

Electronic Supplementary Information (ESI)

N-Doped TiO₂ Nanocatalyst for One-Pot Synthesis of α,β -Unsaturated Esters via the Wittig Reaction: A Highly Efficient and Recyclable Heterogeneous System

Rohinee D. Hoval¹, Santosh T. Shinde^{1*}, Anandrao A. Kale¹, Nanasaheb S. Gaikwad¹, Digambar B. Bankar³, Nitin M. Thorat⁴, Ramesh B. Gawade¹, Kaluram G. Kanade⁵, Dinesh P. Amalnerkar²

¹Post graduate Department and Research Centre of Chemistry, Annasaheb Awate College Manchar-410503, India.

²Pimpri Chinchwad University, Pune -412106, India.

³Post Graduate Department of Chemistry and Research Centre, R. B. Narayanrao Borawake College, Shrirampur (Autonomous), 413709, India.

⁴Post Graduate Department of Chemistry, and Research Centre, Maharaja Jivajirao Shinde Mahavidyalaya, Shrigonda, Ahilyanagar-413701, India.

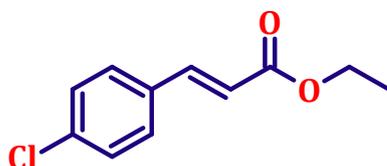
⁵Rajmata Jijau Shikshan Prasarak Mandal's Arts, Commerce & Science College, Landewadi, Bhosari, Pune-411039, India.

Corresponding Autor: Dr. Santosh T. Shinde, drsantoshinde@gmail.com

Supporting Data: ¹H-NMR and ¹³C-NMR spectra of as-synthesized of compounds.

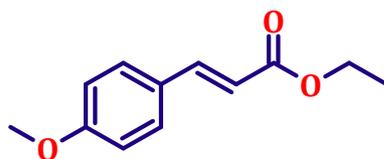
1) Nuclear Magnetic Resonance (NMR) of as-synthesized α,β -Unsaturated Esters (Table 6, entries 1 to 11):

i) Ethyl (E)-3-(4-chlorophenyl) acrylate (Entry 1, Table 6):



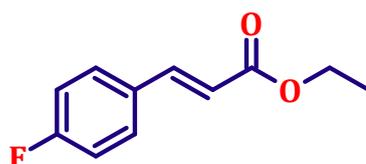
Light yellow, clear liquid; b.p. 138 °C. ¹H NMR (500 MHz, CDCl₃) δ 7.54 (d, J = 16.0 Hz, 1H), 7.37 (d, J = 8.5 Hz, 2H), 7.27 (d, J = 8.5 Hz, 2H), 6.31 (d, J = 16.0 Hz, 1H), 4.17 (q, J = 7.0 Hz, 2H), 1.25 (t, J = 7.0 Hz, 3H). ¹³C NMR (126 MHz, CDCl₃) δ 166.7, 143.1, 136.1, 133.0, 131.2, 128.2, 118.9, 60.7, 14.4.

ii) Ethyl (E)-3-(4-methoxyphenyl) acrylate (Entry 2, Table 6):



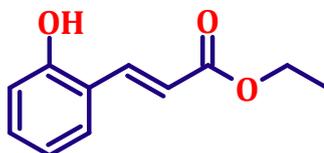
White crystalline powder; m.p. 52 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.55 (d, J = 16.0 Hz, 1H), 7.39 (d, J = 8.5 Hz, 2H), 6.82 (d, J = 8.5 Hz, 2H), 6.22 (d, J = 16.0 Hz, 1H), 4.16 (q, J = 7.5 Hz, 2H), 3.76 (s, 3H), 1.24 (t, J = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.4, 161.3, 144.2, 129.7, 127.2, 115.8, 114.4, 60.4, 55.4, 14.4.

iii) Ethyl (E)-3-(4-fluorophenyl) acrylate (Entry 3, Table 6):



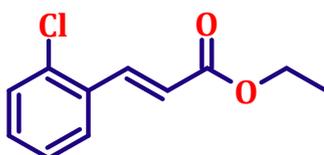
Colourless liquid; b.p. 288 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.56 (d, J = 16.0 Hz, 1H), 7.43–7.47 (m, 2H), 6.98–7.03 (m, 2H), 6.27 (d, J = 16.0 Hz, 1H), 4.17 (q, J = 7.0 Hz, 2H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.9, 162.9, 143.3, 130.8, 129.9, 118.1, 116.0, 60.6, 14.3.

iv) Ethyl (E)-3-(2-hydroxyphenyl) acrylate (Entry 4, Table 6):



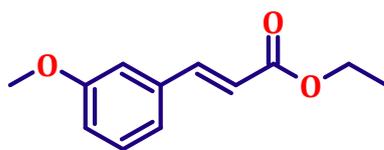
Off-white crystalline powder; m.p. 86 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.92 (d, J = 16.0 Hz, 1H), 7.39 (dd, J = 7.5, 1.3 Hz, 1H), 7.14–7.19 (m, 1H), 6.83 (t, J = 7.5 Hz, 1H), 6.76 (d, J = 8.0 Hz, 1H), 6.52 (d, J = 16.0 Hz, 1H), 4.19 (q, J = 7.0 Hz, 2H), 1.26 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 168.5, 157.1, 140.1, 131.3, 129.2, 121.9, 120.9, 118.9, 116.3, 60.5, 14.4.

v) Ethyl (E)-3-(2-chlorophenyl) acrylate (Entry 5, Table 6):



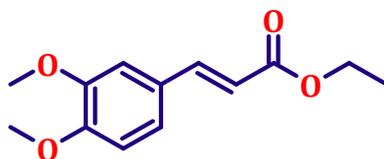
Pale yellow liquid; b.p. 305 °C. ^1H NMR (500 MHz, CDCl_3) δ 8.00 (d, J = 16.0 Hz, 1H), 7.54 (dd, J = 7.5, 2.0 Hz, 1H), 7.34 (dd, J = 7.5, 2.0 Hz, 1H), 7.15–7.26 (m, 2H), 6.34 (d, J = 16.0 Hz, 1H), 4.19 (q, J = 7.0 Hz, 2H), 1.26 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.5, 140.4, 134.9, 132.8, 130.2, 129.1, 127.1, 122.1, 121.0, 60.7, 14.3.

vi) Ethyl (E)-3-(3-methoxyphenyl) acrylate (Entry 6, Table 6):



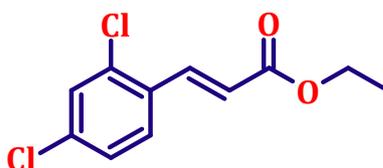
Pale yellow liquid; b.p. 145 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.56 (d, J = 16.0 Hz, 1H), 7.16–7.24 (m, 1H), 7.04 (d, J = 7.8 Hz, 1H), 6.97 (t, J = 2.0 Hz, 1H), 6.85 (dd, J = 8.0 Hz, 1H), 6.34 (d, J = 16.0 Hz, 1H), 4.17 (q, J = 7.0 Hz, 2H), 3.86 (s, 3H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.0, 159.9, 144.5, 135.9, 129.9, 120.8, 118.6, 116.1, 112.9, 60.5, 55.3, 14.3.

vii) Ethyl (E)-3-(3,4-dimethoxyphenyl) acrylate (Entry 7, Table 6):



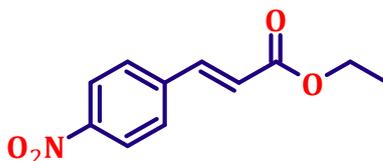
White crystalline powder; m.p. 52 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.54 (d, J = 16.0 Hz, 1H), 7.02 (dd, J = 8.0, 2.0 Hz, 1H), 6.98 (d, J = 2.0 Hz, 1H), 6.78 (d, J = 8.0 Hz, 1H), 6.22 (d, J = 16.0 Hz, 1H), 4.16 (q, J = 7.0 Hz, 2H), 3.84 (s, 6H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.3, 151.2, 149.2, 144.5, 127.5, 122.5, 116.0, 111.1, 109.6, 60.3, 55.9, 14.4.

viii) Ethyl (E)-3-(2,4-dichlorophenyl) acrylate (Entry 8, Table 6):



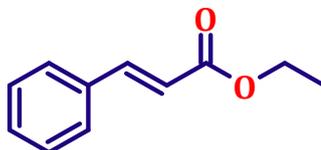
White crystalline powder; m.p. 85 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.91 (d, J = 16.0 Hz, 1H), 7.46 (d, J = 8.5 Hz, 1H), 7.36 (d, J = 2.0 Hz, 1H), 7.18–7.20 (m, 1H), 6.32 (d, J = 16.0 Hz, 1H), 4.18 (q, J = 7.0 Hz, 2H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.2, 139.1, 136.3, 135.5, 131.4, 130.0, 128.4, 127.5, 121.4, 60.7, 14.2.

ix) Ethyl (E)-3-(4-nitrophenyl) acrylate (Entry 9, Table 6):



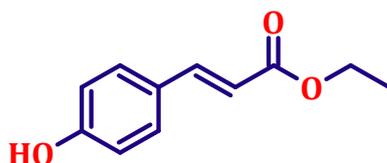
Off-white crystalline powder; m.p. 138 °C. ^1H NMR (500 MHz, CDCl_3) δ 8.17 (d, J = 8.5 Hz, 2H), 7.62 (d, J = 16.0 Hz, 1H), 7.59 (d, J = 8.5 Hz, 2H), 6.47 (d, J = 16.0 Hz, 1H), 4.20 (q, J = 7.0 Hz, 2H), 1.27 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 166.0, 148.5, 141.6, 140.6, 128.6, 124.2, 122.6, 61.0, 14.3.

x) Ethyl cinnamate (Entry 10, Table 6):



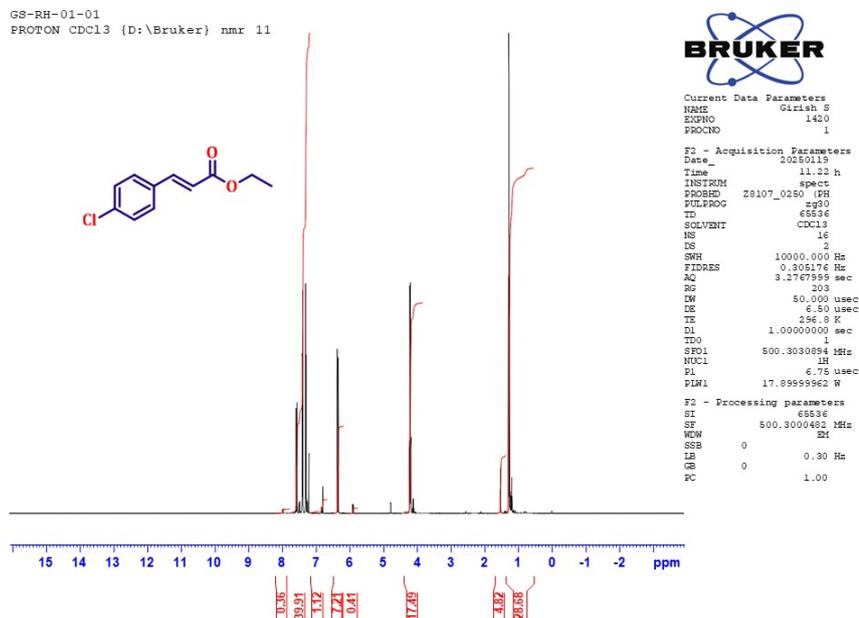
Pale yellow liquid; b.p. 272 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.60 (d, J = 16.0 Hz, 1H), 7.44 (dd, J = 8.0, 2.0 Hz, 2H), 7.29–7.33 (m, 3H), 6.35 (d, J = 16.0 Hz, 1H), 4.17 (q, J = 7.0 Hz, 2H), 1.25 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 167.0, 144.6, 134.5, 130.2, 128.9, 128.1, 118.3, 60.5, 14.3.

xi) Ethyl (E)-3-(4-hydroxyphenyl) acrylate (Entry 11, Table 6):

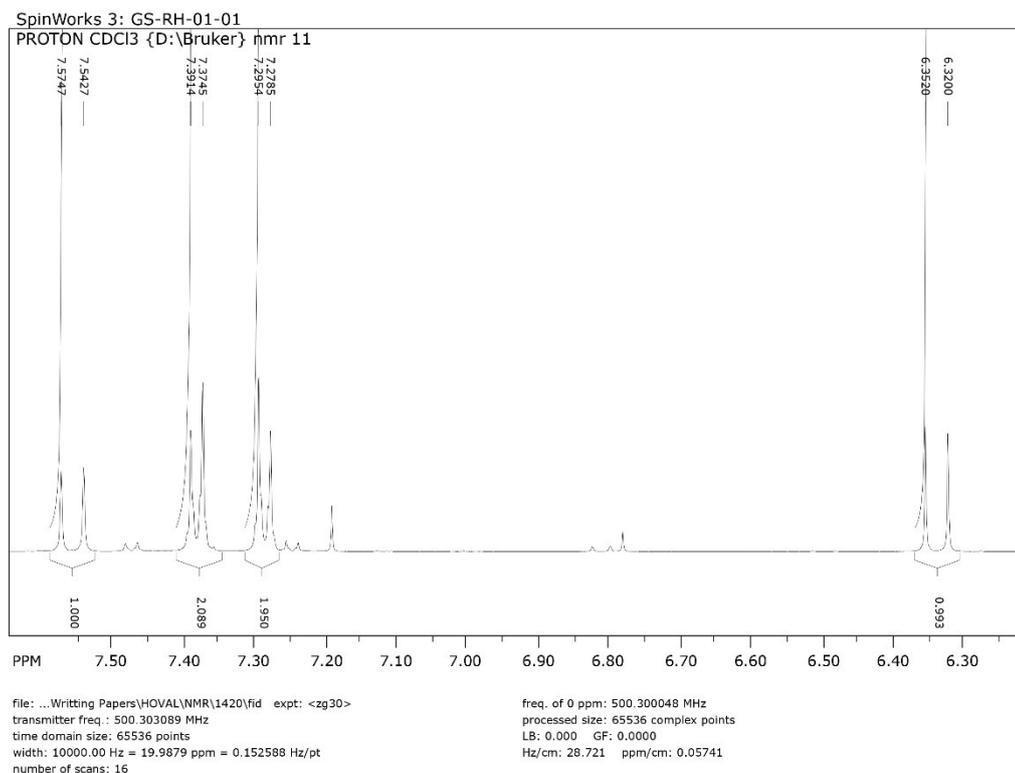


White crystalline powder; m.p. 48 °C. ^1H NMR (500 MHz, CDCl_3) δ 7.54 (d, J = 16.0 Hz, 1H), 7.39 (d, J = 8.5 Hz, 2H), 6.82 (d, J = 8.5 Hz, 2H), 6.23 (d, J = 16.0 Hz, 1H), 4.57 (s, 1H), 4.16 (q, J = 7.0 Hz, 2H), 1.21 (t, J = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, CDCl_3) δ 168.5, 159.4, 143.9, 129.7, 128.3, 116.5, 115.0, 60.4, 14.2.

