Supporting Information

One-pot Construction of Dispirocyclohexanes via K_2CO_3-Mediated Desulfonylative Michael-Michael-aldol (2+2+2) Annulation of Sulfonyl 2-Pyridyl Flavanones and Methylketones

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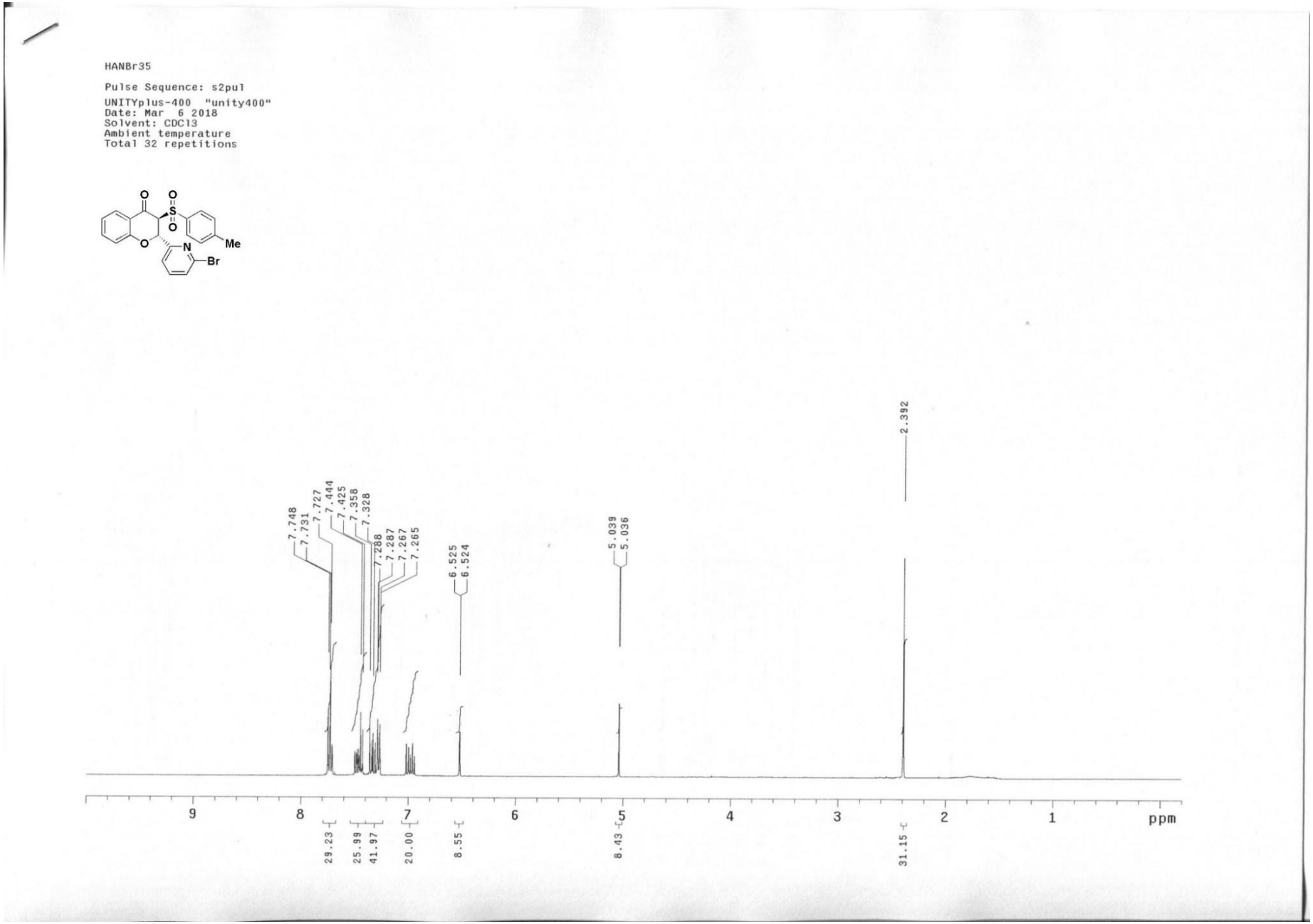
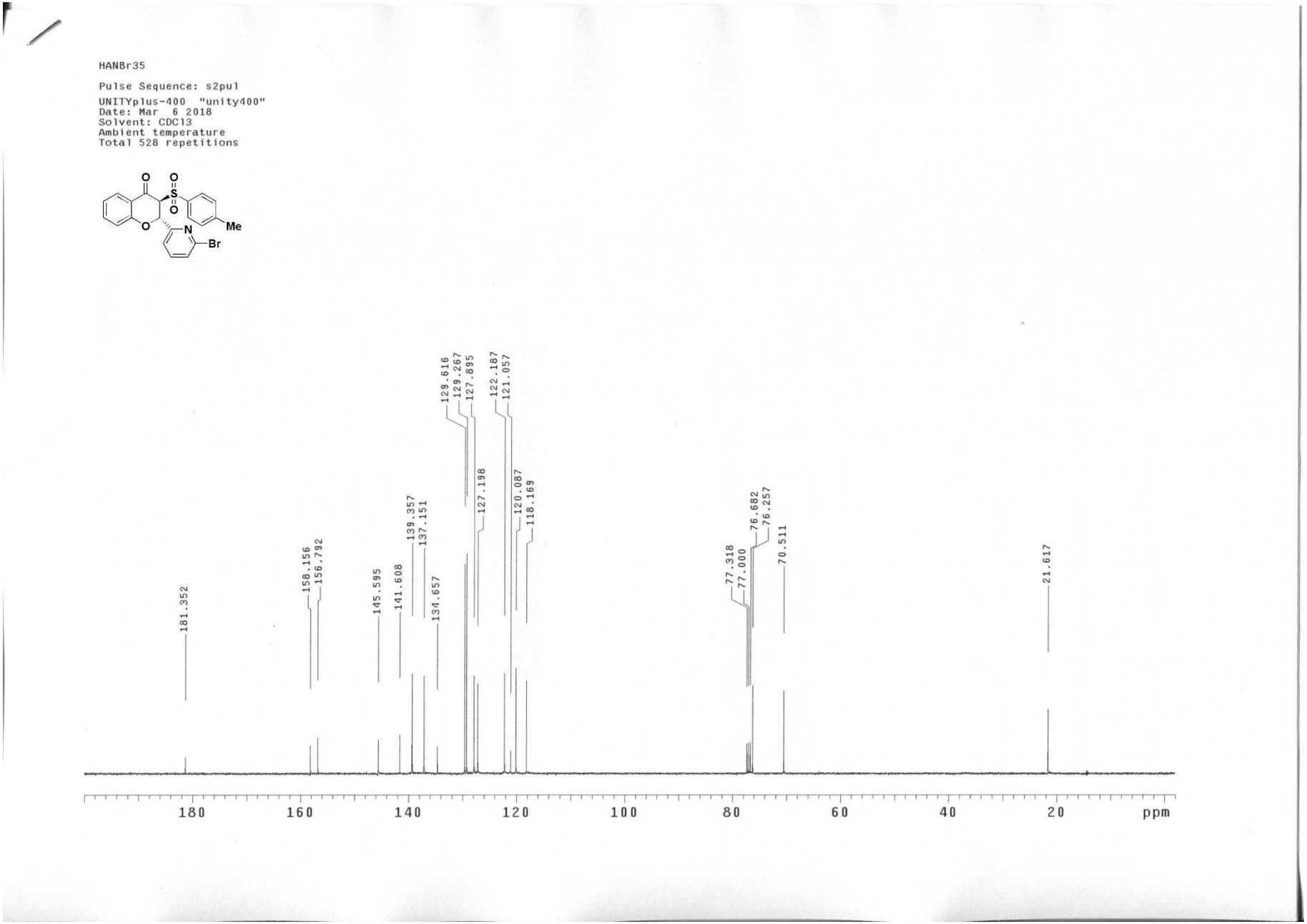
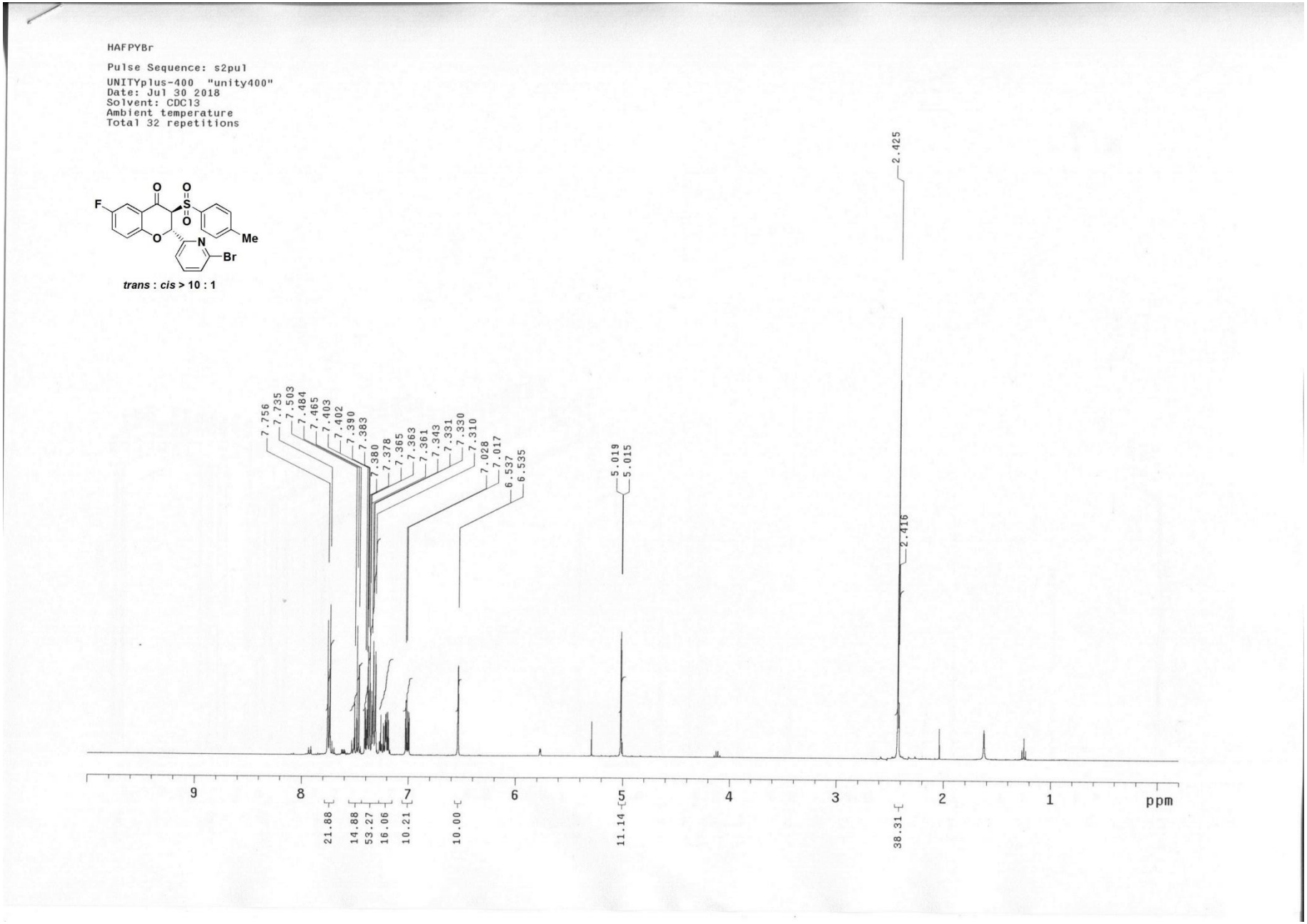
Compound 2a (^{1}H-NMR spectral data)



Compound 2a (^{13}C-NMR spectral data)



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



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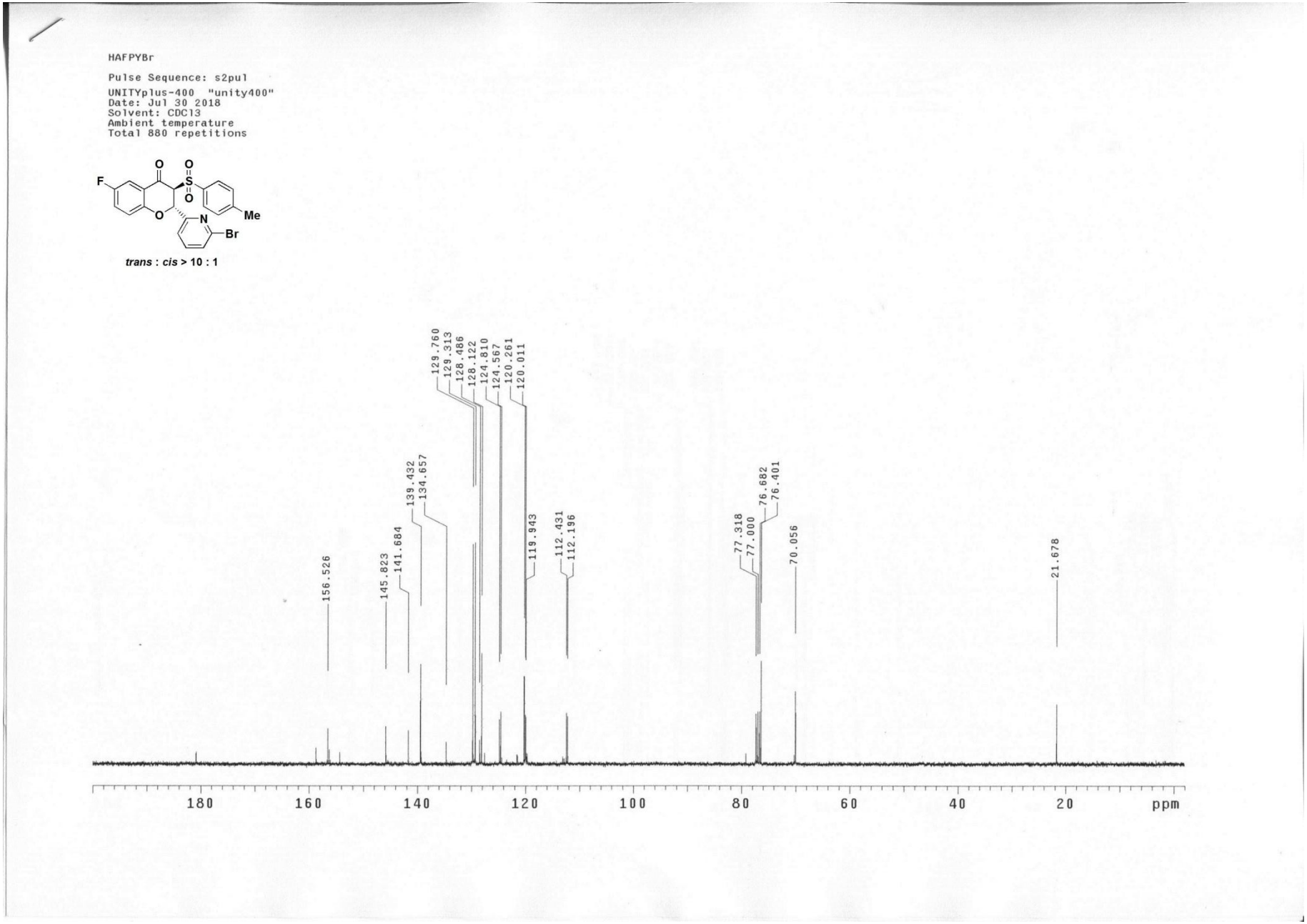

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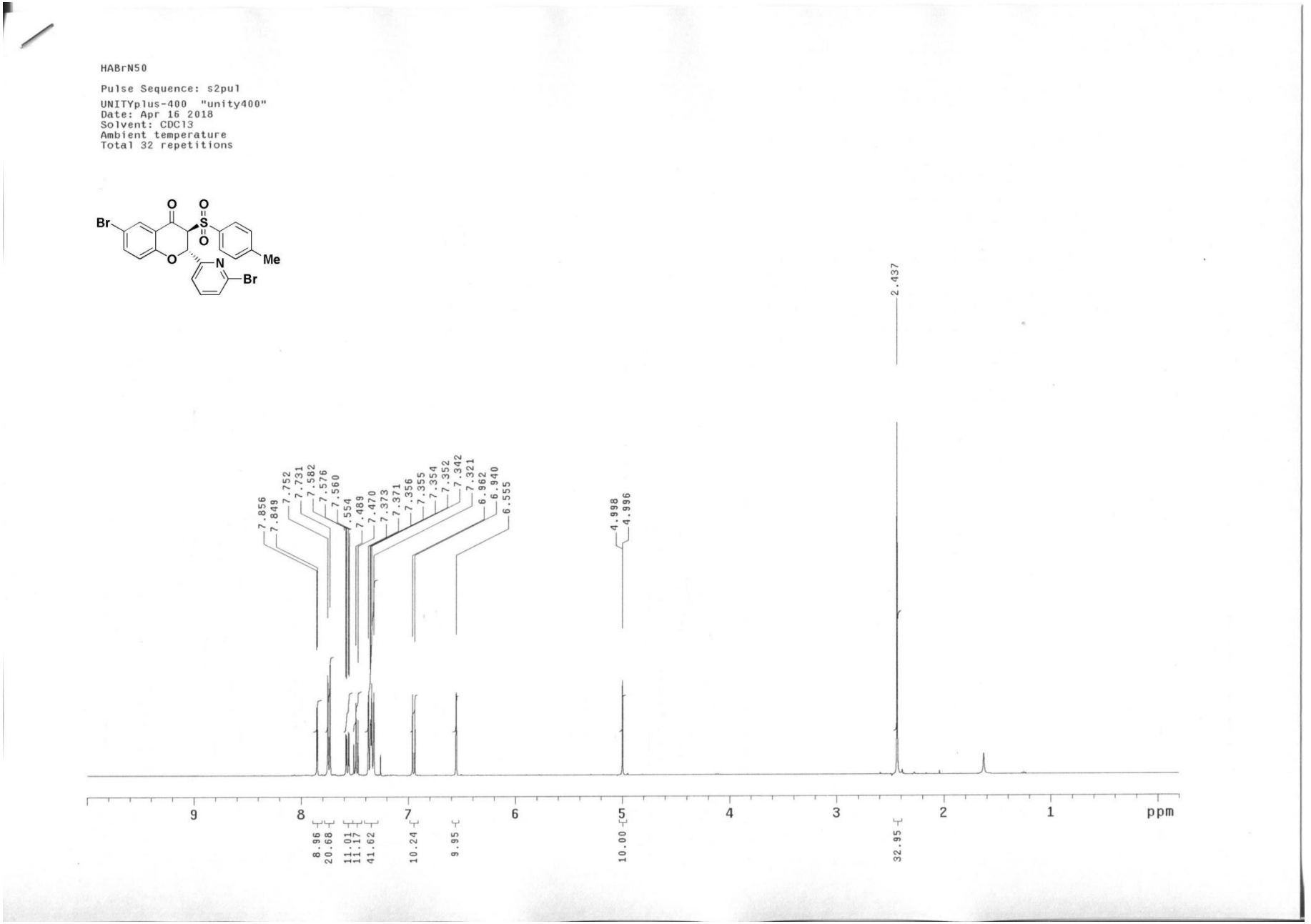

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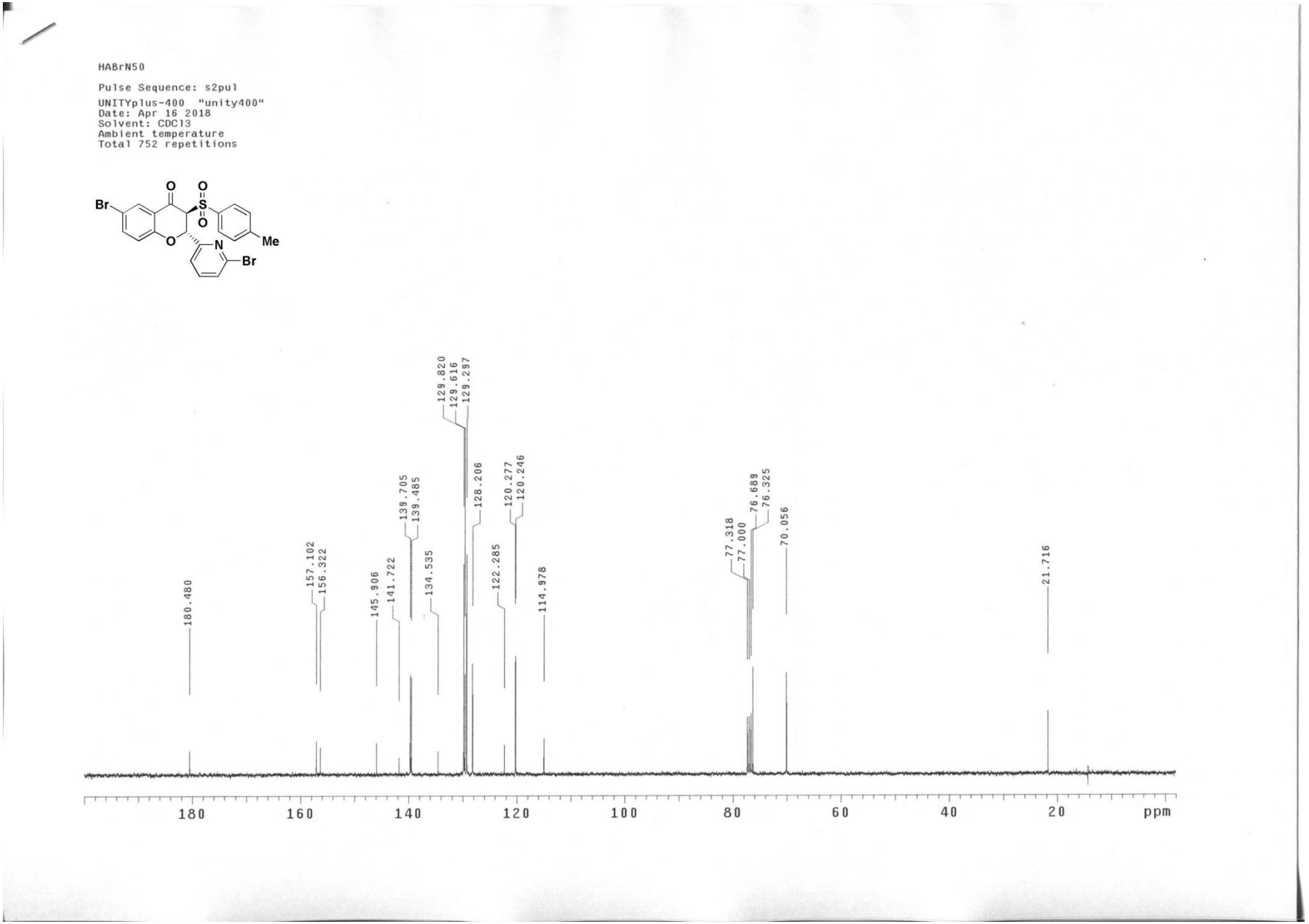

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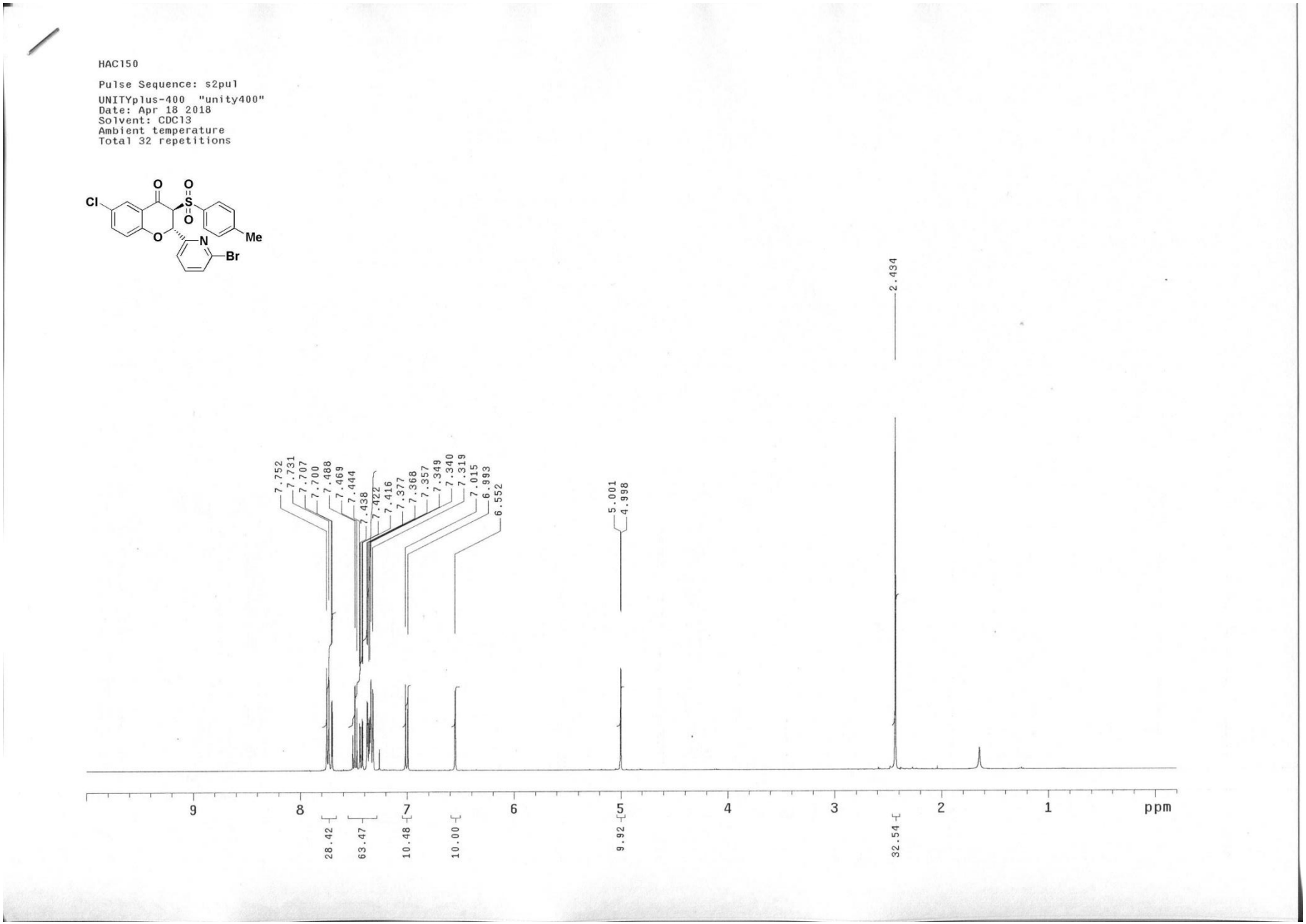

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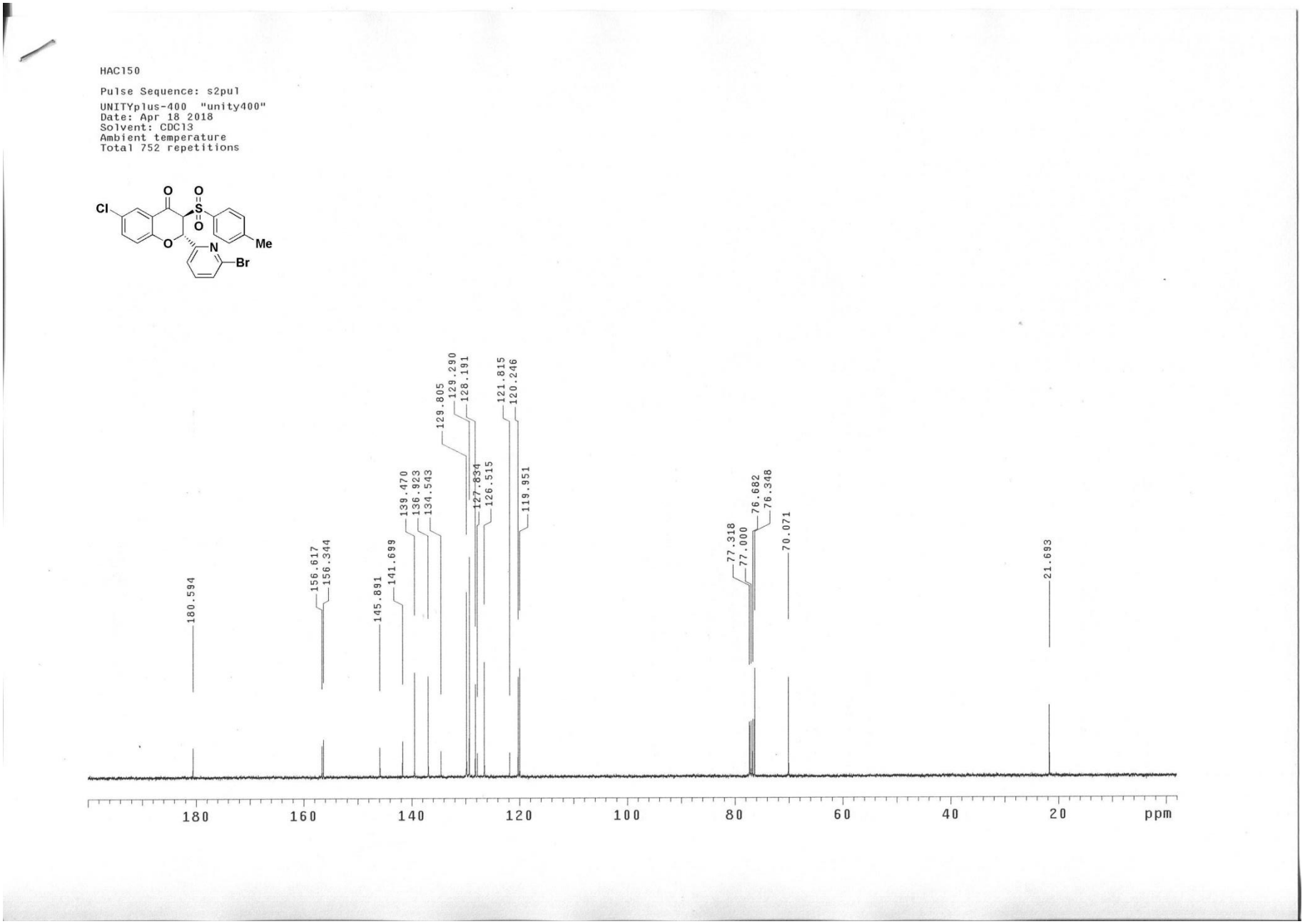

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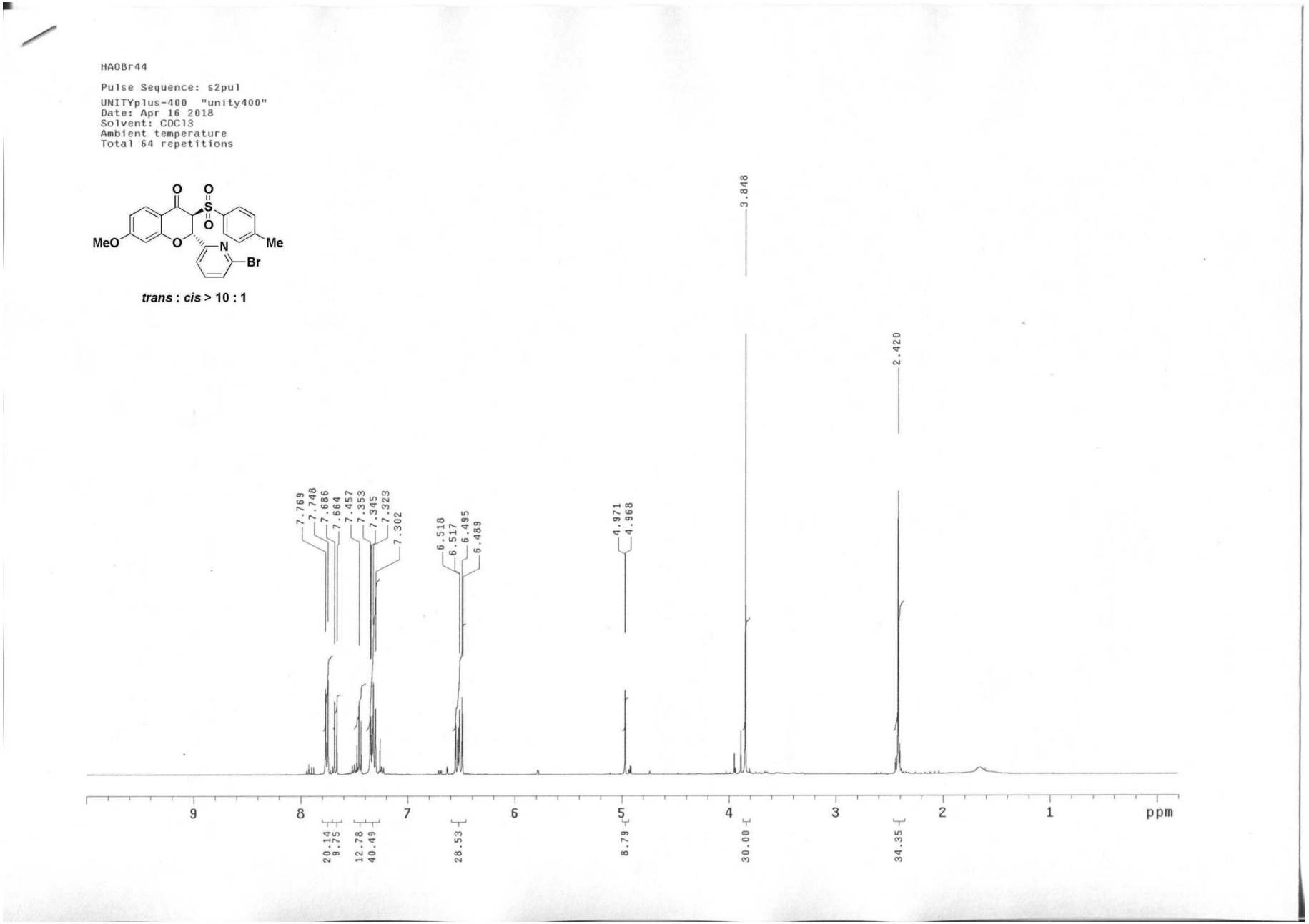

Compound 2e (^{13}C-NMR spectral data)

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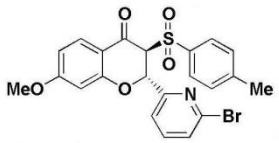
Date: Apr 16 2018
Solvent: CDCl3
Ambient temperature
Total 1200 repetitions



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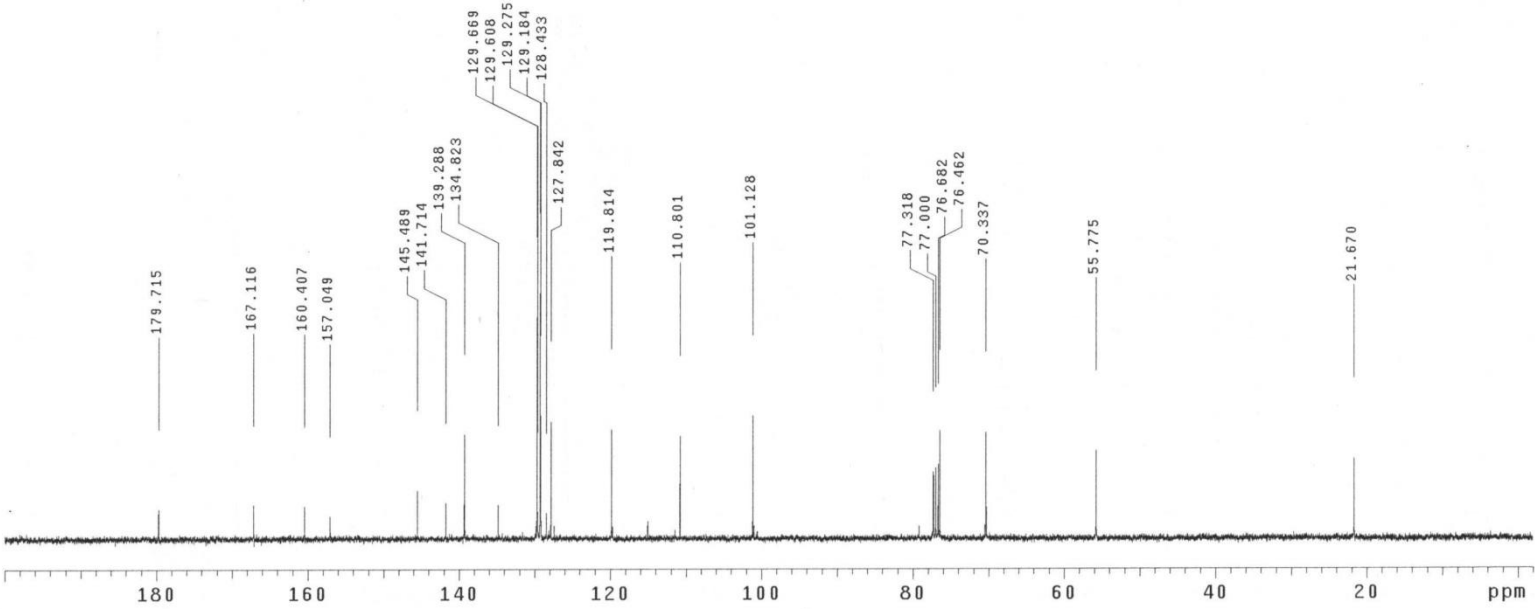

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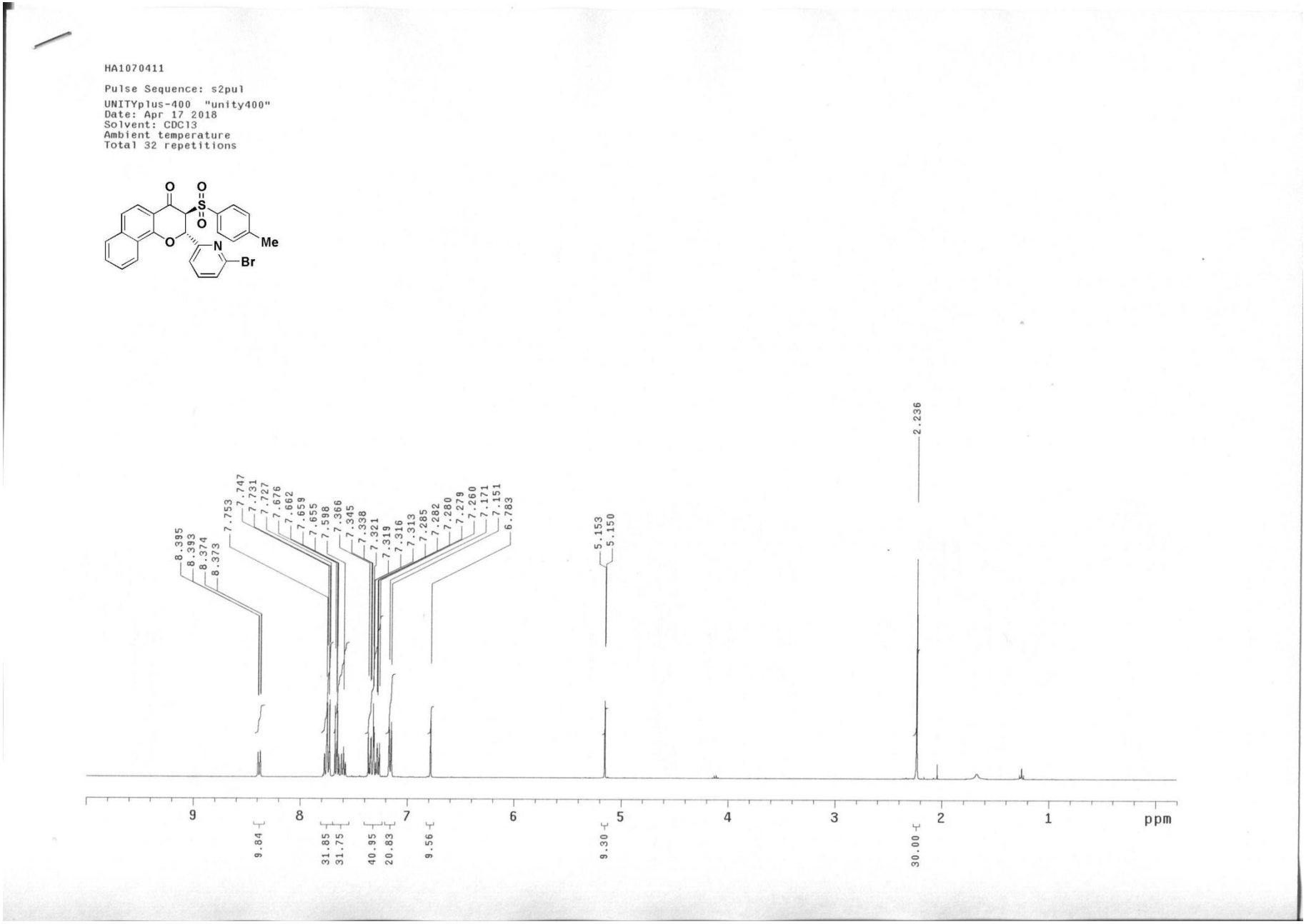

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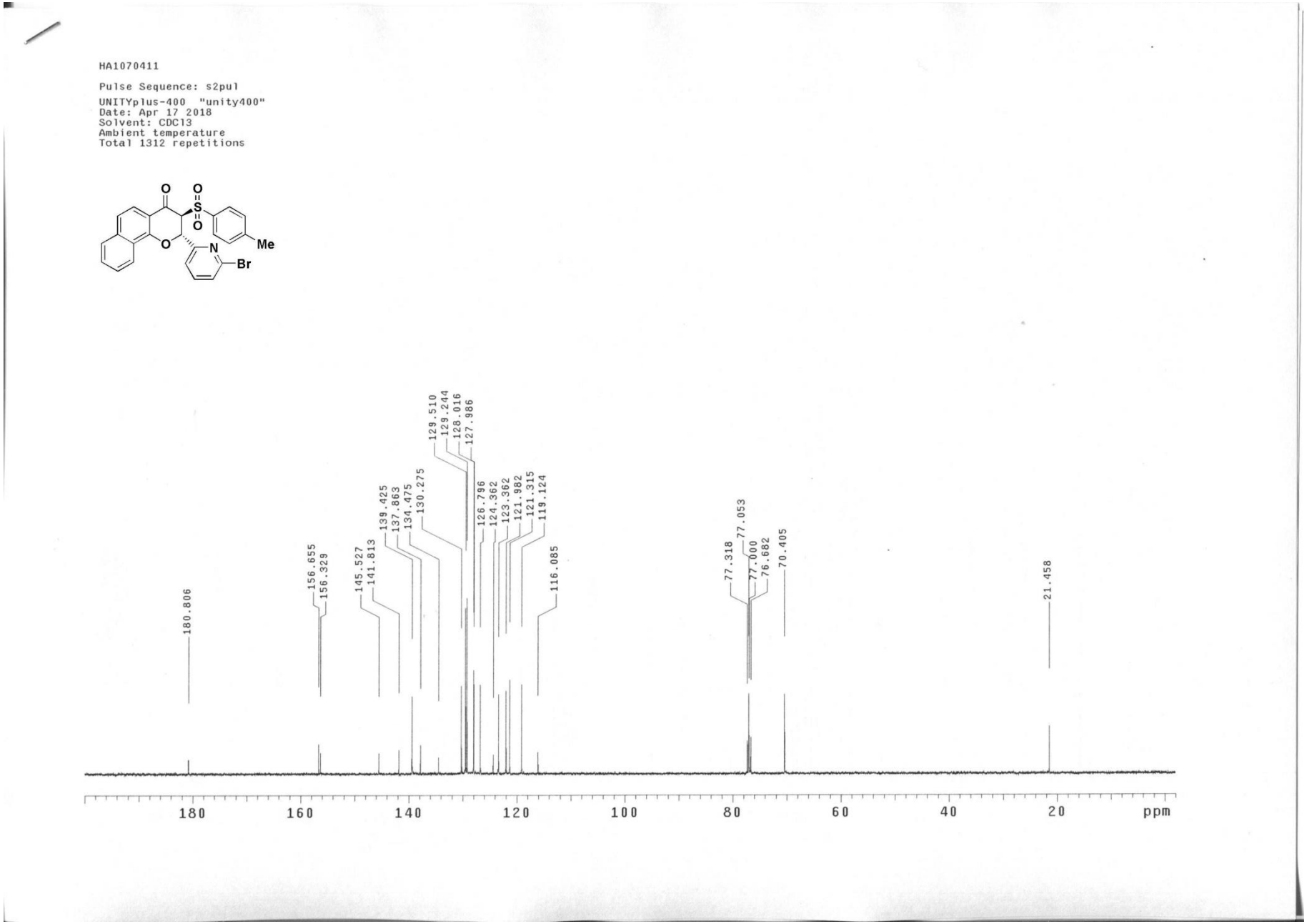

Compound 2g (^{1}H-NMR spectral data)

HA4BrNAPB

Pulse Sequence: s2pul
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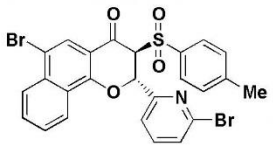
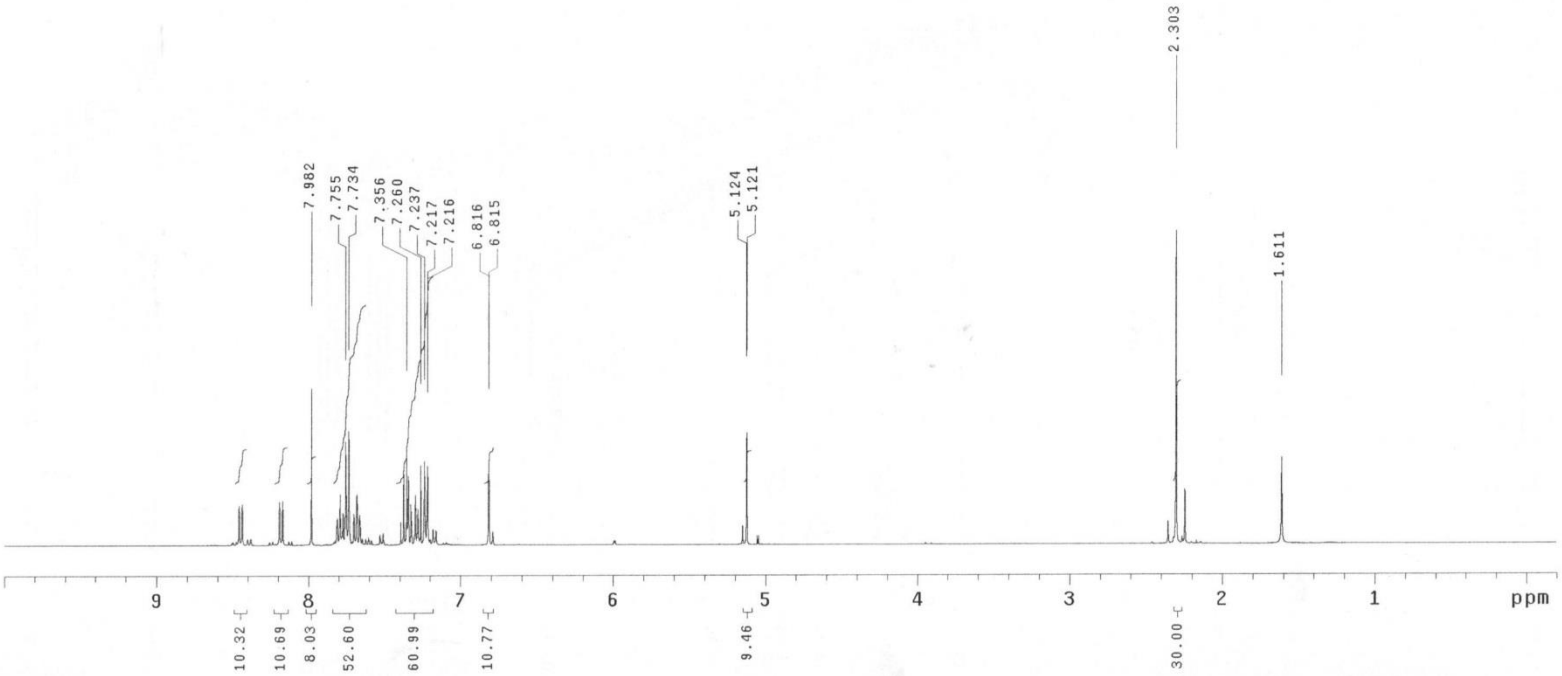
Date: Jan 31 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



trans **: *cis* > 8 : 1**

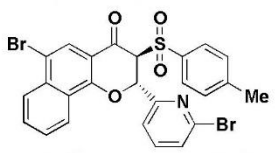


Compound 2g (^{13}C-NMR spectral data)

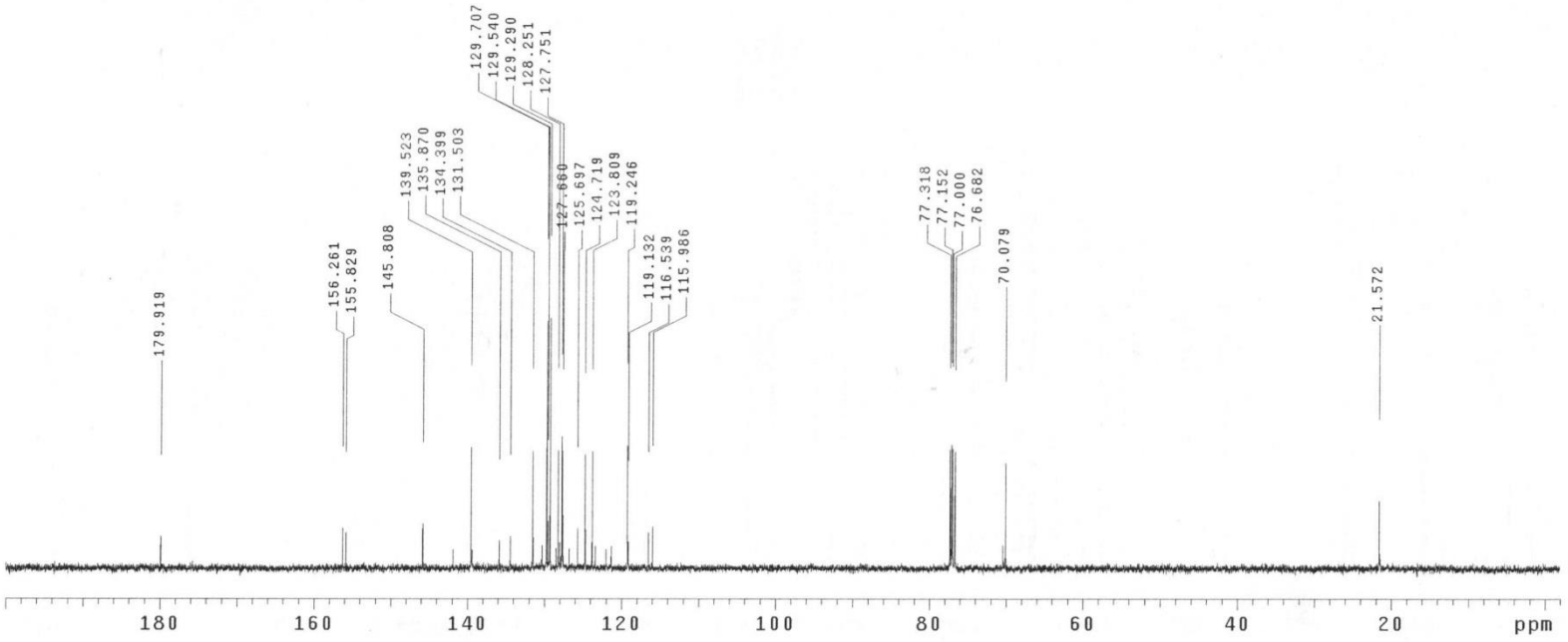
HA4BrNAPB

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jan 31 2019
Solvent: CDCl3
Ambient temperature
Total 1616 repetitions



***trans* : *cis* > 8 : 1**



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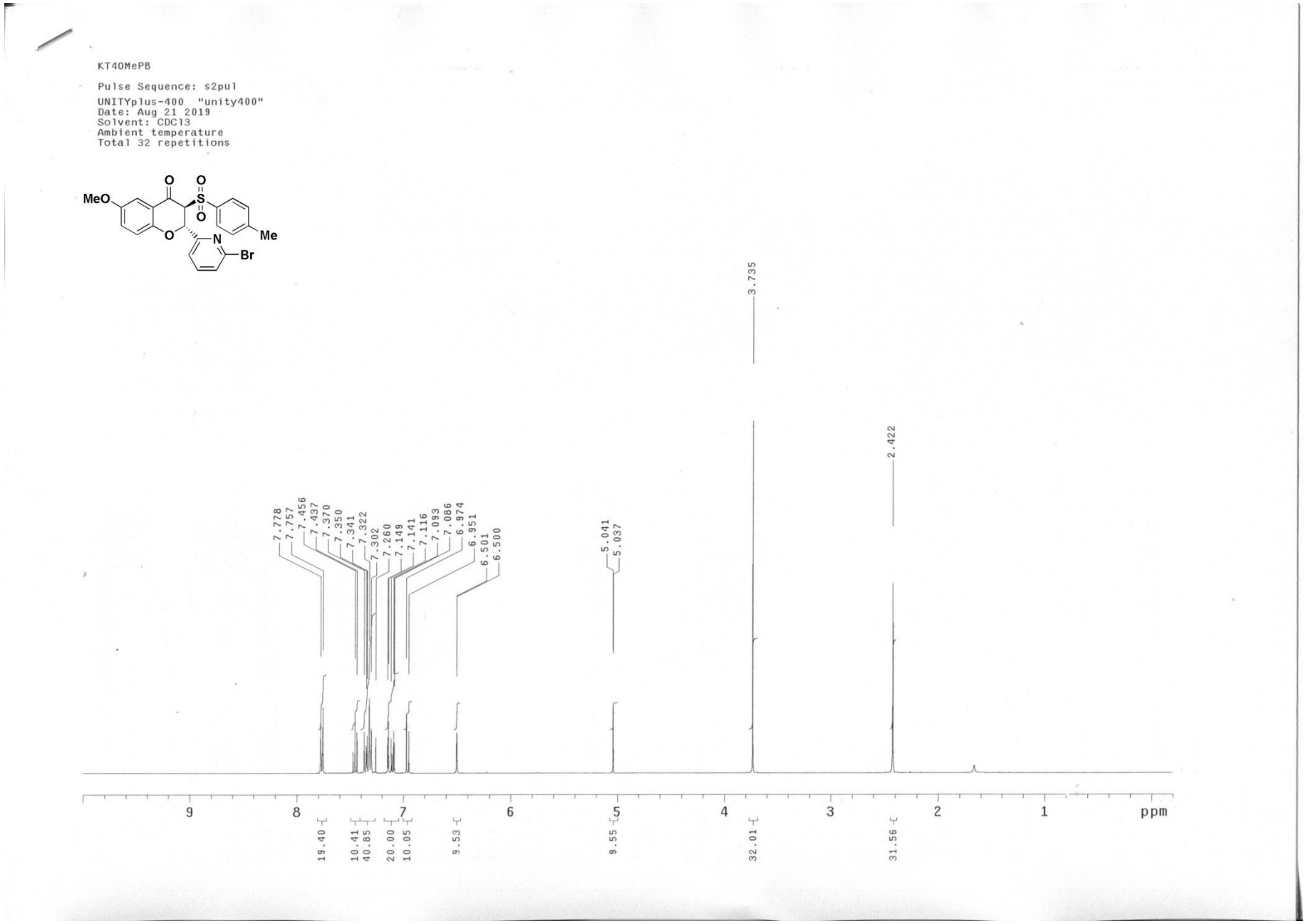

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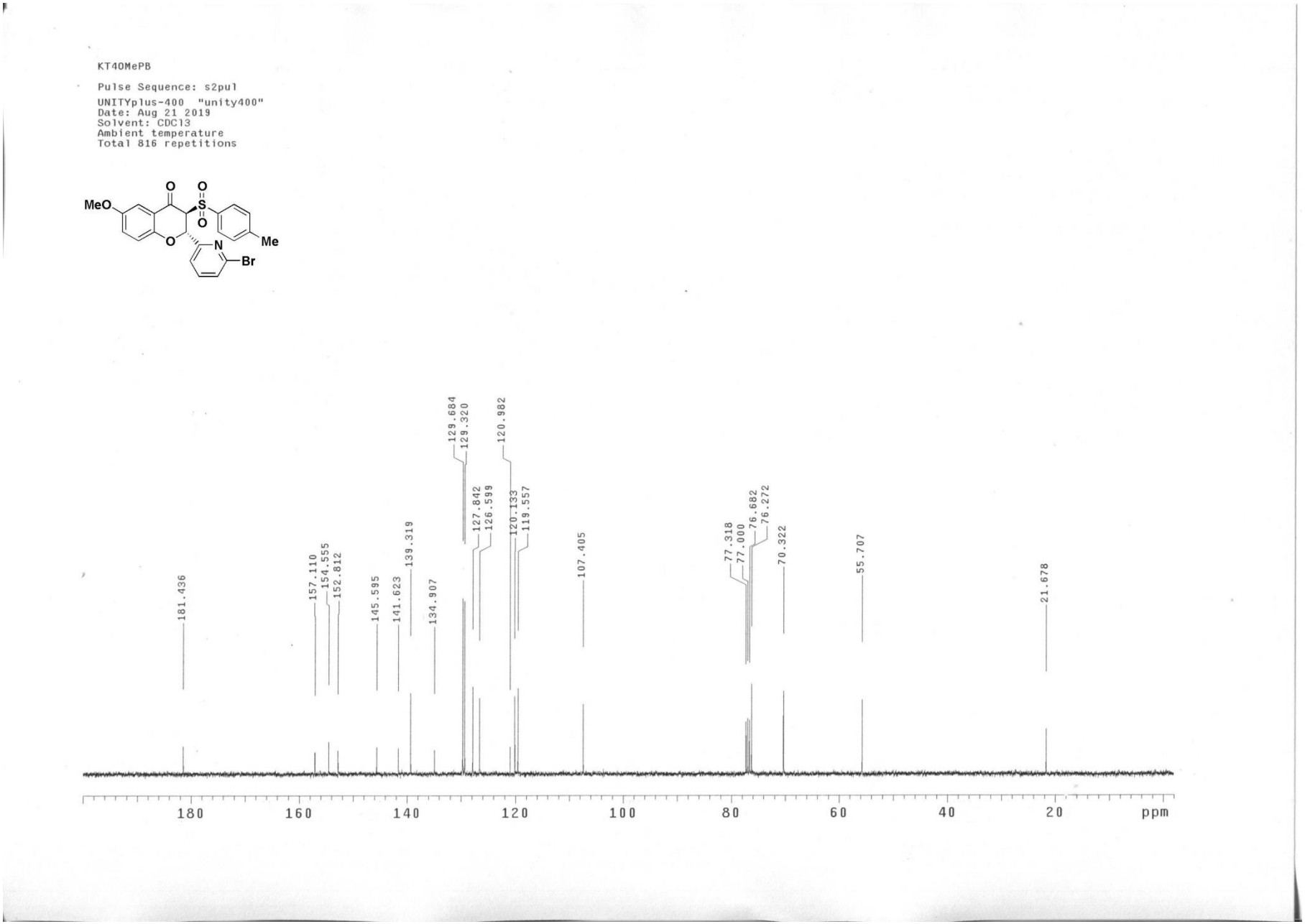

