

Supporting Information
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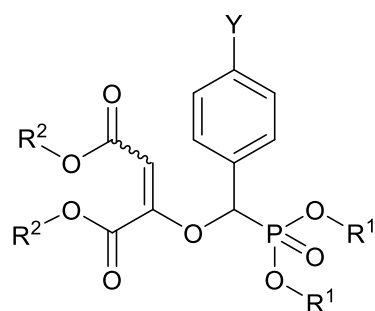
Supplementary Information

The Addition of α -Hydroxy-benzylphosphonates to Dialkyl Acetylenedicarboxylates; Catalytic Hydrogenation of the Adducts

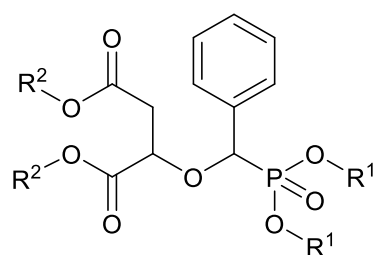
Mátyás Milen, Cintia Bese, Csenge Kovács, András Dancsó, György Keglevich*

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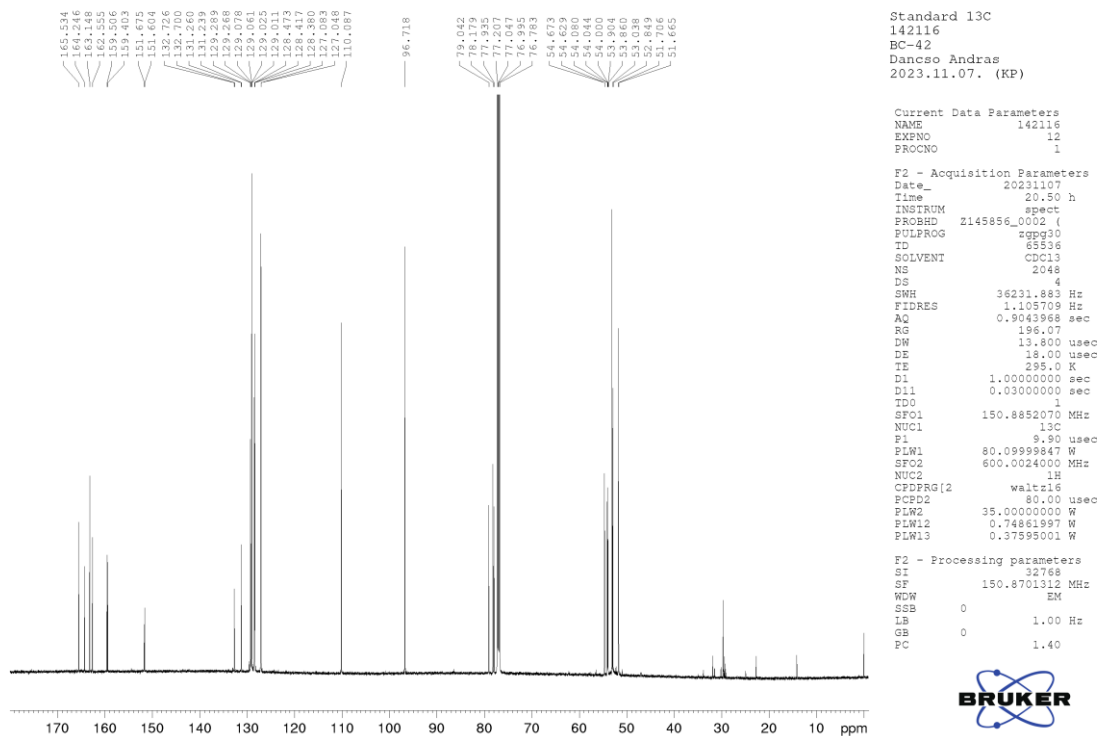
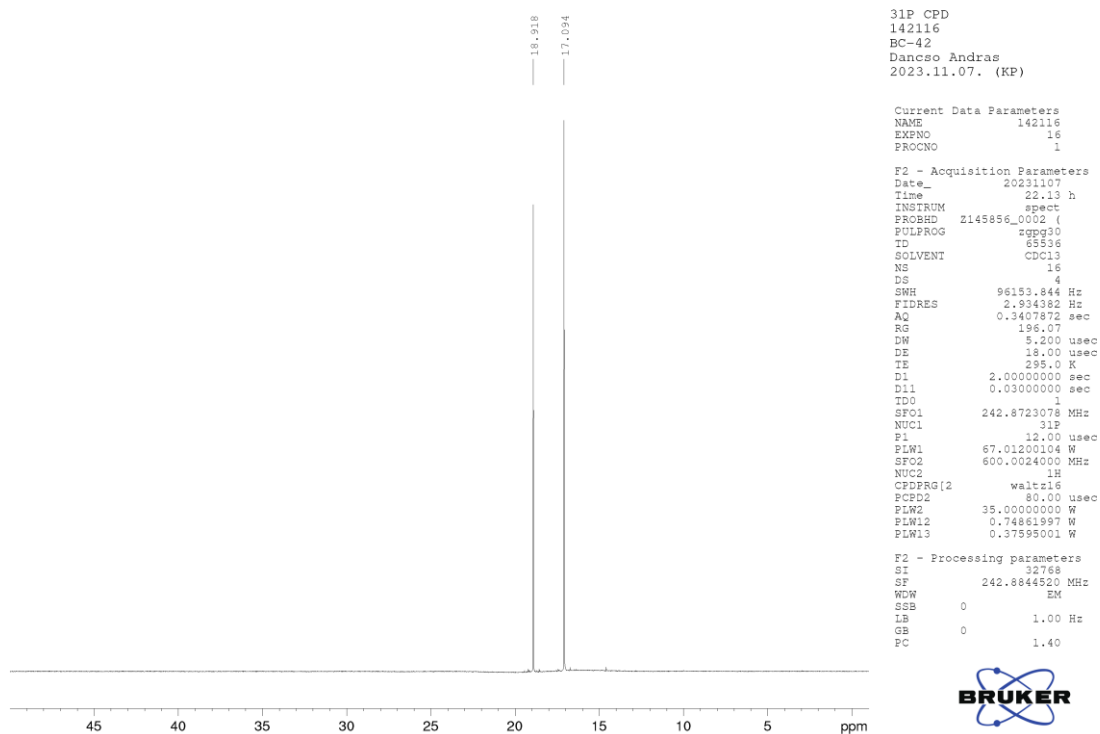
Compound	Y	R ¹	R ²
2Aa	H	Me	Me
2Ab	MeO	Me	Me
2Ac	Me	Me	Me
2Ad	Cl	Me	Me
2Ae	H	Et	Me
2Af	MeO	Et	Me
2Ag	Me	Et	Me
2Ba	H	Me	Et
2Bb	MeO	Me	Et
2Bc	Me	Me	Et
2Bd	Cl	Me	Et
2Be	H	Et	Et

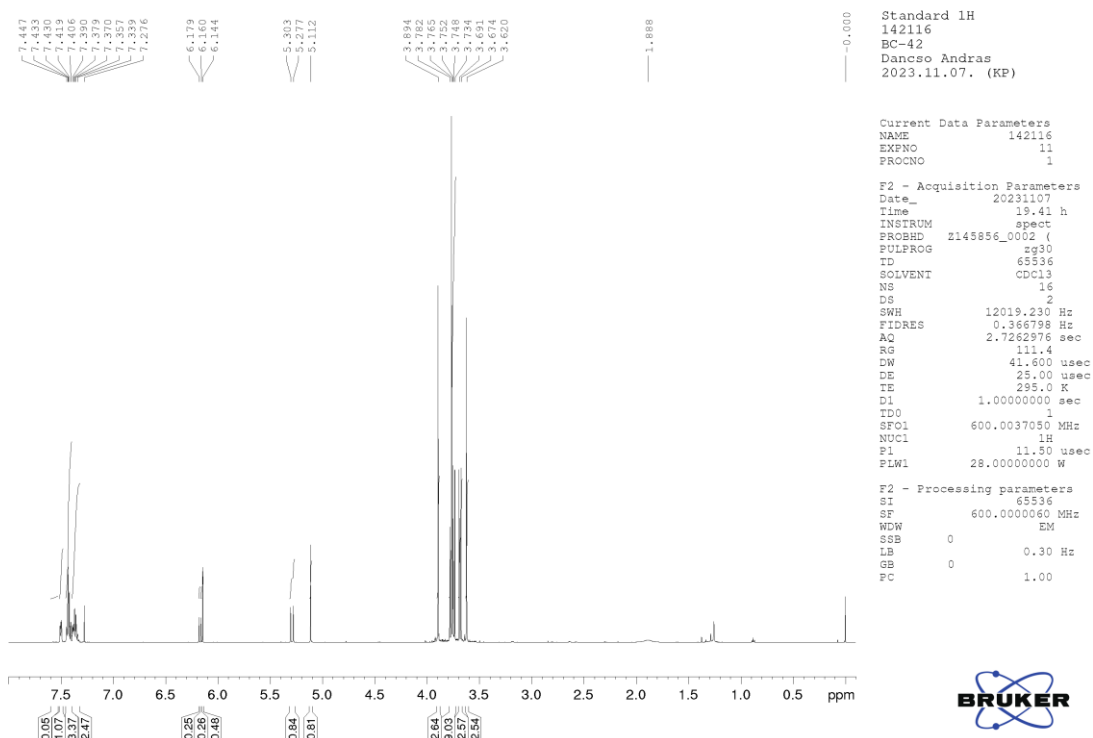


Compound	R ¹	R ²
3Aa	Me	Me
3Ae	Et	Me
3Ba	Me	Et
3Be	Et	Et

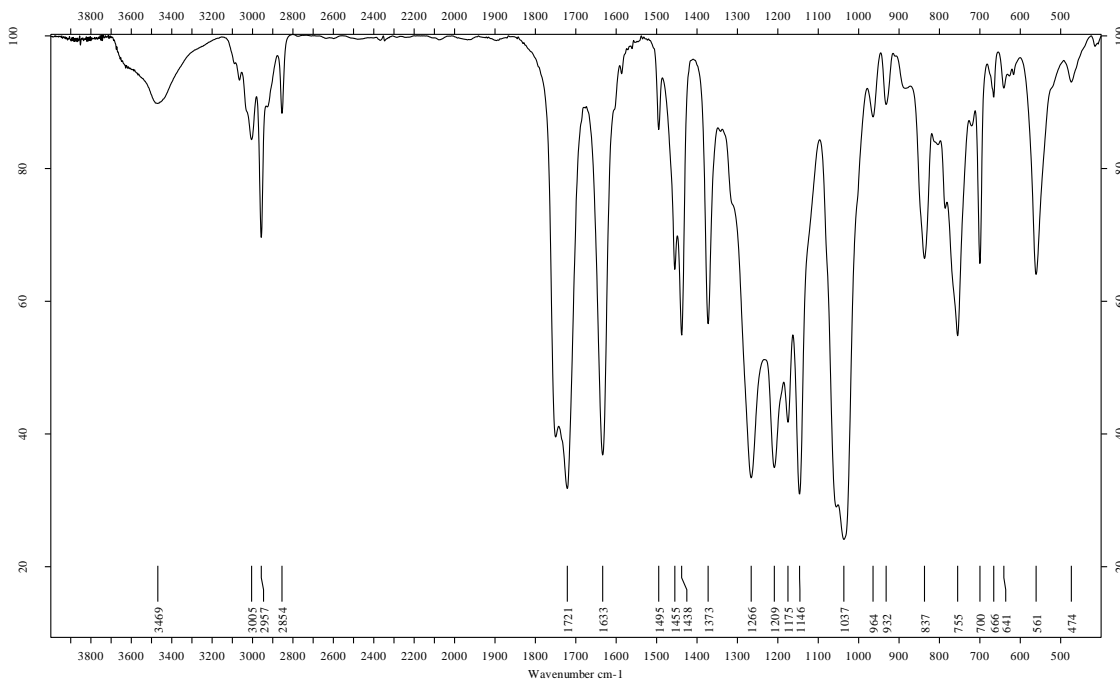
1.) α -Hydroxyphosphonate – dialkyl acetylenedicarboxylate adducts

Dimethyl (1,2-Di(methoxycarbonyl)ethenyloxy)-benzylphosphonate (2Aa)

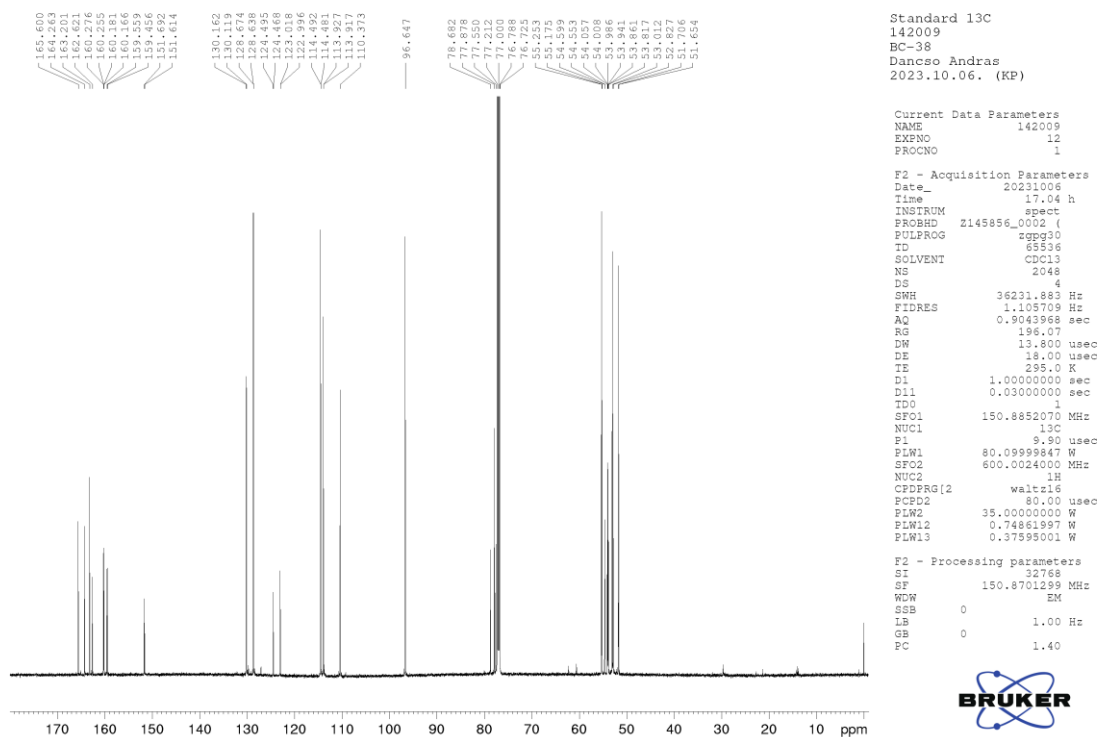
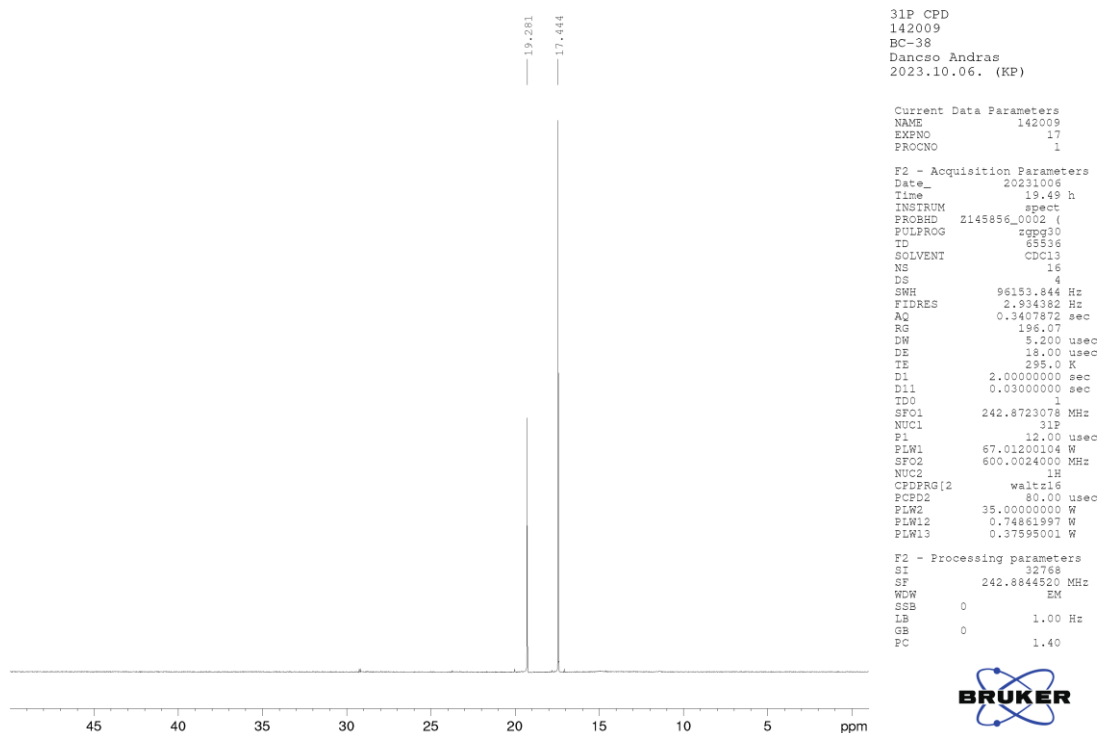


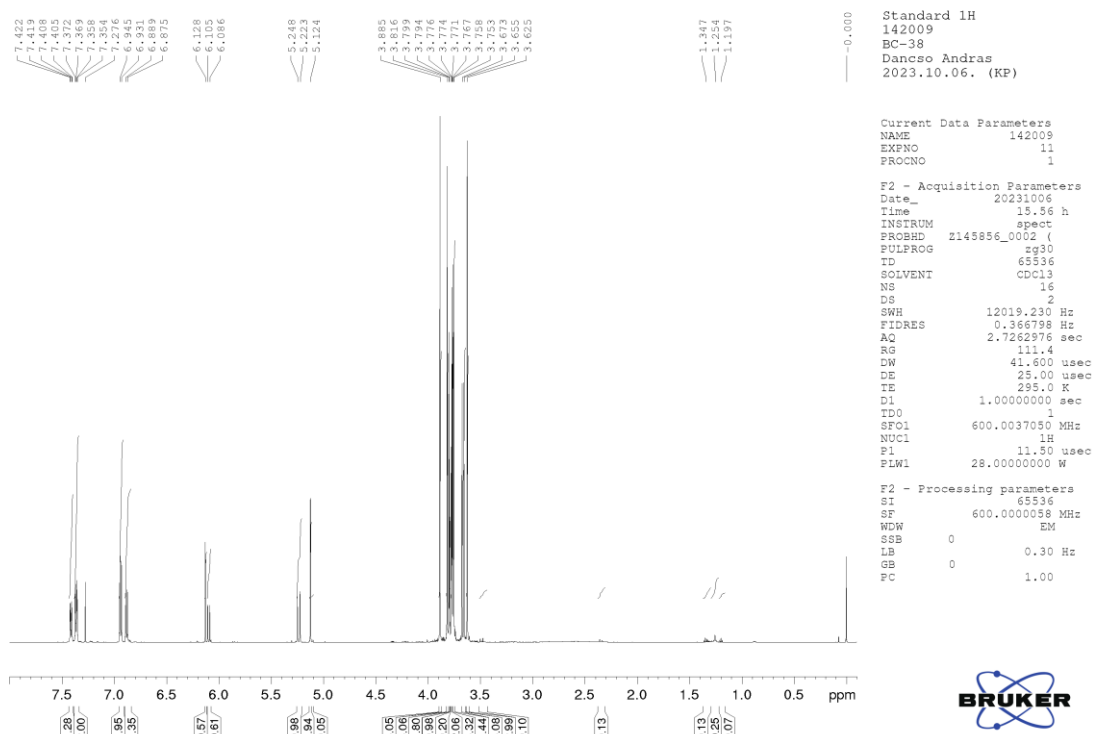


142116	Bese Cintia	DA	BRUKER Alpha
BC-42	film	5/9/2024	Resolution: 2 cm-1
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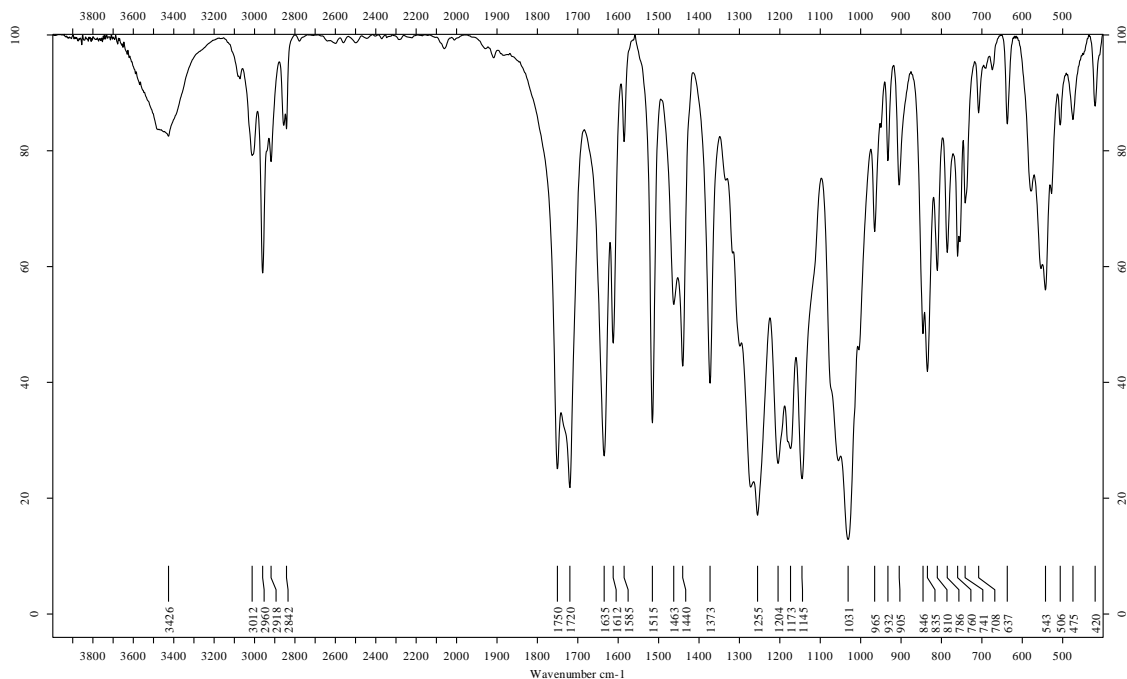


