

Supporting materials

Improved Synthesis of Asymmetric Curcuminoids and their Assessment as Antioxidants

Yang-Je Cheng ¹, Cai-Wei Li ², Cing-Ling Kuo ¹, Tzenge-Lien Shih ^{1,*}, and Jih-Jung Chen ^{3,4,*}

¹ Department of Chemistry, Tamkang University, Tamsui Dist., New Taipei City 251301, Taiwan, Republic of China; jason0110422@gmail.com (Y.-J.C.); rose40635@gmail.com (C.-L.K.)

² Institute of Traditional Medicine, National Yang Ming Chiao Tung University, Taipei 112304, Taiwan; leecw1219.y@nycu.edu.tw (C.-W.L.)

³ Department of Pharmacy, College of Pharmaceutical Sciences, National Yang Ming Chiao Tung University, Taipei 112304, Taiwan

⁴ Department of Medical Research, China Medical University Hospital, China Medical University, Taichung 404332, Taiwan

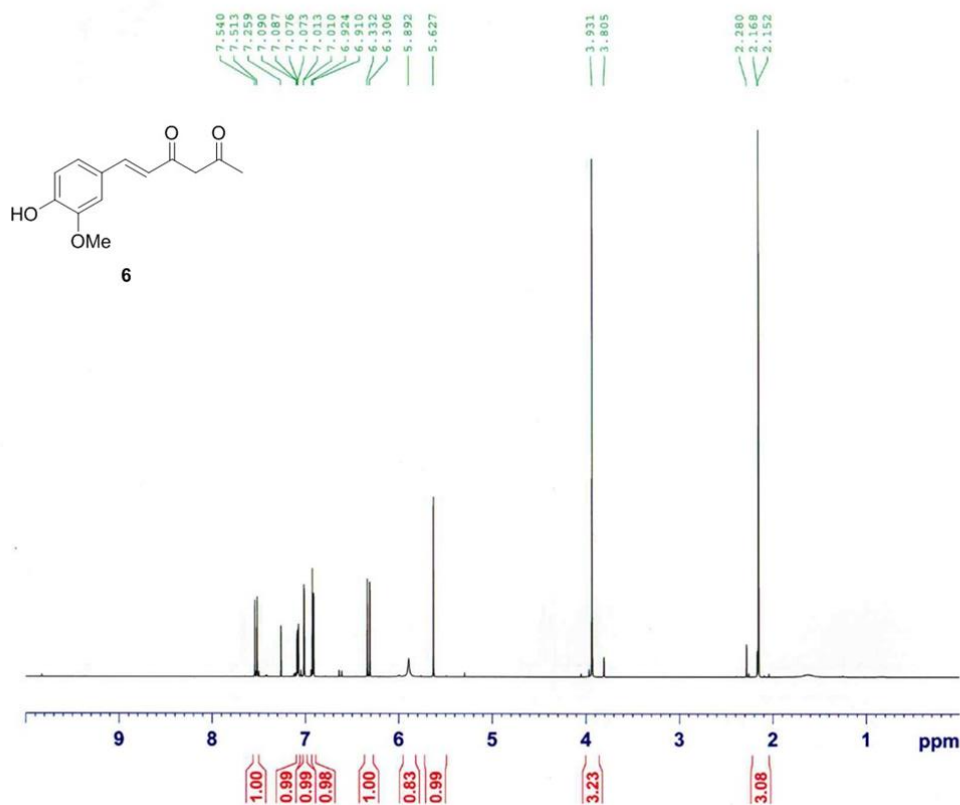
* Correspondence: tlshih@mail.tku.edu.tw (T.-L.S.)*; jjungchen@nycu.edu.tw (J.-J.C.)*; Tel.: +886-2-2826-7195 (J.-J.C.)*; Fax: +886-2-2823-2940 (J.-J.C.)*.

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¹H of CLK1-82up

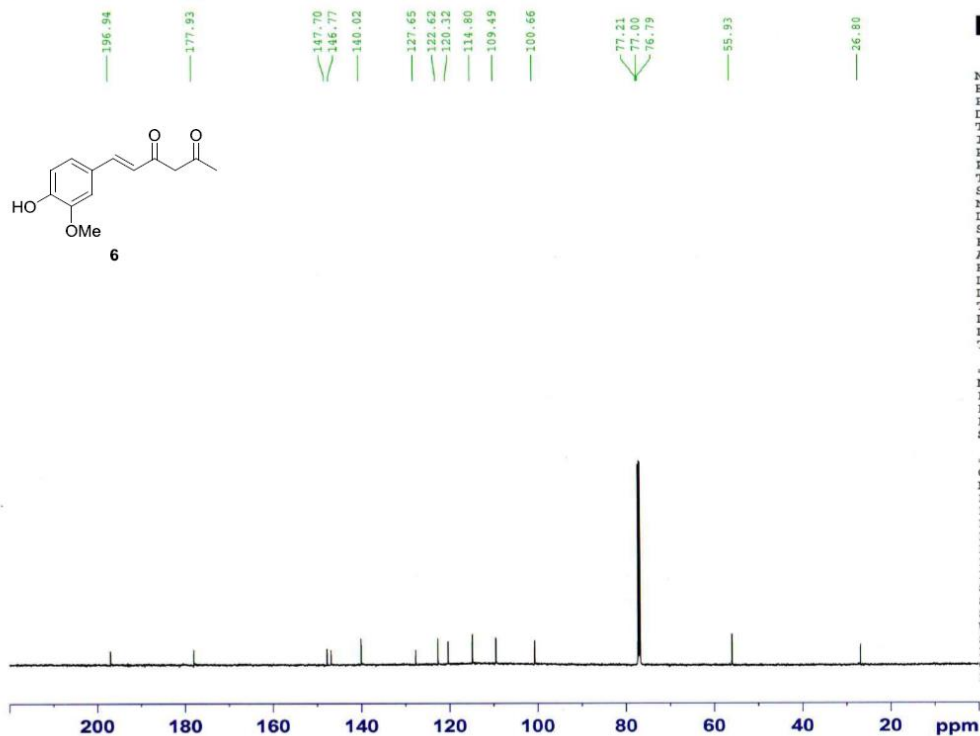


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PROCNO 1
Date_ 20200904
Time 11.12
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PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 7788.162 Hz
FIDRES 0.237676 Hz
AQ 2.1038198 sec
RG 406
DW 64.200 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -0.19 dB
PL1W 20.99374771 W
SFO1 600.1336008 MHz
SI 16384
SF 600.1300113 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Figure S1. ¹H NMR (600 MHz, CDCl₃) for compound 6.

¹³C of CLK1-82up



NAME CLK1-82up
EXPNO 2
PROCNO 1
Date_ 20200904
Time 11.31
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 308
DS 0
SWH 33333.332 Hz
FIDRES 0.508626 Hz
AQ 0.9831050 sec
RG 46300
DW 15.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.40000000 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 -3.55 dB
PL1W 213.31983948 W
SFO1 150.9194083 MHz
===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.80 dB
PL12 18.50 dB
PL13 21.50 dB
PL2W 30.41515160 W
PL12W 0.28385070 W
PL13W 0.14226235 W
SFO2 600.1339008 MHz
SI 32768
SF 150.9028120 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

Figure S2. ¹³C NMR (150 MHz, CDCl₃) for compound 6.

¹H of CLK1-68

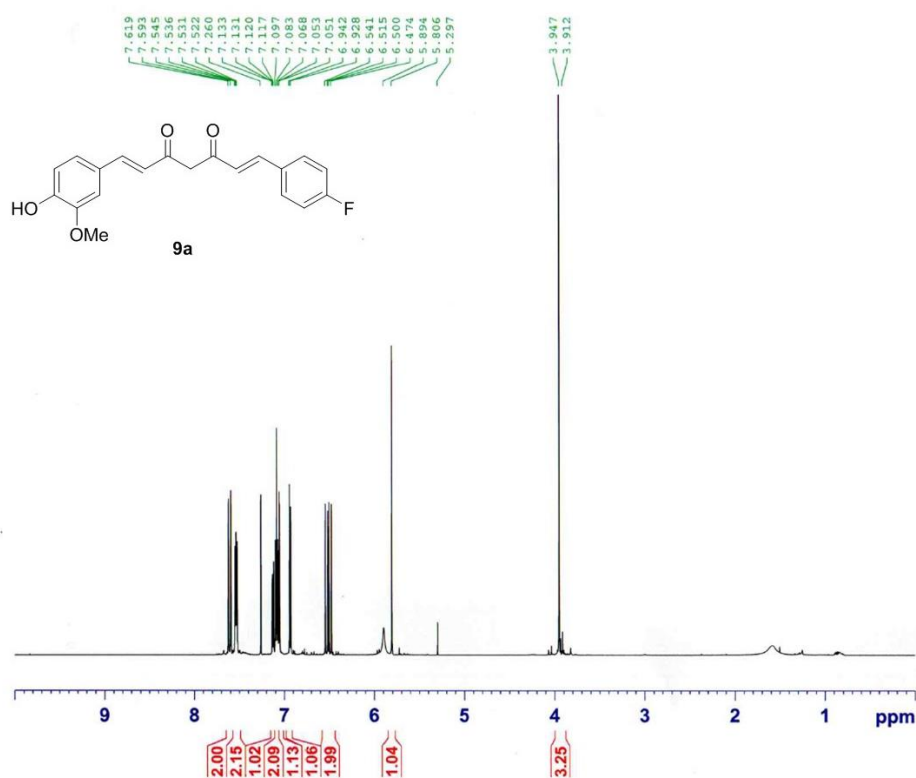


Figure S3. ¹H NMR (600 MHz, CDCl₃) for compound 9a.

¹³C of CLK1-68

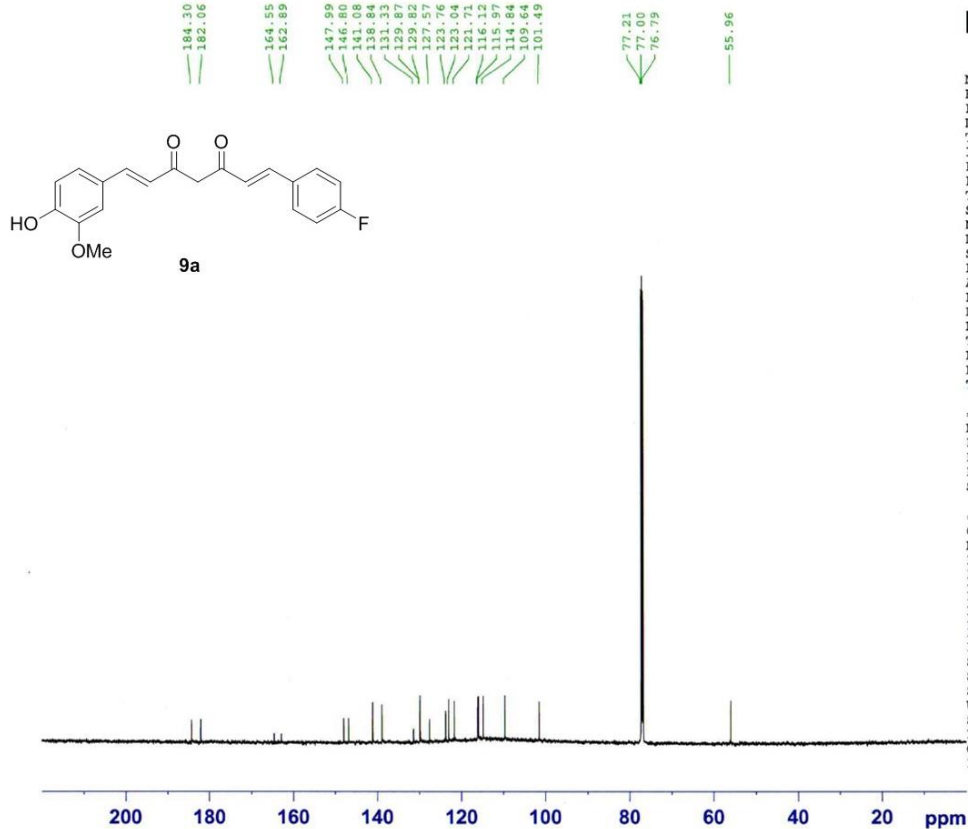


Figure S4. ¹³C NMR (150 MHz, CDCl₃) for compound 9a.