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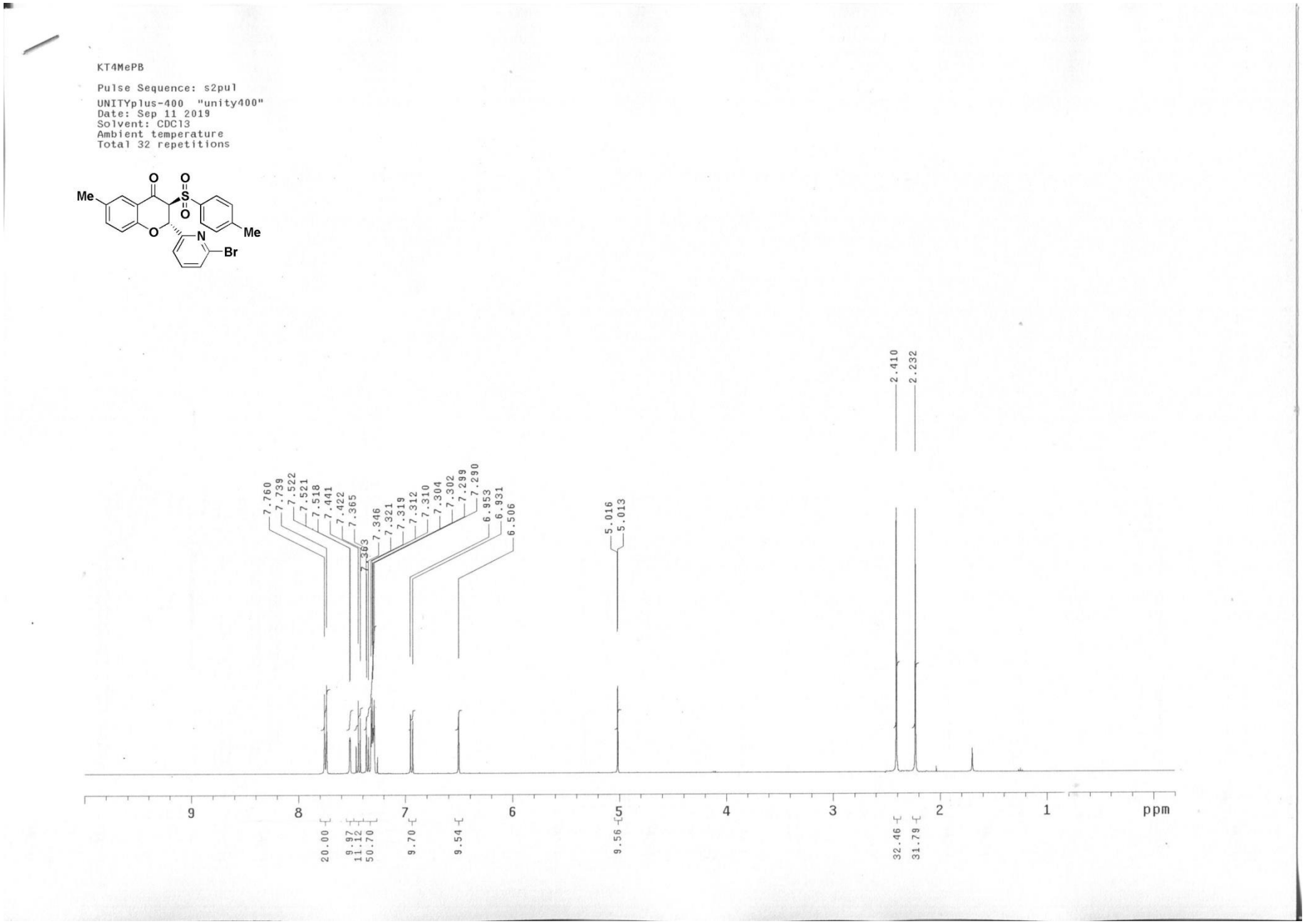
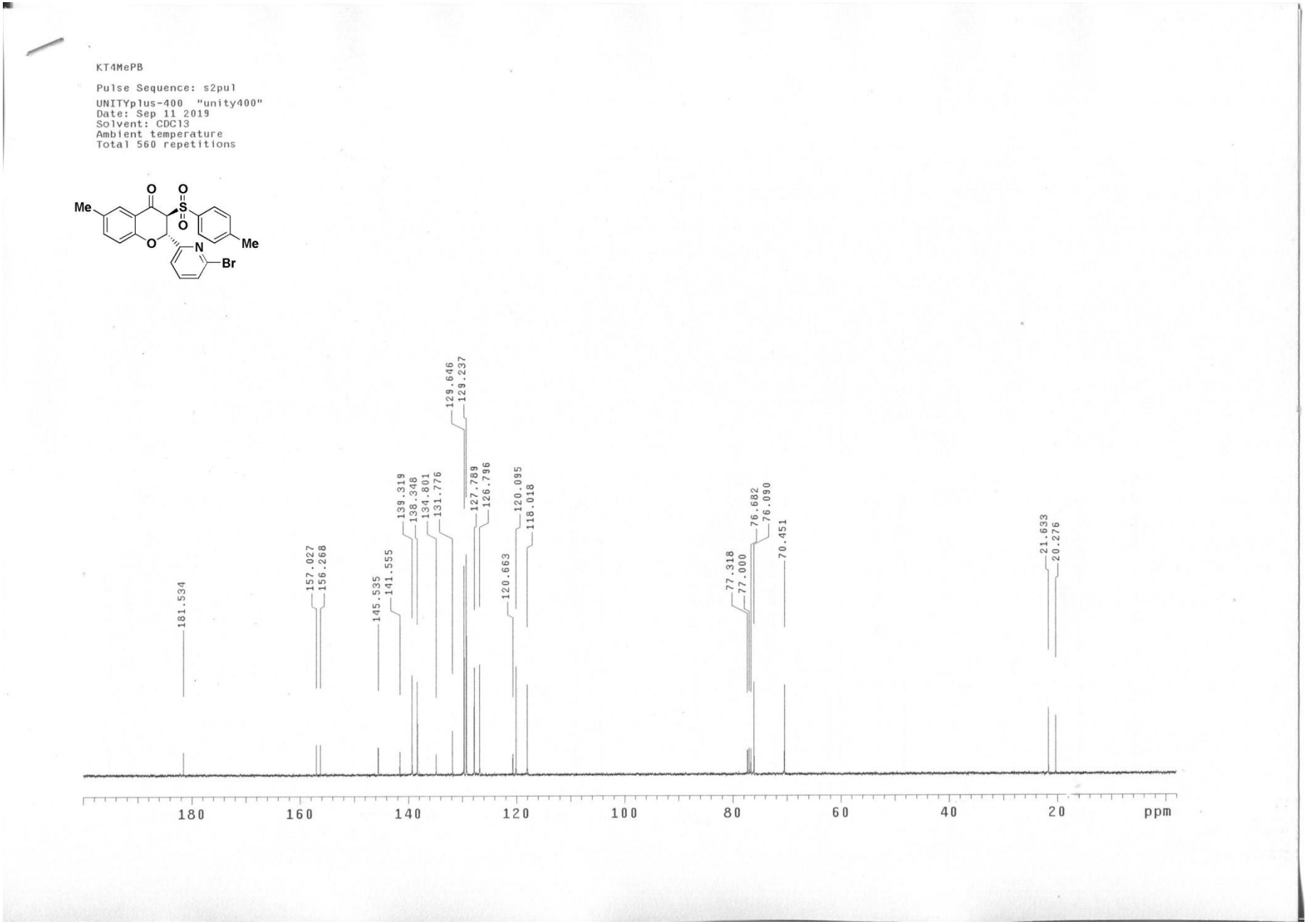
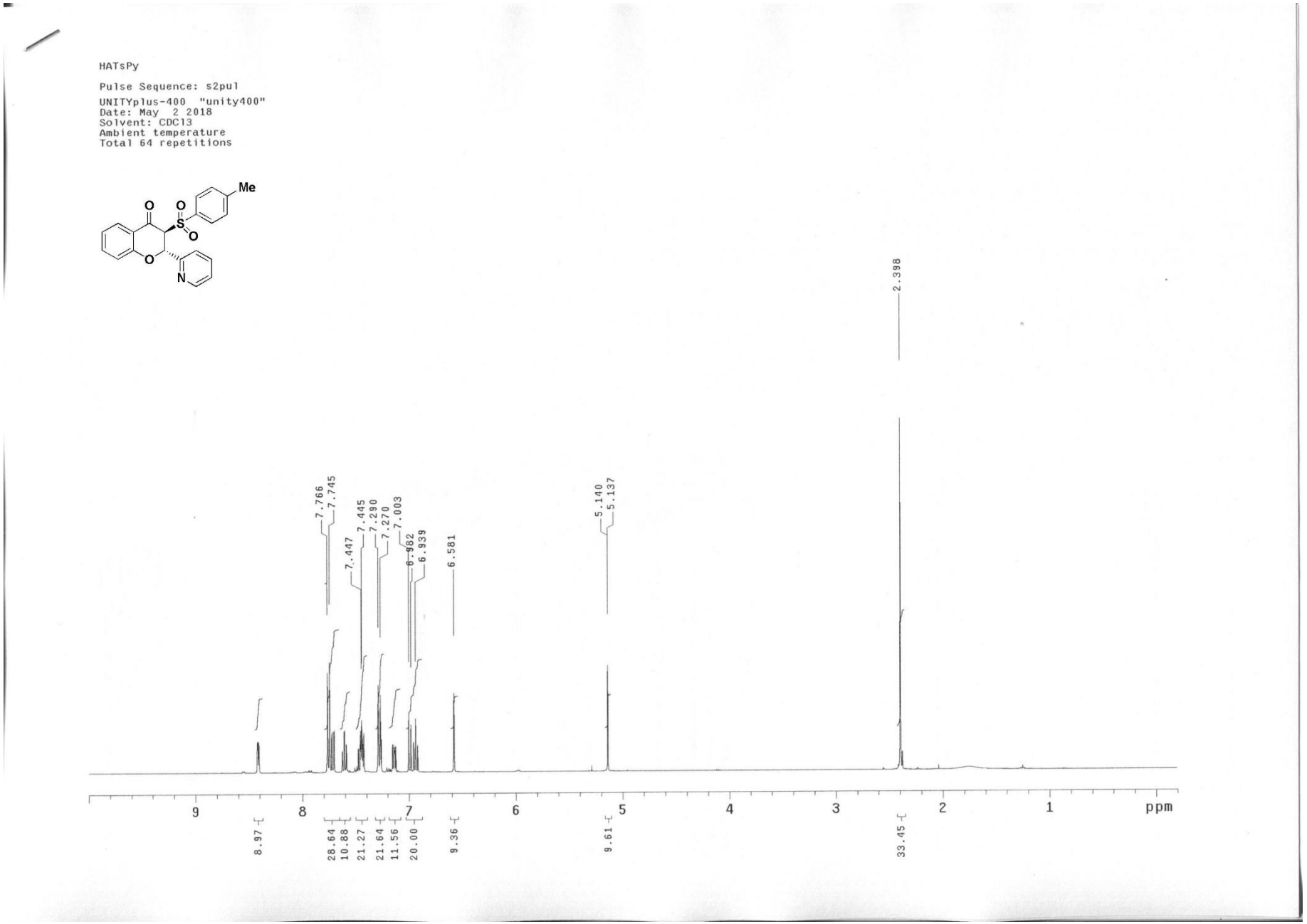


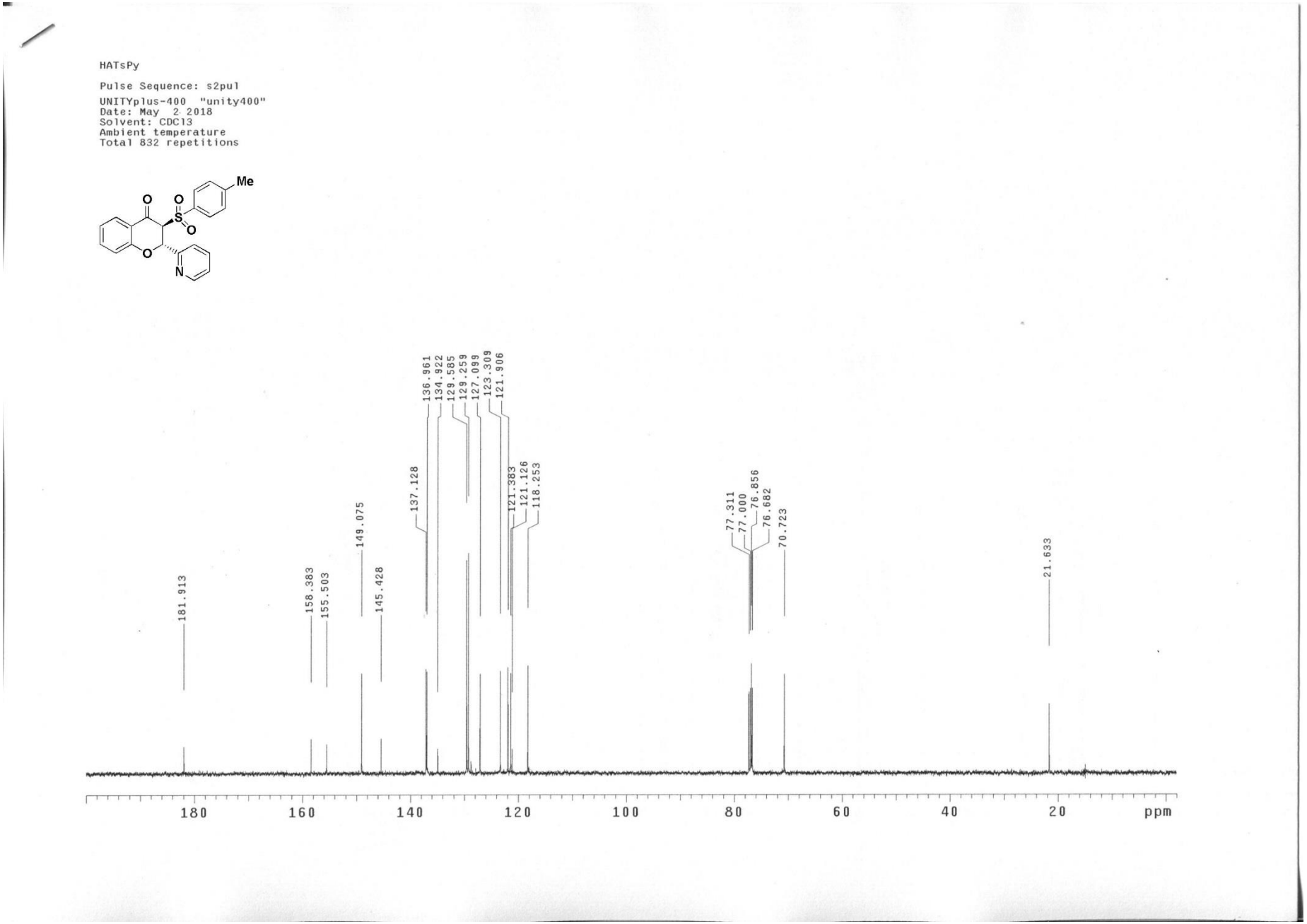
Compound 2i (^{13}C-NMR spectral data)



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



Compound 2j (^{13}C-NMR spectral data)



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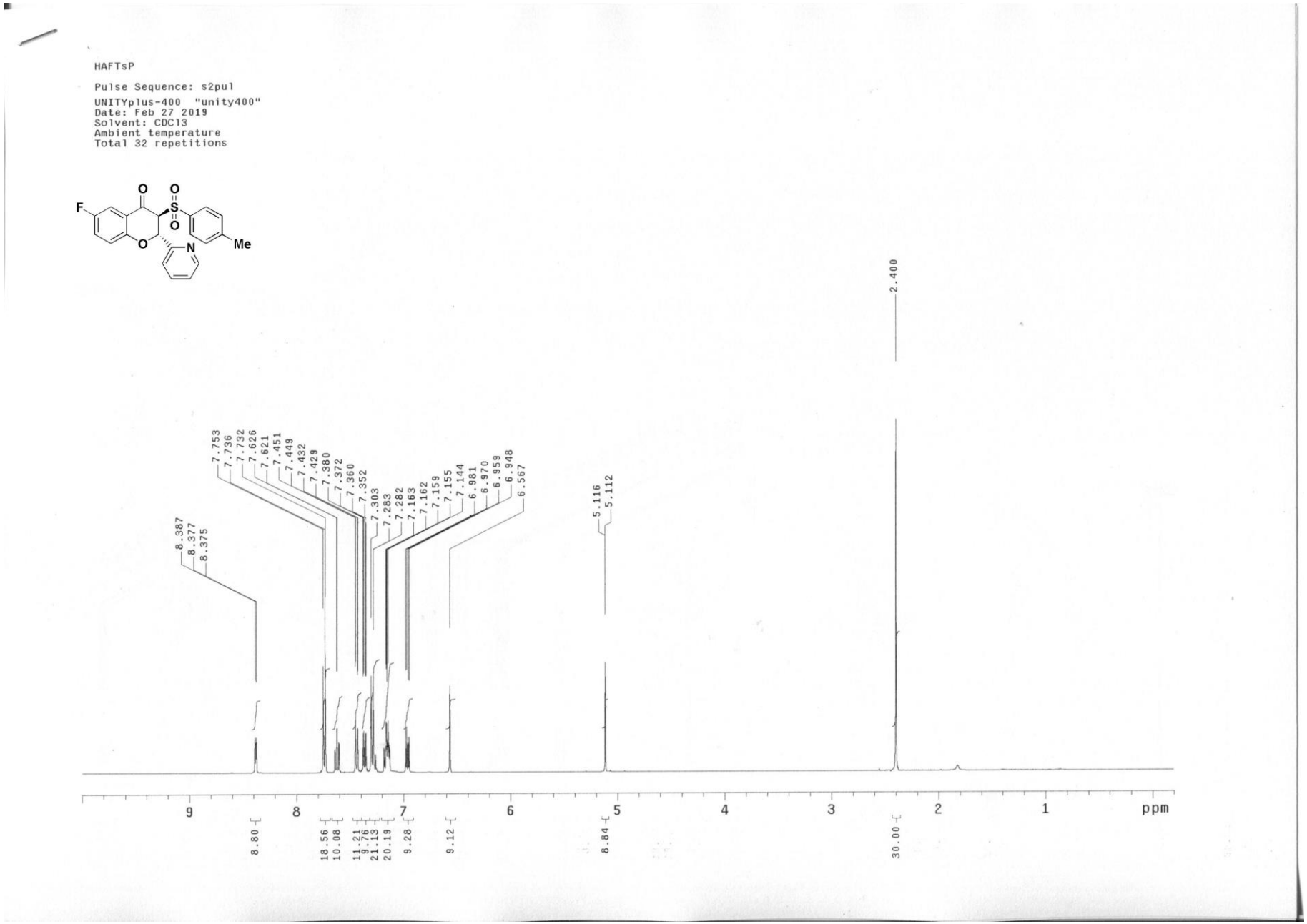
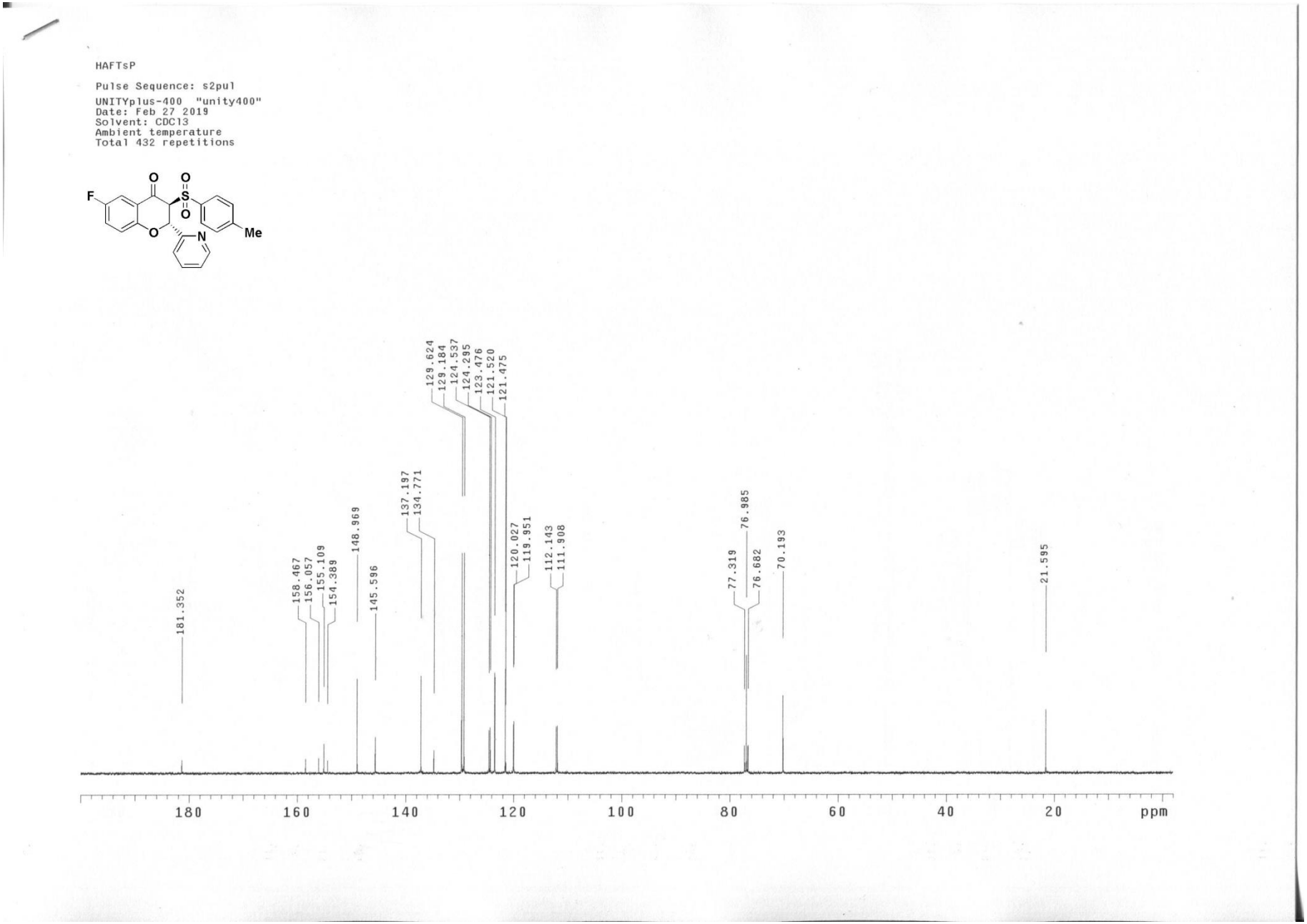


Compound 2k (^{13}C-NMR spectral data)



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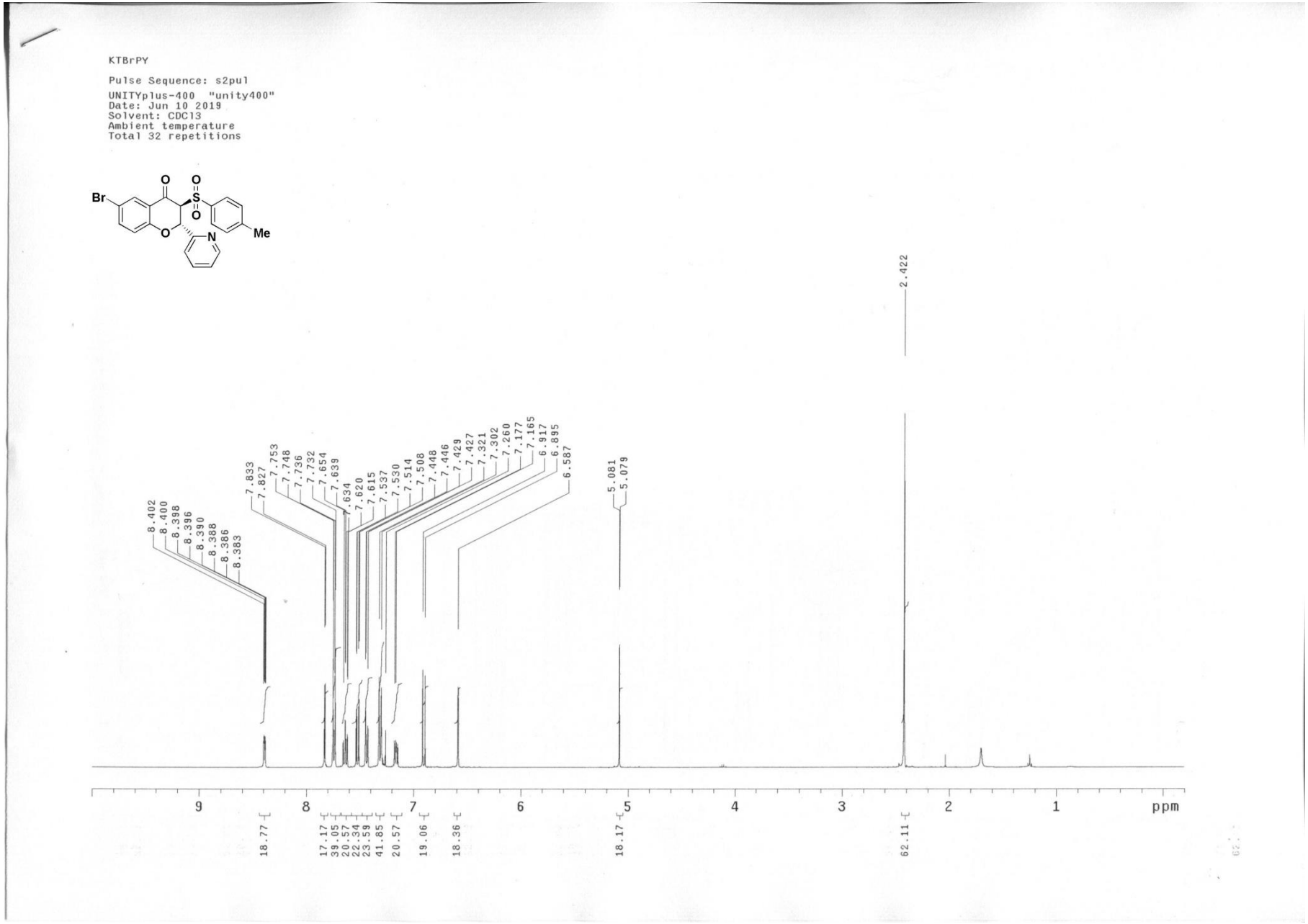
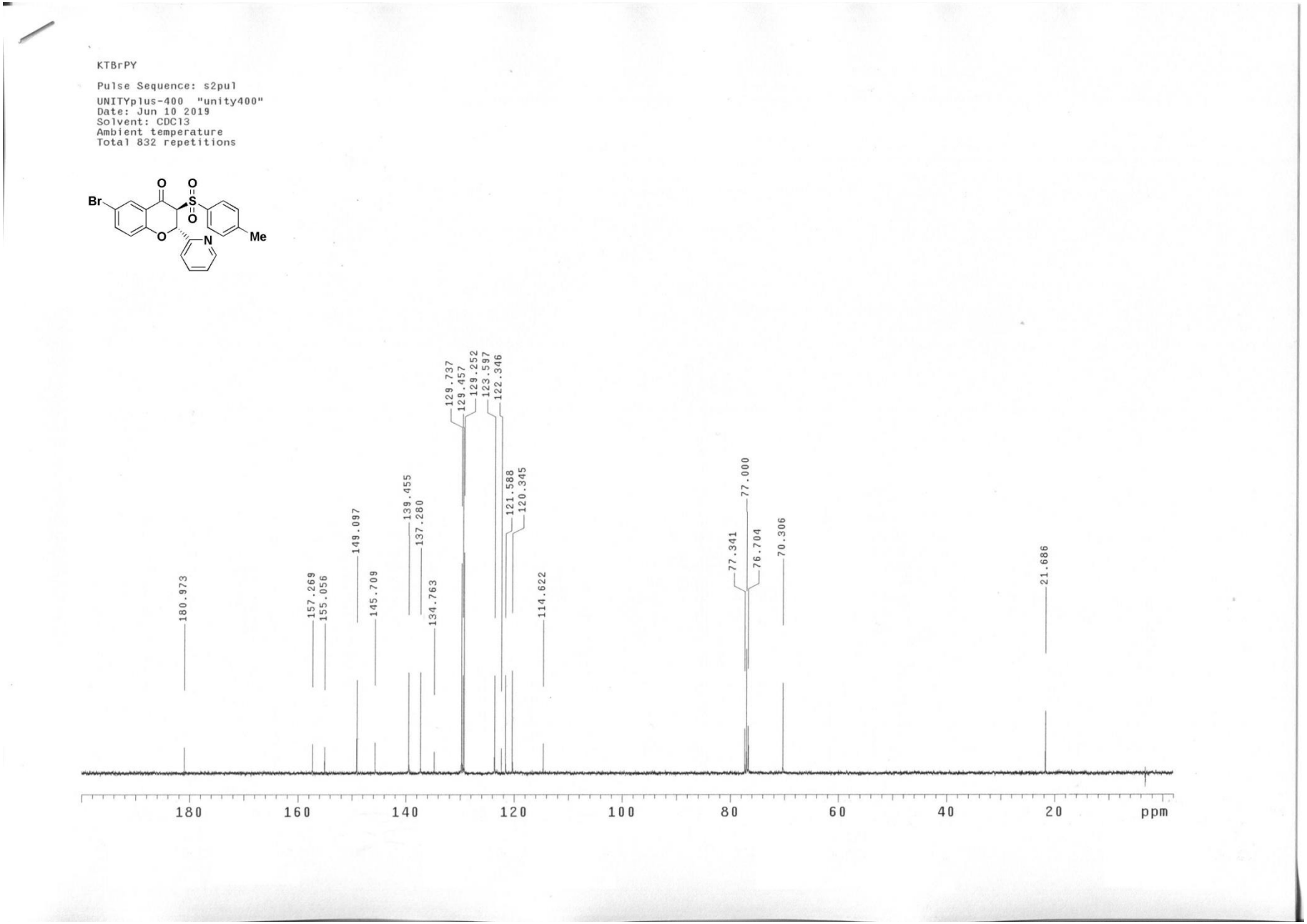


Compound 2l (^{13}C-NMR spectral data)



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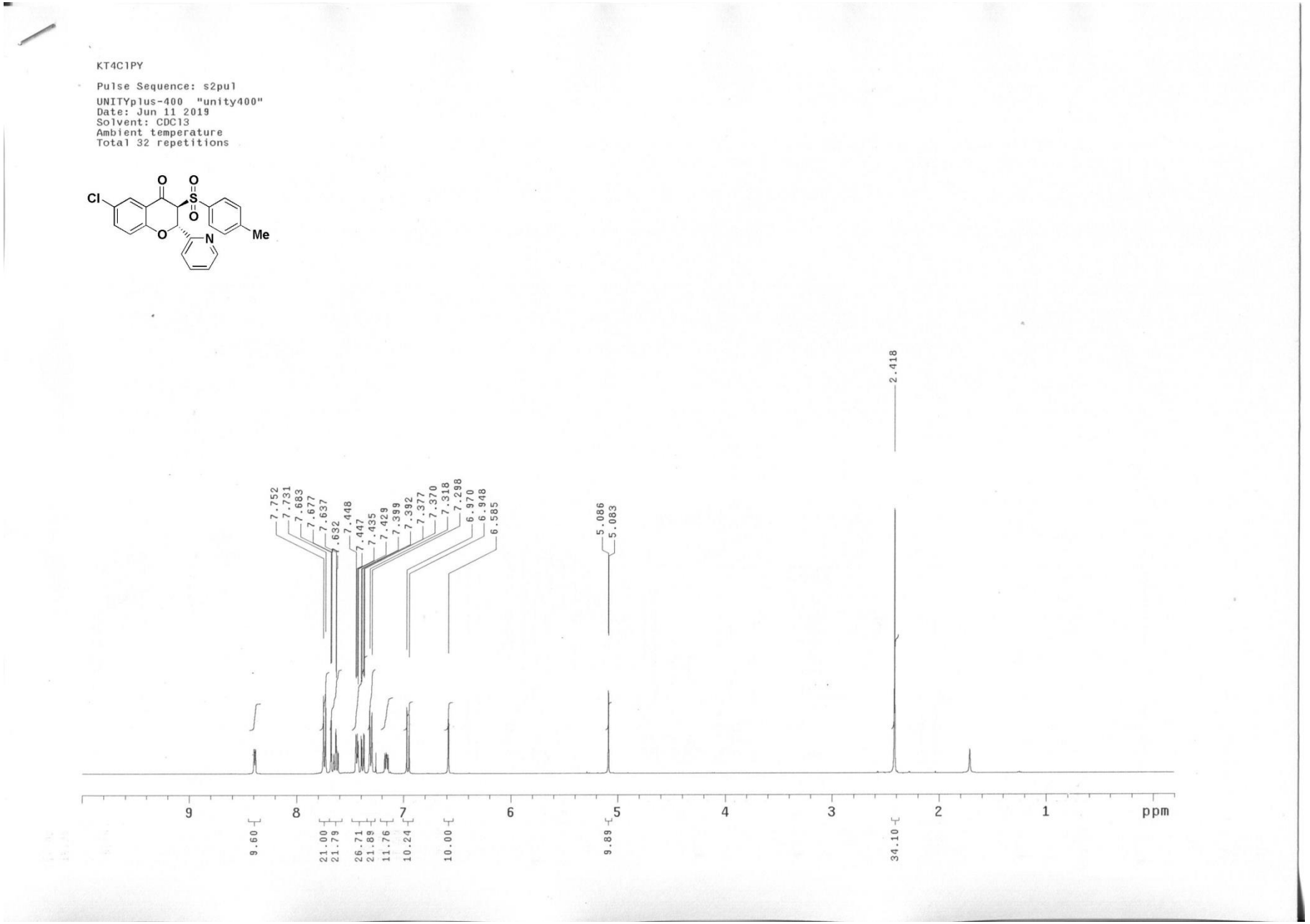
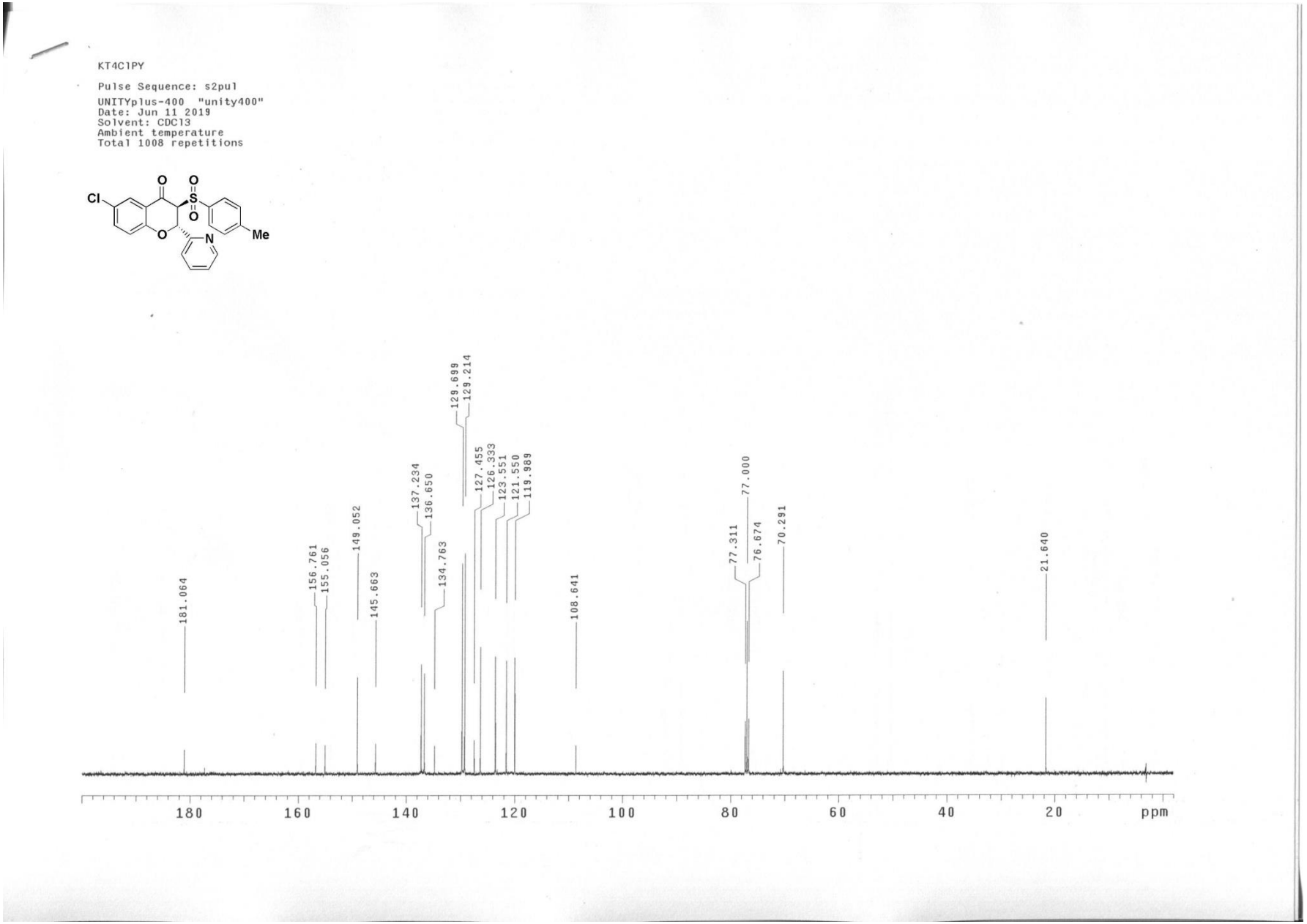
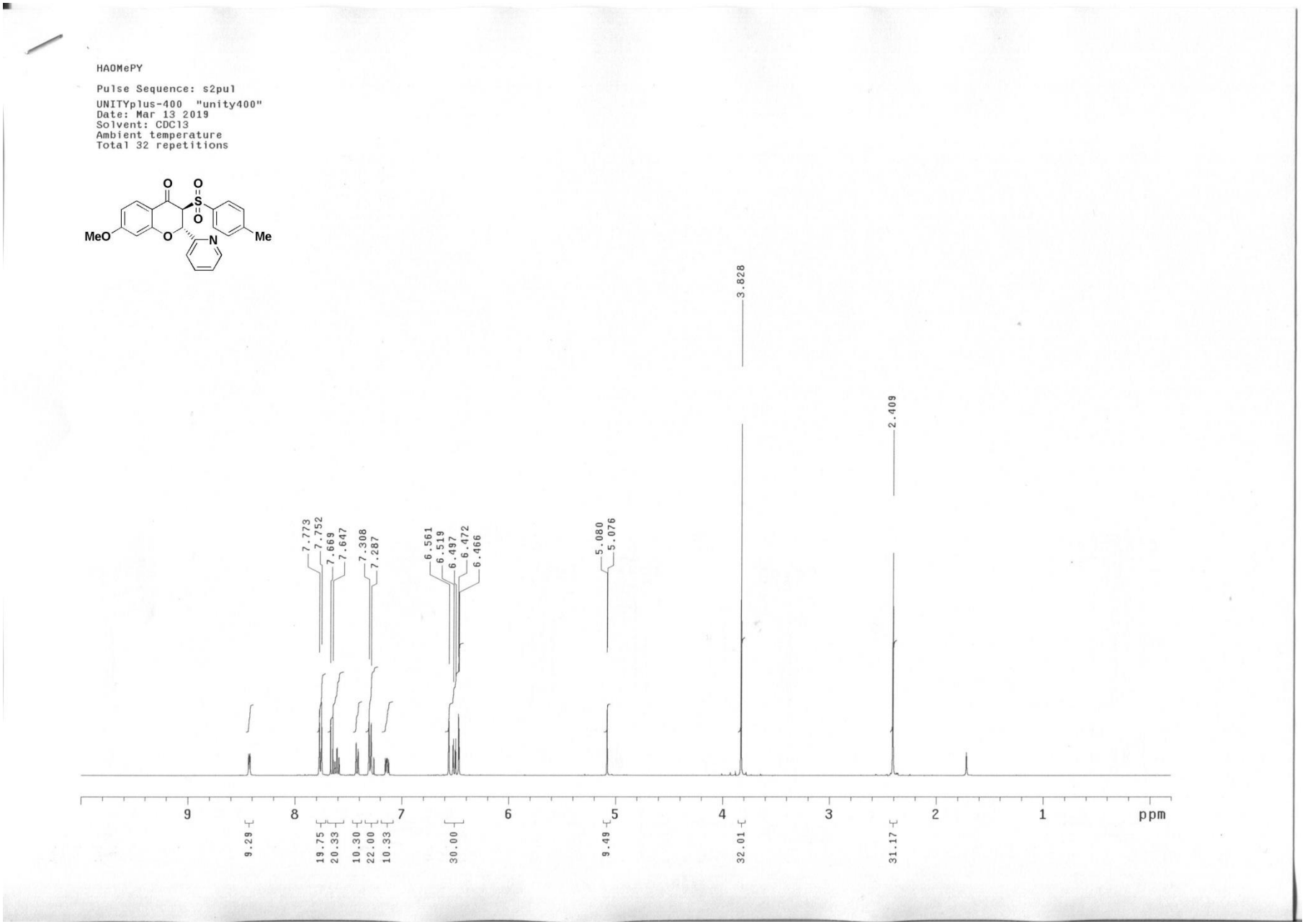


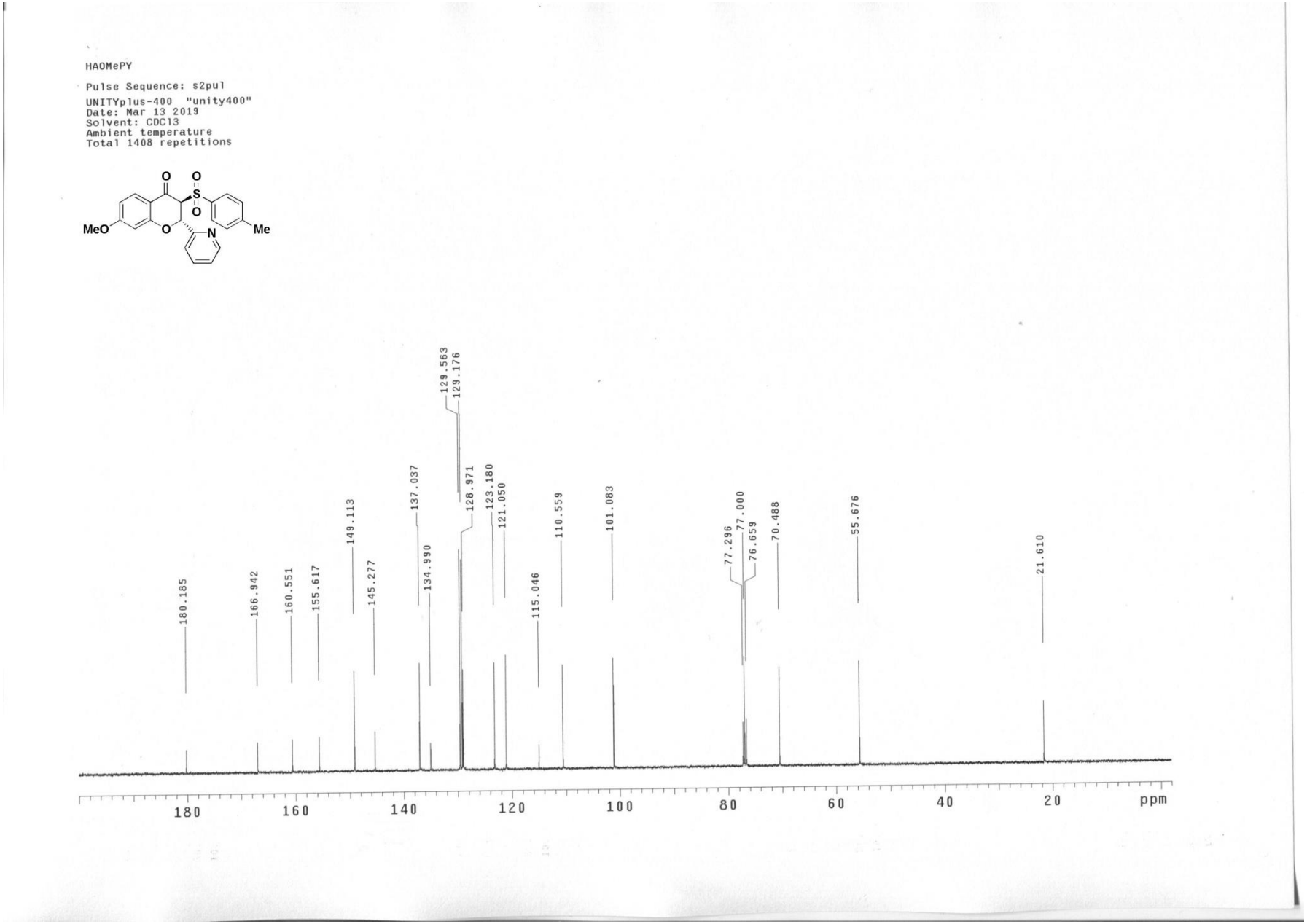
Compound 2m (^{13}C-NMR spectral data)



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



Compound 2n (^{13}C-NMR spectral data)

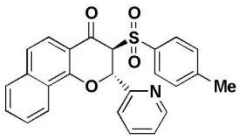


Compound 2o (^{1}H-NMR spectral data)

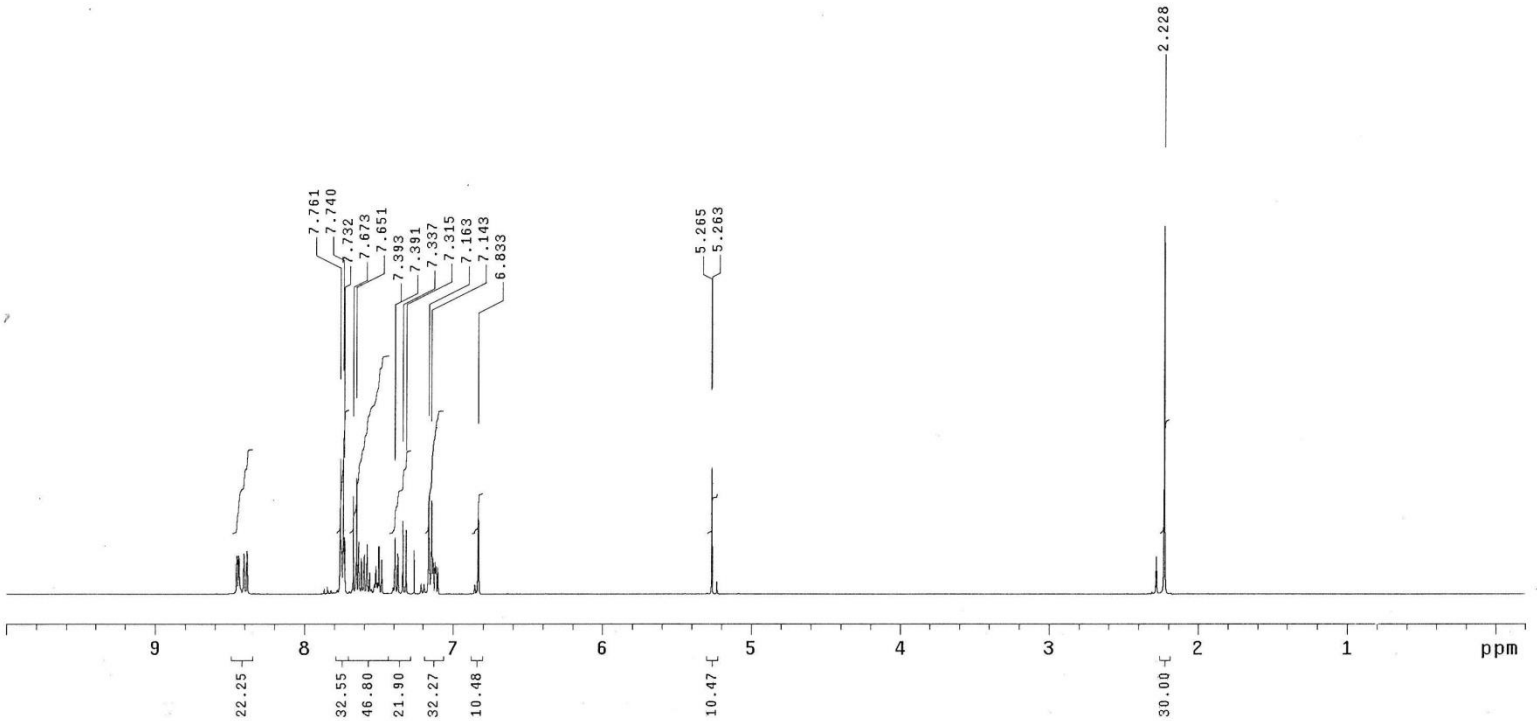
KT1NAPY

Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 5 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions



trans : *cis* > 10 : 1



Compound 2o (^{13}C-NMR spectral data)

KT1NAPY

Pulse Sequence: s2pul

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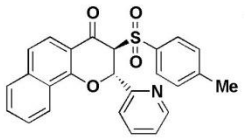
Date: Aug 5 2019
Solvent: CDCl3
Ambient temperature
Total 1296 repetitions



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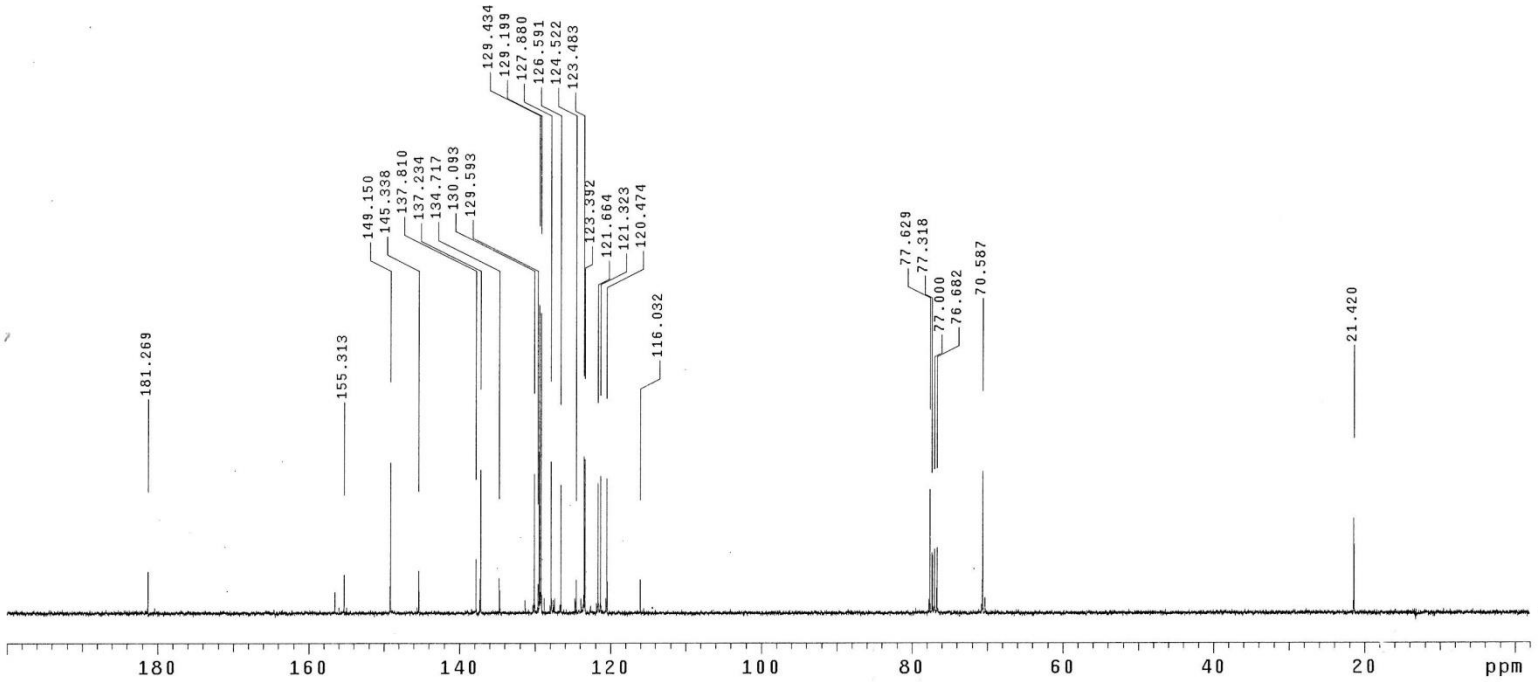

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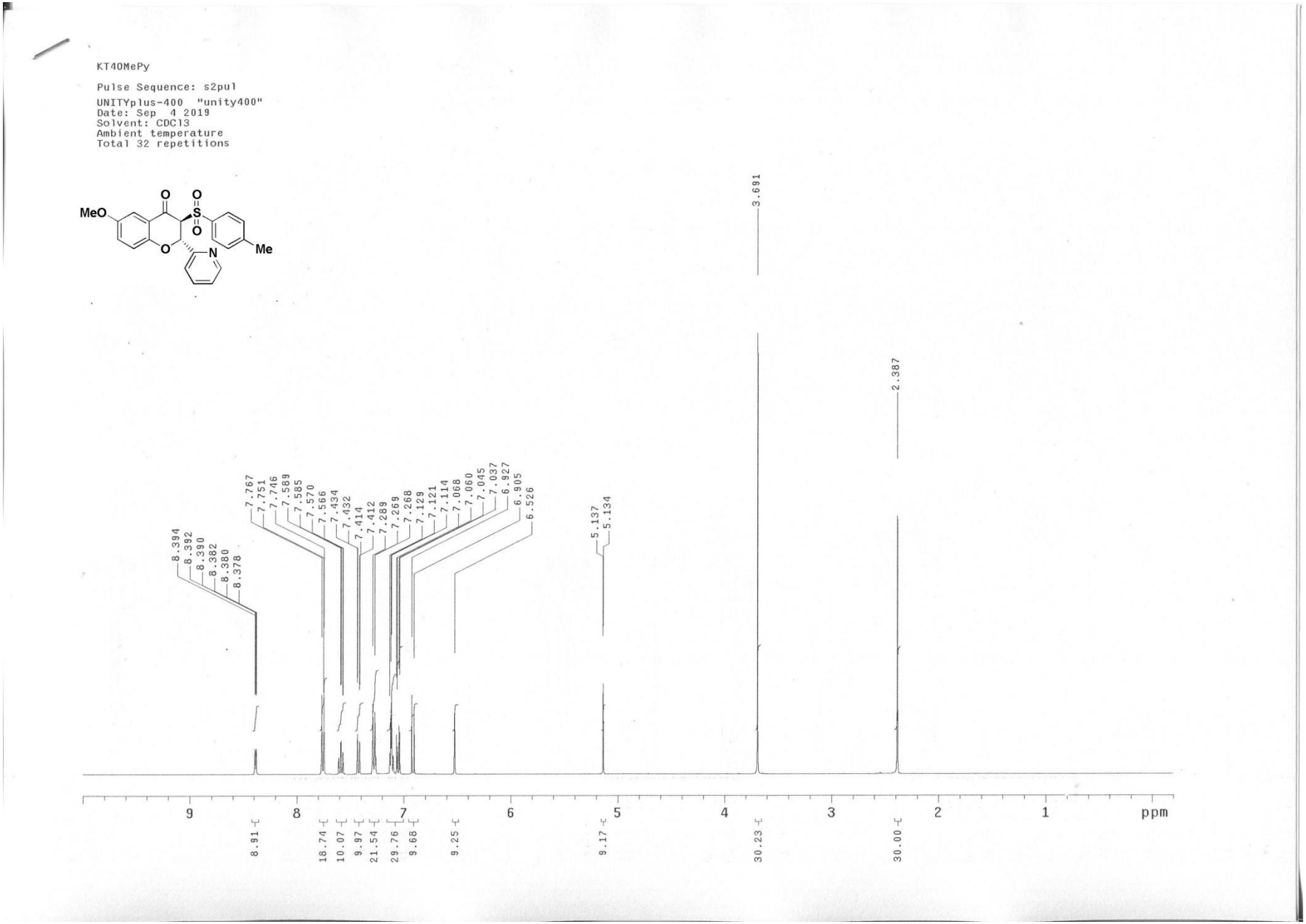
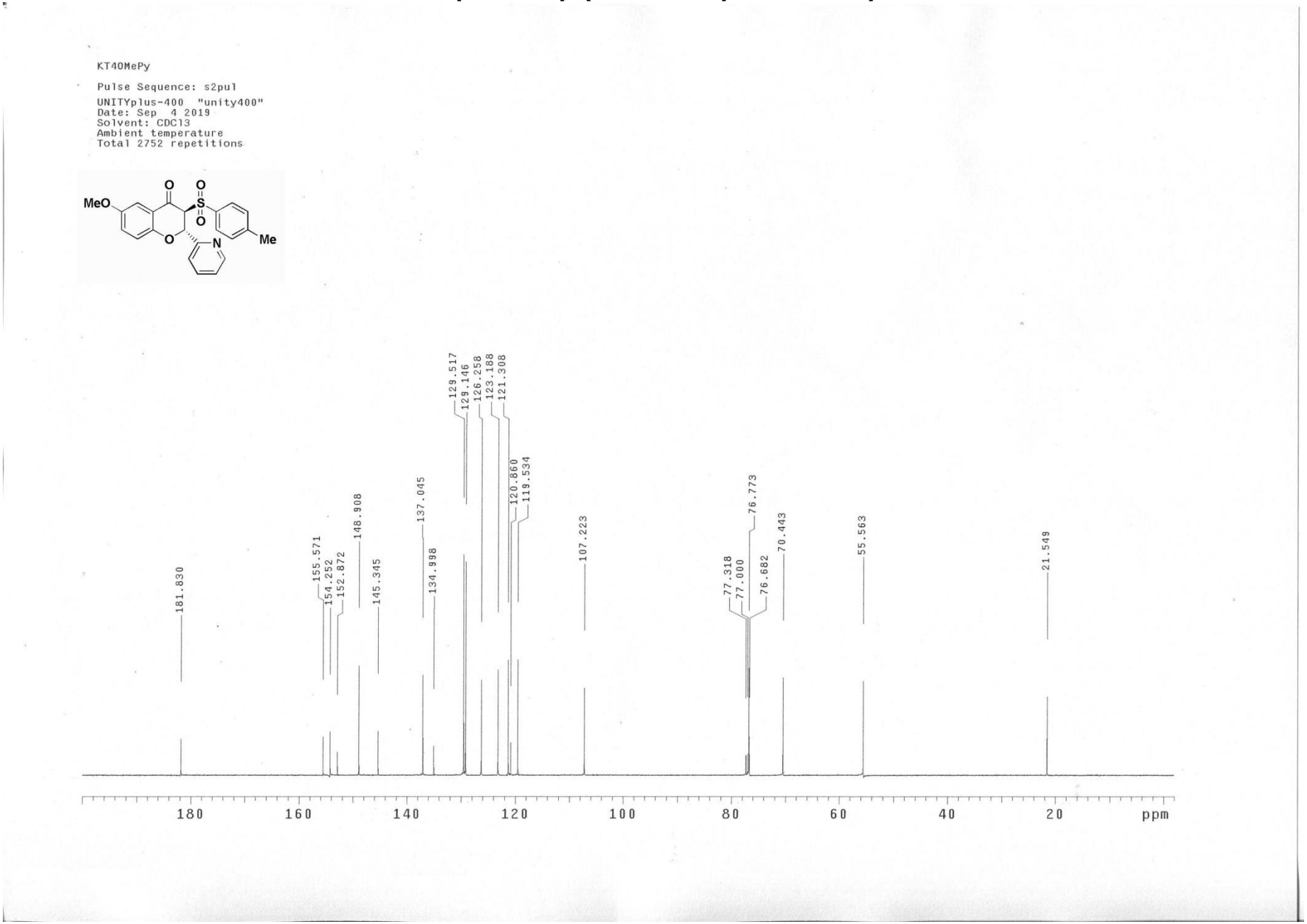


Compound 2p (^{13}C-NMR spectral data)

