

Supporting Information

Bi(OTf)₃-Mediated Tandem Annulation of 1-Aryl Isochroman-3-ones with Oxygenated Arenes. One-Pot Synthesis of Polyoxygenated Homotriptycenes

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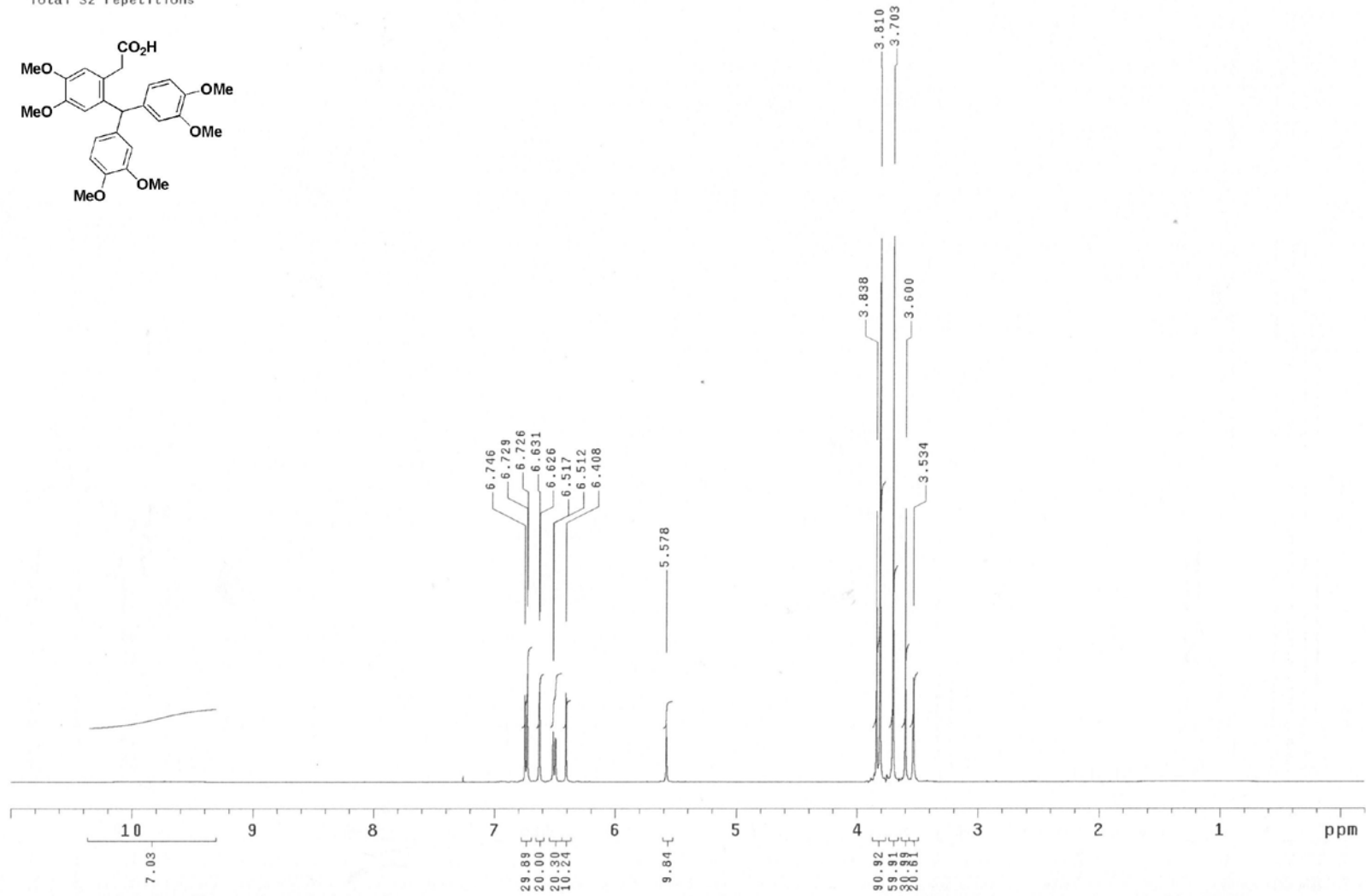
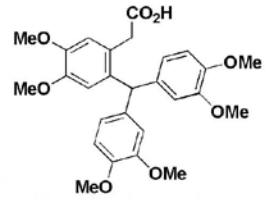
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Compound 4a (¹H-NMR spectral data)

SHBF3

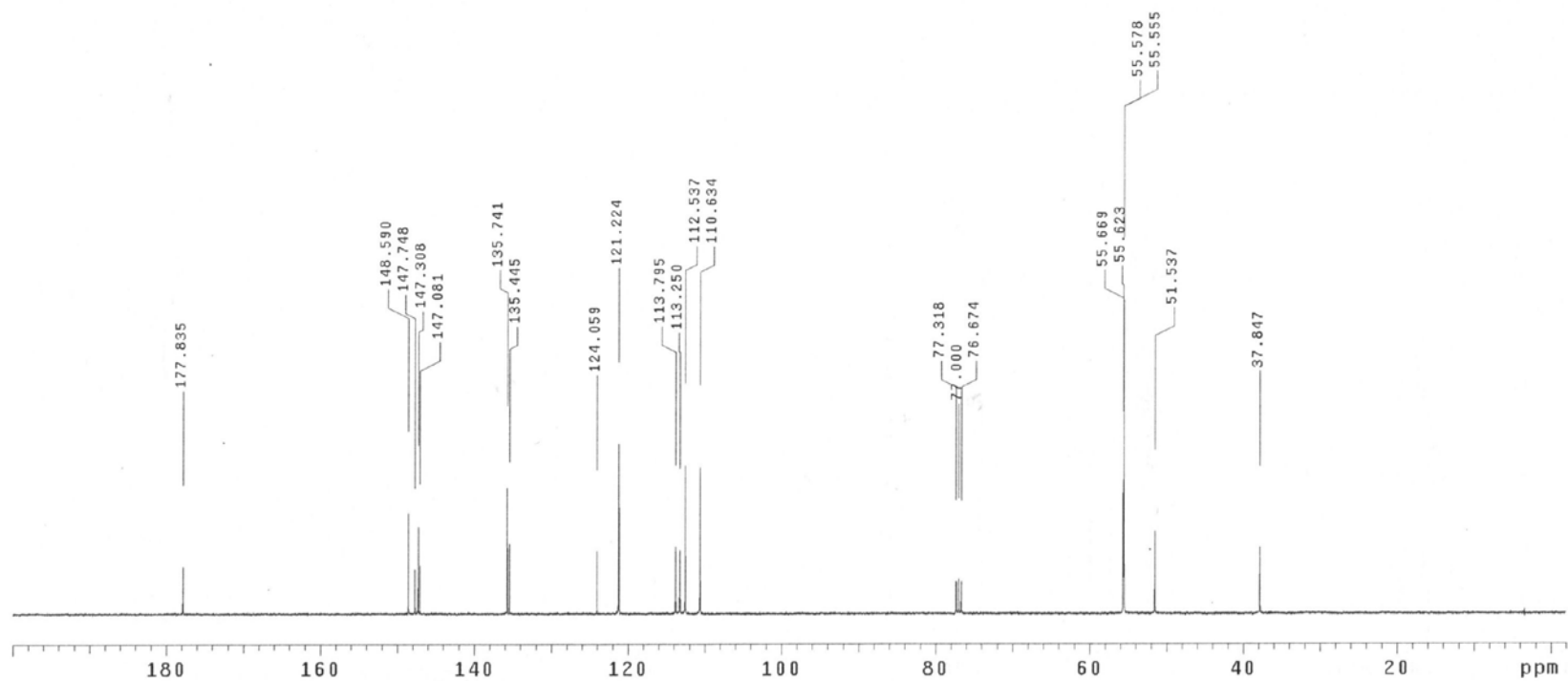
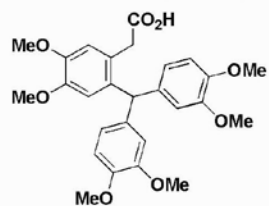
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UNITYplus-400 "unity400"
Date: Nov 15 2018
Solvent: CDC13
Ambient temperature
Total 32 repetitions



Compound 4a (¹³C-NMR spectral data)

SHBF3

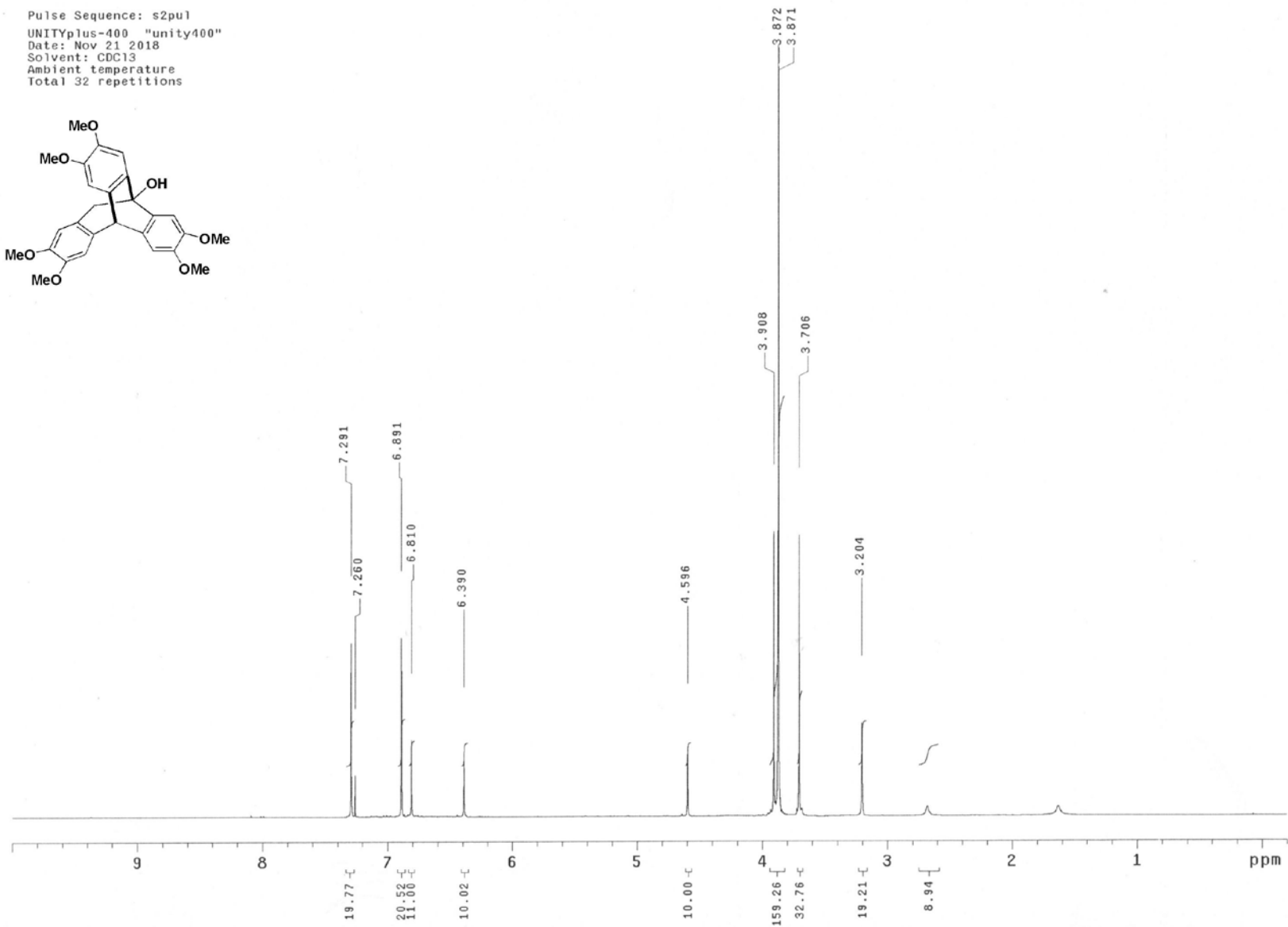
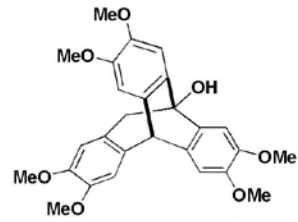
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Nov 15 2018
Solvent: CDCl3
Ambient temperature
Total 480 repetitions



Compound 5a (¹H-NMR spectral data)

SH6760Me6

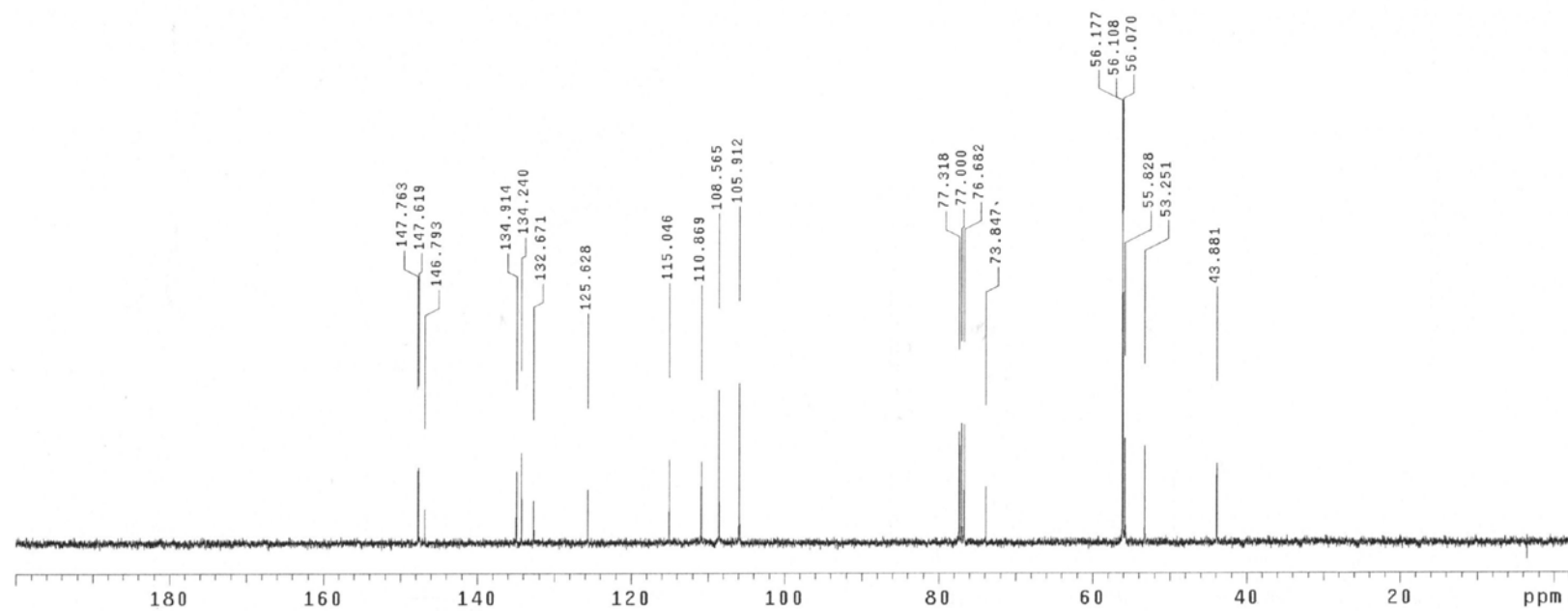
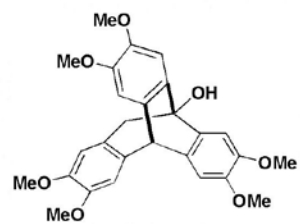
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Nov 21 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 5a (¹³C-NMR spectral data)

SH6760Me6

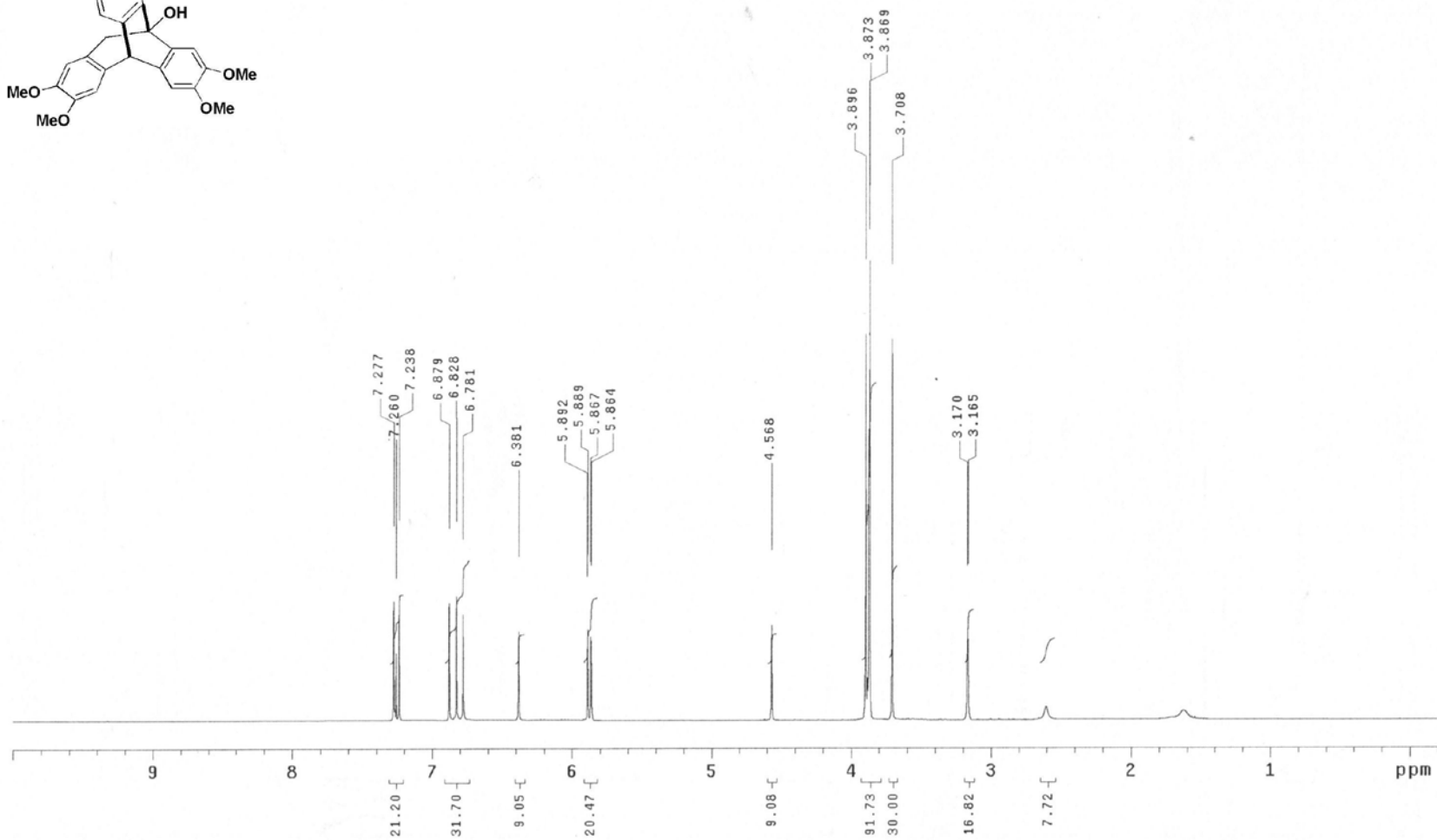
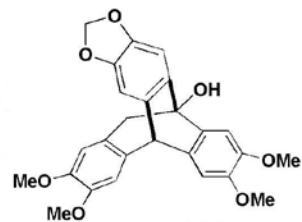
Pulse Sequence: s2pu1
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Date: Nov 21 2018
Solvent: CDCl₃
Ambient temperature
Total 1056 repetitions



Compound 5b (¹H-NMR spectral data)

SH[3.2.2]5b

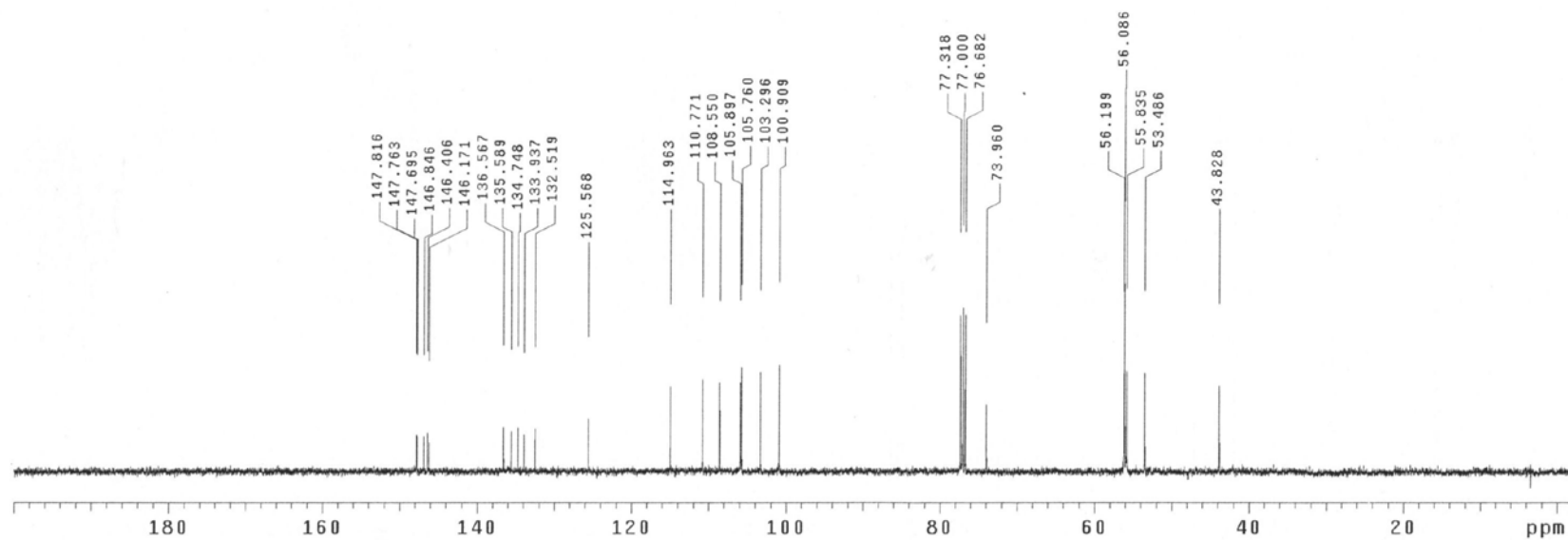
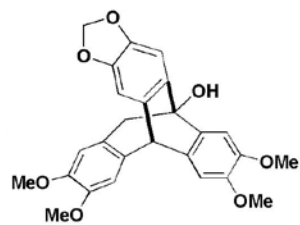
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 13 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 5b (¹³C-NMR spectral data)

SH[3.2.2]5b

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 13 2018
Solvent: CDC13
Ambient temperature
Total 1648 repetitions



Compound 5c (¹H-NMR spectral data)

SH[3.2.2]5C

Pulse Sequence: s2pu1

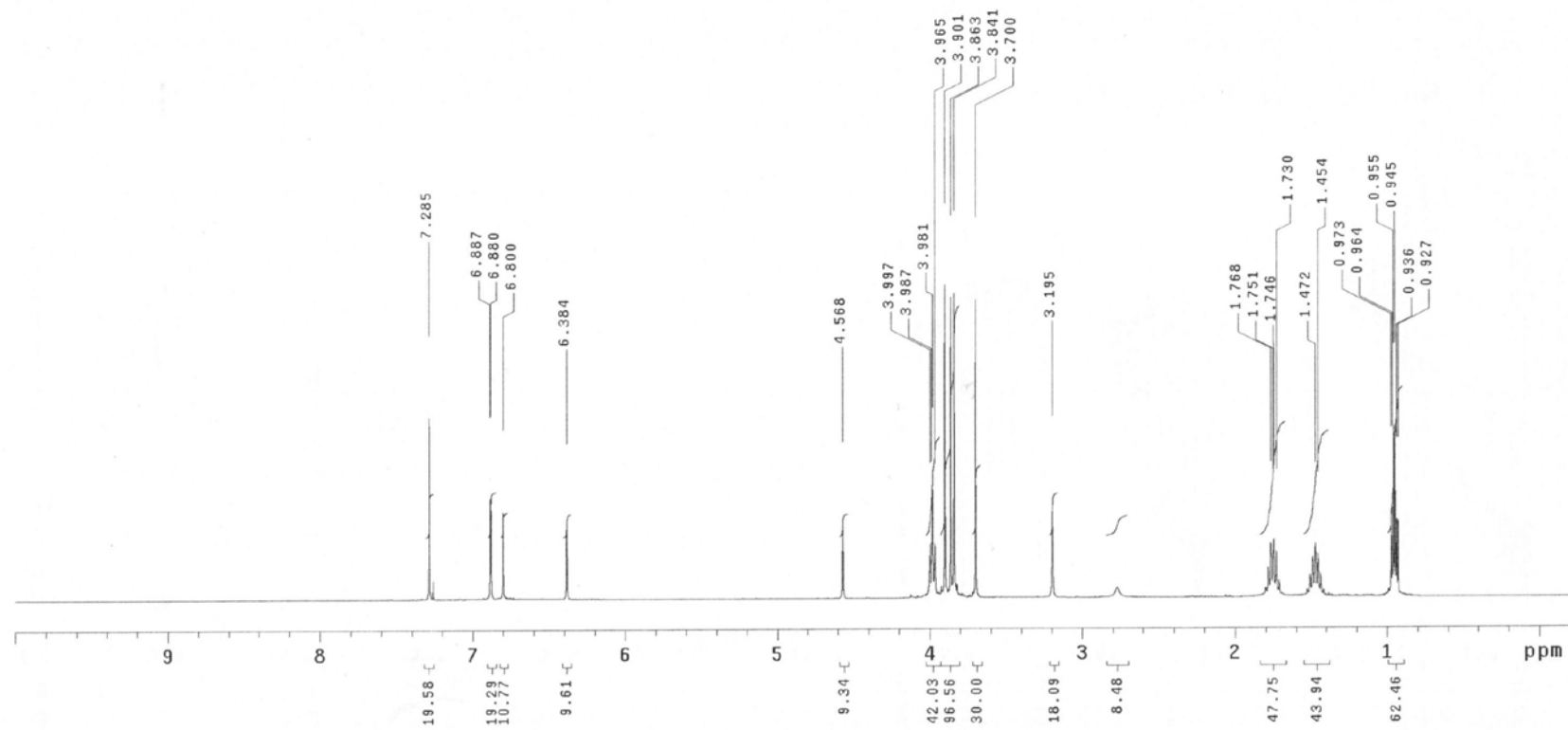
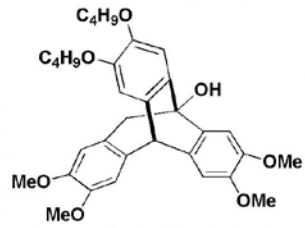
UNITYplus-400 "unity400"

Date: Dec 7 2018

Solvent: CDC13

Ambient temperature

Total 32 repetitions



Compound 5c (¹³C-NMR spectral data)

