Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

BF_3·OEt_2-Mediated Nucleophilic Fluorocyclization of Sulfonyl 3-Methylene-oxabenzocyclooctan-6-ones. Diastereocontrolled Synthesis of Benzofused Fluorooxabicyclo[4.2.1]nonanes

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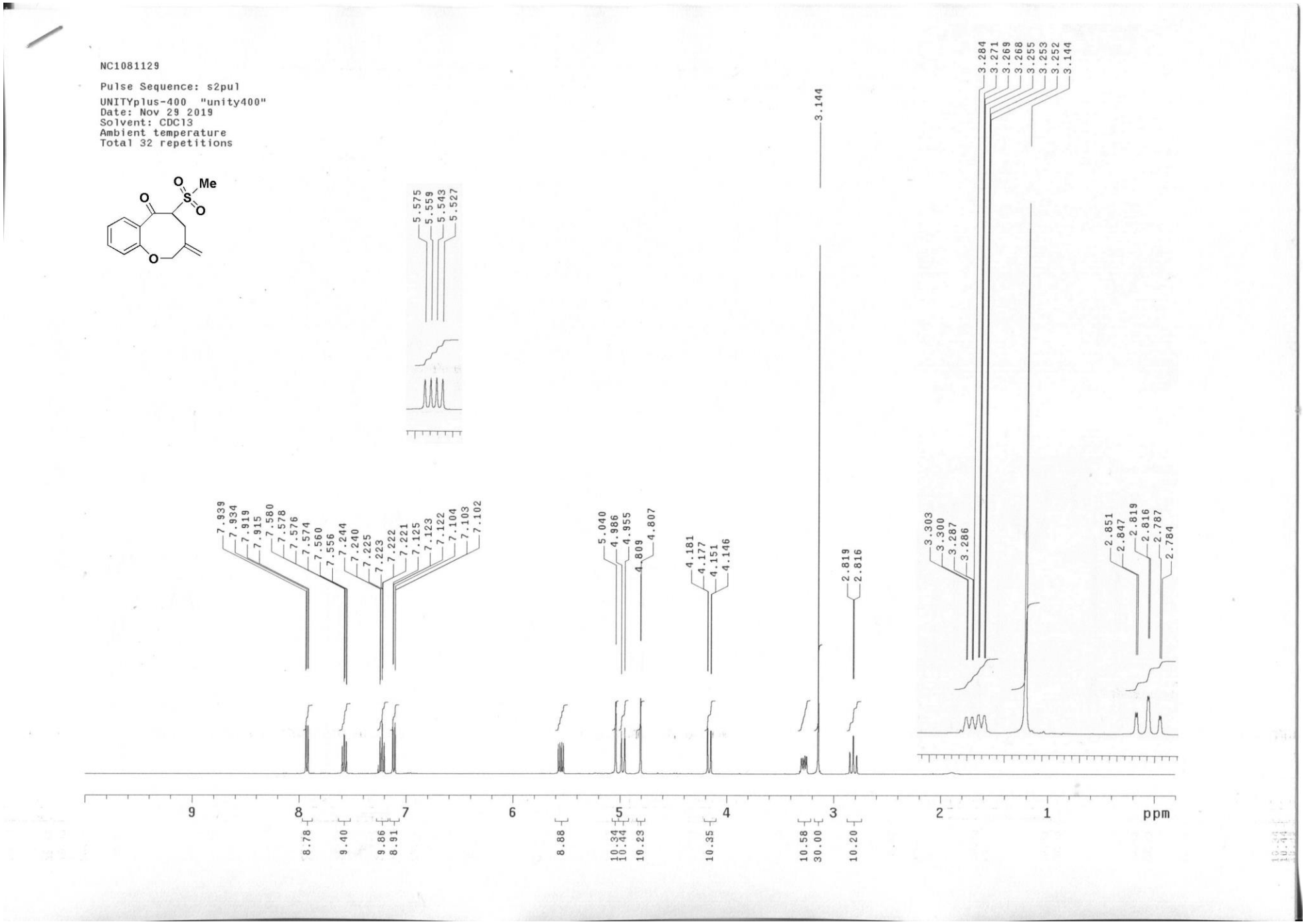
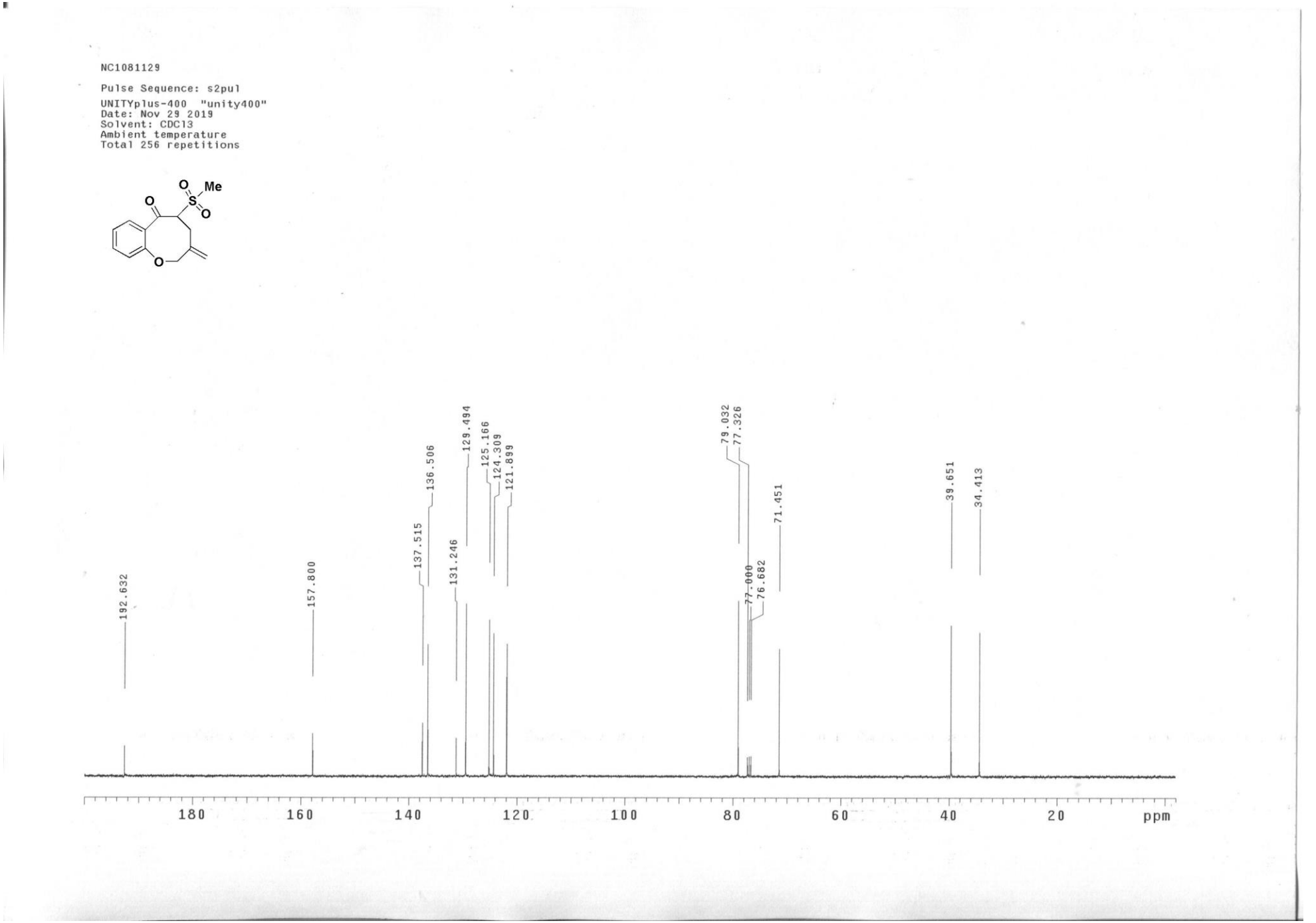
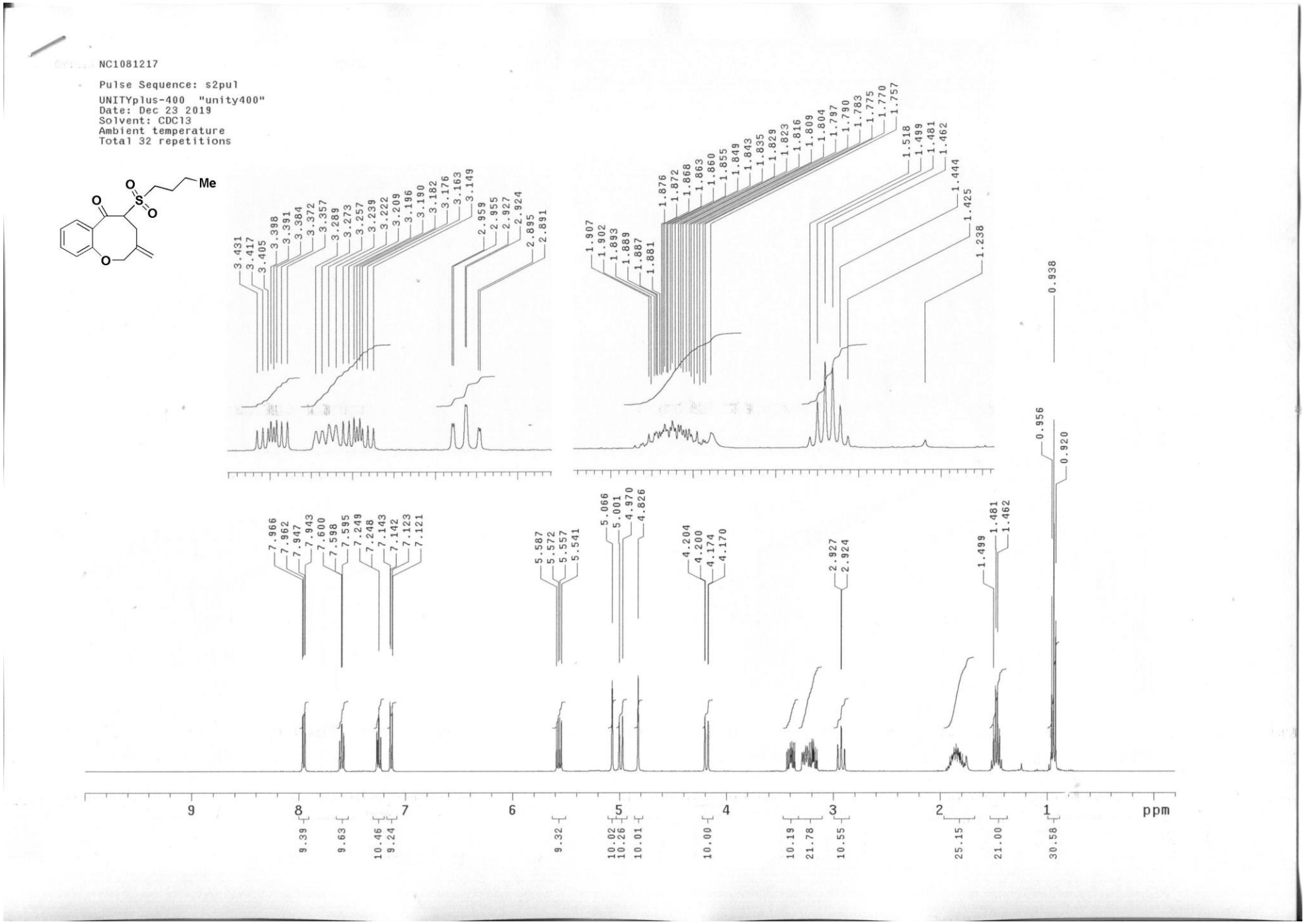
Compound 4a (^{1}H-NMR spectral data)



Compound 4a (^{13}C-NMR spectral data)



Compound 4b (^{1}H-NMR spectral data)



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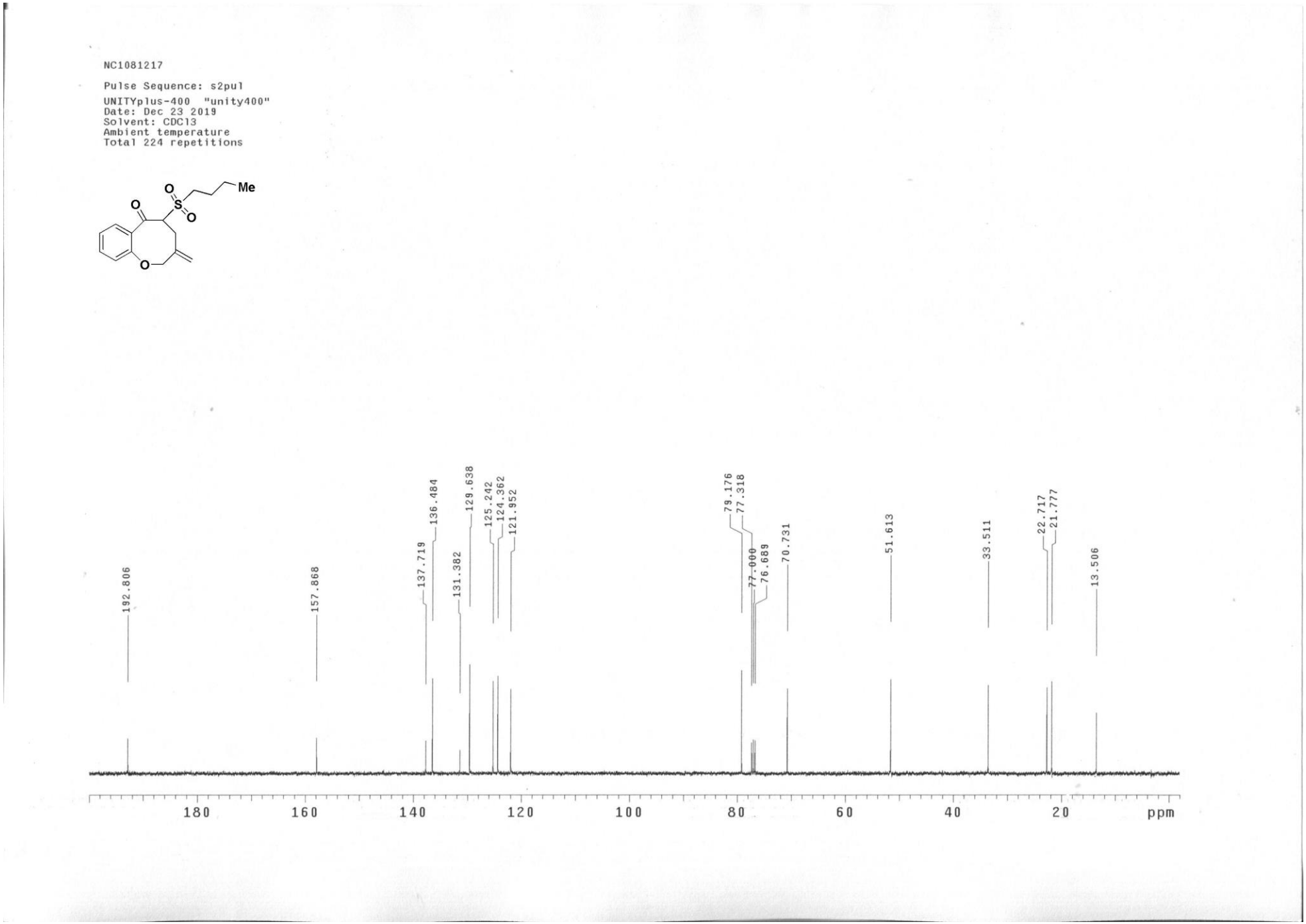

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NC1081128

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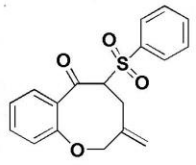
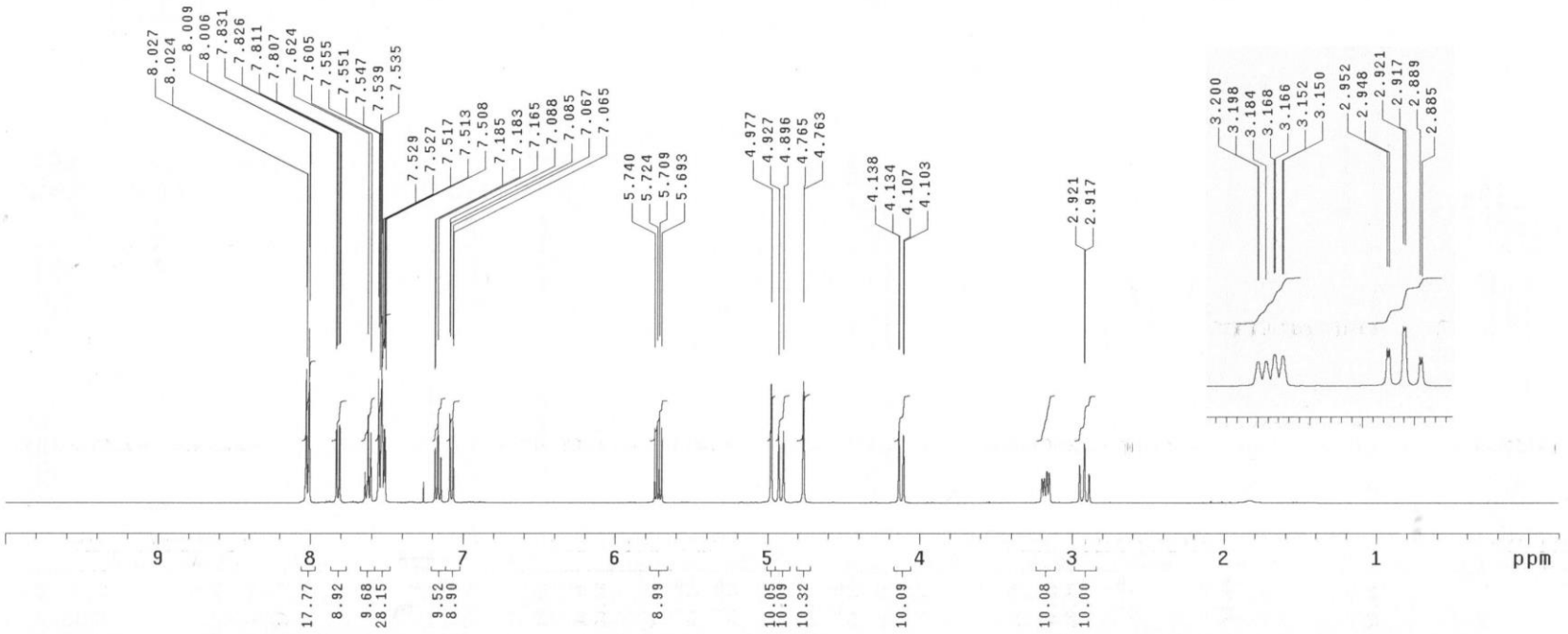
Date: Nov 28 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions





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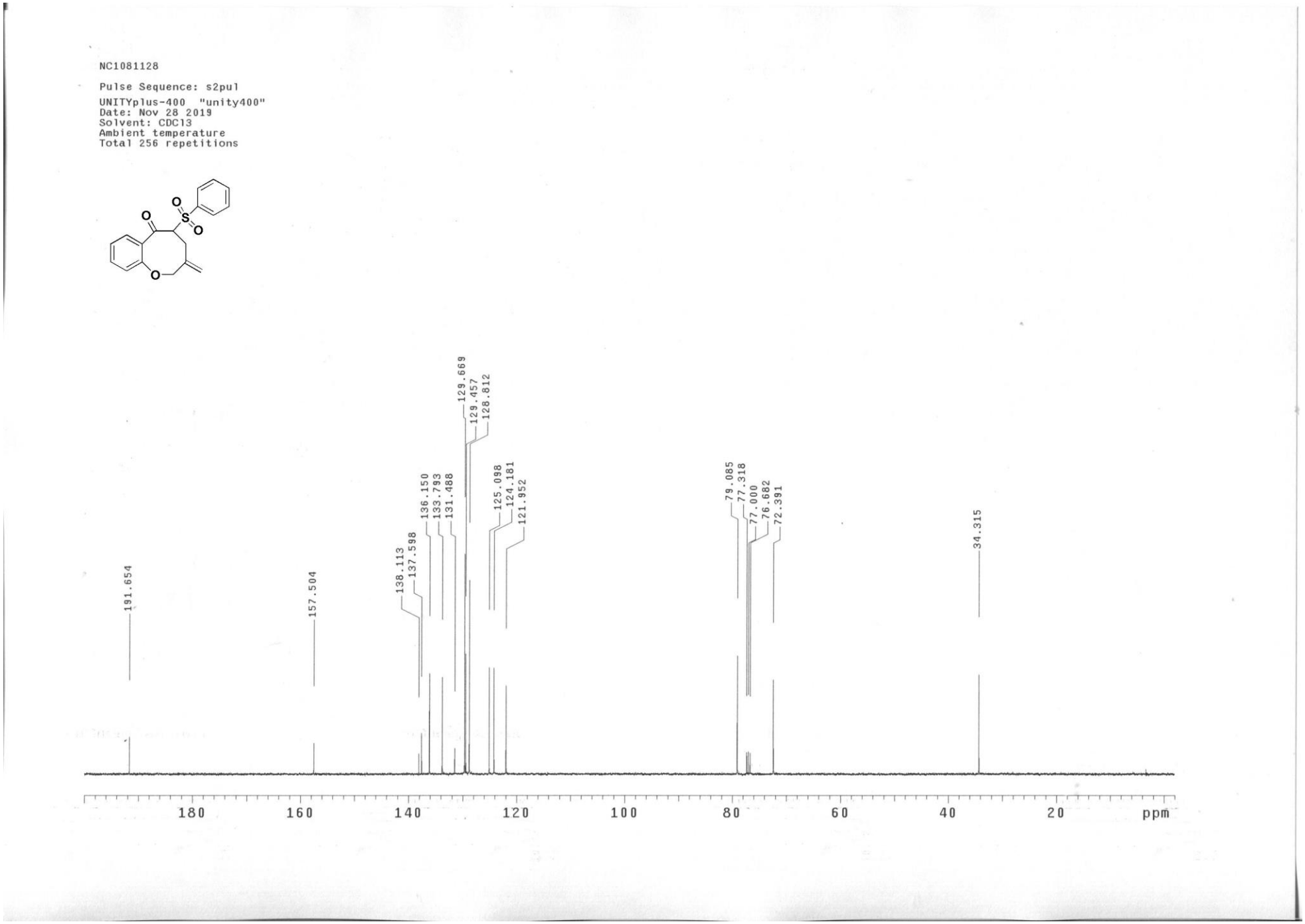

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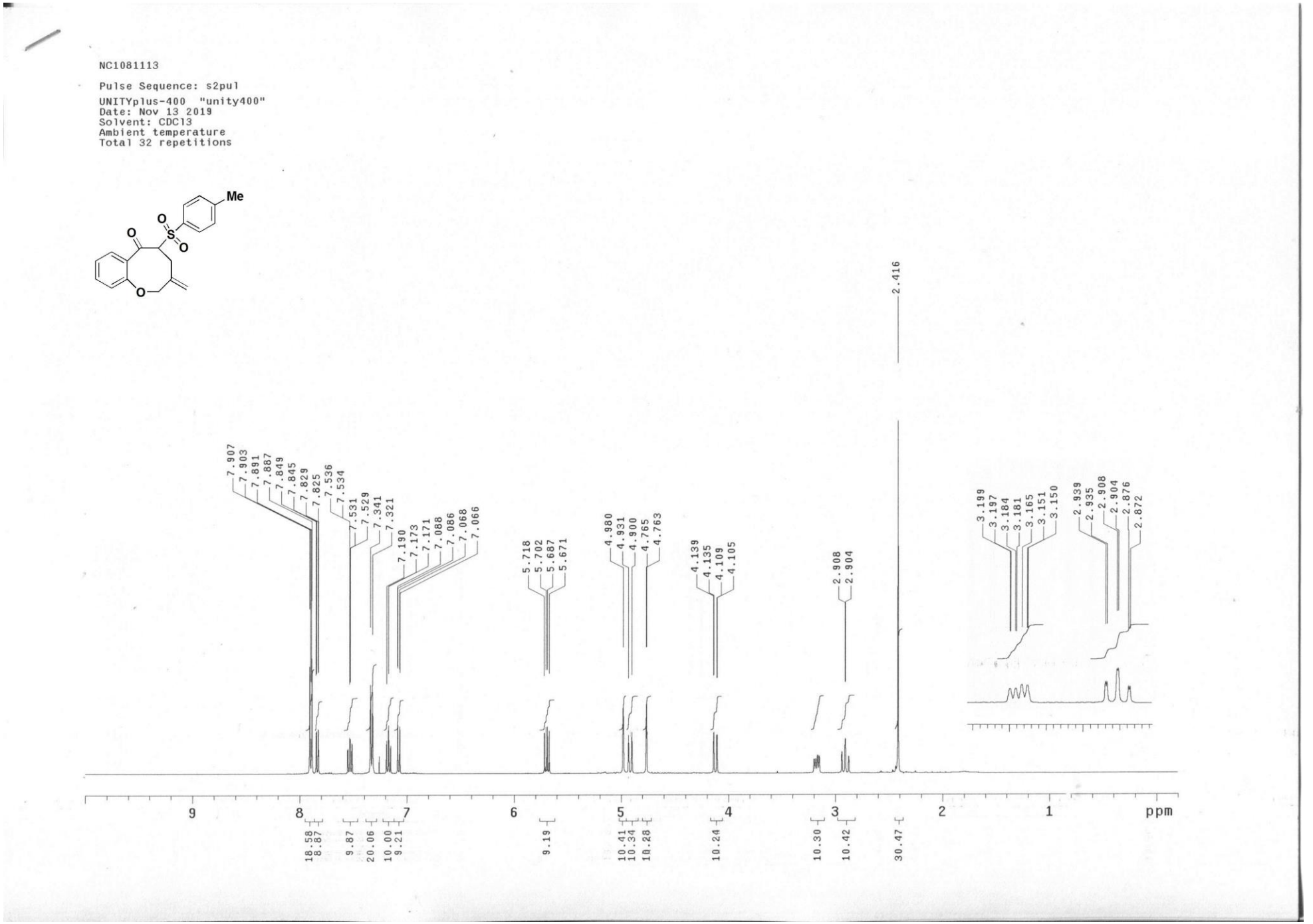

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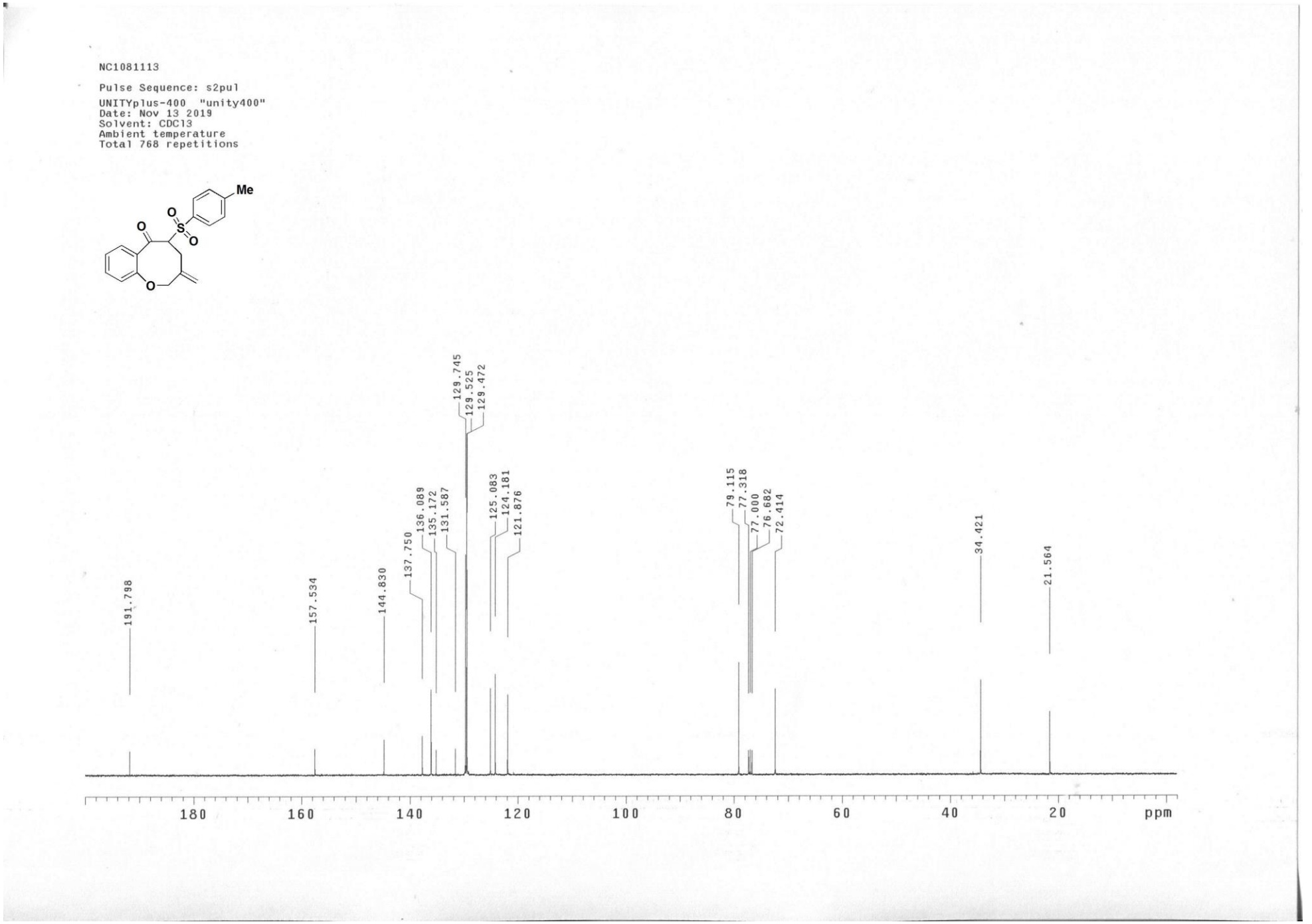

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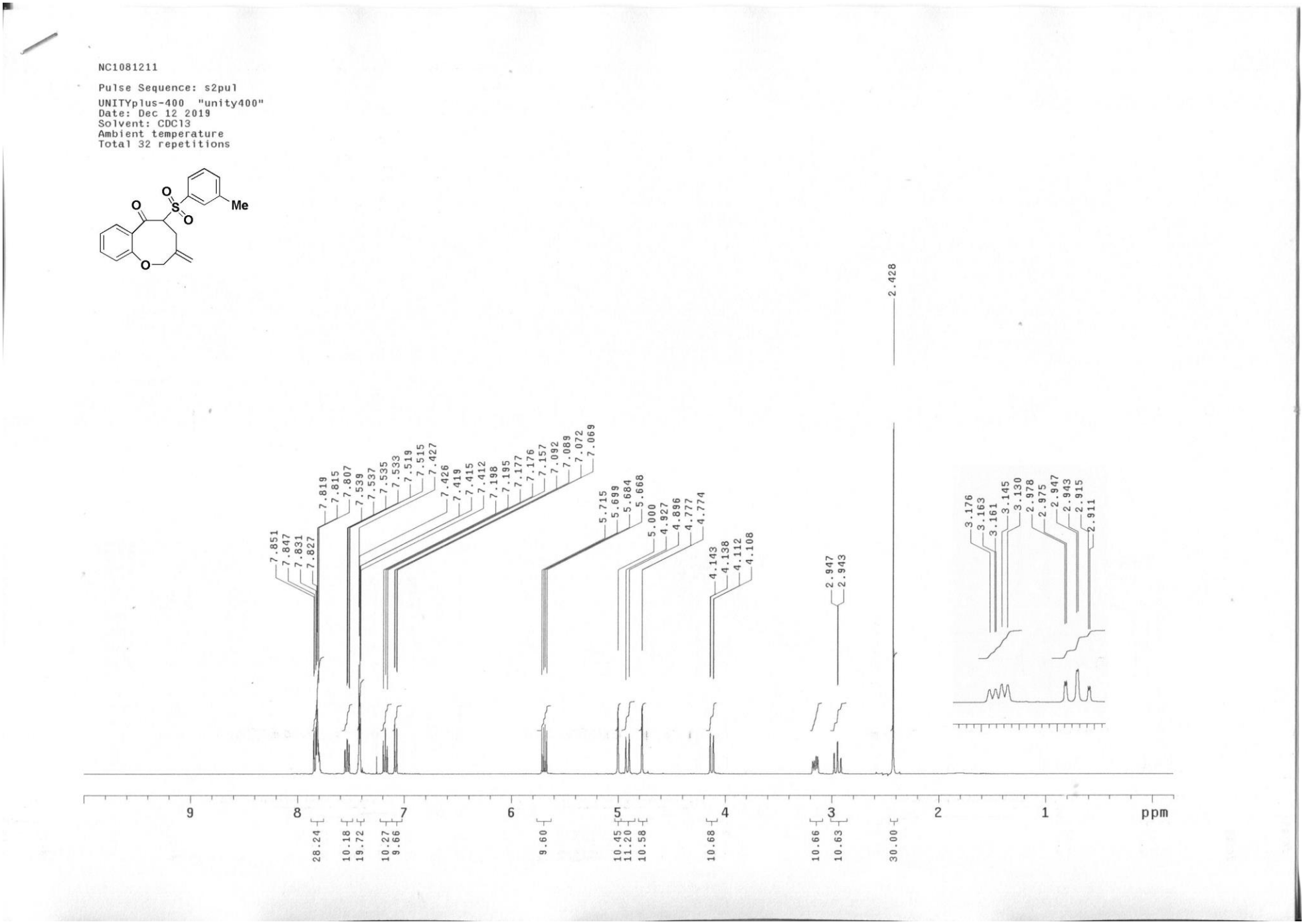

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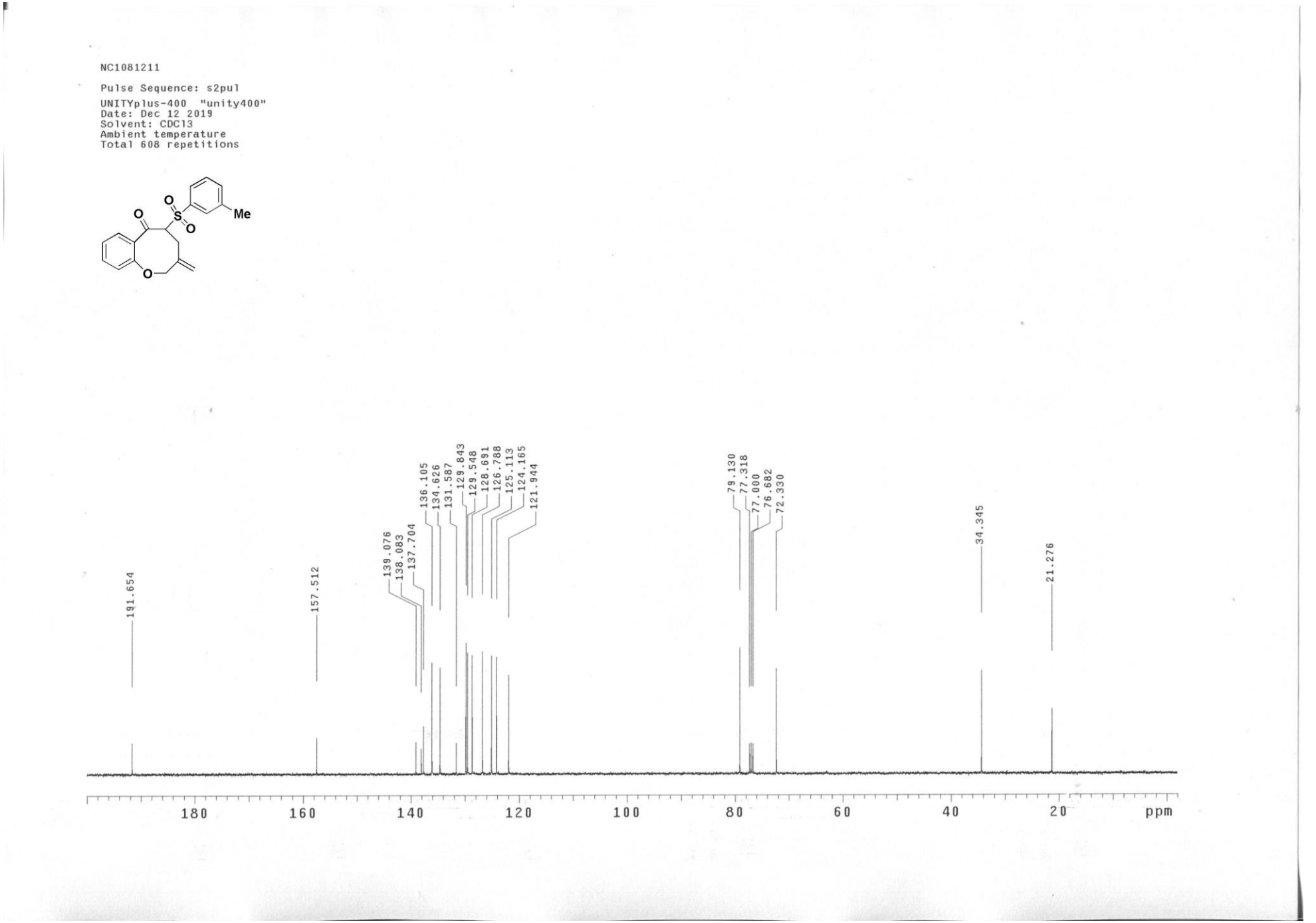

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NC1081202

Pulse Sequence: s2pul

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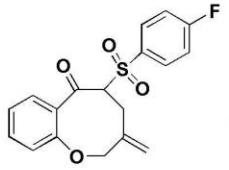
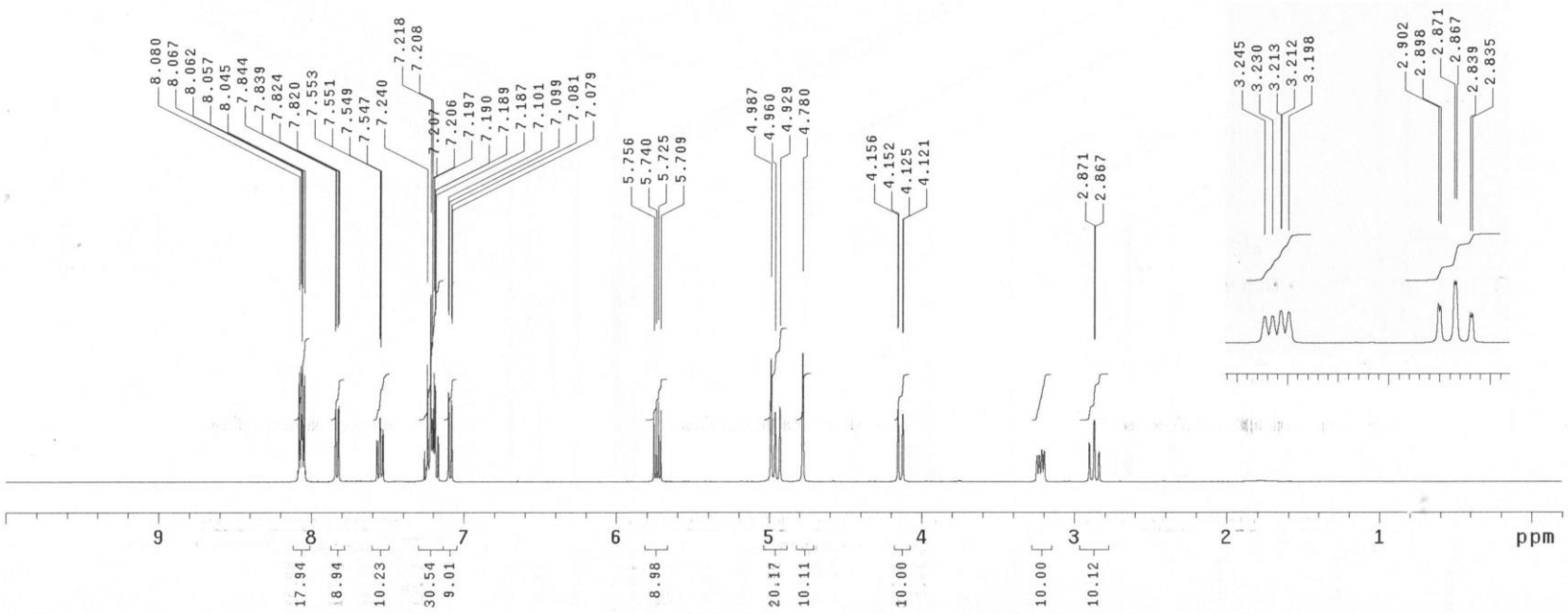
Date: Dec 3 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions





Compound 4f (^{13}C-NMR spectral data)

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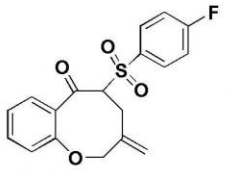
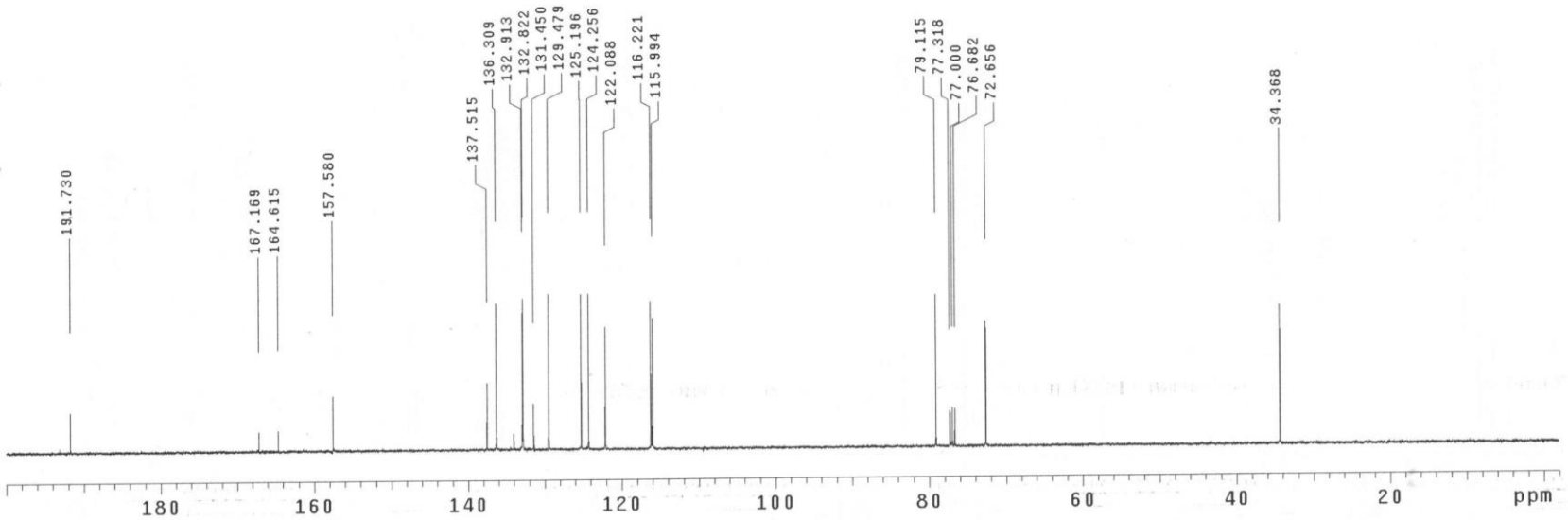
Date: Dec 3 2019
Solvent: CDCl3
Ambient temperature
Total 640 repetitions





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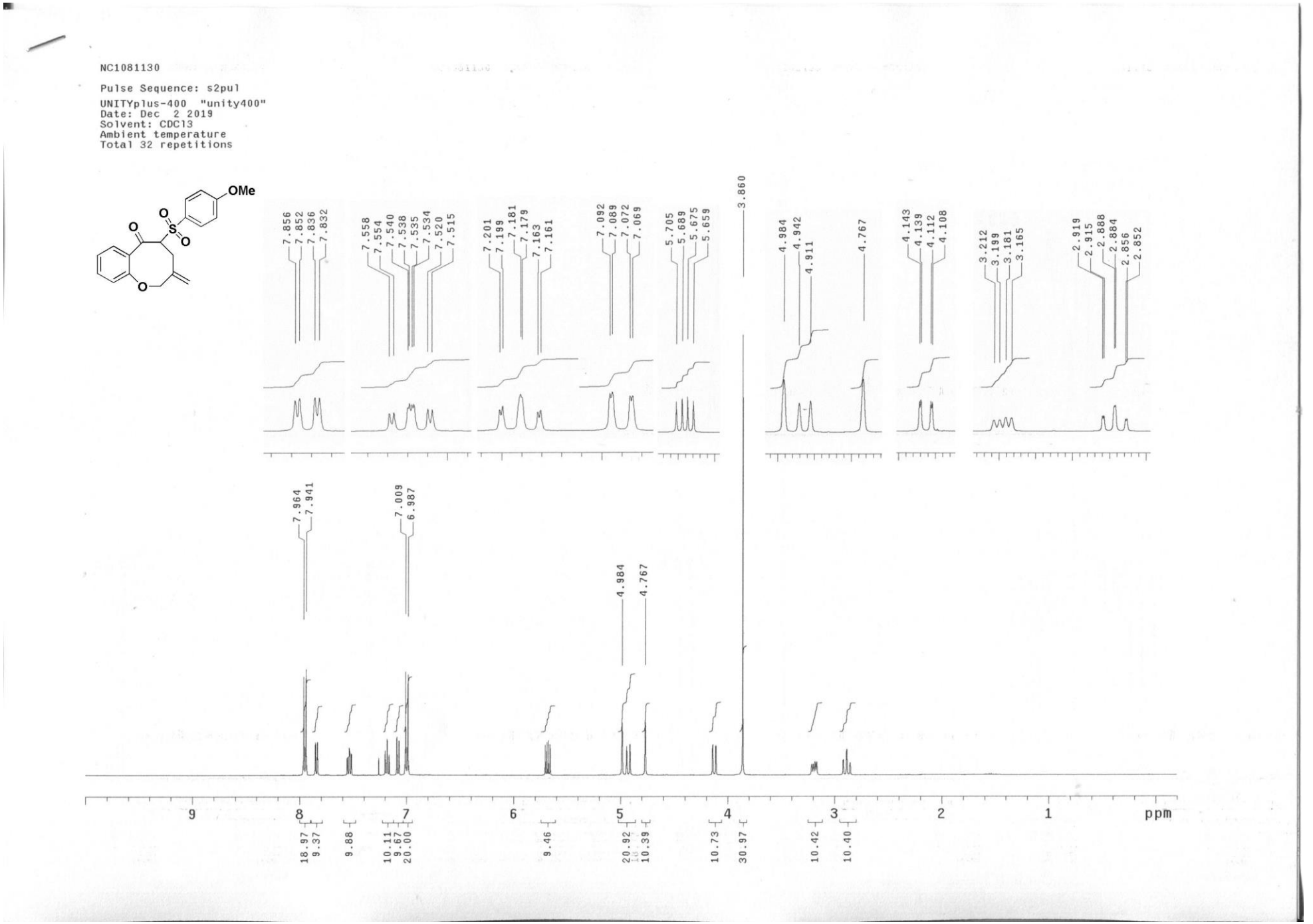

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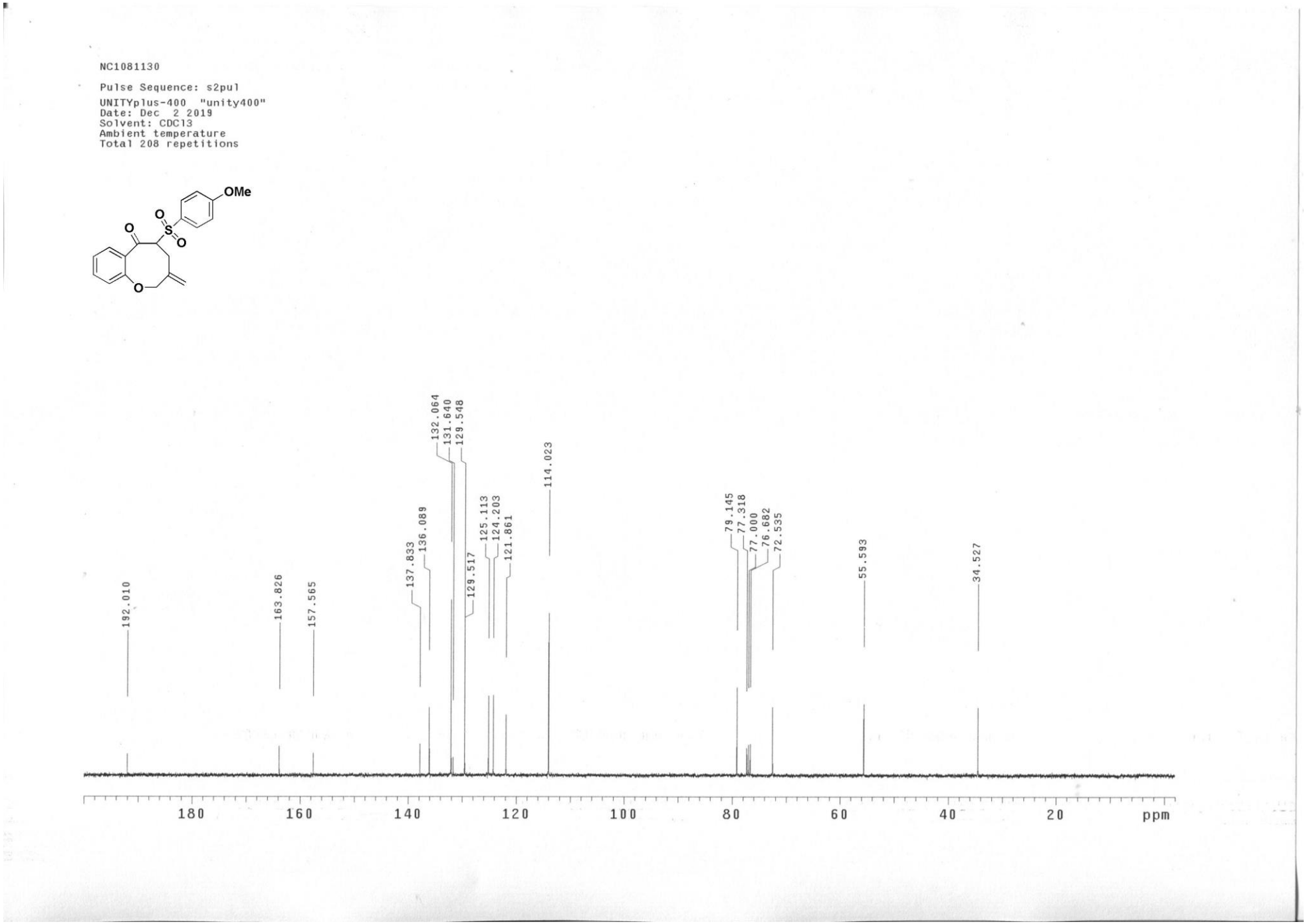

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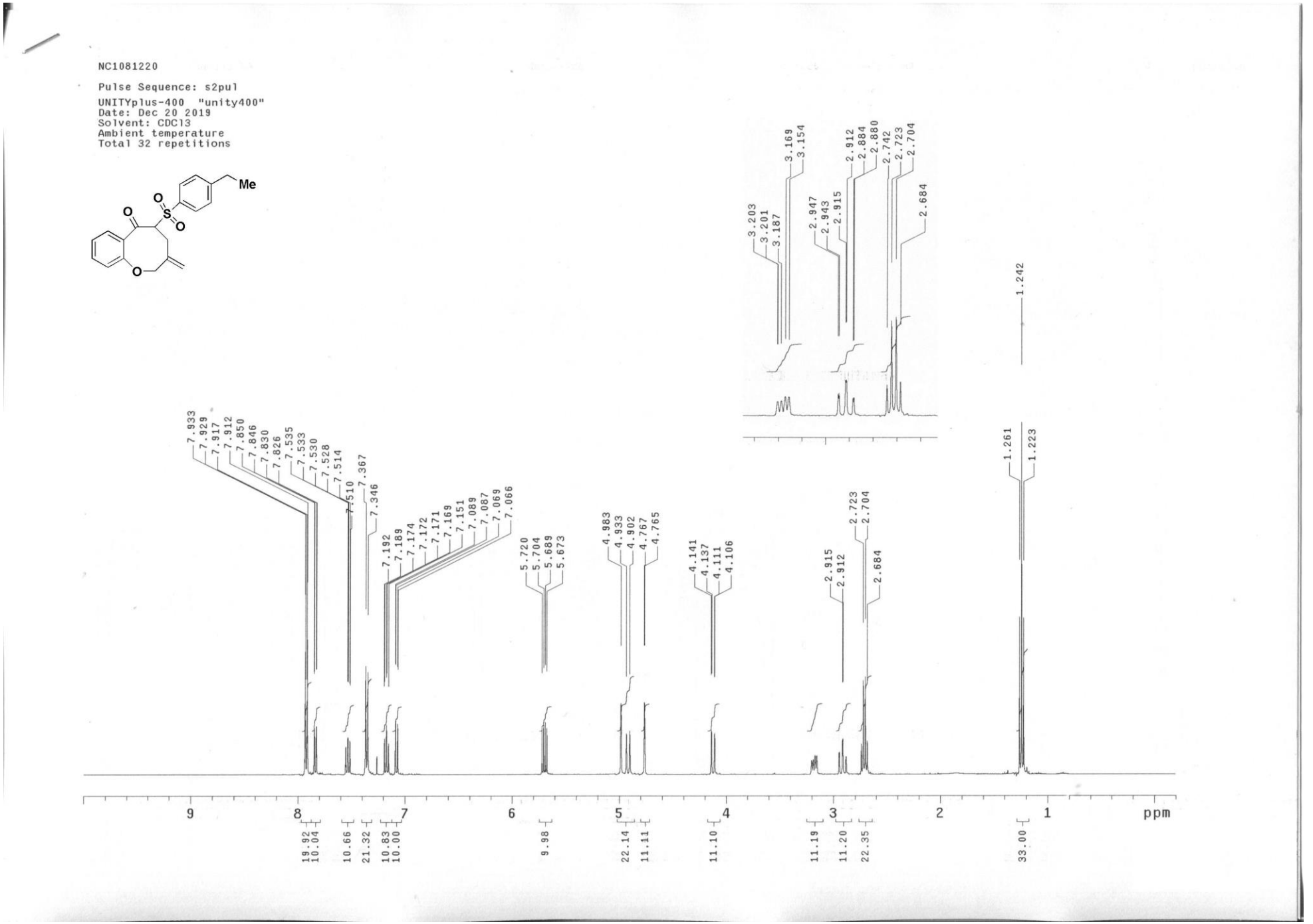

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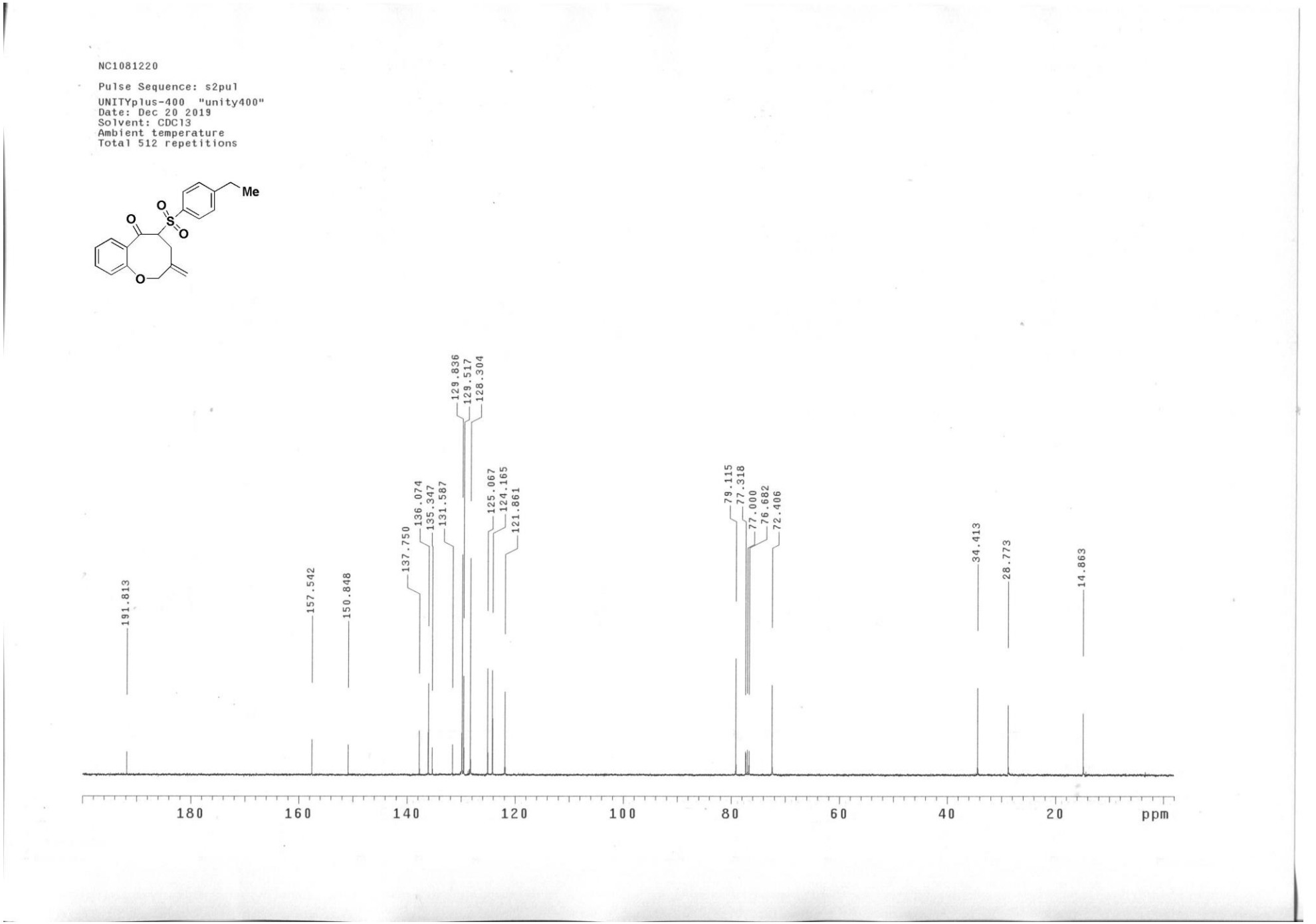