Dimethyl (1,2-Di(methoxycarbonyl)ethenyloxy)-4-methoxybenzylphosphonate (2Ab)

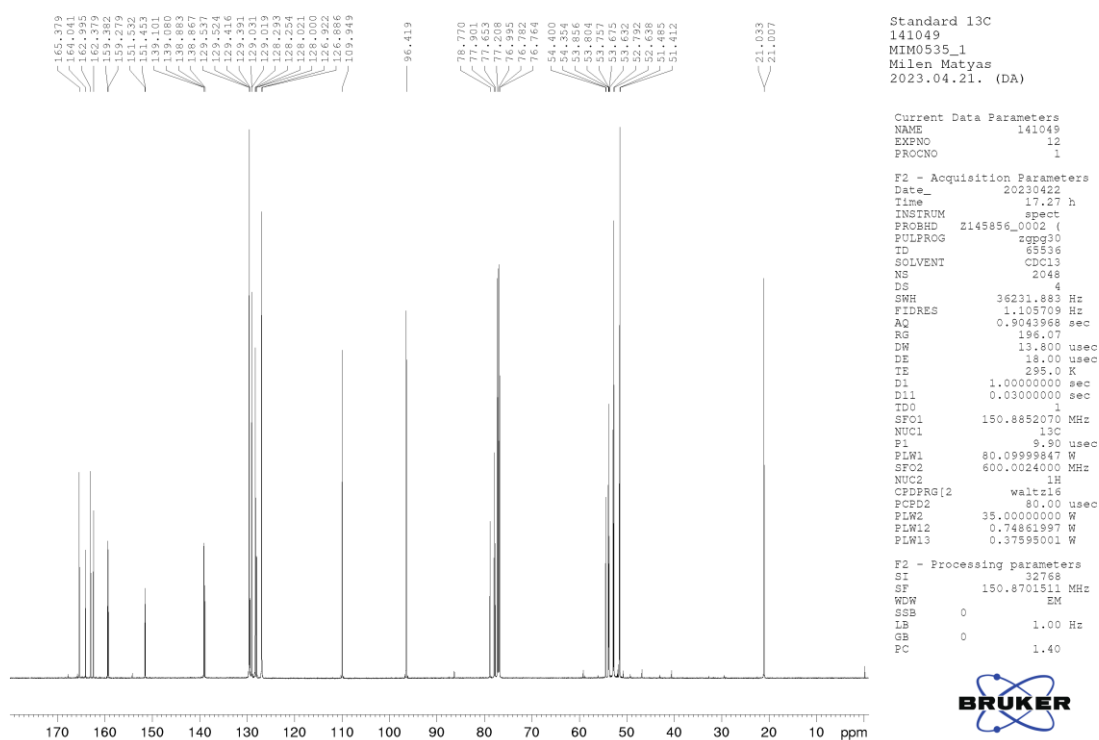
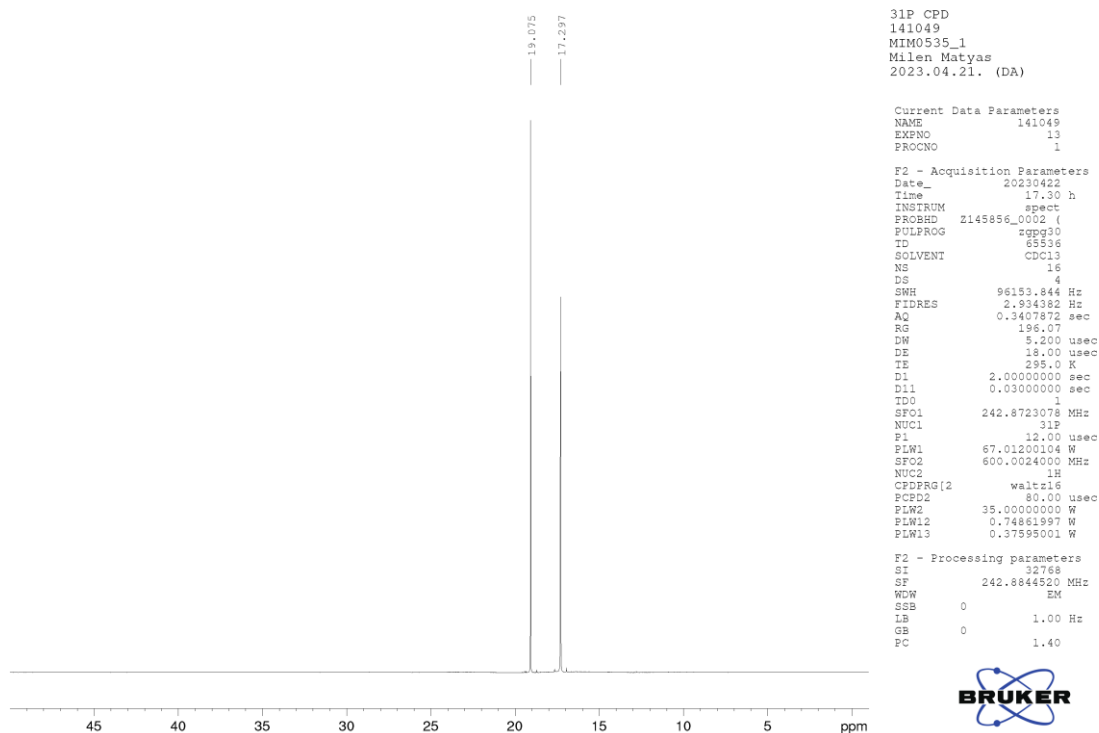


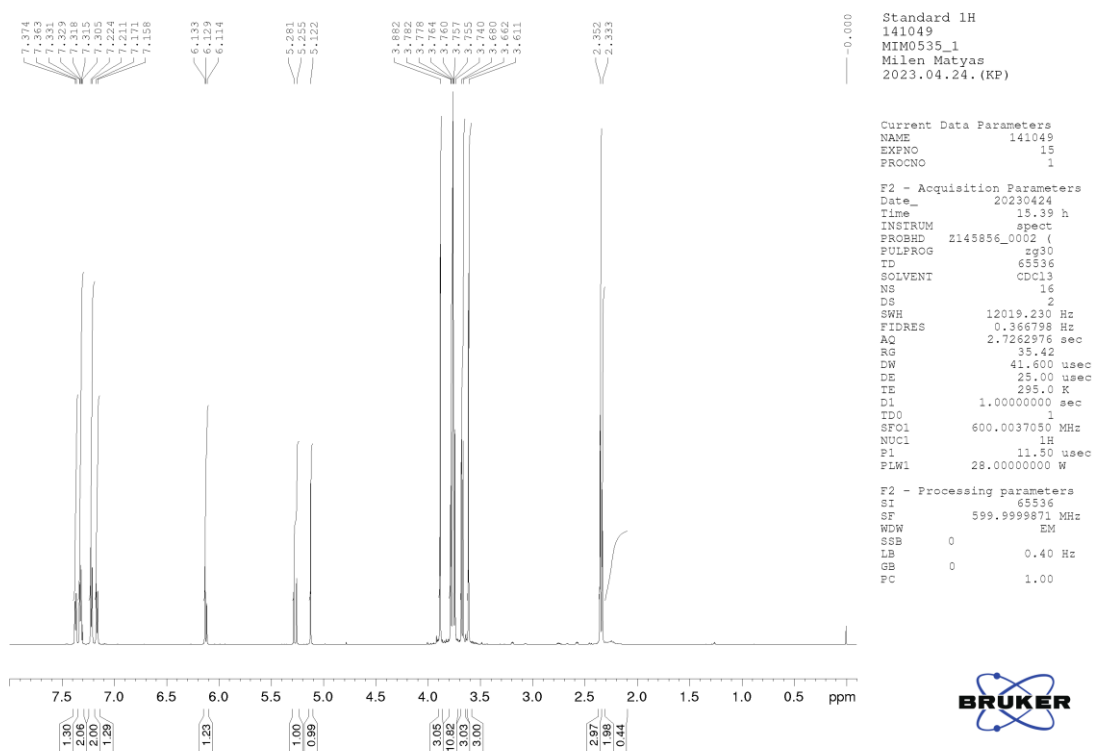


142009	Bese Cintia	DA	BRUKER Alpha
BC-38	KBr	5/21/2024	
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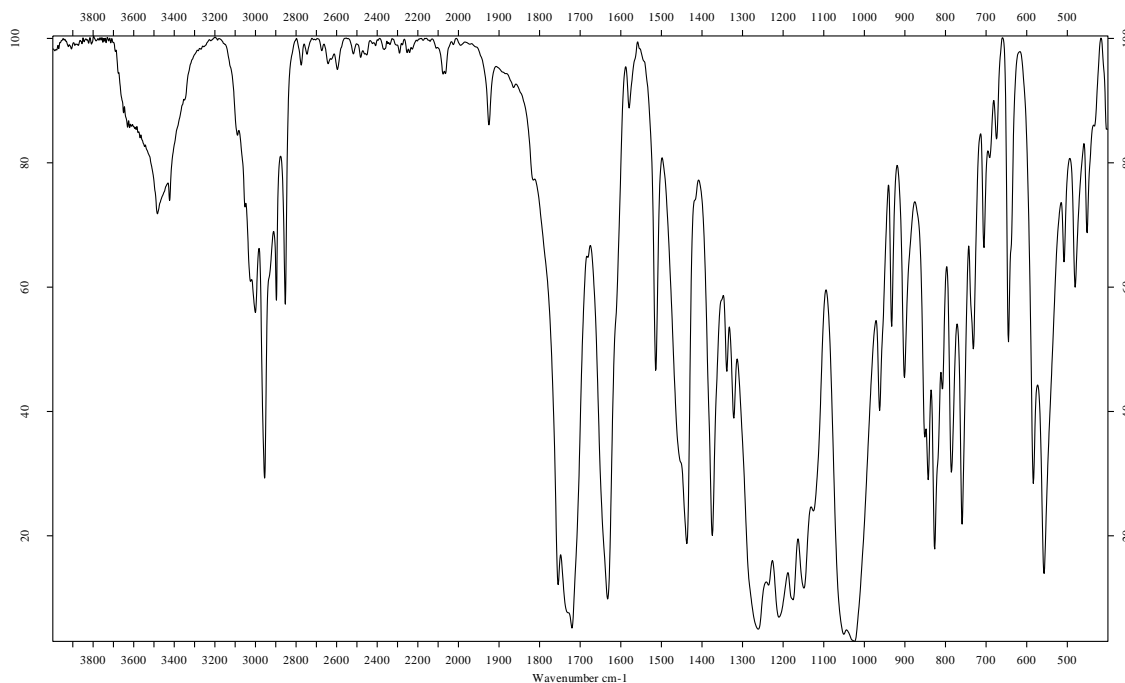


Dimethyl (1,2-Di(methoxycarbonyl)ethenyloxy)-4-methylbenzylphosphonate (2Ac)

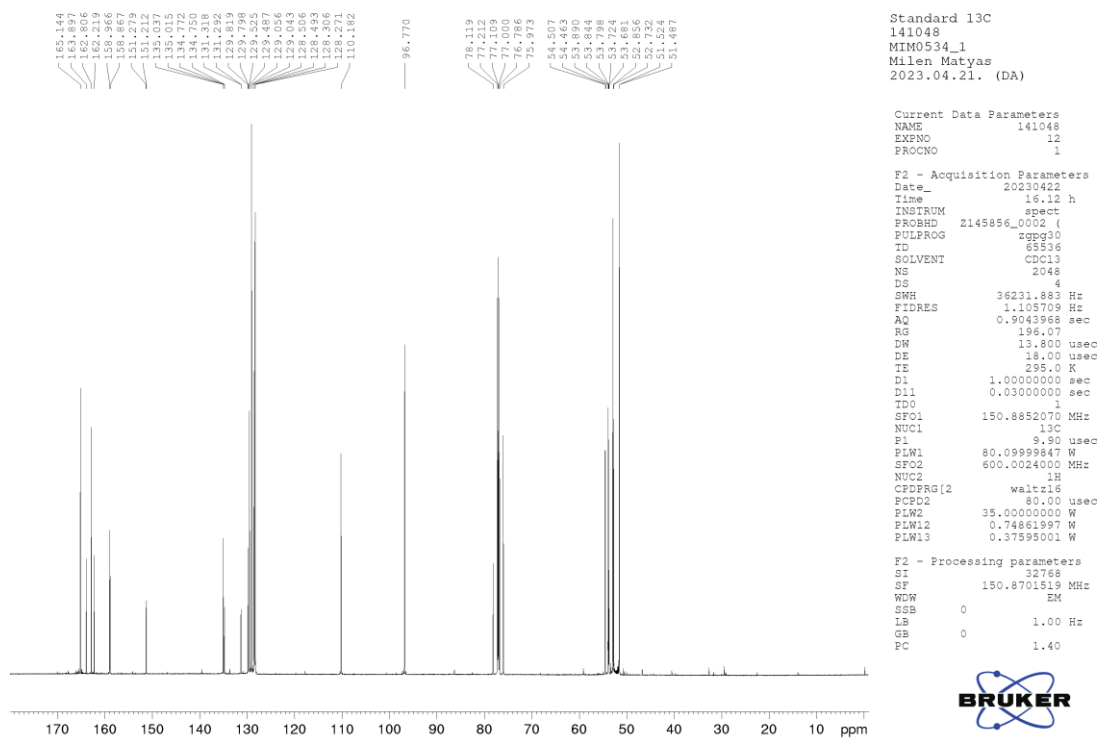


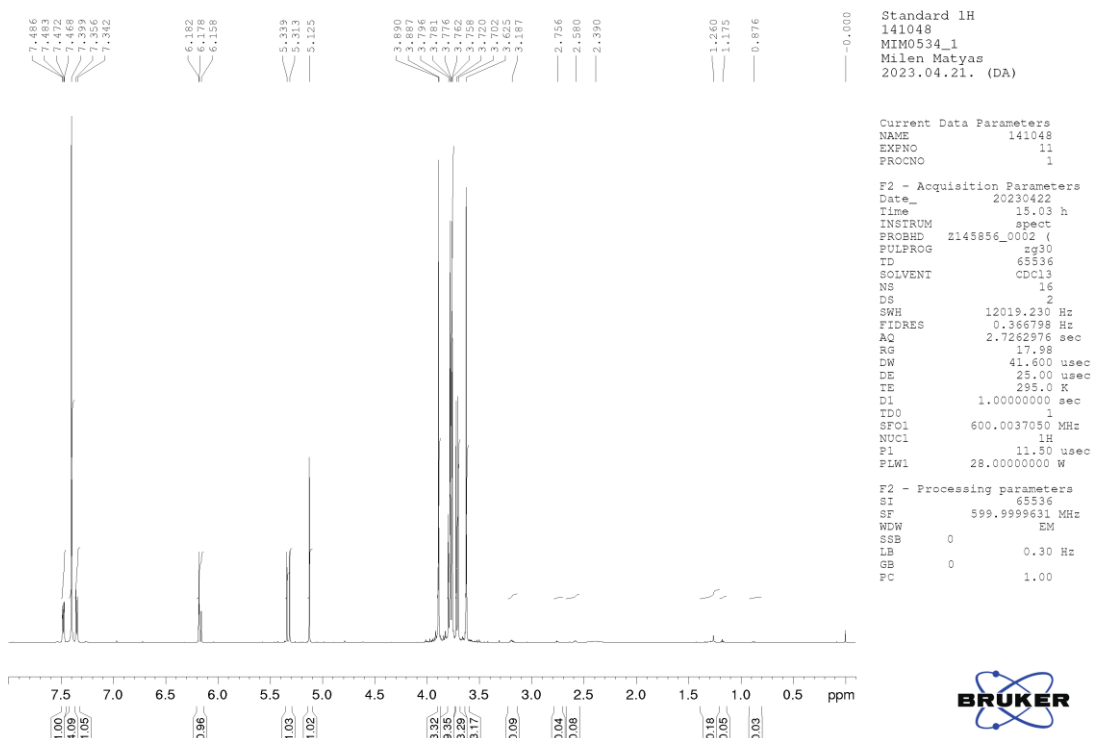


141049	Milen Matyas	KP	BRUKER Alpha
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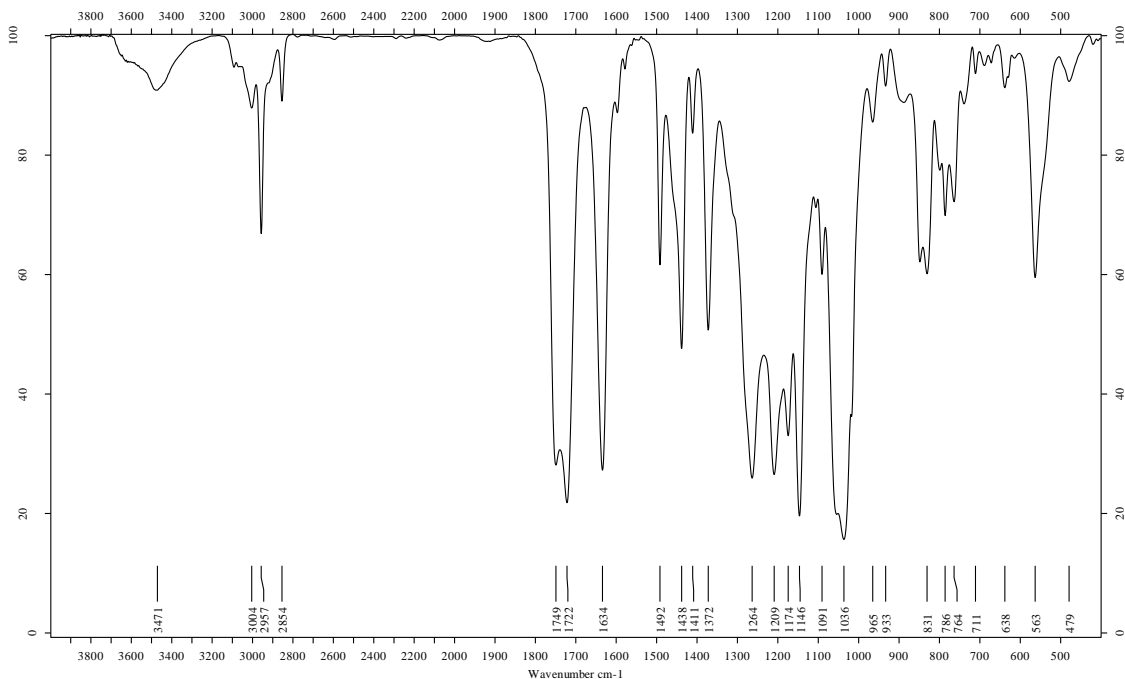


Dimethyl (1,2-Di(methoxycarbonyl)ethenyloxy)-4-chlorobenzylphosphonate (2Ad)

