

Supplementary Material

C-Alkyl bis-phosphoryl chelating systems for the potential recovery of strategic metals

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Jean-Luc Pirat^a, and David Virieux^{a*}

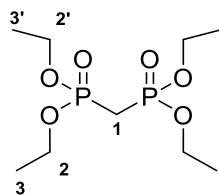
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Université Abdou Moumouni, B.P. 10662, Niamey, Niger.

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S1: ^{31}P , ^1H and ^{13}C NMR spectra of tetraethyl methylenebis(phosphonate) (1)

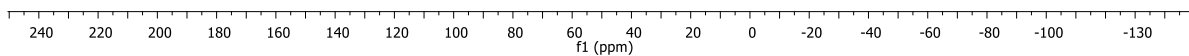
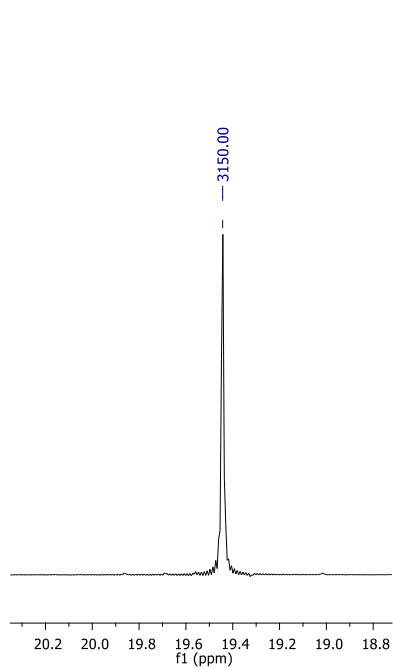
tetraethyl methylenebis(phosphonate)

 $\text{C}_9\text{H}_{22}\text{O}_6\text{P}_2$

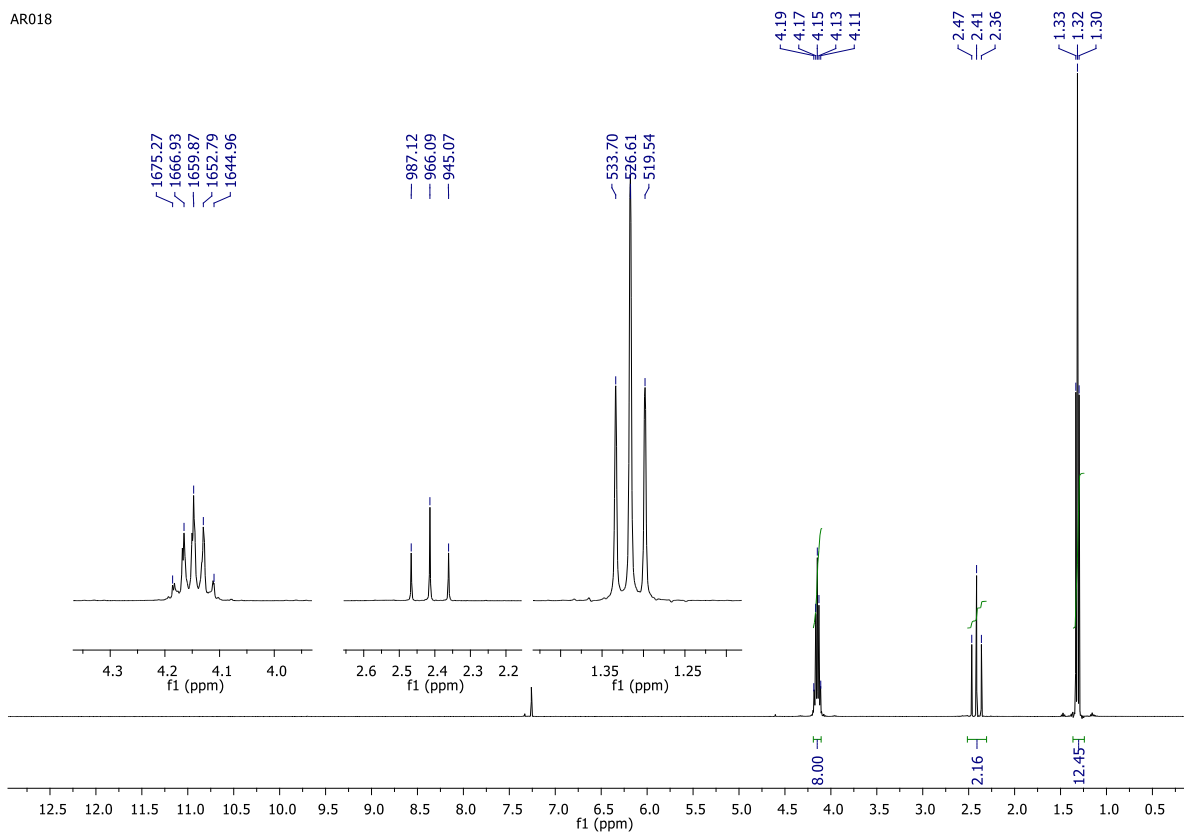
288,22 g/mol

Colorless oil

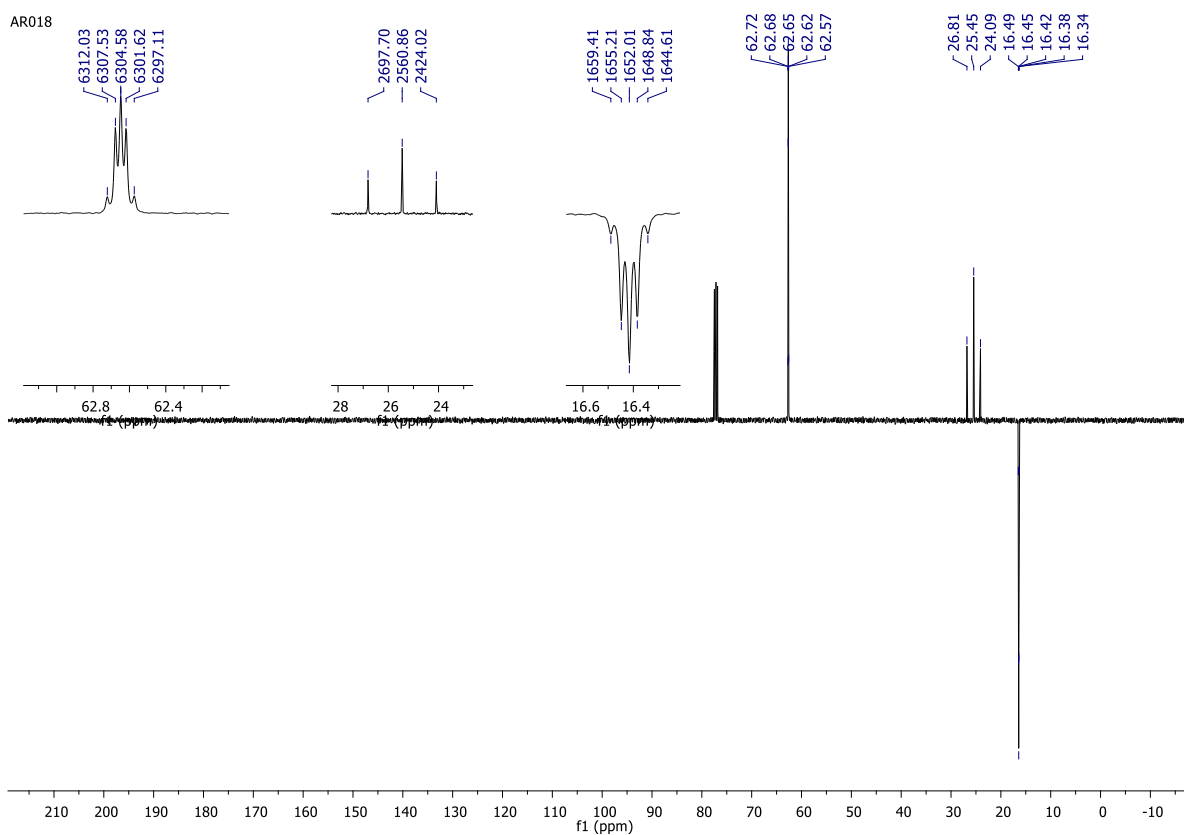
AR018

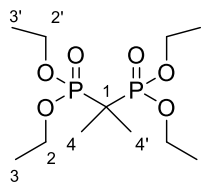


AR018



AR018



S2: ^{31}P , ^1H and ^{13}C NMR spectra of tetraethyl propane-2,2-diylbis(phosphonate) (3a)

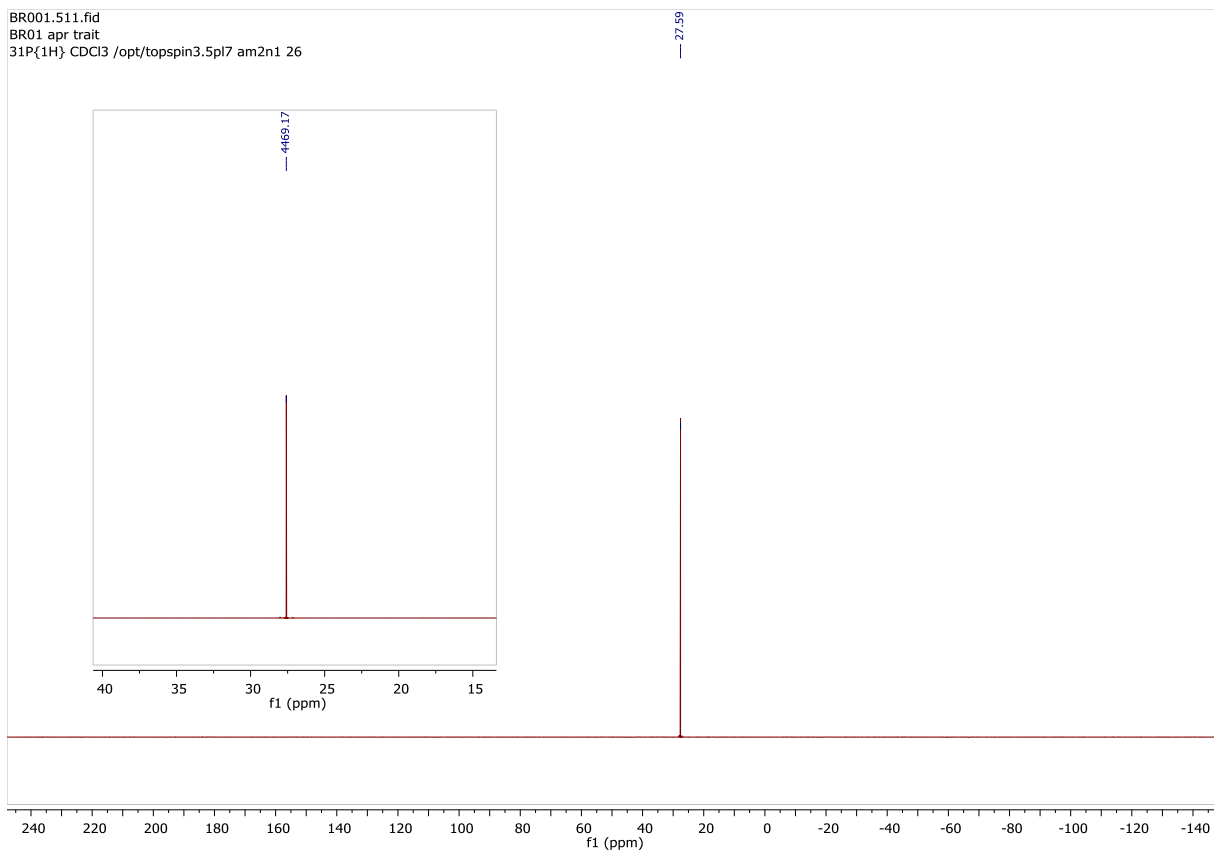
tetraethyl propane-2,2-diylbis(phosphonate)

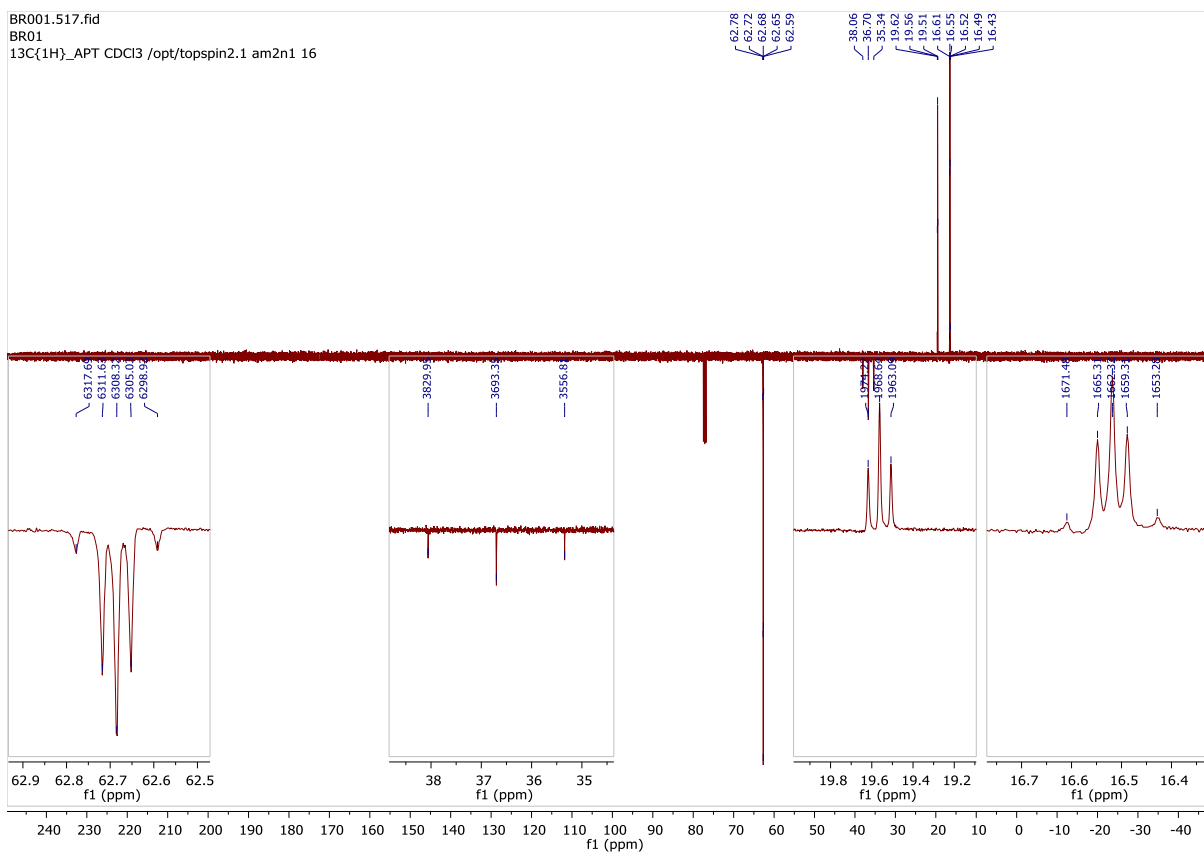
 $\text{C}_{11}\text{H}_{26}\text{O}_6\text{P}_2$

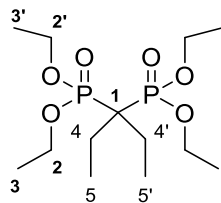
316,27 g/mol

Colorless oil

BR001.511.fid
BR01 apr trait
31P{1H} CDCl3 /opt/topspin3.5pl7 am2n1 26





S3: ^{31}P , ^1H and ^{13}C NMR Spectra of tetraethyl pentane-3,3-diylbis(phosphonate) (3b)

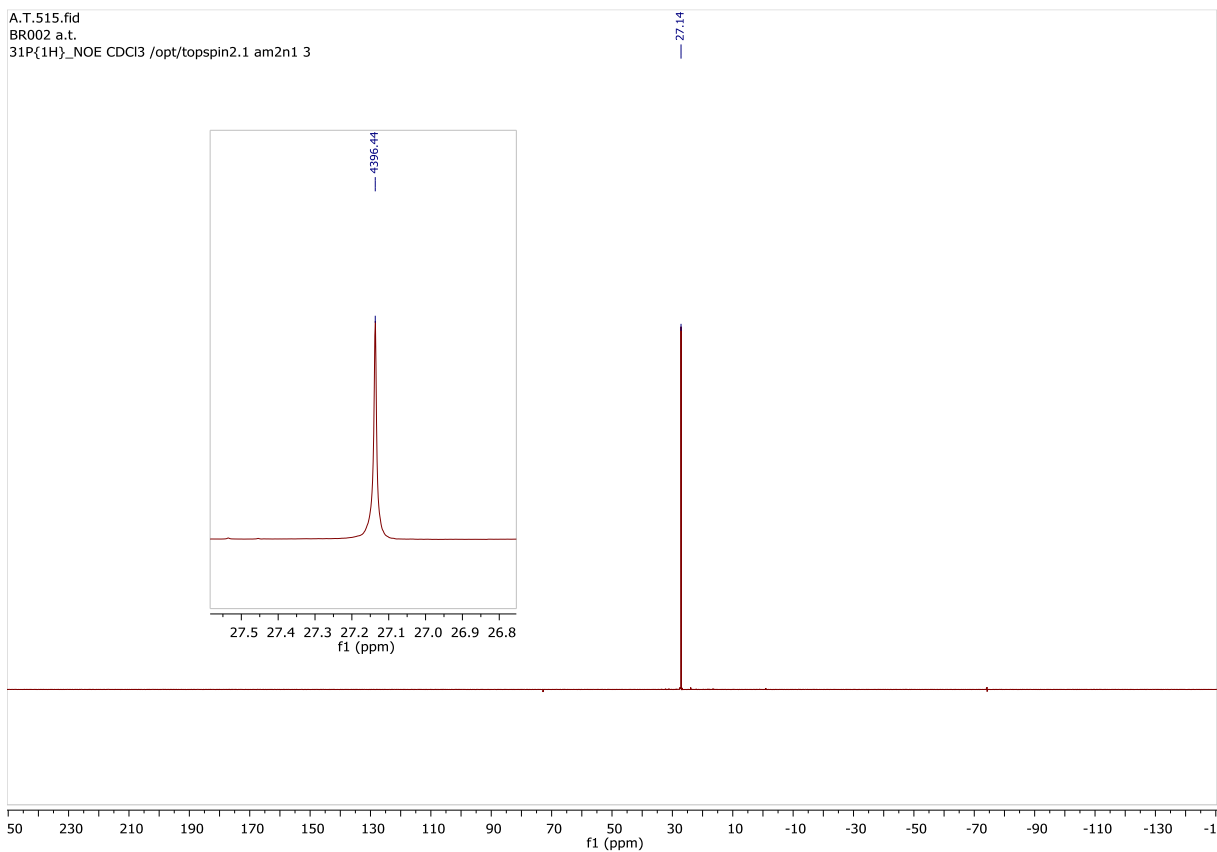
tetraethyl pentane-3,3-diylbis(phosphonate)

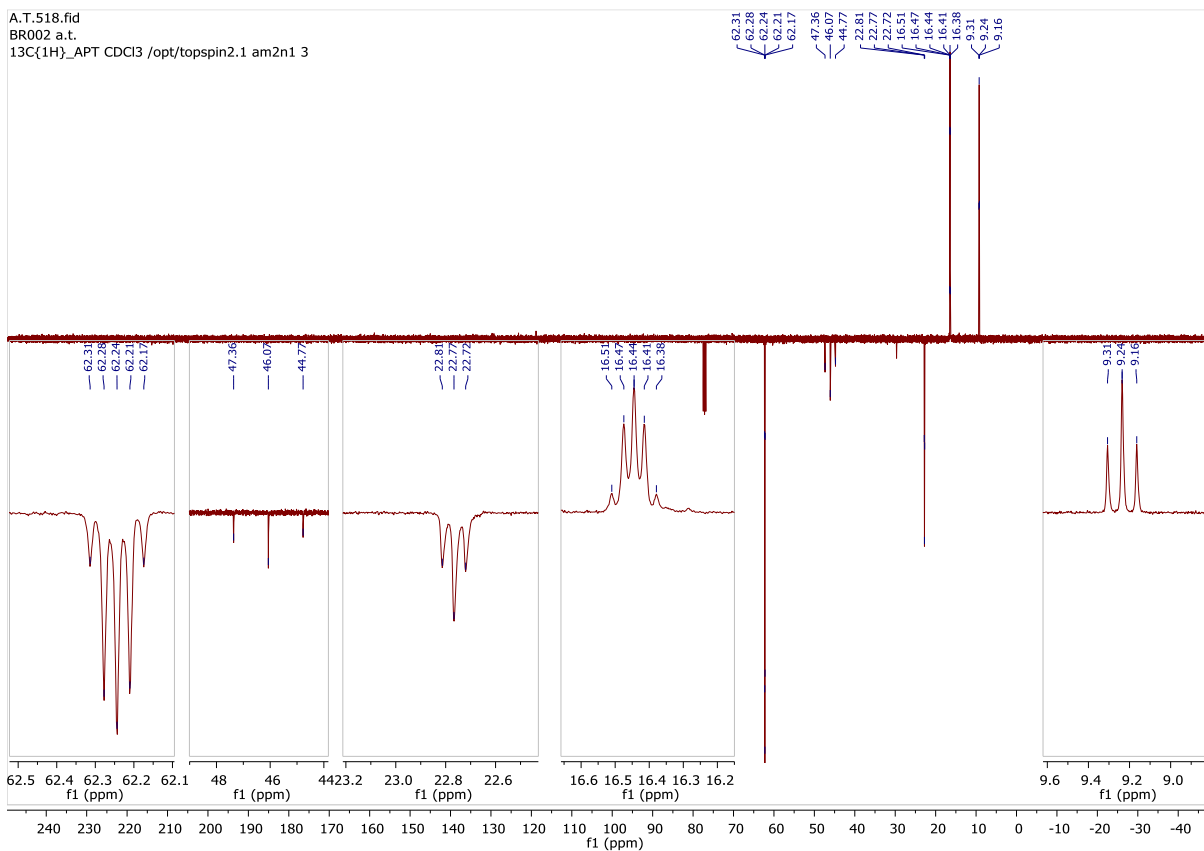
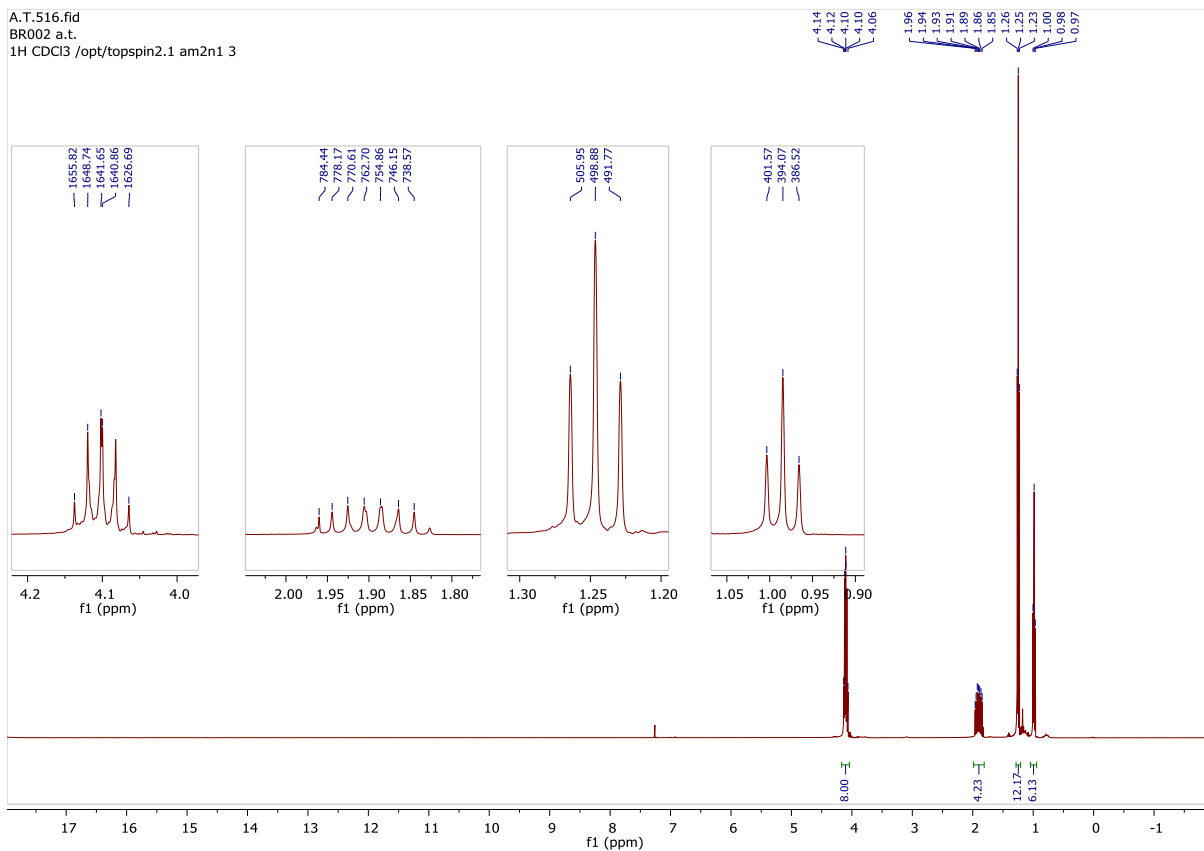
 $\text{C}_{13}\text{H}_{30}\text{O}_6\text{P}_2$

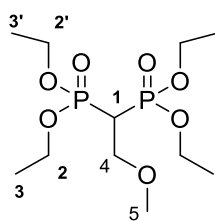
344,32 g/mol

Colorless oil

A.T.515.fid
BR002 a.t.
31P{1H}_NOE CDCl3 /opt/topspin2.1 am2n1 3





S4: ^{31}P , and ^1H , ^{13}C NMR spectra and mass analysis of tetraethyl (2-methoxyethane-1,1-diyl)bis(phosphonate) (4)

tetraethyl (2-methoxyethane-1,1-diyl)bis(phosphonate)

 $\text{C}_{11}\text{H}_{26}\text{O}_7\text{P}_2$

332,27 g/mol

Colorless oil

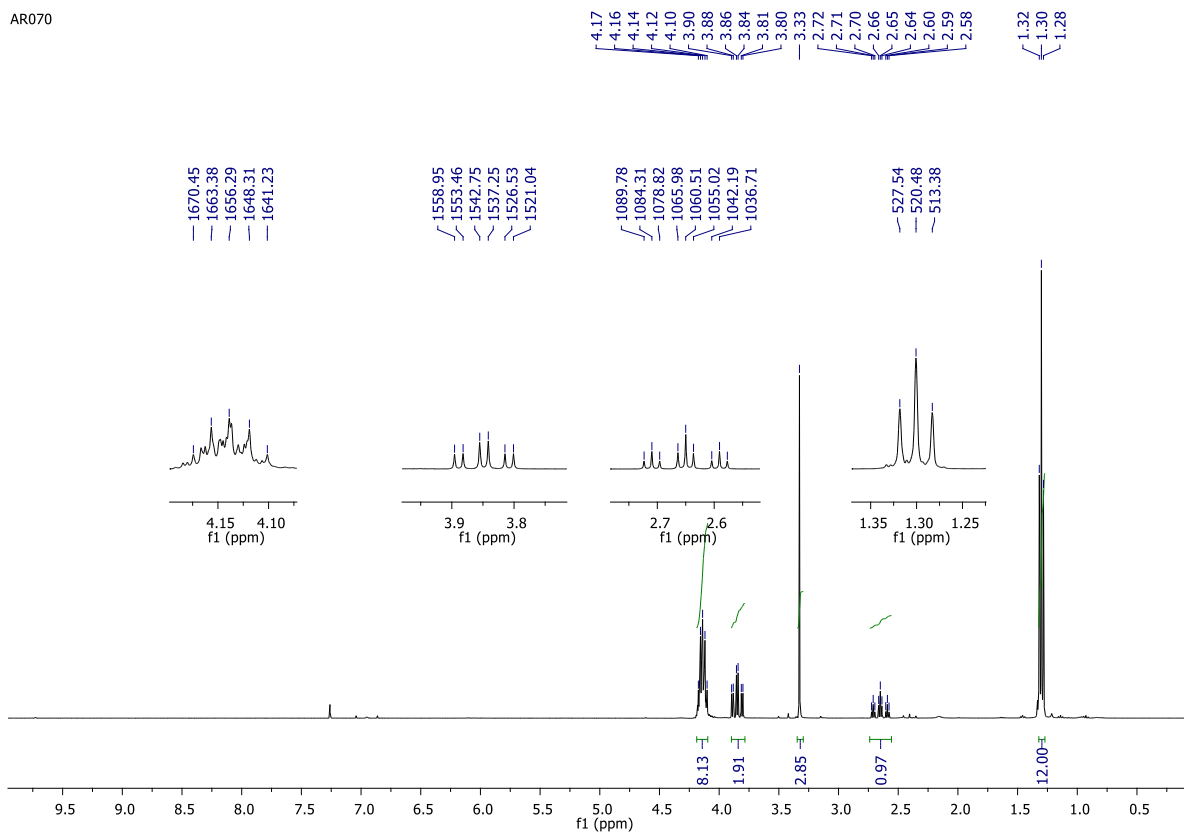
AR070

— 21.20

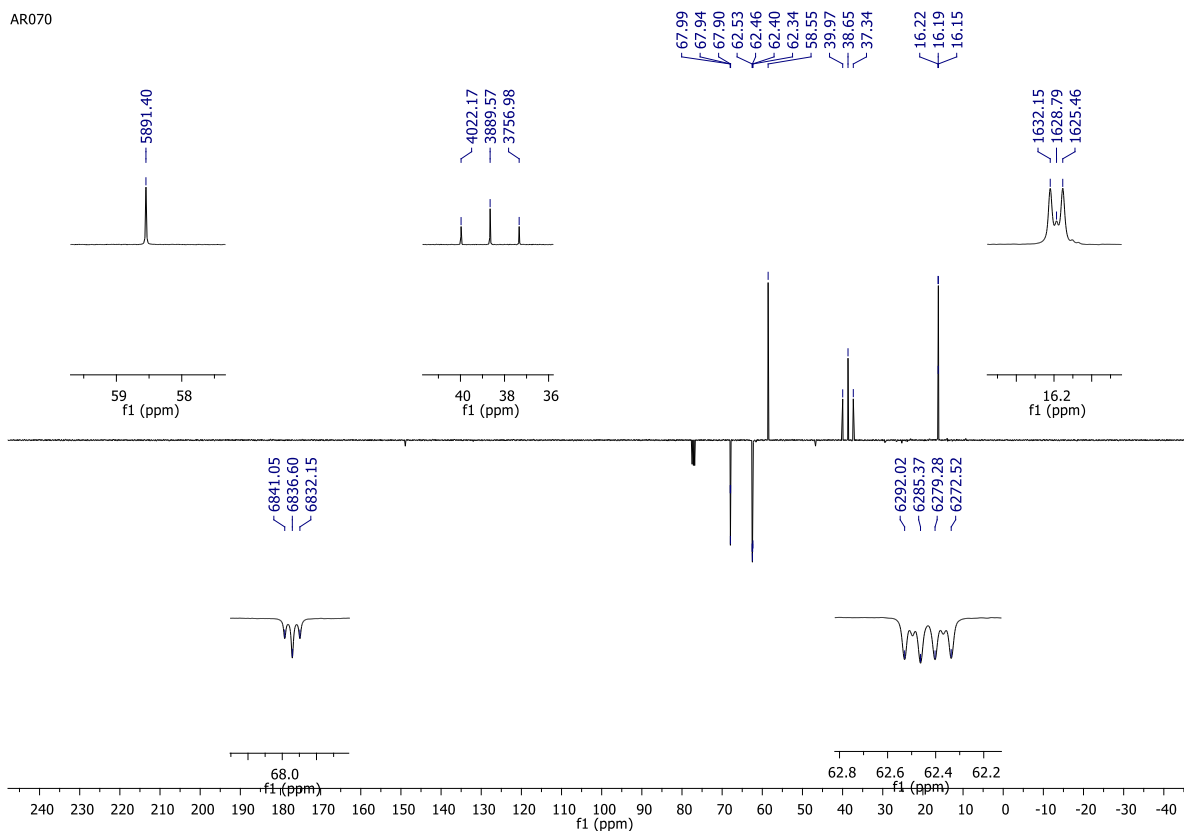
— 3434.87

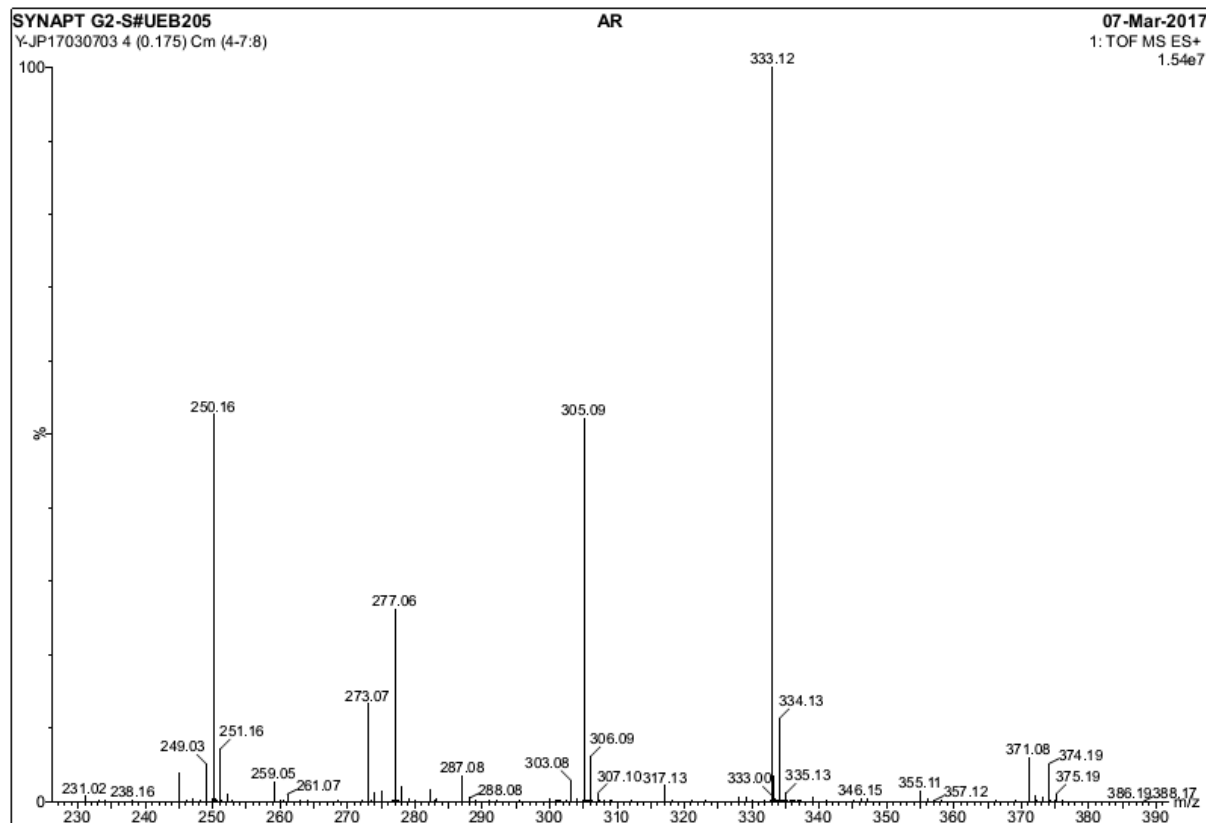
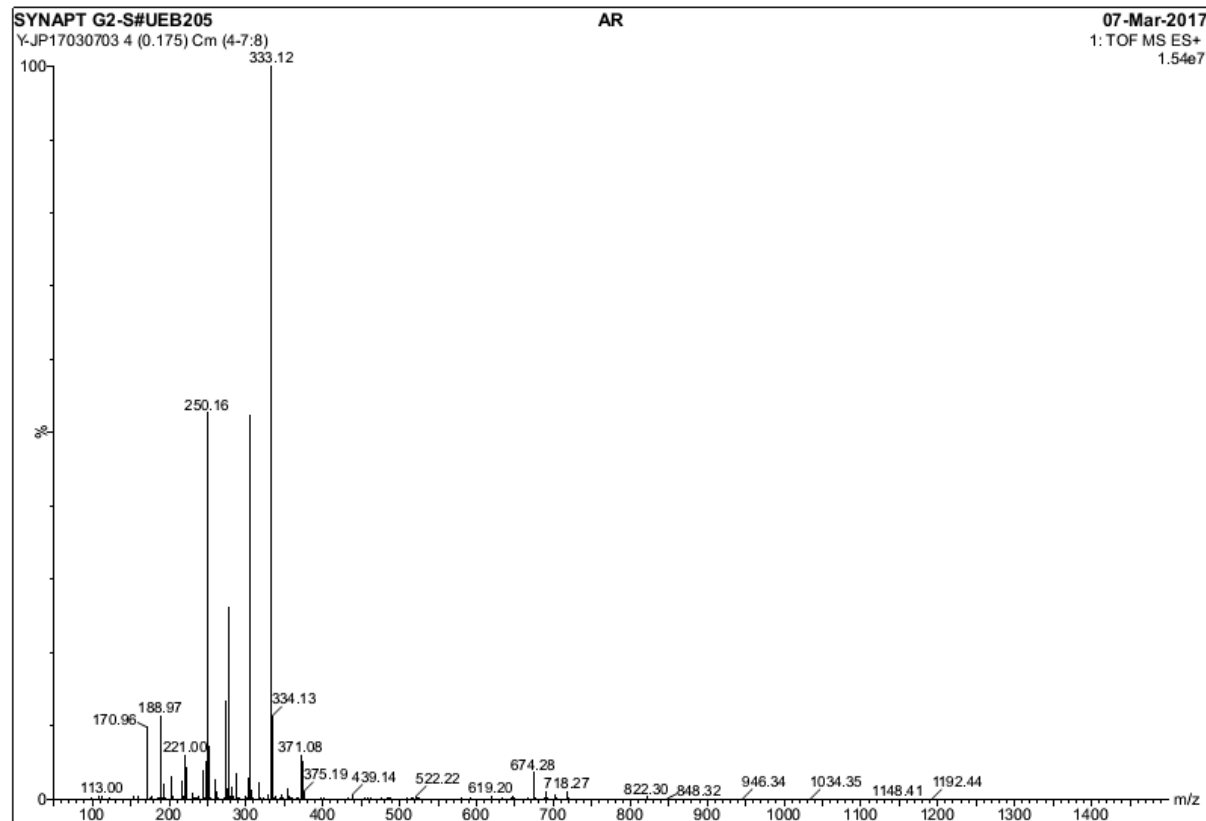
22.0 21.5 21.0 20.5
f1 (ppm)240 220 200 180 160 140 120 100 80 60 40 20 0 -10 -30 -50 -70 -90 -120
f1 (ppm)