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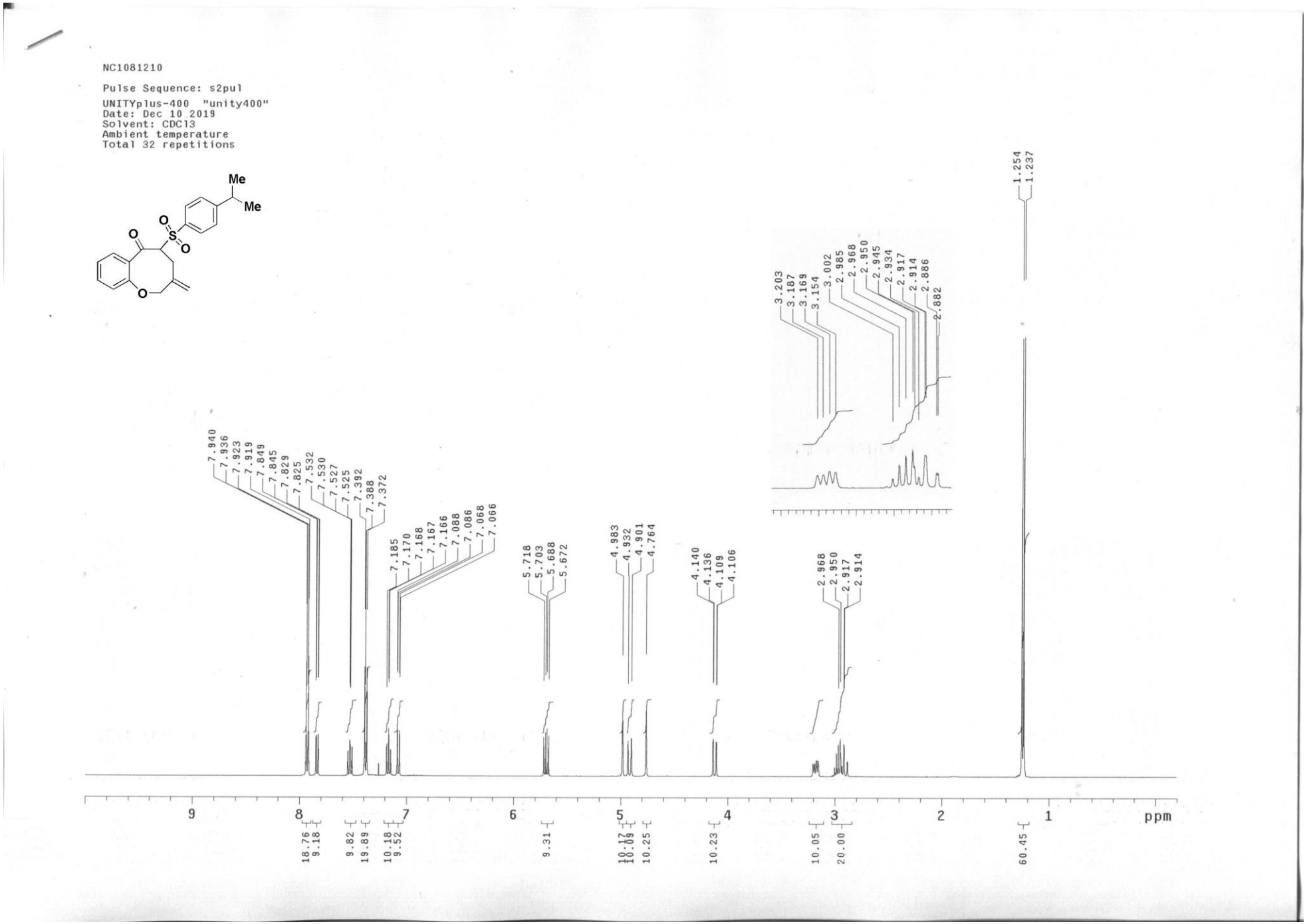

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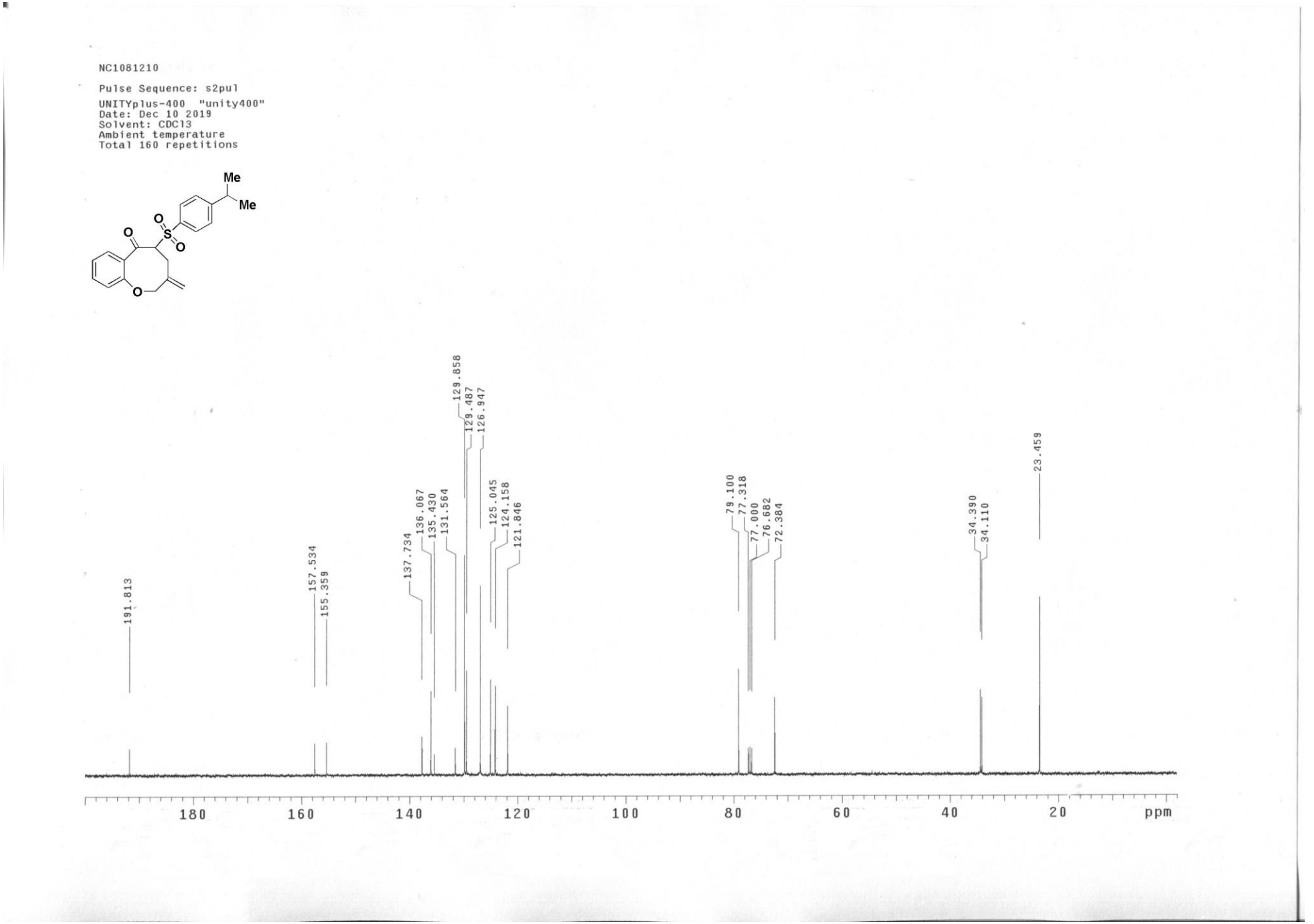

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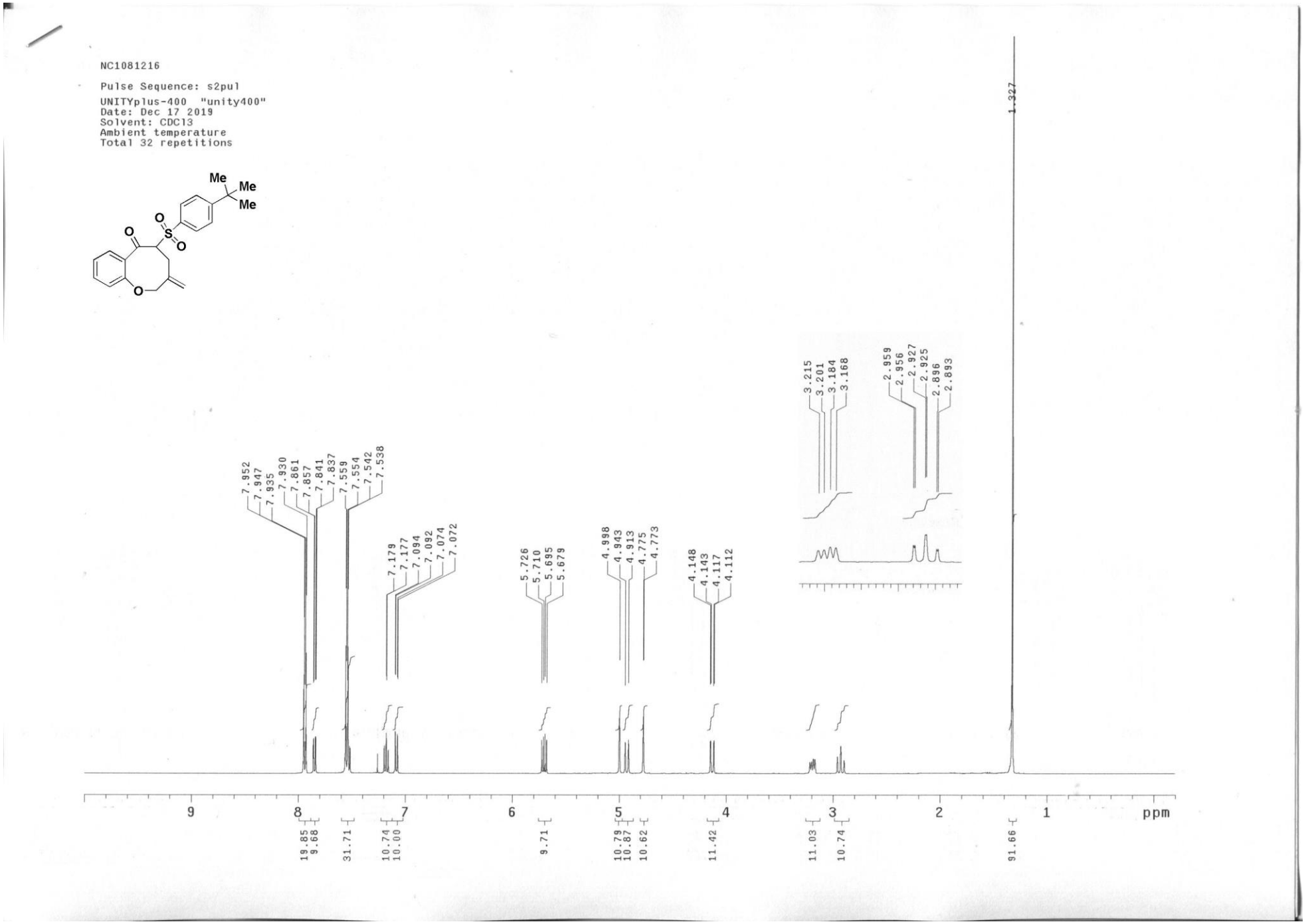

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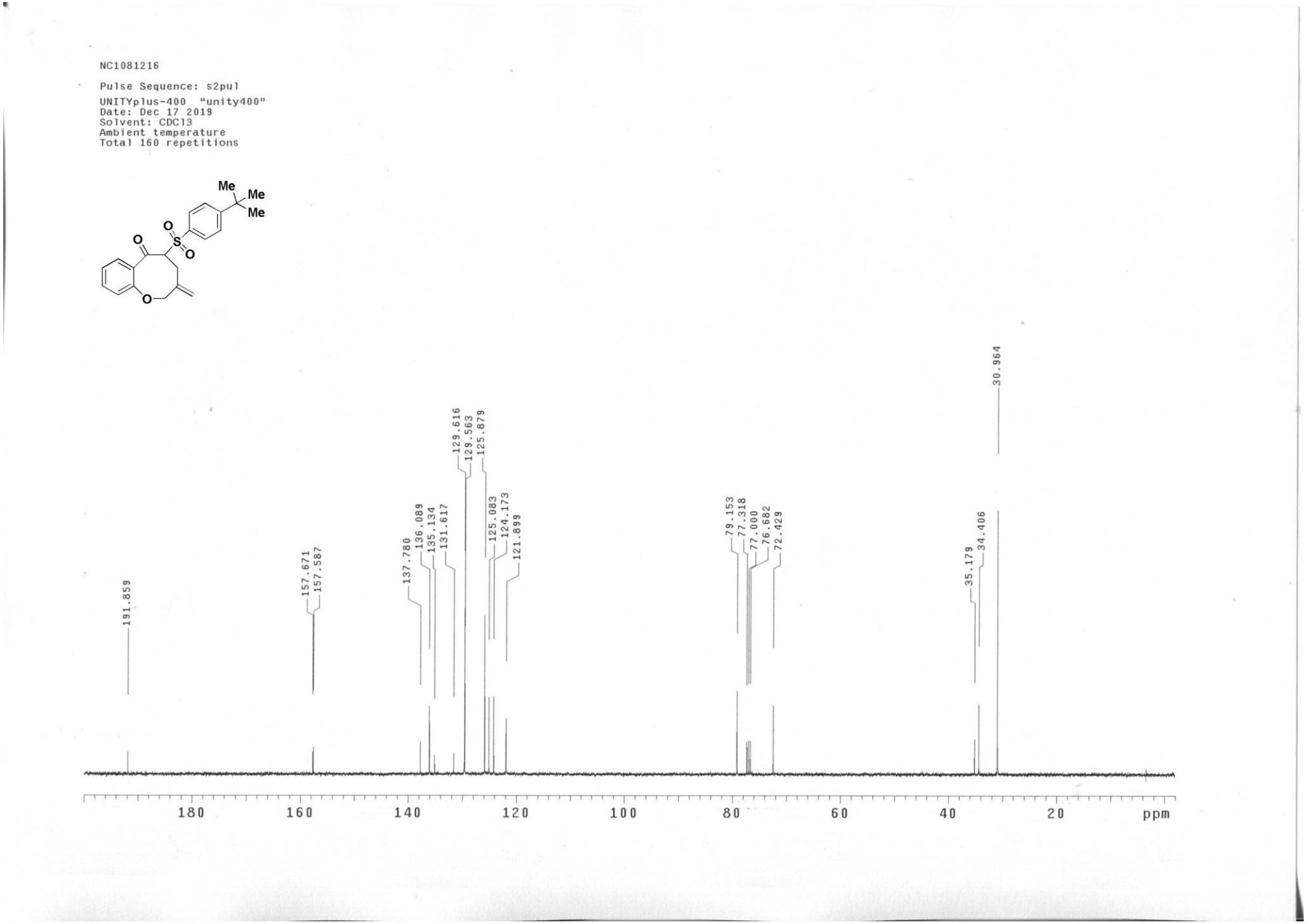

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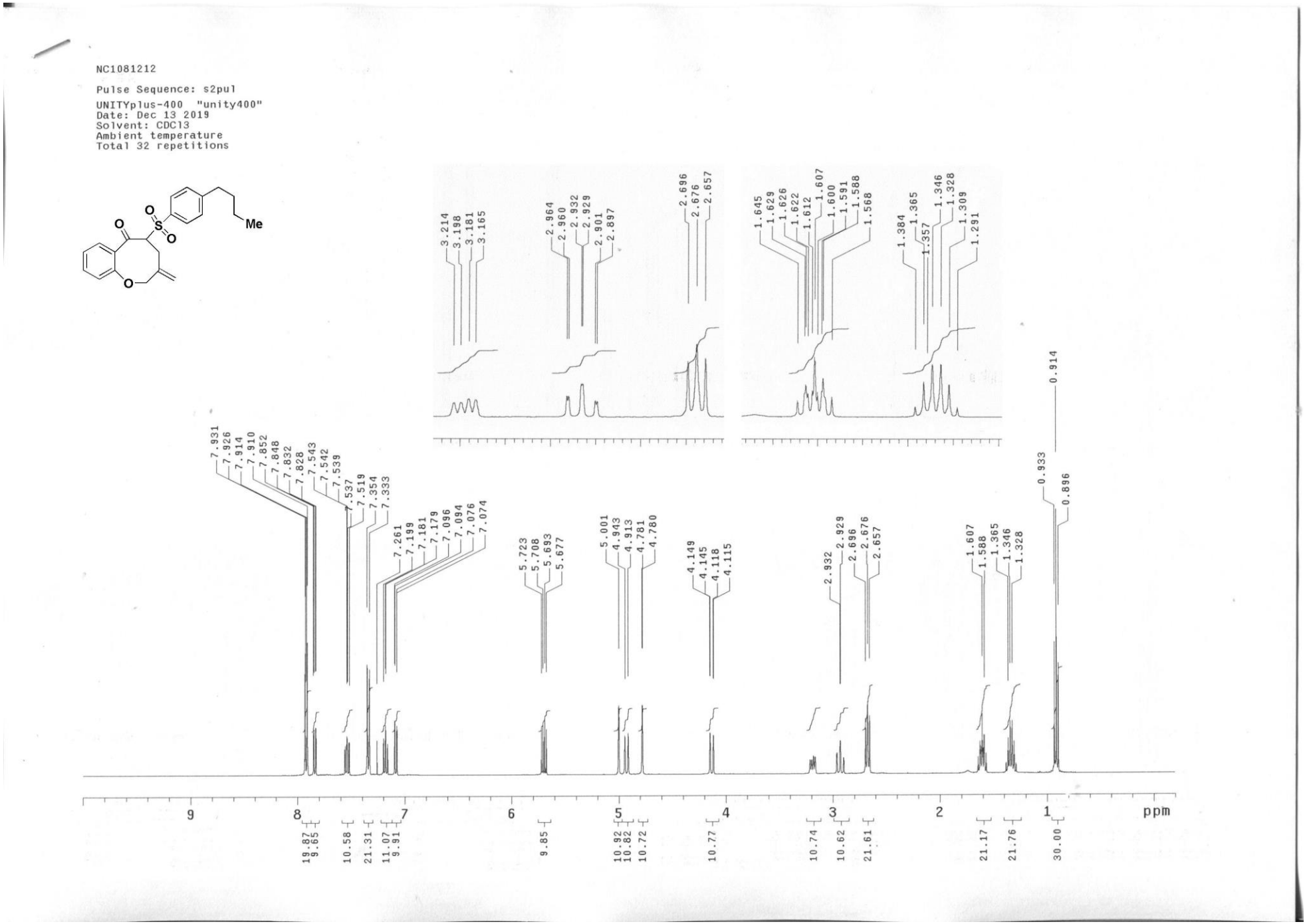

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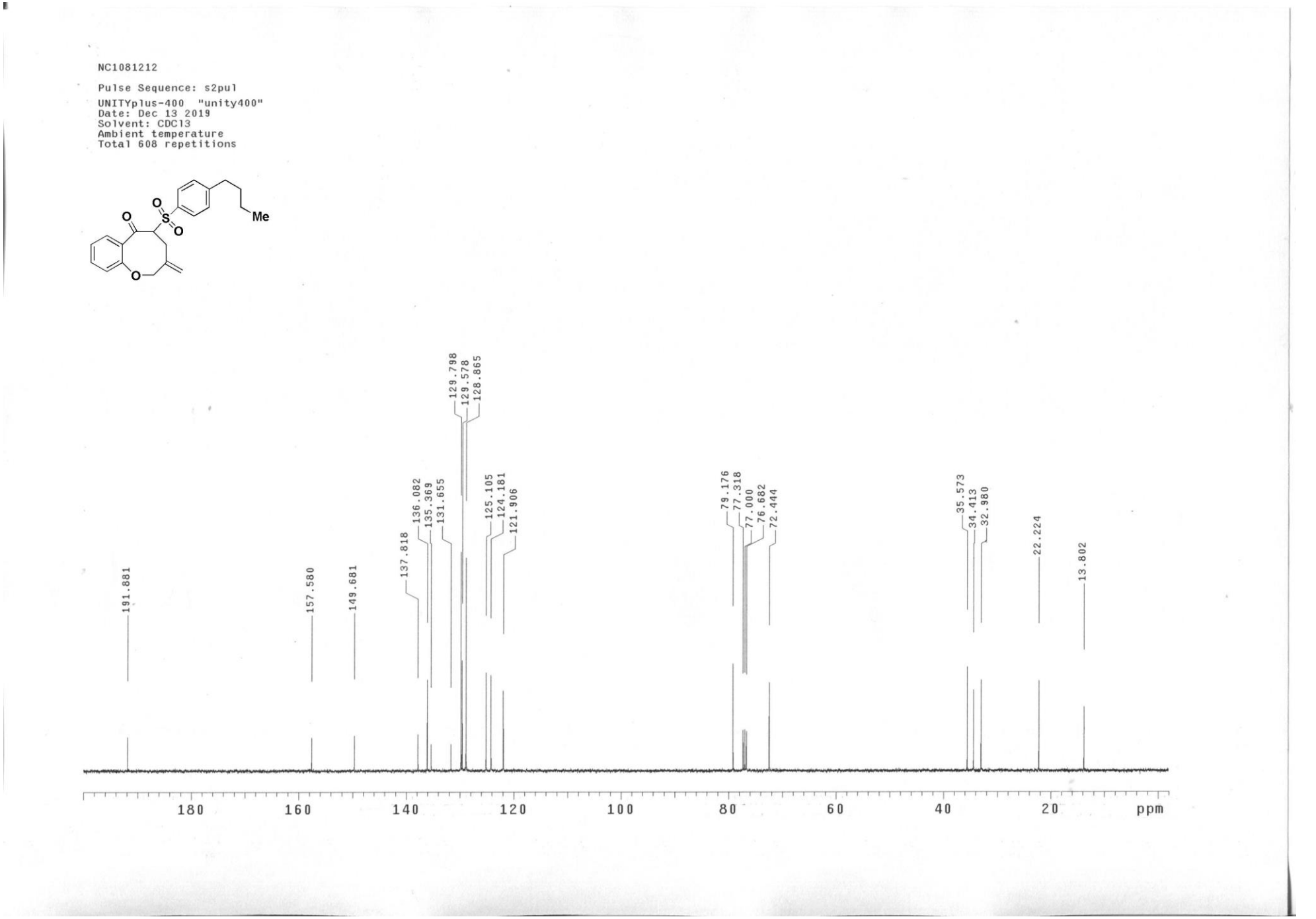

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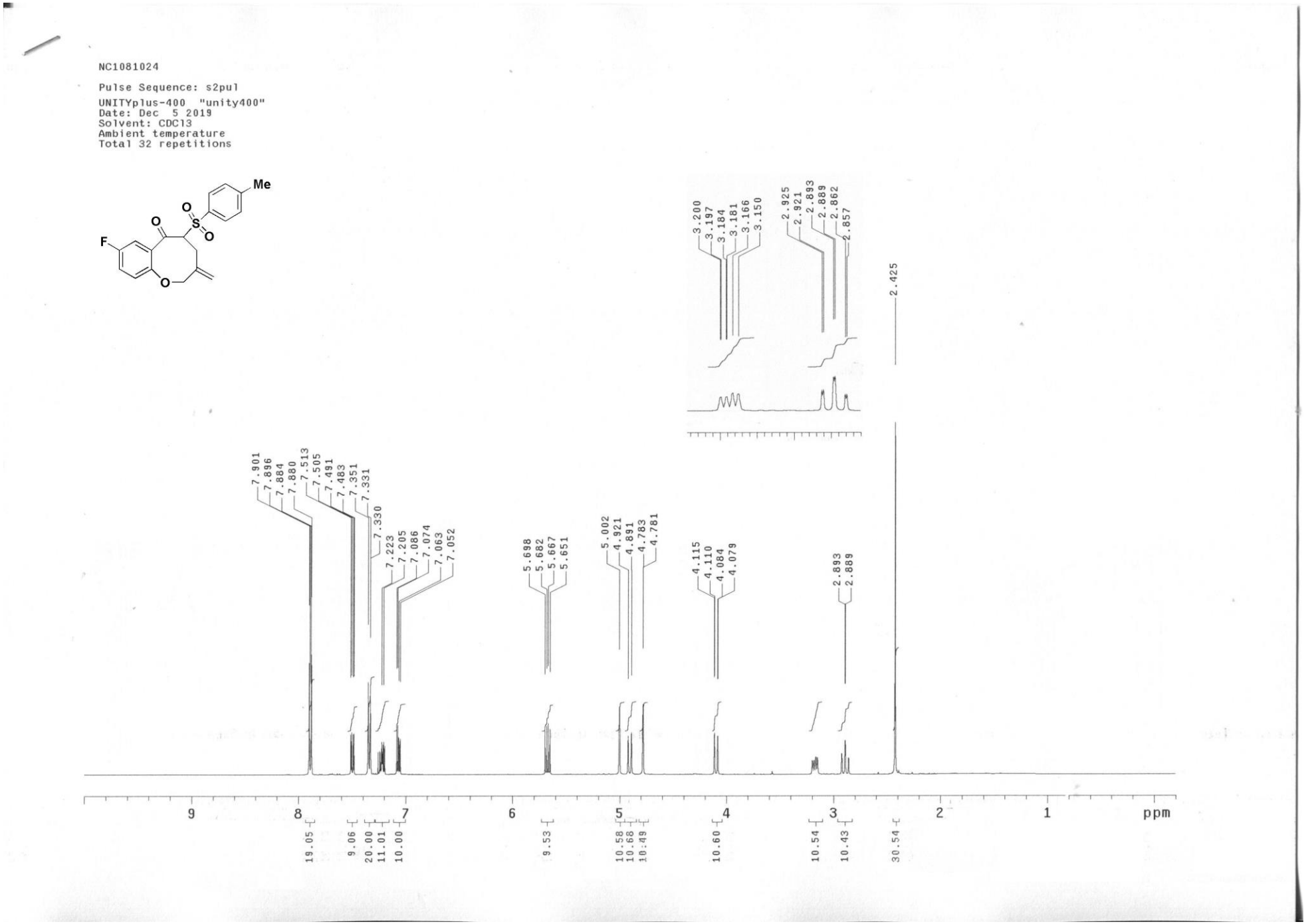
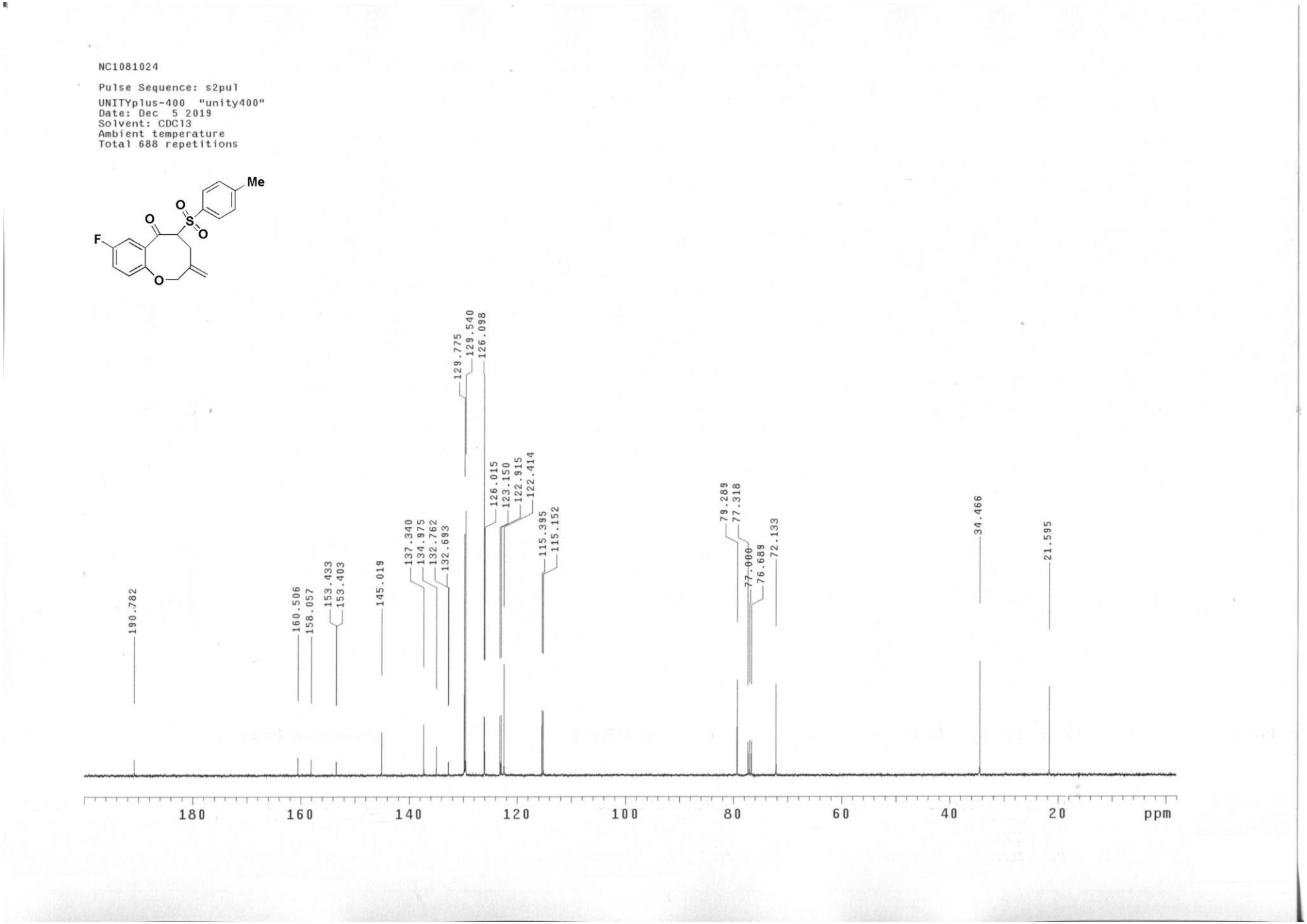


Compound 4l (^{13}C-NMR spectral data)



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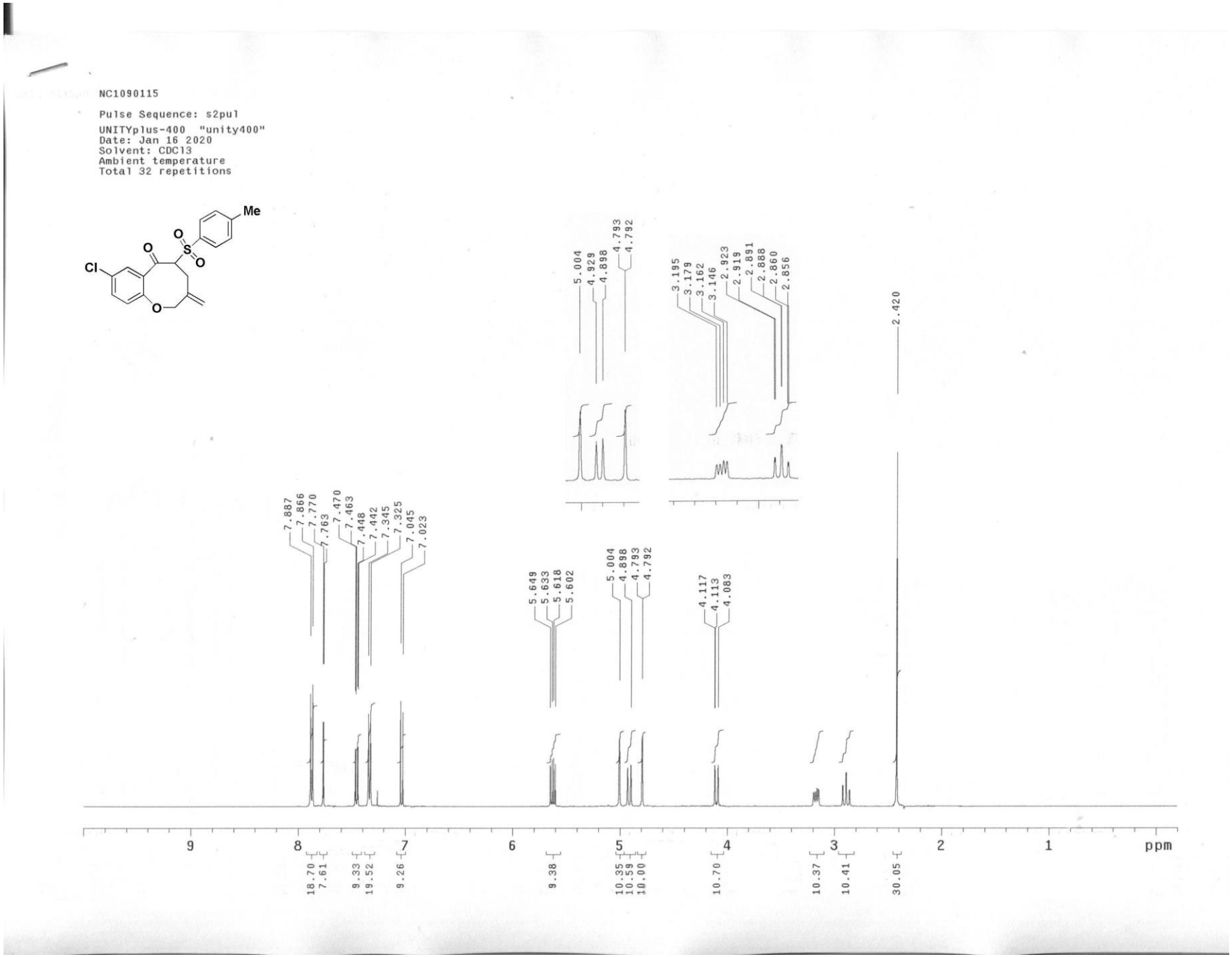

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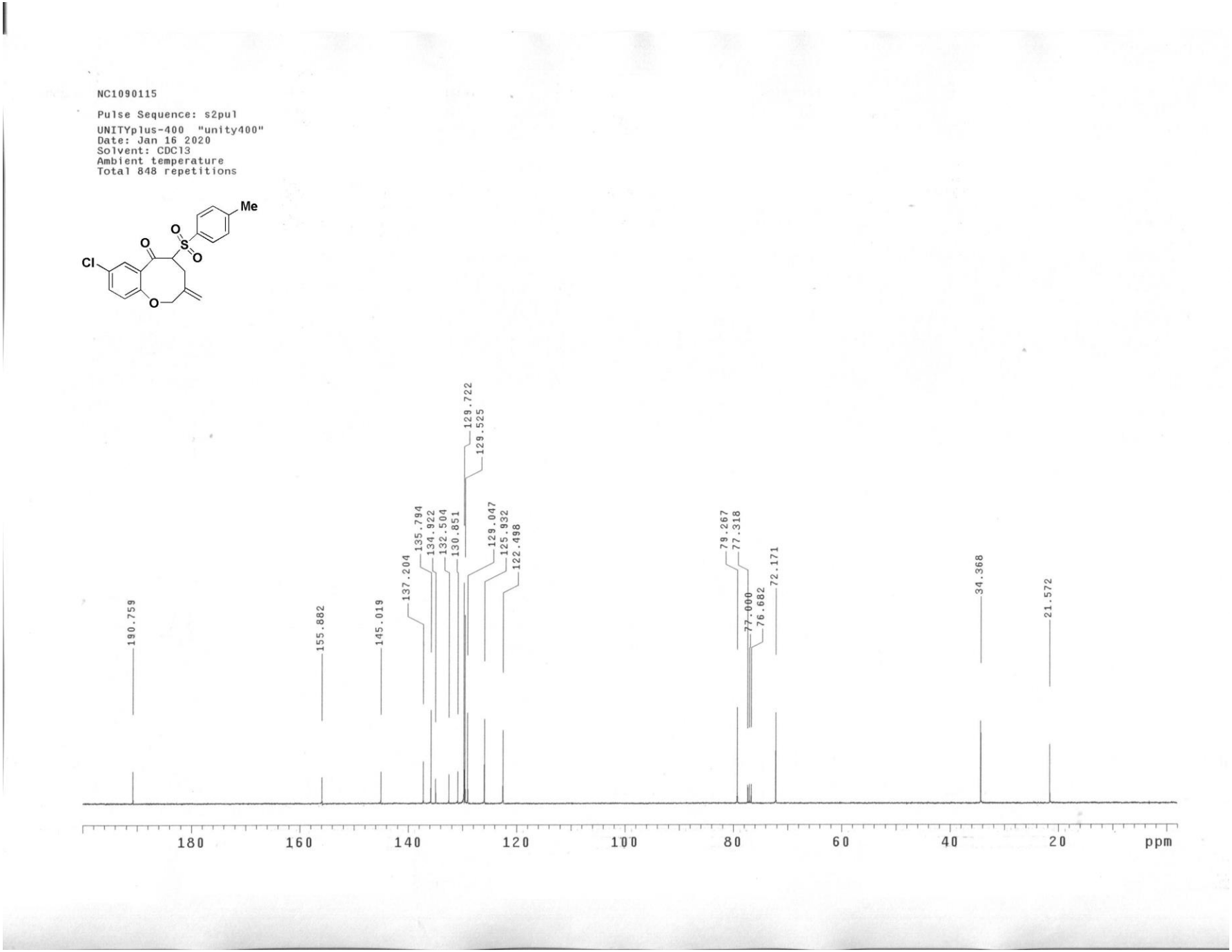

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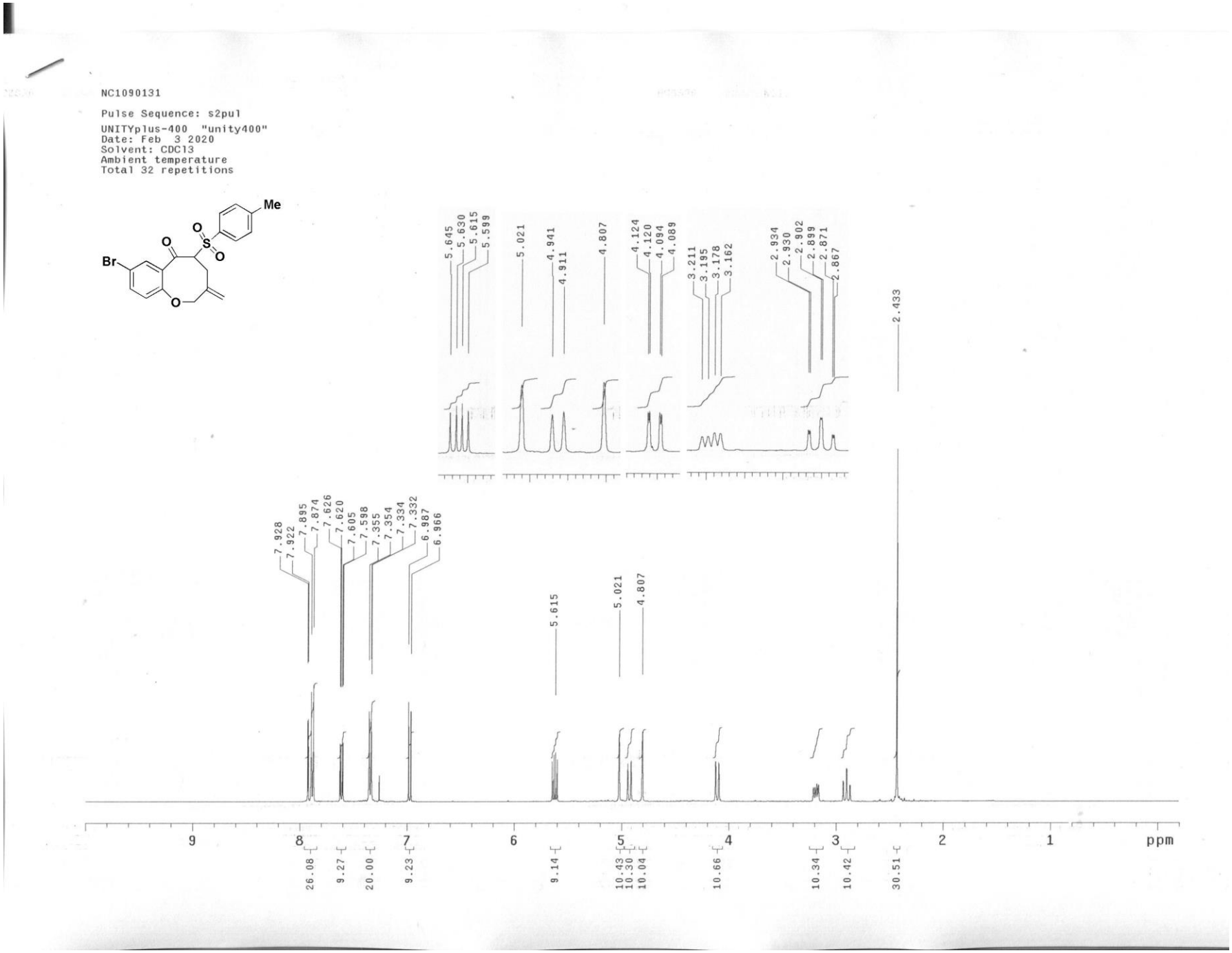

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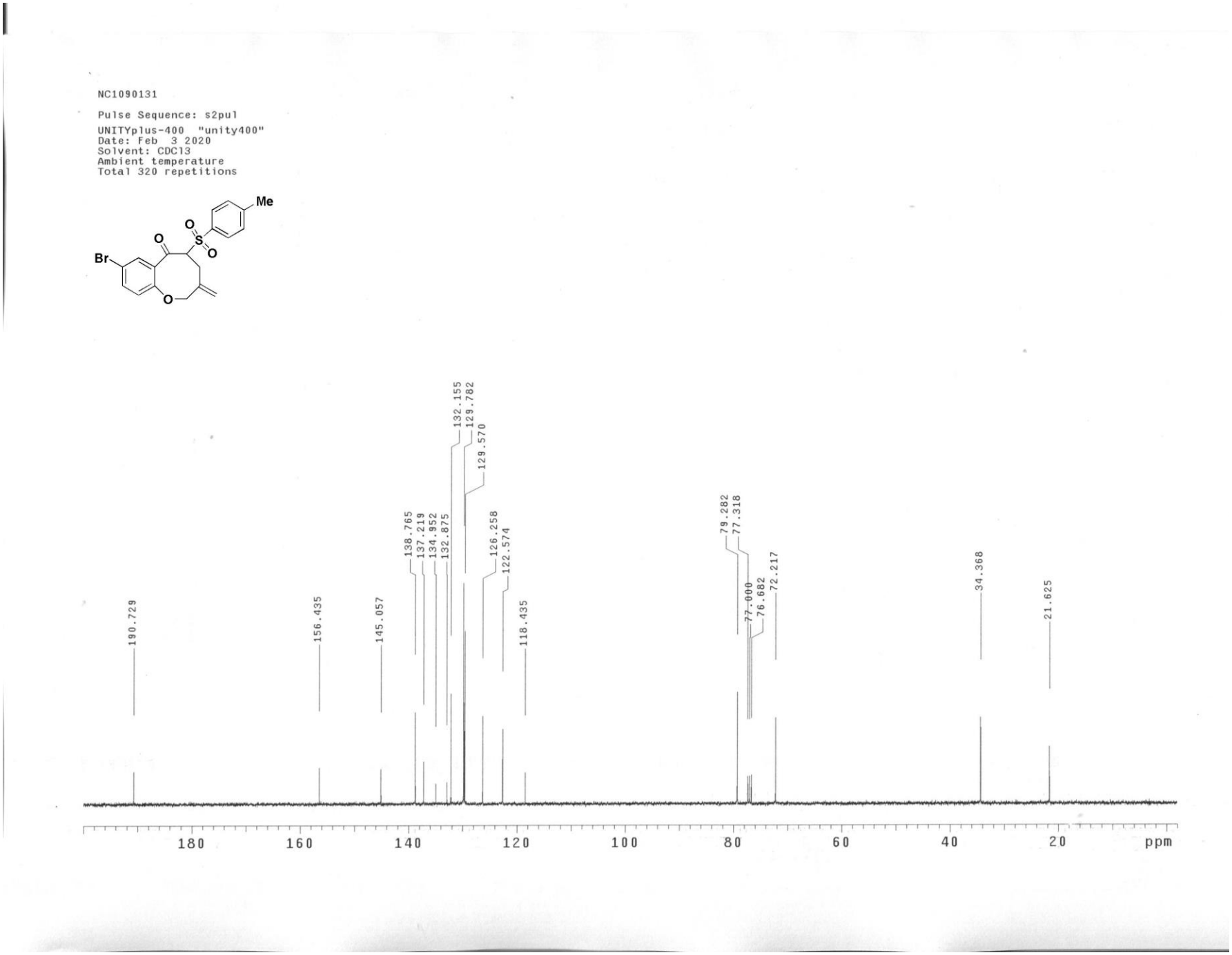

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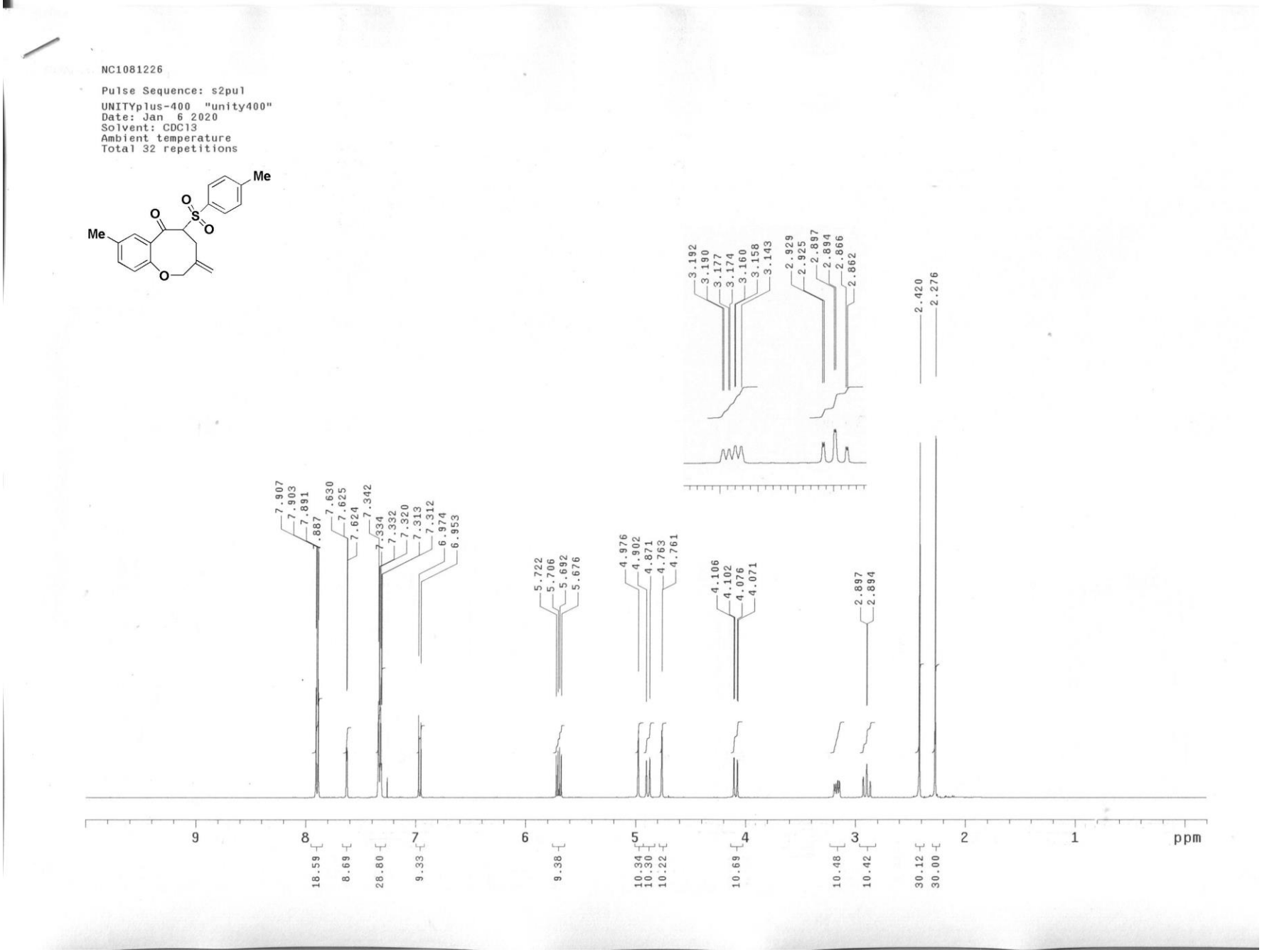
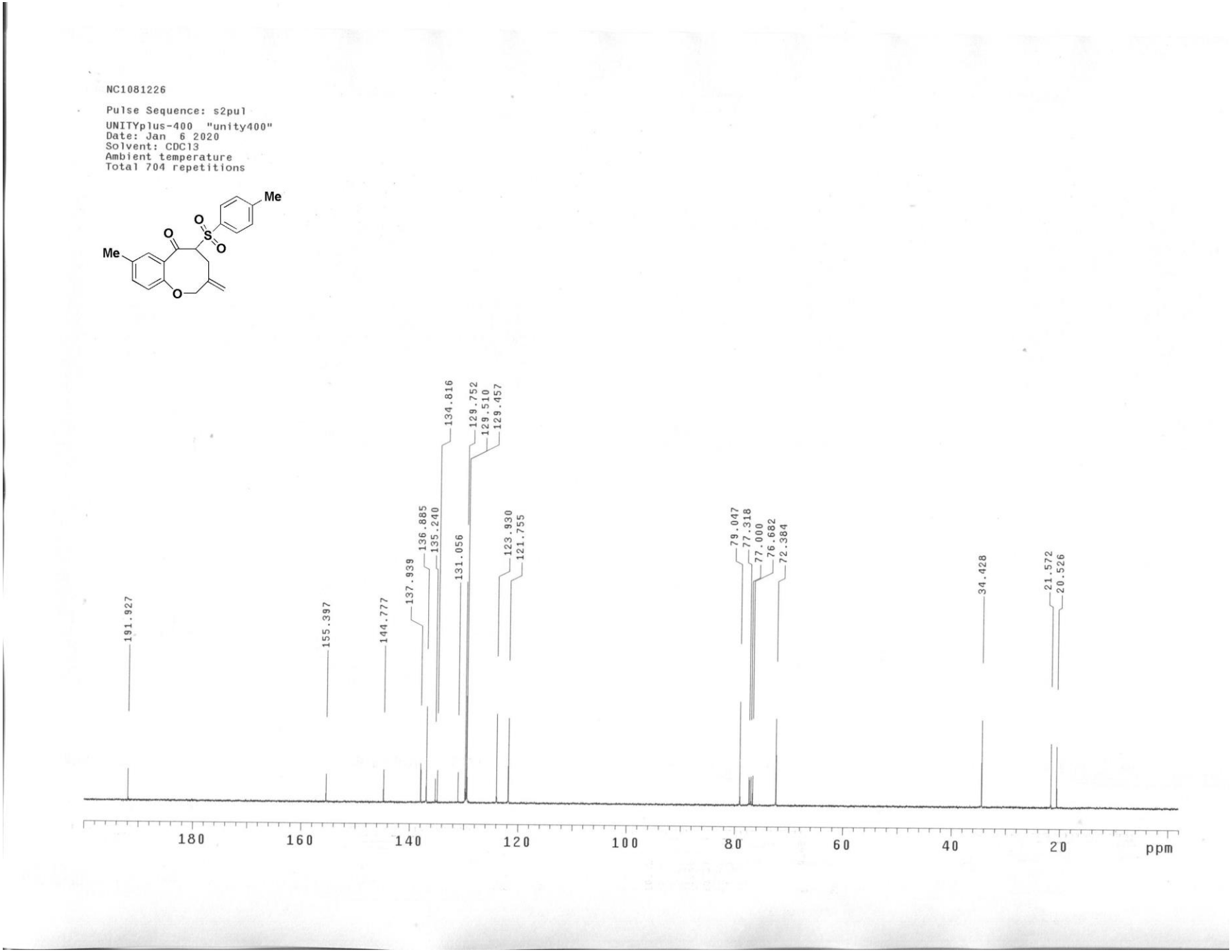


Compound 4o (^{13}C-NMR spectral data)



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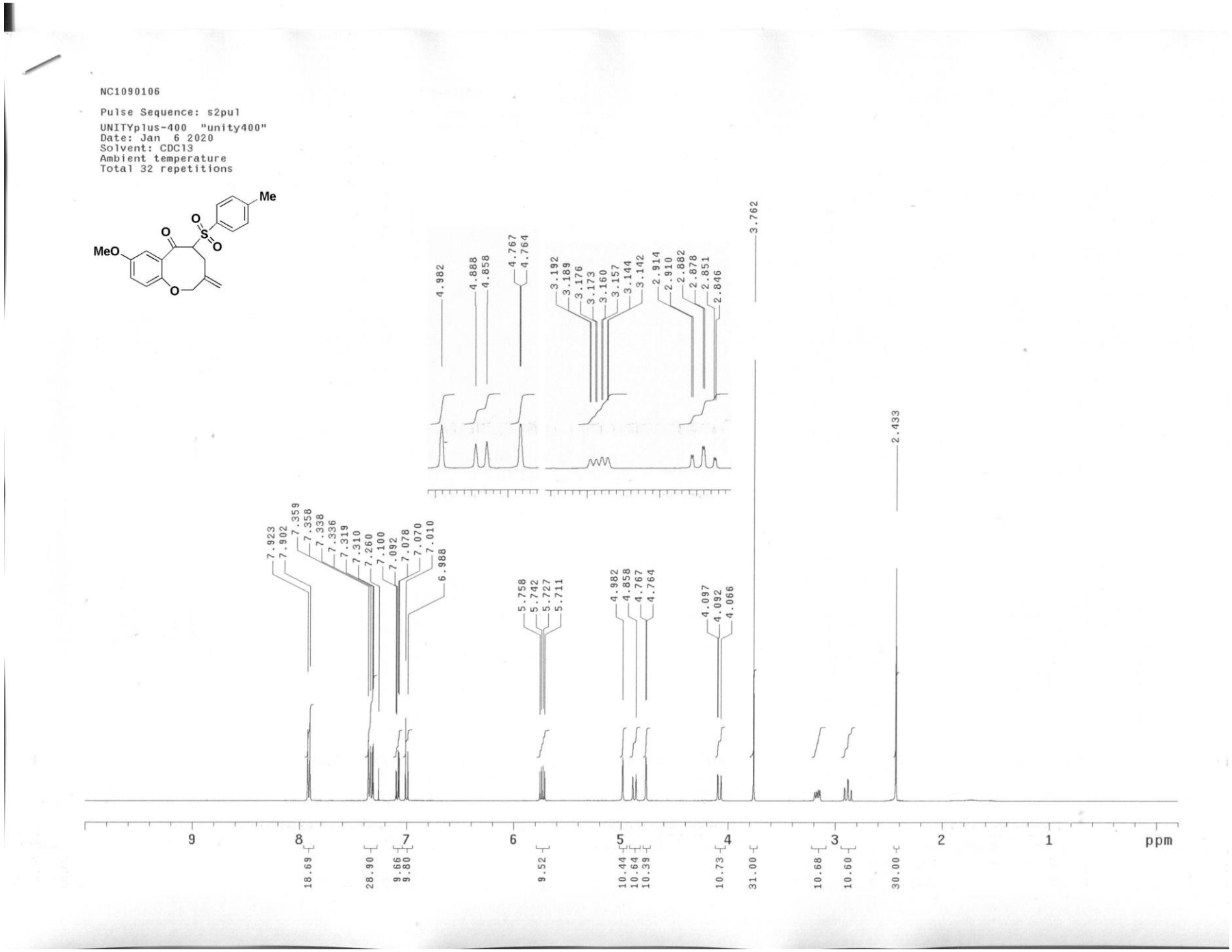