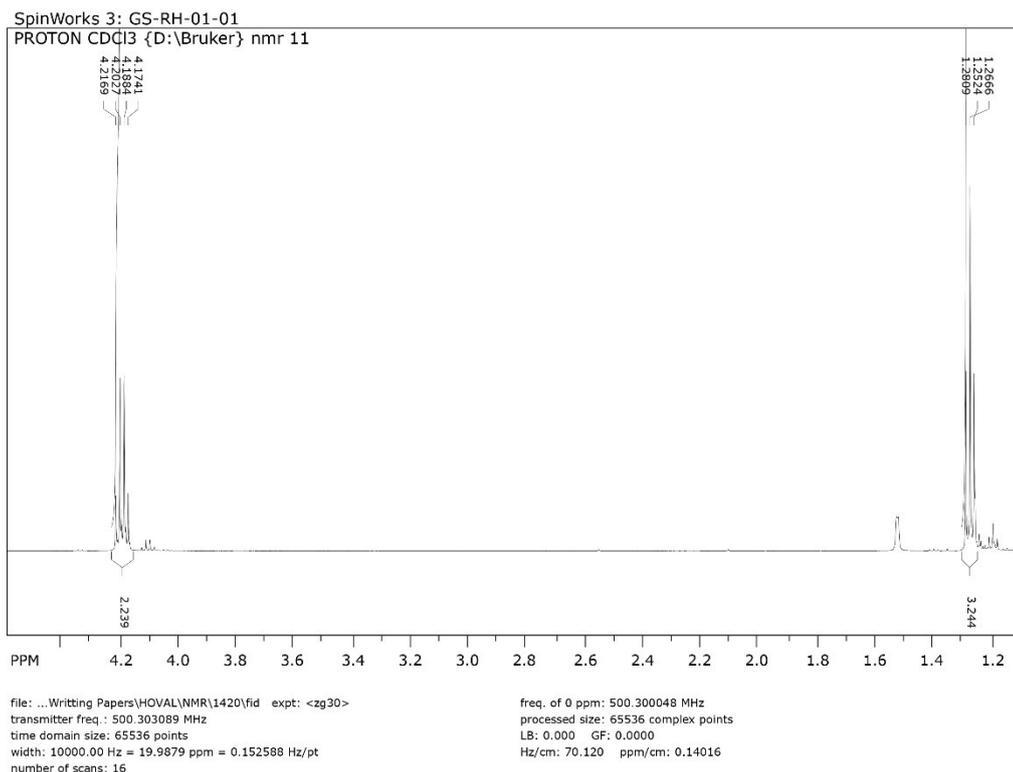
The structures of the synthesized representative compounds (Table 6, entries 1–11) were confirmed by ^1H NMR and ^{13}C NMR spectroscopy. The complete spectra, along with their expanded regions, are provided below. Information.



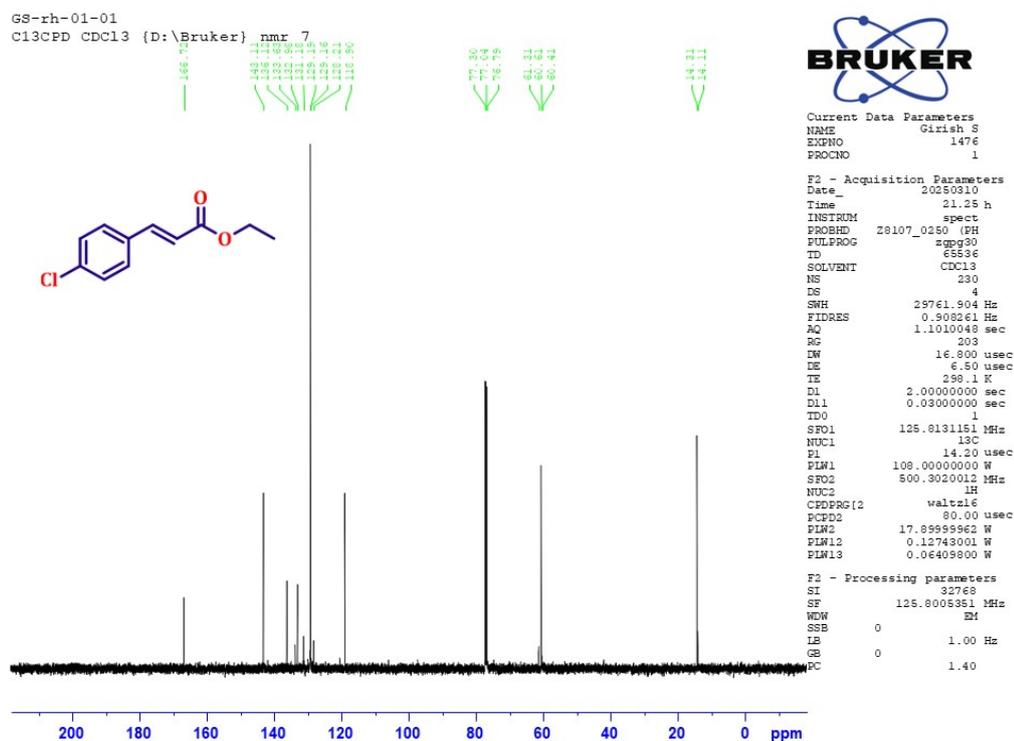
^1H -NMR spectrum of Ethyl (*E*)-3-(4-chlorophenyl)acrylate (Entry 1, Table 6)



^1H -NMR spectrum of Ethyl (*E*)-3-(4-chlorophenyl)acrylate (Entry 1, Table 6, Expansion 1)

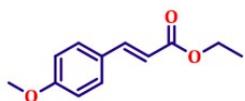


**¹H-NMR spectrum of Ethyl (*E*)-3-(4-chlorophenyl)acrylate
 (Entry 1, Table 6, Expansion 2)**



**¹³C-NMR spectrum of Ethyl (*E*)-3-(4-chlorophenyl)acrylate
 (Entry 1, Table 6)**

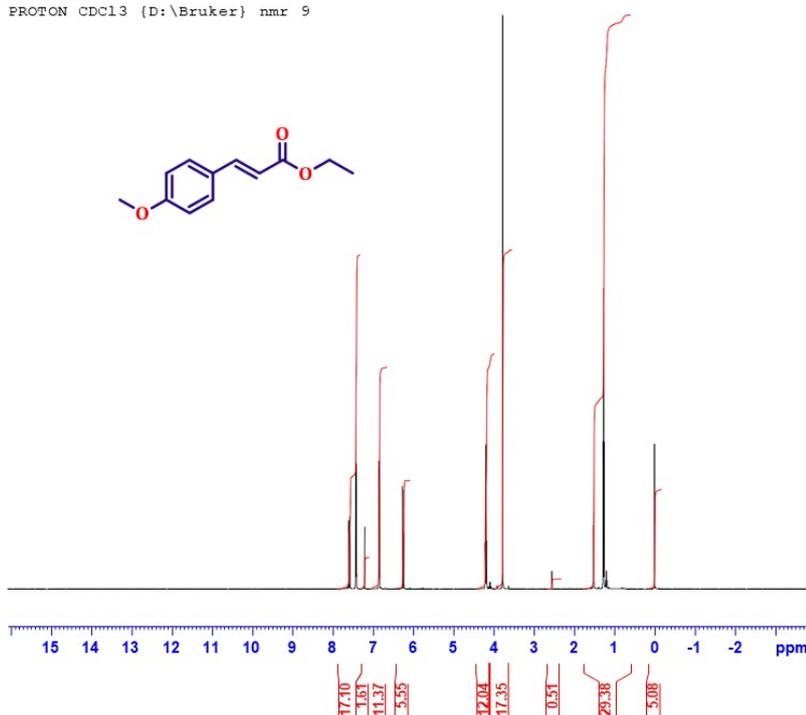
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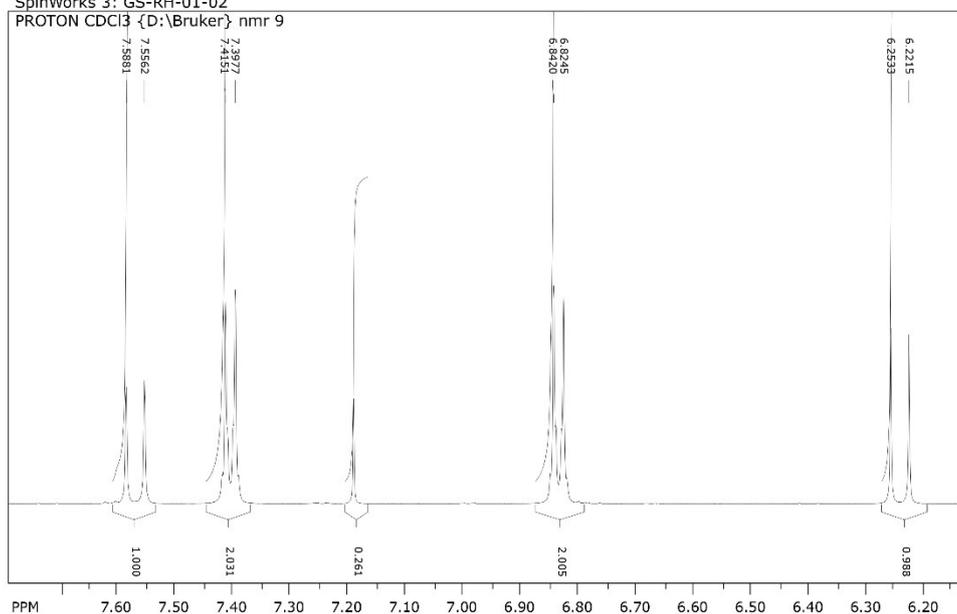
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FIDRES 0.305174 Hz
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RG 203
DW 50.000 usec
DE 6.50 usec
TE 298.3 K
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TD0 1
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NUC1 H
P1 6.75 usec
PLM1 17.89899962 W

F2 - Processing parameters
SI 65536
SF 500.3000481 MHz
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GB 0
PC 1.00



**¹H-NMR spectrum of Ethyl (*E*)-3-(4-methoxyphenyl)acrylate
(Entry 2, Table 6)**

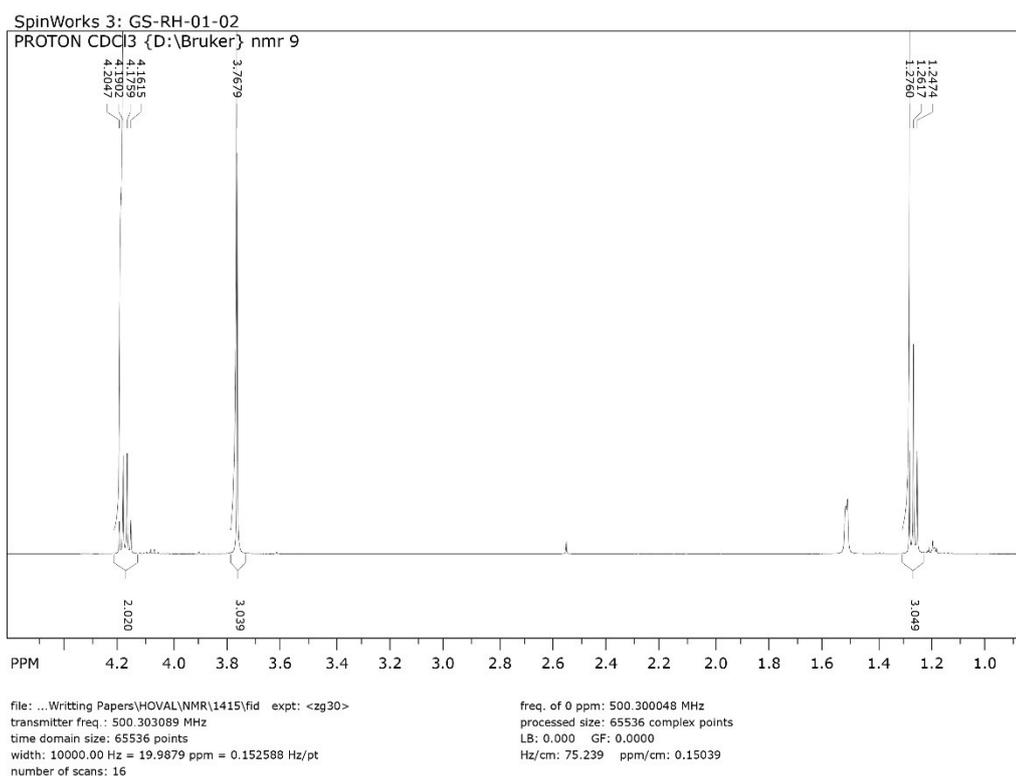
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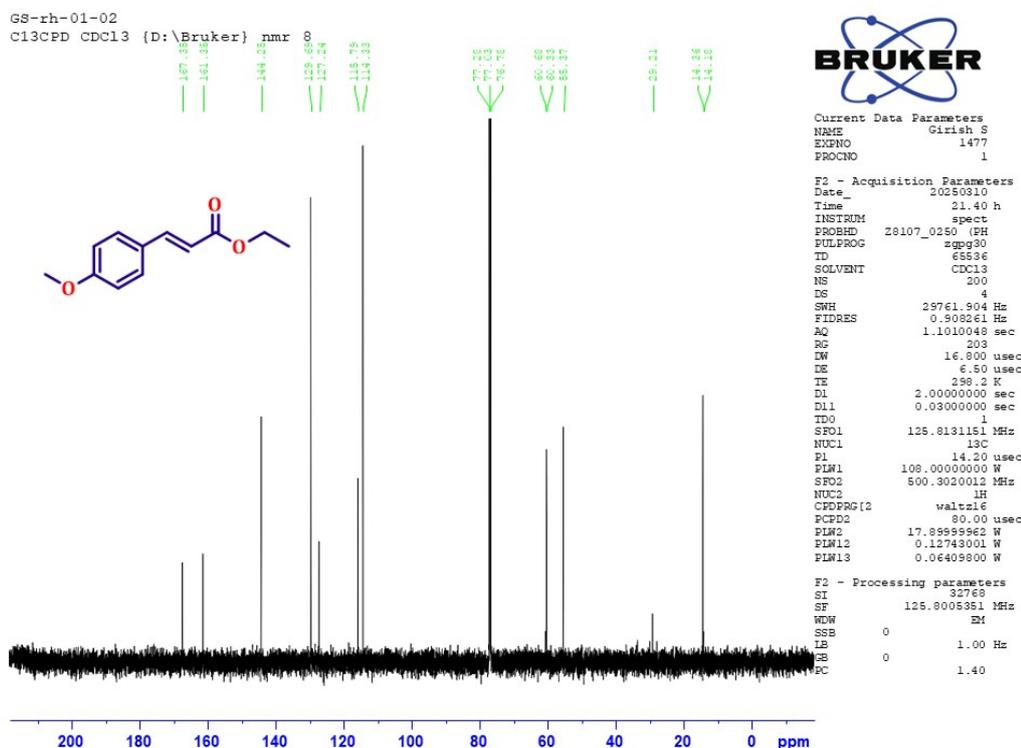
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number of scans: 16

freq. of 0 ppm: 500.300048 MHz
processed size: 65536 complex points
LB: 0.000 GF: 0.0000
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**¹H-NMR spectrum of Ethyl (*E*)-3-(4-methoxyphenyl)acrylate
(Entry 2, Table 6, Expansion 1)**