AR070



AR070





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1098 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 1-2

SYNAPT G2-S#UEB205

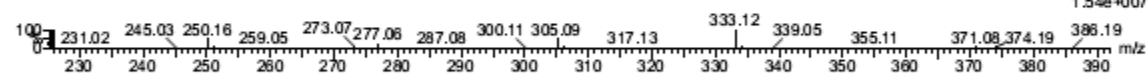
Y-IP17030703 4 (0.175) Cm (4-7:8)

AR

07-Mar-2017

1: TOF MS ES+

1.54e+007

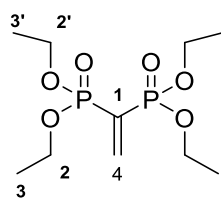


Minimum:

Maximum:

1.0 1.0 -1.5
50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
333.1232	333.1232	0.0	0.0	-0.5	1436.9	0.014	98.58	C11 H27 O7 P2
	333.1229	0.3	0.9	9.5	1441.2	4.257	1.42	C14 H18 N6 O2 P

S5: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl ethene-1,1-diylbis(phosphonate) (5)

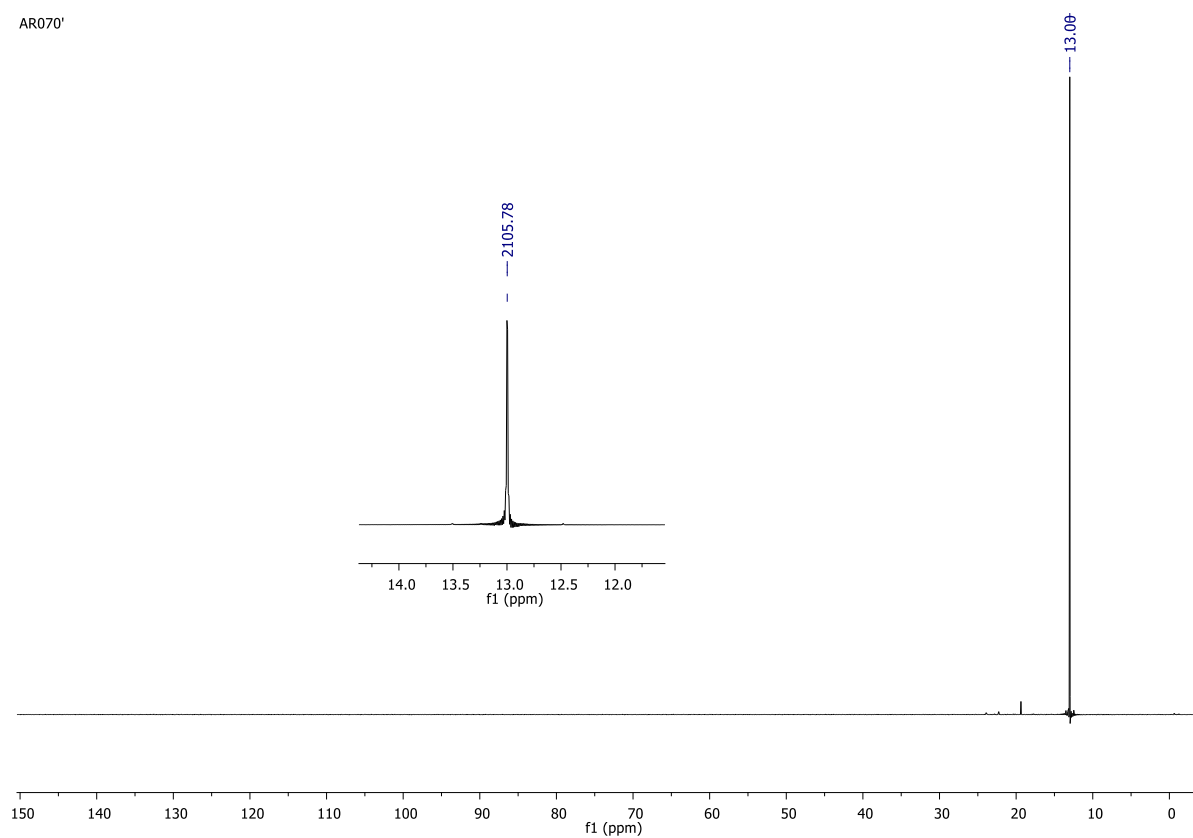
tetraethyl ethene-1,1-diylbis(phosphonate)

 $\text{C}_{10}\text{H}_{22}\text{O}_6\text{P}_2$

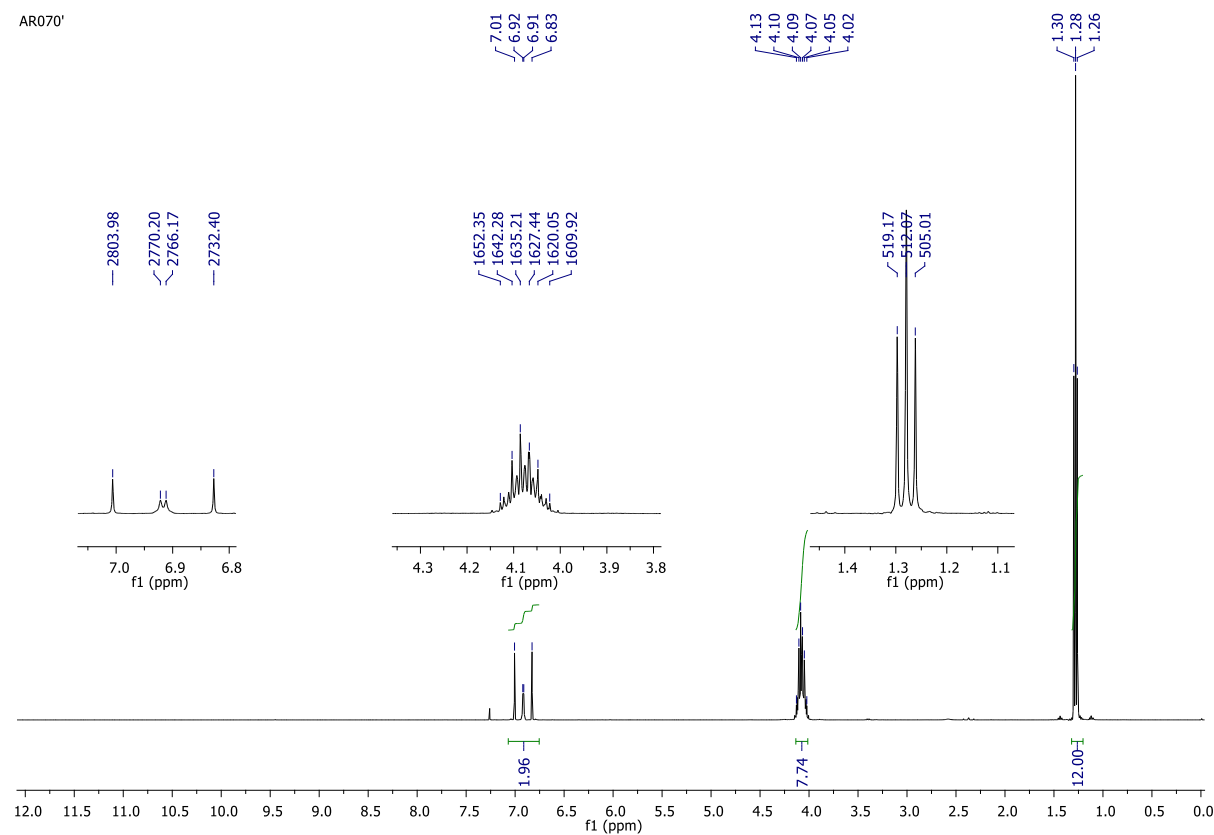
300,23 g/mol

Colorless oil

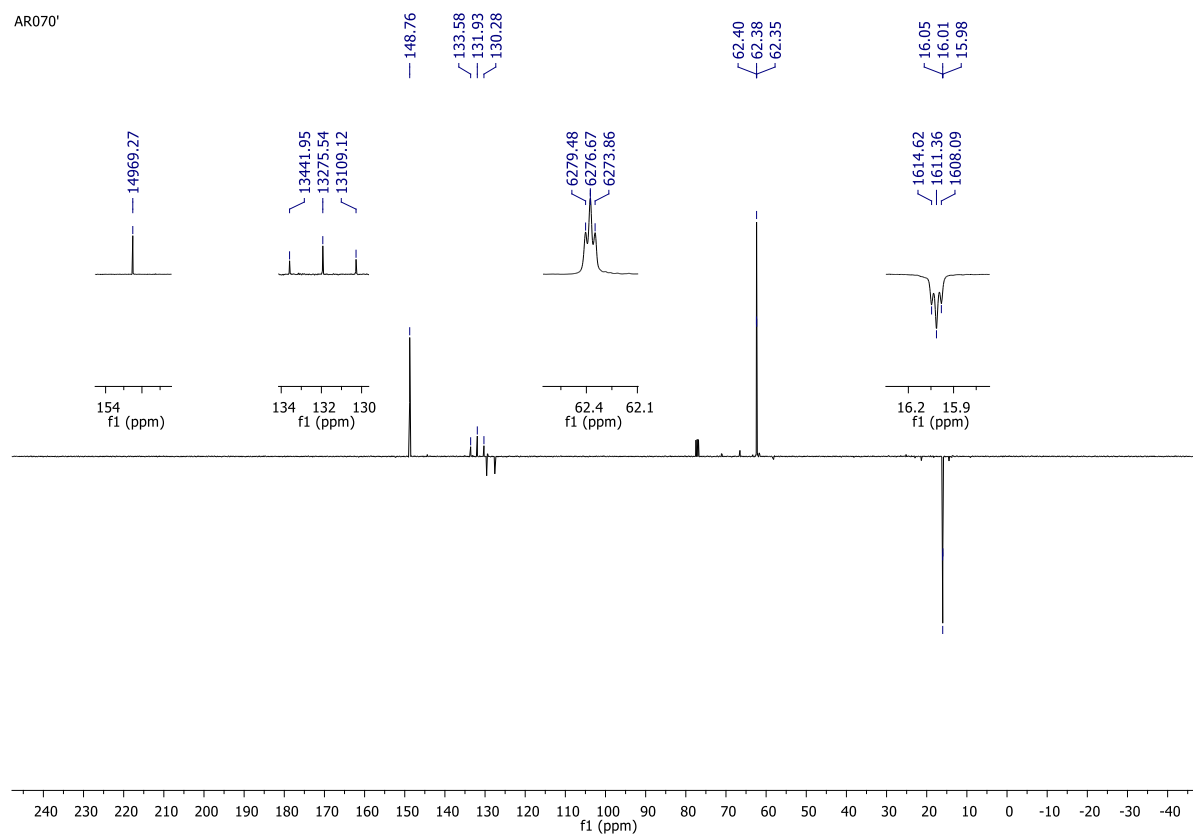
AR070'

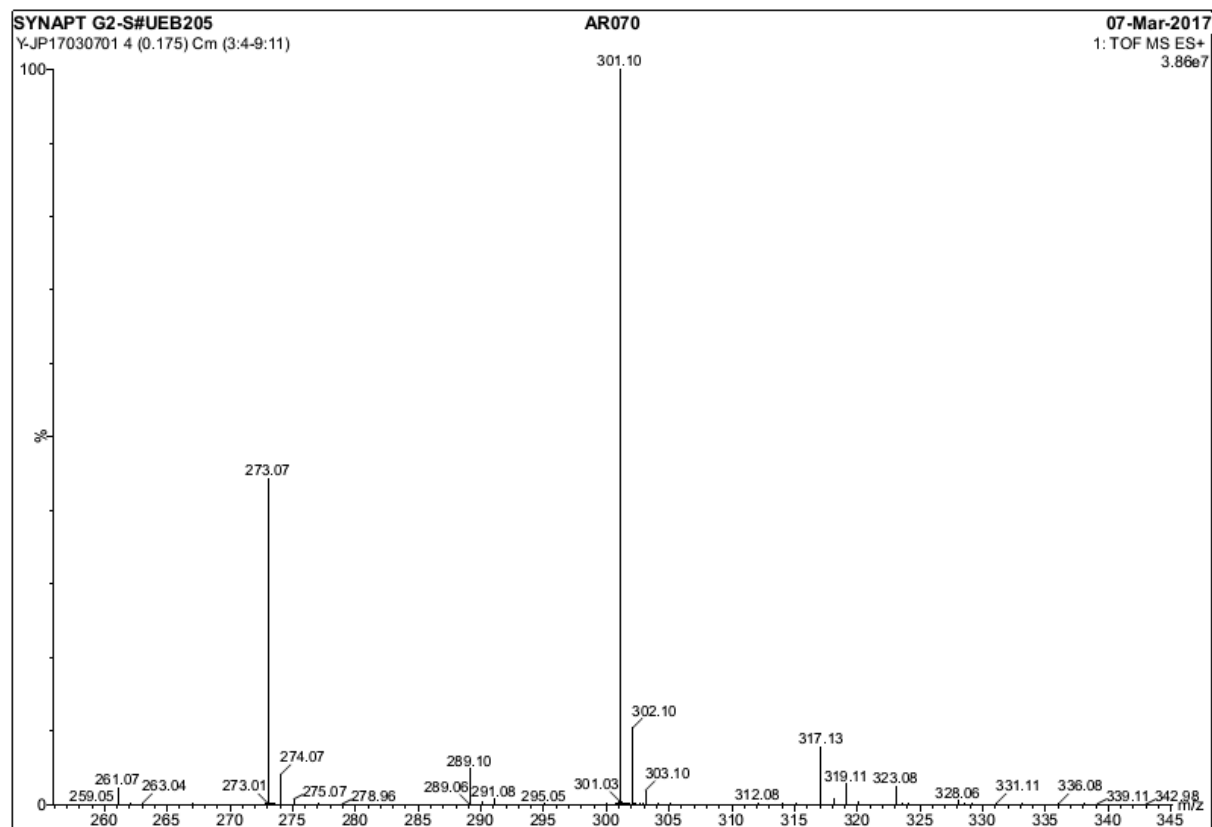
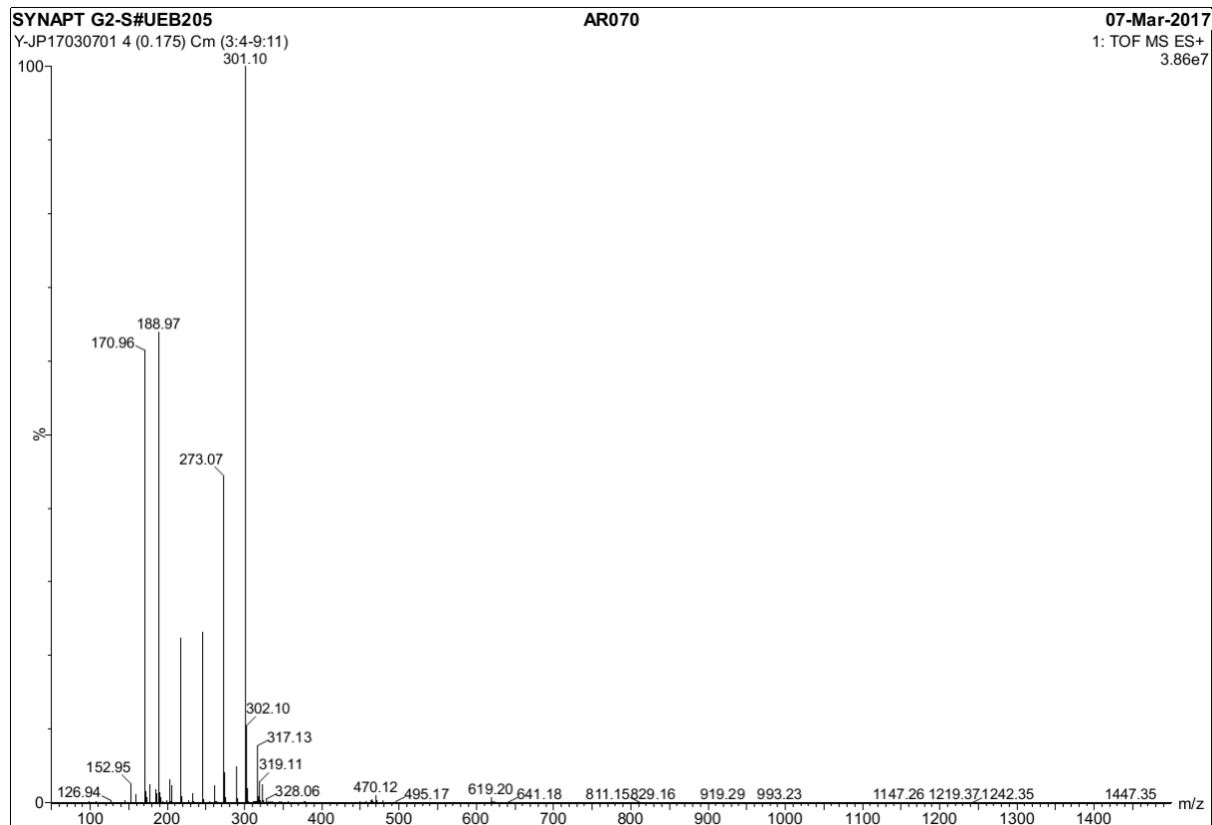


AR070'



AR070'





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

829 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 1-2

SYNAPT G2-SMUEB205

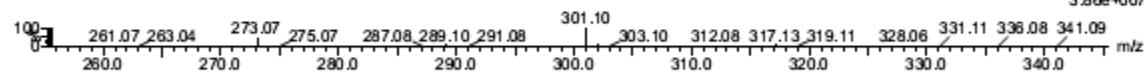
Y-IP17030701 4 (0.175) Cm (3:4-9:11)

AR070

07-Mar-2017

1: TOF MS ES+

3.86e+007

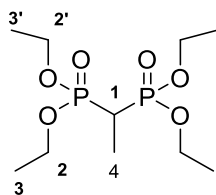


Minimum:

Maximum:

		-1.5
1.0	1.0	50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
301.0969	301.0970	-0.1	-0.3	0.5	2207.2	0.000	100.00	C10 H23 O6 P2
	301.0967	0.2	0.7	10.5	2218.0	10.826	0.00	C13 H14 N6 O P

S6: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl ethane-1,1-diylbis(phosphonate) (2a)

tetraethyl ethane-1,1-diylbis(phosphonate)

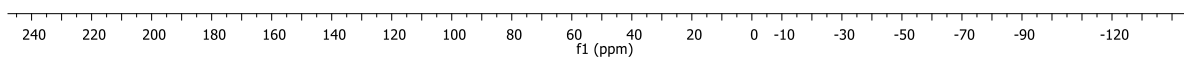
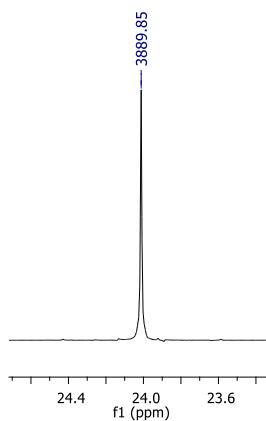
 $\text{C}_{10}\text{H}_{24}\text{O}_6\text{P}_2$

302,24 g/mol

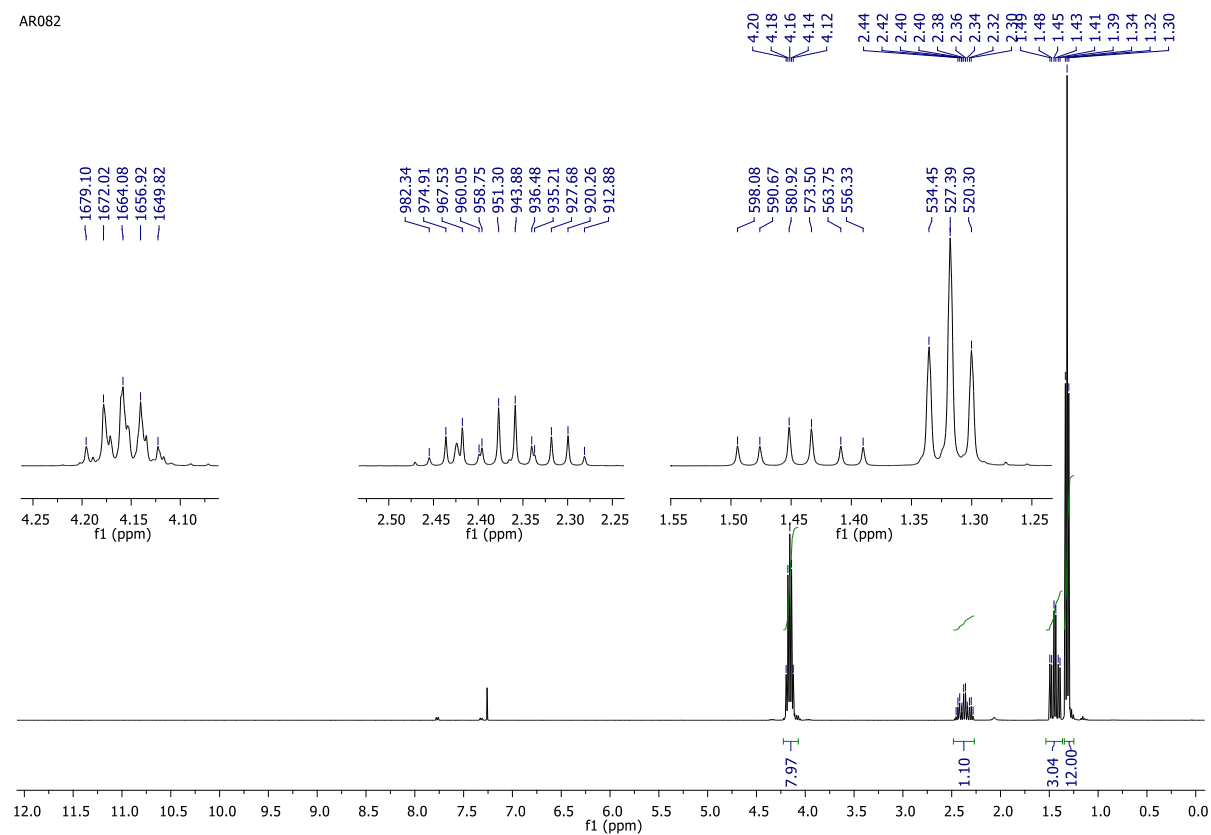
Colorless oil

AR082

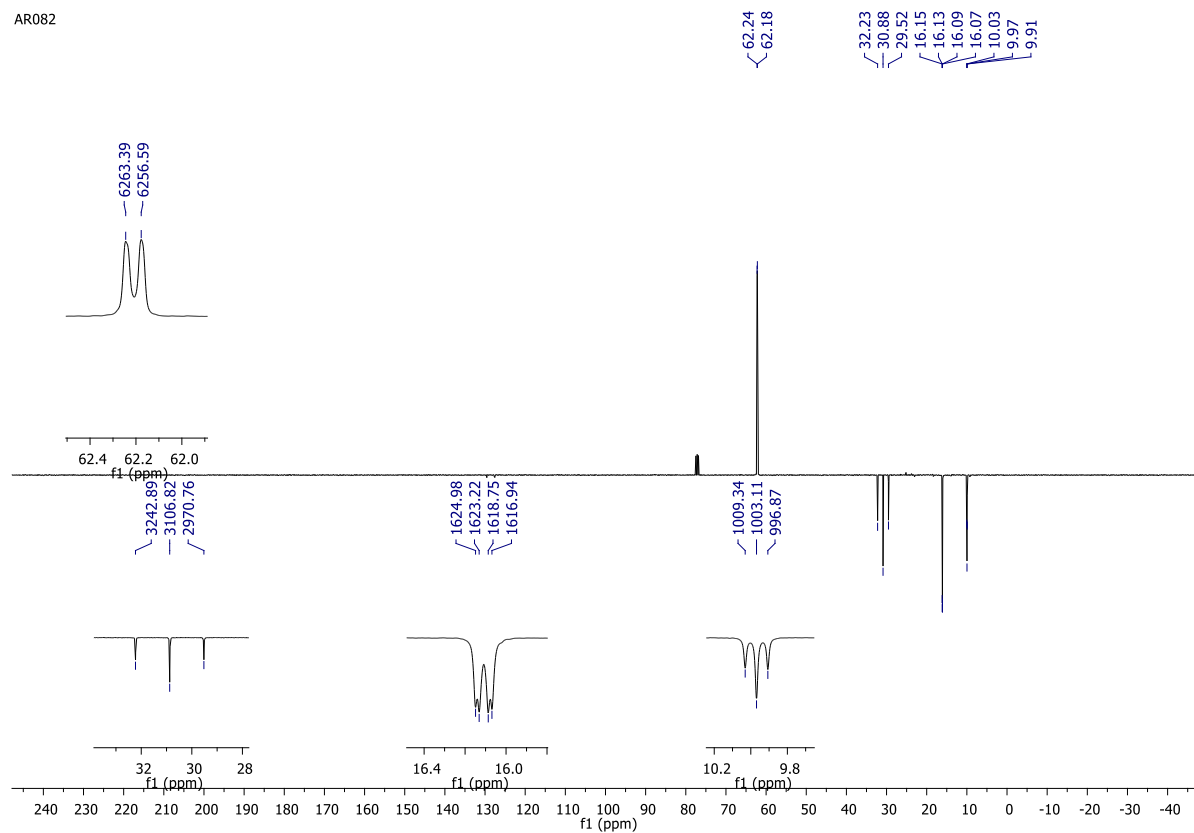
— 24.01

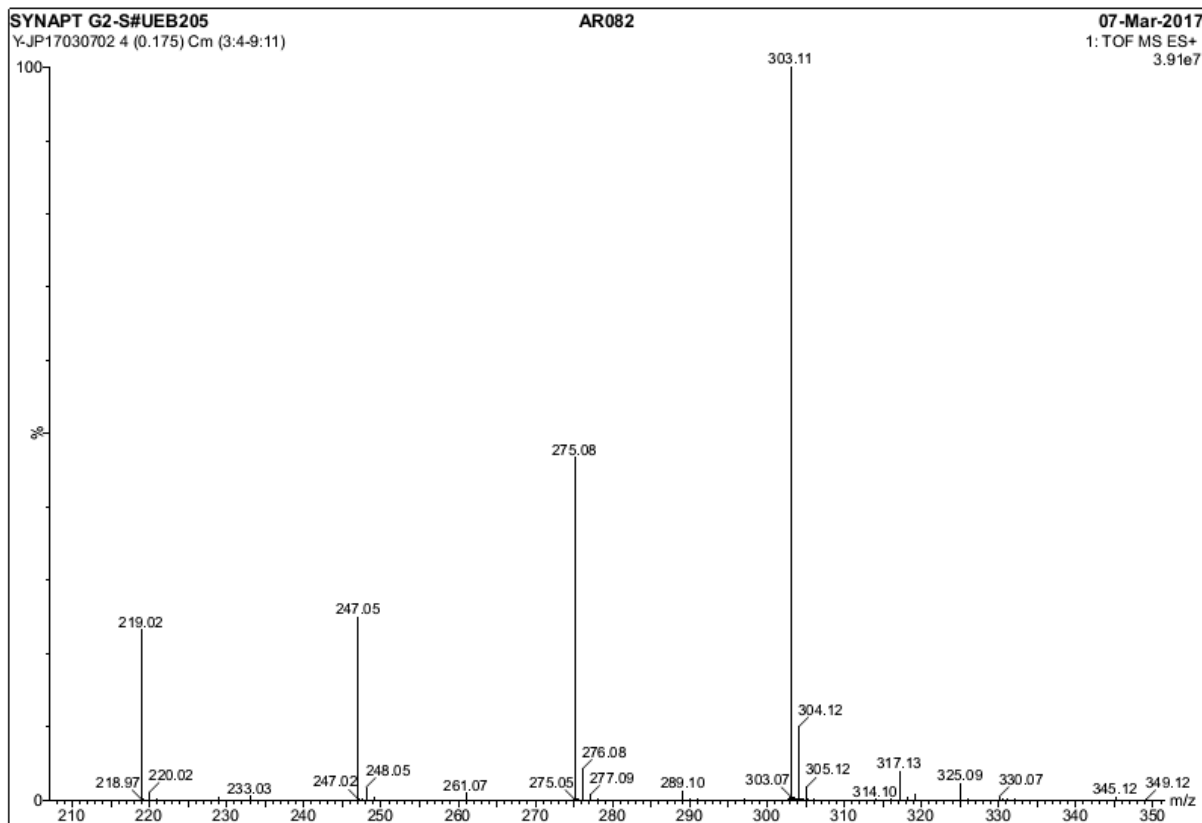
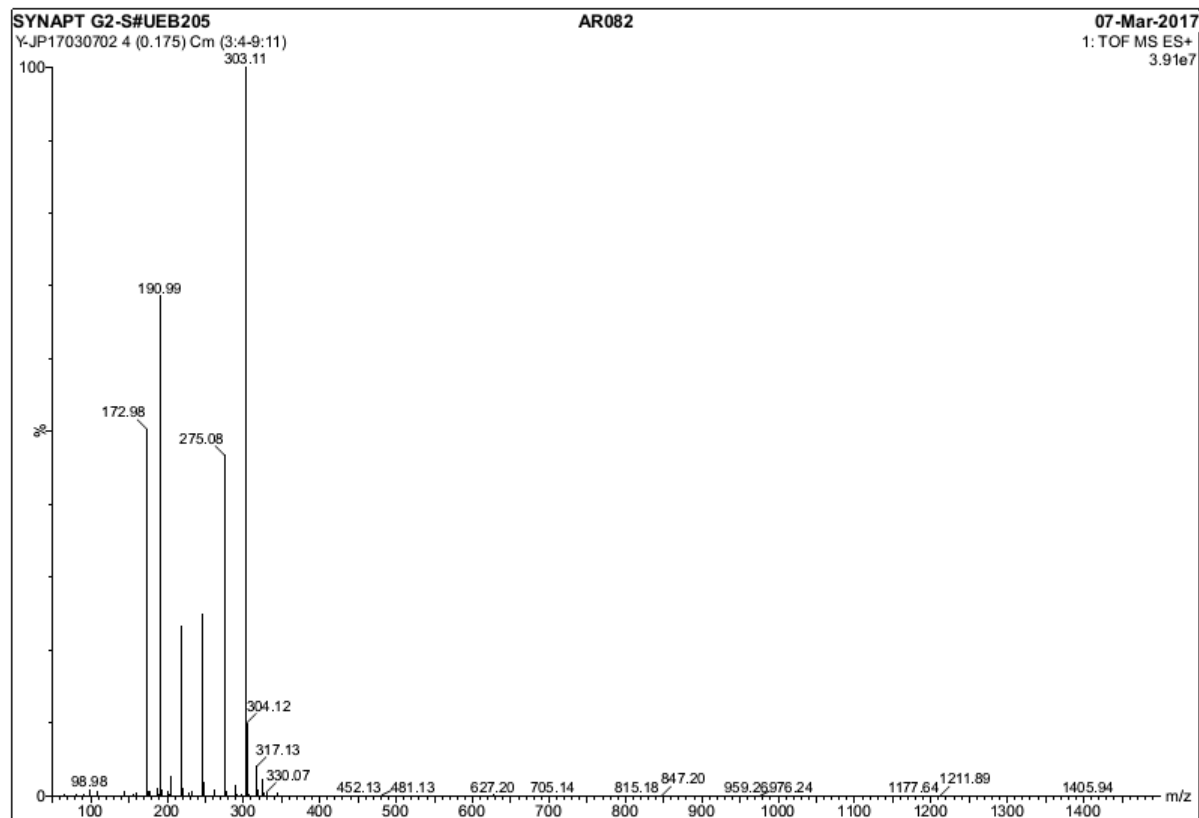


AR082



AR082





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

839 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 1-2

SYNAPT G2-SAMUEB205

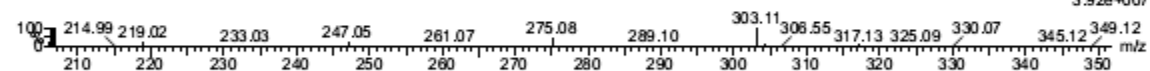
YJP17030702 4 (0.175) Cm (34-9:11)

AR082

07-Mar-2017

1: TOF MS ES+

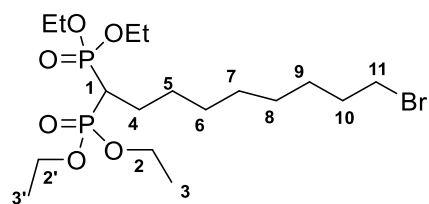
3.92e+007



Minimum:

Maximum:

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
303.1127	303.1126	0.1	0.3	-0.5	1995.2	0.000	99.97	C10 H25 O6 P2
	303.1123	0.4	1.3	9.5	2003.4	8.238	0.03	C13 H16 N6 O P

S7: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl (9-bromononane-1,1-diyl)bis(phosphonate) (2c)

