

# Supporting Information

## One-pot Construction of Dispirocyclohexanes via K<sub>2</sub>CO<sub>3</sub>-Mediated Desulfonylative Michael-Michael-aldol (2+2+2) Annulation of Sulfonyl 2-Pyridyl Flavanones and Methylketones

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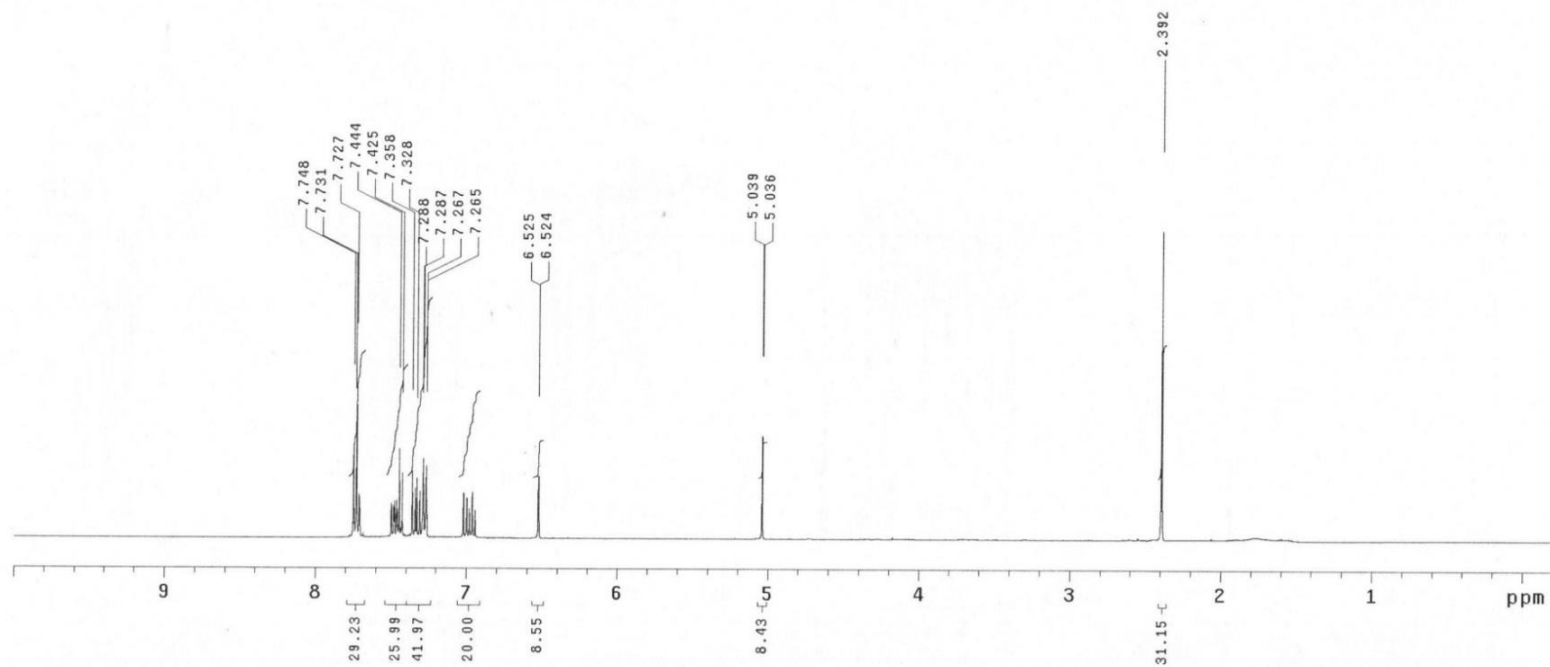
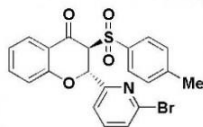
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# Compound 2a (<sup>1</sup>H-NMR spectral data)

HANBr35

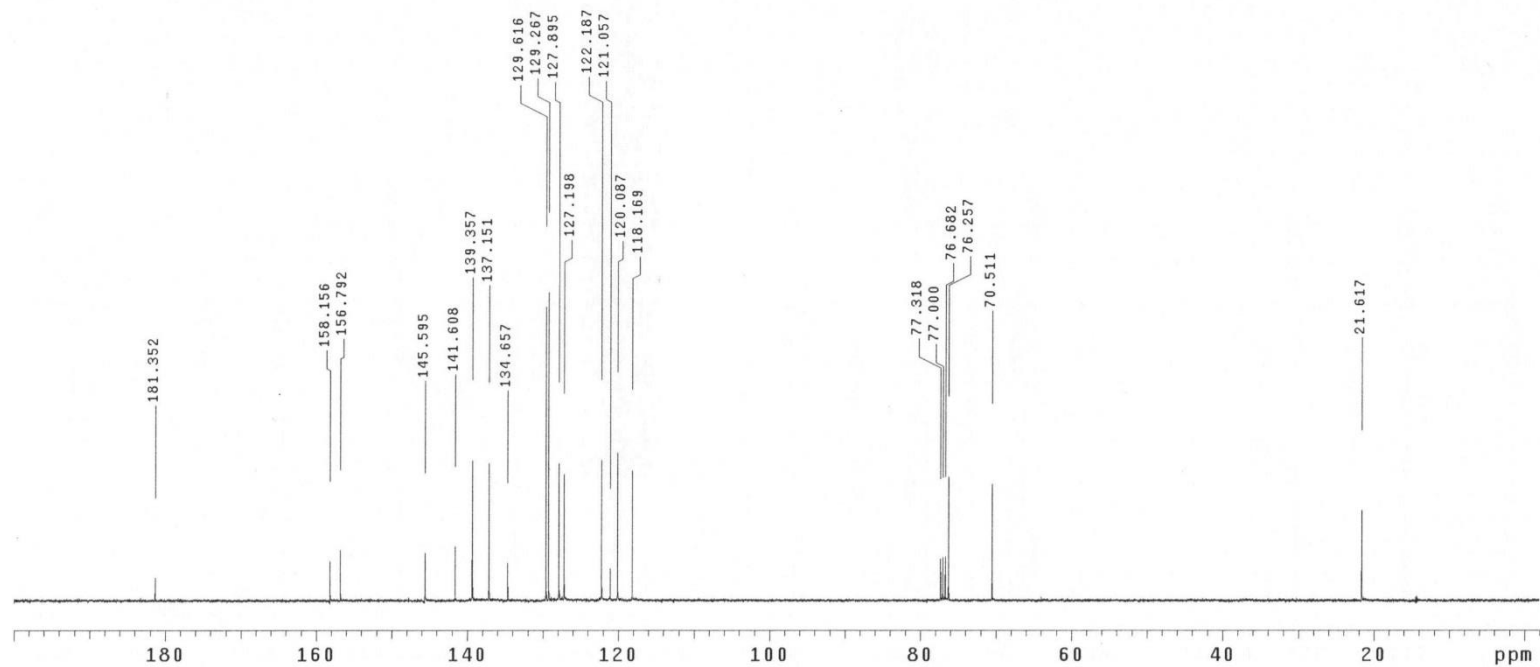
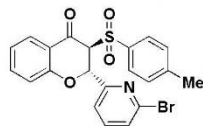
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Date: Mar 6 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2a (<sup>13</sup>C-NMR spectral data)

HANBr35

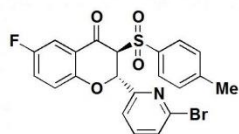
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Total 528 repetitions



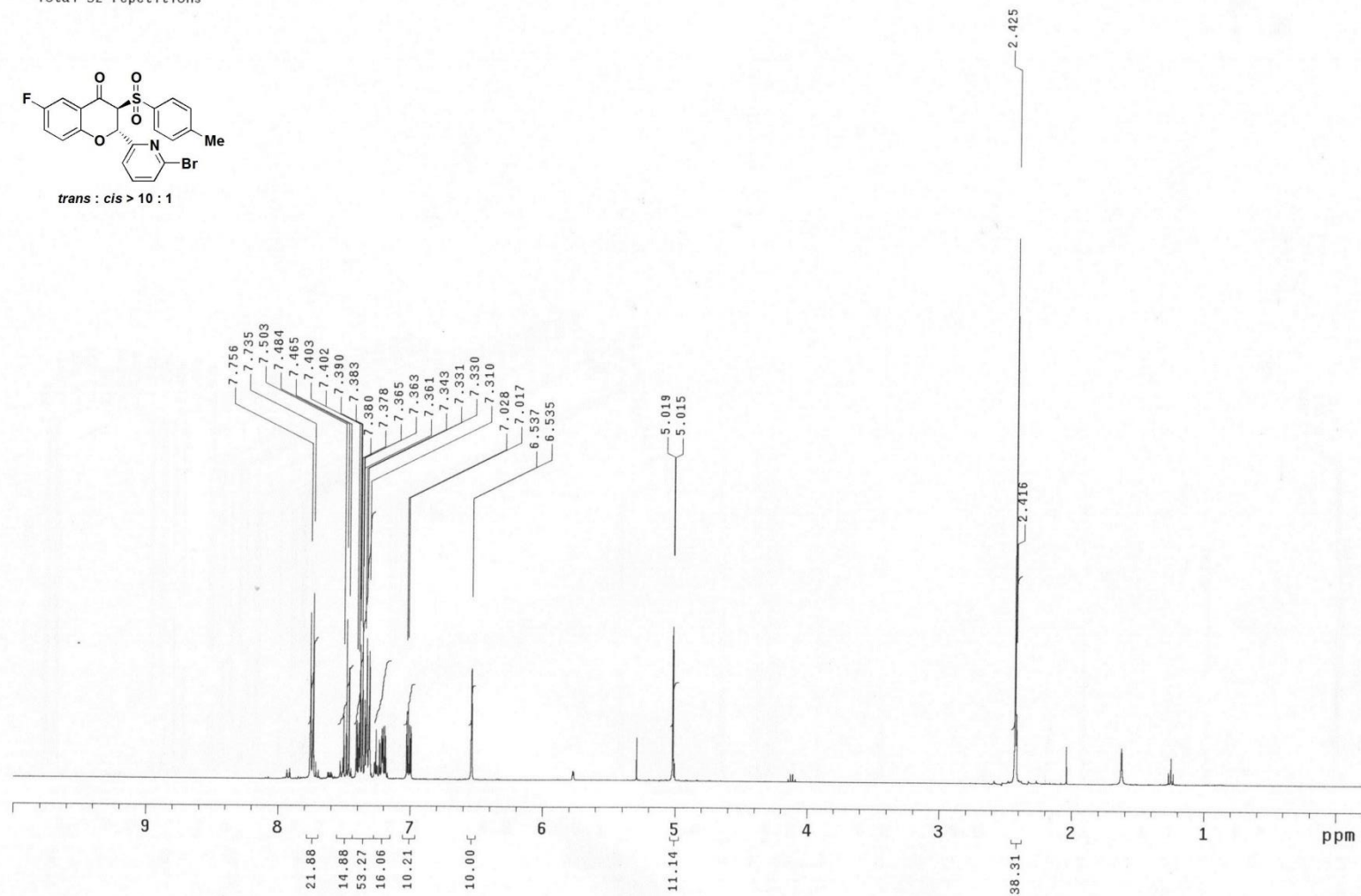
# Compound 2b (<sup>1</sup>H-NMR spectral data)

HAFPYBr

Pulse Sequence: s2pu1  
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Date: Jul 30 2018  
Solvent: CDC13  
Ambient temperature  
Total 32 repetitions



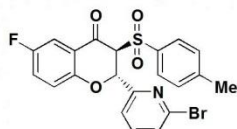
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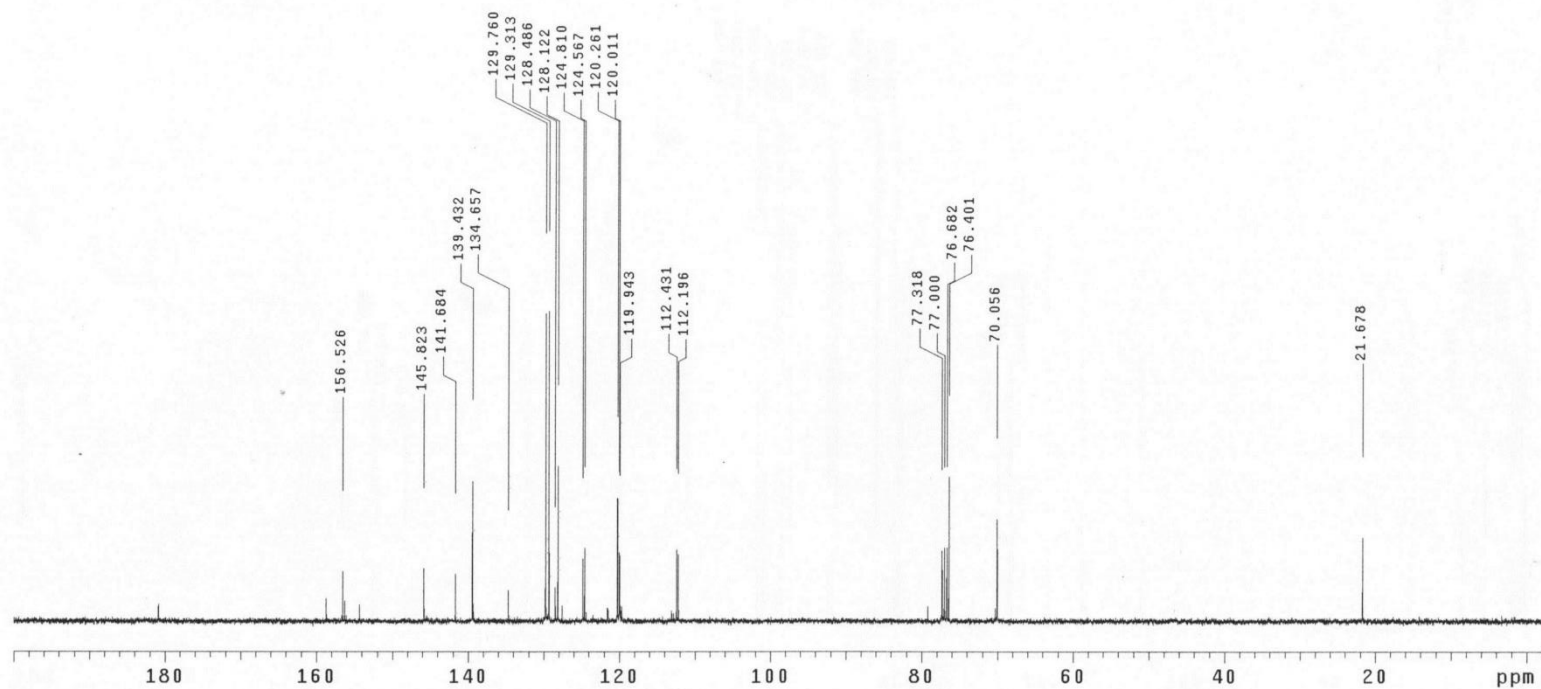
# Compound 2b (<sup>13</sup>C-NMR spectral data)

HAFPYBr

Pulse Sequence: s2pu1  
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Date: Jul 30 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 880 repetitions



*trans* : *cis* > 10 : 1



# Compound 2c (<sup>1</sup>H-NMR spectral data)

HABrN50

Pulse Sequence: s2pu1

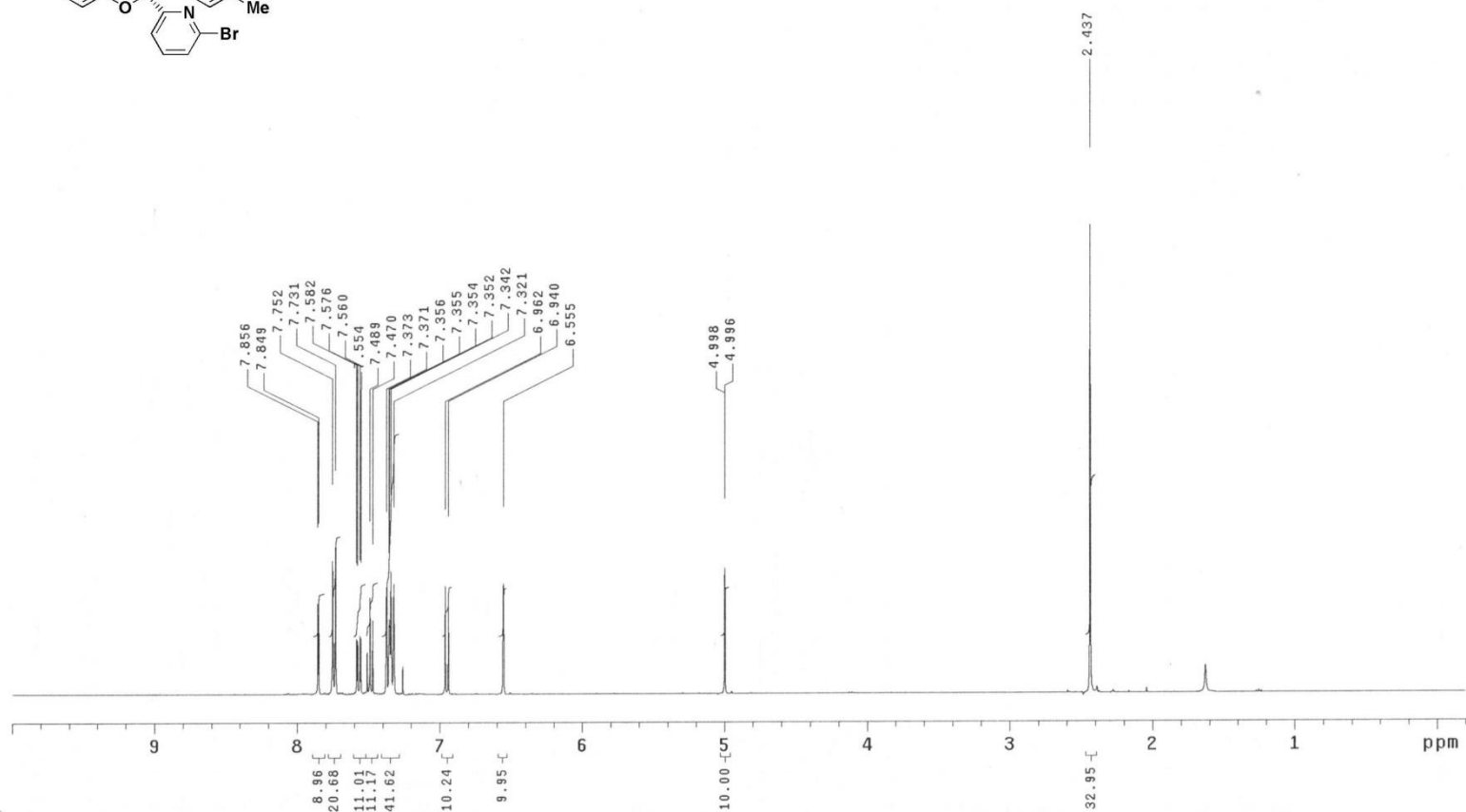
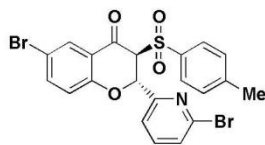
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Date: Apr 16 2018

Solvent: CDCl<sub>3</sub>

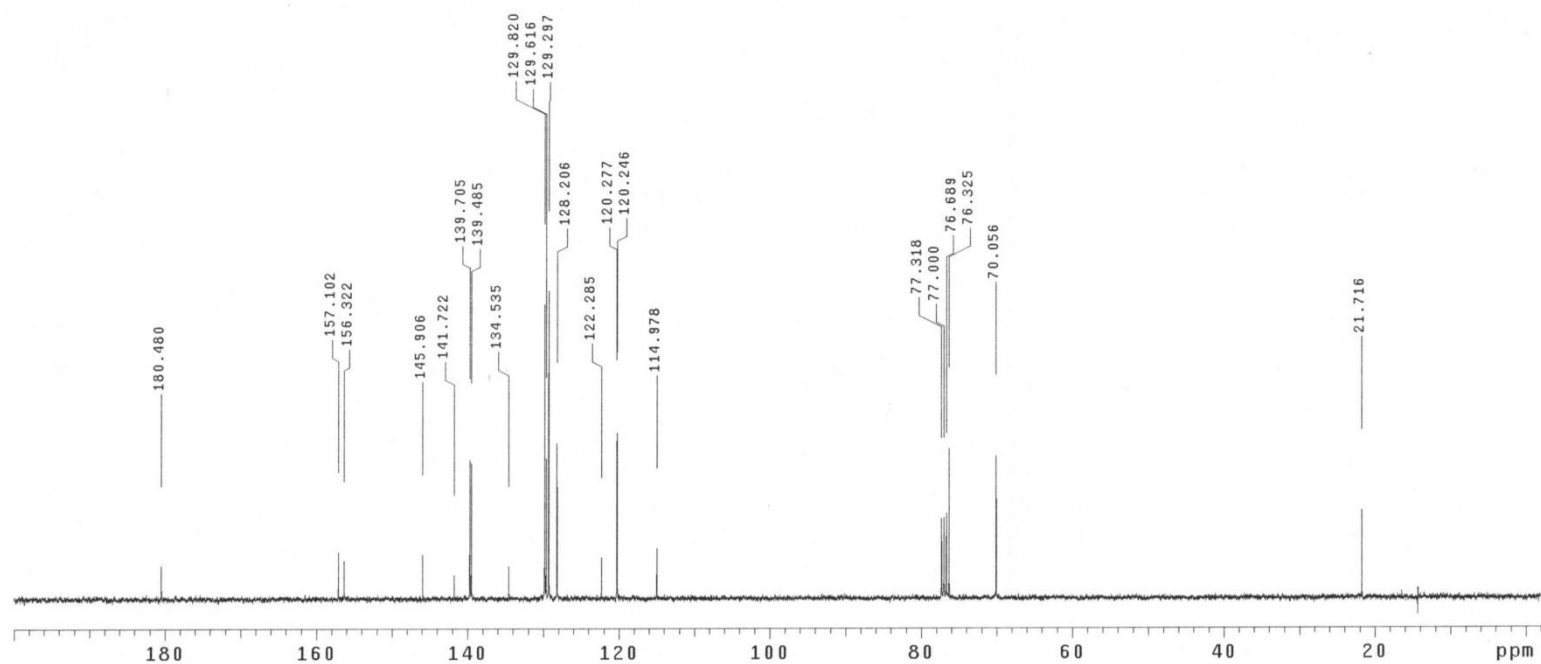
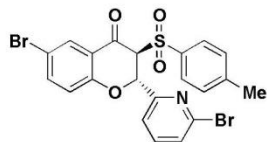
Ambient temperature

Total 32 repetitions



# Compound 2c (<sup>13</sup>C-NMR spectral data)

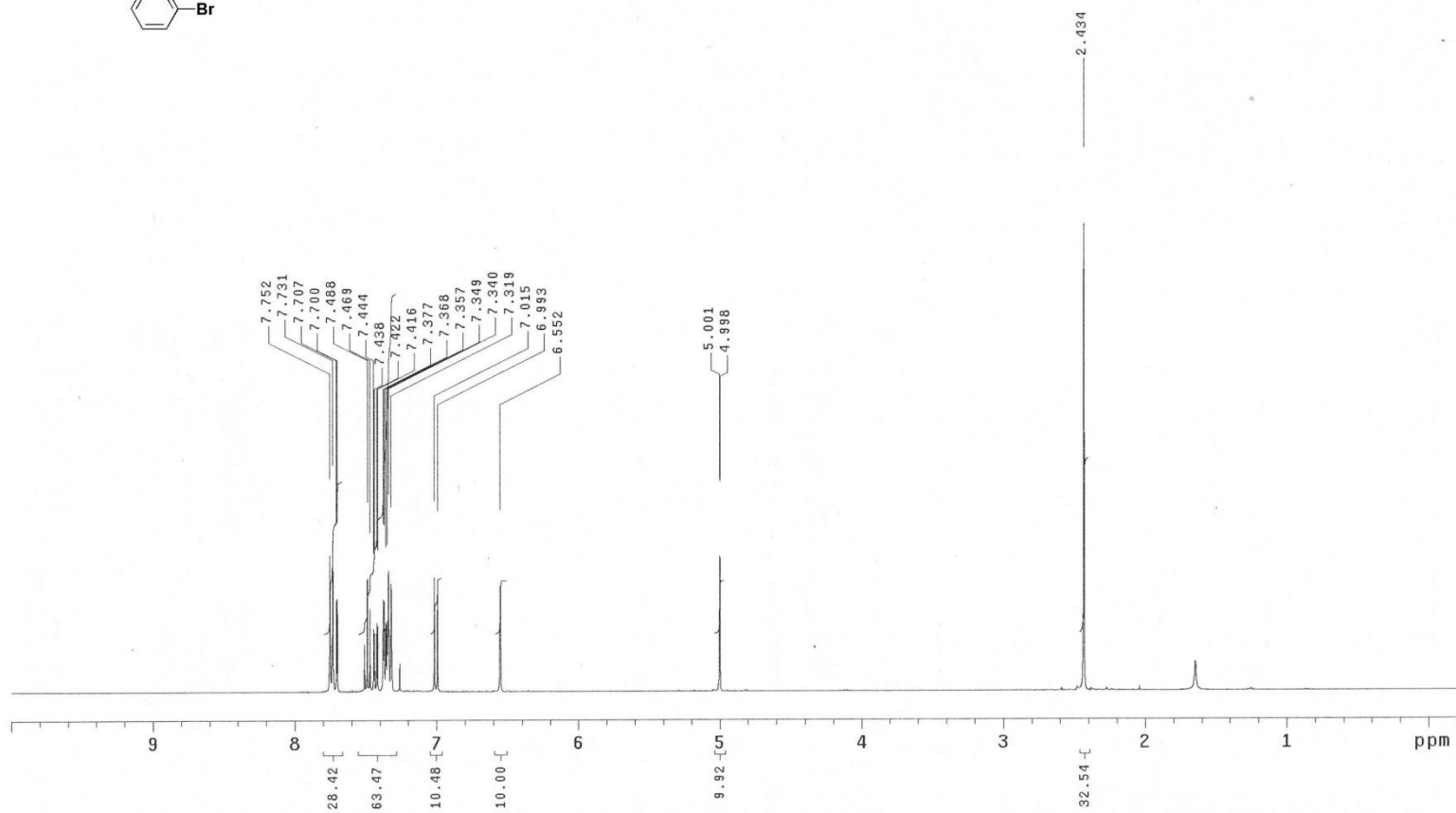
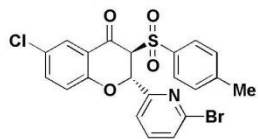
HABrN50  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
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Solvent: CDC13  
Ambient temperature  
Total 752 Repetitions



# Compound 2d (<sup>1</sup>H-NMR spectral data)

HAC150

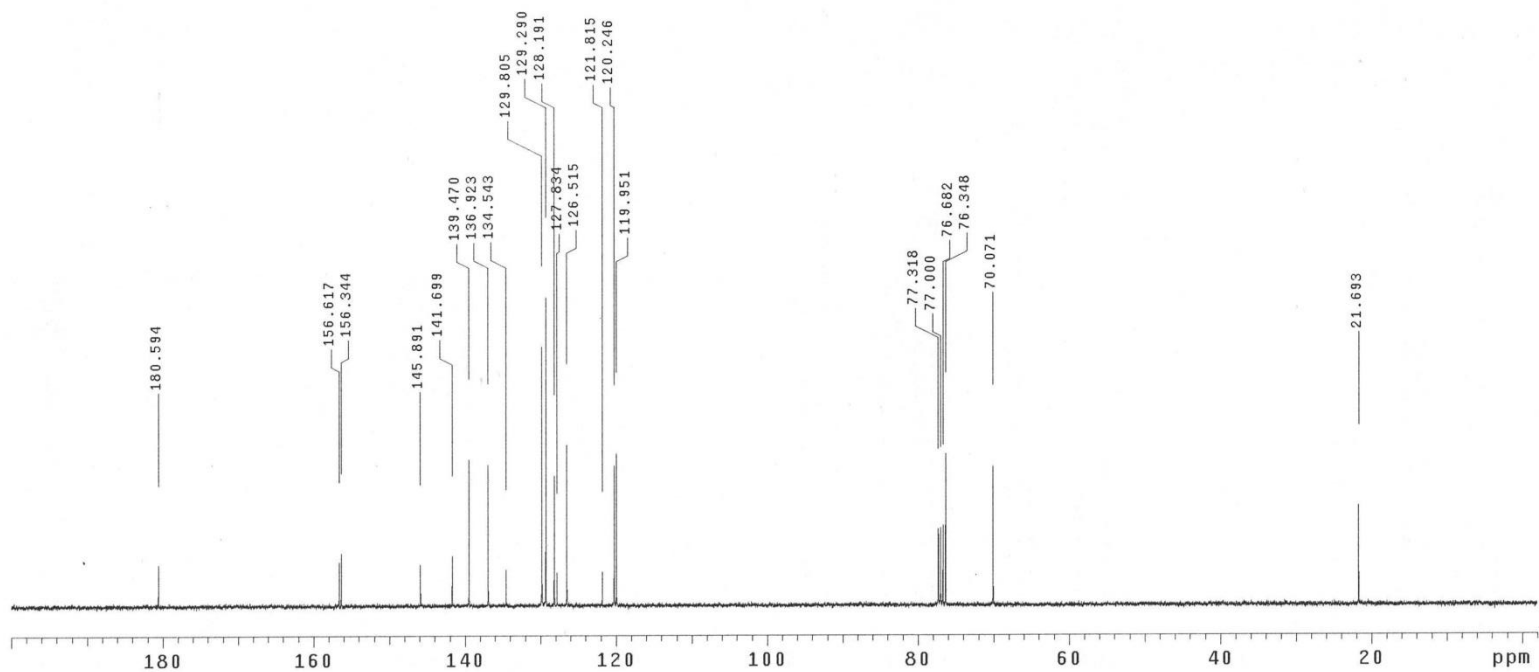
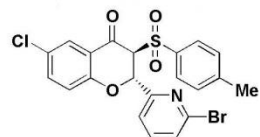
Pulse Sequence: s2pu1  
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Date: Apr 18 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions





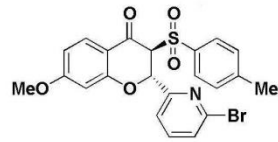
# Compound 2d (<sup>13</sup>C-NMR spectral data)

HAC150  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 18 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 752 repetitions

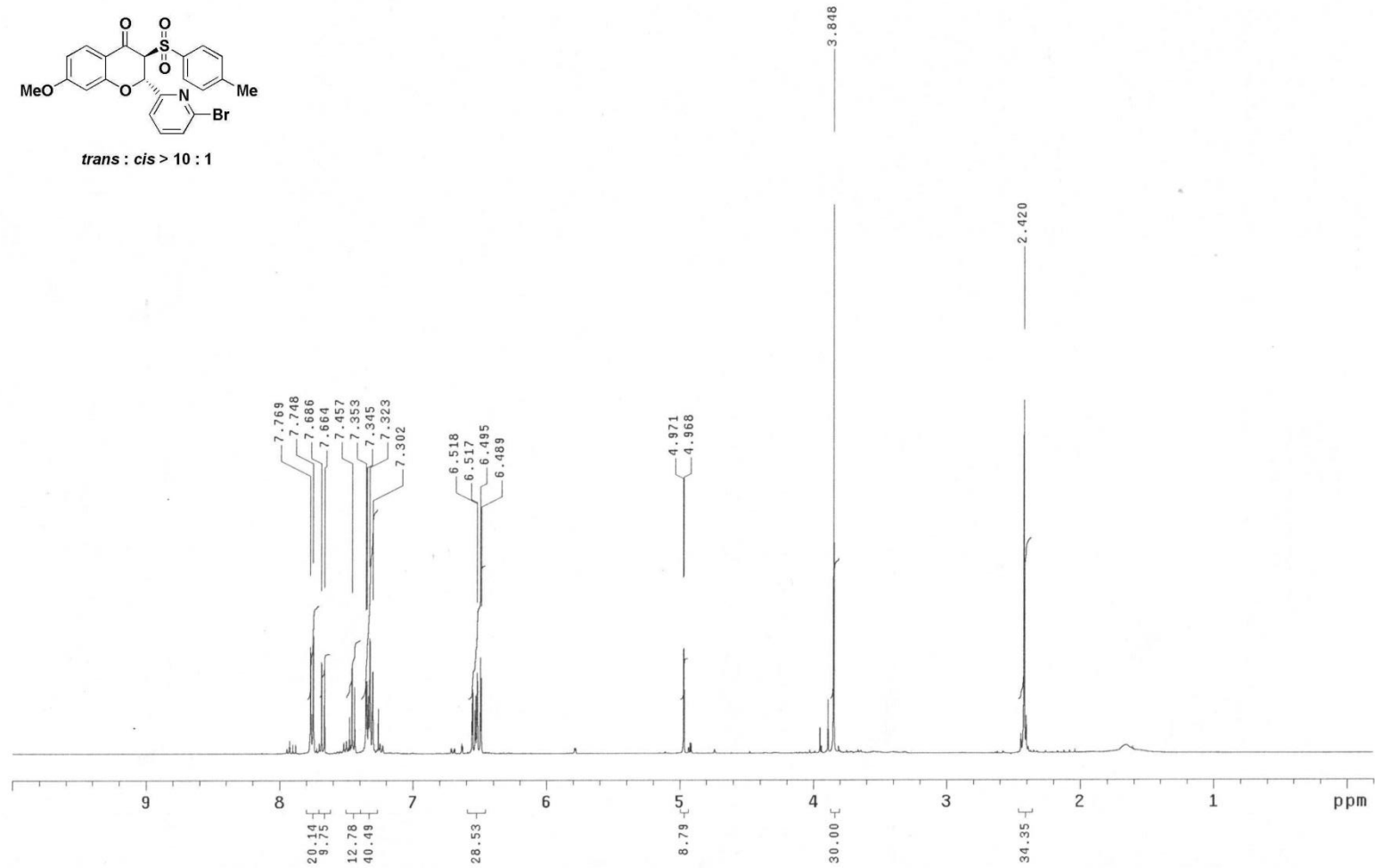


# Compound 2e (<sup>1</sup>H-NMR spectral data)

HA0Br44  
Pulse Sequence: s2pu1  
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Solvent: CDC13  
Ambient temperature  
Total 64 repetitions

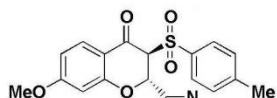


*trans* : *cis* > 10 : 1

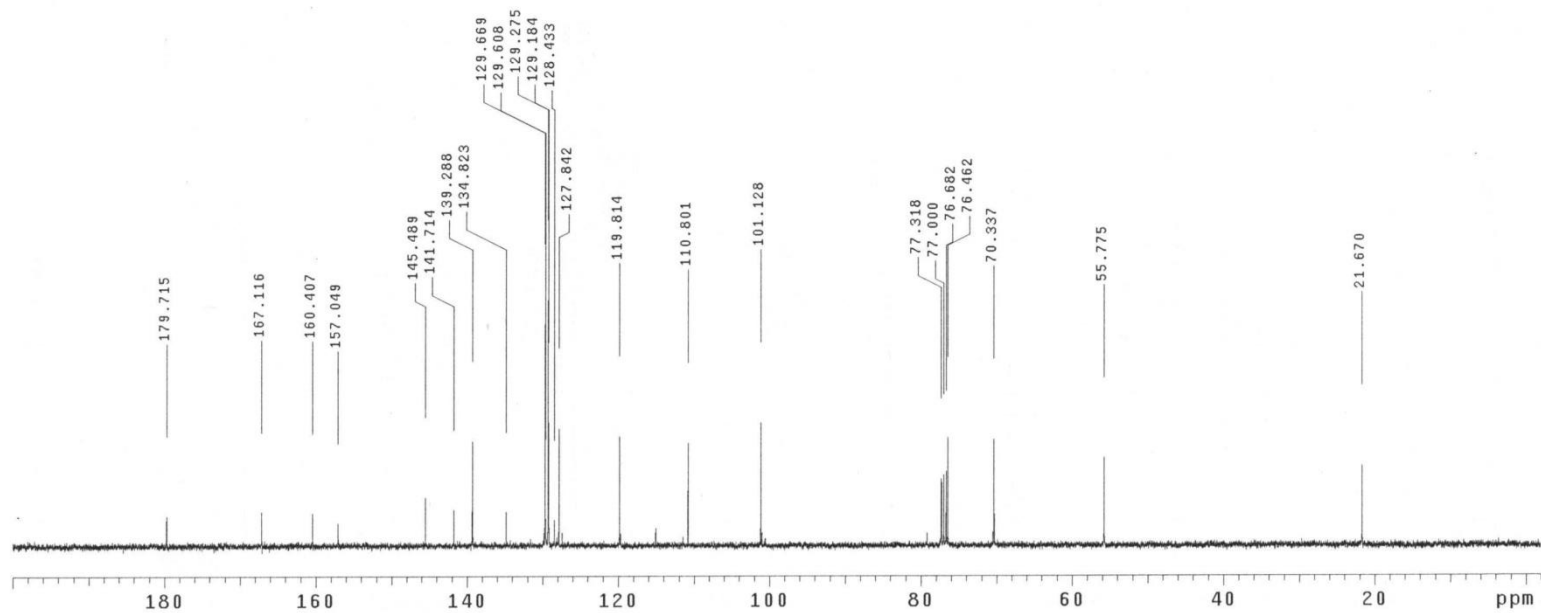


# Compound 2e (<sup>13</sup>C-NMR spectral data)

HA0Br44  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 16 2018  
Solvent: CDC13  
Ambient temperature  
Total 1200 repetitions

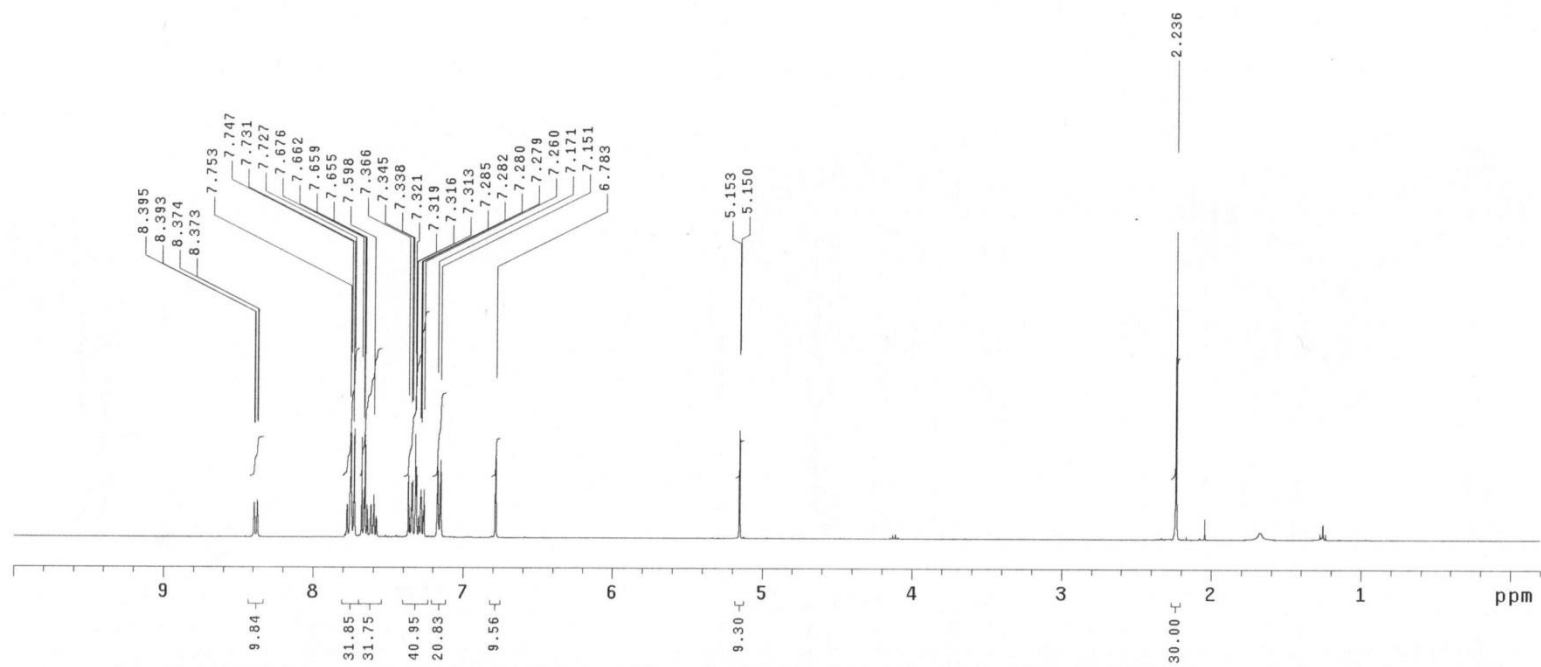
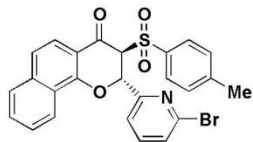


*trans* : *cis* > 10 : 1



# Compound 2f (<sup>1</sup>H-NMR spectral data)

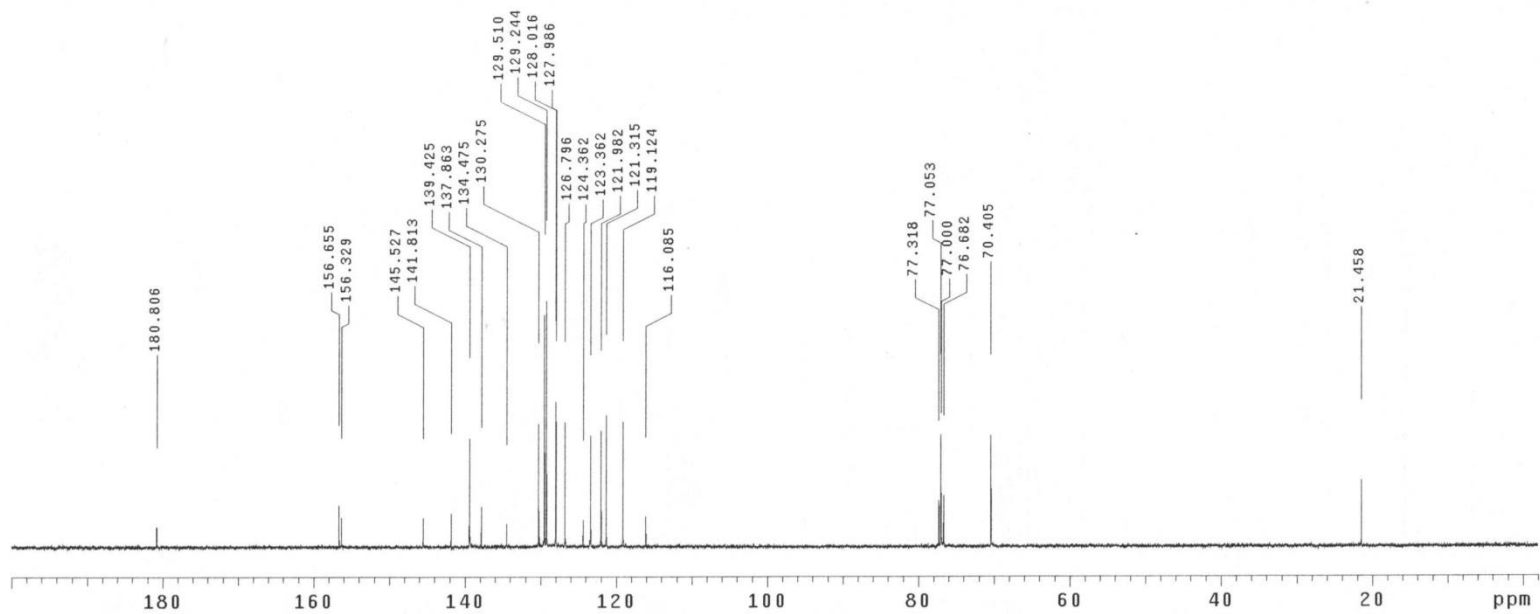
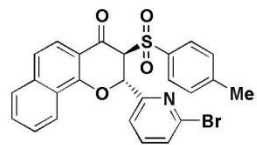
HA1070411  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 17 2018  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions



# Compound 2f (<sup>13</sup>C-NMR spectral data)

HA1070411

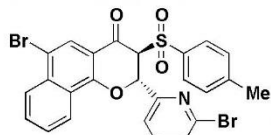
Pulse Sequence: s2pu1  
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Date: Apr 17 2018  
Solvent: CDCl3  
Ambient temperature  
Total 1312 repetitions



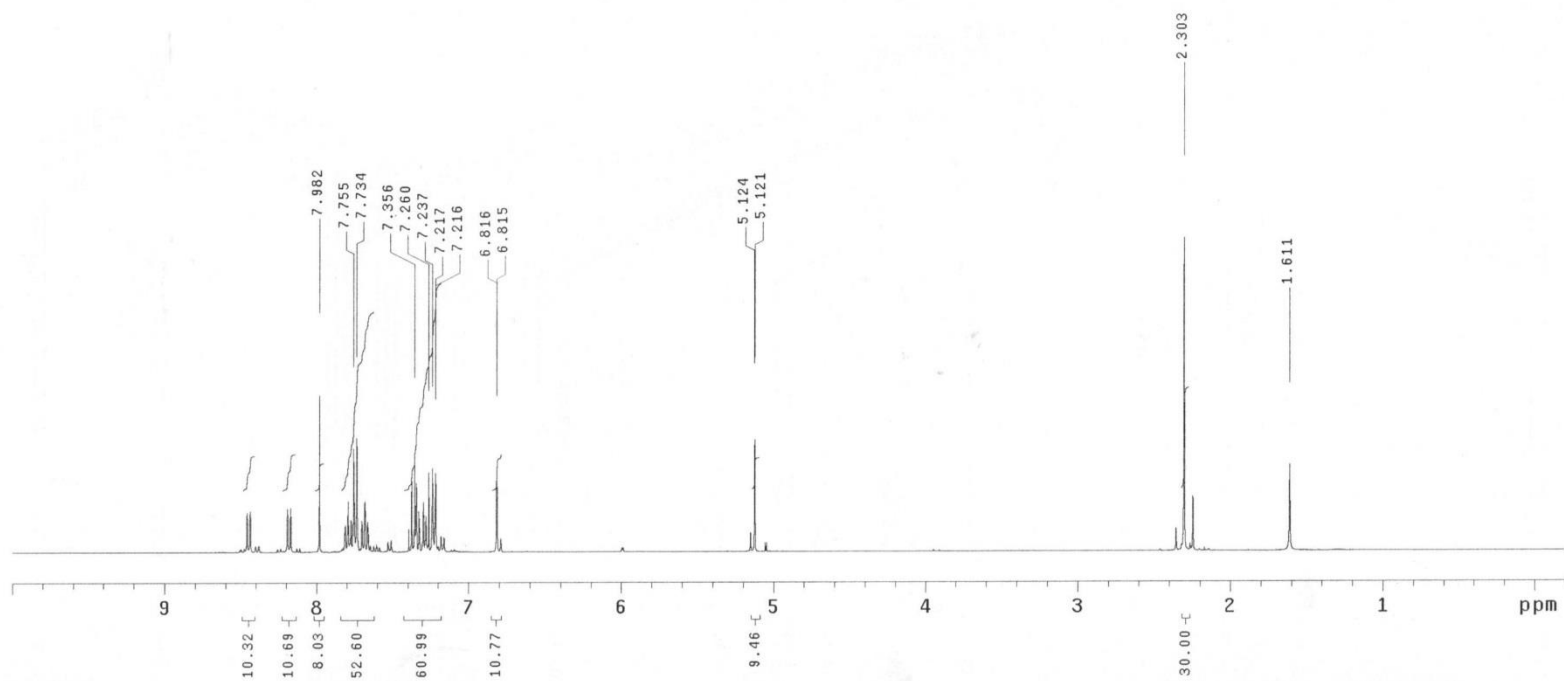
# Compound 2g (<sup>1</sup>H-NMR spectral data)

HA4BrNAPB

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 31 2019  
Solvent: CDC13  
Ambient temperature  
Total 32 repetitions



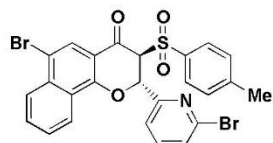
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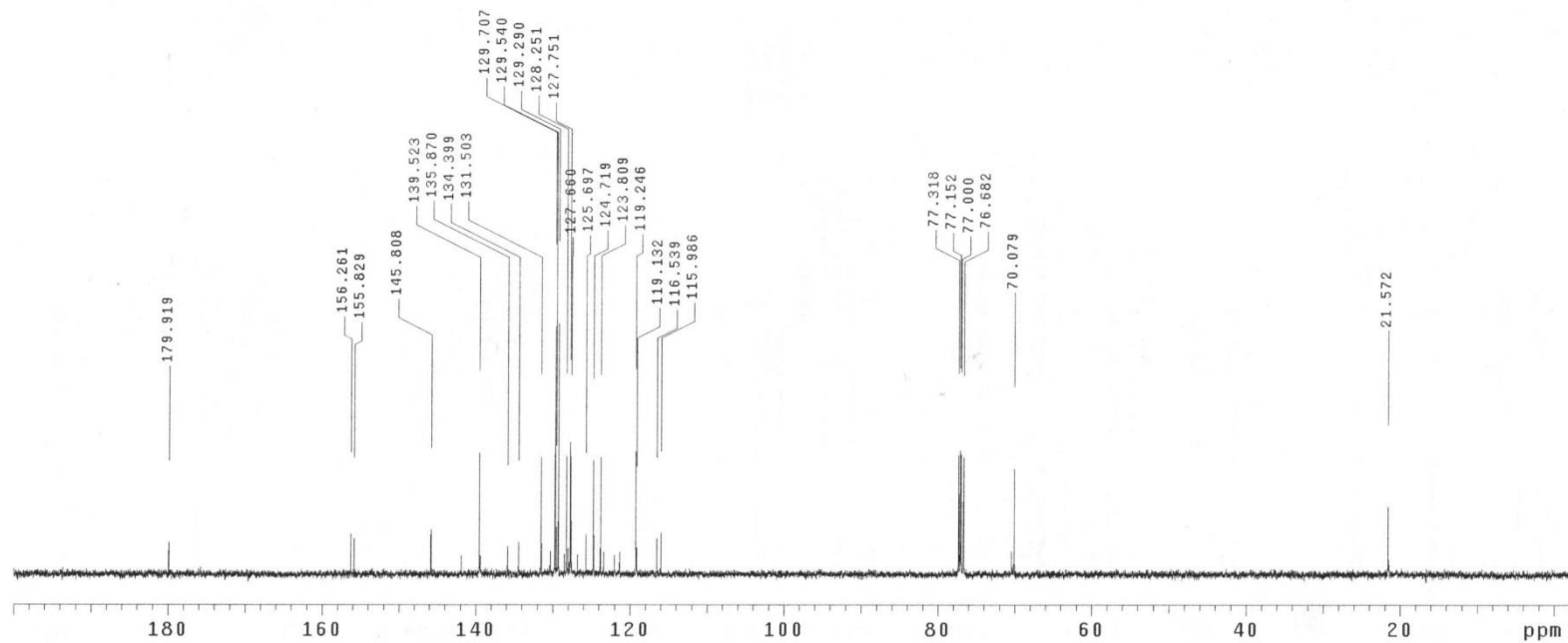
# Compound 2g (<sup>13</sup>C-NMR spectral data)

HA4BrNAPB

Pulse Sequence: s2pu1  
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Date: Jan 31 2019  
Solvent: CDCl3  
Ambient temperature  
Total 1616 repetitions



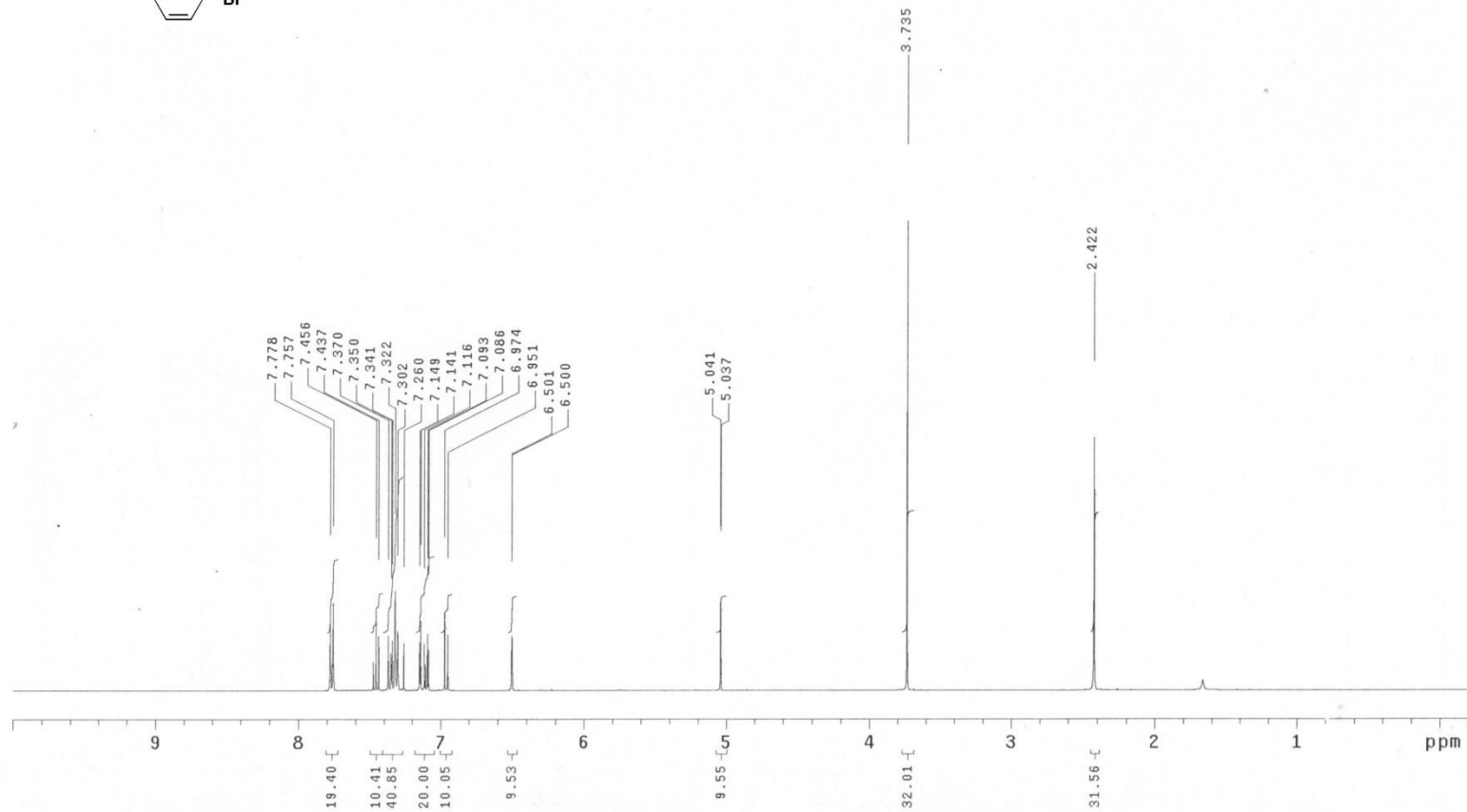
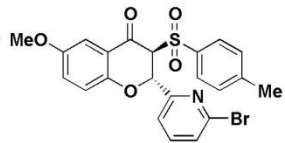
*trans* : *cis* > 8 : 1



# Compound 2h (<sup>1</sup>H-NMR spectral data)

KT40MePB

Pulse Sequence: s2pu1  
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Date: Aug 21 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions