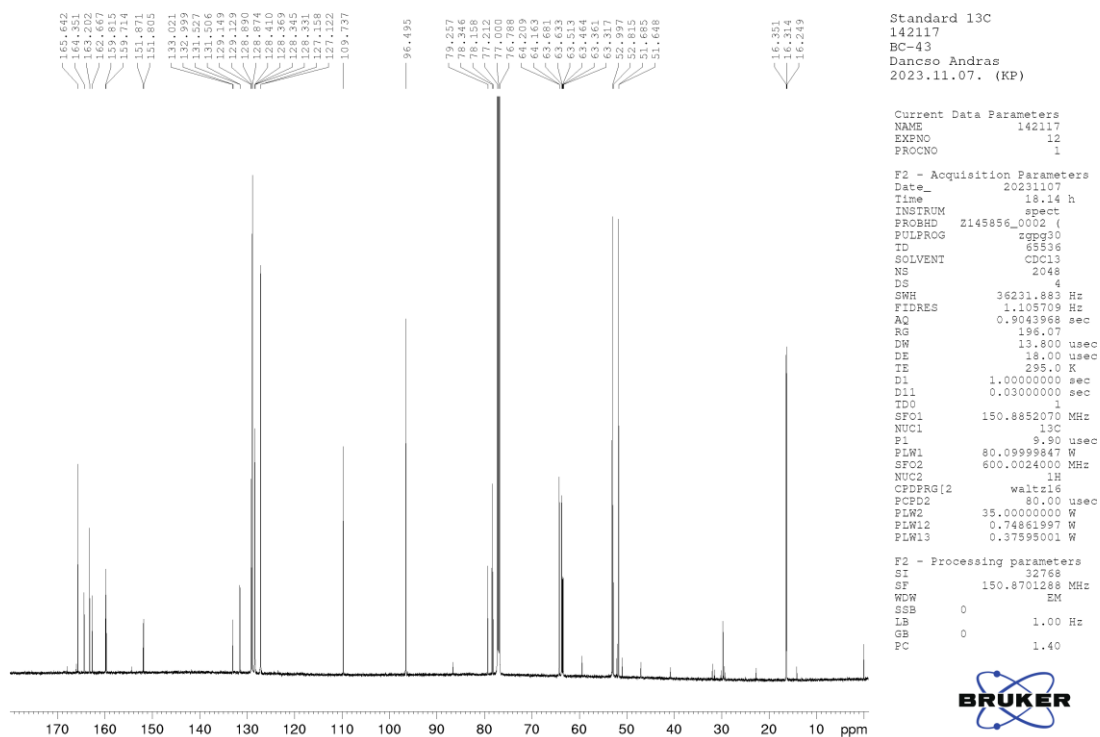
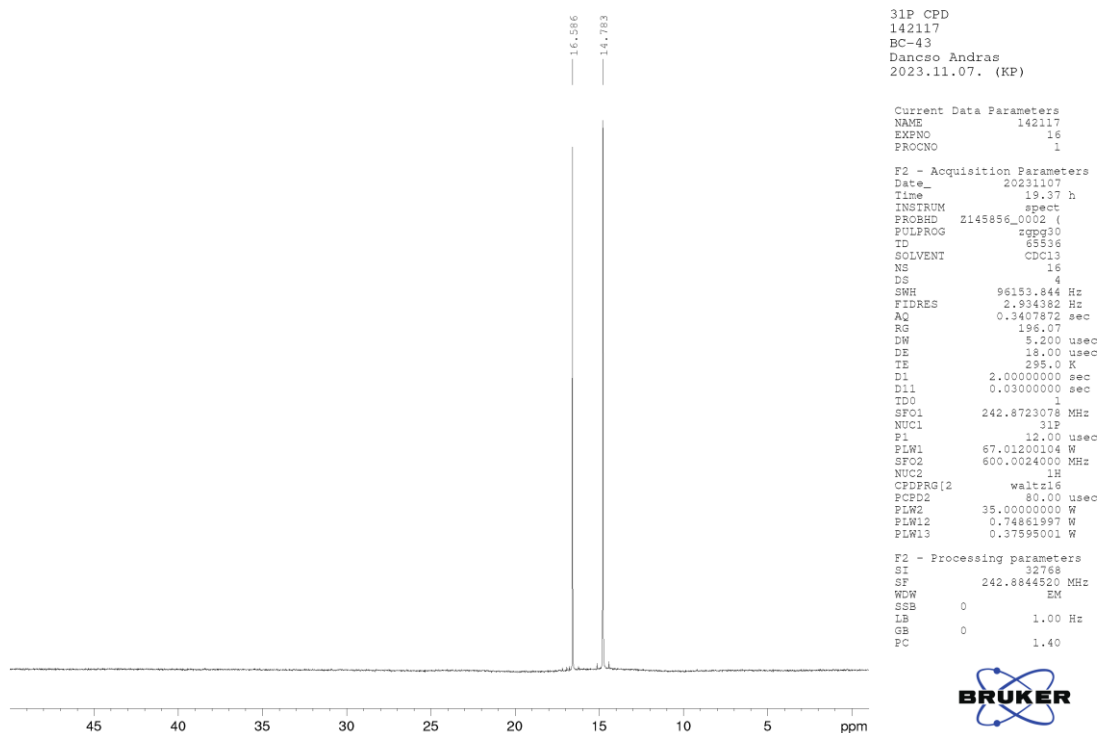


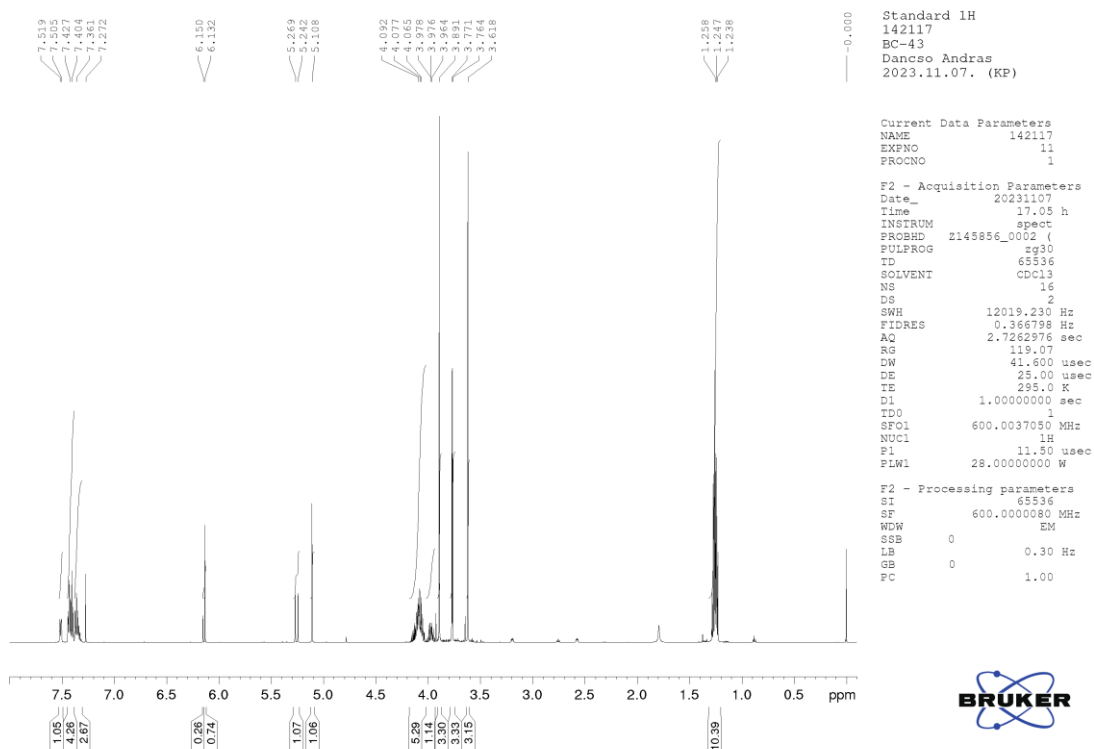


141048	Milen Matyas	KP	BRUKER Alpha
MIM0534_1	Film	5/8/2024	
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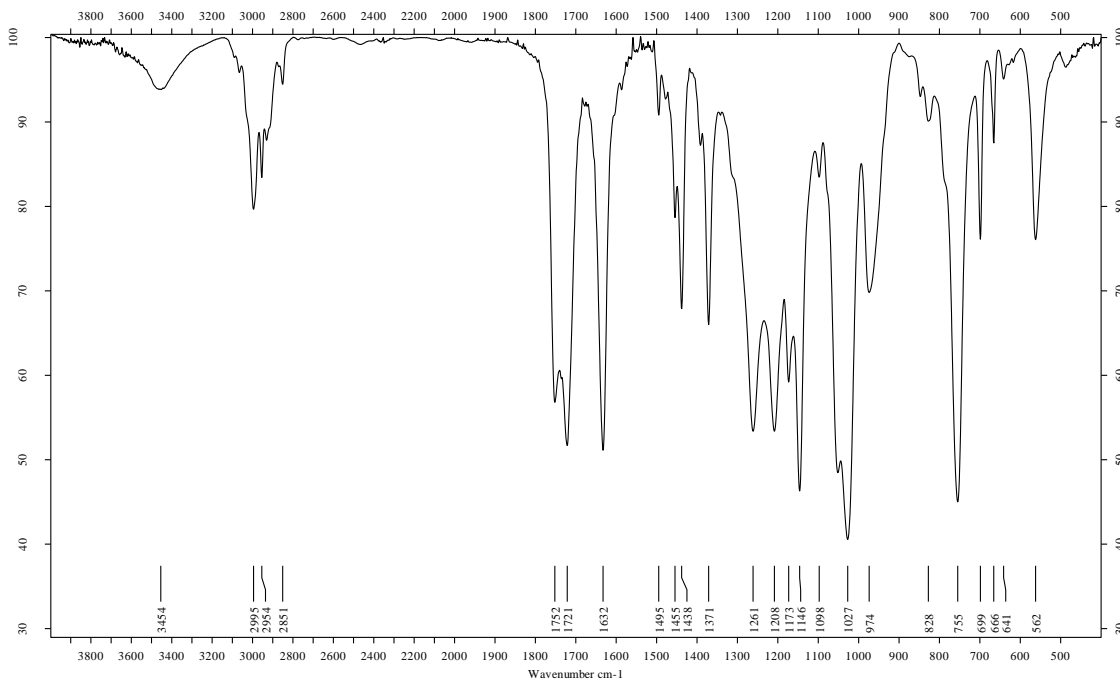




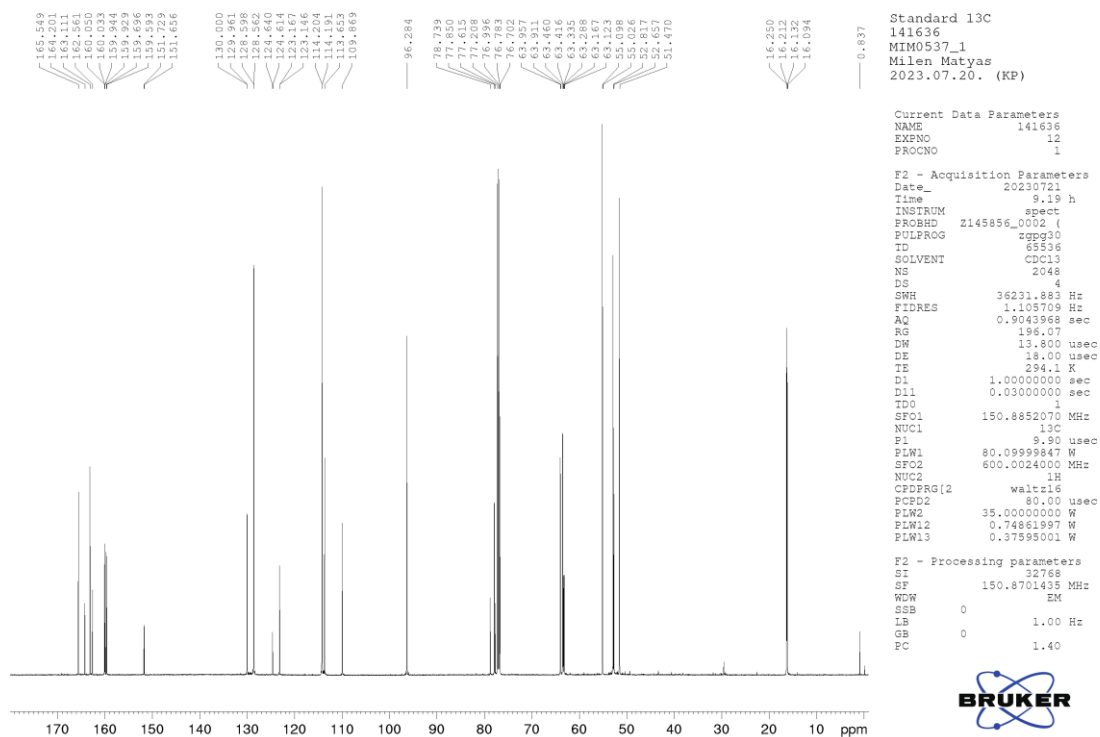
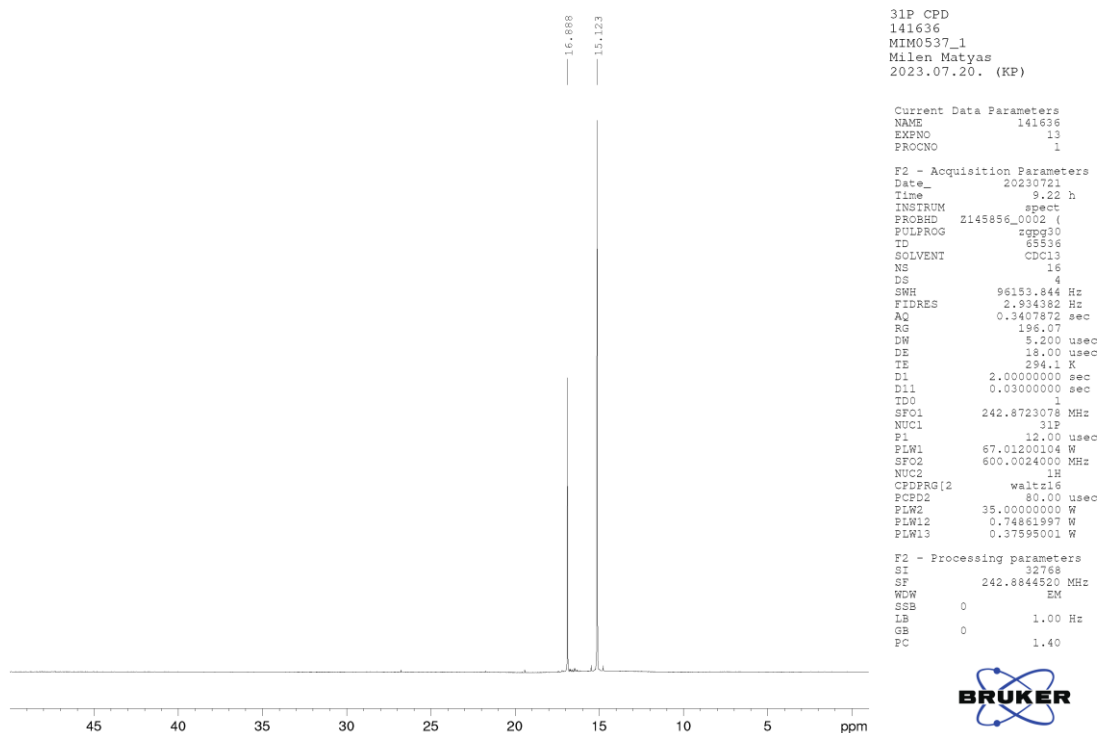
142117
BC-43

