

Supplementary Material

Preparation of key intermediates for the synthesis of Coenzyme Q₁₀ and derivatives via cross-metathesis reactionsTrang Thu Thi Nguyen,¹ and Hung Dinh Mac ² * and Phong Van Pham¹ *¹ Laboratory of Catalysis at Faculty of Chemistry, VNU University of Science, Vietnam National University, Hanoi 110403, Vietnam² Laboratory of Pharmaceutical Chemistry at Faculty of Chemistry, VNU University of Science, Vietnam National University, Hanoi 110403, Vietnam

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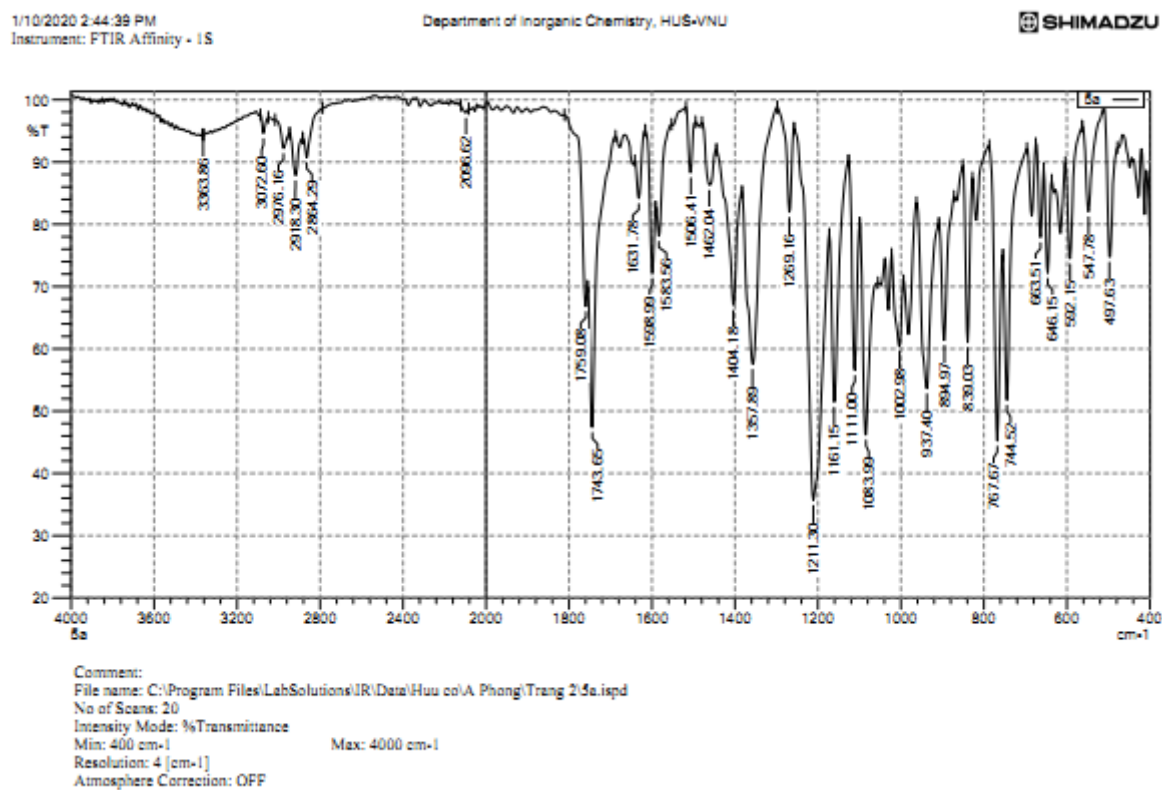
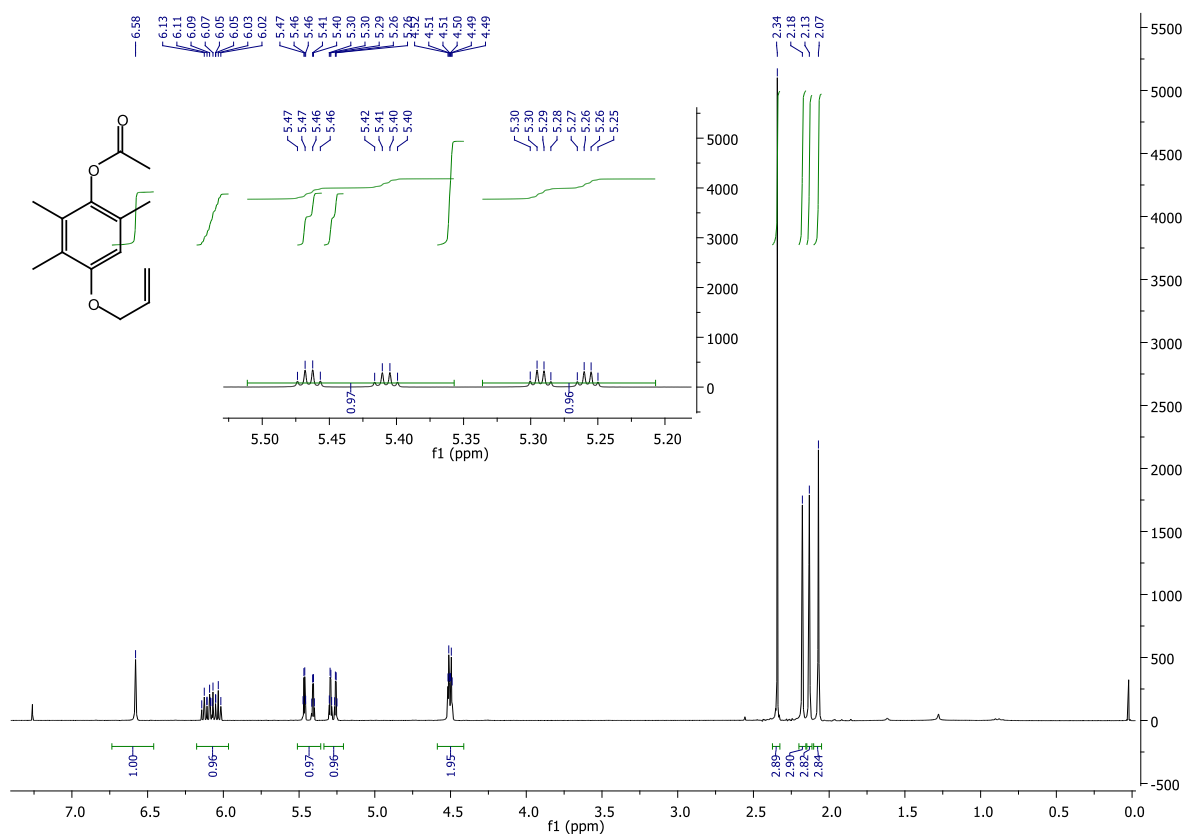
Table of Contents

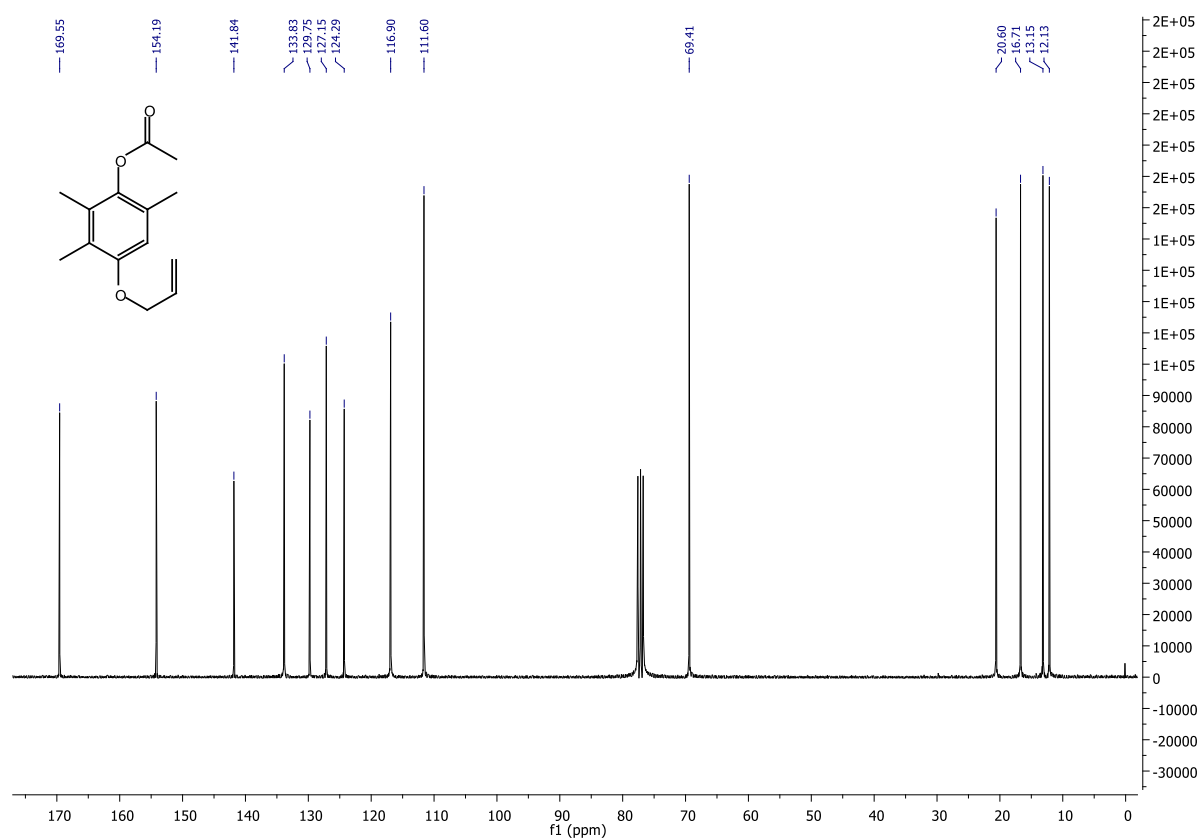
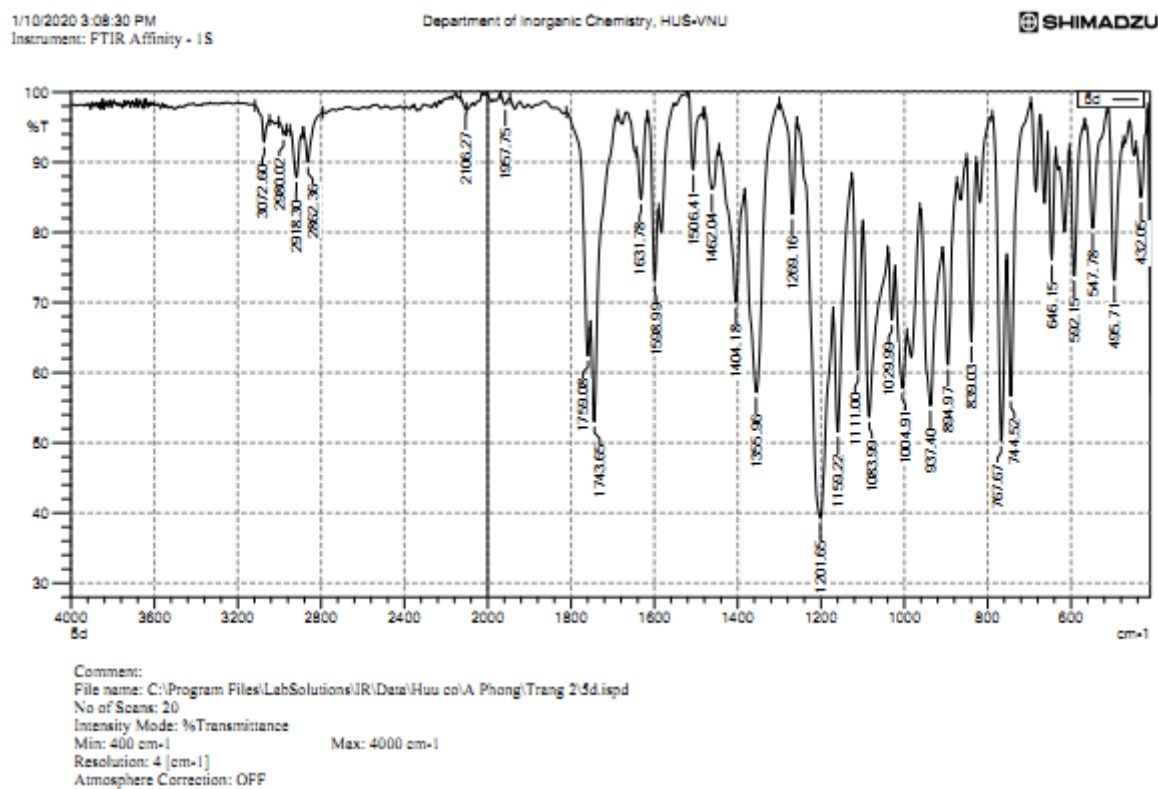
IR Spectrum of 4-(allyloxy)-2,3,6-trimethylphenyl acetate (5a)	S4
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 4-(allyloxy)-2,3,6-trimethylphenyl acetate (5a)	S4
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 4-(allyloxy)-2,3,6-trimethylphenyl acetate (5a)	S5
IR Spectrum of 4-(allyloxy)-2-methylnaphthalen-1-yl acetate (5d)	S5
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 4-(allyloxy)-2-methylnaphthalen-1-yl acetate (5d)	S6
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 4-(allyloxy)-2-methylnaphthalen-1-yl acetate (5d)	S6
IR Spectrum of 3-allyl-4-hydroxy-2,5,6-trimethylphenyl acetate (6a)	S7
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 3-allyl-4-hydroxy-2,5,6-trimethylphenyl acetate (6a)	S7
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 3-allyl-4-hydroxy-2,5,6-trimethylphenyl acetate (6a)	S8
IR Spectrum of 3-allyl-4-hydroxy-2-methylnaphthalen-1-yl acetate (6d)	S8
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 3-allyl-4-hydroxy-2-methylnaphthalen-1-yl acetate (6d)	S9
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 3-allyl-4-hydroxy-2-methylnaphthalen-1-yl acetate (6d)	S9
IR Spectrum of 2-allyl-3,5,6-trimethyl-1,4-phenylene diacetate (7a)	S10
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 2-allyl-3,5,6-trimethyl-1,4-phenylene diacetate (7a)	S10
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 2-allyl-3,5,6-trimethyl-1,4-phenylene diacetate (7a)	S11
IR Spectrum of 1-allyl-2,5-dimethoxy-3,4,6-trimethylbenzene (7b)	S11
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 1-allyl-2,5-dimethoxy-3,4,6-trimethylbenzene (7b)	S12
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 1-allyl-2,5-dimethoxy-3,4,6-trimethylbenzene (7b)	S12
IR Spectrum of 1-allyl-2,3,4,5-tetramethoxy-6-methylbenzene (7c)	S13
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 1-allyl-2,3,4,5-tetramethoxy-6-methylbenzene (7c)	S13
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 1-allyl-2,3,4,5-tetramethoxy-6-methylbenzene (7c)	S14
IR Spectrum (500 MHz, CDCl ₃) of 2-allyl-3-methylnaphthalene-1,4-diyl diacetate (7d)	S14
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 2-allyl-3-methylnaphthalene-1,4-diyl diacetate (7d)	S15

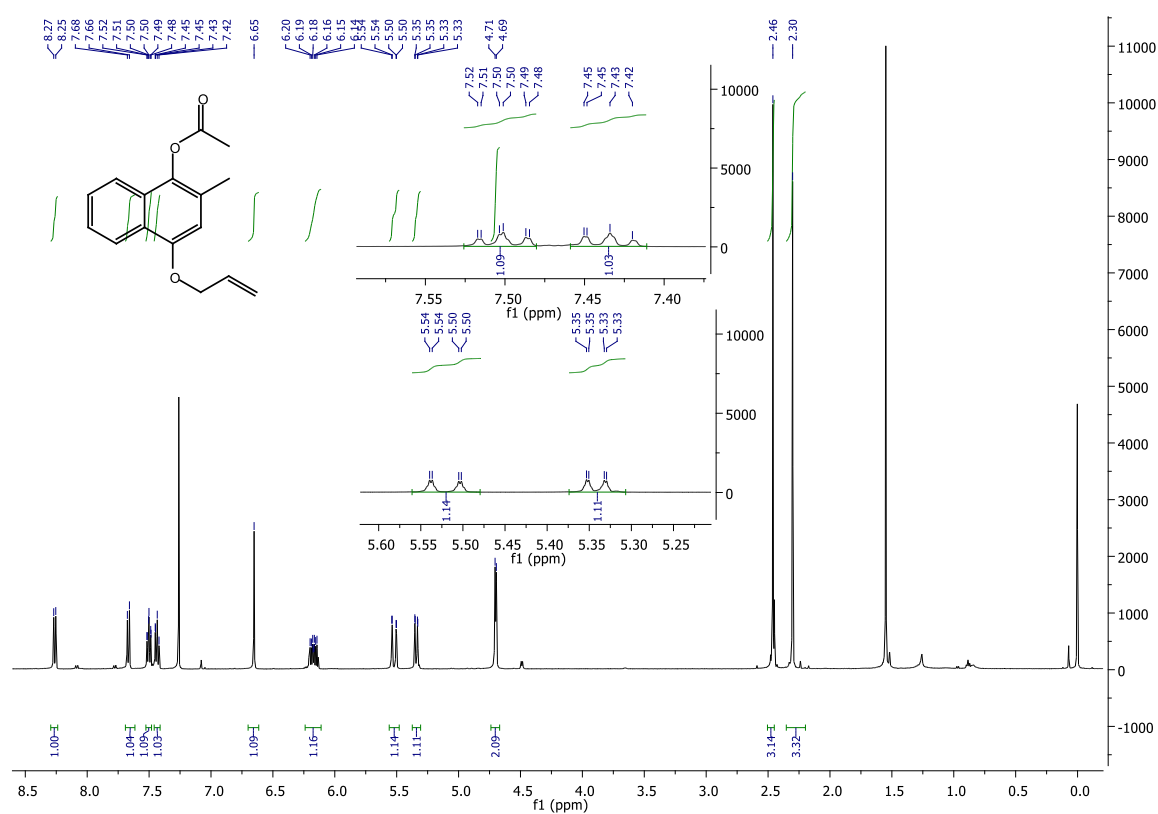
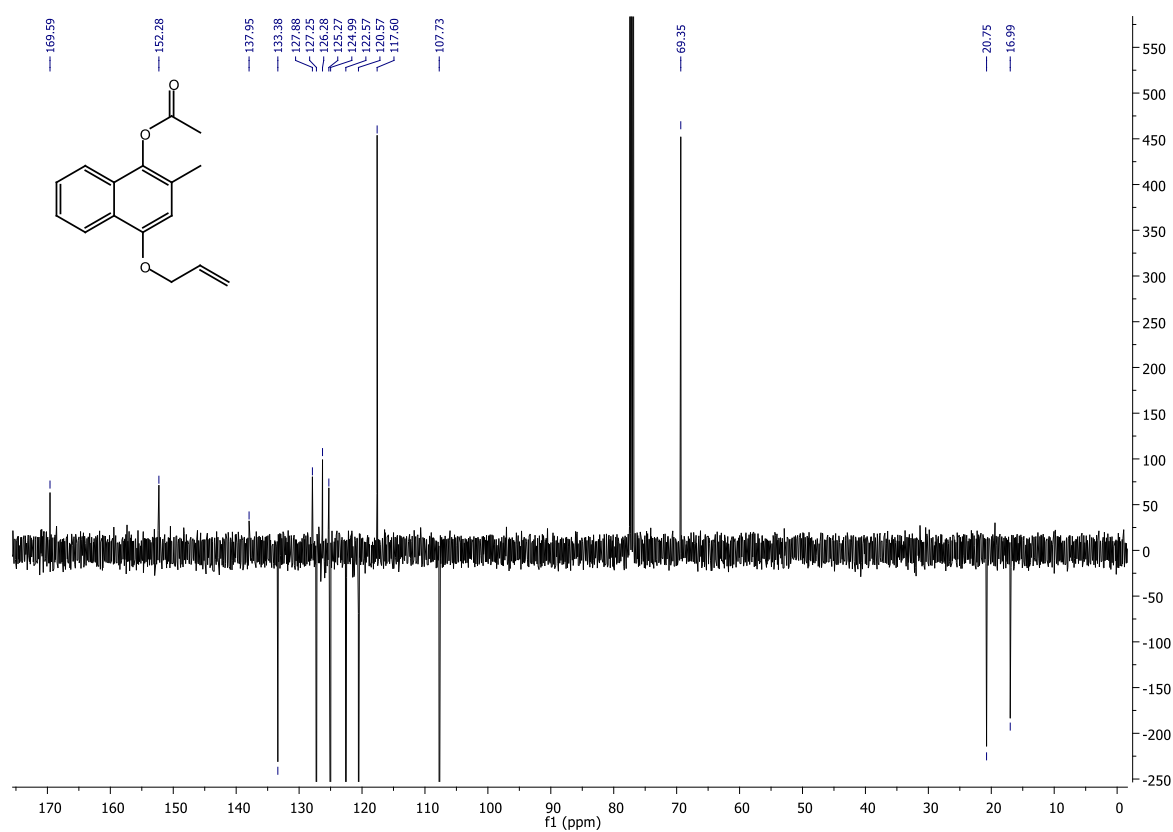
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 2-allyl-3-methylnaphthalene-1,4-diyl diacetate (7d)	S15
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 2-allyl-1,4-dimethoxy-3-methylnaphthalene (7e)	S16
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 2-allyl-1,4-dimethoxy-3-methylnaphthalene (7e)	S16
IR Spectrum of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (8a)	S17
¹ H NMR Spectrum (500 MHz, CDCl ₃) of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (8a)	S17
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (8a)	S18
Noesy spectrum of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (8a)	S18
IR Spectrum of methyl (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (8b)	S19
¹ H NMR Spectrum (500 MHz, CDCl ₃) of methyl (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (8b)	S19
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of methyl (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (8b)	S20
NOESY spectrum of methyl (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (8b)	S20
IR Spectrum of methyl (<i>E</i>)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-enoate (8c)	S21
¹ H NMR Spectrum (500 MHz, CDCl ₃) of methyl (<i>E</i>)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-enoate (8c)	S21
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of methyl (<i>E</i>)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-enoate (8c)	S22
IR Spectrum of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3-methylnaphthalene-1,4-diyl diacetate (8d)	S22
¹ H NMR Spectrum (500 MHz, CDCl ₃) of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3-methylnaphthalene-1,4-diyl diacetate (8d)	S23
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of (<i>E</i>)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3-methylnaphthalene-1,4-diyl diacetate (8d)	S23
IR Spectrum of methyl (<i>E</i>)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-enoate (8e)	S24
¹ H NMR Spectrum (500 MHz, CDCl ₃) of methyl (<i>E</i>)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-enoate (8e)	S24
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of methyl (<i>E</i>)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-enoate (8e)	S25