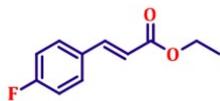


**¹H-NMR spectrum of Ethyl (*E*)-3-(4-methoxyphenyl)acrylate
 (Entry 2, Table 6, Expansion 2)**



**¹³C NMR spectrum of Ethyl (*E*)-3-(4-methoxyphenyl)acrylate
 (Entry 2, Table 6)**

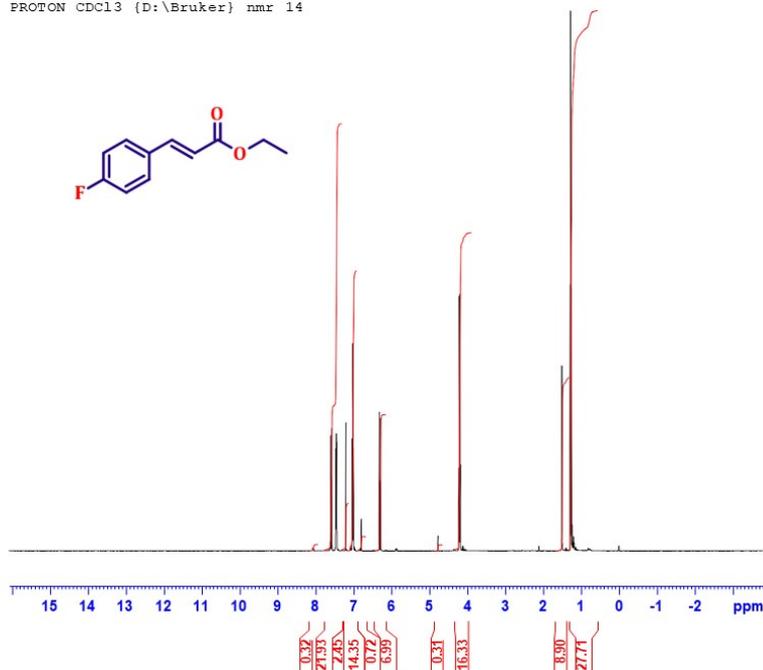
GS-RH-01-03
PROTON CDCl3 {D:\Bruker} nmr 14



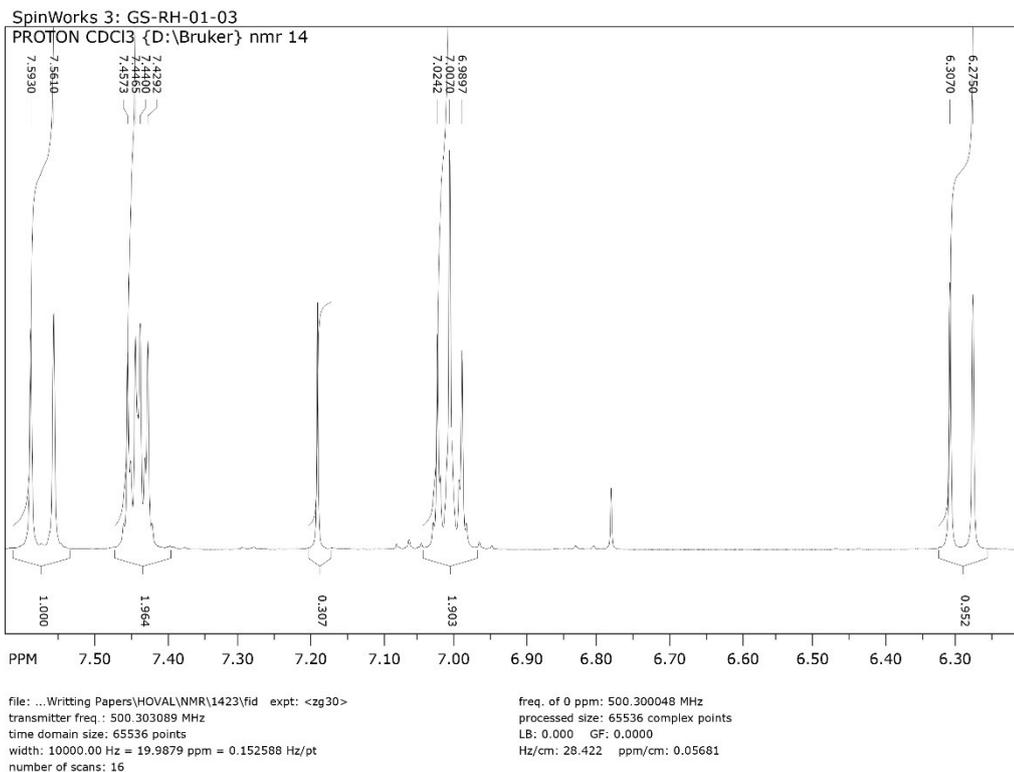
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RG 203
DM 50.000 usec
DE 6.50 usec
IE 256.7 K
FI 1.00000000 sec
TDO 1
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NUC1 1H
EI 6.75 usec
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GB 0
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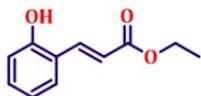


1H-NMR spectrum of Ethyl (*E*)-3-(4-fluorophenyl)acrylate (Entry 3, Table 6)



1H-NMR spectrum of Ethyl (*E*)-3-(4-fluorophenyl)acrylate (Entry 3, Table 6, Expansion 1)

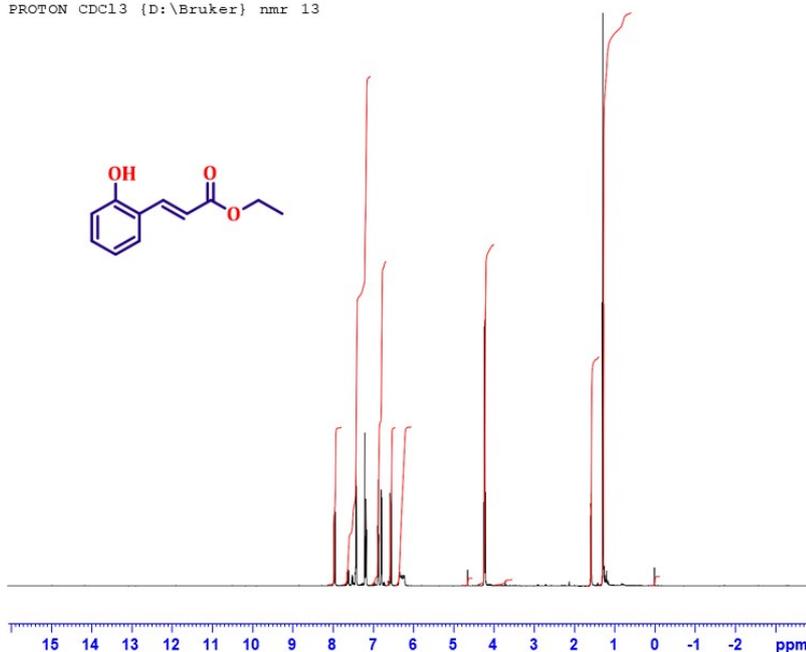
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PROTON CDCl3 {D:\Bruker} nmr 13



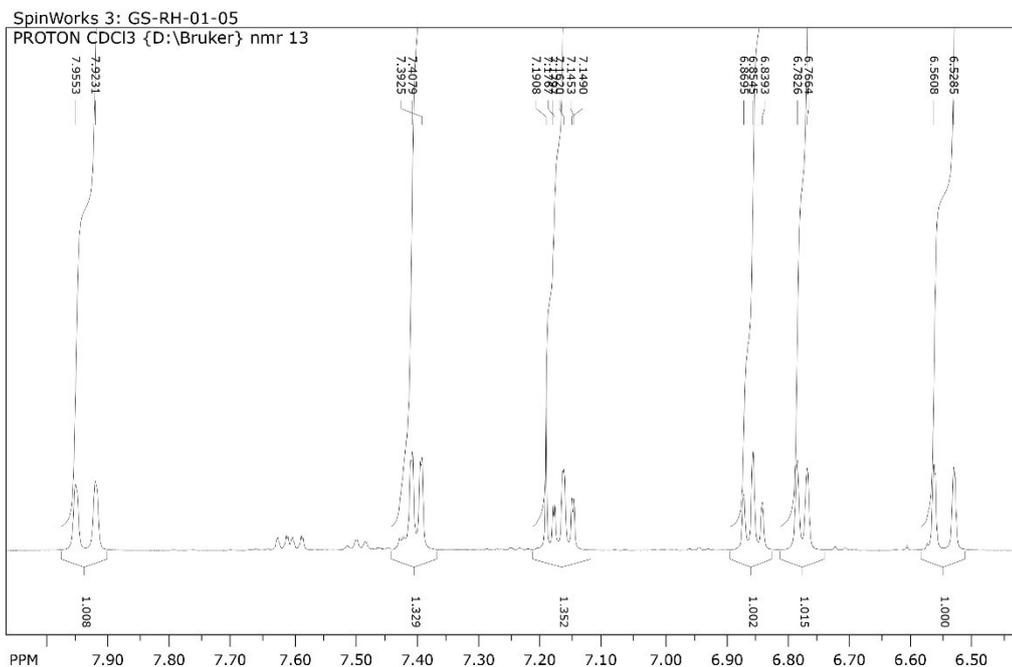
Current Data Parameters
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EXPNO 1422
PROCNO 1

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DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767899 sec
RG 203
LW 50.000 usec
DE 6.50 usec
TE 296.3 K
DL 1.00000000 sec
TDO 1
SFO1 500.3030894 MHz
NUC1 1H
P1 6.75 usec
PLW1 17.89999962 W

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GB 0
PC 1.00



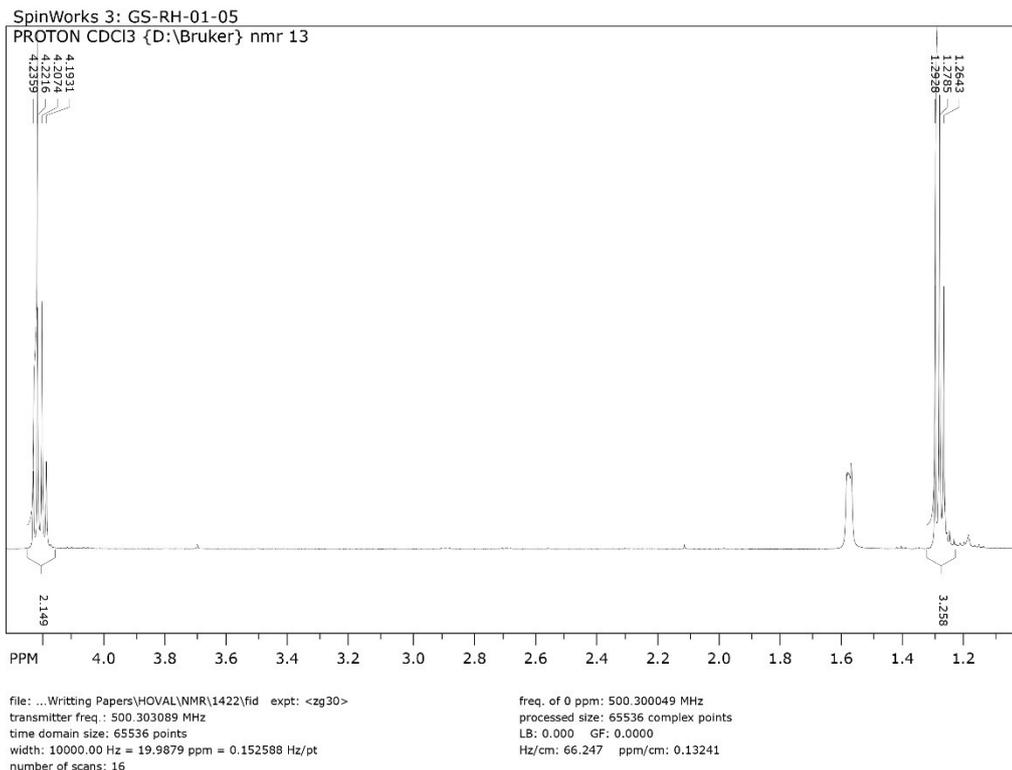
1H-NMR spectrum of Ethyl (*E*)-3-(2-hydroxyphenyl)acrylate (Entry 4, Table 6)



