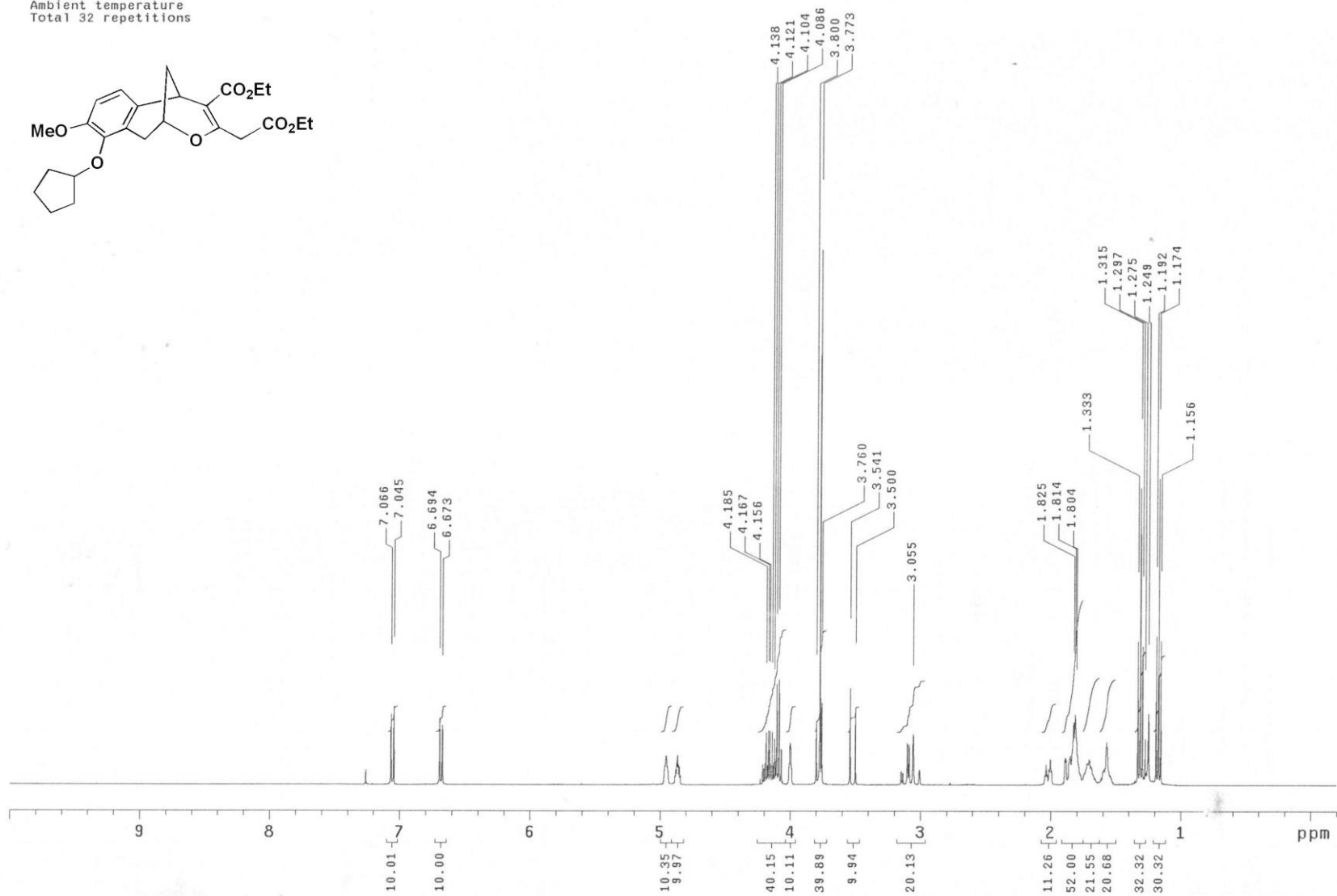
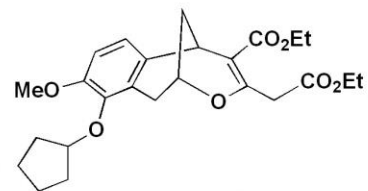
UNITYplus-400 "unity400"

Date: Jul 18 2022

Solvent: CDCl₃

Ambient temperature

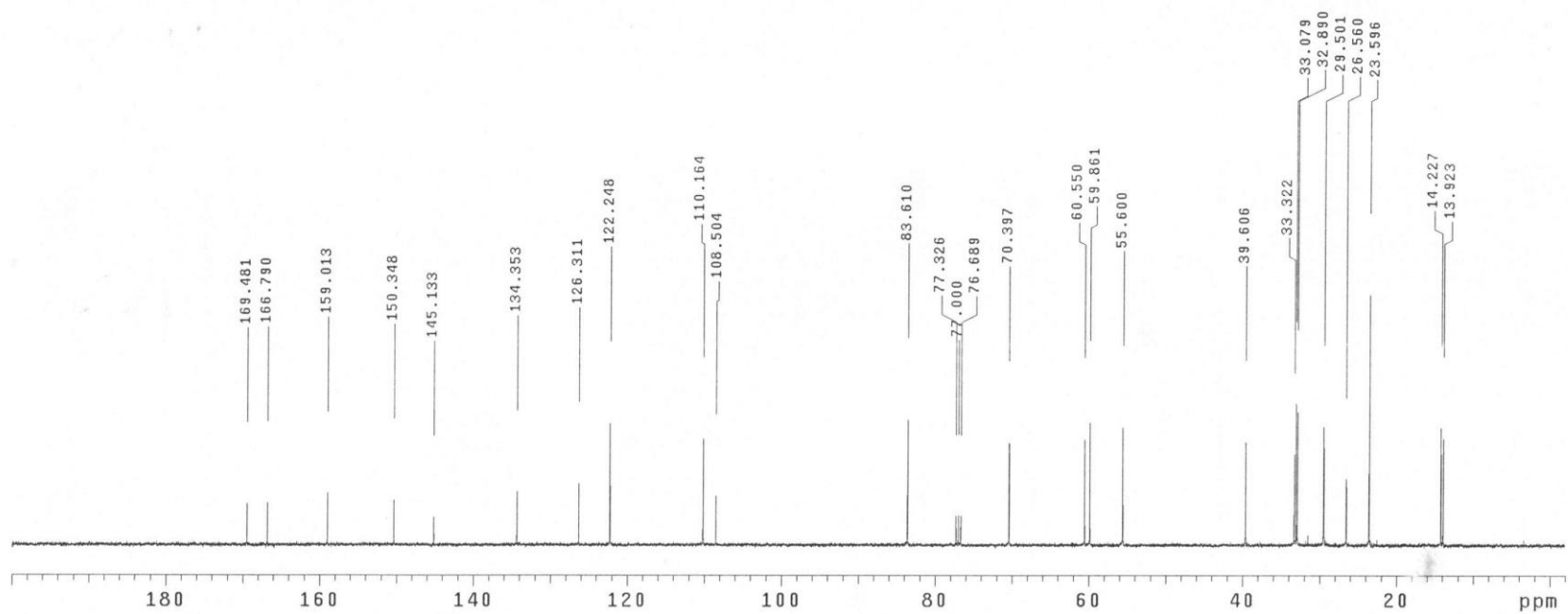
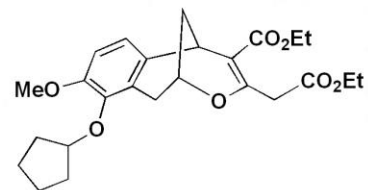
Total 32 repetitions



Compound 6e (¹³C-NMR spectral data)

YNB716

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jul 18 2022
Solvent: CDCl₃
Ambient temperature
Total 224 repetitions



Compound 6f (¹H-NMR spectral data)

YNBC713

Pulse Sequence: s2pu1

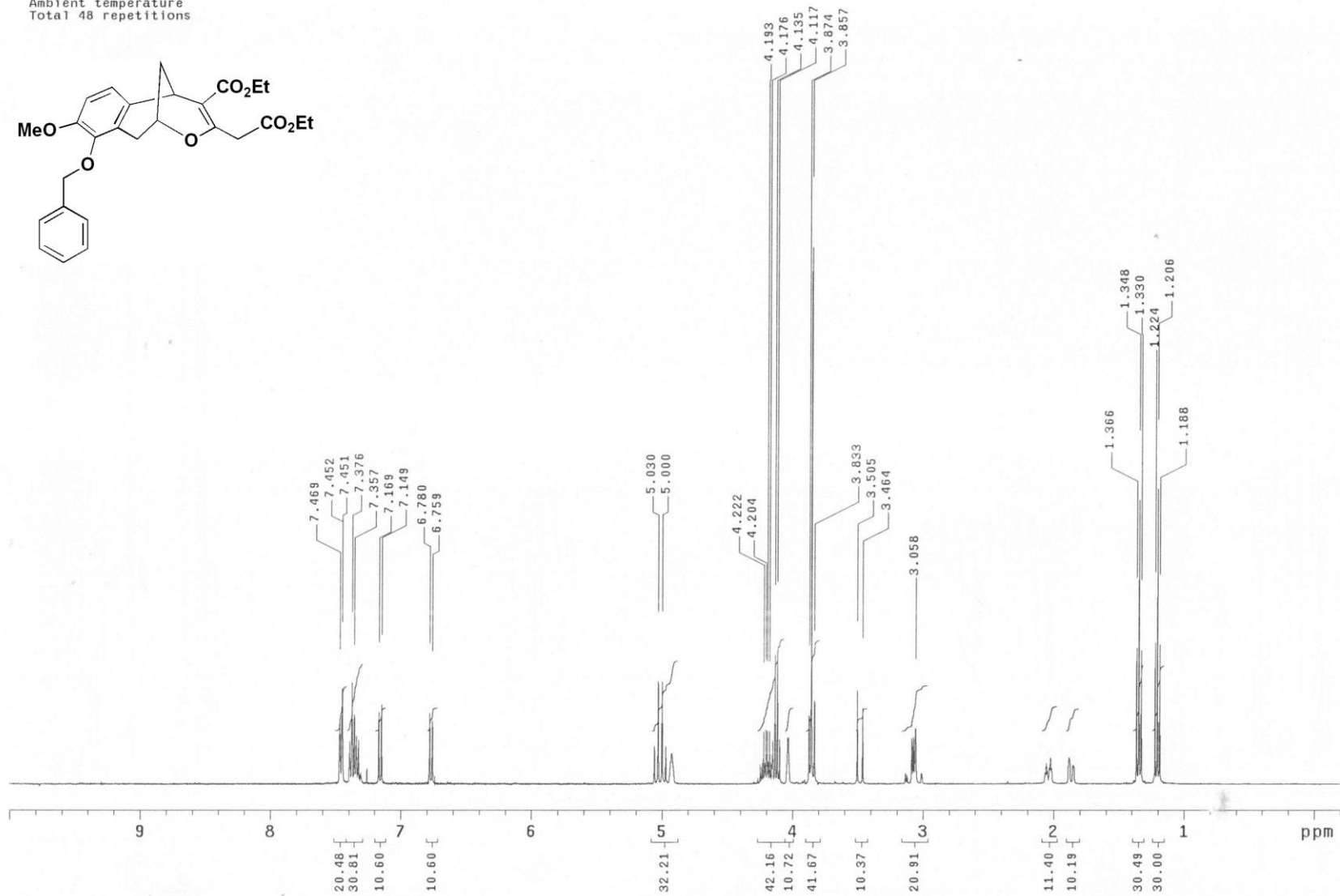
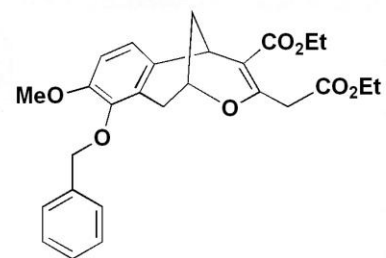
UNITYplus-400 "unity400"

Date: Jul 14 2022

Solvent: CDCl₃

Ambient temperature

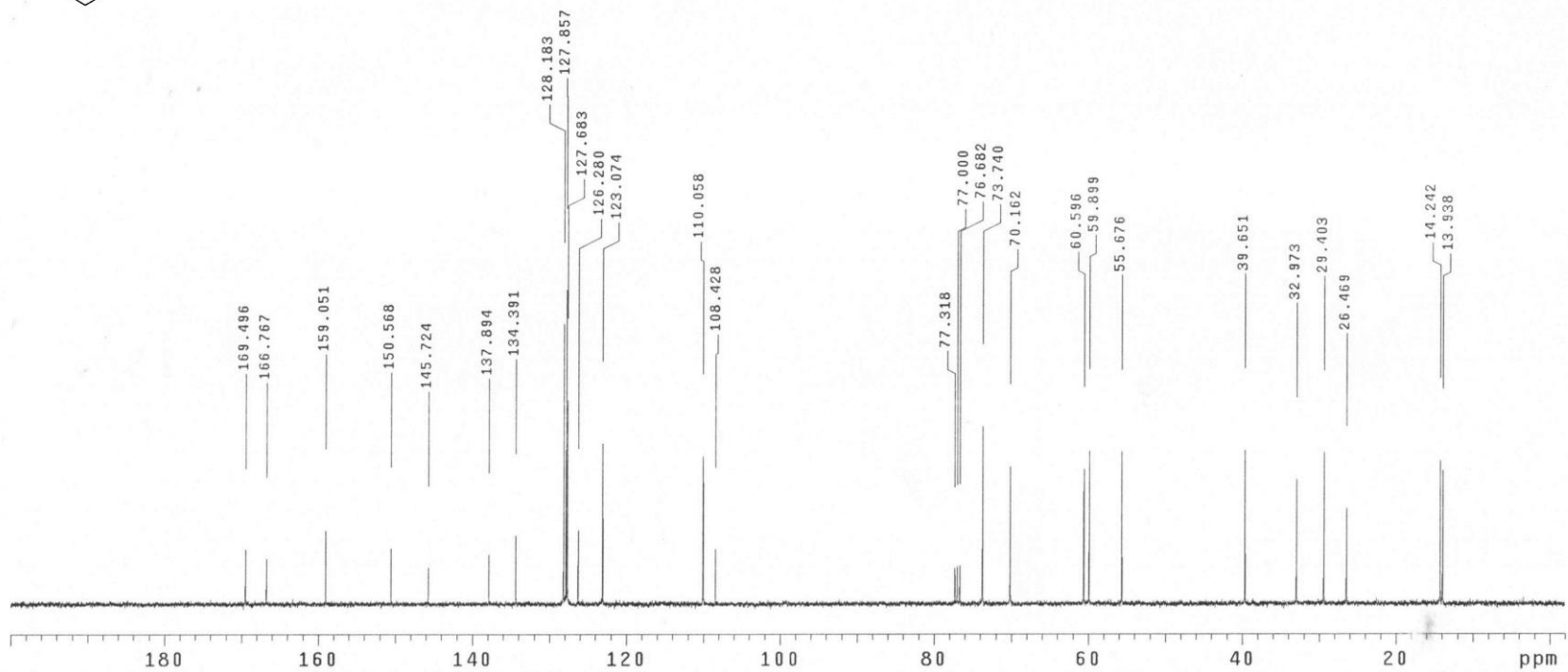
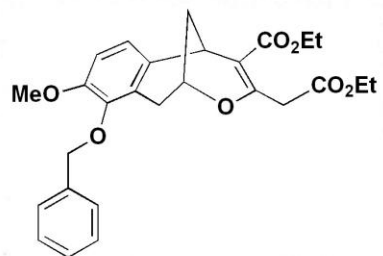
Total 48 repetitions



Compound 6f (¹³C-NMR spectral data)

YNBC713

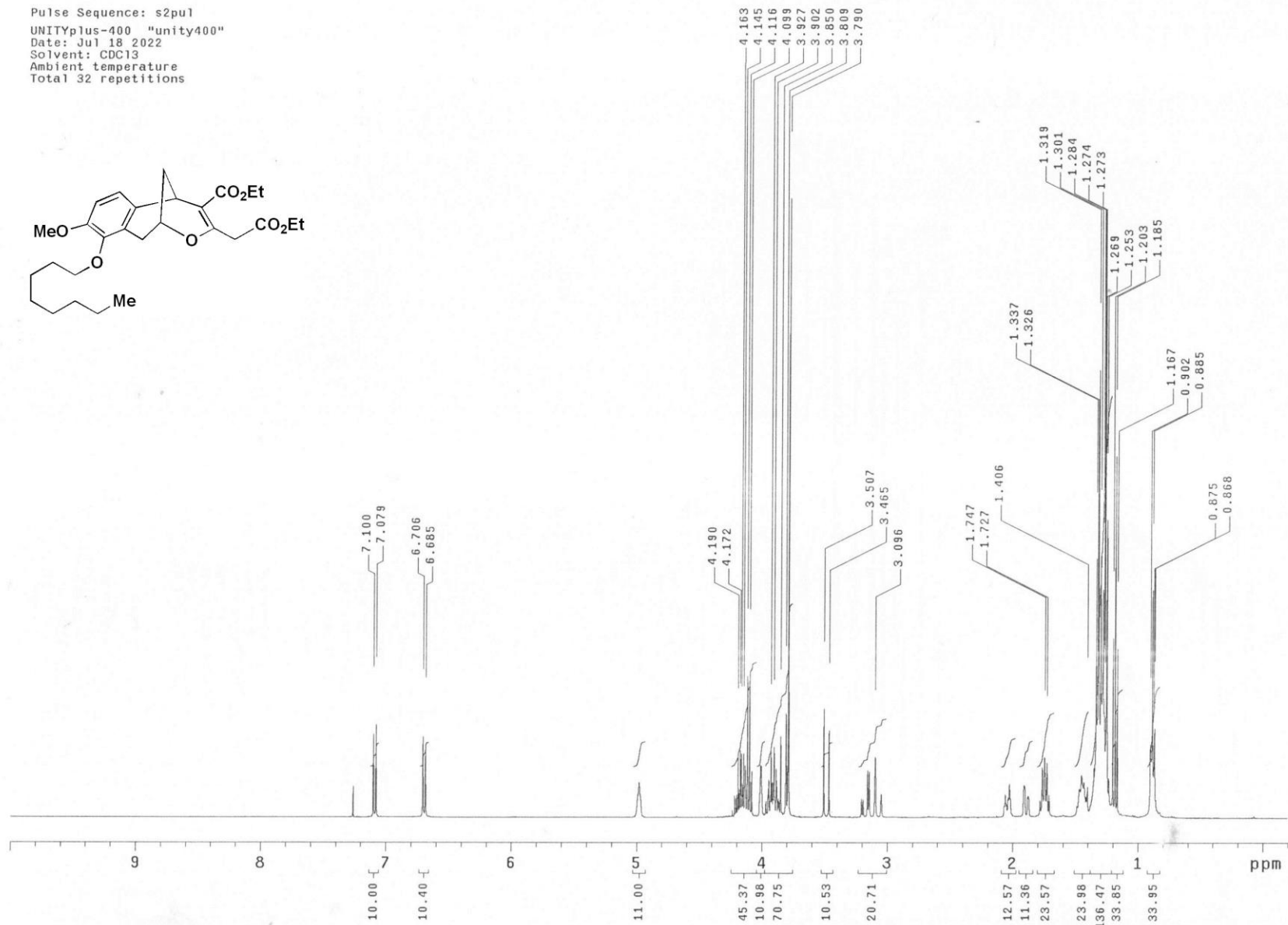
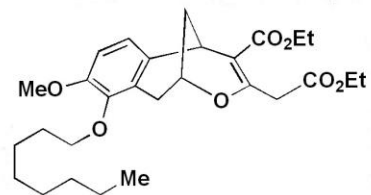
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jul 14 2022
Solvent: CDC13
Ambient temperature
Total 256 repetitions



Compound 6g (¹H-NMR spectral data)