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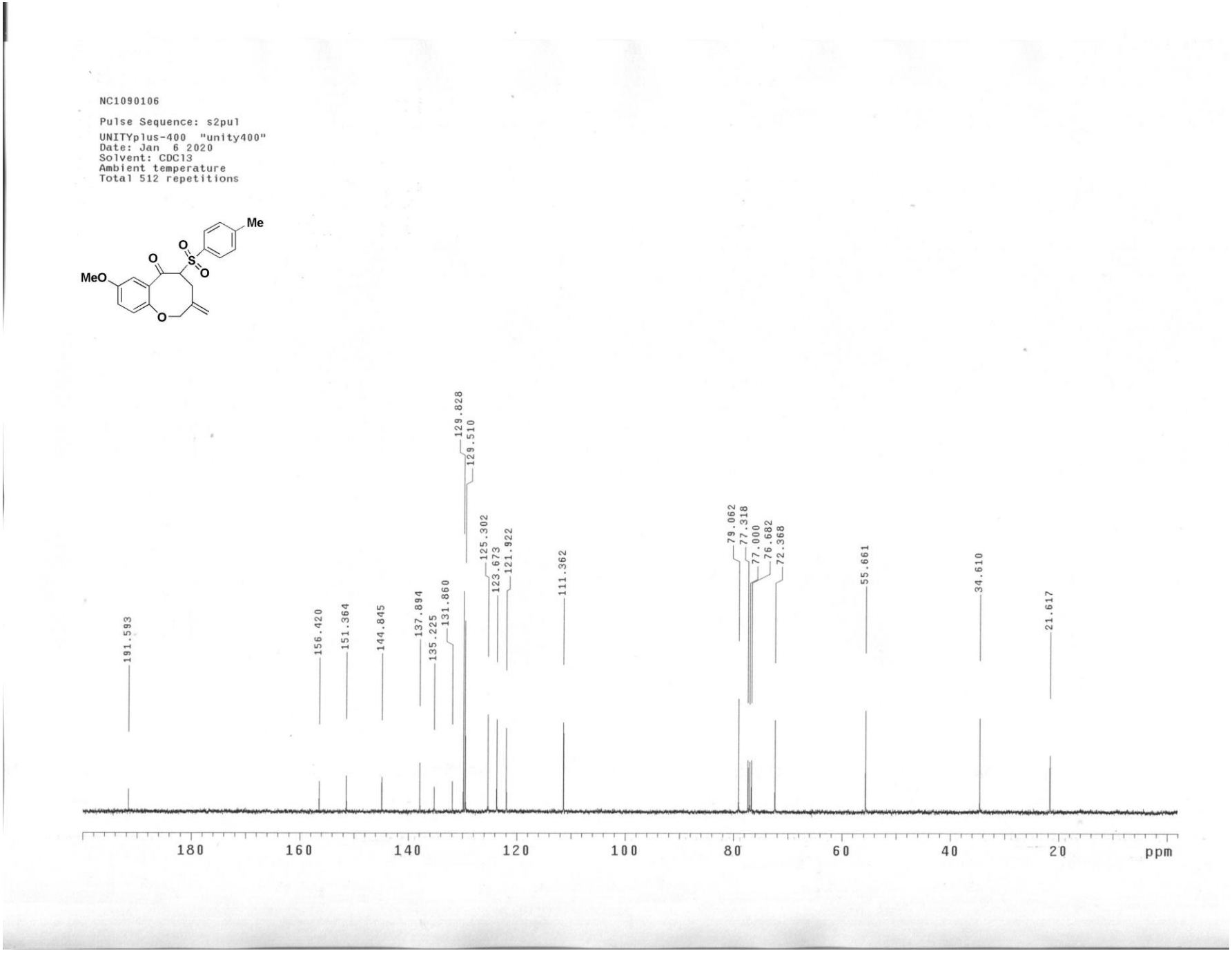

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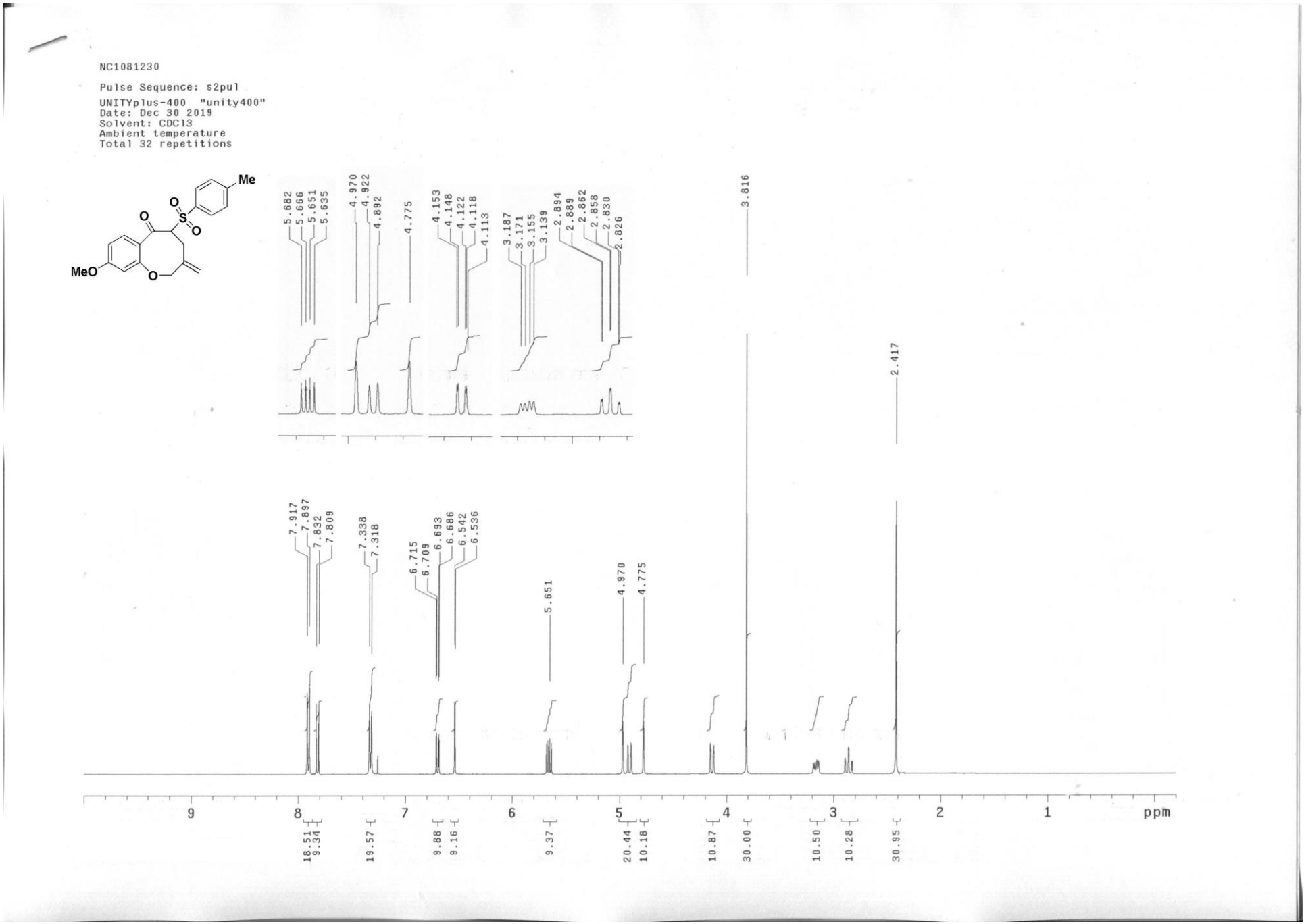

Compound 4q (^{13}C-NMR spectral data)

NC1081230

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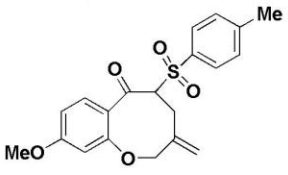
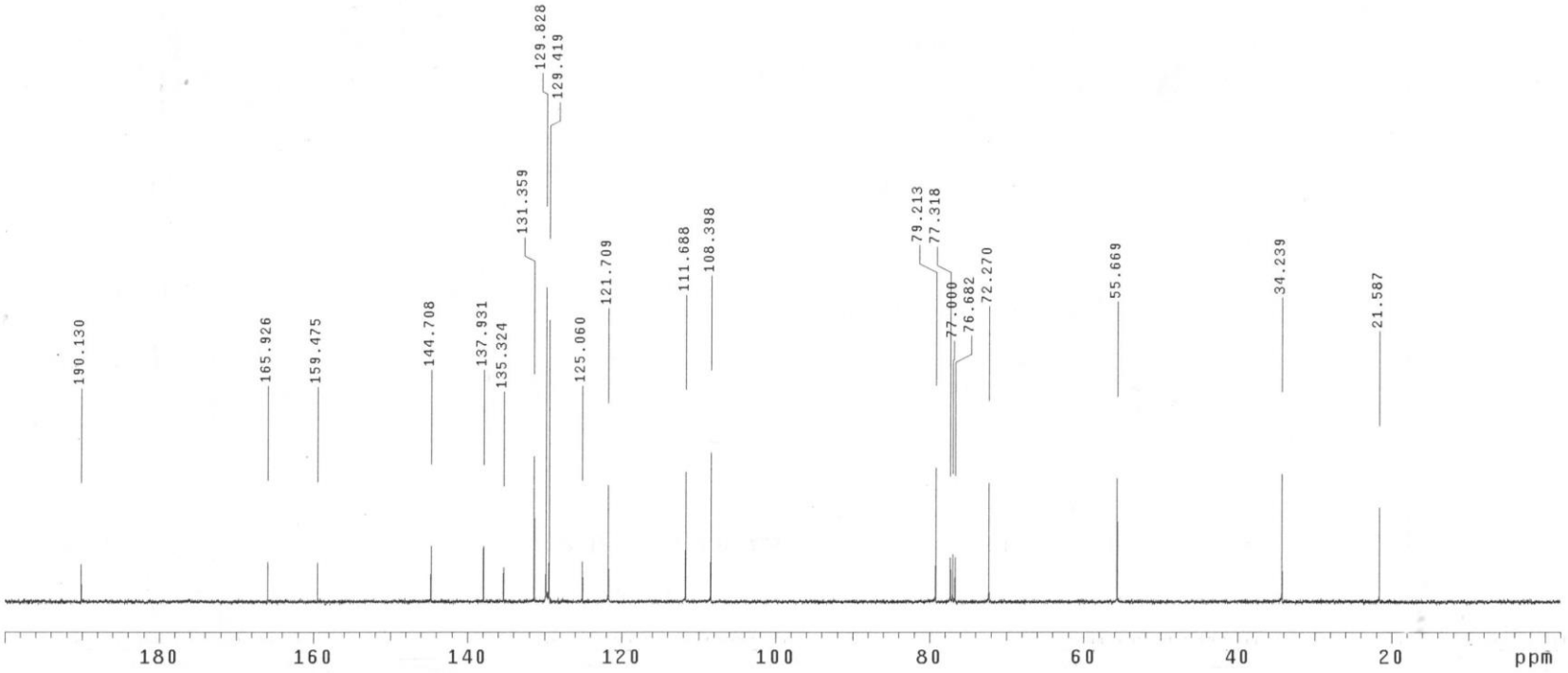
Date: Dec 30 2019
Solvent: CDCl3
Ambient temperature
Total 816 repetitions





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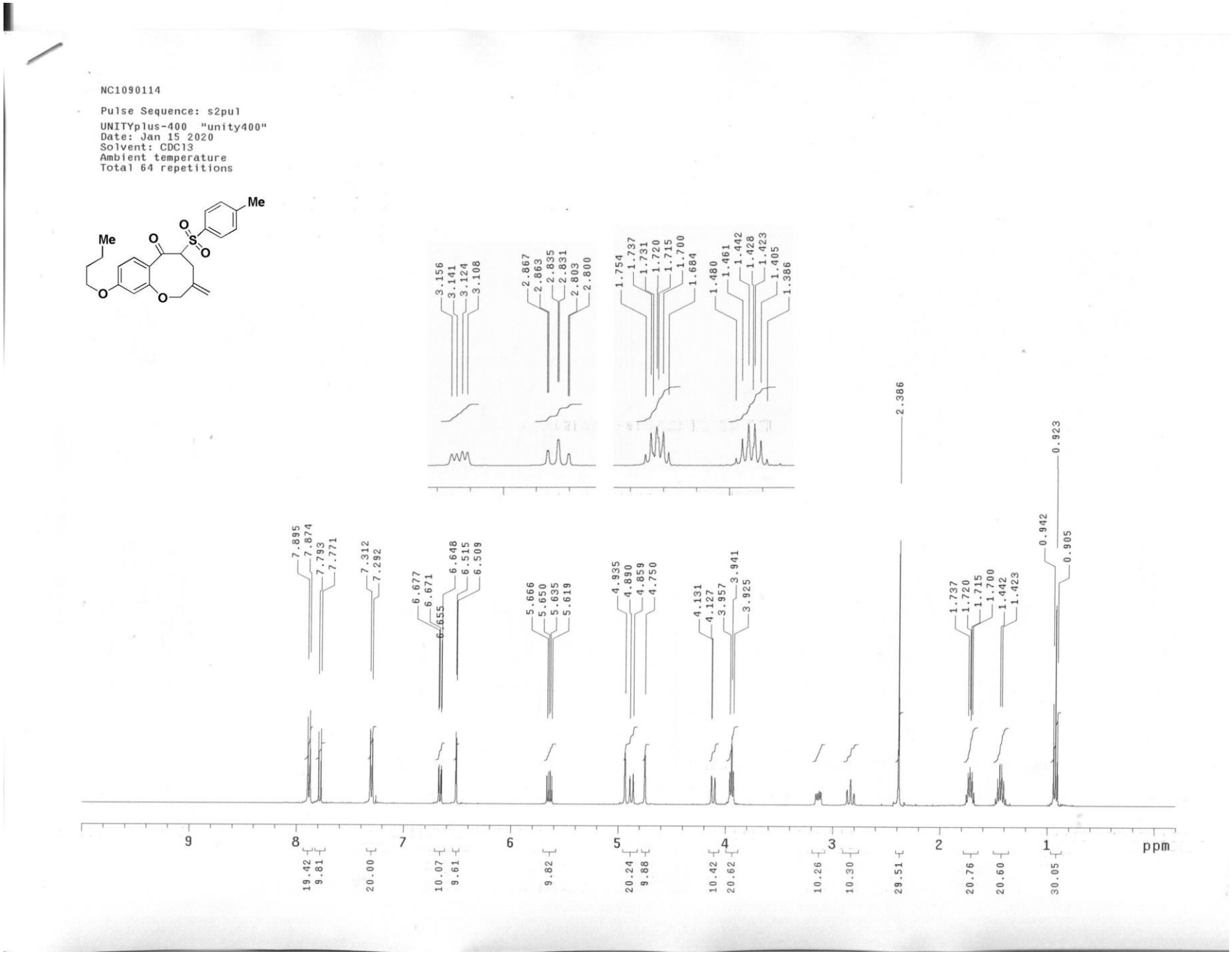
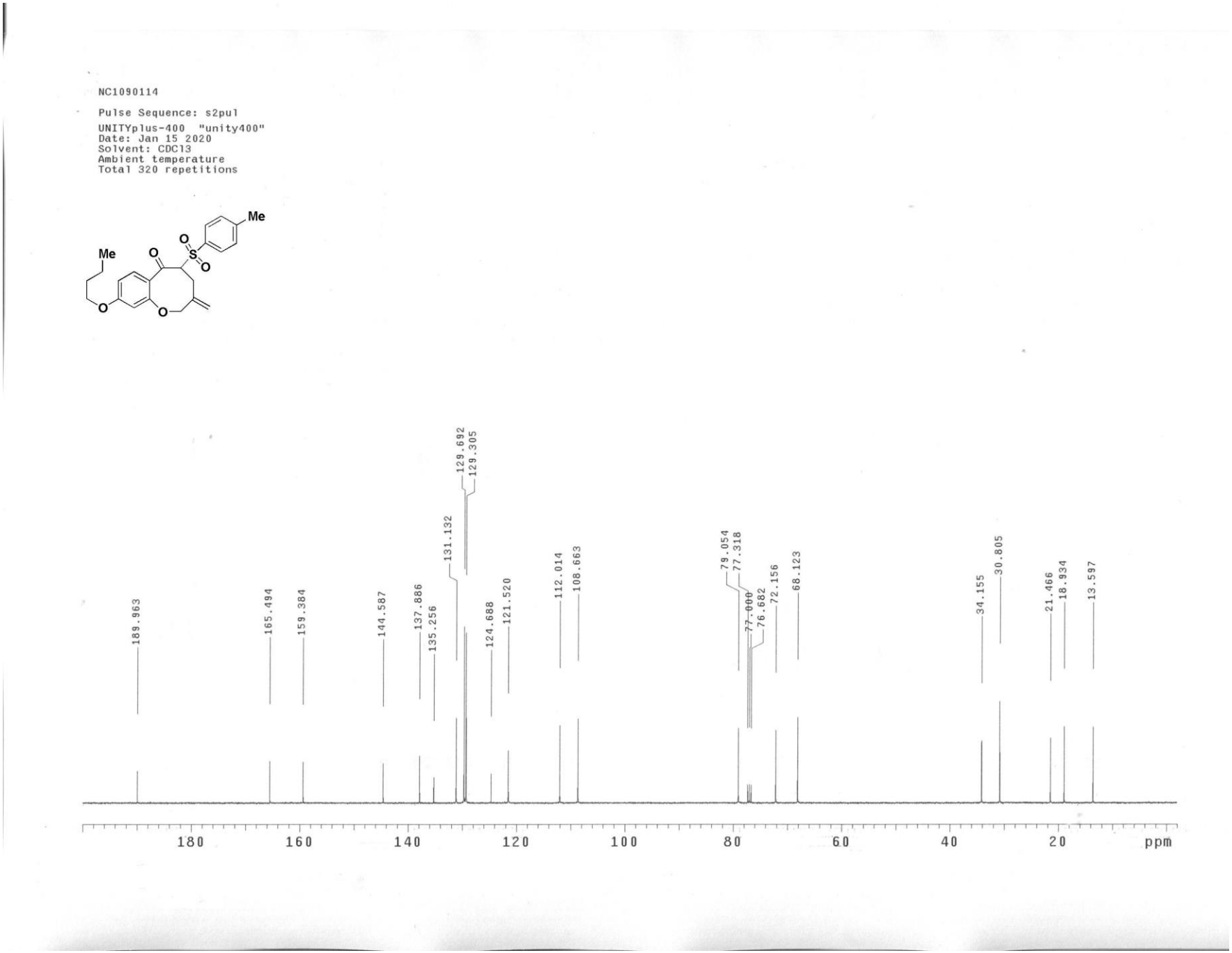


Compound 4r (^{13}C-NMR spectral data)



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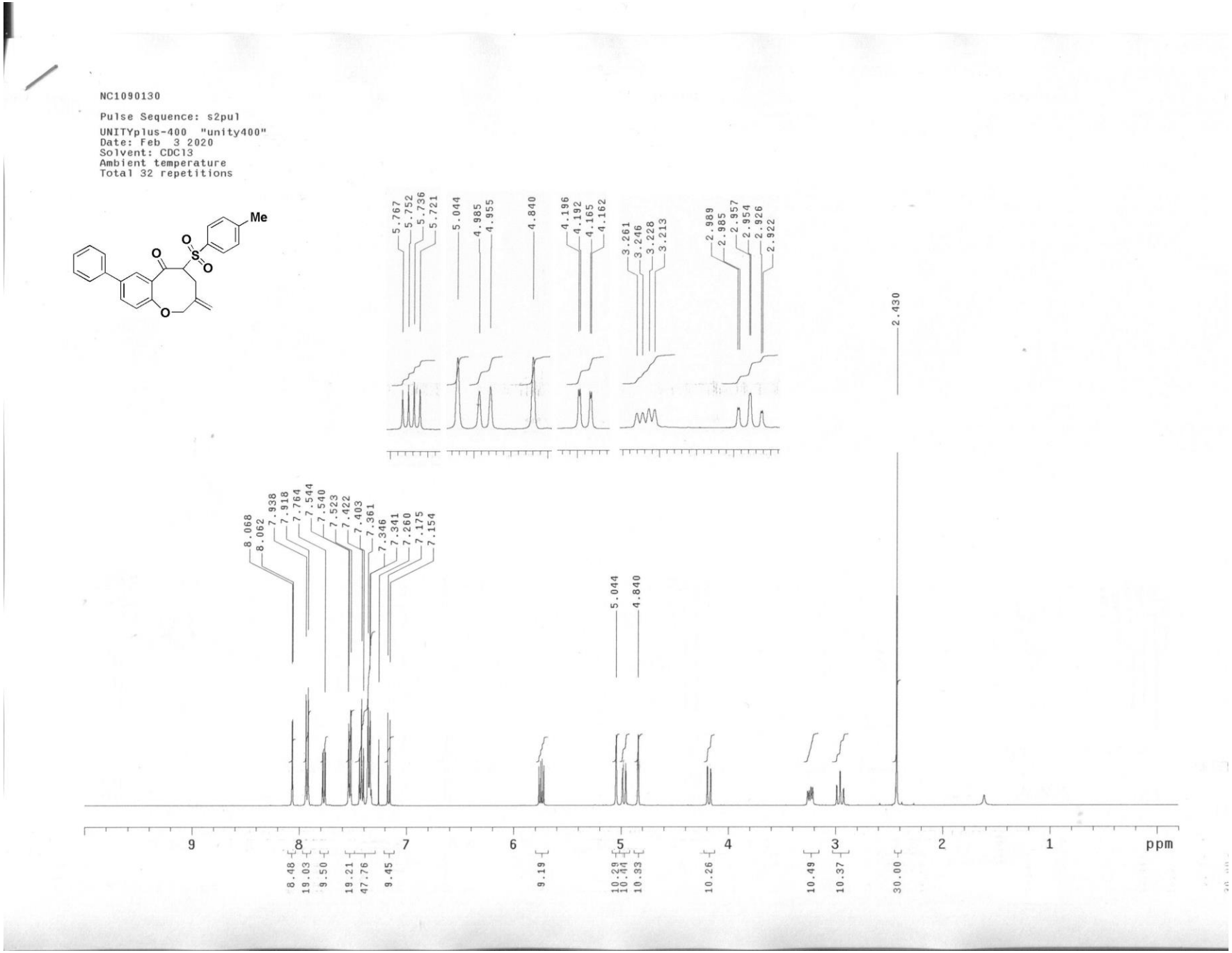

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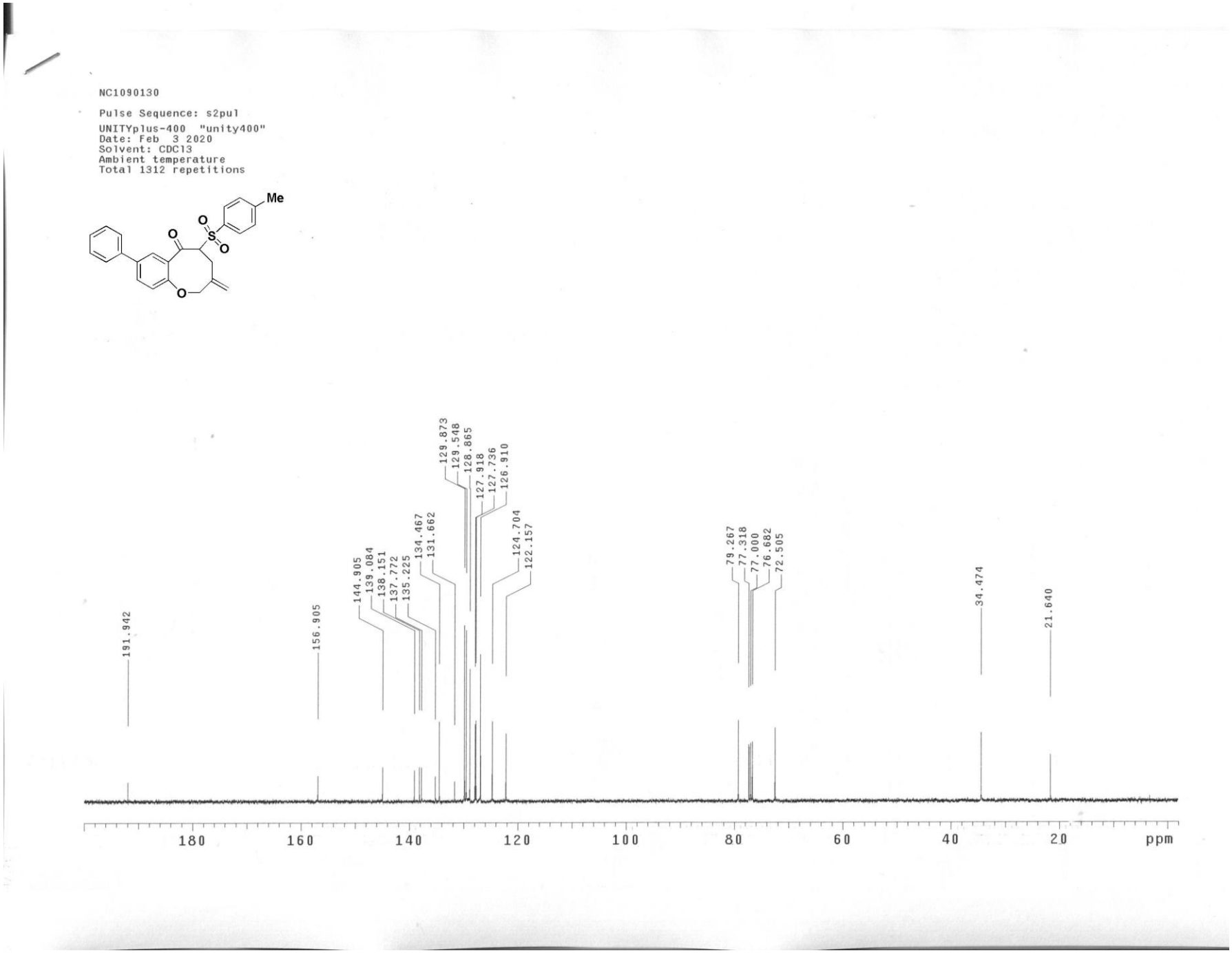
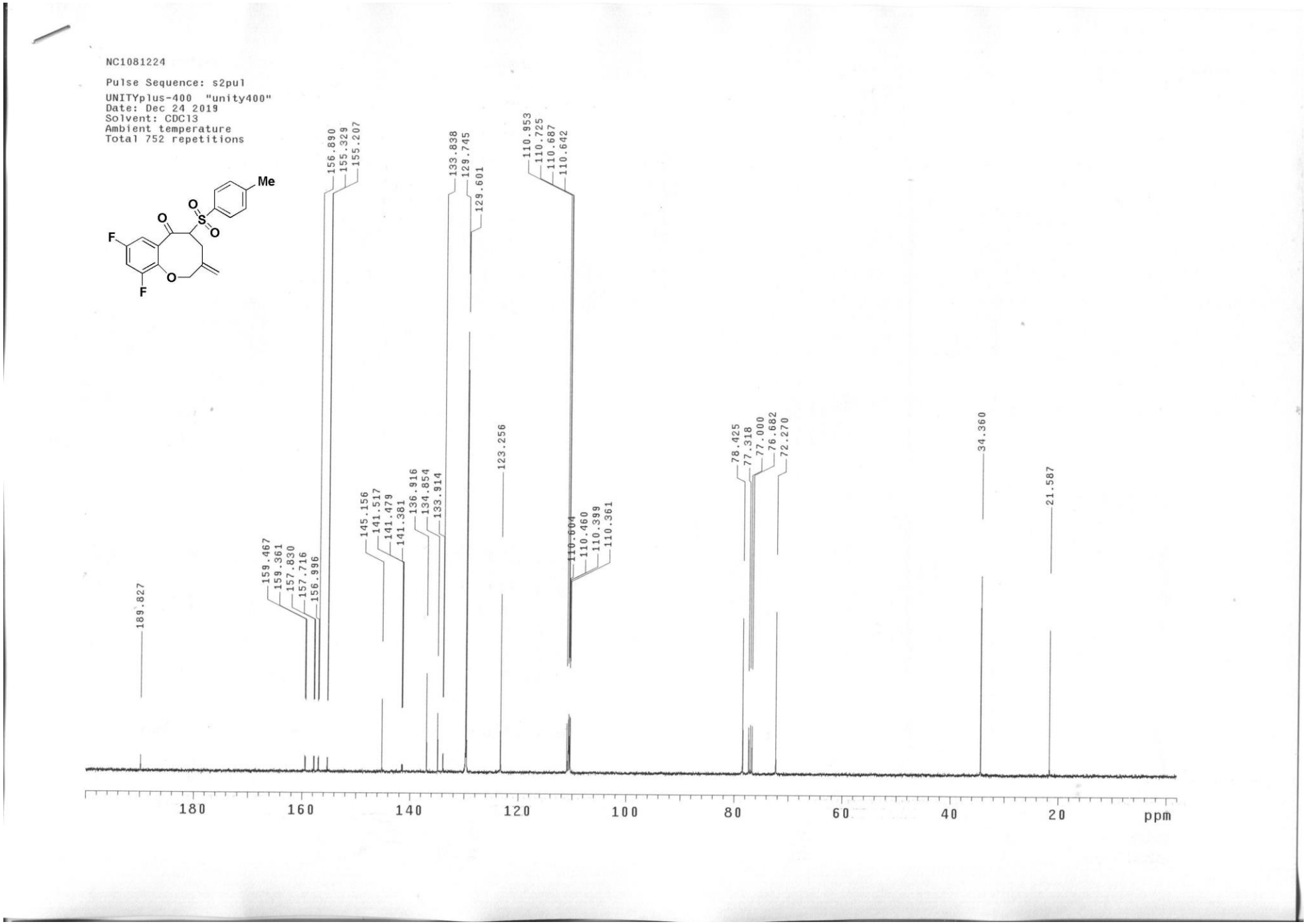


Compound 4t (^{1}H-NMR spectral data)

Compound 4t (^{13}C-NMR spectral data)



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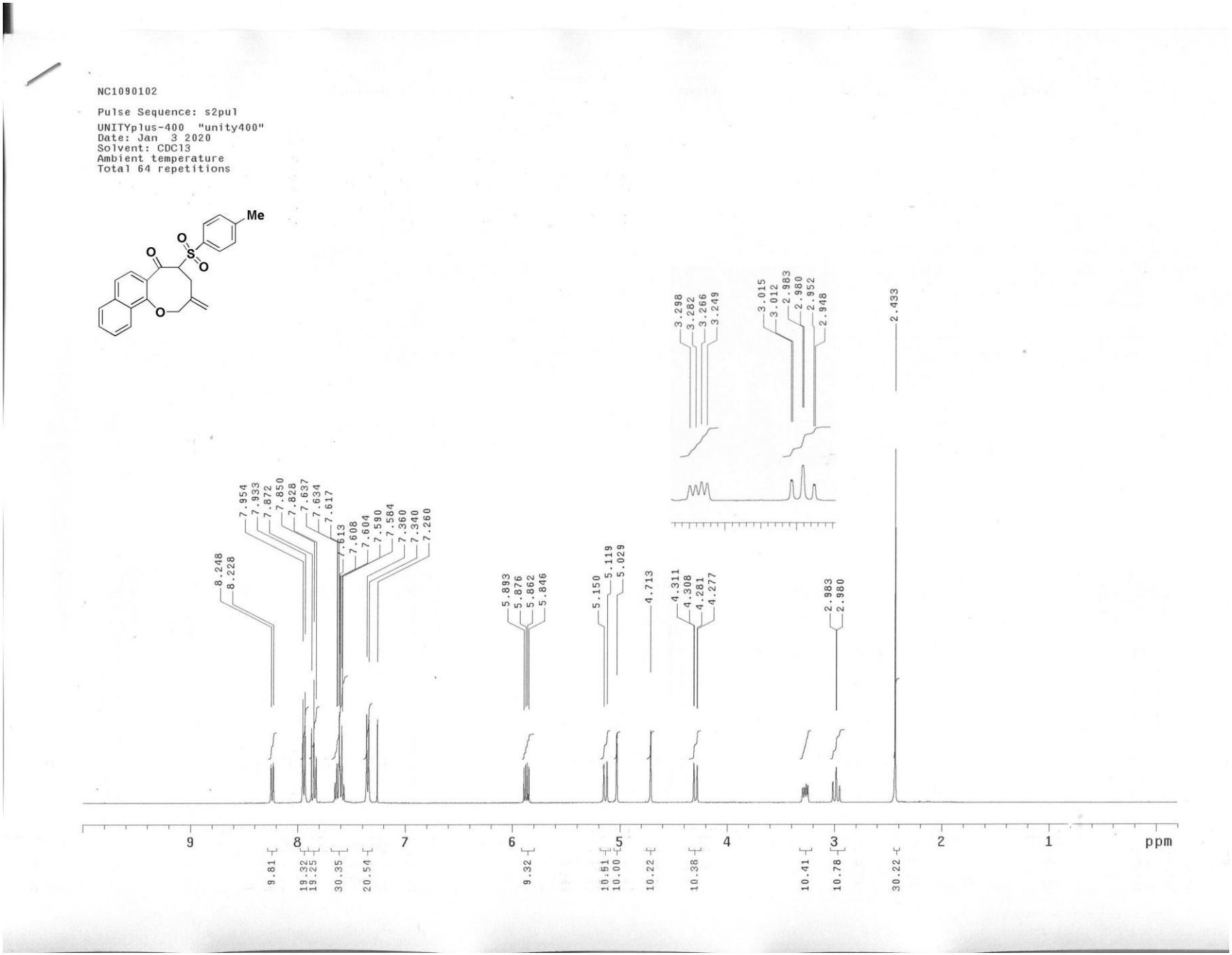
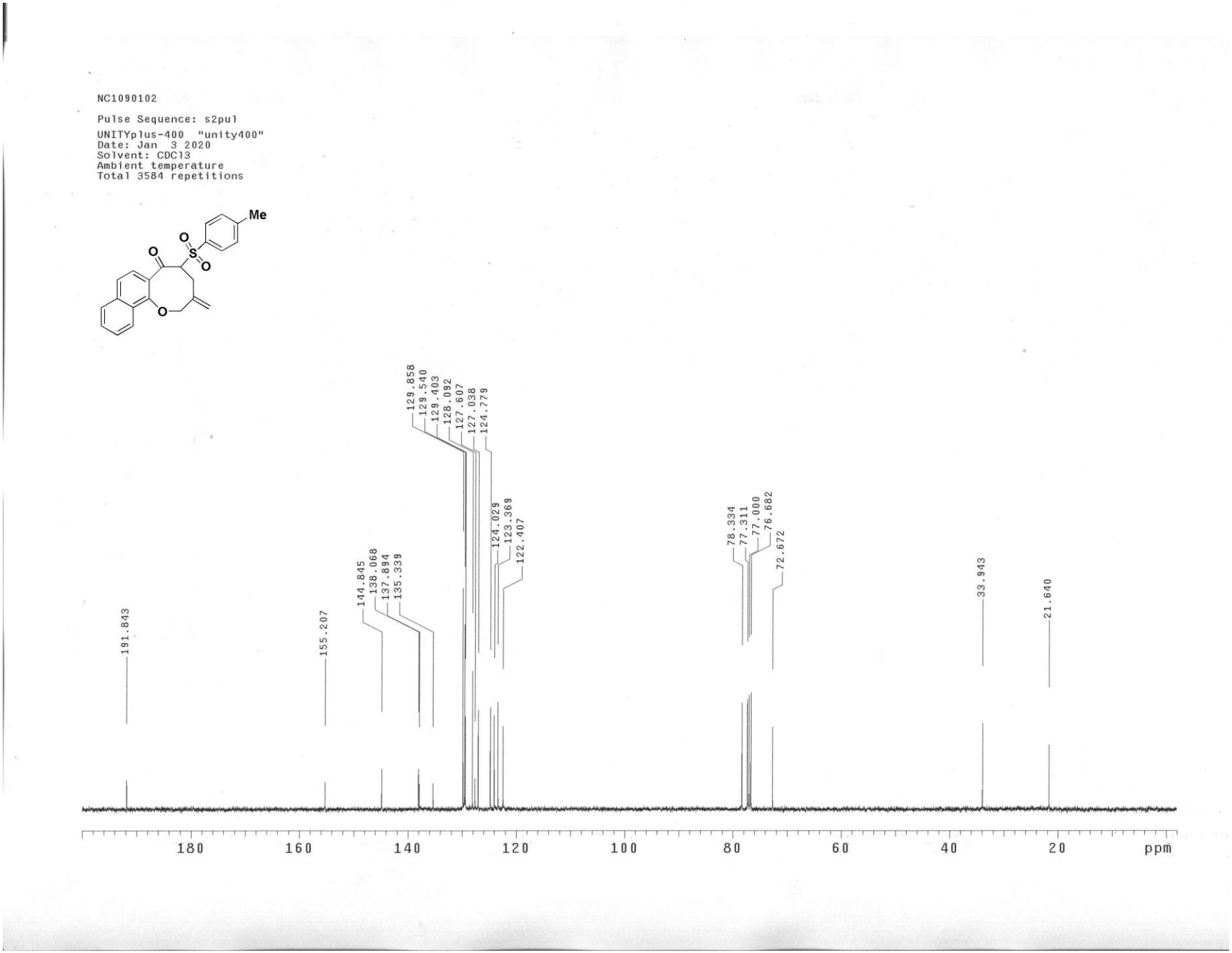
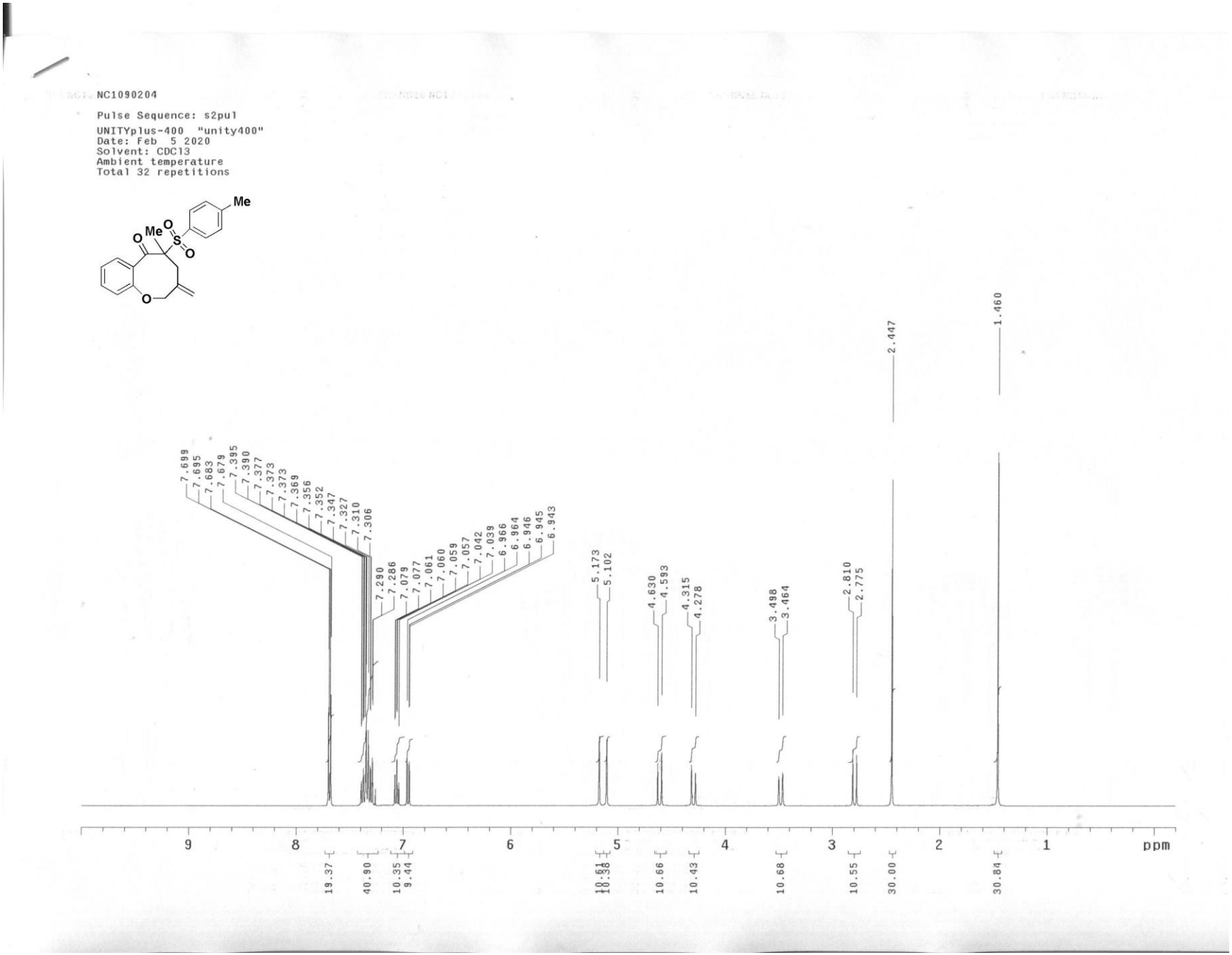


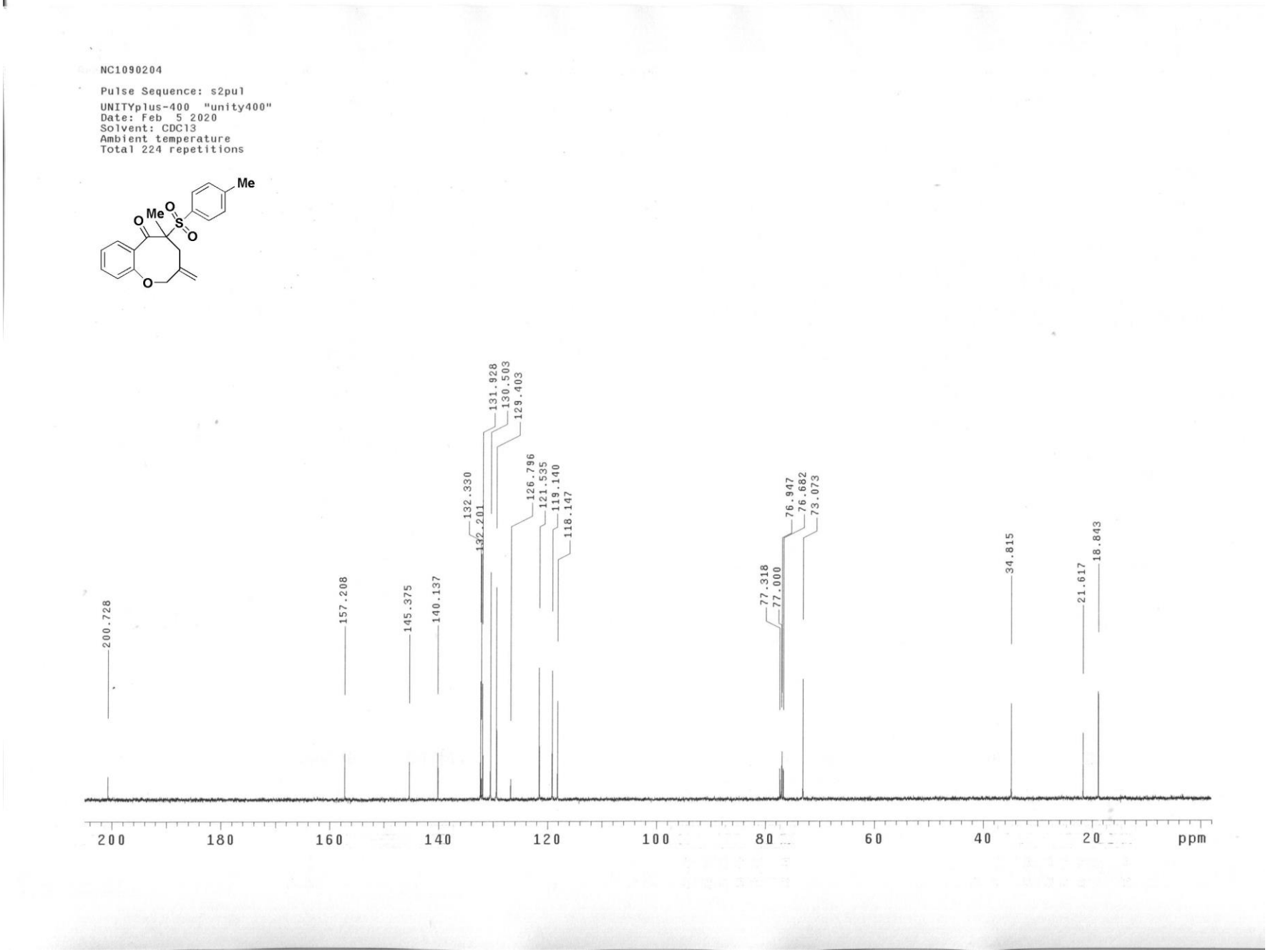
Compound 4u (^{13}C-NMR spectral data)



Compound 4v (^{1}H-NMR spectral data)



Compound 4v (^{13}C-NMR spectral data)



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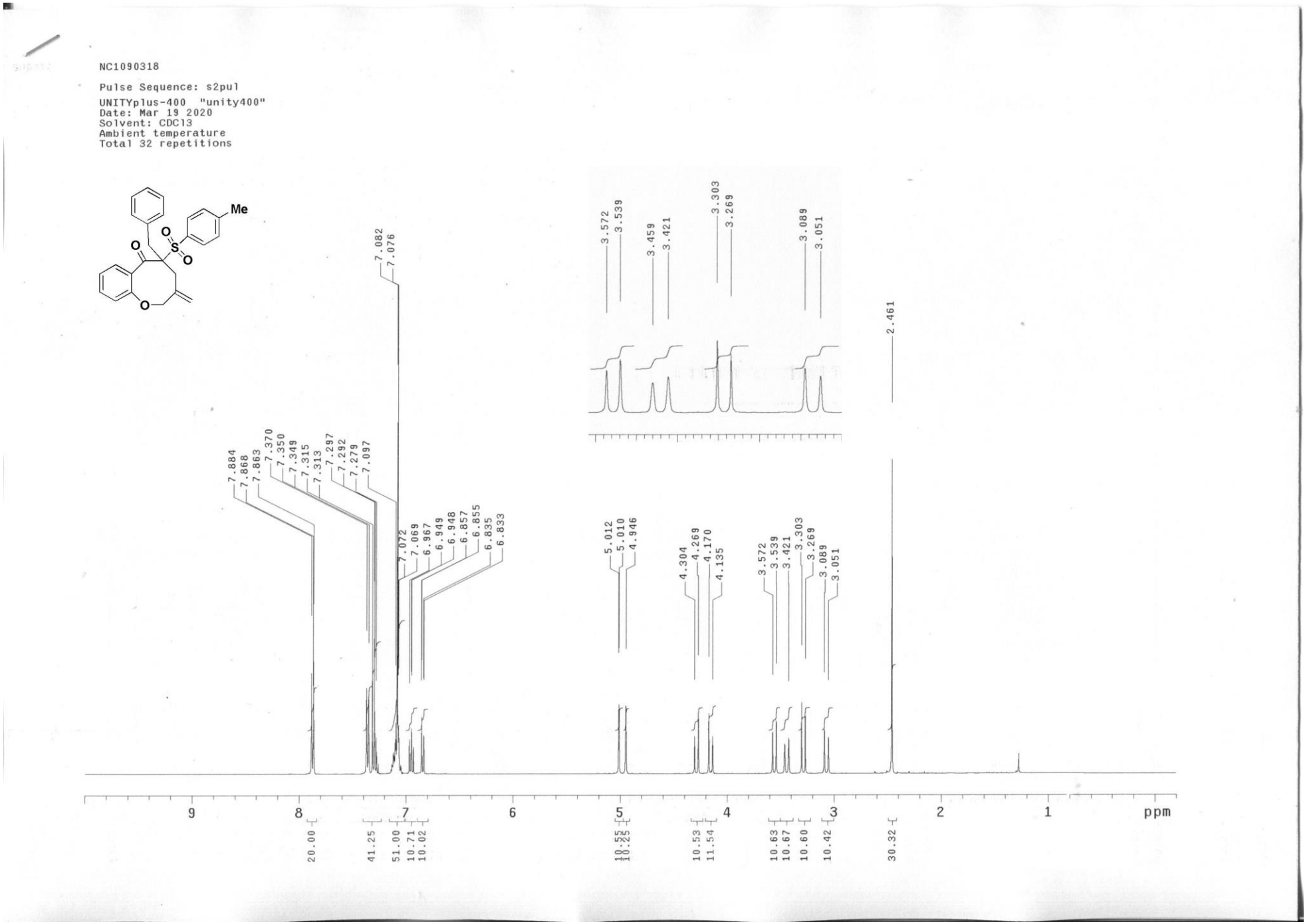

Compound 4w (^{13}C-NMR spectral data)

NC1090318

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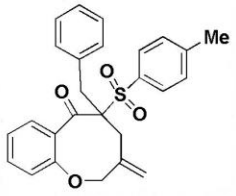
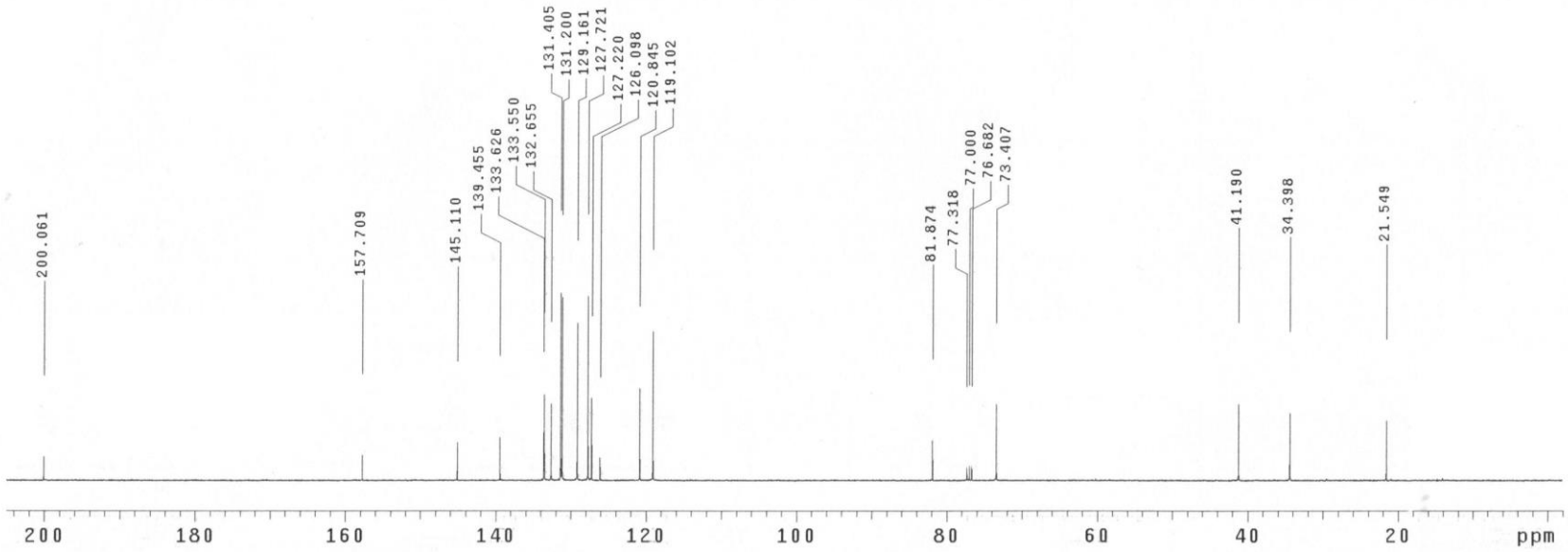
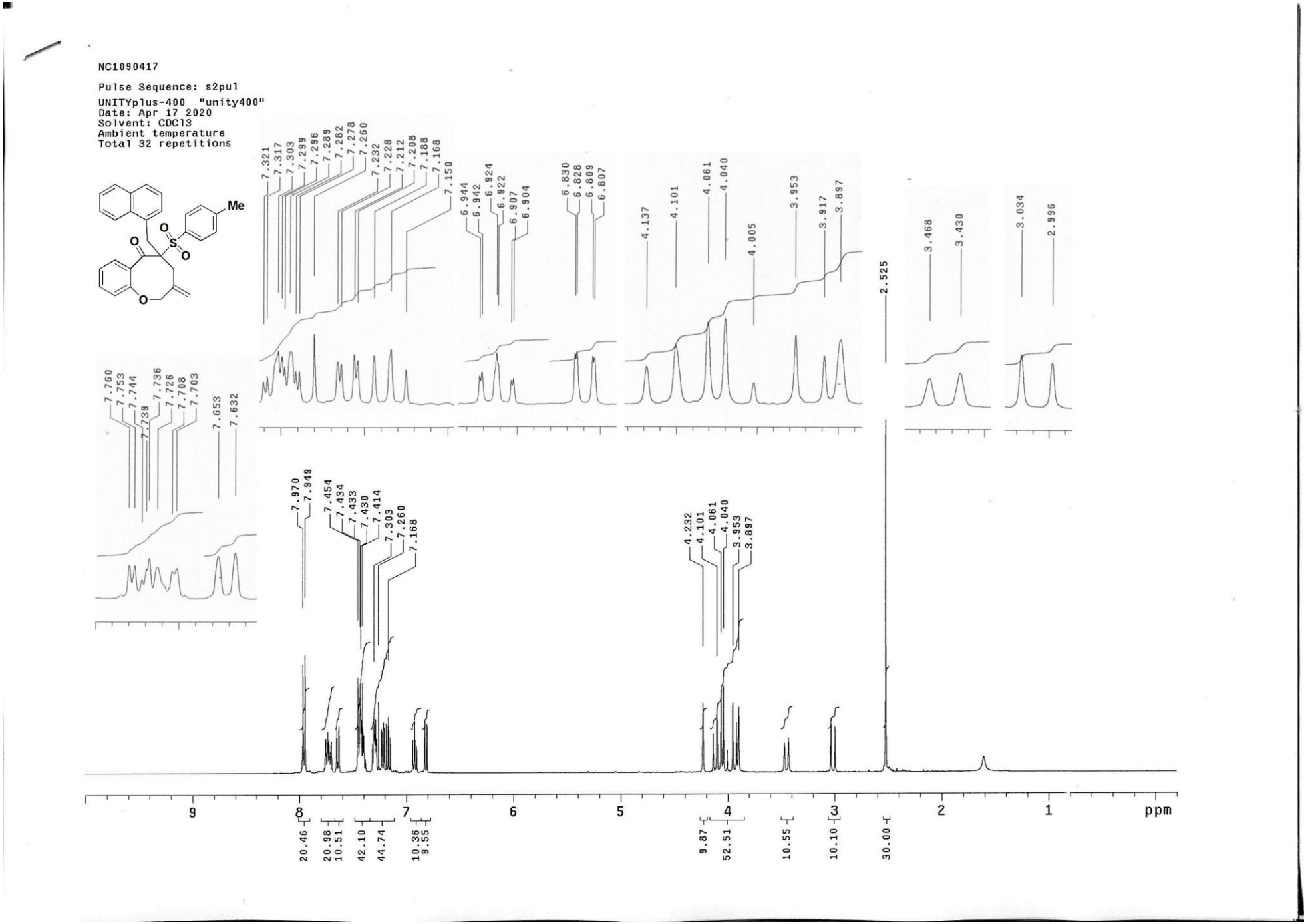
Date: Mar 19 2020
Solvent: CDCl3
Ambient temperature
Total 512 repetitions





Compound 4x (^{1}H-NMR spectral data)



Compound 4x (^{13}C-NMR spectral data)