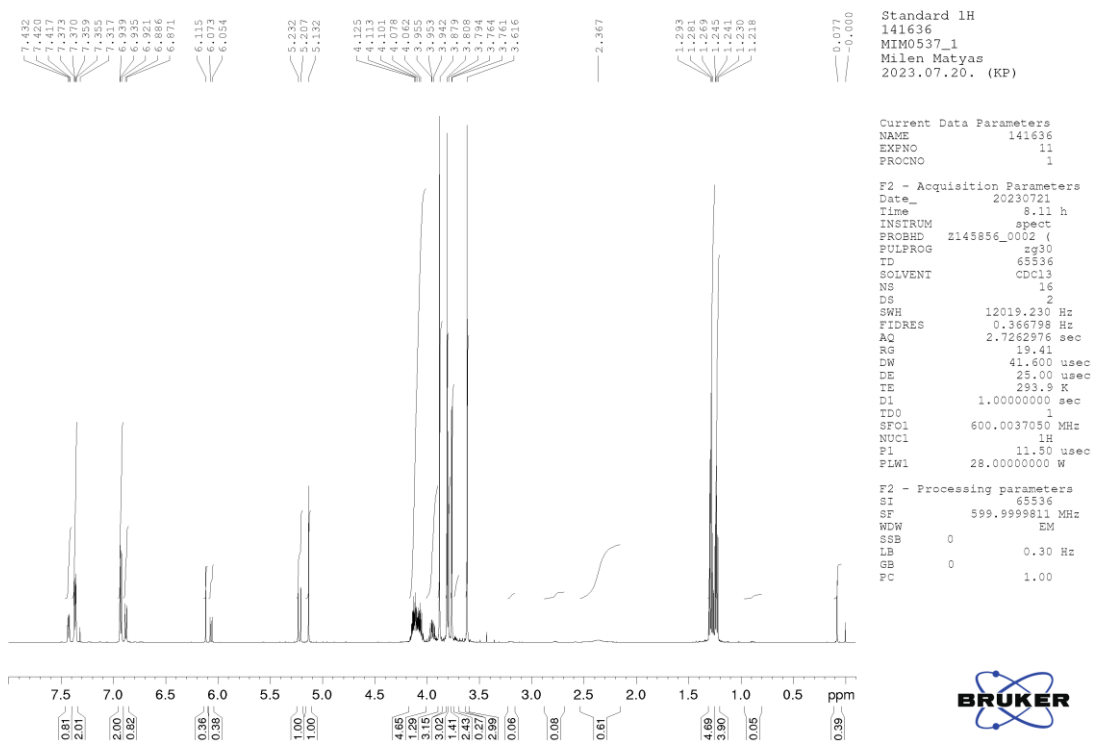
Bese Cintia	DA
film	5/9/2024

BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16

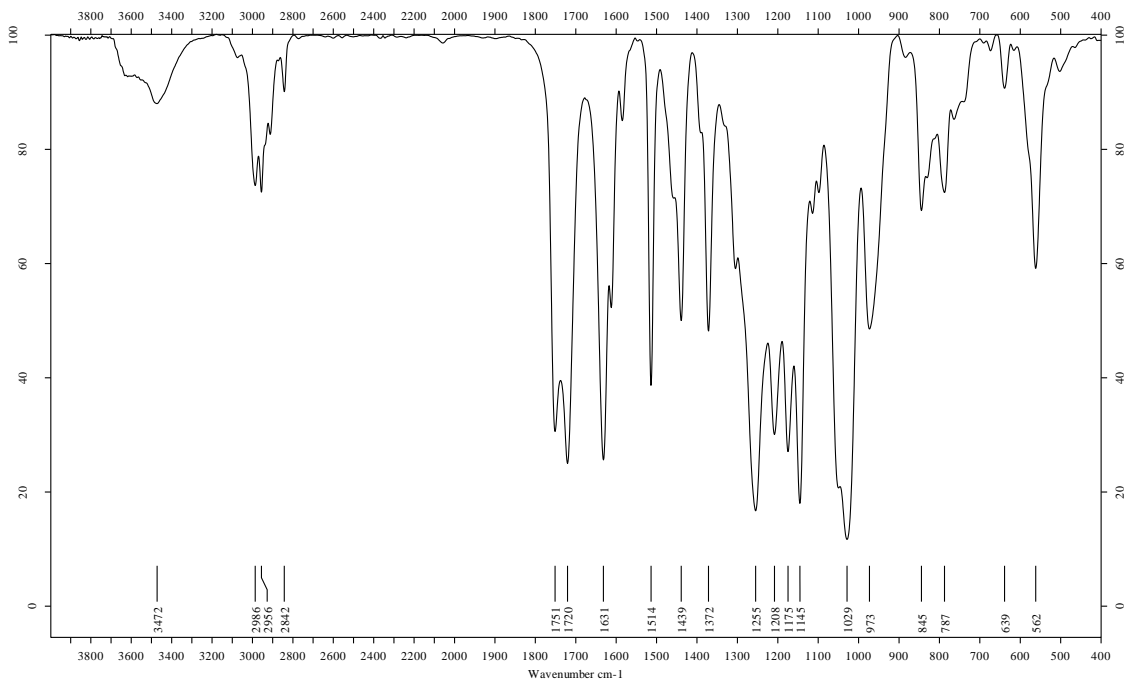


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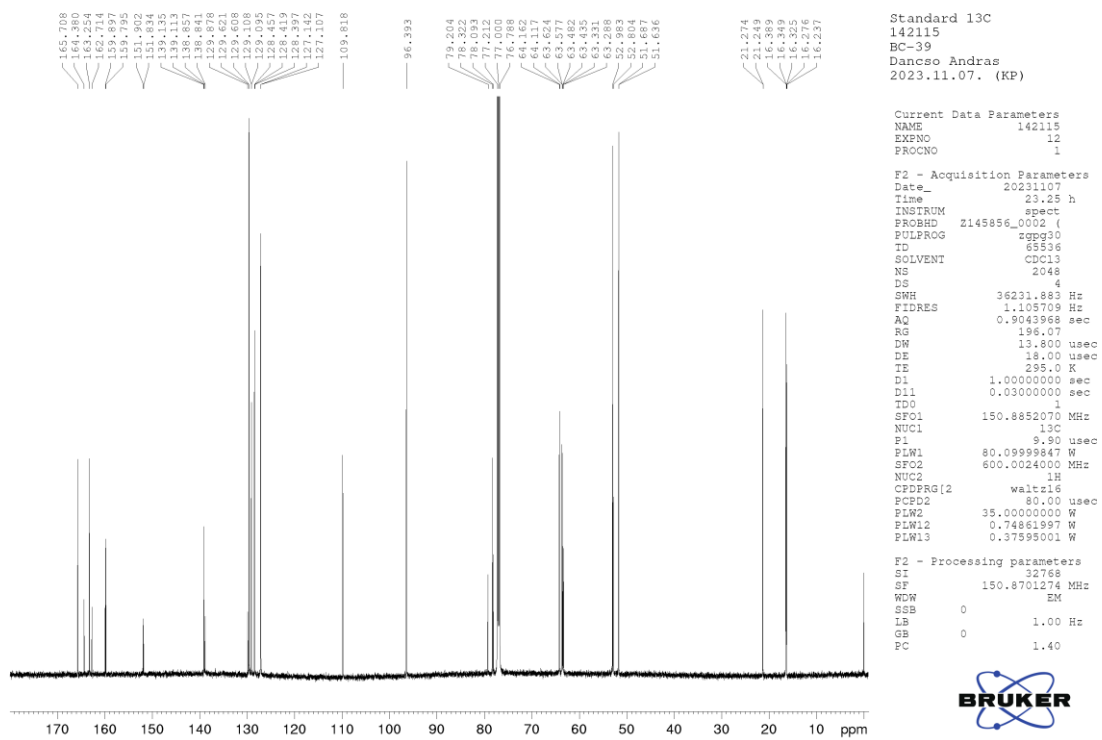
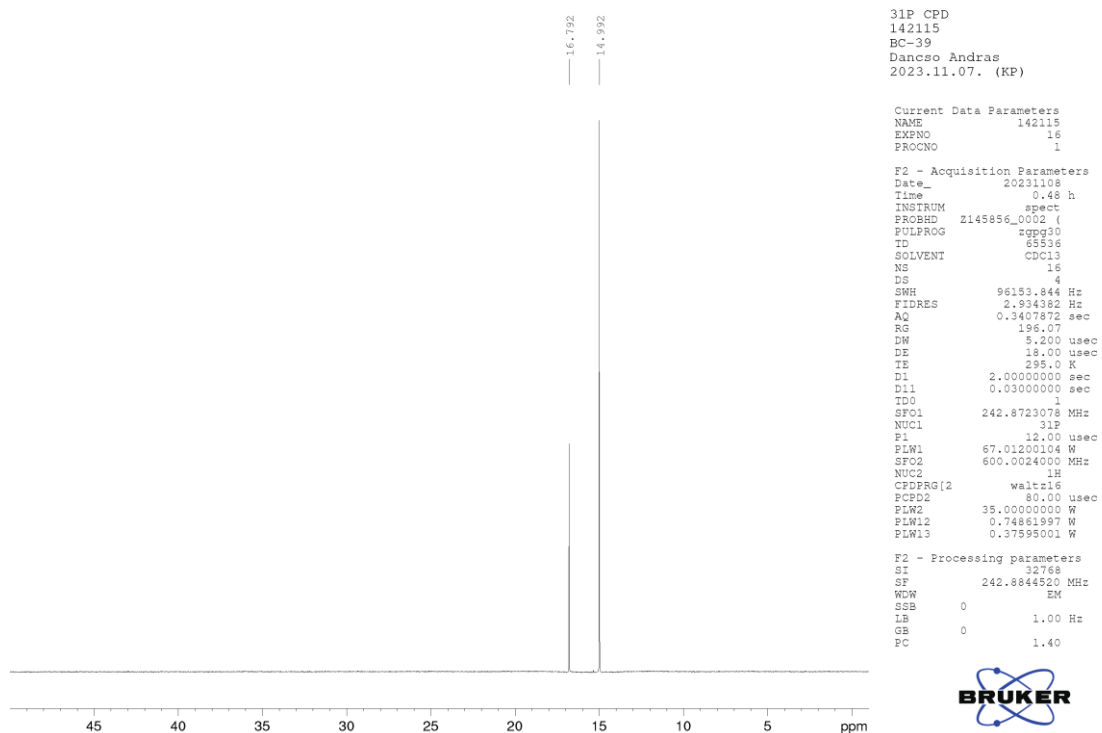


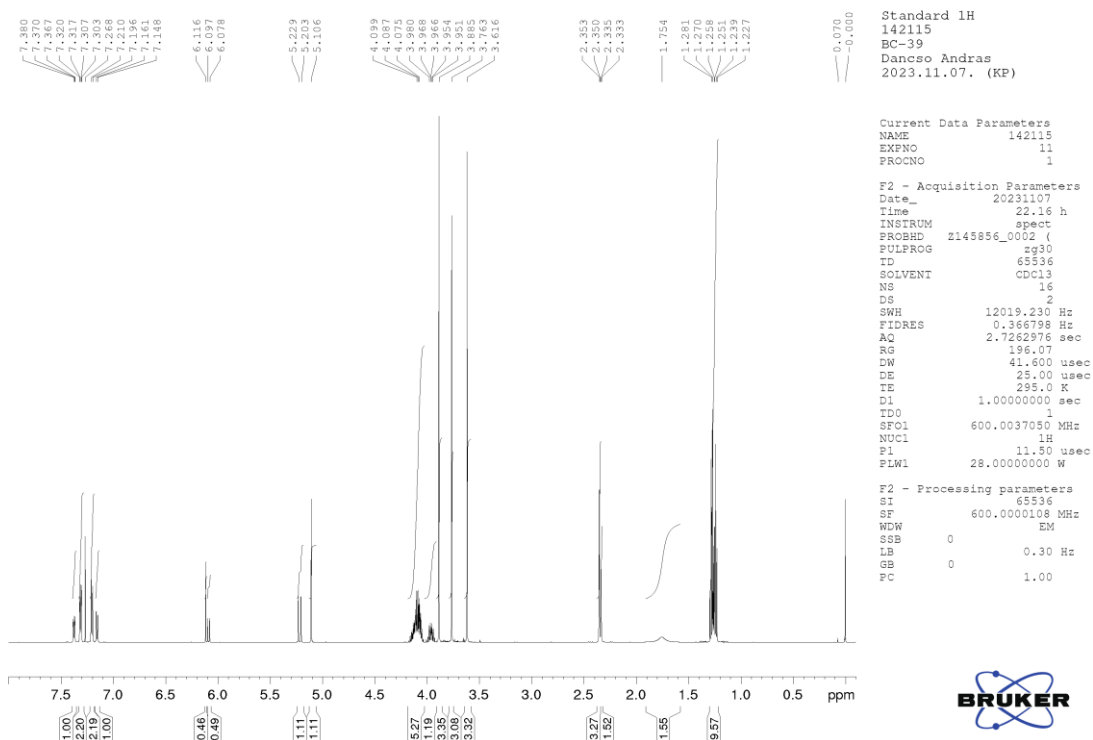


141636	Milen Matyas	KP	BRUKER Alpha
MIM0537_1	Film	7/21/2023	
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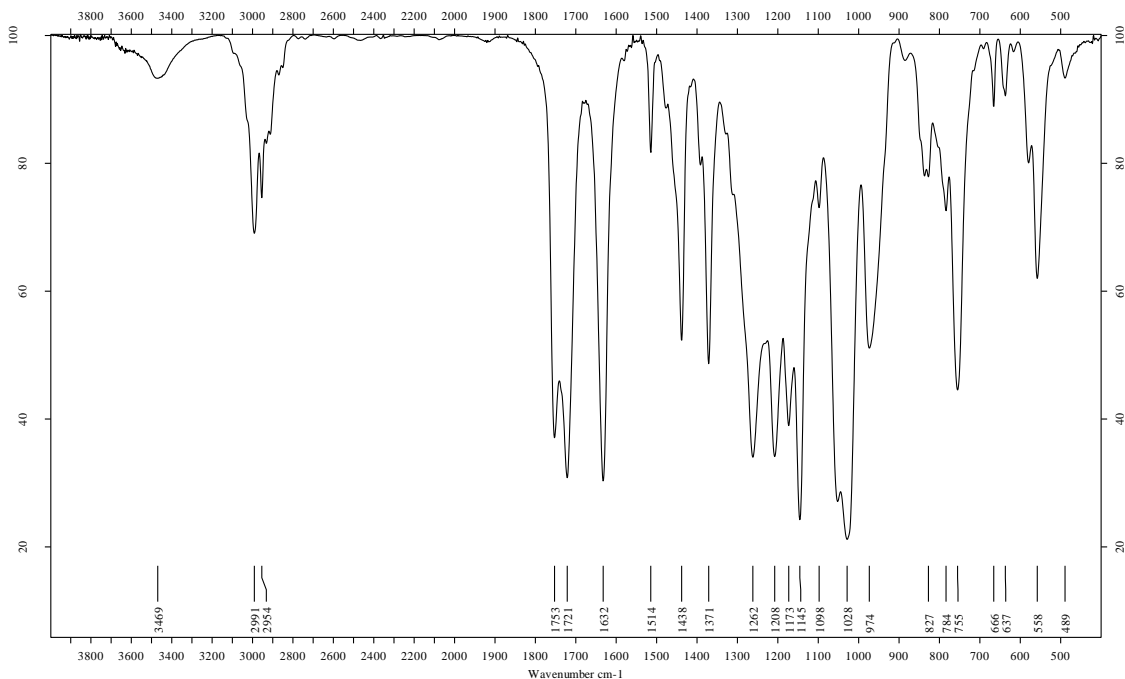


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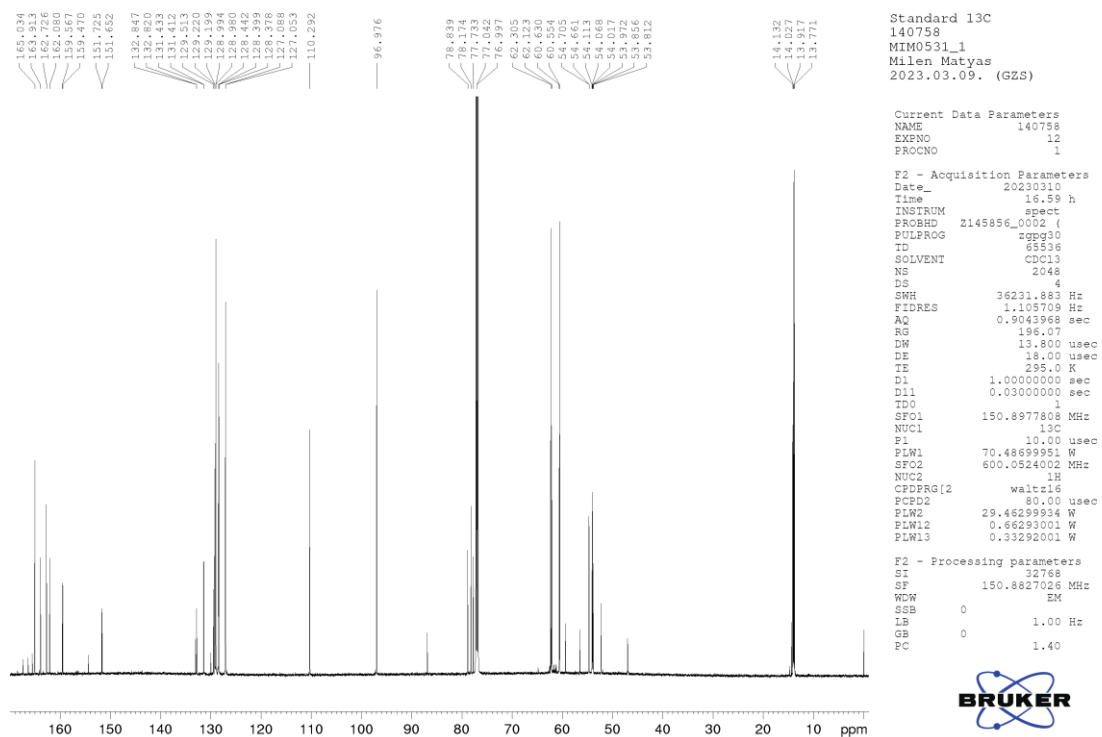
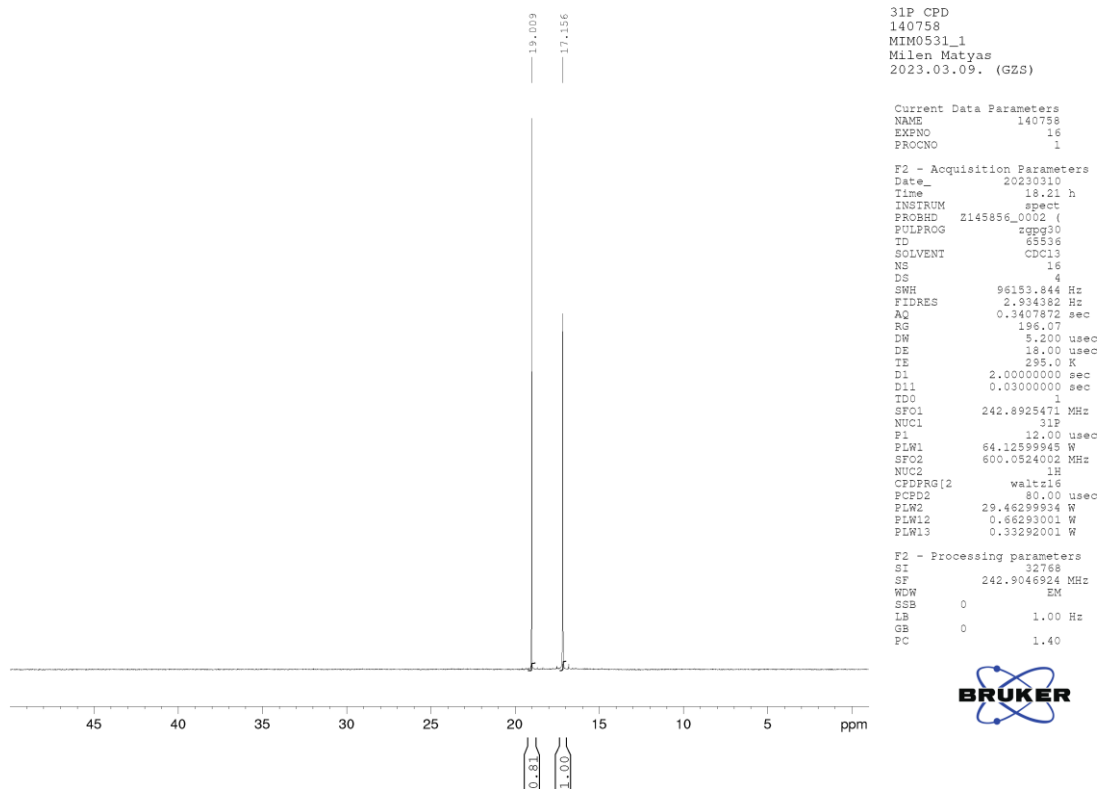


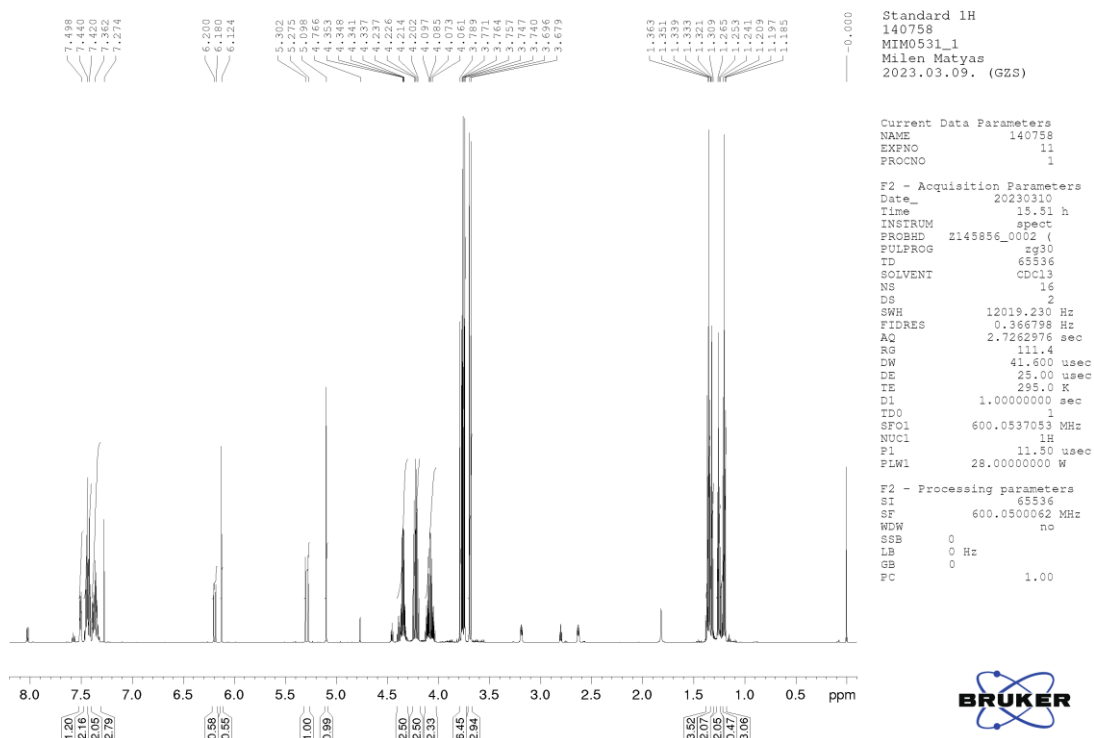


142115	Bese Cintia	DA	BRUKER Alpha
BC-39	film	5/9/2024	
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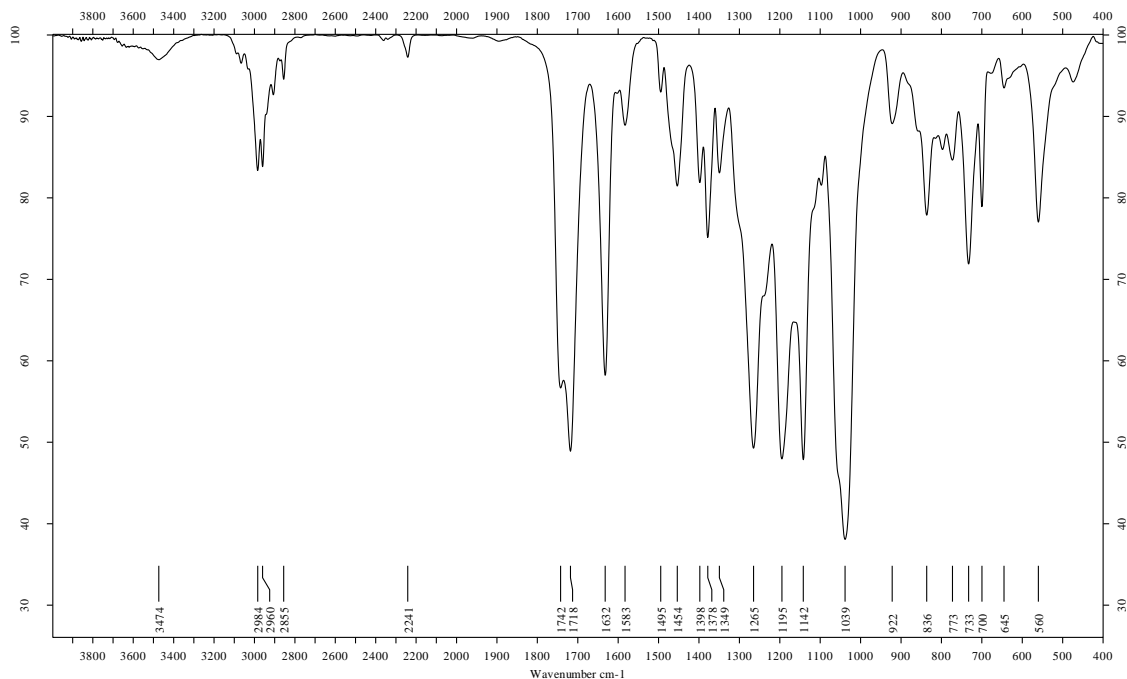