YNB715

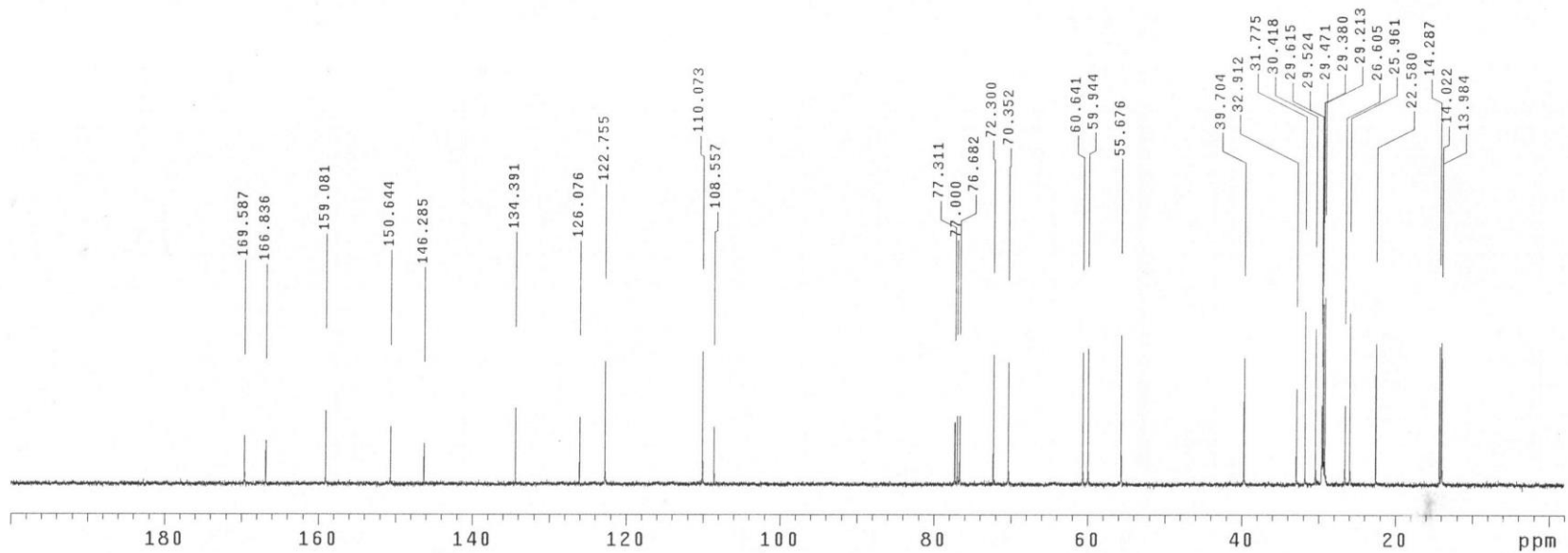
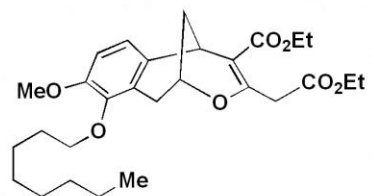
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Jul 18 2022
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 6g (¹³C-NMR spectral data)

YNB715

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jul 18 2022
Solvent: CDCl₃
Ambient temperature
Total 656 repetitions



Compound 6h (¹H-NMR spectral data)

YNB719

Pulse Sequence: s2pu1

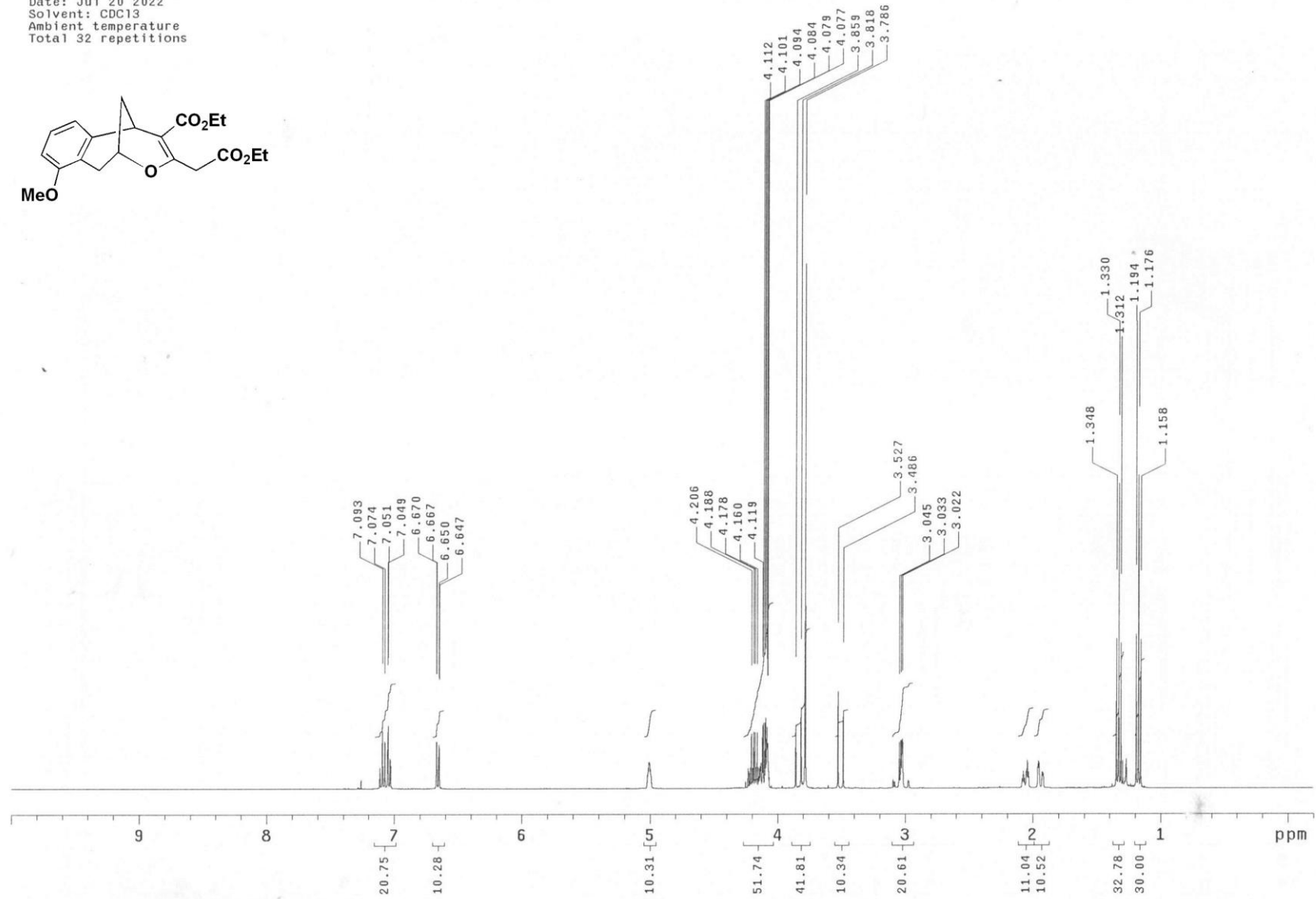
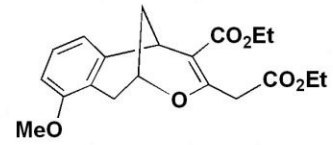
UNITYplus-400 "unity400"

Date: Jul 20 2022

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 6h (¹³C-NMR spectral data)

YNB719

Pulse Sequence: s2pu1

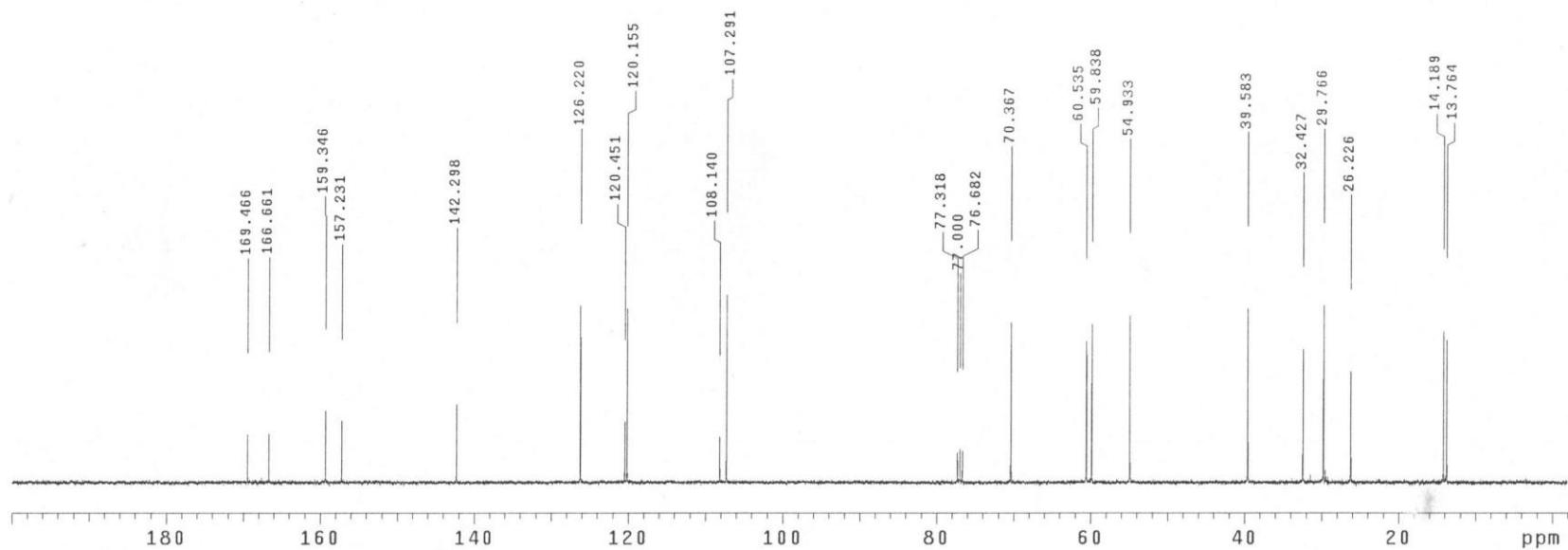
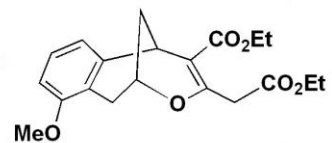
UNITYplus-400 "unity400"

Date: Jul 20 2022

Solvent: CDCl₃

Ambient temperature

Total 128 repetitions



Compound 6i (¹H-NMR spectral data)

YNB725

Pulse Sequence: s2pu1

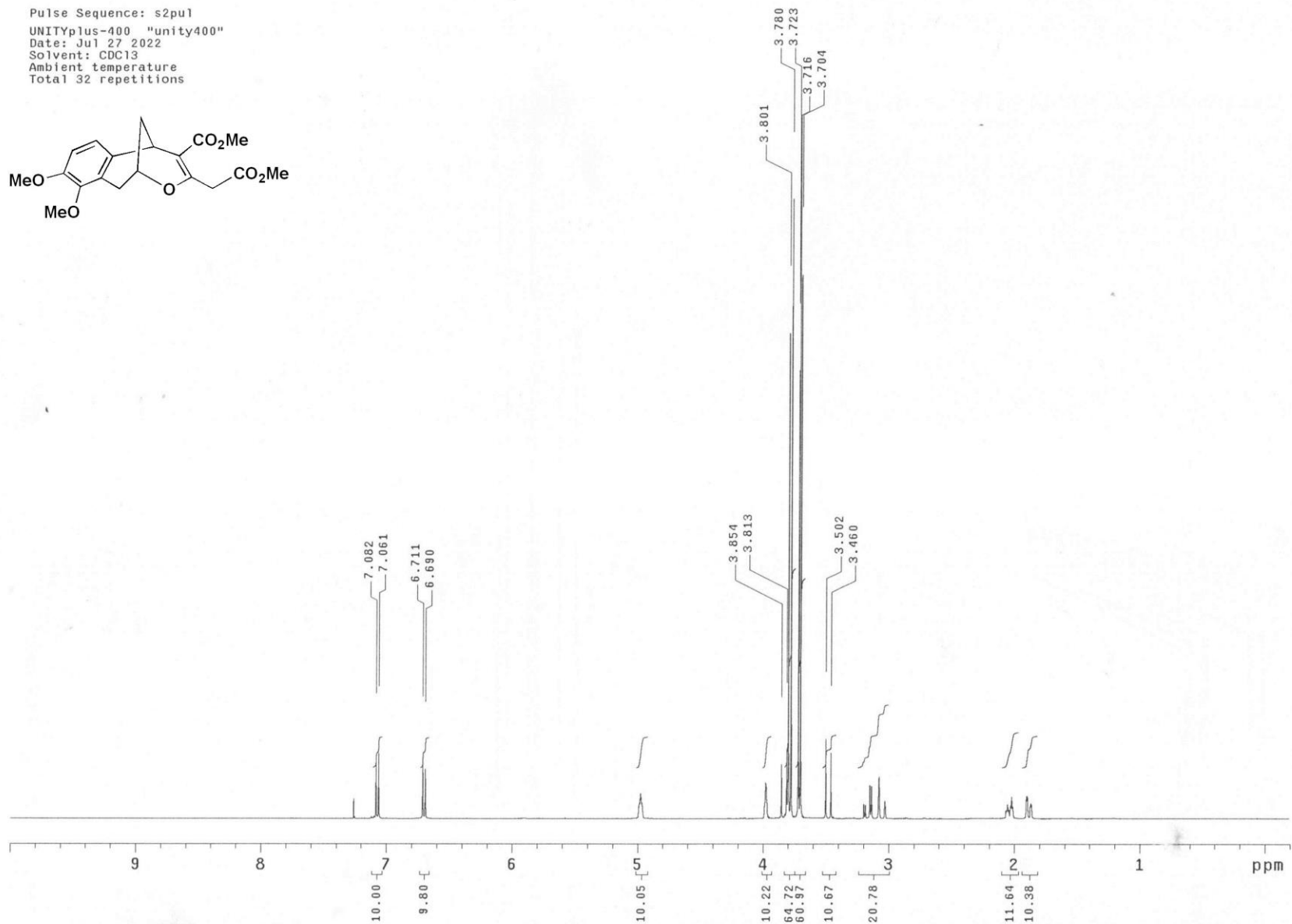
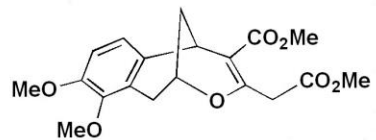
UNITYplus-400 "unity400"

Date: Jul 27 2022

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 6i (¹³C-NMR spectral data)

YNB725

Pulse Sequence: s2pu1

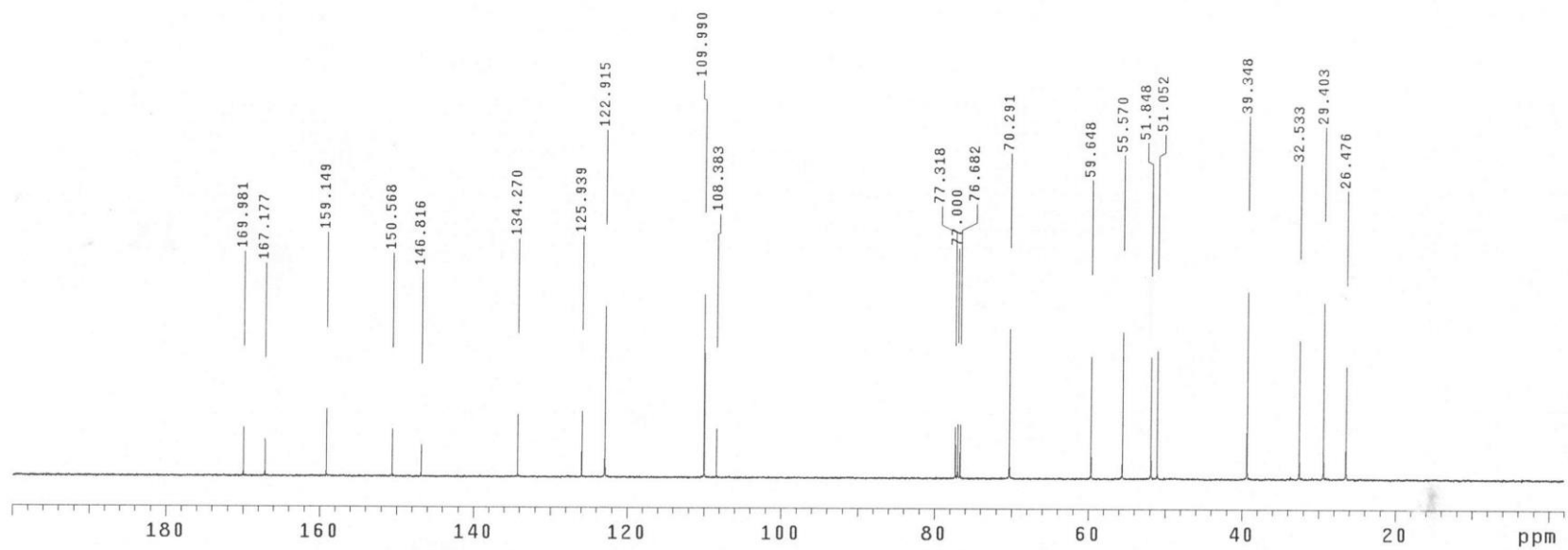
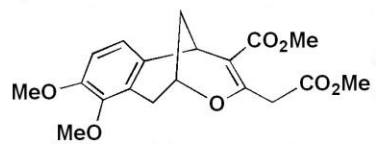
UNITYplus-400 "unity400"

Date: Jul 27 2022

Solvent: CDCl₃

Ambient temperature

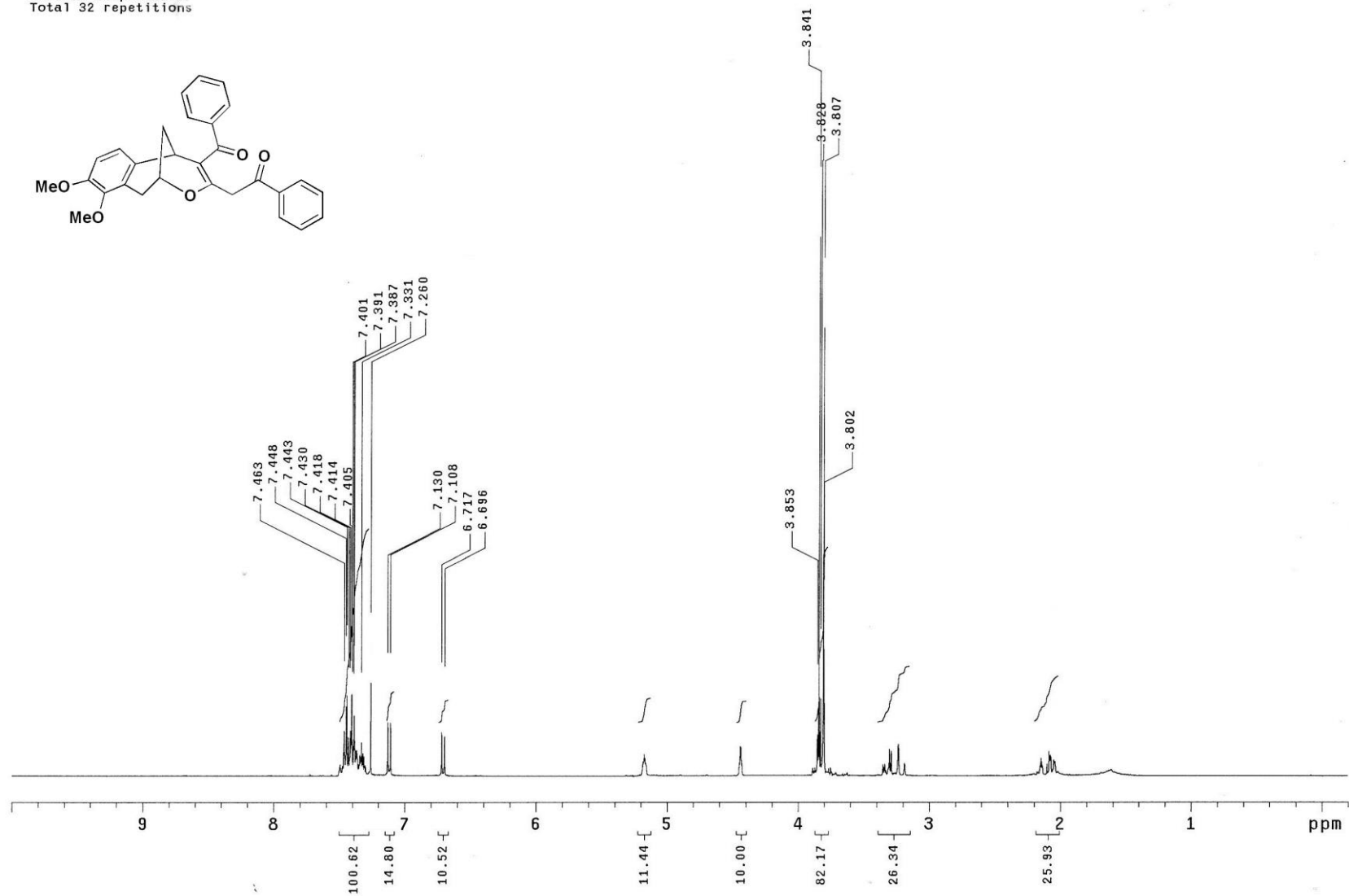
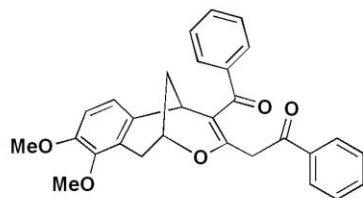
Total 2528 repetitions