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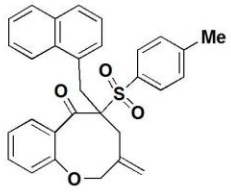
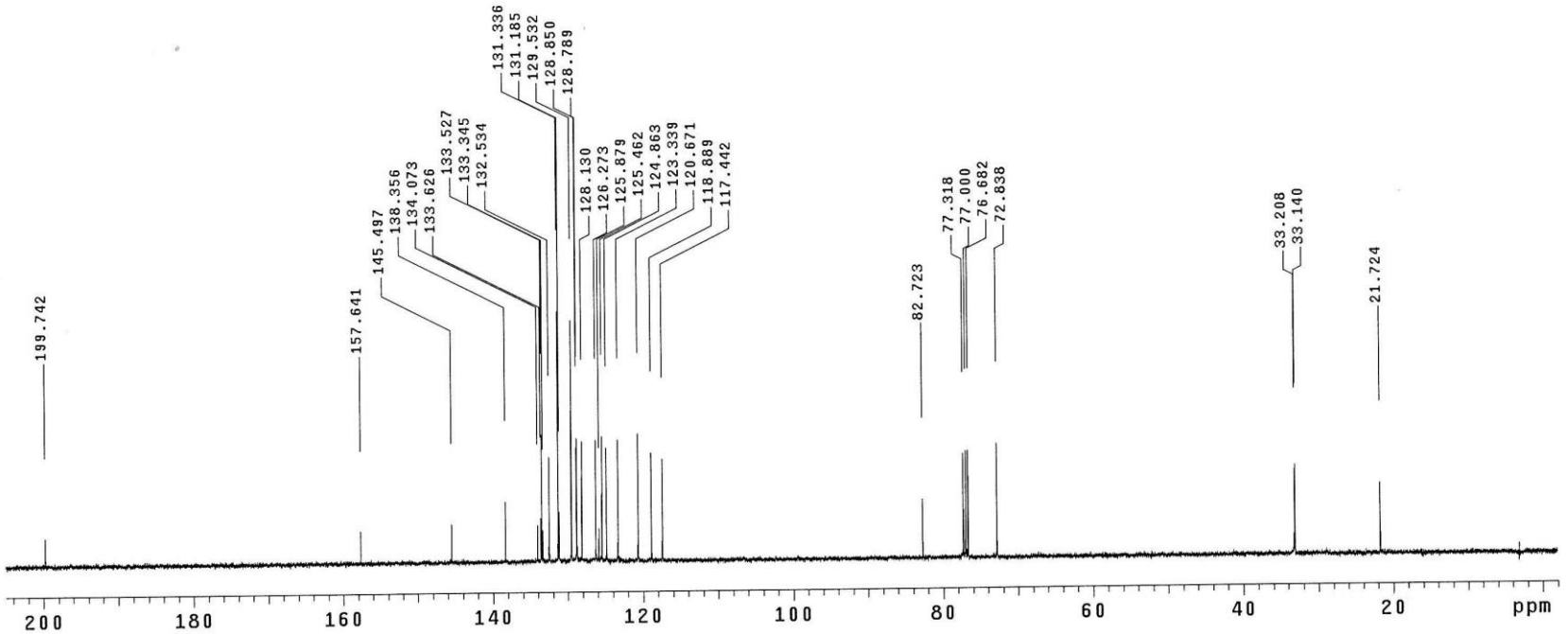
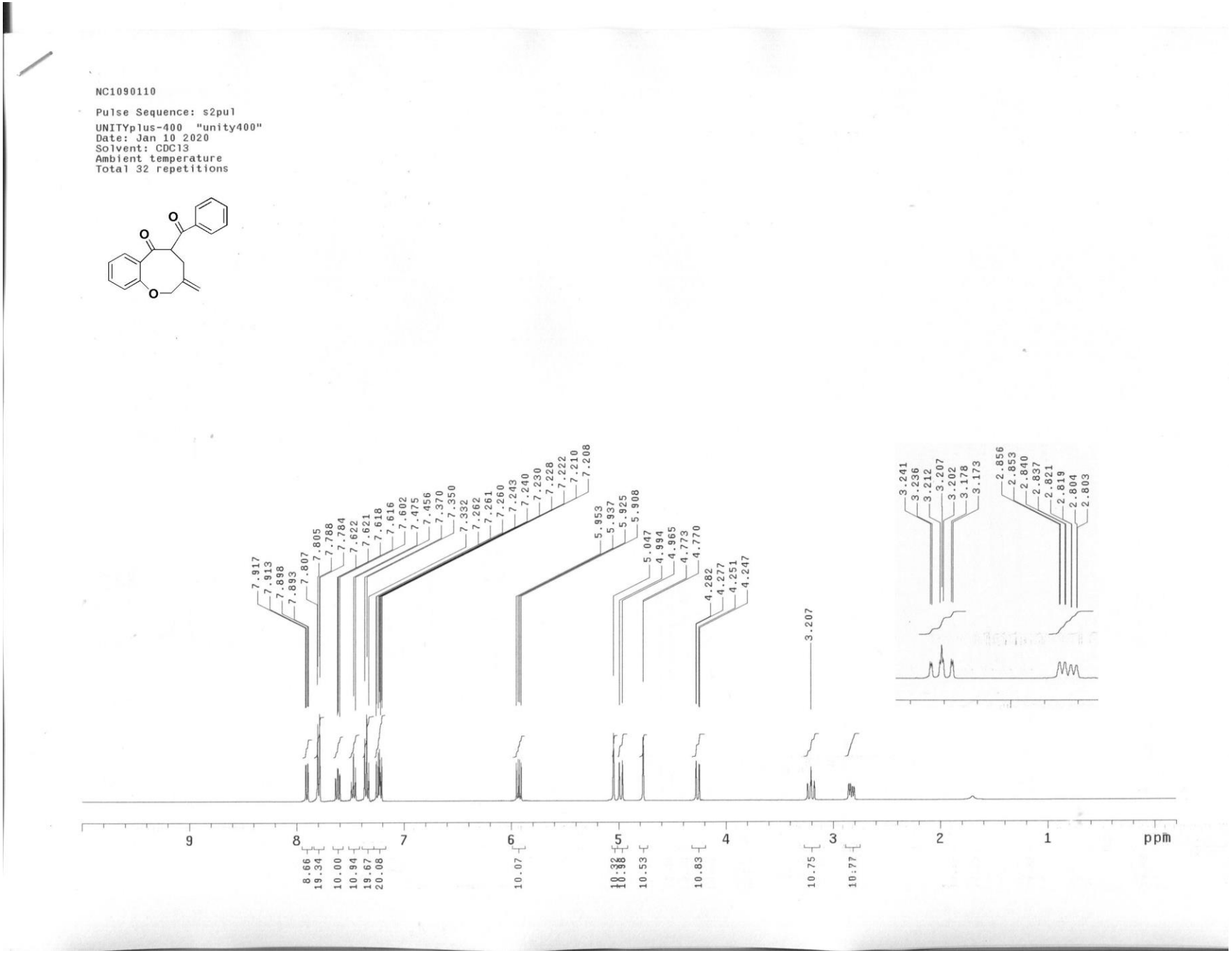
Date: Apr 17 2020
Solvent: CDCl3
Ambient temperature
Total 1792 repetitions

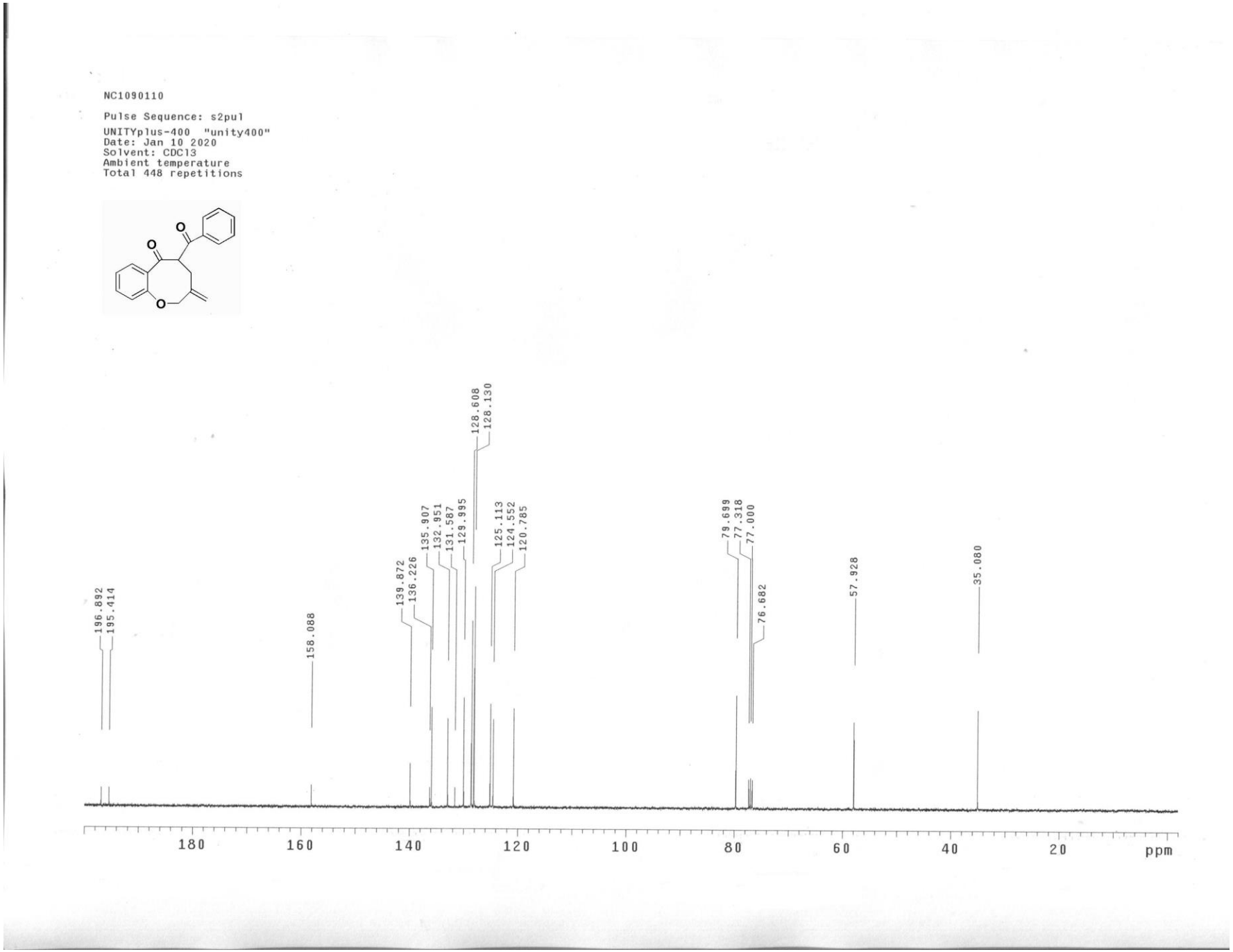




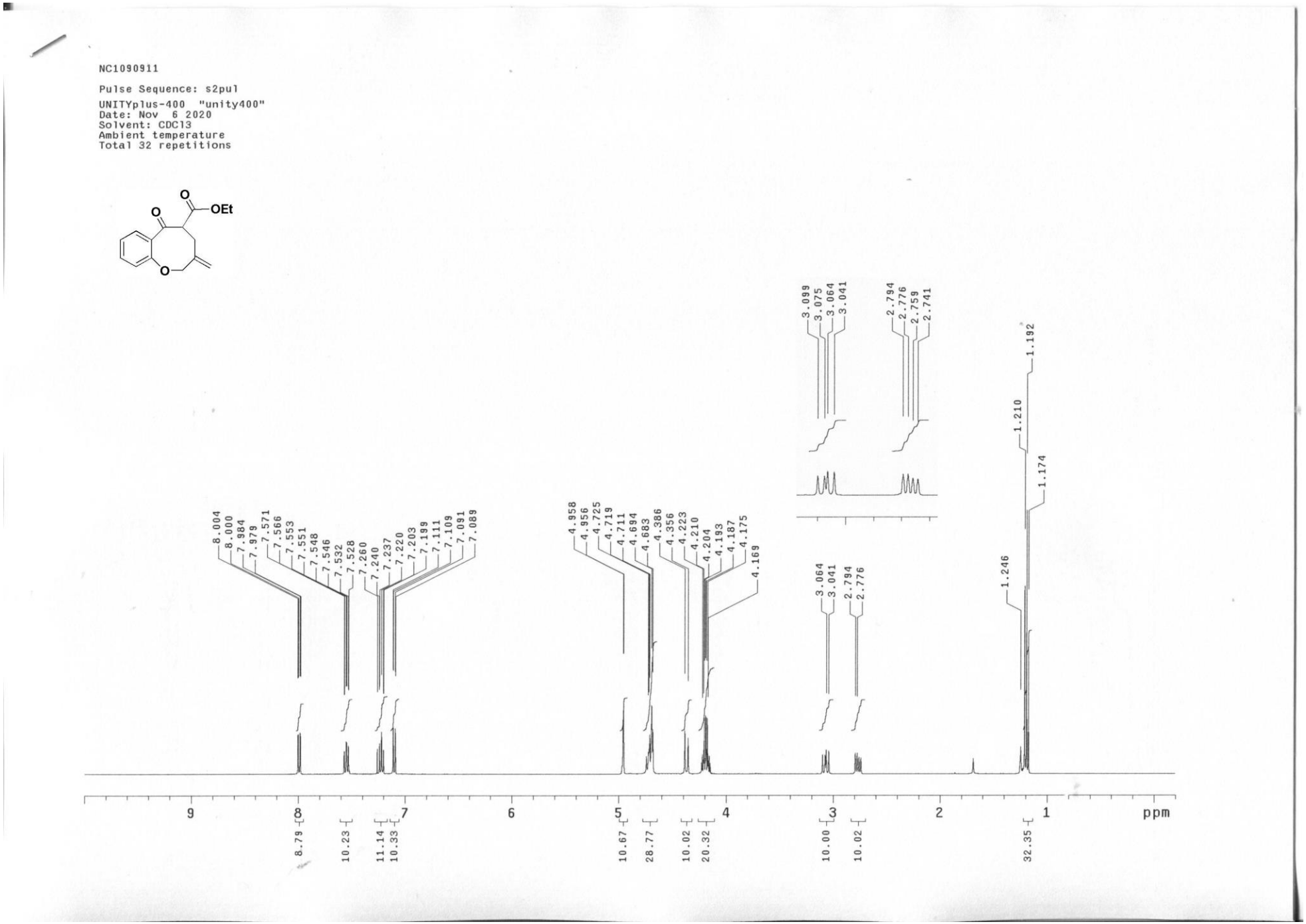
Compound 4y (^{1}H-NMR spectral data)



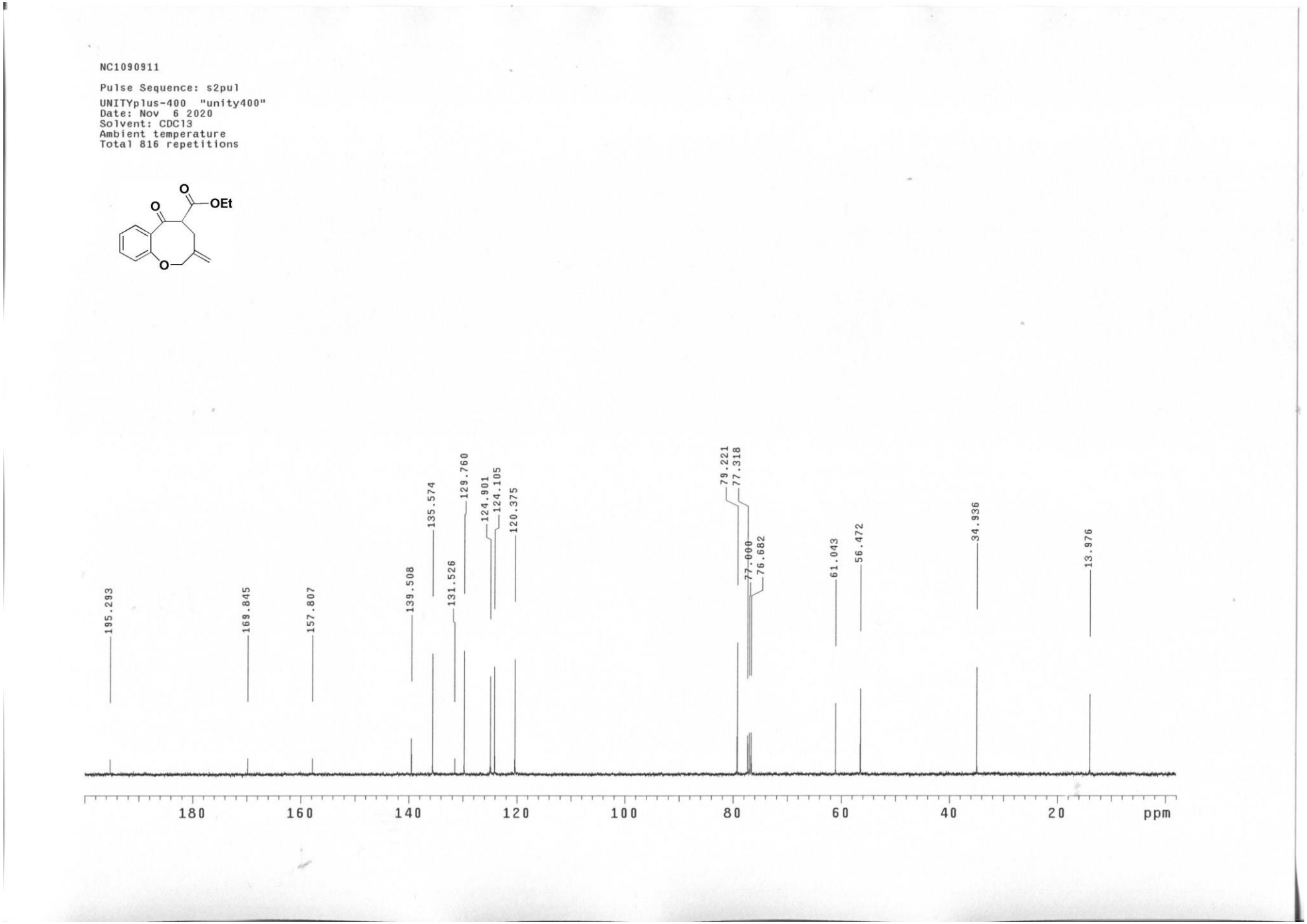
Compound 4y (^{13}C-NMR spectral data)



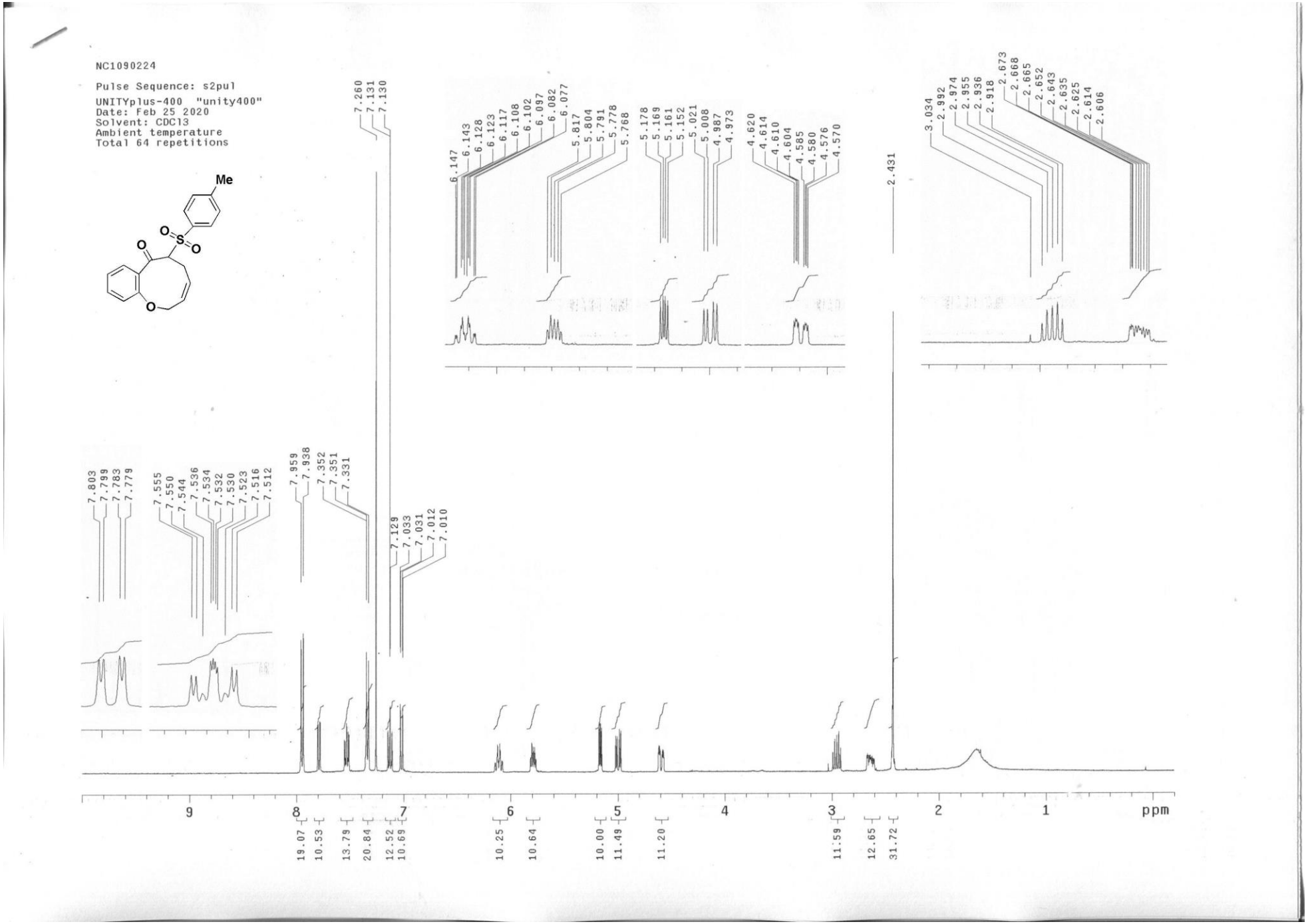
Compound 4z (^{1}H-NMR spectral data)



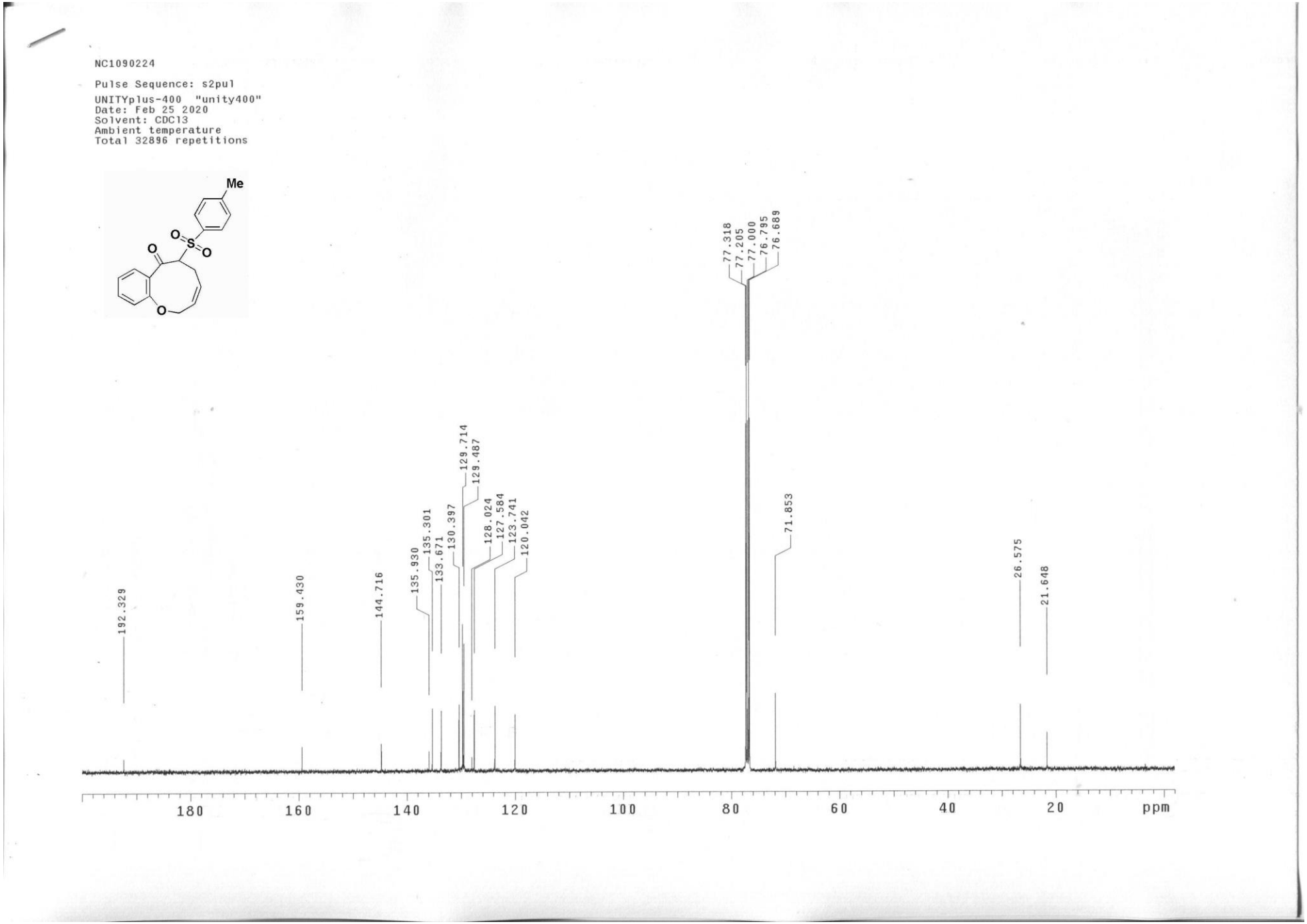
Compound 4z (^{13}C-NMR spectral data)



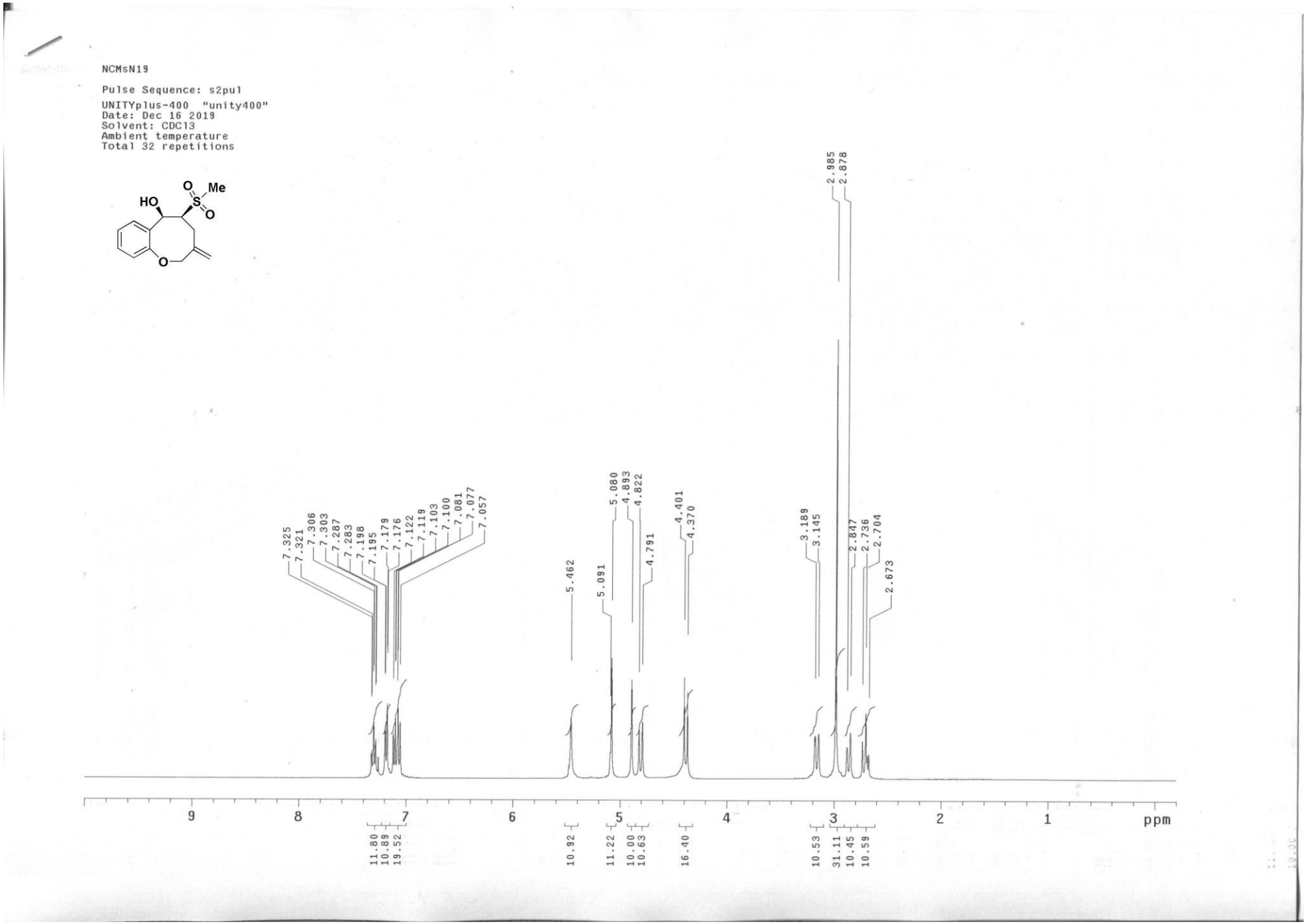
Compound 4aa (^{1}H-NMR spectral data)



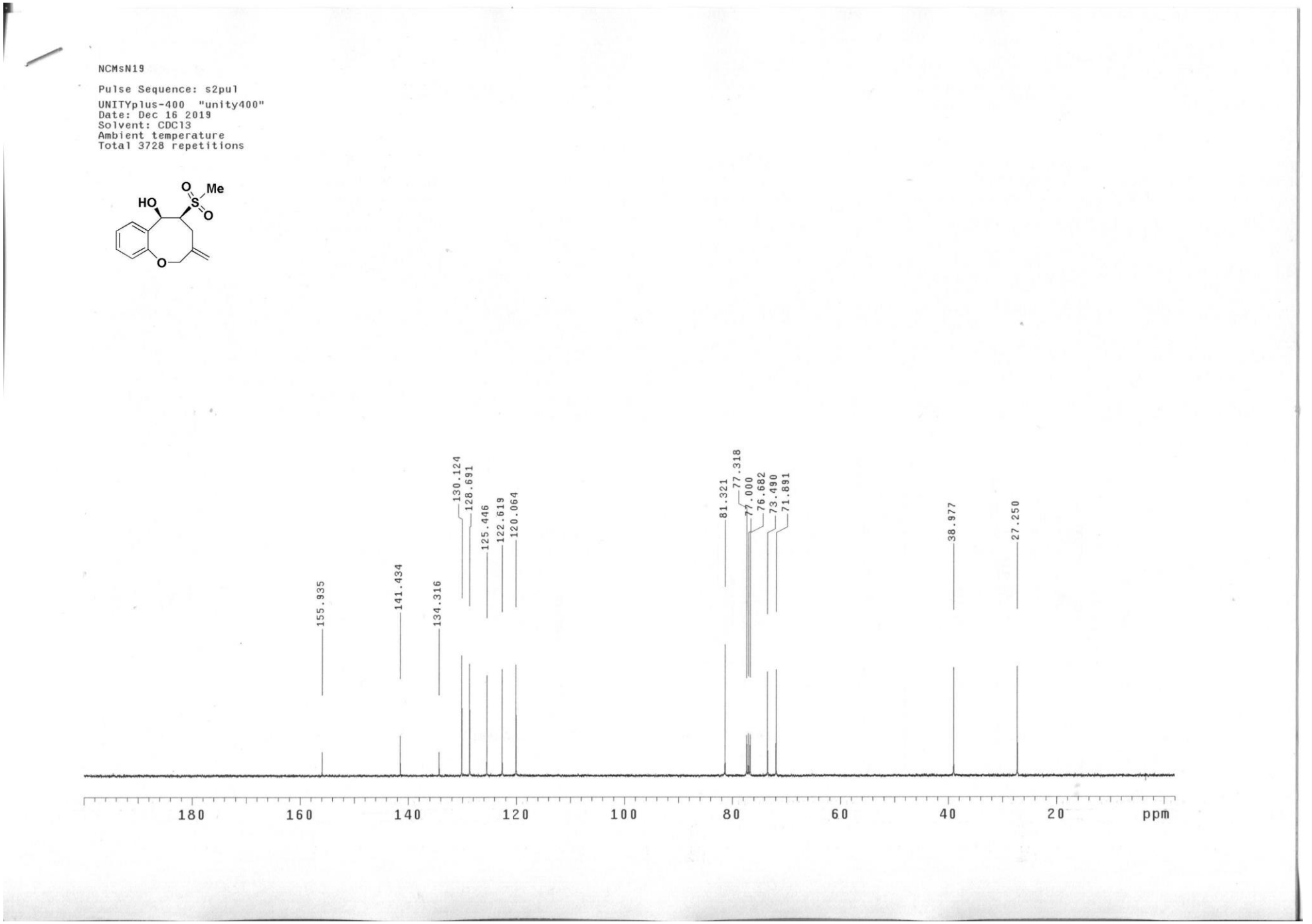
Compound 4aa (^{13}C-NMR spectral data)



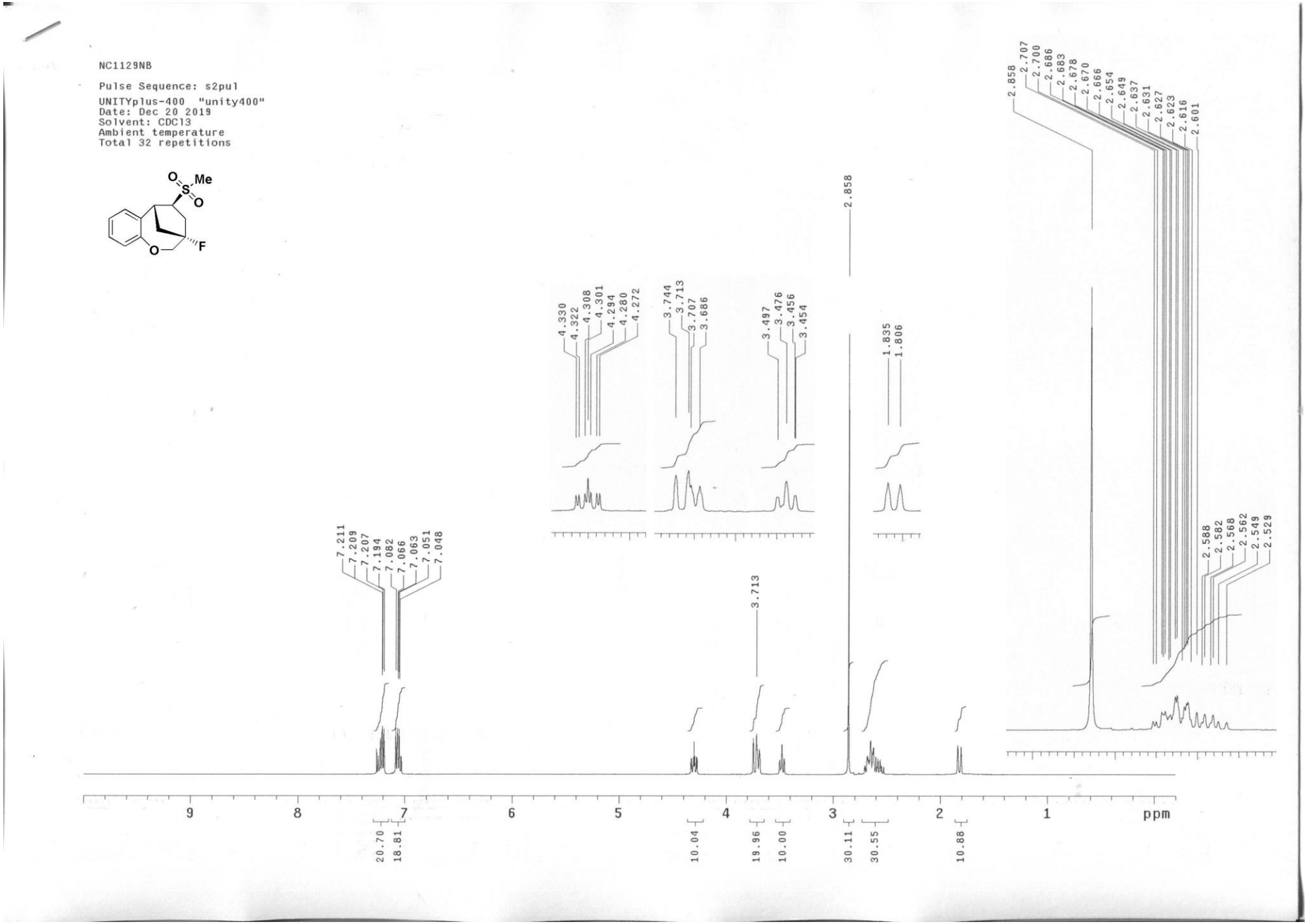
Compound 6a (^{1}H-NMR spectral data)



Compound 6a (^{13}C-NMR spectral data)



Compound 5a (^{1}H-NMR spectral data)

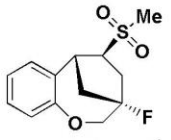


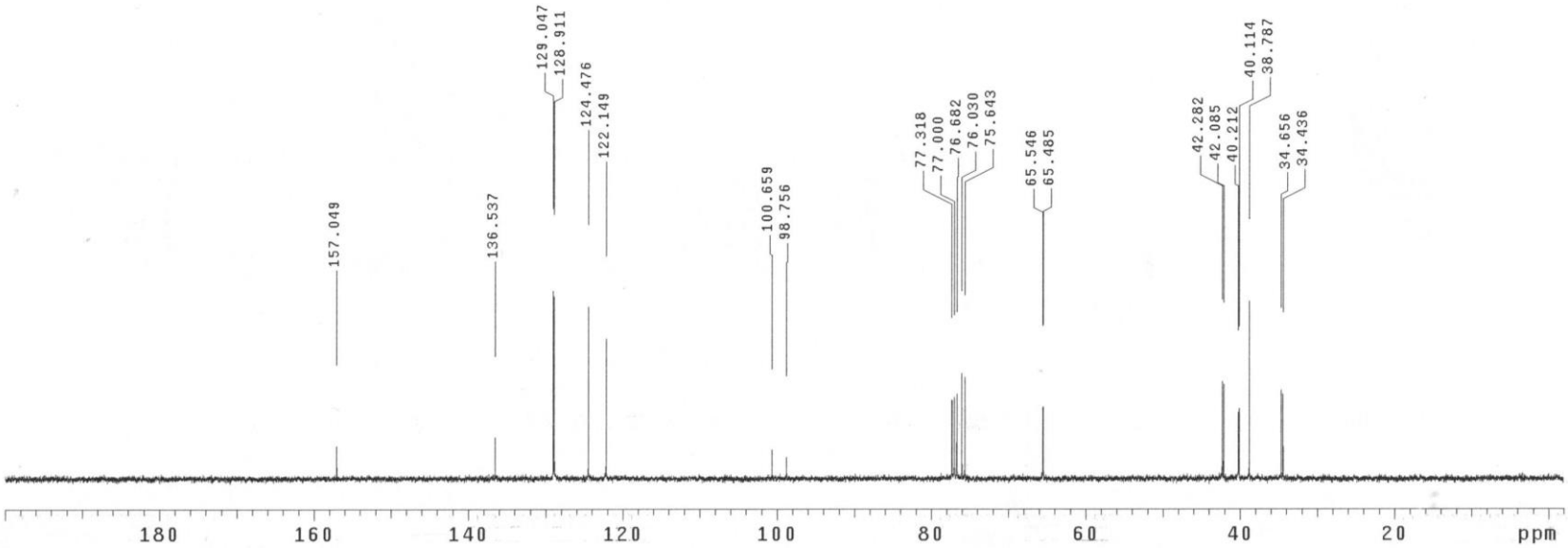
Compound 5a (^{13}C-NMR spectral data)

NC1129NB

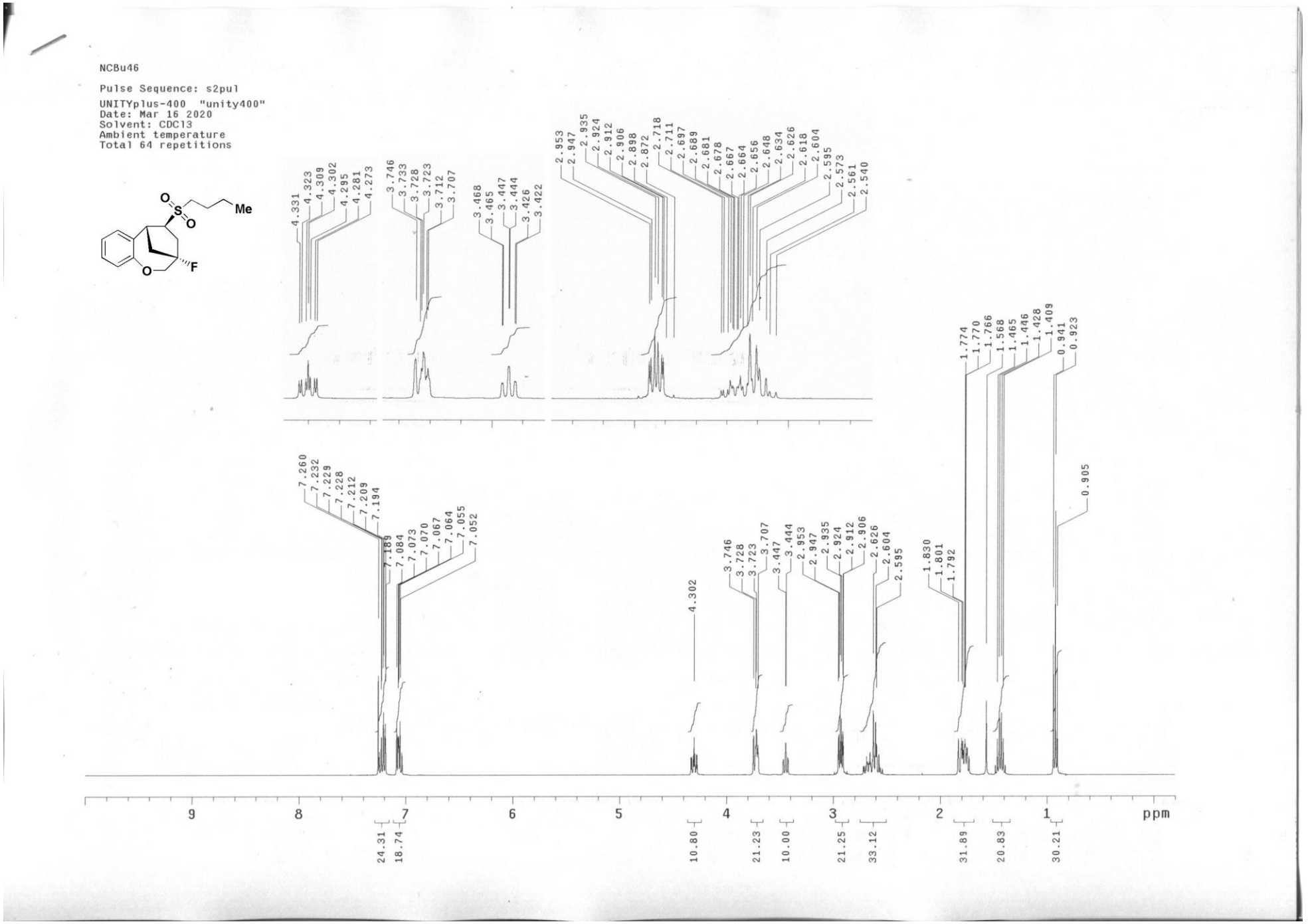
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Dec 20 2019
Solvent: CDCl3
Ambient temperature
Total 560 repetitions

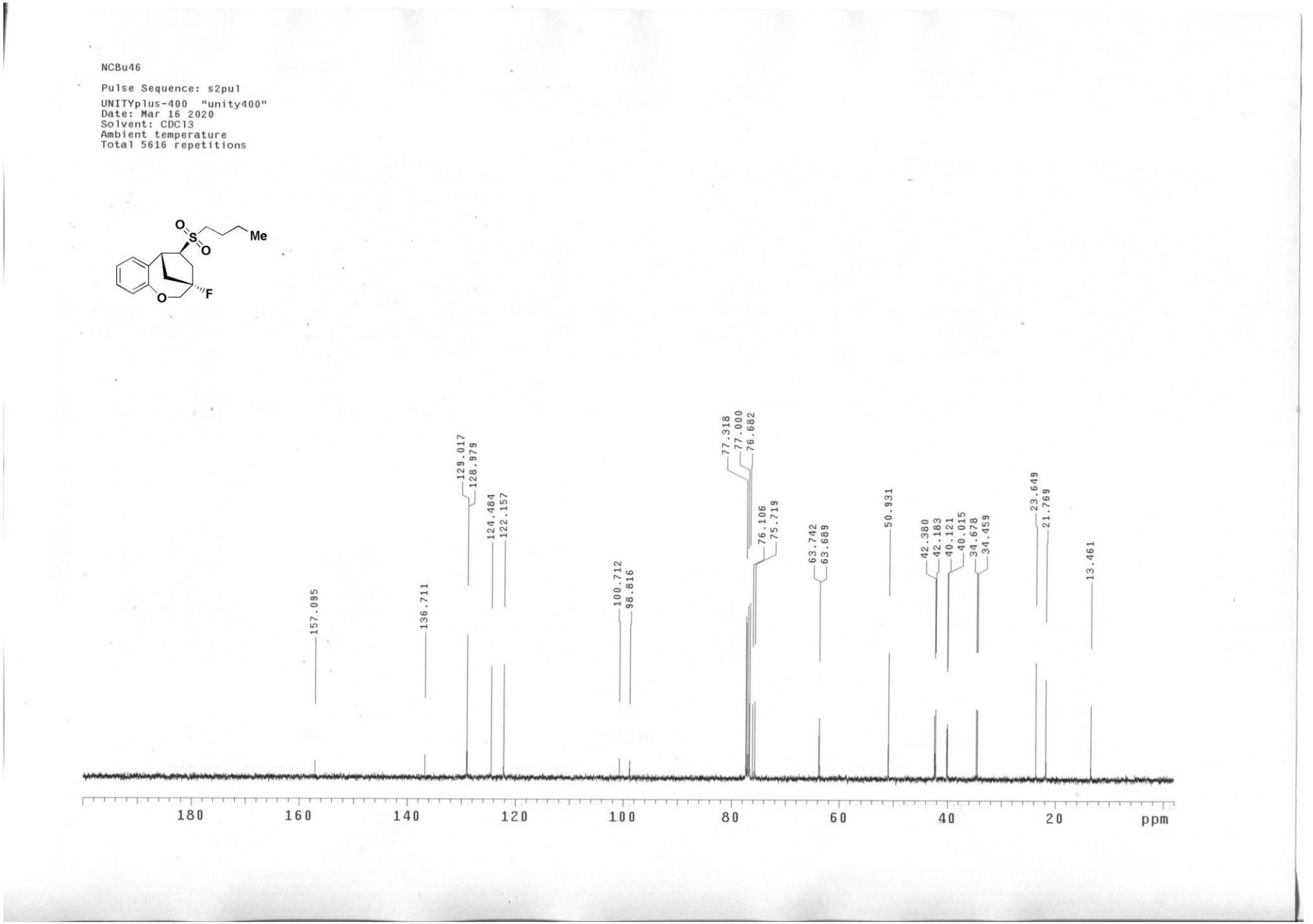




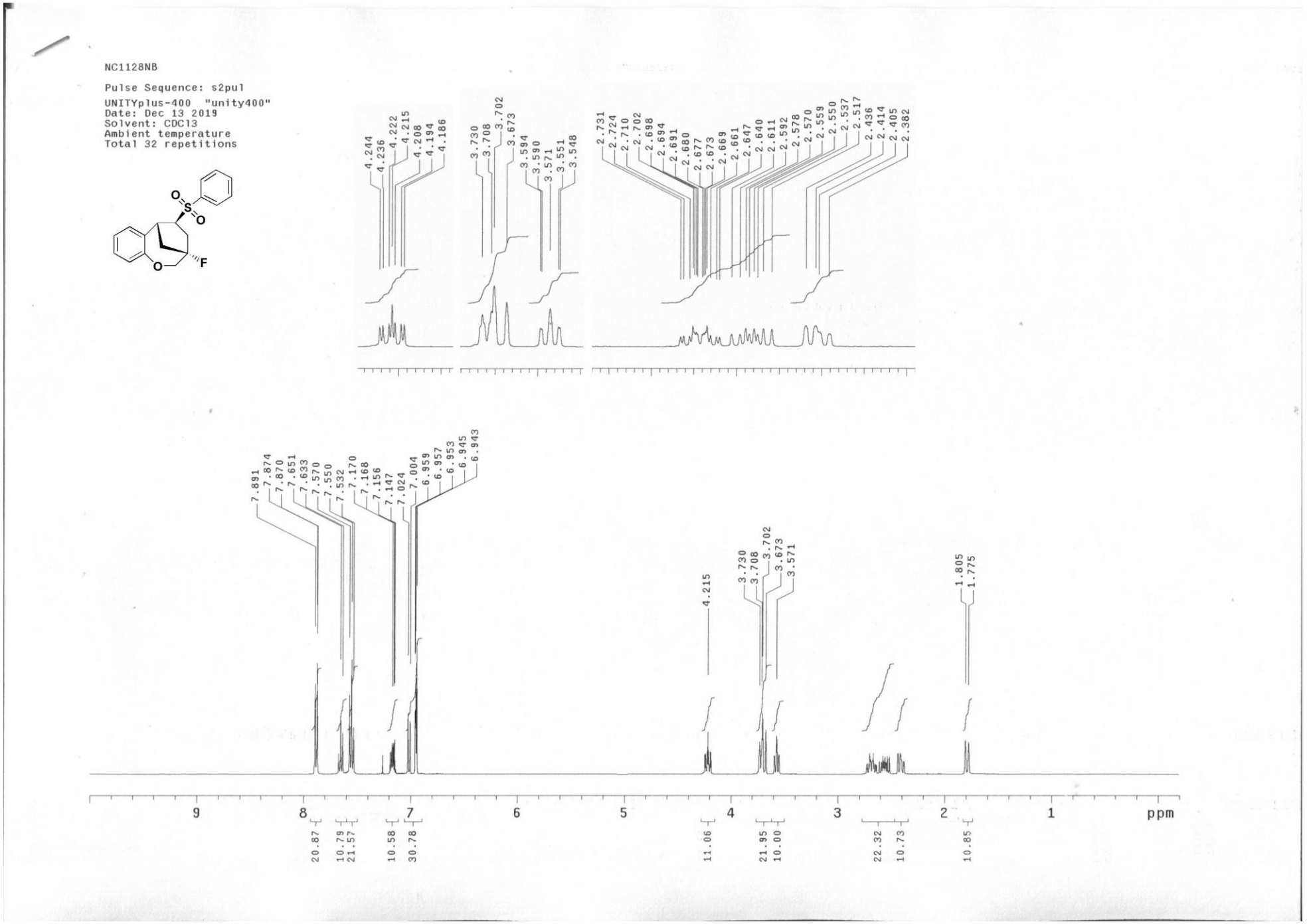
Compound 5b (^{1}H-NMR spectral data)



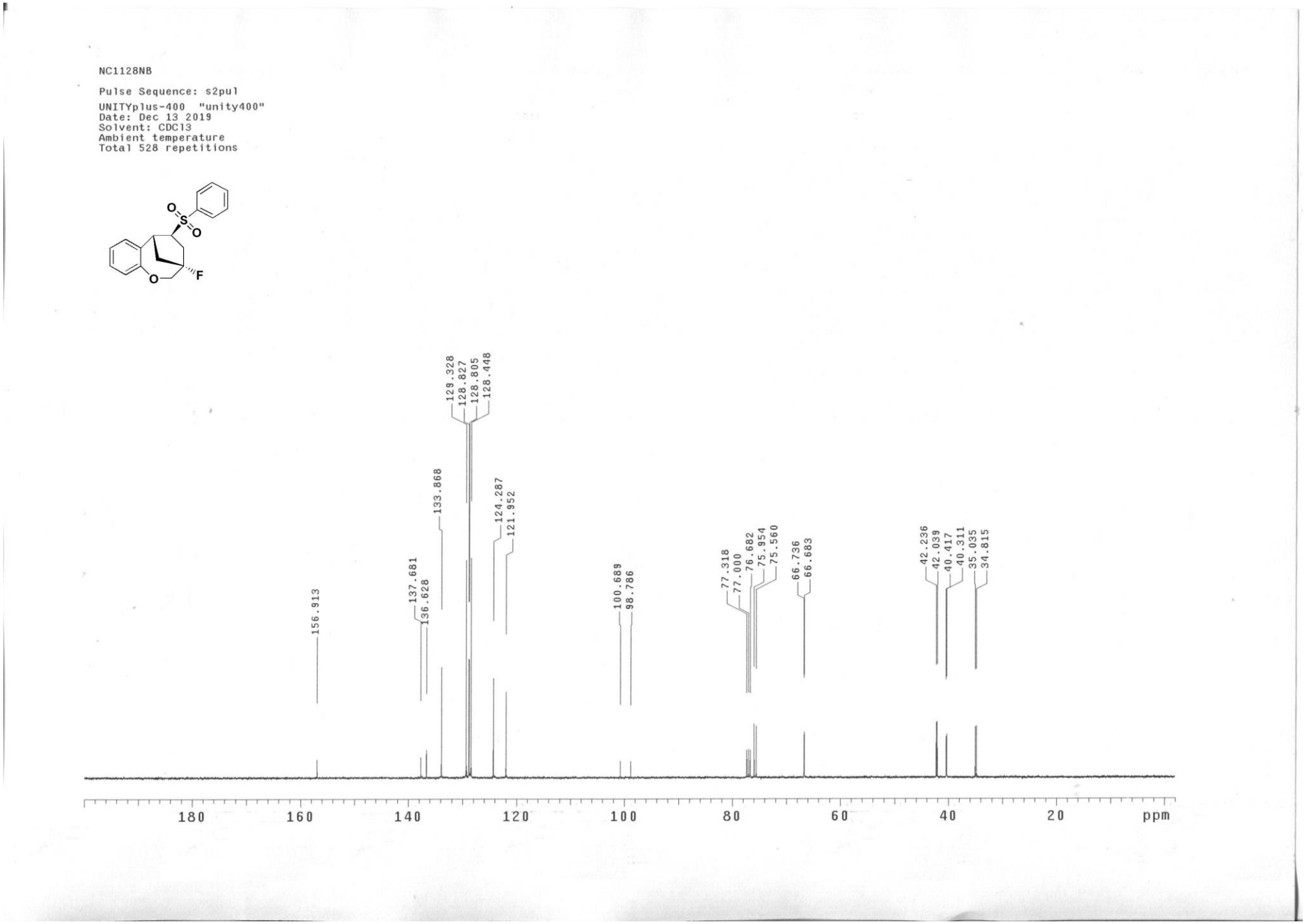
Compound 5b (^{13}C-NMR spectral data)



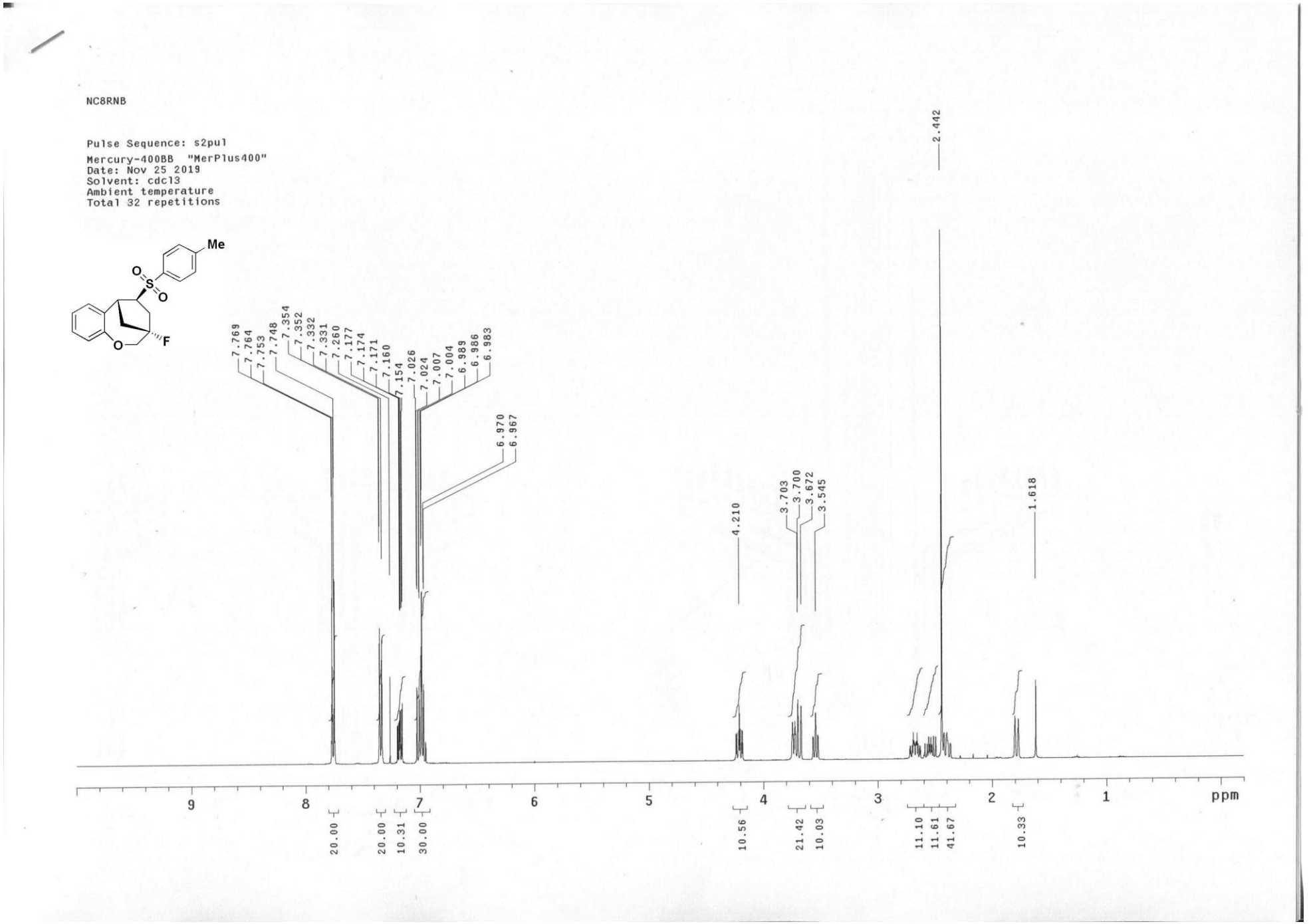
Compound 5c (^{1}H-NMR spectral data)



