

Supporting Information

Ac₂O-Mediated Dearylacetylation Dimerization of 2-Arylacetyl-1-naphthols. Synthesis of Naphtho[1,2-*b*]furan-3-ones

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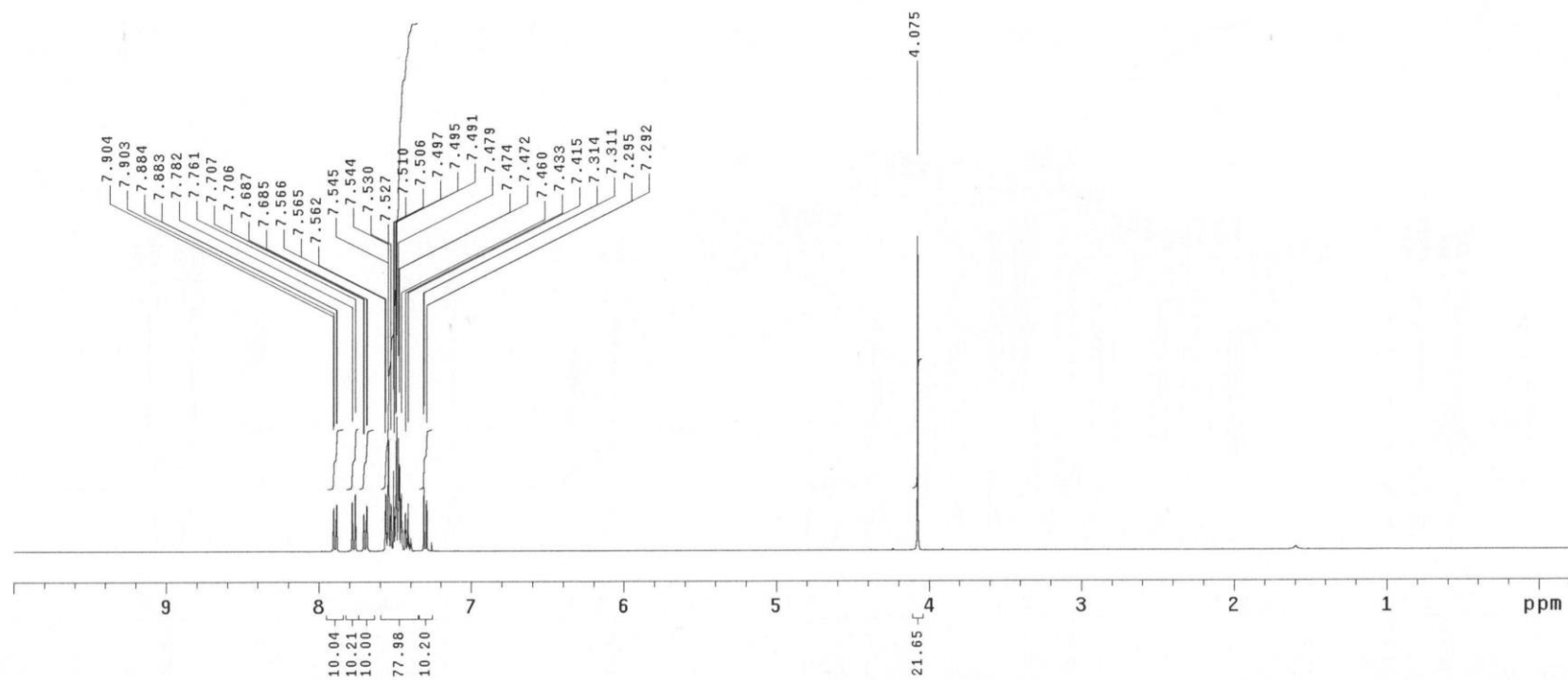
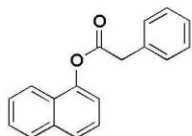
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Compound 3a (¹H-NMR spectral data)

YTN1H

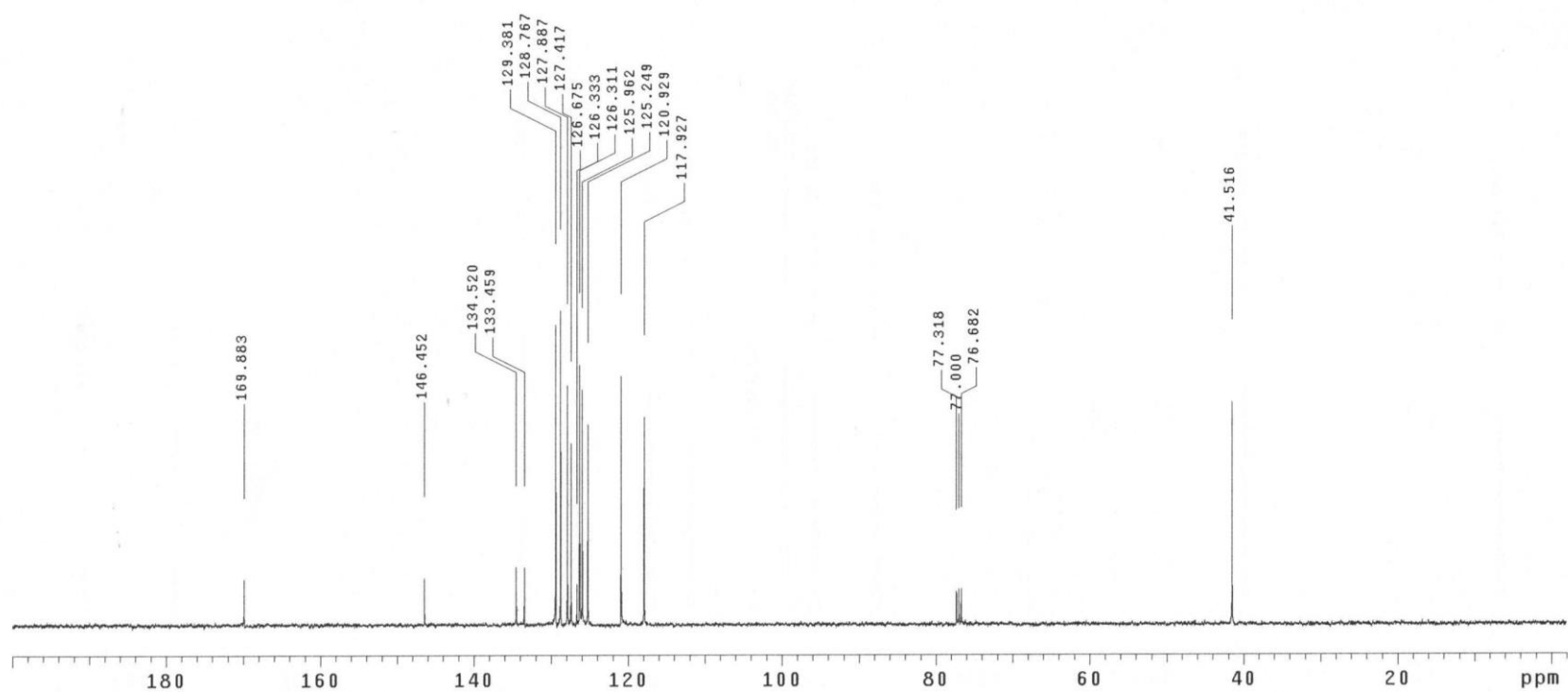
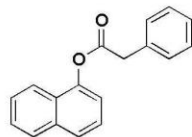
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Date: Dec 14 2017
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 3a (¹³C-NMR spectral data)

YTN1H

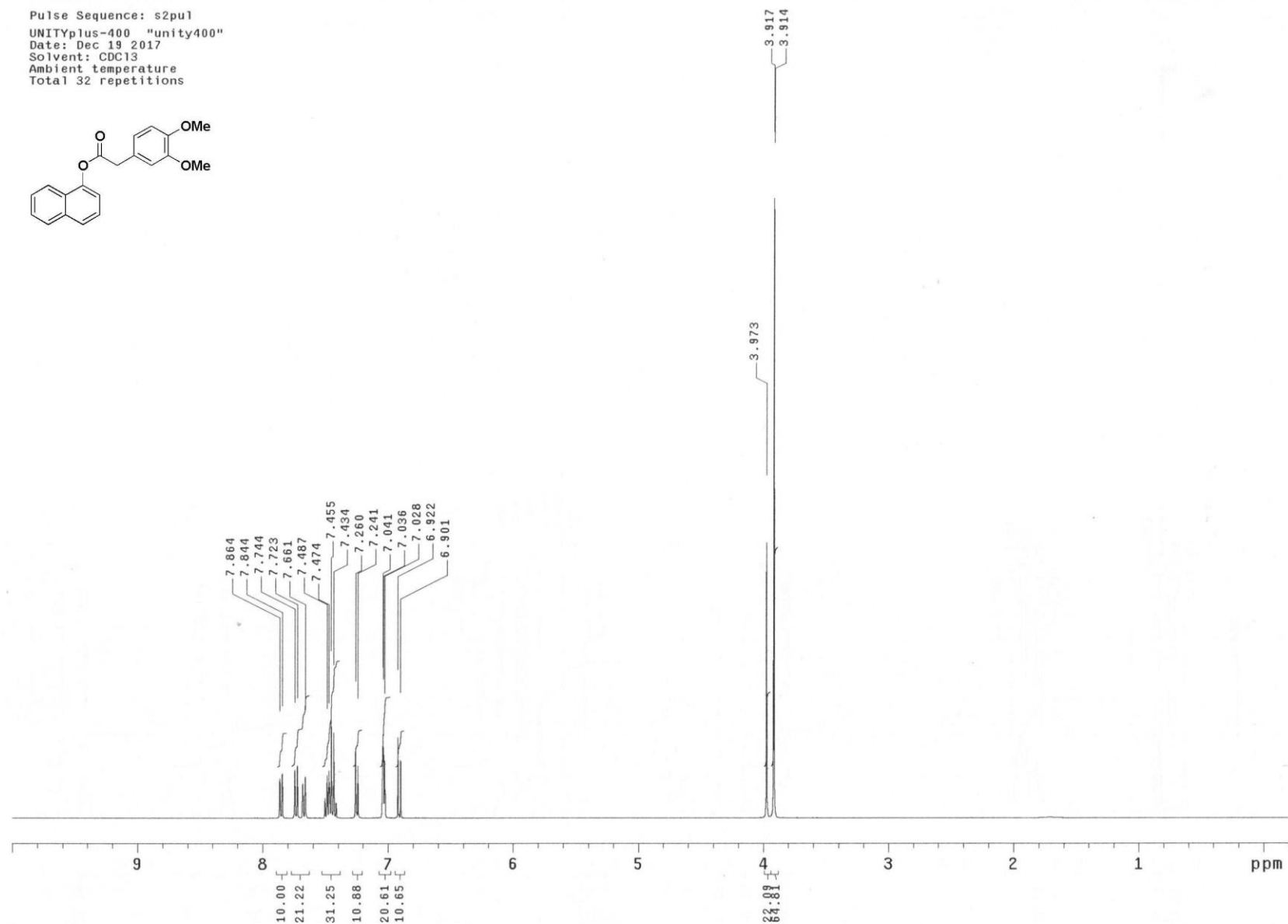
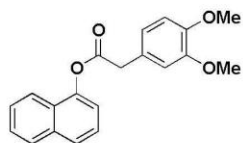
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Date: Dec 14 2017
Solvent: CDCl₃
Ambient temperature
Total 256 repetitions



Compound 3b (¹H-NMR spectral data)

YTN102

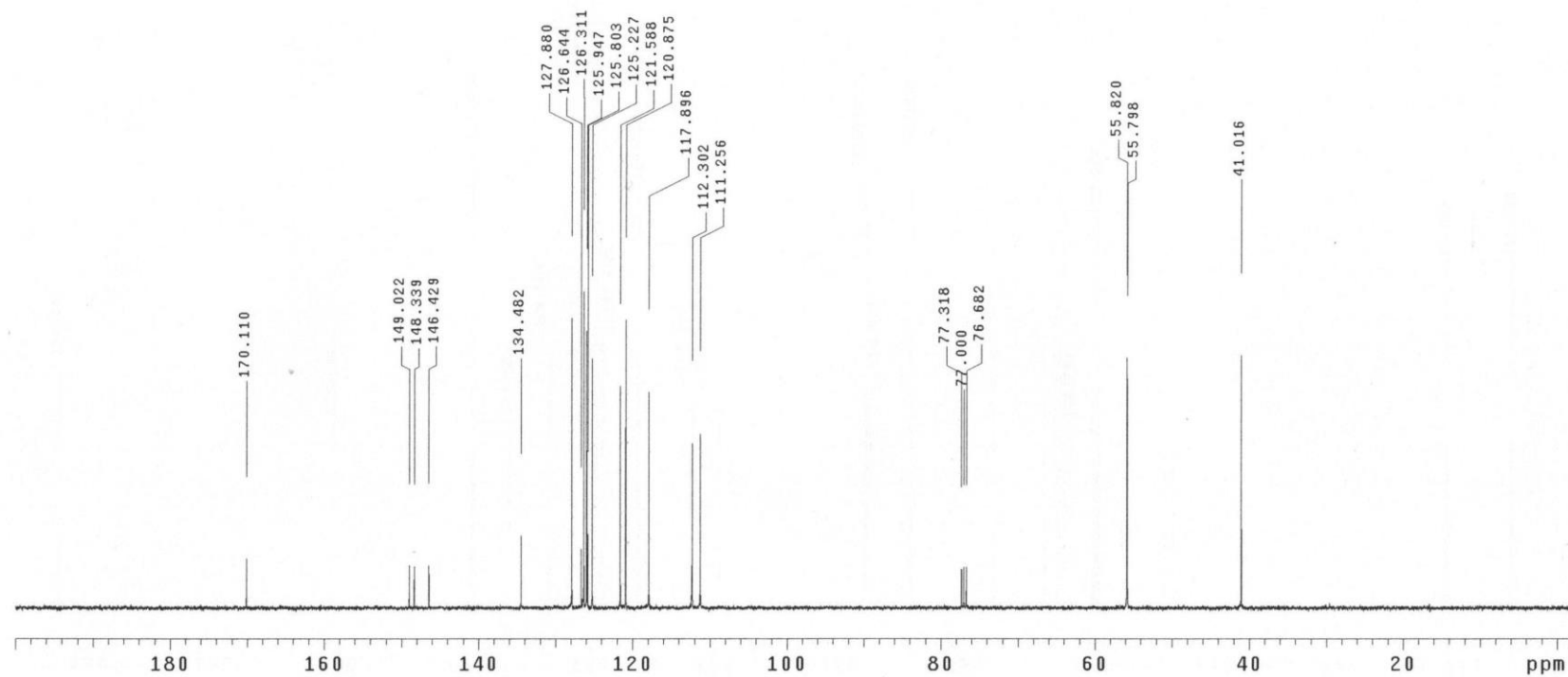
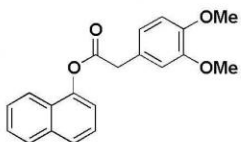
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 UNITYplus-400 "unity400"
 Date: Dec 19 2017
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 3b (¹³C-NMR spectral data)

YTN102

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 19 2017
Solvent: CDCl₃
Ambient temperature
Total 480 repetitions



Compound 3c (¹H-NMR spectral data)

YTN4F

Pulse Sequence: s2pu1

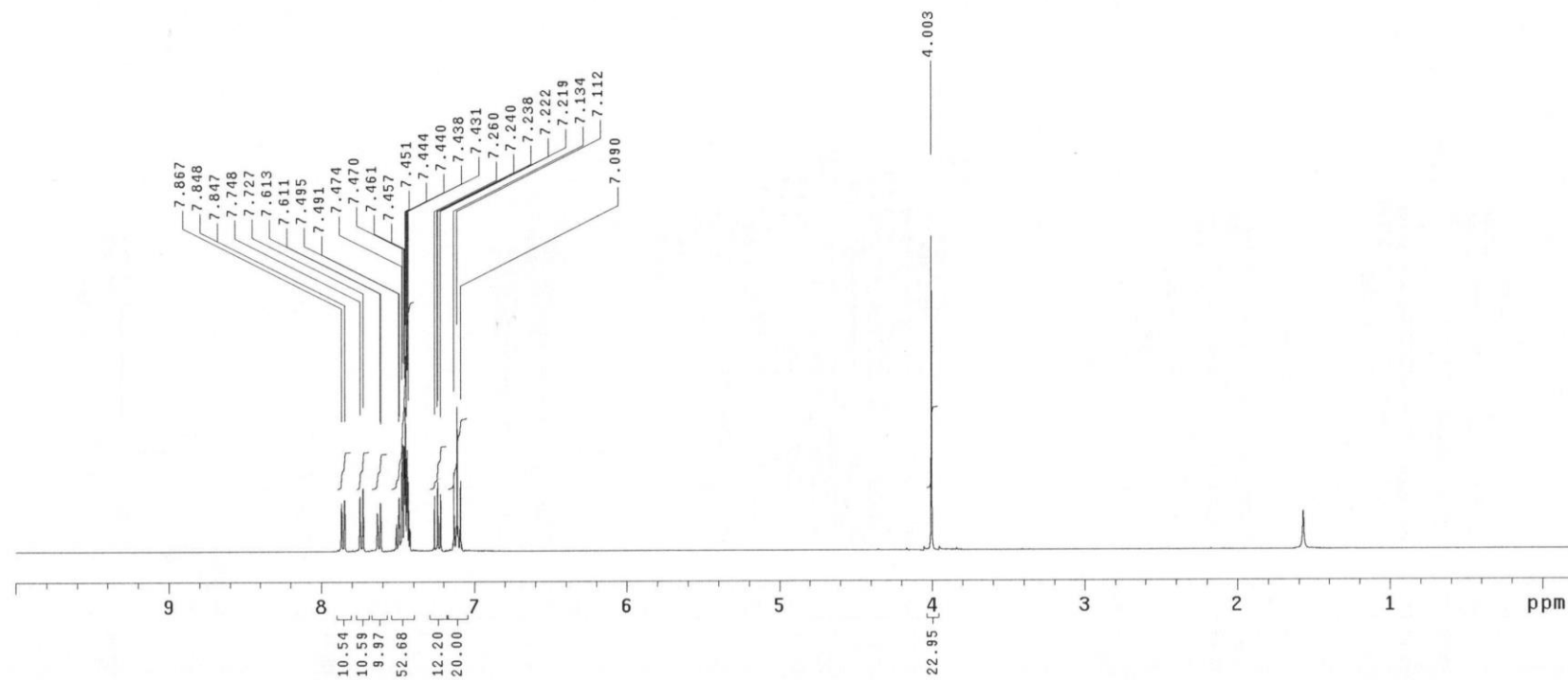
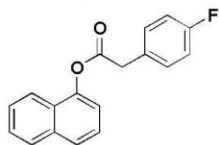
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Date: Jan 3 2018

Solvent: CDCl₃

Ambient temperature

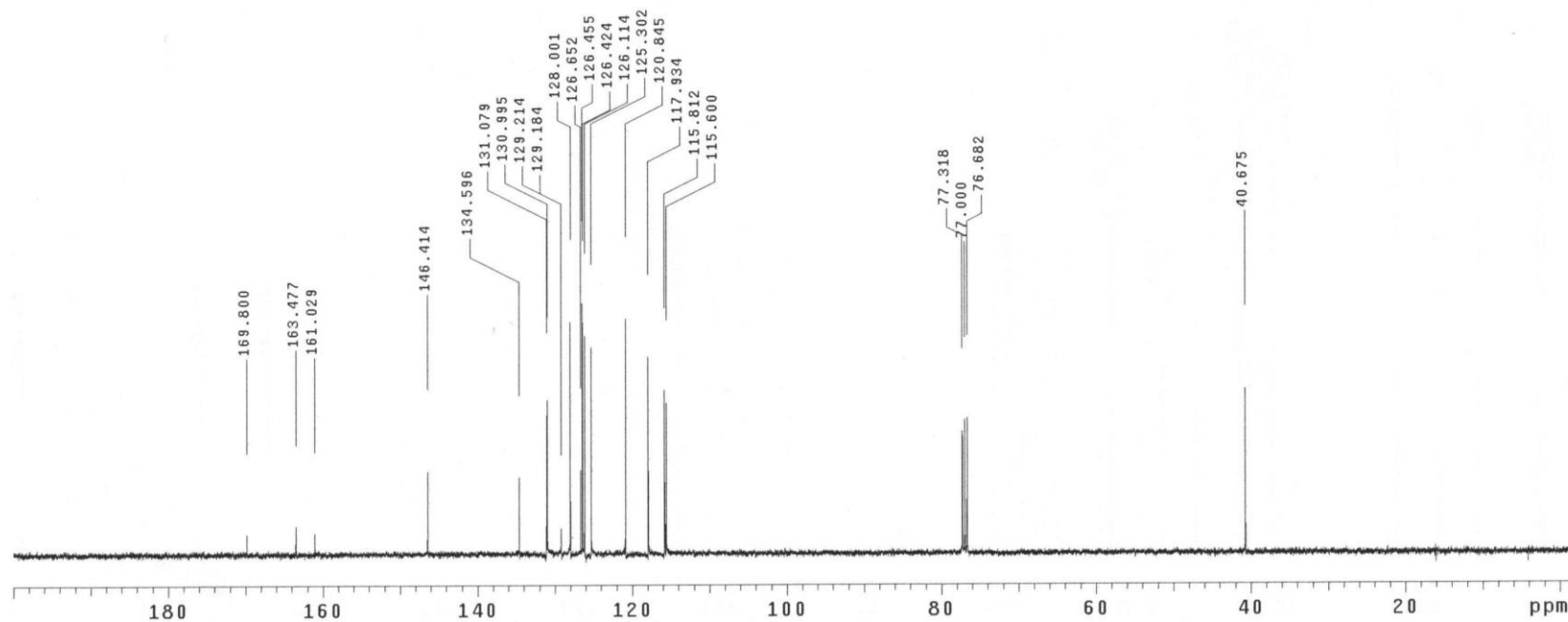
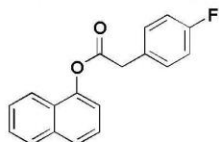
Total 32 repetitions



Compound 3c (¹³C-NMR spectral data)

YTN4F

Pulse Sequence: s2pu1
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Date: Jan 3 2018
Solvent: CDCl₃
Ambient temperature
Total 3280 repetitions



Compound 3d (¹H-NMR spectral data)

YTN4MeH

Pulse Sequence: s2pu1

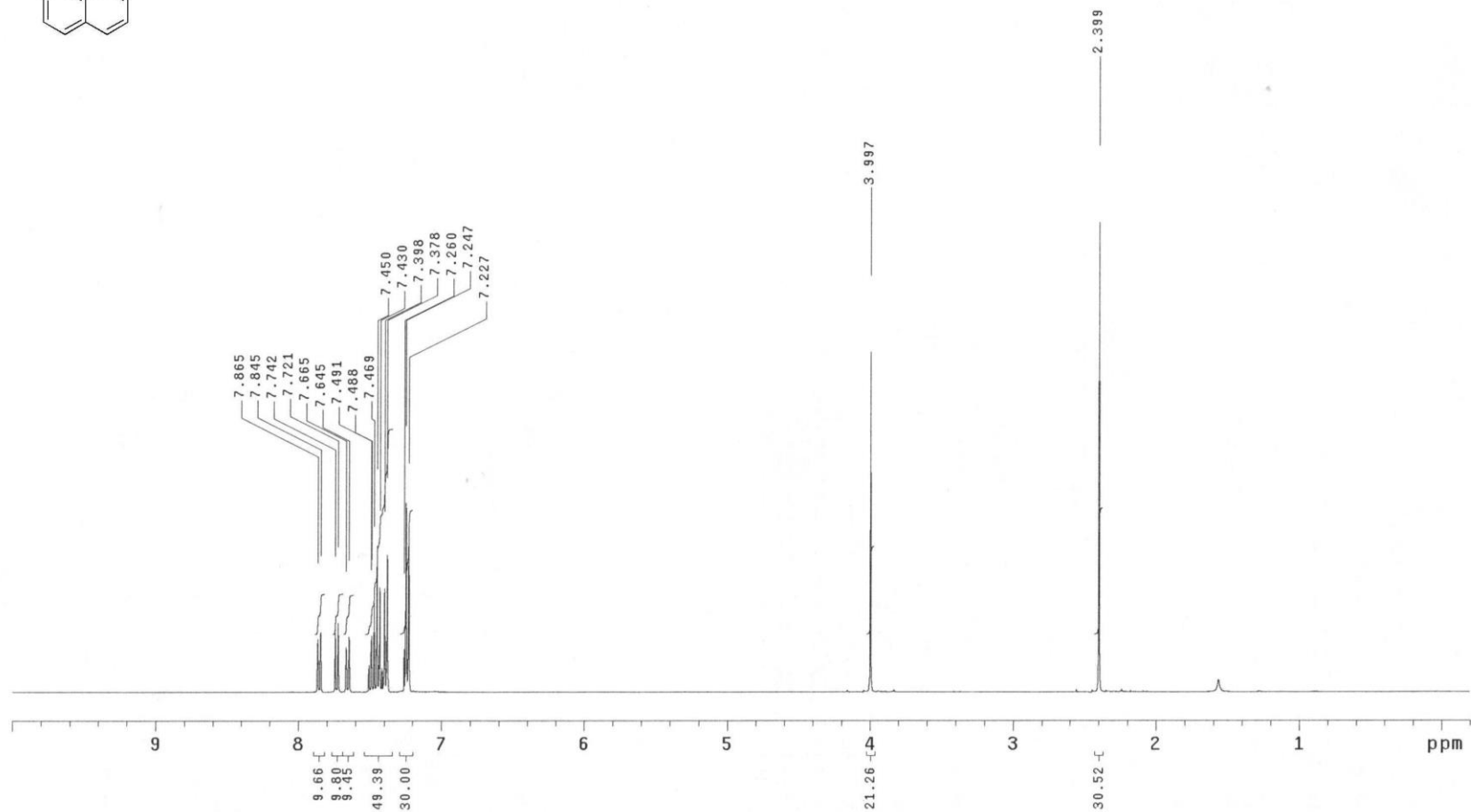
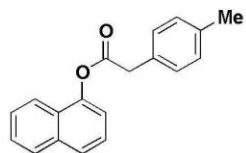
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Date: Jan 12 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3d (¹³C-NMR spectral data)

YTN4MeH

Pulse Sequence: s2pu1

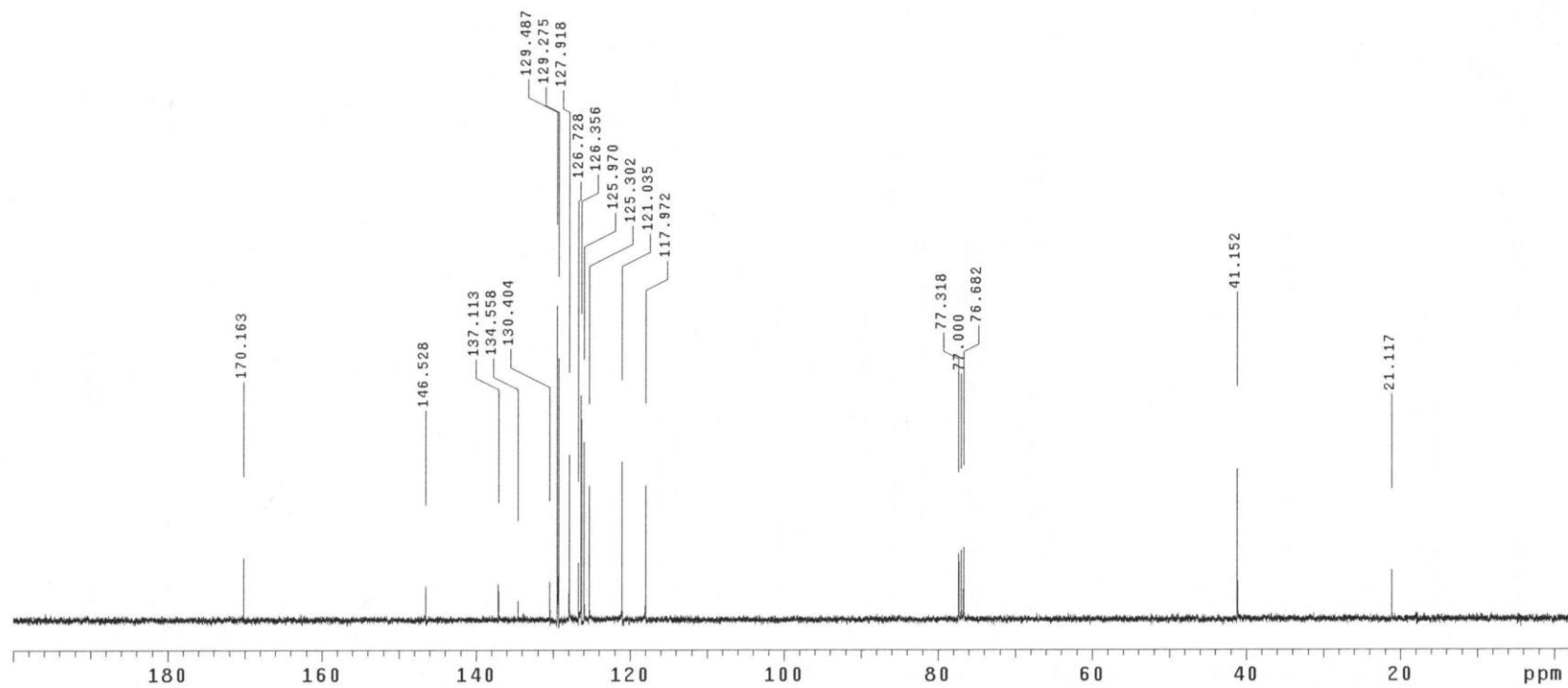
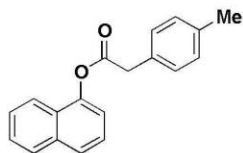
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Date: Jan 12 2018

Solvent: CDCl₃

Ambient temperature

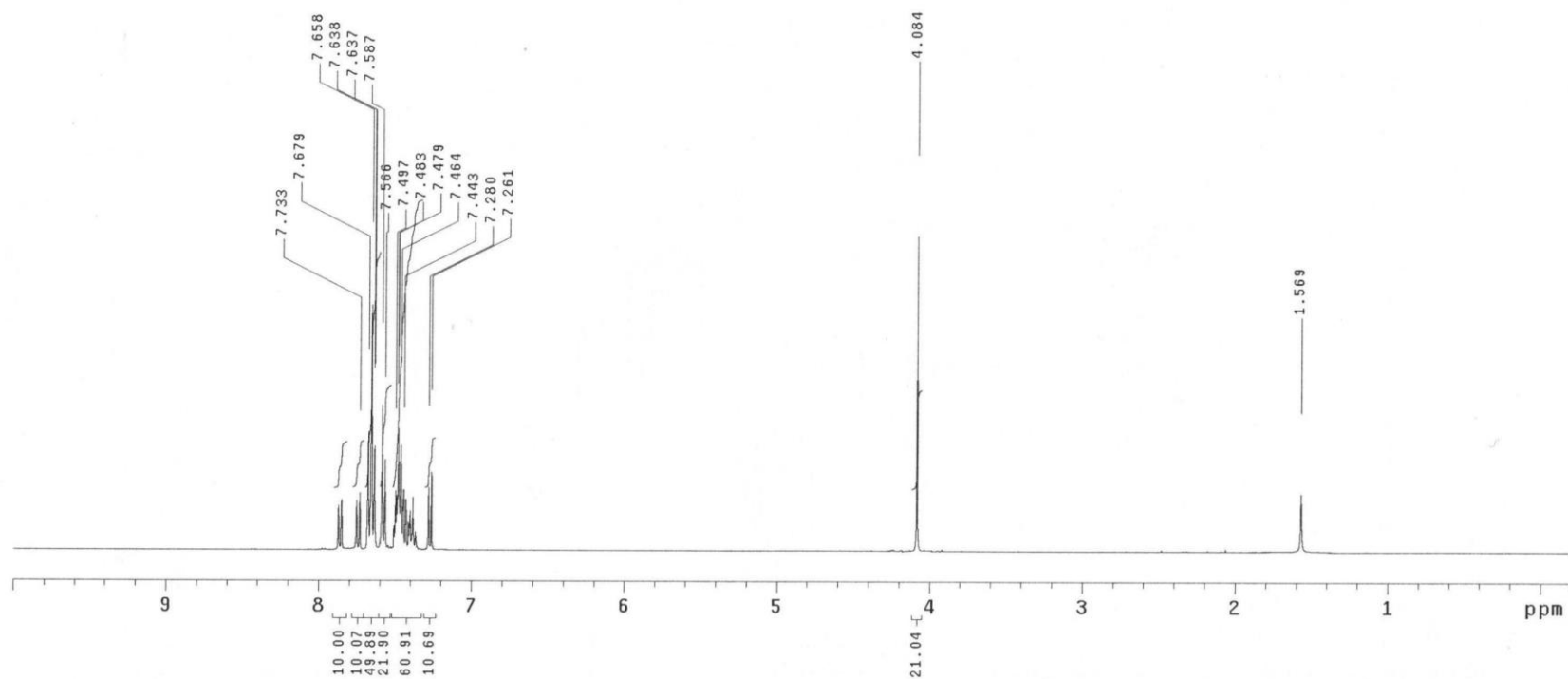
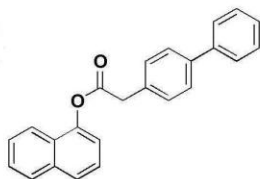
Total 848 repetitions



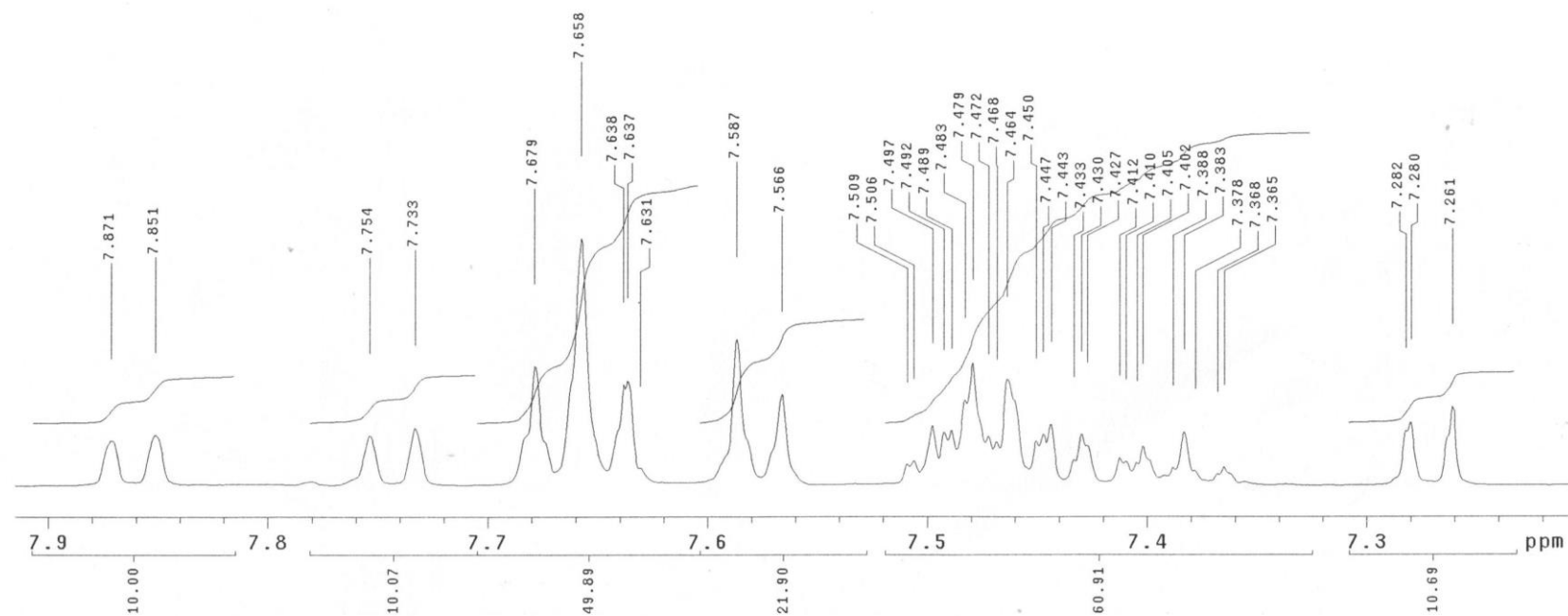
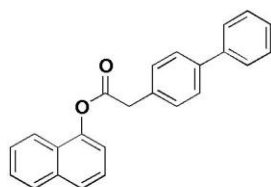
Compound 3e (¹H-NMR spectral data-1)

YTNPhPh

Pulse Sequence: s2pu1
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Date: Mar 16 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



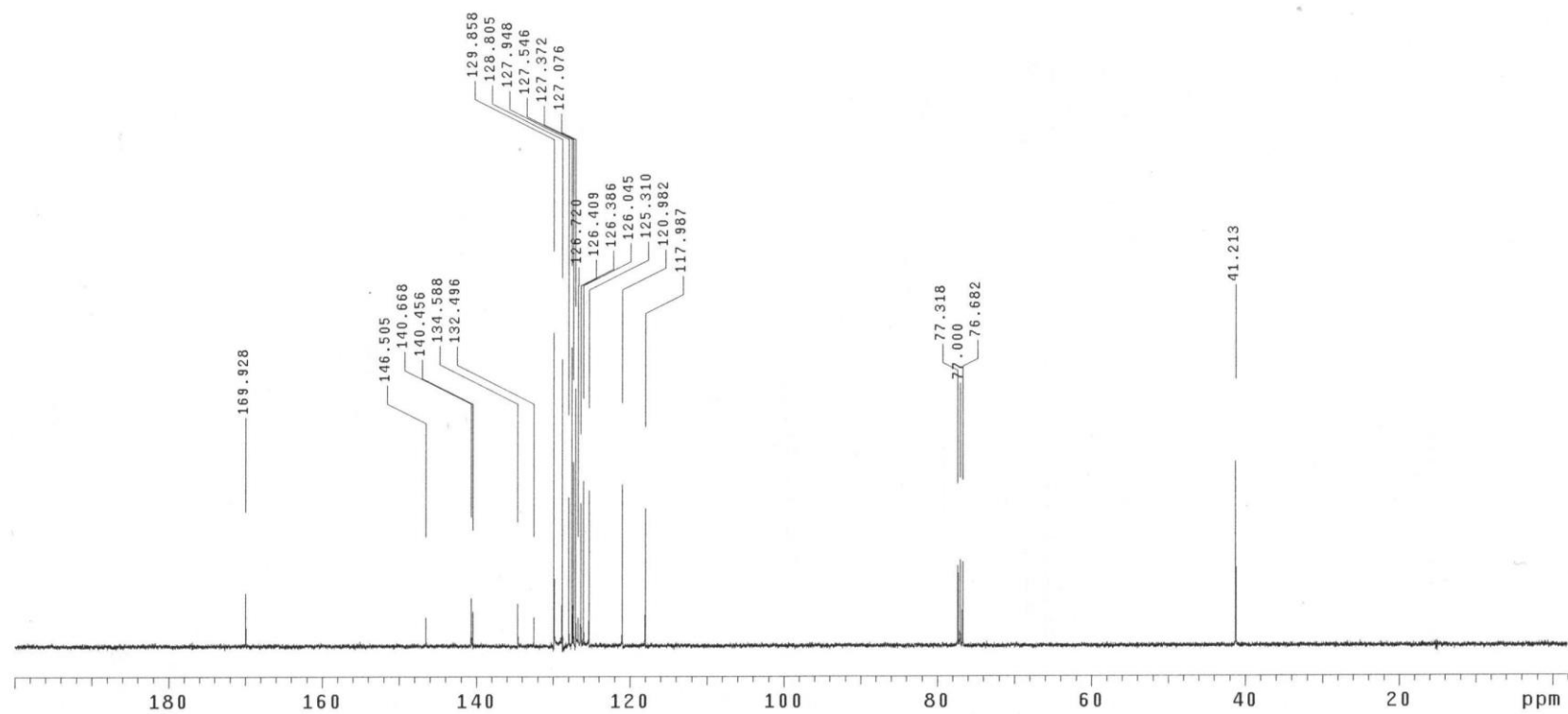
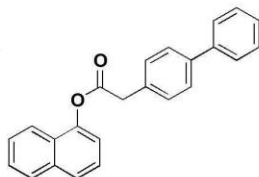
Compound 3e (¹H-NMR spectral data-2)



Compound 3e (¹³C-NMR spectral data)

YTNPhPh

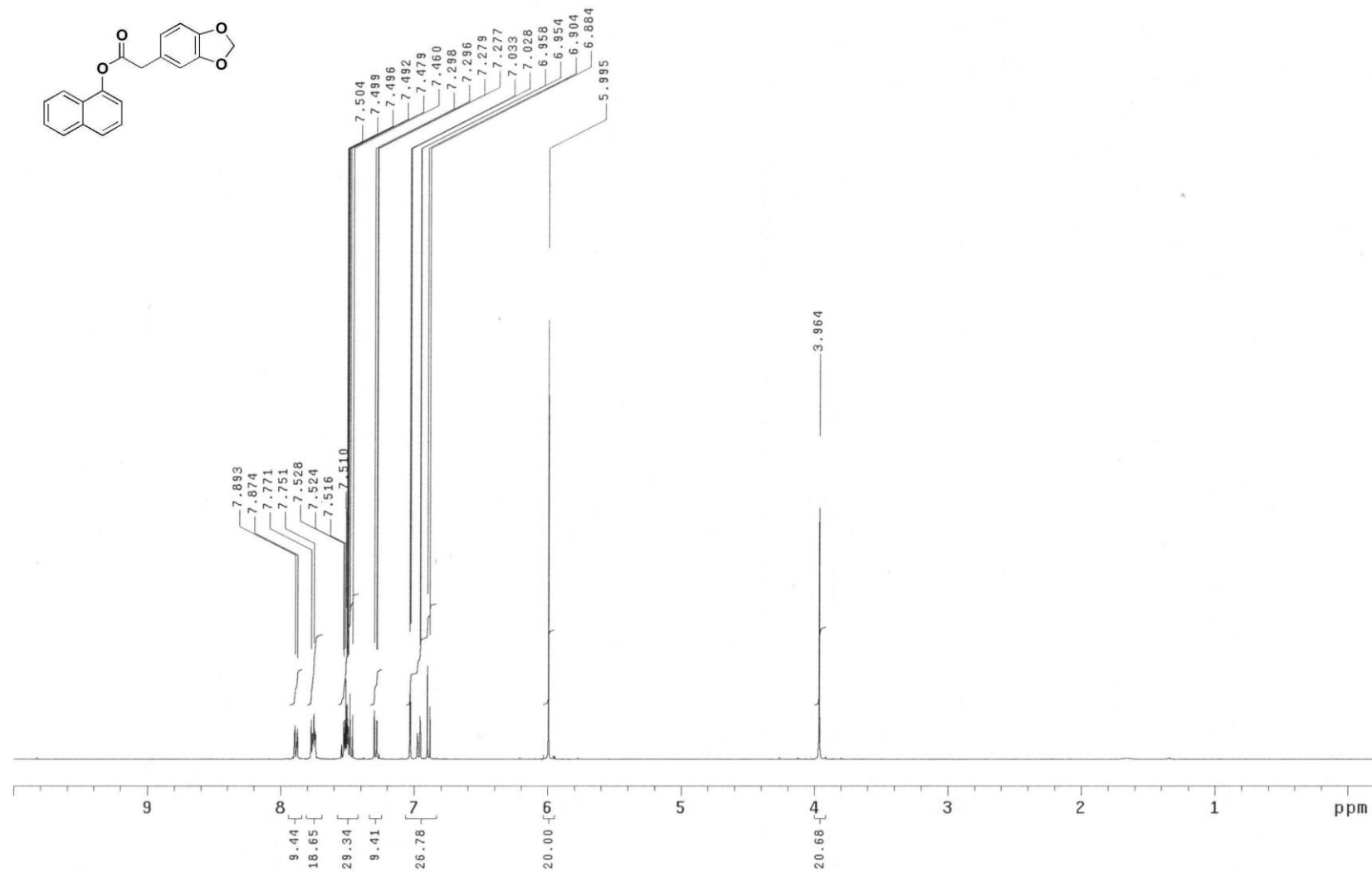
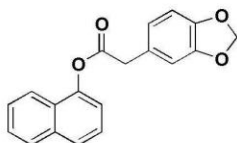
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Solvent: CDCl₃
Ambient temperature
Total 2432 repetitions



Compound 3f (¹H-NMR spectral data)

YTN02H

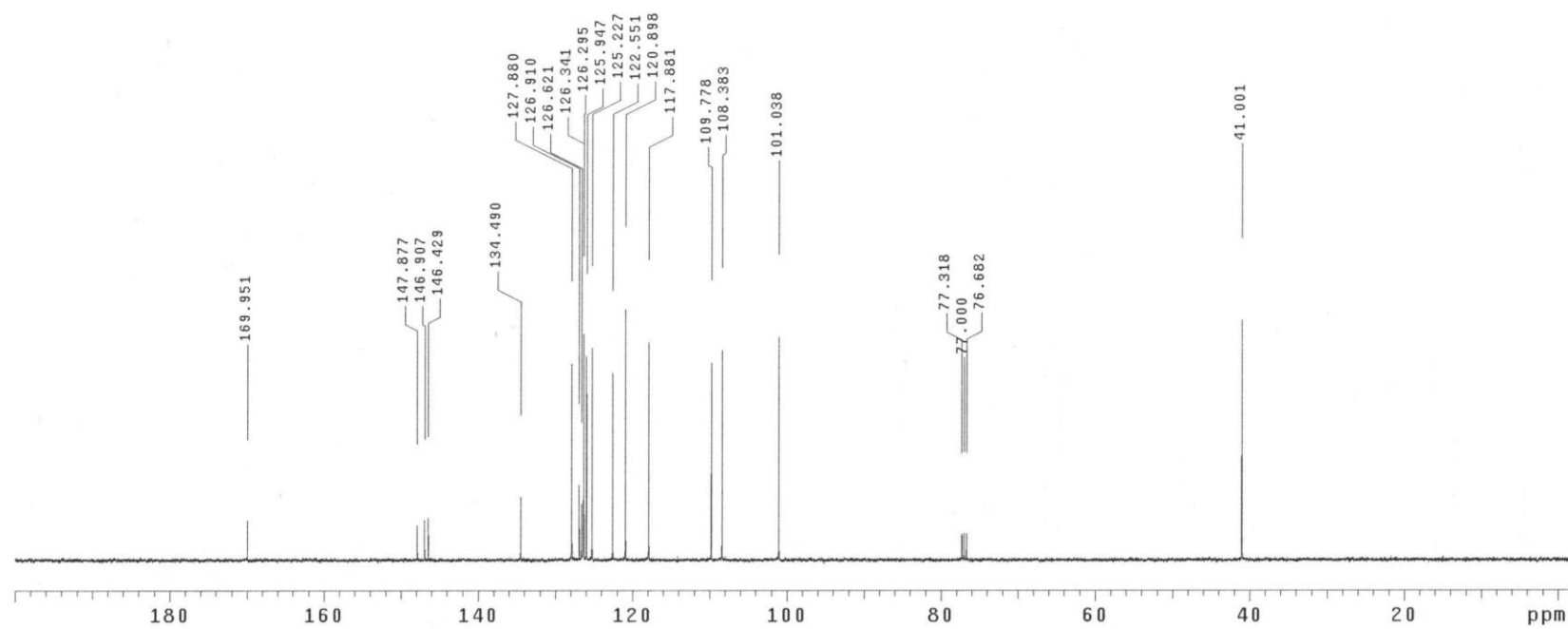
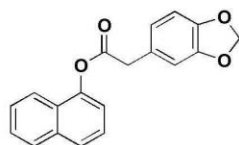
Pulse Sequence: s2pul
 UNITYplus-400 "unity400"
 Date: Mar 28 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



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

YTN02H

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Mar 28 2018
Solvent: CDCl₃
Ambient temperature
Total 128 repetitions



Compound 3g (¹H-NMR spectral data)

SHPPAS

Pulse Sequence: s2pu1

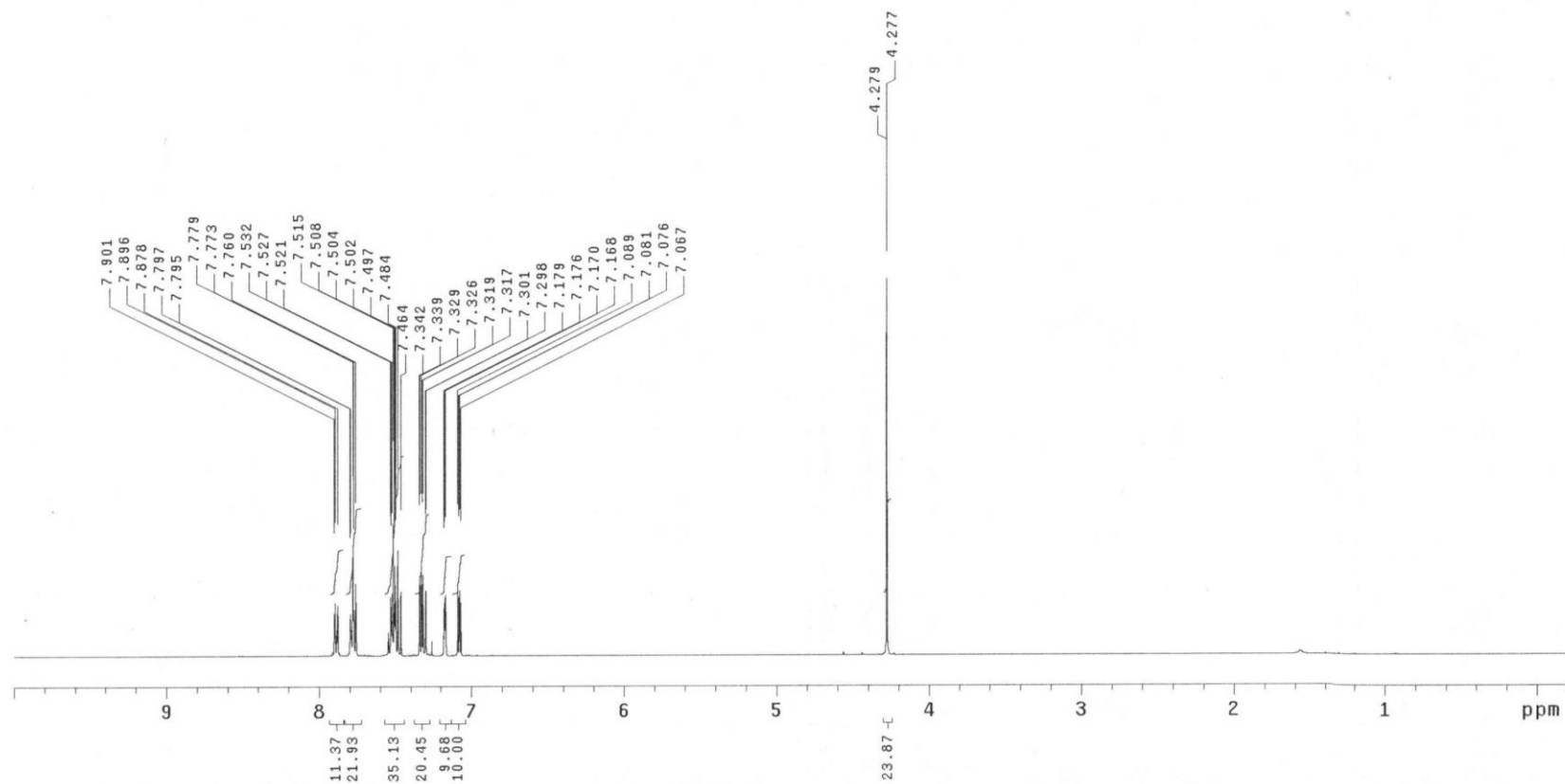
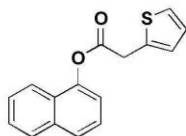
UNITYplus-400 "unity400"

Date: Apr 12 2018

Solvent: CDCl₃

Ambient temperature

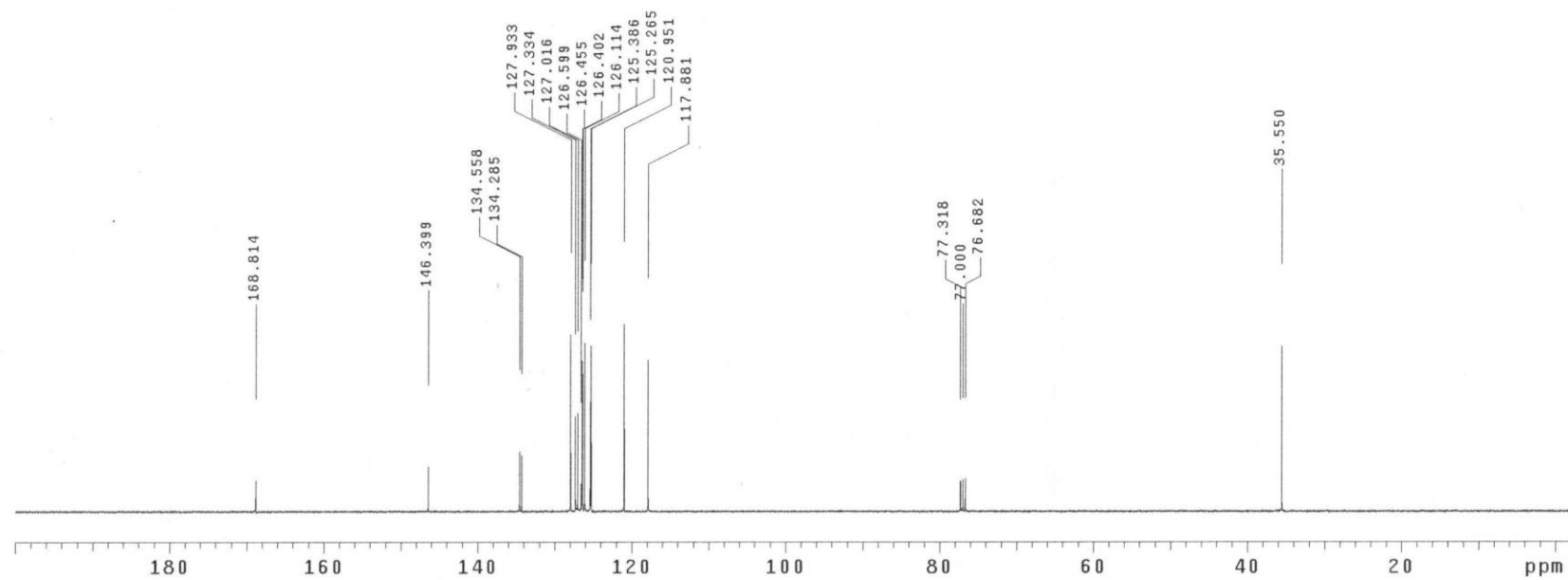
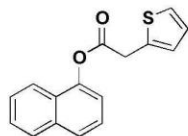
Total 32 repetitions



Compound 3g (¹³C-NMR spectral data)

SHPPAS

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Apr 12 2018
Solvent: CDCl₃
Ambient temperature
Total 736 repetitions



Compound 3h (¹H-NMR spectral data-1)

YT1070409

Pulse Sequence: s2pu1

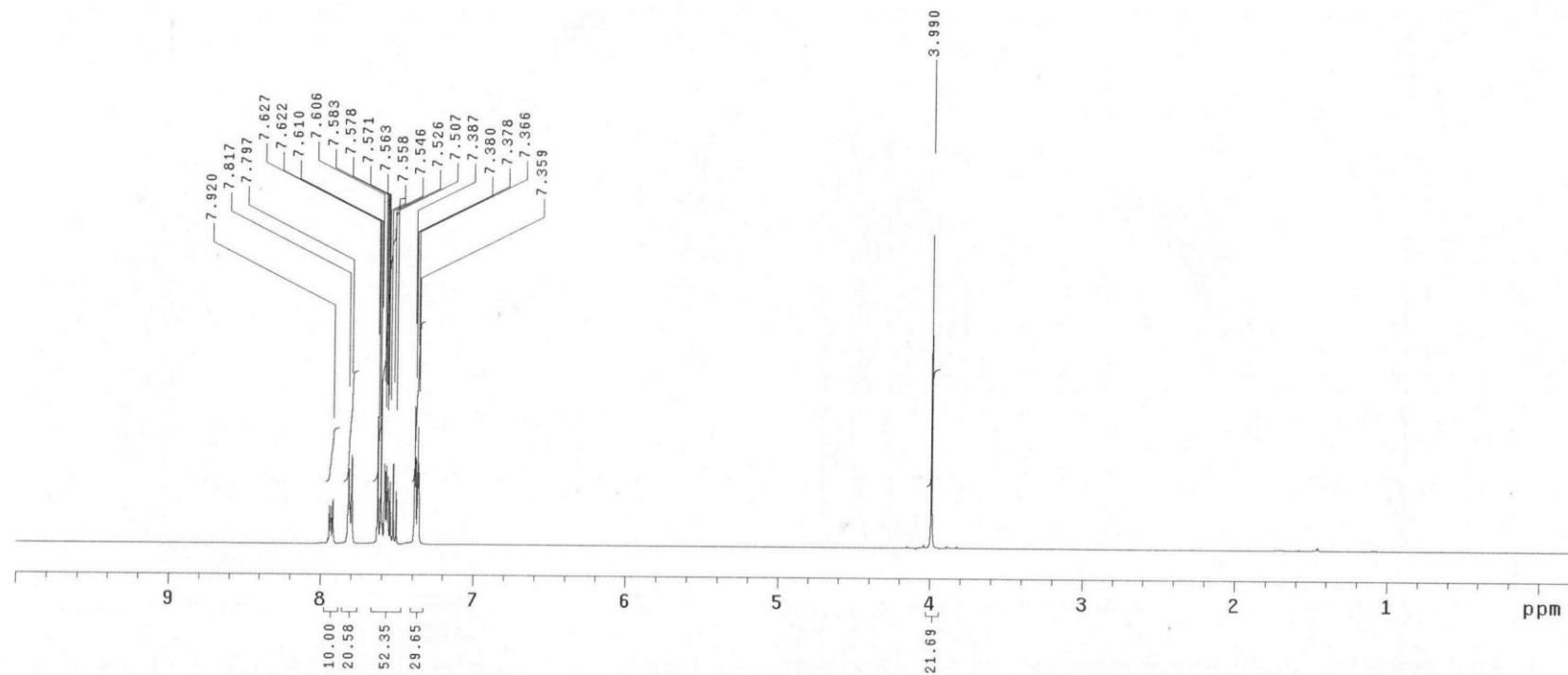
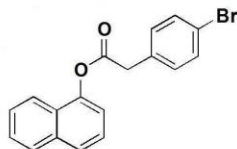
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Date: Apr 10 2018

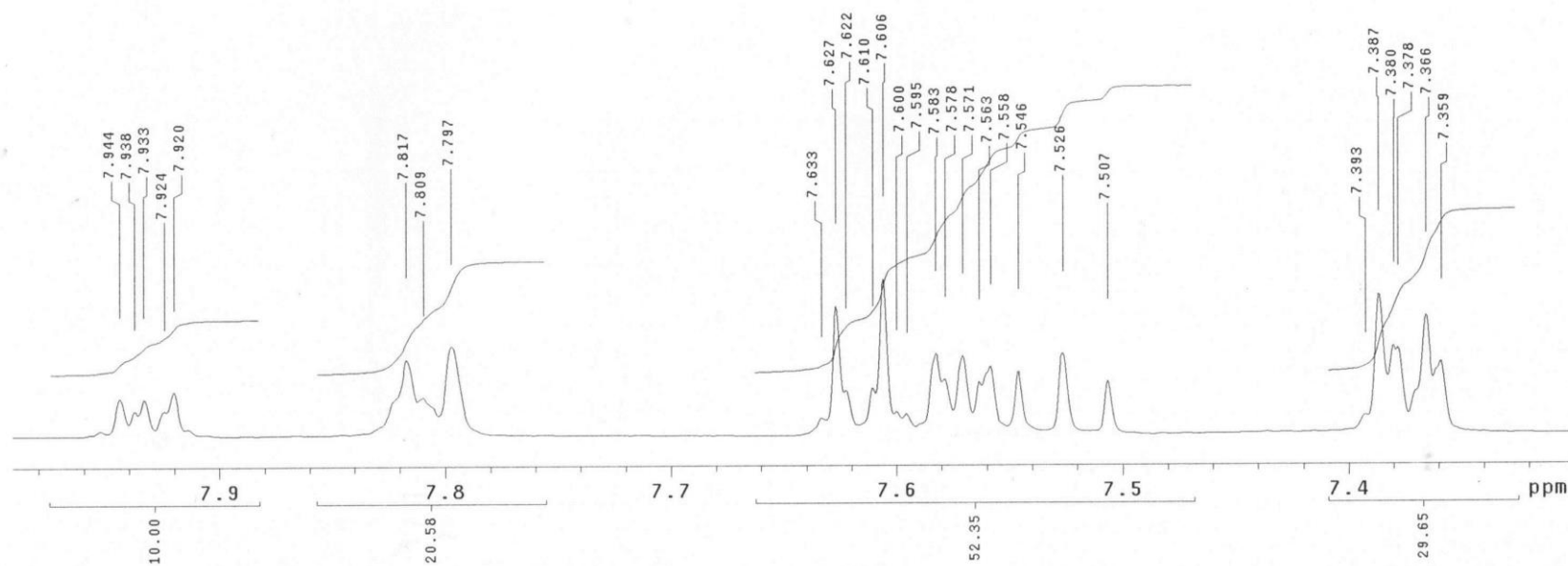
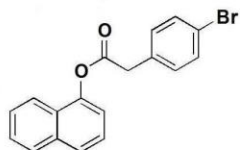
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3h (¹H-NMR spectral data-2)



Compound 3h (¹³C-NMR spectral data)

YT1070409

Pulse Sequence: s2pu1

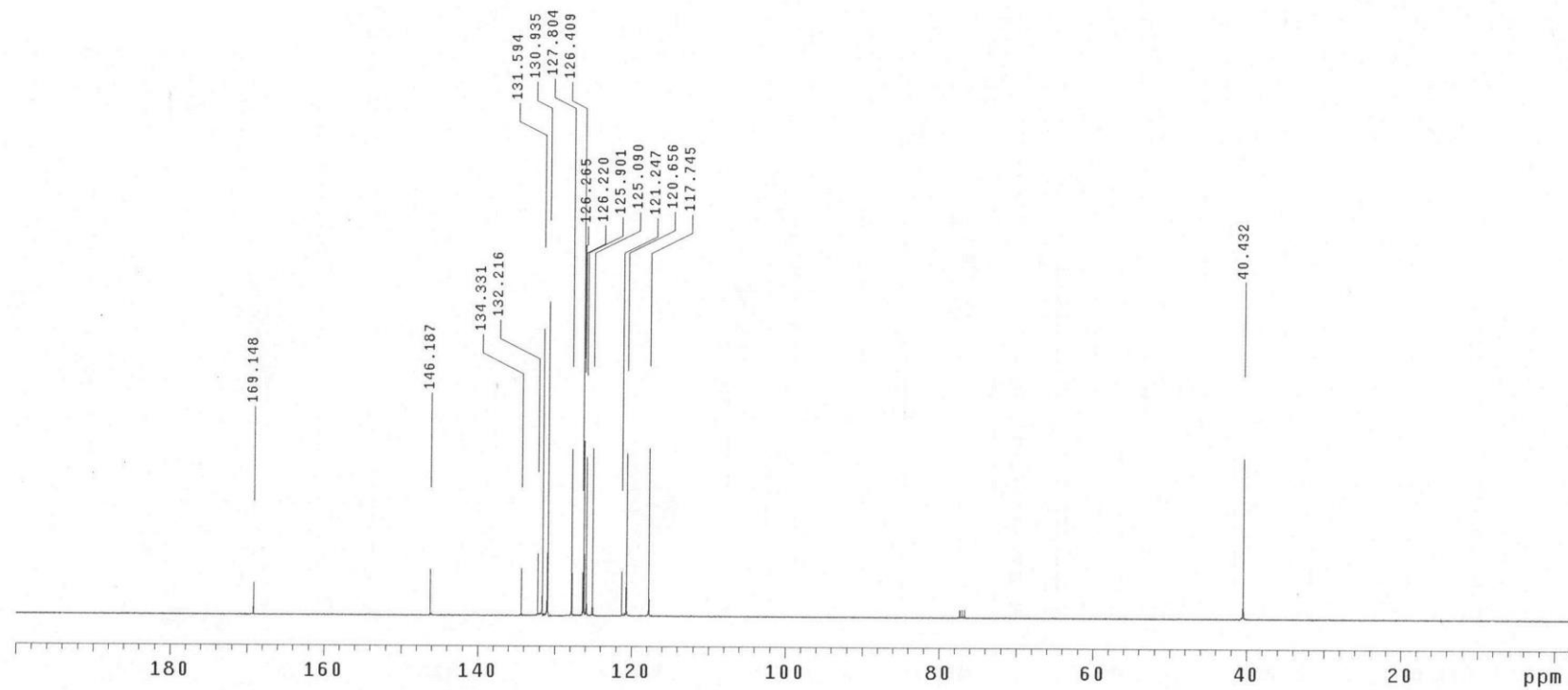
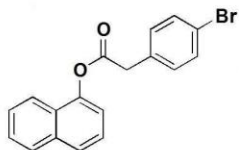
UNITYplus-400 "unity400"

Date: Apr 10 2018

Solvent: CDCl₃

Ambient temperature

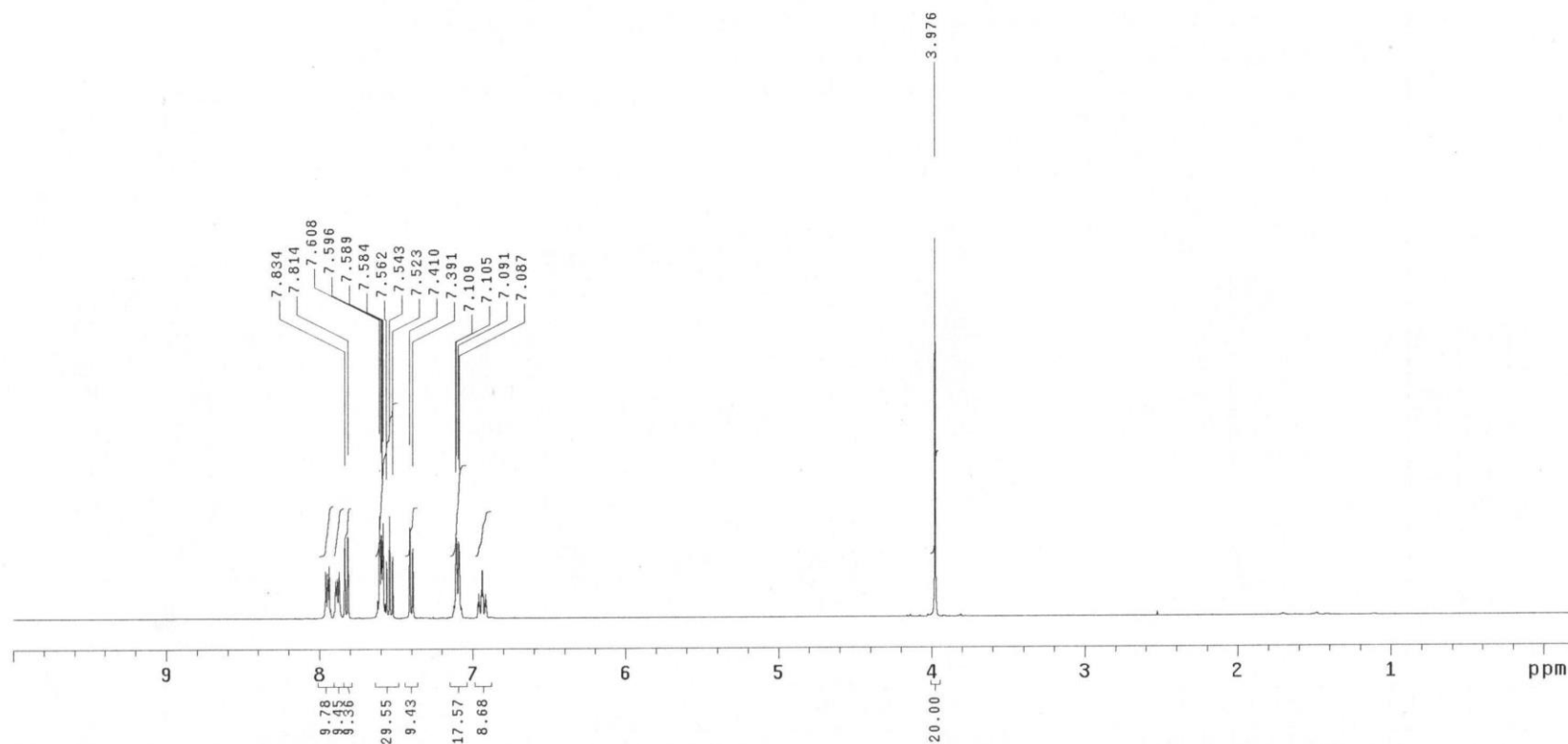
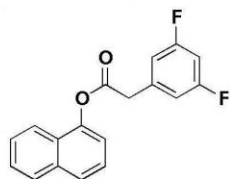
Total 272 repetitions



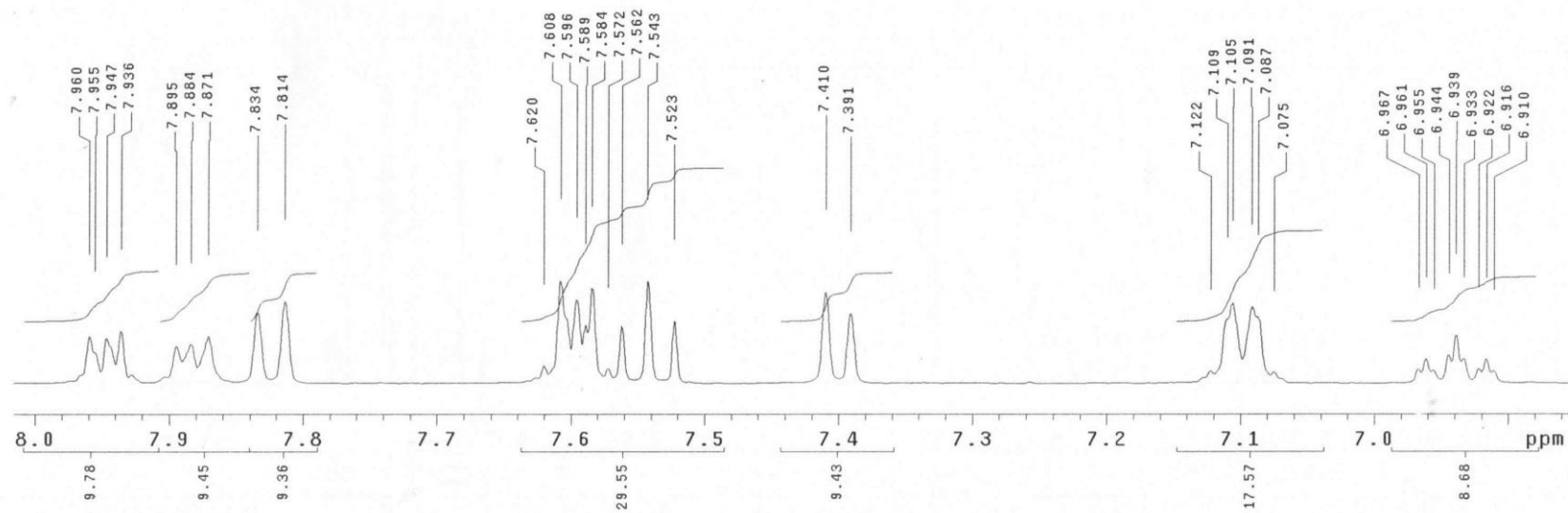
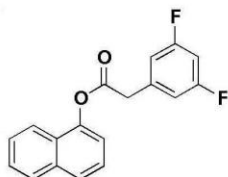
Compound 3i (¹H-NMR spectral data-1)

SMPPA2F

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Apr 16 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 3i (¹H-NMR spectral data-2)



Compound 3i (¹³C-NMR spectral data)

SMPPA2F

Pulse Sequence: s2pul

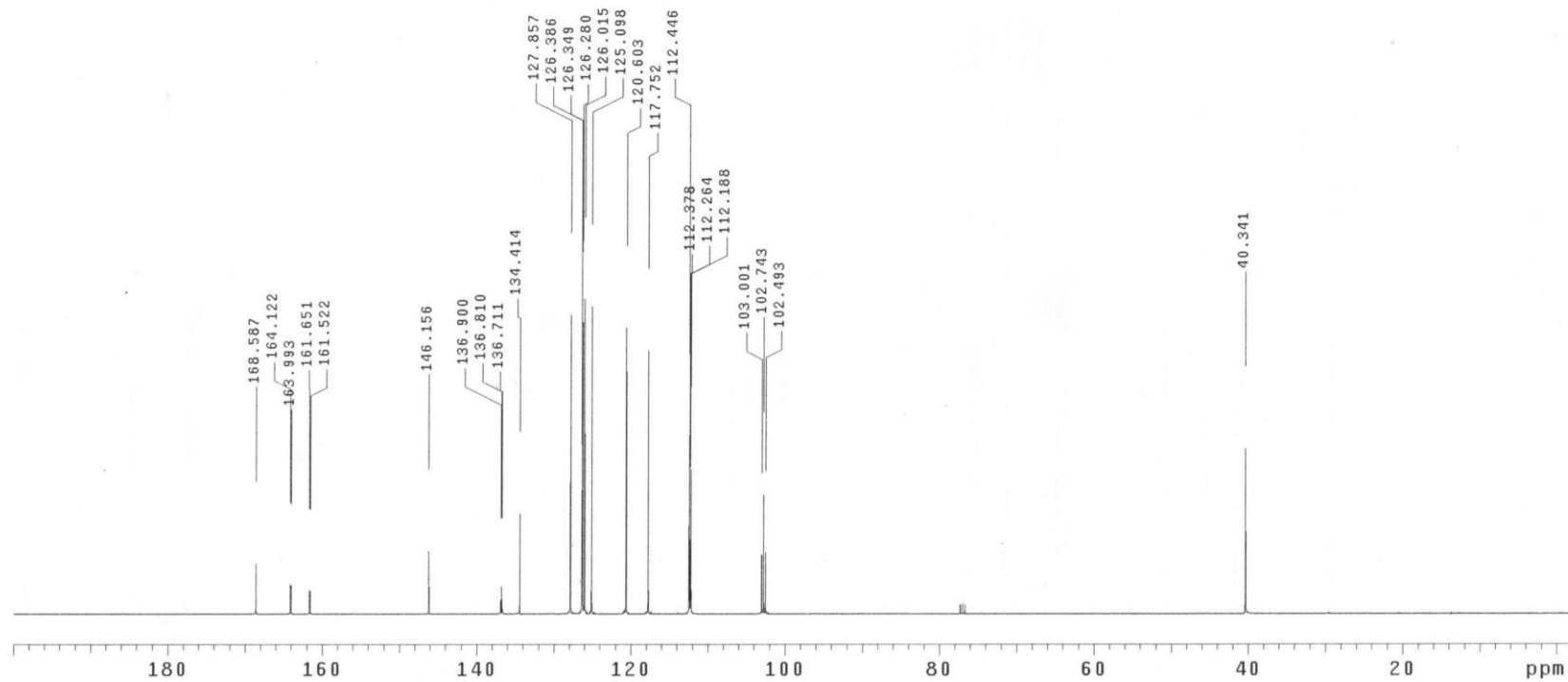
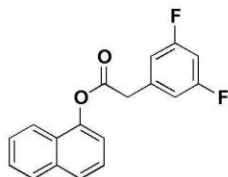
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Date: Apr 16 2018

Solvent: CDCl₃

Ambient temperature

Total 32000 repetitions



Compound 3j (¹H-NMR spectral data)

SHPPACF3

Pulse Sequence: s2pu1

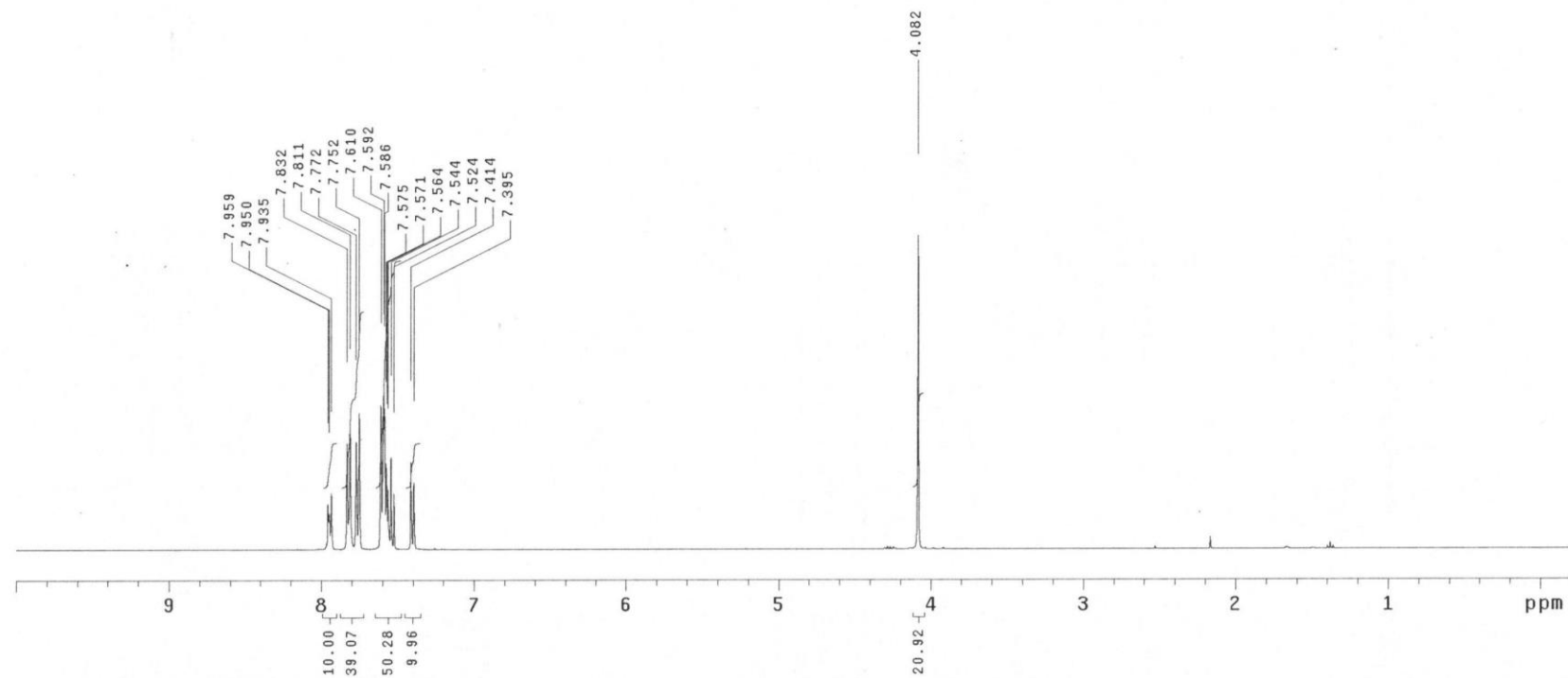
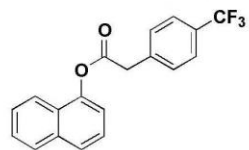
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Date: Apr 23 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3j (¹³C-NMR spectral data)