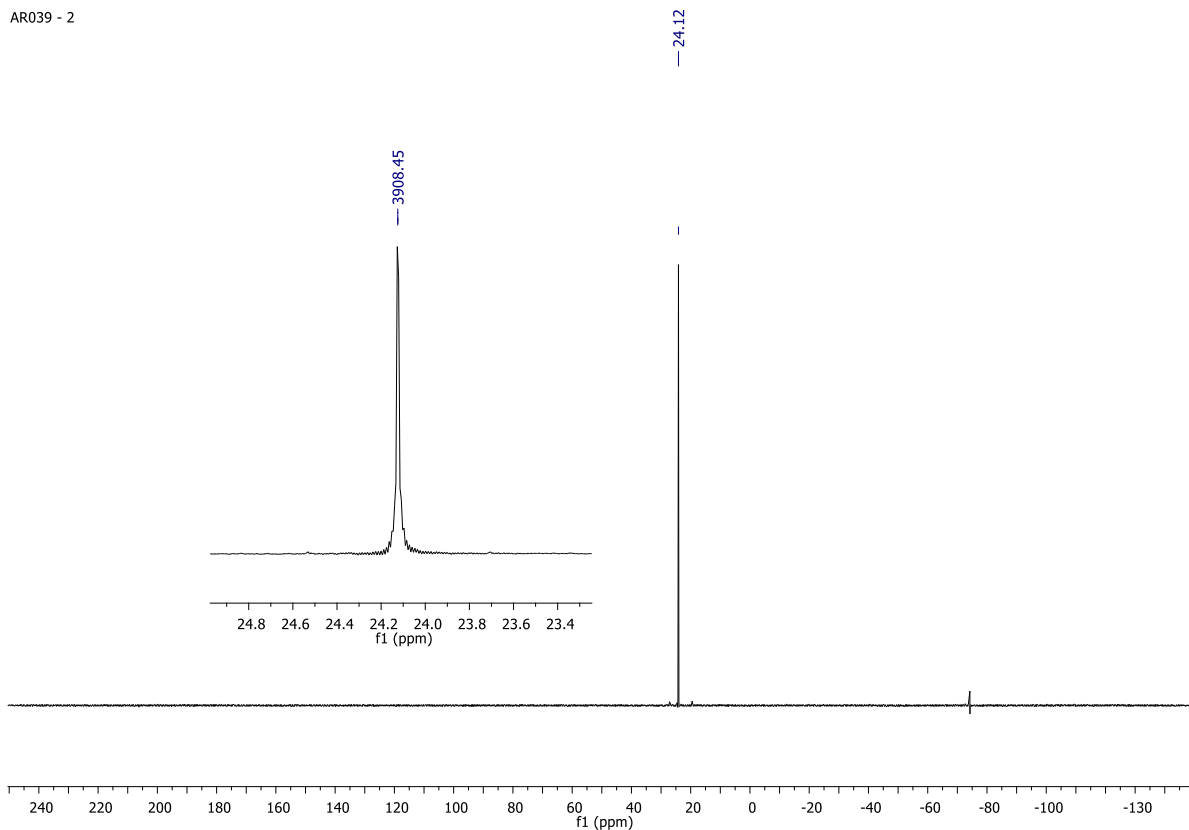
tetraethyl(9-bromononane-1,1-diyl)bis(phosphonate)

 $\text{C}_{17}\text{H}_{37}\text{O}_6\text{BrP}_2$

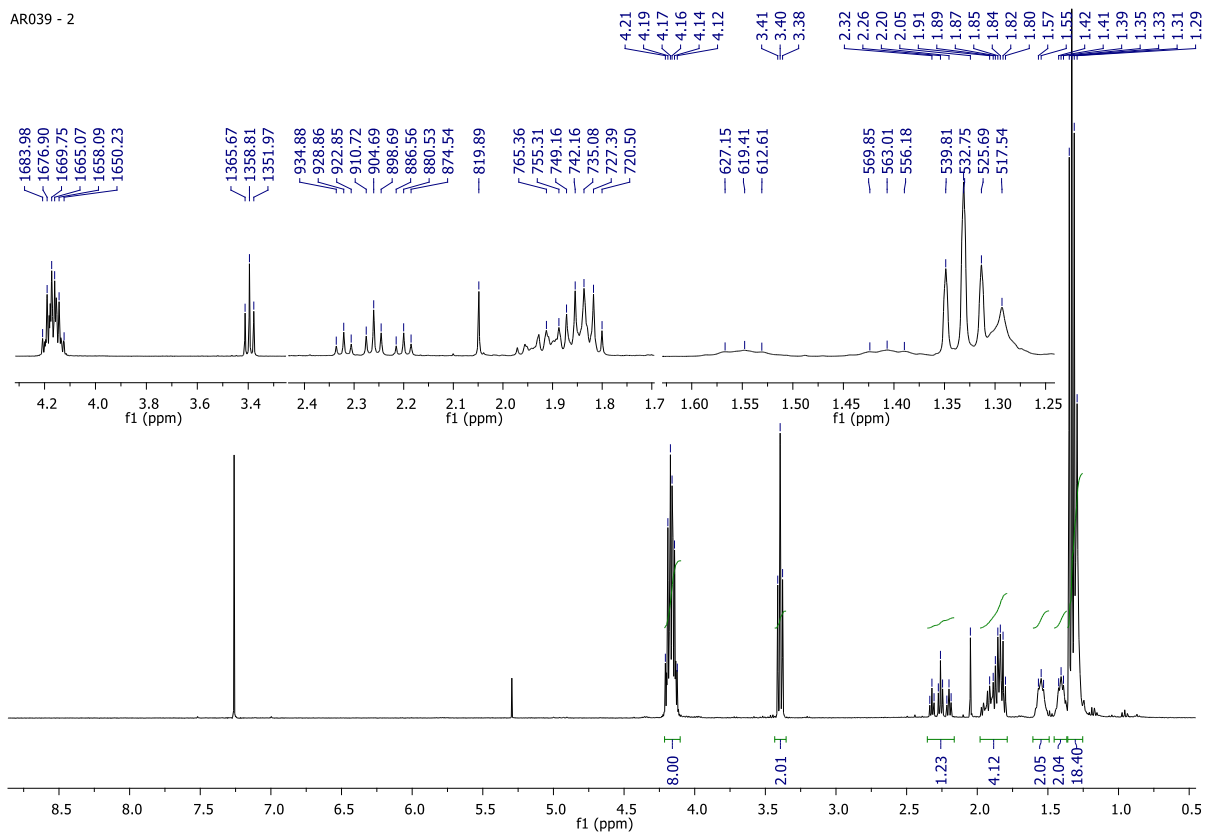
479,33 g/mol

Colorless oil

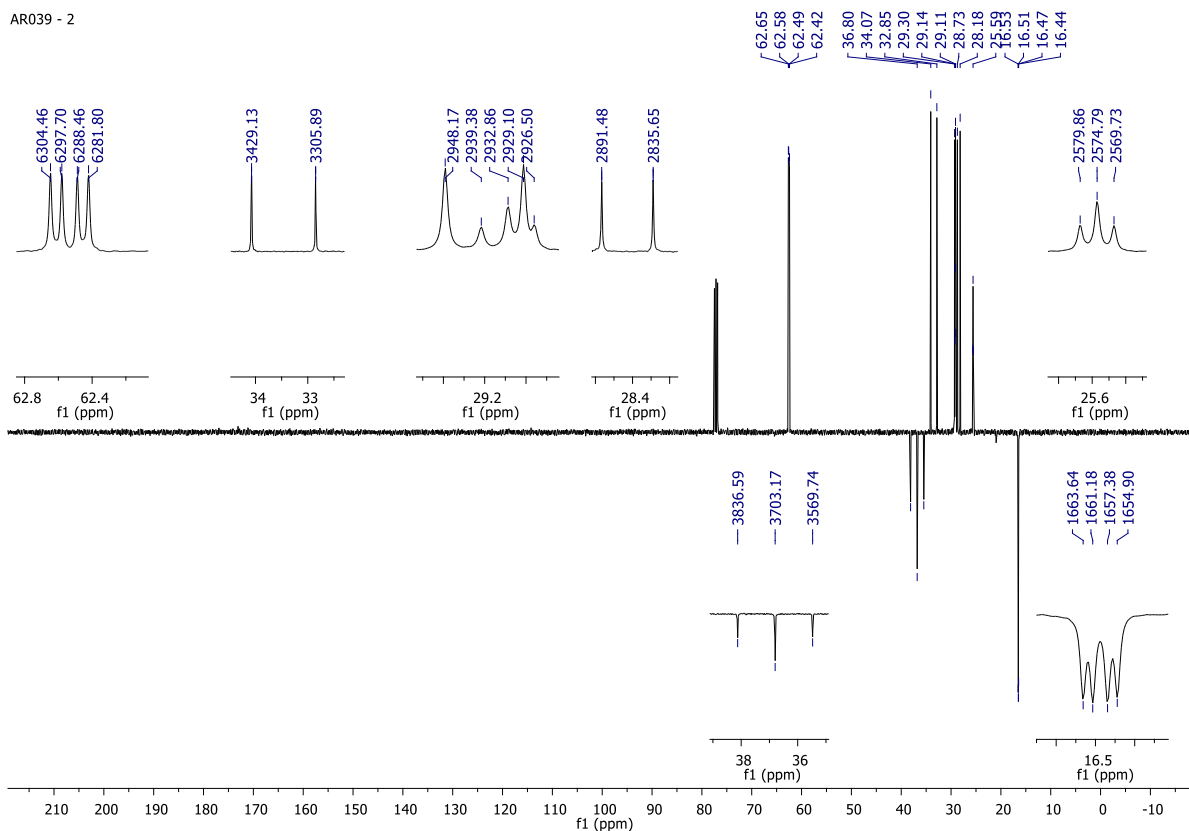
AR039 - 2

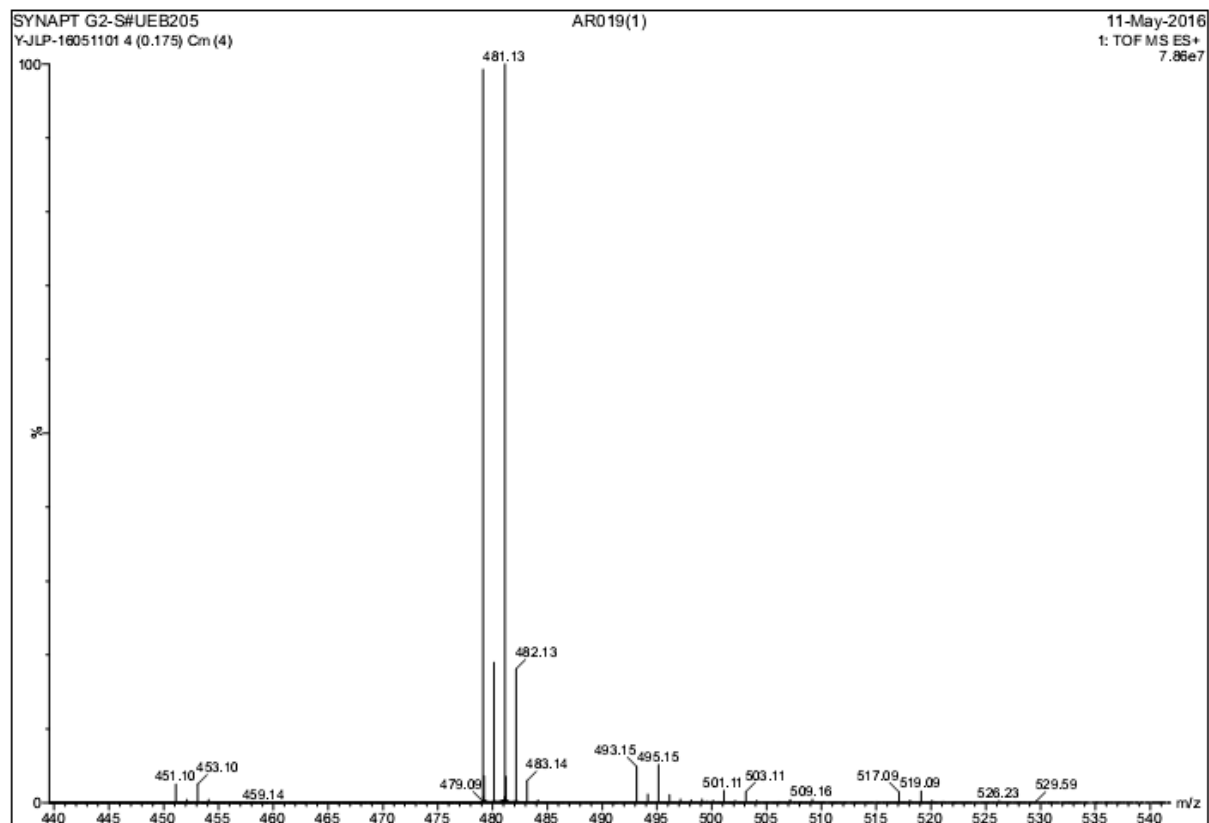
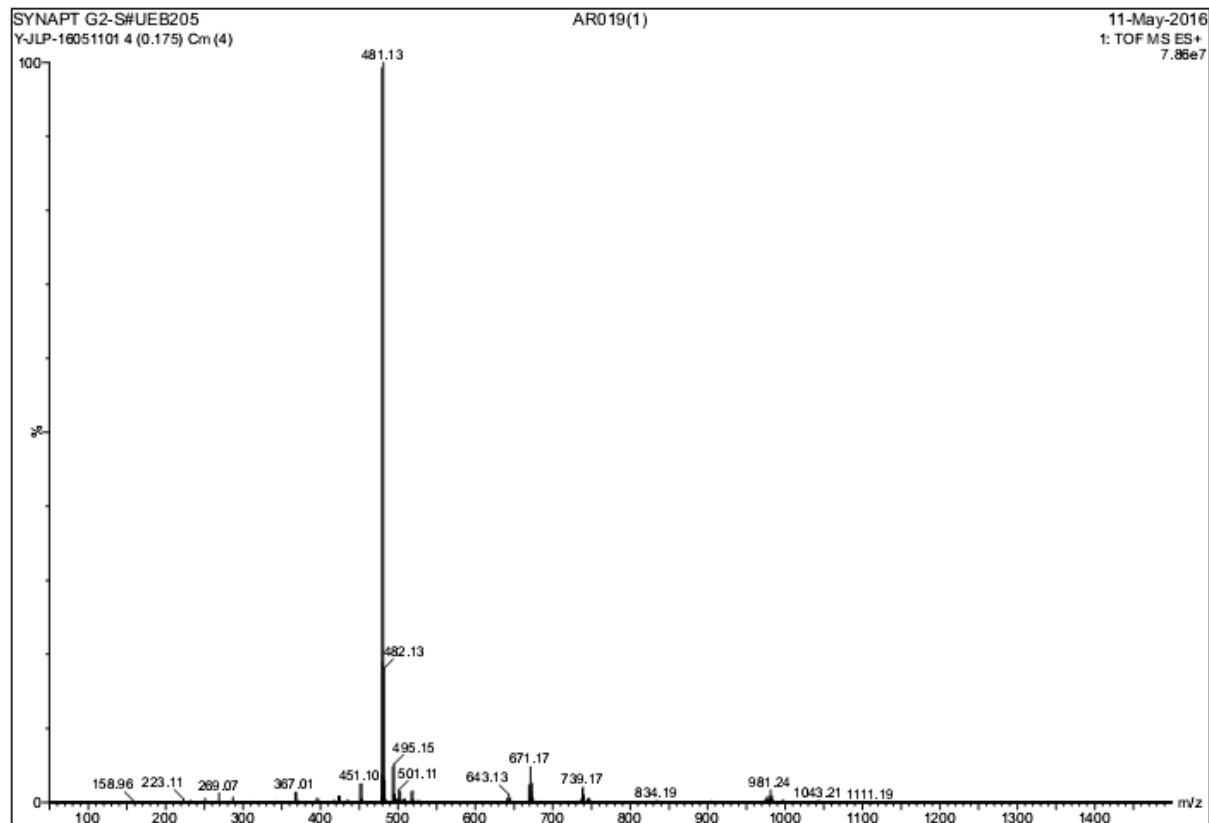


AR039 - 2



AR039 - 2





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 2.0 PPM / DBE: min = -50.0, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2560 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-100 H: 0-150 N: 0-10 O: 0-10 P: 1-2 Br: 1-1

SYNAPT G2-S#UEB205

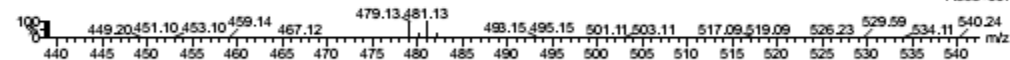
AR019(1)

11-May-2016

YJLP-16051101 4 (0.175) Cm (4)

1: TOF MS ES+

7.86e+007



Minimum:

Maximum:

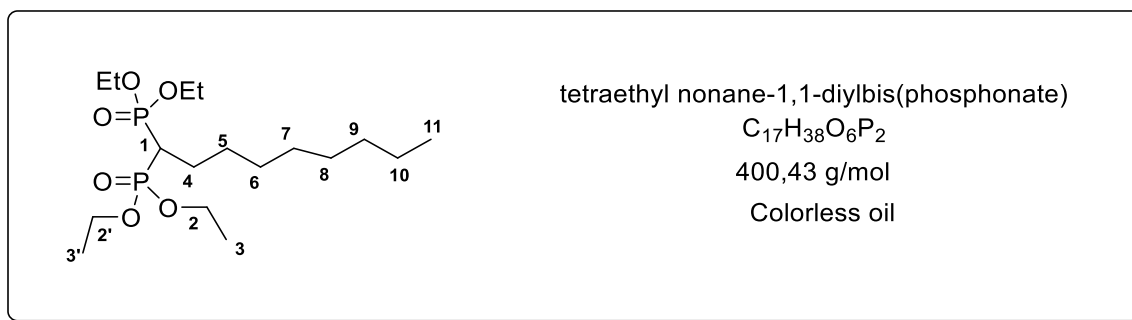
1.1

2.0

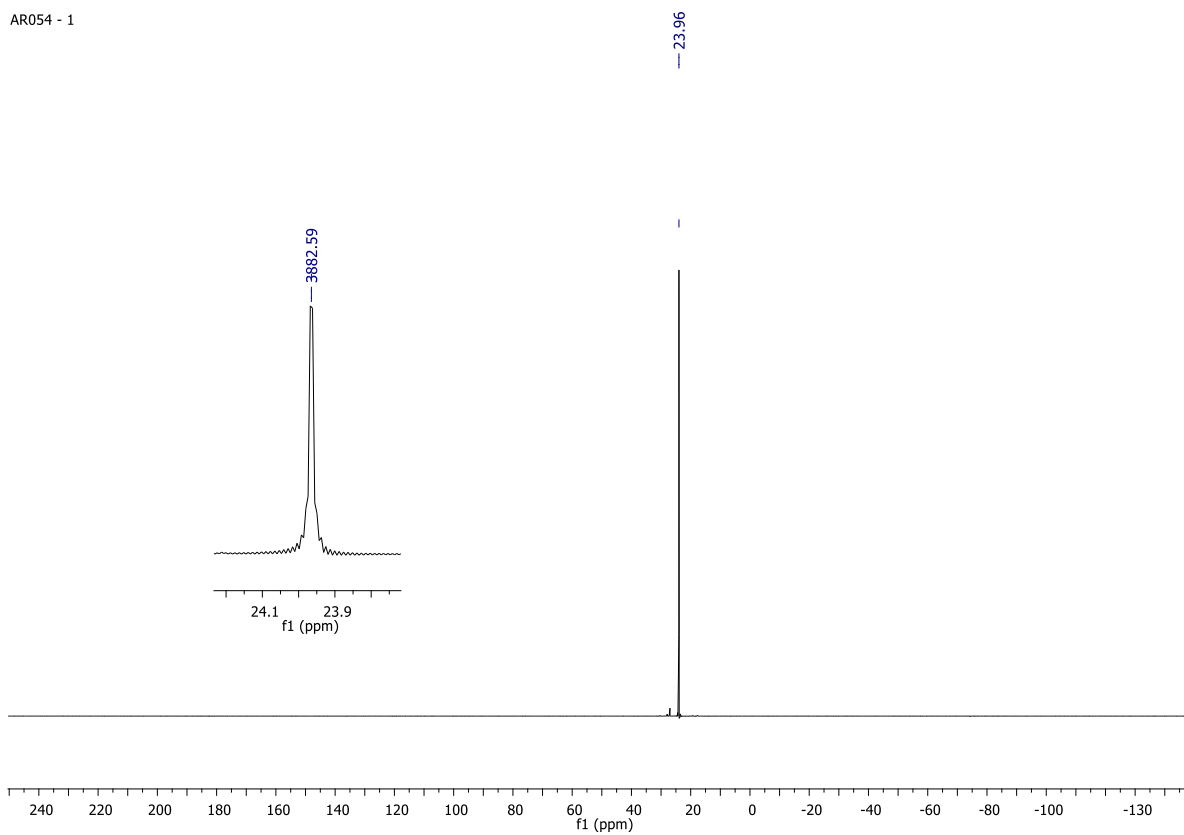
-50.0

100.0

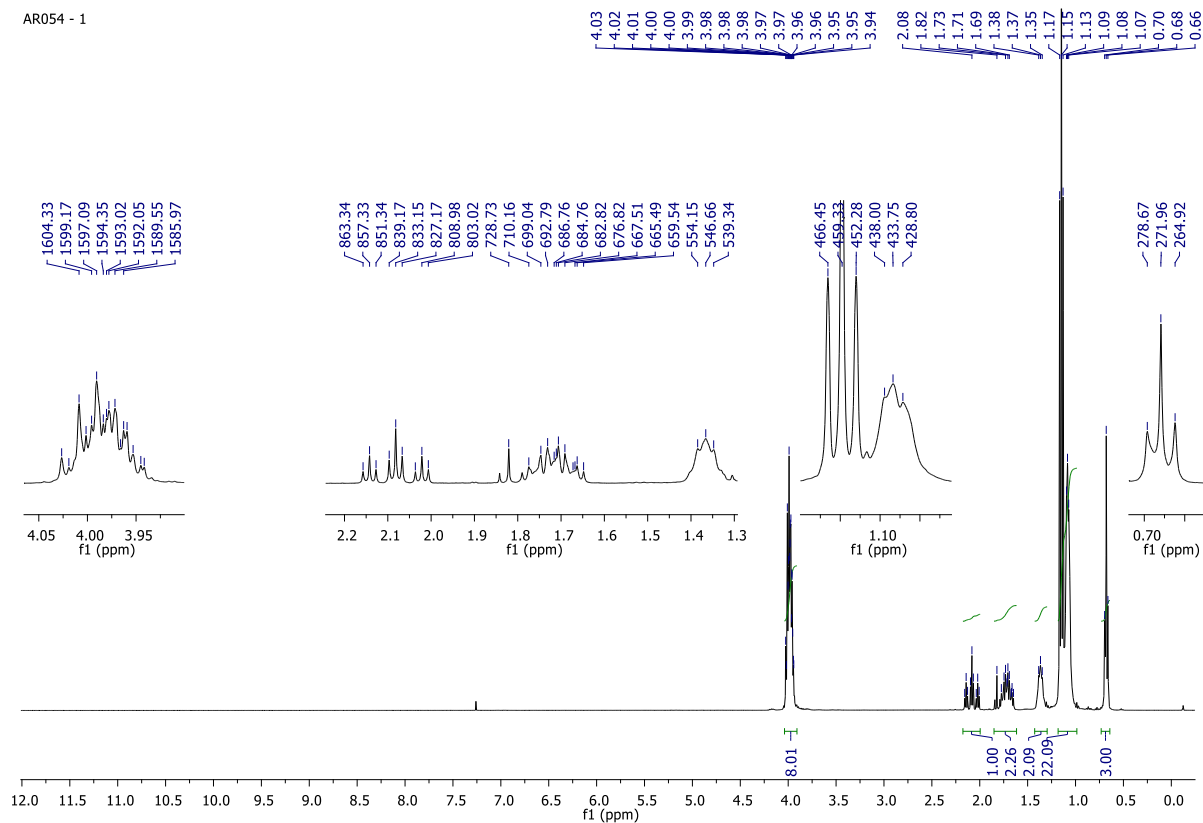
Mass	Calc. Mass	mDa	PM	DBE	i-FIT	Norm	Conf (%)	Formula
479.1328	479.1327	0.1	0.2	-0.5	1653.9	0.000	100.00	C17 H38 O6 P2 Br
	479.1324	0.4	0.8	9.5	1669.5	15.690	0.00	C20 H29 N6 O P Br

S8: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl nonane-1,1-diylbis(phosphonate) (2d)

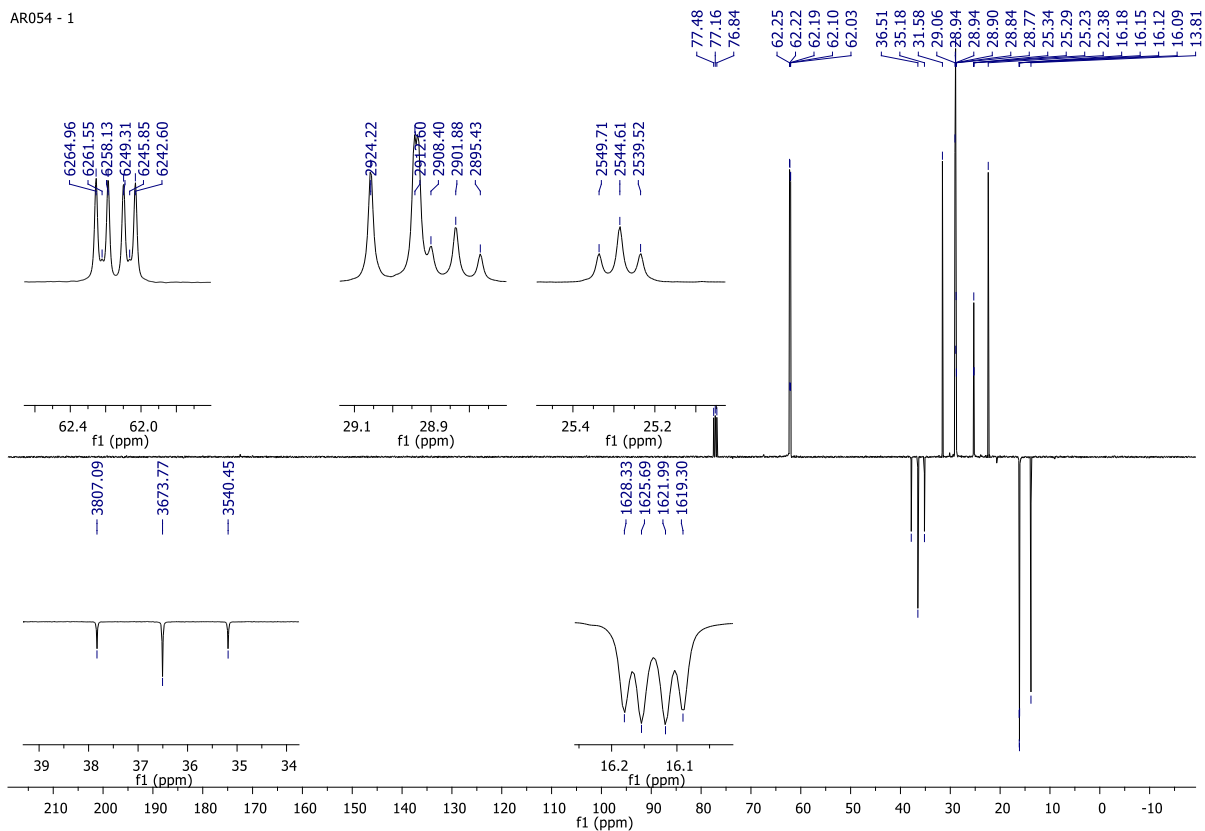
AR054 - 1

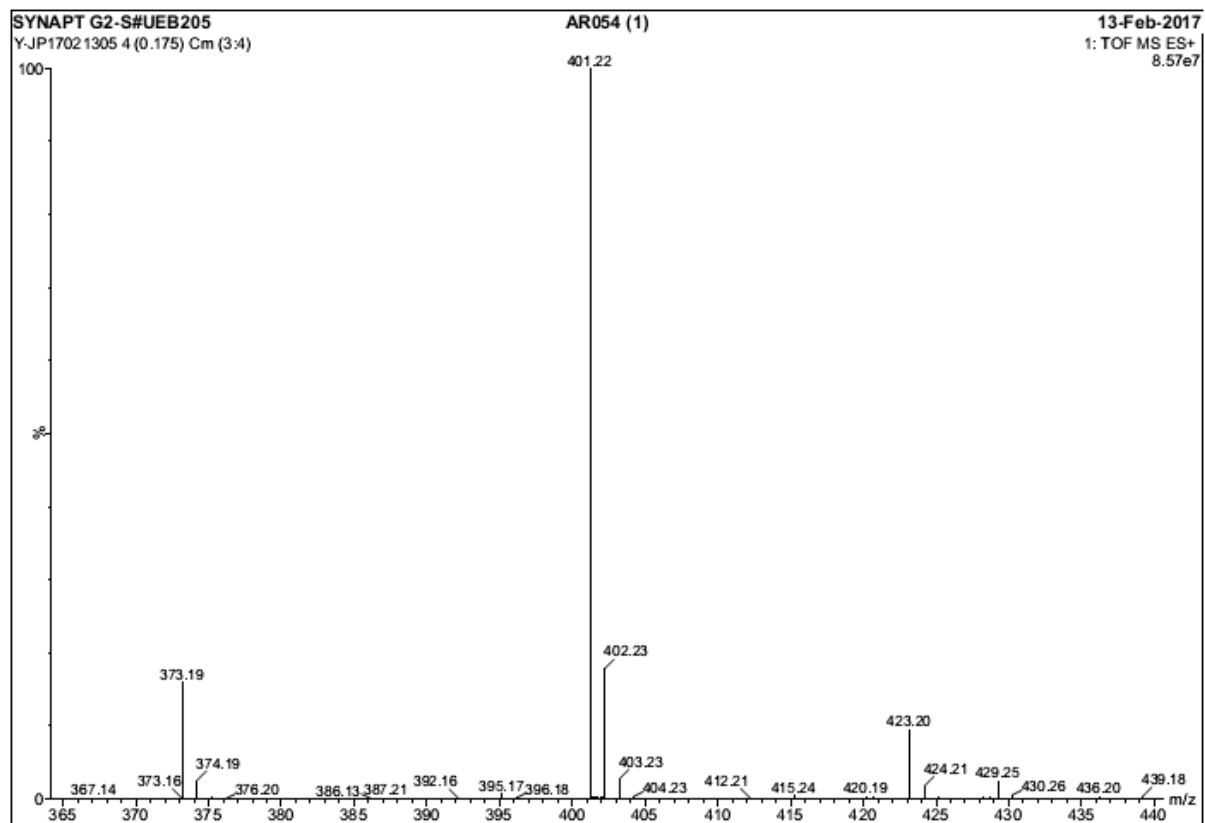
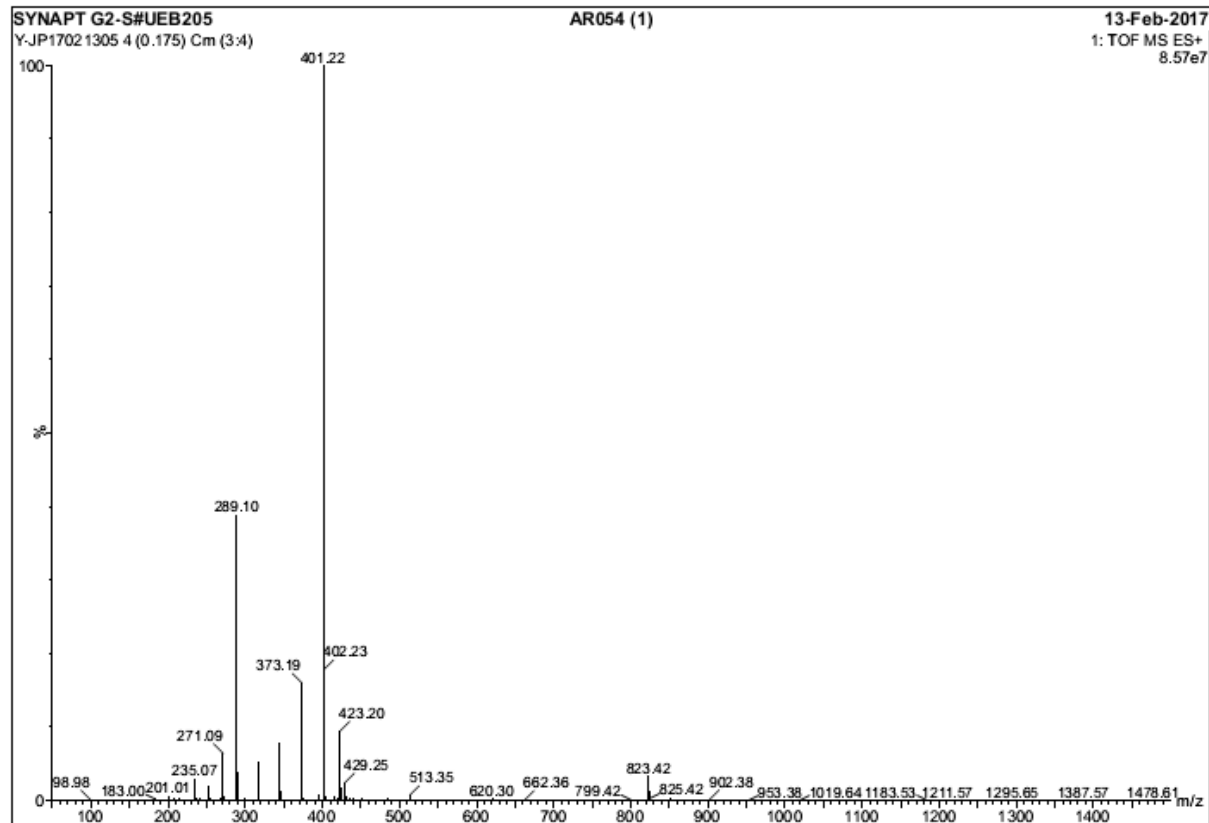


AR054 - 1



AR054 - 1





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

841 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 2-2

SYNAPT G2-S#UEB205

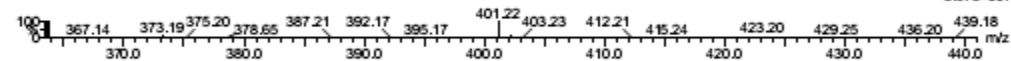
Y-JP17021305 4 (0.175) Cm (3c4)

AR054 (1)

13-Feb-2017

1: TOF MS ES+

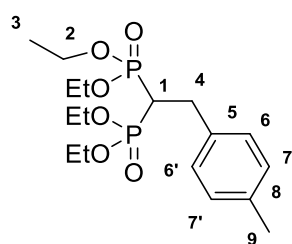
8.57e+007



Minimum: -1.5

Maximum: 10.0 1.0 50.0

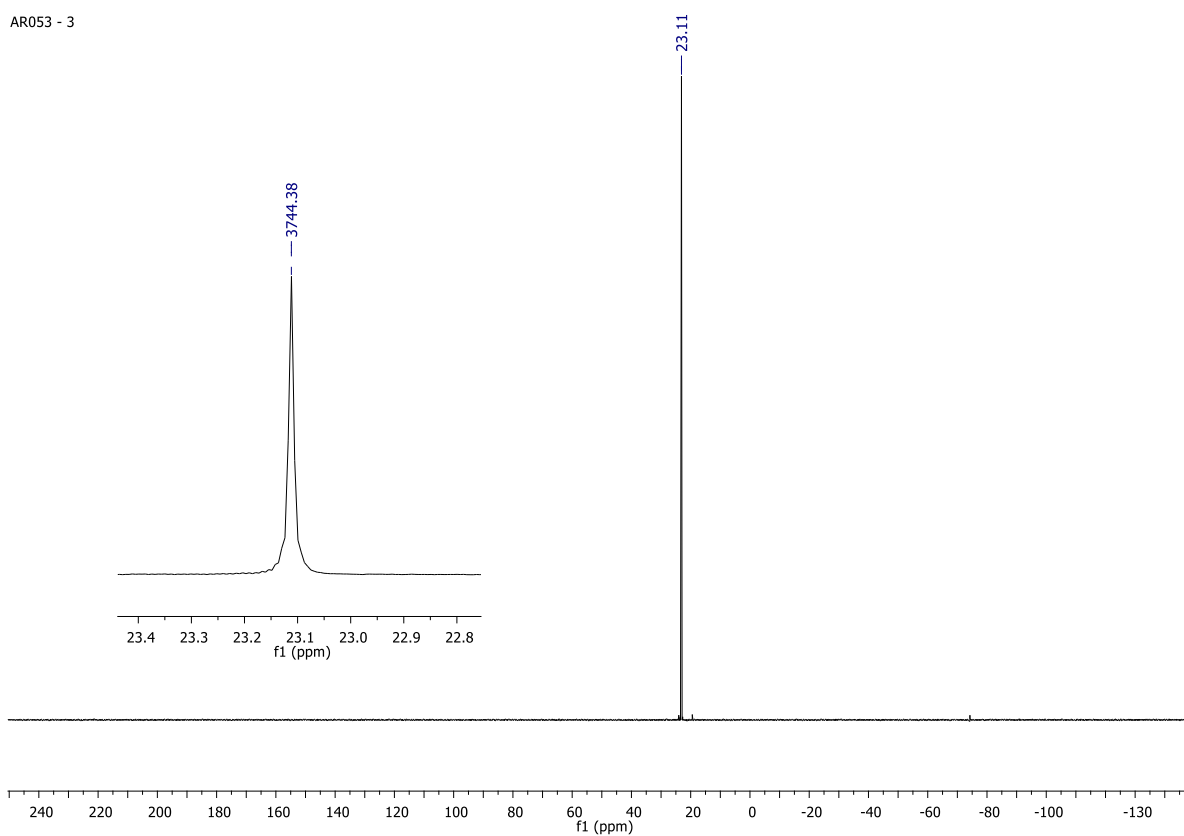
Mass	Calc. Mass	mDa	PM	DBE	i-FIT	Norm	Conf (%)	Formula
401.2219	401.2222	-0.3	-0.7	-0.5	2057.4	n/a	n/a	C17 H39 O6 P2