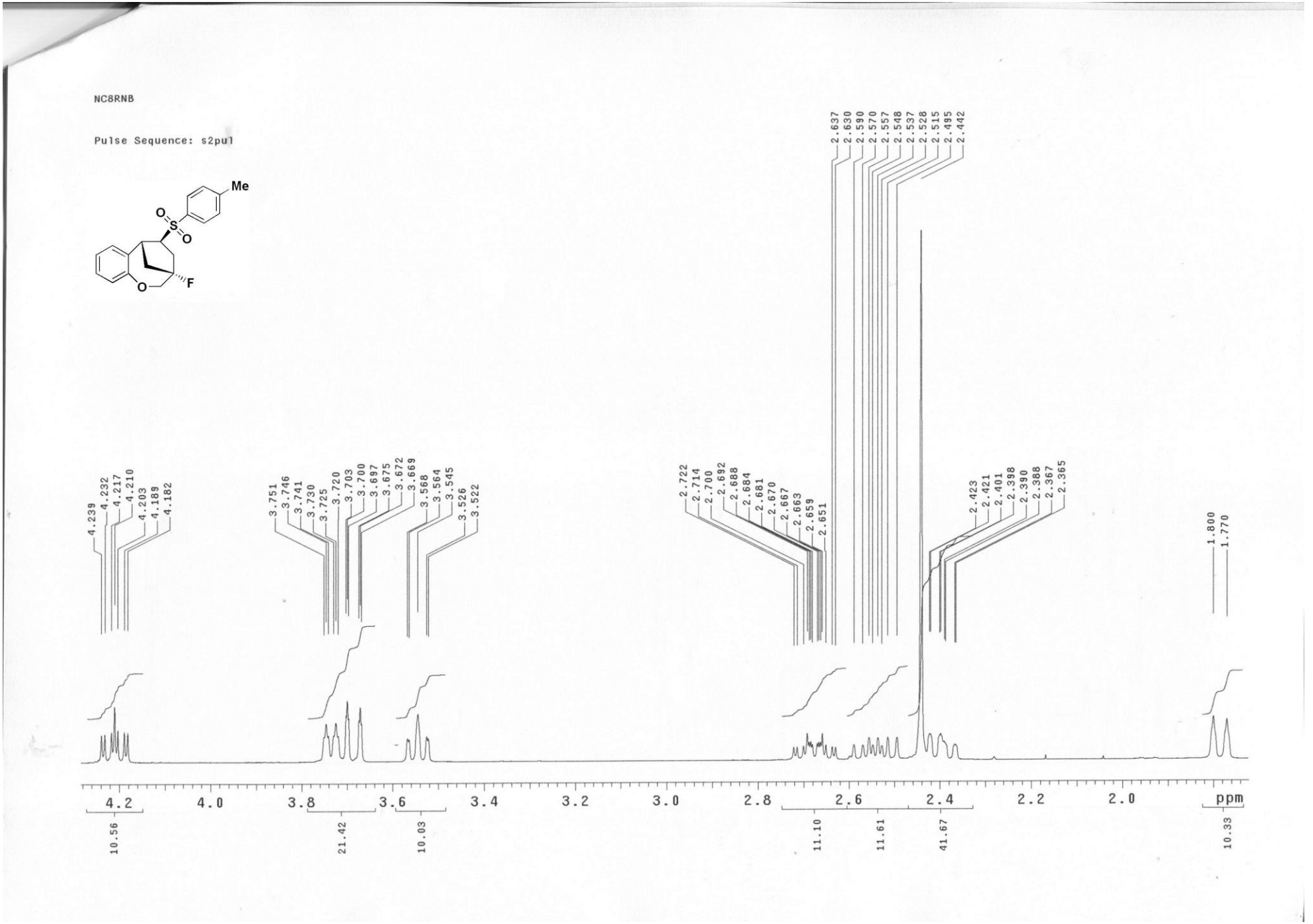
Compound 5c (^{13}C-NMR spectral data)



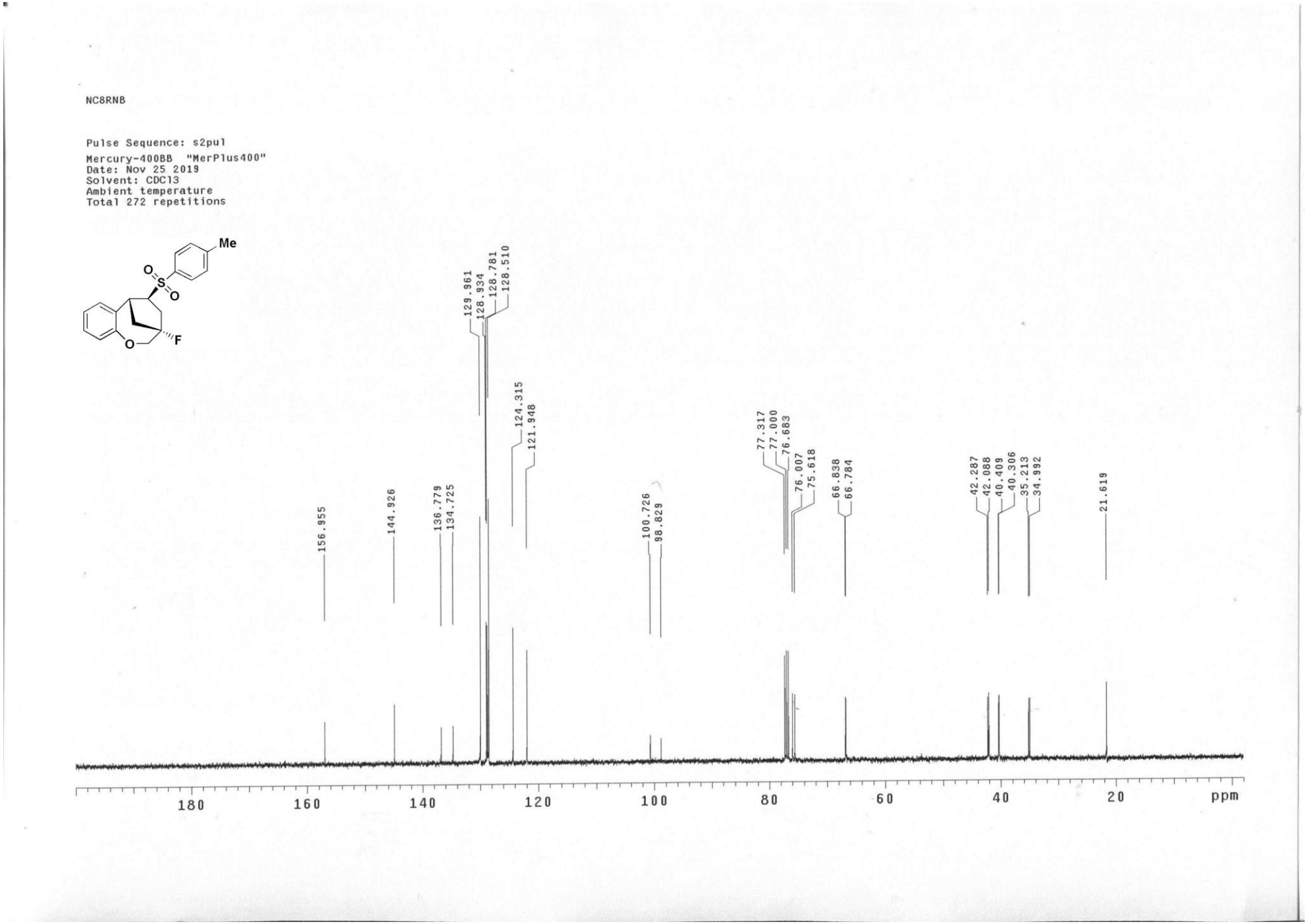
Compound 5d (^{1}H-NMR spectral data-1)



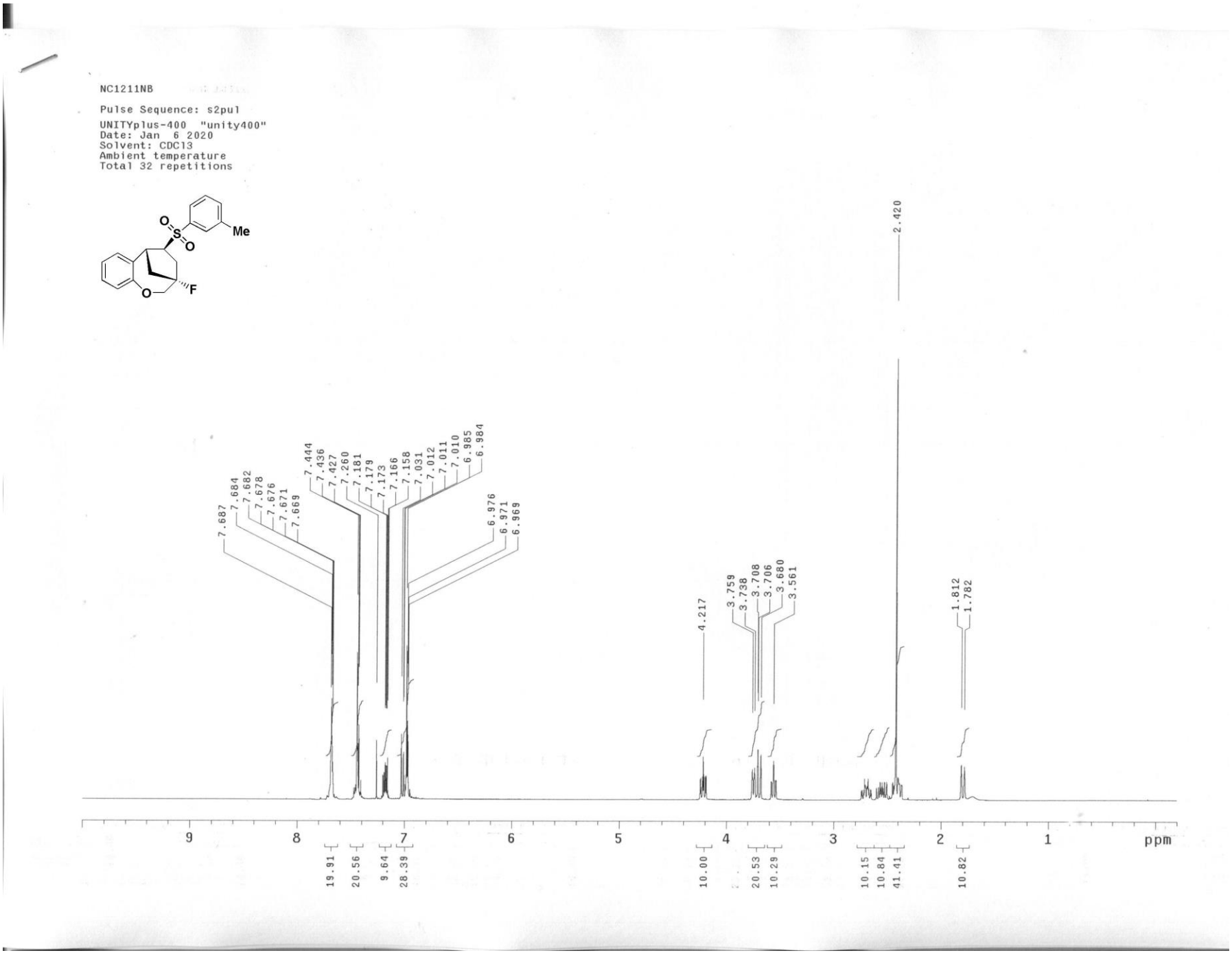
Compound 5d (^{1}H-NMR spectral data-2)



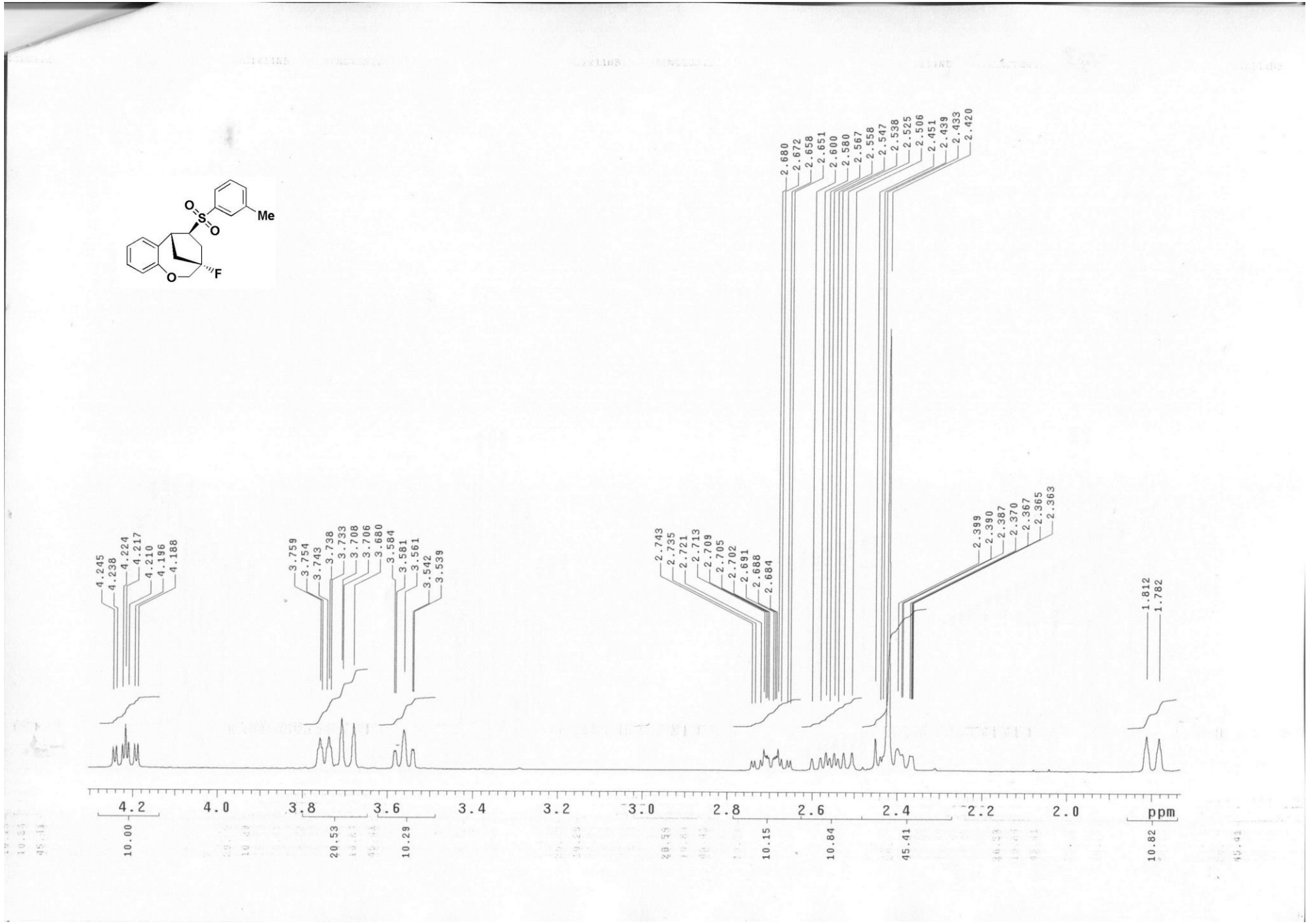
Compound 5d (^{13}C-NMR spectral data)



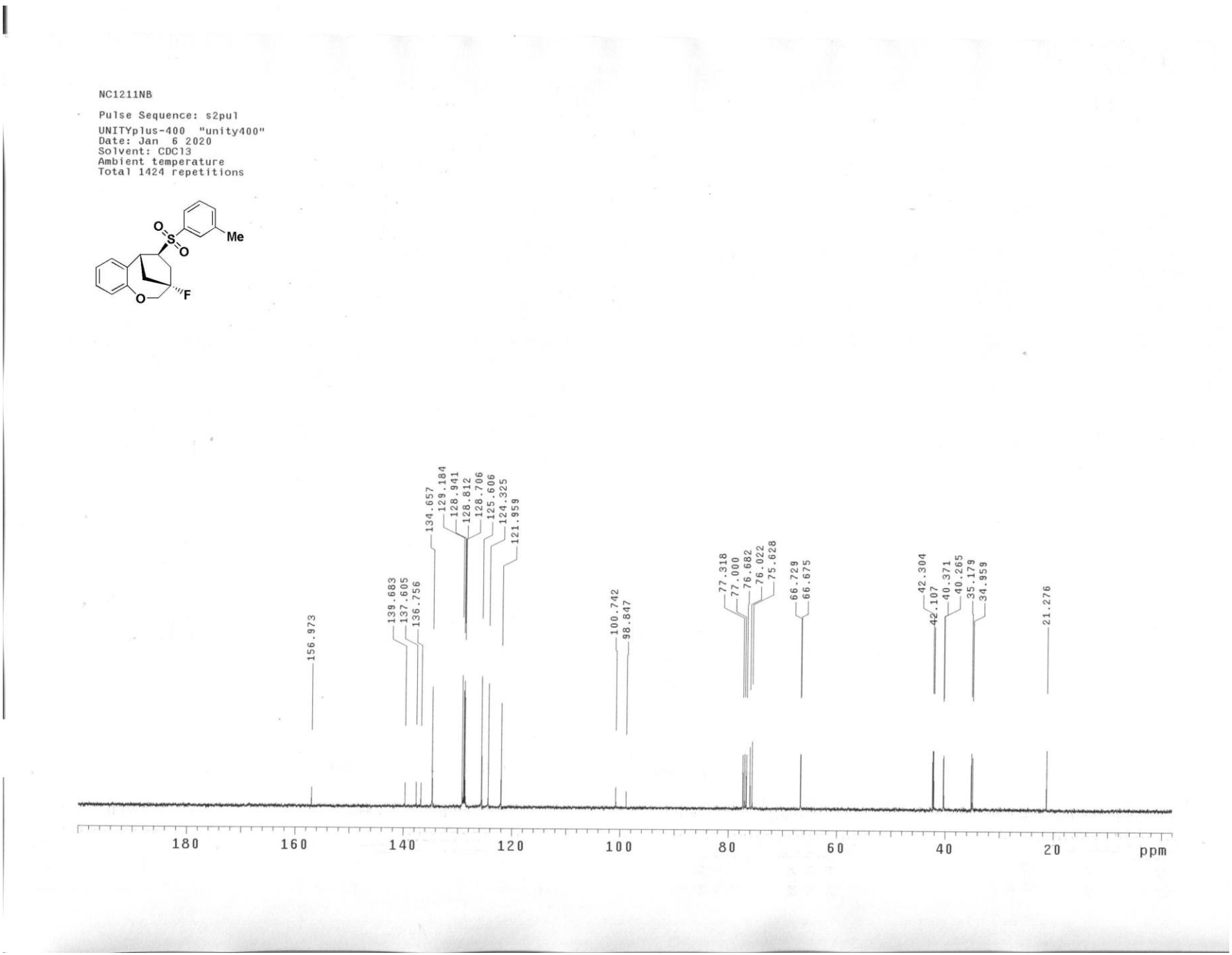
Compound 5e (^{1}H-NMR spectral data-1)



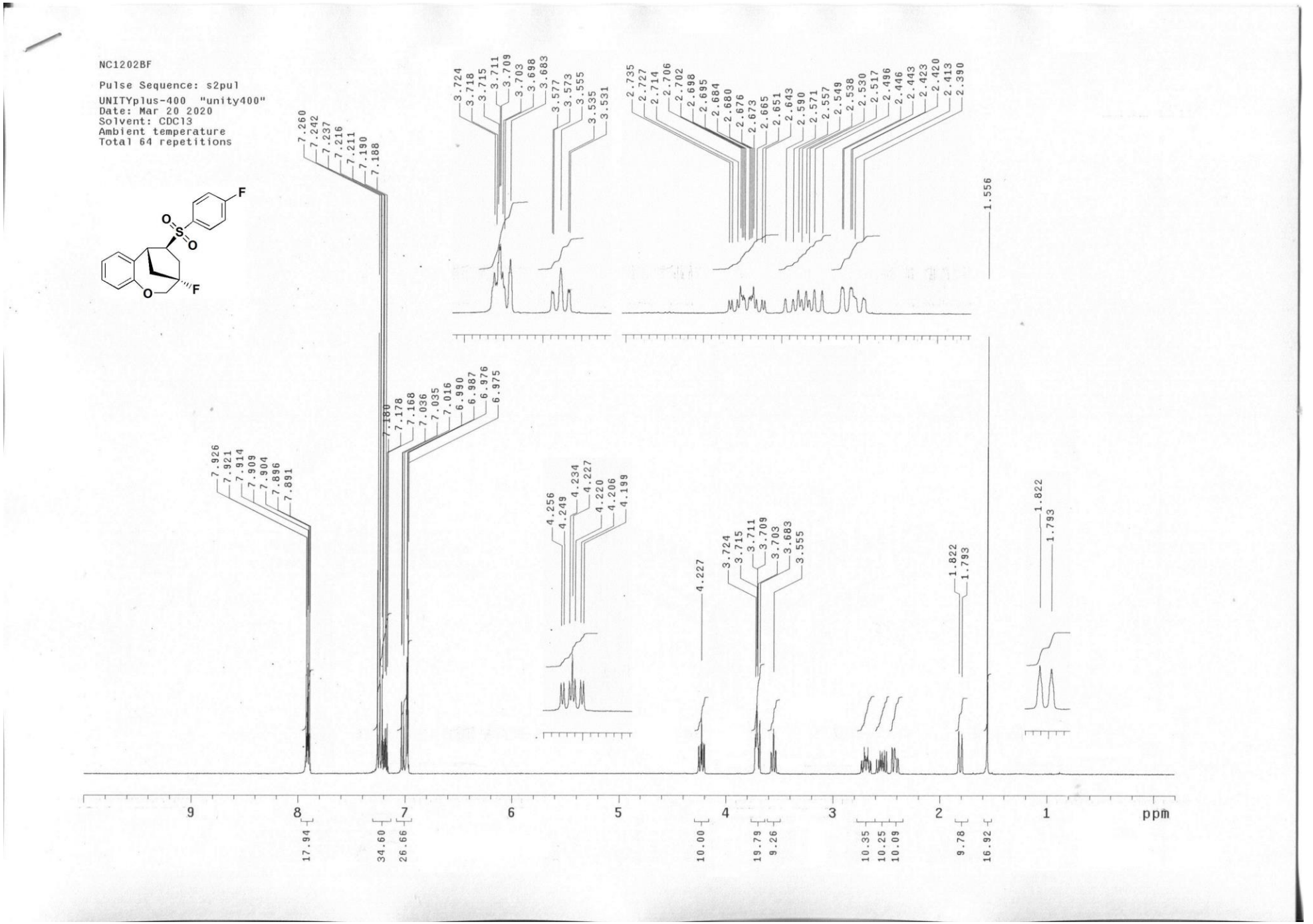
Compound 5e (^{1}H-NMR spectral data-2)



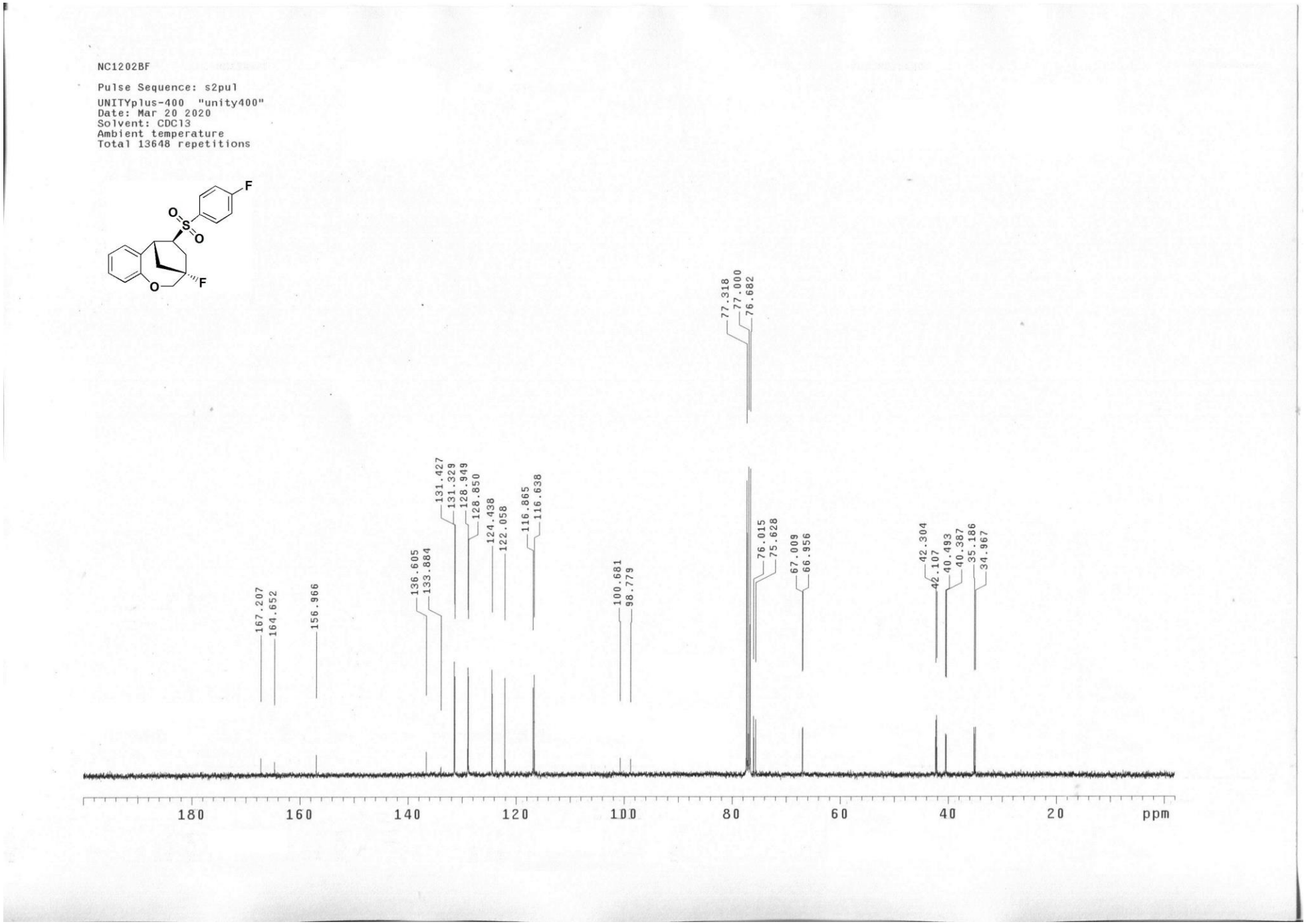
Compound 5e (^{13}C-NMR spectral data)



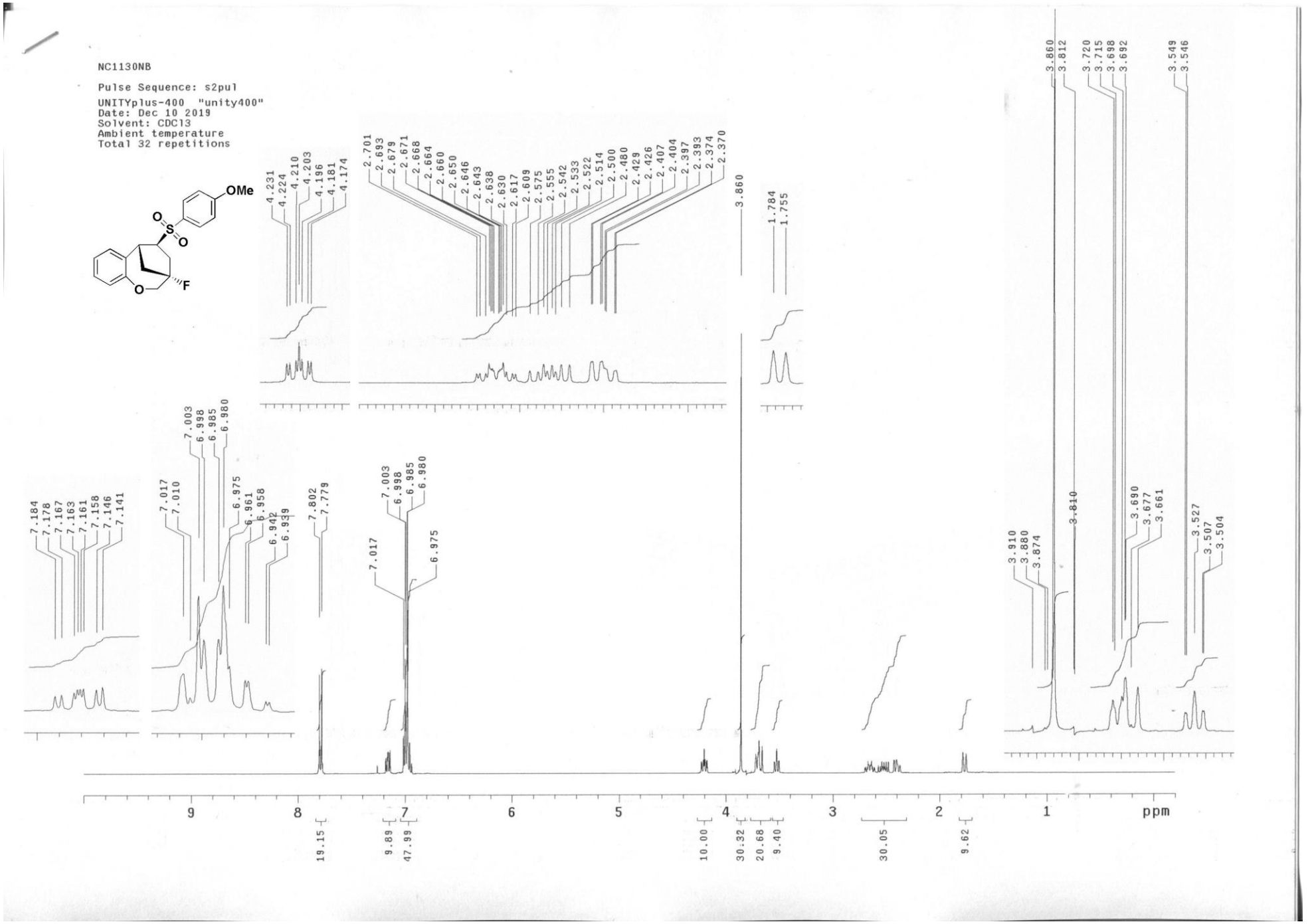
Compound 5f (^{1}H-NMR spectral data)



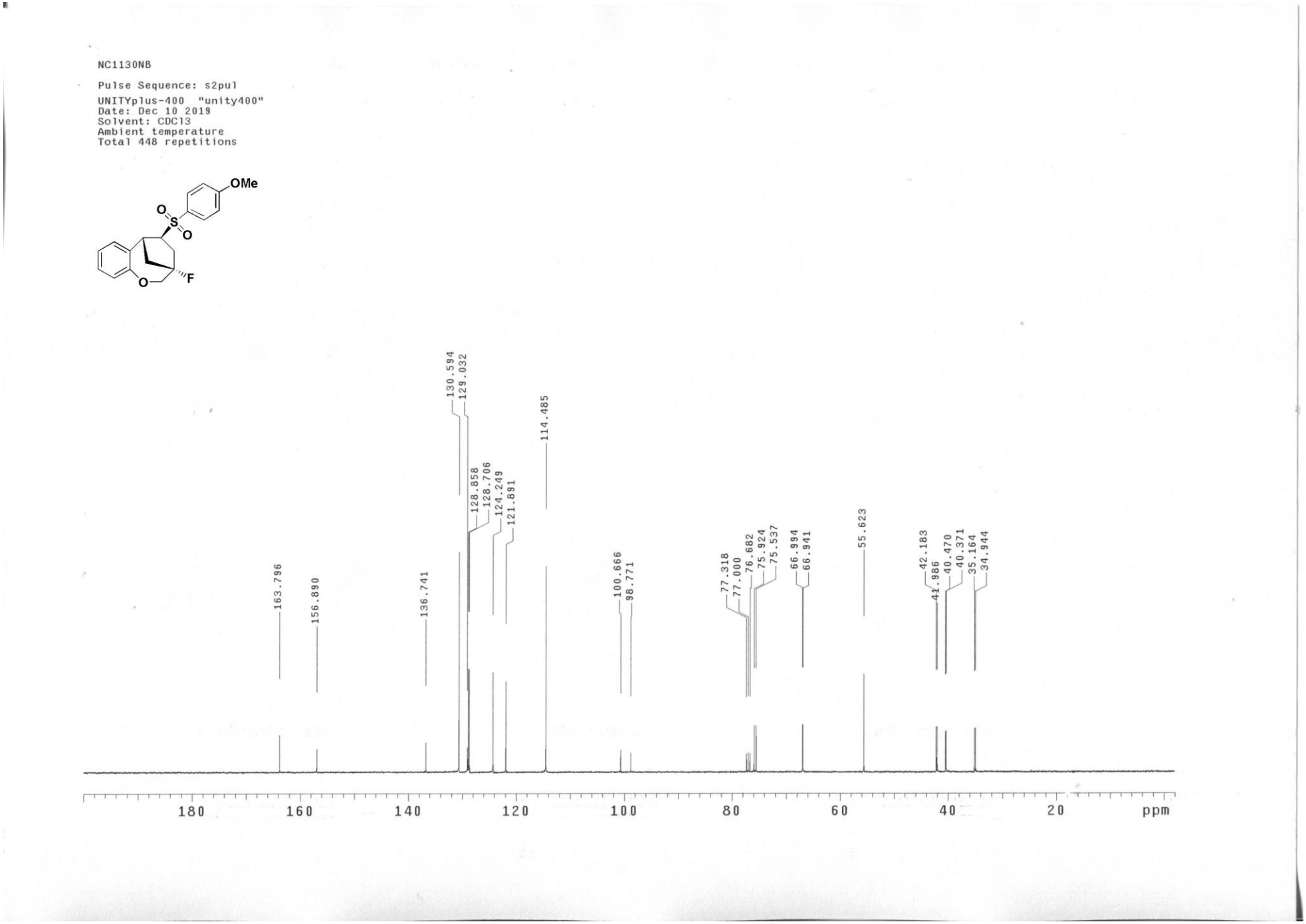
Compound 5f (^{13}C-NMR spectral data)



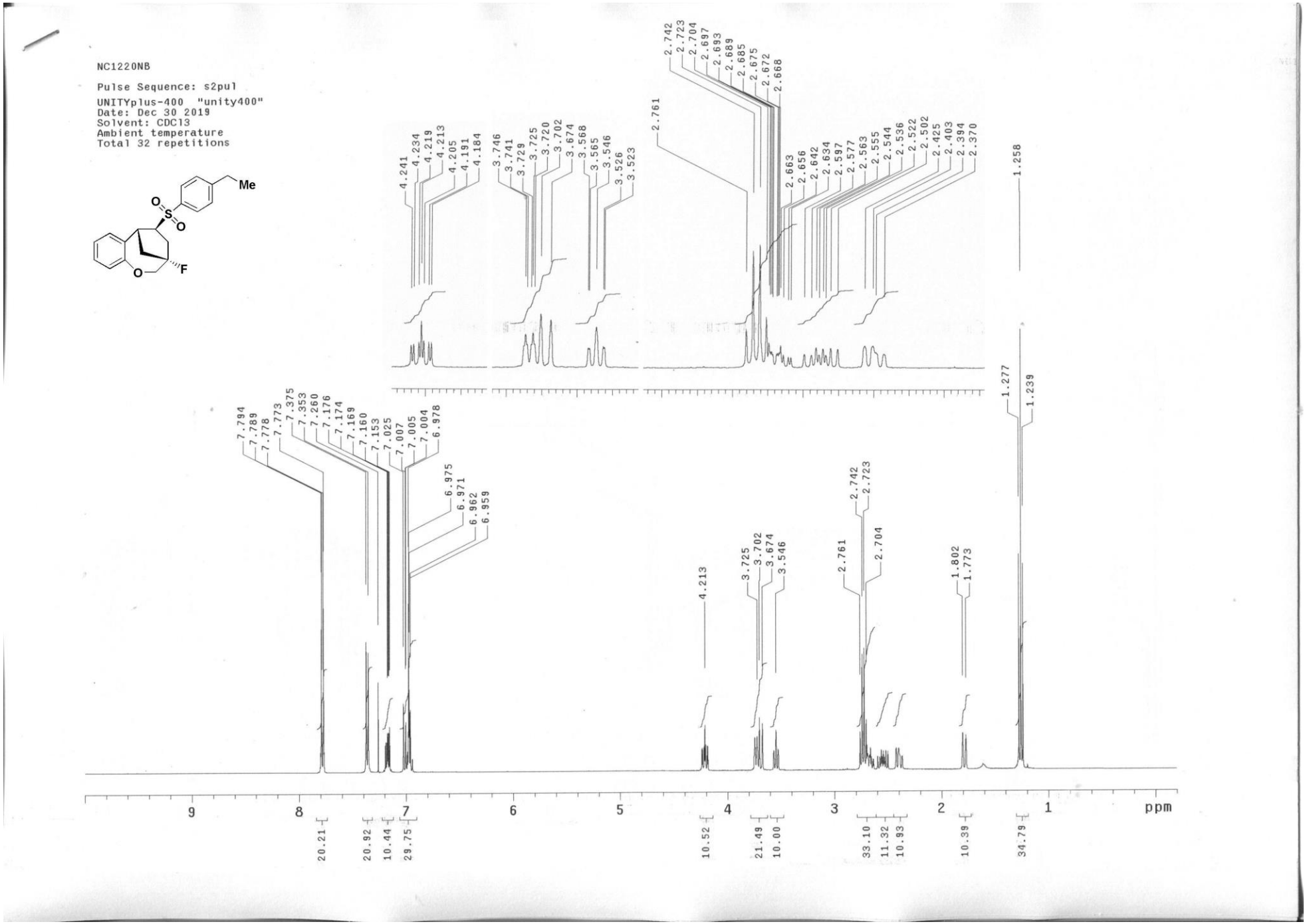
Compound 5g (^{1}H-NMR spectral data)



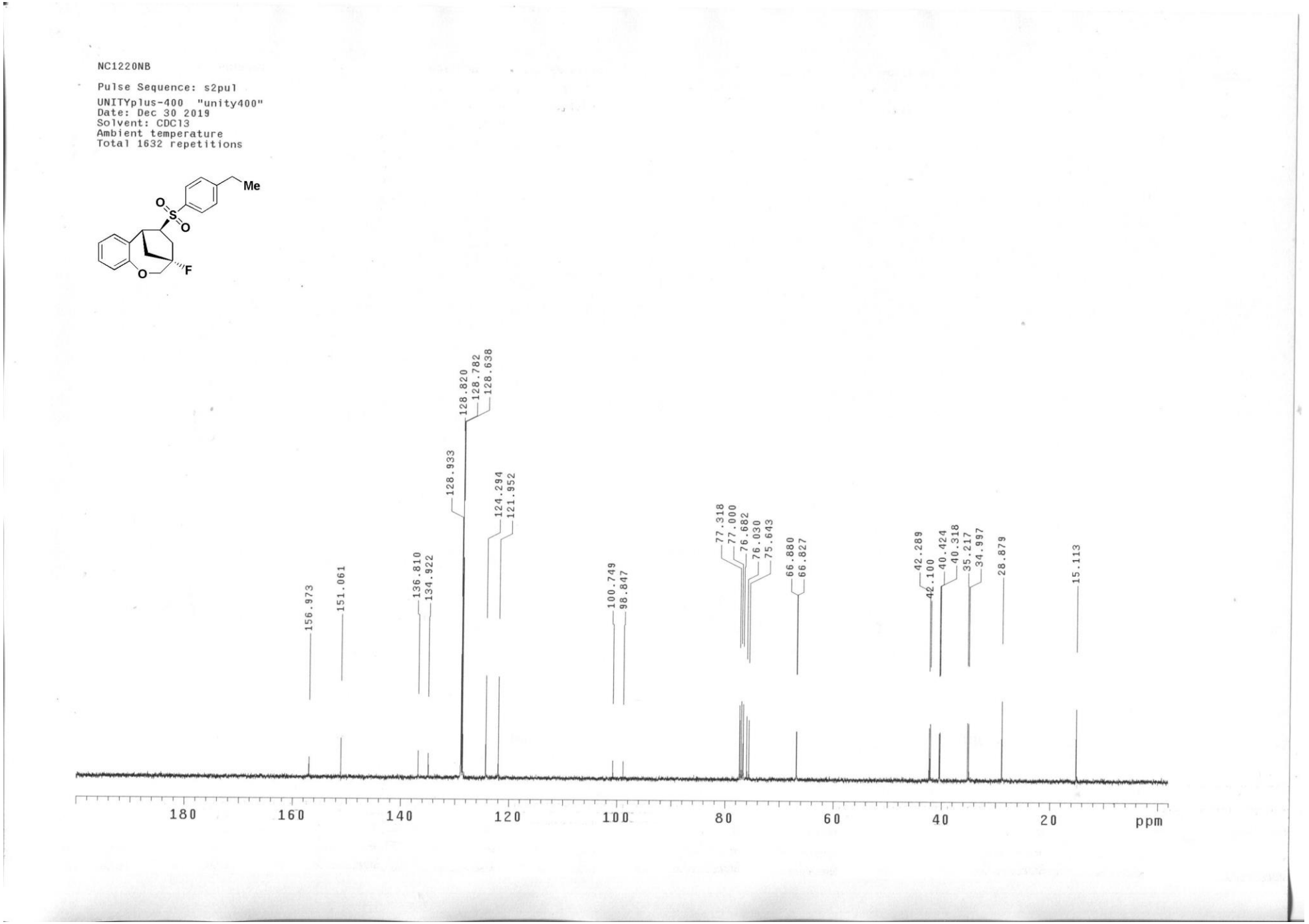
Compound 5g (^{13}C-NMR spectral data)



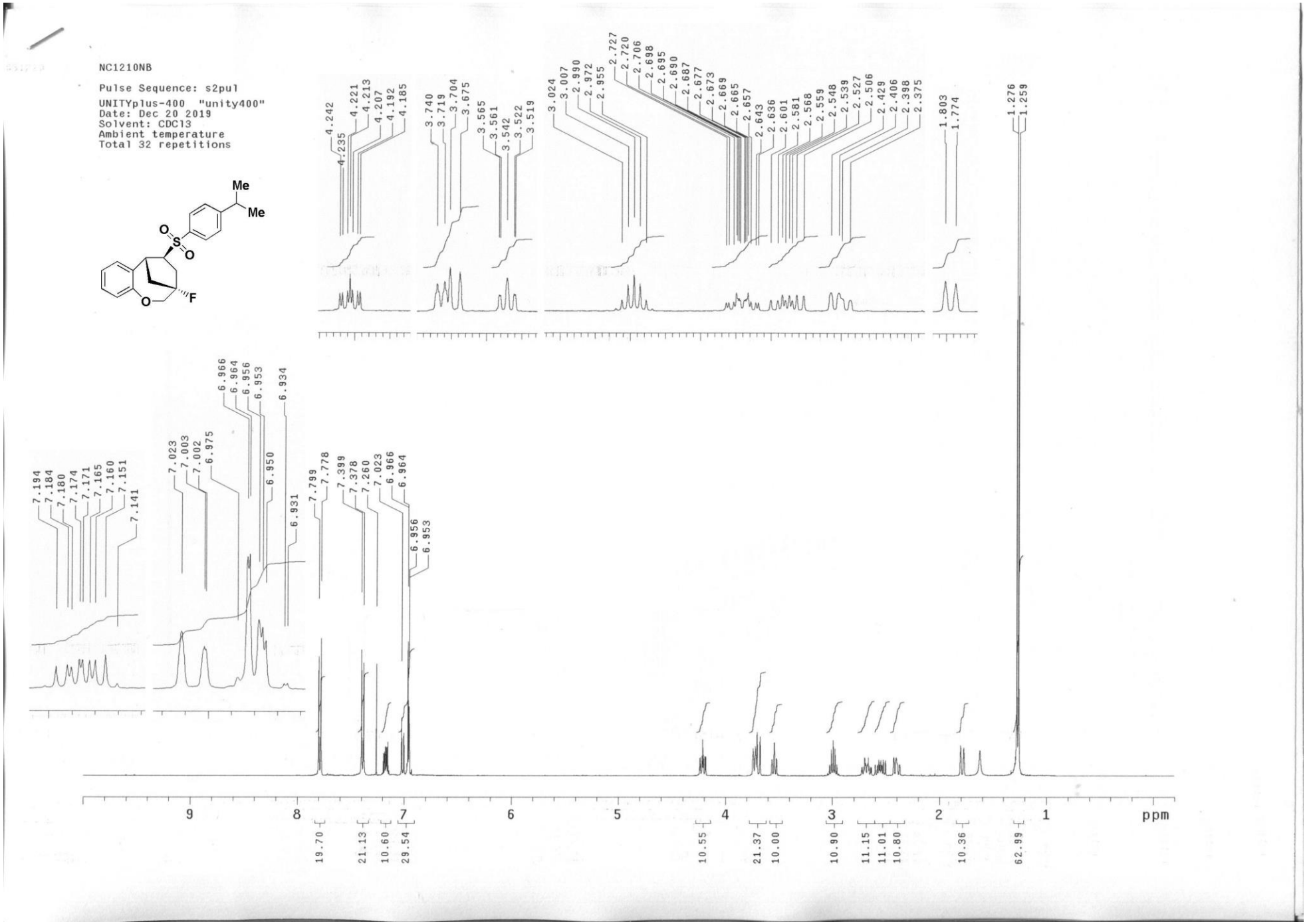
Compound 5h (^{1}H-NMR spectral data)



Compound 5h (^{13}C-NMR spectral data)



Compound 5i (^{1}H-NMR spectral data)

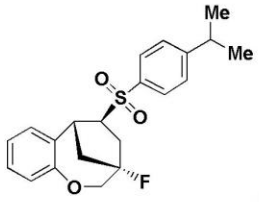


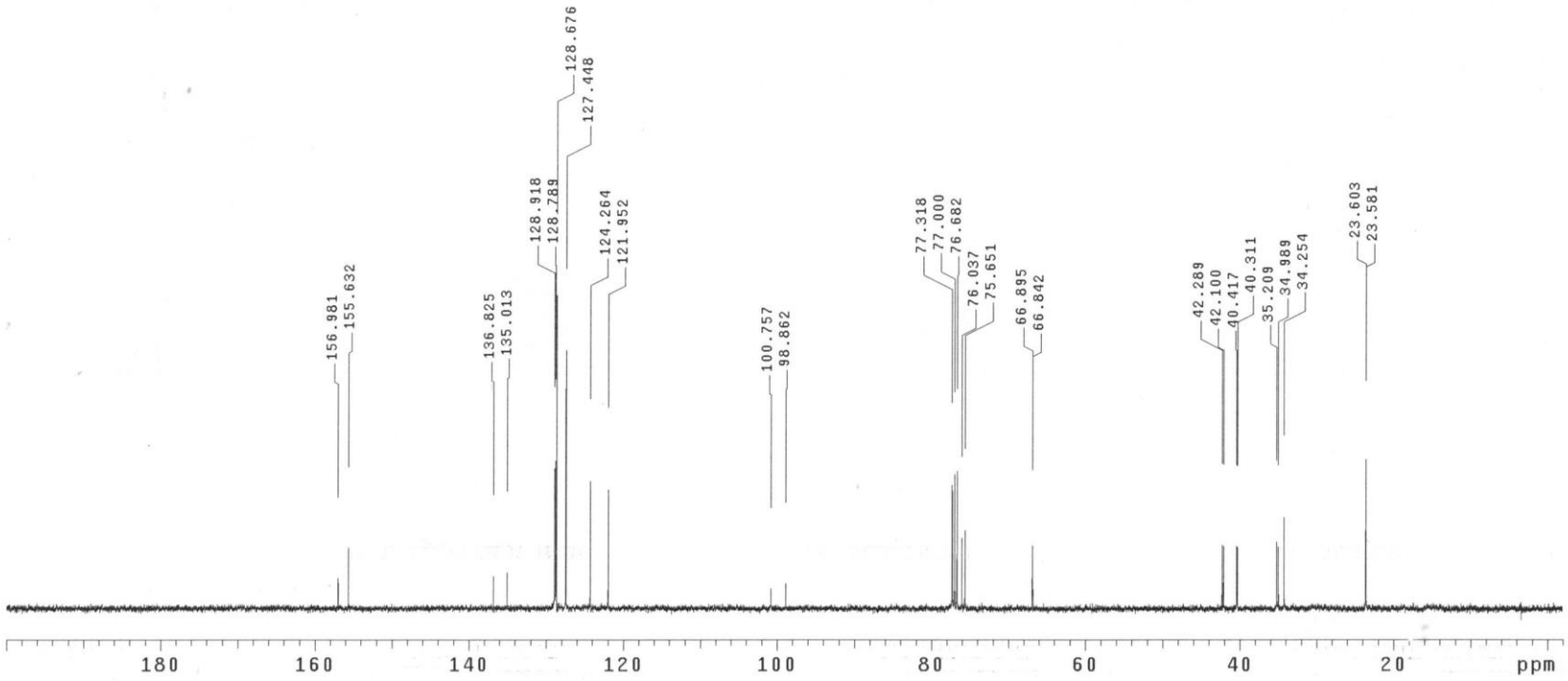
Compound 5i (^{13}C-NMR spectral data)

NC1210NB

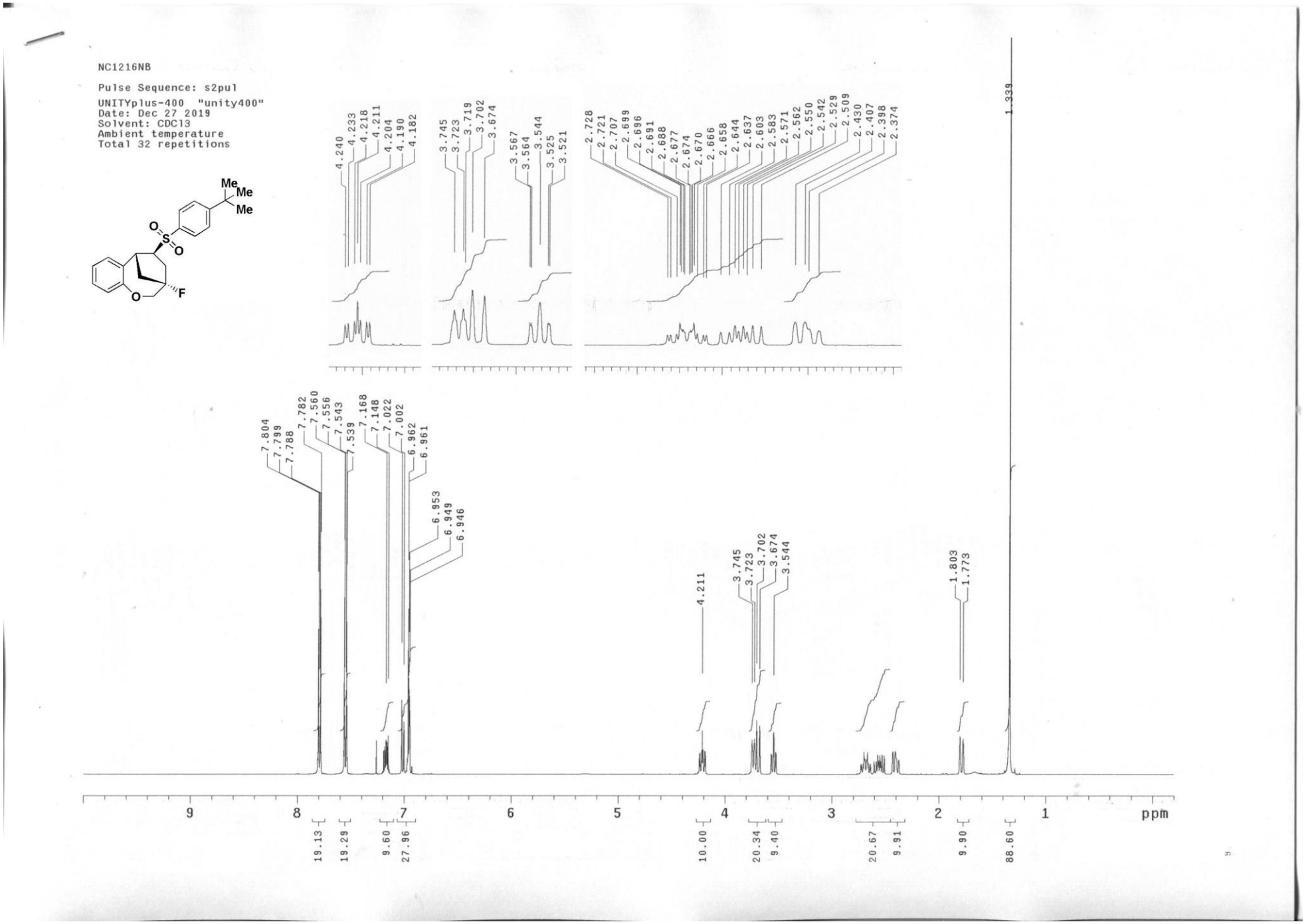
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Dec 20 2019
Solvent: CDCl3
Ambient temperature
Total 2400 repetitions

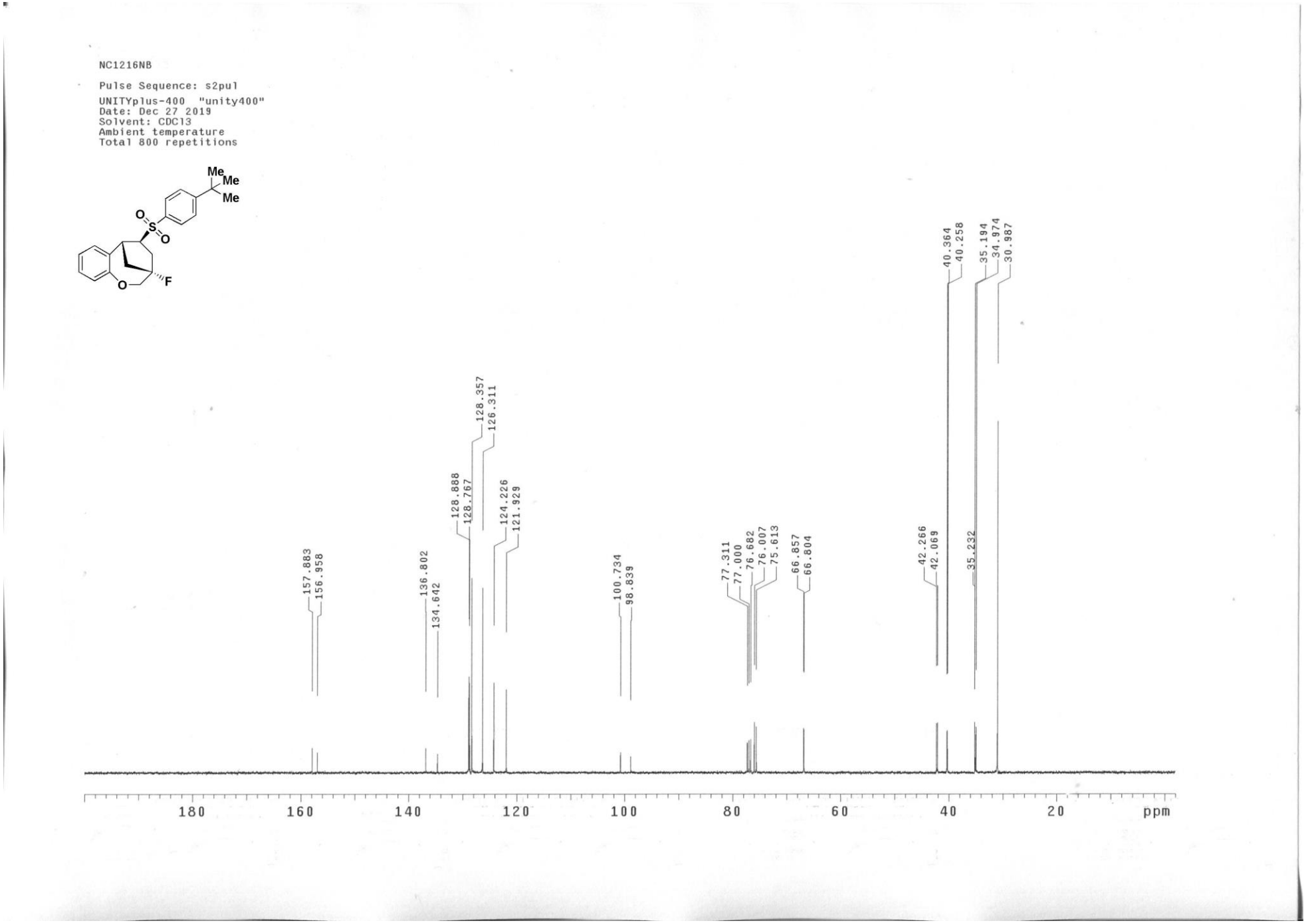




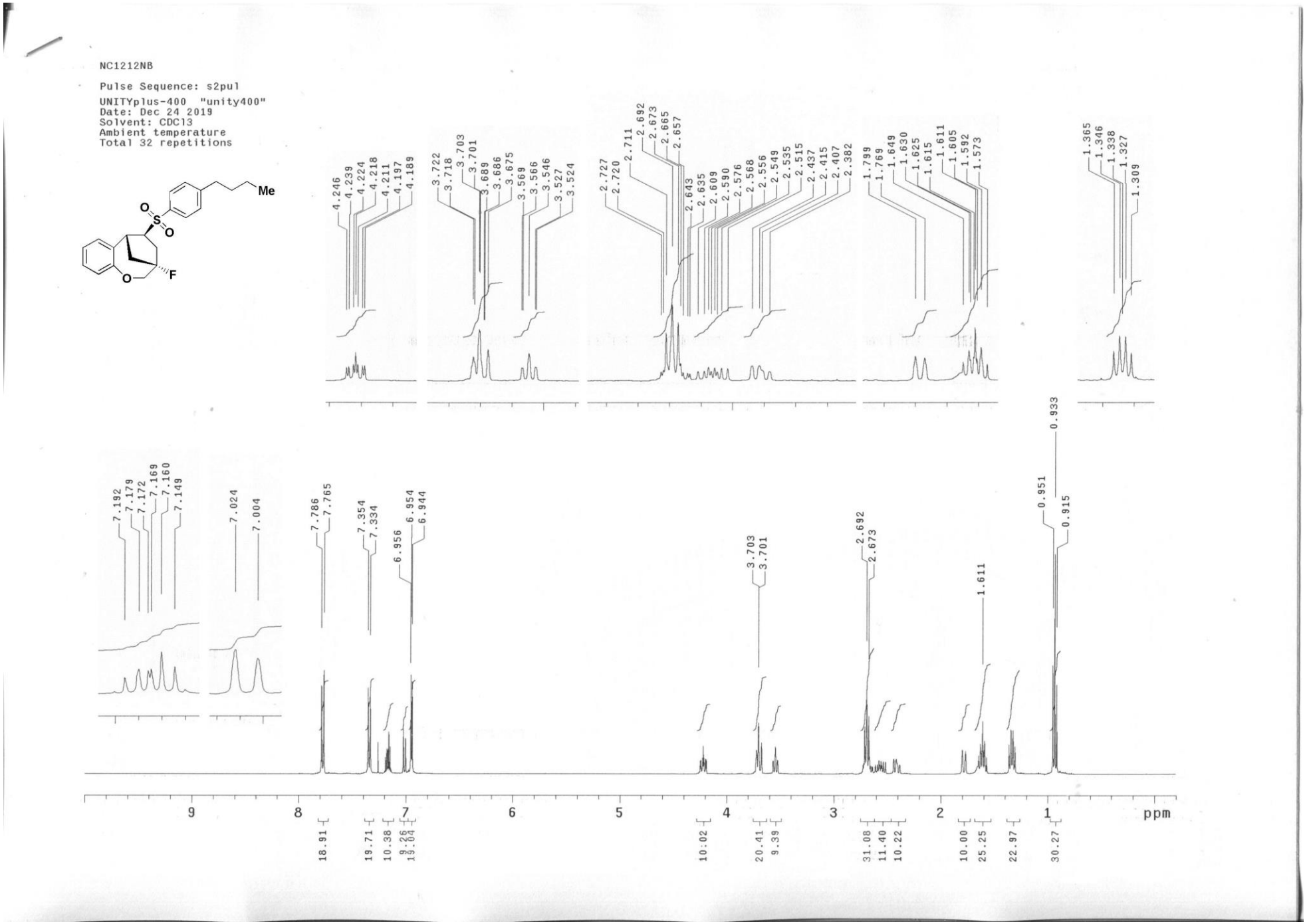
Compound 5j (^{1}H-NMR spectral data)