NAME CLK1-68
EXPNO 1
PROCNO 1
Date_ 20200826
Time 8.52
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530824 sec
RG 1150
DW 59.600 usec
DE 6.00 usec
TE 299.8 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -0.19 dB
PL1W 20.99374771 W
SFO1 600.1342009 MHz
SI 16384
SF 600.1300110 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00



NAME CLK1-68
EXPNO 2
PROCNO 1
Date_ 20200826
Time 9.57
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1012
DS 0
SWH 33333.332 Hz
FIDRES 0.508626 Hz
AQ 0.9831050 sec
RG 46300
DW 15.000 usec
DE 6.00 usec
TE 300.3 K
D1 2.40000010 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 -3.55 dB
PL1W 213.31983948 W
SFO1 150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.80 dB
PL12 18.50 dB
PL13 21.50 dB
PL2W 30.41515160 W
PL12W 0.28385070 W
PL13W 0.14226235 W
SFO2 600.1339008 MHz
SI 32768
SF 150.9028107 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

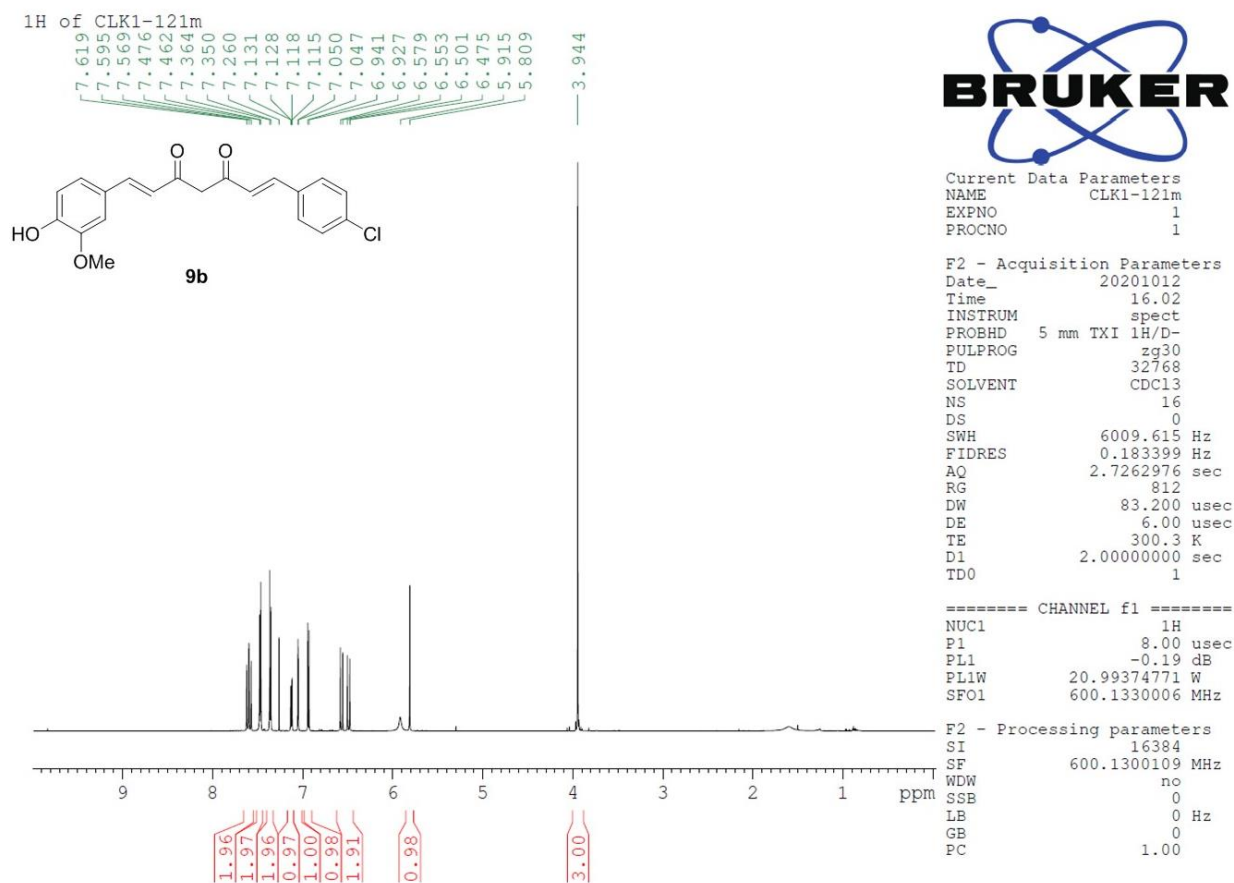


Figure S5. ¹H NMR (600 MHz, CDCl₃) for compound **9b**.

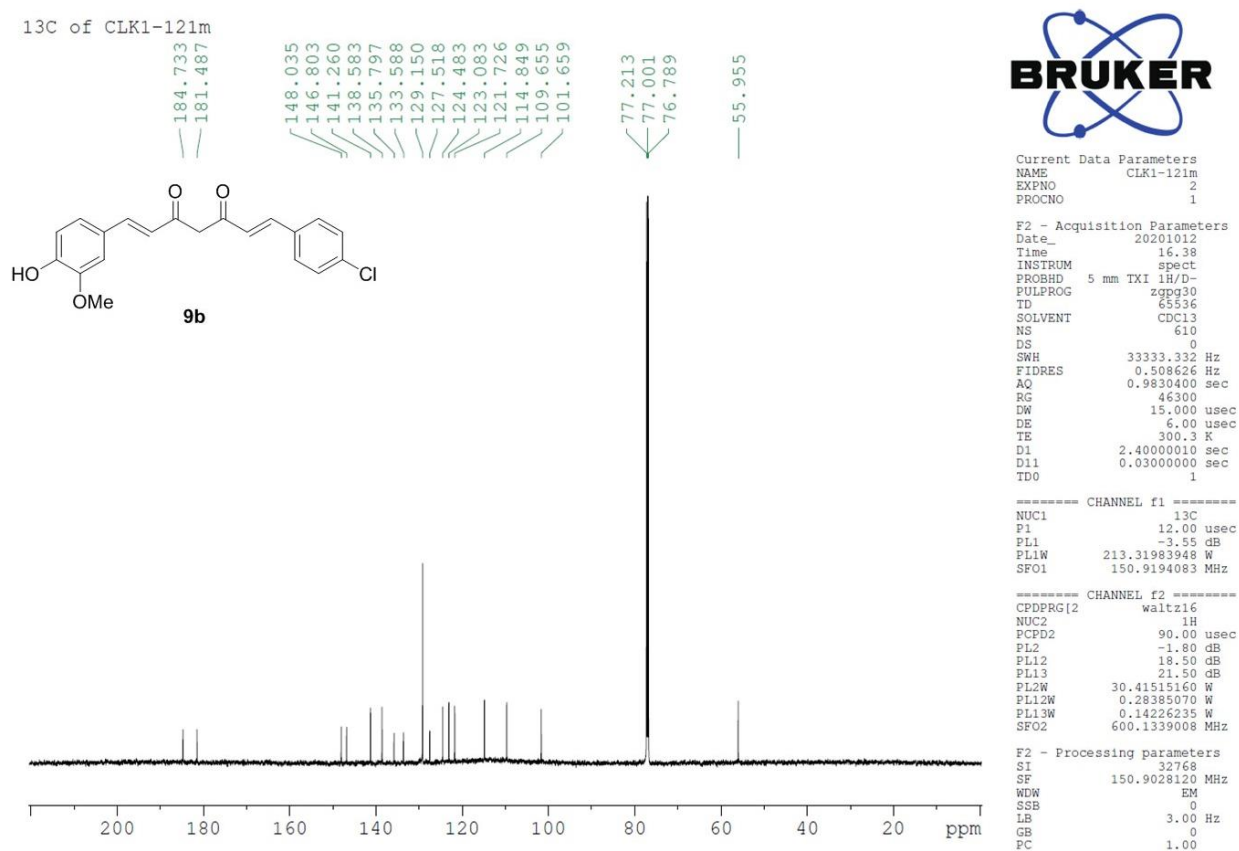
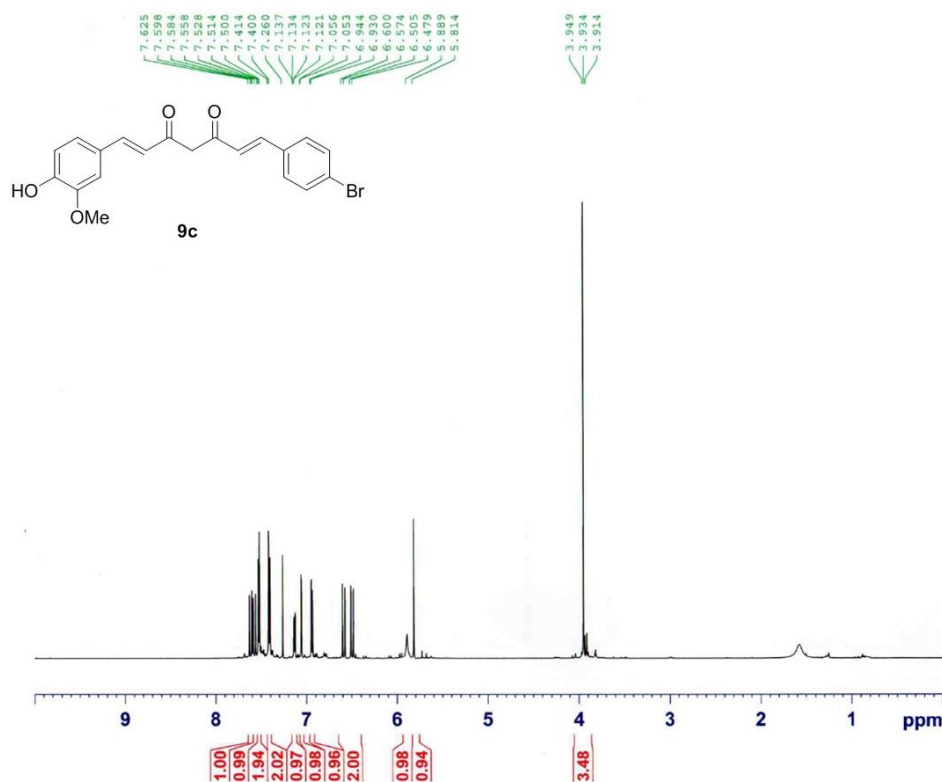


Figure S6. ¹³C NMR (150 MHz, CDCl₃) for compound **9b**.