S9: ^{31}P , ^1H and ^{13}C NMR spectra and mass analysis of tetraethyl (2-(*p*-tolyl)ethane-1,1-diyl)bis(phosphonate) (2e)tetraethyl (2-(*p*-tolyl)ethane-1,1-diyl)bis(phosphonate) $\text{C}_{17}\text{H}_{30}\text{O}_6\text{P}_2$

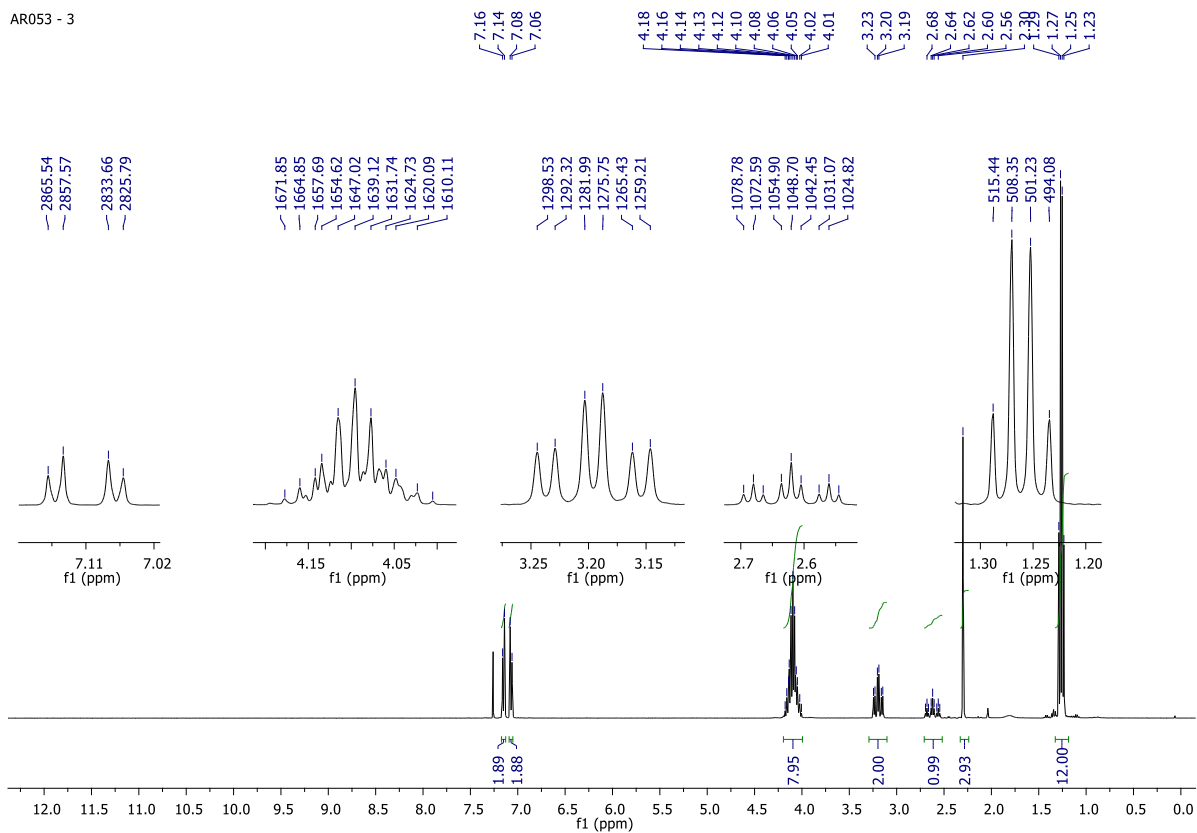
392,37 g/mol

Yellow oil

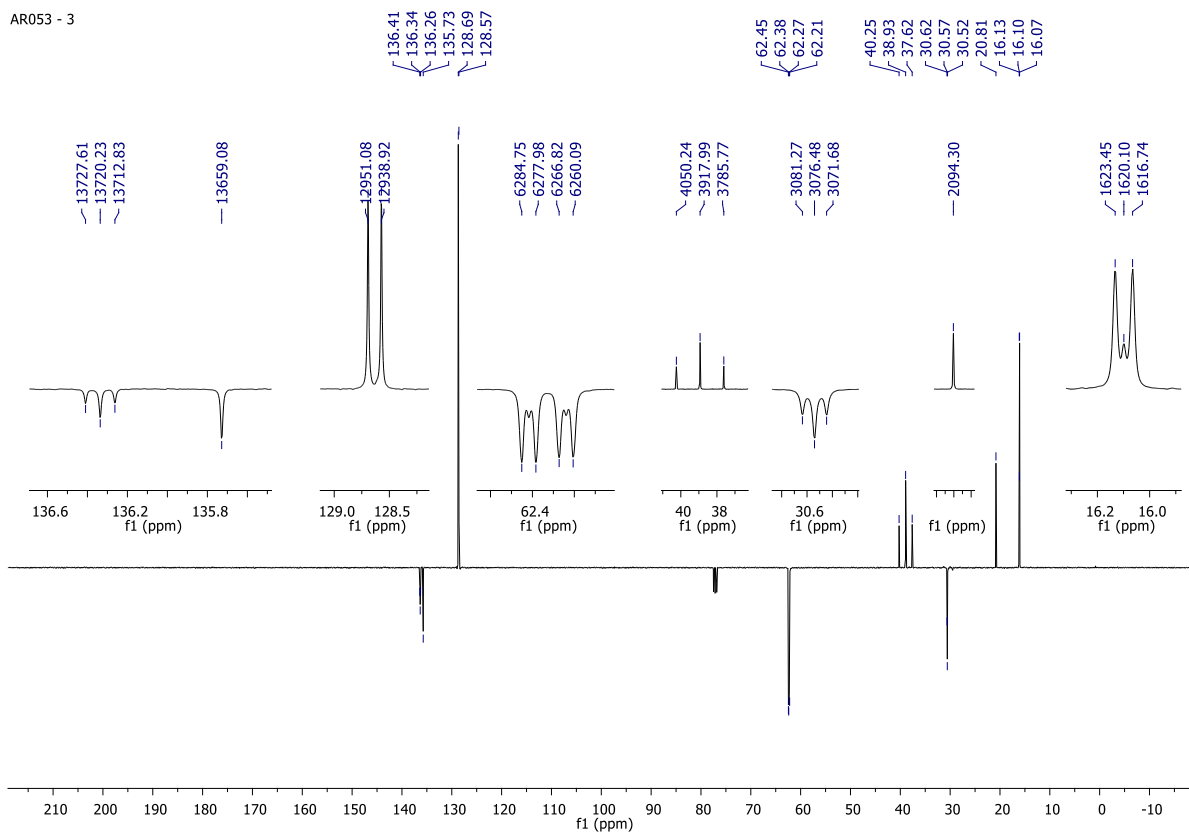
AR053 - 3

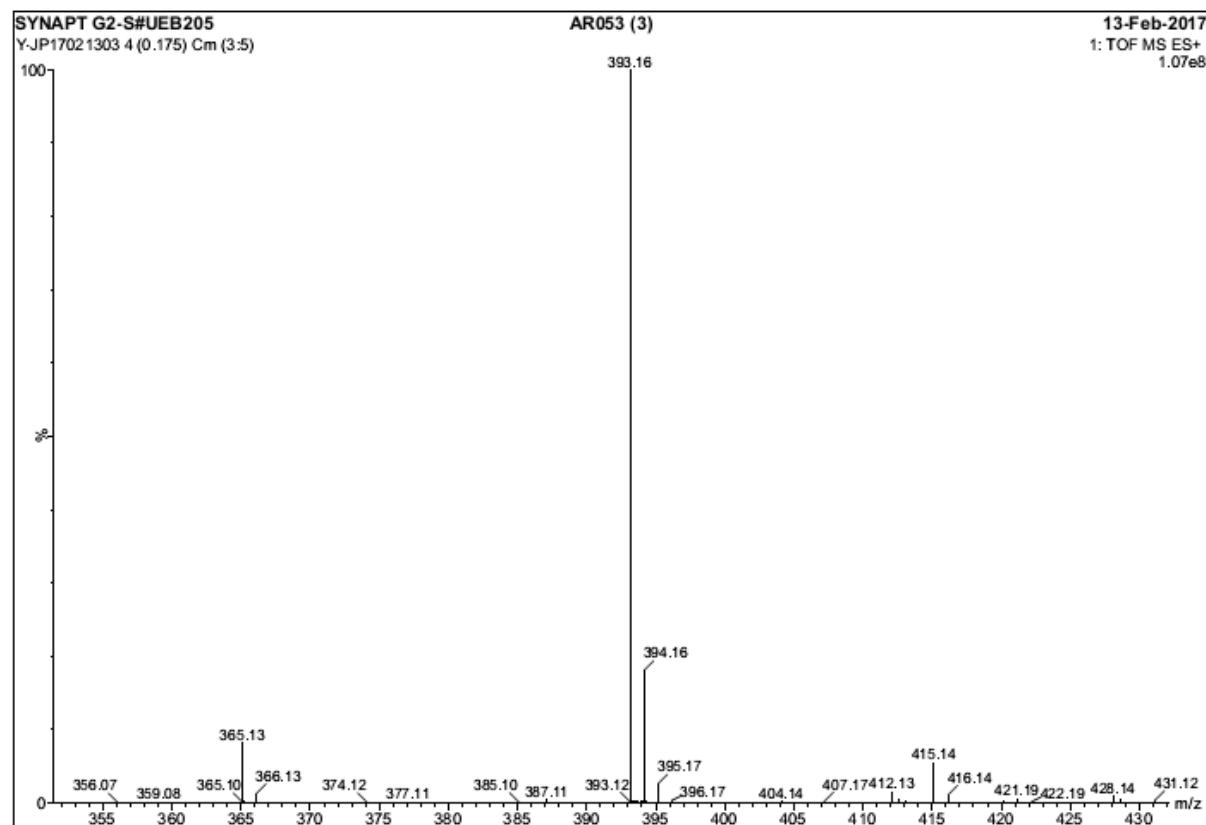
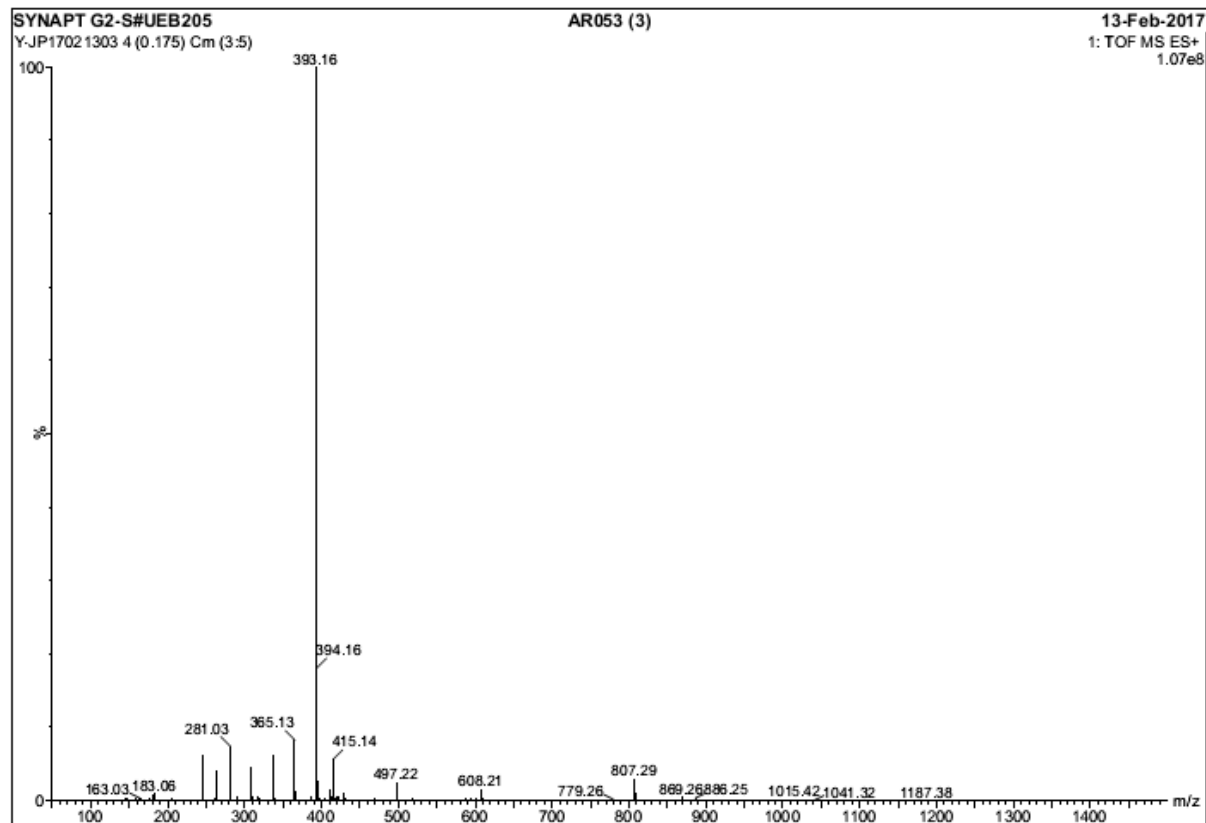


AR053 - 3



AR053 - 3





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

794 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 2-2

SYNAPT G2-S#UEB205

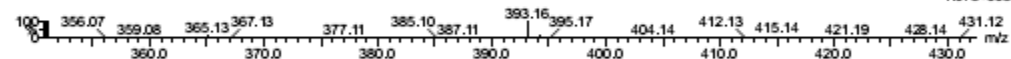
YJPI7021303 4 (0.175) Cm (2d5)

AR053 (3)

13-Feb-2017

1: TOF MS ES+

1.07e+008



Minimum:

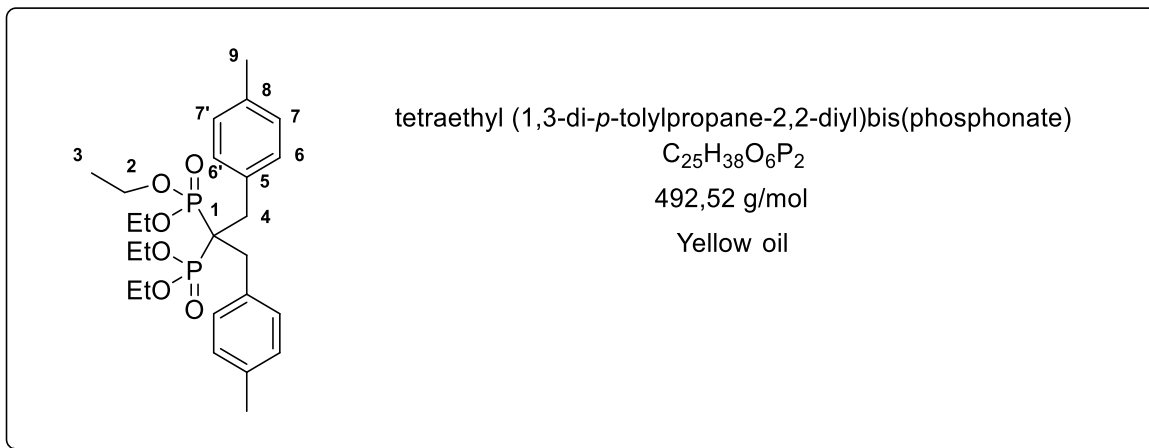
Maximum:

10.0 3.0

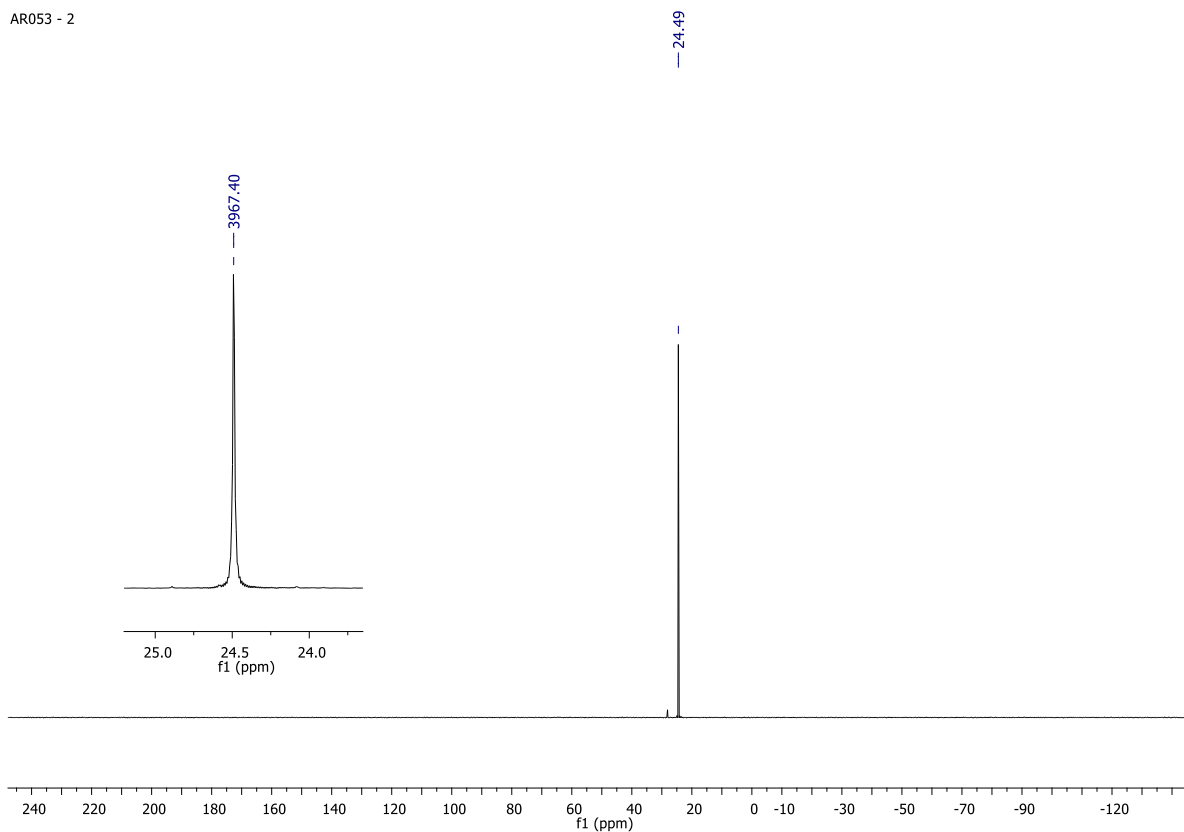
-1.5

50.0

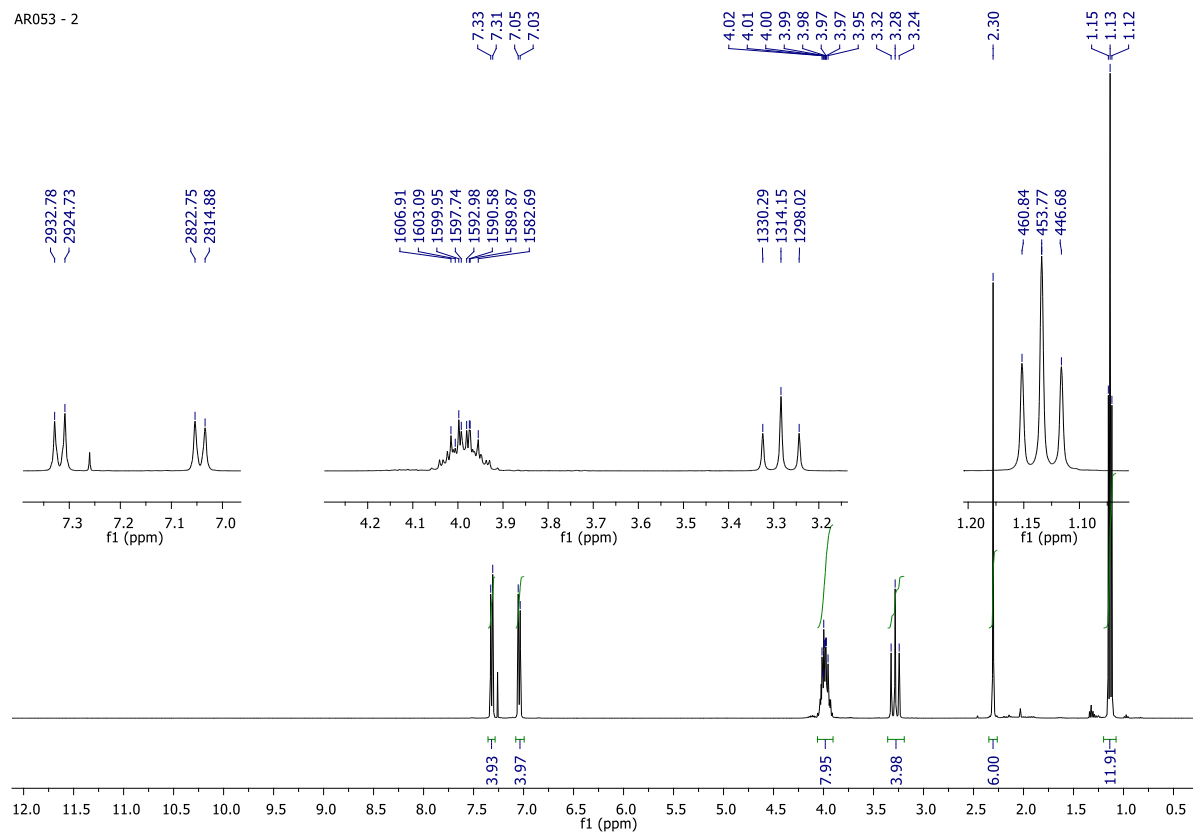
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
393.1598	393.1596	0.2	0.5	3.5	2237.7	0.002	99.83	C17 H31 O6 P2
	393.1609	-1.1	-2.8	8.5	2244.1	6.393	0.17	C18 H27 N4 O2 P2

S10: ^{31}P , ^1H and ^{13}C NMR spectra and mass analysis of tetraethyl (1,3-di-*p*-tolylpropane-2,2-diyl)bis(phosphonate) (3e)

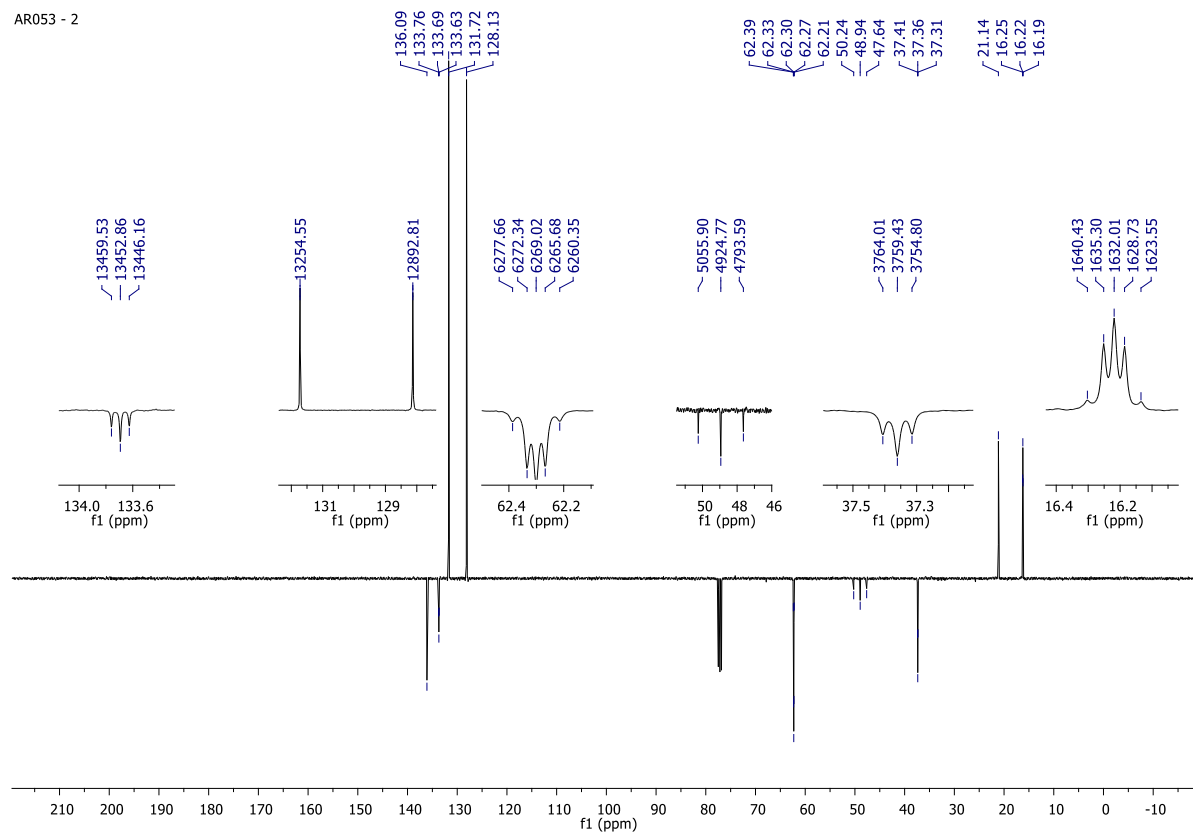
AR053 - 2

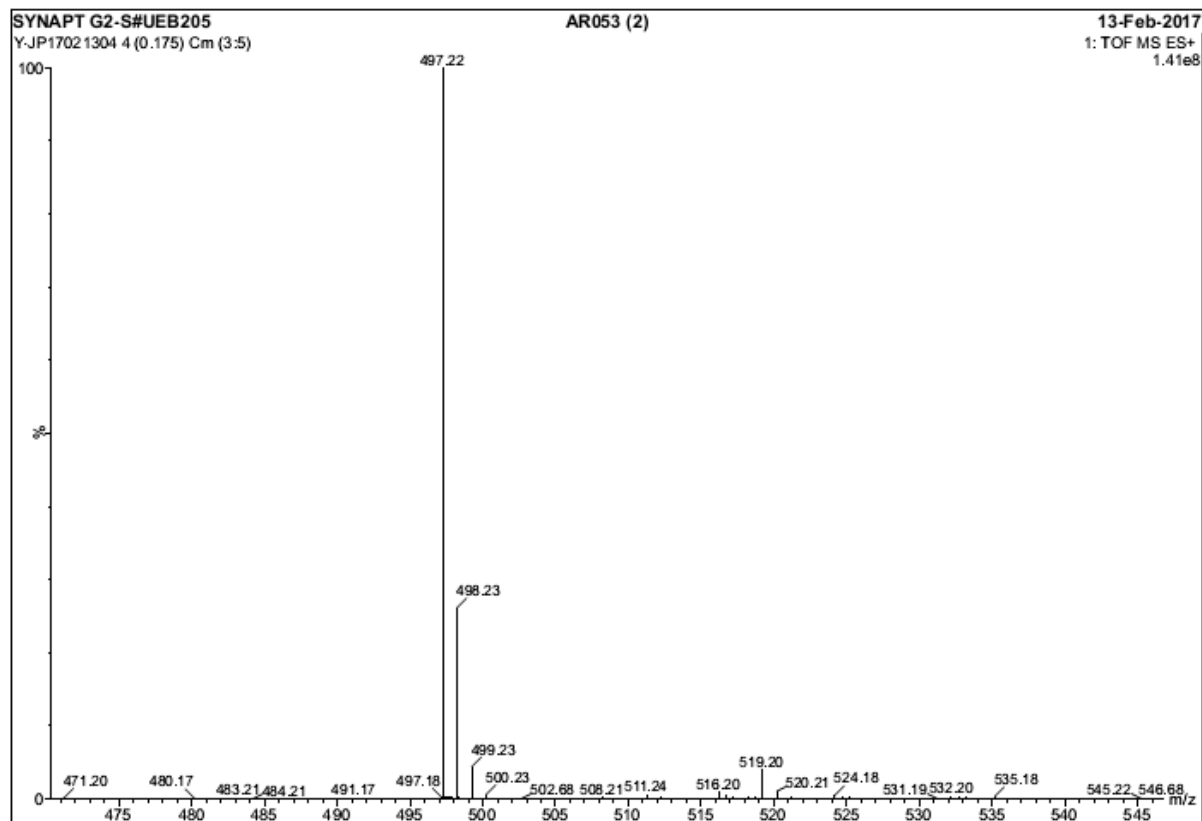
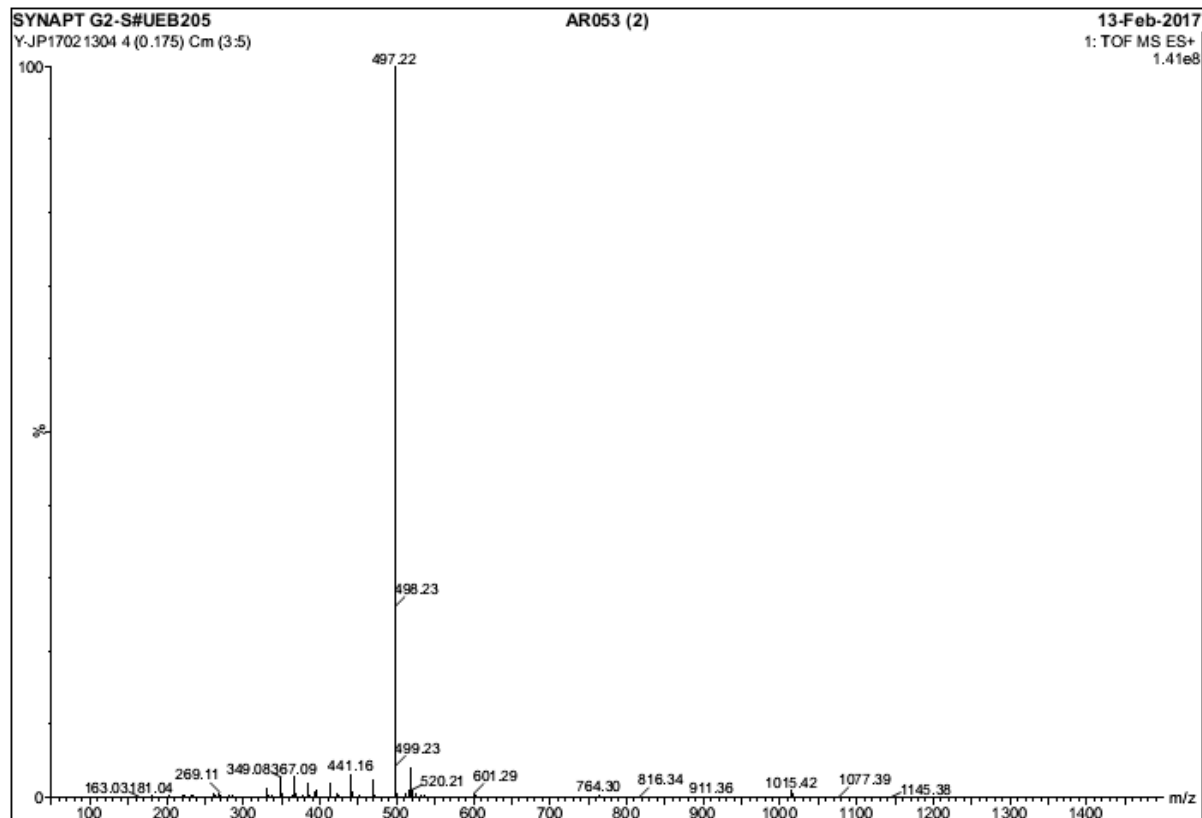


AR053 - 2



AR053 - 2





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 PPM / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1410 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 2-2

SYNAPT G2-S#UEB205

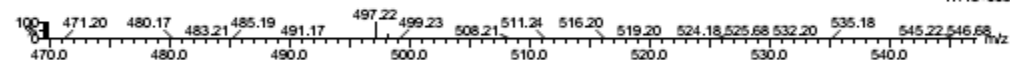
Y-JP17021304.4 (0.175) Cm (3x5)

AR053 (2)

13-Feb-2017

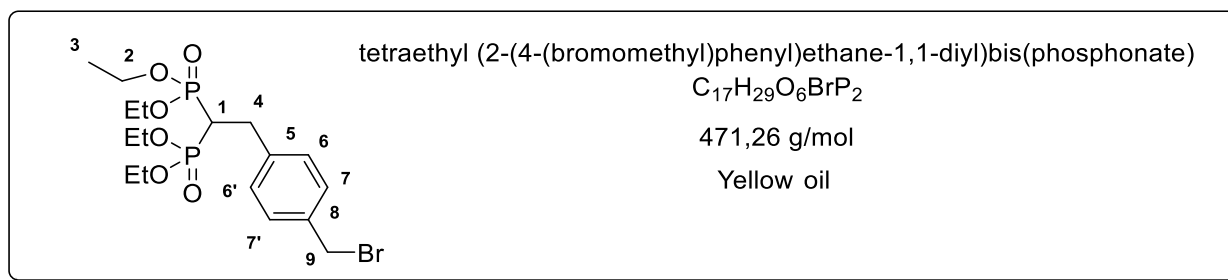
1: TOF MS ES+

1.41e+008

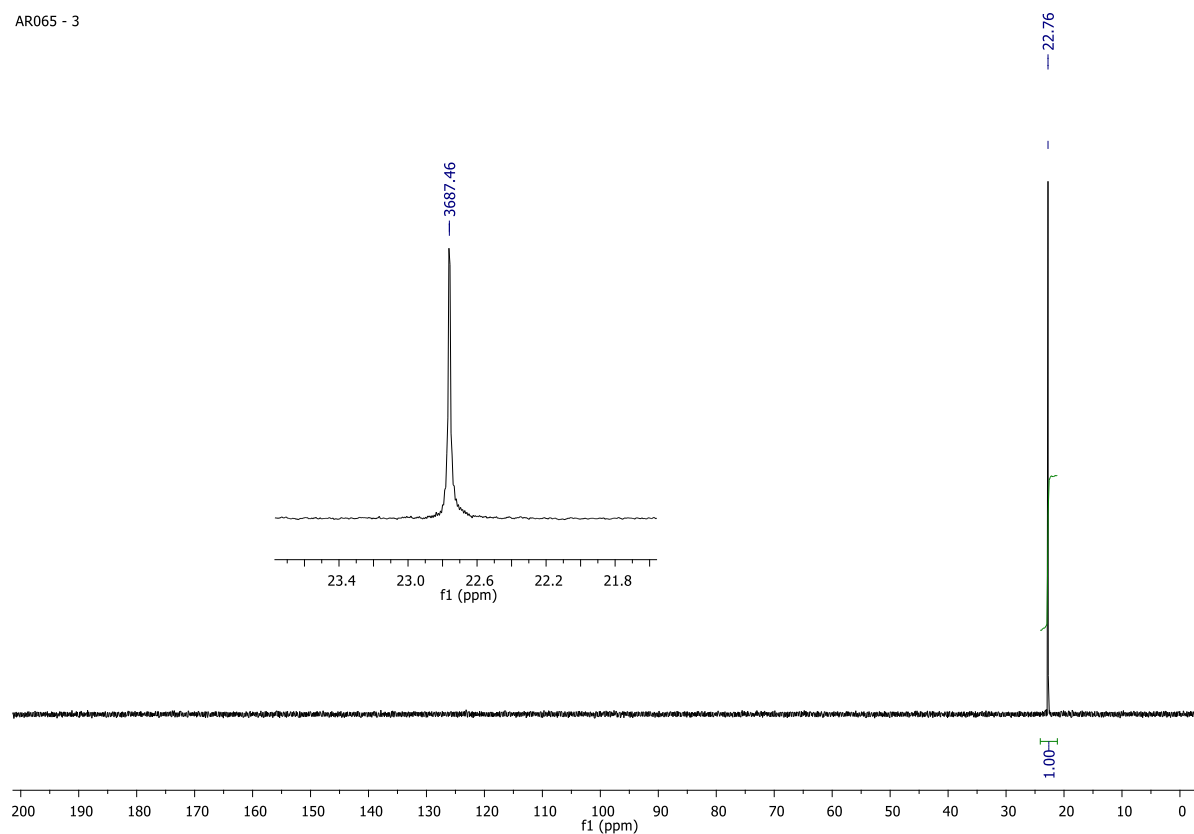


Minimum: -1.5
Maximum: 10.0 1.0 50.0

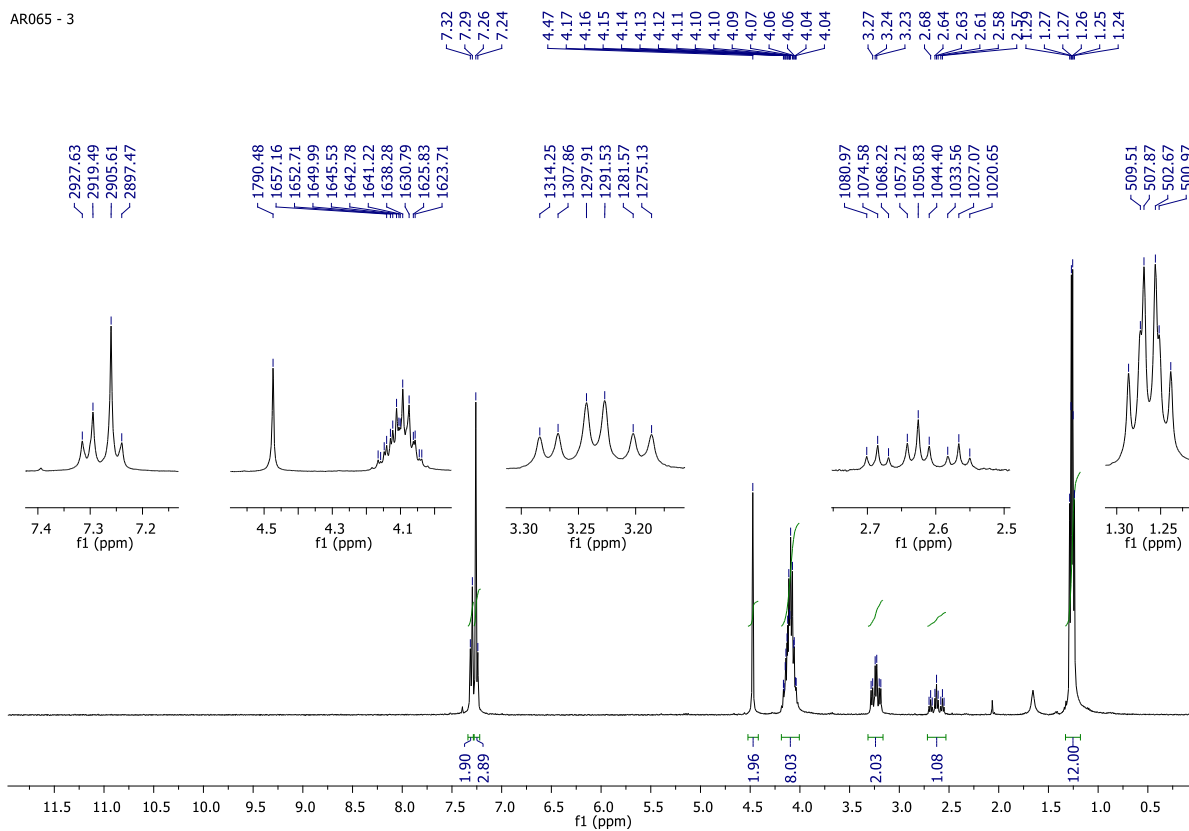
Mass	Calc. Mass	mDa	PM	DBE	i-FIT	Norm	Conf (%)	Formula
497.2223	497.2222	0.1	0.2	7.5	1901.1	0.000	100.00	C25 H39 O6 P2
	497.2227	-0.4	-0.8	0.5	1912.2	11.094	0.00	C10 H35 N12 O7 P2