SHPPACF3

Pulse Sequence: s2pu1

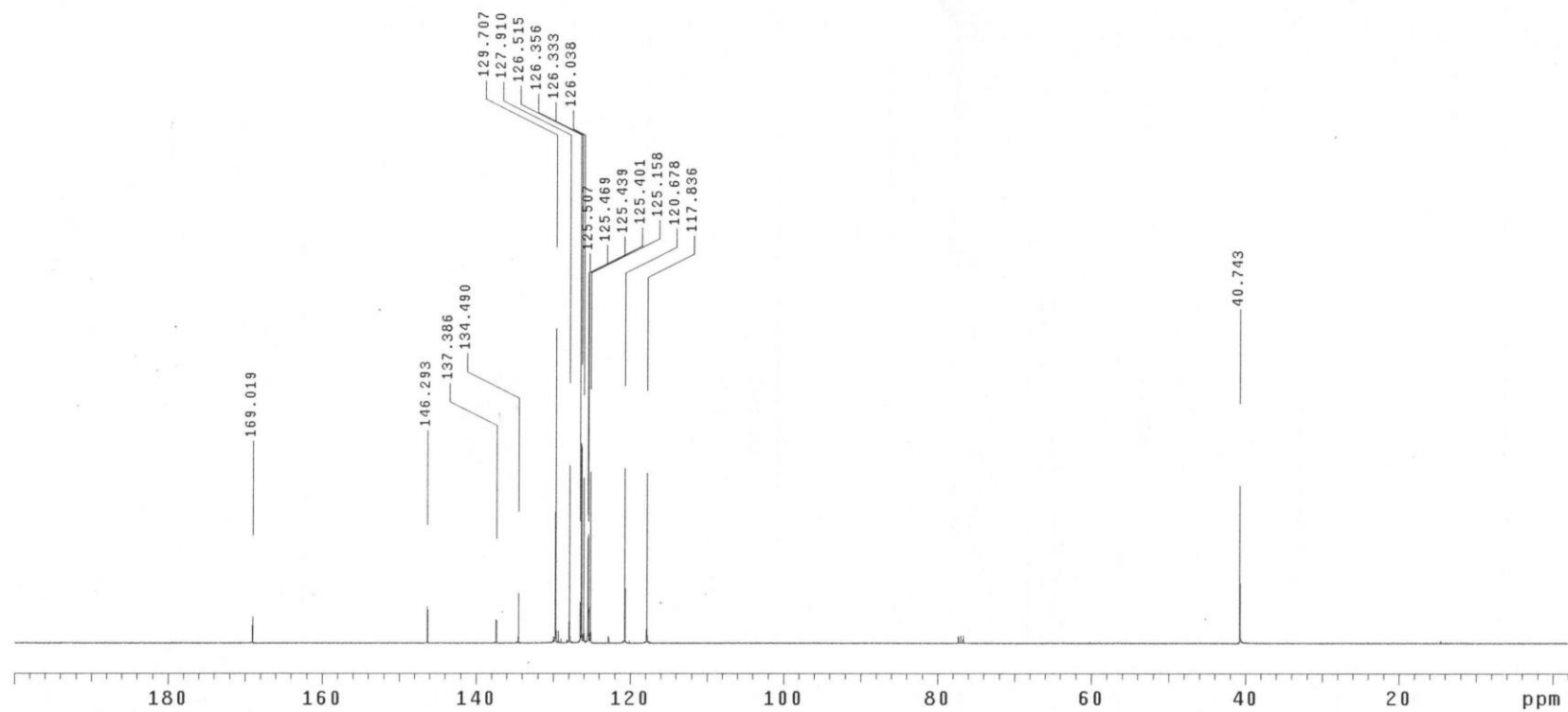
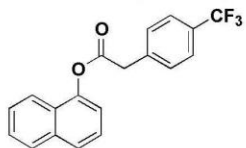
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Date: Apr 23 2018

Solvent: CDCl₃

Ambient temperature

Total 432 repetitions



Compound 3k (¹H-NMR spectral data)

SHNAPPA

Pulse Sequence: s2pu1

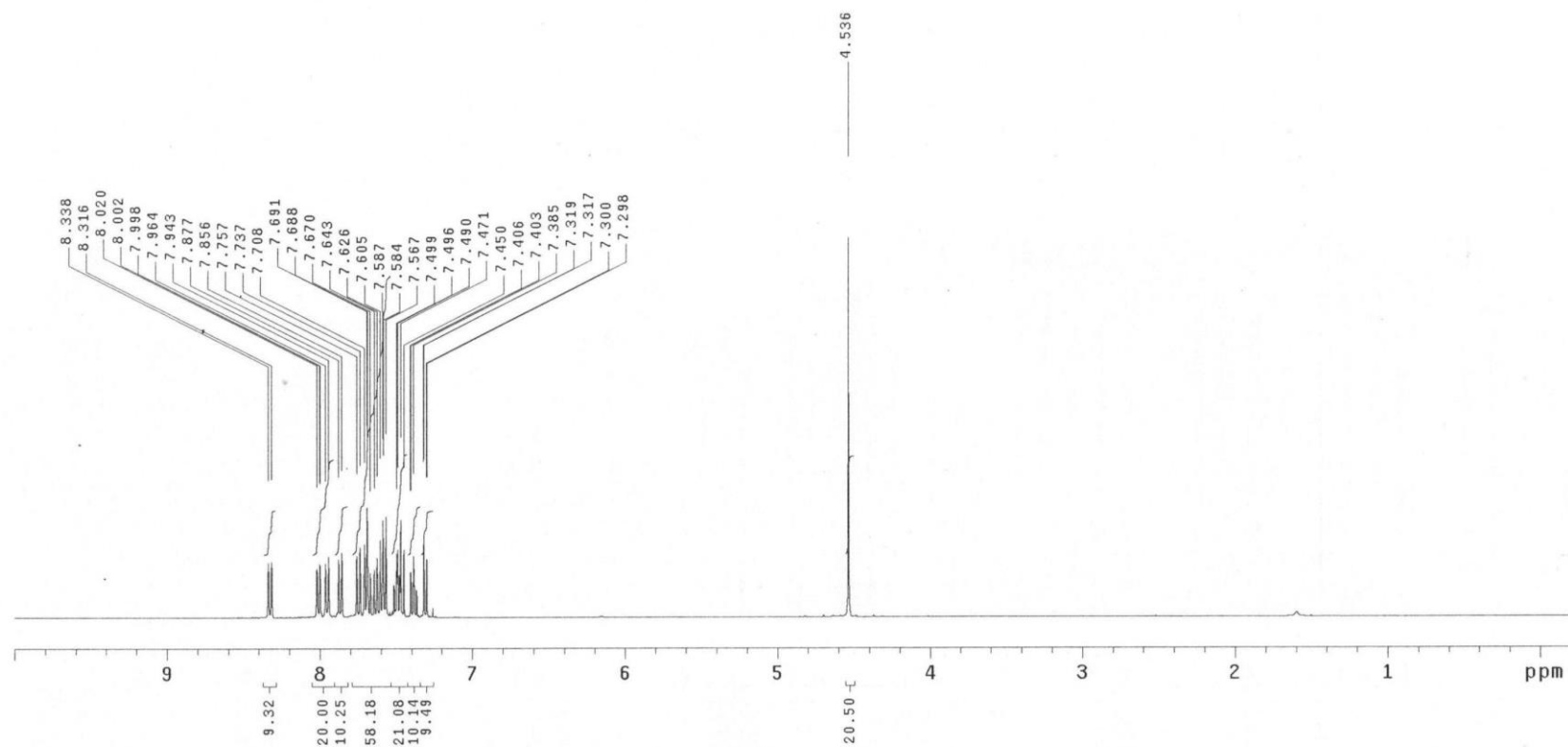
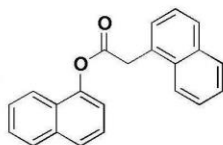
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Date: Apr 26 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3k (¹³C-NMR spectral data)

SHNAPPA

Pulse Sequence: s2pu1

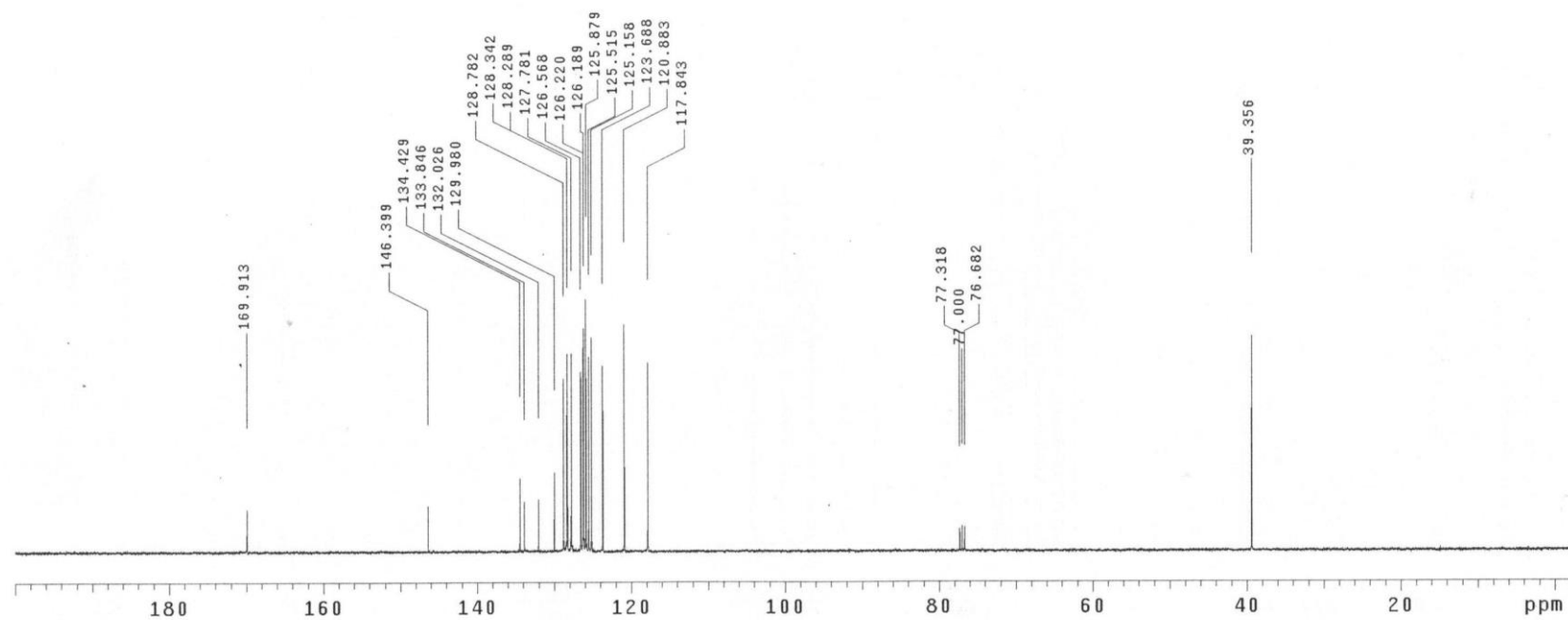
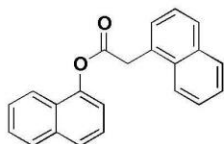
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Date: Apr 26 2018

Solvent: CDCl₃

Ambient temperature

Total 176 repetitions



Compound 3l (¹H-NMR spectral data)

CYNACF3

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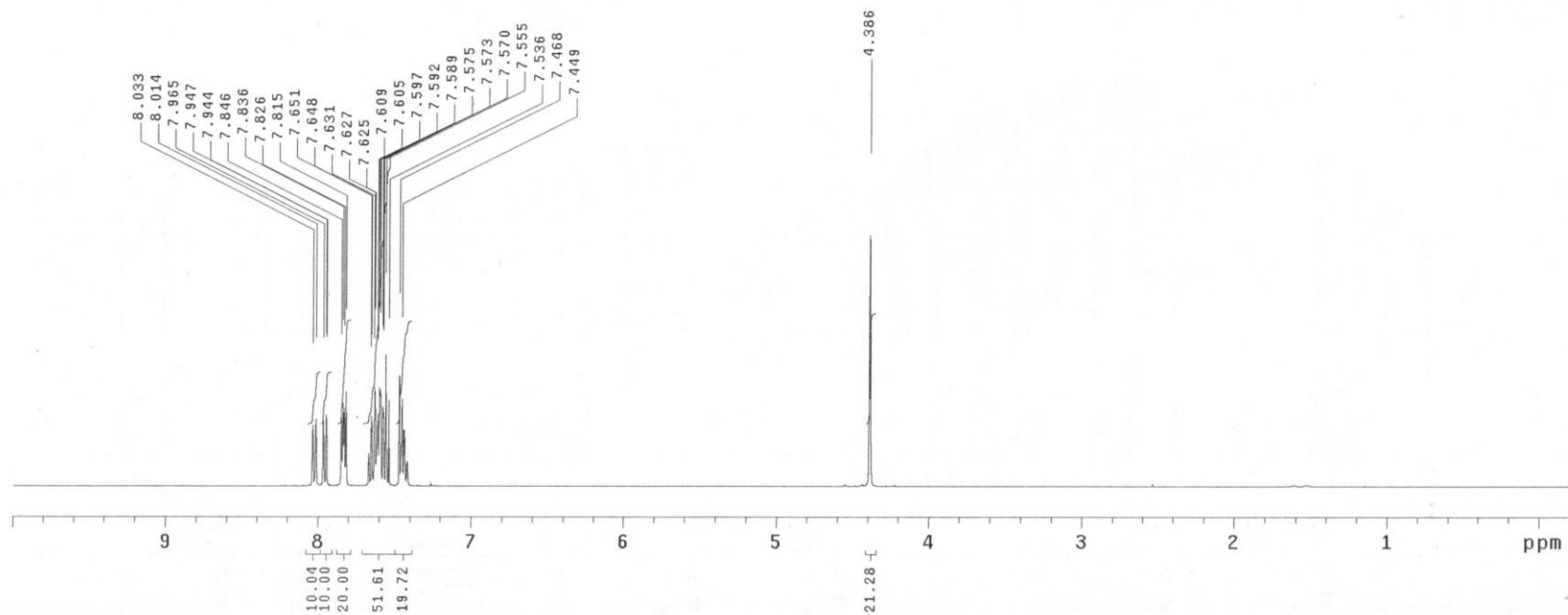
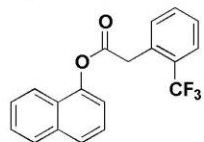
UNITYplus-400 "unity400"

Date: Jul 17 2019

Solvent: CDC13

Ambient temperature

Total 32 repetitions



Compound 3I (¹³C-NMR spectral data)

CYNACF3

Pulse Sequence: s2pu1

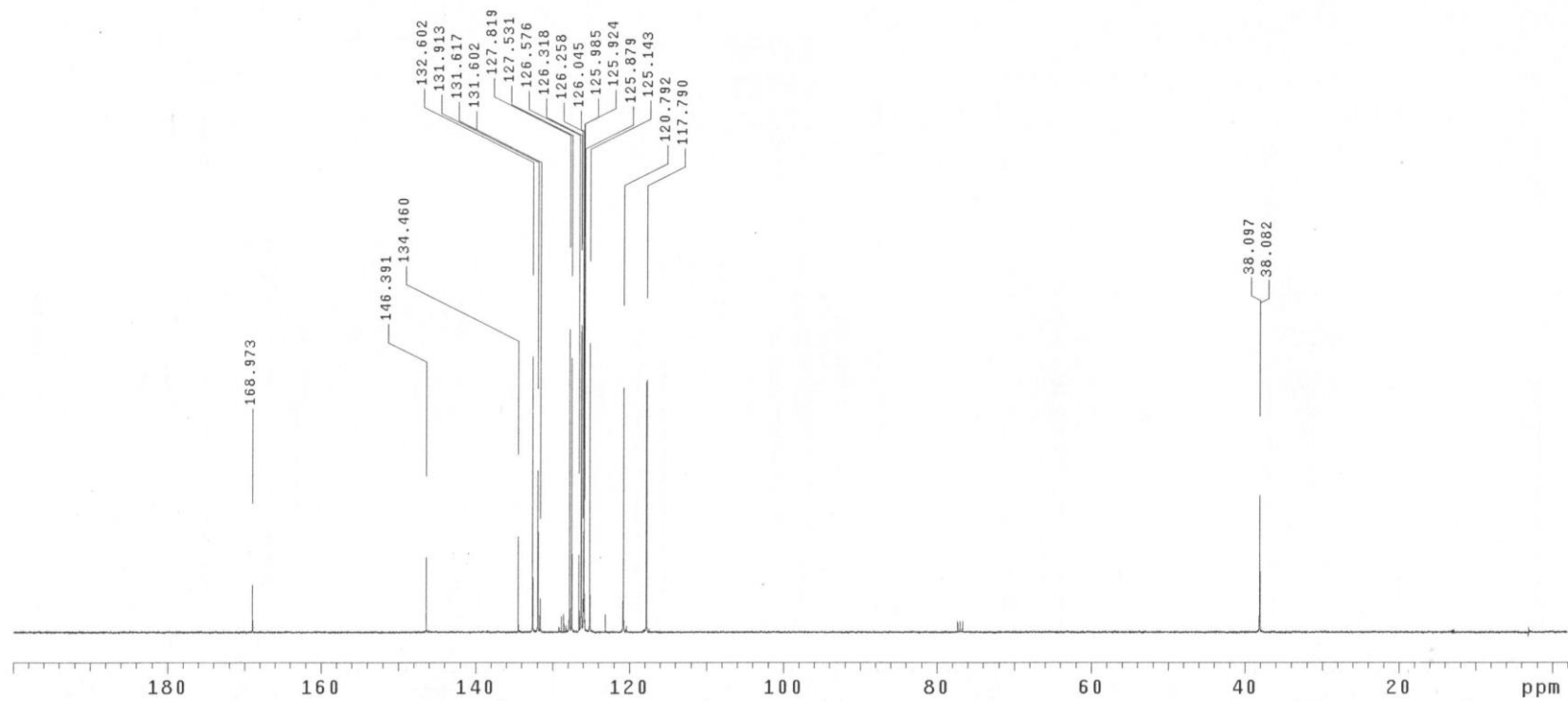
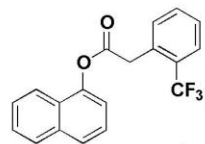
UNITYplus-400 "unity400"

Date: Jul 17 2019

Solvent: CDCl₃

Ambient temperature

Total 128 repetitions



Compound 3m (¹H-NMR spectral data)

SHPPAPh2MeO

Pulse Sequence: s2pu1

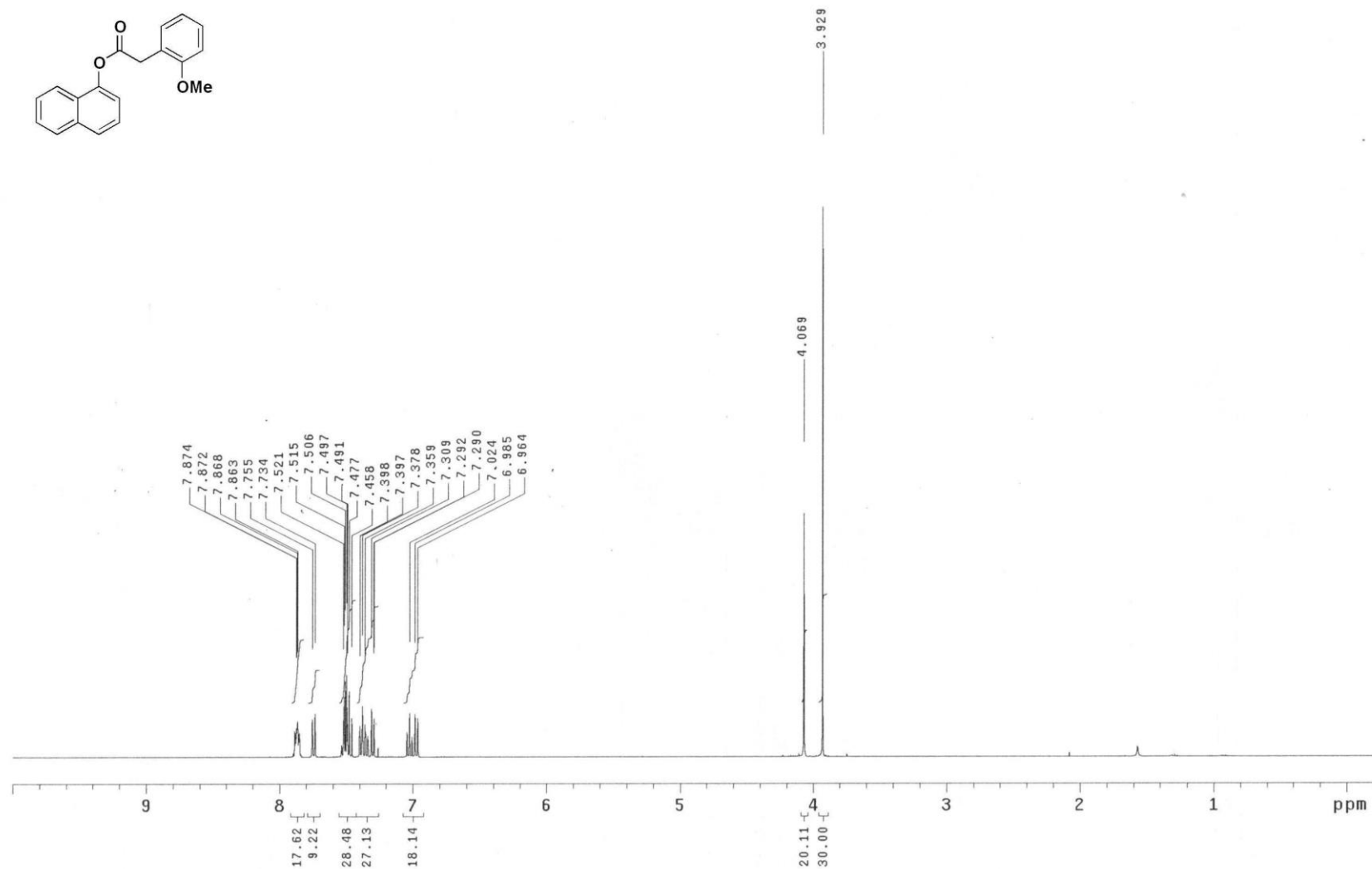
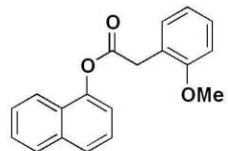
UNITYplus-400 "unity400"

Date: May 3 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3m (¹³C-NMR spectral data)

SHPPAPh2MeO

Pulse Sequence: s2pu1

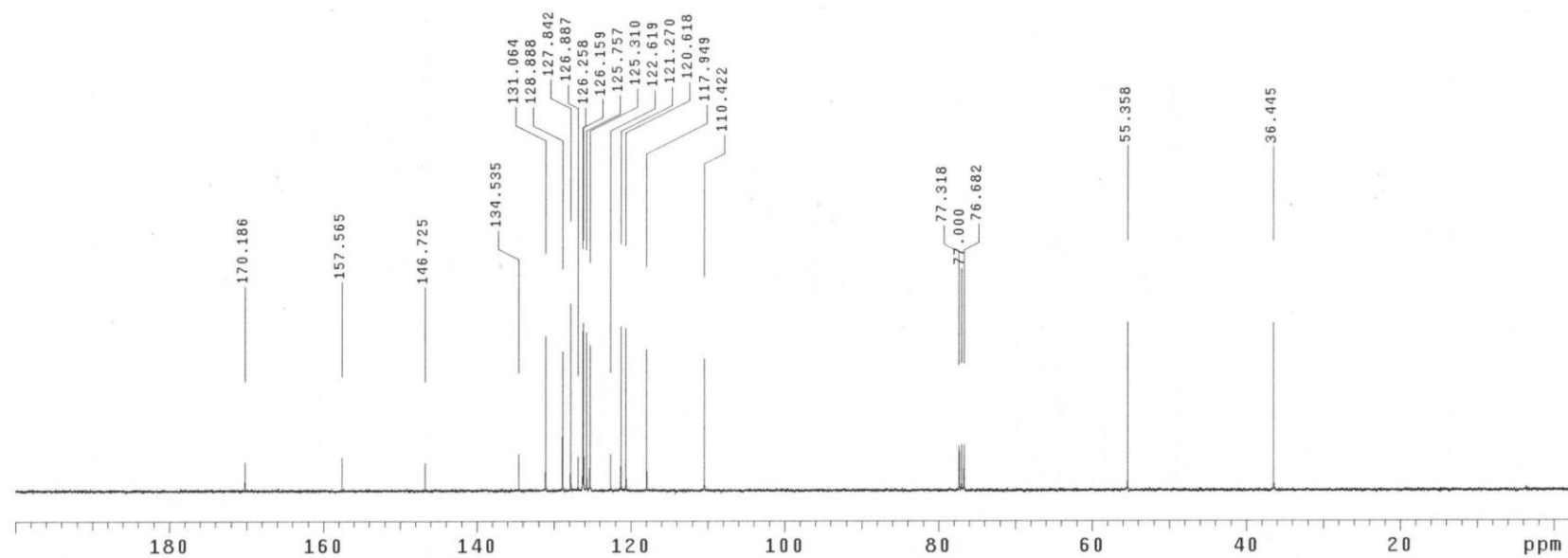
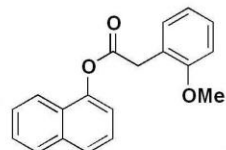
UNITYplus-400 "unity400"

Date: May 3 2018

Solvent: CDCl₃

Ambient temperature

Total 528 repetitions



Compound 3n (¹H-NMR spectral data)

YTOMePh

Pulse Sequence: s2pu1

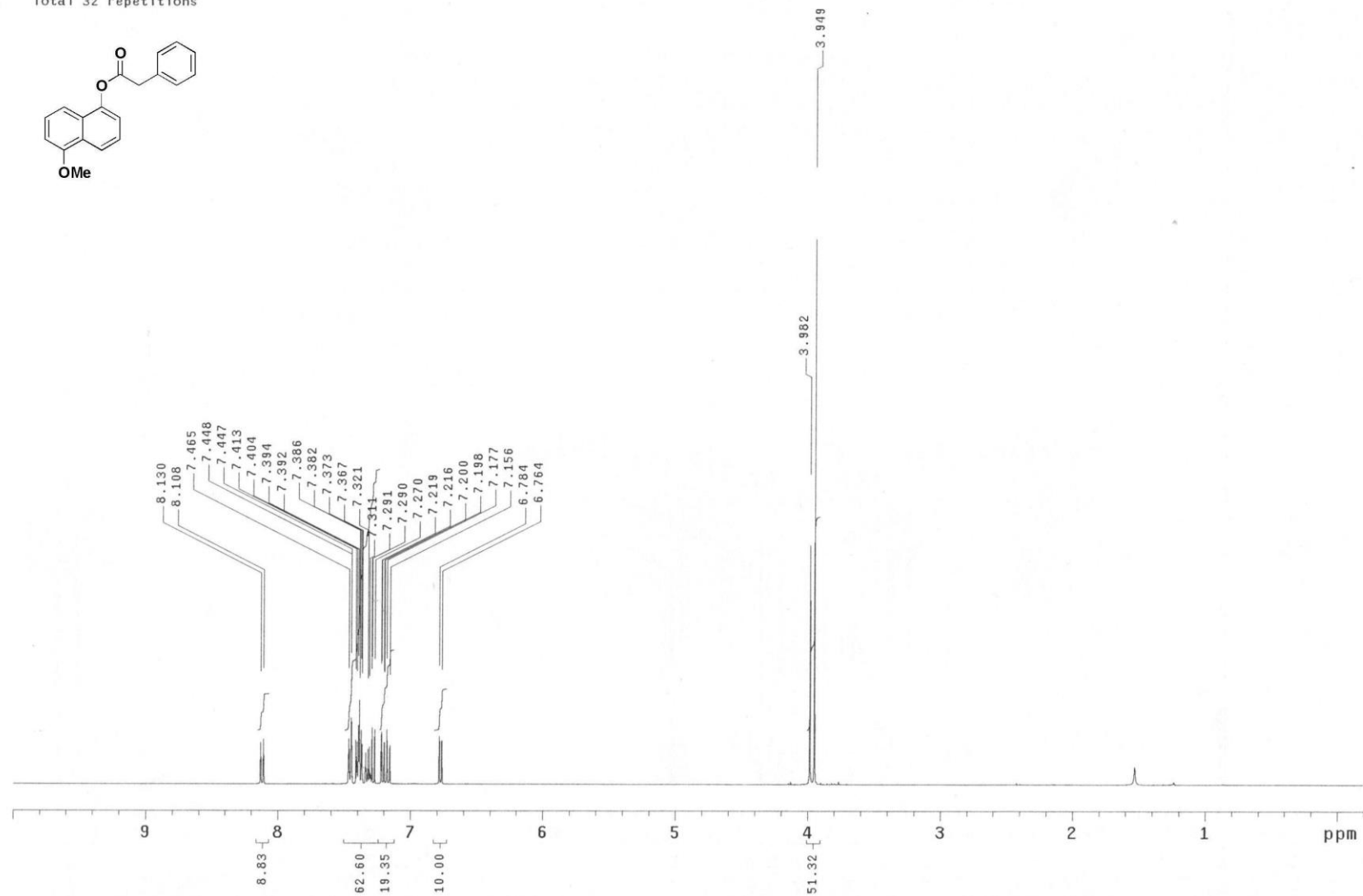
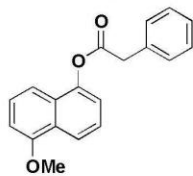
UNITYplus-400 "unity400"

Date: May 31 2018

Solvent: CDCl₃

Ambient temperature

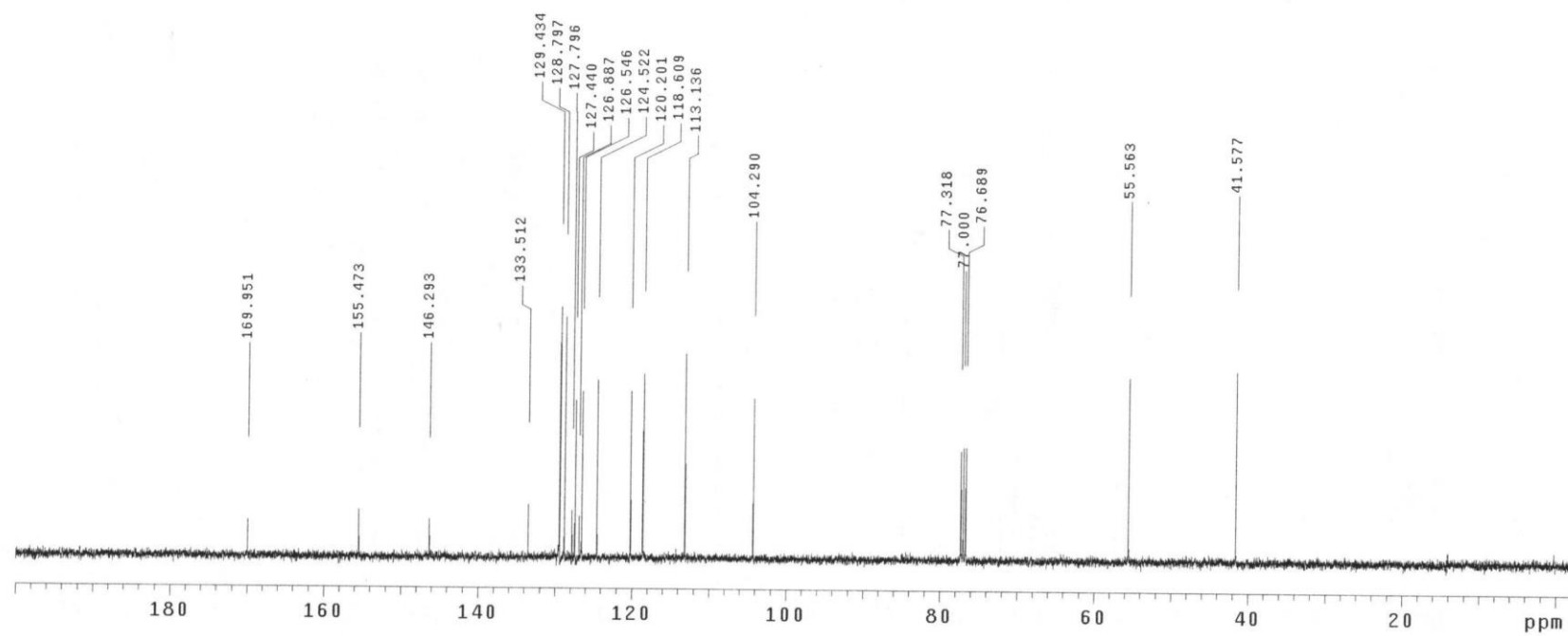
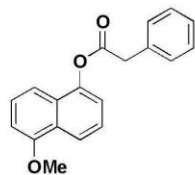
Total 32 repetitions



Compound 3n (¹³C-NMR spectral data)

YTOmePh

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: May 31 2018
Solvent: CDCl₃
Ambient temperature
Total 368 repetitions



Compound 3o (¹H-NMR spectral data-1)

SH40MePPA

Pulse Sequence: s2pu1

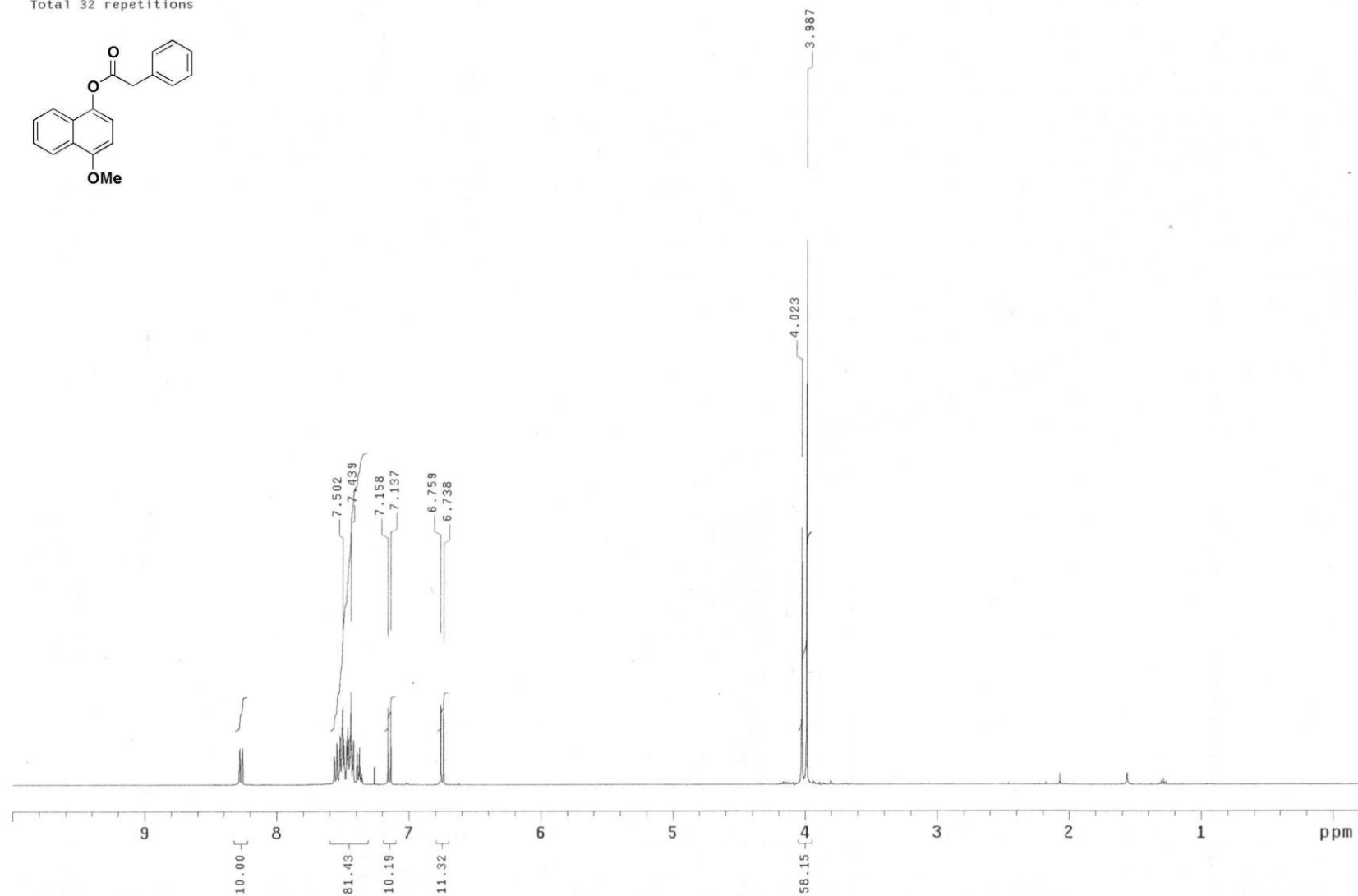
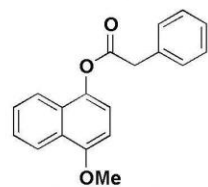
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Date: Jul 26 2018

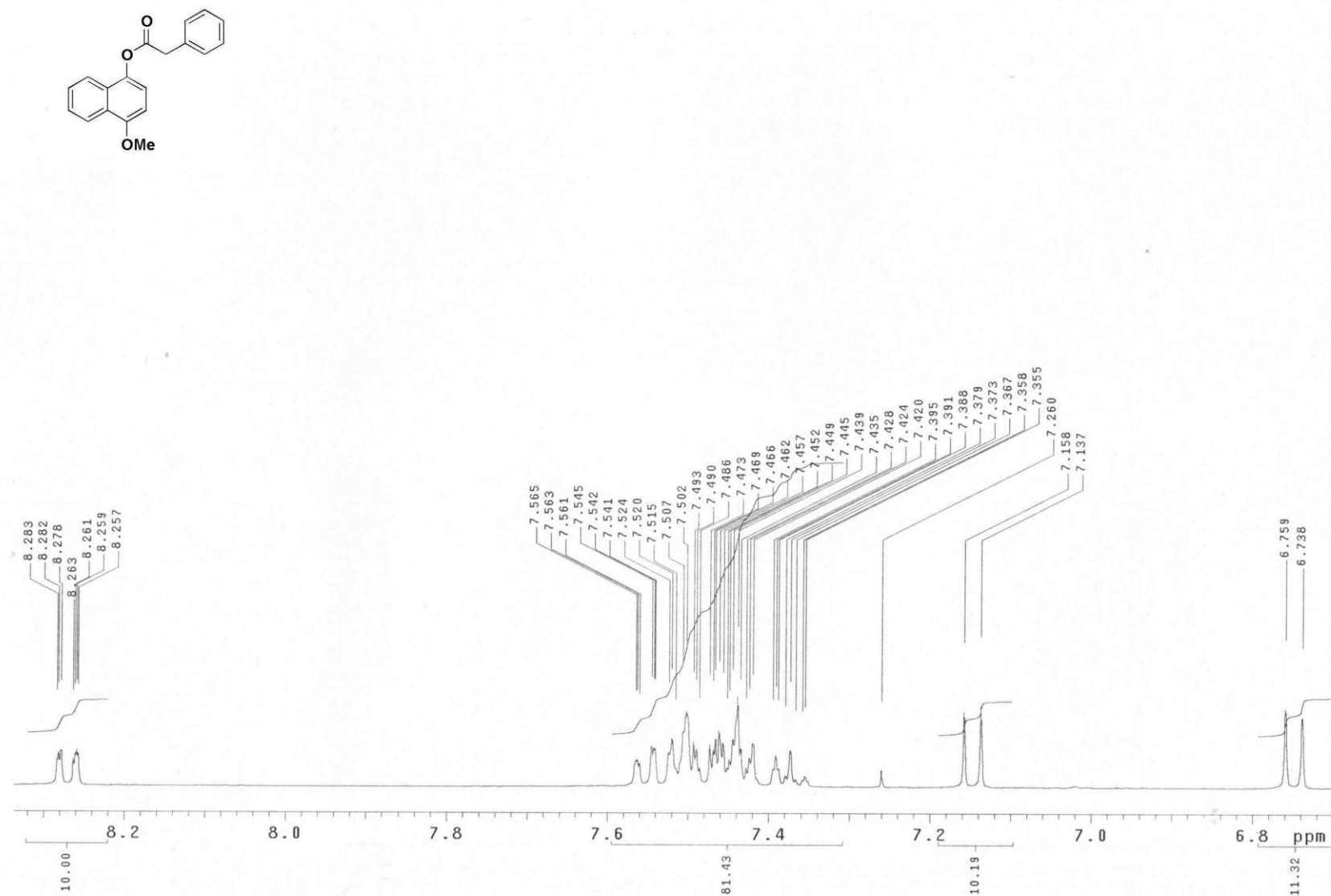
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3o (¹H-NMR spectral data-2)



Compound 3o (¹³C-NMR spectral data)

SH40MePPA

Pulse Sequence: s2pu1

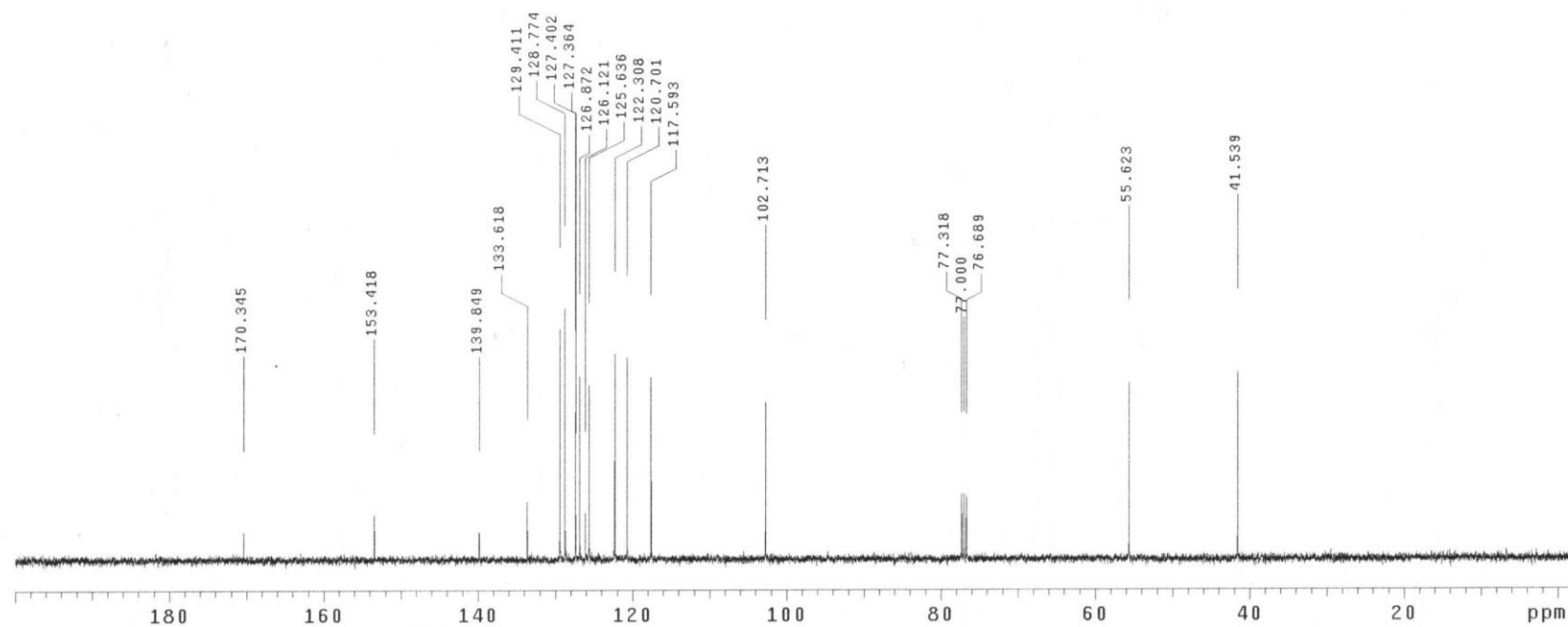
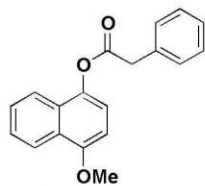
UNITYplus-400 "unity400"

Date: Jul 26 2018

Solvent: CDCl₃

Ambient temperature

Total 128 repetitions



Compound 3p (¹H-NMR spectral data-1)

SH4C1PPA

Pulse Sequence: s2pu1

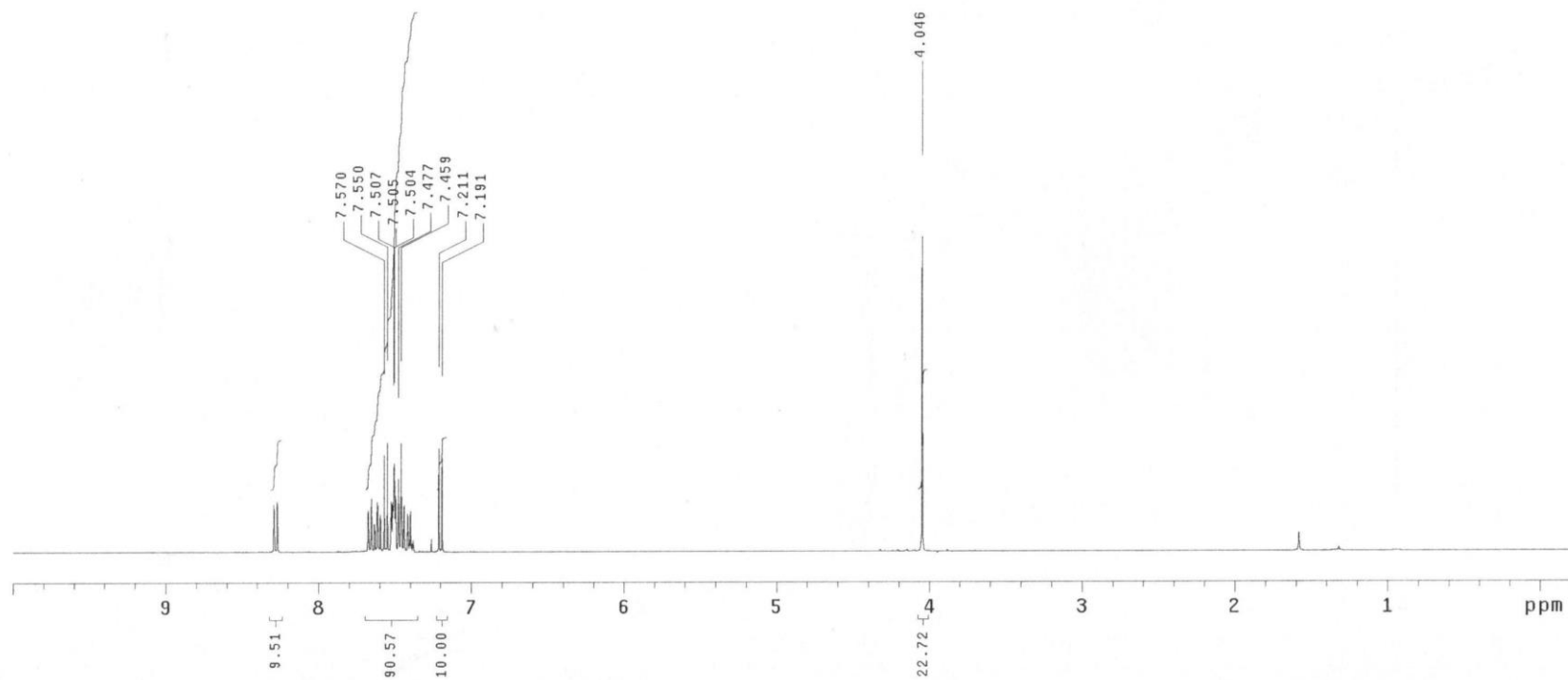
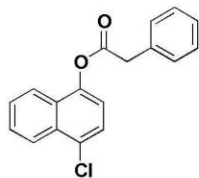
UNITYplus-400 "unity400"

Date: Jul 23 2018

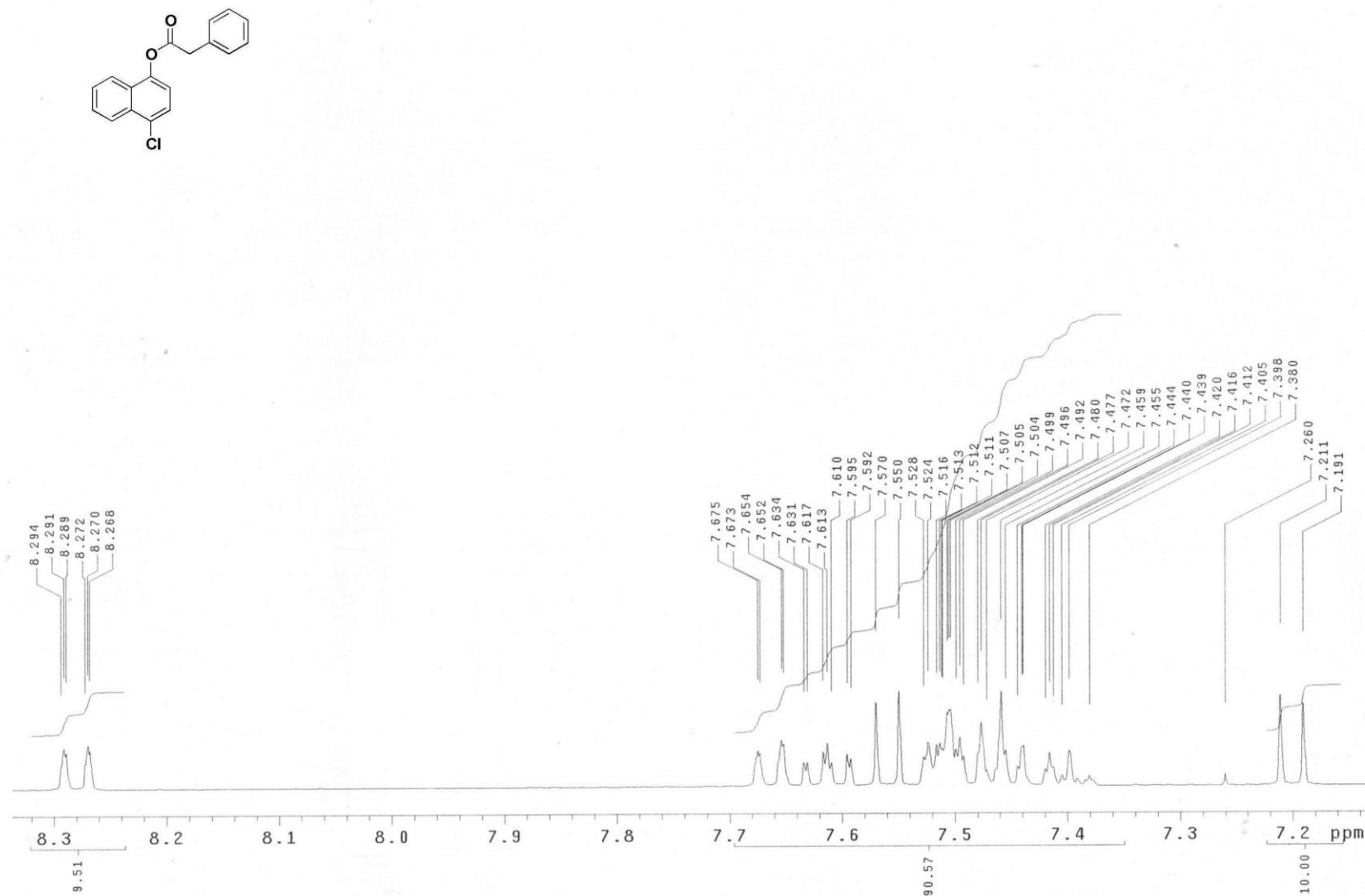
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3p (¹H-NMR spectral data-2)



Compound 3p (¹³C-NMR spectral data)

SH4C1PPA

Pulse Sequence: s2pu1

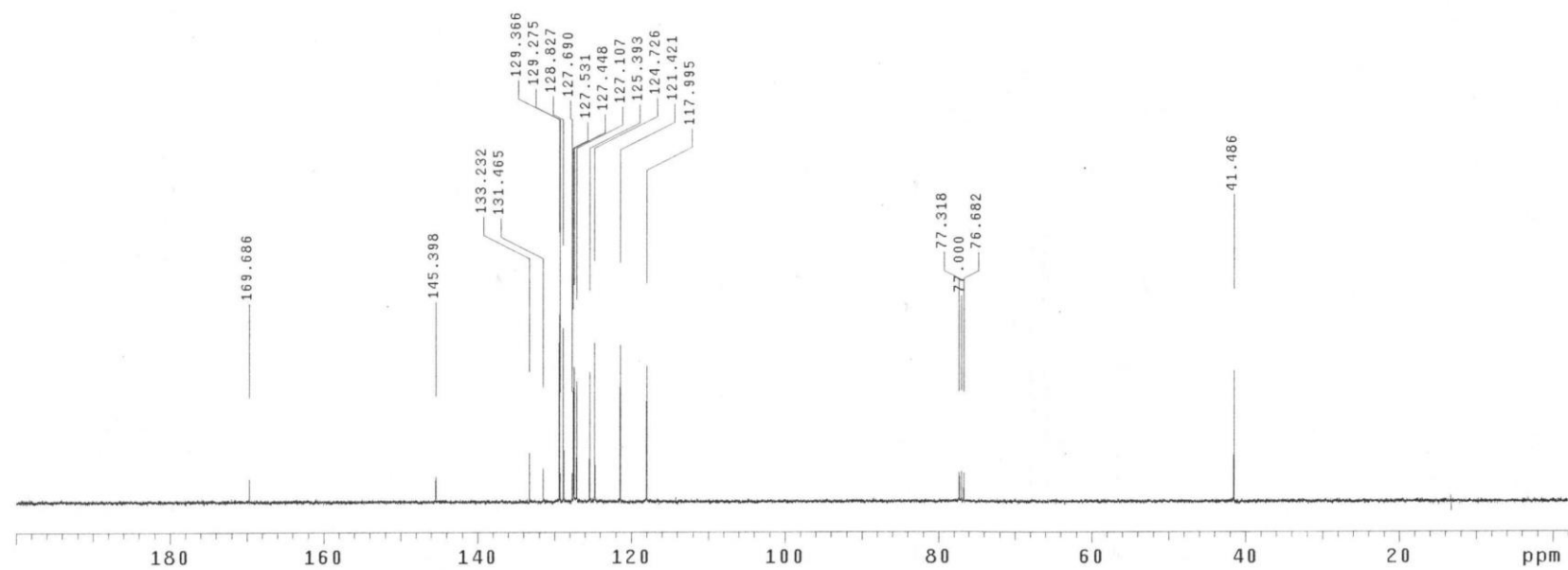
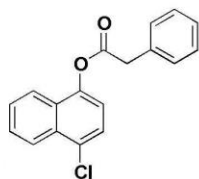
UNITYplus-400 "unity400"

Date: Jul 23 2018

Solvent: CDCl₃

Ambient temperature

Total 112 repetitions



Compound 3q (¹H-NMR spectral data-1)

YT15NAP2

Pulse Sequence: s2pu1

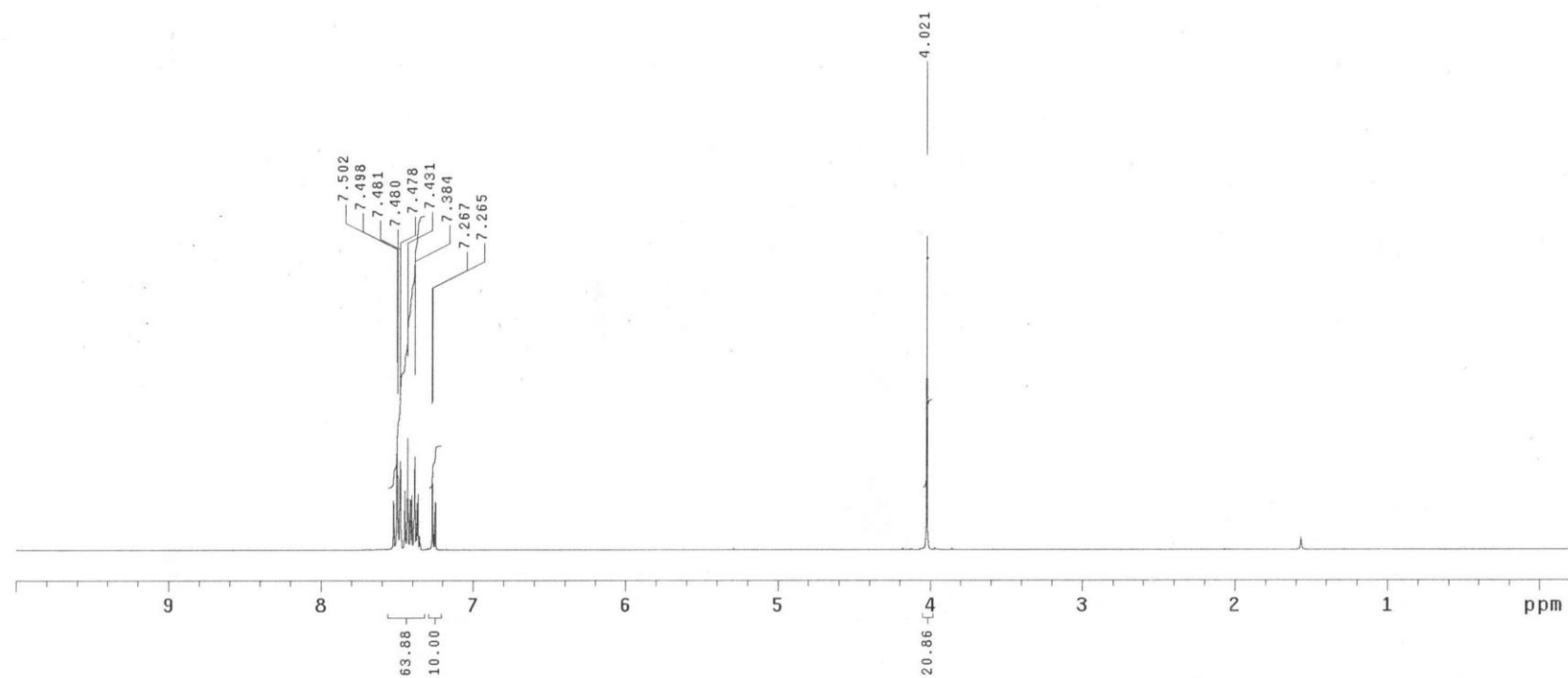
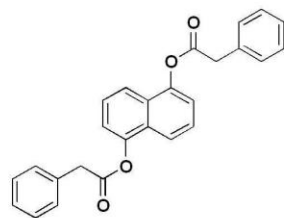
UNITYplus-400 "unity400"

Date: May 10 2018

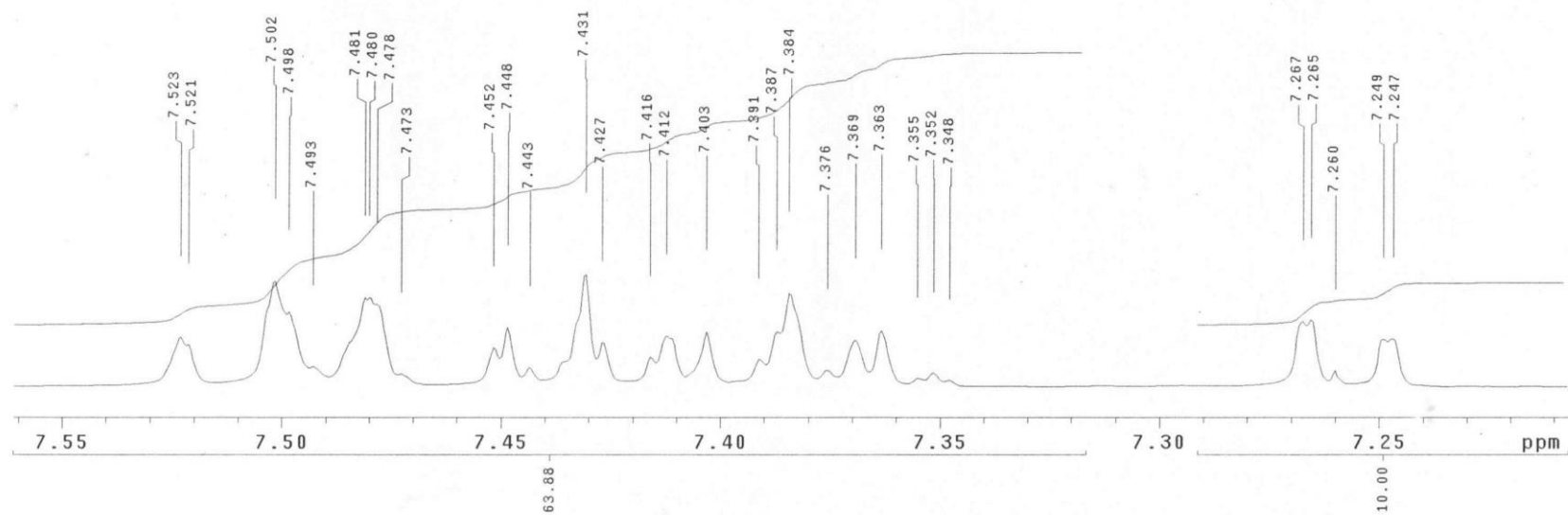
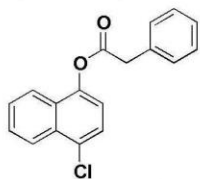
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 3q (¹H-NMR spectral data-2)



Compound 3q (¹³C-NMR spectral data)

YT15NAP2

Pulse Sequence: s2pul

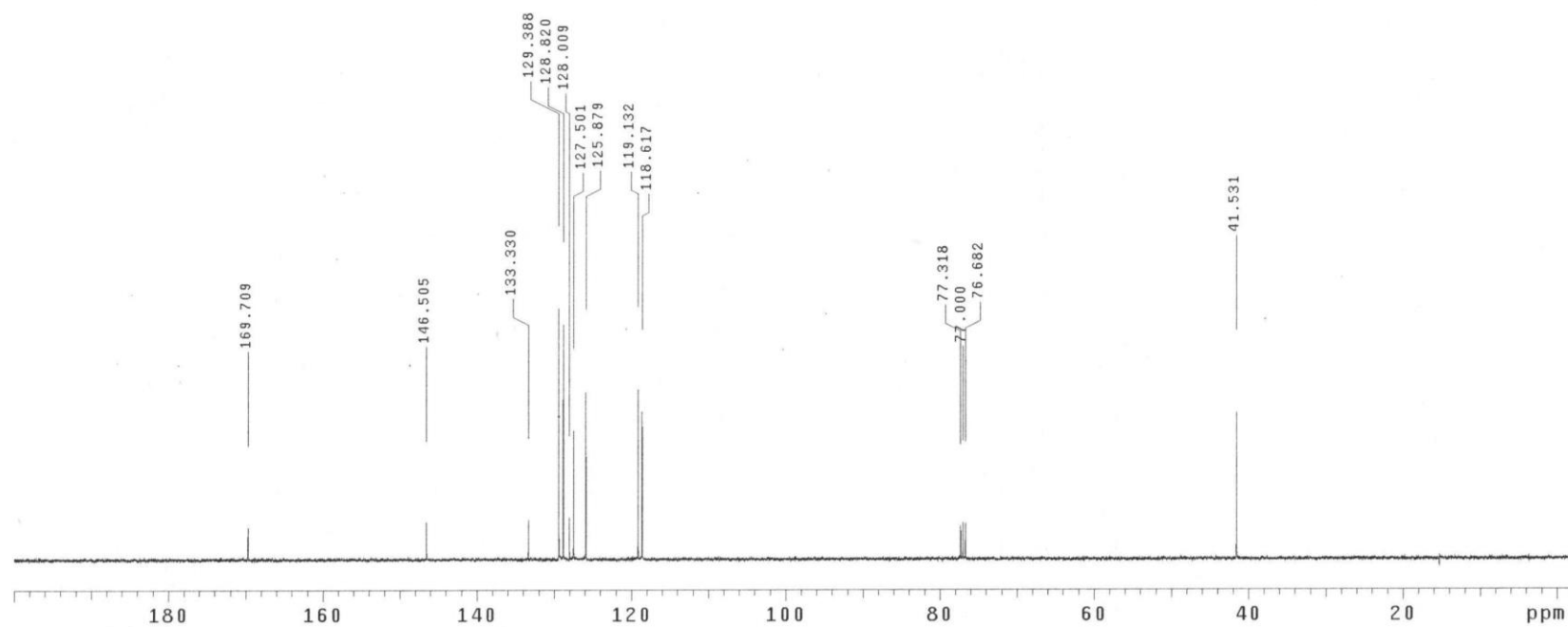
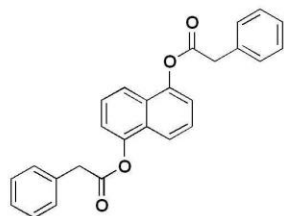
UNITYplus-400 "unity400"

Date: May 10 2018

Solvent: CDCl₃

Ambient temperature

Total 208 repetitions



Compound 4a (¹H-NMR spectral data-1)

YTN1HP

Pulse Sequence: s2pu1

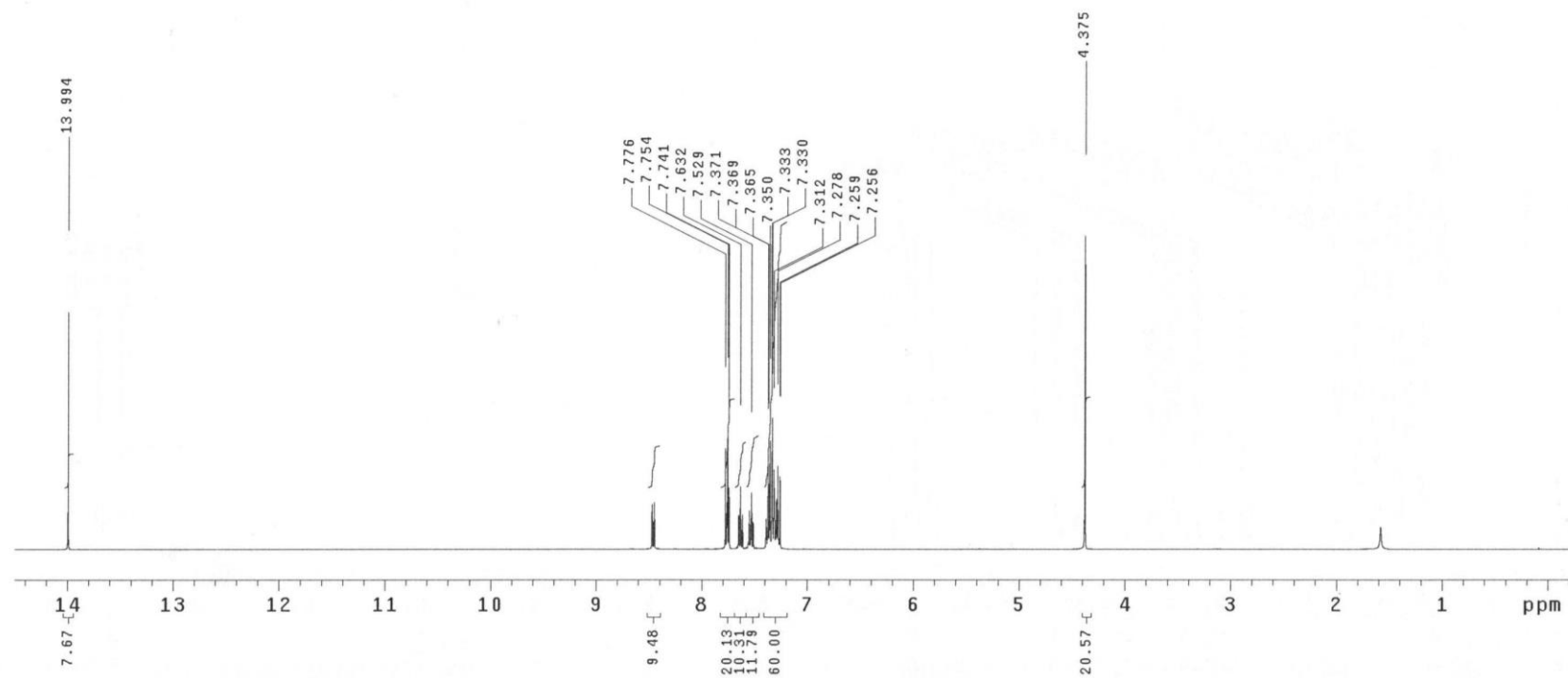
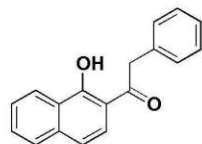
UNITYplus-400 "unity400"

Date: Dec 21 2017

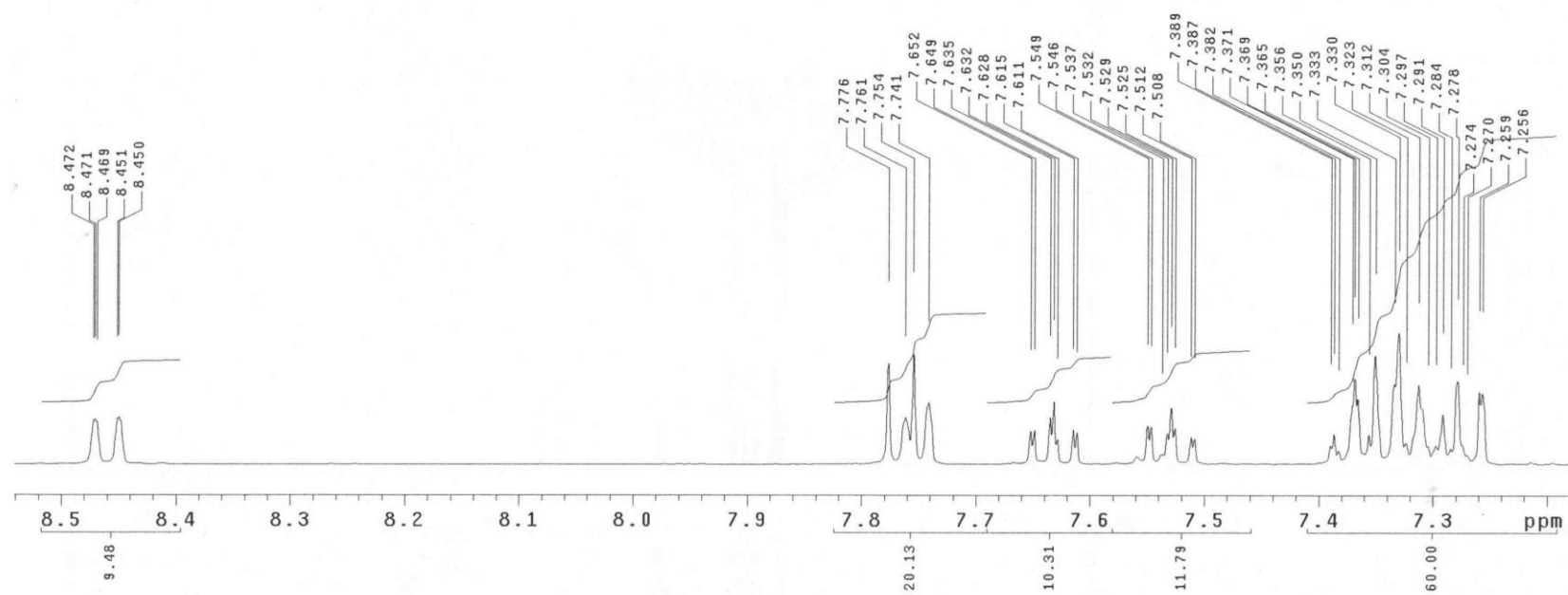
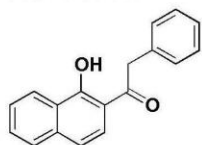
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4a (¹H-NMR spectral data-2)



Compound 4a (¹³C-NMR spectral data)

YTN1HP

Pulse Sequence: s2pul

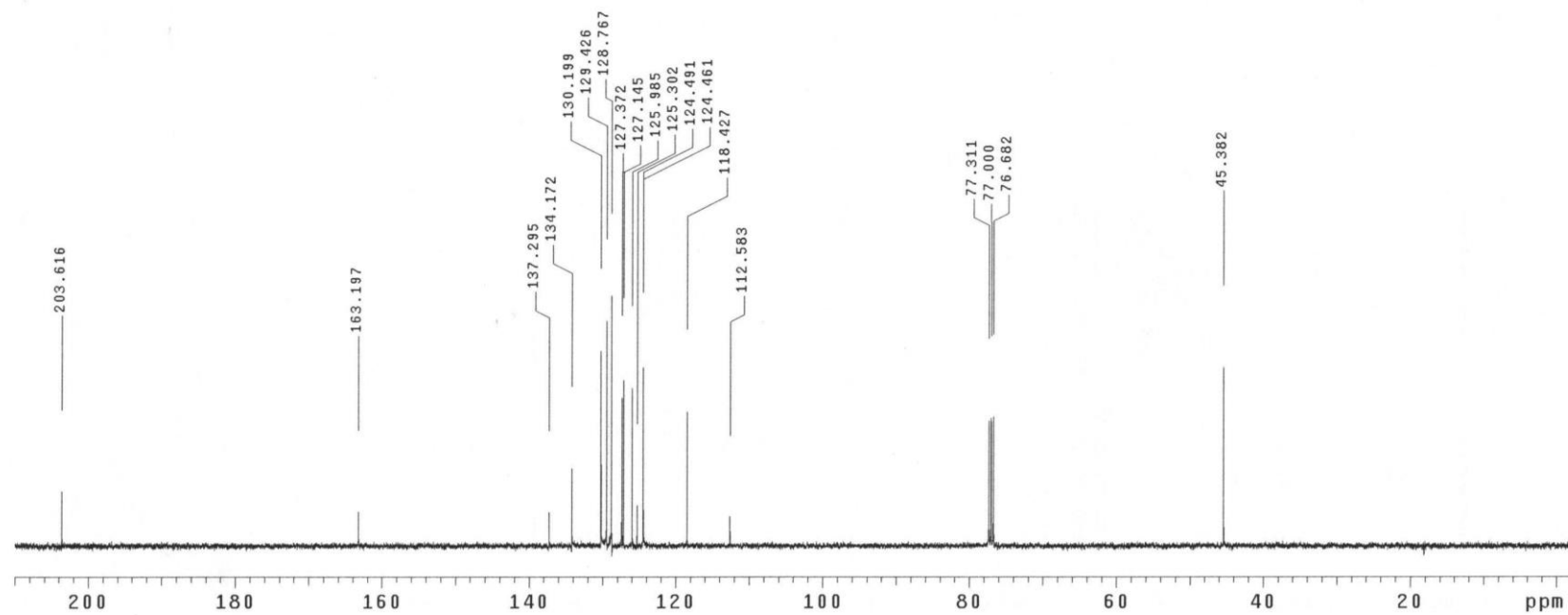
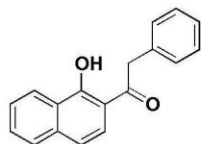
UNITYplus-400 "unity400"

Date: Dec 21 2017

Solvent: CDCl₃

Ambient temperature

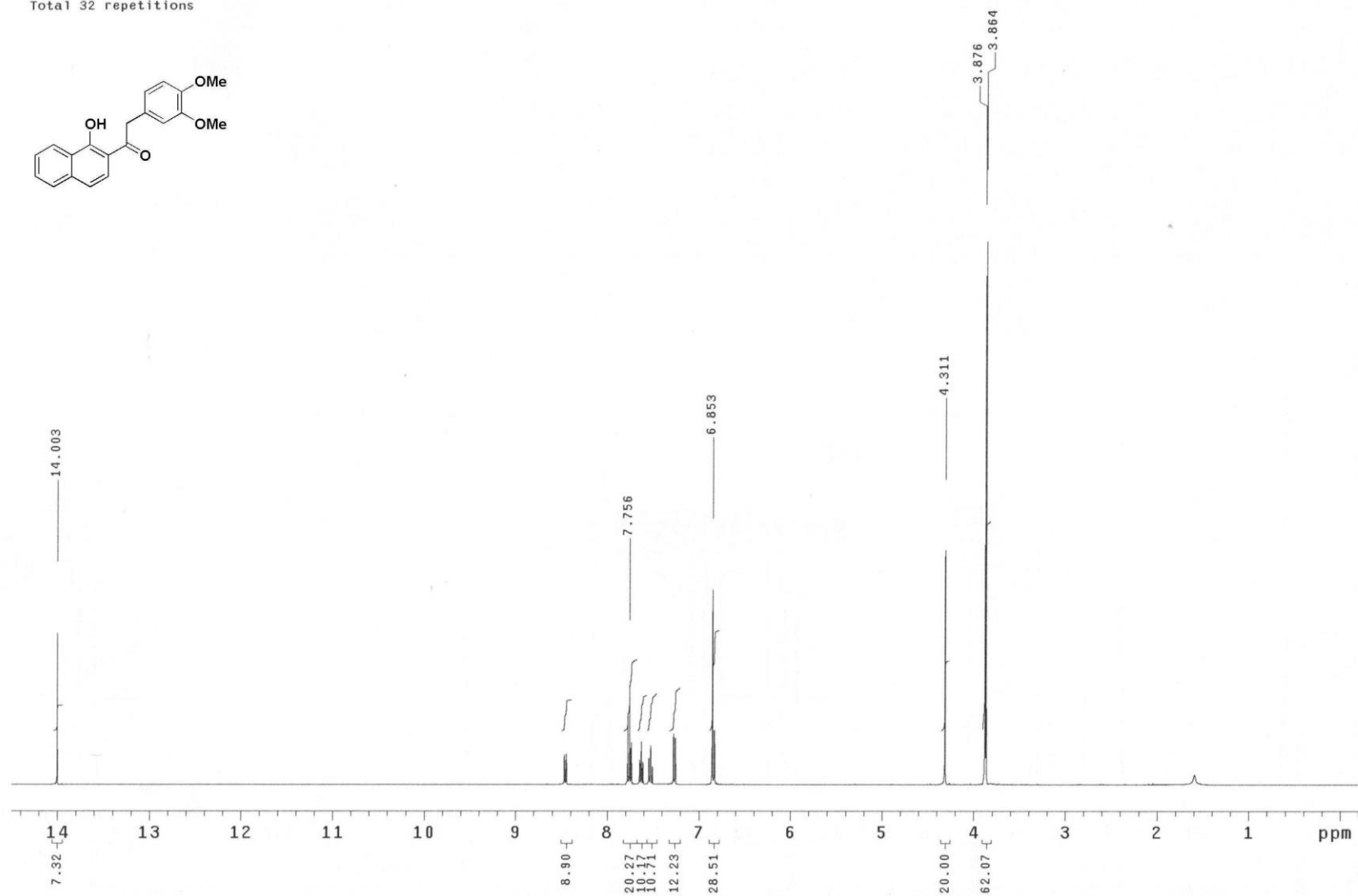
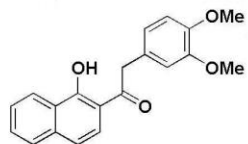
Total 3216 repetitions



