Palladium-Catalyzed C-H functionalization and flame-retardant properties of isophosphinolines

Karen-Pacelye Mengue Me Ndong[1,2], Mina Hariri[1,3], Gabin Mwande-Maguene[2], Jacques Lebibi[2], Fatemeh Darvish[3], Christine Safi[4], Kouceila Abdelli[4], Adam Daïch[4], Claire Negrell[1], Rodolphe Sonnier[5], Loic Dumazert[5], Abdou Rachid Issaka Ibrahim[6], Ilagouma Amadou Tidjani[6], David Virieux[1], Tahar Ayad[1]*and Jean-Luc Pirat[1]*

[1] ICGM, Univ Montpellier, ENSCM, CNRS, Montpellier, France
[2] Université des Sciences et Techniques de Masuku, Franceville, Gabon
[3] Department of Chemistry, K. N. Toosi University of Technology, Tehran, Iran
[4] Université Le Havre Normandie, Normandie Univ, URCOM UR 3221, FR CNRS 3038, Le Havre, France
[5] Polymers Composites and Hybrids (PCH), IMT Mines Ales, 30319 Ales, France
[6] University Abdou Moumouni of Niamey, Niamey, Niger
* Correspondence: jean-luc.pirat@enscm.fr (J.-L.P.); Tel.: (+033. 448 792 014); tahar.ayad@enscm.fr (T.A.); Tel.: (+033. 448 792 015)

Table of Contents

Comments on the residue contents in MCC versus in TGA.....2

$^{31}P\{^{1}H\}$, ^{1}H and $^{13}C\{^{1}H\}$ NMR and HRMS spectra of the synthesized compounds 3

S1 : (*E*)-2-Phenyl-3-styryl-1*H*-isophosphinoline 2-oxide (**3a**) 3

S2: (*Z*)-3-(1,2-Diphenylvinyl)-2-phenyl-1*H*-isophosphinoline 2-oxide (**3b**) 5

S3: 3-(Cyclohex-2-en-1-yl)-2-phenyl-1*H*-isophosphinoline 2-oxide (**3c**) 9

S4: Dimethyl (*E*)-(2-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)vinyl)phosphonate (**3d**) 11

S5: Diethyl (*E*)-(2-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)vinyl)phosphonate (**3e**) 14

S6: Methyl (*E*)-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylate (**3f**) 16

S7: (*E*)-*N,N*-Dimethyl-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylamide (**3g**) 19

S8: (*E*)-3-(2-Oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylonitrile (**3h**) 21

S9: Methyl 2-methyl-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylate **(3ia)**..... 24

S10: Methyl 2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate (**3ib**) 28

S11: Methyl 3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)-2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate (**3ic**)..... 30

S12: Ethyl 2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate (**3ja**) 33

S13: Ethyl 2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate (**3jb**)..... 36

S14: Ethyl 3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)-2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate (**3jc**) 38

S15: (*E*)-*N*-(4-Methoxybenzyl)-3-(2-oxido-2-phenyl-1H-isophosphinolin-3-yl)acrylamide (**3k**)..... 41

S16: (*E*)-2-Phenyl-3-(2-(phenylsulfonyl)vinyl)-1*H*-isophosphinoline 2-oxide (**3l**)..... 43

S17: (*E*)-2-(2-(2-Oxido-2-phenyl-1*H*-isophosphinolin-3-yl)vinyl)isoindoline-1,3-dione (**3m**)..... 45

Crystallographic data of sulfonated product **3l**..... 45

Comments on the residue contents in MCC versus in TGA

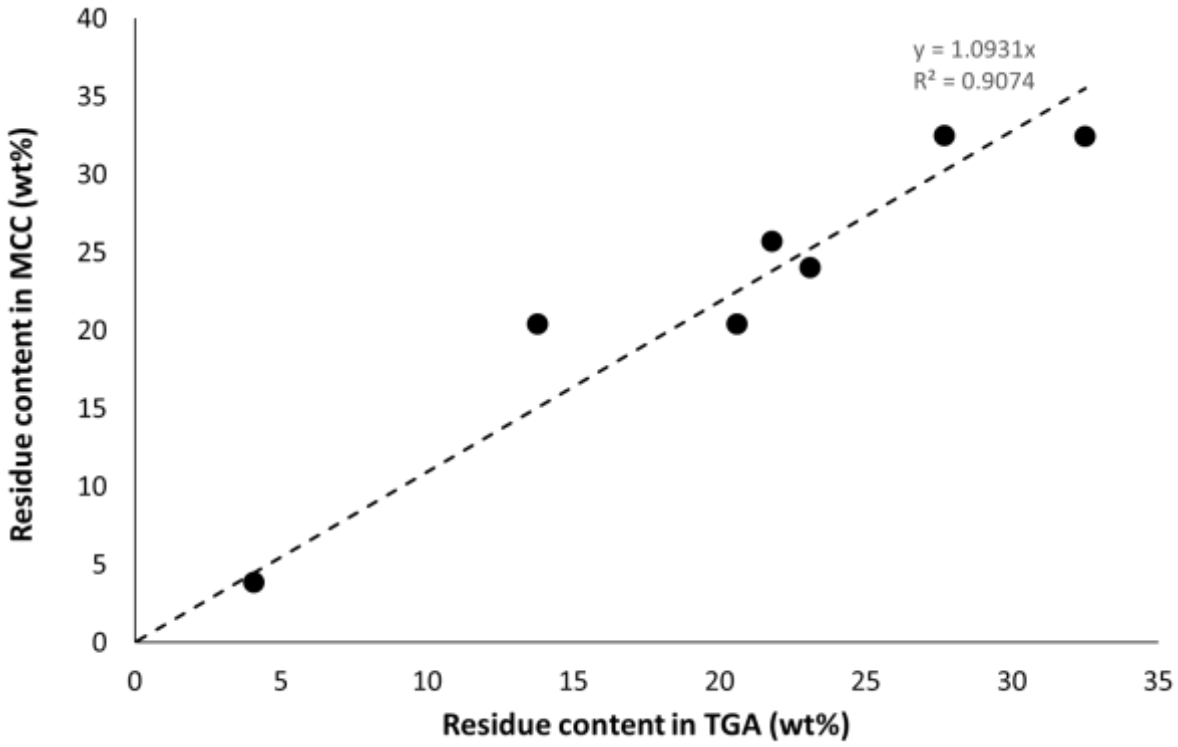
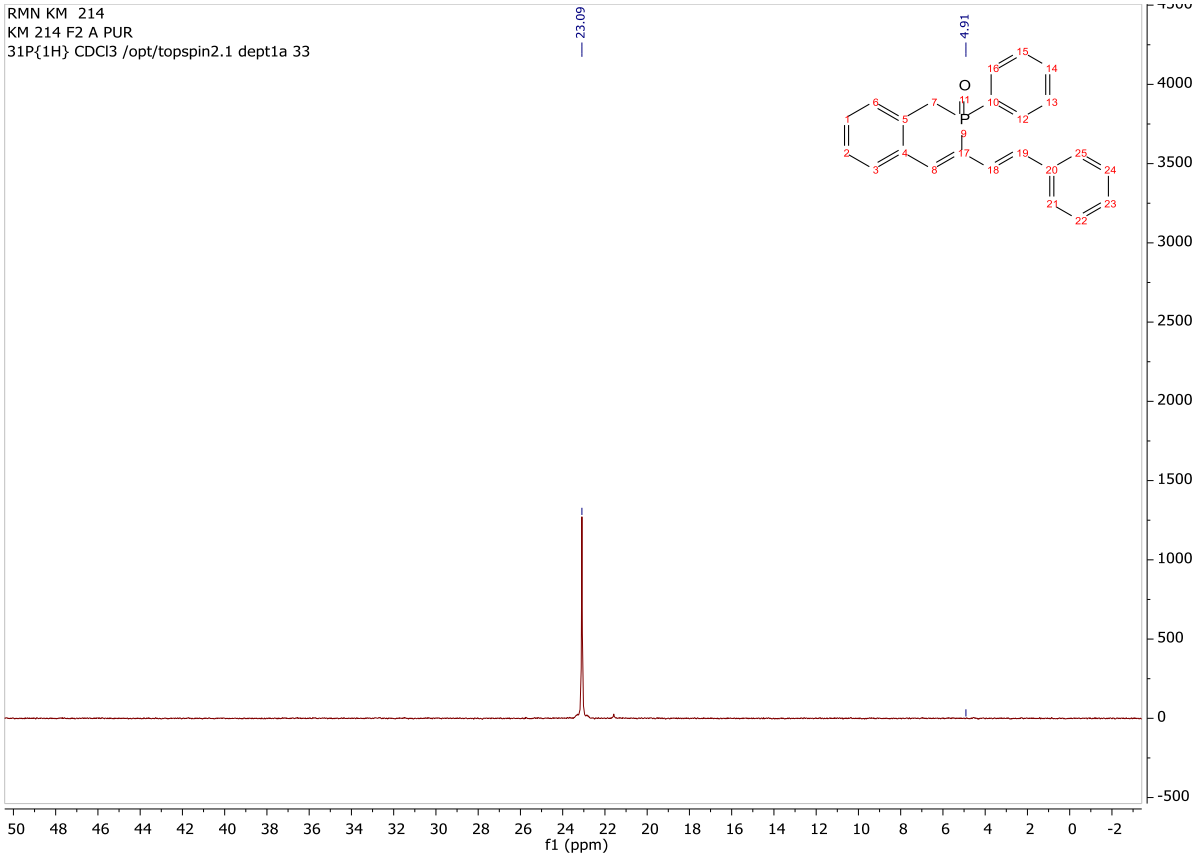


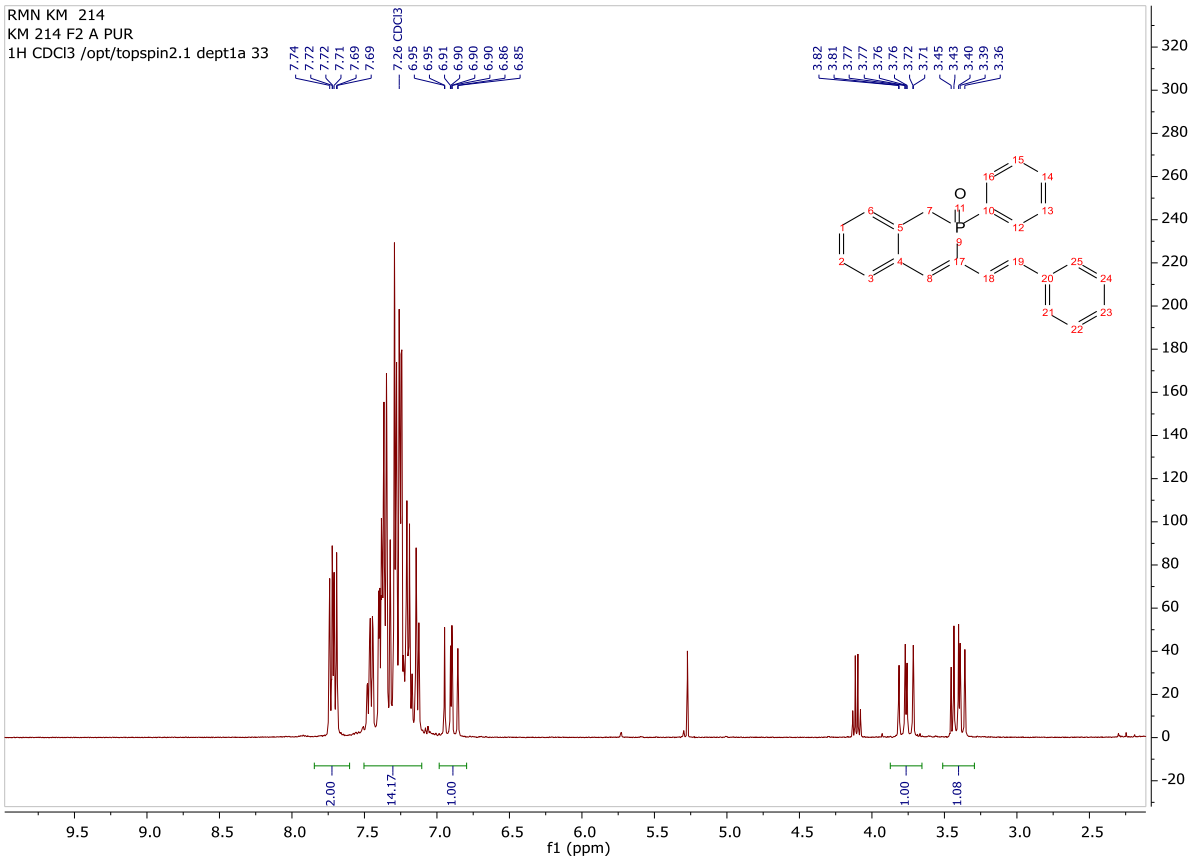
Figure SI1. Residue contents in MCC versus in TGA

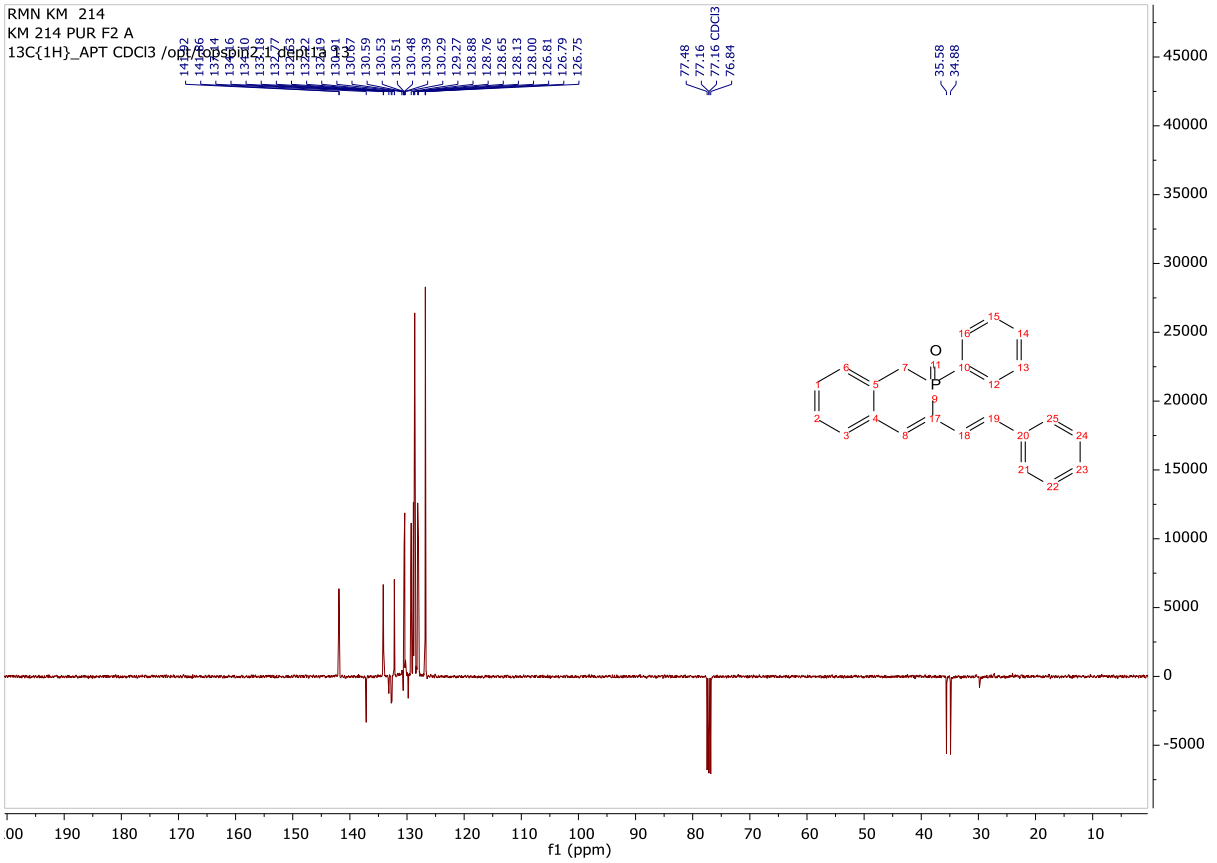
There is a good agreement between the residue contents measured in MCC and in TGA (Figure SI1) despite some differences that may be assigned to the test protocol. The heating rate is much higher in MCC: in some rare cases, the heating rate can impact significantly the pyrolysis pathway and the final residue content.

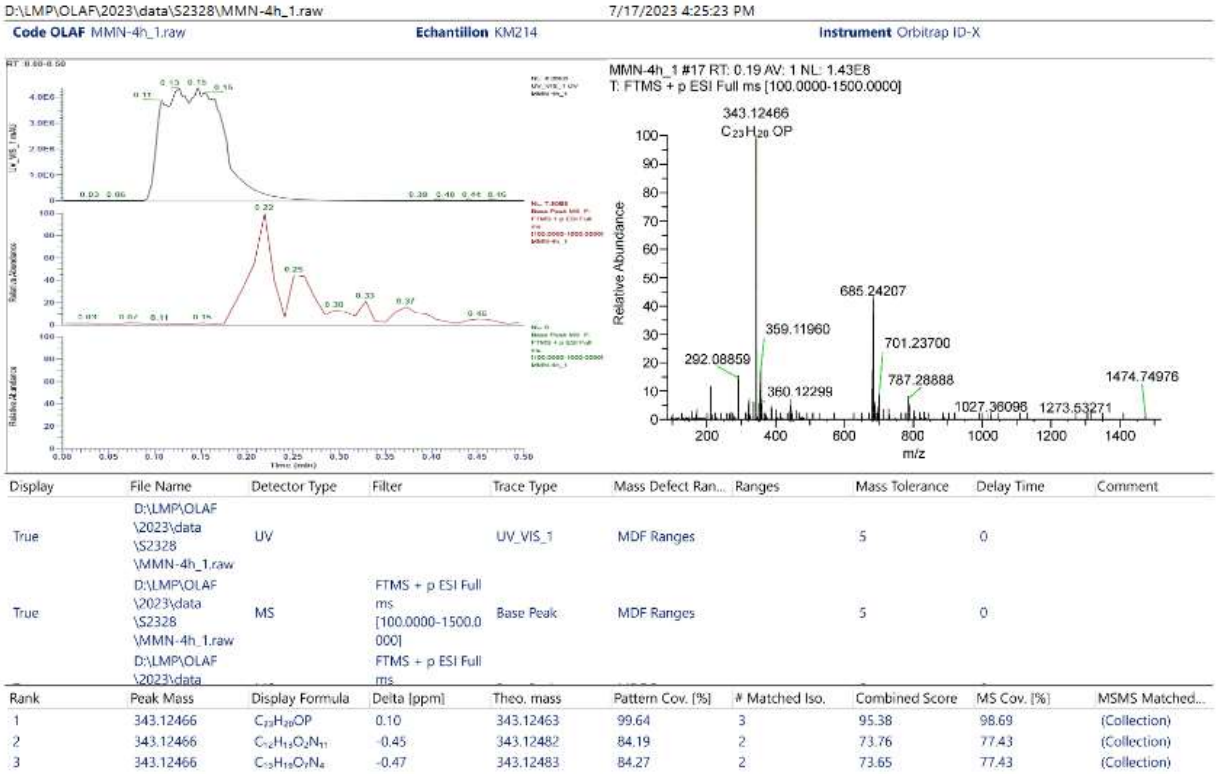
$^{31}P\{^{1}H\}$, ^{1}H and $^{13}C\{^{1}H\}$ NMR and HRMS spectra of the synthesized compounds

S1 : (*E*)-2-Phenyl-3-styryl-1*H*-isophosphinoline 2-oxide **(3a)**





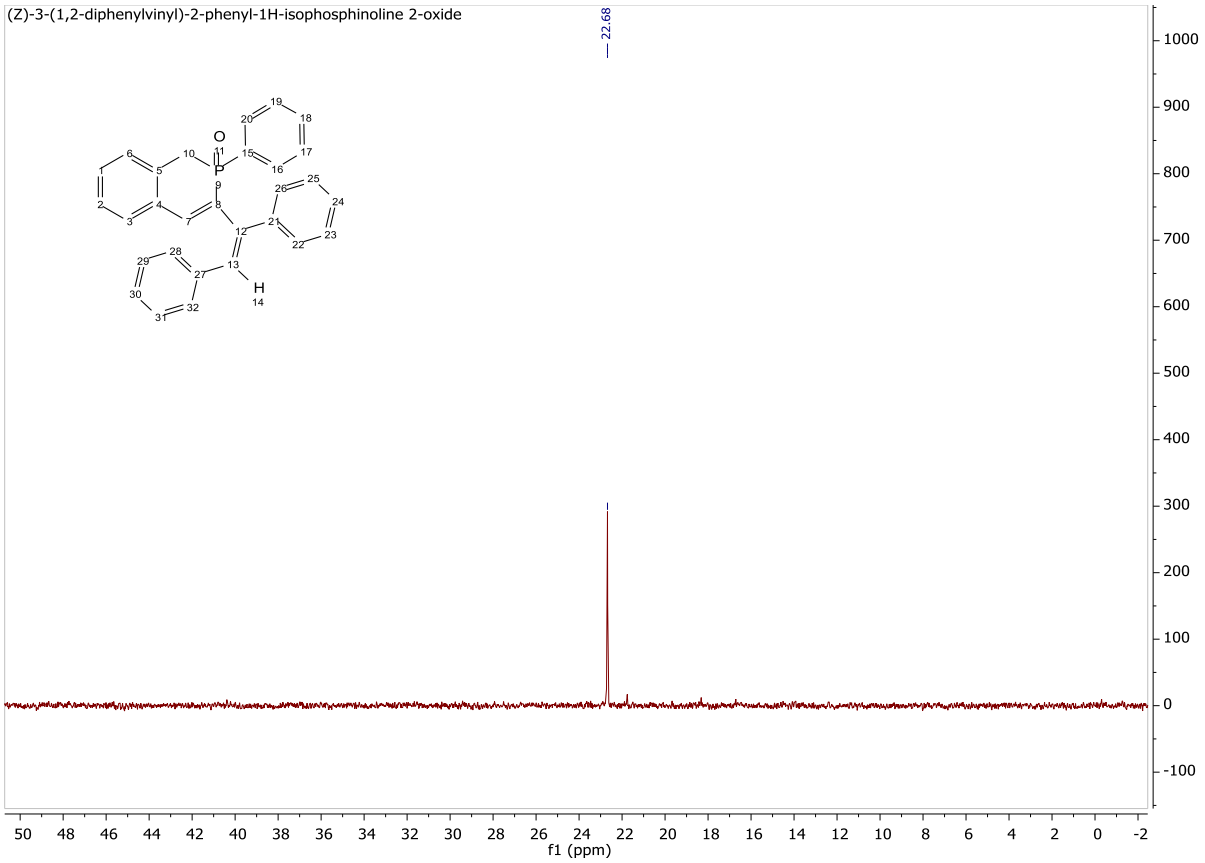


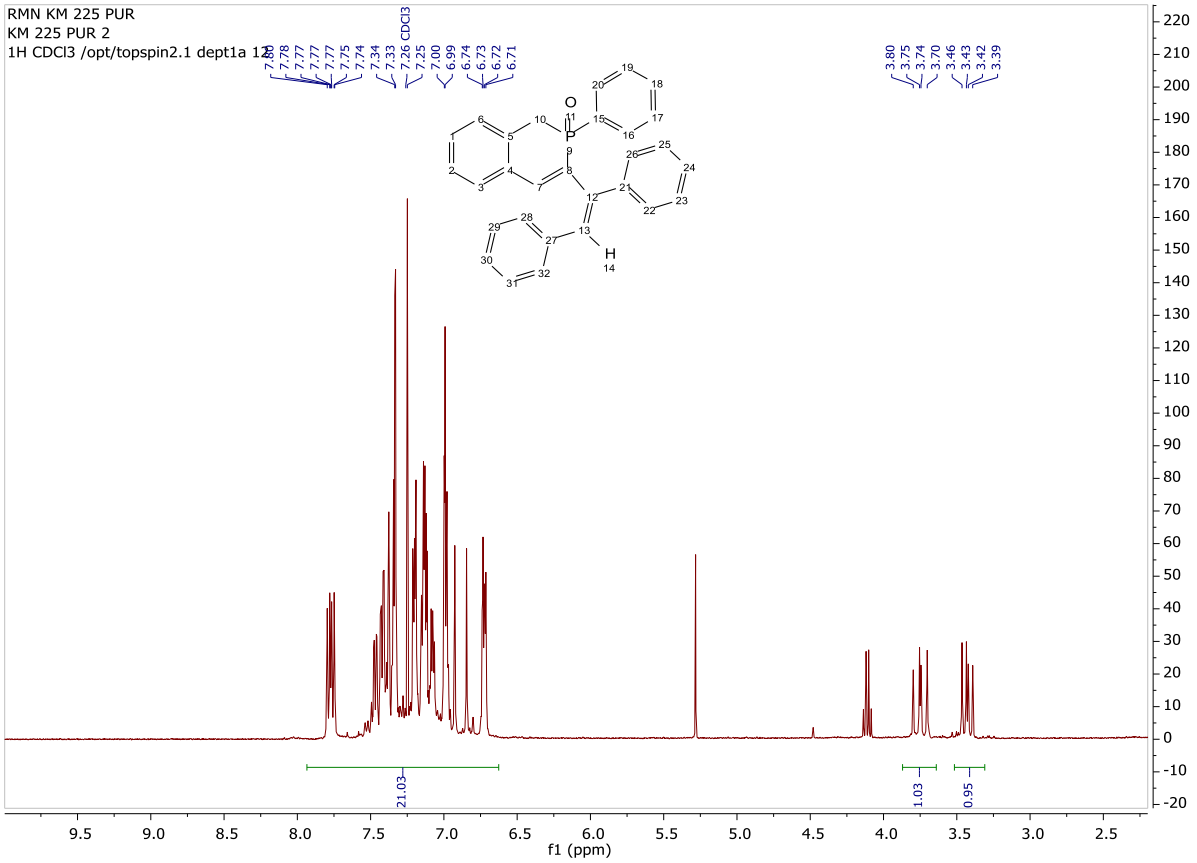


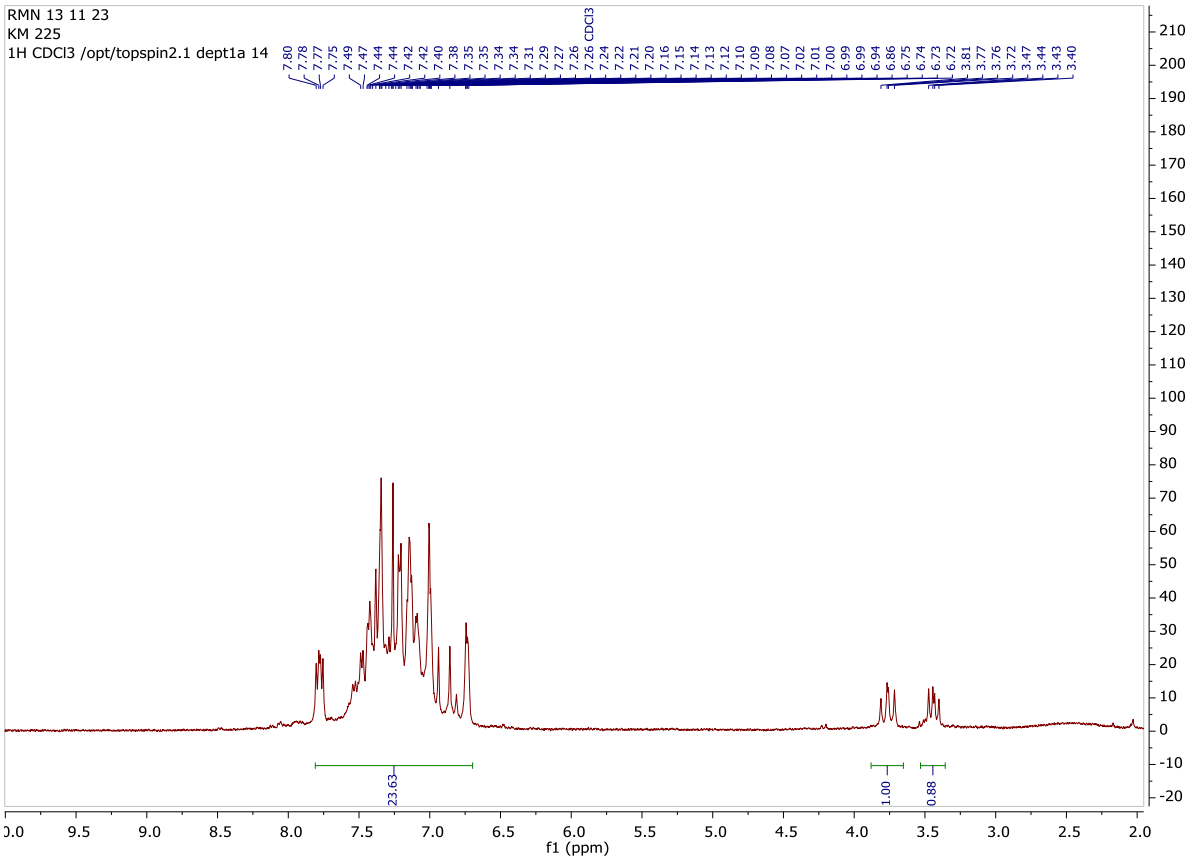
Display	File Name	Detector Type	Filter	Trace Type	Mass Defect Ran...	Ranges	Mass Tolerance	Delay Time	Comment
True	D:\LMP\OLAF\2023\data\S2328\MMN-4h_1.raw	UV		UV_VIS_1	MDF Ranges		5	0	
True	D:\LMP\OLAF\2023\data\S2328\MMN-4h_1.raw	MS	FTMS + p ESI Full ms [100.0000-1500.0000]	Base Peak	MDF Ranges		5	0	
	D:\LMP\OLAF\2023\data		FTMS + p ESI Full ms						