¹H of CLK1-72



```

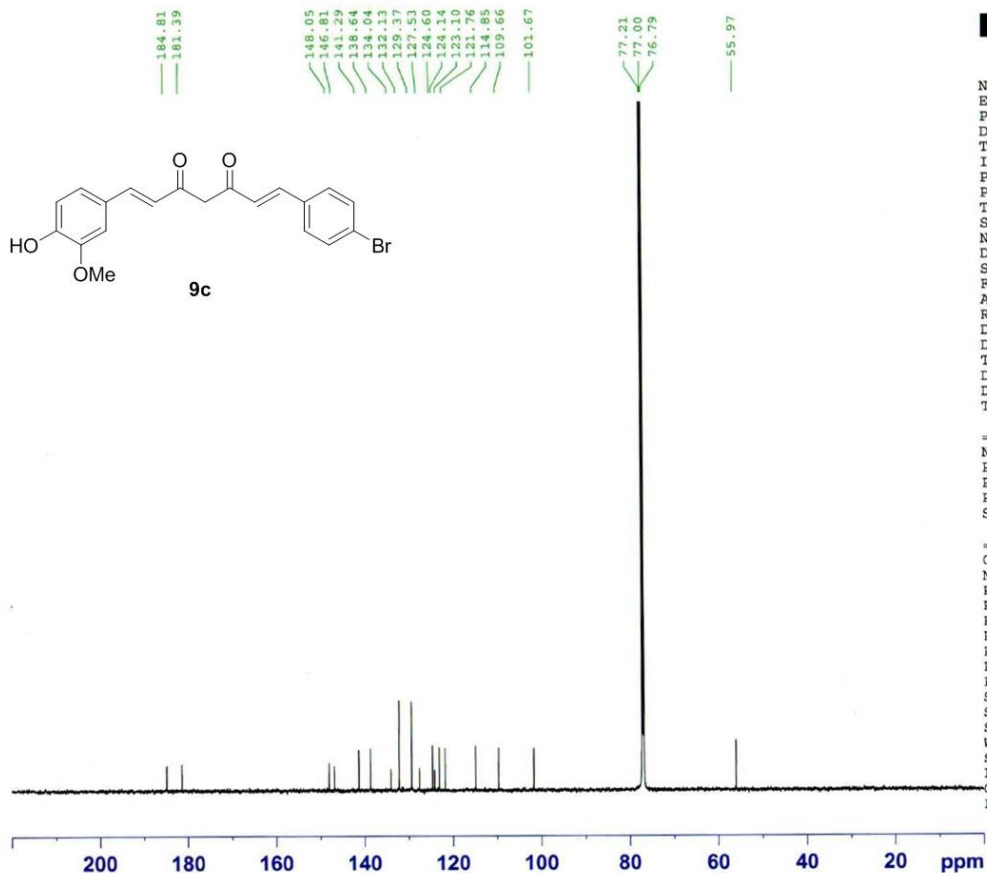
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PROCNO    1
Date_     20200911
Time      16.16
INSTRUM   spect
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PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         16
DS         0
SWH        6613.757 Hz
FIDRES     0.201836 Hz
AQ         2.4773865 sec
RG         812
DW         75.600 usec
DE         6.00 usec
TE         300.4 K
D1         2.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1         15.00 usec
PL1        -1.00 dB
PL1W       25.29822159 W
SFO1       600.1330006 MHz
SI         32768
SF         600.1300111 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```

Figure S7. ¹H NMR (600 MHz, CDCl₃) for compound 9c.

¹³C of CLK1-72



```

NAME      CLK1-72
EXPNO     2
PROCNO    1
Date_     20200910
Time      11.44
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2096
DS         0
SWH        33333.332 Hz
FIDRES     0.508626 Hz
AQ         0.9831050 sec
RG         46300
DW         15.000 usec
DE         6.00 usec
TE         298.7 K
D1         2.40000010 sec
D11        0.03000000 sec
TD0        1
  
```

```

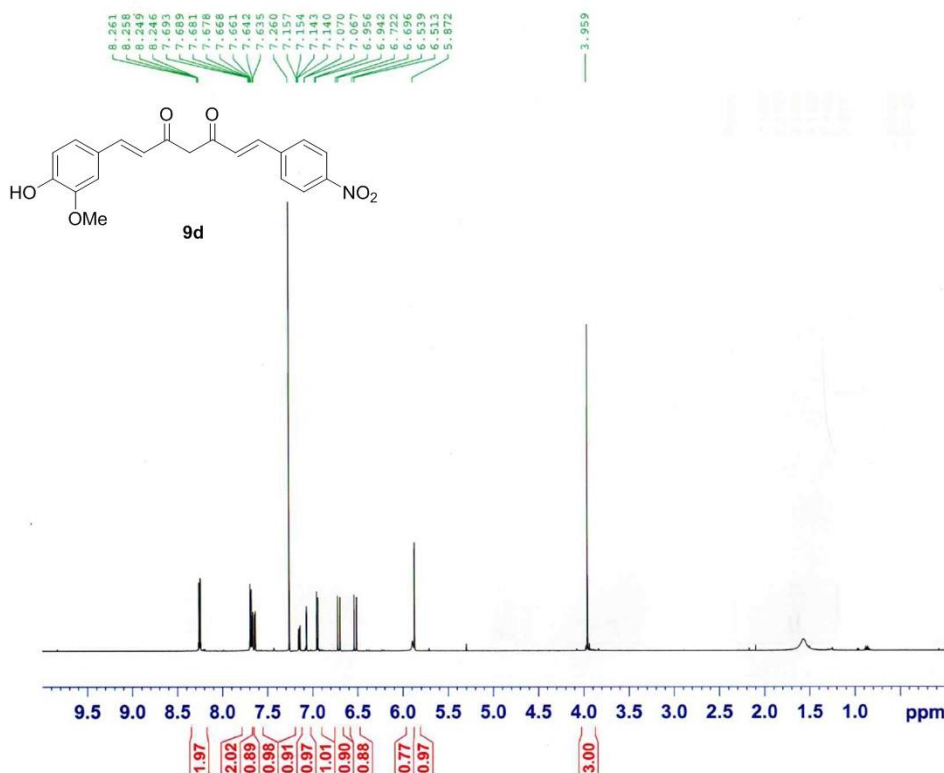
===== CHANNEL f1 =====
NUC1      13C
P1         10.00 usec
PL1        2.80 dB
PL1W       49.43462753 W
SFO1       150.9194083 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -1.60 dB
PL12       13.20 dB
PL13       16.20 dB
PL2W       29.04624367 W
PL12W      0.96181160 W
PL13W      0.48204759 W
SFO2       600.1339008 MHz
SI         32768
SF         150.9028100 MHz
WDW        EM
SSB        0
LB         3.00 Hz
GB         0
PC         1.00
  
```

Figure S8. ¹³C NMR (150 MHz, CDCl₃) for compound 9c.

¹H of CLK1-107up



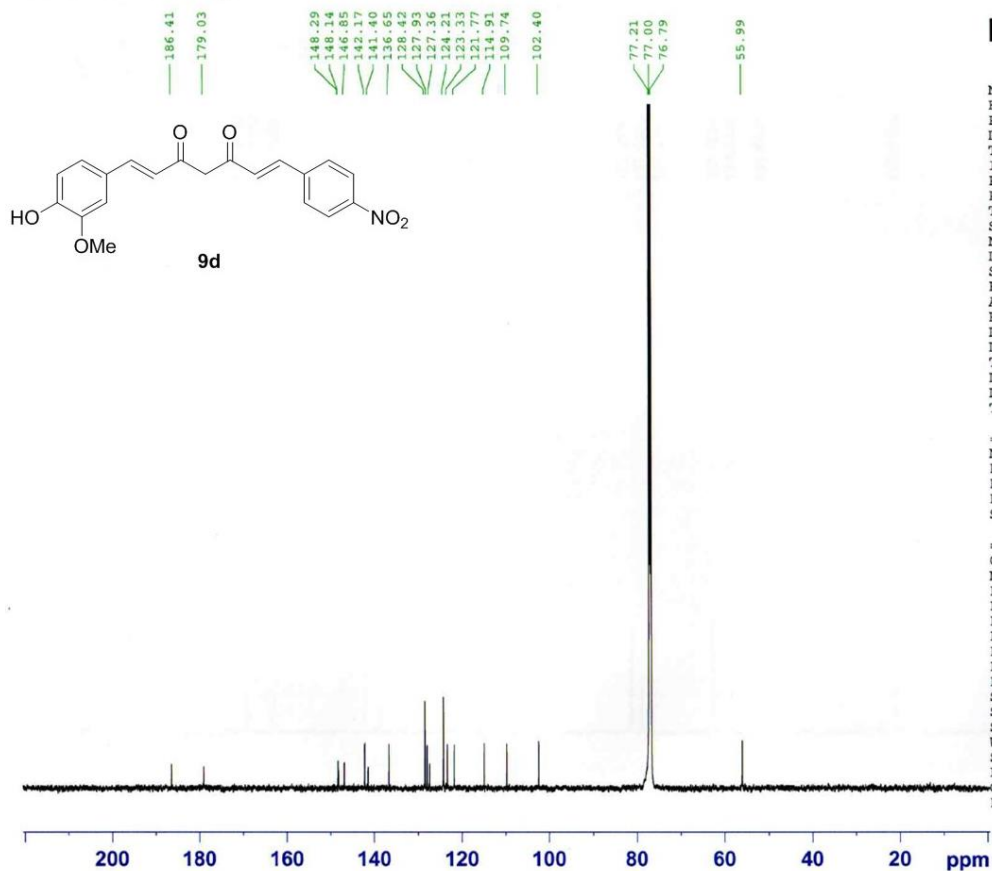
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PROCNO    1
Date_     20210226
Time      8.51
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PULPROG   zg30
TD         32768
SOLVENT   CDCl3
NS         16
DS         0
SWH        6613.757 Hz
FIDRES     0.201836 Hz
AQ         2.4773865 sec
RG         5160
DW         75.600 usec
DE         6.00 usec
TE         300.1 K
D1         2.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         8.00 usec
PL1        -0.40 dB
PL1W       22.03382874 W
SFO1       600.1330006 MHz
SI         32768
SF         600.1300104 MHz
WDW        no
SSB        0
LB         0.00 Hz
GB         0
PC         1.00
  
```

Figure S9. ¹H NMR (600 MHz, CDCl₃) for compound 9d.

¹³C of CLK1-107up



```

NAME      CLK1-107up
EXPNO     2
PROCNO    1
Date_     20210226
Time      6.01
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         16000
DS         0
SWH        33333.332 Hz
FIDRES     0.508626 Hz
AQ         0.9831050 sec
RG         46300
DW         15.000 usec
DE         6.00 usec
TE         298.4 K
D1         2.40000010 sec
D11        0.03000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       13C
P1         10.00 usec
PL1        2.80 dB
PL1W       49.43462753 W
SFO1       150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2        1H
PCPD2      90.00 usec
PL2        -1.60 dB
PL12       13.20 dB
PL13       16.20 dB
PL2W       29.04624367 W
PL12W      0.96181160 W
PL13W      0.48204759 W
SFO2       600.1339008 MHz
SI         32768
SF         150.9028080 MHz
WDW        EM
SSB        0
LB         3.00 Hz
GB         0
PC         1.00
  
```

Figure S10. ¹³C NMR (150 MHz, CDCl₃) for compound 9d.

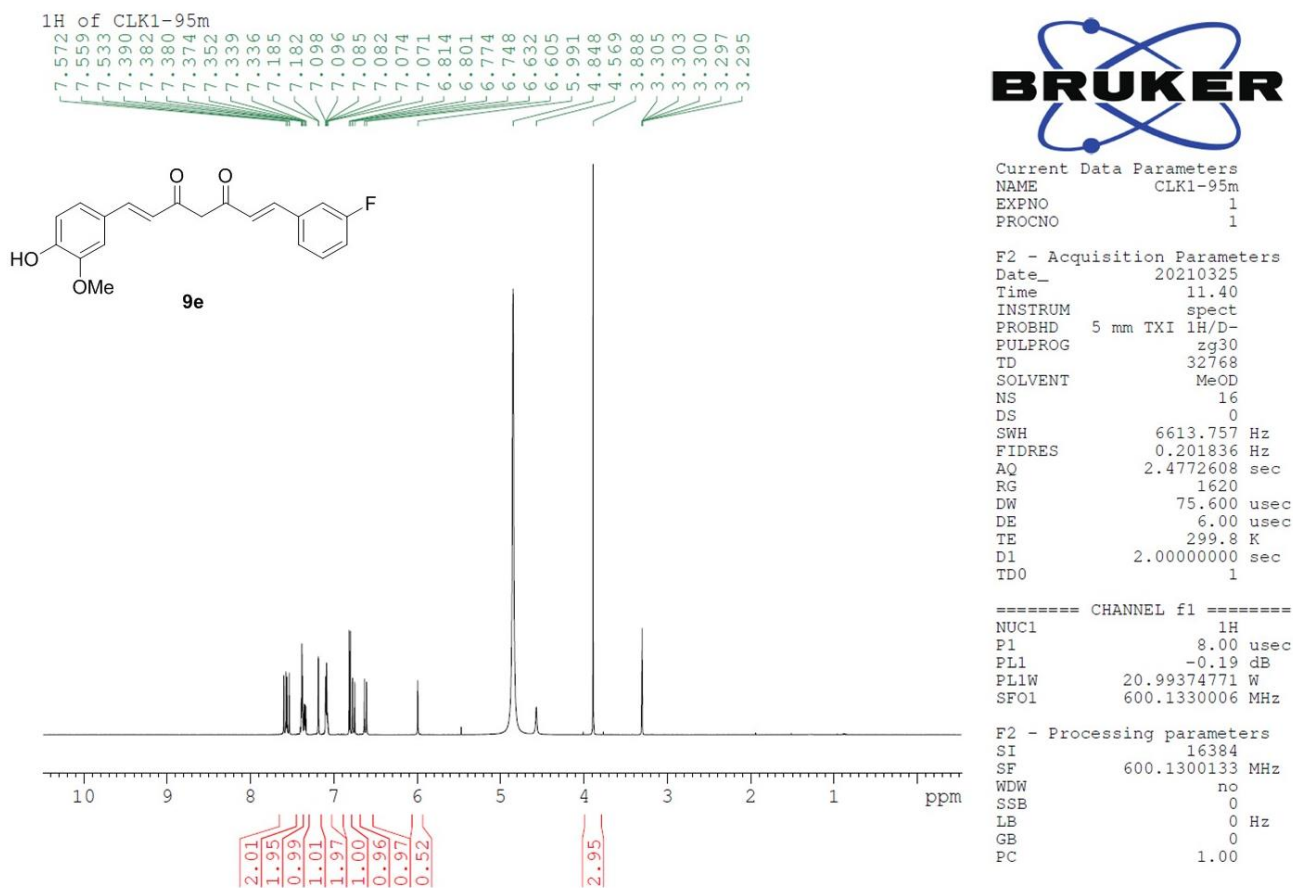


Figure S11. ¹H NMR (600 MHz, CD₃OD) for compound **9e**.