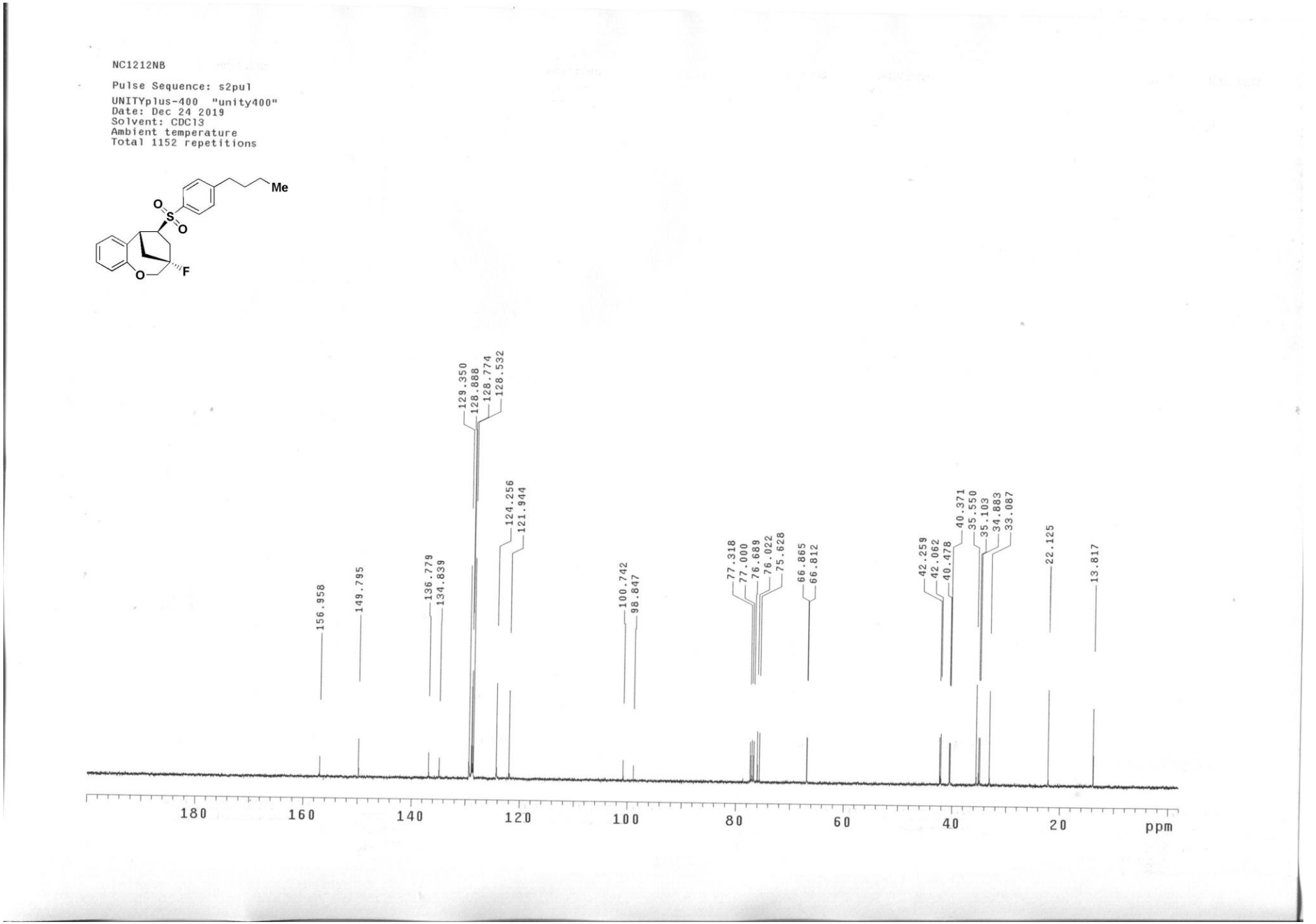
Compound 5j (^{13}C-NMR spectral data)



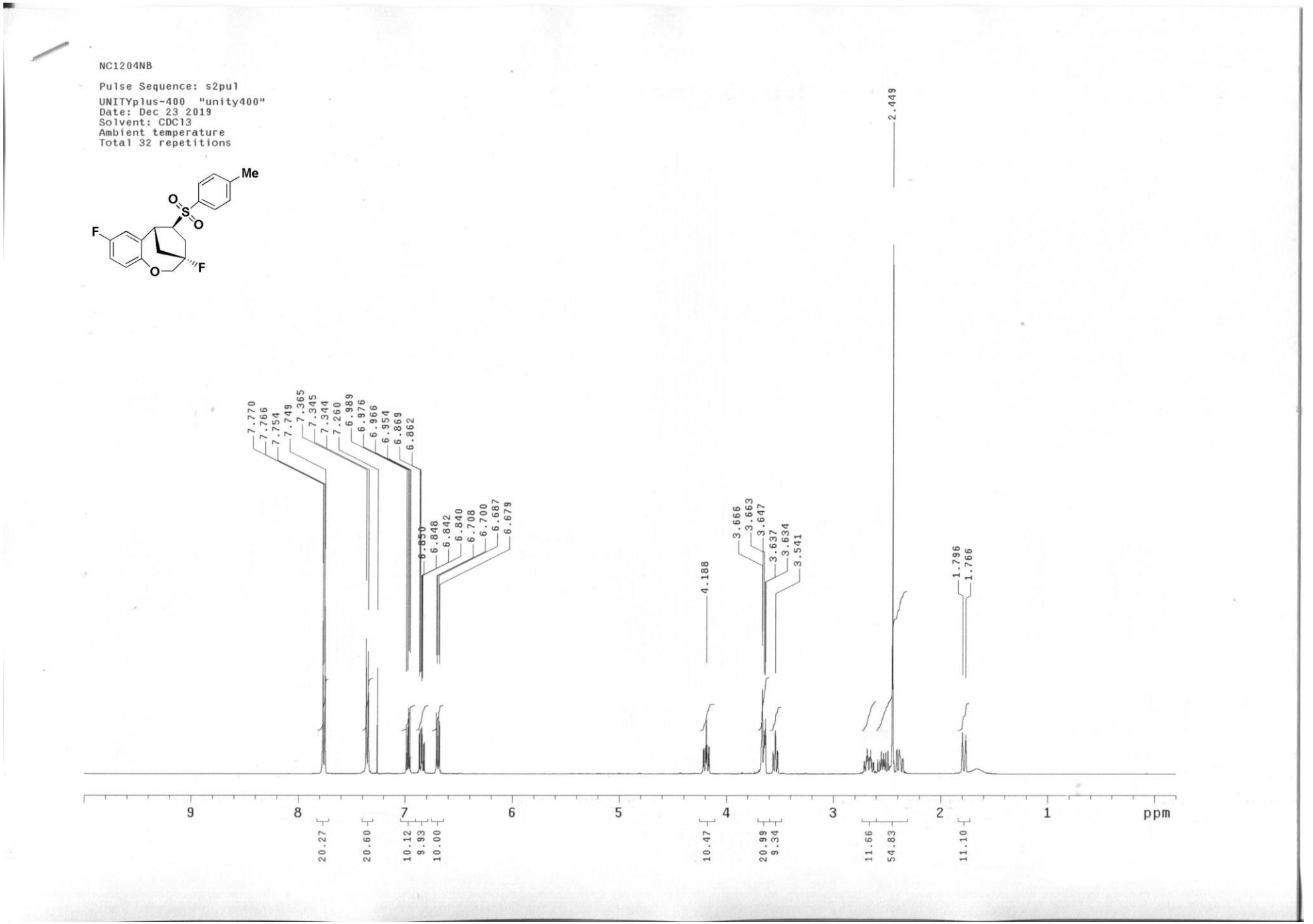
Compound 5k (^{1}H-NMR spectral data)



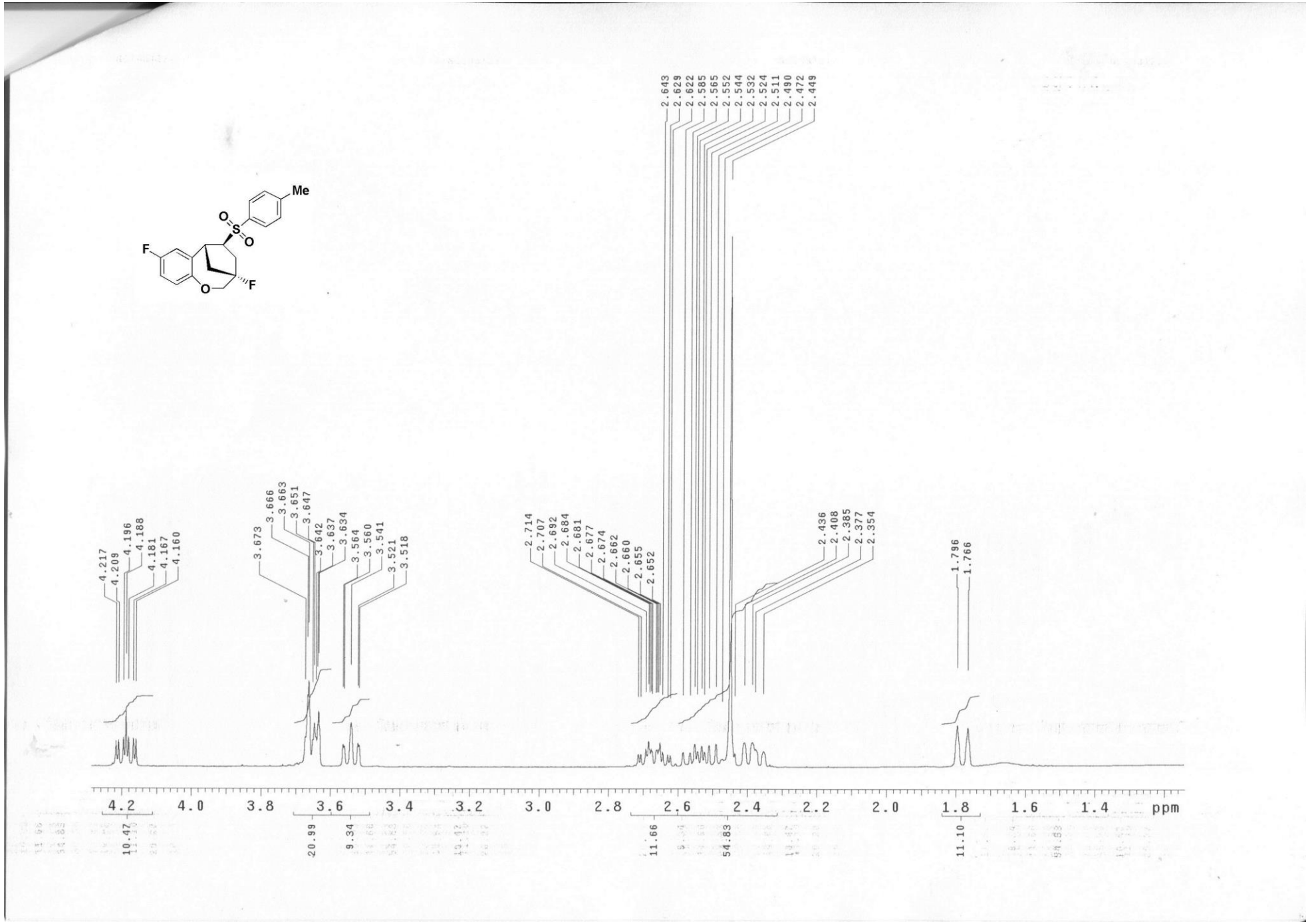
Compound 5k (^{13}C-NMR spectral data)



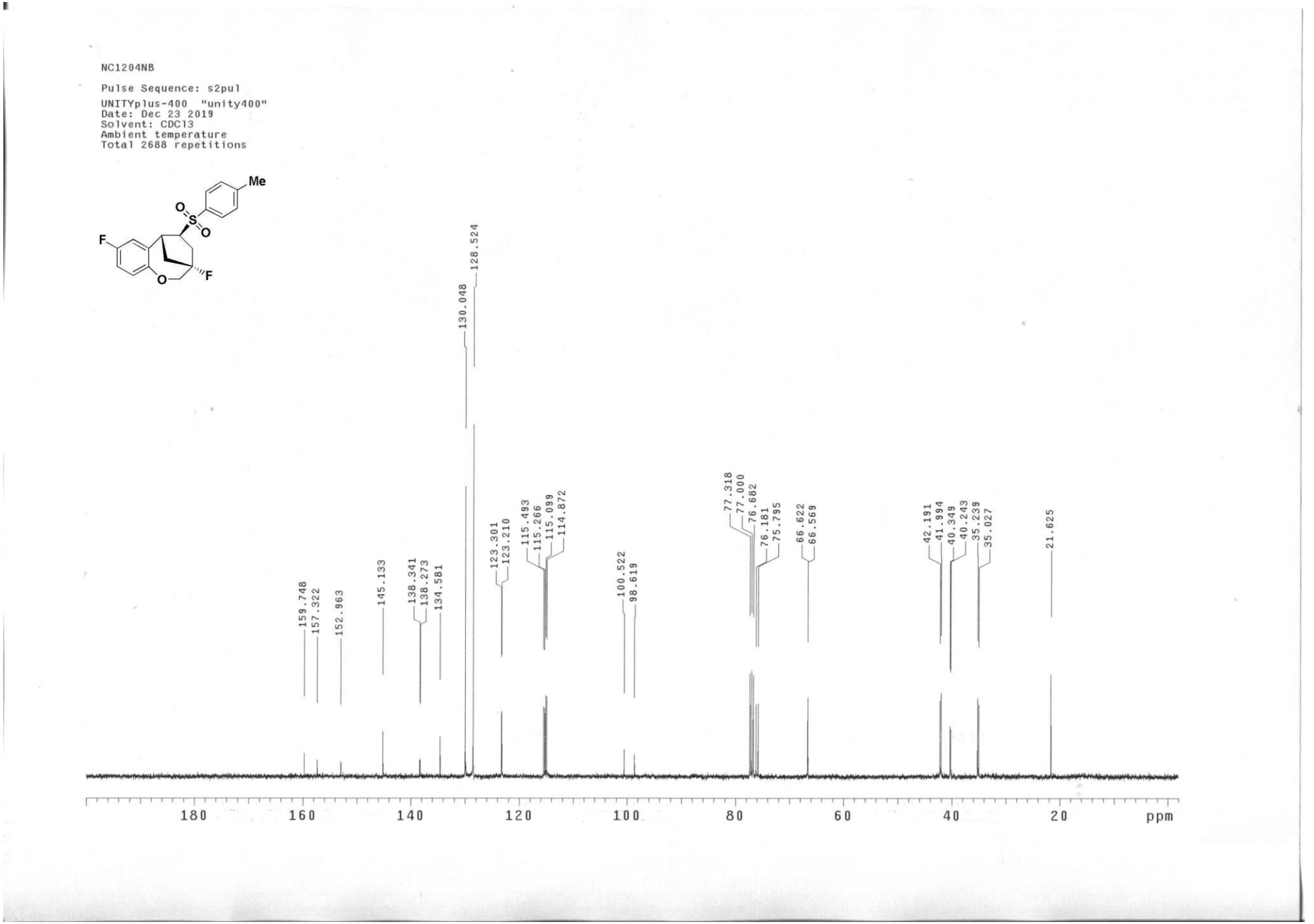
Compound 5l (^{1}H-NMR spectral data-1)



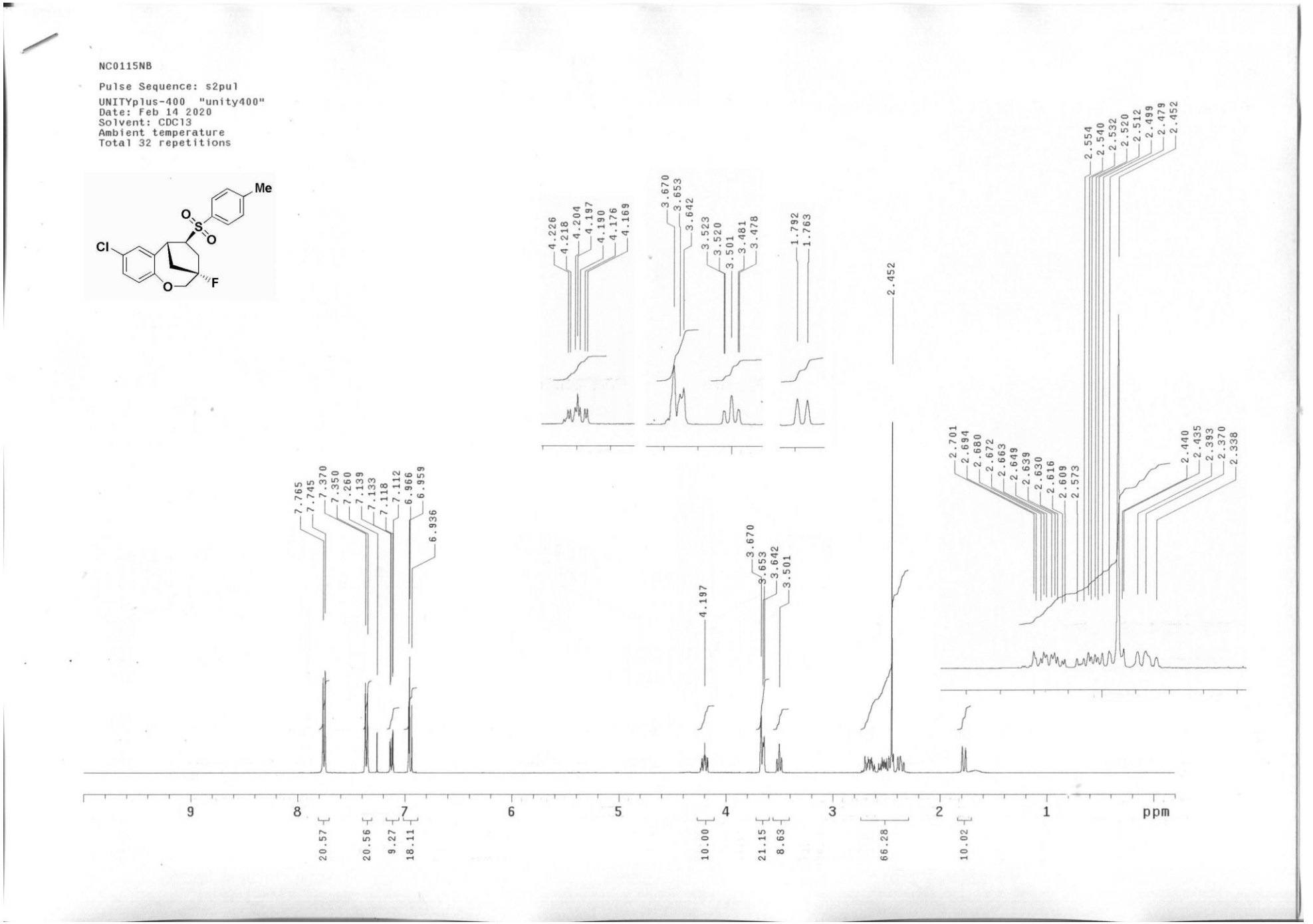
Compound 5l (^{1}H-NMR spectral data-2)



Compound 5l (^{13}C-NMR spectral data)



Compound 5m (^{1}H-NMR spectral data)

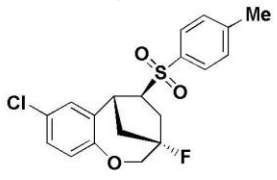


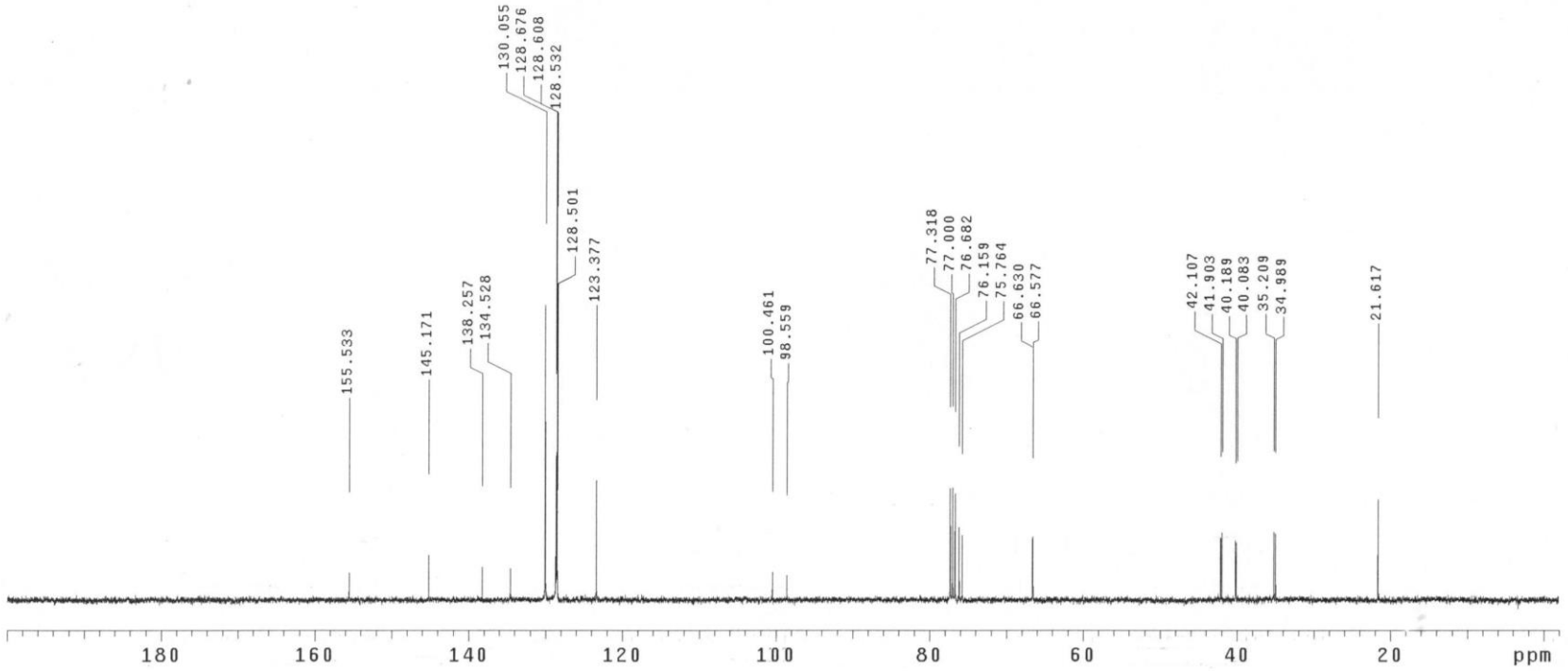
Compound 5m (^{13}C-NMR spectral data)

NC0115NB

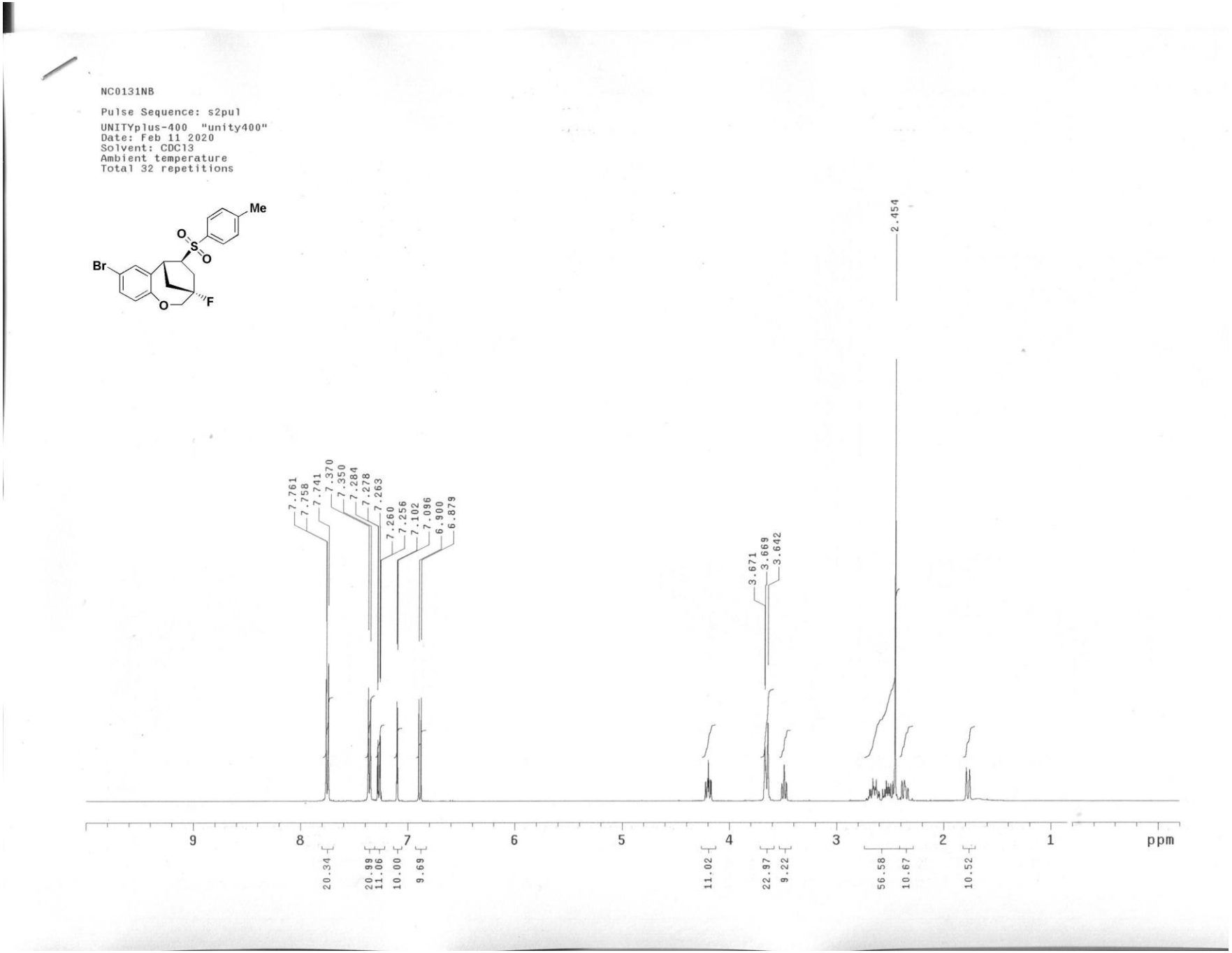
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Feb 14 2020
Solvent: CDCl3
Ambient temperature
Total 1136 repetitions

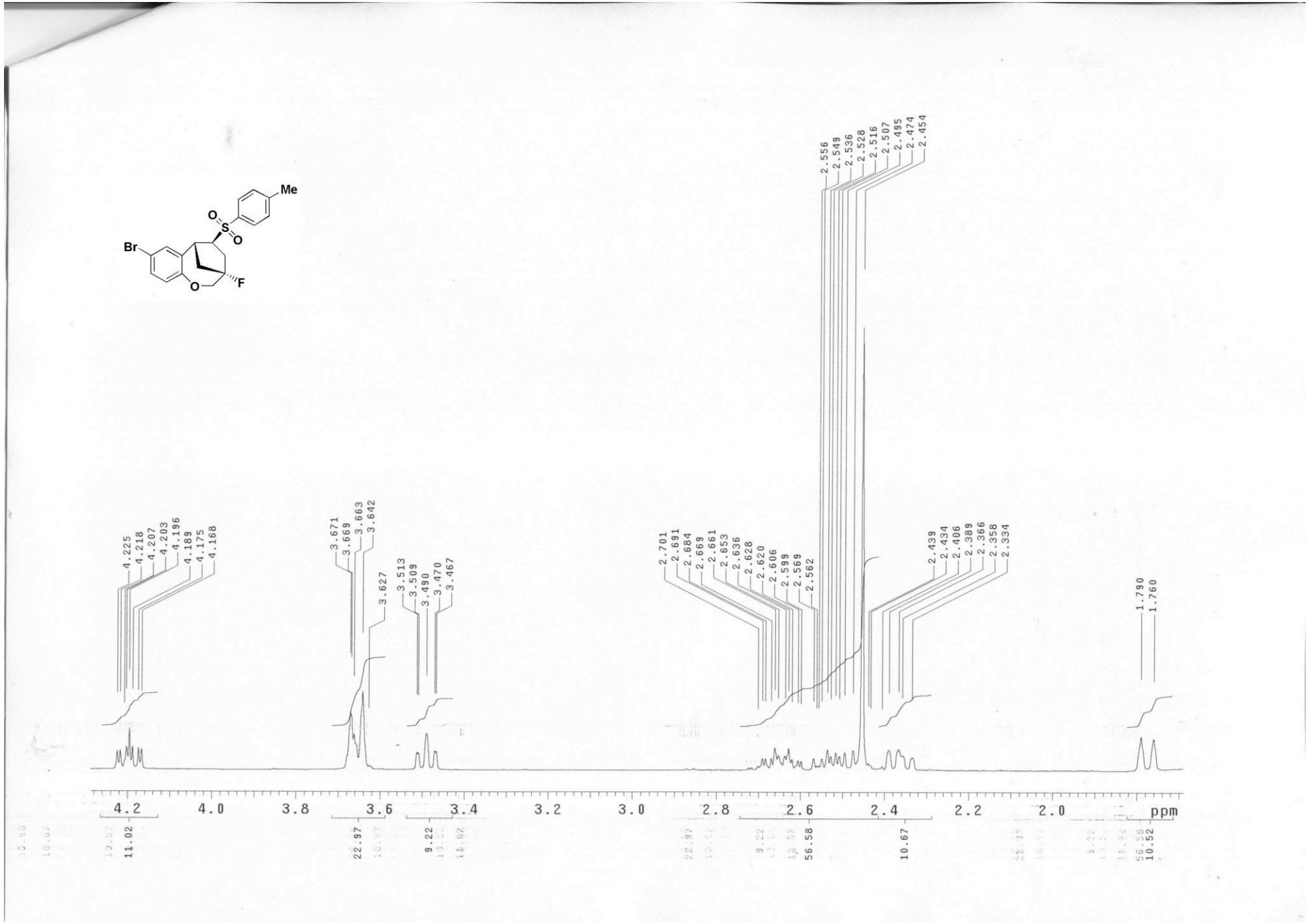




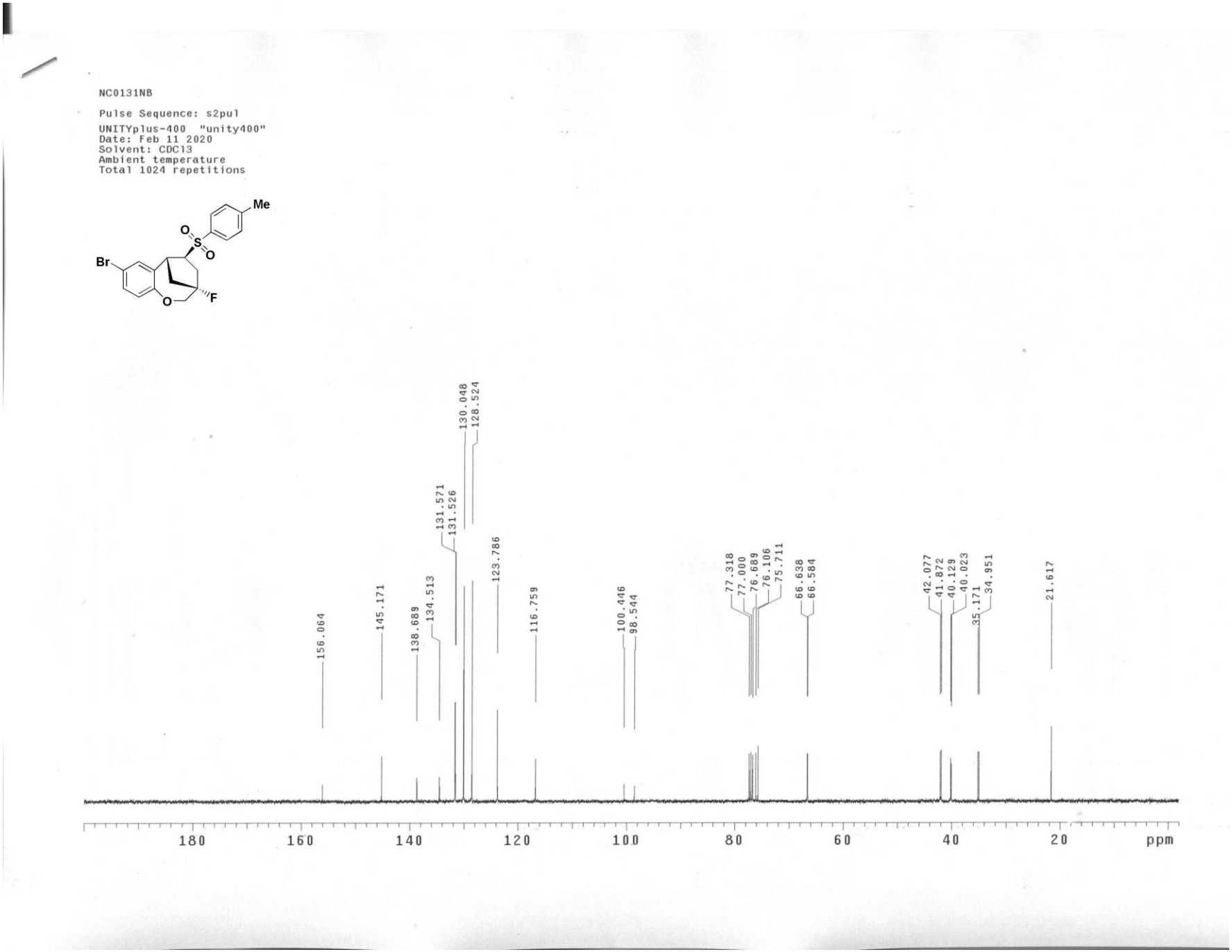
Compound 5n (^{1}H-NMR spectral data-1)



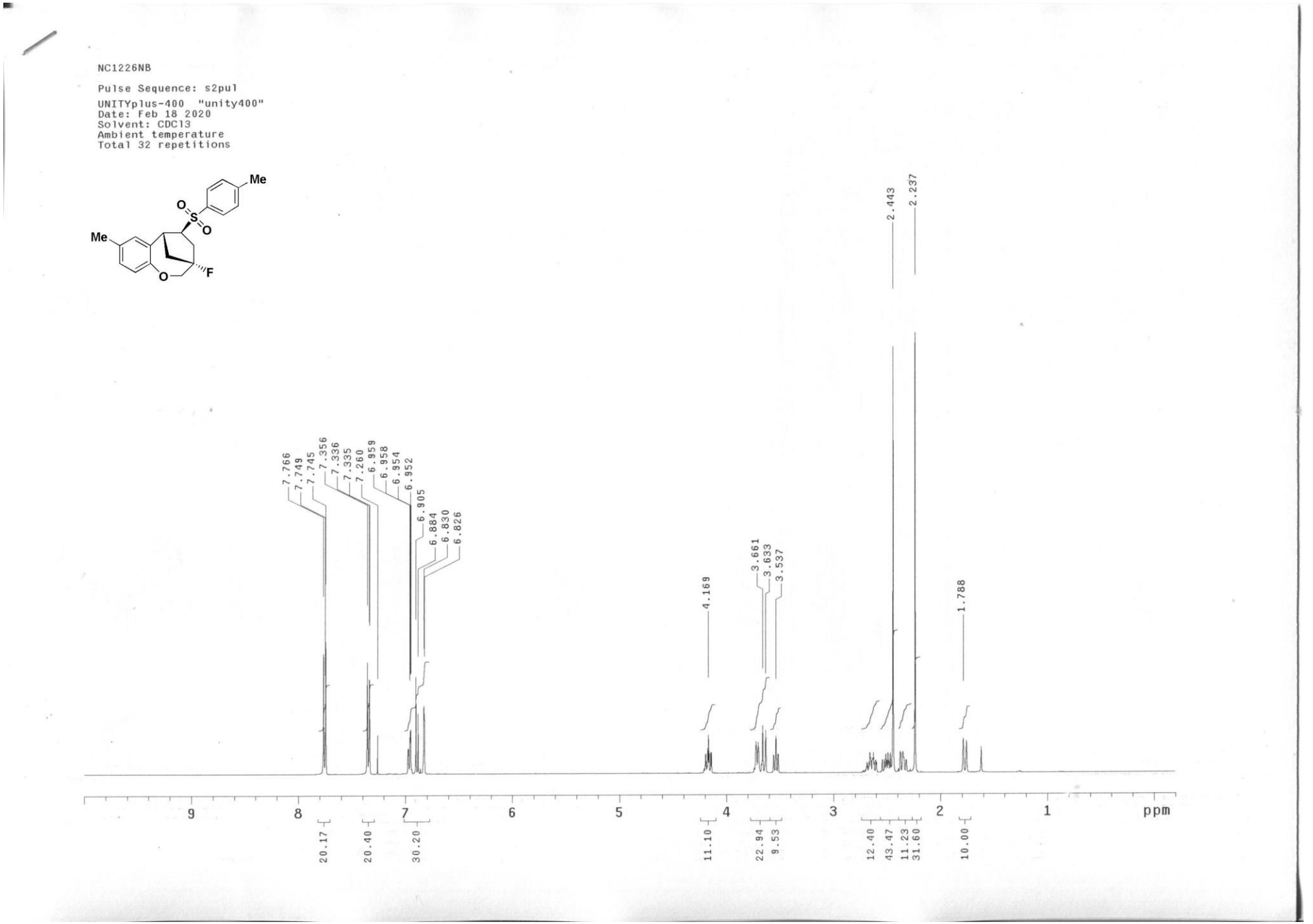
Compound 5n (^{1}H-NMR spectral data-2)



Compound 5n (^{13}C-NMR spectral data)

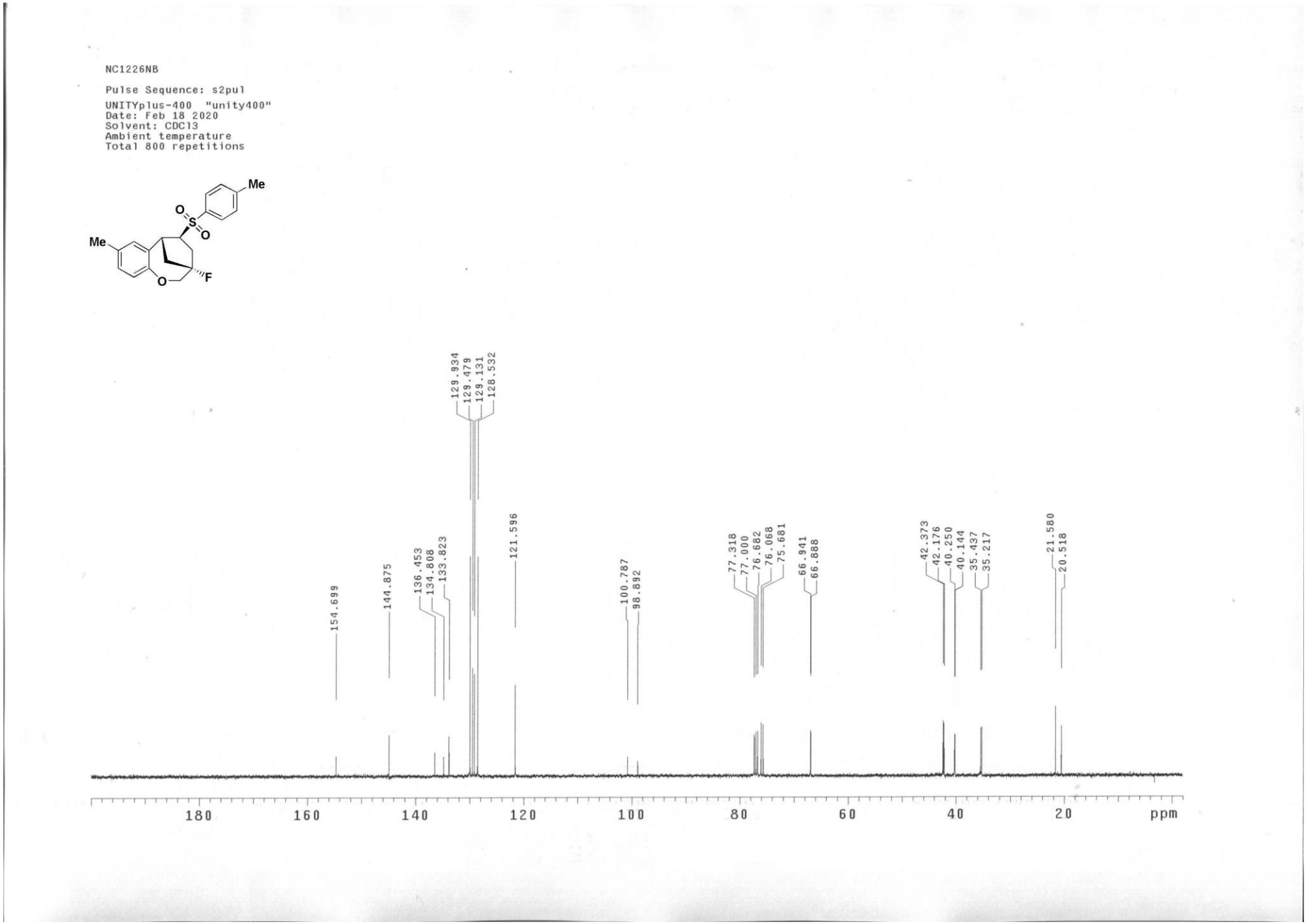


Compound 5o (^{1}H-NMR spectral data-1)

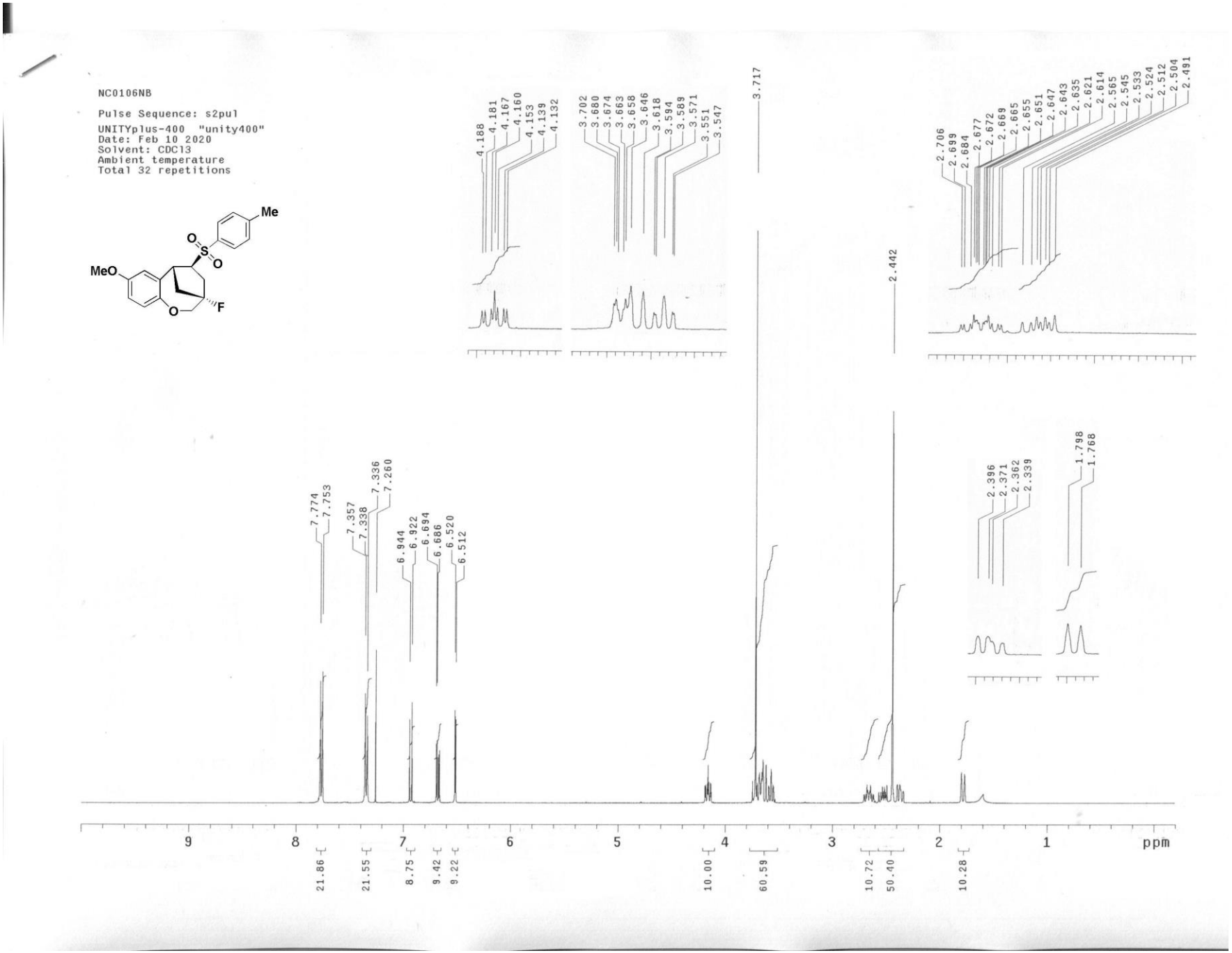


Compound 5o (^{1}H-NMR spectral data-2)

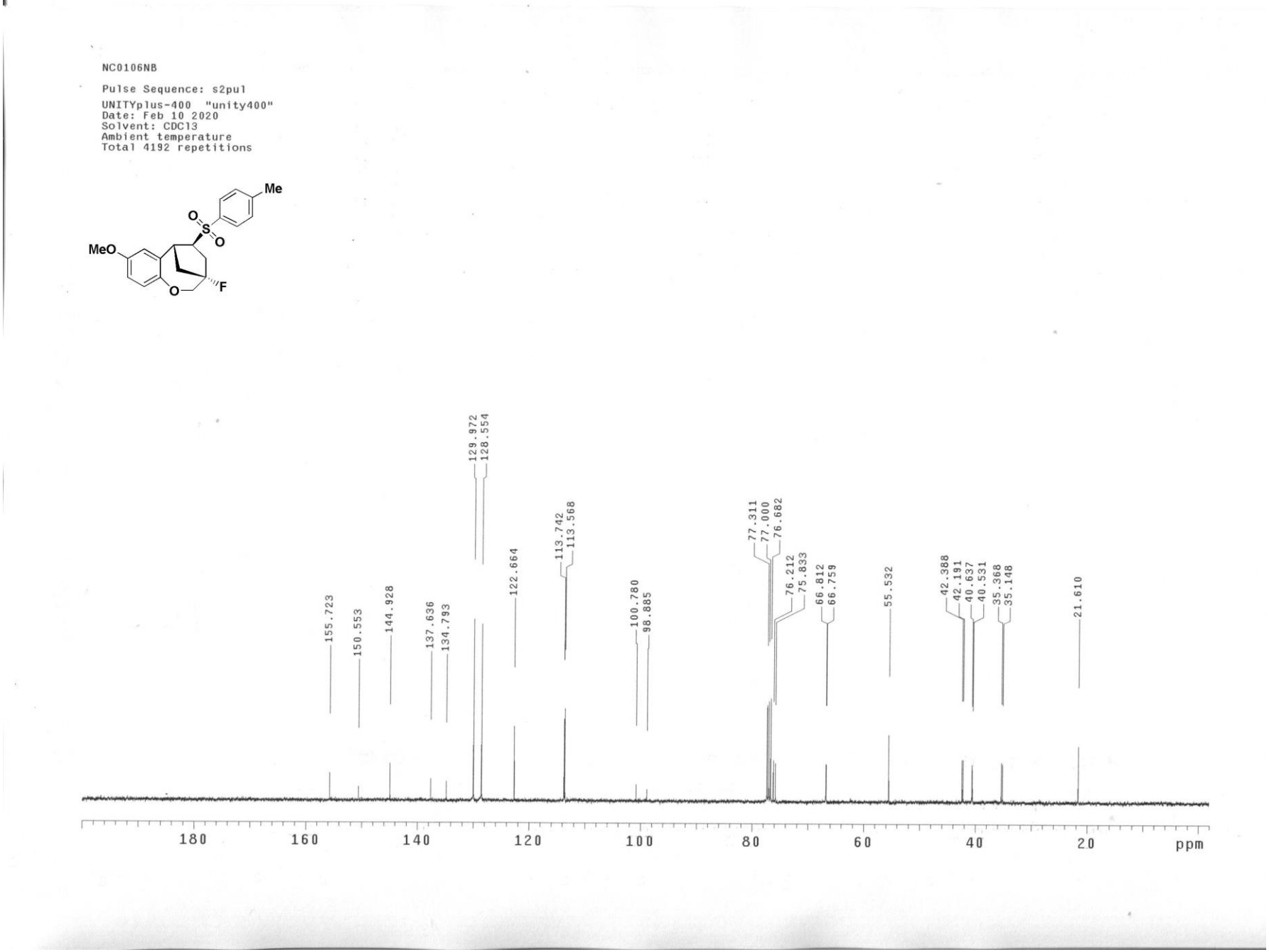
Compound 5o (^{13}C-NMR spectral data)



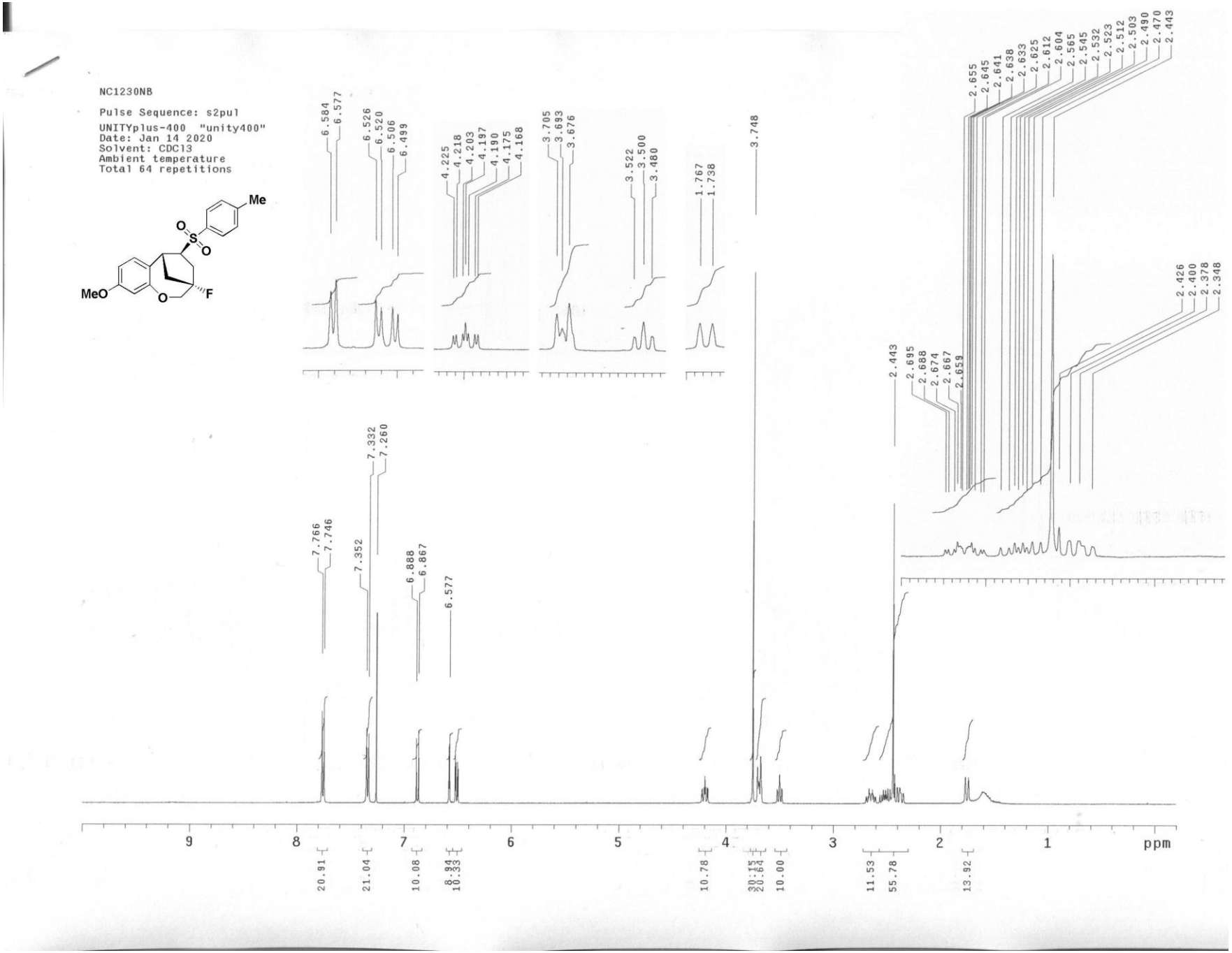
Compound 5p (^{1}H-NMR spectral data)



Compound 5p (^{13}C-NMR spectral data)



Compound 5q (^{1}H-NMR spectral data)

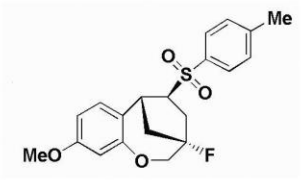


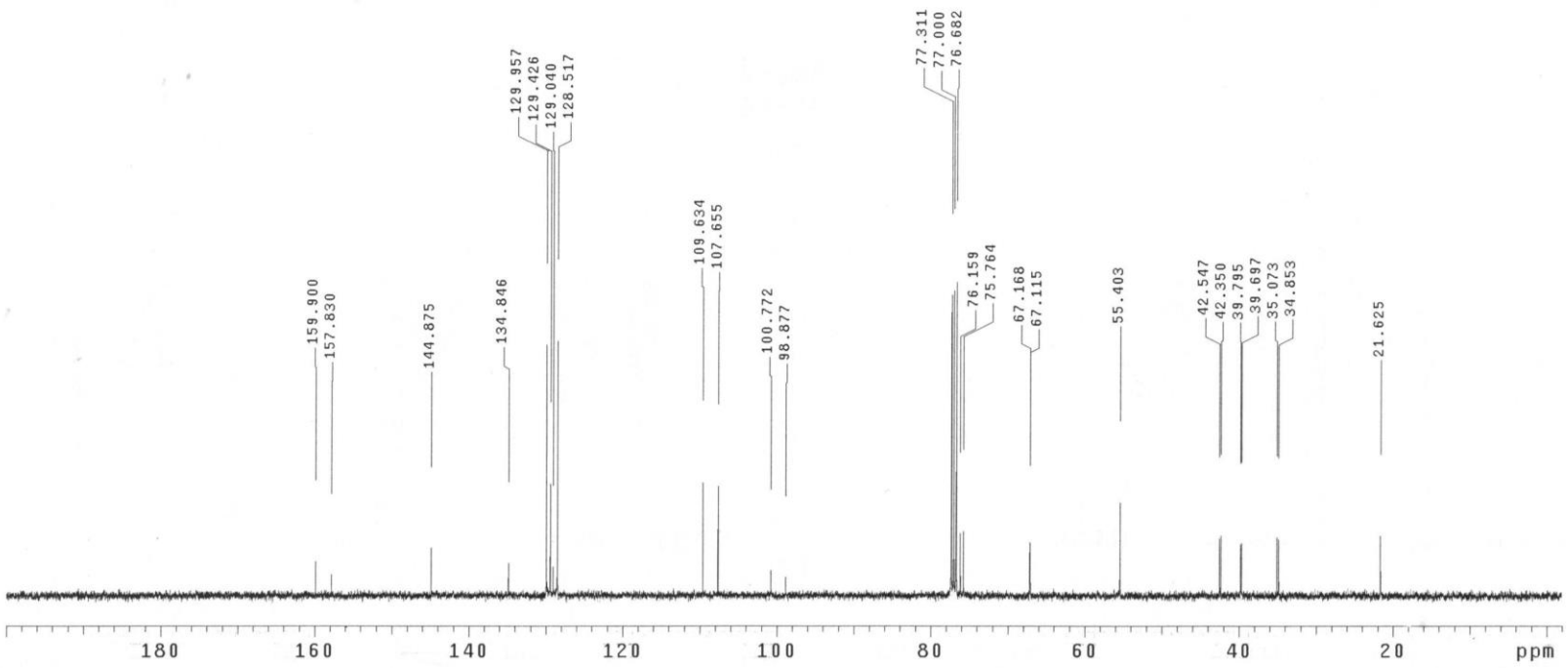
Compound 5q (^{13}C-NMR spectral data)