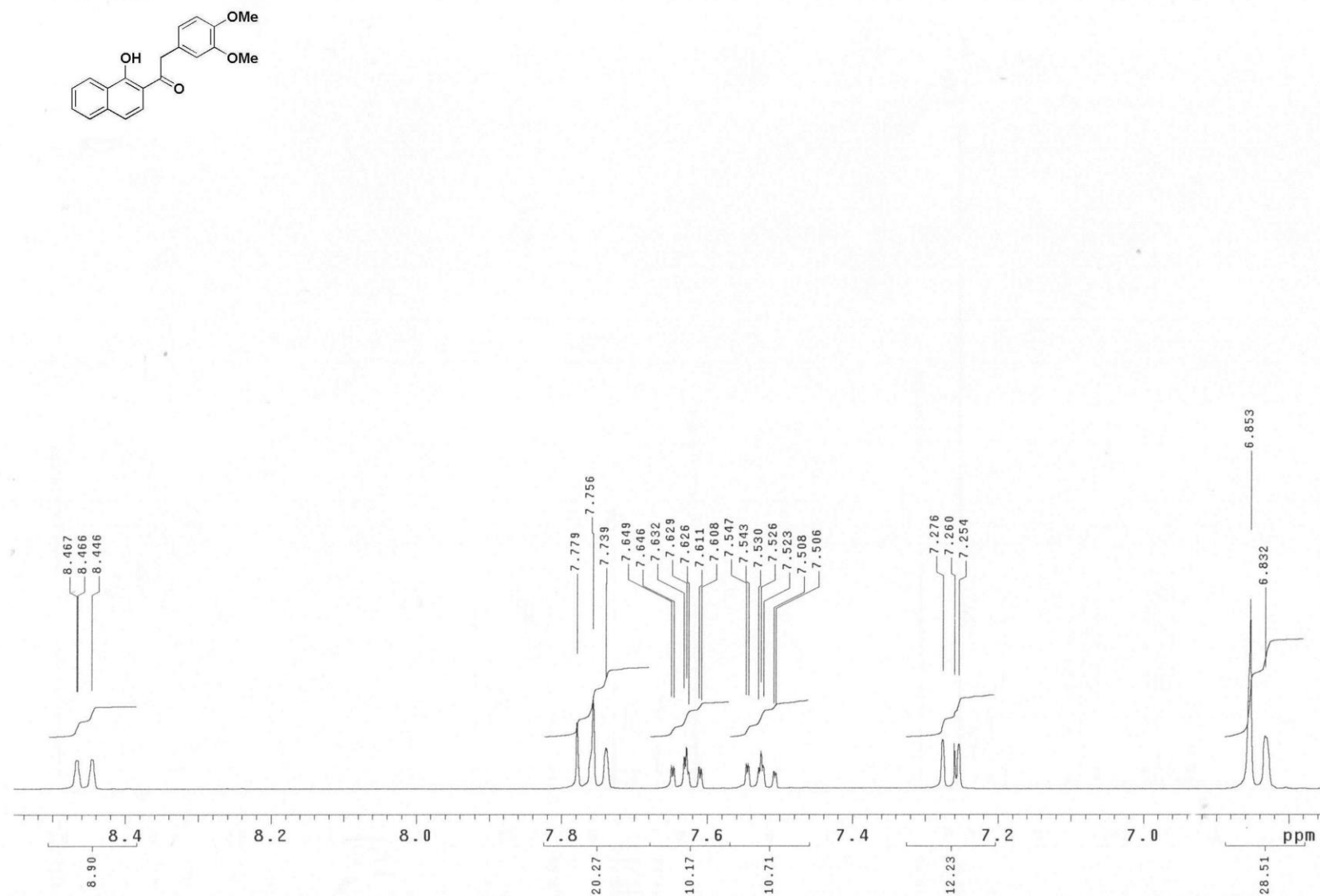
Compound 4b (¹H-NMR spectral data-1)

YTN02P

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 28 2017
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



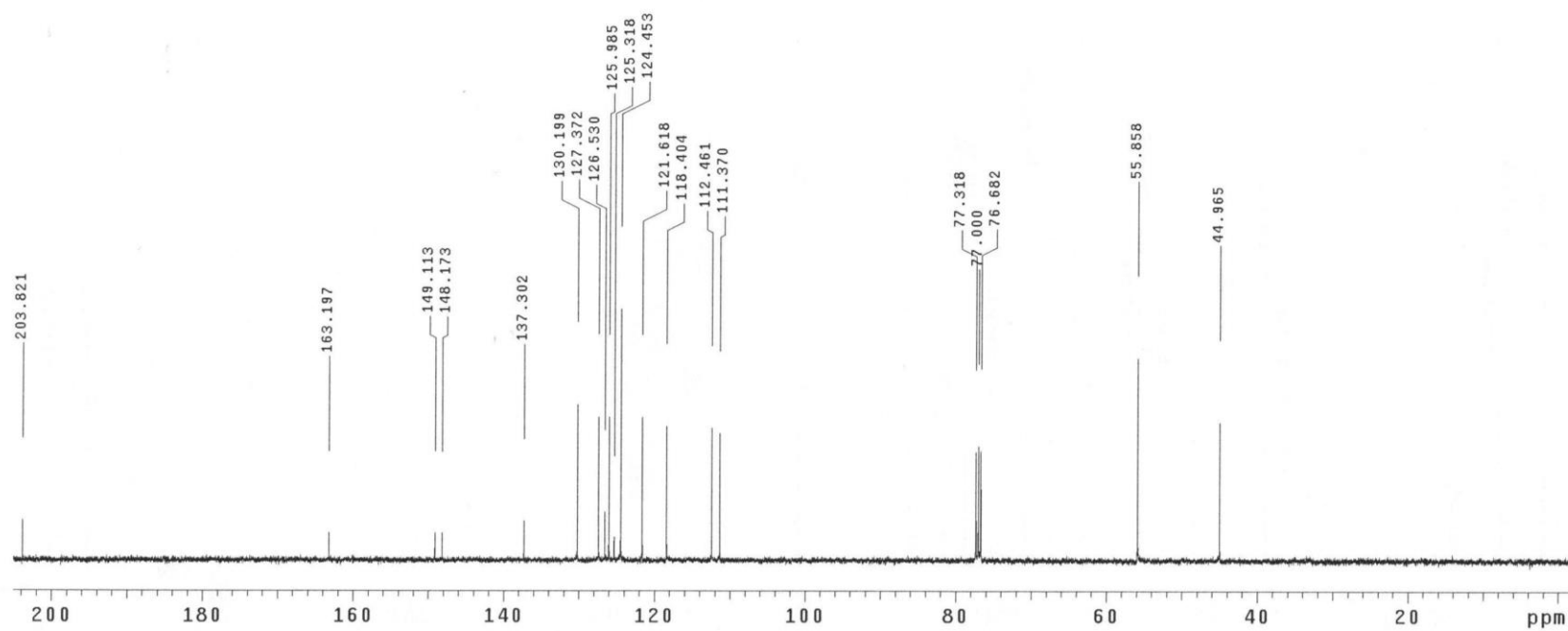
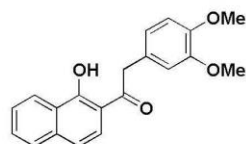
Compound 4b (¹H-NMR spectral data-2)



Compound 4b (¹³C-NMR spectral data)

YTN02P

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Dec 28 2017
Solvent: CDCl₃
Ambient temperature
Total 2048 repetitions



Compound 4c (¹H-NMR spectral data)

YTN4FP

Pulse Sequence: s2pu1

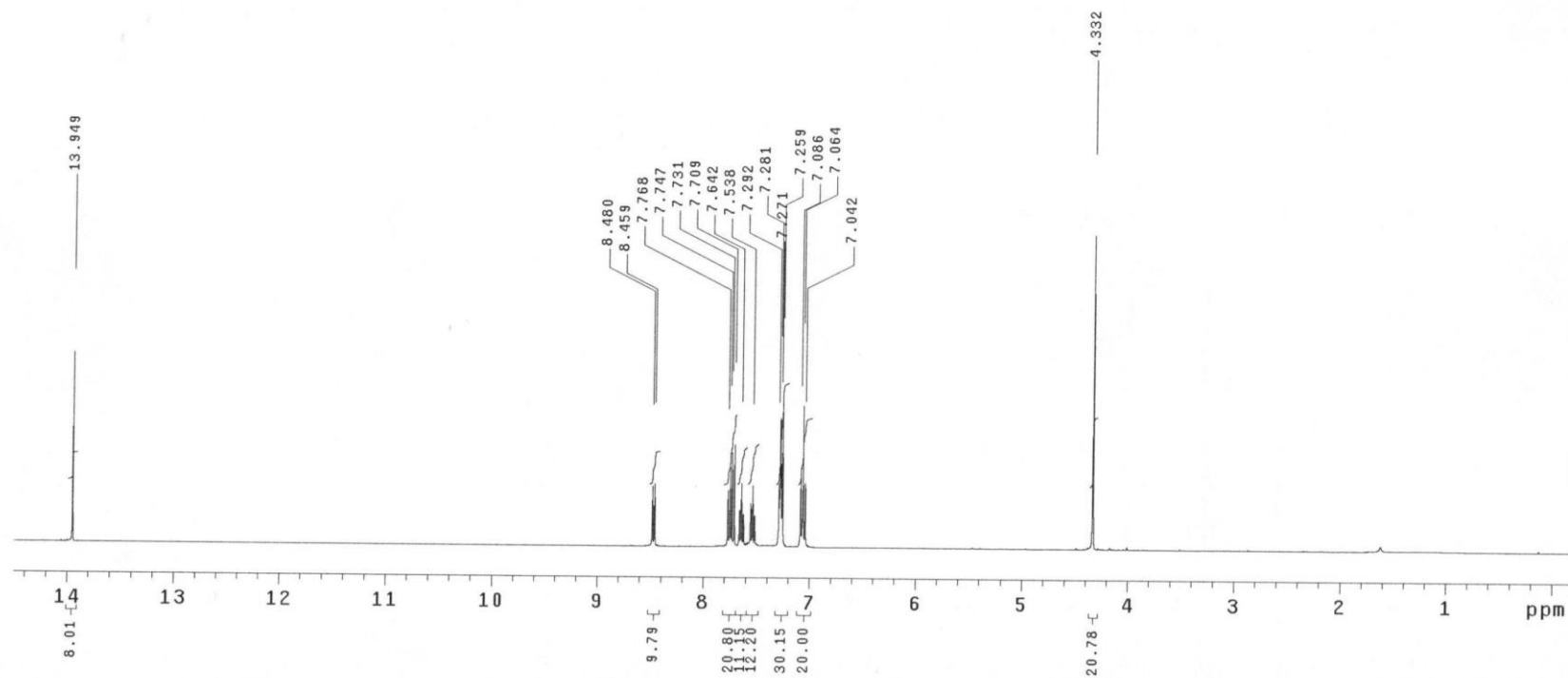
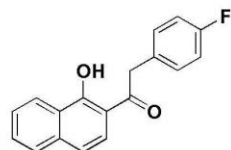
UNITYplus-400 "unity400"

Date: Jan 12 2018

Solvent: CDCl₃

Ambient temperature

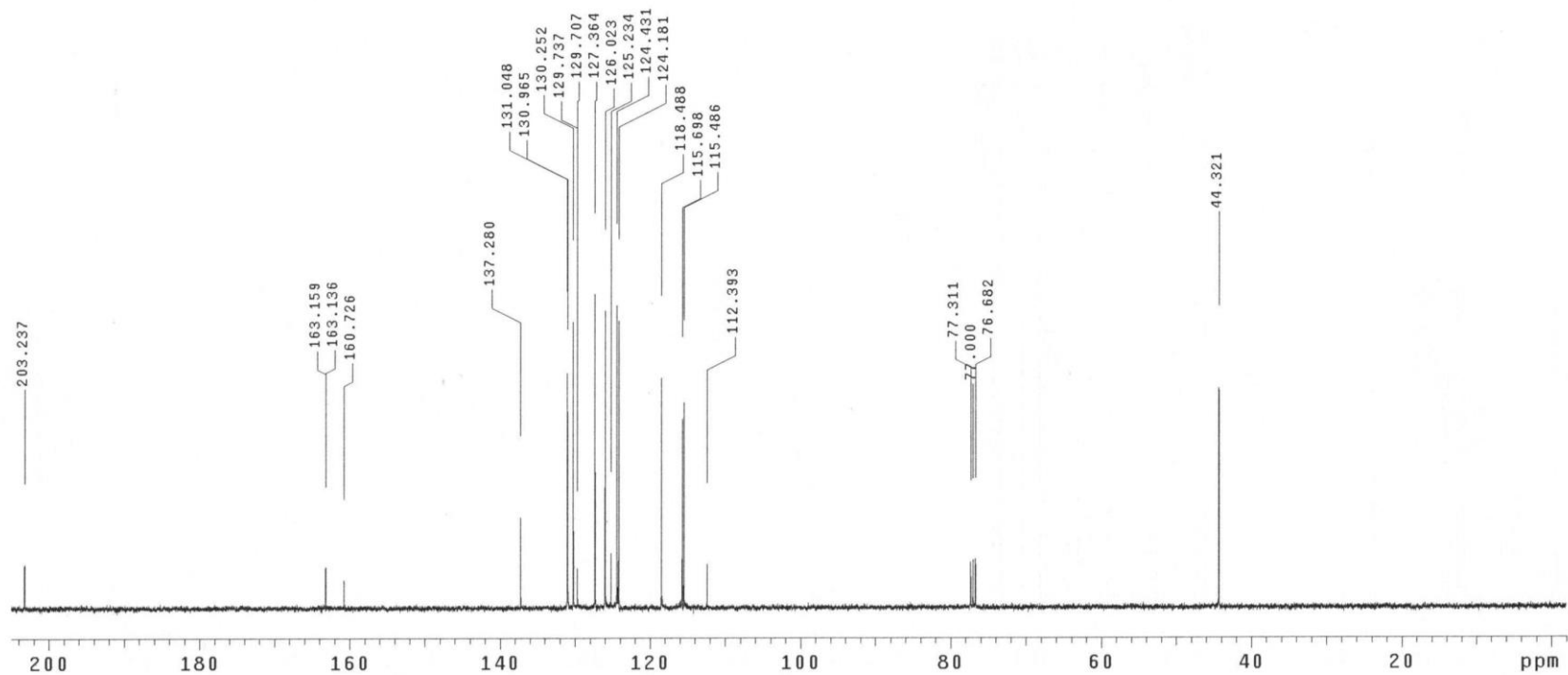
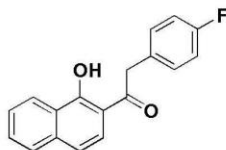
Total 32 repetitions



Compound 4c (¹³C-NMR spectral data)

YTN4FP

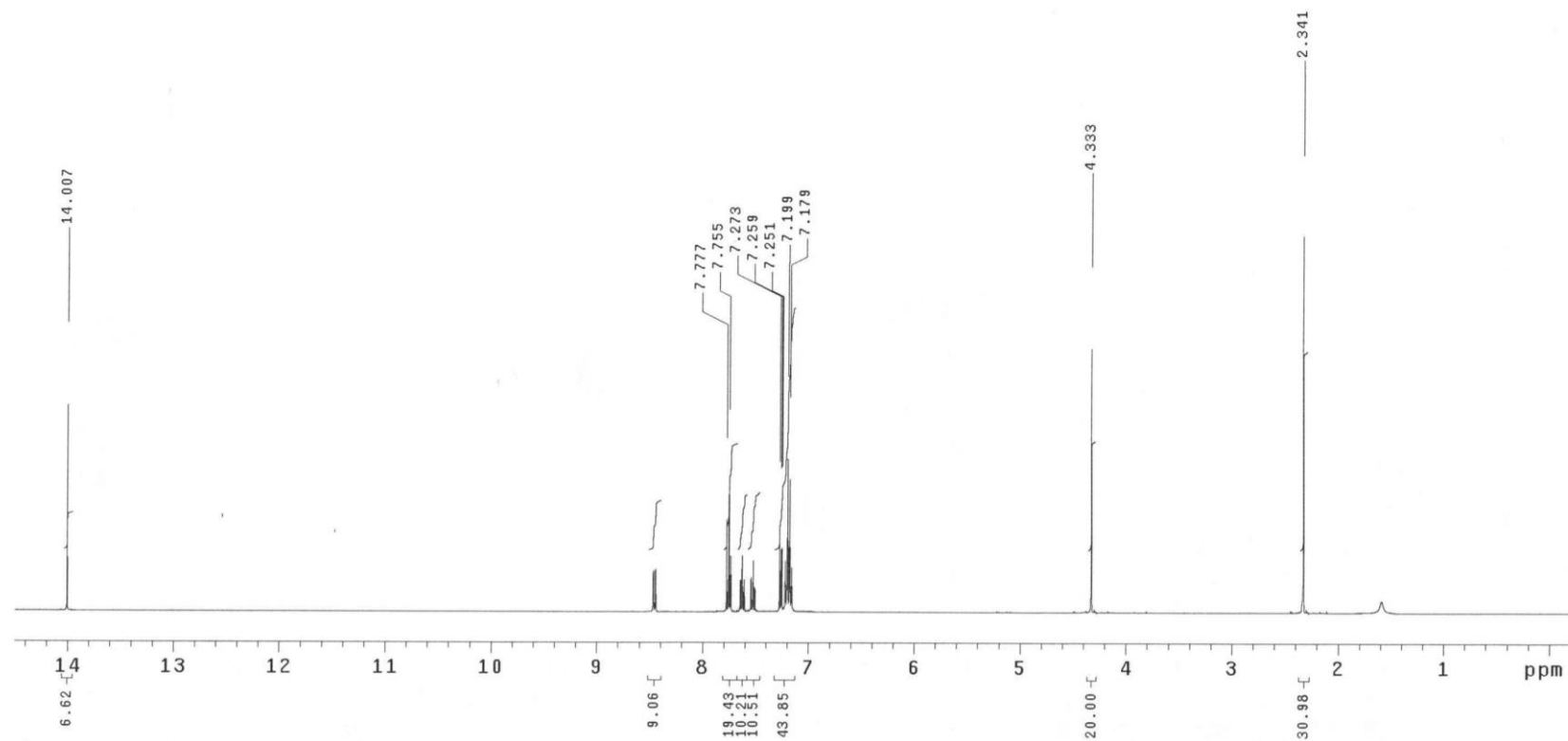
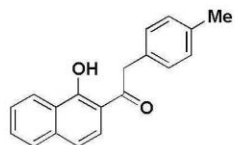
Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 12 2018
Solvent: CDCl₃
Ambient temperature
Total 672 repetitions



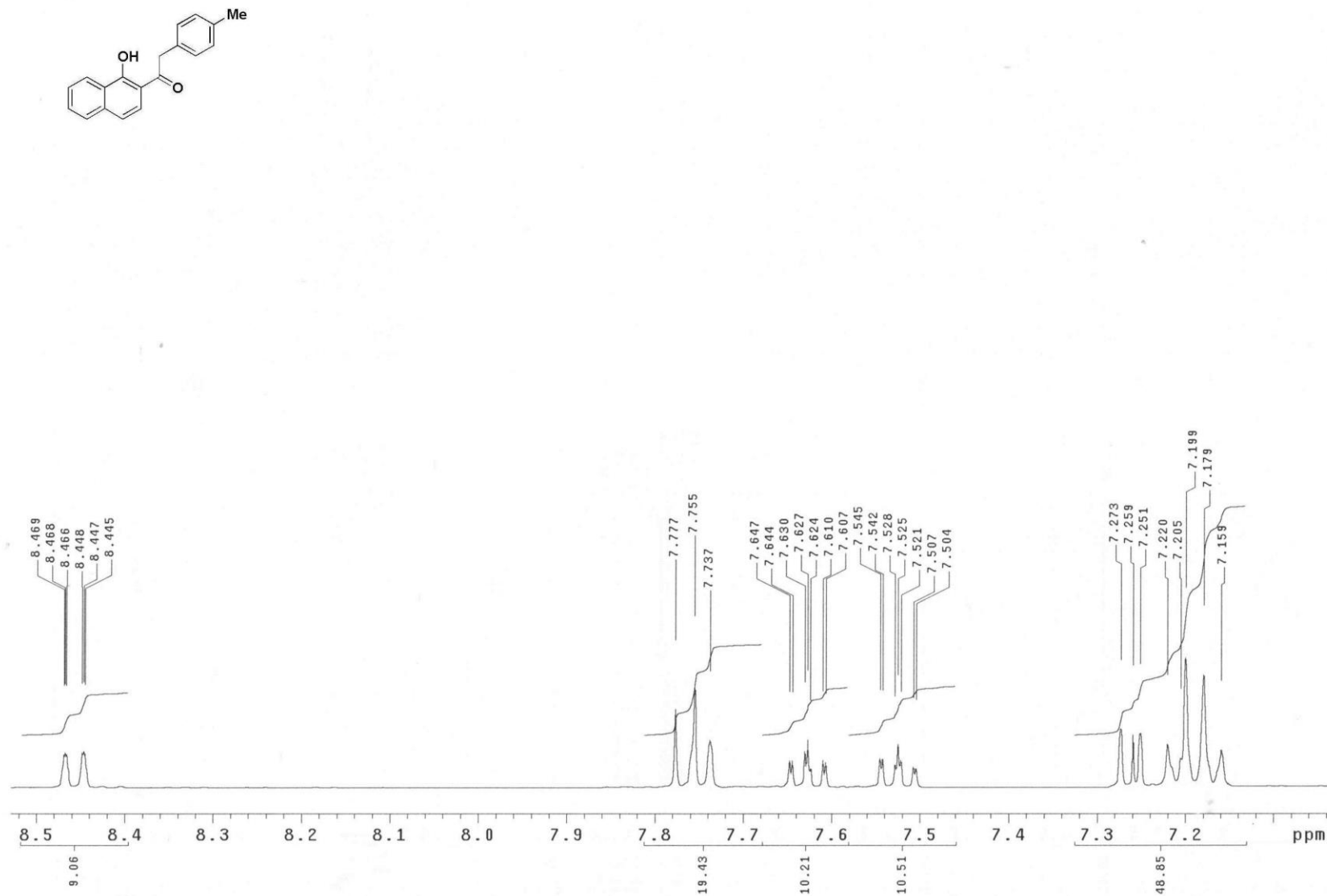
Compound 4d (¹H-NMR spectral data-1)

YTN4MeP

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jan 19 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 4d (¹H-NMR spectral data-2)



Compound 4d (¹³C-NMR spectral data-1)

YTN4MeP

Pulse Sequence: s2pu1

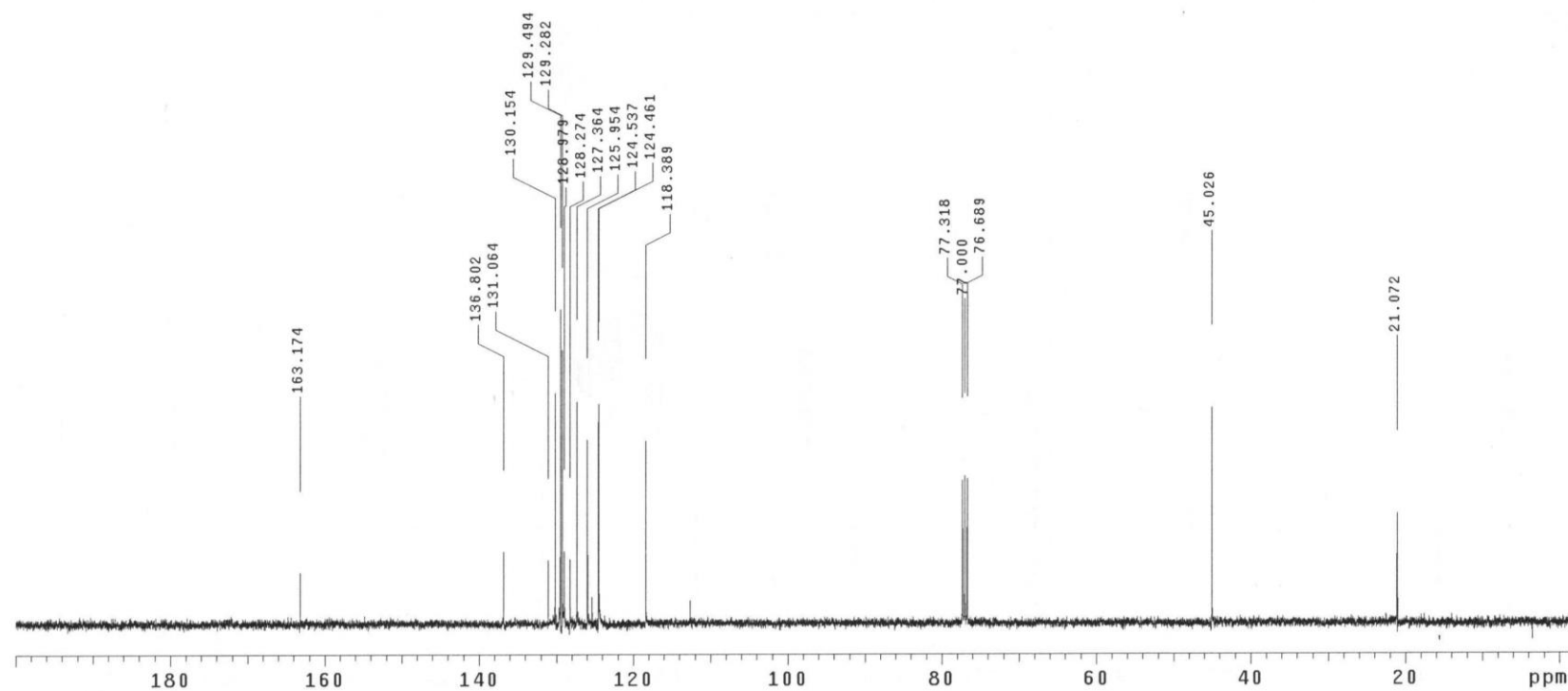
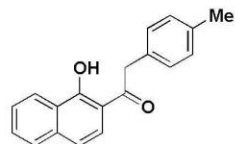
UNITYplus-400 "unity400"

Date: Jan 19 2018

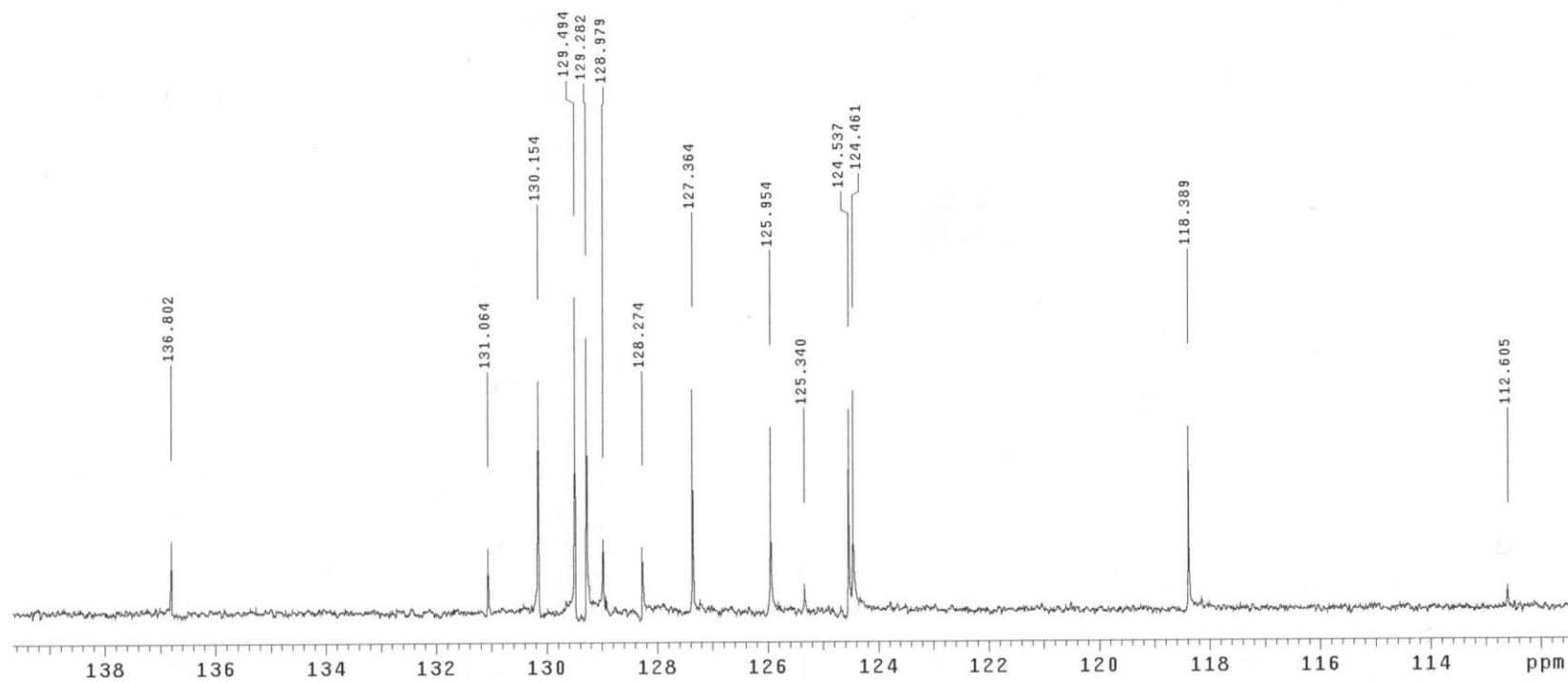
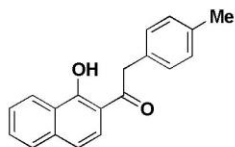
Solvent: CDCl₃

Ambient temperature

Total 2304 repetitions



Compound 4d (^{13}C -NMR spectral data-2)



Compound 4e (¹H-NMR spectral data)

YT4PhP

Pulse Sequence: s2pu1

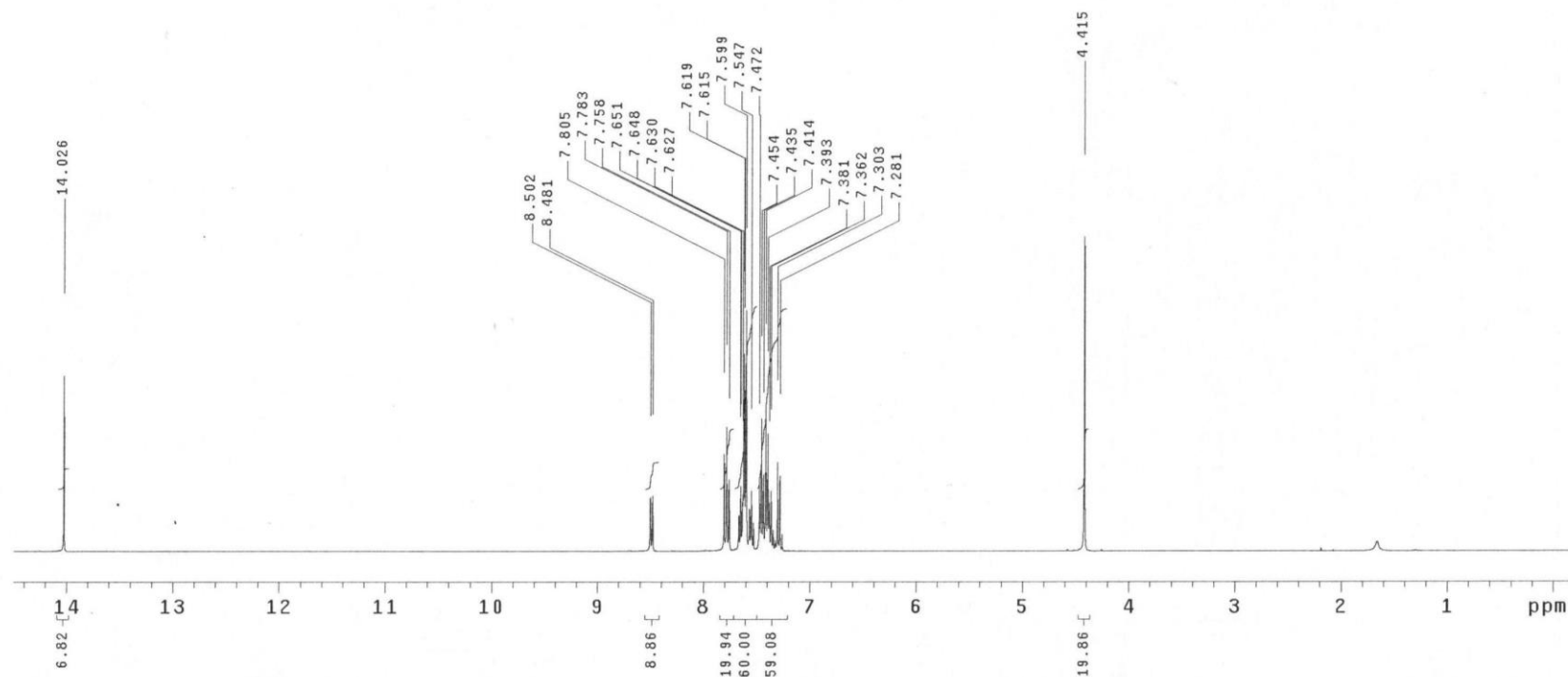
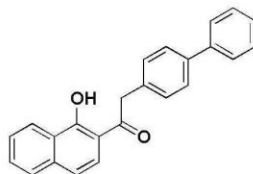
UNITYplus-400 "unity400"

Date: Apr 17 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



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

YT4PhP

Pulse Sequence: s2pu1

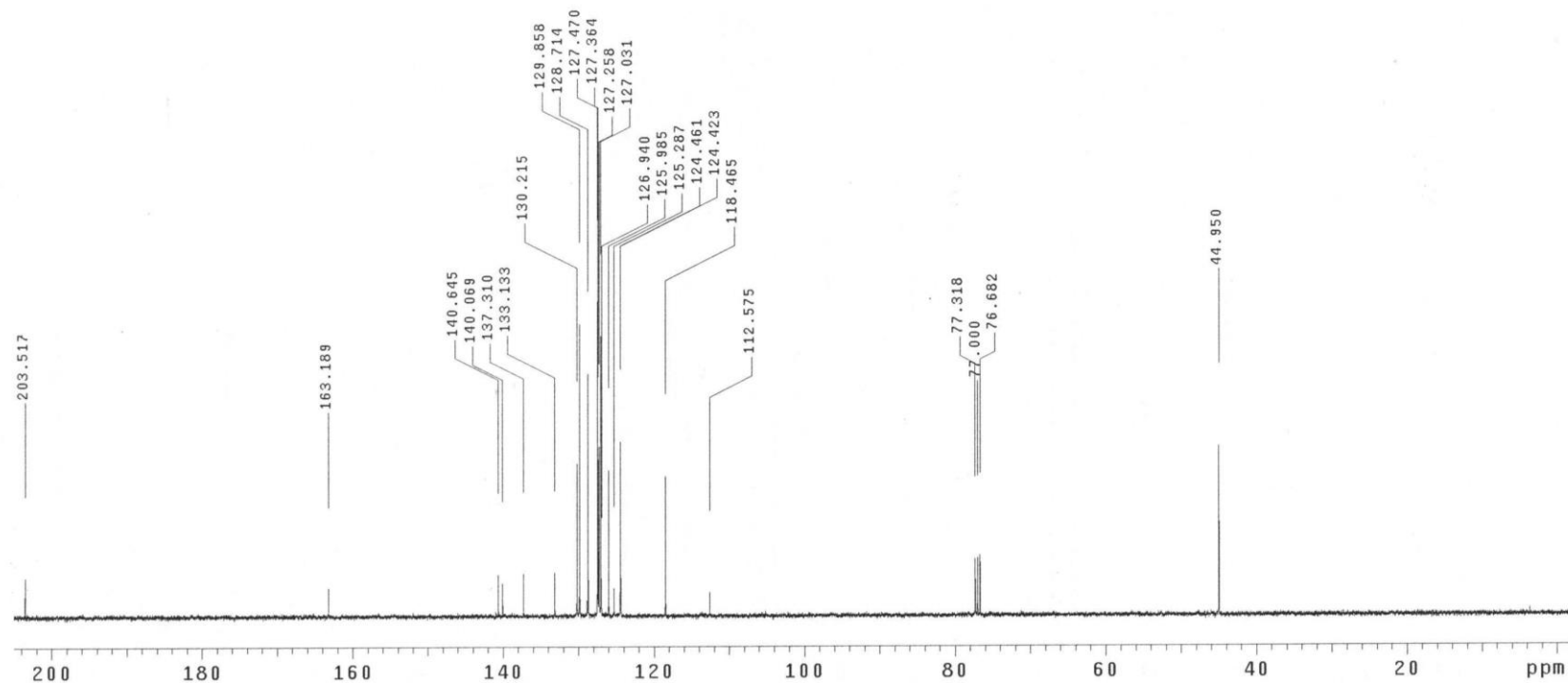
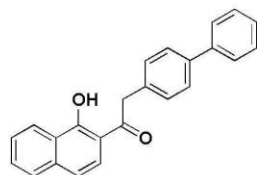
UNITYplus-400 "unity400"

Date: Apr 17 2018

Solvent: CDCl₃

Ambient temperature

Total 448 repetitions



Compound 4f (¹H-NMR spectral data)

SH02P

Pulse Sequence: s2pu1

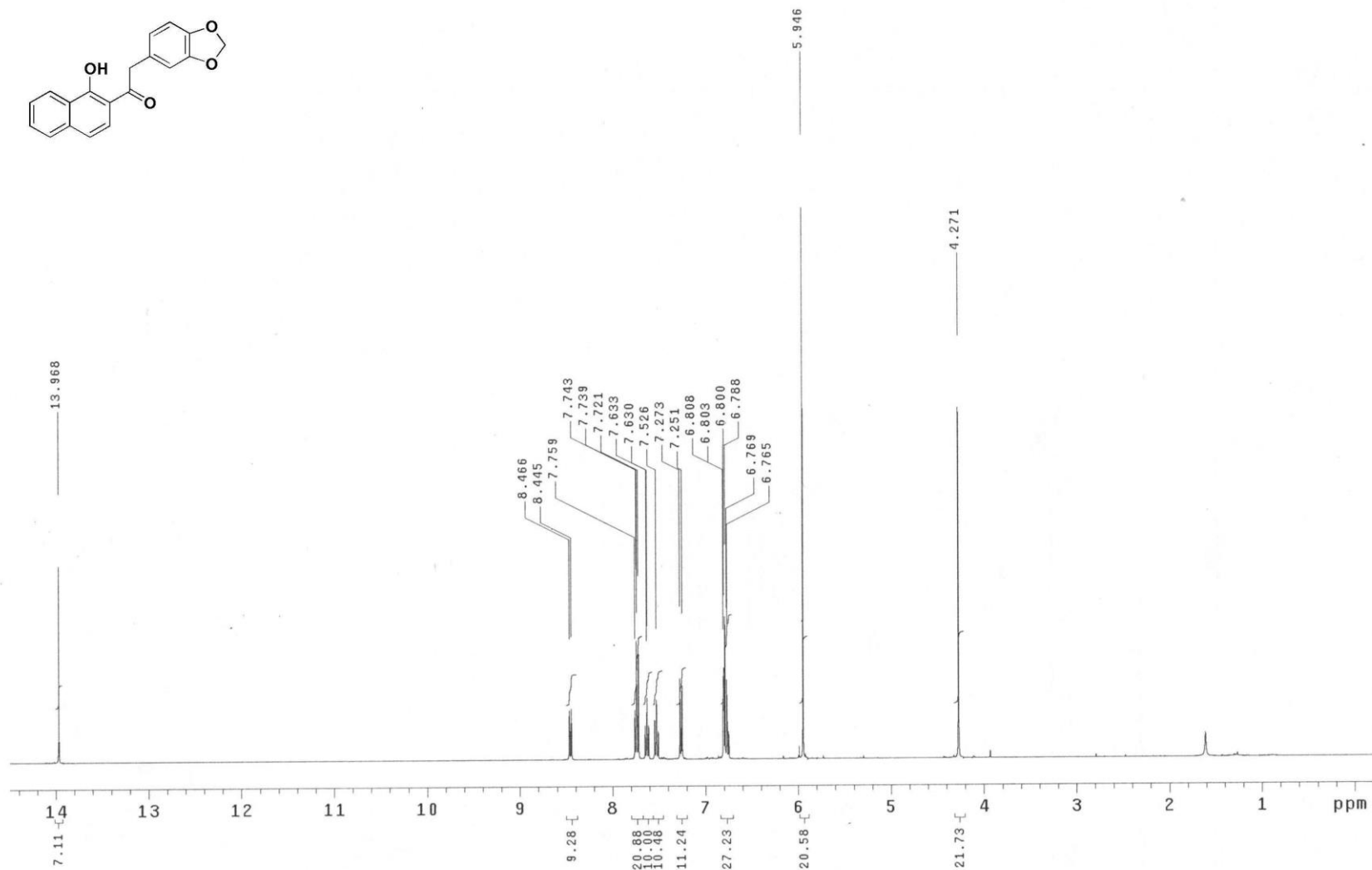
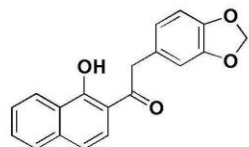
UNITYplus-400 "unity400"

Date: May 10 2018

Solvent: CDCl₃

Ambient temperature

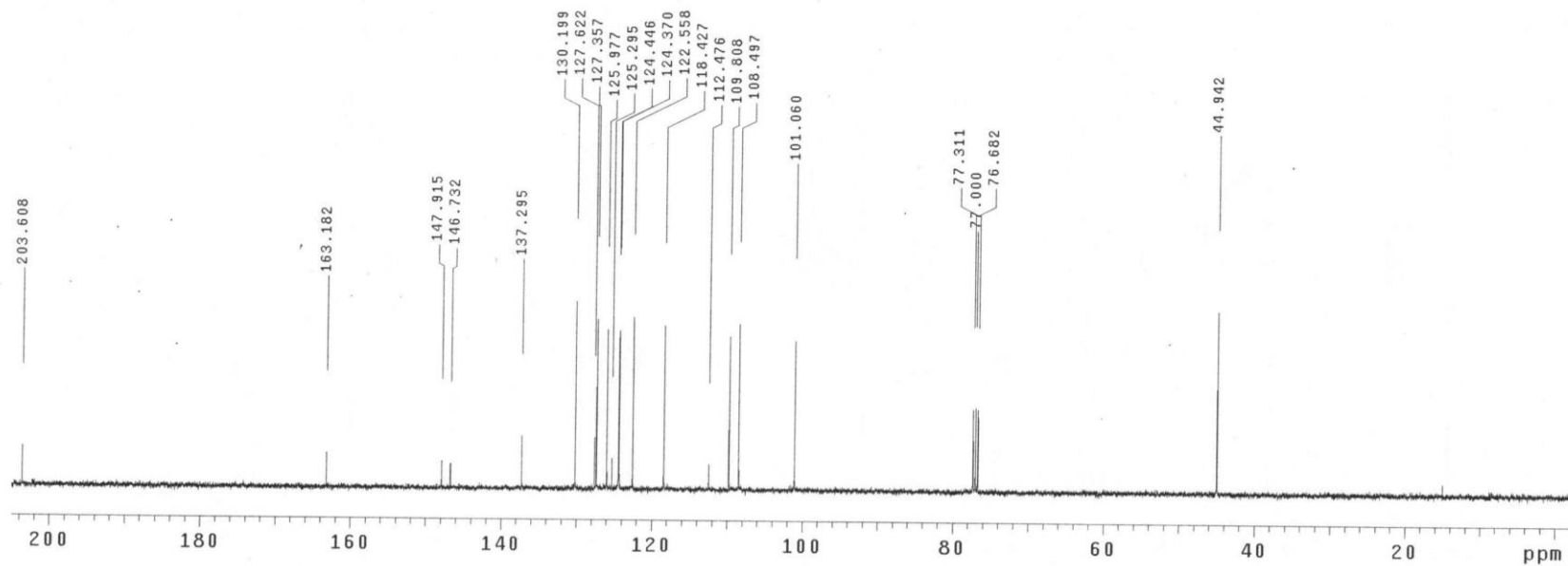
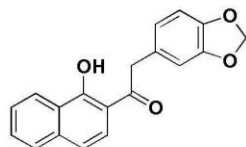
Total 32 repetitions



Compound 4f (¹³C-NMR spectral data)

SH02P

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: May 10 2018
Solvent: CDCl₃
Ambient temperature
Total 752 repetitions



Compound 4g (¹H-NMR spectral data)

YT1NASP

Pulse Sequence: s2pu1

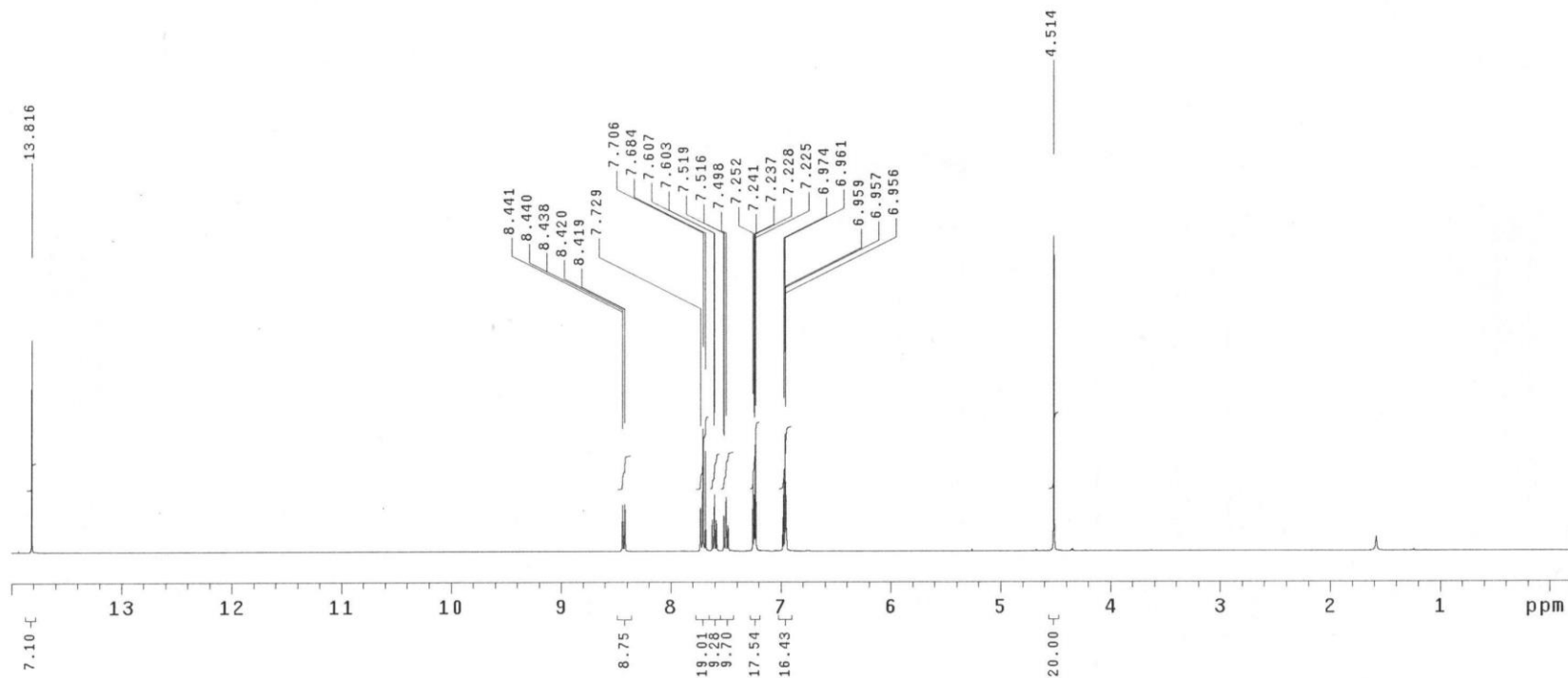
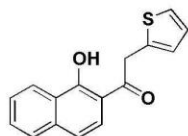
UNITYplus-400 "unity400"

Date: Apr 27 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4g (¹³C-NMR spectral data)

YT1NASP

Pulse Sequence: s2pu1

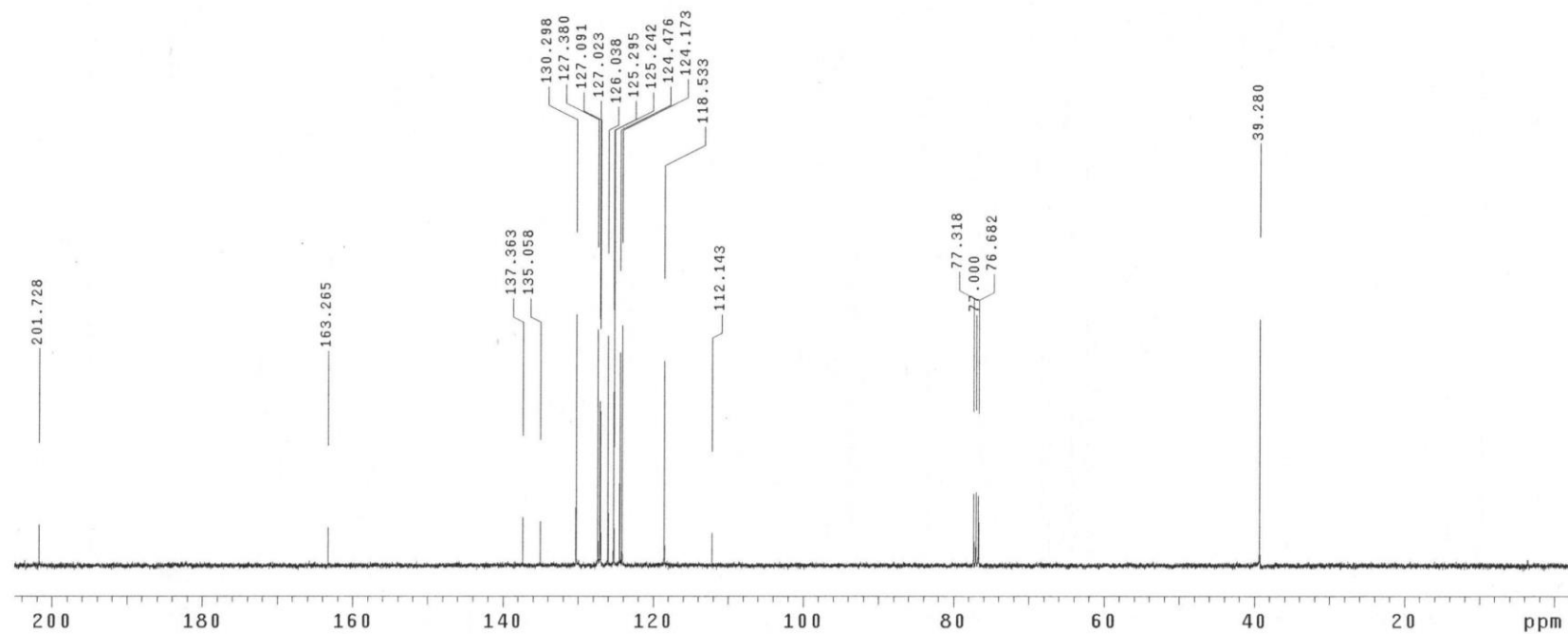
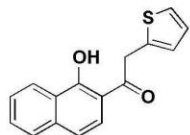
UNITYplus-400 "unity400"

Date: Apr 27 2018

Solvent: CDCl₃

Ambient temperature

Total 480 repetitions



Compound 4h (¹H-NMR spectral data)

YT1NBrP

Pulse Sequence: s2pu1

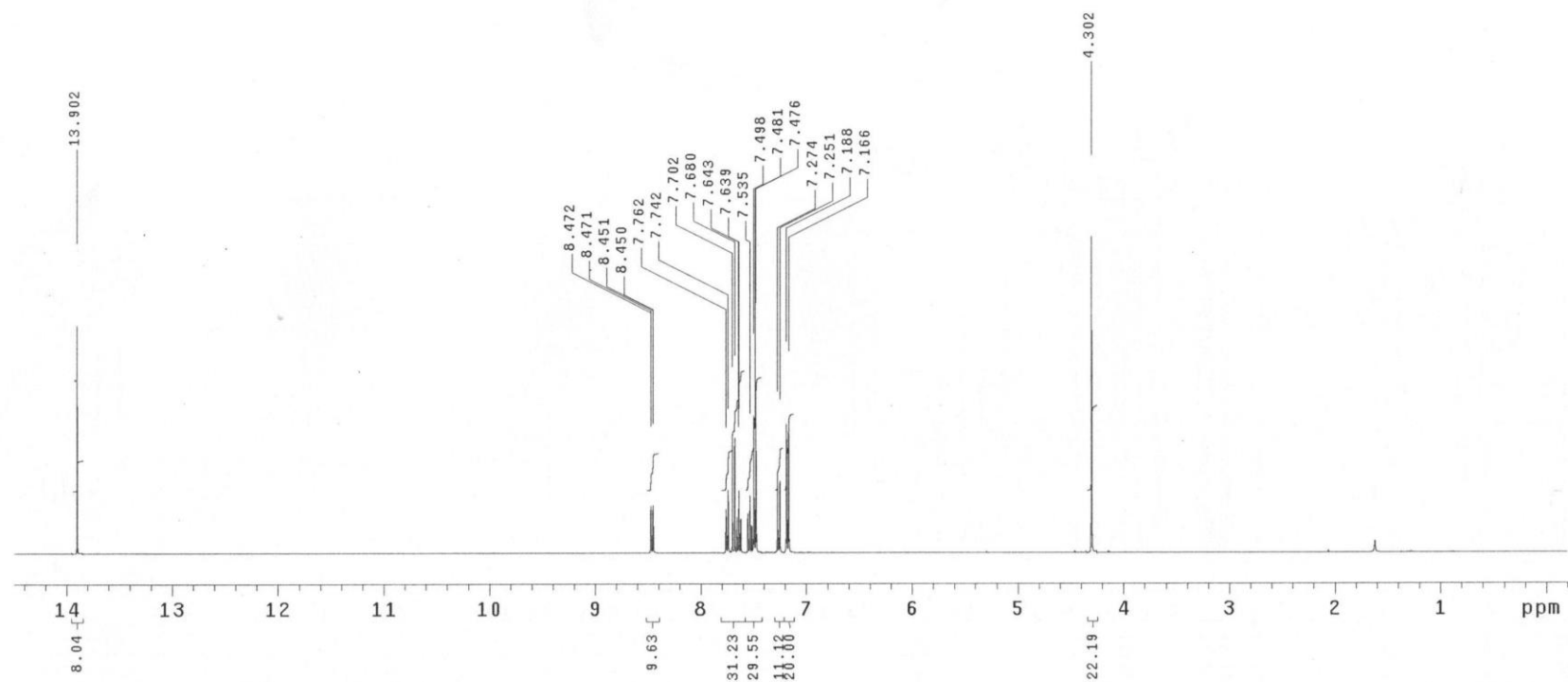
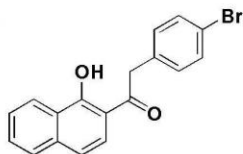
UNITYplus-400 "unity400"

Date: Apr 26 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4h (¹³C-NMR spectral data)

YT1NBrP

Pulse Sequence: s2pu1

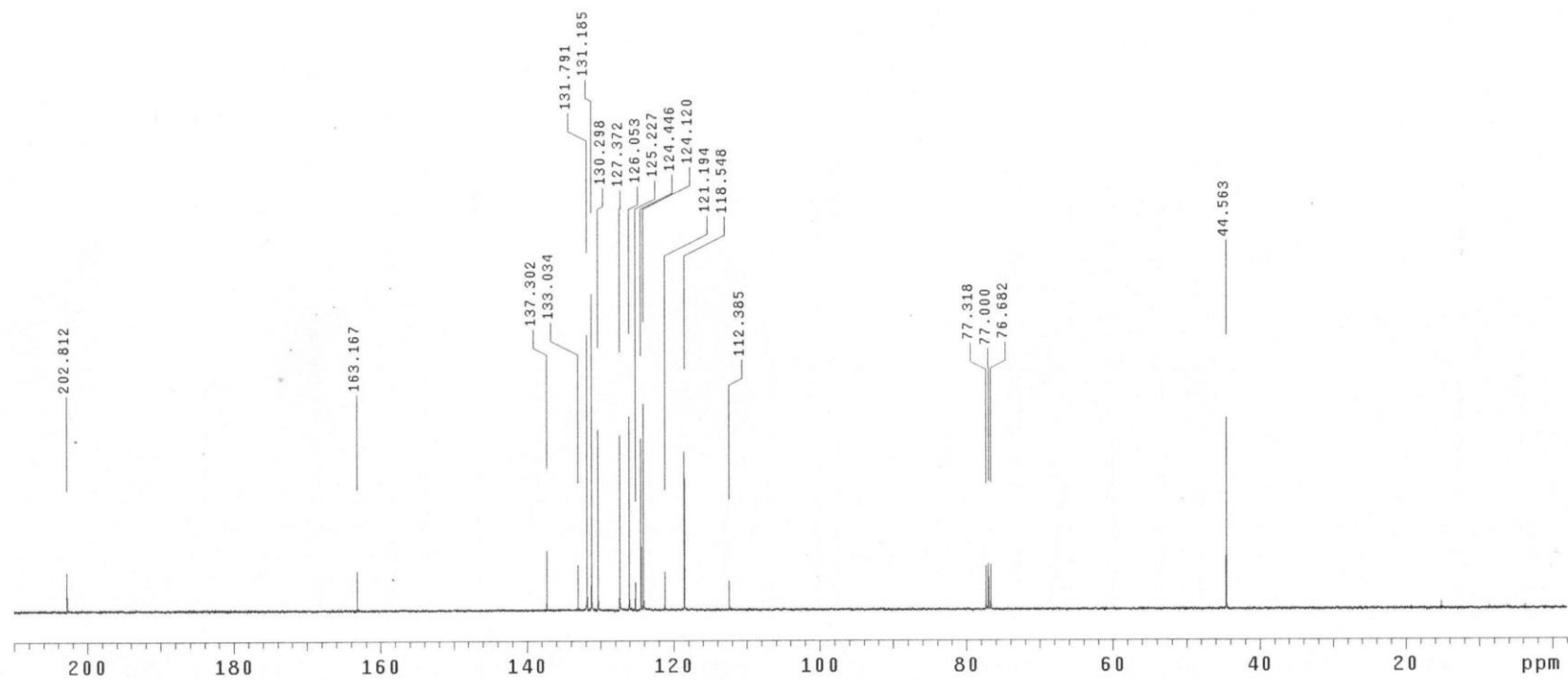
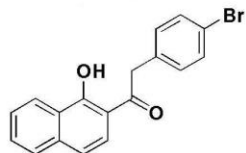
UNITYplus-400 "unity400"

Date: Apr 26 2018

Solvent: CDCl₃

Ambient temperature

Total 720 repetitions



Compound 4i (¹H-NMR spectral data)

YT2FP

Pulse Sequence: s2pu1

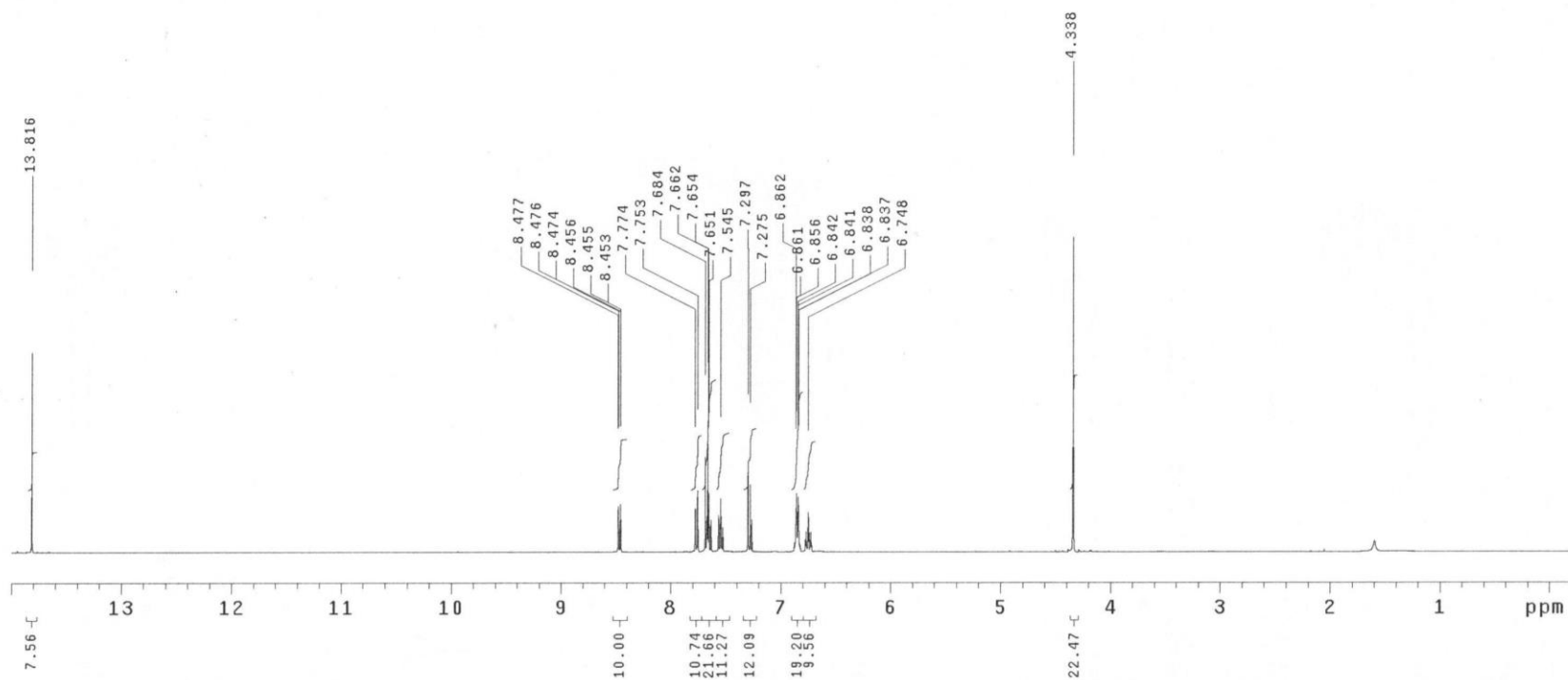
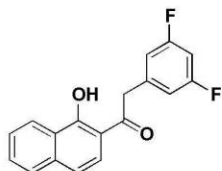
UNITYplus-400 "unity400"

Date: May 11 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4i (¹³C-NMR spectral data)

YT2FP

Pulse Sequence: s2pu1

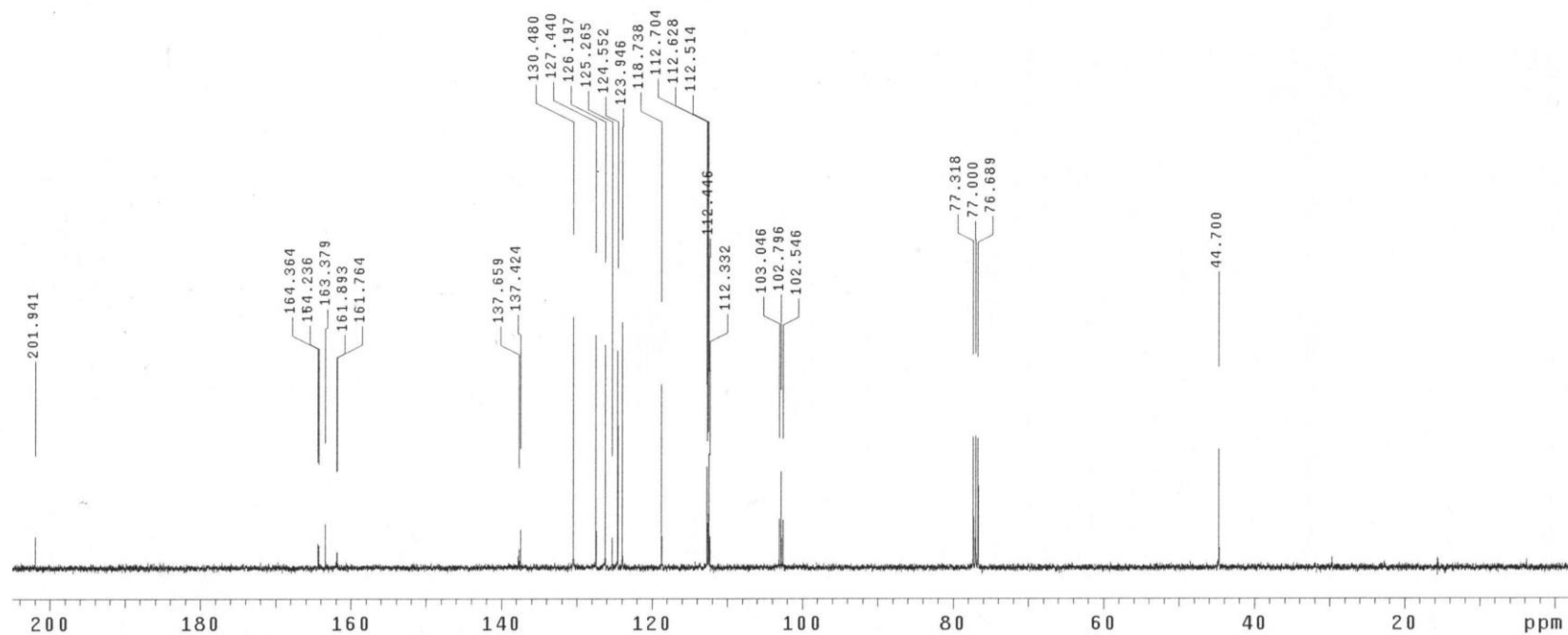
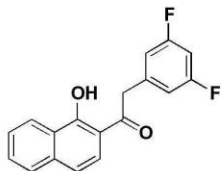
UNITYplus-400 "unity400"

Date: May 11 2018

Solvent: CDCl₃

Ambient temperature

Total 1280 repetitions



Compound 4j (¹H-NMR spectral data)

YTCF3P

Pulse Sequence: s2pu1

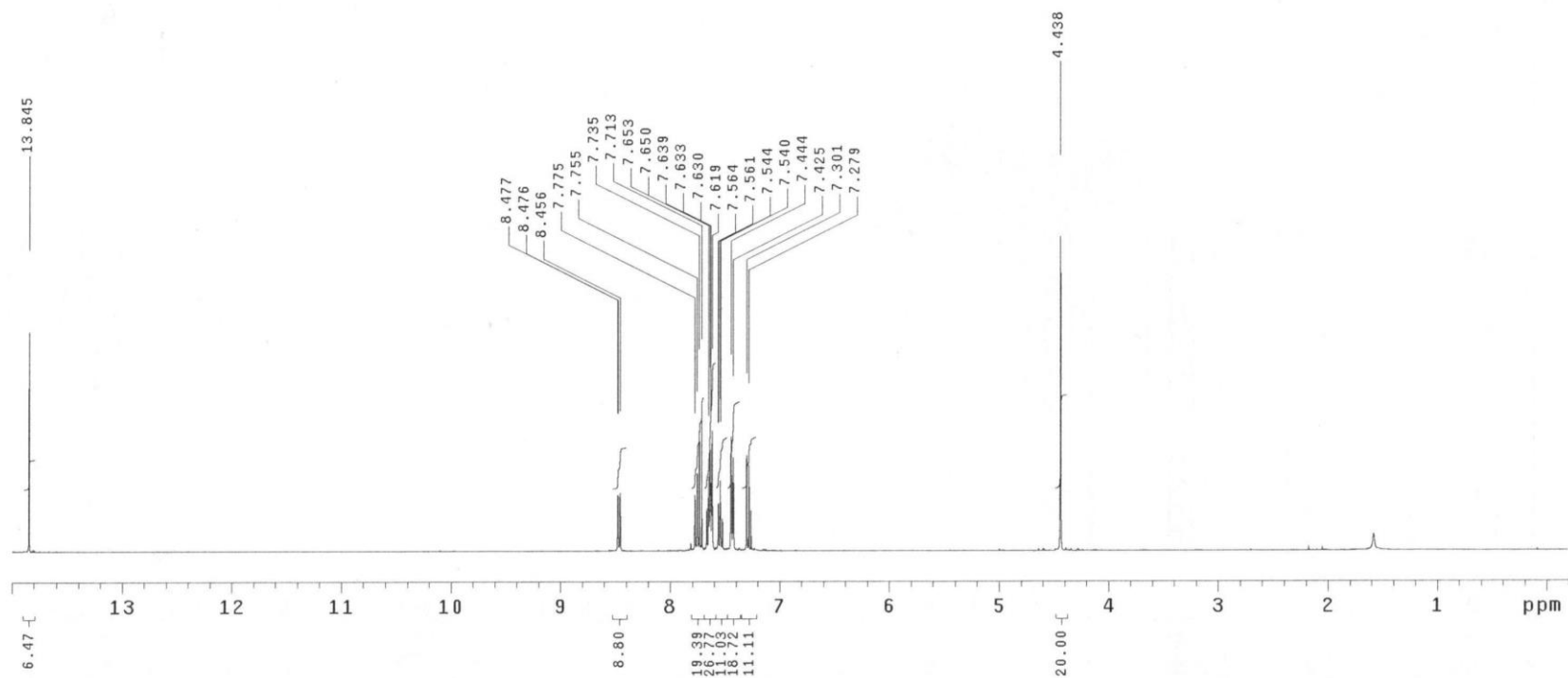
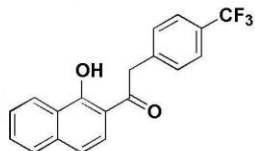
UNITYplus-400 "unity400"

Date: May 31 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4j (¹³C-NMR spectral data)

YTCF3P

Pulse Sequence: s2pu1

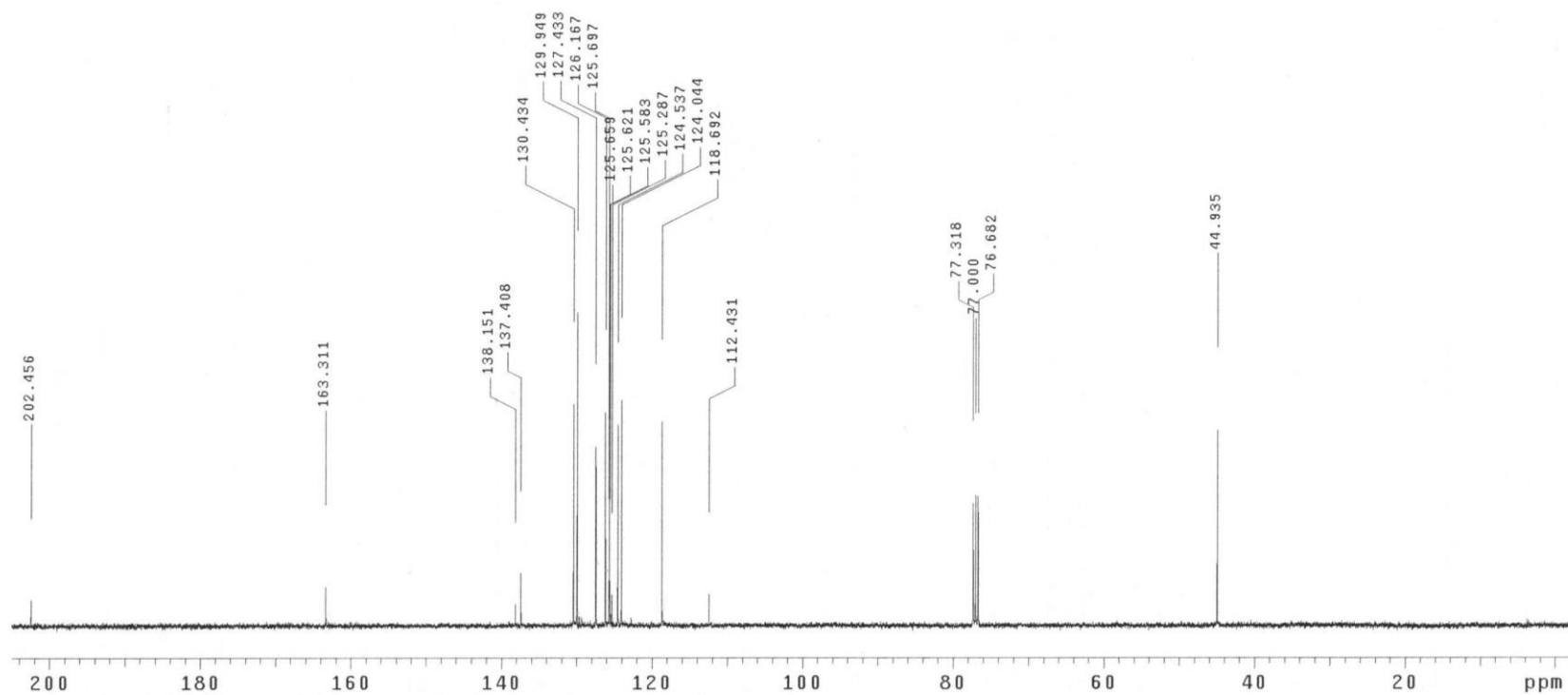
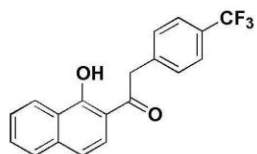
UNITYplus-400 "unity400"

Date: May 31 2018

Solvent: CDCl₃

Ambient temperature

Total 1808 repetitions



Compound 4k (¹H-NMR spectral data)

YT1NAP

Pulse Sequence: s2pu1

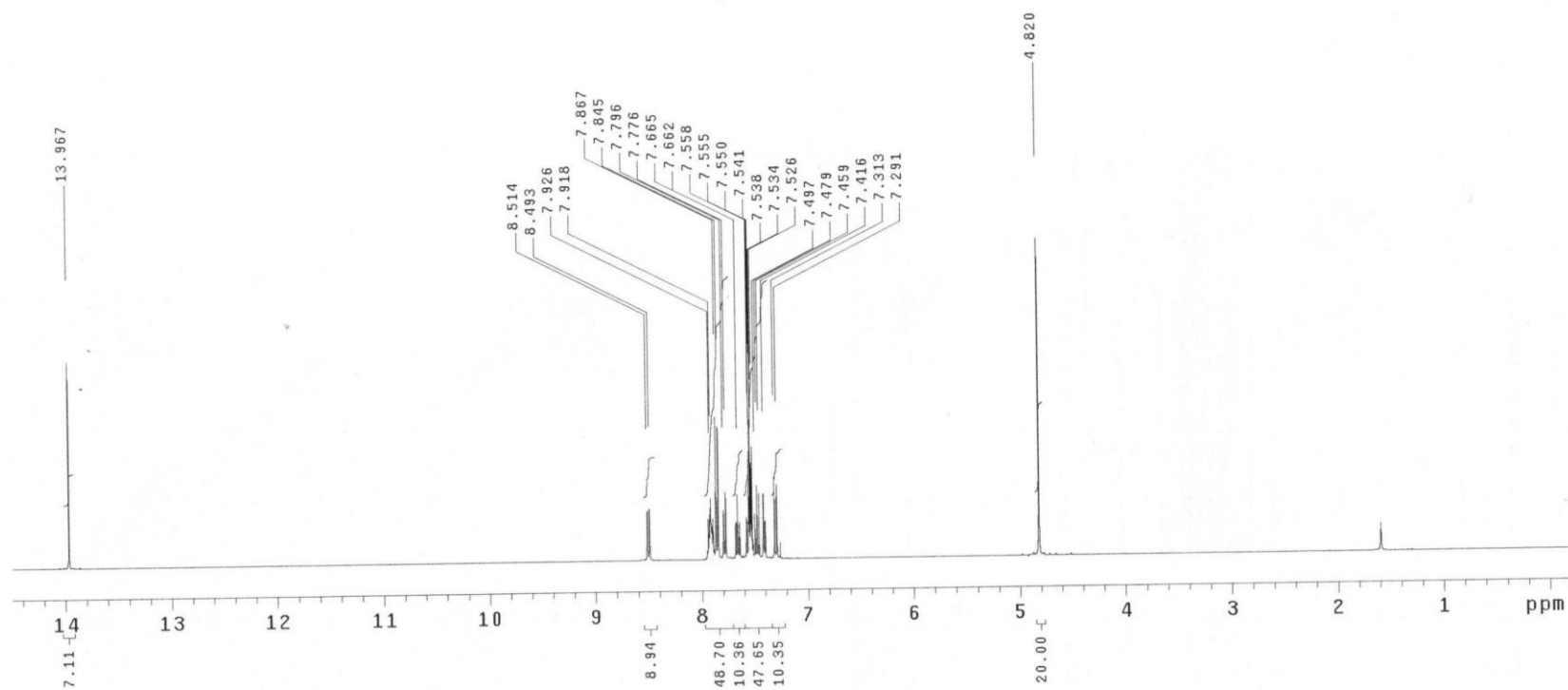
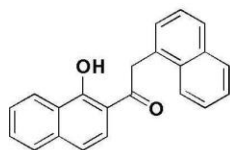
UNITYplus-400 "unity400"

Date: May 25 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4k (¹³C-NMR spectral data)

YT1NAP

Pulse Sequence: s2pu1

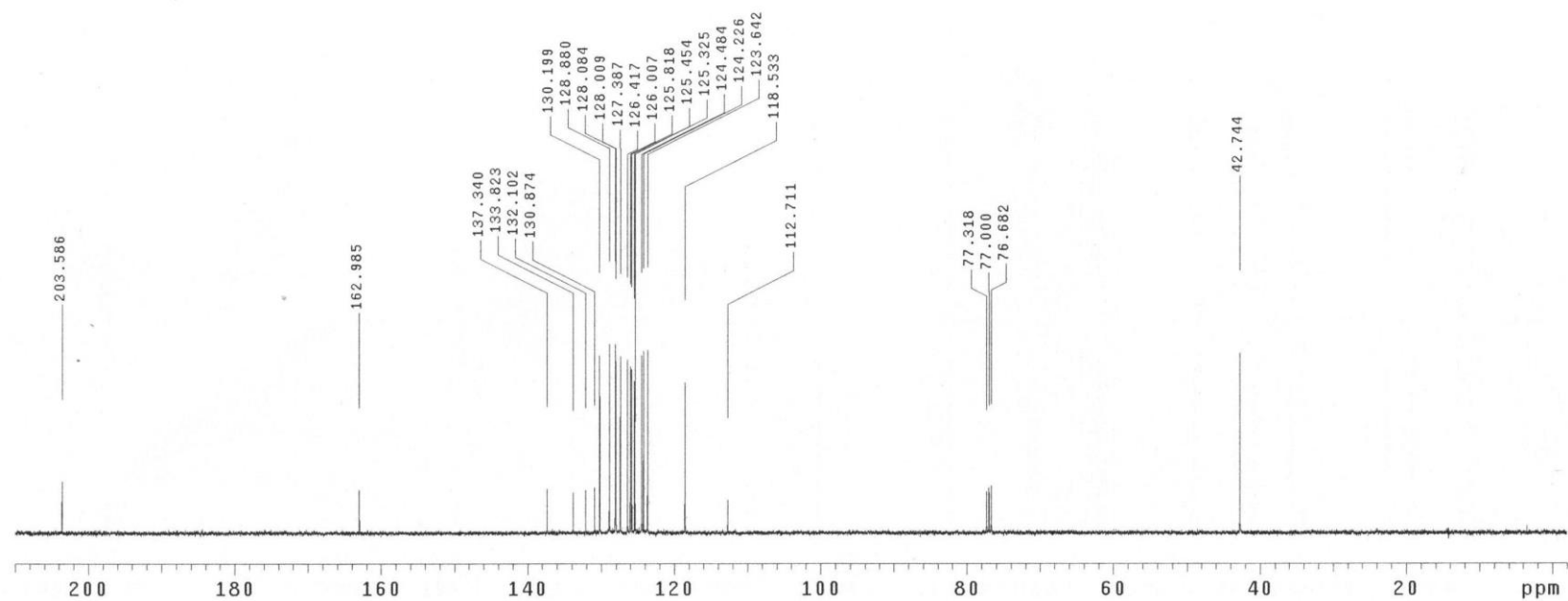
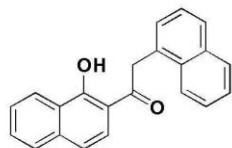
UNITYplus-400 "unity400"

Date: May 25 2018

Solvent: CDCl₃

Ambient temperature

Total 336 repetitions



Compound 4l (¹H-NMR spectral data)