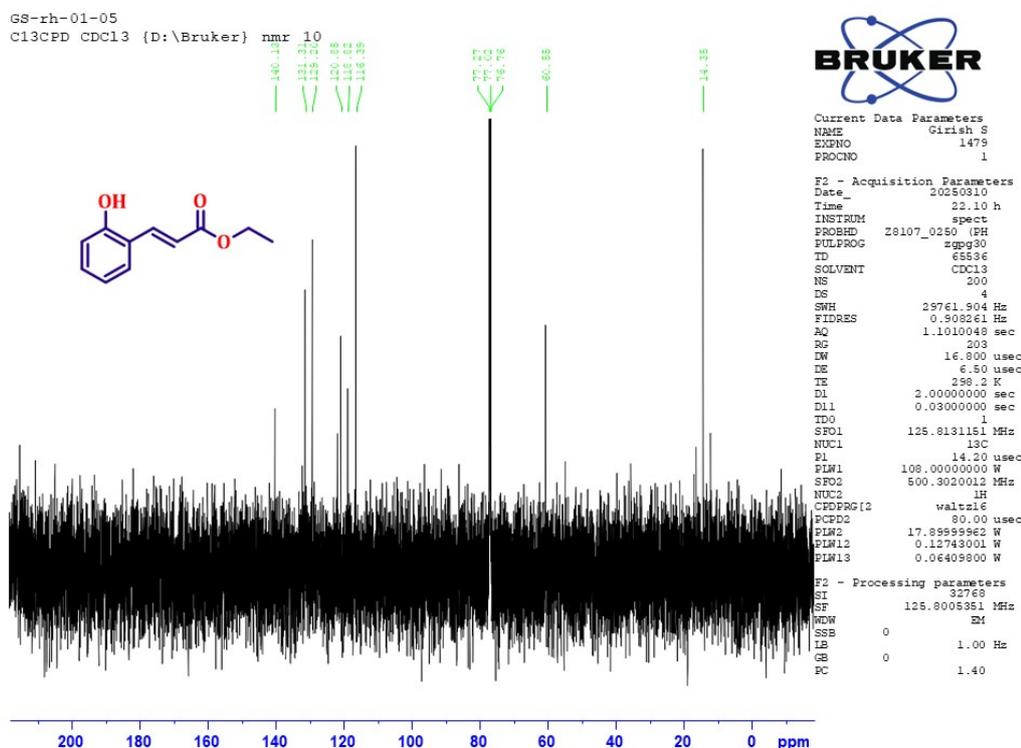
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time domain size: 65536 points
width: 10000.00 Hz = 19.9879 ppm = 0.152588 Hz/pt
number of scans: 16

freq. of 0 ppm: 500.300049 MHz
processed size: 65536 complex points
LB: 0.000 GF: 0.0000
Hz/cm: 32.984 ppm/cm: 0.06593

1H-NMR spectrum of Ethyl (*E*)-3-(2-hydroxyphenyl)acrylate (Entry 4, Table 6, Expansion 1)

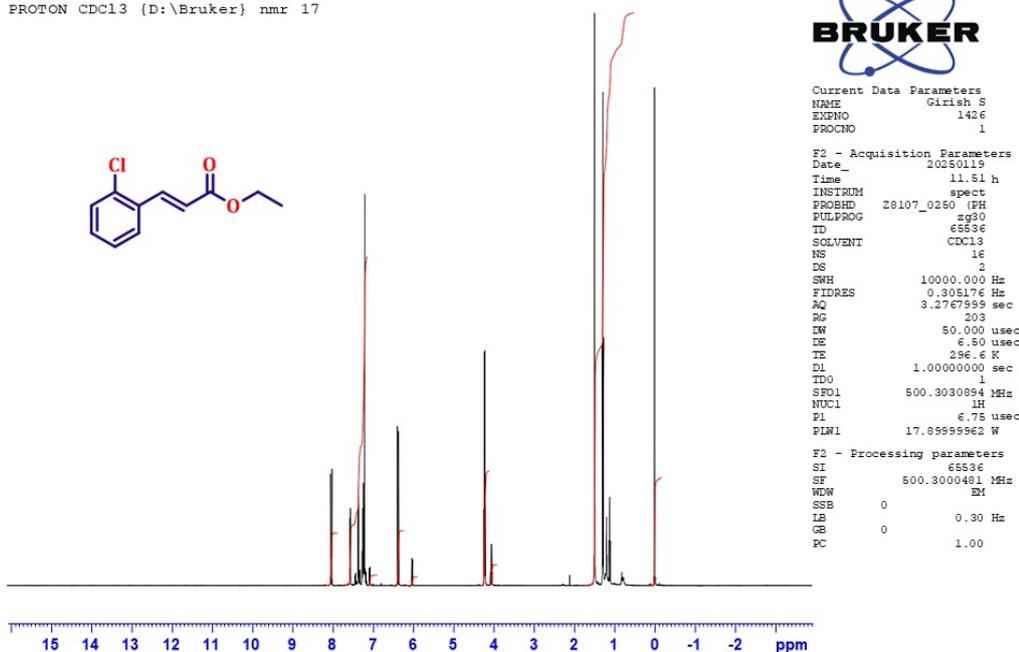
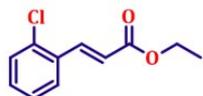


1H-NMR spectrum of Ethyl (*E*)-3-(2-hydroxyphenyl)acrylate (Entry 4, Table 6, Expansion 2)

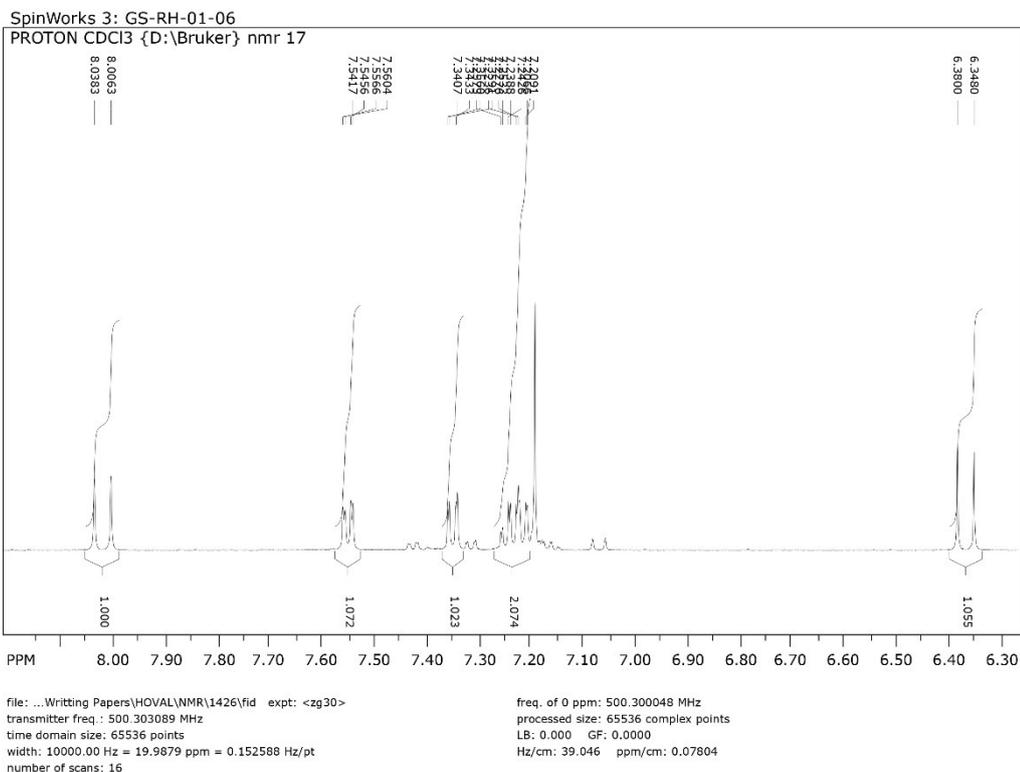


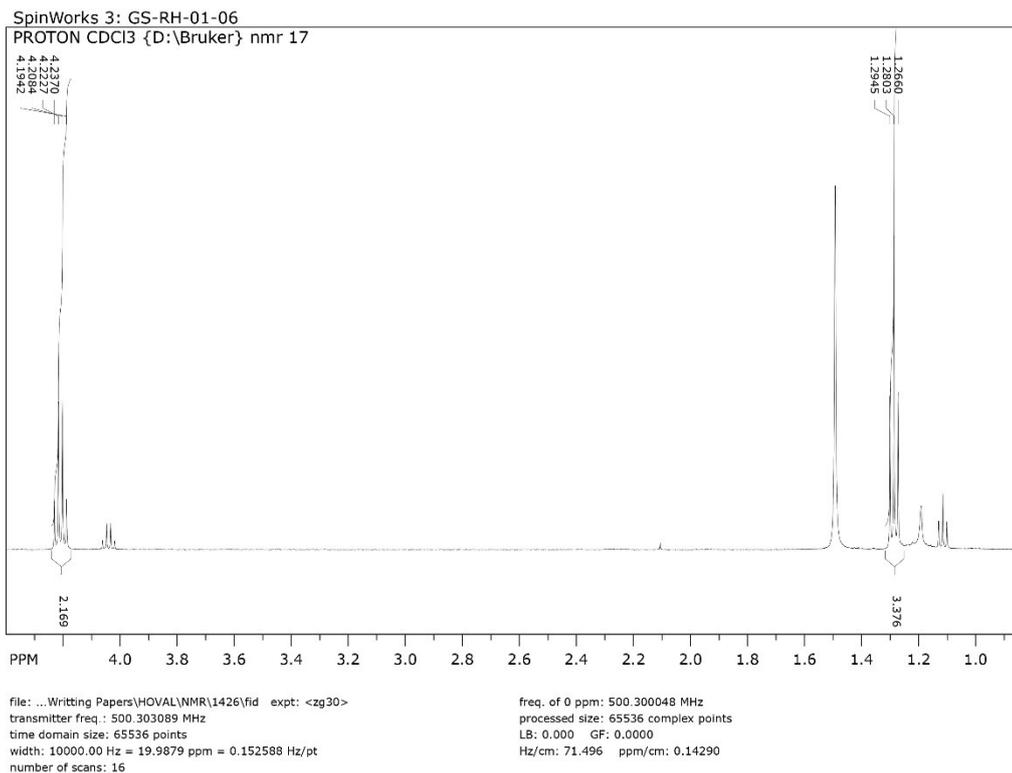
13C NMR spectrum of Ethyl (*E*)-3-(2-hydroxyphenyl)acrylate (Entry 4, Table 6)

GS-RH-01-06
PROTON CDCl3 {D:\Bruker} nmr 17

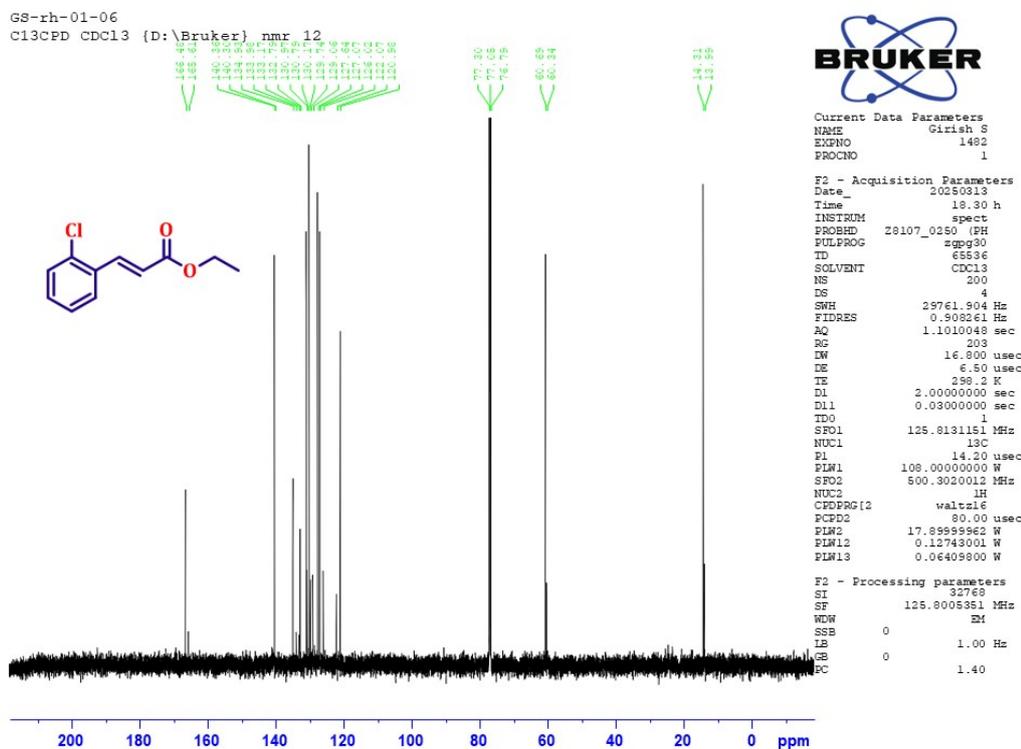


**1H-NMR spectrum of Ethyl (*E*)-3-(2-chlorophenyl)acrylate
(Entry 5, Table 6)**



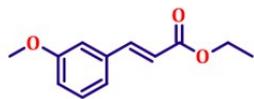


**1H-NMR spectrum of Ethyl (*E*)-3-(2-chlorophenyl)acrylate
 (Entry 5, Table 6, Expansion 2)**



**13C NMR spectrum of Ethyl (*E*)-3-(2-chlorophenyl)acrylate
 (Entry 5, Table 6)**

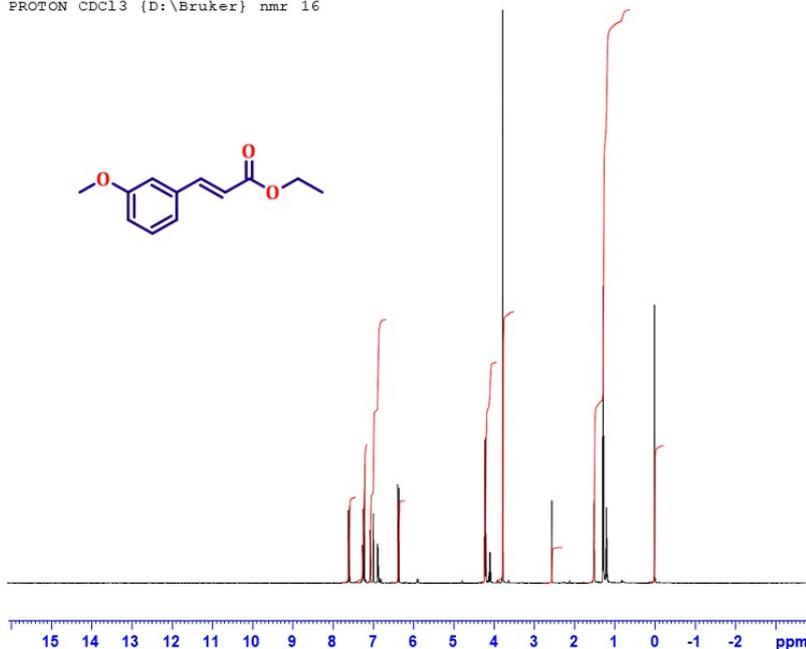
GS-RH-01-08
PROTON CDCl3 {D:\Bruker} nmr 16