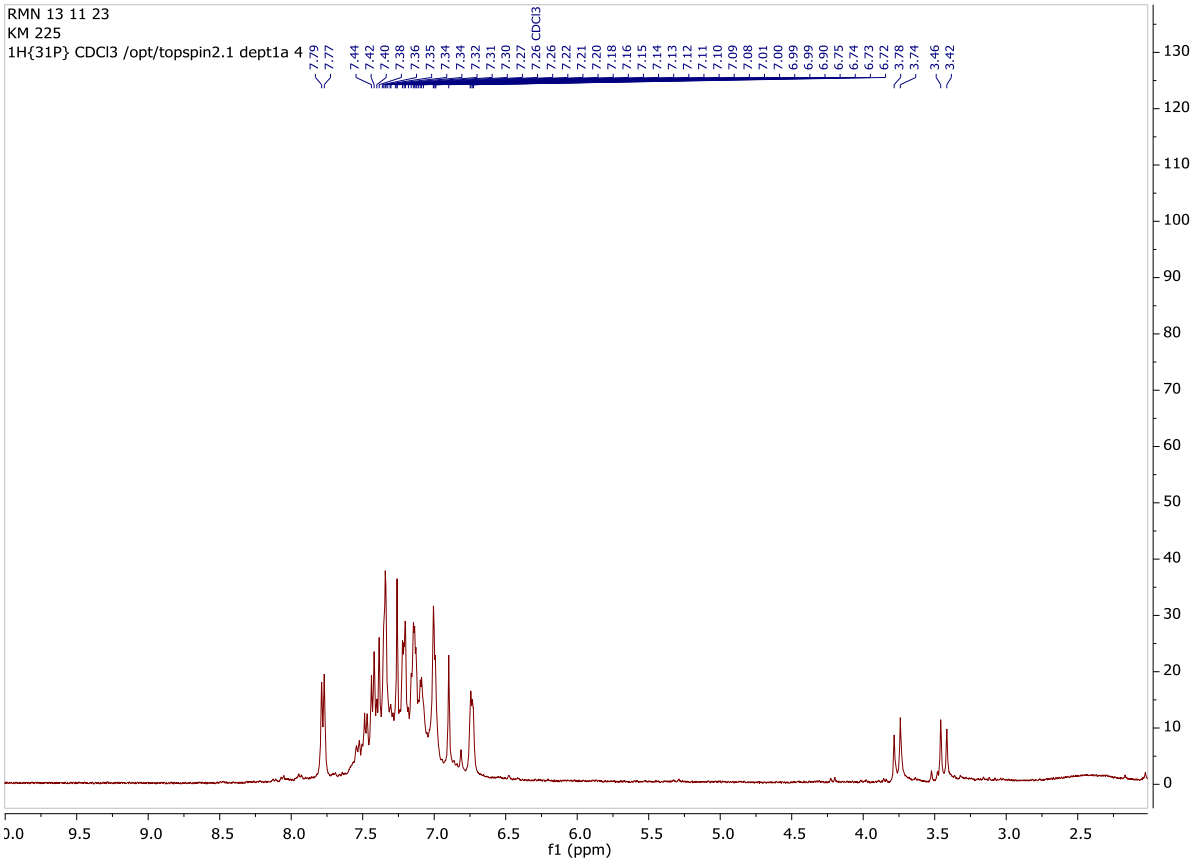
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	343.12466	$C_{23}H_{26}OP$	0.10	343.12463	99.64	3	95.38	98.69	(Collection)
2	343.12466	$C_{24}H_{19}O_2N_{11}$	-0.45	343.12482	84.19	2	73.76	77.43	(Collection)
3	343.12466	$C_{13}H_{15}O_7N_4$	-0.47	343.12483	84.27	2	73.65	77.43	(Collection)

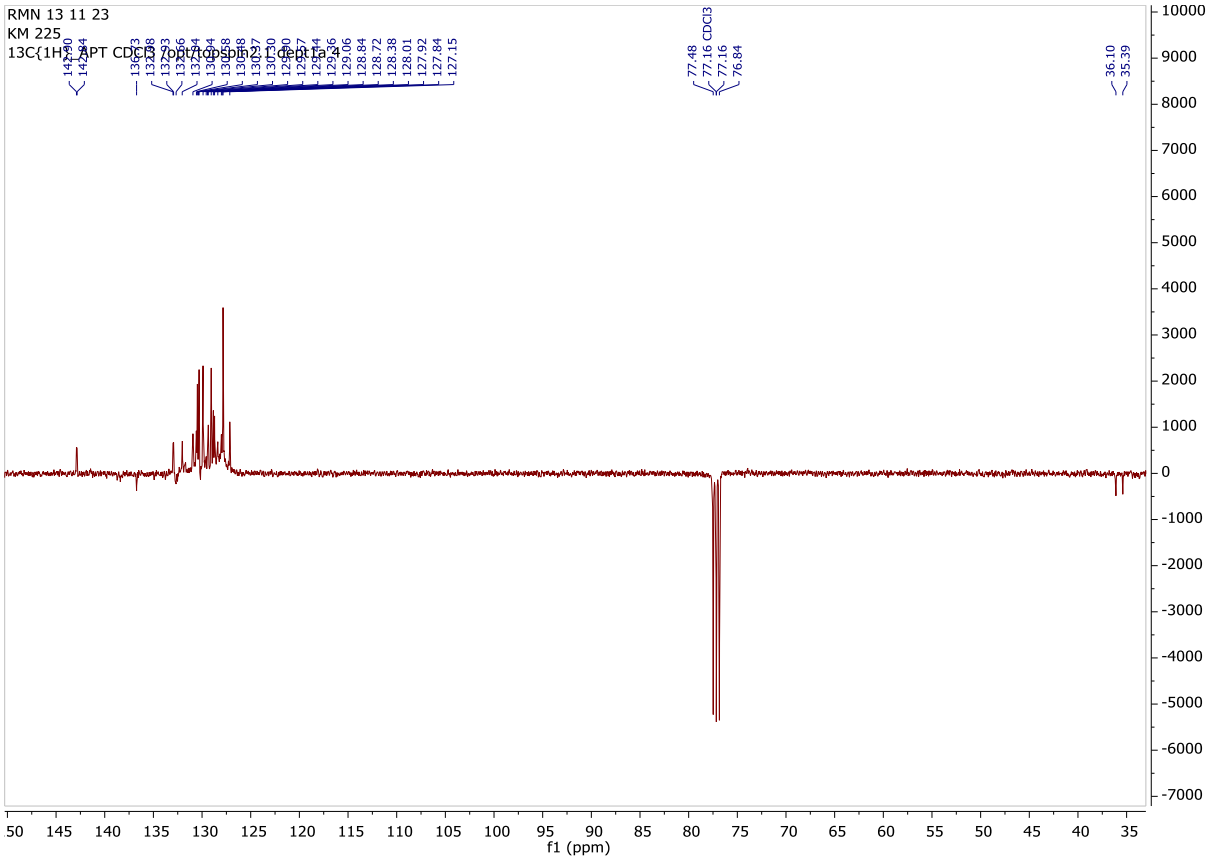
S2: (*Z*)-3-(1,2-Diphenylvinyl)-2-phenyl-1*H*-isophosphinoline 2-oxide **(3b)**

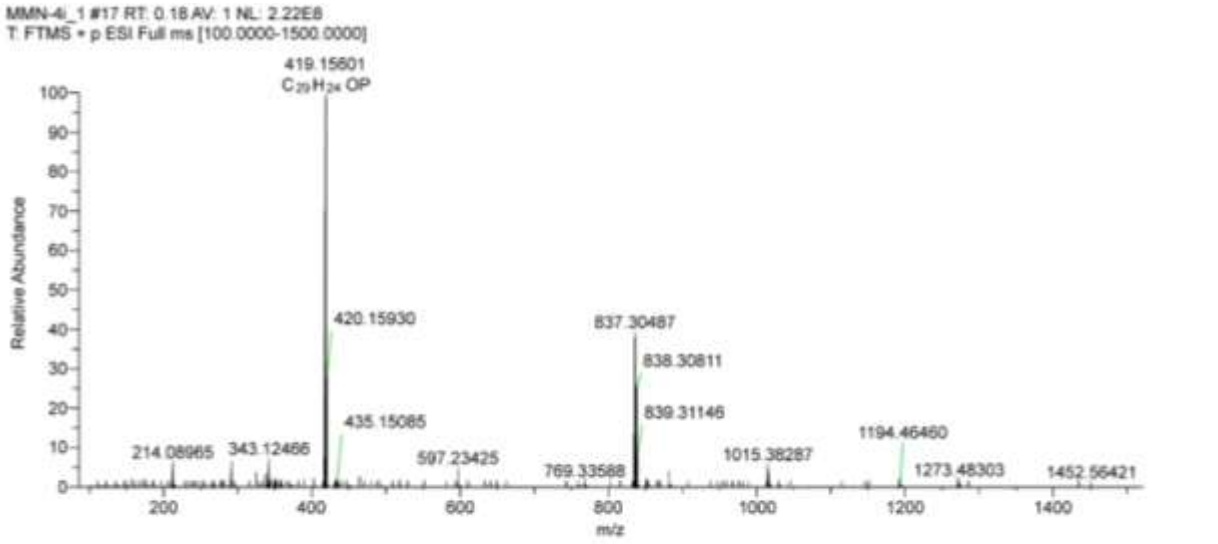



RMN KM 225 PUR
KM 225 PUR 2
1H CDCl3 /opt/topspin2.1 dept1a 12
7.80
7.78
7.77
7.77
7.77
7.75
7.74
7.34
7.33
7.26 CDCl3
7.25
7.00
6.99
6.74
6.73
6.72
6.71
3.80
3.75
3.74
3.70
3.46
3.43
3.42
3.39
21.03
1.03
0.95
f1 (ppm)


RMN 13 11 23
KM 225
1H CDCl3 /opt/topspin2.1 dept1a 14
CDCl3
23.63
1.00
0.88
f1 (ppm)

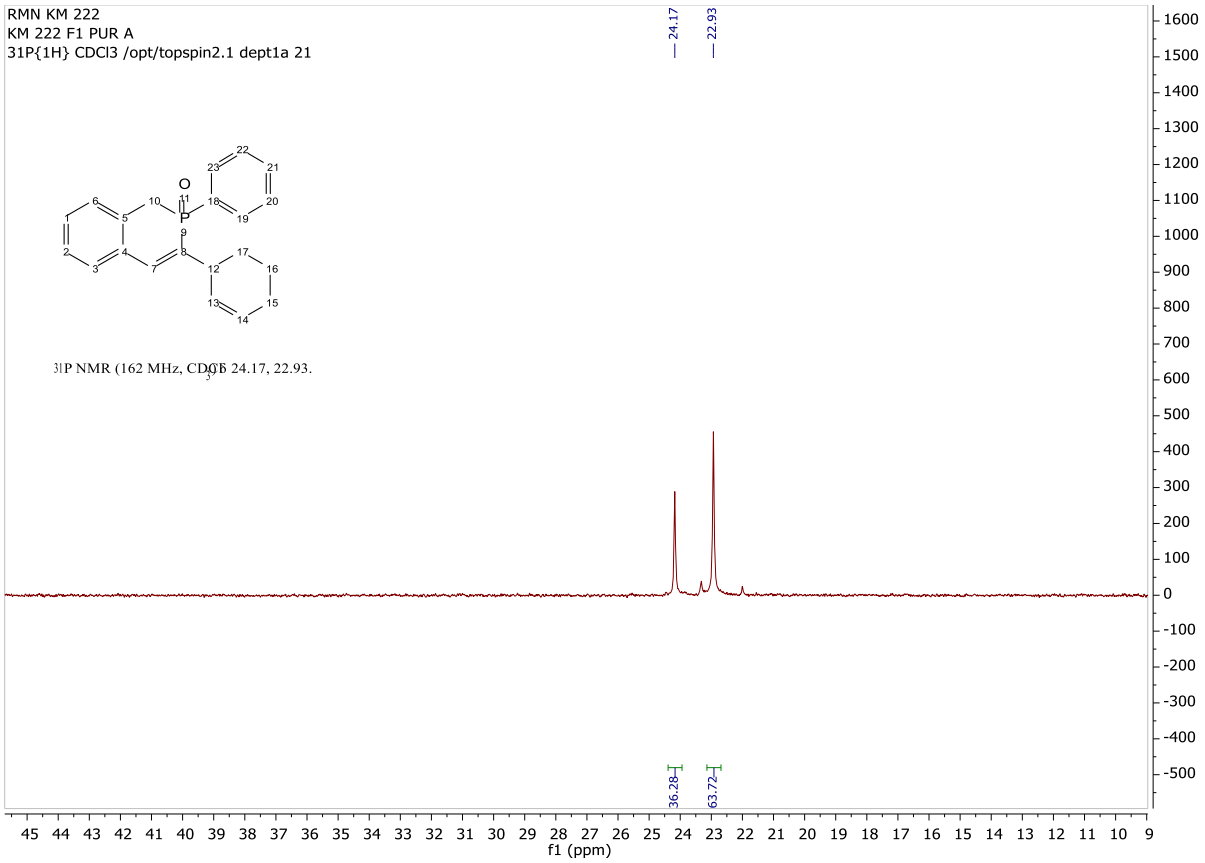

RMN 13 11 23
KM 225
1H{31P} CDCl3 /opt/topspin2.1 dept1a 4
7.79
7.77
7.44
7.42
7.40
7.38
7.36
7.35
7.34
7.34
7.32
7.31
7.30
7.27 CDCl3
7.26
7.22
7.21
7.20
7.18
7.16
7.15
7.14
7.13
7.12
7.11
7.10
7.09
7.08
7.01
7.00
6.99
6.99
6.90
6.75
6.74
6.73
6.72
3.78
3.74
3.46
3.42
f1 (ppm)


RMN 13 11 23
KM 225
13C{1H} APT CDCl3 /opt/topspin2.1 dept1a 4
142.90
142.84
136.23
132.88
132.23
132.06
132.04
130.74
130.68
130.27
130.10
129.00
128.97
128.64
129.46
129.06
128.84
128.72
128.38
128.01
127.92
127.84
127.15
77.48
77.16 CDCl3
77.16
76.84
36.10
35.39
f1 (ppm)

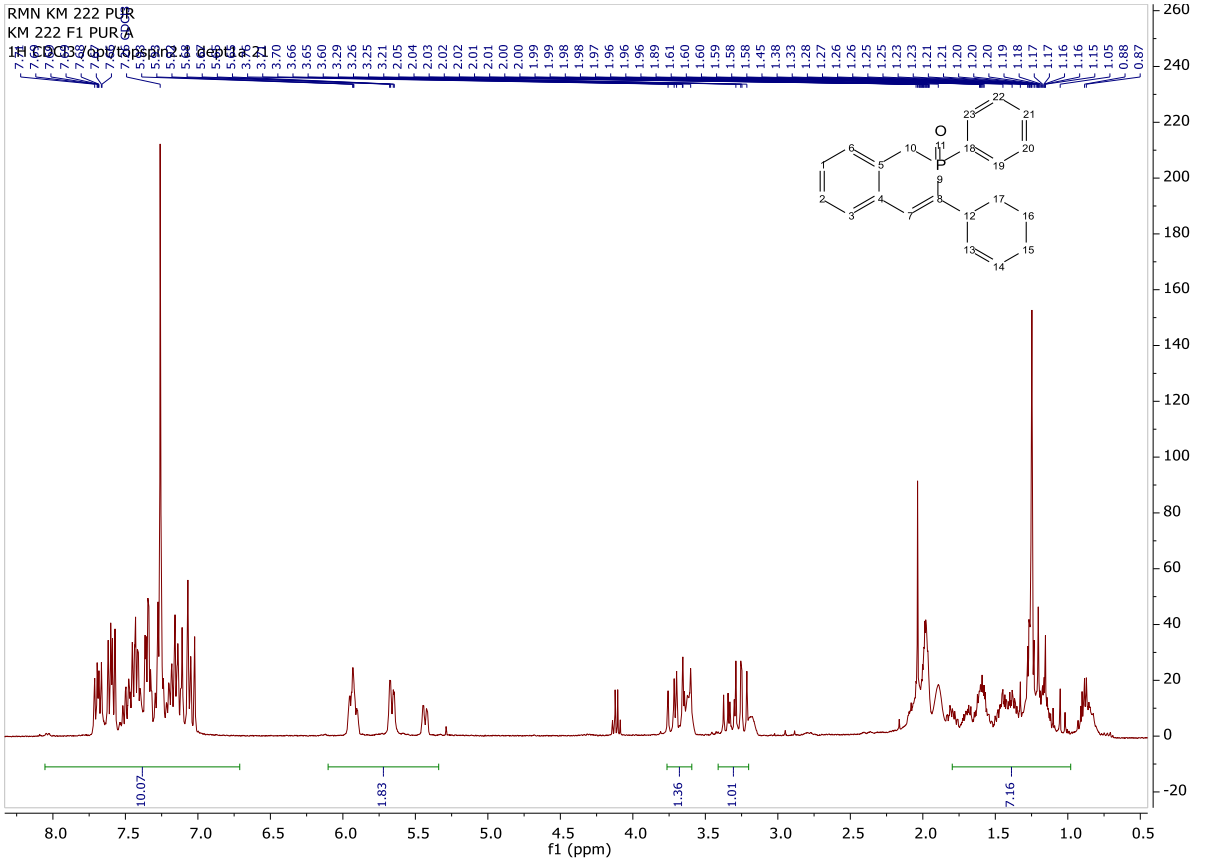


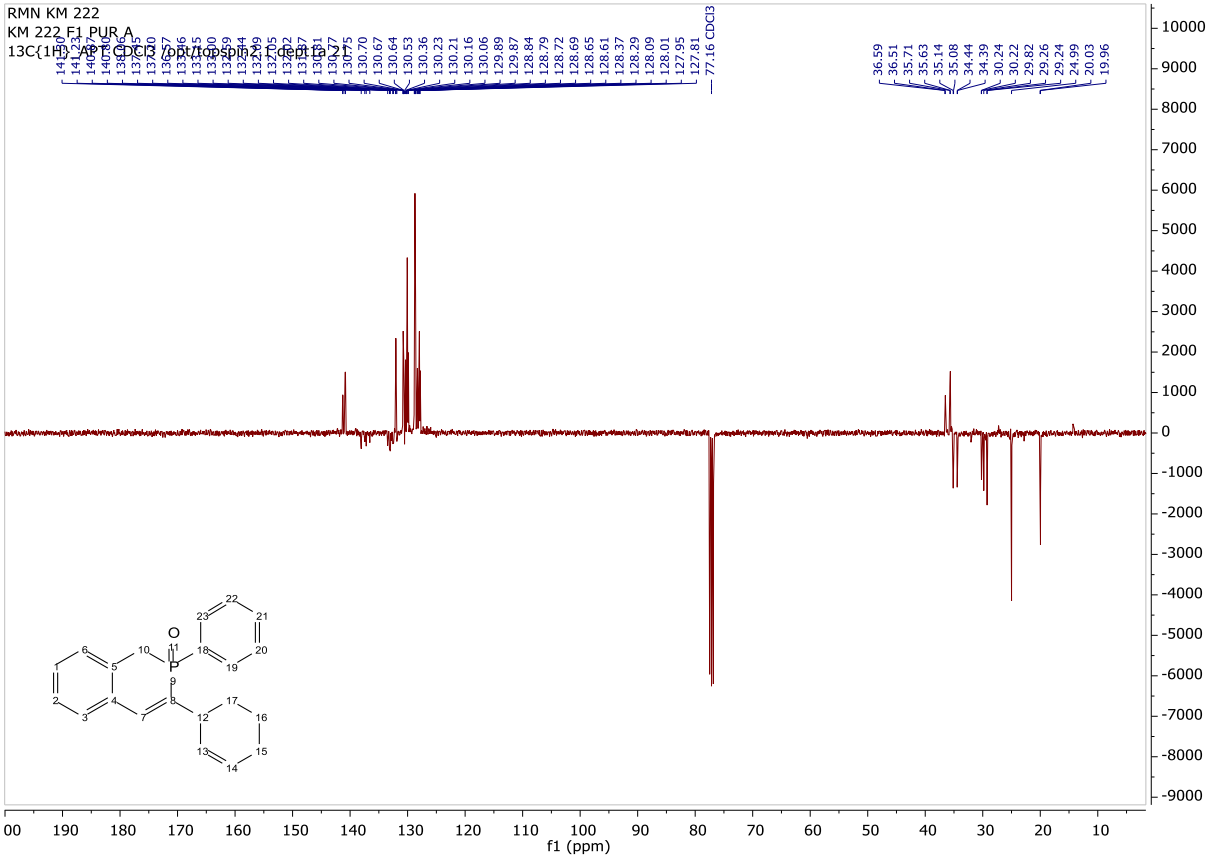
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	419.15601	$C_{29}H_{24}OP$	0.18	419.15593	99.66	4	96.81	99.83	(Collection)
2	419.15601	$C_{28}H_{19}O_2N_{2+}$	-0.27	419.15612	79.18	4	71.81	75.33	(Collection)
3	419.15601	$C_{28}H_{23}O_2N_4$	-0.29	419.15613	79.12	3	71.74	75.33	(Collection)

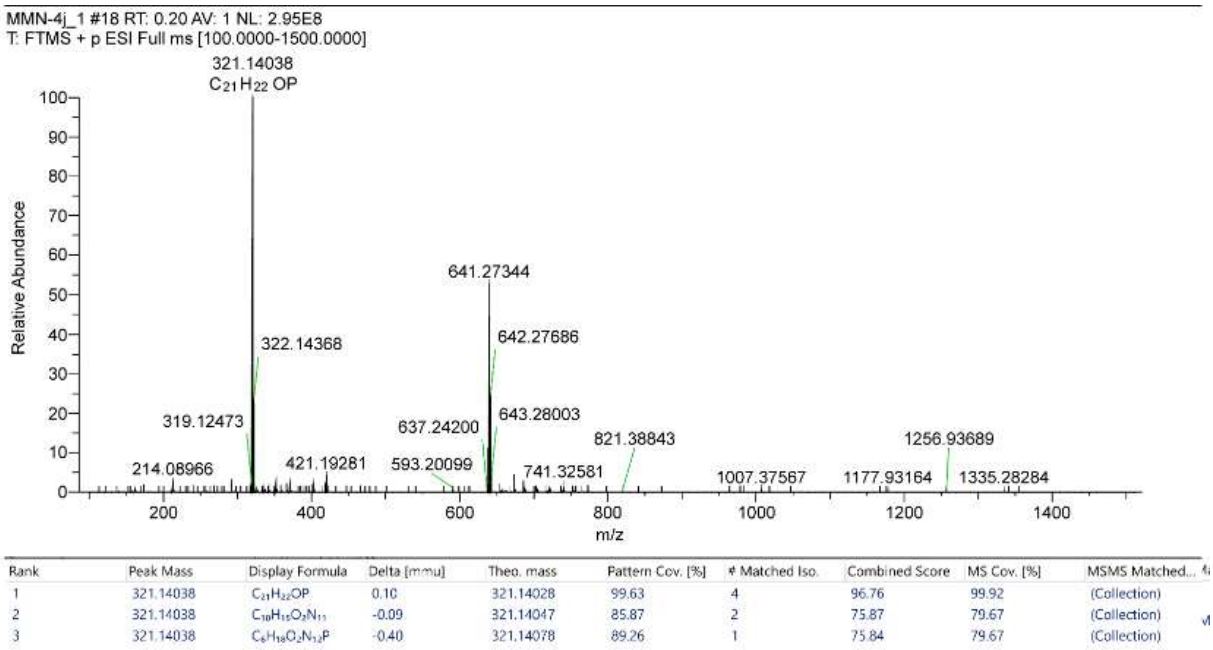
S3: 3-(Cyclohex-2-en-1-yl)-2-phenyl-1*H*-isophosphinoline 2-oxide **(3c)**



31P NMR (162 MHz, $CDCl_3$) δ 24.17, 22.93.

