Supplementary Material

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

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E-mail : david.virieux@enscm.fr

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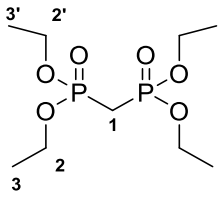
S1: ^{31}P, ^{1}H and ^{13}C NMR spectra of tetraethyl methylenebis(phosphonate) (1)

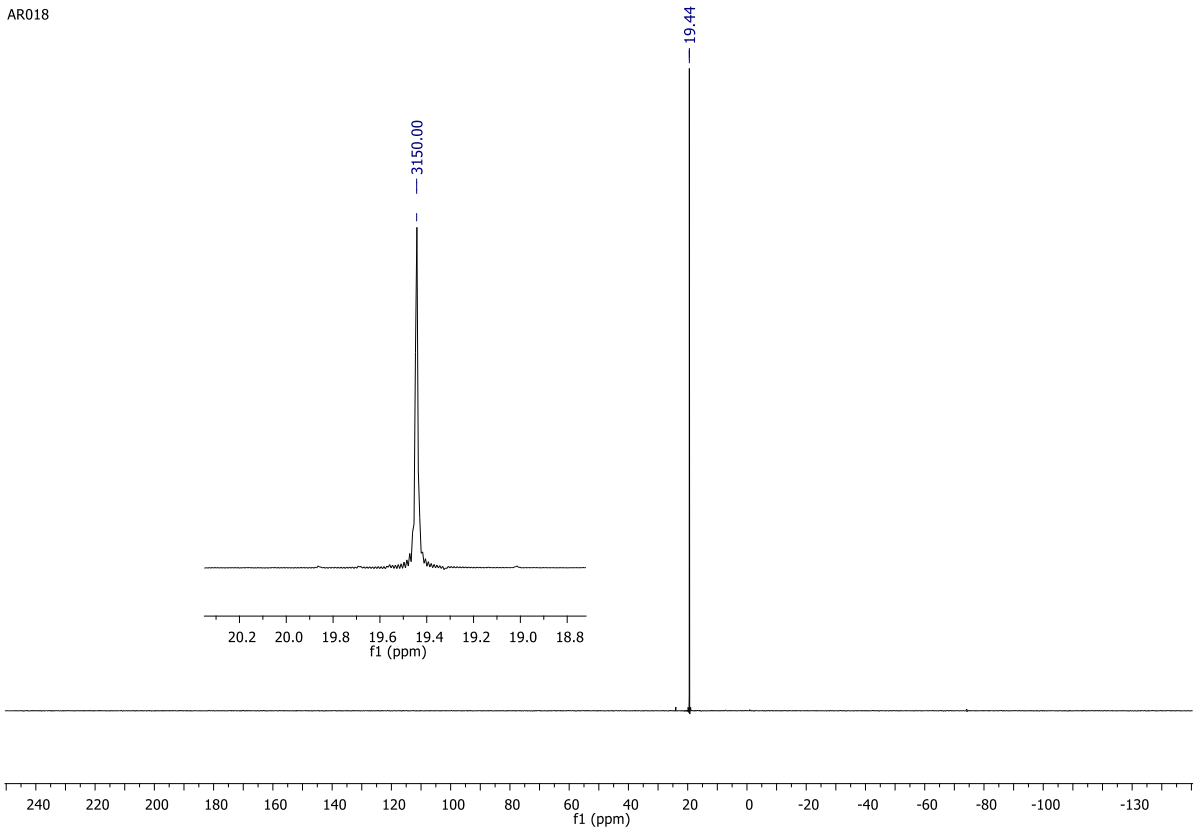


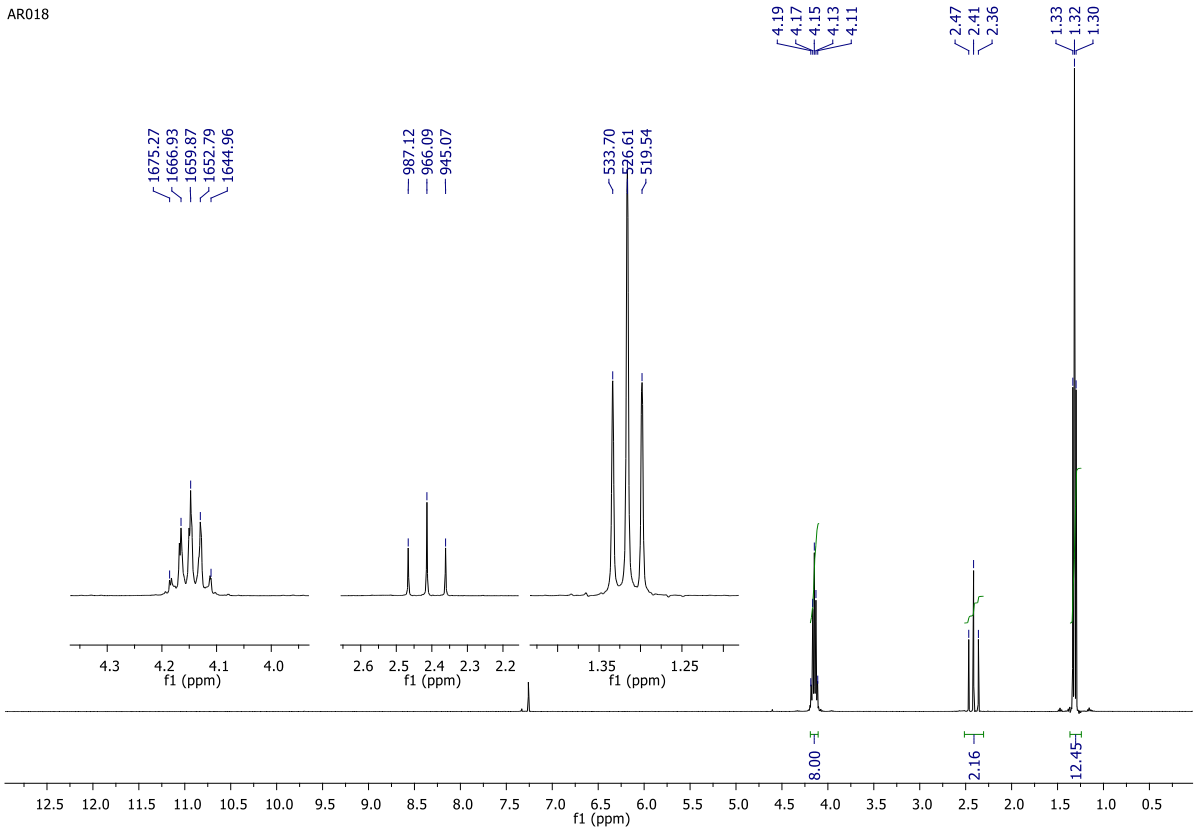
tetraethyl methylenebis(phosphonate)