NC1230NB

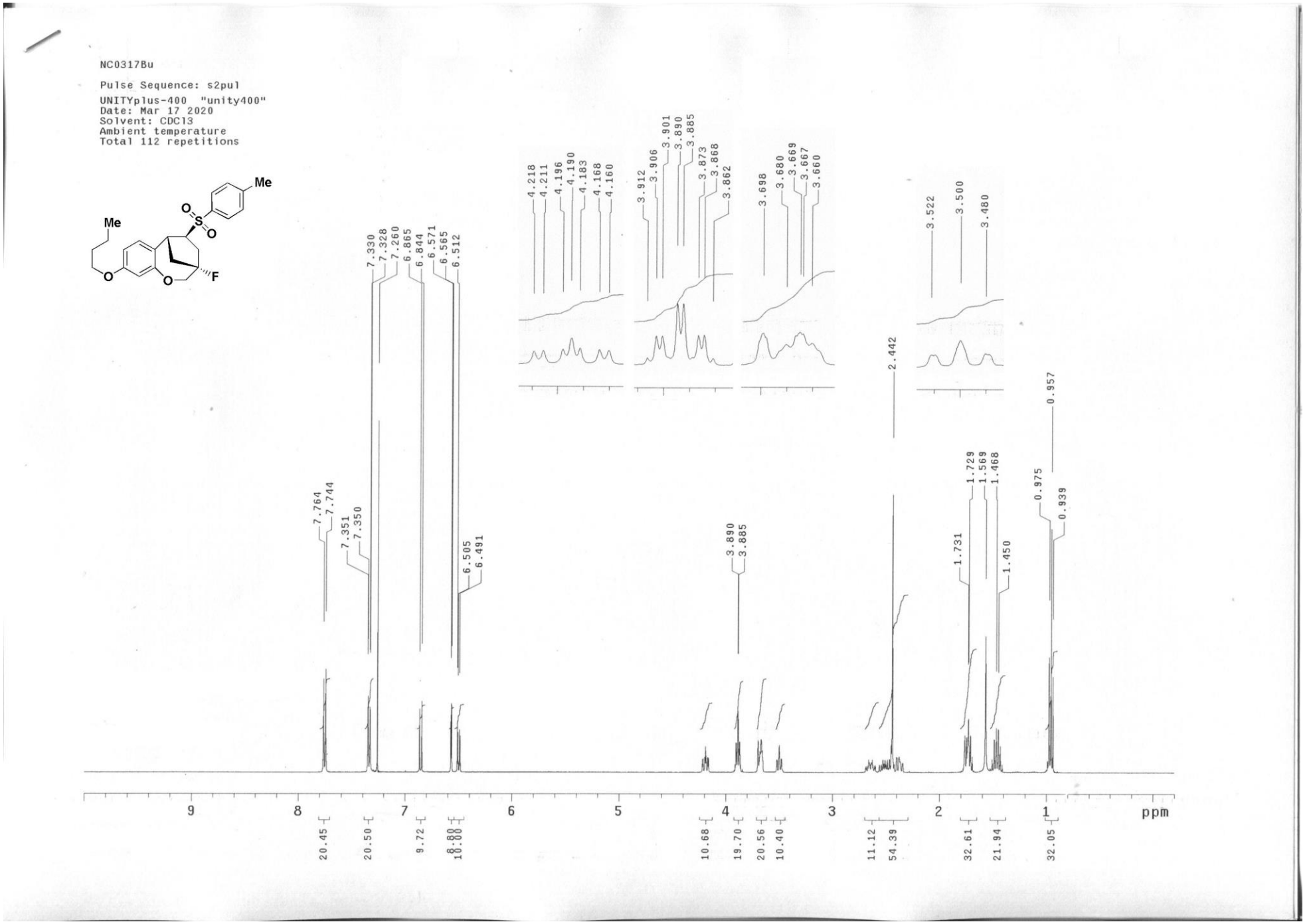
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jan 14 2020
Solvent: CDCl3
Ambient temperature
Total 8304 repetitions

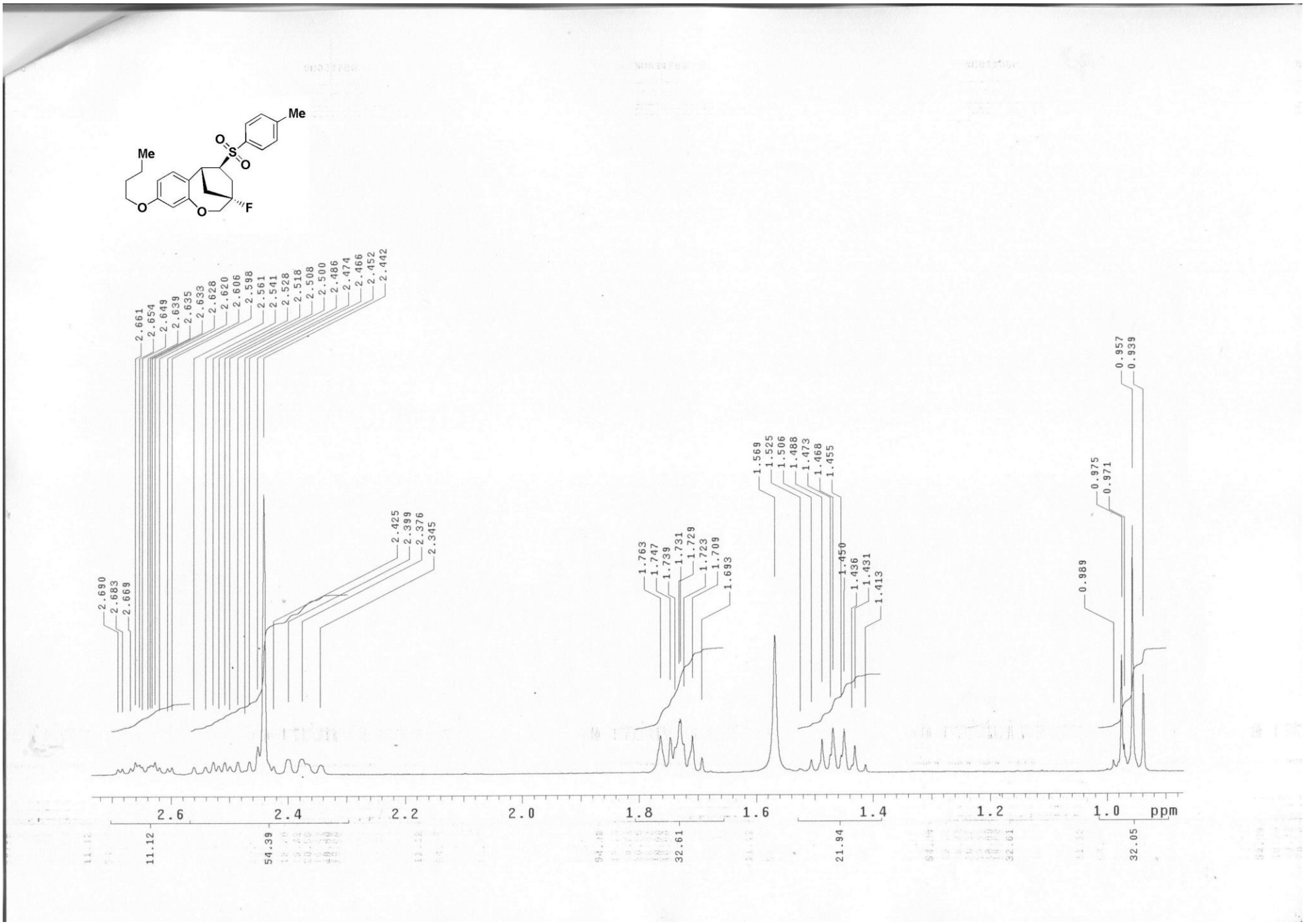




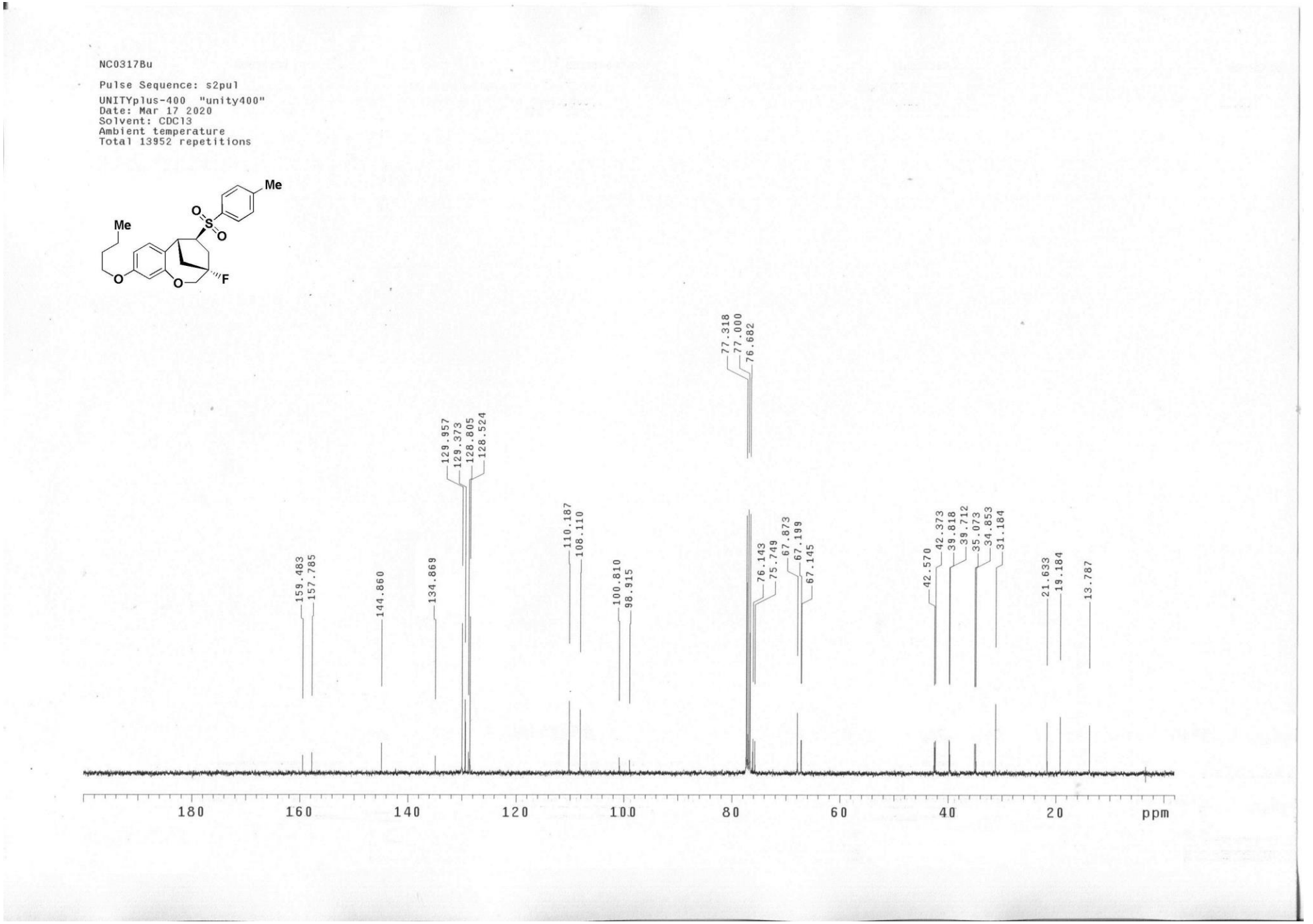
Compound 5r (^{1}H-NMR spectral data-1)



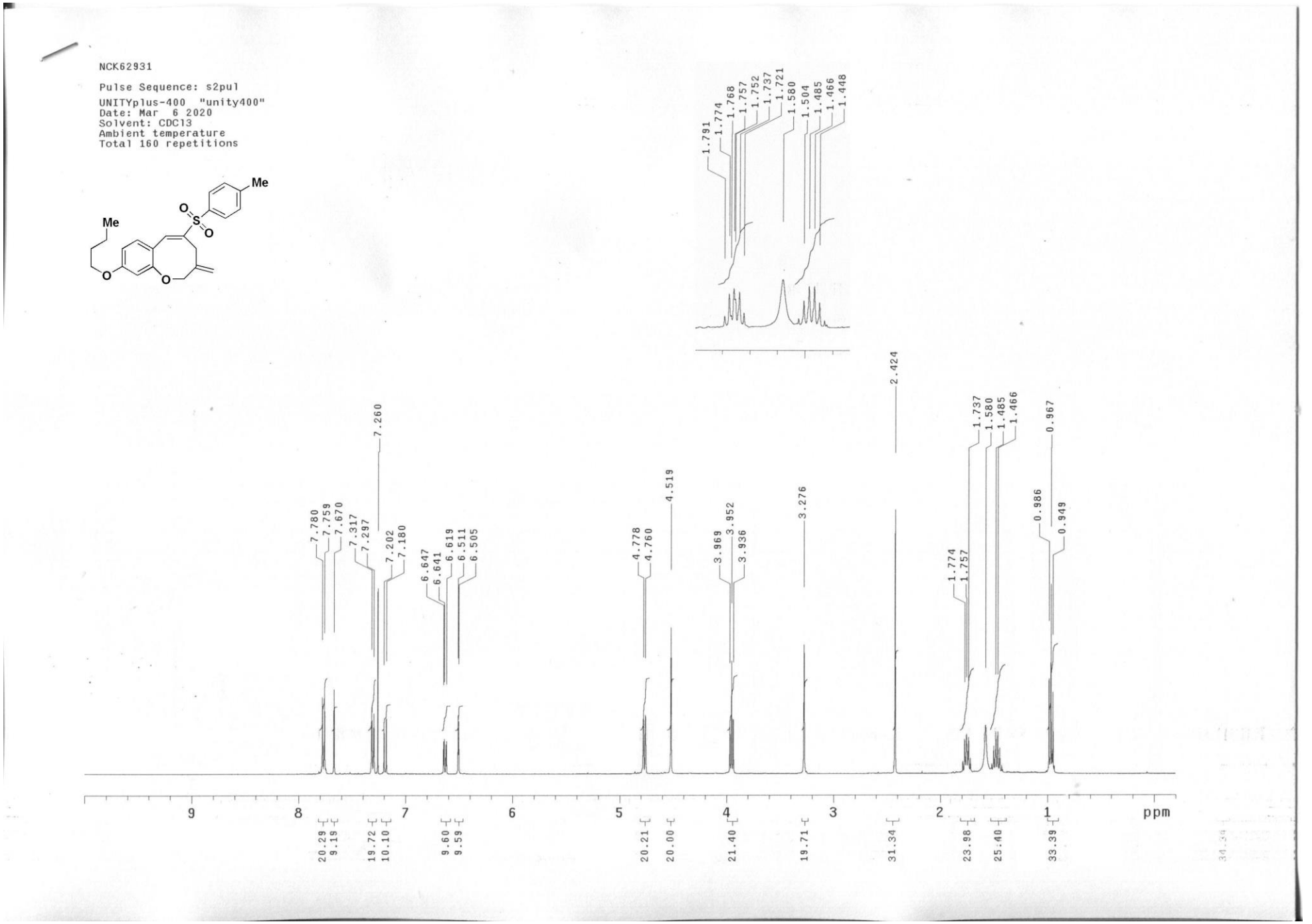
Compound 5r (^{1}H-NMR spectral data-2)



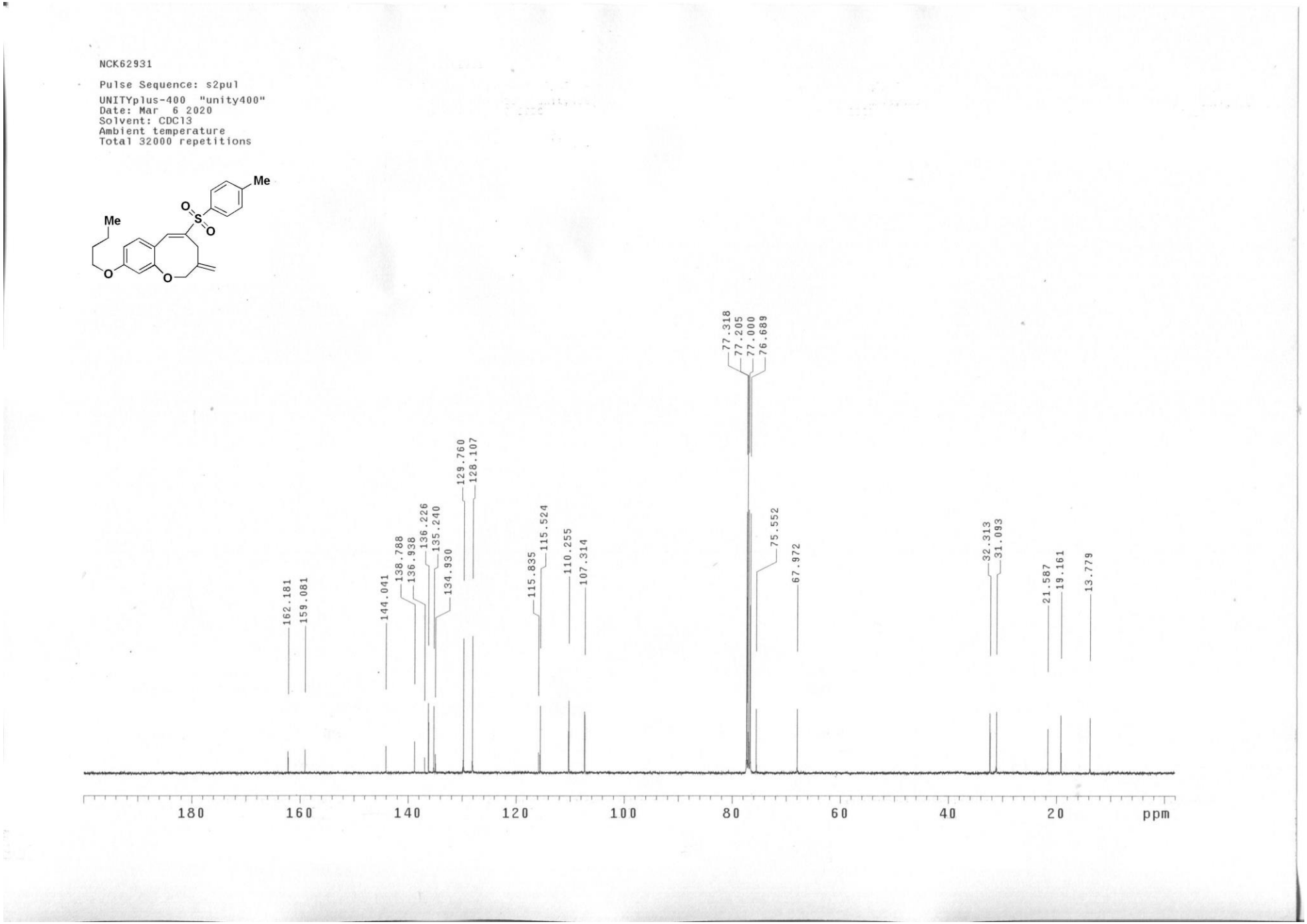
Compound 5r (^{13}C-NMR spectral data)



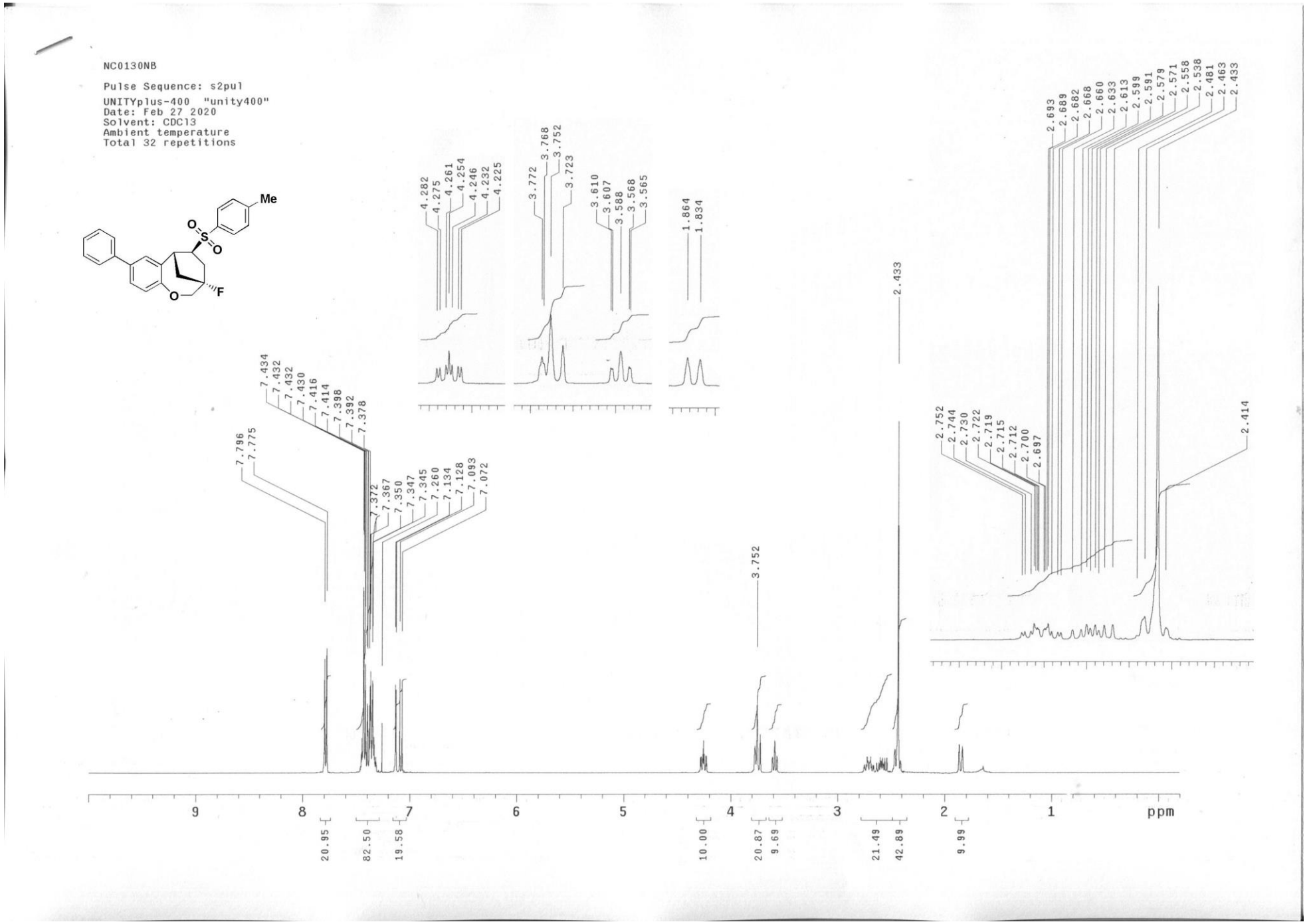
Compound 5r-1 (^{1}H-NMR spectral data)



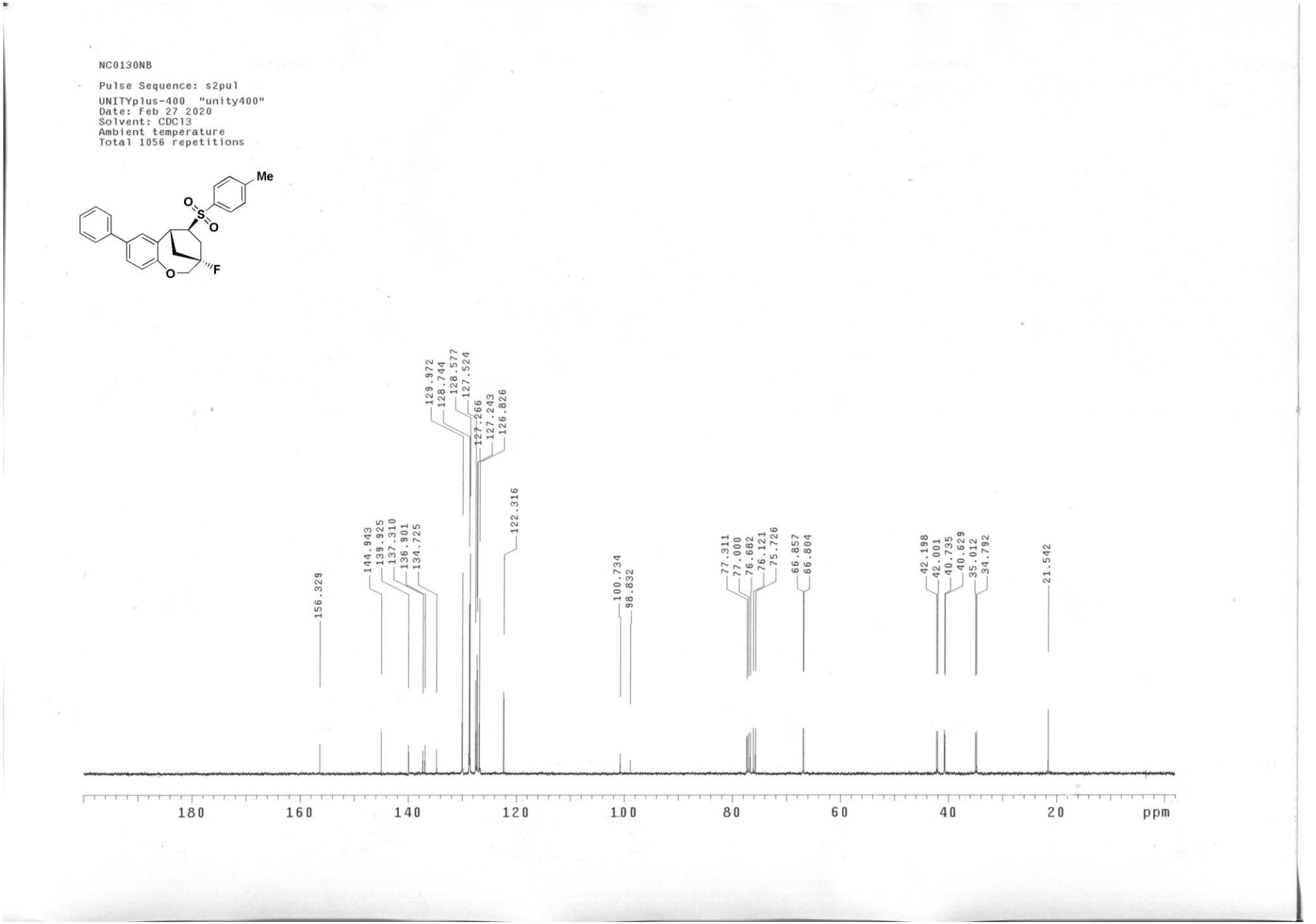
Compound 5r-1 (^{13}C-NMR spectral data)



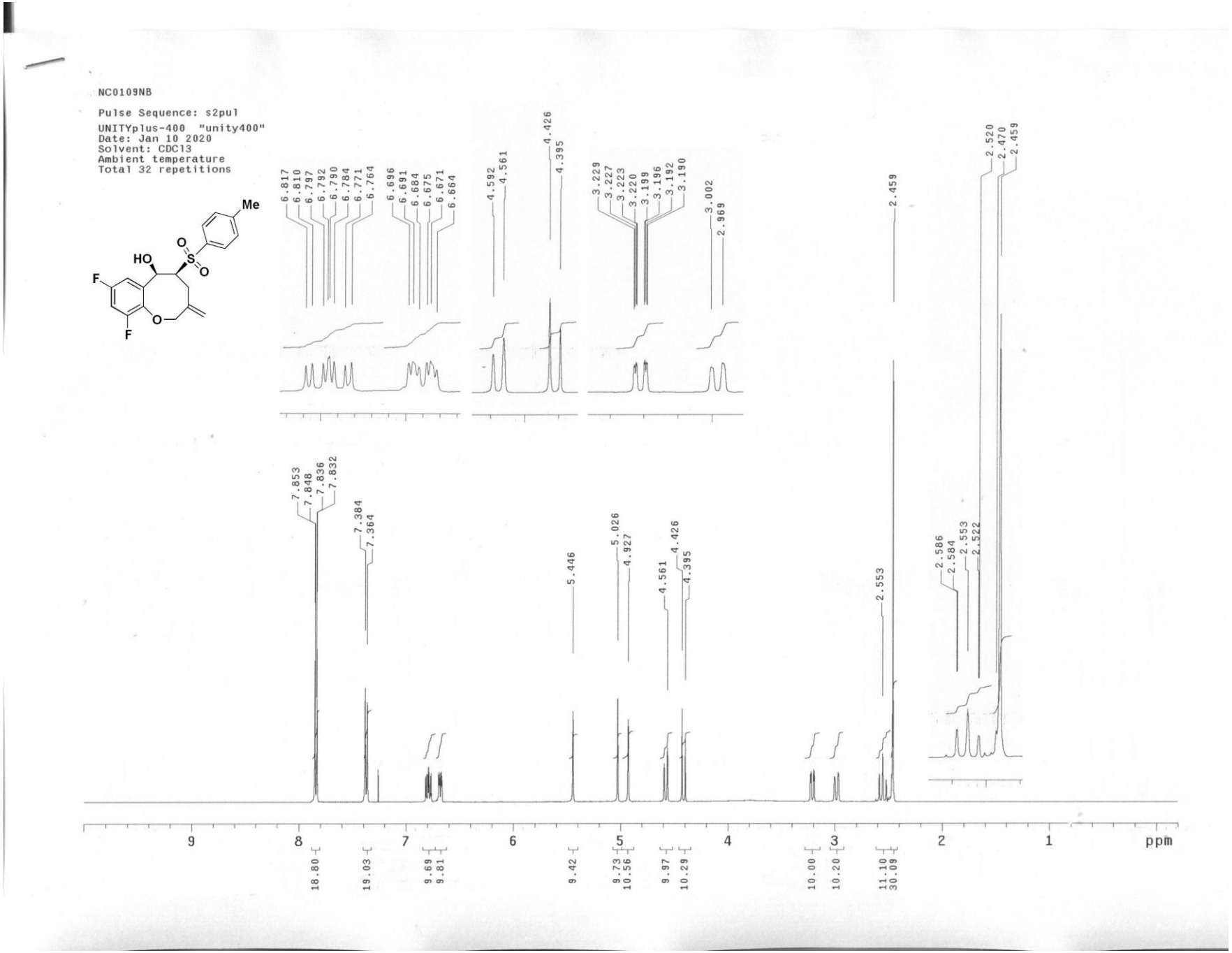
Compound 5s (^{1}H-NMR spectral data)



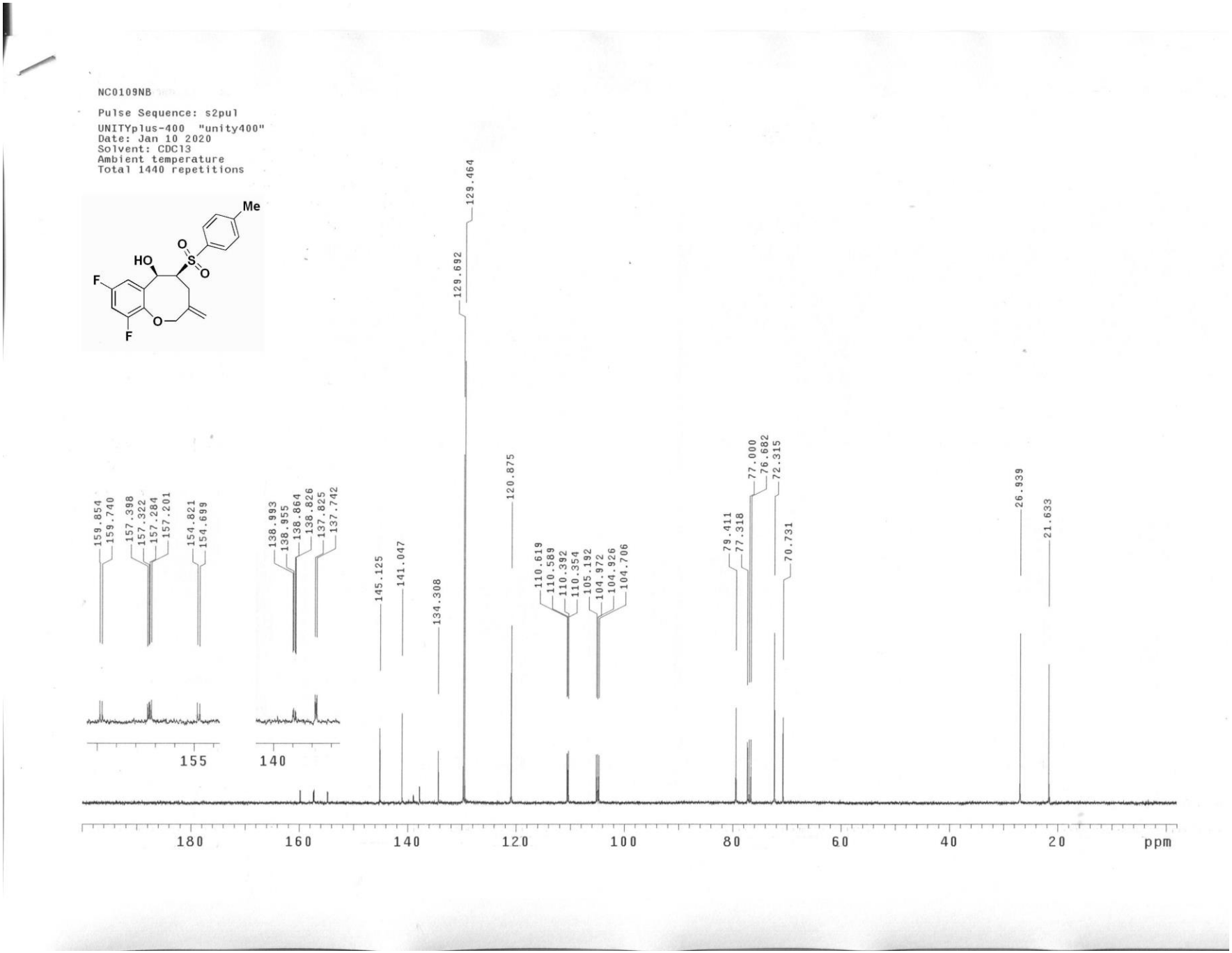
Compound 5s (^{13}C-NMR spectral data)



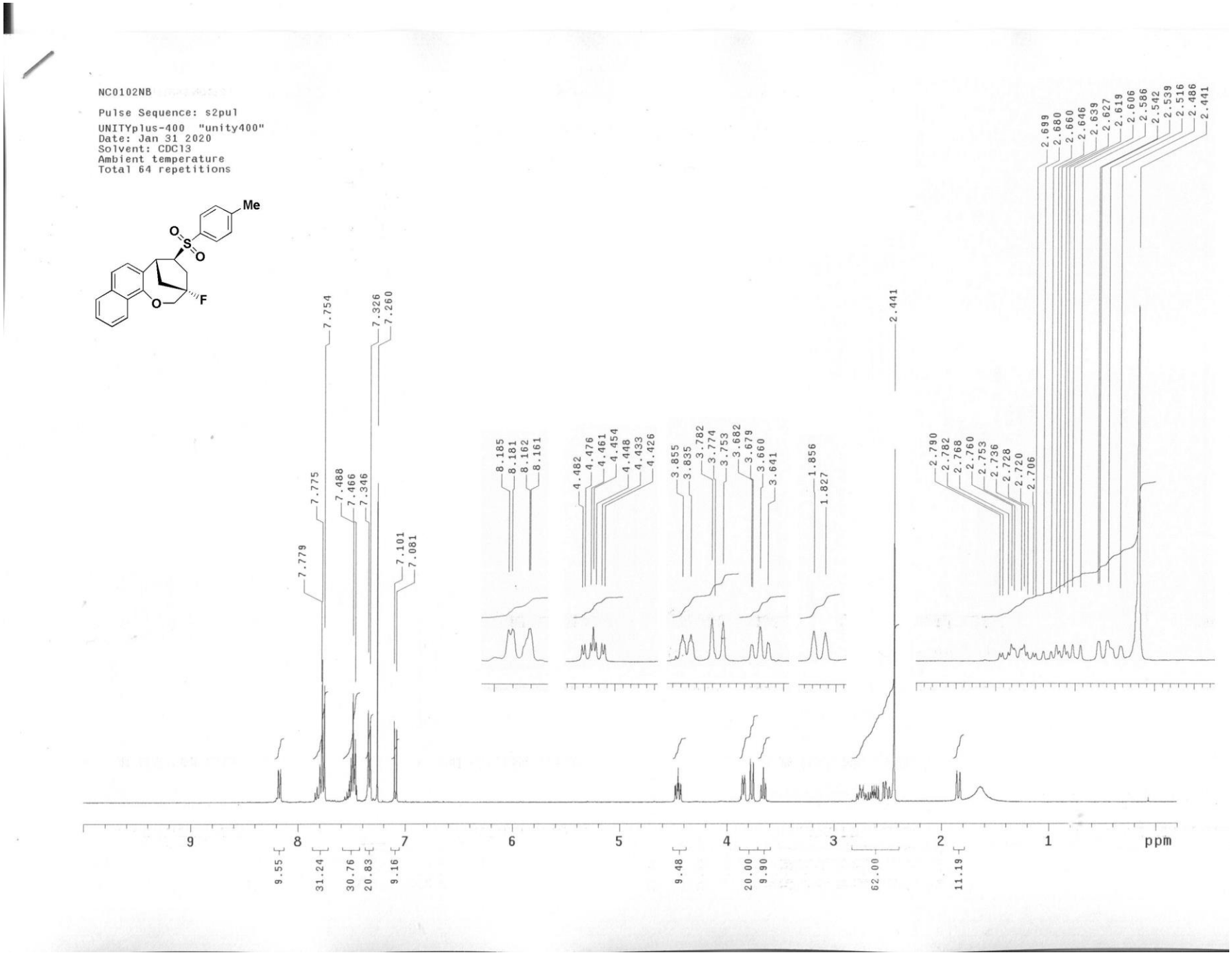
Compound 6t (^{1}H-NMR spectral data)



Compound 6t (^{13}C-NMR spectral data)



Compound 5u (^{1}H-NMR spectral data)

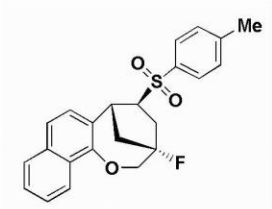


Compound 5u (^{13}C-NMR spectral data)

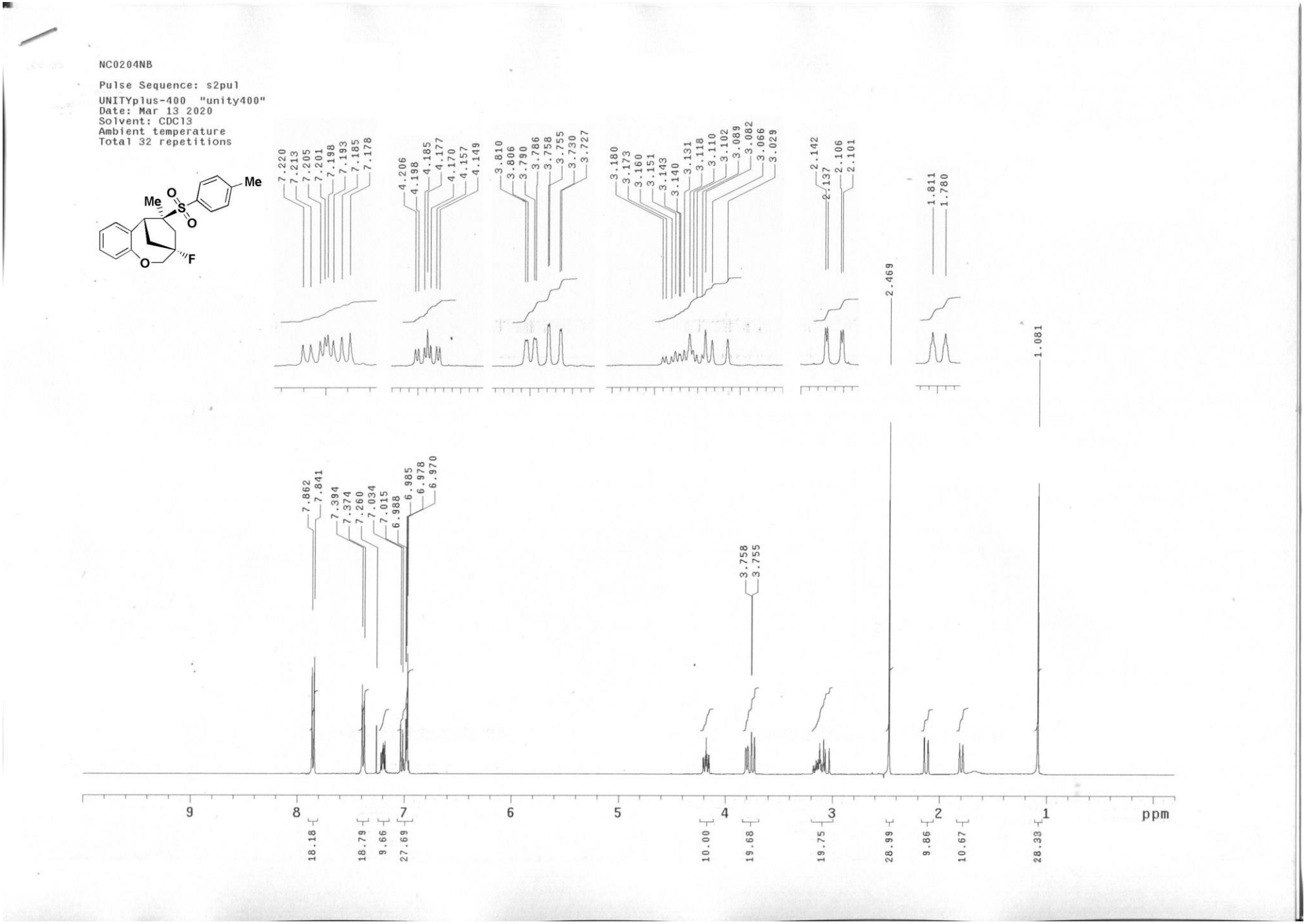
NC0102NB

Pulse Sequence: s2pul

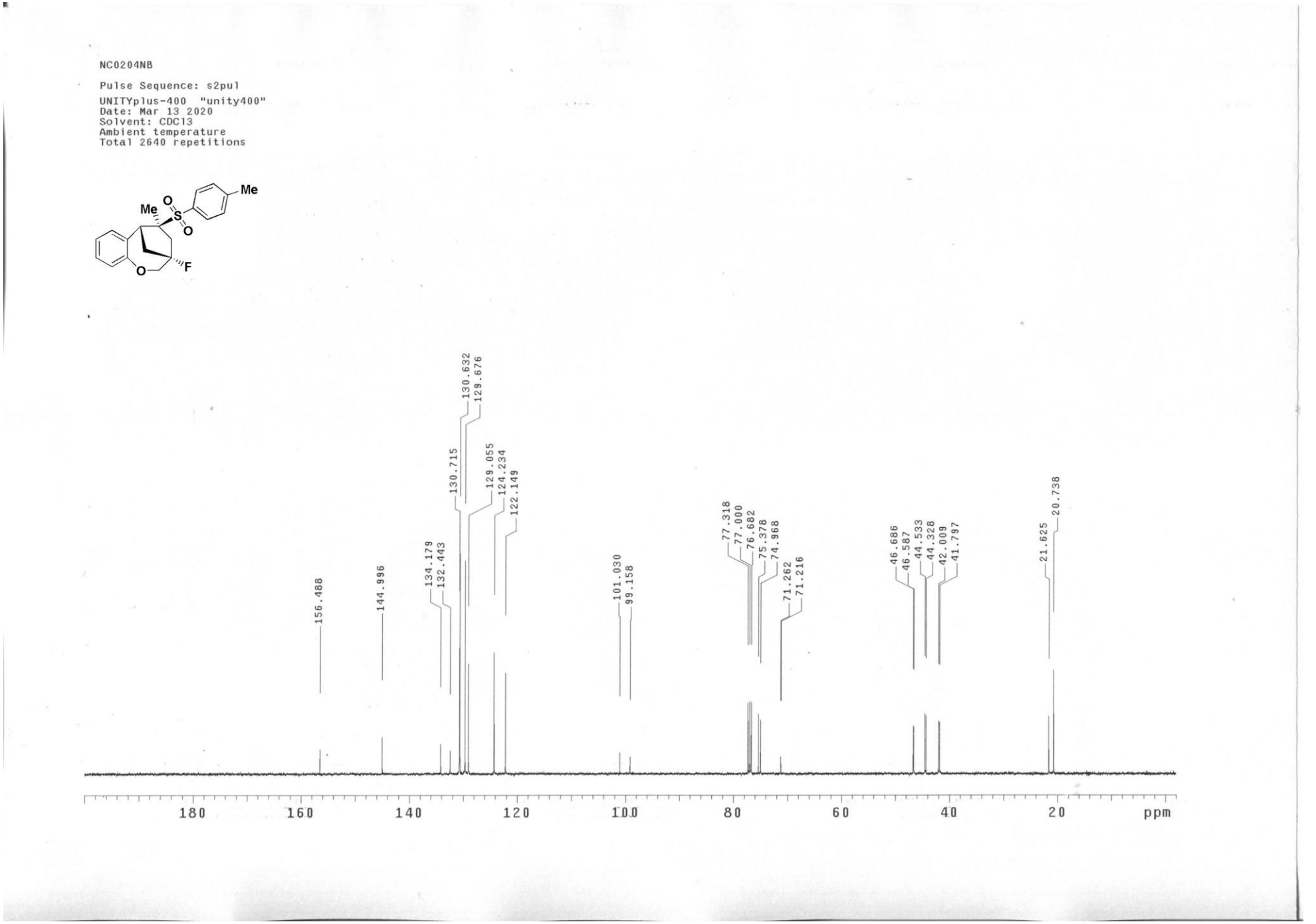
UNITYplus-400 "unity400"
Date: Jan 31 2020
Solvent: CDCl3
Ambient temperature
Total 6720 repetitions



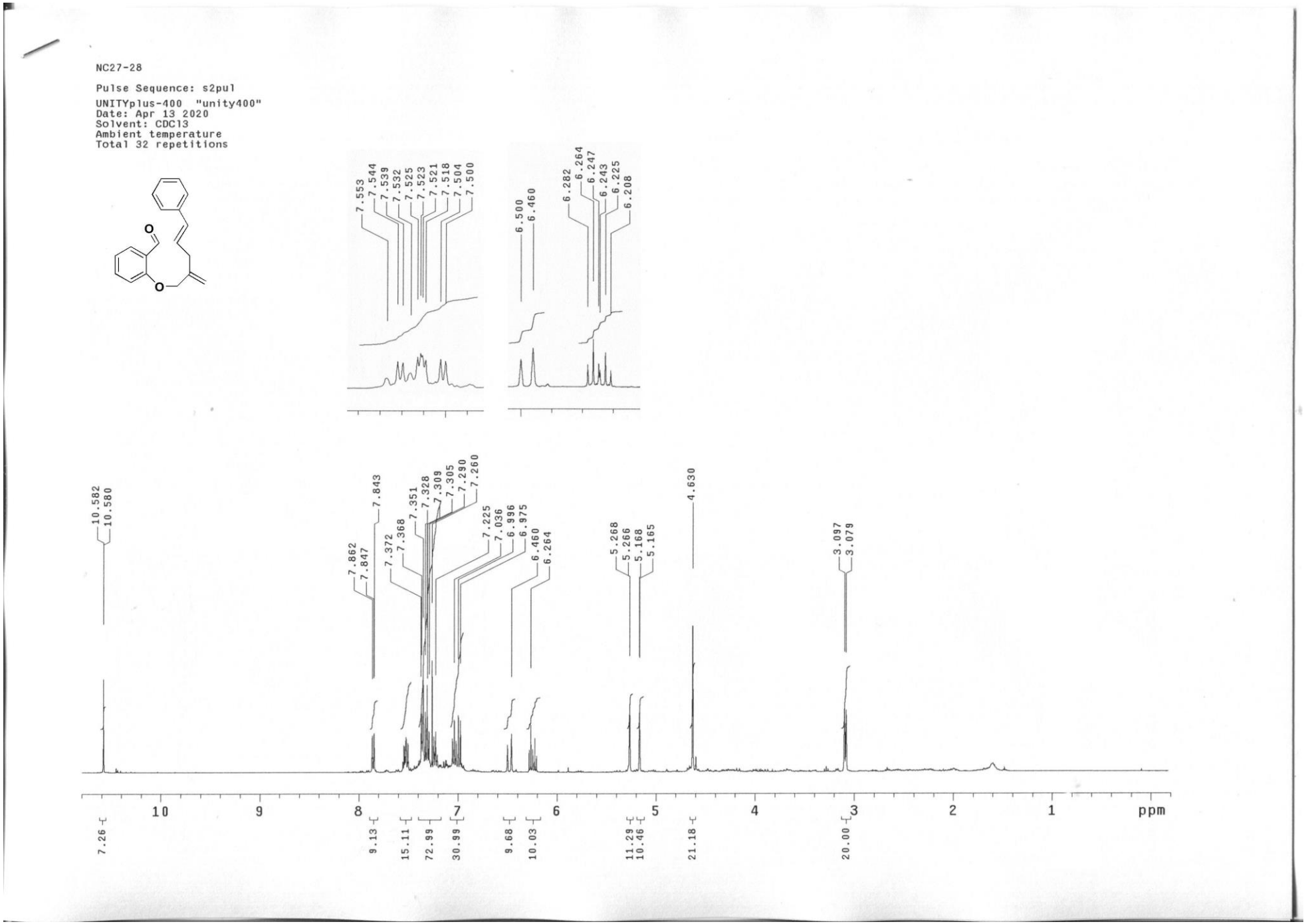
Compound 5v (^{1}H-NMR spectral data)



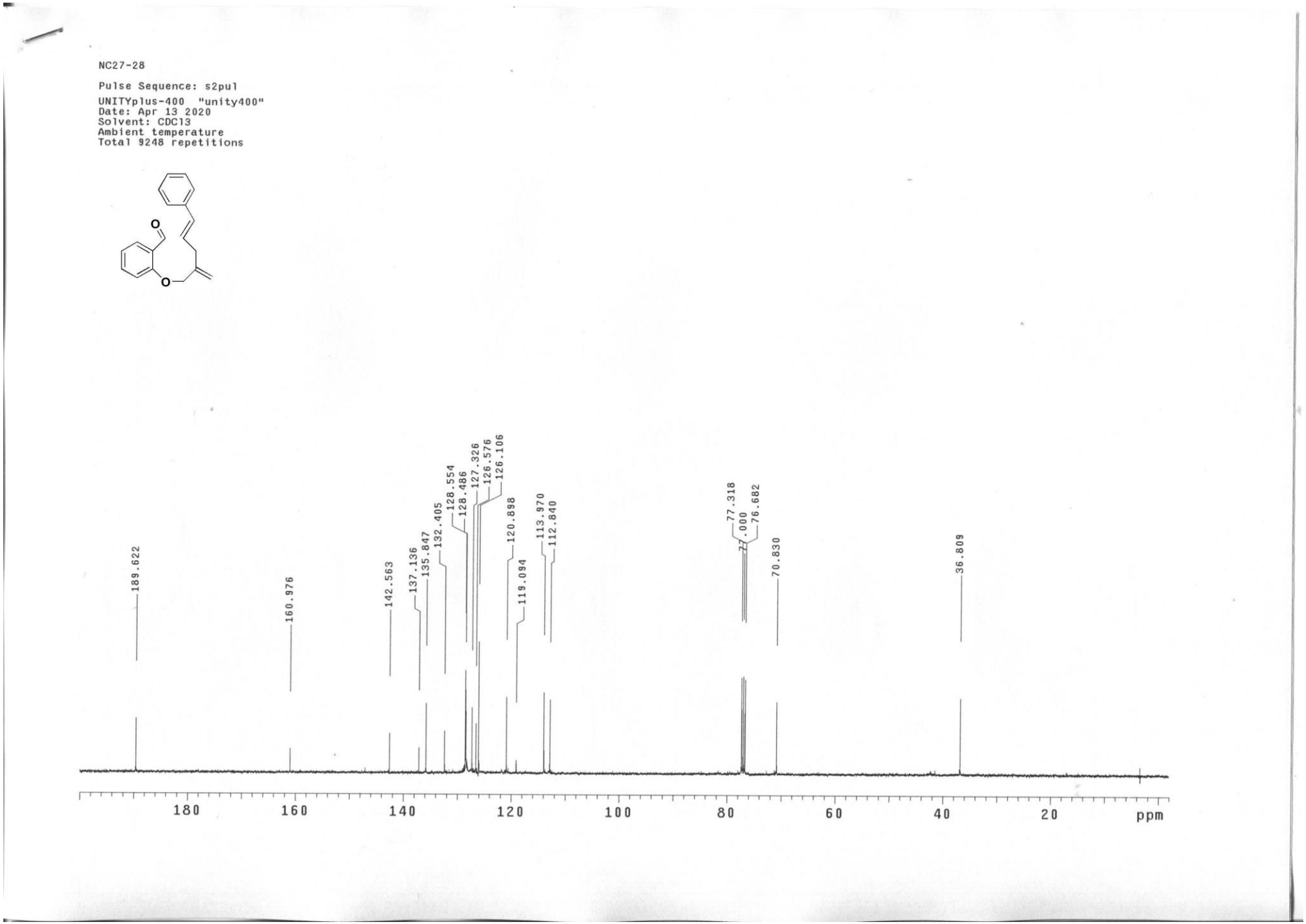
Compound 5v (^{13}C-NMR spectral data)



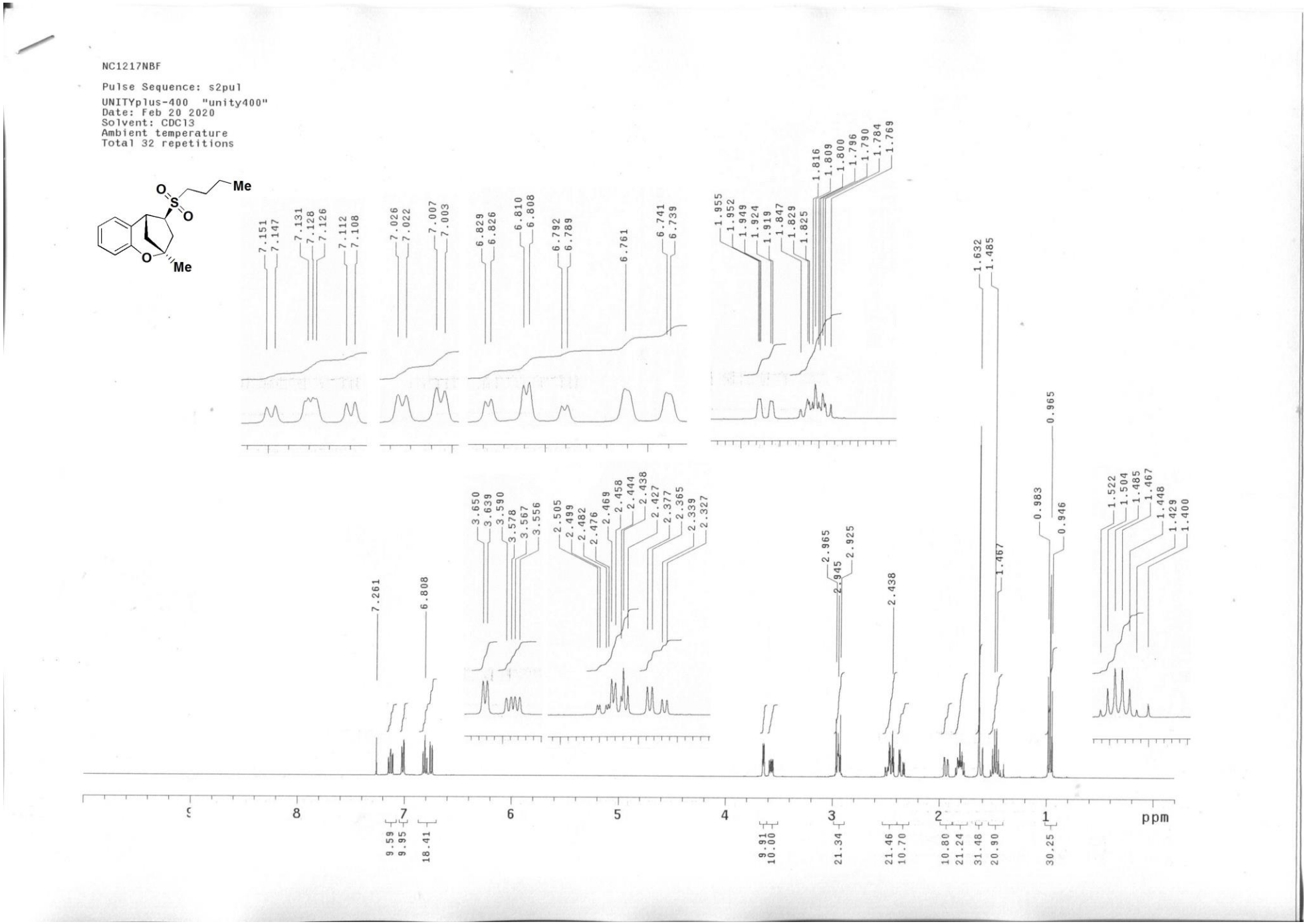
Compound 5y (^{1}H-NMR spectral data)