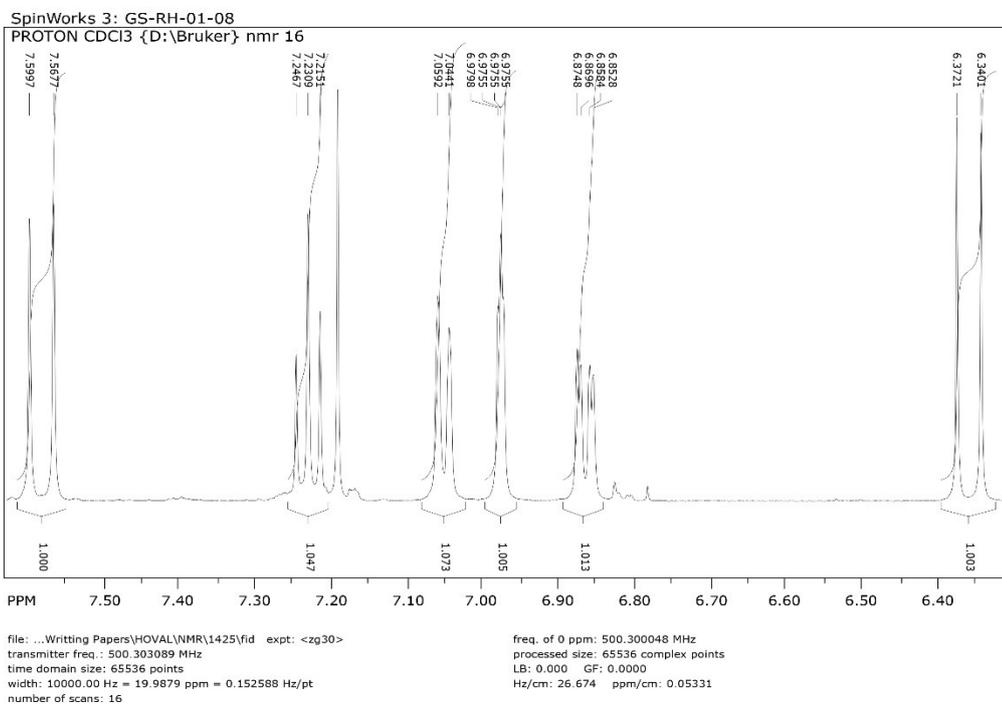
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NAME Girish S
EXPNO 1425
PROCNO 1

F2 - Acquisition Parameters
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TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767899 sec
RG 203
LW 50.000 usec
DE 6.50 usec
TE 298.2 K
DL 1.00000000 sec
TDO 1
SFO1 500.3030894 MHz
NUC1 H
P1 6.75 usec
PLW1 17.89999962 W

F2 - Processing parameters
SI 65536
SF 500.3000481 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



**1H-NMR spectrum of Ethyl (*E*)-3-(3-methoxyphenyl)acrylate
(Entry 6, Table 6)**



**1H-NMR spectrum of Ethyl (*E*)-3-(3-methoxyphenyl)acrylate
(Entry 6, Table 6, Expansion 1)**

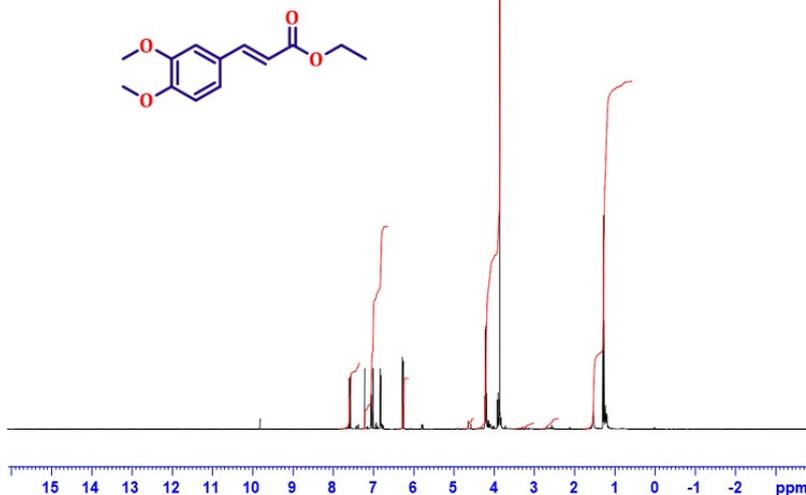
GS-RH-01-09
PROTON CDCl3 {D:\Bruker} nmr 15



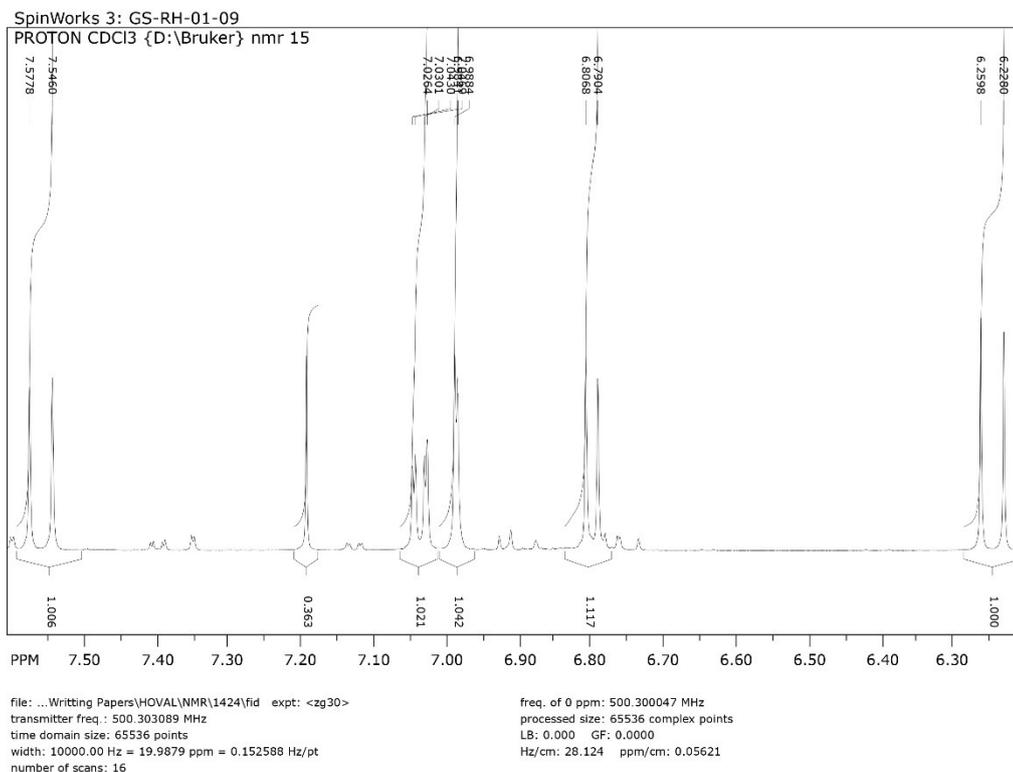
Current Data Parameters
NAME Girish S
EXPNO 1424
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250119
Time 11.41 h
INSTRUM spect
PROBHD Z8107_0250 (PH
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 203
LW 50.000 usec
DE 6.50 usec
TE 296.2 K
DL 1.00000000 sec
TDO 1
SFO1 500.3030894 MHz
NUC1 1H
P1 6.75 usec
PLW1 17.89999962 W

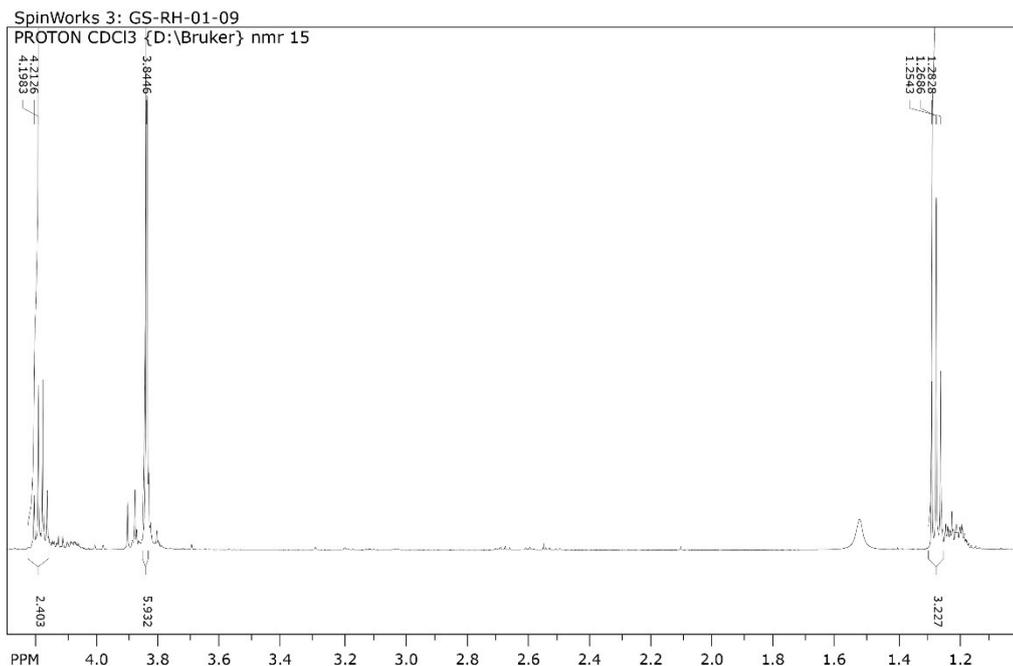
F2 - Processing parameters
SI 65536
SF 500.3000469 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



**1H-NMR spectrum of Ethyl (*E*)-3-(3,4-dimethoxyphenyl)acrylate
(Entry 7, Table 6)**



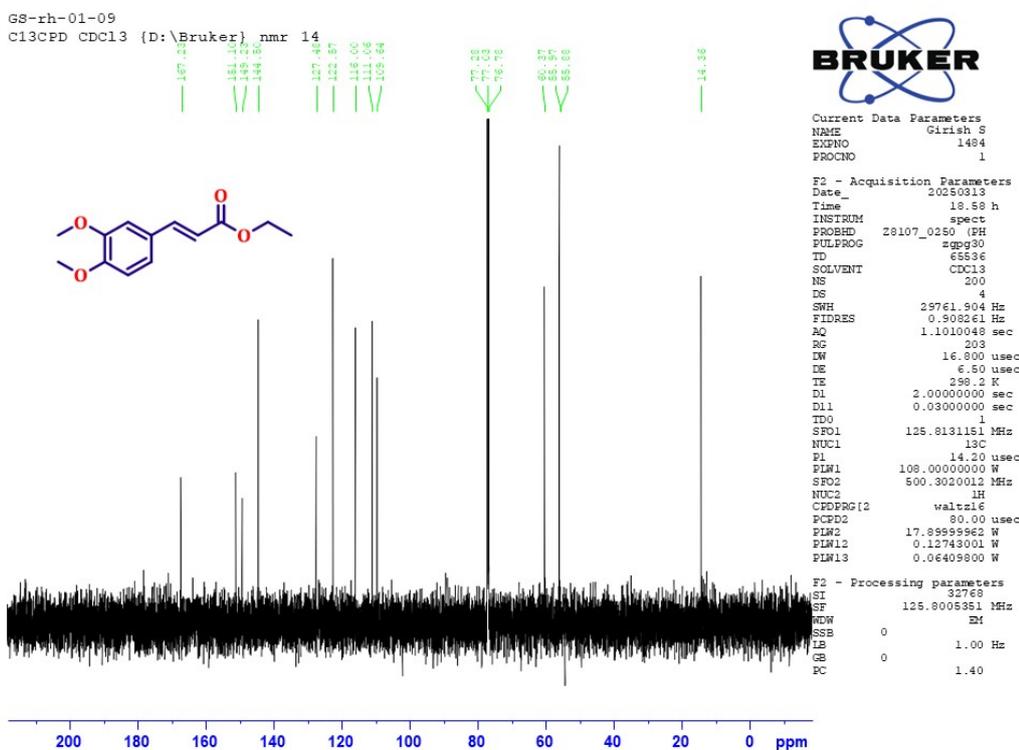
**1H-NMR spectrum of Ethyl (*E*)-3-(3,4-dimethoxyphenyl)acrylate
(Entry 7, Table 6, Expansion 1)**