Compound 6j (¹H-NMR spectral data)

YNB815

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Feb 6 2023
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6j (¹³C-NMR spectral data)

YNB815

Pulse Sequence: s2pu1

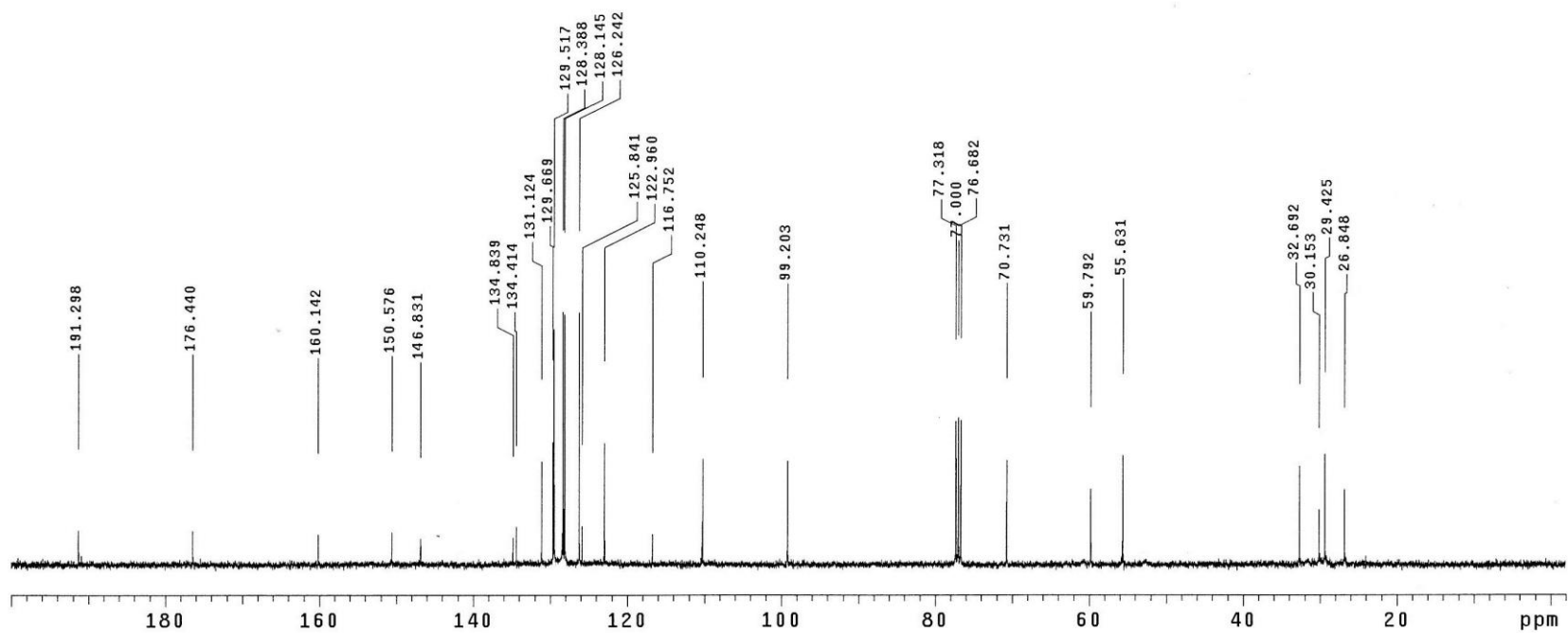
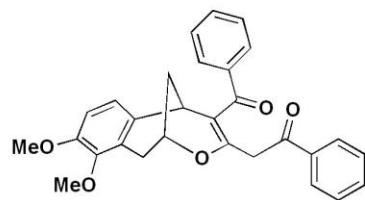
UNITYplus-400 "unity400"

Date: Feb 6 2023

Solvent: CDC13

Ambient temperature

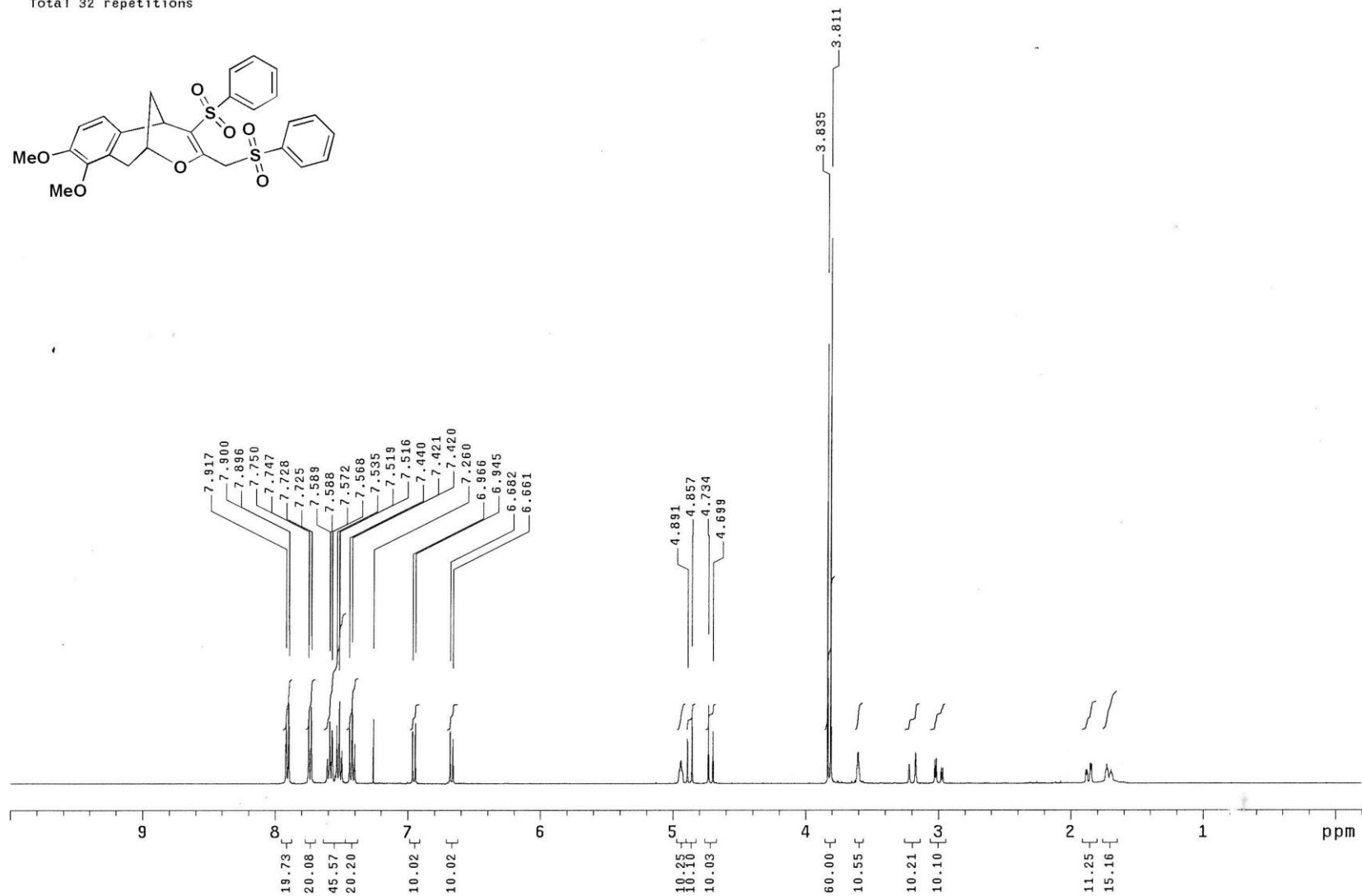
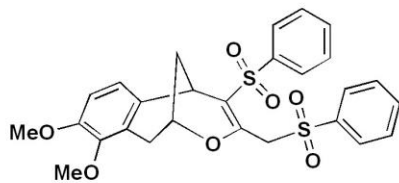
Total 2464 repetitions



Compound 6k (¹H-NMR spectral data)

YN8810

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Aug 11 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6k (¹³C-NMR spectral data)

YNB810

Pulse Sequence: s2pu1

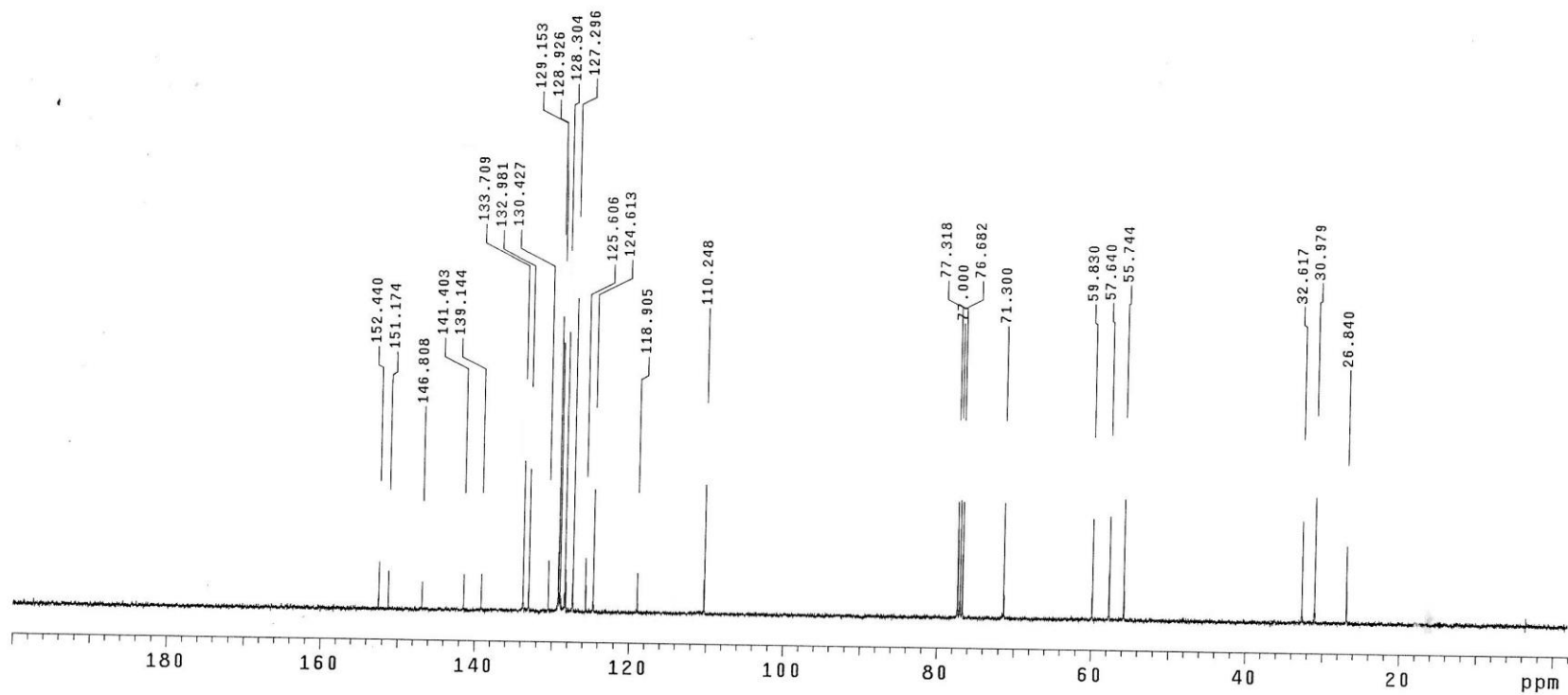
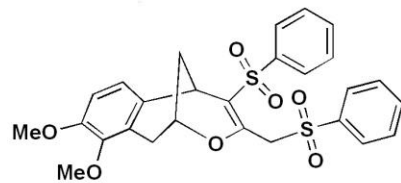
UNITYplus-400 "unity400"

Date: Aug 11 2022

Solvent: CDCl₃

Ambient temperature

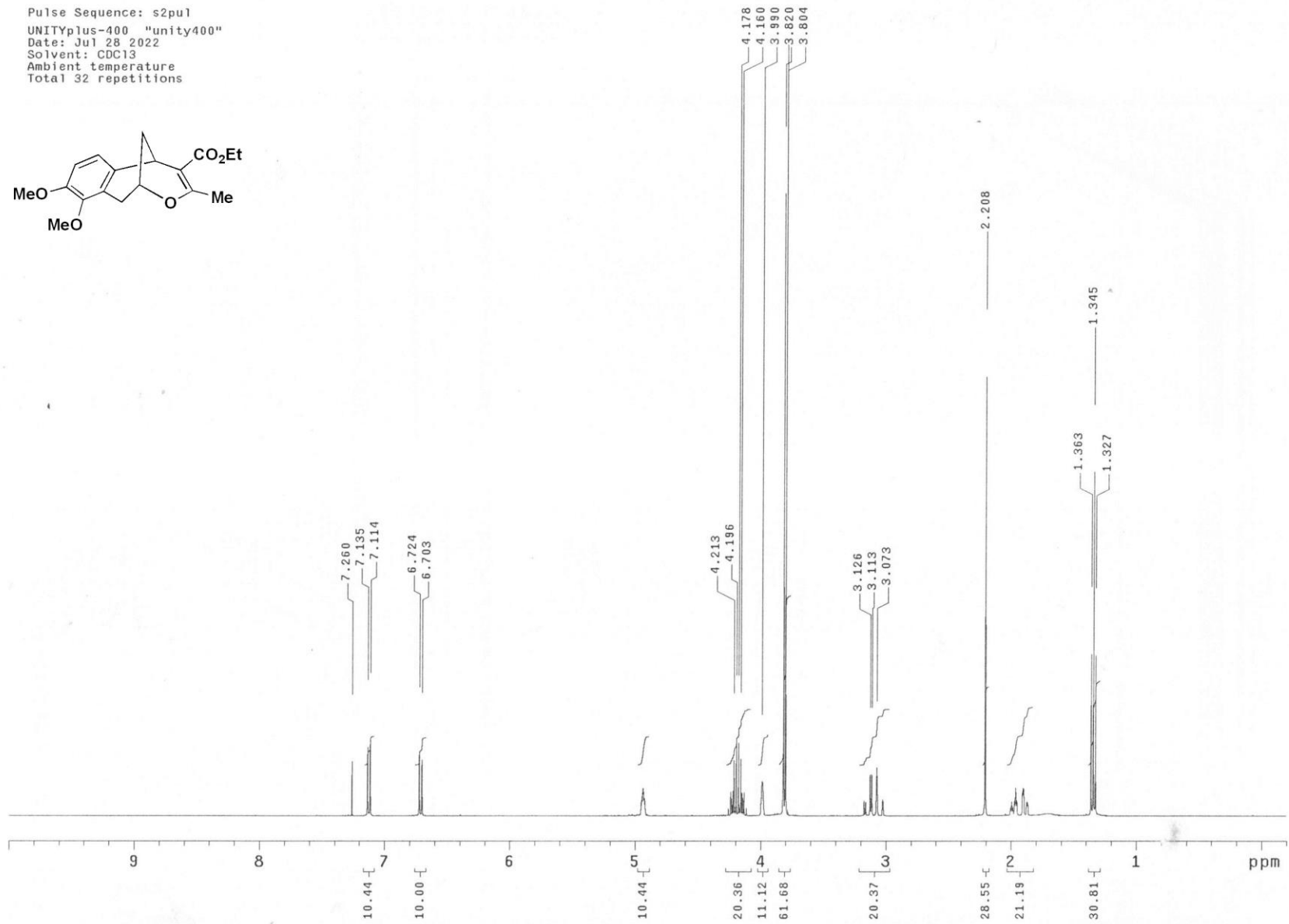
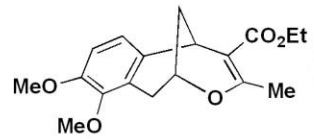
Total 2432 repetitions



Compound 6I (¹H-NMR spectral data)

YNB727

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jul 28 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6I (¹³C-NMR spectral data)

YNB727

Pulse Sequence: s2pu1

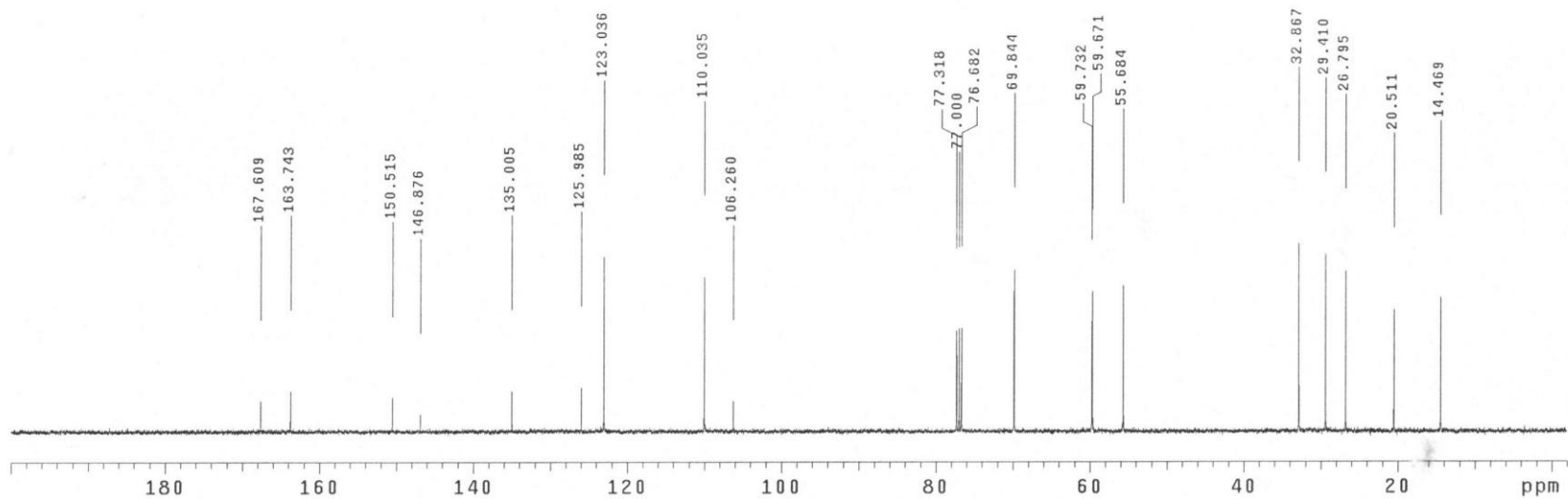
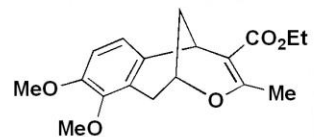
UNITYplus-400 "unity400"