Dimethyl (1,2-Di(ethoxycarbonyl)ethenyloxy)-benzylphosphonate (2Ba)

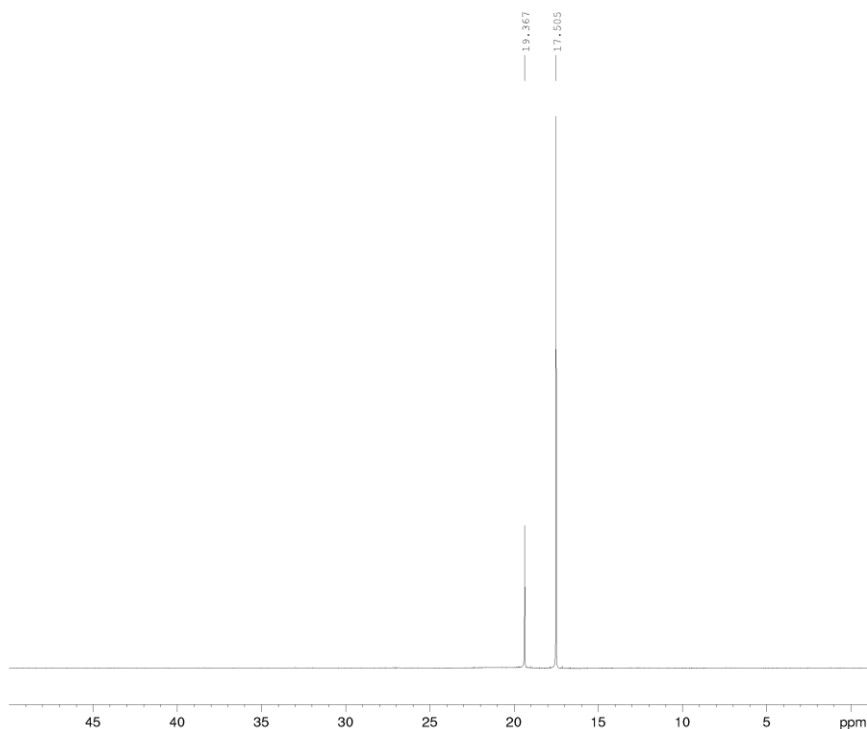




140758	Milen Matyas	KP	BRUKER Alpha
MIM0531_1	Film	3/23/2023	
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Dimethyl (1,2-Di(ethoxycarbony)ethenyloxy)-4-methoxybenzylphosphonate (2Bb)

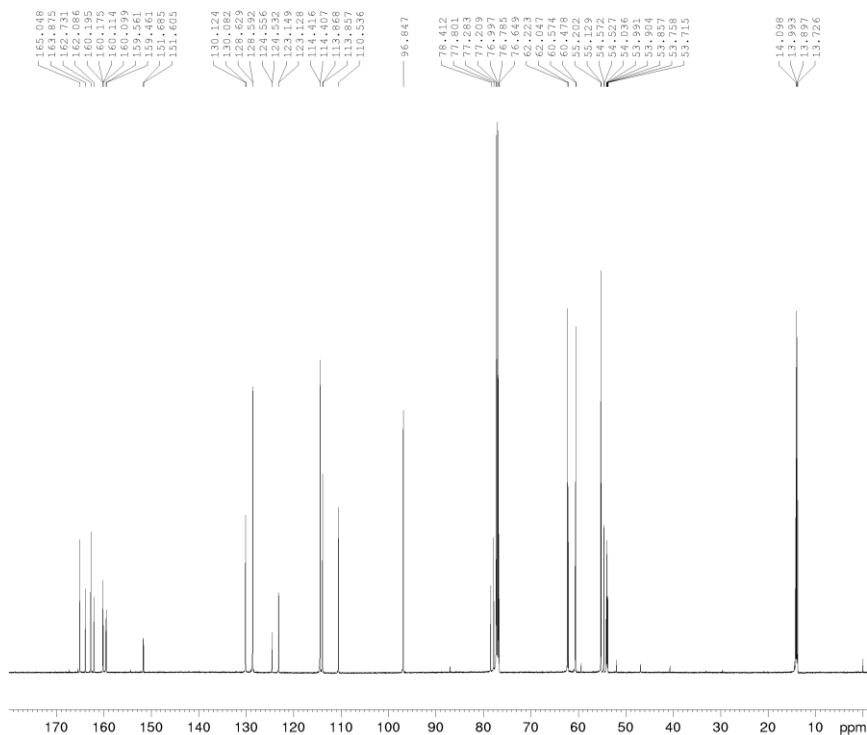


31P CPD
141041
MIM0533_1
Milen Matyas
2023.04.21. (DA)

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SOLVENT CDC13
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FIDRES 2.934382 Hz
AQ 0.3407872 sec
RG 196.07
DW 5.200 usec
DE 18.00 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 242.8723078 MHz
NUC1 31P
P1 12.00 usec
PLW1 67.01200104 W
SFO2 600.0024000 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
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PLW12 0.74861997 W
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F2 - Processing parameters
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WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



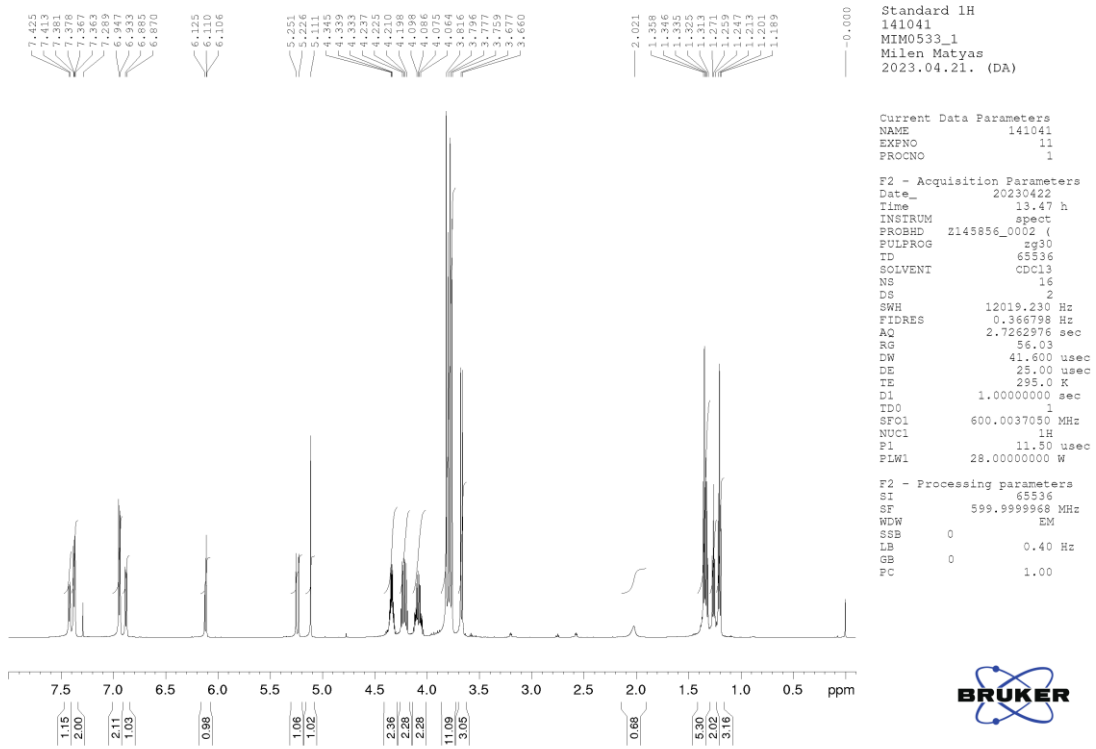
Standard 13C
141041
MIM0533_1
Milen Matyas
2023.04.21. (DA)

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EXPNO 12
PROCNO 1

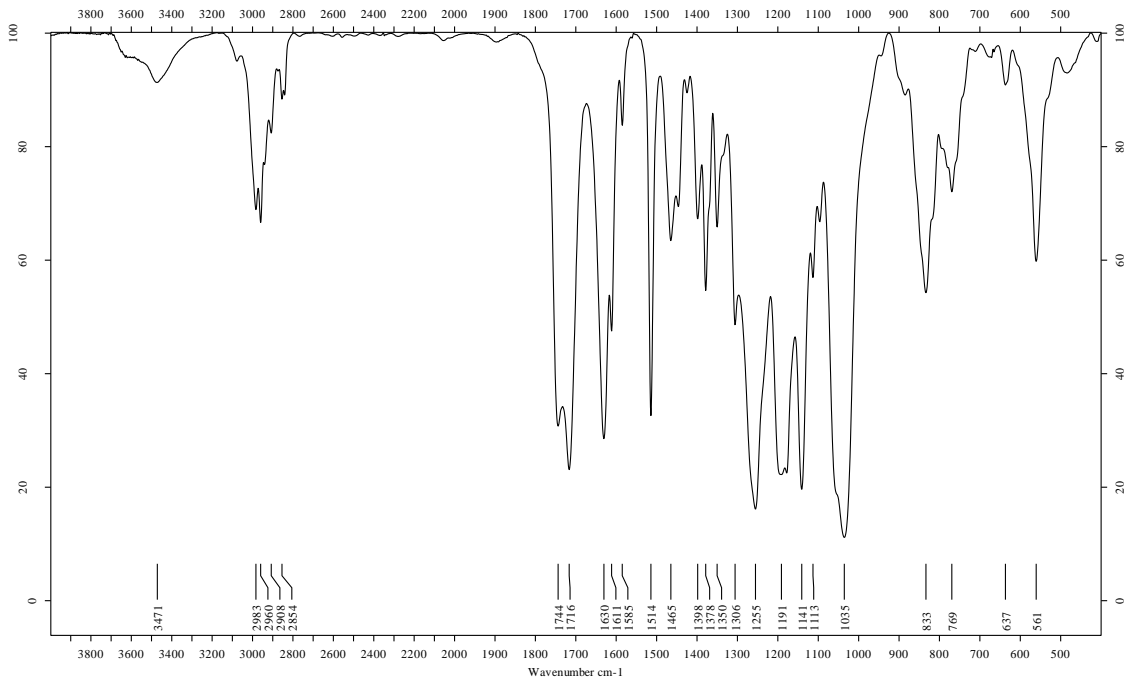
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FIDRES 1.105709 Hz
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RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8852070 MHz
NUC1 13C
P1 9.90 usec
PLW1 80.09999847 W
SFO2 600.0024000 MHz
NUC2 1H
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PCPD2 80.00 usec
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F2 - Processing parameters
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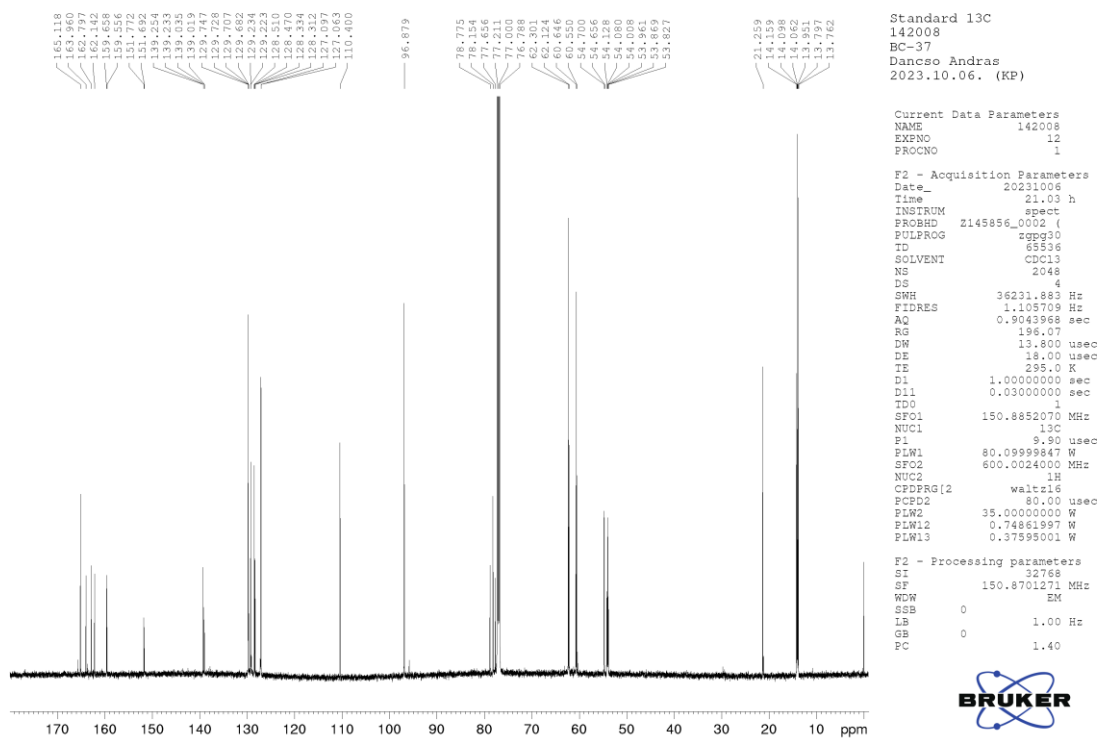
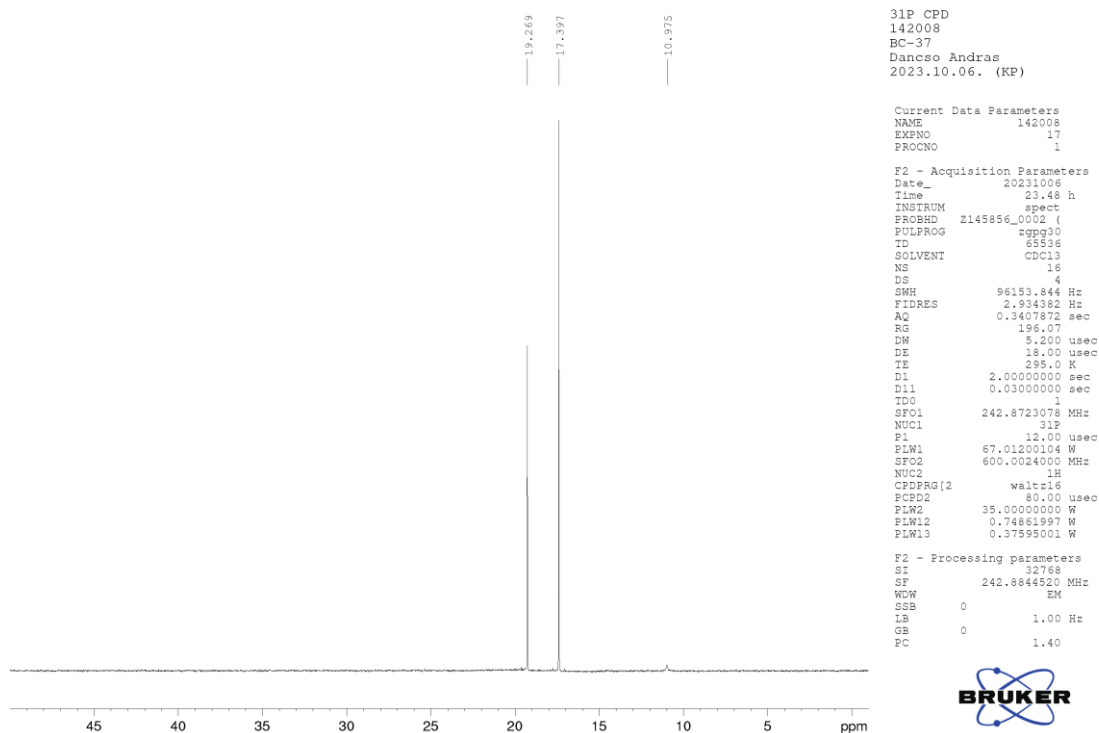


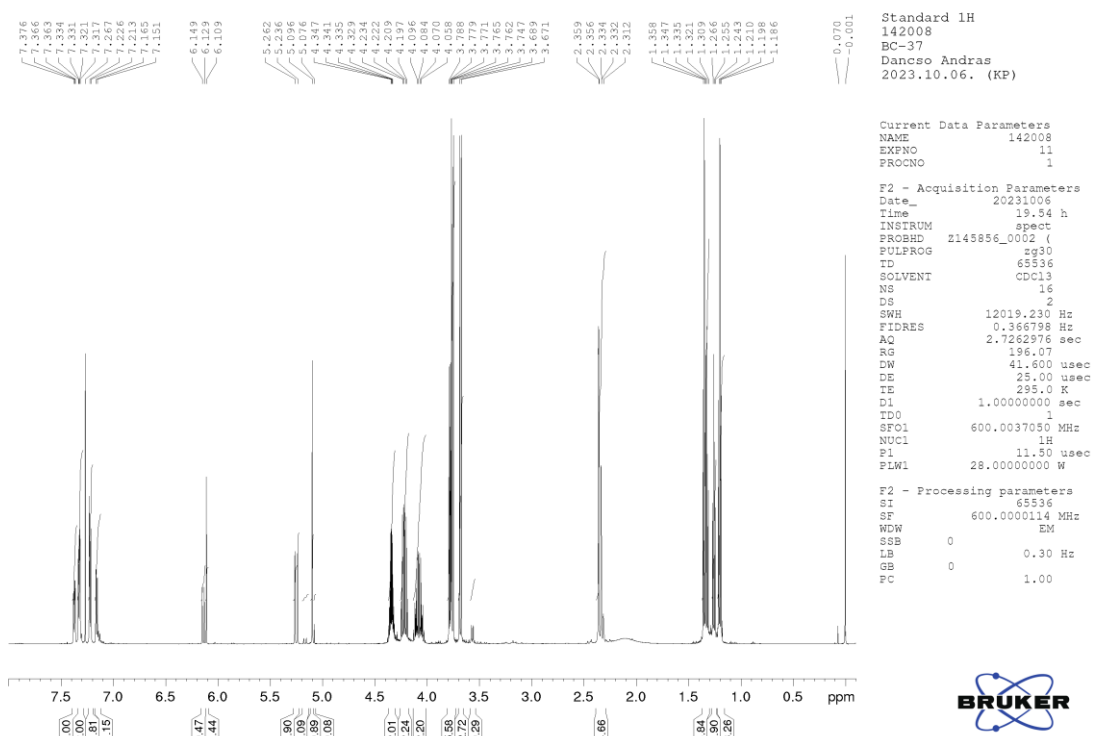


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				Number of Scans: 16

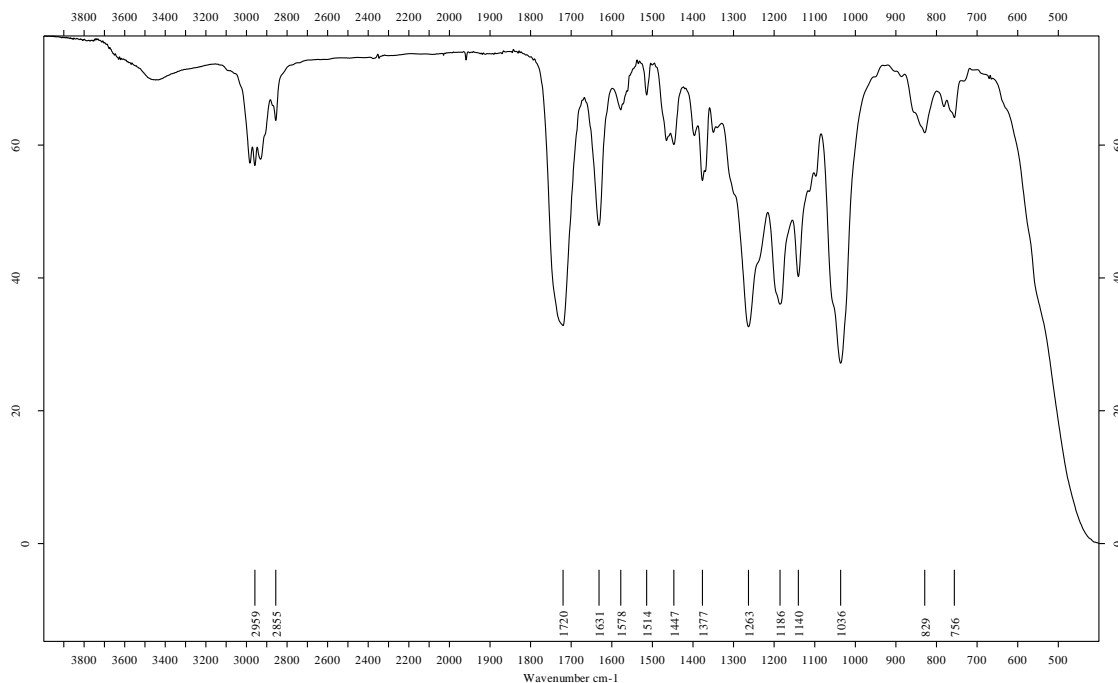


Dimethyl (1,2-Di(ethoxycarbony)ethenyl)oxy-4-methylbenzylphosphonate (2Bc)