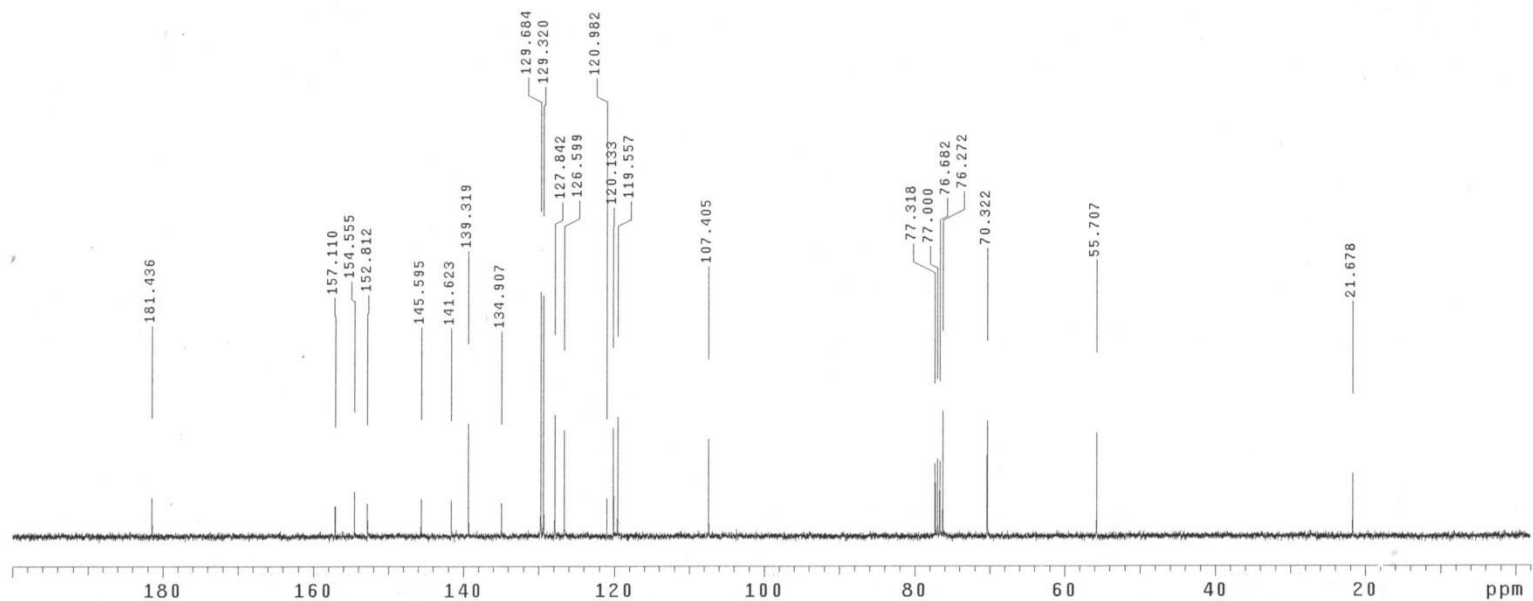
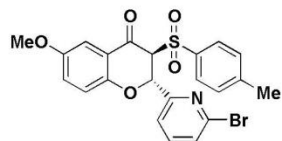




# Compound 2h (<sup>13</sup>C-NMR spectral data)

KT40MePB

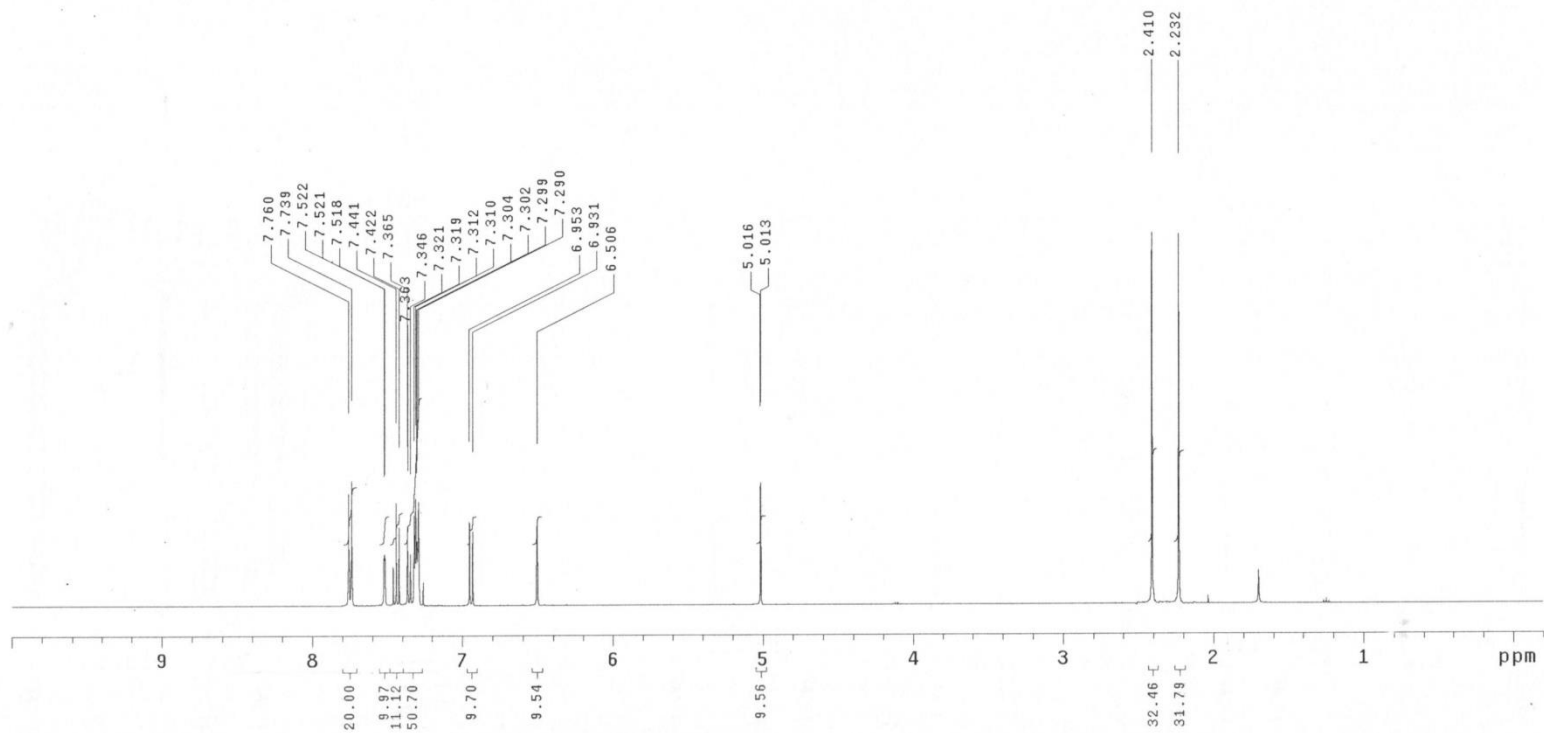
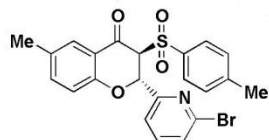
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 21 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 816 repetitions



# Compound 2i (<sup>1</sup>H-NMR spectral data)

KT4MePB

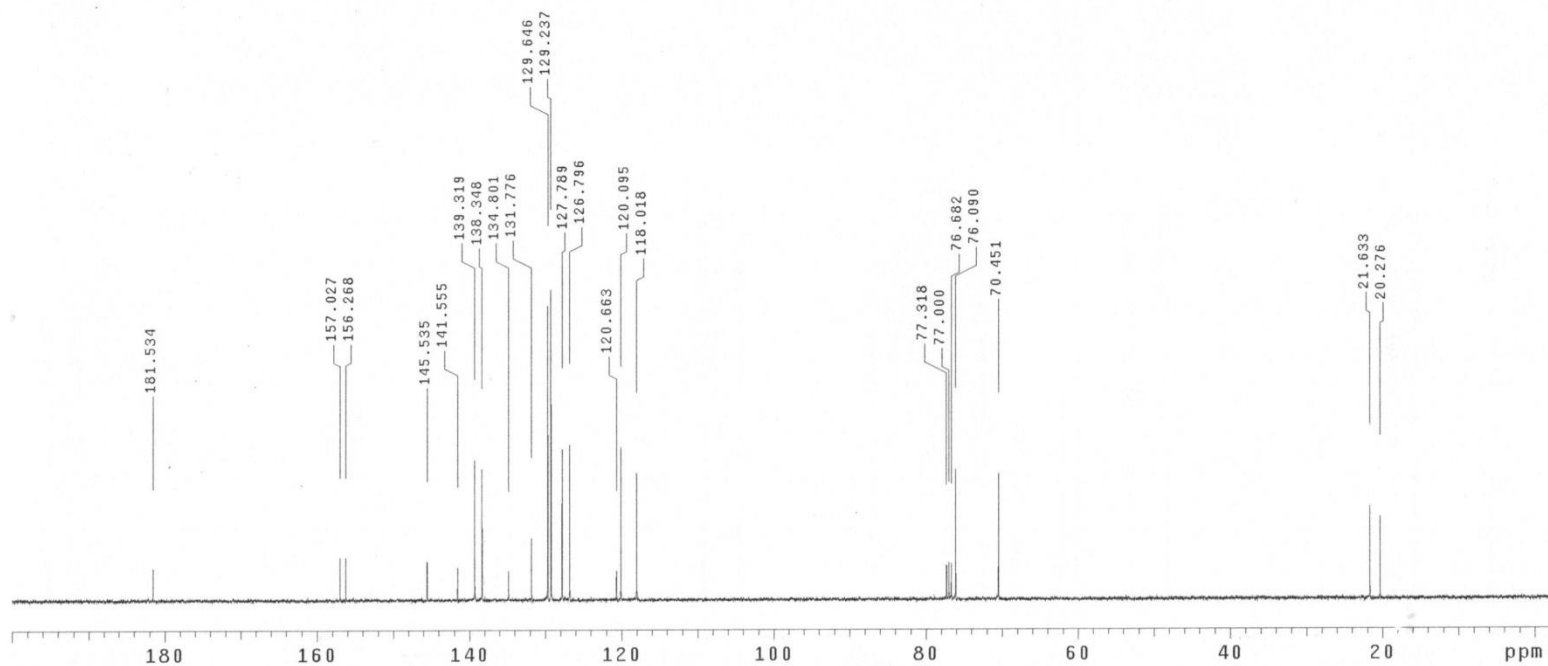
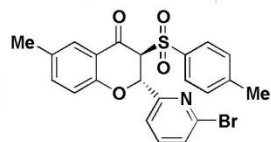
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 11 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions



# Compound 2i (<sup>13</sup>C-NMR spectral data)

KT4MePB

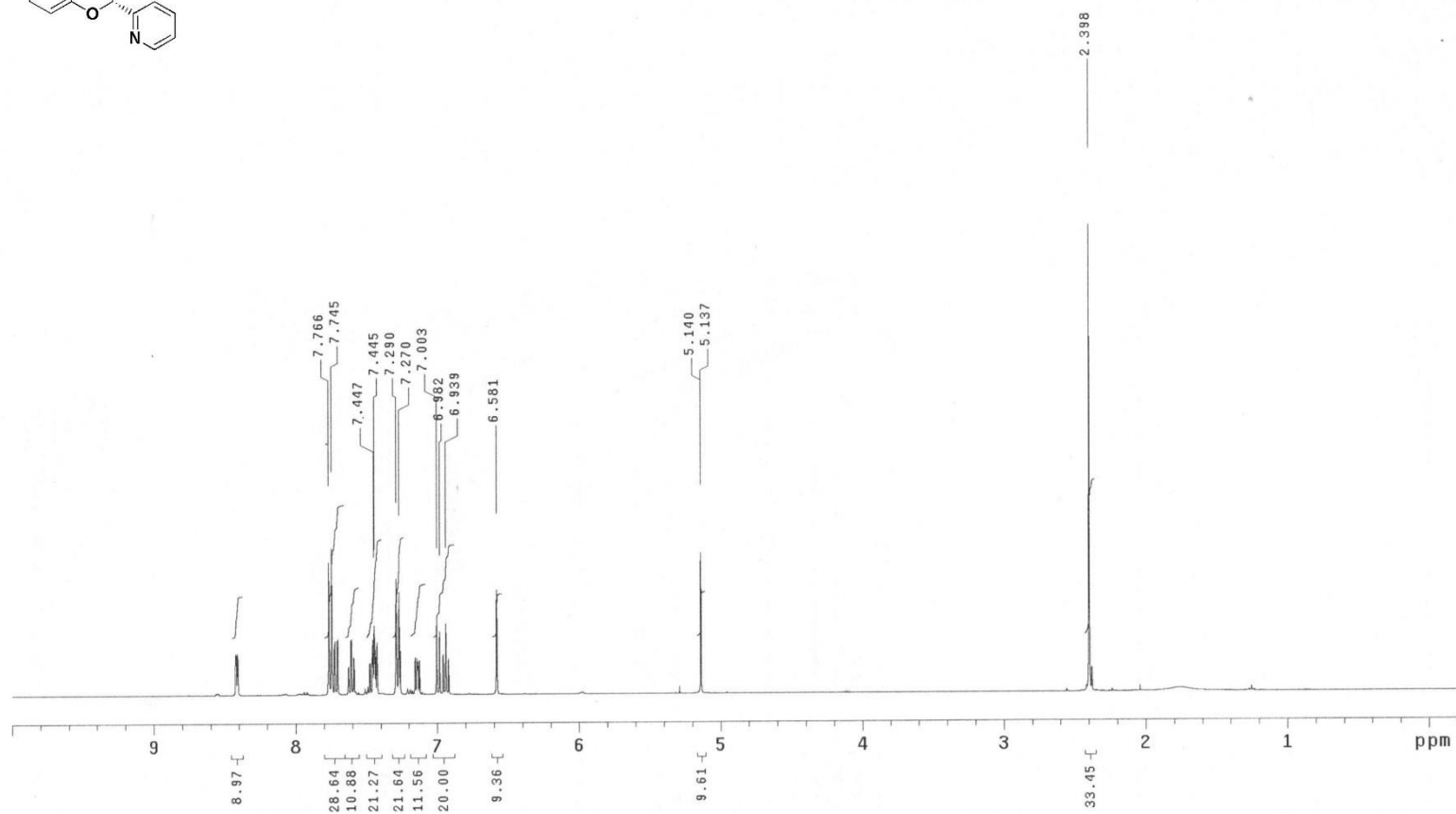
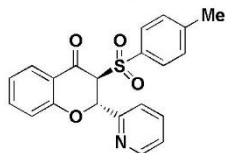
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 11 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 560 repetitions



# Compound 2j (<sup>1</sup>H-NMR spectral data)

HATsPy

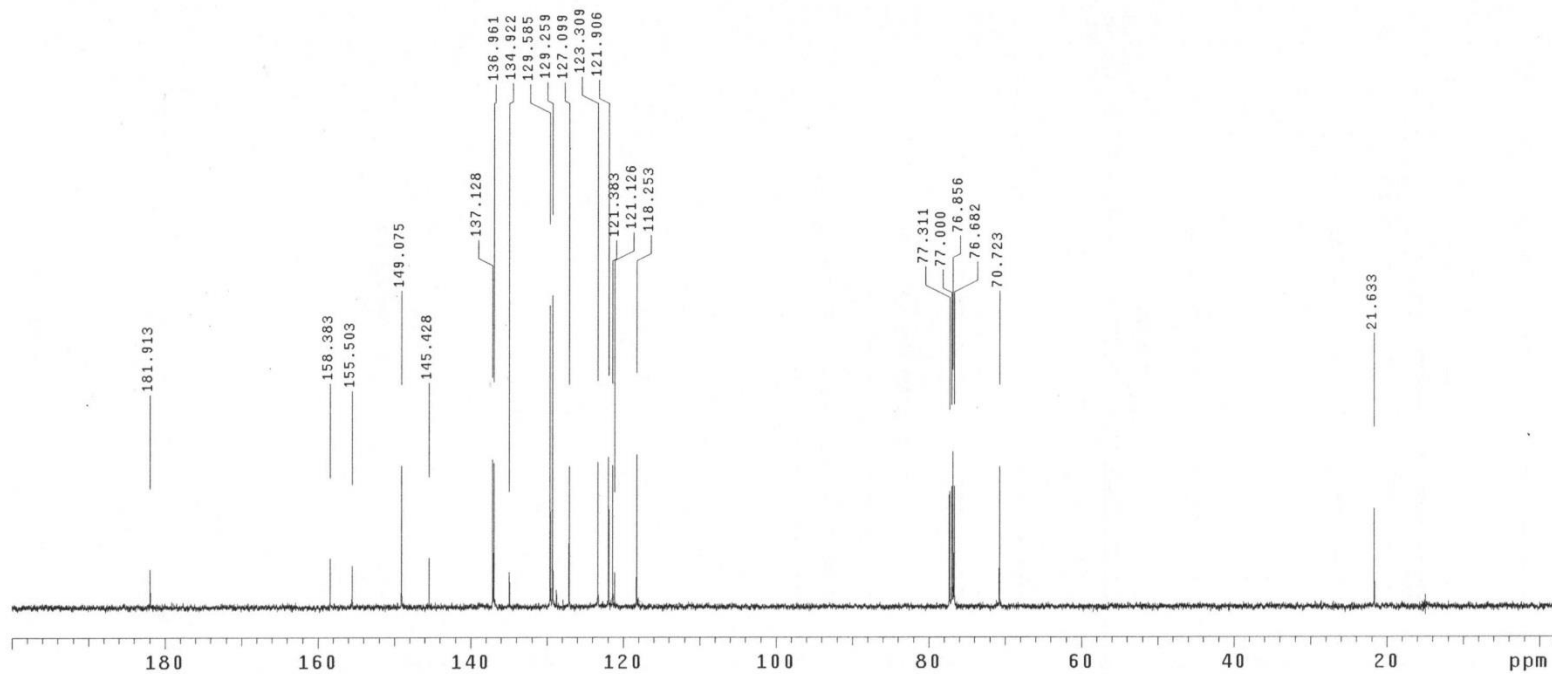
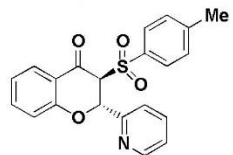
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Date: May 2 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 64 repetitions



# Compound 2j (<sup>13</sup>C-NMR spectral data)

HATsPy

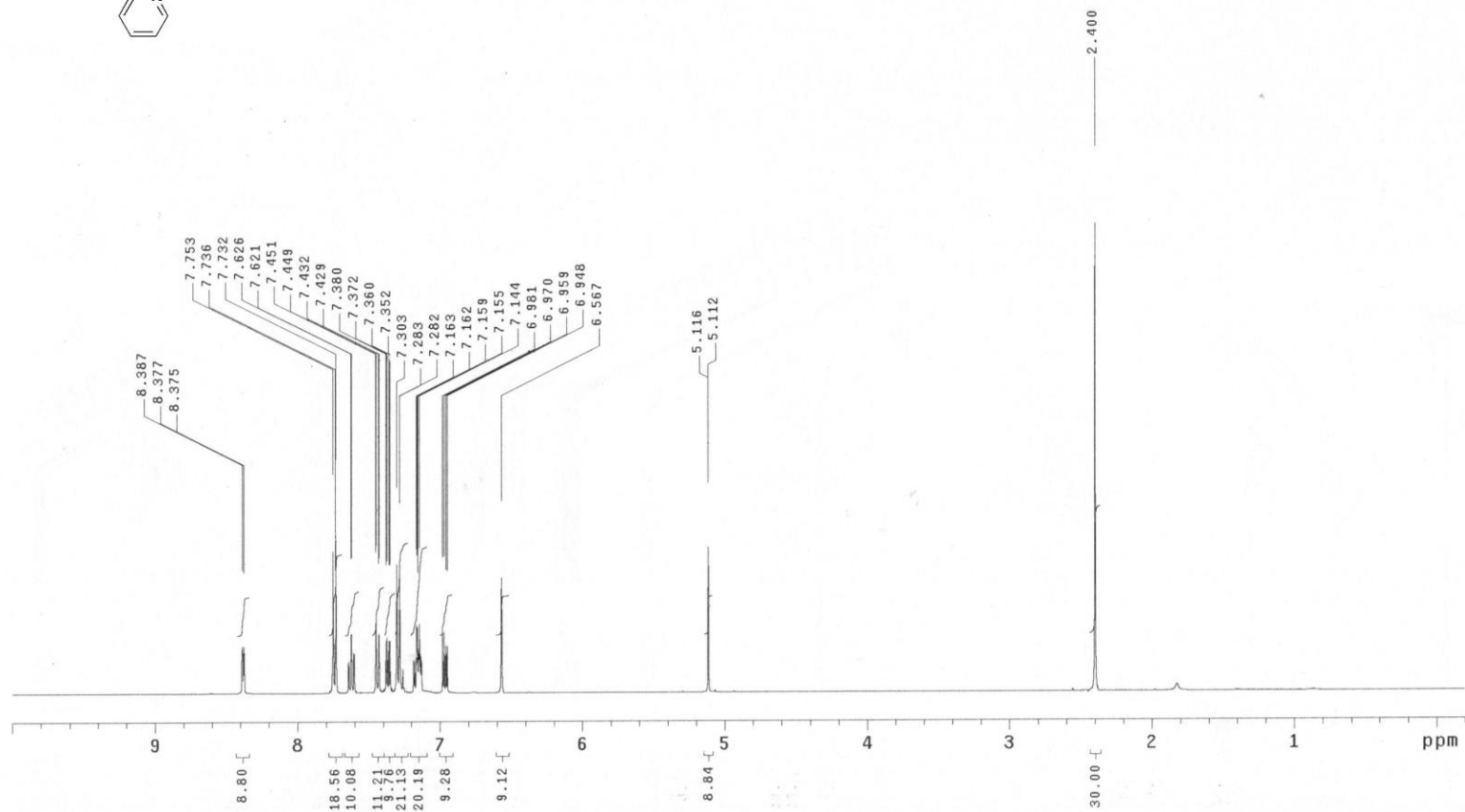
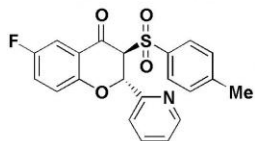
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Ambient temperature  
Total 832 repetitions



# Compound 2k (<sup>1</sup>H-NMR spectral data)

HAFTsP

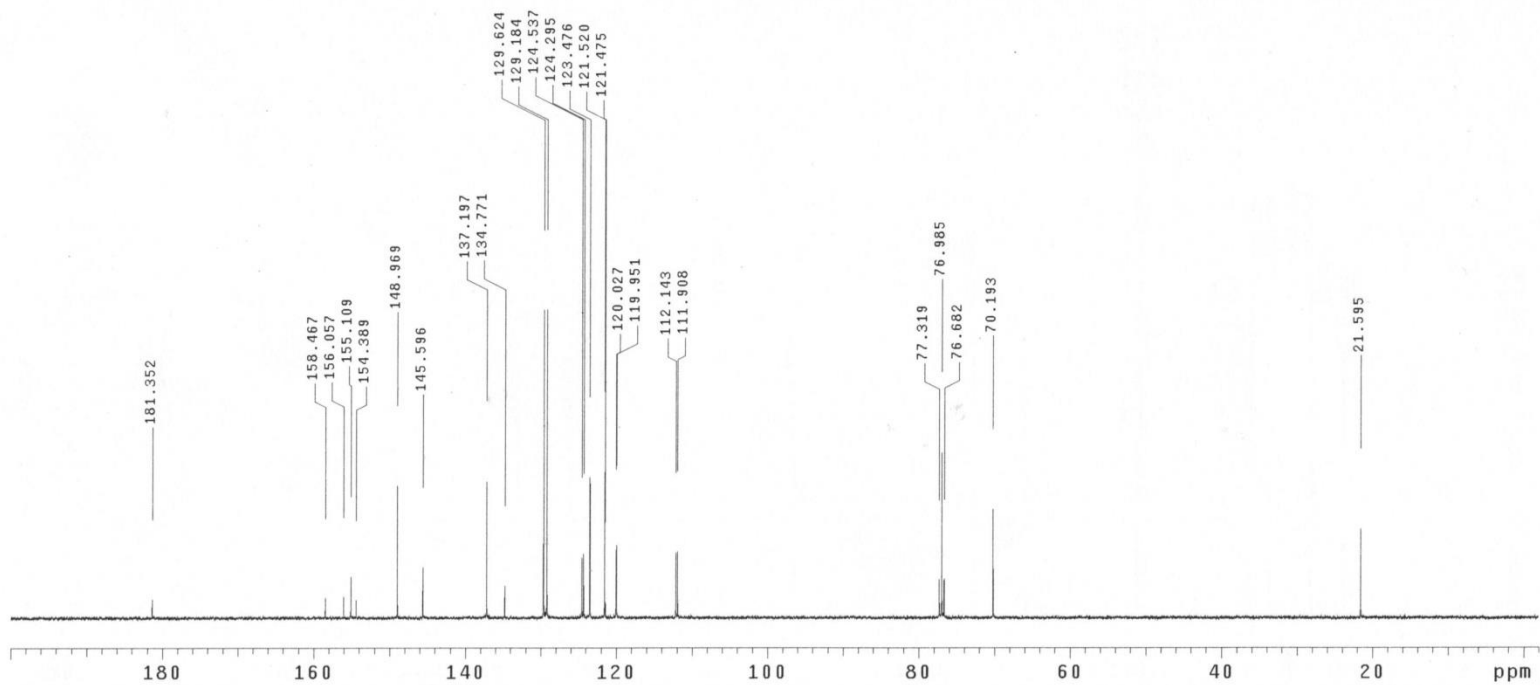
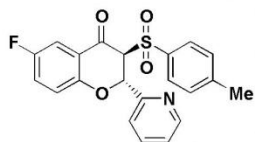
Pulse Sequence: s2pu1  
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Date: Feb 27 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2k (<sup>13</sup>C-NMR spectral data)

HAFTsP

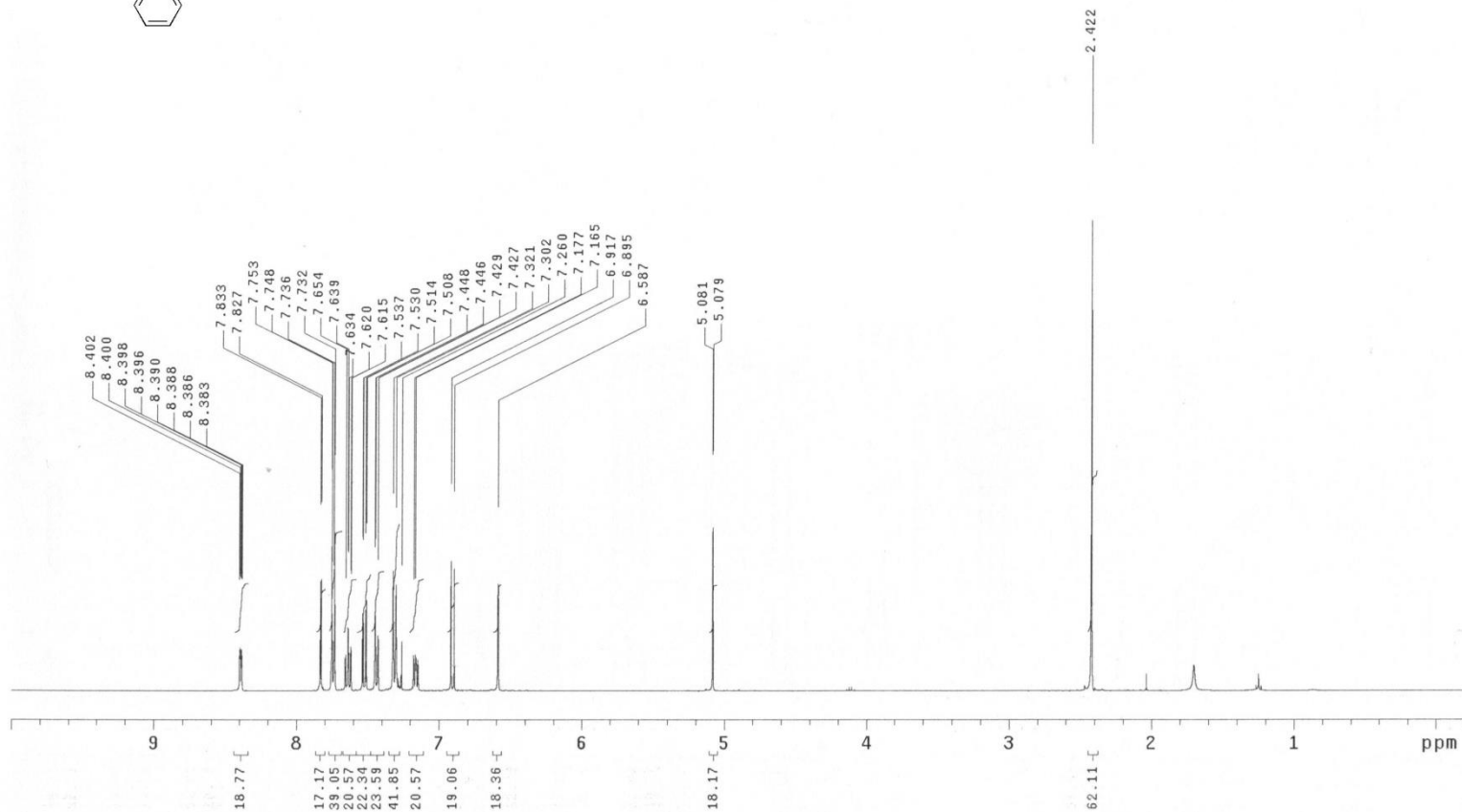
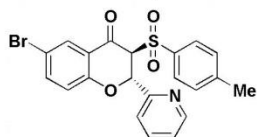
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Date: Feb 27 2019  
Solvent: CDCl3  
Ambient temperature  
Total 432 repetitions



# Compound 2I (<sup>1</sup>H-NMR spectral data)

KTBrPY

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jun 10 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions

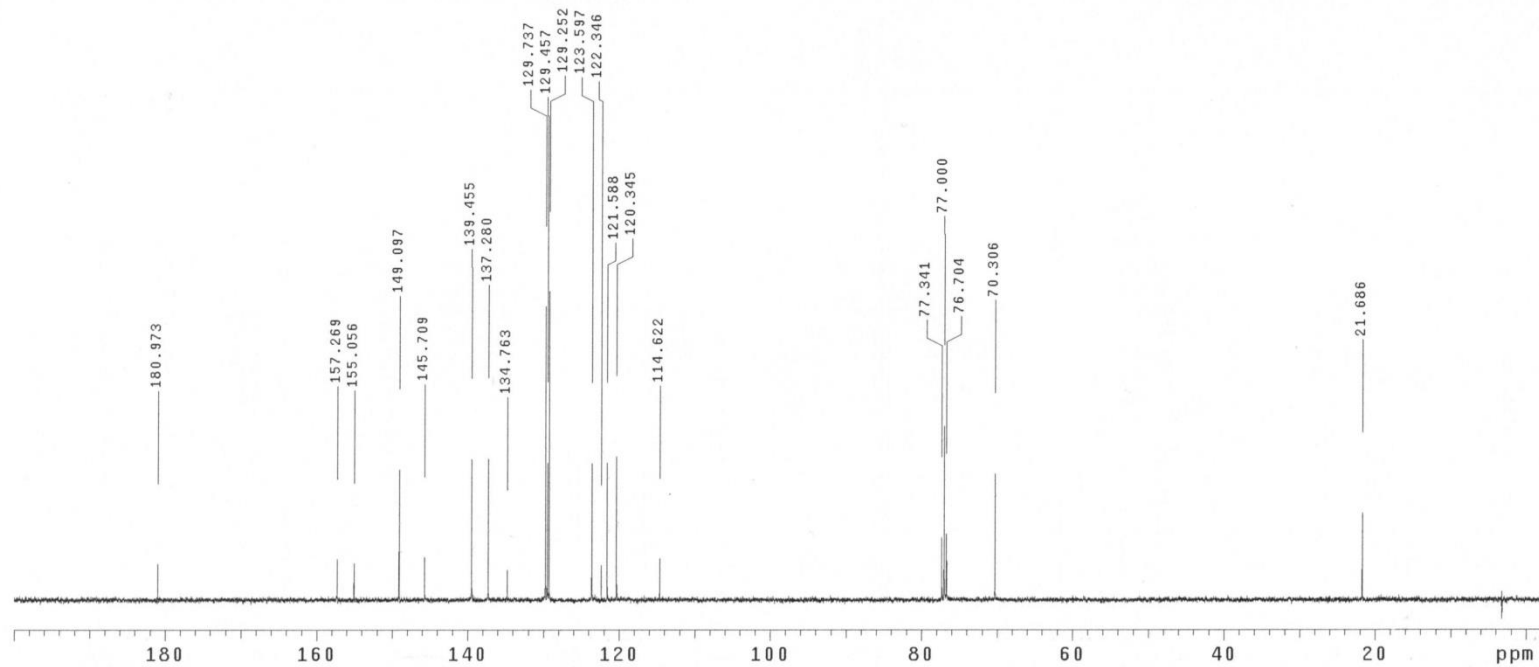
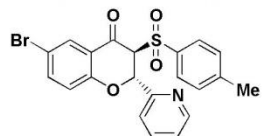




# Compound 2l (<sup>13</sup>C-NMR spectral data)

KTBrPY

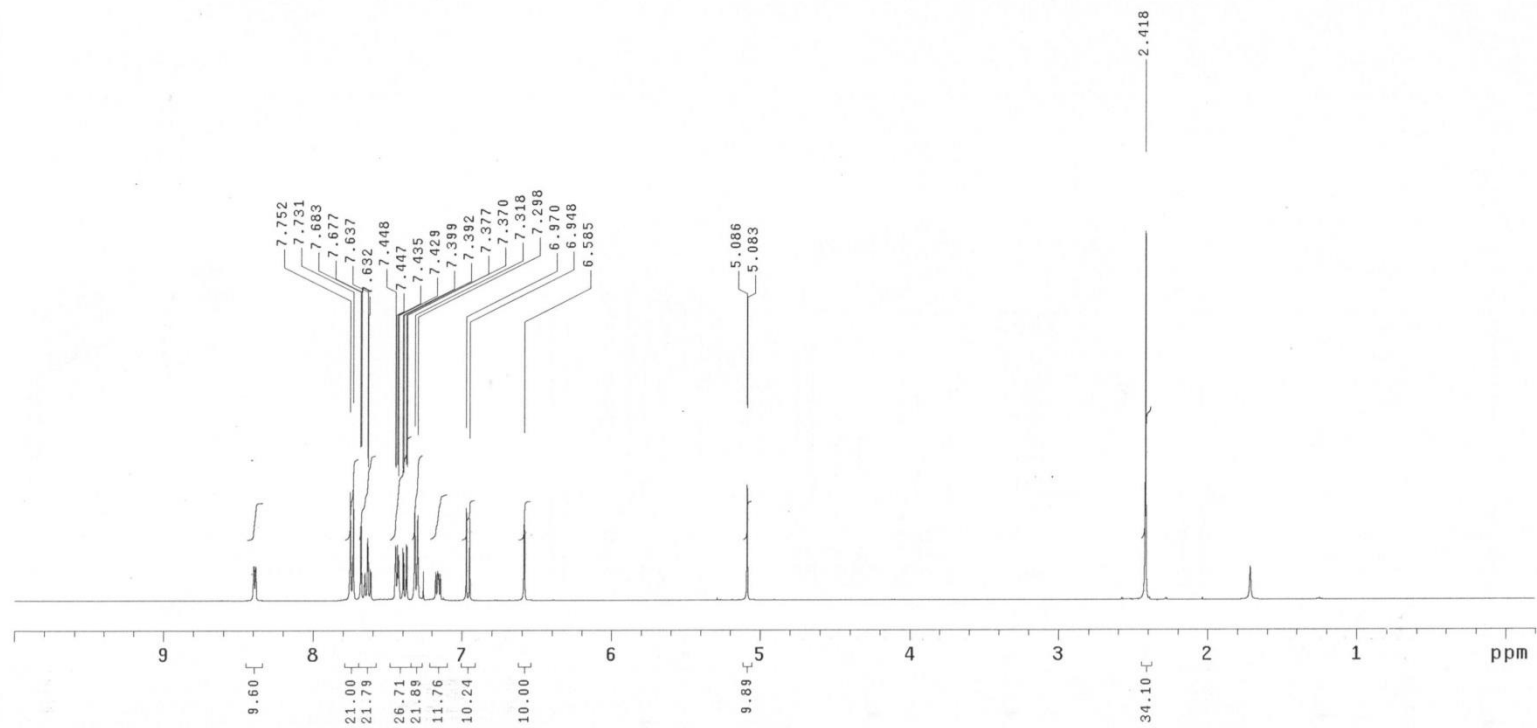
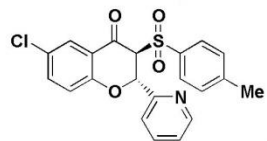
Pulse Sequence: s2pul  
UNITYplus-400 "unity400"  
Date: Jun 10 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 832 repetitions



# Compound 2m (<sup>1</sup>H-NMR spectral data)

KT4C1PY

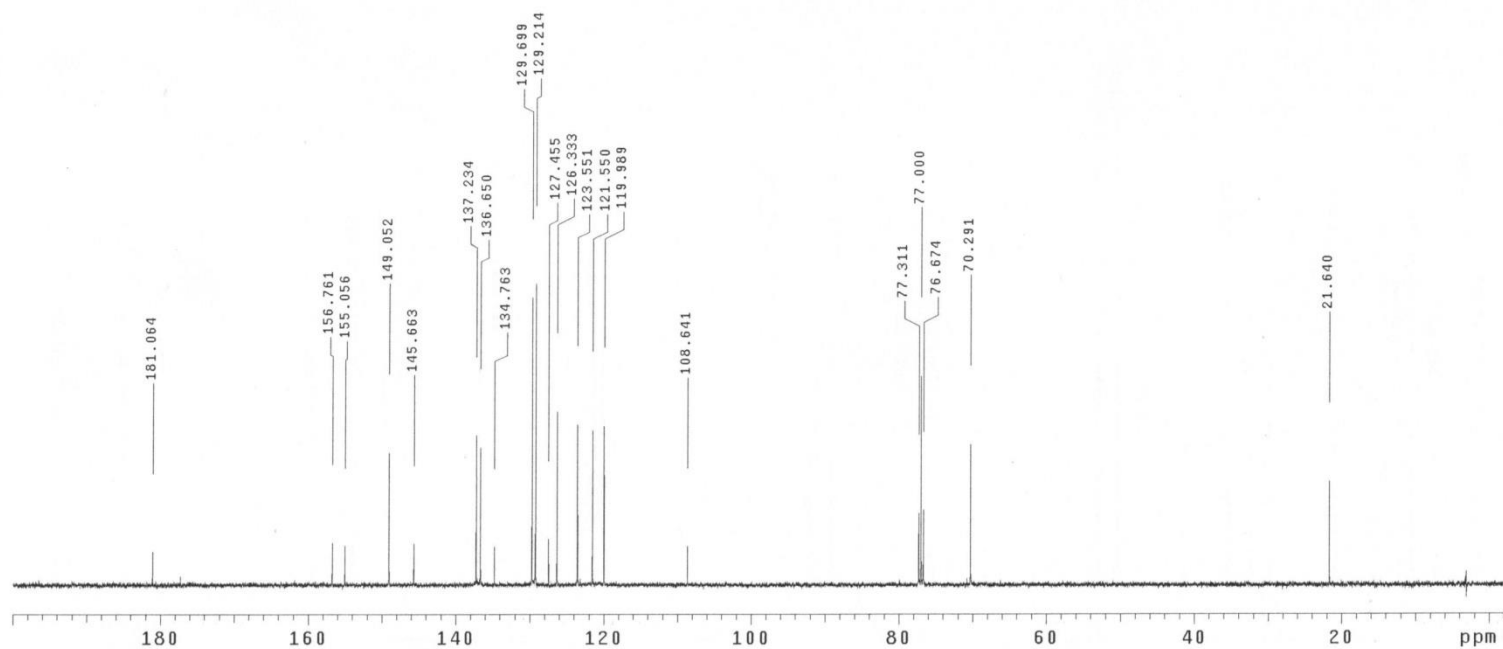
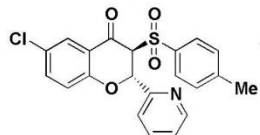
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jun 11 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2m (<sup>13</sup>C-NMR spectral data)

KT4C1PY

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jun 11 2019  
Solvent: CDCl3  
Ambient temperature  
Total 1008 repetitions



# Compound 2n (<sup>1</sup>H-NMR spectral data)

HA0MePY

Pulse Sequence: s2pu1

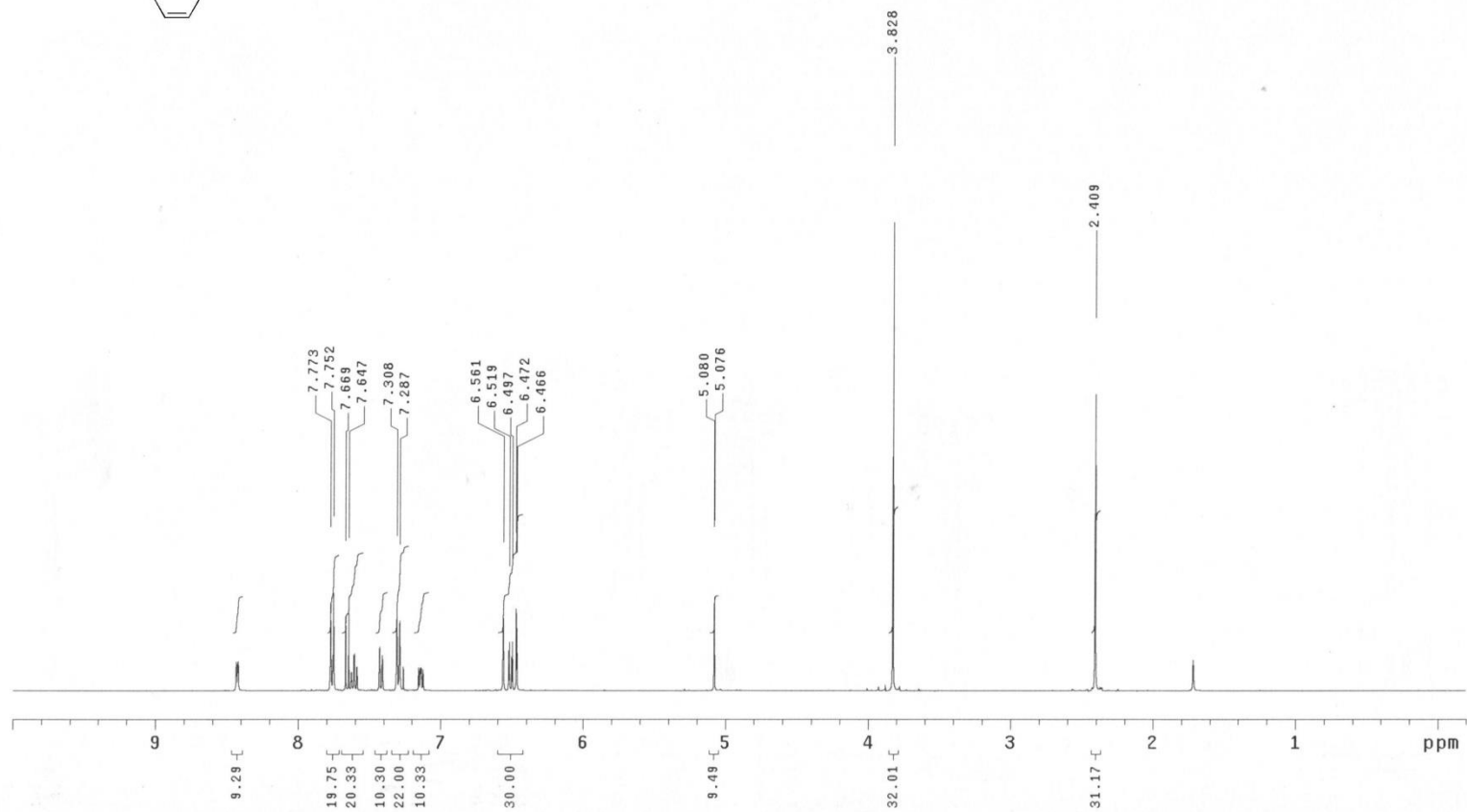
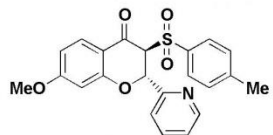
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Date: Mar 13 2019

Solvent: CDC13

Ambient temperature

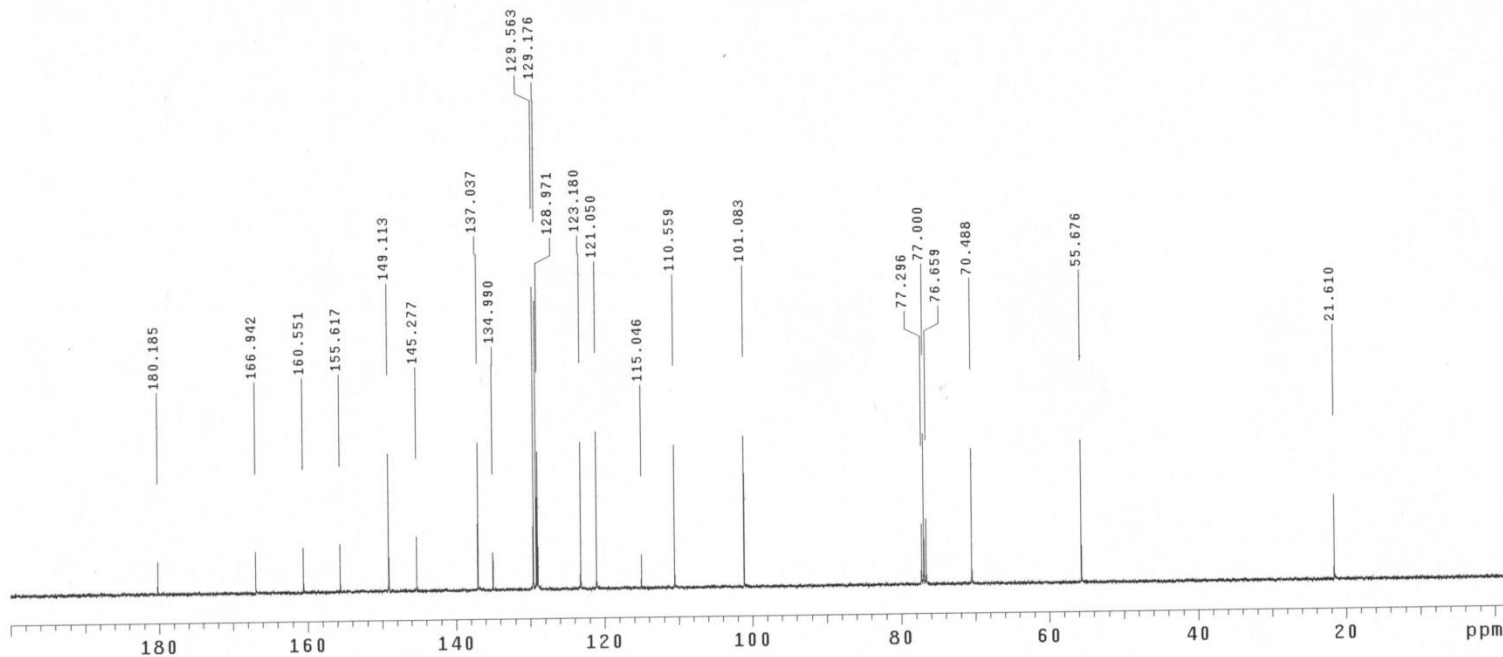
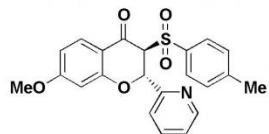
Total 32 repetitions



# Compound 2n (<sup>13</sup>C-NMR spectral data)

HA0MePY

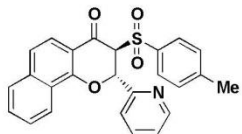
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Solvent: CDCl3  
Ambient temperature  
Total 1408 repetitions



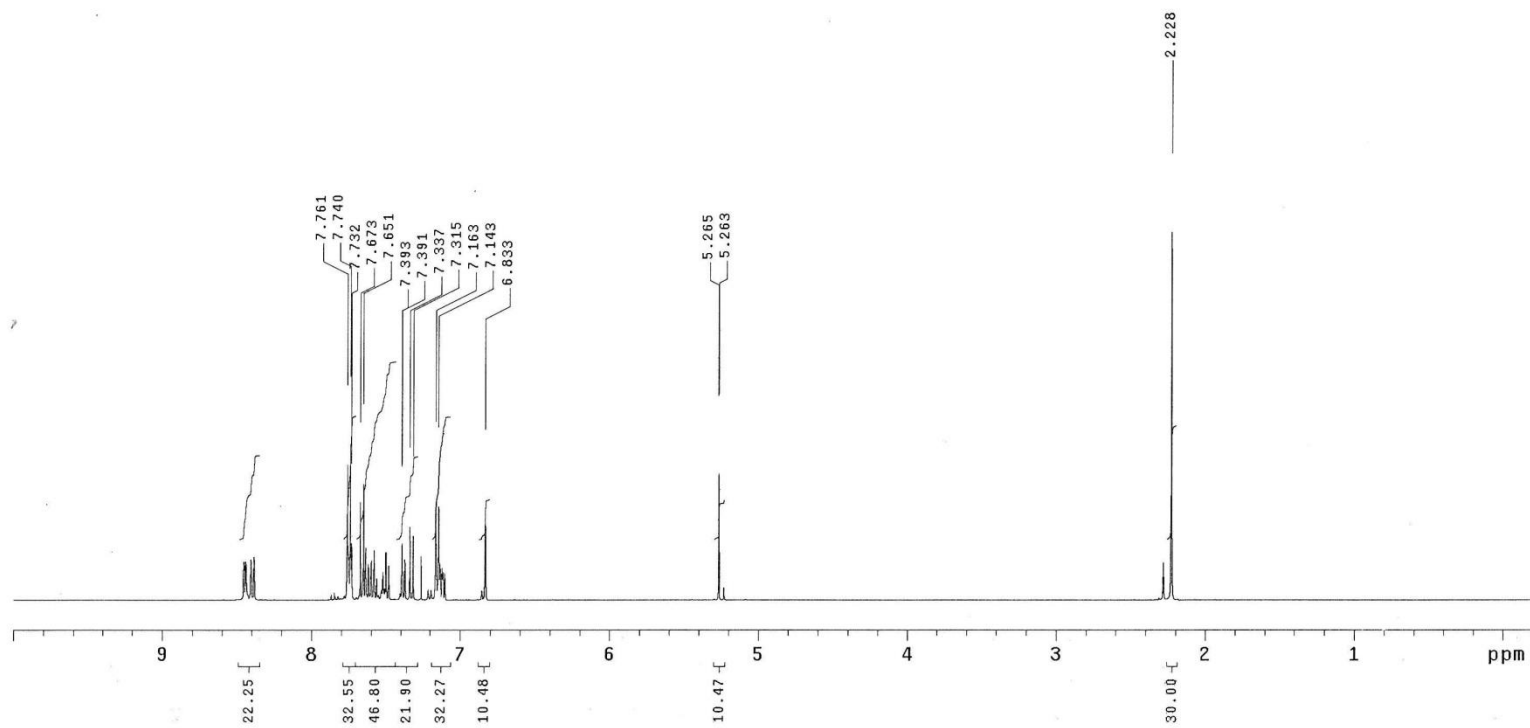
# Compound 2o (<sup>1</sup>H-NMR spectral data)

KT1NAPY

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Date: Aug 5 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



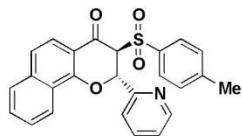
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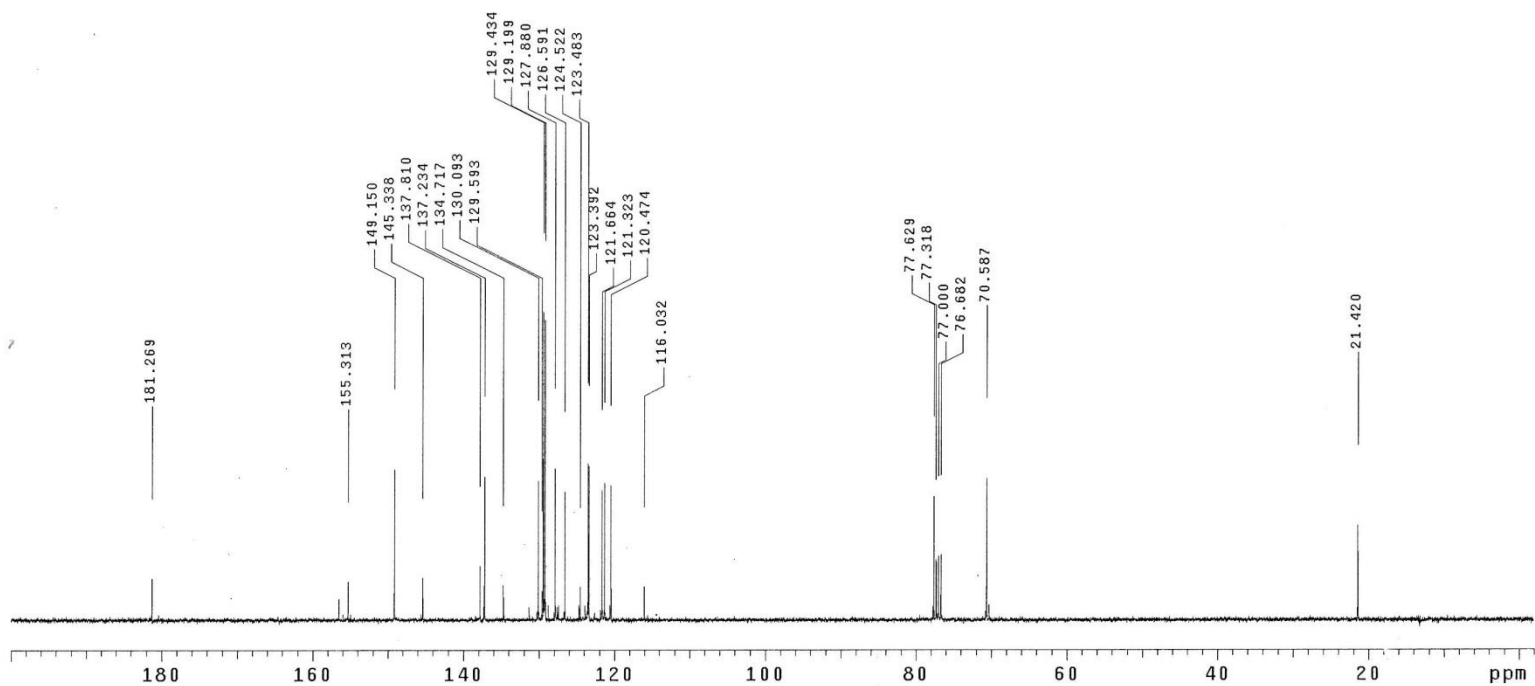
# Compound 2o (<sup>13</sup>C-NMR spectral data)

KT1NAPY

Pulse Sequence: s2pu1  
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Date: Aug 5 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 1296 repetitions



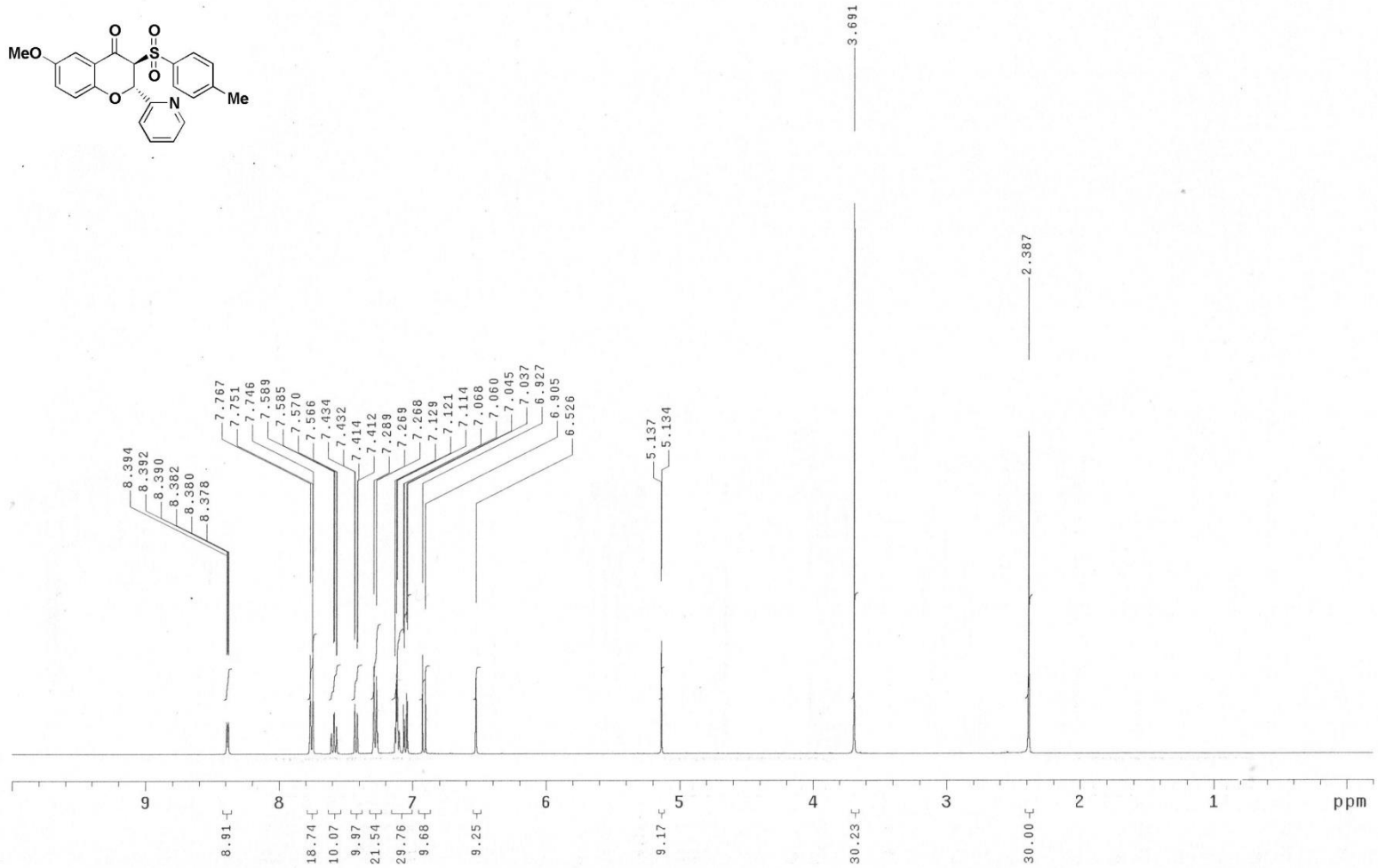
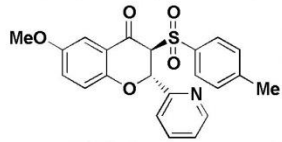
trans : cis > 10 : 1