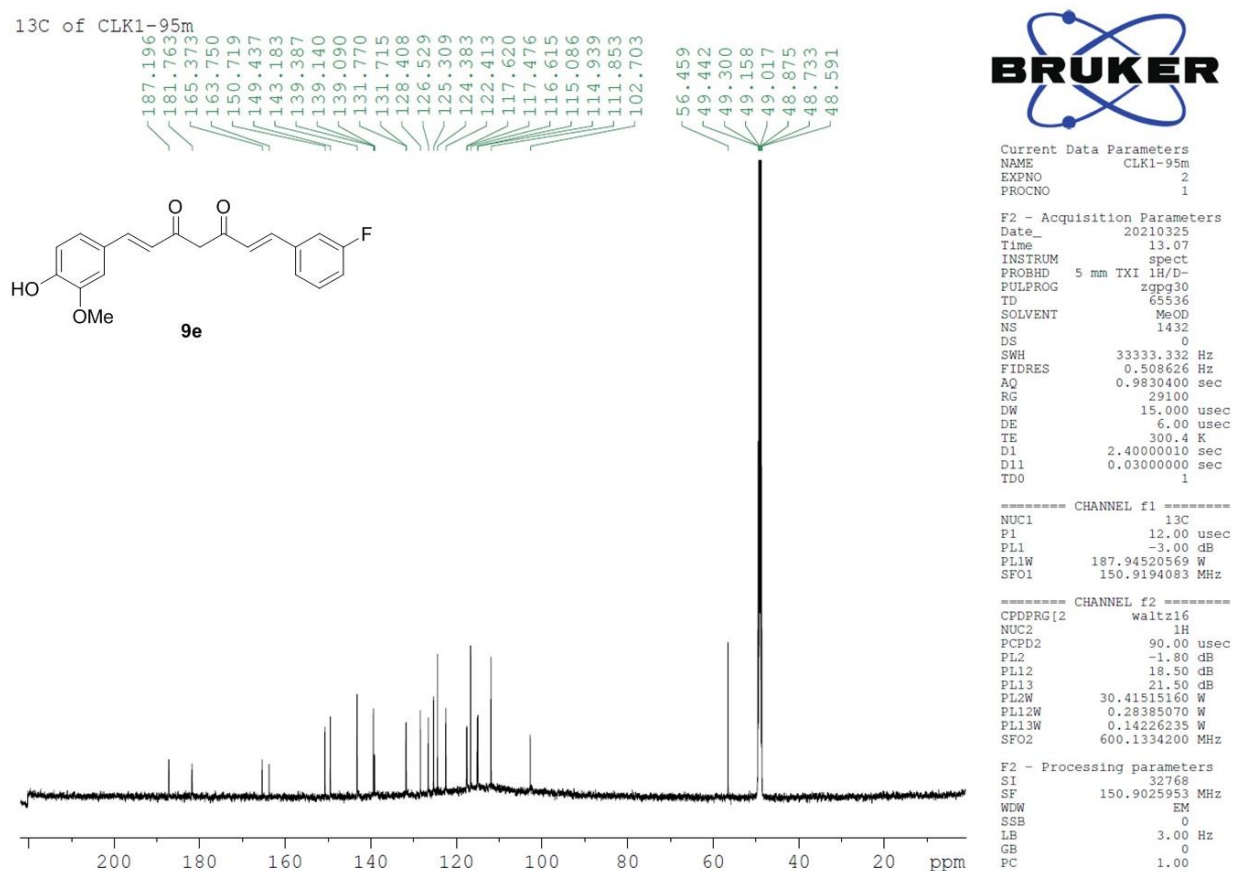
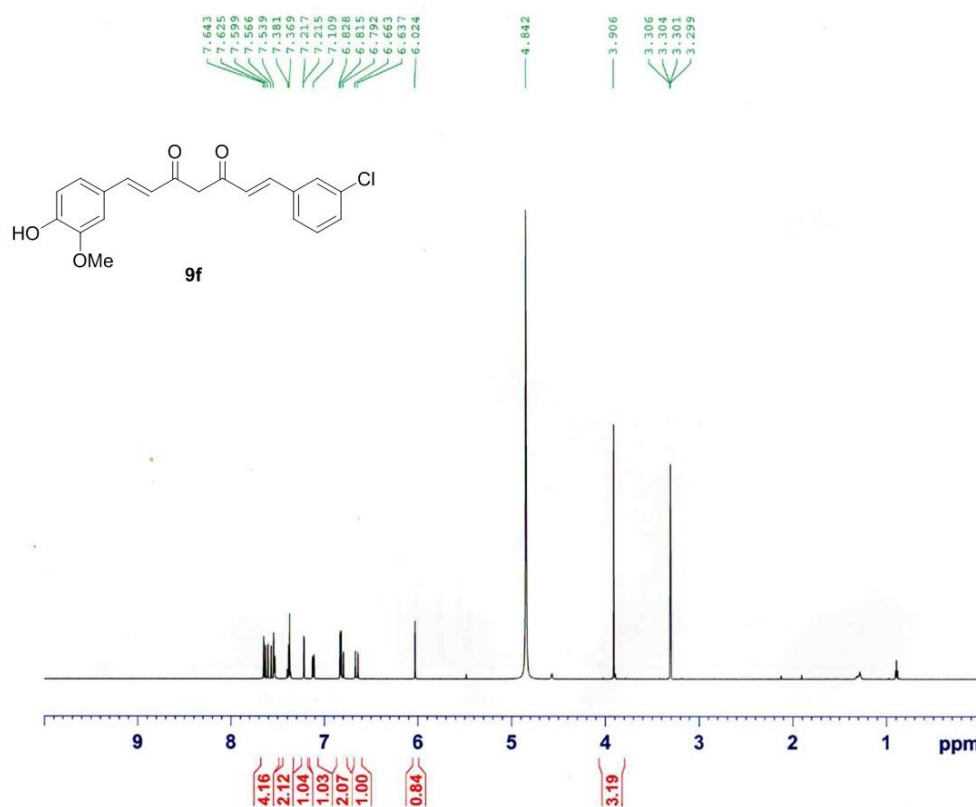


Figure S12. ¹³C NMR (150 MHz, CD₃OD) for compound **9e**.

¹H of CLK1-97m

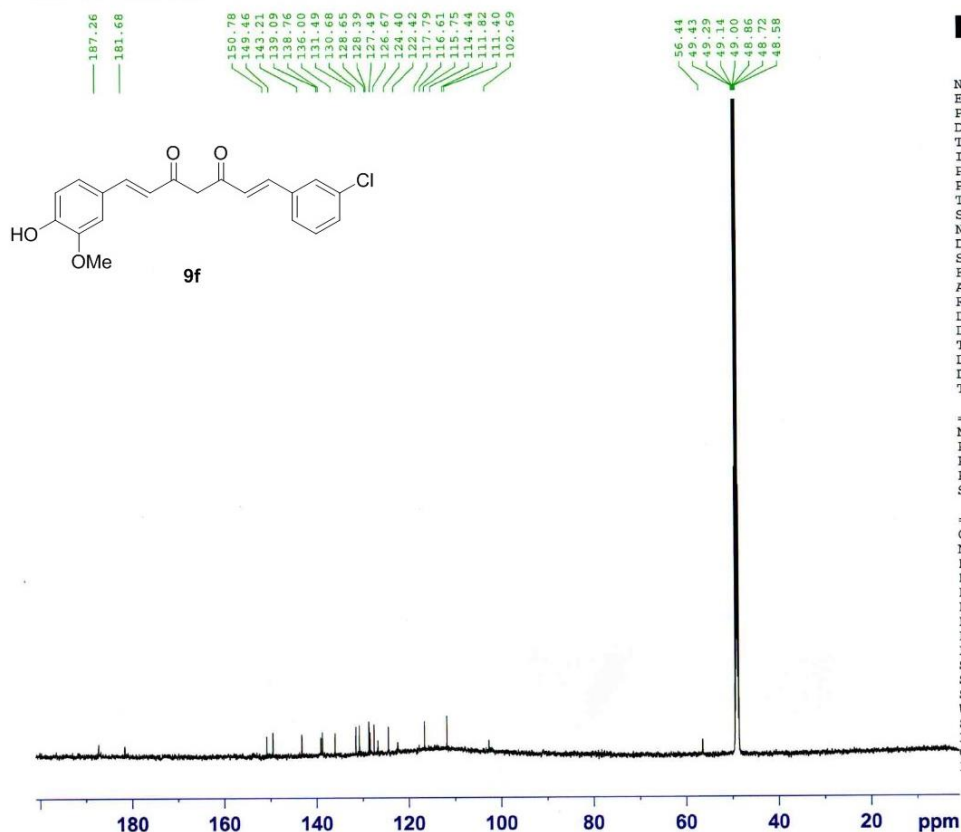


NAME CLK1-97m
EXPNO 11
PROCNO 1
Date_ 20210315
Time 9.51
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 32768
SOLVENT MeOD
NS 16
DS 0
SWH 6613.757 Hz
FIDRES 0.201836 Hz
AQ 2.4773865 sec
RG 6500
DW 75.600 usec
DE 6.00 usec
TE 300.4 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -0.19 dB
PL1W 20.9937471 W
SFO1 600.1330006 MHz
SI 16384
SF 600.1300111 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Figure S13. ¹H NMR (600 MHz, CD₃OD) for compound 9f.

¹³C of CLK1-97m



NAME CLK1-97m
EXPNO 12
PROCNO 1
Date_ 20210315
Time 11.42
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 1822
DS 0
SWH 33333.332 Hz
FIDRES 0.508626 Hz
AQ 0.9831050 sec
RG 29100
DW 15.000 usec
DE 6.00 usec
TE 300.4 K
D1 2.40000010 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.95 usec
PL1 -4.00 dB
PL1W 236.60900879 W
SFO1 150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 2.00 dB
PL12 19.00 dB
PL13 21.00 dB
PL2W 12.67914581 W
PL12W 0.25298220 W
PL13W 0.15962099 W
SFO2 600.1339008 MHz
SI 32768
SF 150.9025953 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

Figure S14. ¹³C NMR (150 MHz, CD₃OD) for compound 9f.