IR Spectrum of (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylcyclohexa-1,5-dienyl)-2-methylbut-2-en-1-ol (2b)	S25
¹ H NMR Spectrum (500 MHz, CDCl ₃) of (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylcyclohexa-1,5-dienyl)-2-methylbut-2-en-1-ol (2b)	S26
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of (<i>E</i>)-4-(2,5-dimethoxy-3,4,6-trimethylcyclohexa-1,5-dienyl)-2-methylbut-2-en-1-ol (2b)	S26
IR Spectrum of (<i>E</i>)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-en-1-ol (2c)	S27
¹ H NMR Spectrum (500 MHz, CDCl ₃) of methyl (<i>E</i>)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-en-1-ol (2c)	S27
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of (<i>E</i>)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-en-1-ol (2c)	S28
IR Spectrum of (<i>E</i>)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-en-1-ol (2e)	S28
¹ H NMR Spectrum (500 MHz, CDCl ₃) of (<i>E</i>)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-en-1-ol (2e)	S29
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of (<i>E</i>)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-en-1-ol (2e)	S29
IR Spectrum of (<i>E</i>)-1,4-dimethoxy-2,3,5-trimethyl-6-(3-methyl-4-(phenylsulfonyl)but-2-enyl)benzene (10)	S30
¹ H NMR Spectrum (500 MHz, CDCl ₃) of (<i>E</i>)-1,4-dimethoxy-2,3,5-trimethyl-6-(3-methyl-4-(phenylsulfonyl)but-2-enyl)benzene (10)	S30
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of (<i>E</i>)-1,4-dimethoxy-2,3,5-trimethyl-6-(3-methyl-4-(phenylsulfonyl)but-2-enyl)benzene (10)	S31
IR Spectrum of 1-((2 <i>E</i> ,6 <i>E</i> ,10 <i>E</i> ,14 <i>E</i> ,18 <i>E</i> ,22 <i>E</i> ,26 <i>E</i> ,30 <i>E</i> ,34 <i>E</i>)-3,7,11,15,19,23,27,31,35,39-decamethyl-4-(phenylsulfonyl)tetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (11)	S31
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 1-((2 <i>E</i> ,6 <i>E</i> ,10 <i>E</i> ,14 <i>E</i> ,18 <i>E</i> ,22 <i>E</i> ,26 <i>E</i> ,30 <i>E</i> ,34 <i>E</i>)-3,7,11,15,19,23,27,31,35,39-decamethyl-4-(phenylsulfonyl)tetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (11)	S32
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 1-((2 <i>E</i> ,6 <i>E</i> ,10 <i>E</i> ,14 <i>E</i> ,18 <i>E</i> ,22 <i>E</i> ,26 <i>E</i> ,30 <i>E</i> ,34 <i>E</i>)-3,7,11,15,19,23,27,31,35,39-decamethyl-4-(phenylsulfonyl)tetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (11)	S32
IR Spectrum of 1-((2 <i>E</i> ,6 <i>E</i> ,10 <i>E</i> ,14 <i>E</i> ,18 <i>E</i> ,22 <i>E</i> ,26 <i>E</i> ,30 <i>E</i> ,34 <i>E</i>)-3,7,11,15,19,23,27,31,35,39-decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (12)	S33
¹ H NMR Spectrum (500 MHz, CDCl ₃) of 1-((2 <i>E</i> ,6 <i>E</i> ,10 <i>E</i> ,14 <i>E</i> ,18 <i>E</i> ,22 <i>E</i> ,26 <i>E</i> ,30 <i>E</i> ,34 <i>E</i>)-3,7,11,15,19,23,27,31,35,39-decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (12)	S33
¹³ C NMR Spectrum (125 MHz, CDCl ₃) of 1-((2 <i>E</i> ,6 <i>E</i> ,10 <i>E</i> ,14 <i>E</i> ,18 <i>E</i> ,22 <i>E</i> ,26 <i>E</i> ,30 <i>E</i> ,34 <i>E</i>)-3,7,11,15,19,23,27,31,35,39-decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (12)	S34

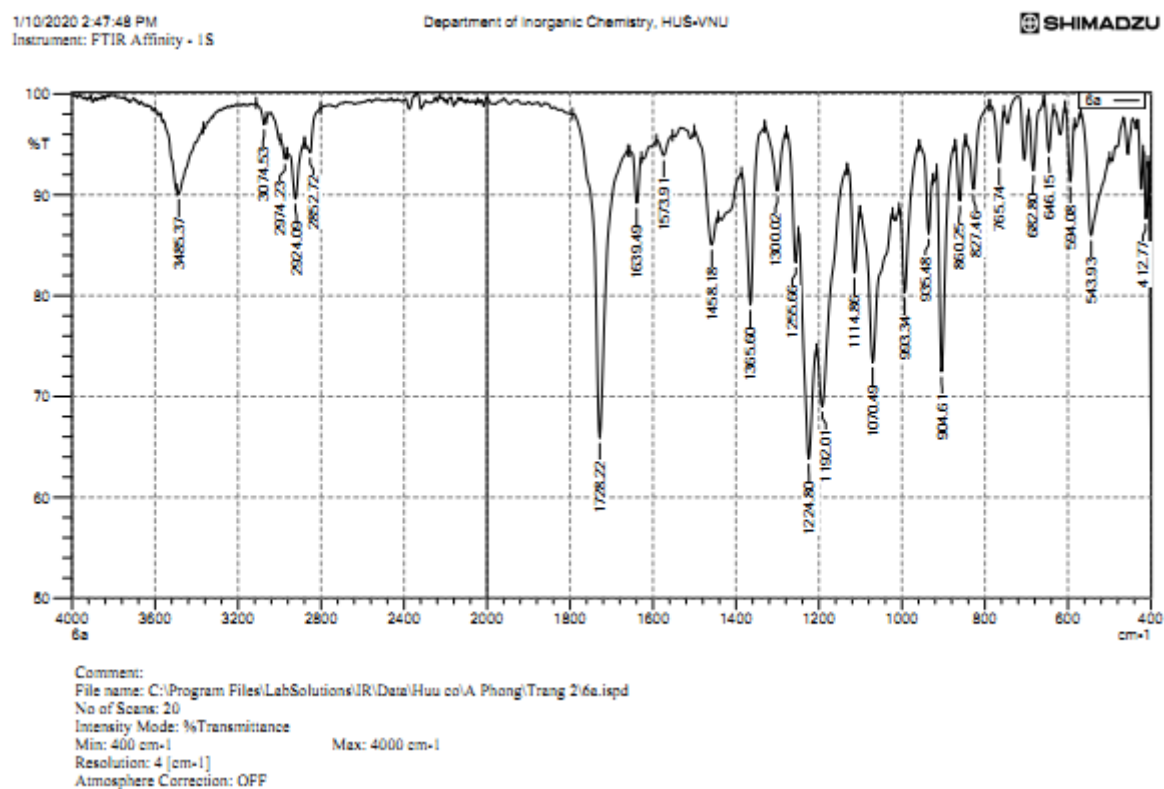
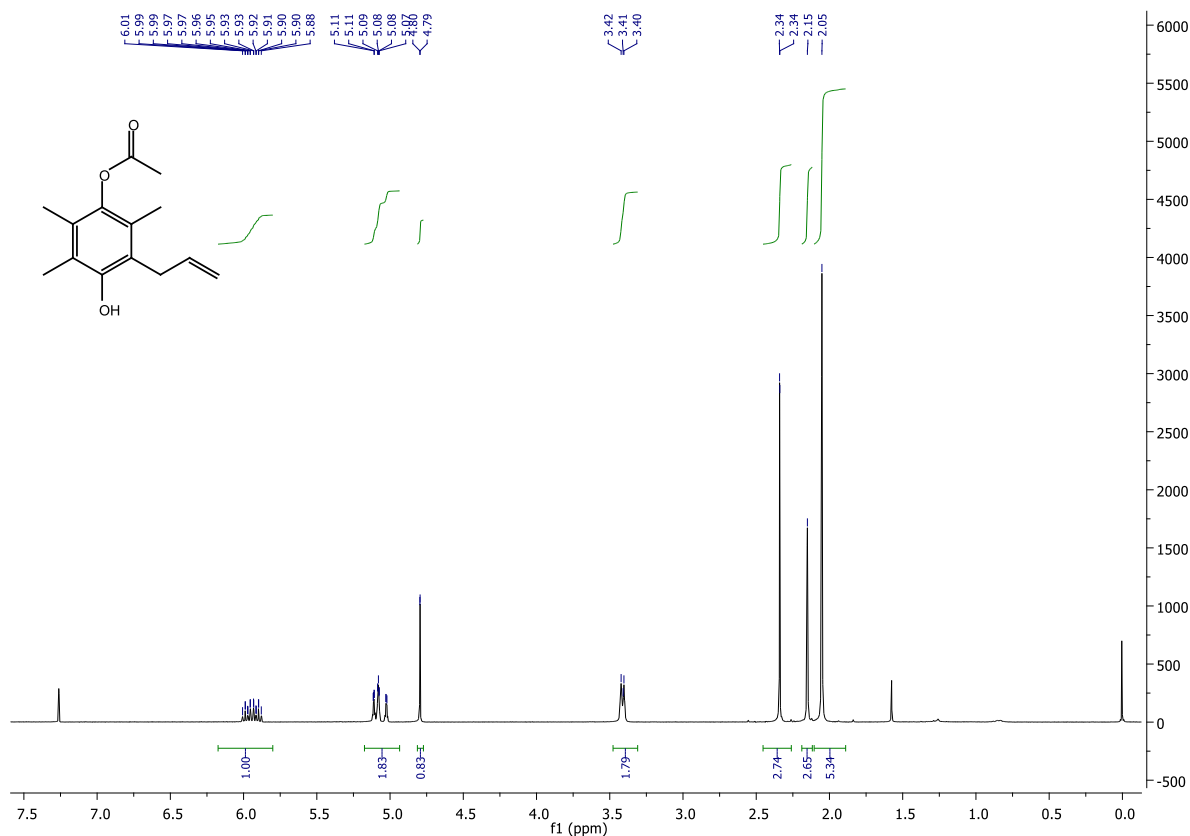
IR Spectrum of 4-(allyloxy)-2,3,6-trimethylphenyl acetate (5a)

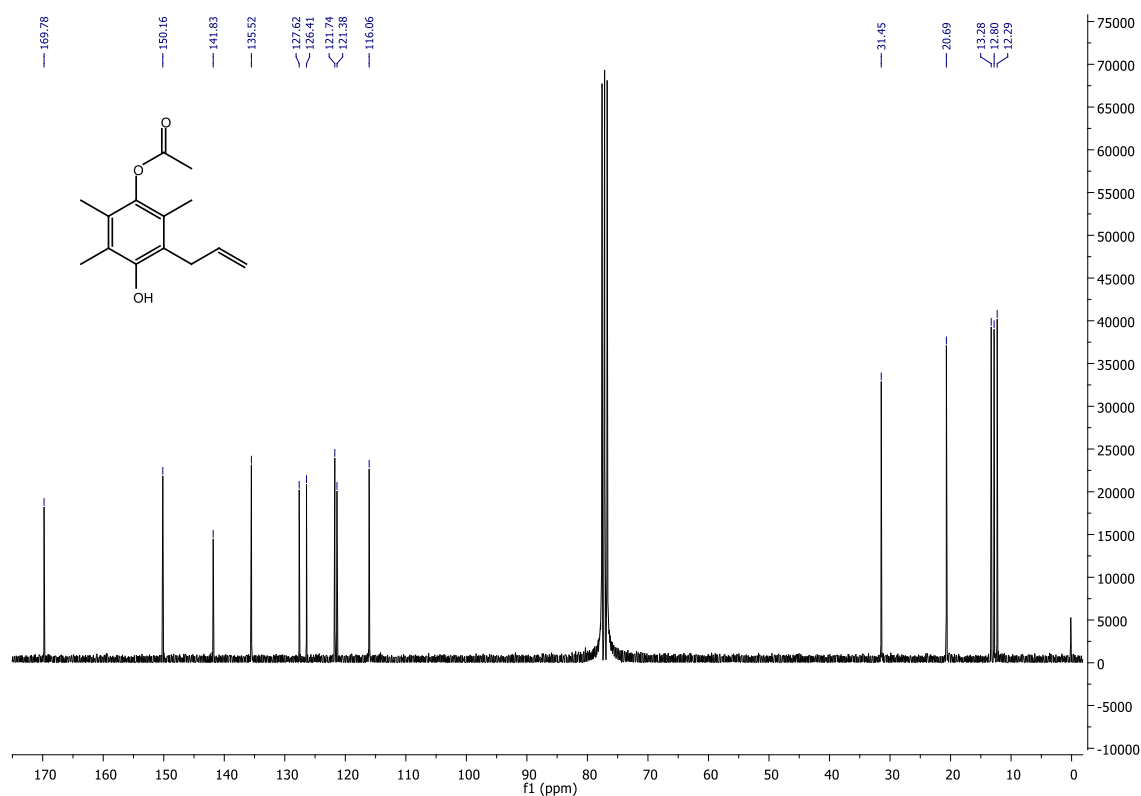
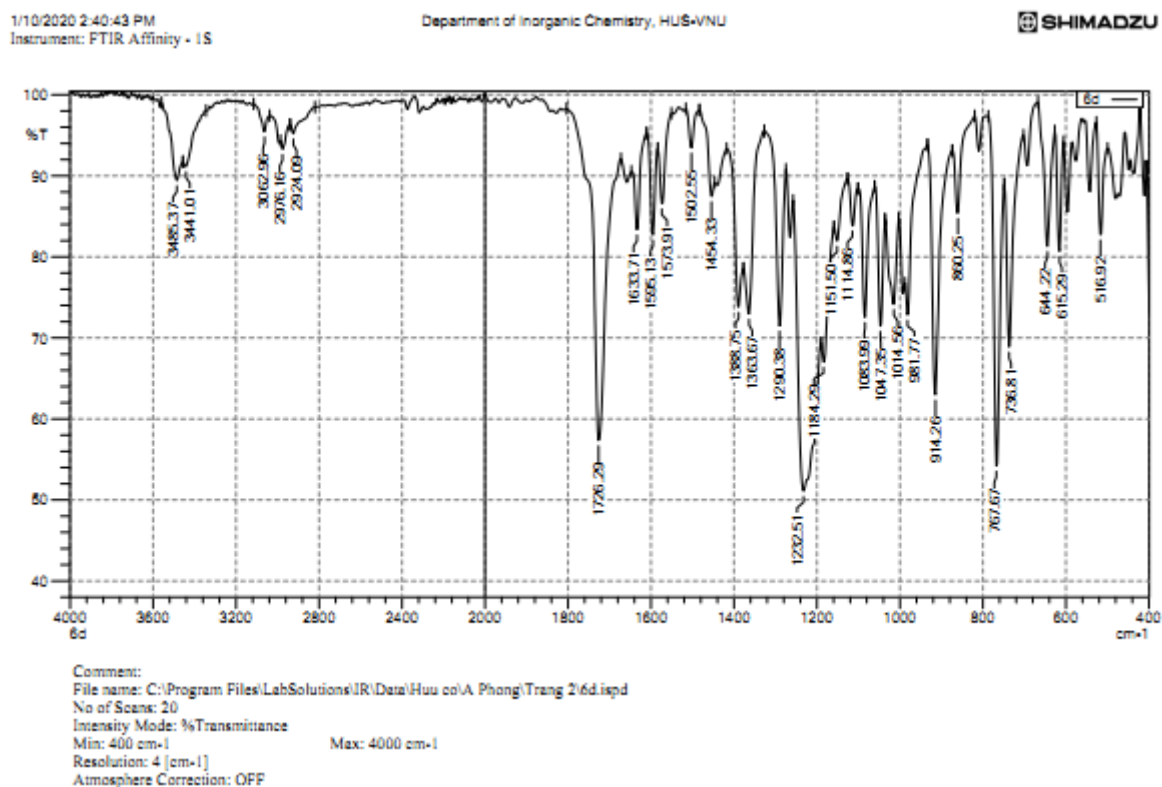
¹H NMR Spectrum (500 MHz, CDCl₃) of 4-(allyloxy)-2,3,6-trimethylphenyl acetate (5a)

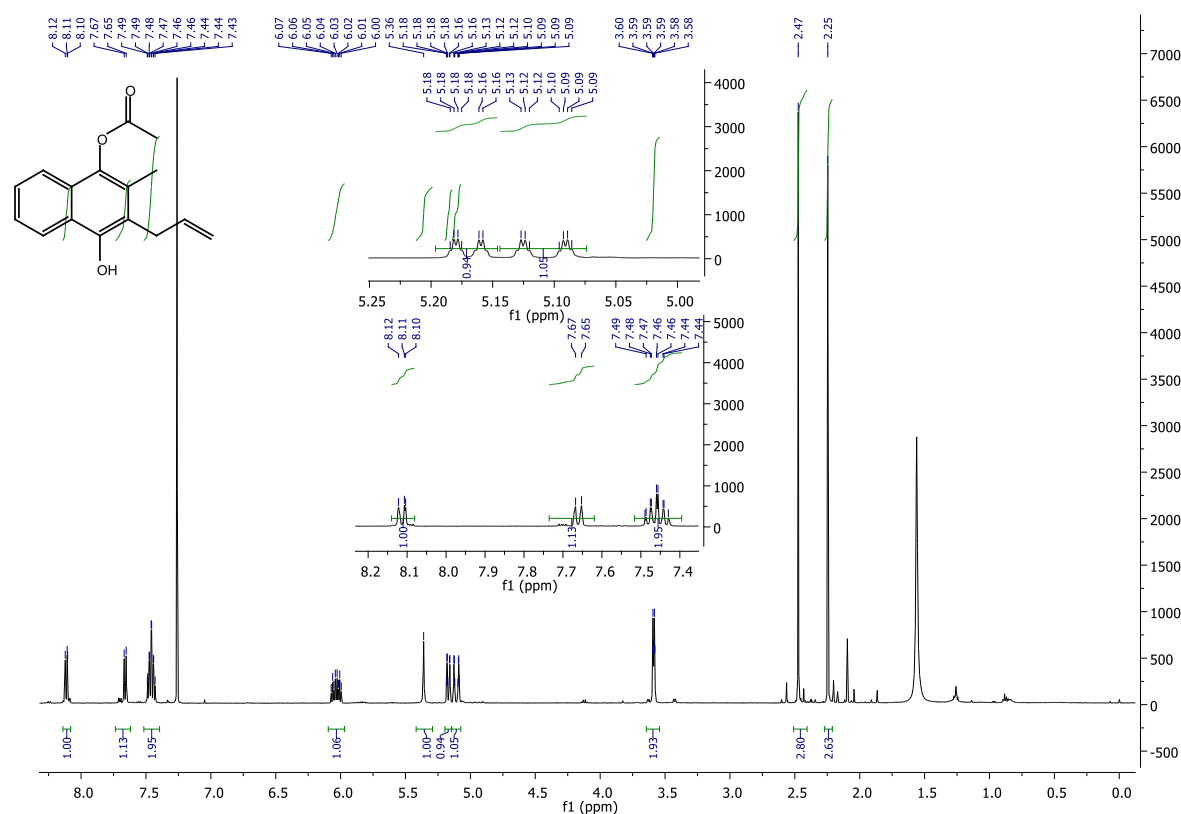
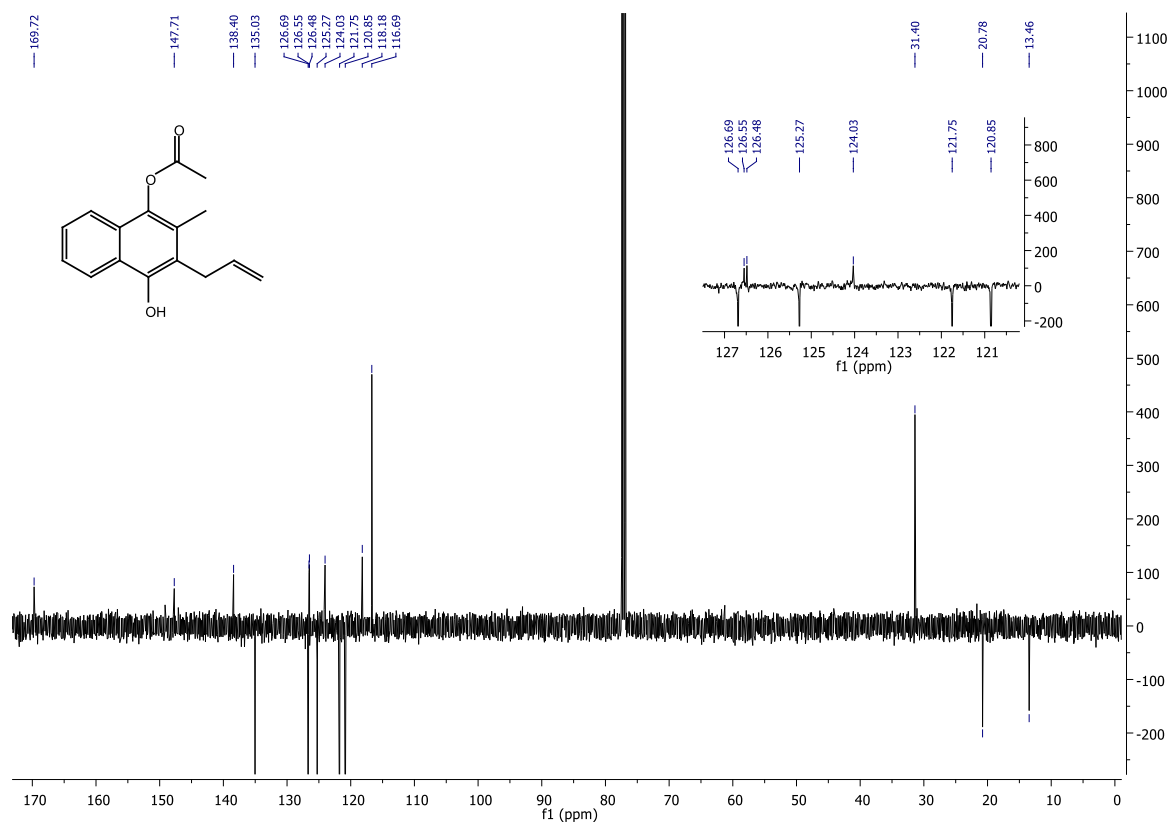
^{13}C NMR Spectrum (125 MHz, CDCl_3) of 4-(allyloxy)-2,3,6-trimethylphenyl acetate (**5a**)IR Spectrum of 4-(allyloxy)-2-methylnaphthalen-1-yl acetate (**5d**)

^1H NMR Spectrum (500 MHz, CDCl_3) of 4-(allyloxy)-2-methylnaphthalen-1-yl acetate (**5d**) ^{13}C NMR Spectrum (125 MHz, CDCl_3) of 4-(allyloxy)-2-methylnaphthalen-1-yl acetate (**5d**)