Date: Jul 28 2022

Solvent: CDCl₃

Ambient temperature

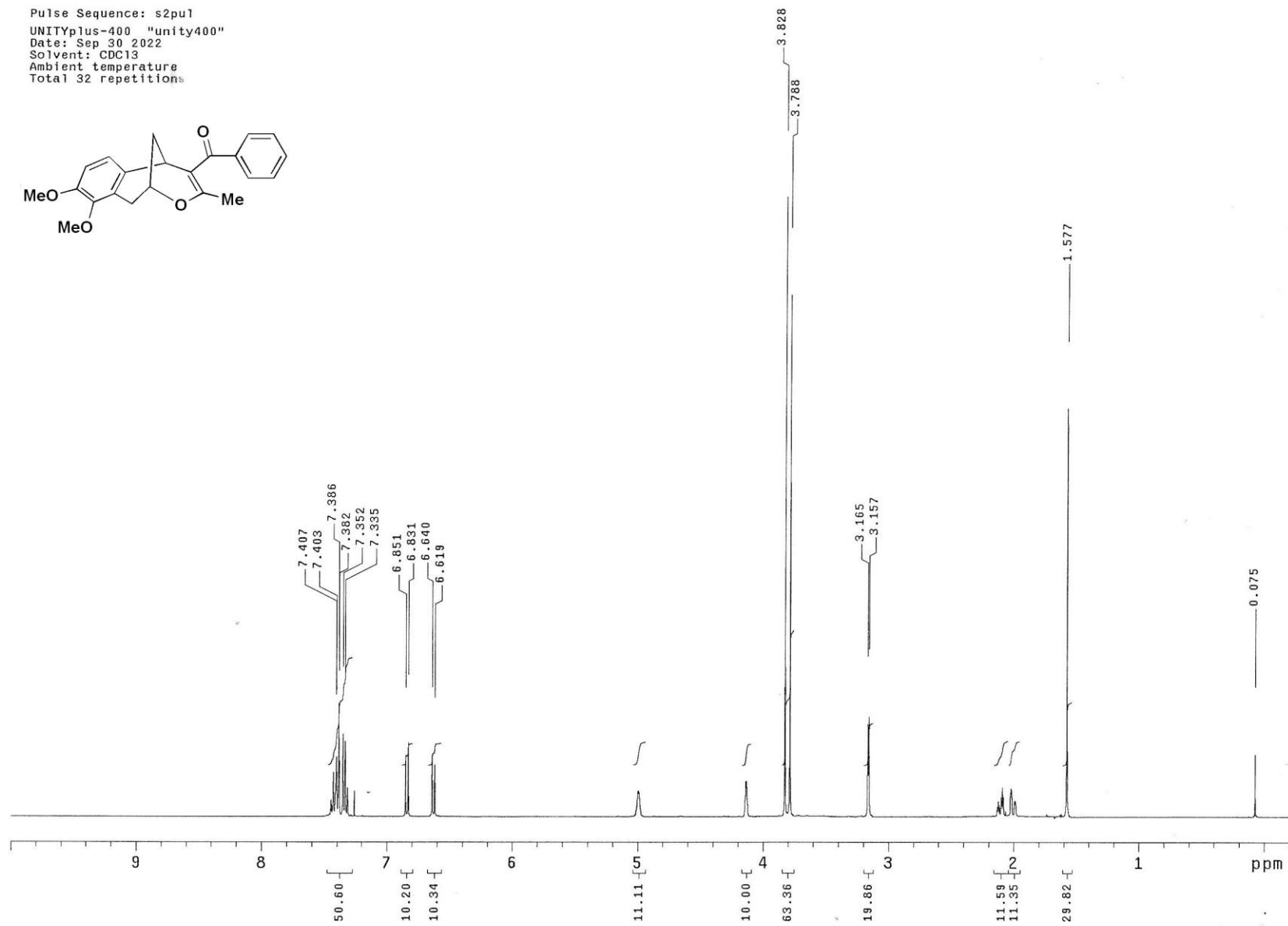
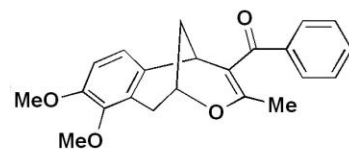
Total 1696 repetitions



Compound 6m (¹H-NMR spectral data)

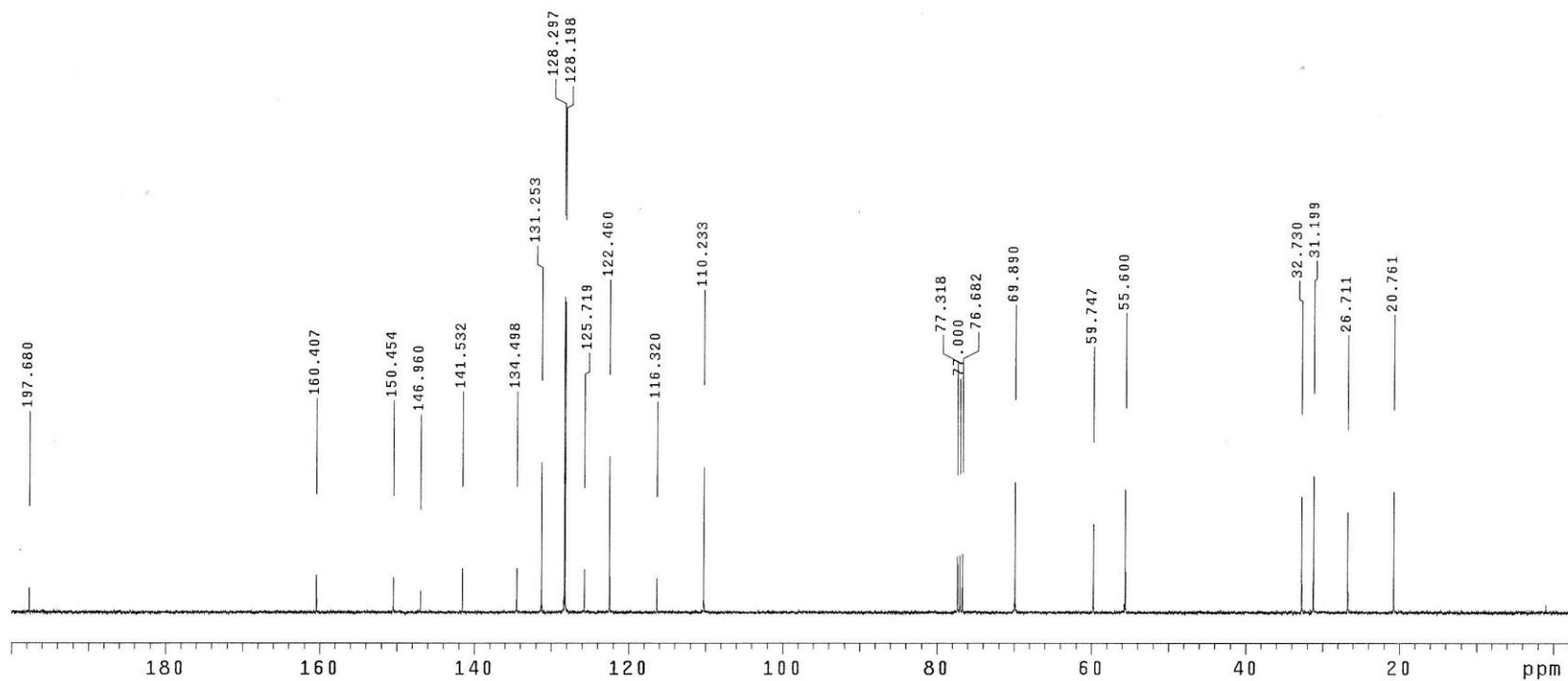
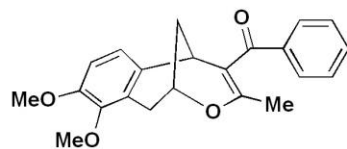
YNB2527

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 30 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6m (¹³C-NMR spectral data)

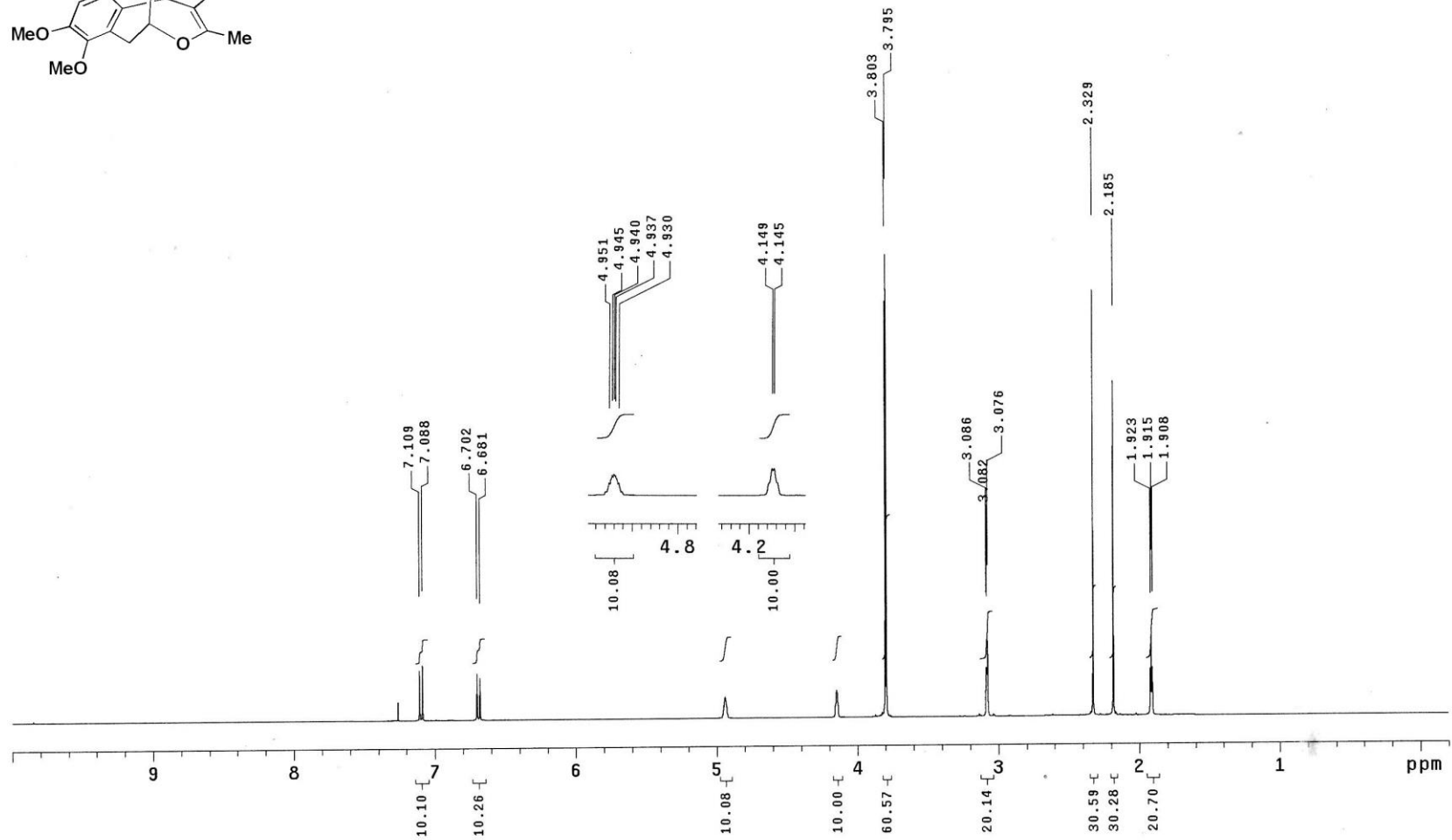
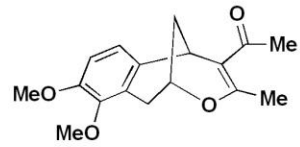
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 30 2022
Solvent: CDC13
Ambient temperature
Total 704 repetitions



Compound 6n (¹H-NMR spectral data)

YNB1205

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 6 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6n (¹³C-NMR spectral data)

YNB1205

Pulse Sequence: s2pu1

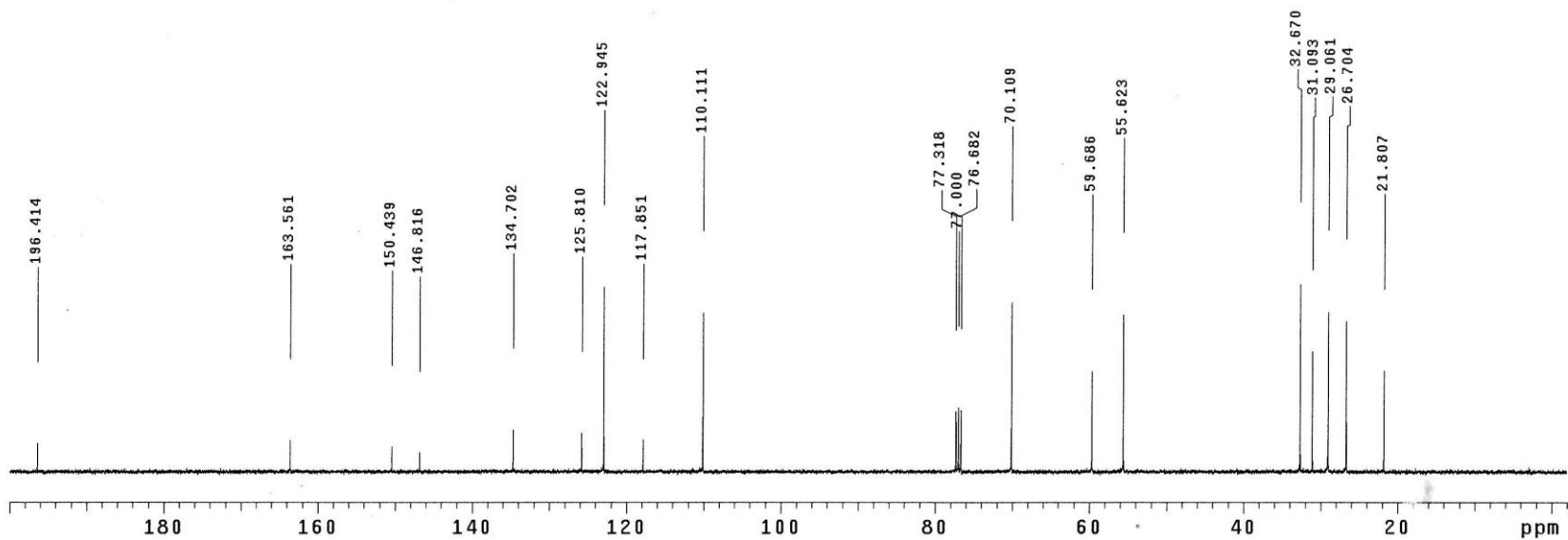
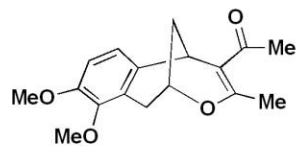
UNITYplus-400 "unity400"

Date: Dec 6 2022

Solvent: CDCl₃

Ambient temperature

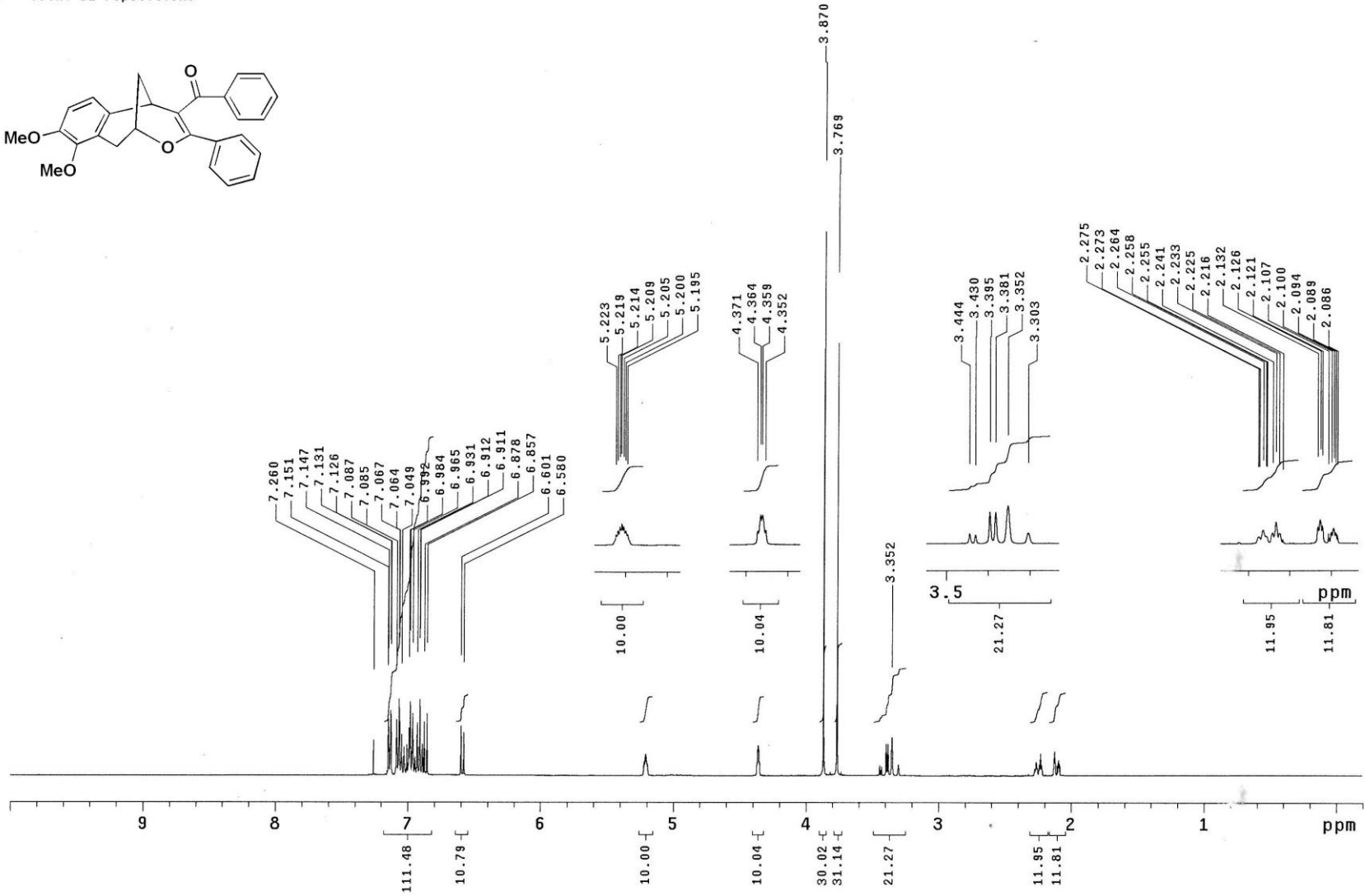
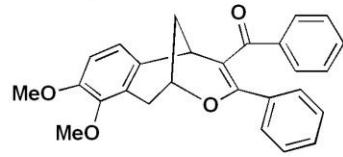
Total 1040 repetitions



Compound 6o (¹H-NMR spectral data)