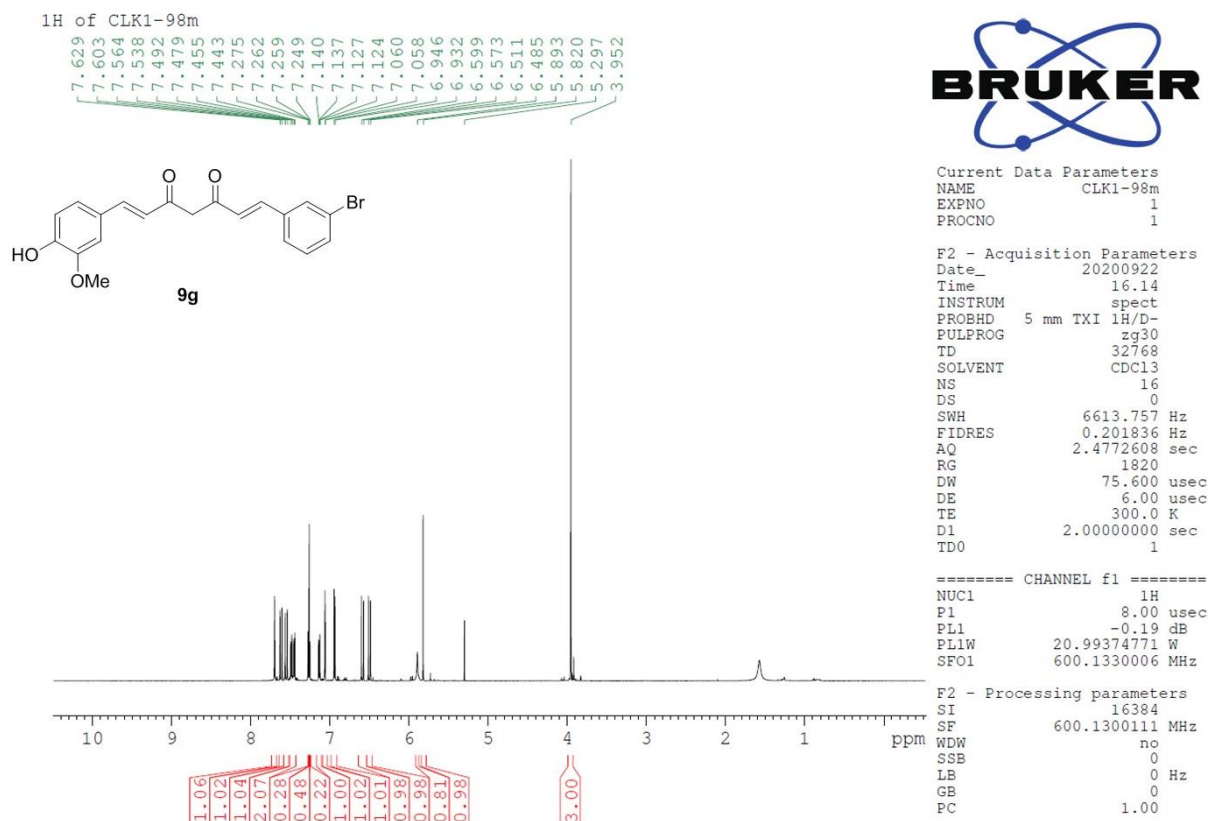


Figure S15. ¹H NMR (600 MHz, CDCl₃) for compound 9g.

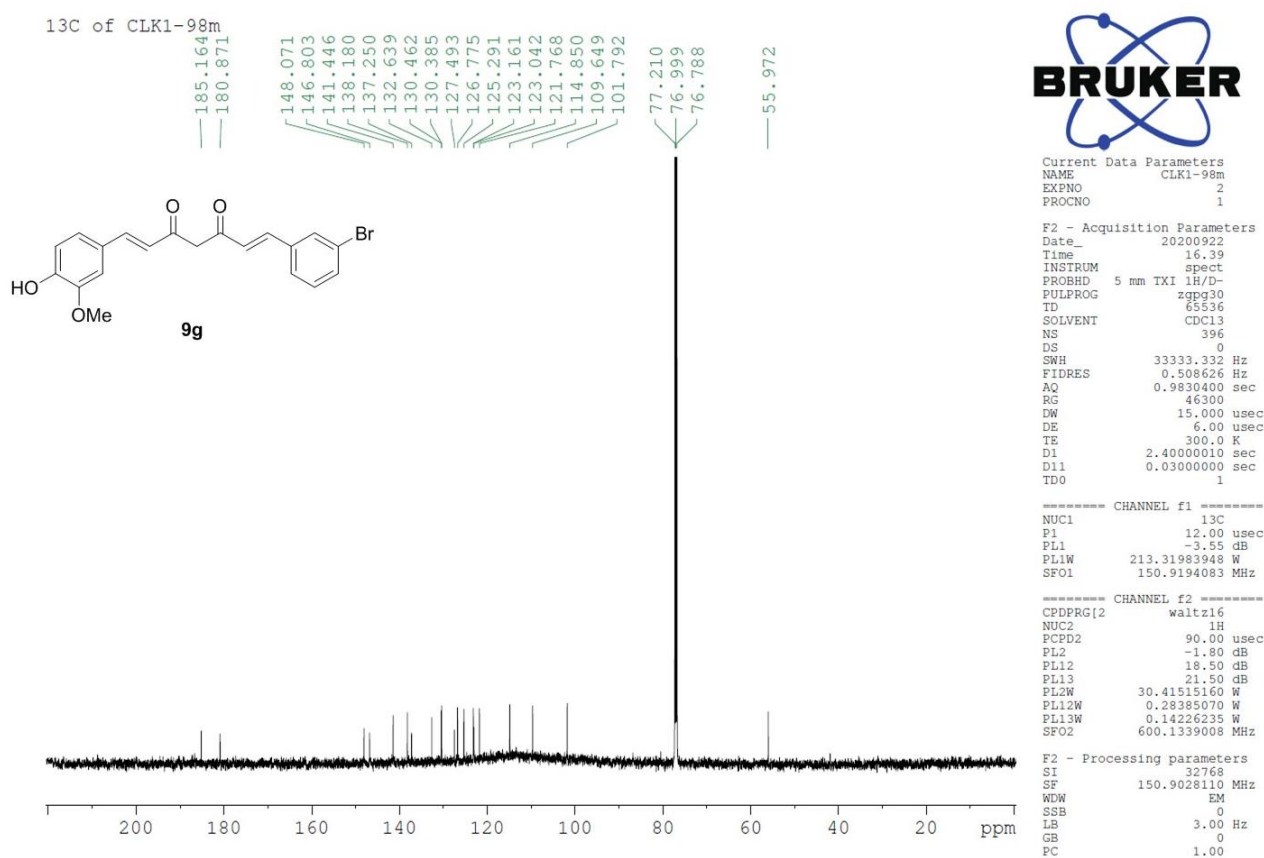
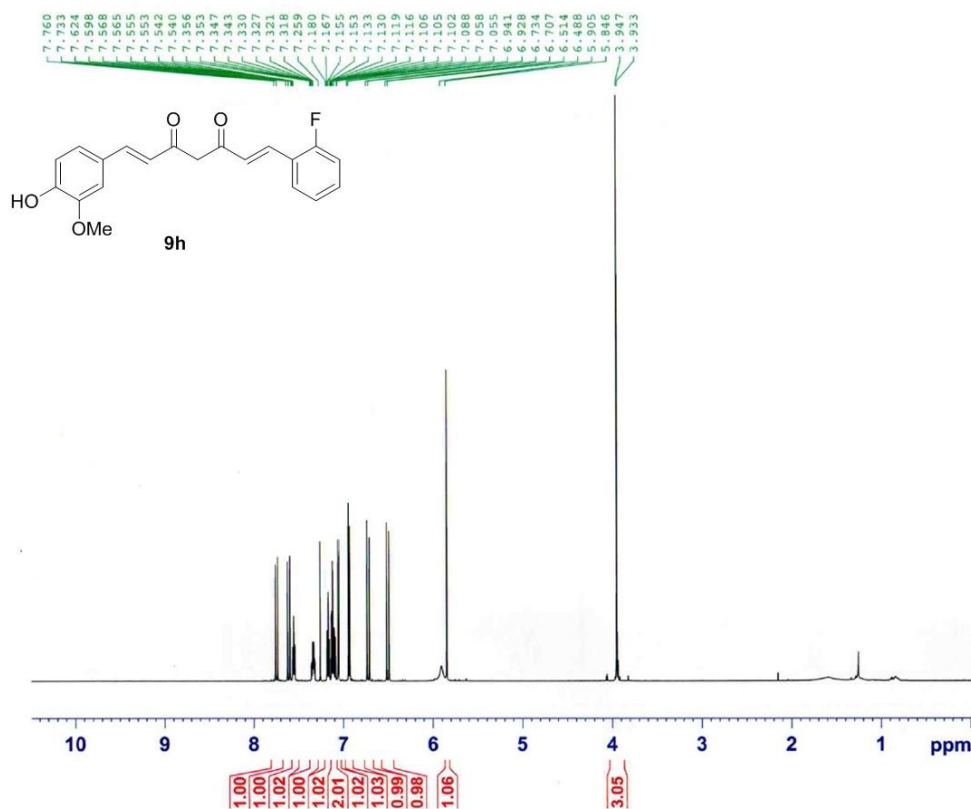


Figure S16. ¹³C NMR (150 MHz, CDCl₃) for compound 9g.

¹H of CLK1-99m

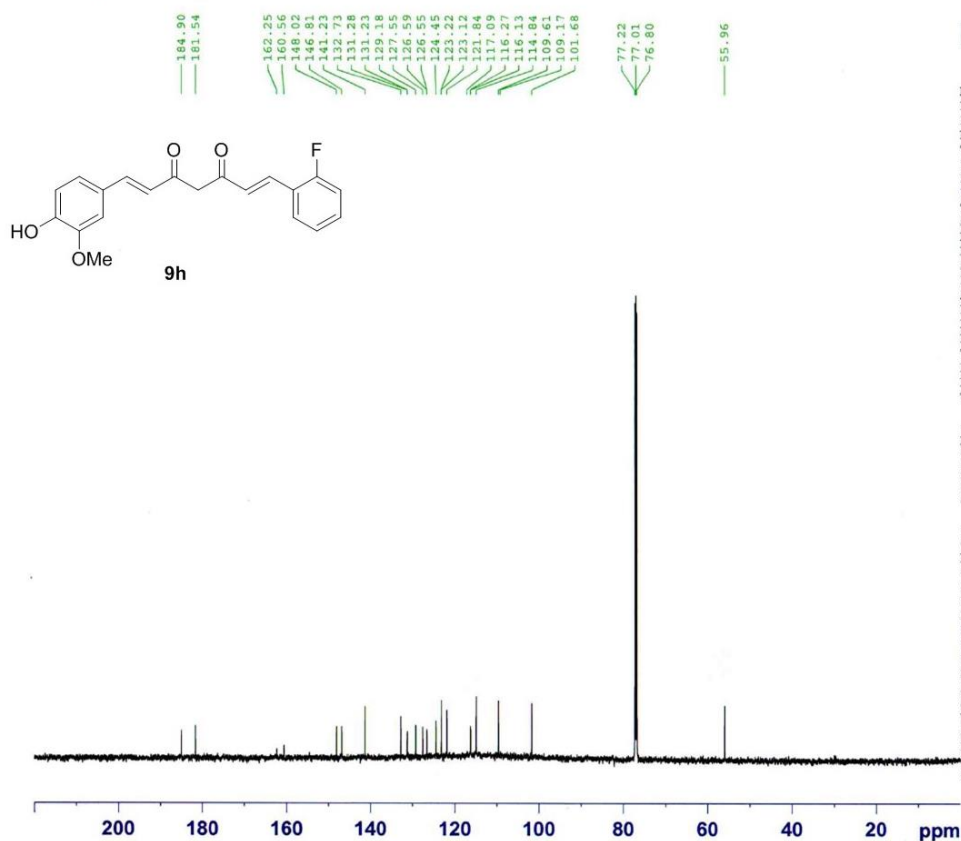


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PROCNO 1
Date_ 20200921
Time 16.06
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6613.757 Hz
FIDRES 0.201836 Hz
AQ 2.4773865 sec
RG 1820
DW 75.600 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -0.19 dB
PL1W 20.99374771 W
SFO1 600.1330006 MHz
SI 16384
SF 600.1330111 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Figure S17. ¹H NMR (600 MHz, CDCl₃) for compound 9h.

¹³C of CLK1-99m



NAME CLK1-99m
EXPNO 2
PROCNO 1
Date_ 20200921
Time 16.24
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 266
DS 0
SWH 33333.332 Hz
FIDRES 0.508626 Hz
AQ 0.9831050 sec
RG 46300
DW 15.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.40000010 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 -3.55 dB
PL1W 213.31983948 W
SFO1 150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.80 dB
PL12 18.50 dB
PL13 21.50 dB
PL2W 30.41515160 W
PL12W 0.28385070 W
PL13W 0.14226235 W
SFO2 600.1339008 MHz
SI 32768
SF 150.9028108 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

Figure S18. ¹³C NMR (150 MHz, CDCl₃) for compound 9h.