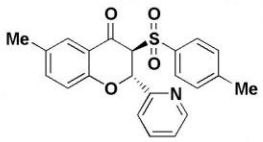


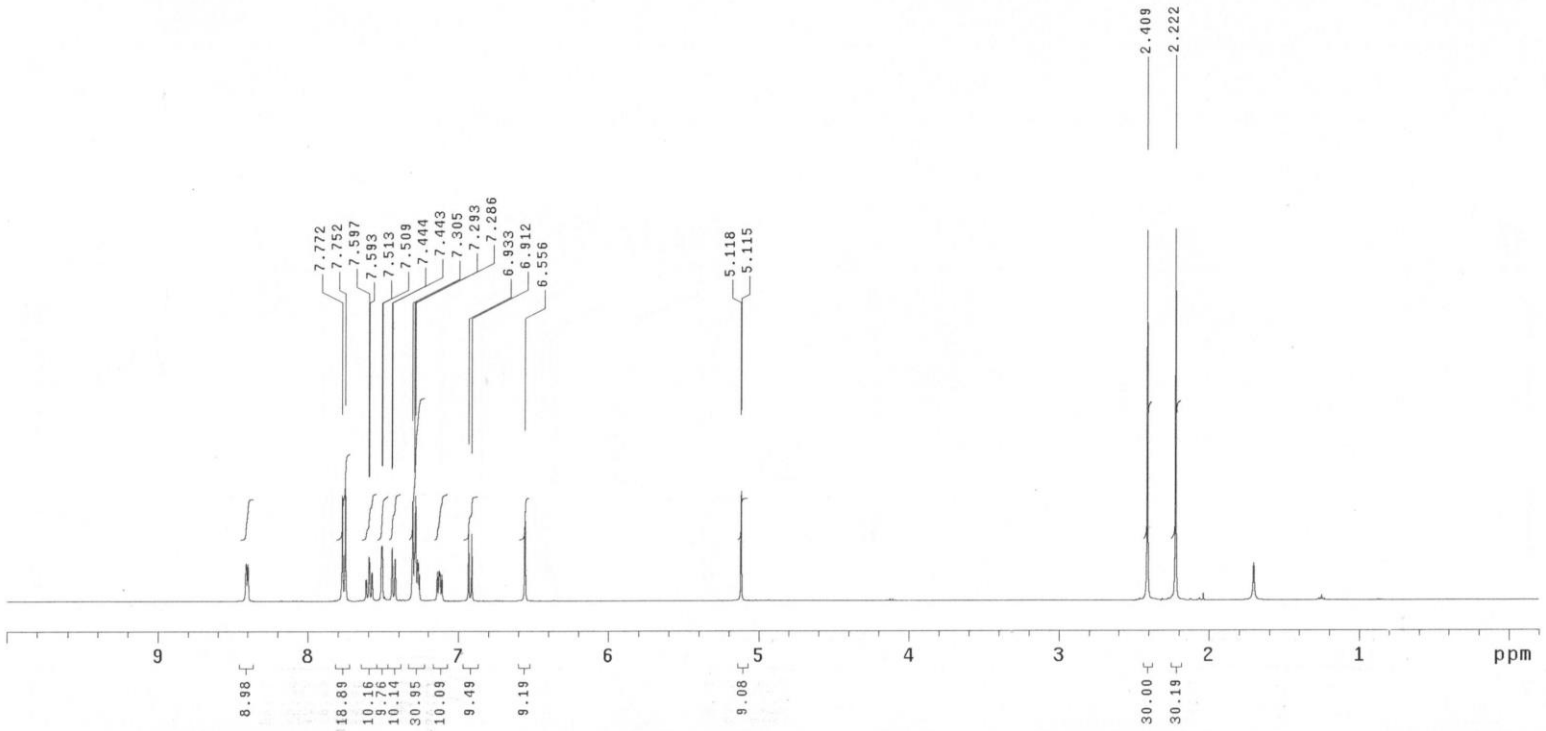
Compound 2q (^{1}H-NMR spectral data)

KT4MePY

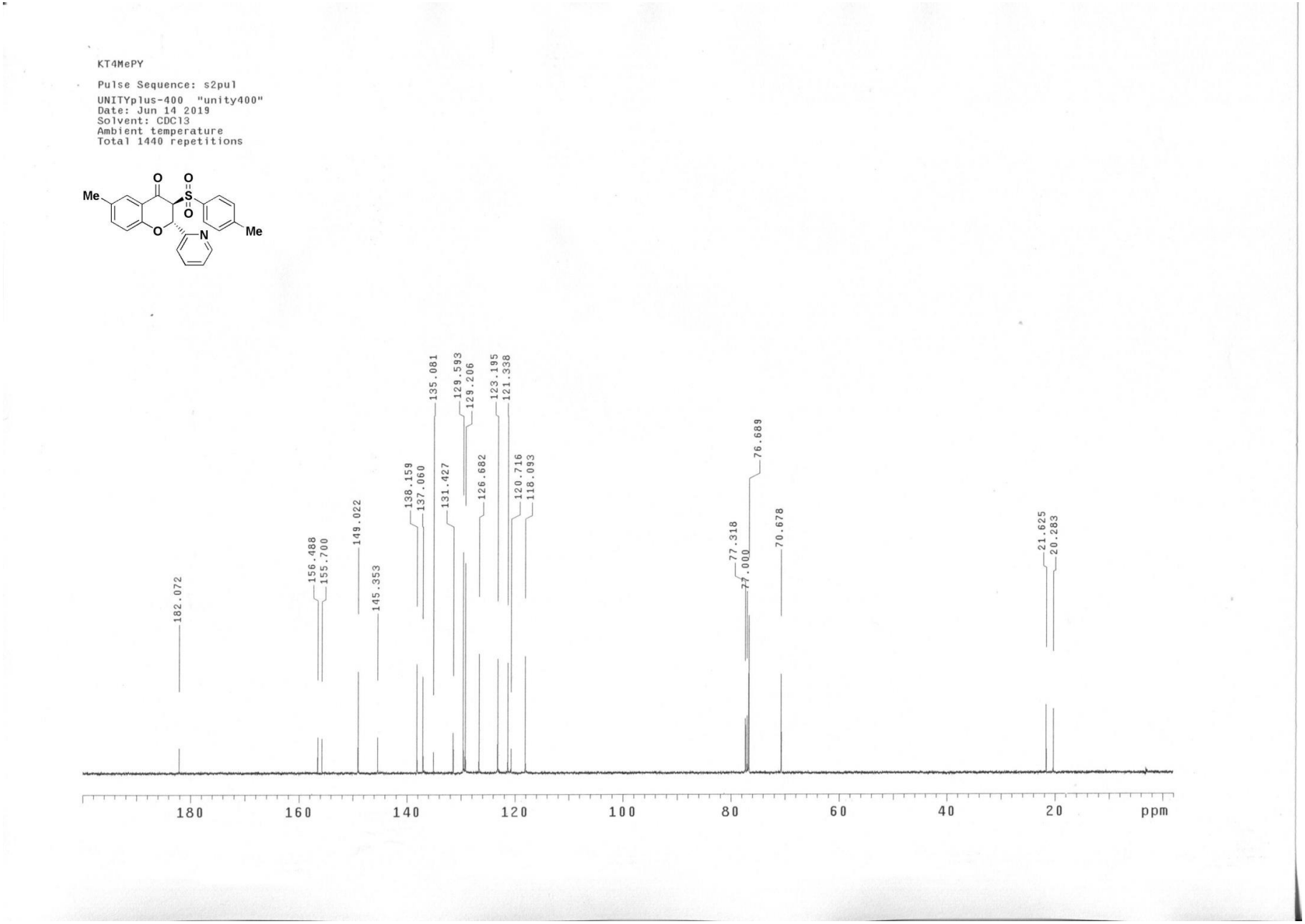
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jun 14 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions





Compound 2q (^{13}C-NMR spectral data)



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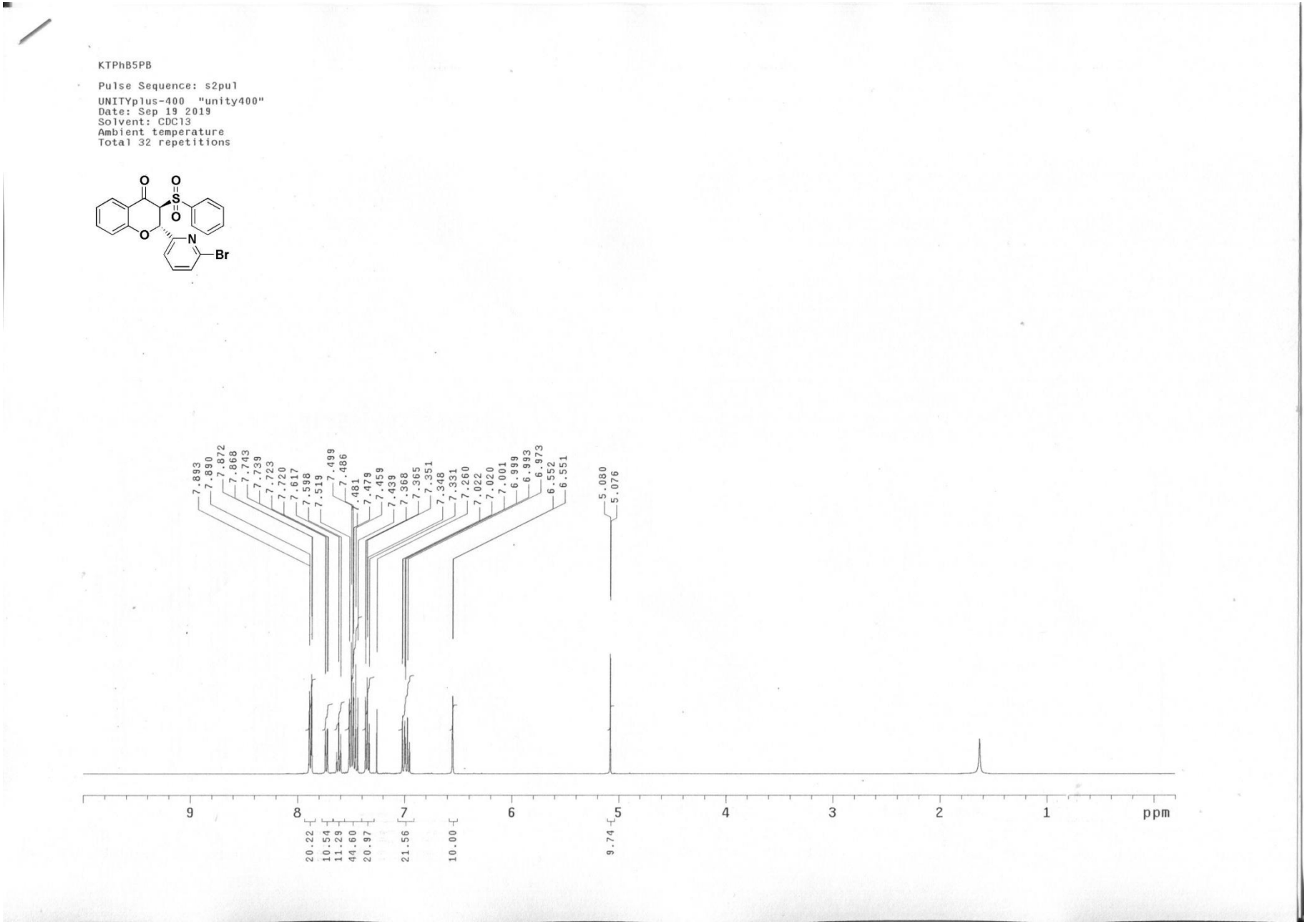

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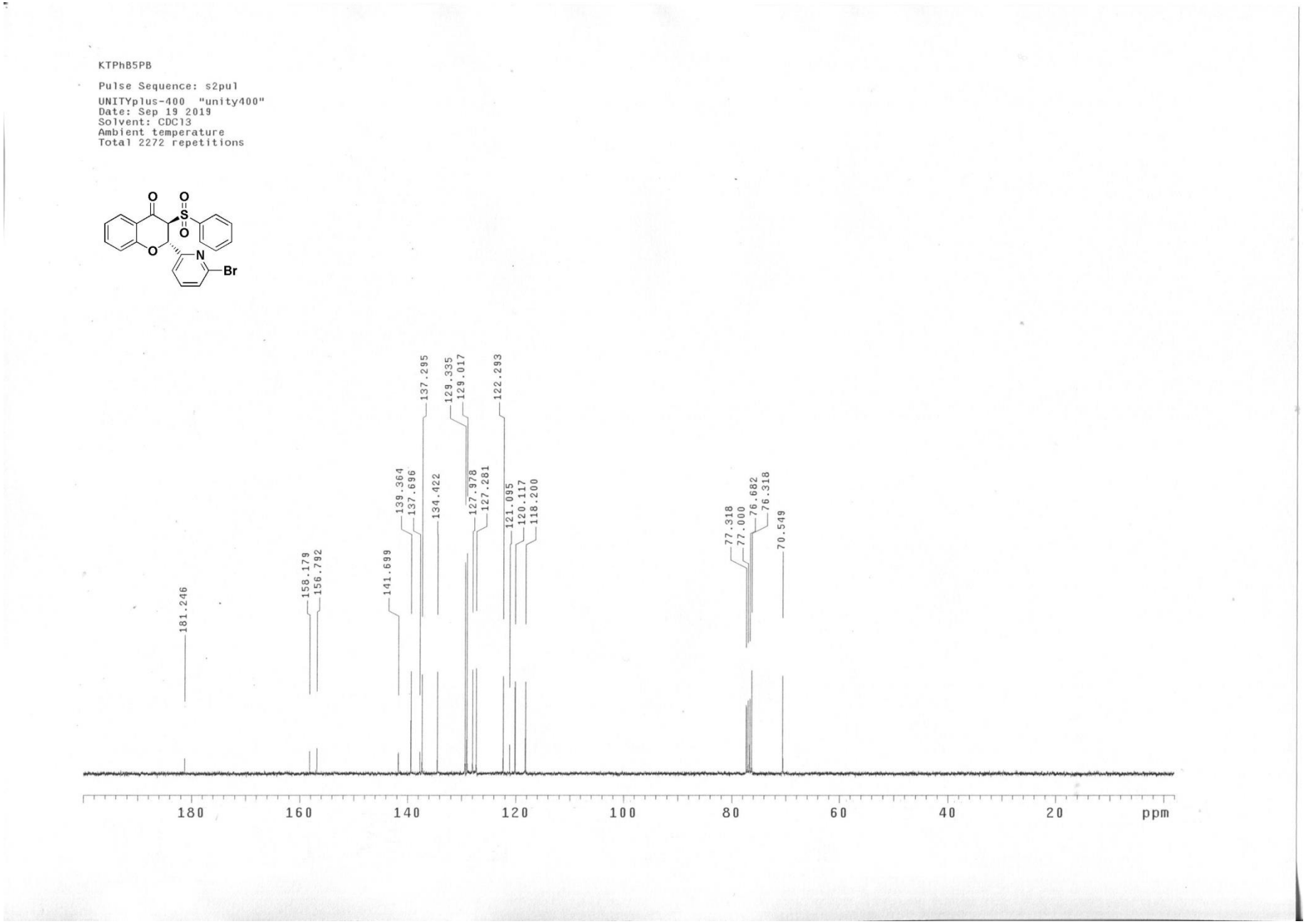

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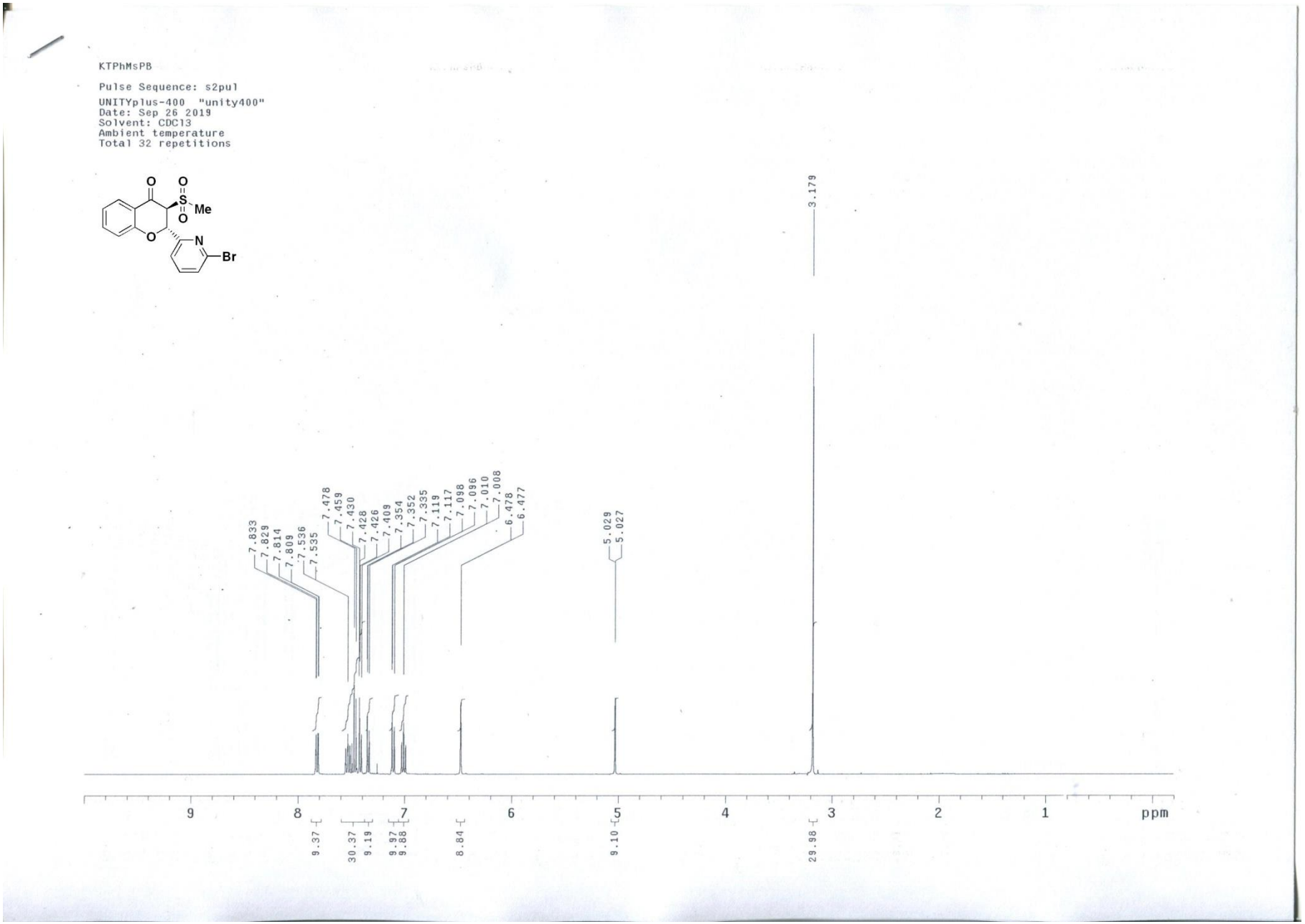
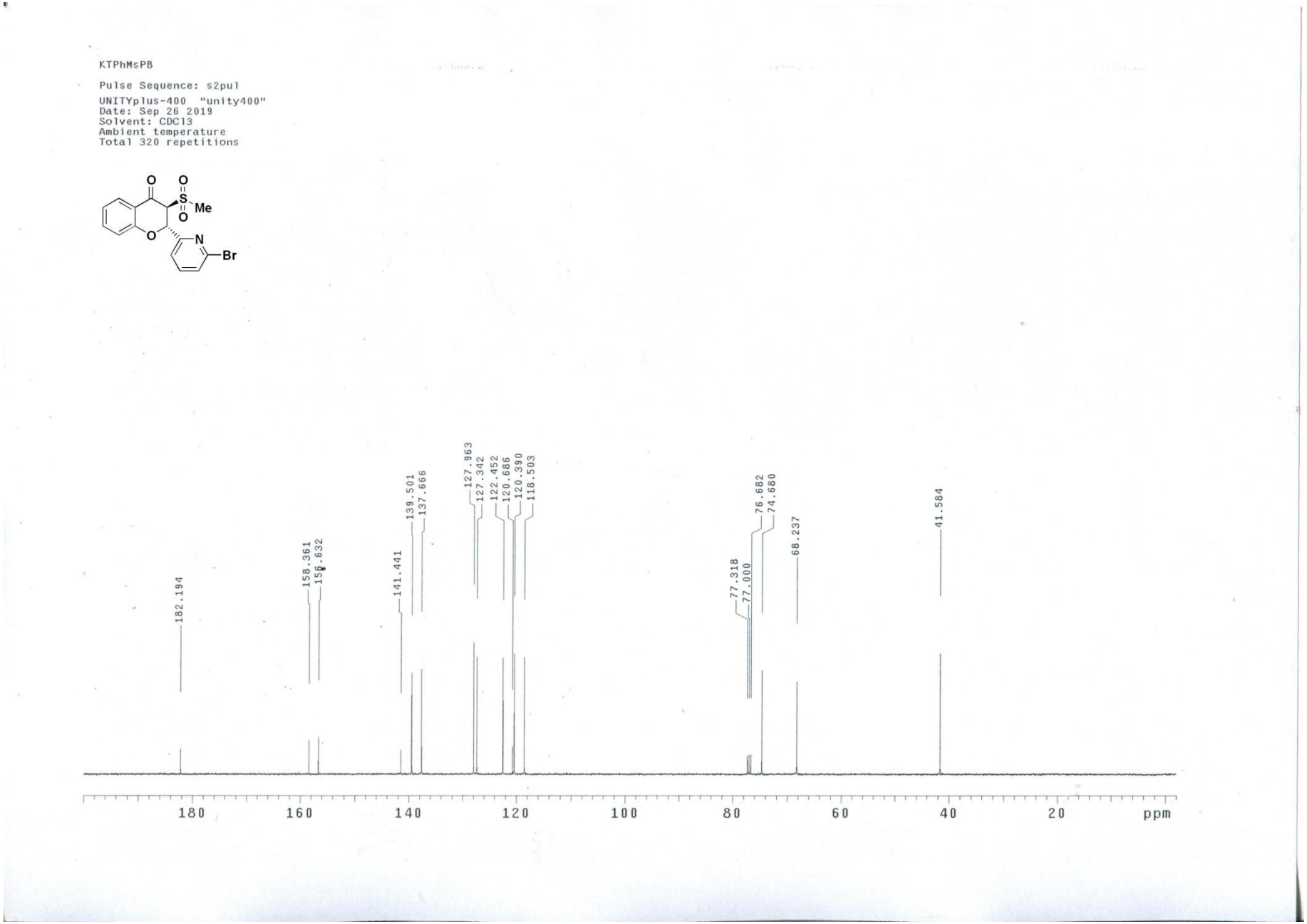
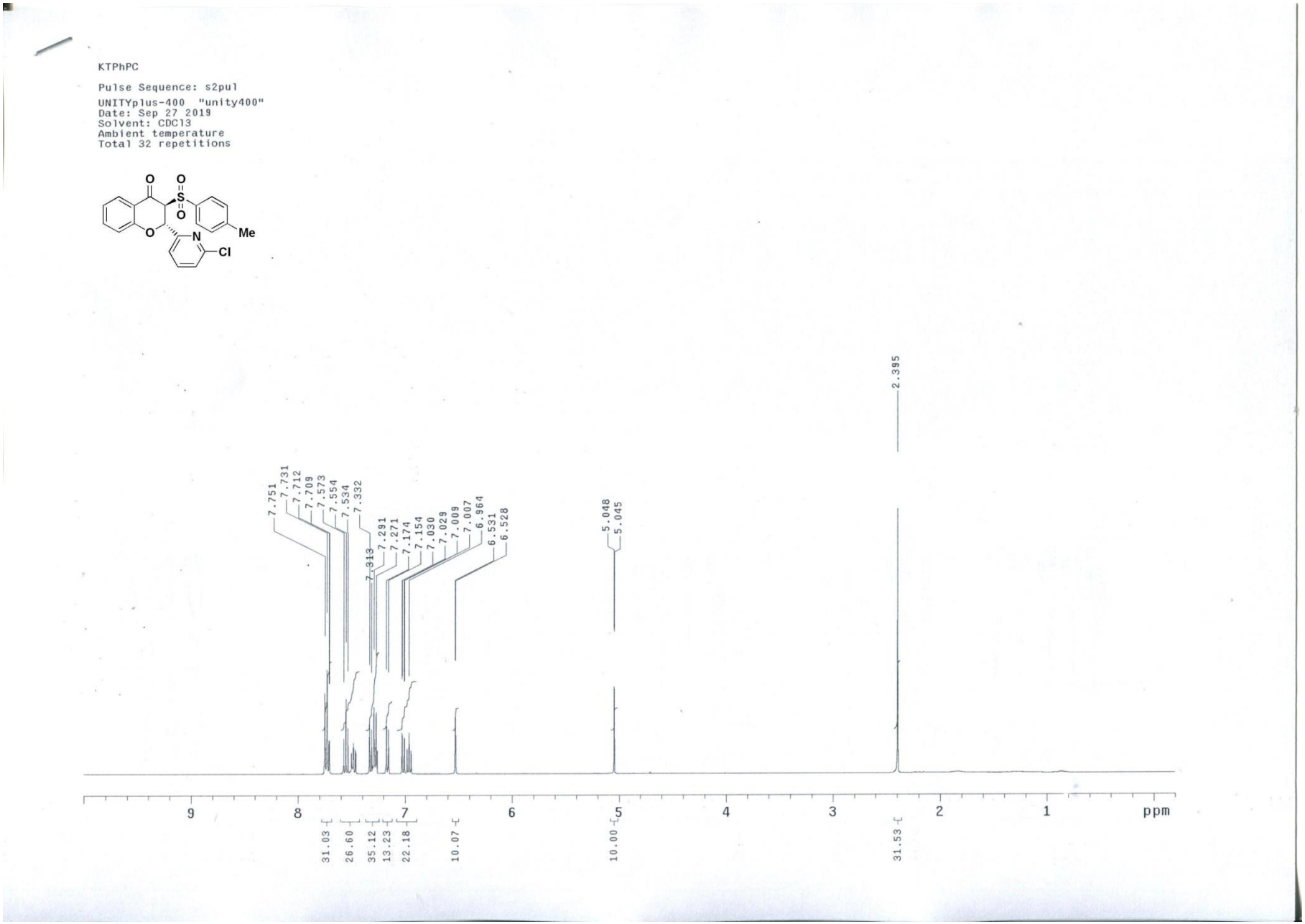


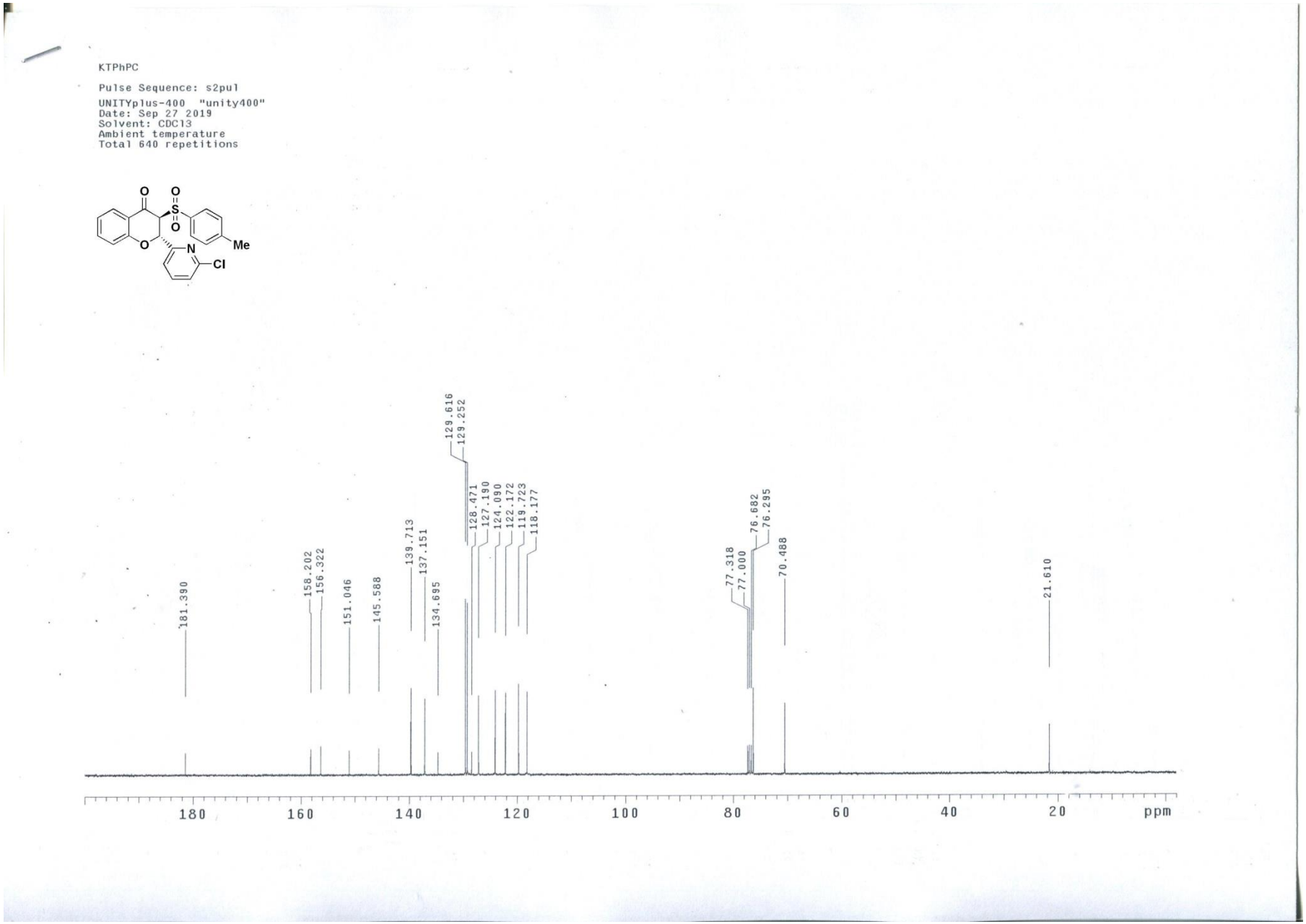
Compound 2s (^{13}C-NMR spectral data)



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



Compound 2t (^{13}C-NMR spectral data)



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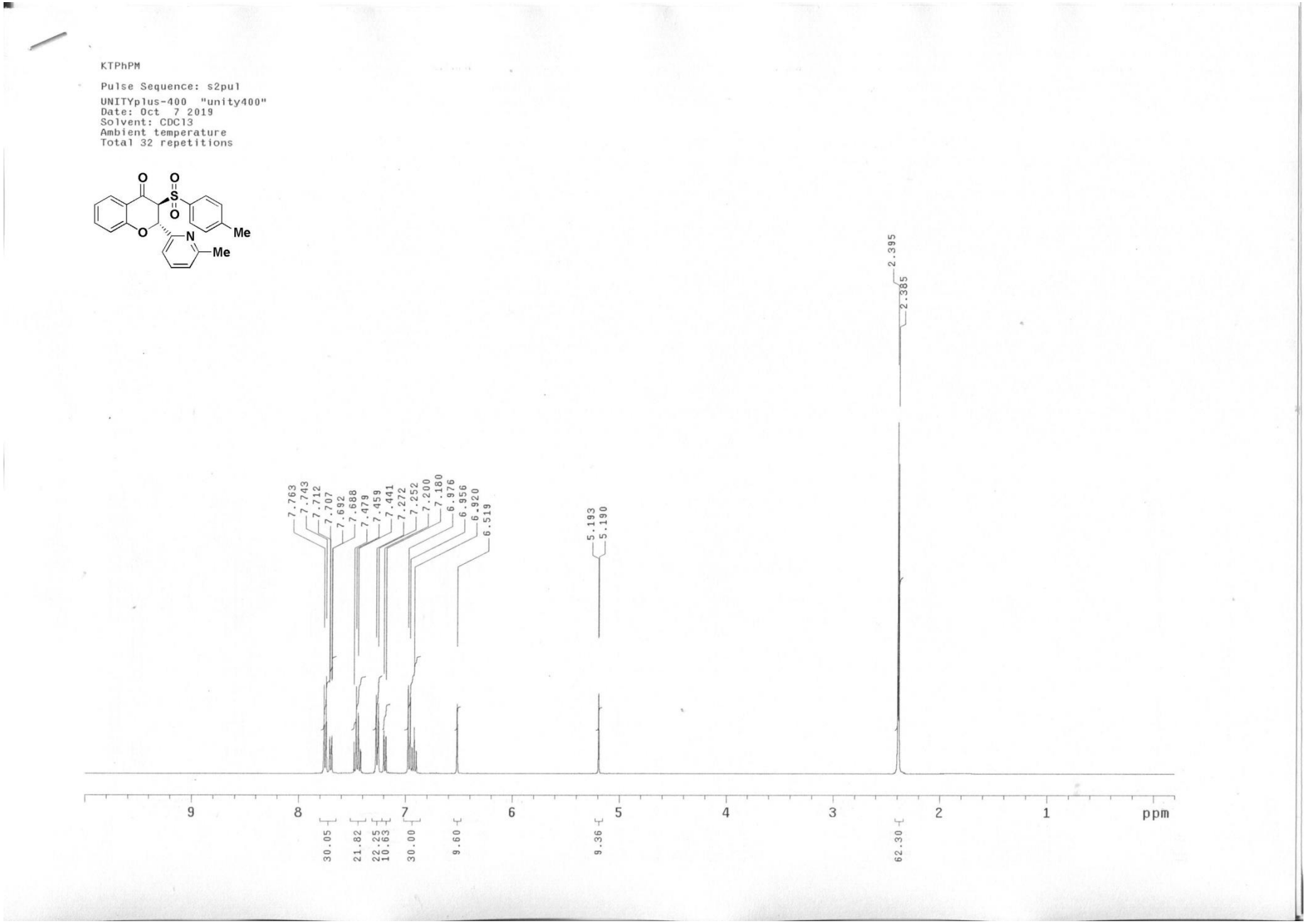
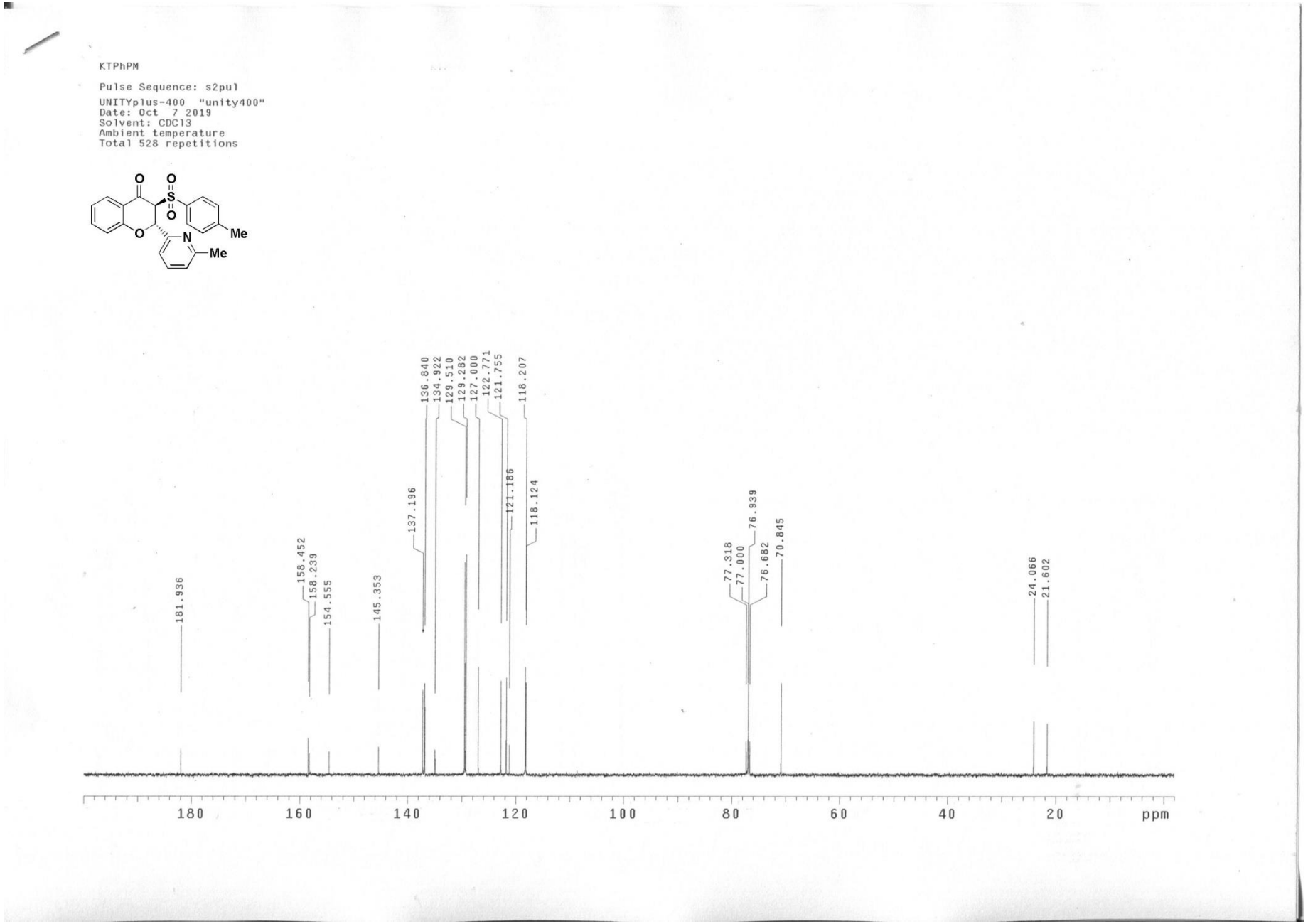
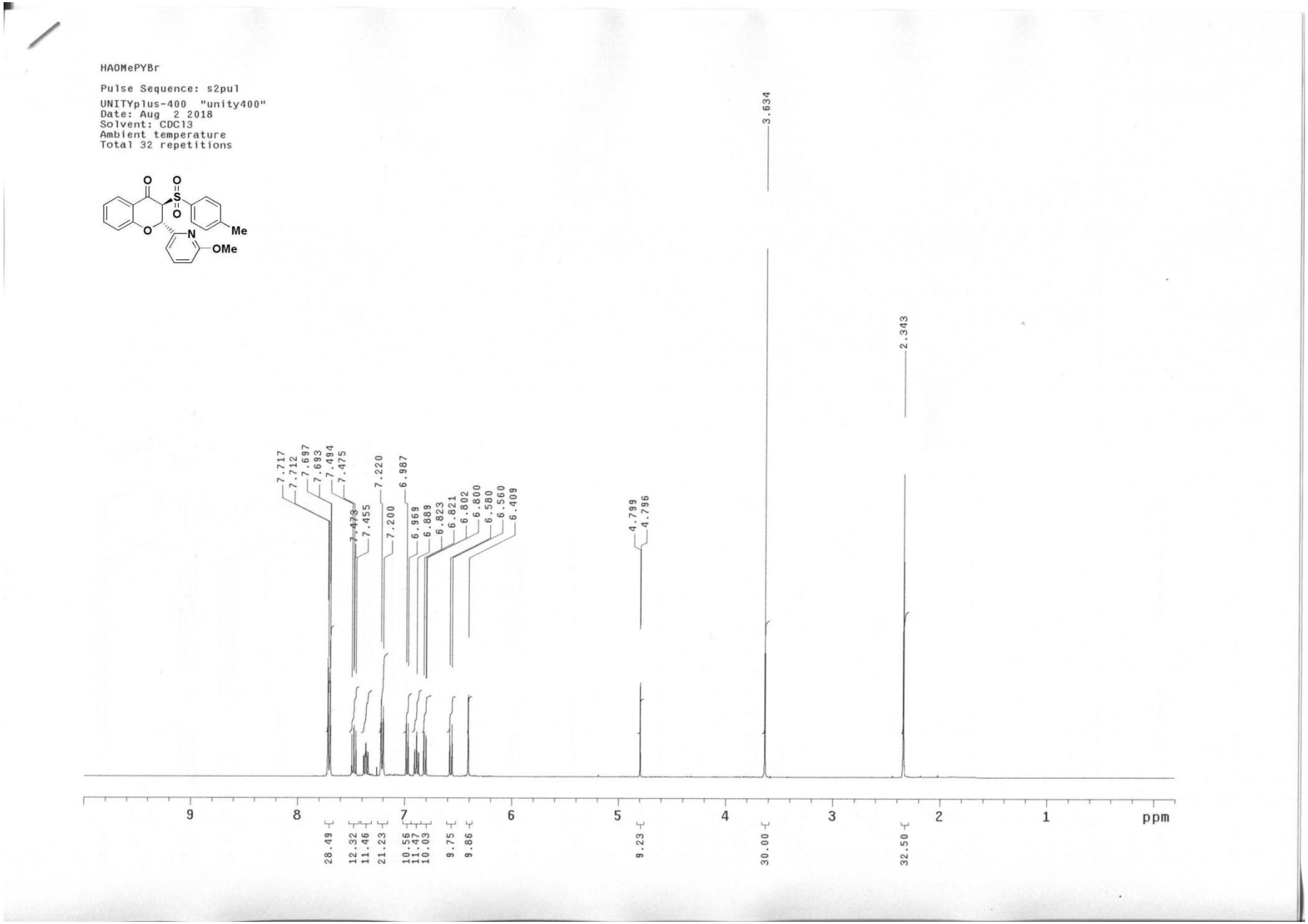


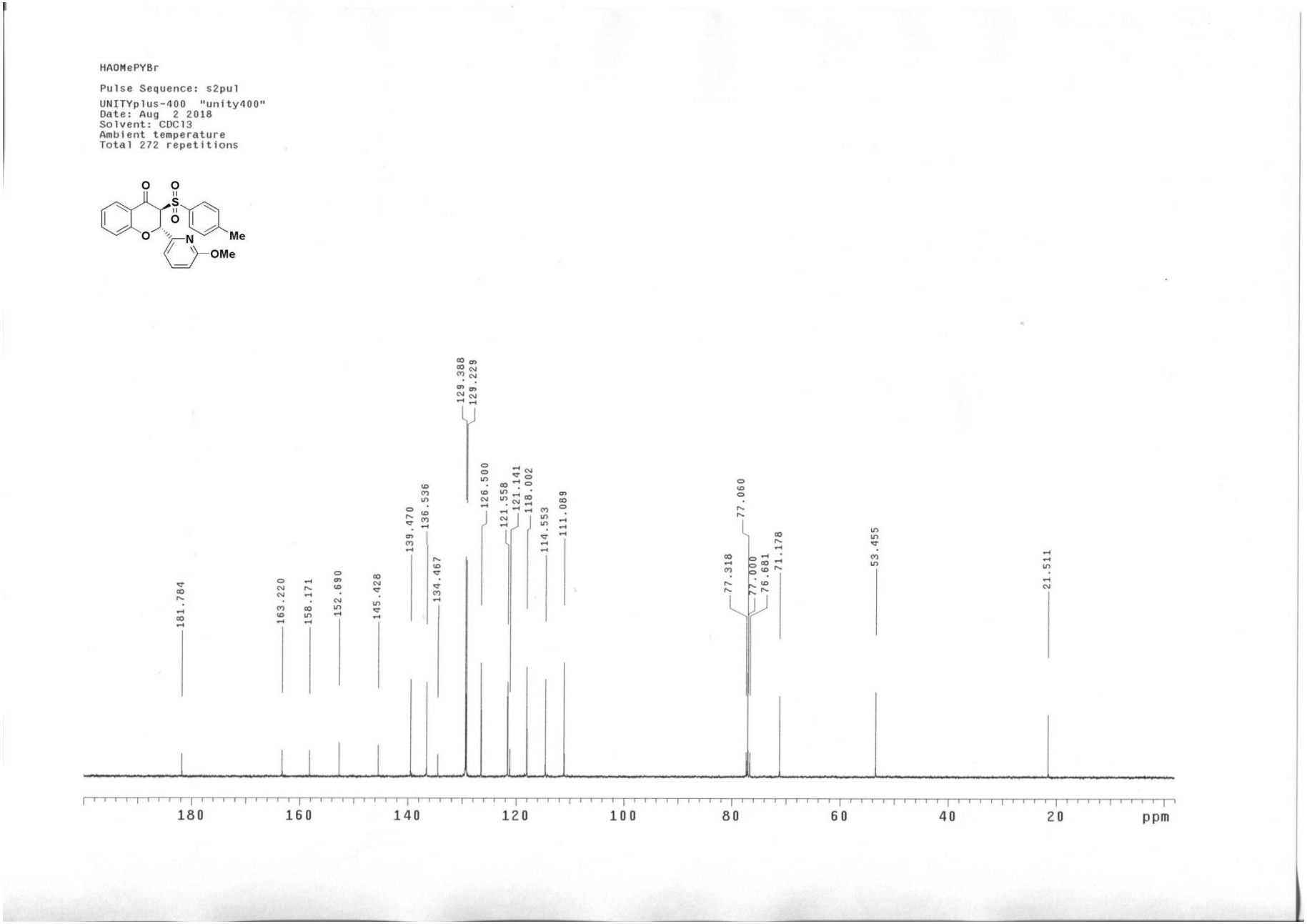
Compound 2u (^{13}C-NMR spectral data)



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



Compound 2v (^{13}C-NMR spectral data)



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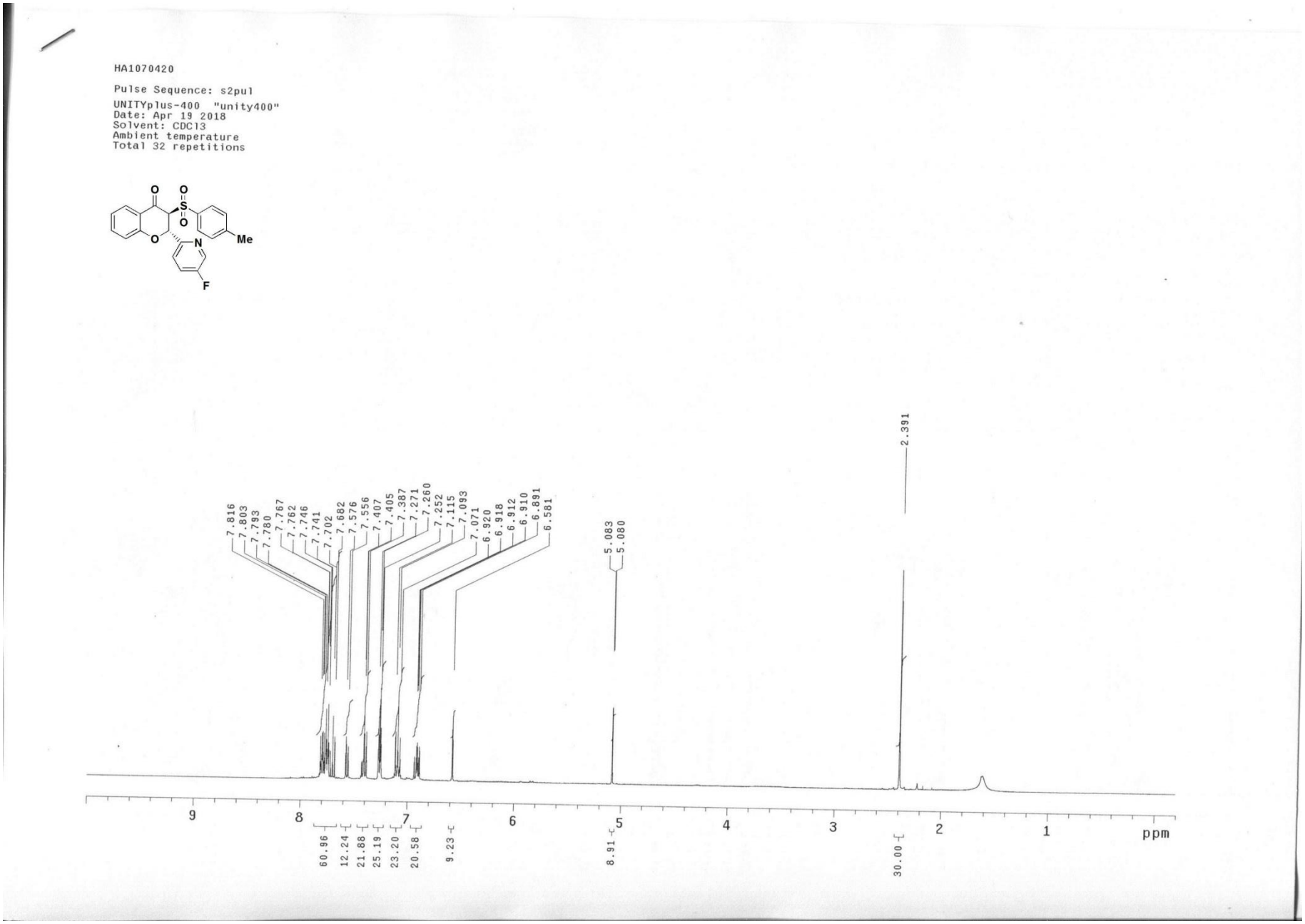
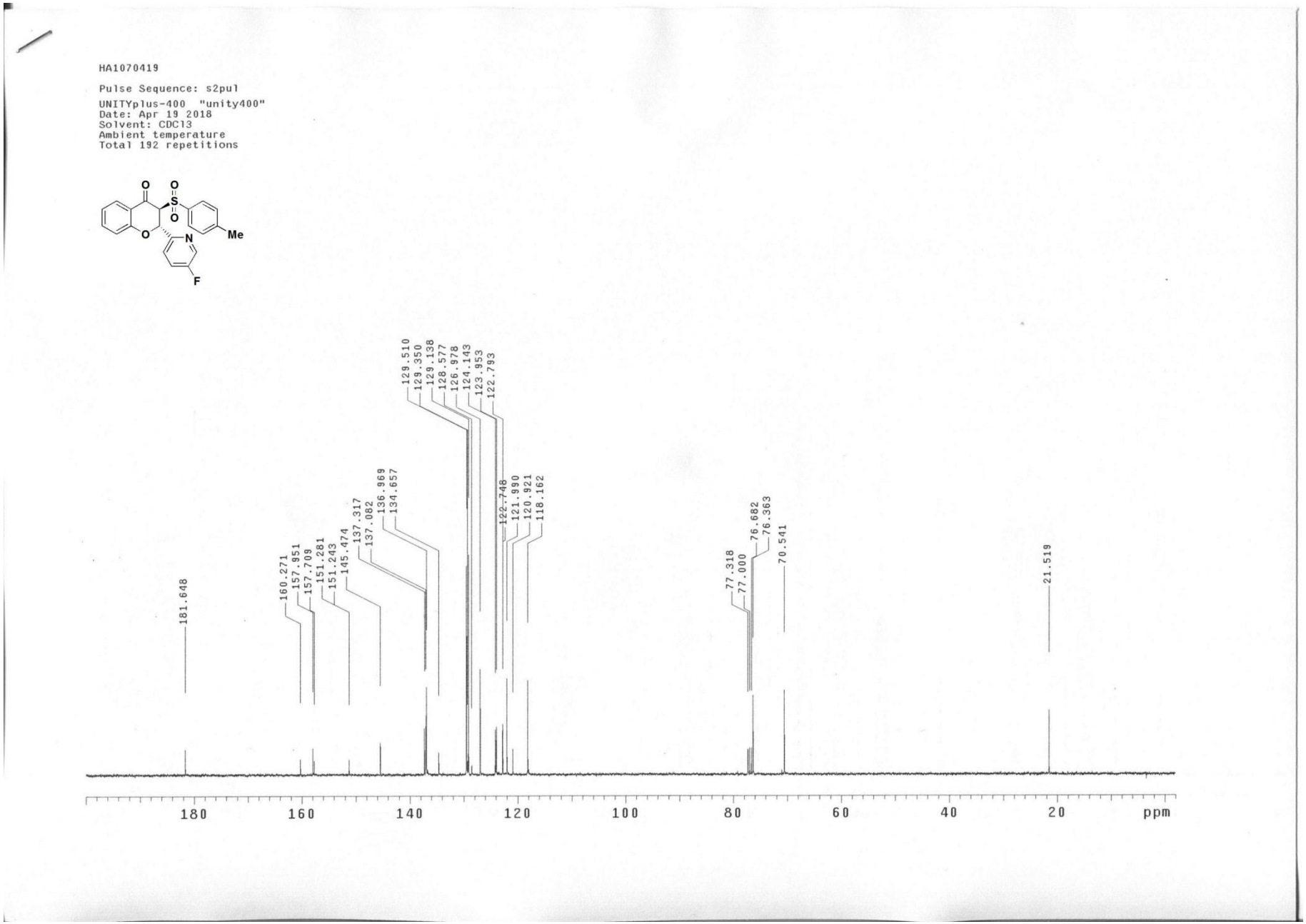
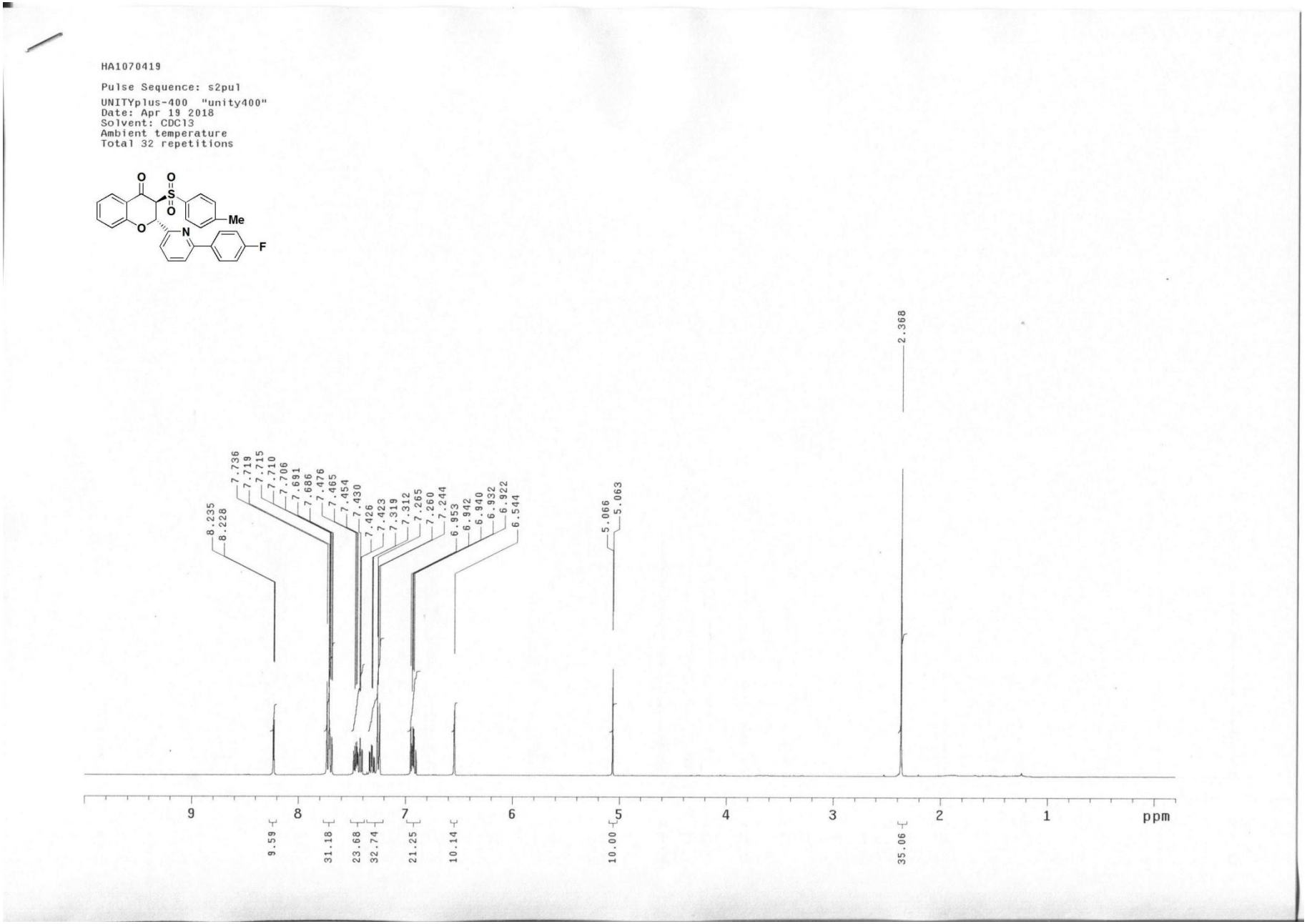


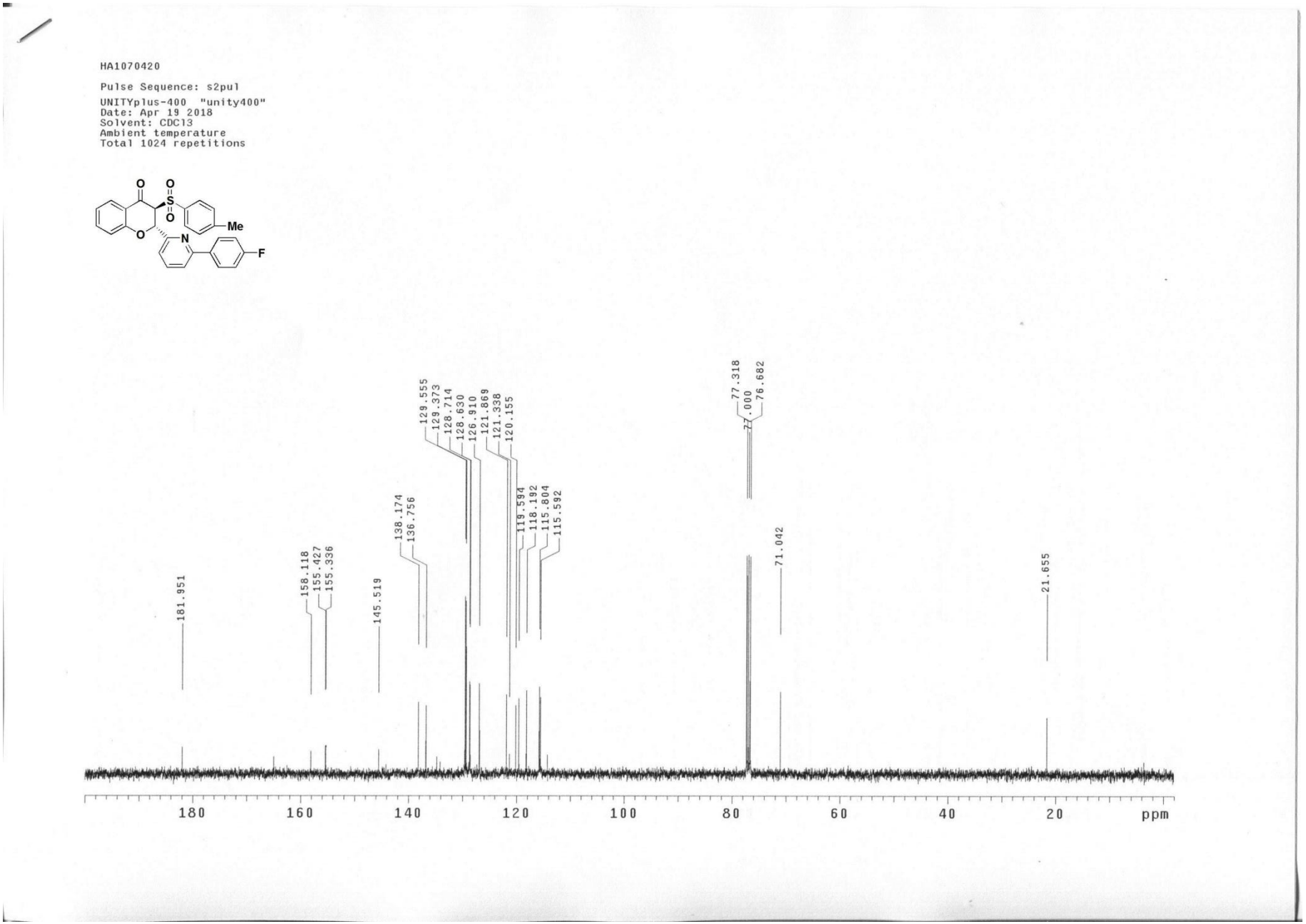
Compound 2w (^{13}C-NMR spectral data)