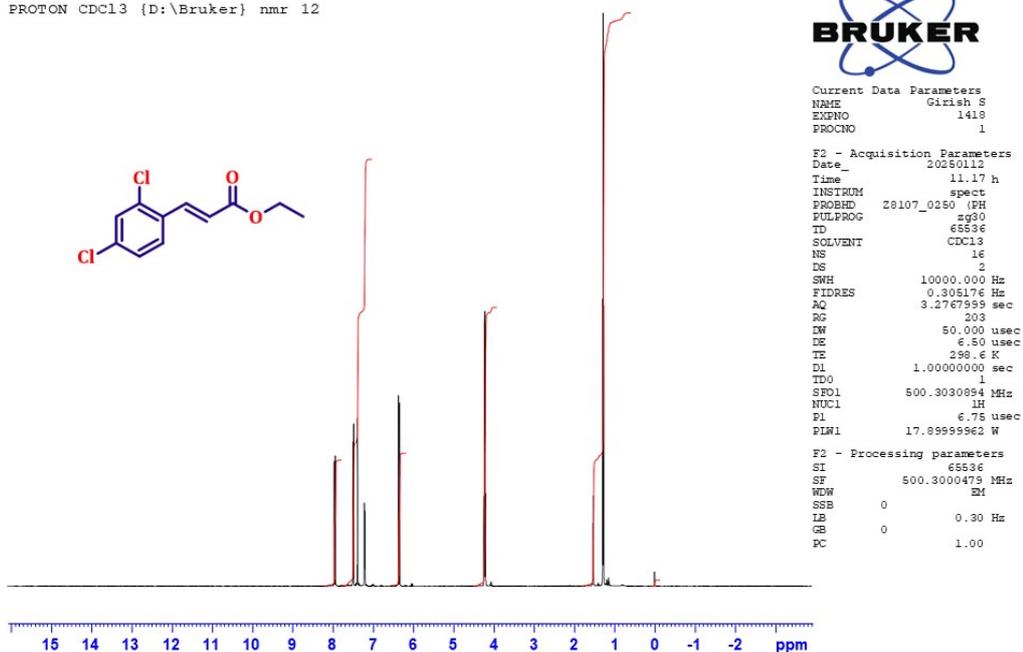
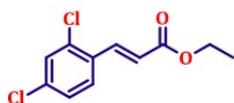
file: ...Writing Papers\HOVAL\NMR\1424\fid exp: <zg30>
 transmitter freq.: 500.303089 MHz
 time domain size: 65536 points
 width: 10000.00 Hz = 19.9879 ppm = 0.152588 Hz/pt
 number of scans: 16

freq. of 0 ppm: 500.300047 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 66.247 ppm/cm: 0.13241

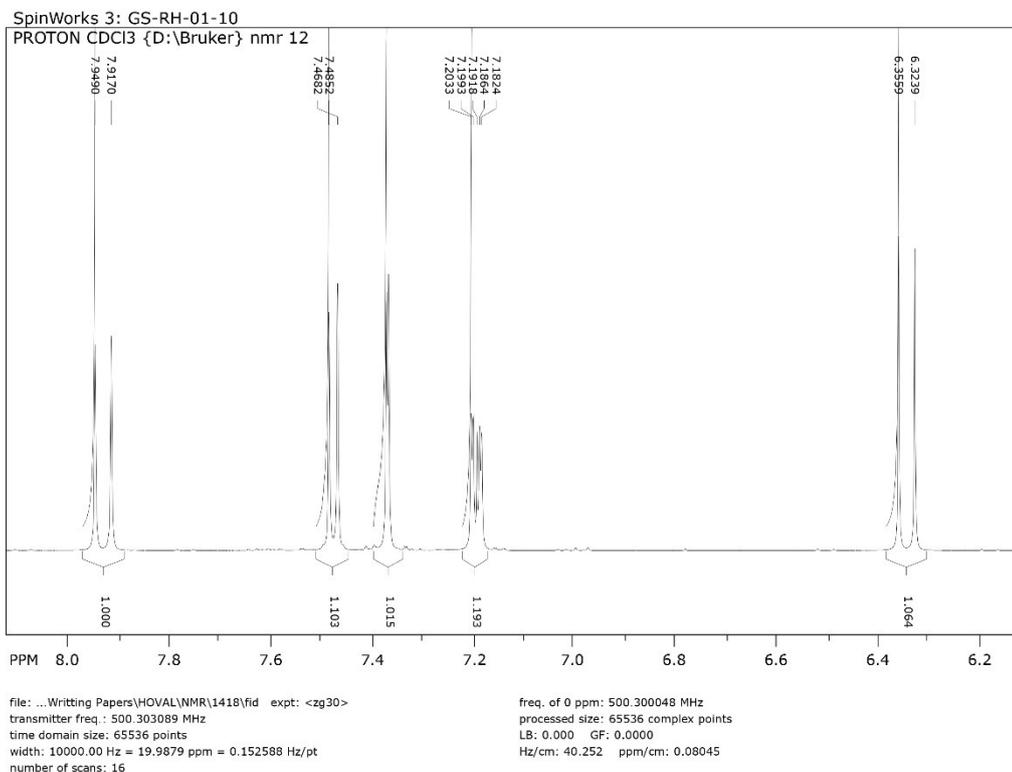
1H-NMR spectrum of Ethyl (*E*)-3-(3,4-dimethoxyphenyl)acrylate (Entry 7, Table 6, Expansion 2)



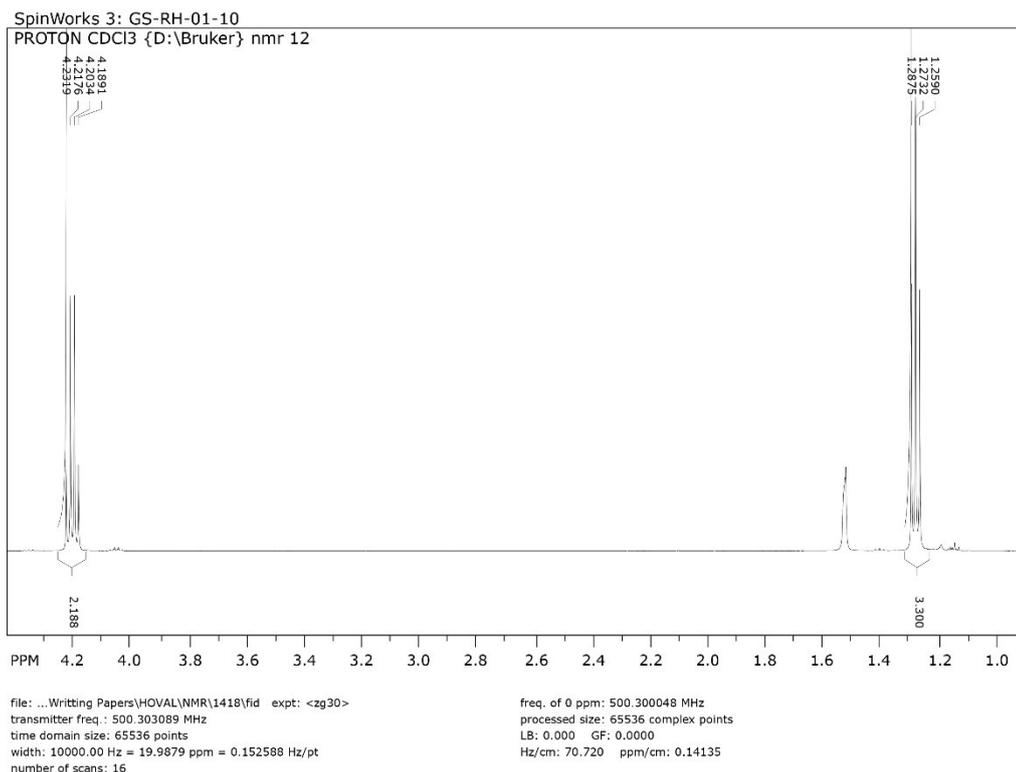
GS-RH-01-10
PROTON CDCl3 {D:\Bruker} nmr 12



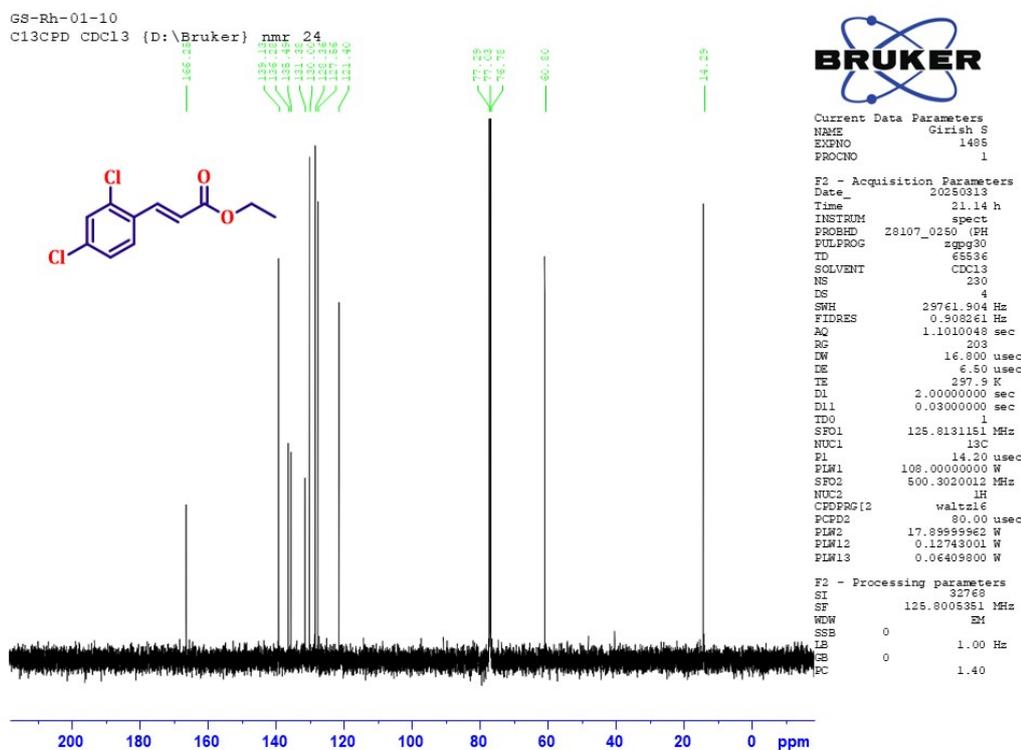
**1H-NMR spectrum of Ethyl (*E*)-3-(2,4-dichlorophenyl)acrylate
(Entry 8, Table 6)**



**1H-NMR spectrum of Ethyl (*E*)-3-(2,4-dichlorophenyl)acrylate
(Entry 8, Table 6, Expansion 1)**

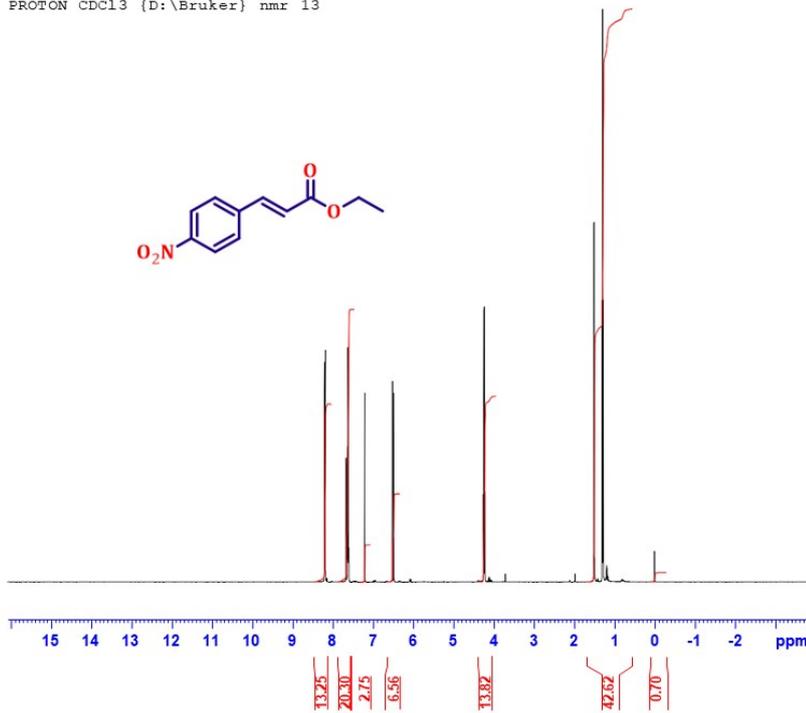
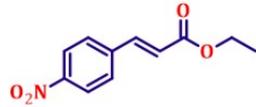