SH[3.2.2]5C

Pulse Sequence: s2pu1

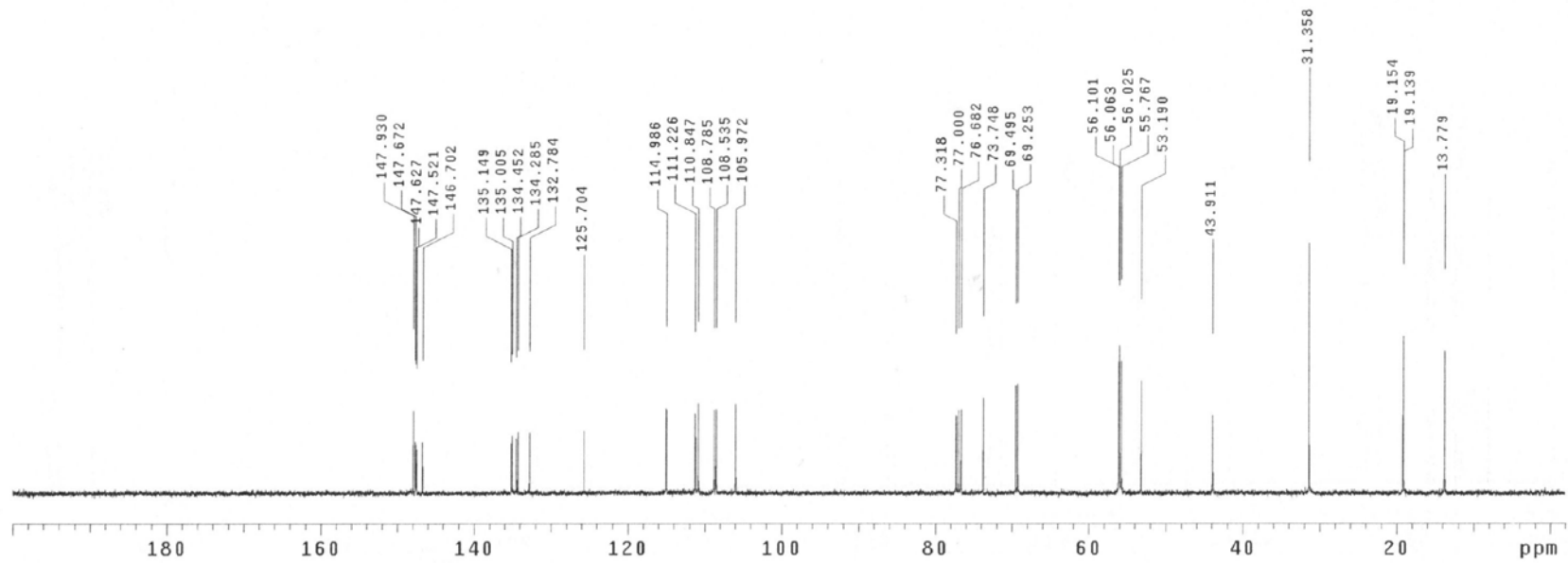
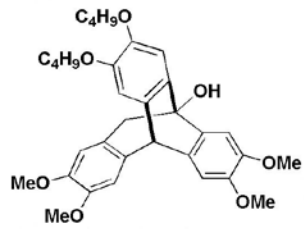
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Date: Dec 7 2018

Solvent: CDCl₃

Ambient temperature

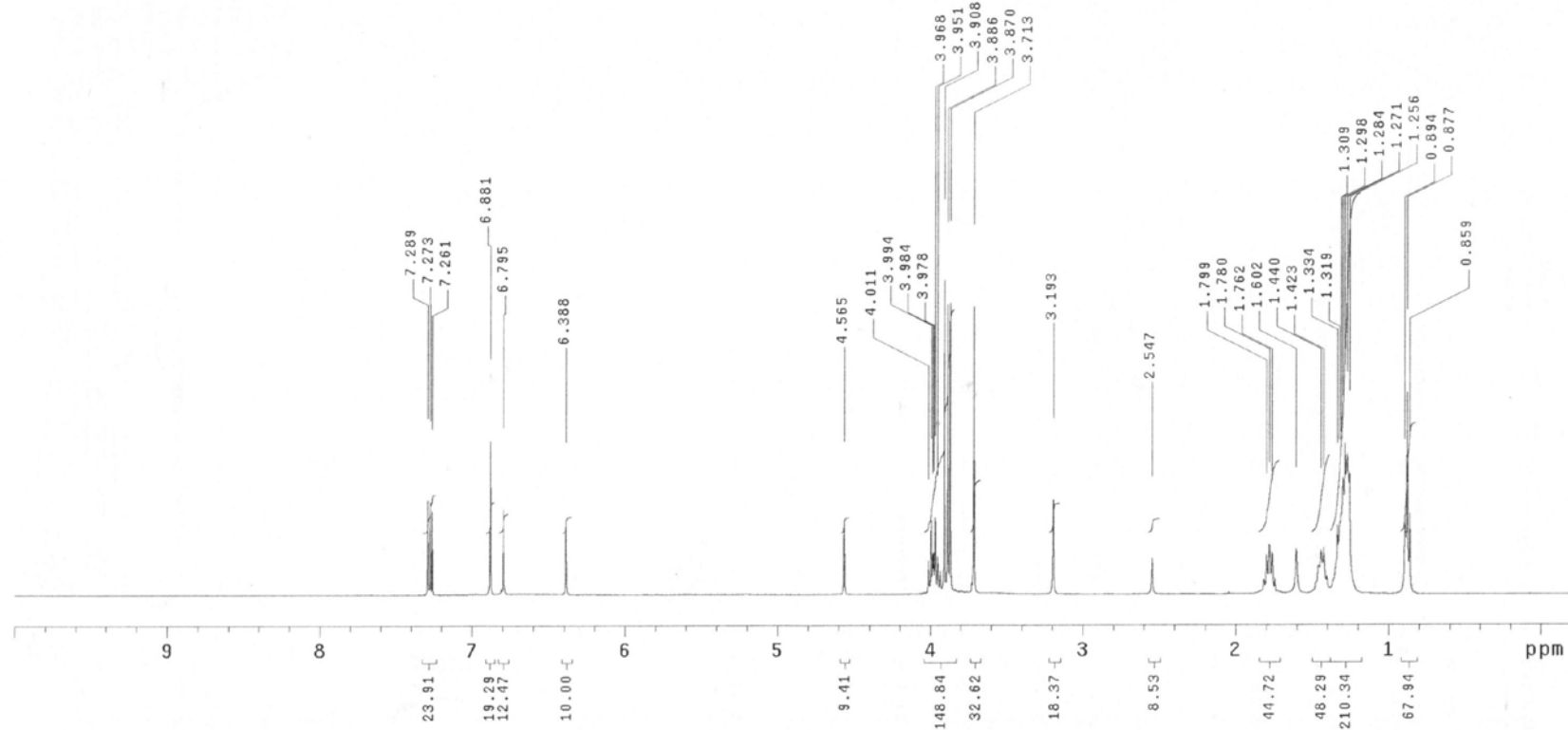
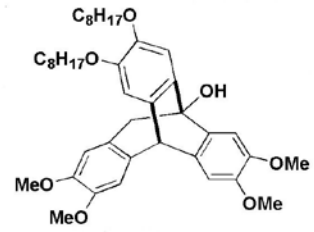
Total 832 repetitions



Compound 5d (¹H-NMR spectral data)

SH[3.2.2]5d

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 9 2019
Solvent: CDC13
Ambient temperature
Total 64 repetitions



Compound 5d (¹³C-NMR spectral data)

SH[3.2.2]5d

Pulse Sequence: s2pu1

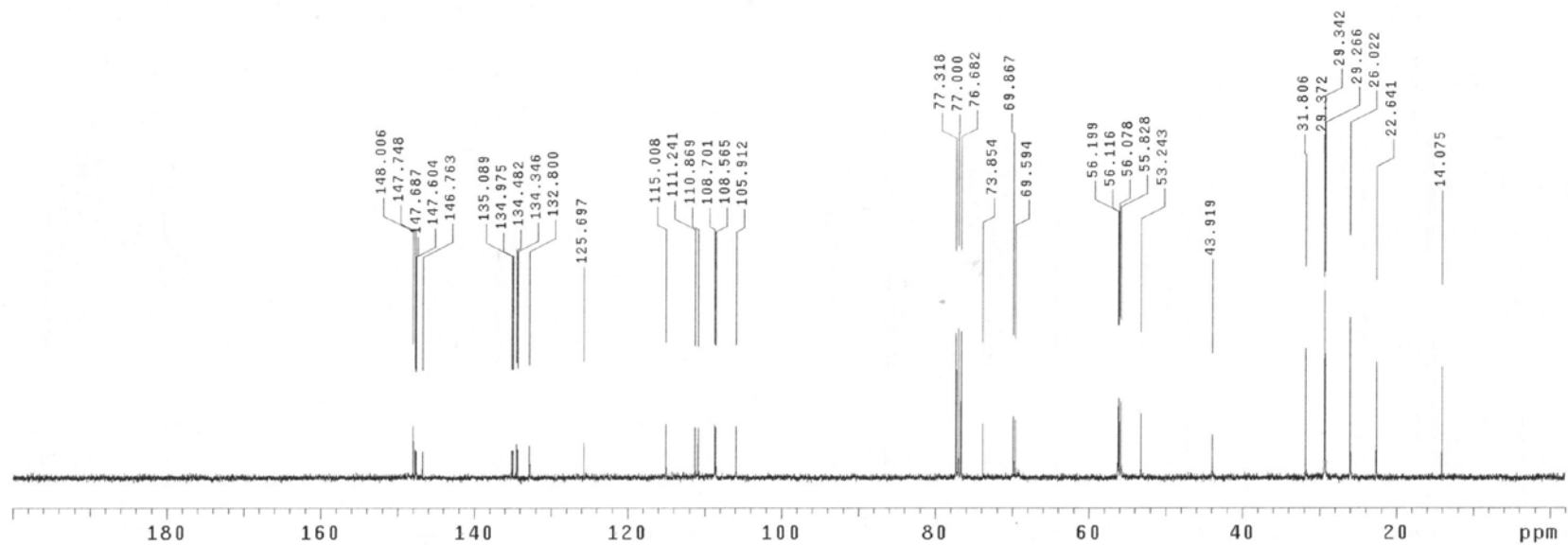
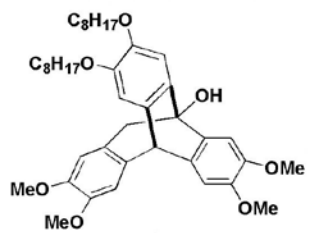
UNITYplus-400 "unity400"

Date: Jan 9 2019

Solvent: CDCl₃

Ambient temperature

Total 2480 repetitions



Compound 5e (¹H-NMR spectral data)

SH[3.2.2]5e

Pulse Sequence: s2pu1

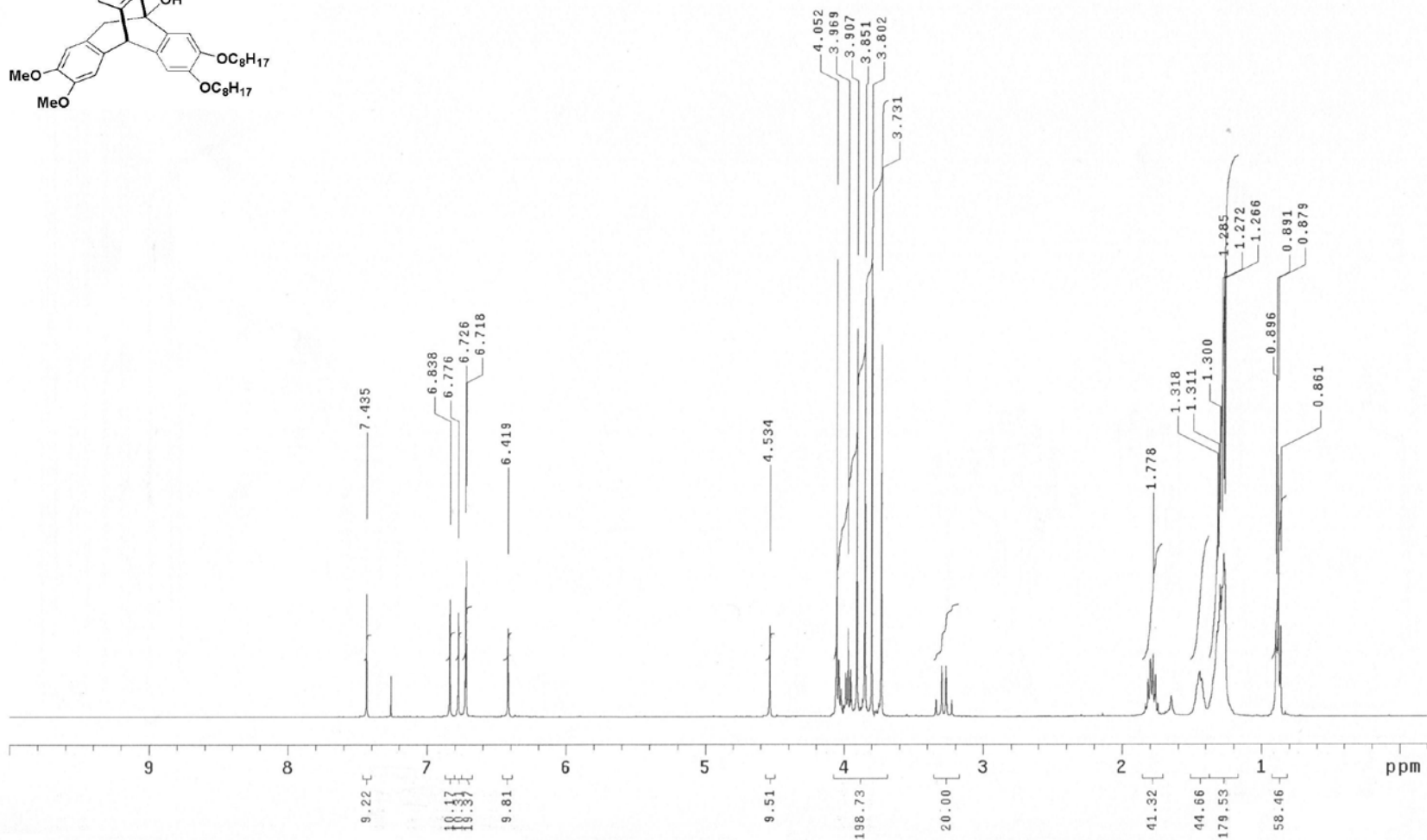
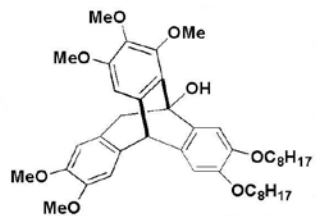
UNITYplus-400 "unity400"

Date: Apr 30 2019

Solvent: CDC13

Ambient temperature

Total 32 repetitions



Compound 5e (¹³C-NMR spectral data)

SH[3.2.2]5e

Pulse Sequence: s2pu1

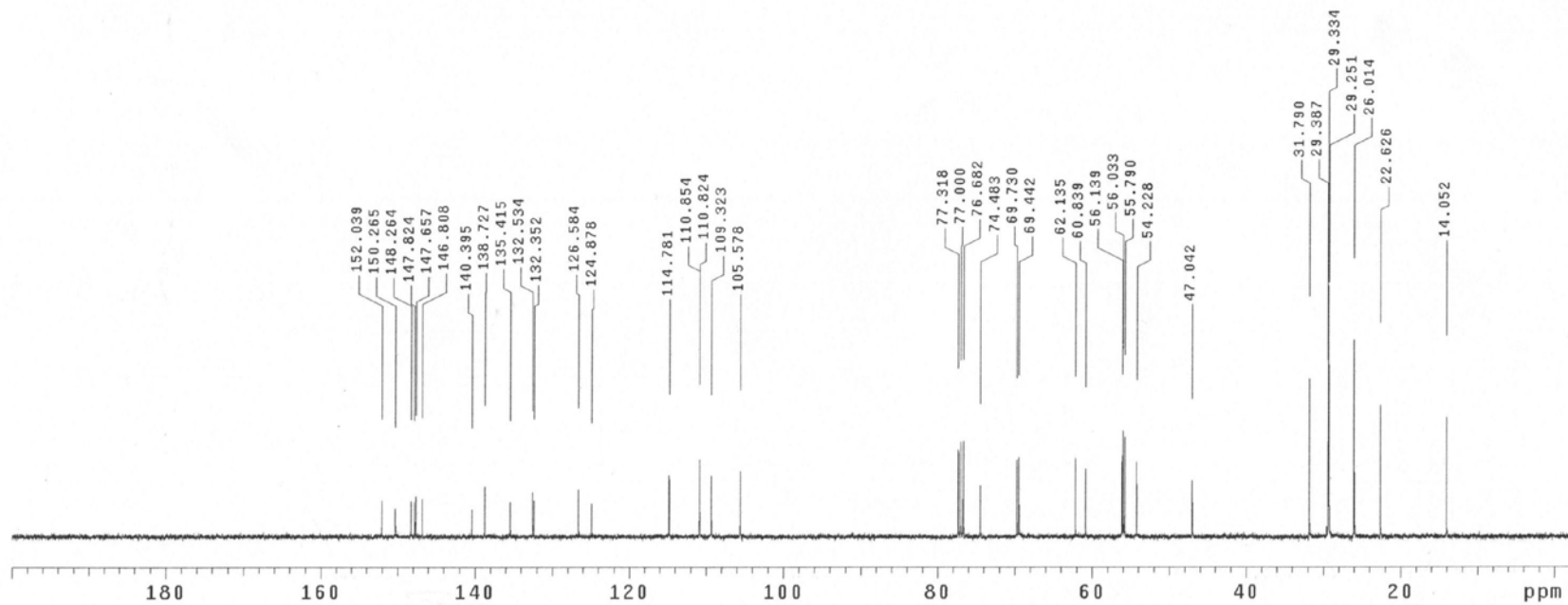
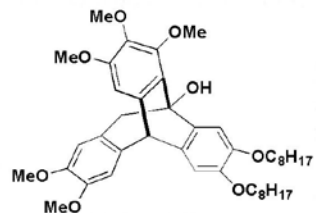
UNITYplus-400 "unity400"

Date: Apr 30 2019

Solvent: CDCl₃

Ambient temperature

Total 1040 repetitions



Compound 5f (¹H-NMR spectral data)

SH[3.2.2]5f

Pulse Sequence: s2pu1

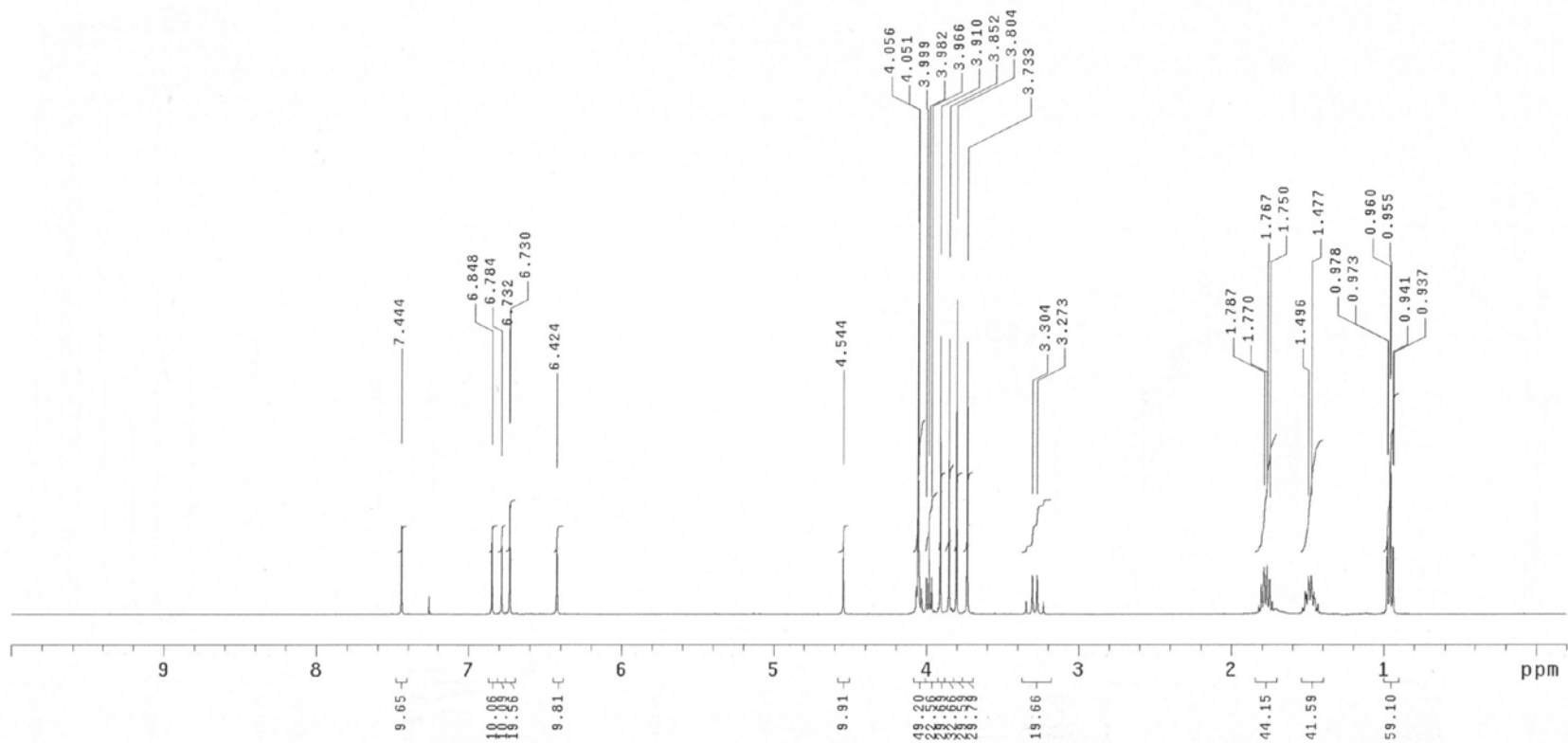
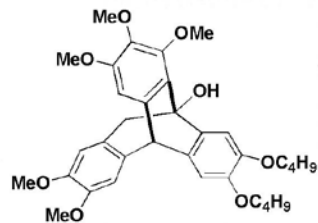
UNITYplus-400 "unity400"

Date: May 7 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5f (¹³C-NMR spectral data)

SH[3.2.2]5f

Pulse Sequence: s2pu1

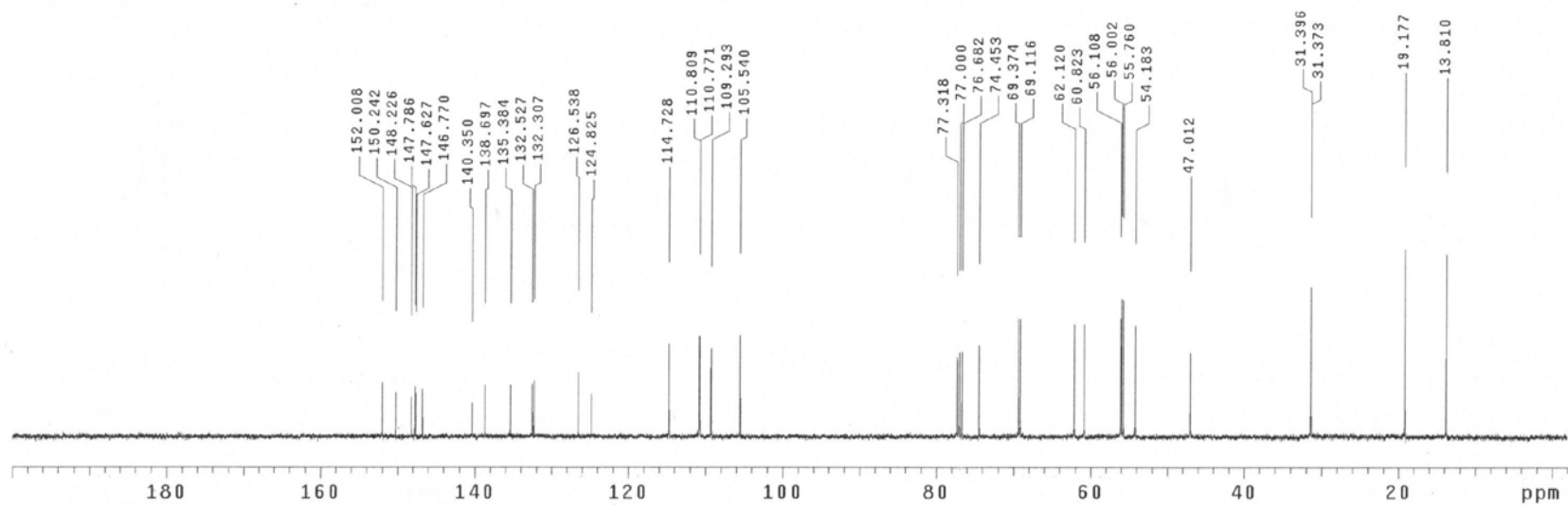
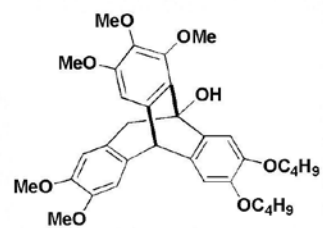
UNITYplus-400 "unity400"

Date: May 7 2019

Solvent: CDC13

Ambient temperature

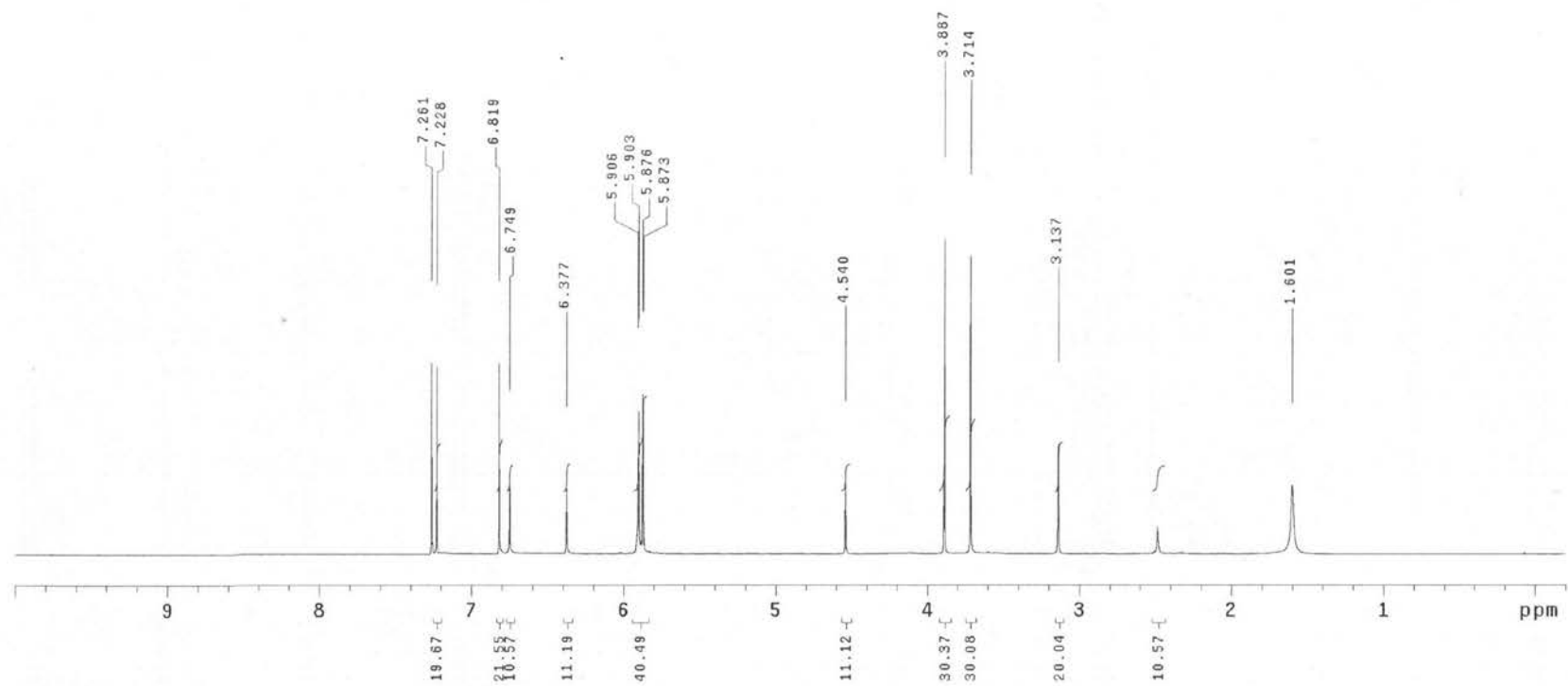
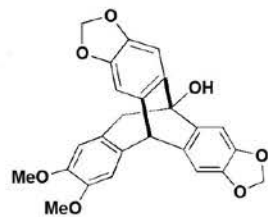
Total 848 repetitions



Compound 5g (¹H-NMR spectral data)