$C_9H_{22}O_6P_2$

288,22 g/mol

Colorless oil

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4.15
4.13
4.11
2.47
2.41
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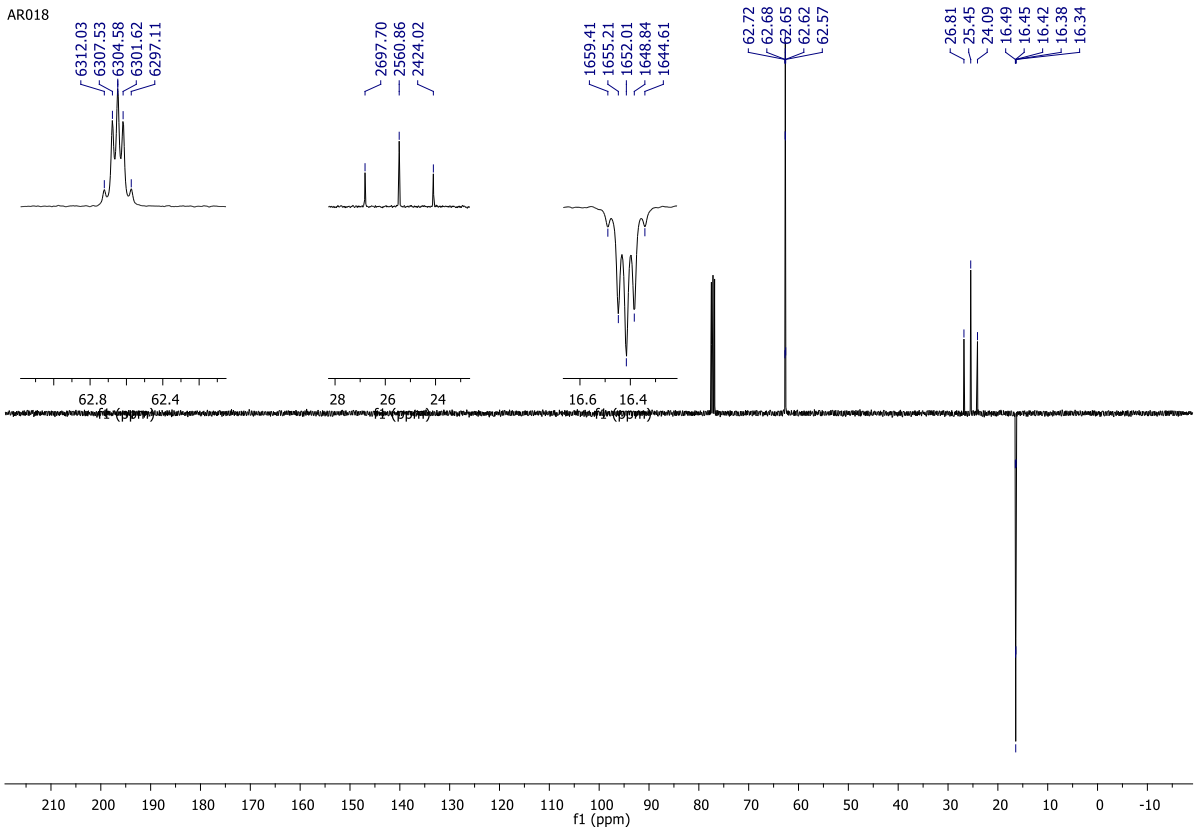
1.33
1.32
1.30
1675.27
1666.93
1659.87
1652.79
1644.96
987.12
966.09
945.07
533.70
526.61
519.54
8.00
2.16
12.45
f1 (ppm)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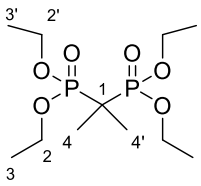
6304.58
6301.62
6297.11
2697.70
2560.86
2424.02
1659.41
1655.21
1652.01
1648.84
1644.61
62.72
62.68
62.65
62.62
62.57
26.81
25.45
24.09
16.49
16.45
16.42
16.38
16.34
f1 (ppm)