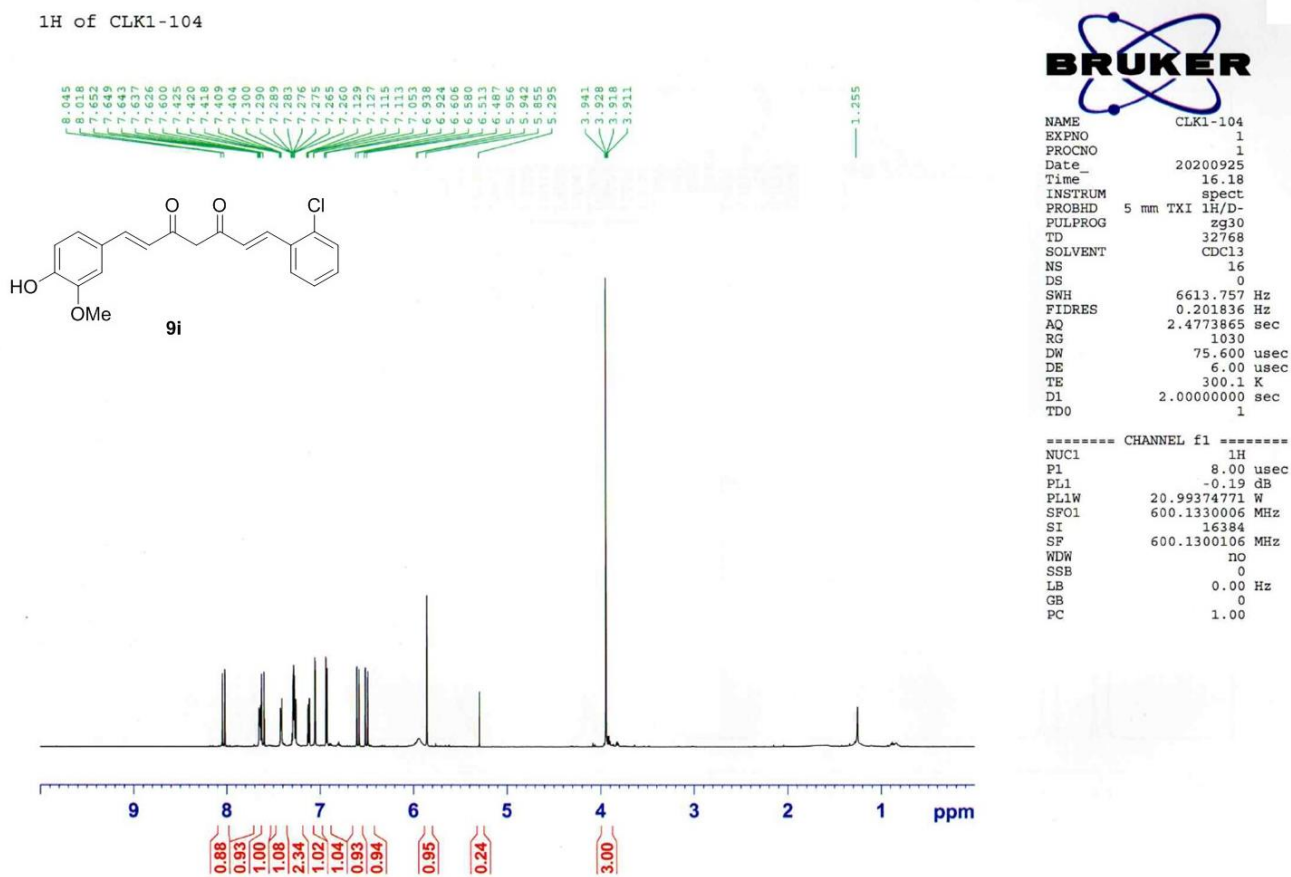


Figure S19. ¹H NMR (600 MHz, CDCl₃) for compound 9i.

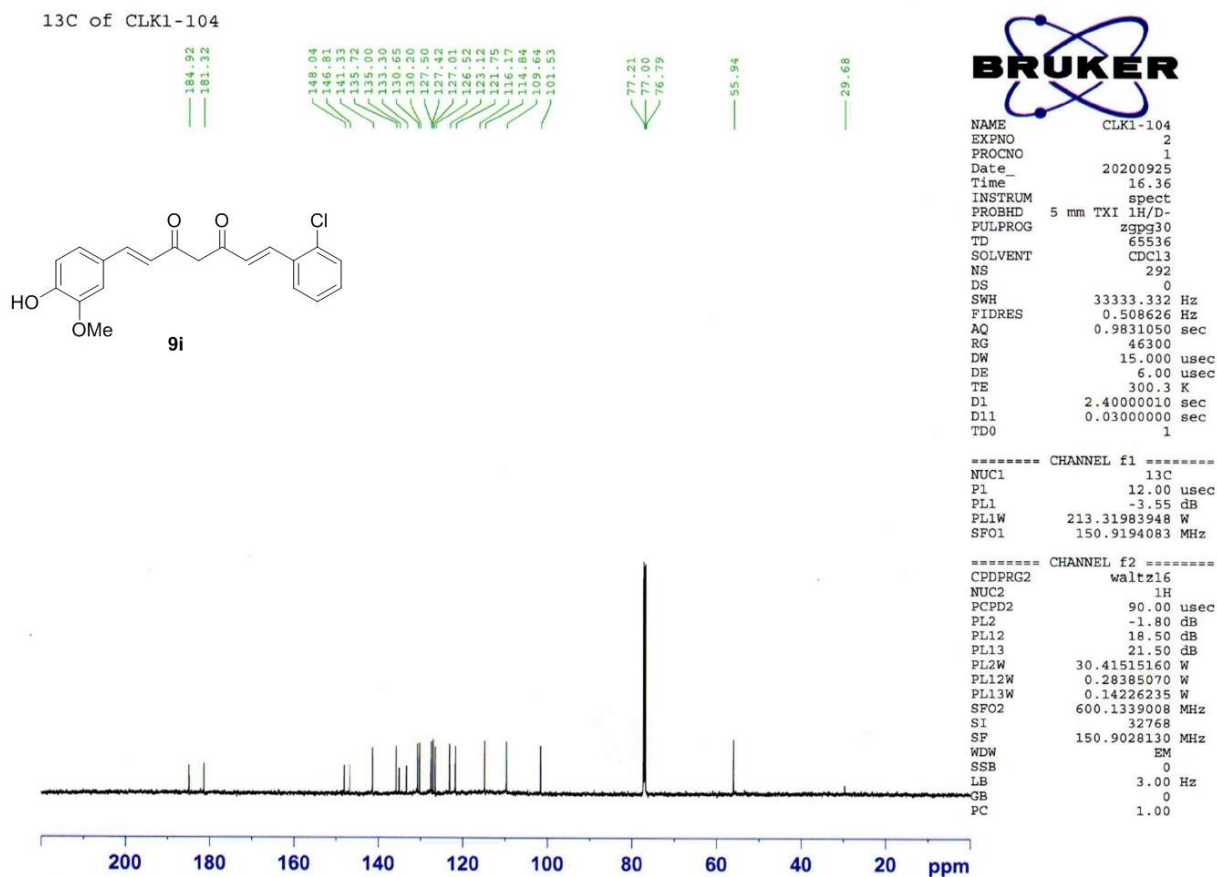
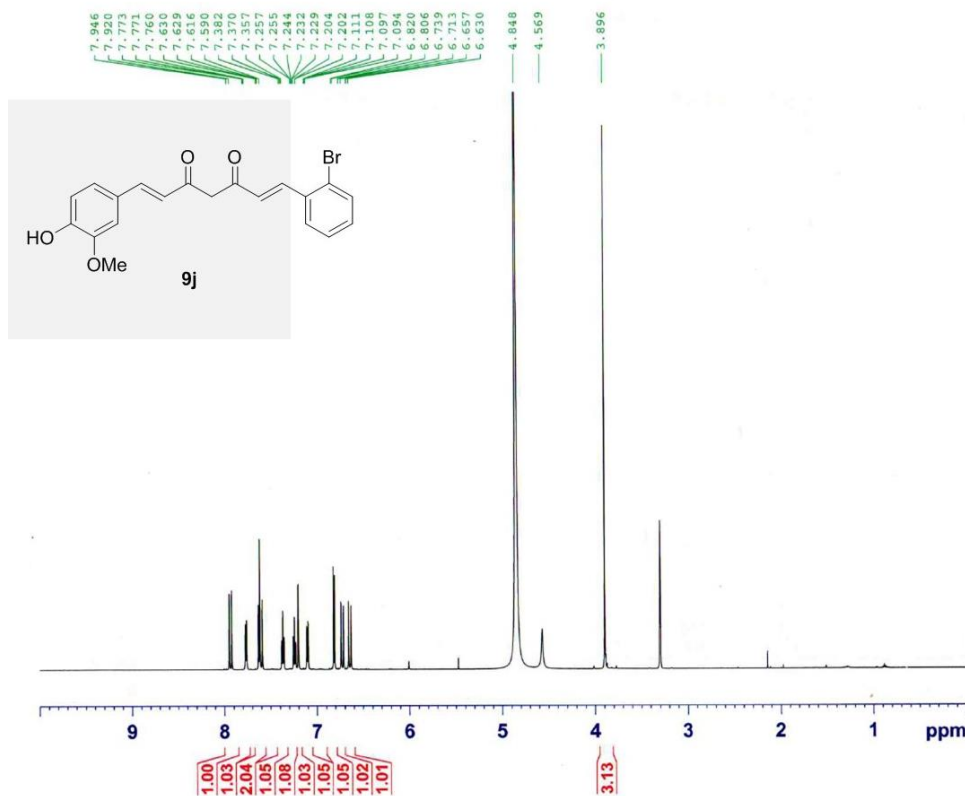


Figure S20. ¹³C NMR (150 MHz, CDCl₃) for compound 9i.

¹H of CLK1-105m

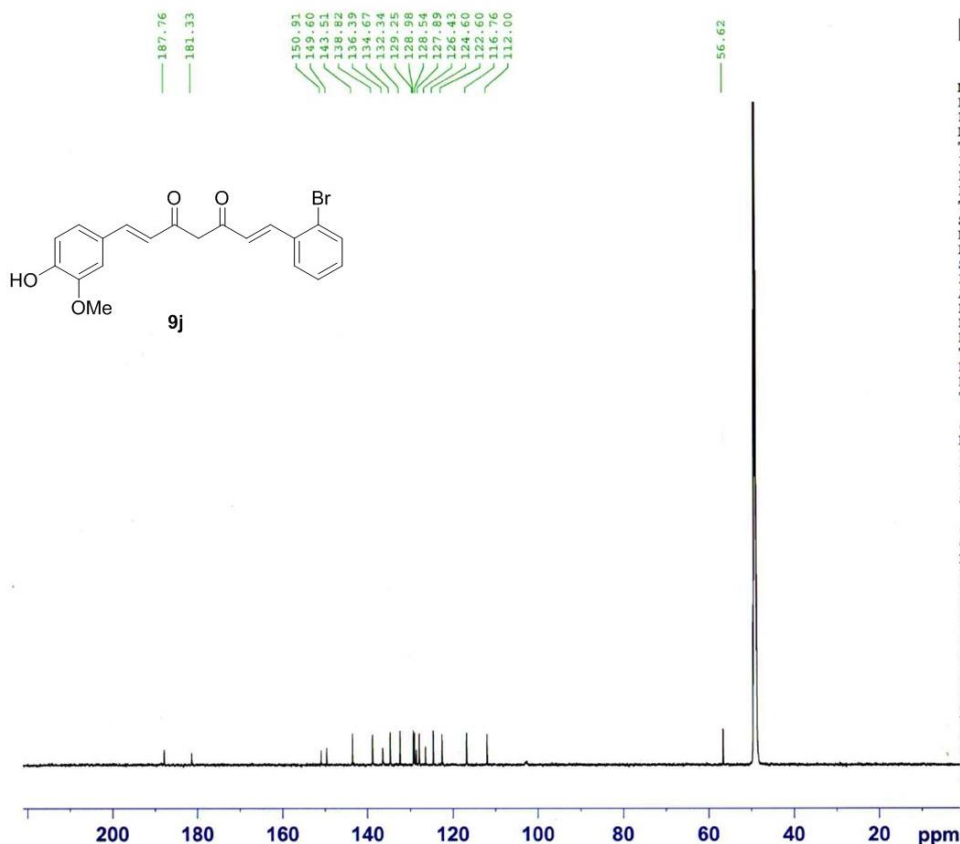


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PROCNO 1
Date_ 20210414
Time_ 15.42
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PULPROG zg30
TD 32768
SOLVENT MeOD
NS 16
DS 0
SWH 6613.757 Hz
FIDRES 0.201836 Hz
AQ 2.4773865 sec
RG 1150
DW 75.600 usec
DE 6.00 usec
TE 299.9 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -0.19 dB
PL1W 20.99374771 W
SFO1 600.1330006 MHz
SI 16384
SF 600.1300131 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

Figure S21. ¹H NMR (600 MHz, CD₃OD) for compound 9j.