SH[3.2.2]5g

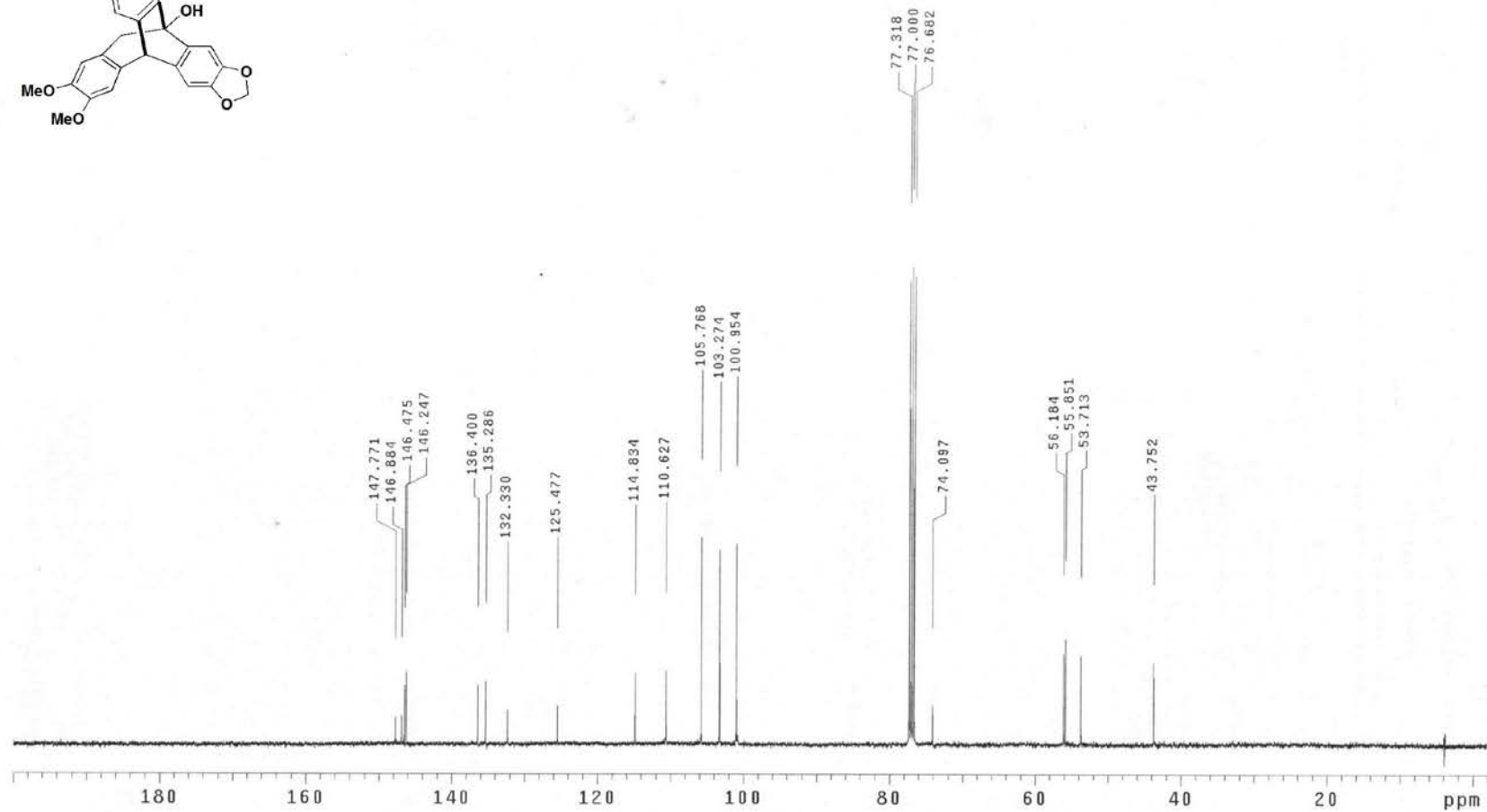
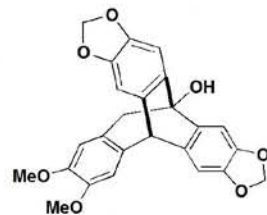
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 3 2019
Solvent: CDCl₃
Ambient temperature
Total 64 repetitions



Compound 5g (¹³C-NMR spectral data)

SH[3.2.2]5g

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 3 2019
Solvent: CDCl₃
Ambient temperature
Total 32816 repetitions



Compound 5h (¹H-NMR spectral data)

SH[3.2.2]5h

Pulse Sequence: s2pu1

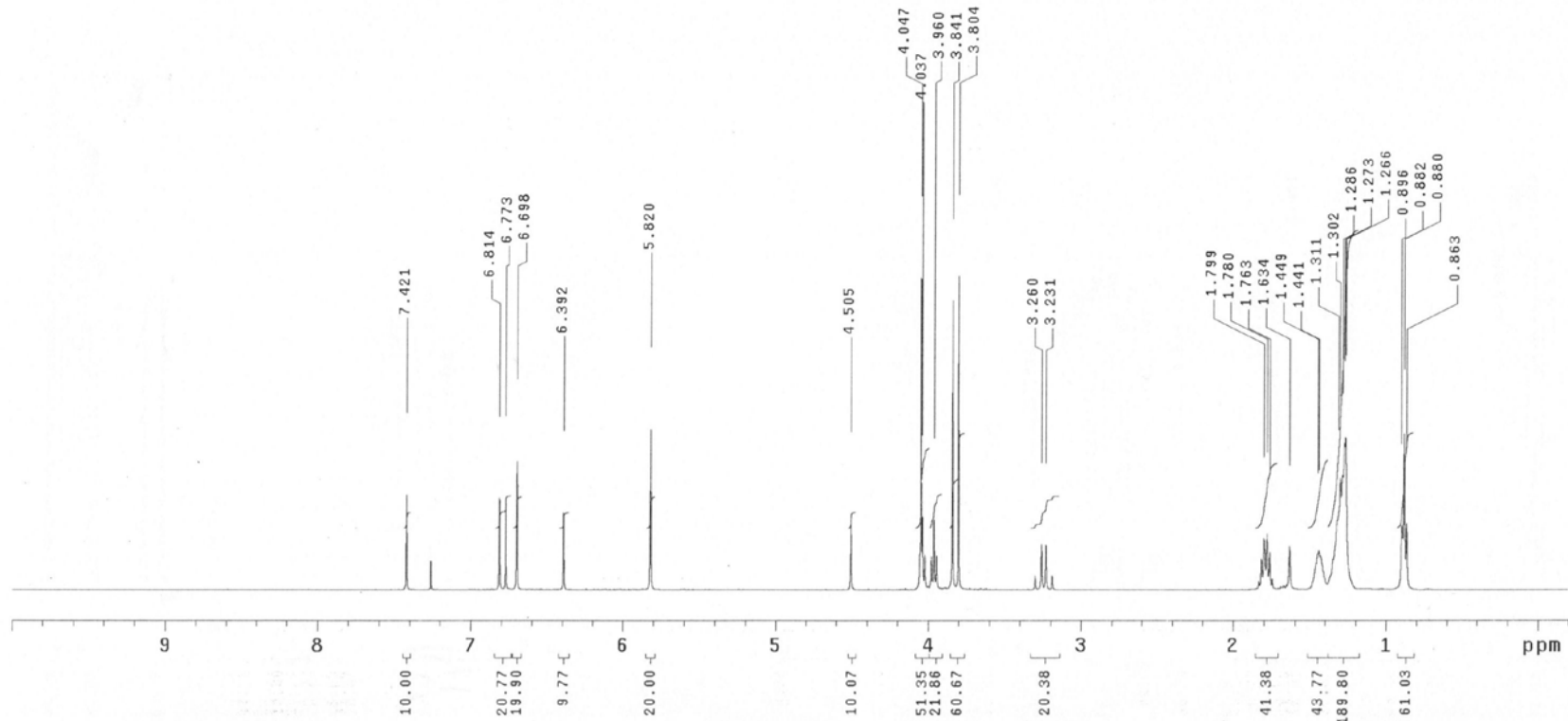
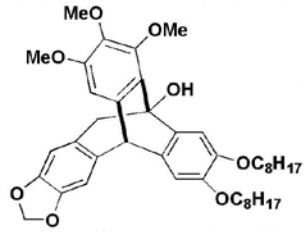
UNITYplus-400 "unity400"

Date: May 15 2019

Solvent: CDC13

Ambient temperature

Total 32 repetitions



Compound 5h (¹³C-NMR spectral data)

SH[3.2.2]5h

Pulse Sequence: s2pu1

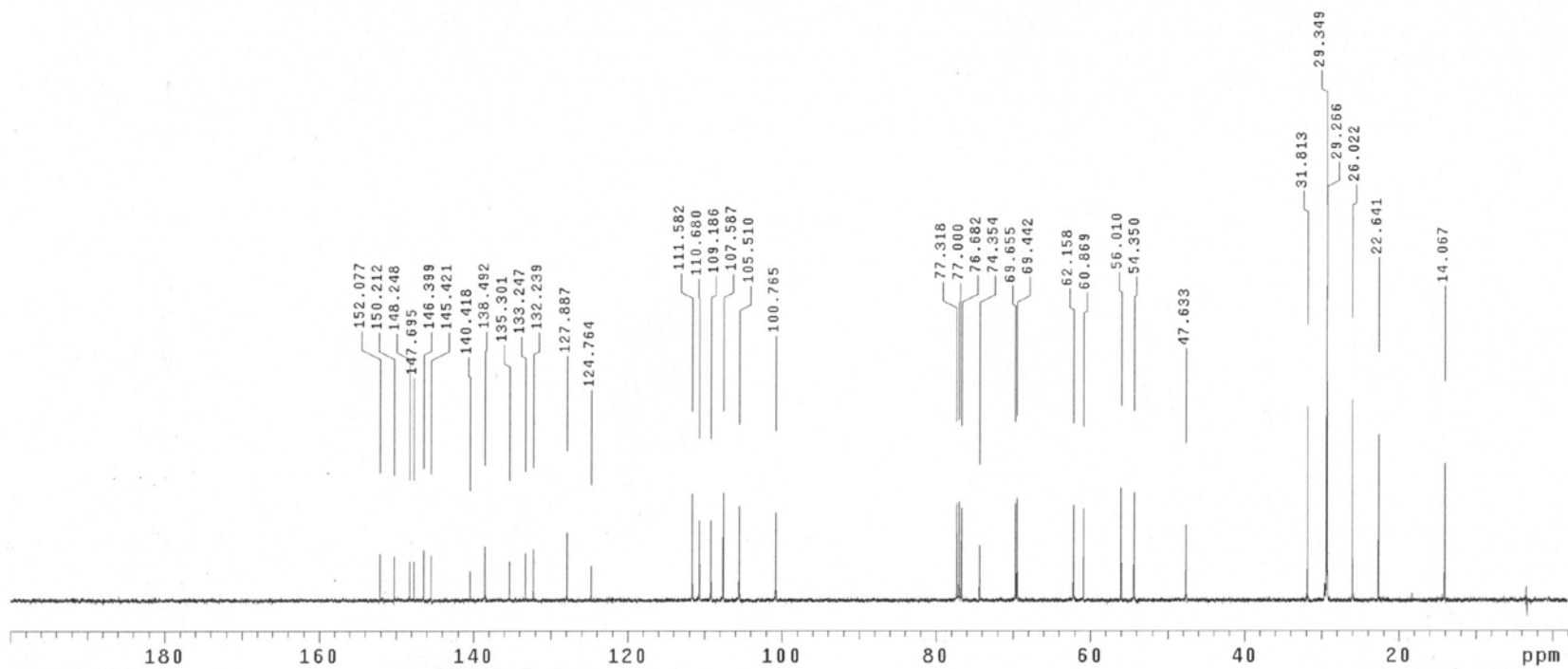
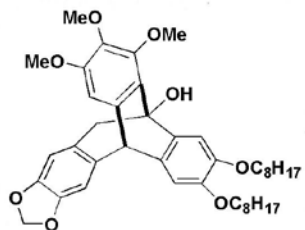
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Date: May 15 2019

Solvent: CDCl₃

Ambient temperature

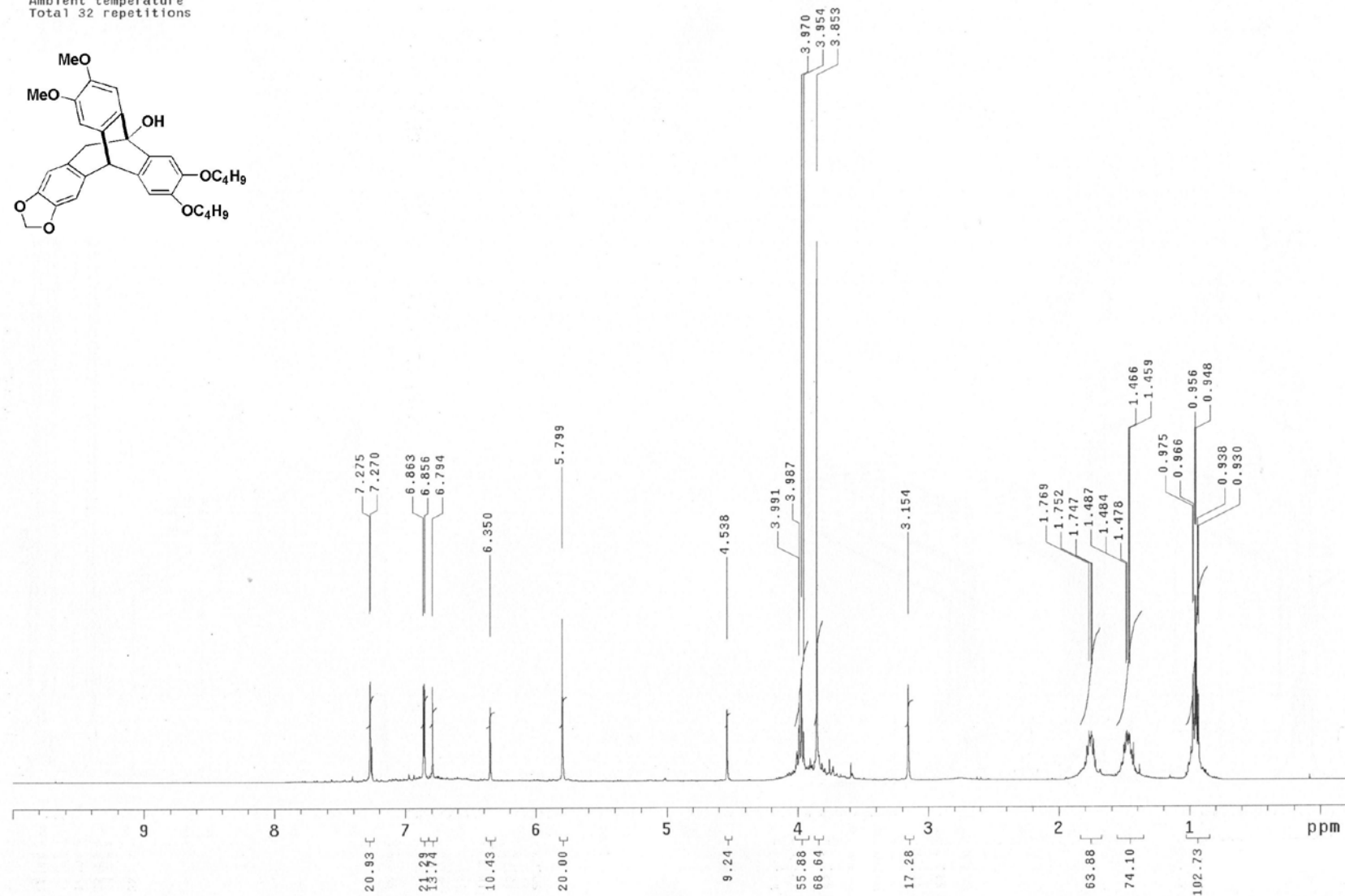
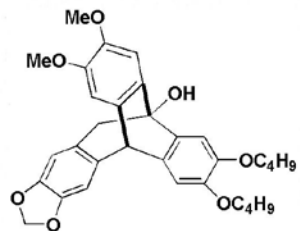
Total 3248 repetitions



Compound 5i (¹H-NMR spectral data)

SH[3.2.2]5i

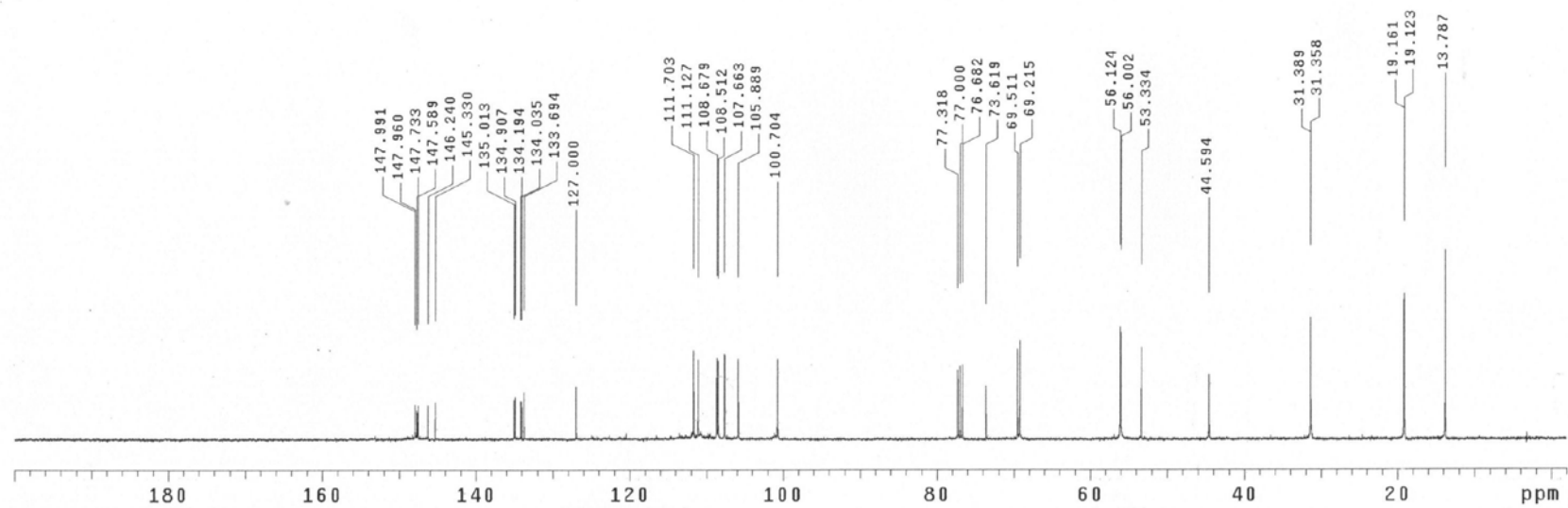
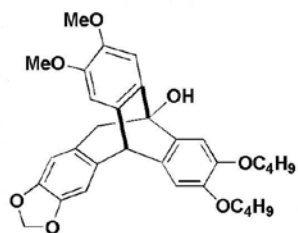
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: May 14 2019
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 5i (¹³C-NMR spectral data)

SH[3.2.2]5i

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: May 14 2019
Solvent: CDC13
Ambient temperature
Total 5360 repetitions



Compound 5j (¹H-NMR spectral data)

SH[3.2.]5j

Pulse Sequence: s2pu1

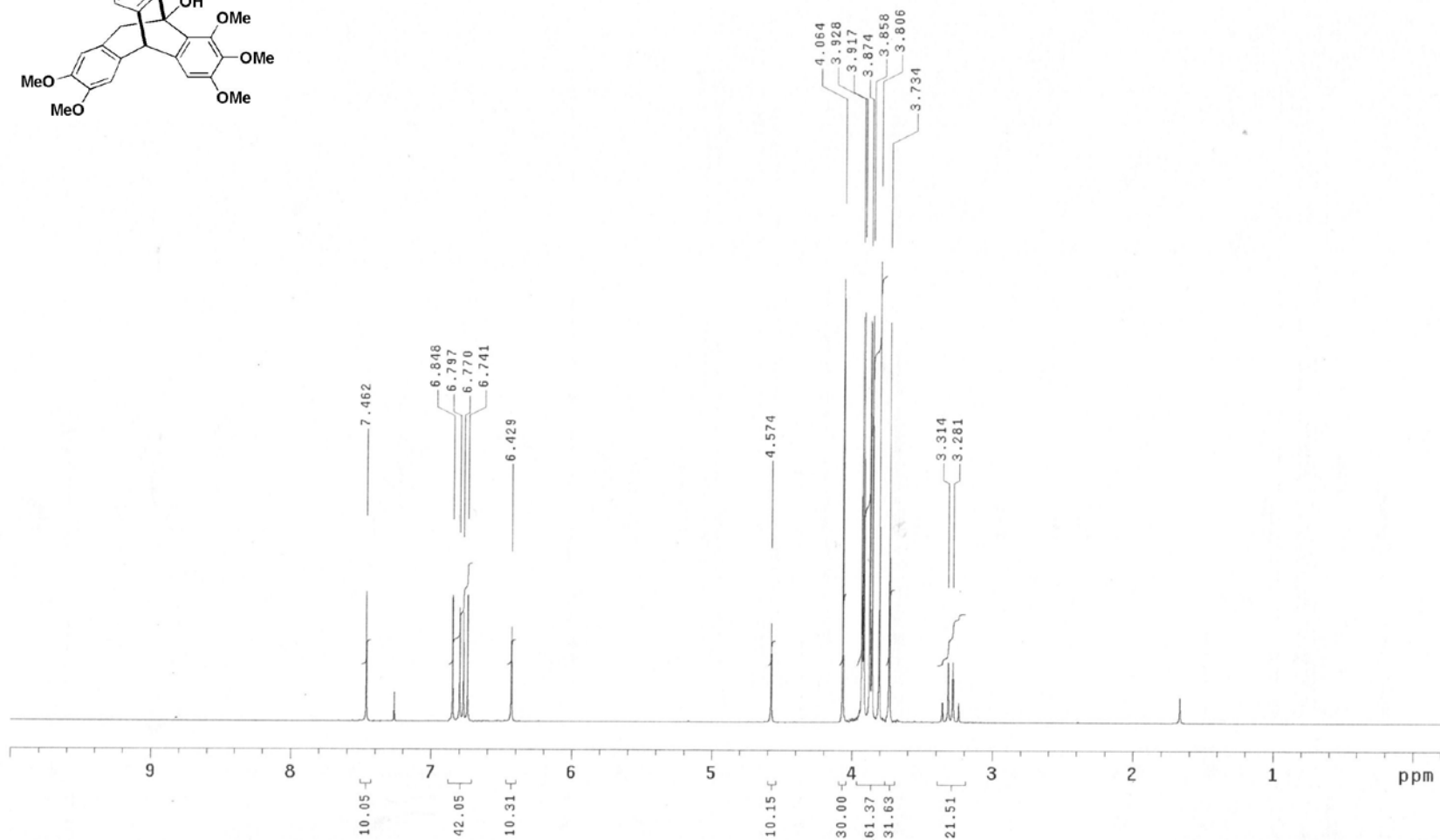
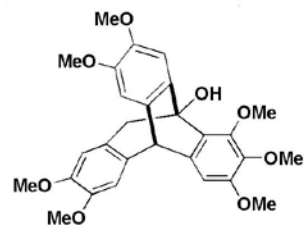
UNITYplus-400 "unity400"

Date: Jan 29 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5j (¹³C-NMR spectral data)

SH[3.2.]5J

Pulse Sequence: s2pu1

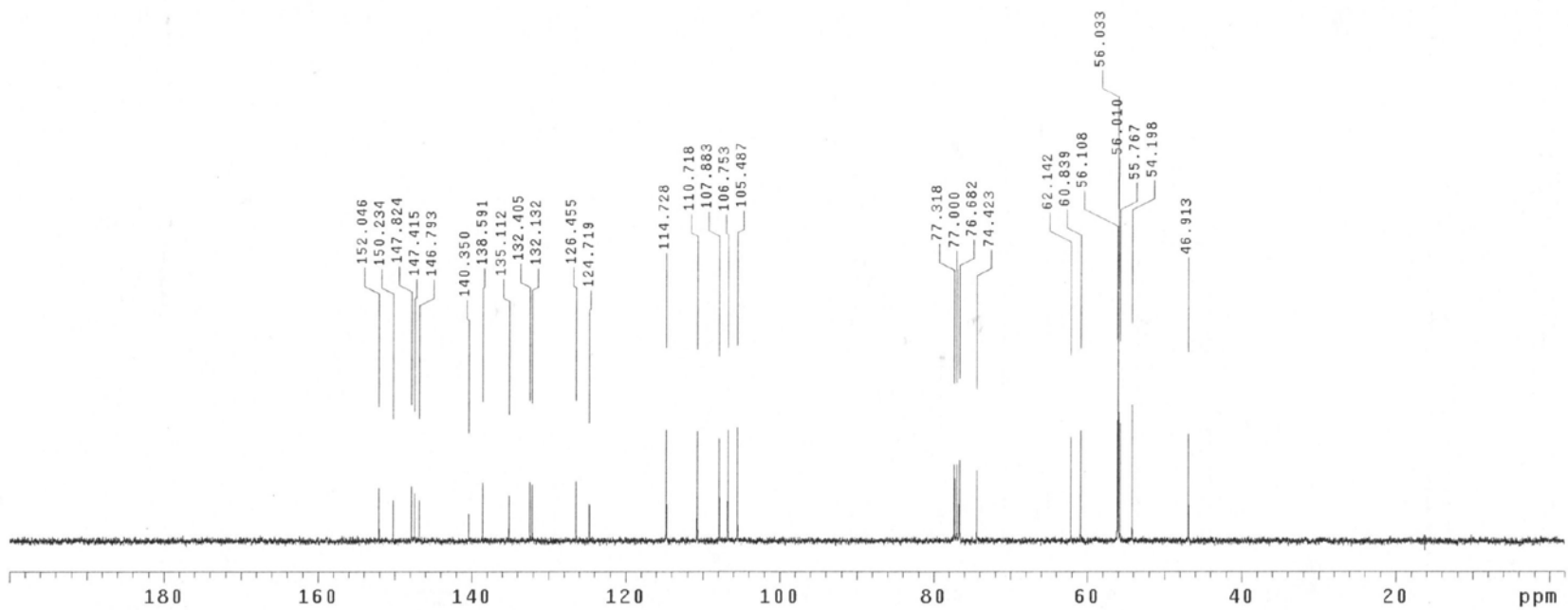
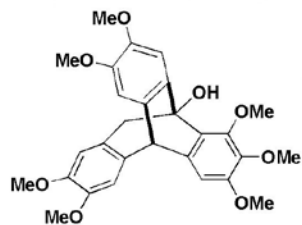
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Date: Jan 29 2019