SHCF3hv

Pulse Sequence: s2pu1

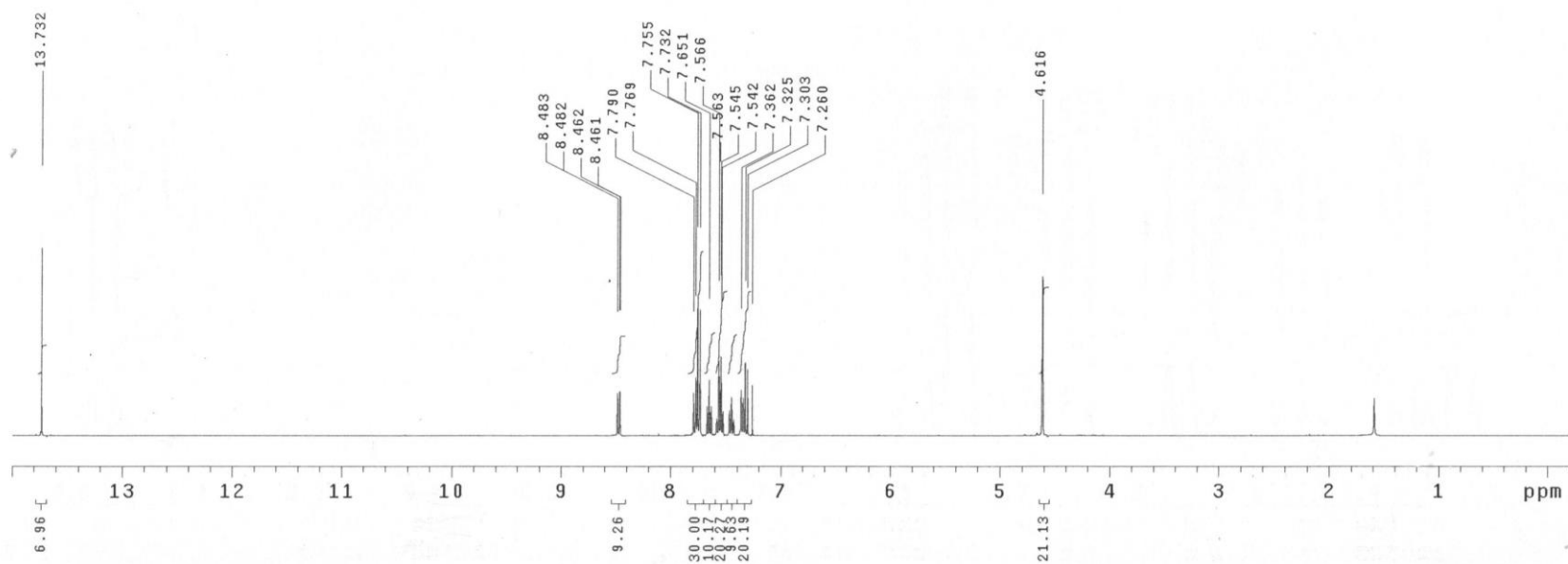
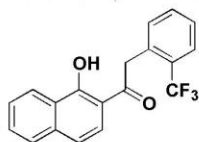
UNITYplus-400 "unity400"

Date: Jul 24 2019

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



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

SHCF3hv

Pulse Sequence: s2pu1

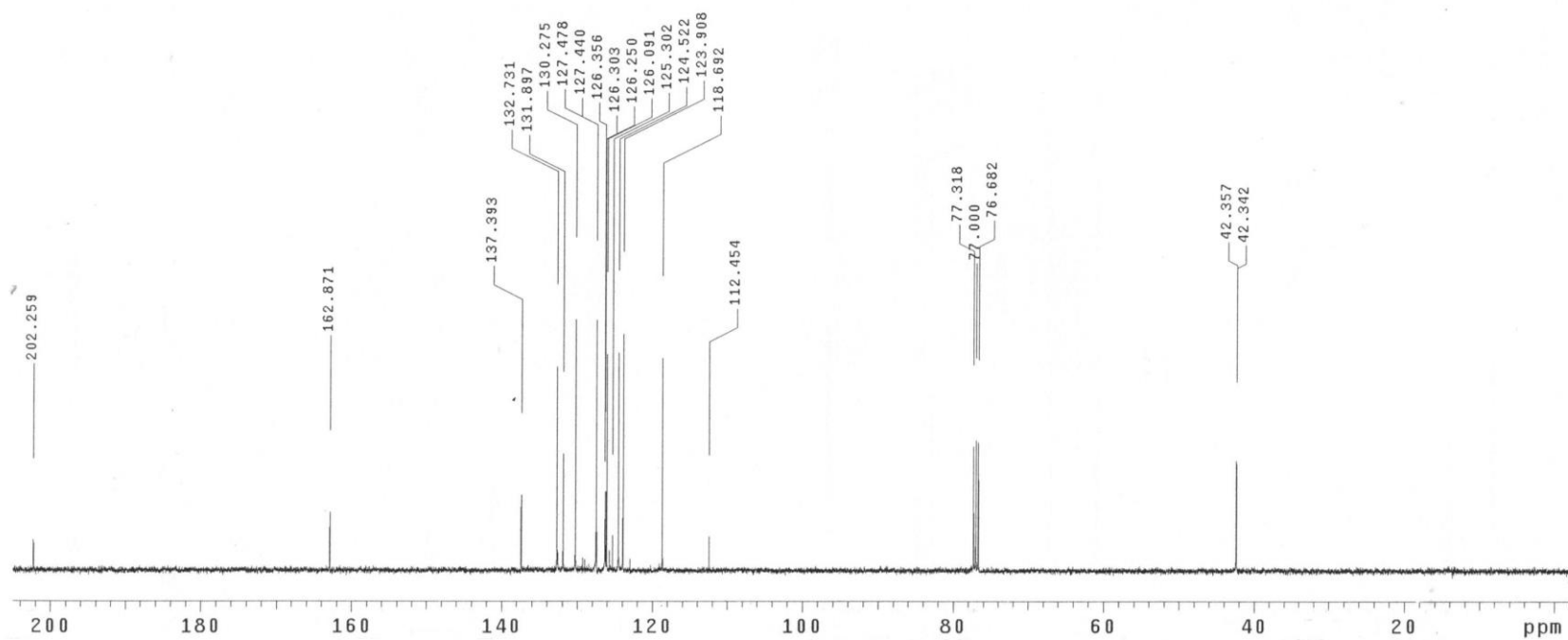
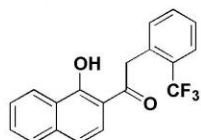
UNITYplus-400 "unity400"

Date: Jul 24 2019

Solvent: CDCl₃

Ambient temperature

Total 2912 repetitions



Compound 4m (¹H-NMR spectral data-1)

YTOMe2P

Pulse Sequence: s2pu1

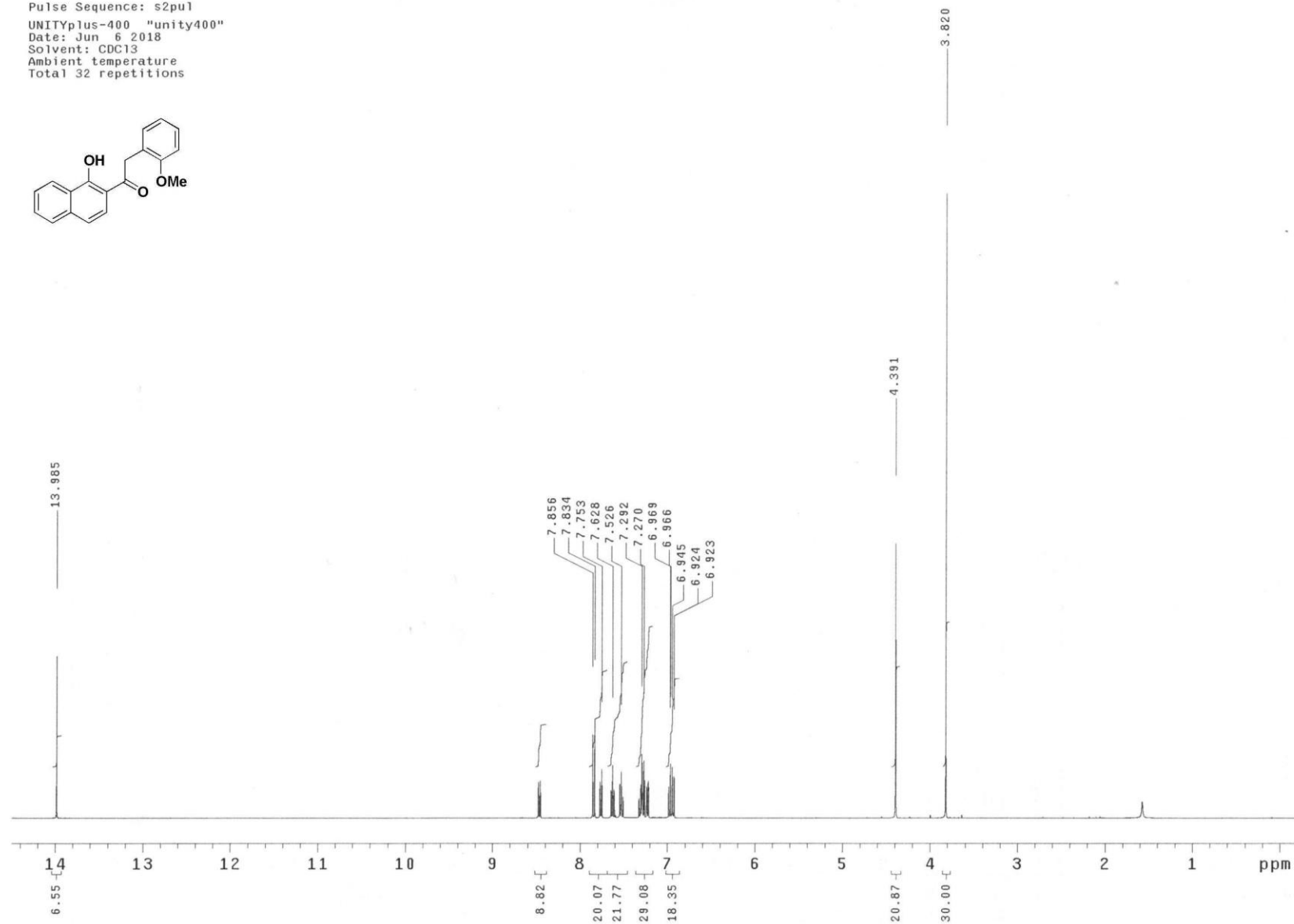
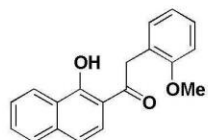
UNITYplus-400 "unity400"

Date: Jun 6 2018

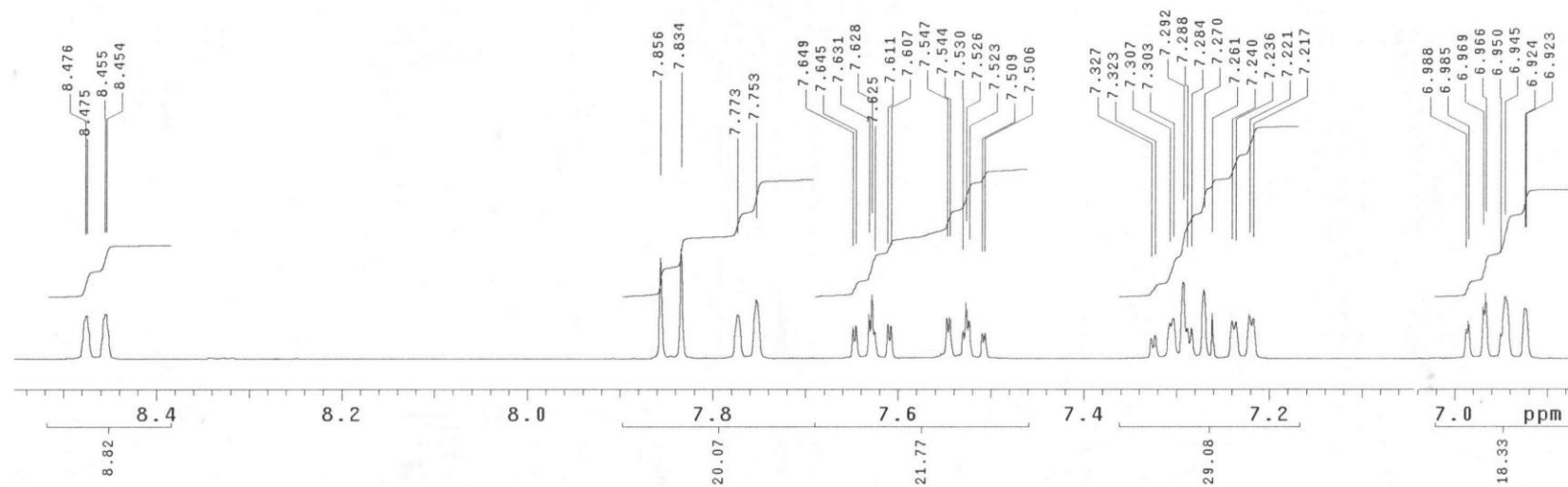
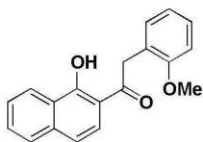
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 4m (¹H-NMR spectral data-2)



Compound 4m (¹³C-NMR spectral data)

YTOMe2P

Pulse Sequence: s2pul

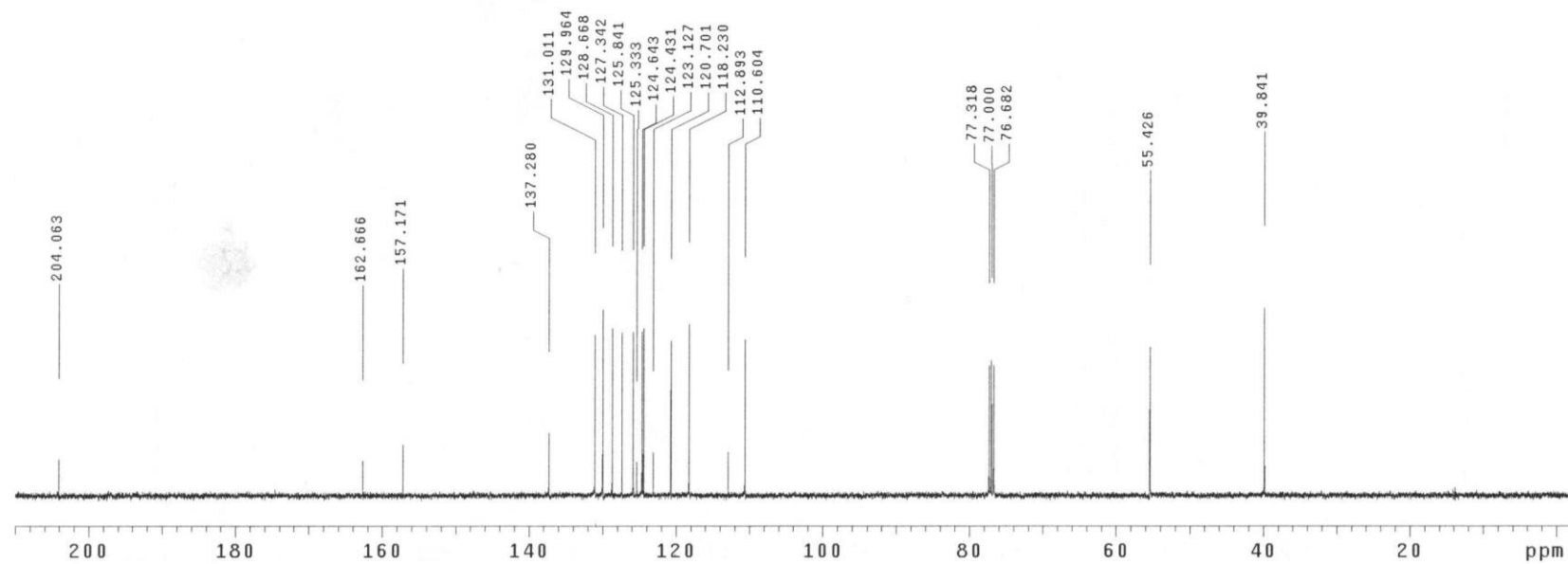
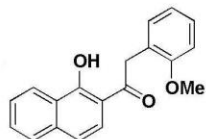
UNITYplus-400 "unity400"

Date: Jun 6 2018

Solvent: CDCl₃

Ambient temperature

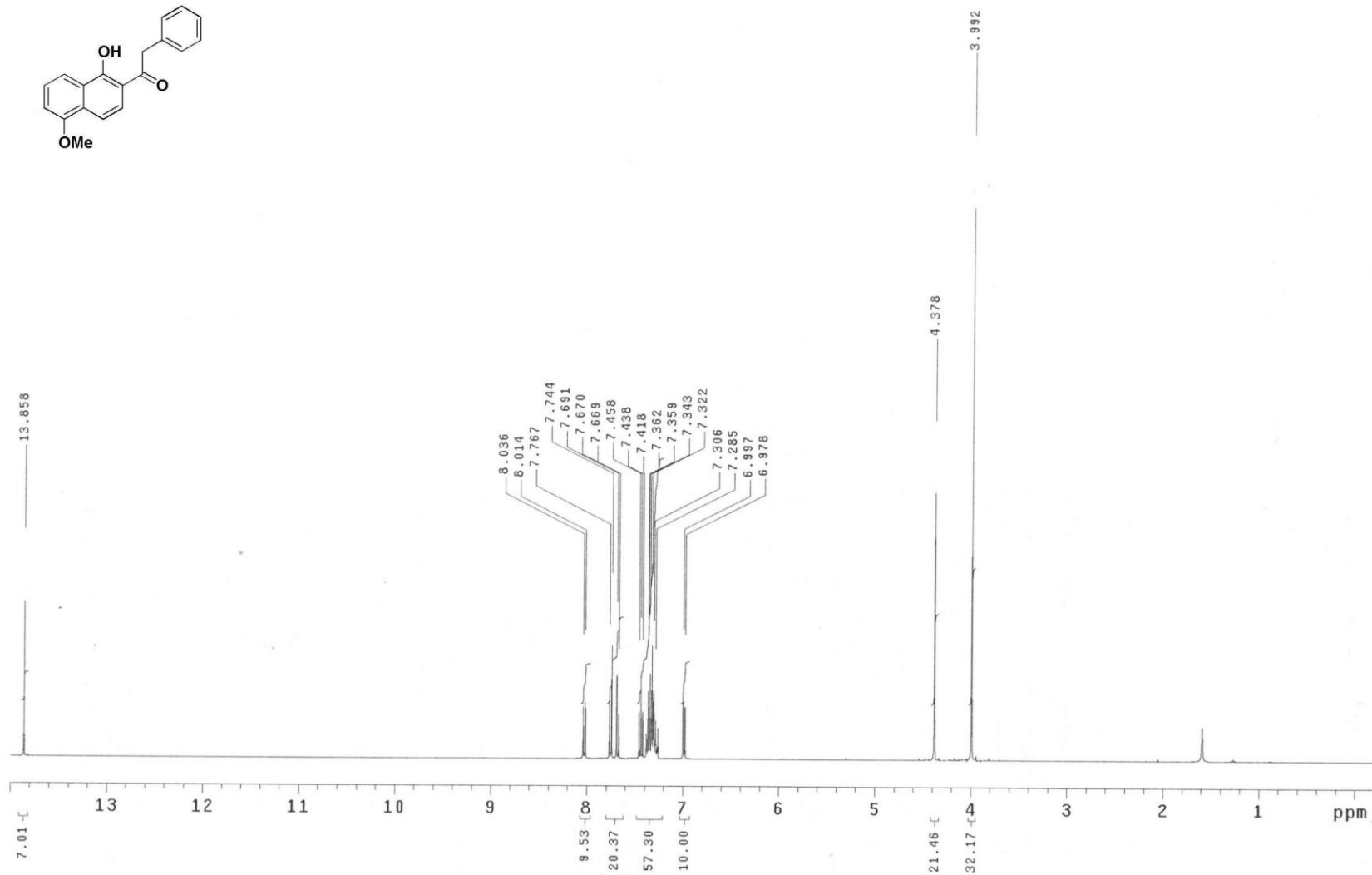
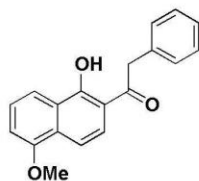
Total 1536 repetitions



Compound 4n (¹H-NMR spectral data)

YTOMeP

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Jun 11 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 4n (¹³C-NMR spectral data)

YTOMeP

Pulse Sequence: s2pu1

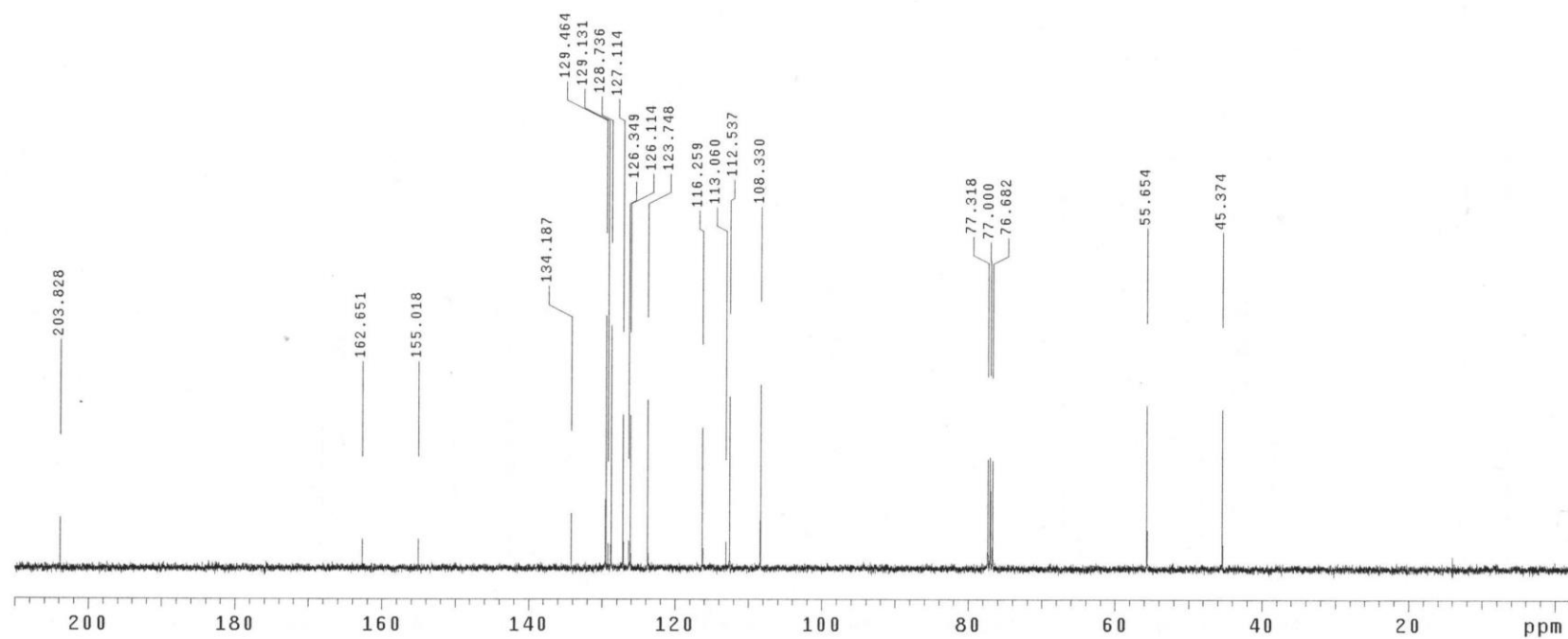
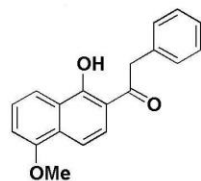
UNITYplus-400 "unity400"

Date: Jun 11 2018

Solvent: CDCl₃

Ambient temperature

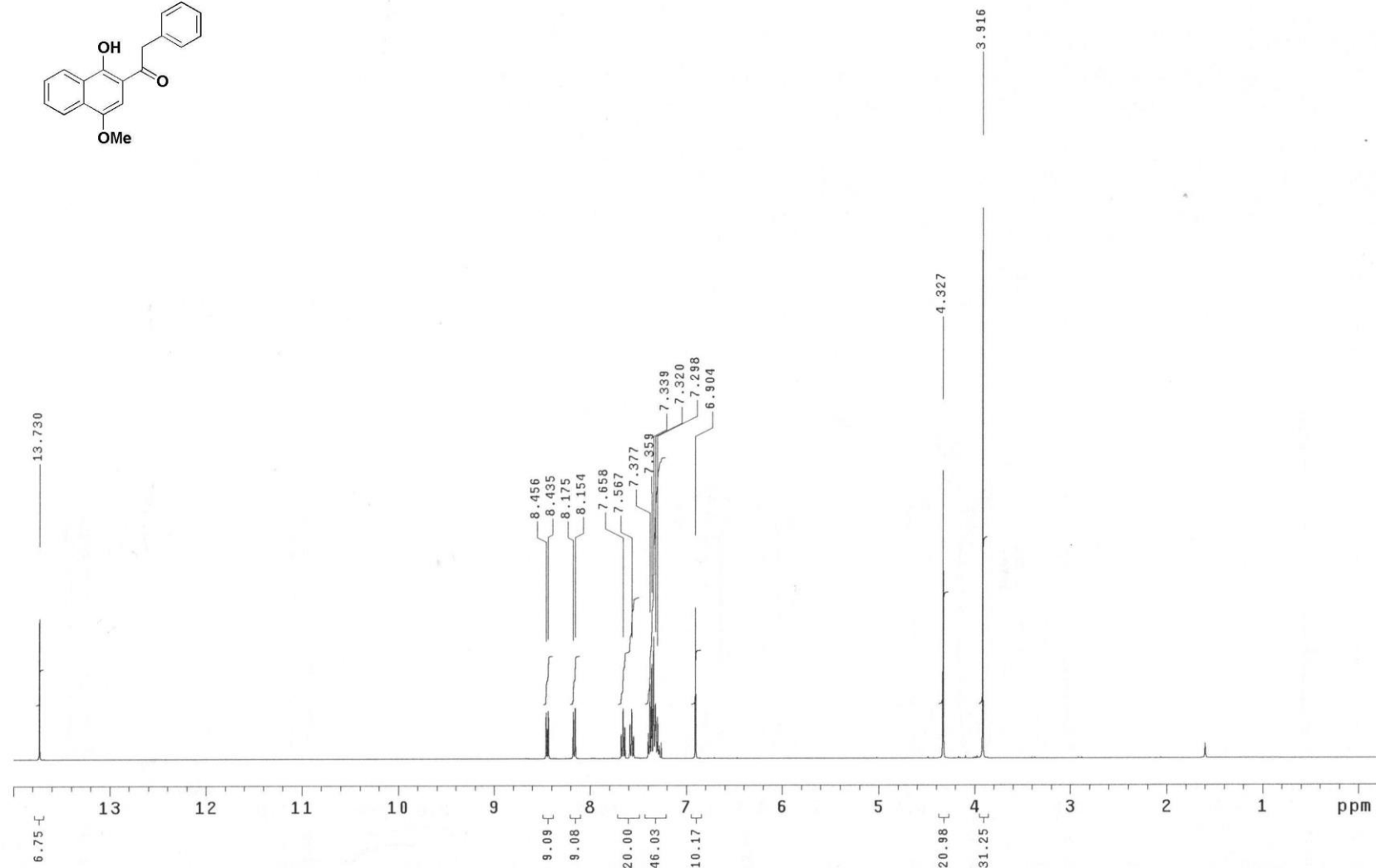
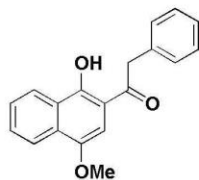
Total 672 repetitions



Compound 4o (¹H-NMR spectral data)

YT4-OMeP

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Aug 6 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 4o (¹³C-NMR spectral data)

YT4-OMeP

Pulse Sequence: s2pu1

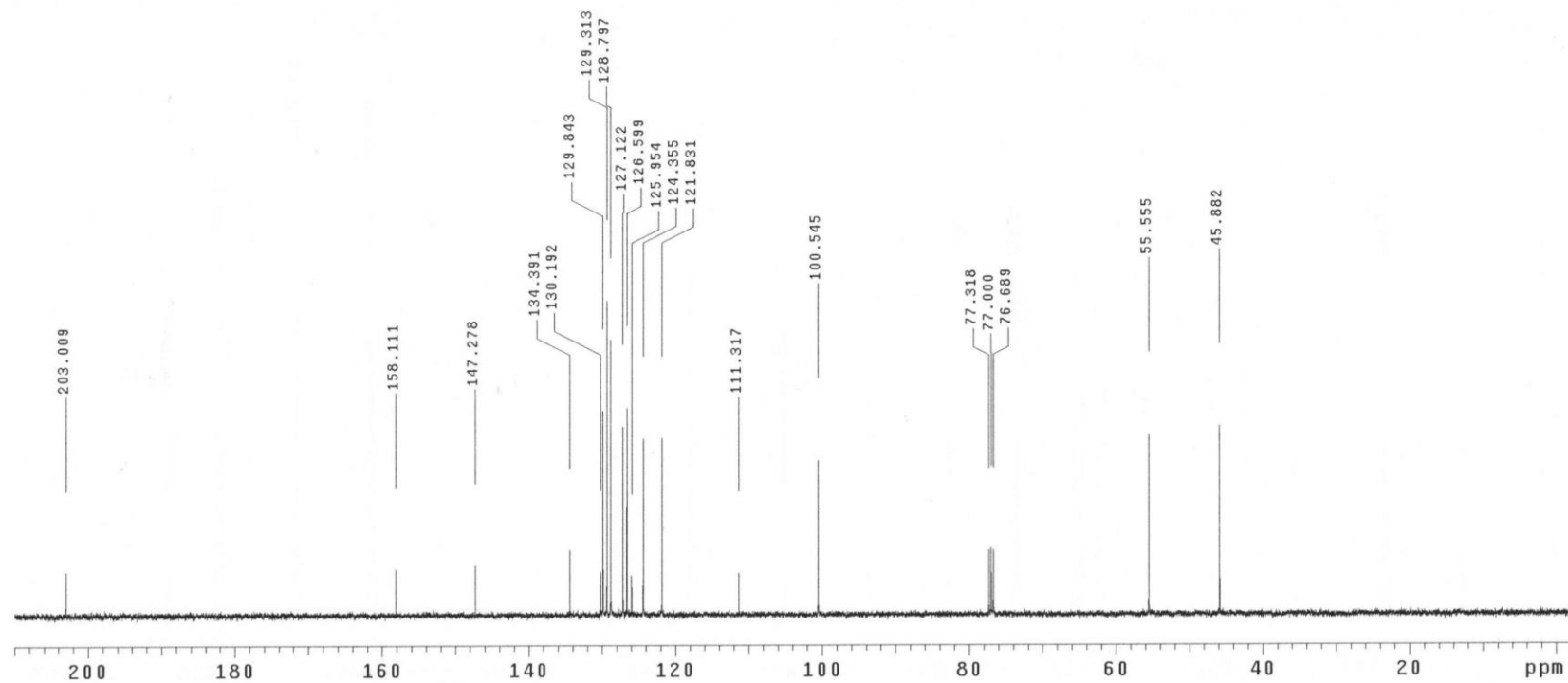
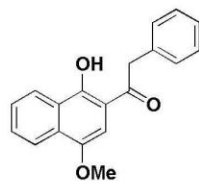
UNITYplus-400 "unity400"

Date: Aug 6 2018

Solvent: CDCl₃

Ambient temperature

Total 256 repetitions



Compound 4p (¹H-NMR spectral data-1)

YT4C1Ph

Pulse Sequence: s2pu1

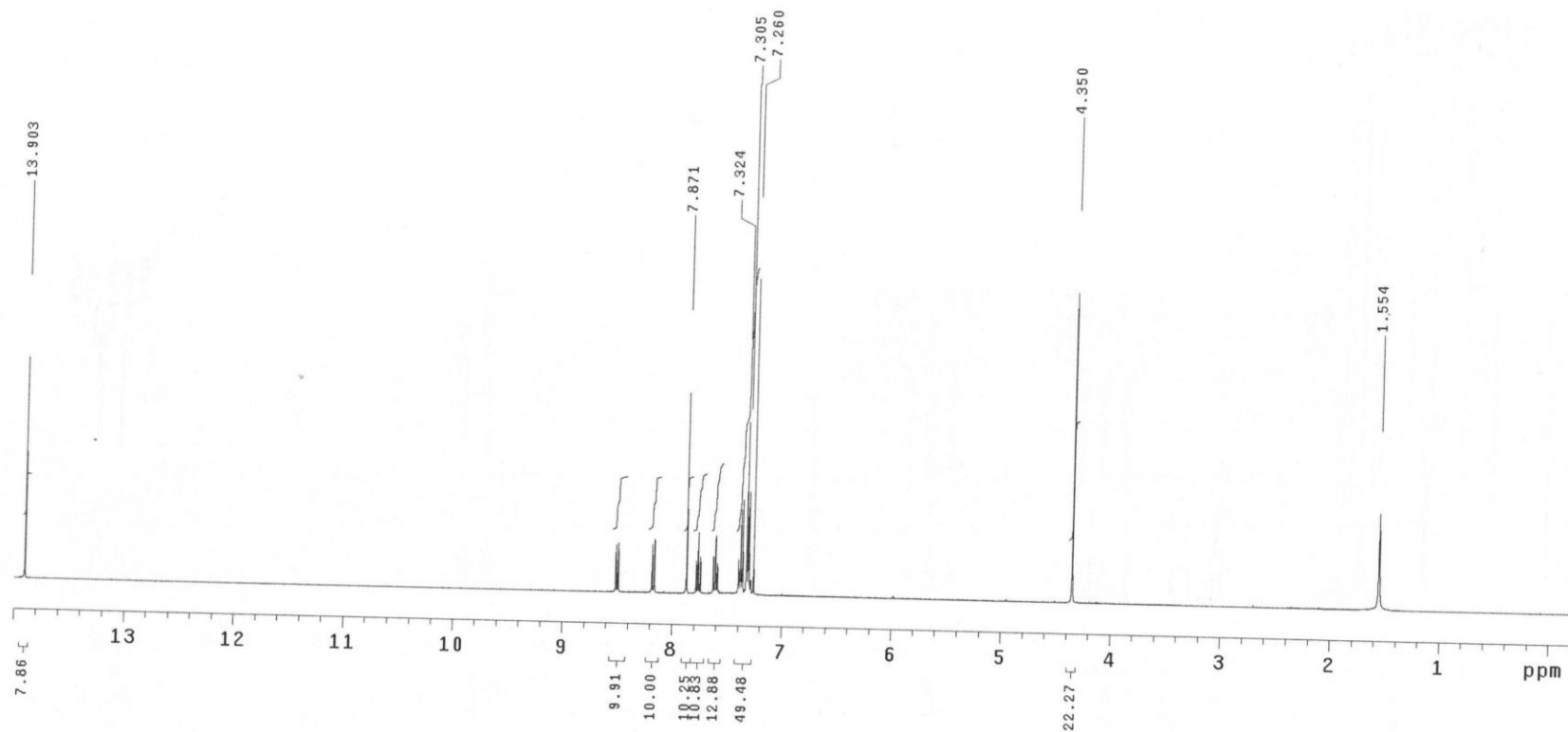
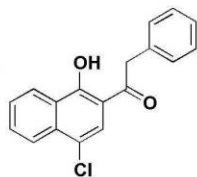
UNITYplus-400 "unity400"

Date: Aug 9 2018

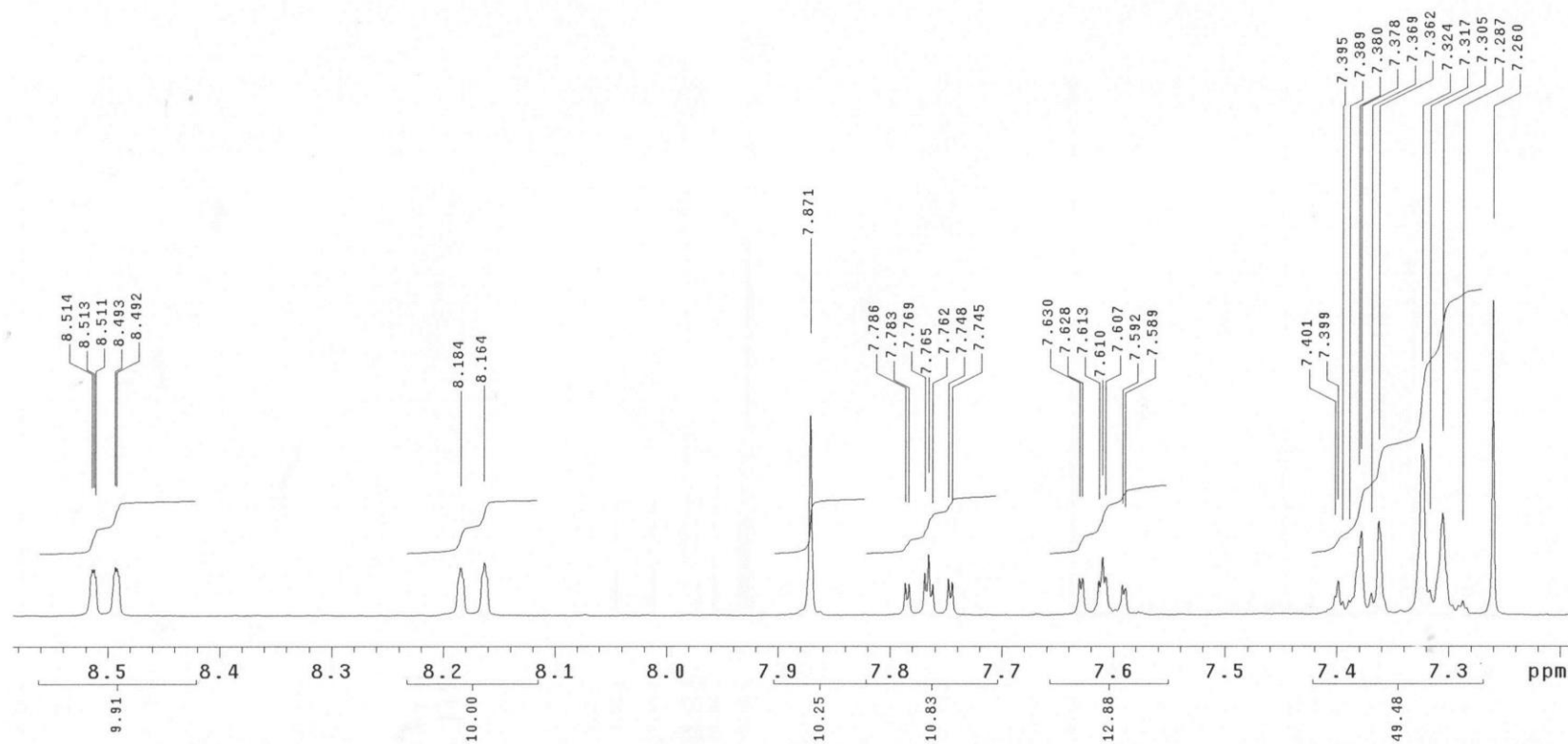
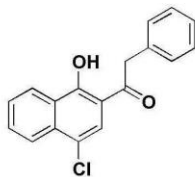
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



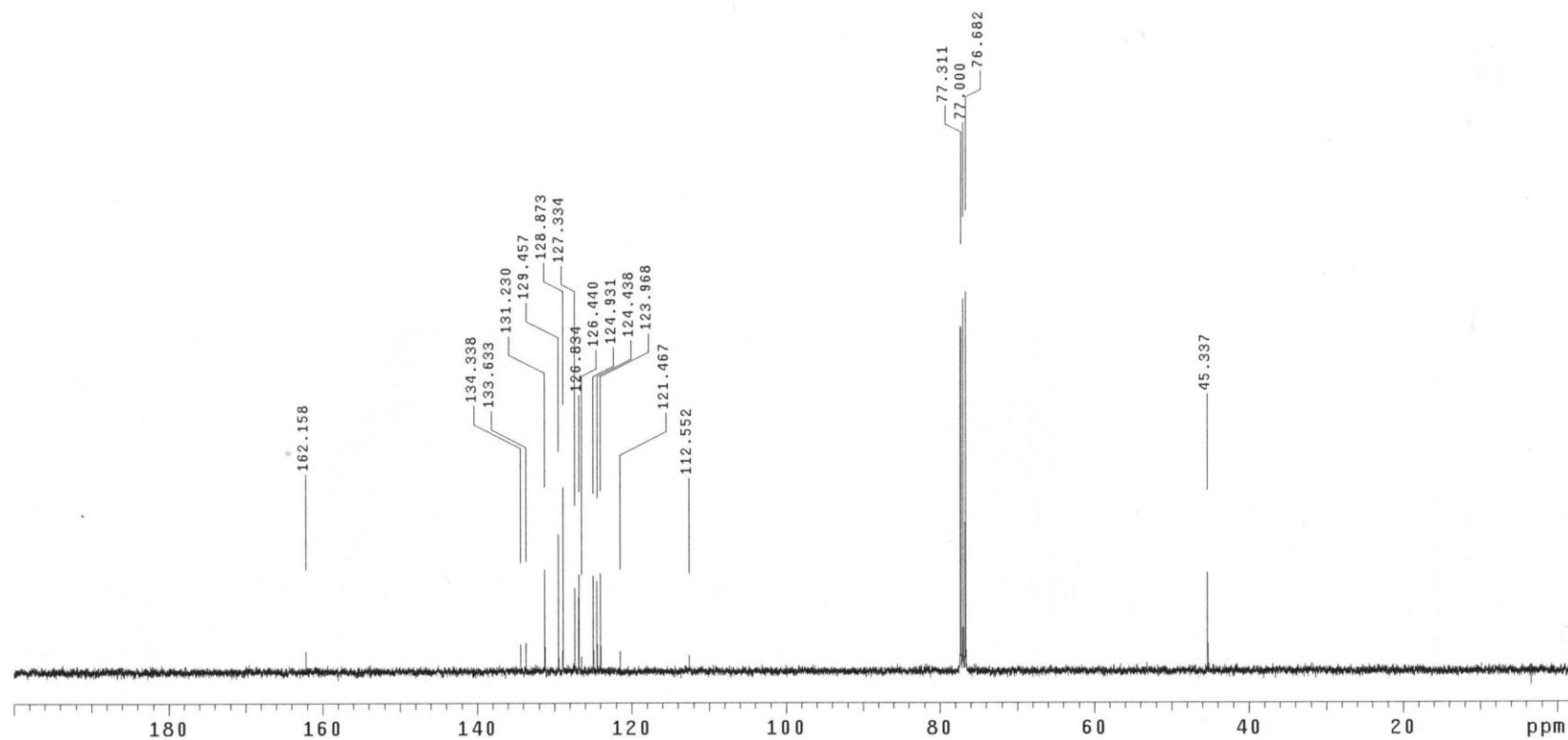
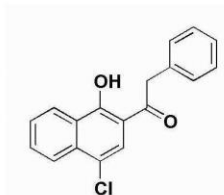
Compound 4p (¹H-NMR spectral data-2)



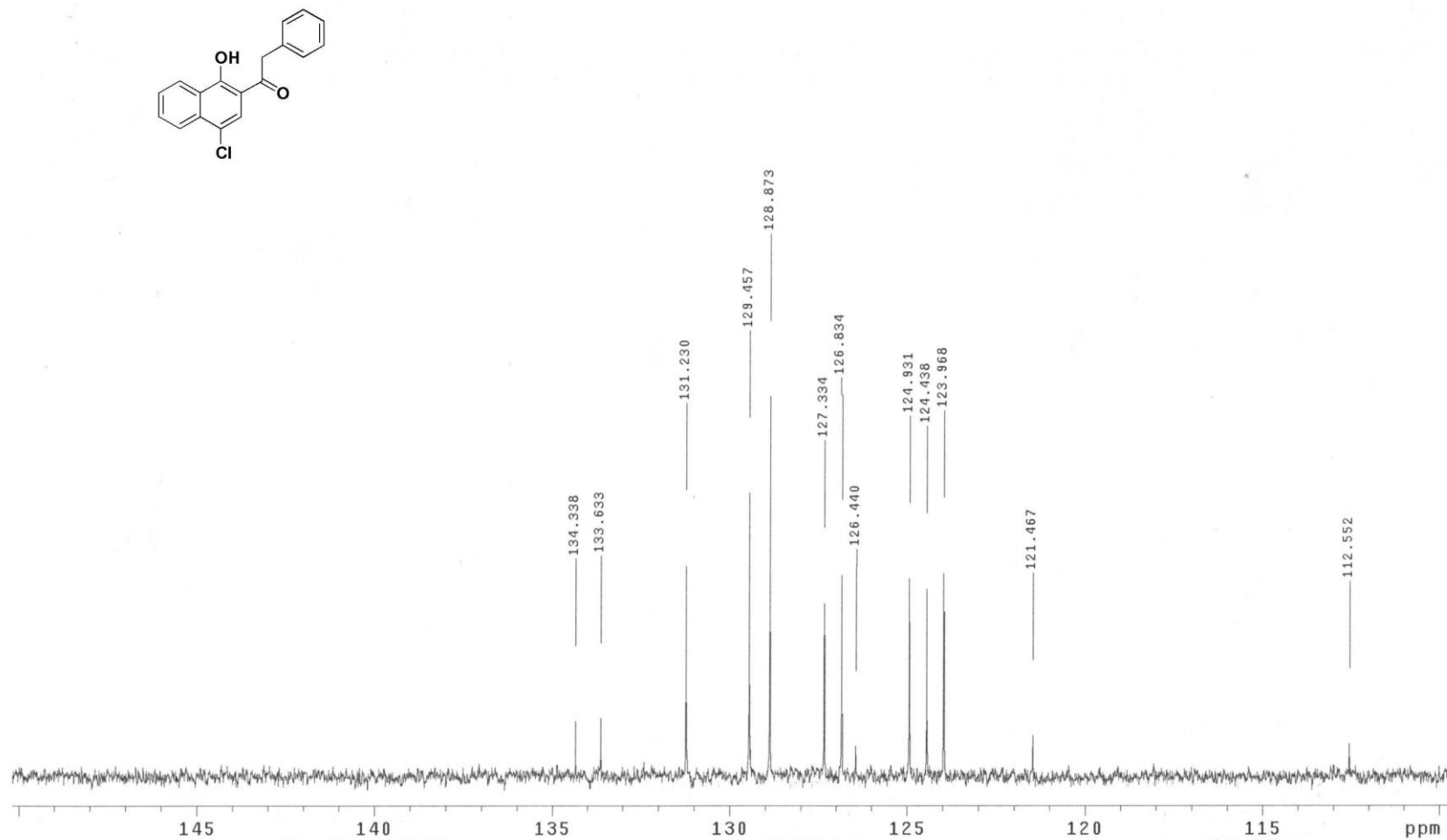
Compound 4p (¹³C-NMR spectral data-1)

YT4C1Ph

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Aug 9 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 5104 repetitions



Compound 4p (^{13}C -NMR spectral data-2)



Compound 4q (¹H-NMR spectral data)

YT2Xup

Pulse Sequence: s2pu1

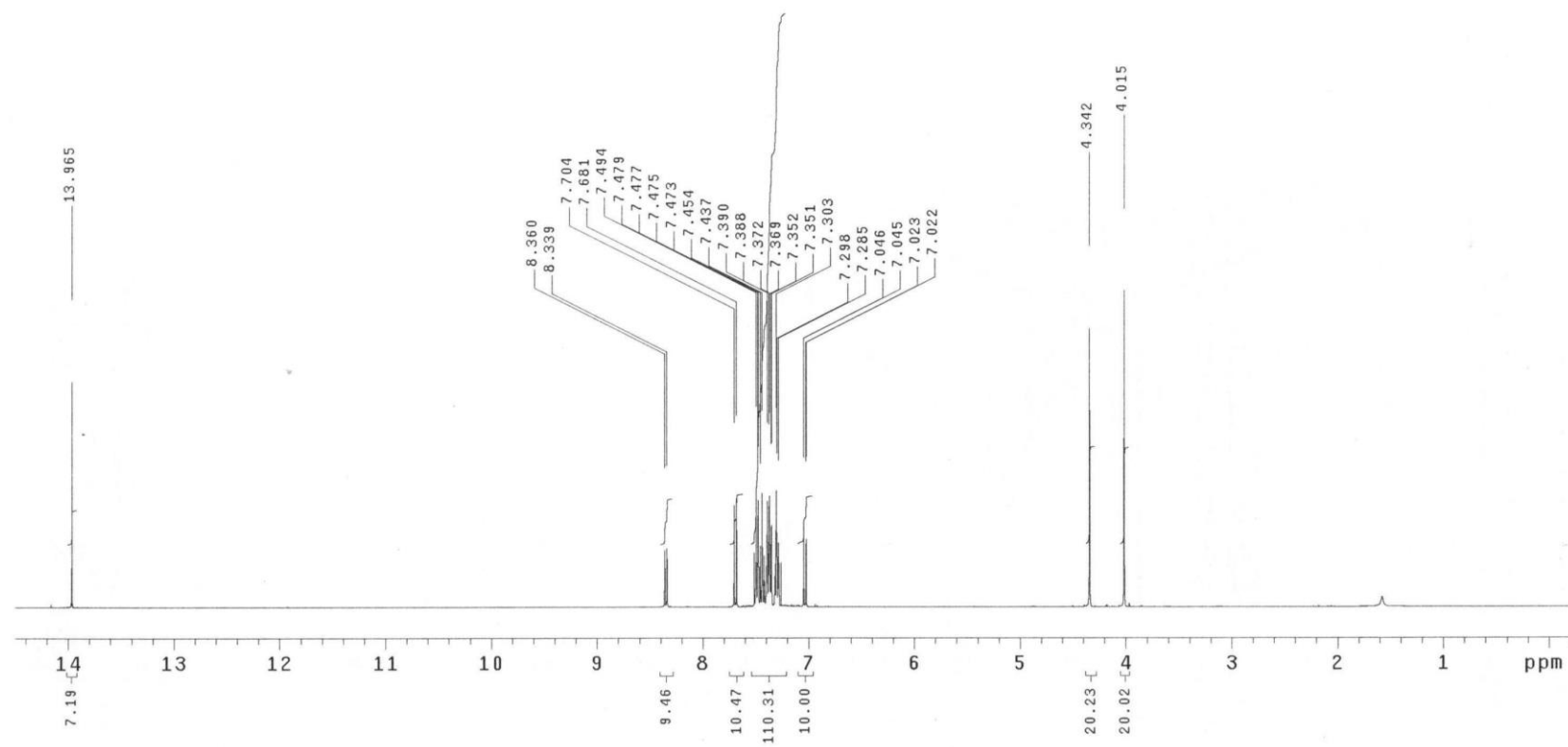
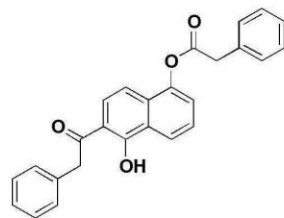
UNITYplus-400 "unity400"

Date: Jun 6 2018

Solvent: CDCl₃

Ambient temperature

Total 32 Repetitions



Compound 4q (¹³C-NMR spectral data)

YT2Xup

Pulse Sequence: s2pu1

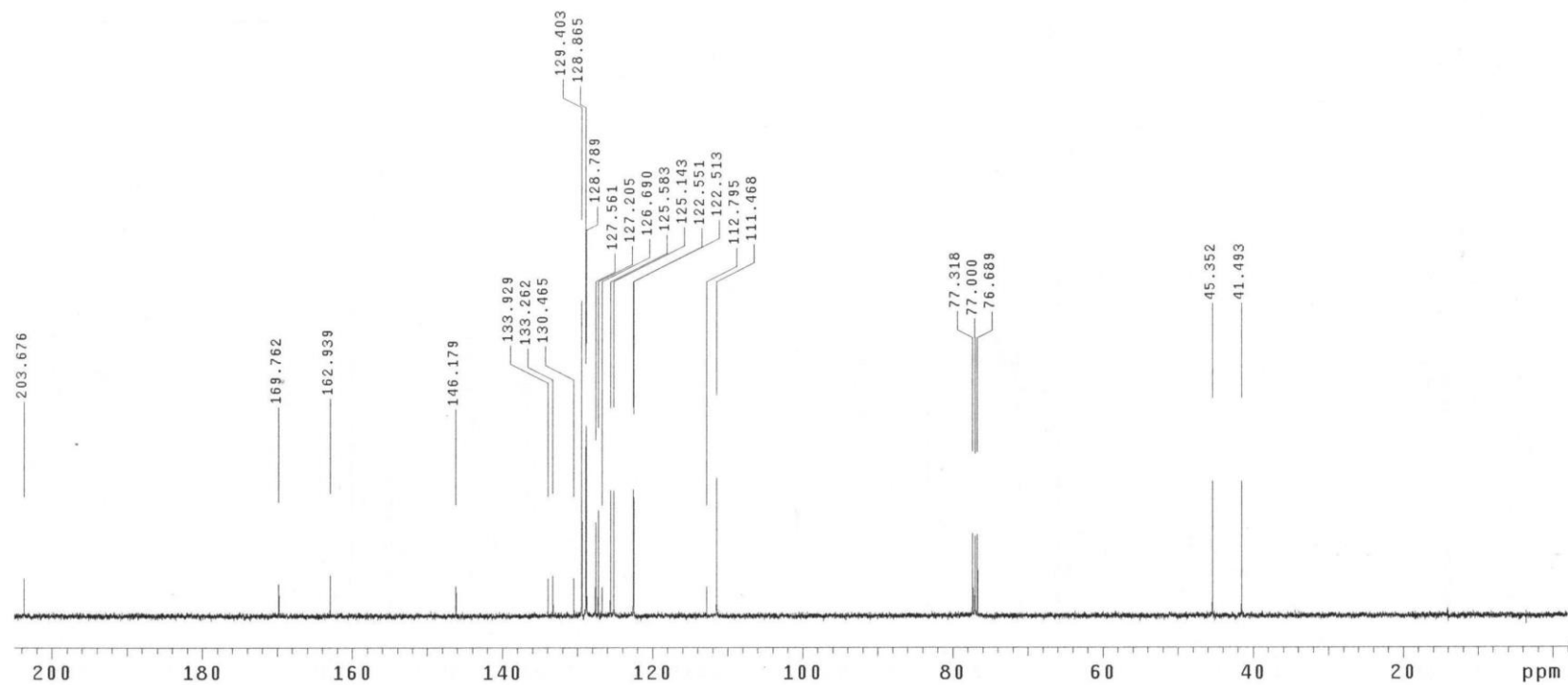
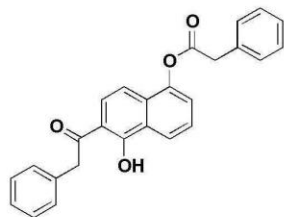
UNITYplus-400 "unity400"

Date: Jun 6 2018

Solvent: CDCl₃

Ambient temperature

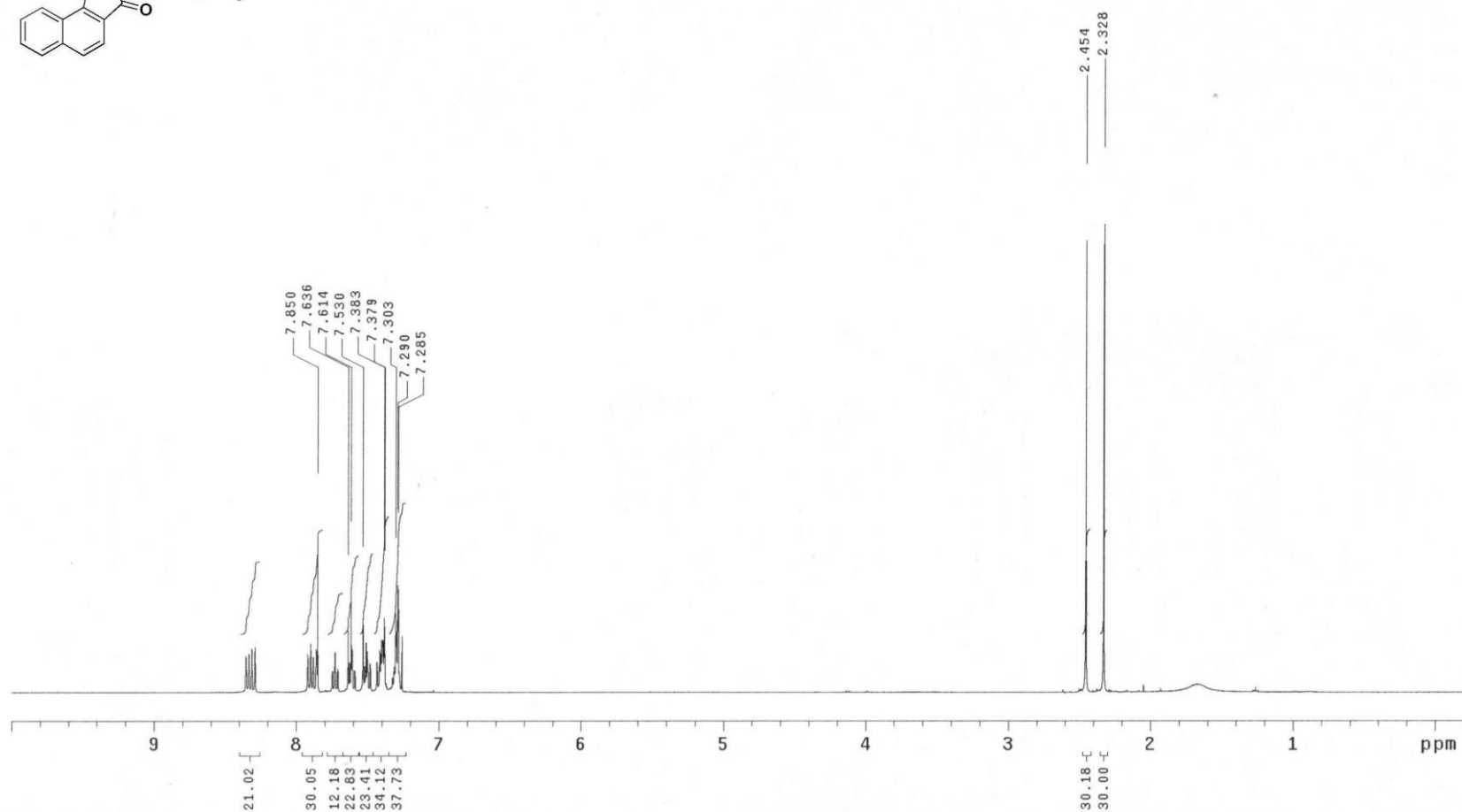
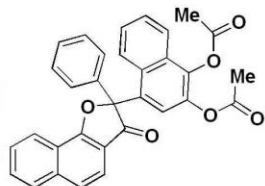
Total 544 repetitions



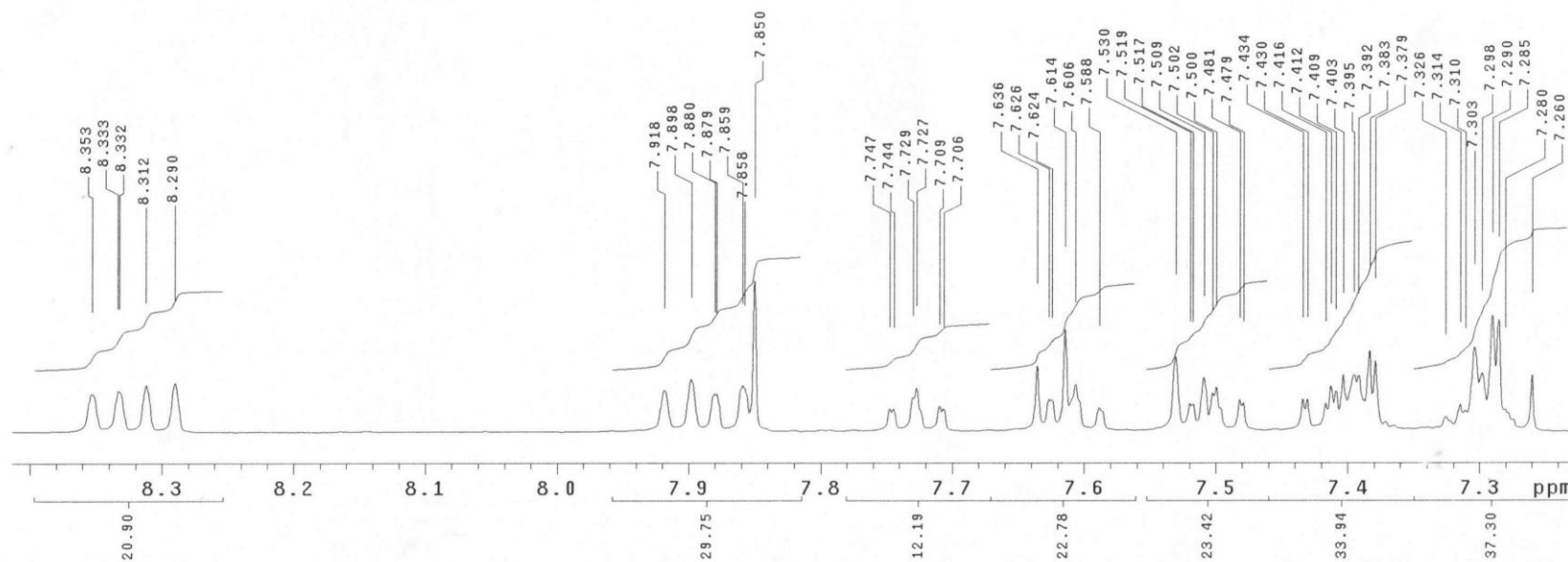
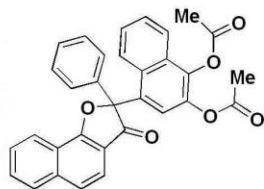
Compound 5a (¹H-NMR spectral data-1)

YT4749

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Mar 5 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 56 repetitions



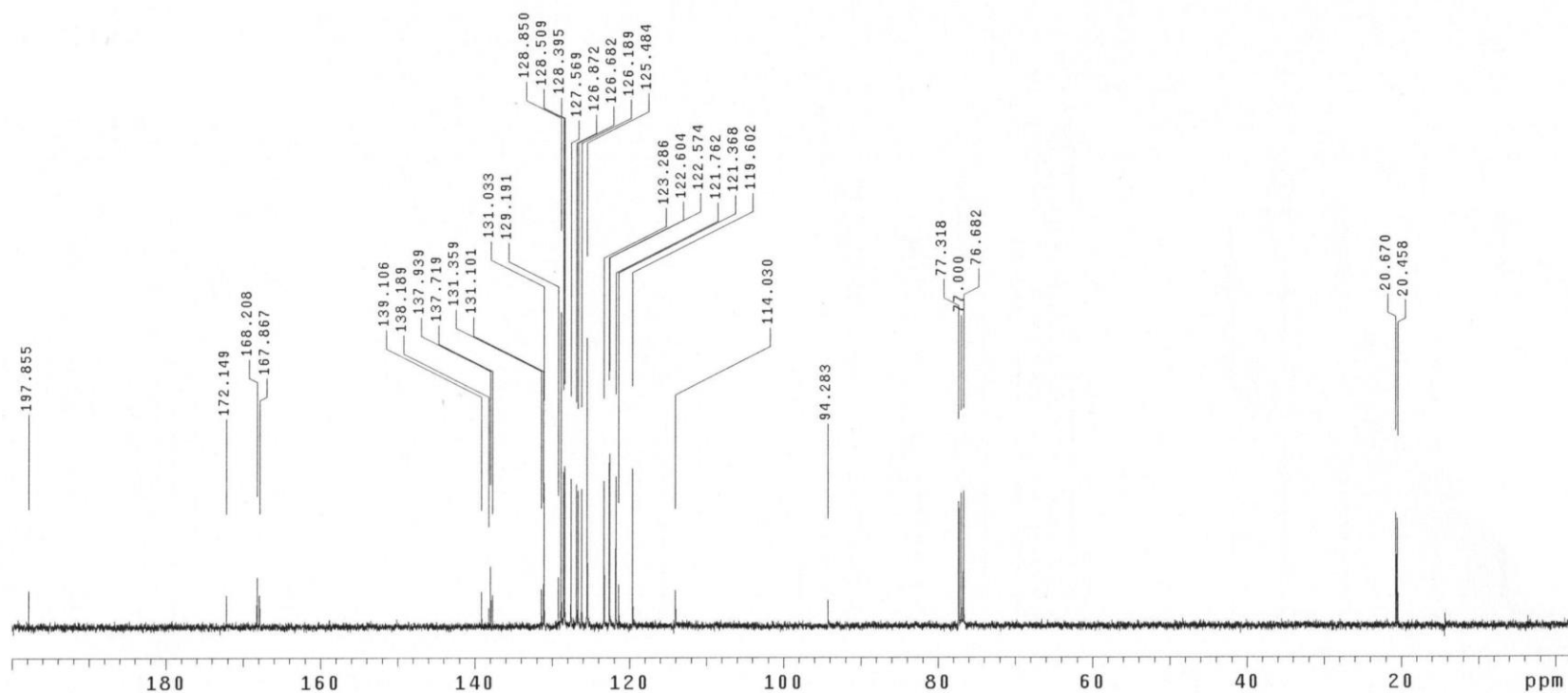
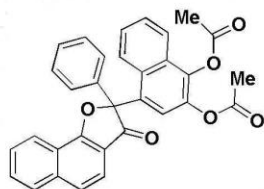
Compound 5a (¹H-NMR spectral data-2)



Compound 5a (¹³C-NMR spectral data)

YT4749

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Mar 5 2018
Solvent: CDCl₃
Ambient temperature
Total 3360 repetitions



Compound 5b (¹H-NMR spectral data-1)

YT2Me0

Pulse Sequence: s2pu1

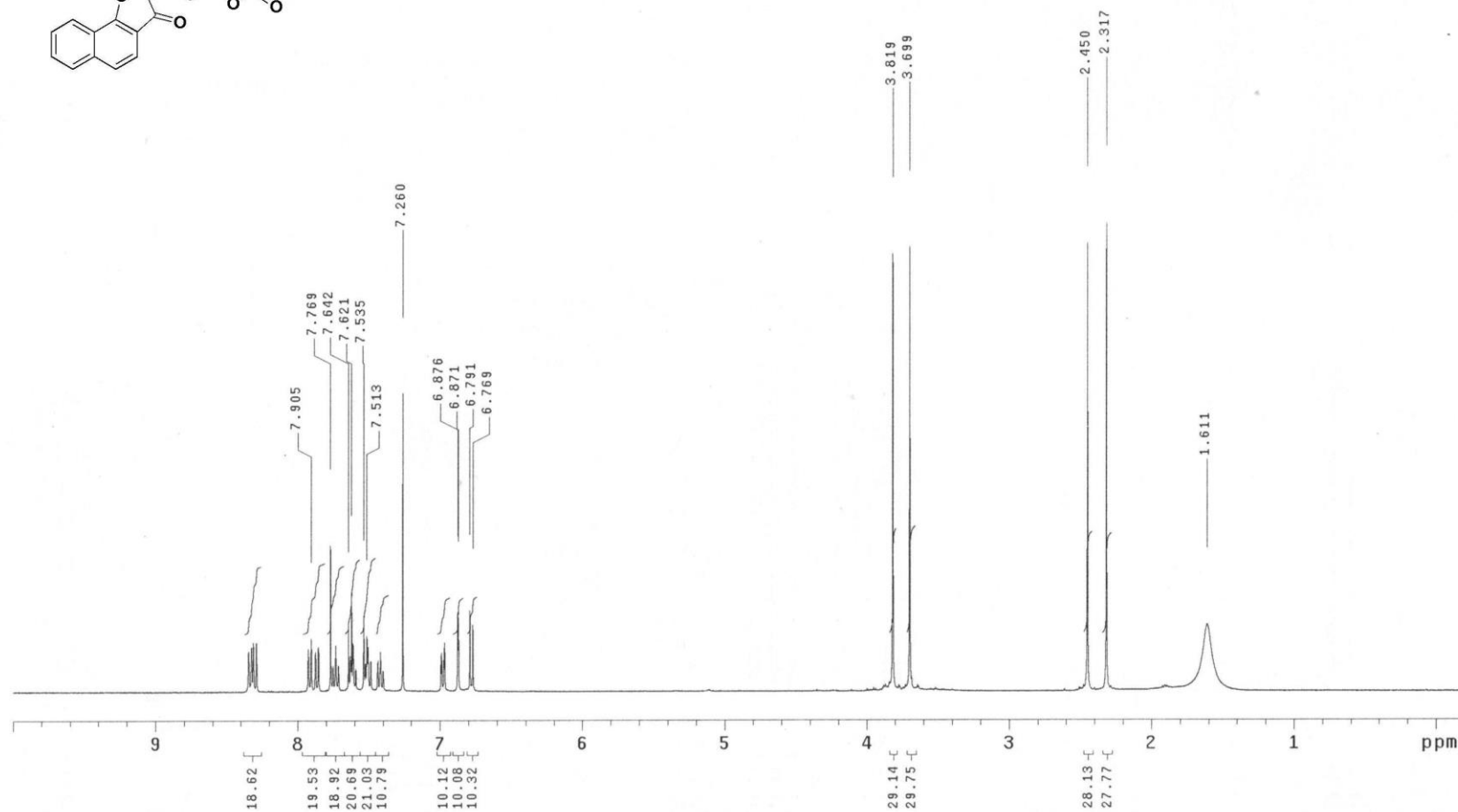
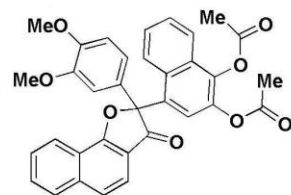
UNITYplus-400 "unity400"

Date: May 10 2018

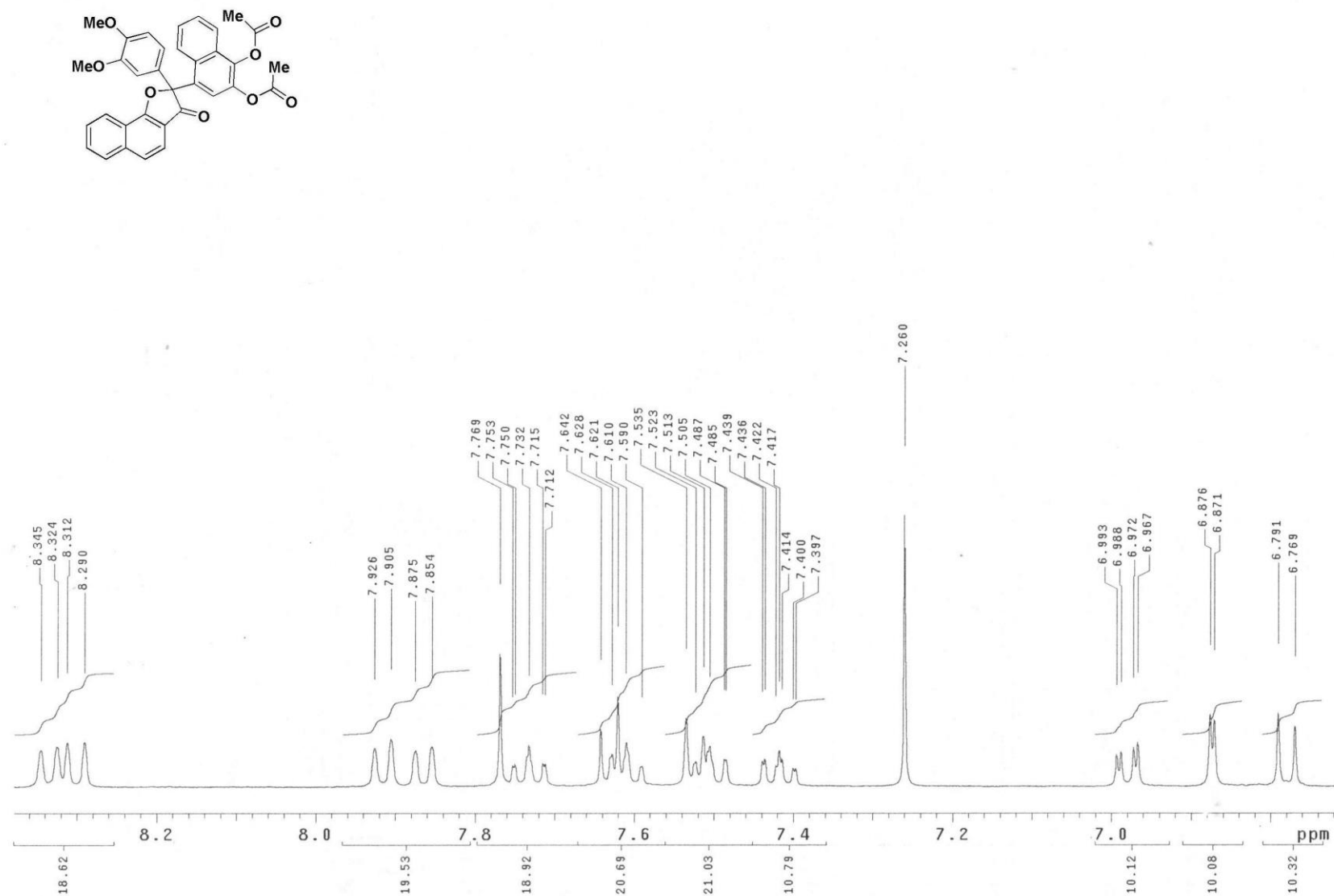
Solvent: CDCl₃

Ambient temperature

Total 80 repetitions



Compound 5b (¹H-NMR spectral data-2)



Compound 5b (¹³C-NMR spectral data-1)

YT2MeO

Pulse Sequence: s2pu1

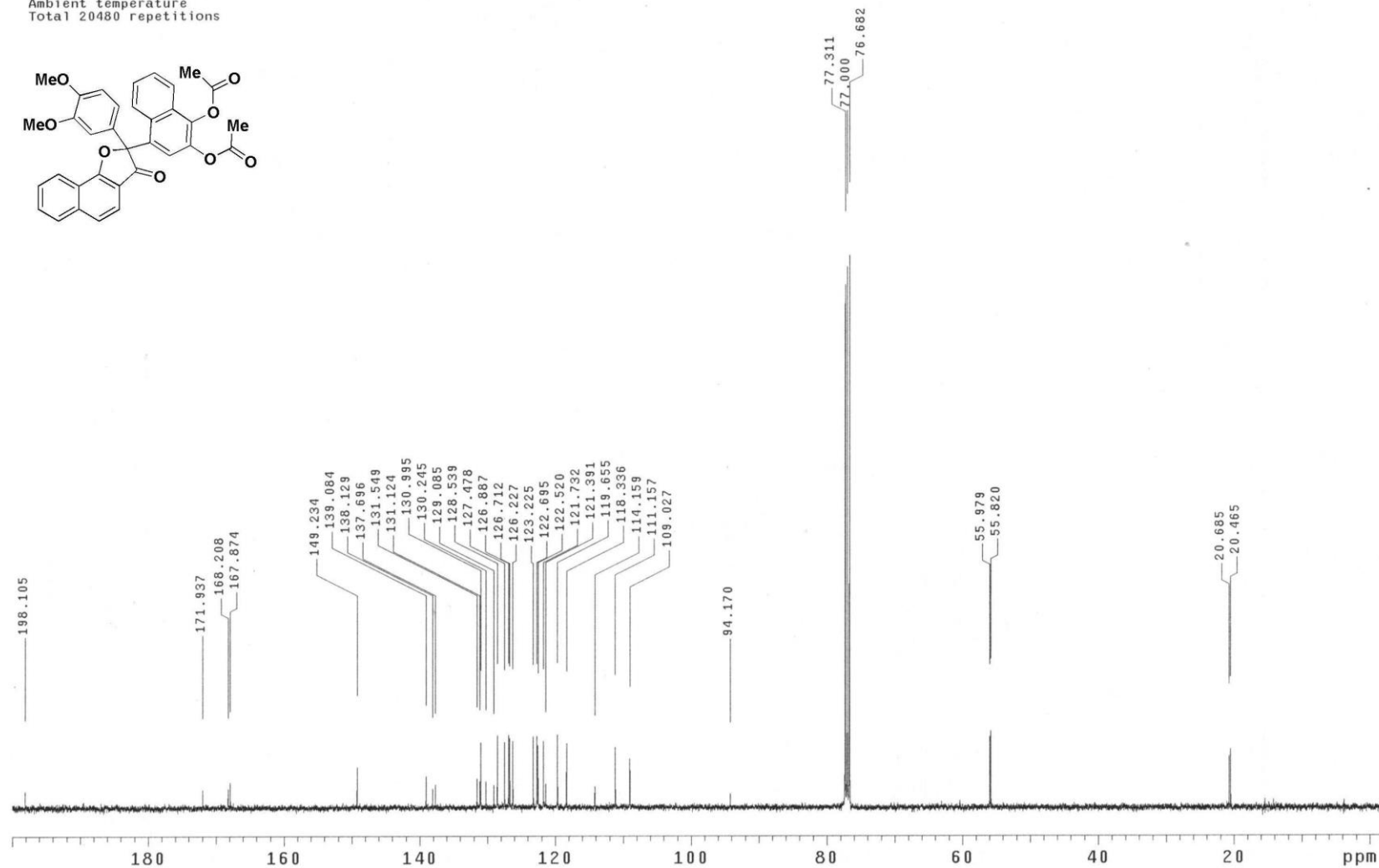
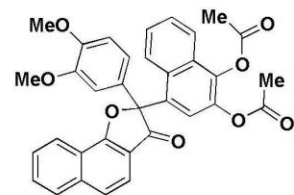
UNITYplus-400 "unity400"

Date: May 10 2018

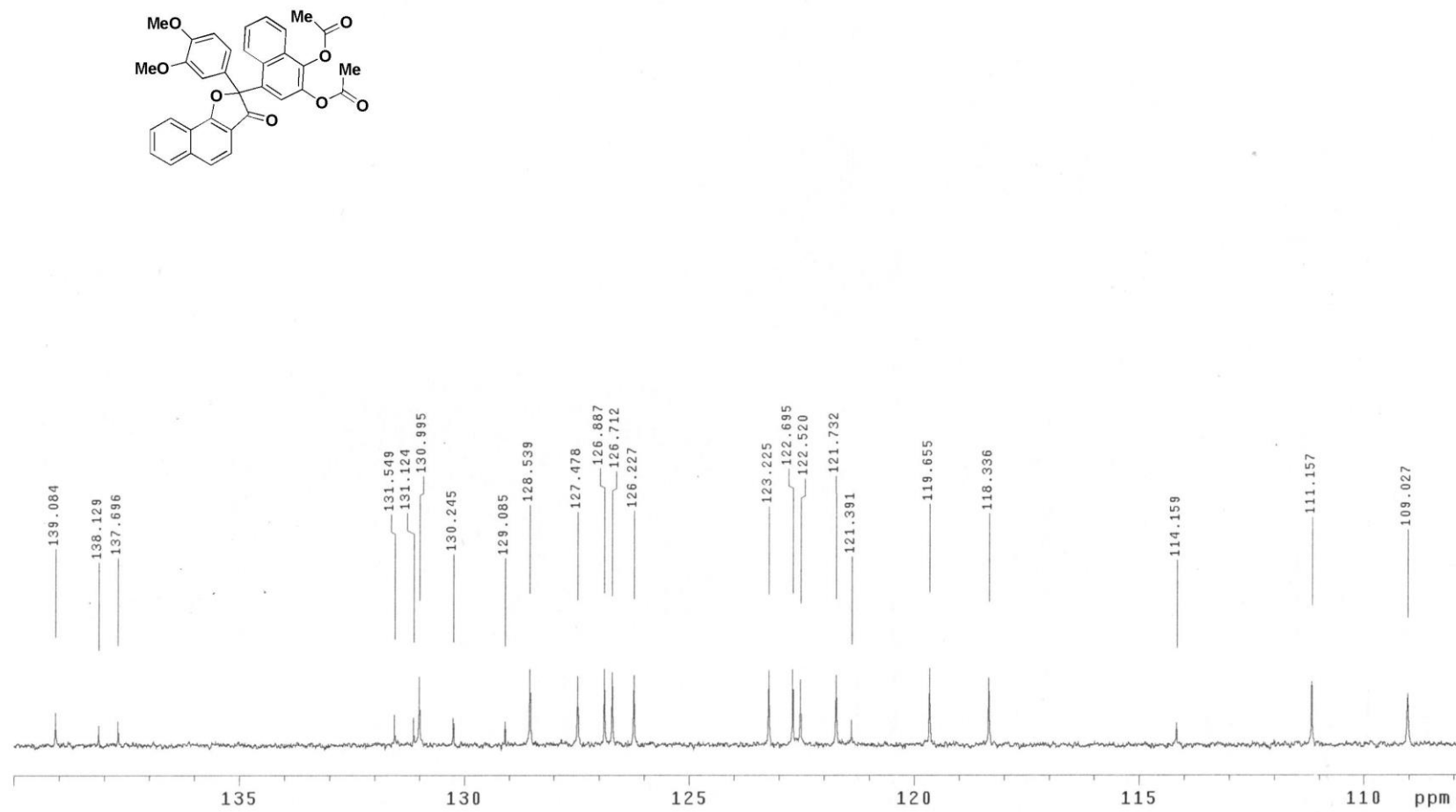
Solvent: CDCl₃

Ambient temperature

Total 20480 repetitions



Compound 5b (^{13}C -NMR spectral data-2)