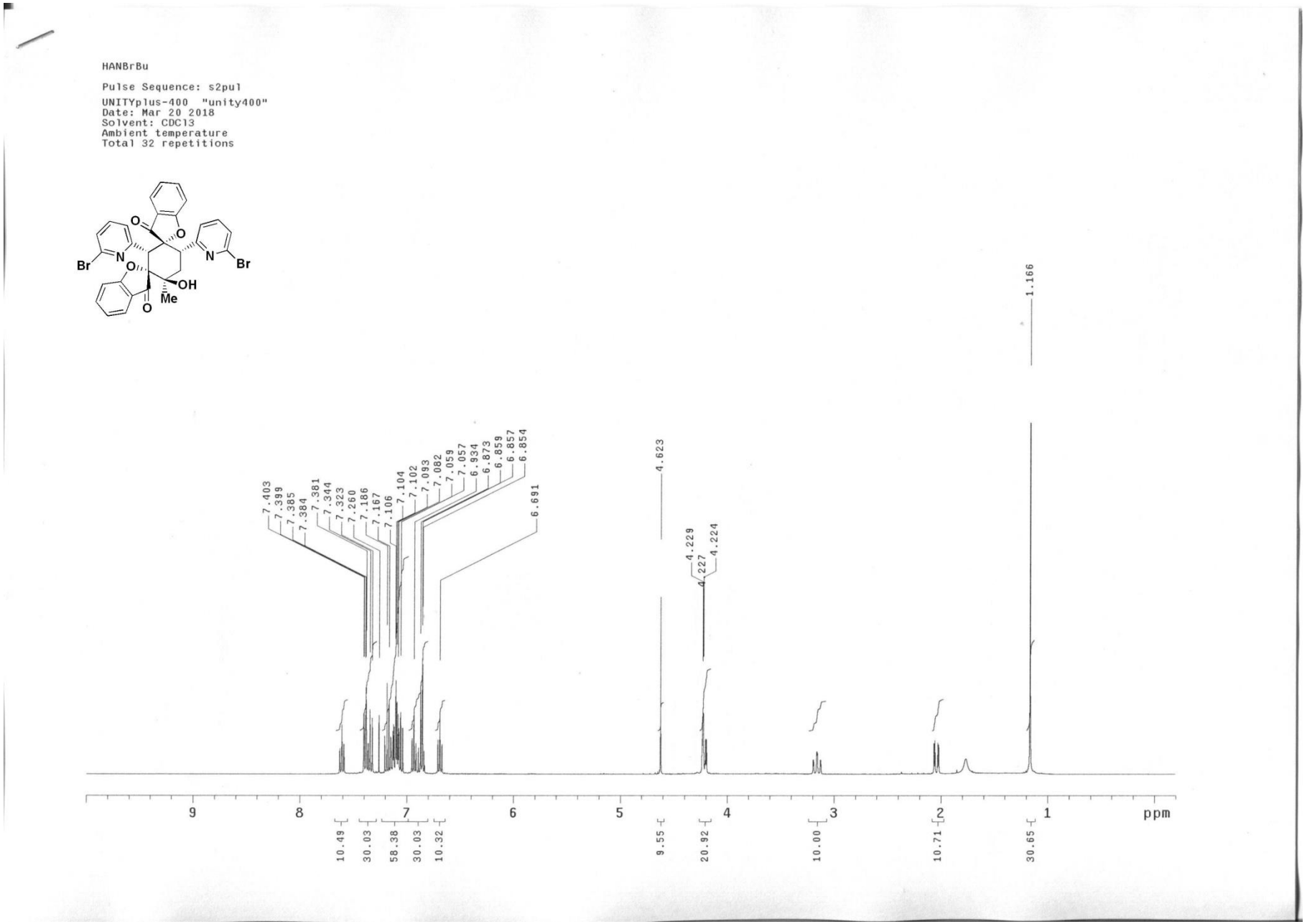
Compound 2x (^{1}H-NMR spectral data)



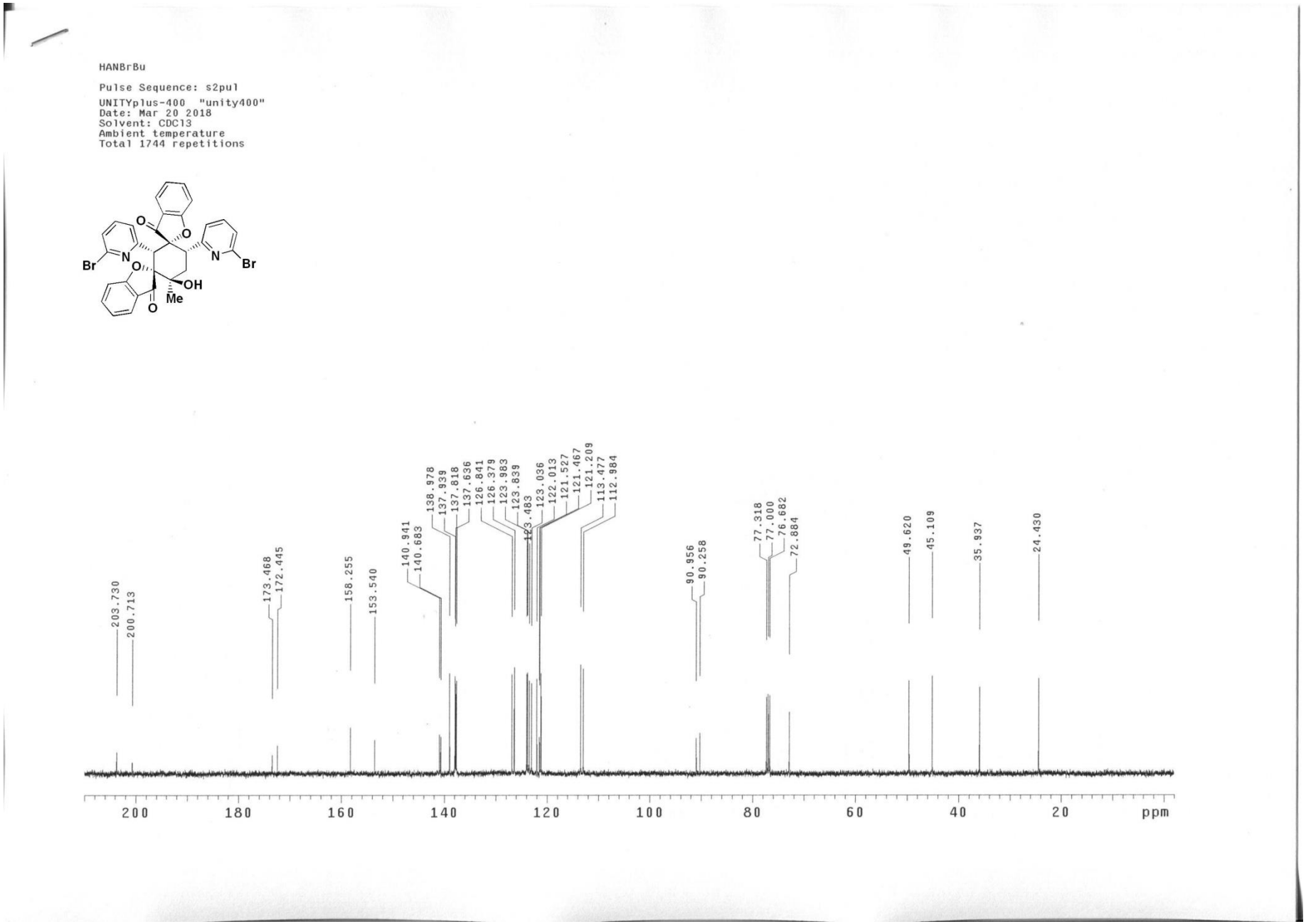
Compound 2x (^{13}C-NMR spectral data)



Compound 3a (^{1}H-NMR spectral data)



Compound 3a (^{13}C-NMR spectral data)



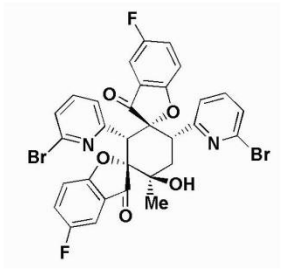
Compound 3b (^{1}H-NMR spectral data)

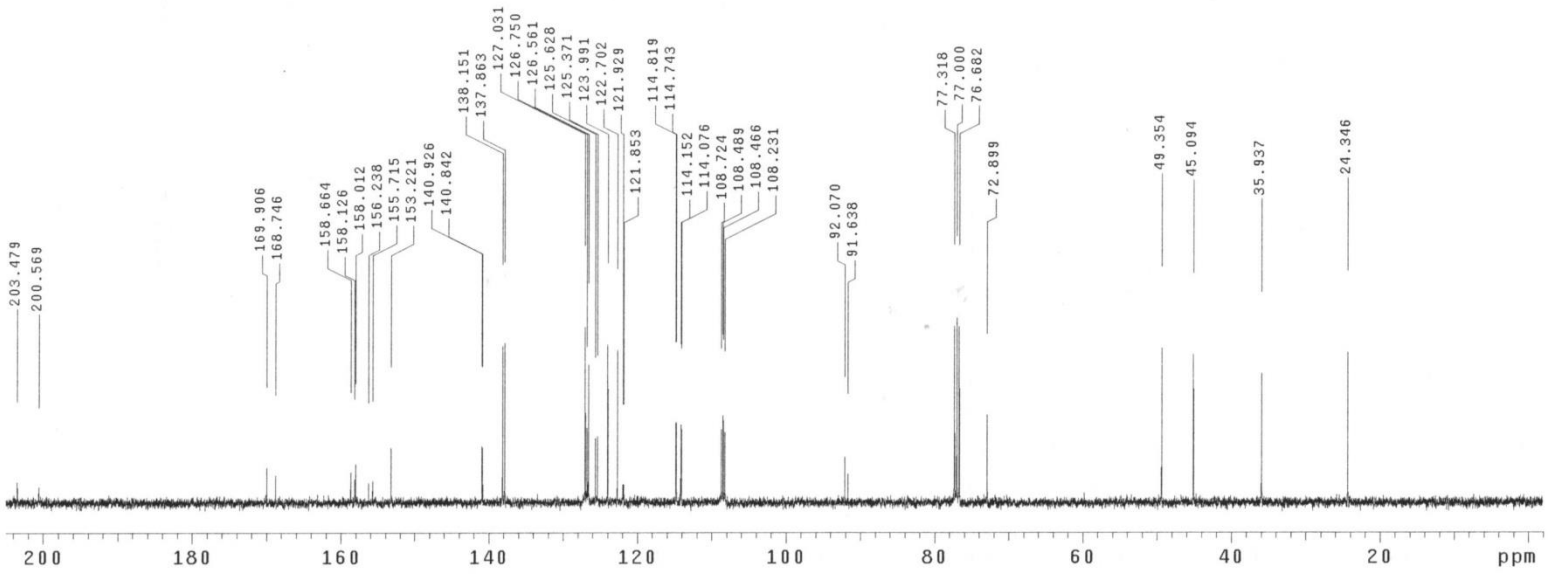
Compound 3b (^{13}C-NMR spectral data)

HAFPyBr

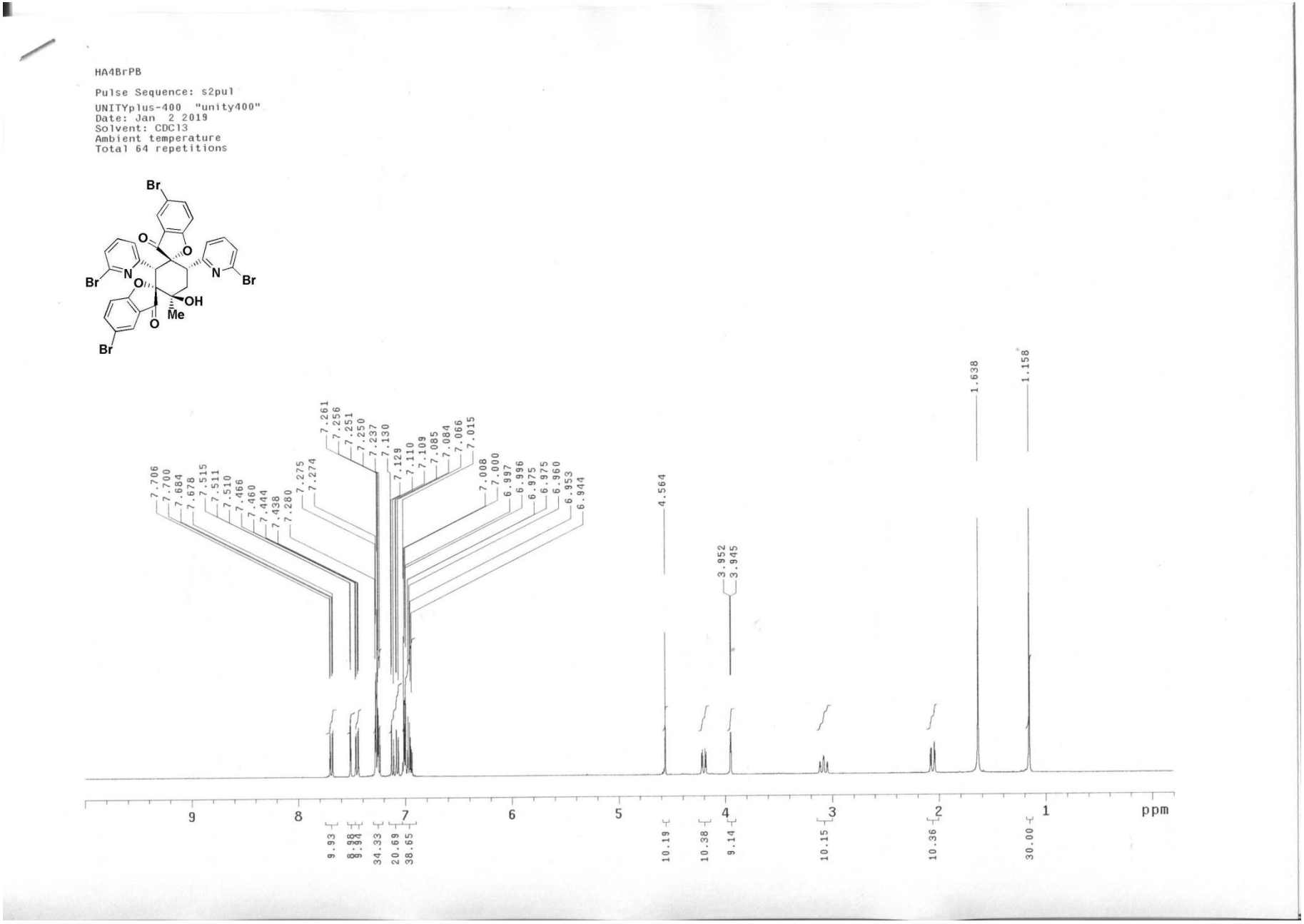
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jan 15 2019
Solvent: CDCl3
Ambient temperature
Total 2048 repetitions

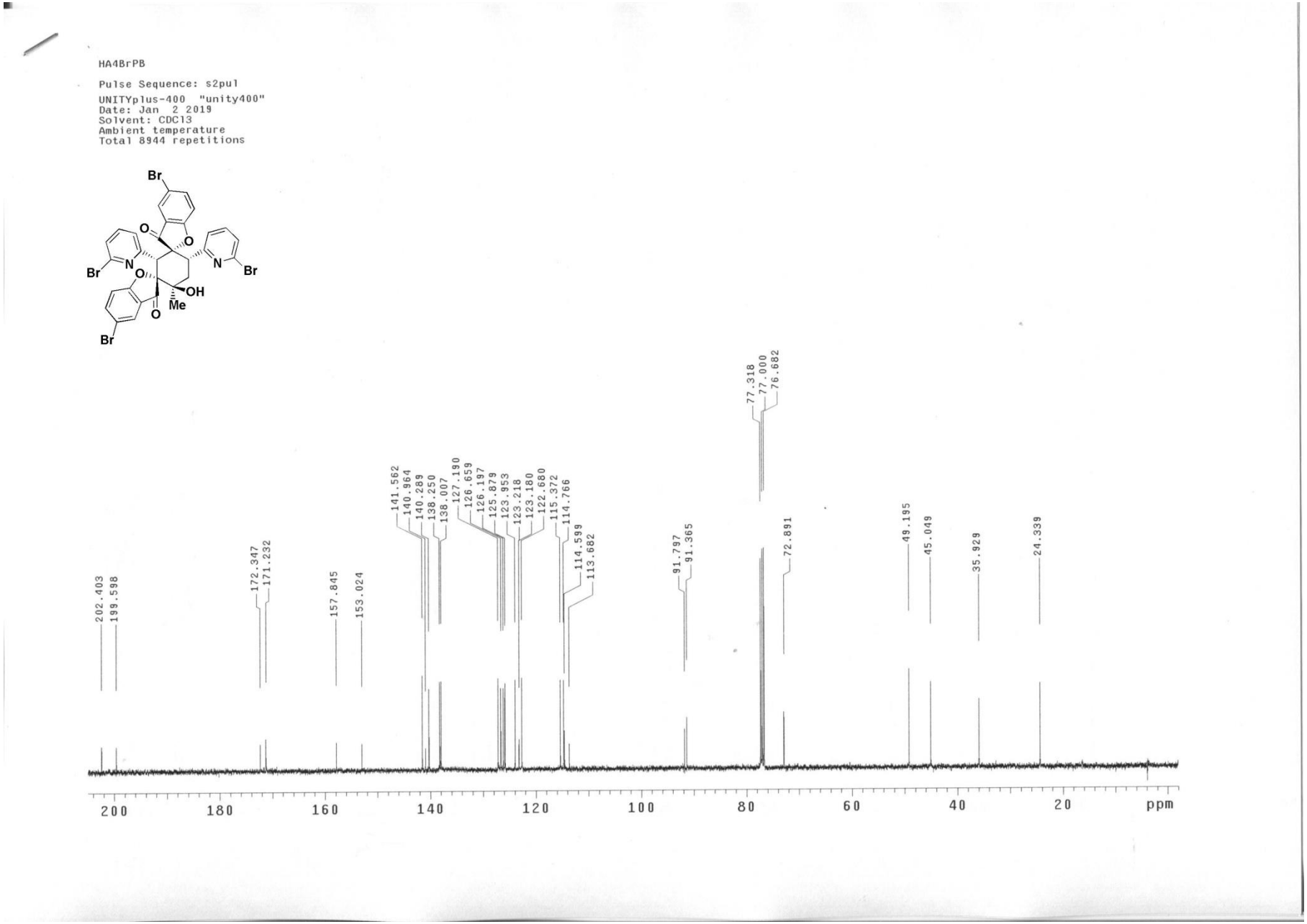




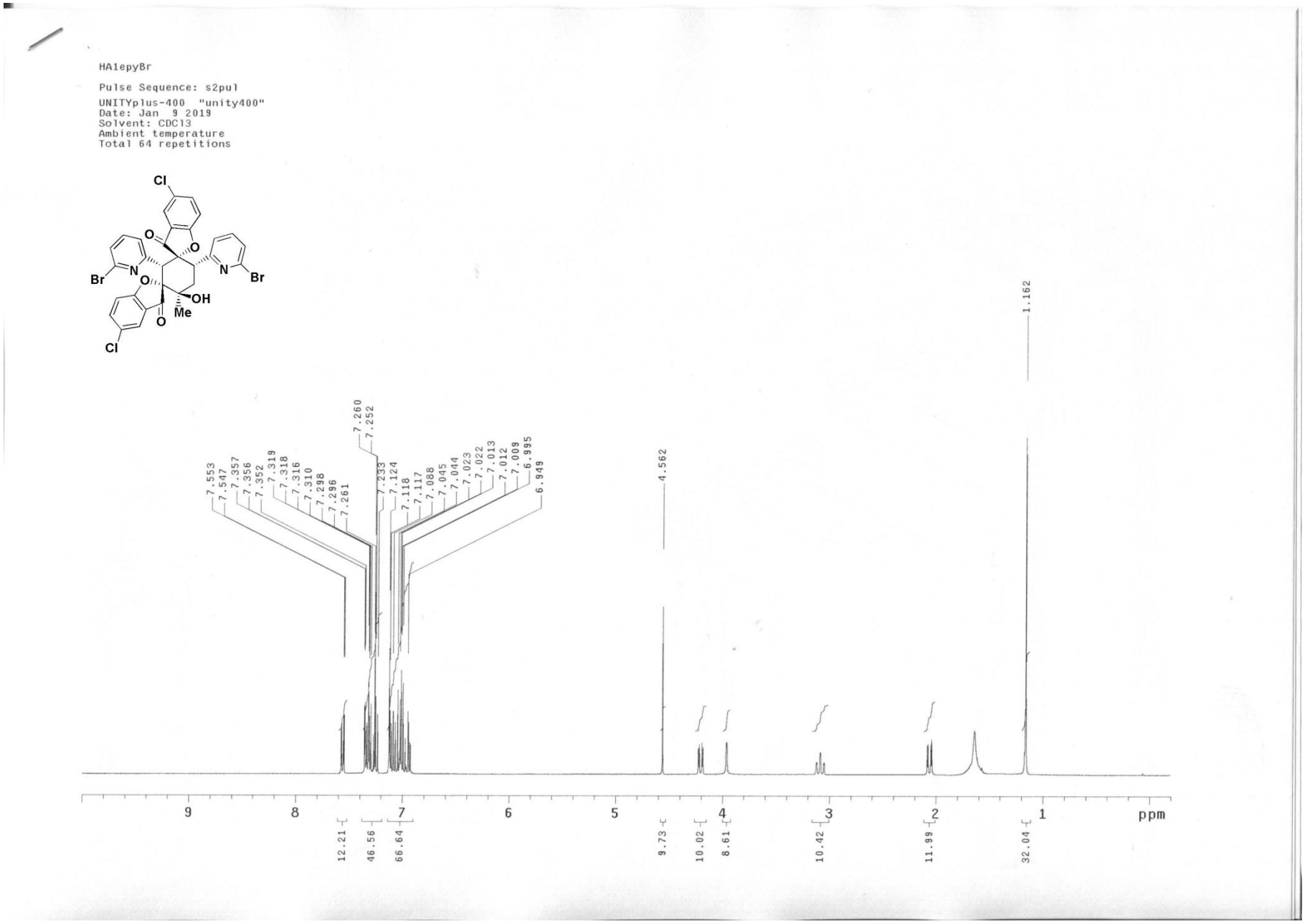
Compound 3c (^{1}H-NMR spectral data)



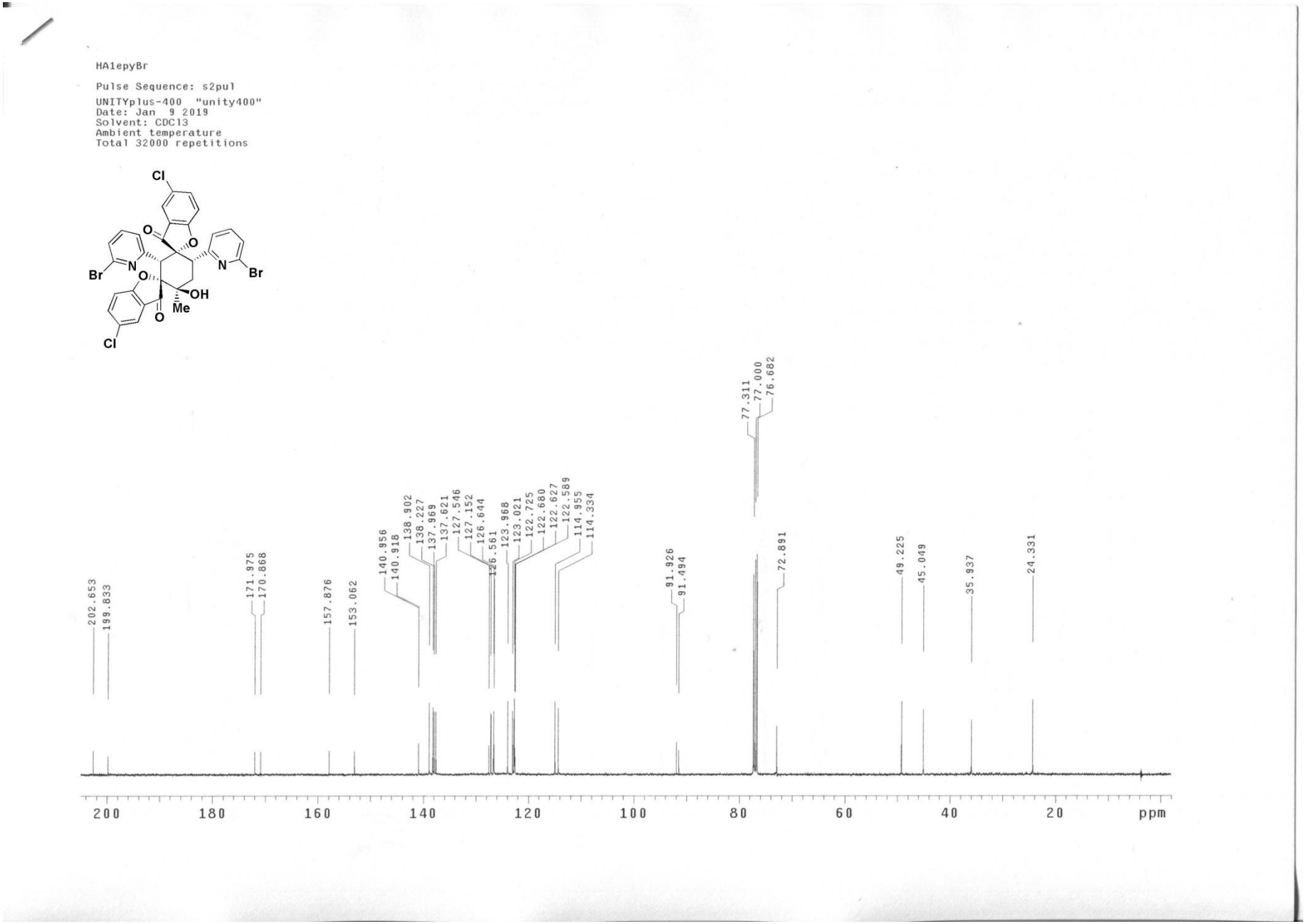
Compound 3c (^{13}C-NMR spectral data)



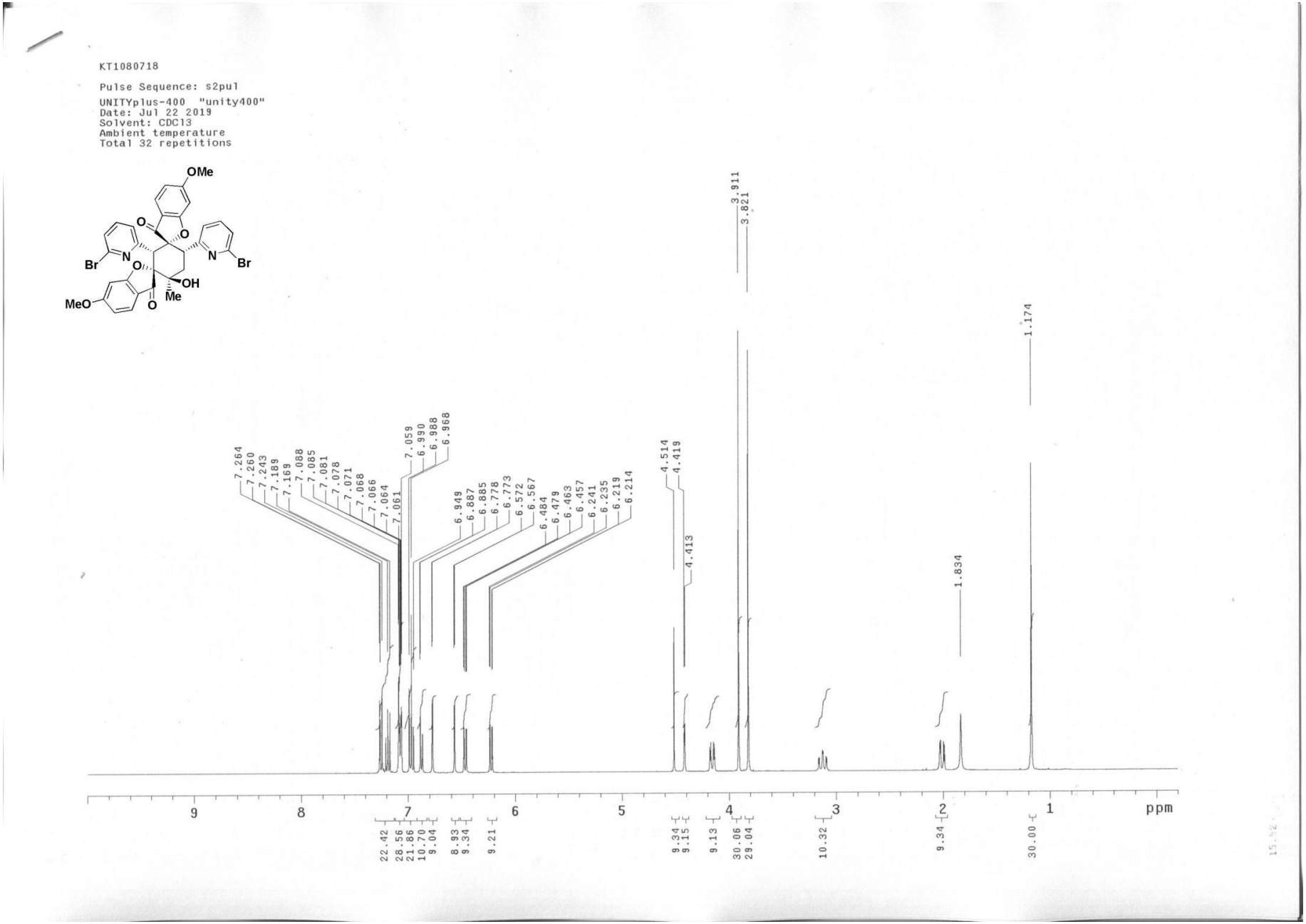
Compound 3d (^{1}H-NMR spectral data)



Compound 3d (^{13}C-NMR spectral data)



Compound 3e (^{1}H-NMR spectral data)

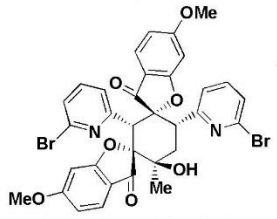


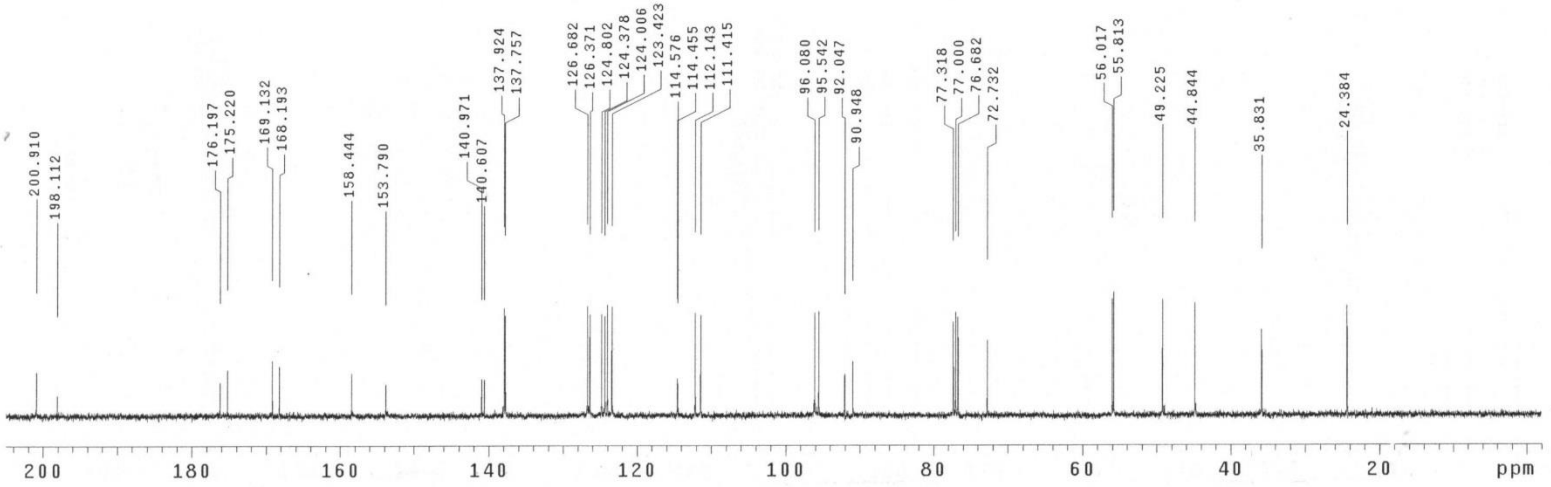
Compound 3e (^{13}C-NMR spectral data)

KT1080718

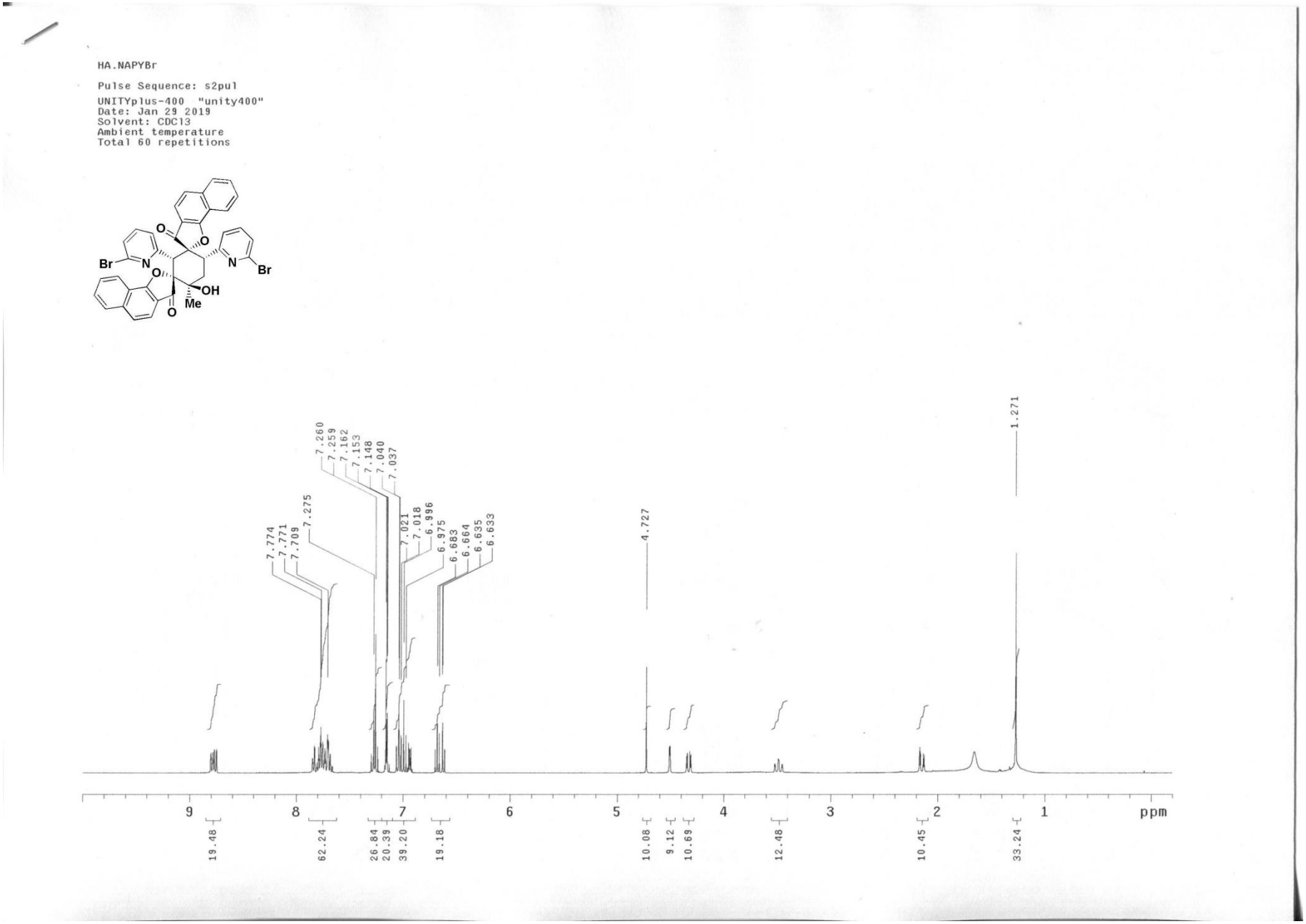
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Jul 22 2019
Solvent: CDCl3
Ambient temperature
Total 1776 repetitions

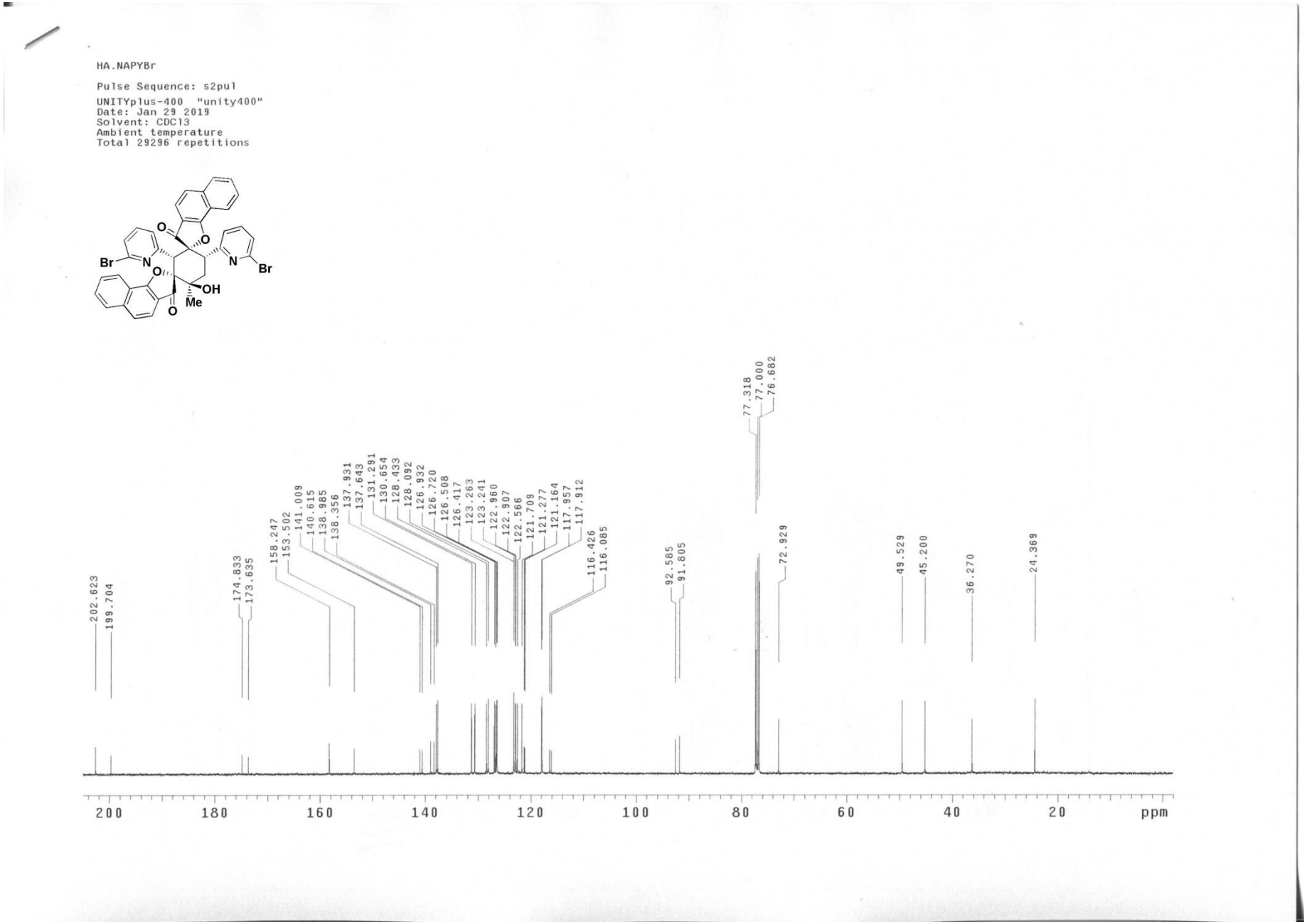




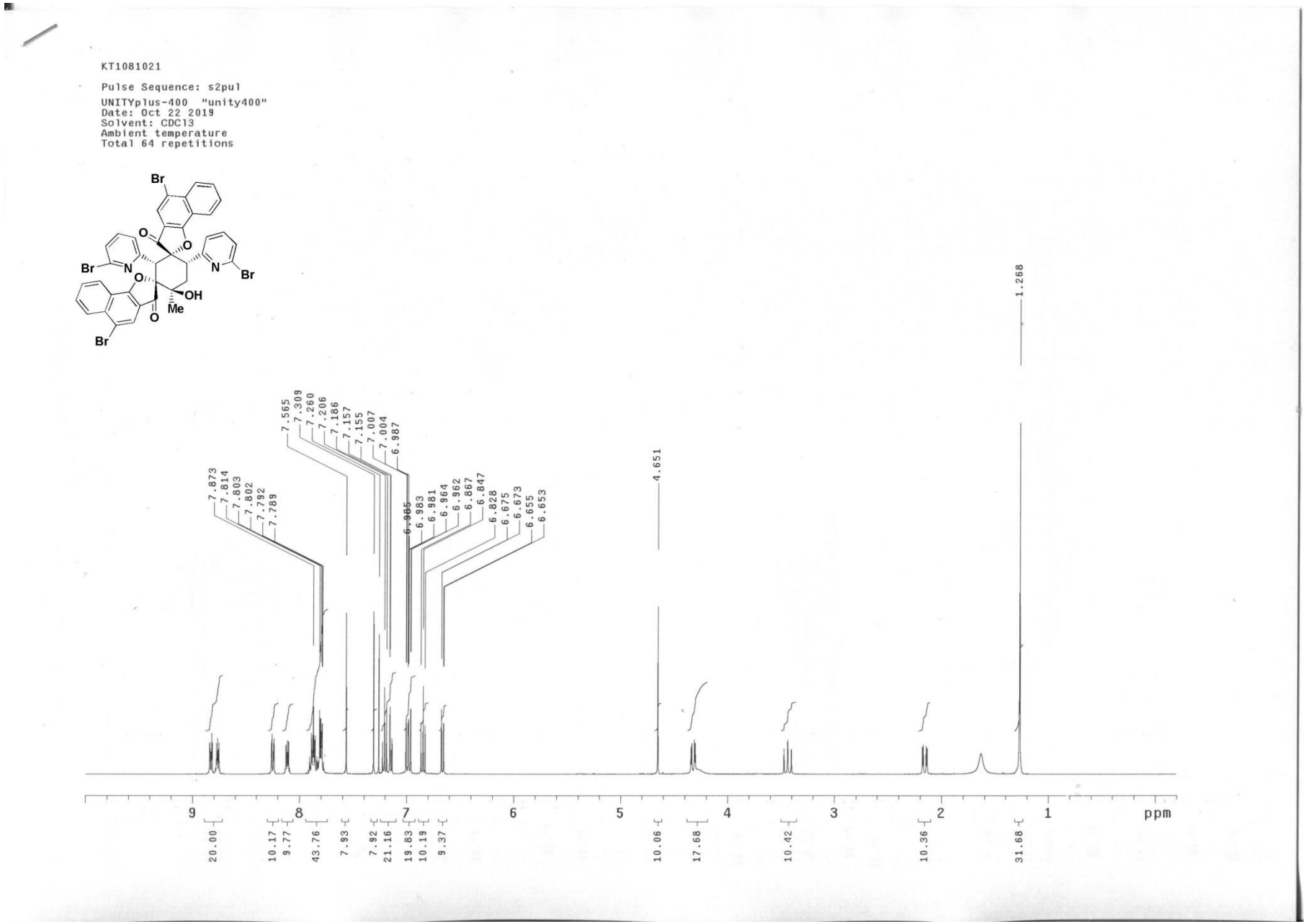
Compound 3f (^{1}H-NMR spectral data)



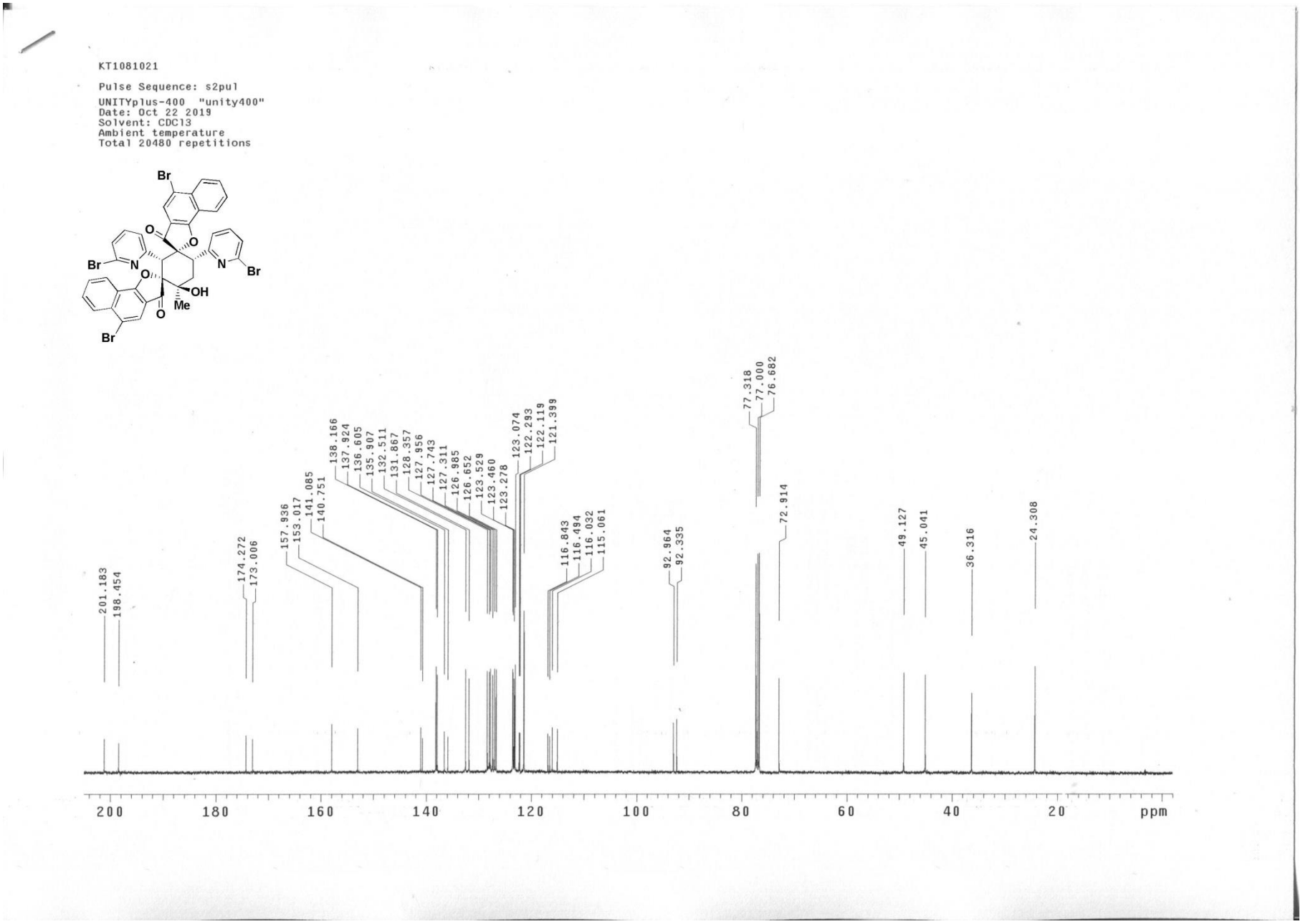
Compound 3f (^{13}C-NMR spectral data)



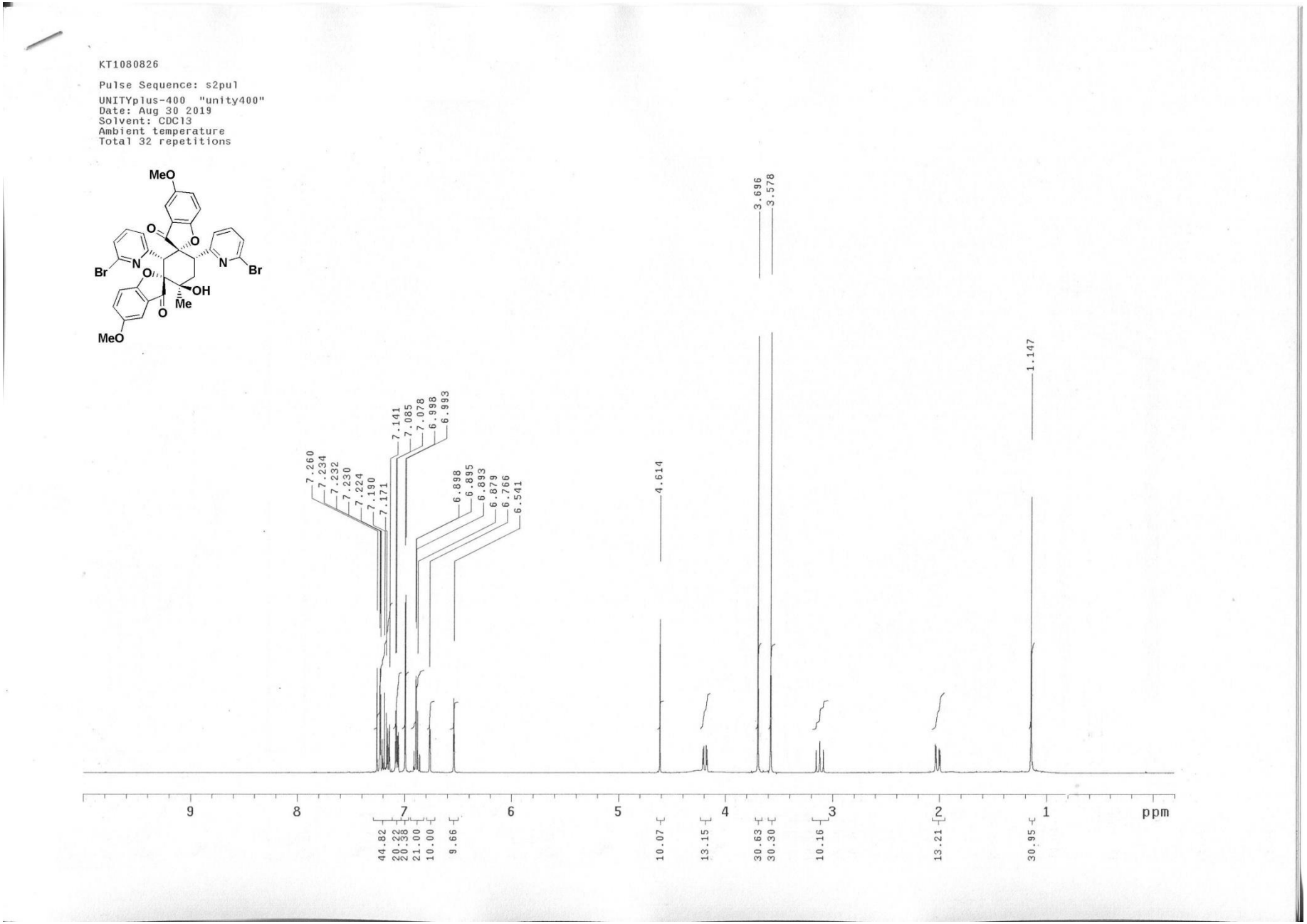
Compound 3g (^{1}H-NMR spectral data)



Compound 3g (^{13}C-NMR spectral data)



Compound 3h (^{1}H-NMR spectral data)

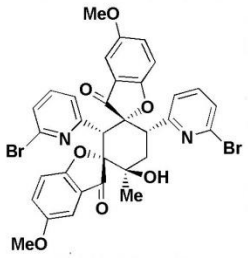


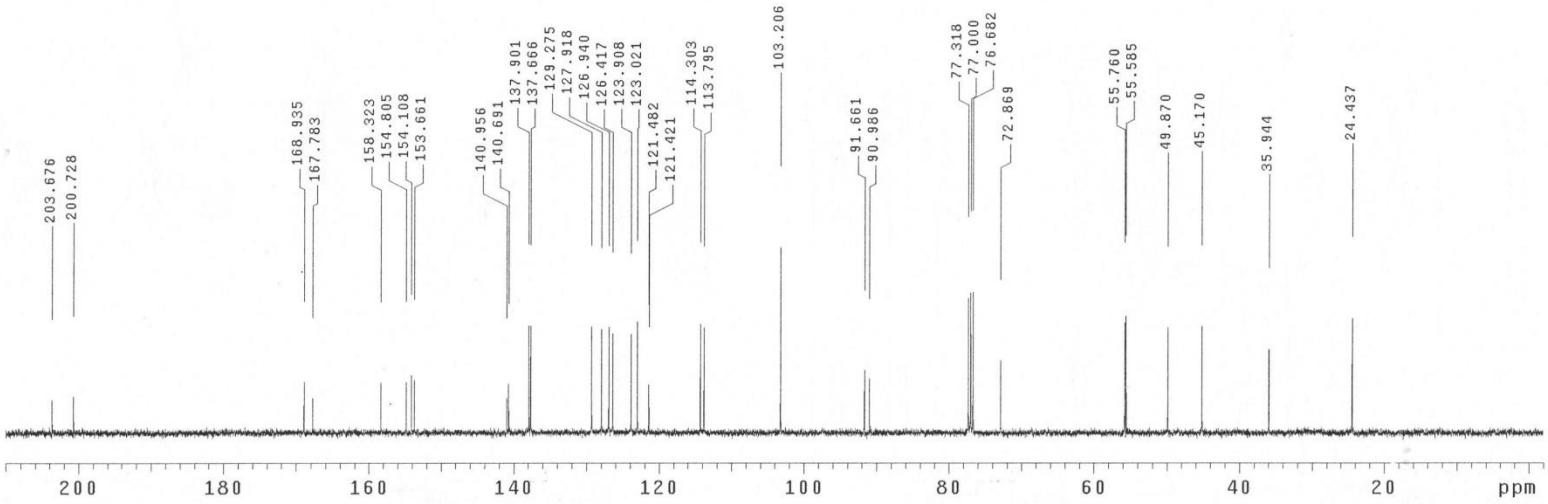
Compound 3h (^{13}C-NMR spectral data)

KT1080826

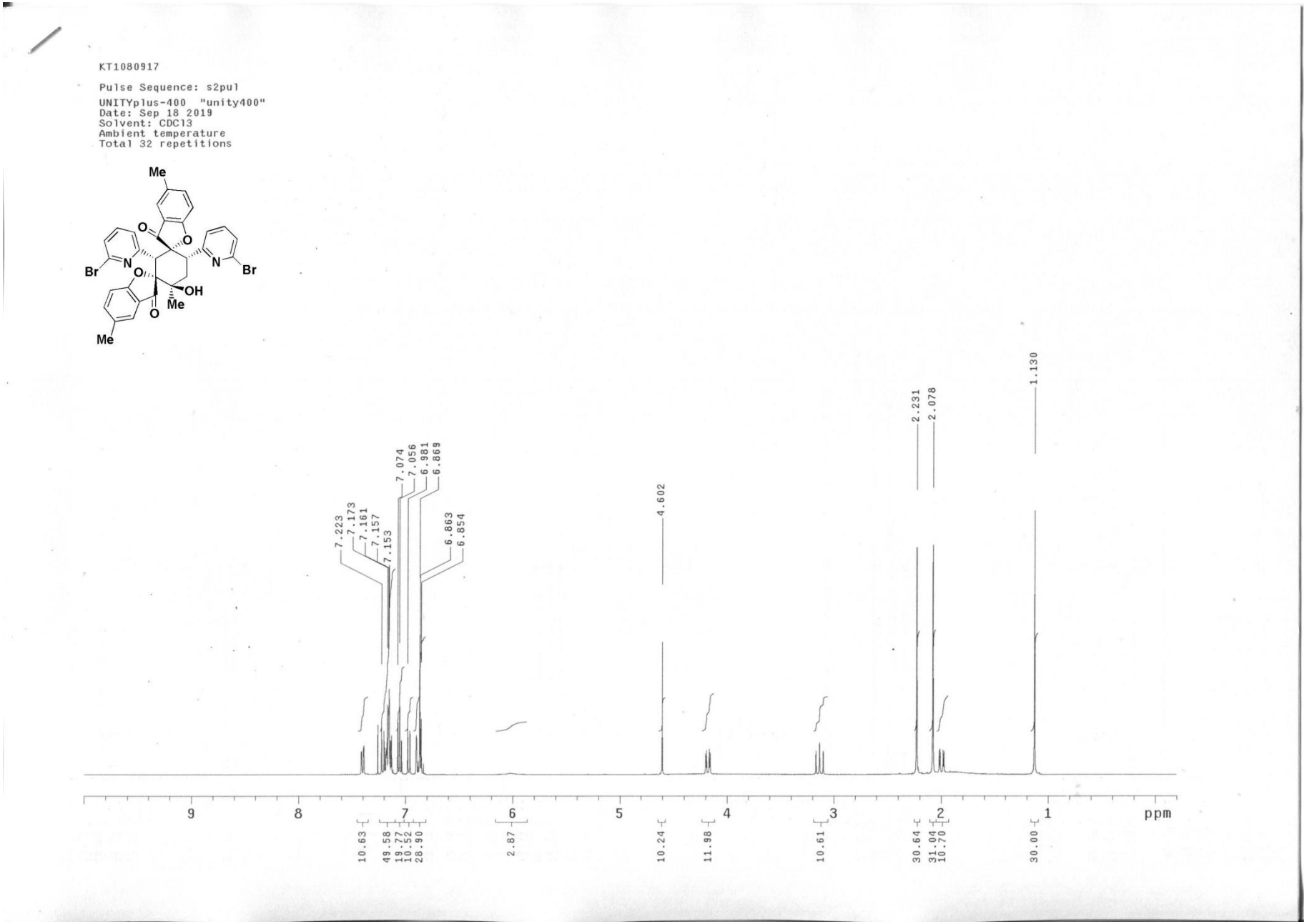
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Aug 30 2019
Solvent: CDCl3
Ambient temperature
Total 2016 repetitions