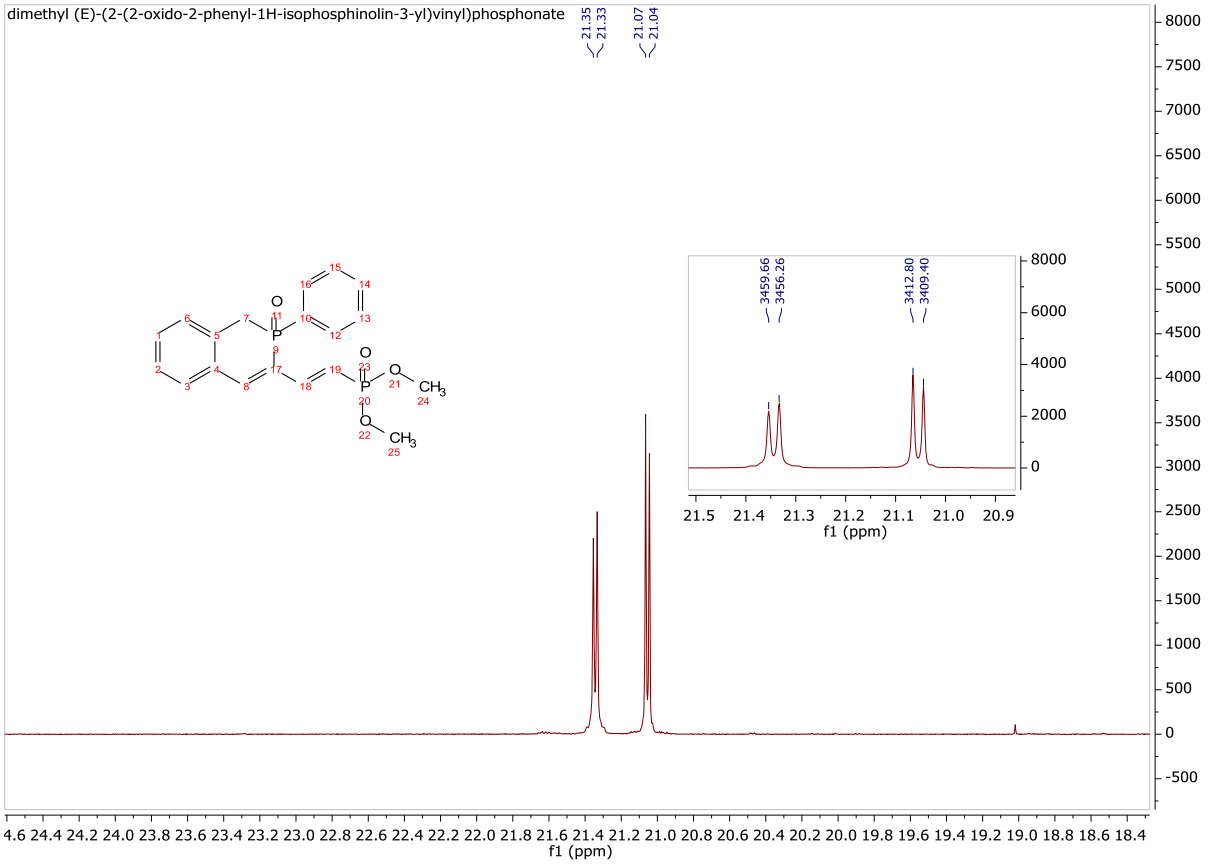


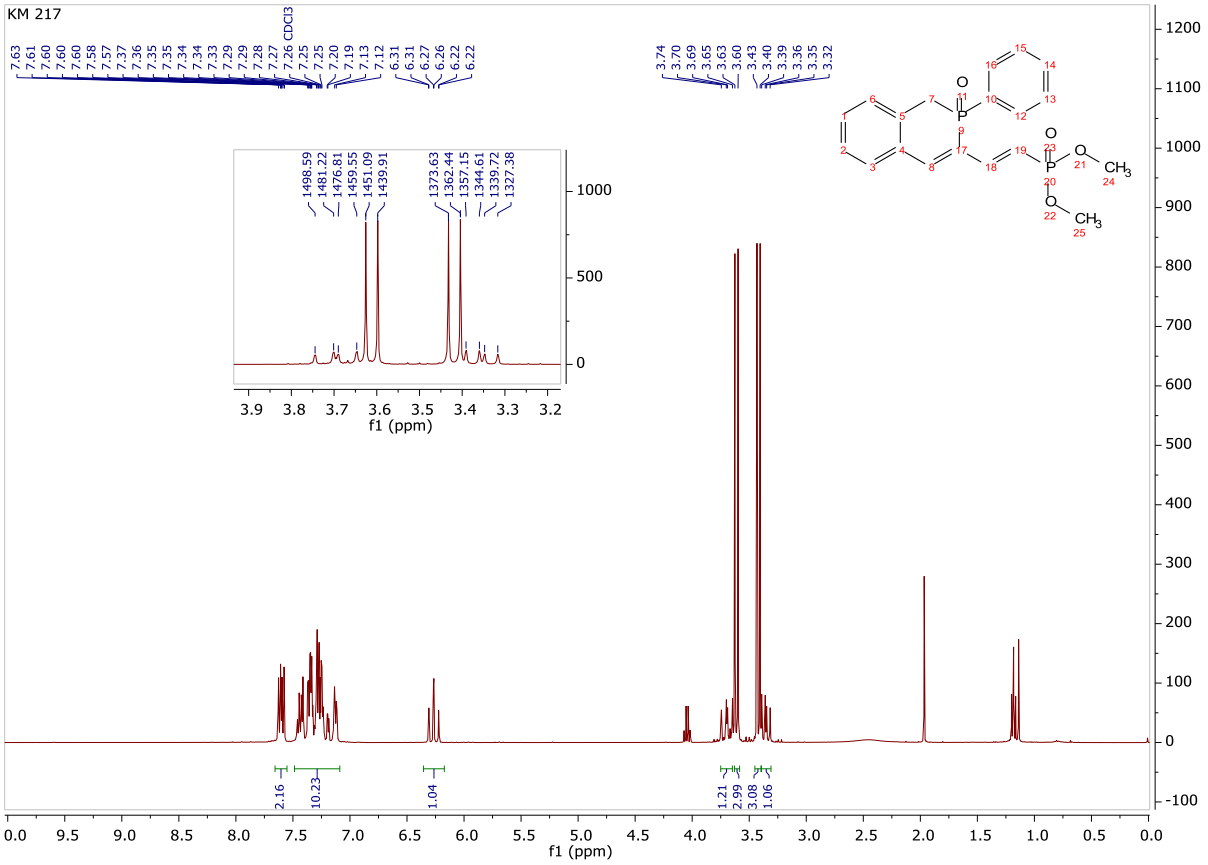


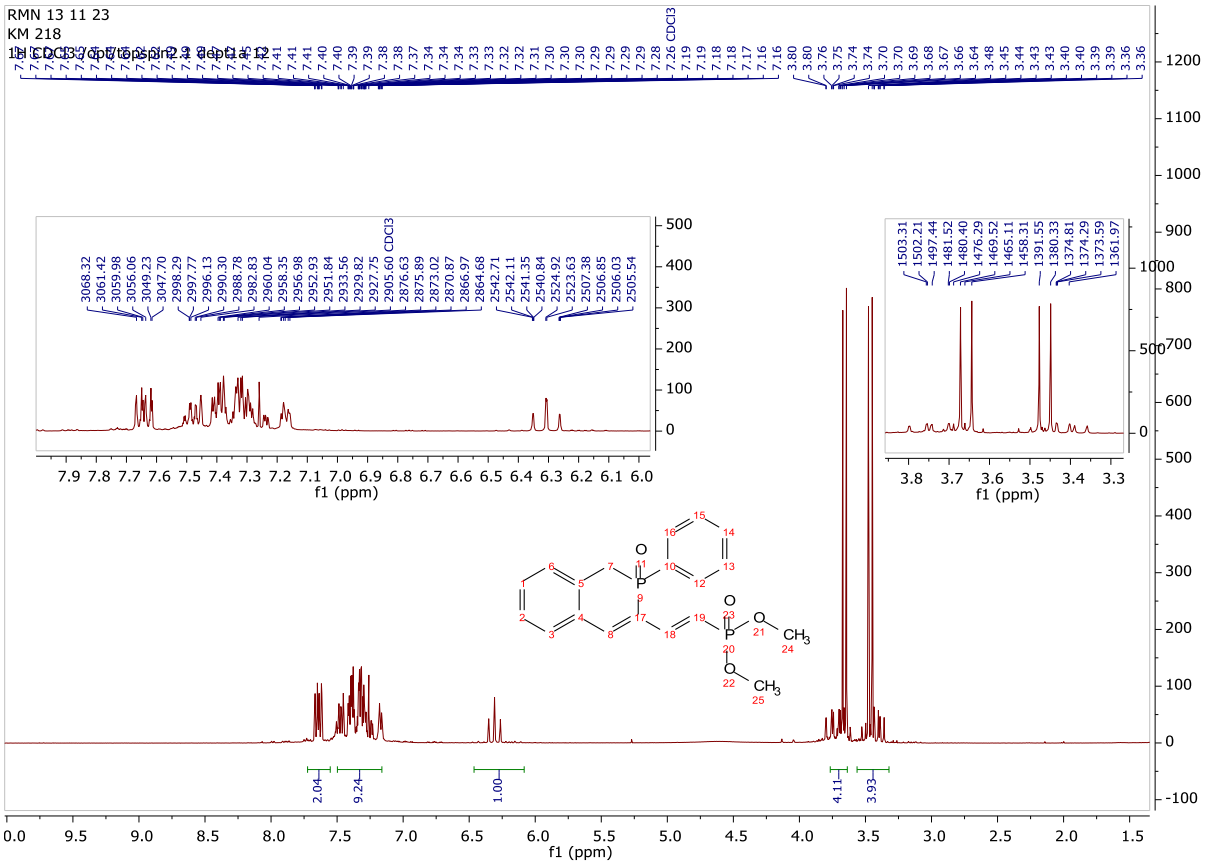


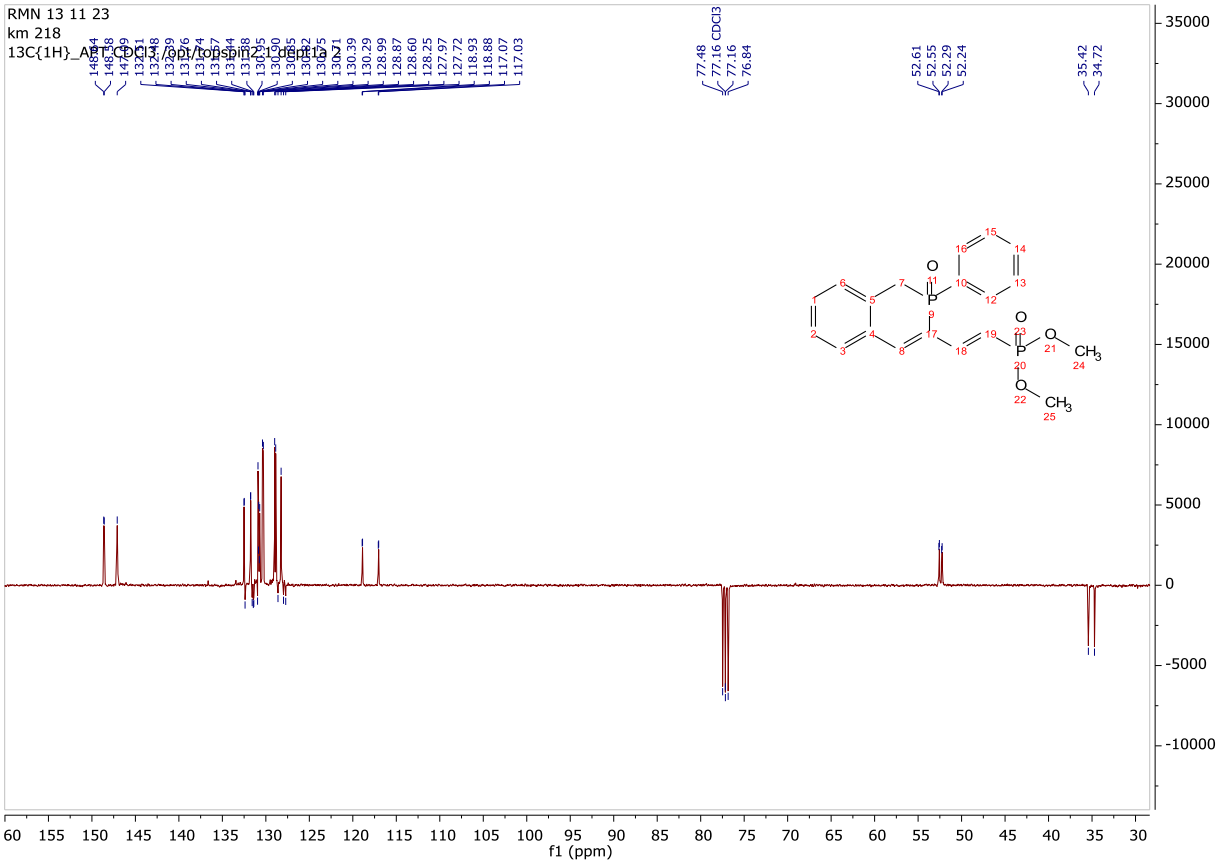
Rank	Peak Mass	Display Formula	Delta [mmu]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	321.14038	$C_{21}H_{22}OP$	0.10	321.14028	99.63	4	96.76	99.92	(Collection)
2	321.14038	$C_{19}H_{15}O_2N_{11}$	-0.09	321.14047	85.87	2	75.87	79.67	(Collection)
3	321.14038	$C_6H_{18}O_2N_{12}P$	-0.40	321.14078	89.26	1	75.84	79.67	(Collection)

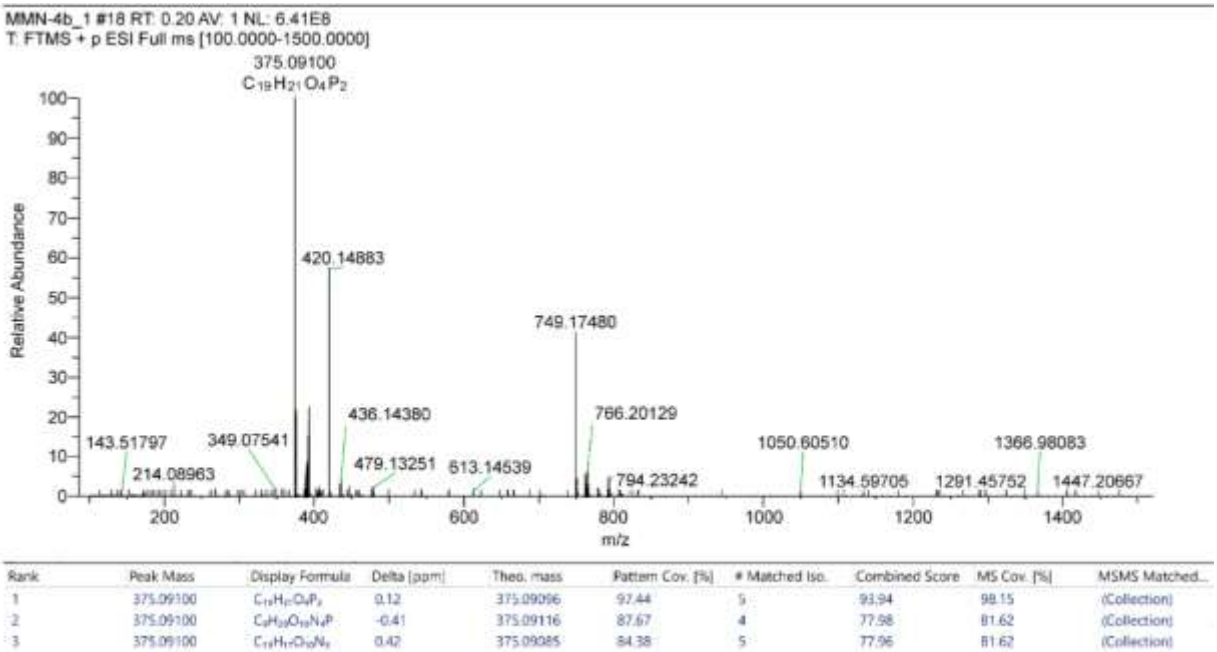
S4: Dimethyl (*E*)-(2-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)vinyl)phosphonate **(3d)**




KM 217
f1 (ppm)

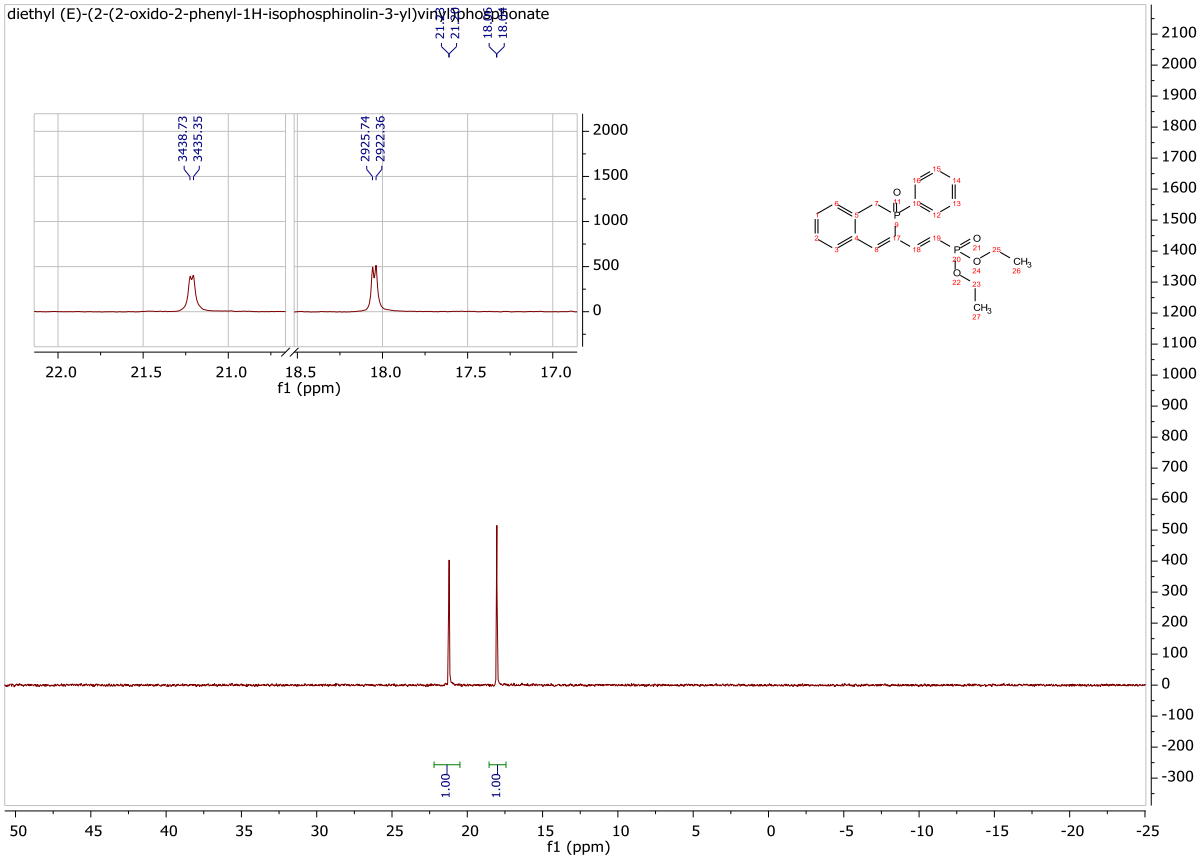

RMN 13 11 23
KM 218
f1 (ppm)

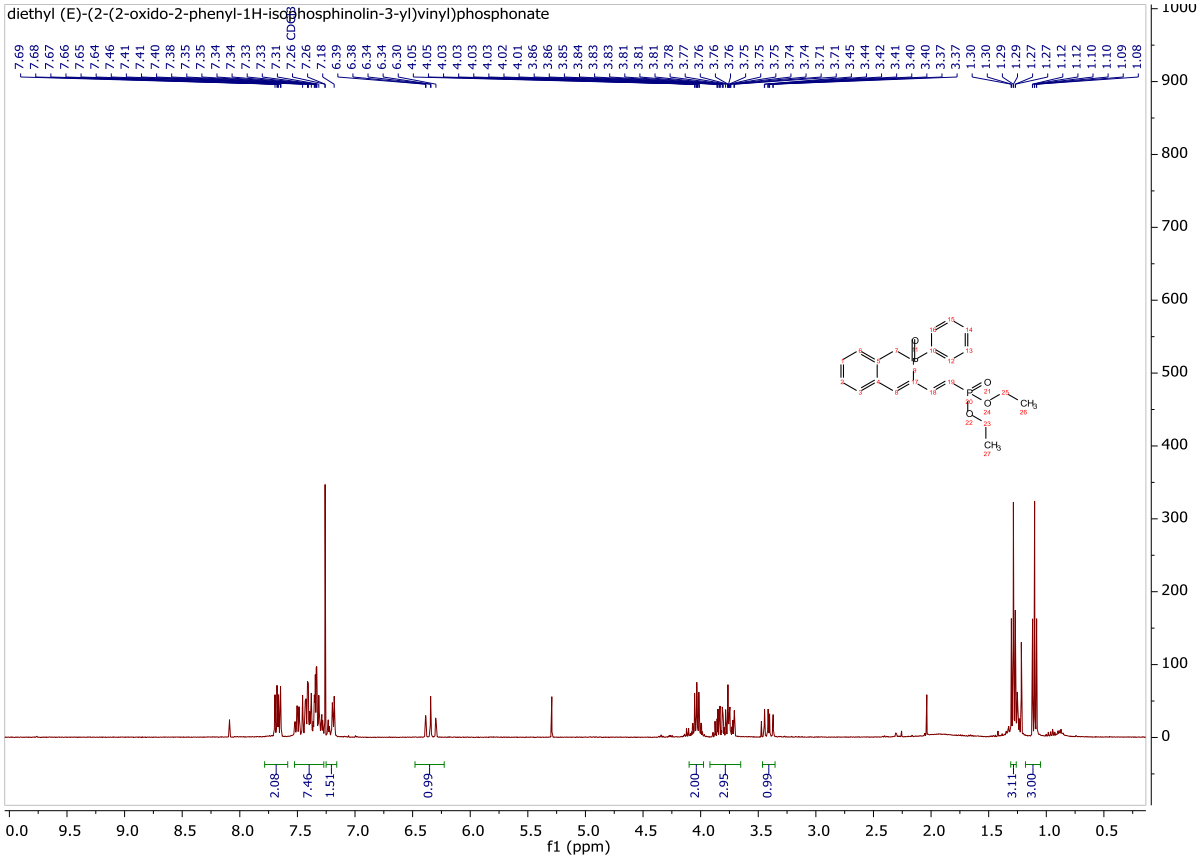


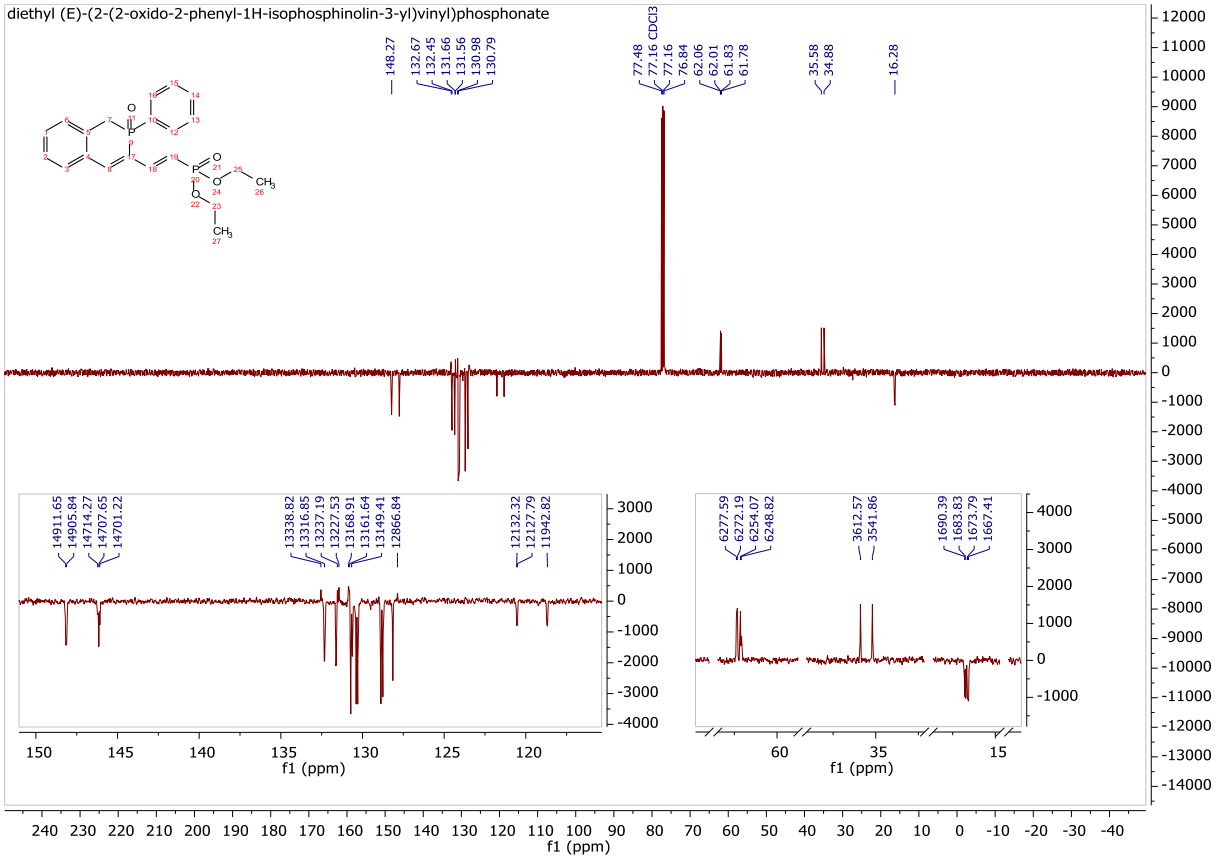


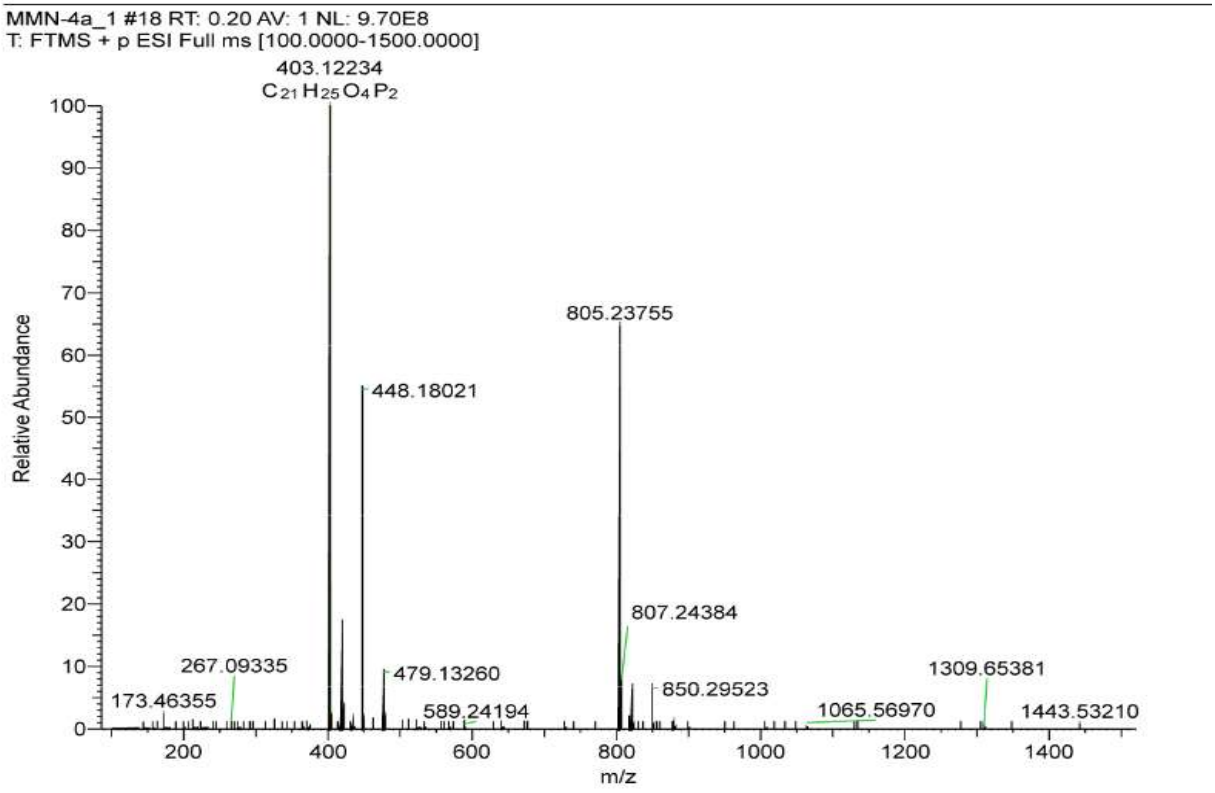
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	375.09100	$C_{19}H_{21}O_4P_2$	0.12	375.09096	97.44	5	93.94	98.15	(Collection)
2	375.09100	$C_{24}H_{20}O_{10}N_4P$	-0.41	375.09116	87.67	4	77.98	81.62	(Collection)
3	375.09100	$C_{19}H_{17}O_{10}N_4$	0.42	375.09085	84.38	5	77.96	81.62	(Collection)

S5: Diethyl (*E*)-(2-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)vinyl)phosphonate **(3e)**