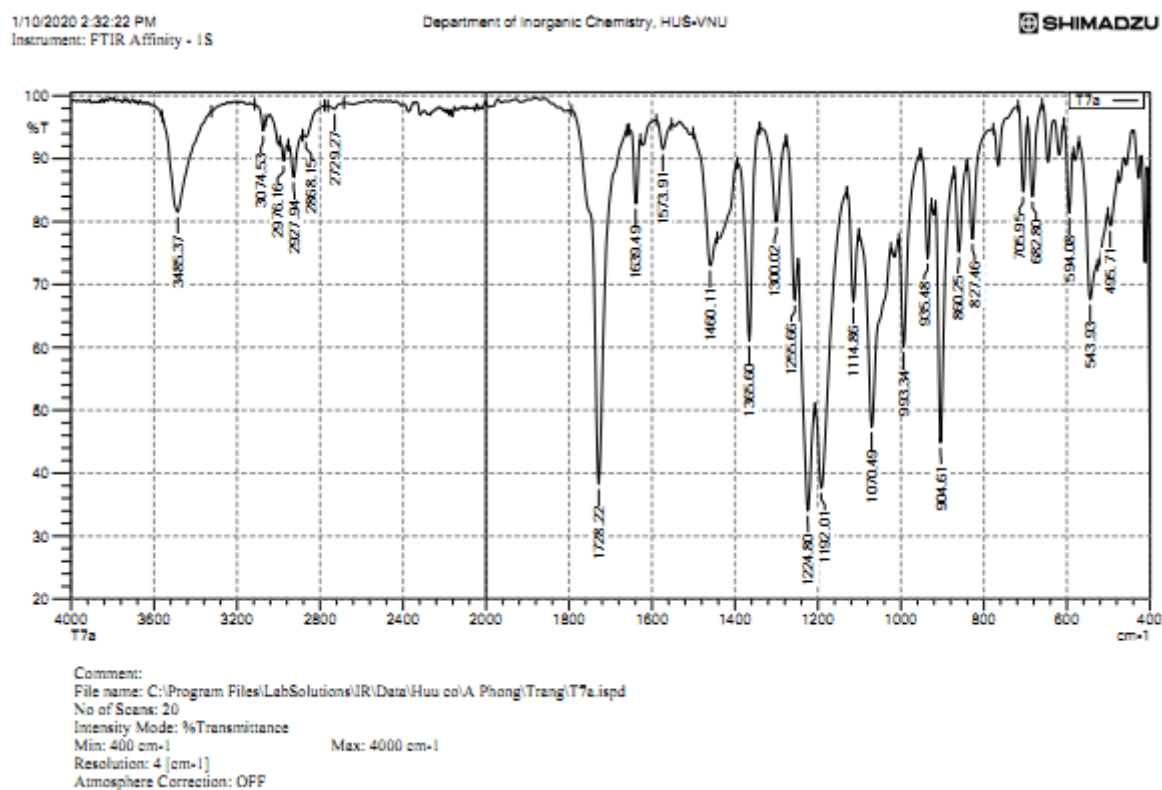
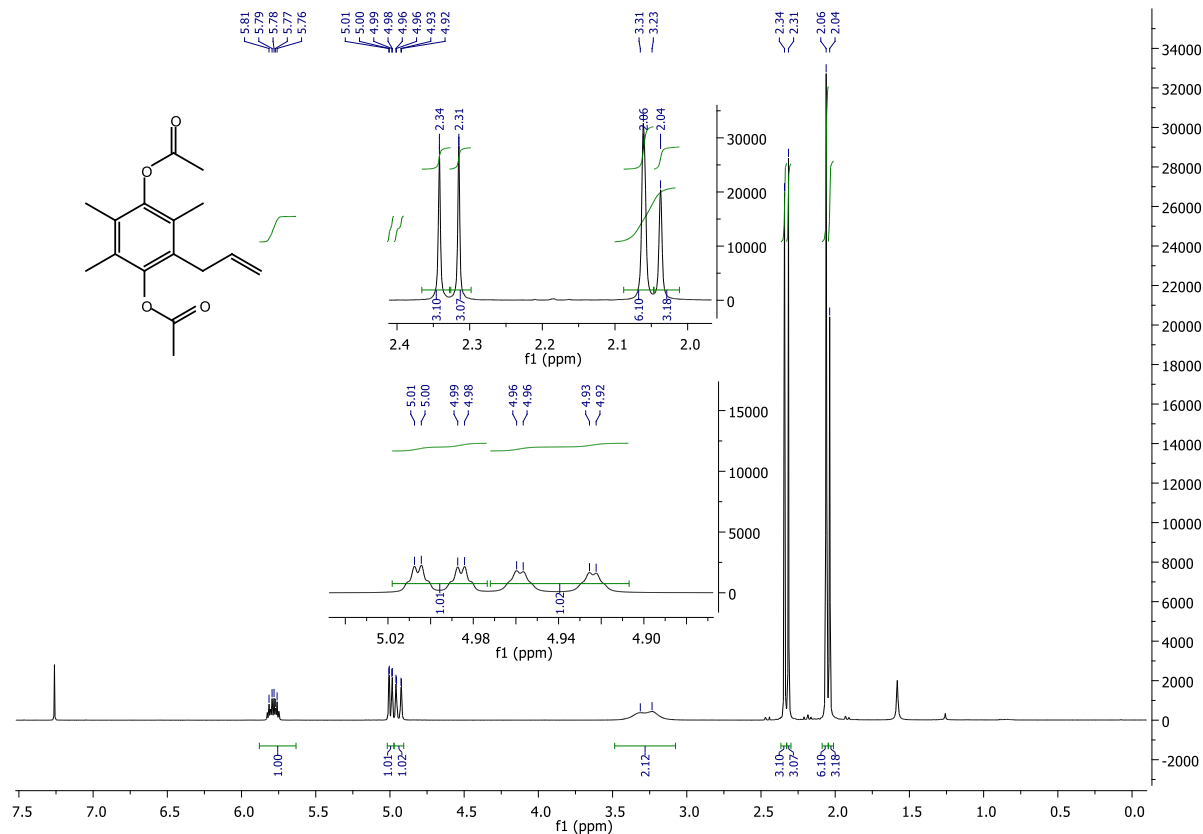
IR Spectrum of 3-allyl-4-hydroxy-2,5,6-trimethylphenyl acetate (6a)

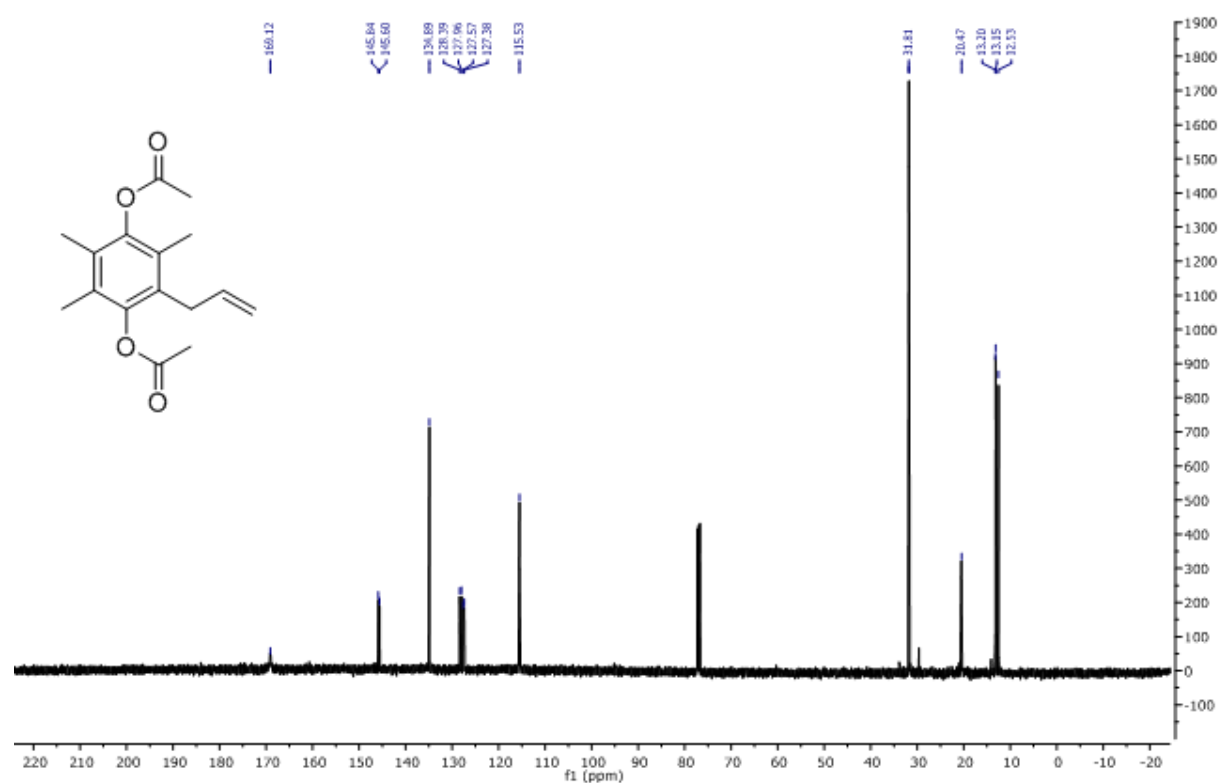
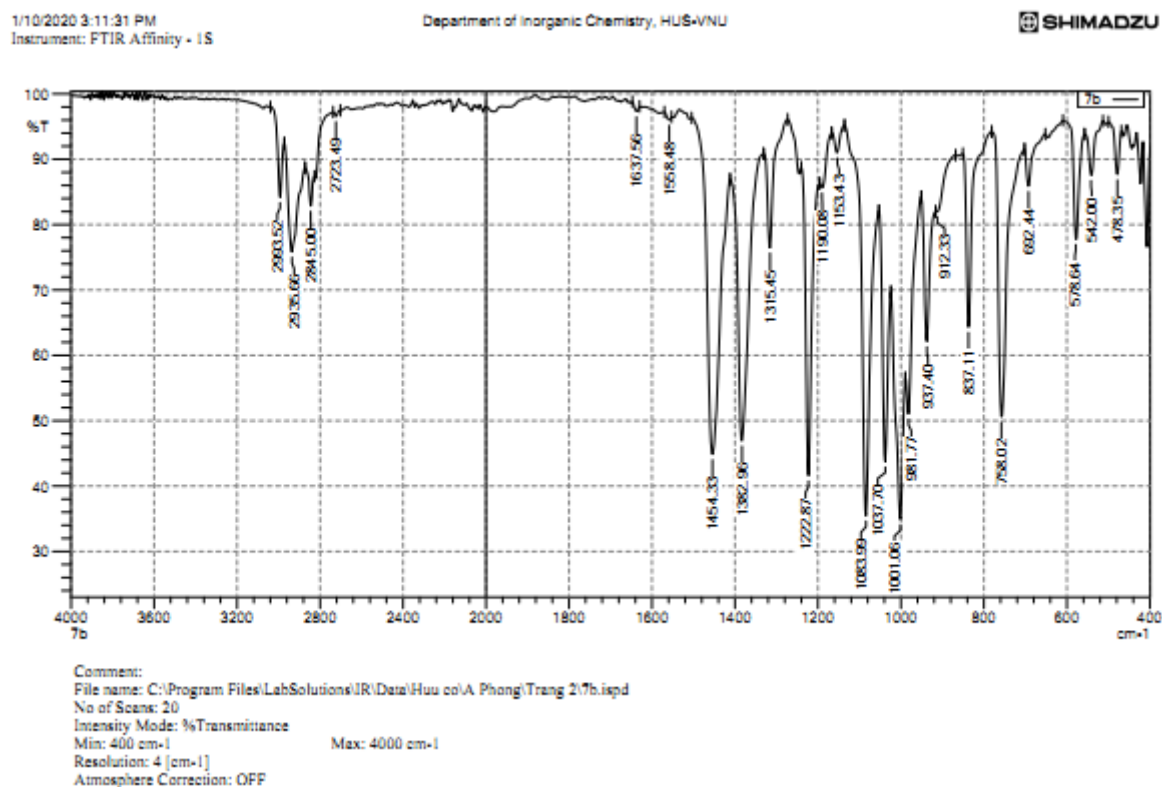
 ^1H NMR Spectrum (500 MHz, CDCl_3) of 3-allyl-4-hydroxy-2,5,6-trimethylphenyl acetate (6a)

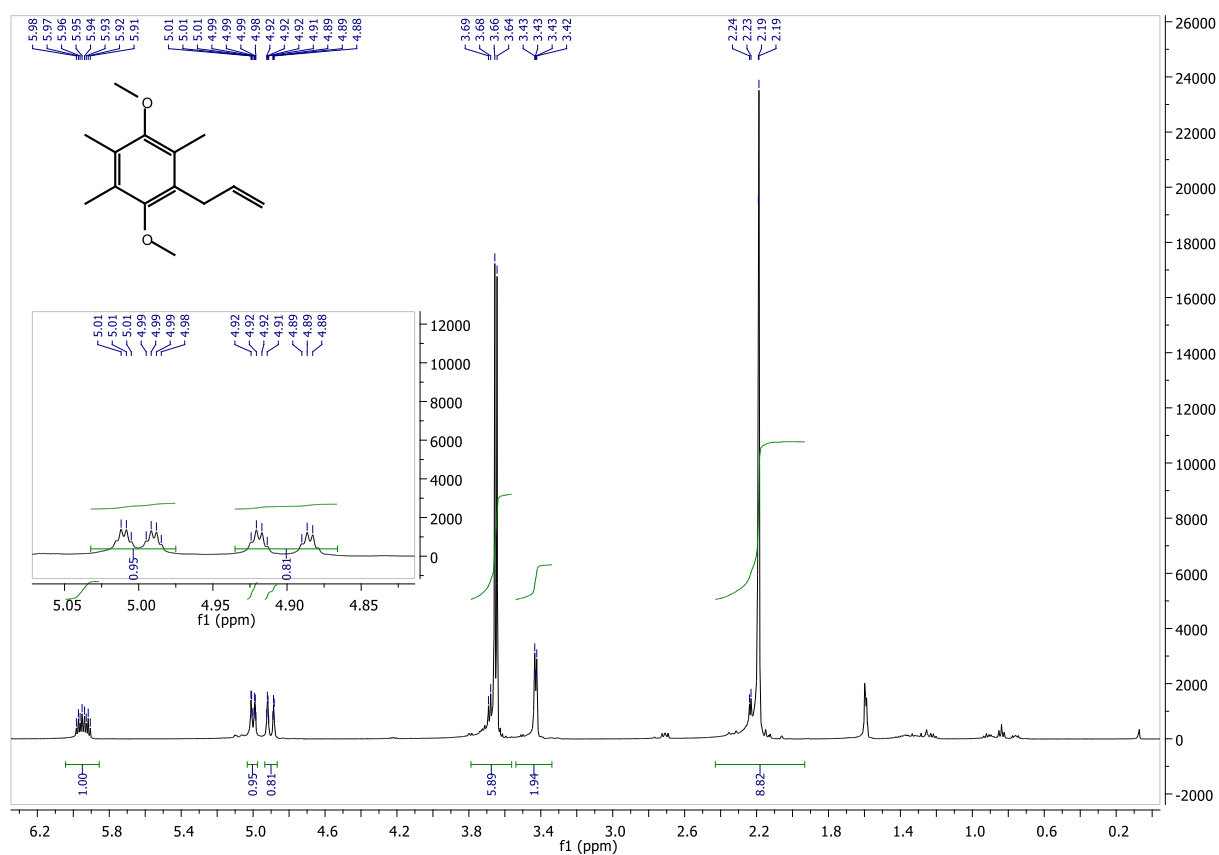
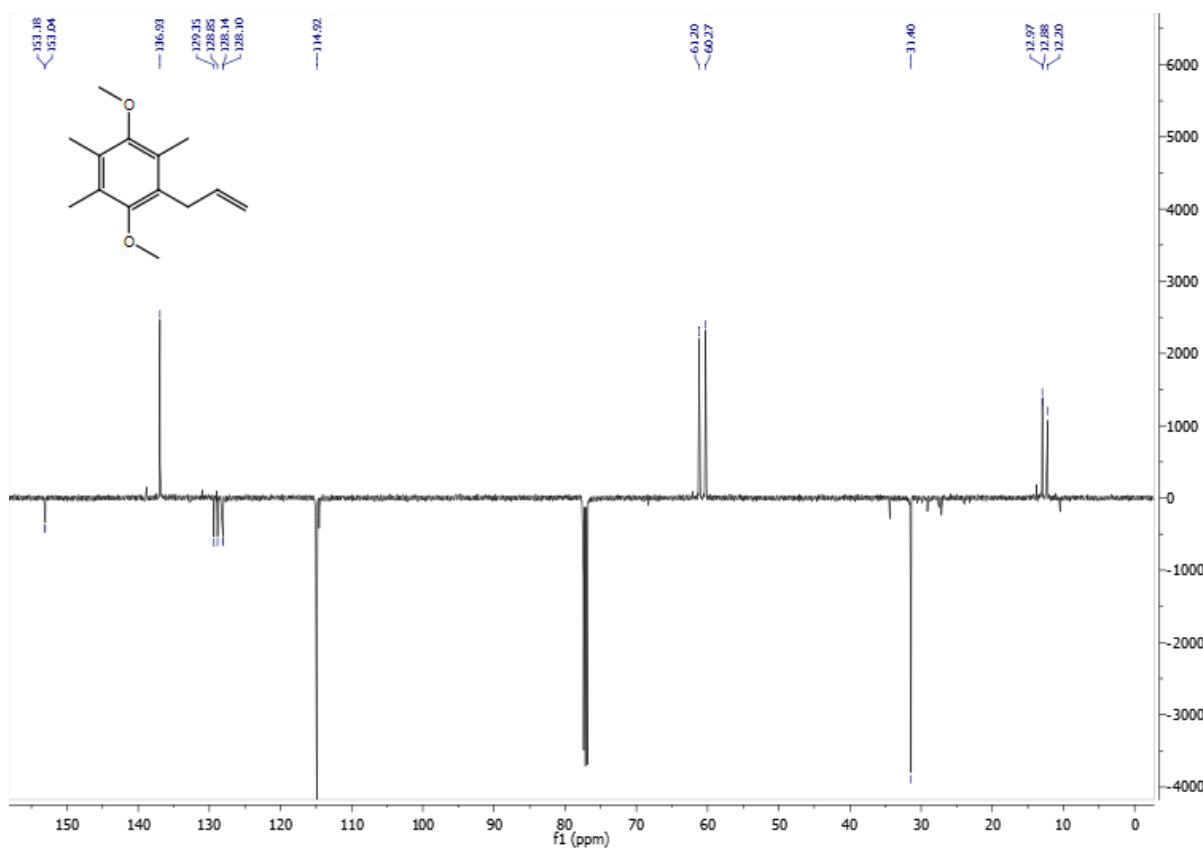
^{13}C NMR Spectrum (125 MHz, CDCl_3) of 3-allyl-4-hydroxy-2,5,6-trimethylphenyl acetate (**6a**)IR Spectrum of 3-allyl-4-hydroxy-2-methylnaphthalen-1-yl acetate (**6d**)

^1H NMR Spectrum (500 MHz, CDCl_3) of 3-allyl-4-hydroxy-2-methylnaphthalen-1-yl acetate (**6d**) ^{13}C NMR Spectrum (125 MHz, CDCl_3) of 3-allyl-4-hydroxy-2-methylnaphthalen-1-yl acetate (**6d**)

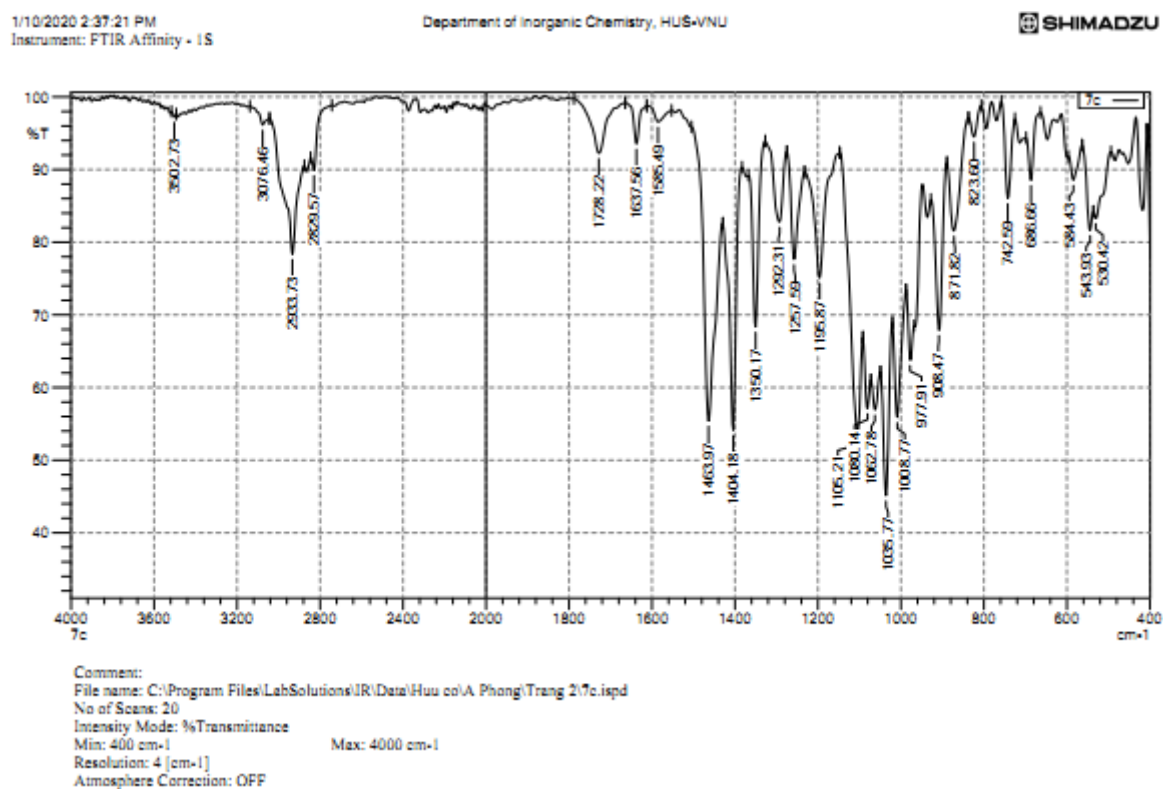
IR Spectrum of 2-allyl-3,5,6-trimethyl-1,4-phenylene diacetate (7a)

¹H NMR Spectrum (500 MHz, CDCl₃) of 2-allyl-3,5,6-trimethyl-1,4-phenylene diacetate (7a)