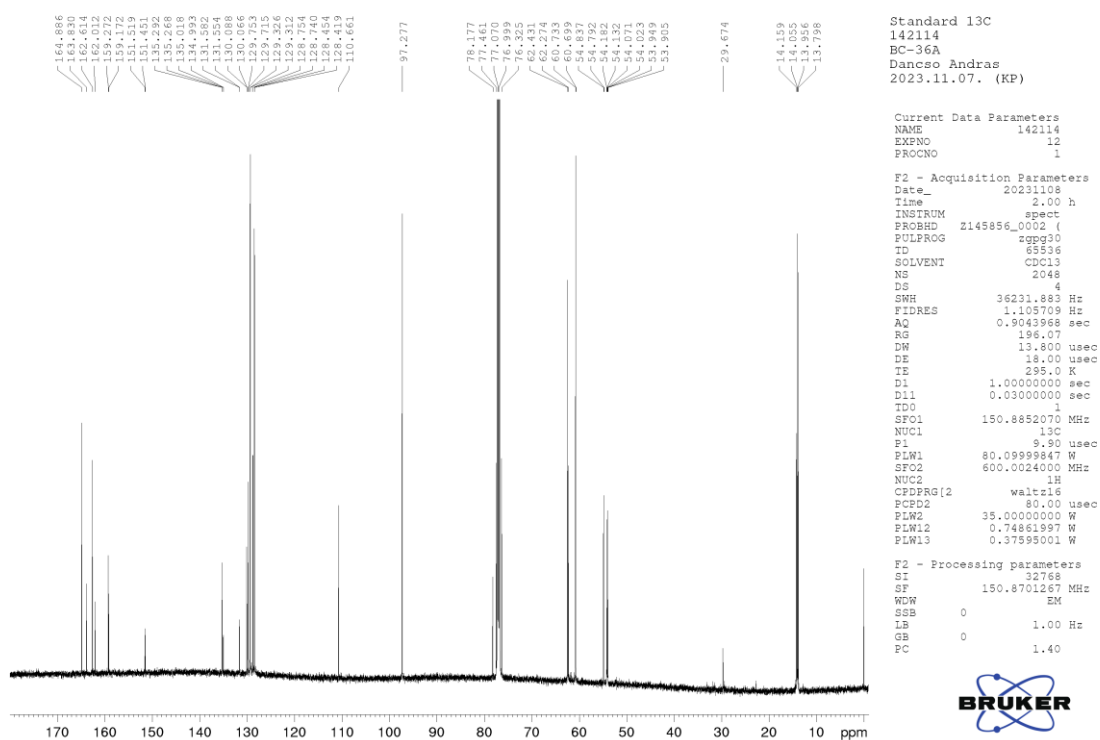
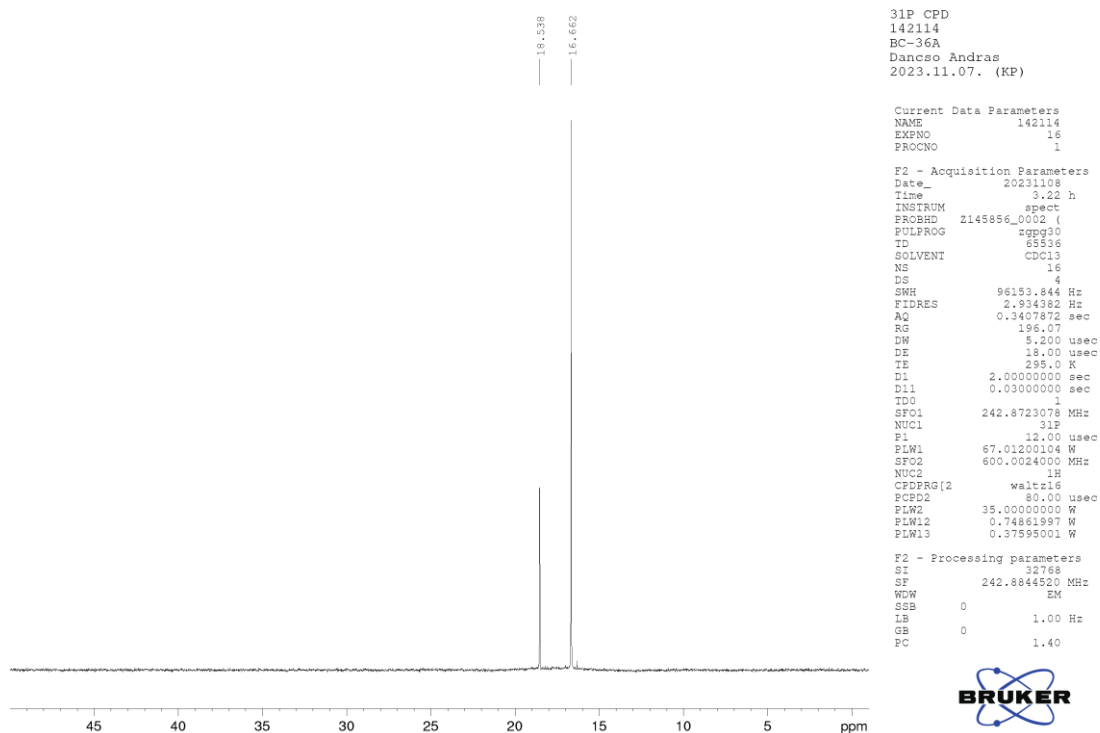


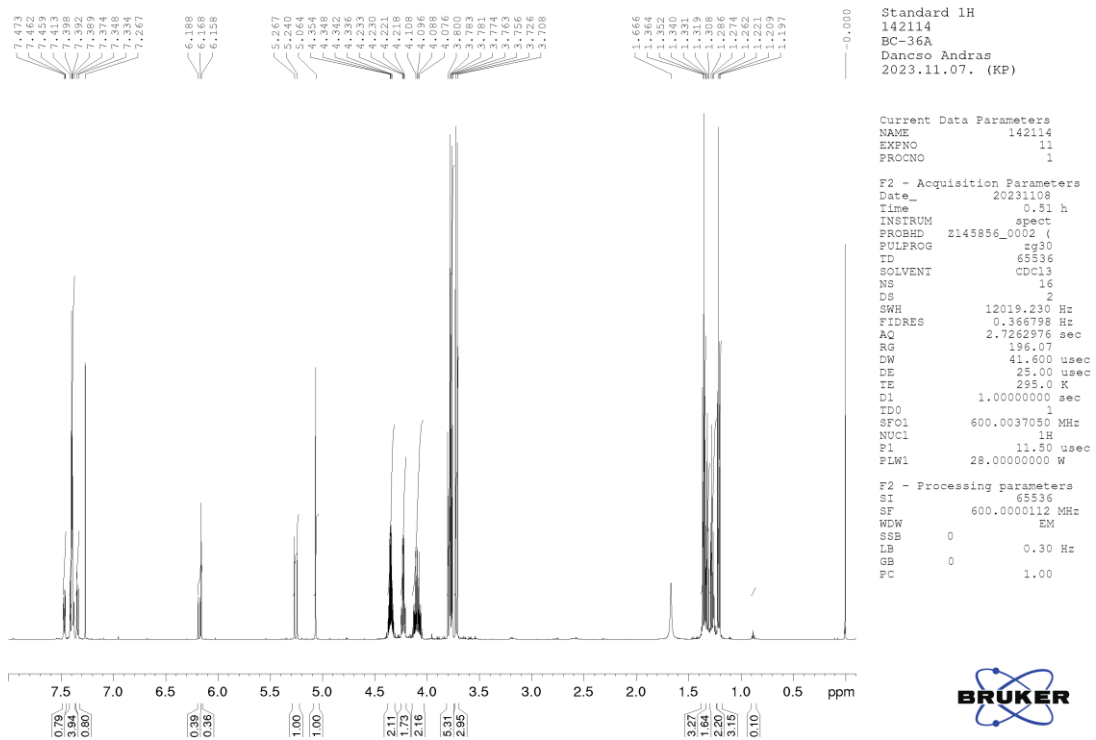


142008	Milen Mátyás	DA	BRUKER Alpha
BC-37	film	5/23/2024	Resolution: 2 cm-1
			Number of Scans: 16



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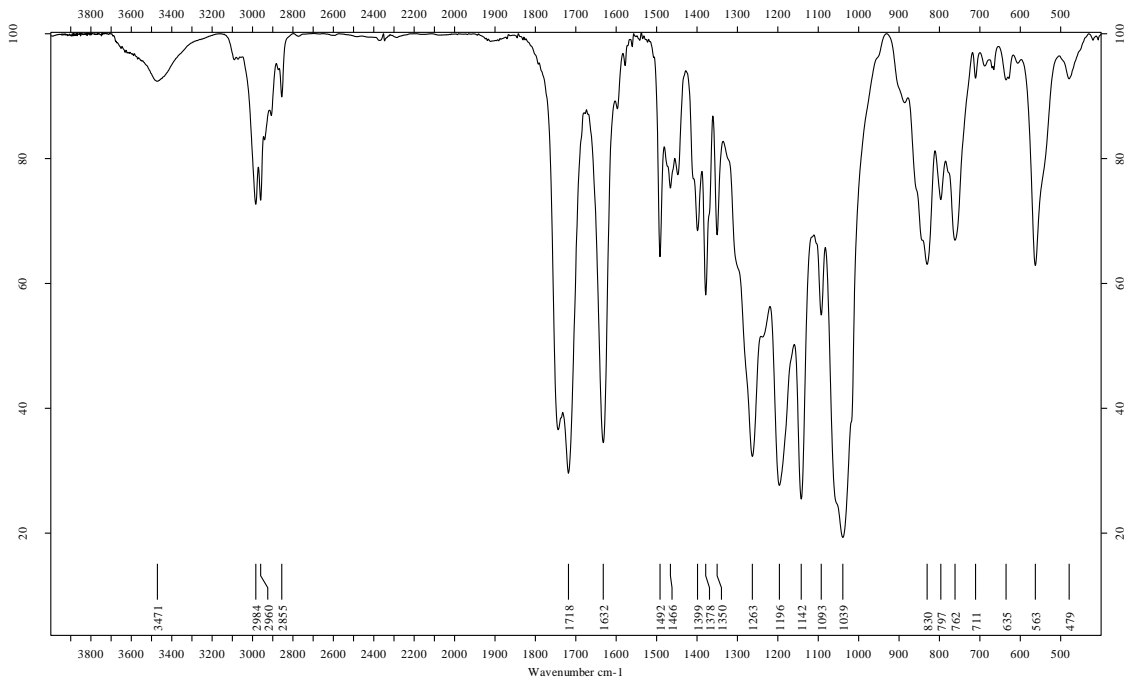




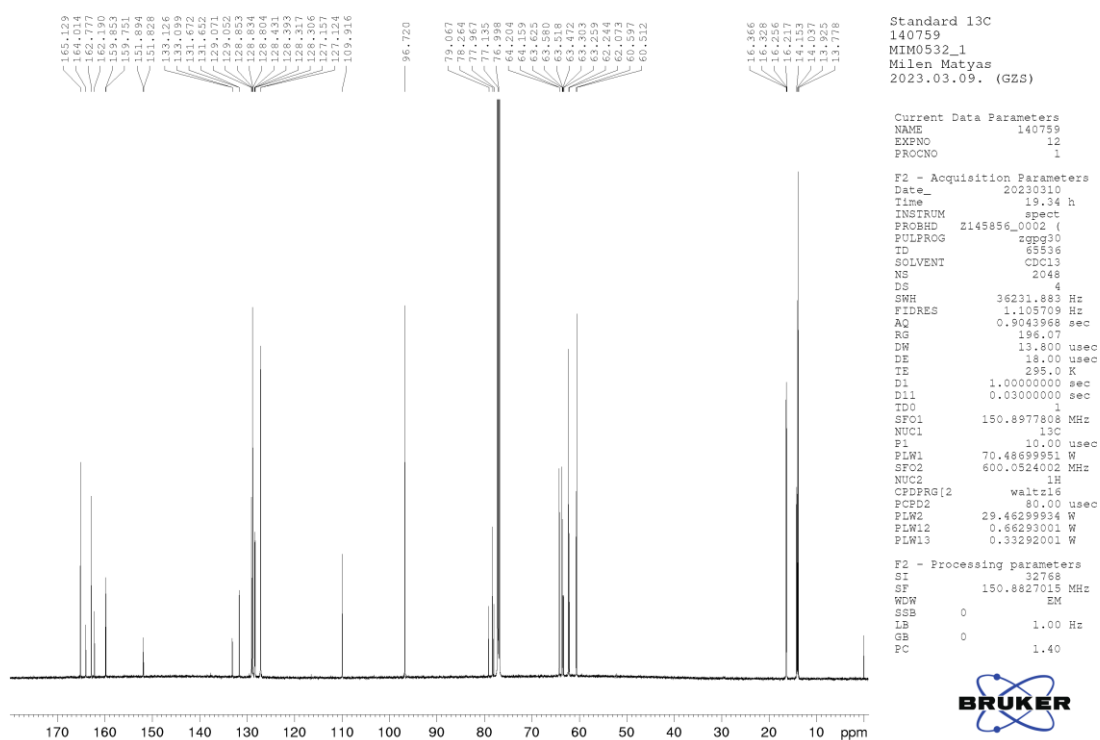
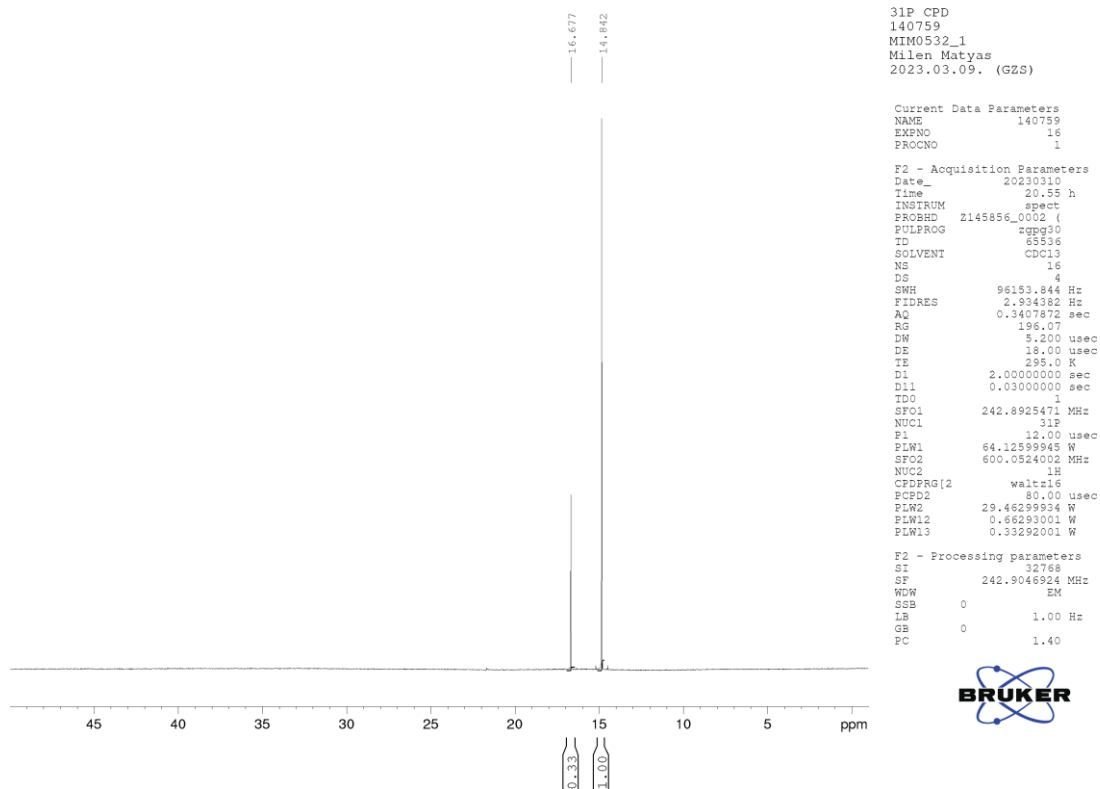
142114
BC-36A

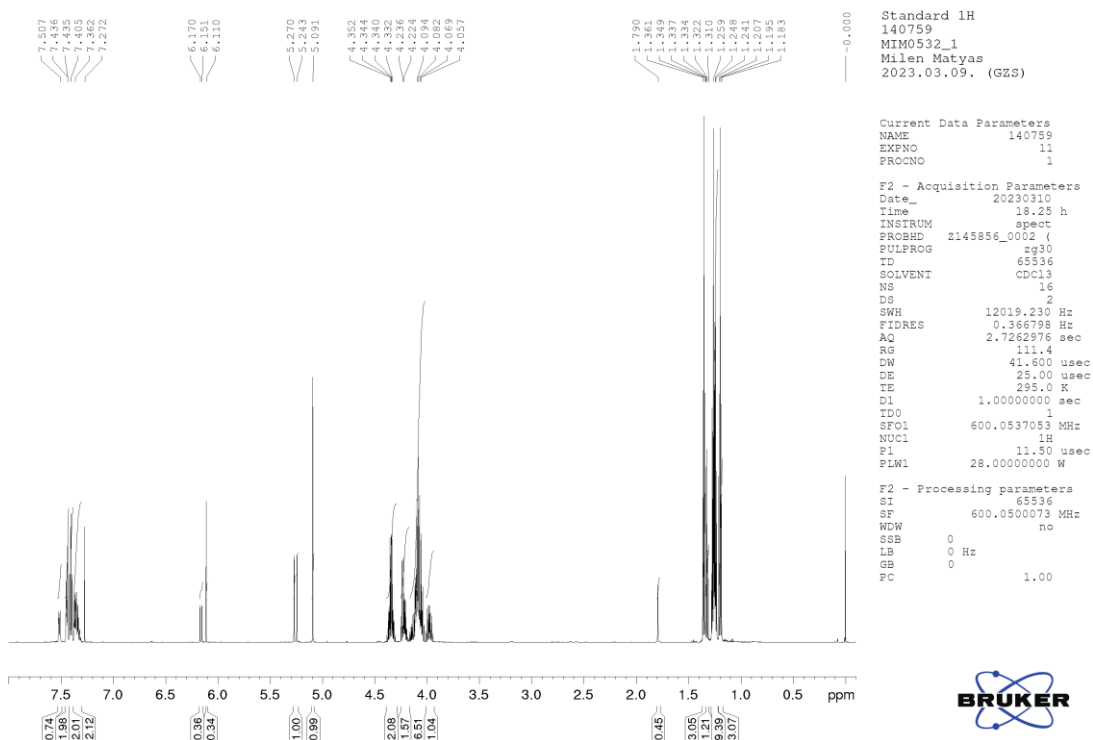
Bese Cintia	DA
film	5/9/2024

BRUKER Alpha
Resolution: 2 cm-1
Number of Scans: 16

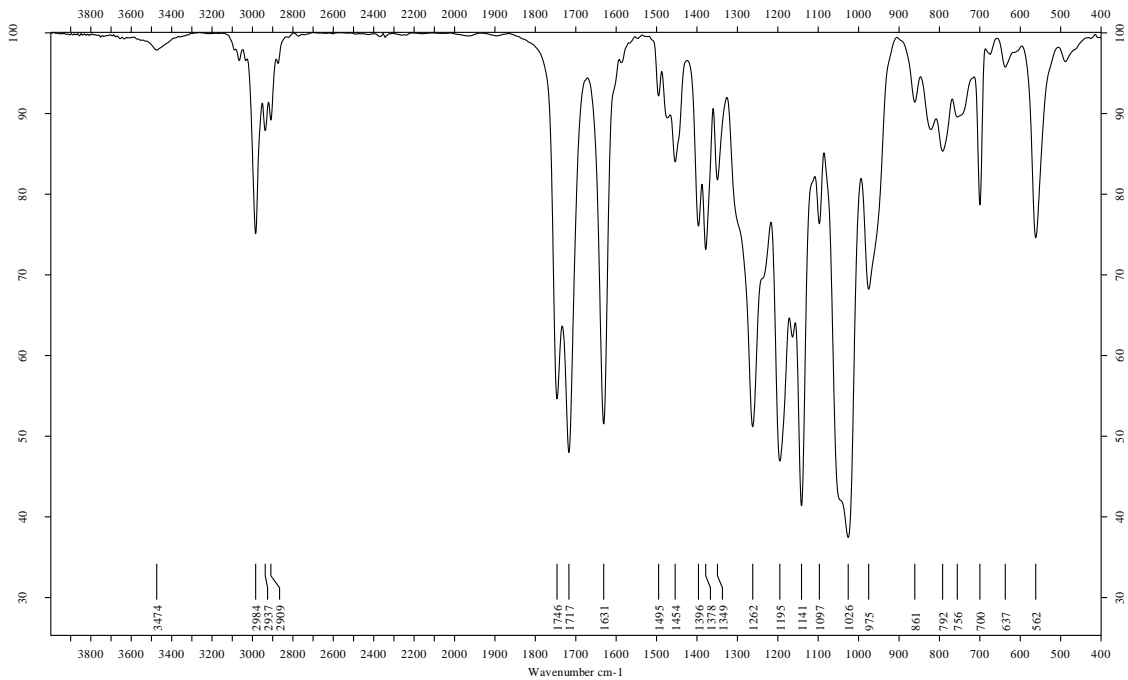


Diethyl (1,2-Di(ethoxycarbonyl)ethenoxy)-benzylphosphonate (2Be)