Compound 5y (^{13}C-NMR spectral data)



Compound 7a (^{1}H-NMR spectral data)

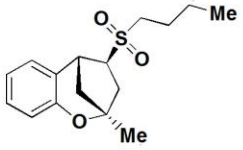


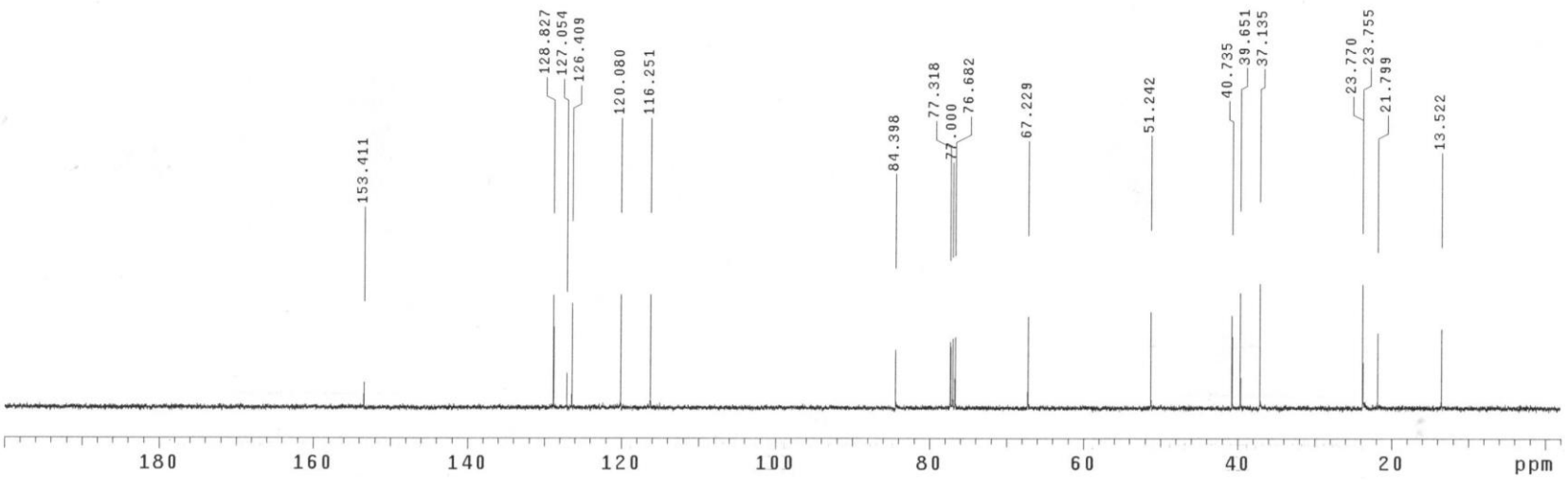
Compound 7a (^{13}C-NMR spectral data)

NC1217NBF

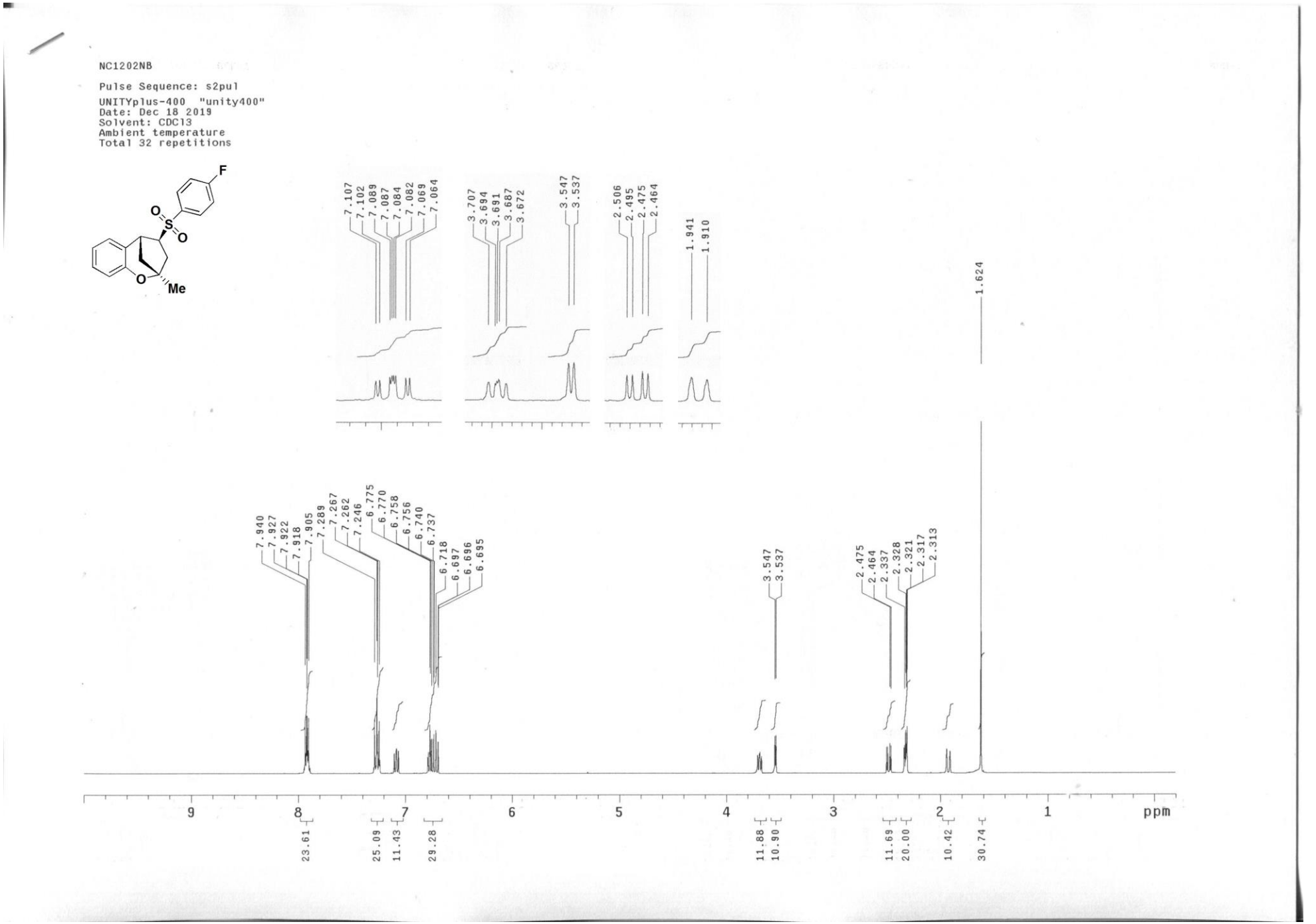
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Feb 20 2020
Solvent: CDCl3
Ambient temperature
Total 1248 repetitions





Compound 7b (^{1}H-NMR spectral data)

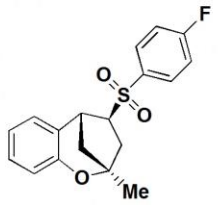


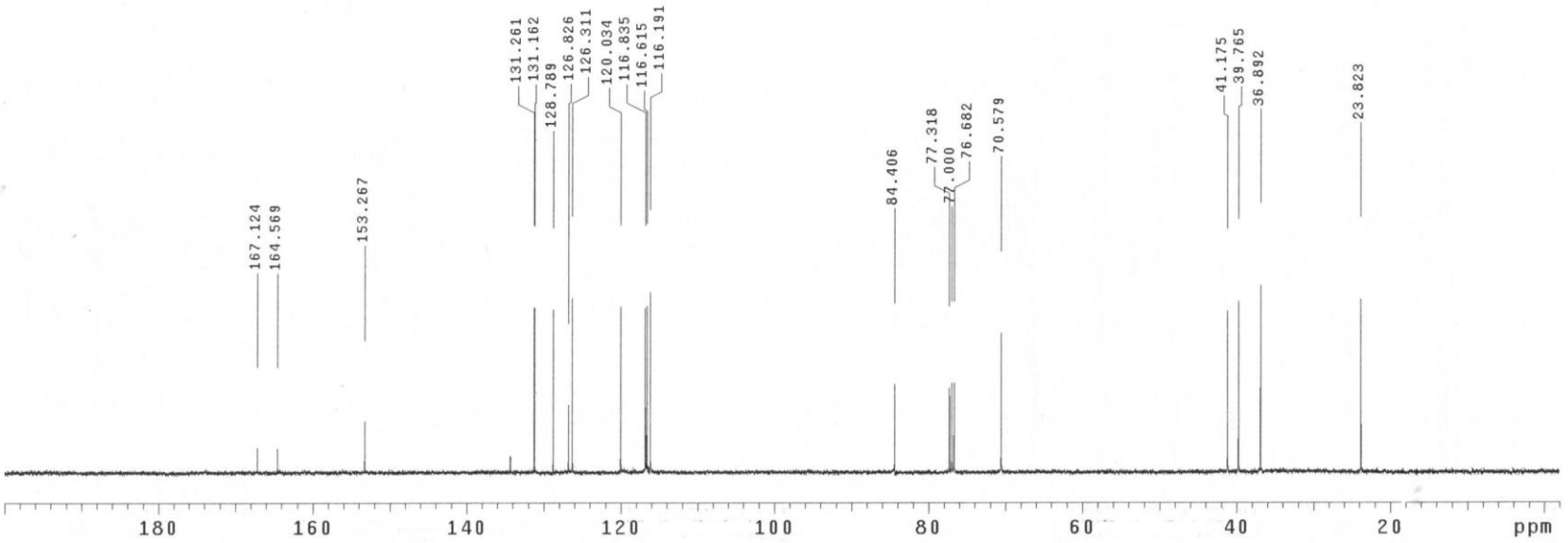
Compound 7b (^{13}C-NMR spectral data)

NC1202NB

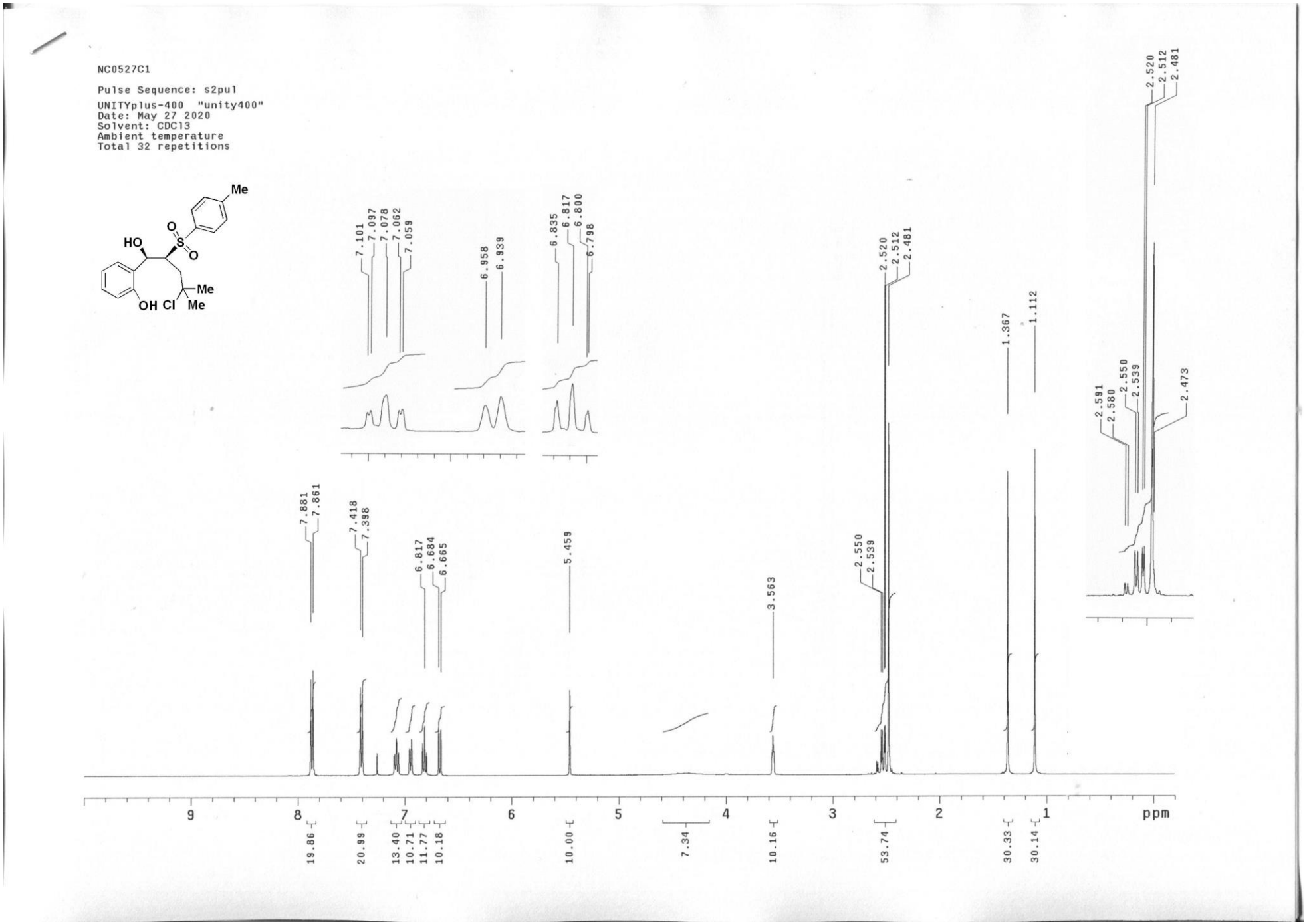
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Dec 18 2019
Solvent: CDCl3
Ambient temperature
Total 1520 repetitions

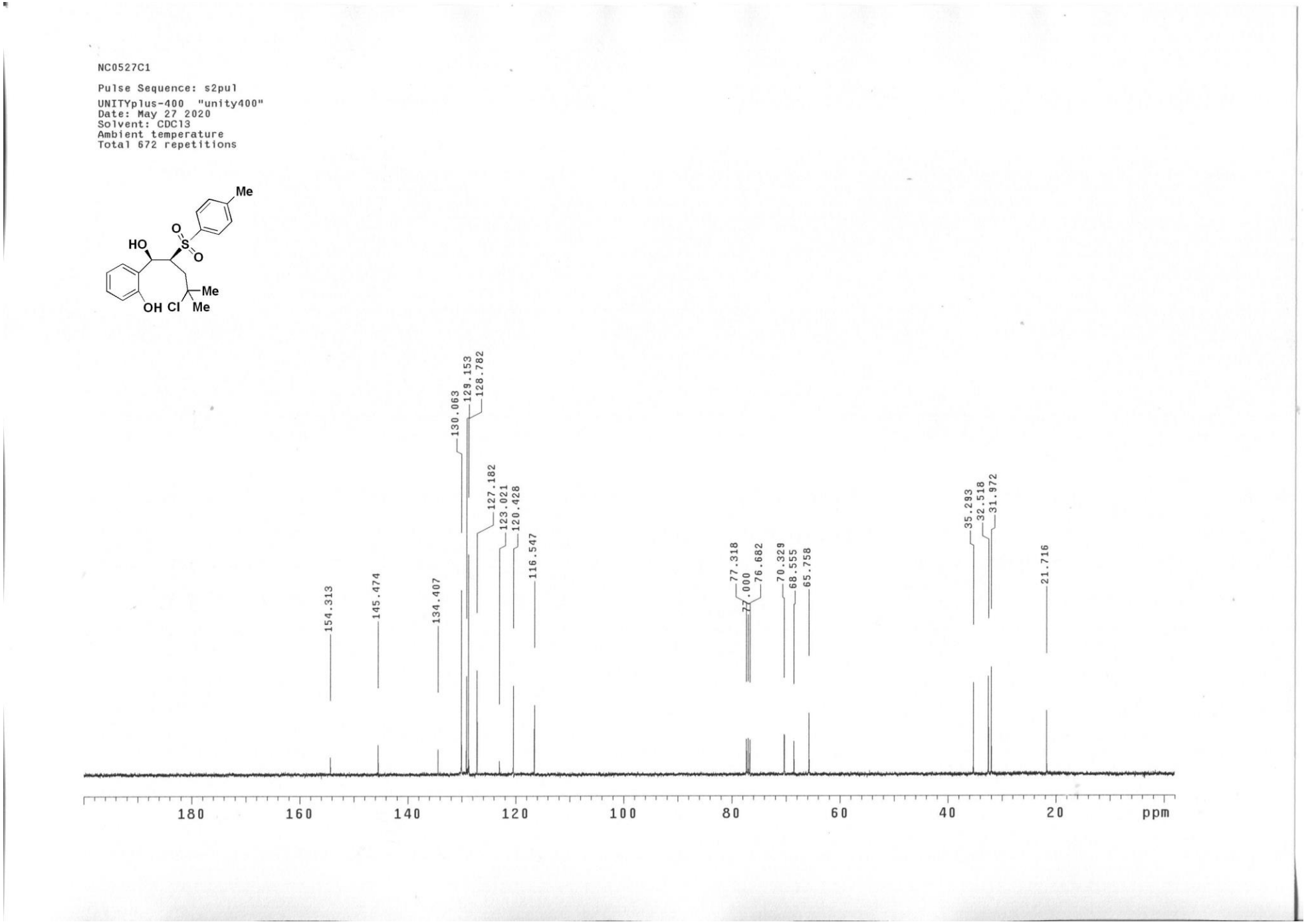




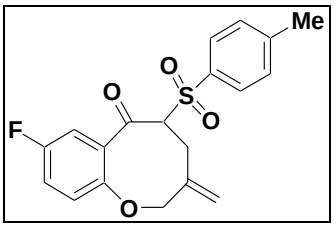
Compound 9 (^{1}H-NMR spectral data)



Compound 9 (^{13}C-NMR spectral data)

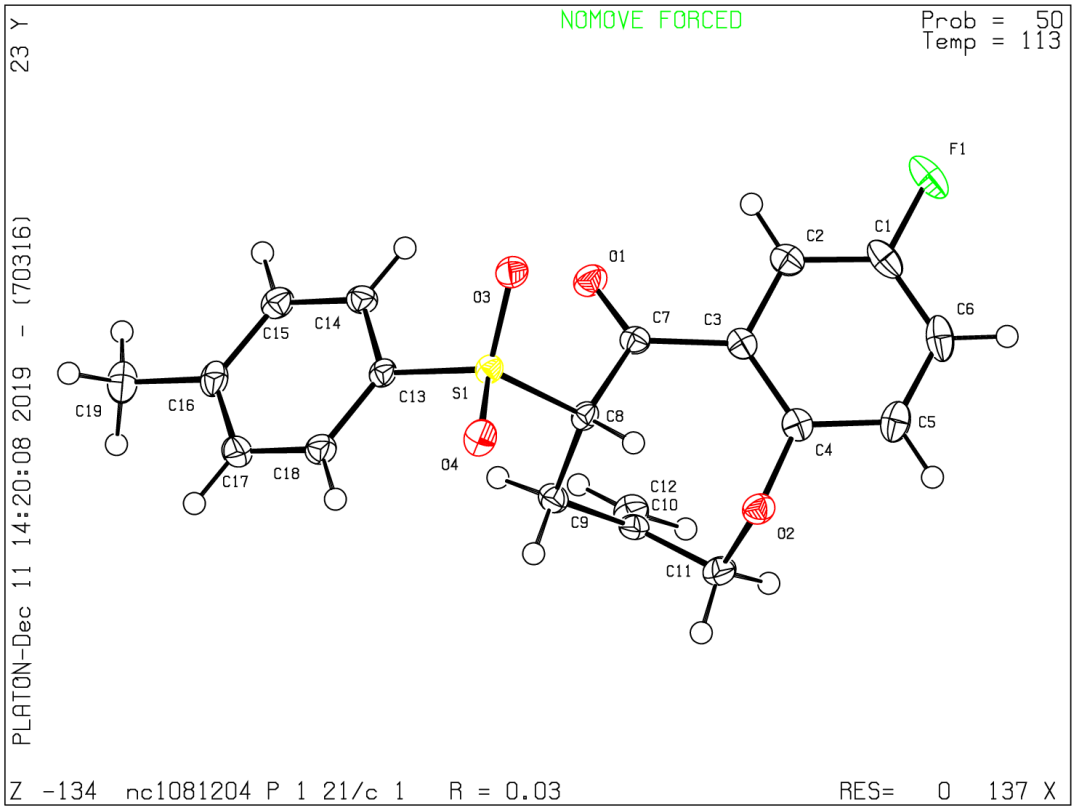


X-ray crystal data of compound 4l (CCDC 2003255)



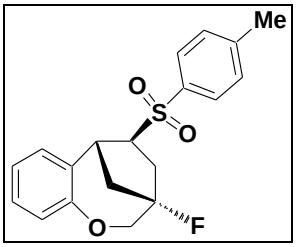
Sample preparation : A solution of compound **4l** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



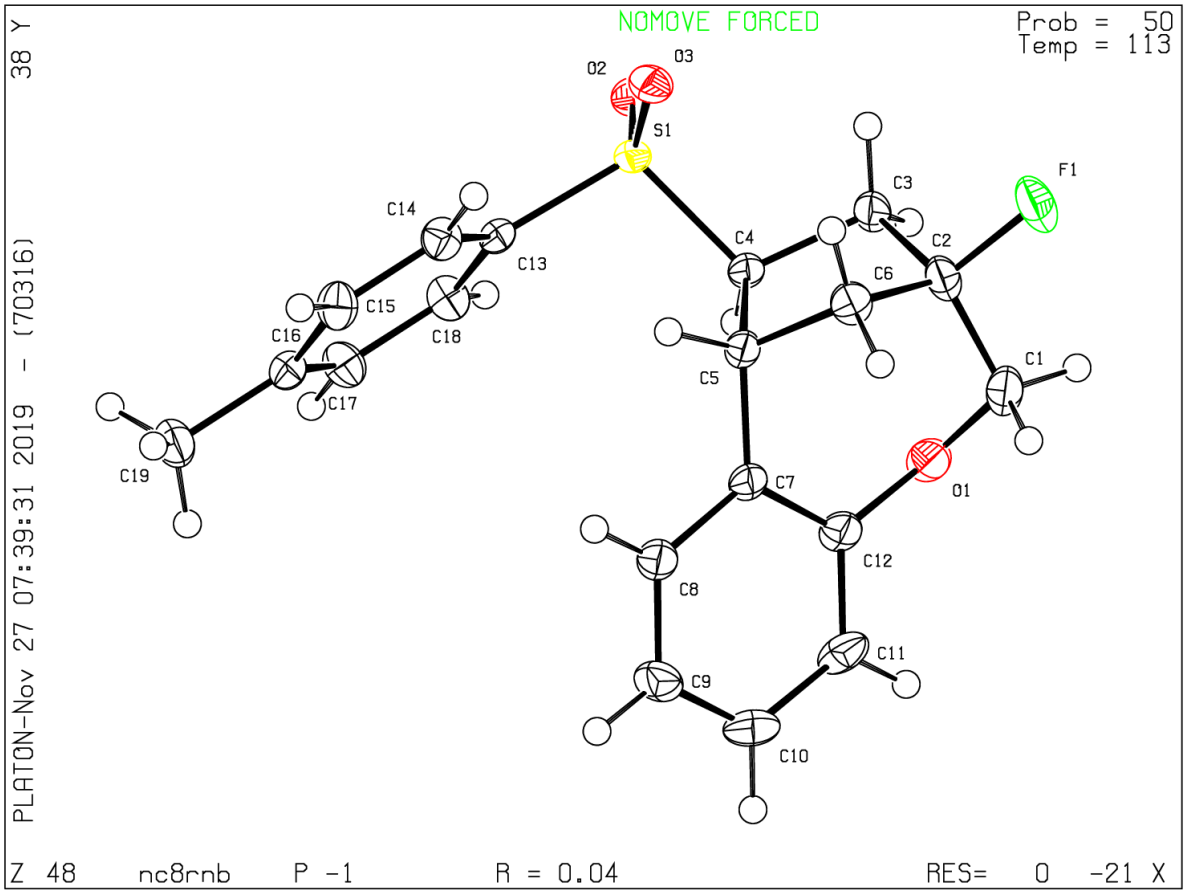
Empirical formula	C19 H17 F O4 S	
Formula weight	360.38	
Temperature	113(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/c 1	
Unit cell dimensions	a = 9.0958(2) Å	α = 90°.
	b = 11.0238(3) Å	β = 98.035(2)°.
	c = 16.3016(3) Å	γ = 90°.
Volume	1618.52(6) $Å^3$	
Z	4	
Density (calculated)	1.479 Mg/m^3	
Absorption coefficient	0.233 mm^{-1}	
F(000)	752	
Crystal size	0.25 x 0.2 x 0.1 mm^3	
Theta range for data collection	2.237 to 26.990°.	
Index ranges	-11<=h<=11, -13<=k<=14, -20<=l<=20	
Reflections collected	16444	
Independent reflections	3383 [R(int) = 0.0259]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.47803	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3383 / 0 / 235	
Goodness-of-fit on F^2	1.037	
Final R indices [I>2sigma(I)]	R1 = 0.0332, wR2 = 0.0755	
R indices (all data)	R1 = 0.0415, wR2 = 0.0783	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.333 and -0.384 $e.Å^{-3}$	

X-ray crystal data of compound 5d (CCDC 2003256)



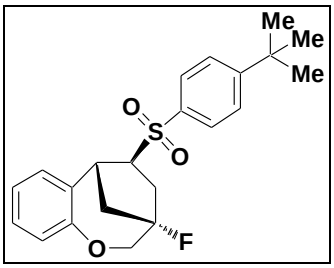
Sample preparation : A solution of compound **5d** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



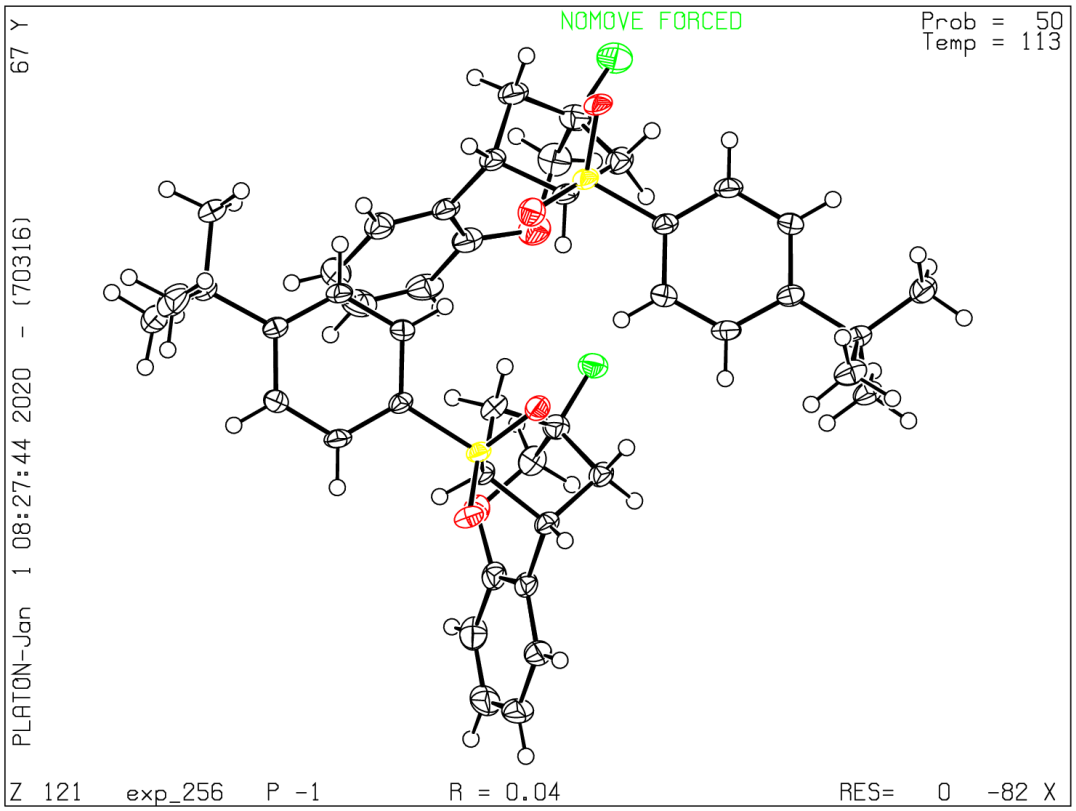
Empirical formula	C19 H19 F O3 S	
Formula weight	346.40	
Temperature	113(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 8.1798(3) Å	α = 88.093(4)°.
	b = 8.2382(4) Å	β= 81.665(4)°.
	c = 12.9438(7) Å	γ = 73.510(4)°.
Volume	827.50(7) $Å^3$	
Z	2	
Density (calculated)	1.390 Mg/m^3	
Absorption coefficient	0.220 mm^{-1}	
F(000)	364	
Crystal size	0.3 x 0.25 x 0.25 mm^3	
Theta range for data collection	2.578 to 27.055°.	
Index ranges	-10<=h<=10, -10<=k<=7, -16<=l<=16	
Reflections collected	12519	
Independent reflections	3425 [R(int) = 0.0379]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.56378	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3425 / 0 / 219	
Goodness-of-fit on F^2	1.060	
Final R indices [I>2sigma(I)]	R1 = 0.0375, wR2 = 0.0928	
R indices (all data)	R1 = 0.0459, wR2 = 0.0971	
Extinction coefficient	0.009(2)	
Largest diff. peak and hole	0.300 and -0.426 $e.Å^{-3}$	

X-ray crystal data of compound 5j (CCDC 2003257)



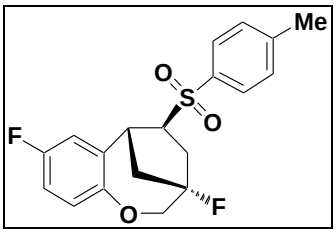
Sample preparation : A solution of compound **5j** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



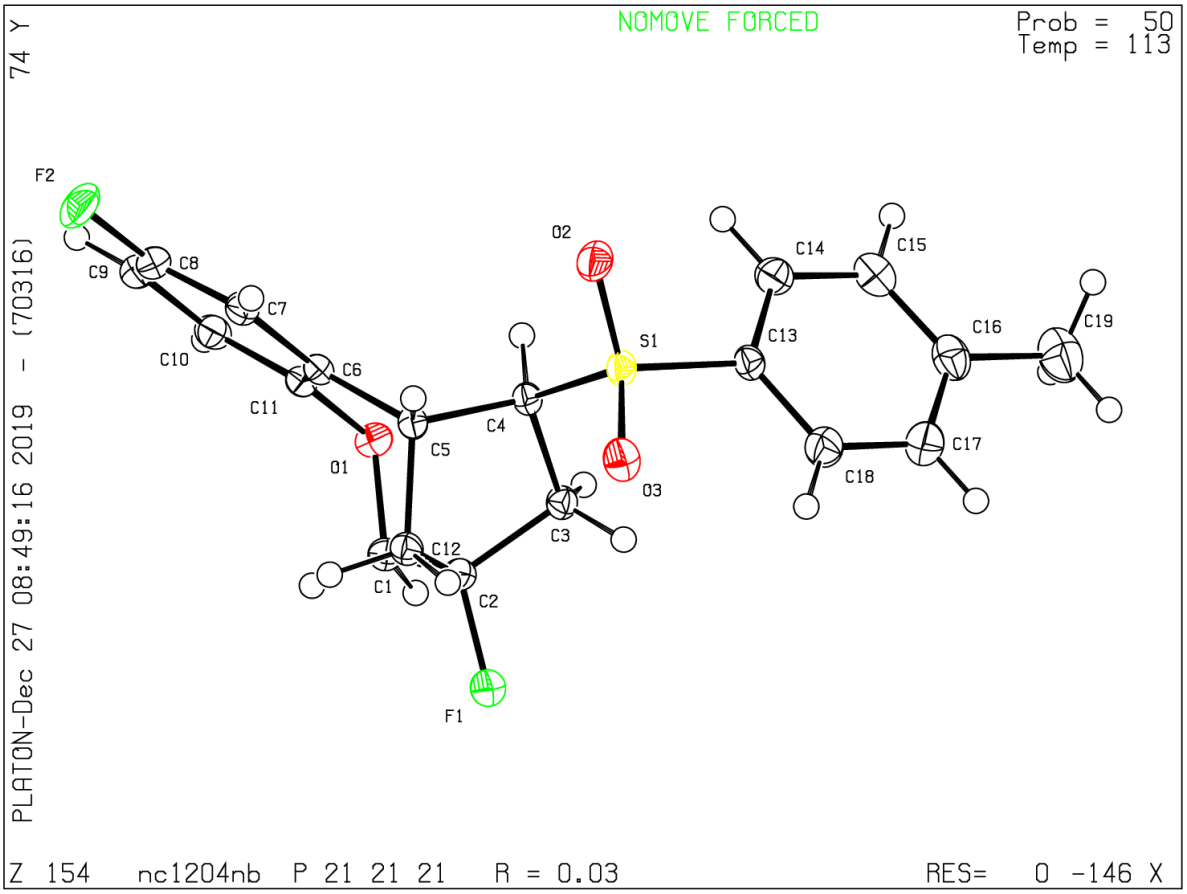
Formula weight	194.24	
Temperature	113(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.5216(3) Å	α = 114.737(2)°.
	b = 14.9812(4) Å	β = 100.424(2)°.
	c = 15.0603(3) Å	γ = 104.503(2)°.
Volume	1974.08(9) $Å^3$	
Z	8	
Density (calculated)	1.307 Mg/m^3	
Absorption coefficient	0.192 mm^{-1}	
F(000)	824	
Crystal size	0.2 x 0.2 x 0.15 mm^3	
Theta range for data collection	2.114 to 27.076°.	
Index ranges	-13<=h<=13, -18<=k<=19, -19<=l<=19	
Reflections collected	33783	
Independent reflections	8215 [R(int) = 0.0422]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.57917	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	8215 / 0 / 493	
Goodness-of-fit on F^2	1.079	
Final R indices [I>2sigma(I)]	R1 = 0.0432, wR2 = 0.1083	
R indices (all data)	R1 = 0.0626, wR2 = 0.1150	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.463 and -0.462 $e.Å^{-3}$	

X-ray crystal data of compound 5l (CCDC 2003258)



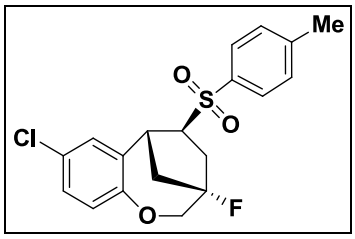
Sample preparation : A solution of compound **5l** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



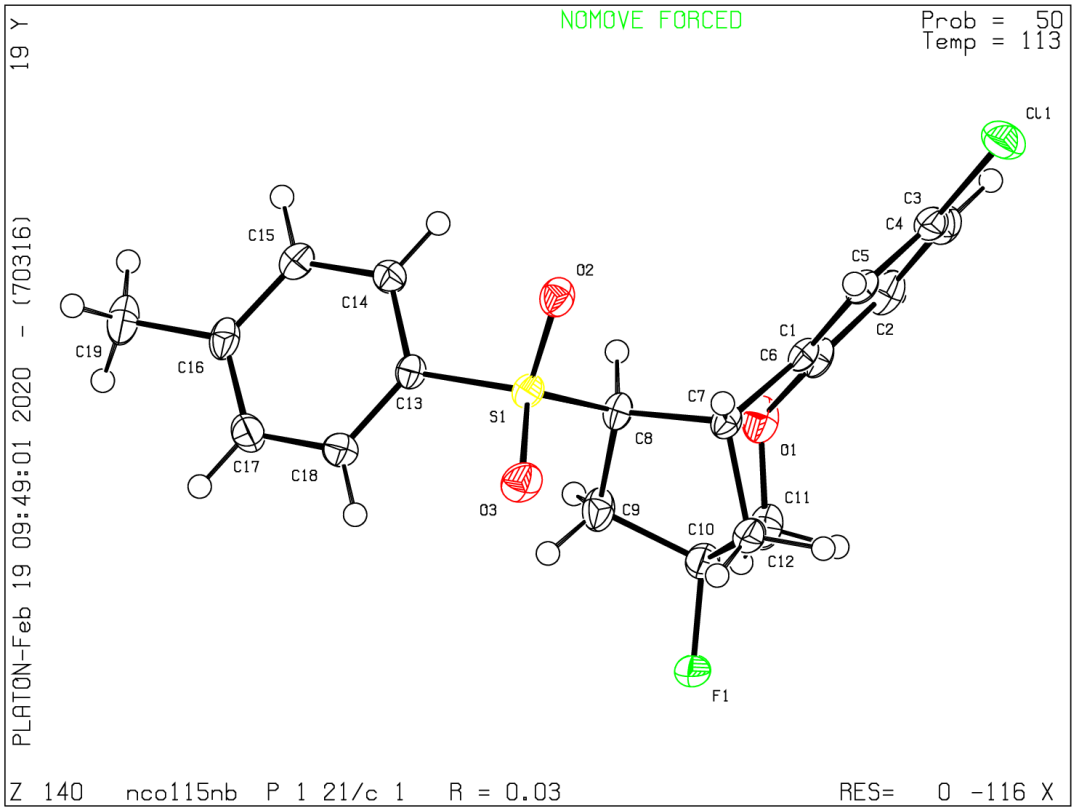
Empirical formula	C19 H18 F2 O3 S	
Formula weight	364.39	
Temperature	113(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	$P2_12_12_1$	
Unit cell dimensions	a = 5.88630(10) Å	α = 90°.
	b = 9.4890(2) Å	β = 90°.
	c = 29.6414(7) Å	γ = 90°.
Volume	1655.62(6) $Å^3$	
Z	4	
Density (calculated)	1.462 Mg/m^3	
Absorption coefficient	0.232 mm^{-1}	
F(000)	760	
Crystal size	0.25 x 0.25 x 0.2 mm^3	
Theta range for data collection	2.254 to 27.000°.	
Index ranges	-7<=h<=7, -12<=k<=12, -33<=l<=37	
Reflections collected	31130	
Independent reflections	3504 [R(int) = 0.0346]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.42029	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3504 / 115 / 227	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0314, wR2 = 0.0767	
R indices (all data)	R1 = 0.0336, wR2 = 0.0780	
Absolute structure parameter	0.00(2)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.324 and -0.282 $e.Å^{-3}$	

X-ray crystal data of compound 5m (CCDC 2003259)



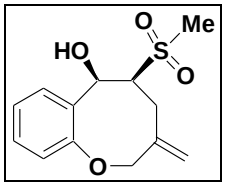
Sample preparation : A solution of compound **5m** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



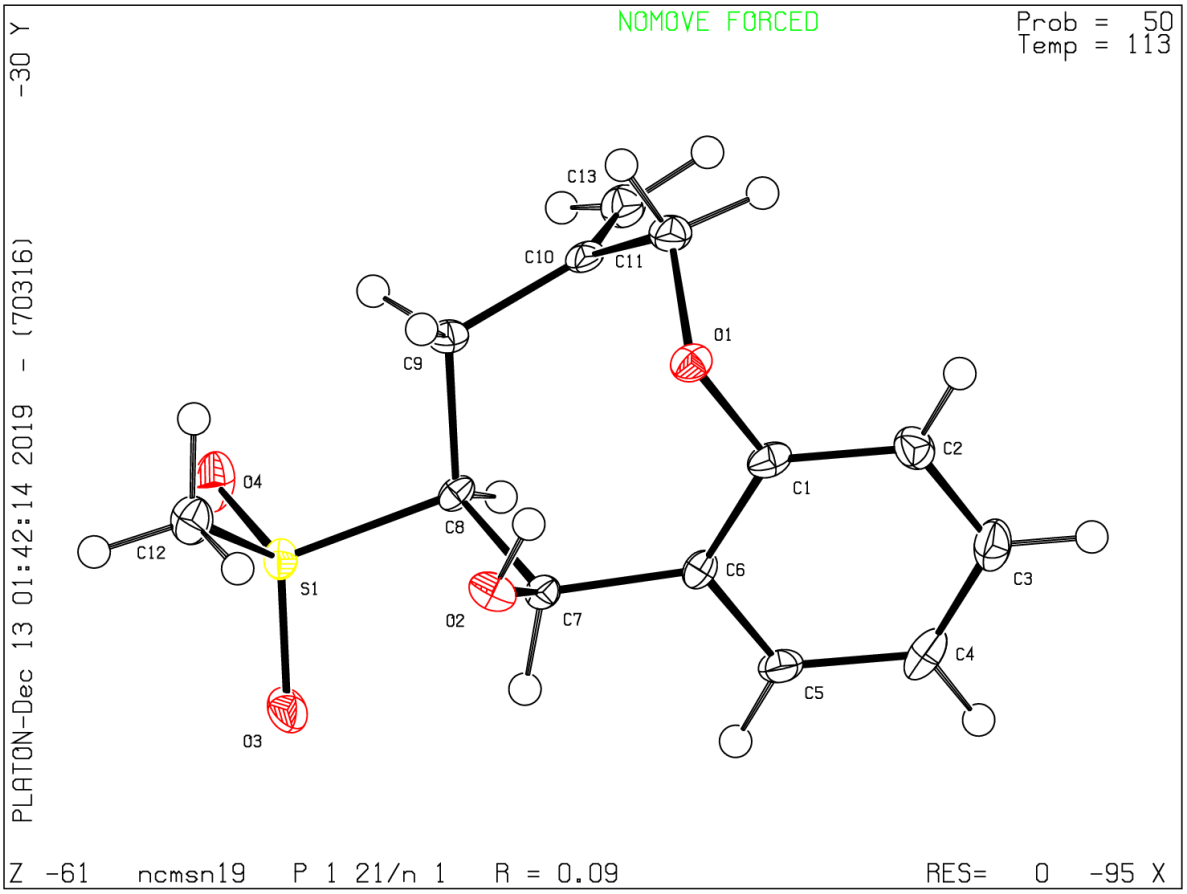
Empirical formula	$C_{19}H_{18}ClFO_3S$
Formula weight	380.84
Temperature/K	113(2)
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	14.9529(3)
b/Å	7.52540(10)
c/Å	15.8144(3)
α/°	90.00
β/°	106.409(2)
γ/°	90.00
Volume/Å^3	1707.06(5)
Z	4
ρ_{calc}g/cm^3	1.482
μ/mm^{-1}	0.372
F(000)	792.0
Crystal size/mm^3	0.2 × 0.2 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	5.32 to 52
Index ranges	$-18 \leq h \leq 17$, $-9 \leq k \leq 9$, $-19 \leq l \leq 17$
Reflections collected	31146
Independent reflections	3362 [R_{int} = 0.0313, R_{sigma} = 0.0181]
Data/restraints/parameters	3362/0/227
Goodness-of-fit on F^2	1.062
Final R indexes [I>=2σ (I)]	R_1 = 0.0307, wR_2 = 0.0783
Final R indexes [all data]	R_1 = 0.0343, wR_2 = 0.0799
Largest diff. peak/hole / e Å^{-3}	0.30/-0.39

X-ray crystal data of compound 6a (CCDC 2003260)



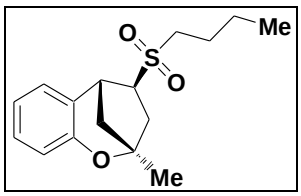
Sample preparation : A solution of compound **6a** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



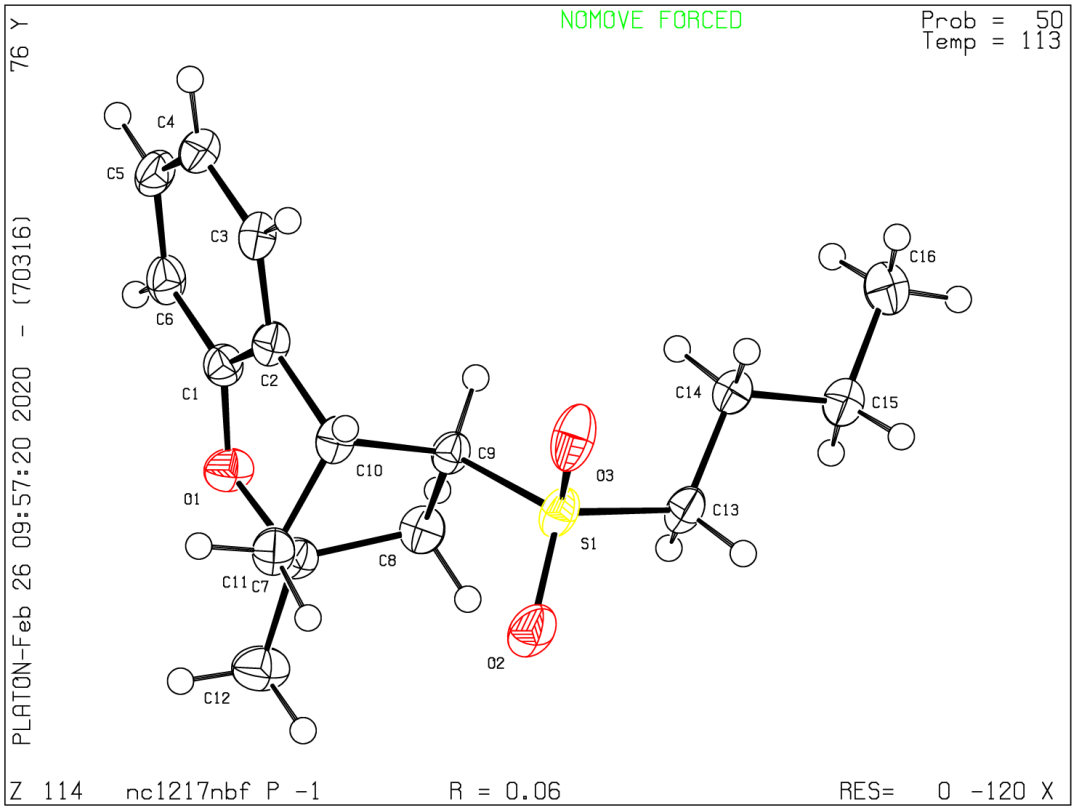
Empirical formula	C13 H16 O4 S	
Formula weight	268.32	
Temperature	113(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 1 21/n 1	
Unit cell dimensions	a = 8.1912(3) Å	α = 90°.
	b = 6.0583(2) Å	β = 92.806(4)°.
	c = 25.2332(11) Å	γ = 90°.
Volume	1250.69(8) $Å^3$	
Z	4	
Density (calculated)	1.425 Mg/m^3	
Absorption coefficient	0.263 mm^{-1}	
F(000)	568	
Crystal size	0.25 x 0.2 x 0.2 mm^3	
Theta range for data collection	2.580 to 27.034°.	
Index ranges	-10<=h<=10, -7<=k<=7, -31<=l<=29	
Reflections collected	10768	
Independent reflections	2550 [R(int) = 0.0372]	
Completeness to theta = 25.242°	98.3 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.47989	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2550 / 0 / 173	
Goodness-of-fit on F^2	1.195	
Final R indices [I>2sigma(I)]	R1 = 0.0854, wR2 = 0.2050	
R indices (all data)	R1 = 0.0921, wR2 = 0.2070	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.228 and -0.464 $e.Å^{-3}$	

X-ray crystal data of compound 7a (CCDC 2003261)



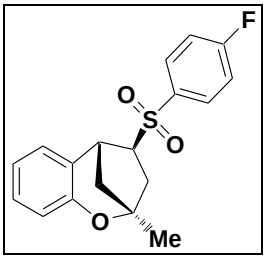
Sample preparation : A solution of compound **7a** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



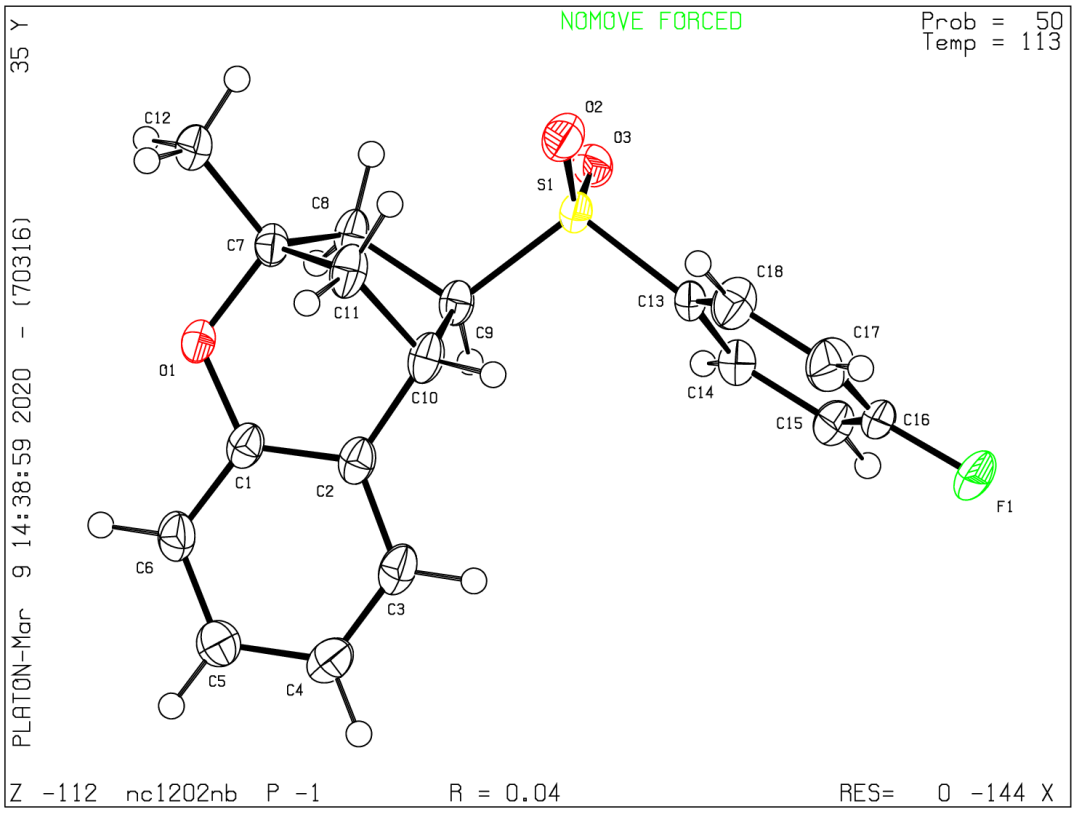
Empirical formula	$C_{16}H_{22}O_3S$
Formula weight	294.39
Temperature/K	113(2)
Crystal system	triclinic
Space group	P-1
a/Å	5.5686(2)
b/Å	9.5682(3)
c/Å	15.1726(5)
α/°	105.346(3)
β/°	92.676(2)
γ/°	102.631(3)
Volume/Å^3	756.02(5)
Z	2
ρ_{calc}g/cm^3	1.293
μ/mm^{-1}	0.219
F(000)	316.0
Crystal size/mm^3	0.15 × 0.1 × 0.1
Radiation	Mo Kα (λ = 0.71073)
2Θ range for data collection/°	4.548 to 54.142
Index ranges	$-7 \leq h \leq 6$, $-12 \leq k \leq 11$, $-19 \leq l \leq 19$
Reflections collected	16987
Independent reflections	3170 [R_{int} = 0.0451, R_{sigma} = 0.0366]
Data/restraints/parameters	3170/0/191
Goodness-of-fit on F^2	1.088
Final R indexes [I>=2σ (I)]	R_1 = 0.0572, wR_2 = 0.1449
Final R indexes [all data]	R_1 = 0.0670, wR_2 = 0.1489
Largest diff. peak/hole / e Å^{-3}	0.61/-0.38

X-ray crystal data of compound 7b (CCDC 2003262)



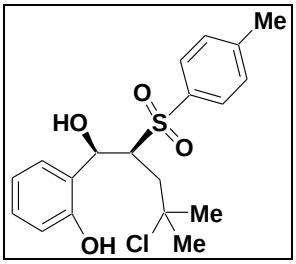
Sample preparation : A solution of compound **7b** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



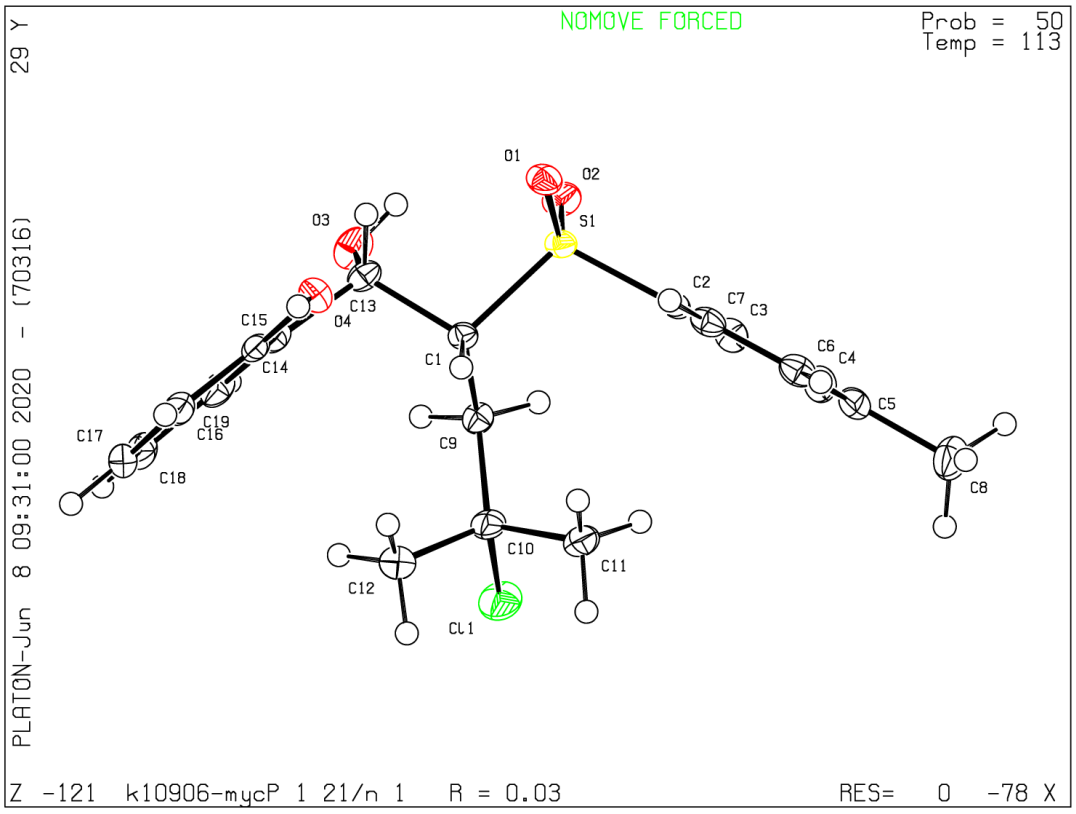
Empirical formula	$C_{18}H_{17}FO_3S$
Formula weight	332.38
Temperature/K	113(2)
Crystal system	triclinic
Space group	P-1
a/Å	6.87130(10)
b/Å	10.4176(3)
c/Å	11.8932(3)
α/°	99.558(2)
β/°	105.692(2)
γ/°	96.770(2)
Volume/Å^3	796.26(3)
Z	2
ρ_{calc}g/cm^3	1.386
μ/mm^{-1}	0.226
F(000)	348.0
Crystal size/mm^3	0.15 × 0.15 × 0.1
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.64 to 52
Index ranges	$-8 \leq h \leq 8$, $-12 \leq k \leq 12$, $-14 \leq l \leq 14$
Reflections collected	12550
Independent reflections	3134 [R_{int} = 0.0226, R_{sigma} = 0.0250]
Data/restraints/parameters	3134/0/209
Goodness-of-fit on F^2	1.076
Final R indexes [I>=2σ (I)]	R_1 = 0.0416, wR_2 = 0.1025
Final R indexes [all data]	R_1 = 0.0484, wR_2 = 0.1059
Largest diff. peak/hole / e Å^{-3}	0.71/-0.52

X-ray crystal data of compound 9 (CCDC 2008547)



Sample preparation : A solution of compound **9** (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	$C_{19}H_{23}ClO_4S$
Formula weight	389.36
Temperature/K	113(2)
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	14.5435(3)
b/Å	6.0581(2)
c/Å	20.5872(4)
α/°	90.00
β/°	93.464(2)
γ/°	90.00
Volume/Å^3	1810.54(8)
Z	4
ρ_{calc}g/cm^3	1.428
μ/mm^{-1}	0.353
F(000)	825.0
Crystal size/mm^3	0.3 × 0.3 × 0.2
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.96 to 54.14
Index ranges	$-18 \leq h \leq 18$, $-6 \leq k \leq 7$, $-26 \leq l \leq 25$
Reflections collected	38190
Independent reflections	3861 [R_{int} = 0.0328, R_{sigma} = 0.0190]
Data/restraints/parameters	3861/0/231
Goodness-of-fit on F^2	1.072
Final R indexes [I>=2σ (I)]	R_1 = 0.0332, wR_2 = 0.0874
Final R indexes [all data]	R_1 = 0.0379, wR_2 = 0.0898
Largest diff. peak/hole / e Å^{-3}	0.38/-0.35