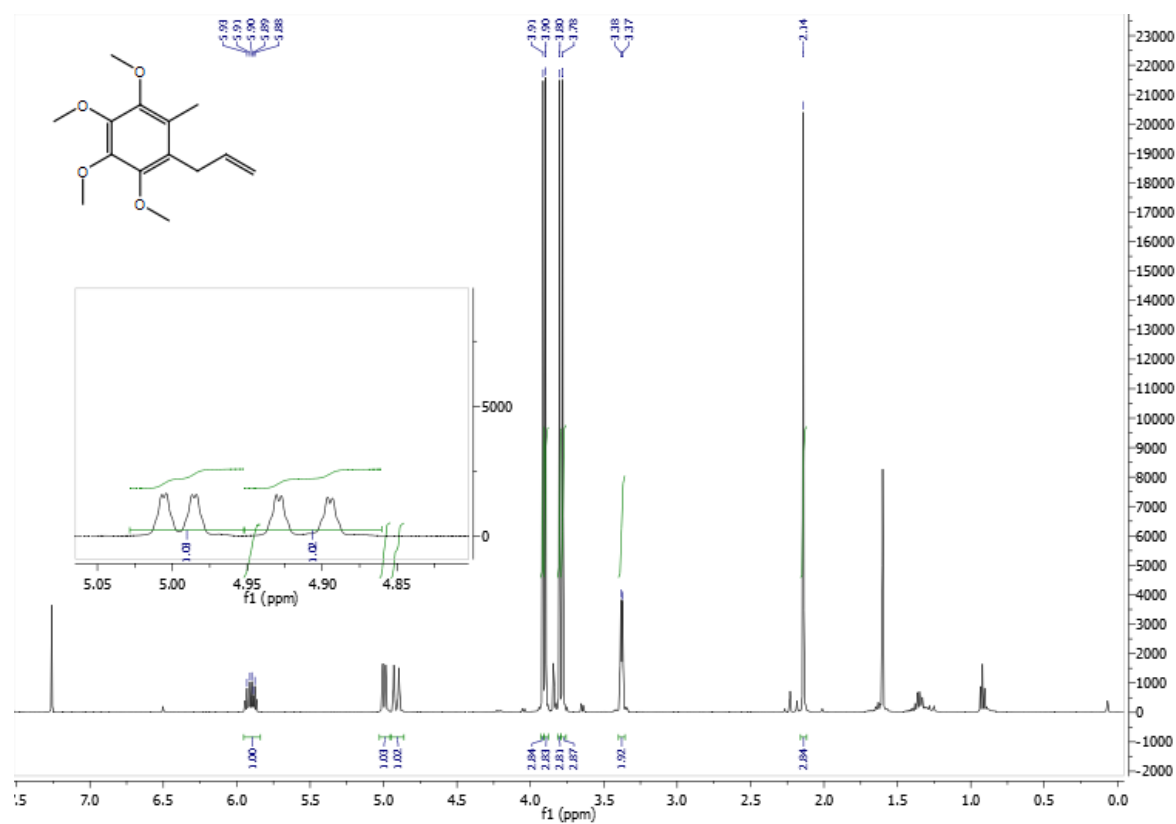
^{13}C NMR Spectrum (125 MHz, CDCl_3) of 2-allyl-3,5,6-trimethyl-1,4-phenylene diacetate (**7a**)IR Spectrum of 1-allyl-2,5-dimethoxy-3,4,6-trimethylbenzene (**7b**)

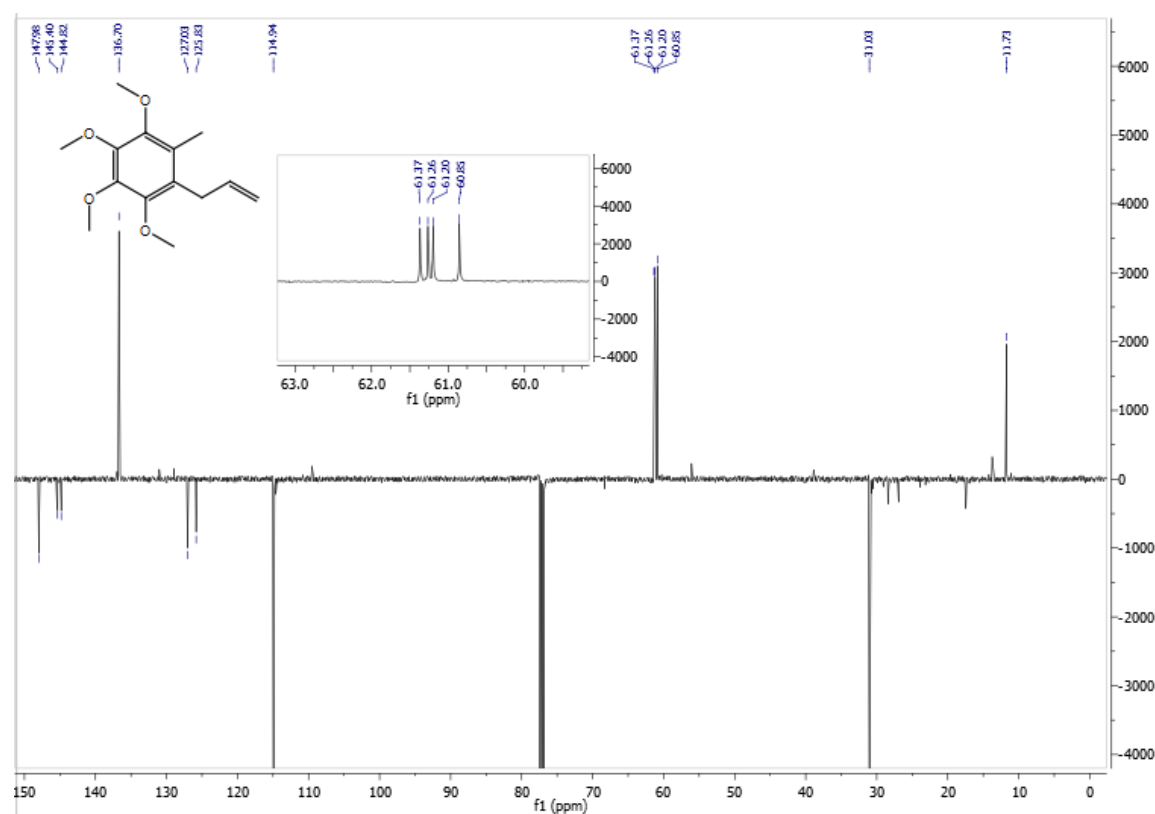
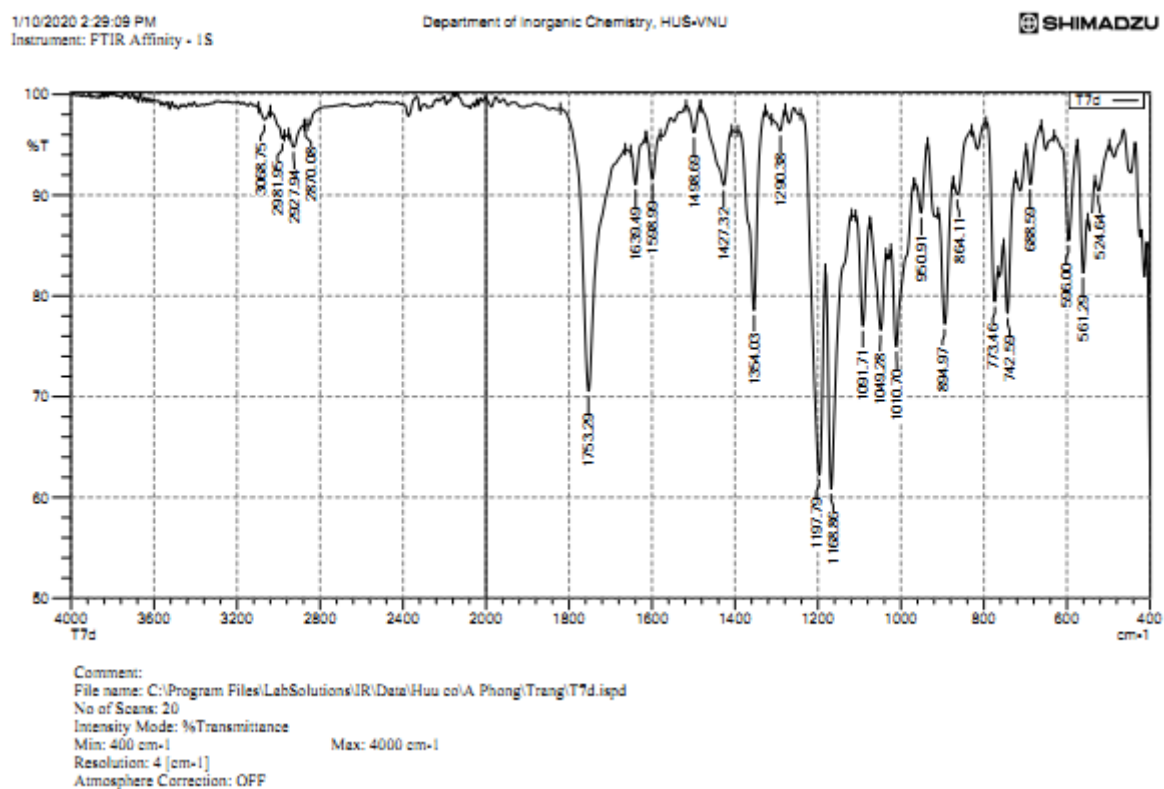
^1H NMR Spectrum (500 MHz, CDCl_3) of 1-allyl-2,5-dimethoxy-3,4,6-trimethylbenzene (**7b**) ^{13}C NMR Spectrum (125 MHz, CDCl_3) of 1-allyl-2,5-dimethoxy-3,4,6-trimethylbenzene (**7b**)

IR Spectrum of 1-allyl-2,3,4,5-tetramethoxy-6-methylbenzene (7c)



¹H NMR Spectrum (500 MHz, CDCl₃) of 1-allyl-2,3,4,5-tetramethoxy-6-methylbenzene (**7c**)



^{13}C NMR Spectrum (125 MHz, CDCl_3) of 1-allyl-2,3,4,5-tetramethoxy-6-methylbenzene (7c)IR Spectrum (500 MHz, CDCl_3) of 2-allyl-3-methylnaphthalene-1,4-diyl diacetate (7d)

Chemical structure of compound 10a is shown in the top left. The ¹H NMR spectrum (CDCl₃) is displayed below, with two zoomed-in regions for the aromatic and aliphatic regions.

Chemical Structure of 10a: CC(=O)OC1=C(C=C(C=C1)C=C)C(=O)OC2=CC=CC=C2

¹H NMR Spectrum (CDCl₃):

- Chemical Shifts (ppm):** 7.73, 7.71, 7.67, 7.65, 7.48, 7.47, 7.46, 5.01, 5.00, 4.98, 4.96, 3.46, 2.27.
- Integrations:** 1.00, 1.09, 2.13, 0.99, 1.03, 1.03, 2.02, 3.08, 3.11, 3.18.

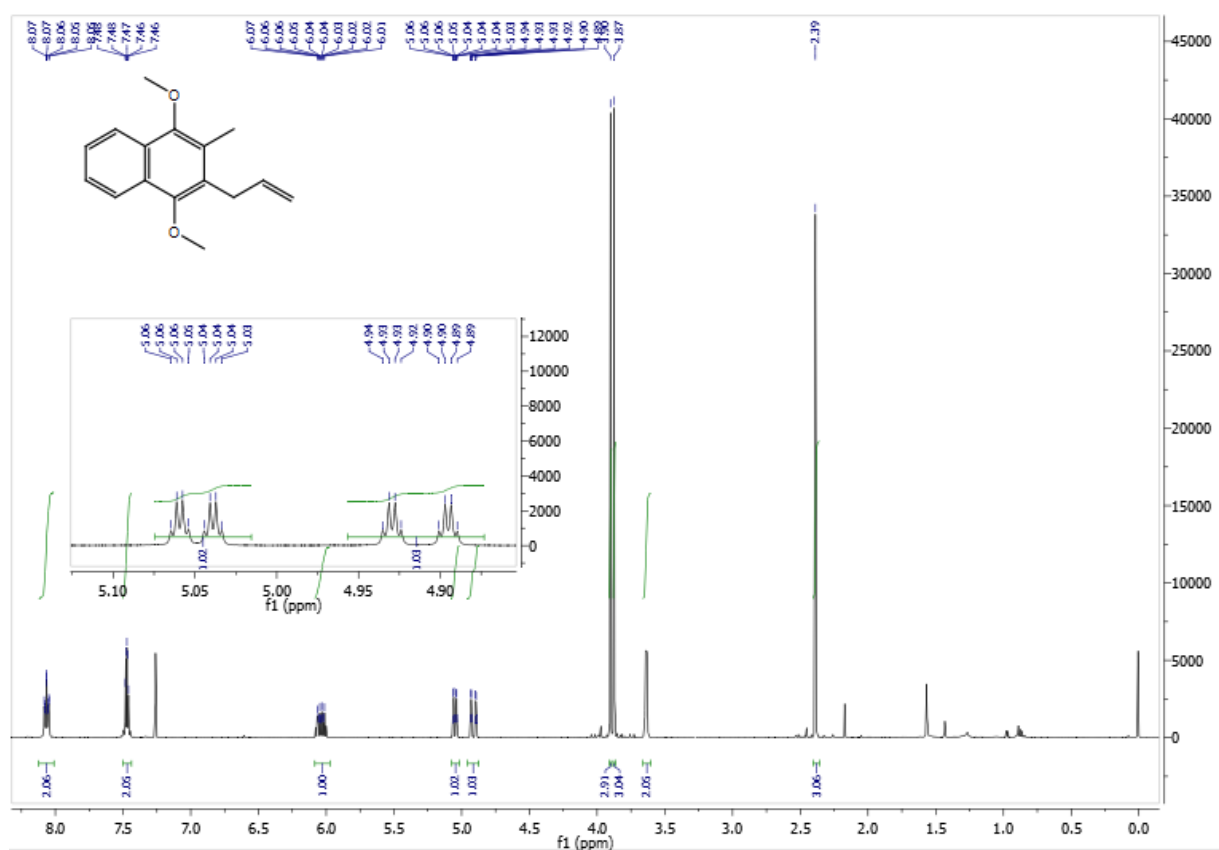
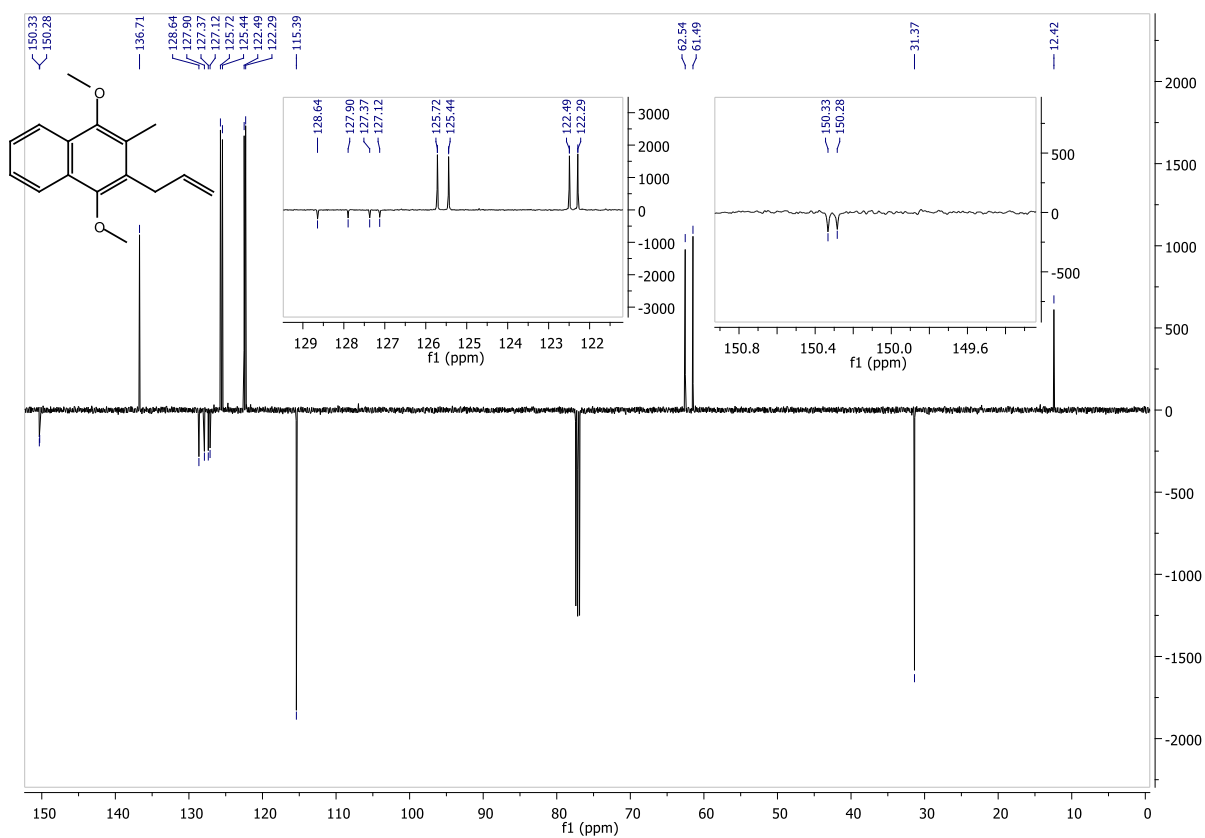
Zoomed-in Regions:

- Aromatic Region (7.45-7.75 ppm):** Peaks at 7.73, 7.71, 7.67, 7.65, 7.48, 7.47, 7.46 ppm. Integrations: 1.00, 1.09, 2.13.
- Aliphatic Region (4.96-5.08 ppm):** Peaks at 5.07, 5.07, 5.07, 5.06, 5.05, 5.05, 5.04, 5.04, 5.01, 5.01, 4.98, 4.98 ppm. Integrations: 1.03, 1.03.

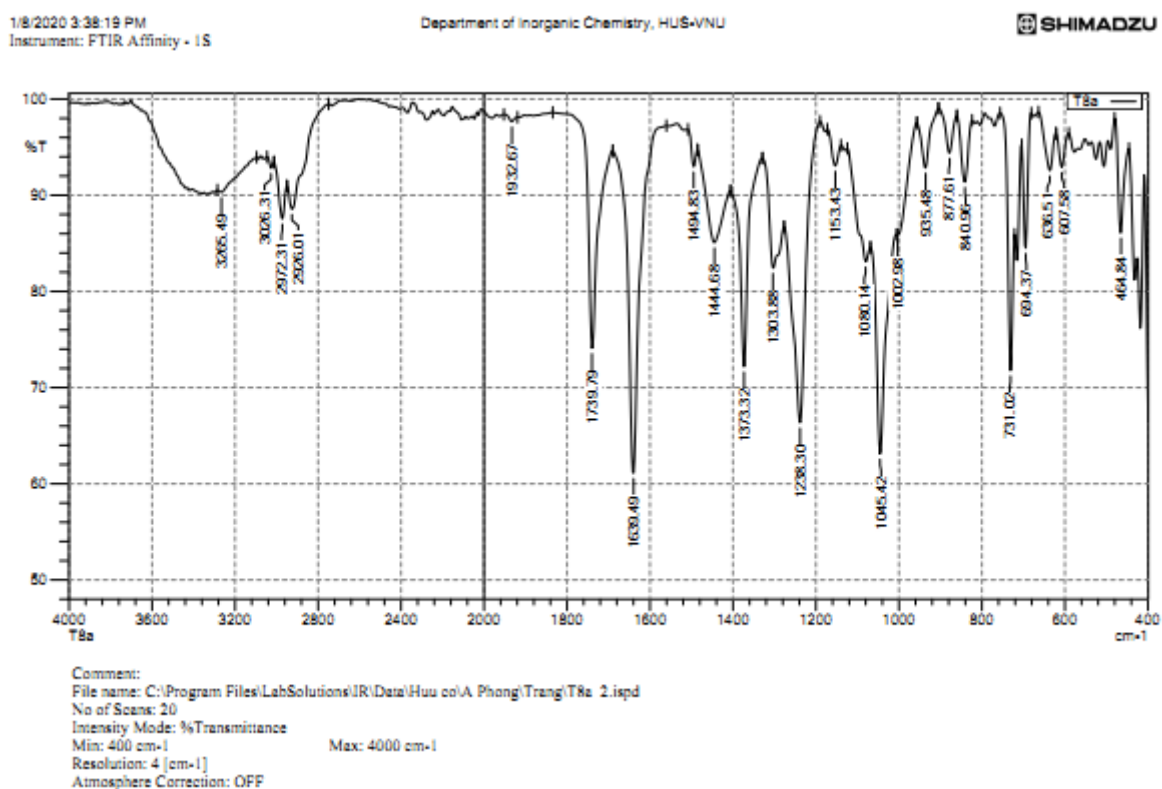
Chemical structure: CC(=O)OC1=C(C)C(C=C)C(OC(=O)C)=C1c2ccccc2

¹³C NMR spectrum (ppm):

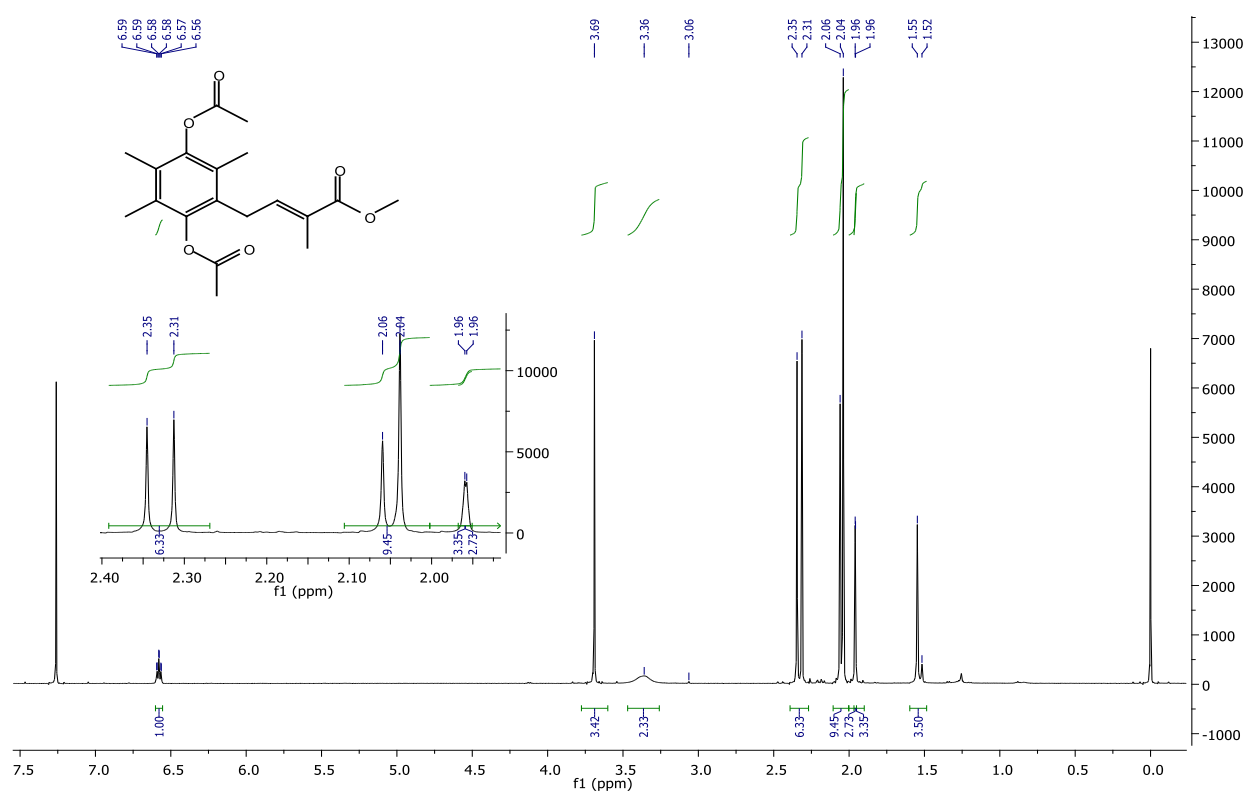
- 169.65
- 142.91
- 142.79
- 134.72
- 128.41
- 127.13
- 126.70
- 126.47
- 126.68
- 126.29
- 121.37
- 116.35
- 32.14
- 20.88
- 20.78
- 13.19
- 121.63
- 121.37
- 142.91
- 142.79
- 134.72