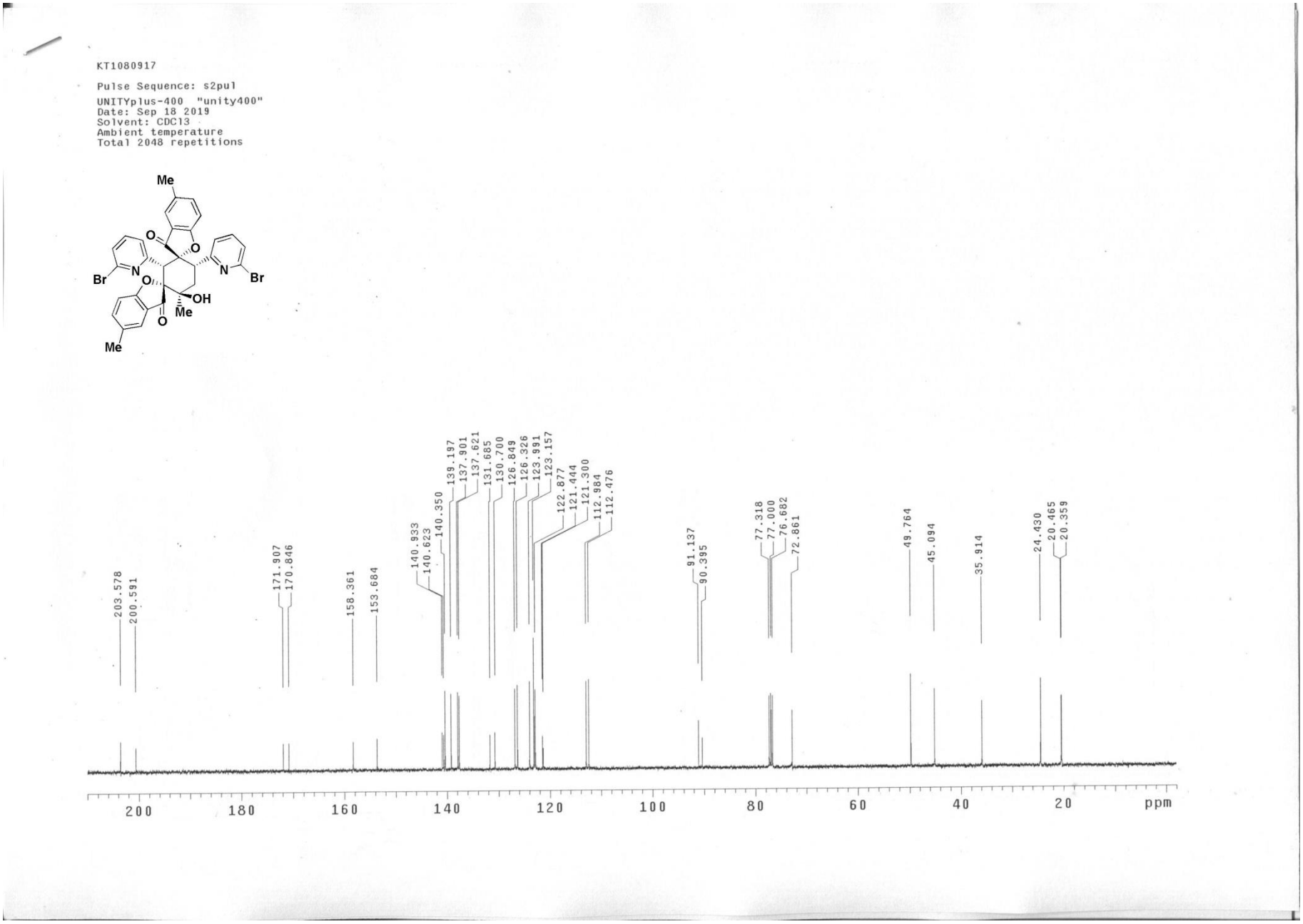




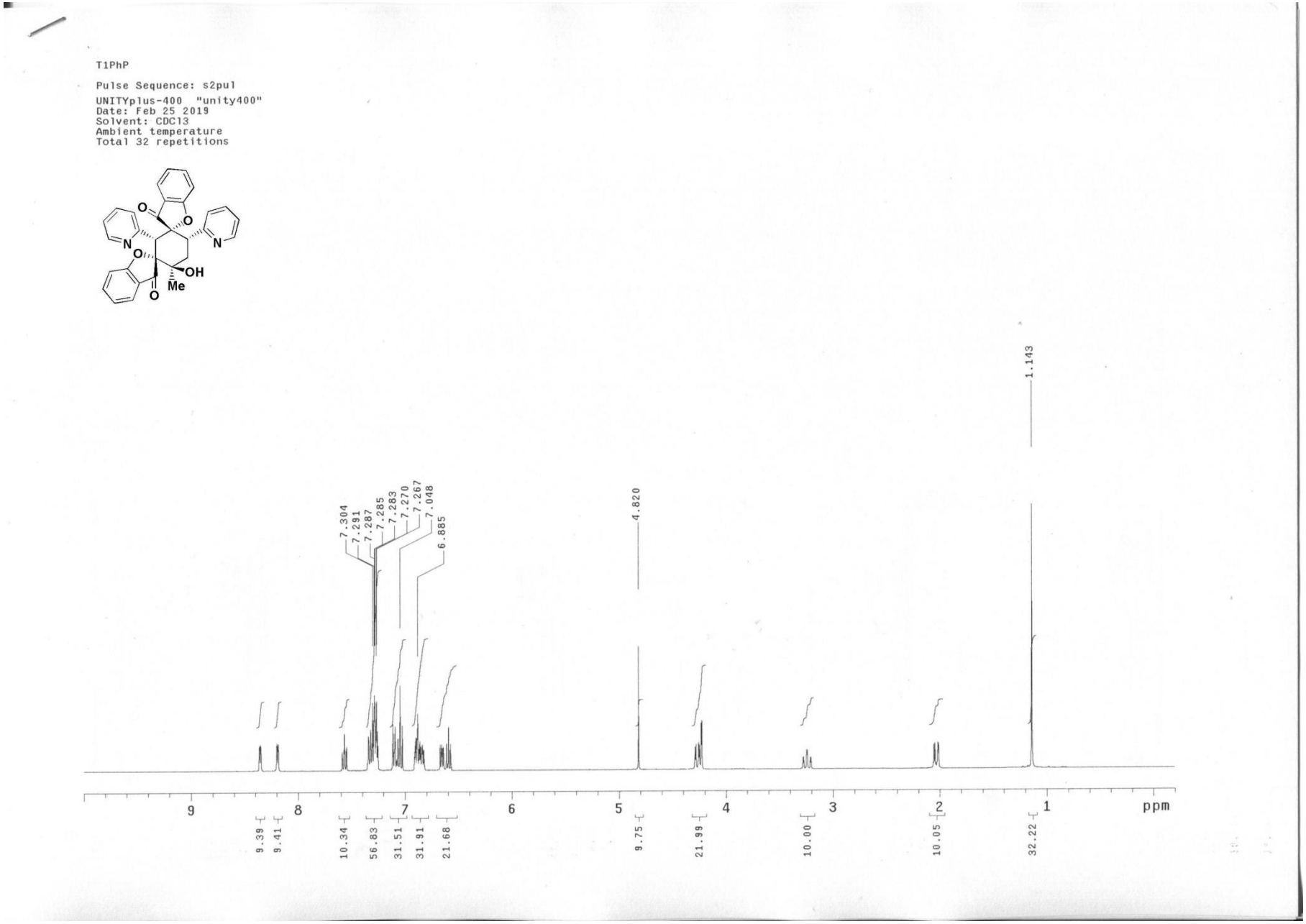
Compound 3i (^{1}H-NMR spectral data)



Compound 3i (^{13}C-NMR spectral data)



Compound 3j (^{1}H-NMR spectral data)

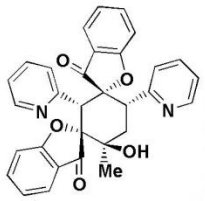


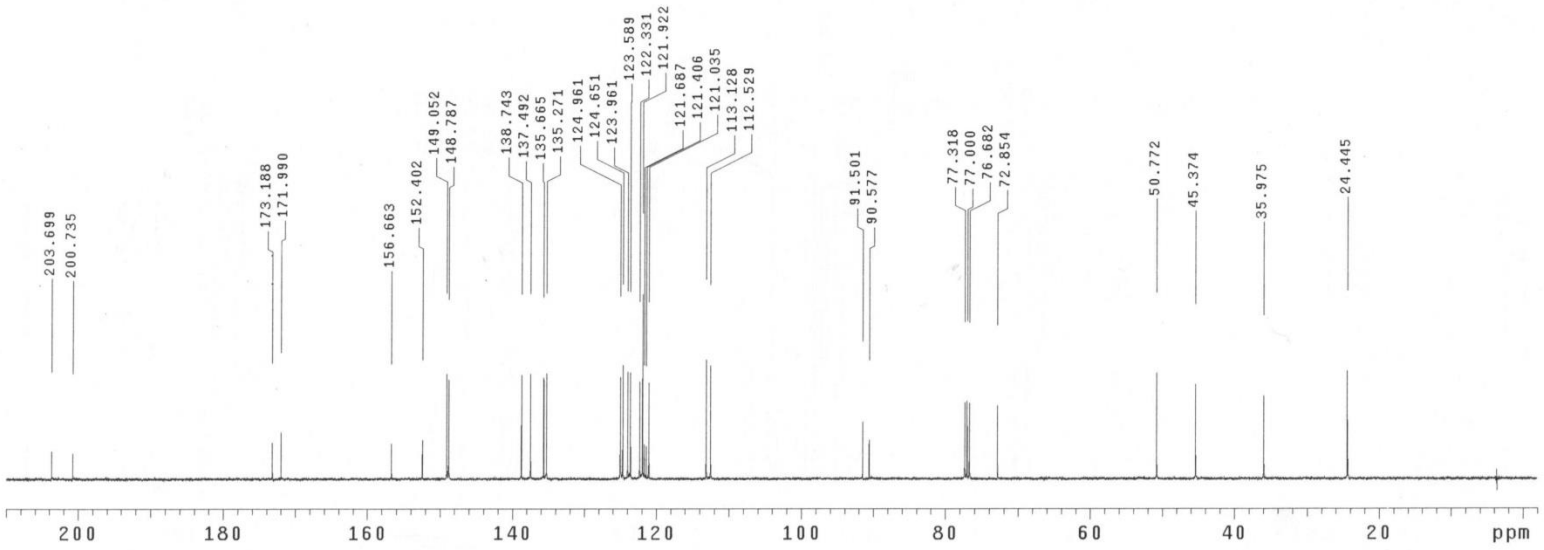
Compound 3j (^{13}C-NMR spectral data)

T1PhP

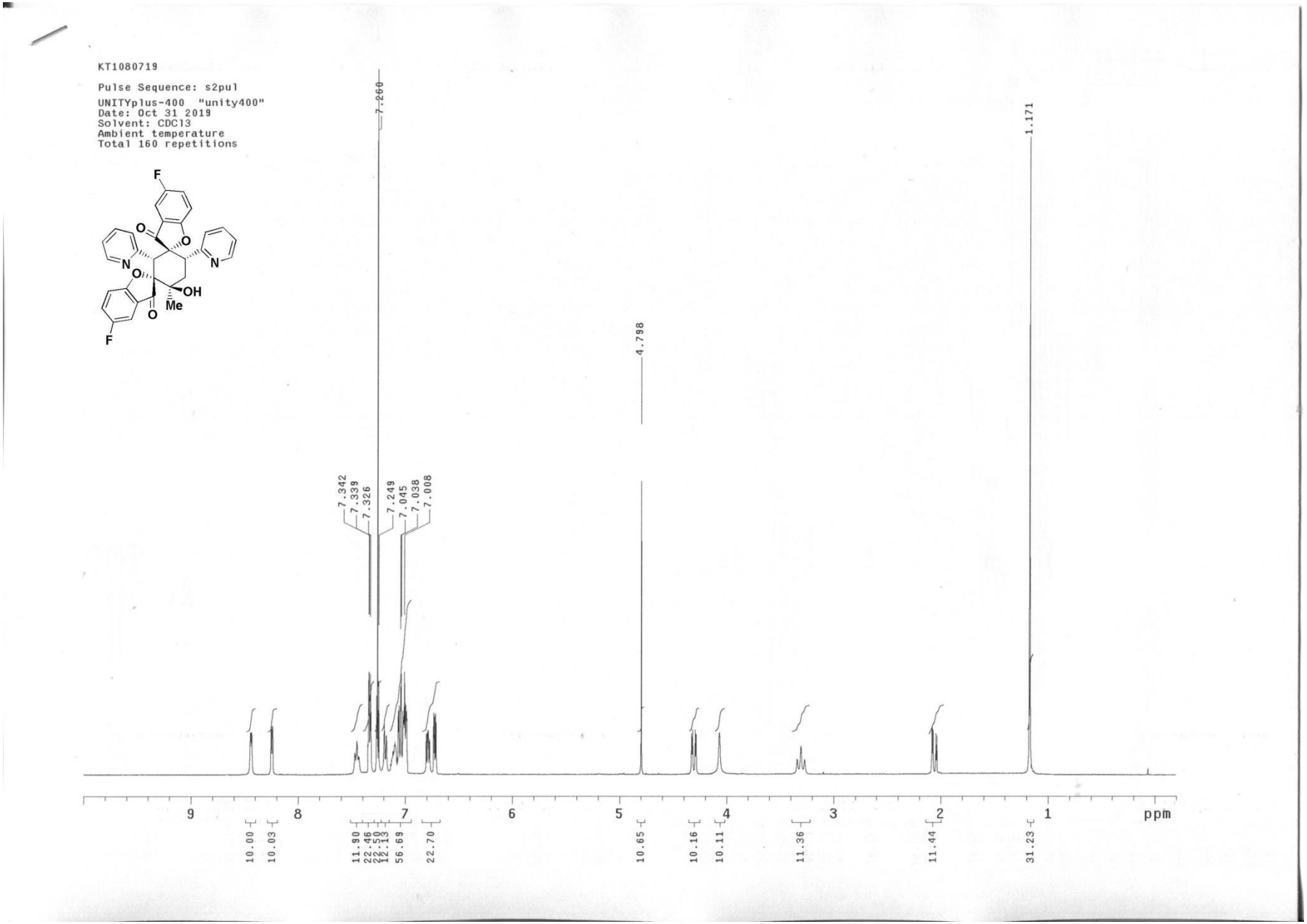
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Feb 25 2019
Solvent: CDCl3
Ambient temperature
Total 3008 repetitions

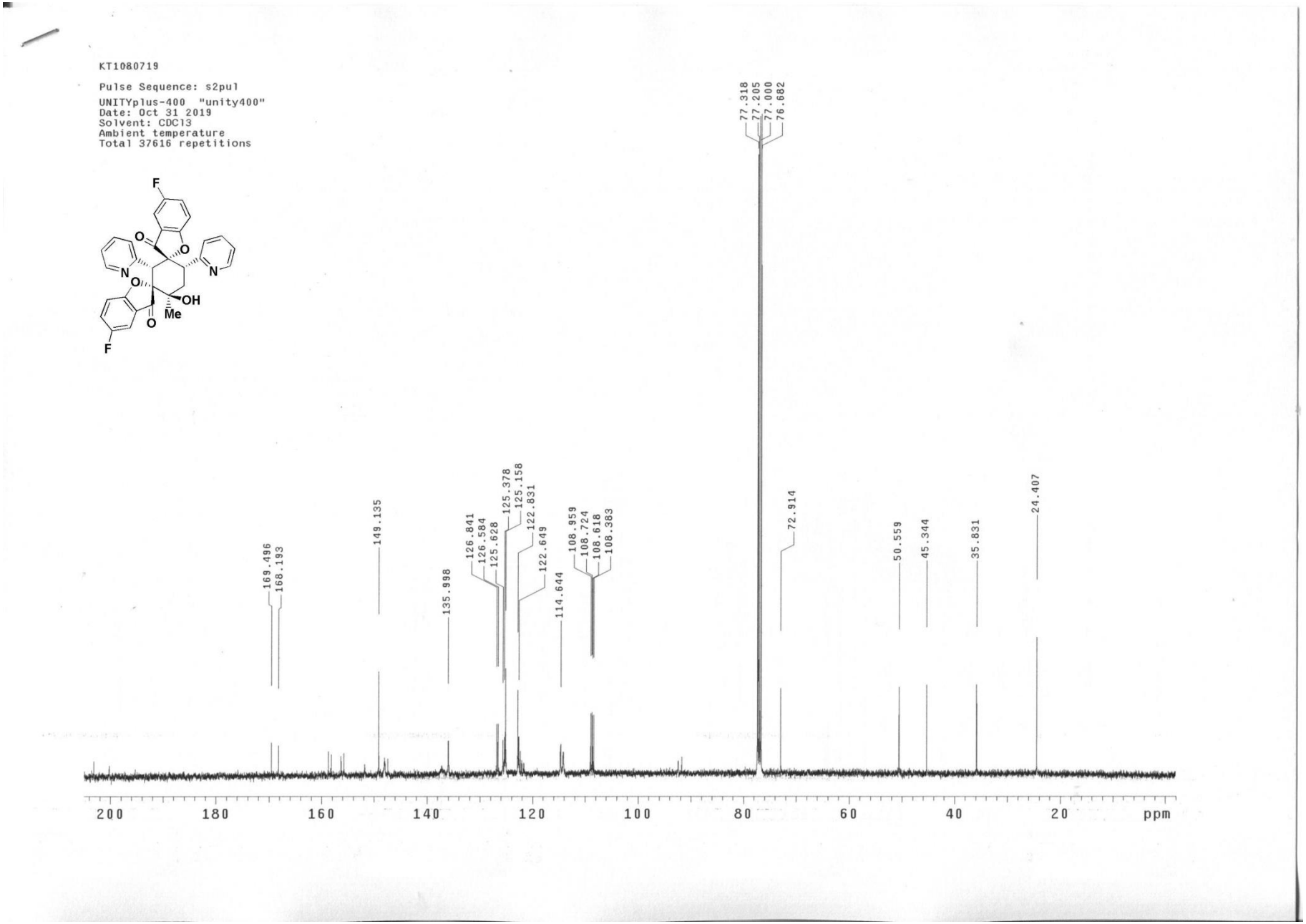




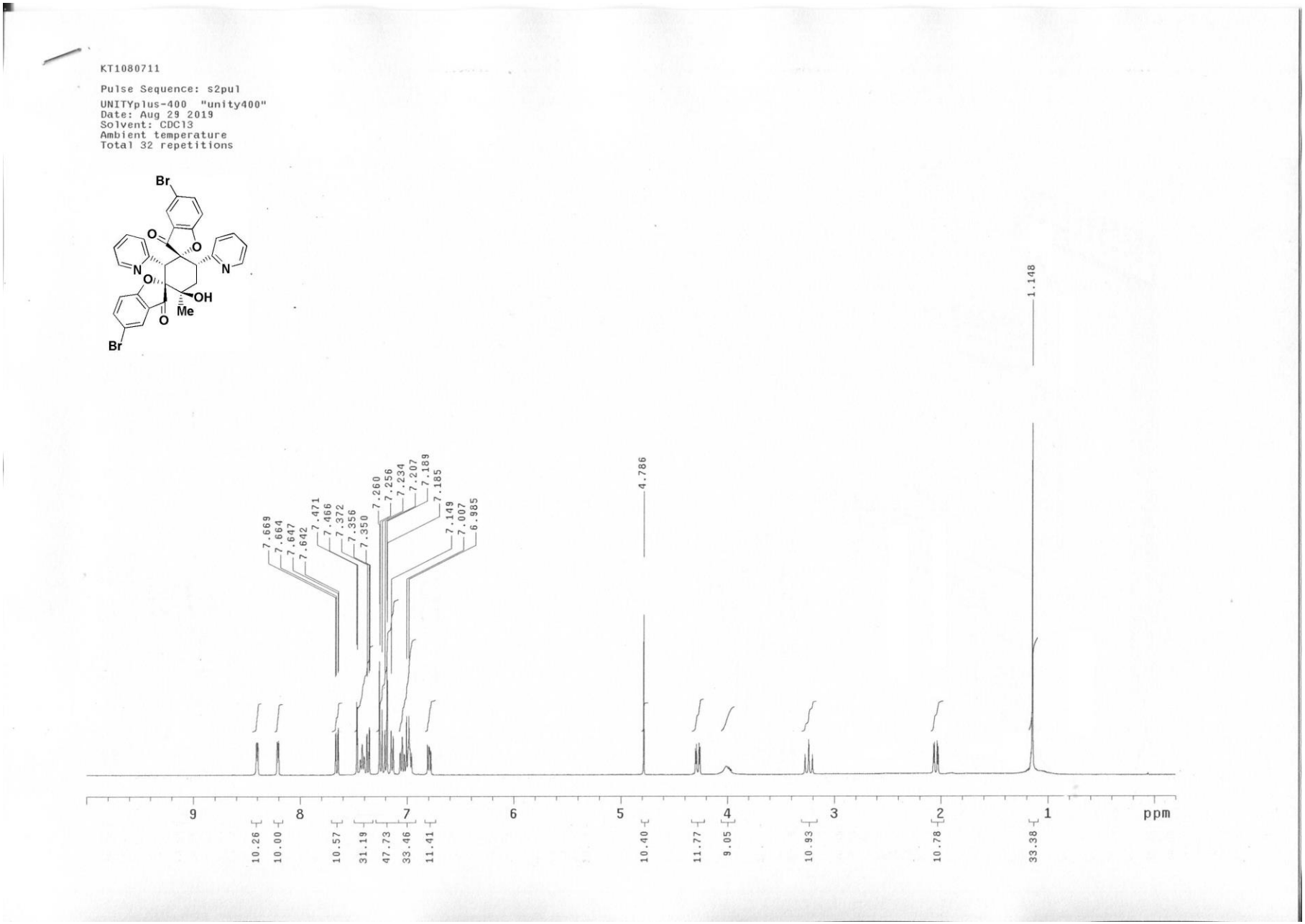
Compound 3k (^{1}H-NMR spectral data)



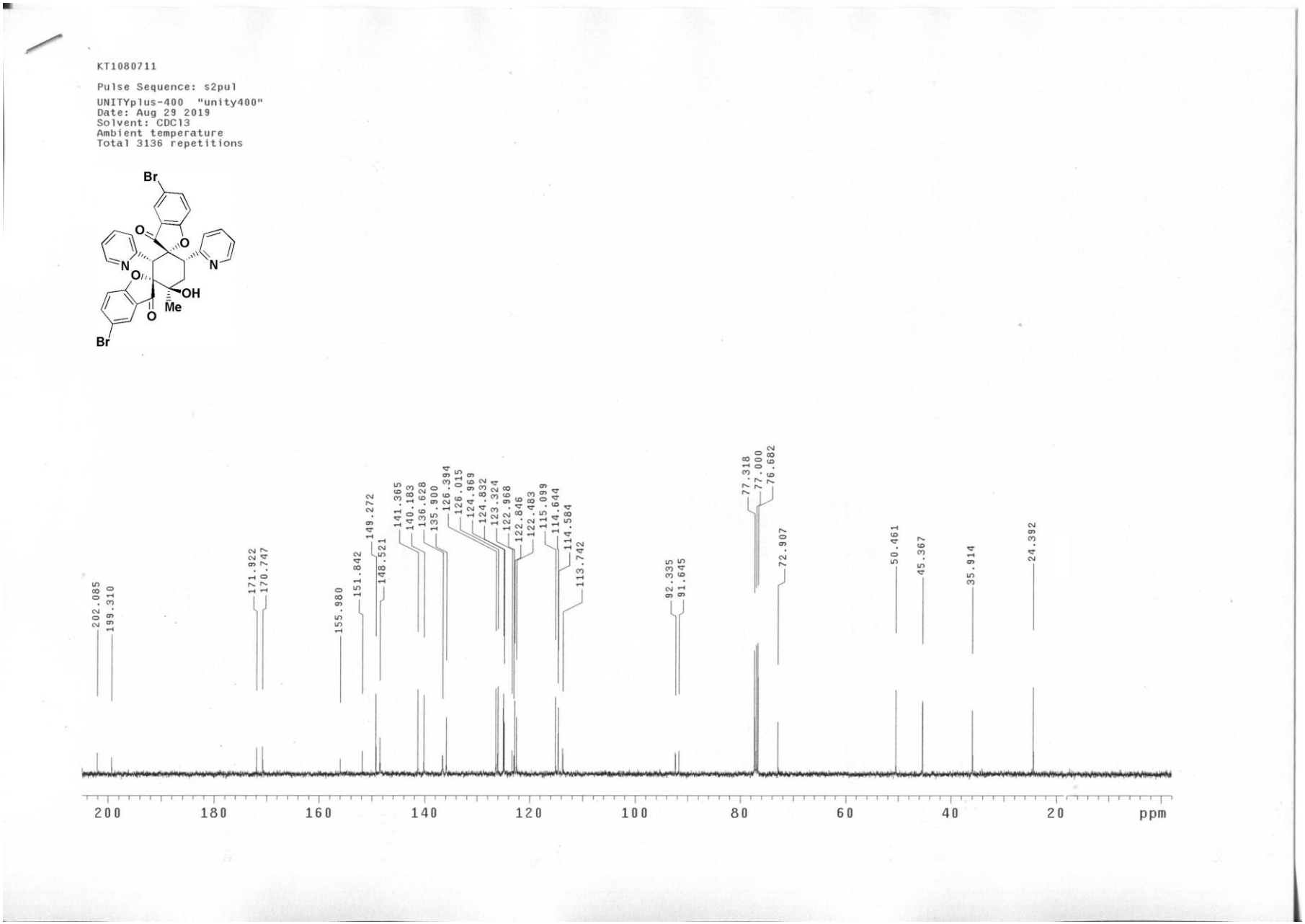
Compound 3k (^{13}C-NMR spectral data)



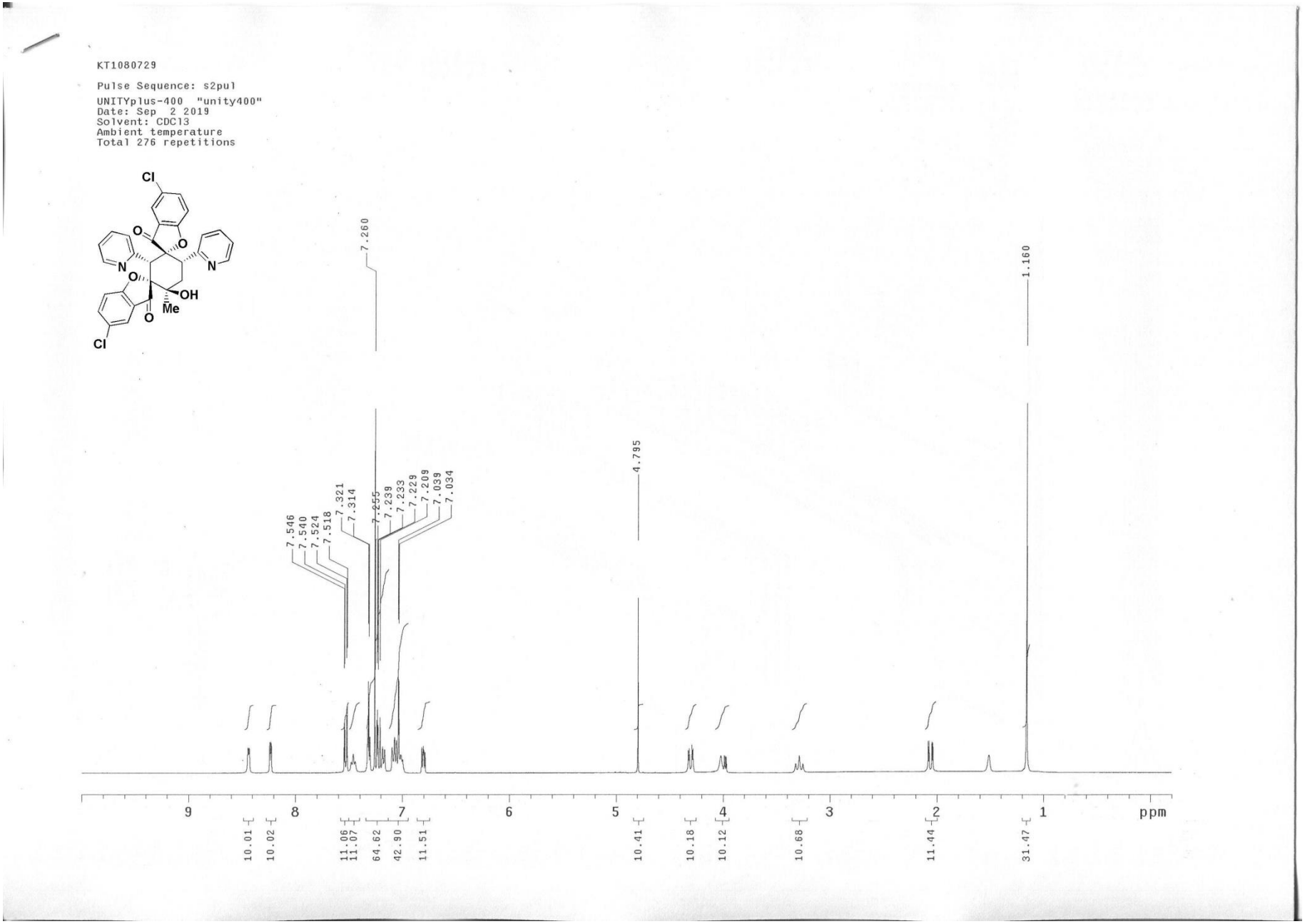
Compound 3l (^{1}H-NMR spectral data)



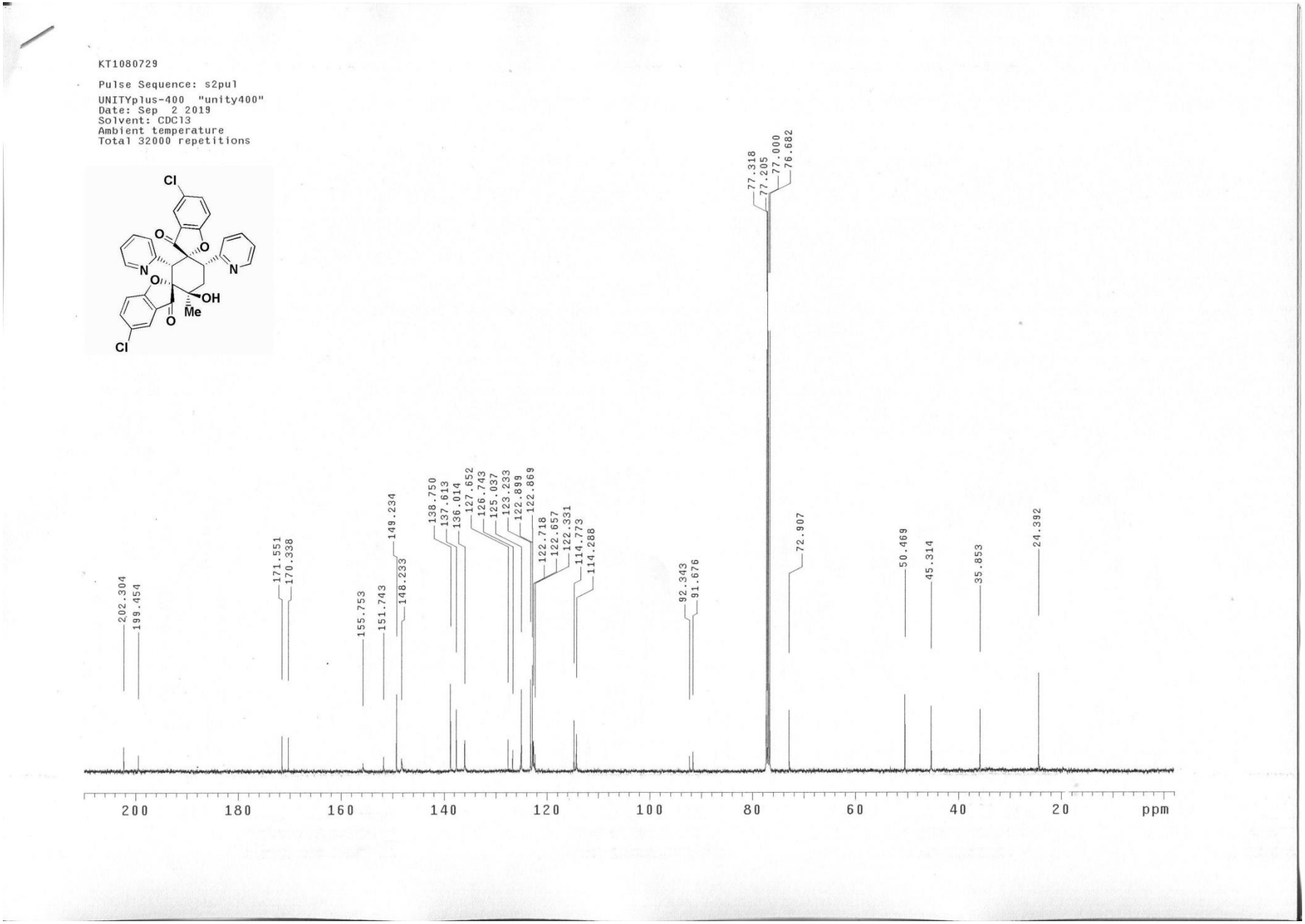
Compound 3l (^{13}C-NMR spectral data)



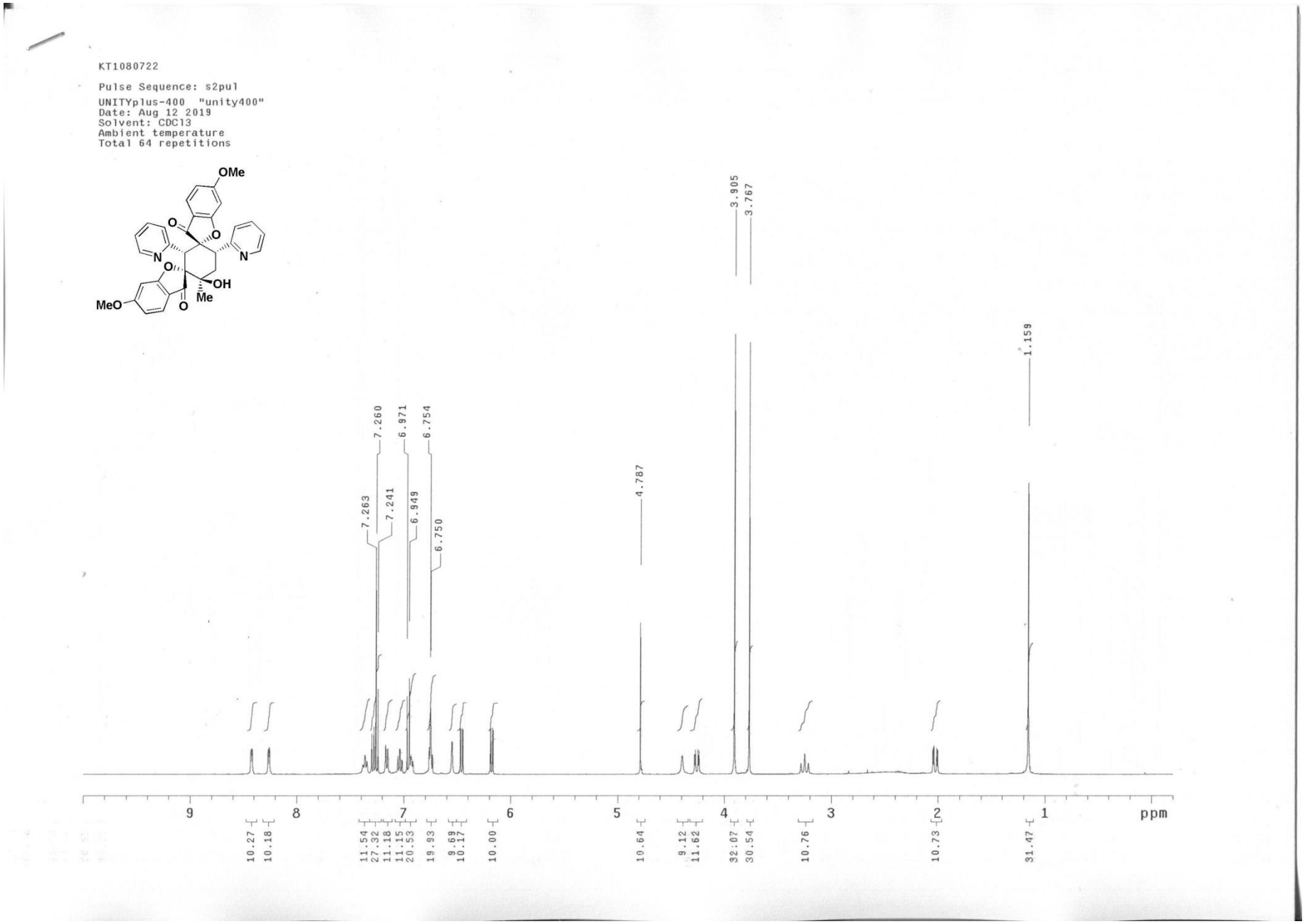
Compound 3m (^{1}H-NMR spectral data)



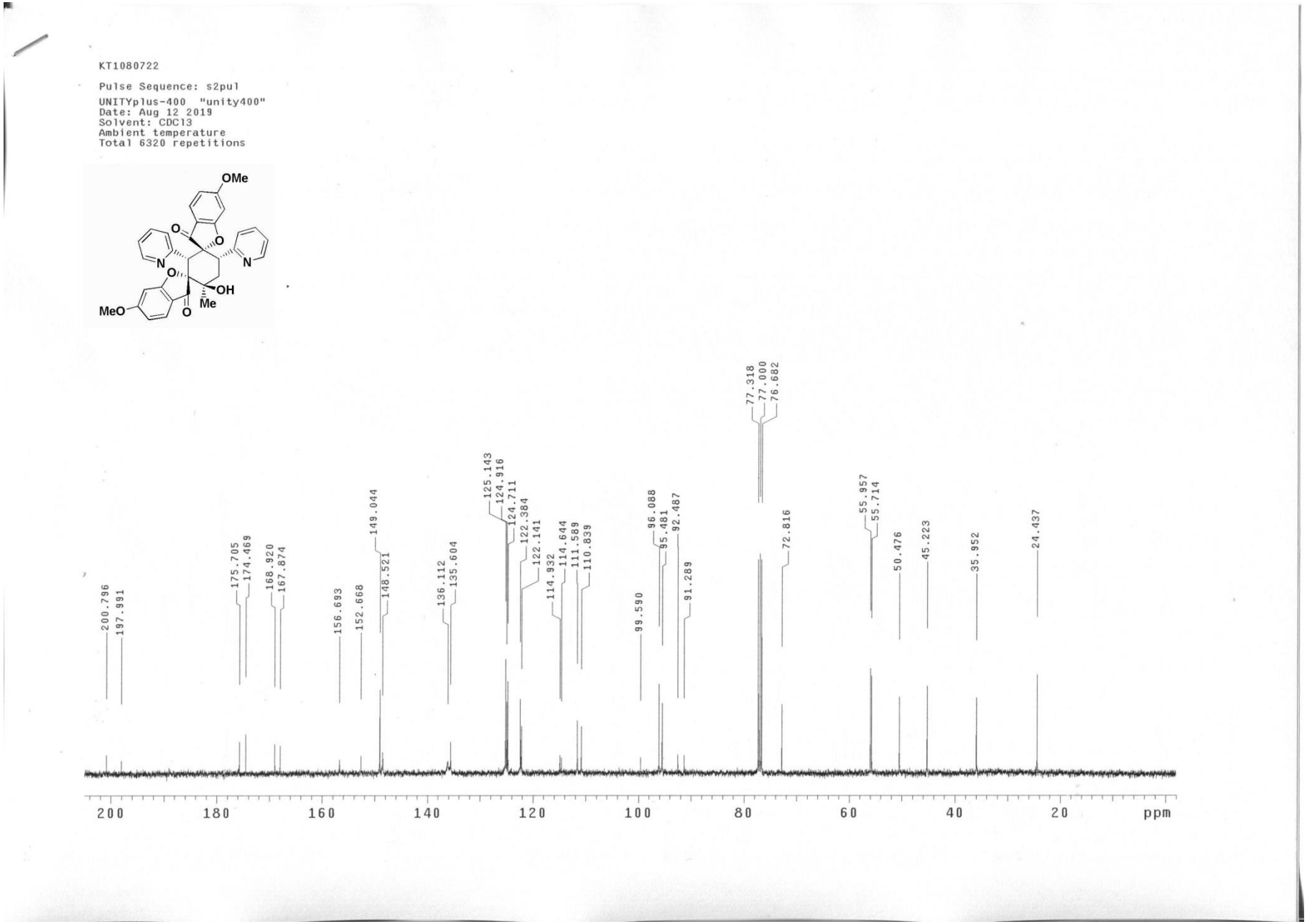
Compound 3m (^{13}C-NMR spectral data)



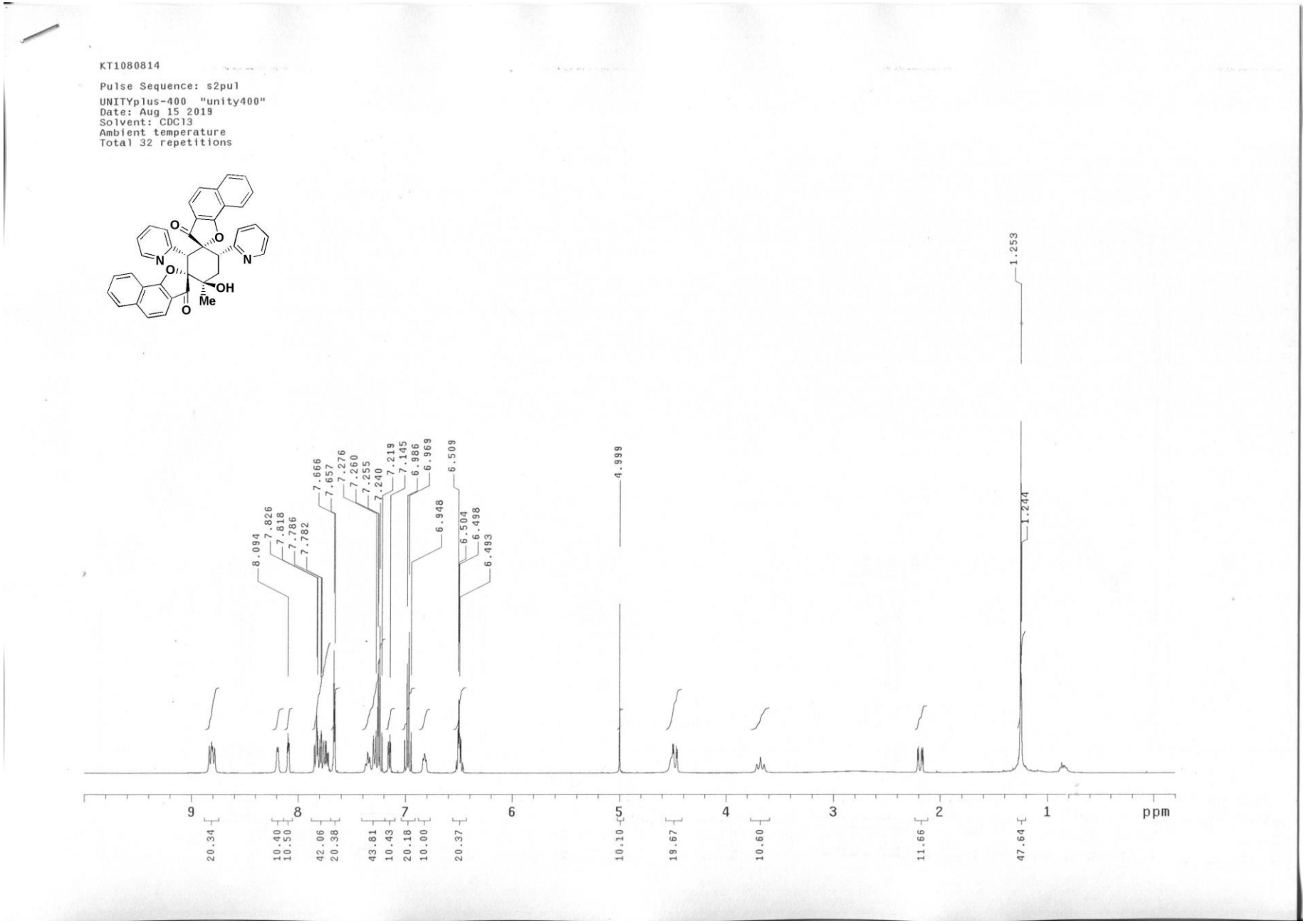
Compound 3n (^{1}H-NMR spectral data)



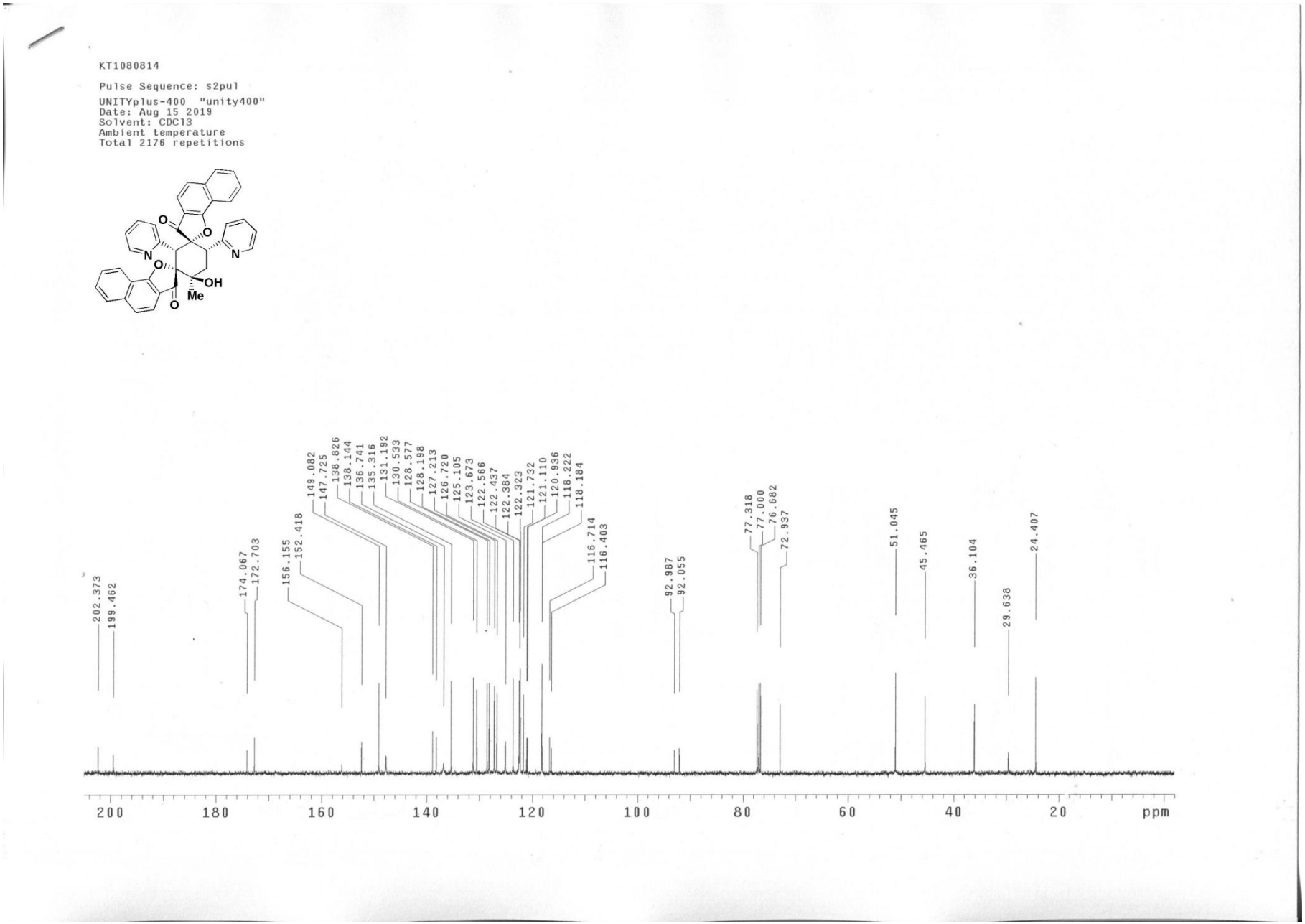
Compound 3n (^{13}C-NMR spectral data)



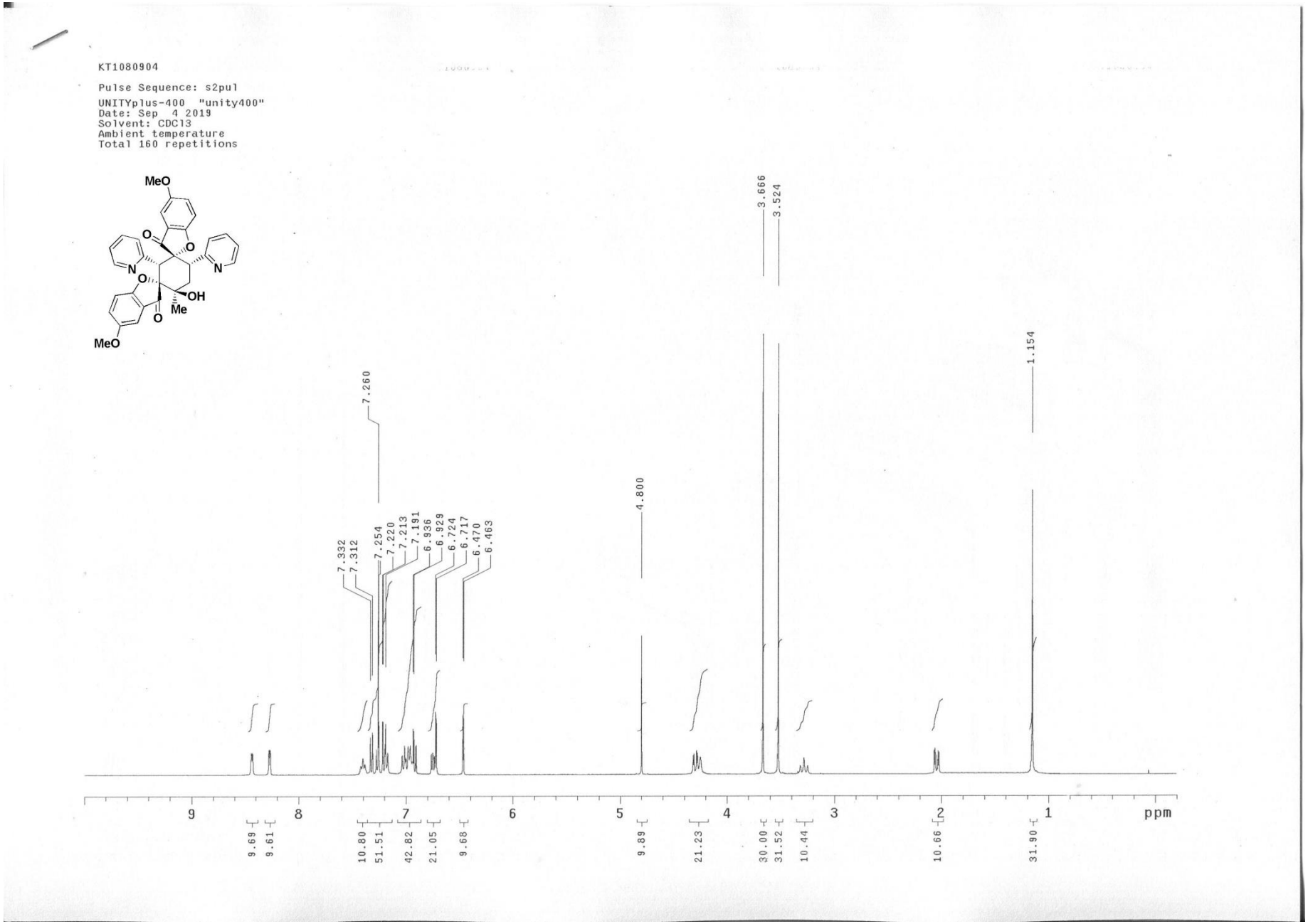
Compound 3o (^{1}H-NMR spectral data)



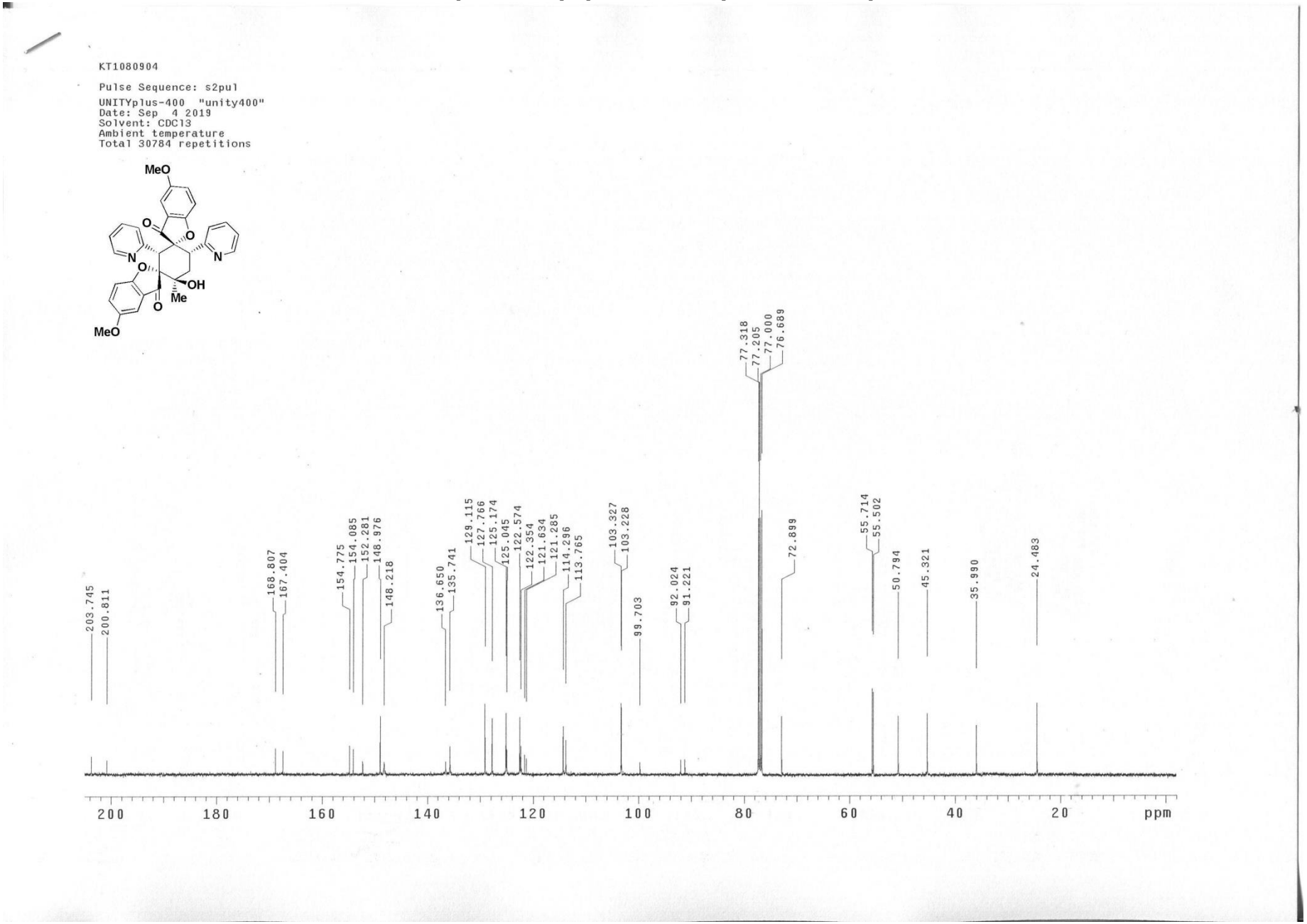
Compound 3o (^{13}C-NMR spectral data)



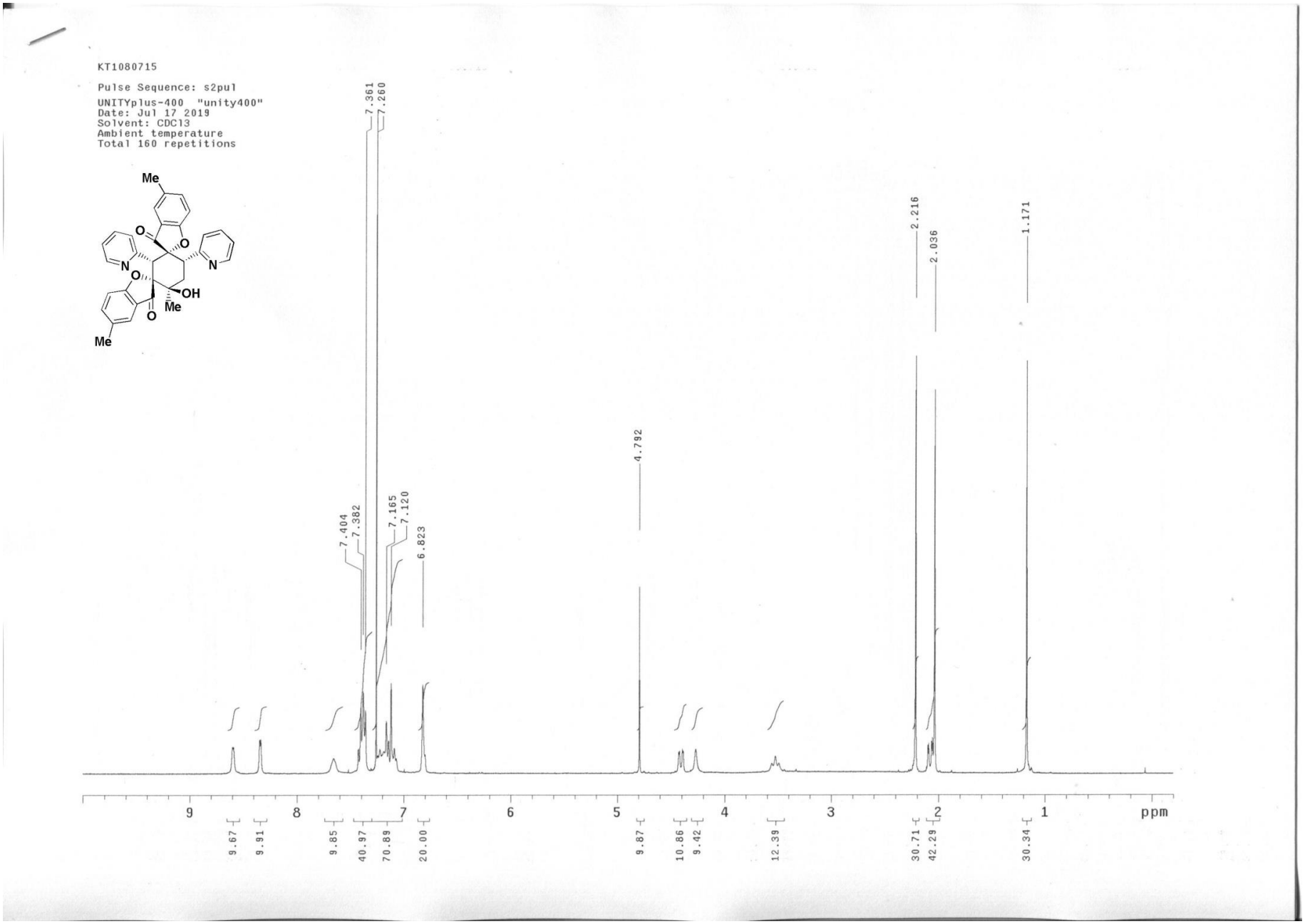
Compound 3p (^{1}H-NMR spectral data)