Solvent: CDCl₃

Ambient temperature

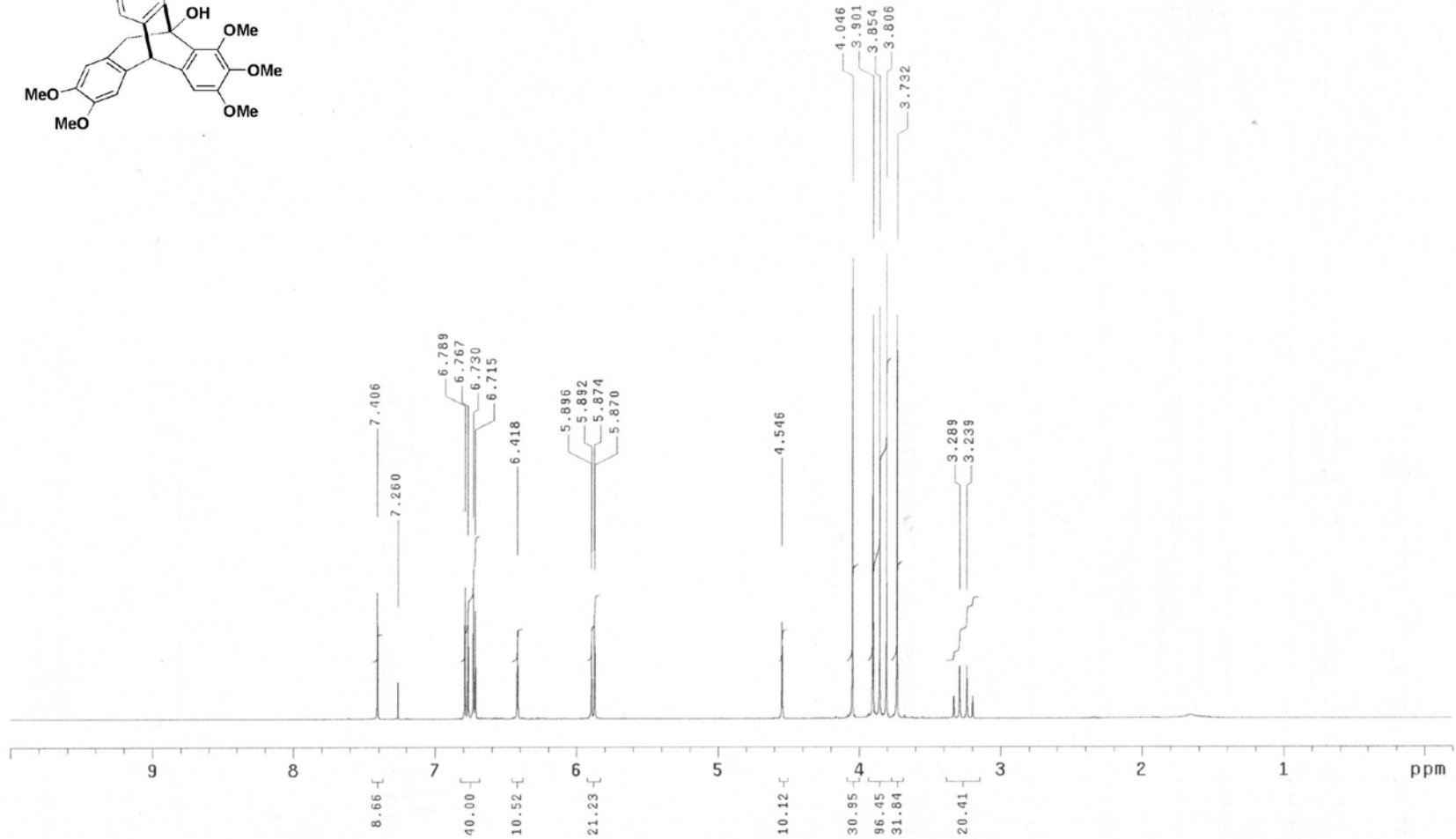
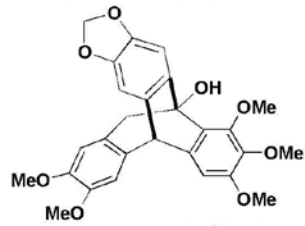
Total 768 repetitions



Compound 5k (¹H-NMR spectral data)

SH[3.2.2]5k

Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Jan 23 2019
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 5k (¹³C-NMR spectral data)

SH[3.2.2]5k

Pulse Sequence: s2pu1

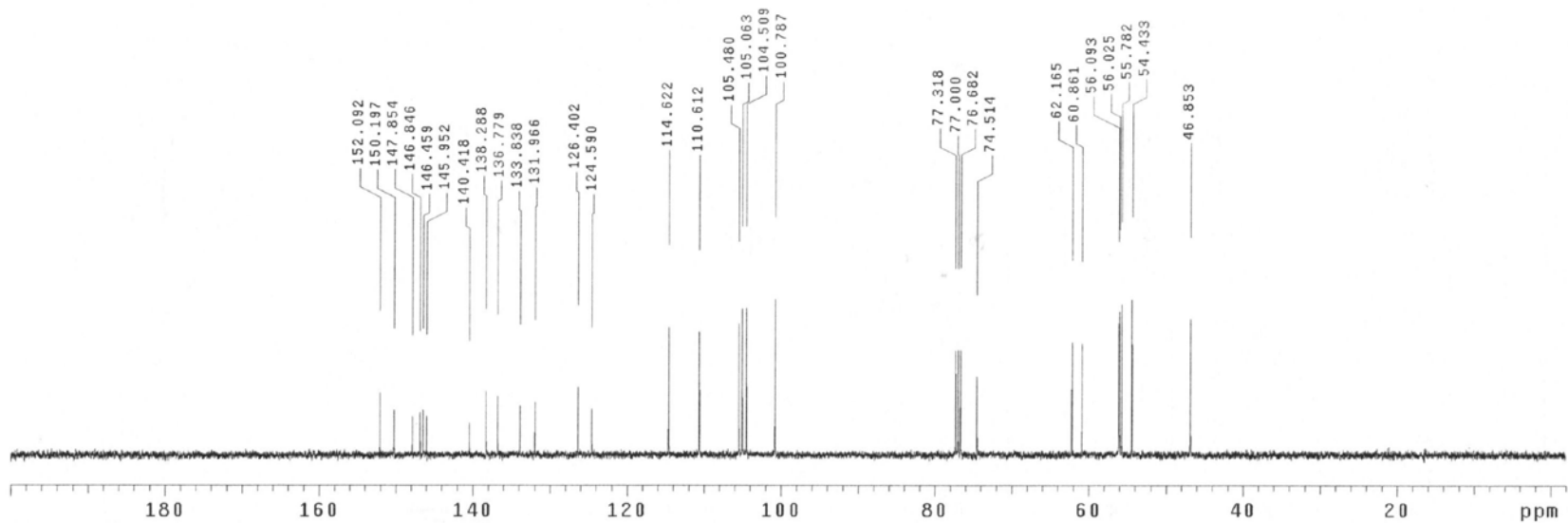
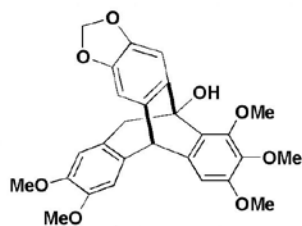
UNITYplus-400 "unity400"

Date: Jan 23 2019

Solvent: CDC13

Ambient temperature

Total 944 repetitions



Compound 5I (¹H-NMR spectral data)

SH[3.2.2]5I

Pulse Sequence: s2pu1

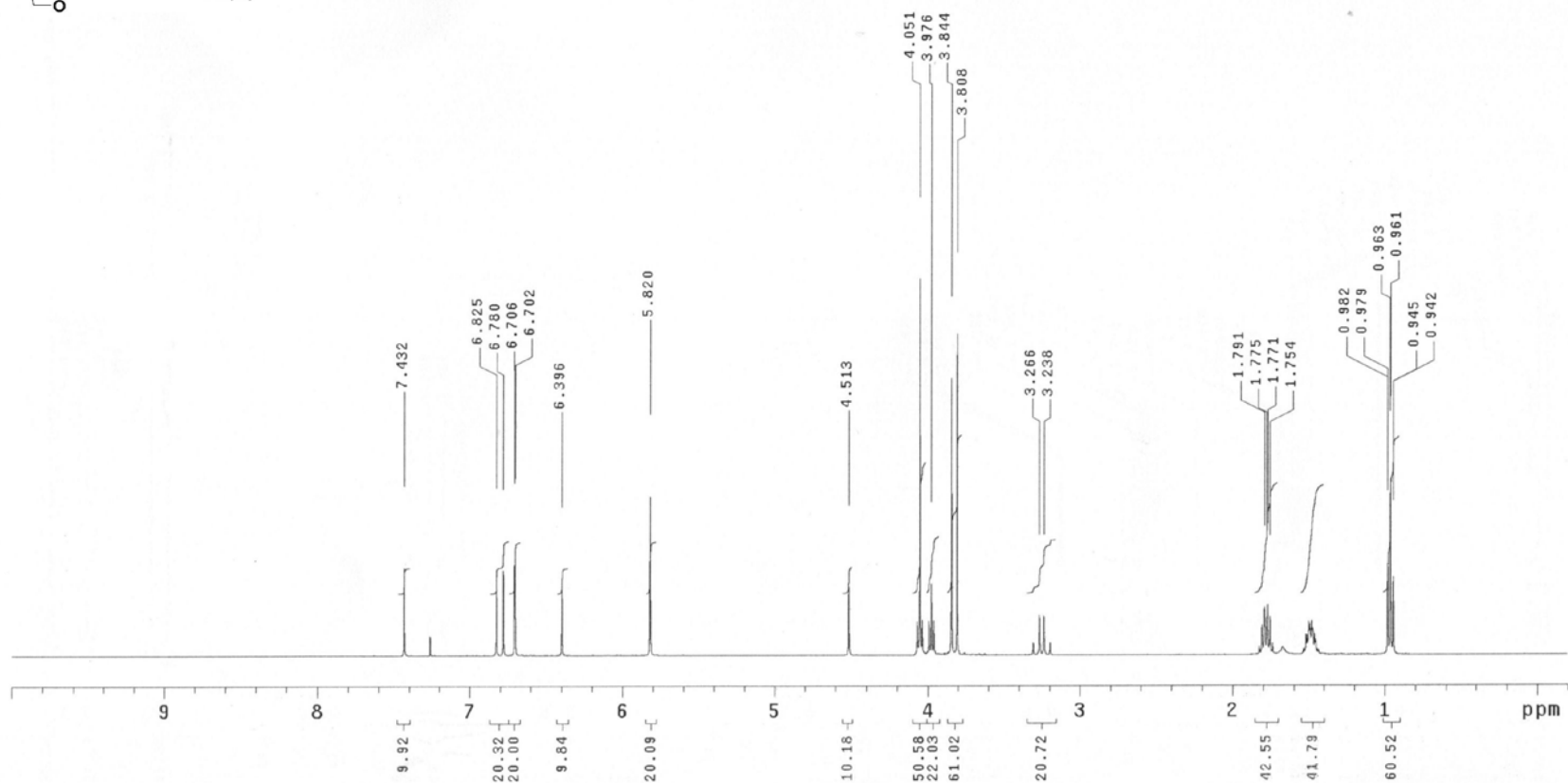
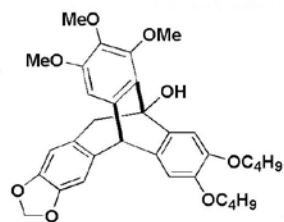
UNITYplus-400 "unity400"

Date: Apr 30 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5I (¹³C-NMR spectral data)

SH[3.2.2]5I

Pulse Sequence: s2pu1

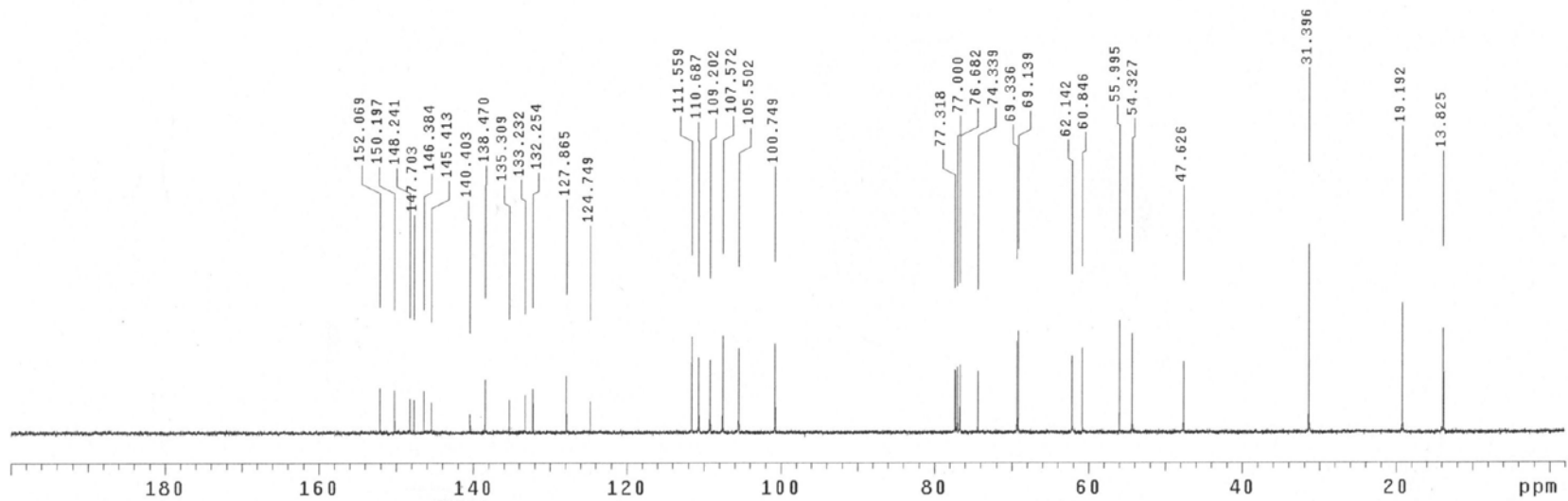
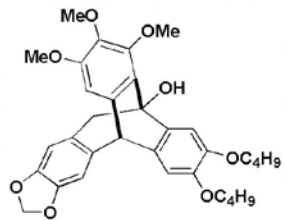
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Date: Apr 30 2019

Solvent: CDCl₃

Ambient temperature

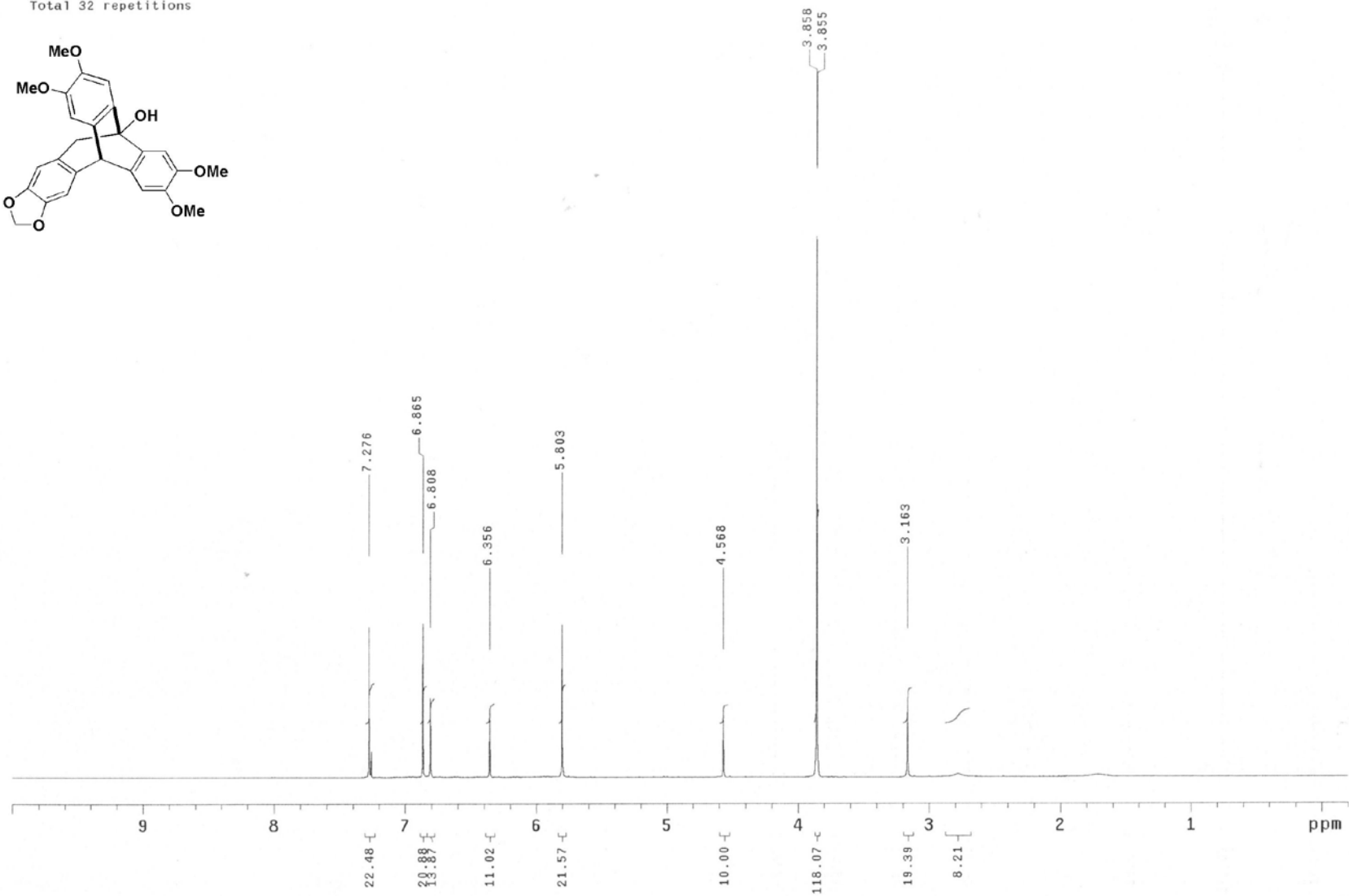
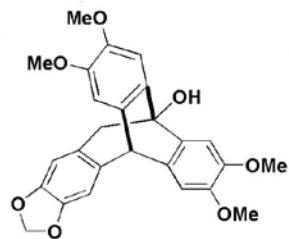
Total 1024 repetitions



Compound 5m (¹H-NMR spectral data)

SH[3.2.2]5m

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 10 2019
Solvent: CDC13
Ambient temperature
Total 32 repetitions



Compound 5m (¹³C-NMR spectral data)

SH[3.2.2]5m

Pulse Sequence: s2pu1

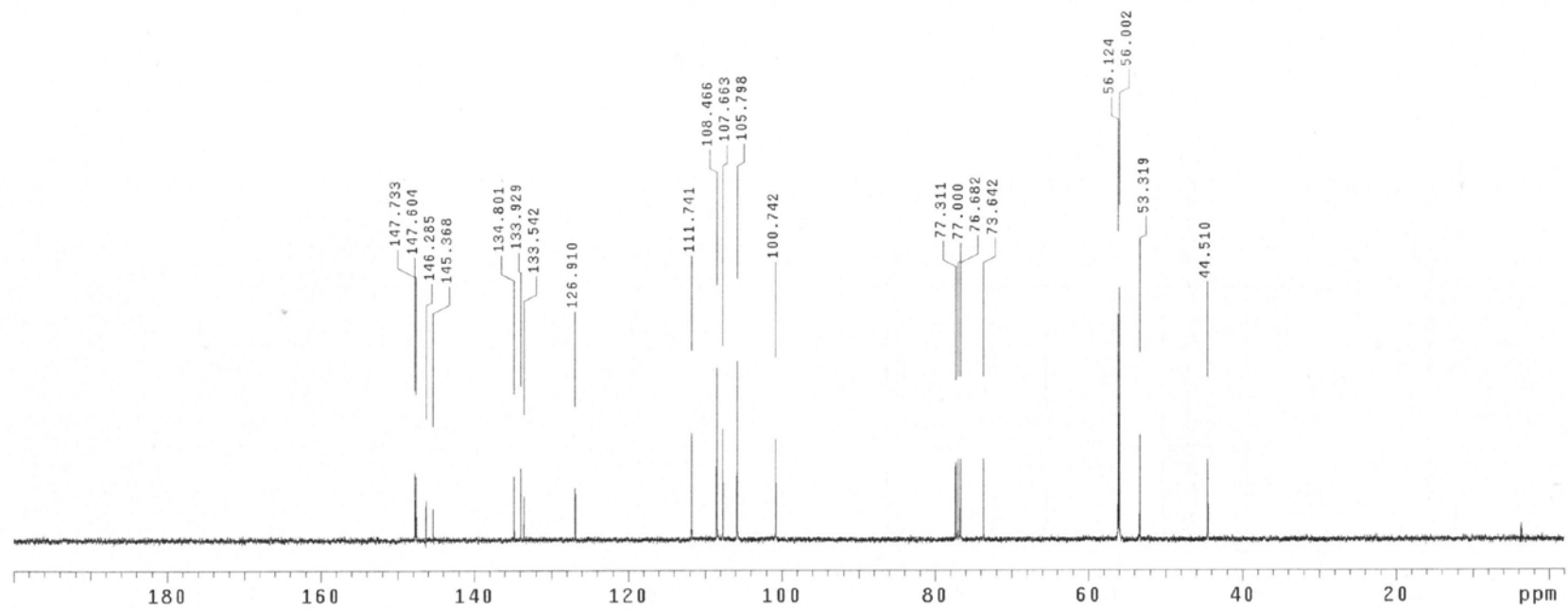
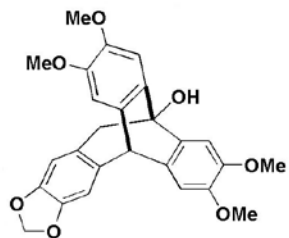
UNITYplus-400 "unity400"

Date: Jan 10 2019

Solvent: CDCl₃

Ambient temperature

Total 1168 repetitions



Compound 5n (¹H-NMR spectral data)

SH[3.2.2]5n

Pulse Sequence: s2pul

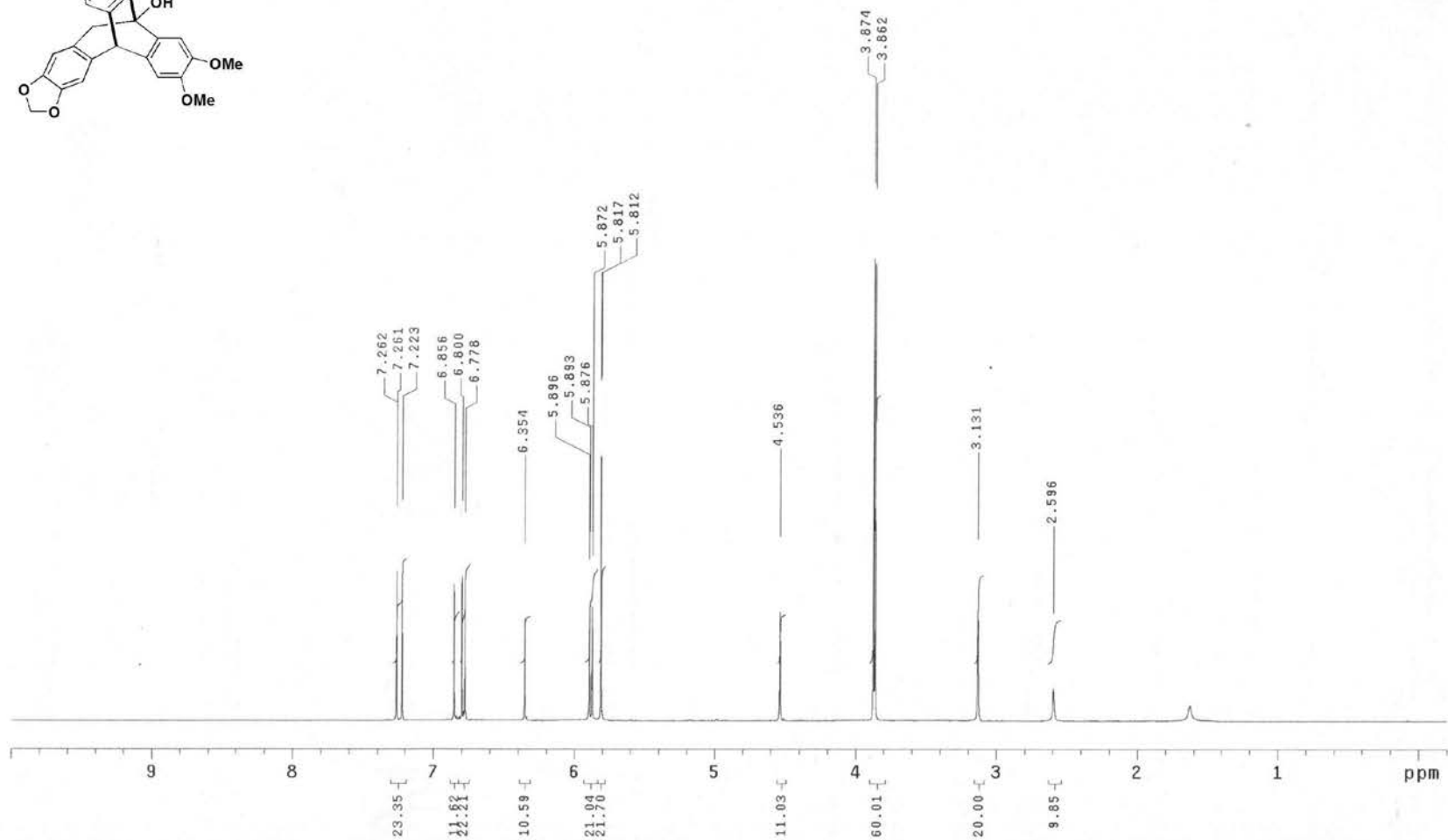
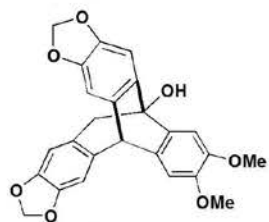
UNITYplus-400 "unity400"

Date: Jan 4 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5n (¹³C-NMR spectral data)

SH[3.2.2]5n

Pulse Sequence: s2pu1

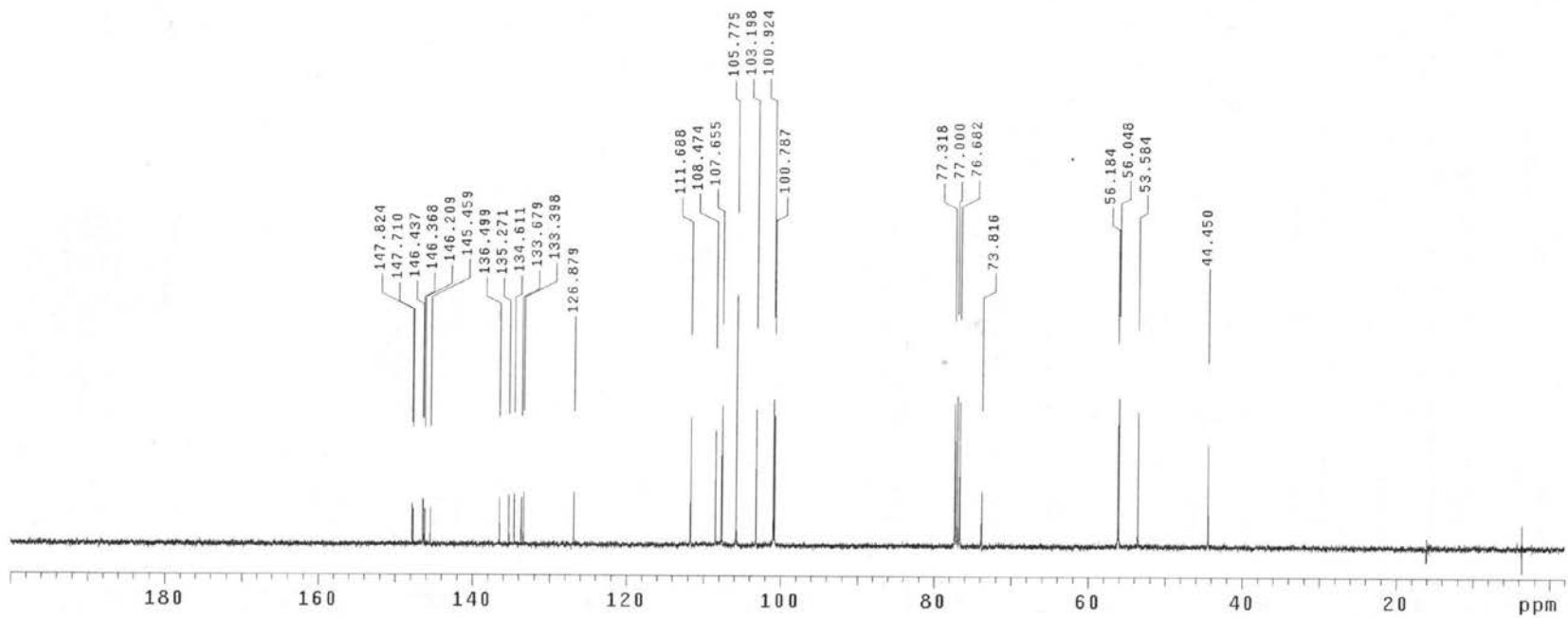
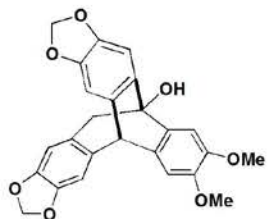
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Date: Jan 4 2019