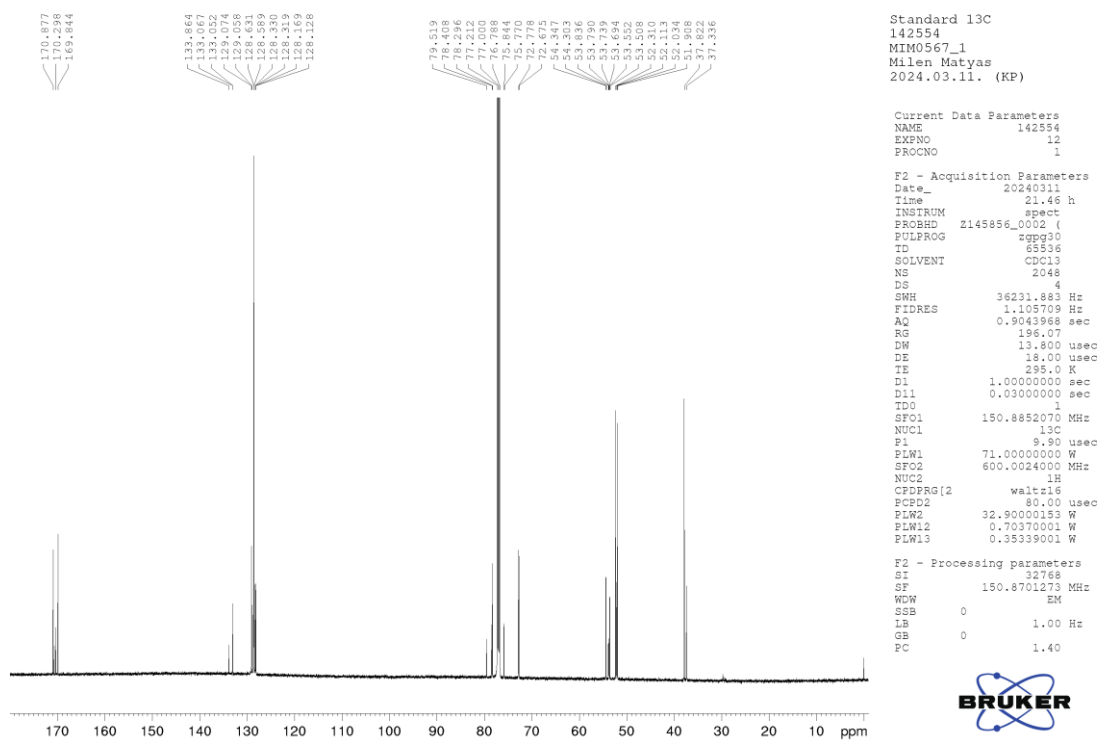
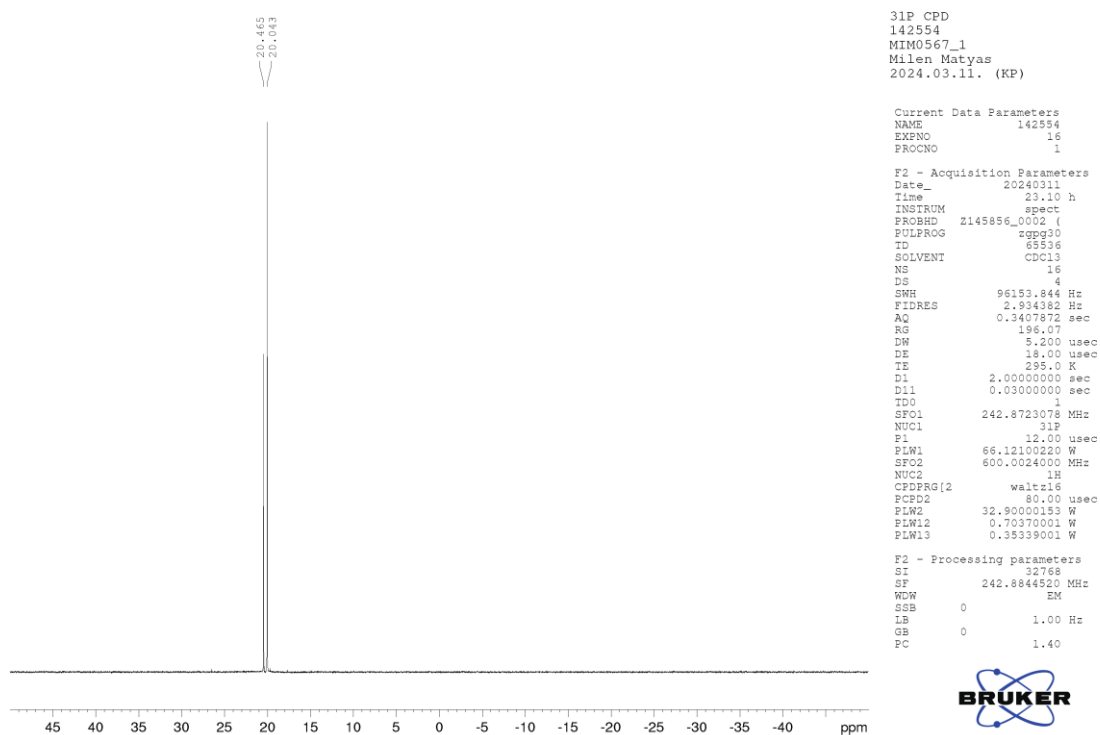


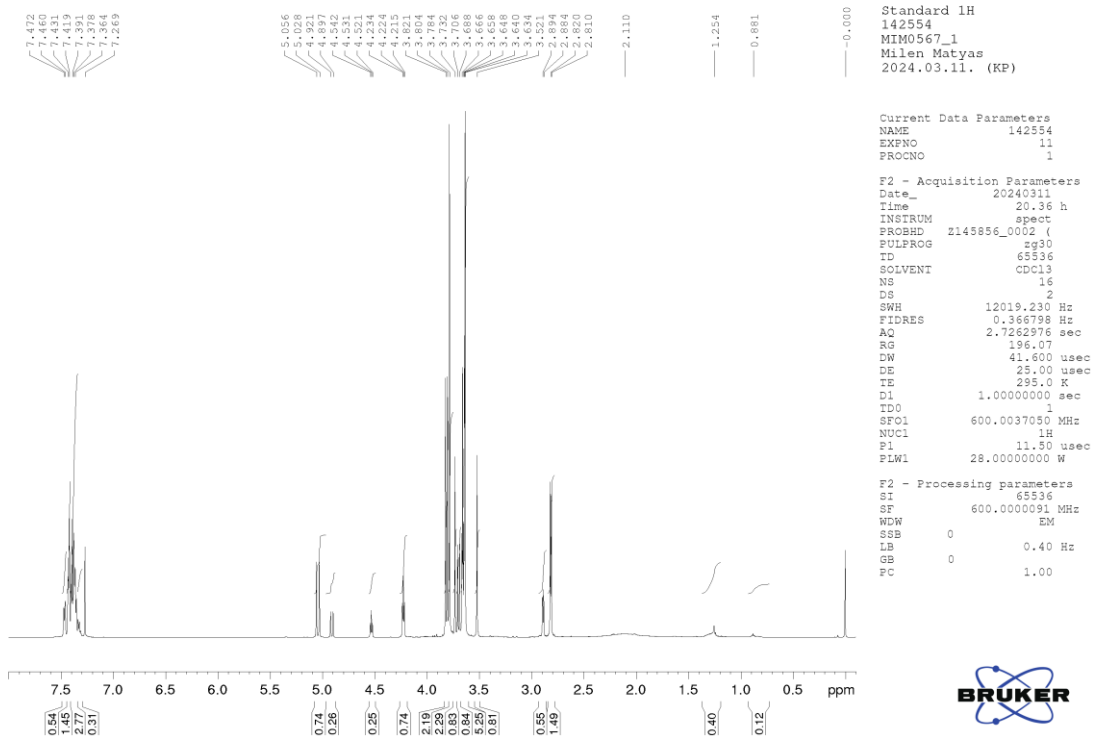
140759	Milen Matyas	KP	BRUKER Alpha
MIM0532_1	Film	3/16/2023	
			Number of Scans: 16



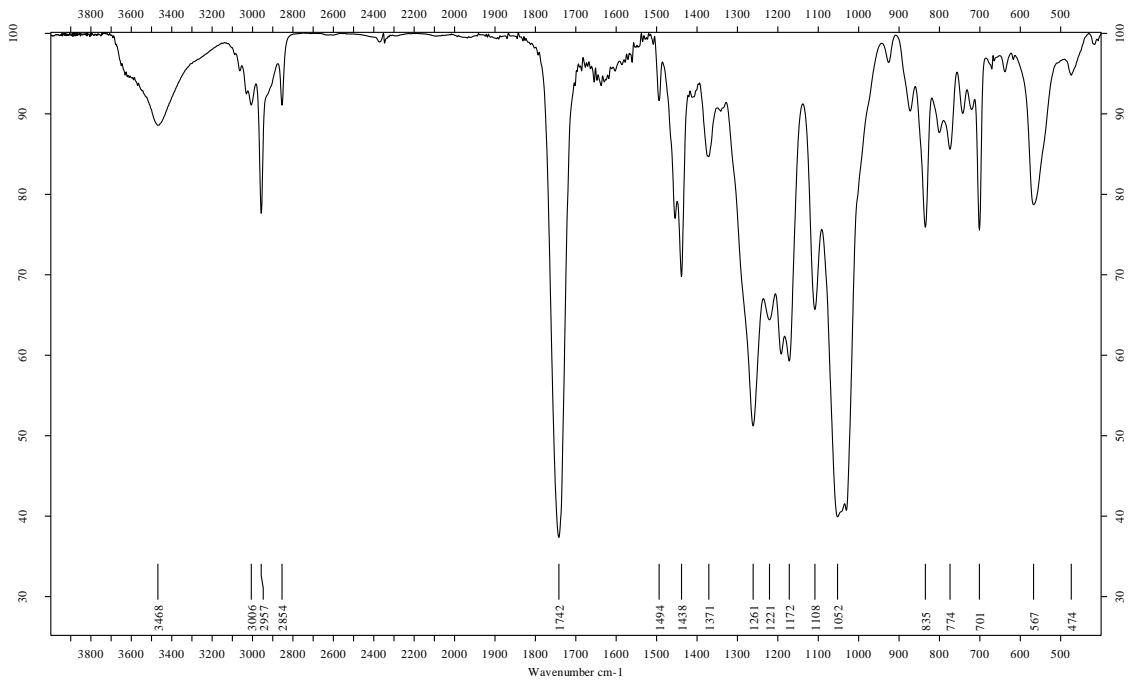
2.) Hydrogenated derivatives

1,4-Dimethyl 2-[(Dimethoxyphosphoryl)(phenyl)methoxy]butanedioate (3Aa)

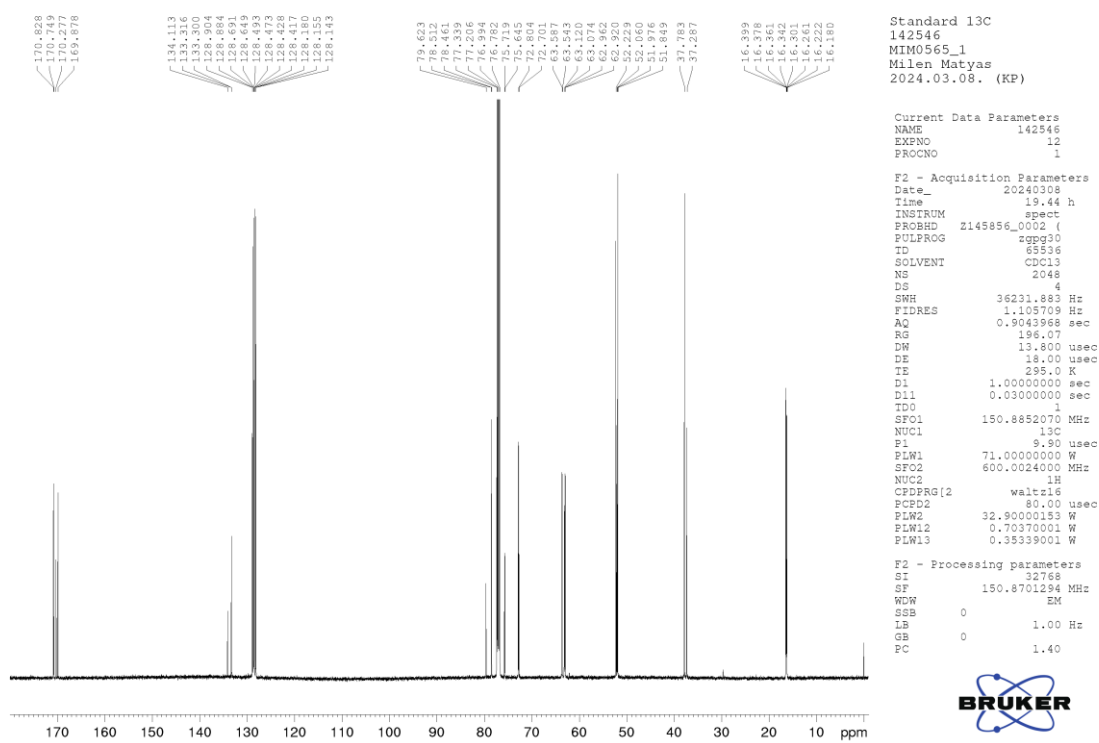
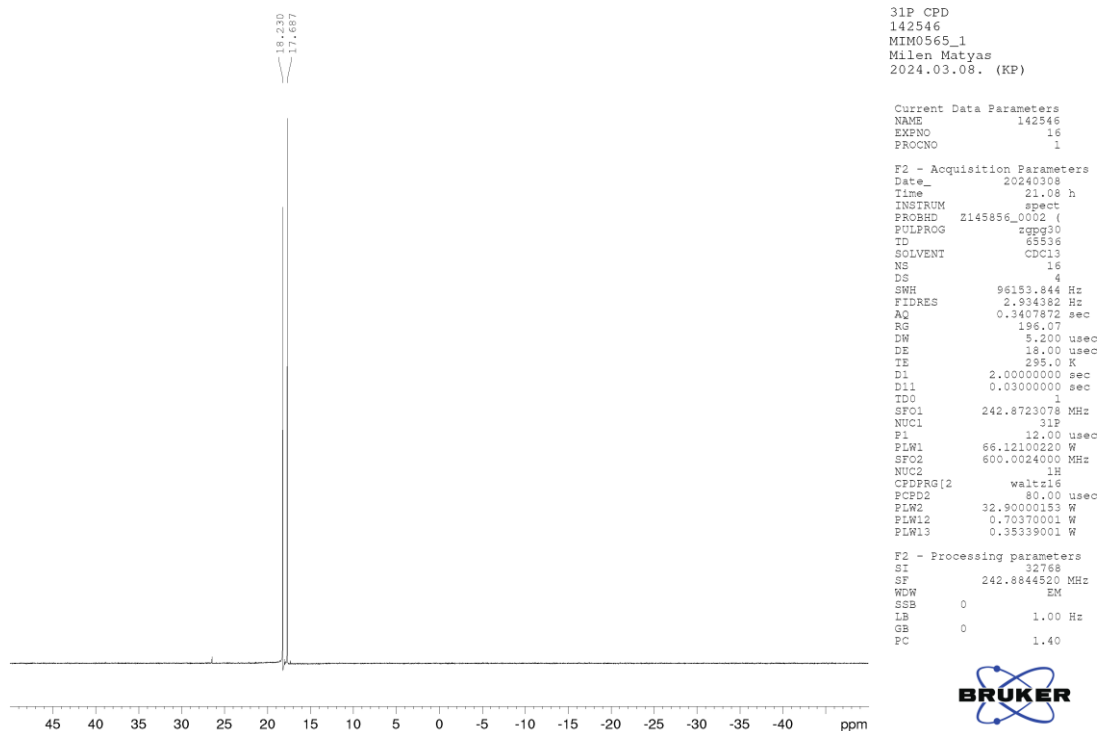


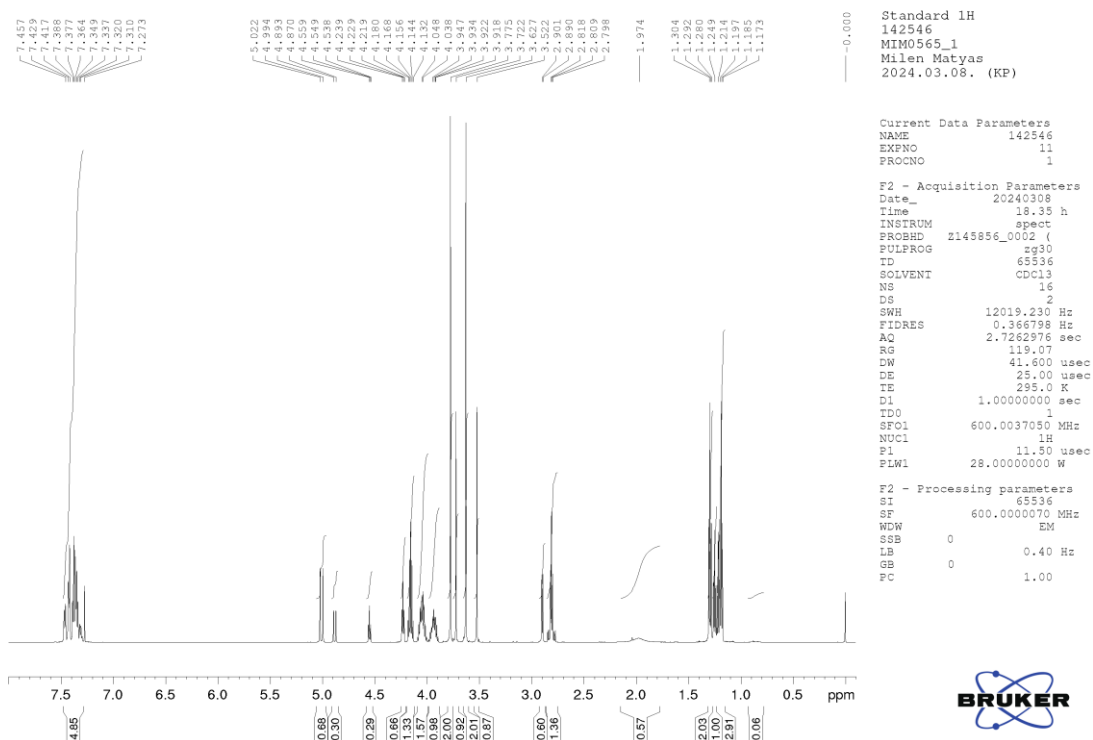


142554	Milen Matyas	KP	BRUKER Alpha
MIM0567_1	Film	3/12/2024	
			Number of Scans: 16

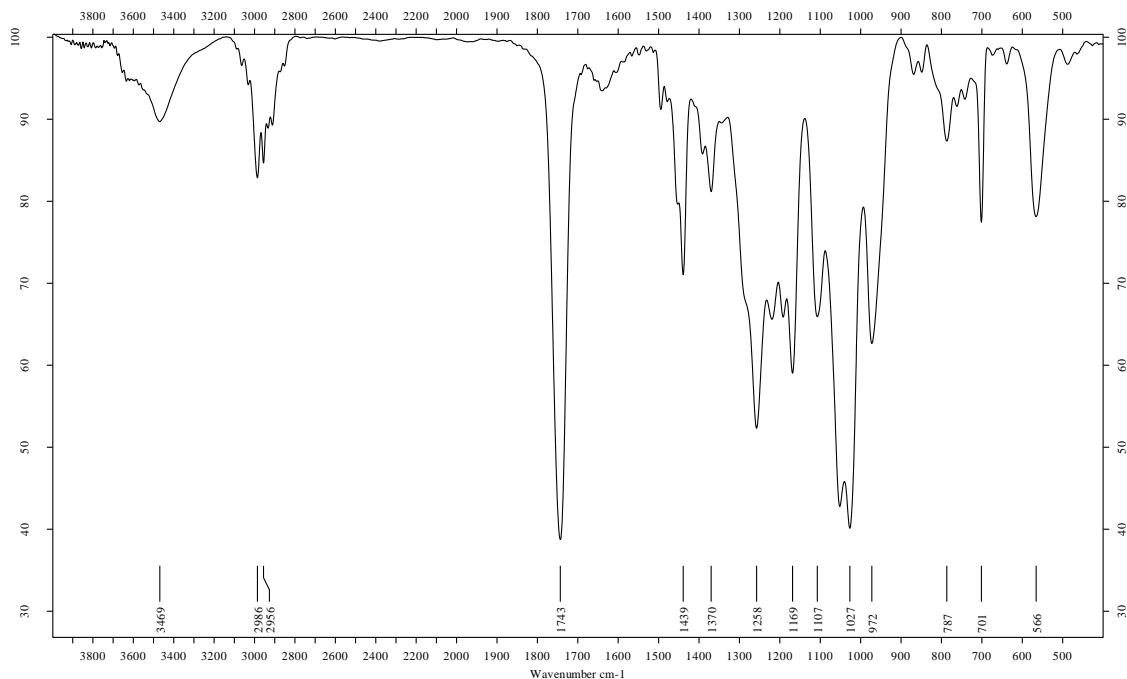


1,4-Dimethyl 2-[(Diethoxyphosphoryl)(phenyl)methoxy]butanedioate (3Ae)