YNB1207

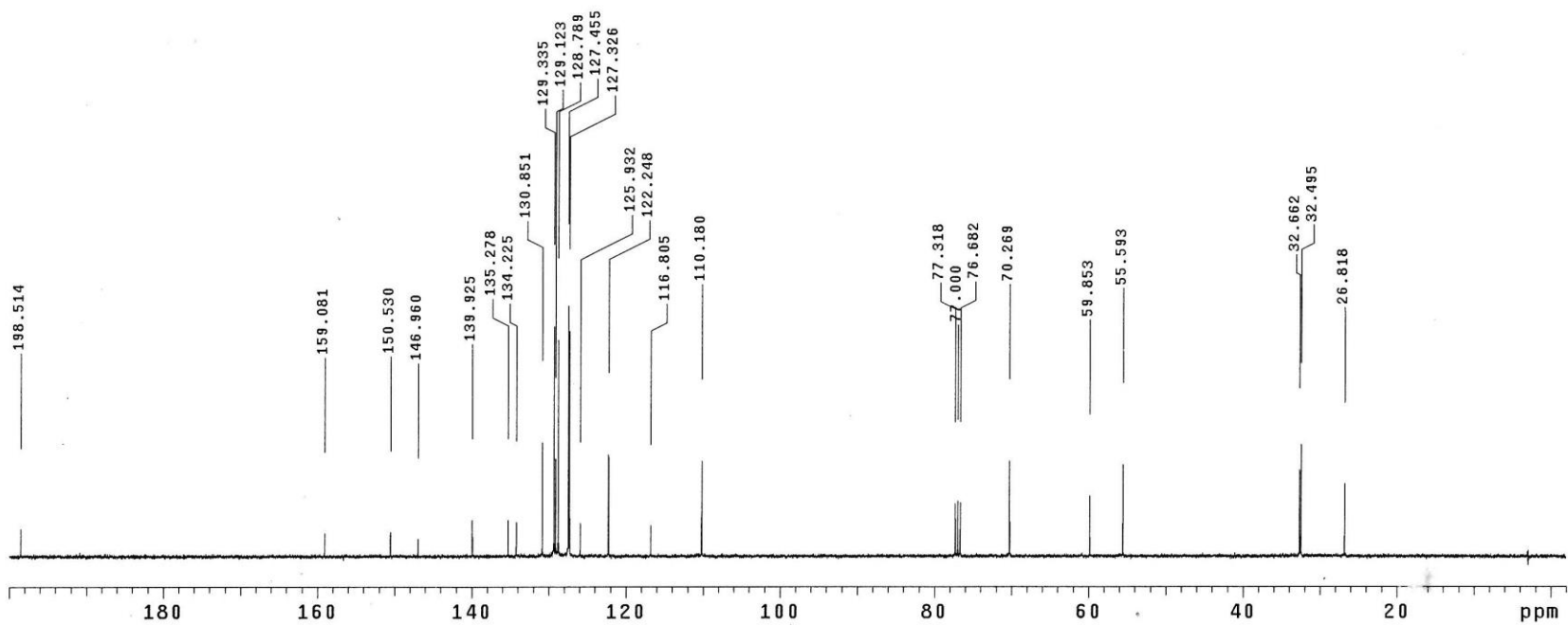
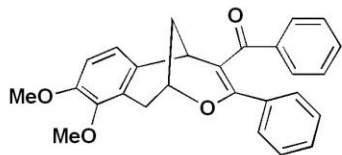
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Dec 8 2022
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 6o (¹³C-NMR spectral data)

YNB1207

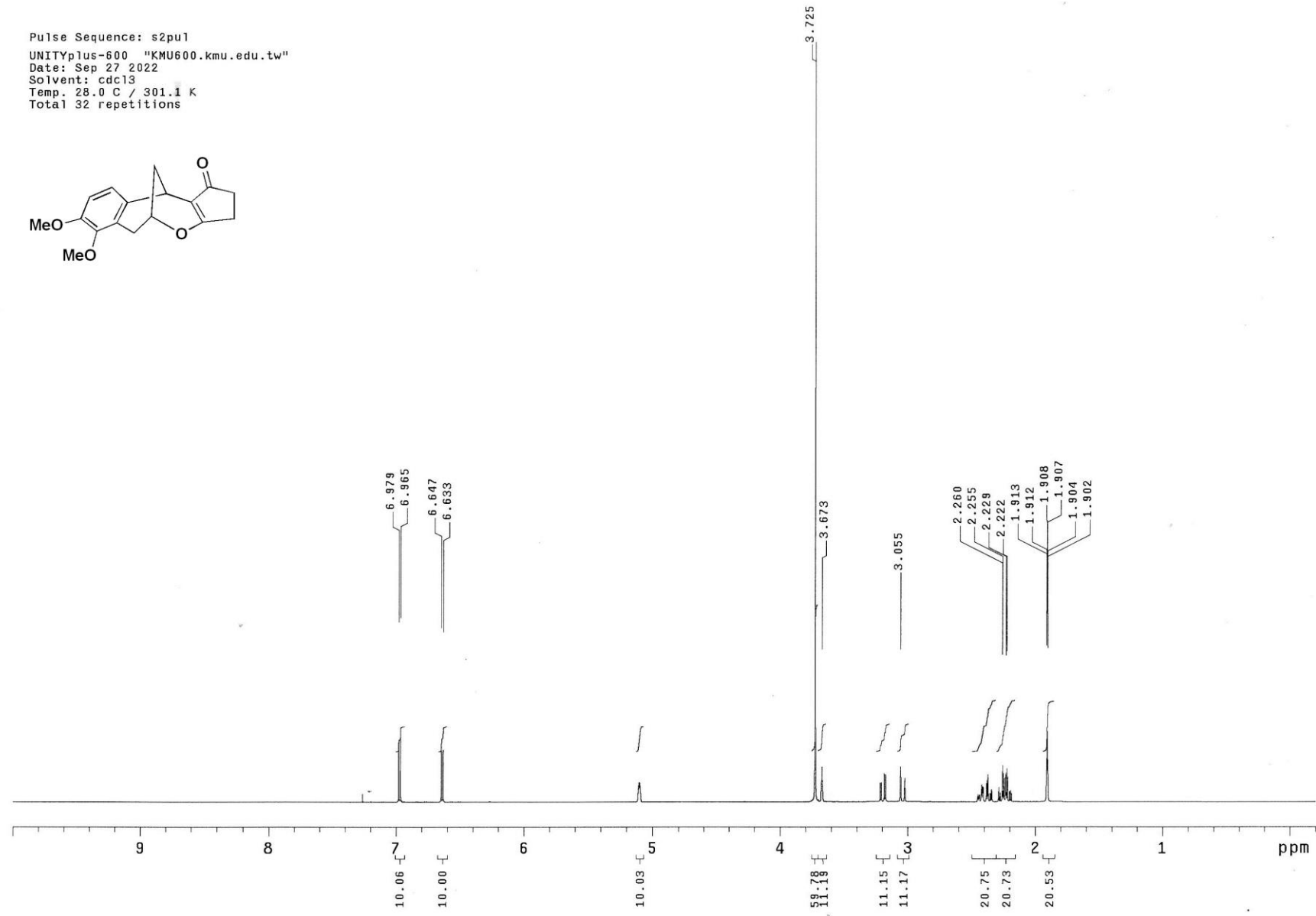
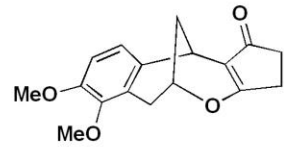
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 8 2022
Solvent: CDCl₃
Ambient temperature
Total 1024 repetitions



Compound 6p (¹H-NMR spectral data)

YNB926

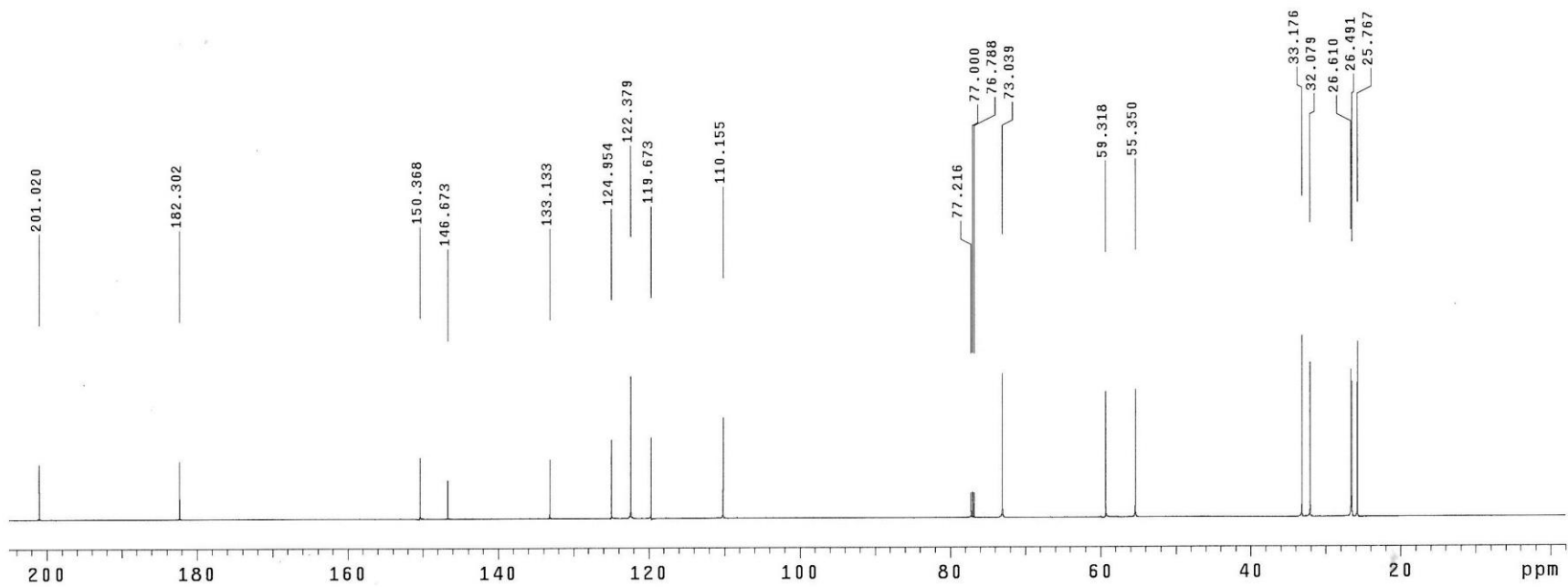
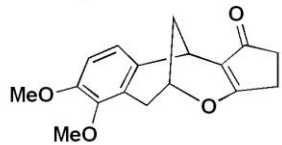
Pulse Sequence: s2pu1
UNITYplus-600 "KMU600.kmu.edu.tw"
Date: Sep 27 2022
Solvent: cdc13
Temp. 28.0 C / 301.1 K
Total 32 repetitions



Compound 6p (¹³C-NMR spectral data)

YNB926

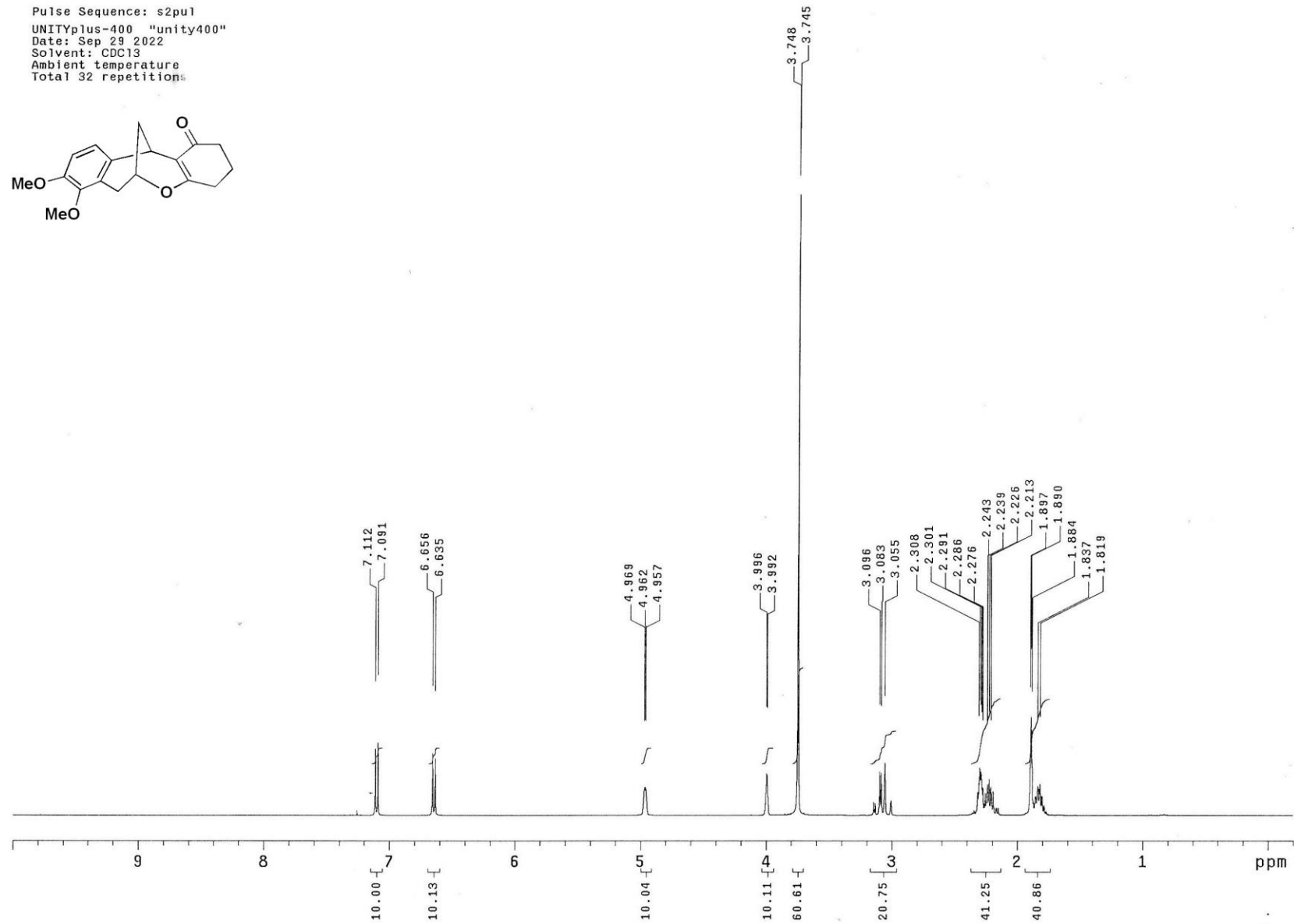
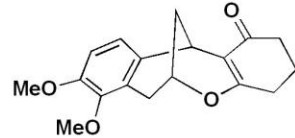
Pulse Sequence: s2pu1
UNITYplus-600 "KMU600.kmu.edu.tw"
Date: Sep 27 2022
Solvent: cdc13
Temp: 28.0 C / 301.1 K
Total 936 repetitions



Compound 6q (¹H-NMR spectral data)

YNB928

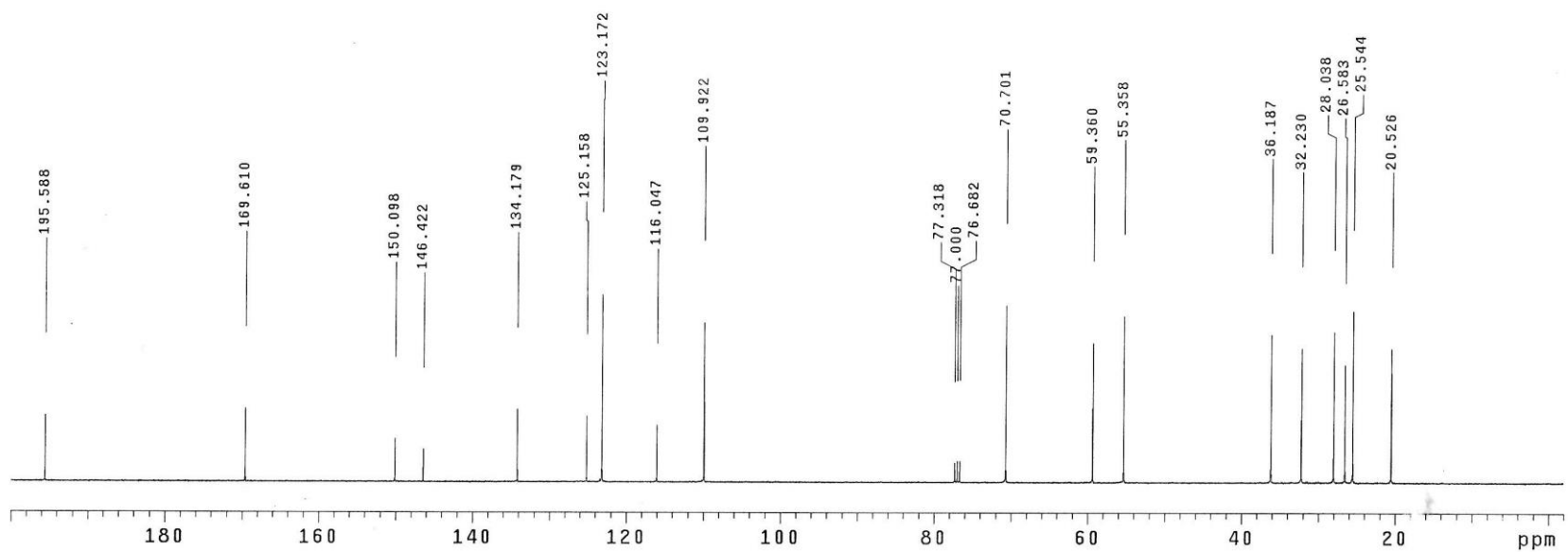
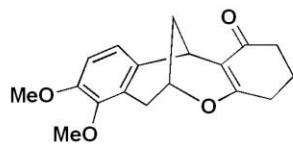
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 29 2022
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 6q (¹³C-NMR spectral data)