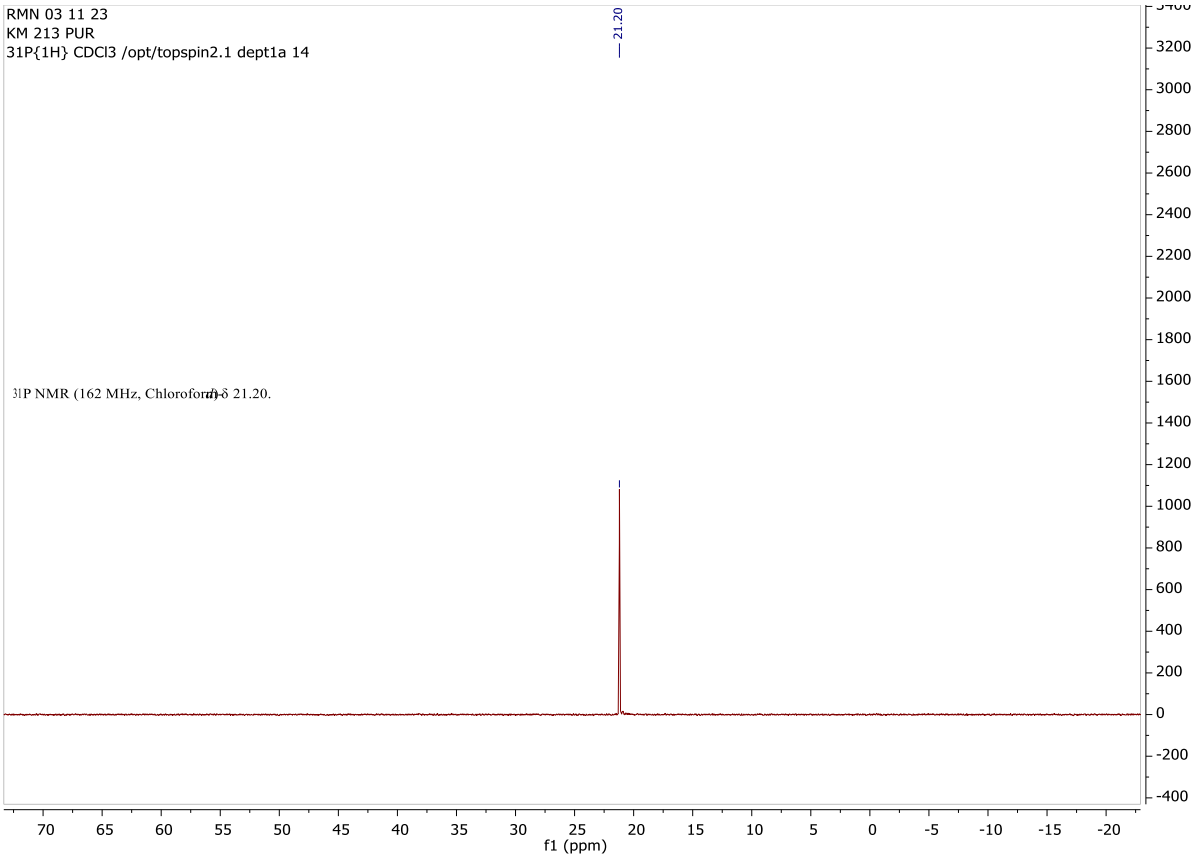


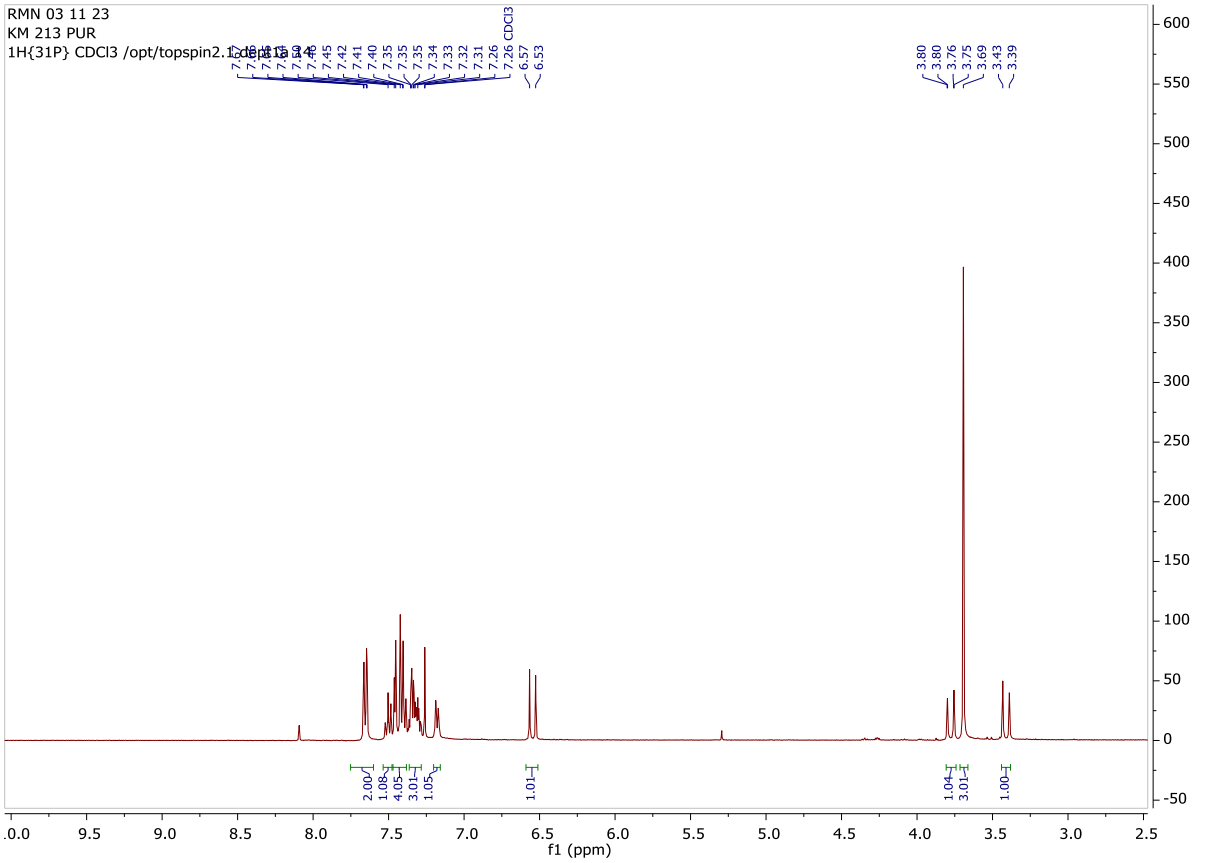


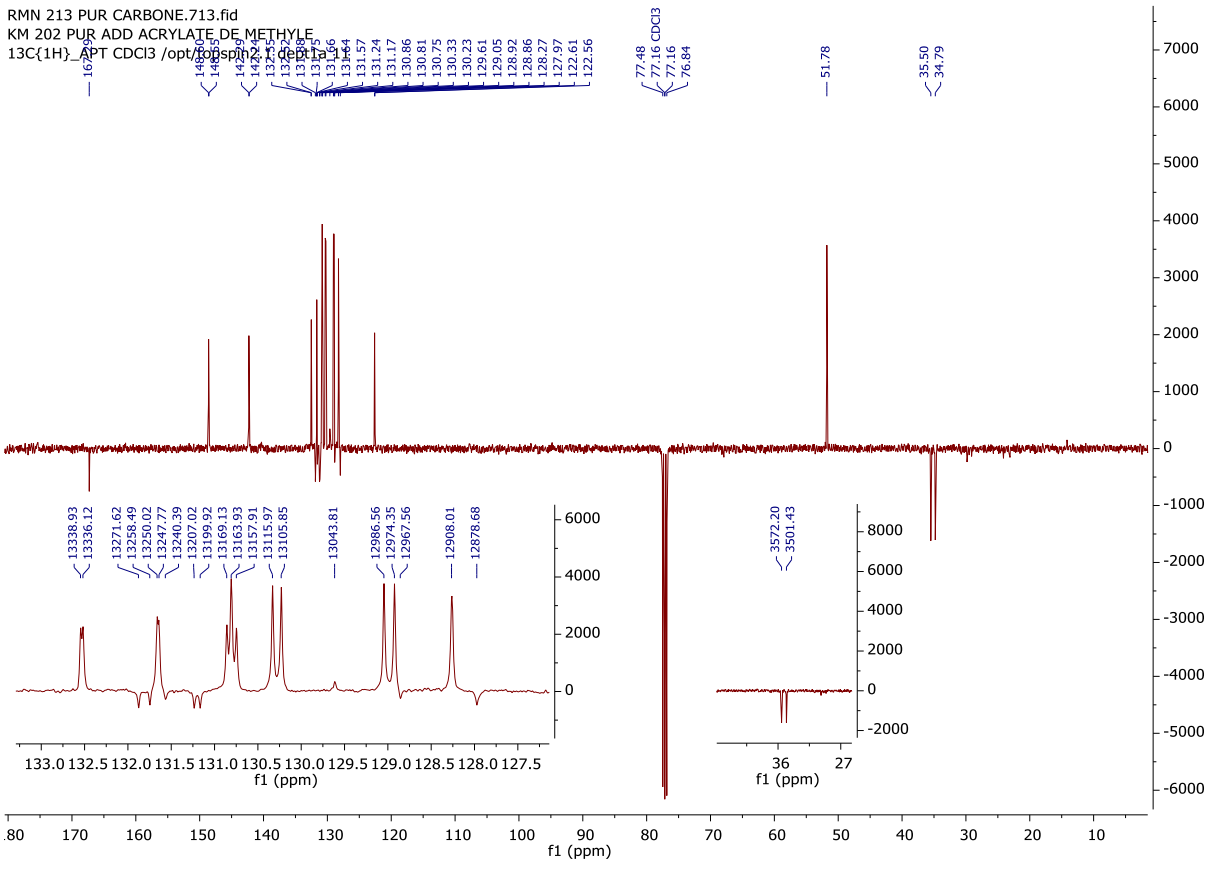


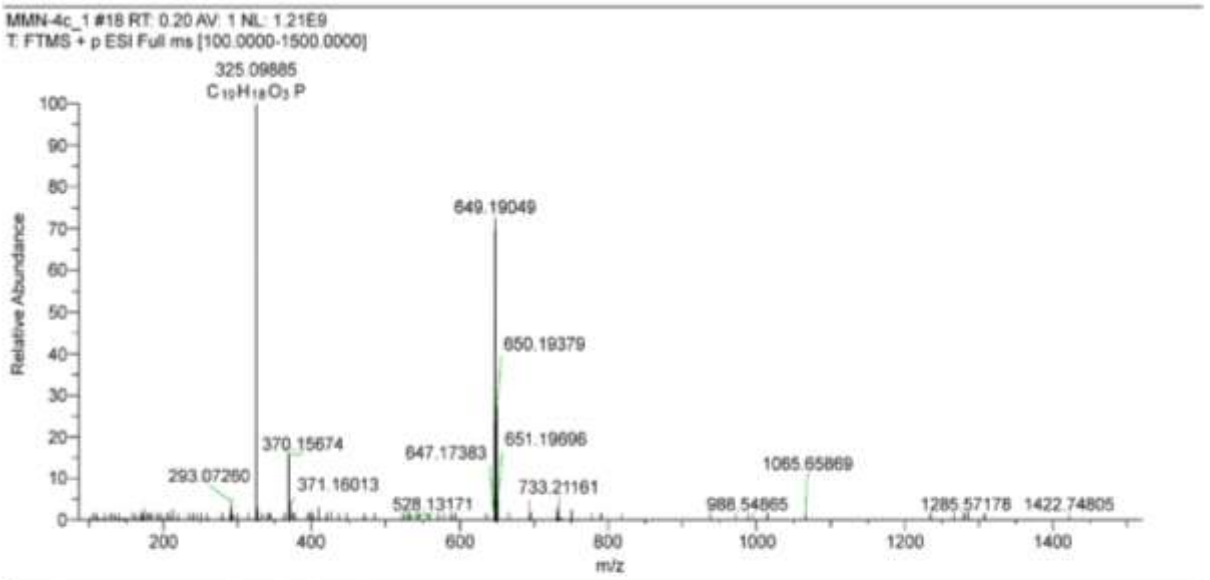
Rank	Peak Mass	Display For...	Delta [mmu]	Theo. mass	Pattern Cov....	# Matched I...	Combined S...	MS Cov. [%]	MSMS Matc...
1	403.12234	$C_{21}H_{25}O_4P_2$	0.09	403.12226	97.72	3	93.9	97.93	(Collection)
2	403.12234	$C_{15}H_{21}O_{10}N_3$	0.20	403.12215	95.86	4	93.46	97.93	(Collection)
3	403.12234	$C_6H_{21}O_5N_{12}P_2$	-0.42	403.12276	88.99	2	77.79	81.58	(Collection)

S6: Methyl (*E*)-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylate **(3f)**



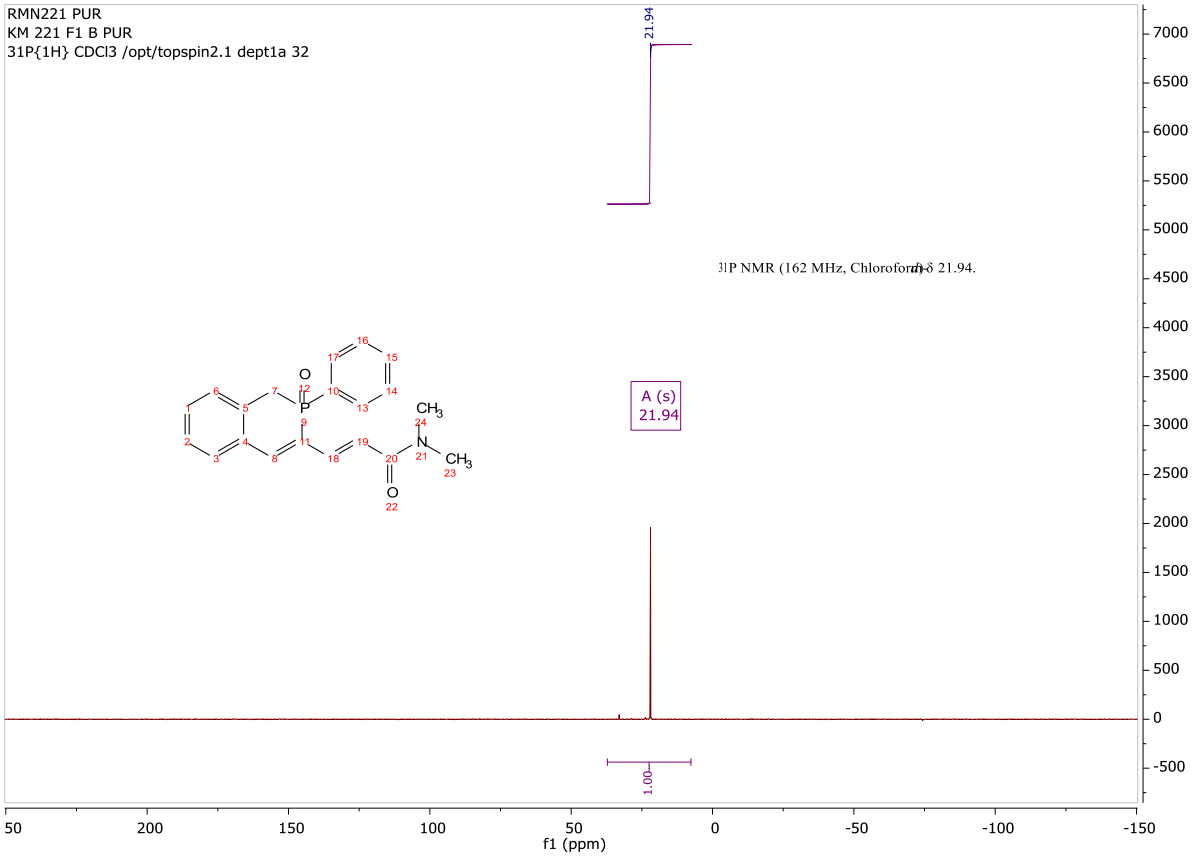

RMN 03 11 23
KM 213 PUR
1H{31P} CDCl3 /opt/topspin2.1_dept13.14
f1 (ppm)


RMN 213 PUR CARBONE.713.fid
KM 202 PUR ADD ACRYLATE DE METHYLE
13C{1H}_APT CDCl3 /opt/topspin2.1_dept13.14
f1 (ppm)

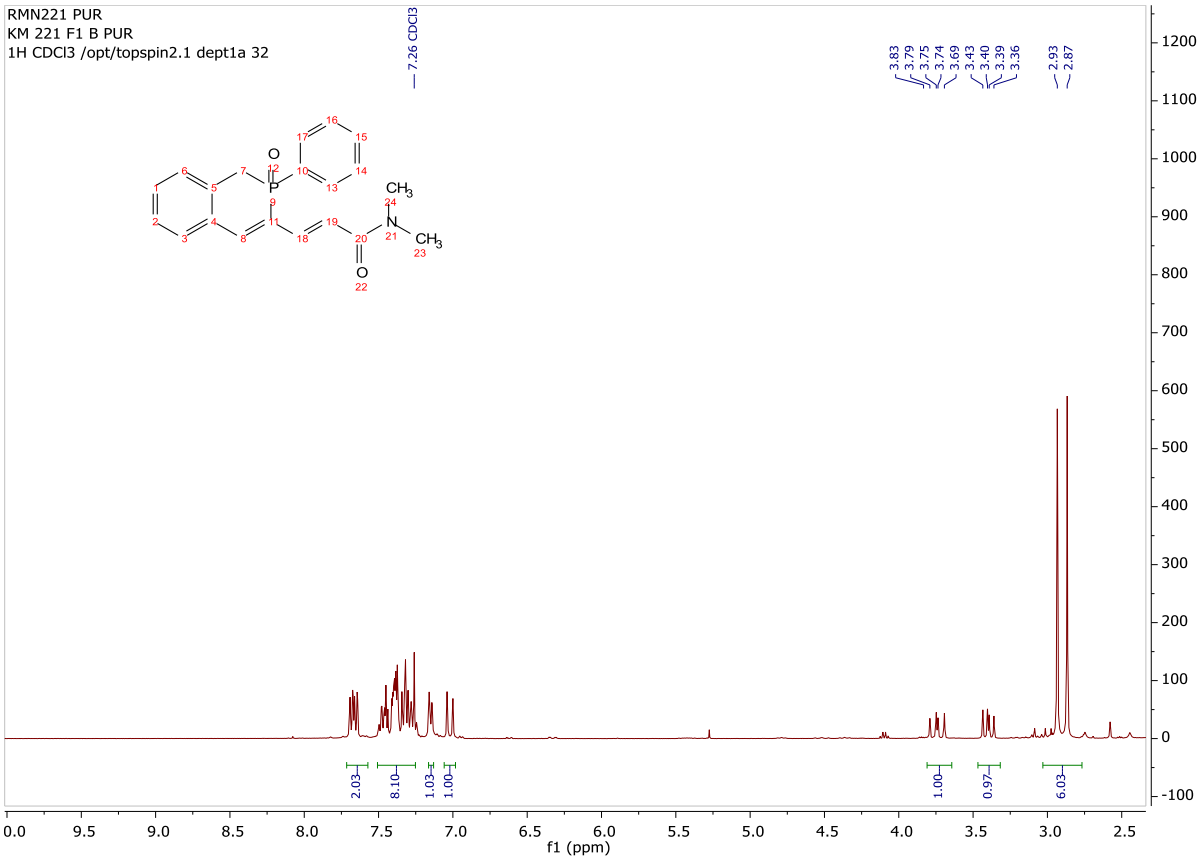


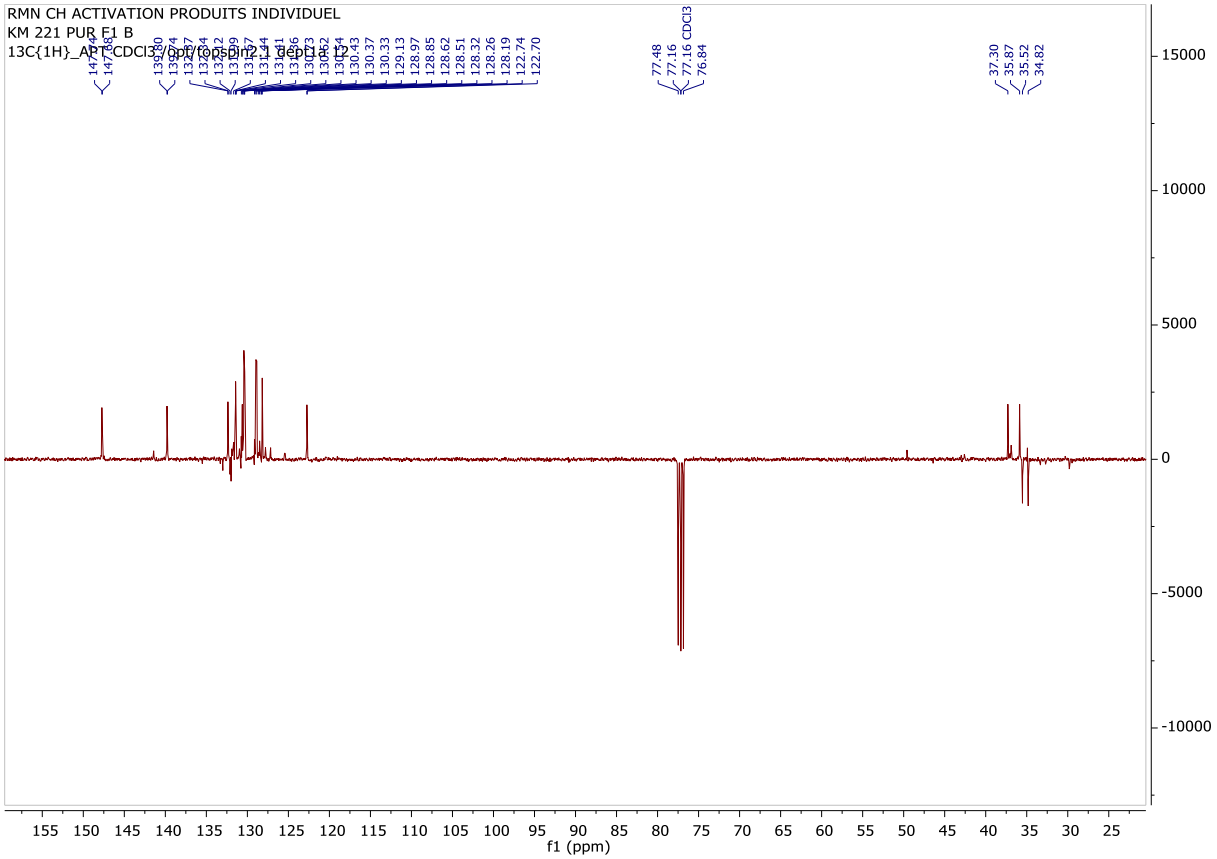
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	325.09885	$C_{19}H_{18}O_3P$	0.12	325.09881	99.13	5	95.6	99.58	(Collection)
2	325.09885	$C_9H_{15}O_4N_4$	-0.49	325.09900	87.75	4	77.85	81.52	(Collection)
3	325.09885	$C_8H_{13}O_5N_4$	-0.47	325.09900	87.4	2	77.44	81.38	(Collection)

S7: (*E*)-*N*,*N*-Dimethyl-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylamide **(3g)**



^{31}P NMR (162 MHz, Chloroform-*d*) δ 21.94.

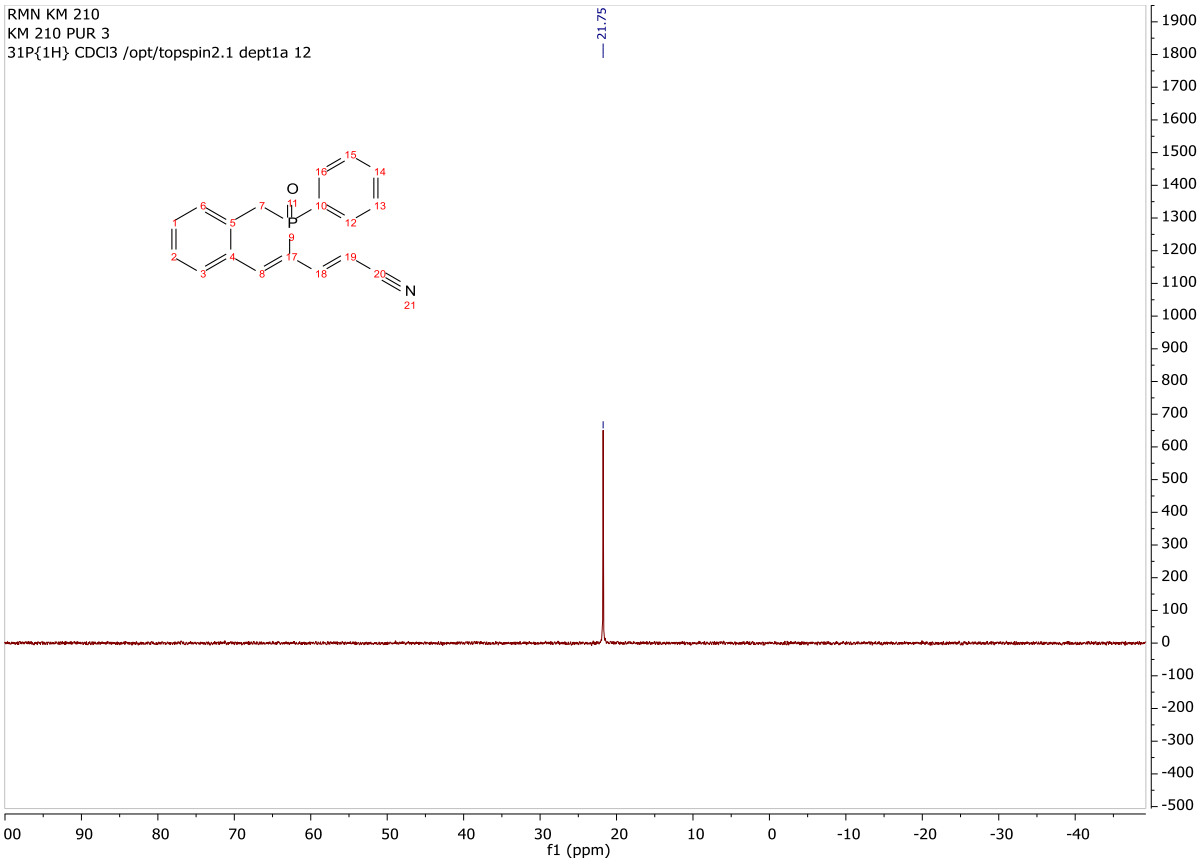


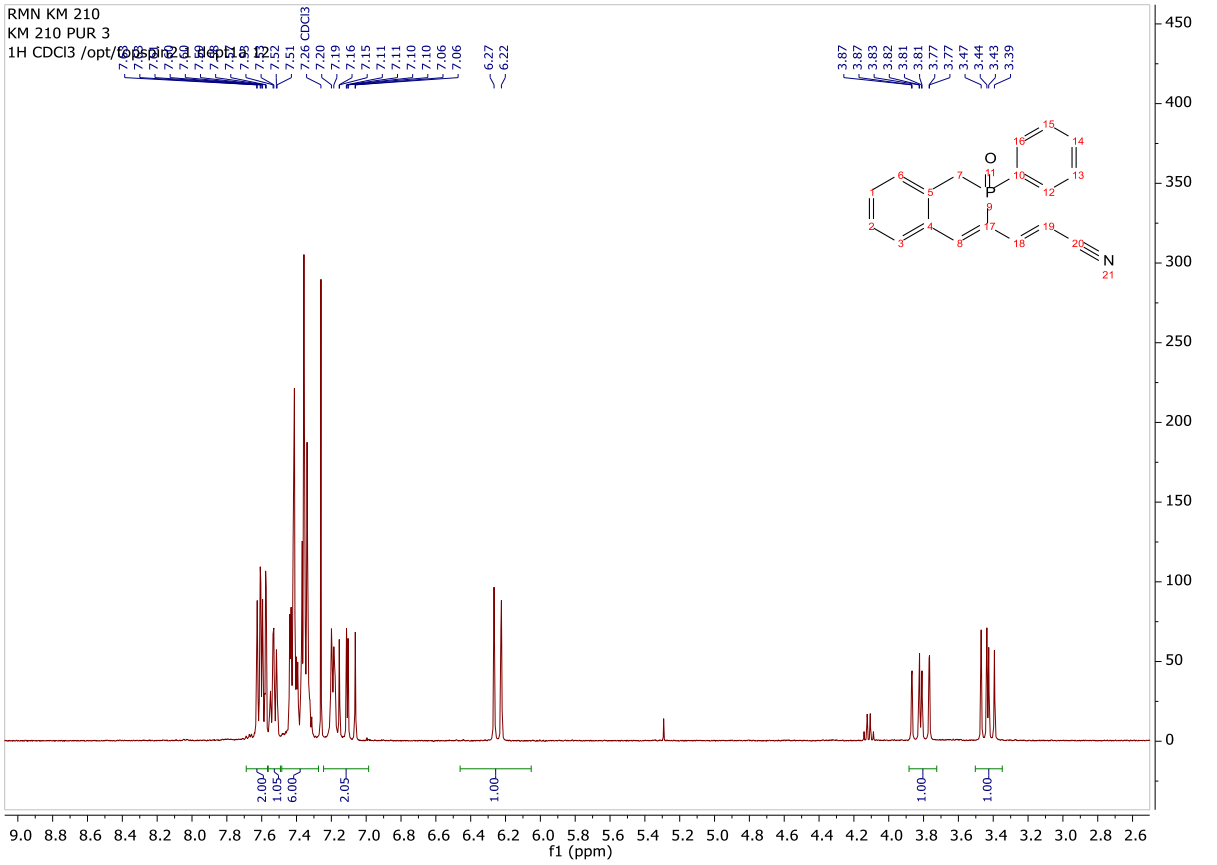


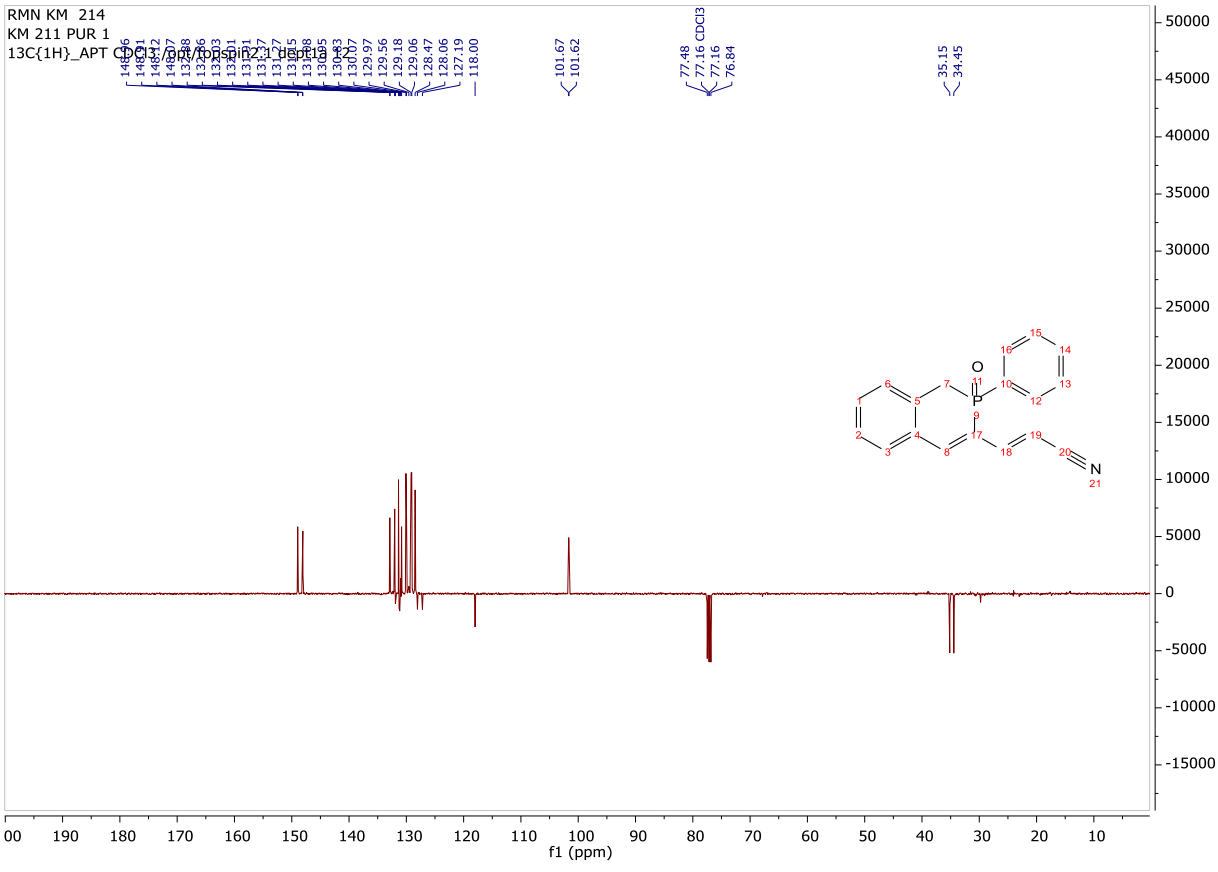


Rank	Peak Mass	Display Formula	Delta (ppm)	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	338.13052	$C_{20}H_{21}O_2NP$	0.24	338.13044	99.36	4	95.72	99.26	(Collection)
2	338.13052	$C_{24}H_{19}O_2N_2$	-0.33	338.13063	86.31	3	77.47	81.38	(Collection)
3	338.13052	$C_{19}H_{18}O_4N_2$	-0.34	338.13064	86.5	3	77.42	81.38	(Collection)

S8: (*E*)-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylonitrile **(3h)**