^1H NMR Spectrum (500 MHz, CDCl_3) of 2-allyl-1,4-dimethoxy-3-methylnaphthalene (7e) ^{13}C NMR Spectrum (125 MHz, CDCl_3) of 2-allyl-1,4-dimethoxy-3-methylnaphthalene (7e)

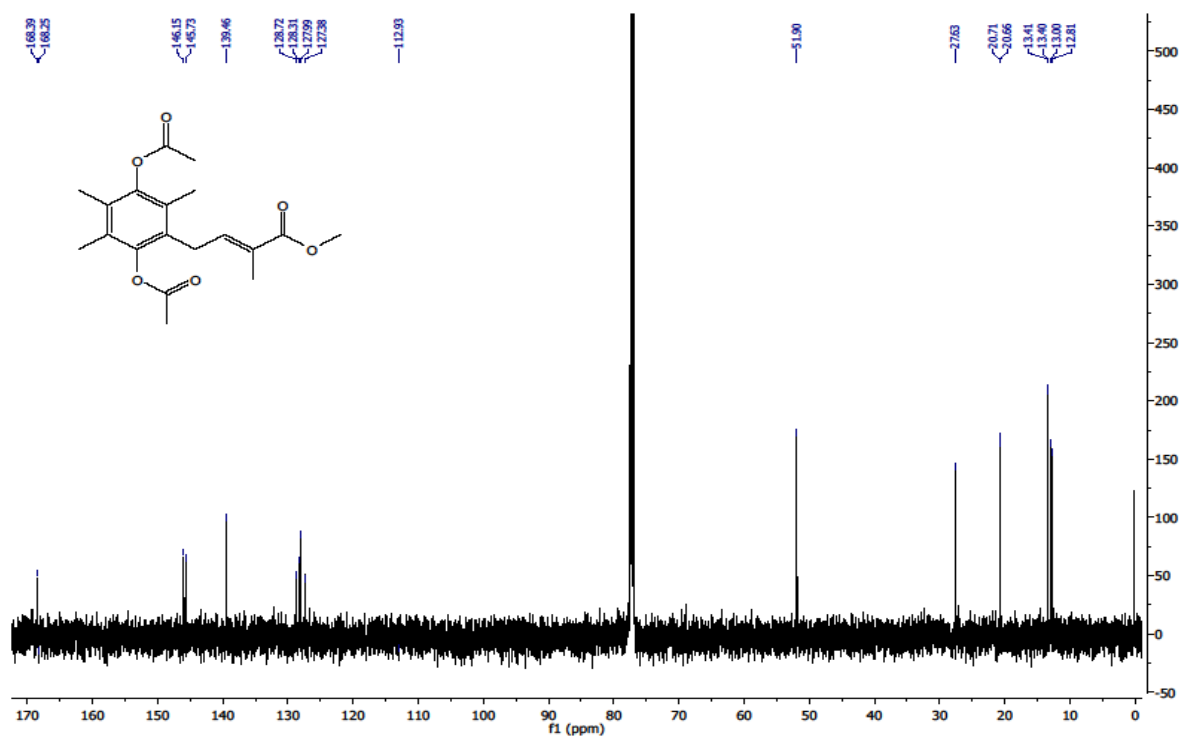
IR Spectrum of (*E*)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (**8a**)



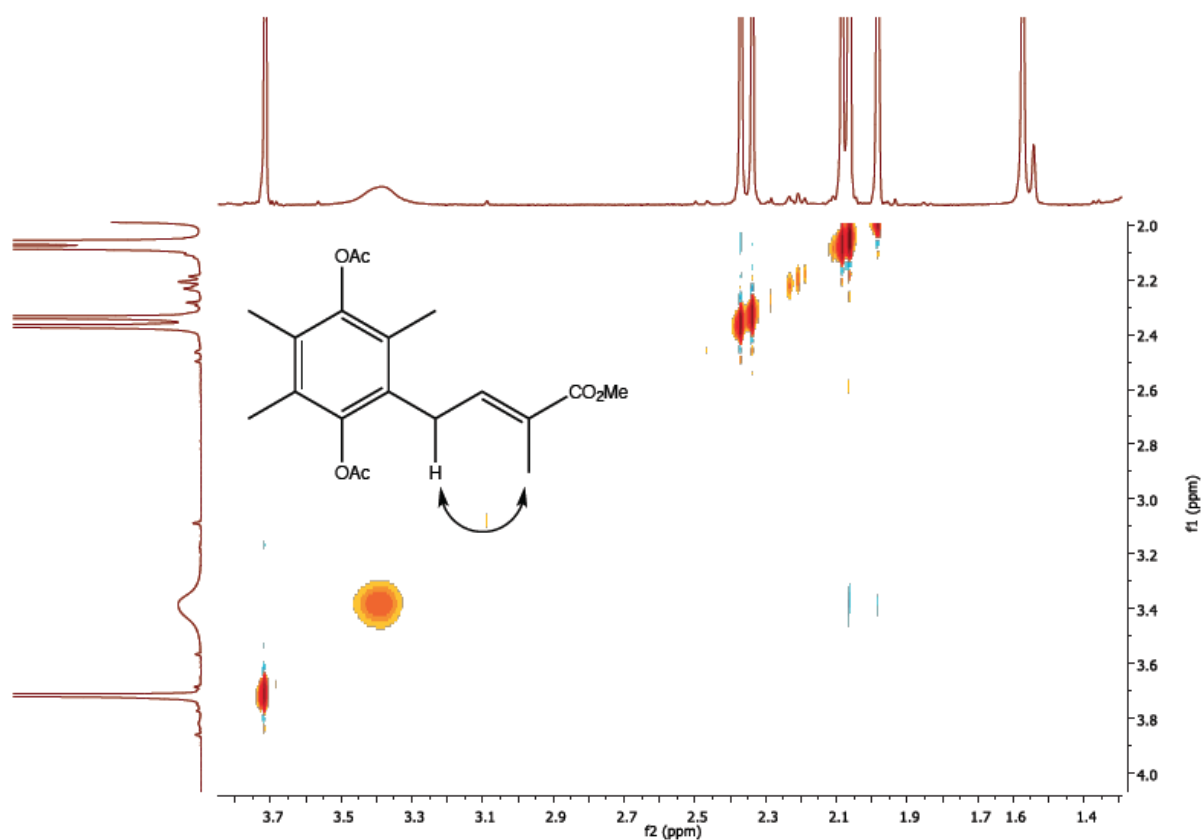
¹H NMR Spectrum (500 MHz, CDCl₃) of (*E*)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (**8a**)

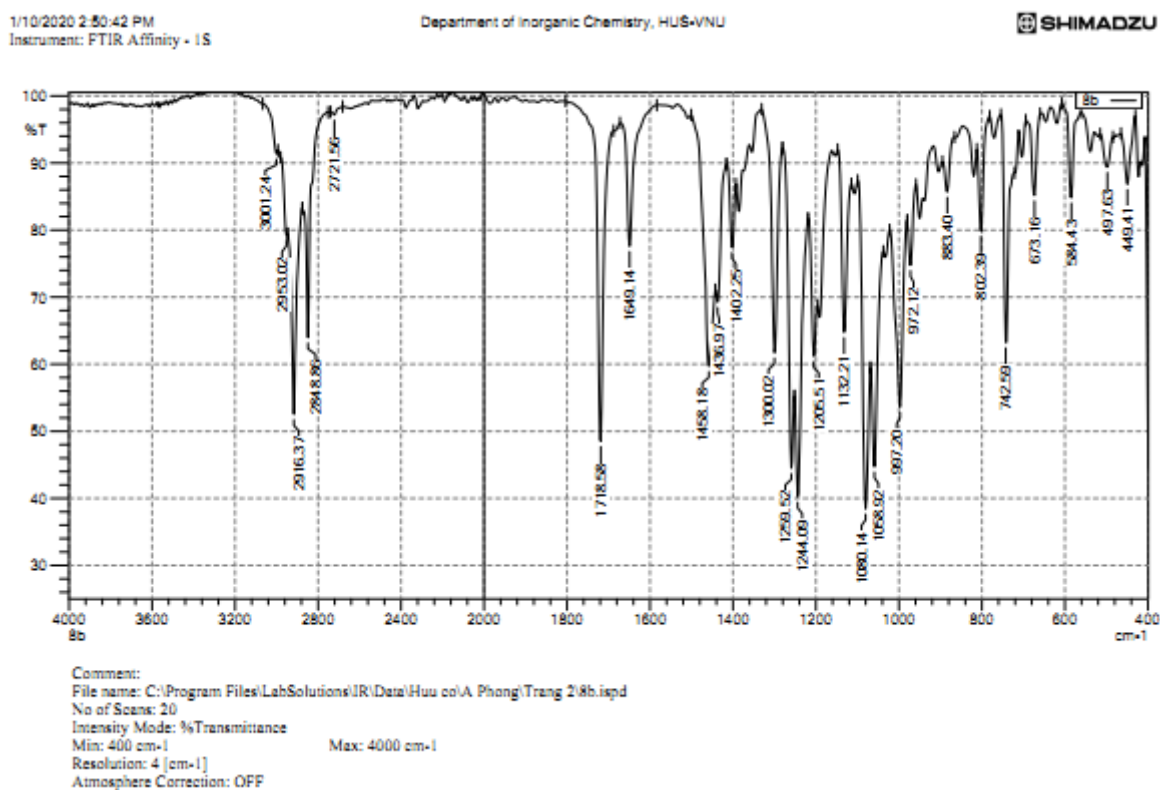
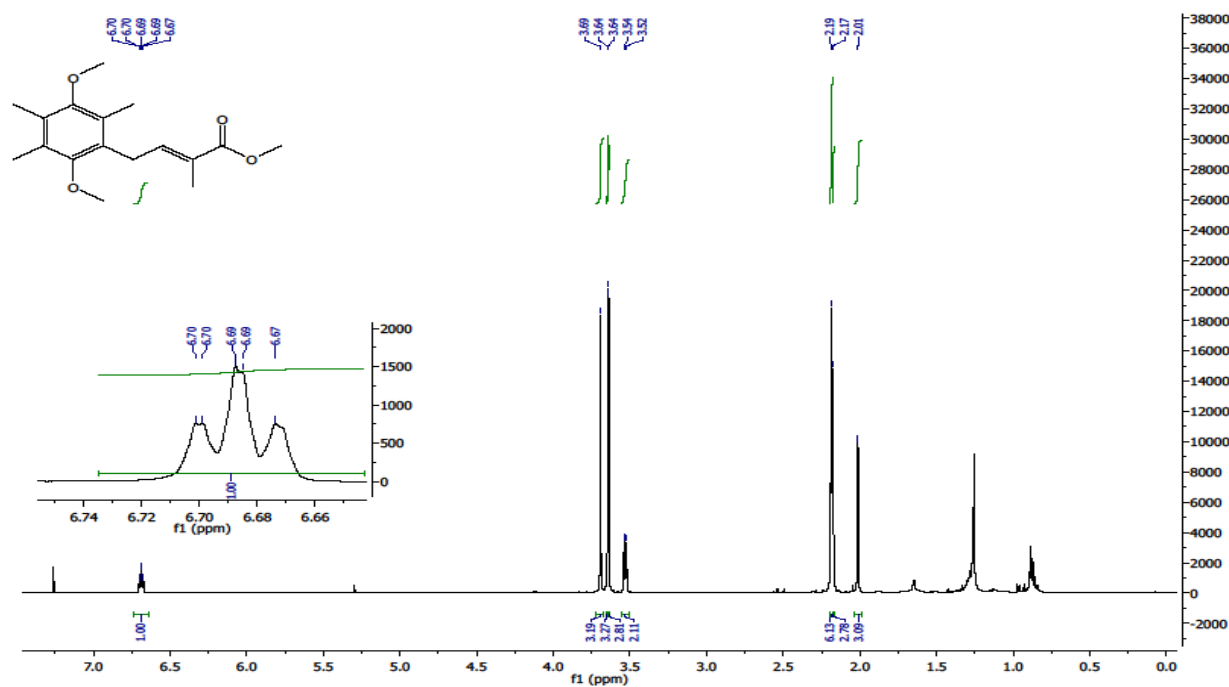


^{13}C NMR Spectrum (125 MHz, CDCl_3) of (*E*)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (**8a**)

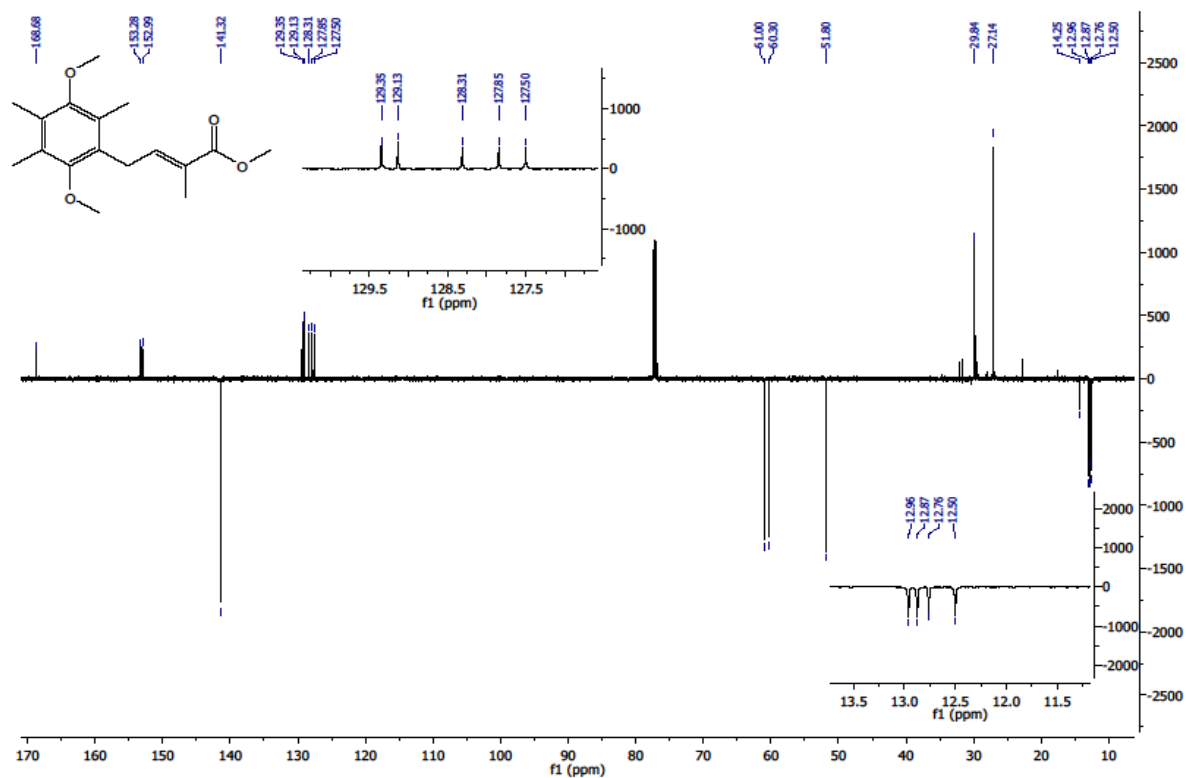


Noesy Spectrum (125 MHz, CDCl_3) of (*E*)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3,5,6-trimethyl-1,4-phenylene diacetate (**8a**)

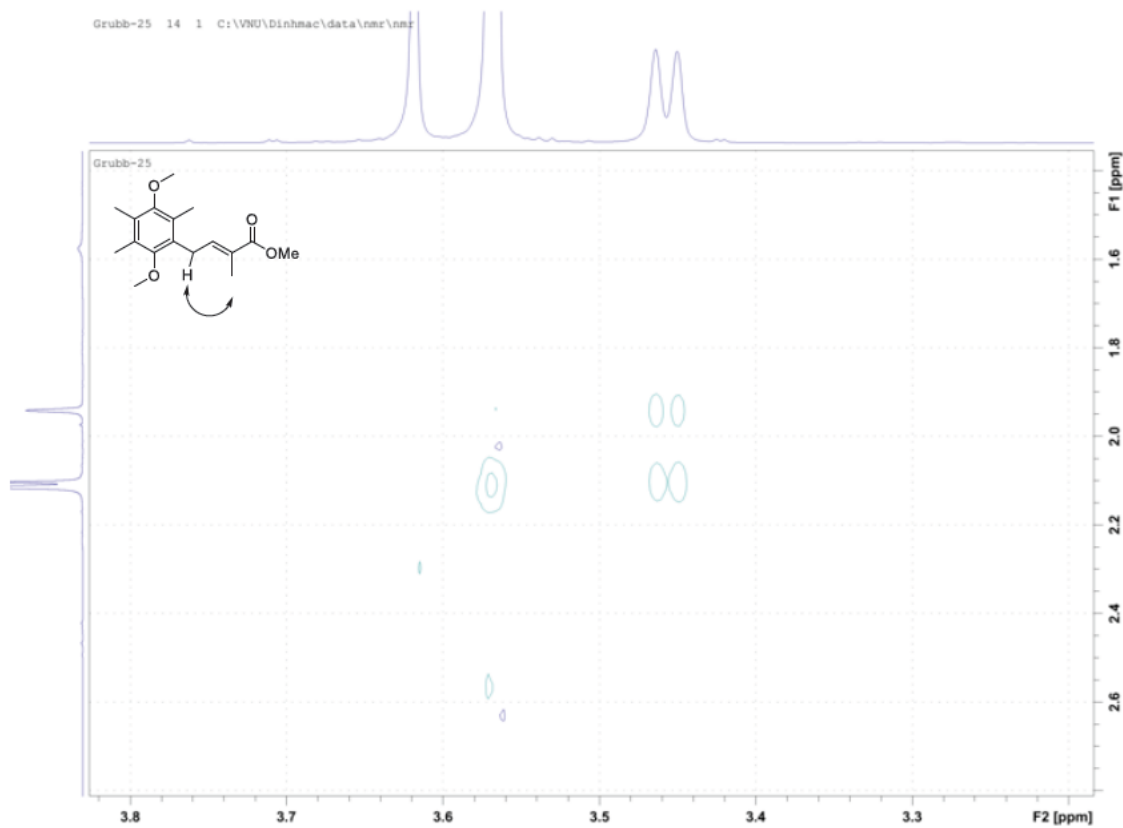


IR Spectrum of methyl (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (**8b**)¹H NMR Spectrum (500 MHz, CDCl₃) of methyl (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (**8b**)

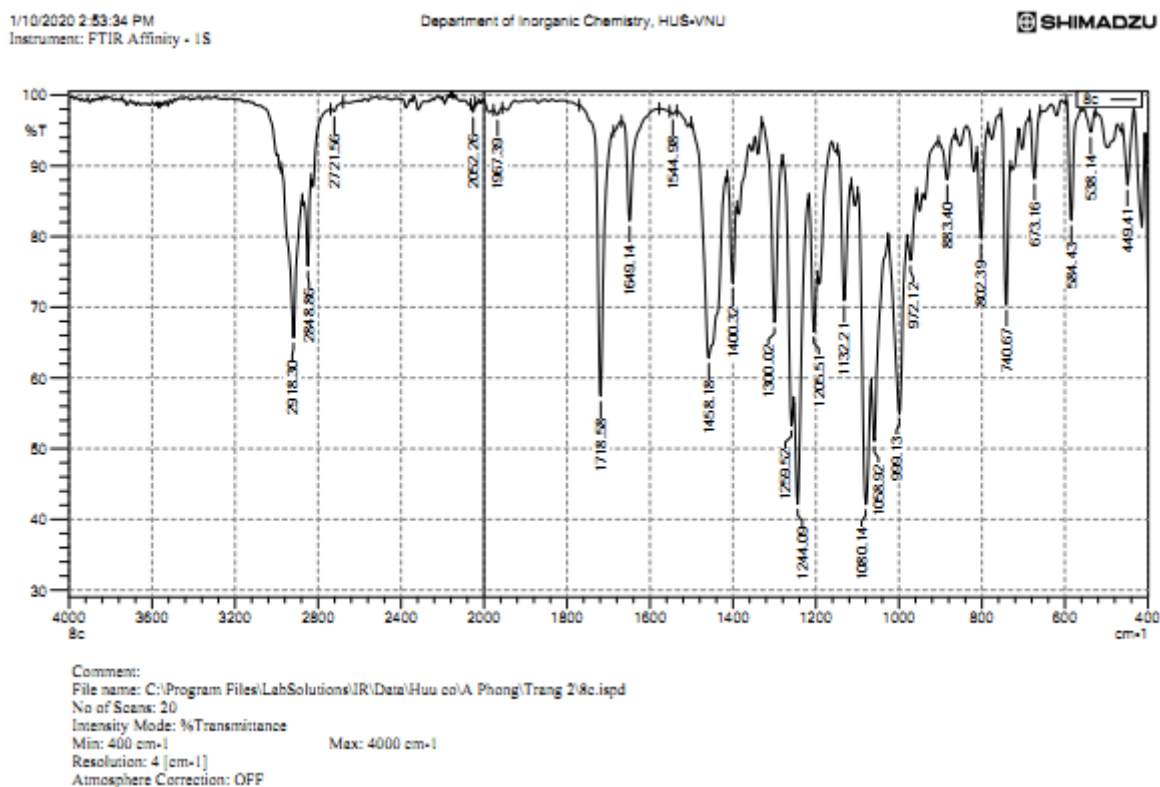
^{13}C NMR Spectrum (125 MHz, CDCl_3) of methyl (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (**8b**)