# Compound 2p (<sup>1</sup>H-NMR spectral data)

KT40MePy

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 4 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions

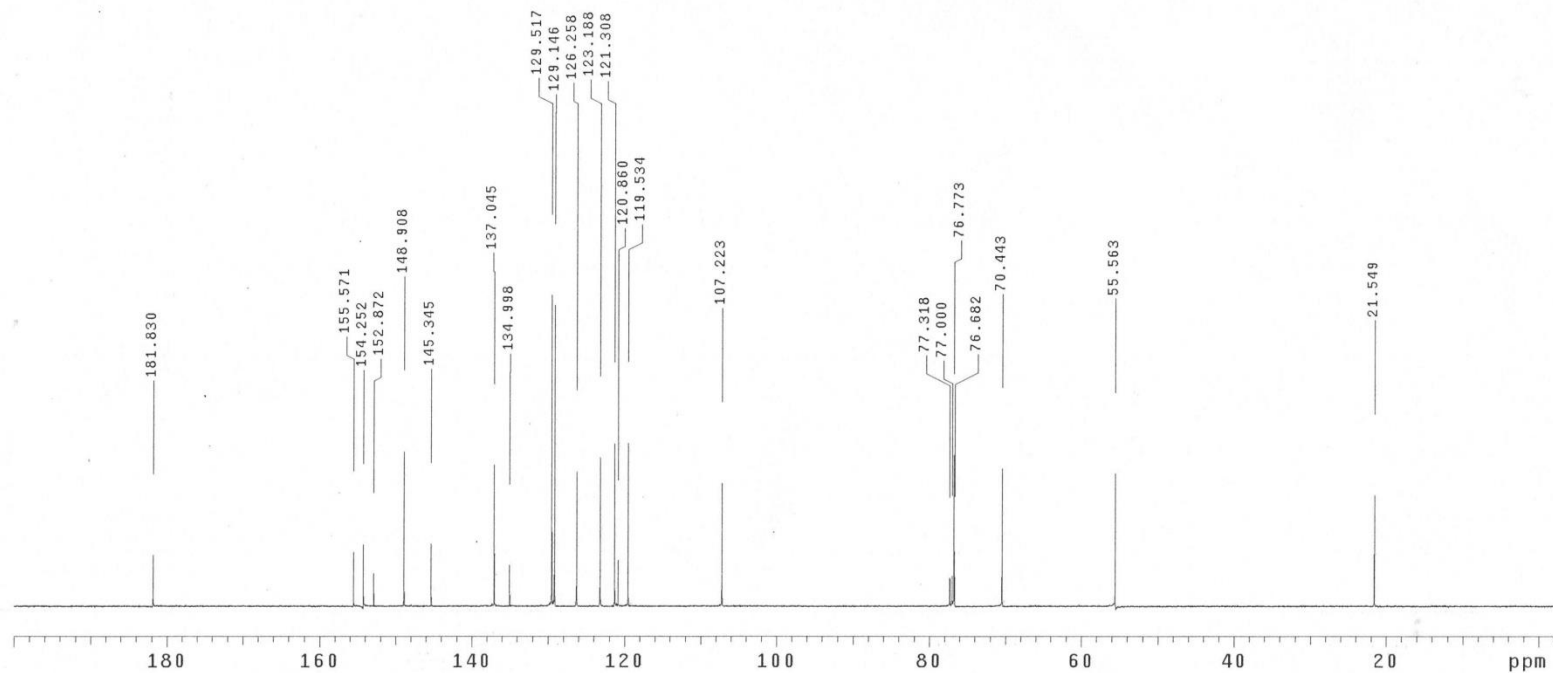
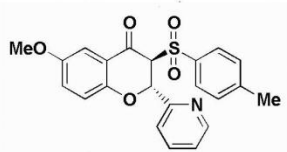




# Compound 2p (<sup>13</sup>C-NMR spectral data)

KT40MePy

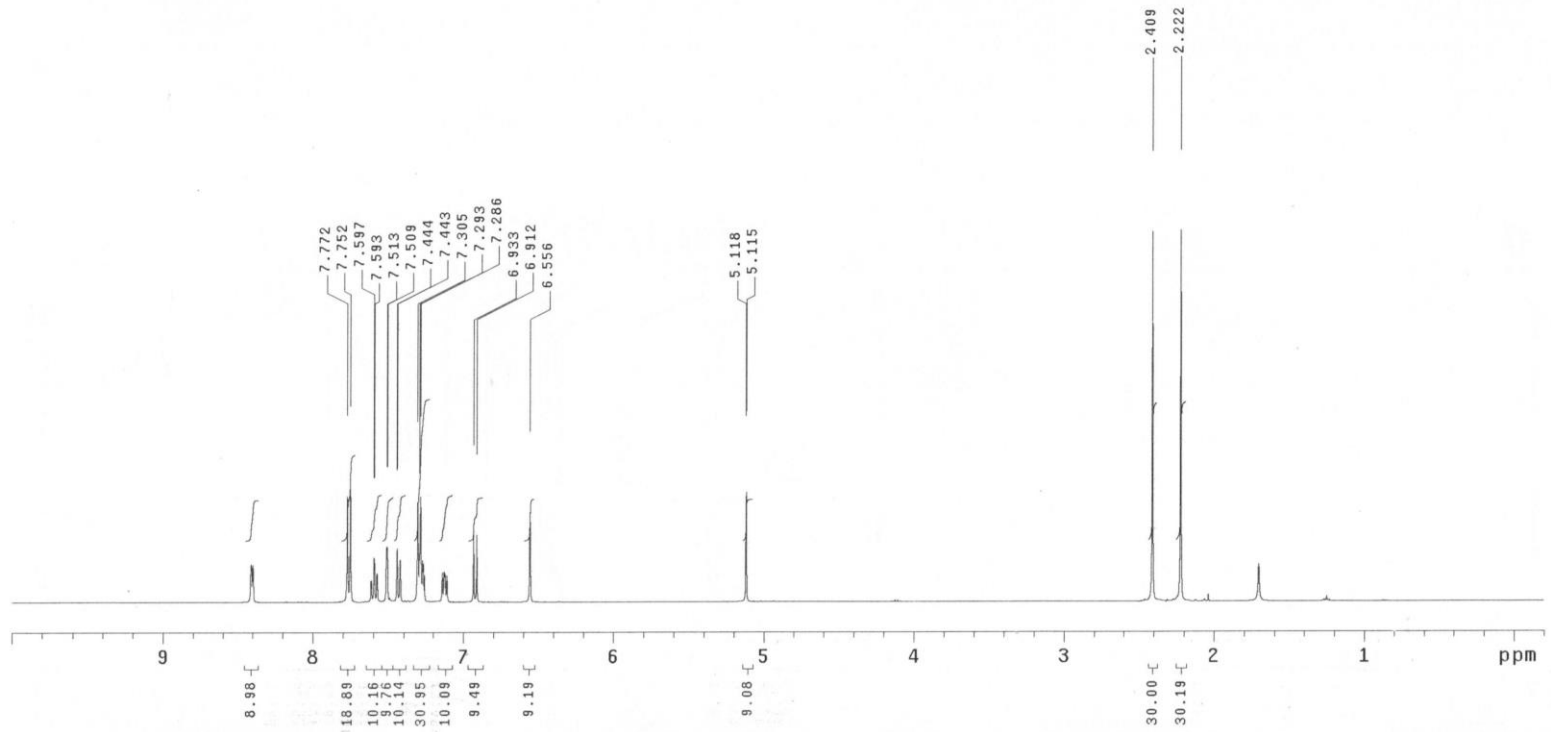
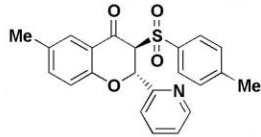
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 4 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2752 repetitions



# Compound 2q (<sup>1</sup>H-NMR spectral data)

KT4MePY

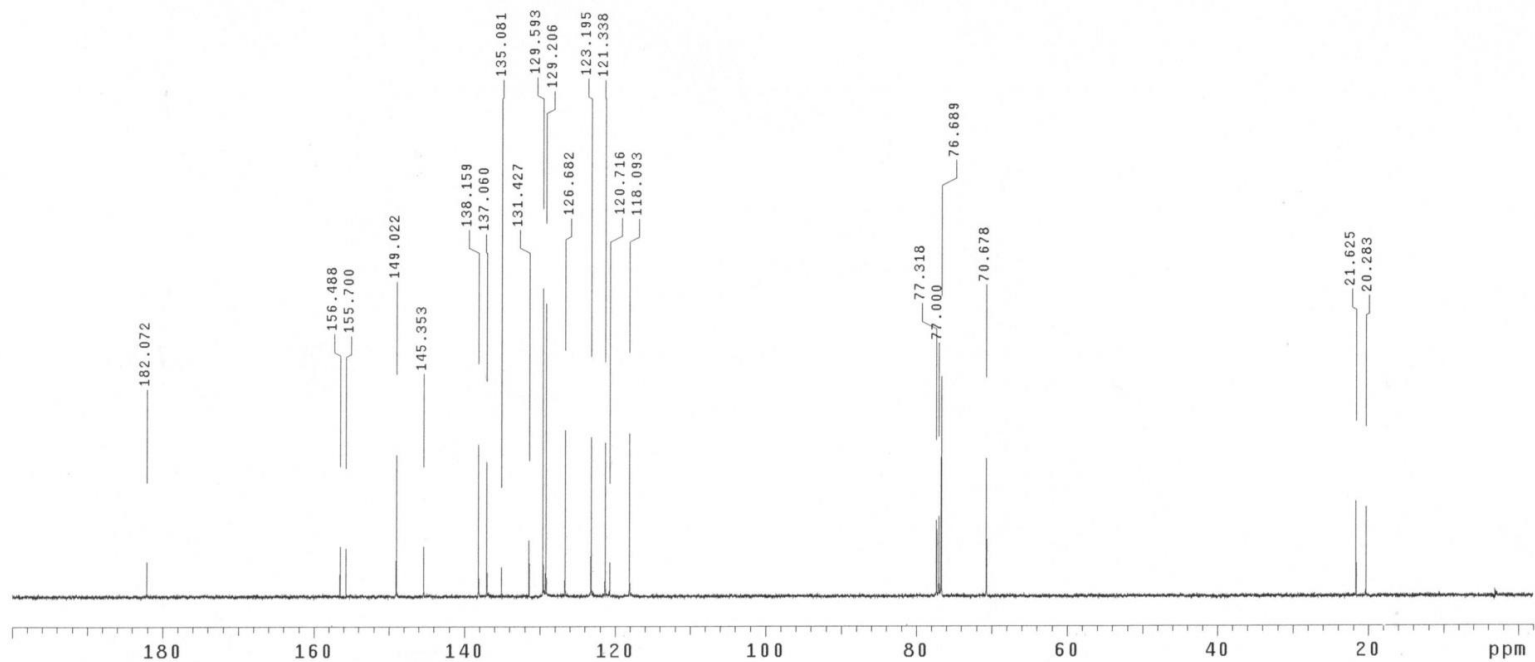
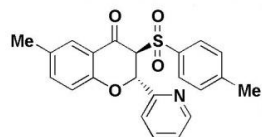
Pulse Sequence: s2pul  
UNITYplus-400 "unity400"  
Date: Jun 14 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2q (<sup>13</sup>C-NMR spectral data)

KT4MePY

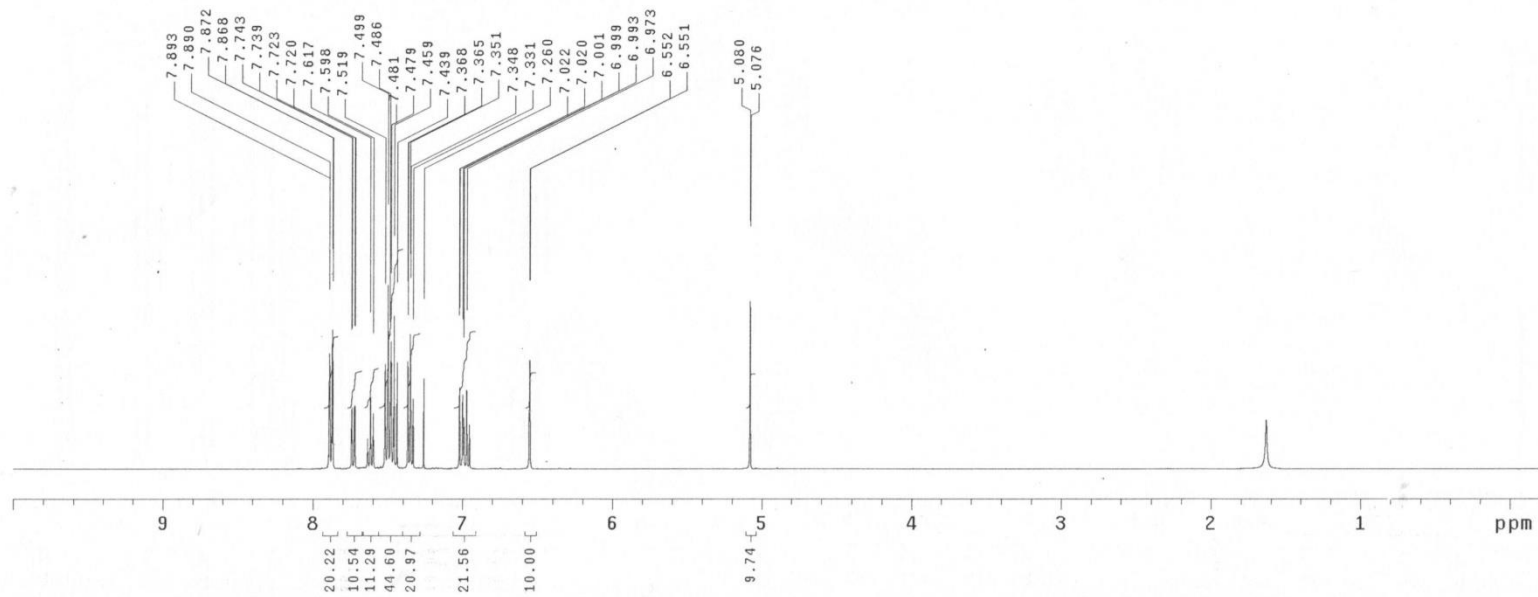
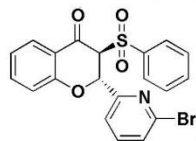
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jun 14 2019  
Solvent: CDC13  
Ambient temperature  
Total 1440 repetitions



# Compound 2r (<sup>1</sup>H-NMR spectral data)

KTPhB5PB

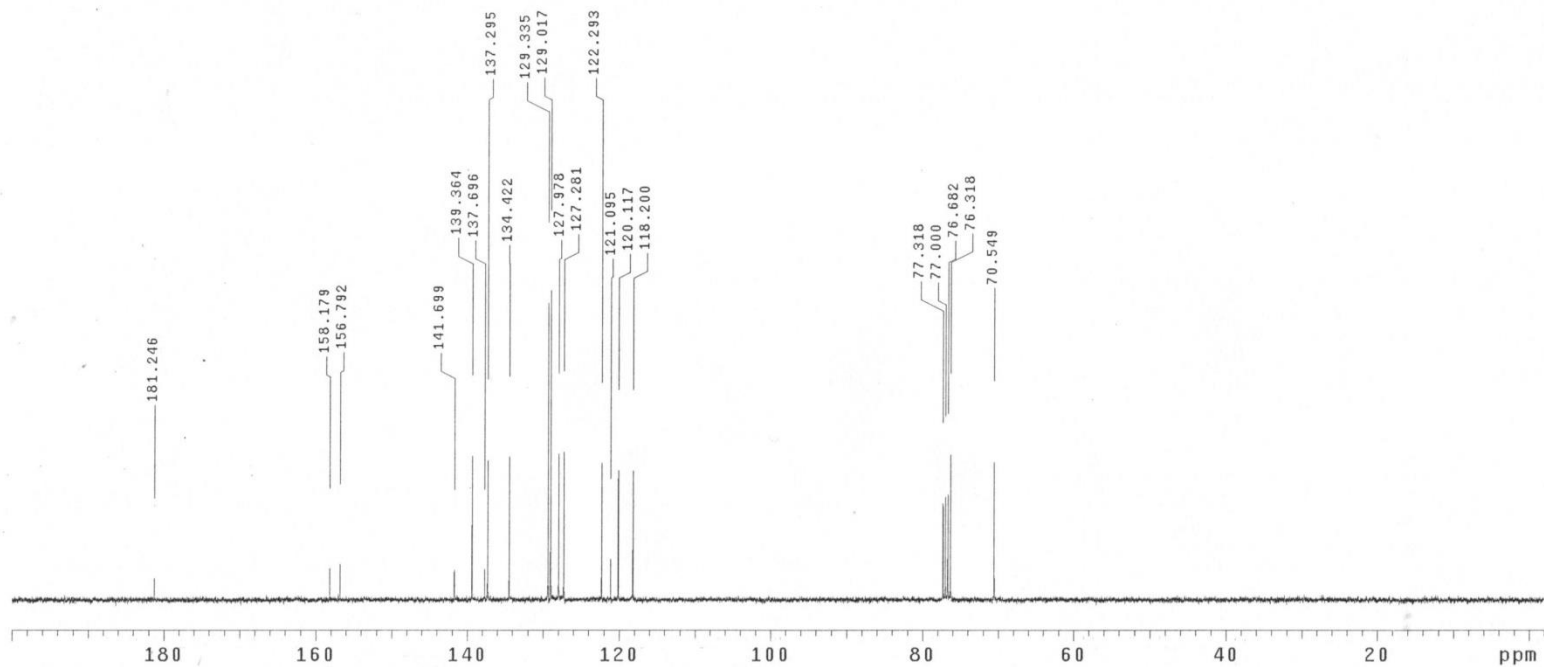
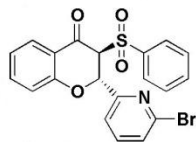
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 19 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2r (<sup>13</sup>C-NMR spectral data)

KTPbB5PB

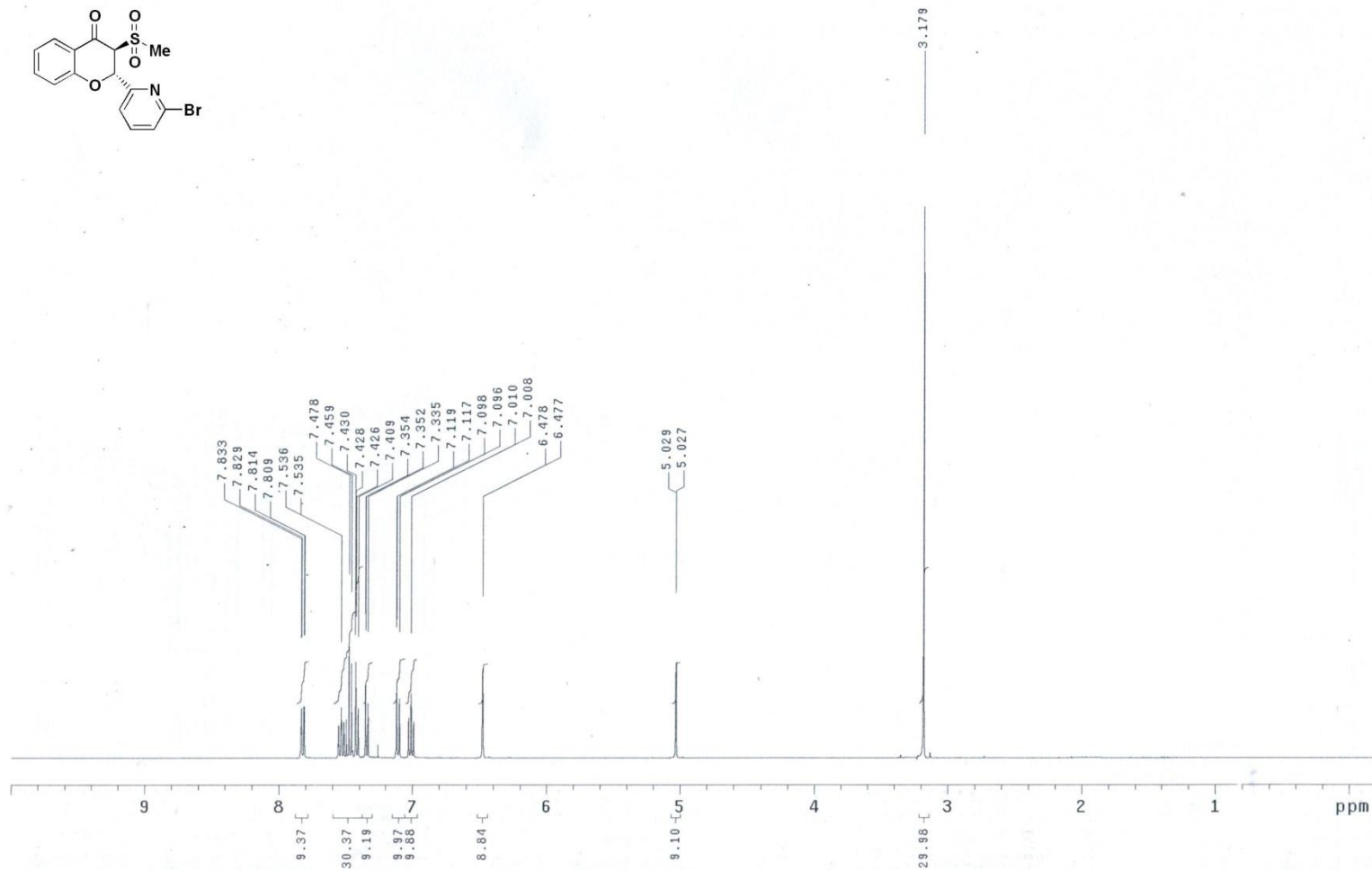
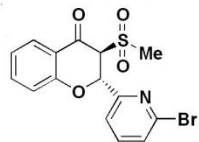
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 19 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2272 repetitions



# Compound 2s (<sup>1</sup>H-NMR spectral data)

KTPHMsPB

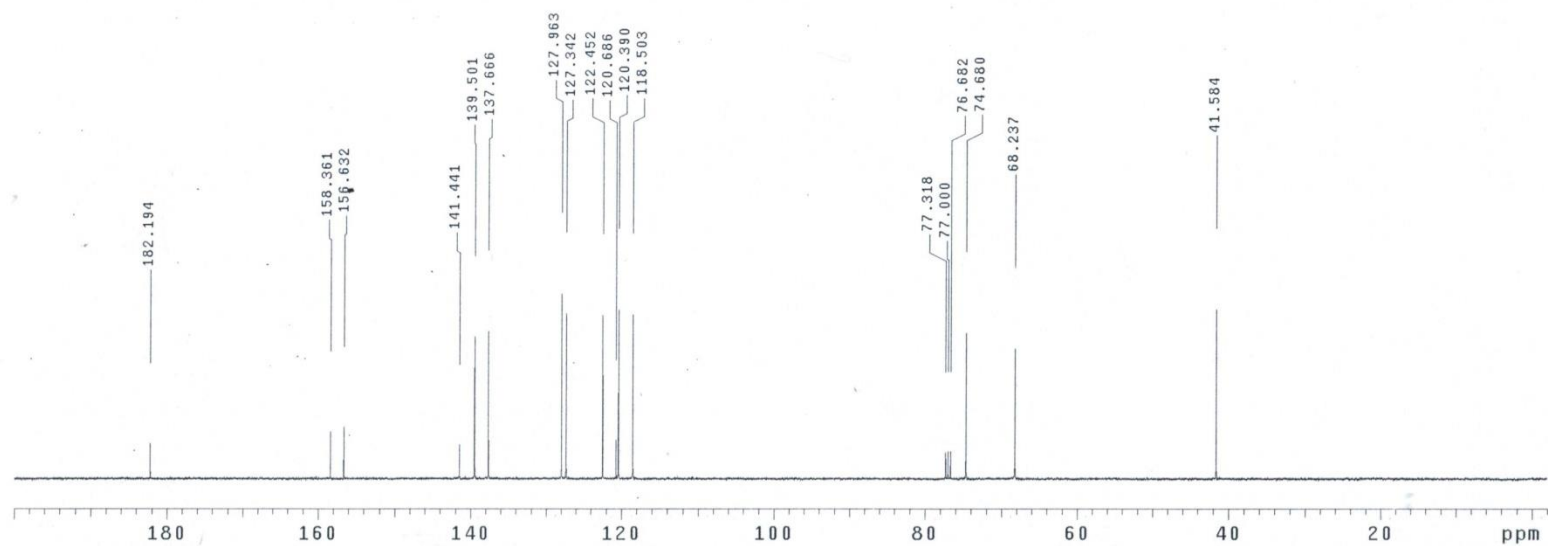
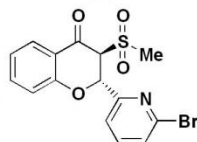
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 26 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2s (<sup>13</sup>C-NMR spectral data)

KTPHMsPB

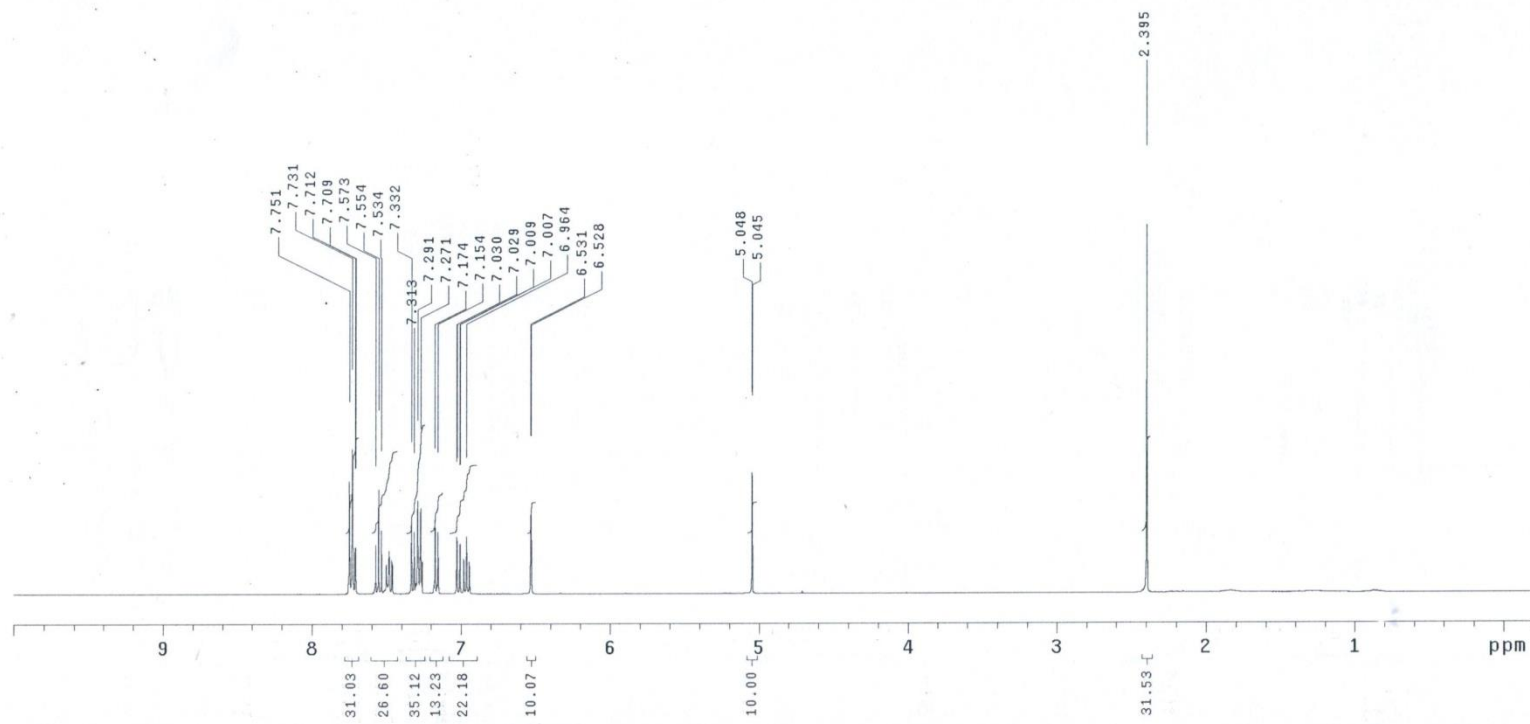
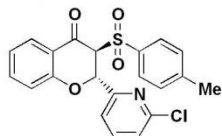
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 26 2019  
Solvent: CDC13  
Ambient temperature  
Total 320 repetitions



# Compound 2t (<sup>1</sup>H-NMR spectral data)

KTPPhC

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 27 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions





# Compound 2t (<sup>13</sup>C-NMR spectral data)

KTPPhC

Pulse Sequence: s2pu1

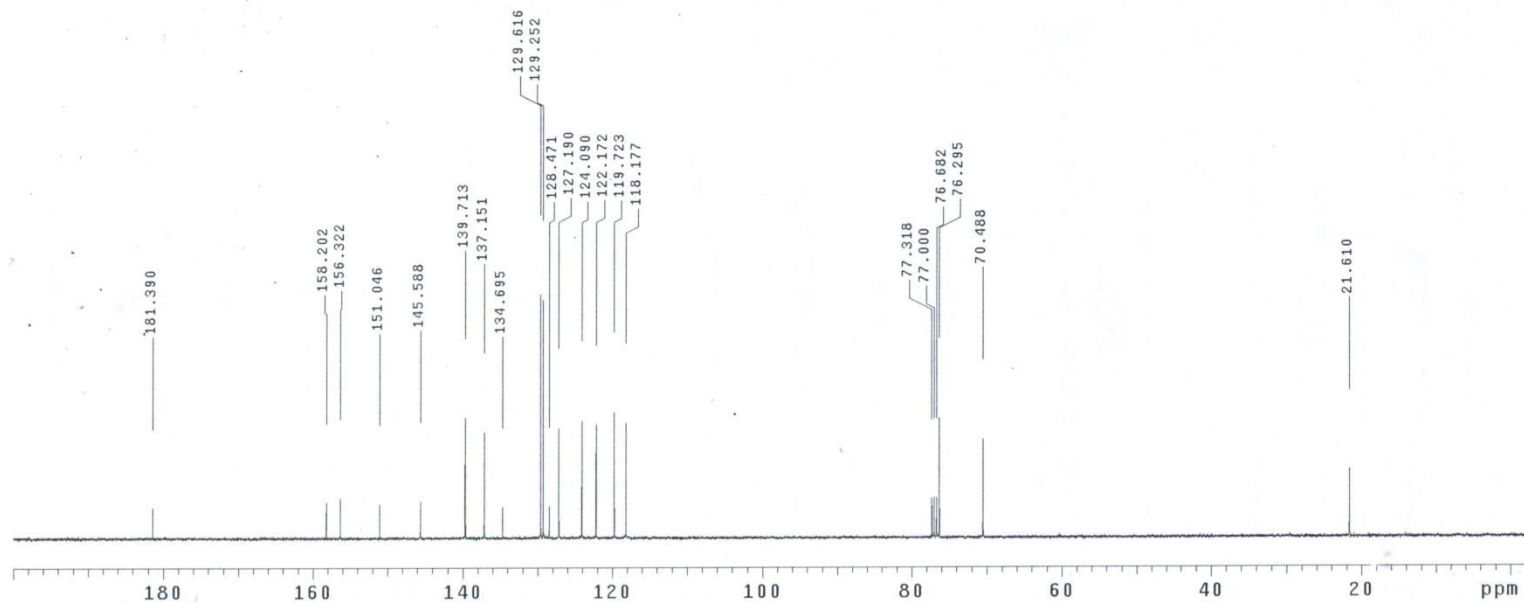
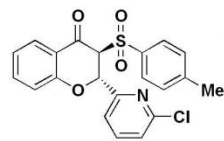
UNITYplus-400 "unity400"

Date: Sep 27 2019

Solvent: CDCl<sub>3</sub>

Ambient temperature

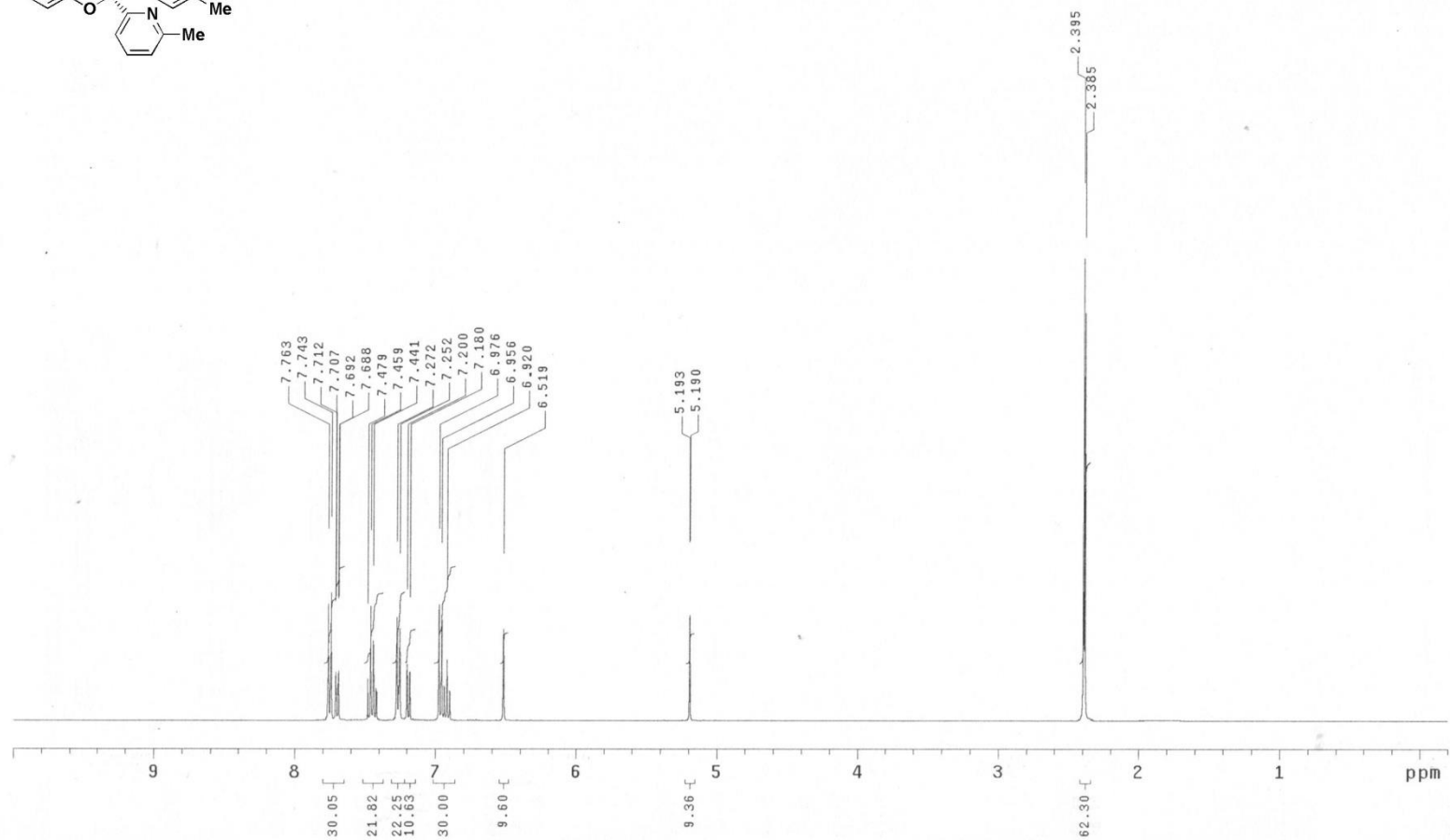
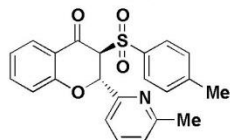
Total 640 repetitions



# Compound 2u (<sup>1</sup>H-NMR spectral data)

KTPhPM

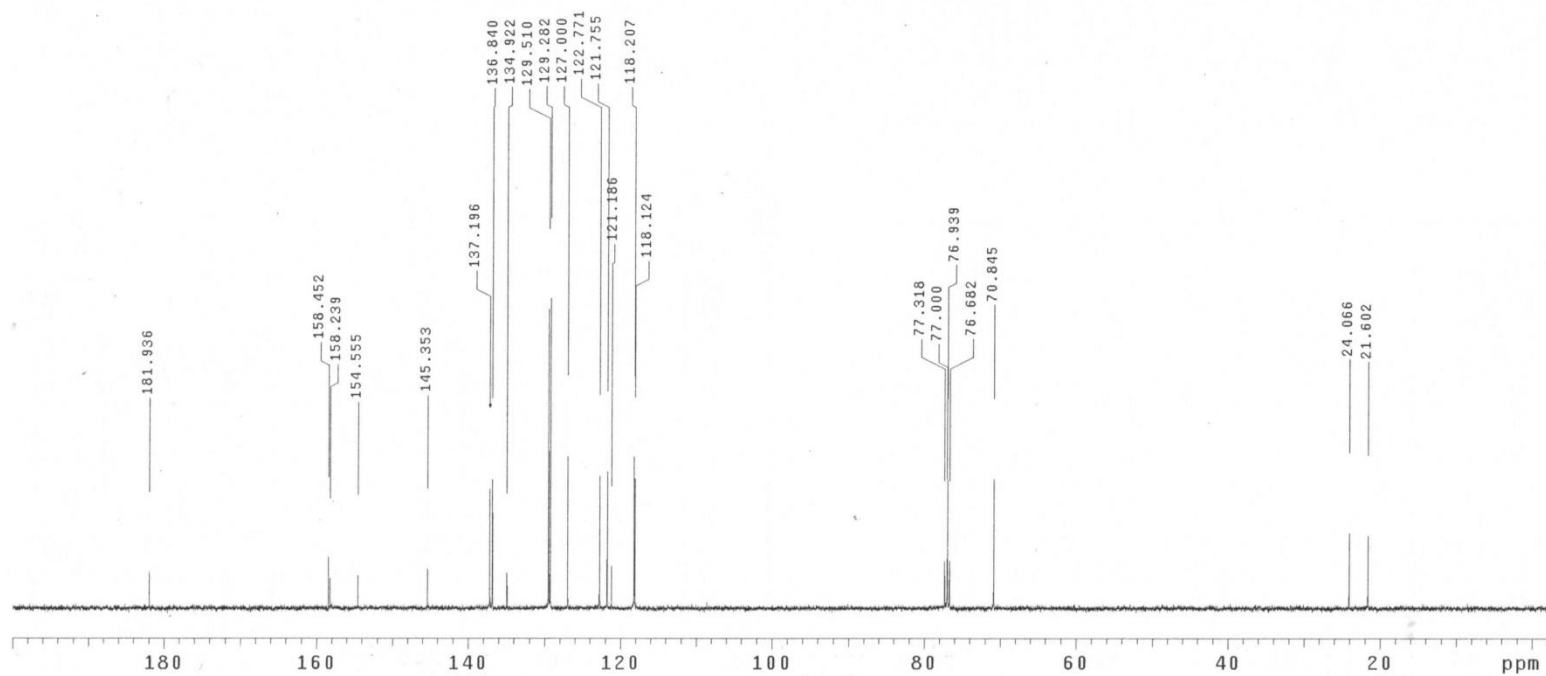
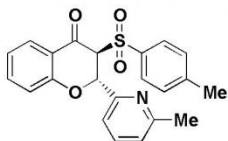
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 7 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions



# Compound 2u (<sup>13</sup>C-NMR spectral data)

KTPbPM

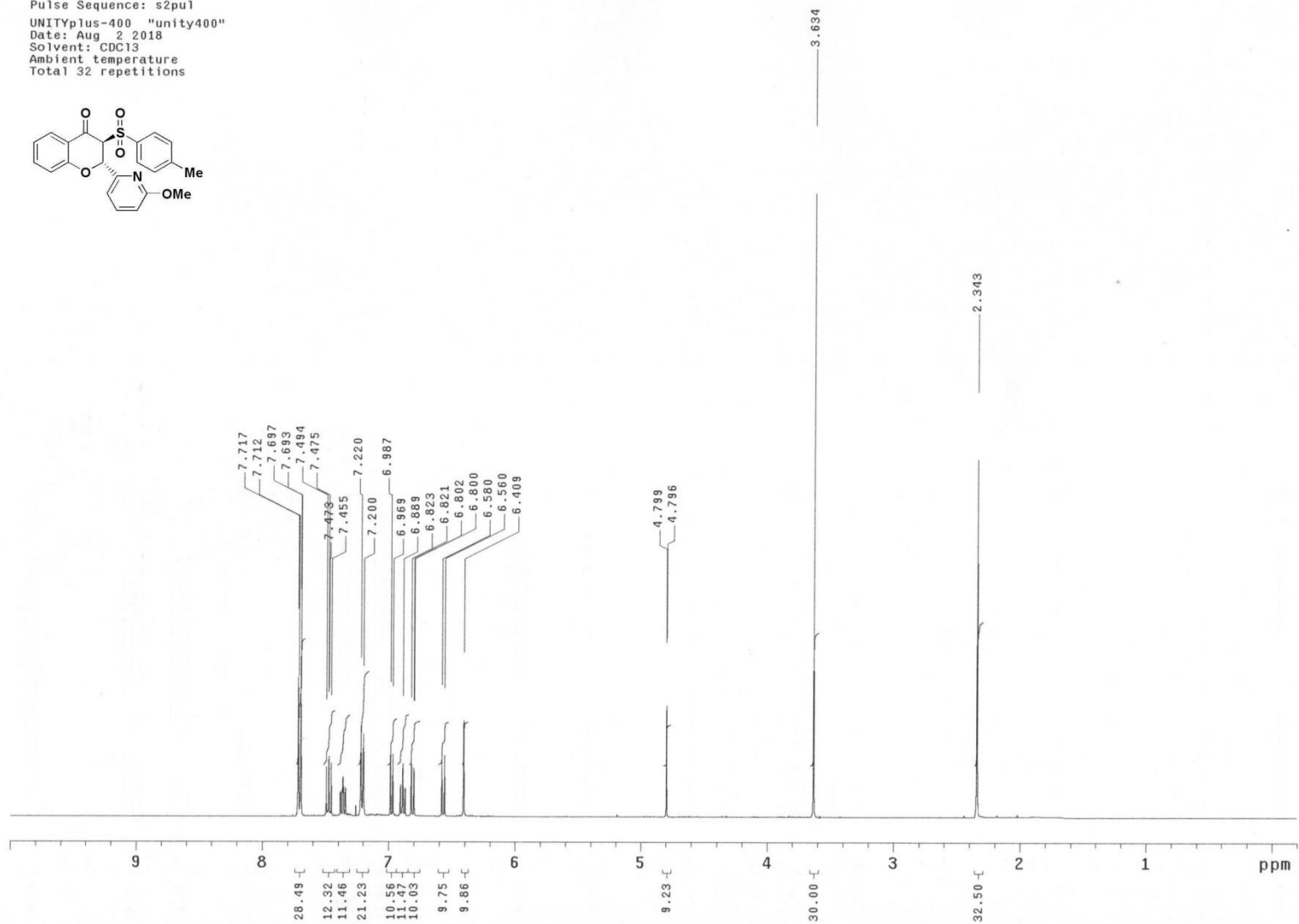
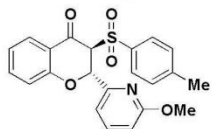
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 7 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 528 repetitions



# Compound 2v (<sup>1</sup>H-NMR spectral data)

HAOMePYBr

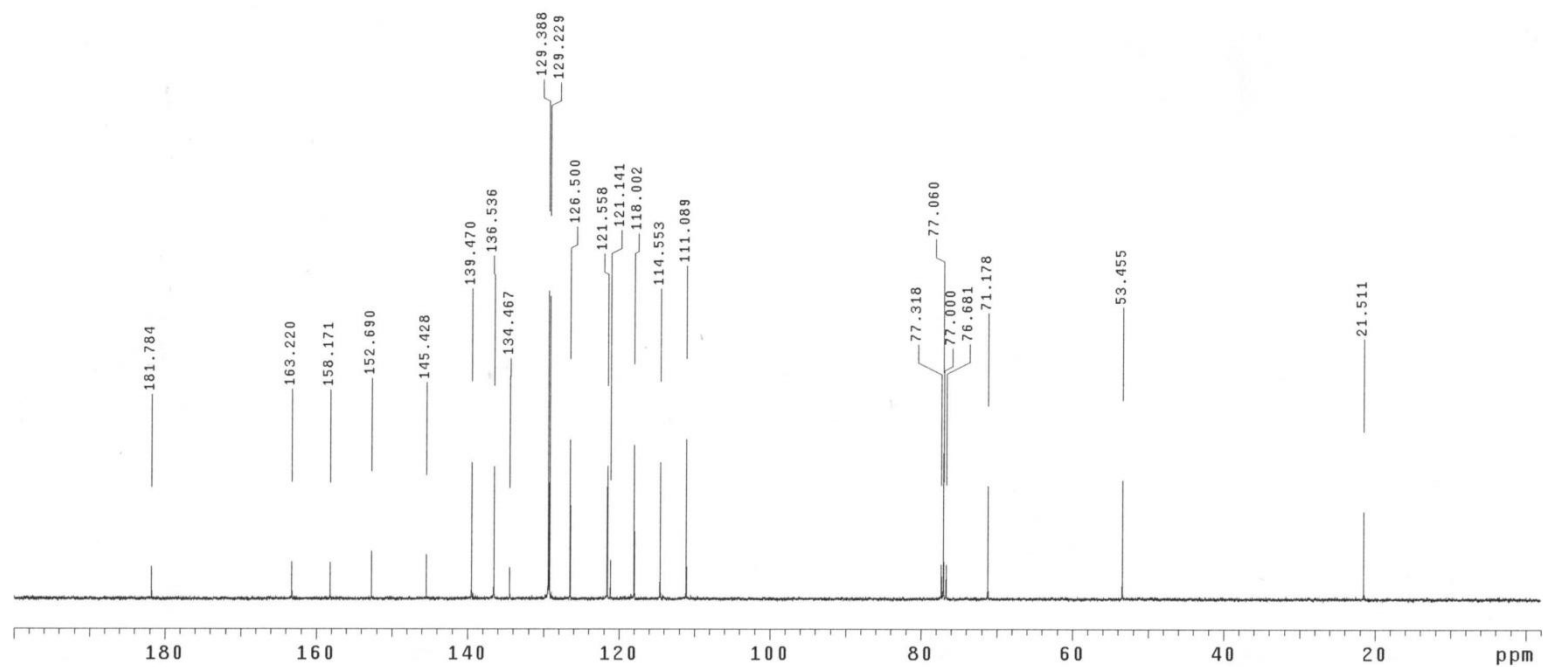
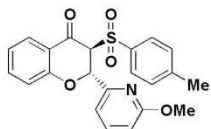
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 2 2018  
Solvent: CDC13  
Ambient temperature  
Total 32 repetitions



# Compound 2v (<sup>13</sup>C-NMR spectral data)

HAOMePYBr

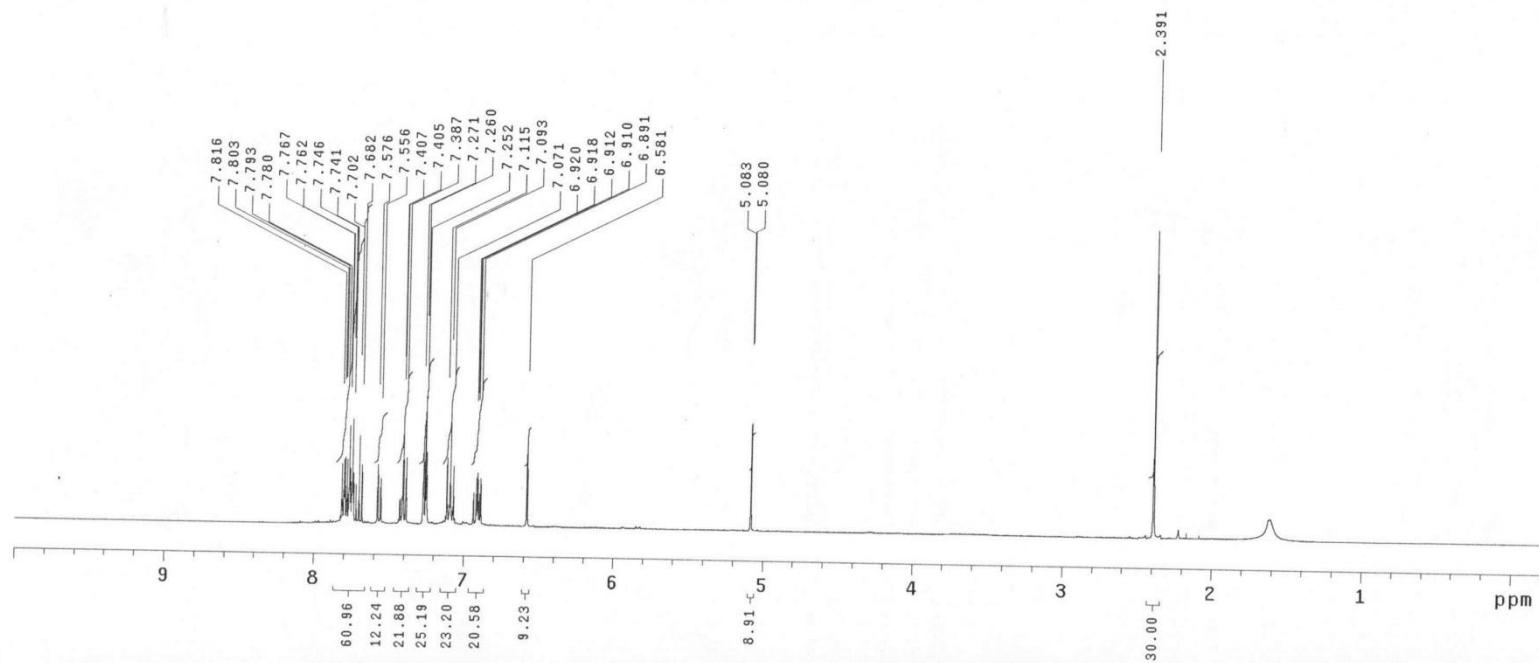
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 2 2018  
Solvent: CDCl3  
Ambient temperature  
Total 272 repetitions



# Compound 2w (<sup>1</sup>H-NMR spectral data)

HA1070420

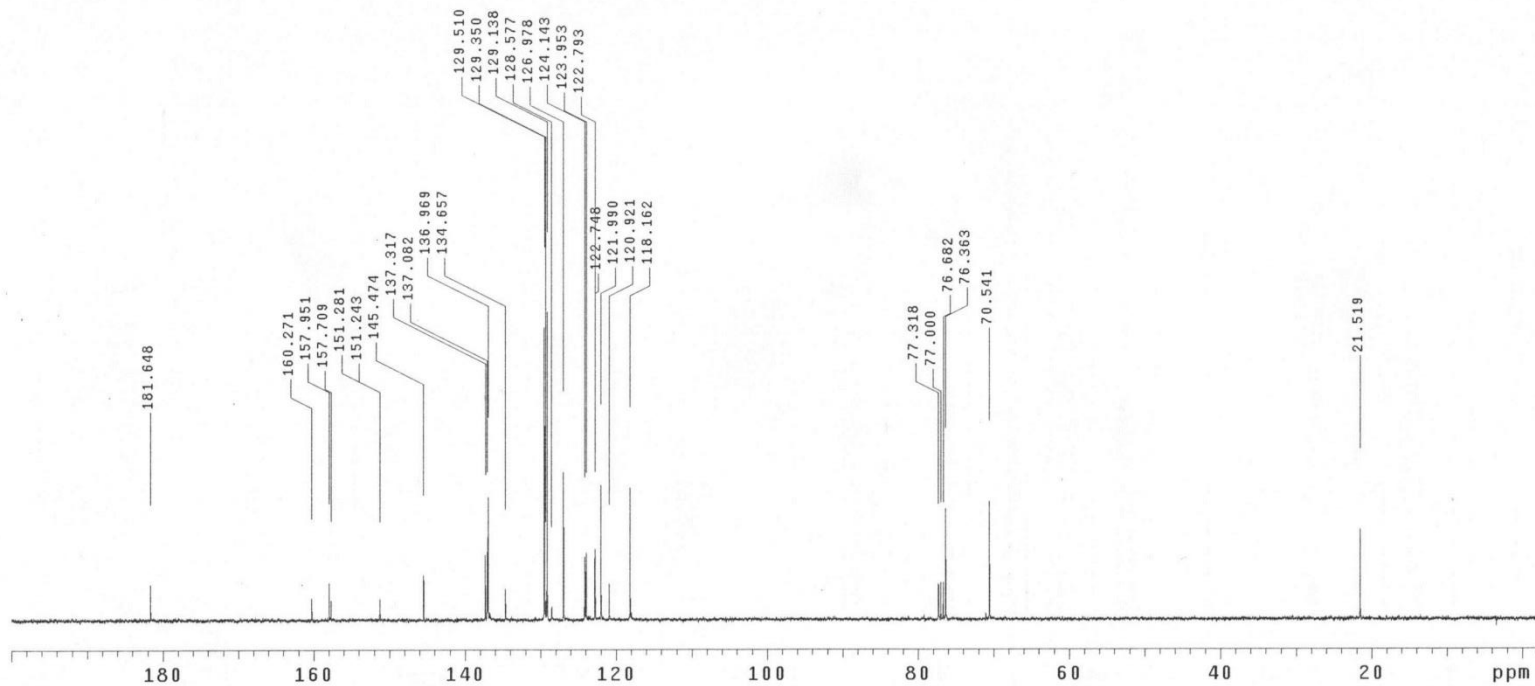
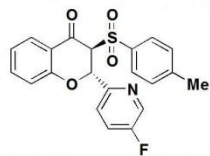
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 13 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 2w (<sup>13</sup>C-NMR spectral data)

HA1070419

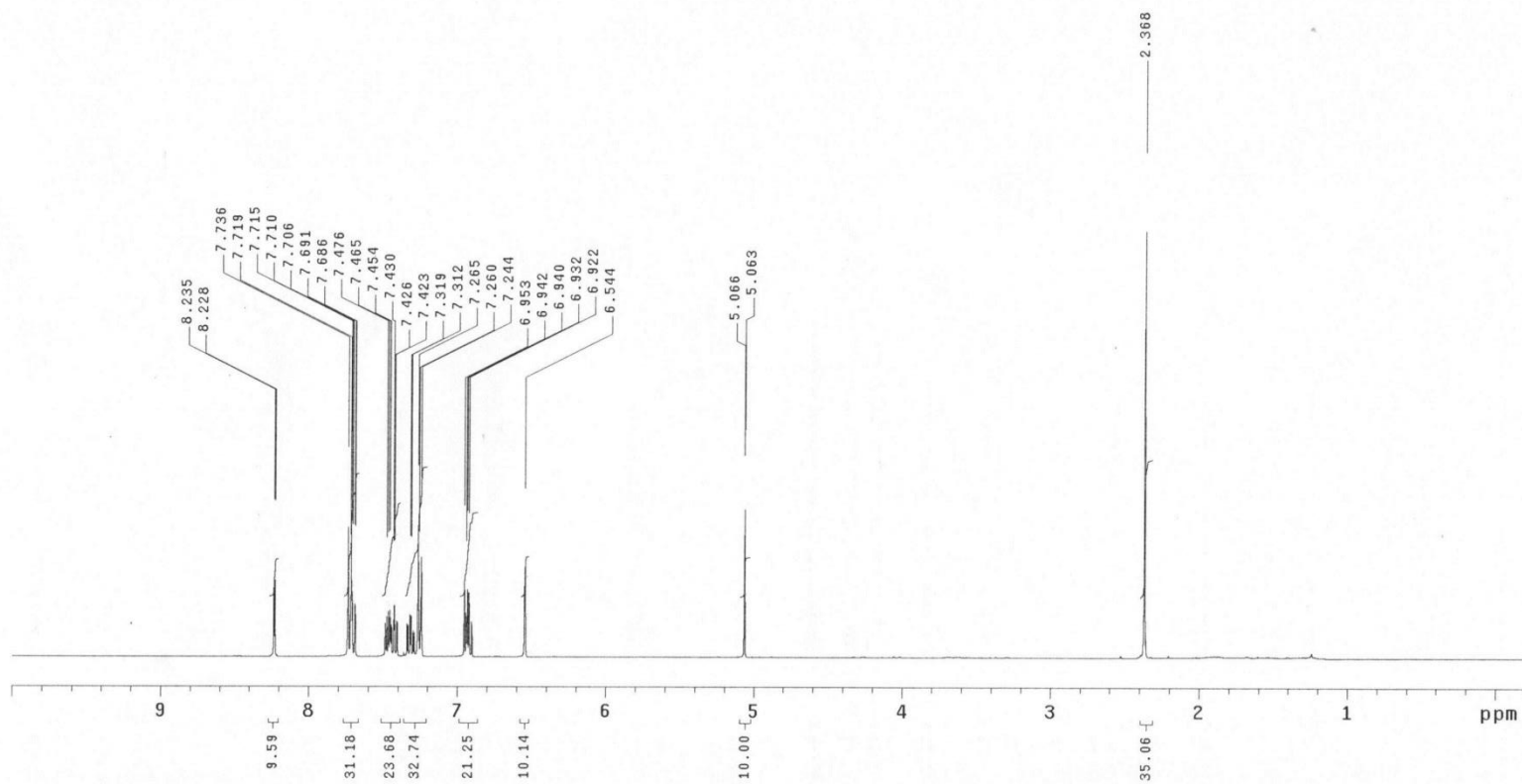
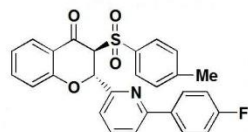
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 19 2018  
Solvent: CDC13  
Ambient temperature  
Total 192 repetitions