Compound 5c (¹H-NMR spectral data-1)

YTF4749

Pulse Sequence: s2pu1

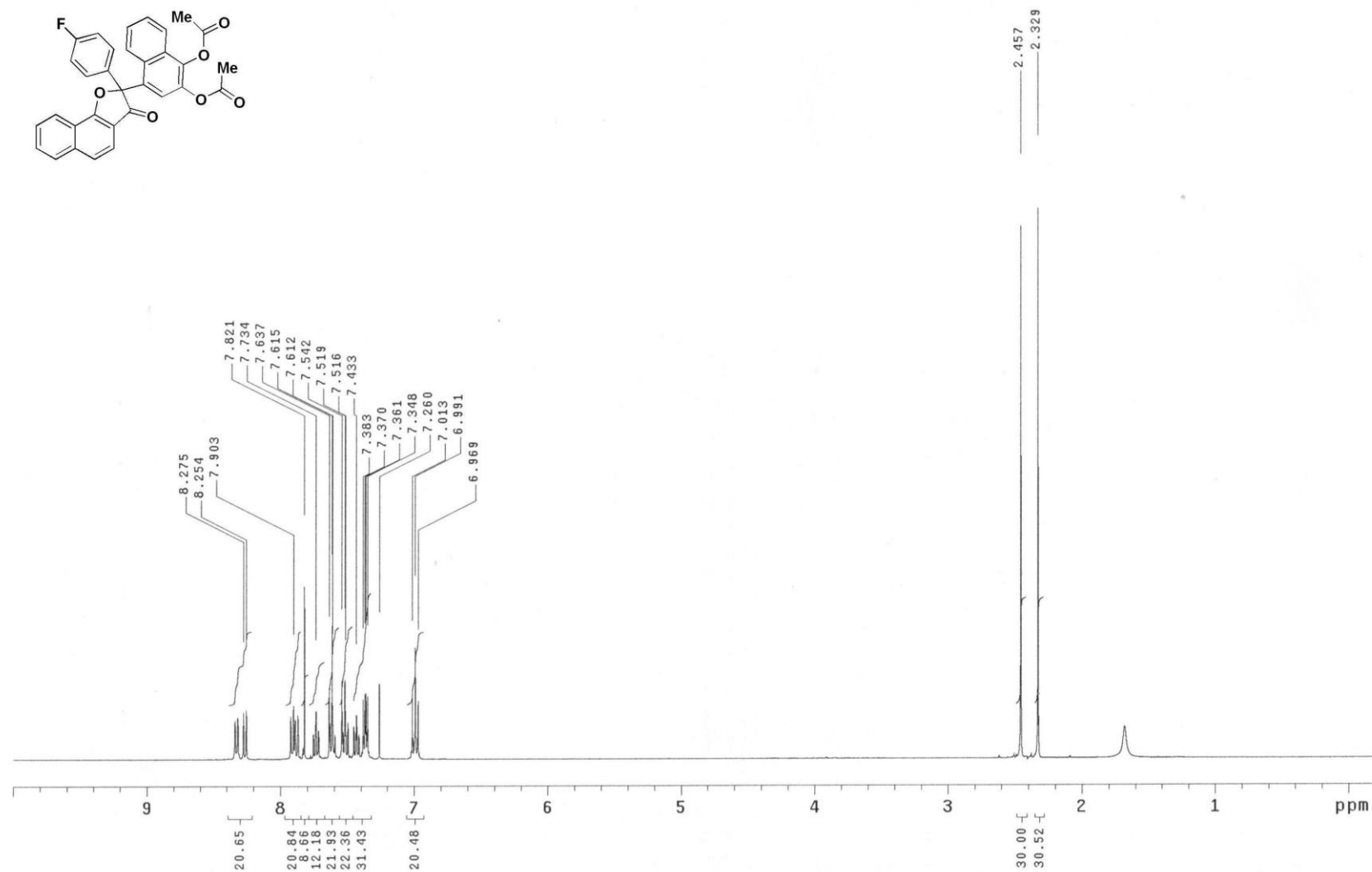
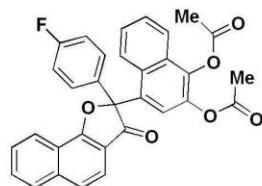
UNITYplus-400 "unity400"

Date: Mar 27 2018

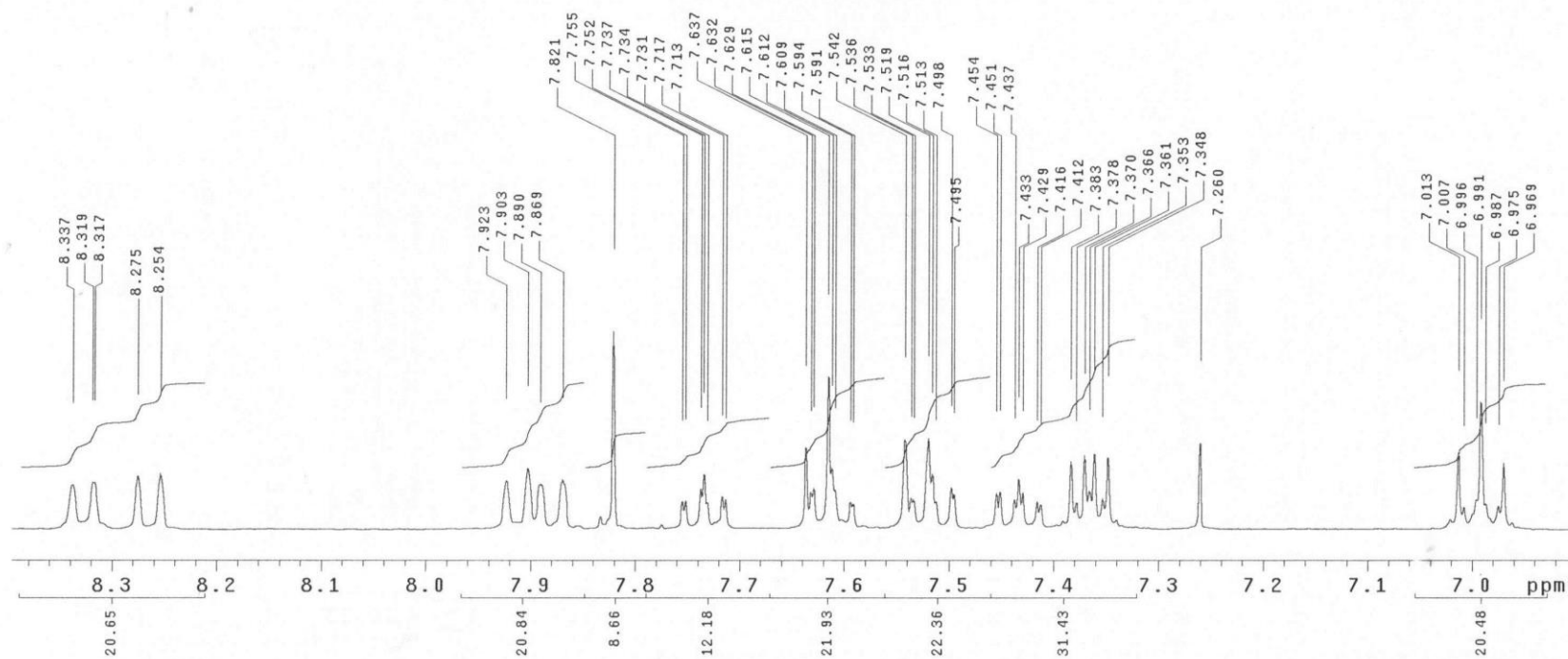
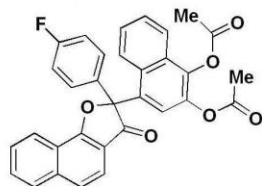
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5c (¹H-NMR spectral data-2)



Compound 5c (¹³C-NMR spectral data)

YTF4749

Pulse Sequence: s2pu1

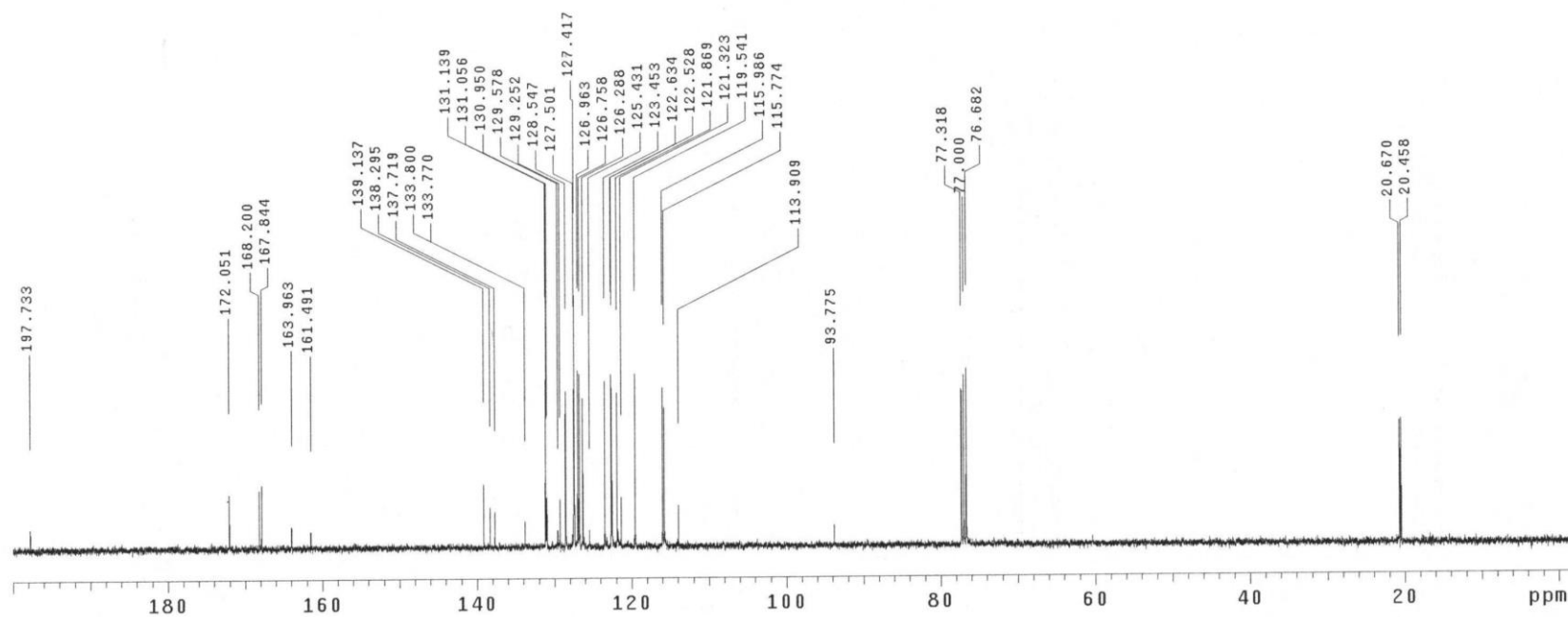
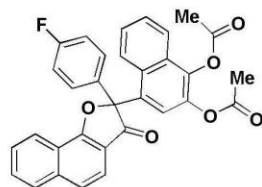
UNITYplus-400 "unity400"

Date: Mar 27 2018

Solvent: CDCl₃

Ambient temperature

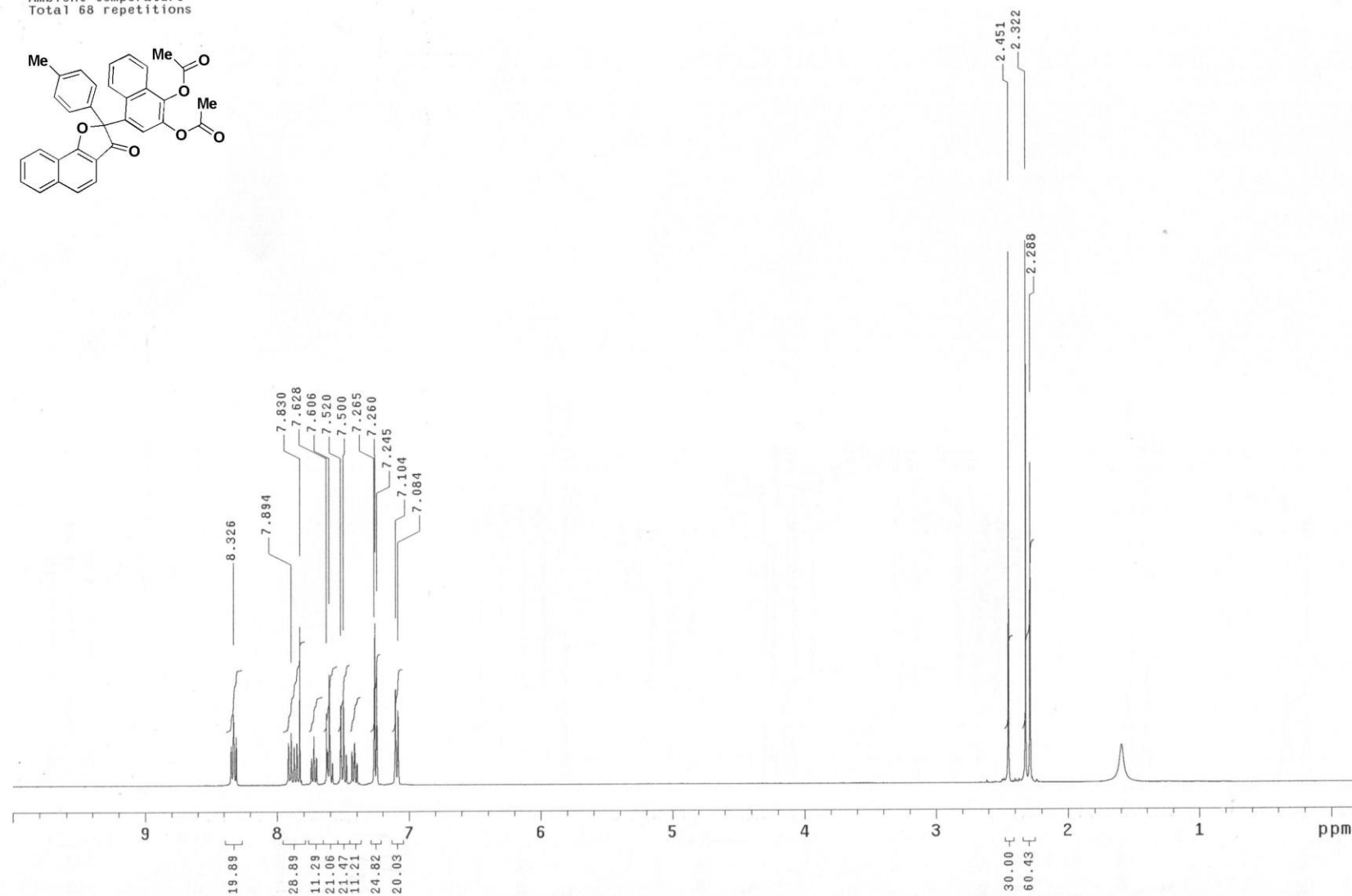
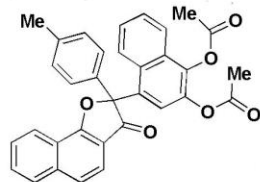
Total 5056 repetitions



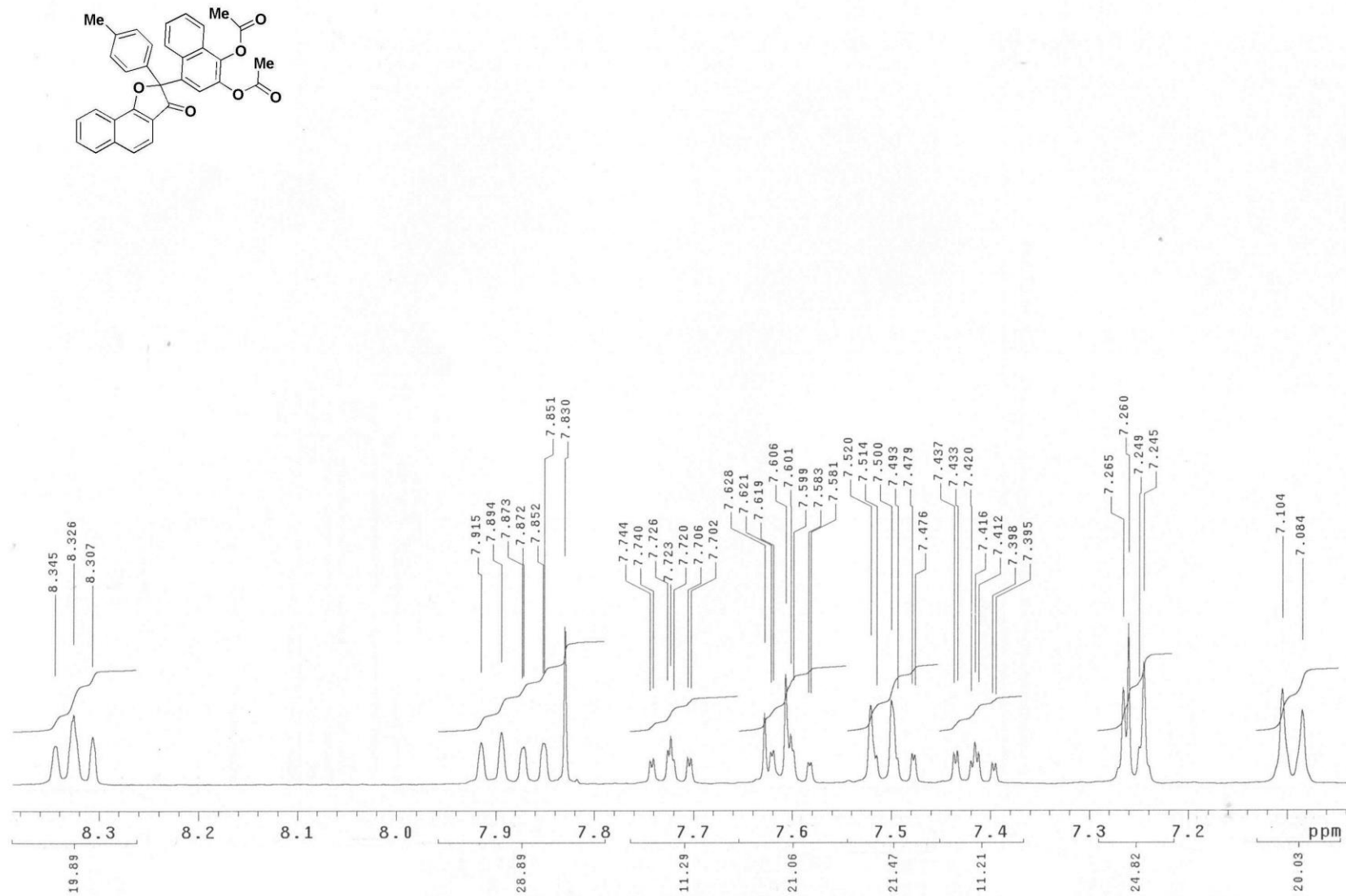
Compound 5d (¹H-NMR spectral data-1)

YTM47

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Jun 27 2019
 Solvent: CDCl₃
 Ambient temperature
 Total 68 repetitions



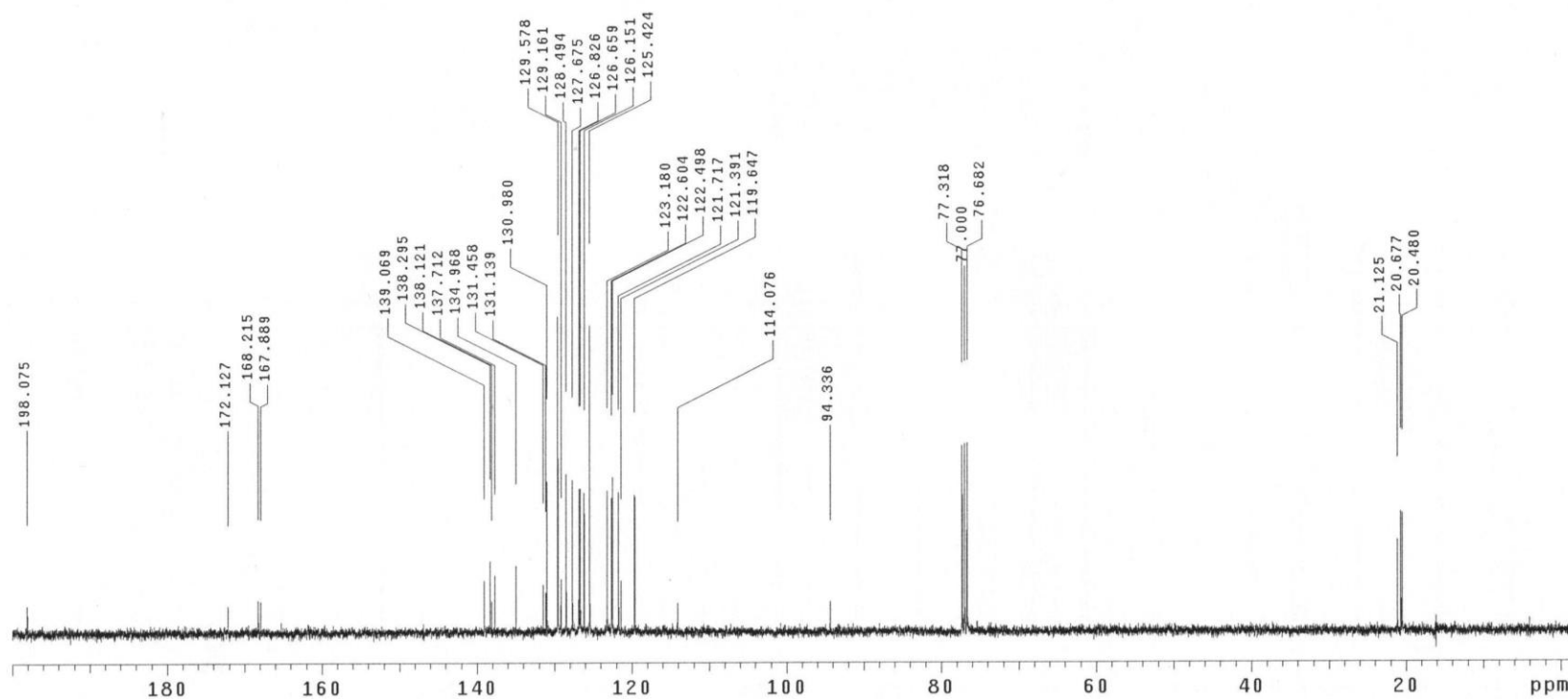
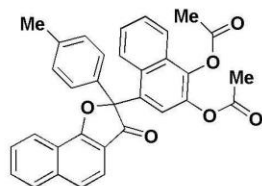
Compound 5d (¹H-NMR spectral data-2)



Compound 5d (¹³C-NMR spectral data)

YTM47

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Mar 12 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 2896 repetitions



Compound 5e (¹H-NMR spectral data-1)

YTPH60

Pulse Sequence: s2pu1

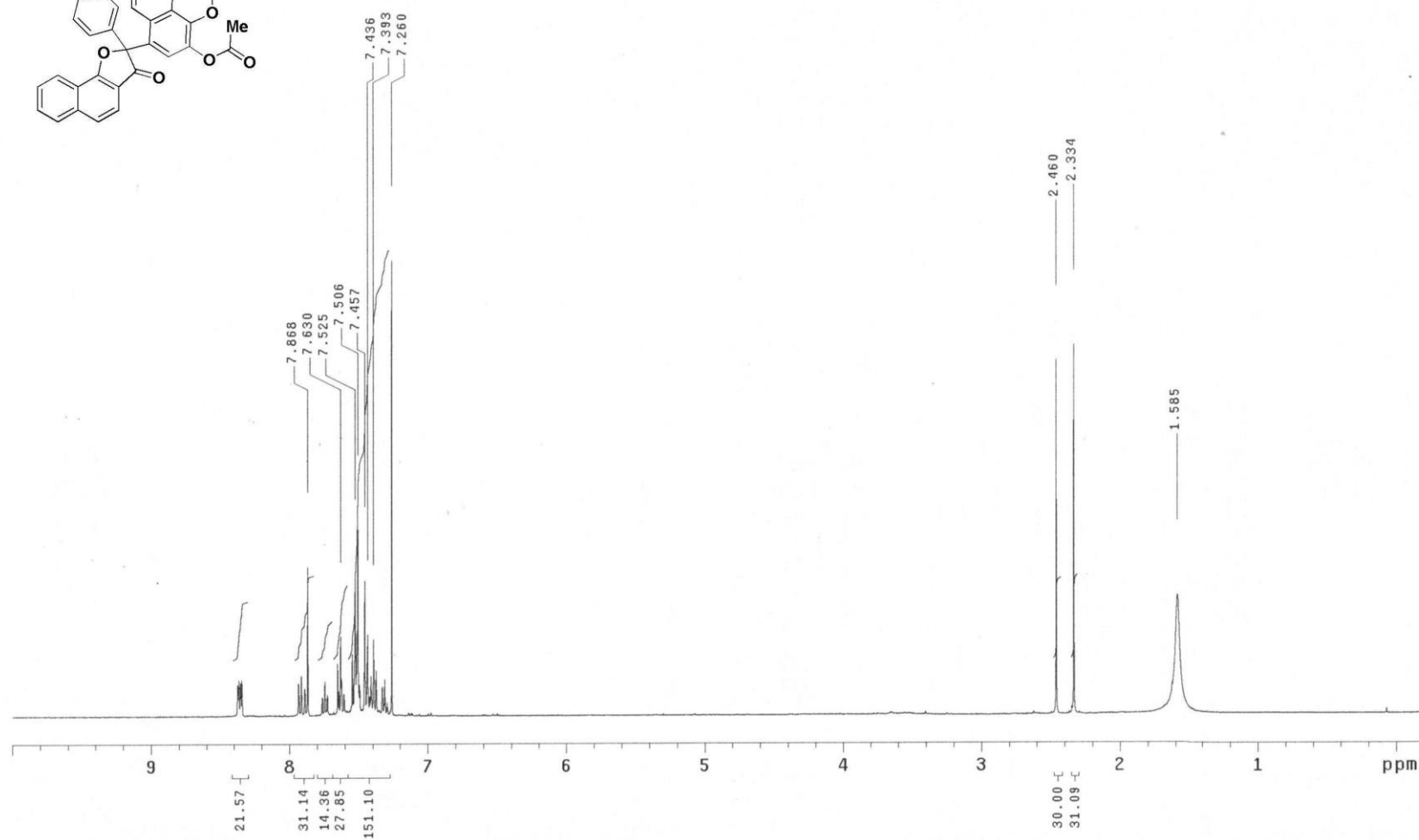
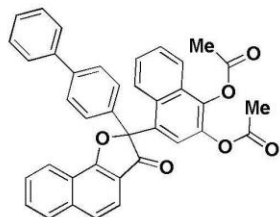
UNITYplus-400 "unity400"

Date: May 3 2018

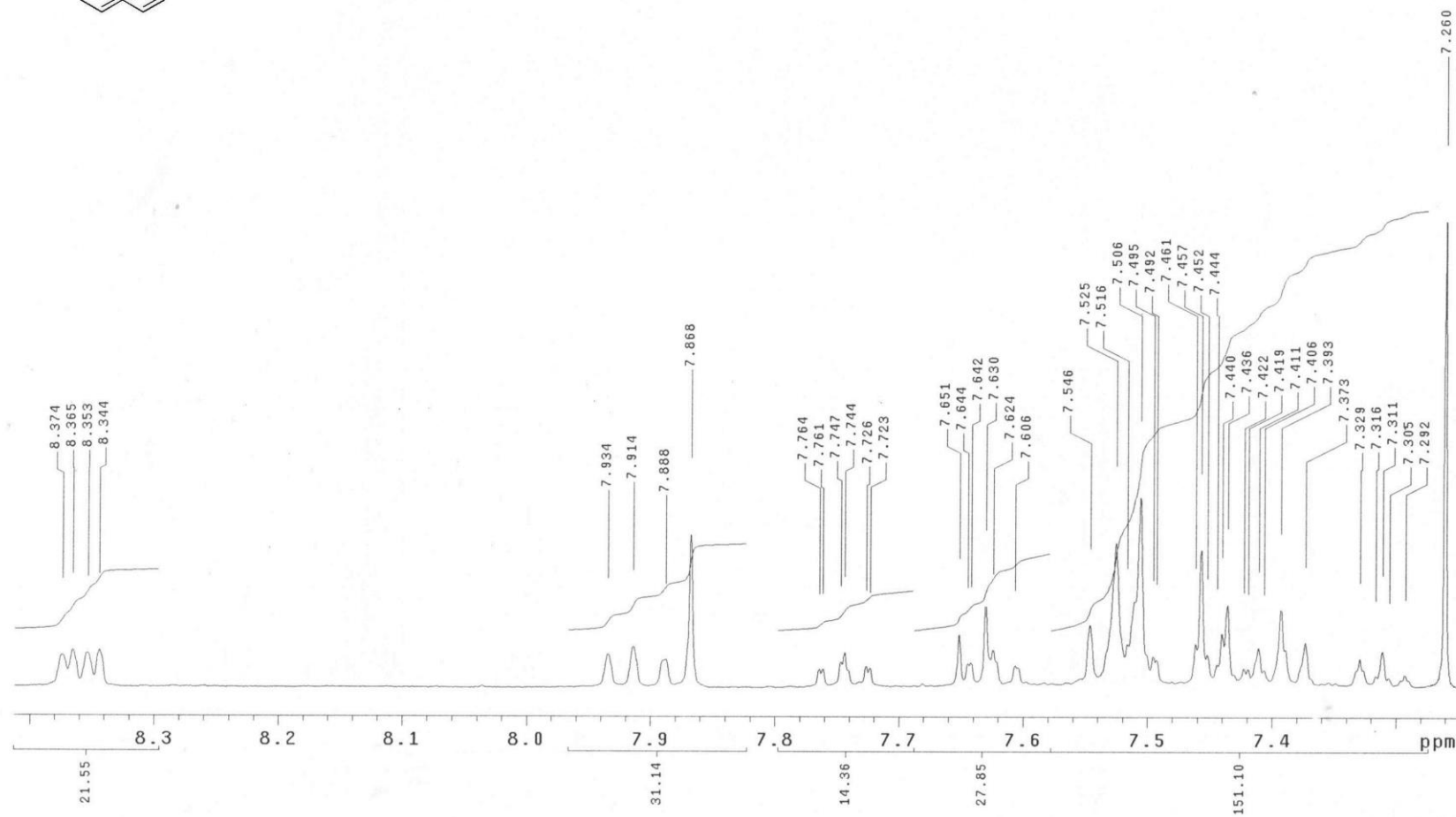
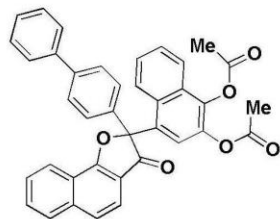
Solvent: CDCl₃

Ambient temperature

Total 80 repetitions



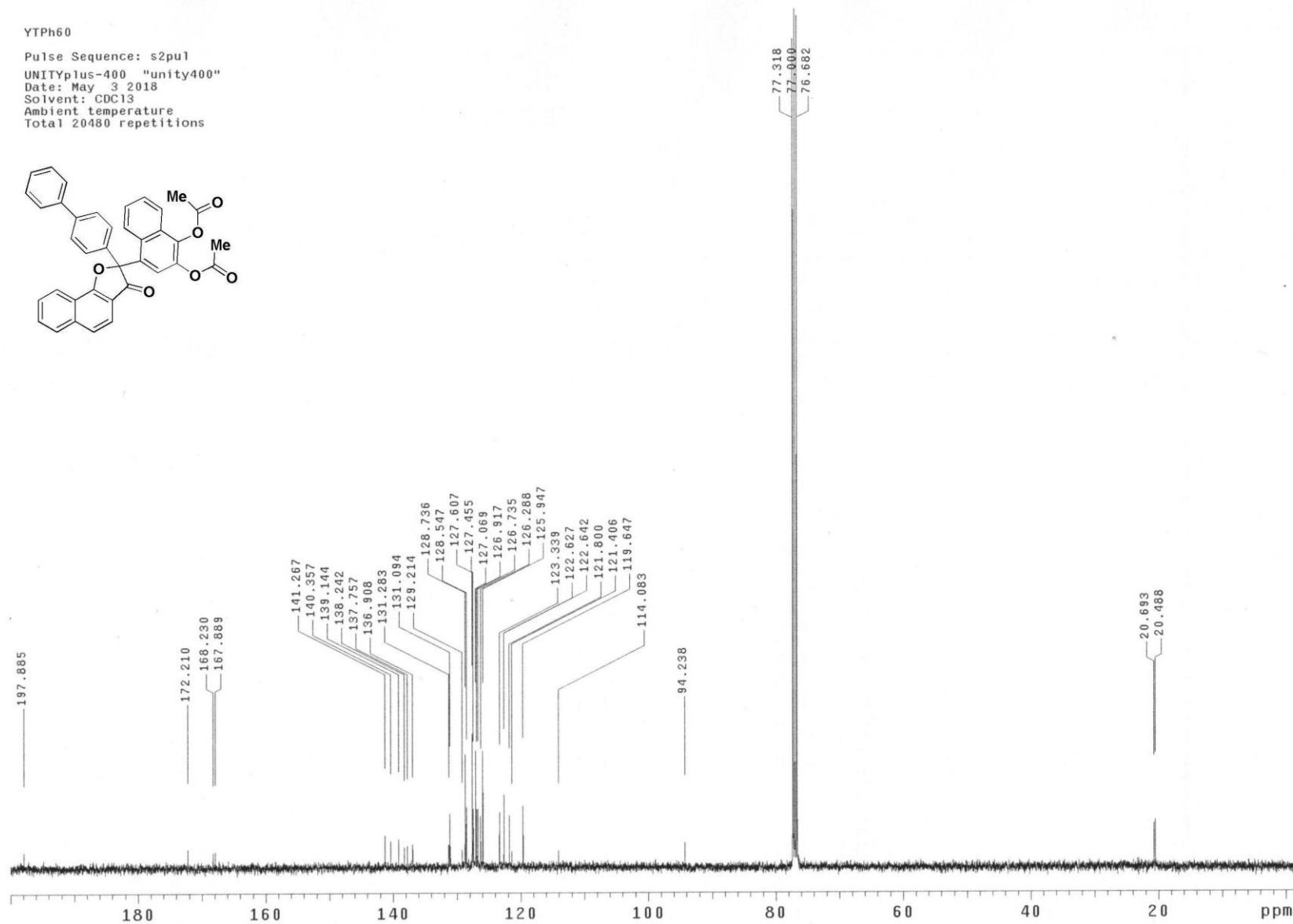
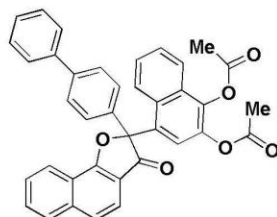
Compound 5e (¹H-NMR spectral data-2)



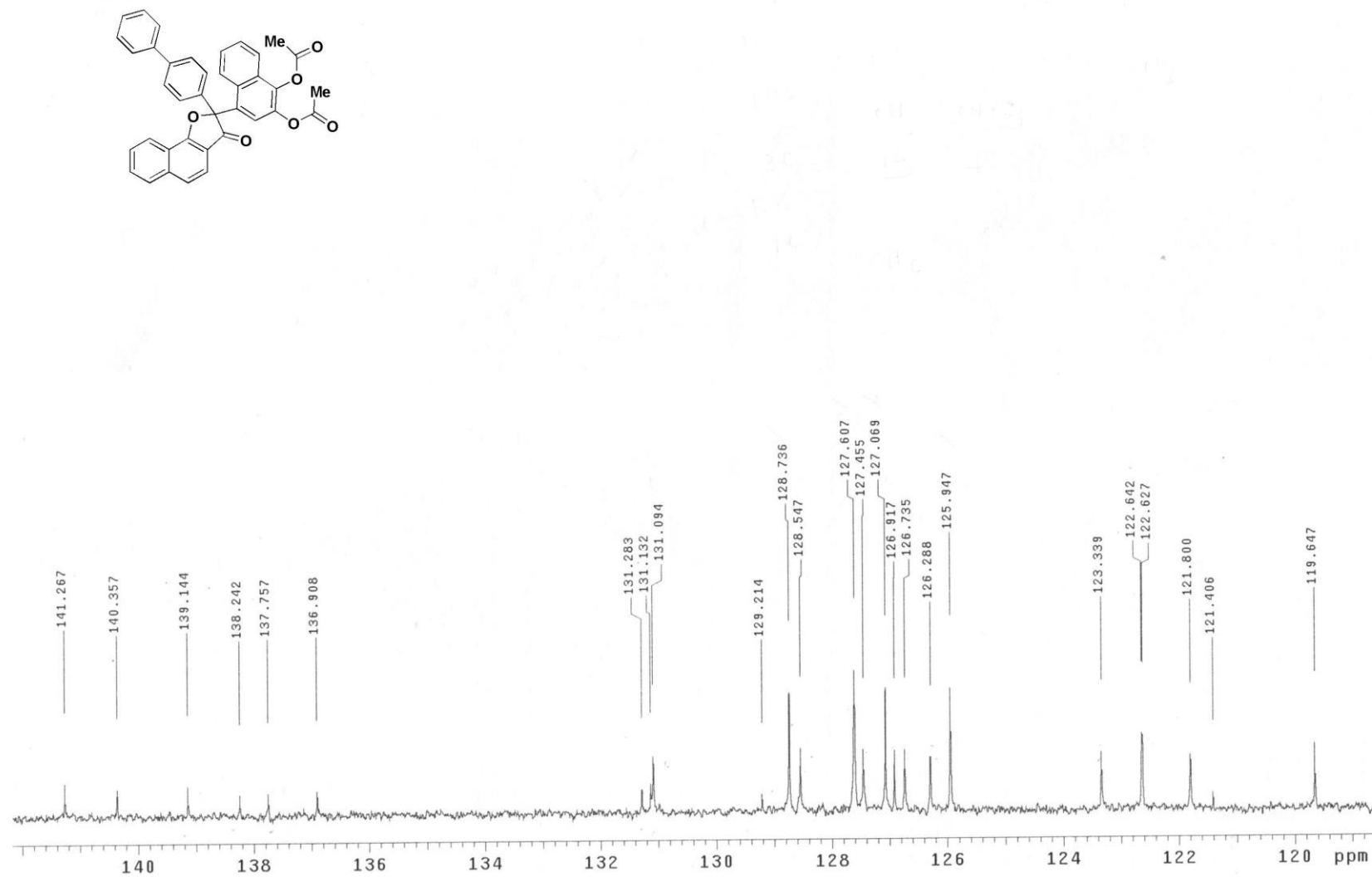
Compound 5e (¹³C-NMR spectral data-1)

YTPH60

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: May 3 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 20480 repetitions



Compound 5e (¹³C-NMR spectral data-2)



Compound 5f (¹H-NMR spectral data-1)

YT02AC20

Pulse Sequence: s2pu1

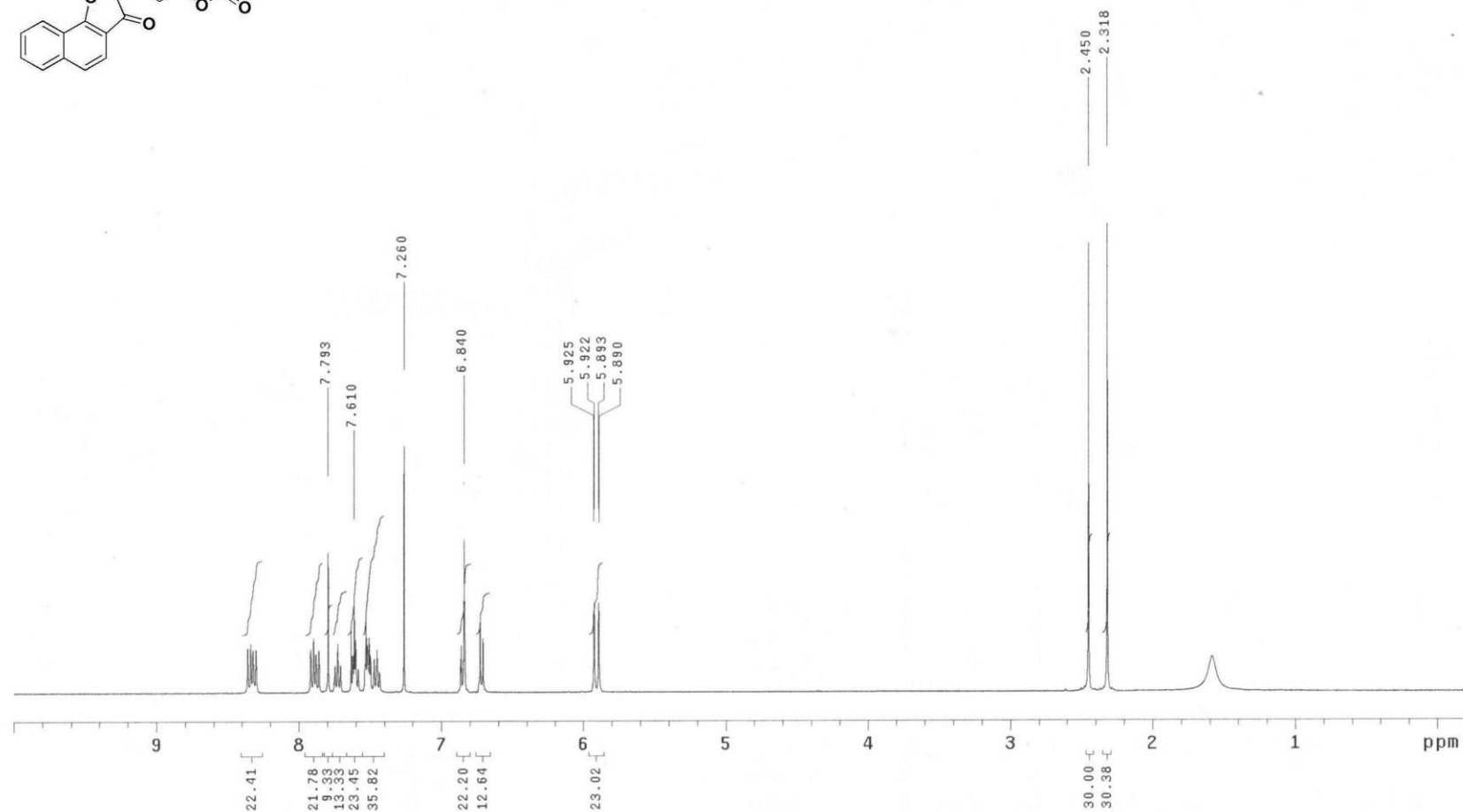
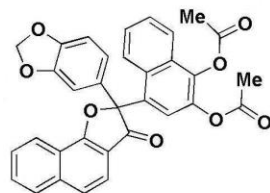
UNITYplus-400 "unity400"

Date: Jun 22 2018

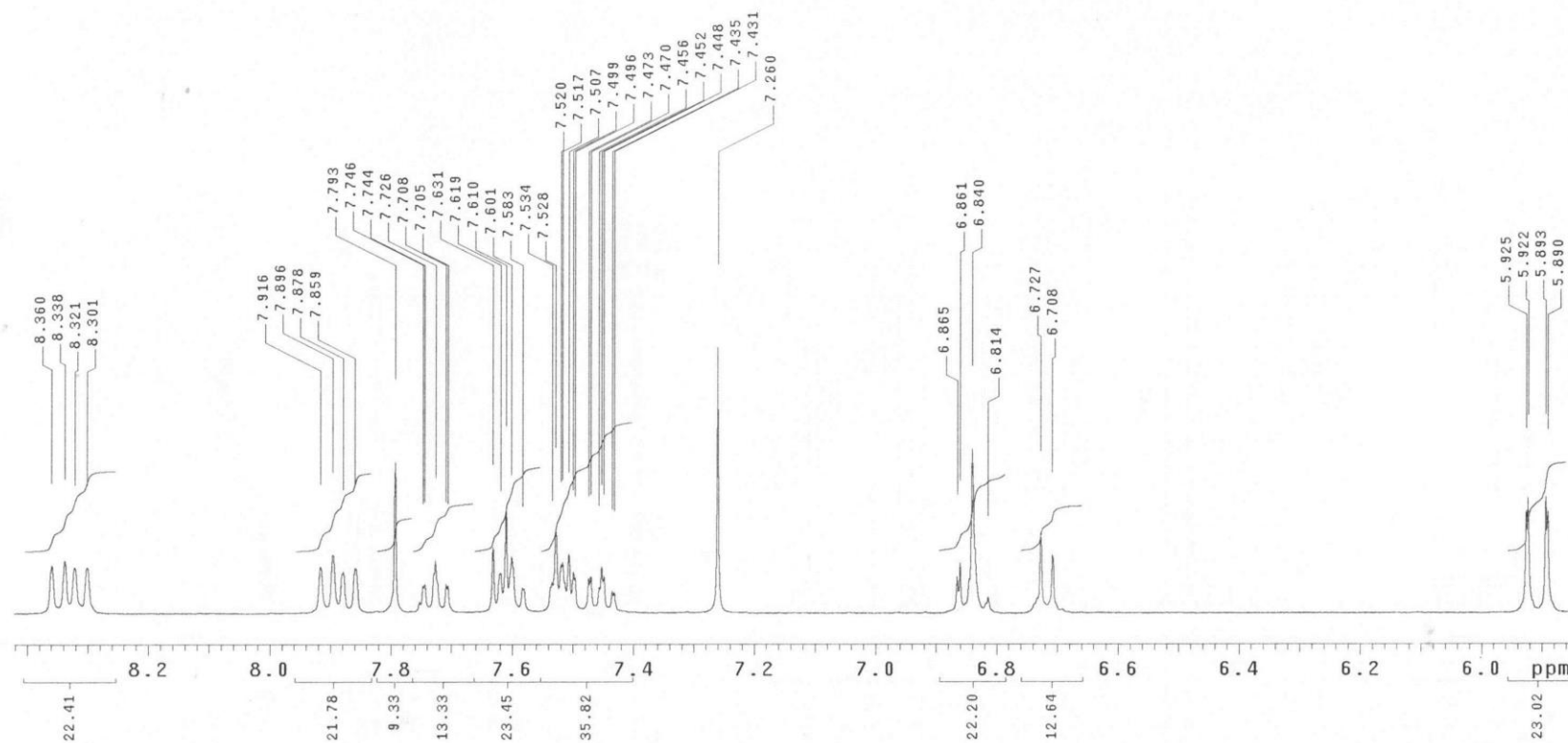
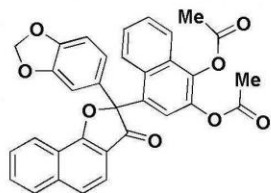
Solvent: CDCl₃

Ambient temperature

Total 64 repetitions



Compound 5f (¹H-NMR spectral data-2)



Compound 5f (¹³C-NMR spectral data)

YT02AC20

Pulse Sequence: s2pu1

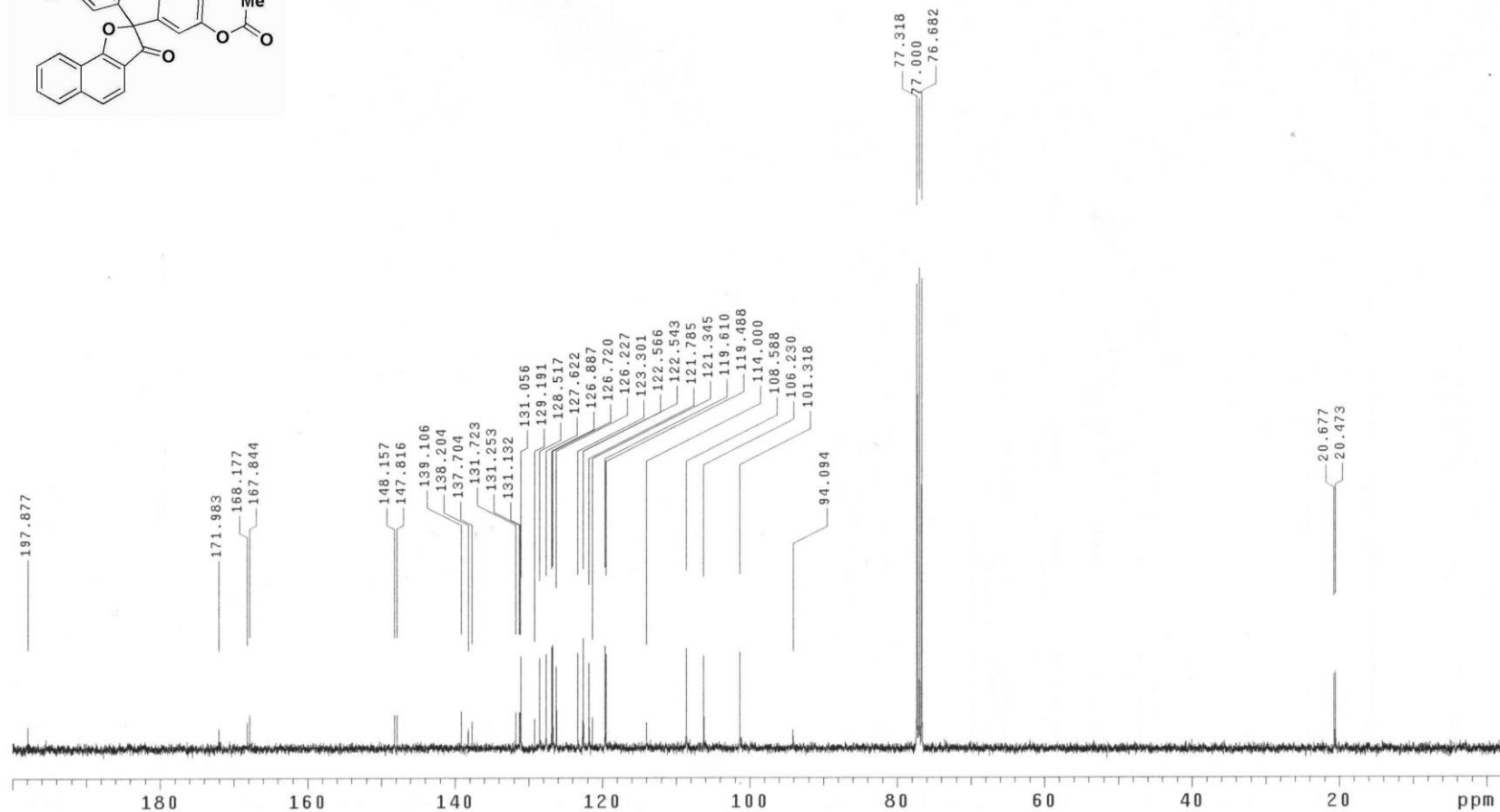
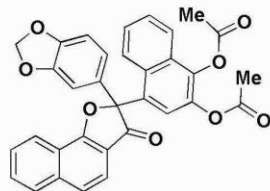
UNITYplus-400 "unity400"

Date: Jun 22 2018

Solvent: CDCl₃

Ambient temperature

Total 7824 repetitions



Compound 5g (¹H-NMR spectral data-1)

YTN1SP

Pulse Sequence: s2pu1

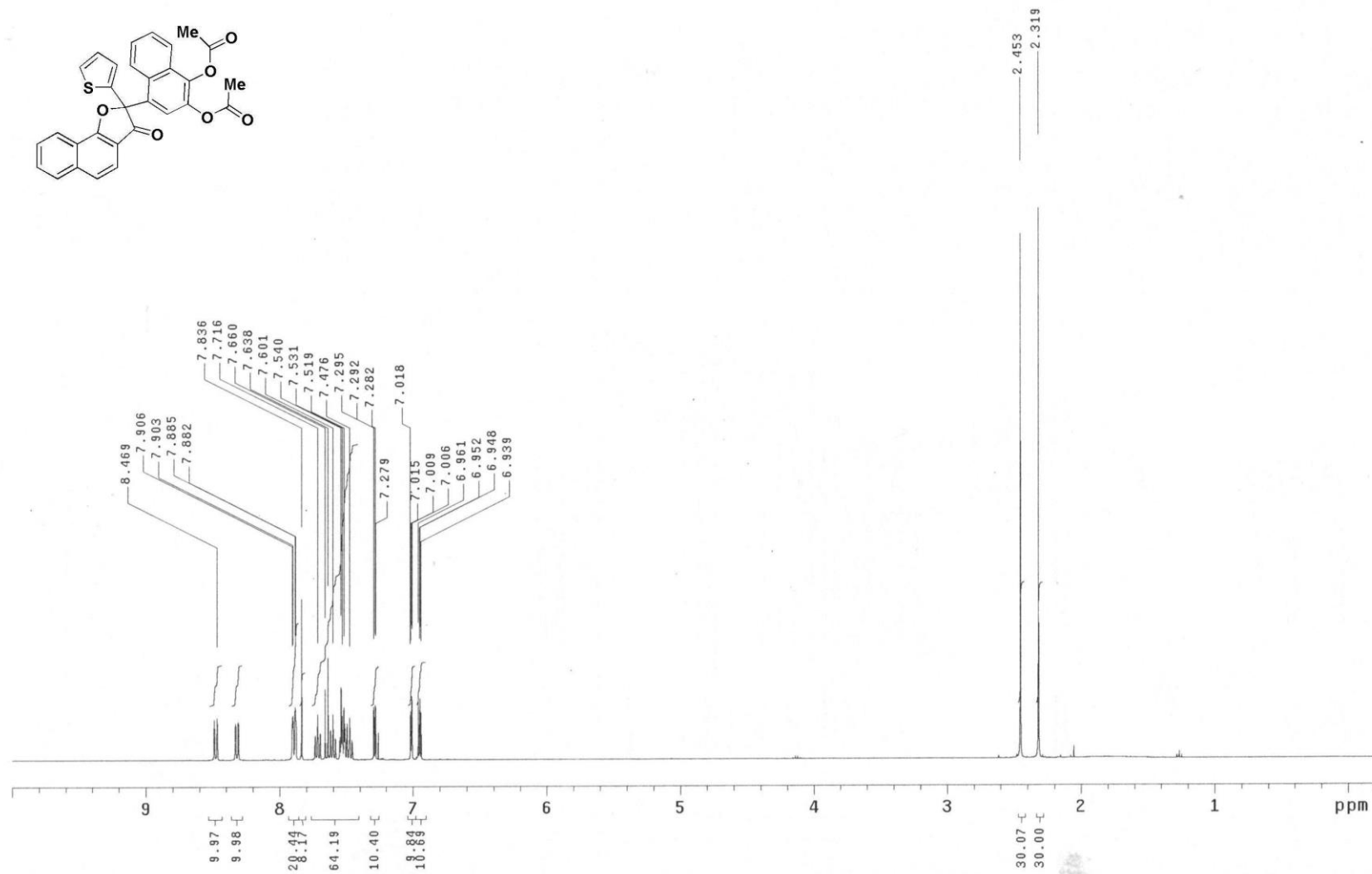
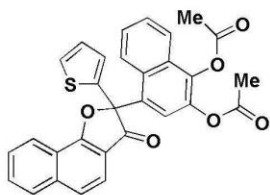
UNITYplus-400 "unity400"

Date: May 4 2018

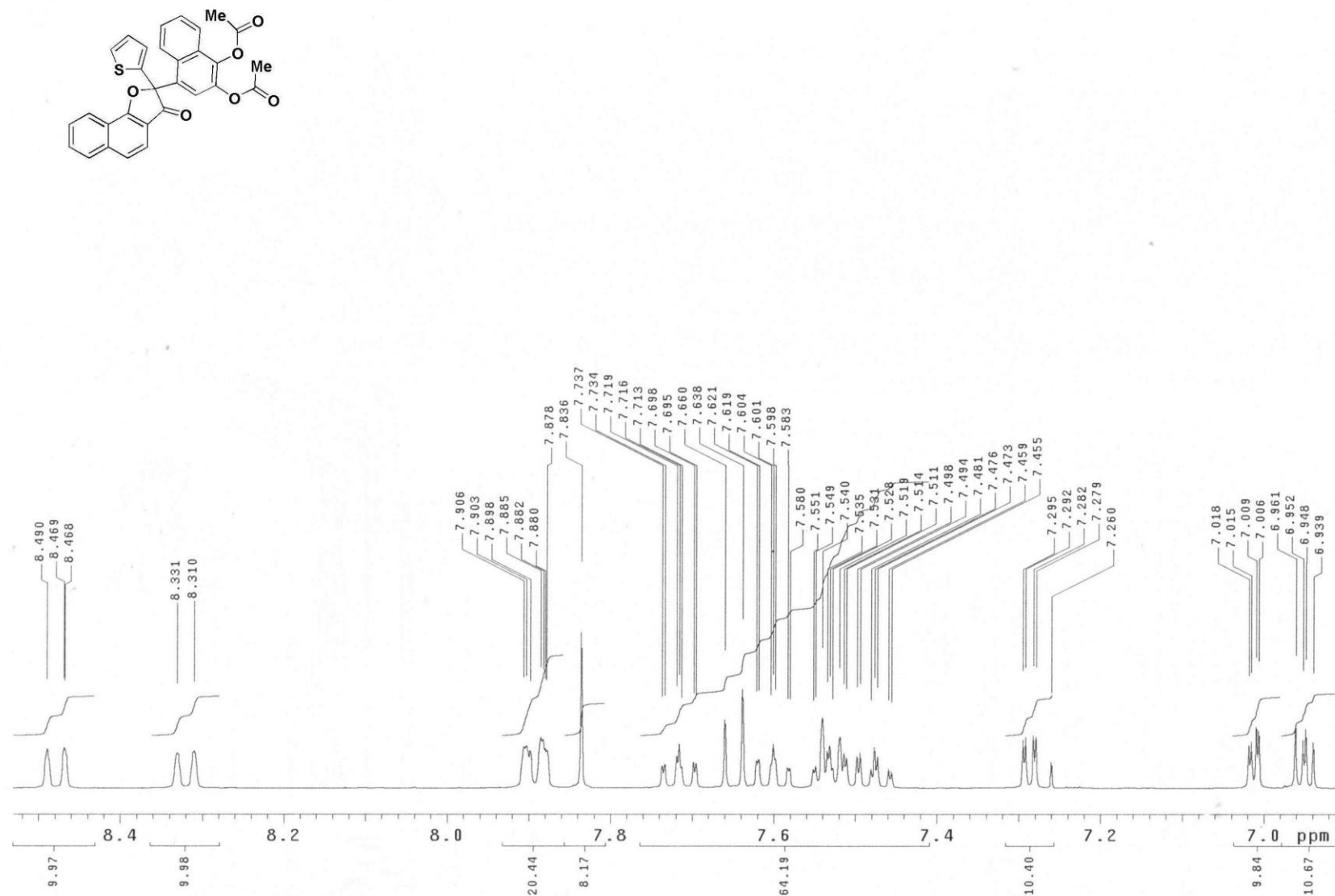
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 5g (¹H-NMR spectral data-2)



Compound 5g (¹³C-NMR spectral data)

YTN1SP

Pulse Sequence: s2pu1

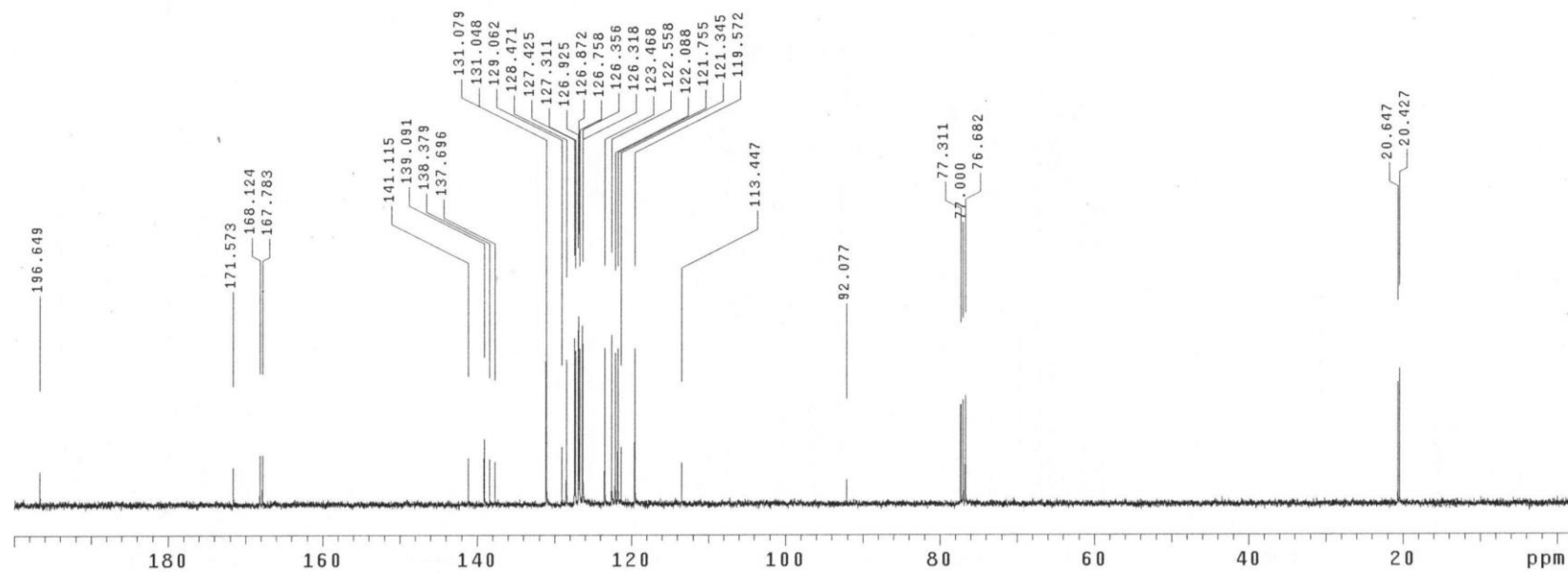
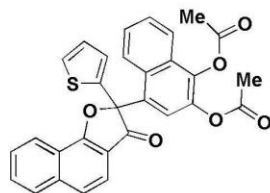
UNITYplus-400 "unity400"

Date: May 4 2018

Solvent: CDCl₃

Ambient temperature

Total 576 repetitions



Compound 5h (¹H-NMR spectral data-1)

YTB3435

Pulse Sequence: s2pu1

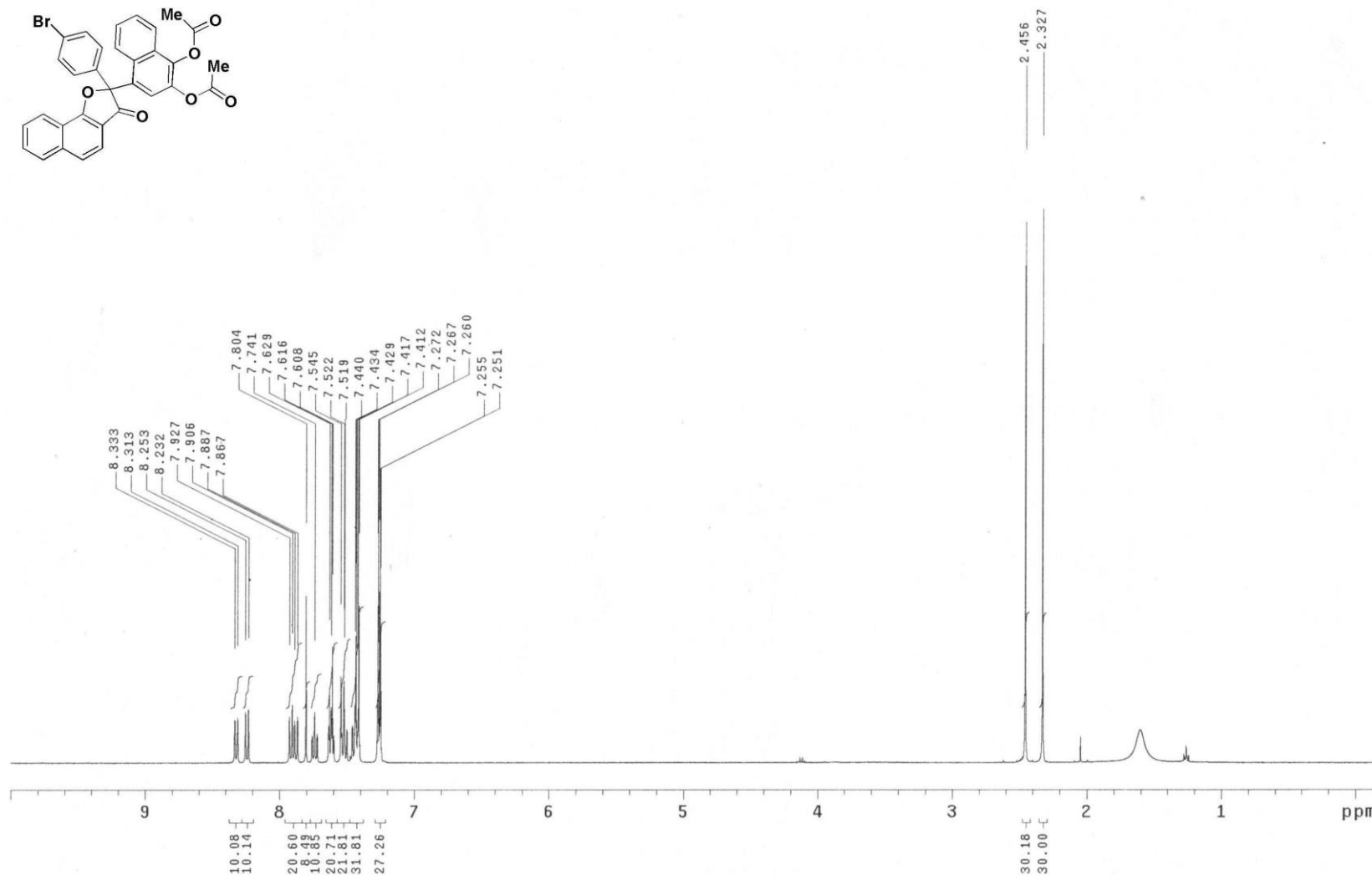
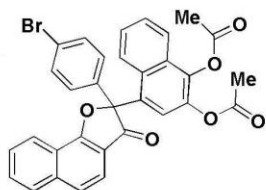
UNITYplus-400 "unity400"

Date: May 10 2018

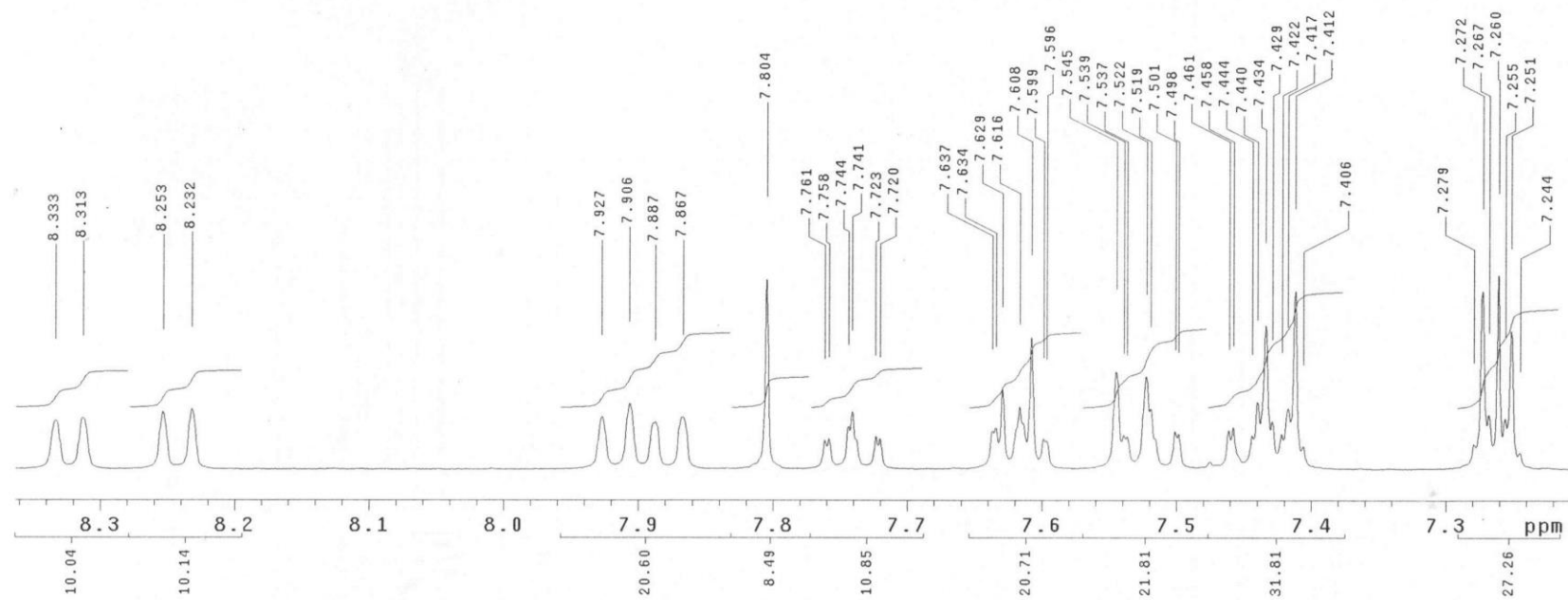
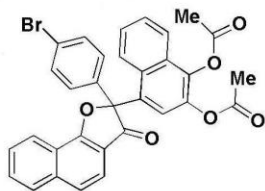
Solvent: CDCl₃

Ambient temperature

Total 64 repetitions



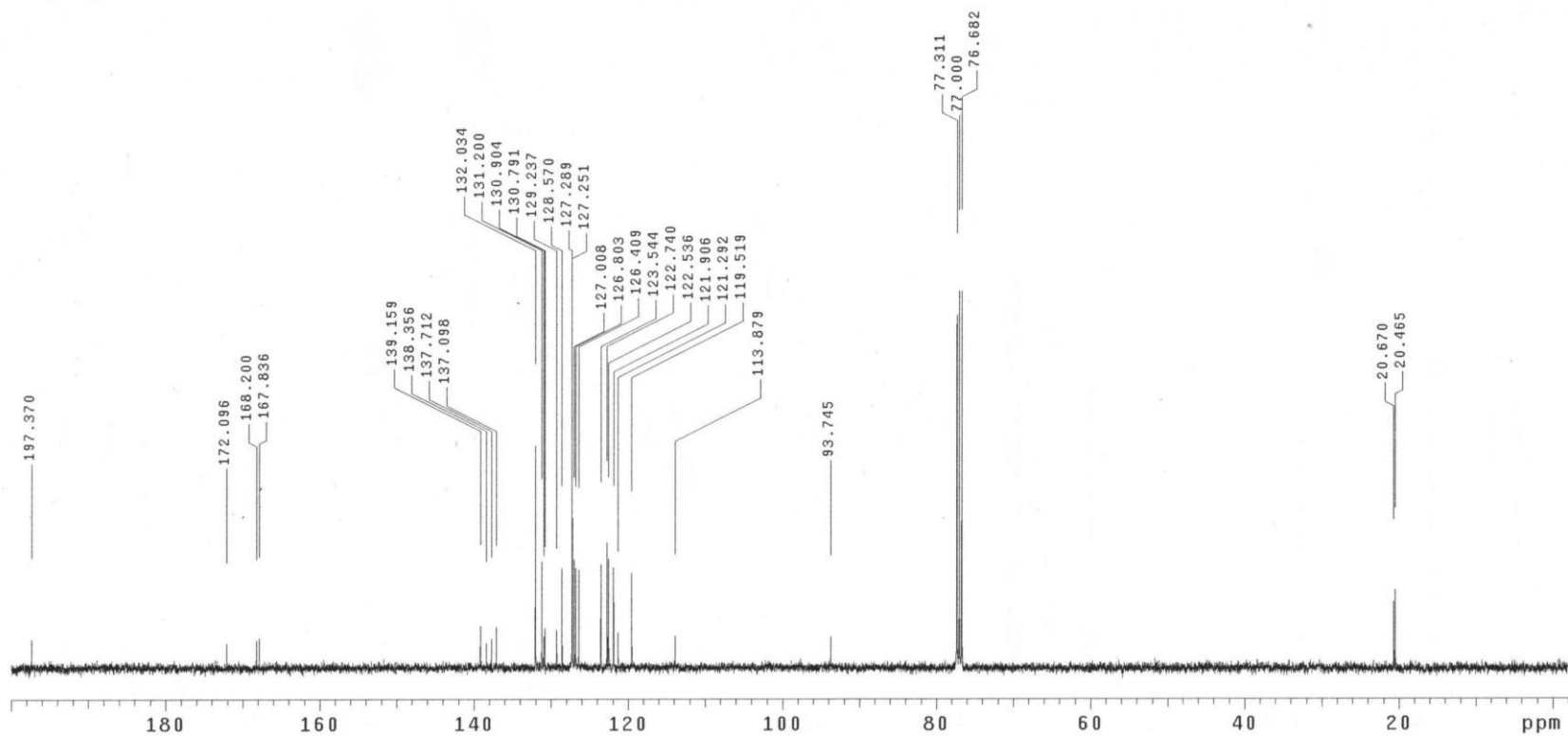
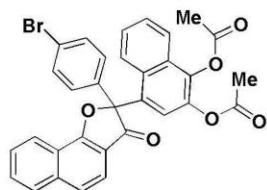
Compound 5h (¹H-NMR spectral data-2)



Compound 5h (¹³C-NMR spectral data)

YTB3435

Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: May 10 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 4592 repetitions



Compound 5i (¹H-NMR spectral data-1)

YT2FAC20

Pulse Sequence: s2pu1

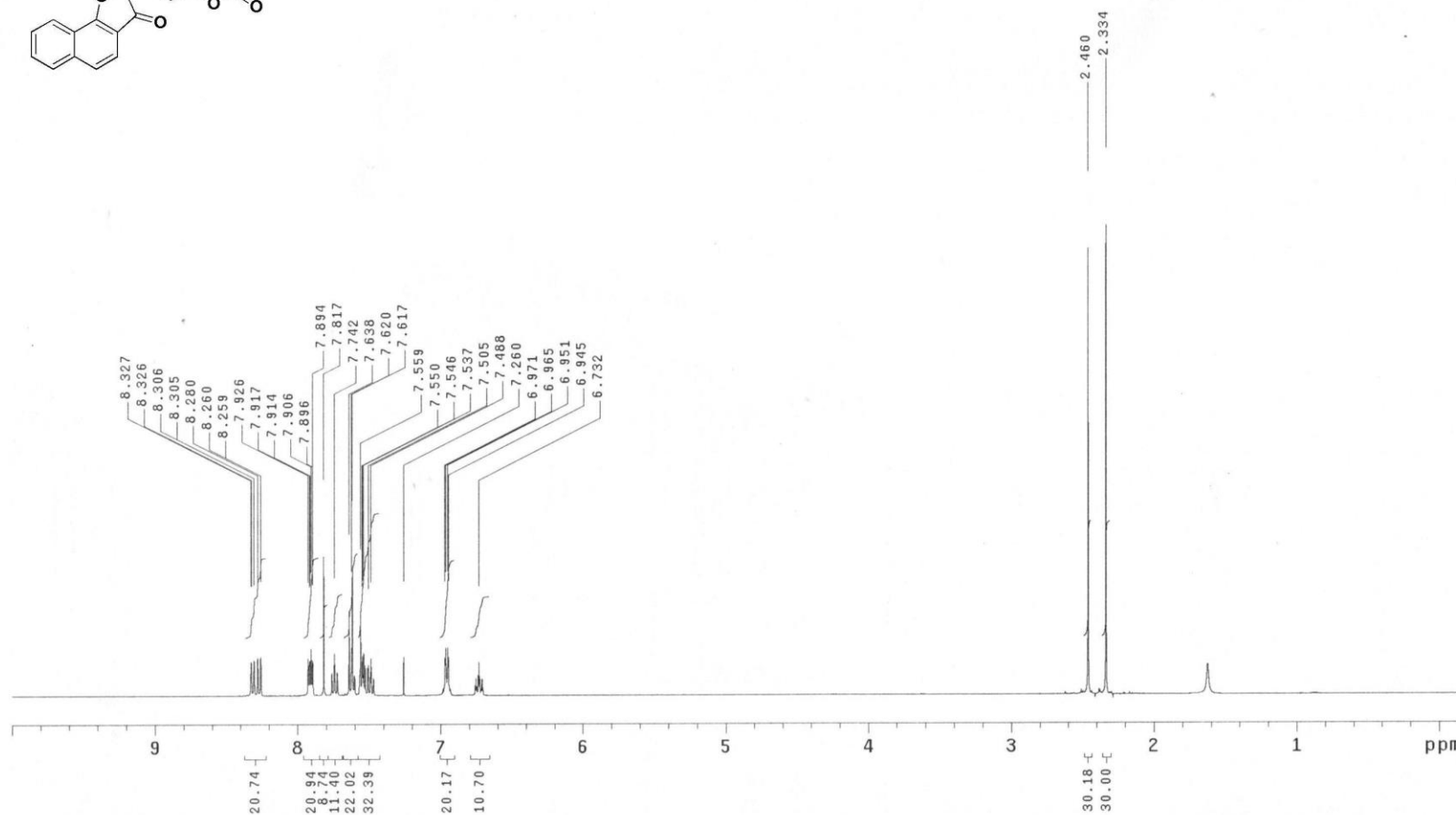
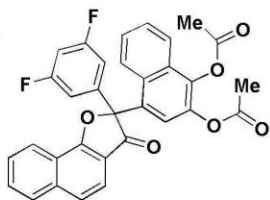
UNITYplus-400 "unity400"

Date: May 16 2018

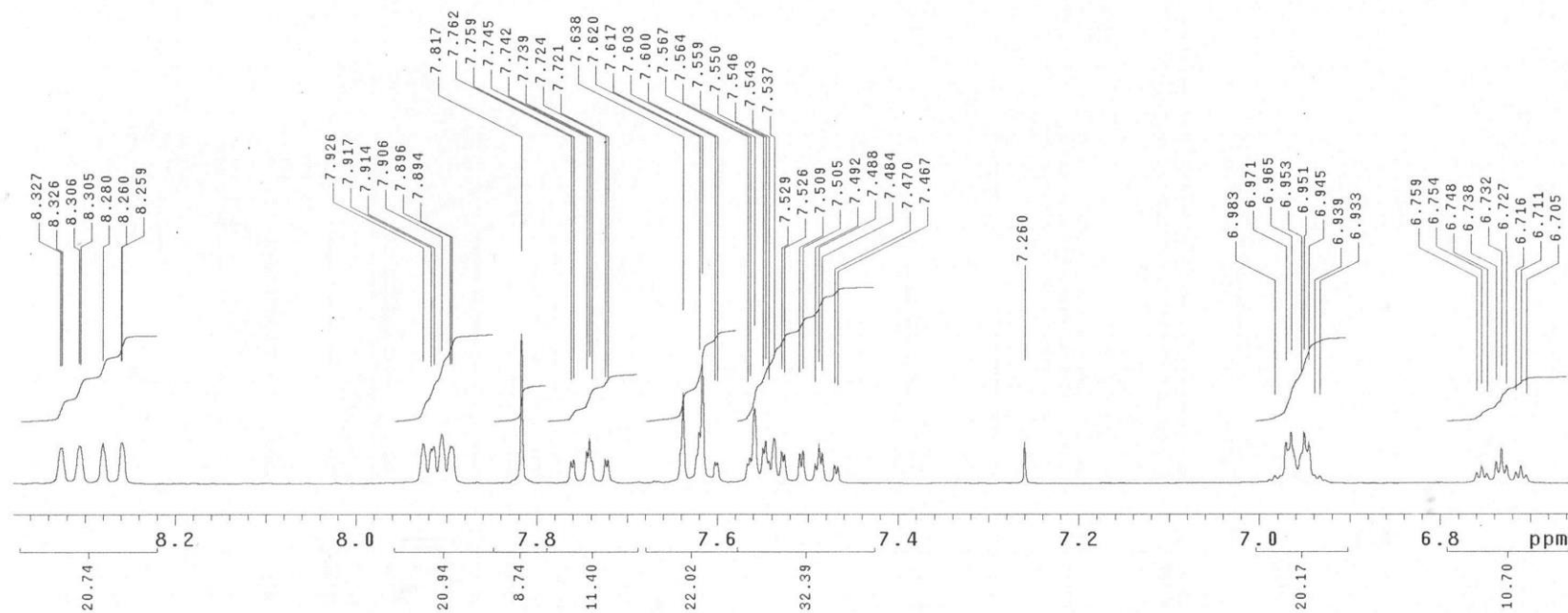
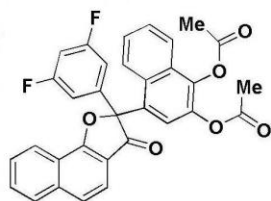
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