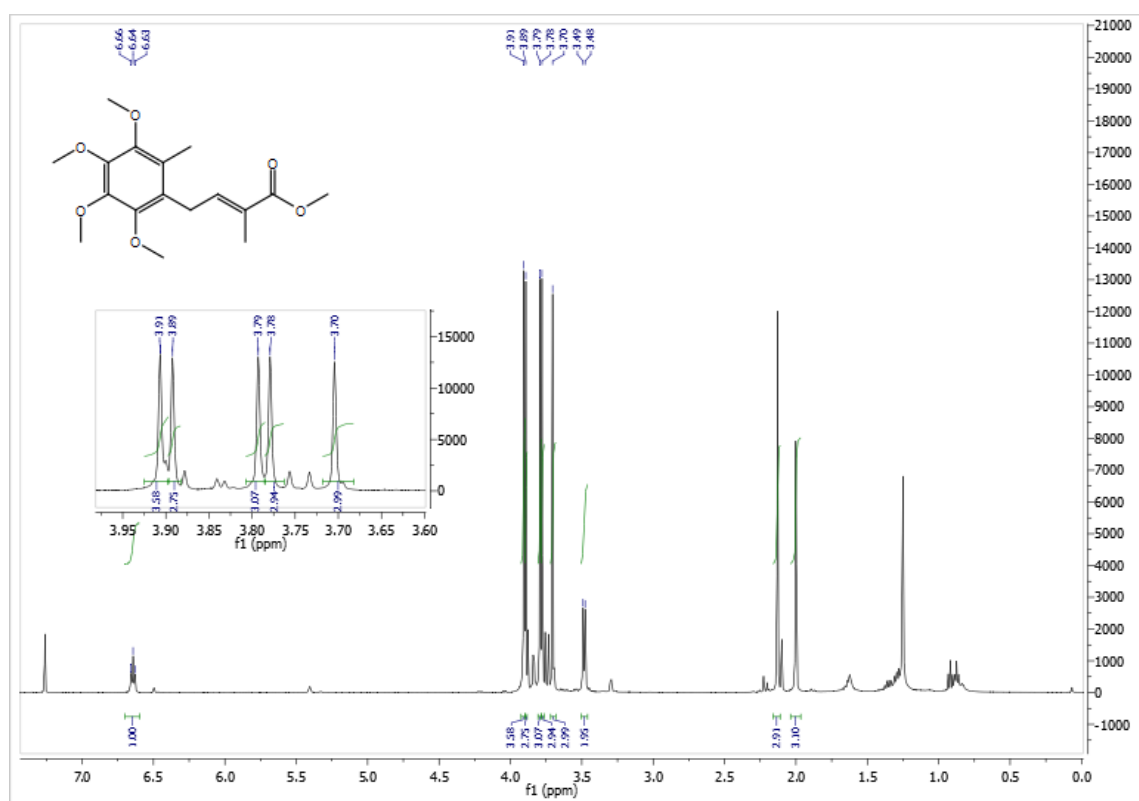
NOESY spectrum of methyl (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylphenyl)-2-methylbut-2-enoate (**8b**)



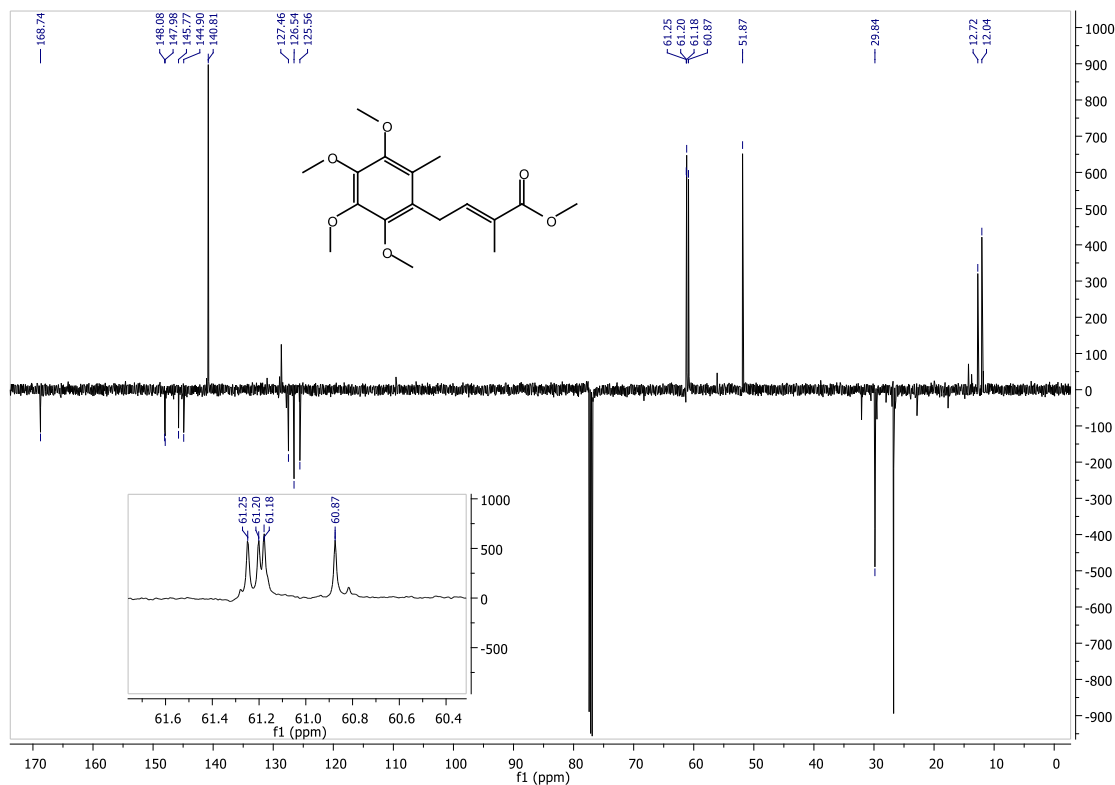
IR Spectrum of methyl (*E*)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-enoate (**8c**)



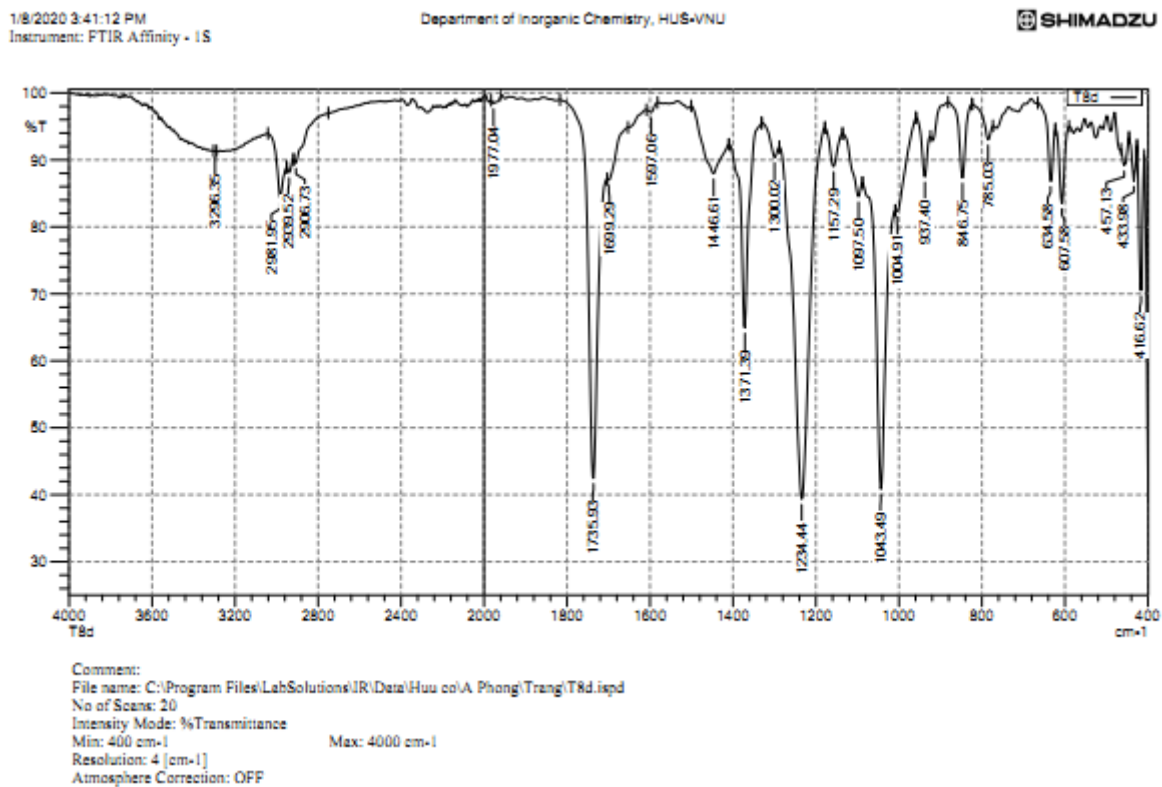
¹H NMR Spectrum (500 MHz, CDCl₃) of methyl (*E*)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-enoate (**8c**)



^{13}C NMR Spectrum (125 MHz, CDCl_3) of methyl (*E*)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-enoate (**8c**)



IR Spectrum of (*E*)-2-(4-methoxy-3-methyl-4-oxobut-2-en-1-yl)-3-methylnaphthalene-1,4-diyl diacetate (**8d**)

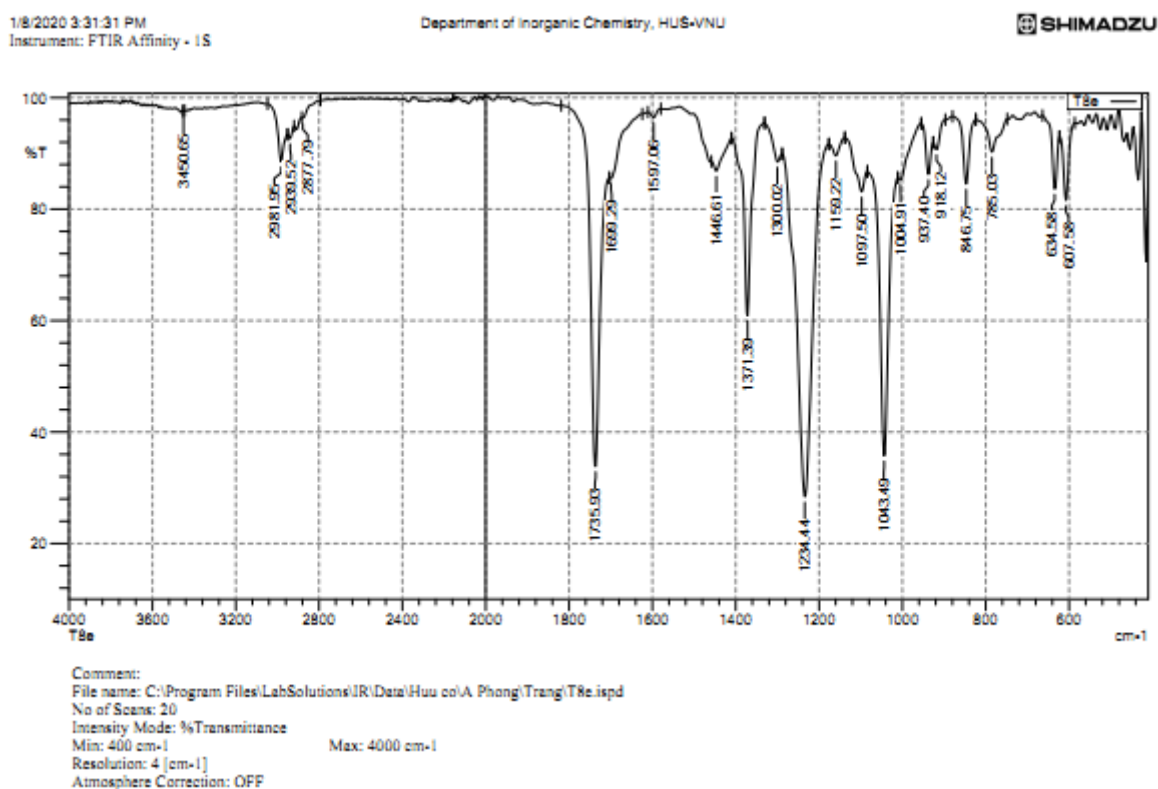


Chemical structure of compound 10 is shown above the spectrum. The spectrum displays two regions: the top region (120-170 ppm) shows aromatic and carbonyl signals, while the bottom region (0-60 ppm) shows aliphatic signals. Key peaks are labeled with their chemical shifts in ppm.

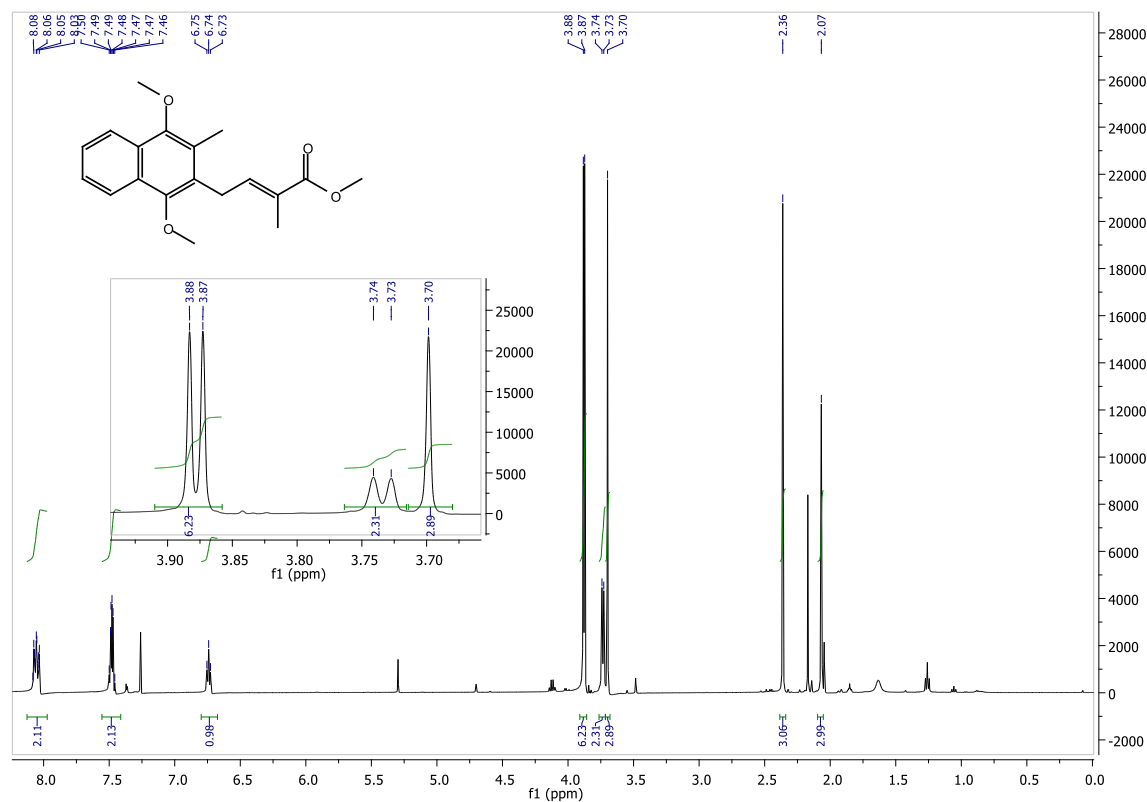
Chemical Shifts (ppm):

- 169.37, 168.30
- 142.95, 142.83
- 138.86
- 142.95, 142.83, 138.86
- 128.57, 128.56, 128.55, 126.96, 126.84, 126.69, 126.64, 121.44
- 51.96
- 27.87
- 20.80, 20.77
- 13.44, 12.91

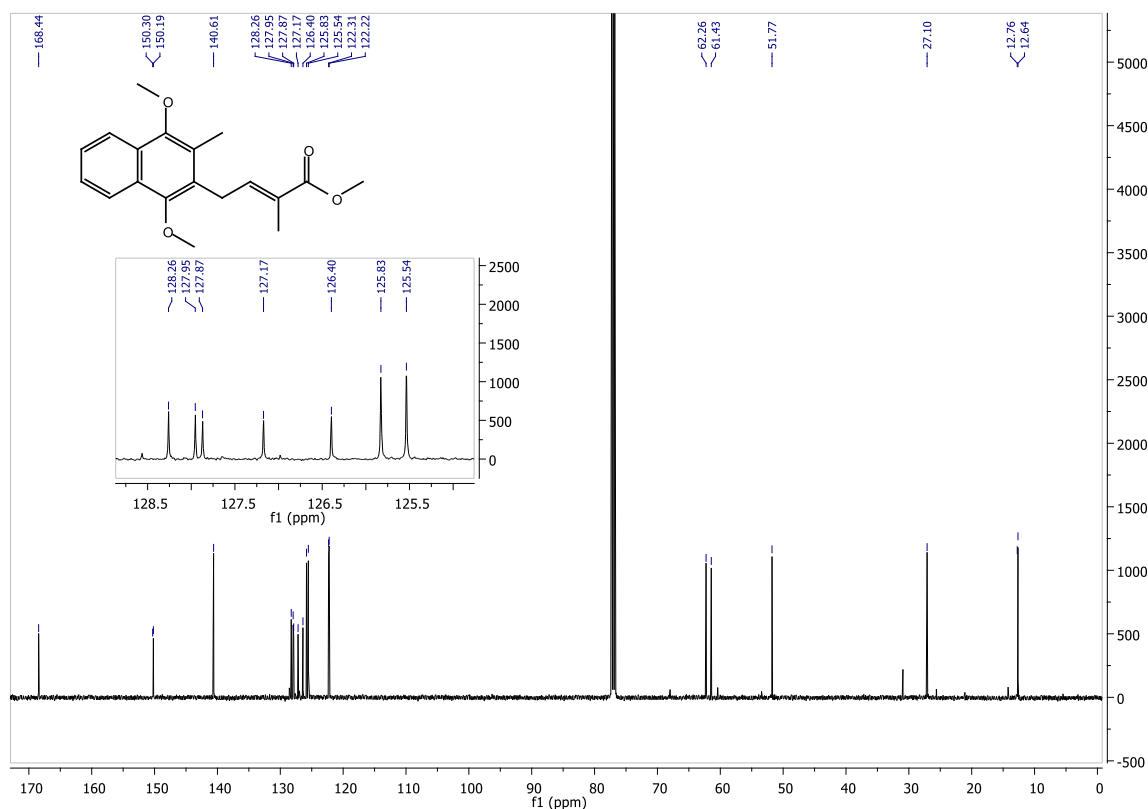
IR Spectrum of methyl (*E*)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-enoate (**8e**)



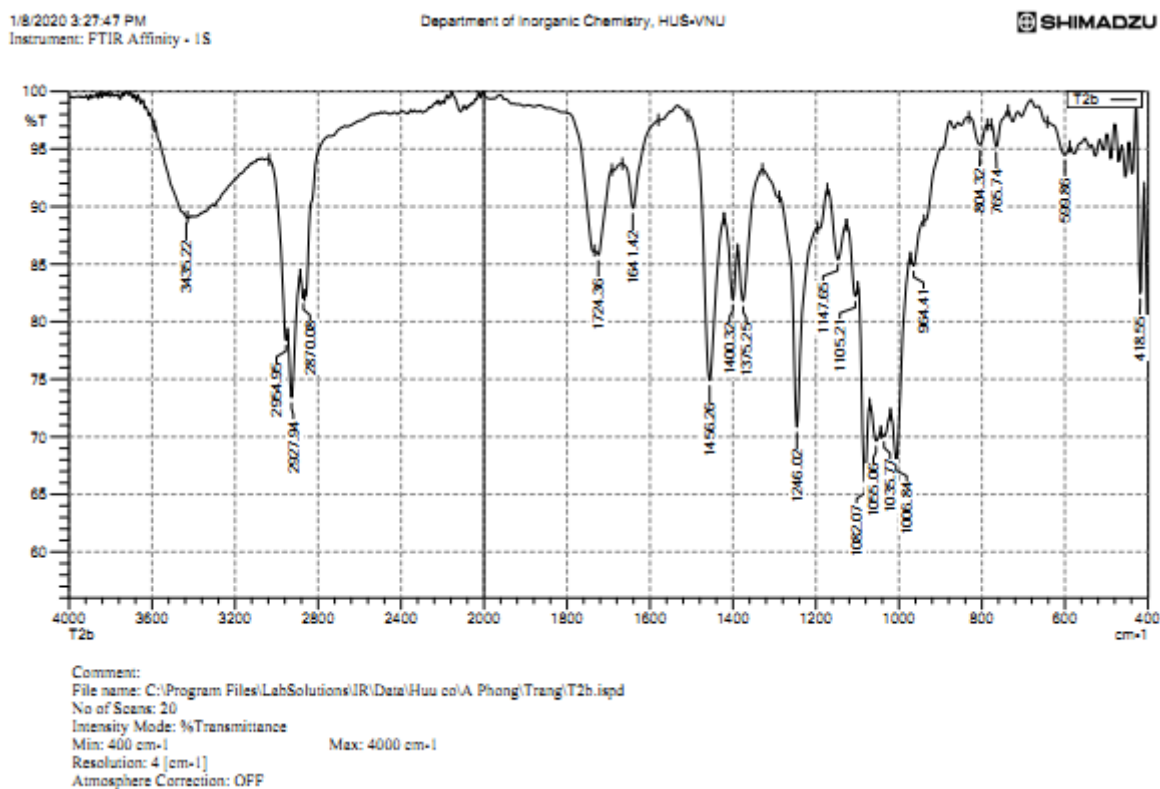
¹H NMR Spectrum (500 MHz, CDCl₃) of methyl (*E*)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-enoate (**8e**)



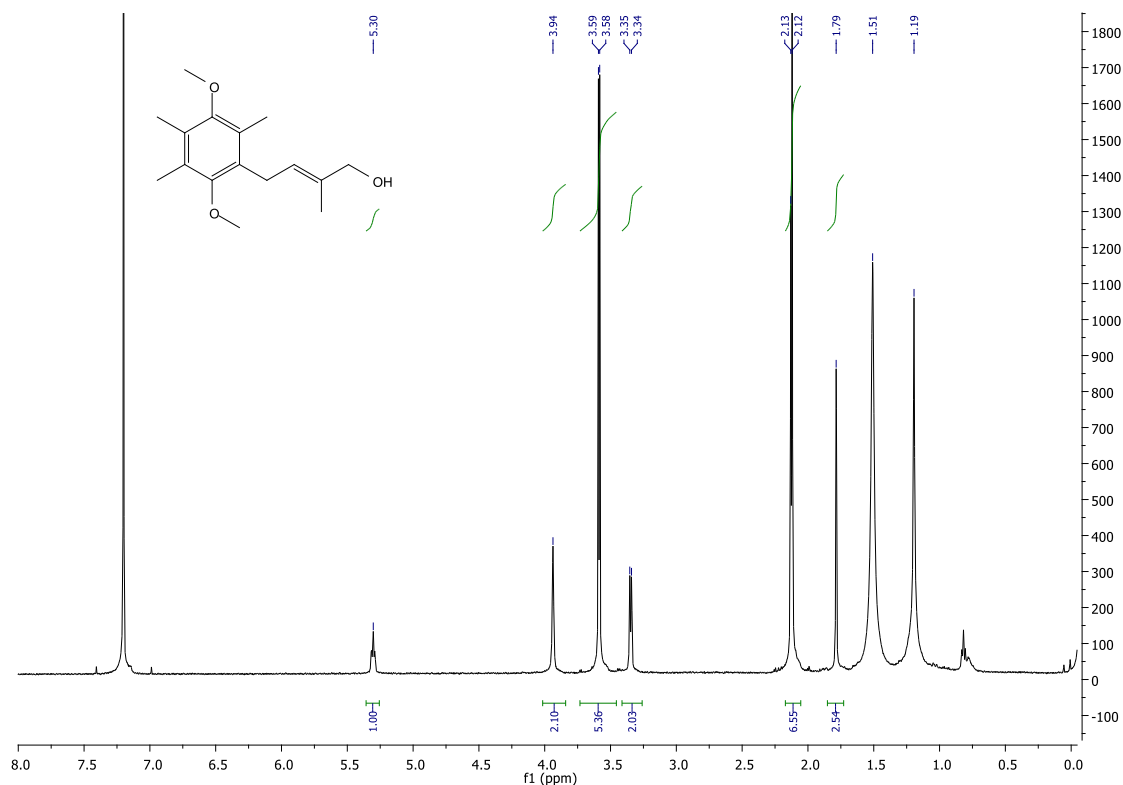
^{13}C NMR Spectrum (125 MHz, CDCl_3) of methyl (*E*)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-enoate (**8e**)