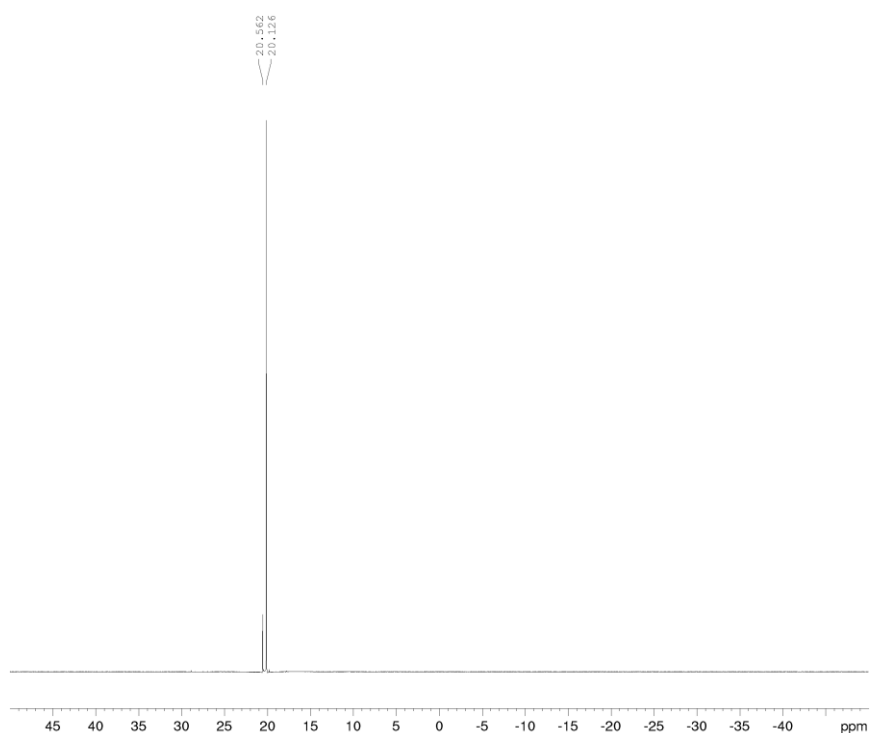




142546	Milen Matyas	KP	BRUKER Alpha
MIM0565_1	Film	3/11/2024	
			Number of Scans: 16



1,4-Diethyl 2-[(Dimethoxyphosphoryl)(phenyl)methoxy]butanedioate (3Ba)

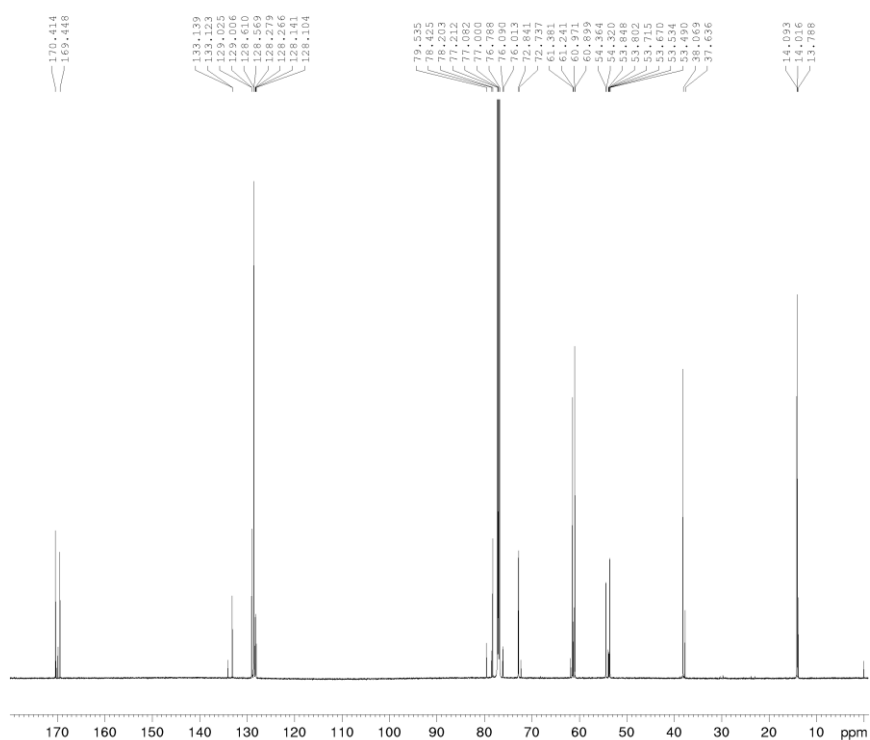


31P CPD
142487
MIM0561_2
Milen Matyas
2024.02.23. (KP)

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PROCNO 1

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DE 18.00 usec
TE 295.0 K
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D11 0.03000000 sec
TD0 1
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NUC1 31P
P1 12.00 usec
PLW1 66.12100220 W
SFO2 600.0024000 MHz
NUC2 1H
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PLW13 0.35339001 W

F2 - Processing parameters
SI 32768
SF 242.8844520 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



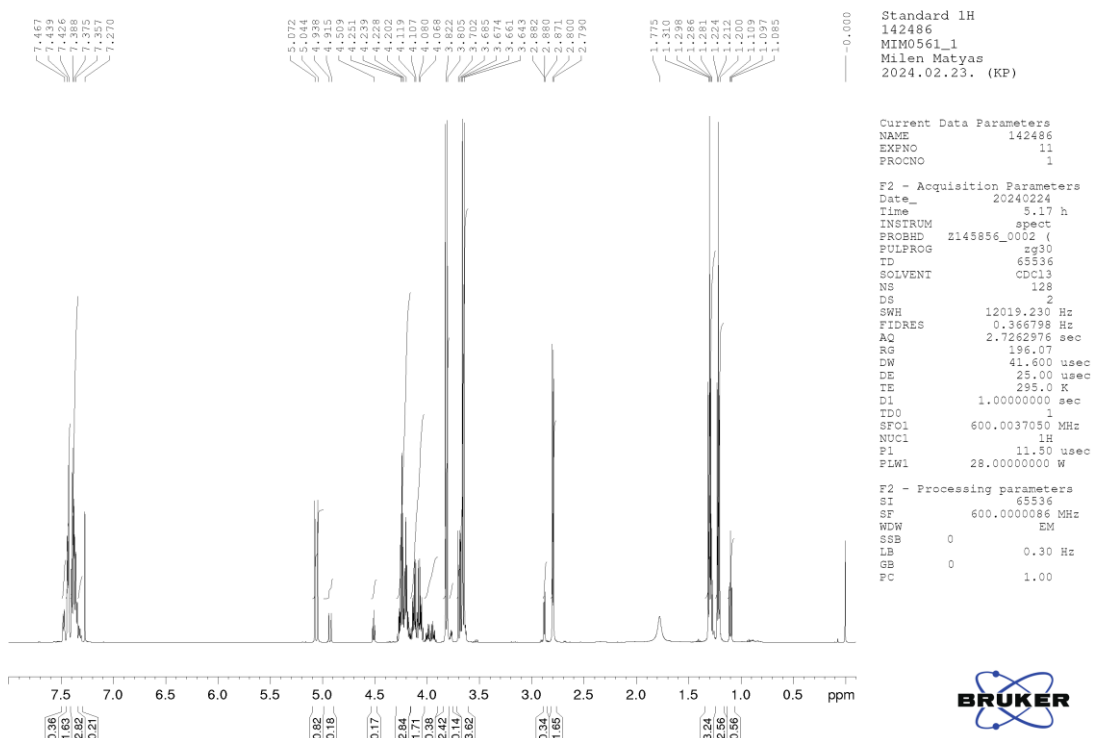
Standard 13C
142486
MIM0561_1
Milen Matyas
2024.02.23. (KP)

Current Data Parameters
NAME 142486
EXPNO 12
PROCNO 1

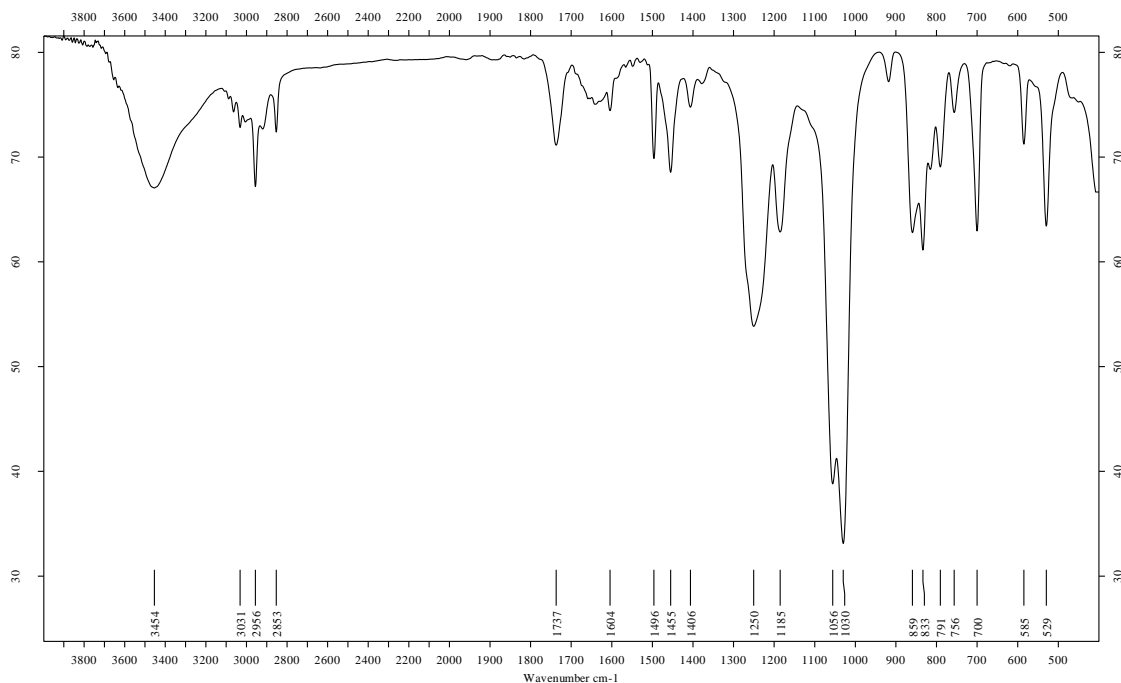
F2 - Acquisition Parameters
Date_ 20240224
Time 9.47 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zgpg30
ID 65536
SOLVENT CDC13
NS 8192
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9043968 sec
RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.00000000 sec
D11 0.03000000 sec
TD0 1
SFO1 150.8852070 MHz
NUC1 13C
P1 9.80 usec
PLW1 71.00000000 W
SFO2 600.0024000 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 32.9000135 W
PLW12 0.70370001 W
PLW13 0.35339001 W

F2 - Processing parameters
SI 32768
SF 150.8701275 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

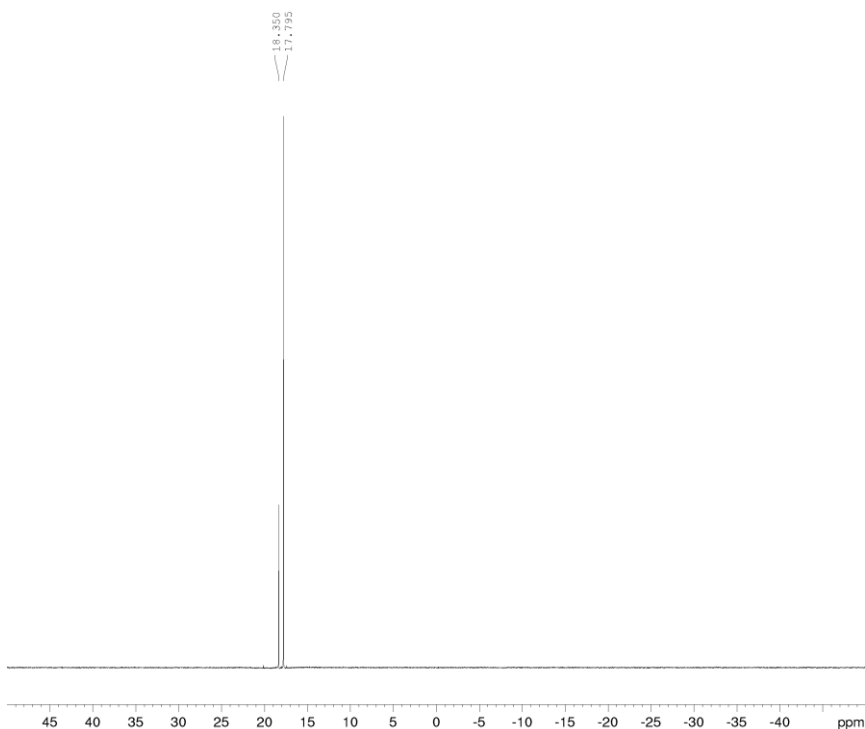




142486	Milen Matyas	KP	BRUKER Alpha
MIM0561_1	KBr	2/26/2024	
			Number of Scans: 16



1,4-Diethyl 2-[(Diethoxyphosphoryl)(phenyl)methoxy]butanedioate (3Be)

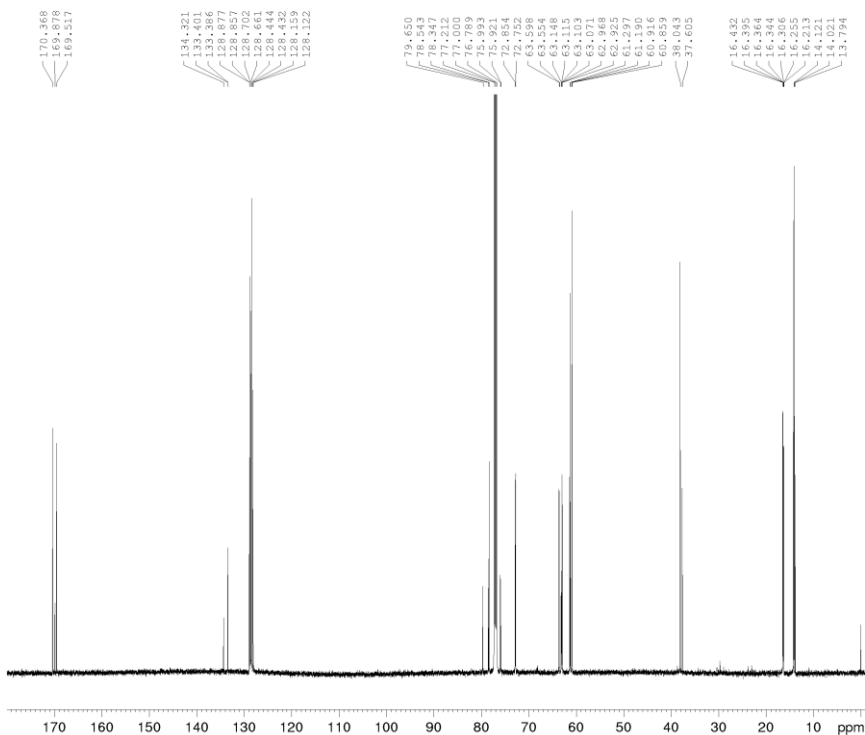


31P CPD
142519
MIM0560_1A
Milen Matyas
2024.03.04. (KP)

Current Data Parameters
NAME 142519
EXPNO 16
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240304
Time 20.51 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zgpg30
ID 65536
SOLVENT CDC13
NS 16
DS 4
SWH 96153.844 Hz
FIDRES 2.934382 Hz
AQ 0.3407872 sec
RG 196.07
DW 5.200 usec
DE 18.00 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
ID0 1
SFO1 242.8723078 MHz
NUC1 31P
P1 12.00 usec
PLW1 66.12100220 W
SFO2 600.0024000 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 32.90000155 W
PLW12 0.70370001 W
PLW13 0.35339001 W

F2 - Processing parameters
SI 32768
SF 242.8844520 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



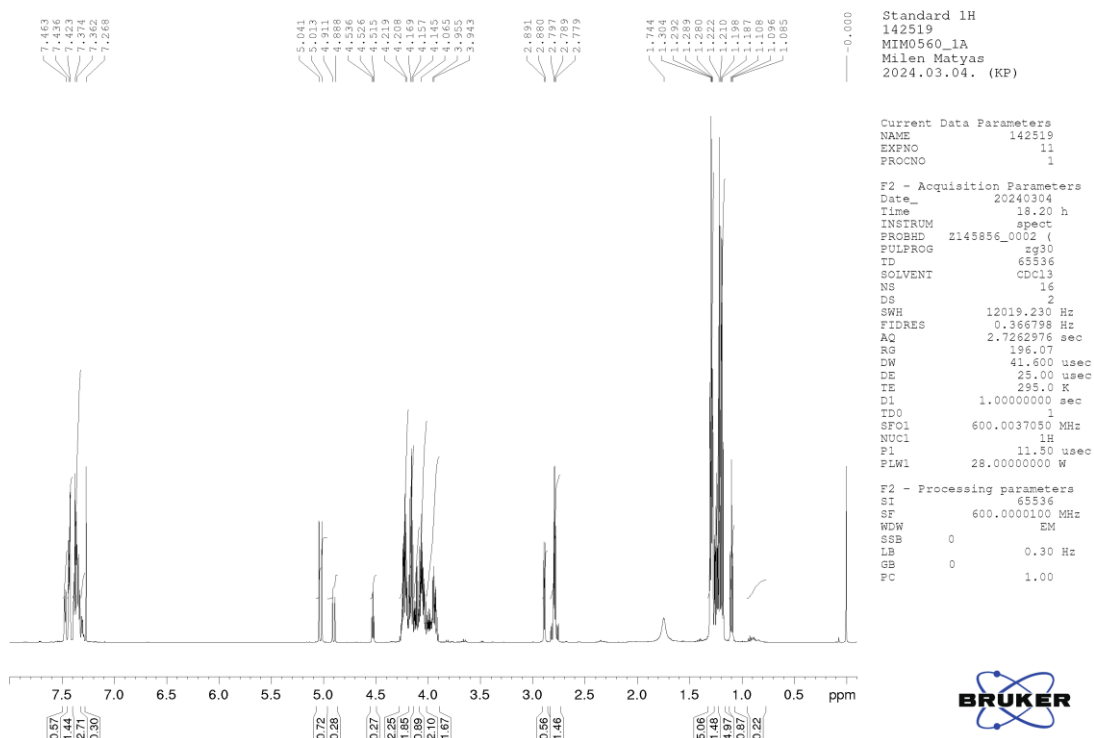
Standard 13C
142519
MIM0560_1A
Milen Matyas
2024.03.04. (KP)

Current Data Parameters
NAME 142519
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20240304
Time 19.28 h
INSTRUM spect
PROBHD Z145856_0002 ()
PULPROG zgpg30
ID 65536
SOLVENT CDC13
NS 2048
DS 4
SWH 36231.883 Hz
FIDRES 1.105709 Hz
AQ 0.9043968 sec
RG 196.07
DW 13.800 usec
DE 18.00 usec
TE 295.0 K
D1 1.00000000 sec
D11 0.03000000 sec
ID0 1
SFO1 150.8852070 MHz
NUC1 13C
P1 9.90 usec
PLW1 71.00000000 W
SFO2 600.0024000 MHz
NUC2 1H
CPDPRG2 waltz16
PCPD2 80.00 usec
PLW2 32.90000155 W
PLW12 0.70370001 W
PLW13 0.35339001 W

F2 - Processing parameters
SI 32768
SF 150.8701268 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





142485	Milen Matyas	KP	BRUKER Alpha
MIM0560_1	KBr	2/26/2024	
			Number of Scans: 16