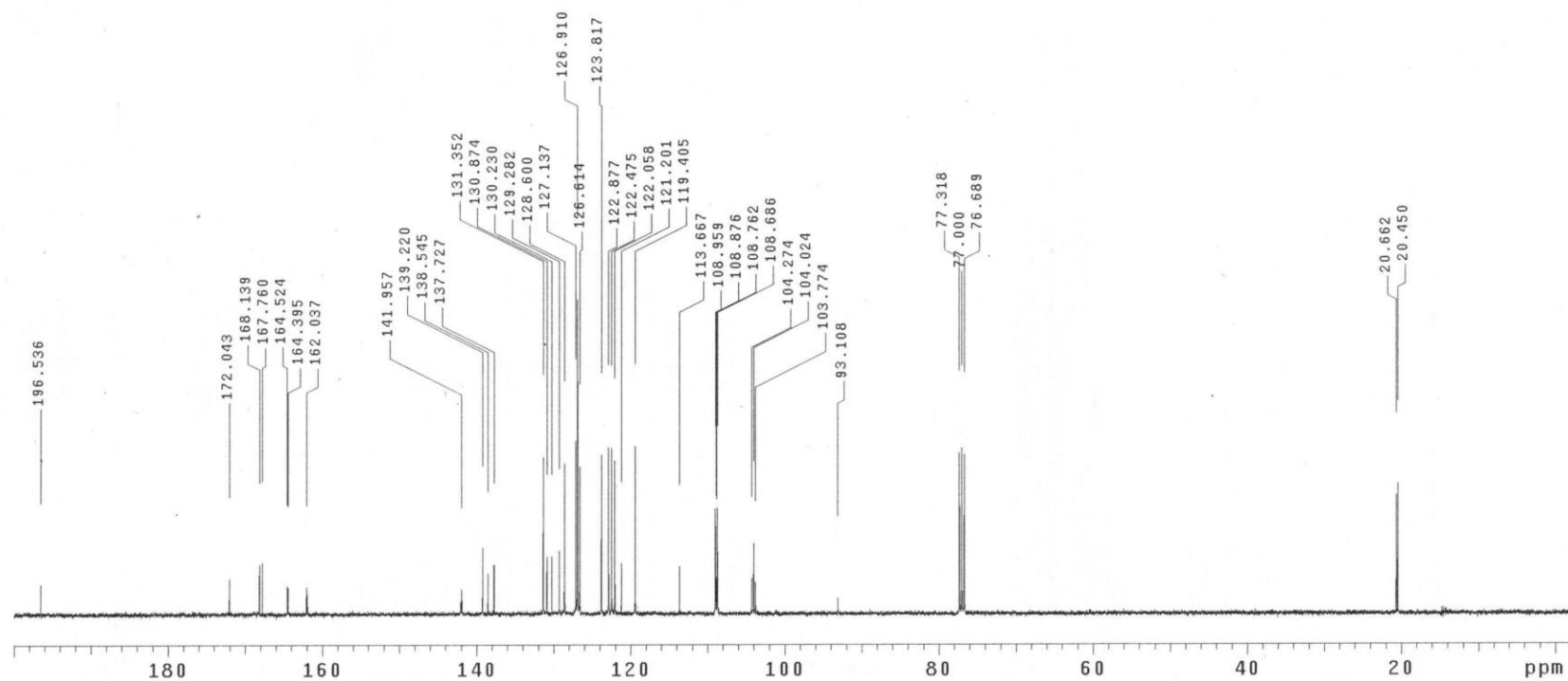
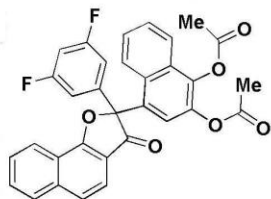
Compound 5i (¹H-NMR spectral data-2)



Compound 5i (¹³C-NMR spectral data)

YT2FAC20

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: May 16 2018
Solvent: CDCl₃
Ambient temperature
Total 3680 repetitions



Compound 5j (¹H-NMR spectral data-1)

YTCF3AC20

Pulse Sequence: s2pu1

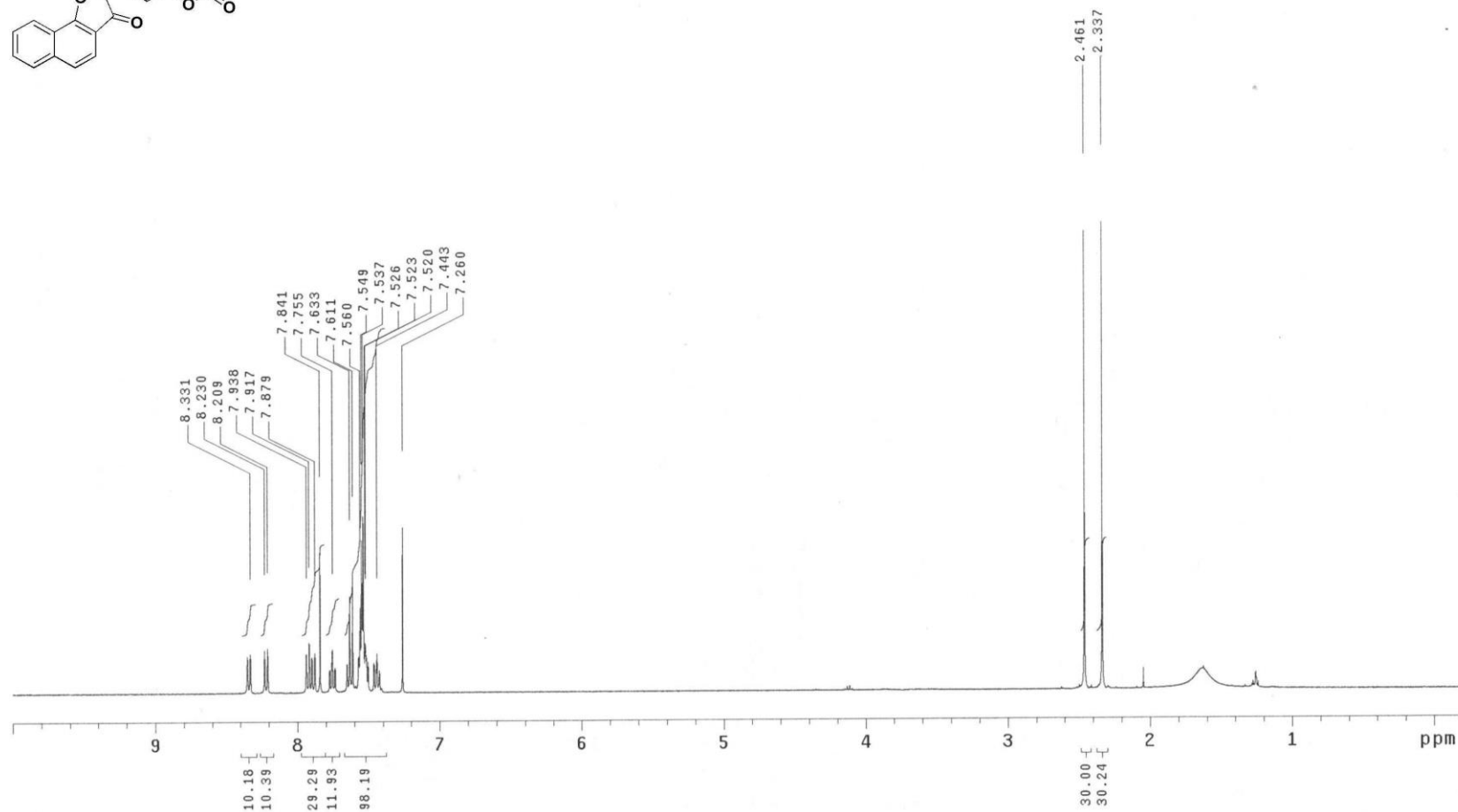
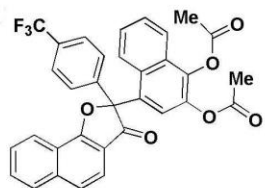
UNITYplus-400 "unity400"

Date: Jun 7 2018

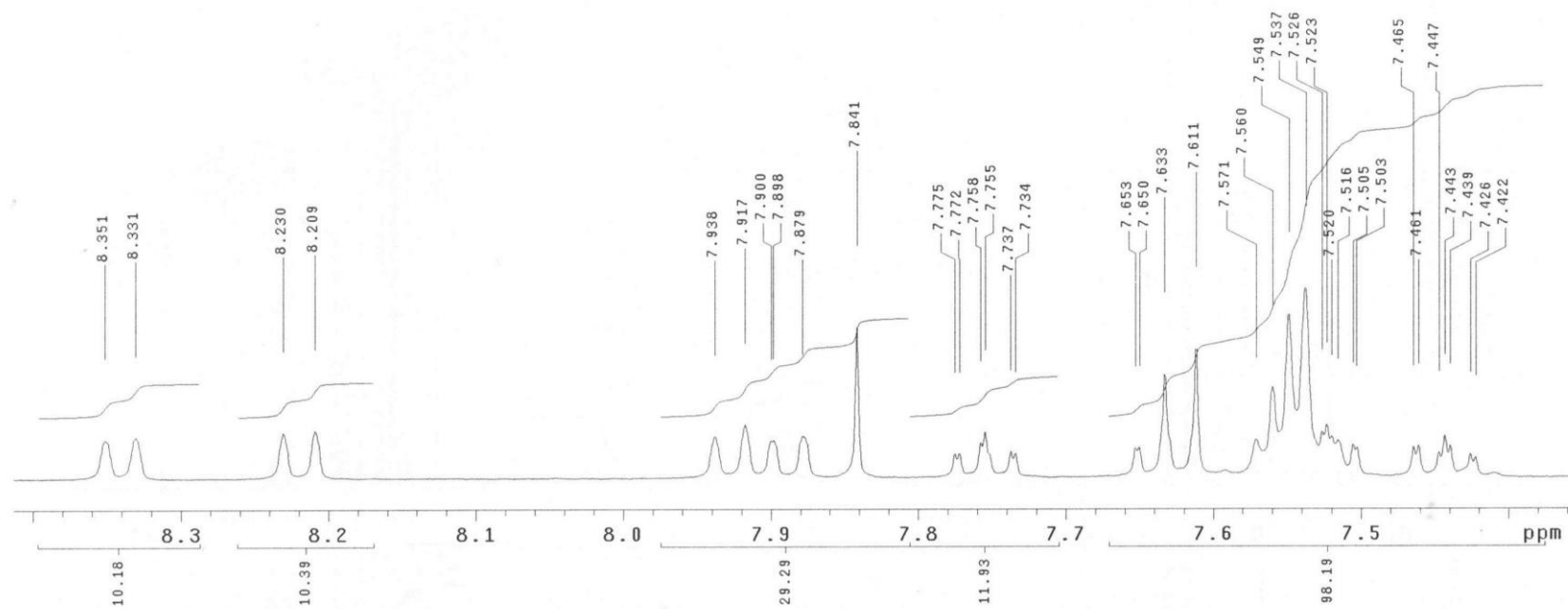
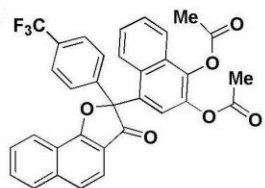
Solvent: CDC13

Ambient temperature

Total 32 repetitions



Compound 5j (¹H-NMR spectral data-2)



Compound 5j (¹³C-NMR spectral data)

YTCF3AC20

Pulse Sequence: s2pu1

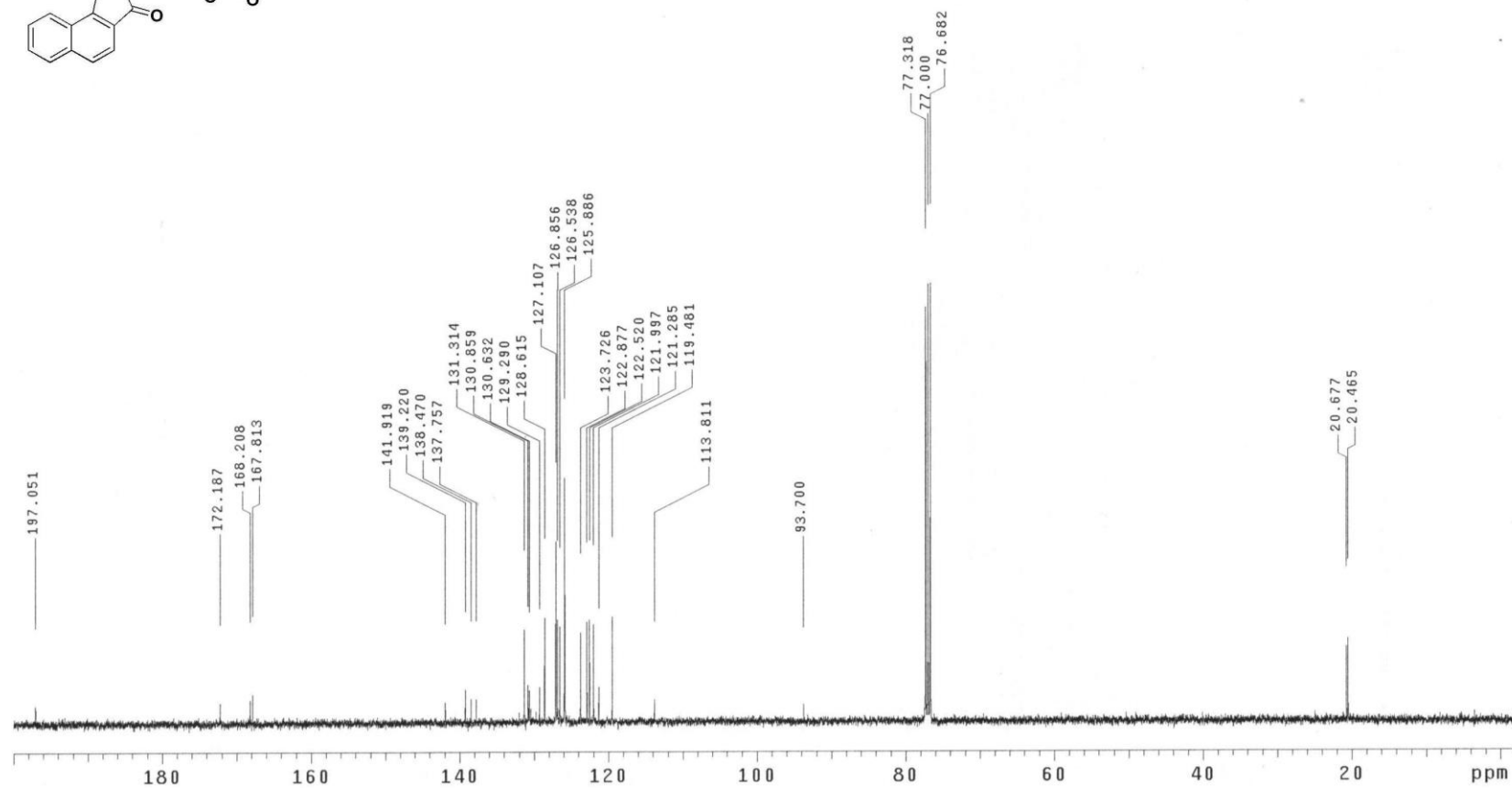
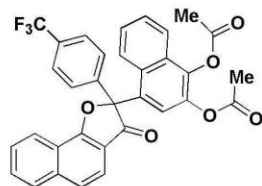
UNITYplus-400 "unity400"

Date: Jun 7 2018

Solvent: CDCl₃

Ambient temperature

Total 7568 repetitions



Compound 5k (¹H-NMR spectral data-1)

YTNAN

Pulse Sequence: s2pu1

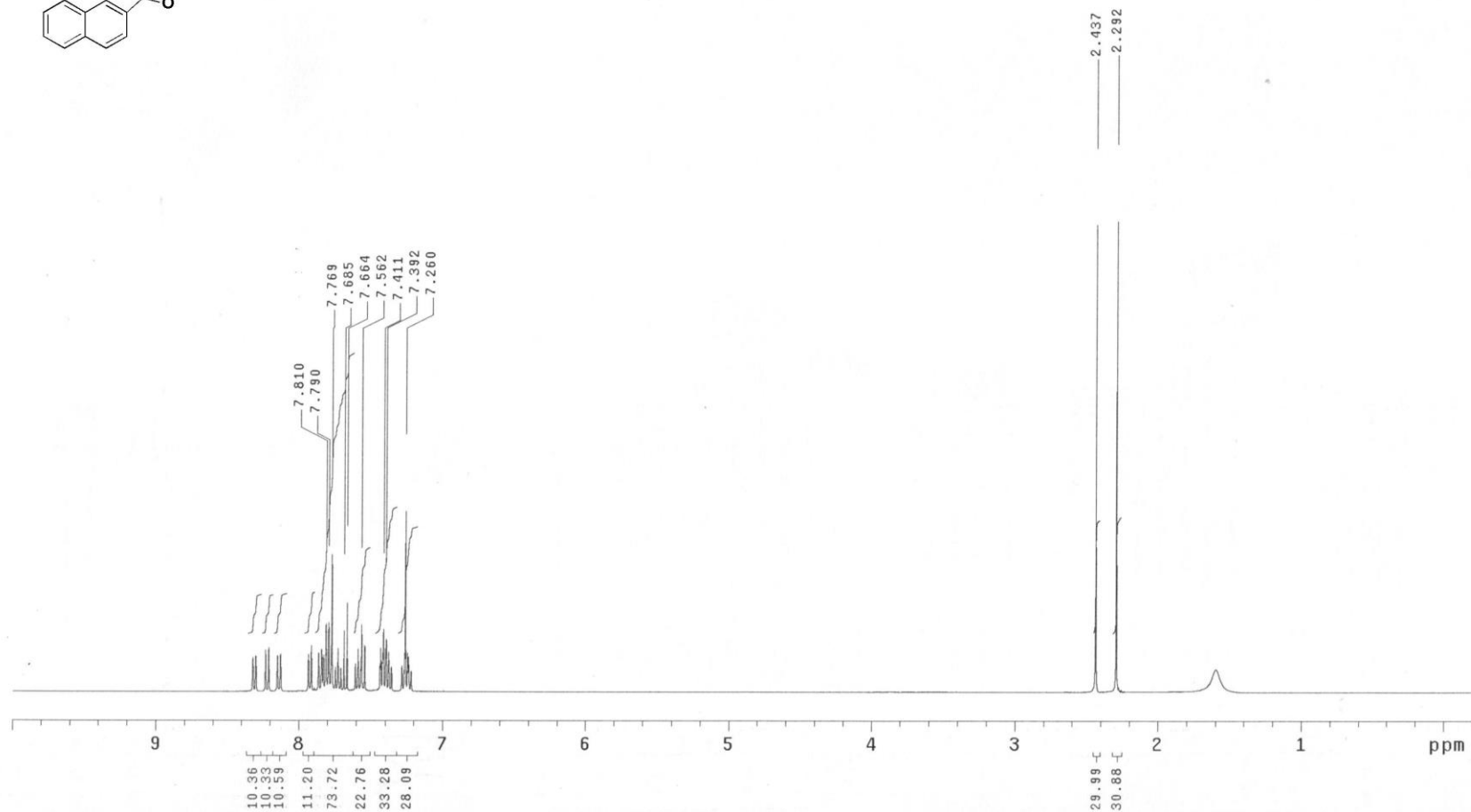
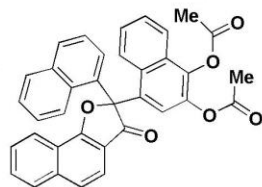
UNITYplus-400 "unity400"

Date: Jun 27 2019

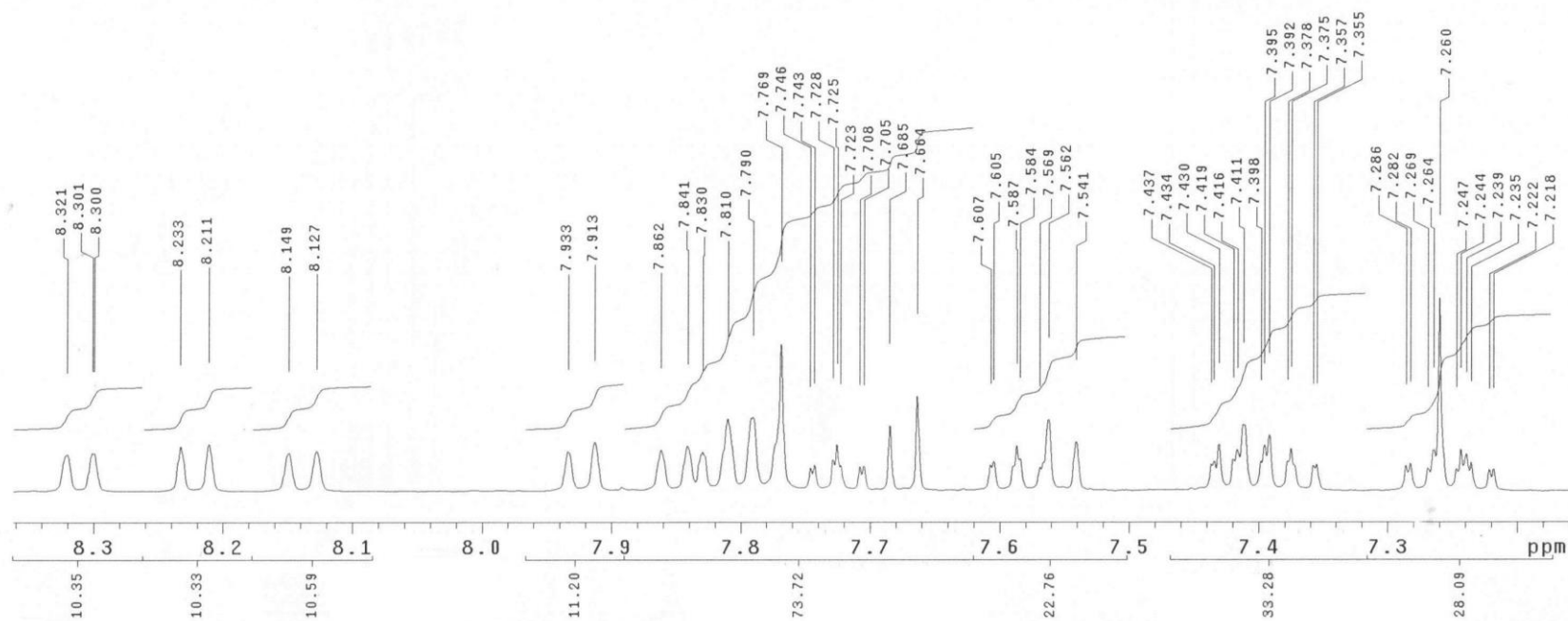
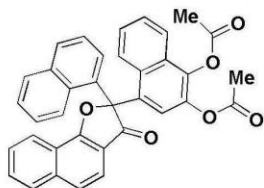
Solvent: CDCl₃

Ambient temperature

Total 96 repetitions



Compound 5k (¹H-NMR spectral data-2)



Compound 5k (¹³C-NMR spectral data-1)

YTNAAc02

Pulse Sequence: s2pu1

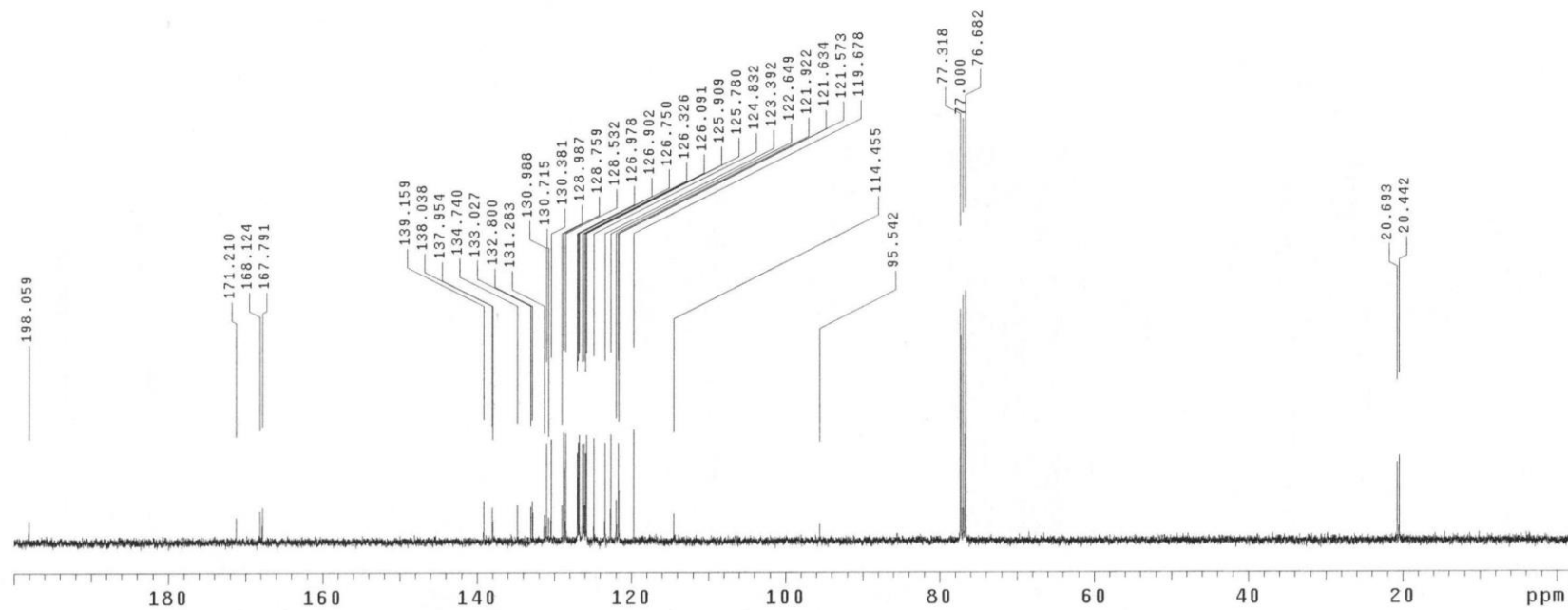
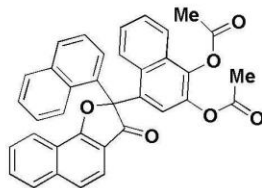
UNITYplus-400 "unity400"

Date: Jun 1 2018

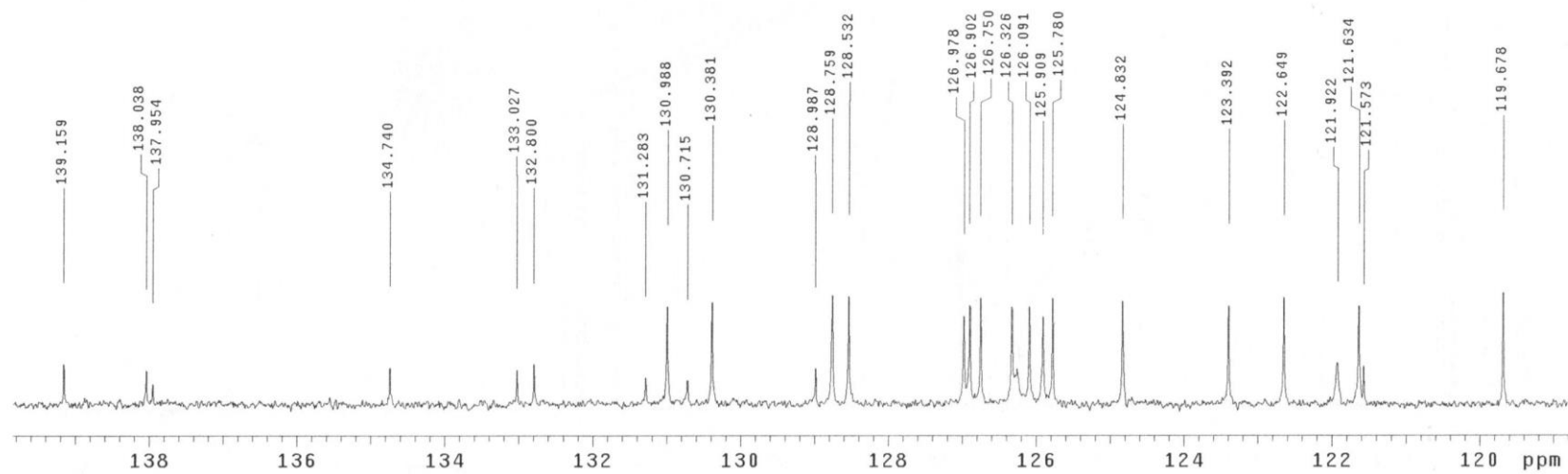
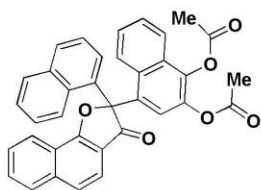
Solvent: CDCl₃

Ambient temperature

Total 2160 repetitions



Compound 5k (^{13}C -NMR spectral data-2)



Compound 5l (¹H-NMR spectral data)

KTCF30AC

Pulse Sequence: s2pu1

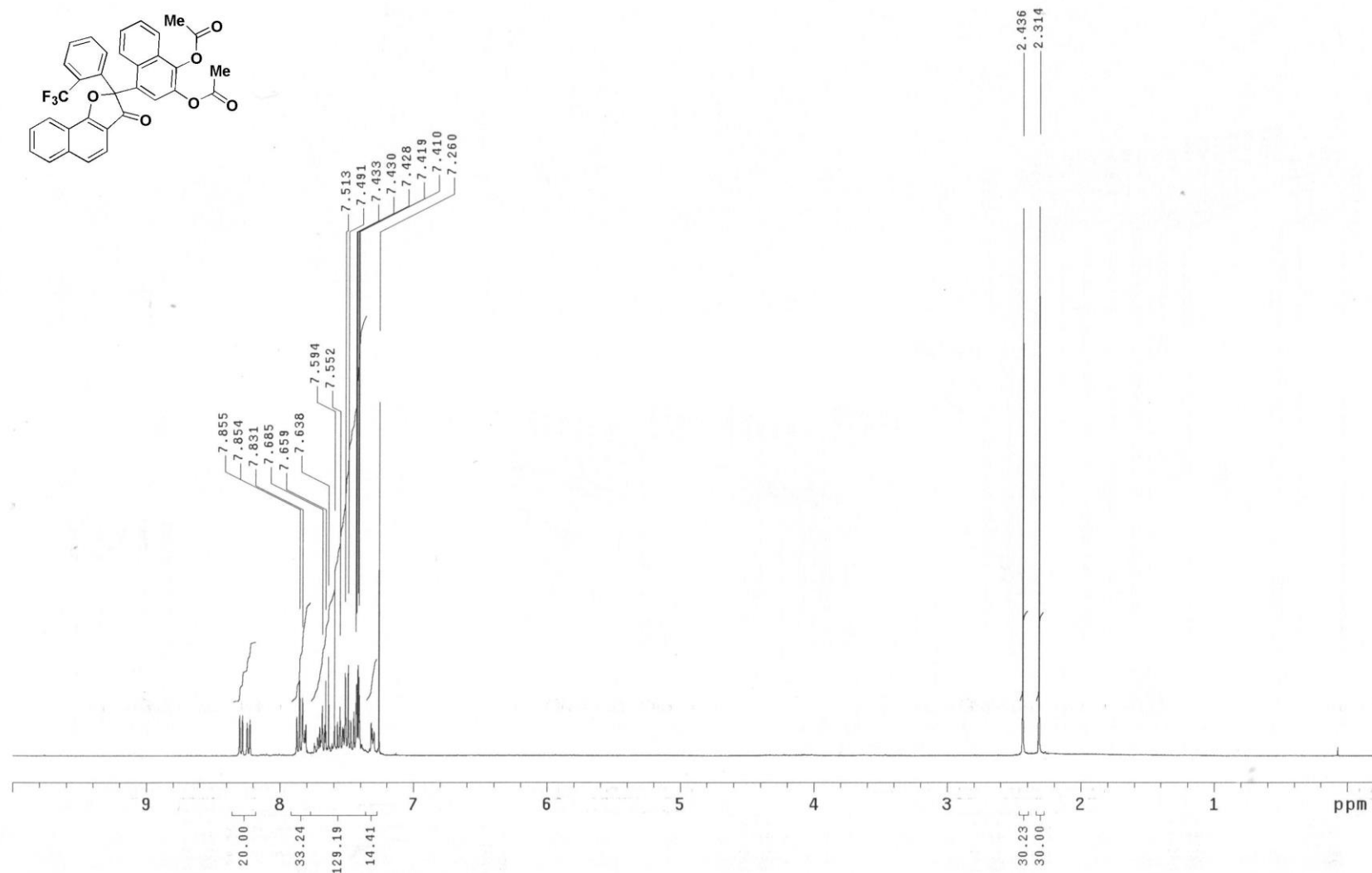
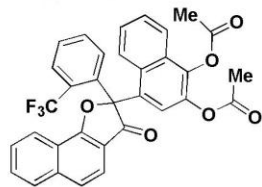
UNITYplus-400 "unity400"

Date: Dec 3 2019

Solvent: CDCl₃

Ambient temperature

Total 80 repetitions



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

KTCF30AC

Pulse Sequence: s2pu1

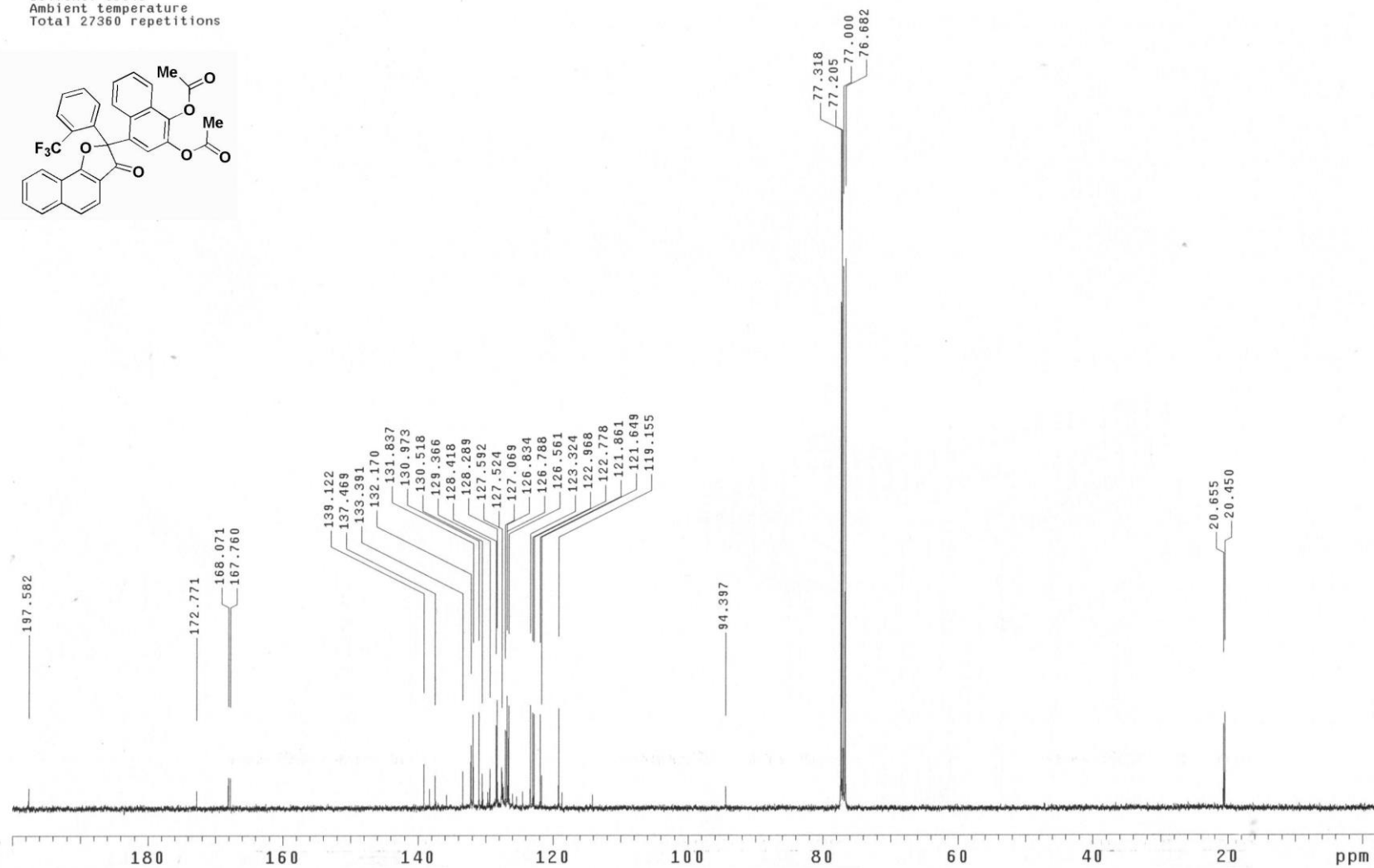
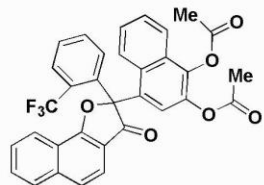
UNITYplus-400 "unity400"

Date: Dec 3 2019

Solvent: CDCl_3

Ambient temperature

Total 27360 repetitions



Compound 6m (¹H-NMR spectral data-1)

YT2Me0Ac20

Pulse Sequence: s2pu1

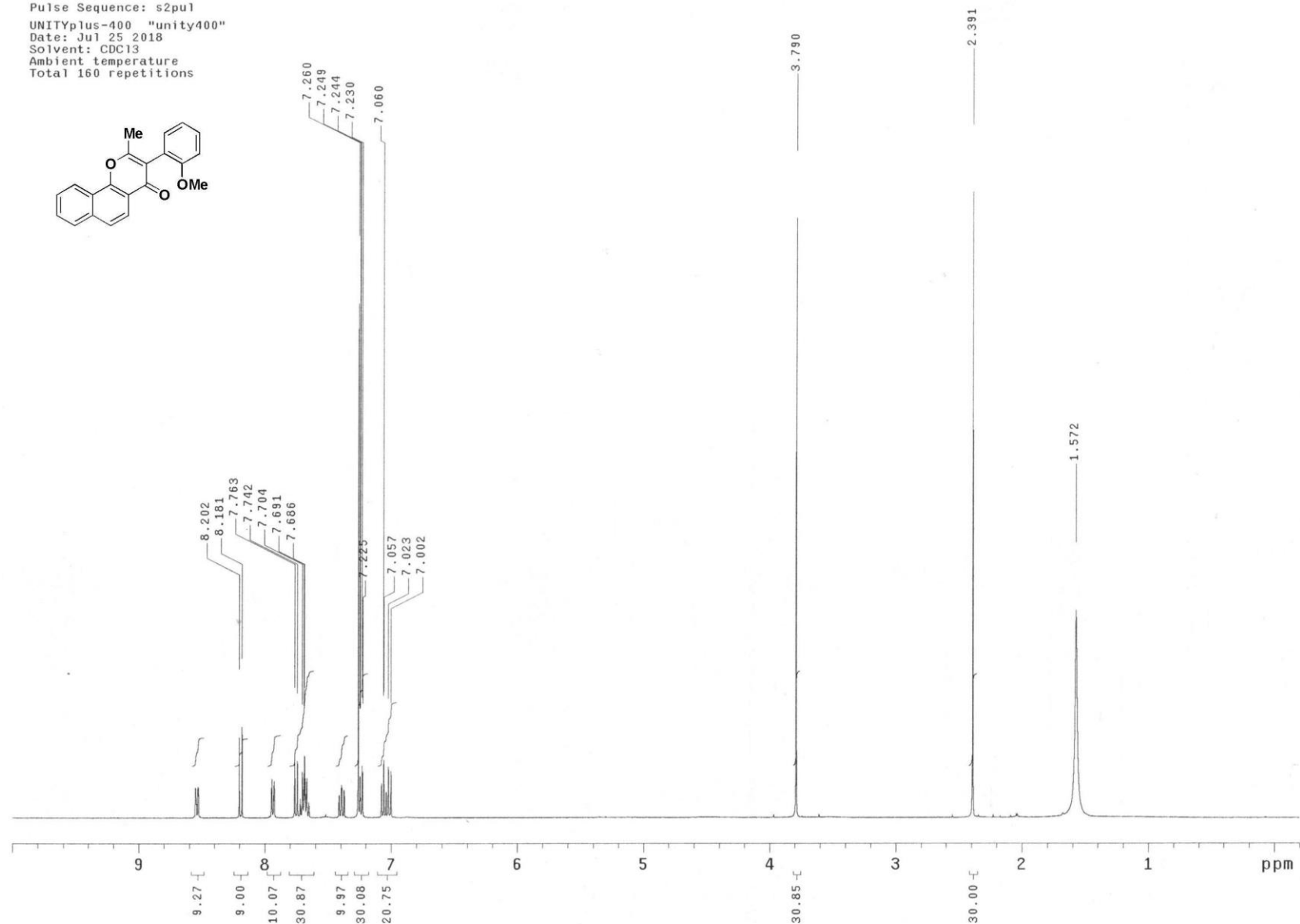
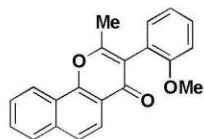
UNITYplus-400 "unity400"

Date: Jul 25 2018

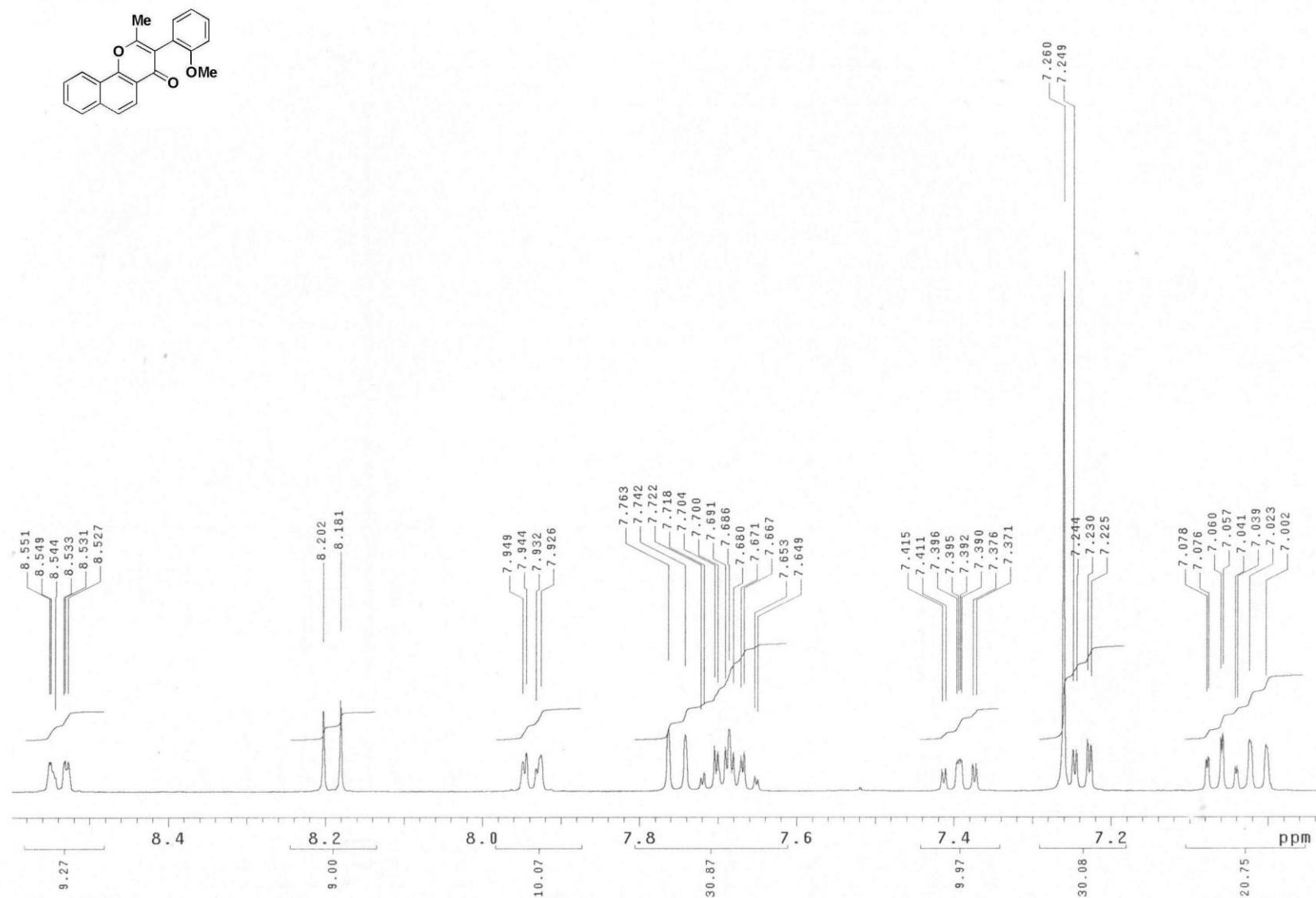
Solvent: CDCl₃

Ambient temperature

Total 160 repetitions



Compound 6m (¹H-NMR spectral data-2)



Compound 6m (¹³C-NMR spectral data-1)

YT2Me0Ac20

Pulse Sequence: s2pu1

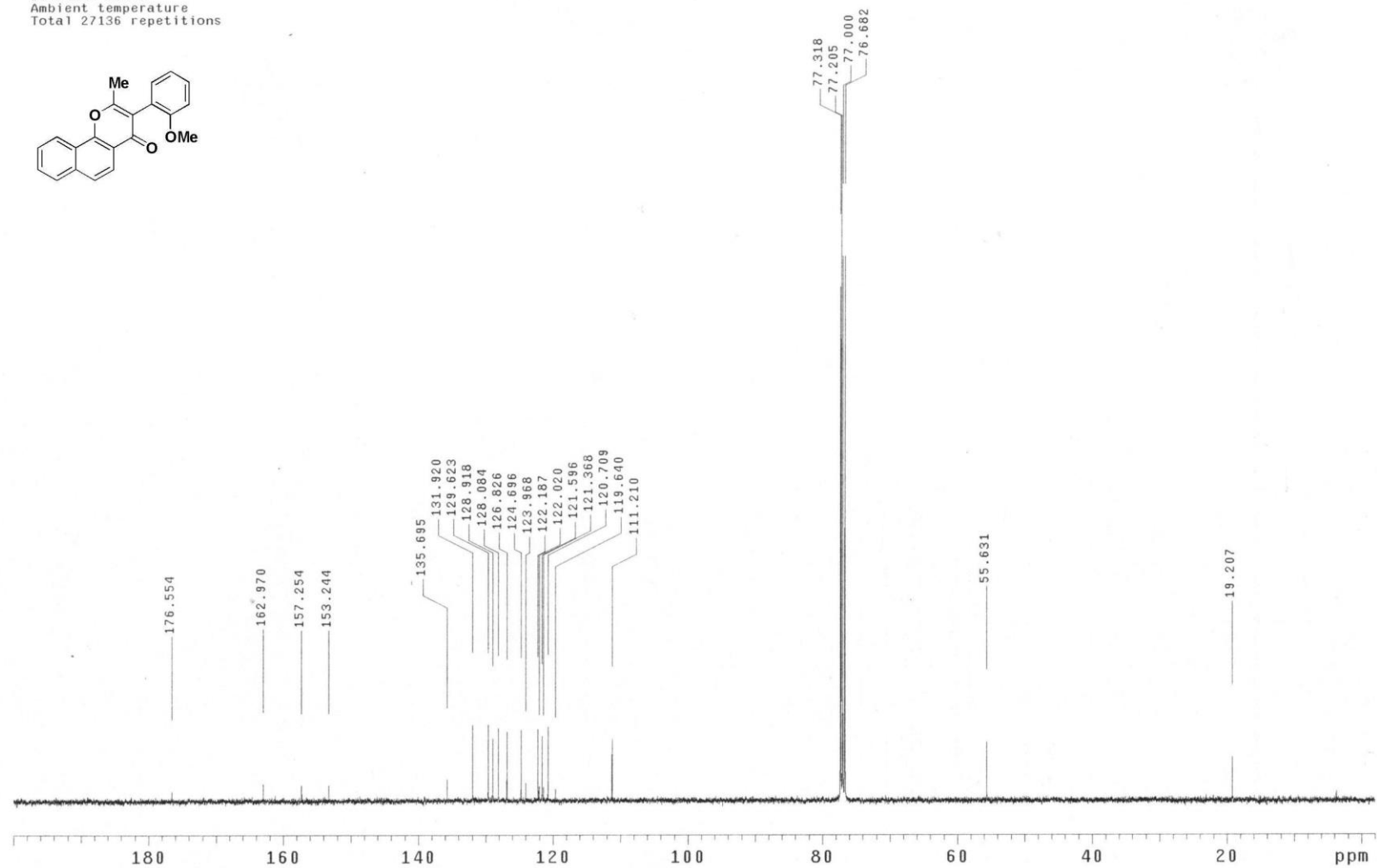
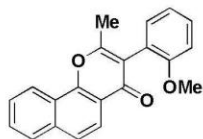
UNITYplus-400 "unity400"

Date: Jul 25 2018

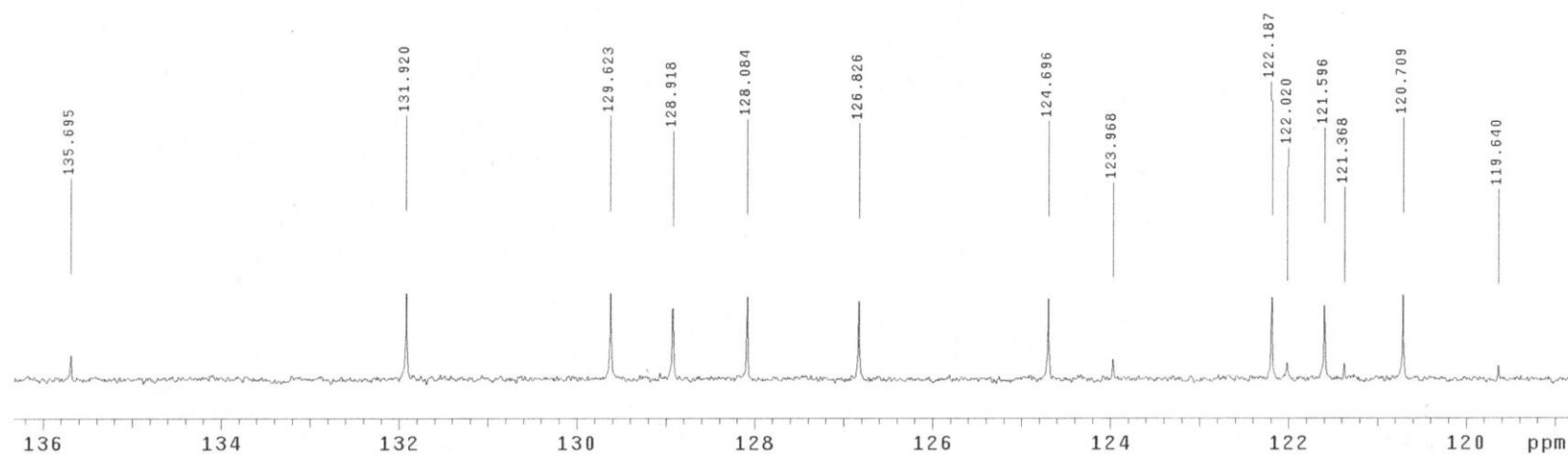
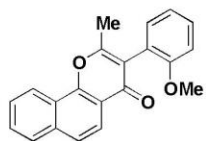
Solvent: CDCl₃

Ambient temperature

Total 27136 repetitions



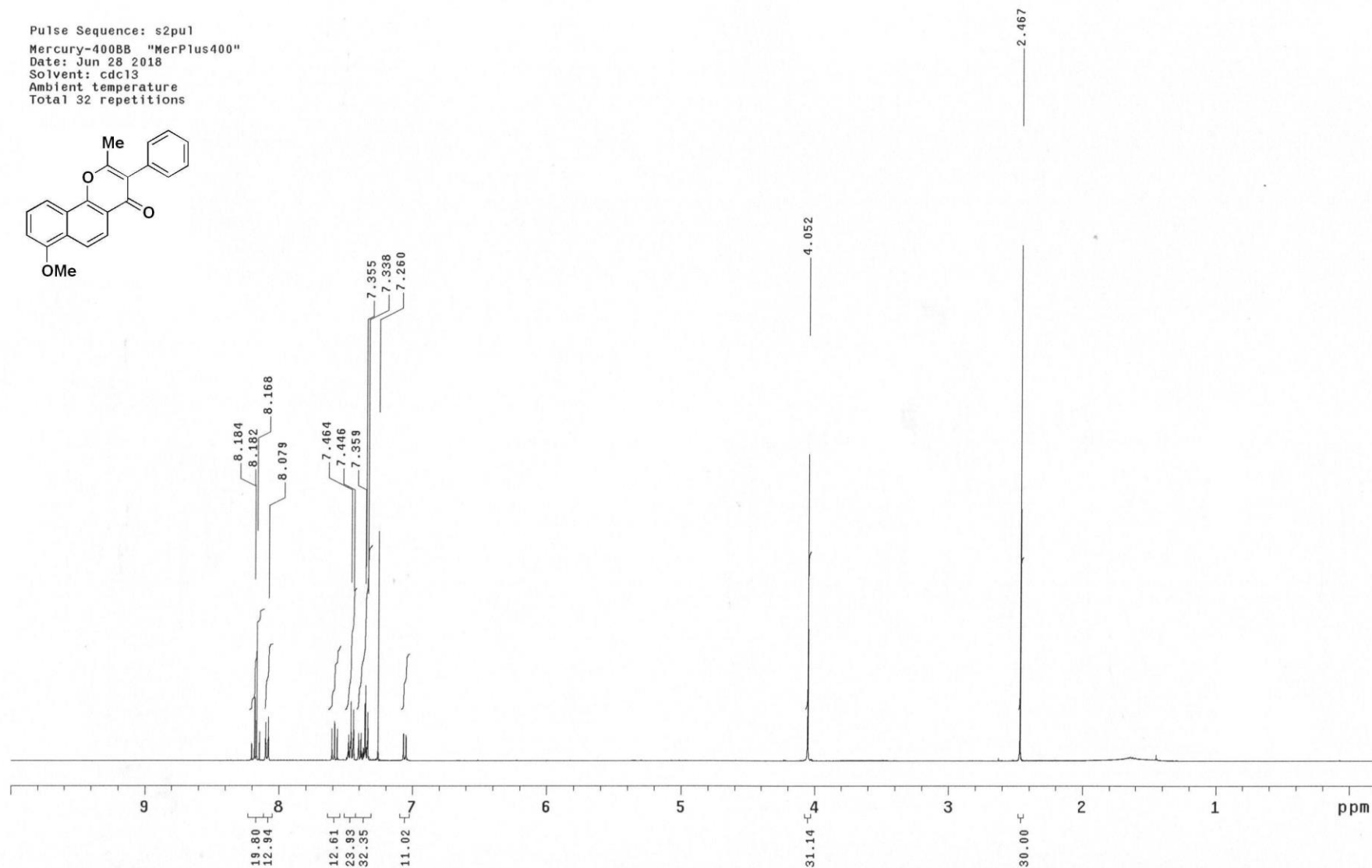
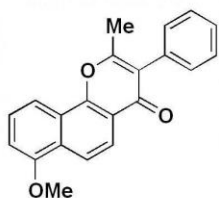
Compound 6m (¹³C-NMR spectral data-2)



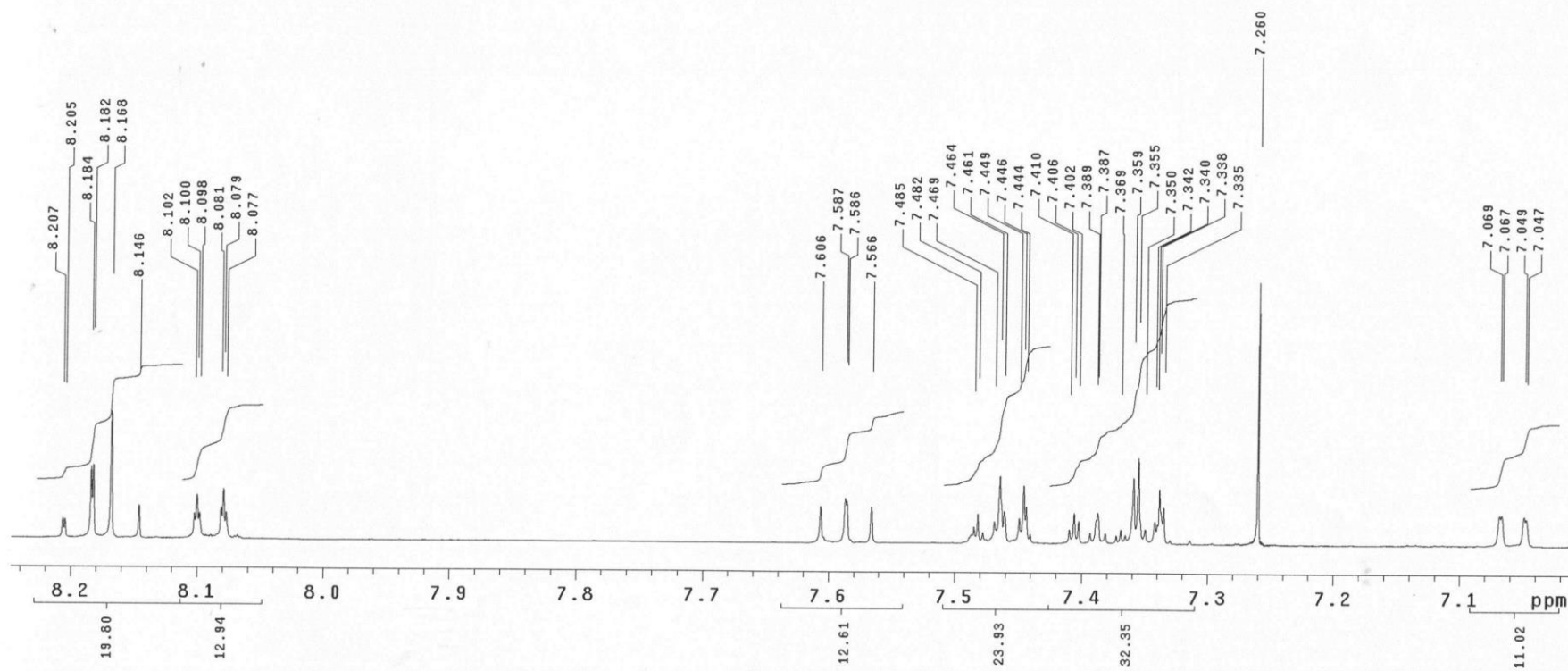
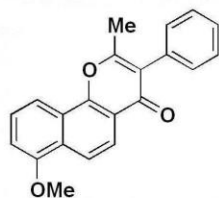
Compound 6n (¹H-NMR spectral data-1)

YT150Me

Pulse Sequence: s2pu1
Mercury-400BB "MerPlus400"
Date: Jun 28 2018
Solvent: cdcl3
Ambient temperature
Total 32 repetitions



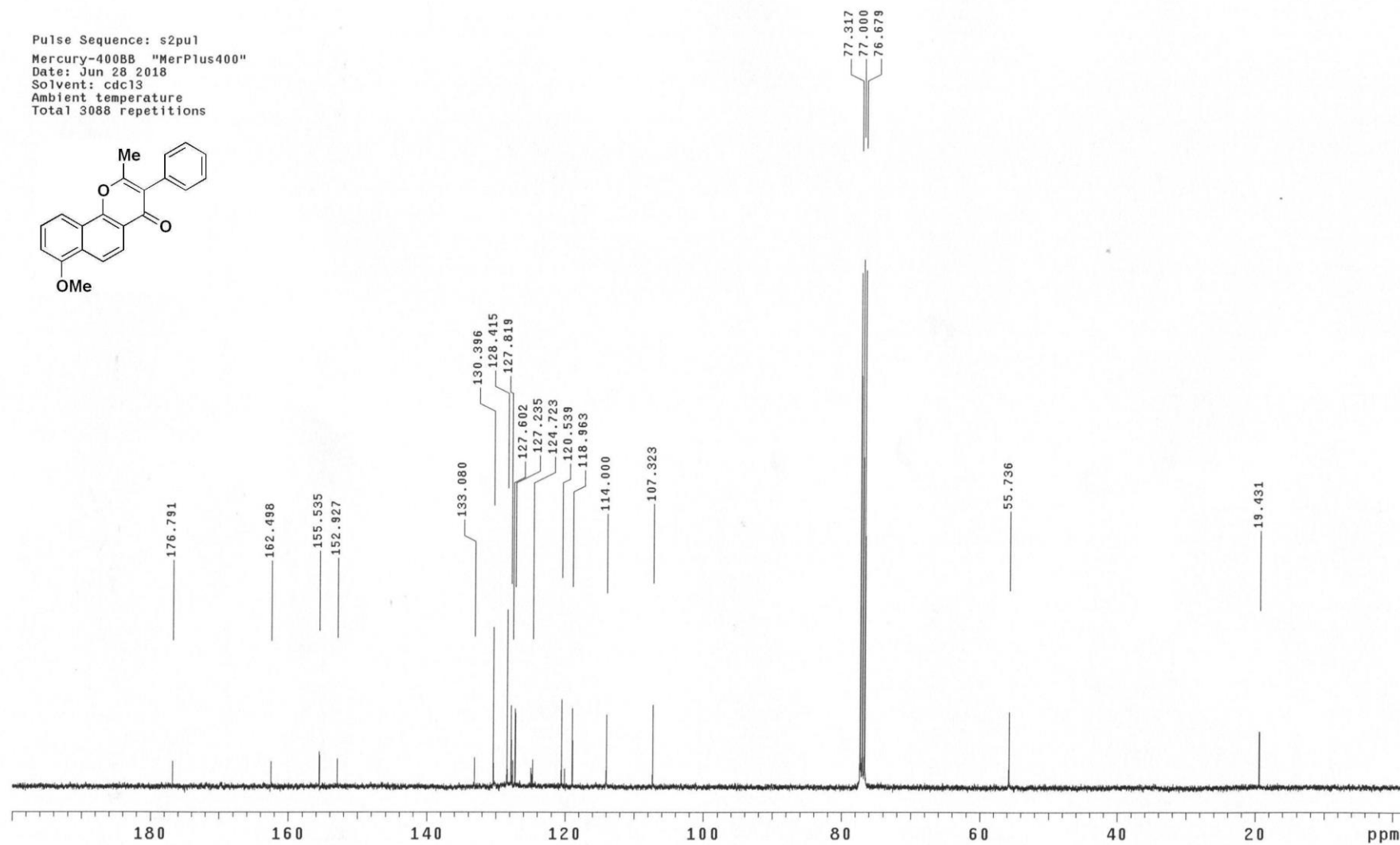
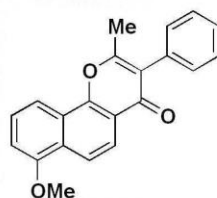
Compound 6n (¹H-NMR spectral data-2)



Compound 6n (¹³C-NMR spectral data)

YT150Me

Pulse Sequence: s2pu1
Mercury-400BB "MerPlus400"
Date: Jun 28 2018
Solvent: cdcl3
Ambient temperature
Total 3088 repetitions



Compound 6o (¹H-NMR spectral data-1)

YT4-OMeAC20

Pulse Sequence: s2pul

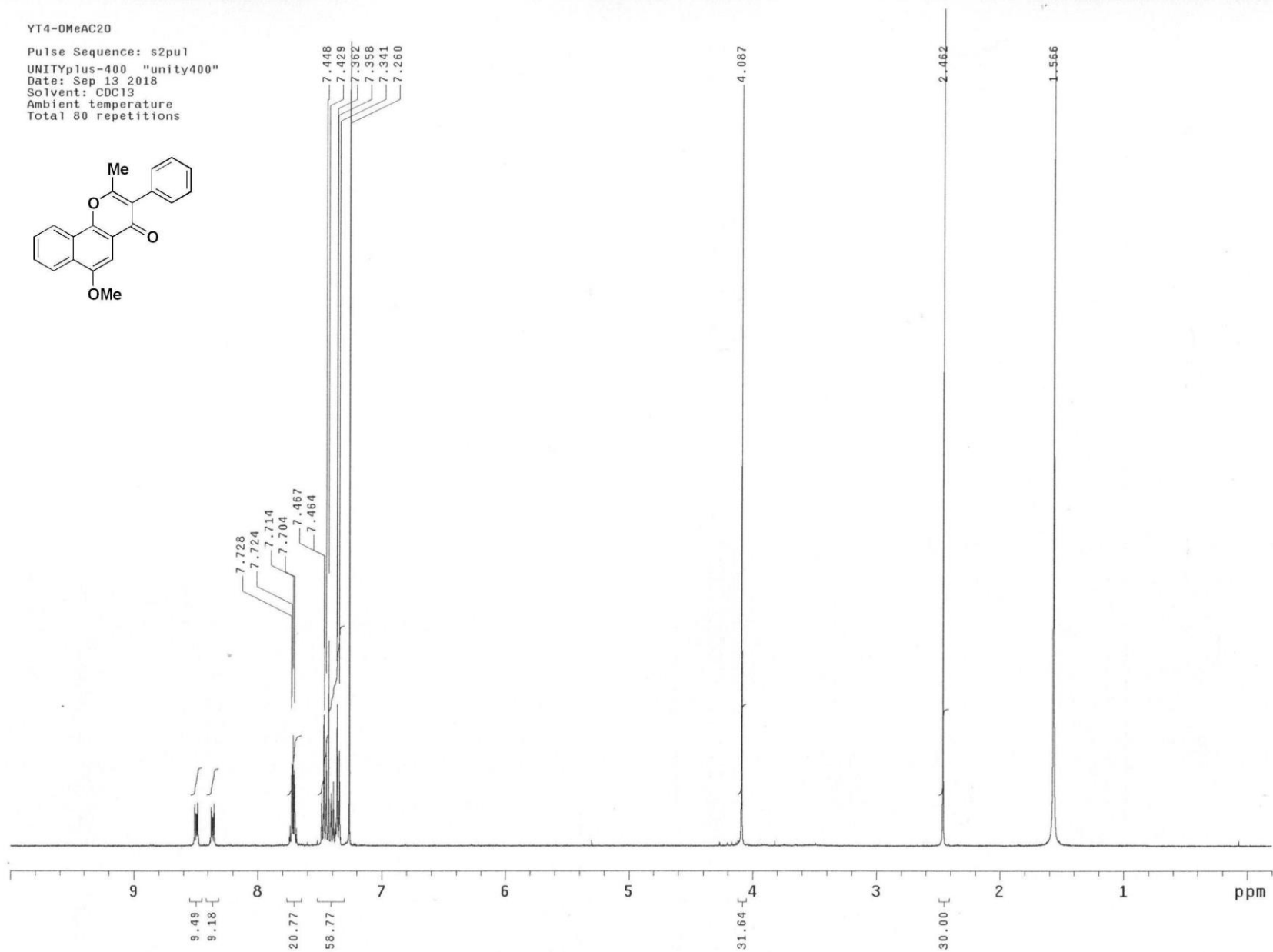
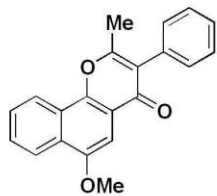
UNITYplus-400 "unity400"

Date: Sep 13 2018

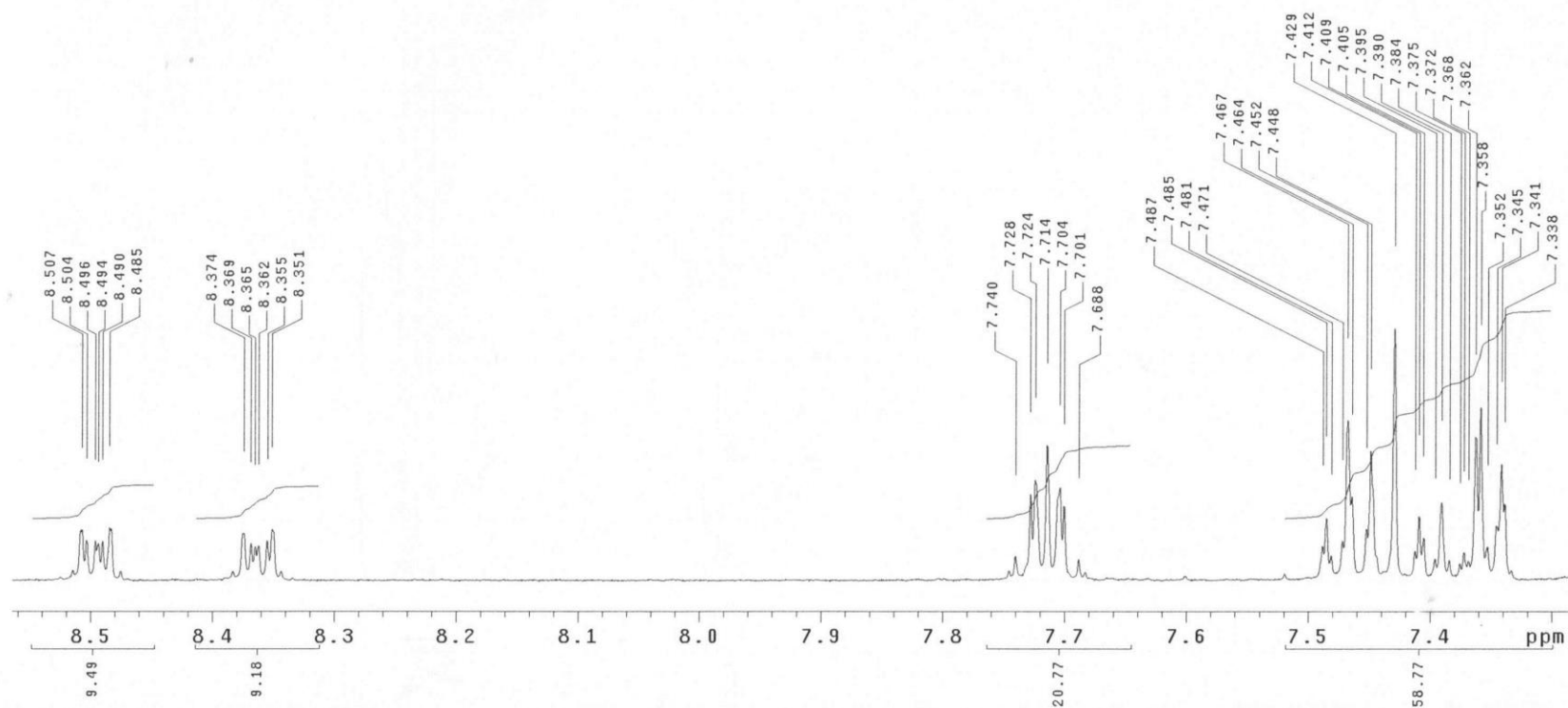
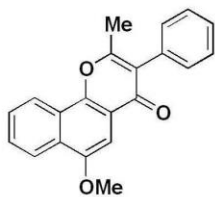
Solvent: CDCl₃

Ambient temperature

Total 80 repetitions



Compound 6o (¹H-NMR spectral data-2)



Compound 6o (¹³C-NMR spectral data)

YT4-OMeAC20

Pulse Sequence: s2pu1

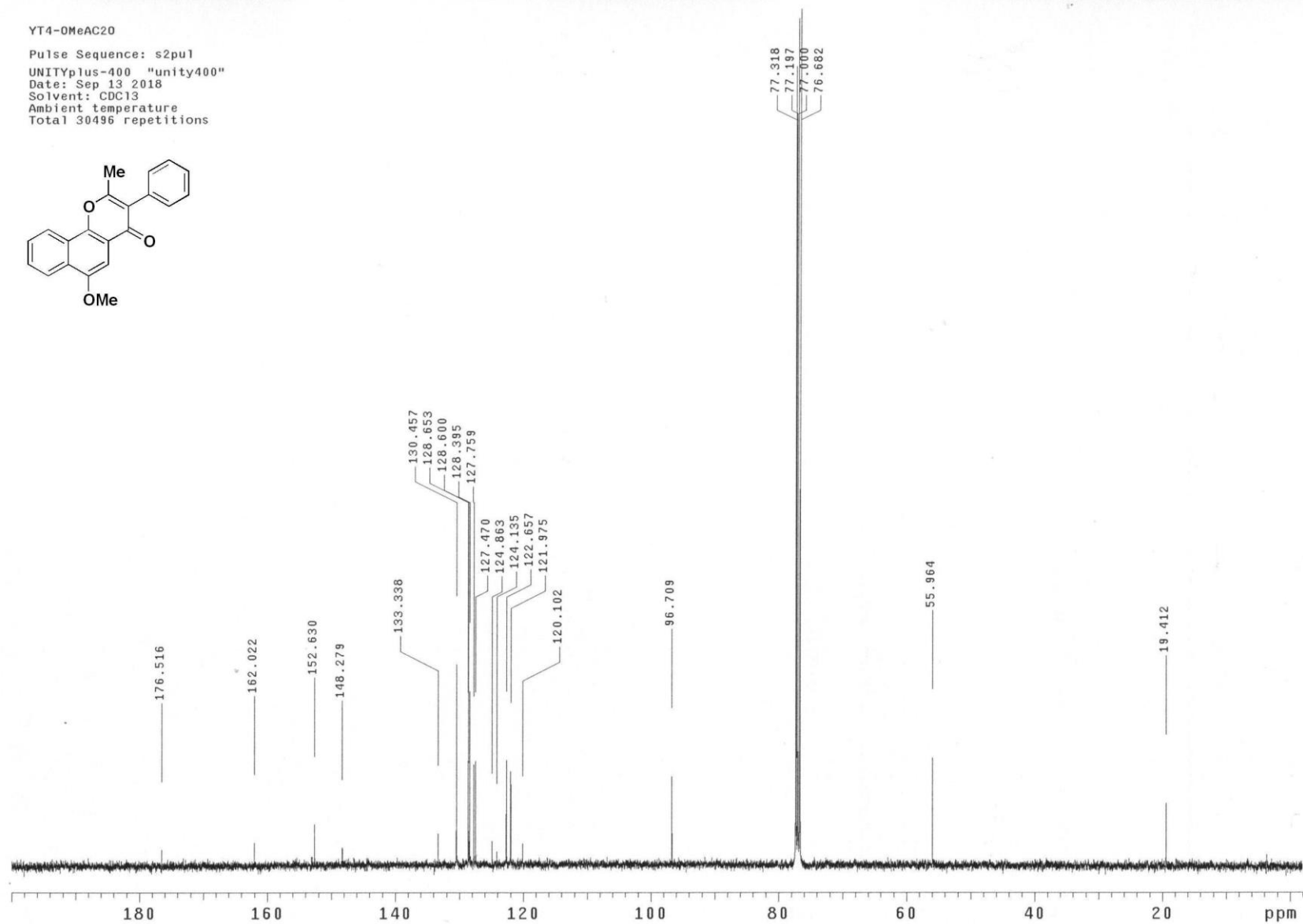
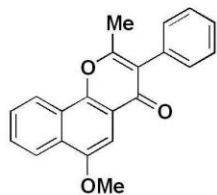
UNITYplus-400 "unity400"

Date: Sep 13 2018

Solvent: CDCl₃

Ambient temperature

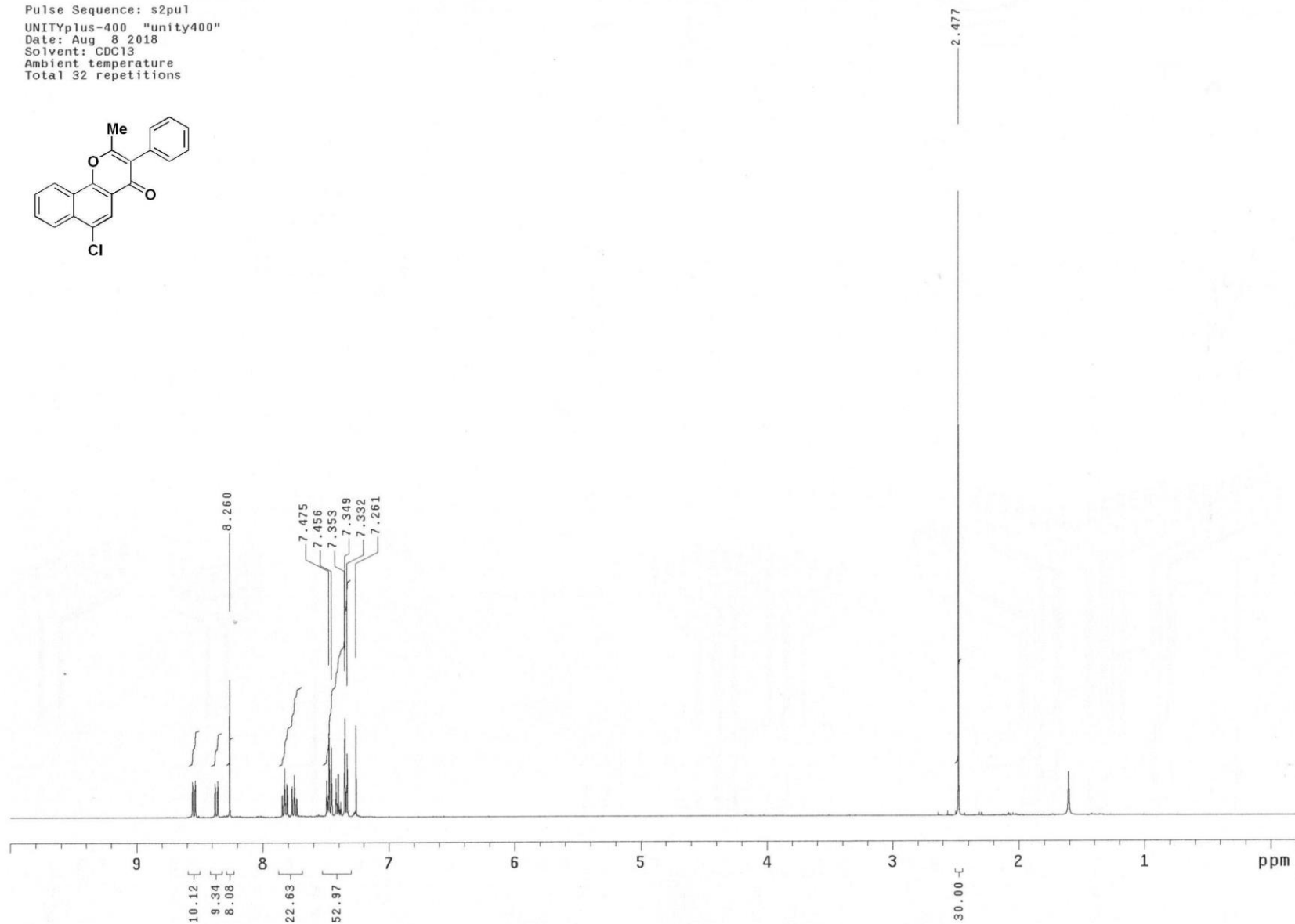
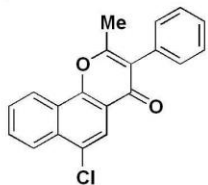
Total 30496 repetitions