S11: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl (1,3-bis(4-(bromomethyl)phenyl)propane-2,2-diyl)bis(phosphonate) (2f)

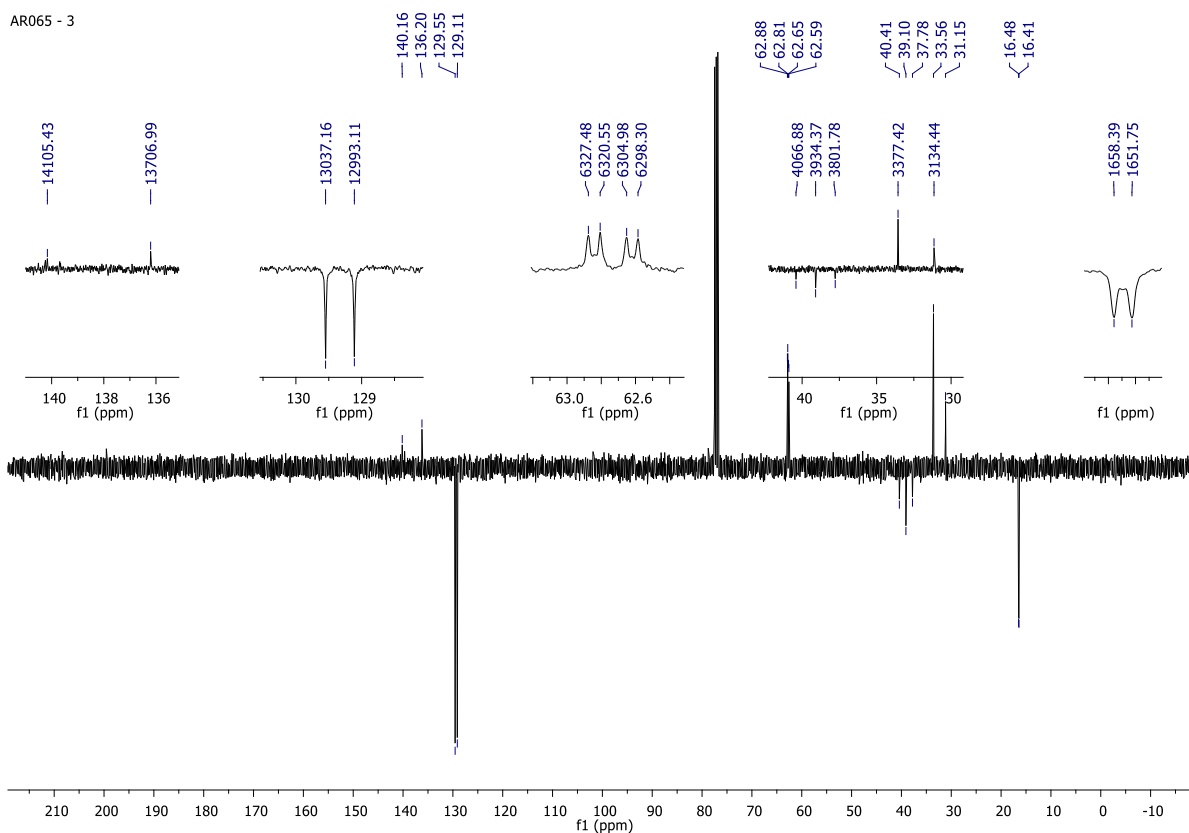
AR065 - 3

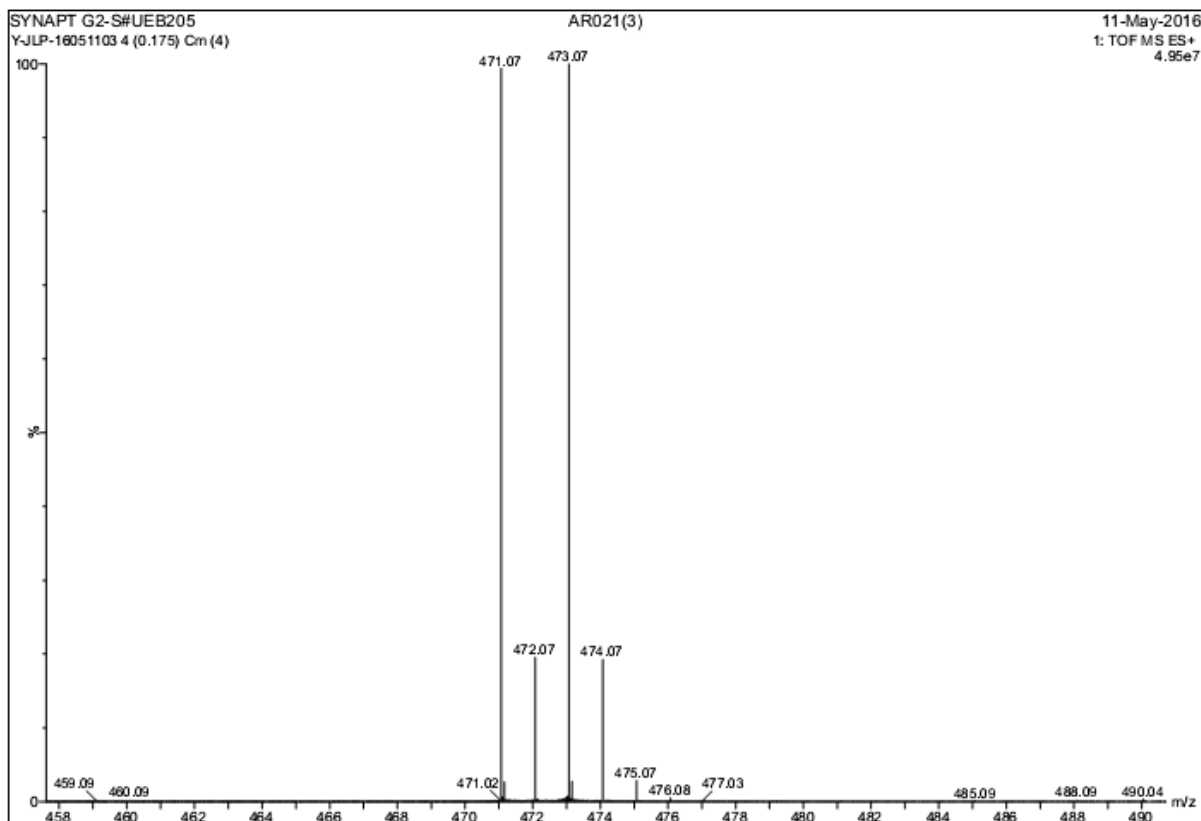
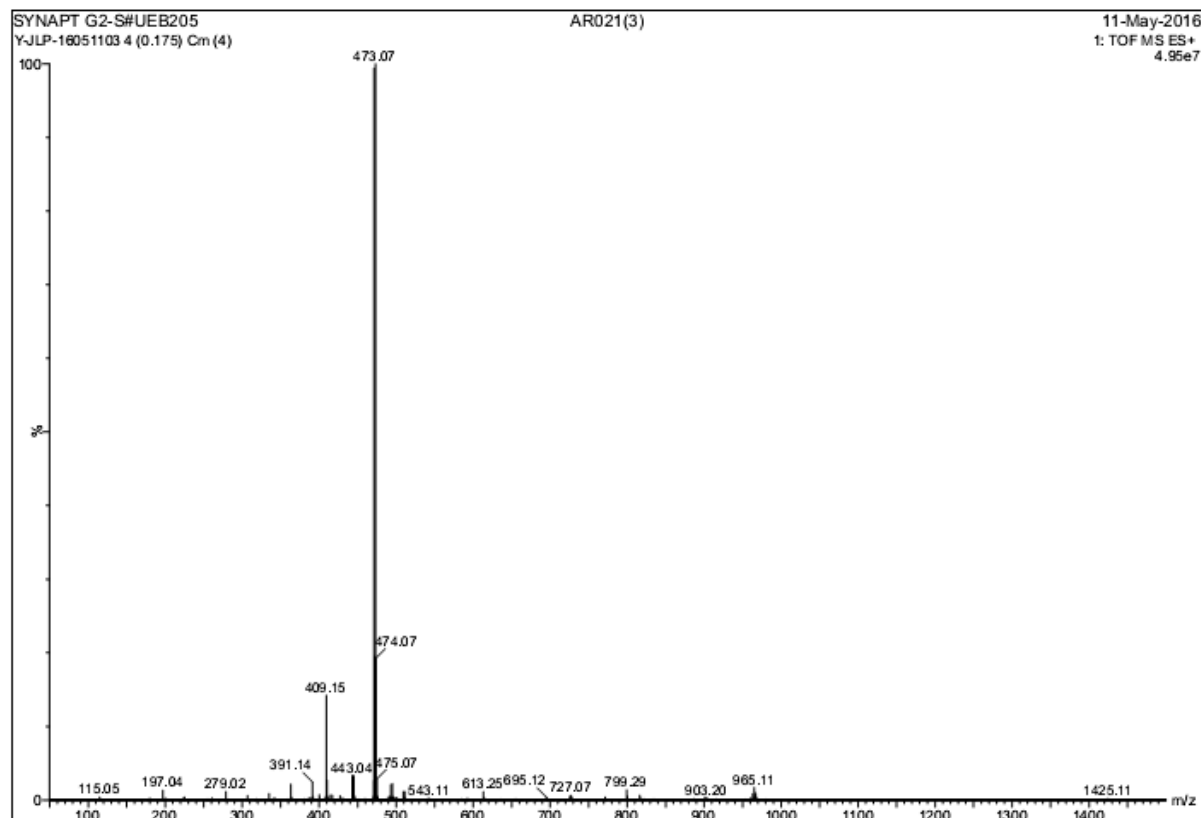


AR065 - 3



AR065 - 3





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 2.0 PPM / DBE: min = -50.0, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

4436 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-100 H: 0-150 N: 0-10 O: 0-10 P: 1-2 Br: 1-2

SYNAPT G2-S#UEB205

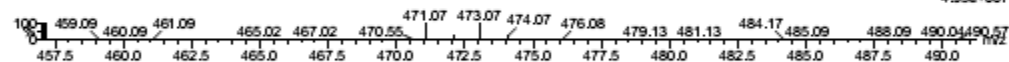
AR021(3)

YJLP-16051103 4 (0.175) Cm (4)

11-May-2016

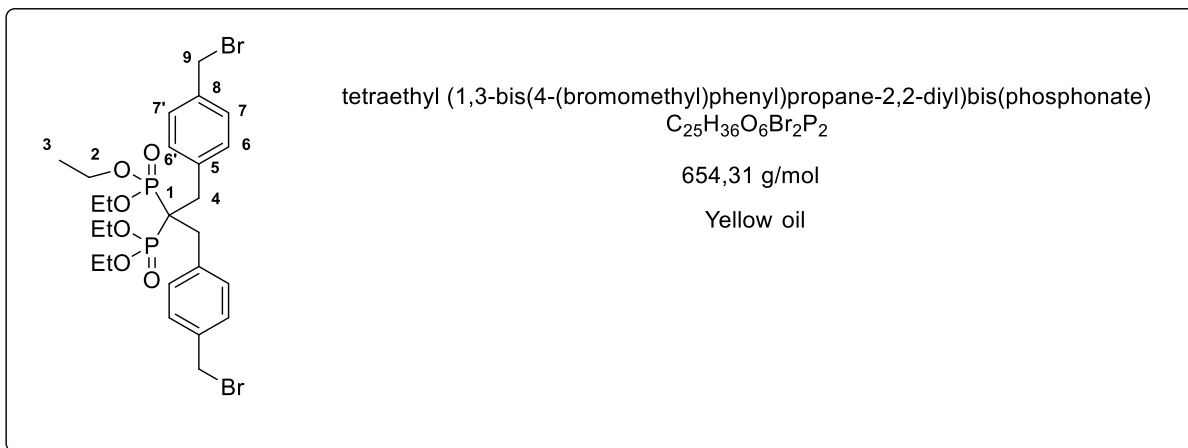
1: TOF MS ES+

4.95e+007

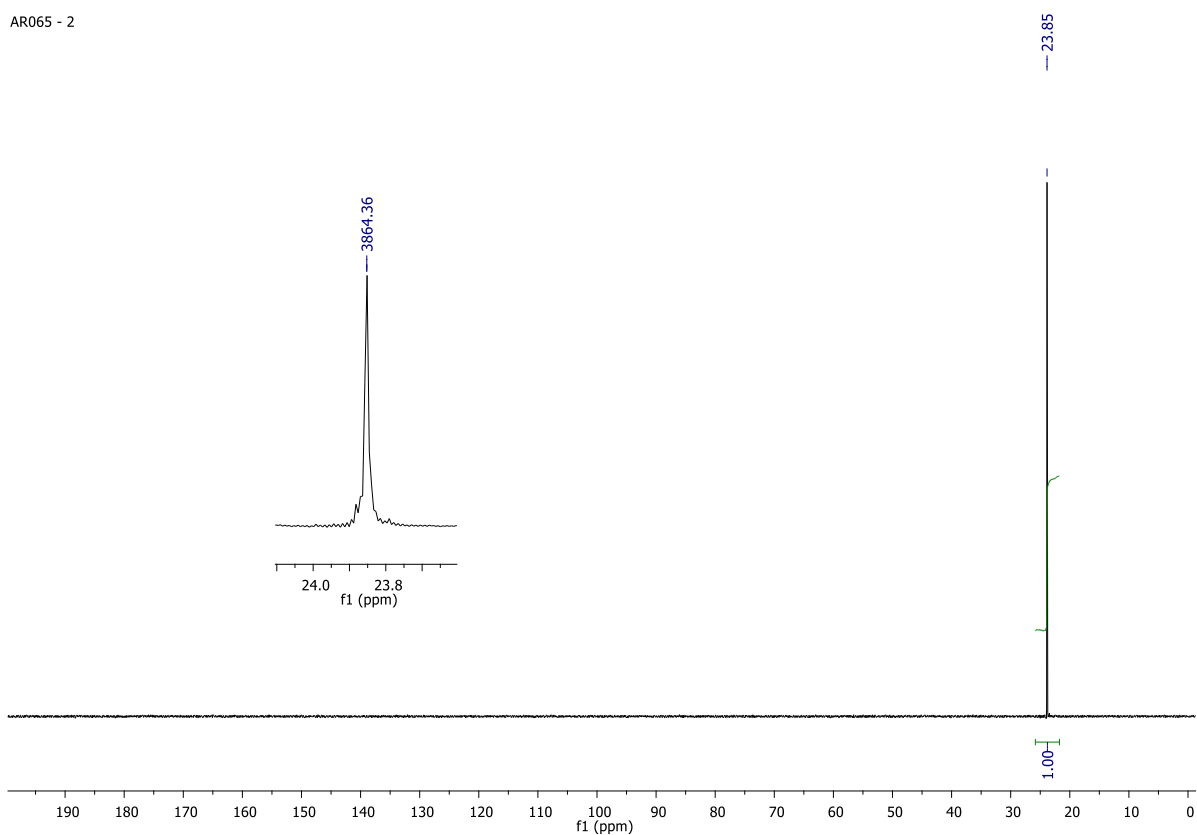


Minimum: -50.0
Maximum: 100.0

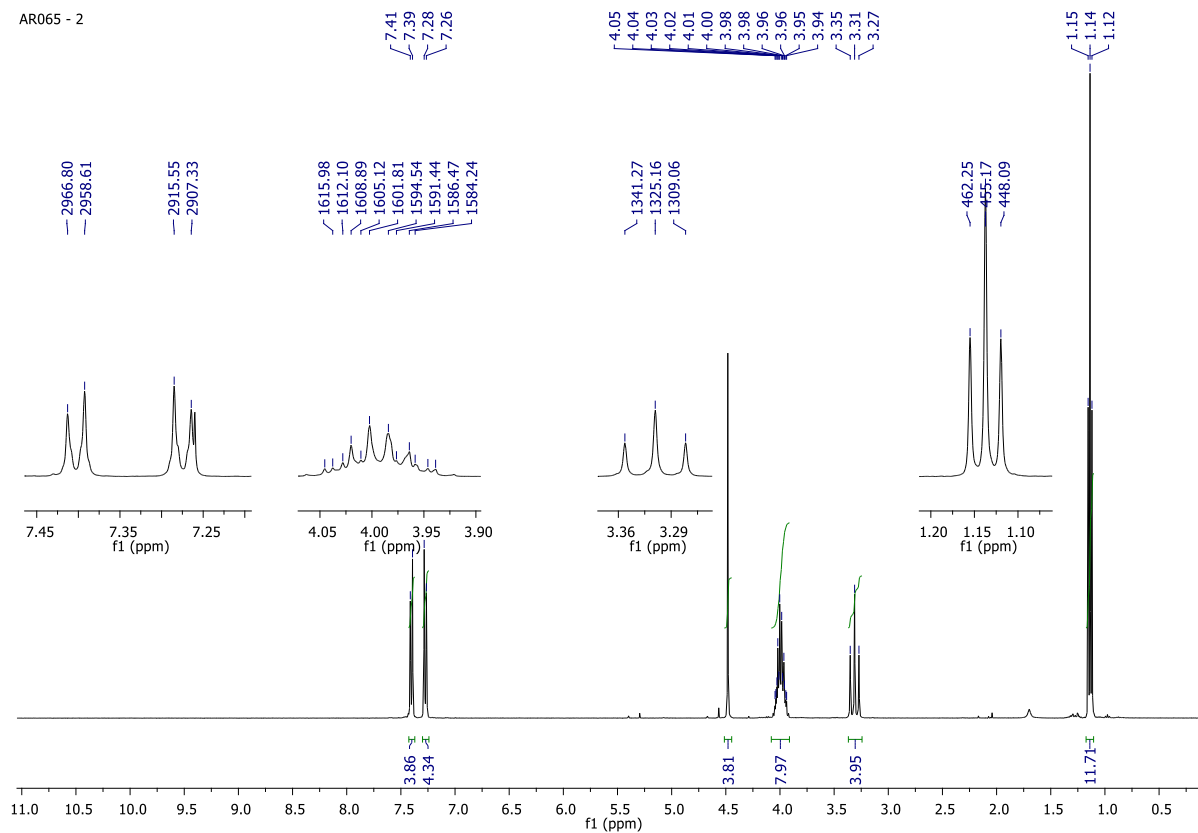
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
471.0704	471.0701	0.3	0.6	3.5	1608.0	0.000	99.99	C17 H30 O6 P2 Br
	471.0698	0.6	1.3	13.5	1617.1	9.086	0.01	C20 H21 N6 O P Br
	471.0698	0.6	1.3	-16.5	1623.5	15.463	0.00	C4 H43 O10 P2 Br2
	471.0712	-0.8	-1.7	-11.5	1624.8	16.714	0.00	C5 H39 N4 O6 P2 Br2
	471.0708	-0.4	-0.8	-1.5	1626.0	17.976	0.00	C8 H30 N10 O P Br2
	471.0695	0.9	1.9	-6.5	1626.3	18.290	0.00	C7 H34 N6 O5 P Br2

S12: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl (1,3-bis(4-(bromomethyl)phenyl)propane-2,2-diyl)bis(phosphonate) (3f)

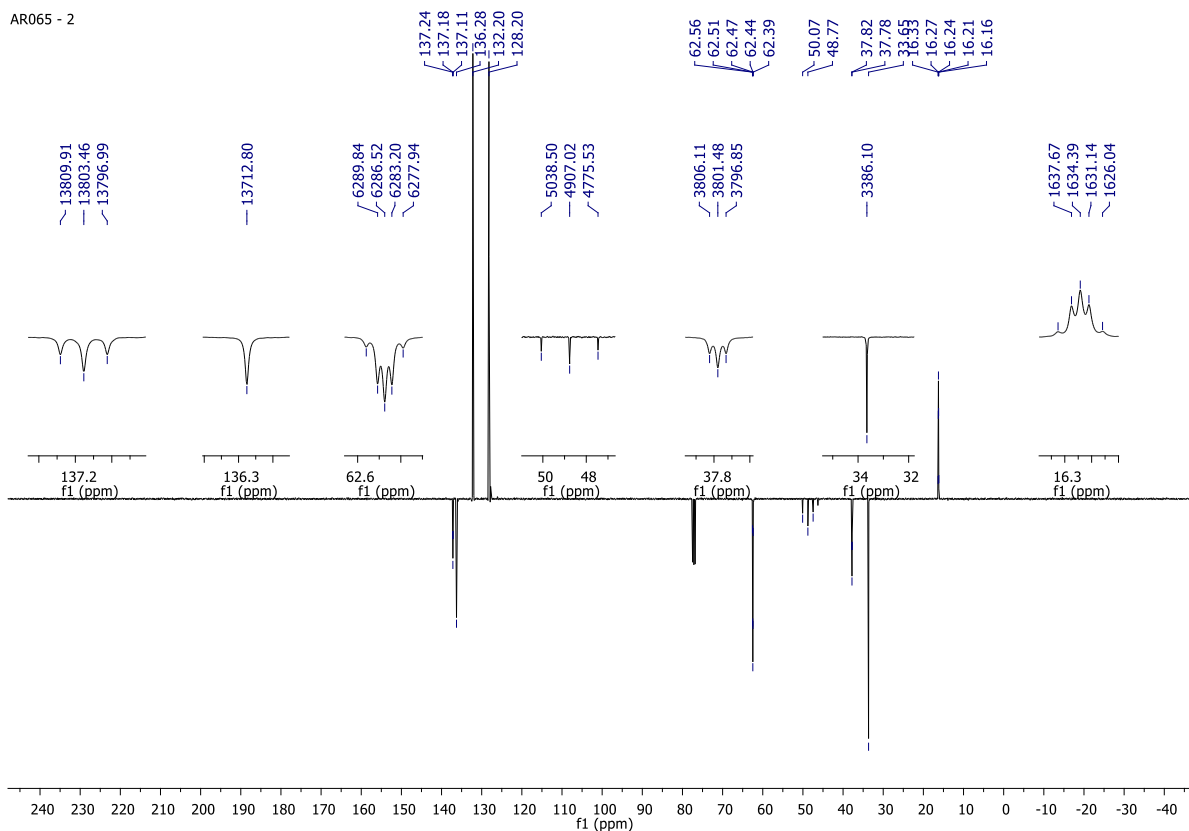
AR065 - 2

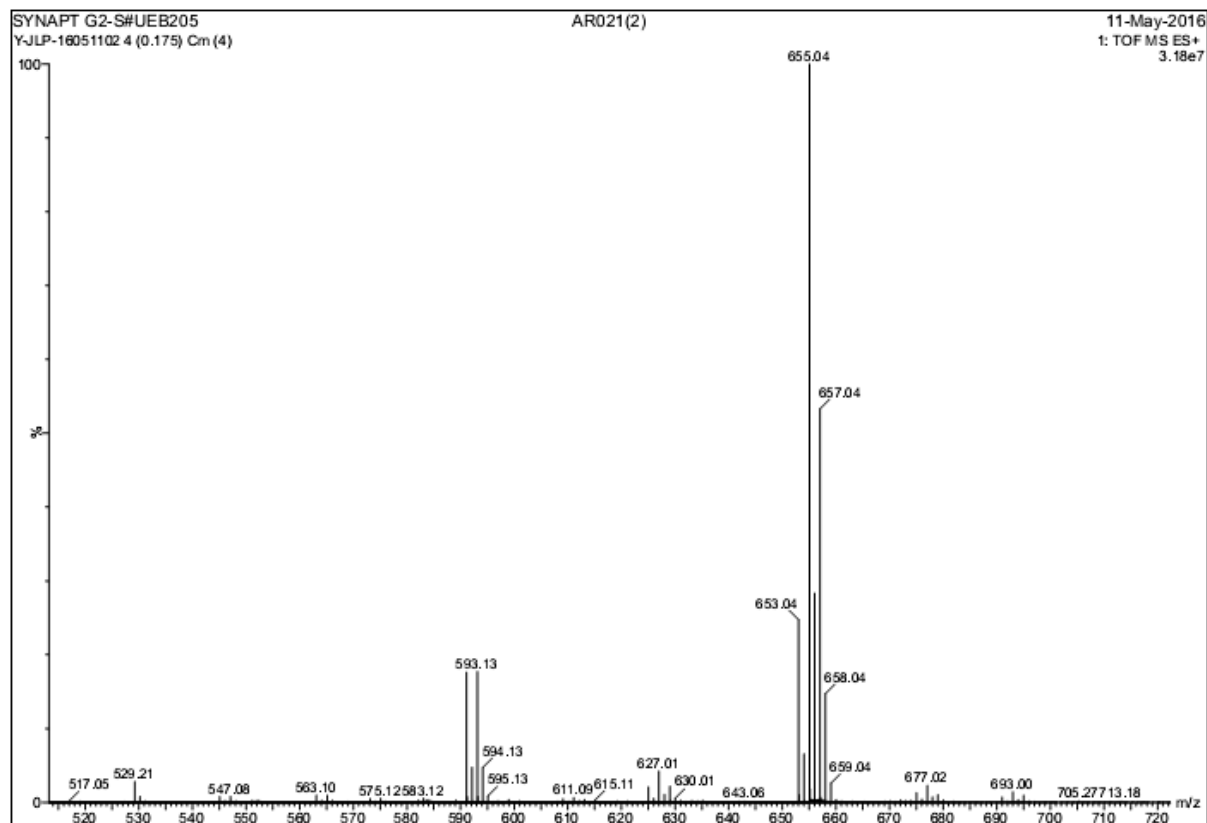
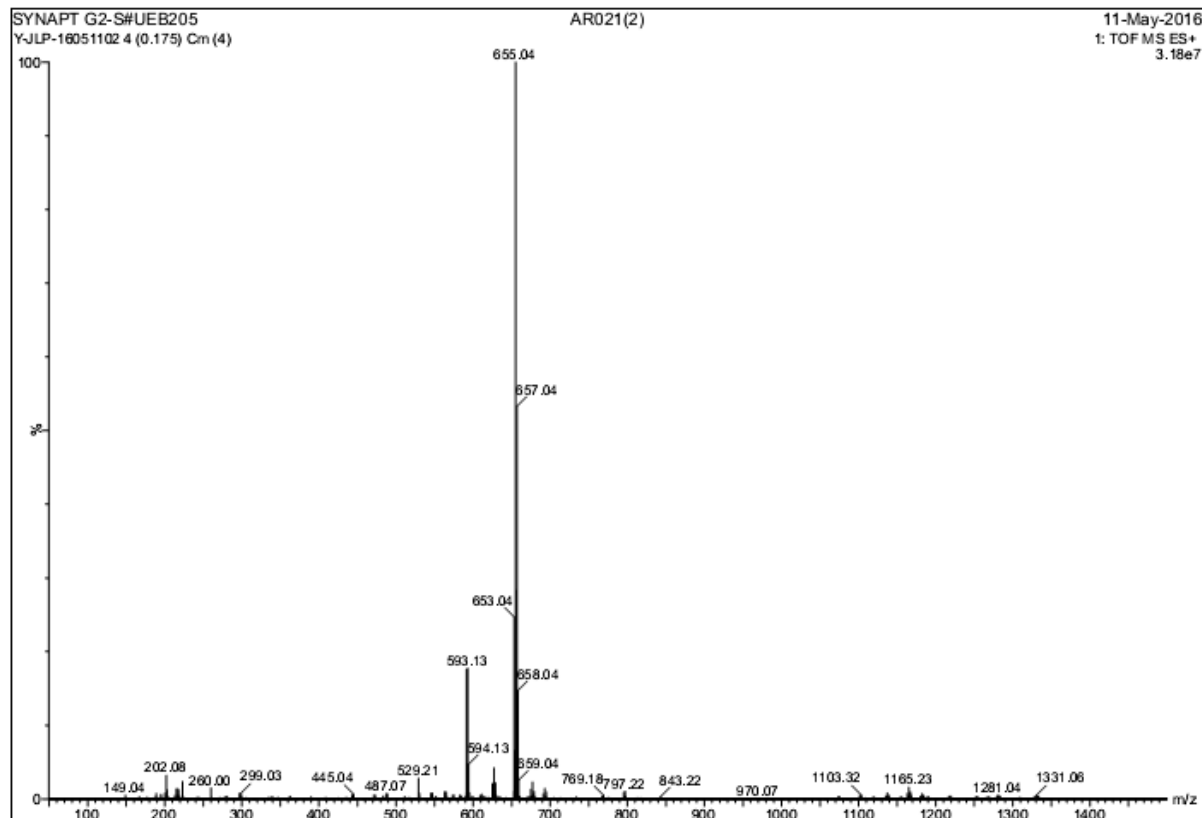


AR065 - 2



AR065 - 2





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 2.0 PPM / DBE: min = -50.0, max = 100.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

2857 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)

Elements Used:

C: 0-100 H: 0-150 N: 0-10 O: 0-10 P: 1-2 Br: 2-2

SYNAPT G2-S#UEB205

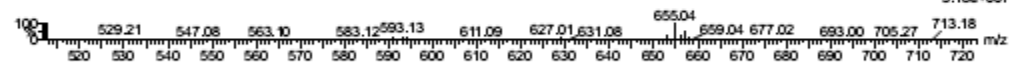
AR021(2)

11-May-2016

YJLP-16051102.4 (0.175) Cm (4)

1: TOF MS ES+

3.18e+007



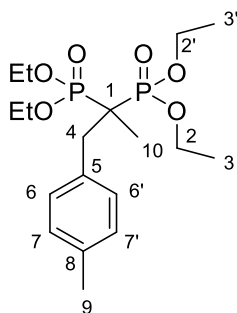
Minimum:

Maximum:

1.1 2.0 -50.0

100.0

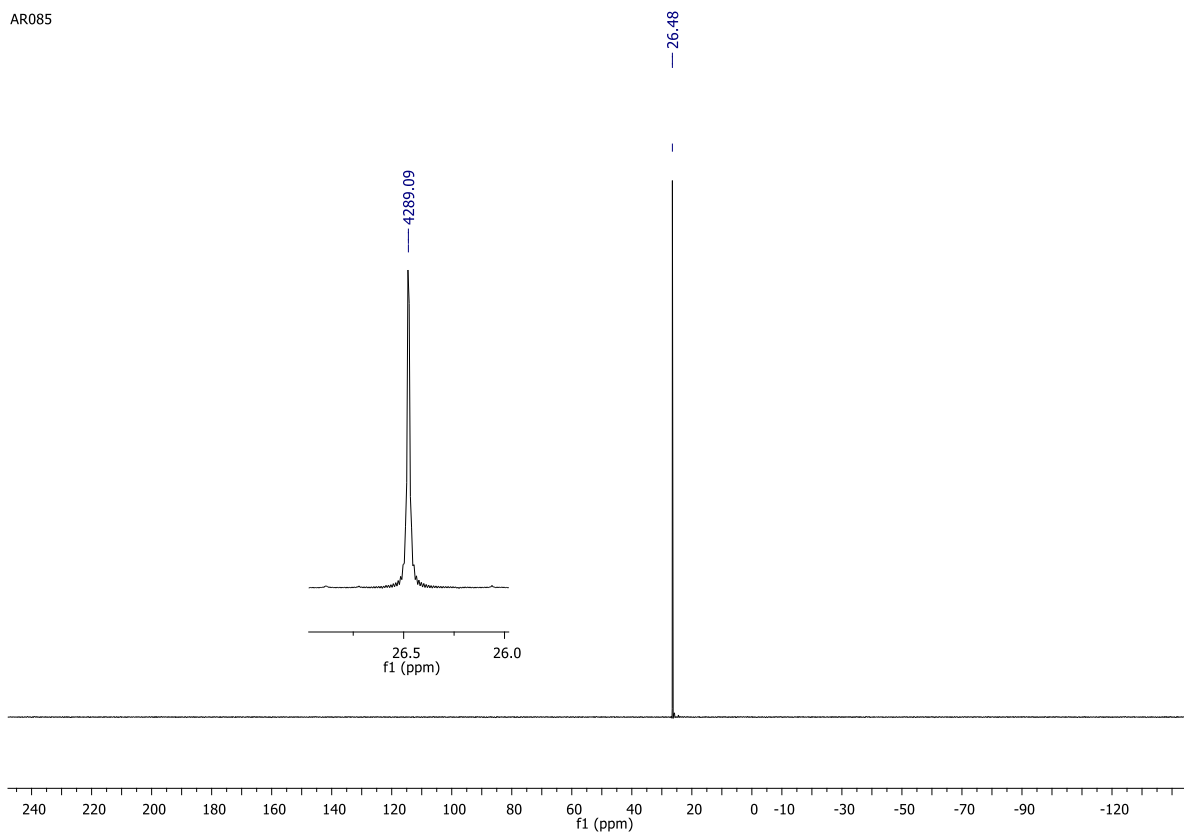
Mass	Calc. Mass	mDa	PM	DBE	i-FIT	Norm	Conf (%)	Formula
653.0433	653.0432	0.1	0.2	7.5	1666.3	0.432	64.91	C25 H37 O6 P2 Br2
	653.0429	0.4	0.6	17.5	1667.5	1.563	20.96	C28 H28 N6 O P Br2
	653.0446	-1.3	-2.0	12.5	1667.9	1.957	14.13	C26 H33 N4 O2 P2 Br2