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

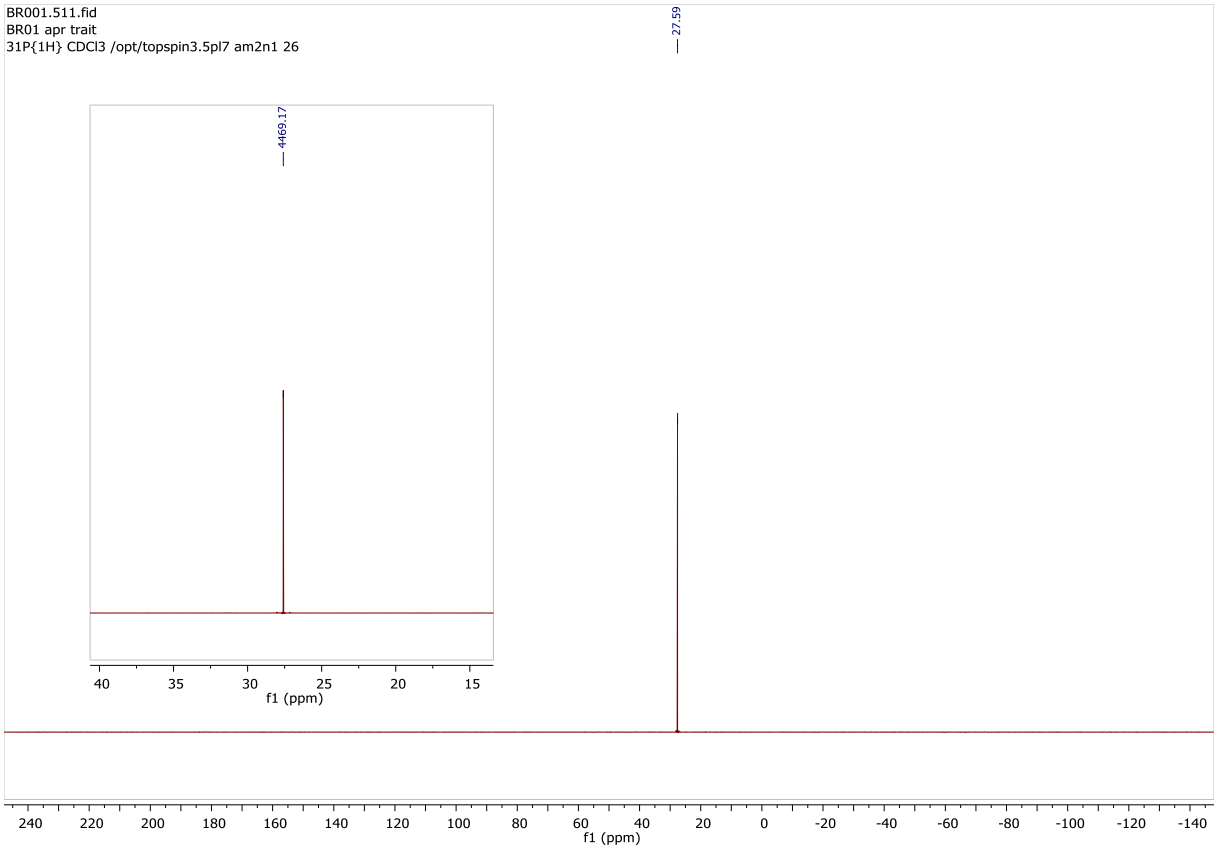


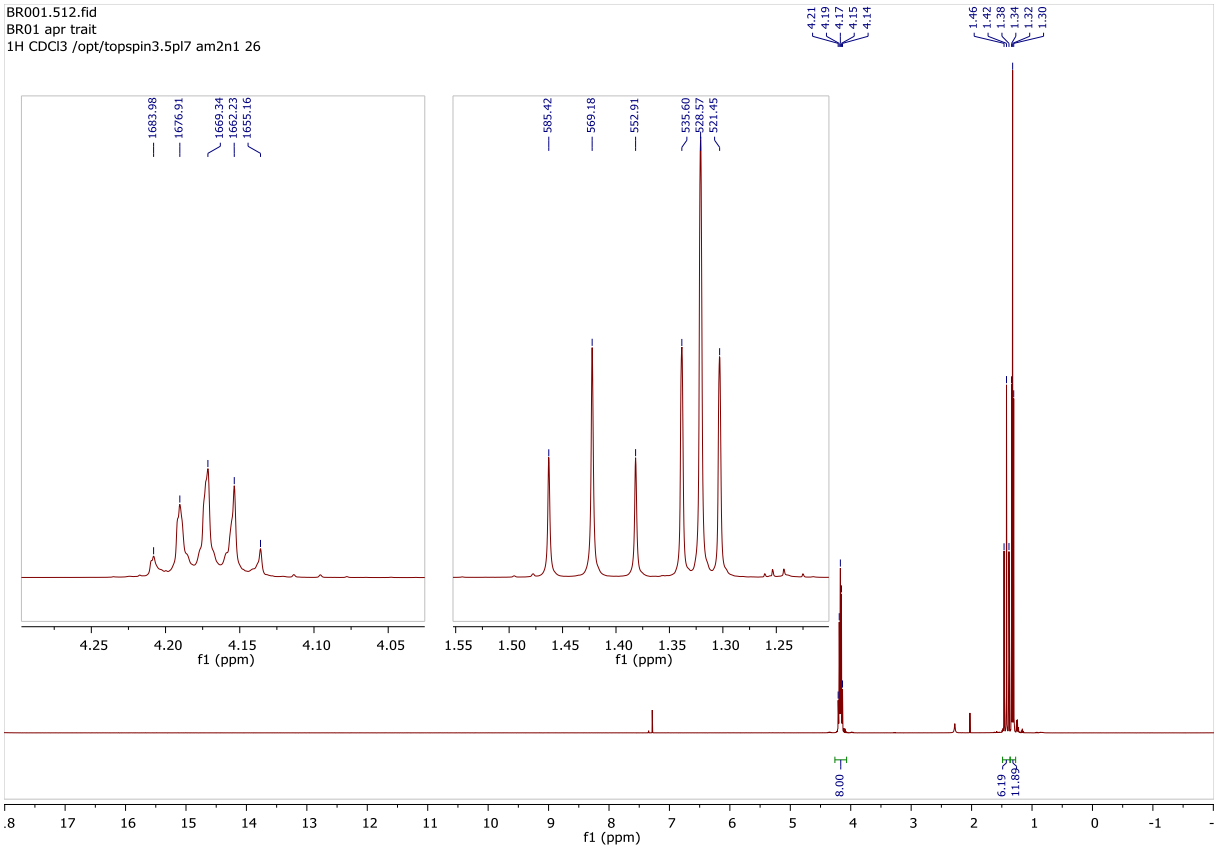
tetraethyl propane-2,2-diylbis(phosphonate)

$C_{11}H_{26}O_6P_2$

316,27 g/mol

Colorless oil

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BR01 apr trait
1H CDCl3 /opt/topspin3.5pl7 am2n1 26
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4.17
4.15
4.14
1.46
1.42
1.38
1.34
1.32
1.30
1683.98
1676.91
1669.34
1662.23
1655.16
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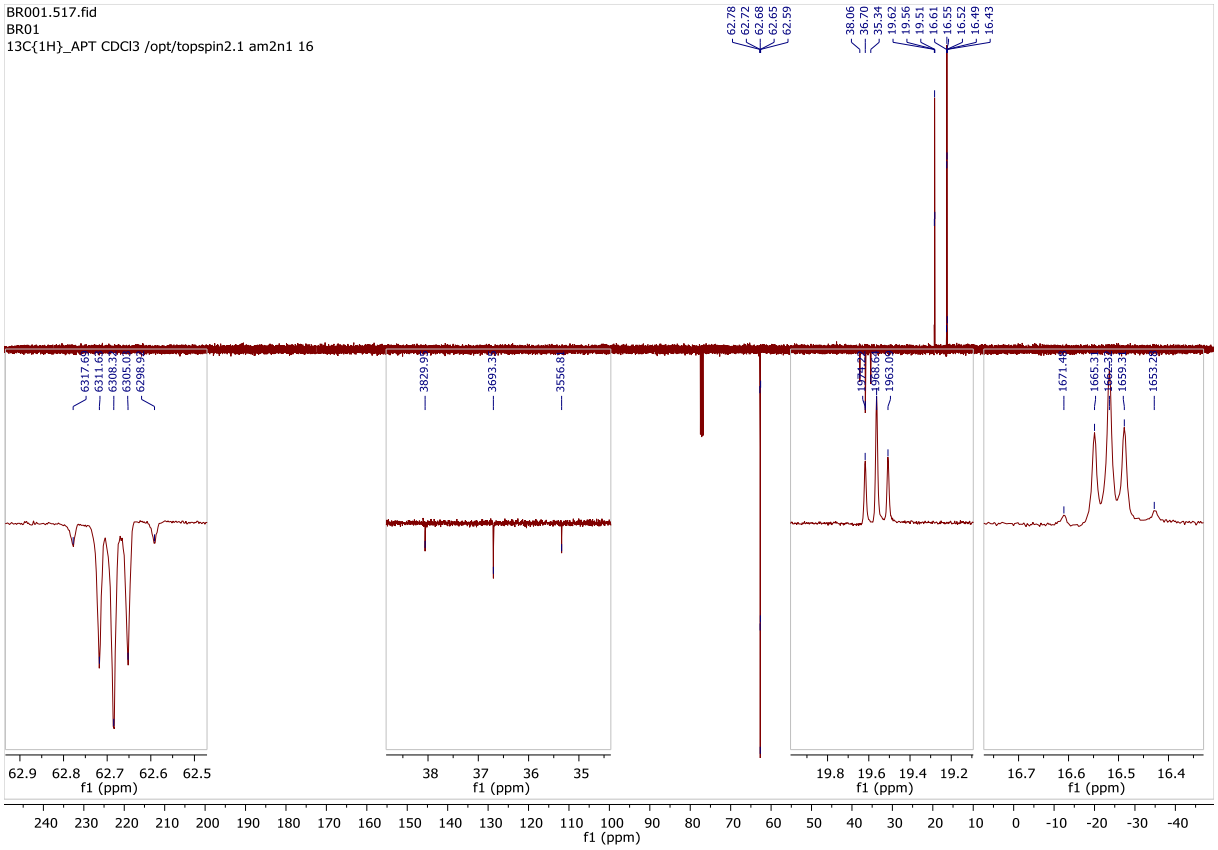
569.18
552.91
535.60
528.57
521.45
8.00
6.19
11.89
f1 (ppm)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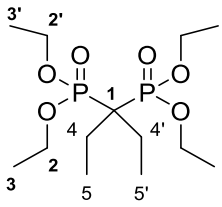
38.06
36.70
35.34
19.62
19.56
19.51
16.61
16.55
16.52
16.49
16.43
f1 (ppm)