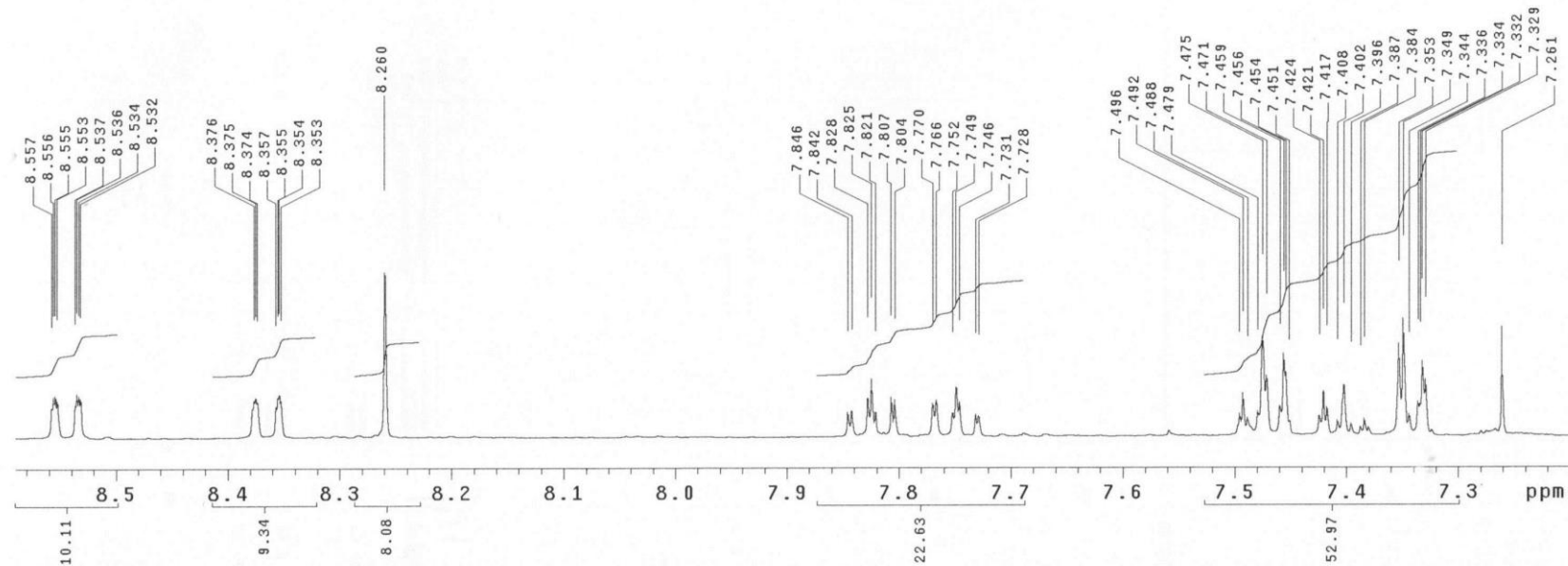
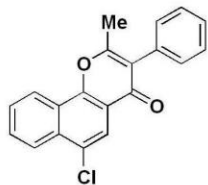
Compound 6p (¹H-NMR spectral data-1)

YT4-C1AC20

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Aug 8 2018
Solvent: CDCl₃
Ambient temperature
Total 32 Repetitions



Compound 6p (¹H-NMR spectral data-2)



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

YT4-C1AC20

Pulse Sequence: s2pu1

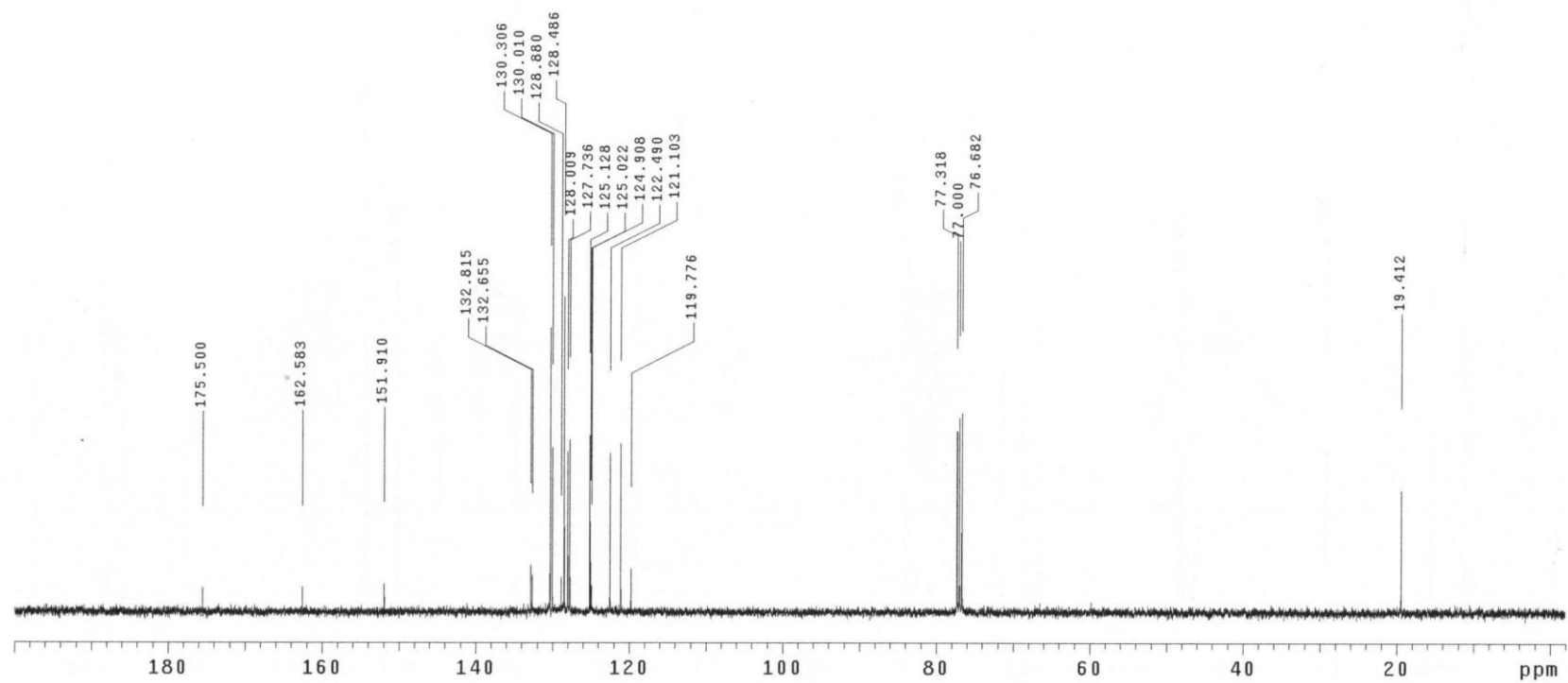
UNITYplus-400 "unity400"

Date: Aug 8 2018

Solvent: CDCl_3

Ambient temperature

Total 1104 repetitions



Compound 8a (¹H-NMR spectral data)

SPhPh20

Pulse Sequence: s2pu1

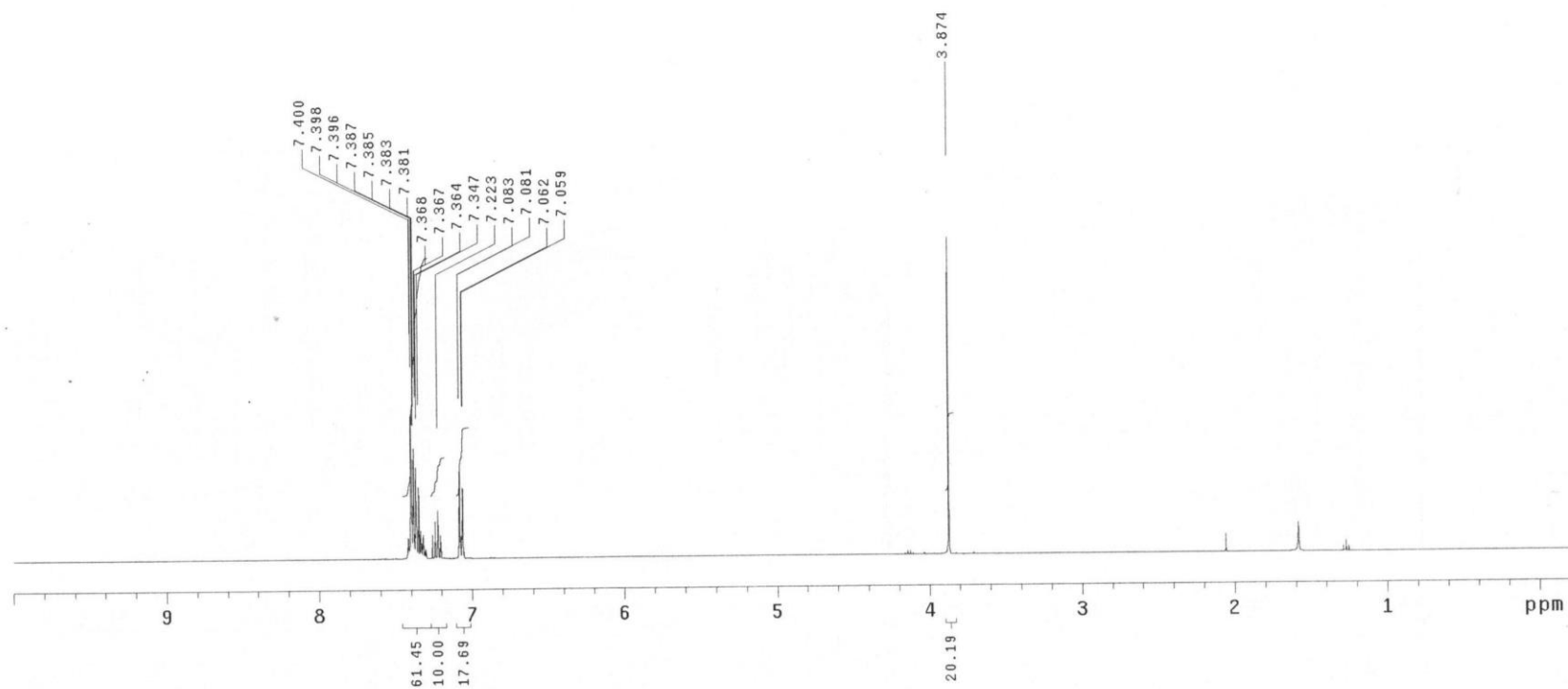
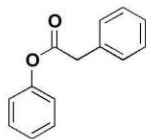
UNITYplus-400 "unity400"

Date: May 23 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 8a (^{13}C -NMR spectral data)

SHPh20

Pulse Sequence: s2pu1

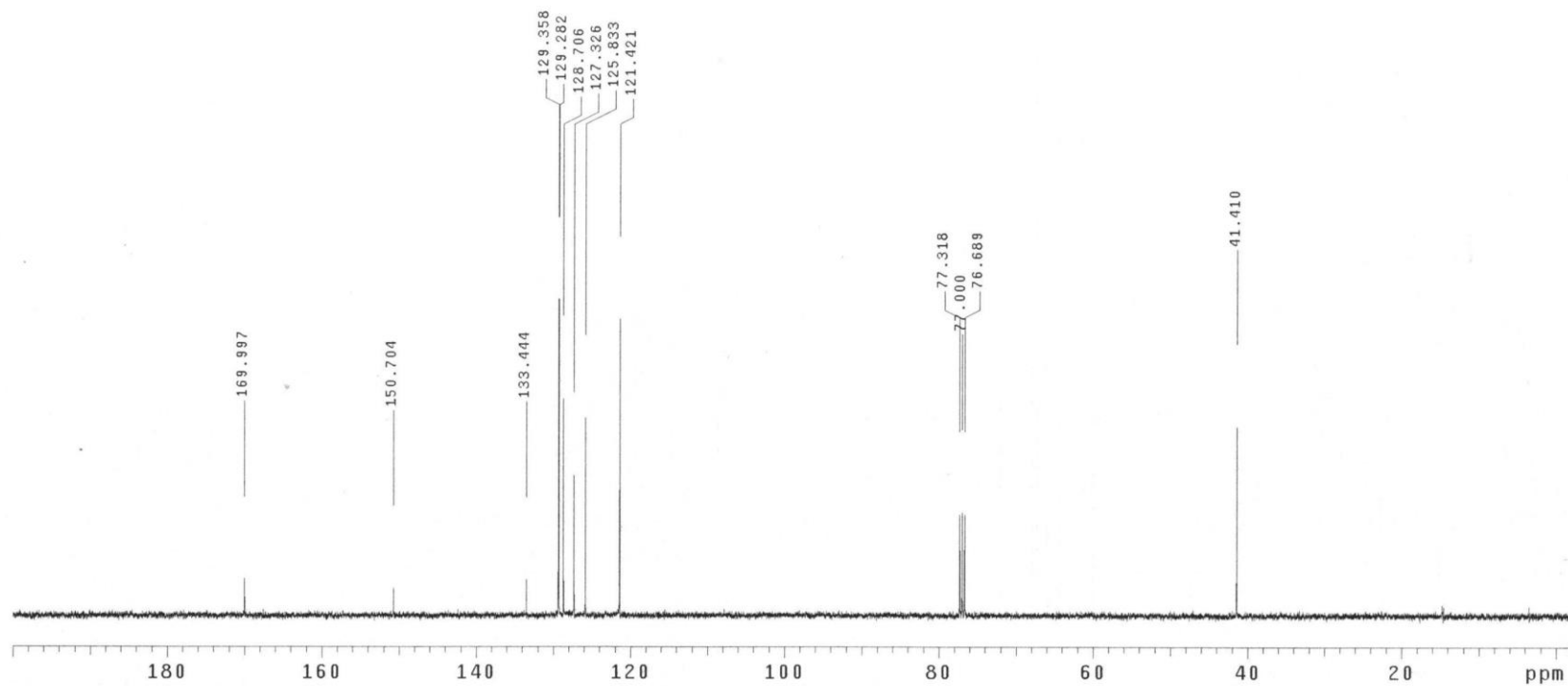
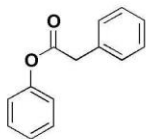
UNITYplus-400 "unity400"

Date: May 23 2018

Solvent: CDCl_3

Ambient temperature

Total 560 repetitions



Compound 8b (¹H-NMR spectral data)

SHF20

Pulse Sequence: s2pu1

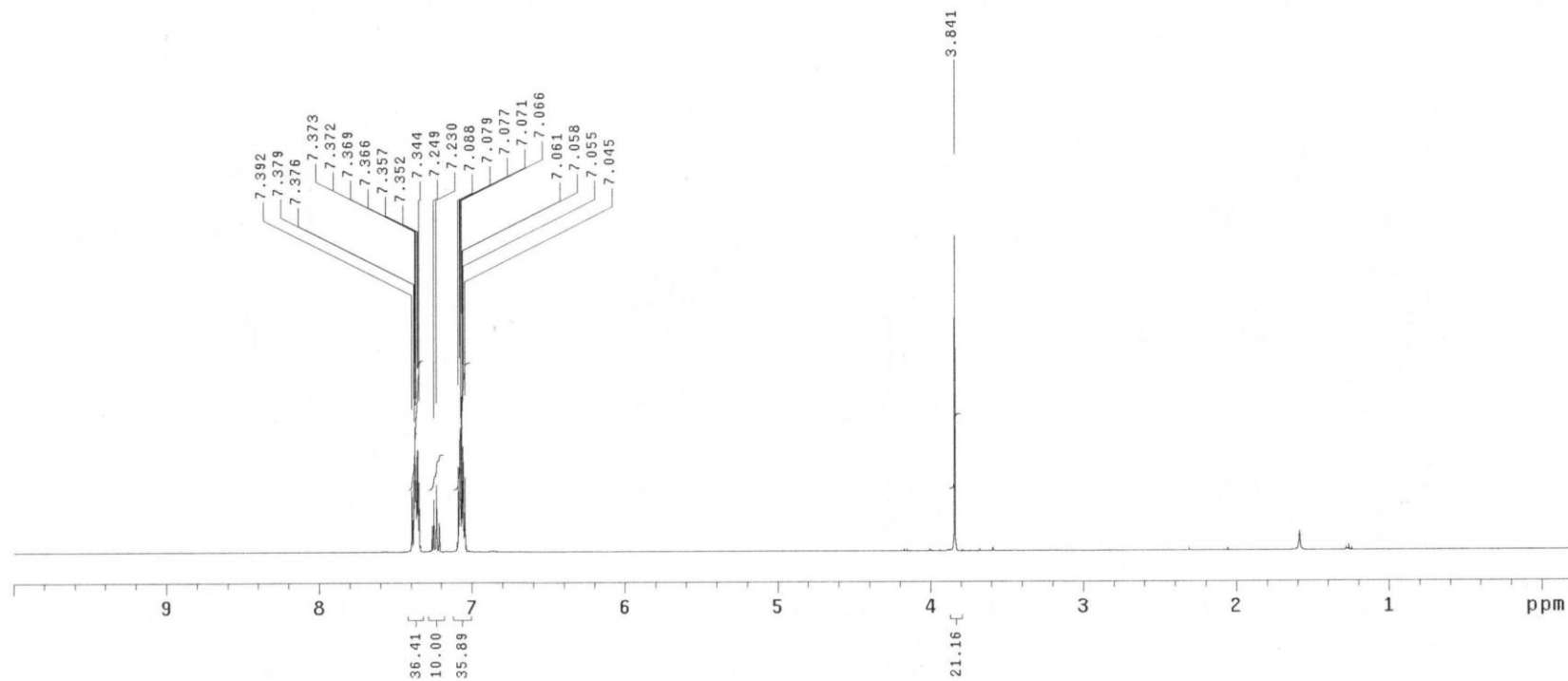
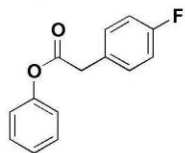
UNITYplus-400 "unity400"

Date: May 28 2018

Solvent: CDCl₃

Ambient temperature

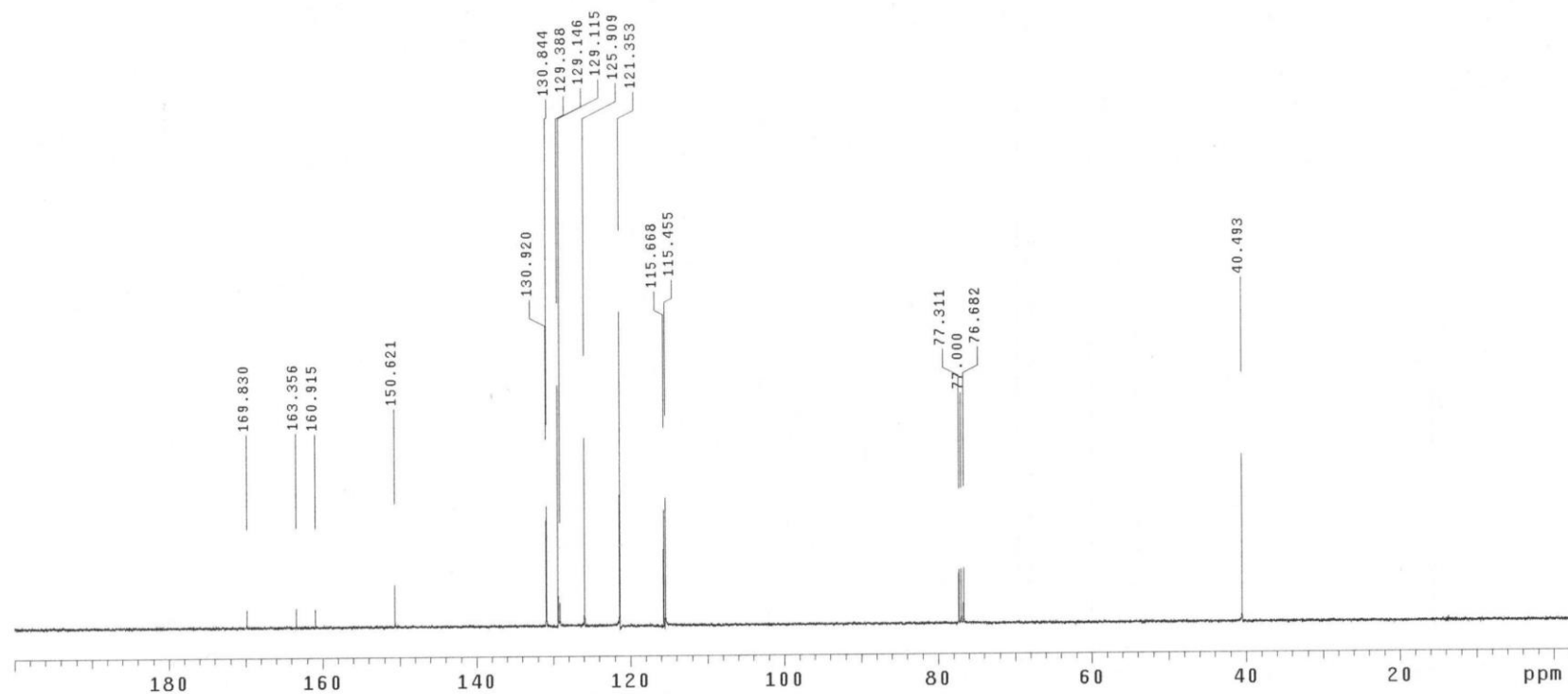
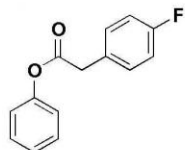
Total 32 repetitions



Compound 8b (^{13}C -NMR spectral data)

SHF20

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: May 28 2018
Solvent: CDCl₃
Ambient temperature
Total 1136 repetitions



Compound 8c (¹H-NMR spectral data)

SHB1Ph

Pulse Sequence: s2pu1

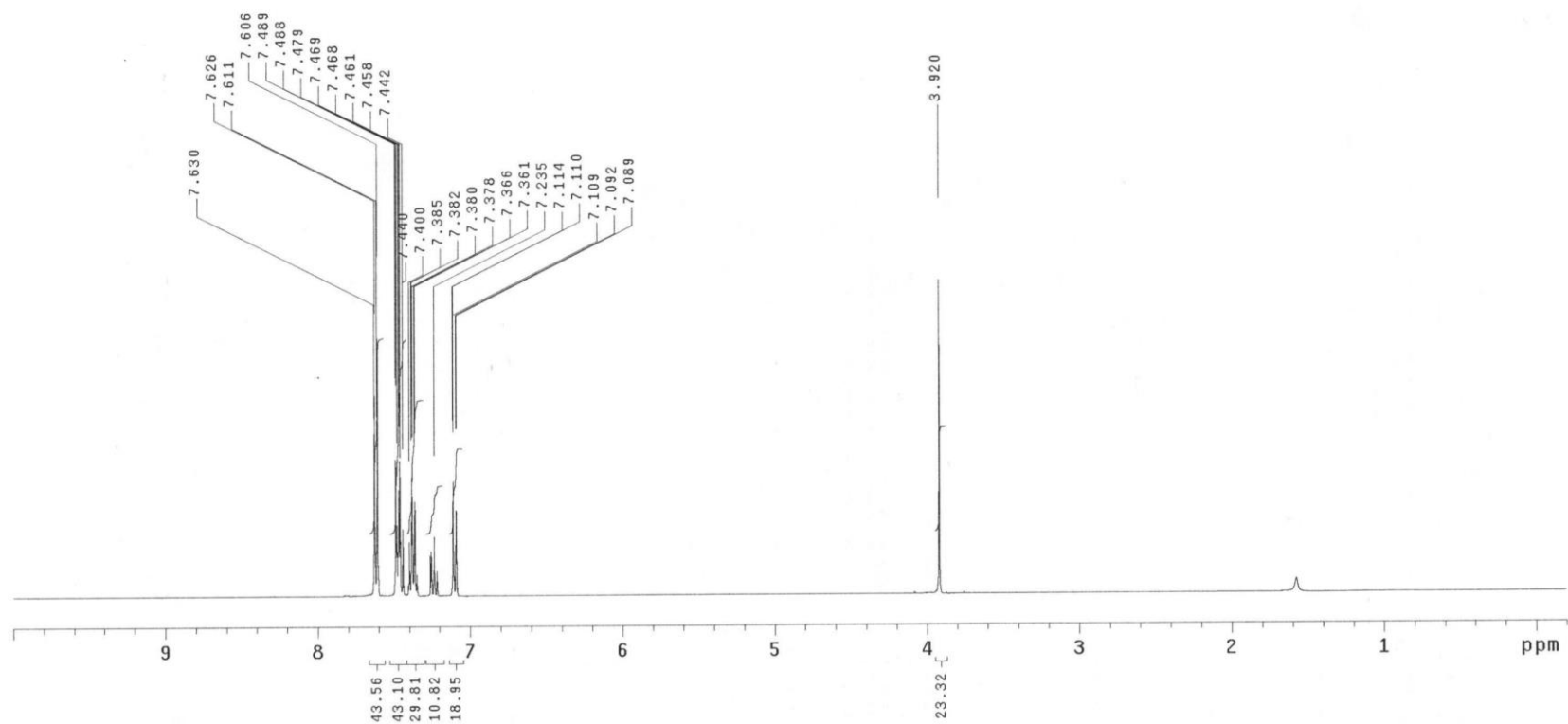
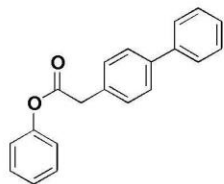
UNITYplus-400 "unity400"

Date: Jun 8 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 8c (¹³C-NMR spectral data)

SHB1Ph

Pulse Sequence: s2pu1

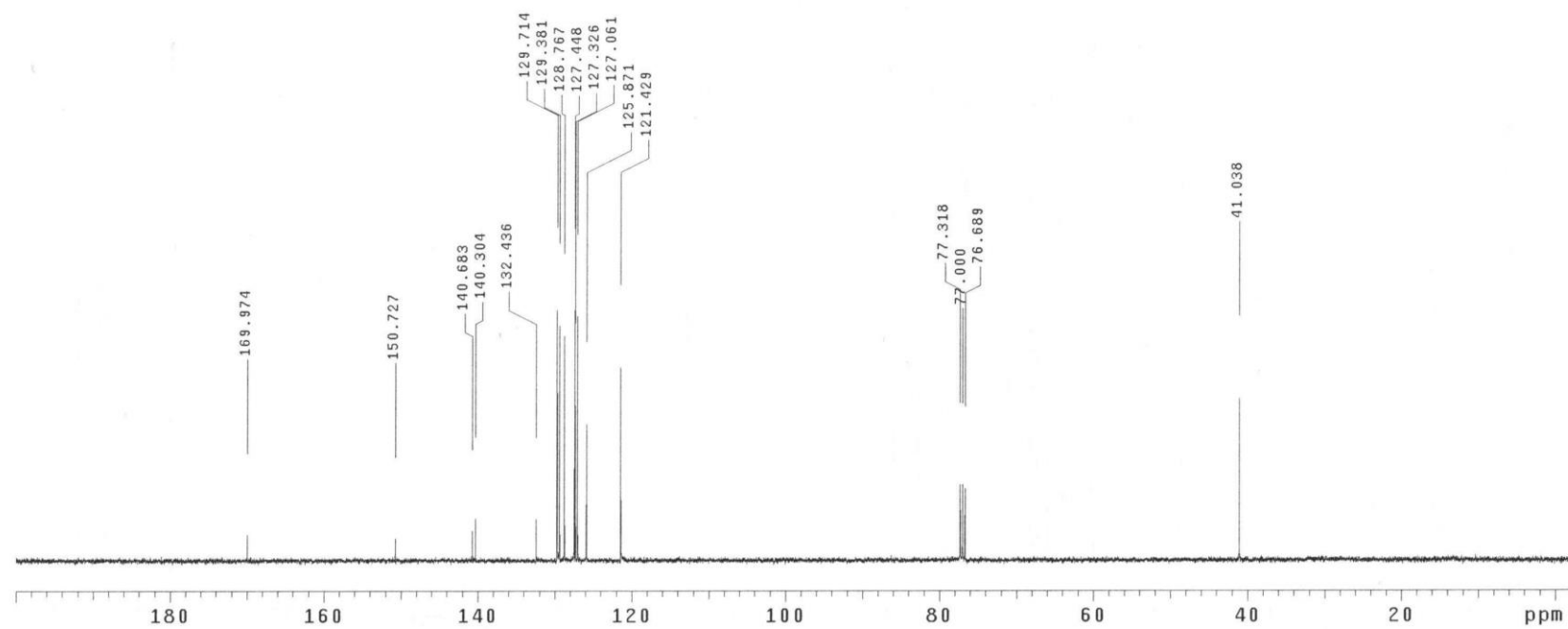
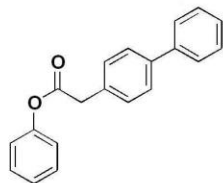
UNITYplus-400 "unity400"

Date: Jun 8 2018

Solvent: CDCl₃

Ambient temperature

Total 704 repetitions



Compound 8d (¹H-NMR spectral data)

SH2F19

Pulse Sequence: s2pu1

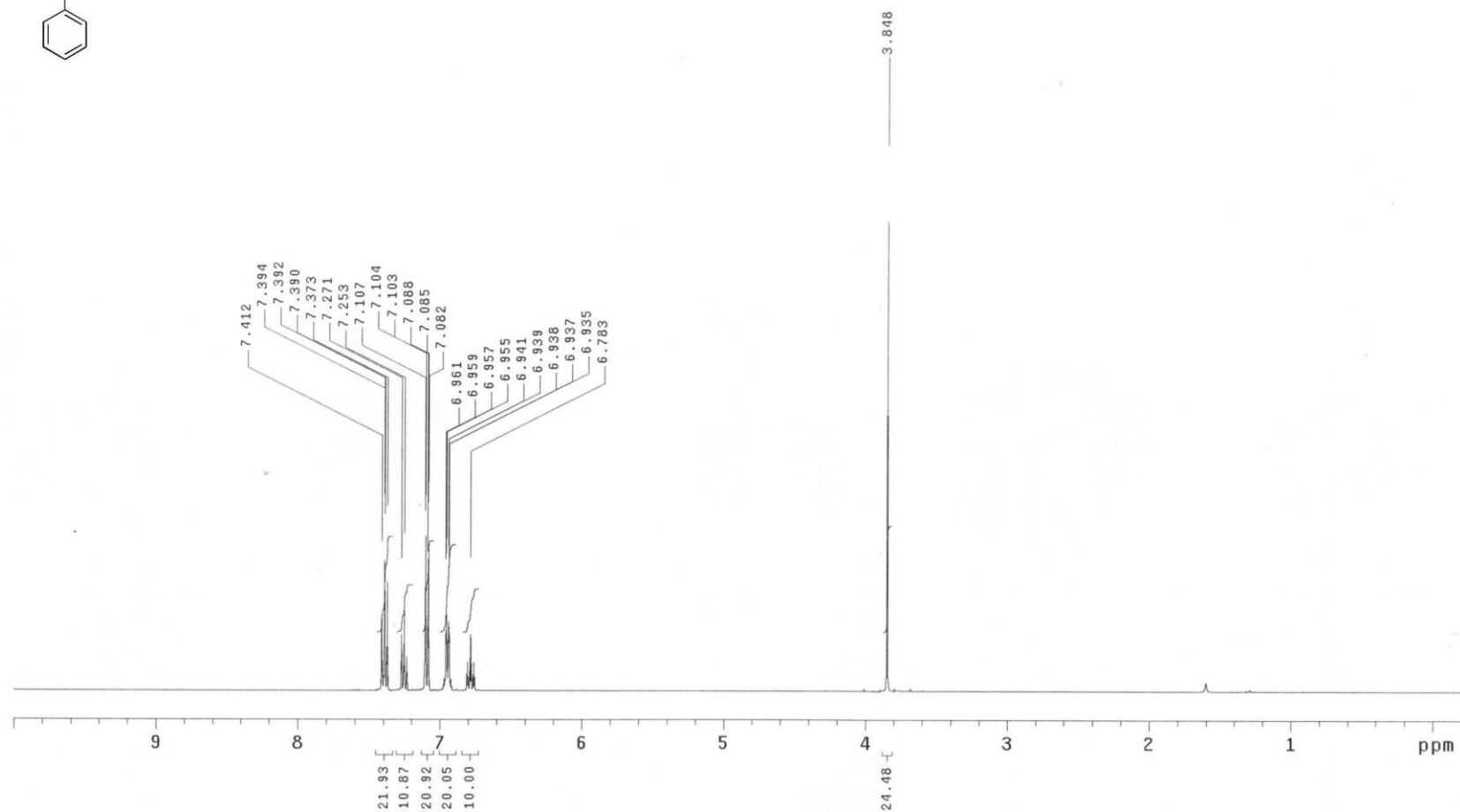
UNITYplus-400 "unity400"

Date: Jun 6 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 8d (¹³C-NMR spectral data)

SH2F19

Pulse Sequence: s2pu1

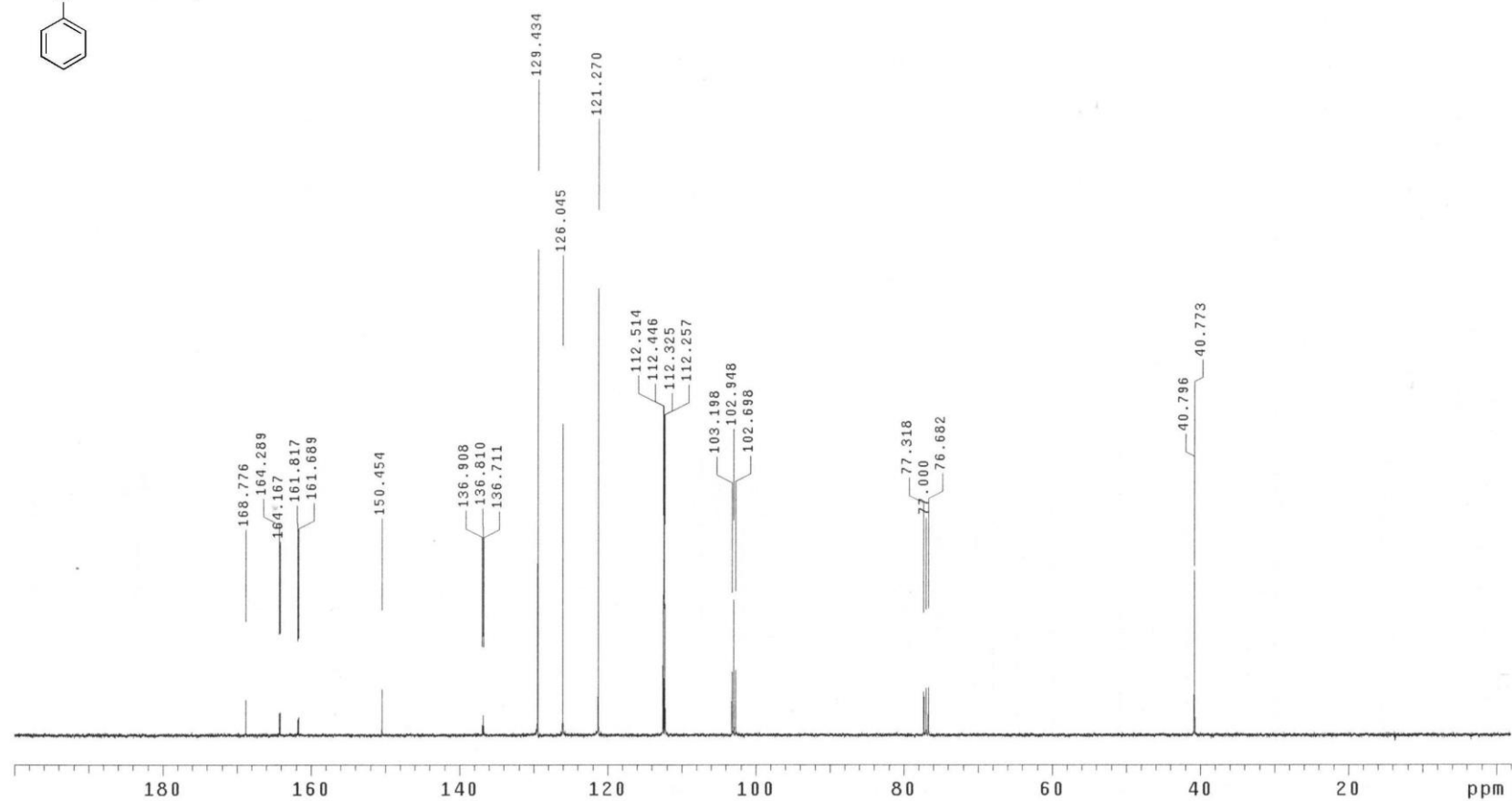
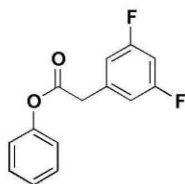
UNITYplus-400 "unity400"

Date: Jun 6 2018

Solvent: CDCl₃

Ambient temperature

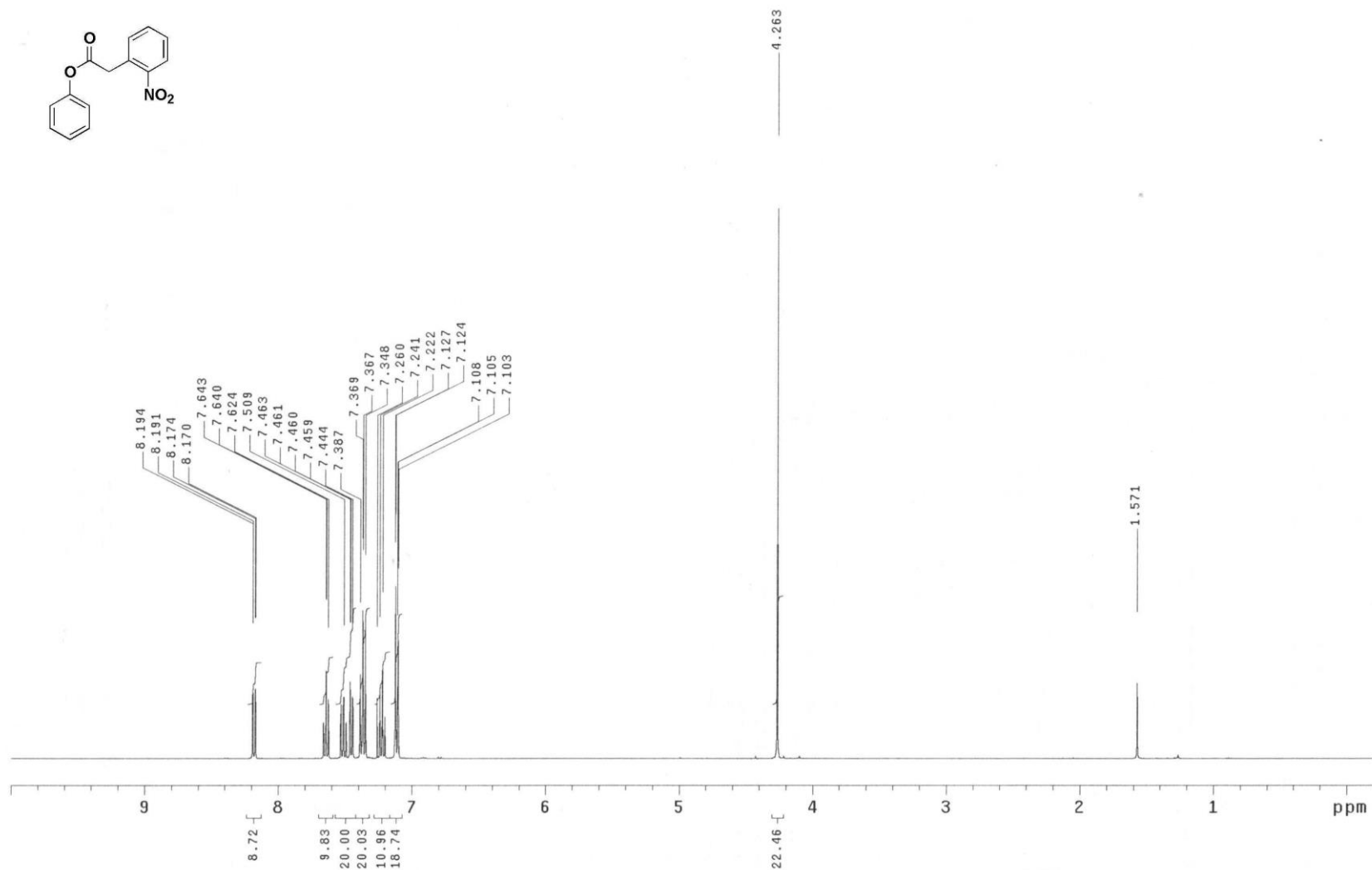
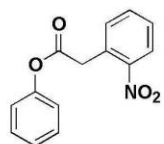
Total 512 repetitions



Compound 8e (¹H-NMR spectral data)

SHN02 32

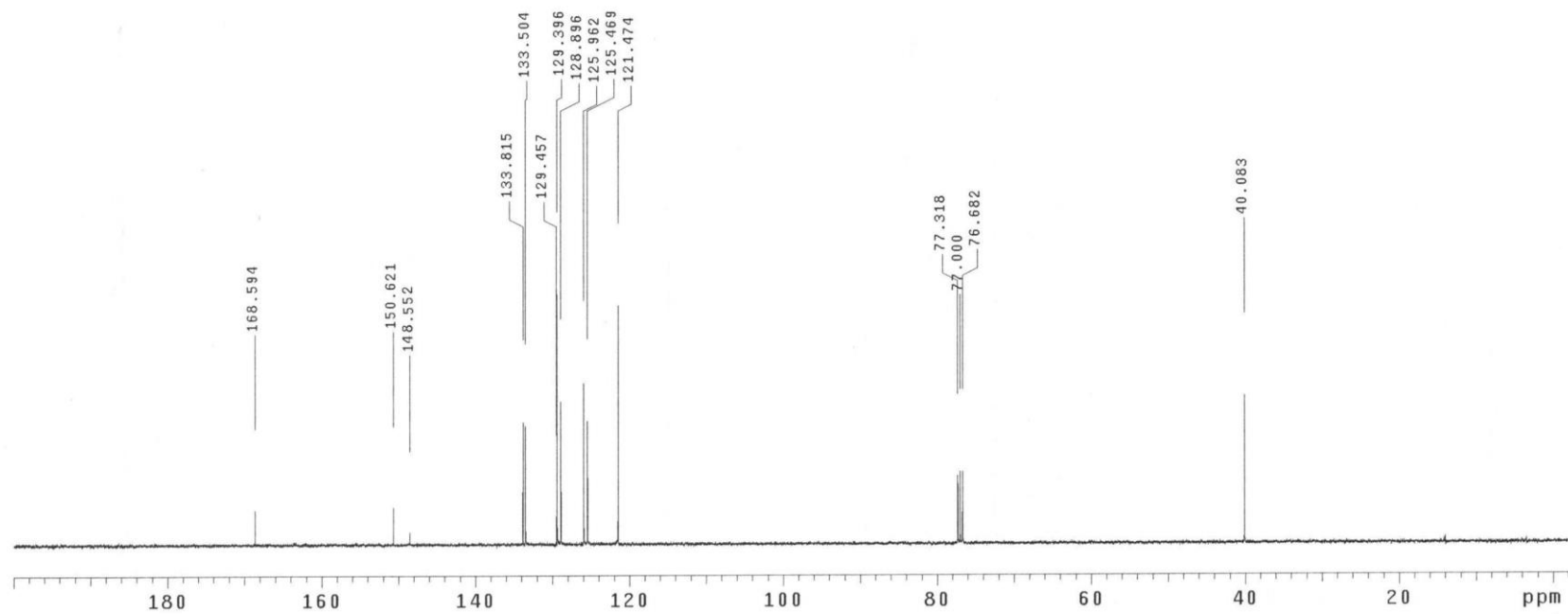
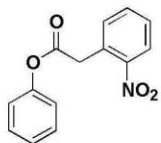
Pulse Sequence: s2pu1
 UNITYplus-400 "unity400"
 Date: Jun 11 2018
 Solvent: CDCl₃
 Ambient temperature
 Total 32 repetitions



Compound 8e (¹³C-NMR spectral data)

SHN02 32

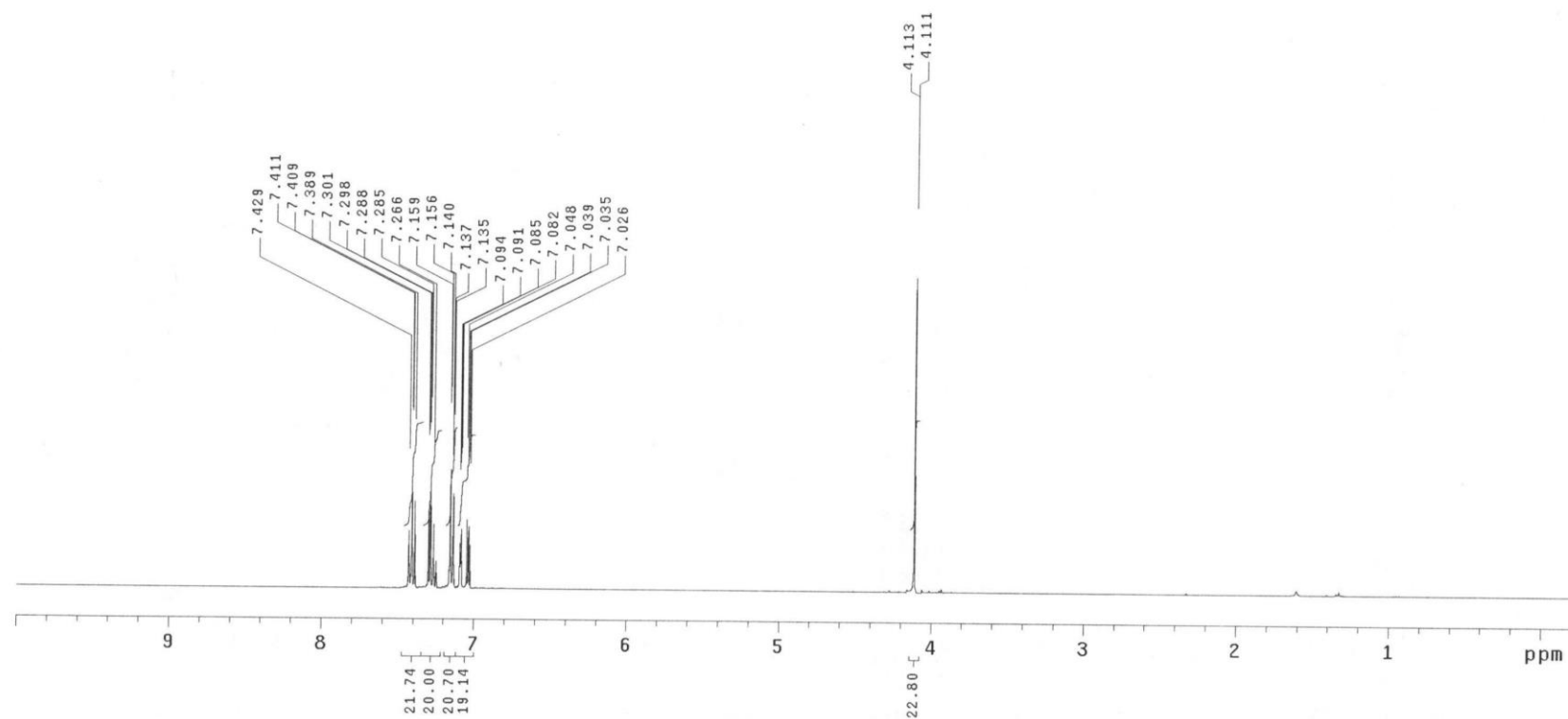
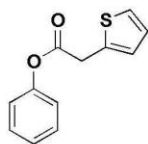
Pulse Sequence: s2pul
UNITYplus-400 "unity400"
Date: Jun 11 2018
Solvent: CDCl₃
Ambient temperature
Total 1216 repetitions



Compound 8f (¹H-NMR spectral data)

SHS20

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Jun 5 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 8f (¹³C-NMR spectral data)

SHS20

Pulse Sequence: s2pu1

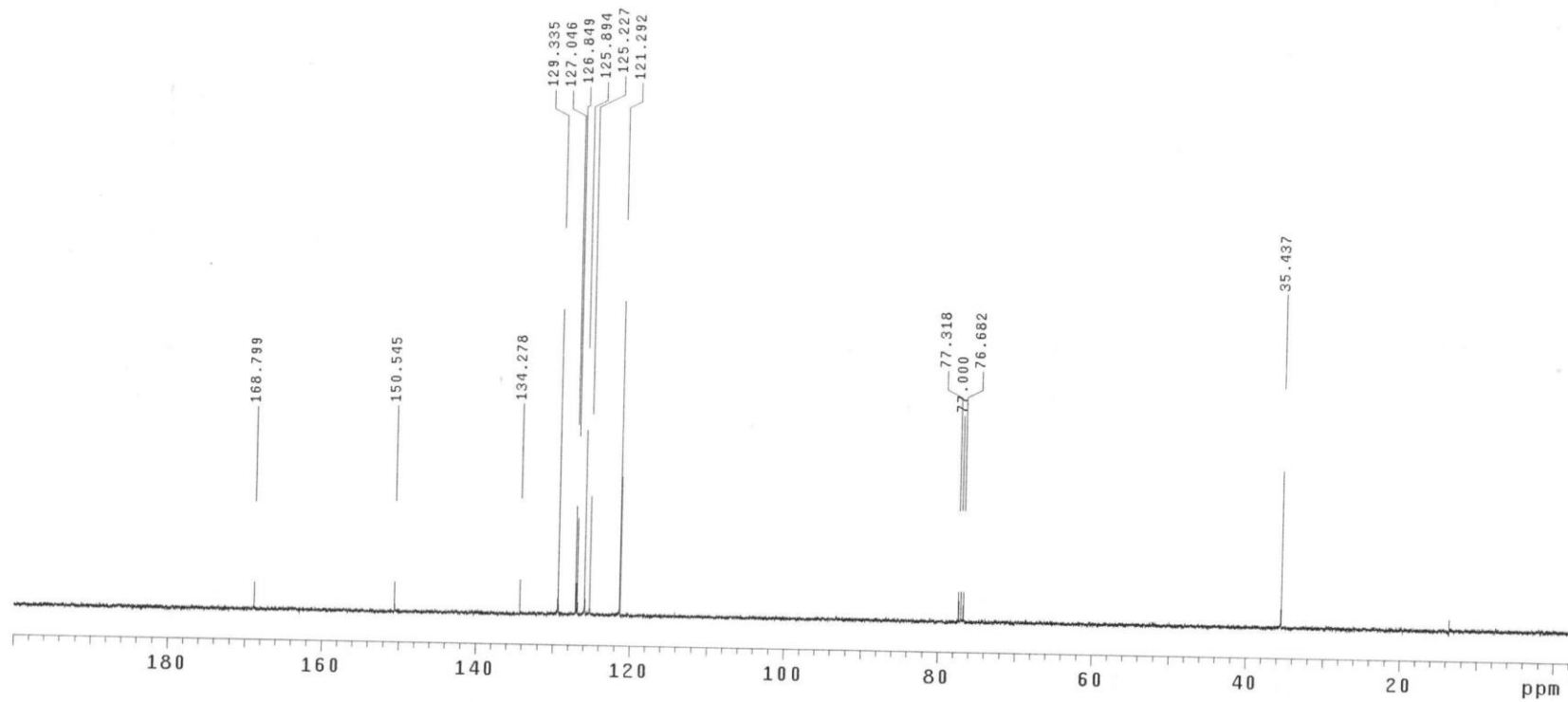
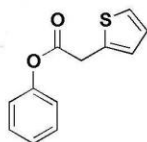
UNITYplus-400 "unity400"

Date: Jun 5 2018

Solvent: CDCl₃

Ambient temperature

Total 64 repetitions



Compound 9a (¹H-NMR spectral data)

YTPH1C13

Pulse Sequence: s2pu1

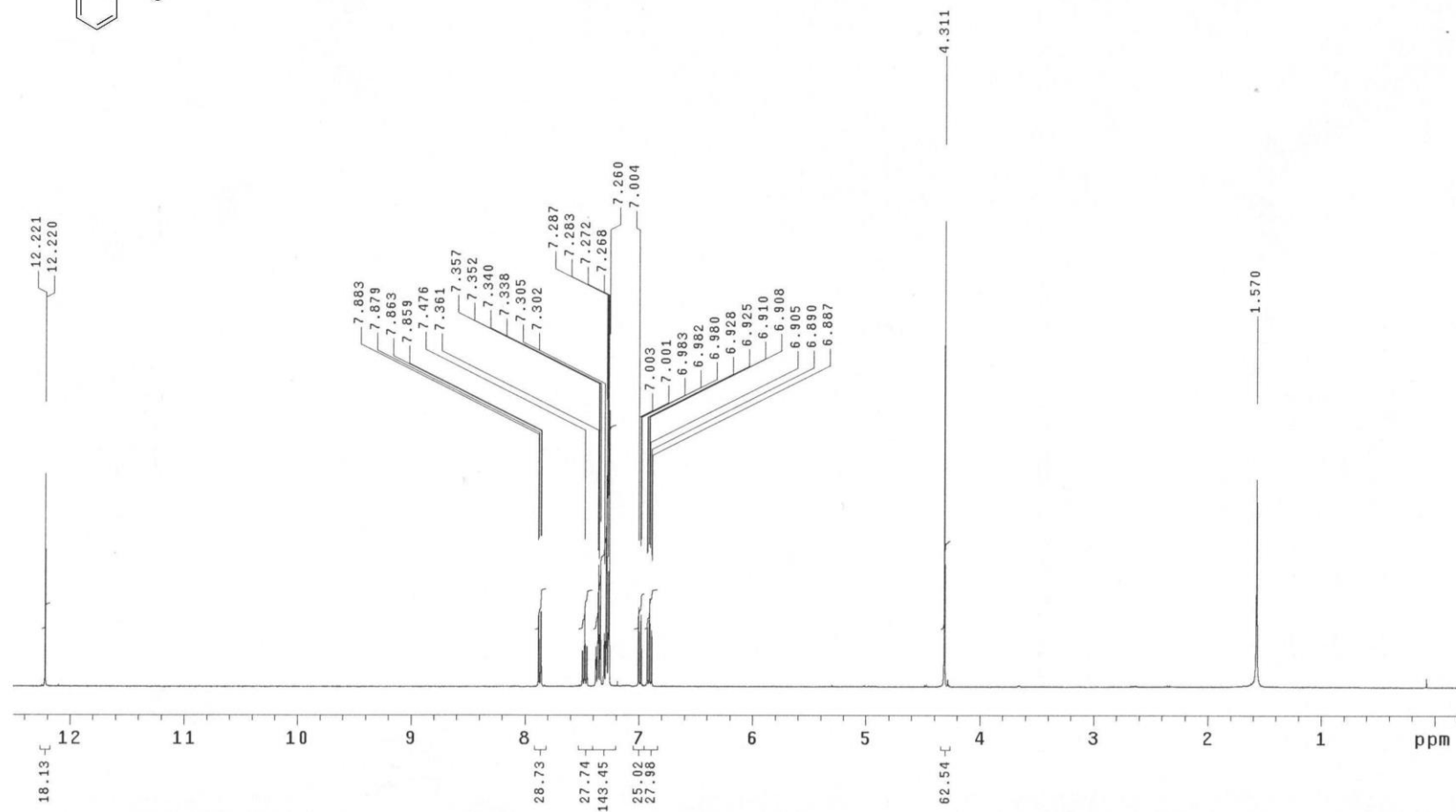
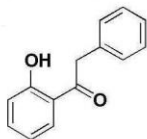
UNITYplus-400 "unity400"

Date: Aug 30 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



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

YTPH41C13

Pulse Sequence: s2pul

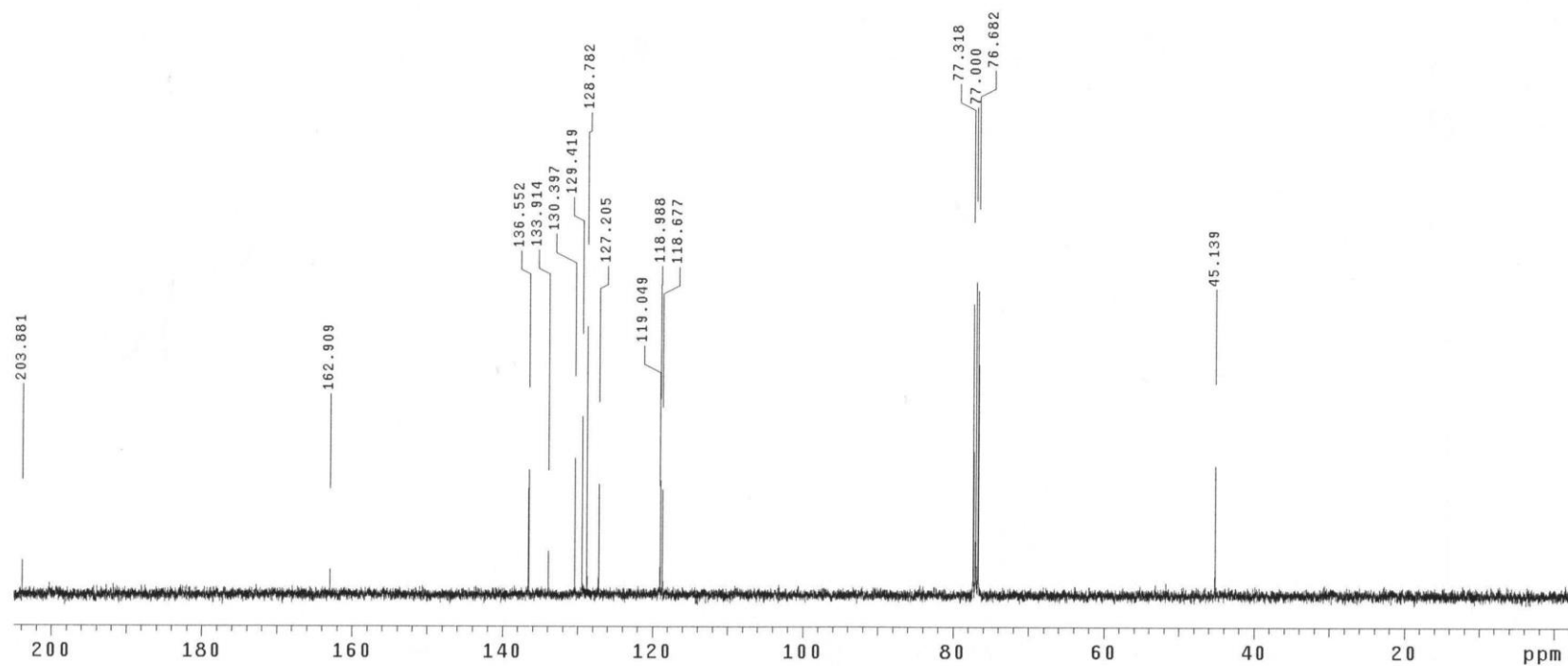
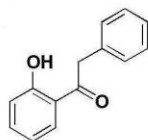
UNITYplus-400 "unity400"

Date: Aug 30 2018

Solvent: CDCl₃

Ambient temperature

Total 2720 repetitions



Compound 9b (¹H-NMR spectral data)

SHFA1C13

Pulse Sequence: s2pu1

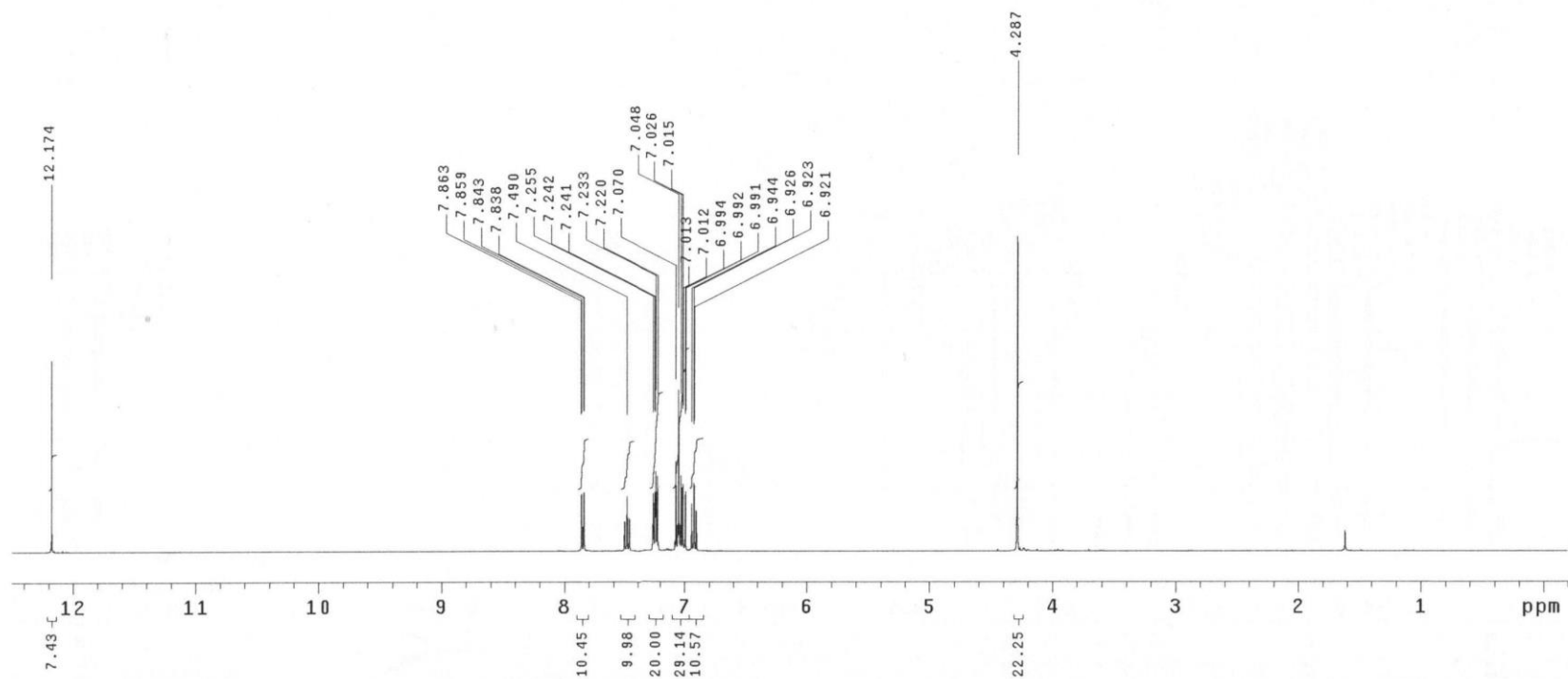
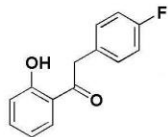
UNITYplus-400 "unity400"

Date: Aug 2 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 9b (¹³C-NMR spectral data)

SHFA1C13

Pulse Sequence: s2pu1

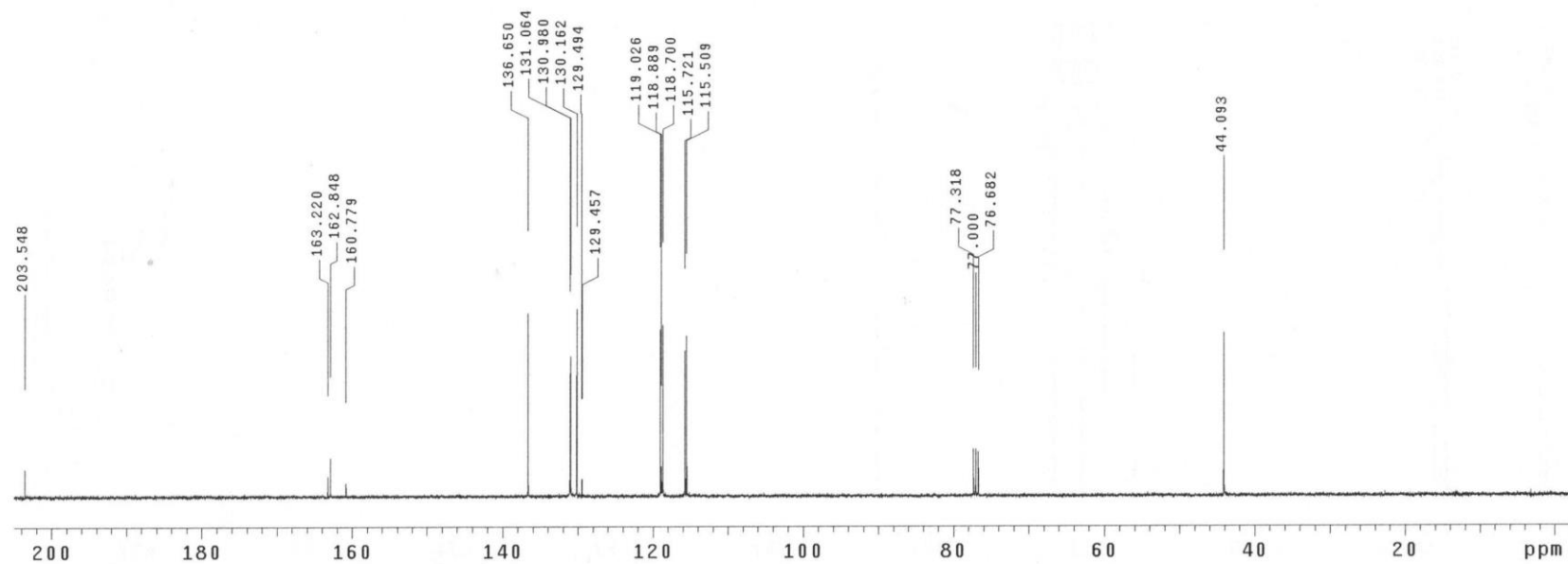
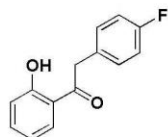
UNITYplus-400 "unity400"

Date: Aug 2 2018

Solvent: CDCl₃

Ambient temperature

Total 544 repetitions



Compound 9c (¹H-NMR spectral data-1)

SHBiPhA1C13

Pulse Sequence: s2pu1

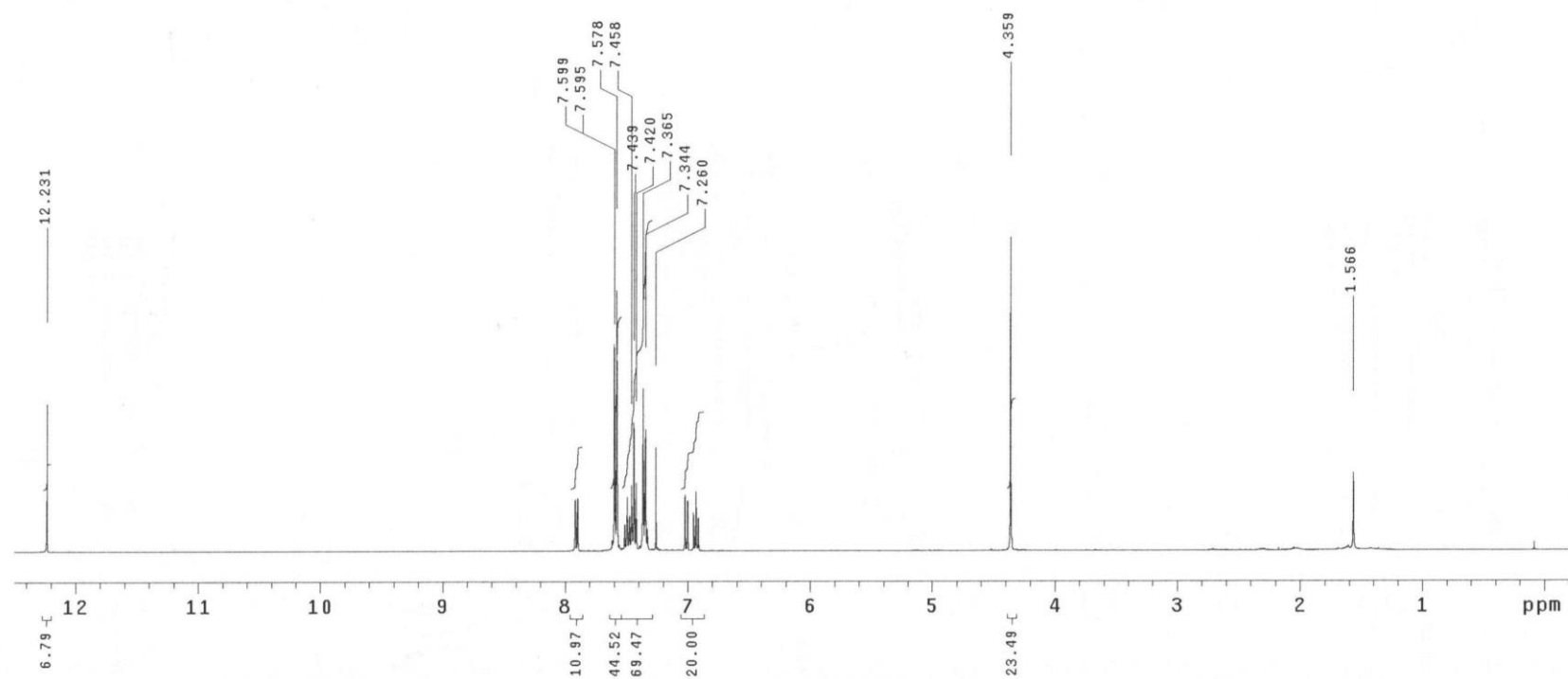
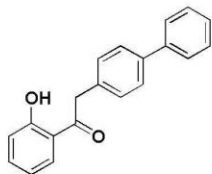
UNITYplus-400 "unity400"

Date: Aug 8 2018

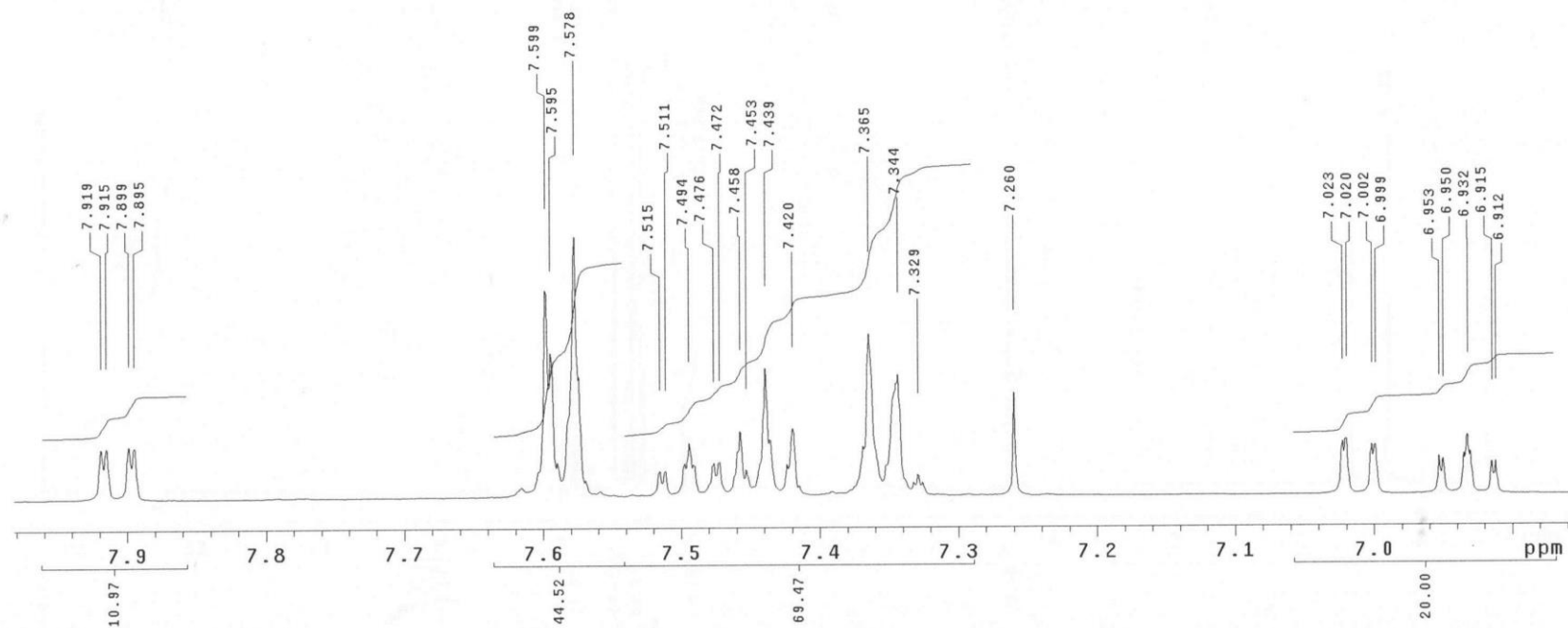
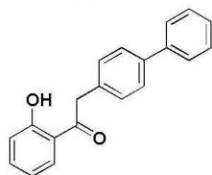
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 9c (¹H-NMR spectral data-2)



Compound 9c (¹³C-NMR spectral data)

SHBiPhA1C13

Pulse Sequence: s2pu1

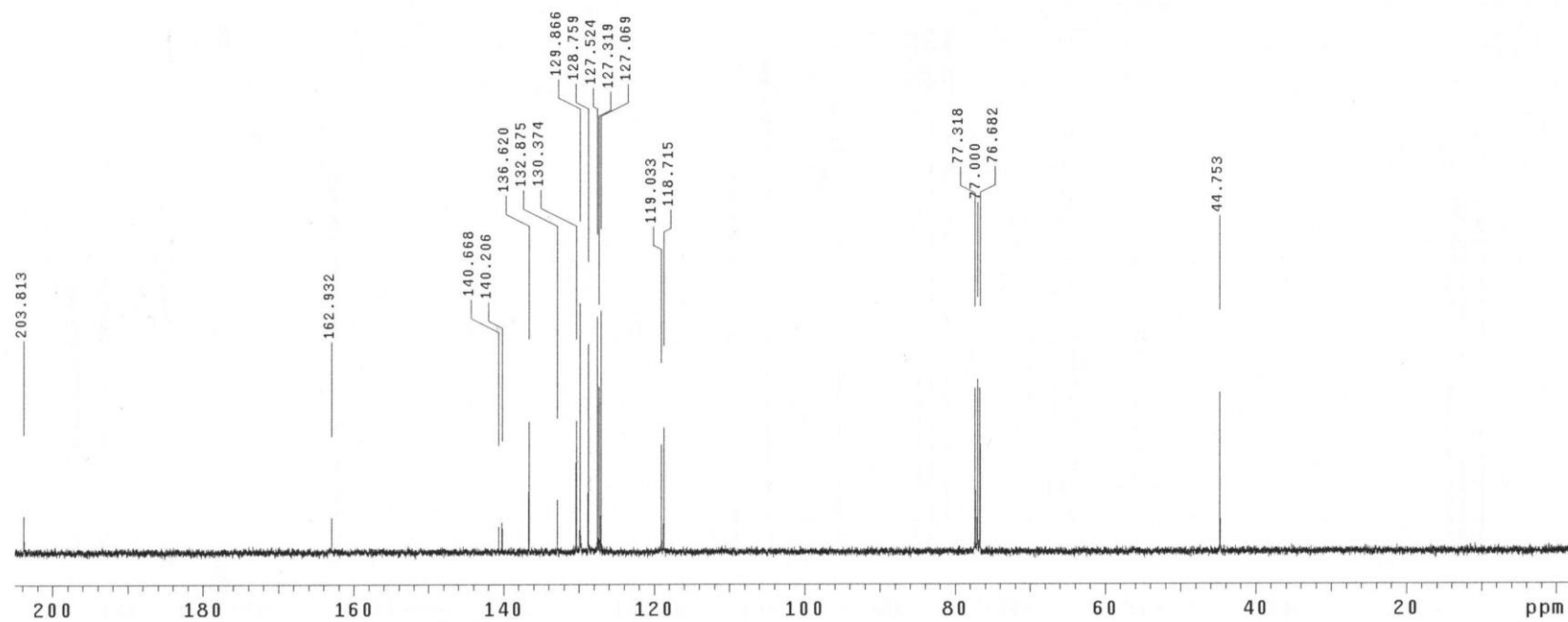
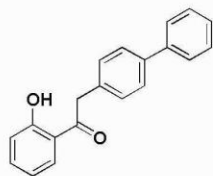
UNITYplus-400 "unity400"

Date: Aug 8 2018

Solvent: CDCl₃

Ambient temperature

Total 1440 repetitions



Compound 9d (¹H-NMR spectral data)

SH2FA1C13

Pulse Sequence: s2pu1

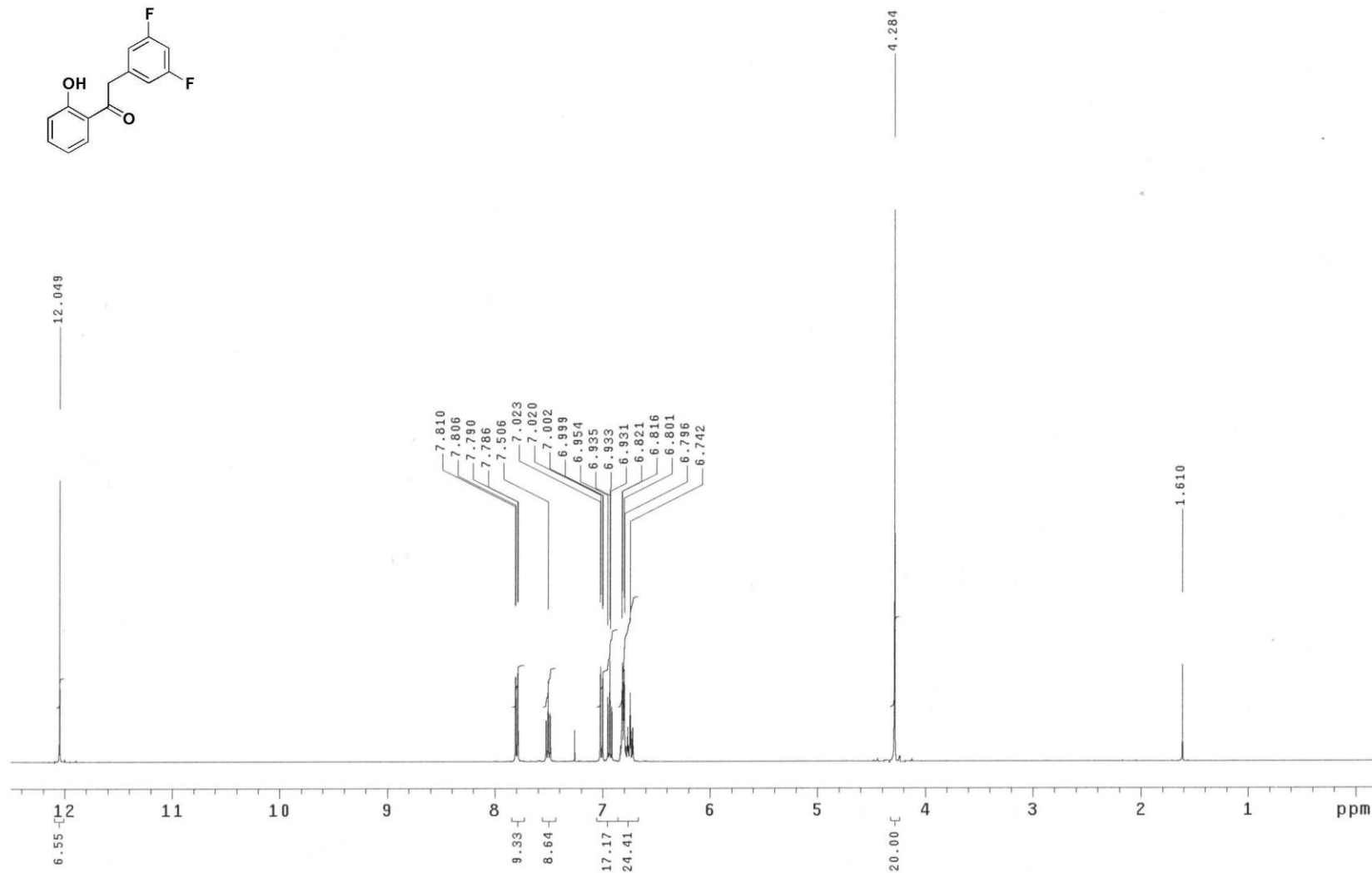
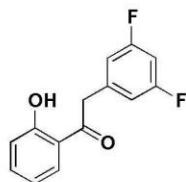
UNITYplus-400 "unity400"