YNB928

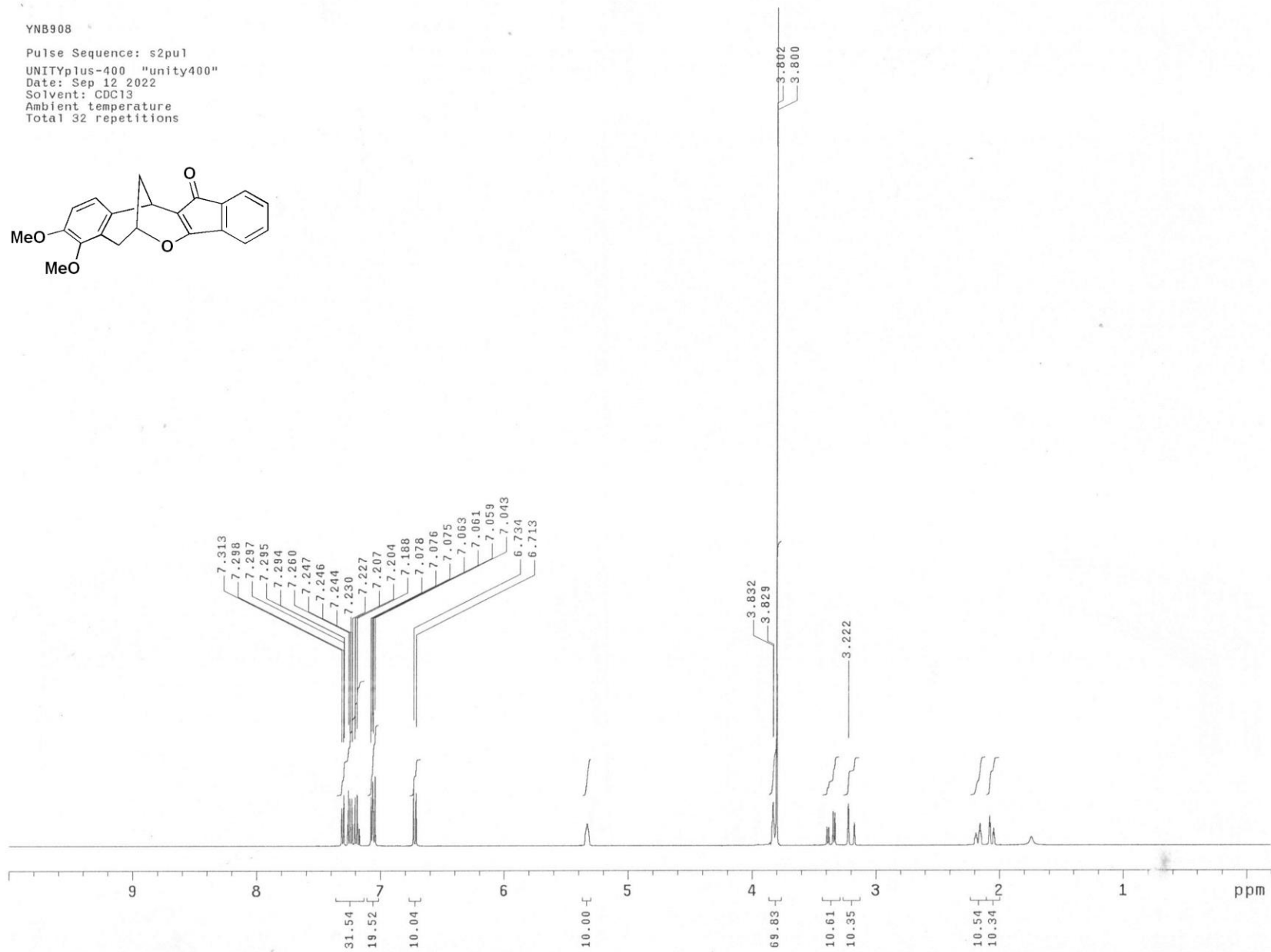
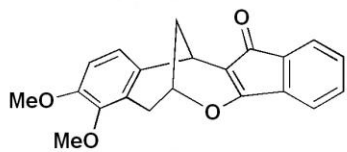
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 29 2022
Solvent: CDCl₃
Ambient temperature
Total 1056 repetitions



Compound 6r (¹H-NMR spectral data)

YNB908

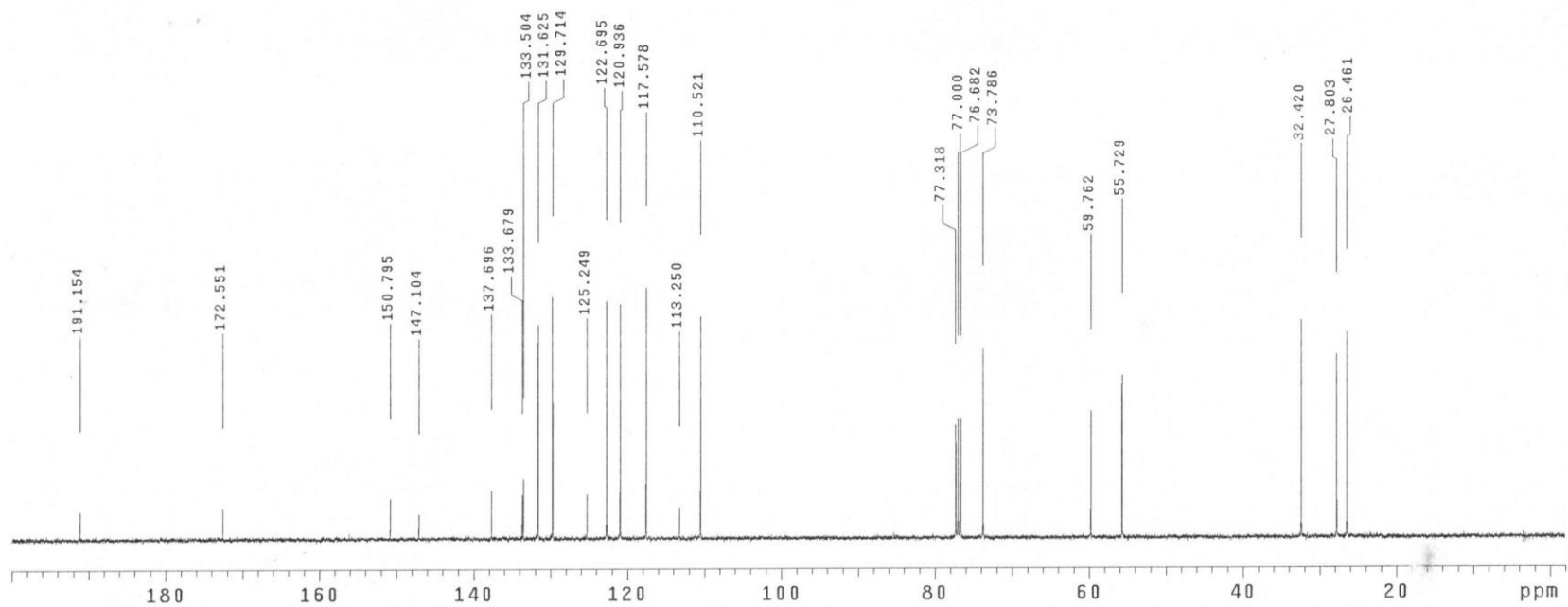
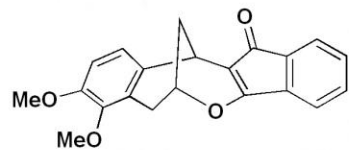
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 12 2022
Solvent: CDC13
Ambient temperature
Total 32 repetitions



Compound 6r (¹³C-NMR spectral data)

YNB908

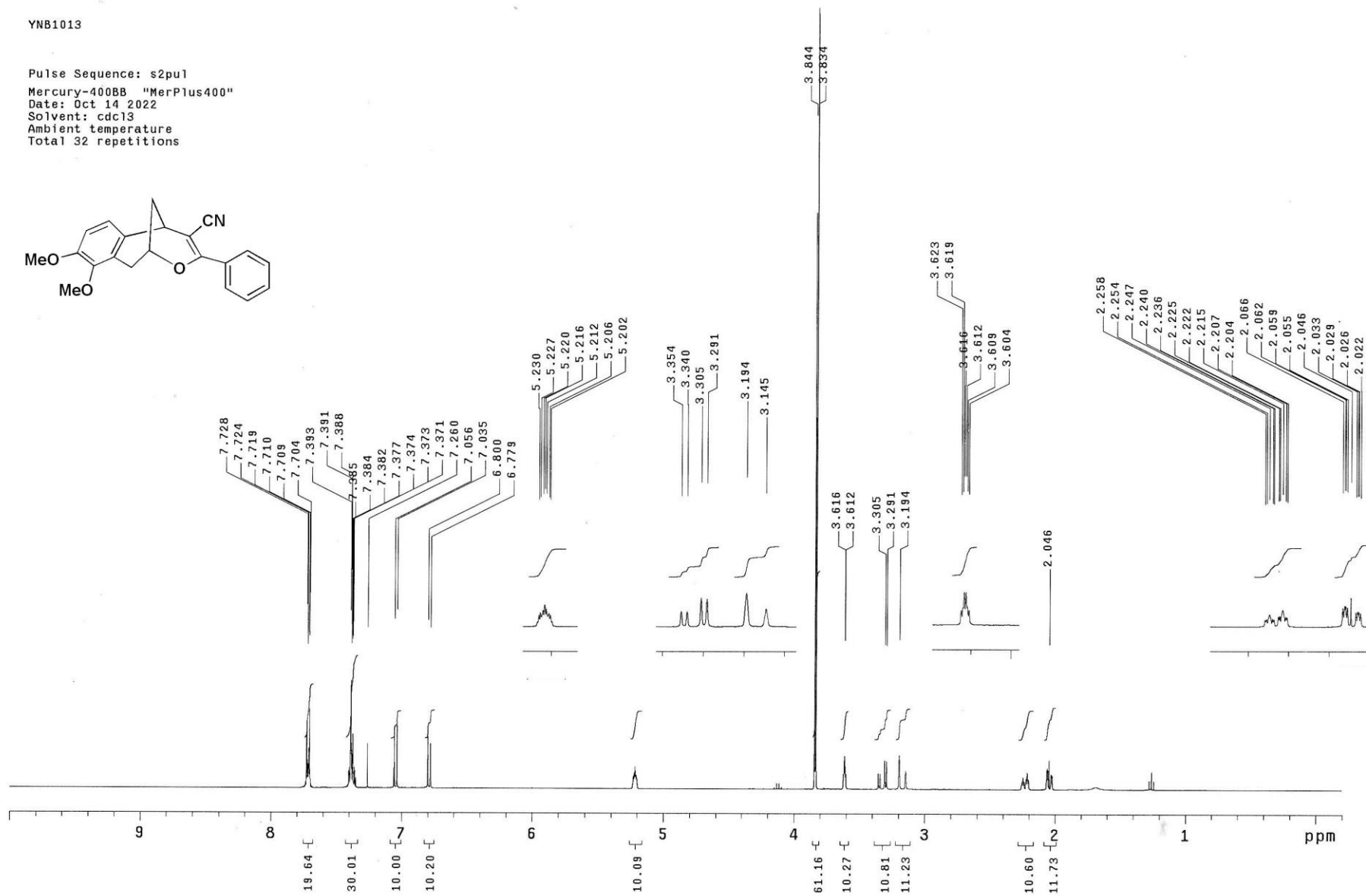
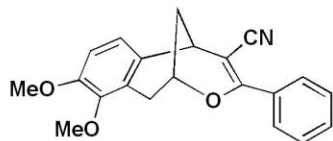
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 12 2022
Solvent: CDCl₃
Ambient temperature
Total 3216 repetitions



Compound 6s (¹H-NMR spectral data)

YNB1013

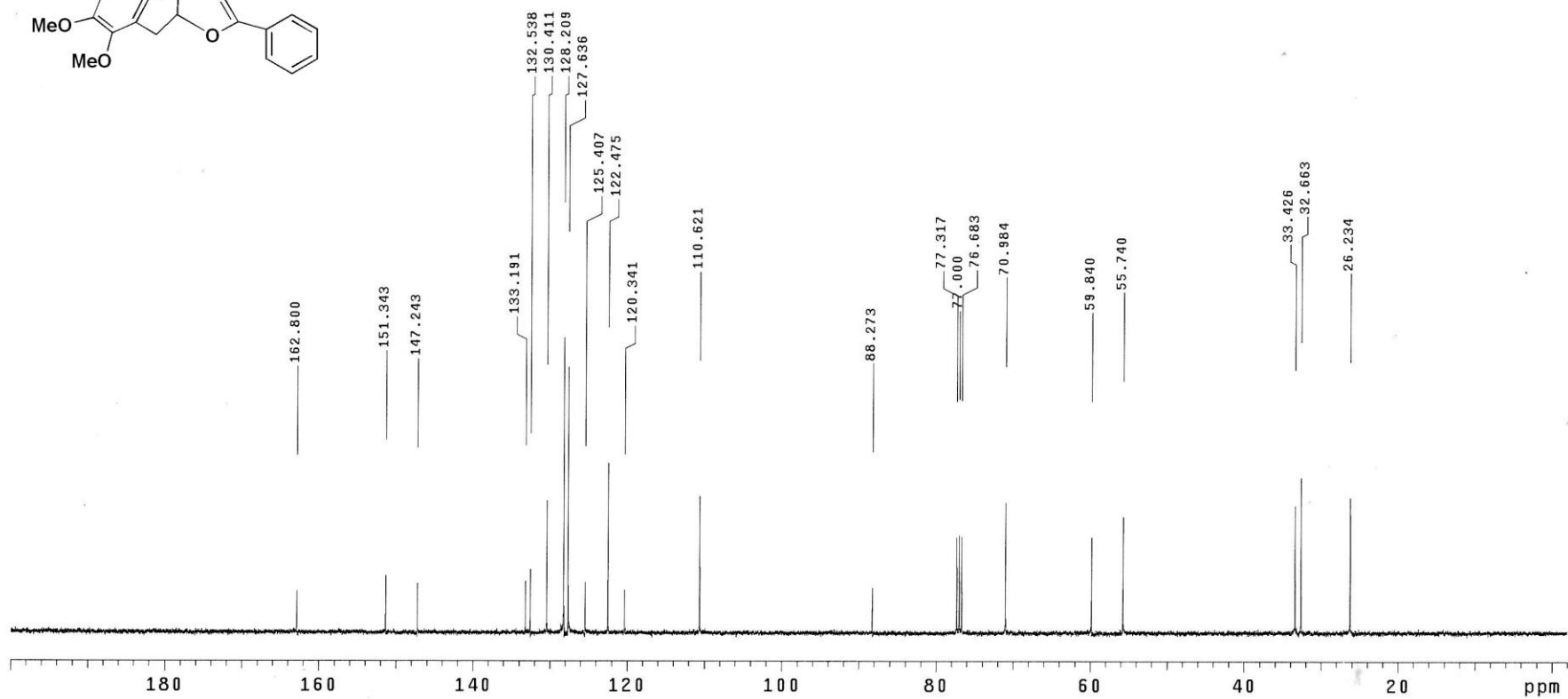
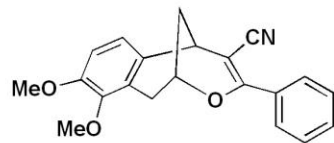
Pulse Sequence: s2pu1
 Mercury-400BB "MerPlus400"
 Date: Oct 14 2022
 Solvent: cdc13
 Ambient temperature
 Total 32 repetitions



Compound 6s (¹³C-NMR spectral data)

YNB1013

Pulse Sequence: s2pu1
Mercury-400BB "MerPlus400"
Date: Oct 14 2022
Solvent: cdc13
Ambient temperature
Total 400 repetitions



Compound 6t (¹H-NMR spectral data-1)

YNB33-38

Pulse Sequence: s2pu1

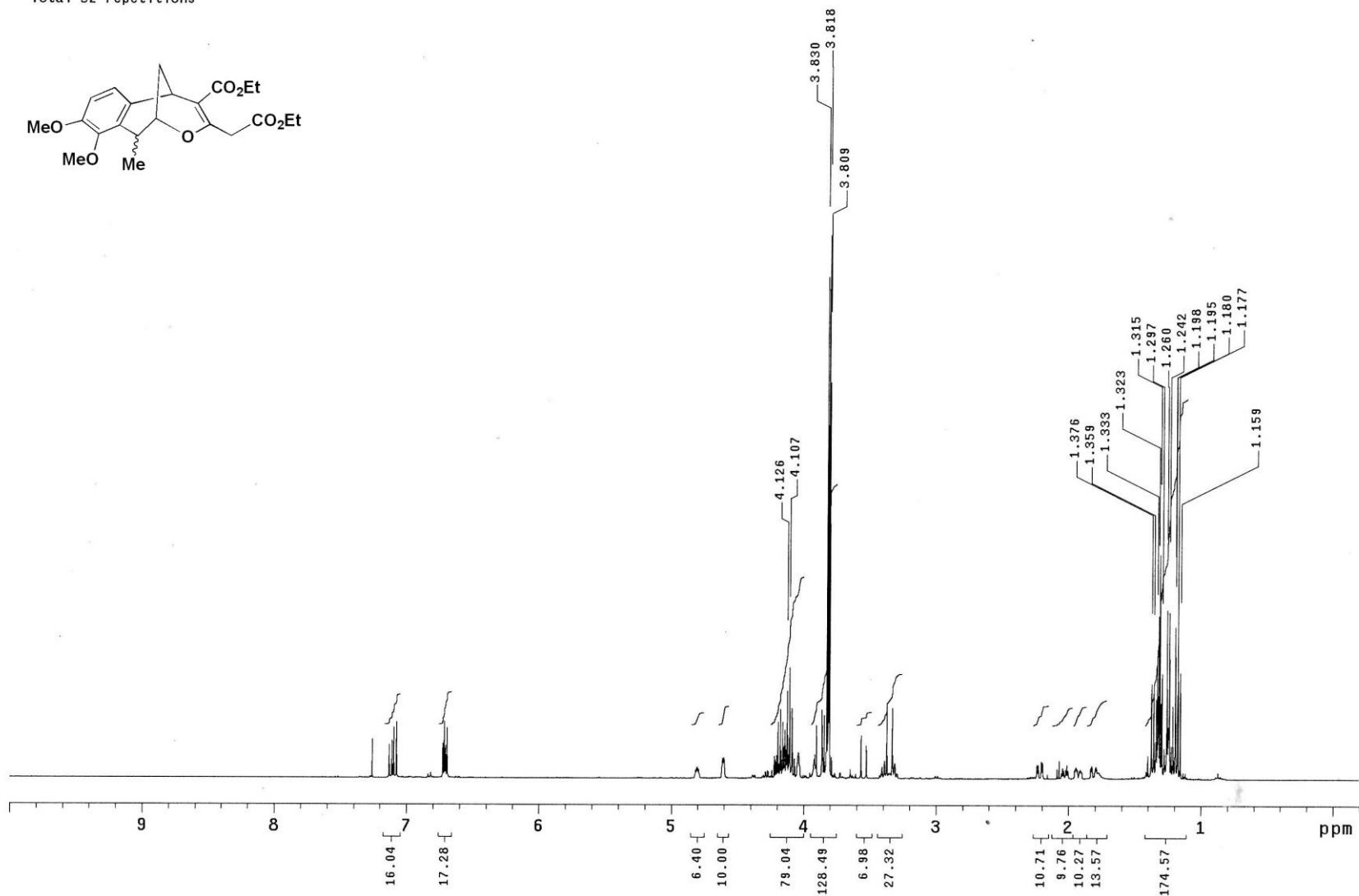
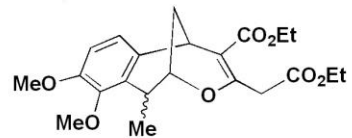
UNITYplus-400 "unity400"

Date: Feb 8 2023

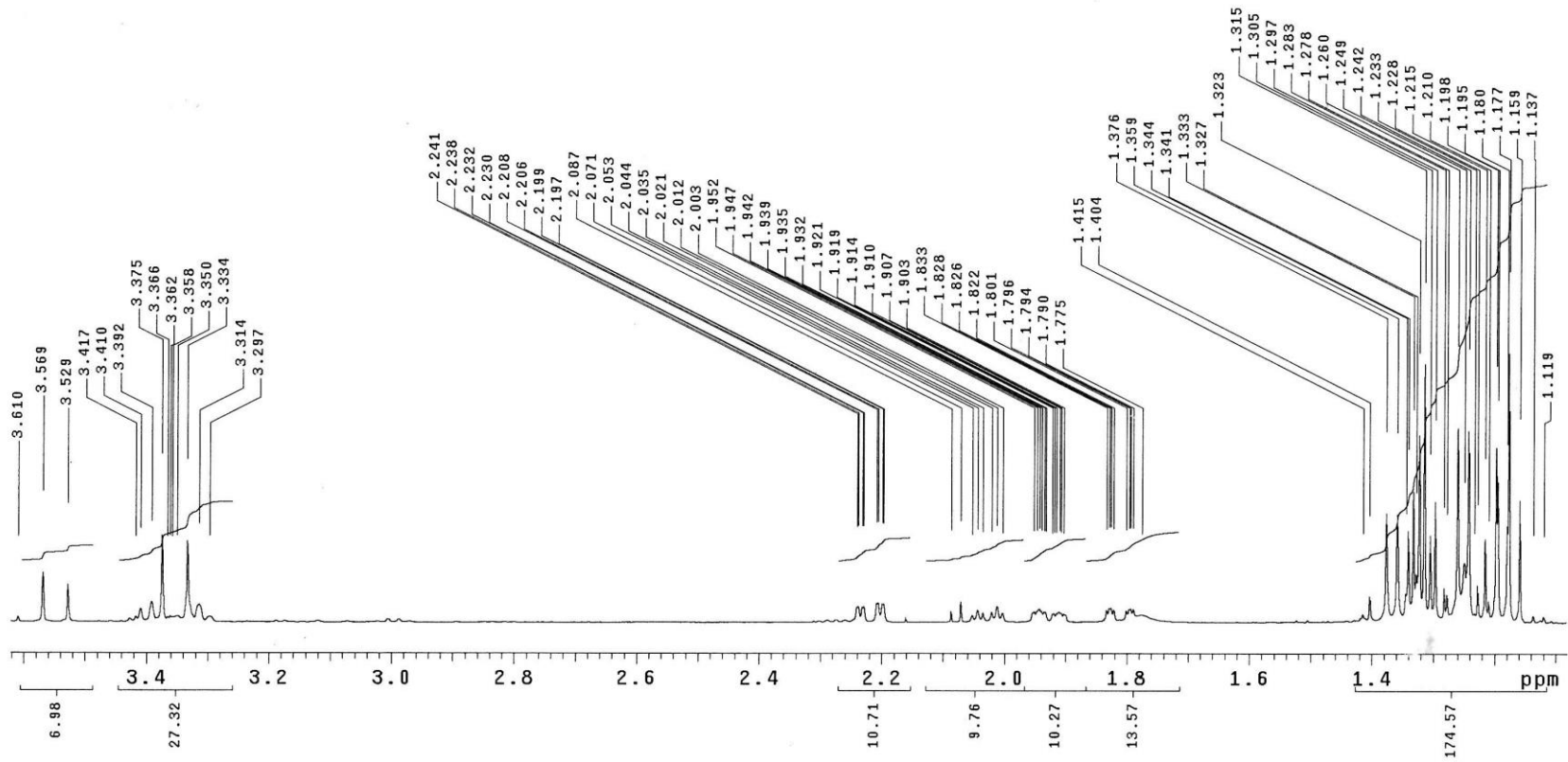
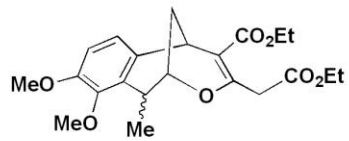
Solvent: CDC13