Solvent: CDC13

Ambient temperature

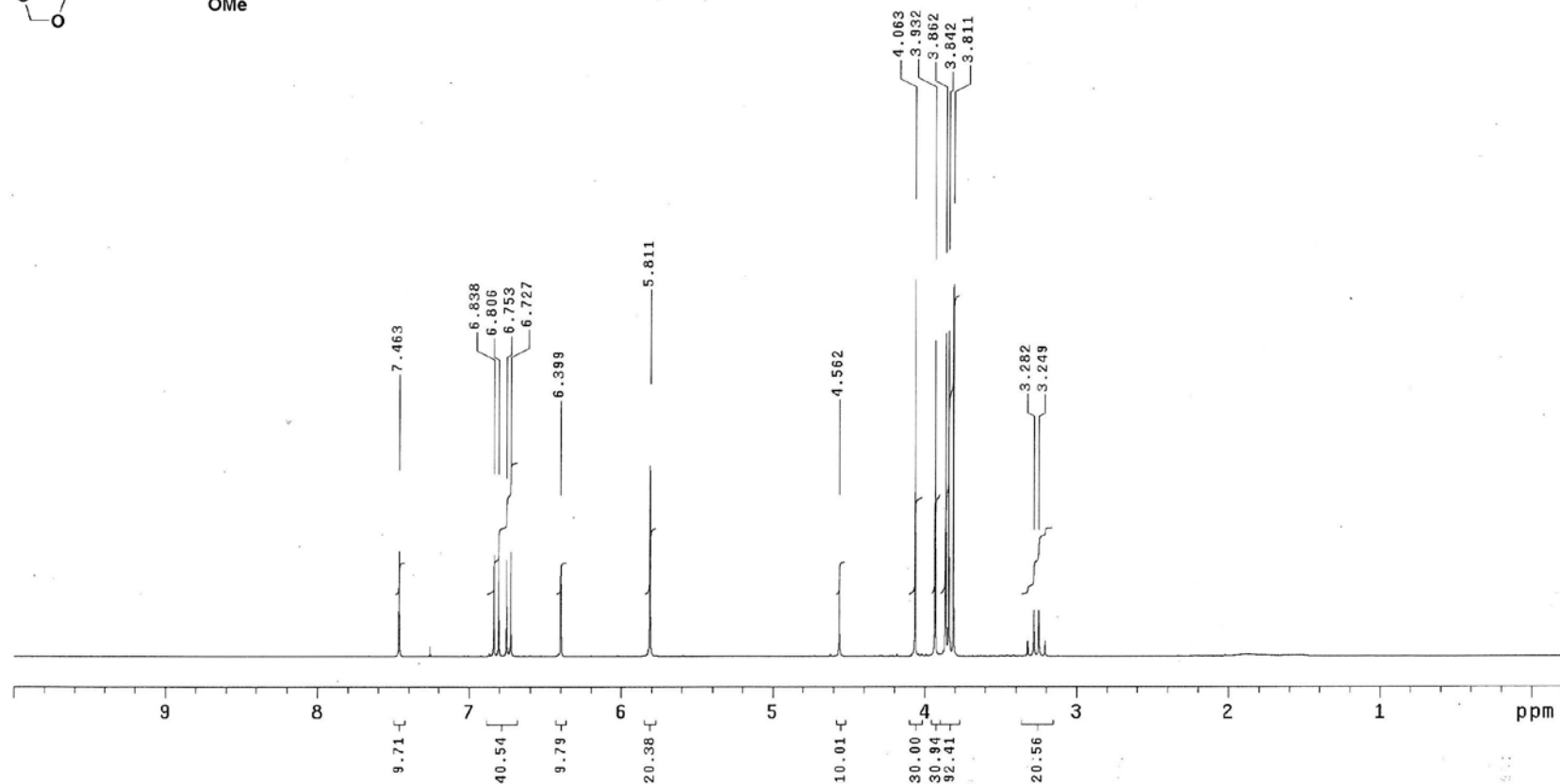
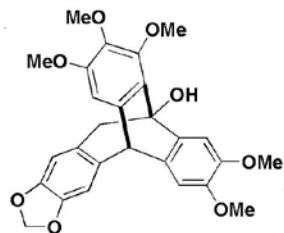
Total 2432 repetitions



Compound 5o (¹H-NMR spectral data)

SH[3.2.2]5o

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Feb 18 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



Compound 5o (¹³C-NMR spectral data)

SH[3.2.2]5o

Pulse Sequence: s2pu1

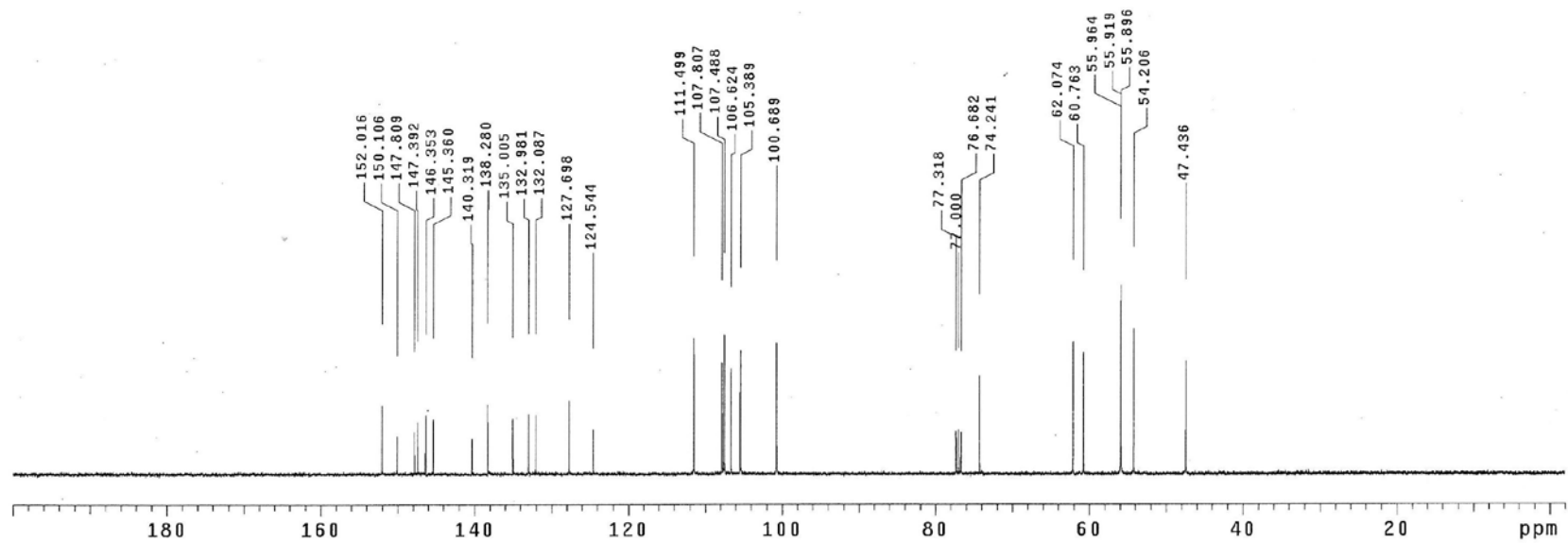
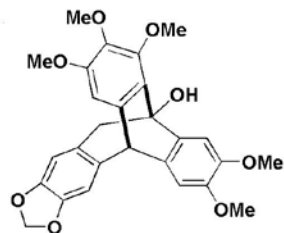
UNITYplus-400 "unity400"

Date: Feb 18 2019

Solvent: CDC13

Ambient temperature

Total 480 repetitions



Compound 5p (¹H-NMR spectral data)

SH[3.2.2]5p

Pulse Sequence: s2pu1

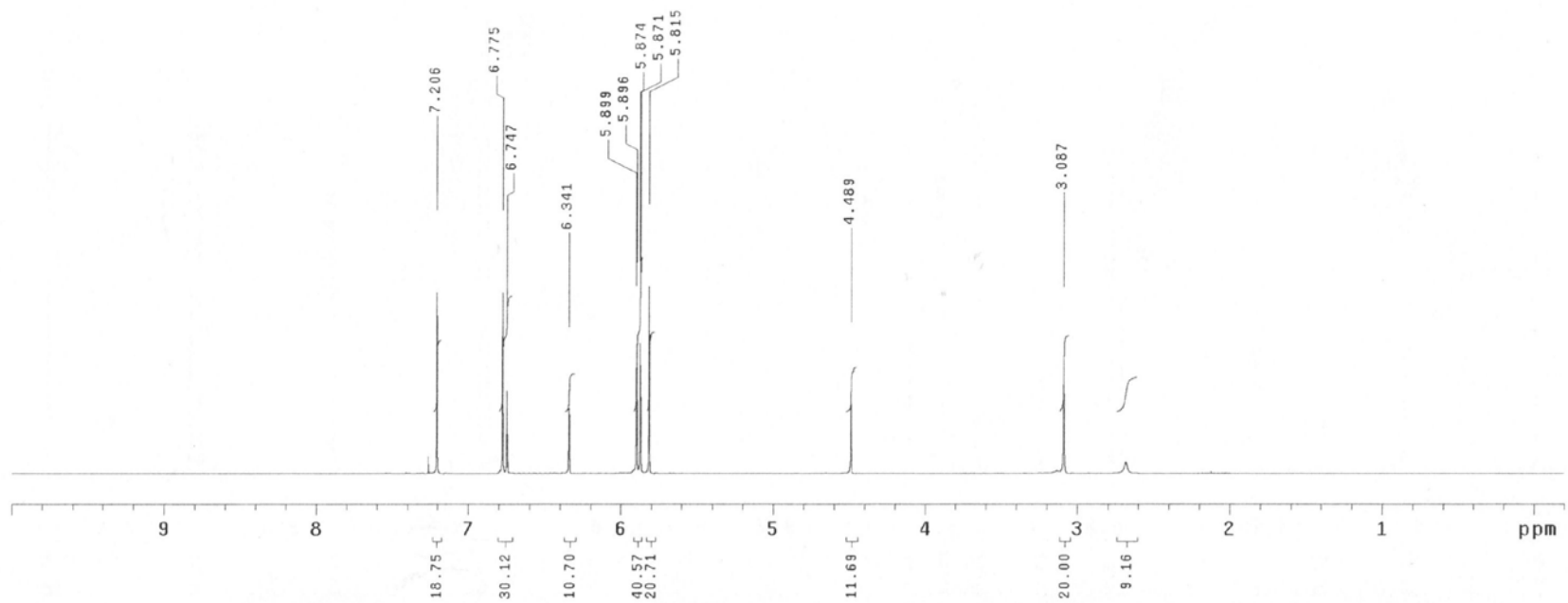
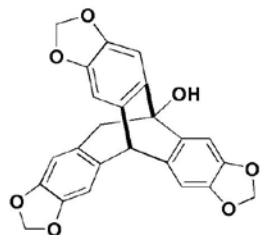
UNITYplus-400 "unity400"

Date: Jan 23 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5p (¹³C-NMR spectral data)

SH[3.2.2]5p

Pulse Sequence: s2pu1

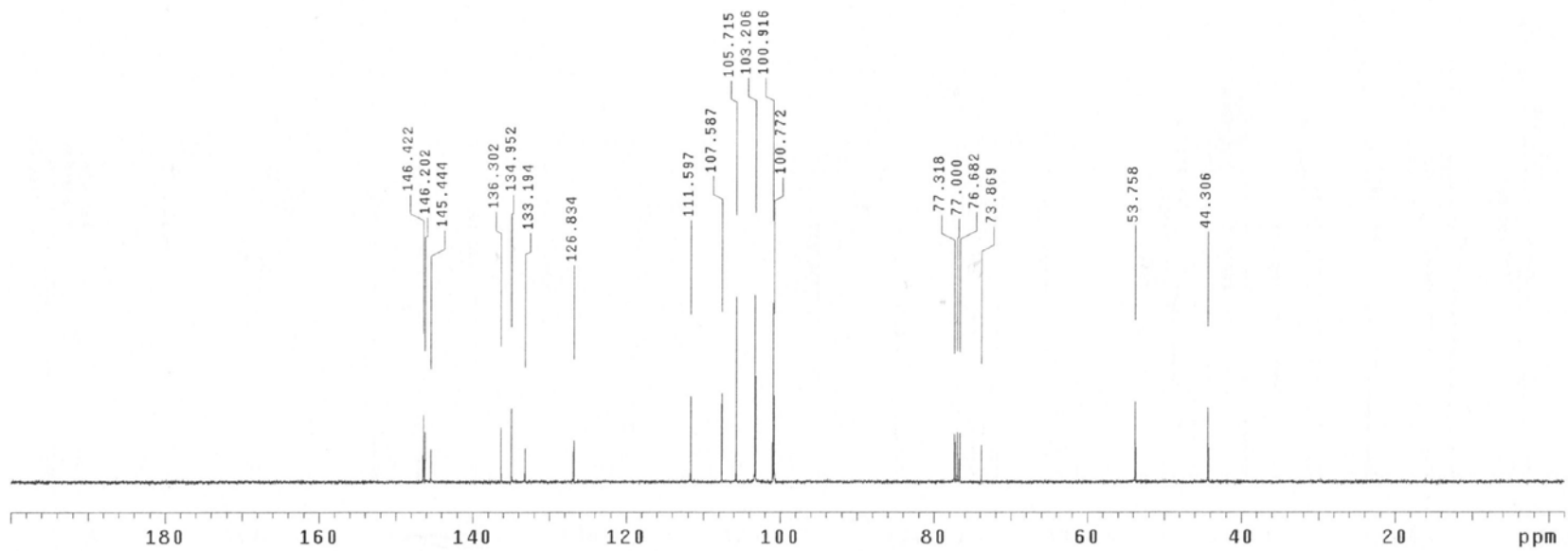
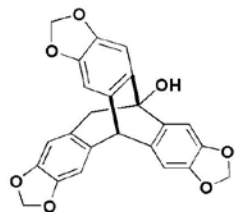
UNITYplus-400 "unity400"

Date: Jan 23 2019

Solvent: CDCl₃

Ambient temperature

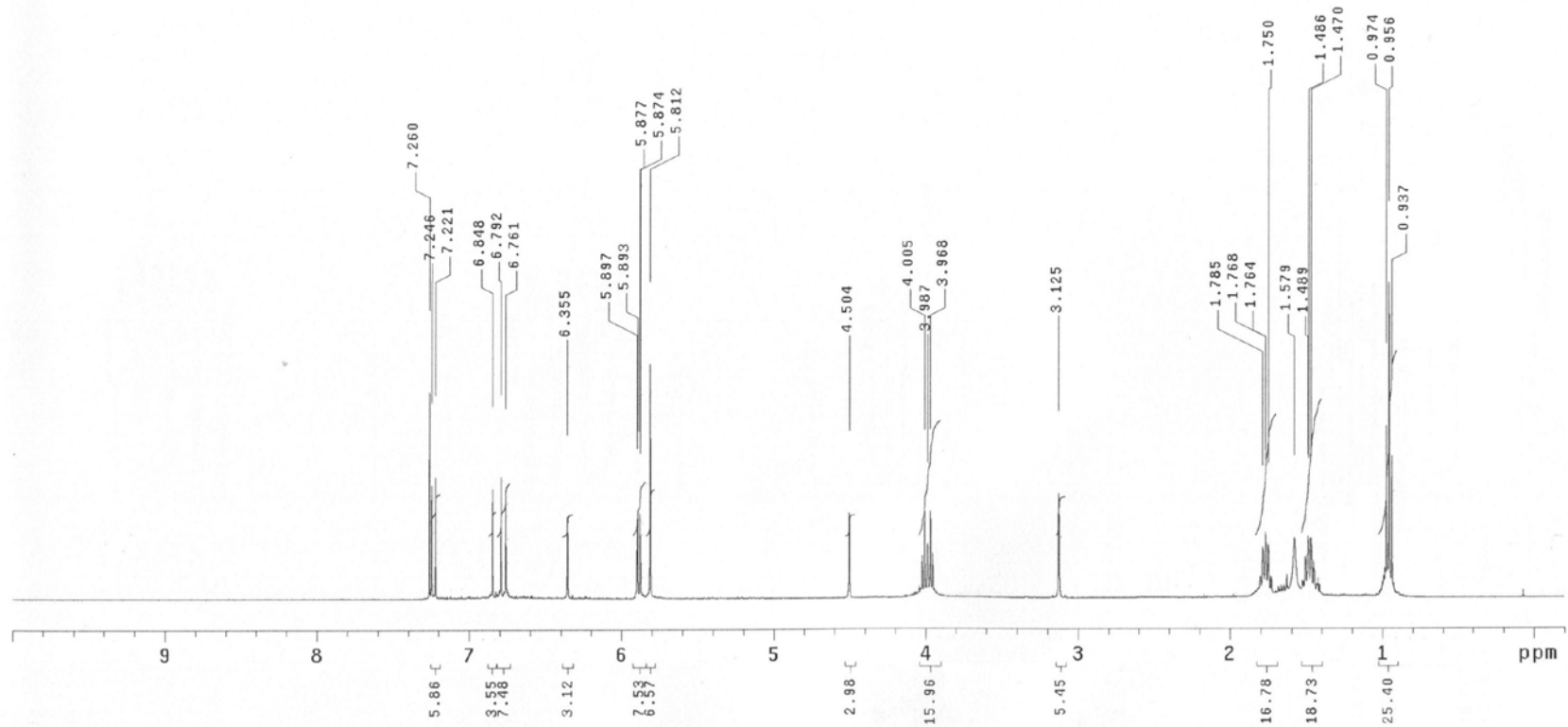
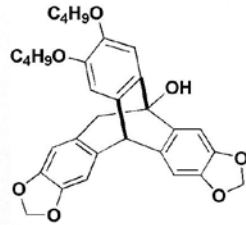
Total 768 repetitions



Compound 5q (¹H-NMR spectral data)

SH[3.2.2]5q

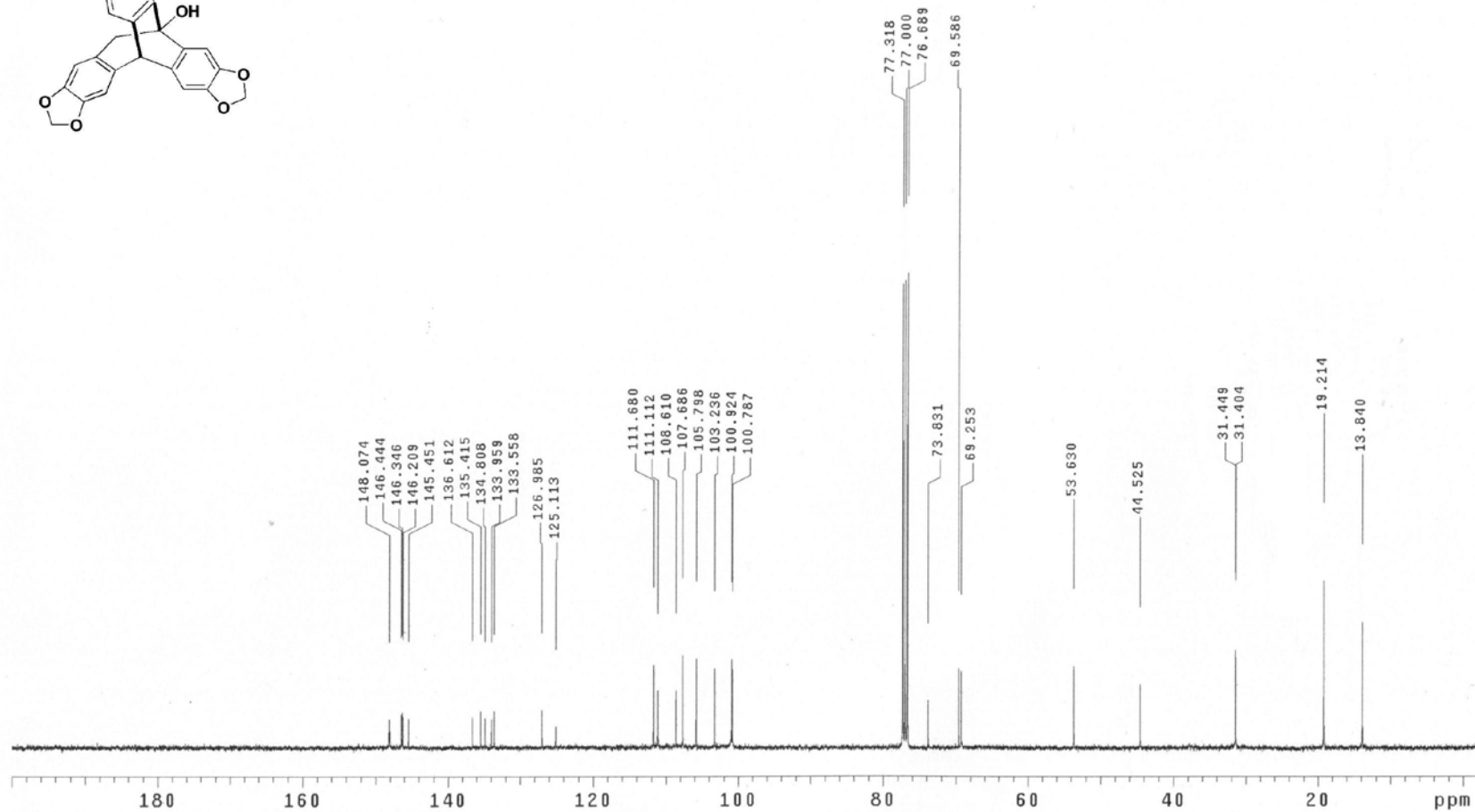
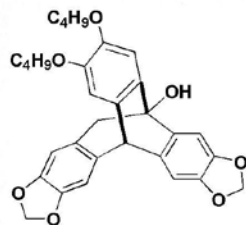
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: May 20 2019
 Solvent: CDCl3
 Ambient temperature
 Total 64 repetitions



Compound 5q (¹³C-NMR spectral data)

SH[3.2.2]5q

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: May 20 2019
Solvent: CDCl₃
Ambient temperature
Total 28864 repetitions



Compound 5r (¹H-NMR spectral data)

SH[3.2.2]5r-1

Pulse Sequence: s2pul

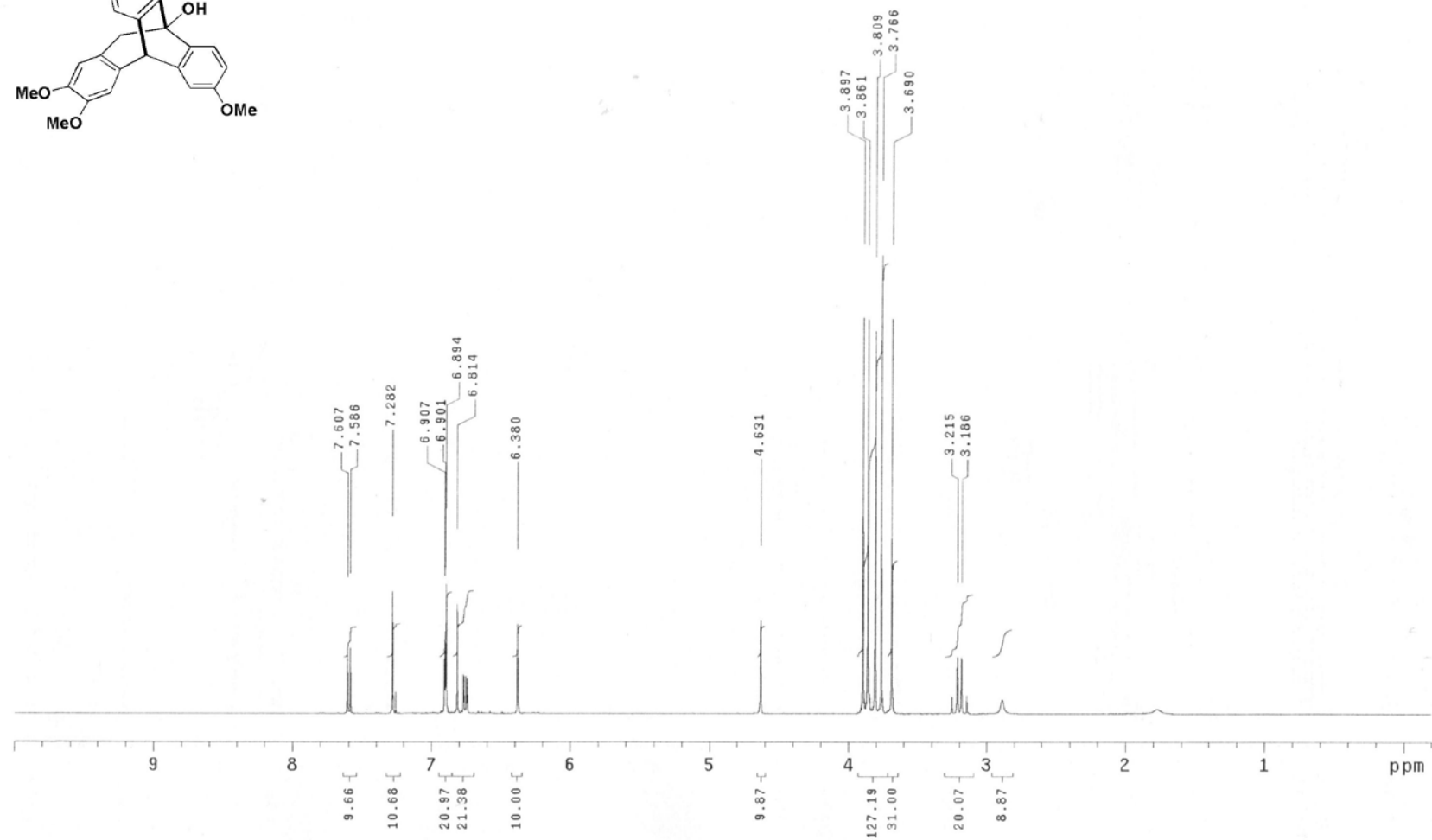
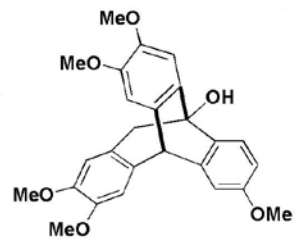
UNITYplus-400 "unity400"

Date: Jan 22 2019

Solvent: CDCl₃

Ambient temperature

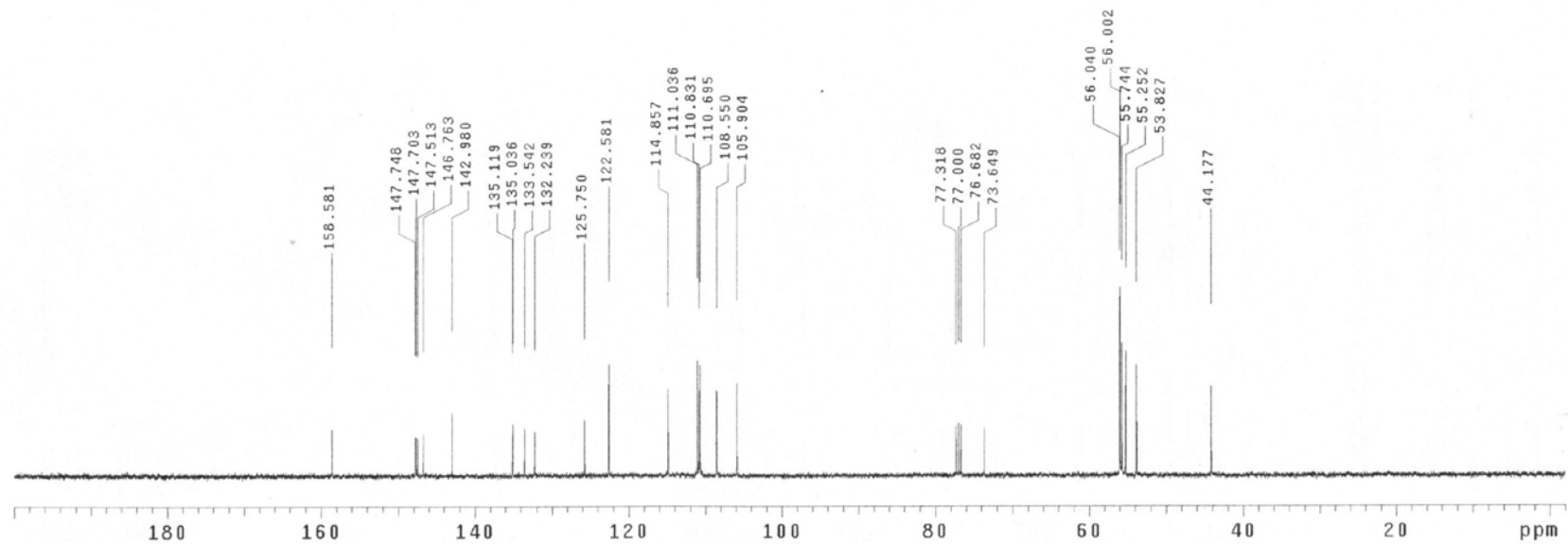
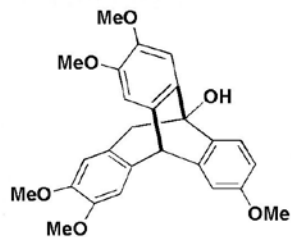
Total 32 repetitions