# Compound 2x (<sup>1</sup>H-NMR spectral data)

HA1070419

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 19 2018  
Solvent: CDC13  
Ambient temperature  
Total 32 repetitions

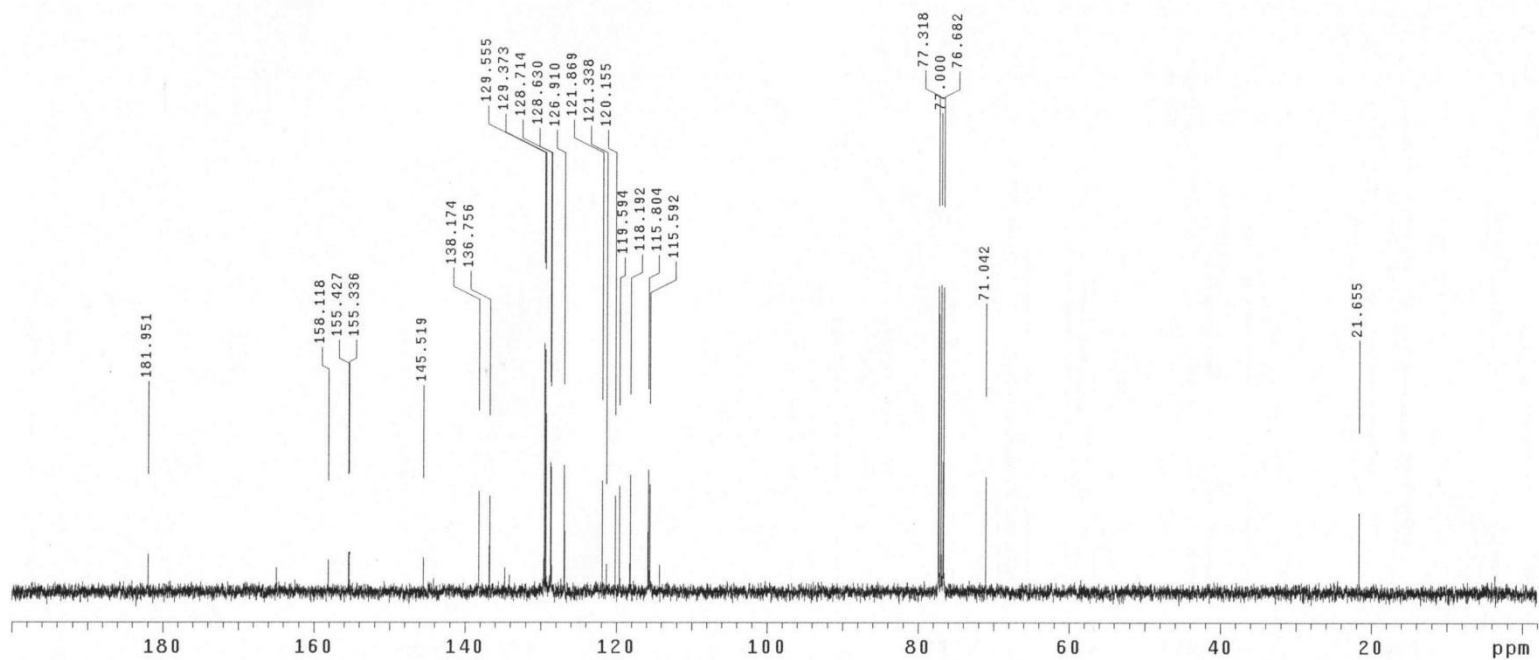
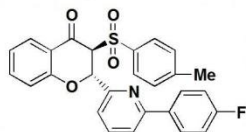




# Compound 2x (<sup>13</sup>C-NMR spectral data)

HA1070420

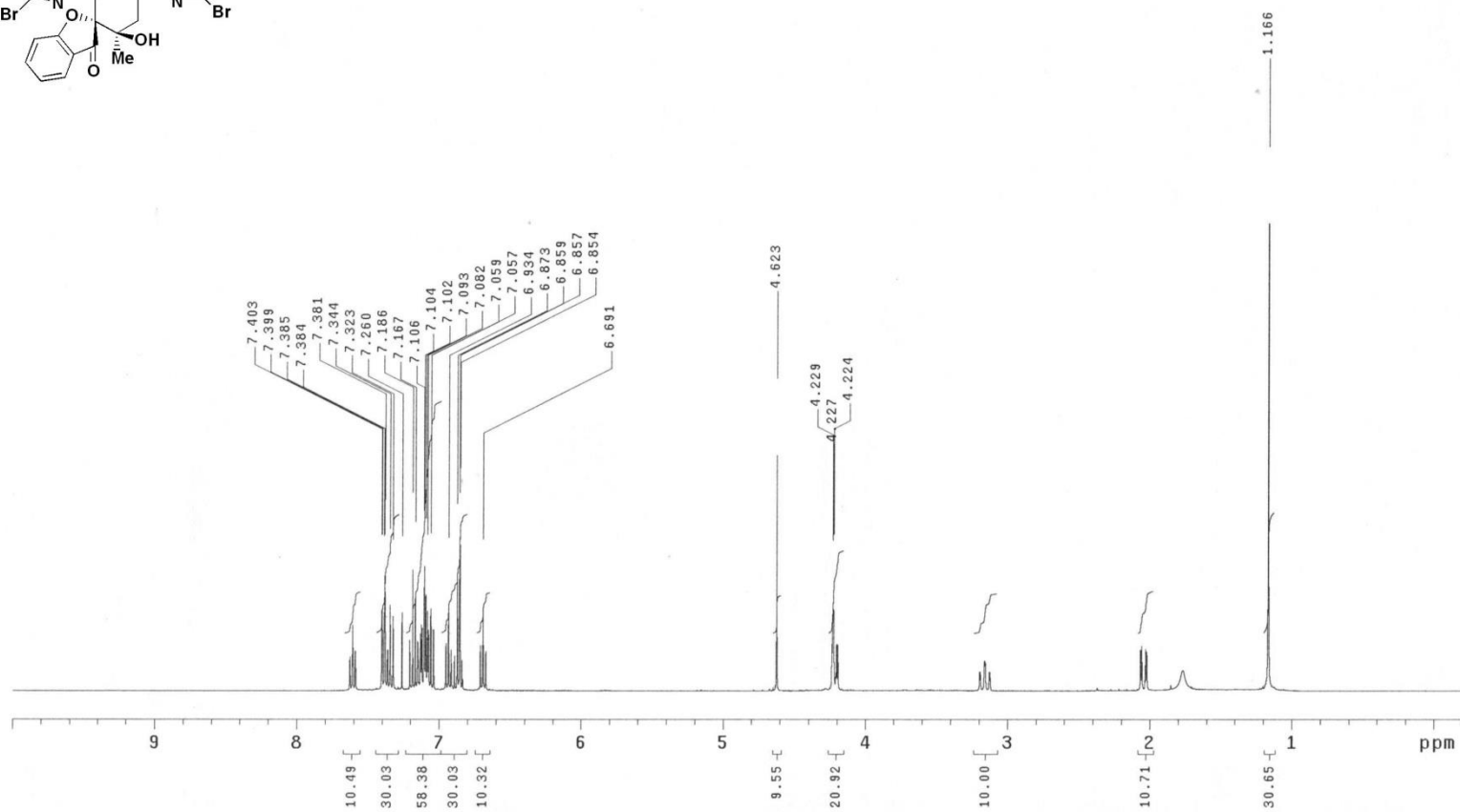
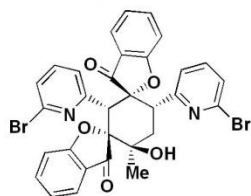
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Apr 19 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 1024 repetitions



# Compound 3a (<sup>1</sup>H-NMR spectral data)

HANBrBu

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Mar 20 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions





# Compound 3b (<sup>1</sup>H-NMR spectral data)

HAFPyBr

Pulse Sequence: s2pu1

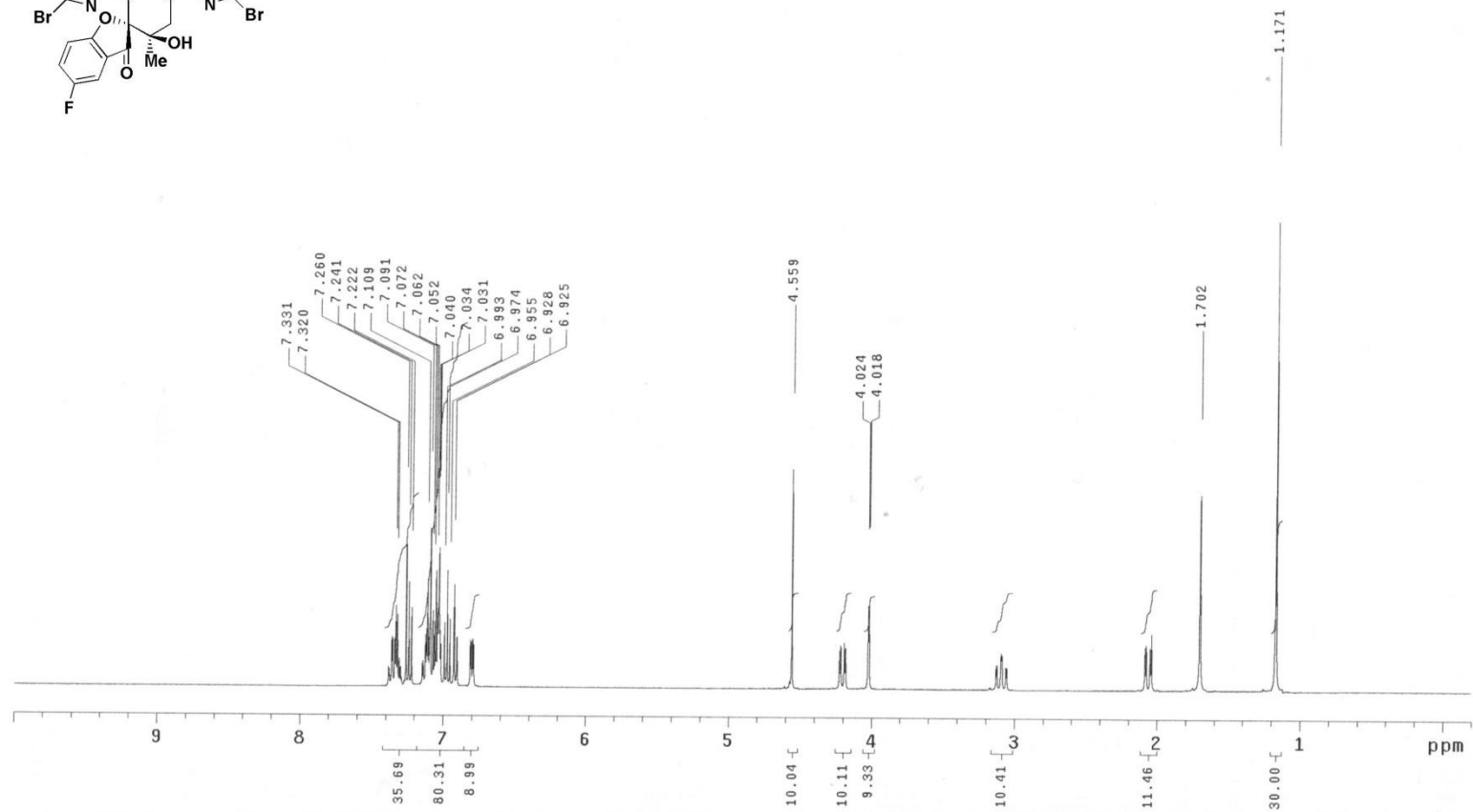
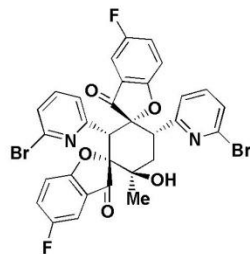
UNITYplus-400 "unity400"

Date: Jan 15 2019

Solvent: CDCl<sub>3</sub>

Ambient temperature

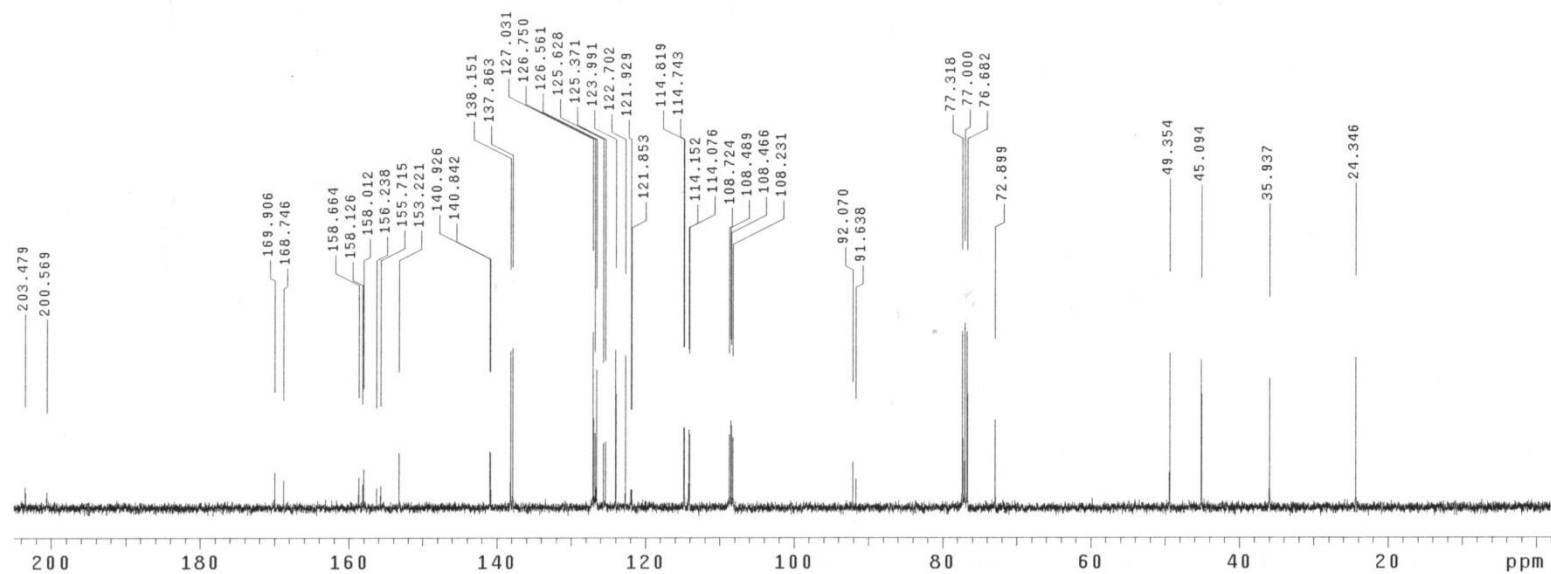
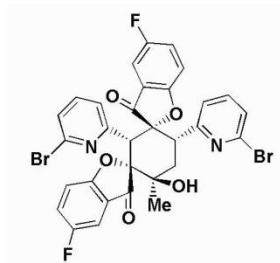
Total 32 repetitions



# Compound 3b (<sup>13</sup>C-NMR spectral data)

HAFPyBr

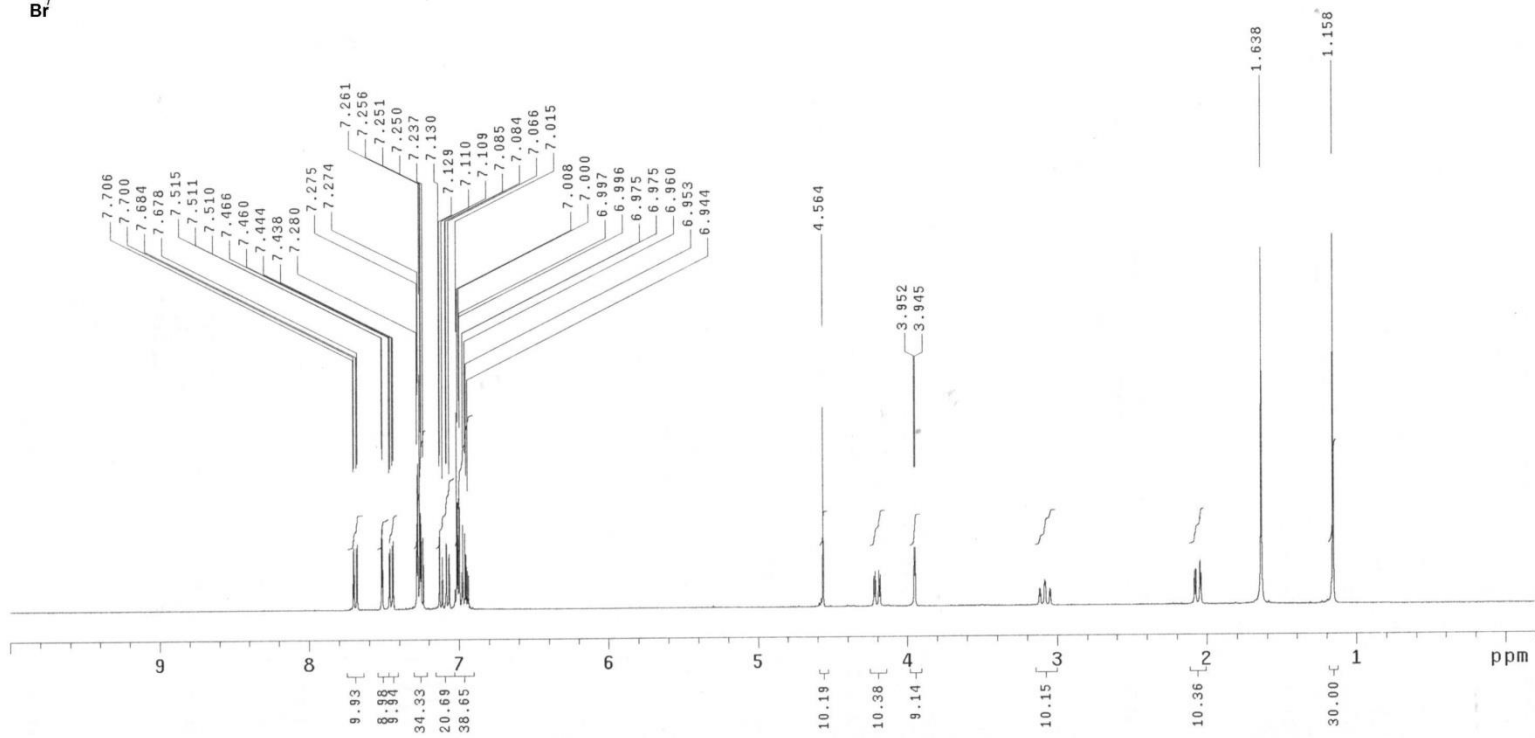
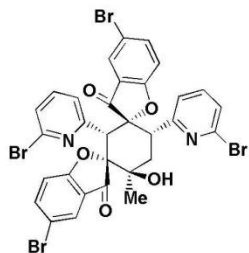
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 15 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2048 repetitions



# Compound 3c (<sup>1</sup>H-NMR spectral data)

HA4BrPB

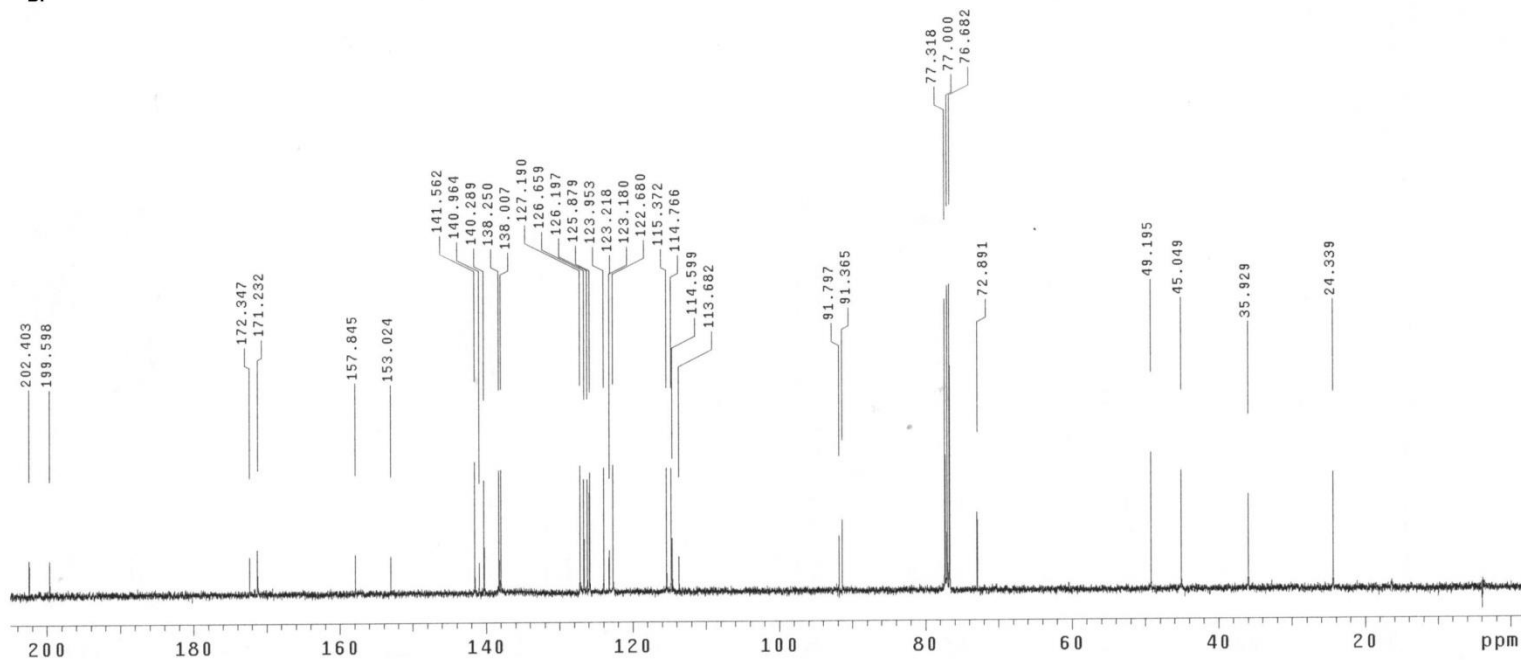
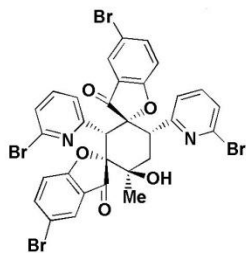
Pulse Sequence: s2pul  
UNITYplus-400 "unity400"  
Date: Jan 2 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 64 repetitions



# Compound 3c (<sup>13</sup>C-NMR spectral data)

HA4BrPB

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 2 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 8944 repetitions



# Compound 3d (<sup>1</sup>H-NMR spectral data)

HA1epyBr

Pulse Sequence: s2pu1

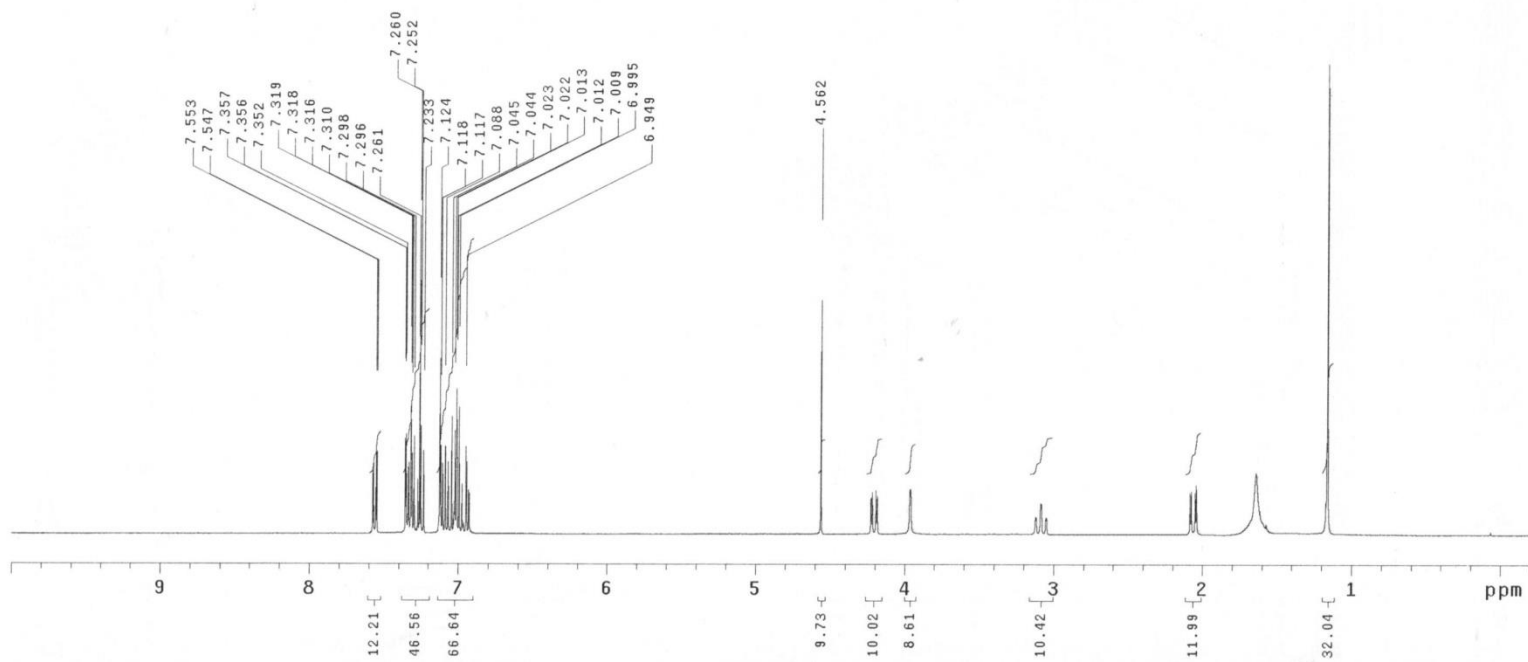
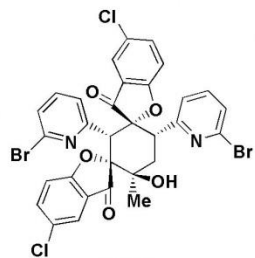
UNITYplus-400 "unity400"

Date: Jan 9 2019

Solvent: CDCl<sub>3</sub>

Ambient temperature

Total 64 repetitions

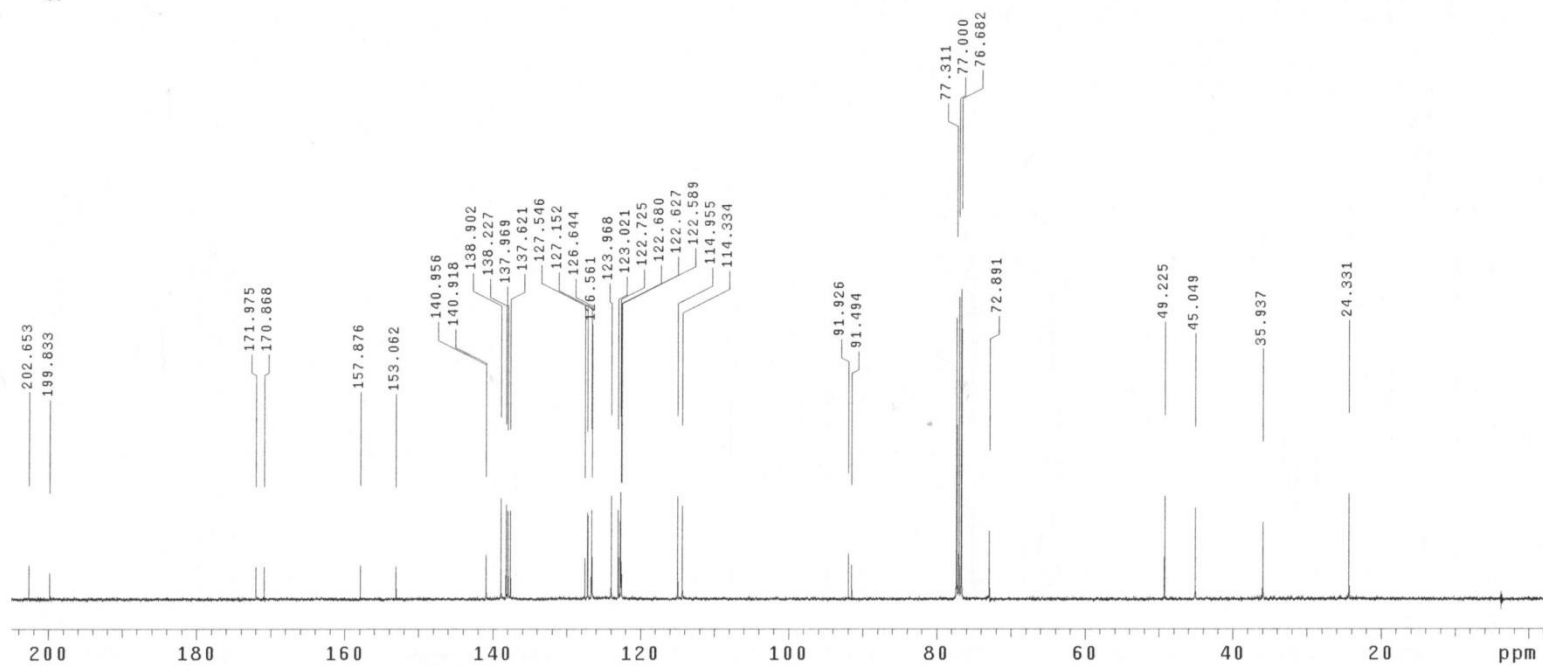
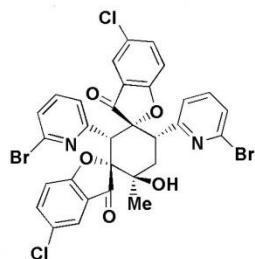




# Compound 3d (<sup>13</sup>C-NMR spectral data)

HA1epyBr

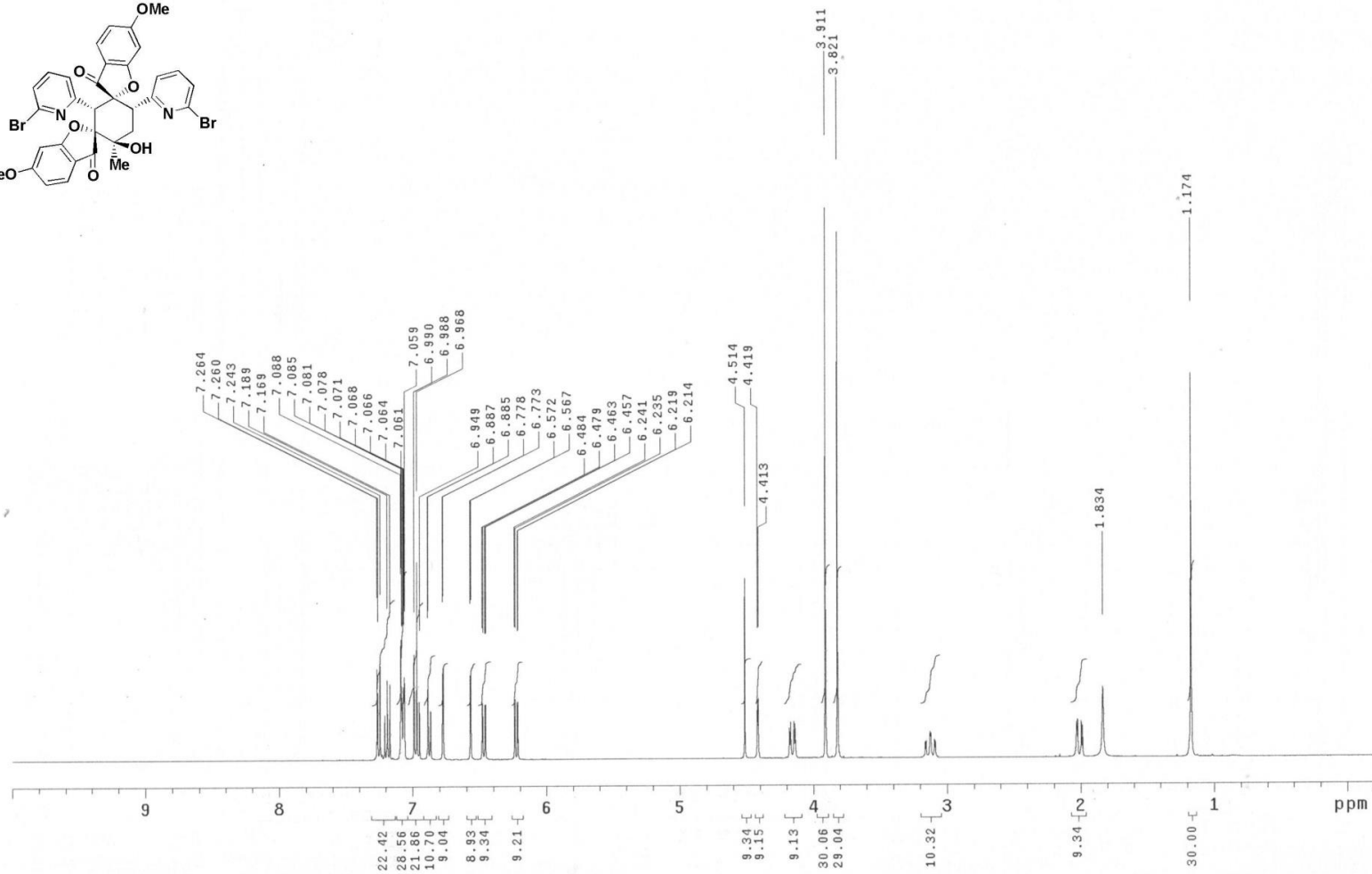
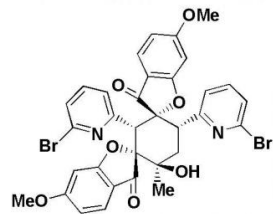
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 9 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32000 repetitions



# Compound 3e (<sup>1</sup>H-NMR spectral data)

KT1080718

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jul 22 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions

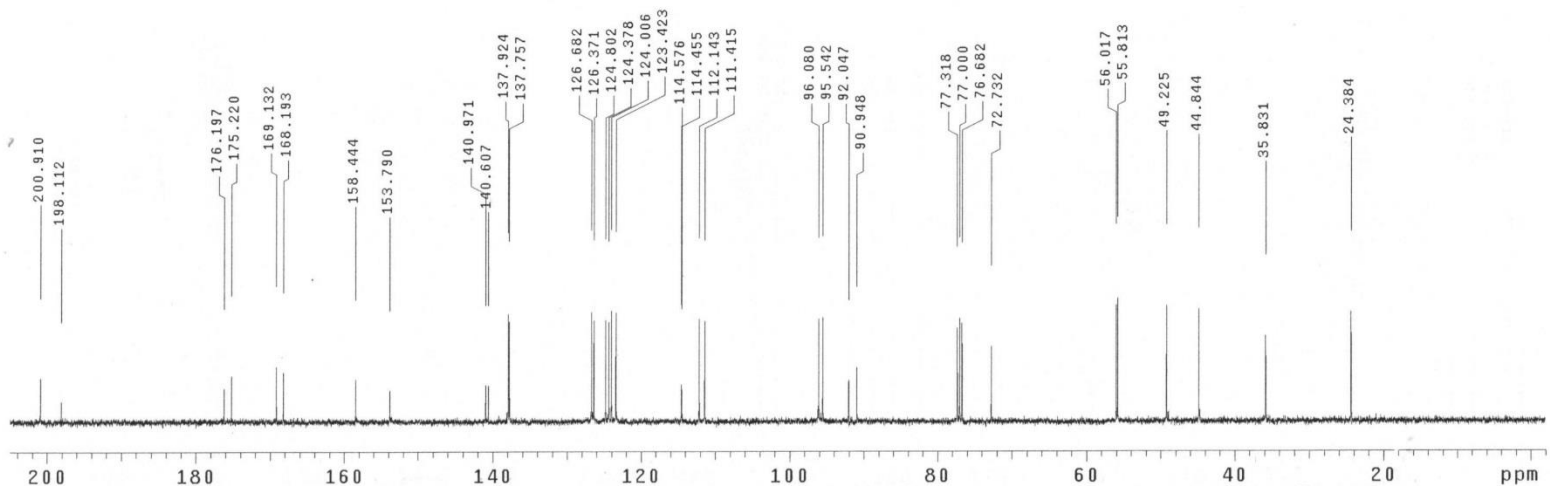
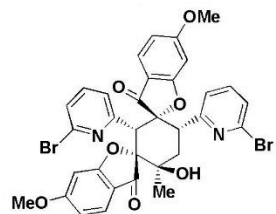


15.82

# Compound 3e (<sup>13</sup>C-NMR spectral data)

KT1080718

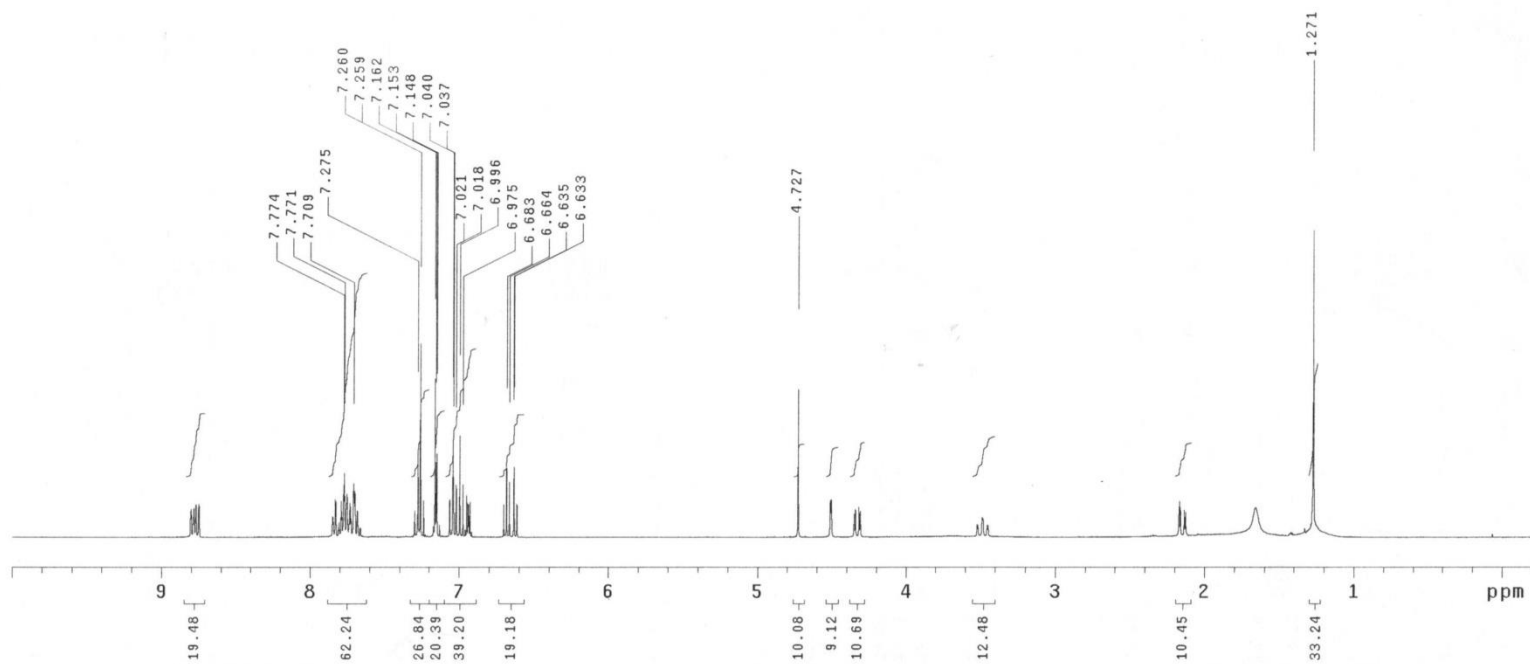
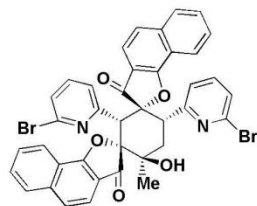
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jul 22 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 1776 repetitions



# Compound 3f (<sup>1</sup>H-NMR spectral data)

HA.NAPYBr

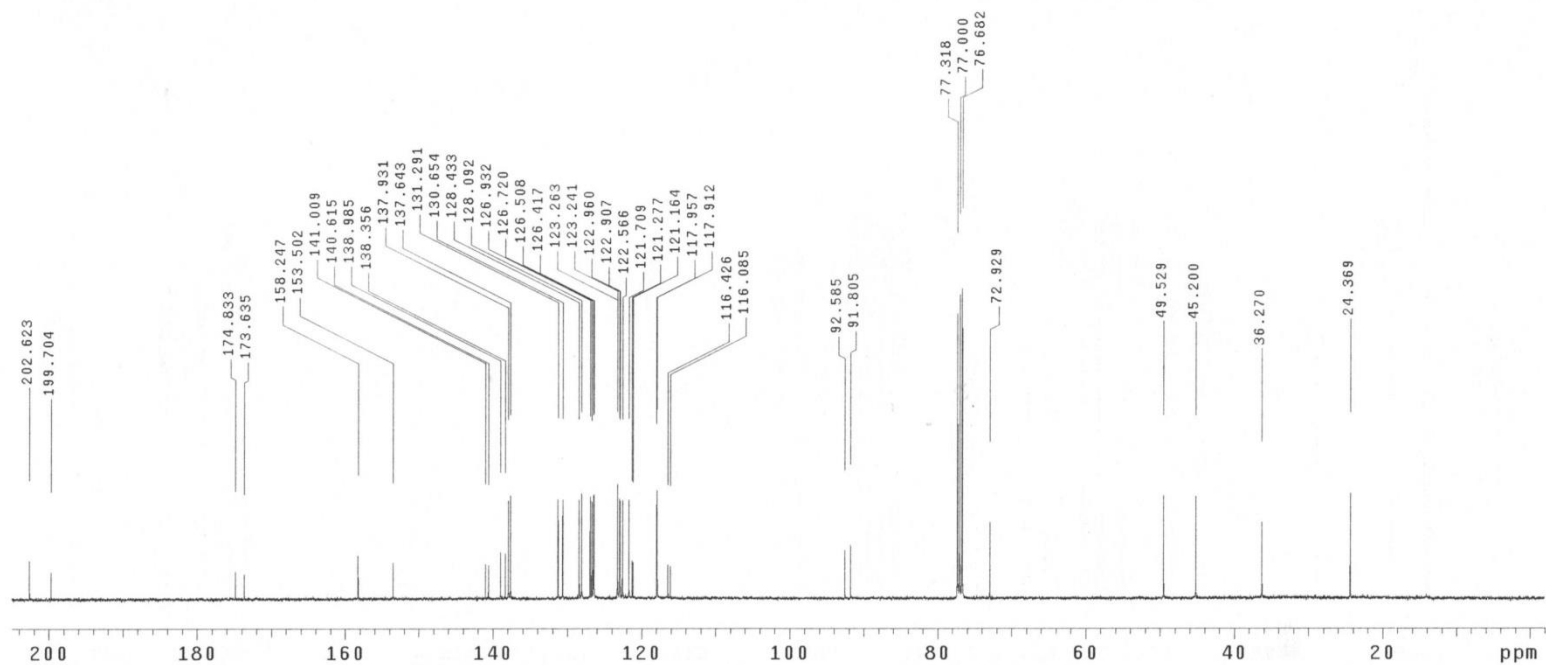
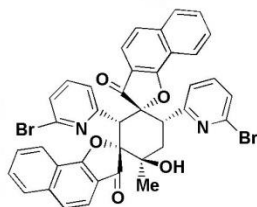
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 29 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 60 repetitions



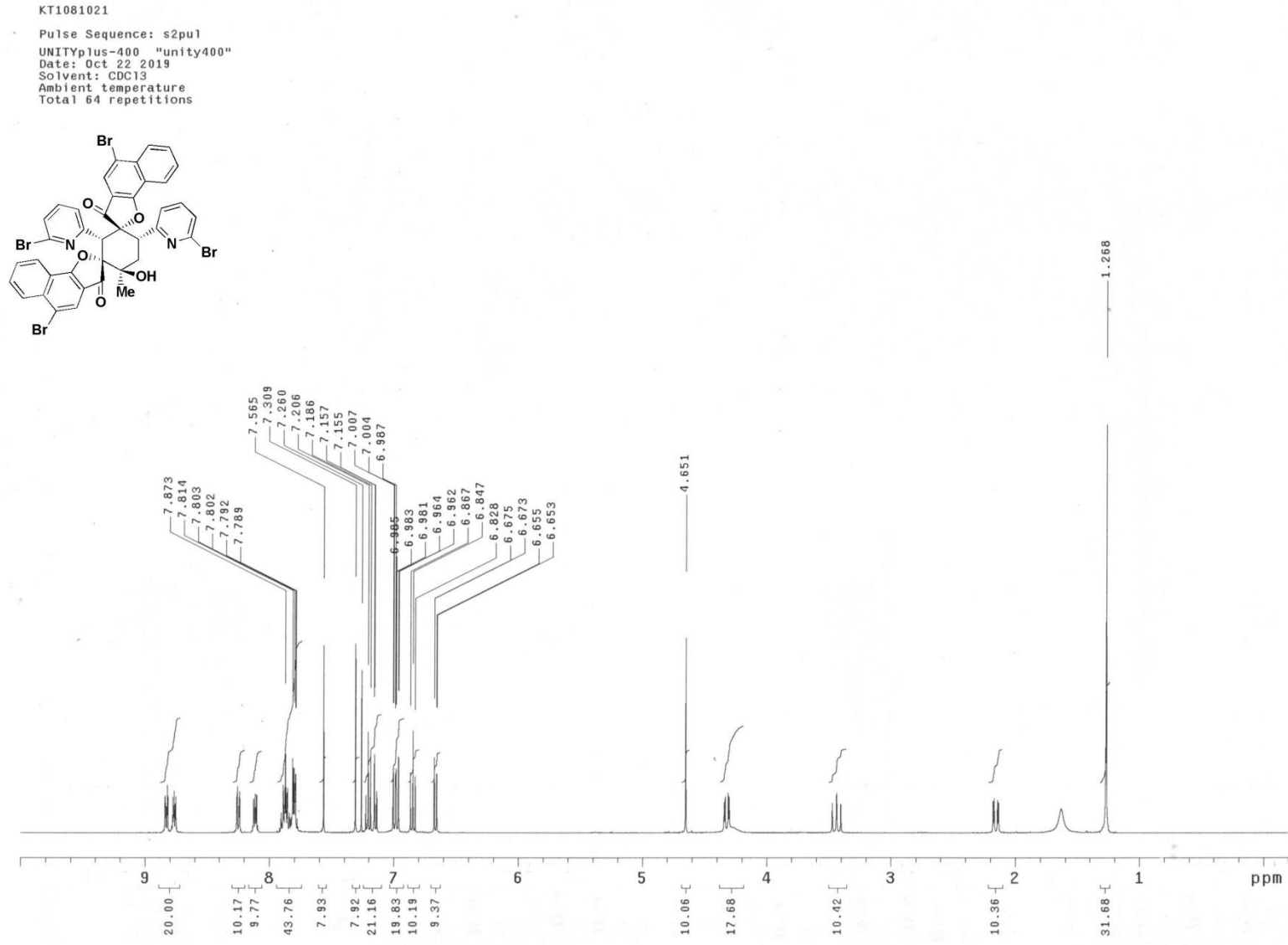
# Compound 3f (<sup>13</sup>C-NMR spectral data)

HA.NAPYBr

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 29 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 29296 repetitions



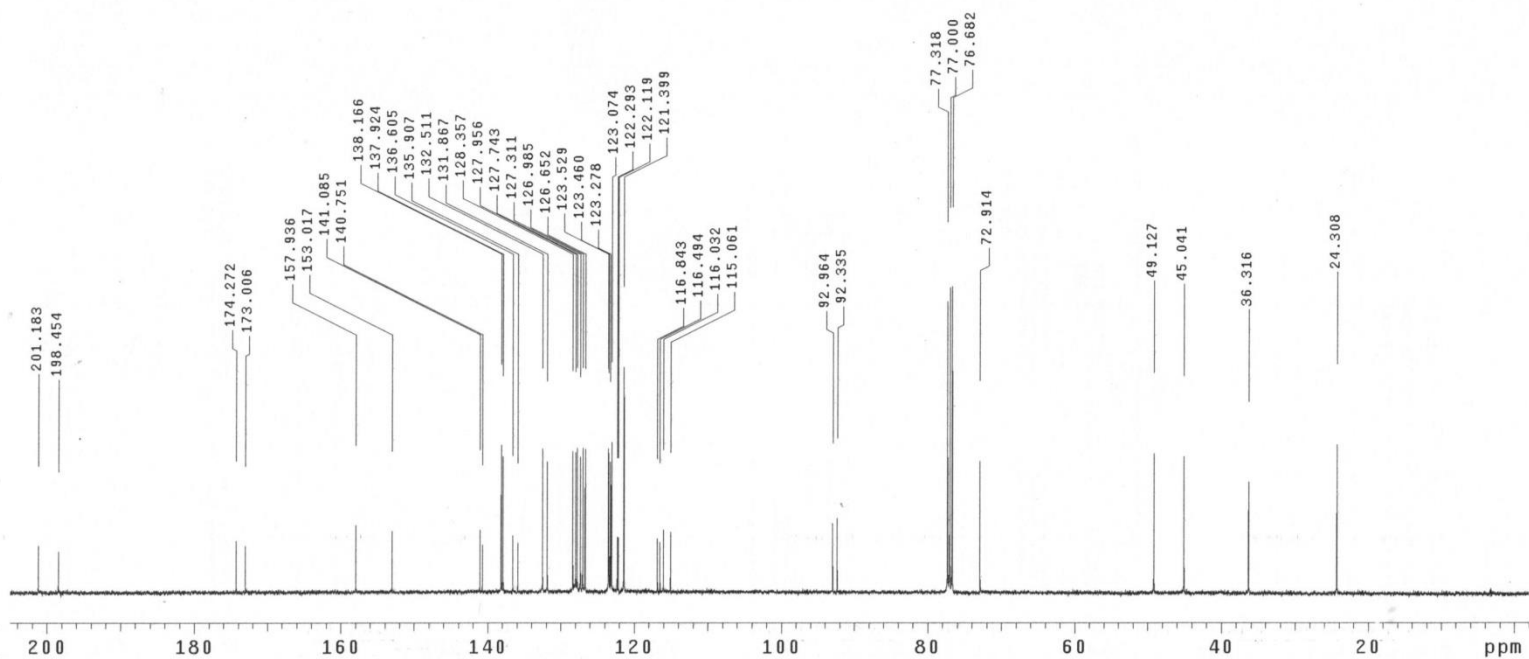
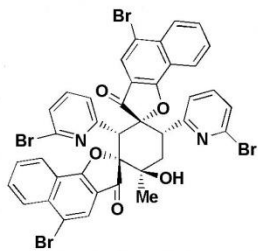
# Compound 3g (<sup>1</sup>H-NMR spectral data)



# Compound 3g (<sup>13</sup>C-NMR spectral data)

KT1081021

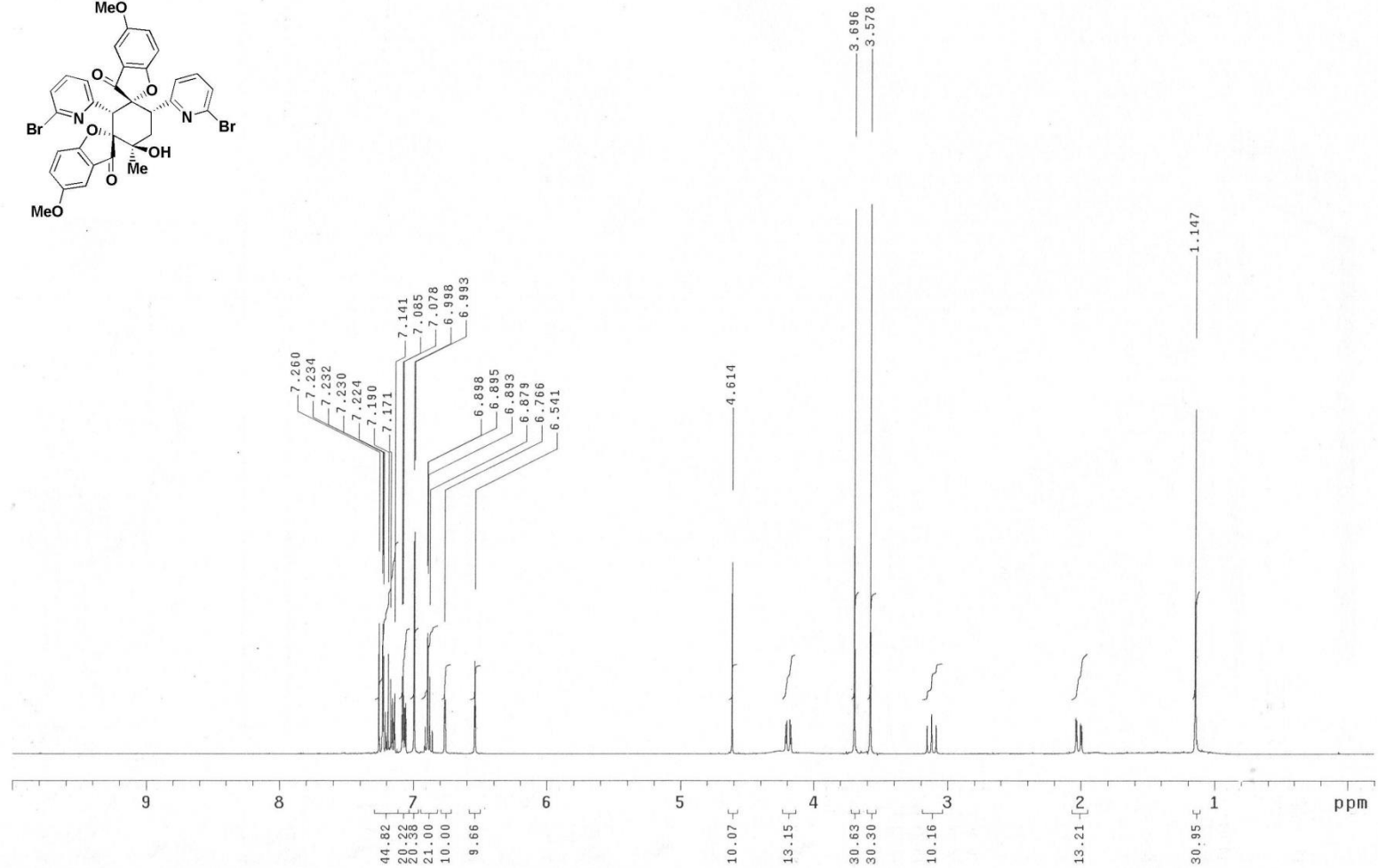
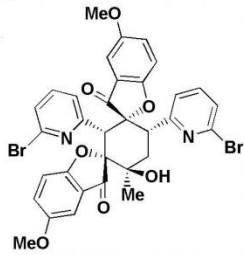
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 22 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 20480 repetitions



# Compound 3h (<sup>1</sup>H-NMR spectral data)

KT1080826

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 30 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions