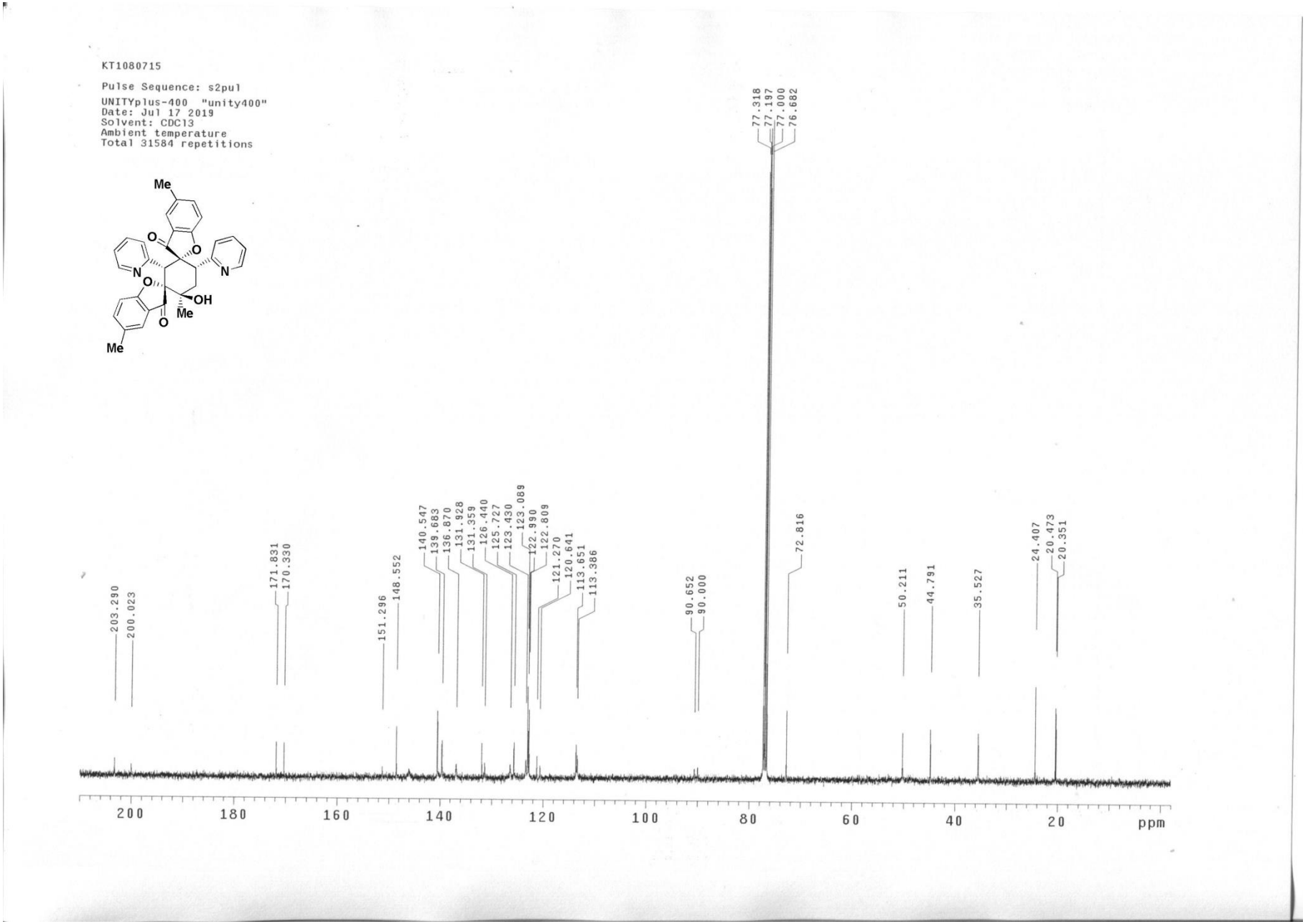
Compound 3p (^{13}C-NMR spectral data)



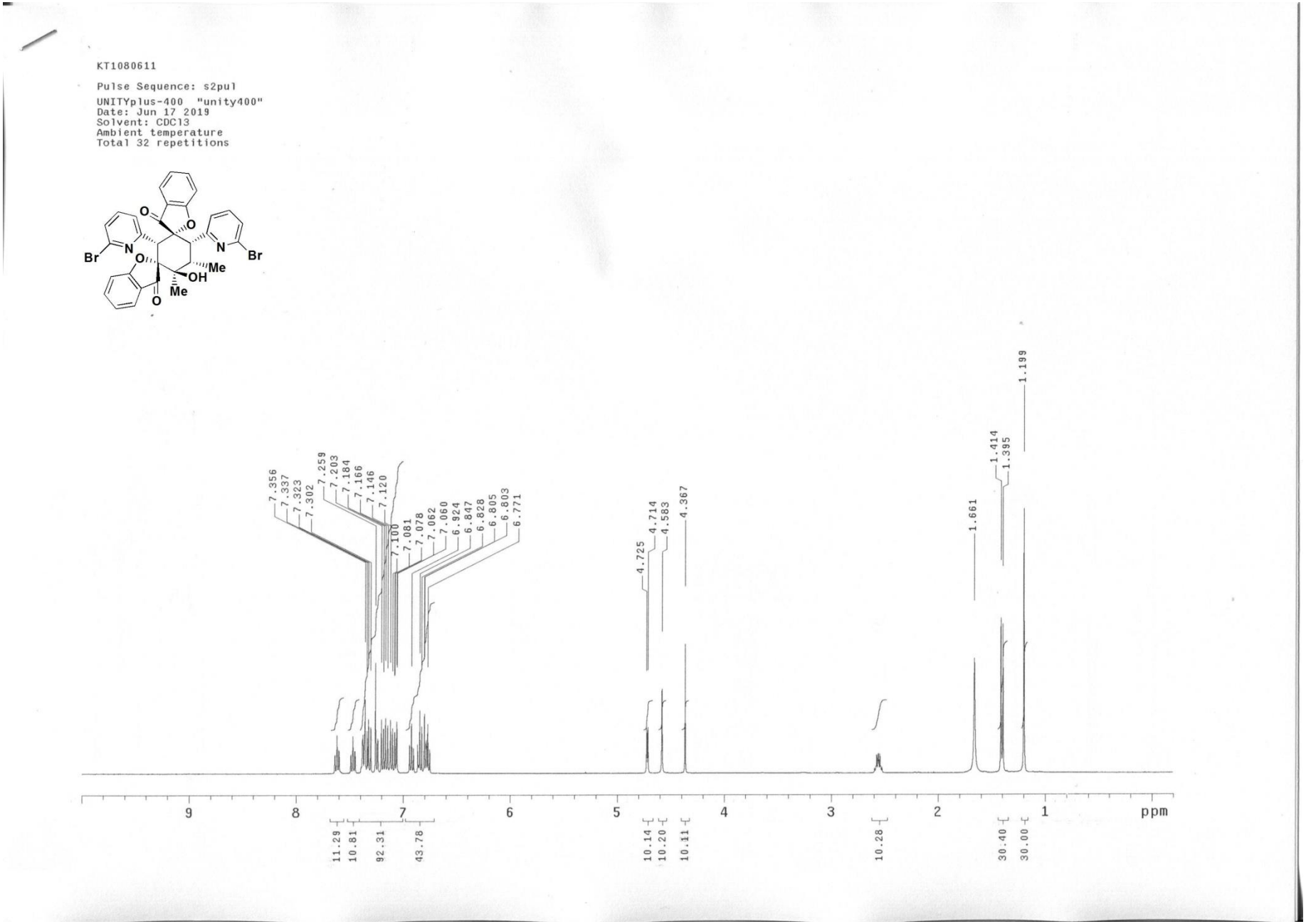
Compound 3q (^{1}H-NMR spectral data)



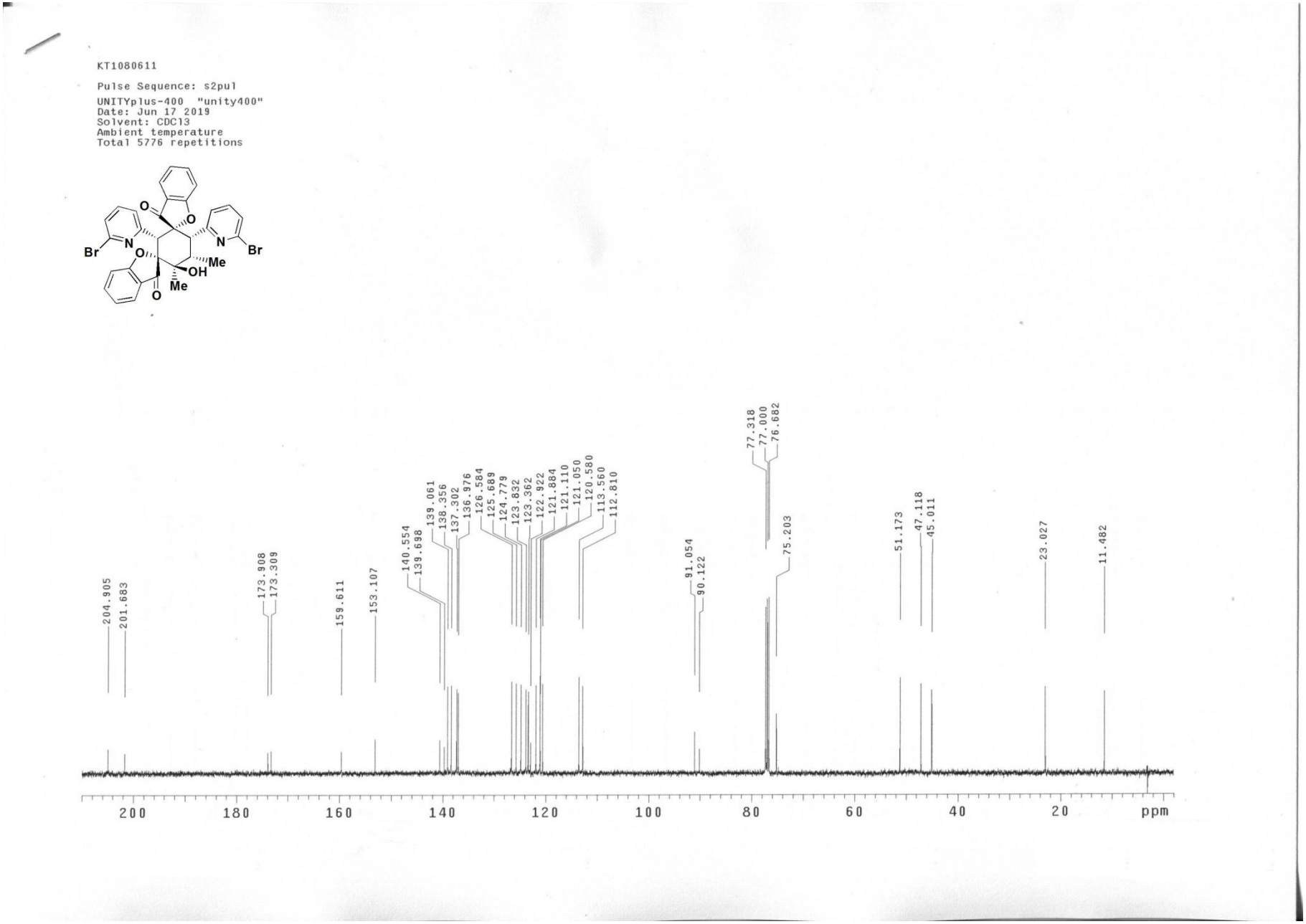
Compound 3q (^{13}C-NMR spectral data)



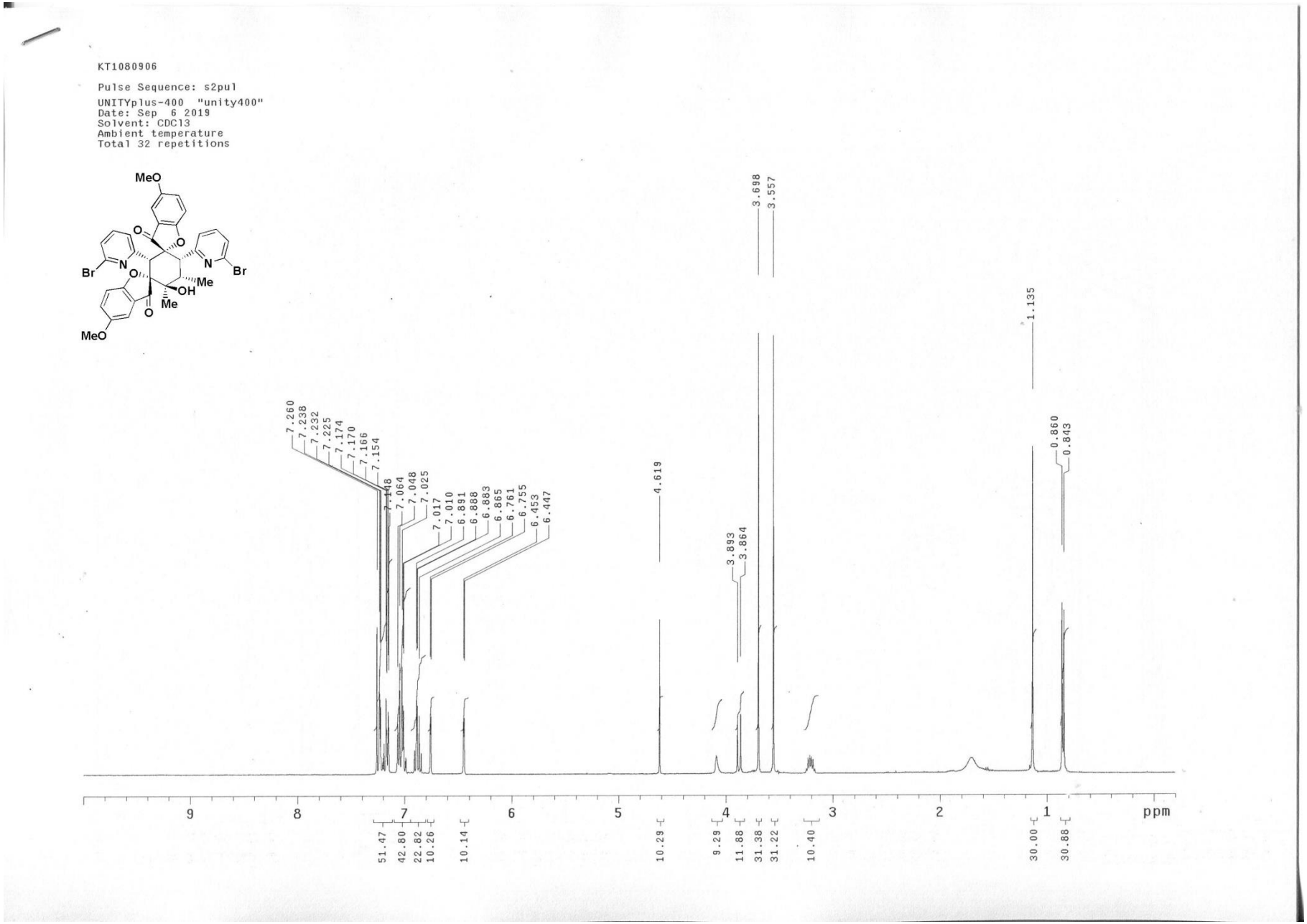
Compound 3r (^{1}H-NMR spectral data)



Compound 3r (^{13}C-NMR spectral data)



Compound 3s (^{1}H-NMR spectral data)

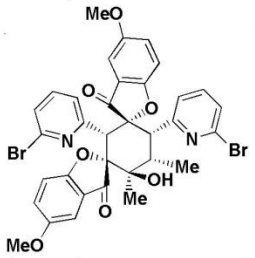


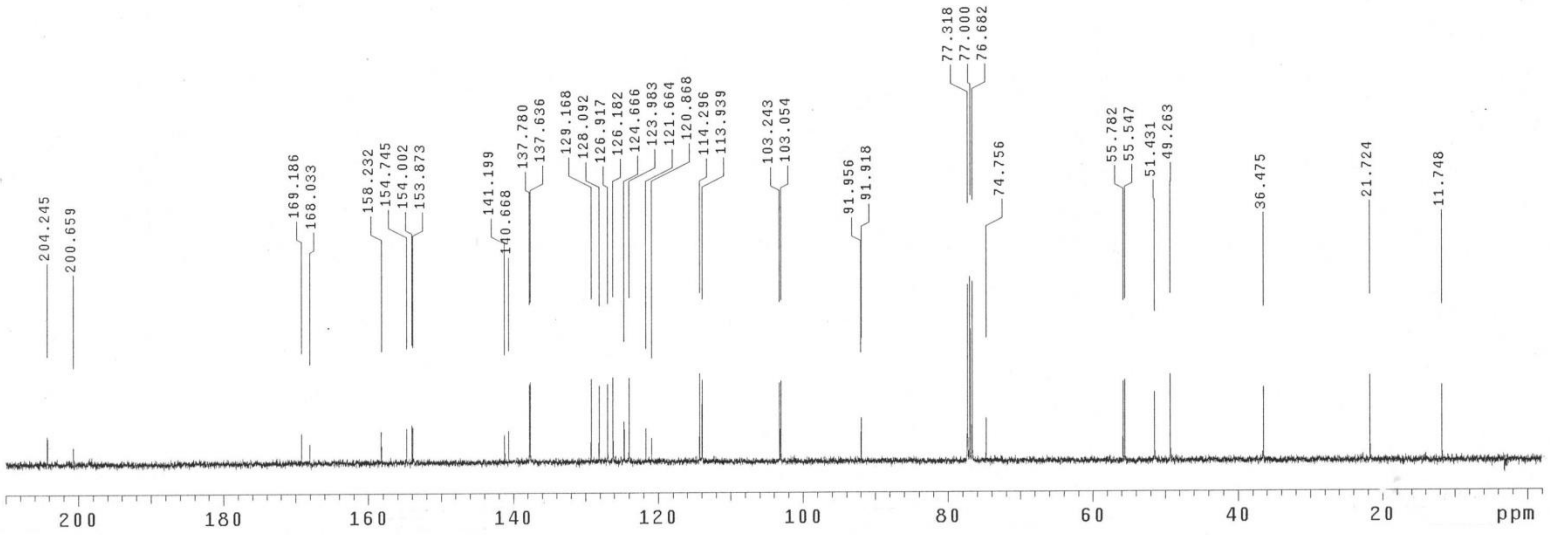
Compound 3s (^{13}C-NMR spectral data)

KT1080906

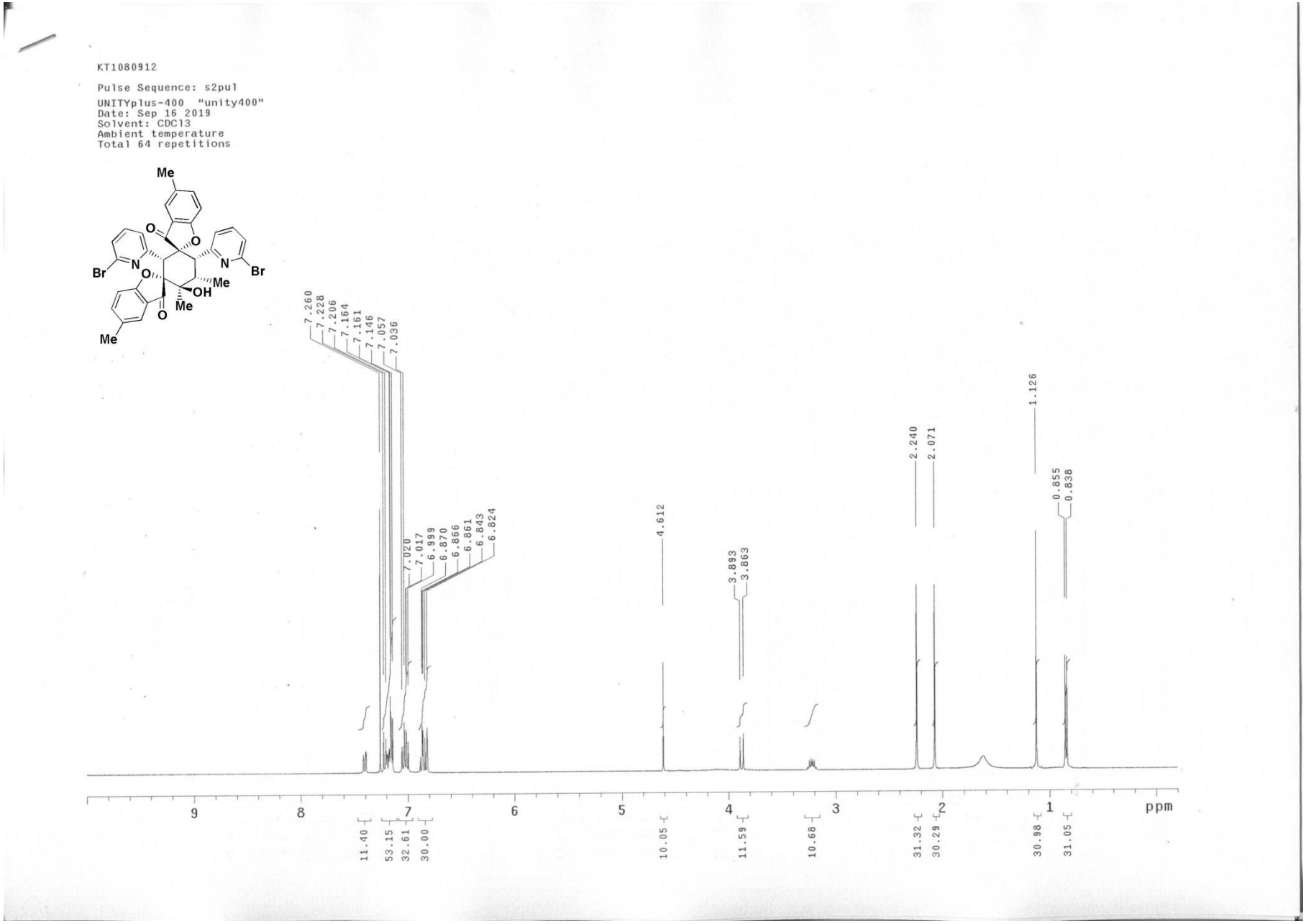
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Sep 6 2019
Solvent: CDCl3
Ambient temperature
Total 4400 repetitions

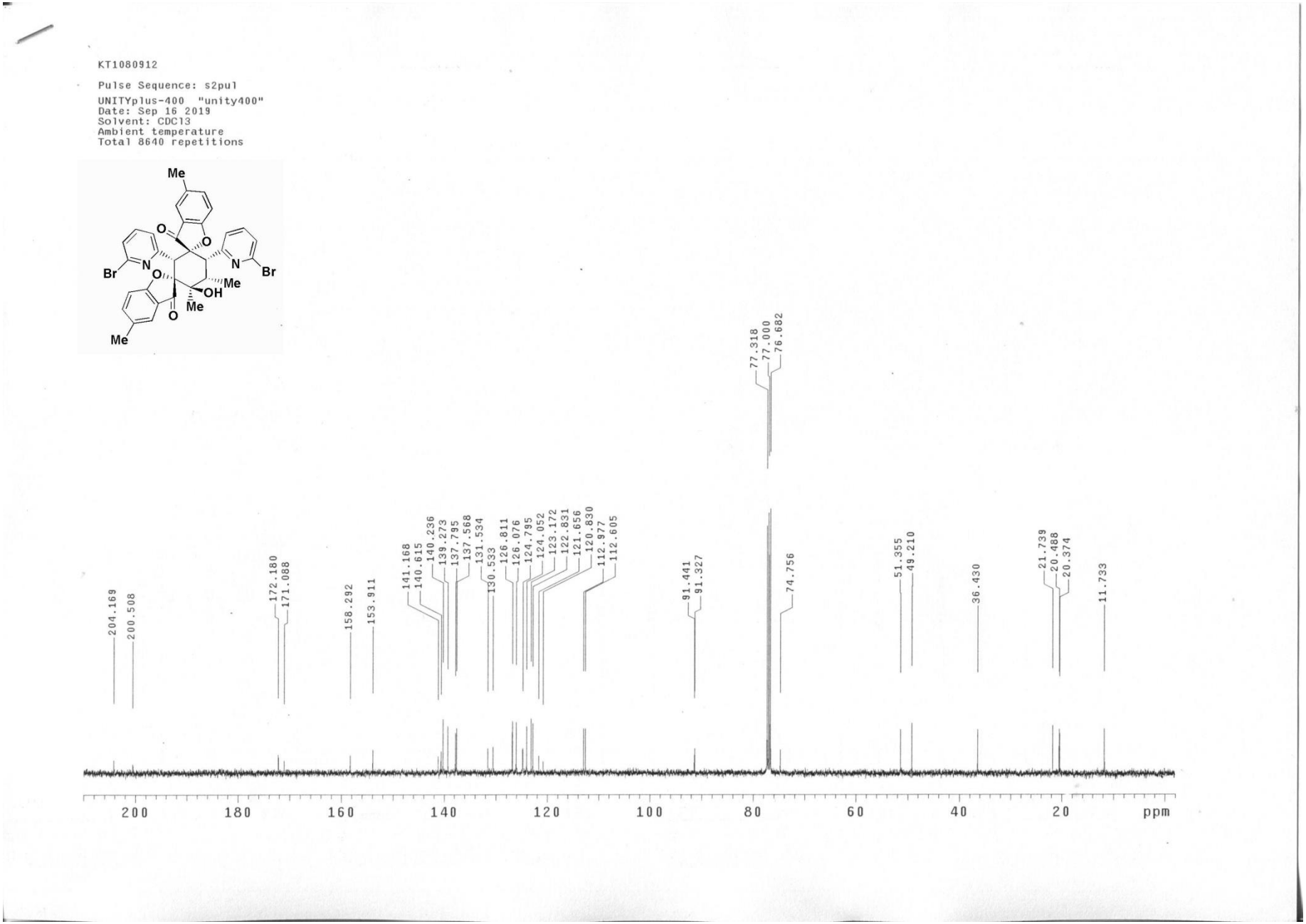




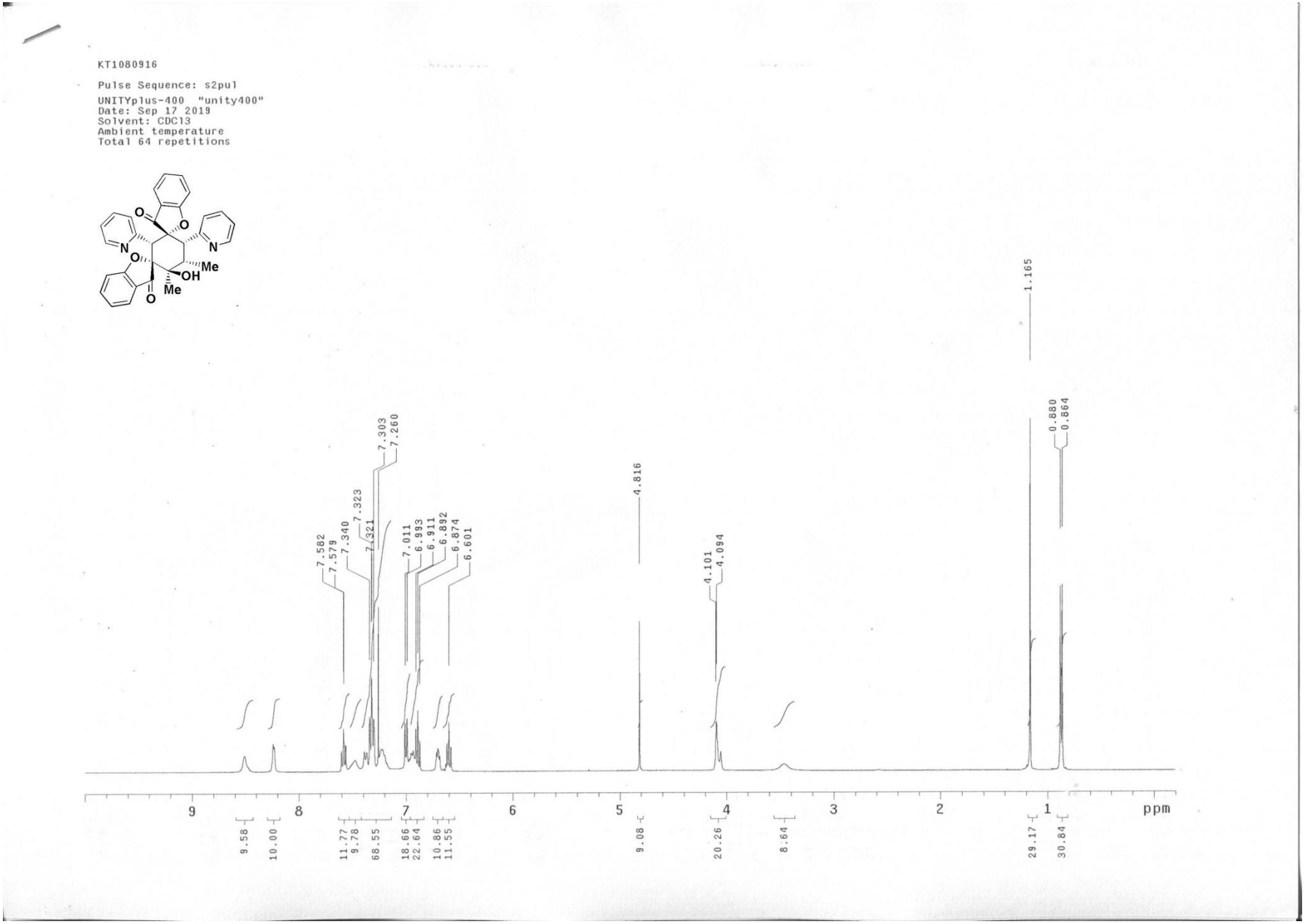
Compound 3t (^{1}H-NMR spectral data)



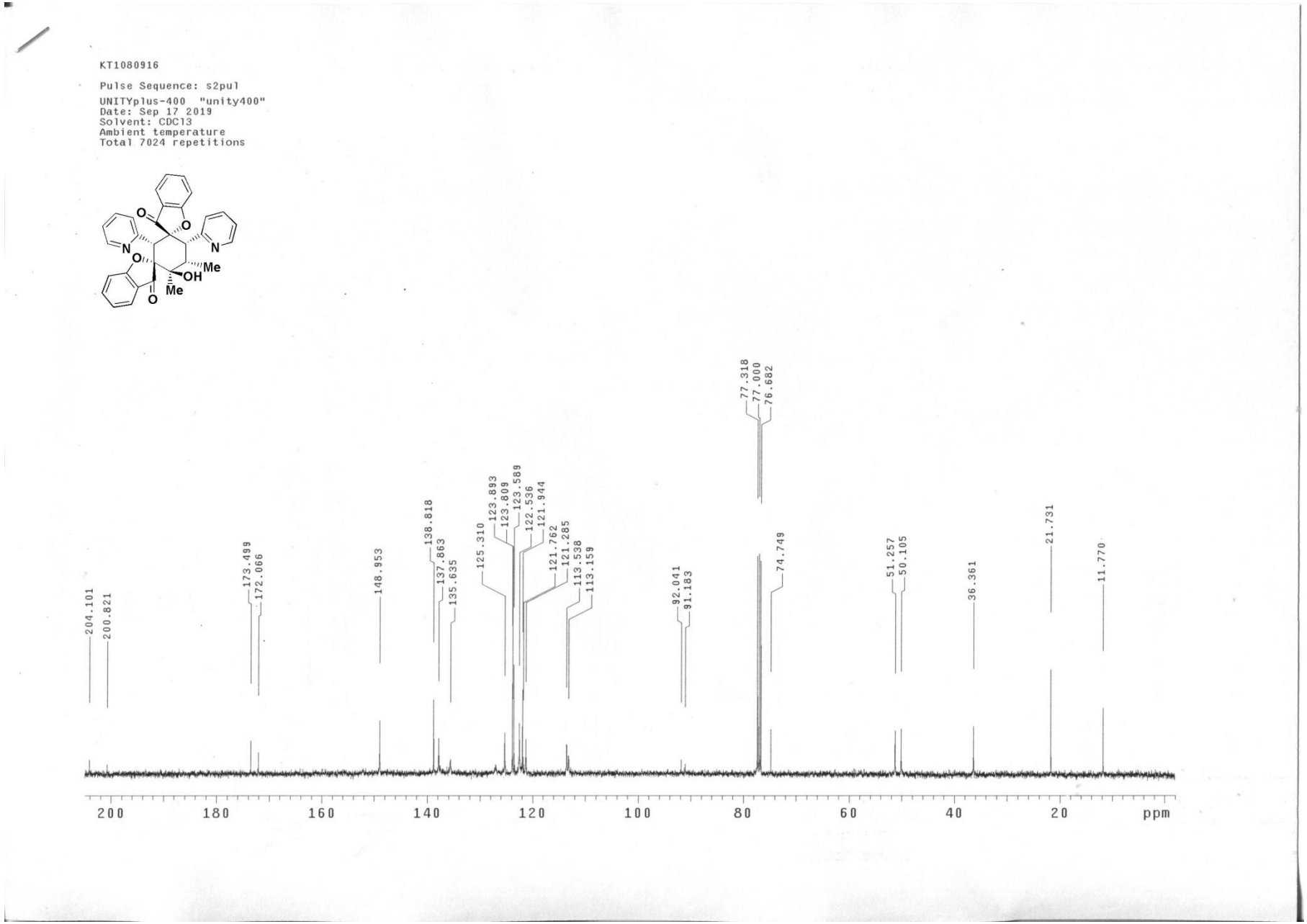
Compound 3t (^{13}C-NMR spectral data)



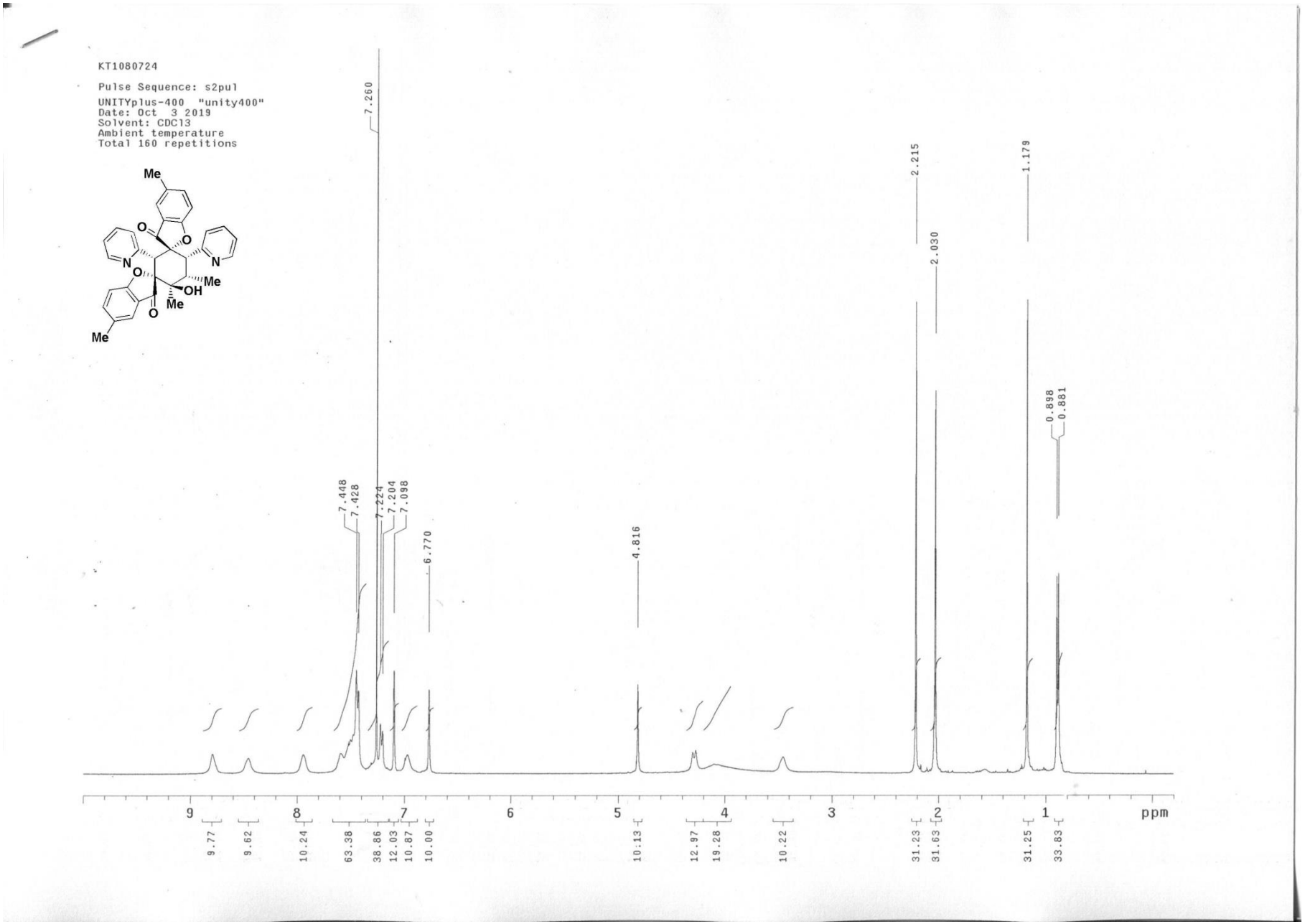
Compound 3u (^{1}H-NMR spectral data)



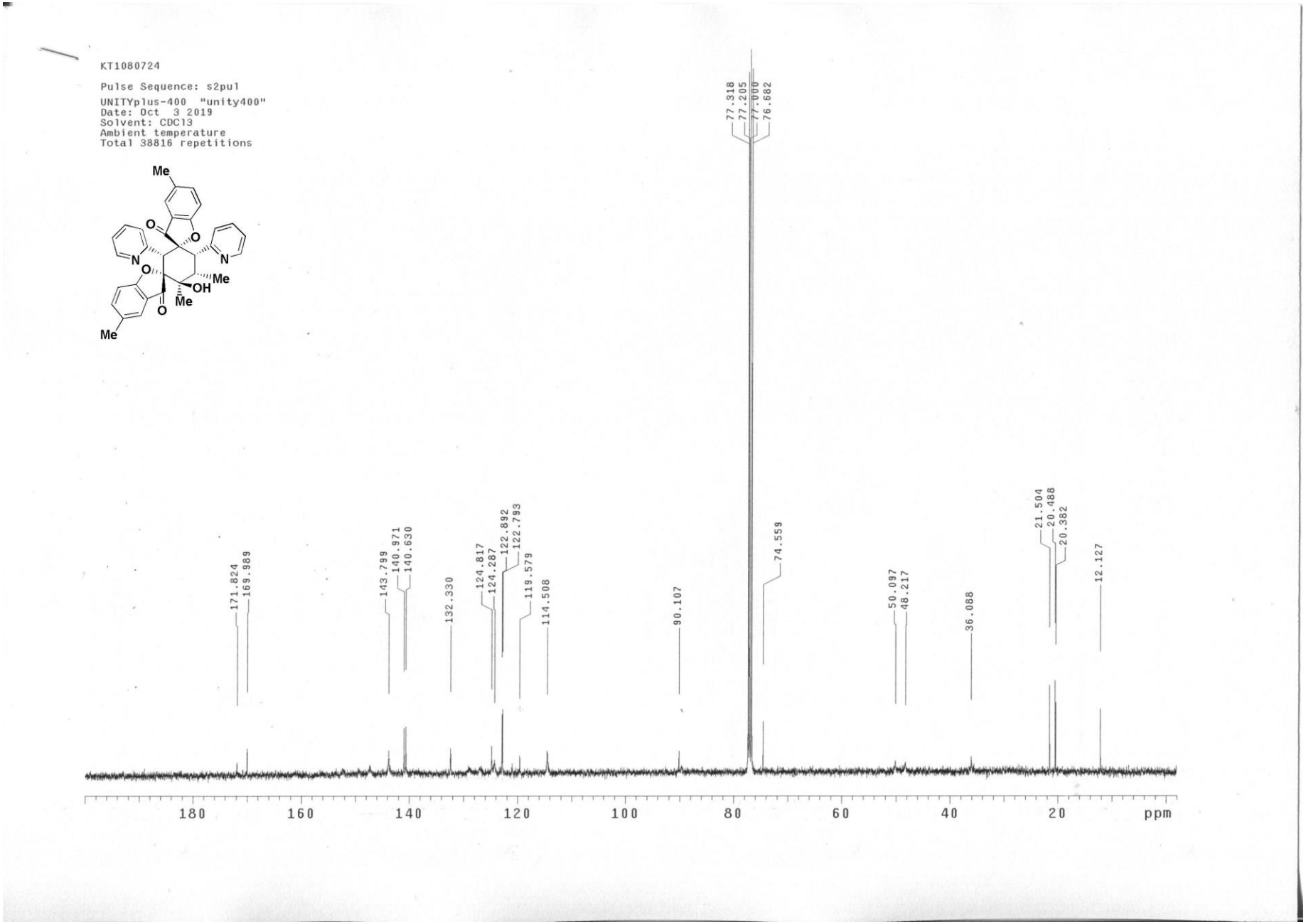
Compound 3u (^{13}C-NMR spectral data)



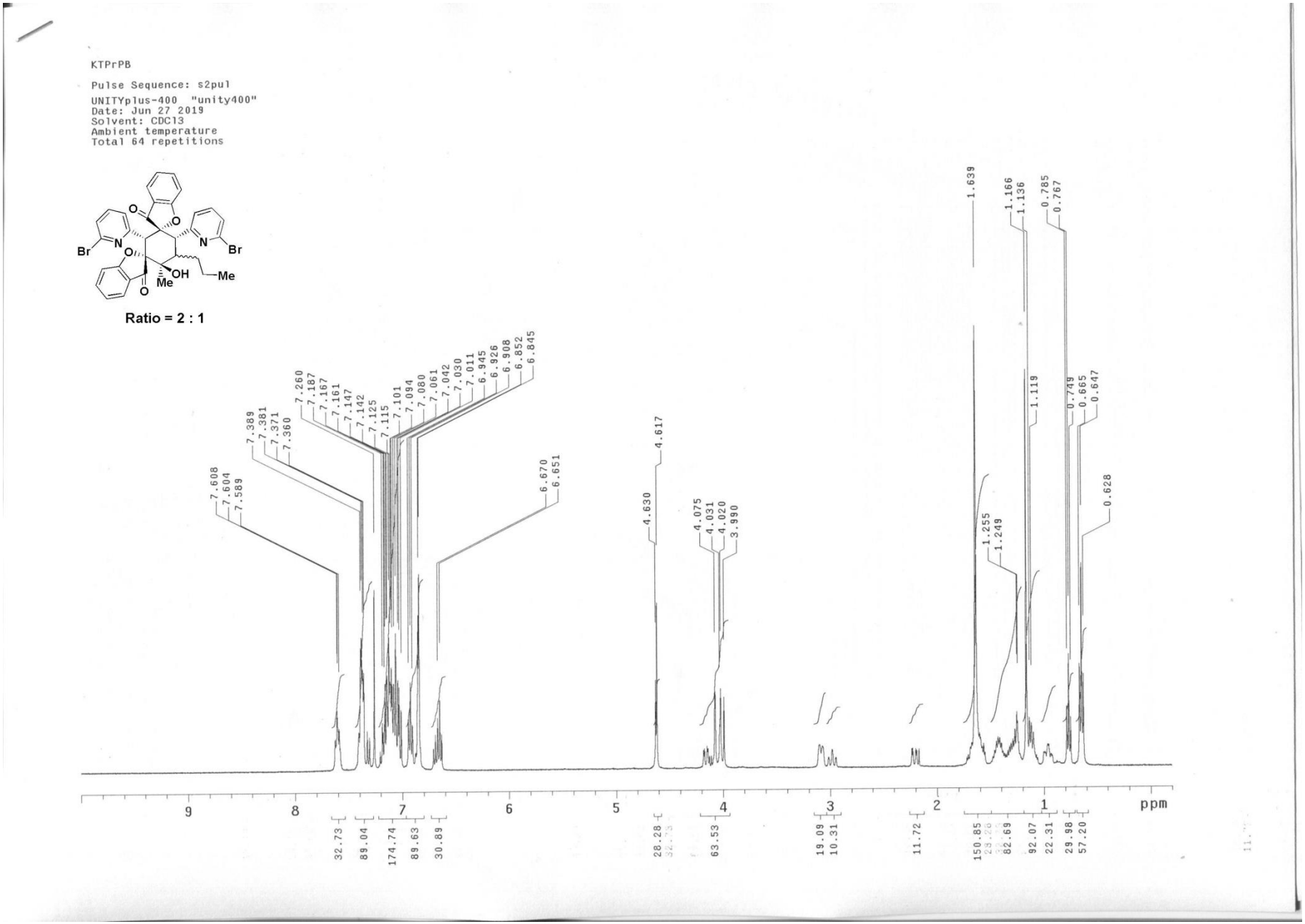
Compound 3v (^{1}H-NMR spectral data)



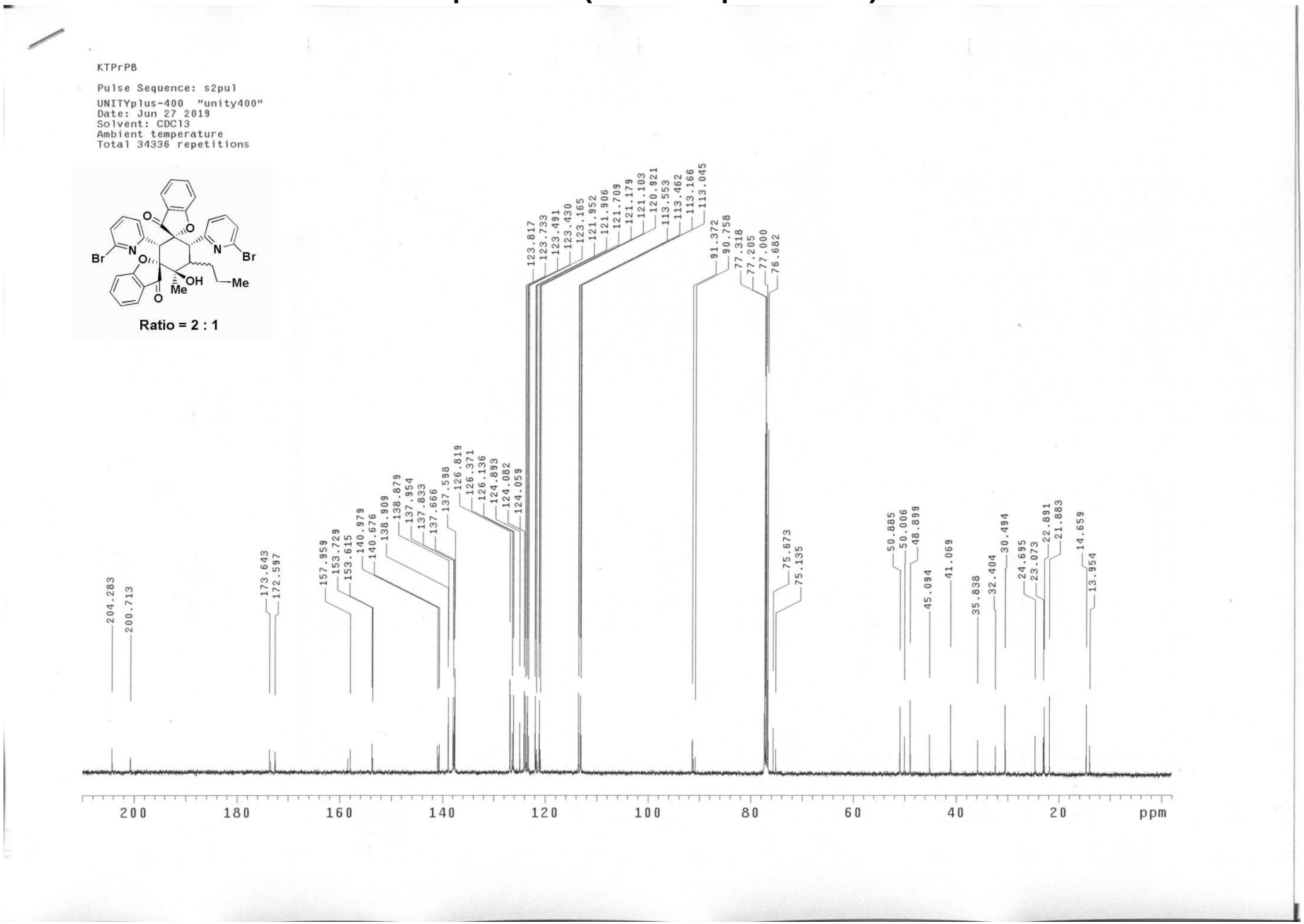
Compound 3v (^{13}C-NMR spectral data)



Compound 3w (^{1}H-NMR spectral data)



Compound 3w (^{13}C-NMR spectral data)

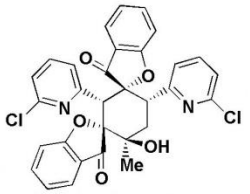


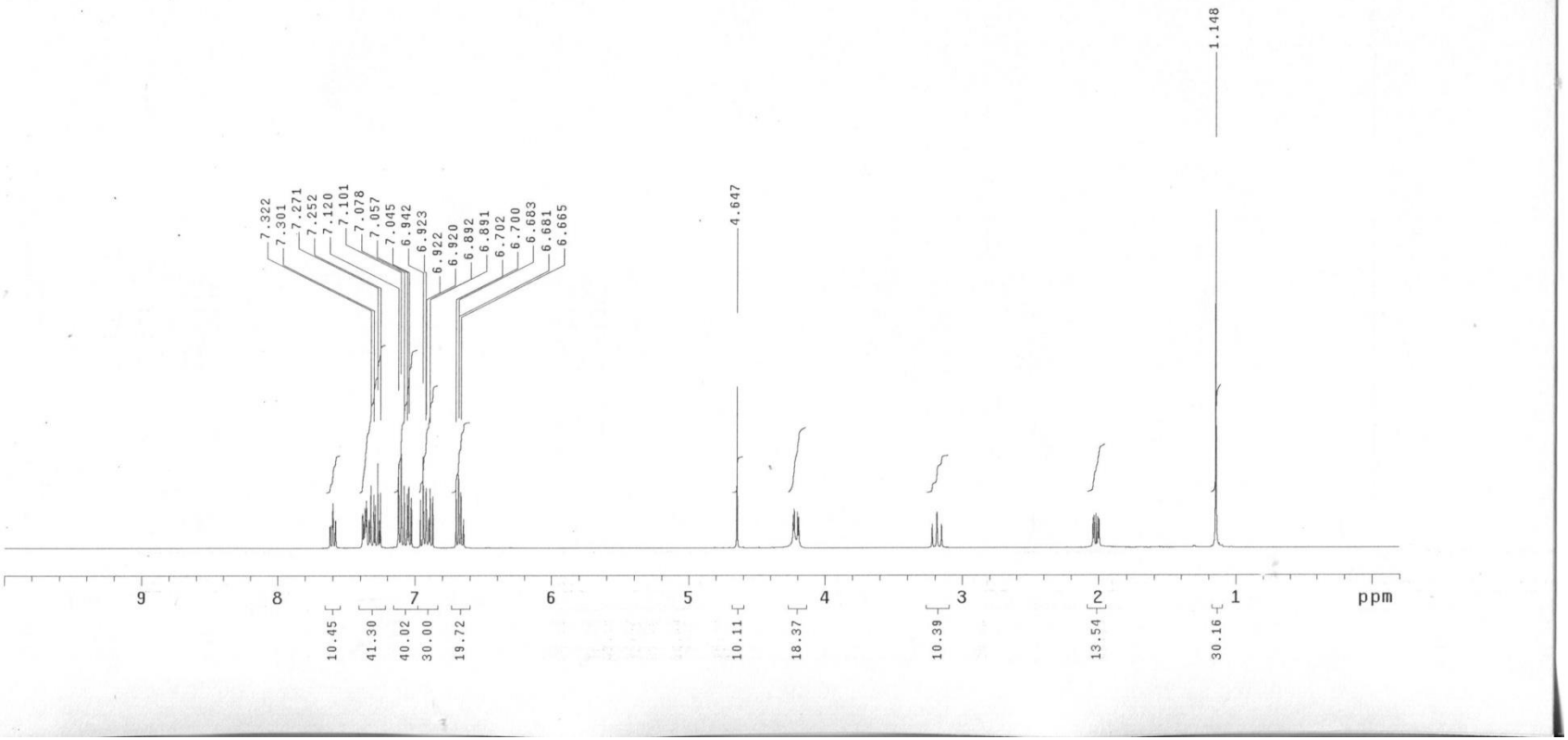
Compound 3y (^{1}H-NMR spectral data)

KT1081003

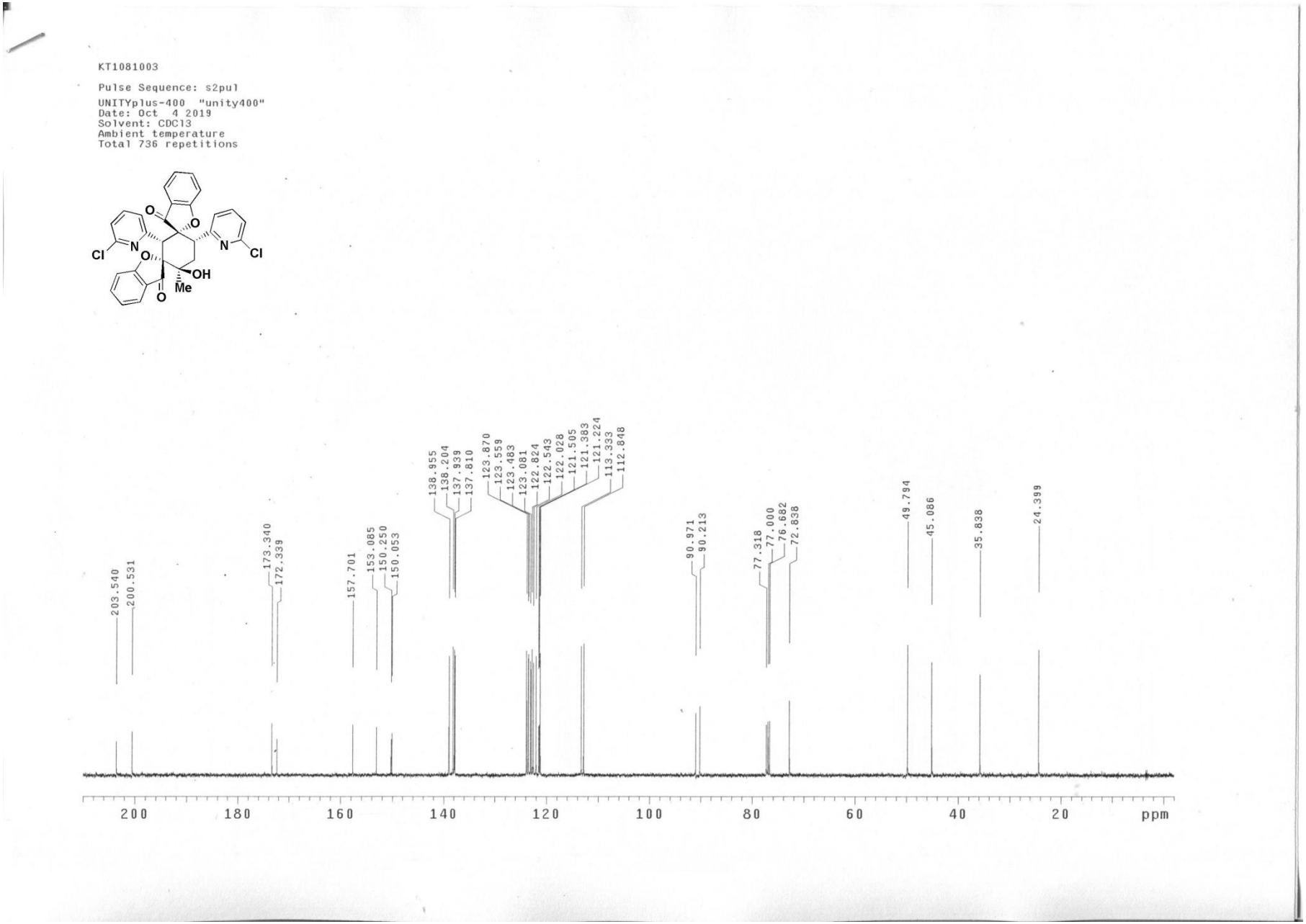
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Oct 4 2019
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

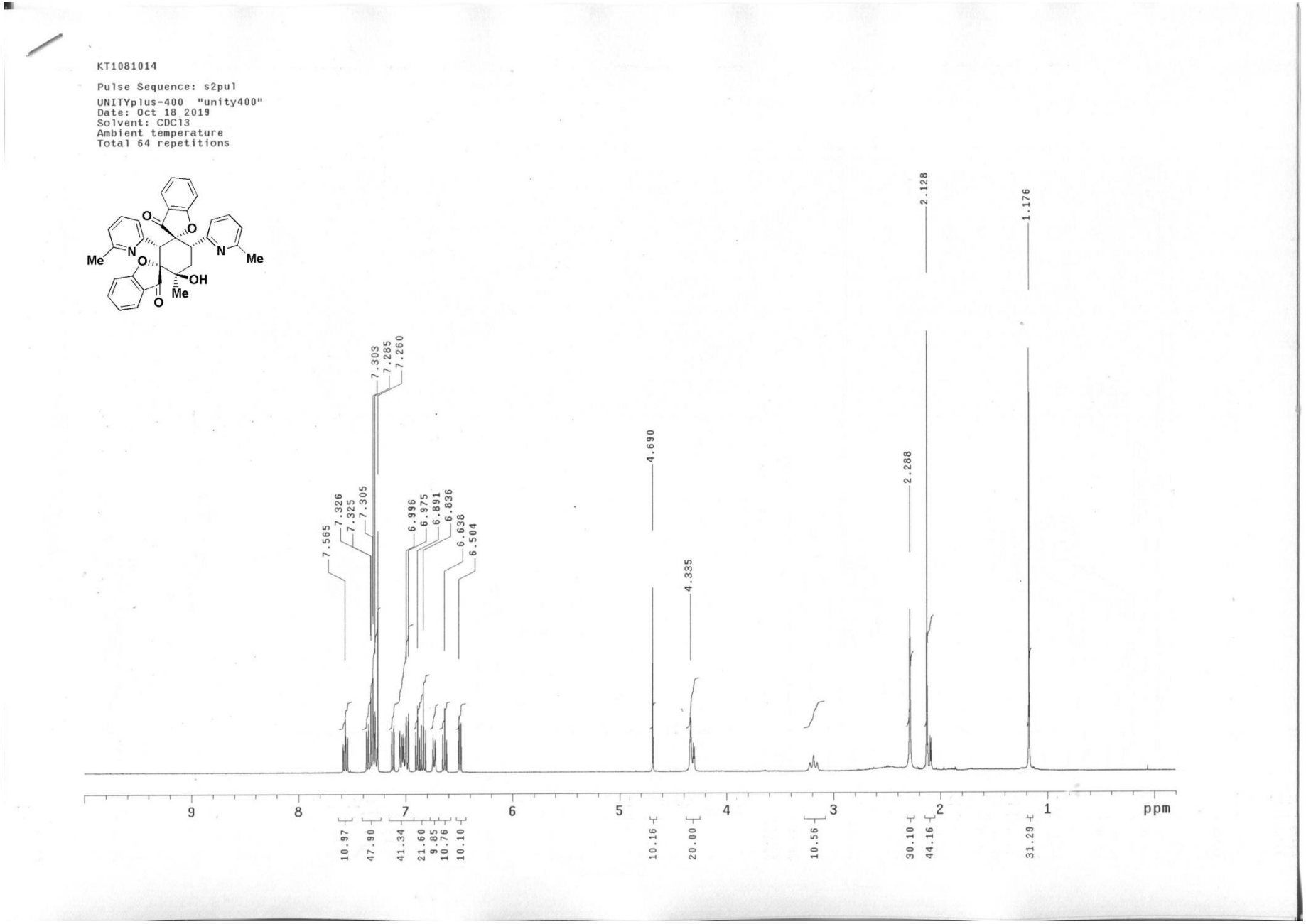




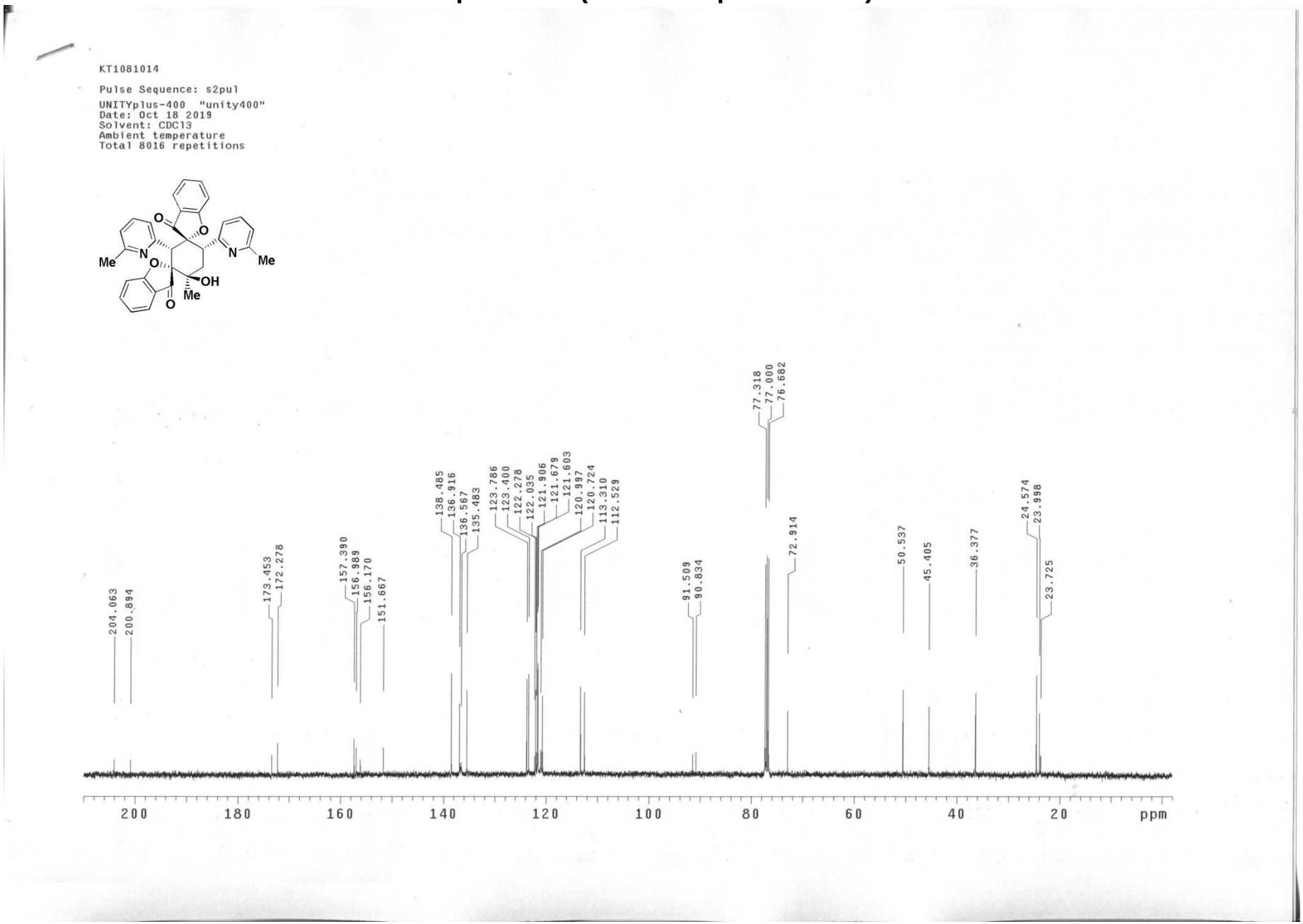
Compound 3y (^{13}C-NMR spectral data)