Ambient temperature

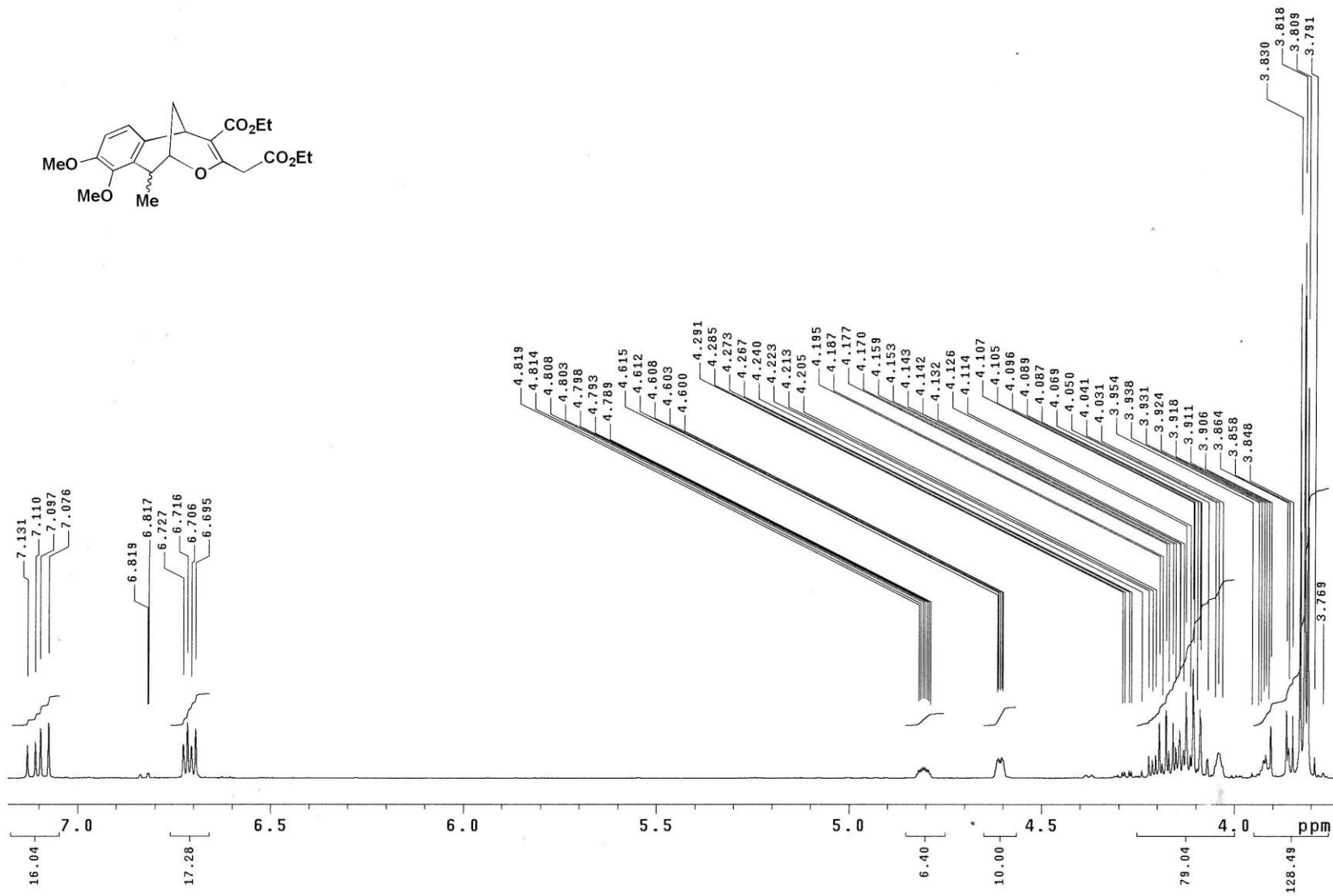
Total 32 repetitions



Compound 6t (¹H-NMR spectral data-2)



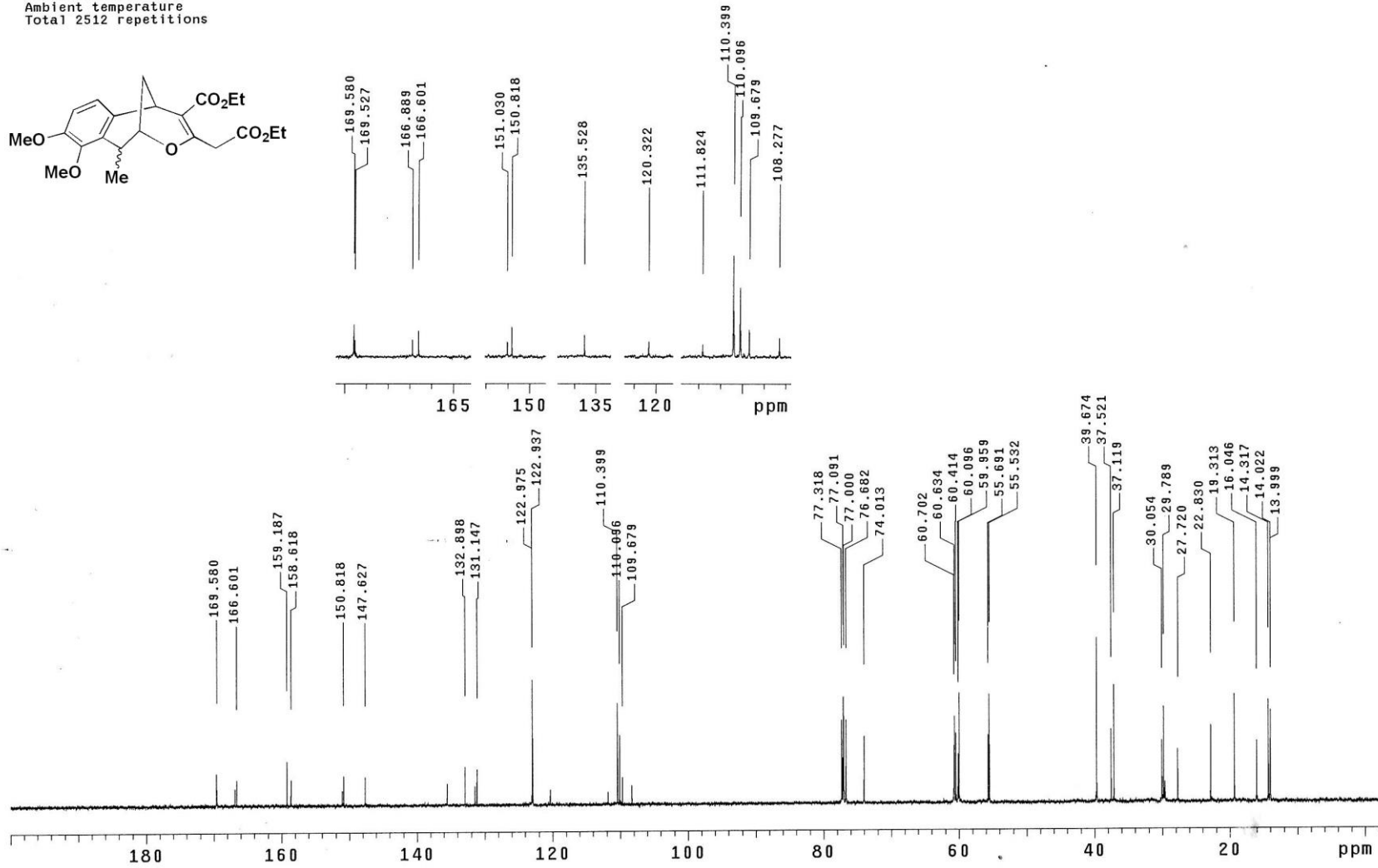
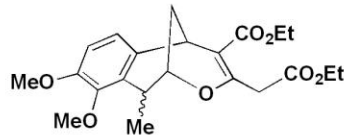
Compound 6t (¹H-NMR spectral data-3)



Compound 6t (¹³C-NMR spectral data)

YNB33-38

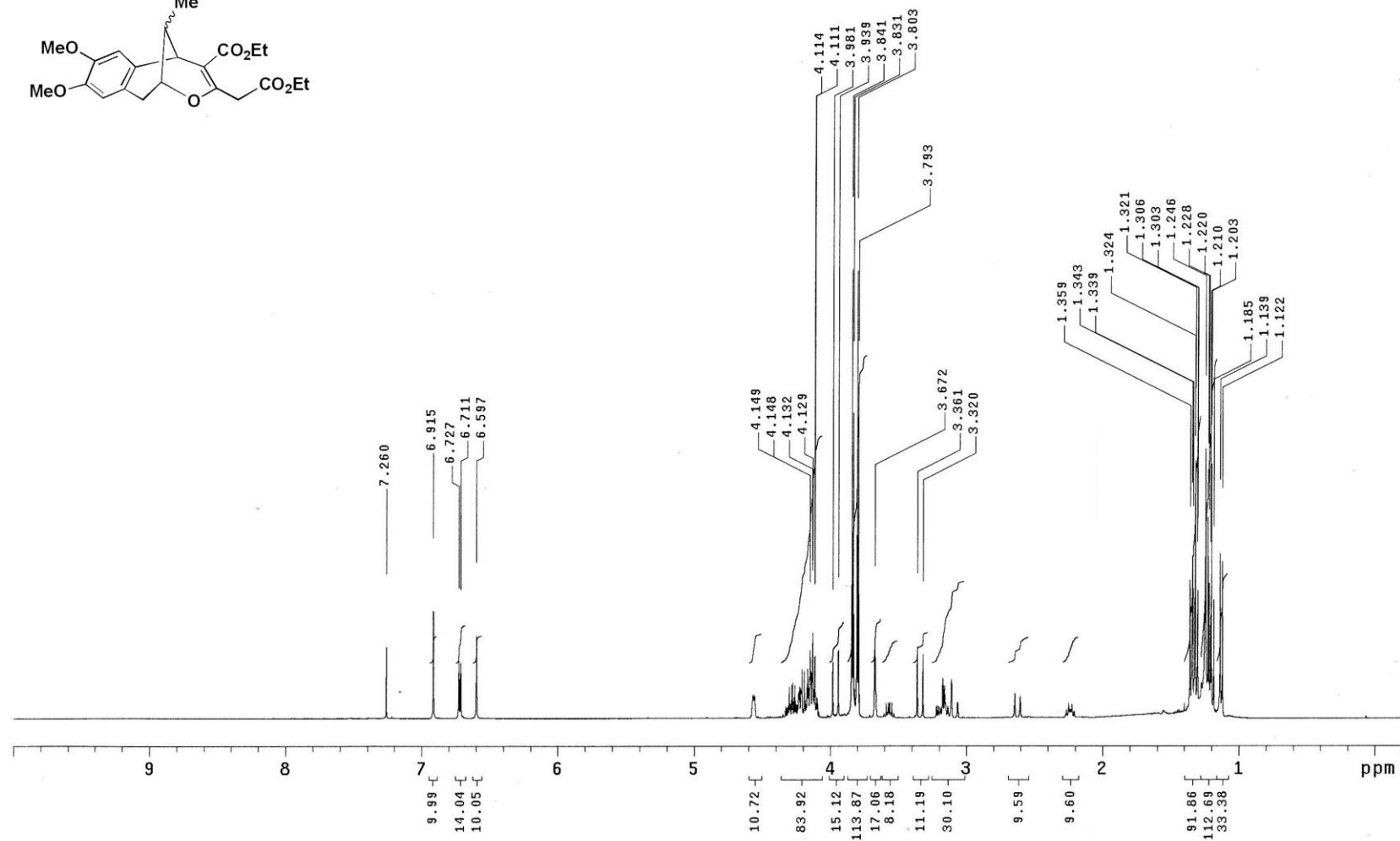
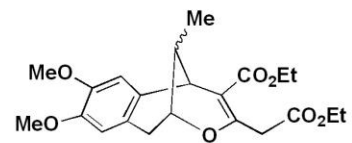
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Feb 8 2023
Solvent: CDCl₃
Ambient temperature
Total 2512 repetitions



Compound 6u (¹H-NMR spectral data)

YNB0206

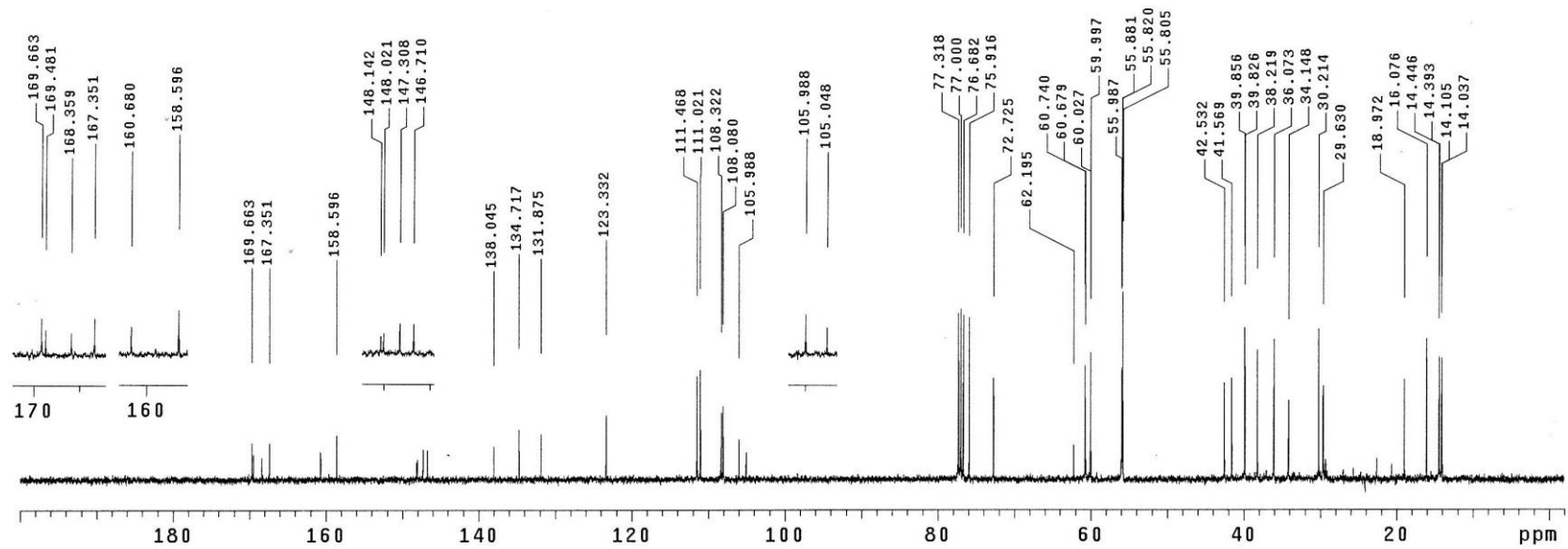
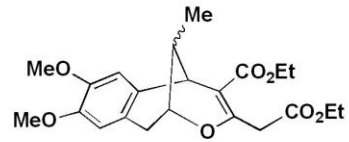
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Feb 7 2023
 Solvent: CDCl₃
 Ambient temperature
 Total 64 repetitions



Compound 6u (¹³C-NMR spectral data)

YNB0206

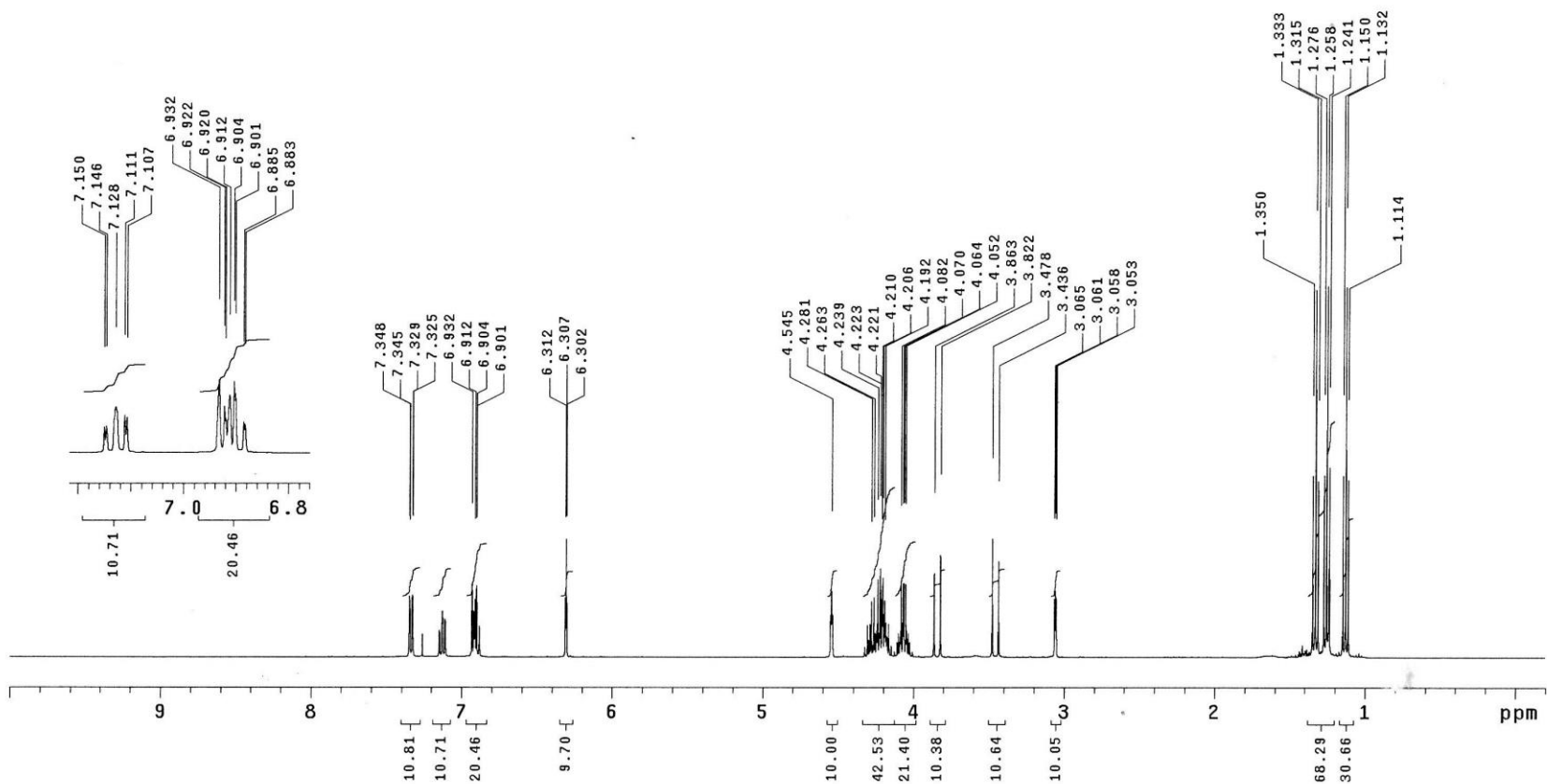
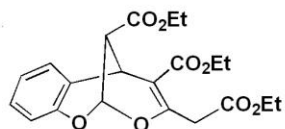
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Feb 7 2023
Solvent: CDCl₃
Ambient temperature
Total 3376 repetitions