**¹H-NMR spectrum of Ethyl (*E*)-3-(2,4-dichlorophenyl)acrylate
 (Entry 8, Table 6, Expansion 2)**



**¹³C NMR spectrum of Ethyl (*E*)-3-(2,4-dichlorophenyl)acrylate
 (Entry 8, Table 6)**

GS-RH-01-11
PROTON CDCl3 {D:\Bruker} nmr 13

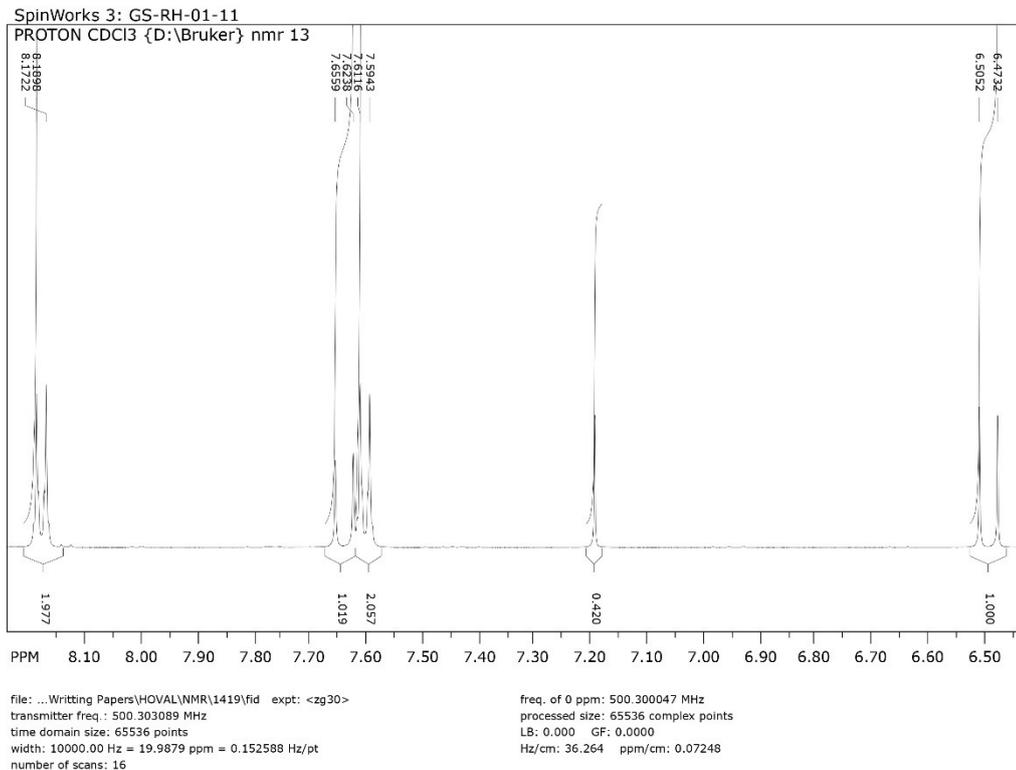


Current Data Parameters
NAME Girish S
EXPNO 1419
PROCNO 1

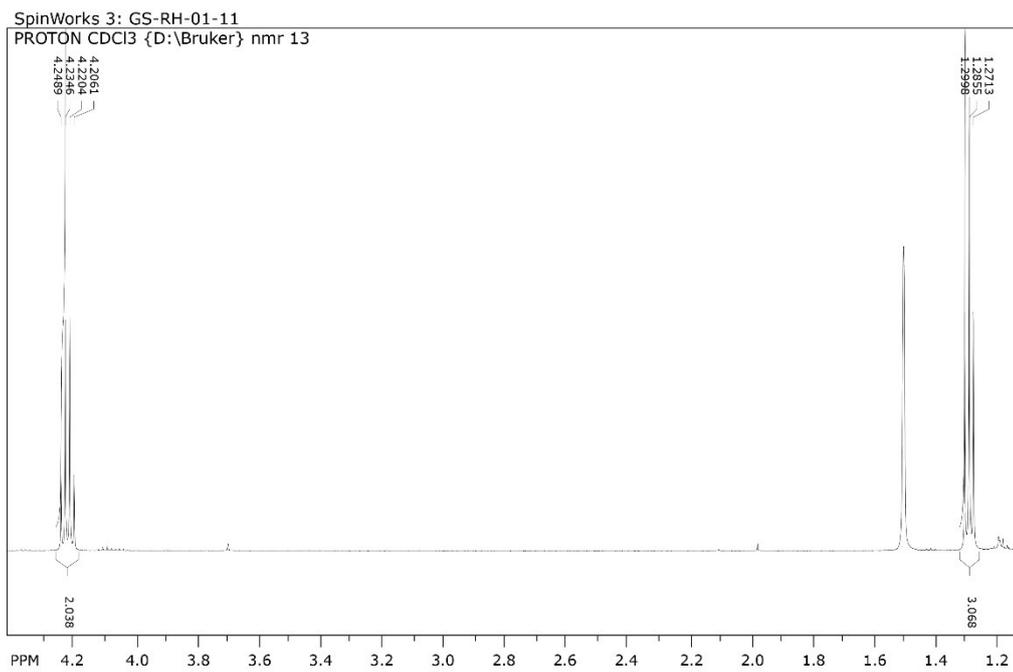
F2 - Acquisition Parameters
Date_ 20250112
Time 11.22 h
INSTRUM spect
PROBHD zgpg30
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 503
IN 50.000 usec
DE 6.50 usec
TE 298.7 K
D1 1.00000000 sec
TD0 1
SFO1 500.3030894 MHz
NUC1 1H
P1 6.75 usec
PLW1 17.89999962 W

F2 - Processing parameters
SI 65536
SF 500.3000475 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

1H-NMR spectrum of Ethyl (E)-3-(4-nitrophenyl)acrylate (Entry 9, Table 6)



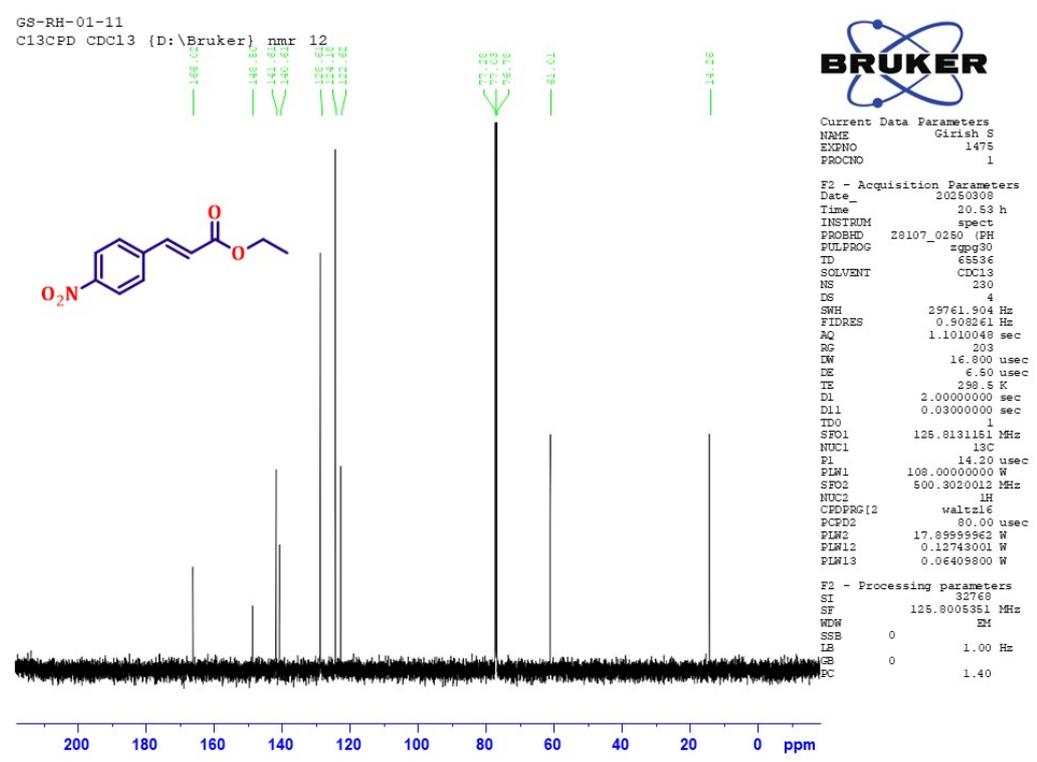
1H-NMR spectrum of Ethyl (E)-3-(4-nitrophenyl)acrylate (Entry 9, Table 6, Expansion 1)



file: ...Writing Papers\HOVAL\NMR\1419\fid exp: <zg30>
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 time domain size: 65536 points
 width: 10000.00 Hz = 19.9879 ppm = 0.152588 Hz/pt
 number of scans: 16

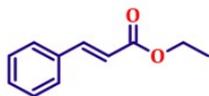
freq. of 0 ppm: 500.300047 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 66.247 ppm/cm: 0.13241

1H-NMR spectrum of Ethyl (*E*)-3-(4-nitrophenyl)acrylate (Entry 9, Table 6, Expansion 2)



13C NMR spectrum of Ethyl (*E*)-3-(4-nitrophenyl)acrylate (Entry 9, Table 6)

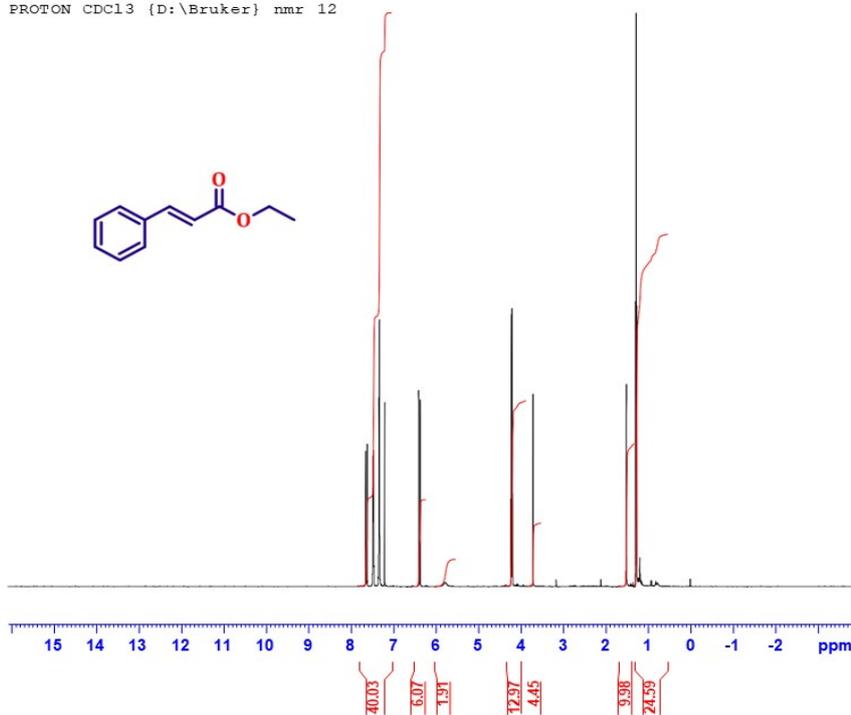
GS-RH-01-12
 PROTON CDCl3 {D:\Bruker} nmr 12



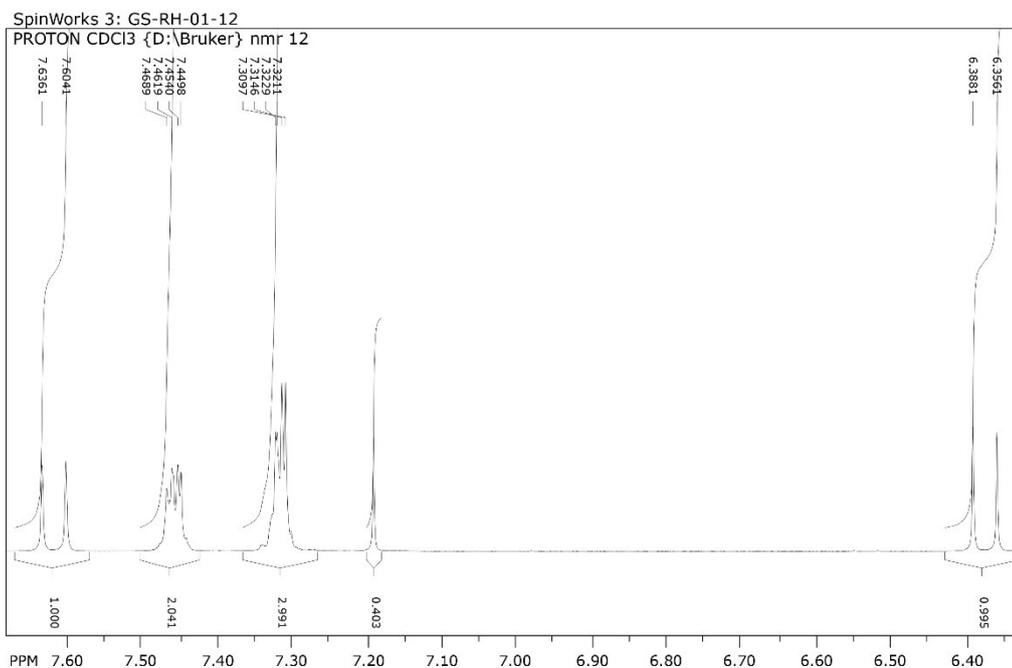
Current Data Parameters
 NAME Girish S
 EXPNO 1421
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 20250119
 Time 11.27 h
 INSTRUM spect
 PROBHD 28107_0250 (PH
 PULPROG zg30
 TD 65536
 SOLVENT CDCl3
 NS 16
 DS 2
 SWH 10000.000 Hz
 FIDRES 0.305176 Hz
 AQ 3.2767959 sec
 RG 203
 INW 50.000 usec
 DE 6.50 usec
 TE 296.8 K
 D1 1.00000000 sec
 TDO 1
 SFO1 500.3030894 MHz
 NUC1 1H
 P1 6.75 usec
 PLW1 17.89999562 W

F2 - Processing parameters
 SI 65536
 SF 500.3000461 MHz
 WDW EM
 SSB 0
 LB 0.30 Hz
 GB 0
 PC 1.00



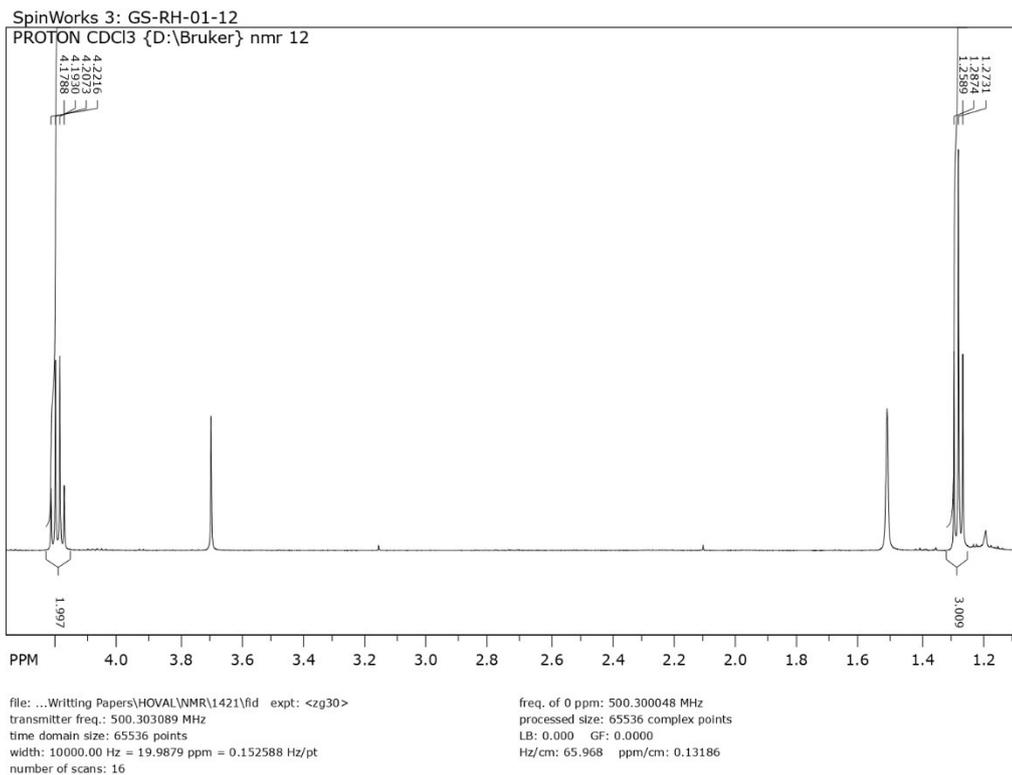
1H-NMR spectrum of Ethyl (*E*)-cinnamate (Entry 10, Table 6)