S13: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl (1-(*p*-tolyl)propane-2,2-diyl)bis(phosphonate) (6)tetraethyl (1-(*p*-tolyl)propane-2,2-diyl)bis(phosphonate) $\text{C}_{18}\text{H}_{32}\text{O}_6\text{P}_2$

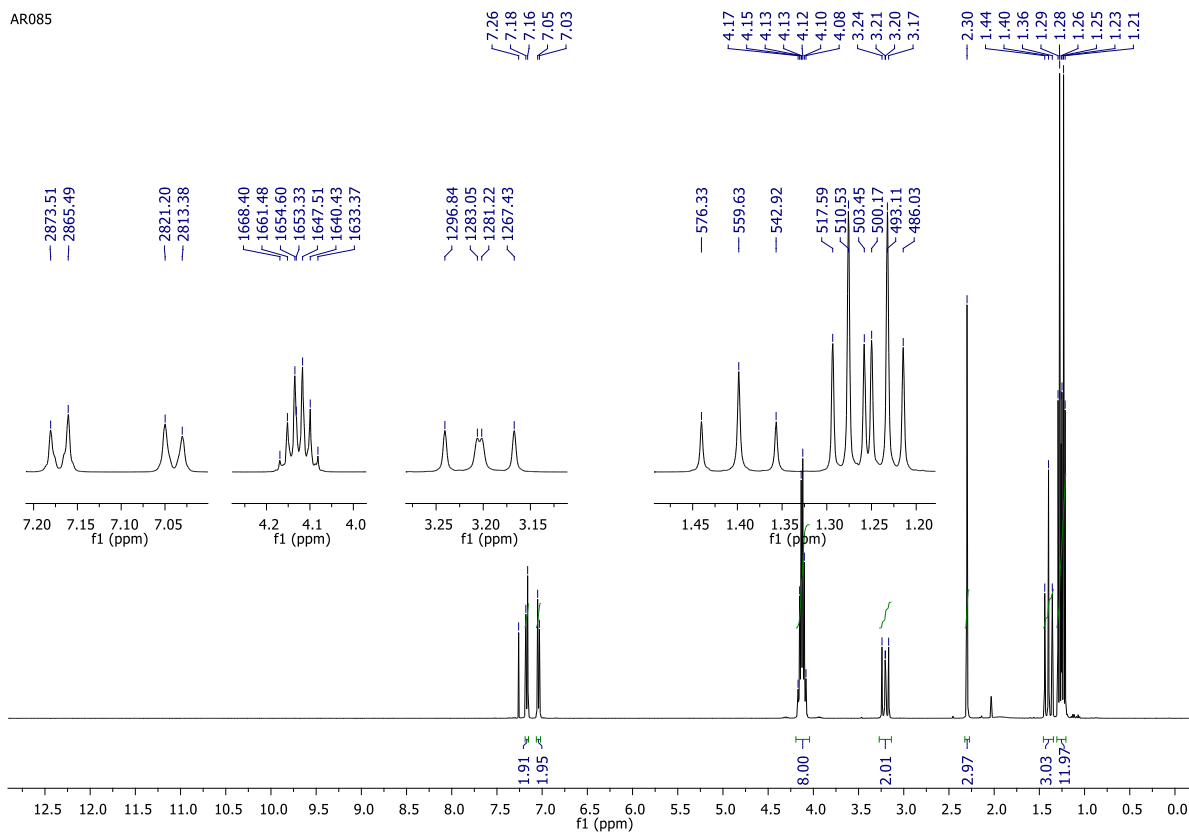
406,40 g/mol

Yellow oil

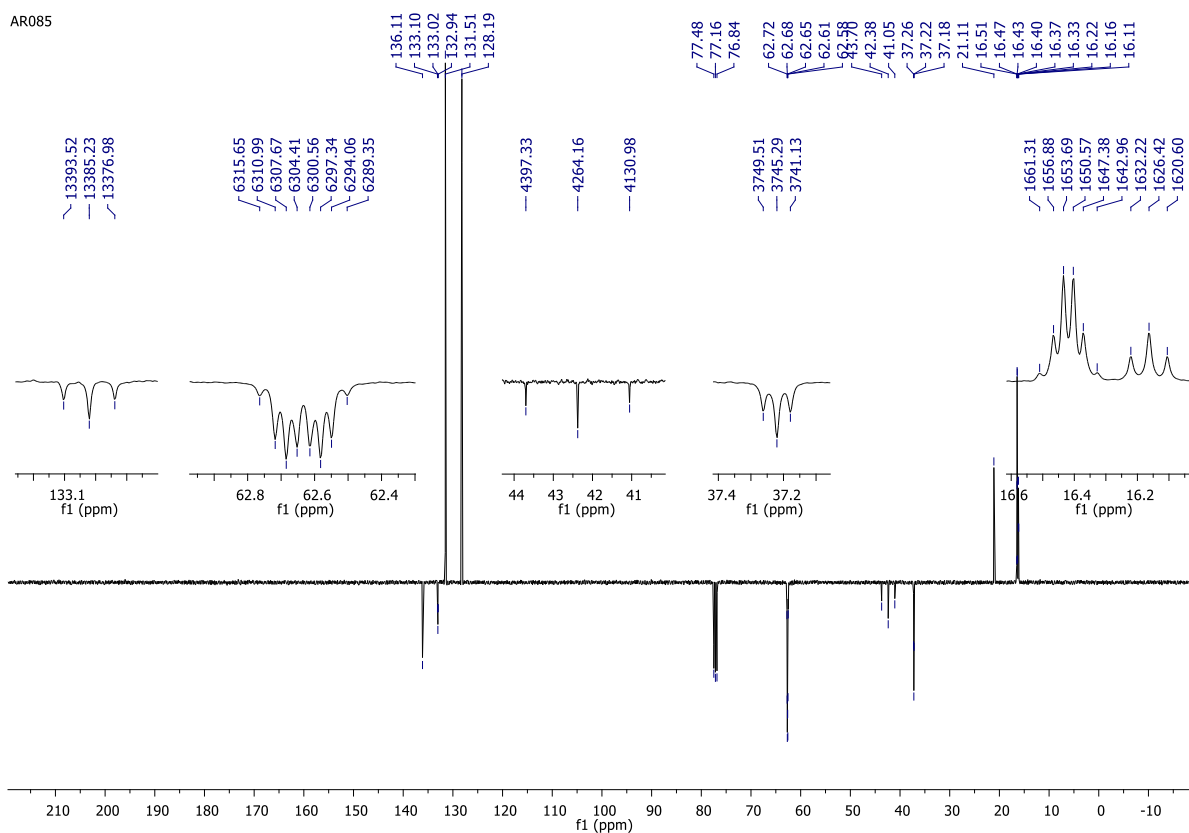
AR085

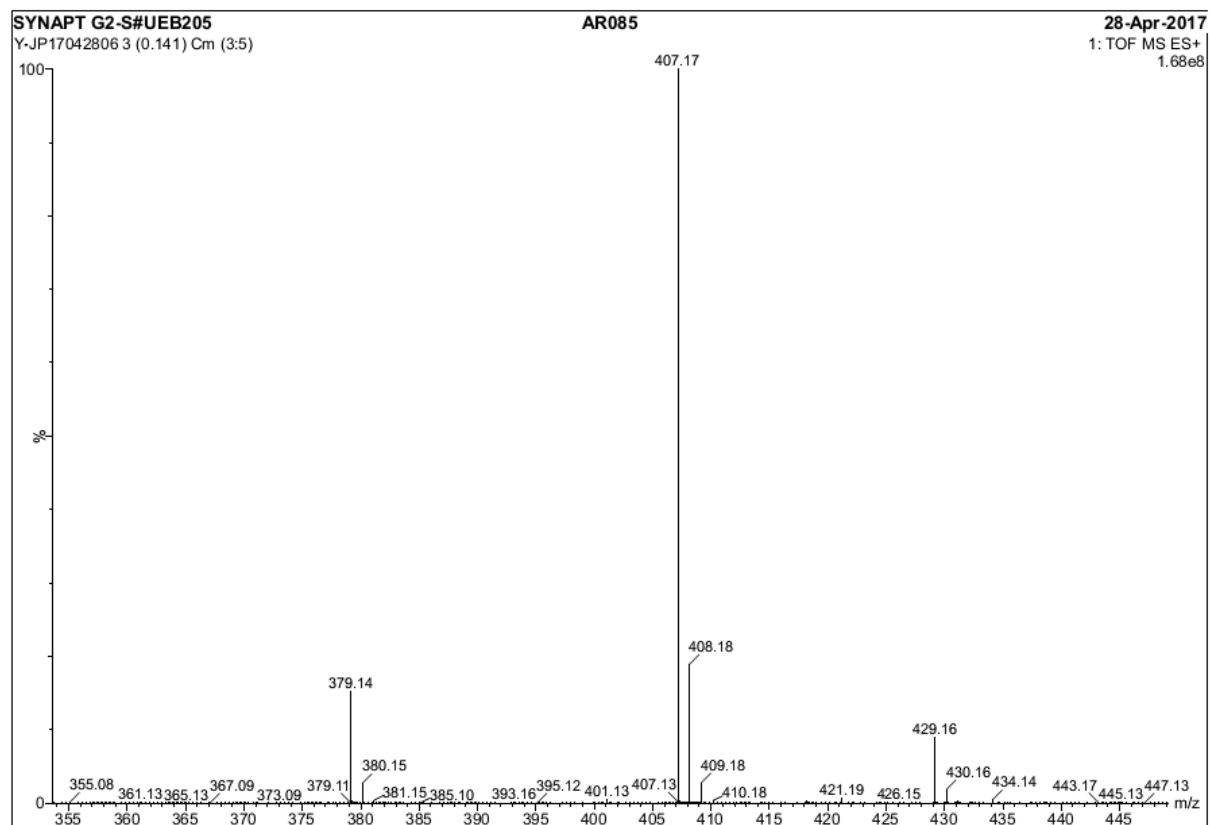
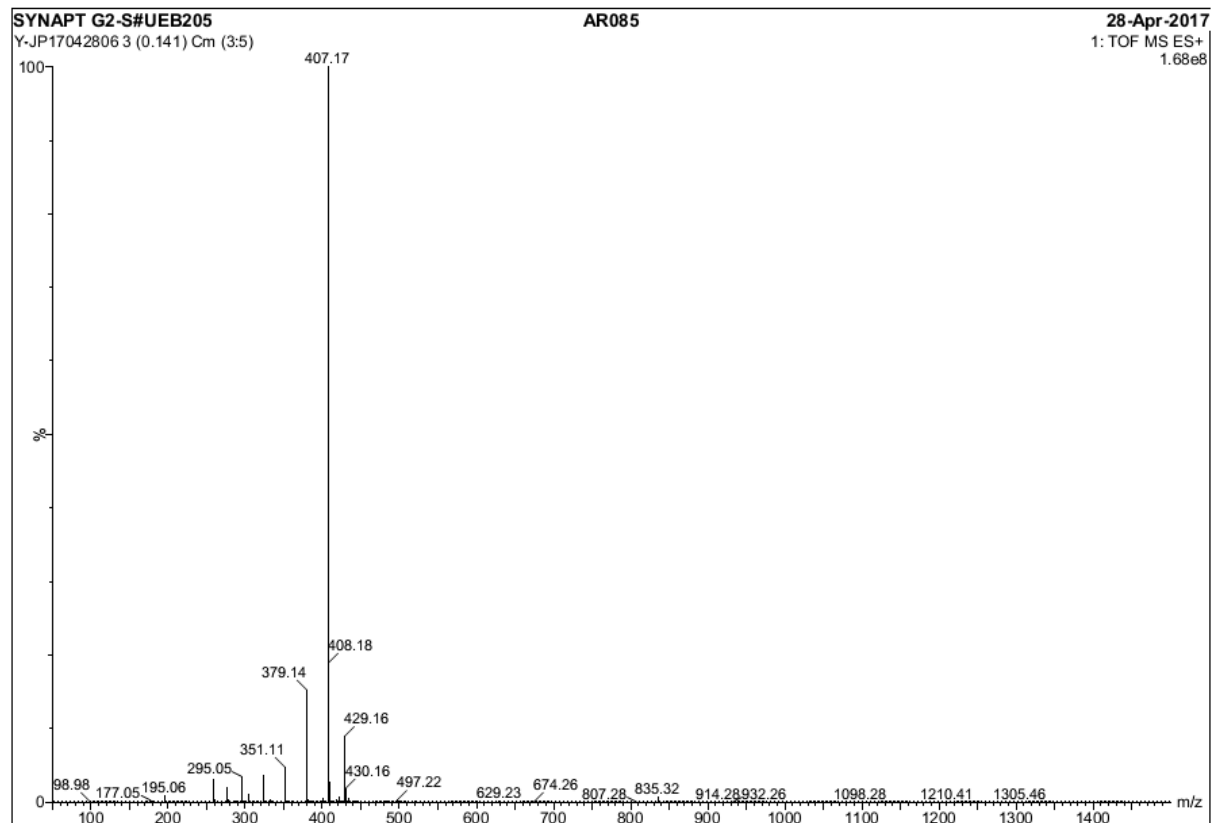


AR085



AR085





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

3015 formula(e) evaluated with 8 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 1-100 H: 0-100 N: 0-20 O: 0-20 P: 0-2

SYNAPT G2-S#UEB205

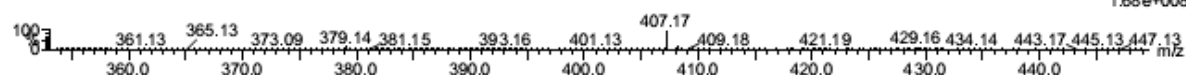
Y-JP17042806 3 (0.141) Cm (3:5)

AR085

28-Apr-2017

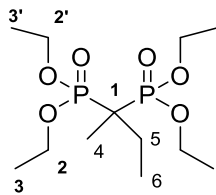
1: TOF MS ES+

1.68e+008



Minimum: -1.5
Maximum: 1.0 3.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
407.1747	407.1752	-0.5	-1.2	3.5	2314.7	0.005	99.46	C18 H33 O6 P2
	407.1749	-0.2	-0.5	13.5	2320.3	5.606	0.37	C21 H24 N6 O P
	407.1751	-0.4	-1.0	5.5	2321.5	6.836	0.11	C11 H23 N10 O7
	407.1739	0.8	2.0	9.5	2322.1	7.427	0.06	C15 H25 N10 P2
	407.1738	0.9	2.2	0.5	2325.0	10.306	0.00	C10 H27 N6 O11
	407.1738	0.9	2.2	11.5	2327.6	12.962	0.00	C8 H15 N20 O
	407.1754	-0.7	-1.7	6.5	2328.4	13.738	0.00	C6 H20 N18 O2 P
	407.1741	0.6	1.5	1.5	2328.9	14.207	0.00	C5 H24 N14 O6 P

S14: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl butane-2,2-diylbis(phosphonate) (7)

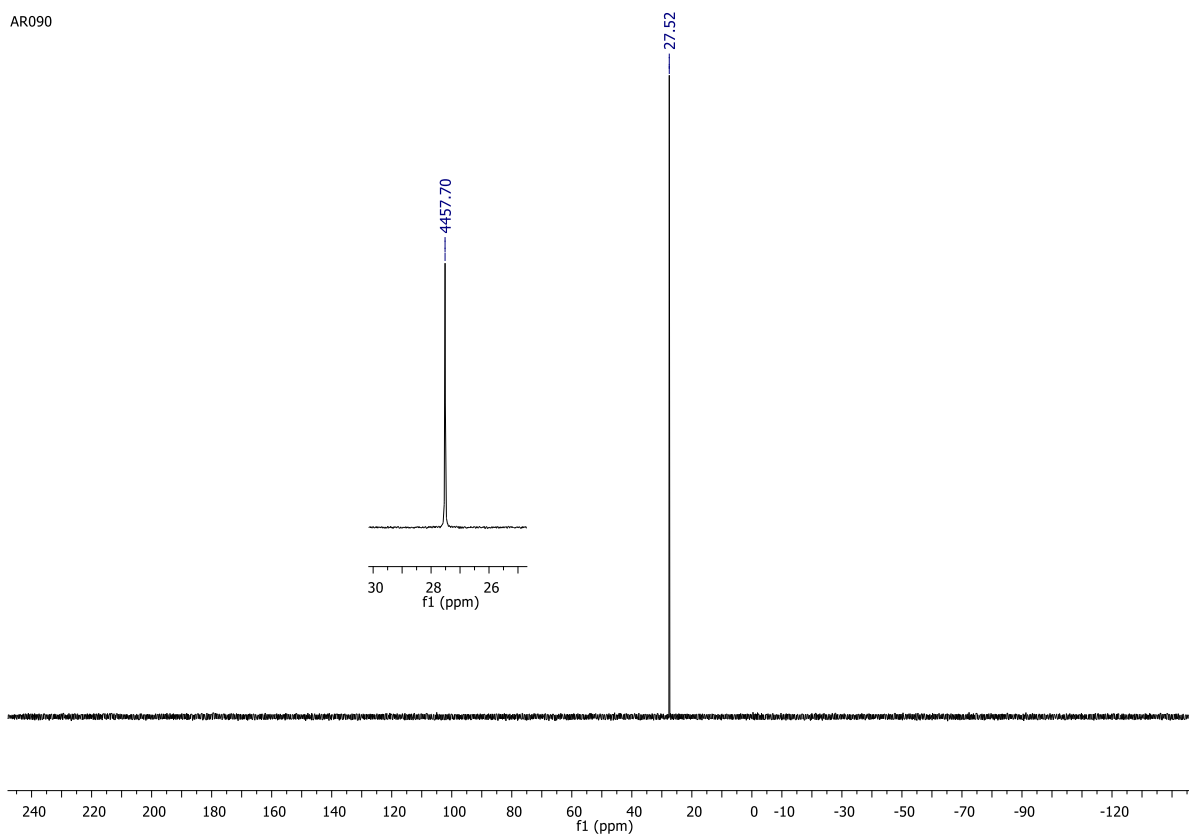
tetraethyl butane-2,2-diylbis(phosphonate)

 $\text{C}_{12}\text{H}_{28}\text{O}_6\text{P}_2$

330,30 g/mol

Colorless oil

AR090

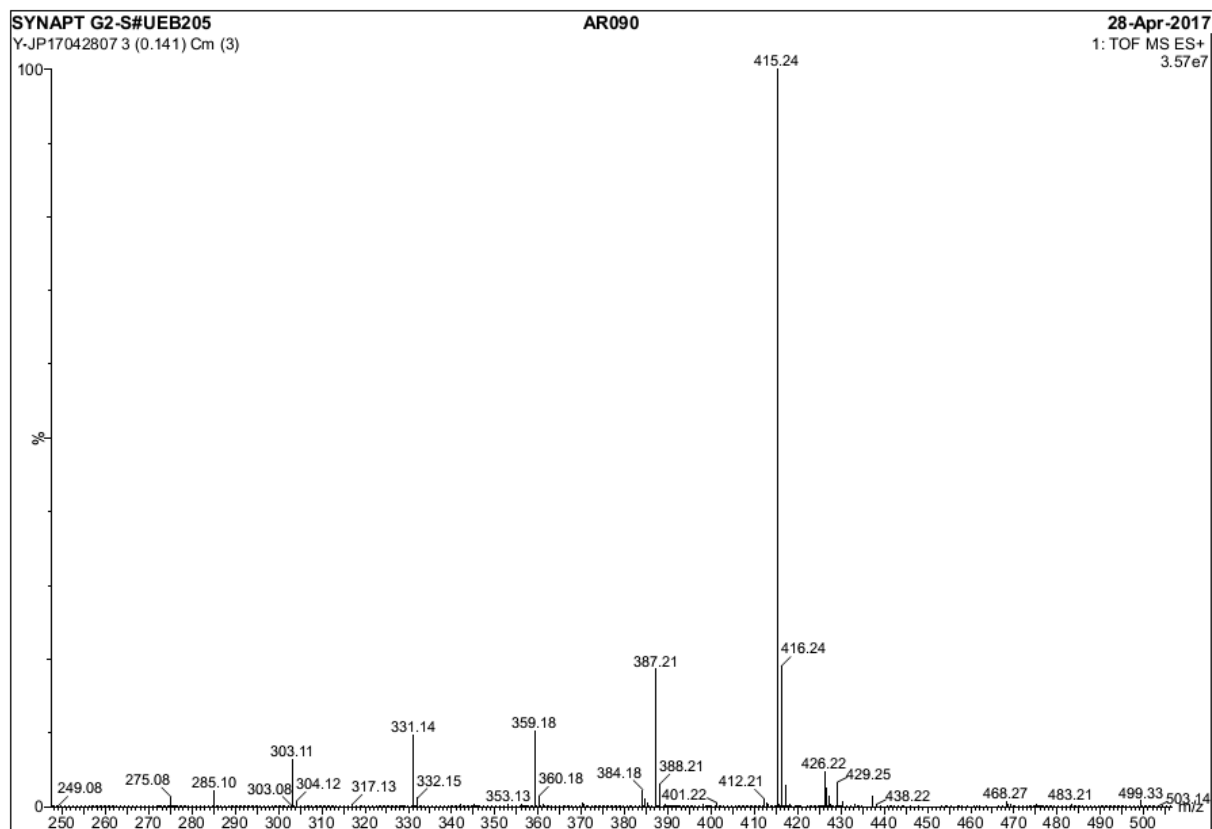
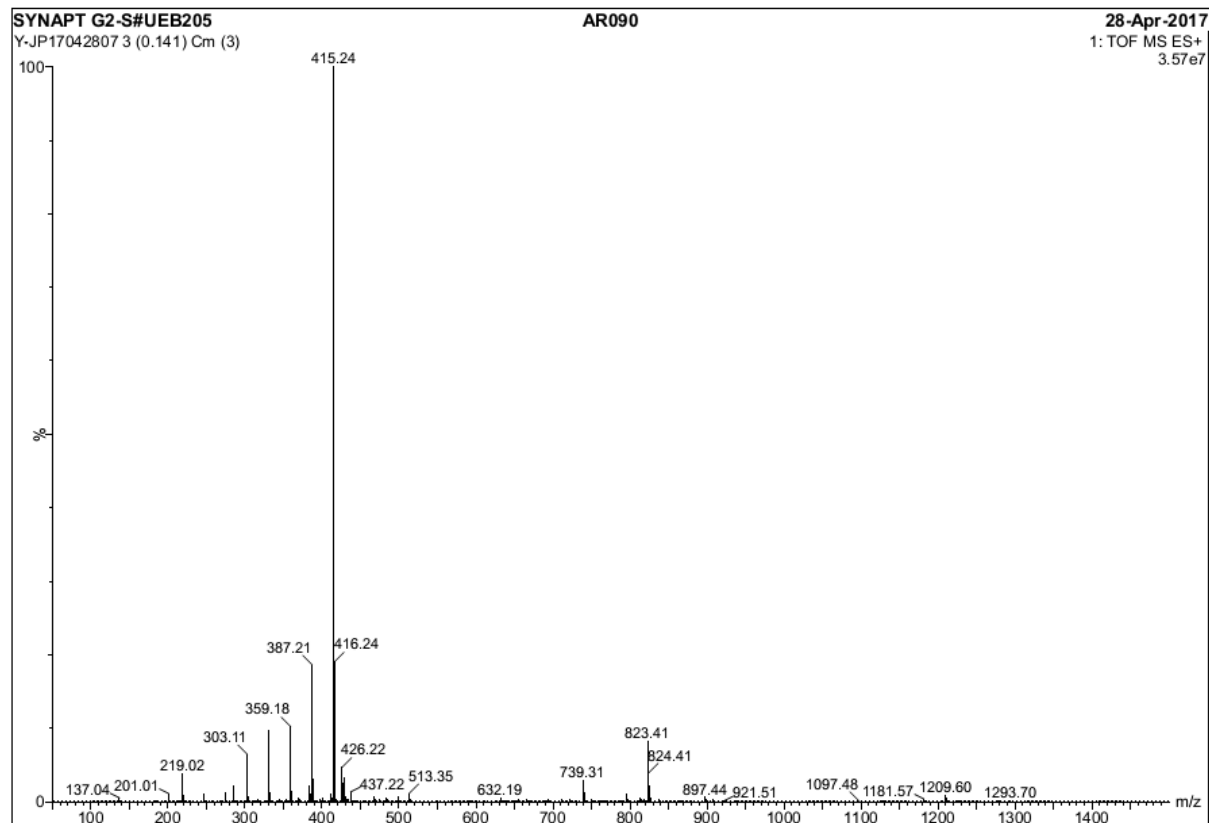


¹H NMR (400 MHz, CDCl₃)

Chemical Shift (ppm)	Integration
4.21, 4.19, 4.18, 4.16, 4.14	7.97
1.97, 1.95, 1.93, 1.91, 1.89, 1.87, 1.45, 1.41, 1.37, 1.34, 1.32, 1.30, 1.10, 1.08, 1.06	2.23, 3.14, 12.00, 3.01

¹³C NMR (100 MHz, CDCl₃)

Chemical Shift (ppm)
1685.70, 1678.59, 1670.82, 1663.64, 1656.54
796.35, 788.75, 781.13, 772.51, 764.91, 756.25, 748.65, 741.16
580.15, 563.52, 546.89, 536.24, 529.19, 522.10
440.71, 433.19, 425.63



Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

1781 formula(e) evaluated with 4 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 1-100 H: 0-100 N: 0-20 O: 0-20 P: 0-2

SYNAPT G2-S#UEB205

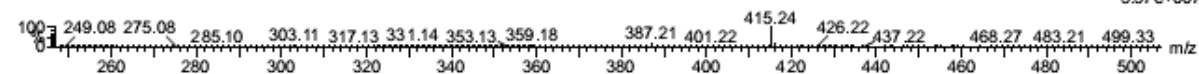
Y-JP17042807 3 (0.141) Cm (3)

AR090

28-Apr-2017

1: TOF MS ES+

3.57e+007



Minimum:

Maximum:

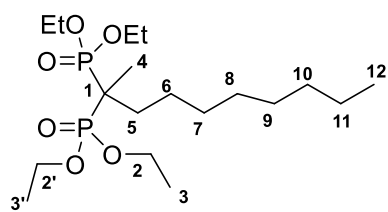
1.0

3.0

-1.5

50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
331.1441	331.1439	0.2	0.6	-0.5	1968.1	0.001	99.86	C12 H29 O6 P2
	331.1436	0.5	1.5	9.5	1975.3	7.164	0.08	C15 H20 N6 O P
	331.1438	0.3	0.9	1.5	1975.6	7.472	0.06	C5 H19 N10 O7
	331.1447	-0.6	-1.8	13.5	1979.6	11.423	0.00	C21 H19 N2 O2

S15: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of tetraethyl decane-2,2-diylbis(phosphonate) (8)

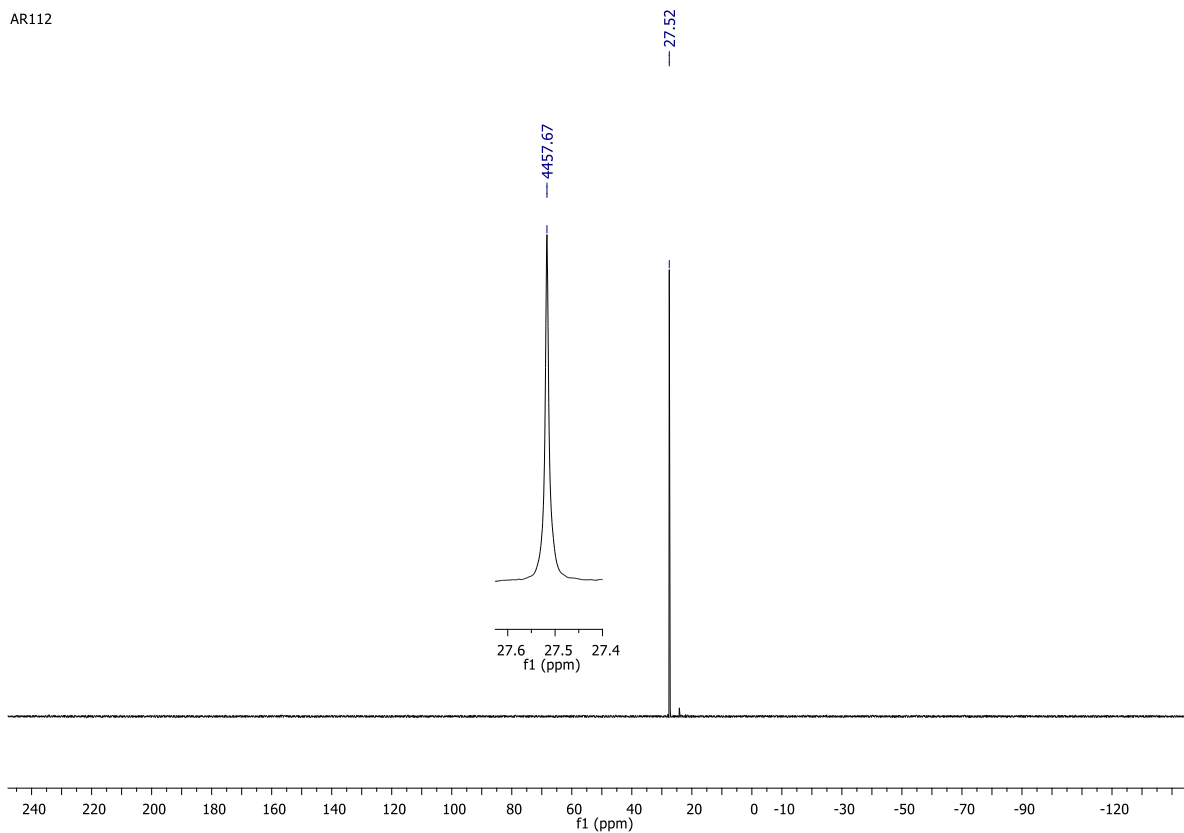
tetraethyl decane-2,2-diylbis(phosphonate)