Compound 6v (¹H-NMR spectral data)

YNB1202

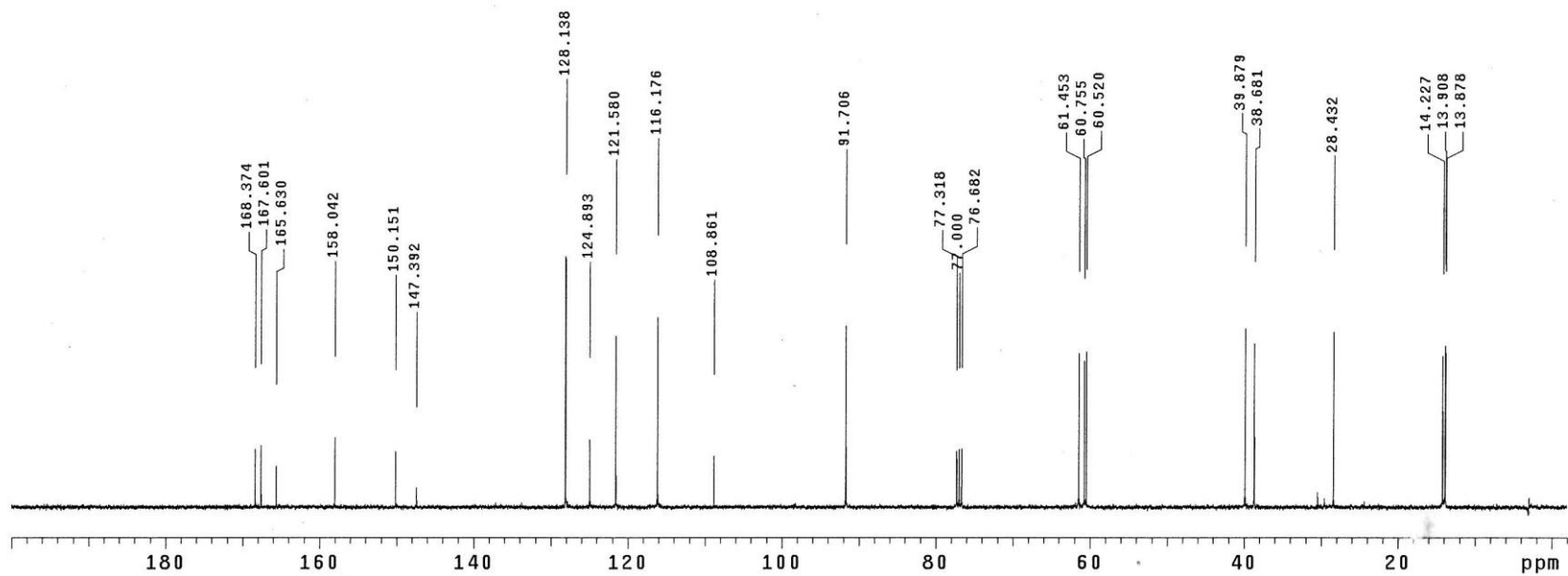
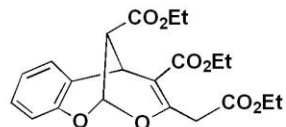
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Dec 2 2022
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 6v (¹³C-NMR spectral data)

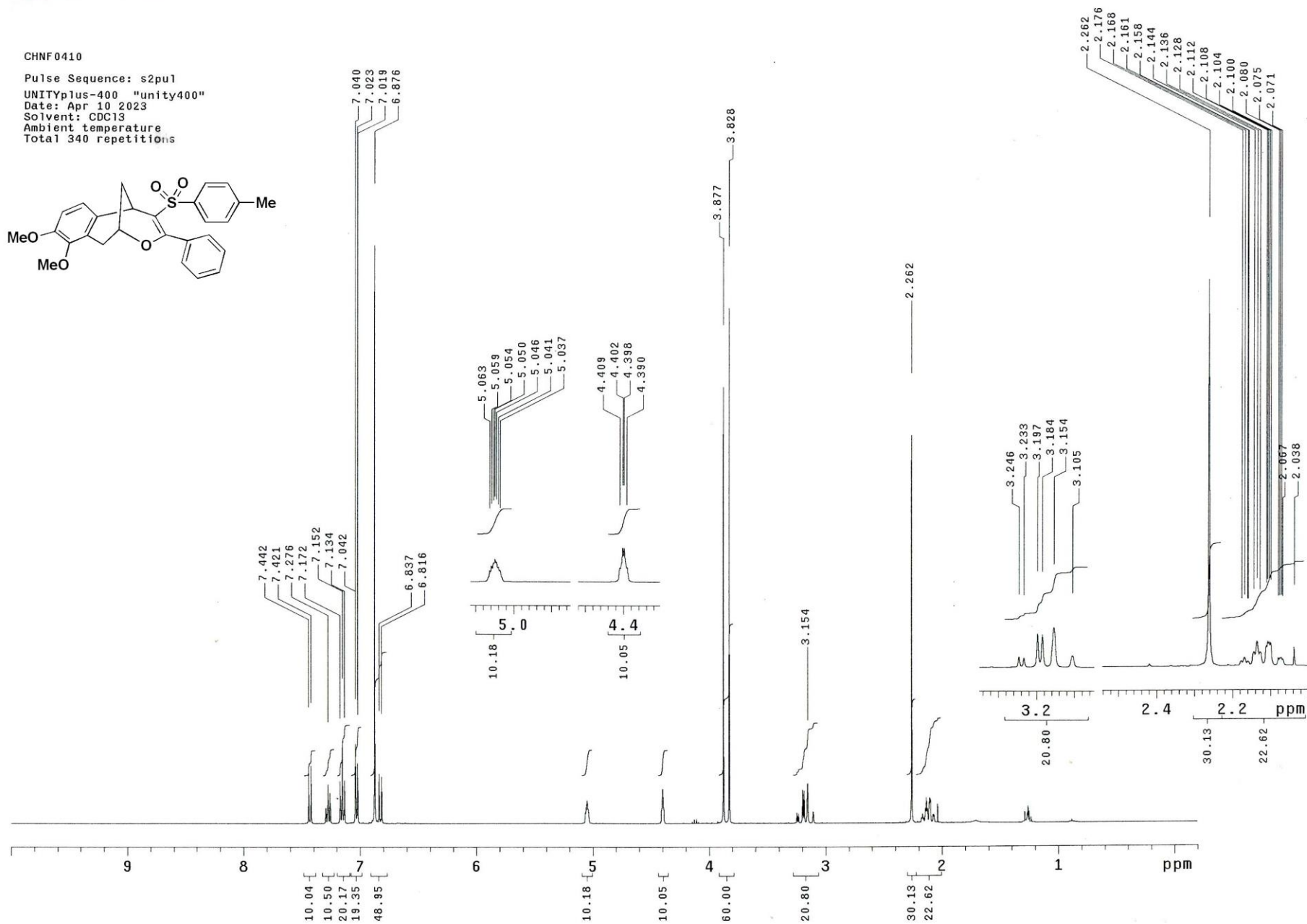
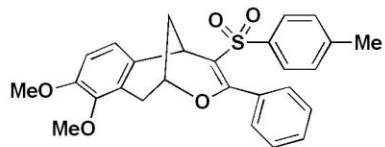
YNB1202

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 2 2022
Solvent: CDCl₃
Ambient temperature
Total 720 repetitions



Compound 6x (¹H-NMR spectral data)

CHNF0410
 Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Apr 10 2023
 Solvent: CDCl3
 Ambient temperature
 Total 340 repetitions



Compound 6x (¹³C-NMR spectral data)

CHNF0410

Pulse Sequence: s2pu1

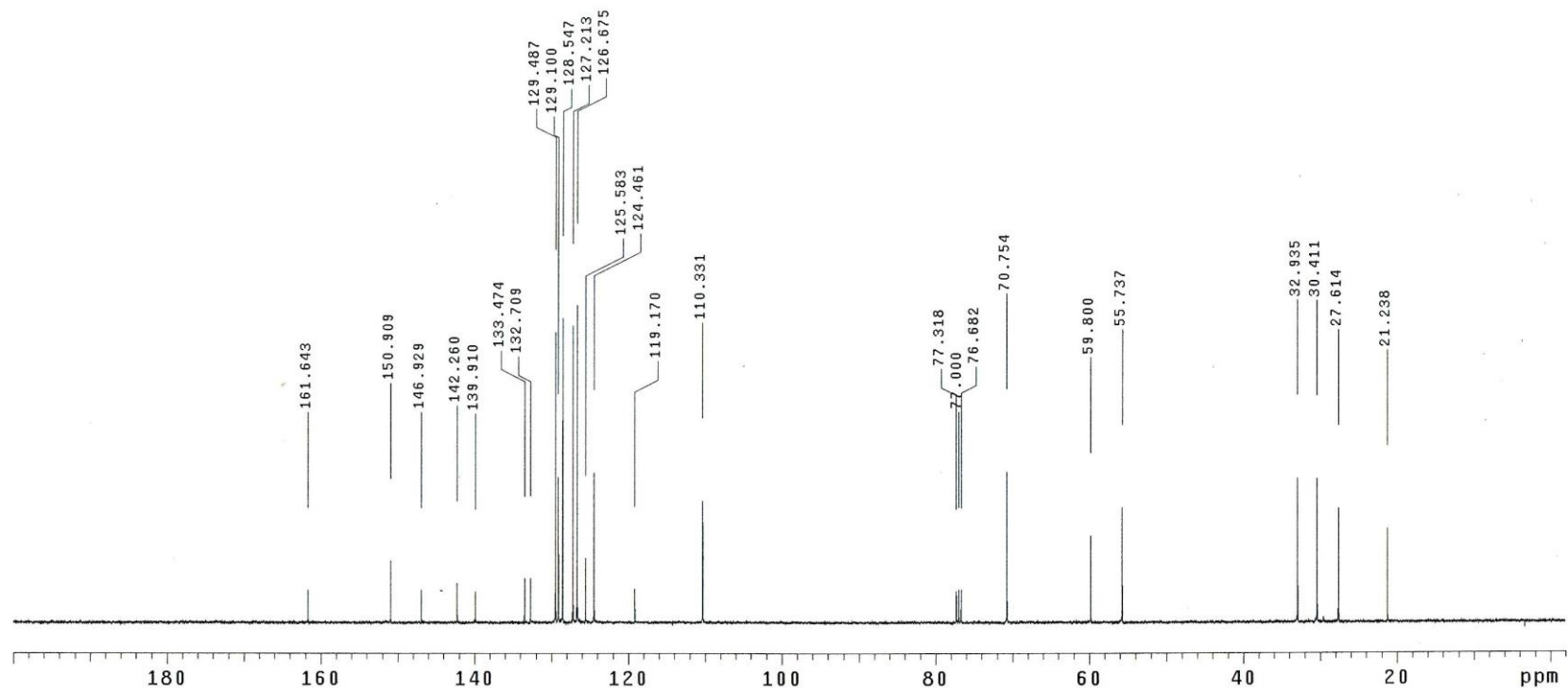
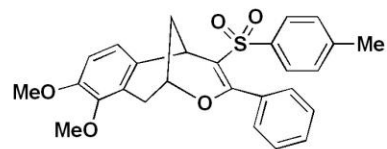
UNITYplus-400 "unity400"

Date: Sep 21 2023

Solvent: CDCl₃

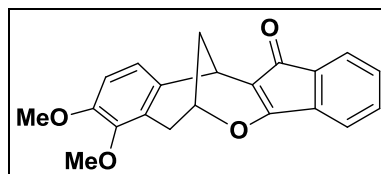
Ambient temperature

Total 4512 repetitions



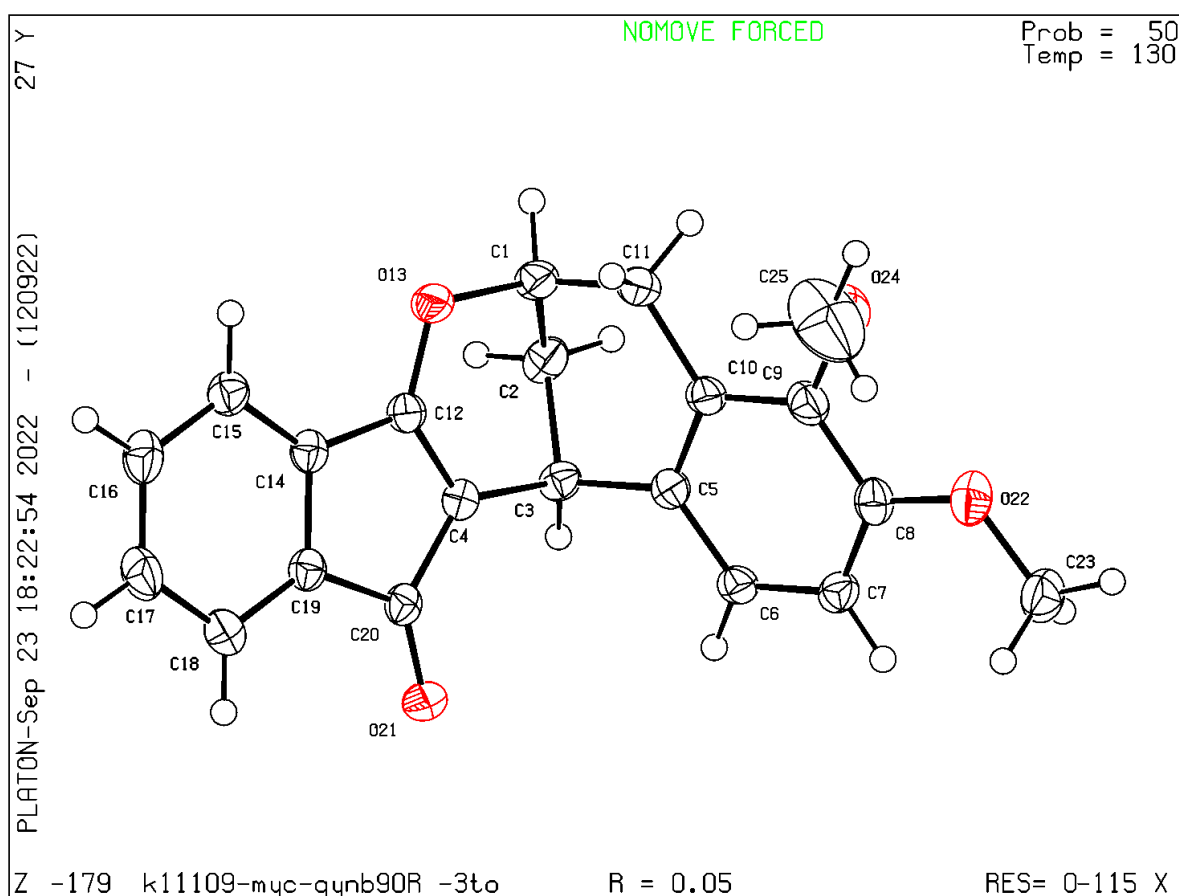
Empirical formula	C ₁₈ H ₂₀ O ₄
Formula weight	298.35
Temperature/K	130(2)
Crystal system	monoclinic
Space group	P2 ₁ /n
a/Å	7.7589(4)
b/Å	10.0726(7)
c/Å	18.9645(10)
α/°	90
β/°	95.207(5)
γ/°	90
Volume/Å ³	1476.00(15)
Z	4
ρ _{calc} /cm ³	1.343
μ/mm ⁻¹	0.091
F(000)	636.0
Crystal size/mm ³	0.5 × 0.5 × 0.5
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection/°	4.314 to 54.134
Index ranges	-9 ≤ h ≤ 9, -12 ≤ k ≤ 12, -24 ≤ l ≤ 23
Reflections collected	23702
Independent reflections	3082 [R _{int} = 0.0789, R _{sigma} = 0.0539]
Data/restraints/parameters	3082/0/201
Goodness-of-fit on F ²	1.078
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0718, wR ₂ = 0.1837
Final R indexes [all data]	R ₁ = 0.0845, wR ₂ = 0.1897
Largest diff. peak/hole / e Å ⁻³	0.42/-0.28

X-ray crystal data of compound 6r



Sample preparation : A solution of compound **6r** (30 mg) in CH₂Cl₂ (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

Crystal measurement : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



Empirical formula	C ₂₁ H ₁₈ O ₄
Formula weight	334.35
Temperature/K	130(2)
Crystal system	trigonal
Space group	R-3
a/Å	36.9306(9)
b/Å	36.9306(9)
c/Å	6.4911(2)
α/°	90
β/°	90
γ/°	120
Volume/Å ³	7666.9(4)
Z	18
ρ _{calc} /cm ³	1.303
μ/mm ⁻¹	0.090
F(000)	3168.0
Crystal size/mm ³	0.4 × 0.2 × 0.2
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	3.82 to 49.98
Index ranges	-43 ≤ h ≤ 43, -43 ≤ k ≤ 43, -7 ≤ l ≤ 7
Reflections collected	51392
Independent reflections	3005 [R _{int} = 0.0960, R _{sigma} = 0.0329]
Data/restraints/parameters	3005/126/228
Goodness-of-fit on F ²	1.066
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0466, wR ₂ = 0.1258
Final R indexes [all data]	R ₁ = 0.0550, wR ₂ = 0.1311
Largest diff. peak/hole / e Å ⁻³	0.79/-0.36

Empirical formula	C ₂₁ H ₁₉ NO ₃
Formula weight	333.37
Temperature/K	130(2)
Crystal system	triclinic
Space group	P-1
a/Å	8.3860(2)
b/Å	8.6370(2)
c/Å	12.3414(3)
α/°	103.452(2)
β/°	95.930(2)
γ/°	103.910(2)
Volume/Å ³	831.78(4)
Z	2
ρ _{calc} /cm ³	1.331
μ/mm ⁻¹	0.089
F(000)	352.0
Crystal size/mm ³	0.3 × 0.2 × 0.2
Radiation	Mo Kα (λ = 0.71073)
2θ range for data collection/°	3.442 to 54.116
Index ranges	-10 ≤ h ≤ 10, -11 ≤ k ≤ 10, -15 ≤ l ≤ 15
Reflections collected	18919
Independent reflections	3472 [R _{int} = 0.0350, R _{sigma} = 0.0276]
Data/restraints/parameters	3472/0/228
Goodness-of-fit on F ²	1.106
Final R indexes [I ≥ 2σ (I)]	R ₁ = 0.0403, wR ₂ = 0.1022
Final R indexes [all data]	R ₁ = 0.0500, wR ₂ = 0.1077
Largest diff. peak/hole / e Å ⁻³	0.20/-0.23