Date: Aug 15 2018

Solvent: CDCl₃

Ambient temperature

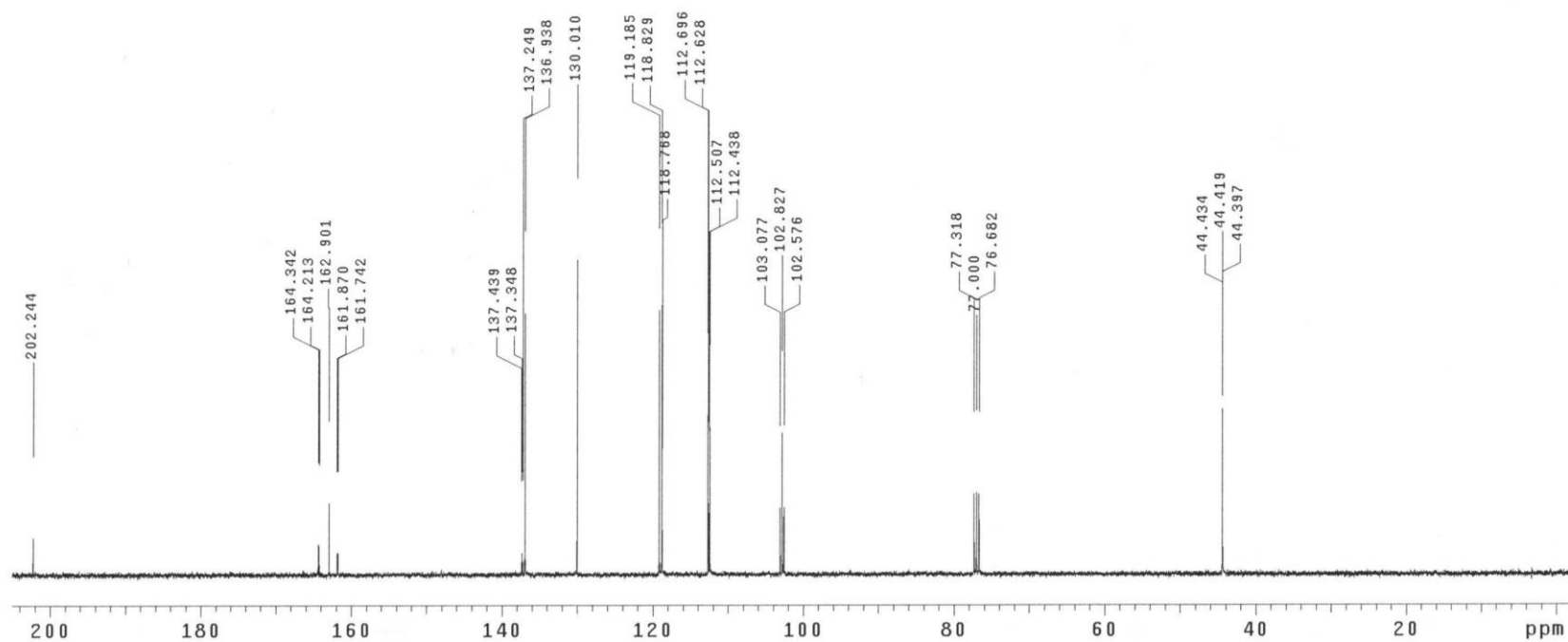
Total 32 repetitions



Compound 9d (¹³C-NMR spectral data)

SH2FA1C13

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Aug 15 2018
Solvent: CDCl₃
Ambient temperature
Total 1072 repetitions



Compound 9e (¹H-NMR spectral data)

SHN02A1C13

Pulse Sequence: s2pu1

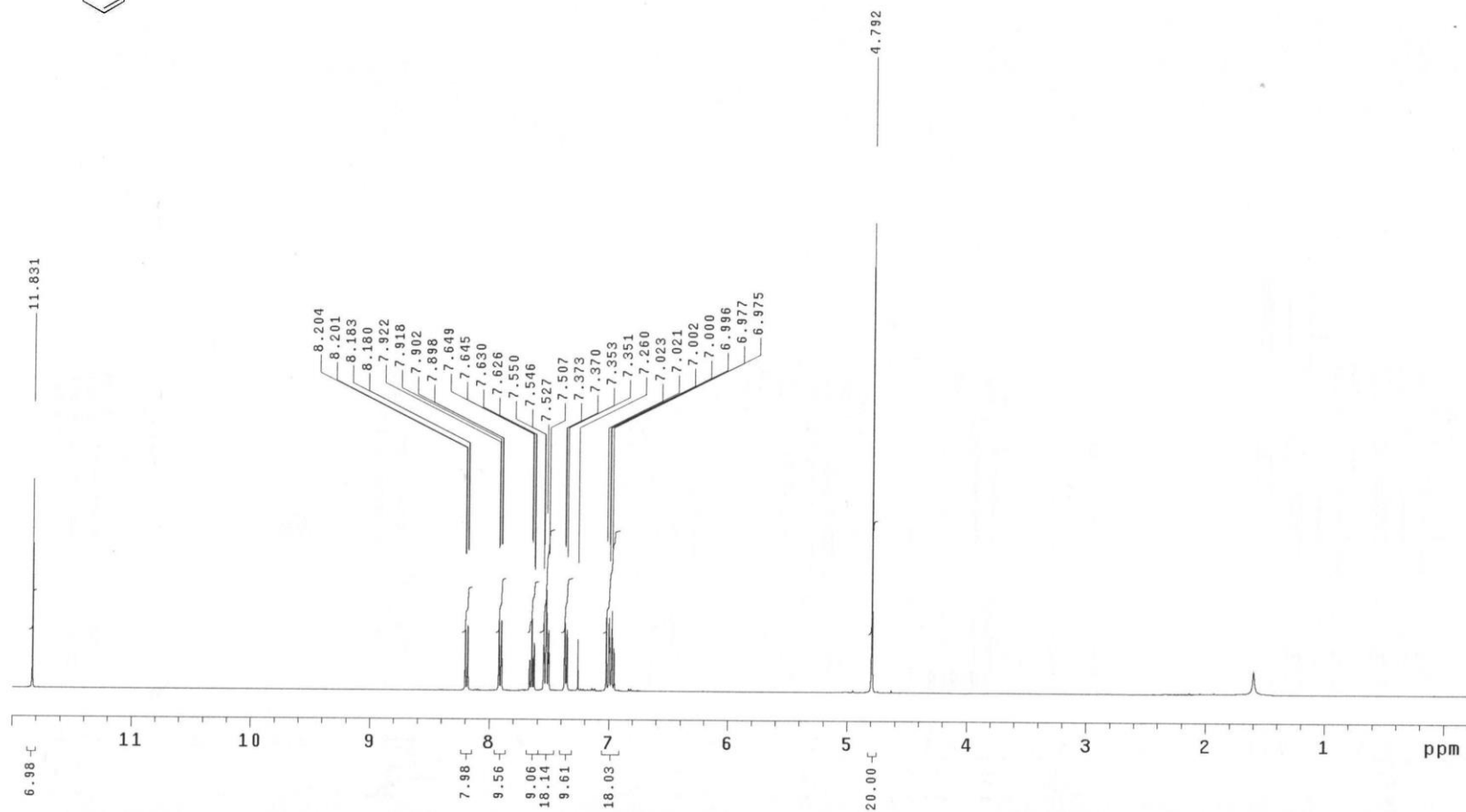
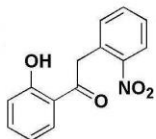
UNITYplus-400 "unity400"

Date: Aug 23 2018

Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 9e (¹³C-NMR spectral data)

SHN02A1C13

Pulse Sequence: s2pu1

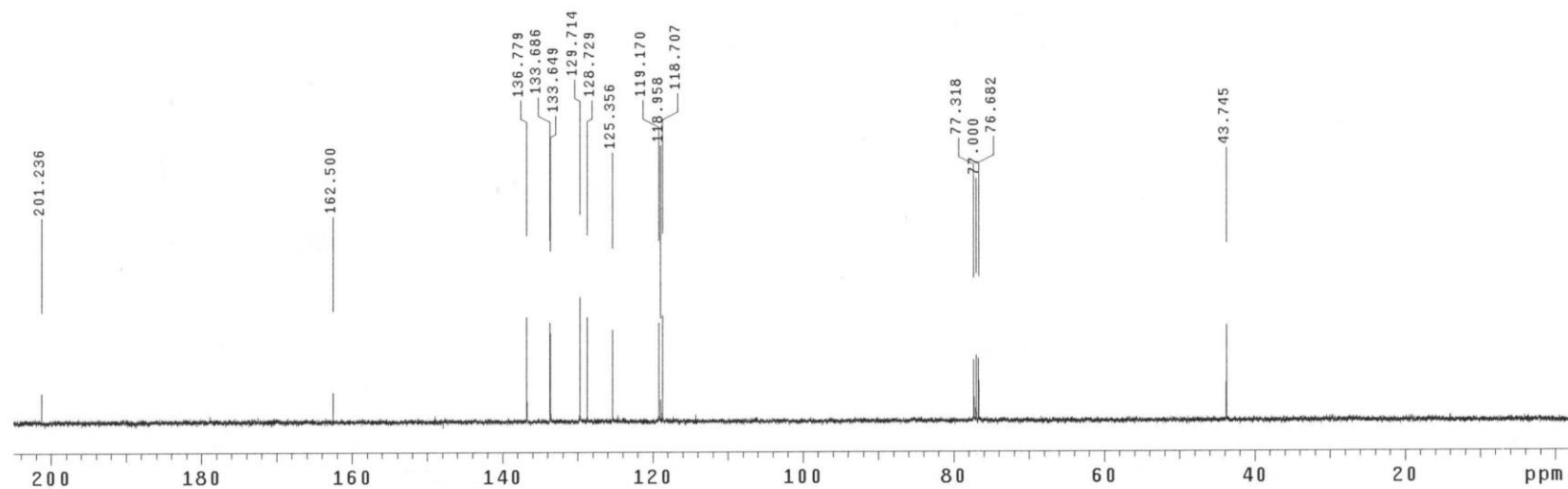
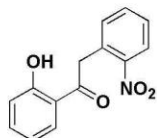
UNITYplus-400 "unity400"

Date: Aug 23 2018

Solvent: CDCl₃

Ambient temperature

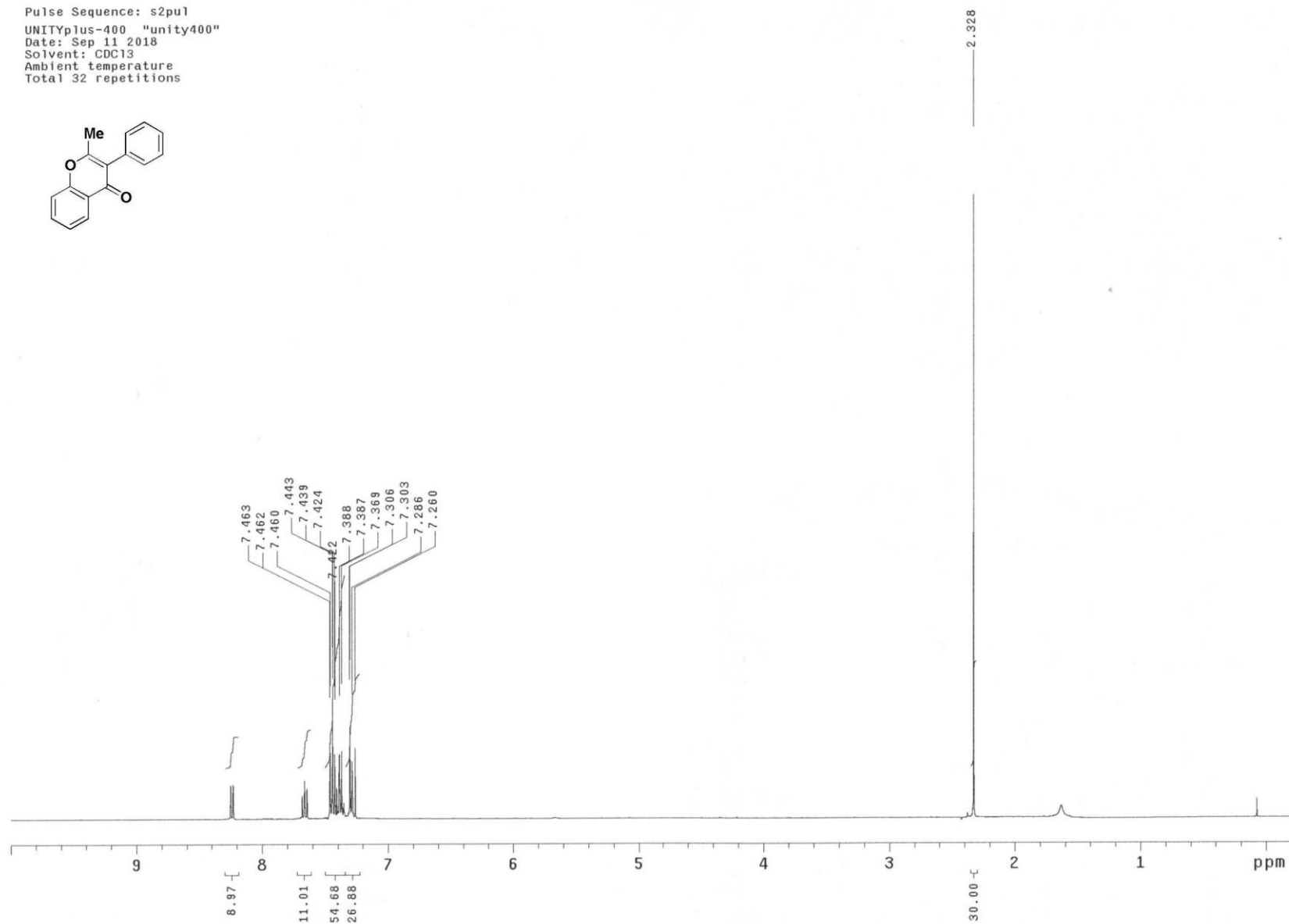
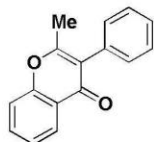
Total 480 repetitions



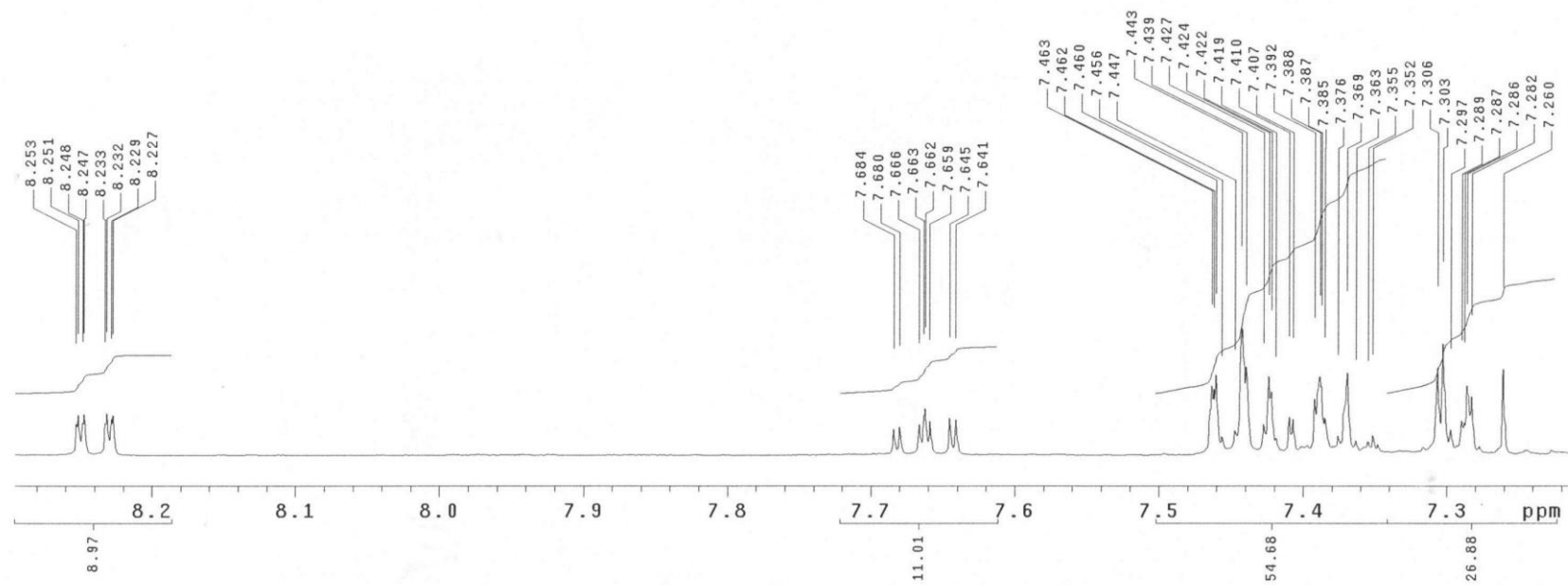
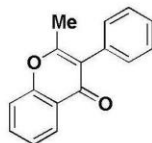
Compound 10a (¹H-NMR spectral data-1)

YTPHAC20

Pulse Sequence: s2pu1
UNITYplus-400 "unity400"
Date: Sep 11 2018
Solvent: CDCl₃
Ambient temperature
Total 32 repetitions



Compound 10a (¹H-NMR spectral data-2)



Compound 10a (¹³C-NMR spectral data)

YTPHAC20

Pulse Sequence: s2pul

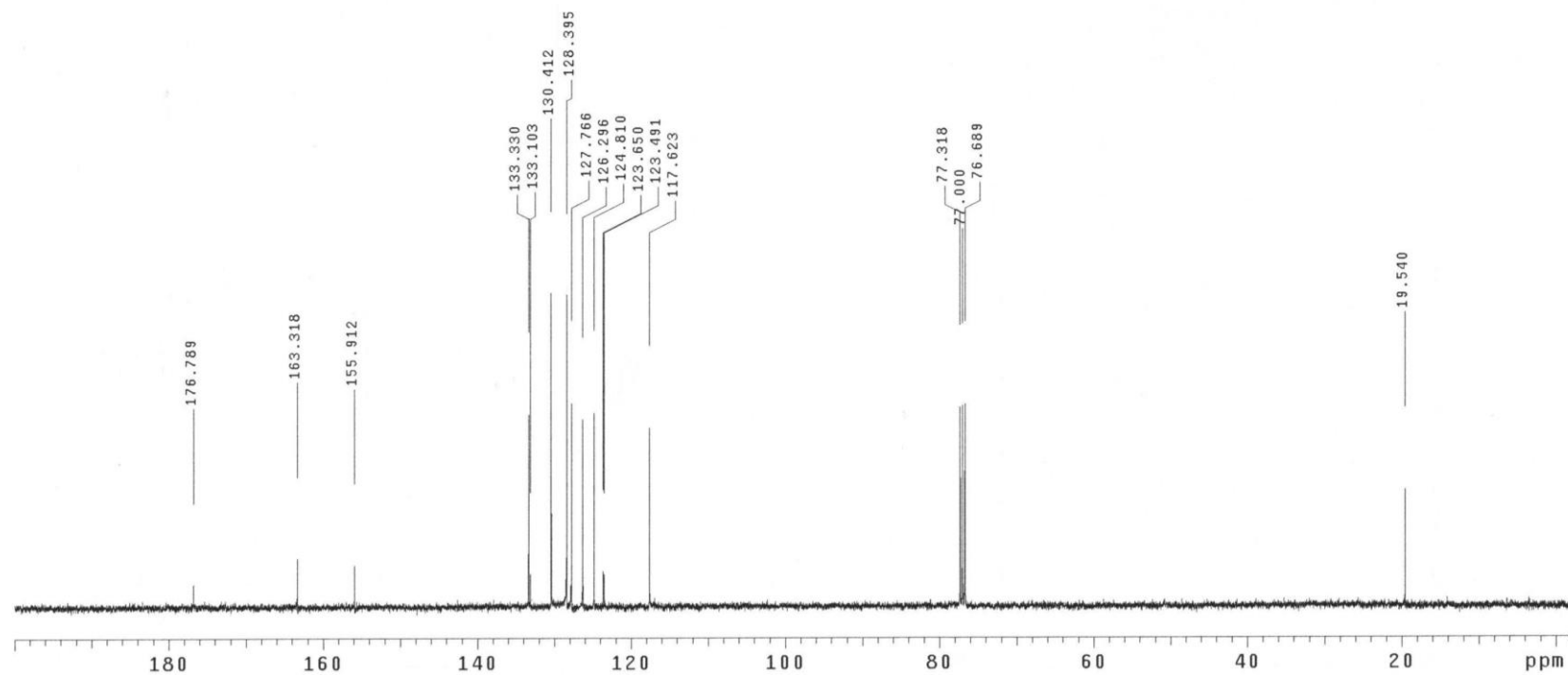
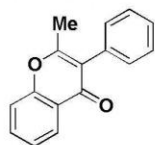
UNITYplus-400 "unity400"

Date: Sep 11 2018

Solvent: CDCl₃

Ambient temperature

Total 2848 repetitions



Compound 10b (¹H-NMR spectral data-1)

YTFAC20up

Pulse Sequence: s2pu1

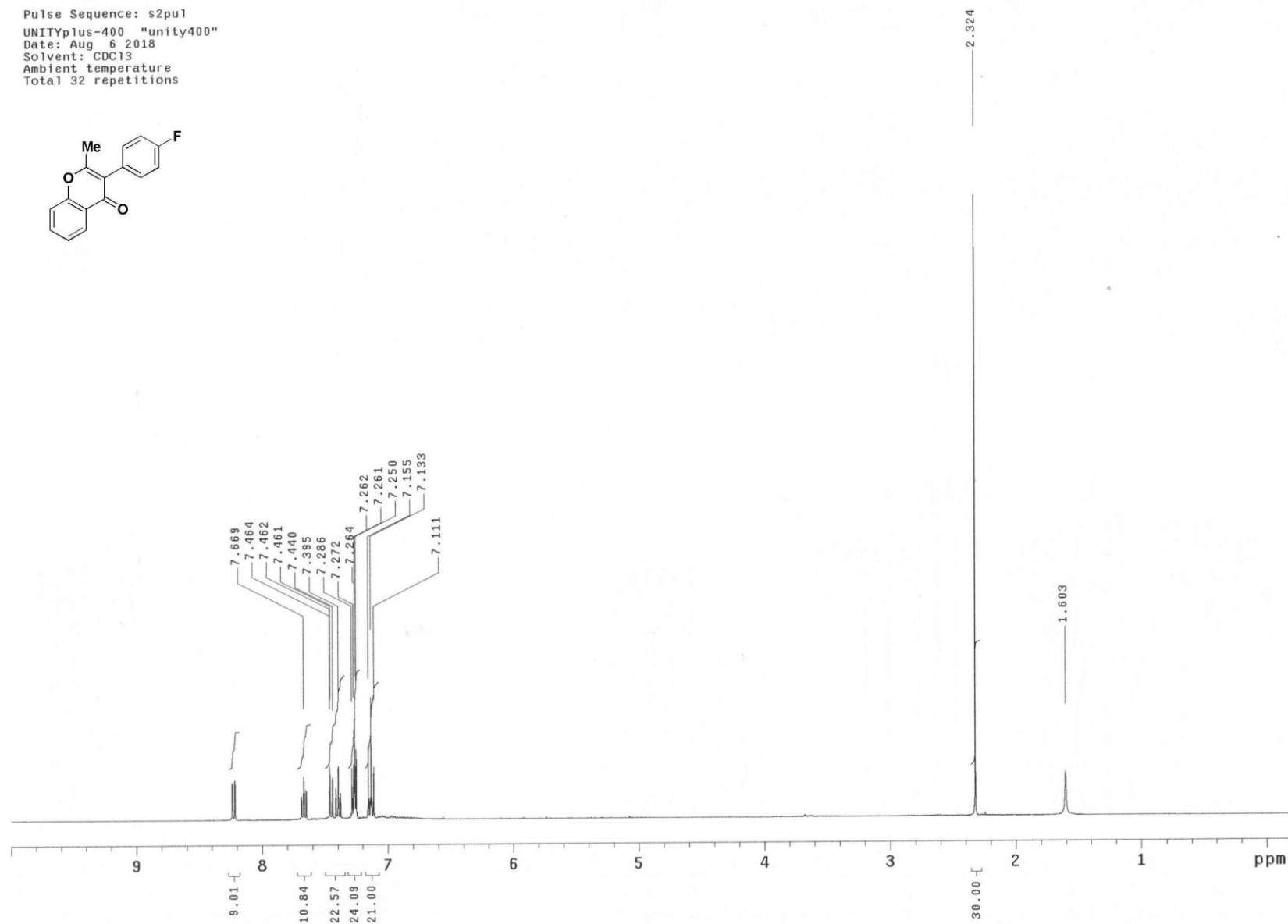
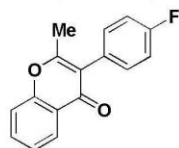
UNITYplus-400 "unity400"

Date: Aug 6 2018

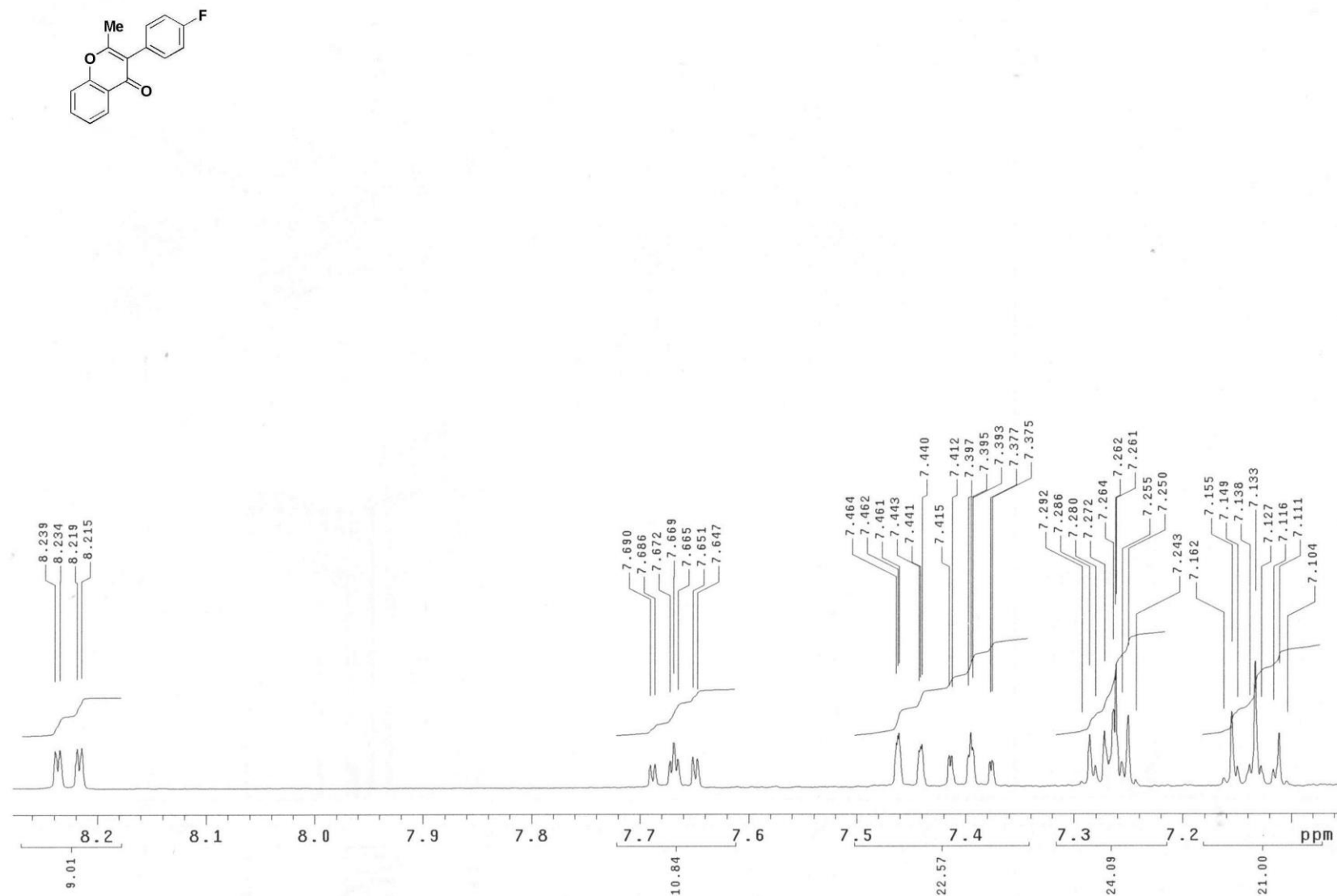
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 10b (¹H-NMR spectral data-2)



Compound 10b (¹³C-NMR spectral data)

YTFAC20up

Pulse Sequence: s2pu1

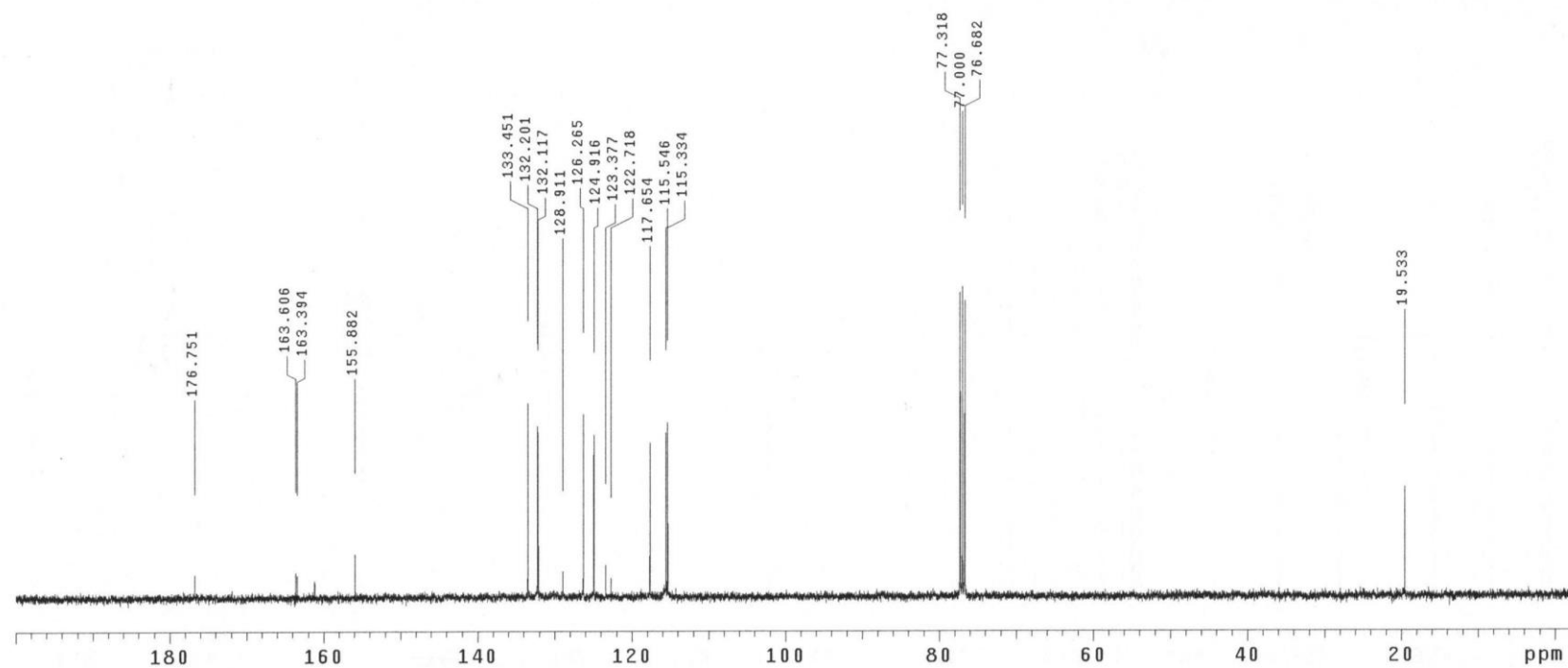
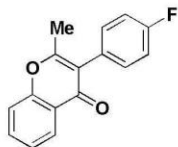
UNITYplus-400 "unity400"

Date: Aug 6 2018

Solvent: CDCl₃

Ambient temperature

Total 3504 repetitions



YTB i PhAC20

Pulse Sequence: s2pu1

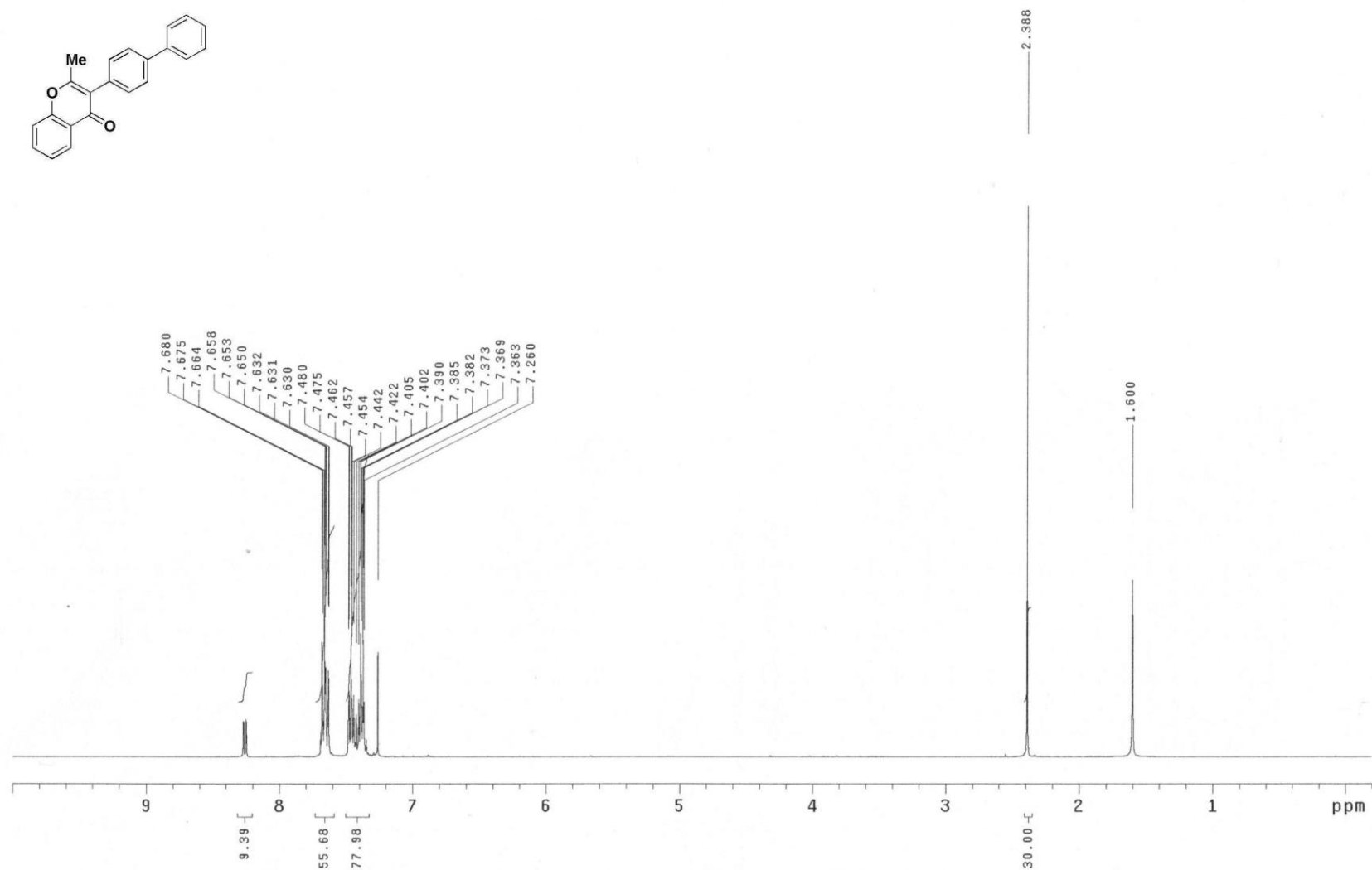
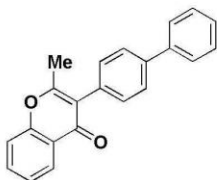
UNITYplus-400 "unity400"

Date: Sep 13 2018

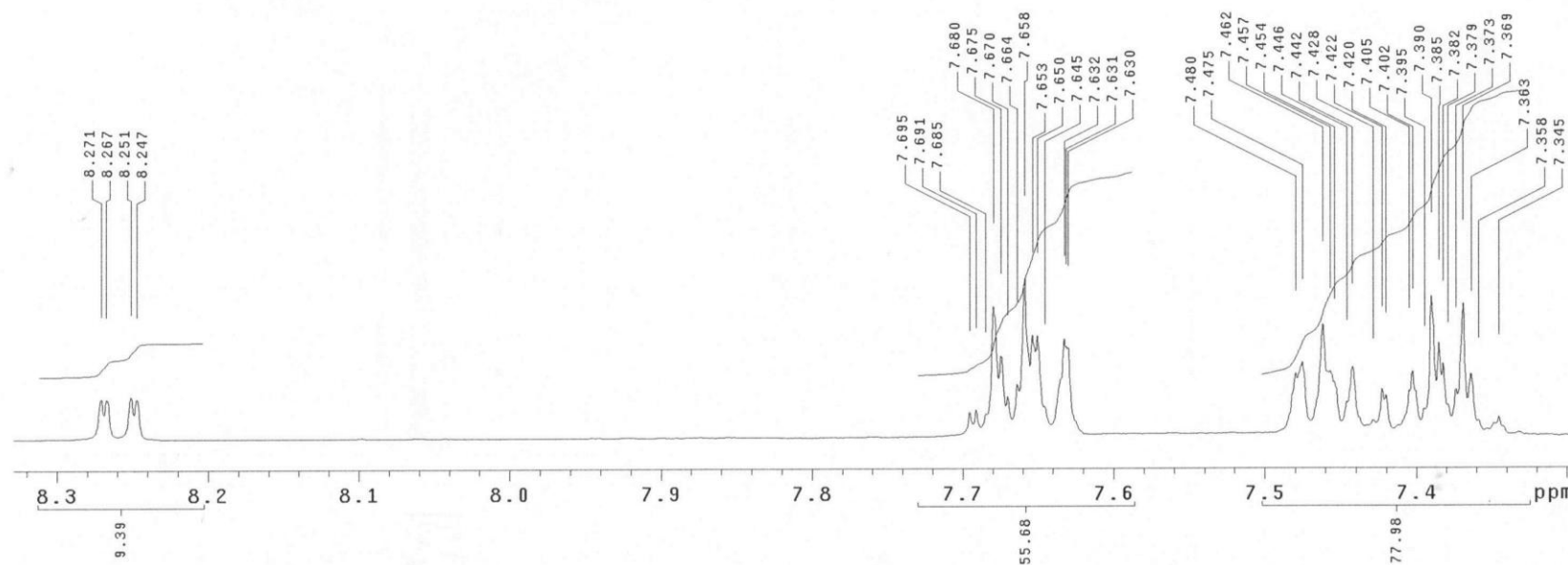
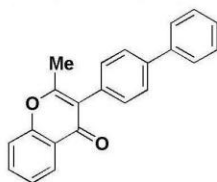
Solvent: CDC13

Ambient temperature

Total 32 repetitions



Compound 10c (¹H-NMR spectral data-2)



Compound 10c (^{13}C -NMR spectral data)

YTBiPhAC20

Pulse Sequence: s2pu1

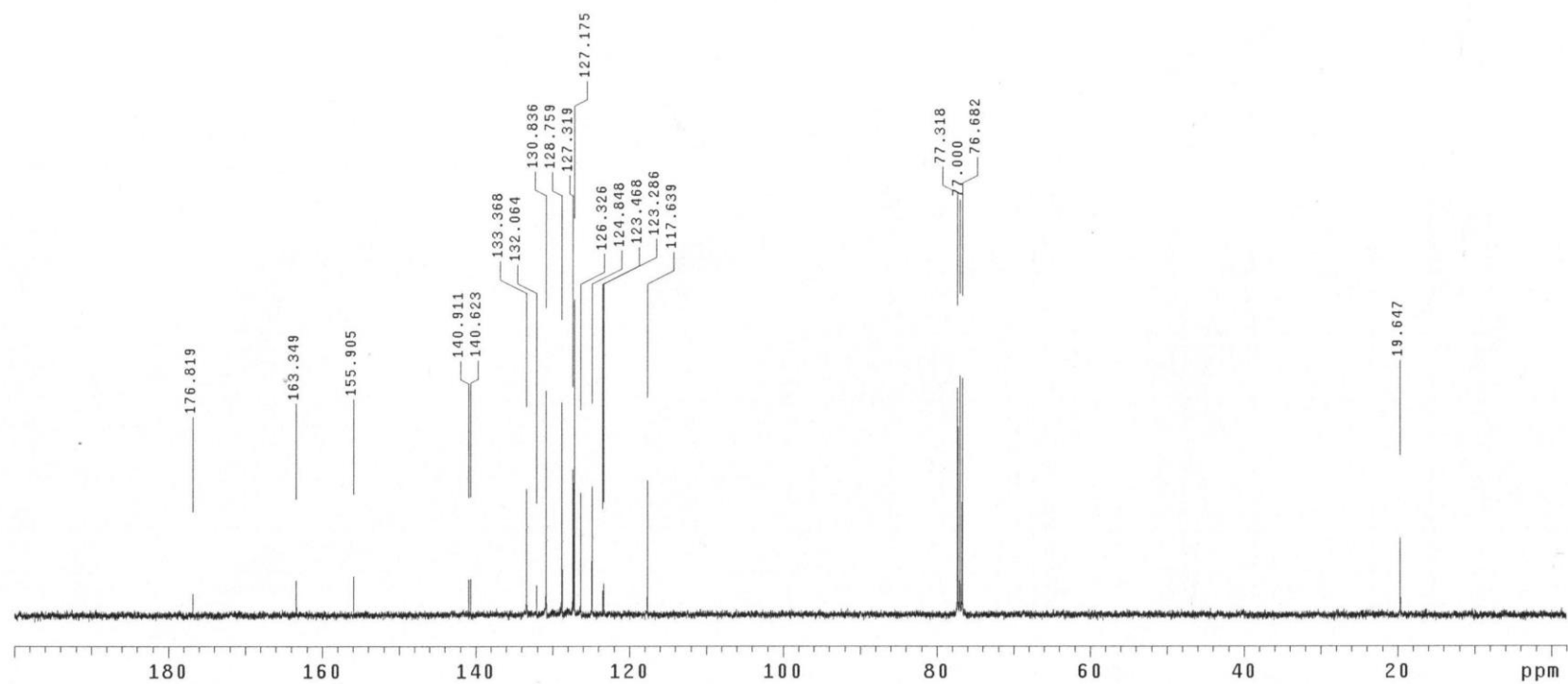
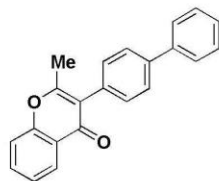
UNITYplus-400 "unity400"

Date: Sep 13 2018

Solvent: CDCl₃

Ambient temperature

Total 4288 repetitions



Compound 12 (¹H-NMR spectral data-1)

3H2-NAPh

Pulse Sequence: s2pu1

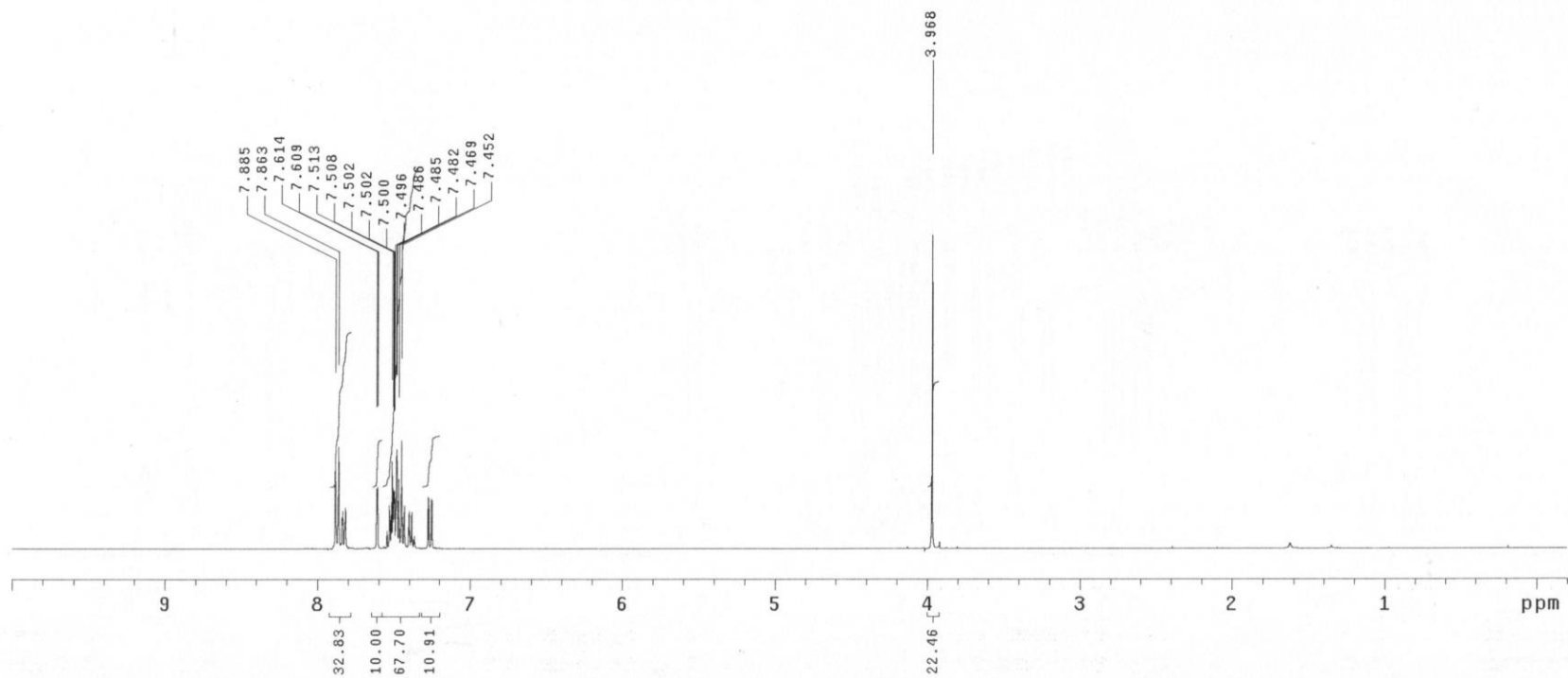
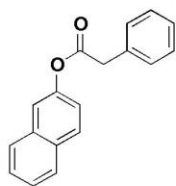
UNITYplus-400 "unity400"

Date: Jun 24 2019

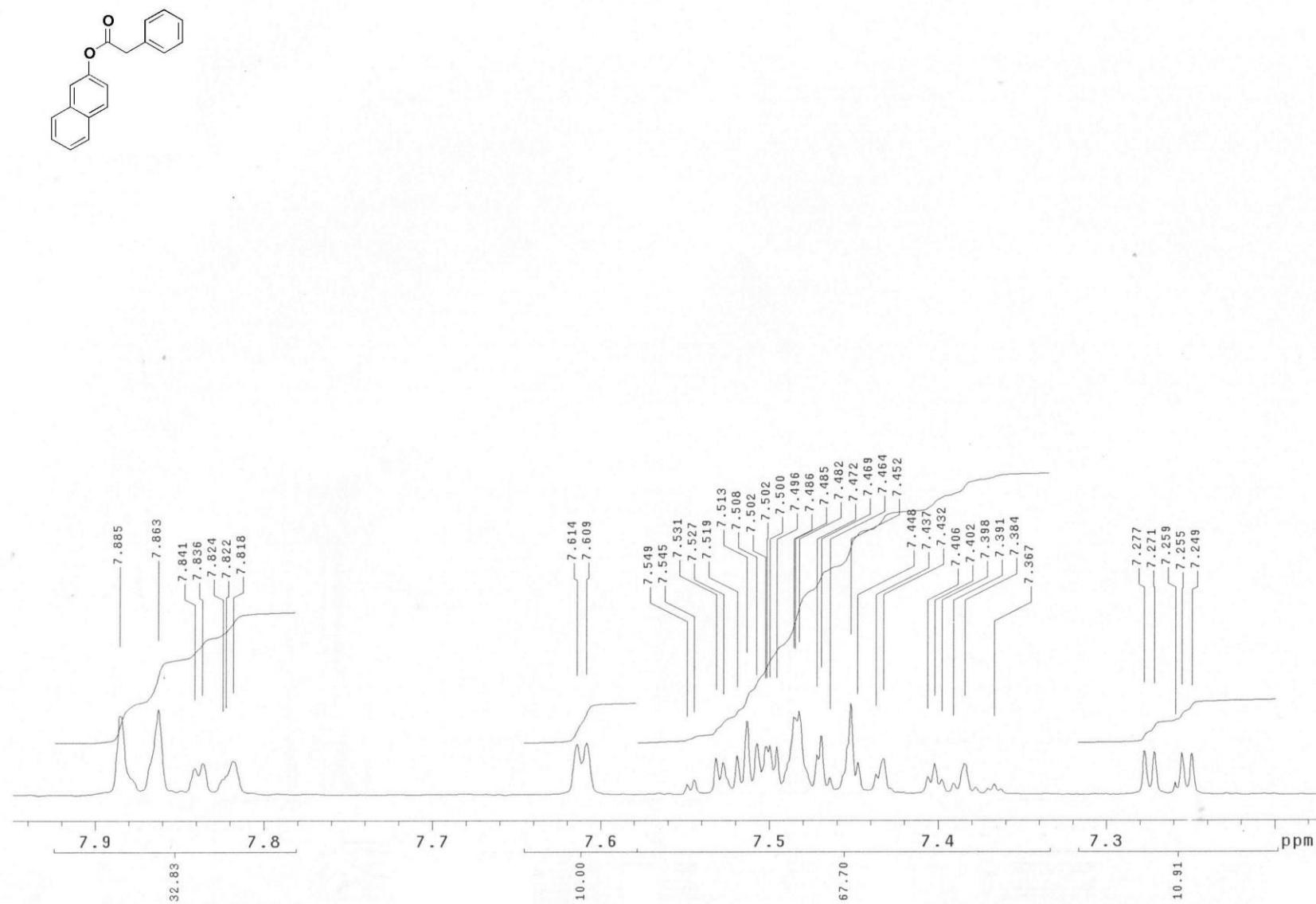
Solvent: CDCl₃

Ambient temperature

Total 32 repetitions



Compound 12 (¹H-NMR spectral data-2)



Compound 12 (¹³C-NMR spectral data)

3H2-NAPh

Pulse Sequence: s2pul

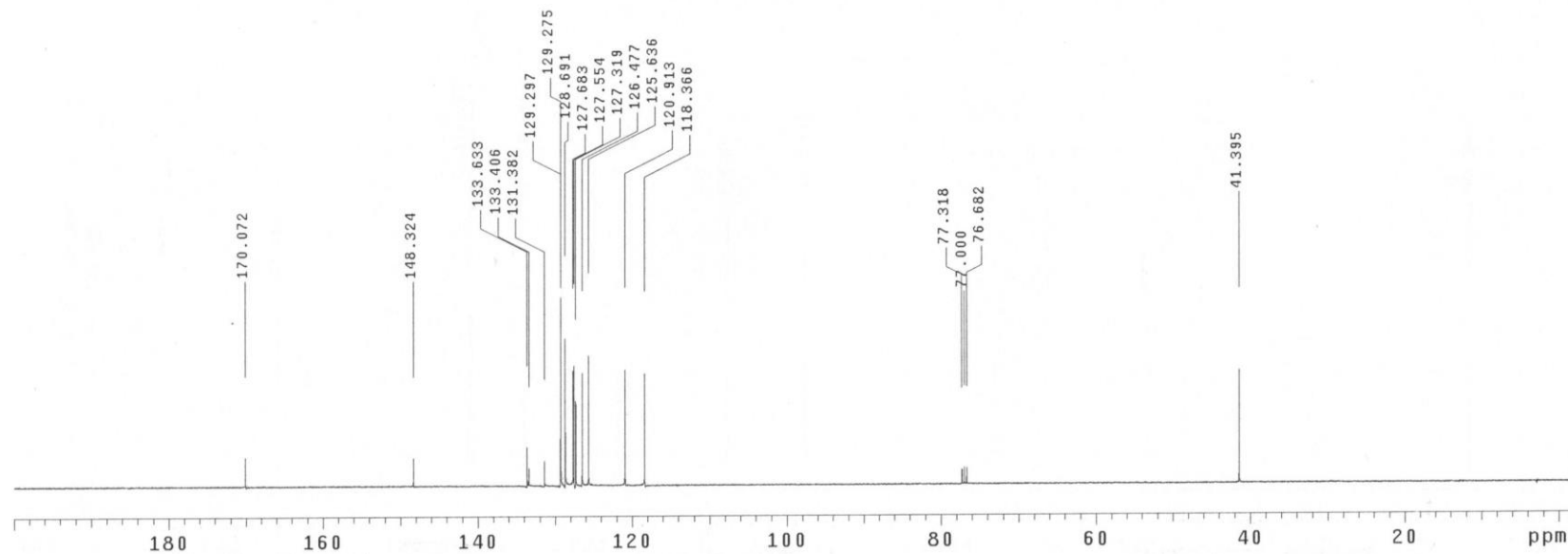
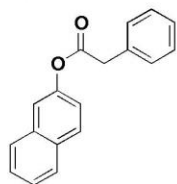
UNITYplus-400 "unity400"

Date: Jun 24 2019

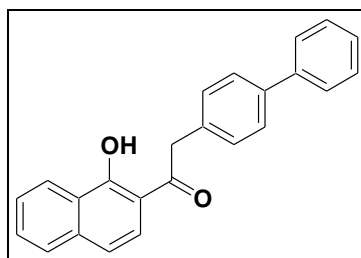
Solvent: CDCl₃

Ambient temperature

Total 1024 repetitions

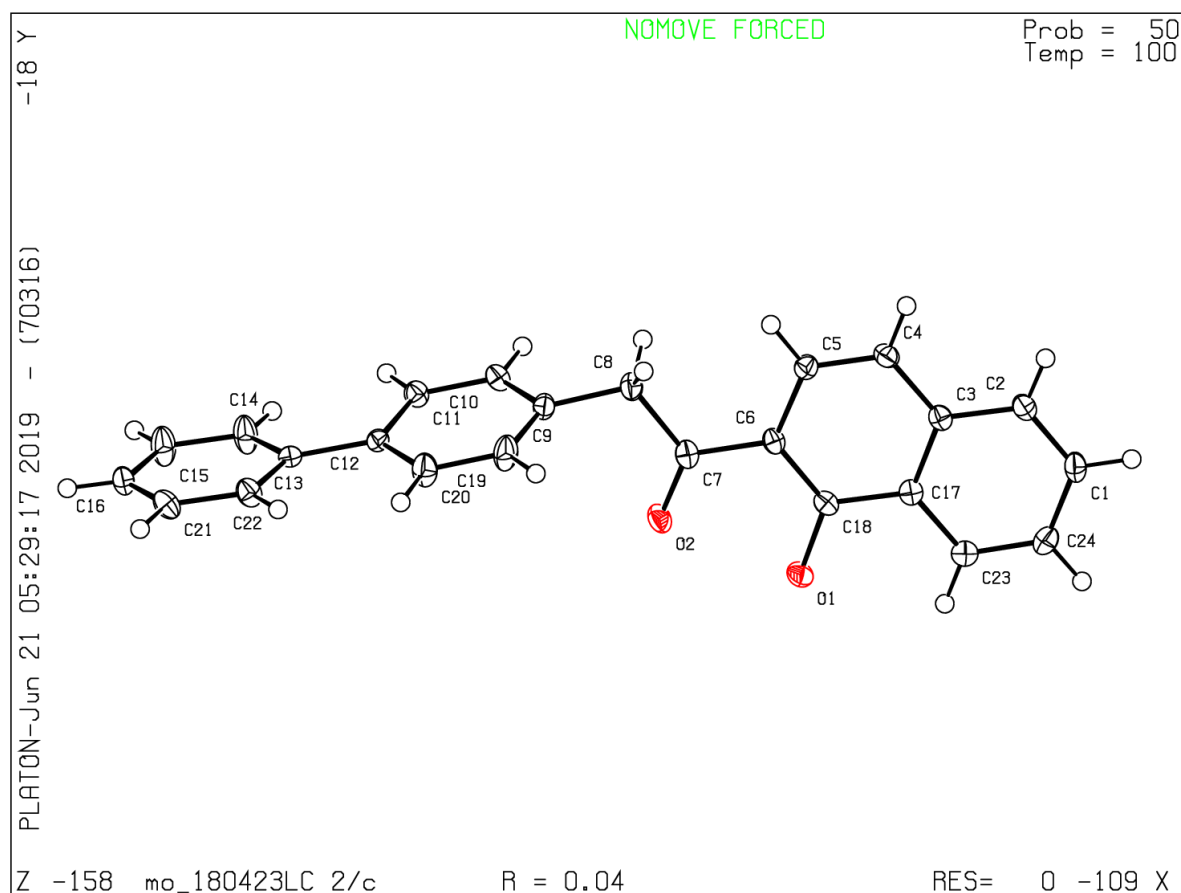


X-ray crystal data of compound 4e (CCDC 1894542)



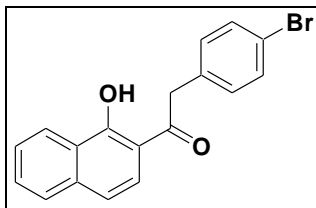
Sample preparation : A solution of compound **4e** (30 mg) in warm CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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



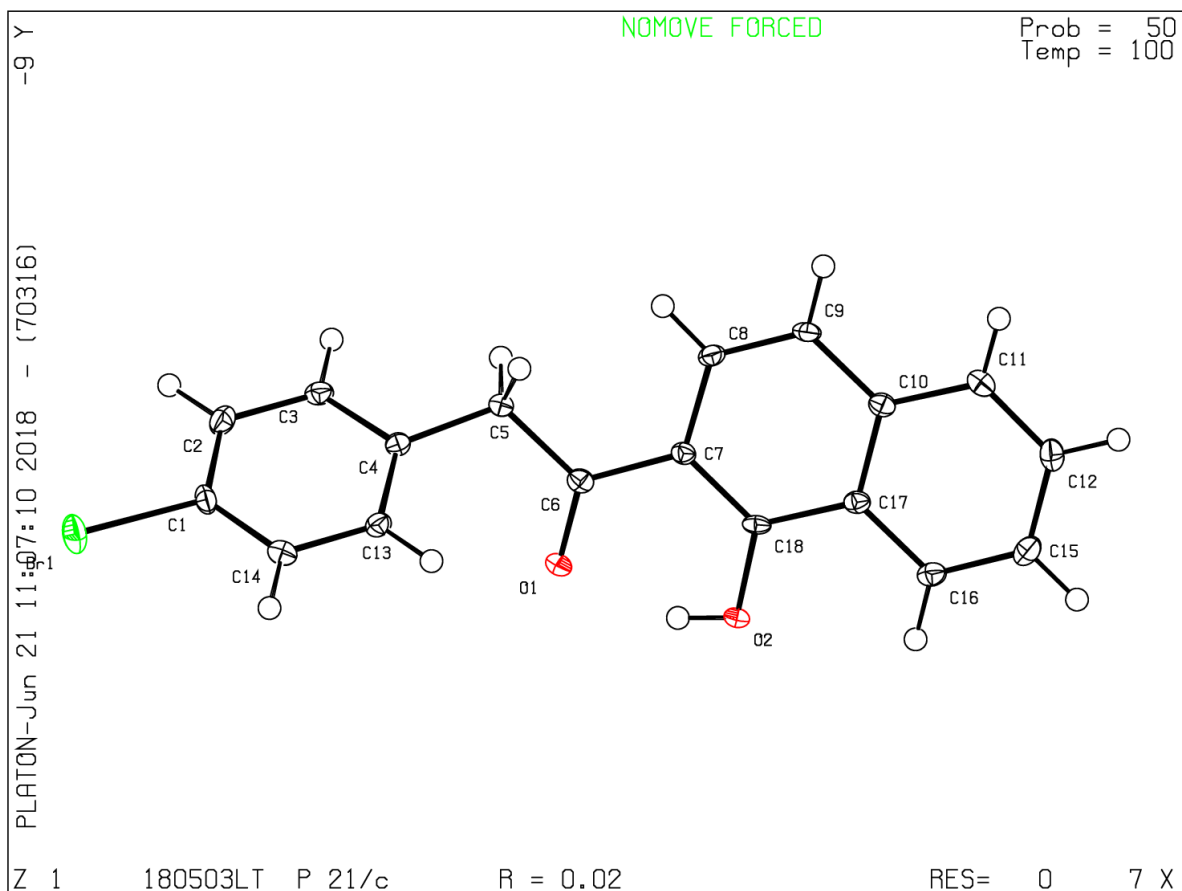
| | |
|-----------------------------------|--|
| Empirical formula | C ₂₄ H ₁₇ O ₂ |
| Formula weight | 337.37 |
| Temperature | 100(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | C2/c |
| Unit cell dimensions | a = 25.037(5) Å α = 90°. b = 8.0888(15) Å β = 118.618(5)°. c = 19.202(3) Å γ = 90°. |
| Volume | 3413.6(11) Å ³ |
| Z | 8 |
| Density (calculated) | 1.313 Mg/m ³ |
| Absorption coefficient | 0.082 mm ⁻¹ |
| F(000) | 1416 |
| Crystal size | 0.25 x 0.23 x 0.19 mm ³ |
| Theta range for data collection | 1.853 to 26.536°. |
| Index ranges | -31 ≤ h ≤ 31, -10 ≤ k ≤ 10, -21 ≤ l ≤ 24 |
| Reflections collected | 24471 |
| Independent reflections | 3514 [R(int) = 0.0306] |
| Completeness to theta = 25.242° | 99.8 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.9485 and 0.9108 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 3514 / 0 / 235 |
| Goodness-of-fit on F ² | 1.038 |
| Final R indices [I > 2σ(I)] | R1 = 0.0430, wR2 = 0.1219 |
| R indices (all data) | R1 = 0.0536, wR2 = 0.1327 |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 0.717 and -0.220 e.Å ⁻³ |

X-ray crystal data of compound 4h (CCDC 1894541)



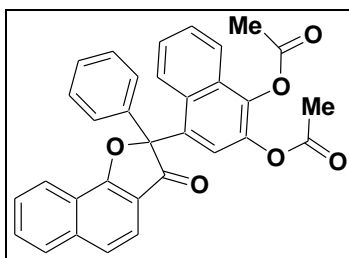
Sample preparation : A solution of compound **4h** (30 mg) in warm CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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



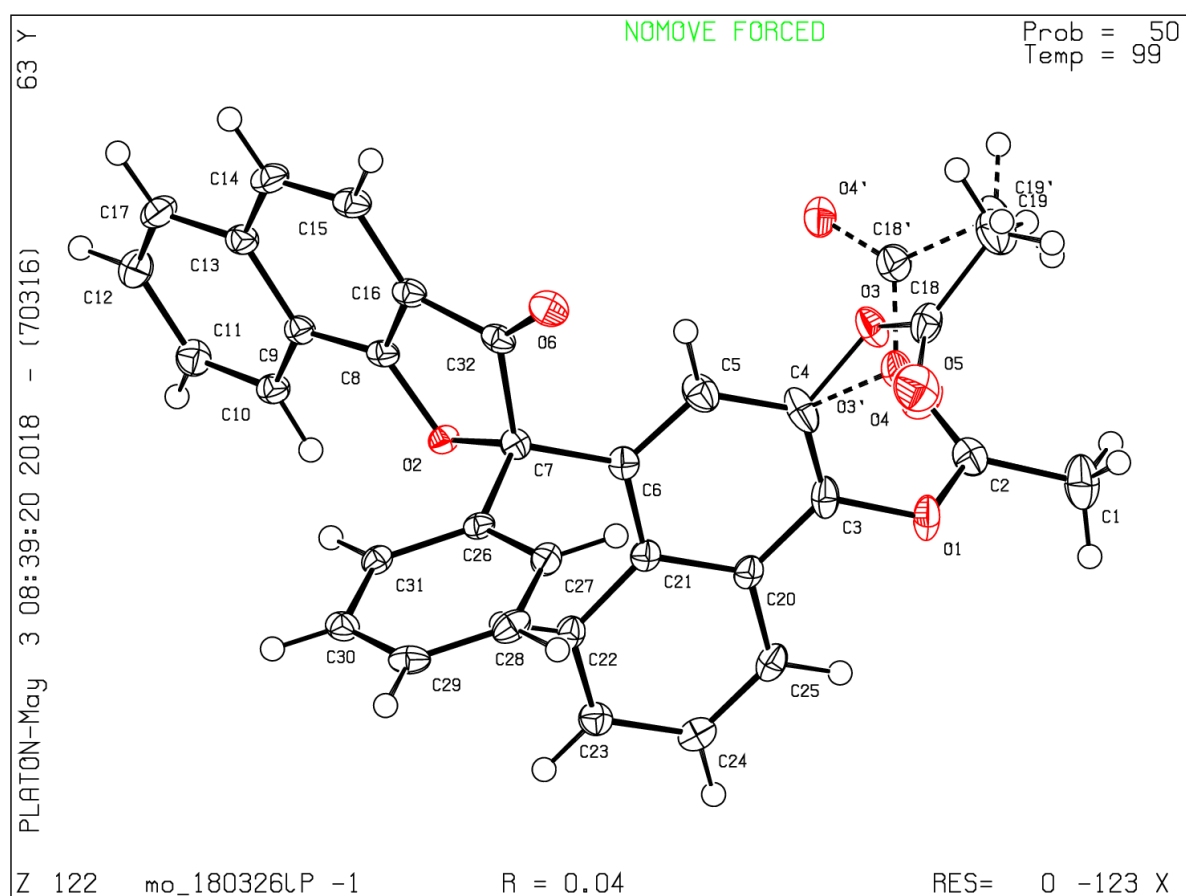
| | |
|-----------------------------------|---|
| Empirical formula | C ₁₈ H ₁₃ Br O ₂ |
| Formula weight | 341.19 |
| Temperature | 100(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | P 21/c |
| Unit cell dimensions | a = 16.3545(7) Å α = 90°. b = 11.9821(5) Å β = 100.2100(10)°. c = 7.3657(3) Å γ = 90°. |
| Volume | 1420.53(10) Å ³ |
| Z | 4 |
| Density (calculated) | 1.595 Mg/m ³ |
| Absorption coefficient | 2.895 mm ⁻¹ |
| F(000) | 688 |
| Crystal size | 0.15 x 0.13 x 0.12 mm ³ |
| Theta range for data collection | 1.265 to 26.419°. |
| Index ranges | -20 ≤ h ≤ 20, -14 ≤ k ≤ 14, -8 ≤ l ≤ 9 |
| Reflections collected | 24966 |
| Independent reflections | 2924 [R(int) = 0.0294] |
| Completeness to theta = 25.242° | 100.0 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.9485 and 0.8032 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 2924 / 0 / 193 |
| Goodness-of-fit on F ² | 1.077 |
| Final R indices [I > 2σ(I)] | R1 = 0.0231, wR2 = 0.0592 |
| R indices (all data) | R1 = 0.0284, wR2 = 0.0632 |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 0.462 and -0.434 e.Å ⁻³ |

X-ray crystal data of compound 5a (CCDC 1894548)



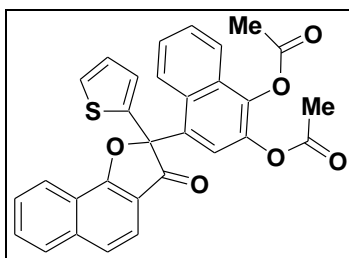
Sample preparation : A solution of compound **5a** (30 mg) in warm CH₂Cl₂ (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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



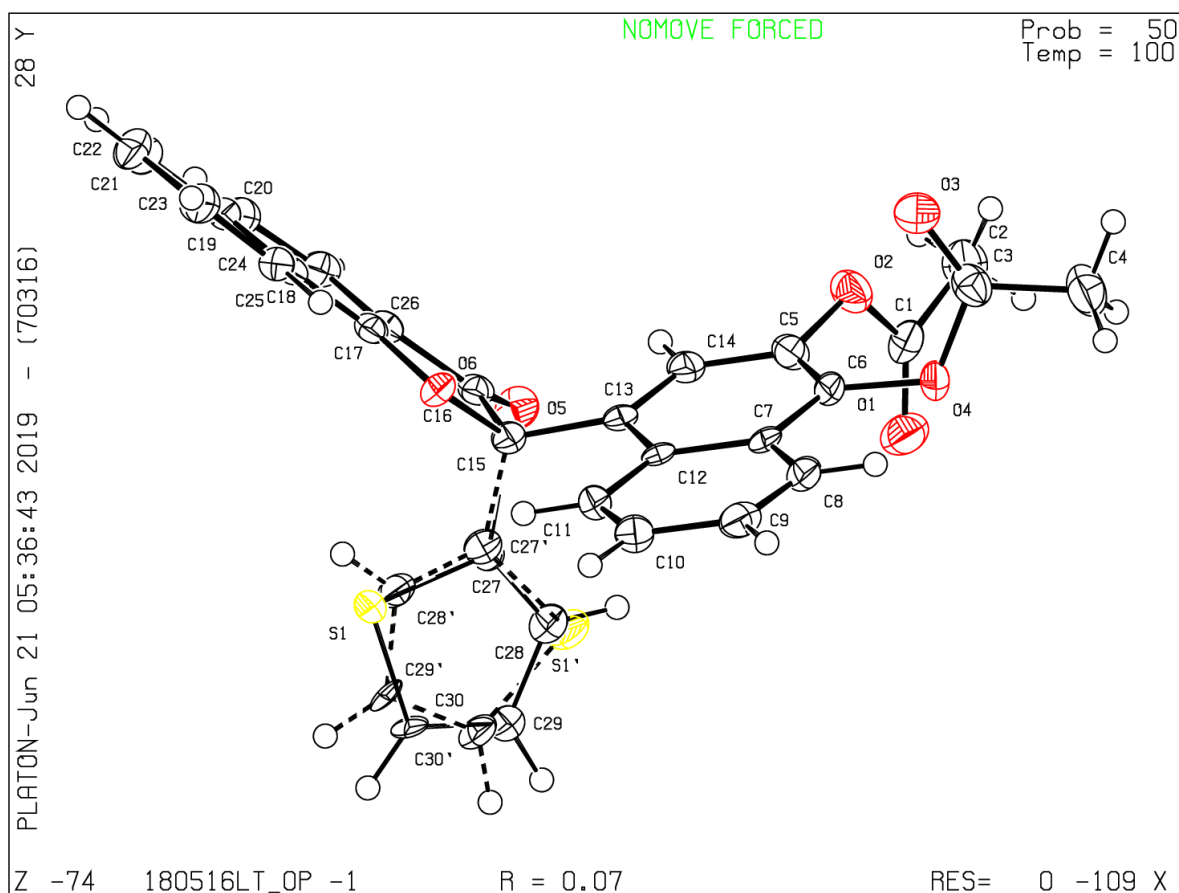
| | |
|-----------------------------------|--|
| Empirical formula | C ₃₂ H ₂₂ O ₆ |
| Formula weight | 502.49 |
| Temperature | 99(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Triclinic |
| Space group | P -1 |
| Unit cell dimensions | a = 8.1280(4) Å α = 84.715(2)°. b = 10.6123(6) Å β = 79.253(2)°. c = 14.7375(8) Å γ = 75.963(2)°. |
| Volume | 1210.15(11) Å ³ |
| Z | 2 |
| Density (calculated) | 1.379 Mg/m ³ |
| Absorption coefficient | 0.095 mm ⁻¹ |
| F(000) | 524 |
| Crystal size | 0.28 x 0.25 x 0.23 mm ³ |
| Theta range for data collection | 1.408 to 26.527°. |
| Index ranges | -10 ≤ h ≤ 9, -13 ≤ k ≤ 13, -18 ≤ l ≤ 18 |
| Reflections collected | 29726 |
| Independent reflections | 5023 [R(int) = 0.0436] |
| Completeness to theta = 25.242° | 100.0 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.9485 and 0.8979 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 5023 / 108 / 383 |
| Goodness-of-fit on F ² | 1.041 |
| Final R indices [I > 2σ(I)] | R1 = 0.0438, wR2 = 0.0998 |
| R indices (all data) | R1 = 0.0577, wR2 = 0.1079 |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 0.380 and -0.376 e.Å ⁻³ |

X-ray crystal data of compound **5g** (CCDC 1894543)



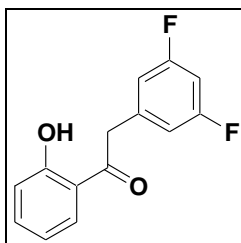
Sample preparation : A solution of compound **5g** (30 mg) in warm CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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



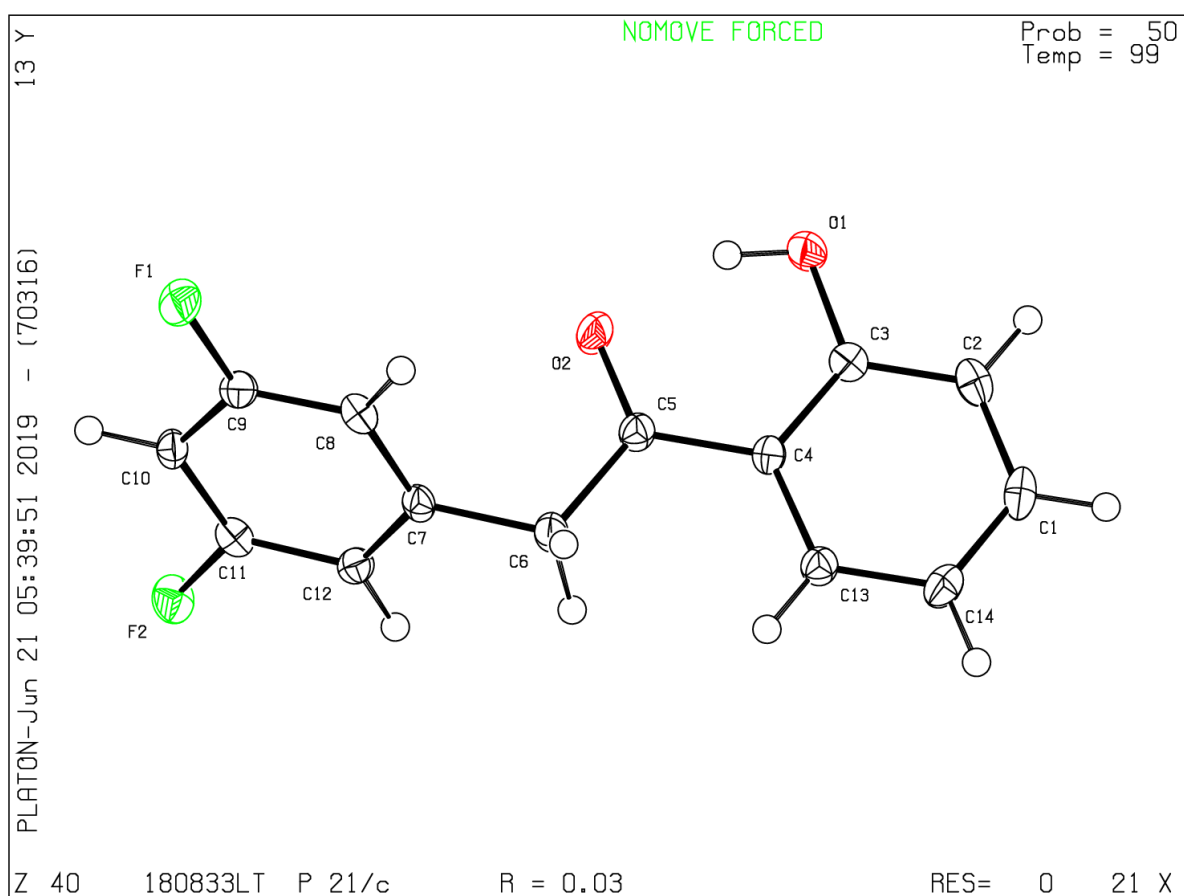
| | |
|-----------------------------------|---|
| Empirical formula | C ₃₀ H ₂₀ O ₆ S |
| Formula weight | 508.52 |
| Temperature | 100(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Triclinic |
| Space group | P-1 |
| Unit cell dimensions | a = 7.9733(10) Å α = 84.455(7)°. b = 10.7024(14) Å β = 80.196(6)°. c = 14.7112(19) Å γ = 74.507(6)°. |
| Volume | 1190.3(3) Å ³ |
| Z | 2 |
| Density (calculated) | 1.419 Mg/m ³ |
| Absorption coefficient | 0.182 mm ⁻¹ |
| F(000) | 528 |
| Crystal size | 0.26 x 0.25 x 0.23 mm ³ |
| Theta range for data collection | 1.407 to 26.569°. |
| Index ranges | -9 ≤ h ≤ 9, -13 ≤ k ≤ 13, -18 ≤ l ≤ 18 |
| Reflections collected | 17883 |
| Independent reflections | 4778 [R(int) = 0.0437] |
| Completeness to theta = 25.242° | 98.0 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.9485 and 0.8566 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 4778 / 549 / 382 |
| Goodness-of-fit on F ² | 1.115 |
| Final R indices [I > 2σ(I)] | R1 = 0.0715, wR2 = 0.1944 |
| R indices (all data) | R1 = 0.0953, wR2 = 0.2046 |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 0.671 and -0.375 e.Å ⁻³ |

X-ray crystal data of compound 9d (CCDC 1894544)



Sample preparation : A solution of compound **9d** (30 mg) in warm CH_2Cl_2 (10 mL) was placed in a tube (10 mL). EtOAc (2 mL) was added slowly to the vial with a dropper. The vial was closed with little cotton and kept at room temperature for 2 days. Then, colorless prisms were observed.

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



| | |
|-----------------------------------|--|
| Empirical formula | C ₁₄ H ₁₀ F ₂ O ₂ |
| Formula weight | 248.22 |
| Temperature | 99(2) K |
| Wavelength | 0.71073 Å |
| Crystal system | Monoclinic |
| Space group | P2 ₁ /c |
| Unit cell dimensions | a = 7.9744(3) Å α = 90°. b = 20.0084(6) Å β = 103.779(2)°. c = 7.1794(2) Å γ = 90°. |
| Volume | 1112.54(6) Å ³ |
| Z | 4 |
| Density (calculated) | 1.482 Mg/m ³ |
| Absorption coefficient | 0.121 mm ⁻¹ |
| F(000) | 512 |
| Crystal size | 0.20 x 0.19 x 0.08 mm ³ |
| Theta range for data collection | 2.036 to 26.406°. |
| Index ranges | -9 ≤ h ≤ 9, -24 ≤ k ≤ 25, -8 ≤ l ≤ 8 |
| Reflections collected | 15302 |
| Independent reflections | 2259 [R(int) = 0.0199] |
| Completeness to theta = 25.242° | 99.5 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.7454 and 0.7176 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 2259 / 0 / 166 |
| Goodness-of-fit on F ² | 1.052 |
| Final R indices [I > 2σ(I)] | R ₁ = 0.0323, wR ₂ = 0.0800 |
| R indices (all data) | R ₁ = 0.0356, wR ₂ = 0.0822 |
| Extinction coefficient | n/a |
| Largest diff. peak and hole | 0.261 and -0.175 e.Å ⁻³ |