 $\text{C}_{18}\text{H}_{40}\text{O}_6\text{P}_2$

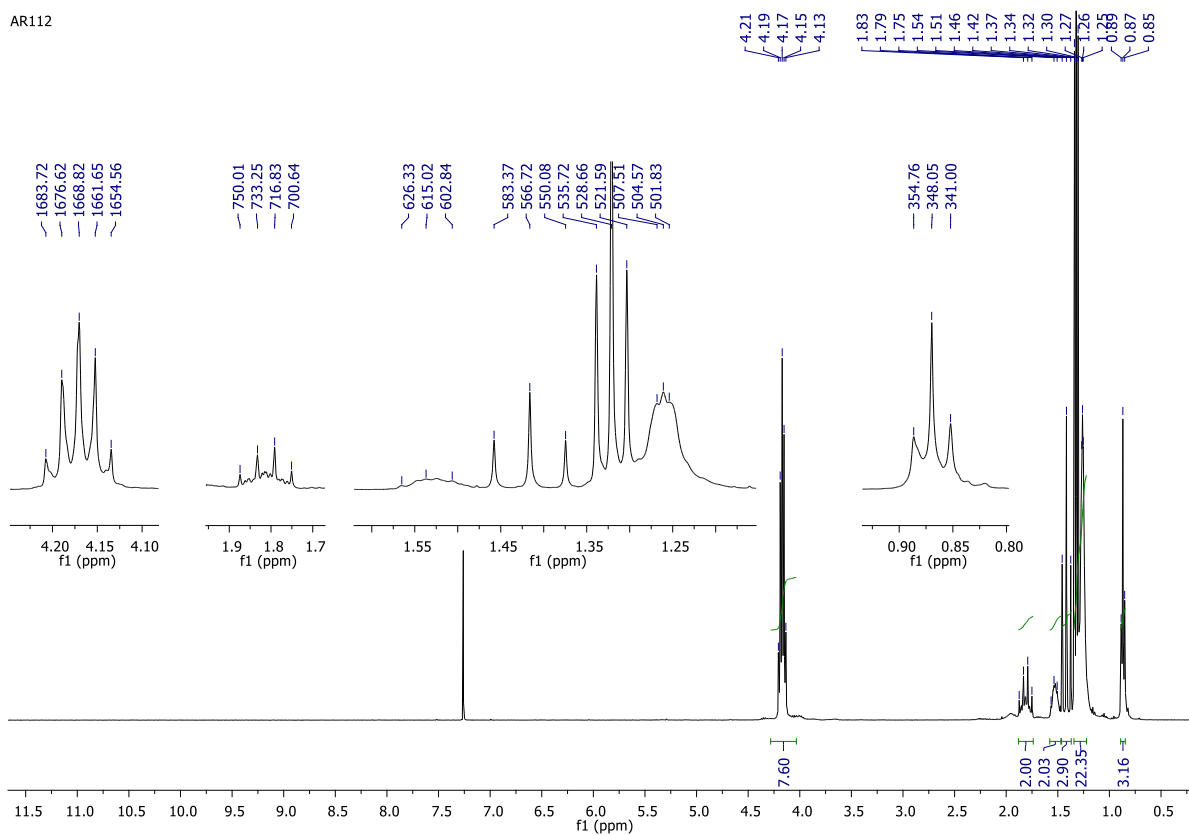
414,46 g/mol

Yellow oil

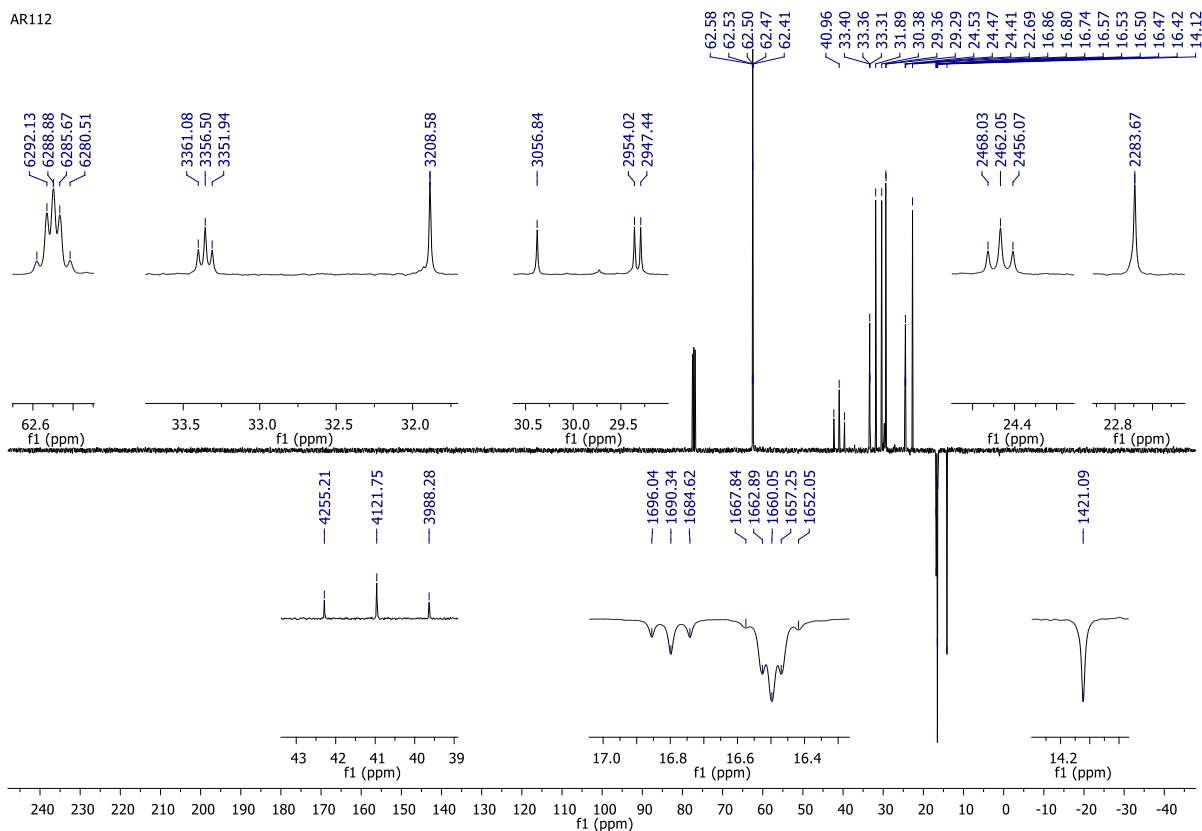
AR112

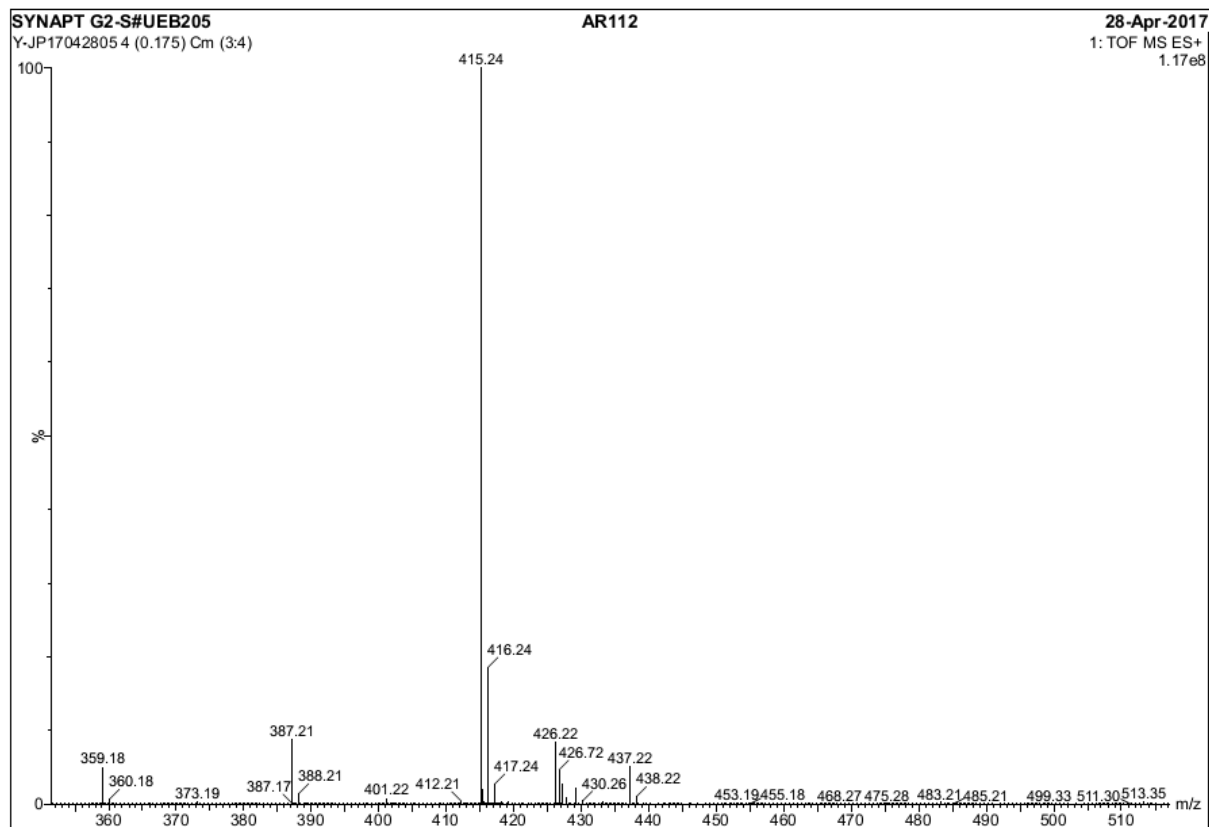
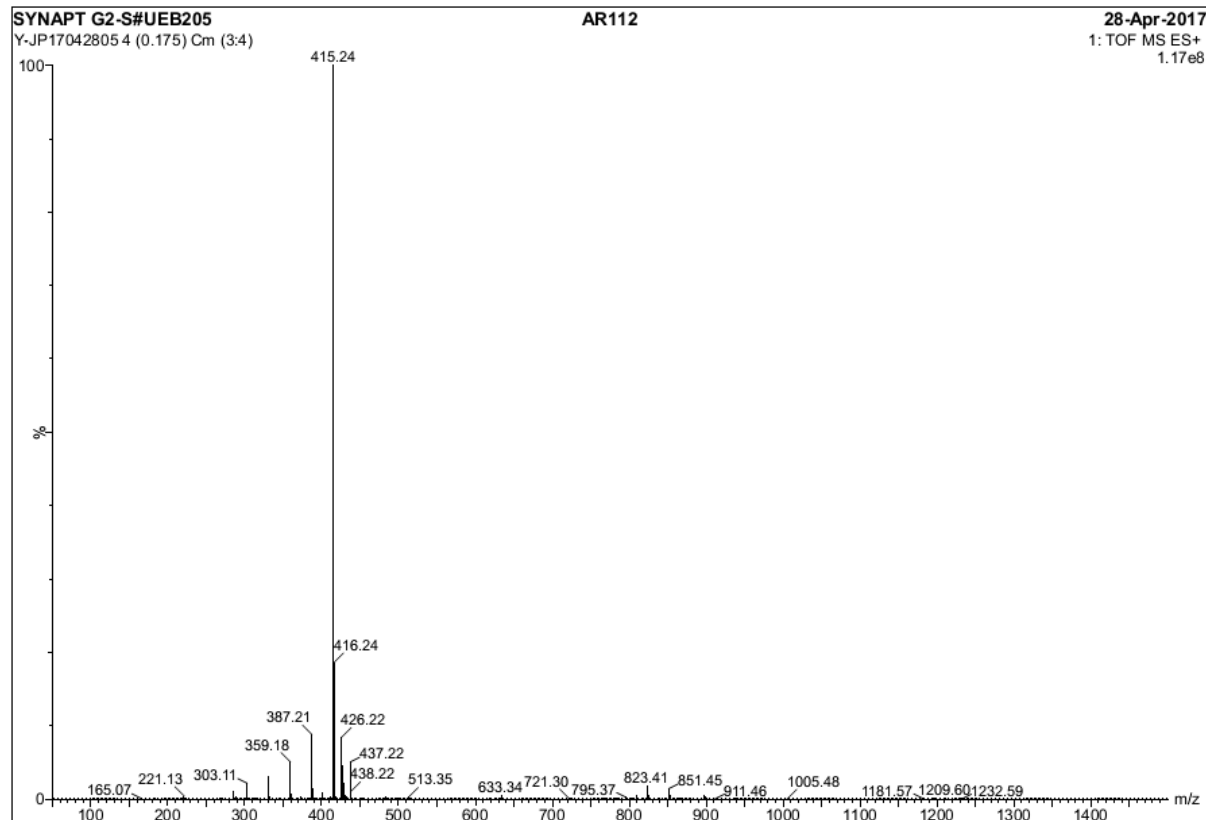


AR112



AR112





Elemental Composition Report

Page 1

Single Mass Analysis

Tolerance = 1.0 mDa / DBE: min = -1.5, max = 50.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

3153 formula(e) evaluated with 5 results within limits (up to 50 best isotopic matches for each mass)

Elements Used:

C: 1-100 H: 0-100 N: 0-20 O: 0-20 P: 0-2

SYNAPT G2-S#UEB205

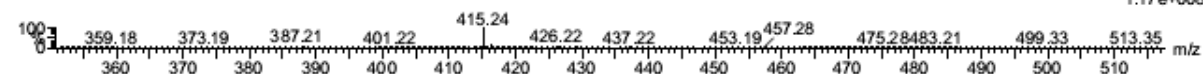
Y-JP17042805 4 (0.175) Cm (3:4)

AR112

28-Apr-2017

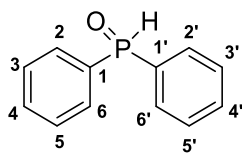
1: TOF MS ES+

1.17e+008



Minimum: -1.5
Maximum: 1.0 3.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula
415.2374	415.2378	-0.4	-1.0	-0.5	2068.3	0.012	98.85	C18 H41 O6 P2
	415.2377	-0.3	-0.7	1.5	2073.5	5.180	0.56	C11 H31 N10 O7
	415.2375	-0.1	-0.2	9.5	2073.7	5.366	0.47	C21 H32 N6 O P
	415.2365	0.9	2.2	5.5	2075.0	6.712	0.12	C15 H33 N10 P2
	415.2380	-0.6	-1.4	2.5	2080.9	12.590	0.00	C6 H28 N18 O2 P

S16: ^{31}P , ^1H and ^{13}C NMR spectra of diphenylphosphine oxide (10)

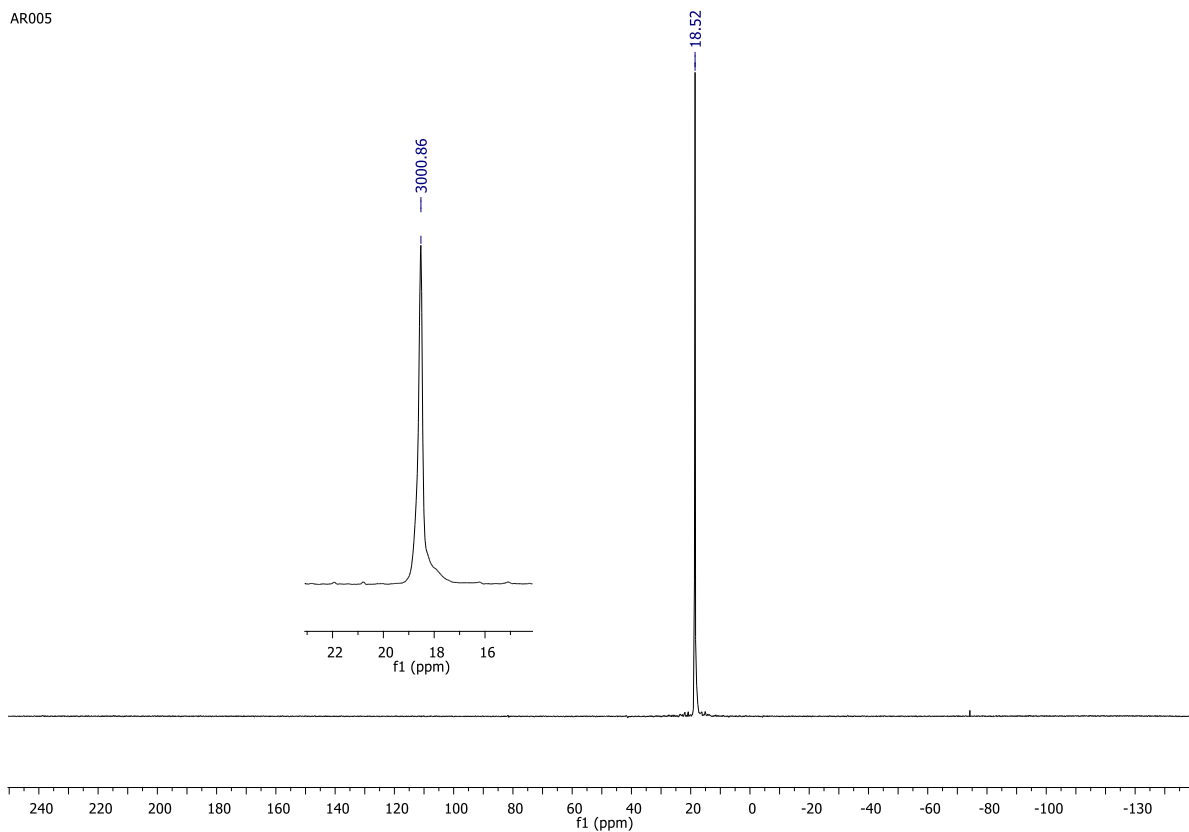
Diphenylphosphine oxide

 $\text{C}_{12}\text{H}_{11}\text{OP}$

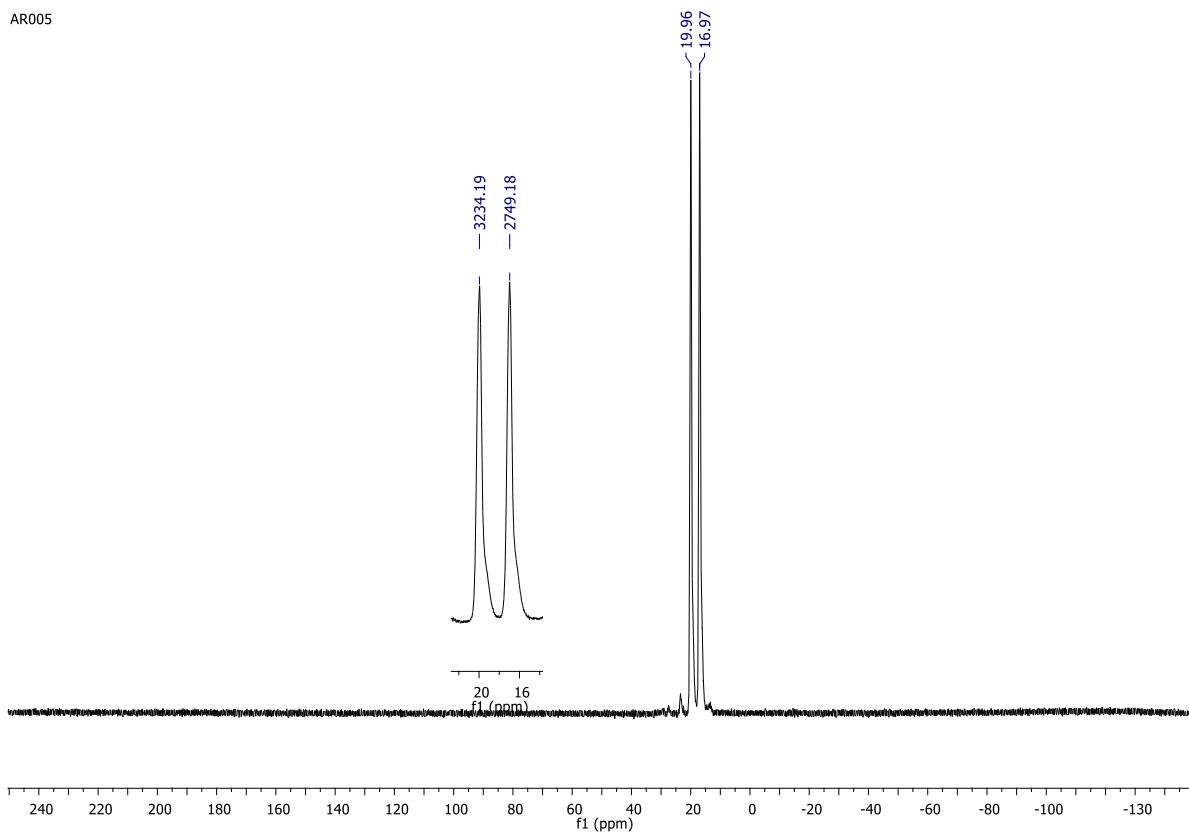
202,19 g/mol

White solid

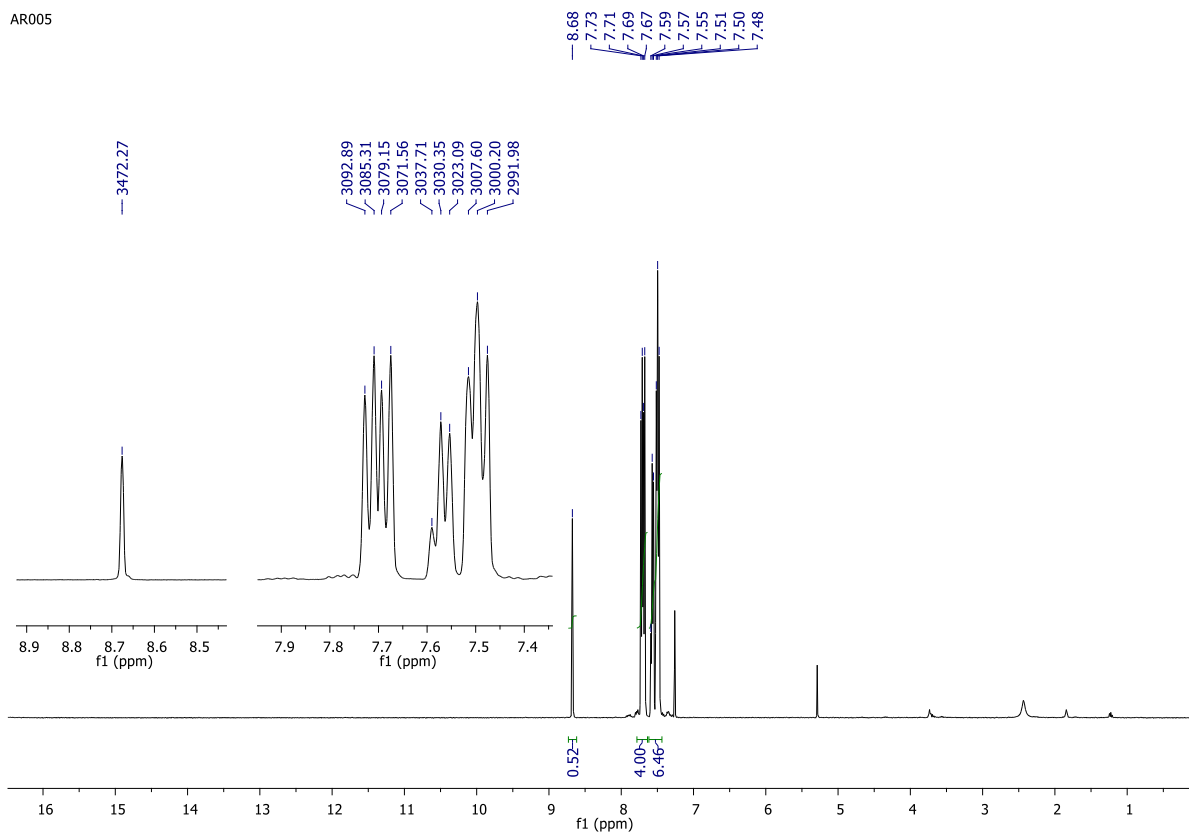
AR005



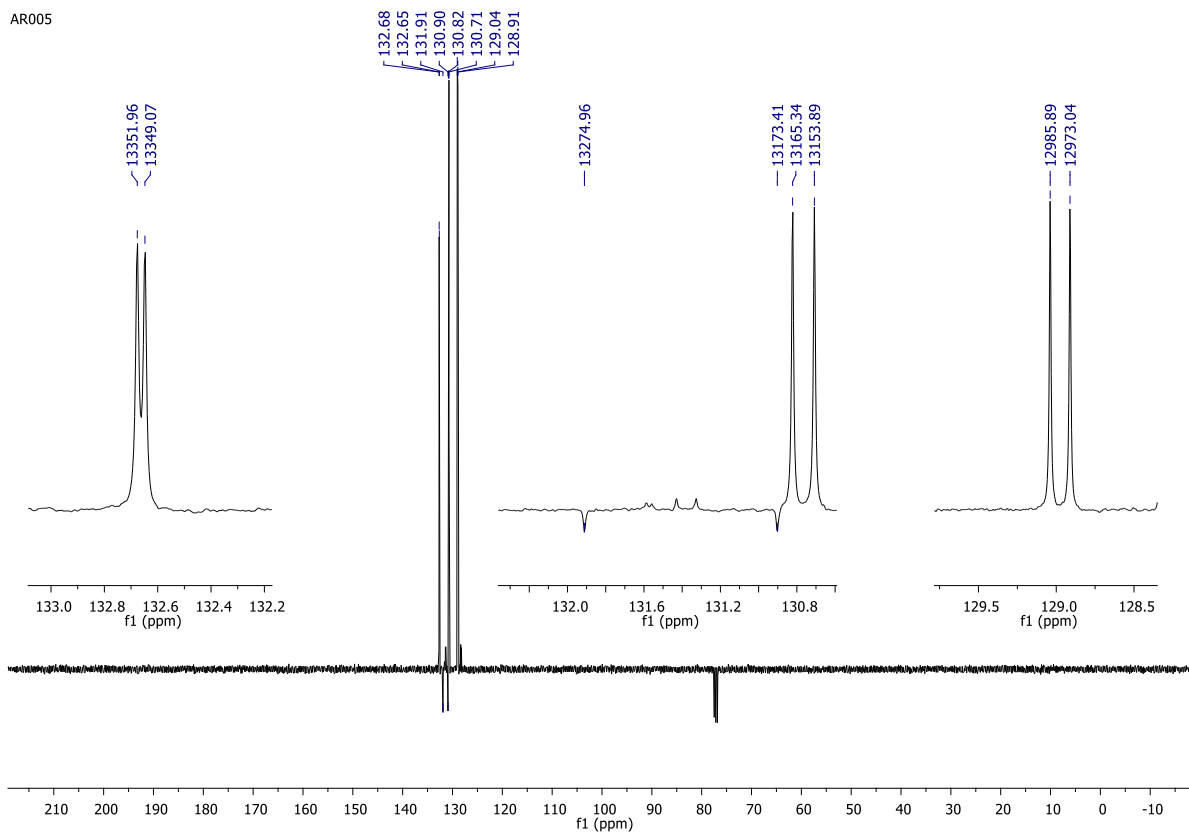
AR005

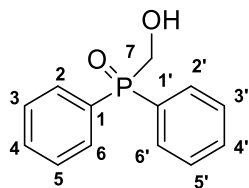


AR005



AR005



S17: ^{31}P , ^1H and ^{13}C NMR spectra of diphenylhydroxymethylphosphine oxide (11)

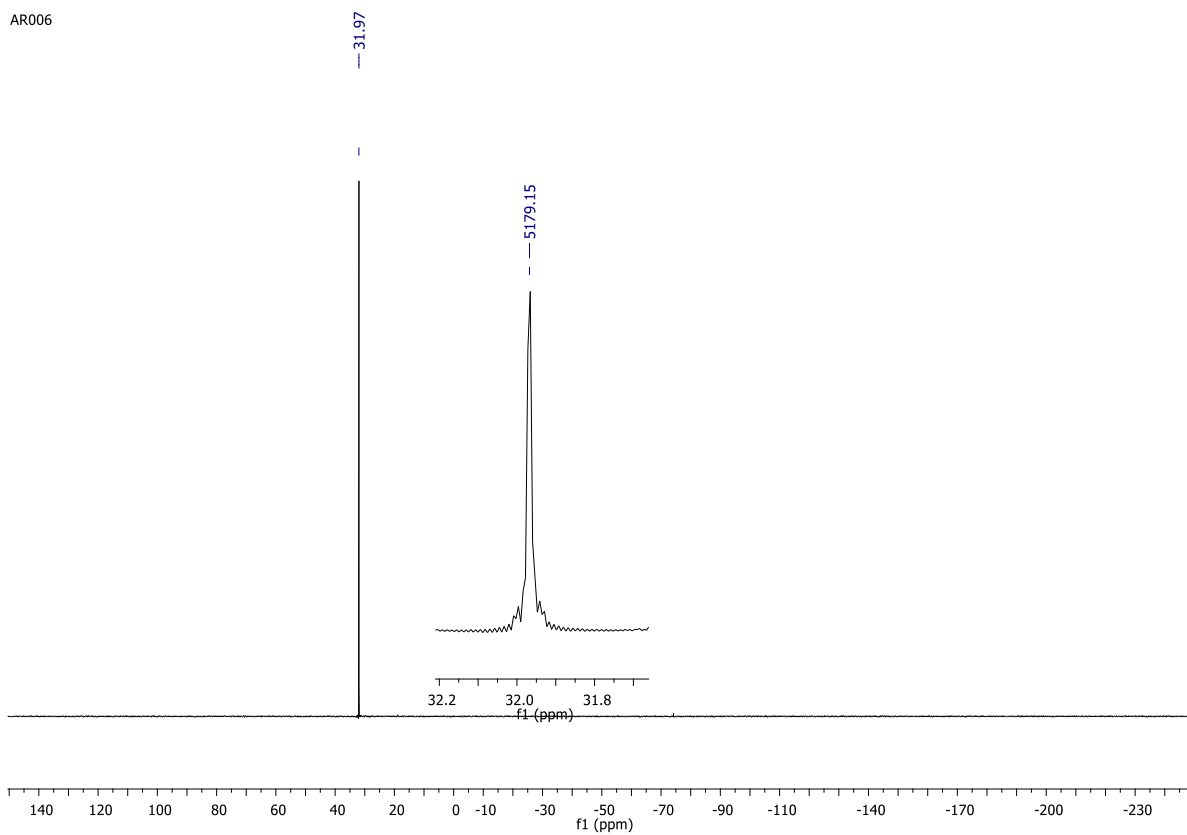
(hydroxymethyl)diphenylphosphine oxide

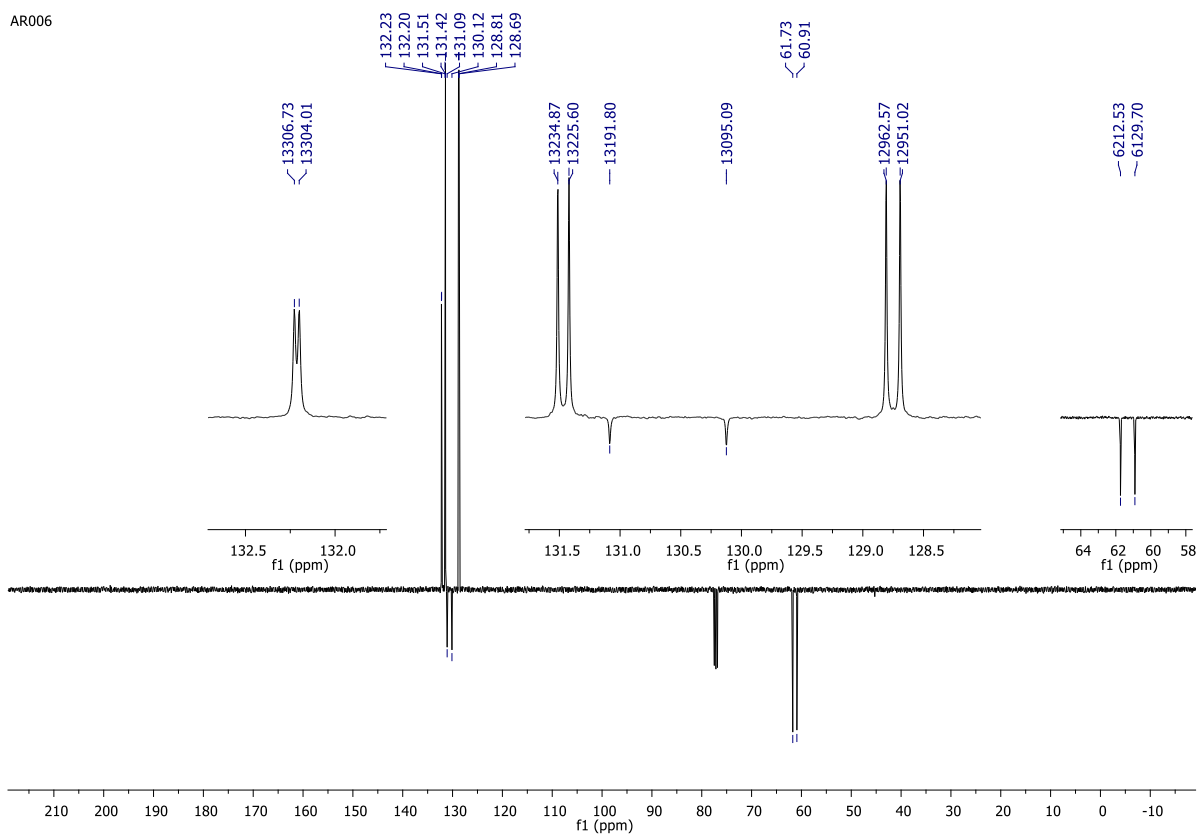
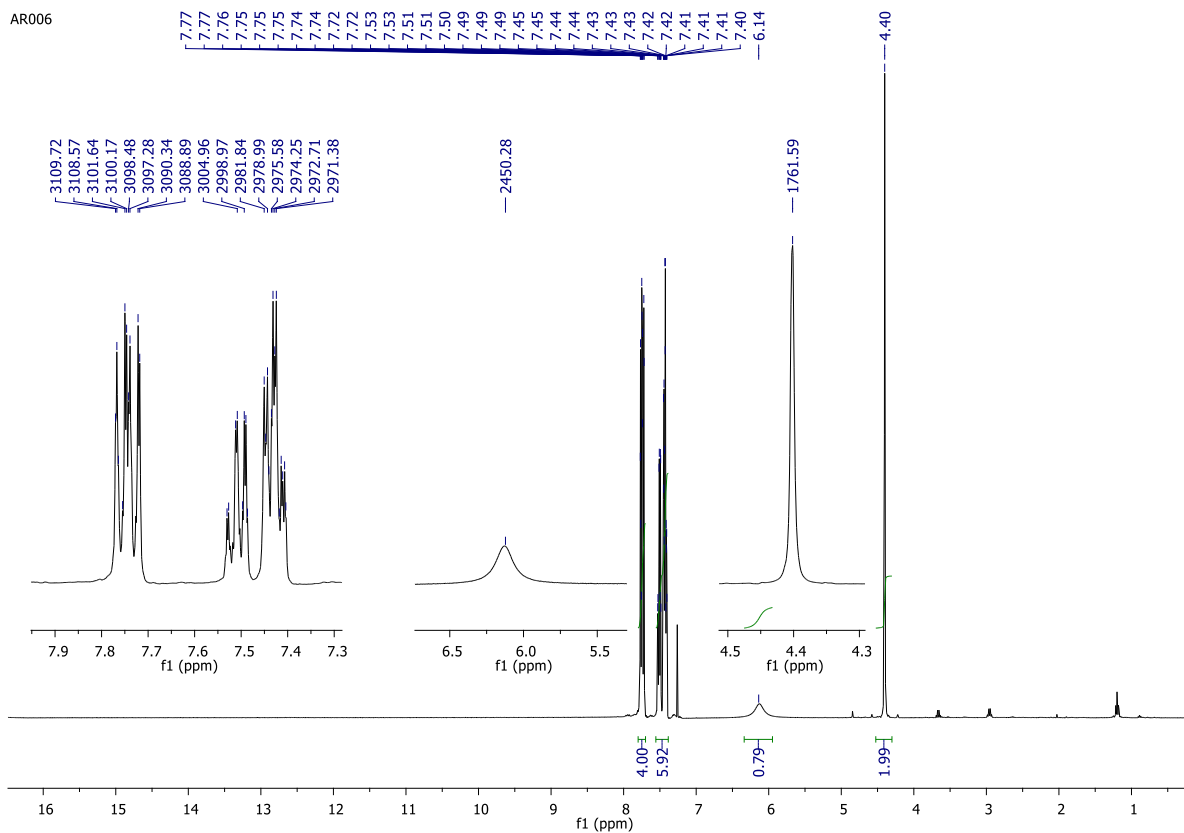
 $\text{C}_{13}\text{H}_{13}\text{O}_2\text{P}$

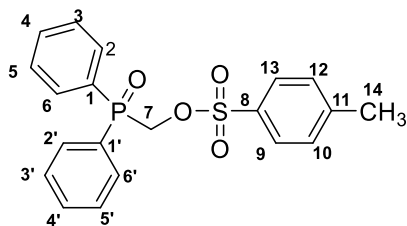
232,22 g/mol

White solid

AR006





S18: ^{31}P , ^1H and ^{13}C NMR spectra of (diphenylphosphoryl)methyl 4-methylbenzenesulfonate (12)

(diphenylphosphoryl)methyl 4-methylbenzenesulfonate

 $\text{C}_{20}\text{H}_{19}\text{O}_4\text{SP}$

386,40 g/mol

White solid

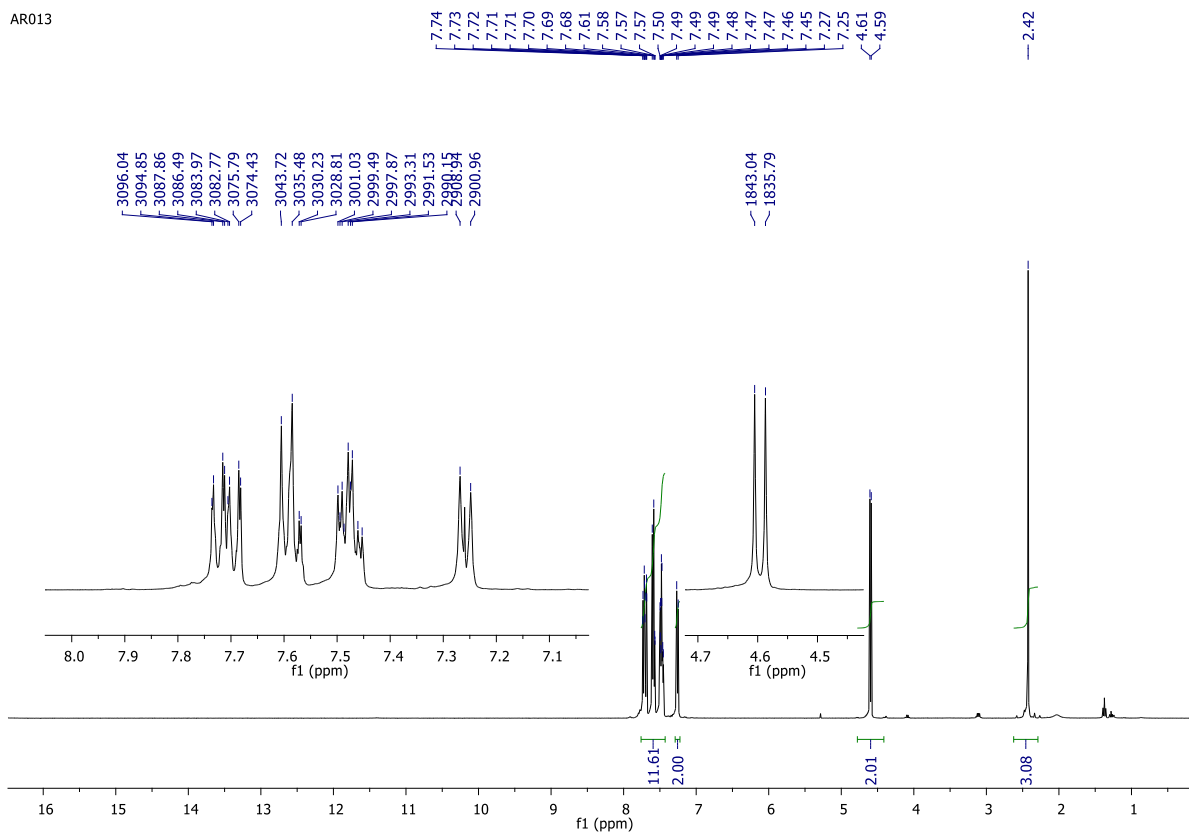
AR013

— 25.81

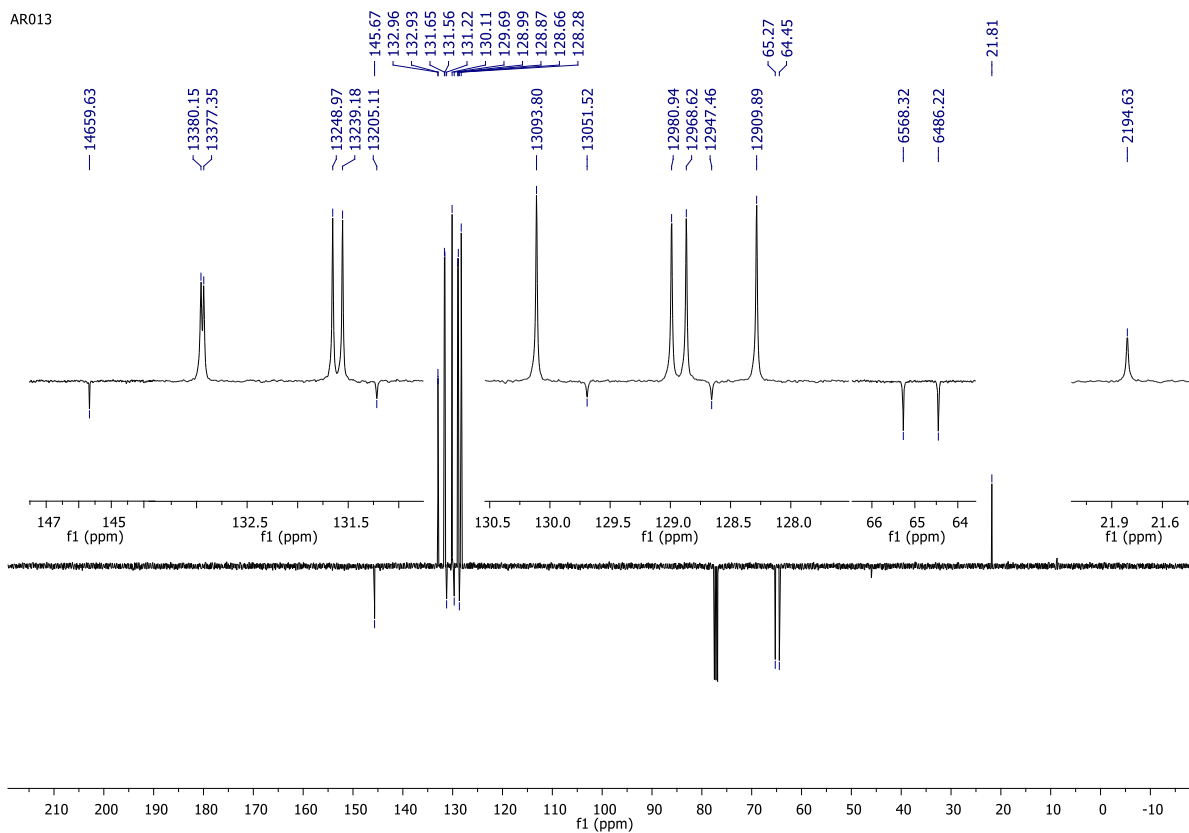
— 4181.26

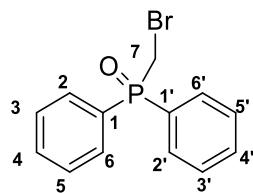
26.0 25.5
f1 (ppm)140 120 100 80 60 40 20 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 -220 -230
f1 (ppm)

AR013



AR013



S19: ^{31}P , ^1H and ^{13}C NMR spectra of (bromomethyl)diphenylphosphine oxide (13)

(bromomethyl)diphenylphosphine oxide

 $\text{C}_{13}\text{H}_{12}\text{OBrP}$

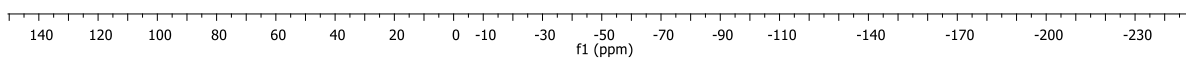
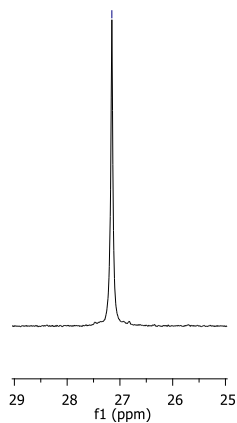
295,12 g/mol

White solid

AR008

— 27.15

— 4399.12



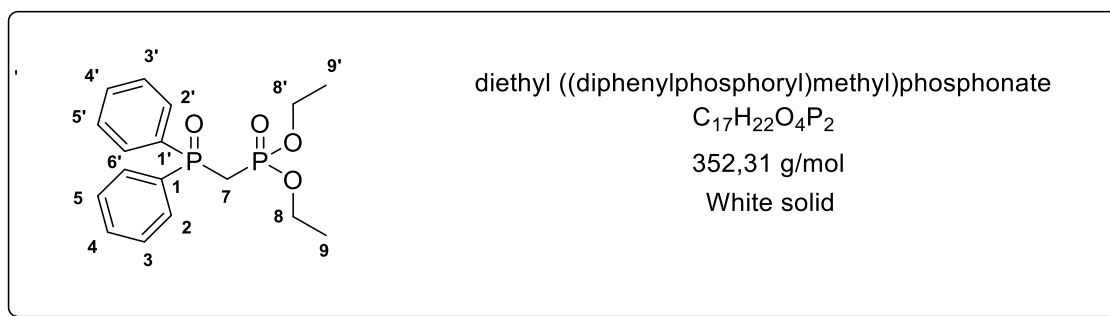
13C NMR spectrum of compound 10a in CDCl₃.