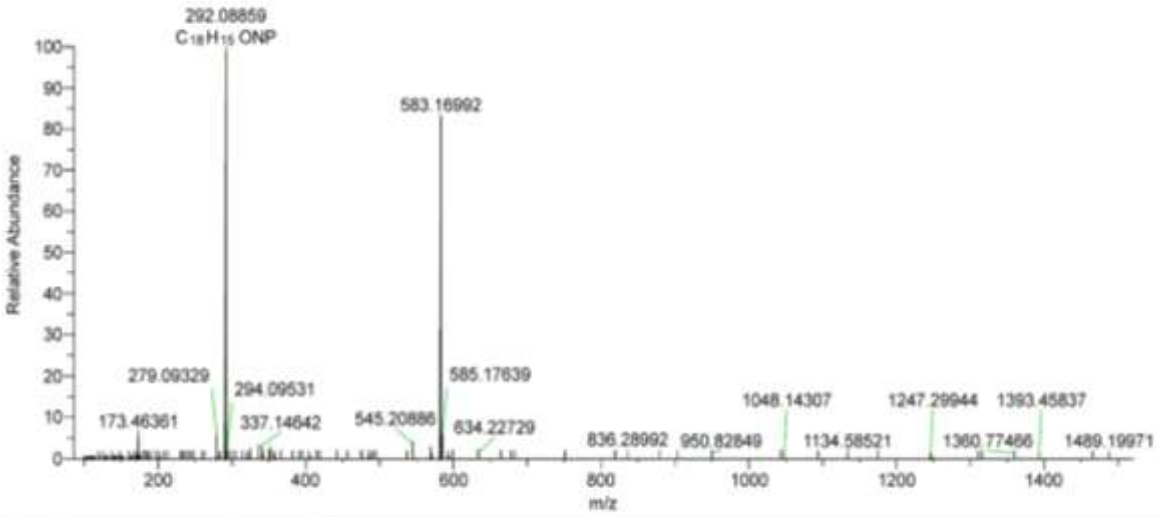

RMN KM 210
KM 210 PUR 3
1H CDCl3 /opt/topspin2.1_beptia_12
7.26 CDCl3
6.27
6.22
3.87
3.87
3.83
3.82
3.81
3.81
3.77
3.77
3.47
3.44
3.43
3.39
2.00
1.05
6.00
2.05
1.00
1.00
1.00
f1 (ppm)


RMN KM 214
KM 211 PUR 1
13C{1H}_APT CDCl3
101.67
101.62
77.48
77.16 CDCl3
77.16
76.84
35.15
34.45
f1 (ppm)

D:\LMP\OLAF\2023\data\S2328\MMN-4f_1.raw 7/17/2023 4:24:23 PM

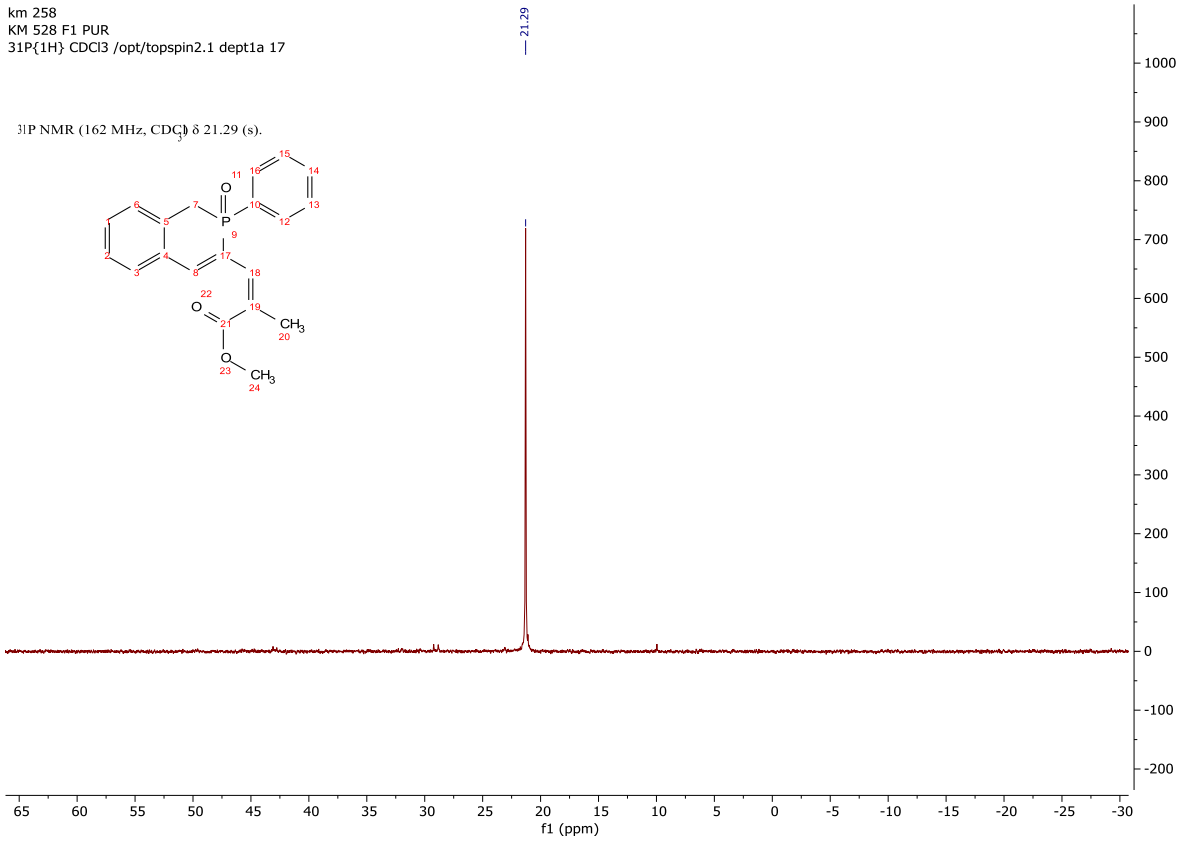
Code OLAF MMN-4f_1.raw **Echantillon** KM212 **Instrument** Orbitrap ID-X

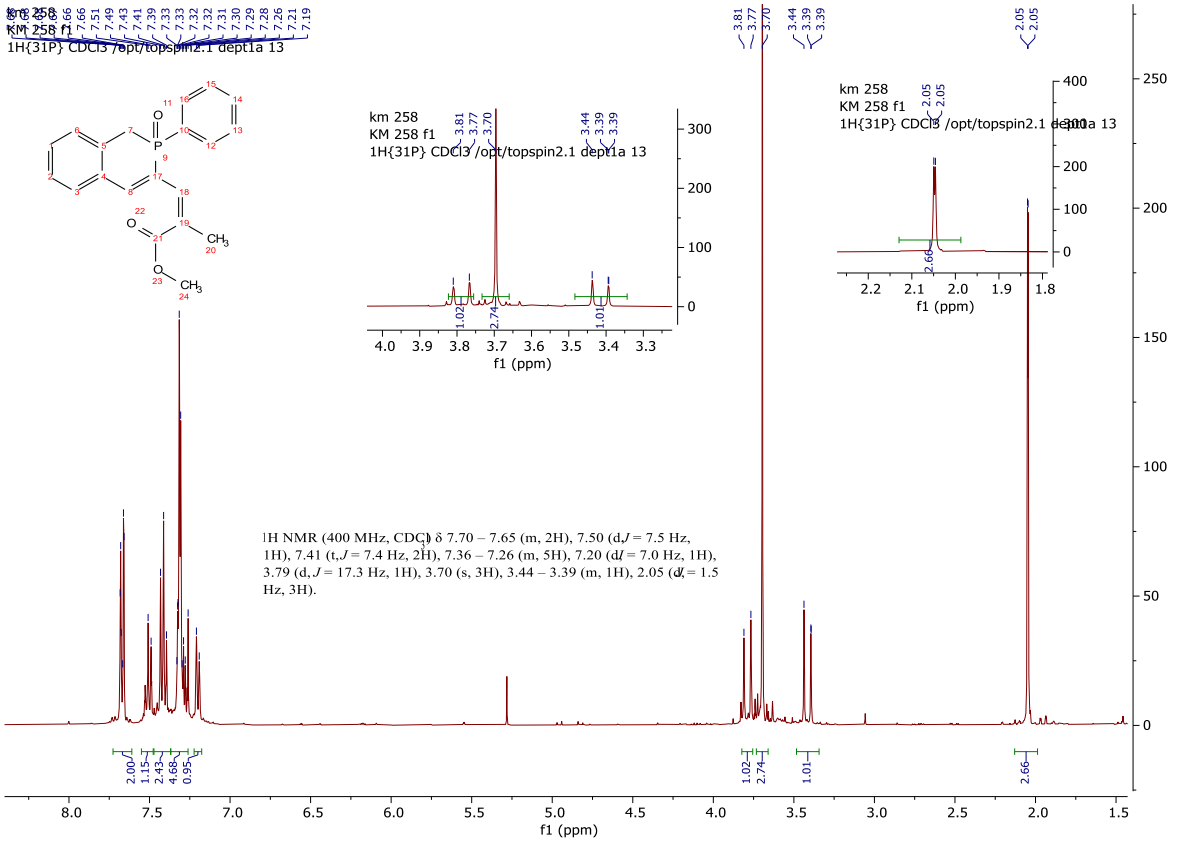
MMN-4f_1 #19 RT: 0.21 AV: 1 NL: 1.31E9
T: FTMS + p ESI Full ms [100.0000-1500.0000]



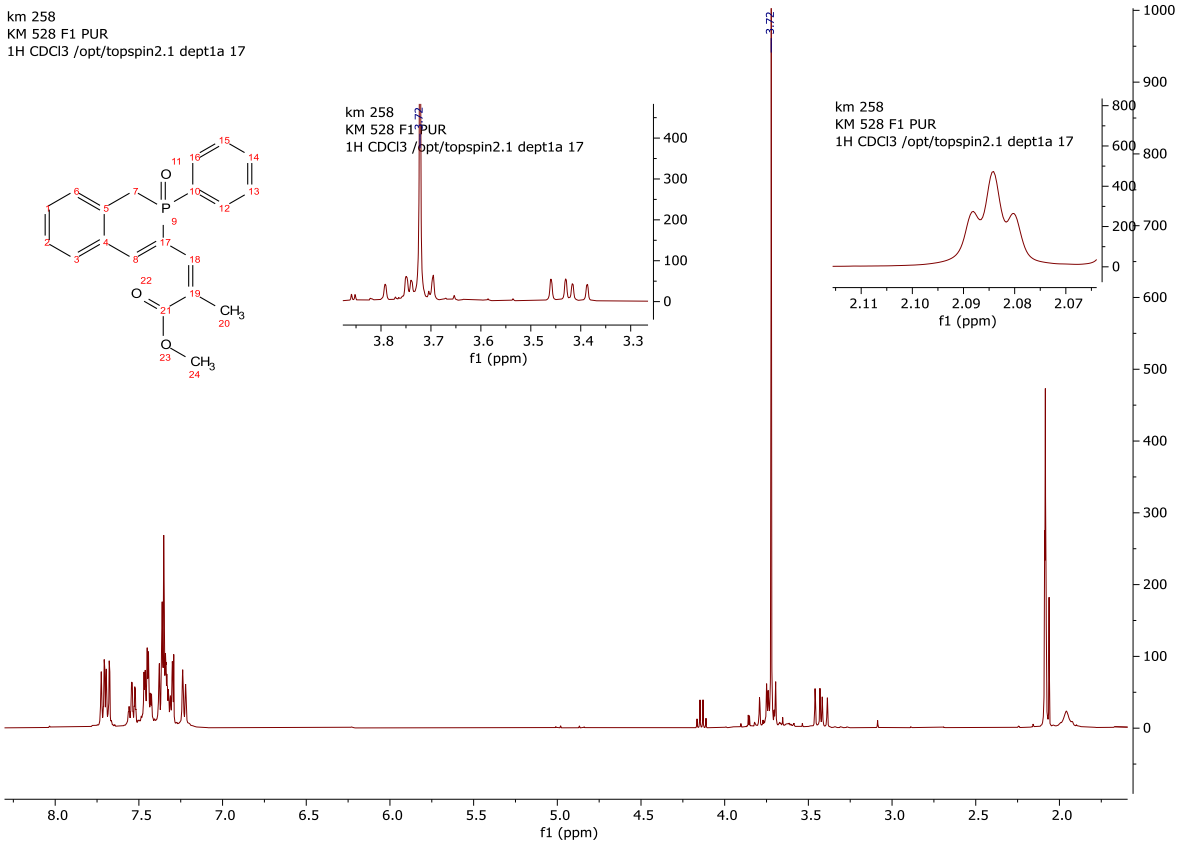
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	292.08859	$C_{18}H_{15}ONP$	0.05	292.08858	99.7	3	96.39	99.51	(Collection)
2	292.08859	$C_{17}H_{11}O_2N_2$	-0.60	292.08877	88.68	1	78.12	81.99	(Collection)
3	292.08859	$C_{16}H_{11}O_3N_2$	-0.62	292.08877	89.01	1	78.11	81.99	(Collection)

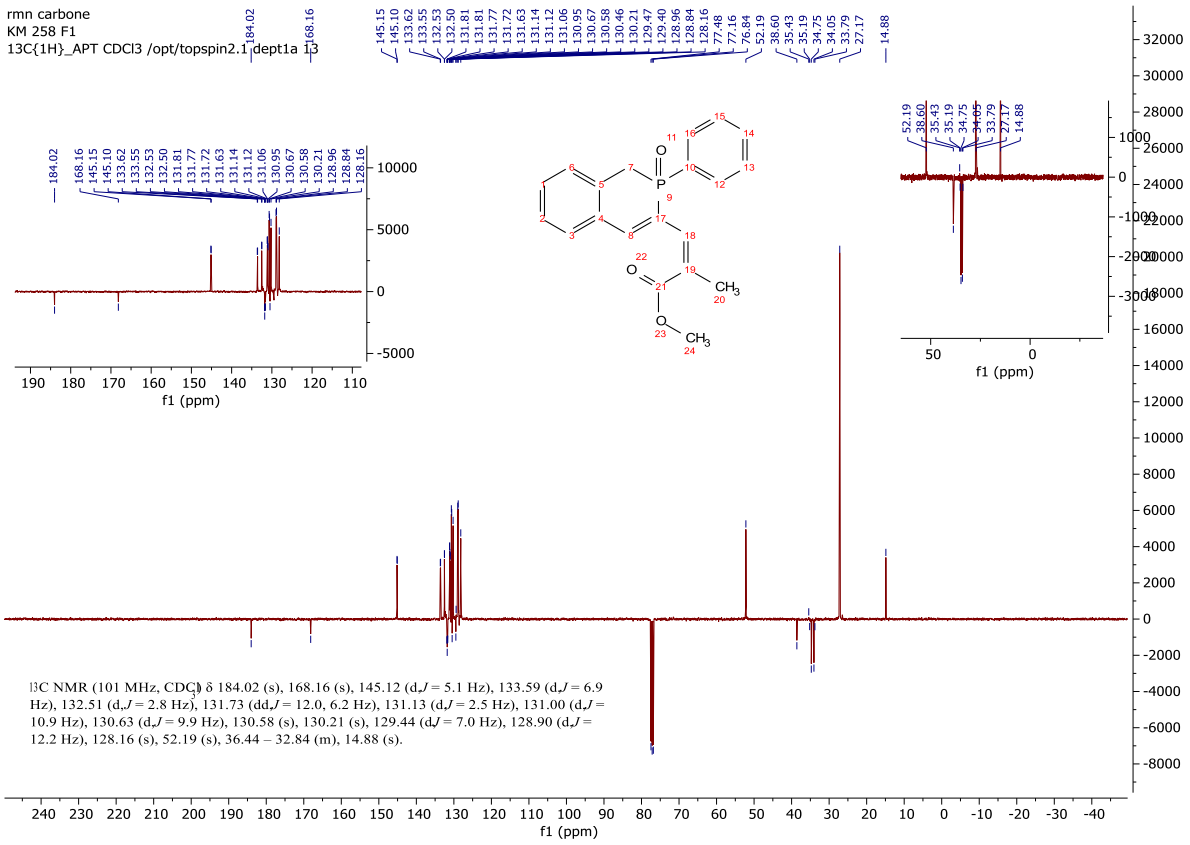
S9: Methyl 2-methyl-3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)acrylate **(3ia)**



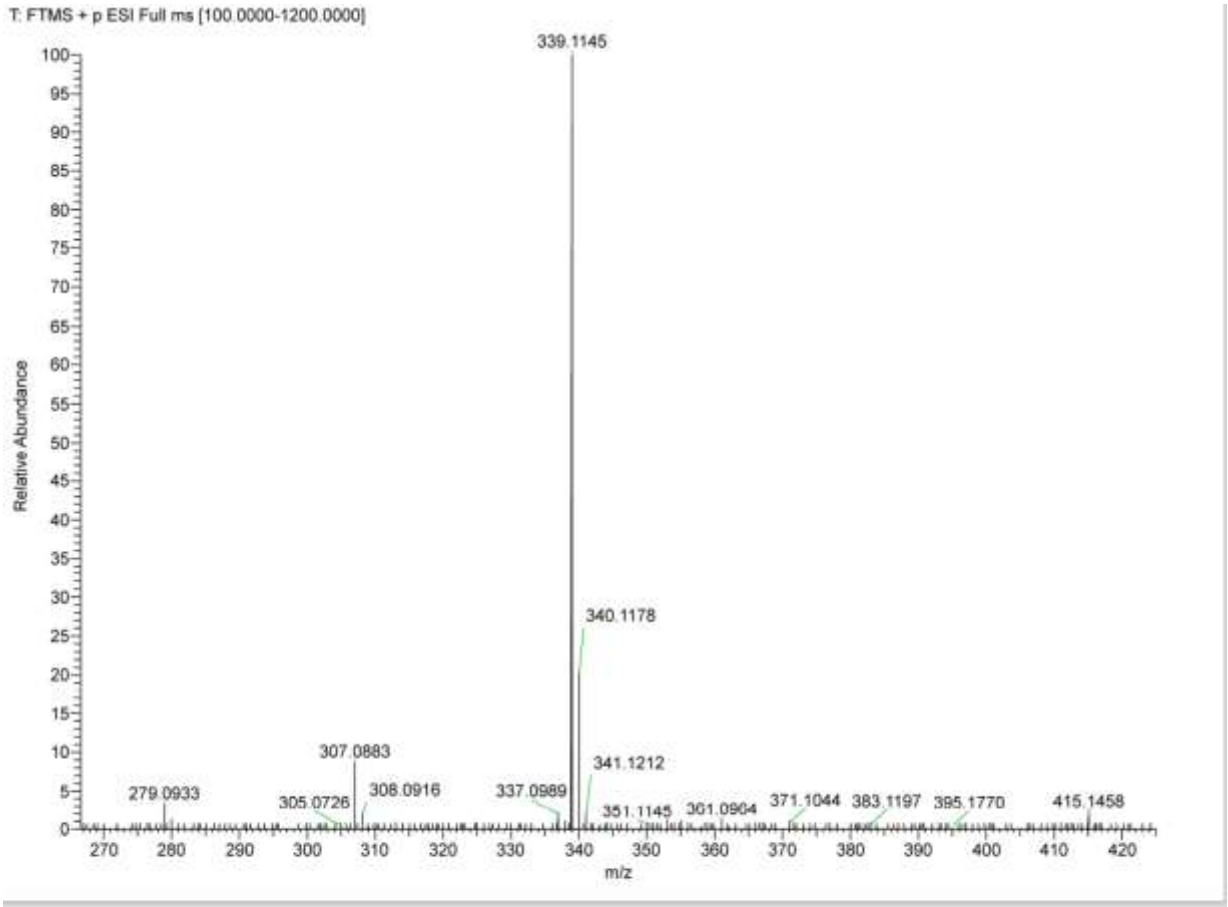


^{1}H NMR (400 MHz, CDCl$_3$) δ 7.70 – 7.65 (m, 2H), 7.50 (d, J = 7.5 Hz, 1H), 7.41 (t, J = 7.4 Hz, 2H), 7.36 – 7.26 (m, 5H), 7.20 (d, J = 7.0 Hz, 1H), 3.79 (d, J = 17.3 Hz, 1H), 3.70 (s, 3H), 3.44 – 3.39 (m, 1H), 2.05 (d, J = 1.5 Hz, 3H).





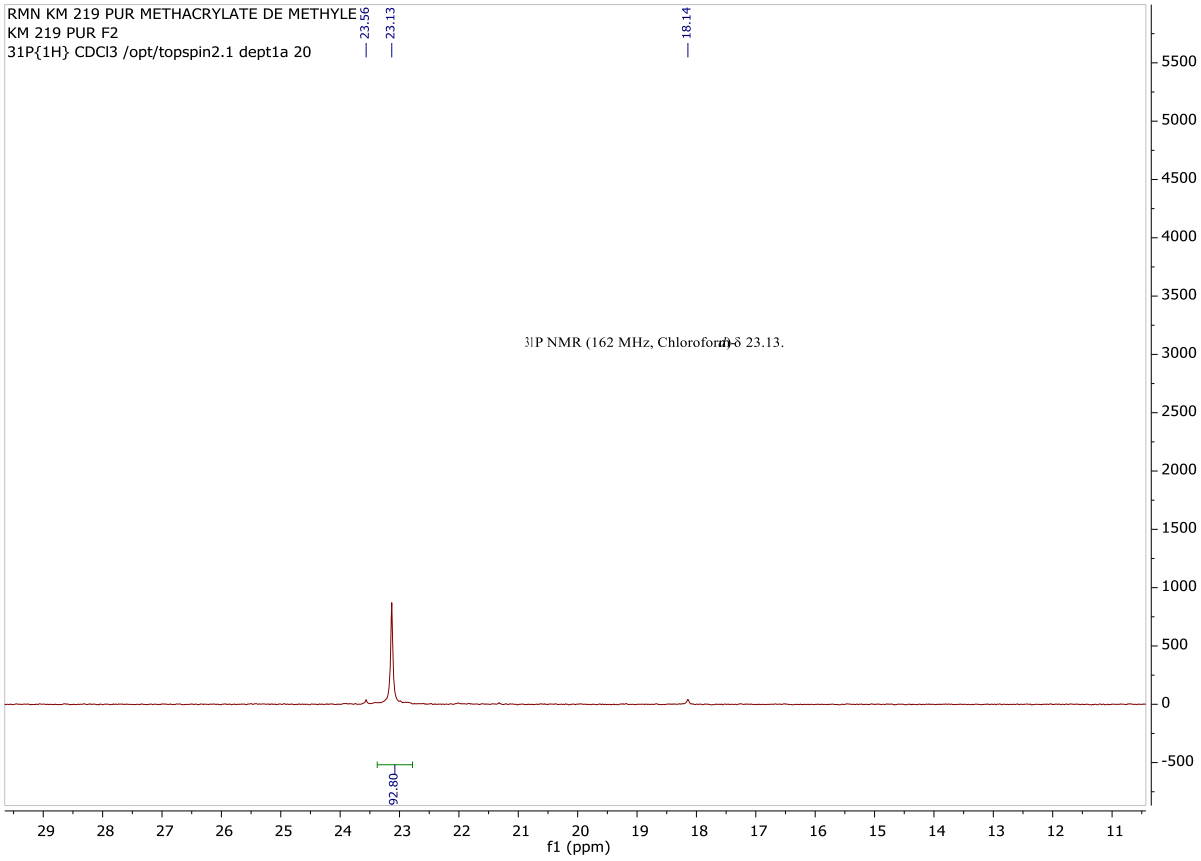
^{13}C NMR (101 MHz, $CDCl_3$) δ 184.02 (s), 168.16 (s), 145.12 (d, J = 5.1 Hz), 133.59 (d, J = 6.9 Hz), 132.51 (d, J = 2.8 Hz), 131.73 (dd, J = 12.0, 6.2 Hz), 131.13 (d, J = 2.5 Hz), 131.00 (d, J = 10.9 Hz), 130.63 (d, J = 9.9 Hz), 130.58 (s), 130.21 (s), 129.44 (d, J = 7.0 Hz), 128.90 (d, J = 12.2 Hz), 128.16 (s), 52.19 (s), 36.44 – 32.84 (m), 14.88 (s).



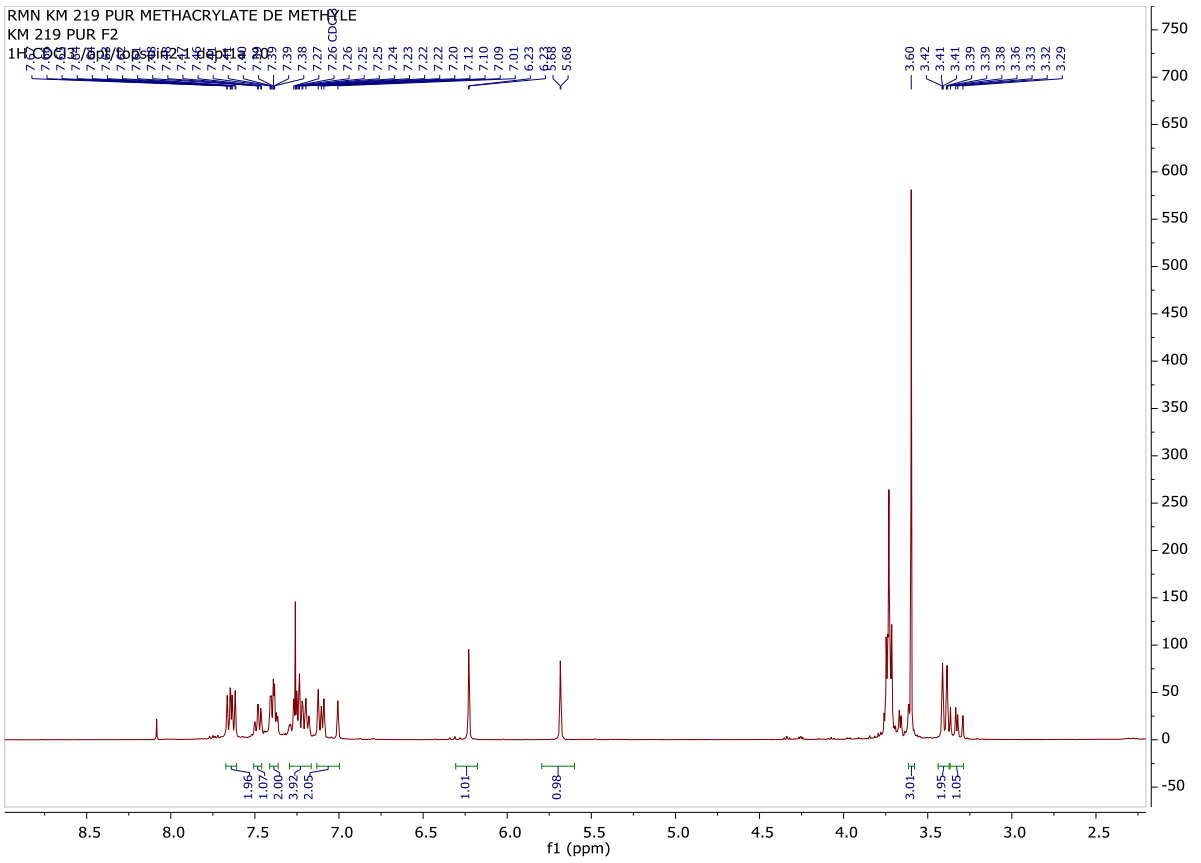
D:\LMP\OLAF\2024\data\S2408\MMN-7a.raw 2/20/2024 11:48:10 AM

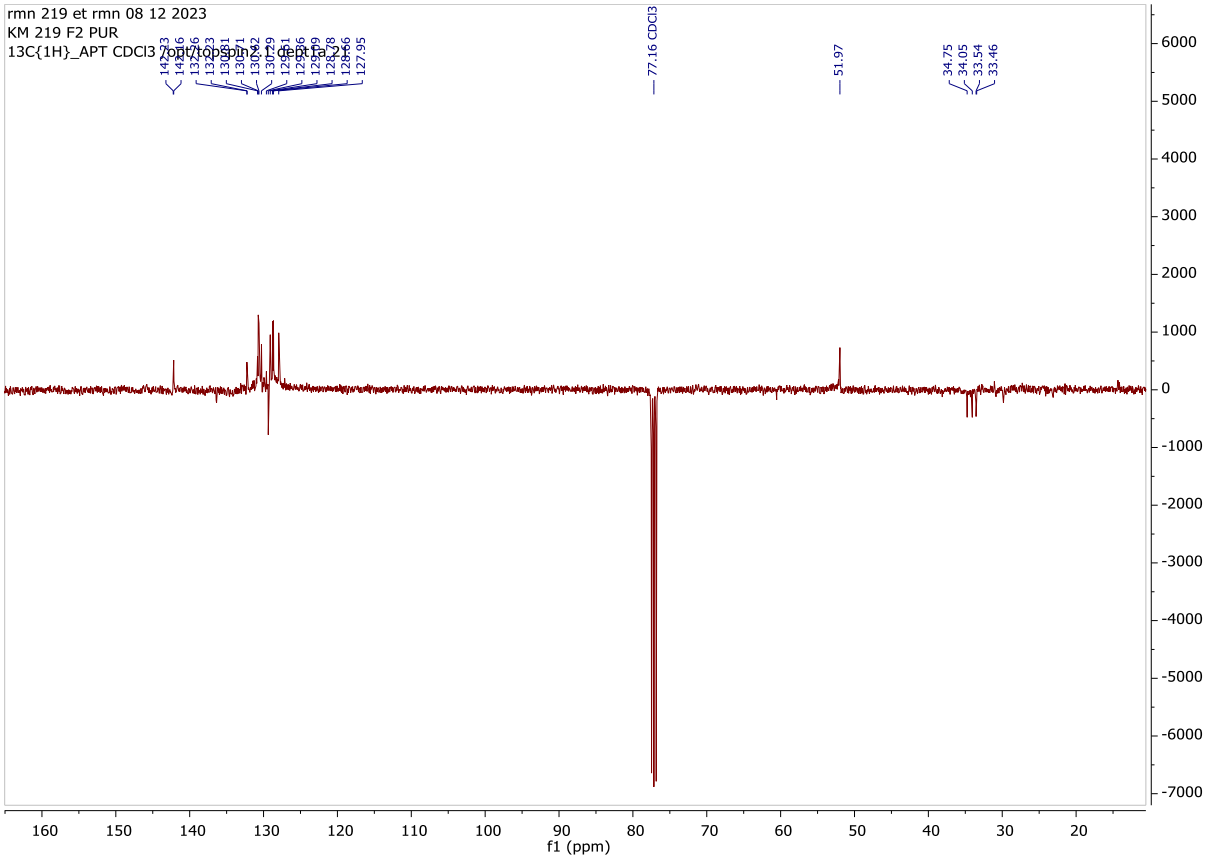
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched Fragments
1	339.1145	$C_{16}H_{20}O_7P$	0.12	339.11446	97.32	6	93.75	98.1	(Collection)
2	339.1145	$C_{16}H_{22}O_5NP_2$	-0.80	339.11477	84.45	5	78.35	82.2	(Collection)

S10: Methyl 2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate **(3ib)**



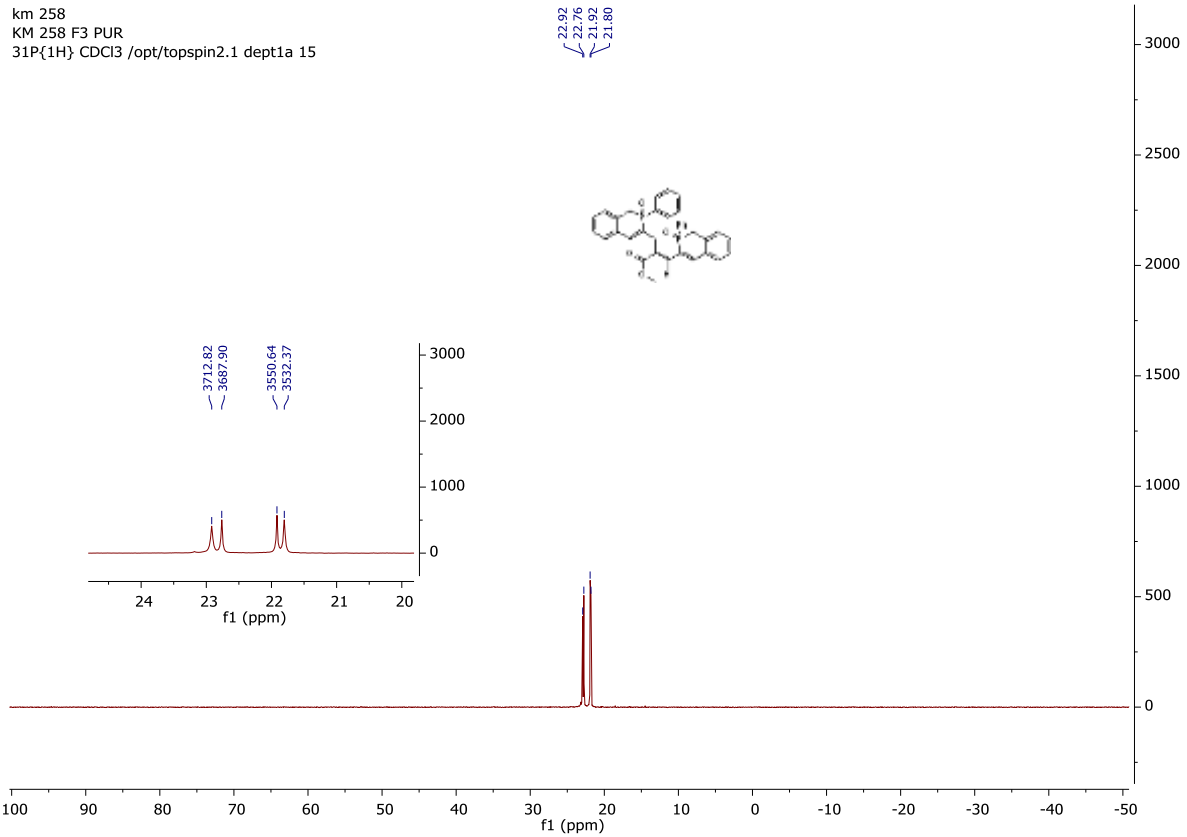
^{31}P NMR (162 MHz, Chloroform-*d*) δ 23.13.