¹³C of CLK1-105m

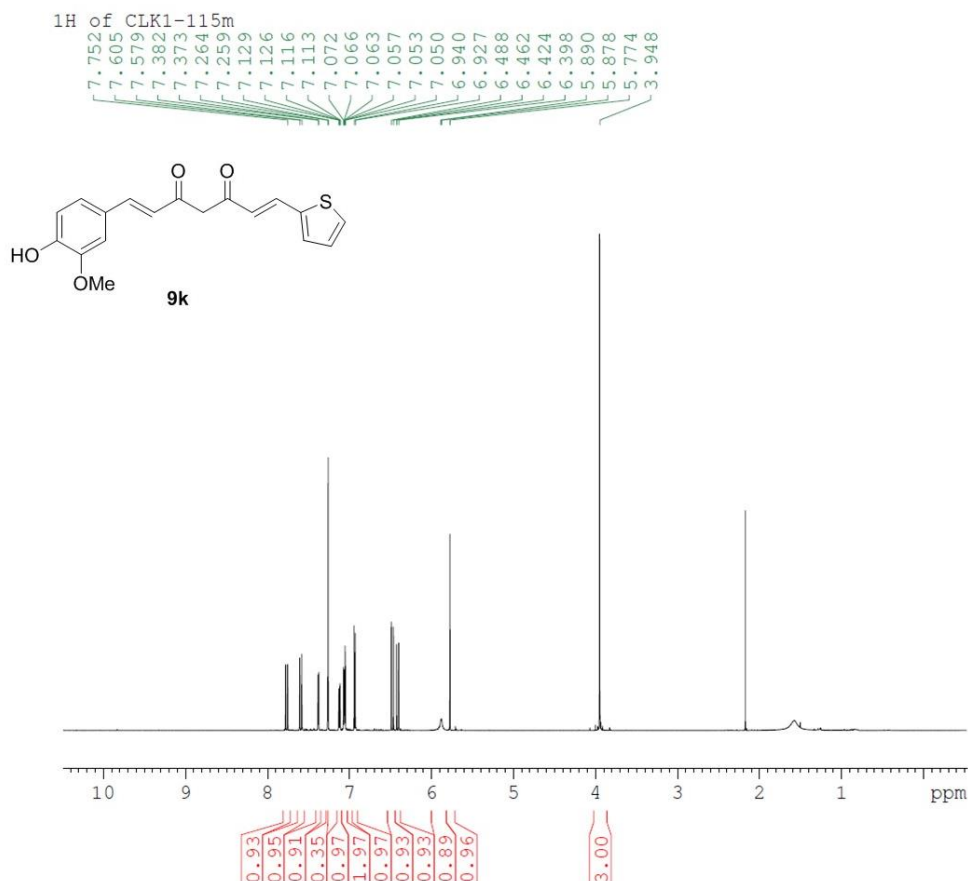


NAME CLK1-105m
EXPNO 202
PROCNO 1
Date_ 20210414
Time_ 13.52
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT MeOD
NS 3123
DS 0
SWH 33333.332 Hz
FIDRES 0.508626 Hz
AQ 0.9831050 sec
RG 46300
DW 15.000 usec
DE 6.00 usec
TE 298.4 K
D1 2.40000010 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 10.00 usec
PL1 2.80 dB
PL1W 49.43462753 W
SFO1 150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.60 dB
PL12 13.20 dB
PL13 16.20 dB
PL2W 29.04624367 W
PL12W 0.96181160 W
PL13W 0.48204759 W
SFO2 600.1339008 MHz
SI 32768
SF 150.9025731 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

Figure S22. ¹³C NMR (150 MHz, CD₃OD) for compound 9j.



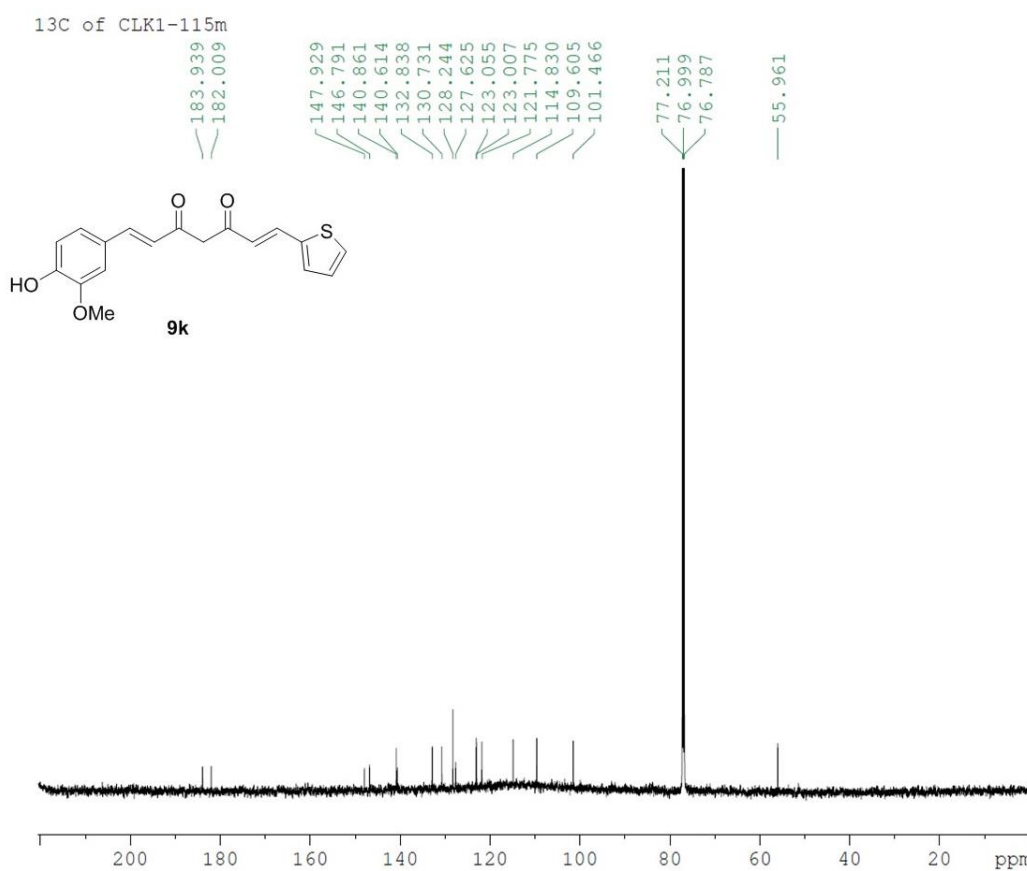
Current Data Parameters
NAME CLK1-115m
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200918
Time 8.51
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 6613.757 Hz
FIDRES 0.201836 Hz
AQ 2.4772608 sec
RG 4100
DW 75.600 usec
DE 6.00 usec
TE 299.8 K
D1 2.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 8.00 usec
PL1 -0.19 dB
PL1W 20.99374771 W
SFO1 600.1330006 MHz

F2 - Processing parameters
SI 16384
SF 600.1300111 MHz
WDW no
SSB 0
LB 0 Hz
GB 0
PC 1.00

Figure S23. ¹H NMR (600 MHz, CDCl₃) for compound 9k.



Current Data Parameters
NAME CLK1-115m
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20200918
Time 9.18
INSTRUM spect
PROBHD 5 mm TXI 1H/D-
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 412
DS 0
SWH 33333.332 Hz
FIDRES 0.508626 Hz
AQ 0.9830400 sec
RG 46300
DW 15.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.40000010 sec
D11 0.03000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 13C
P1 12.00 usec
PL1 -3.55 dB
PL1W 213.31983948 W
SFO1 150.9194083 MHz

===== CHANNEL f2 =====
CPDPRG[2] waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -1.80 dB
PL12 18.50 dB
PL13 21.50 dB
PL2W 30.41515160 W
PL12W 0.28385070 W
PL13W 0.14226235 W
SFO2 600.1339008 MHz

F2 - Processing parameters
SI 32768
SF 150.9028108 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

Figure S24. ¹³C NMR (150 MHz, CDCl₃) for compound 9k.

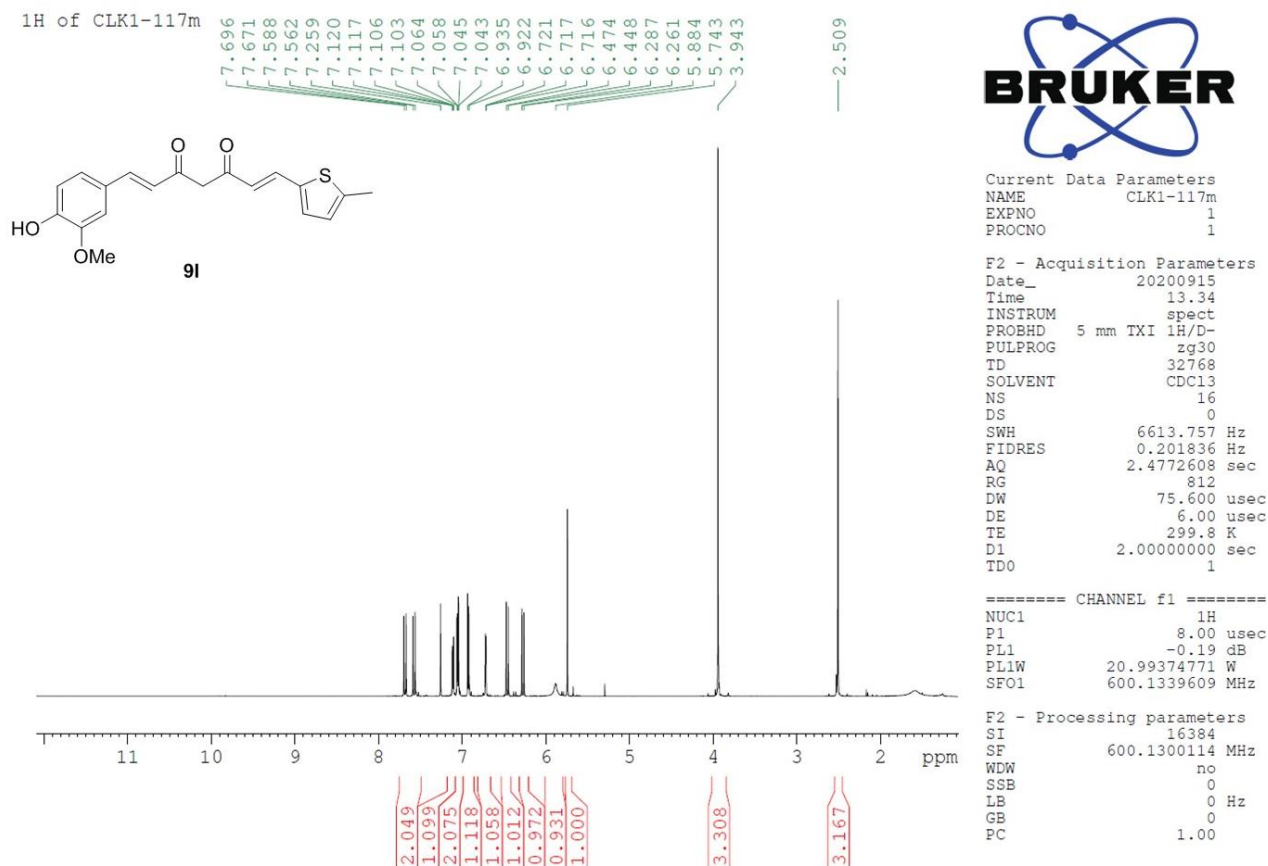


Figure S25. ¹H NMR (600 MHz, CDCl₃) for compound 9l.