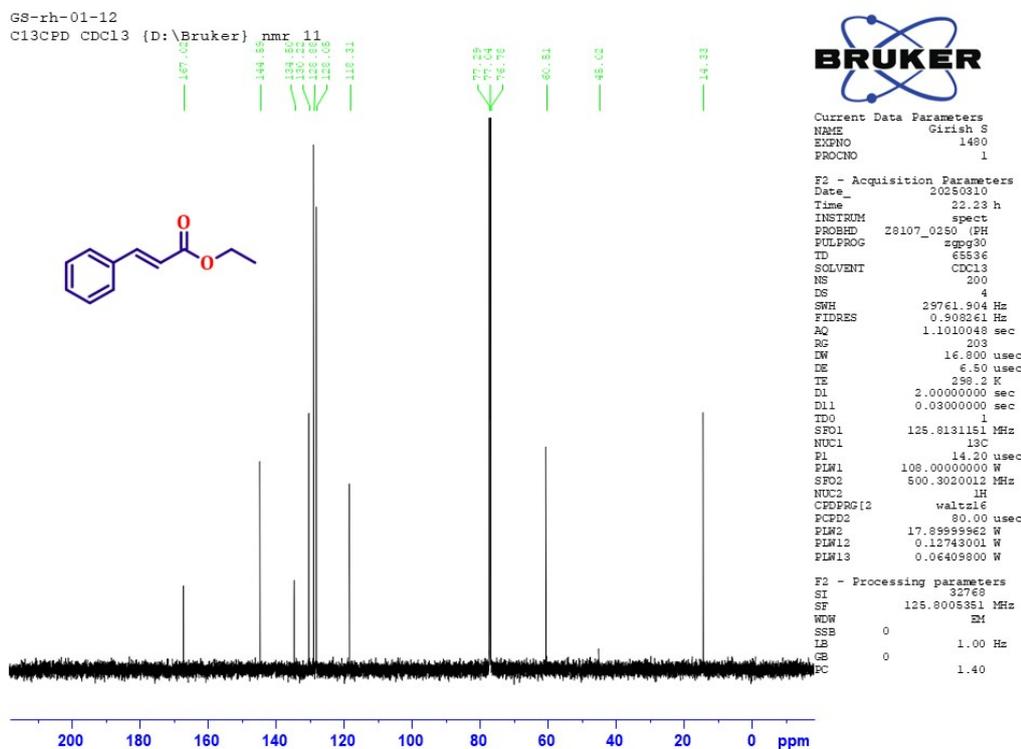
file: ...Writing Papers\HOVAL\NMR\1421\fid exp: <zg30>
 transmitter freq: 500.303089 MHz
 time domain size: 65536 points
 width: 10000.00 Hz = 19.9879 ppm = 0.152588 Hz/pt
 number of scans: 16

freq. of 0 ppm: 500.300048 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 27.216 ppm/cm: 0.05440

1H-NMR spectrum of Ethyl (*E*)-cinnamate (Entry 10, Table 6, Expansion 1)

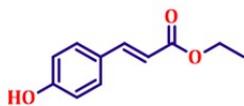


1H-NMR spectrum of Ethyl (*E*)-cinnamate (Entry 10, Table 6, Expansion 2)



13C NMR spectrum of Ethyl (*E*)-cinnamate (Entry 10, Table 6)

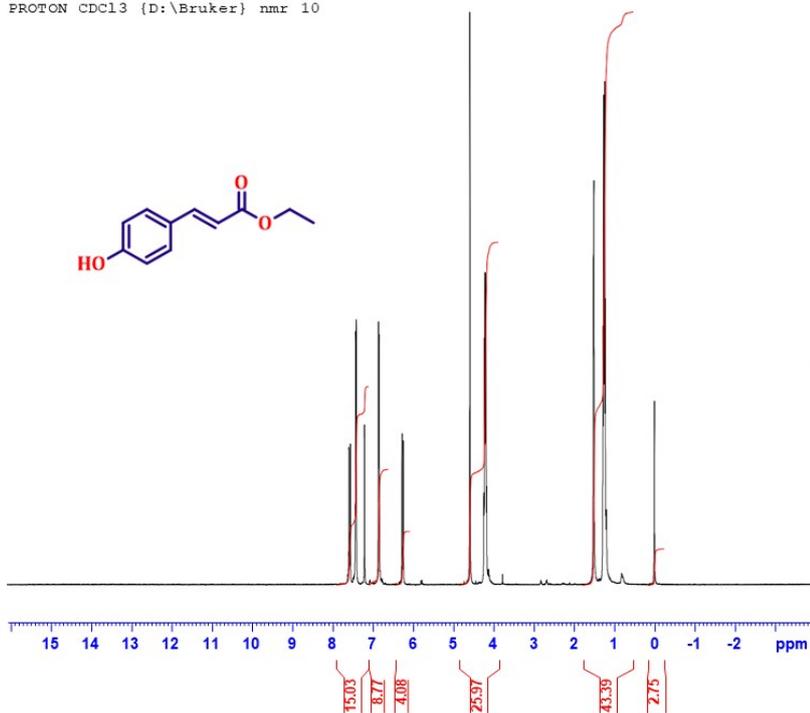
GS-RH-01-04
PROTON CDCl3 {D:\Bruker} nmr 10



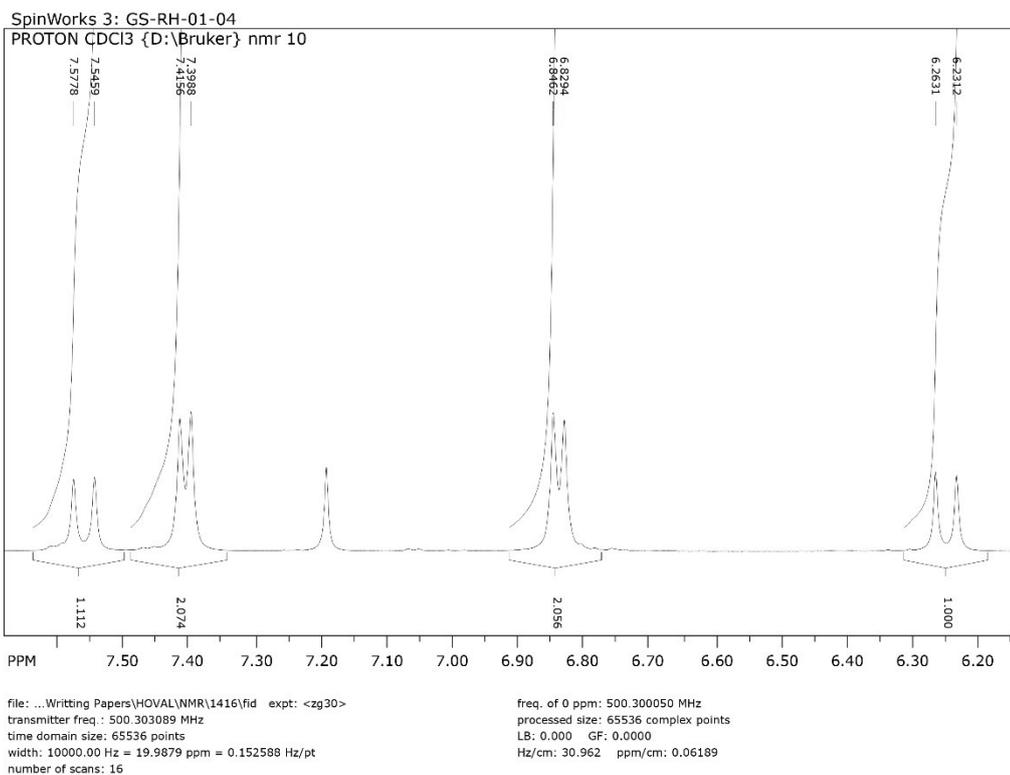
Current Data Parameters
NAME Girish S
EXPNO 1416
PROCNO 1

F2 - Acquisition Parameters
Date_ 20250112
Time 11.05 h
INSTRUM spect
PROBHD Z8107_0250 (PH
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 3.2767999 sec
RG 203
RW 50.000 usec
DE 6.50 usec
TE 298.4 K
D1 1.00000000 sec
TD0 1
SFO1 500.3030894 MHz
NUC1 1H
P1 6.75 usec
PLW1 17.89999962 W

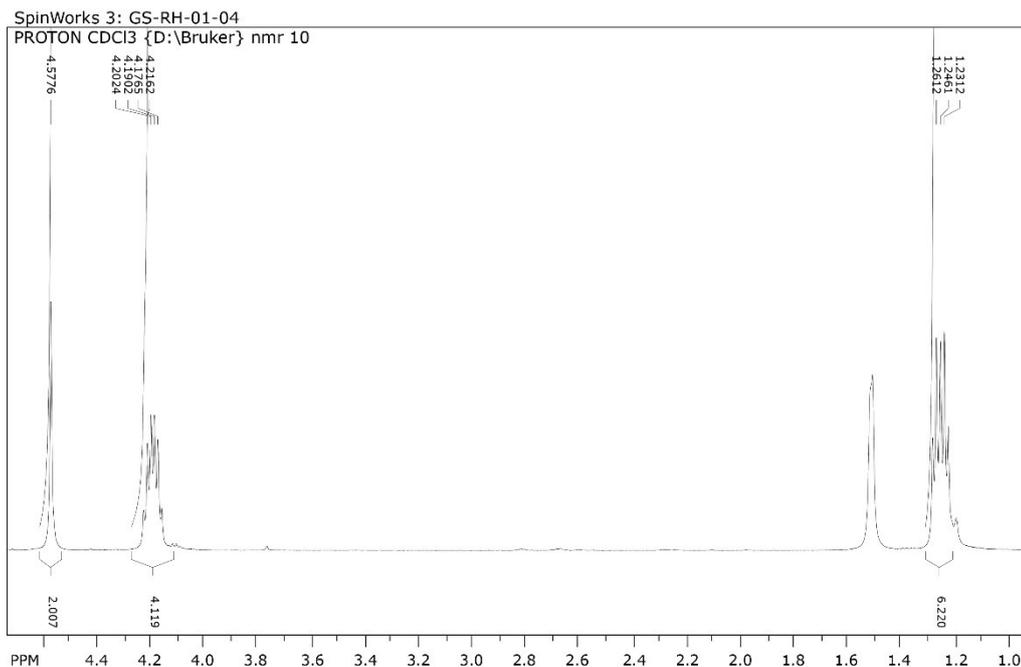
F2 - Processing parameters
SI 65536
SF 500.3000496 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



**1H-NMR spectrum of Ethyl (*E*)-3-(4-hydroxyphenyl)acrylate
(Entry 11, Table 6)**



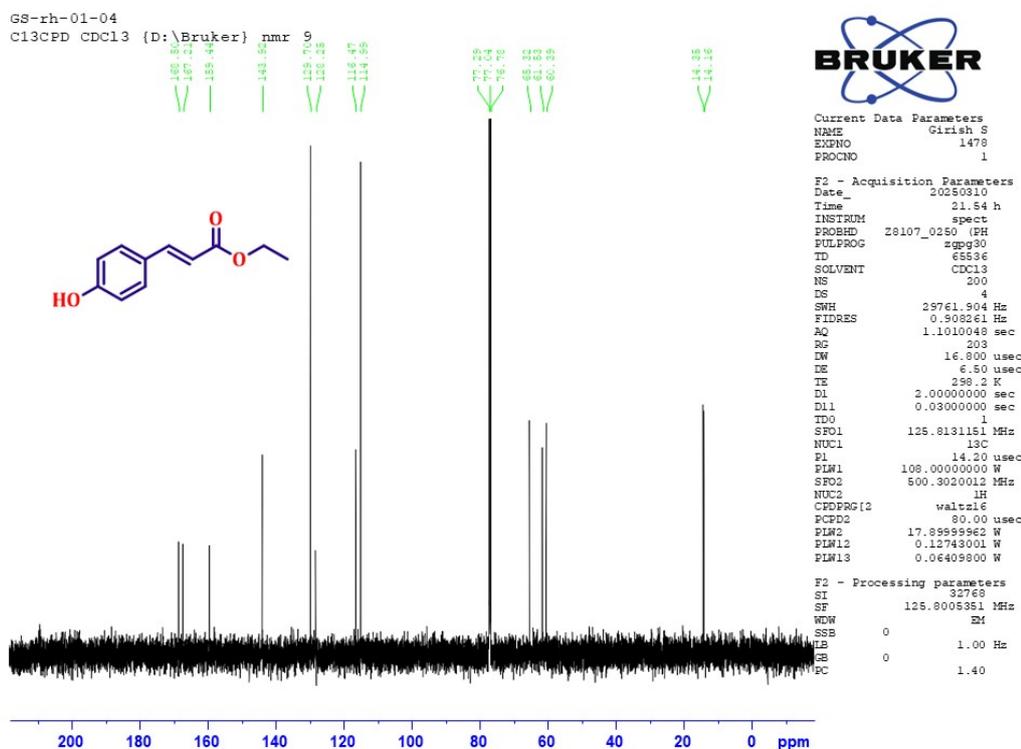
**1H-NMR spectrum of Ethyl (*E*)-3-(4-hydroxyphenyl)acrylate
(Entry 11, Table 6, Expansion 1)**



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 transmitter freq: 500.303089 MHz
 time domain size: 65536 points
 width: 10000.00 Hz = 19.9879 ppm = 0.152588 Hz/pt
 number of scans: 16

freq. of 0 ppm: 500.300050 MHz
 processed size: 65536 complex points
 LB: 0.000 GF: 0.0000
 Hz/cm: 76.031 ppm/cm: 0.15197

1H-NMR spectrum of Ethyl (*E*)-3-(4-hydroxyphenyl)acrylate (Entry 11, Table 6, Expansion 2)



13C NMR spectrum of Ethyl (*E*)-3-(4-hydroxyphenyl)acrylate (Entry 11, Table 6)