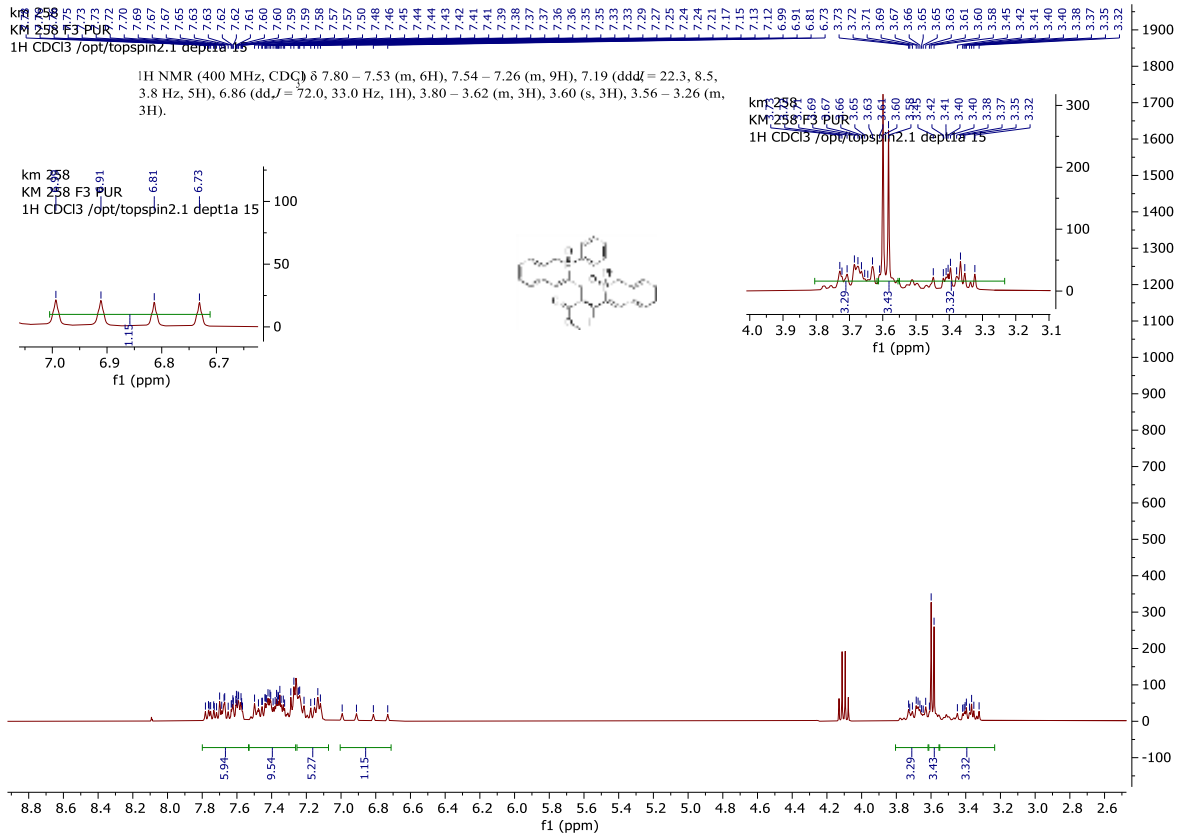


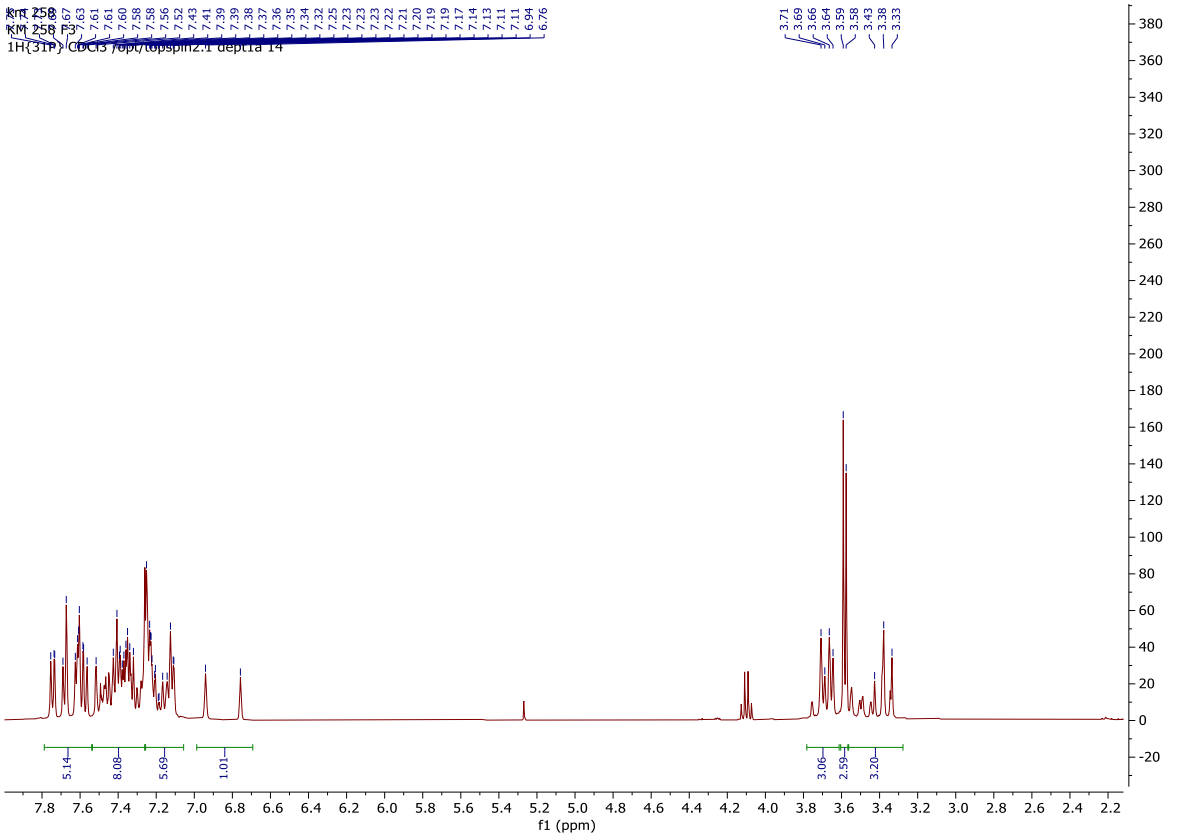

rmn 219 et rmn 08 12 2023
KM 219 F2 PUR
13C{1H}_APT CDCl3 /opt/topspin4.1 dept1a 28
77.16 CDCl3
51.97
f1 (ppm)

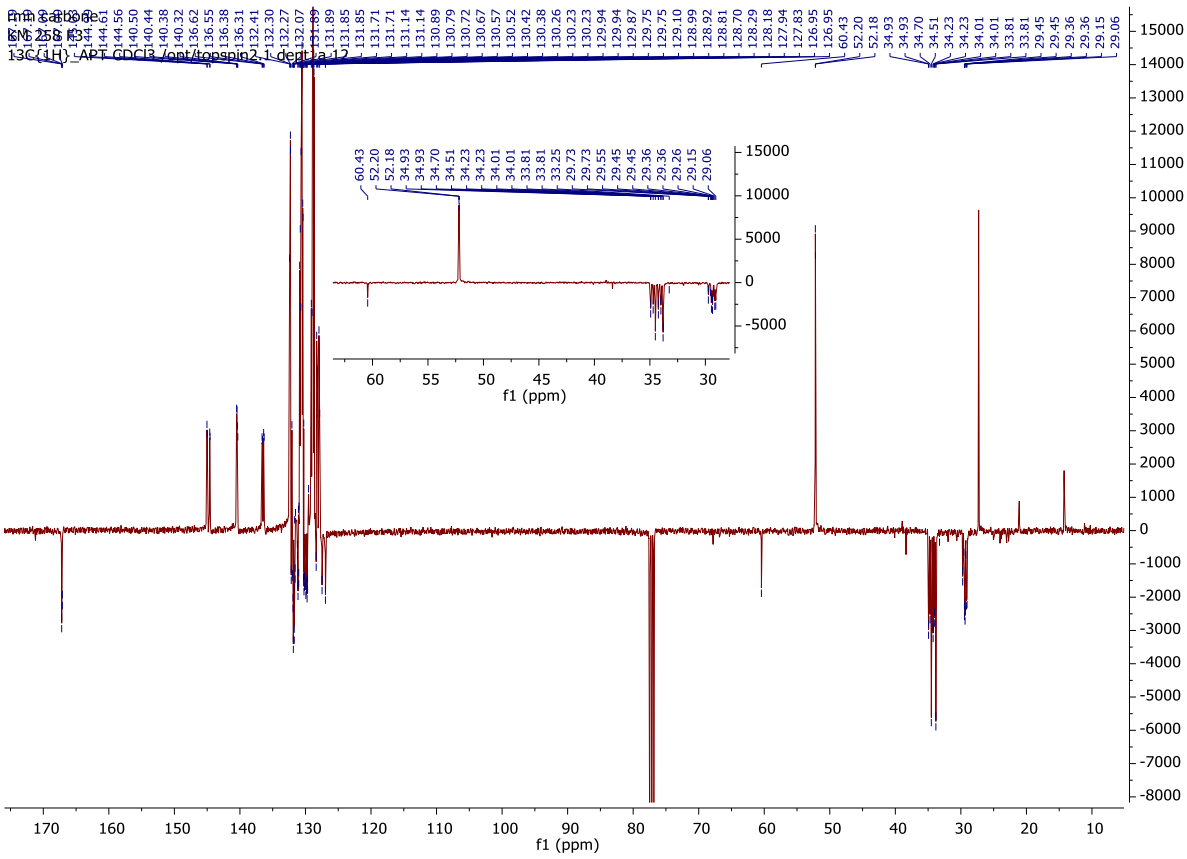

MMN-4d_1 #18 RT: 0.19 AV: 1 NL: 4.62E8
T: FTMS + p ESI Full ms [100.0000-1500.0000]
339.11450
C20H20O5P
340.11780
325.09885
214.08961
437.15128
577.16931
677.22156
775.25848
873.29523
1047.03699
1134.58130
1285.53967
1455.29236
Relative Abundance
m/z

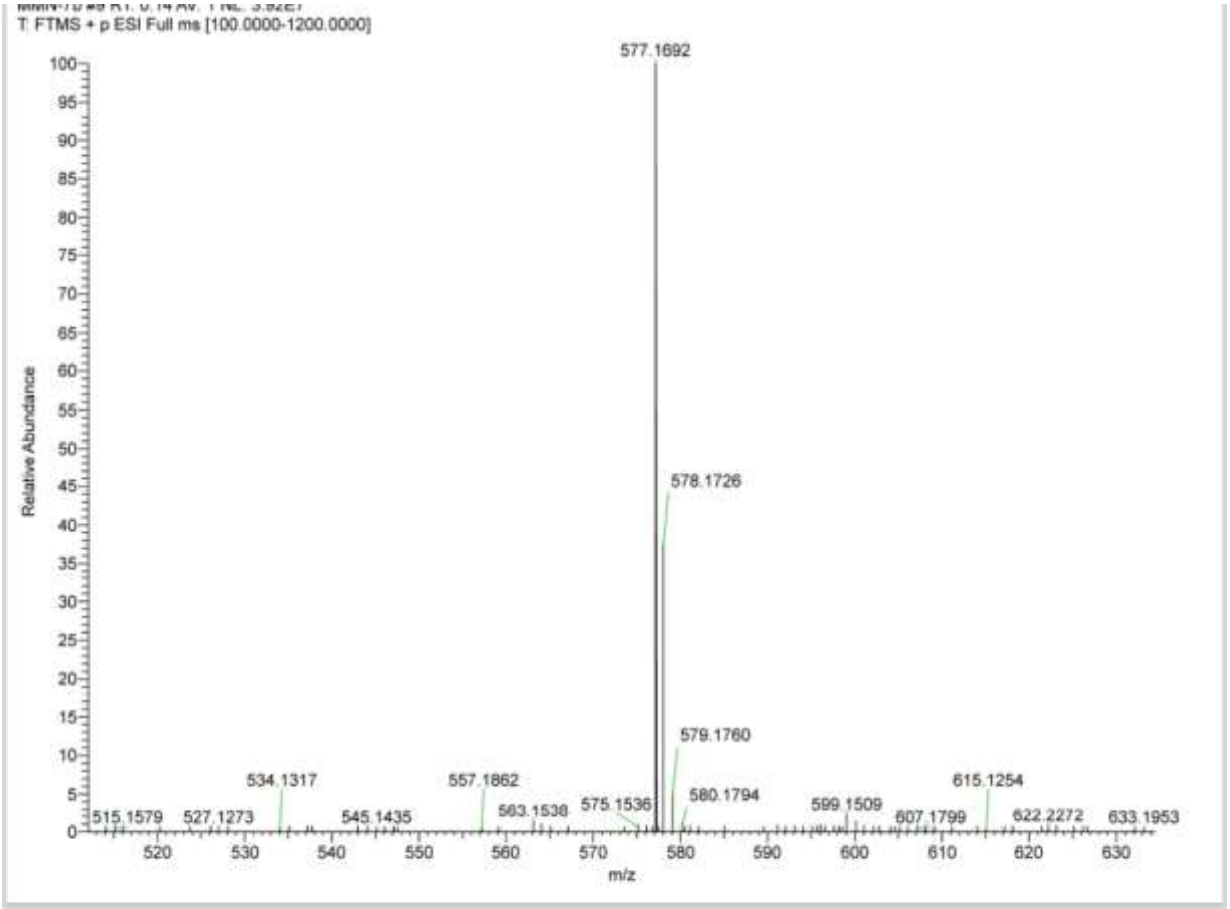
S11: Methyl 3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)-2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate **(3ic)**






KM 258
KM 258 F3
1H{31P} CDCl3 /opt/topspin2.1 dept1a 14
f1 (ppm)

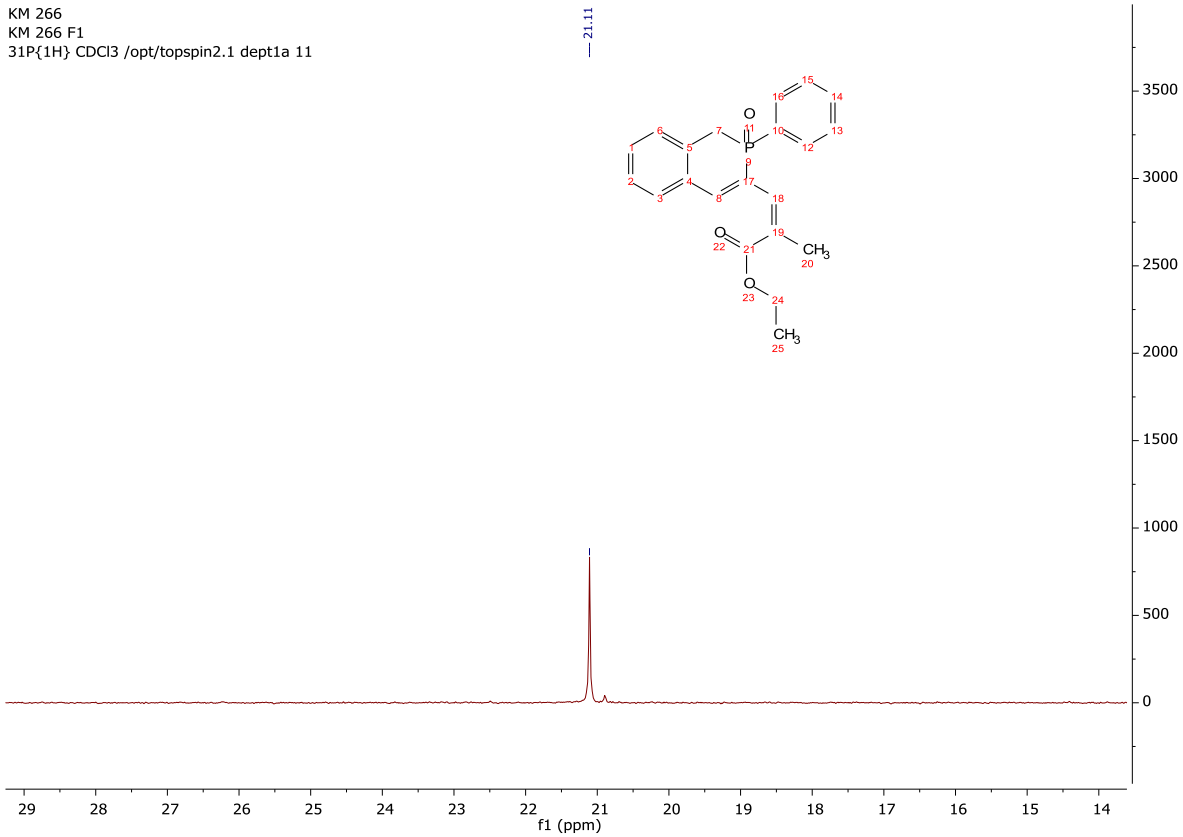

KM 258 F3
13C{1H} APT CDCl3 /opt/topspin2.1 dept1a 12
f1 (ppm)

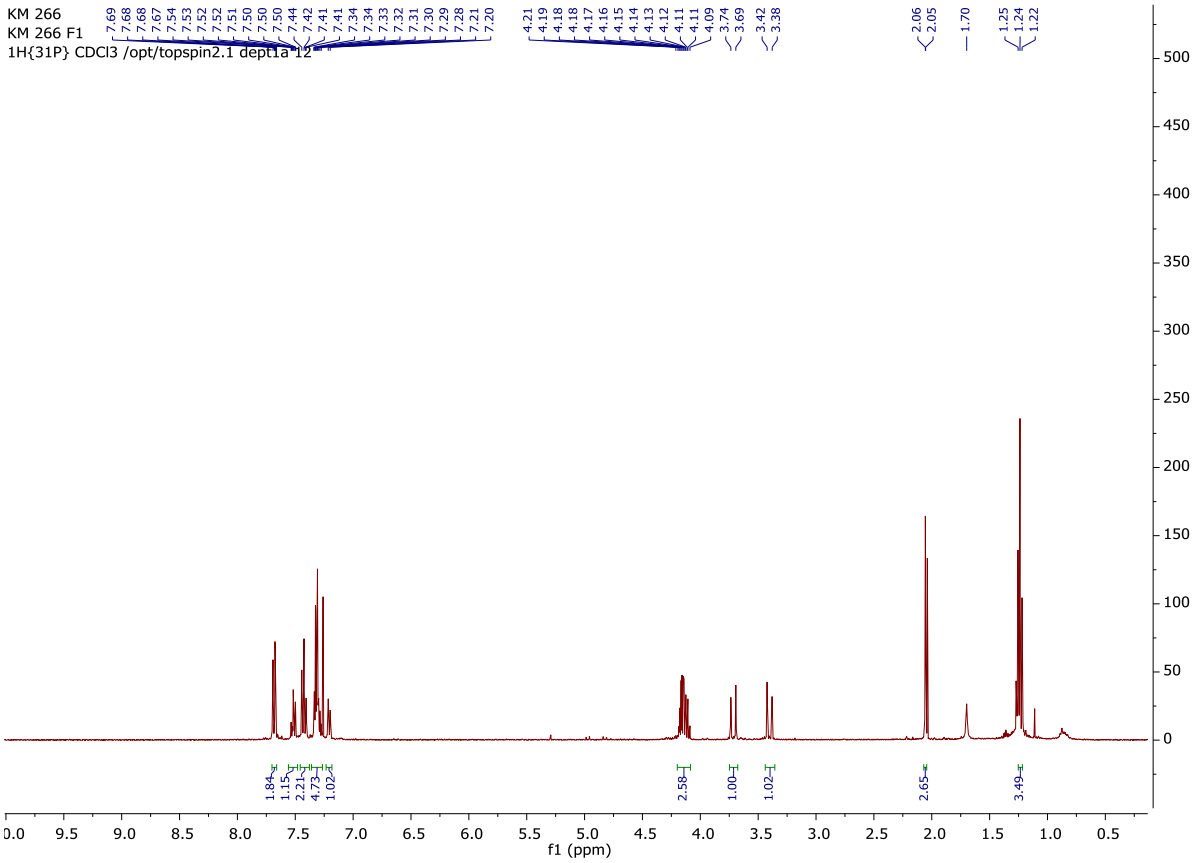


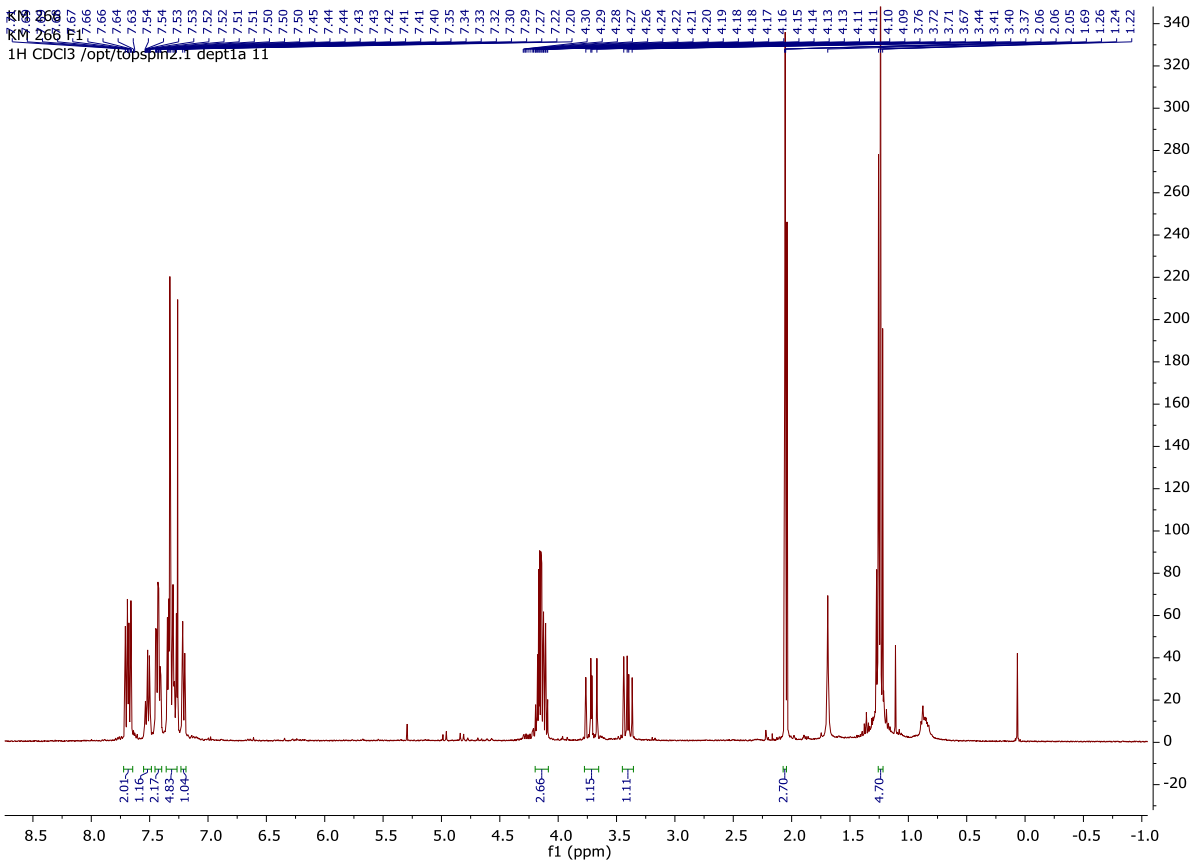
D:\LMP\OLAF\2024\data\S2408\MMN-7b.raw 2/20/2024 11:48:49 AM

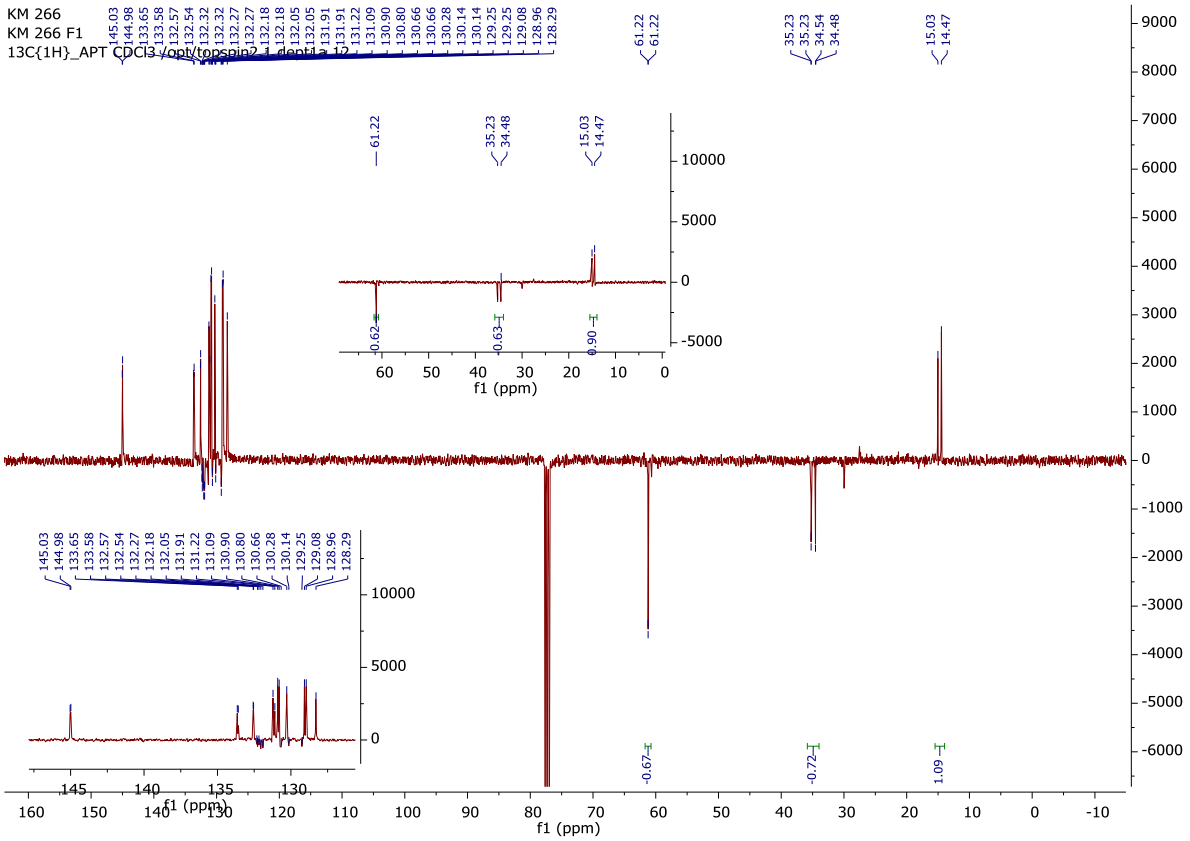
Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched Fragments
1	577.1694	$C_{30}H_{31}O_4P_3$	0.39	577.16921	94.42	5	92.34	96.22	(Collection)
3	577.1694	$C_4H_{31}O_{16}N_{10}P_3$	0.97	577.16888	87.15	1	66.89	70.25	(Collection)
8	577.1694	$C_{25}H_{35}O_{14}N_4P_3$	-0.49	577.16972	76.68	5	66.75	70.25	(Collection)