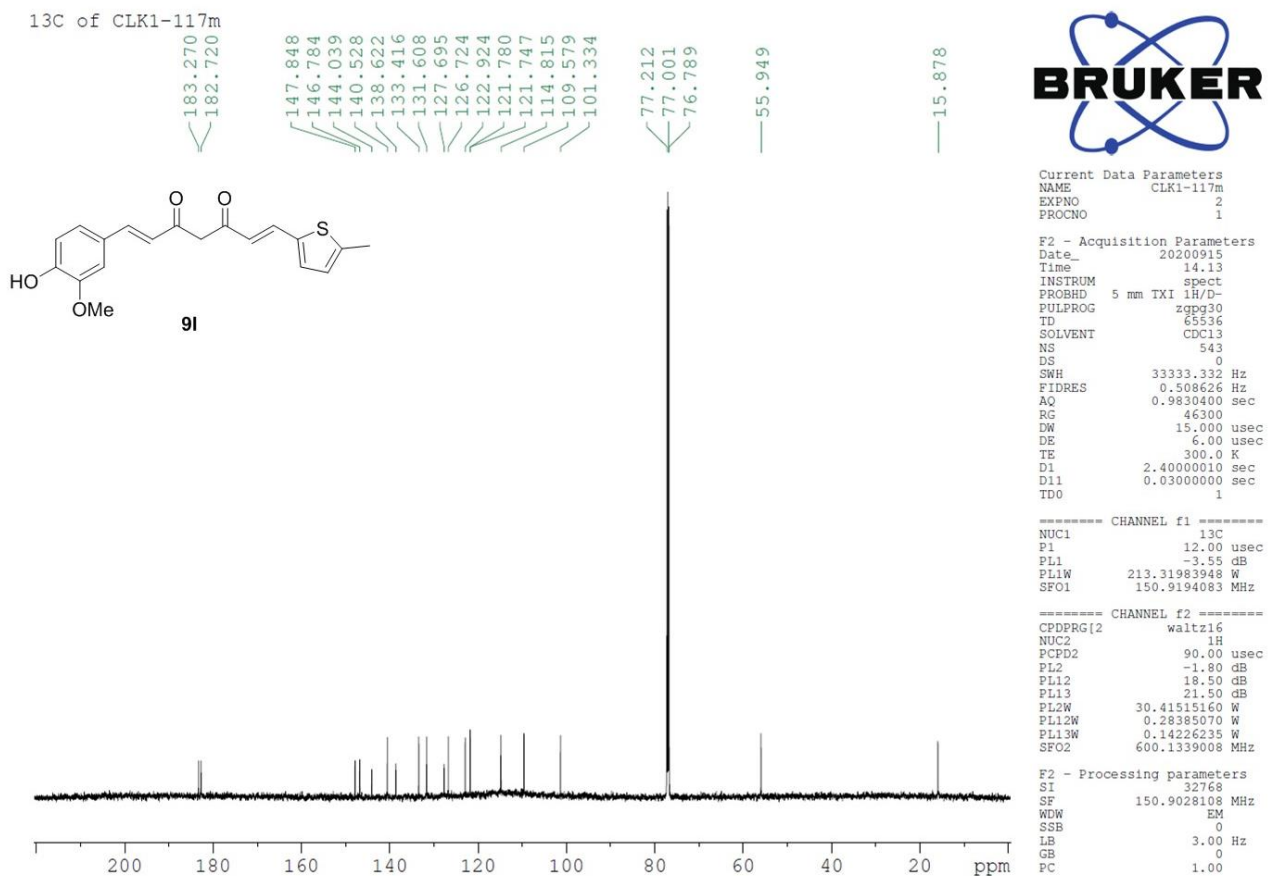


Figure S26. ¹³C NMR (150 MHz, CDCl₃) for compound 9l.

¹H of KYL-26

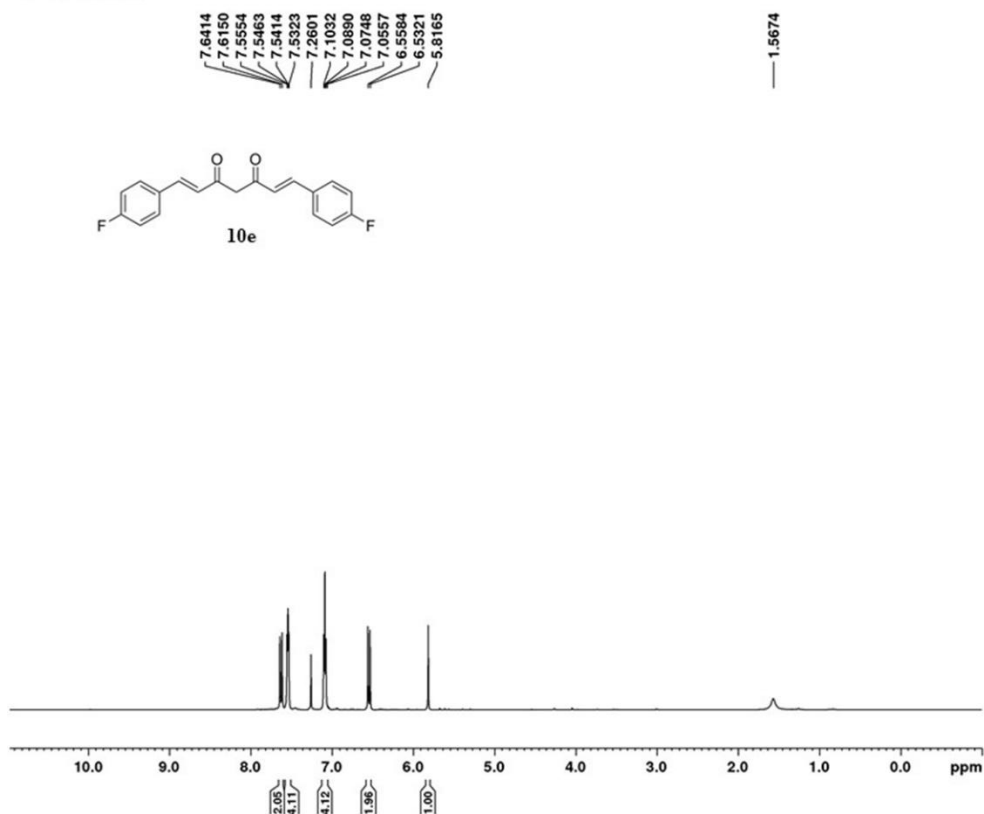


Figure S27. ¹H NMR (600 MHz, CDCl₃) for compound 10e.

¹³C of KYL-26

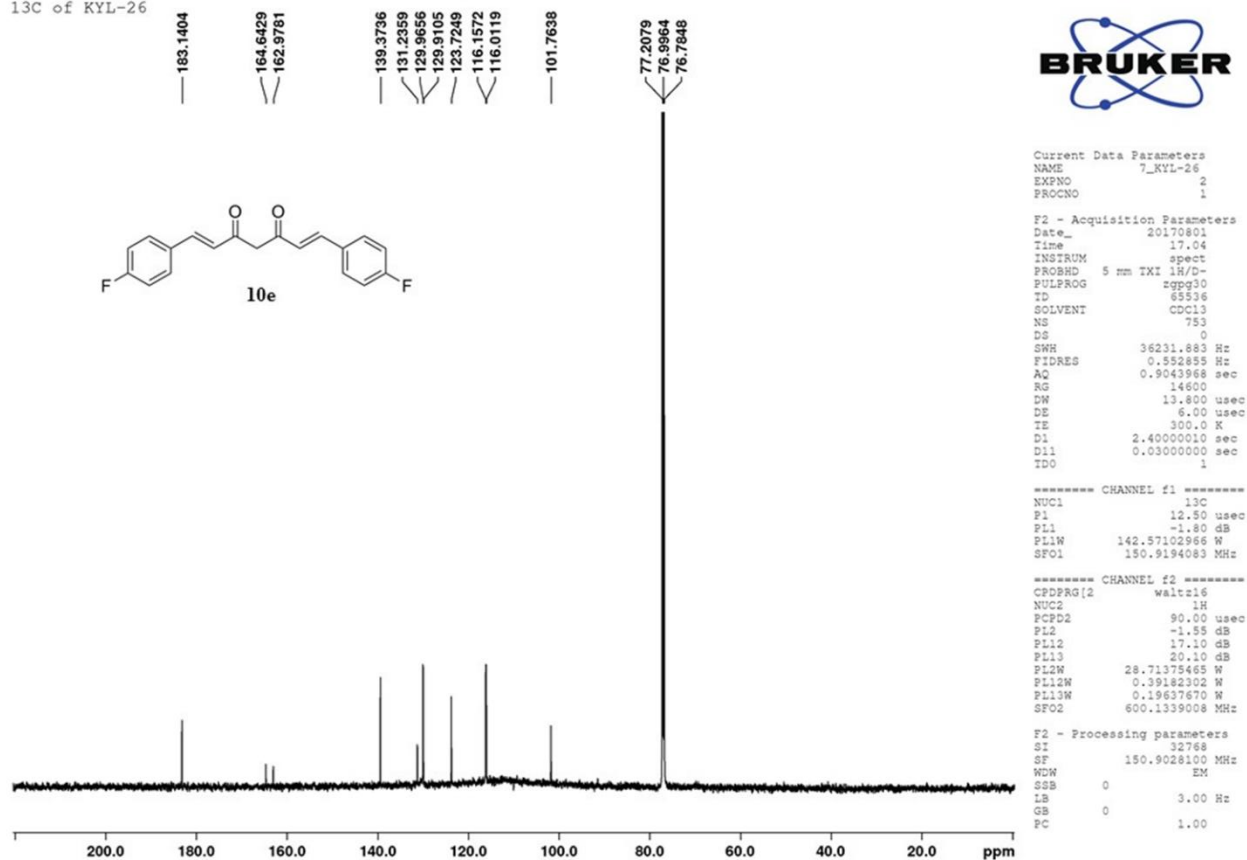
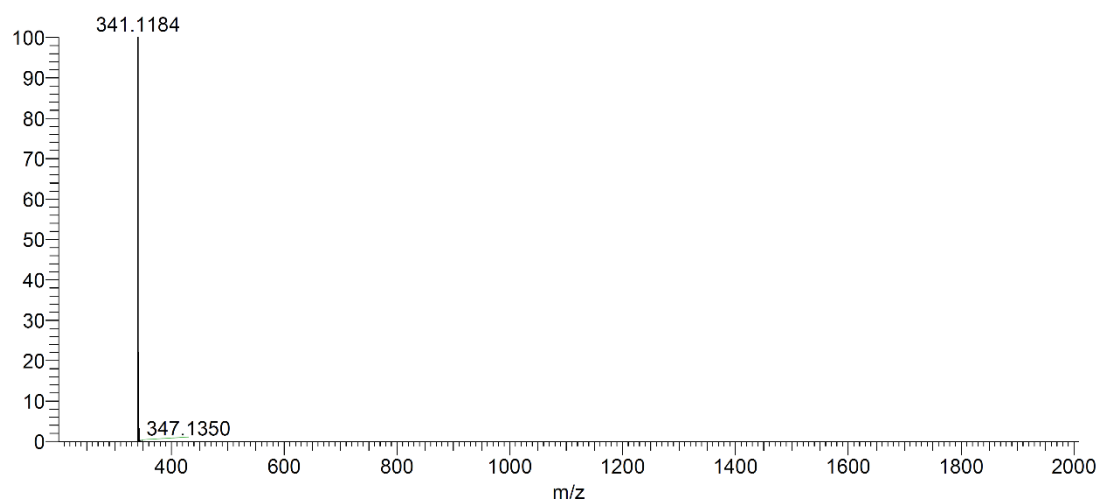


Figure S28. ¹³C NMR (150 MHz, CDCl₃) for compound 10e.



NL:
7.97E5
C₂₀ H₁₇ FO₄ +H:
C₂₀ H₁₈ F₁ O₄
c (gss, s /p:40)(Val) Chrg 1
R: 20000 Res .Pwr . @FWHM

Figure S29. HR-ESI-MS for compound **9a**