Chemical structure of 10a: O=C1C=CC(=C2C=CC(=CC=C2)C=C1)C3=CC=CC=C3

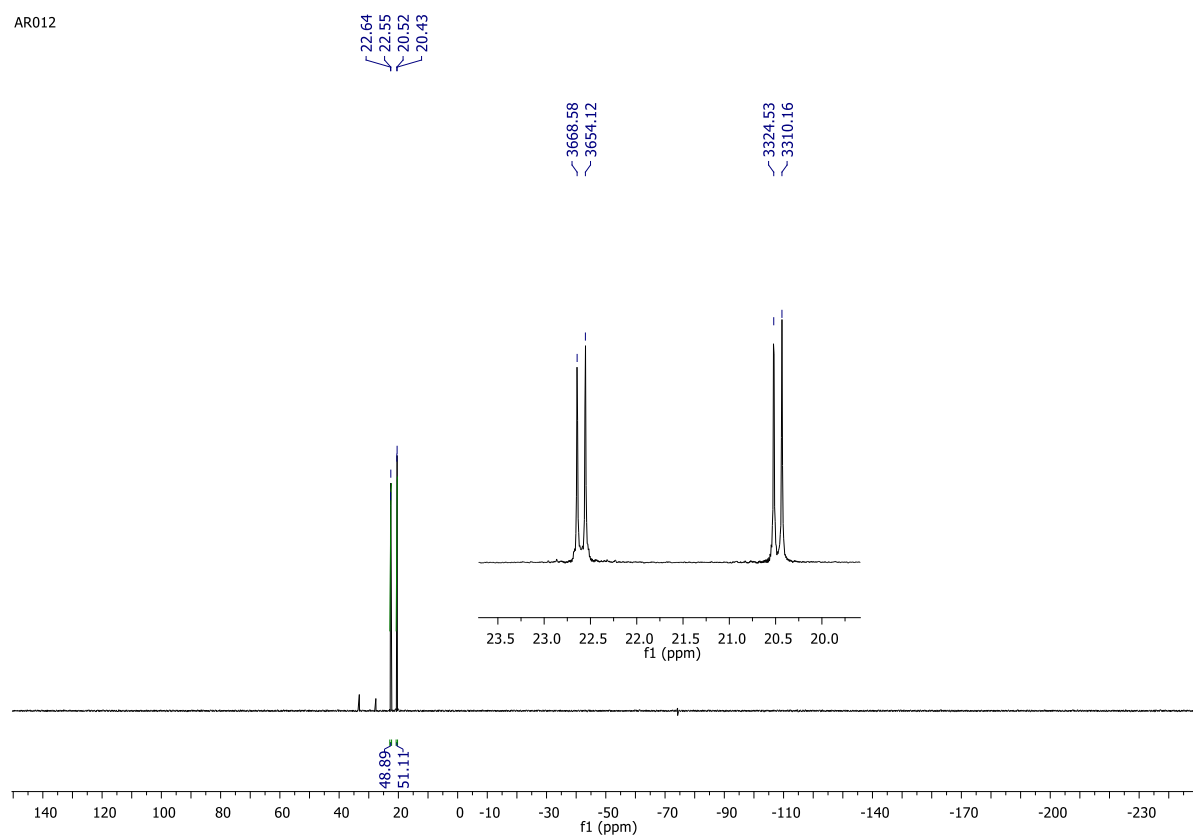
Peak list (ppm):

Region	Peak (ppm)
Aromatic	1356.11, 1353.31, 13245.81, 13236.37, 13146.13, 13041.19, 12976.60, 12964.40
Carbonyl	197.69, 197.62, 197.53, 197.63, 197.59, 197.95, 198.83
Solvent (CDCl ₃)	77.48, 77.16, 76.84
Aliphatic	2413.08, 2343.46, 23.98, 23.29

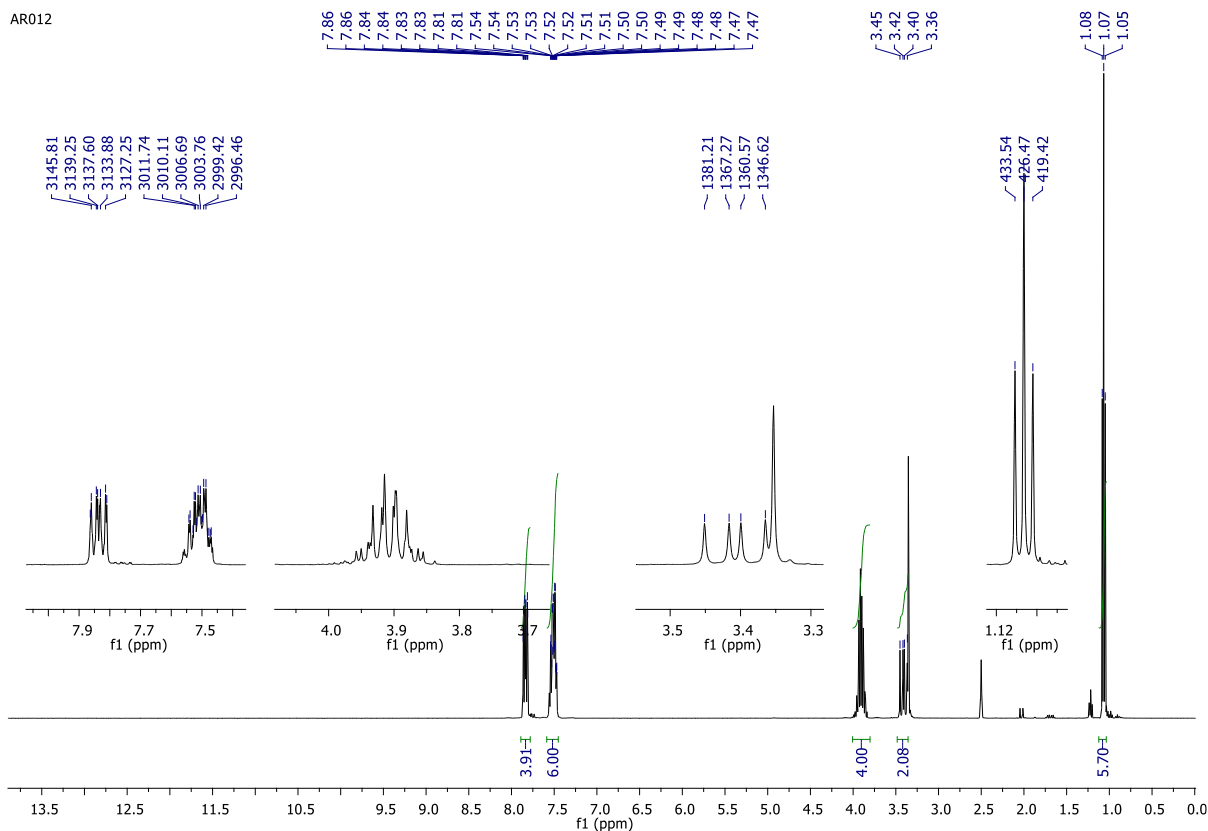
Inset: Aliphatic region (20-30 ppm) showing peaks at 2413.08 and 2343.46 ppm.

S20: ^{31}P , ^1H and ^{13}C NMR spectra of diethyl ((diphenylphosphoryl)methyl)phosphonate (14)

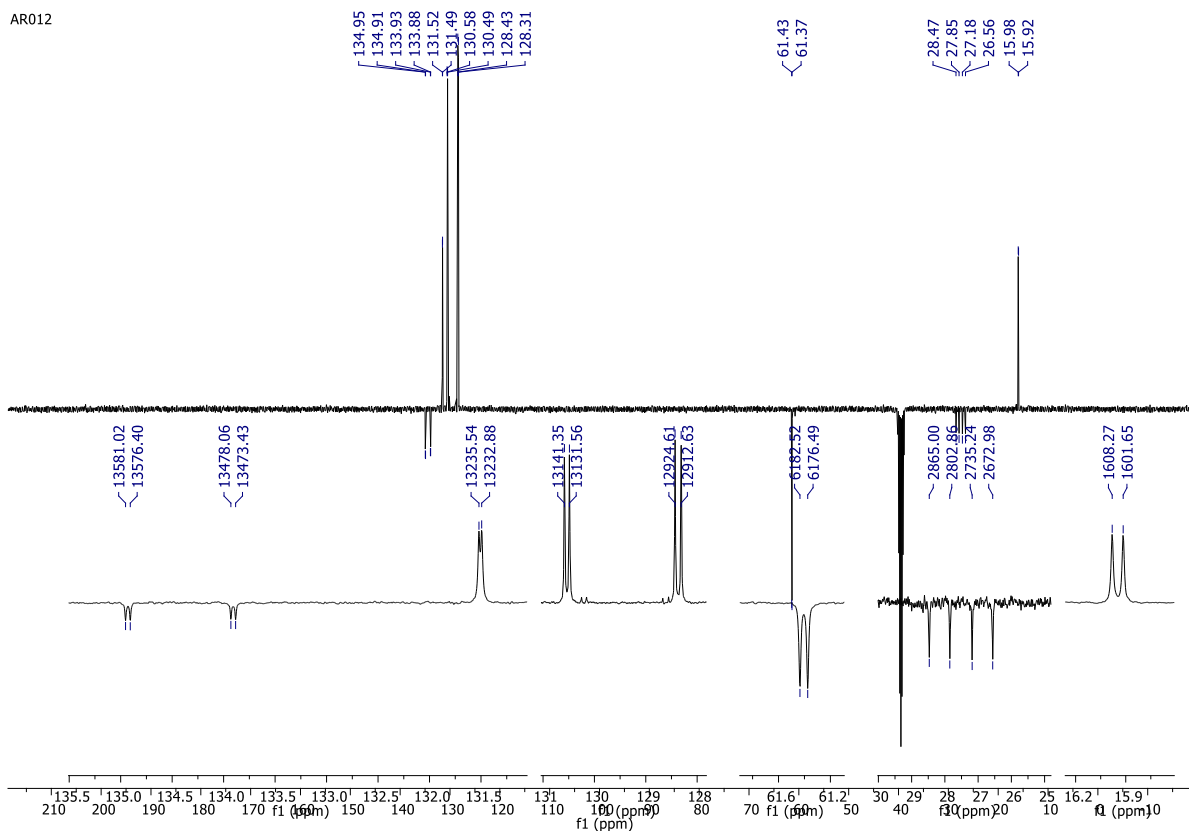
AR012

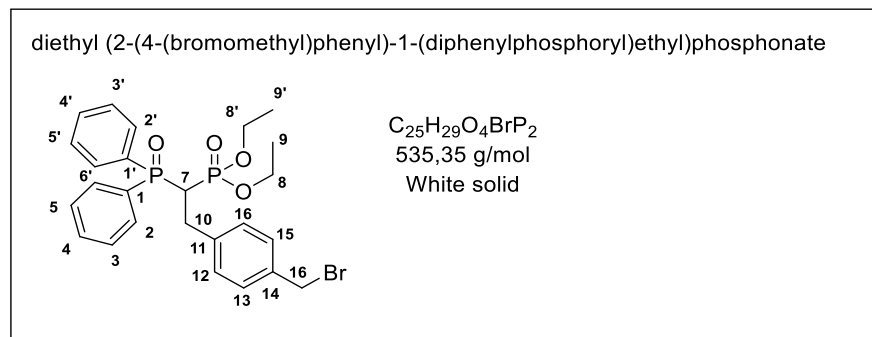


AR012

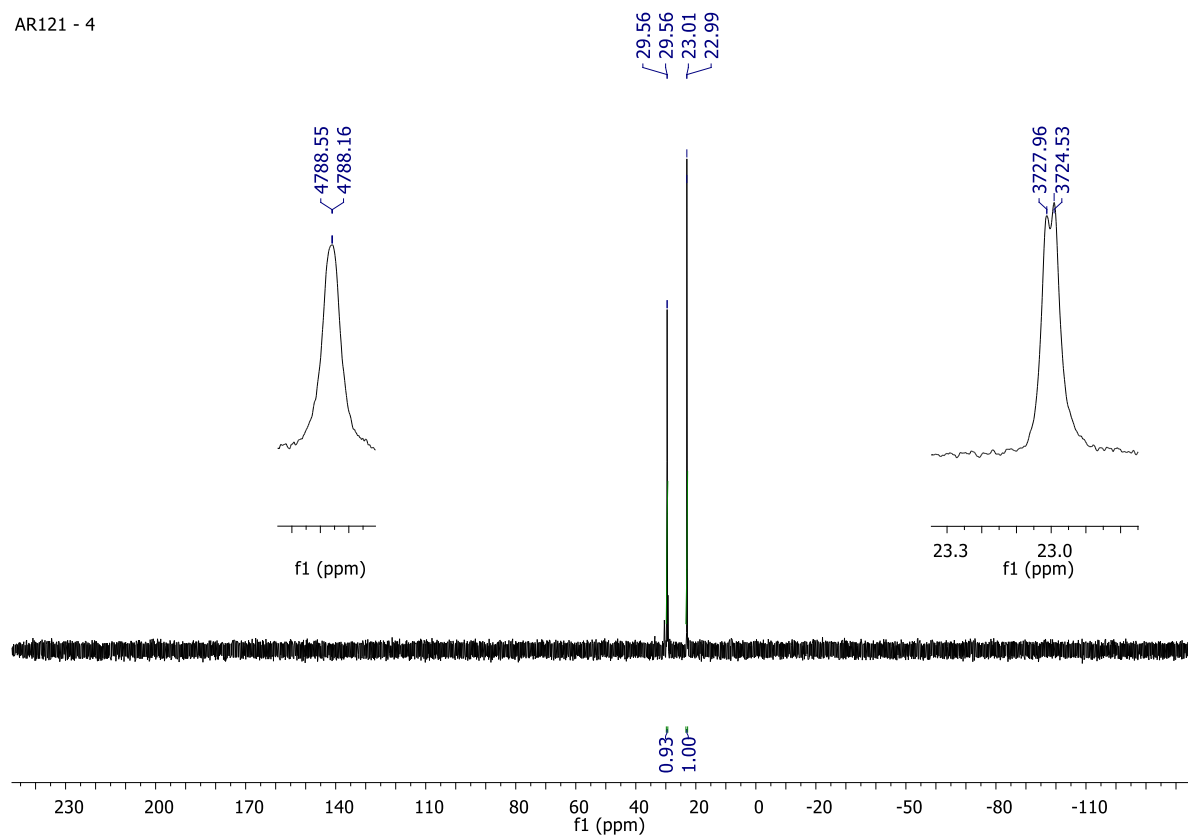


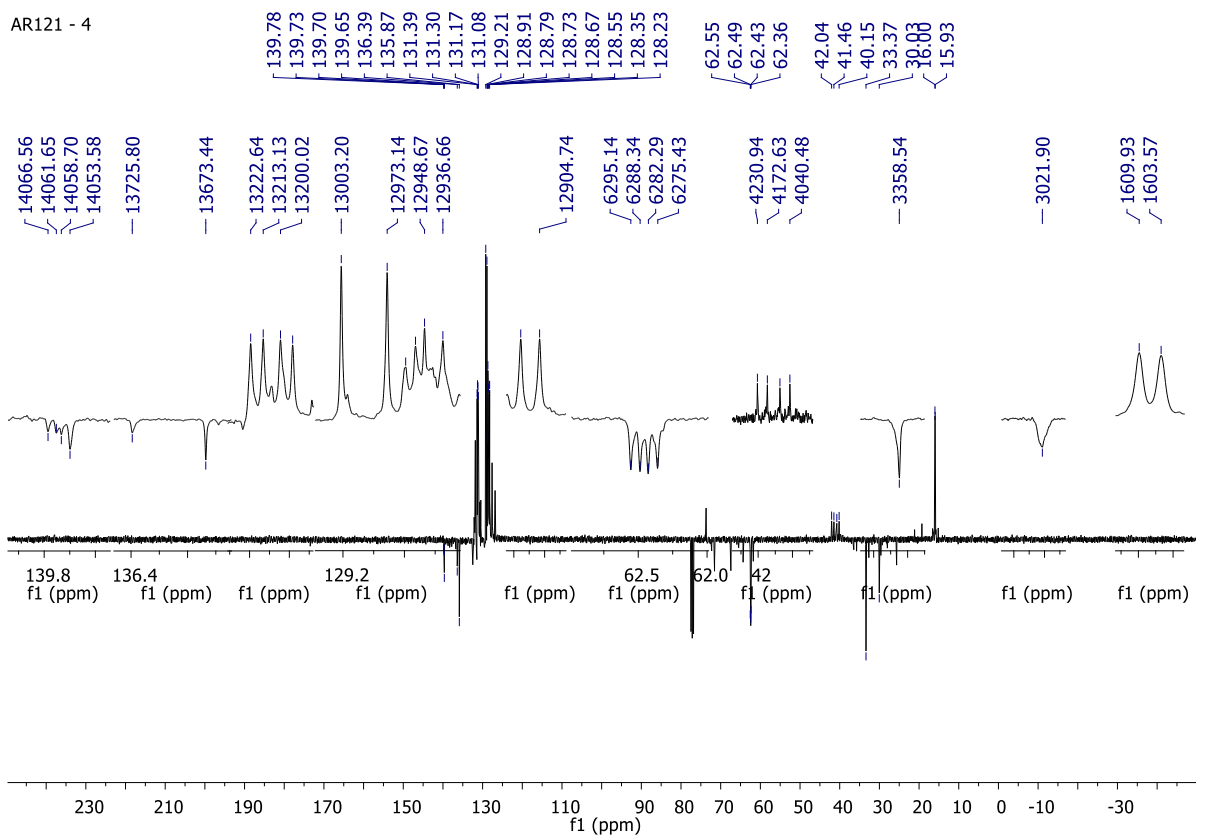
AR012

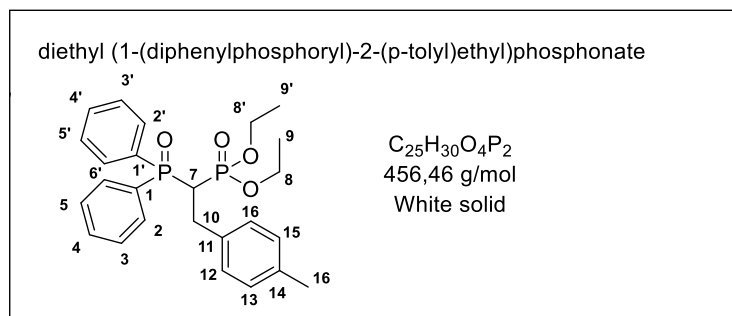


S21: ^{31}P , ^1H and ^{13}C NMR spectra of diethyl (2-(4-(bromomethyl)phenyl)-1-(diphenylphosphoryl)ethyl)phosphonate (15a)

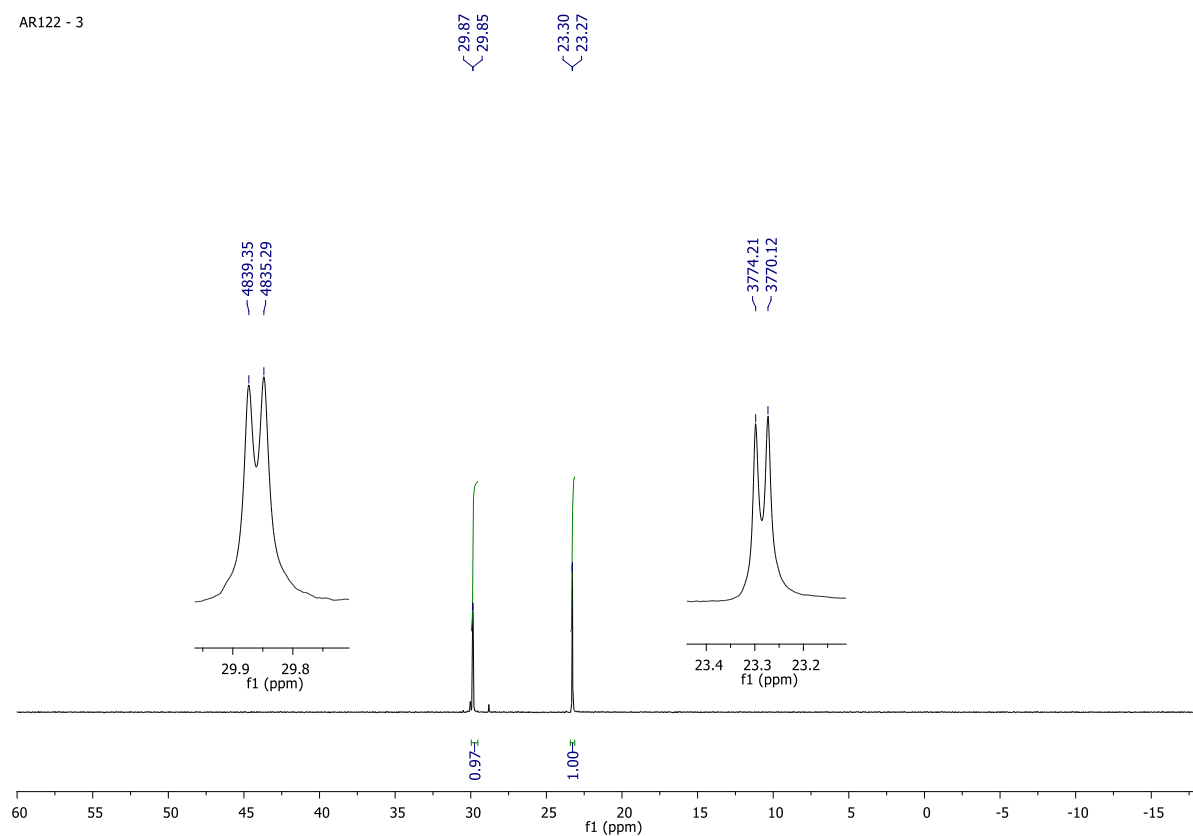
AR121 - 4



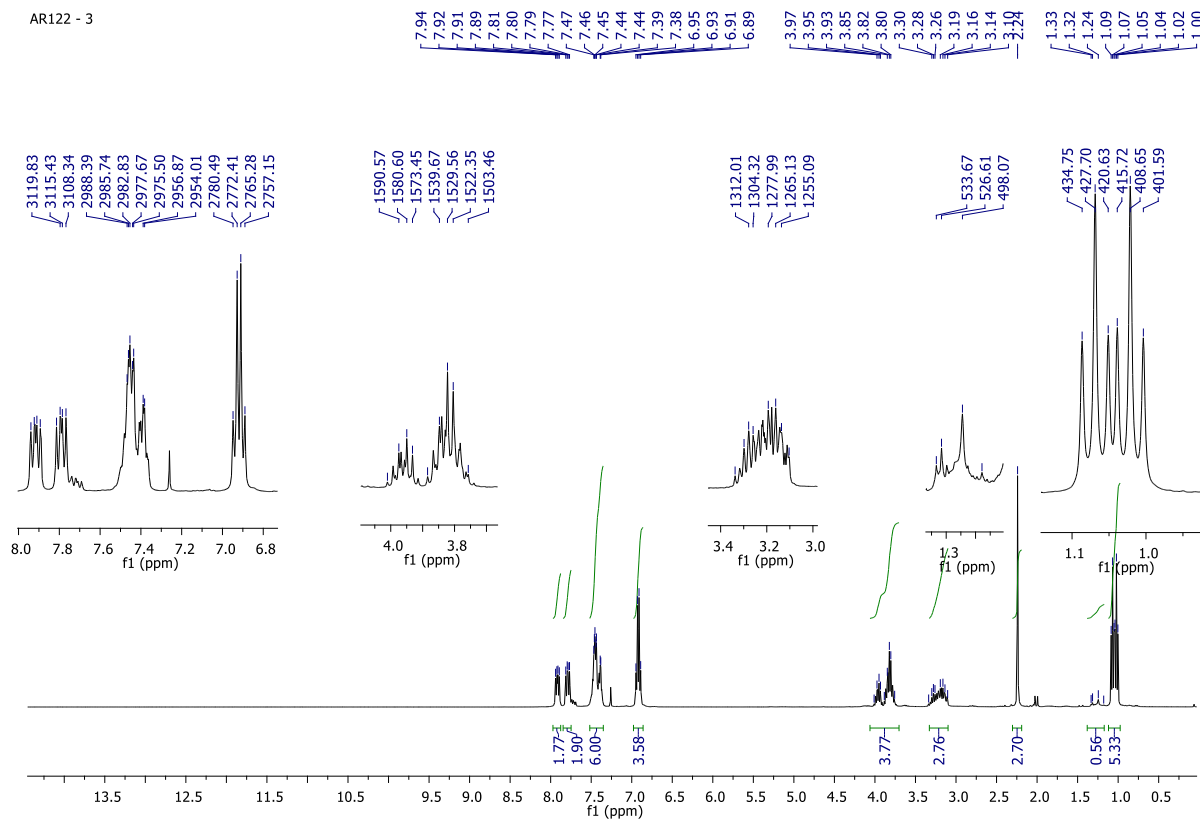


S22: ^{31}P , ^1H , and ^{13}C NMR spectra and mass analysis of diethyl (1-(diphenylphosphoryl)-2-(p-tolyl)ethyl)phosphonate (15b)

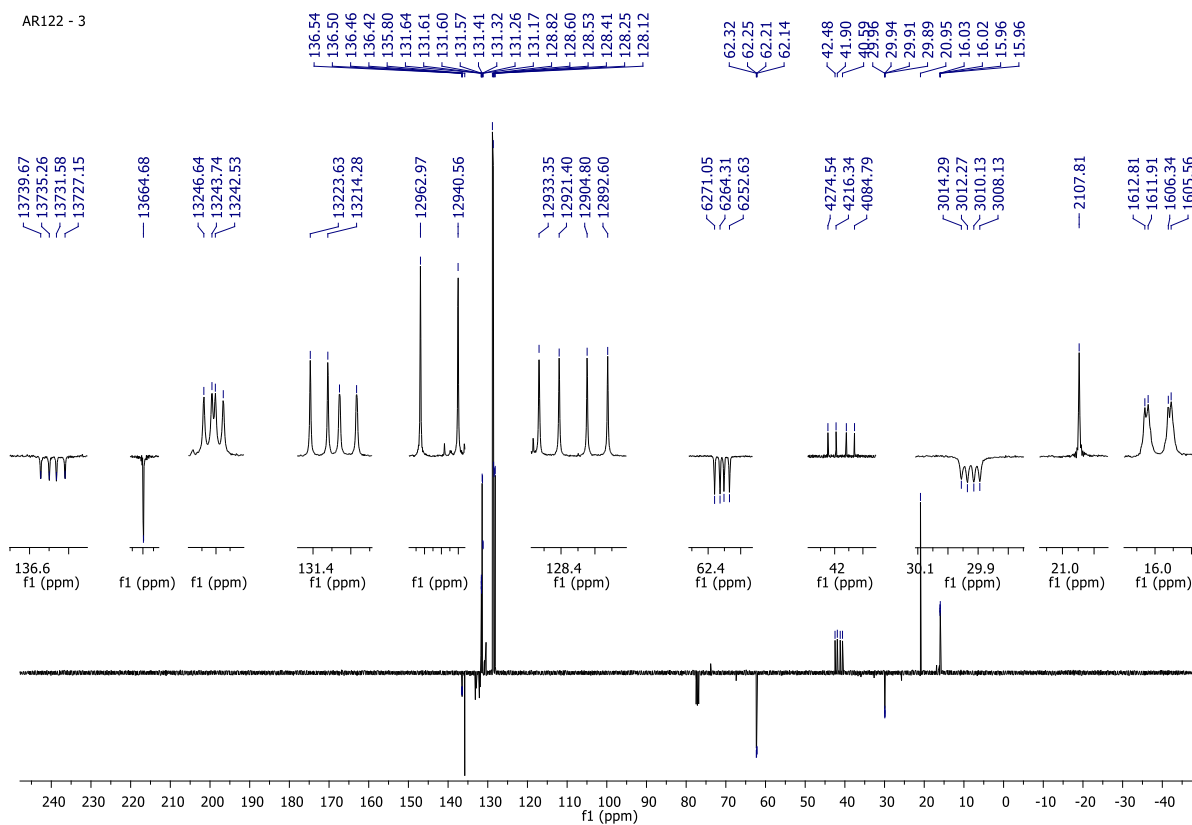
AR122 - 3



AR122 - 3



AR122 - 3

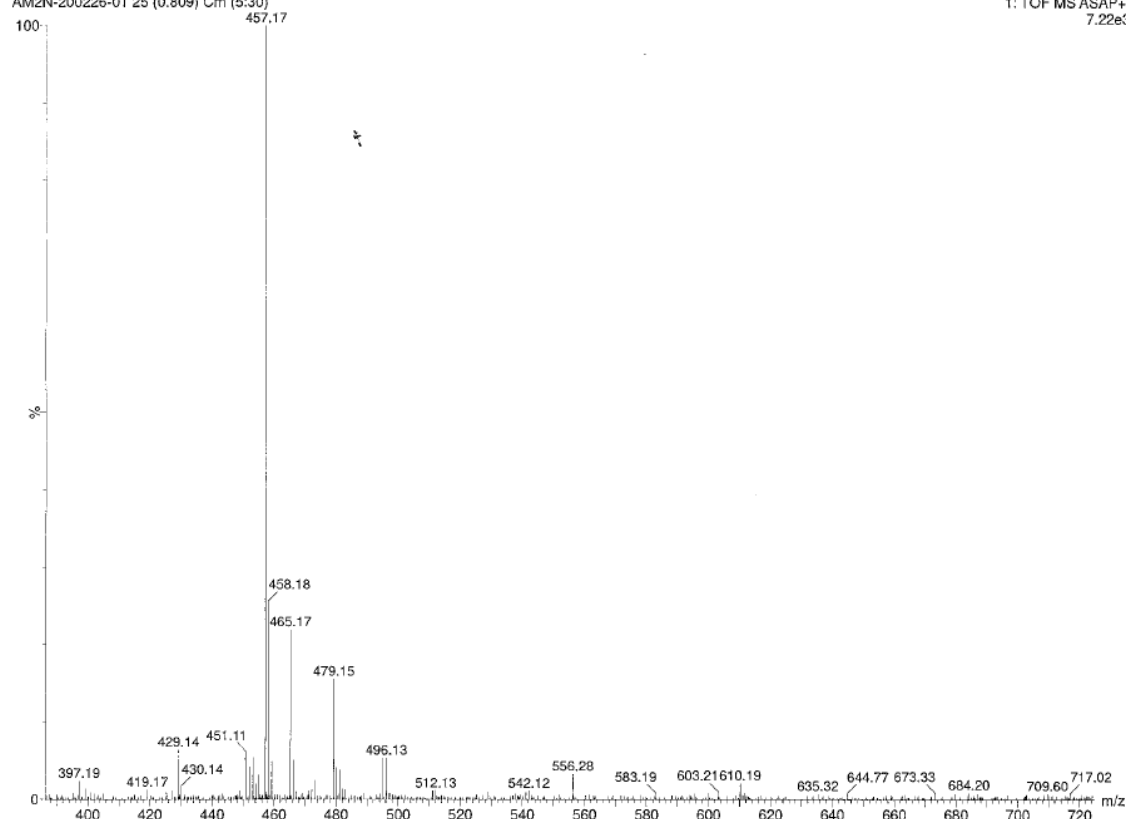


Synapt G2S UEB205

AR121-4

26-Feb-2020

AM2N-200226-01 25 (0.809) Cm (5:30)

1: TOF MS ASAP+
7.22e3

High Resolution Mass Result

Analysis Info

Sample Name

AR122-3

Acquisition Date

2/24/2020 2:16:32 PM

Instrument / Ser#

micrOTOF-Q 228888.10300

Acquisition Parameter

Source Type

ESI

Ion Polarity

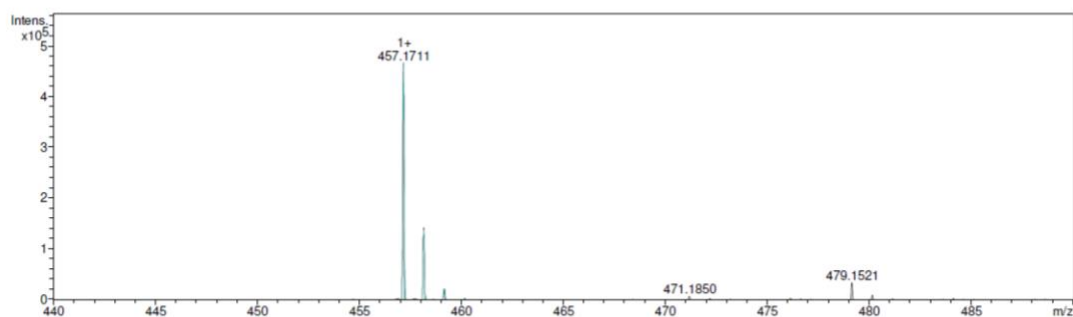
Positive

Scan Begin

50 m/z

Scan End

2200 m/z



Meas. m/z	#	Ion Formula	Score	m/z	err [mDa]	err [ppm]	rdb	e ⁻ Conf	N-Rule
457.1711	1	C ₂₆ H ₂₇ N ₄ P ₂	100.00	457.1705	-0.6	-1.3	16.5	even	ok
	2	C ₂₅ H ₃₁ O ₄ P ₂	35.58	457.1692	1.9	4.2	11.5	even	ok

Mass Result

Analysis Info

Sample Name **AR122-3**

Acquisition Date 2/24/2020 2:16:32 PM

Instrument / Ser# micrOTOF-Q 228888.10300

Acquisition Parameter

Source Type ESI Ion Polarity Positive Scan Begin 50 m/z Scan End 2200 m/z