S12: Ethyl 2-((2-oxido-2-phenyl-1H-isophosphinolin-3-yl)methyl)acrylate **(3ja)**




KM 266
KM 266 F1
1H{31P} CDCl3 /opt/topspin2.1 dept1a 12
f1 (ppm)


KM 266 F1
1H CDCl3 /opt/topspin2.1 dept1a 11
f1 (ppm)



High Resolution Mass Result

Analysis Info

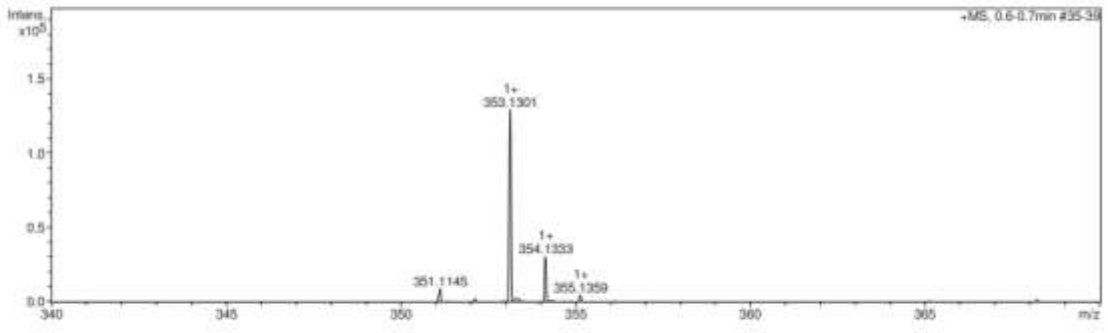
Sample Name **MMN-9 a _ KM-25**

Acquisition Date 3/11/2024 1:10:15 PM

Instrument / Ser# micrOTOF-Q 228888.10300

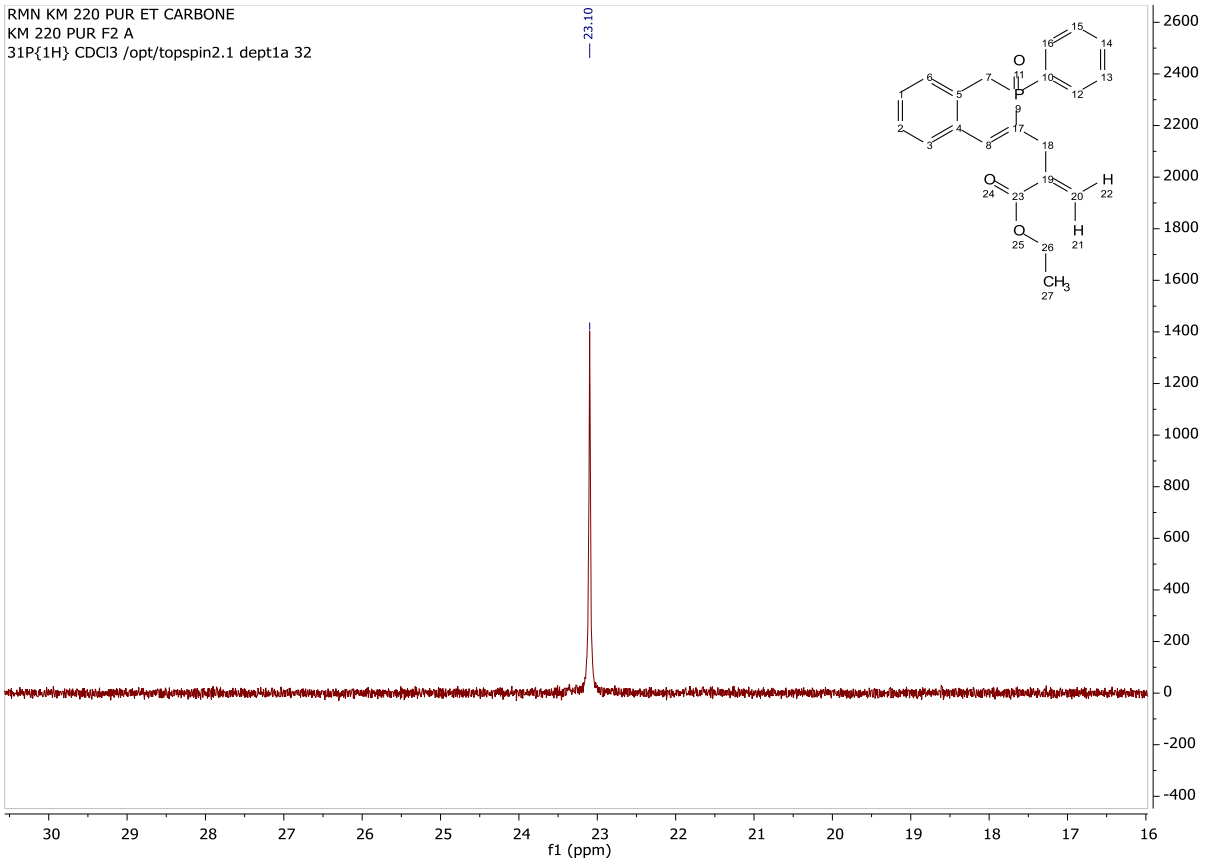
Acquisition Parameter

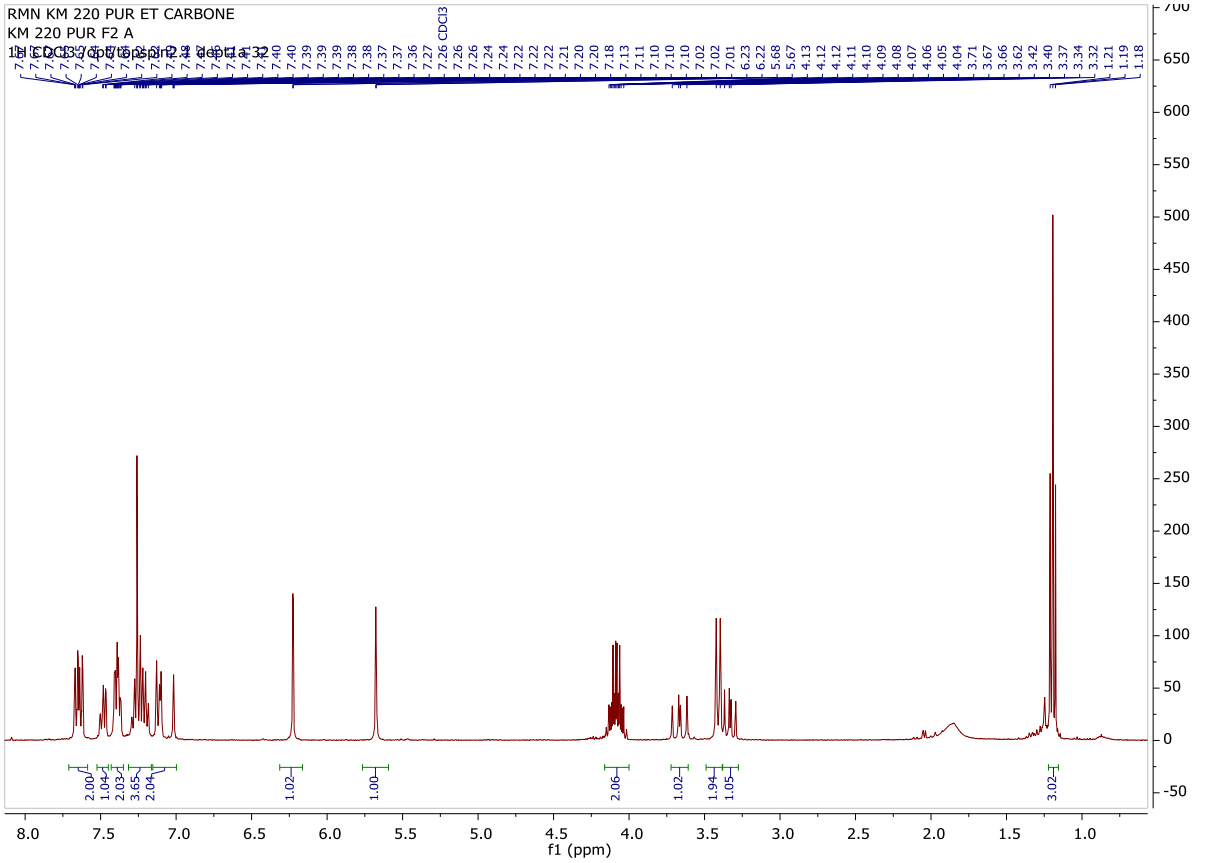
Source Type ESI | Ion Polarity Positive | Scan Begin 50 m/z | Scan End 3000 m/z

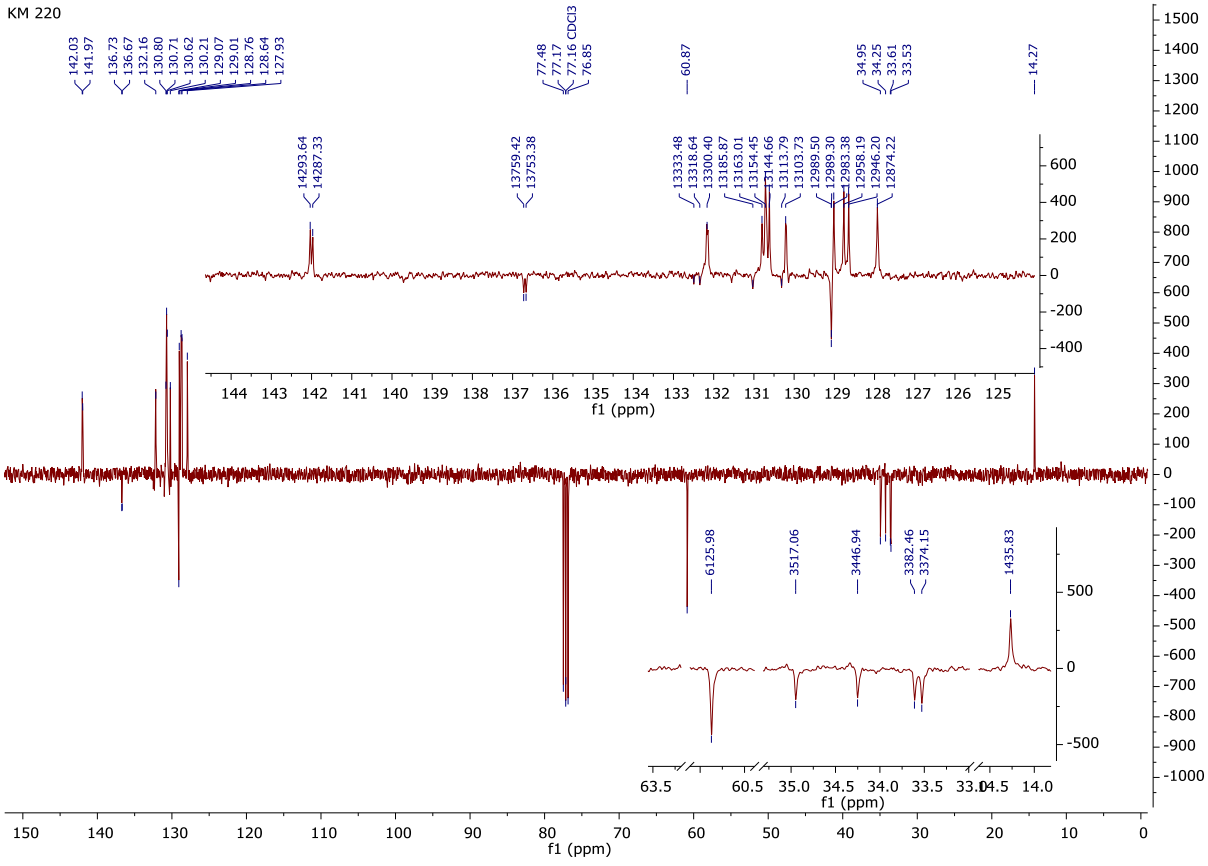


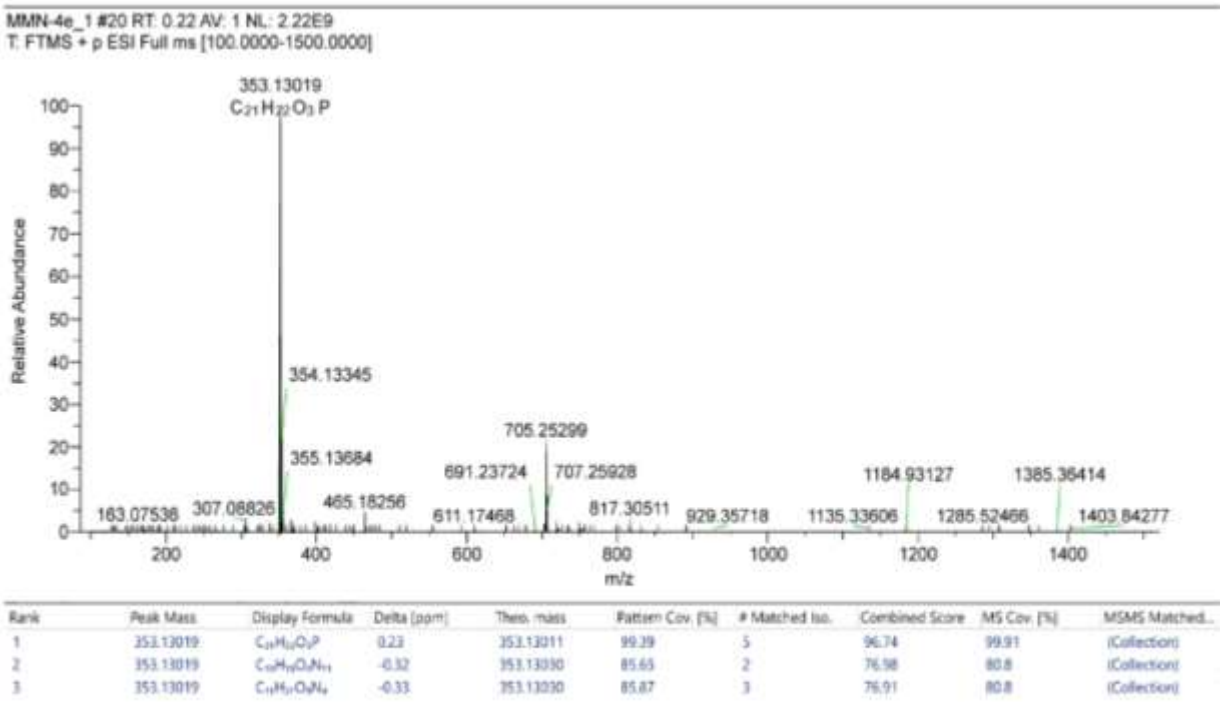
Meas. m/z	#	Ion Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule	Adduct
353.1301	1	C21H22O3P	100.00	353.1301	-0.0	-0.0	2.8	11.5	even	ok	M+H

S13: Ethyl 2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate **(3jb)**



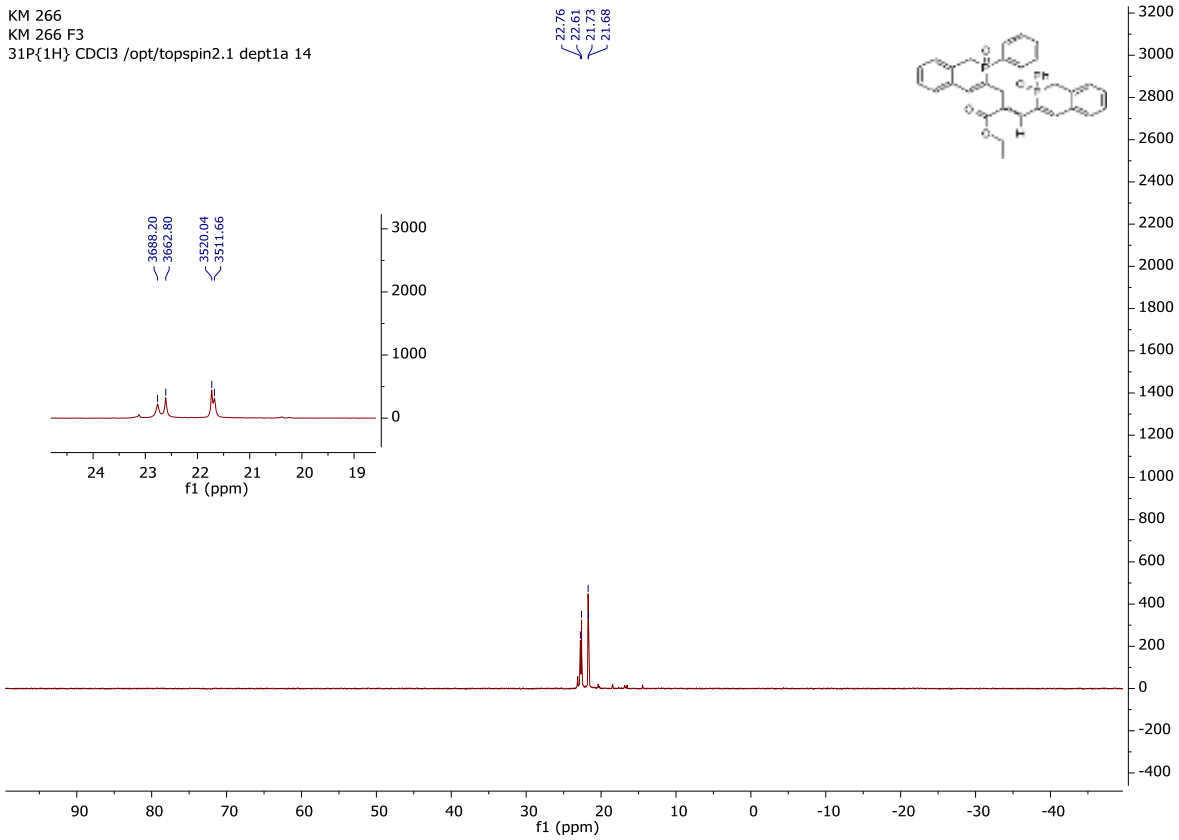

RMN KM 220 PUR ET CARBONE
KM 220 PUR F2 A
f1 (ppm)

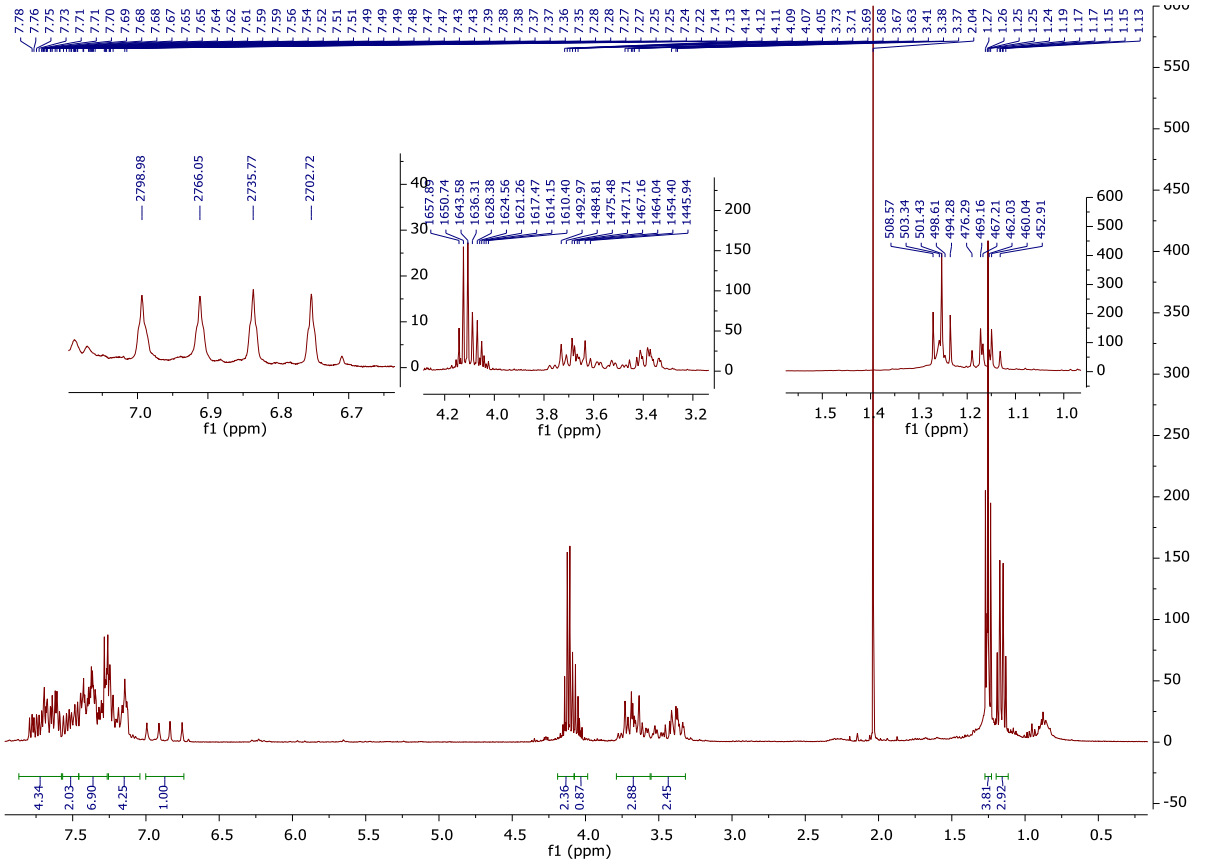

KM 220
f1 (ppm)

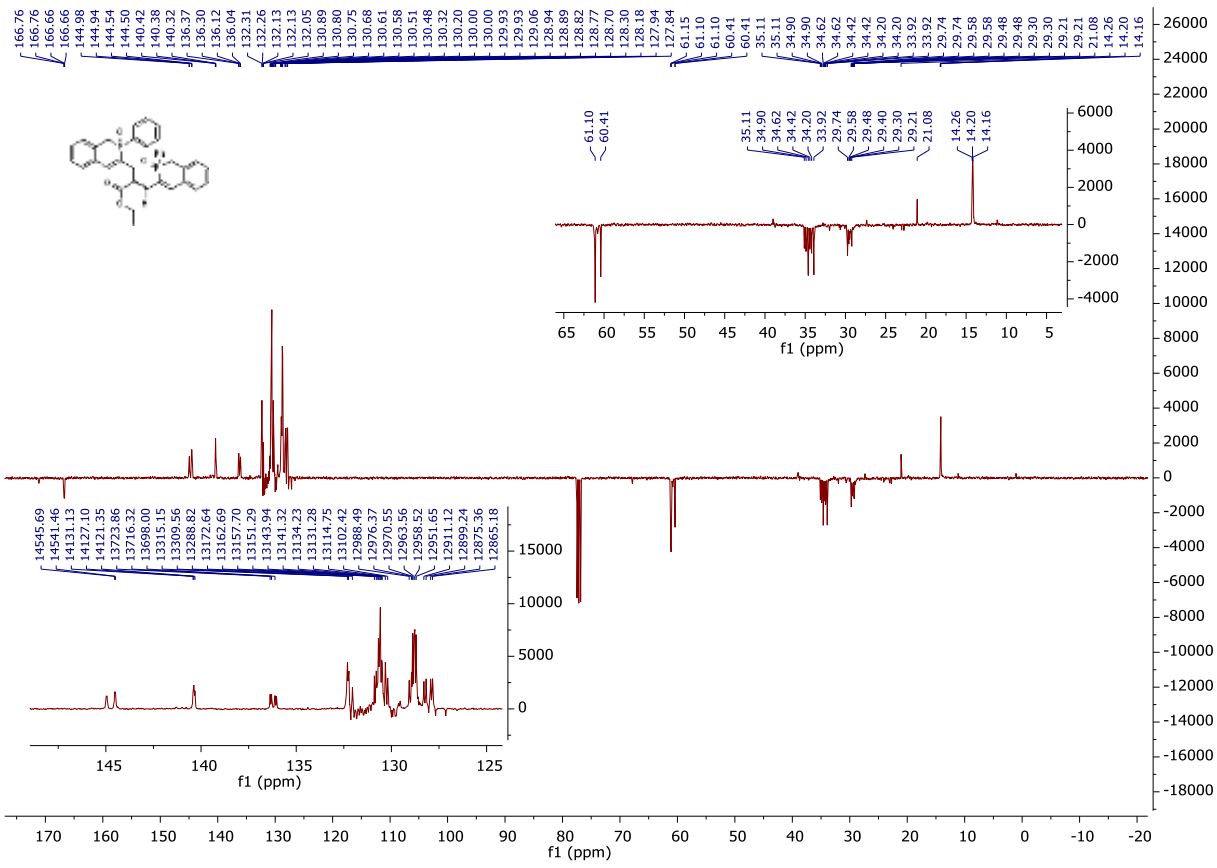


Rank	Peak Mass	Display Formula	Delta [ppm]	Theo. mass	Pattern Cov. [%]	# Matched Iso.	Combined Score	MS Cov. [%]	MSMS Matched...
1	353.13019	$C_{21}H_{22}O_3P$	0.23	353.13011	99.39	5	96.74	99.91	(Collection)
2	353.13019	$C_{20}H_{18}O_4N_3$	-0.32	353.13030	85.65	2	76.98	80.8	(Collection)
3	353.13019	$C_{19}H_{17}O_5N_4$	-0.33	353.13030	85.87	3	76.91	80.8	(Collection)

S14: Ethyl 3-(2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)-2-((2-oxido-2-phenyl-1*H*-isophosphinolin-3-yl)methyl)acrylate **(3jc)**




f1 (ppm)


f1 (ppm)

High Resolution Mass Result

Analysis Info

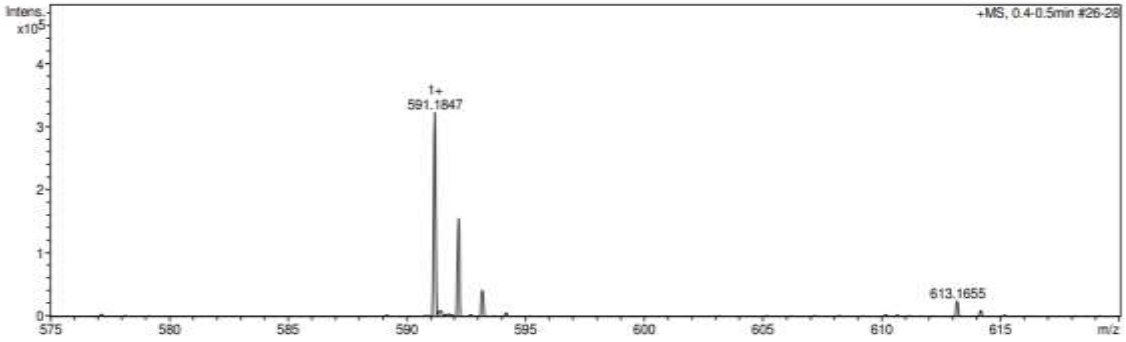
Sample Name **MMN-9 c _ KM-27**

Acquisition Date 3/11/2024 1:28:35 PM

Instrument / Ser# micrOTOF-Q 228888.10300

Acquisition Parameter

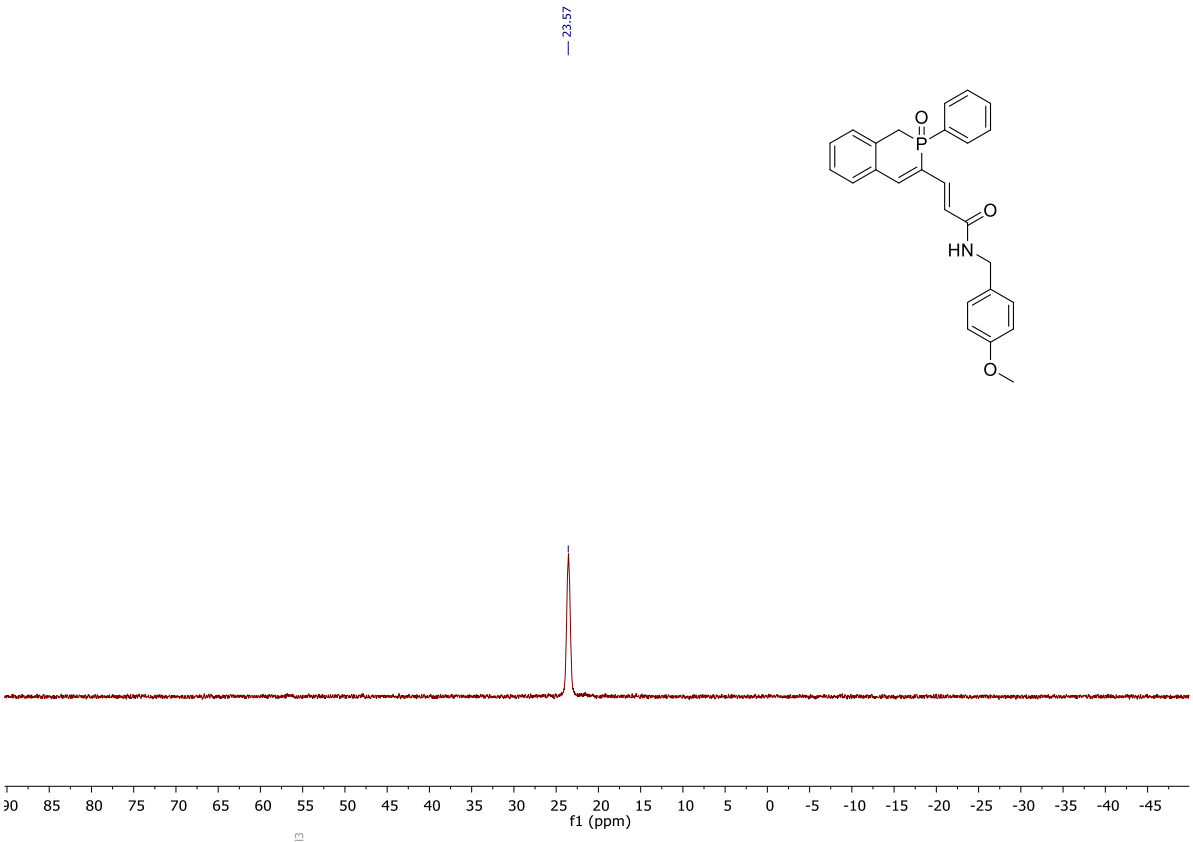
Source Type ESI | Ion Polarity Positive | Scan Begin 50 m/z | Scan End 3000 m/z