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

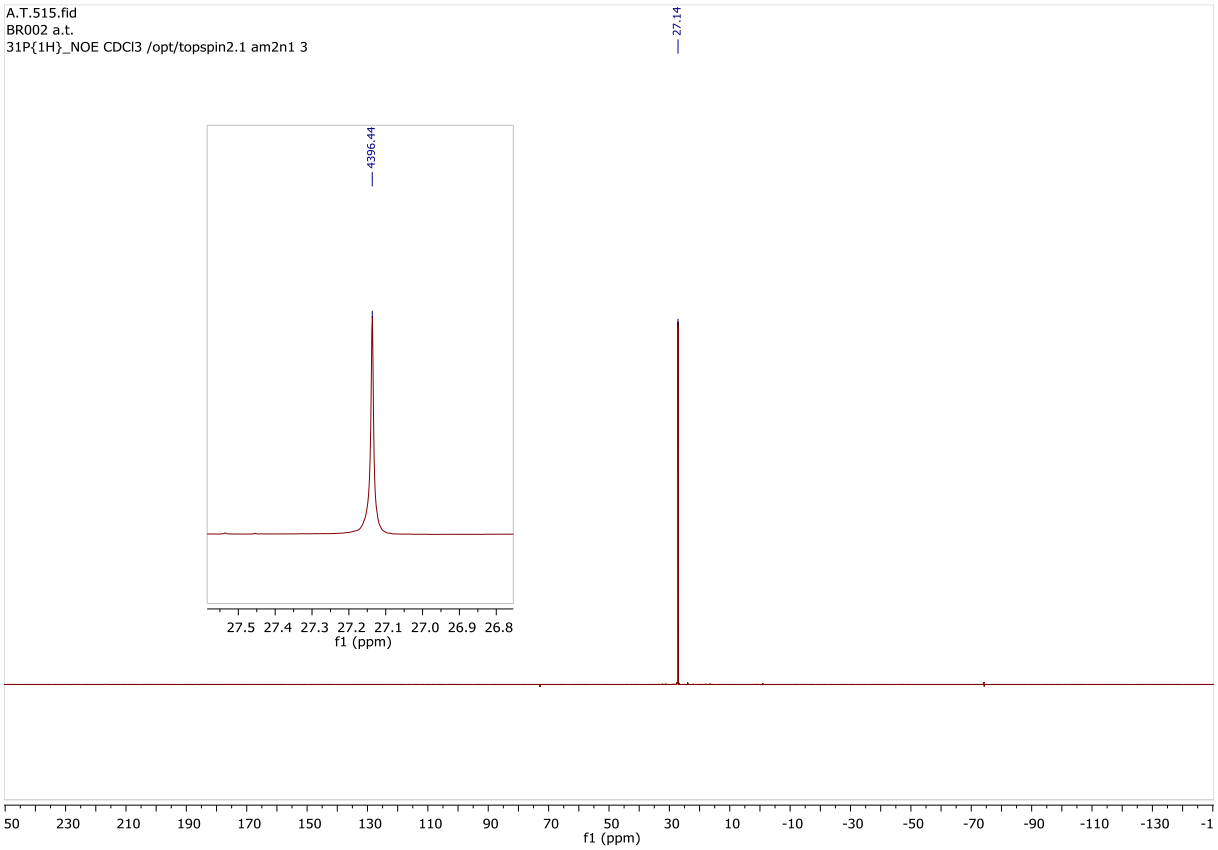


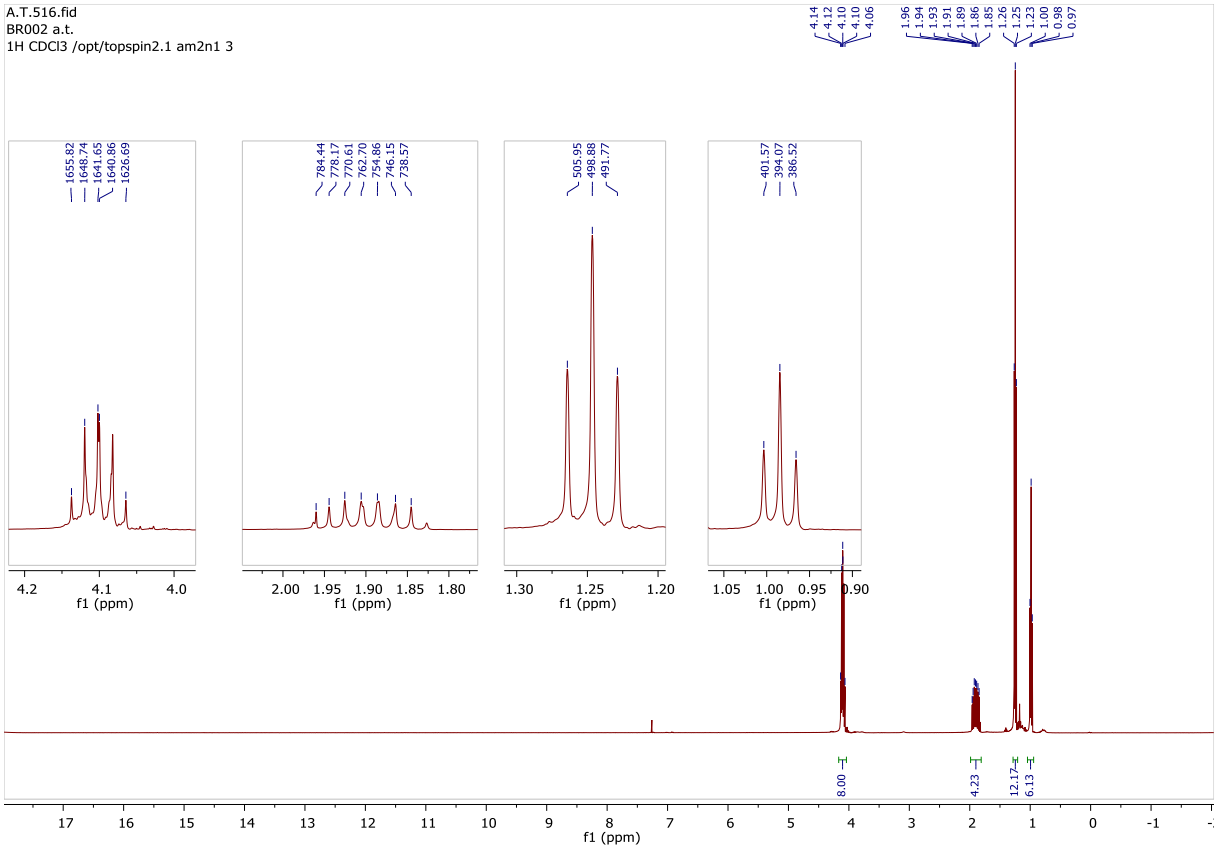
tetraethyl pentane-3,3-diylbis(phosphonate)

$C_{13}H_{30}O_6P_2$

344,32 g/mol

Colorless oil

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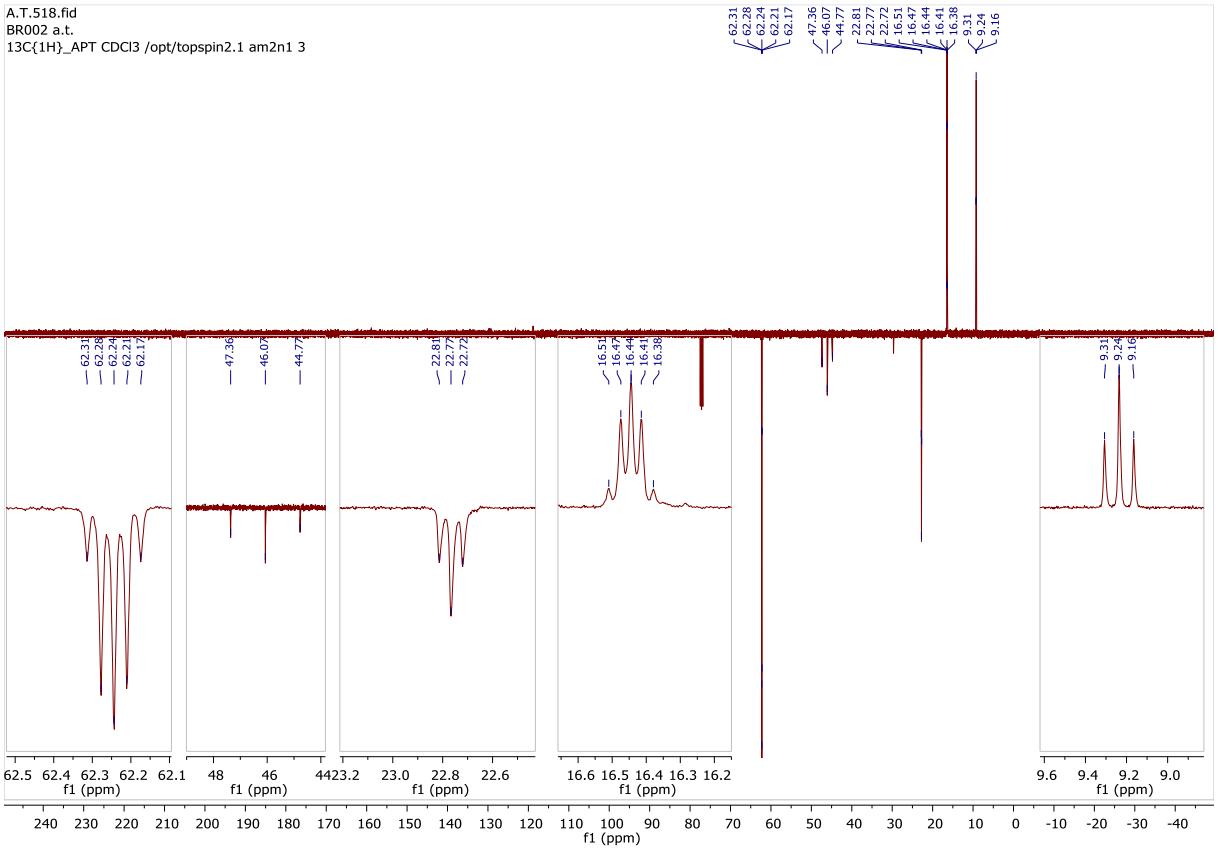
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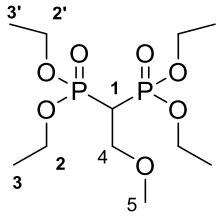
S4: ^{31}P, and ^{1}H, ^{13}C NMR spectra and mass analysis of tetraethyl (2-methoxyethane-1,1-diyl)bis(phosphonate) (4)