Compound 5r (¹³C-NMR spectral data)

SH[3.2.2]5r-1

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 22 2019
Solvent: CDCl3
Ambient temperature
Total 464 repetitions



Compound 5r-1 (¹H-NMR spectral data)

SH[3.2.2]5r

Pulse Sequence: s2pu1

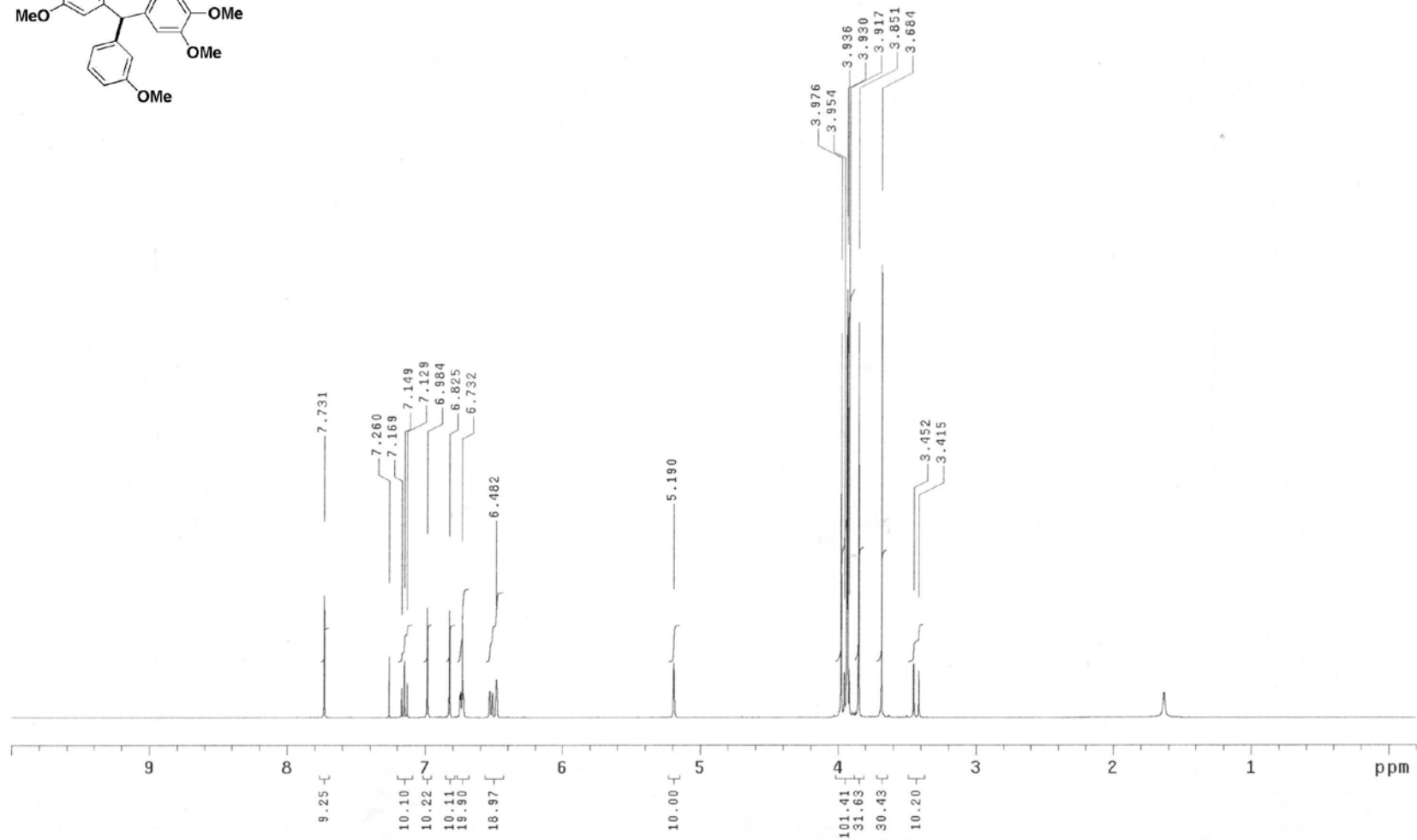
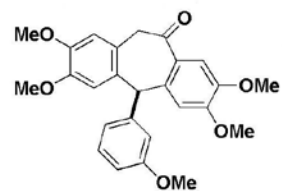
UNITYplus-400 "unity400"

Date: Jan 18 2019

Solvent: CDCl₃

Ambient temperature

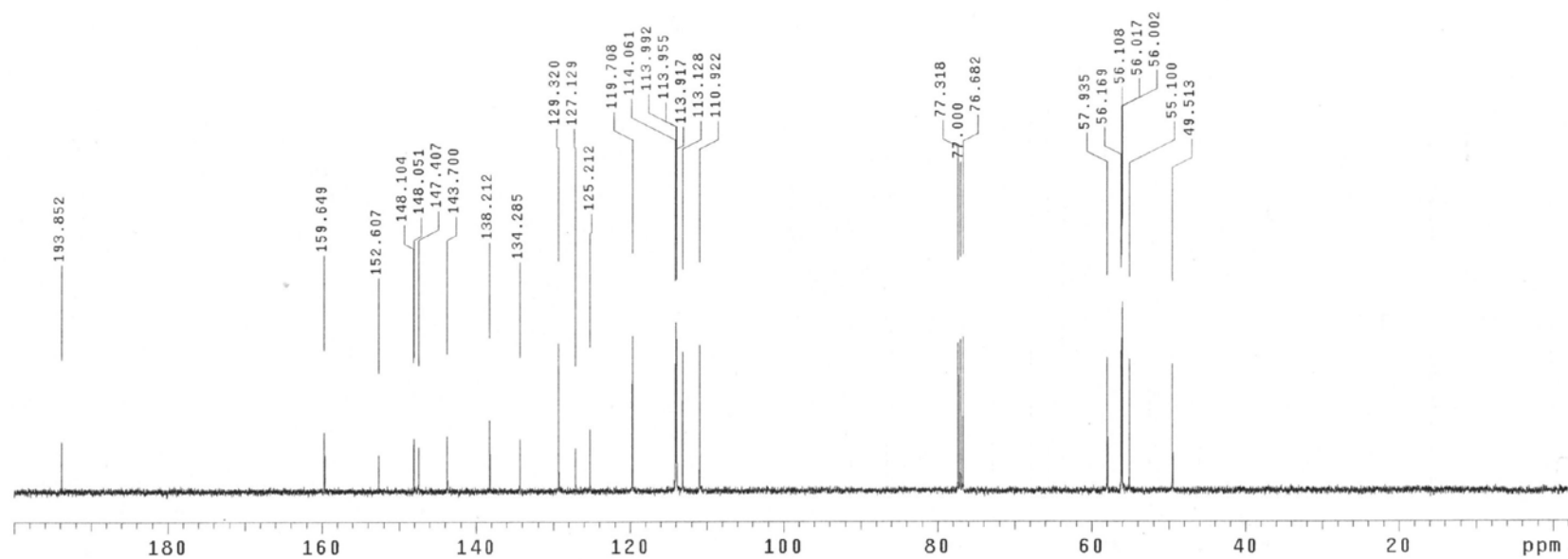
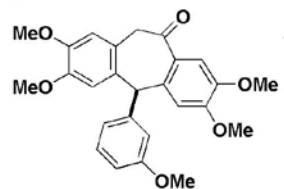
Total 32 repetitions



Compound 5r-1 (¹³C-NMR spectral data)

SH[3.2.2]5r

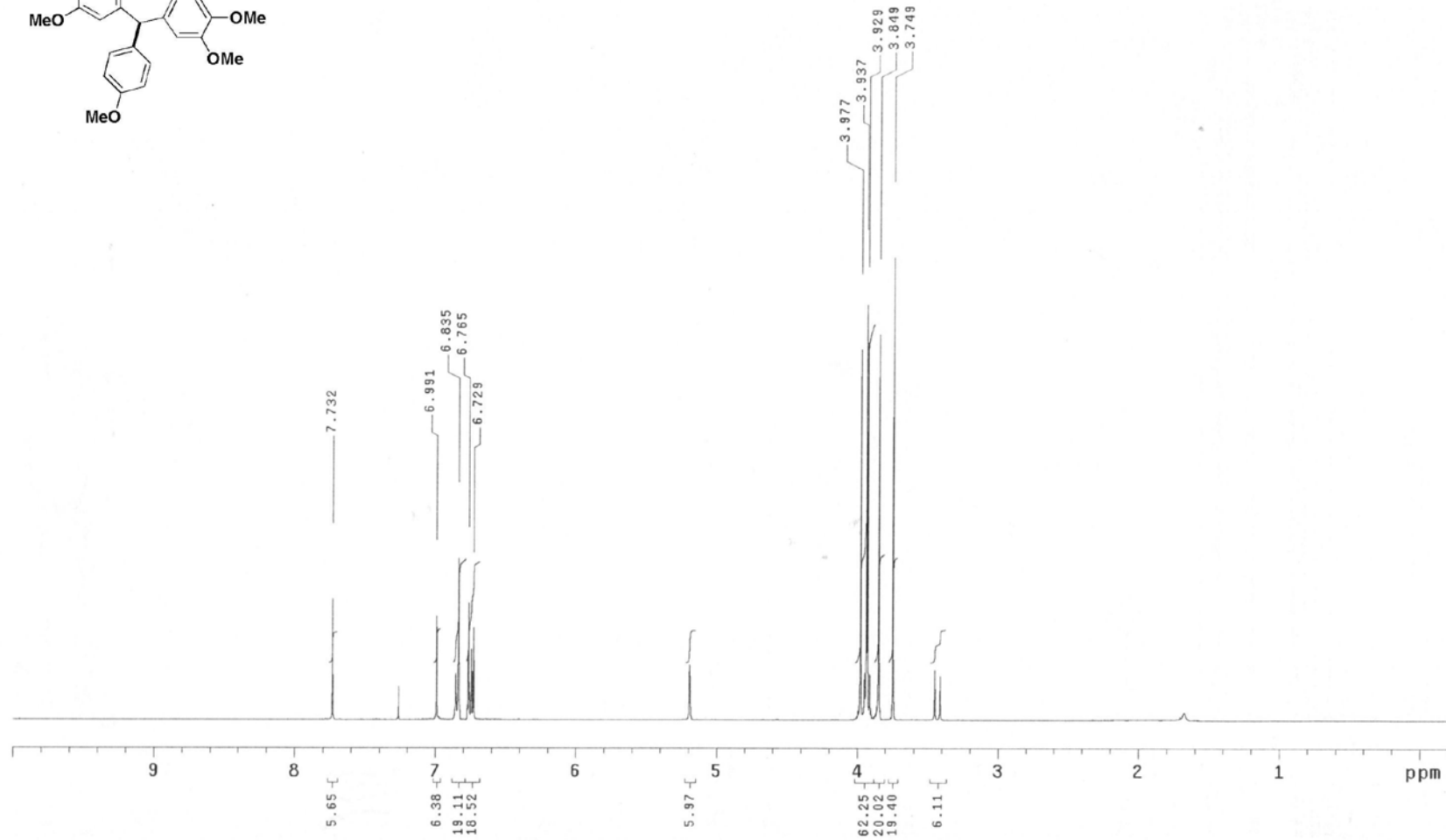
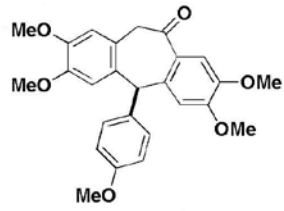
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 18 2019
Solvent: CDCl3
Ambient temperature
Total 2544 repetitions



Compound 5s-1 (¹H-NMR spectral data)

SH[3.2.2]5s

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 17 2019
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 5s-1 (¹³C-NMR spectral data)

SH[3.2.2]5s

Pulse Sequence: s2pu1

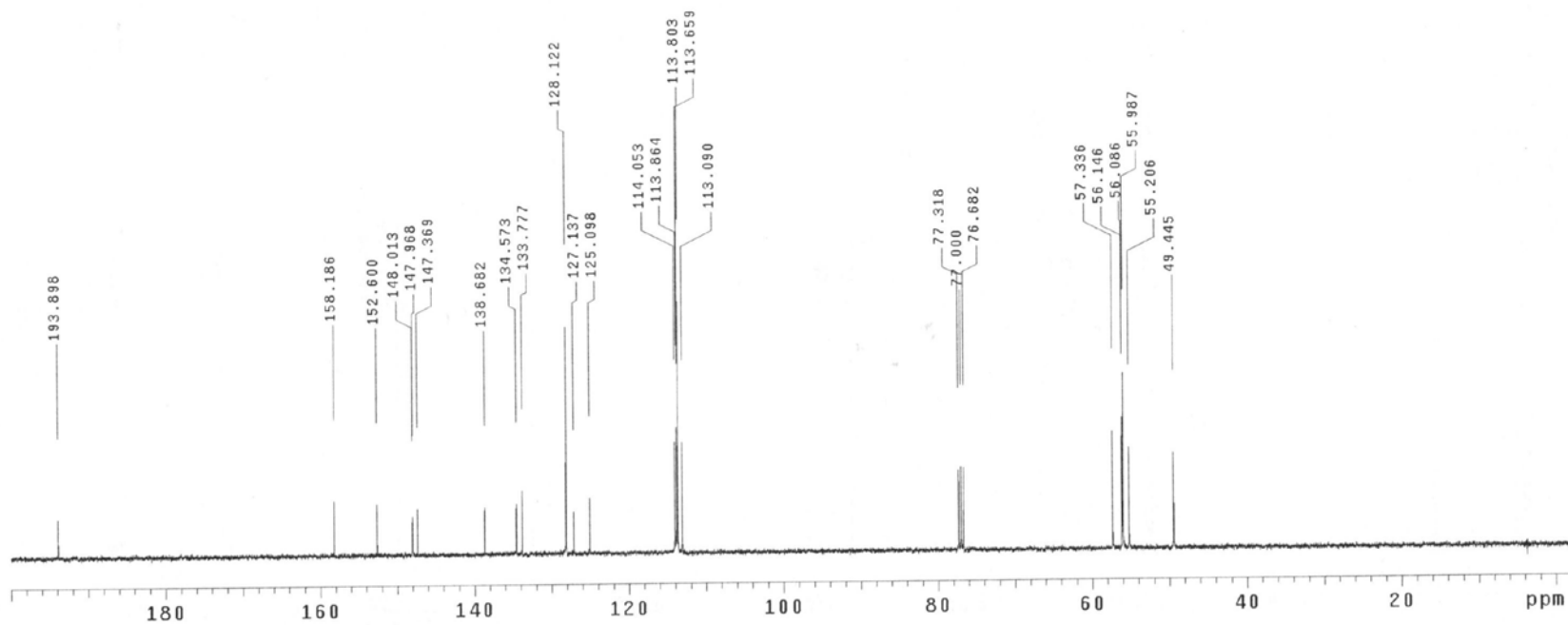
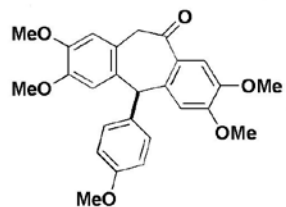
UNITYplus-400 "unity400"

Date: Jan 17 2019

Solvent: CDCl₃

Ambient temperature

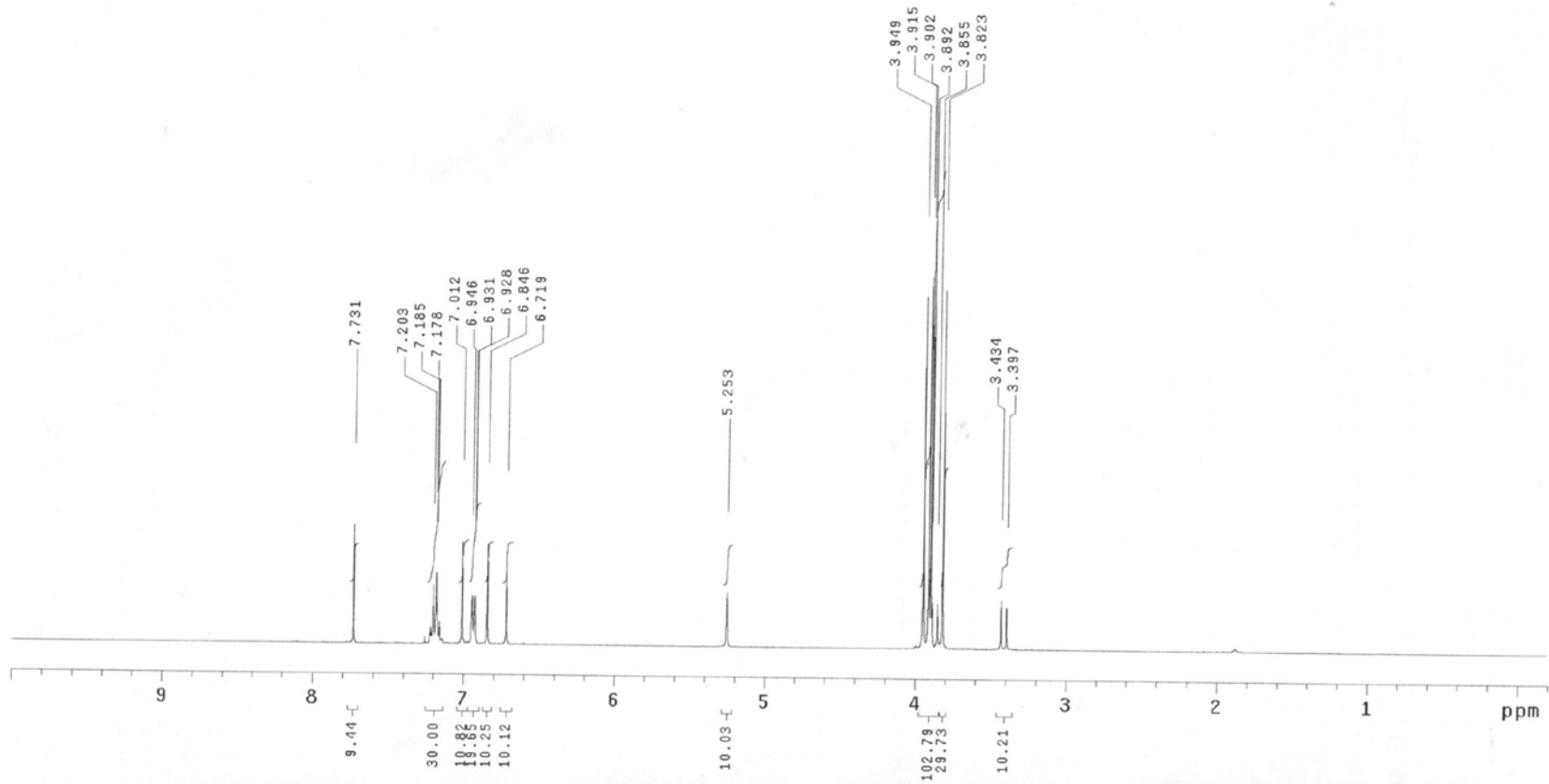
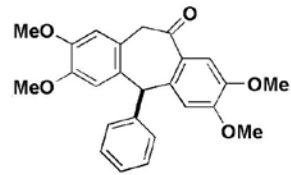
Total 1088 repetitions



Compound 5t-1 (¹H-NMR spectral data)

SH[3.2.2]5t

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 7 2019
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 5t-1 (¹³C-NMR spectral data)

SH[3.2.2]5t

Pulse Sequence: s2pu1

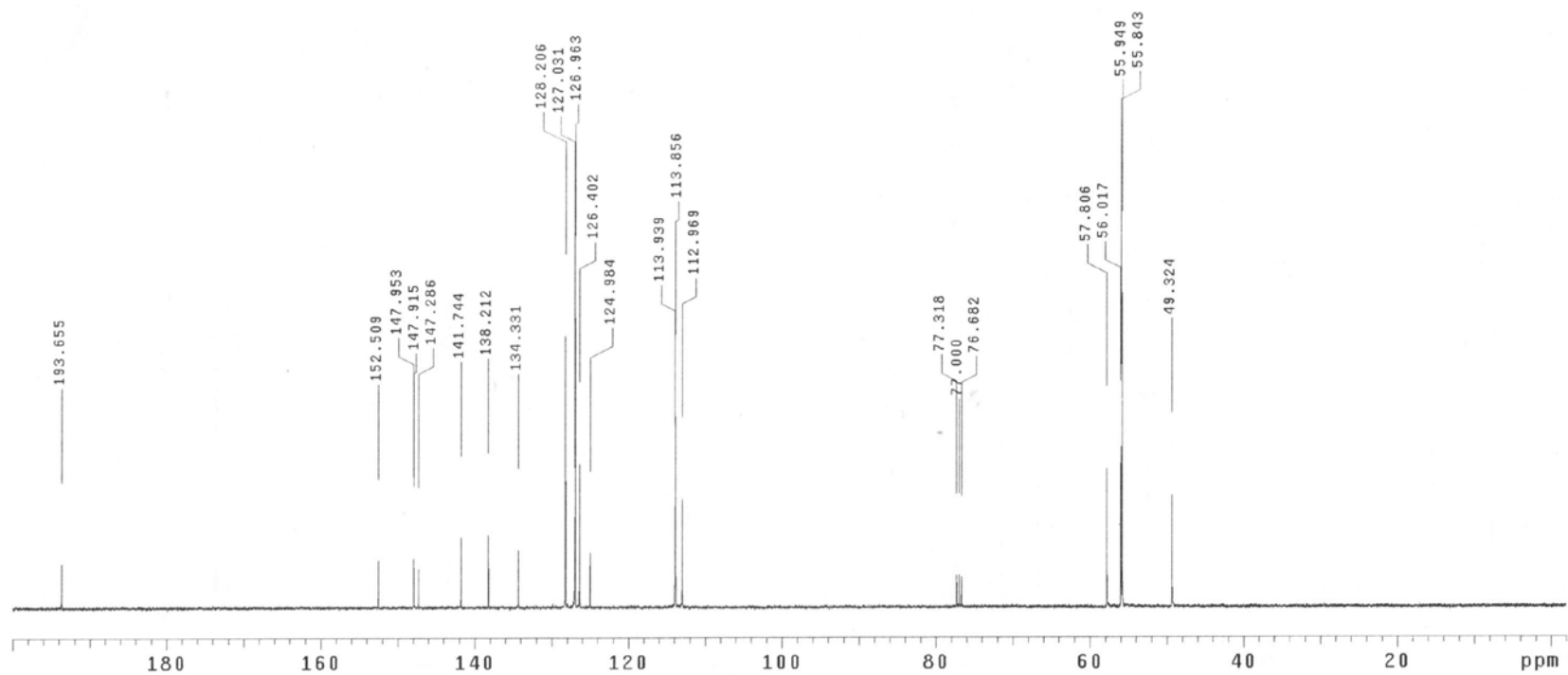
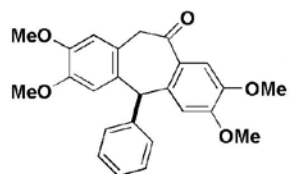
UNITYplus-400 "unity400"

Date: Jan 7 2019

Solvent: CDCl₃

Ambient temperature

Total 240 repetitions



Compound 5u-1 (¹H-NMR spectral data)

SH[3.2.2]5u

Pulse Sequence: s2pu1

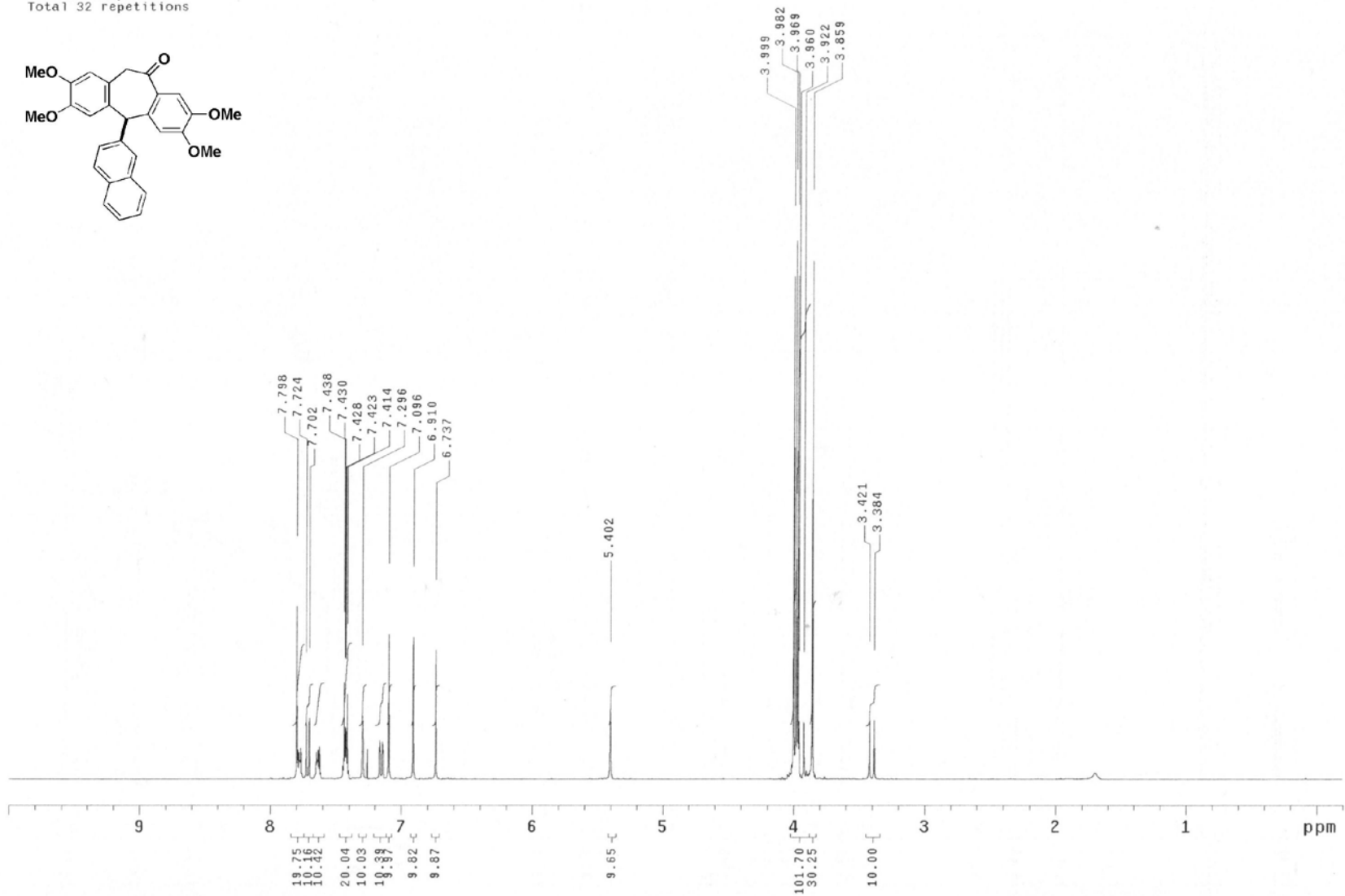
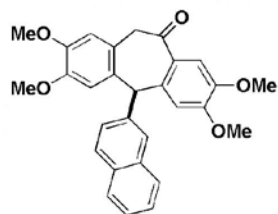
UNITYplus-400 "unity400"