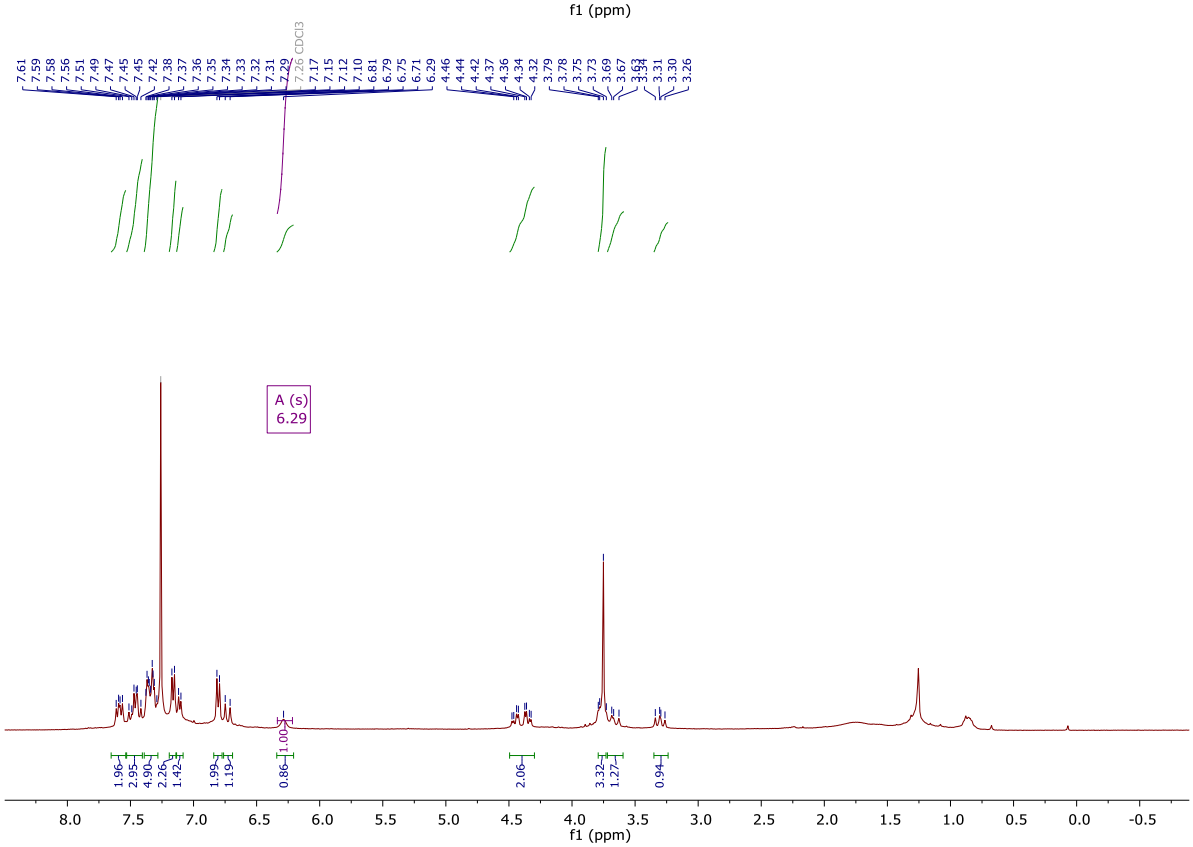


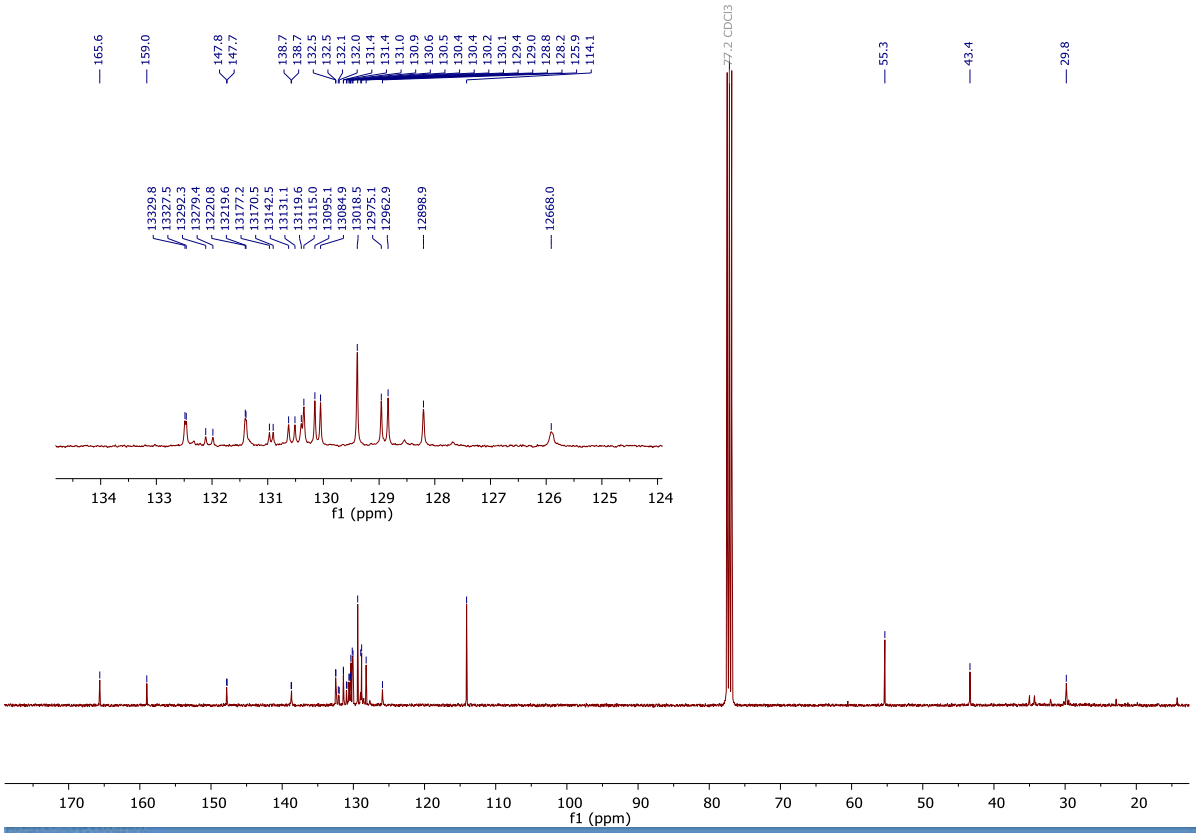
Meas. m/z	#	Ion Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e⁻ Conf	N-Rule	Adduct
591.1847	1	C37H29N4P2	65.28	591.1862	1.5	2.6	37.1	26.5	even	ok	M+H
	2	C36H33O4P2	100.00	591.1849	-0.2	-0.3	47.8	21.5	even	ok	M+H
613.1655	1	C36H32NaO4P2	100.00	613.1668	-1.3	-2.2	4.7	21.5	even	ok	M+Na
1181.3600	1	C72H65O8P4	100.00	1181.3624	2.4	2.1	101.3	42.5	even	ok	2M+H
1198.3887	1	C72H68NO8P4	100.00	1198.3890	-0.2	-0.2	505.3	41.5	even	ok	2M+NH4
1203.3425	1	C72H64NaO8P4	100.00	1203.3444	-1.9	-1.6	7.3	42.5	even	ok	2M+Na

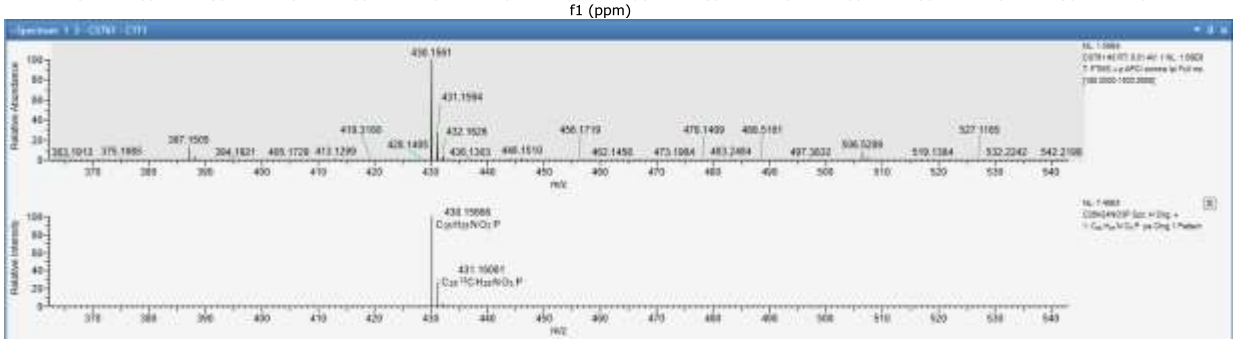
S15: (E)-N-(4-methoxybenzyl)-3-(2-oxido-2-phenyl-1H-isophosphinolin-3-yl)acrylamide

3k

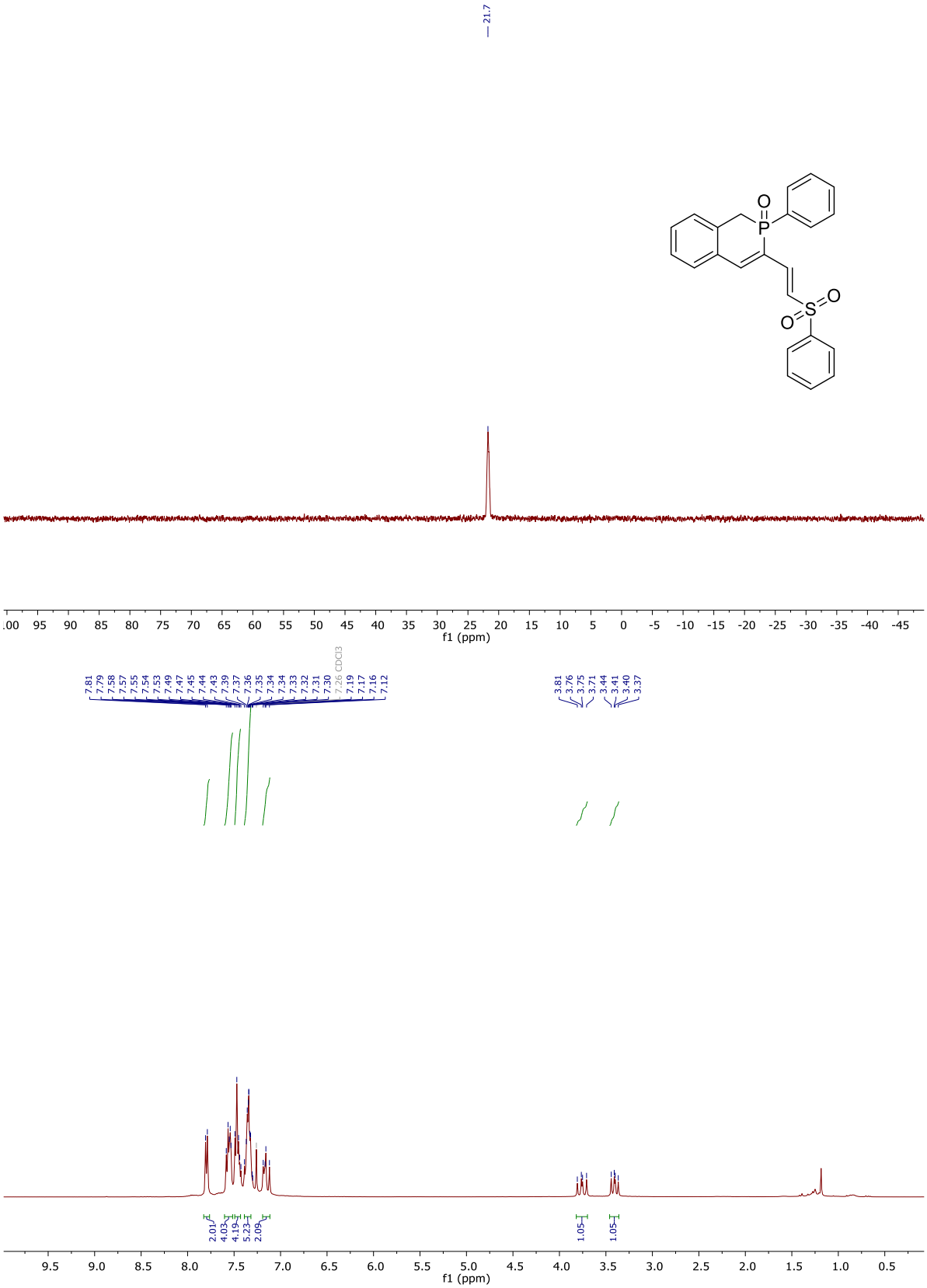


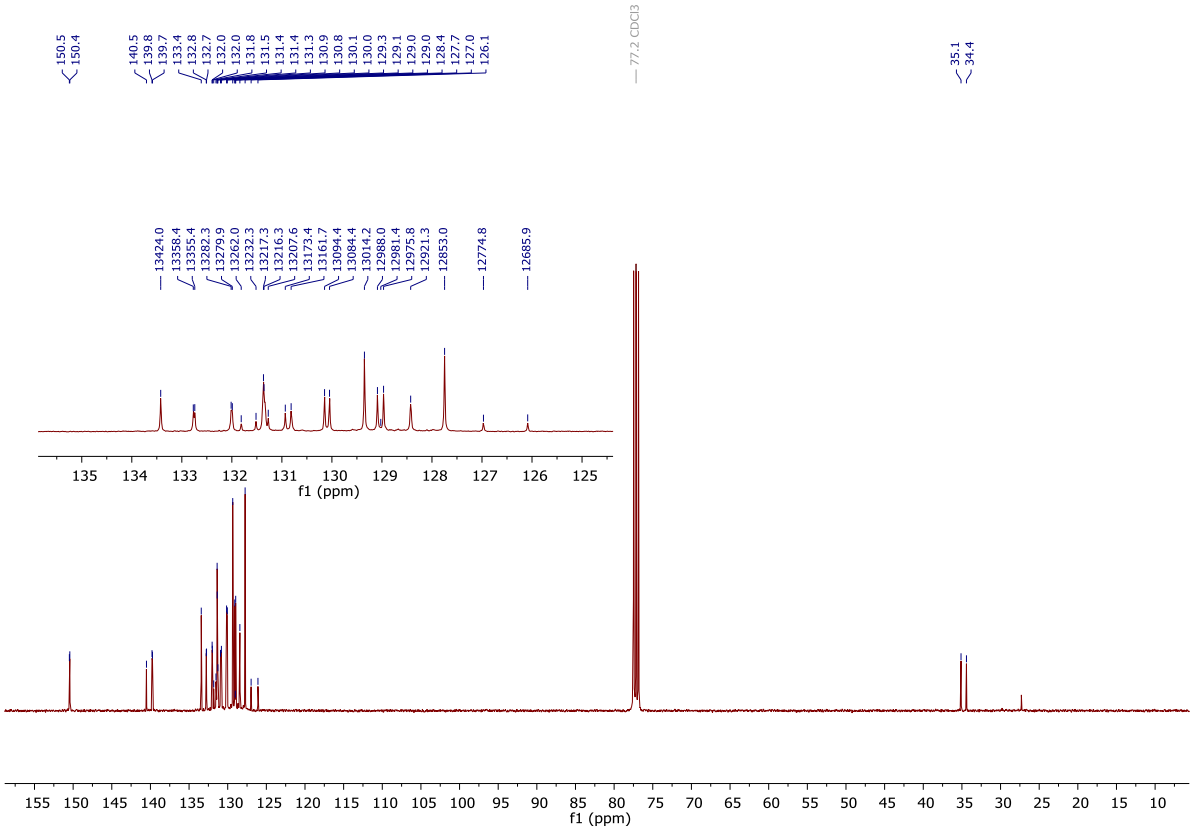


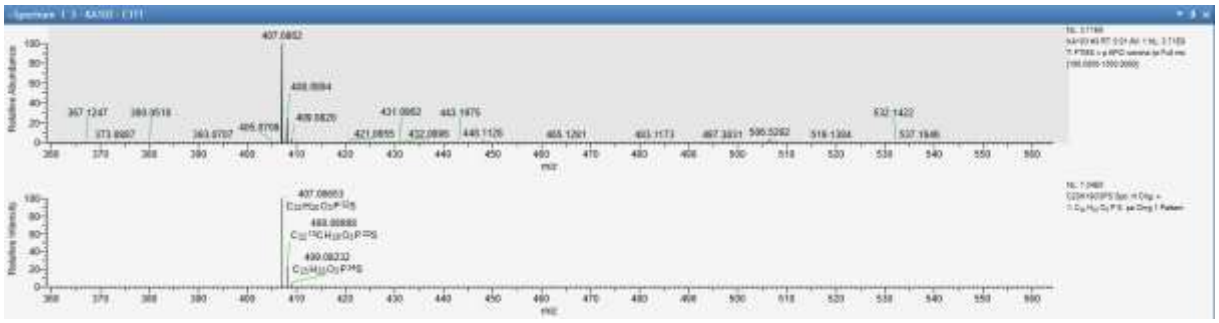

165.6
159.0
147.8
147.7
138.7
138.7
132.5
132.1
132.0
131.4
131.4
131.0
130.9
130.6
130.5
130.4
130.4
130.2
130.1
129.4
129.0
128.8
128.2
125.9
114.1
77.2 CDCl3
55.3
43.4
29.8
13329.8
13327.5
13292.3
13279.4
13220.8
13219.6
13177.2
13170.5
13142.5
13131.1
13119.6
13115.0
13095.1
13084.9
13018.5
12975.1
12962.9
12898.9
12668.0
134 133 132 131 130 129 128 127 126 125 124
f1 (ppm)
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20
f1 (ppm)


436.1661
431.1594
418.3100
432.1628
428.1495
456.1719
476.1499
488.5181
527.1165
387.1508
384.1821
405.1728
413.1299
436.1363
448.1510
462.1458
473.1084
483.2484
497.3632
506.5299
519.1384
532.2242
542.2108
m/z
438.15886
C29H34NO3P
431.16061
C28 13CH34NO3P
m/z

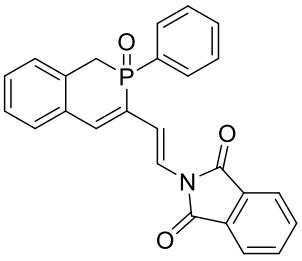
S16: (E)-2-Phenyl-3-(2-(phenylsulfonyl)vinyl)-1H-isophosphinoline 2-oxide **3l**

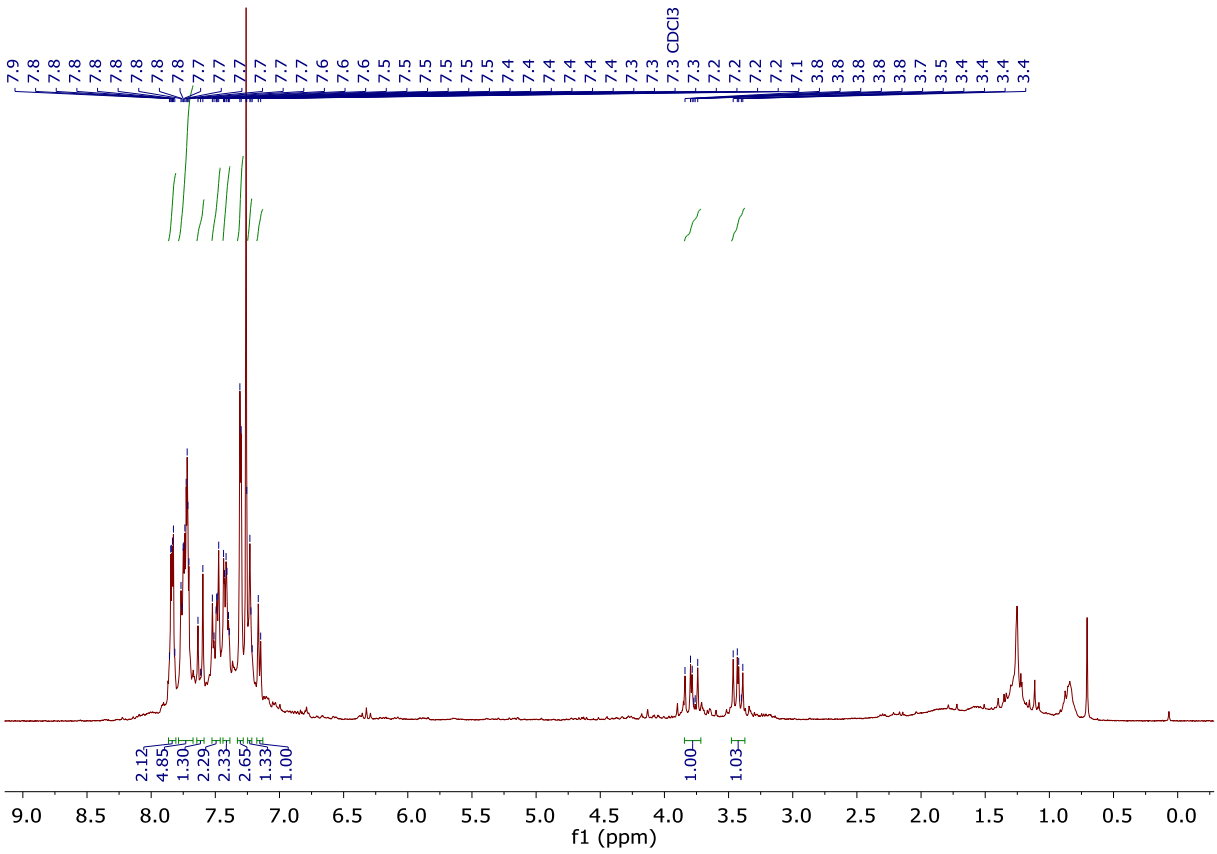


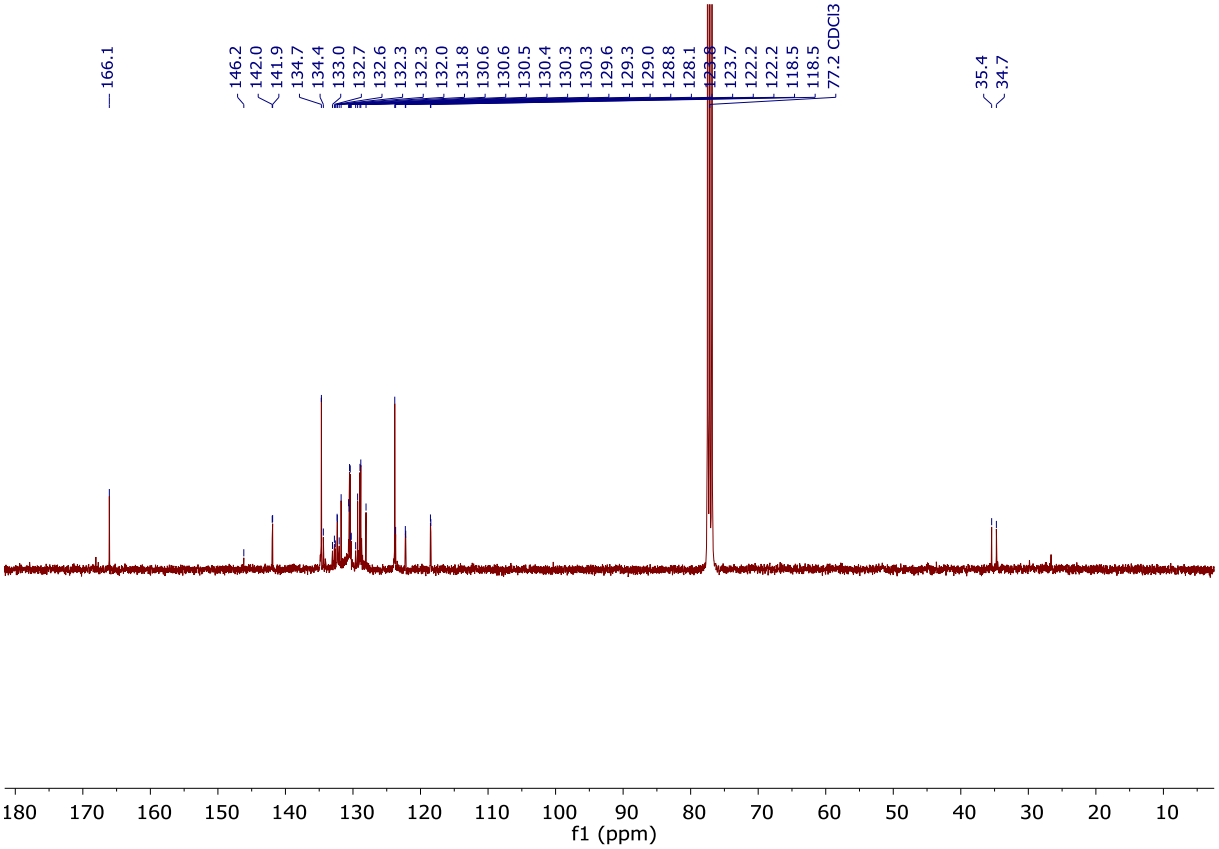

150.5
150.4
140.5
139.8
139.7
133.4
132.8
132.7
132.0
132.0
131.8
131.5
131.4
131.4
131.3
130.9
130.8
130.1
130.0
129.3
129.1
129.0
129.0
128.4
127.7
127.0
126.1
77.2 CDCl3
35.1
34.4
13424.0
13358.4
13355.4
13282.3
13279.9
13262.0
13232.3
13217.3
13216.3
13207.6
13173.4
13161.7
13094.4
13084.4
13014.2
12988.0
12981.4
12975.8
12921.3
12853.0
12774.8
12685.9
135 134 133 132 131 130 129 128 127 126 125
f1 (ppm)
155 150 145 140 135 130 125 120 115 110 105 100 95 90 85 80 75 70 65 60 55 50 45 40 35 30 25 20 15 10
f1 (ppm)

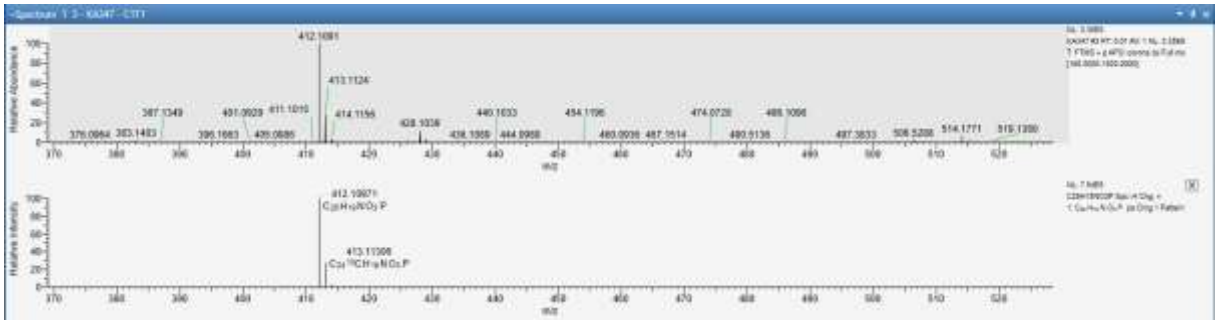


S17: (E)-2-(2-(2-oxido-2-phenyl-1H-isophosphinolin-3-yl)vinyl)isoindoline-1,3-dione **3m**






166.1
146.2
142.0
141.9
134.7
134.4
133.0
132.7
132.6
132.3
132.3
132.0
131.8
130.6
130.6
130.5
130.4
130.3
130.3
129.6
129.3
129.0
128.8
128.1
123.8
123.7
122.2
122.2
118.5
77.2 CDCl3
35.4
34.7
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)



Crystallographic data of sulfonated product **3l**