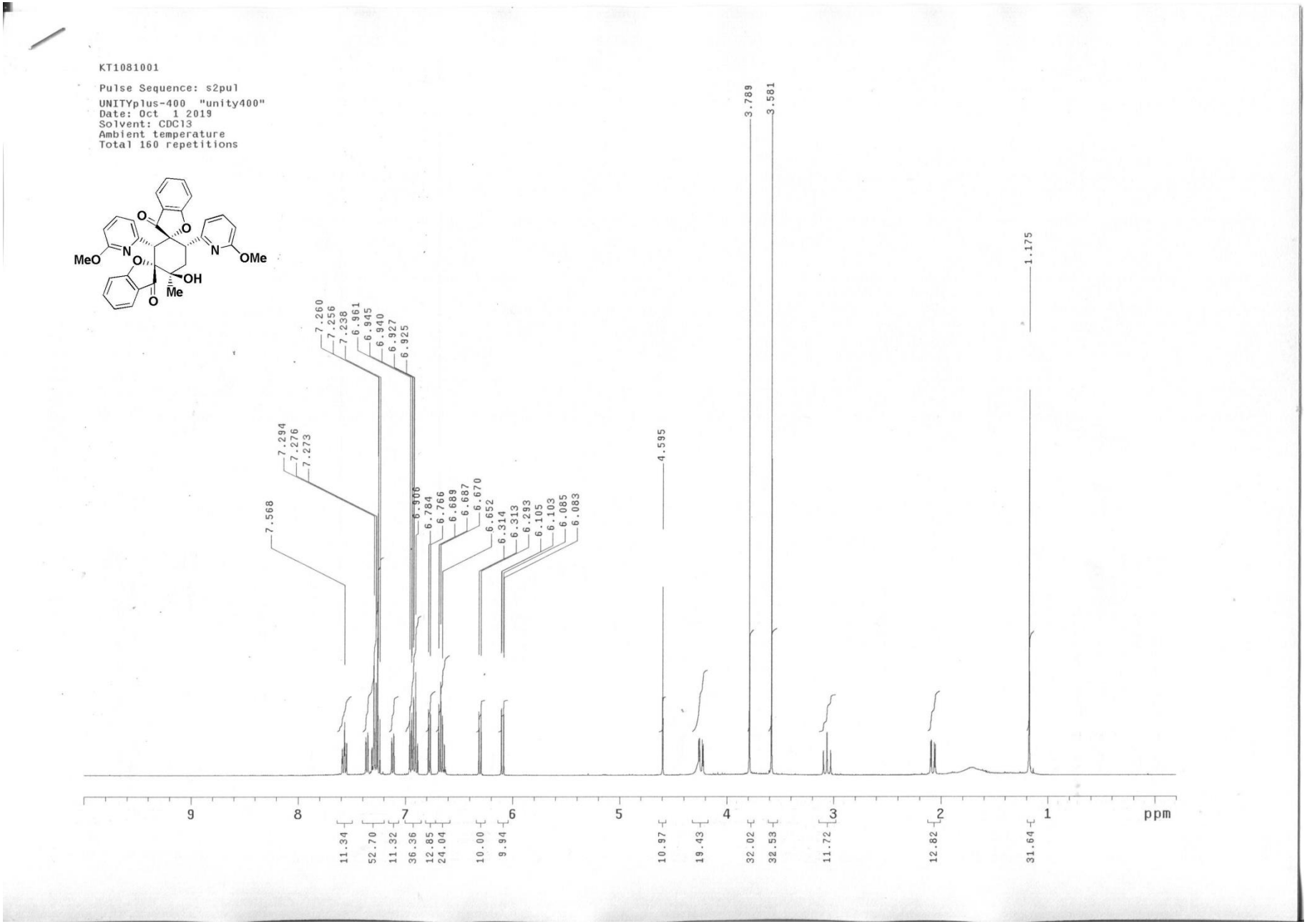
Compound 3z (^{1}H-NMR spectral data)



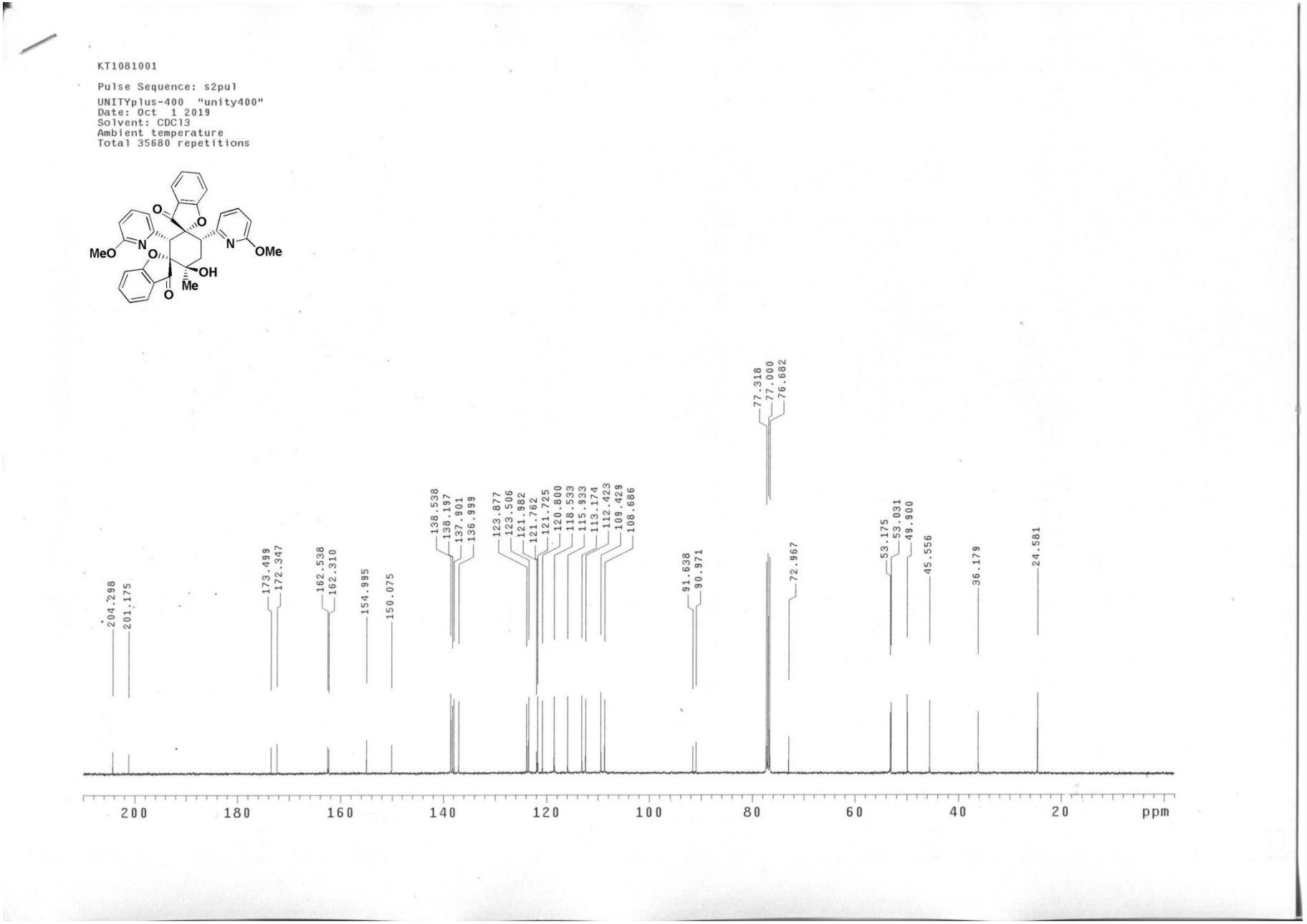
Compound 3z (^{13}C-NMR spectral data)



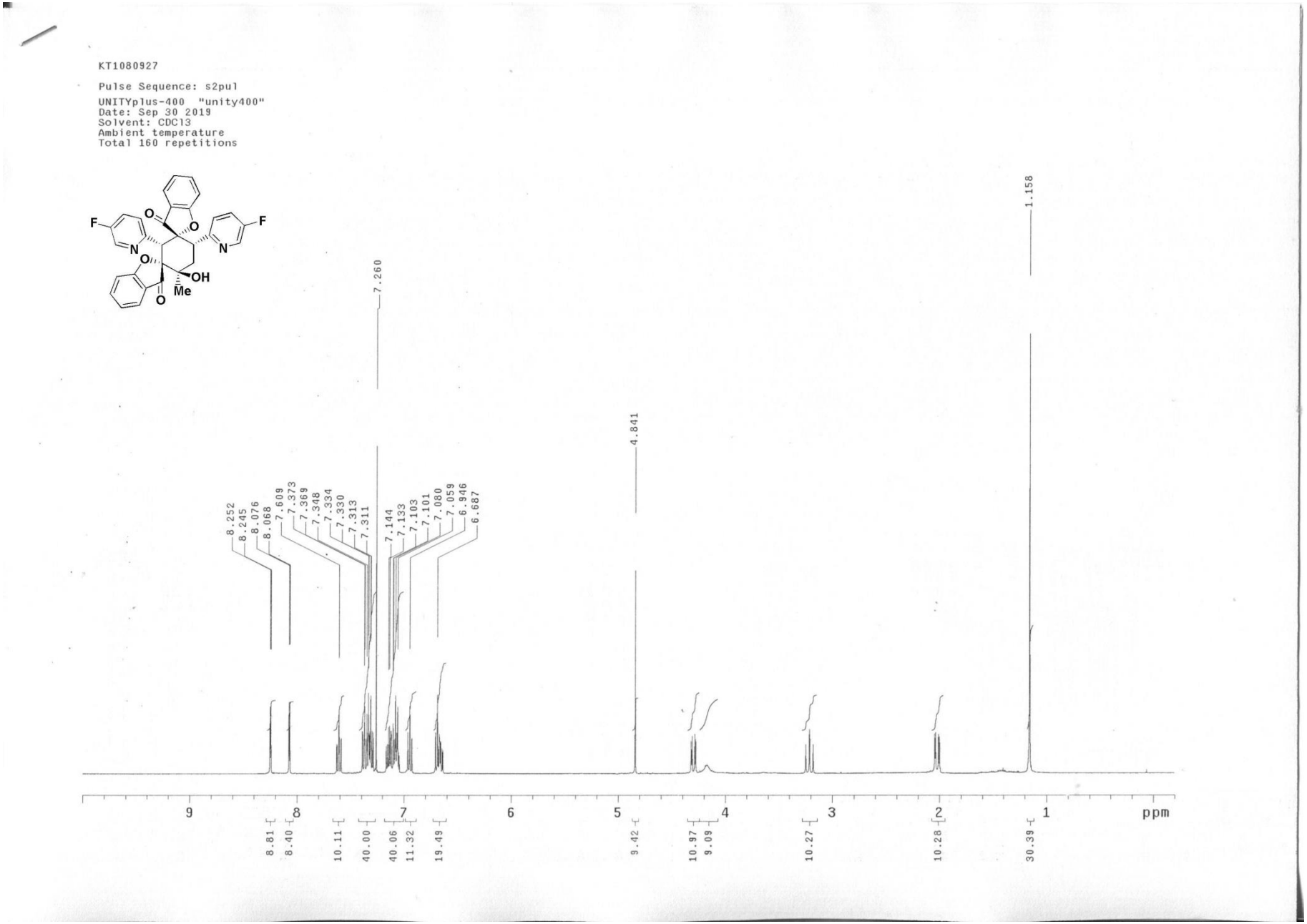
Compound 3aa (^{1}H-NMR spectral data)



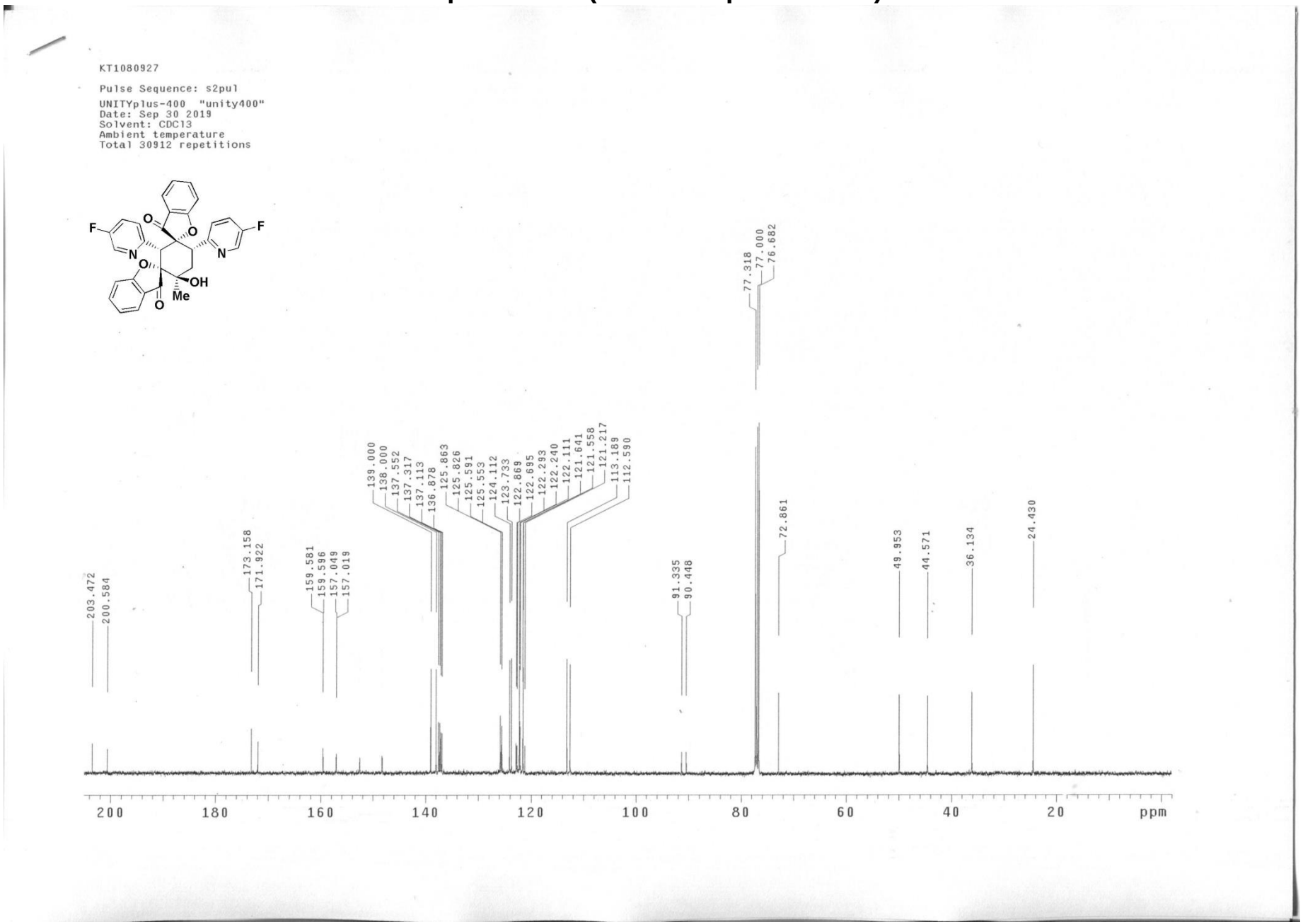
Compound 3aa (^{13}C-NMR spectral data)



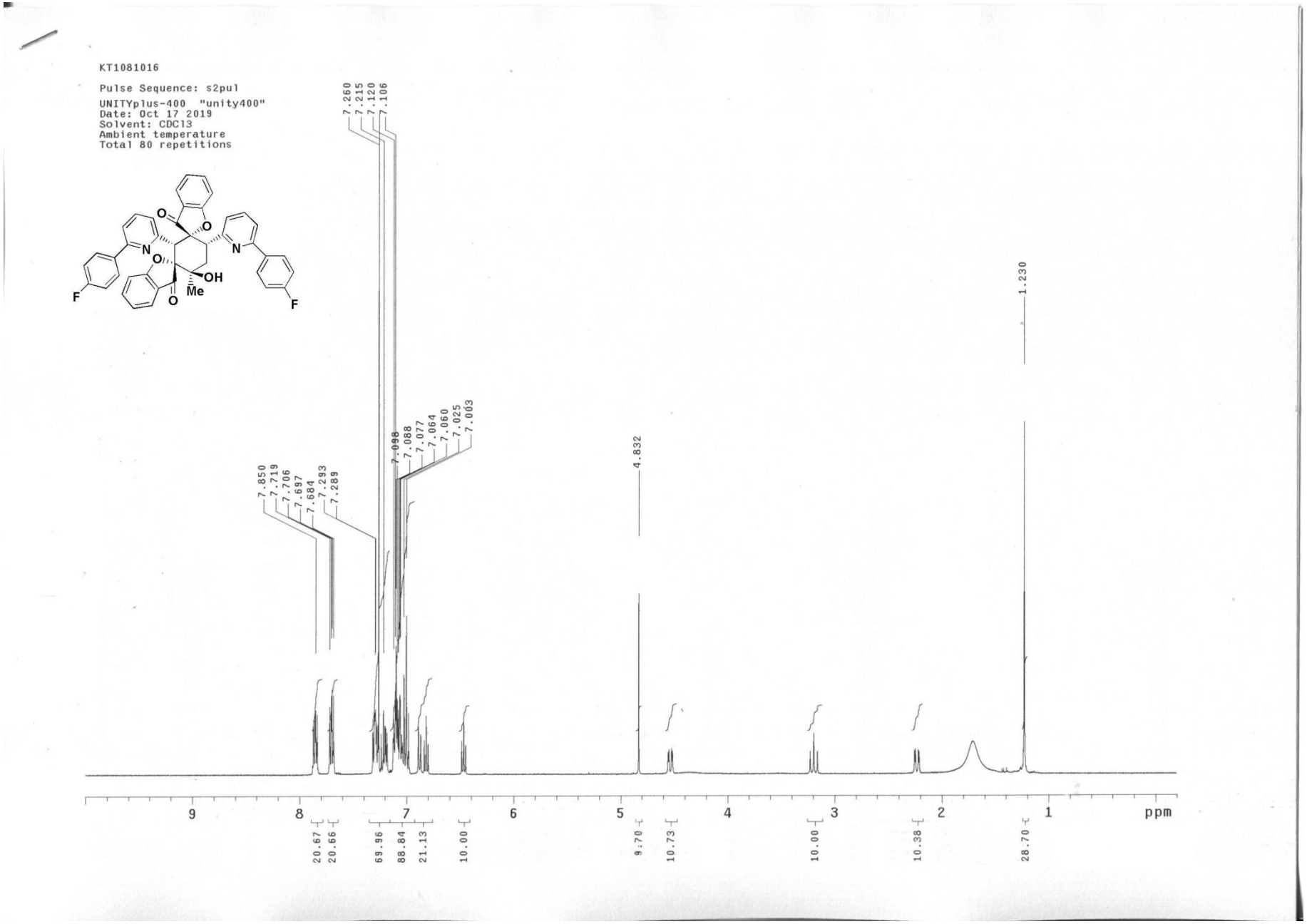
Compound 3ab (^{1}H-NMR spectral data)



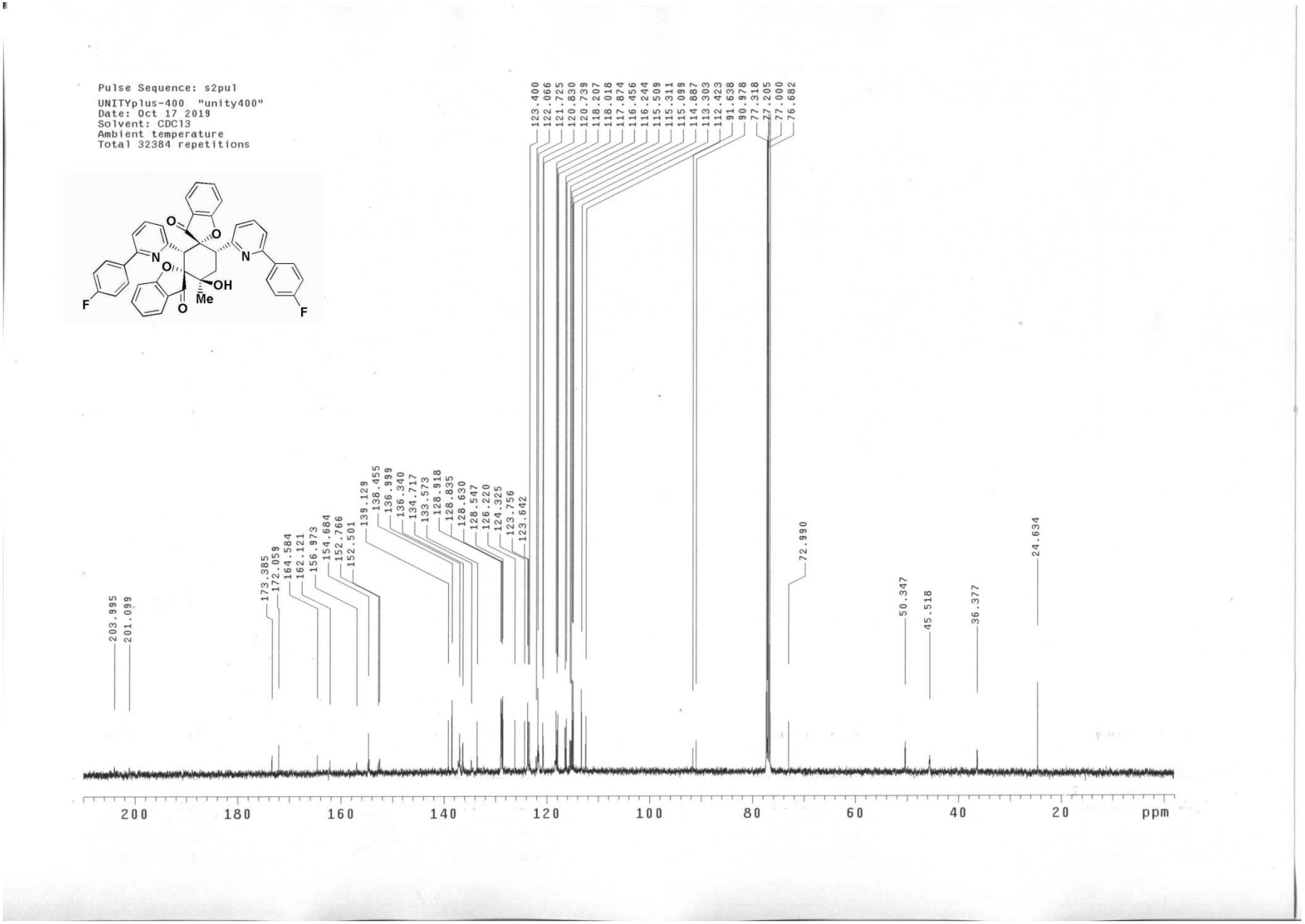
Compound 3ab (^{13}C-NMR spectral data)



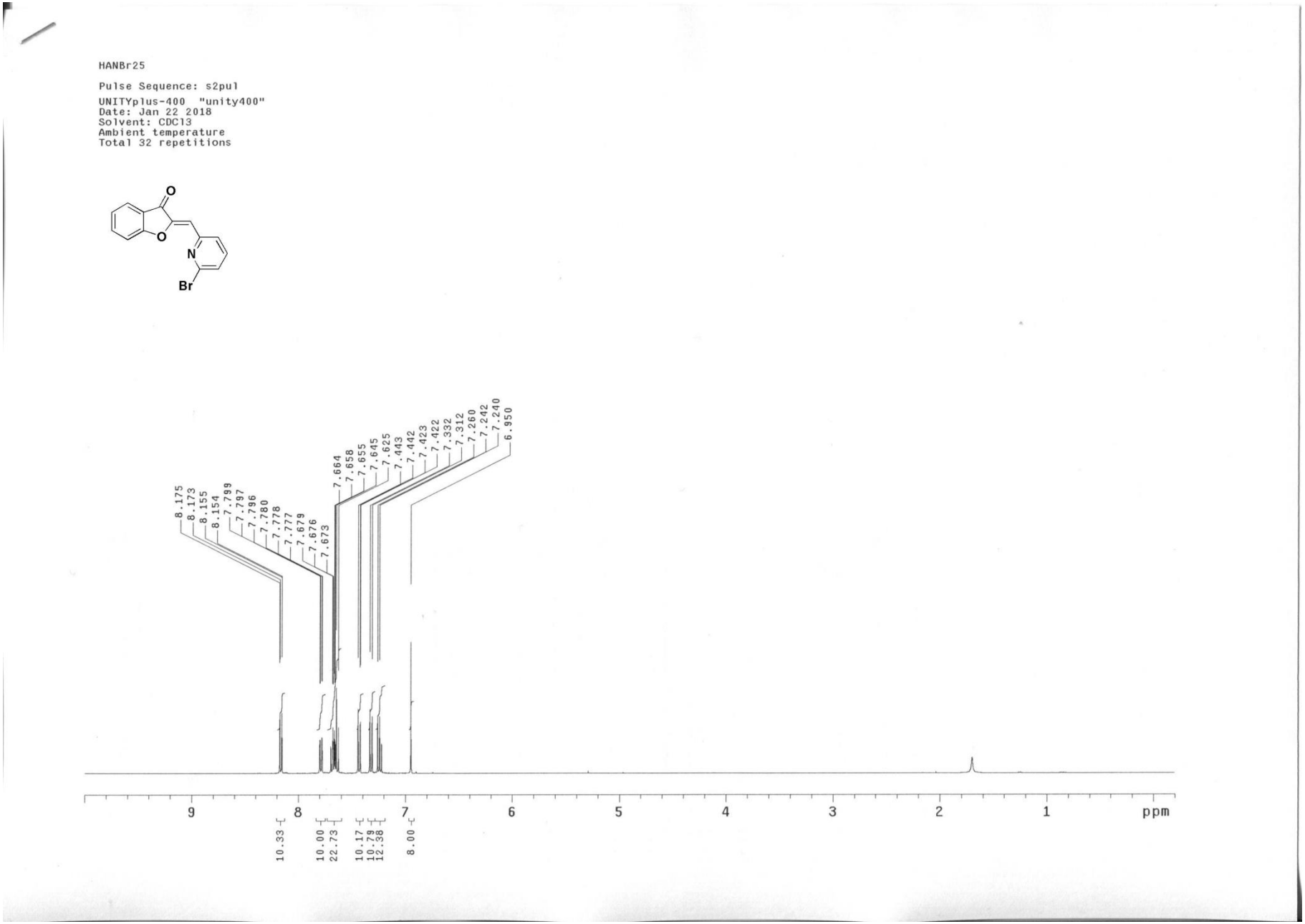
Compound 3ac (^{1}H-NMR spectral data)



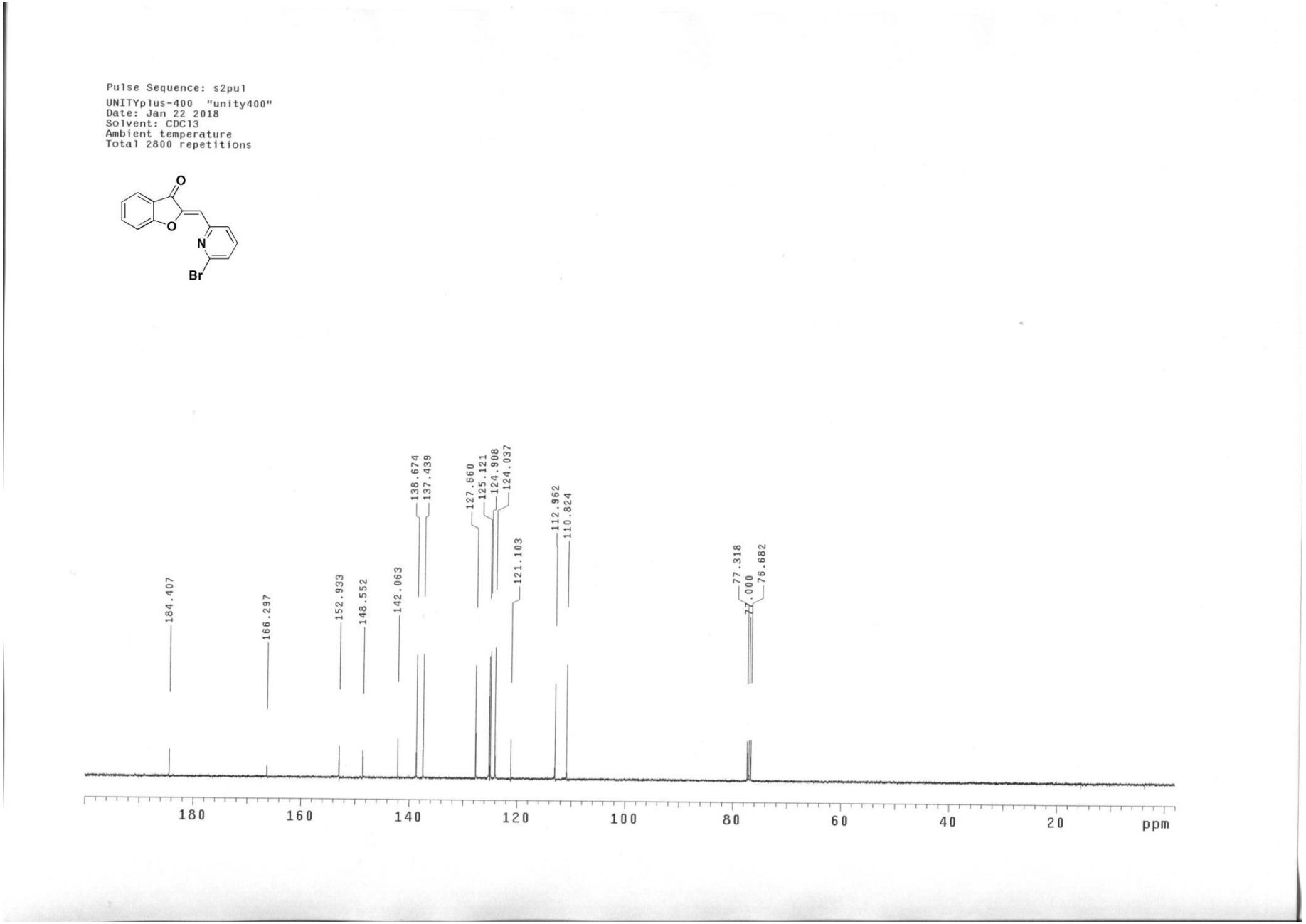
Compound 3ac (^{13}C-NMR spectral data)



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



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

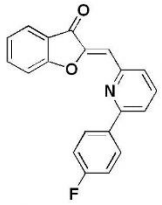


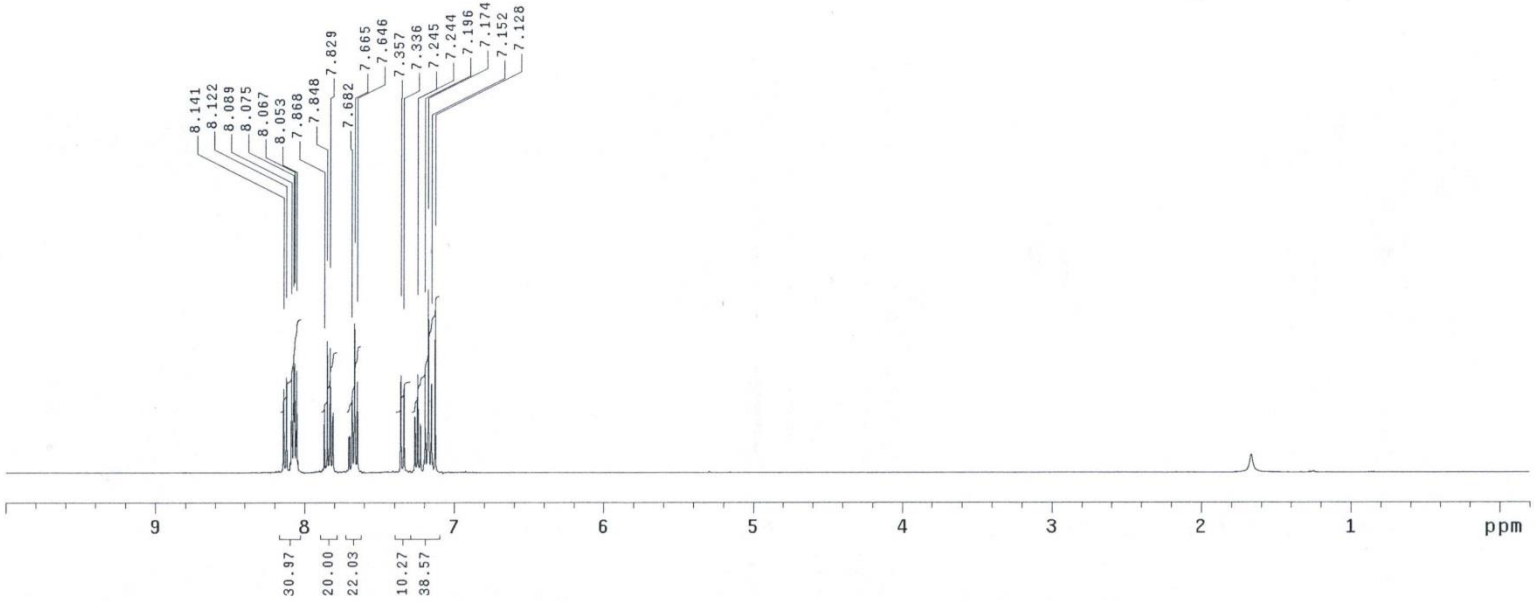
Compound 4ac (^{1}H-NMR spectral data)

HANFPh30

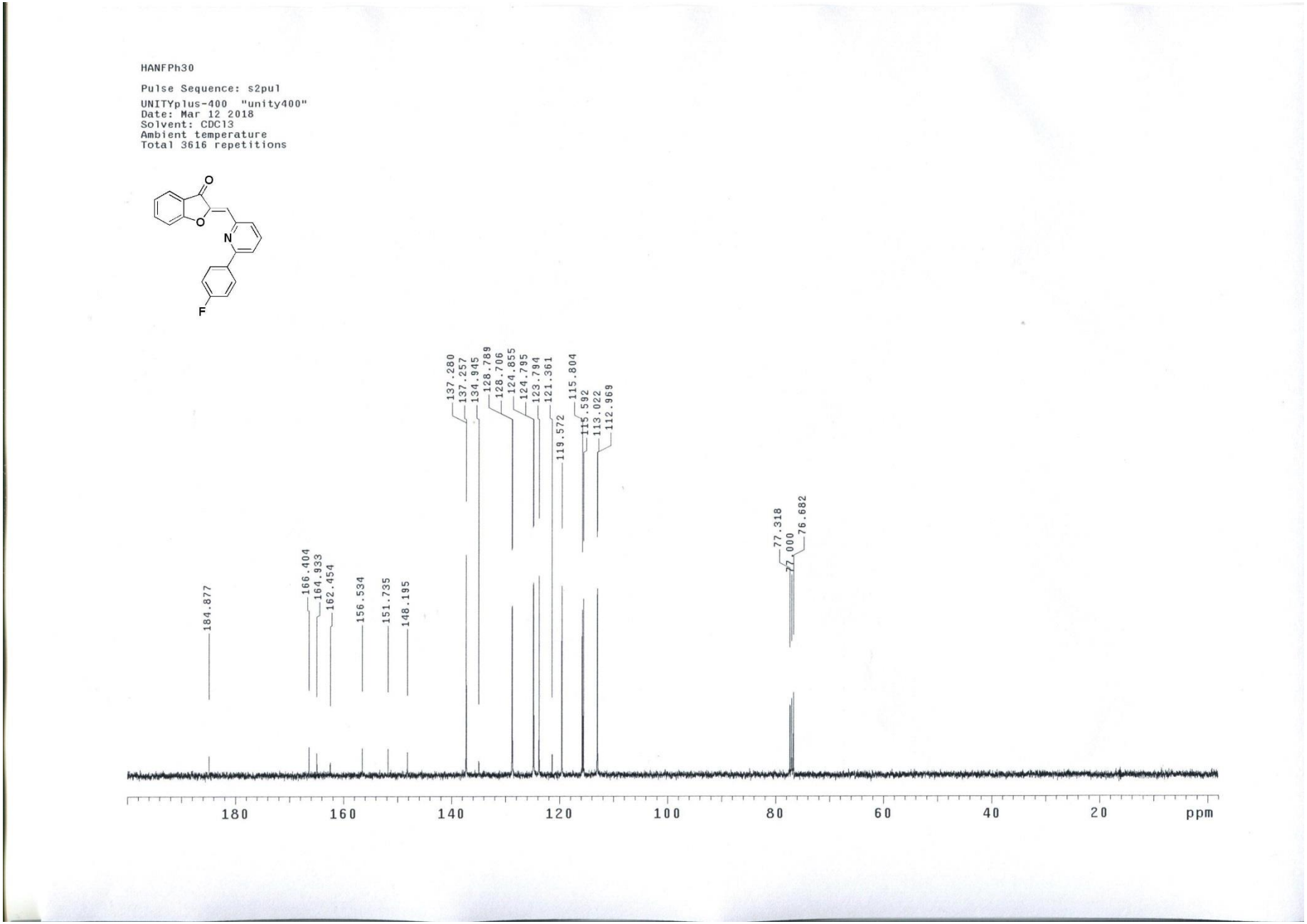
Pulse Sequence: s2pul

UNITYplus-400 "unity400"
Date: Mar 12 2018
Solvent: CDCl3
Ambient temperature
Total 32 repetitions

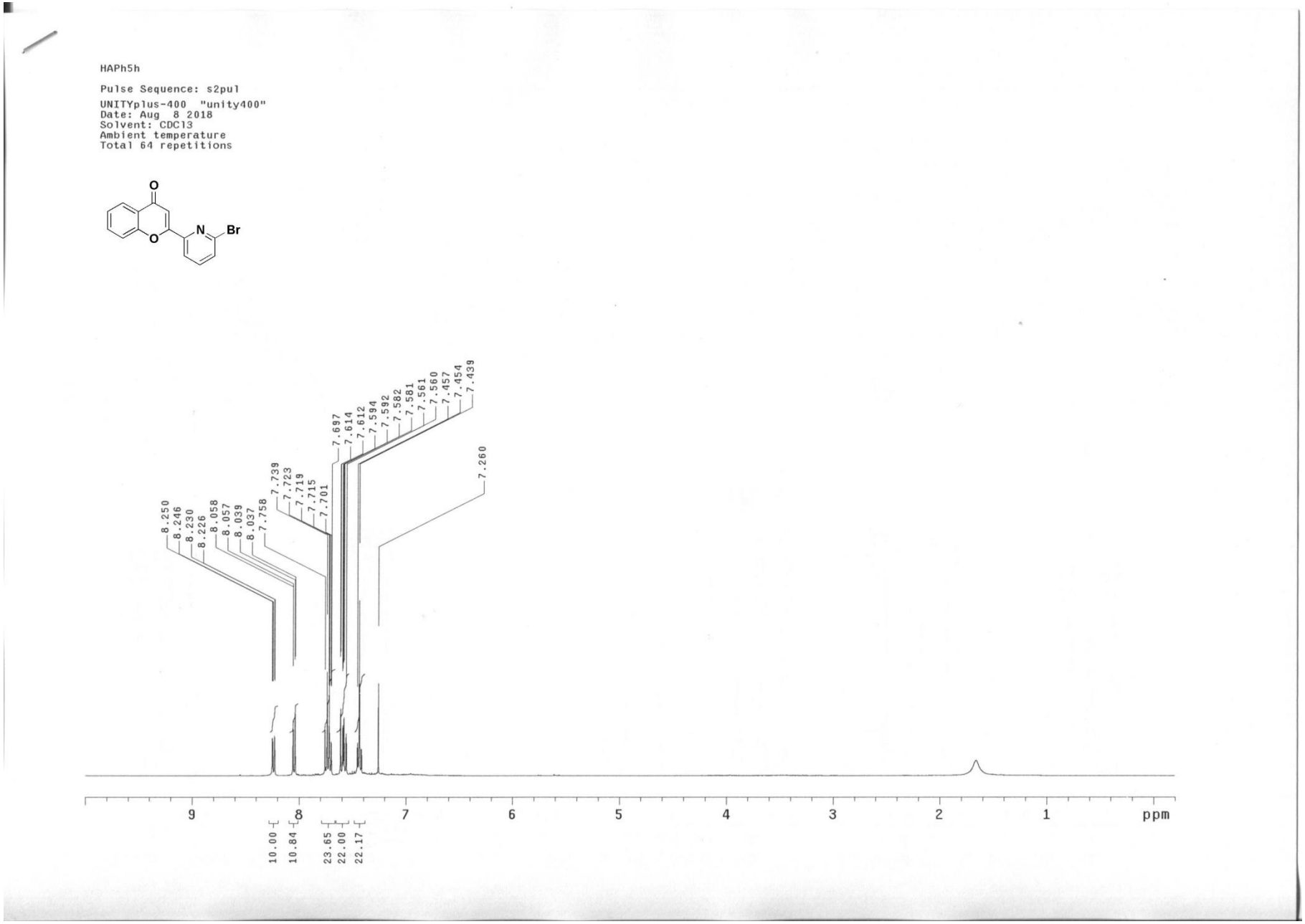




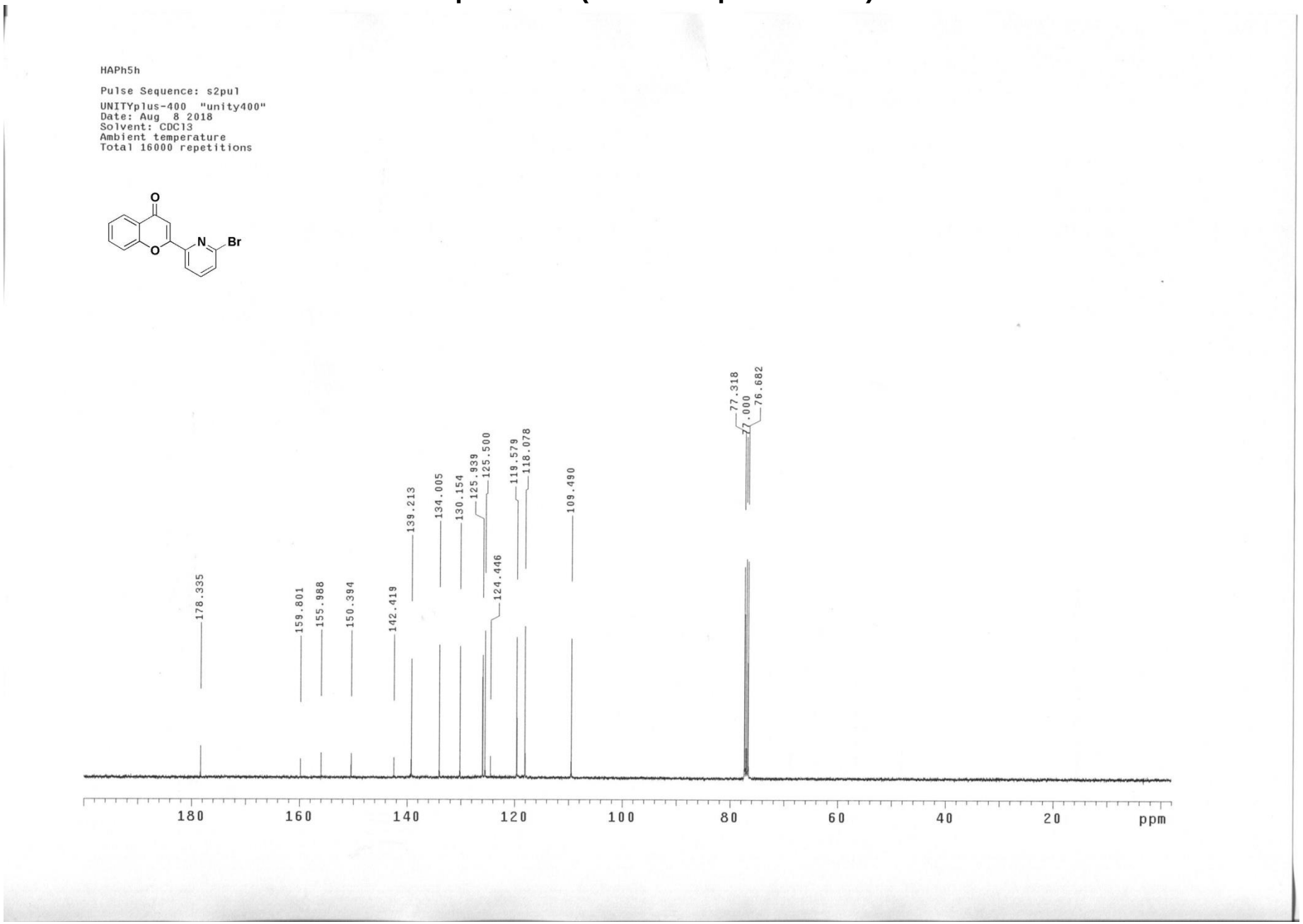
Compound 4ac (^{13}C-NMR spectral data)



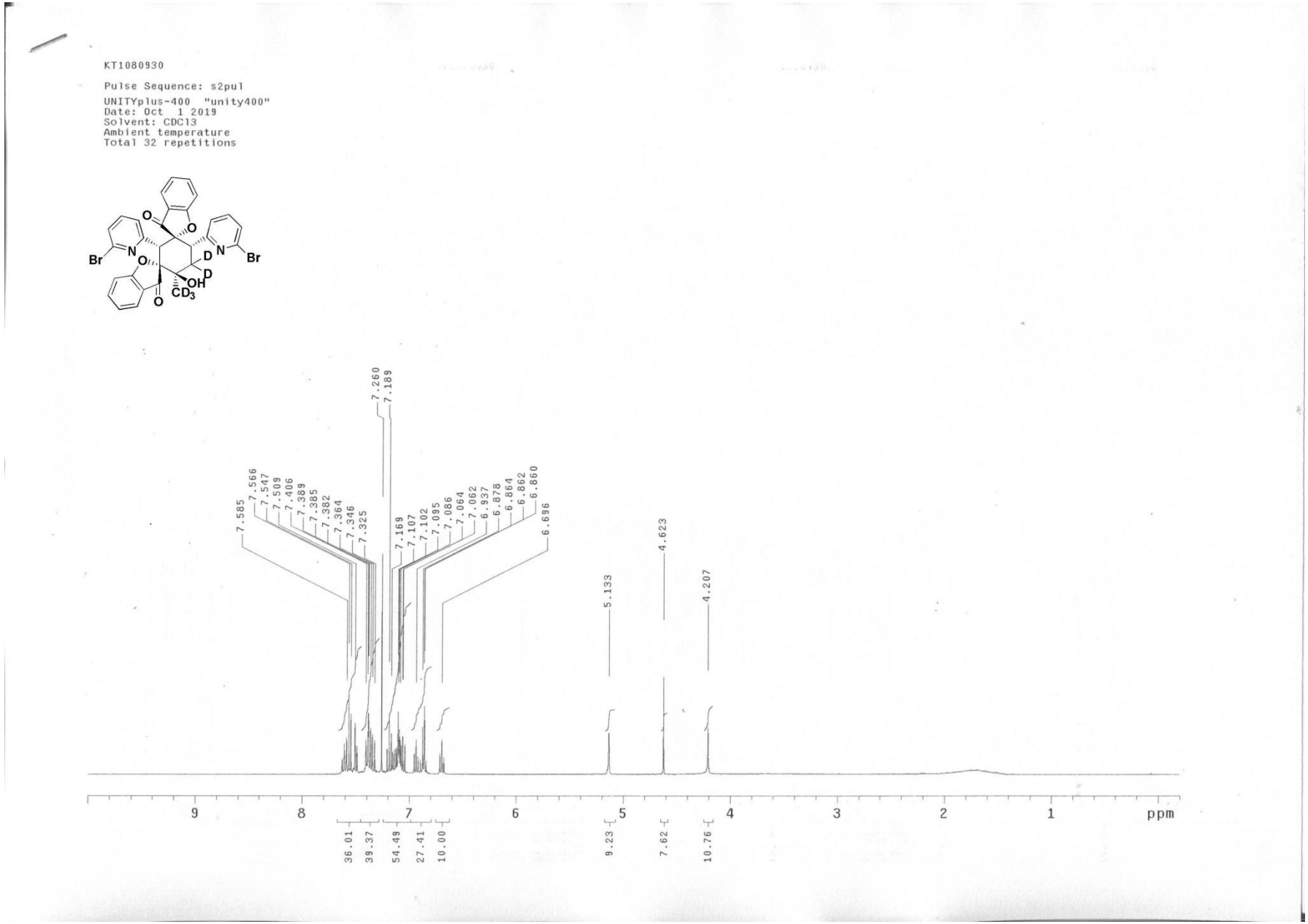
Compound 5a (^{1}H-NMR spectral data)



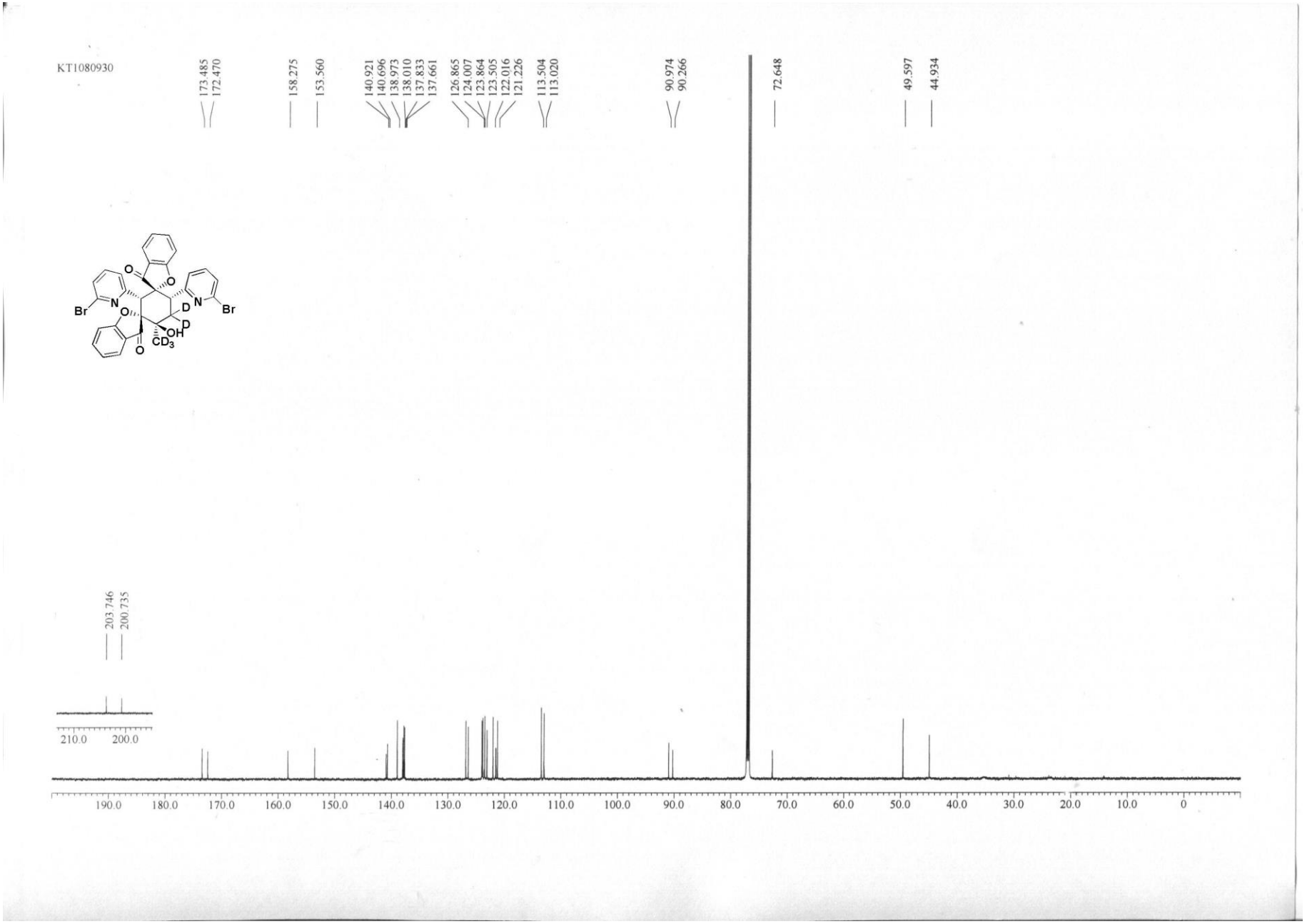
Compound 5a (^{13}C-NMR spectral data)



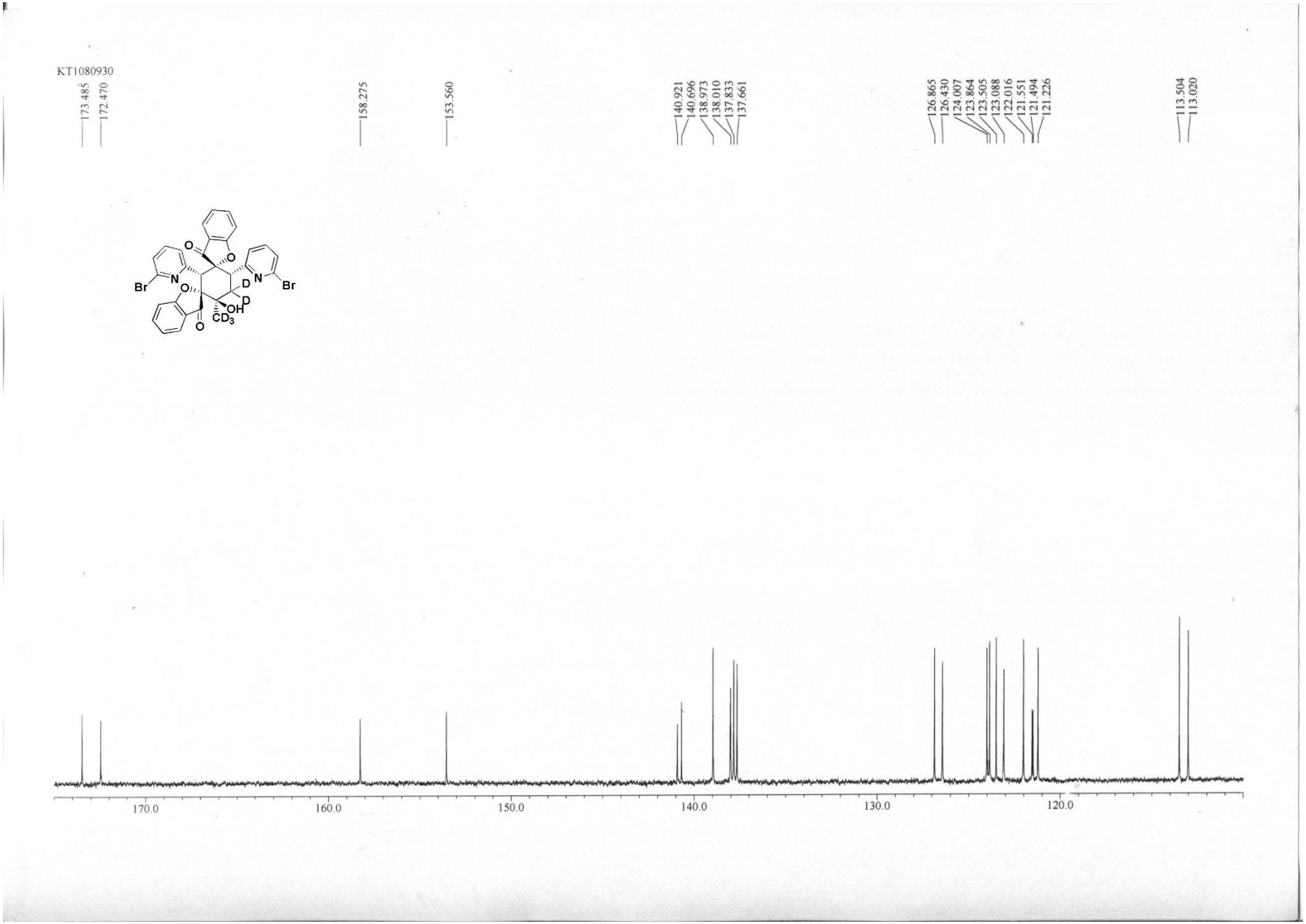
Compound 6 (^{1}H-NMR spectral data-400 MHz)