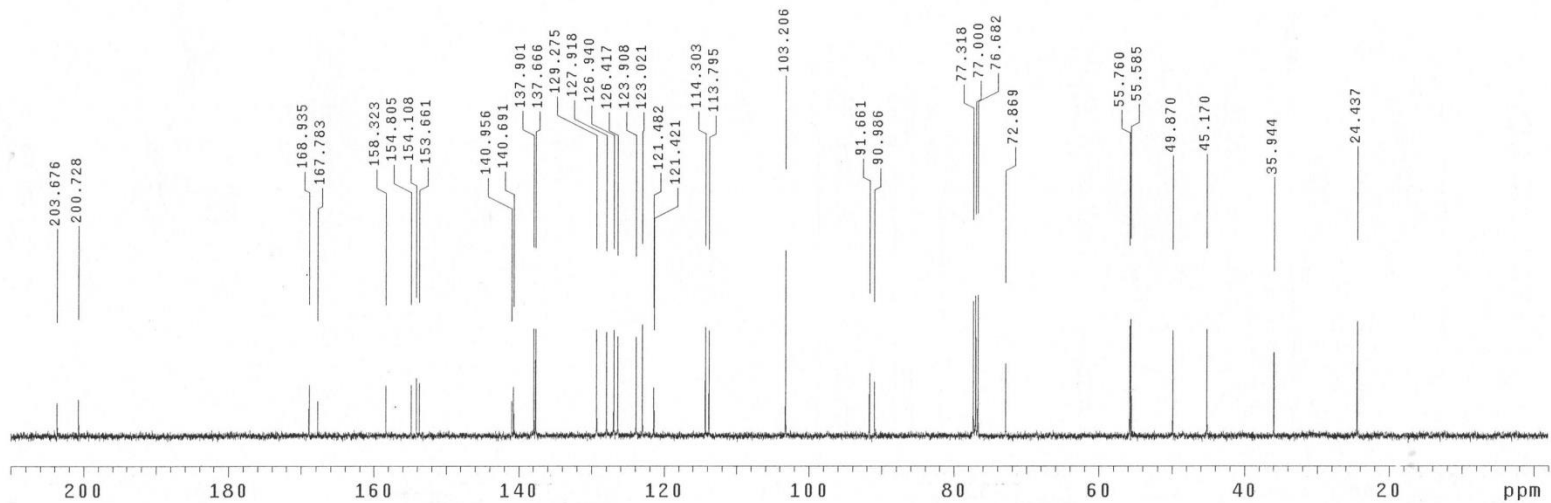
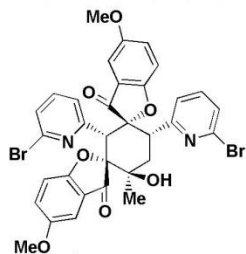




# Compound 3h (<sup>13</sup>C-NMR spectral data)

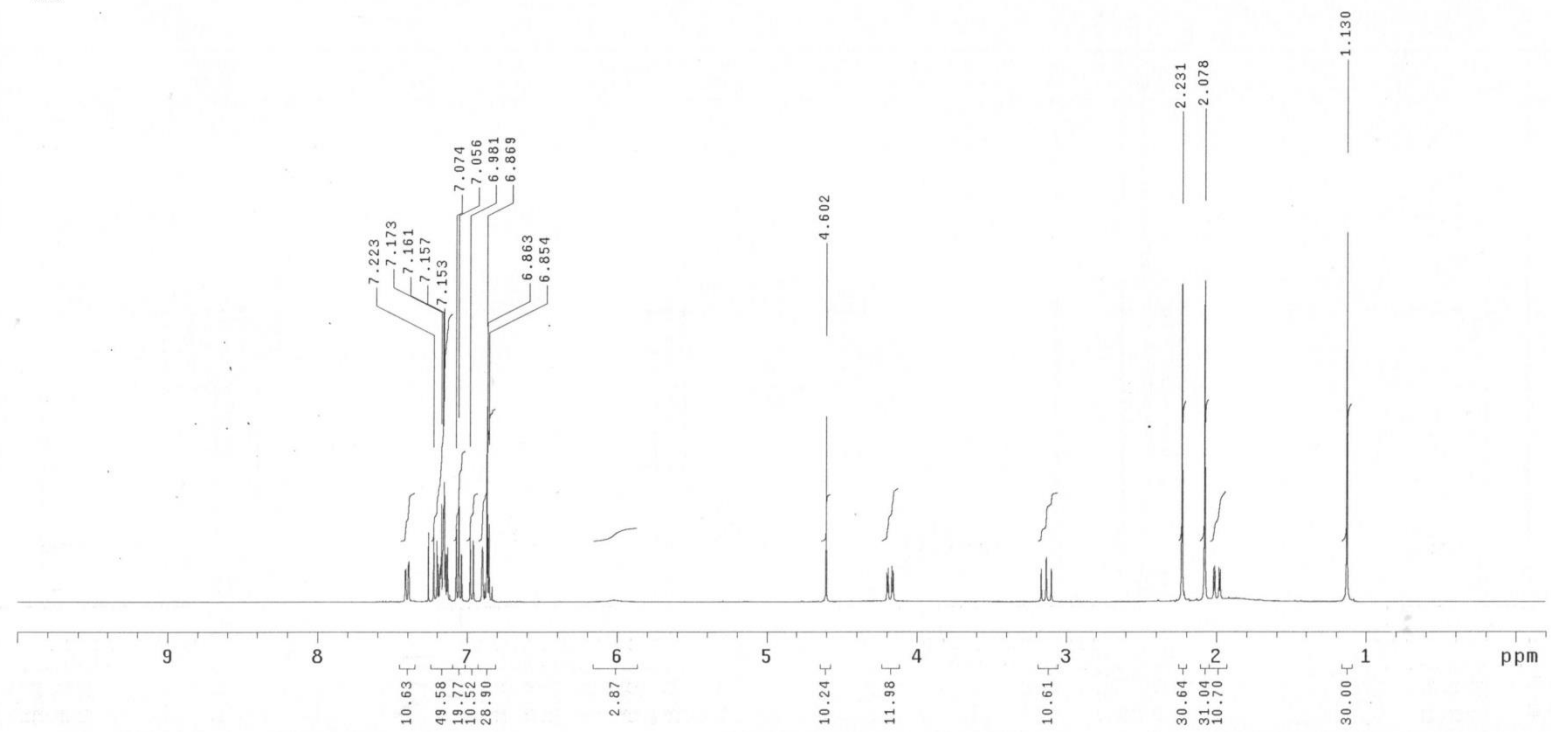
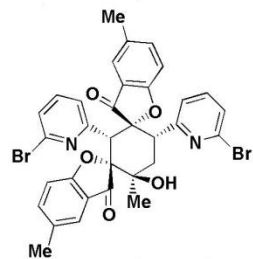
KT1080826

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 30 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2016 repetitions



# Compound 3i (<sup>1</sup>H-NMR spectral data)

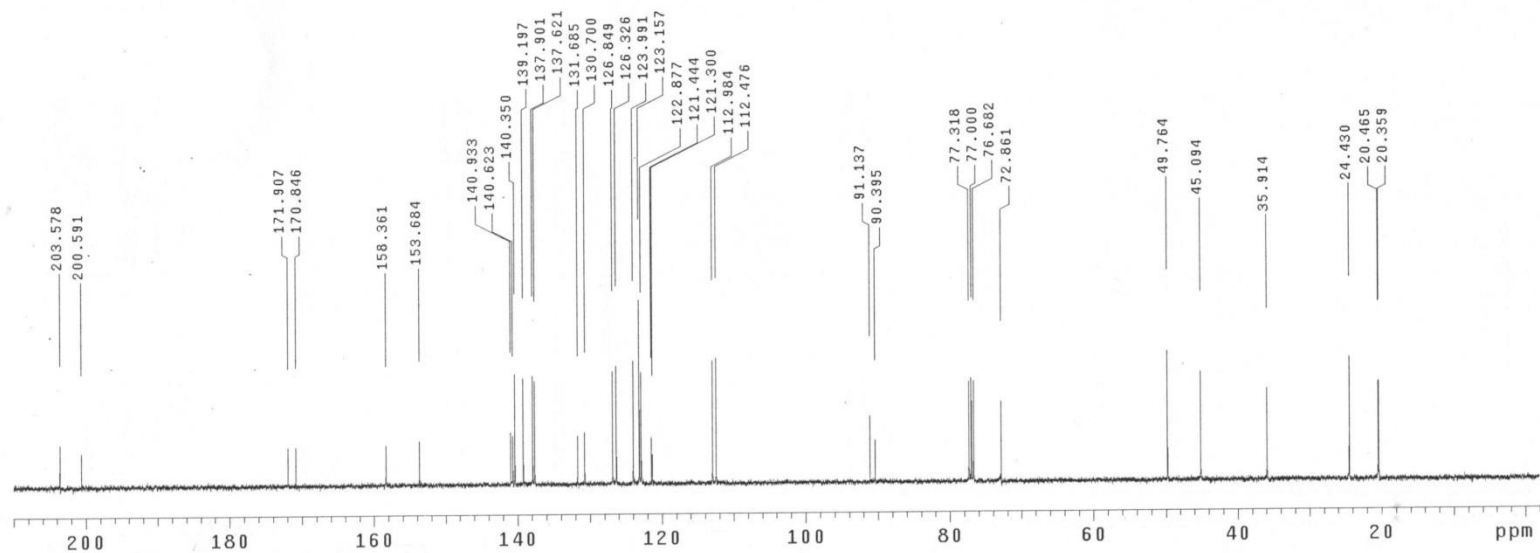
KT1080917  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 18 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions



# Compound 3i (<sup>13</sup>C-NMR spectral data)

KT1080917

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 18 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2048 repetitions

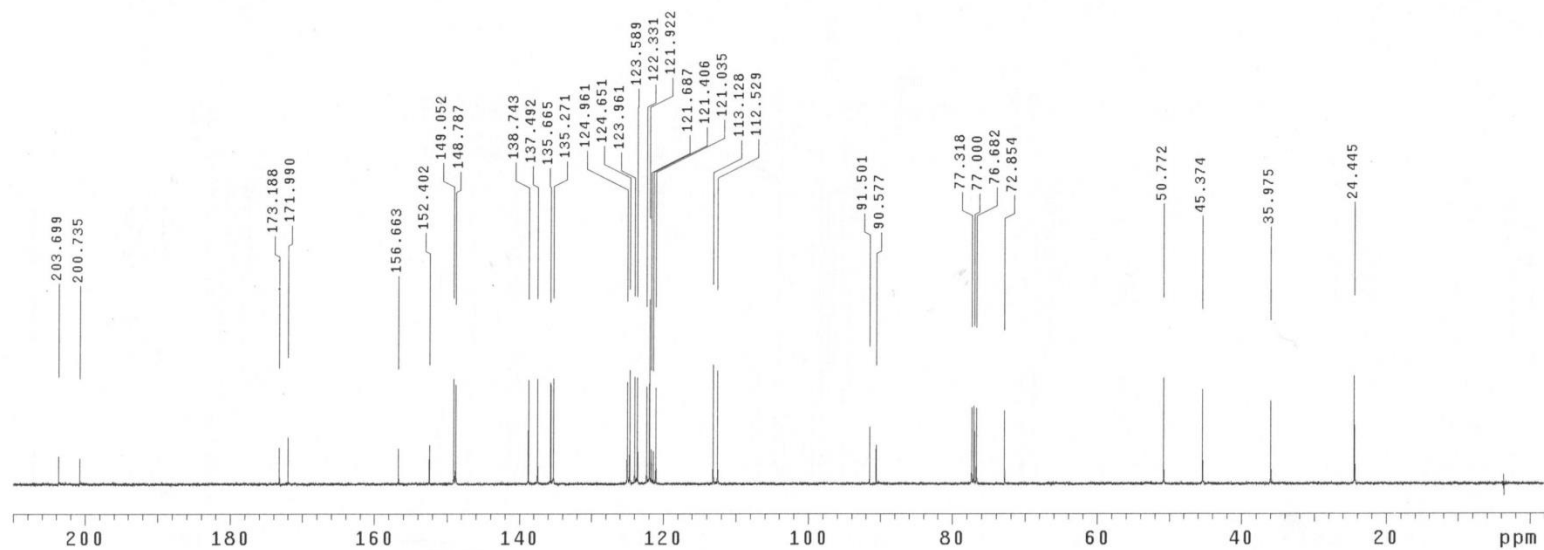
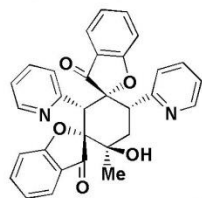




# Compound 3j (<sup>13</sup>C-NMR spectral data)

T1PhP

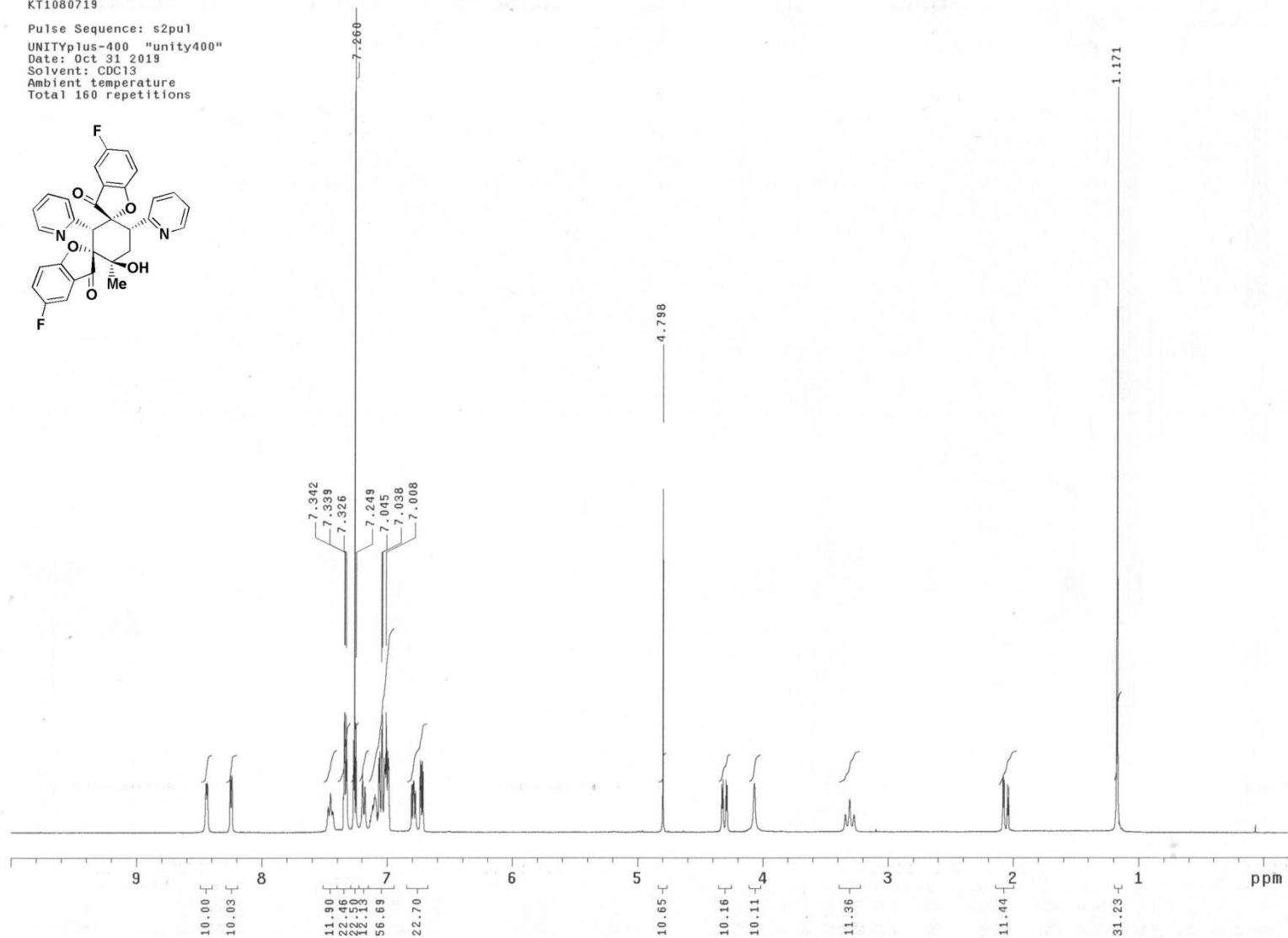
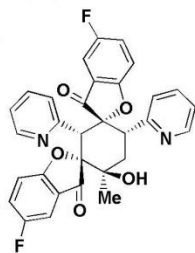
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Feb 25 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 3008 repetitions



# Compound 3k (<sup>1</sup>H-NMR spectral data)

KT1080719

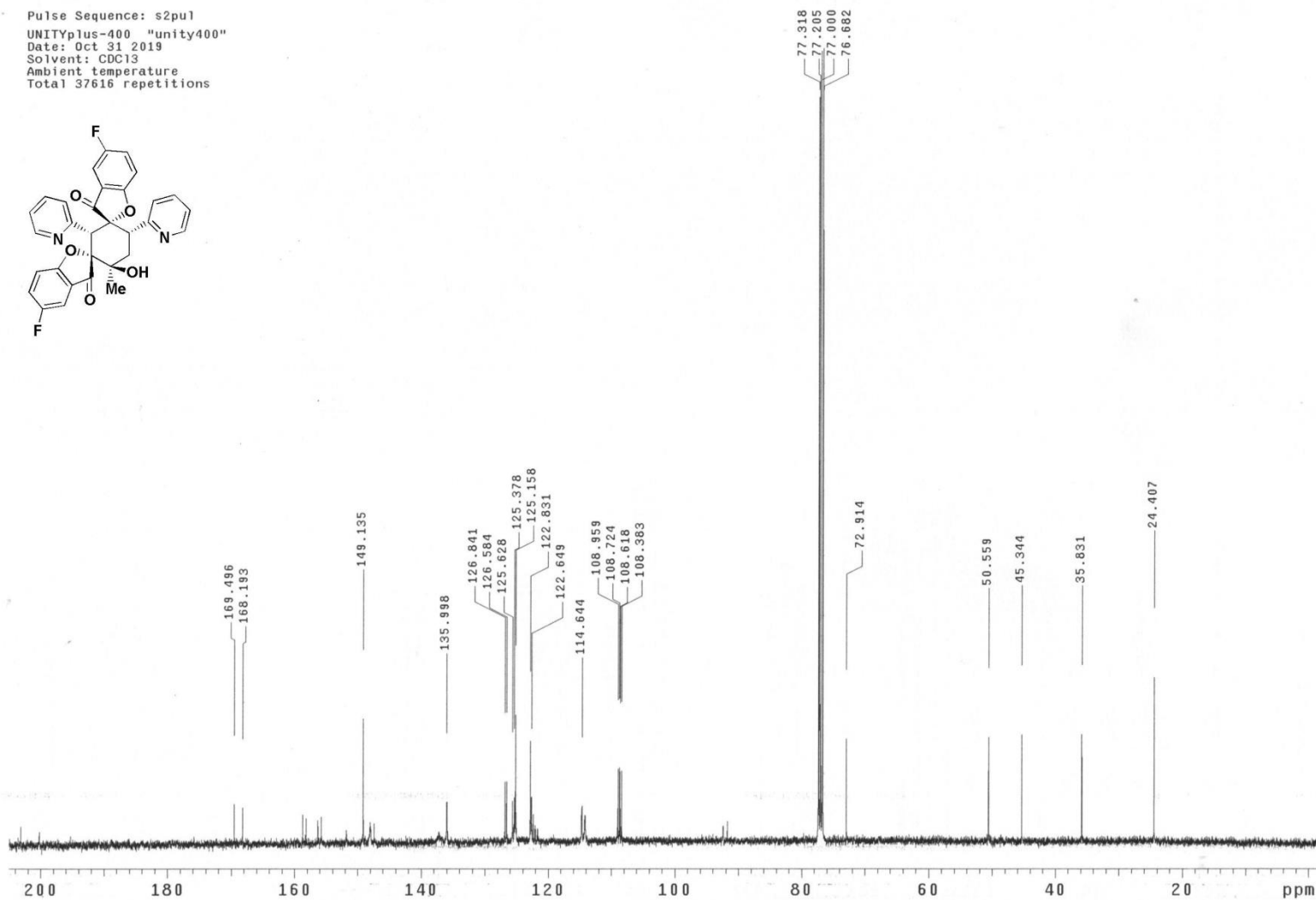
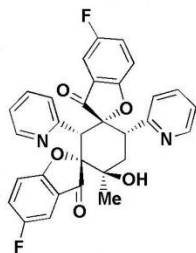
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 31 2019  
Solvent: CDC13  
Ambient temperature  
Total 160 repetitions



# Compound 3k (<sup>13</sup>C-NMR spectral data)

KT1080719

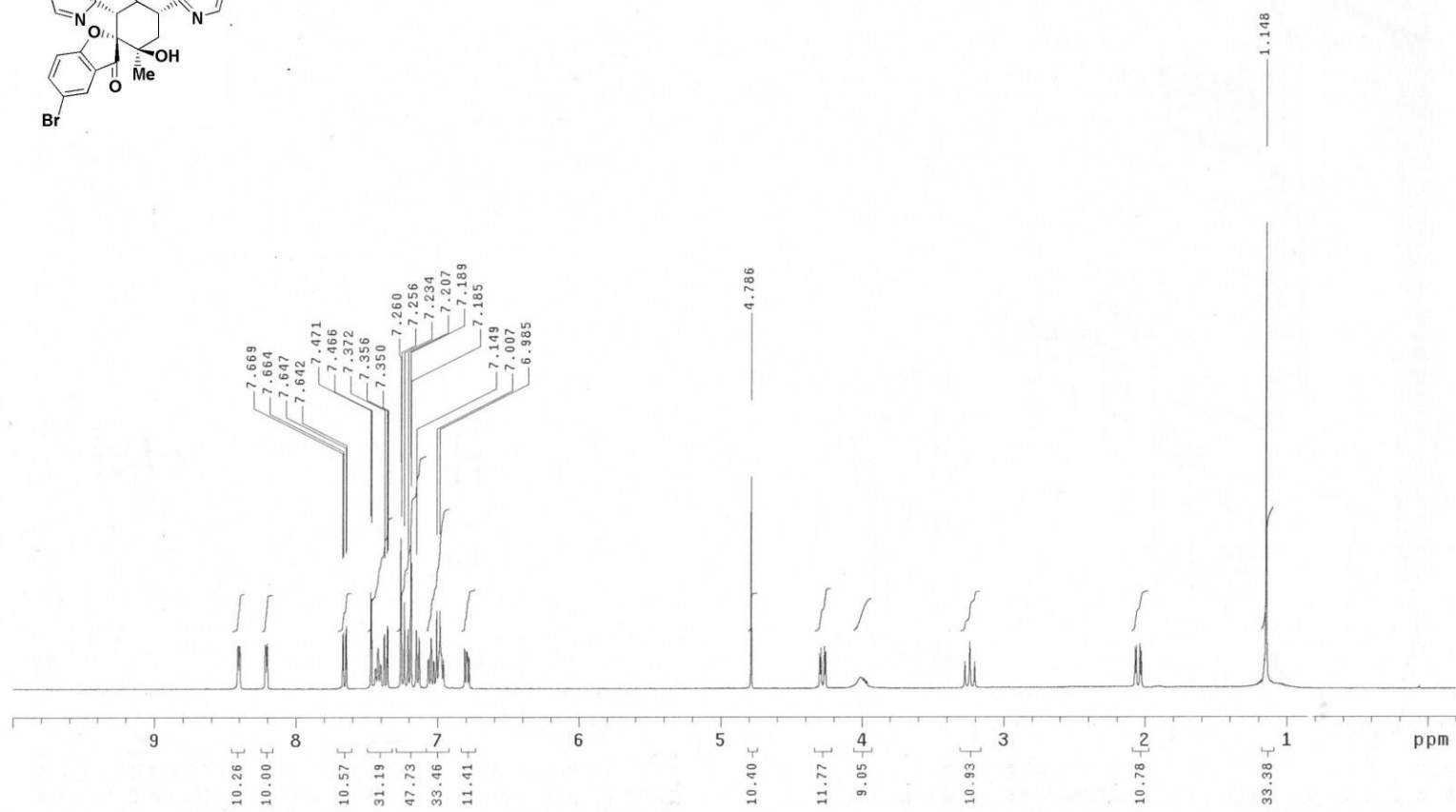
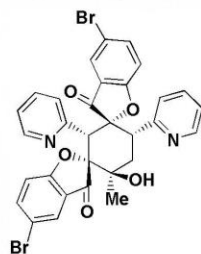
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 31 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 37616 repetitions



# Compound 3I (<sup>1</sup>H-NMR spectral data)

KT1080711

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 29 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions

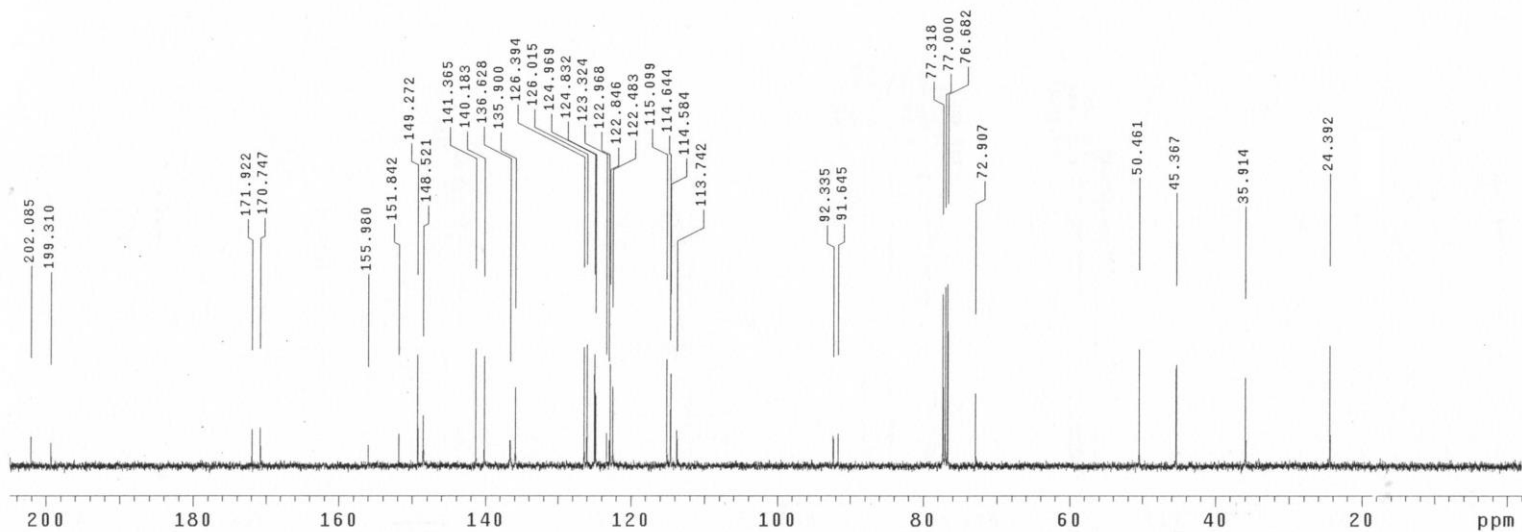
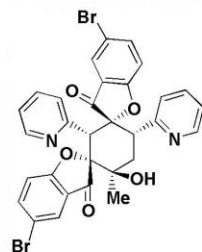




# Compound 3l (<sup>13</sup>C-NMR spectral data)

KT1080711

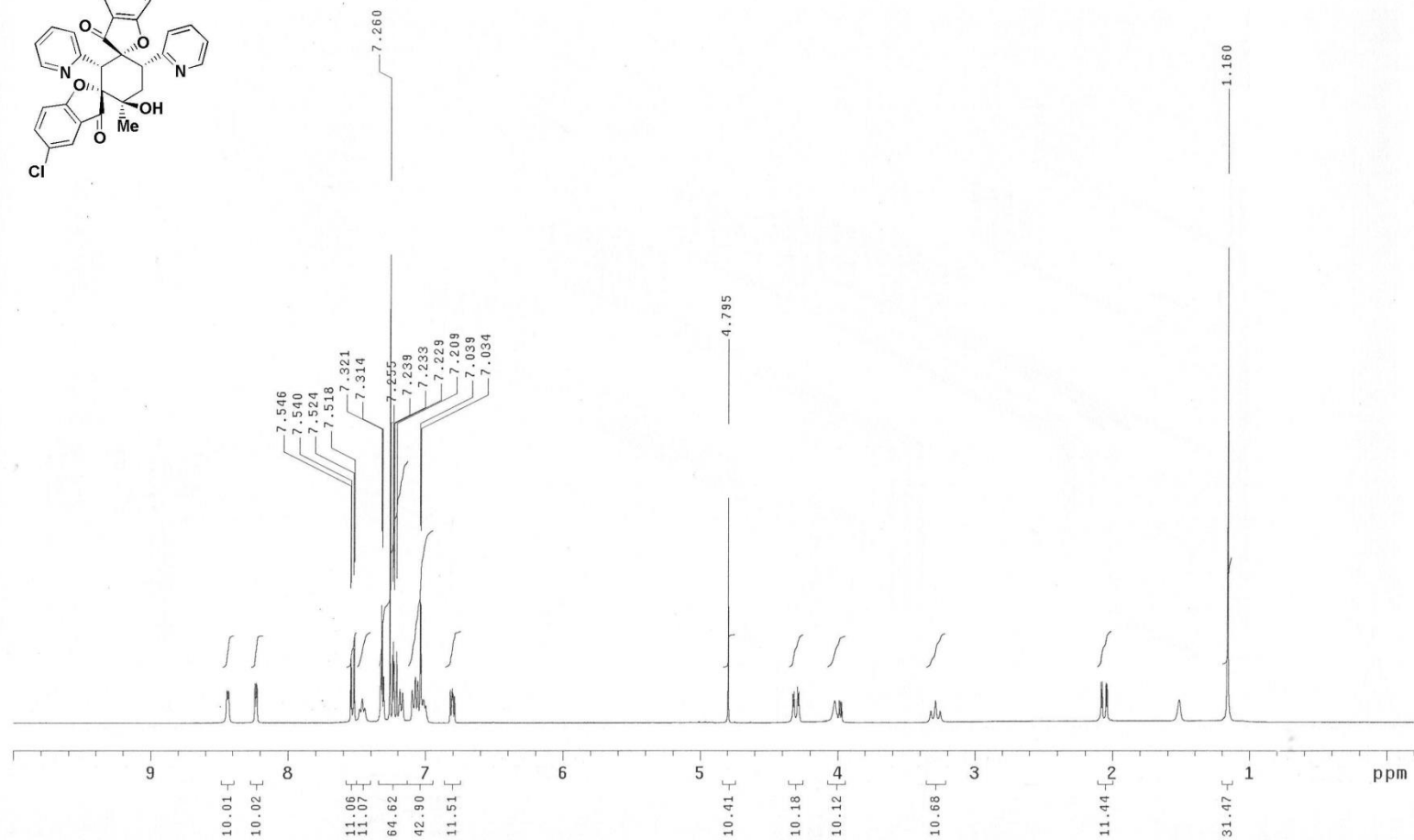
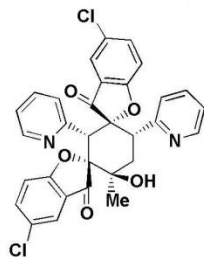
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 29 2019  
Solvent: CDC13  
Ambient temperature  
Total 3136 repetitions



# Compound 3m (<sup>1</sup>H-NMR spectral data)

KT1080729

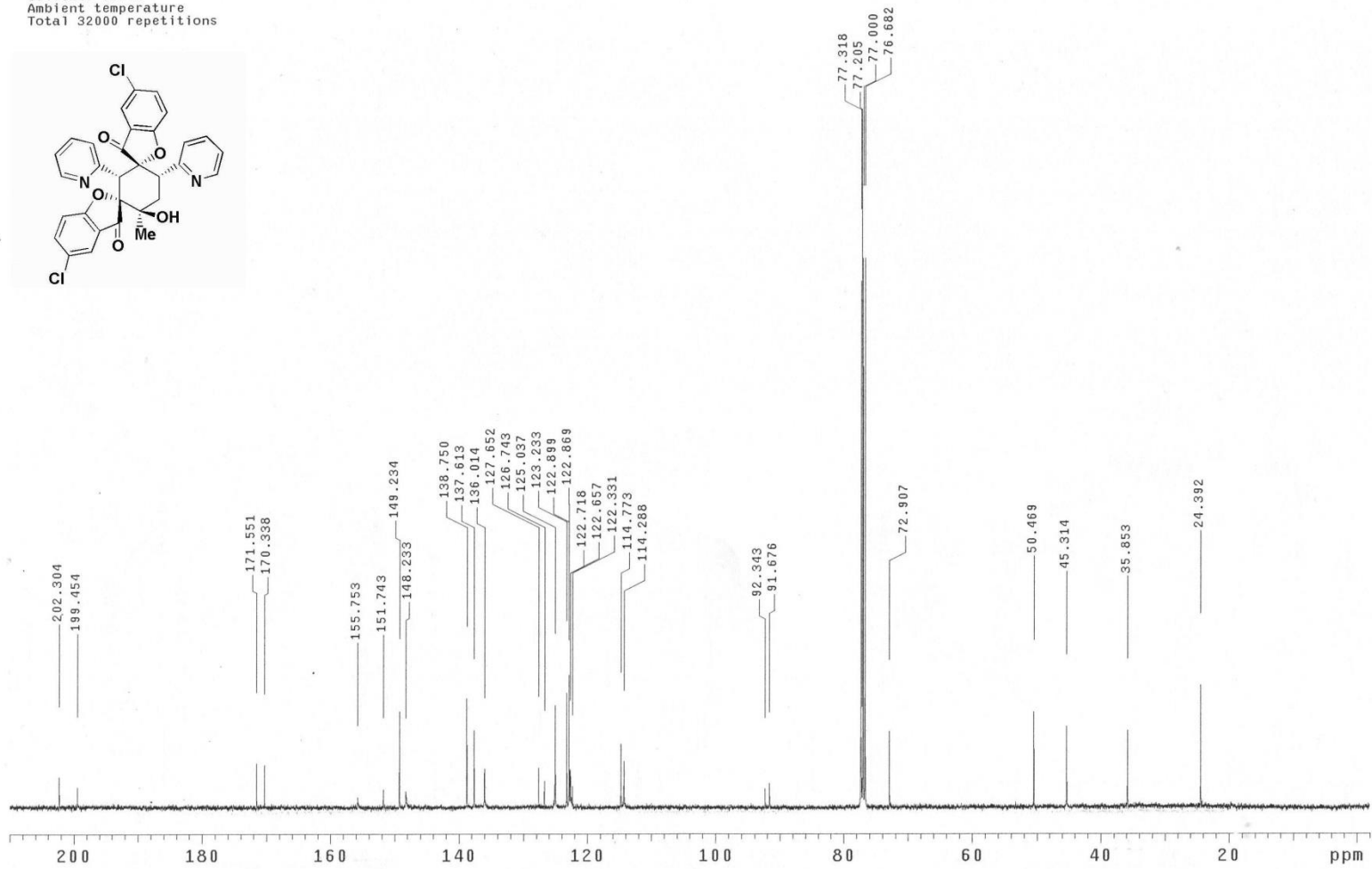
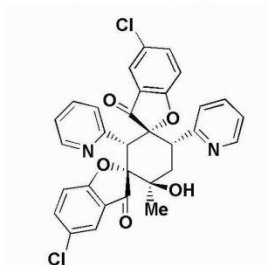
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 2 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 276 repetitions



# Compound 3m (<sup>13</sup>C-NMR spectral data)

KT1080729

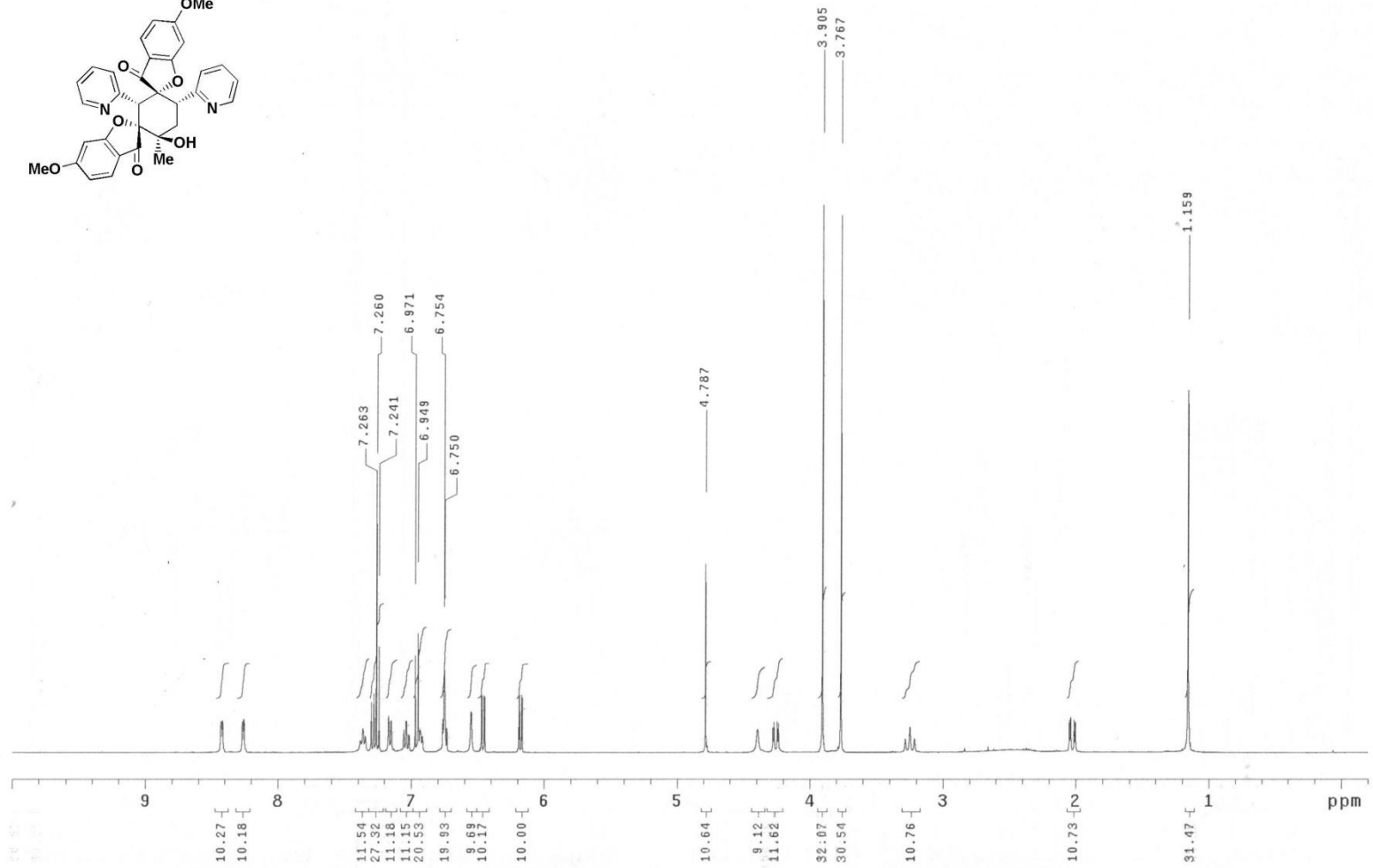
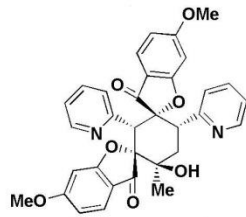
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 2 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32000 repetitions



# Compound 3n (<sup>1</sup>H-NMR spectral data)

KT1080722

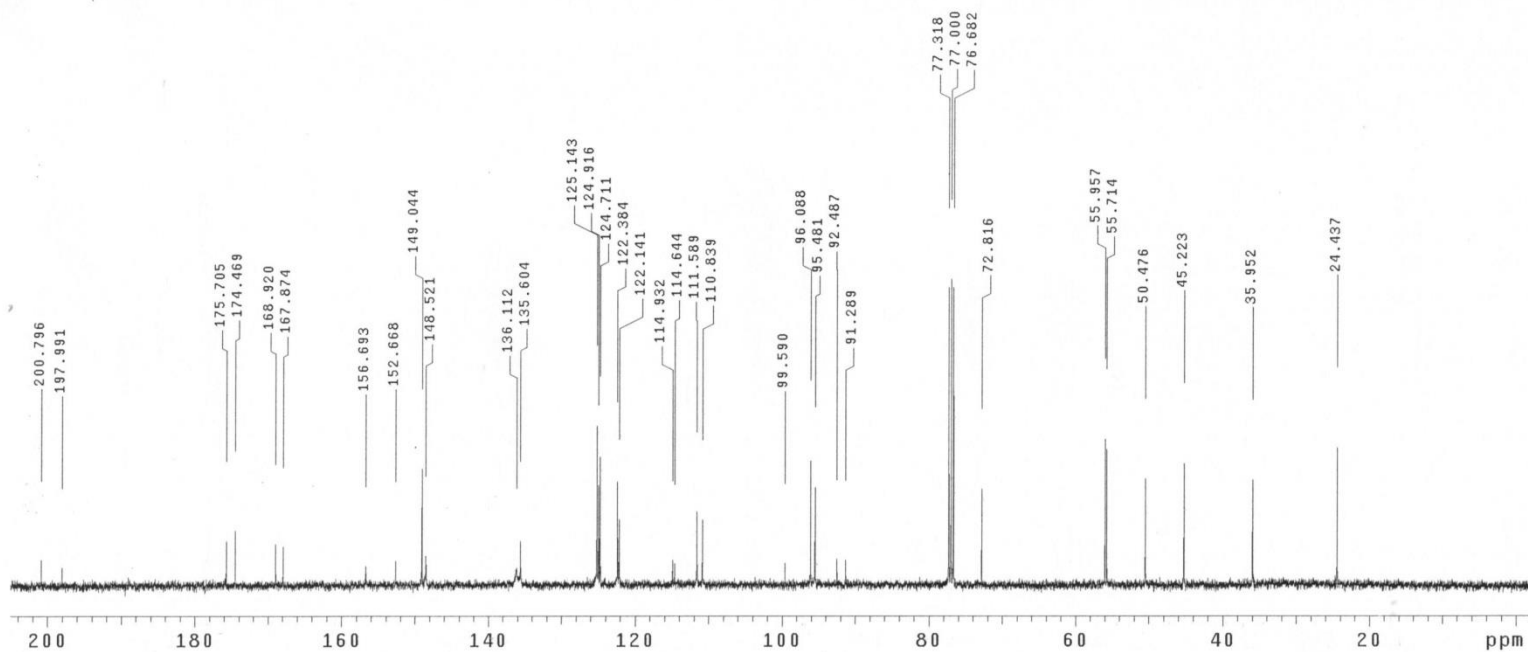
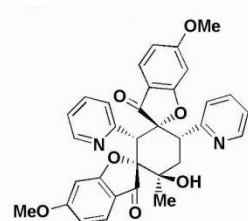
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 12 2019  
Solvent: CDCl3  
Ambient temperature  
Total 64 repetitions



# Compound 3n (<sup>13</sup>C-NMR spectral data)

KT1080722

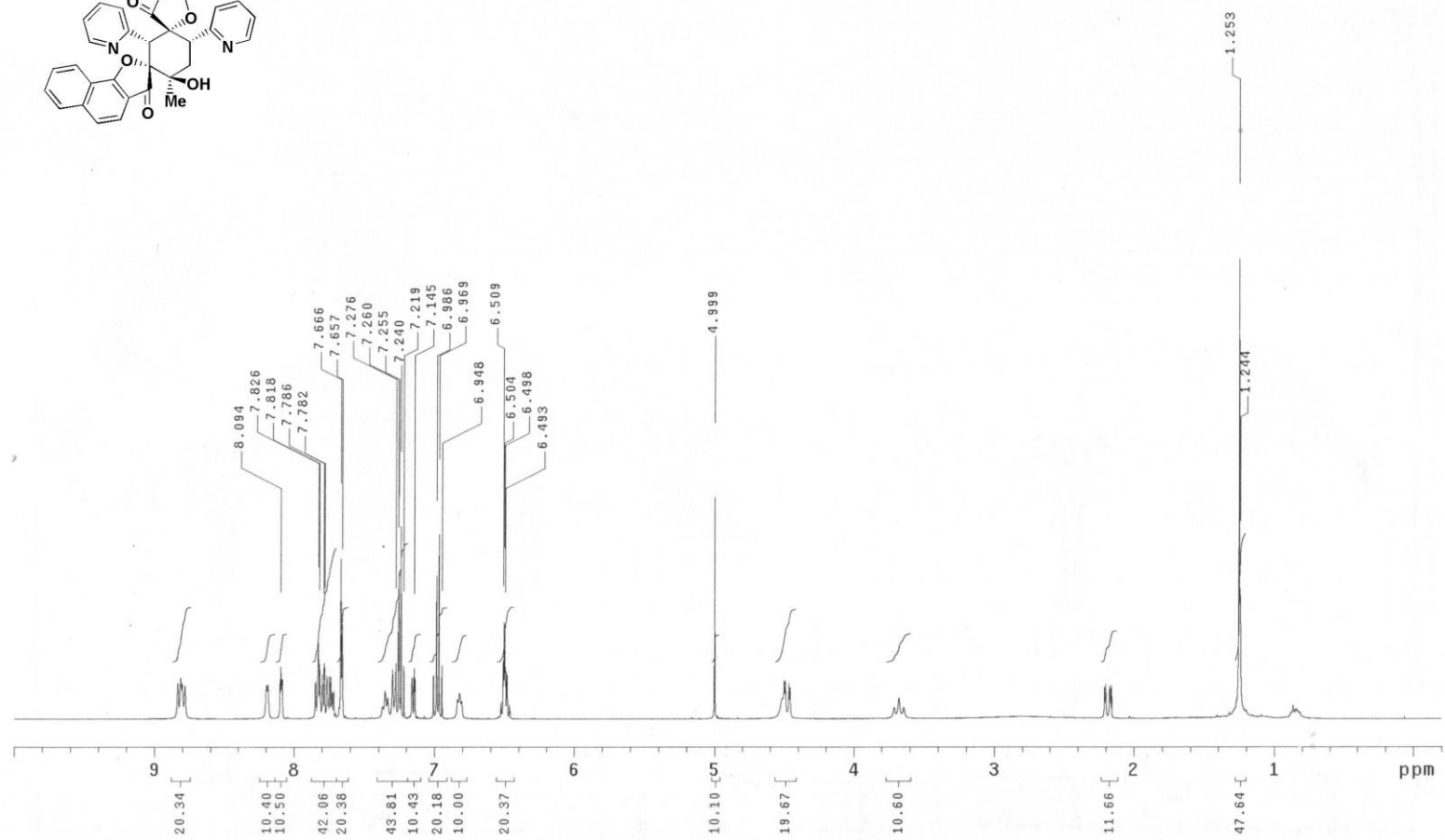
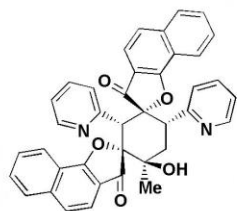
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 12 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 6320 repetitions



# Compound 3o (<sup>1</sup>H-NMR spectral data)

KT1080814

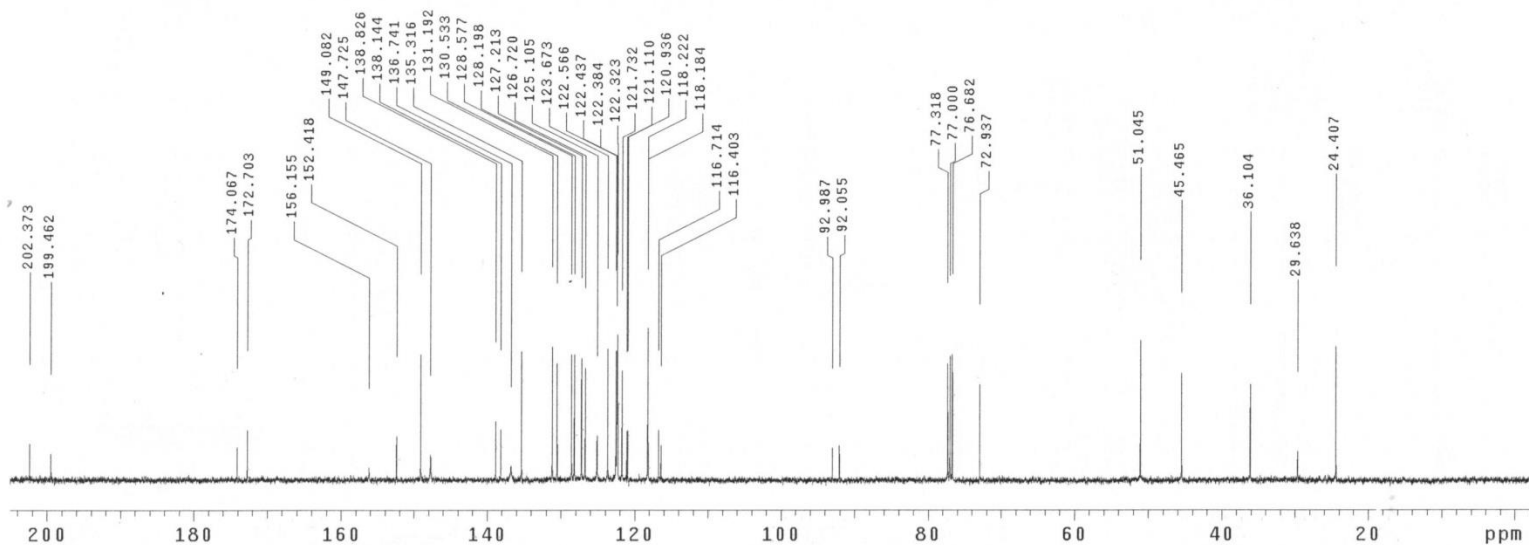
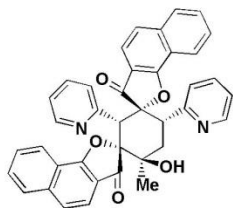
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 15 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 3o (<sup>13</sup>C-NMR spectral data)

KT1080814

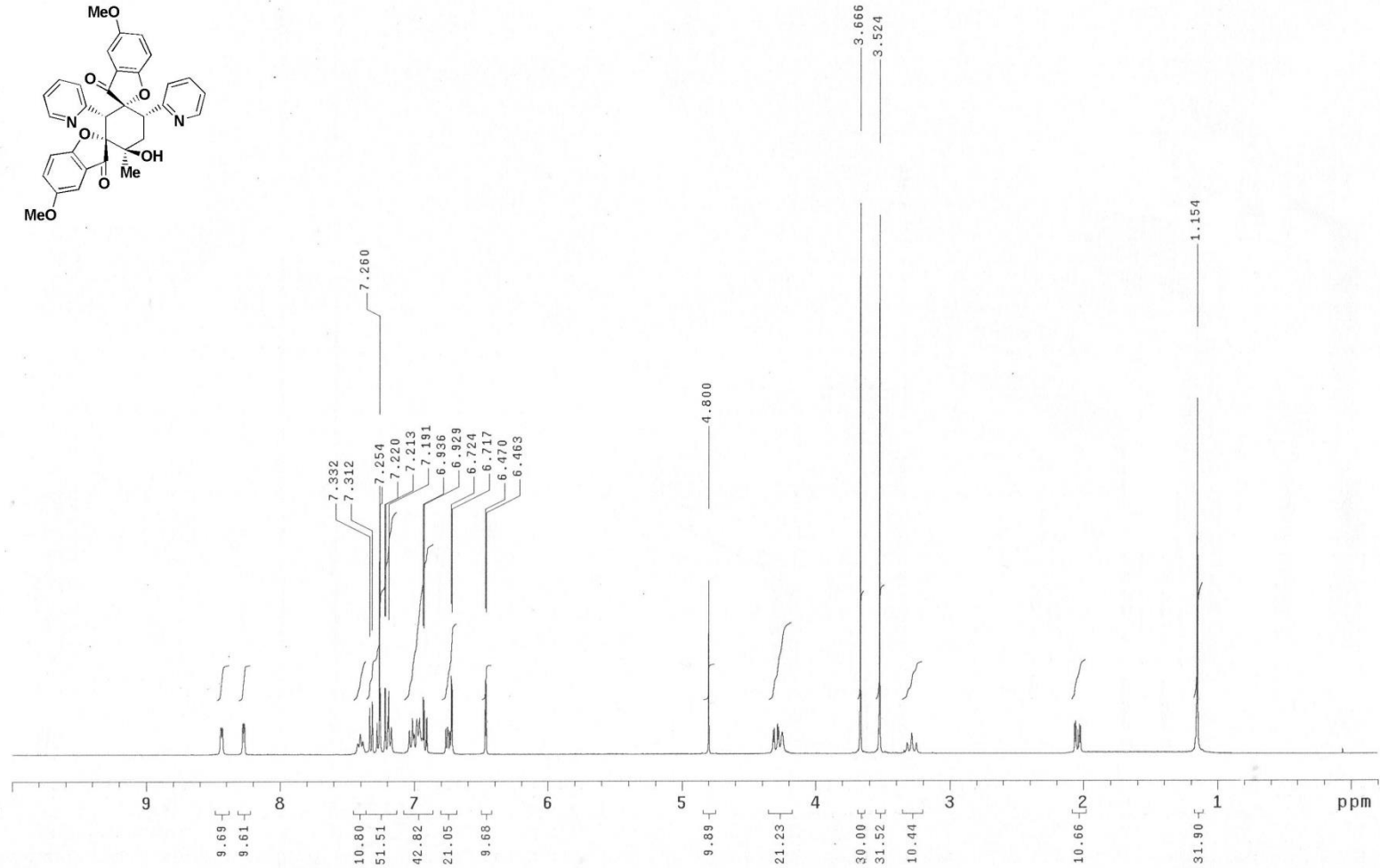
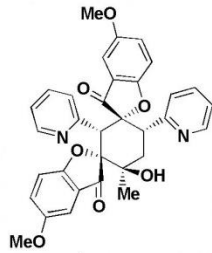
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 15 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2176 repetitions



# Compound 3p (<sup>1</sup>H-NMR spectral data)

KT1080904

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 4 2019  
Solvent: CDCl3  
Ambient temperature  
Total 160 repetitions



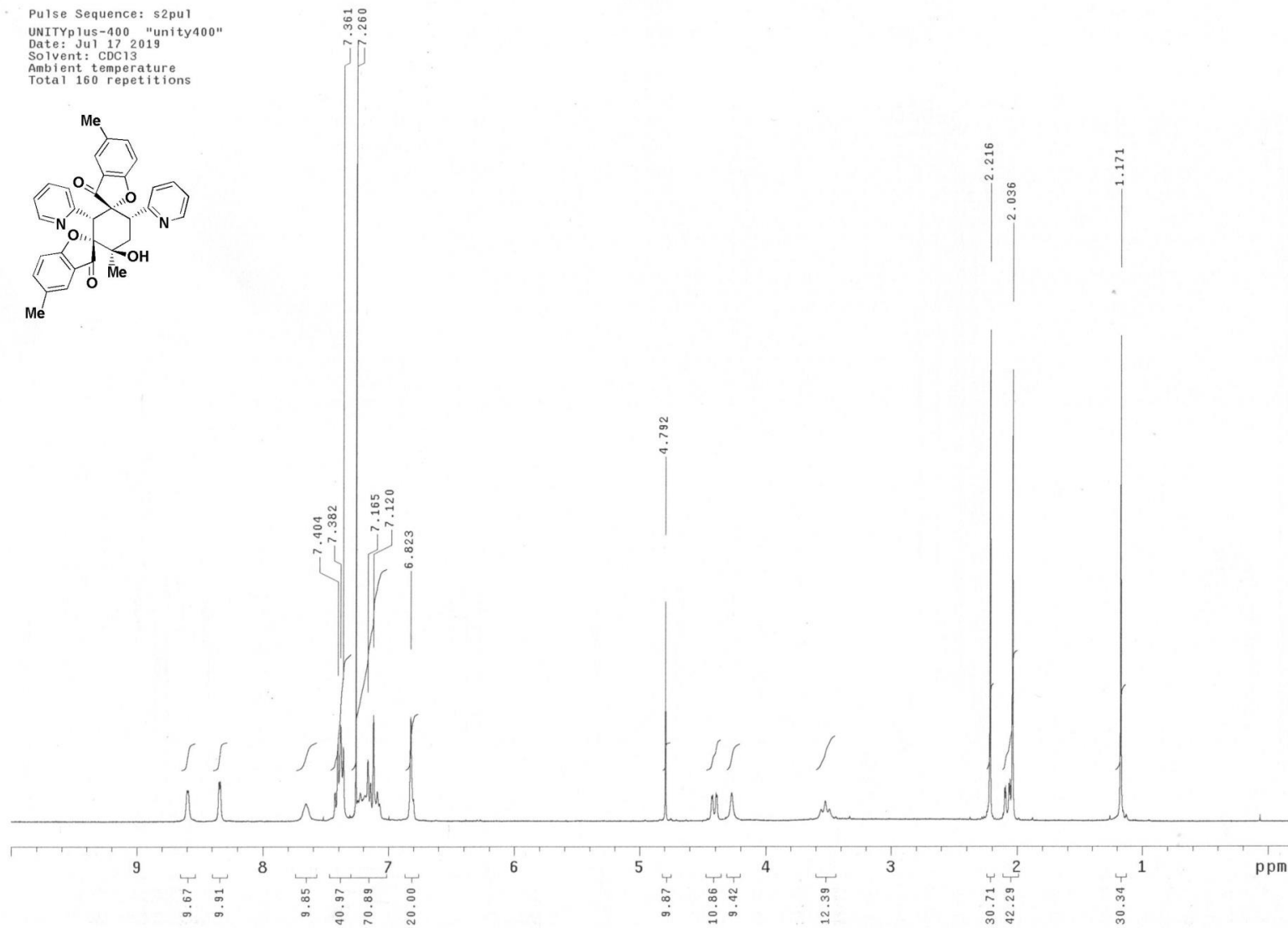
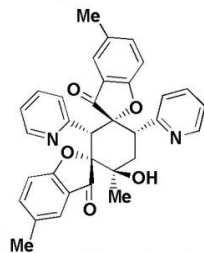