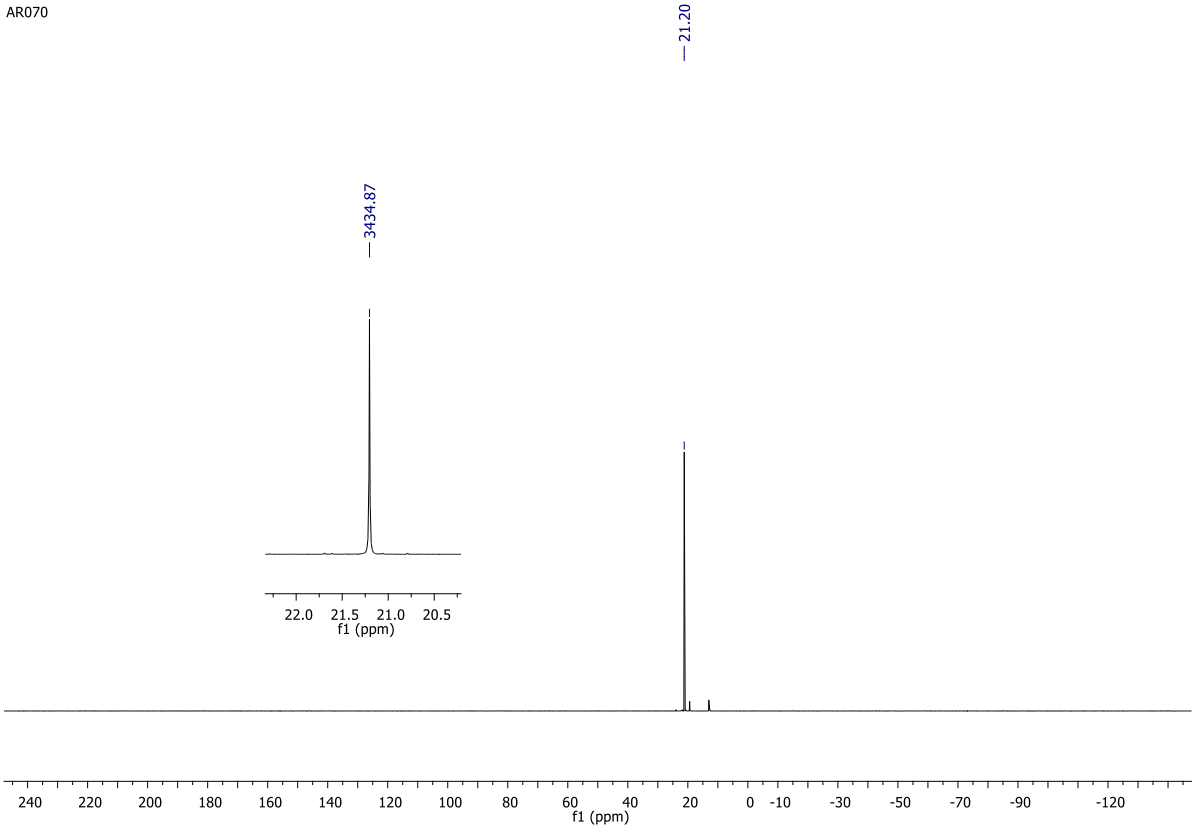


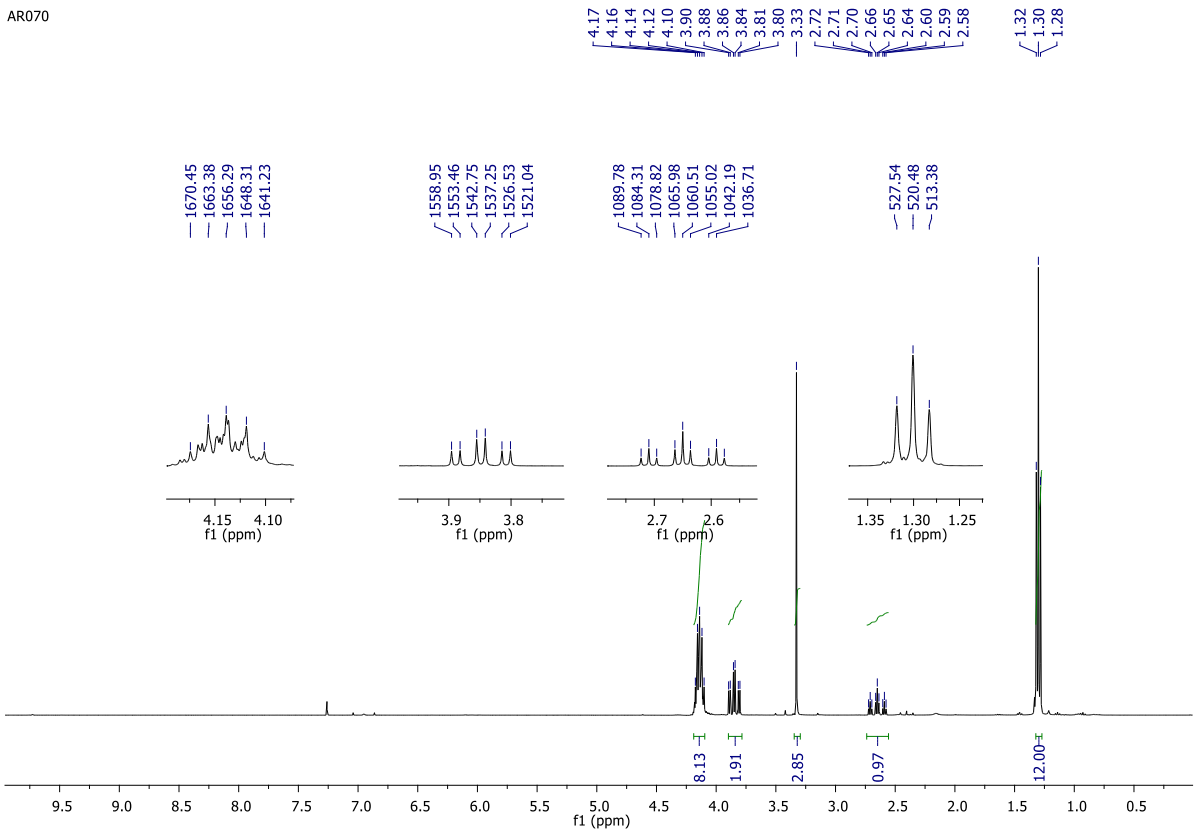
tetraethyl (2-methoxyethane-1,1-diyl)bis(phosphonate)