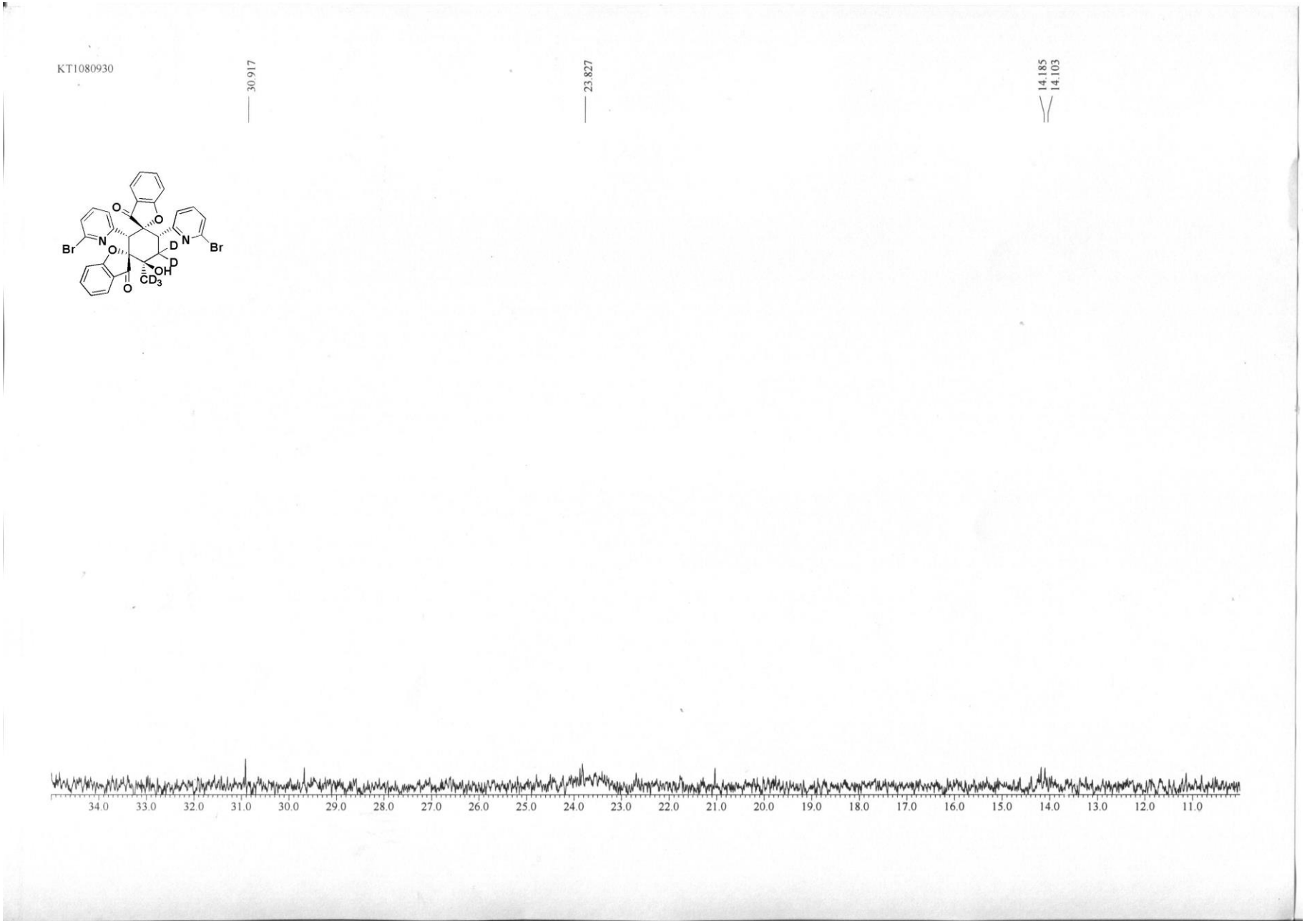
Compound 6 (^{13}C-NMR spectral data-1)



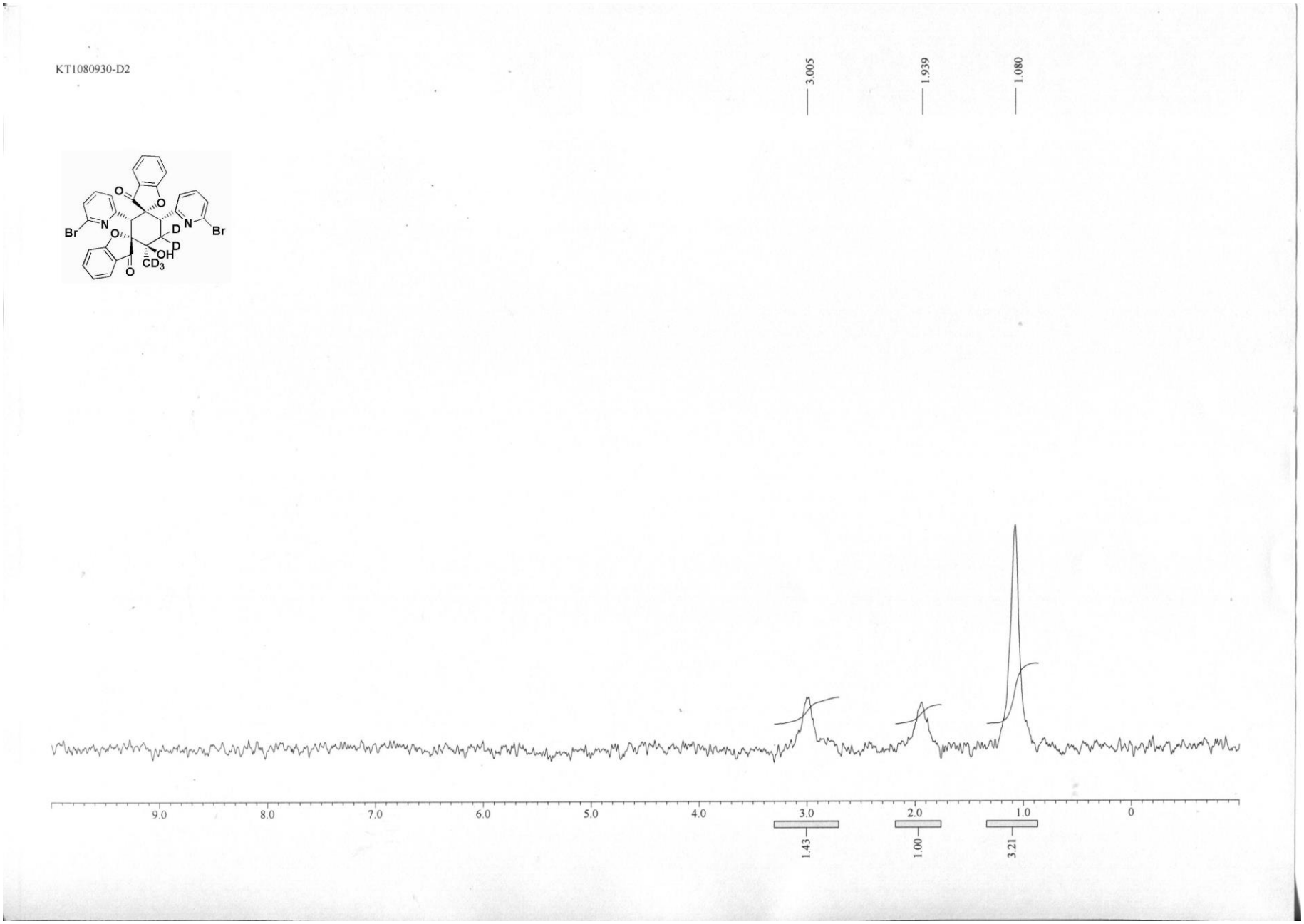
Compound 6 (^{13}C-NMR spectral data-2)



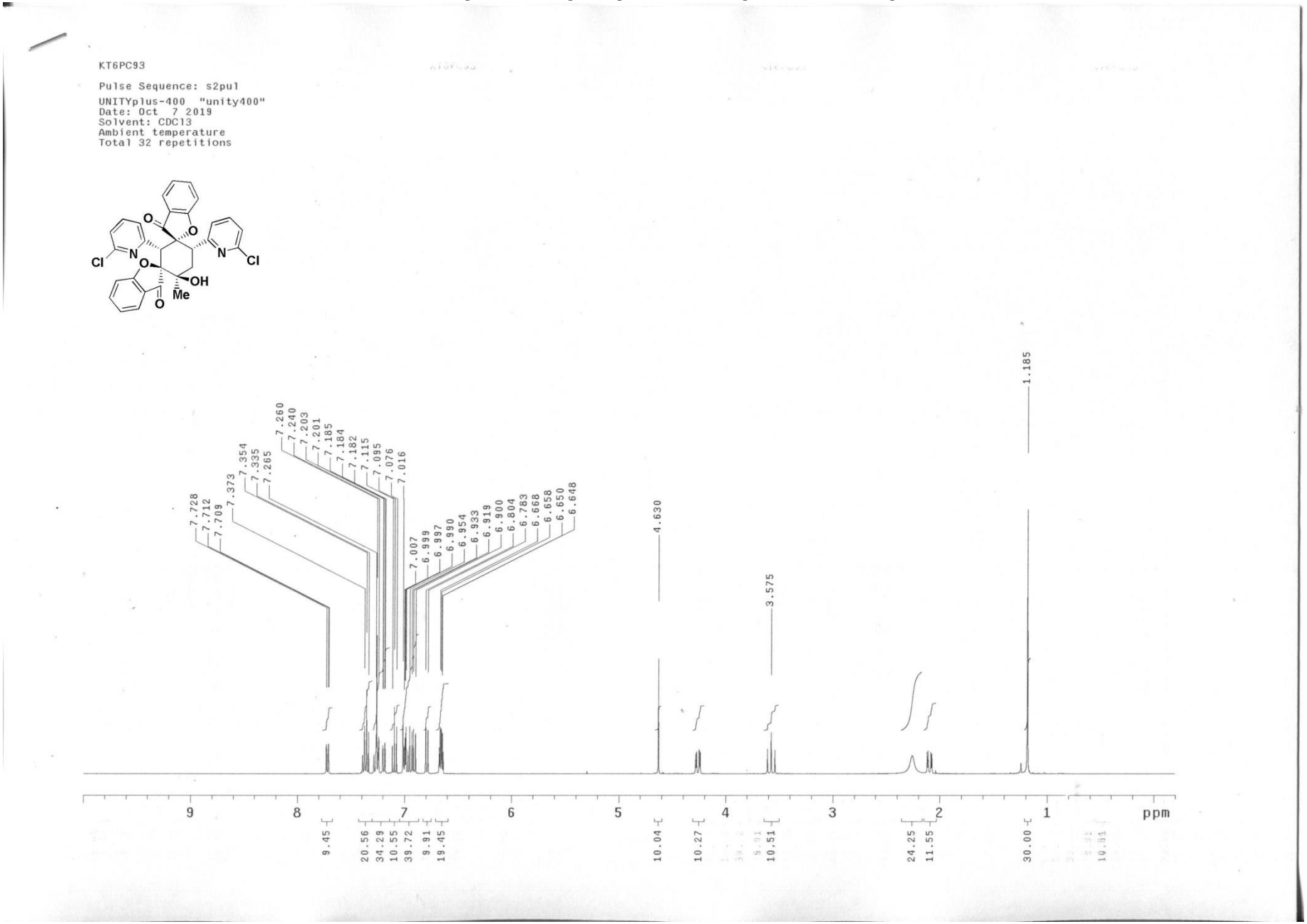
Compound 6 (^{13}C-NMR spectral data-3)



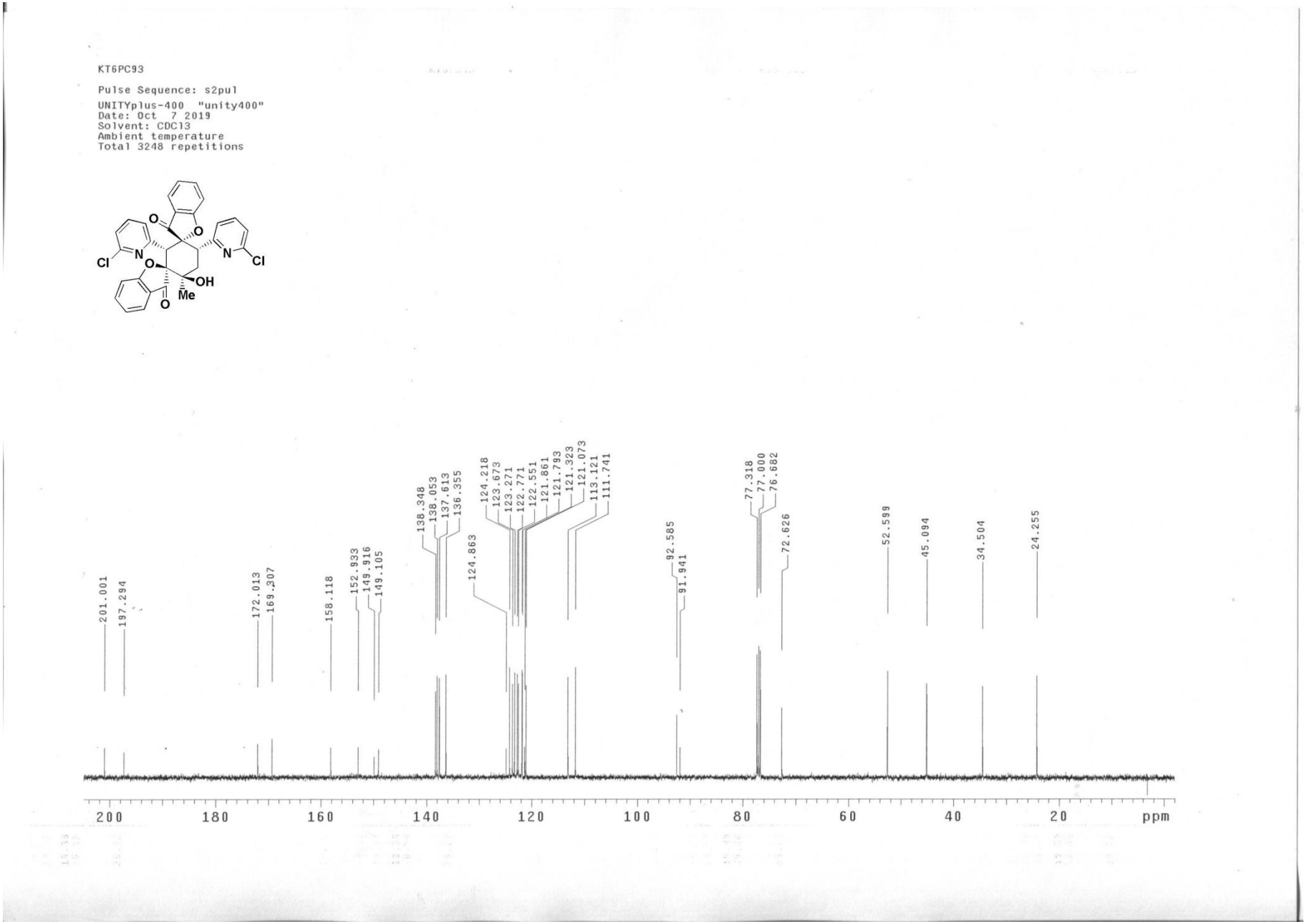
Compound 6 (^{2}H-NMR spectral data)



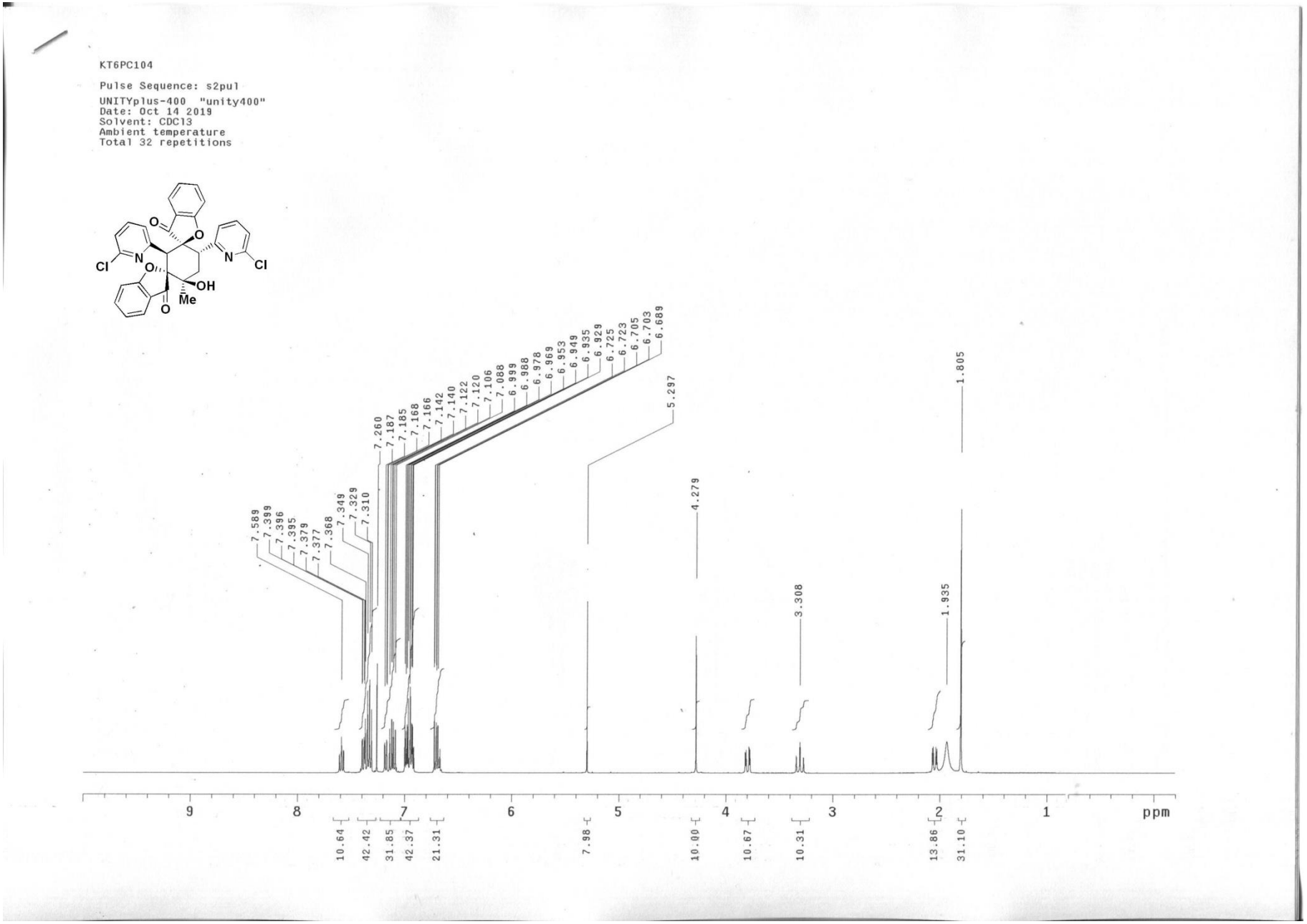
Compound 3y-1 (^{1}H-NMR spectral data)



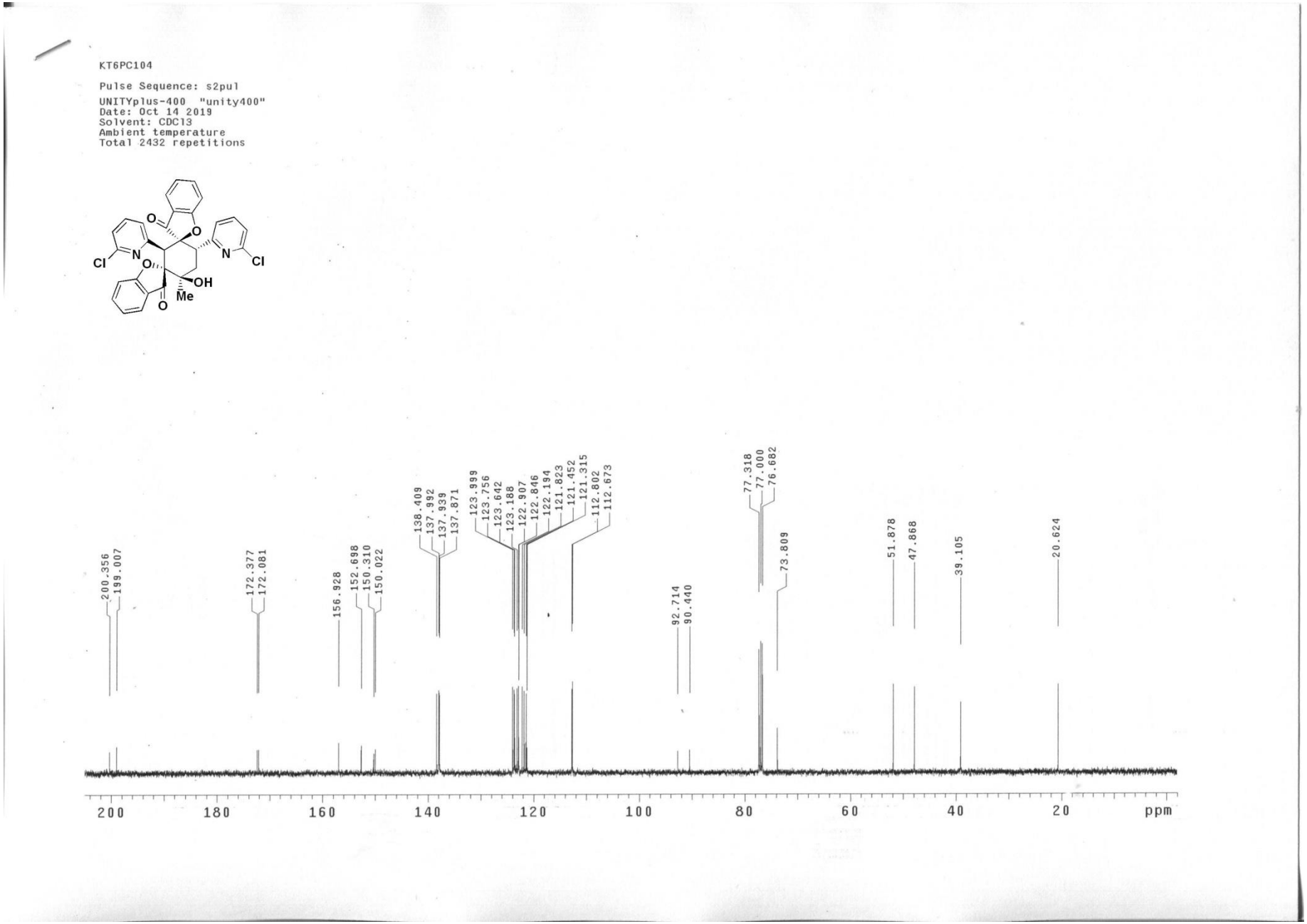
Compound 3y-1 (^{13}C-NMR spectral data)



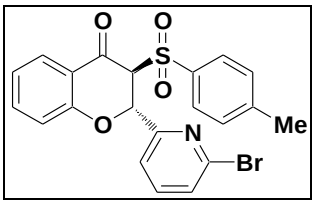
Compound 3y-2 (^{1}H-NMR spectral data)



Compound 3y-2 (^{13}C-NMR spectral data)

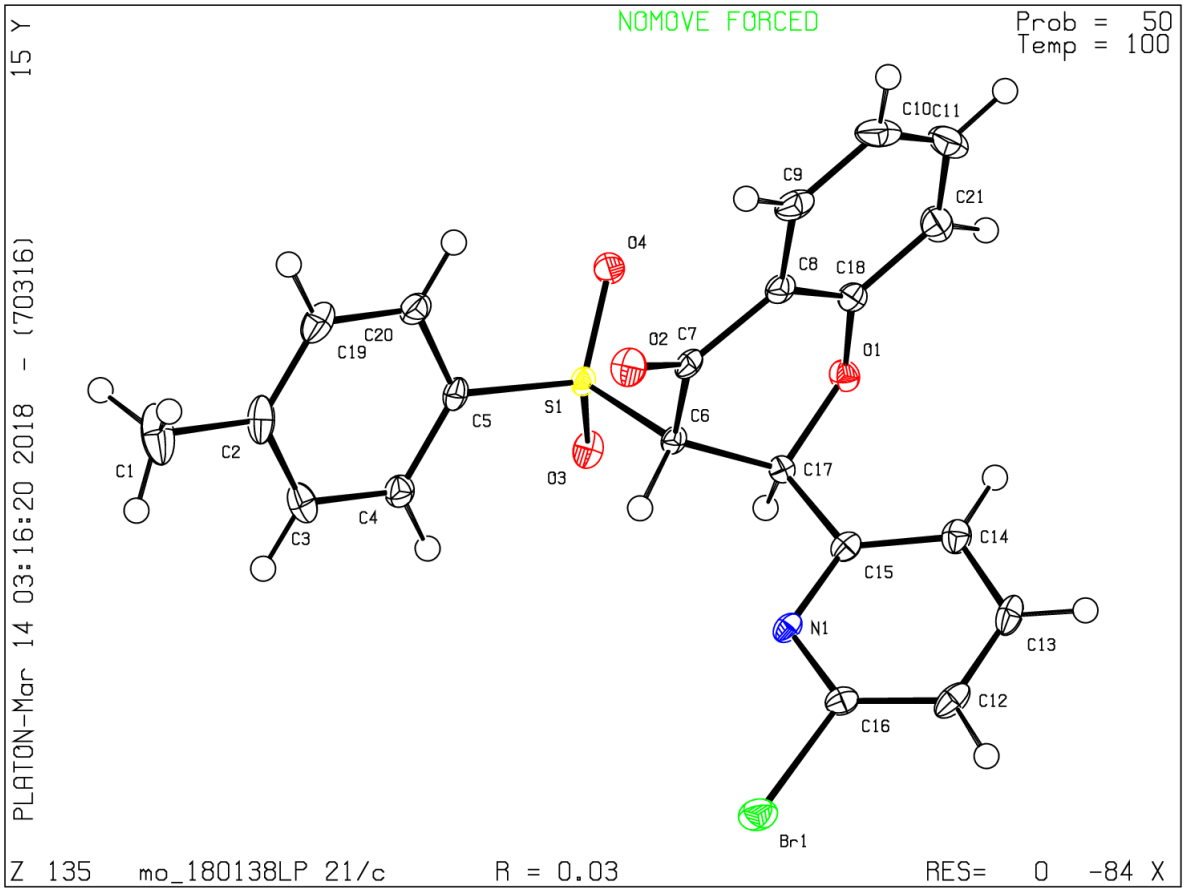


X-ray crystal data of compound 2a (CCDC 1965985)



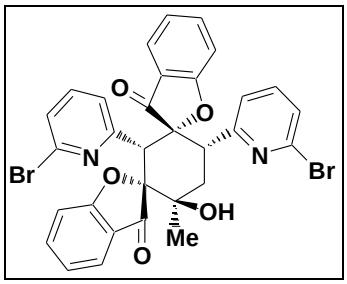
Sample preparation : A solution of compound **2a** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



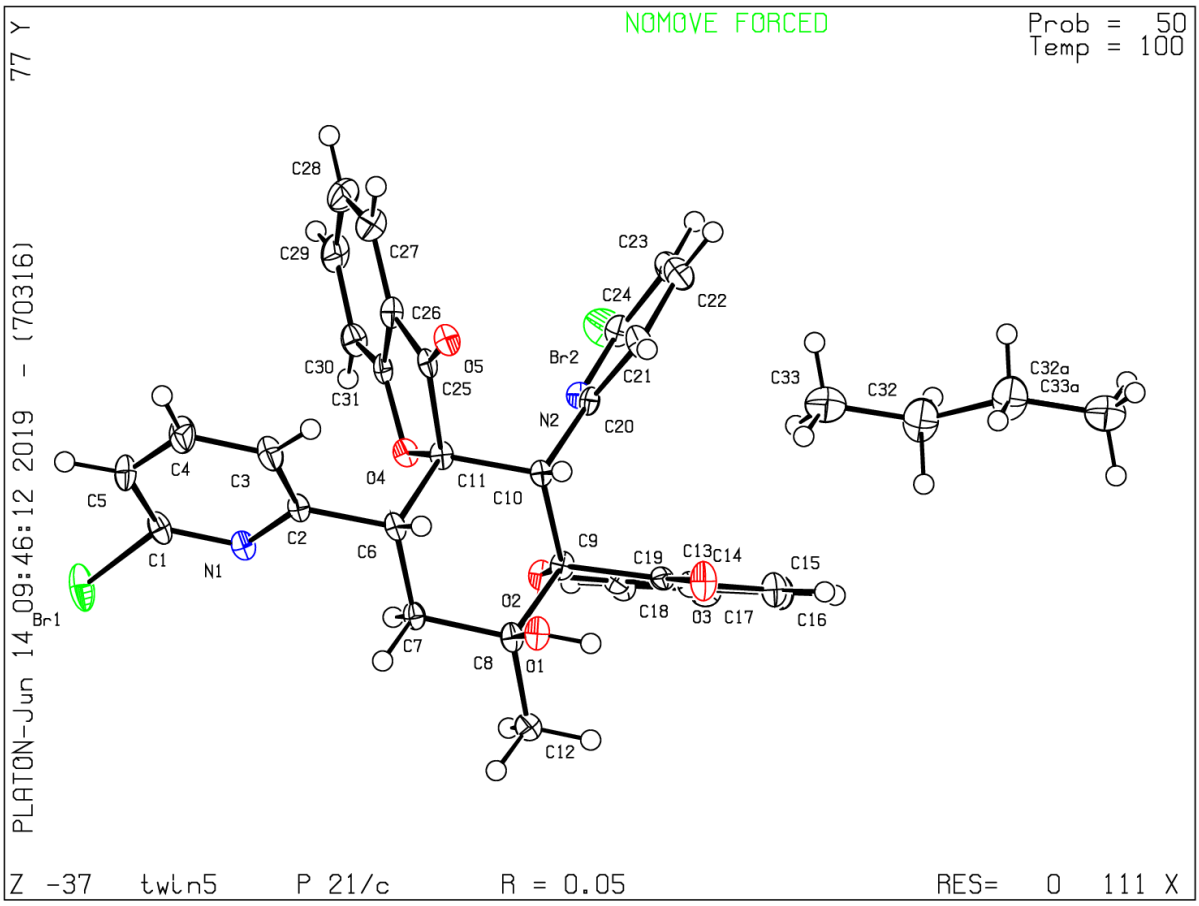
Empirical formula	C21 H16 Br N O4 S	
Formula weight	458.32	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 21/c	
Unit cell dimensions	a = 8.2647(4) Å	α = 90°.
	b = 19.7204(11) Å	β = 95.308(2)°.
	c = 11.6058(6) Å	γ = 90°.
Volume	1883.44(17) $Å^3$	
Z	4	
Density (calculated)	1.616 Mg/m^3	
Absorption coefficient	2.322 mm^{-1}	
F(000)	928	
Crystal size	0.20 x 0.18 x 0.17 mm^3	
Theta range for data collection	2.043 to 26.697°.	
Index ranges	-10<=h<=10, -24<=k<=23, -14<=l<=14	
Reflections collected	30351	
Independent reflections	3930 [R(int) = 0.0460]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9485 and 0.8806	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3930 / 0 / 254	
Goodness-of-fit on F^2	1.041	
Final R indices [I>2sigma(I)]	R1 = 0.0278, wR2 = 0.0598	
R indices (all data)	R1 = 0.0372, wR2 = 0.0629	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.438 and -0.388 e.$Å^{-3}$	

X-ray crystal data of compound 3a (CCDC 1965986)



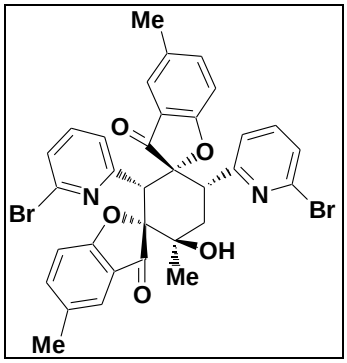
Sample preparation : A solution of compound **3a** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



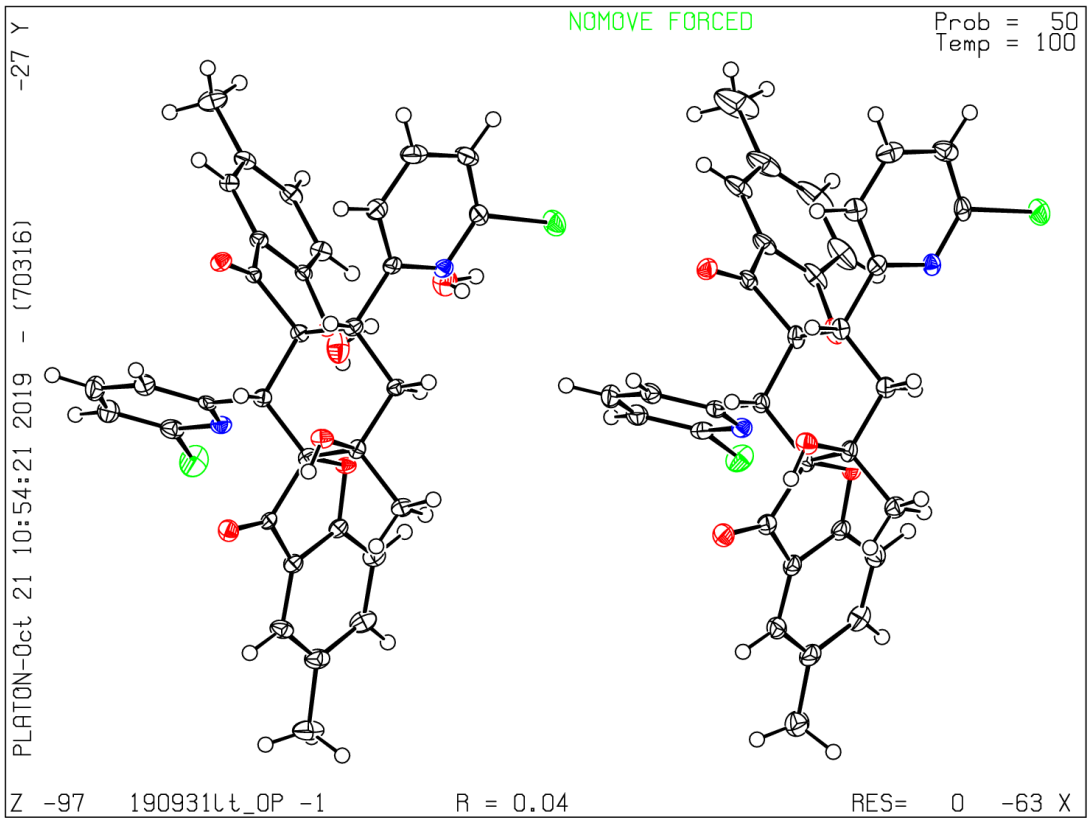
Empirical formula	C33 H27 Br2 N2 O5	
Formula weight	691.38	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 13.1453(16) Å	α = 90°.
	b = 12.9849(18) Å	β = 96.328(5)°.
	c = 16.874(3) Å	γ = 90°.
Volume	2862.6(7) $Å^3$	
Z	4	
Density (calculated)	1.604 Mg/m^3	
Absorption coefficient	2.878 mm^{-1}	
F(000)	1396	
Crystal size	0.22 x 0.20 x 0.19 mm^3	
Theta range for data collection	1.559 to 26.494°.	
Index ranges	-16<=h<=16, 0<=k<=16, 0<=l<=21	
Reflections collected	6408	
Independent reflections	6408 [R(int) = 0.0719]	
Completeness to theta = 25.242°	99.5 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.5536	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	6408 / 366 / 383	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0504, wR2 = 0.1123	
R indices (all data)	R1 = 0.1029, wR2 = 0.1318	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.941 and -0.845 $e.Å^{-3}$	

X-ray crystal data of compound 3i (CCDC 1965987)



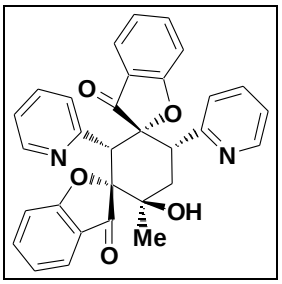
Sample preparation : A solution of compound **3i** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



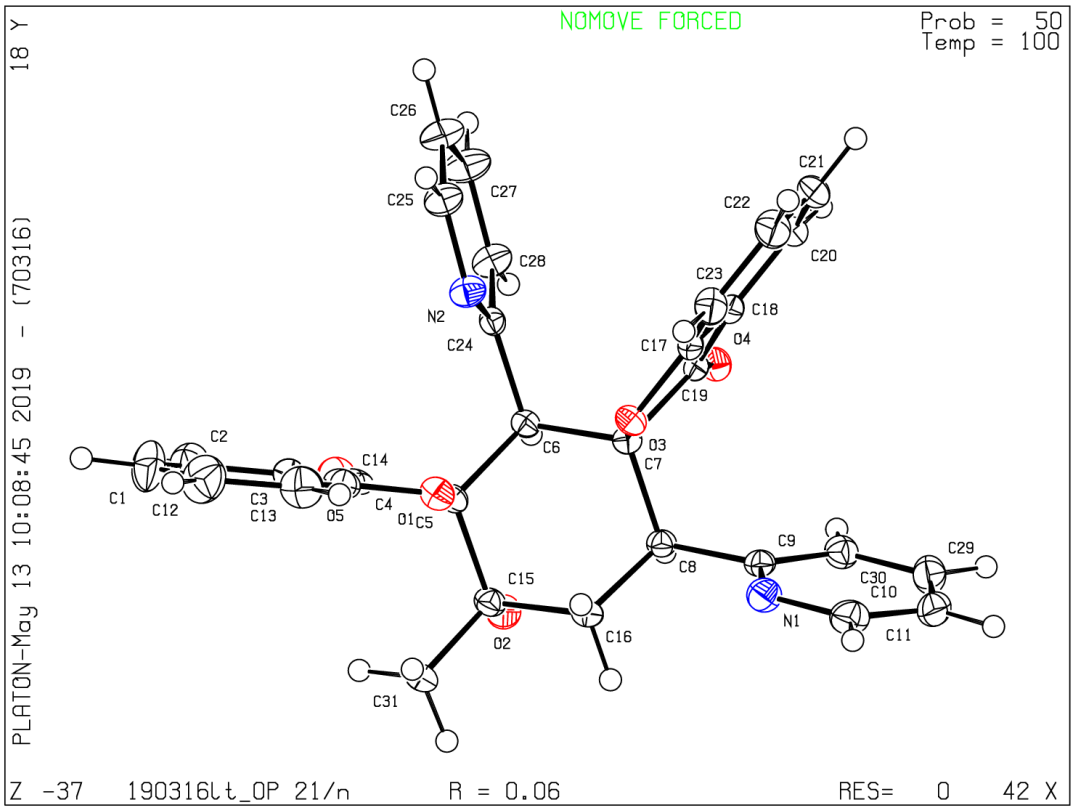
Empirical formula	C66 H56 Br4 N4 O12	
Formula weight	1416.78	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.3171(6) Å	α = 77.235(2)°.
	b = 15.1045(7) Å	β = 76.167(2)°.
	c = 16.7543(6) Å	γ = 67.821(3)°.
Volume	2998.0(2) $Å^3$	
Z	2	
Density (calculated)	1.569 Mg/m^3	
Absorption coefficient	2.753 mm^{-1}	
F(000)	1432	
Crystal size	0.10 x 0.08 x 0.08 mm^3	
Theta range for data collection	1.265 to 26.434°.	
Index ranges	-16<=h<=16, -18<=k<=18, -20<=l<=20	
Reflections collected	46340	
Independent reflections	12292 [R(int) = 0.0439]	
Completeness to theta = 25.242°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.6801	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	12292 / 6 / 799	
Goodness-of-fit on F^2	1.023	
Final R indices [I>2sigma(I)]	R1 = 0.0376, wR2 = 0.0805	
R indices (all data)	R1 = 0.0597, wR2 = 0.0879	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.880 and -1.097 e.$Å^{-3}$	

X-ray crystal data of compound 3j (CCDC 1965988)



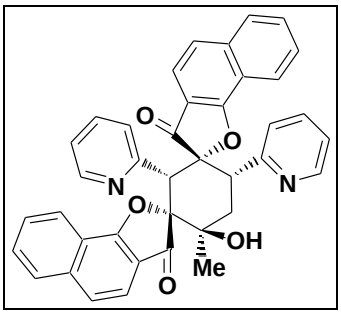
Sample preparation : A solution of compound **3j** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



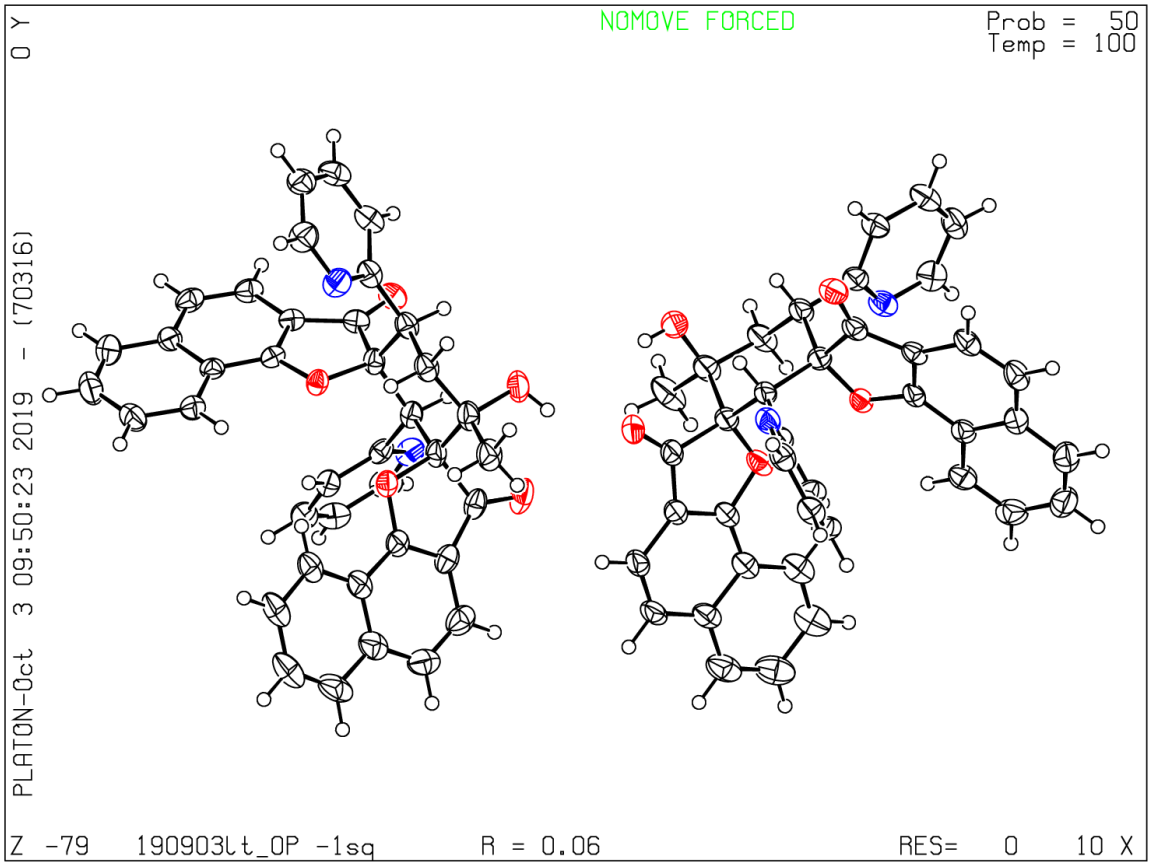
Empirical formula	C31 H23 N2 O5	
Formula weight	503.51	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	$P2_1/n$	
Unit cell dimensions	a = 11.8834(7) Å	α = 90°.
	b = 8.8469(5) Å	β = 91.109(2)°.
	c = 23.6723(12) Å	γ = 90°.
Volume	2488.2(2) $Å^3$	
Z	4	
Density (calculated)	1.344 Mg/m^3	
Absorption coefficient	0.092 mm^{-1}	
F(000)	1052	
Crystal size	0.20 x 0.18 x 0.16 mm^3	
Theta range for data collection	1.903 to 26.396°.	
Index ranges	-14<=h<=14, -11<=k<=10, -26<=l<=29	
Reflections collected	20298	
Independent reflections	5099 [R(int) = 0.0314]	
Completeness to theta = 25.242°	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.7087	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5099 / 0 / 344	
Goodness-of-fit on F^2	1.034	
Final R indices [I>2sigma(I)]	R1 = 0.0571, wR2 = 0.1530	
R indices (all data)	R1 = 0.0692, wR2 = 0.1635	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.466 and -0.378 $e.Å^{-3}$	

X-ray crystal data of compound 3o (CCDC 1965996)



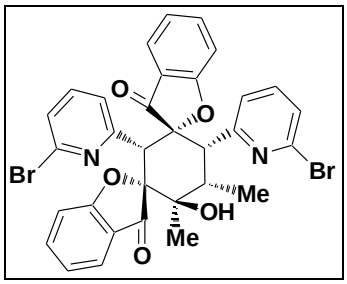
Sample preparation : A solution of compound **3o** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



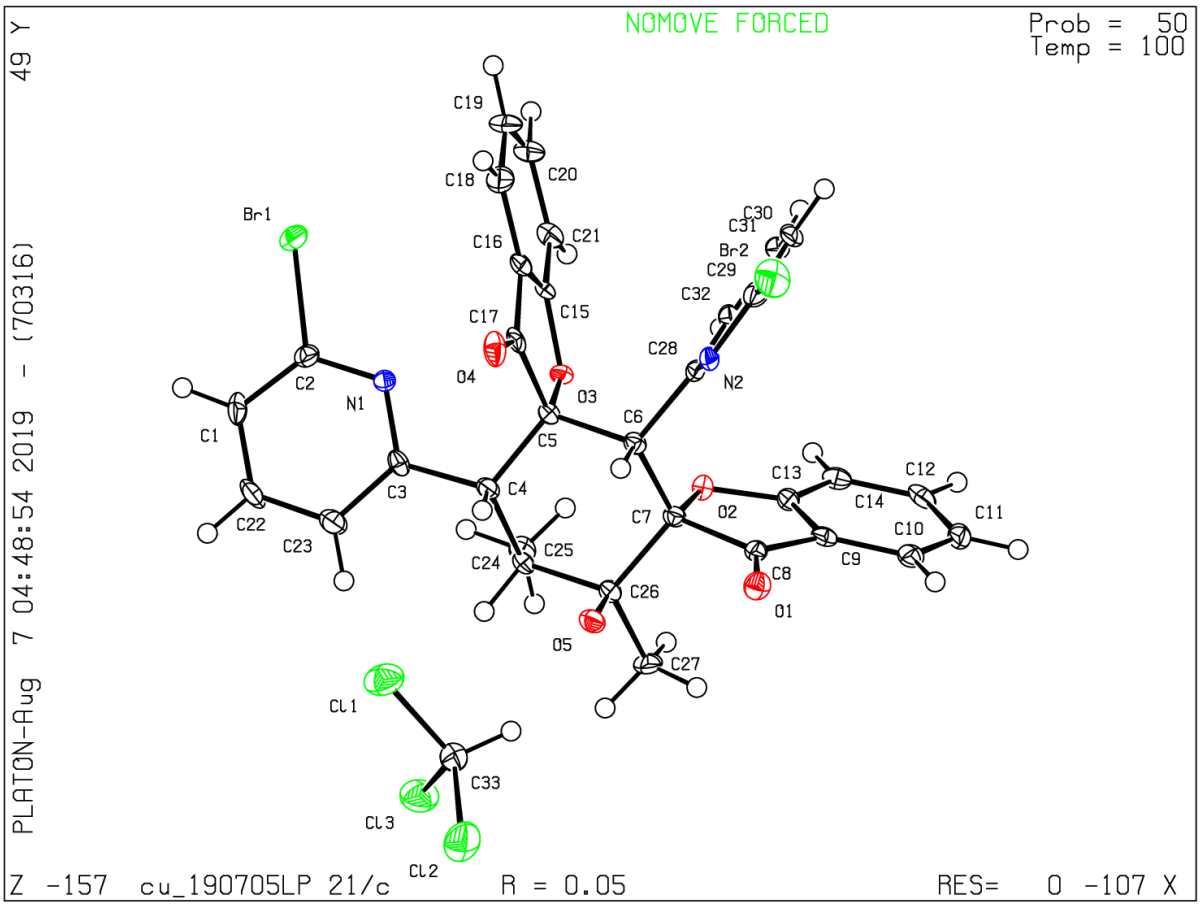
Empirical formula	C39 H28 N2 O5	
Formula weight	604.63	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.8900(7) Å	α = 64.295(2)°.
	b = 16.8786(9) Å	β = 88.306(2)°.
	c = 17.2527(9) Å	γ = 73.213(2)°.
Volume	3467.7(3) $Å^3$	
Z	4	
Density (calculated)	1.158 Mg/m^3	
Absorption coefficient	0.077 mm^{-1}	
F(000)	1264	
Crystal size	0.18 x 0.16 x 0.15 mm^3	
Theta range for data collection	1.318 to 26.437°.	
Index ranges	-17<=h<=16, -21<=k<=21, -21<=l<=21	
Reflections collected	53940	
Independent reflections	14196 [R(int) = 0.0435]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7454 and 0.6755	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	14196 / 0 / 833	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0572, wR2 = 0.1346	
R indices (all data)	R1 = 0.0901, wR2 = 0.1558	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.268 and -0.251 $e.Å^{-3}$	

X-ray crystal data of compound 3r (CCDC 1965997)



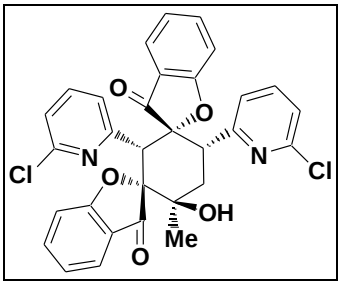
Sample preparation : A solution of compound **3r** (30 mg) in $CHCl_3$ (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



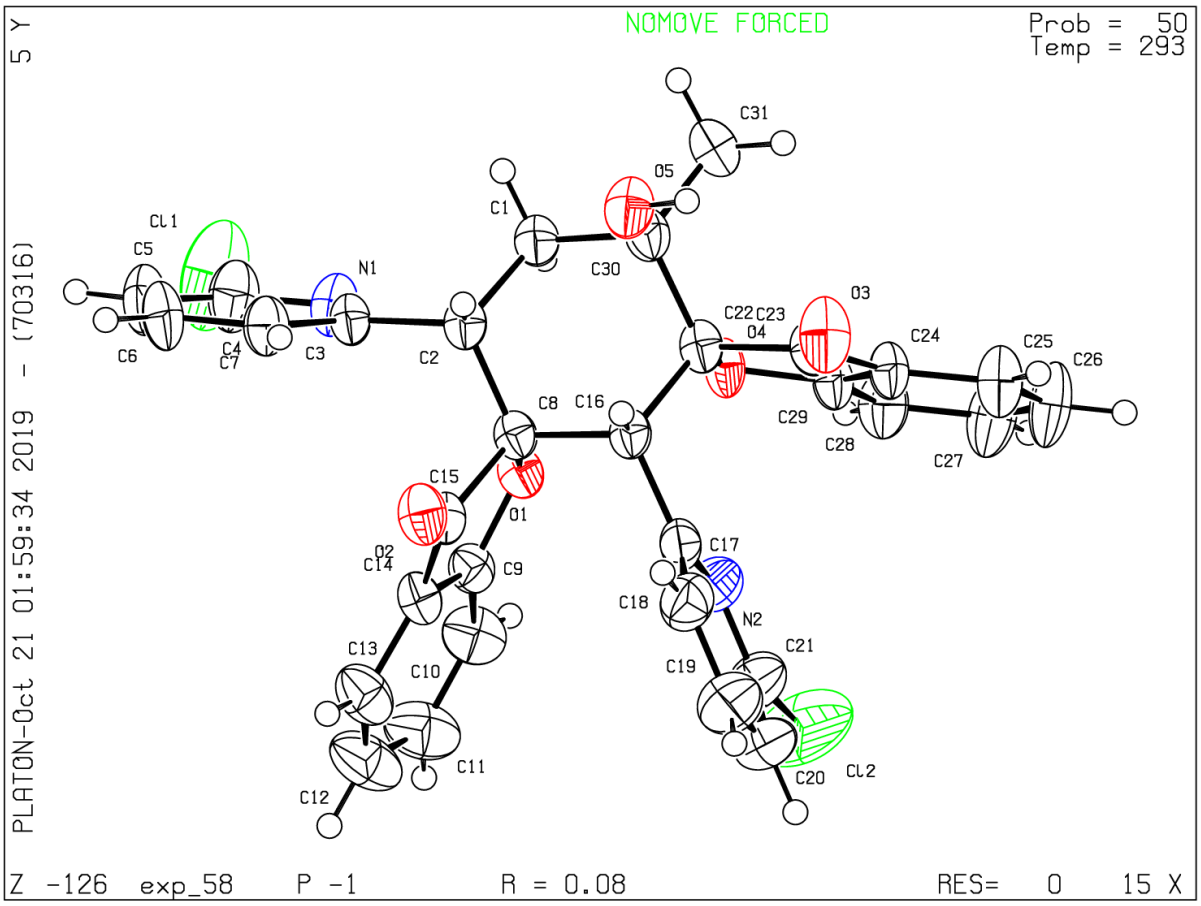
Empirical formula	C33 H24 Br2 Cl3 N2 O5	
Formula weight	794.71	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	$P2_1/c$	
Unit cell dimensions	a = 10.5330(4) Å	α = 90°.
	b = 26.8610(11) Å	β = 115.3340(10)°.
	c = 12.2233(5) Å	γ = 90°.
Volume	3125.7(2) $Å^3$	
Z	4	
Density (calculated)	1.689 Mg/m^3	
Absorption coefficient	6.036 mm^{-1}	
F(000)	1588	
Crystal size	0.15 x 0.13 x 0.12 mm^3	
Theta range for data collection	3.290 to 66.562°.	
Index ranges	-12<=h<=12, -31<=k<=26, -12<=l<=14	
Reflections collected	13631	
Independent reflections	5087 [R(int) = 0.0429]	
Completeness to theta = 67.679°	89.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.7528 and 0.4490	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5087 / 0 / 408	
Goodness-of-fit on F^2	1.120	
Final R indices [I>2sigma(I)]	R1 = 0.0518, wR2 = 0.1341	
R indices (all data)	R1 = 0.0531, wR2 = 0.1430	
Extinction coefficient	n/a	
Largest diff. peak and hole	1.171 and -1.191 $e.Å^{-3}$	

X-ray crystal data of compound 3y (CCDC 1965998)



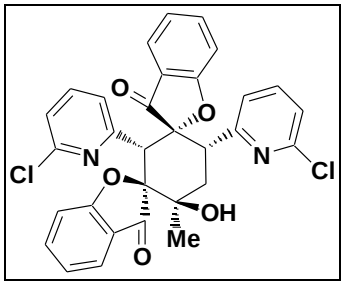
Sample preparation : A solution of compound **3y** (30 mg) in CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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.



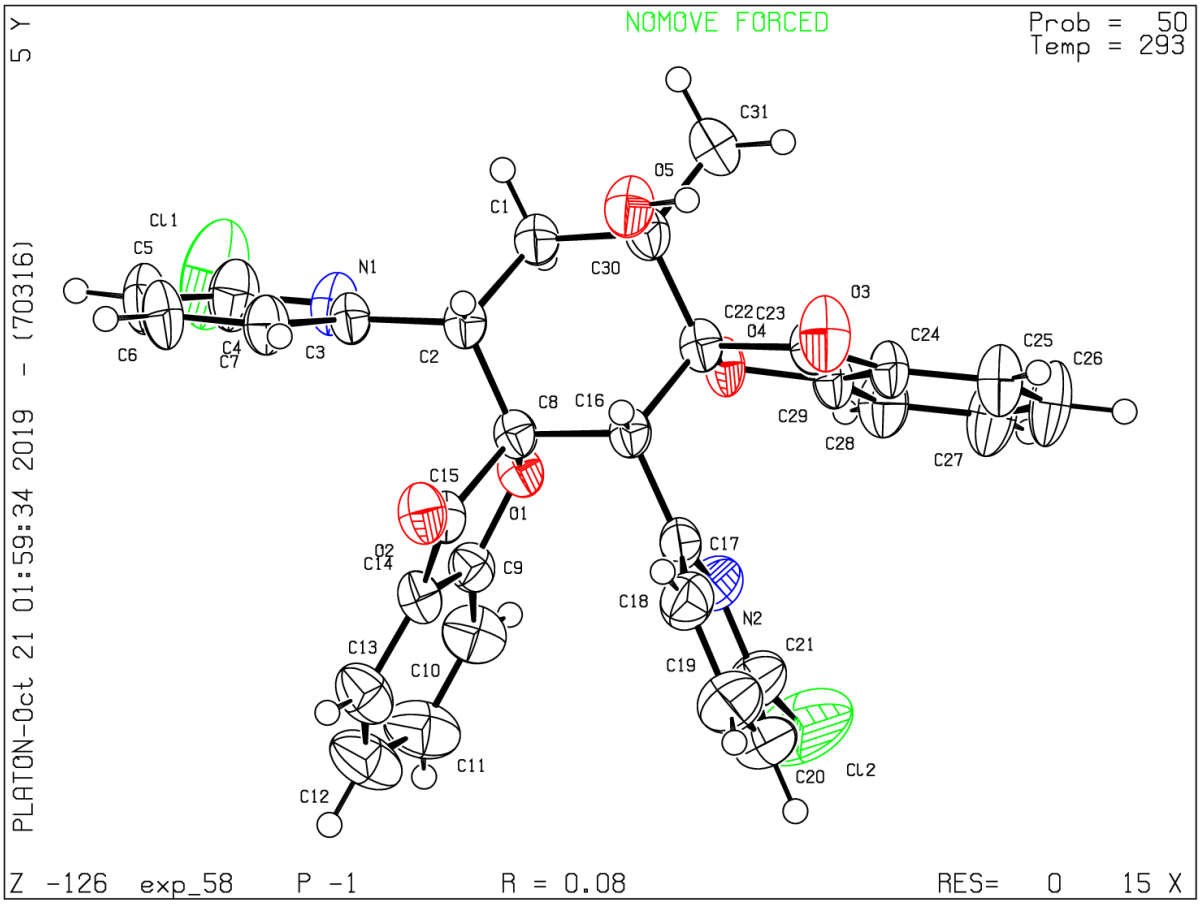
Empirical formula	C31 H22 Cl2 N2 O5	
Formula weight	573.40	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.8001(6) Å	α = 89.110(4)°.
	b = 11.3693(6) Å	β = 67.910(4)°.
	c = 13.4193(5) Å	γ = 68.542(5)°.
Volume	1406.67(13) $Å^3$	
Z	2	
Density (calculated)	1.354 Mg/m^3	
Absorption coefficient	0.274 mm^{-1}	
F(000)	592	
Crystal size	0.2 x 0.2 x 0.2 mm^3	
Theta range for data collection	2.169 to 27.038°.	
Index ranges	-13<=h<=12, -13<=k<=14, -17<=l<=16	
Reflections collected	19610	
Independent reflections	5795 [R(int) = 0.0493]	
Completeness to theta = 25.242°	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.55616	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5795 / 12 / 363	
Goodness-of-fit on F^2	1.063	
Final R indices [I>2sigma(I)]	R1 = 0.0846, wR2 = 0.2025	
R indices (all data)	R1 = 0.1426, wR2 = 0.2297	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.822 and -0.419 e.$Å^{-3}$	

X-ray crystal data of compound 3y-1 (CCDC 1965999)



Sample preparation : A solution of compound **3y-1** (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	C31 H22 Cl2 N2 O5	
Formula weight	573.40	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.9174(14) Å	α = 69.112(11)°.
	b = 11.9111(14) Å	β = 87.877(10)°.
	c = 12.3891(14) Å	γ = 65.530(12)°.
Volume	1358.3(3) $Å^3$	
Z	2	
Density (calculated)	1.402 Mg/m^3	
Absorption coefficient	0.284 mm^{-1}	
F(000)	592	
Crystal size	0.2 x 0.2 x 0.2 mm^3	
Theta range for data collection	2.026 to 27.091°.	
Index ranges	-9<=h<=13, -15<=k<=14, -15<=l<=15	
Reflections collected	17787	
Independent reflections	5593 [R(int) = 0.1441]	
Completeness to theta = 25.242°	99.6 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1.00000 and 0.47499	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5593 / 198 / 363	
Goodness-of-fit on F^2	1.014	
Final R indices [I>2sigma(I)]	R1 = 0.1112, wR2 = 0.2688	
R indices (all data)	R1 = 0.1930, wR2 = 0.3225	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.576 and -0.497 $e.Å^{-3}$	