# Compound 3q (<sup>1</sup>H-NMR spectral data)

KT1080715

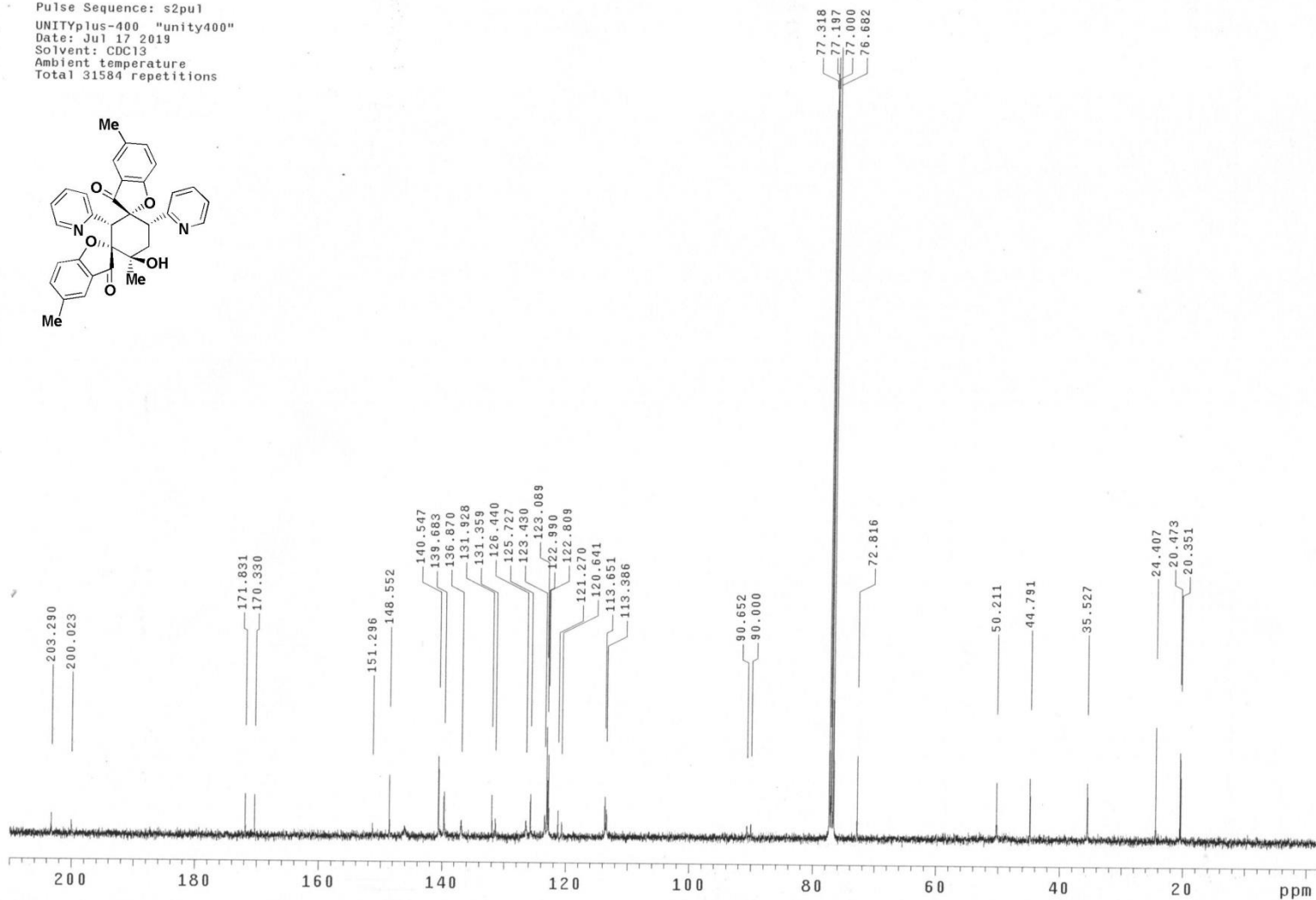
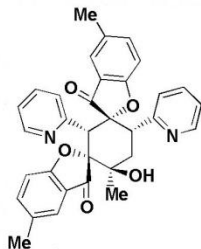
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jul 17 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 160 repetitions



# Compound 3q (<sup>13</sup>C-NMR spectral data)

KT1080715

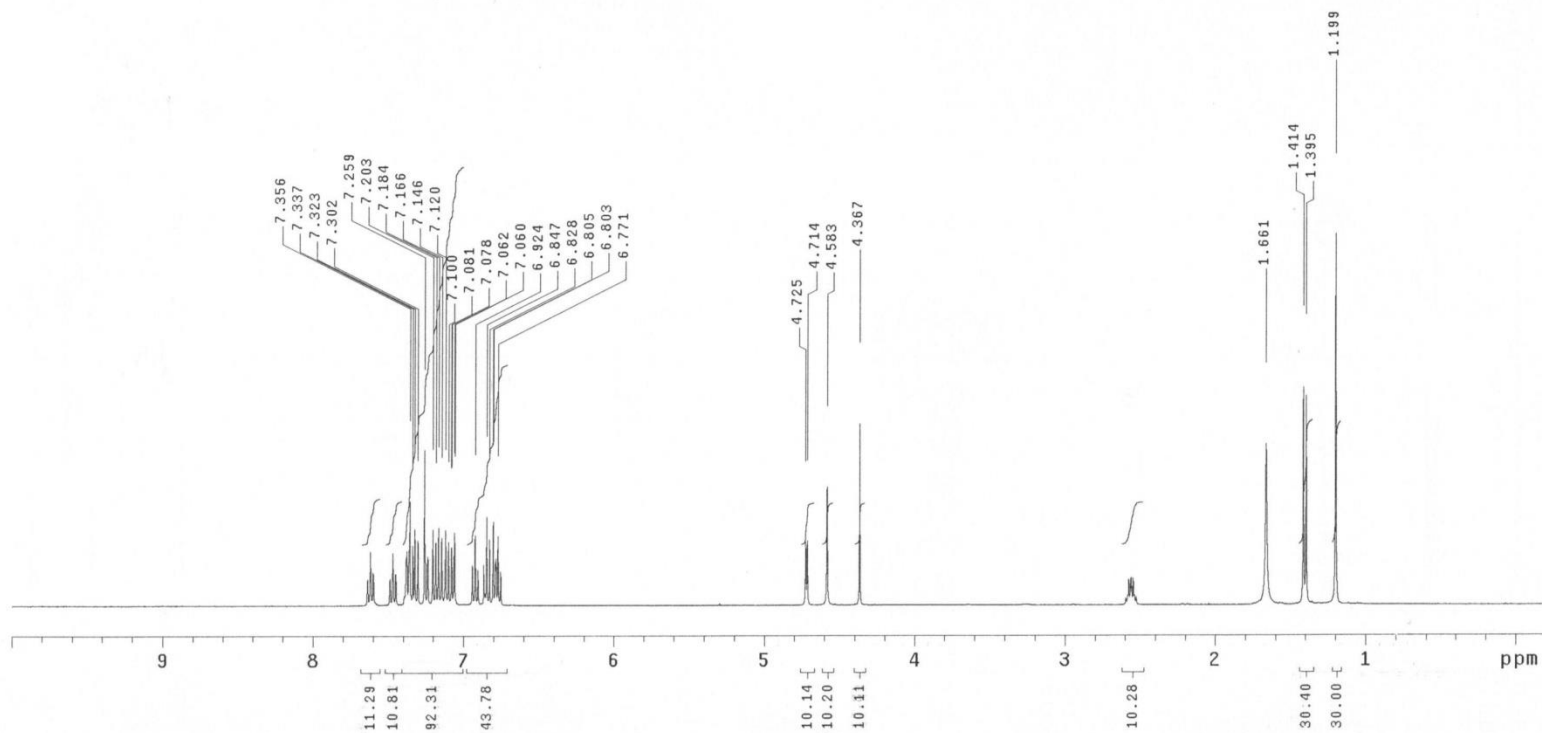
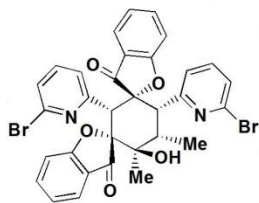
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jul 17 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 31584 repetitions



# Compound 3r (<sup>1</sup>H-NMR spectral data)

KT1080611

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jun 17 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions

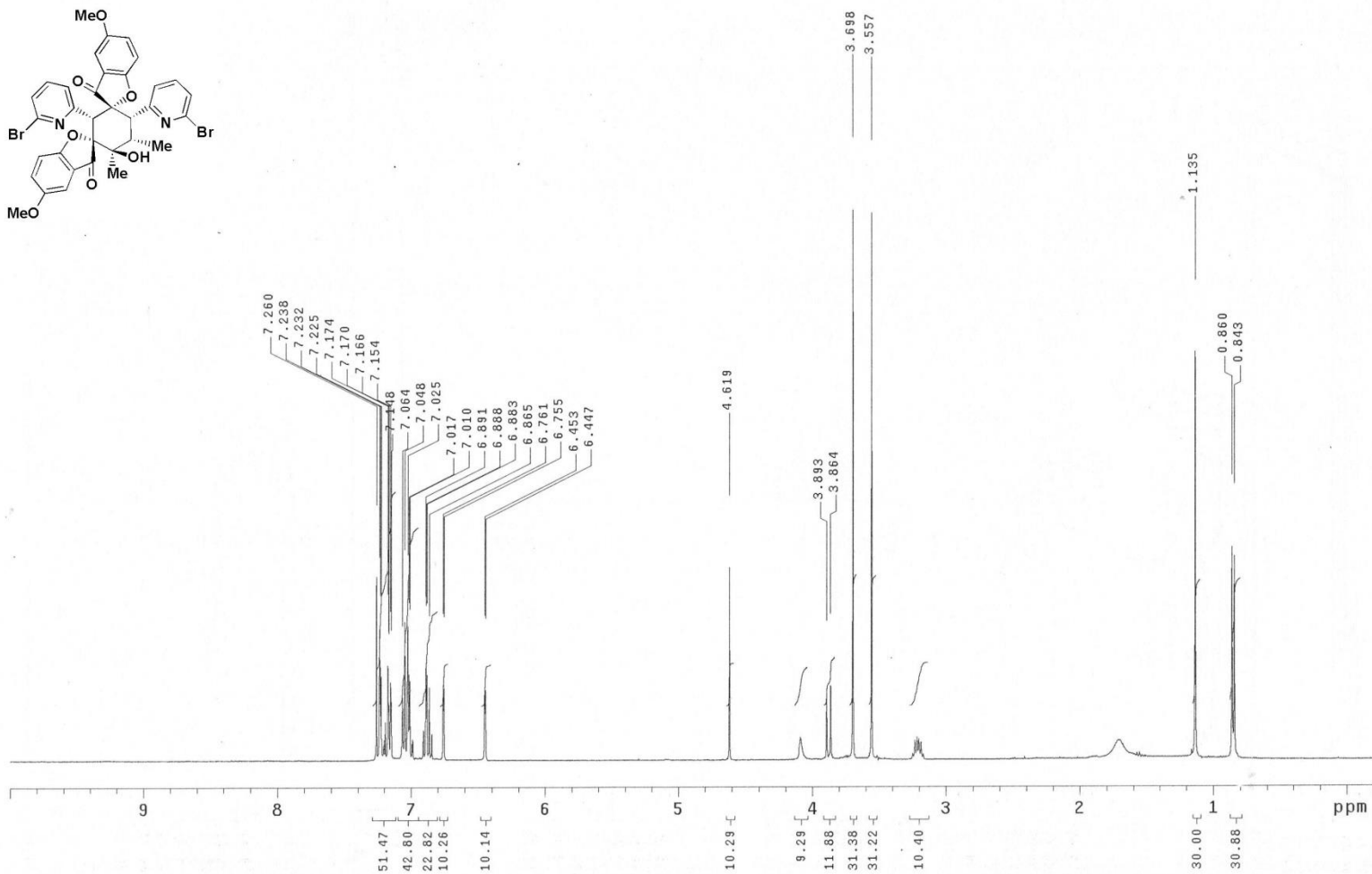
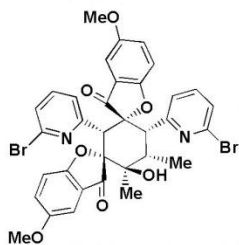




# Compound 3s (<sup>1</sup>H-NMR spectral data)

KT1080906

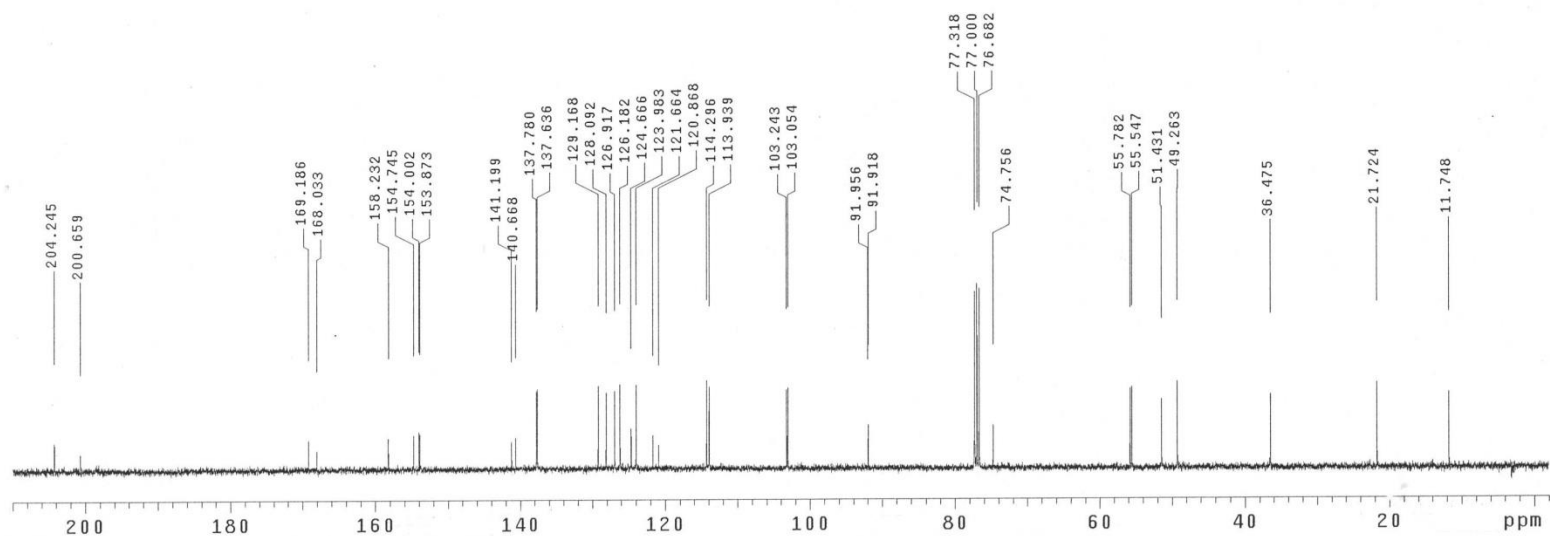
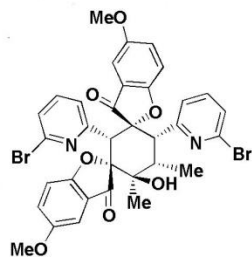
Pulse Sequence: s2pu1  
 UNITYplus-400 "unity400"  
 Date: Sep 6 2019  
 Solvent: CDCl<sub>3</sub>  
 Ambient temperature  
 Total 32 repetitions



# Compound 3s (<sup>13</sup>C-NMR spectral data)

KT1080906

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 6 2019  
Solvent: CDCl3  
Ambient temperature  
Total 4400 repetitions



# Compound 3t (<sup>1</sup>H-NMR spectral data)

KT1080912

Pulse Sequence: s2pu1

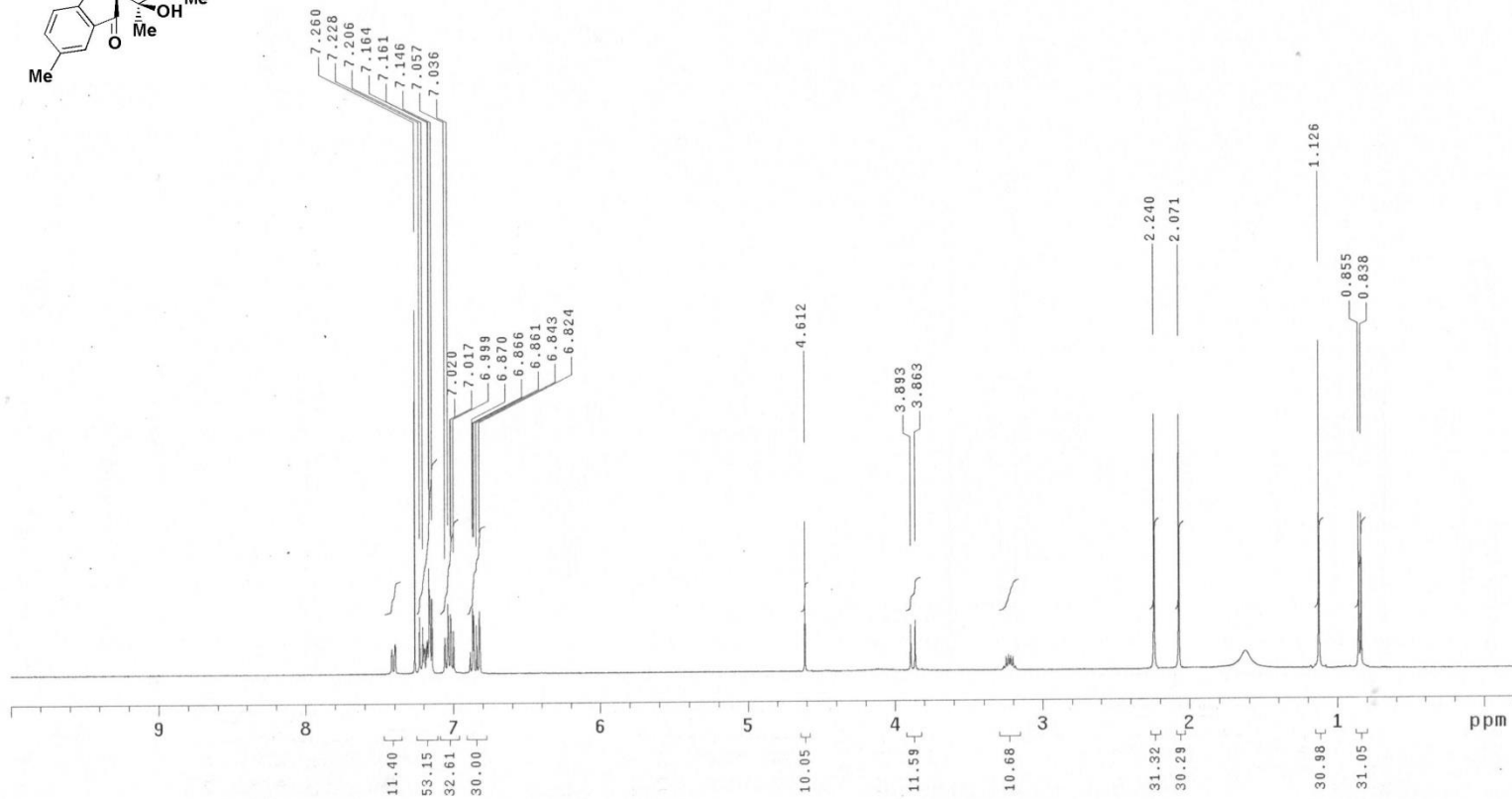
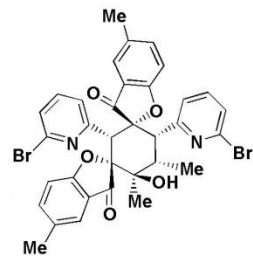
UNITYplus-400 "unity400"

Date: Sep 16 2019

Solvent: CDCl<sub>3</sub>

Ambient temperature

Total 64 repetitions

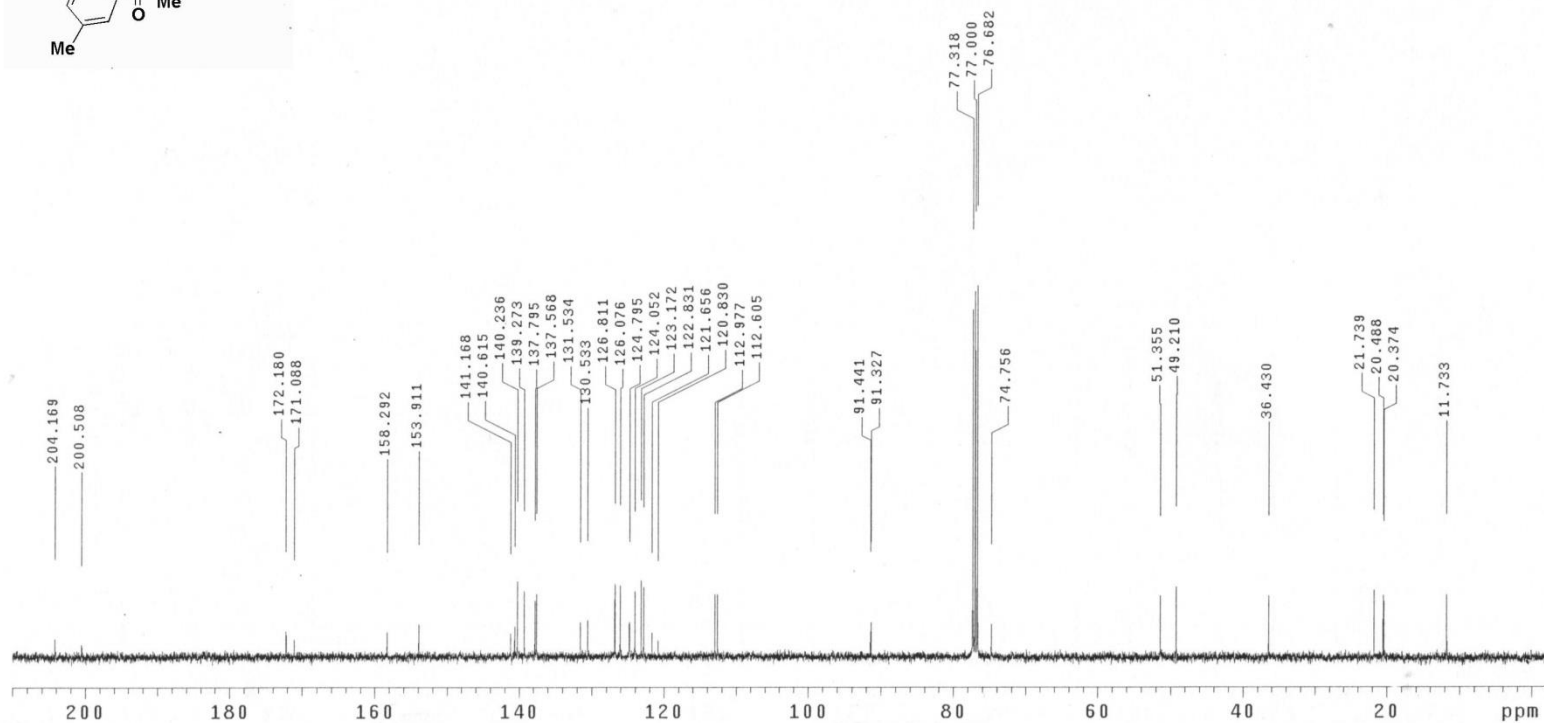
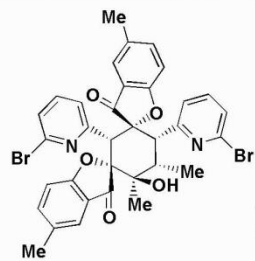




# Compound 3t (<sup>13</sup>C-NMR spectral data)

KT1080912

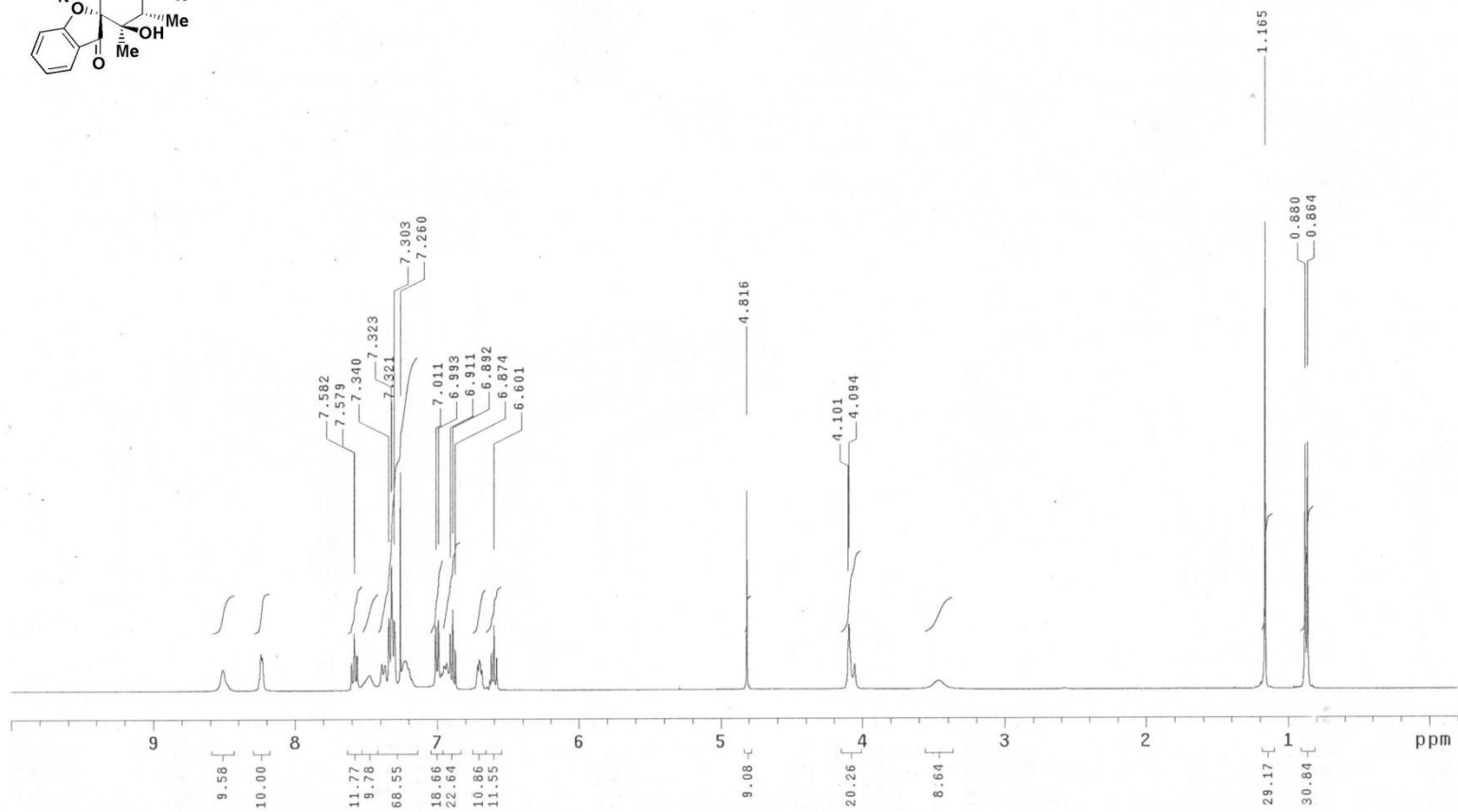
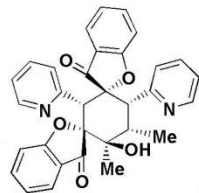
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 16 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 8640 repetitions



# Compound 3u (<sup>1</sup>H-NMR spectral data)

KT1080916

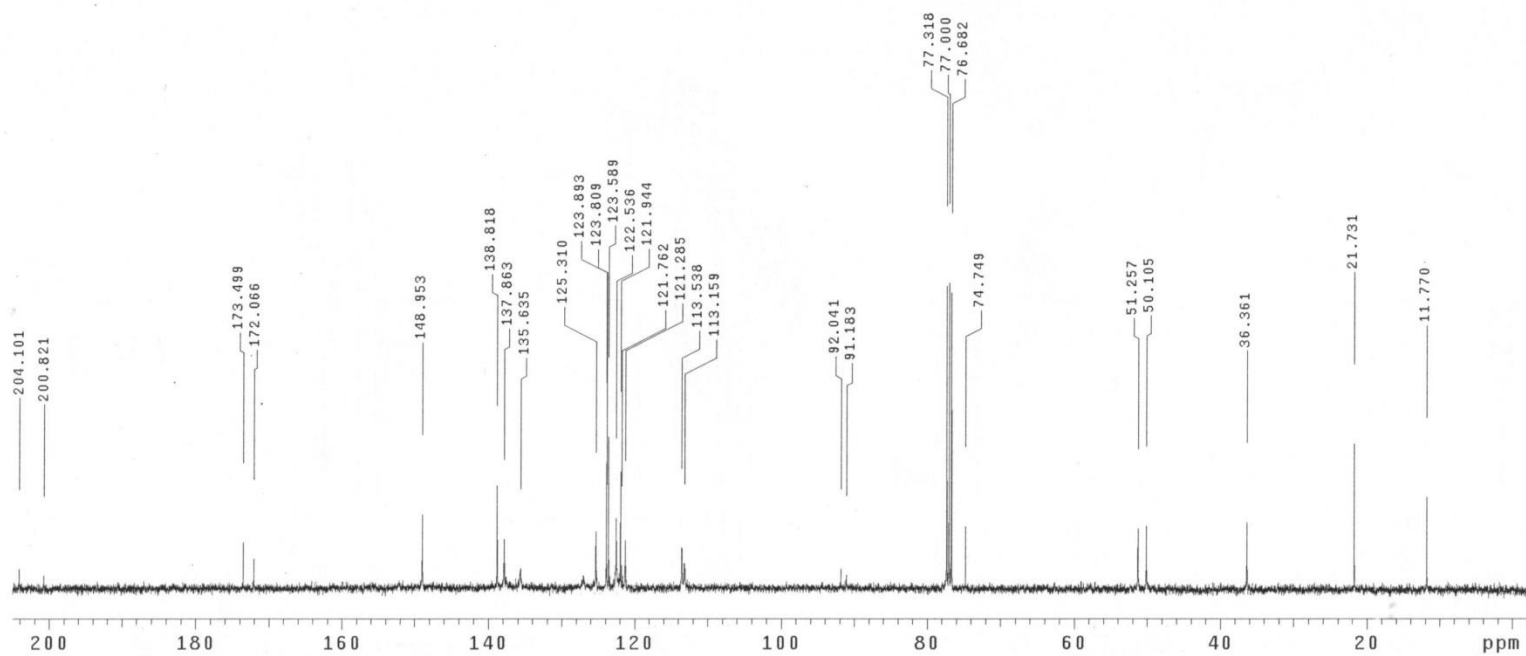
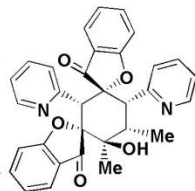
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 17 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 64 repetitions



# Compound 3u (<sup>13</sup>C-NMR spectral data)

KT1080916

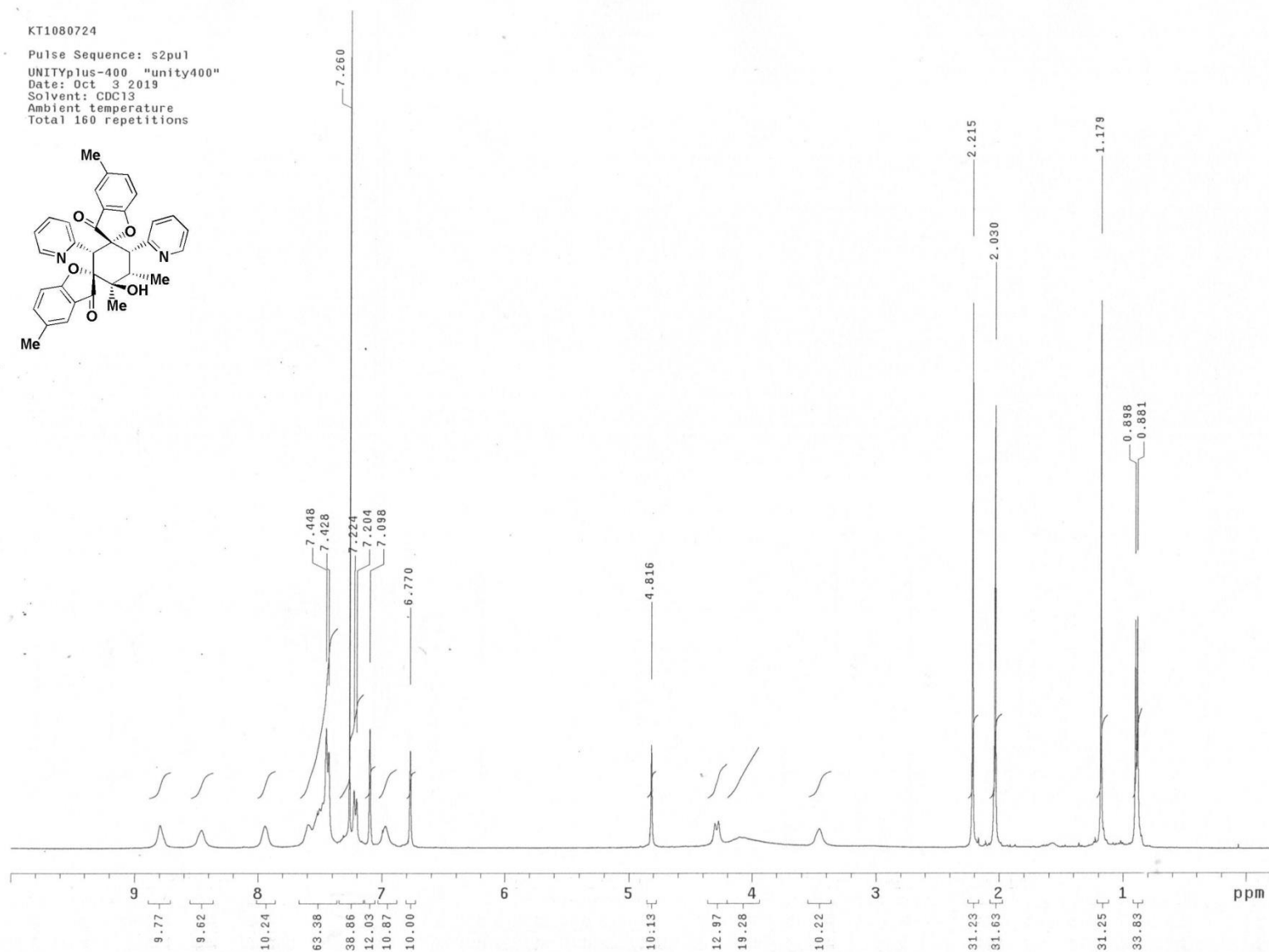
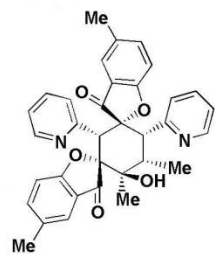
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 17 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 7024 repetitions



# Compound 3v (<sup>1</sup>H-NMR spectral data)

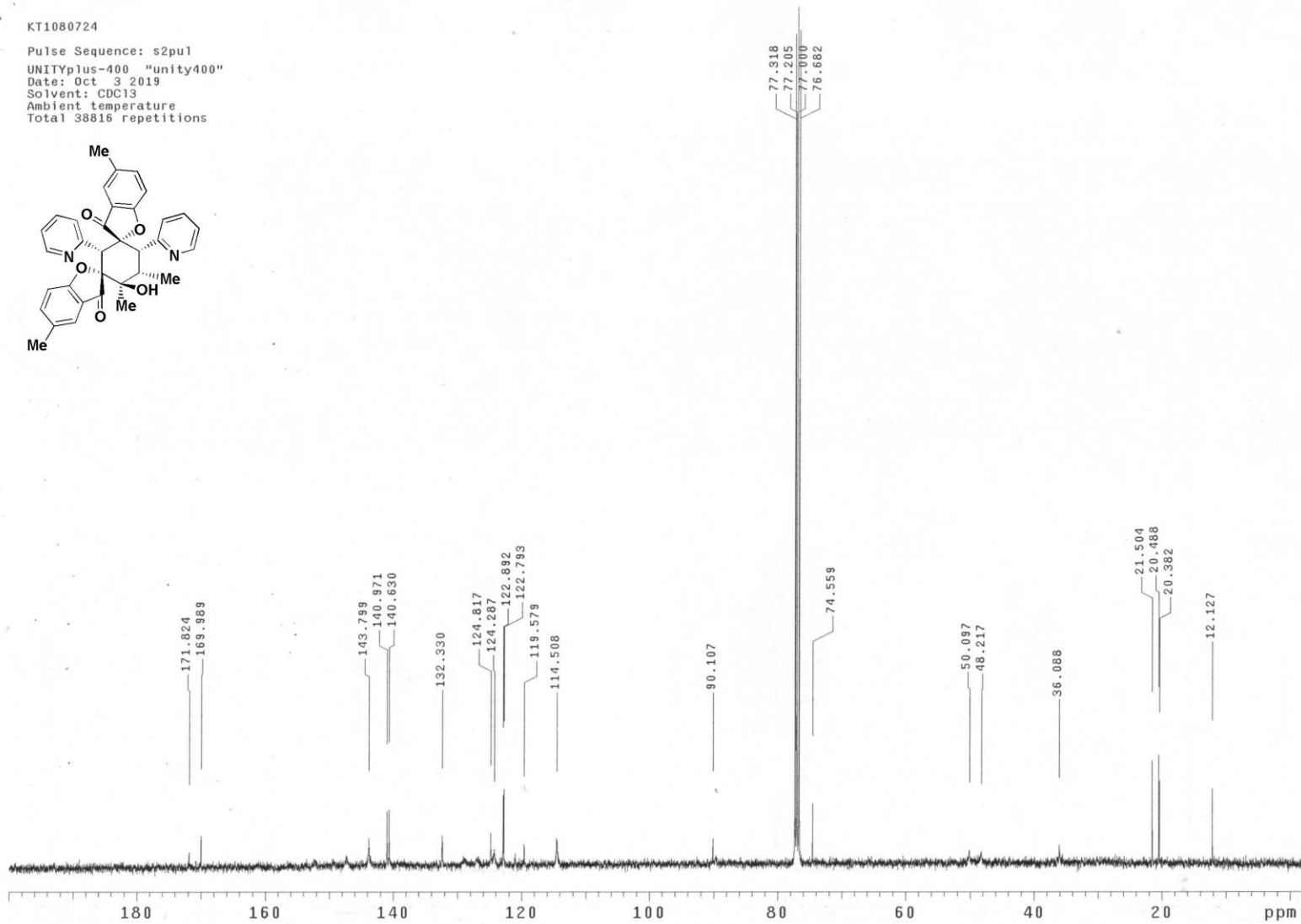
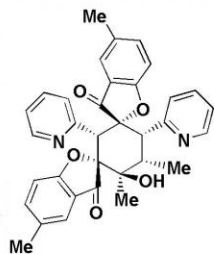
KT1080724

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 3 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 160 repetitions



# Compound 3v (<sup>13</sup>C-NMR spectral data)

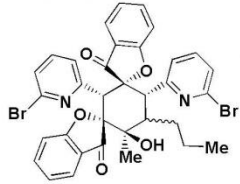
KT1080724  
Pulse Sequence: s2pul  
UNITYplus-400 "unity400"  
Date: Oct 3 2019  
Solvent: CDCl3  
Ambient temperature  
Total 38816 repetitions



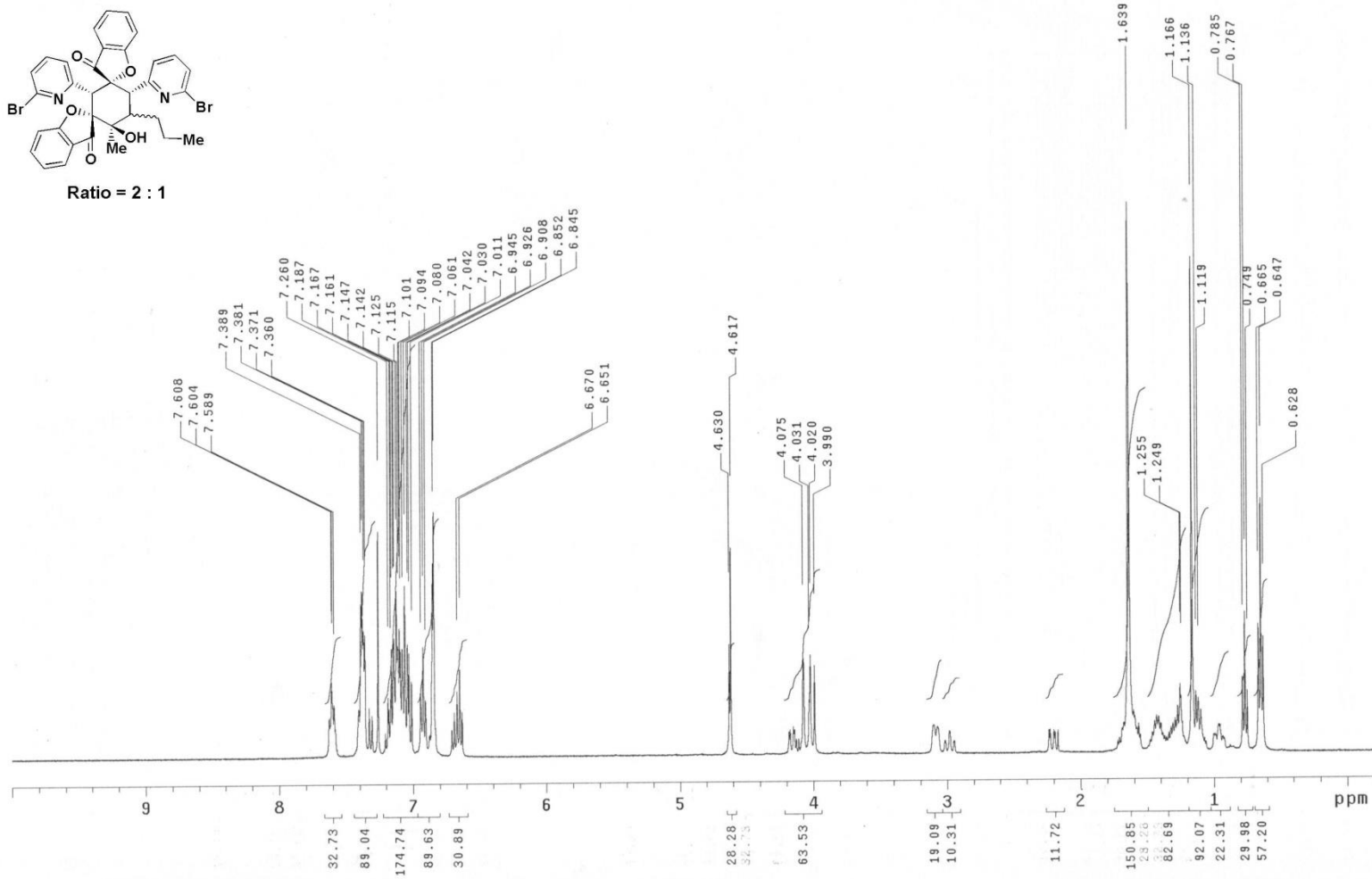
# Compound 3w (<sup>1</sup>H-NMR spectral data)

KTPPrPB

Pulse Sequence: s2pul  
 UNITYplus-400 "unity400"  
 Date: Jun 27 2019  
 Solvent: CDCl<sub>3</sub>  
 Ambient temperature  
 Total 64 repetitions



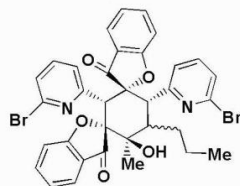
Ratio = 2 : 1



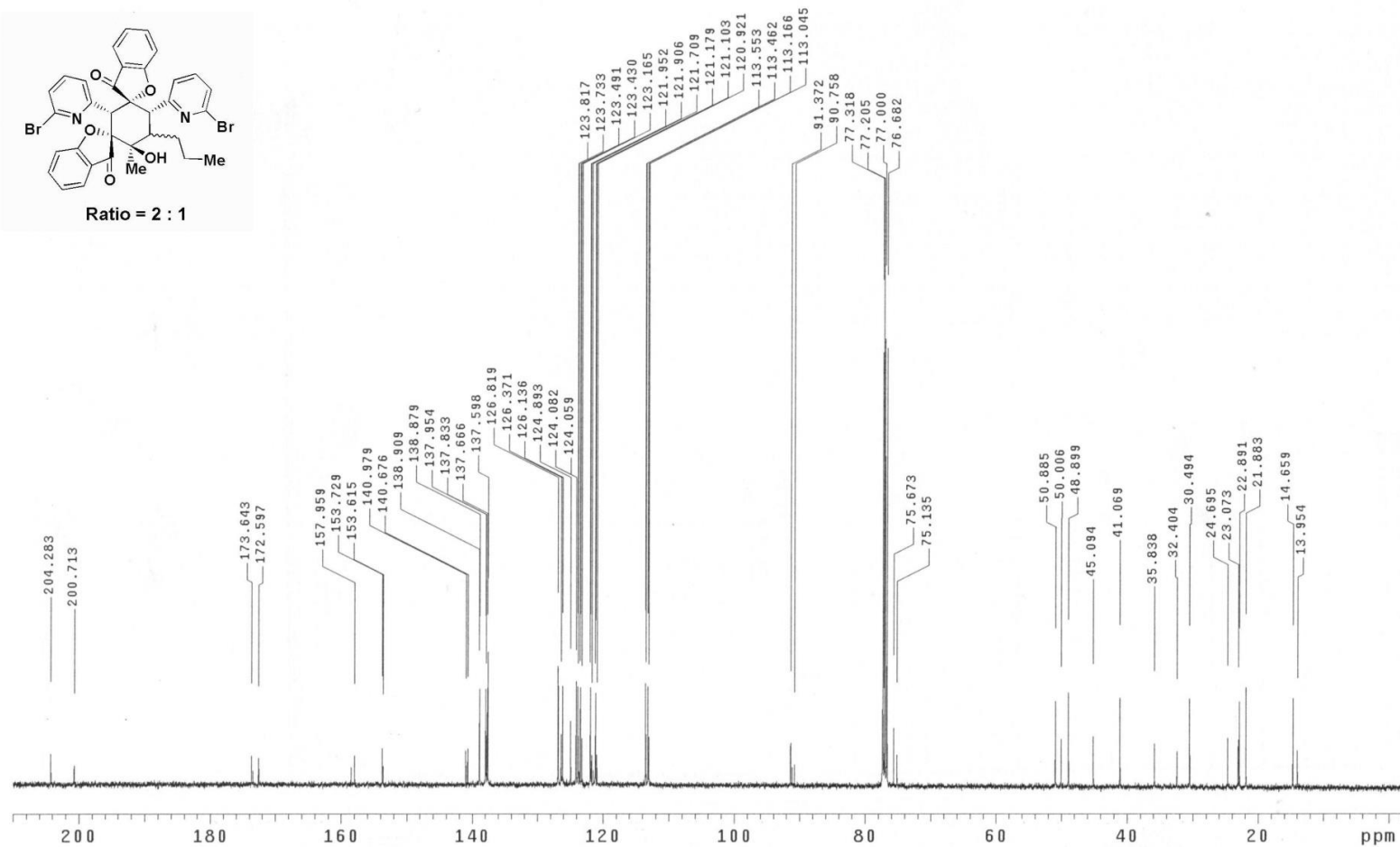
# Compound 3w (<sup>13</sup>C-NMR spectral data)

KTPPrPB

Pulse Sequence: s2pu1  
 UNITYplus-400 "unity400"  
 Date: Jun 27 2019  
 Solvent: CDCl<sub>3</sub>  
 Ambient temperature  
 Total 34336 repetitions

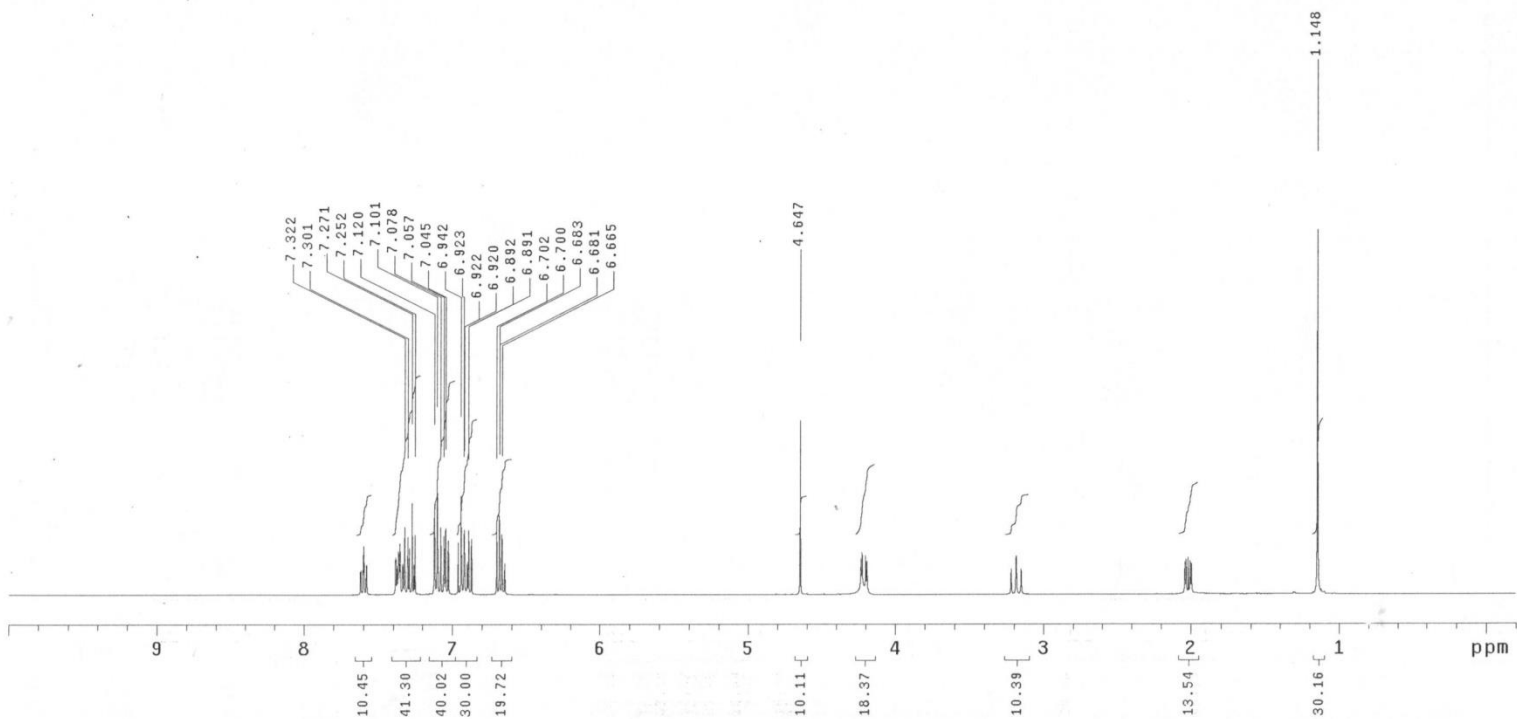
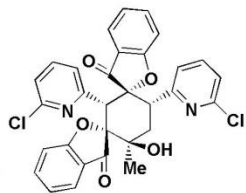


Ratio = 2 : 1



# Compound 3y (<sup>1</sup>H-NMR spectral data)

KT1081003  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 4 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions

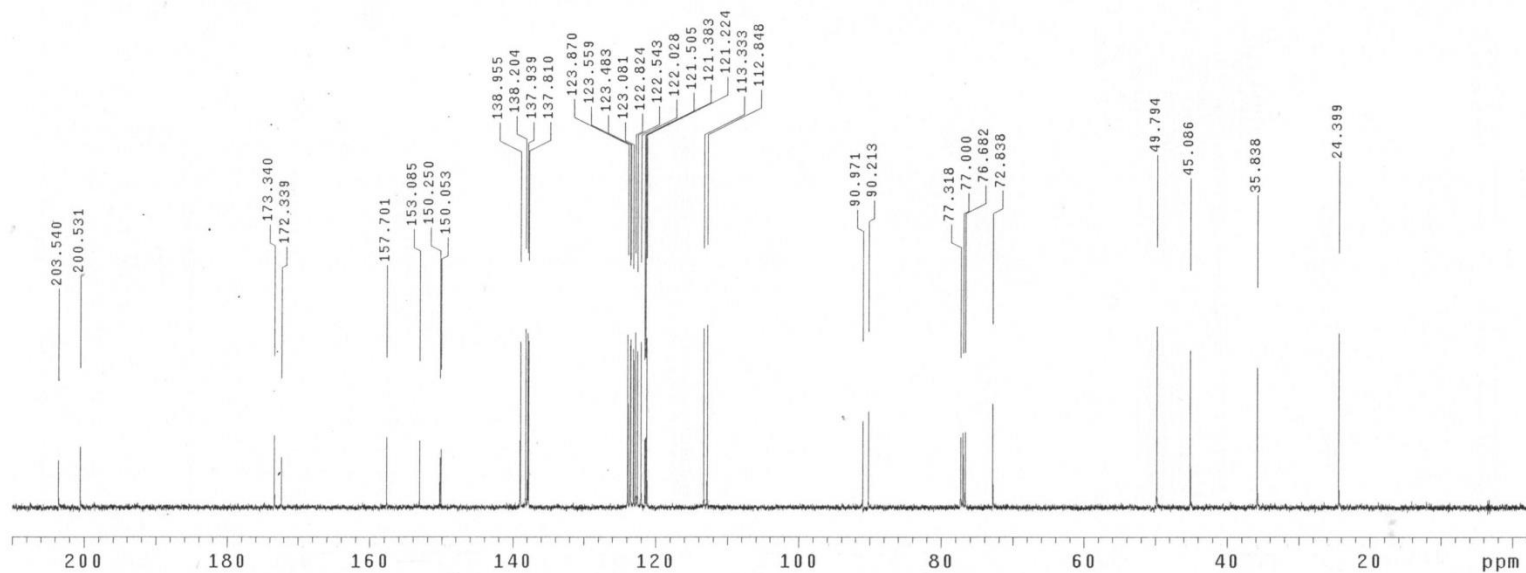
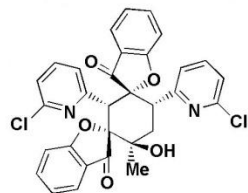