$C_{11}H_{26}O_7P_2$

332,27 g/mol

Colorless oil

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3.84
3.81
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2.72
2.71
2.70
2.66
2.65
2.64
2.60
2.59
2.58
1.32
1.30
1.28
1670.45
1663.38
1656.29
1648.31
1641.23
1558.95
1553.46
1542.75
1537.25
1526.53
1521.04
1089.78
1084.31
1078.82
1065.98
1060.51
1055.02
1042.19
1036.71
527.54
520.48
513.38
4.15 4.10
f1 (ppm)
3.9 3.8
f1 (ppm)
2.7 2.6
f1 (ppm)
1.35 1.30 1.25
f1 (ppm)
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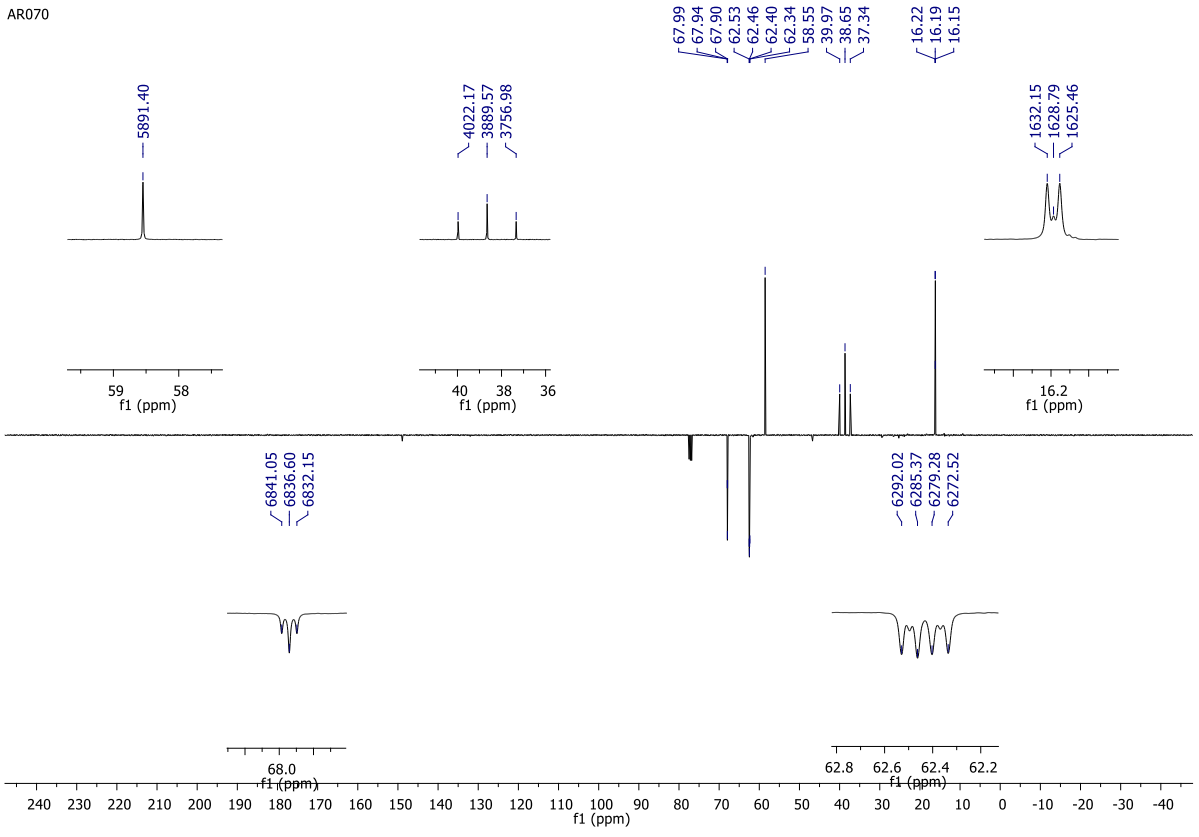
1.91
2.85
0.97
12.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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16.15
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1632.15
1628.79
1625.46
59 58
f1 (ppm)
40 38 36
f1 (ppm)
16.2
f1 (ppm)
6841.05
6836.60
6832.15
6292.02
6285.37
6279.28
6272.52
68.0
f1 (ppm)
62.8 62.6 62.4 62.2
f1 (ppm)
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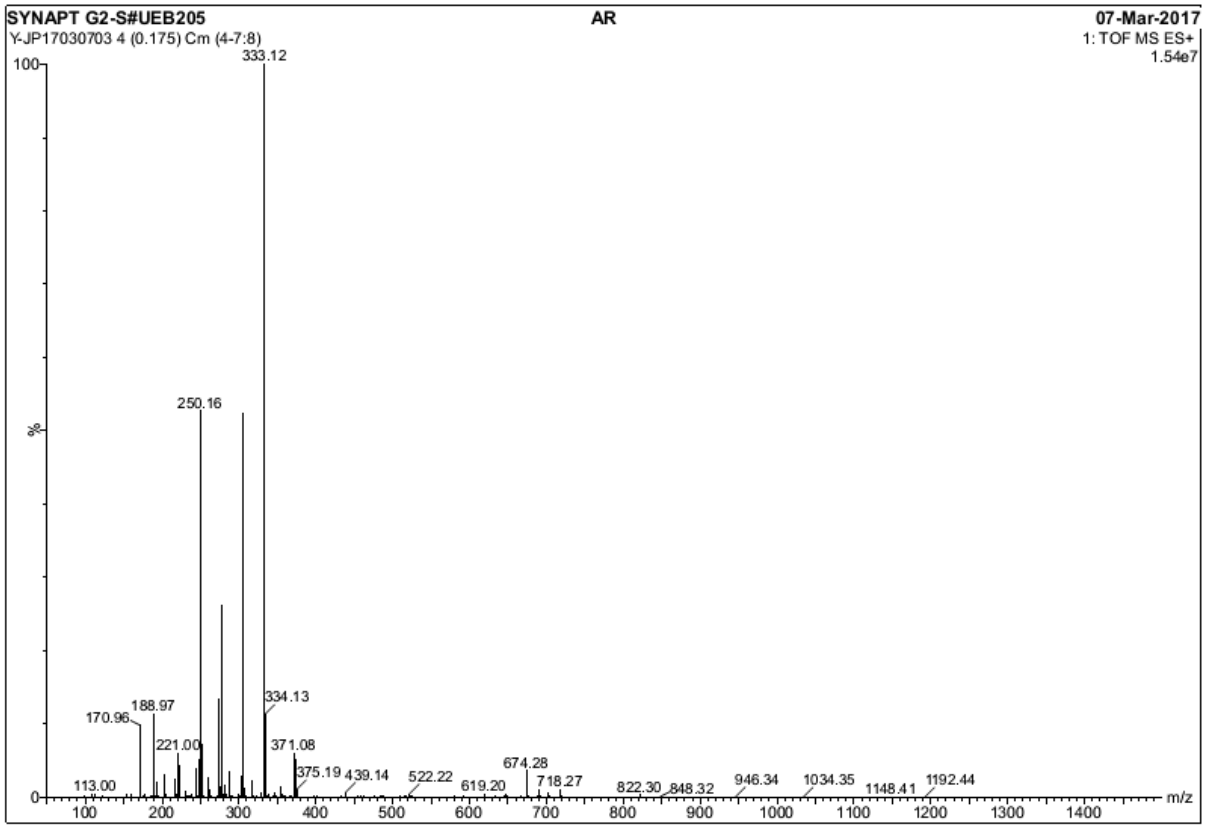
f1 (ppm)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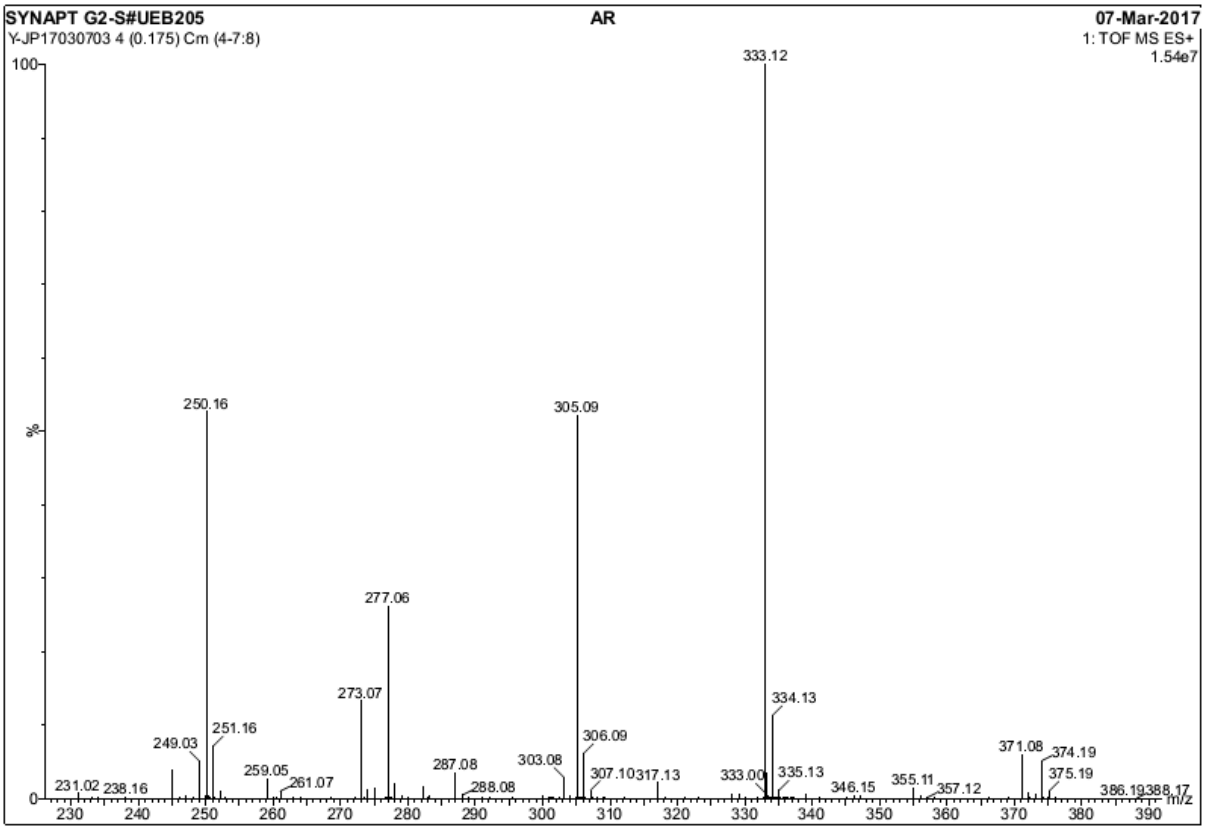
170.96
221.00
371.08
375.19
439.14
522.22
619.20
674.28
718.27
822.30
848.32
946.34
1034.35
1148.41
1192.44
113.00
100 200 300 400 500 600 700 800 900 1000 1100 1200 1300 1400
m/z