Date: Jan 15 2019

Solvent: CDCl₃

Ambient temperature

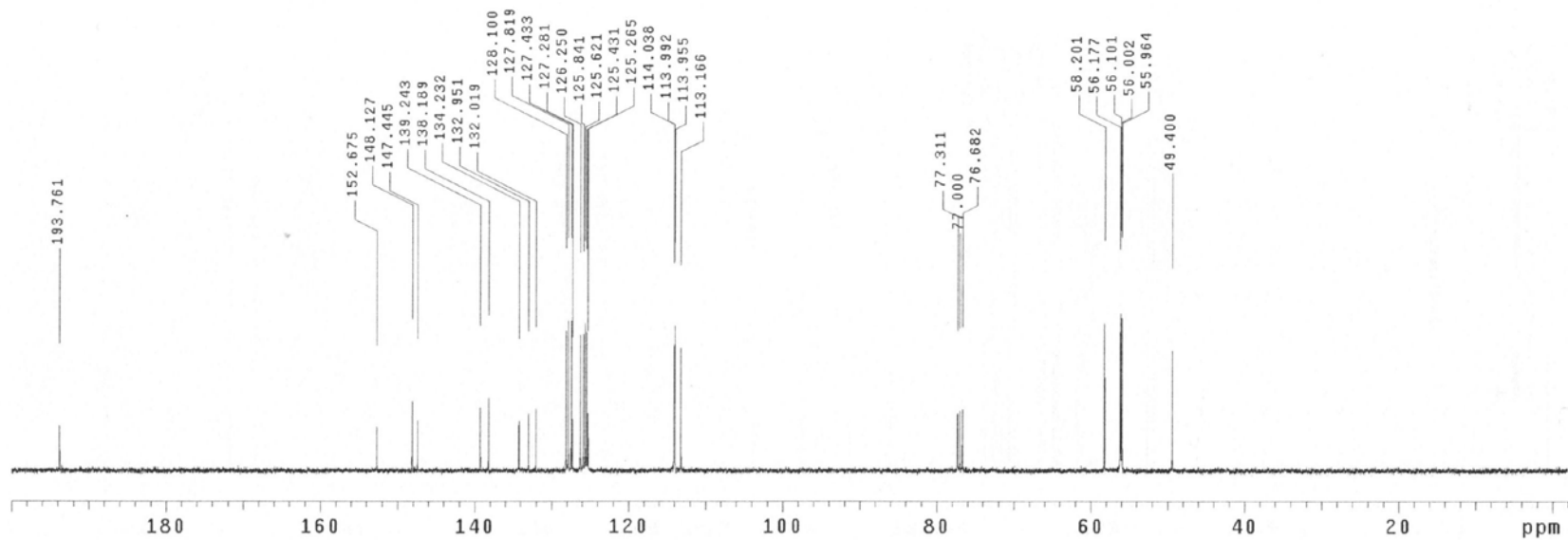
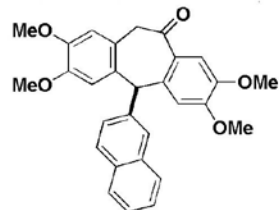
Total 32 repetitions



Compound 5u-1 (¹³C-NMR spectral data)

SH[3.2.2]5u

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 15 2019
Solvent: CDCl₃
Ambient temperature
Total 640 repetitions



Compound 5v-1 (¹H-NMR spectral data)

SH[3.2.2]4v

Pulse Sequence: s2pu1

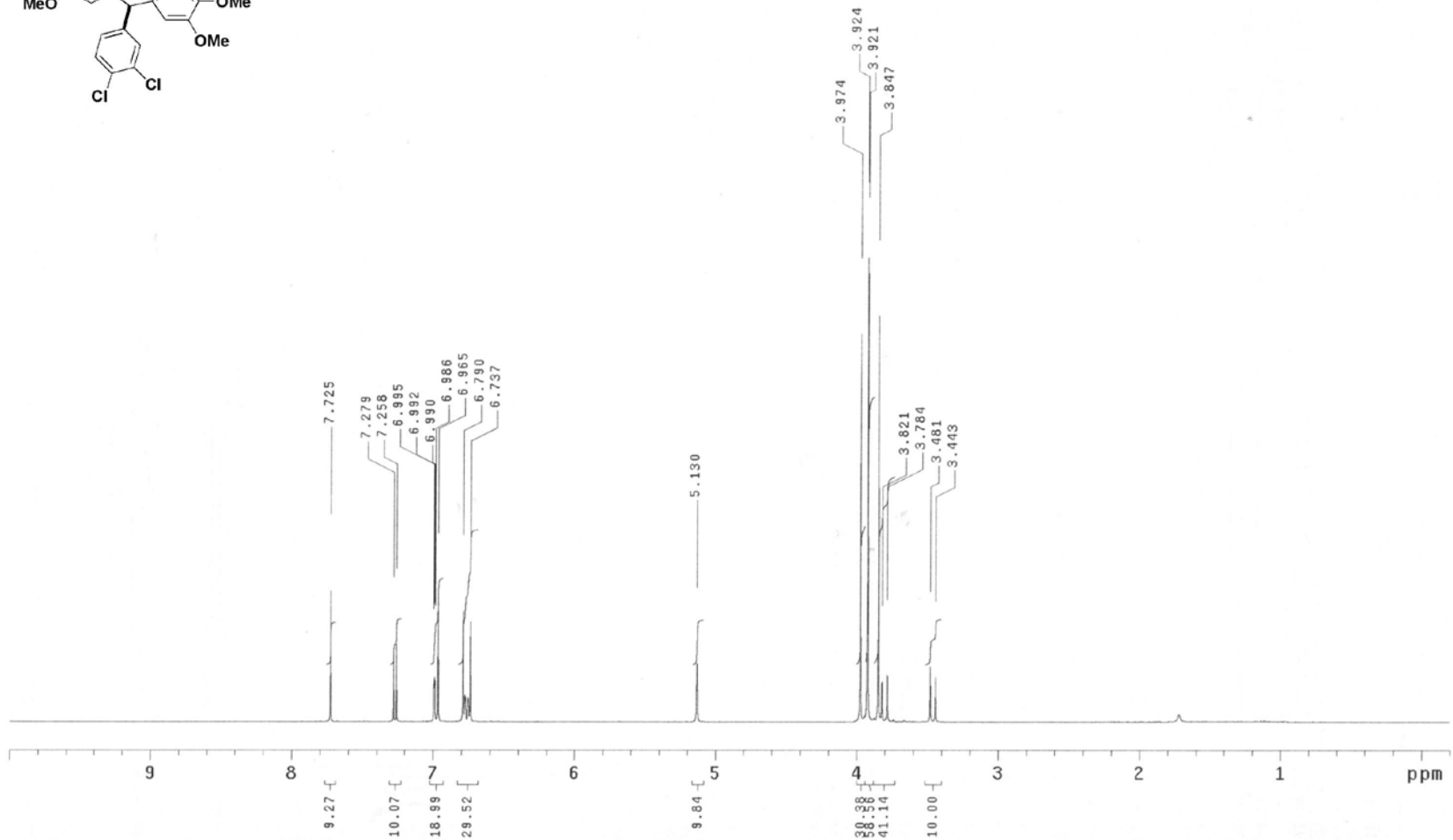
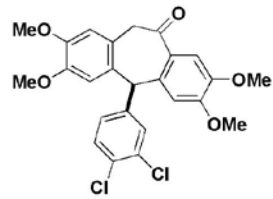
UNITYplus-400 "unity400"

Date: Jan 18 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5v-1 (¹³C-NMR spectral data)

SH[3.2.2]4v

Pulse Sequence: s2pu1

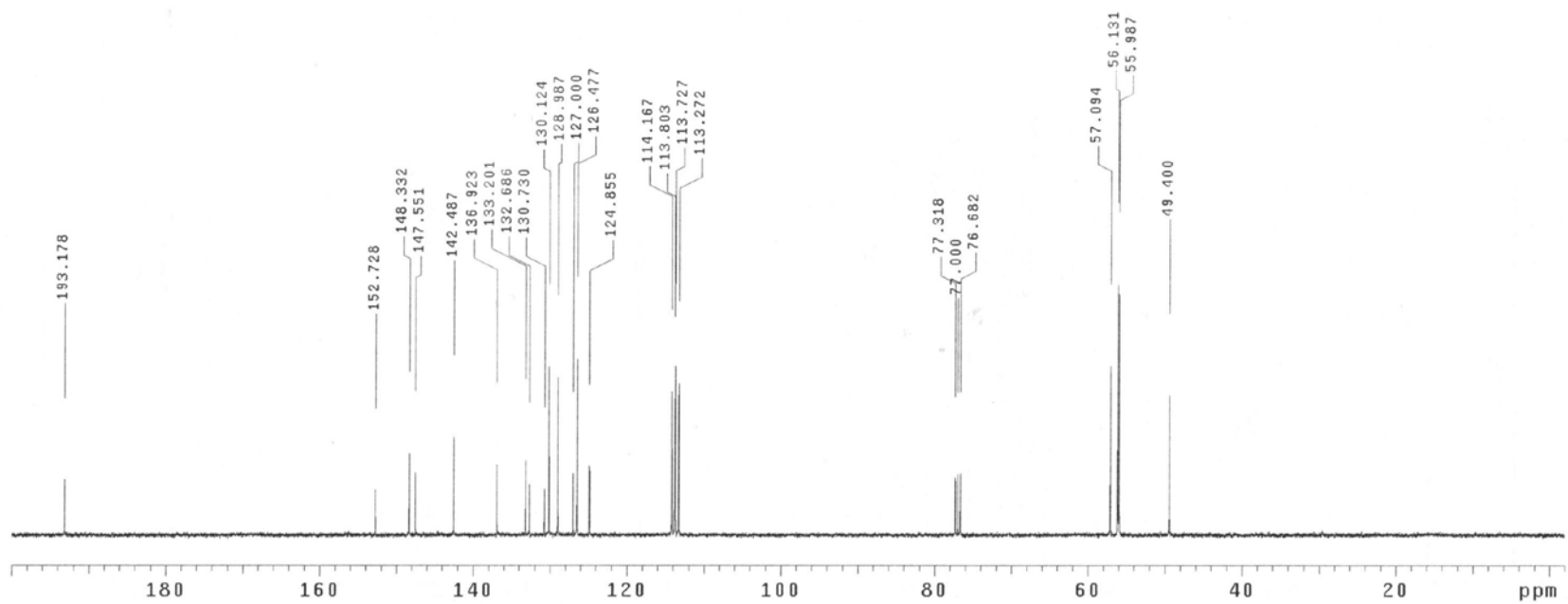
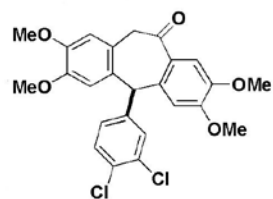
UNITYplus-400 "unity400"

Date: Jan 18 2019

Solvent: CDCl₃

Ambient temperature

Total 1040 repetitions



Compound 5w-1 (¹H-NMR spectral data)

SH[3.2.2]5w

Pulse Sequence: s2pu1

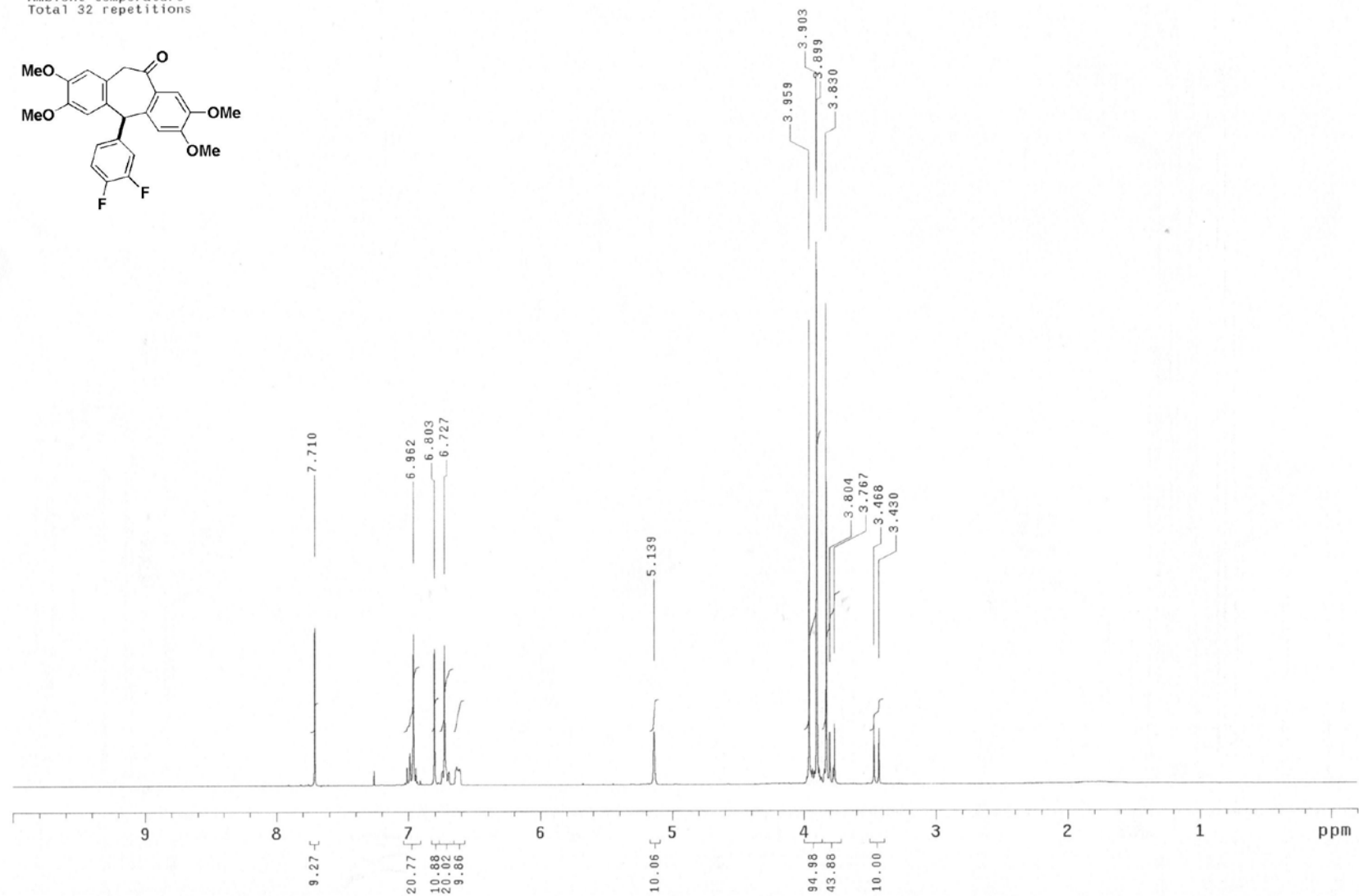
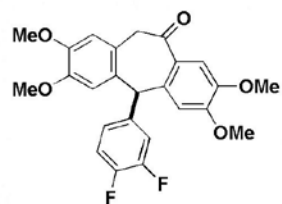
UNITYplus-400 "unity400"

Date: Jan 29 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5w-1 (¹³C-NMR spectral data)

SH[3.2.2]5w

Pulse Sequence: s2pu1

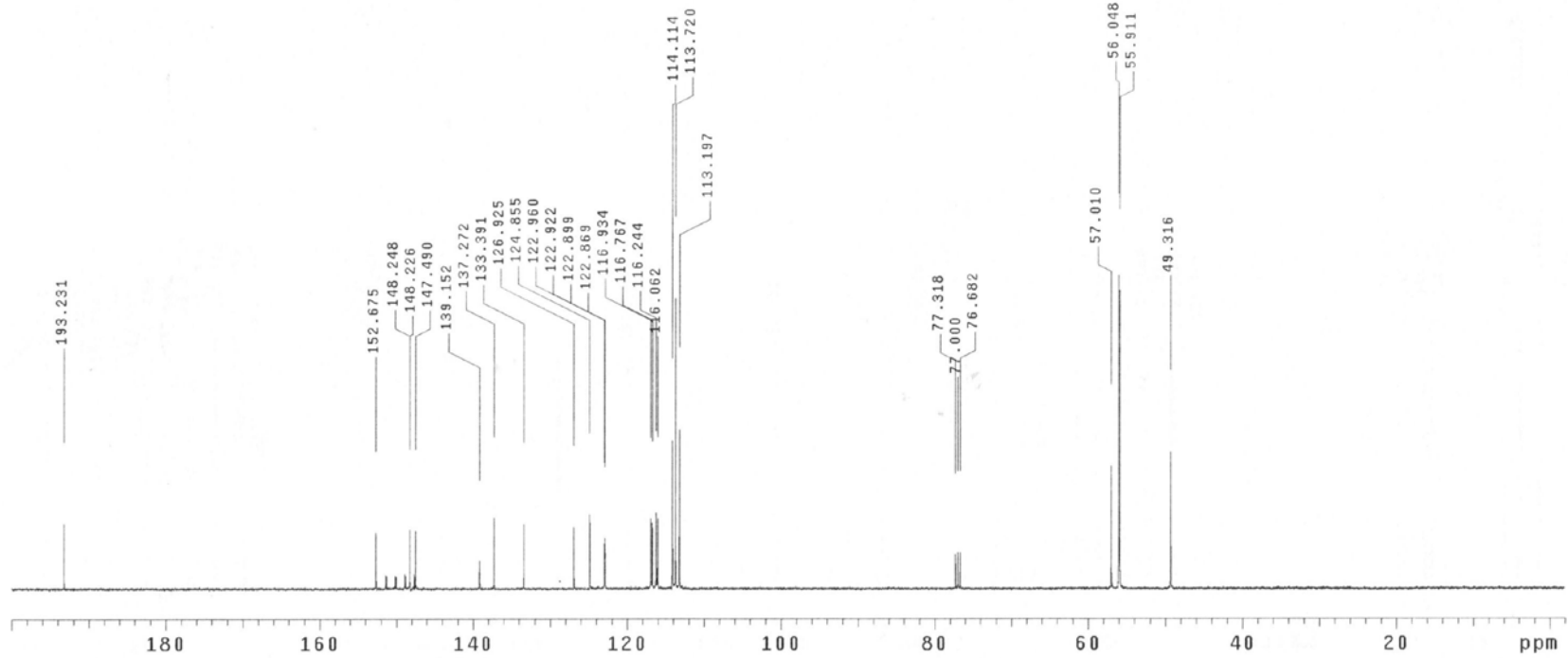
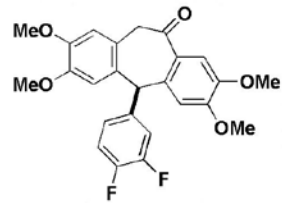
UNITYplus-400 "unity400"

Date: Jan 29 2019

Solvent: CDCl₃

Ambient temperature

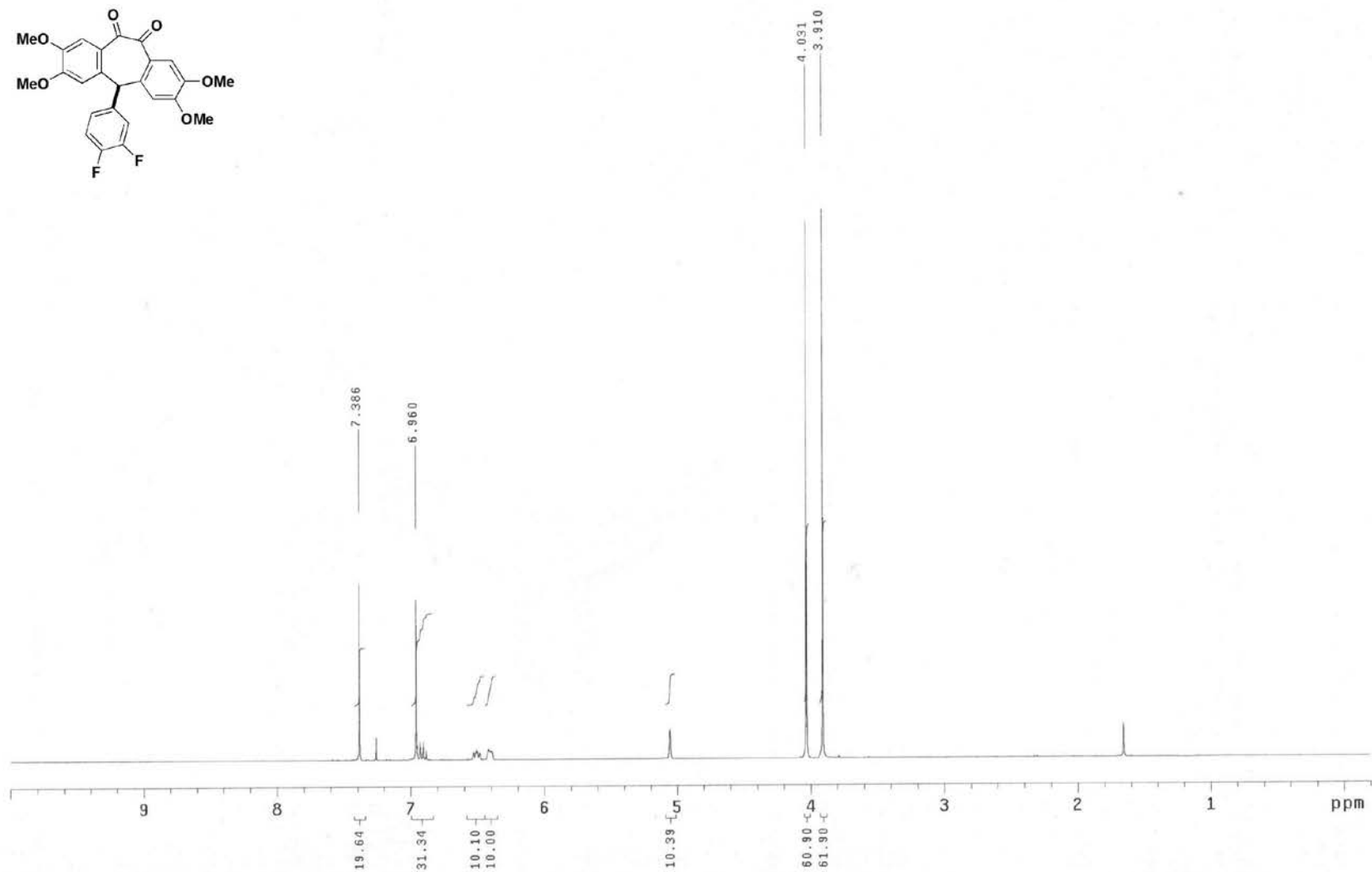
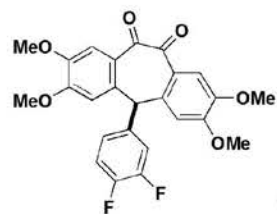
Total 1152 repetitions



Compound 5w-2 (¹H-NMR spectral data)

SH5W-1

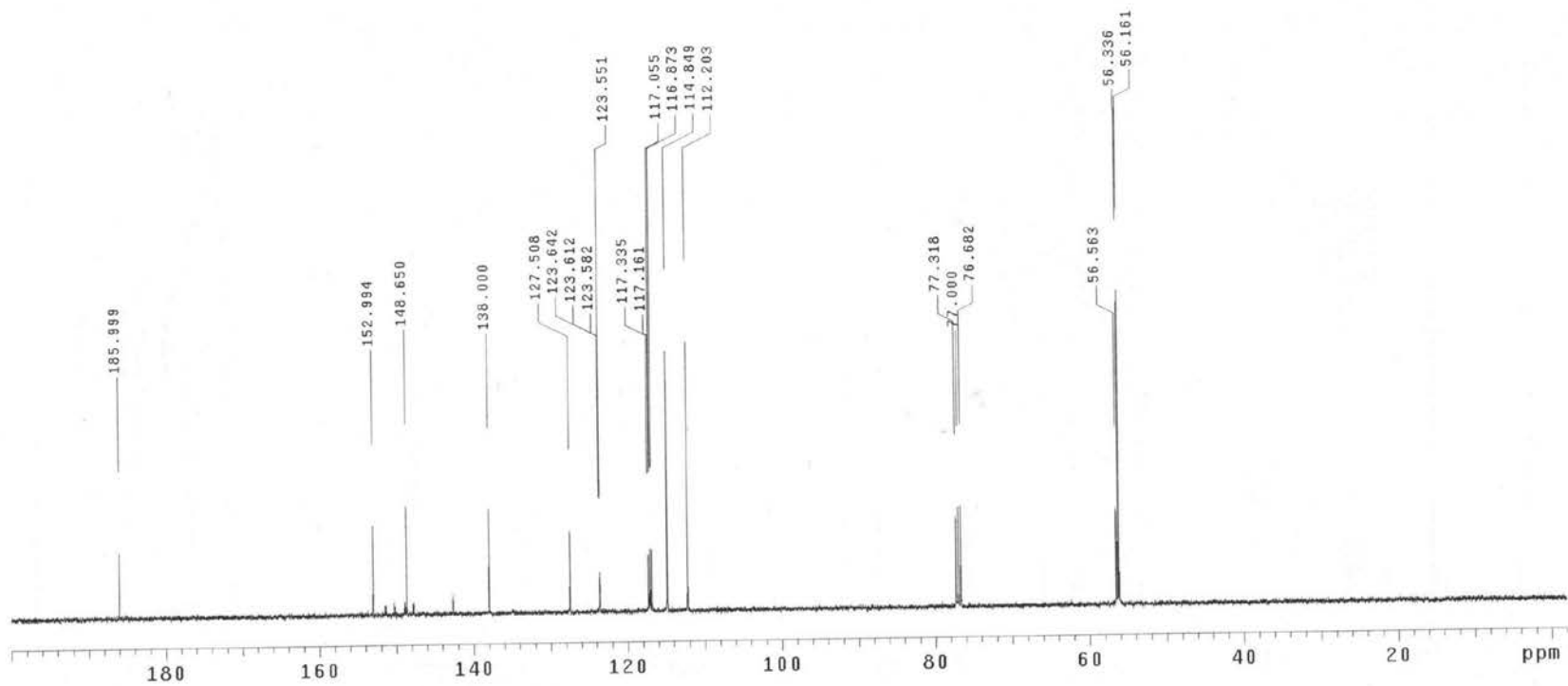
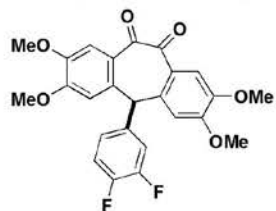
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 18 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



Compound 5w-2 (¹³C-NMR spectral data)

SH5W-1

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 18 2019
Solvent: CDCl₃
Ambient temperature
Total 1616 repetitions



Compound 5x (¹H-NMR spectral data)

SH[3.2.2]5bb

Pulse Sequence: s2pu1

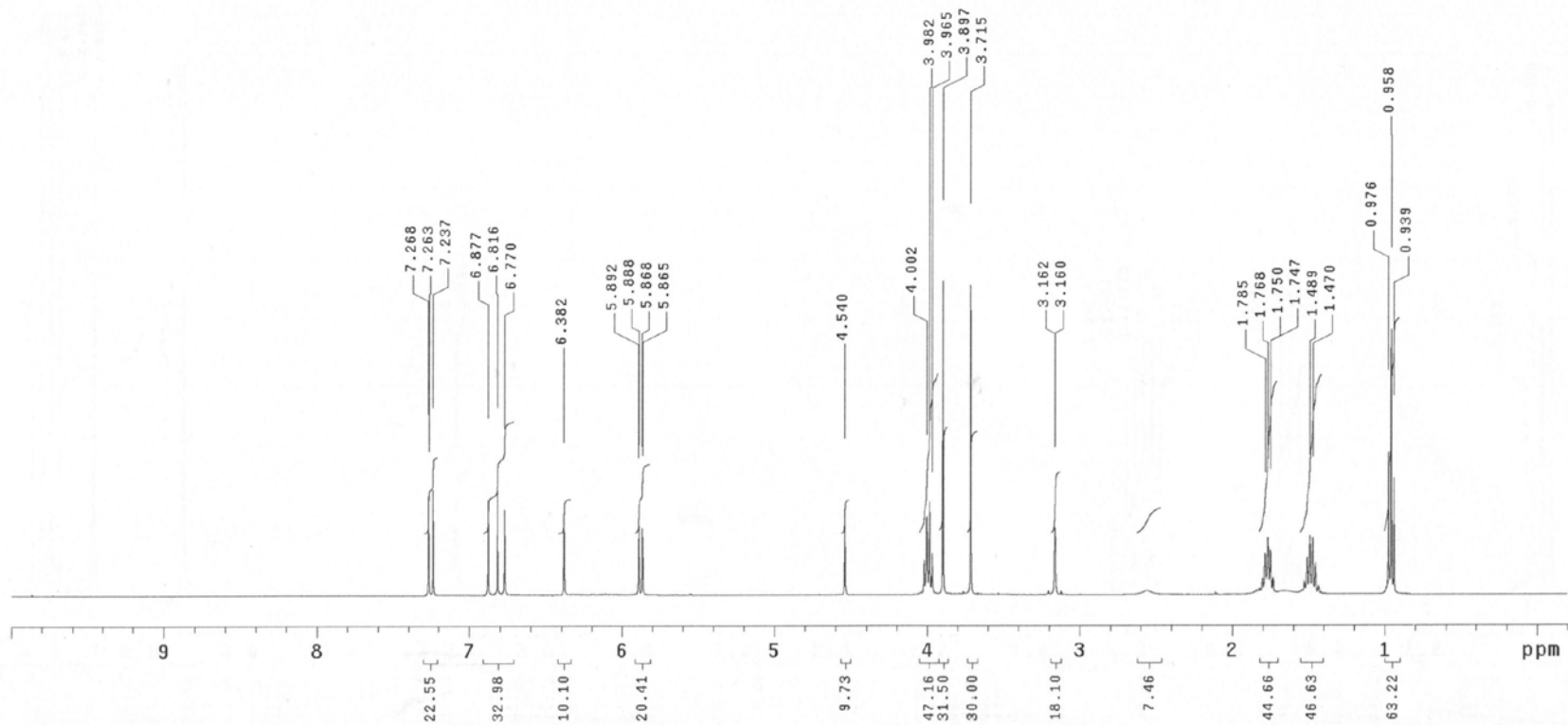
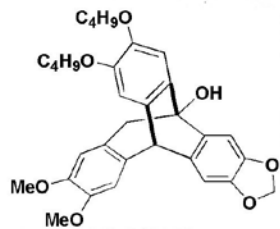
UNITYplus-400 "unity400"