# Compound 3y (<sup>13</sup>C-NMR spectral data)

KT1081003

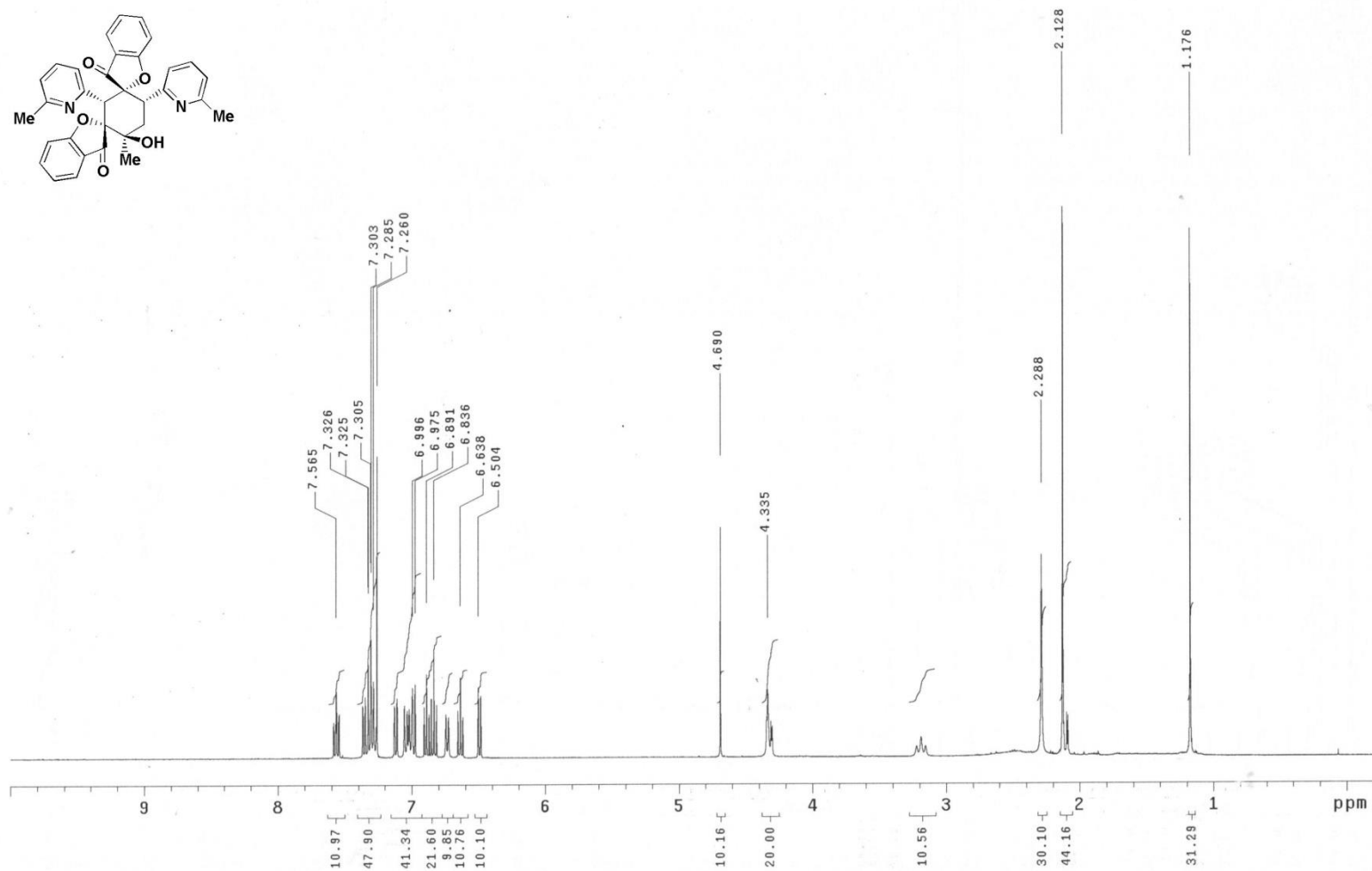
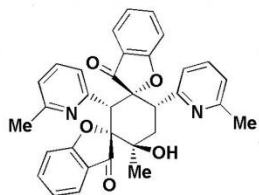
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 4 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 736 repetitions



# Compound 3z (<sup>1</sup>H-NMR spectral data)

KT1081014

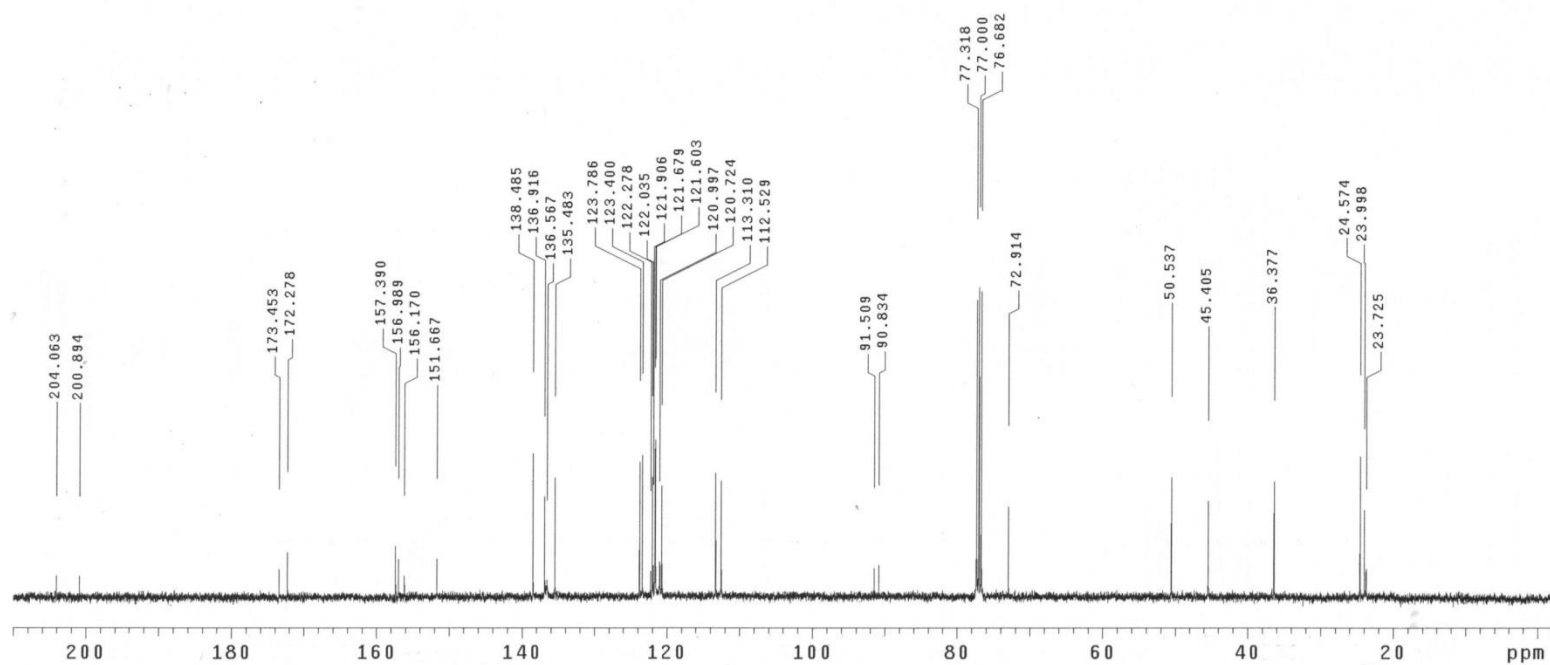
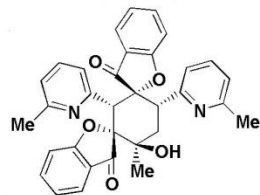
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 18 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 64 repetitions



# Compound 3z (<sup>13</sup>C-NMR spectral data)

KT1081014

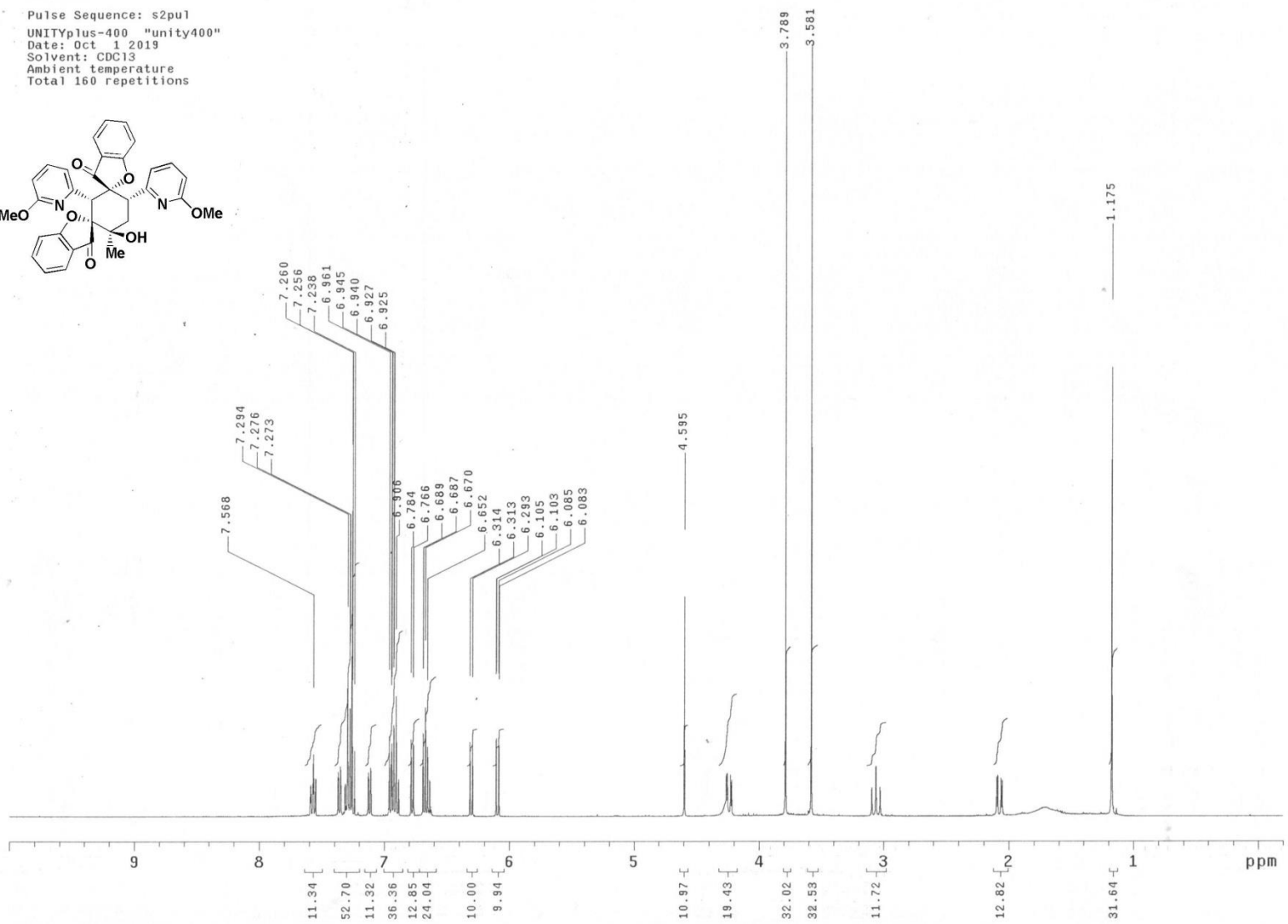
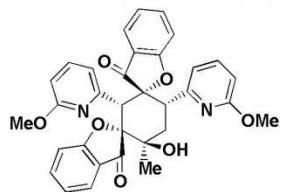
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 18 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 8016 repetitions



# Compound 3aa (<sup>1</sup>H-NMR spectral data)

KT1081001

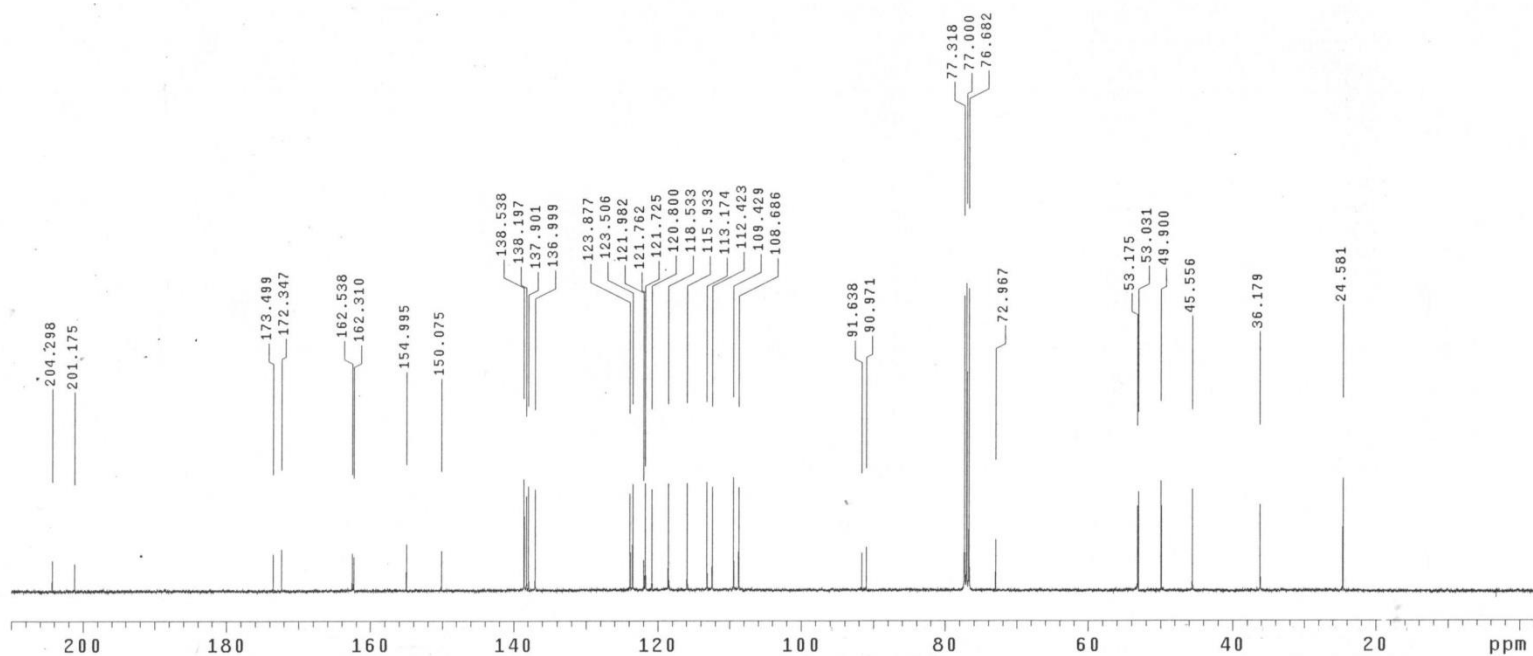
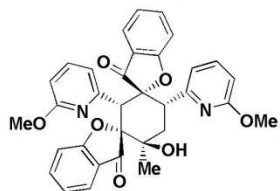
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 1 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 160 repetitions



# Compound 3aa (<sup>13</sup>C-NMR spectral data)

KT1081001

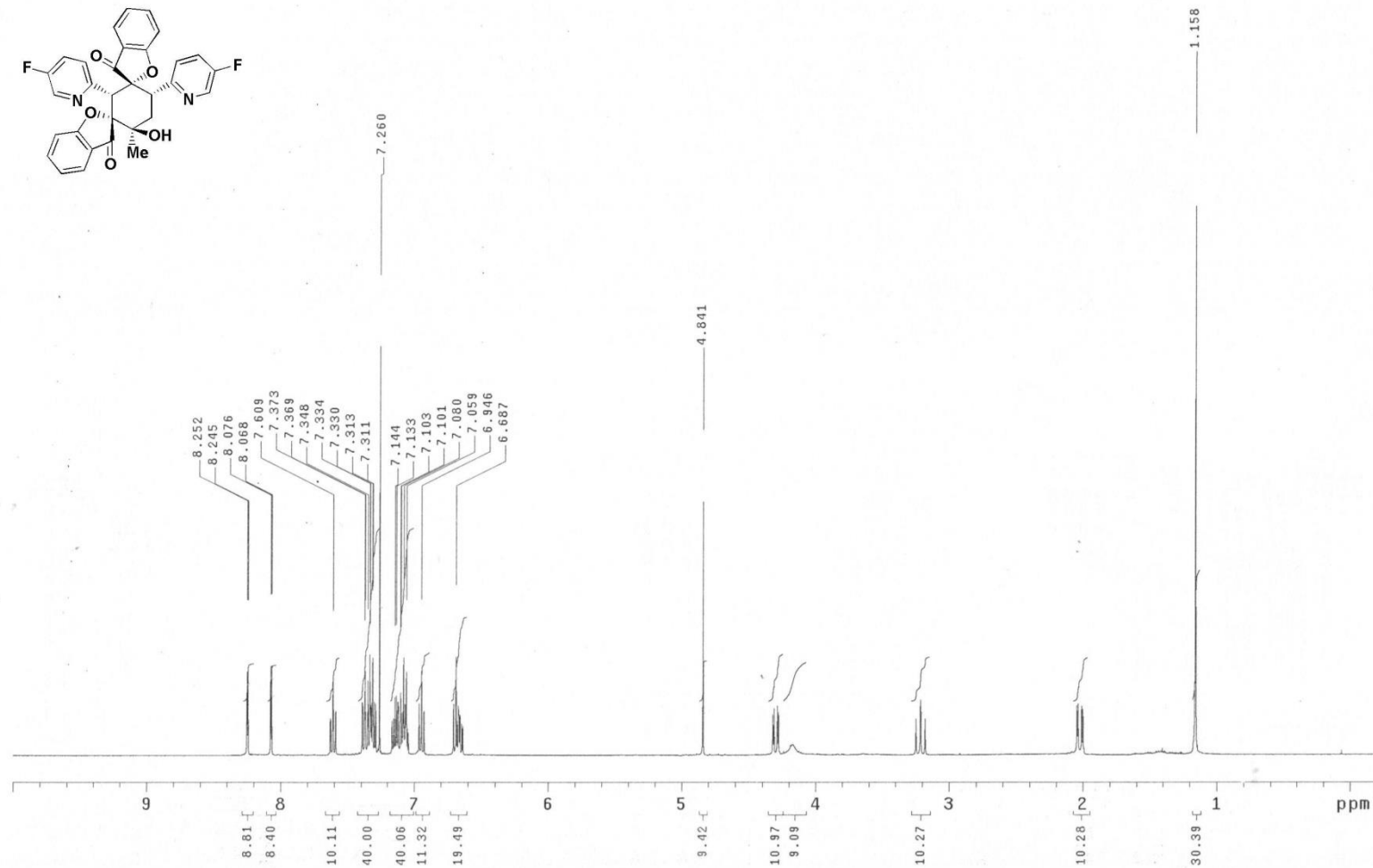
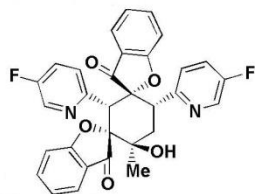
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 1 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 35680 repetitions



# Compound 3ab (<sup>1</sup>H-NMR spectral data)

KT1080927

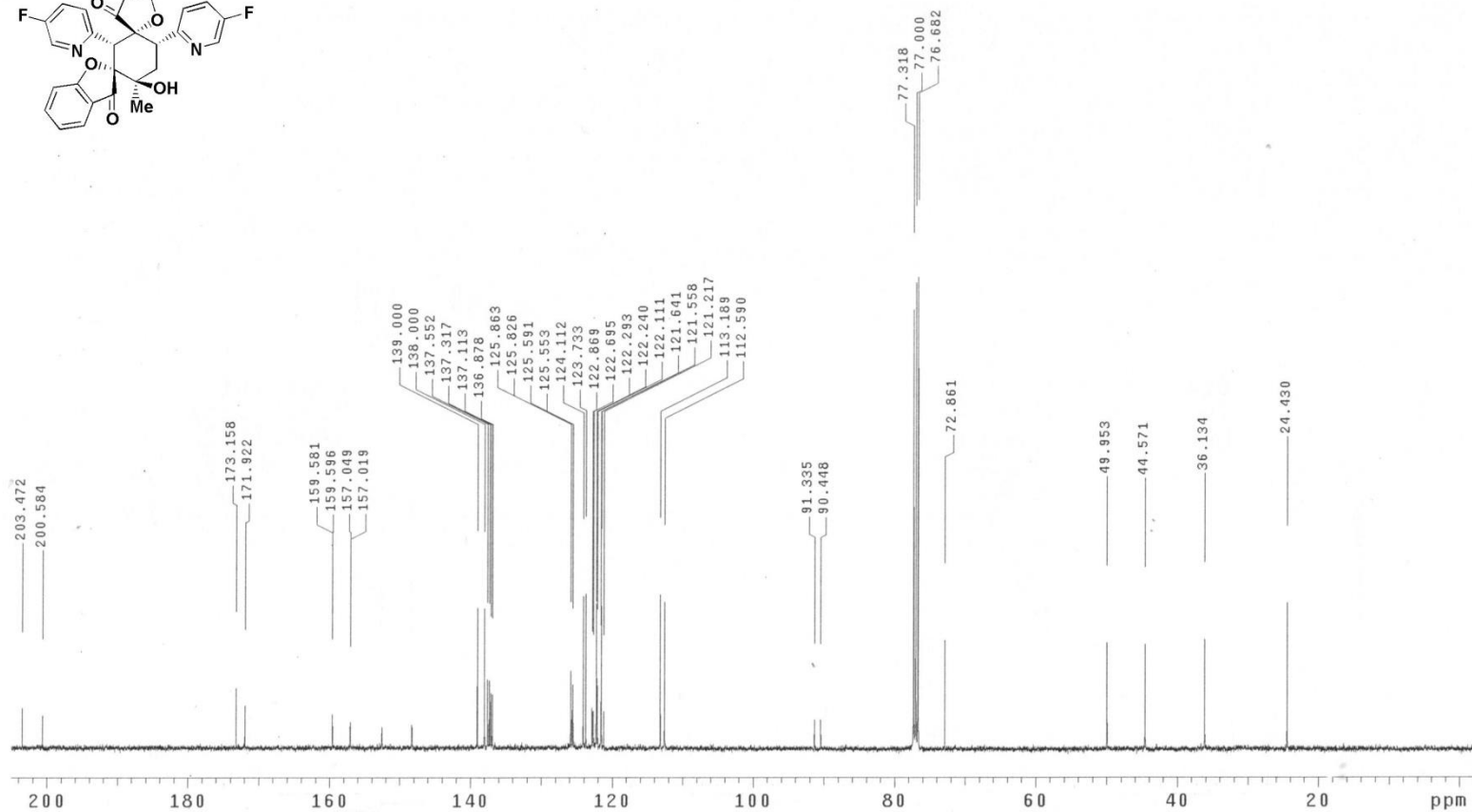
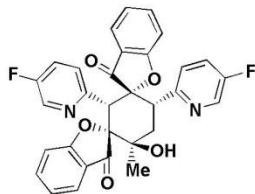
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Sep 30 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 160 repetitions



# Compound 3ab (<sup>13</sup>C-NMR spectral data)

KT1080927

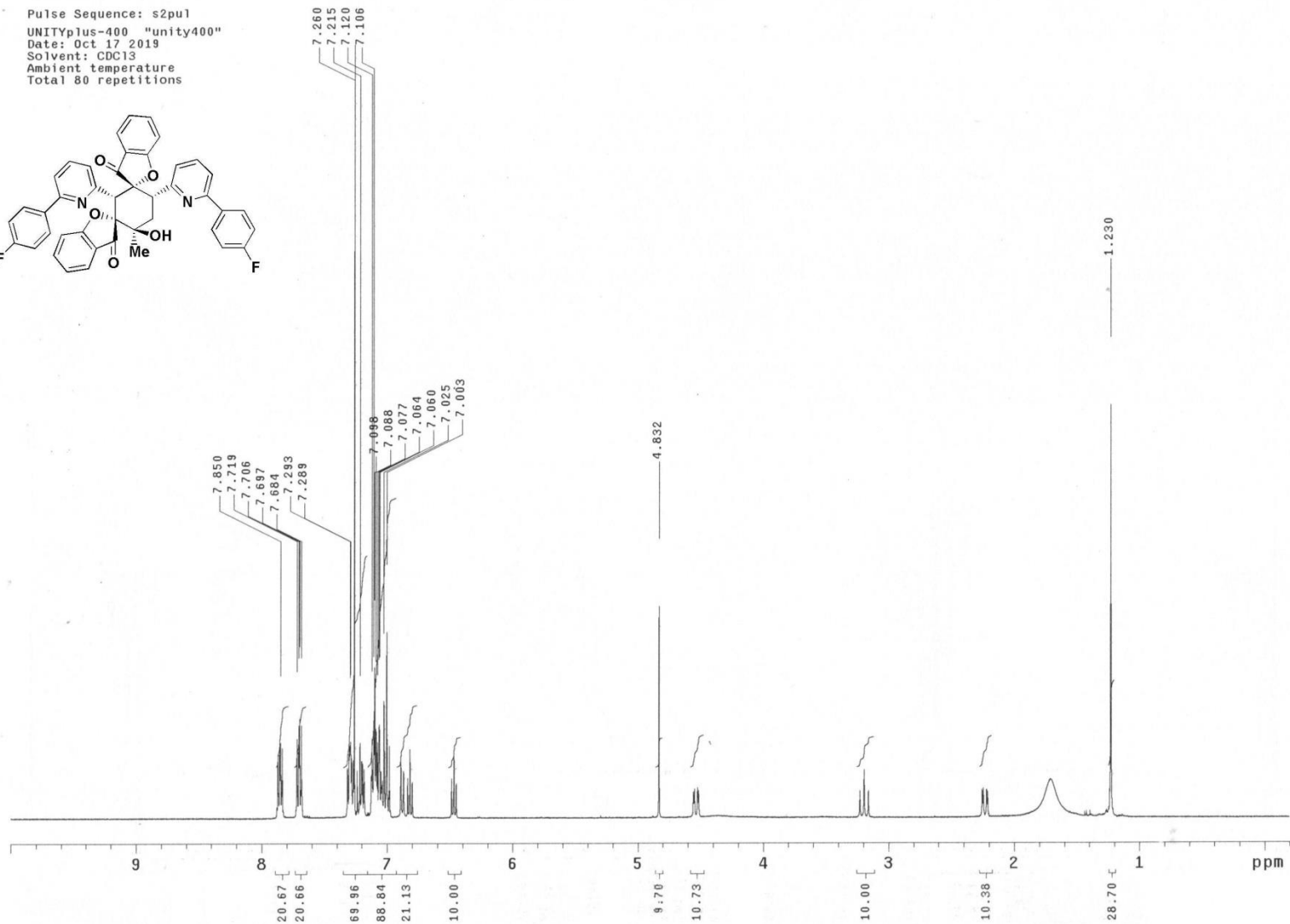
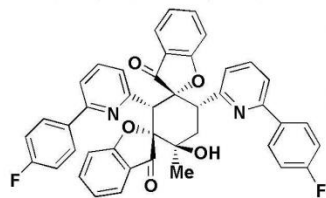
Pulse Sequence: s2pul  
UNITYplus-400 "unity400"  
Date: Sep 30 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 30912 repetitions



# Compound 3ac (<sup>1</sup>H-NMR spectral data)

KT1081016

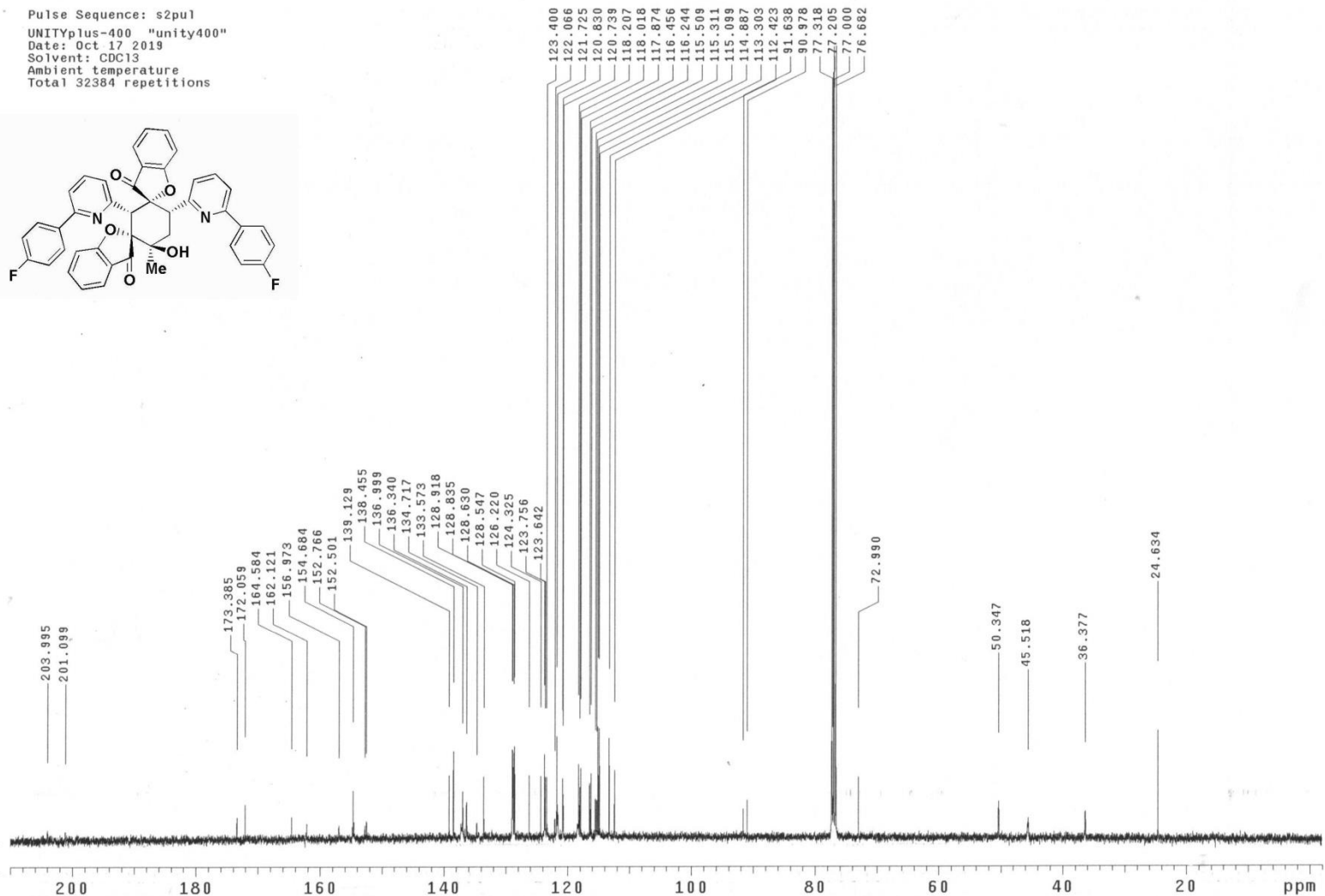
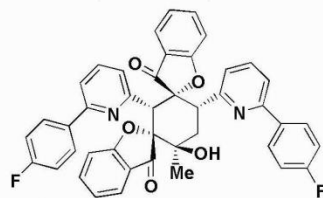
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 17 2019  
Solvent: CDCl3  
Ambient temperature  
Total 80 repetitions





# Compound 3ac (<sup>13</sup>C-NMR spectral data)

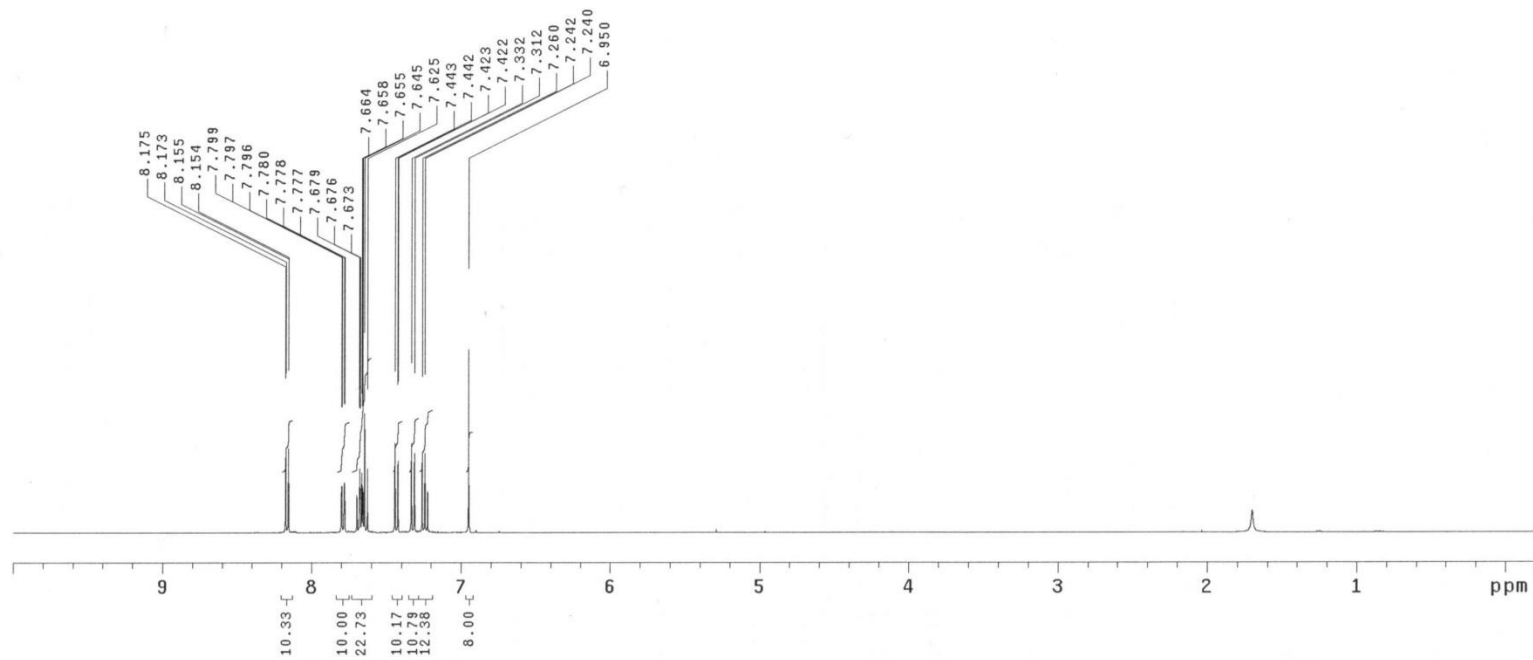
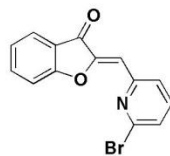
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 17 2019  
Solvent: CDC13  
Ambient temperature  
Total 32384 repetitions



# Compound 4a (<sup>1</sup>H-NMR spectral data)

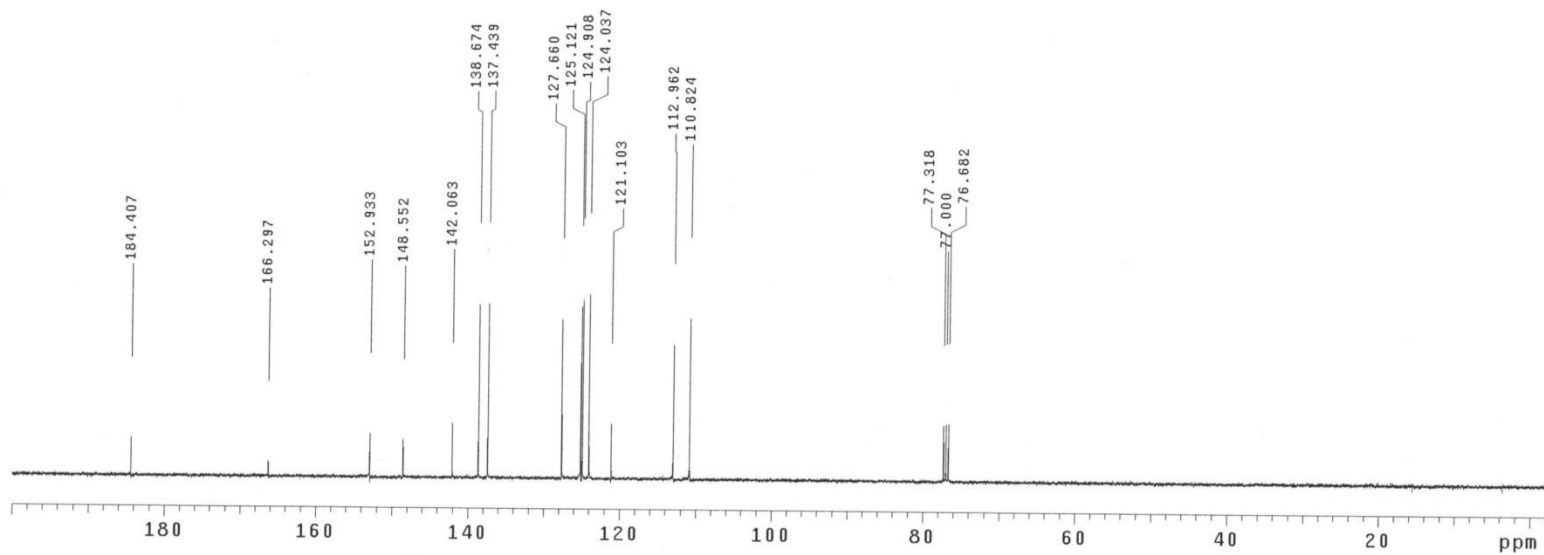
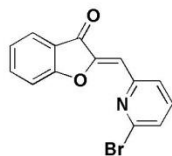
HANBr25

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 22 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



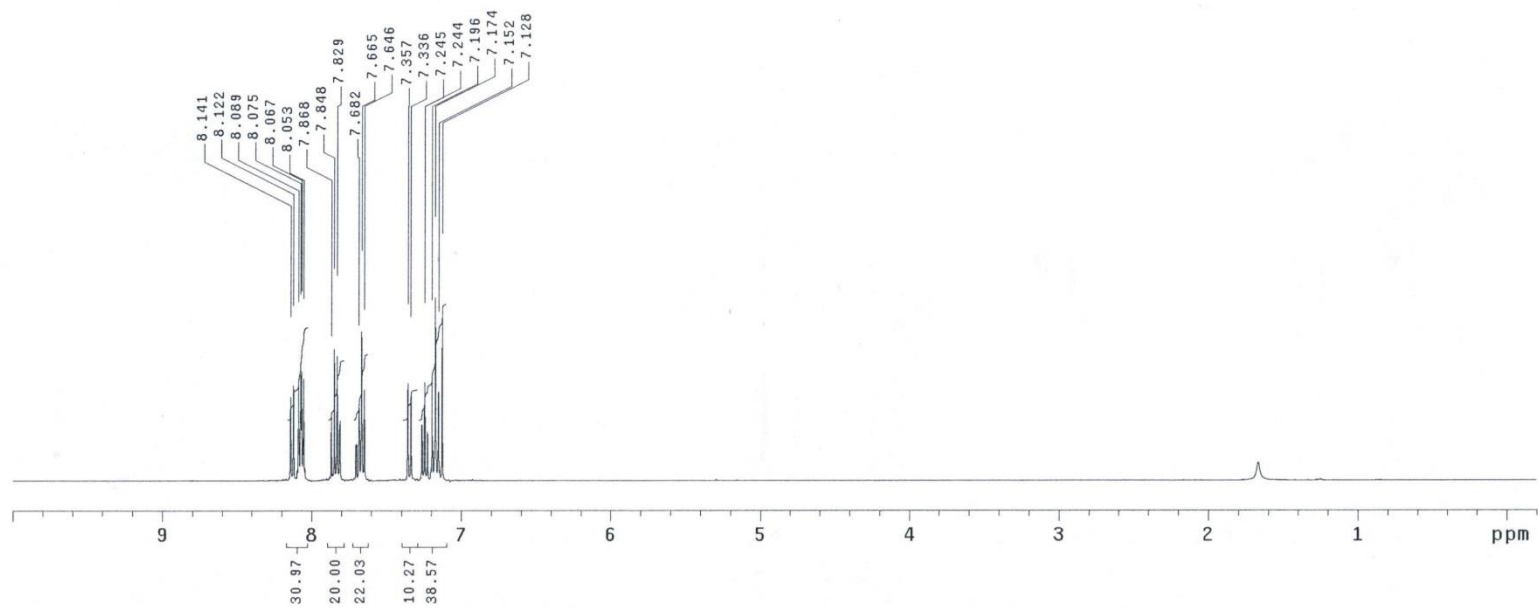
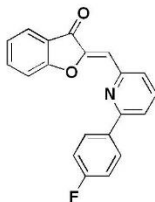
# Compound 4a (<sup>13</sup>C-NMR spectral data)

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Jan 22 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2800 repetitions



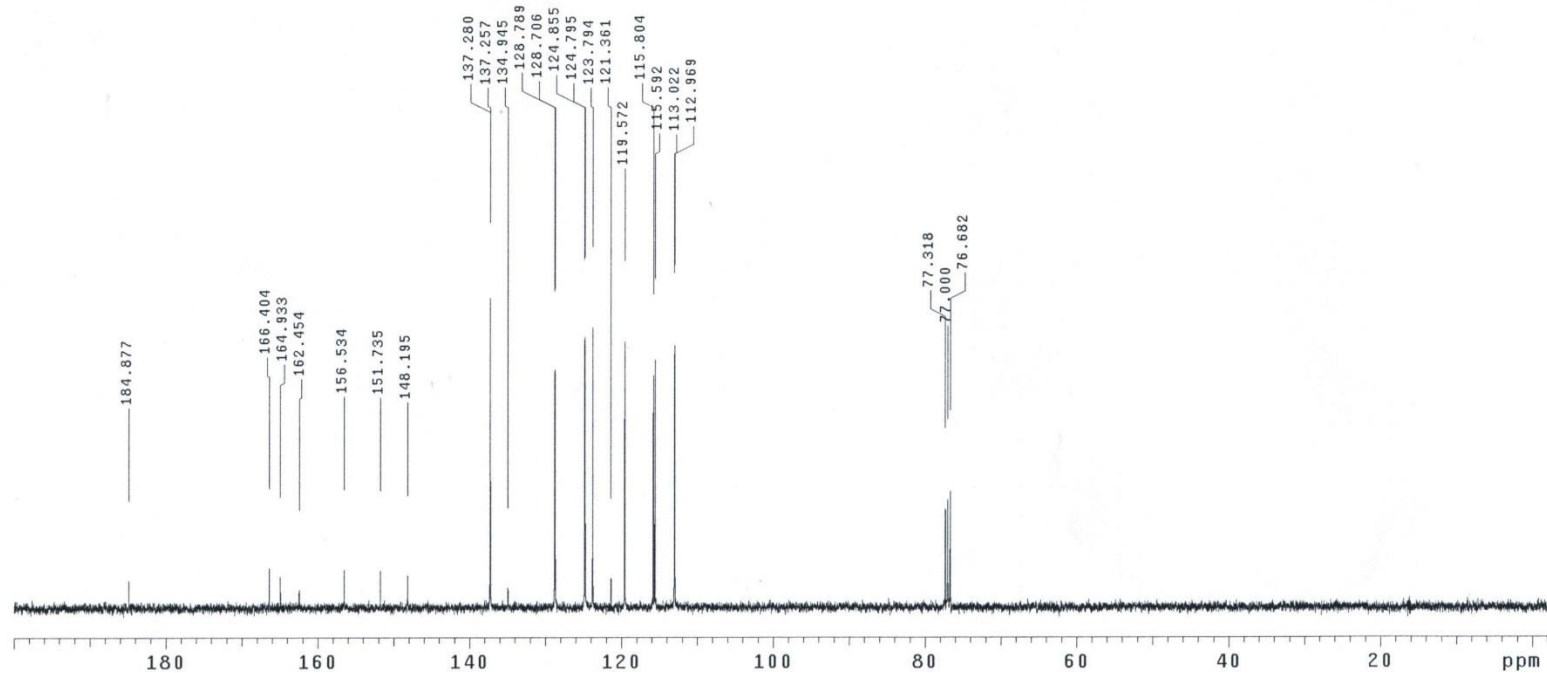
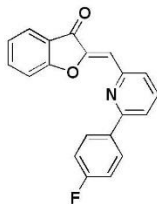
# Compound 4ac (<sup>1</sup>H-NMR spectral data)

HANFPh30  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Mar 12 2018  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 4ac (<sup>13</sup>C-NMR spectral data)

HANFPh30  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Mar 12 2018  
Solvent: CDC13  
Ambient temperature  
Total 3616 repetitions



# Compound 5a (<sup>1</sup>H-NMR spectral data)

HAPh5h

Pulse Sequence: s2pu1

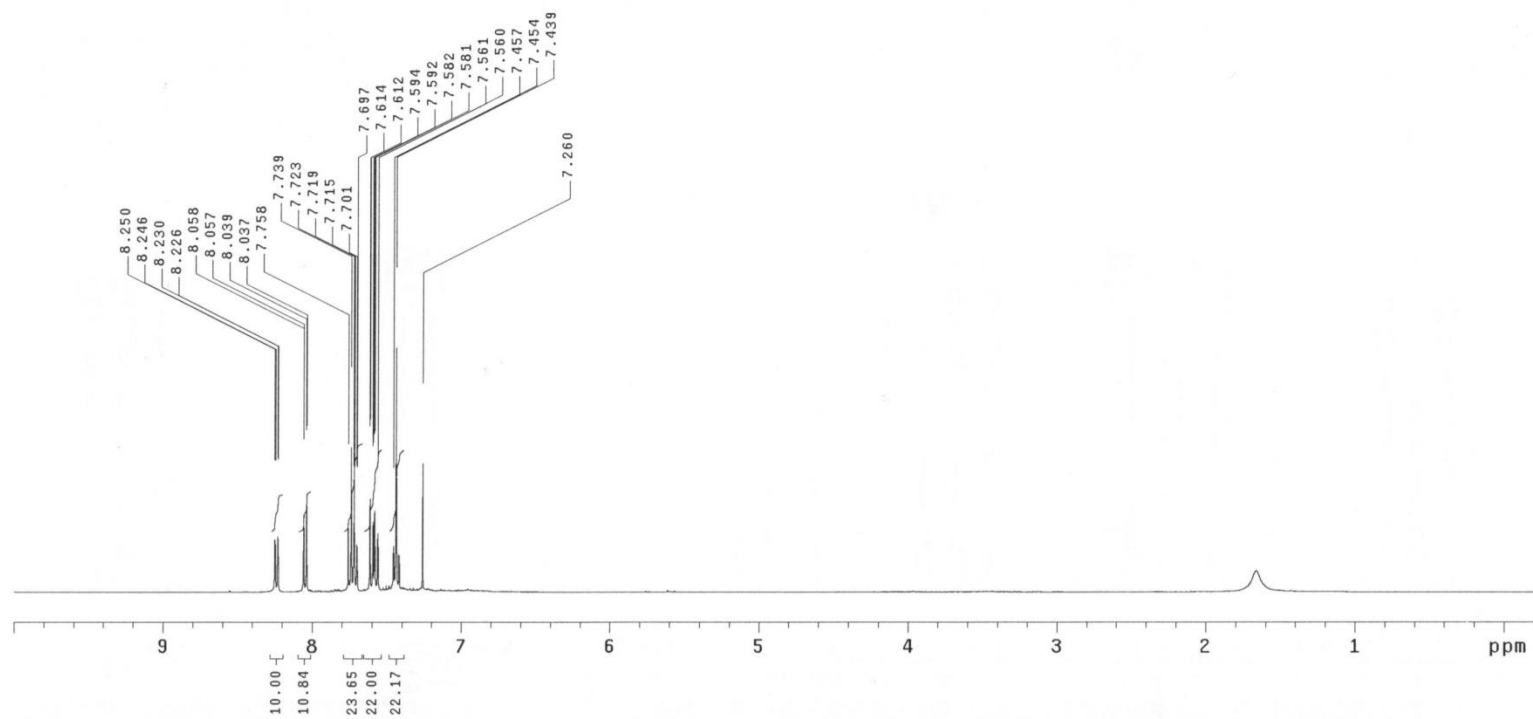
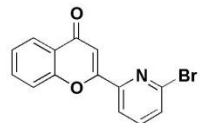
UNITYplus-400 "unity400"

Date: Aug 8 2018

Solvent: CDC13

Ambient temperature

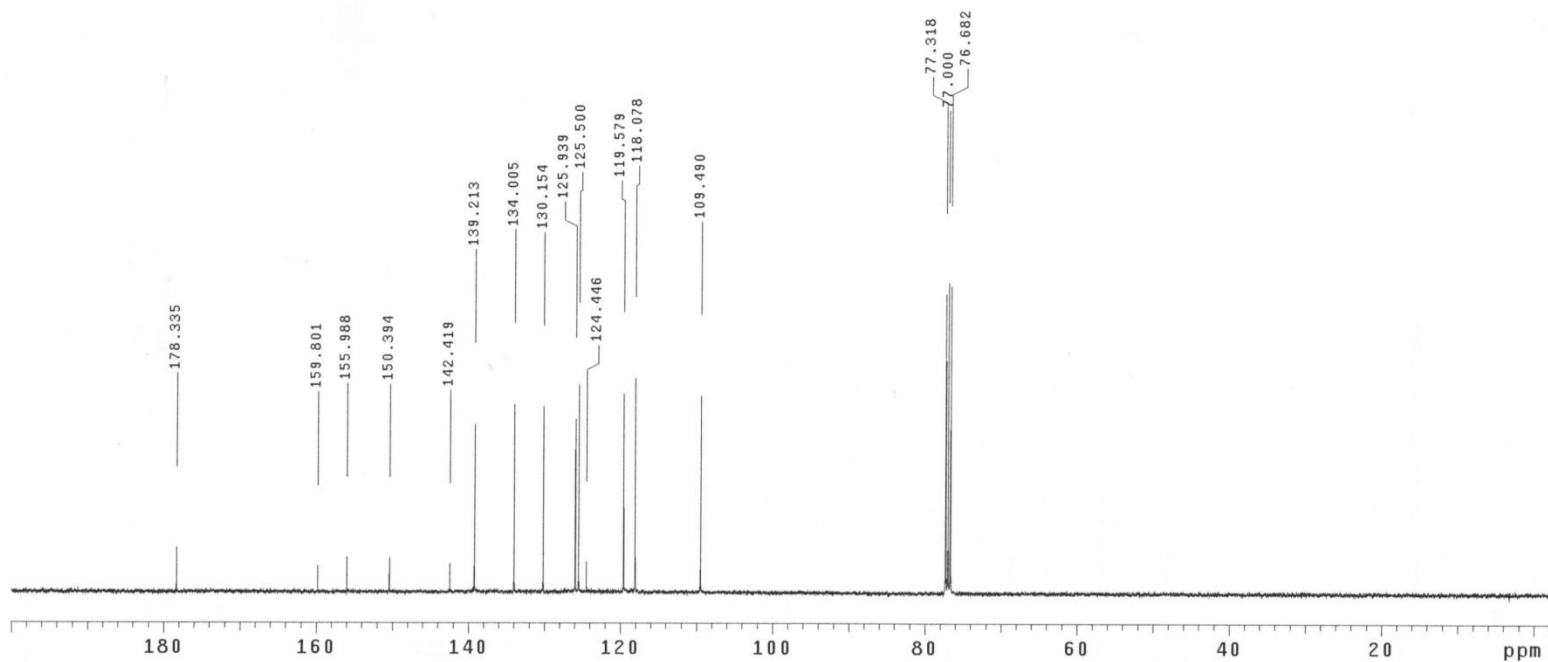
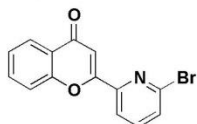
Total 64 repetitions



# Compound 5a (<sup>13</sup>C-NMR spectral data)

HAPh5h

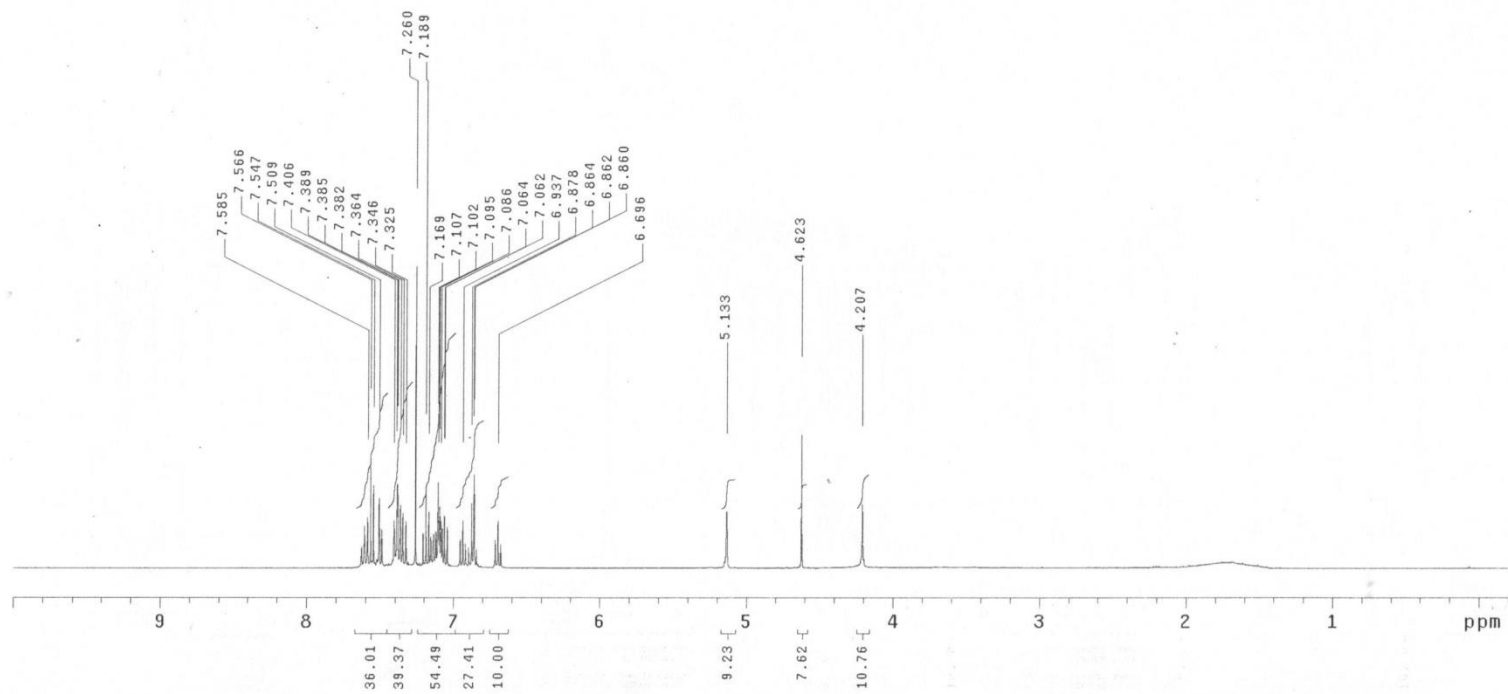
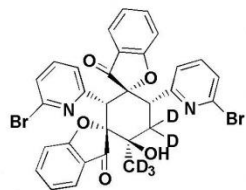
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Aug 8 2018  
Solvent: CDCl3  
Ambient temperature  
Total 16000 repetitions



# Compound 6 (<sup>1</sup>H-NMR spectral data-400 MHz)

KT1080930

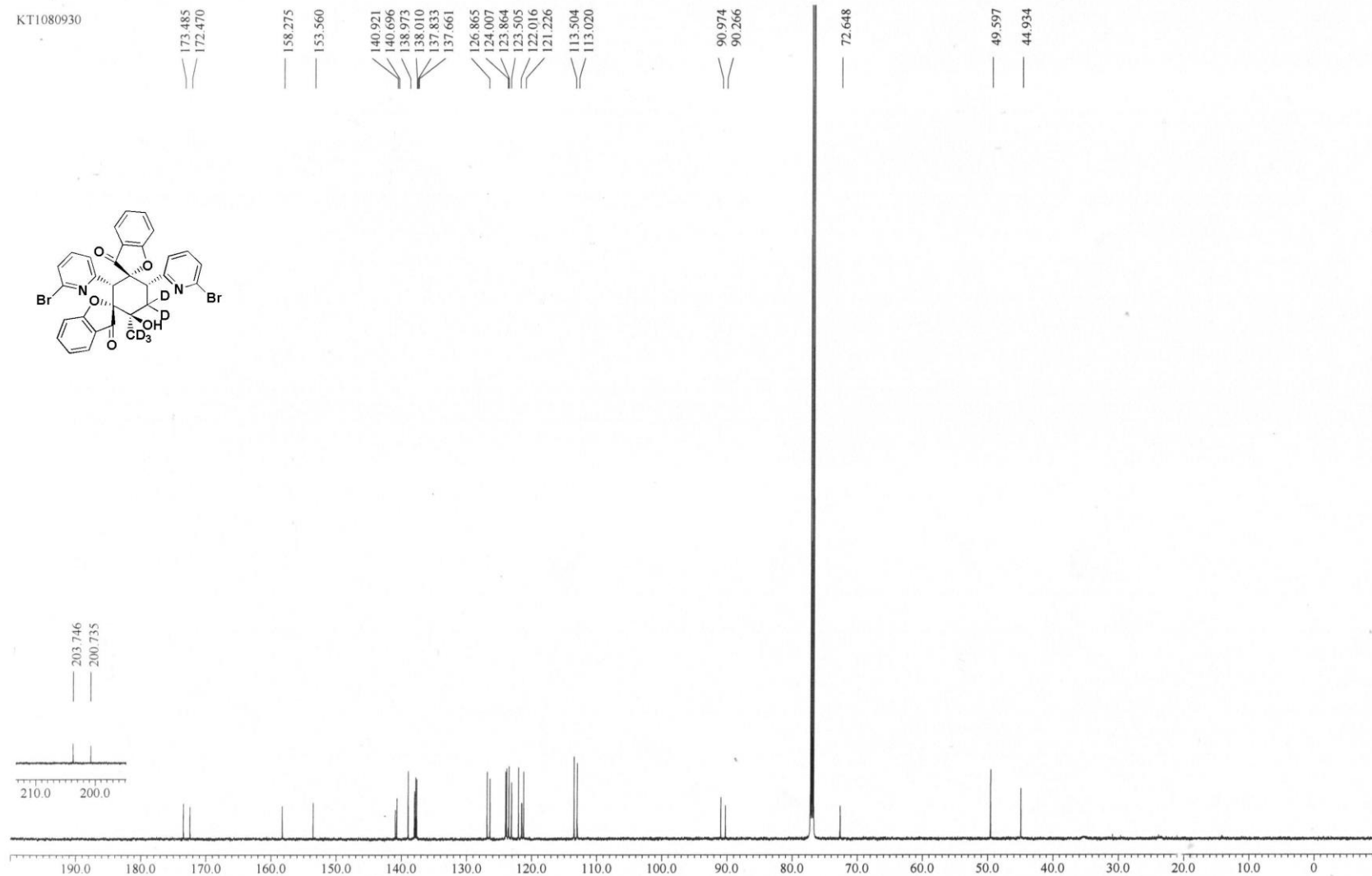
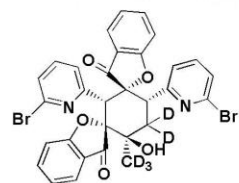
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 1 2019  
Solvent: CDC13  
Ambient temperature  
Total 32 repetitions