SYNAPT G2-S#UEB205
AR
07-Mar-2017
Y-JP17030703 4 (0.175) Cm (4-7:8)
1: TOF MS ES+
1.54e7
100
%
0
333.12
250.16
305.09
277.06
273.07
334.13
249.03
251.16
306.09
371.08
374.19
375.19
259.05
261.07
287.08
288.08
303.08
307.10
317.13
333.00
335.13
346.15
355.11
357.12
231.02
238.16
386.19
388.17
230 240 250 260 270 280 290 300 310 320 330 340 350 360 370 380 390
m/z

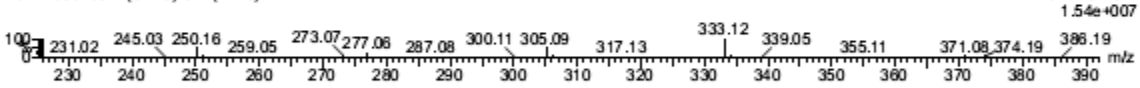
Elemental Composition Report

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 AR 07-Mar-2017
Y-JP17030703 4 (0.175) Cm (4-7:8) 1: TOF MS ES+
1.54e+007



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Maximum:		1.0	1.0	50.0				
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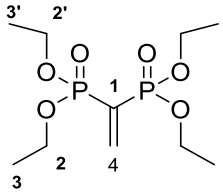
	333.1229	0.3	0.9	9.5	1441.2	4.257	1.42	C14 H18 N6 O2 P