Date: Apr 11 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5x (¹³C-NMR spectral data)

SH[3.2.2]5bb

Pulse Sequence: s2pu1

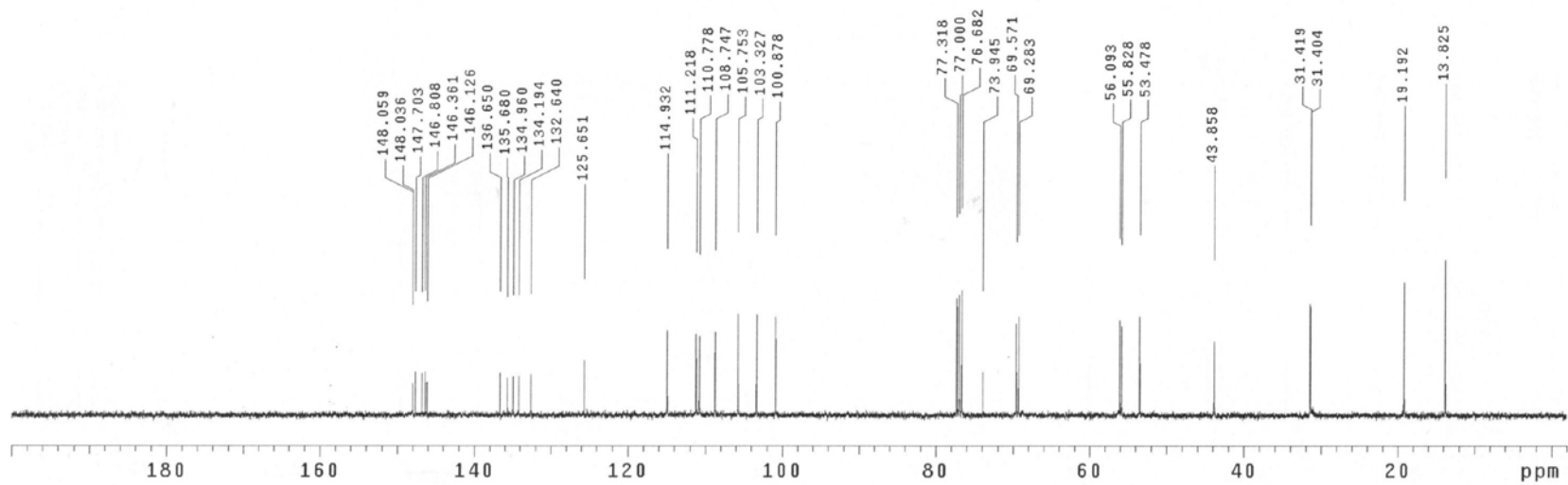
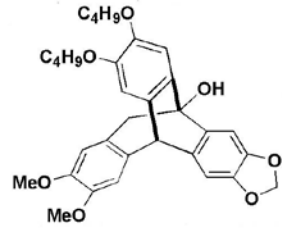
UNITYplus-400 "unity400"

Date: Apr 11 2019

Solvent: CDCl₃

Ambient temperature

Total 1824 repetitions



Compound 5y (¹H-NMR spectral data)

SH[322]5aa

Pulse Sequence: s2pu1

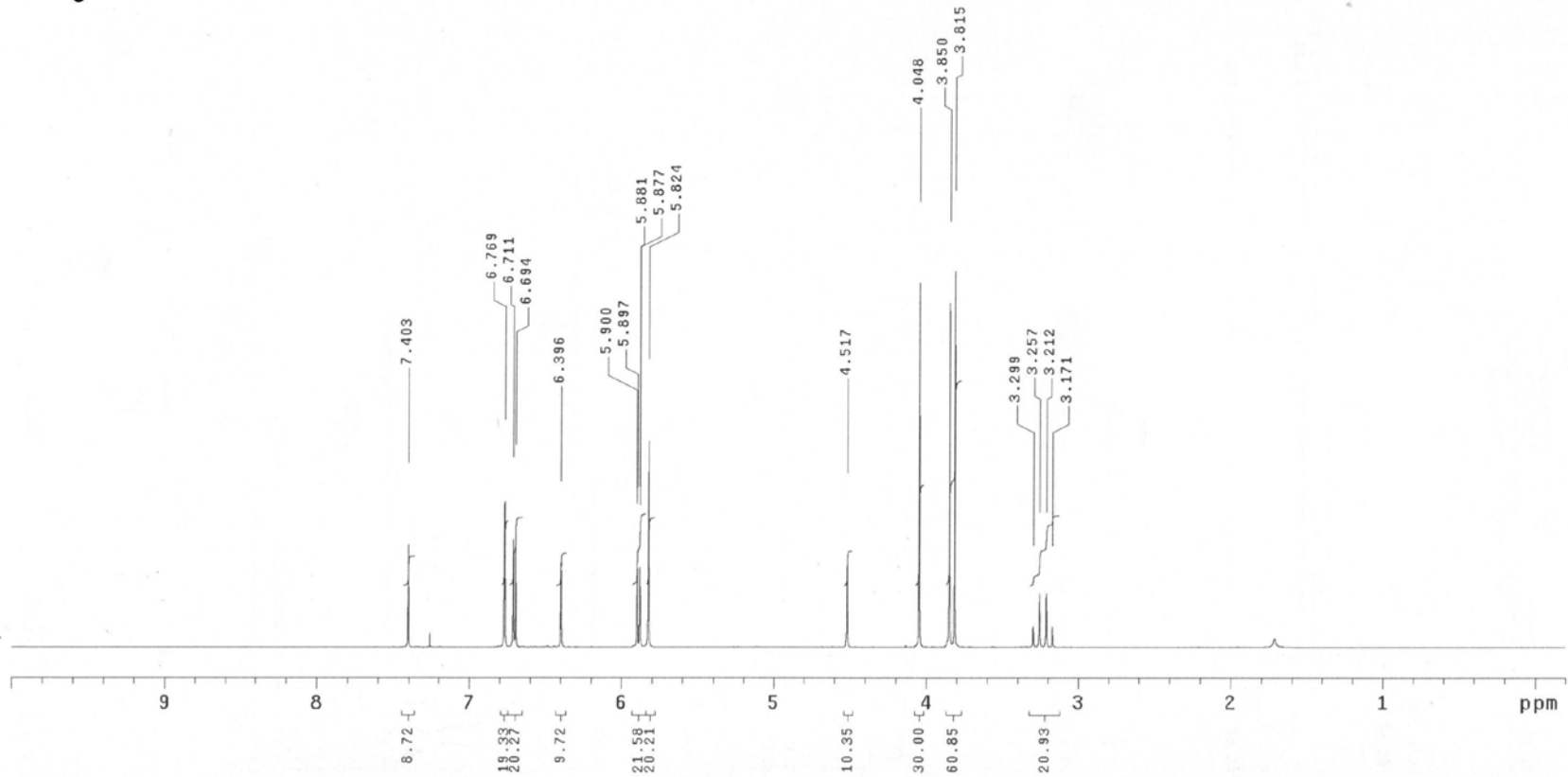
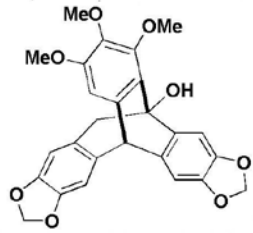
UNITYplus-400 "unity400"

Date: Apr 16 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5y (¹³C-NMR spectral data)

SH[322]5aa

Pulse Sequence: s2pu1

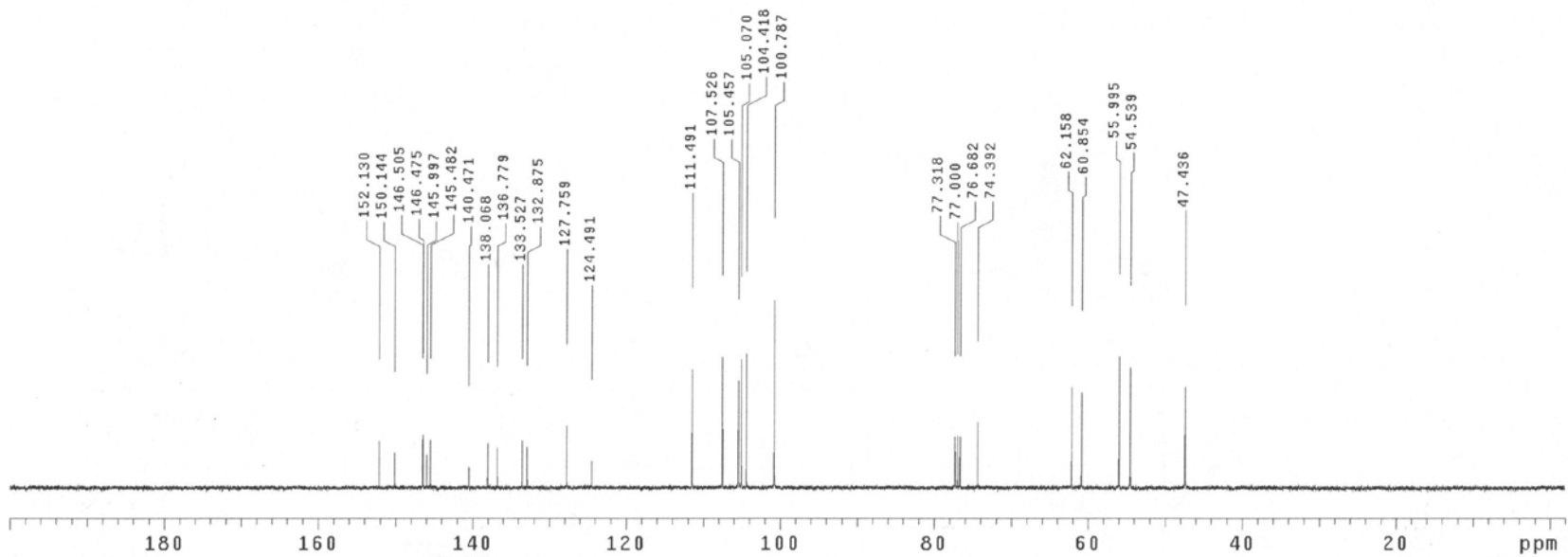
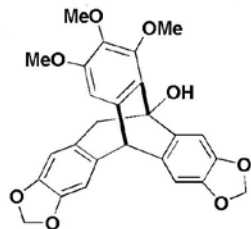
UNITYplus-400 "unity400"

Date: Apr 16 2019

Solvent: CDCl₃

Ambient temperature

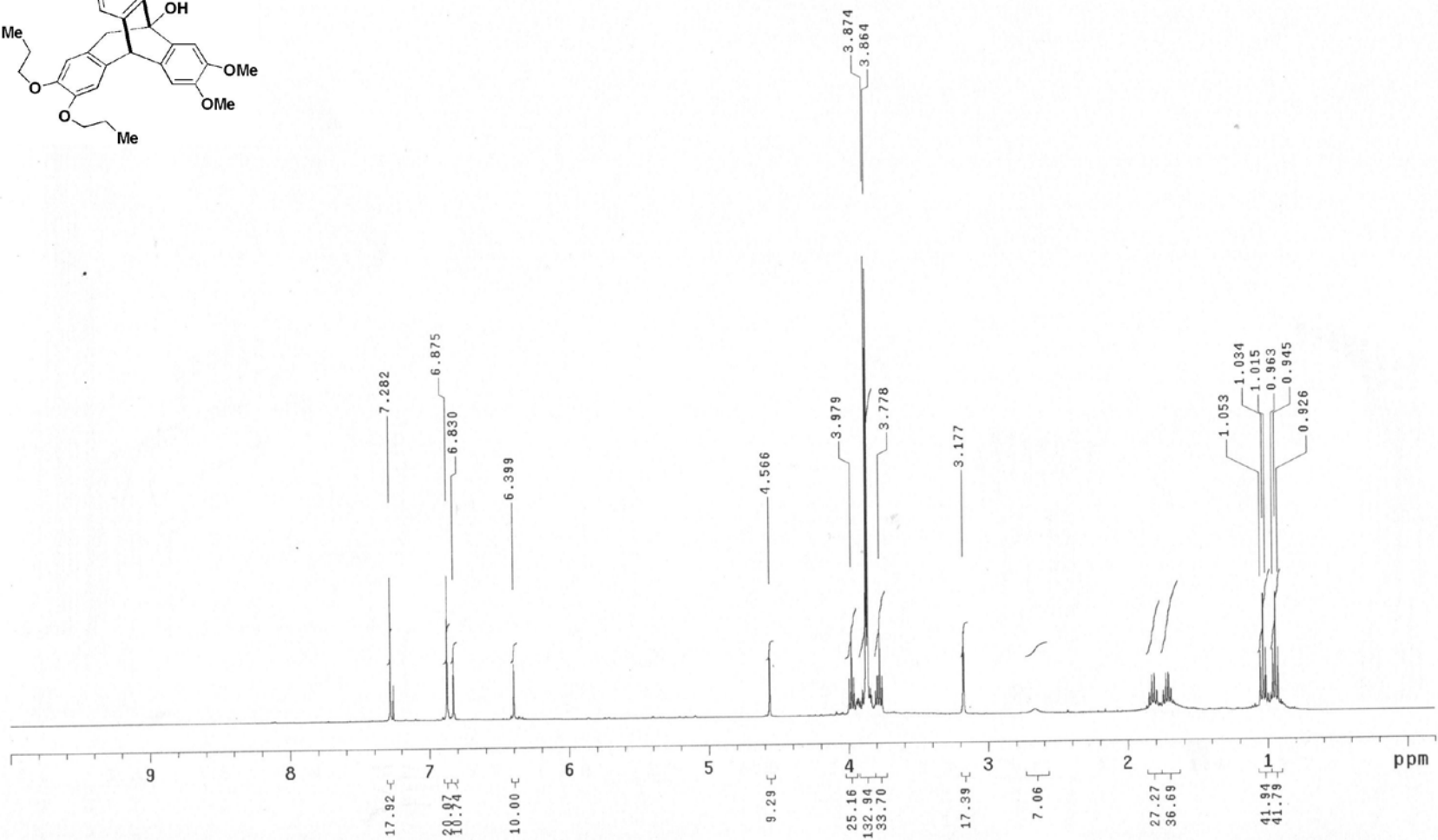
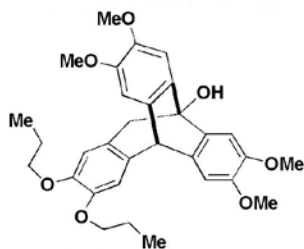
Total 464 repetitions



Compound 5z (¹H-NMR spectral data)

SH[3.2.2]5Z

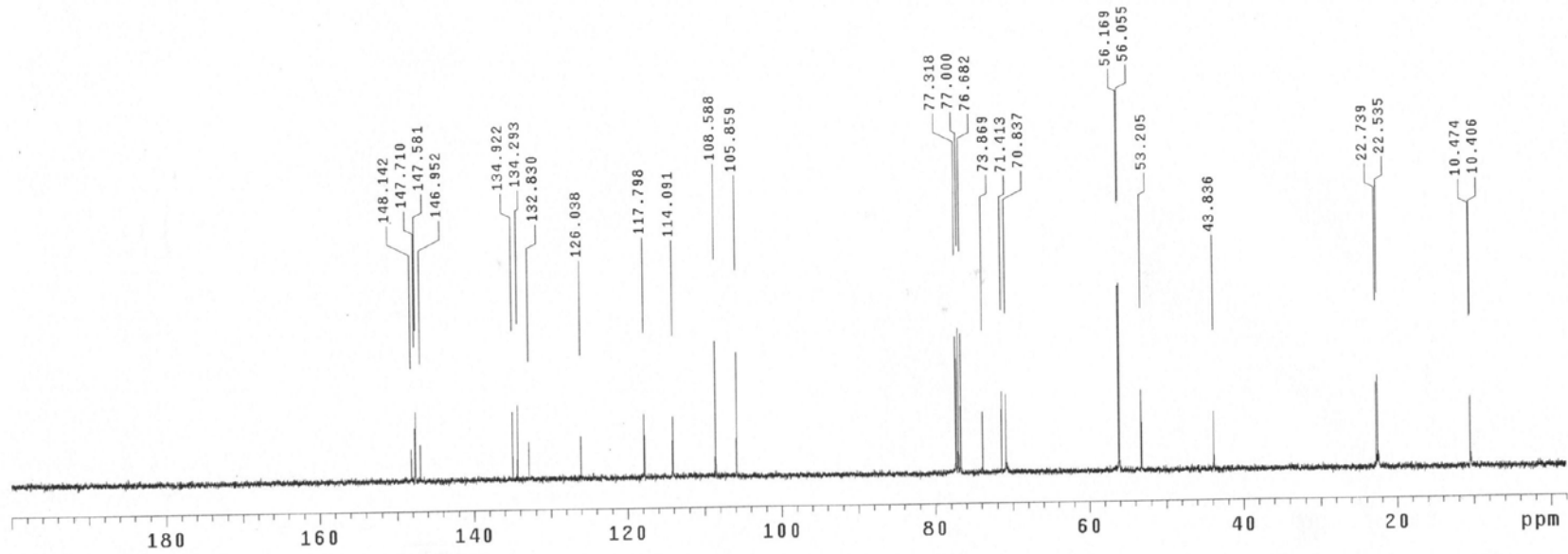
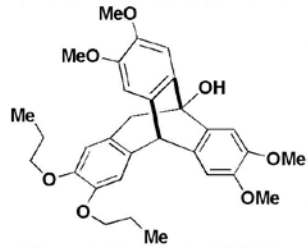
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Feb 12 2019
 Solvent: CDCl3
 Ambient temperature
 Total 32 repetitions



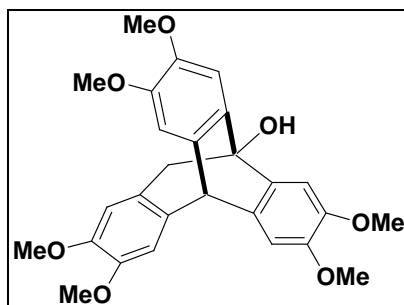
Compound 5z (¹³C-NMR spectral data)

SH[3.2.2]5Z

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Feb 12 2019
Solvent: CDCl₃
Ambient temperature
Total 2080 repetitions

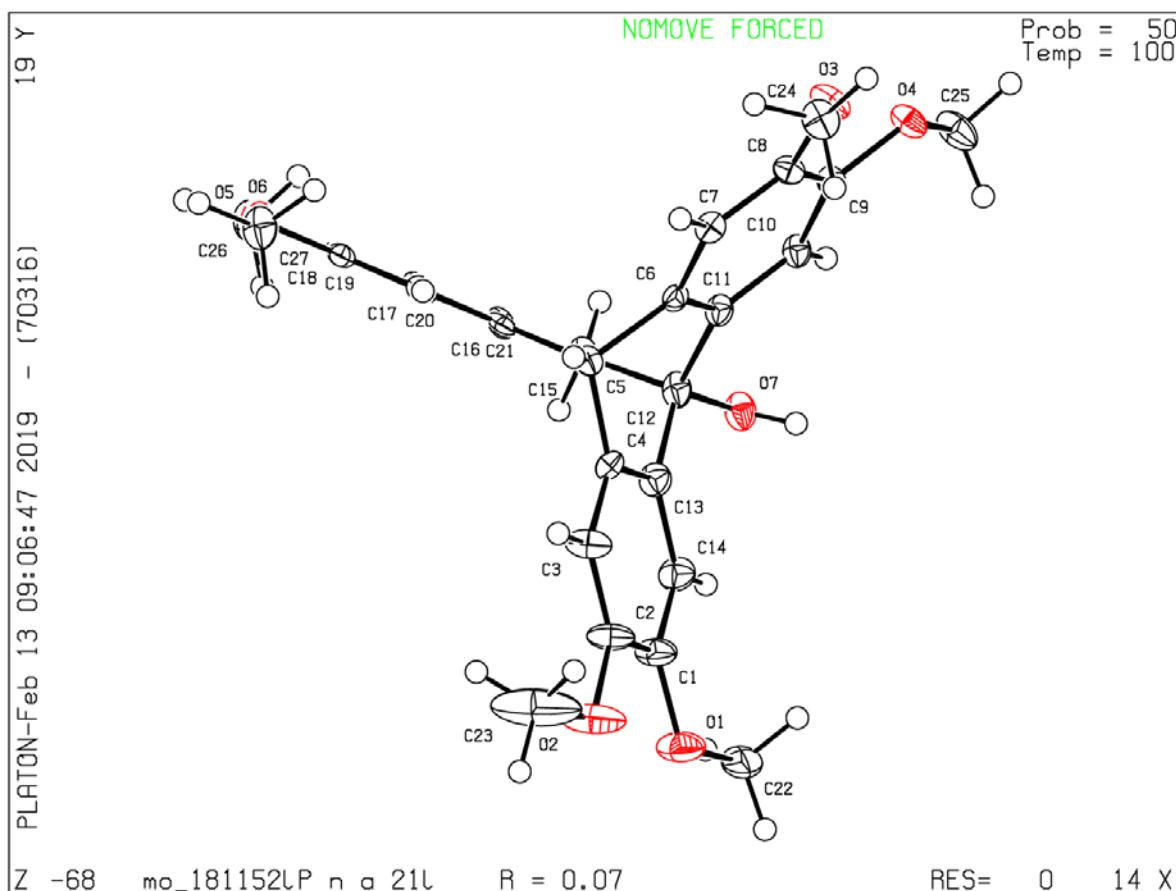


X-ray crystal data of compound 5a (CCDC 1938168)



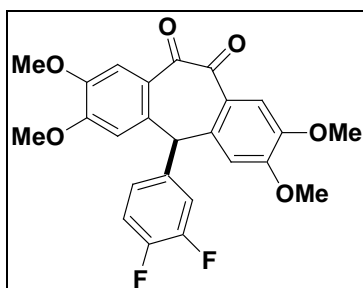
Sample preparation : A solution of compound **5a** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



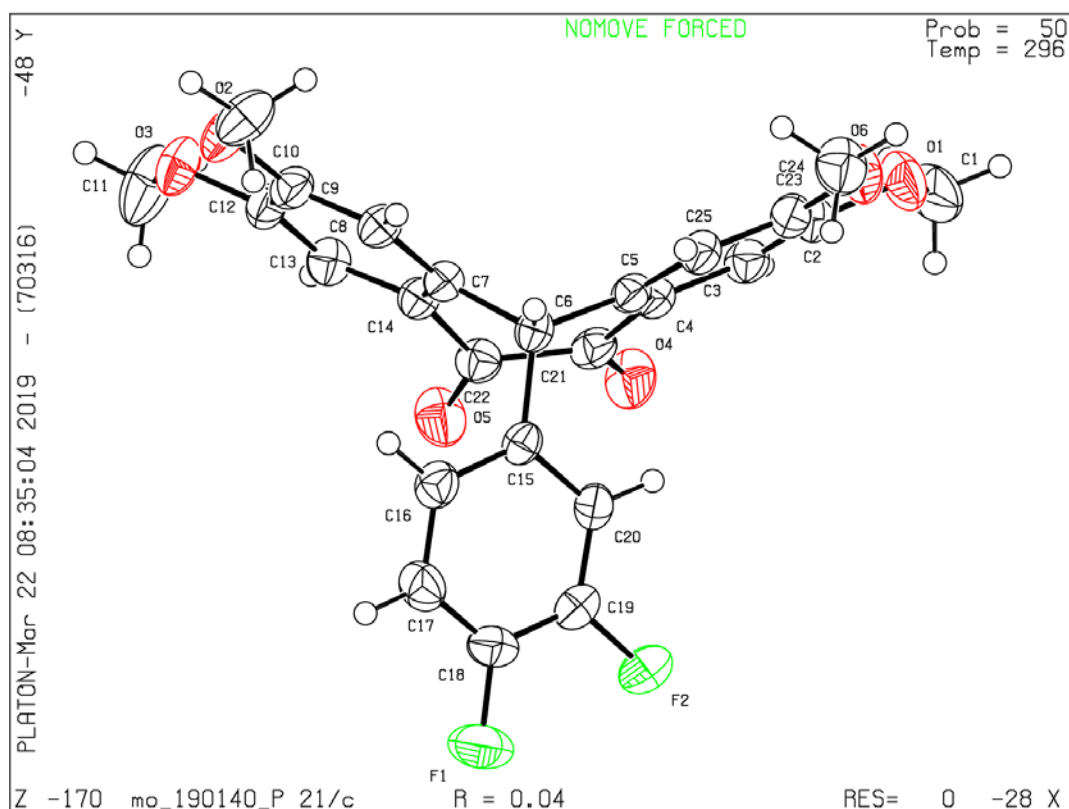
Empirical formula	C ₂₇ H ₂₈ O ₇
Formula weight	464.49
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Orthorhombic
Space group	P n a 21
Unit cell dimensions	a = 15.2055(18) Å α = 90°. b = 17.986(2) Å β = 90°. c = 8.4082(11) Å γ = 90°.
Volume	2299.5(5) Å ³
Z	4
Density (calculated)	1.342 Mg/m ³
Absorption coefficient	0.097 mm ⁻¹
F(000)	984
Crystal size	0.10 x 0.08 x 0.02 mm ³
Theta range for data collection	1.754 to 26.510°.
Index ranges	-19<=h<=18, -22<=k<=21, -10<=l<=10
Reflections collected	24767
Independent reflections	4680 [R(int) = 0.0980]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.6687
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4680 / 1 / 315
Goodness-of-fit on F ²	1.053
Final R indices [I>2sigma(I)]	R1 = 0.0690, wR2 = 0.1332
R indices (all data)	R1 = 0.1004, wR2 = 0.1476
Absolute structure parameter	-2(2)
Extinction coefficient	n/a
Largest diff. peak and hole	0.401 and -0.249 e.Å ⁻³

X-ray crystal data of compound 5w-2 (CCDC 1938169)



Sample preparation : A solution of compound **5w-2** (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 ₂₅ H ₂₀ F ₂ O ₆
Formula weight	454.41
Temperature	296(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2 ₁ /c
Unit cell dimensions	a = 11.9481(8) Å α = 90°. b = 15.6973(9) Å β = 111.052(3)°. c = 12.0408(7) Å γ = 90°.
Volume	2107.6(2) Å ³
Z	4
Density (calculated)	1.432 Mg/m ³
Absorption coefficient	0.113 mm ⁻¹
F(000)	944
Crystal size	0.17 x 0.14 x 0.06 mm ³
Theta range for data collection	1.826 to 26.446°.
Index ranges	-13 ≤ h ≤ 14, -19 ≤ k ≤ 19, -15 ≤ l ≤ 12
Reflections collected	29528
Independent reflections	4328 [R(int) = 0.0422]
Completeness to theta = 25.242°	100.0 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7454 and 0.6846
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4328 / 0 / 302
Goodness-of-fit on F ²	1.004
Final R indices [I > 2σ(I)]	R1 = 0.0407, wR2 = 0.0917
R indices (all data)	R1 = 0.0725, wR2 = 0.1061
Extinction coefficient	n/a
Largest diff. peak and hole	0.150 and -0.160 e.Å ⁻³