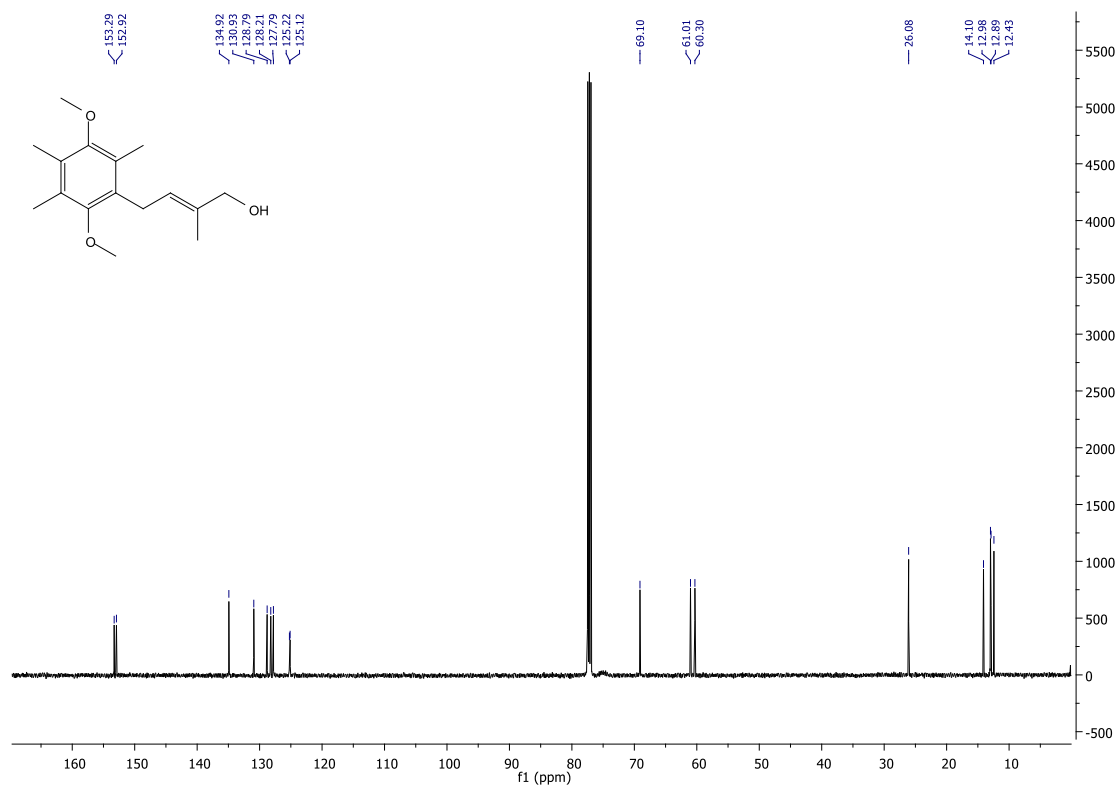
IR Spectrum of (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylcyclohexa-1,5-dienyl)-2-methylbut-2-en-1-ol (**2b**)



^1H NMR Spectrum (500 MHz, CDCl_3) of (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylcyclohexa-1,5-dienyl)-2-methylbut-2-en-1-ol (**2b**)



^{13}C NMR Spectrum (125 MHz, CDCl_3) of (*E*)-4-(2,5-dimethoxy-3,4,6-trimethylcyclohexa-1,5-dienyl)-2-methylbut-2-en-1-ol (**2b**)

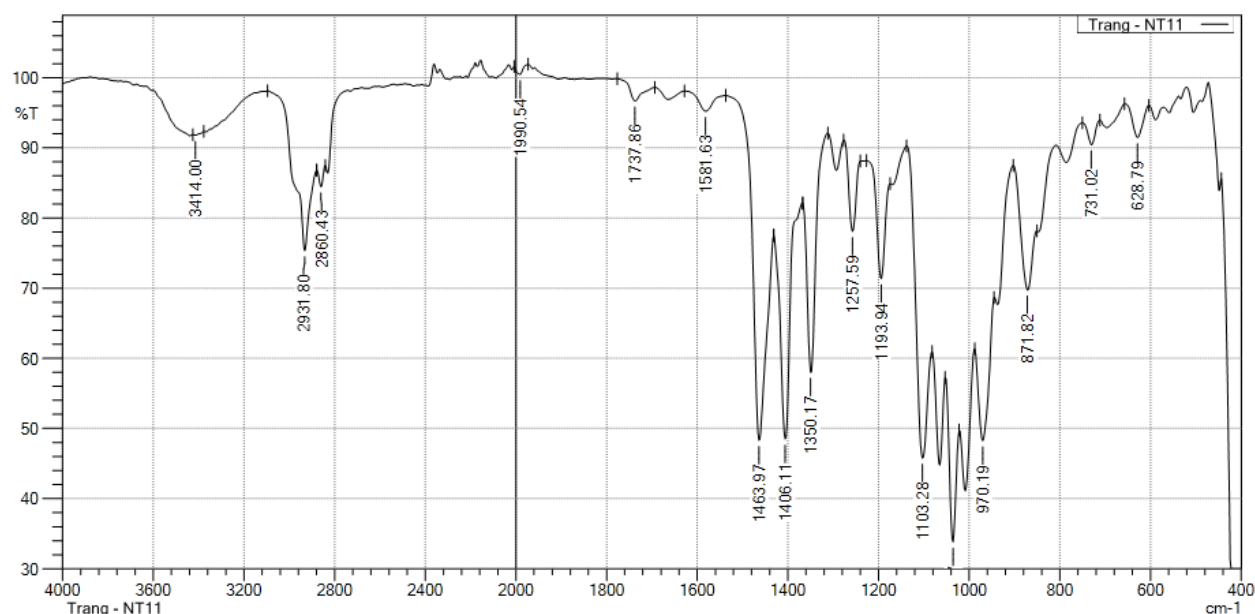


IR Spectrum of (*E*)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-en-1-ol (**2c**)

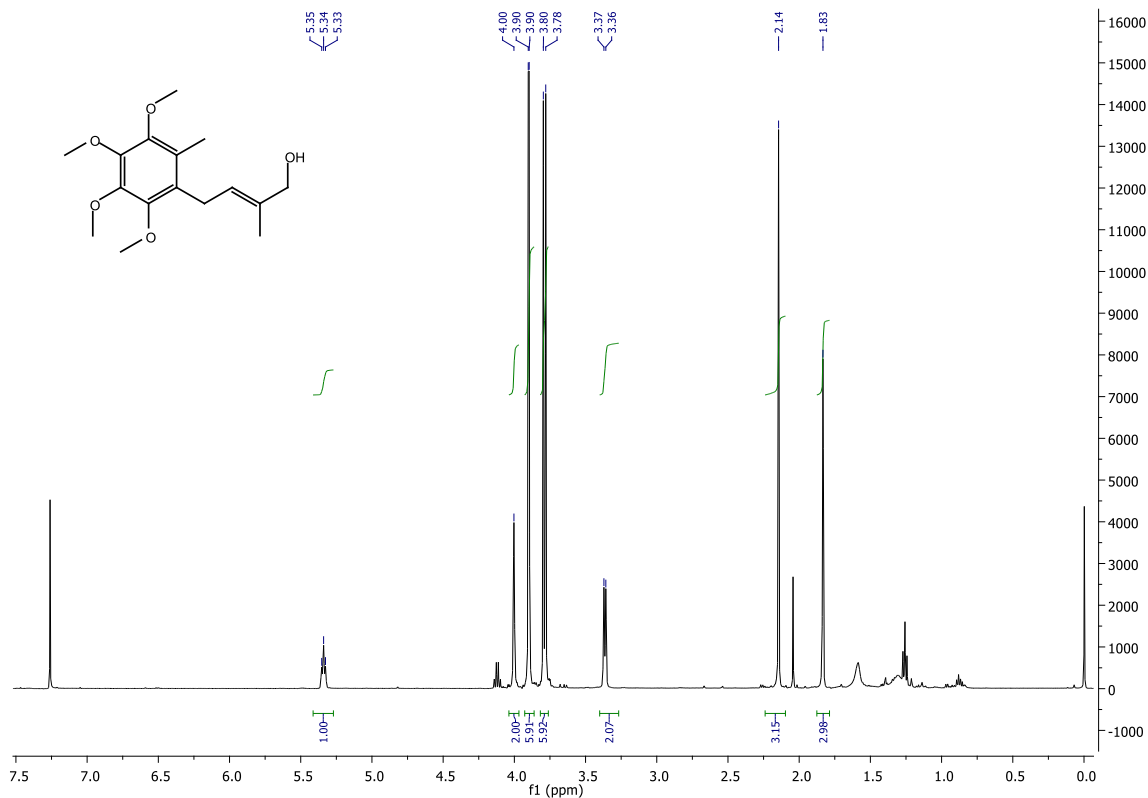
2/8/2018 9:50:59 AM
Instrument: FTIR Affinity - 1S

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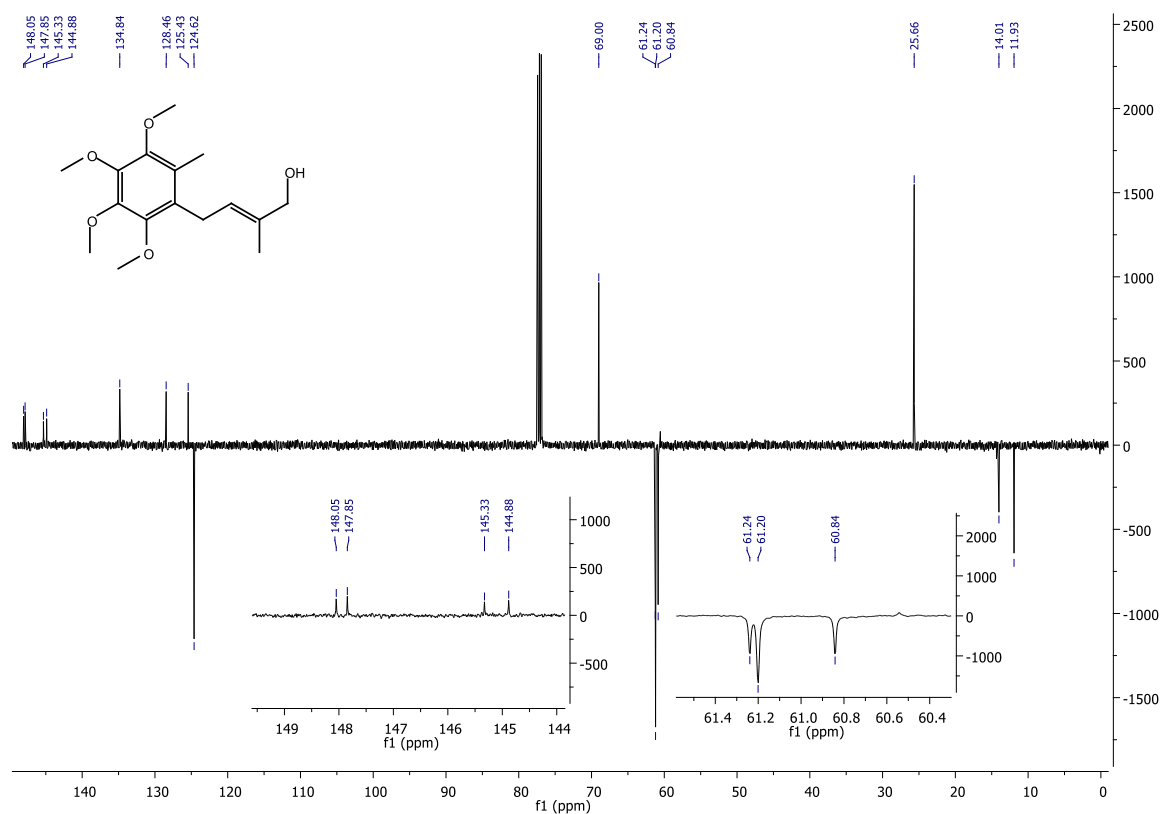
SHIMADZU



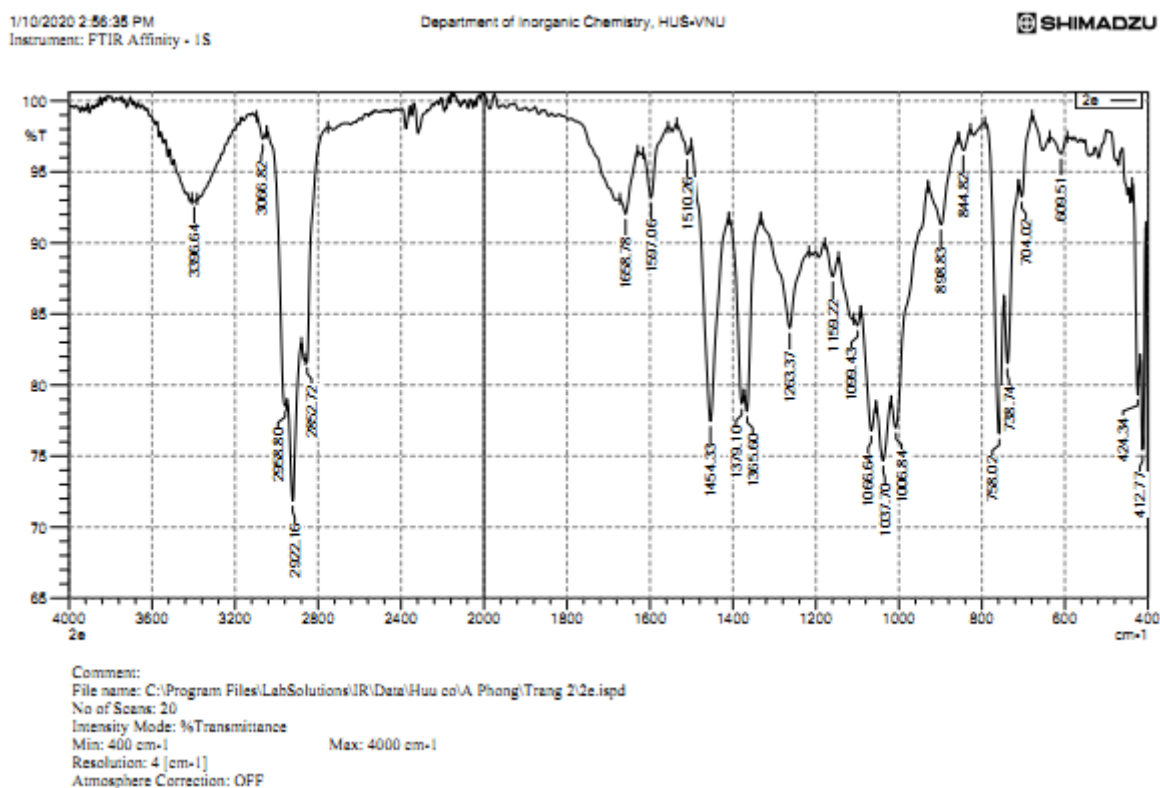
Spectrum (500 MHz, CDCl₃) of methyl (*E*)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-en-1-ol (**2c**)



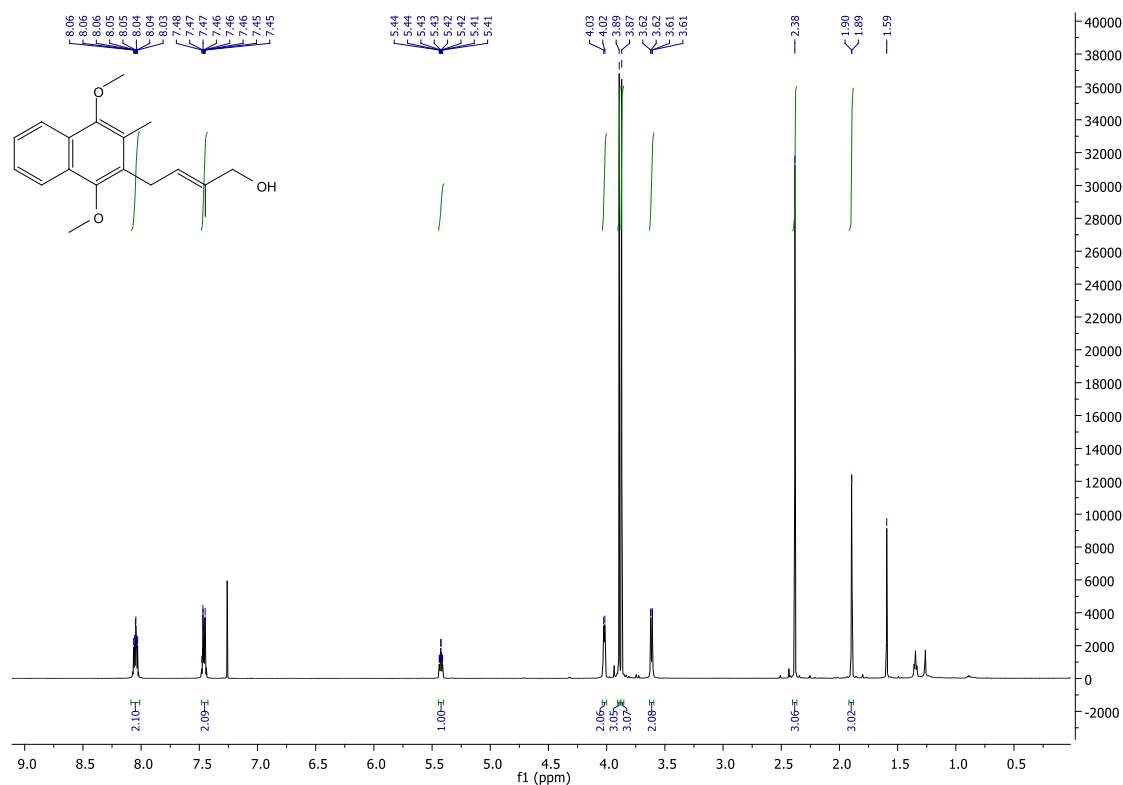
¹³C NMR Spectrum (125 MHz, CDCl₃) of (*E*)-2-methyl-4-(2,3,4,5-tetramethoxy-6-methylphenyl)but-2-en-1-ol (**2c**)



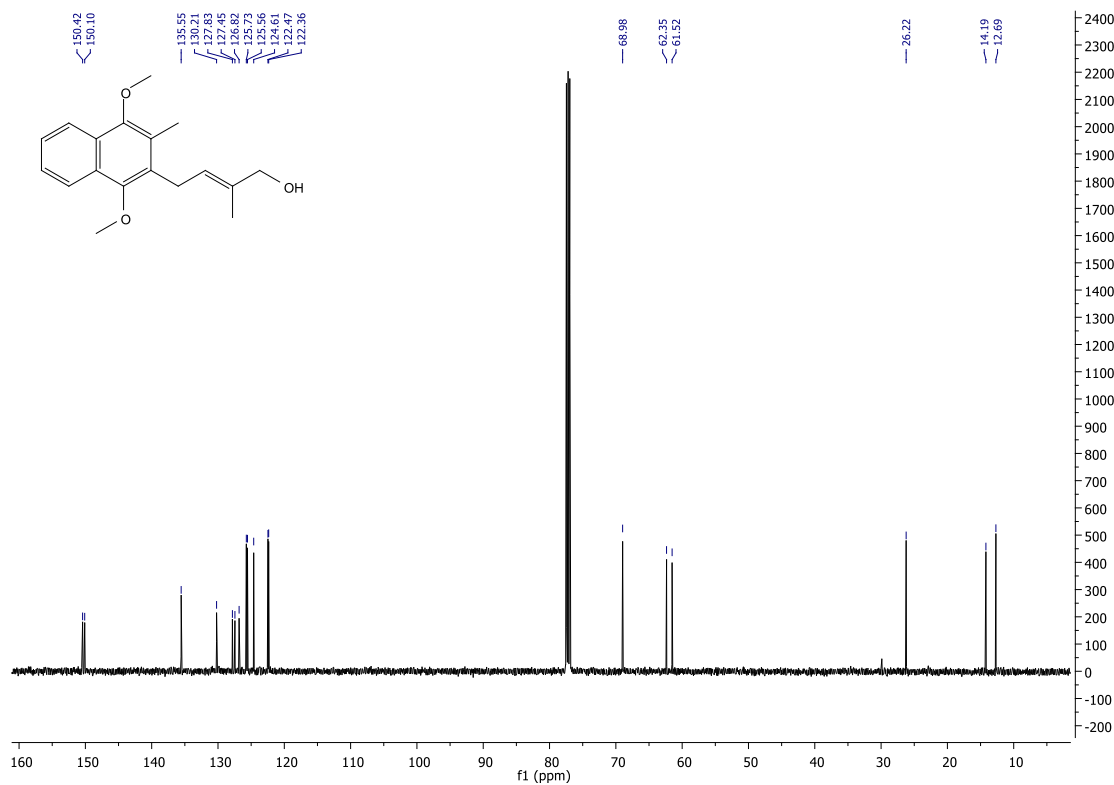
IR Spectrum of (E)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-en-1-ol (2e)



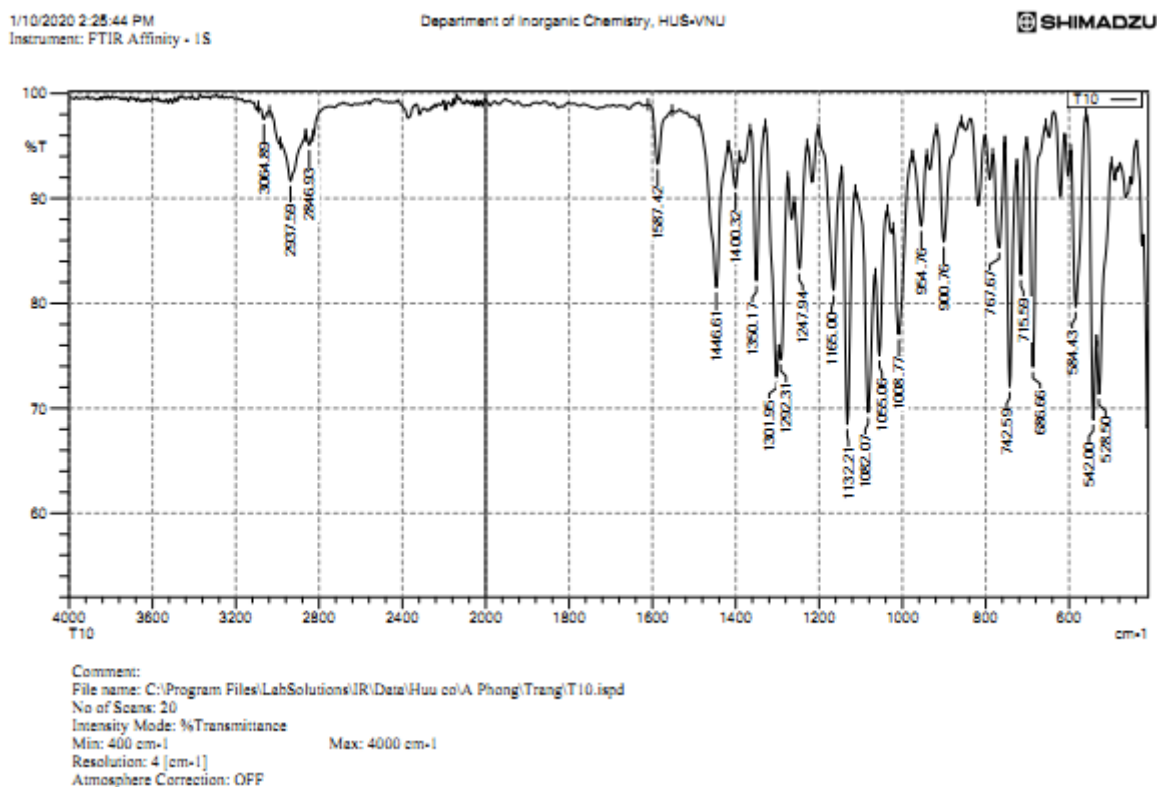
^1H NMR Spectrum (500 MHz, CDCl_3) of (*E*)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-en-1-ol (**2e**)