# Compound 6 (<sup>13</sup>C-NMR spectral data-1)

KT1080930



# Compound 6 (<sup>13</sup>C-NMR spectral data-2)

KT1080930

173.485  
172.470

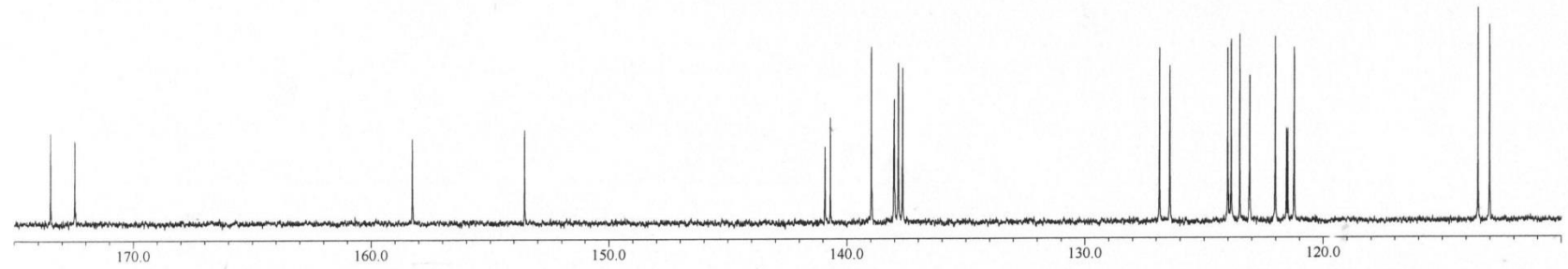
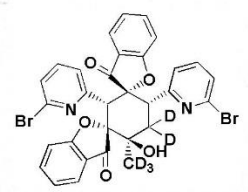
158.275

153.560

140.921  
140.696  
138.973  
138.010  
137.833  
137.661

126.865  
126.430  
124.007  
123.864  
123.505  
123.088  
122.016  
121.551  
121.494  
121.226

113.504  
113.020



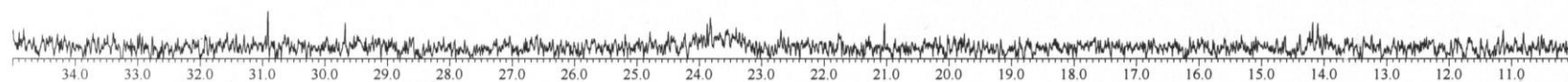
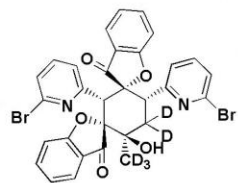
# Compound 6 (<sup>13</sup>C-NMR spectral data-3)

KT1080930

30.917

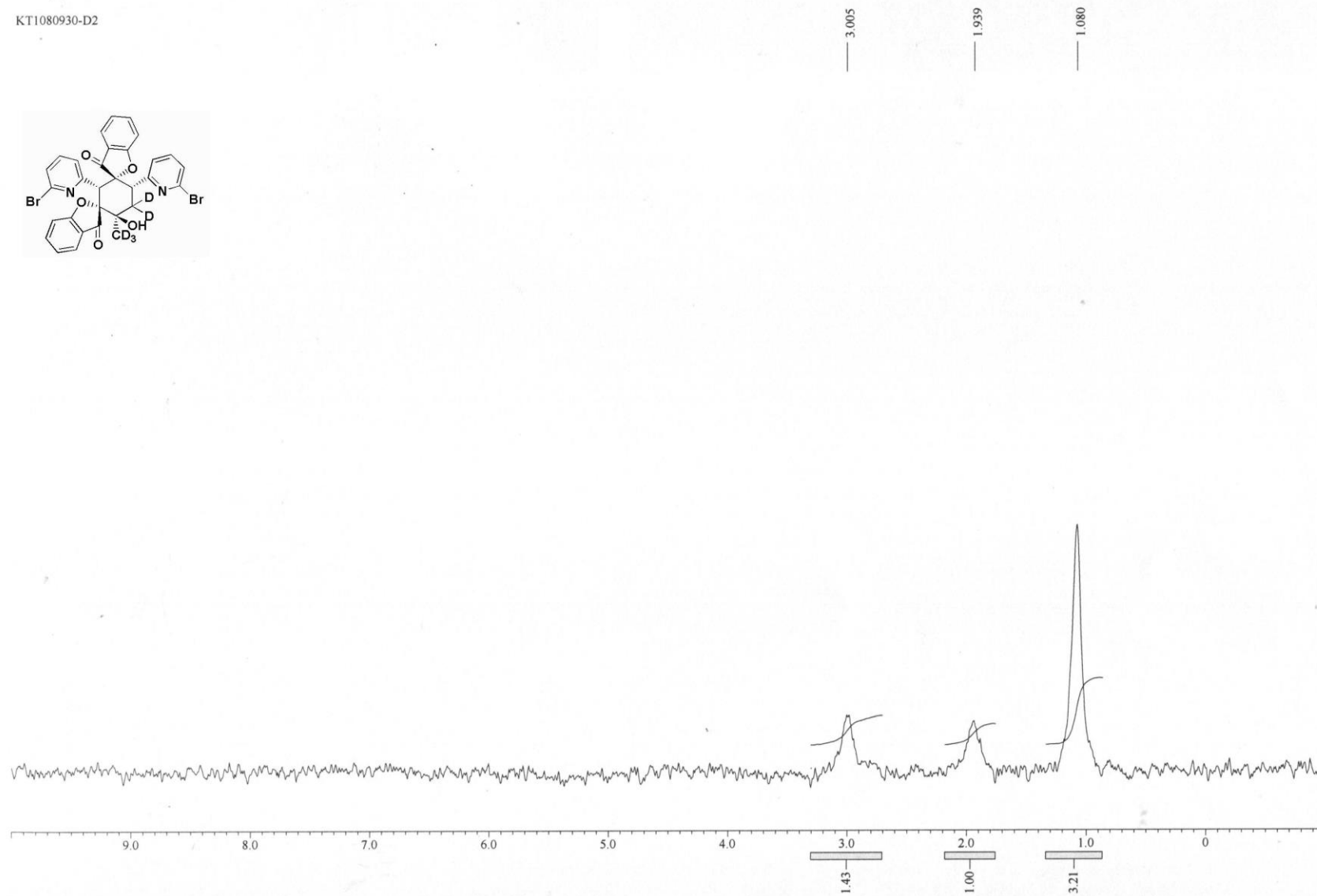
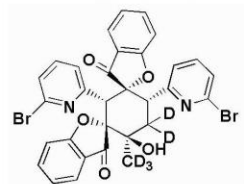
23.827

14.185  
14.103



# Compound 6 (<sup>2</sup>H-NMR spectral data)

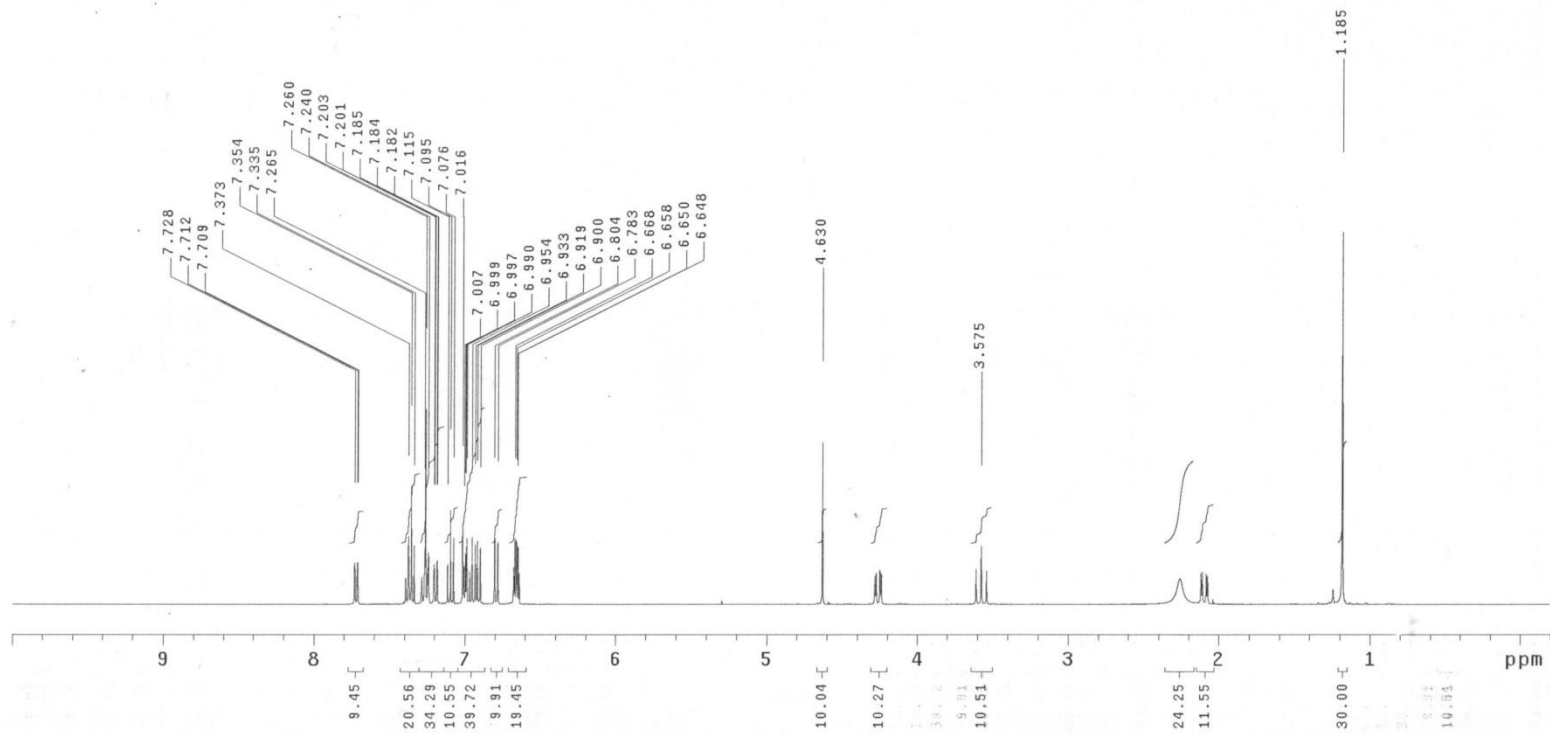
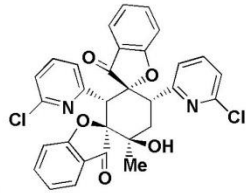
KT1080930-D2



# Compound 3y-1 (<sup>1</sup>H-NMR spectral data)

KT6PC93

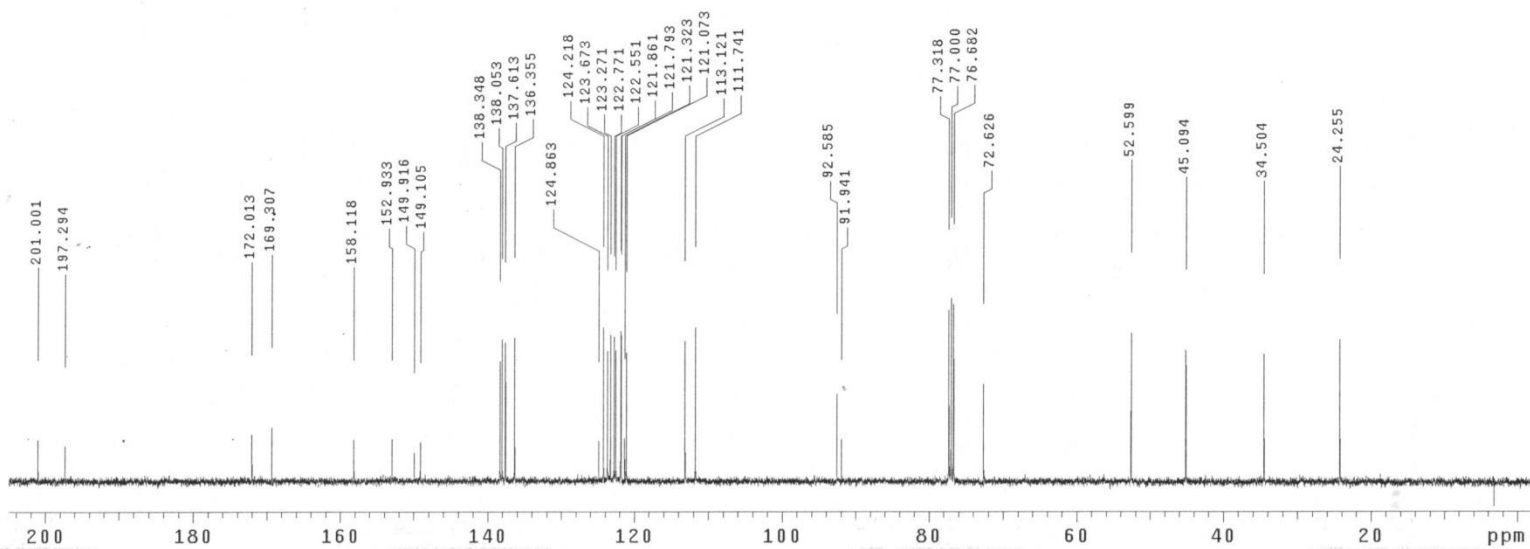
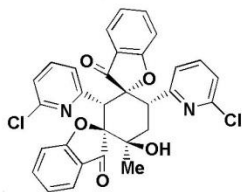
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 7 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 32 repetitions



# Compound 3y-1 (<sup>13</sup>C-NMR spectral data)

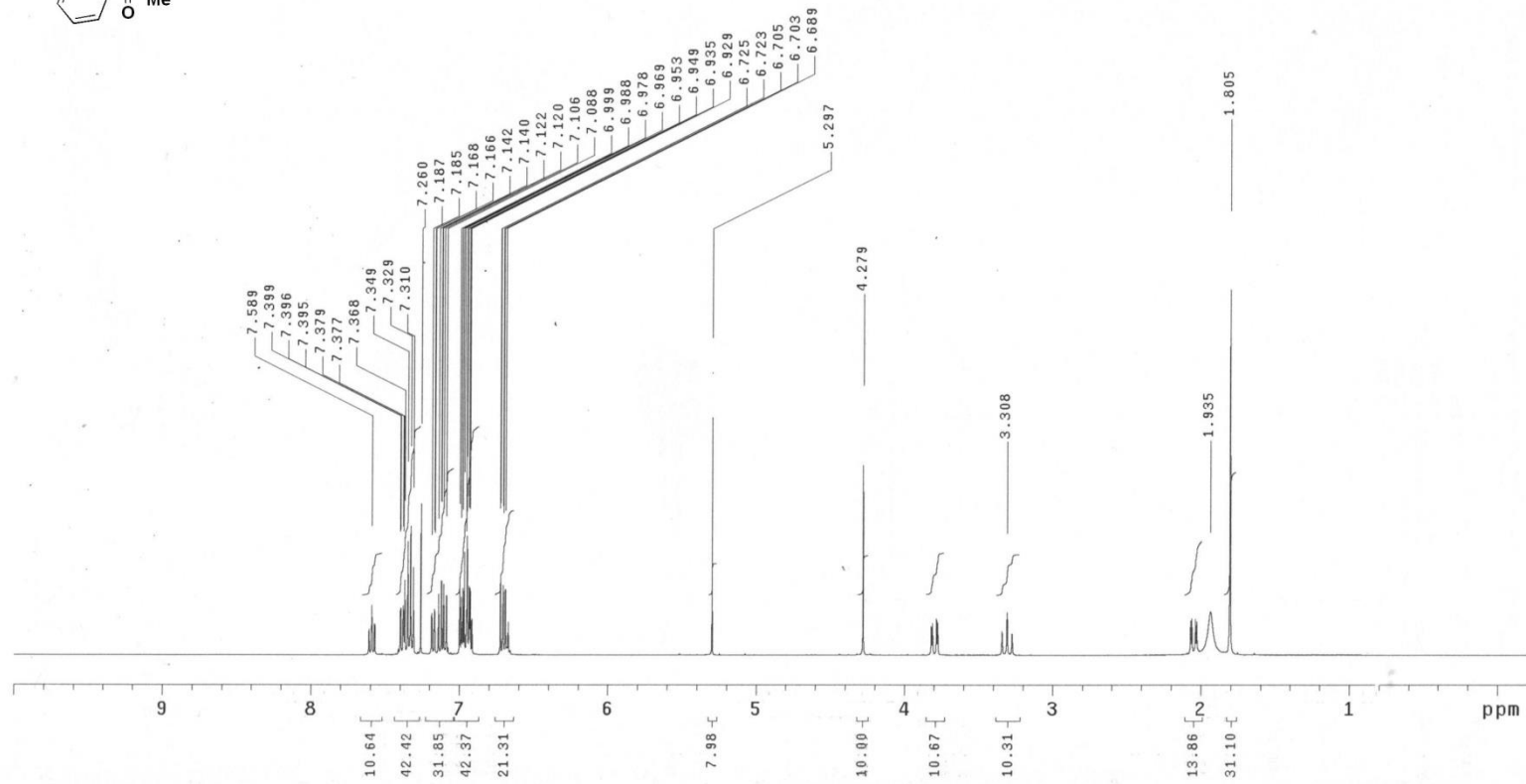
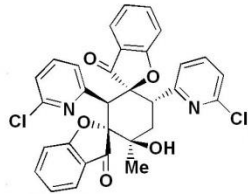
KT6PC93

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 7 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 3248 repetitions



# Compound 3y-2 (<sup>1</sup>H-NMR spectral data)

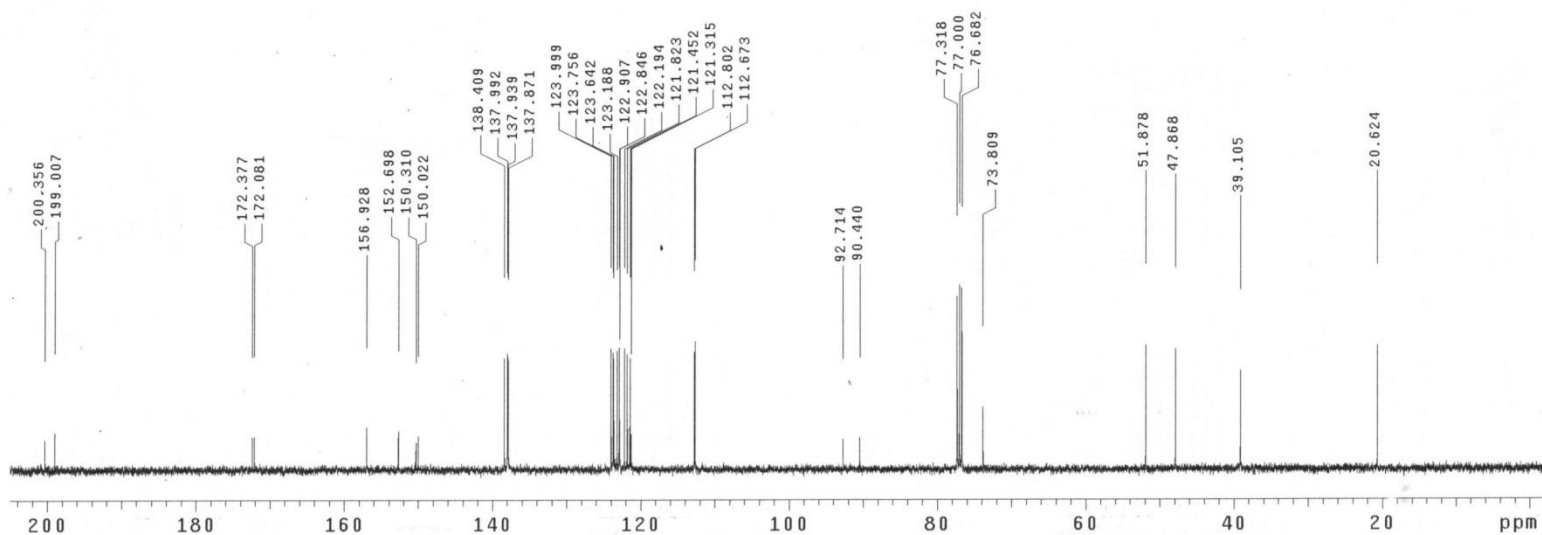
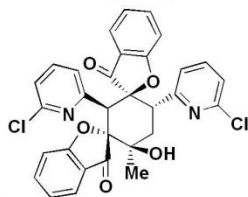
KT6PC104  
Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 14 2019  
Solvent: CDCl3  
Ambient temperature  
Total 32 repetitions



# Compound 3y-2 (<sup>13</sup>C-NMR spectral data)

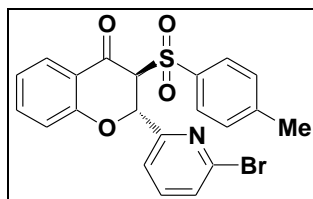
KT6PC104

Pulse Sequence: s2pu1  
UNITYplus-400 "unity400"  
Date: Oct 14 2019  
Solvent: CDCl<sub>3</sub>  
Ambient temperature  
Total 2432 repetitions



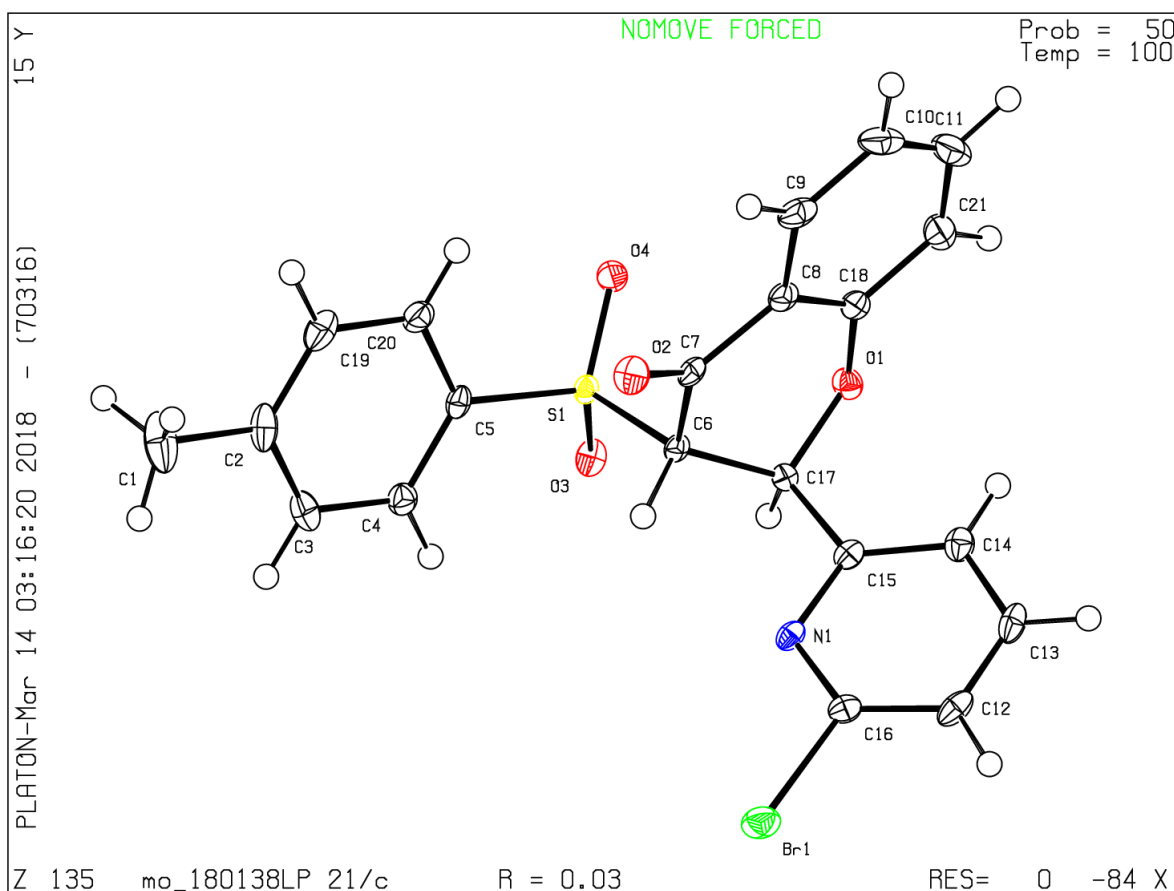


## X-ray crystal data of compound 2a (CCDC 1965985)



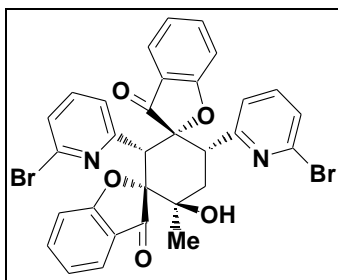
**Sample preparation** : A solution of compound **2a** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

**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.



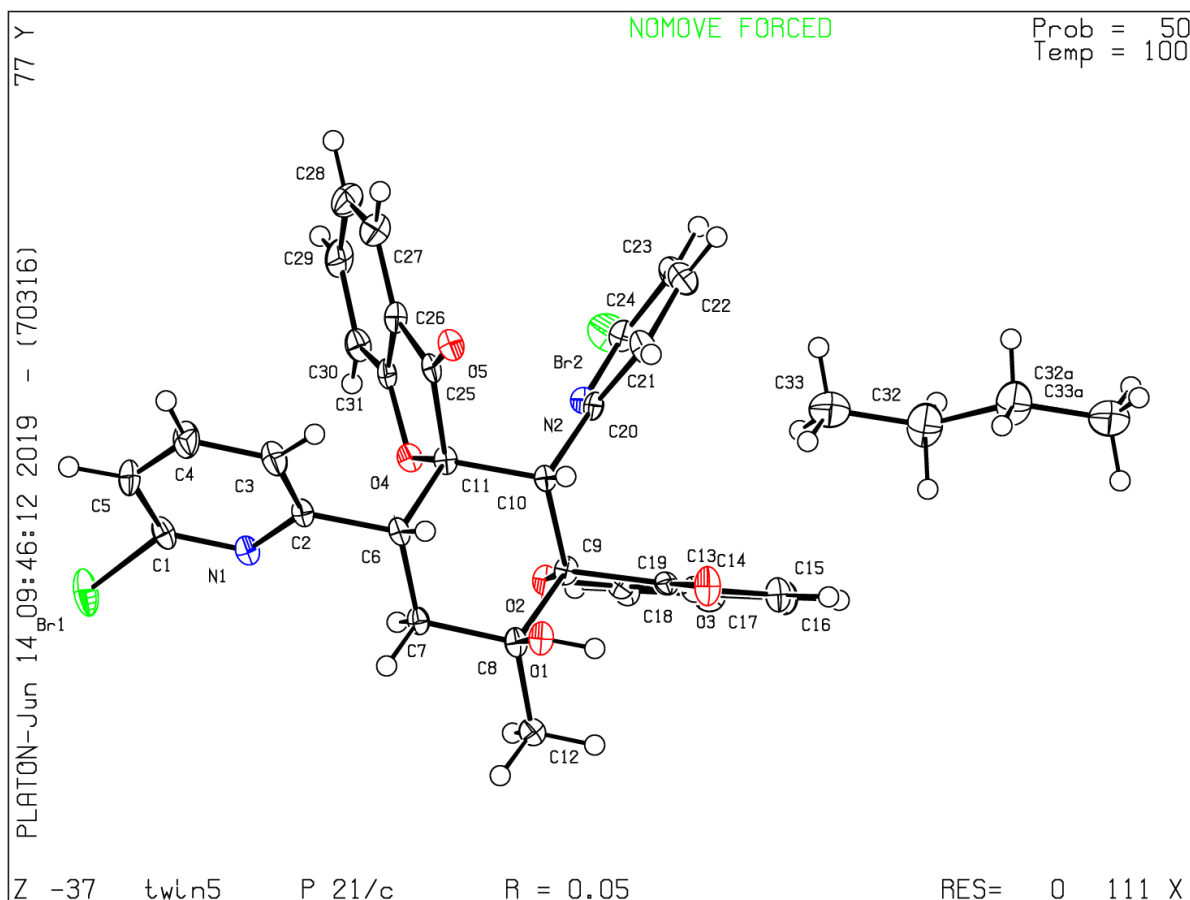
Empirical formula	C <sub>21</sub> H <sub>16</sub> Br N O <sub>4</sub> S	
Formula weight	458.32	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P 2 <sub>1</sub> /c	
Unit cell dimensions	a = 8.2647(4) Å	α = 90°.
	b = 19.7204(11) Å	β = 95.308(2)°.
	c = 11.6058(6) Å	γ = 90°.
Volume	1883.44(17) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.616 Mg/m <sup>3</sup>	
Absorption coefficient	2.322 mm <sup>-1</sup>	
F(000)	928	
Crystal size	0.20 x 0.18 x 0.17 mm <sup>3</sup>	
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 <sup>2</sup>	
Data / restraints / parameters	3930 / 0 / 254	
Goodness-of-fit on F <sup>2</sup>	1.041	
Final R indices [I > 2σ(I)]	R <sub>1</sub> = 0.0278, wR <sub>2</sub> = 0.0598	
R indices (all data)	R <sub>1</sub> = 0.0372, wR <sub>2</sub> = 0.0629	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.438 and -0.388 e.Å <sup>-3</sup>	

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



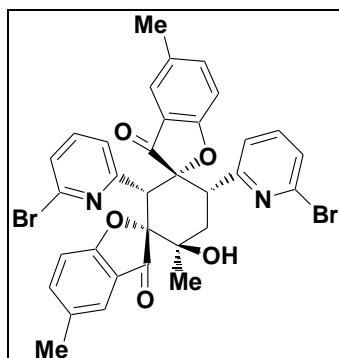
**Sample preparation** : A solution of compound **3a** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

**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.



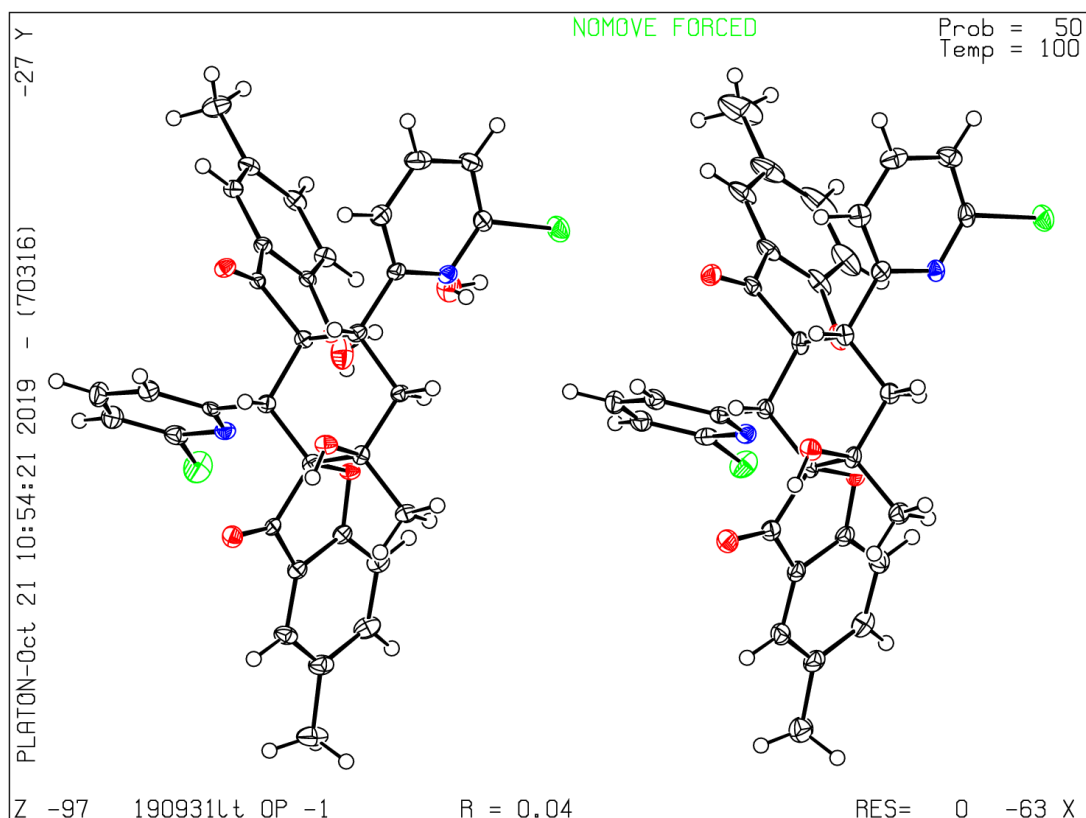
Empirical formula	C33 H27 Br2 N2 O5
Formula weight	691.38
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2 <sub>1</sub> /c
Unit cell dimensions	a = 13.1453(16) Å      α = 90°. b = 12.9849(18) Å      β = 96.328(5)°. c = 16.874(3) Å      γ = 90°.
Volume	2862.6(7) Å <sup>3</sup>
Z	4
Density (calculated)	1.604 Mg/m <sup>3</sup>
Absorption coefficient	2.878 mm <sup>-1</sup>
F(000)	1396
Crystal size	0.22 x 0.20 x 0.19 mm <sup>3</sup>
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 <sup>2</sup>
Data / restraints / parameters	6408 / 366 / 383
Goodness-of-fit on F <sup>2</sup>	1.043
Final R indices [I > 2σ(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 <sup>-3</sup>

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



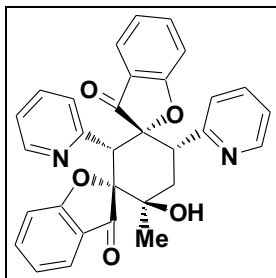
**Sample preparation :** A solution of compound **3i** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

**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.



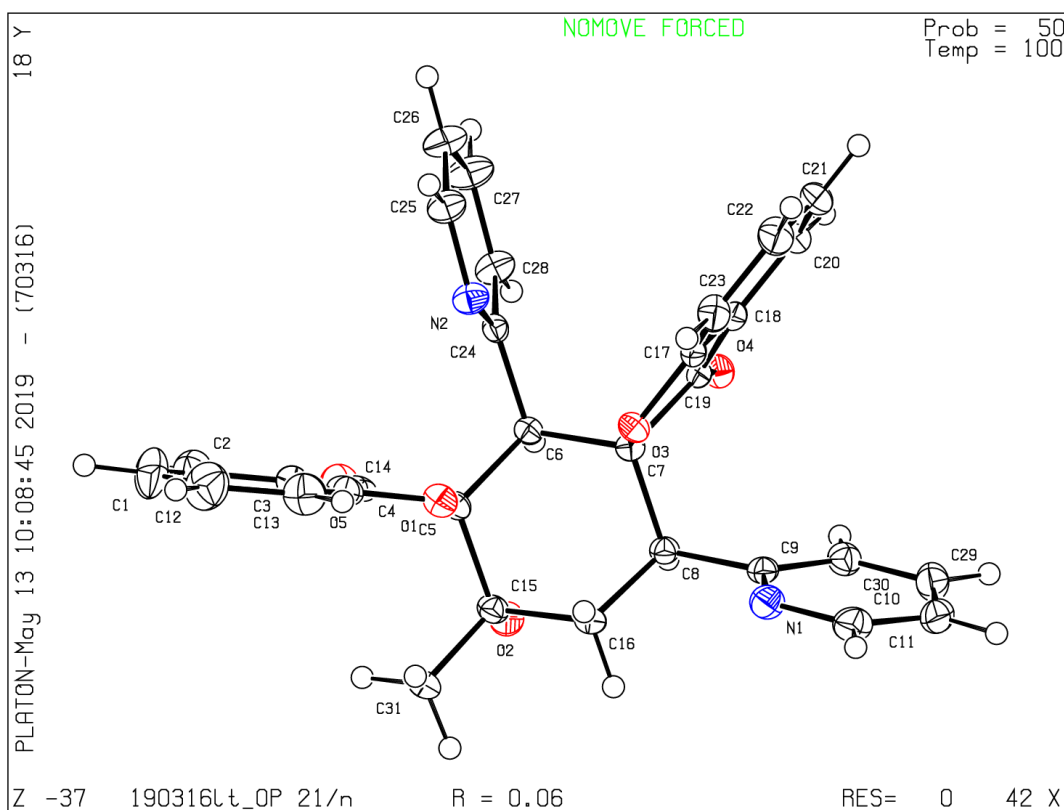
Empirical formula	C66 H56 Br4 N4 O12	
Formula weight	1416.78	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.3171(6) Å	$\alpha = 77.235(2)^\circ$ .
	b = 15.1045(7) Å	$\beta = 76.167(2)^\circ$ .
	c = 16.7543(6) Å	$\gamma = 67.821(3)^\circ$ .
Volume	2998.0(2) Å <sup>3</sup>	
Z	2	
Density (calculated)	1.569 Mg/m <sup>3</sup>	
Absorption coefficient	2.753 mm <sup>-1</sup>	
F(000)	1432	
Crystal size	0.10 x 0.08 x 0.08 mm <sup>3</sup>	
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 <sup>2</sup>	
Data / restraints / parameters	12292 / 6 / 799	
Goodness-of-fit on F <sup>2</sup>	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 <sup>-3</sup>	

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



**Sample preparation** : A solution of compound **3j** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (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.

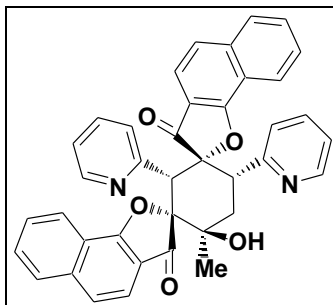
**Crystal measurement** : X-ray crystal structures were determined with a Bruker Enraf-Nonius single-crystal diffractometer (CAD4, Kappa CCD). Thermal ellipsoids are drawn at 50% probability level.



Empirical formula	C31 H23 N2 O5
Formula weight	503.51
Temperature	100(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	P2 <sub>1</sub> /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 <sup>3</sup>
Z	4
Density (calculated)	1.344 Mg/m <sup>3</sup>
Absorption coefficient	0.092 mm <sup>-1</sup>
F(000)	1052
Crystal size	0.20 x 0.18 x 0.16 mm <sup>3</sup>
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 <sup>2</sup>
Data / restraints / parameters	5099 / 0 / 344
Goodness-of-fit on F <sup>2</sup>	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 <sup>-3</sup>

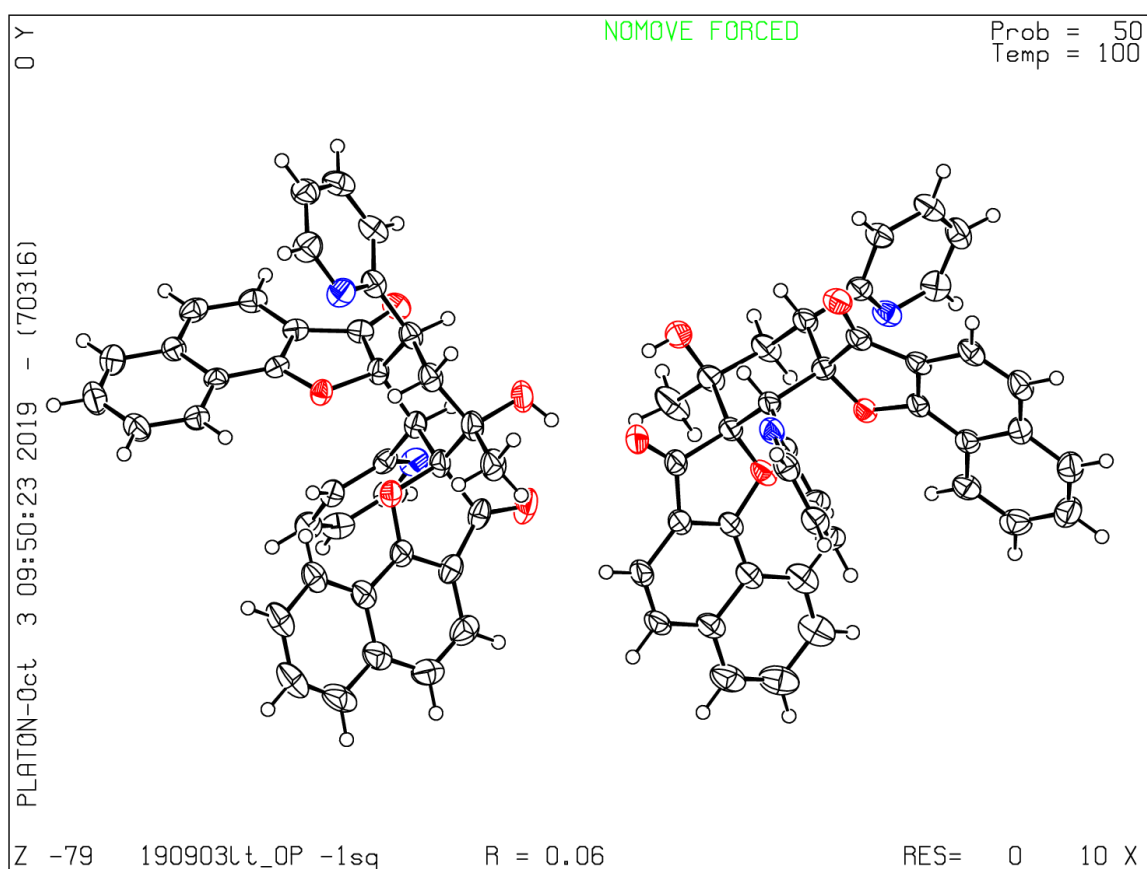


## X-ray crystal data of compound 3o (CCDC 1965996)



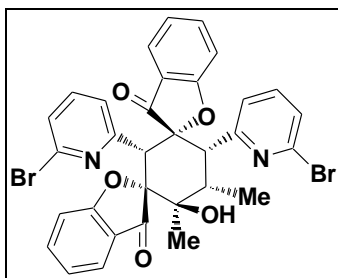
**Sample preparation :** A solution of compound **3o** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

**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.



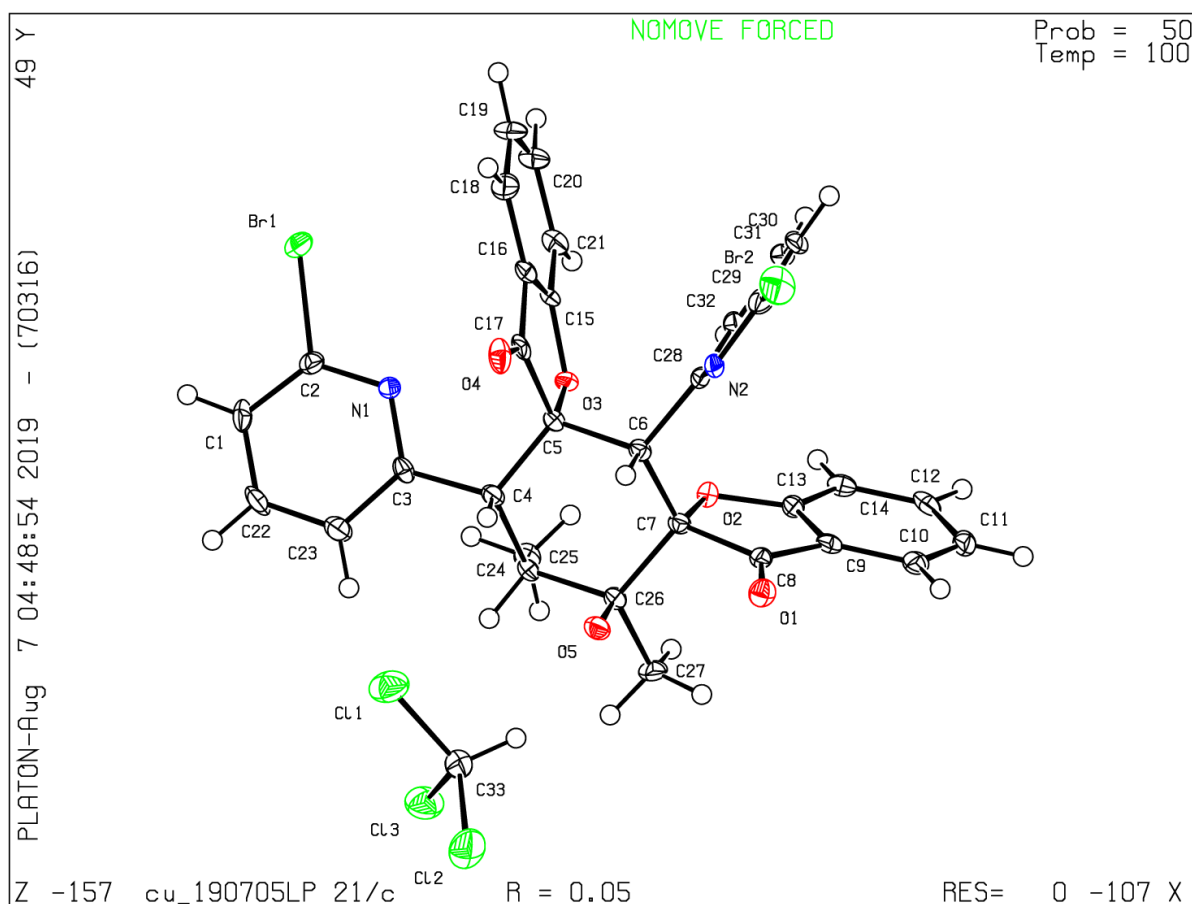
Empirical formula	C39 H28 N2 O5	
Formula weight	604.63	
Temperature	100(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 13.8900(7) Å	$\alpha = 64.295(2)^\circ$ .
	b = 16.8786(9) Å	$\beta = 88.306(2)^\circ$ .
	c = 17.2527(9) Å	$\gamma = 73.213(2)^\circ$ .
Volume	3467.7(3) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.158 Mg/m <sup>3</sup>	
Absorption coefficient	0.077 mm <sup>-1</sup>	
F(000)	1264	
Crystal size	0.18 x 0.16 x 0.15 mm <sup>3</sup>	
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 <sup>2</sup>	
Data / restraints / parameters	14196 / 0 / 833	
Goodness-of-fit on F <sup>2</sup>	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 <sup>-3</sup>	

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



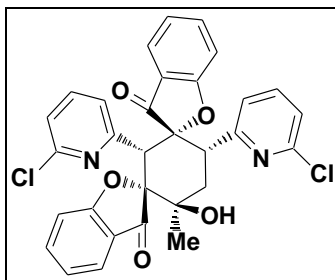
**Sample preparation** : A solution of compound **3r** (30 mg) in  $\text{CHCl}_3$  (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

**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.



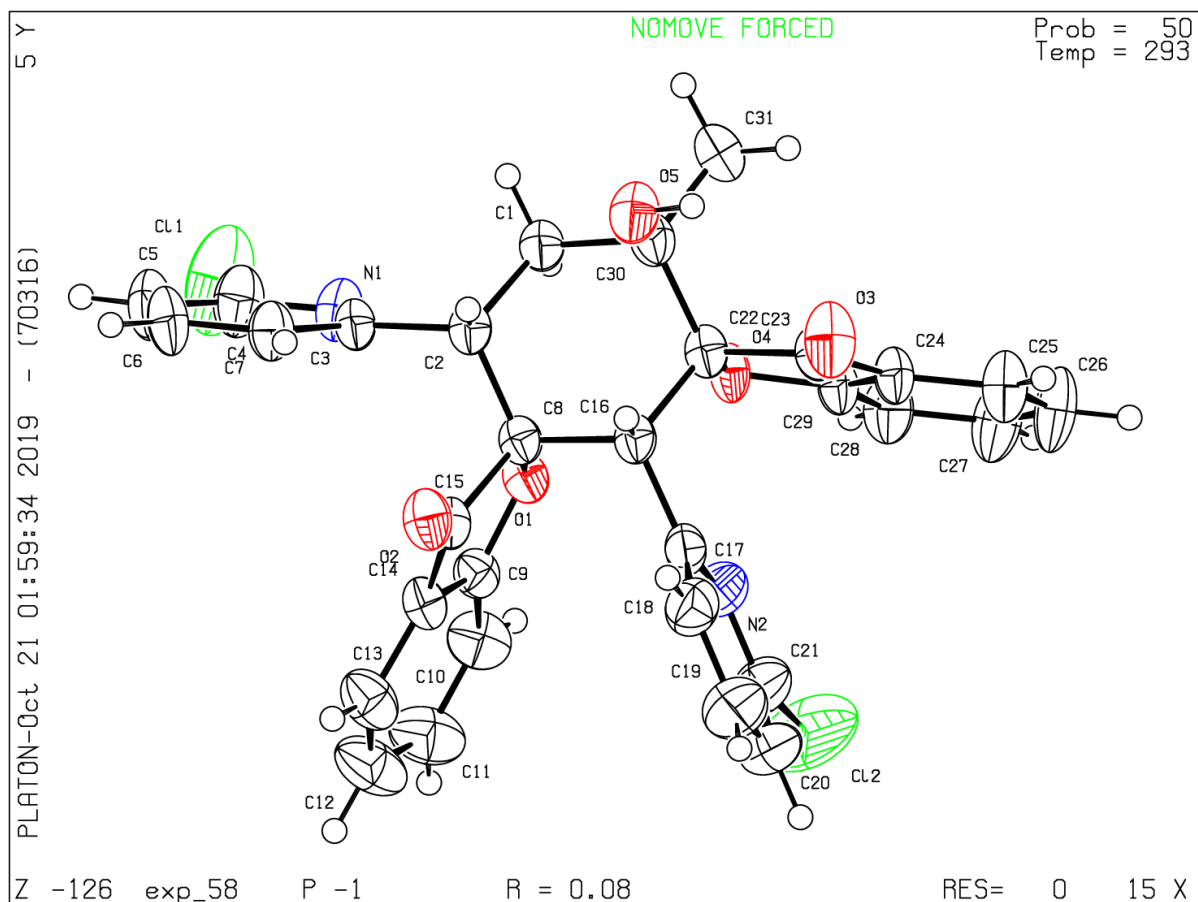
Empirical formula	C33 H24 Br2 Cl3 N2 O5	
Formula weight	794.71	
Temperature	100(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2 <sub>1</sub> /c	
Unit cell dimensions	a = 10.5330(4) Å	α = 90°.
	b = 26.8610(11) Å	β = 115.3340(10)°.
	c = 12.2233(5) Å	γ = 90°.
Volume	3125.7(2) Å <sup>3</sup>	
Z	4	
Density (calculated)	1.689 Mg/m <sup>3</sup>	
Absorption coefficient	6.036 mm <sup>-1</sup>	
F(000)	1588	
Crystal size	0.15 x 0.13 x 0.12 mm <sup>3</sup>	
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 <sup>2</sup>	
Data / restraints / parameters	5087 / 0 / 408	
Goodness-of-fit on F <sup>2</sup>	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 <sup>-3</sup>	

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



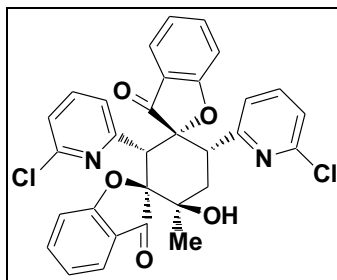
**Sample preparation** : A solution of compound **3y** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

**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.



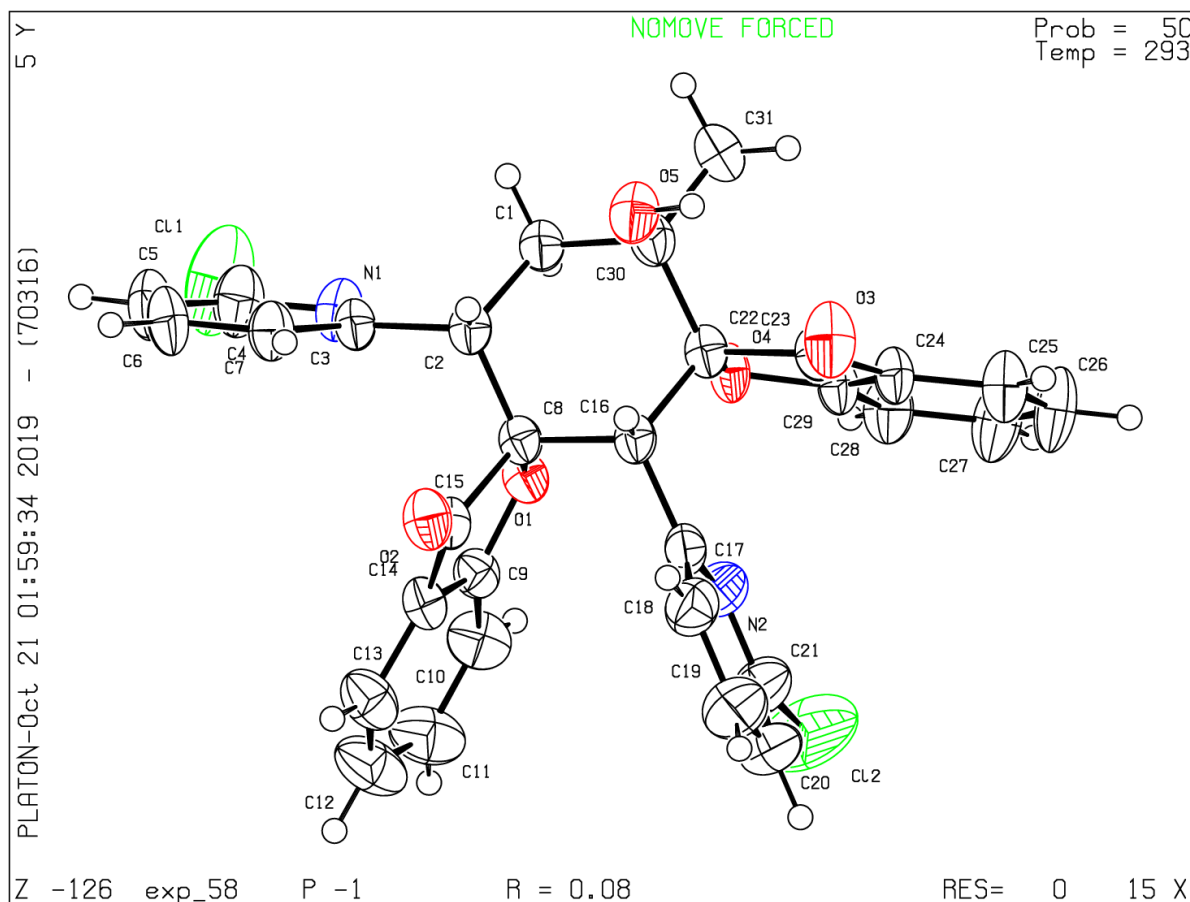
Empirical formula	C31 H22 Cl2 N2 O5	
Formula weight	573.40	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 10.8001(6) Å	$\alpha = 89.110(4)^\circ$ .
	b = 11.3693(6) Å	$\beta = 67.910(4)^\circ$ .
	c = 13.4193(5) Å	$\gamma = 68.542(5)^\circ$ .
Volume	1406.67(13) Å <sup>3</sup>	
Z	2	
Density (calculated)	1.354 Mg/m <sup>3</sup>	
Absorption coefficient	0.274 mm <sup>-1</sup>	
F(000)	592	
Crystal size	0.2 x 0.2 x 0.2 mm <sup>3</sup>	
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 <sup>2</sup>	
Data / restraints / parameters	5795 / 12 / 363	
Goodness-of-fit on F <sup>2</sup>	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 <sup>-3</sup>	

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



**Sample preparation** : A solution of compound **3y-1** (30 mg) in CH<sub>2</sub>Cl<sub>2</sub> (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	$\alpha = 69.112(11)^\circ$ .
	b = 11.9111(14) Å	$\beta = 87.877(10)^\circ$ .
	c = 12.3891(14) Å	$\gamma = 65.530(12)^\circ$ .
Volume	1358.3(3) Å <sup>3</sup>	
Z	2	
Density (calculated)	1.402 Mg/m <sup>3</sup>	
Absorption coefficient	0.284 mm <sup>-1</sup>	
F(000)	592	
Crystal size	0.2 x 0.2 x 0.2 mm <sup>3</sup>	
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 <sup>2</sup>	
Data / restraints / parameters	5593 / 198 / 363	
Goodness-of-fit on F <sup>2</sup>	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 <sup>-3</sup>	