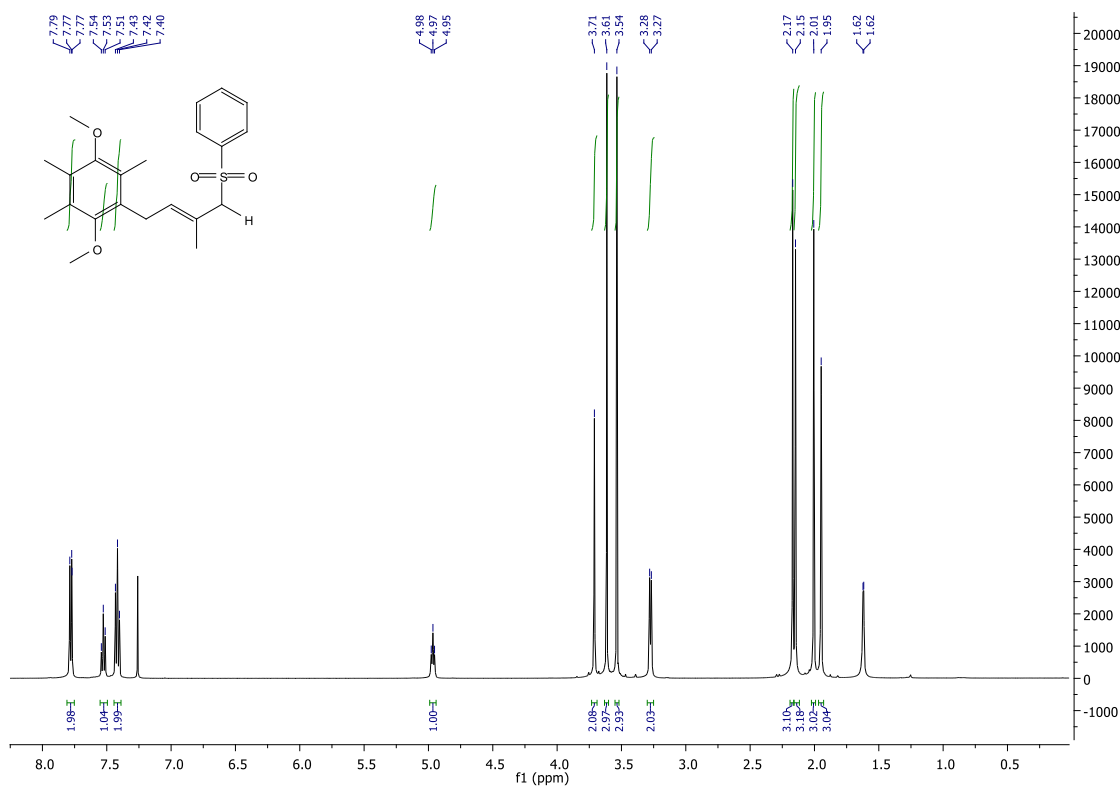
^{13}C NMR Spectrum (125 MHz, CDCl_3) of (*E*)-4-(1,4-dimethoxy-3-methylnaphthalen-2-yl)-2-methylbut-2-en-1-ol (**2e**)



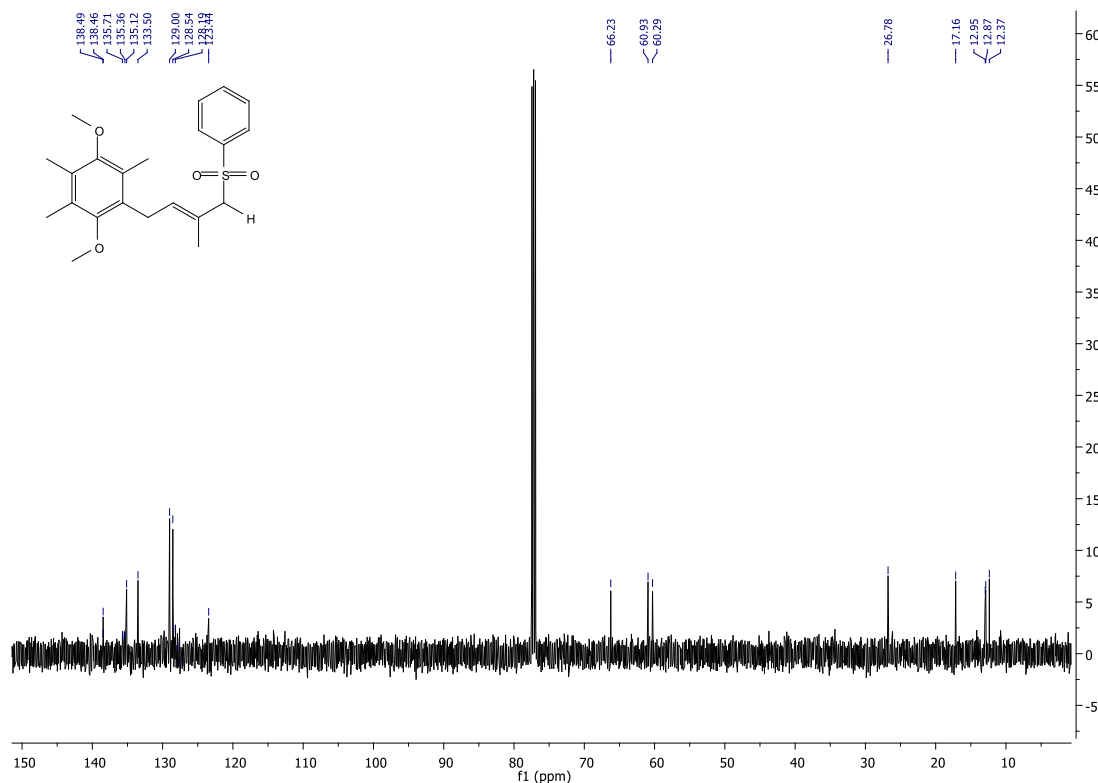
IR Spectrum of (*E*)-1,4-dimethoxy-2,3,5-trimethyl-6-(3-methyl-4-(phenylsulfonyl)but-2-enyl)benzene (**10**)



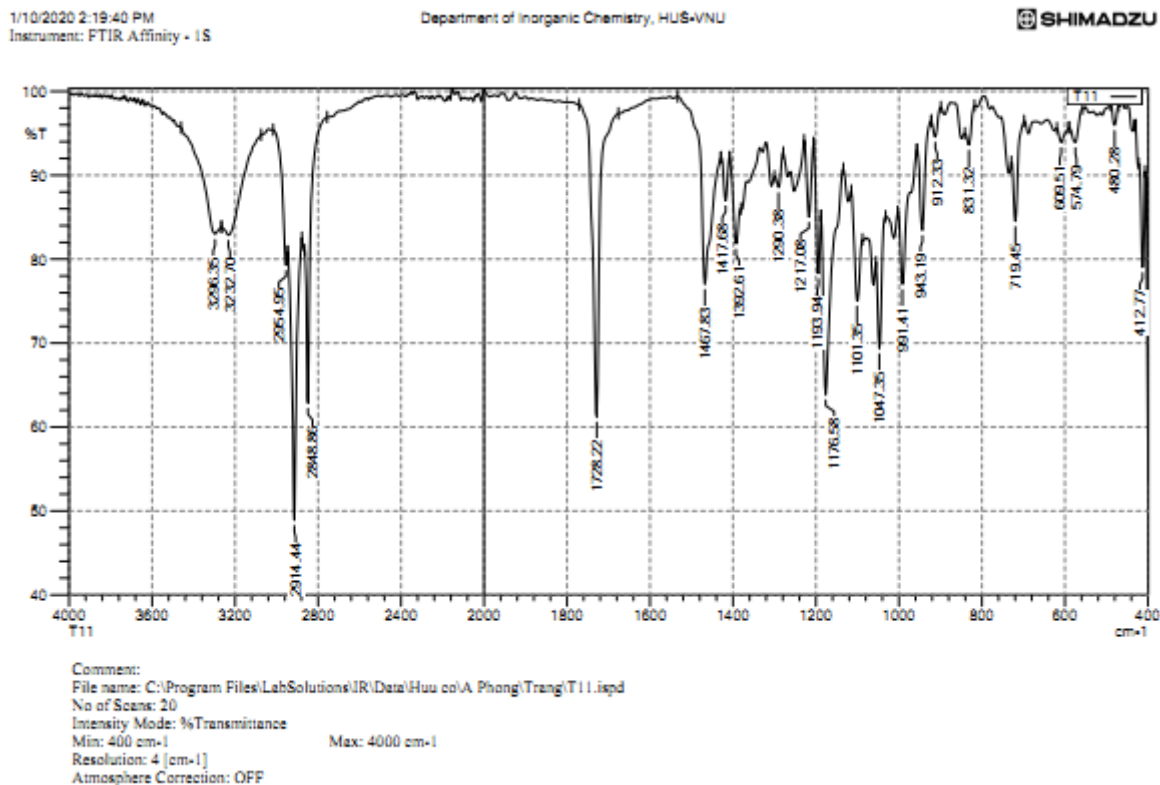
¹H NMR Spectrum (500 MHz, CDCl₃) of (*E*)-1,4-dimethoxy-2,3,5-trimethyl-6-(3-methyl-4-(phenylsulfonyl)but-2-enyl)benzene (**10**)



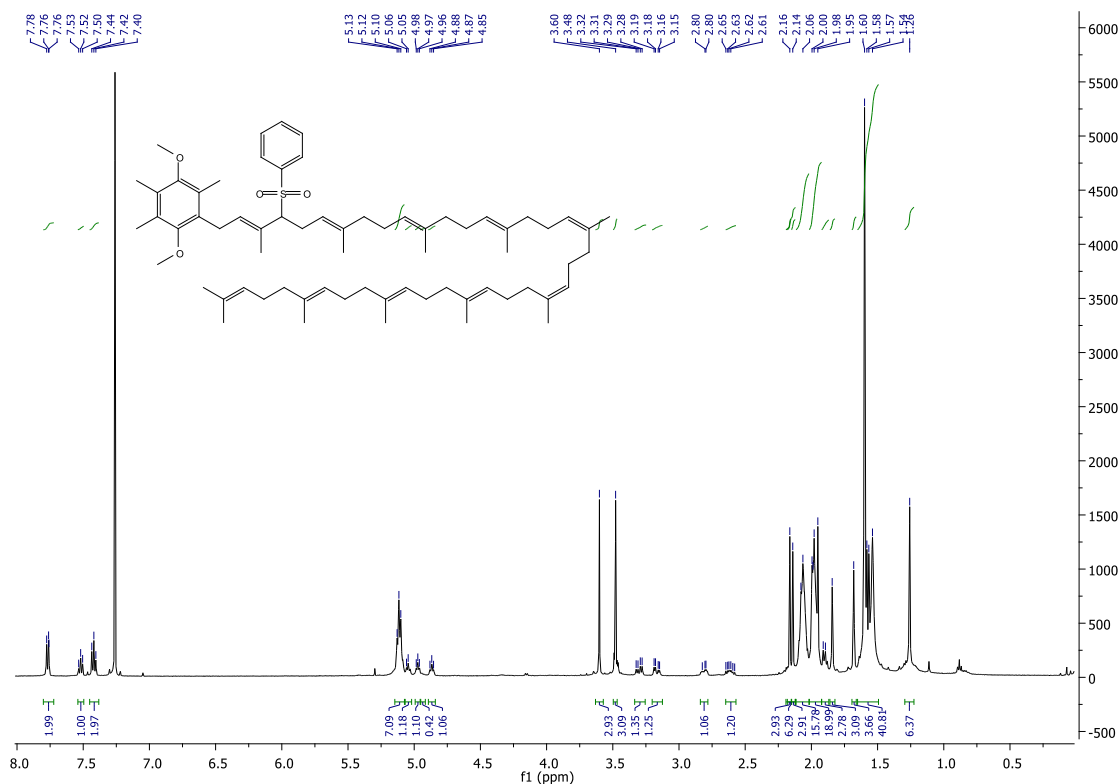
^{13}C NMR Spectrum (125 MHz, CDCl_3) of (*E*)-1,4-dimethoxy-2,3,5-trimethyl-6-(3-methyl-4-(phenylsulfonyl)but-2-enyl)benzene (**10**)



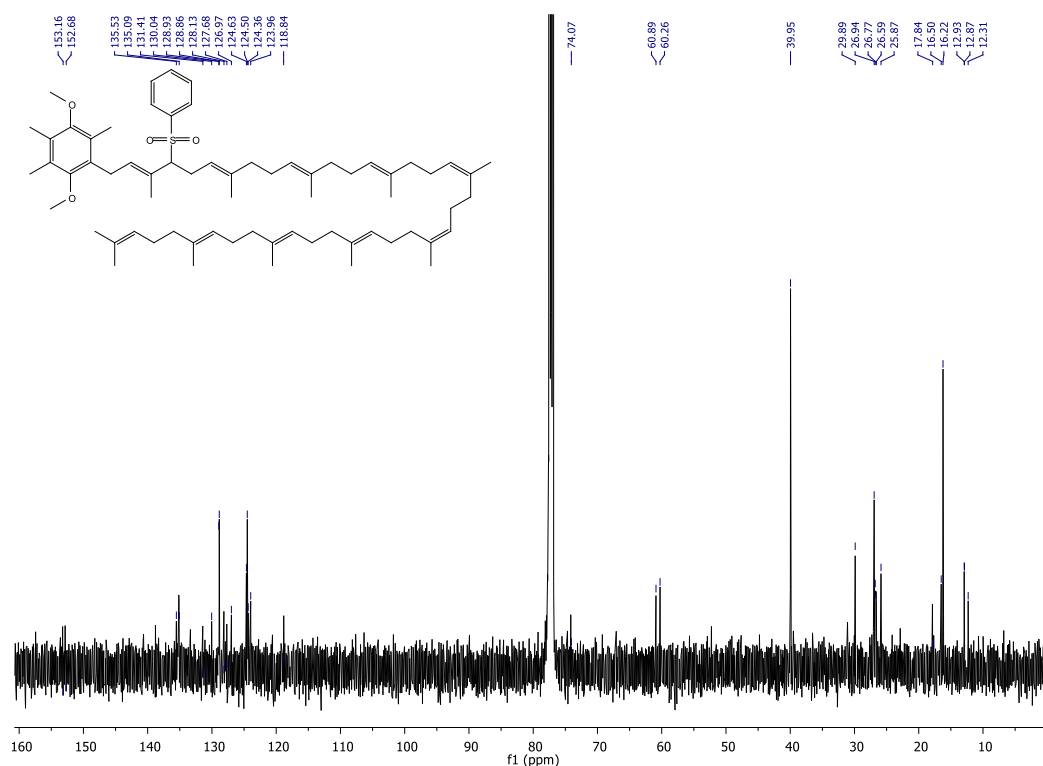
IR Spectrum of 1-((2*E*,6*E*,10*E*,14*E*,18*E*,22*E*,26*E*,30*E*,34*E*)-3,7,11,15,19,23,27,31,35,39-decamethyl-4-(phenylsulfonyl)tetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (**11**)



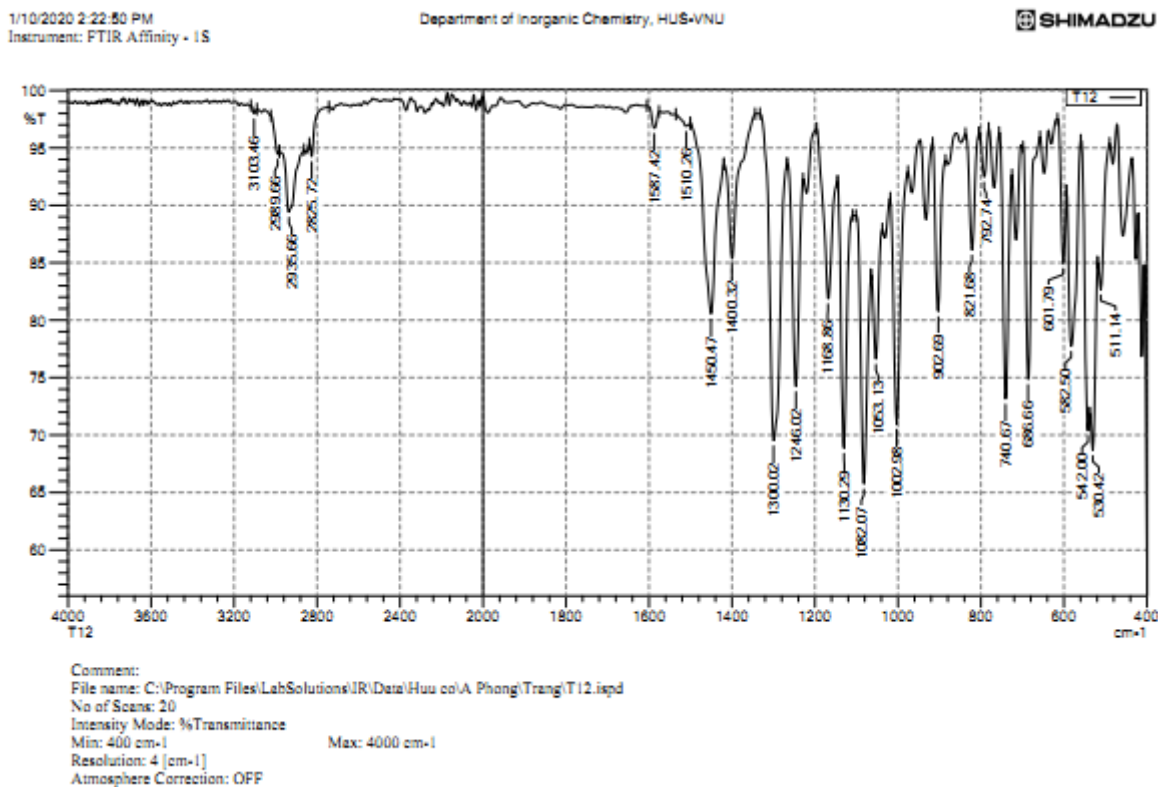
^1H NMR Spectrum (500 MHz, CDCl_3) of 1-((2*E*,6*E*,10*E*,14*E*,18*E*,22*E*,26*E*,30*E*,34*E*)-3,7,11,15,19,23,27,31,35,39-decamethyl-4-(phenylsulfonyl)tetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (**11**)



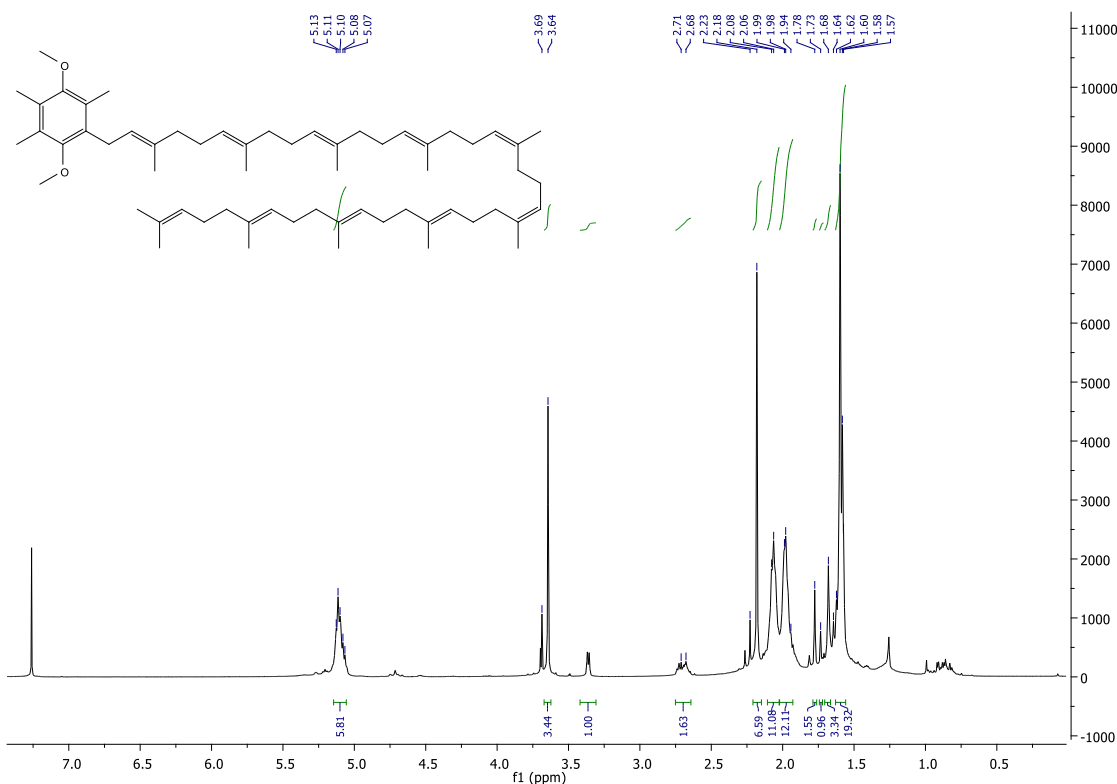
^{13}C NMR Spectrum (125 MHz, CDCl_3) of 1-((2*E*,6*E*,10*E*,14*E*,18*E*,22*E*,26*E*,30*E*,34*E*)-3,7,11,15,19,23,27,31,35,39-decamethyl-4-(phenylsulfonyl)tetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (**11**)



IR Spectrum of 1-((2*E*,6*E*,10*E*,14*E*,18*E*,22*E*,26*E*,30*E*,34*E*)-3,7,11,15,19,23,27,31,35,39-decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (**12**)



¹H NMR Spectrum (500 MHz, CDCl₃) of 1-((2*E*,6*E*,10*E*,14*E*,18*E*,22*E*,26*E*,30*E*,34*E*)-3,7,11,15,19,23,27,31,35,39-decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (**12**)



^{13}C NMR Spectrum (125 MHz, CDCl_3) of 1-((2E,6E,10E,14E,18E,22E,26E,30E,34E)-3,7,11,15,19,23,27,31,35,39-decamethyltetraconta-2,6,10,14,18,22,26,30,34,38-decaenyl)-2,5-dimethoxy-3,4,6-trimethylbenzene (**12**)