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

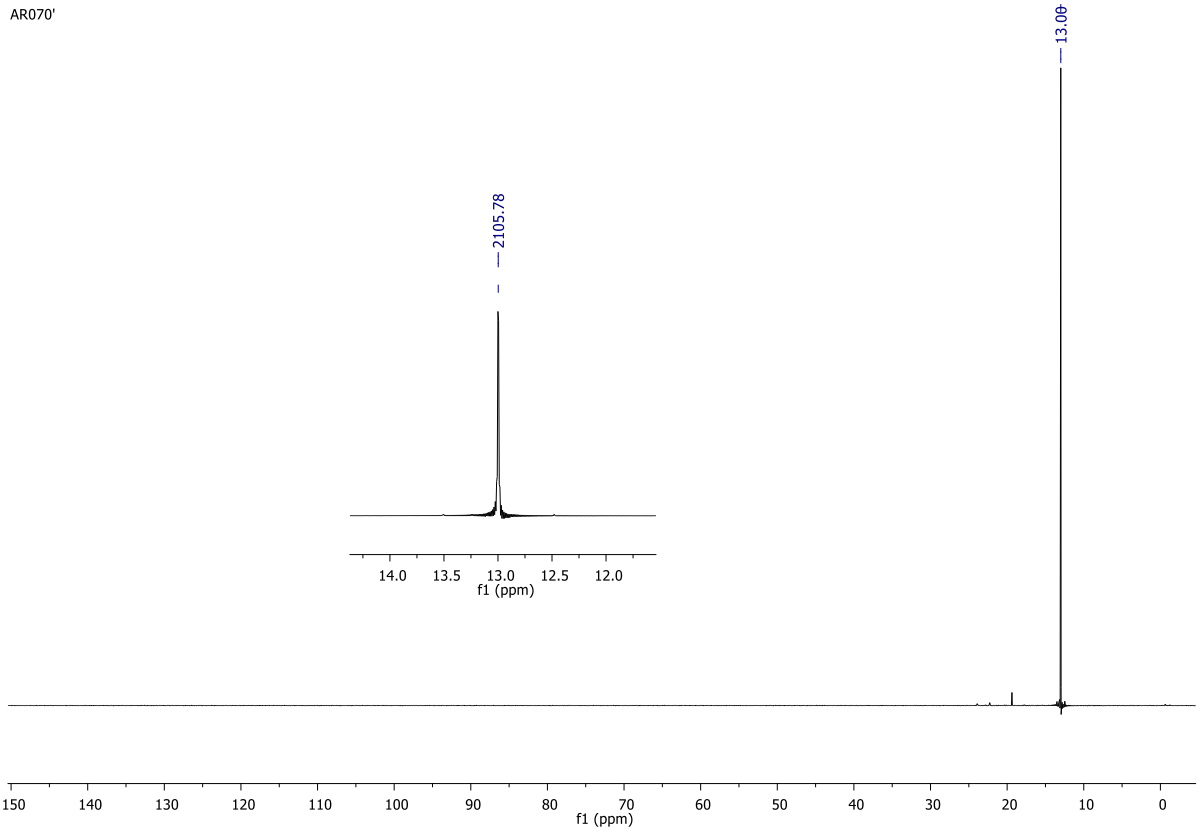


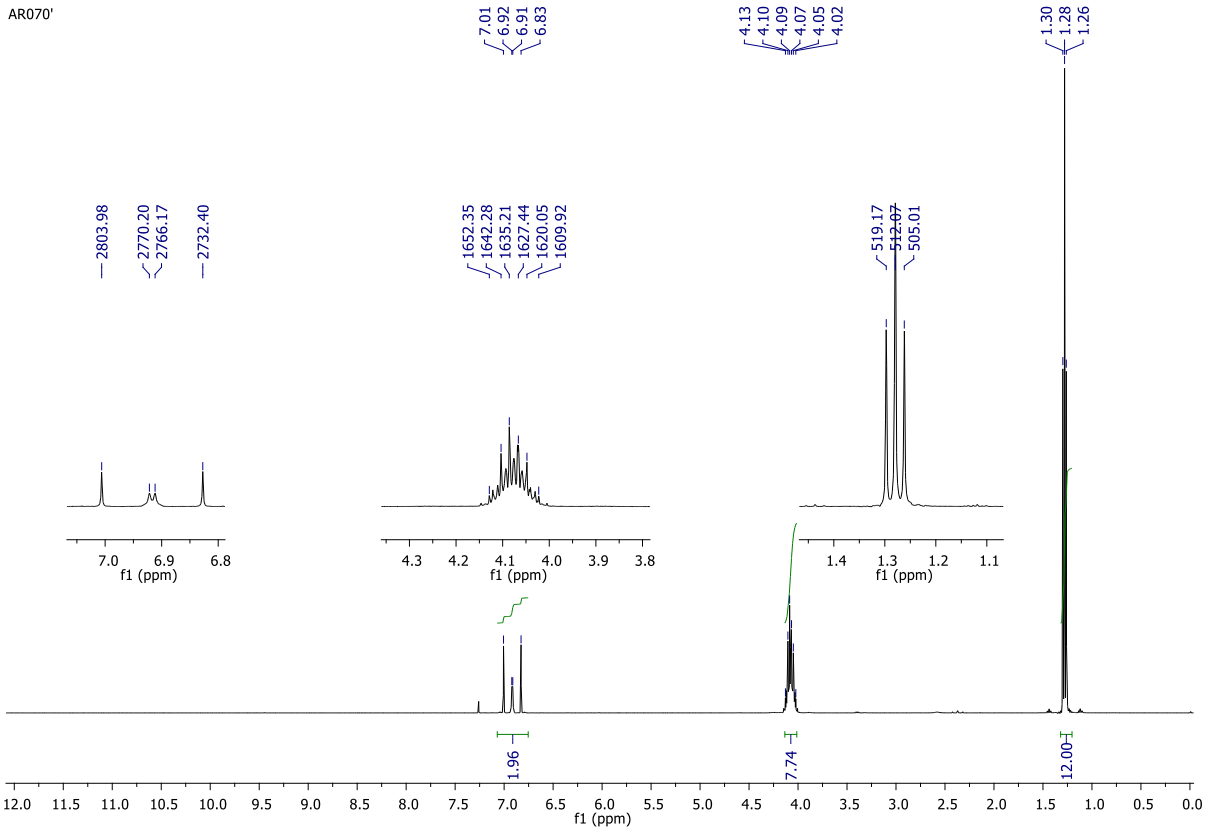
tetraethyl ethene-1,1-diylbis(phosphonate)

$C_{10}H_{22}O_6P_2$

300,23 g/mol

Colorless oil

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4.07
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1.28
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2770.20
2766.17
2732.40
1652.35
1642.28
1635.21
1627.44
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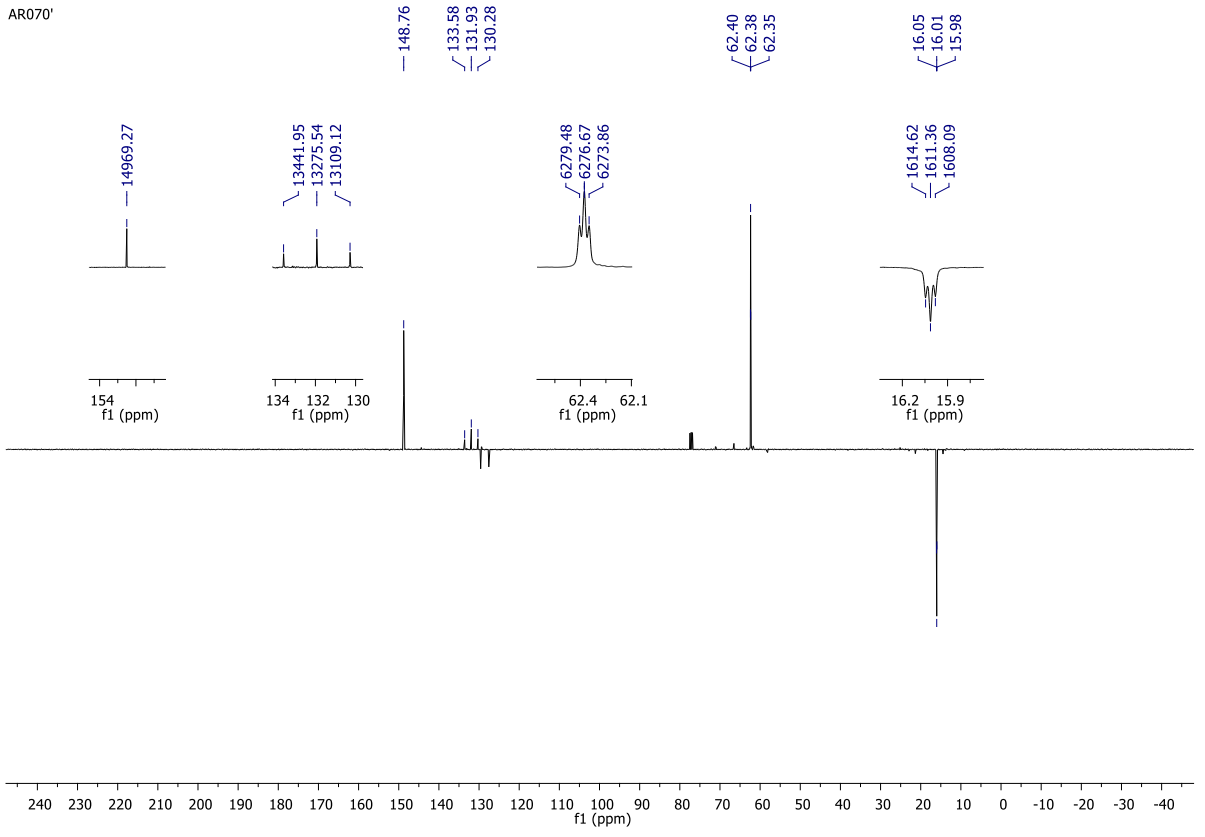
1.96
7.74
12.00
f1 (ppm)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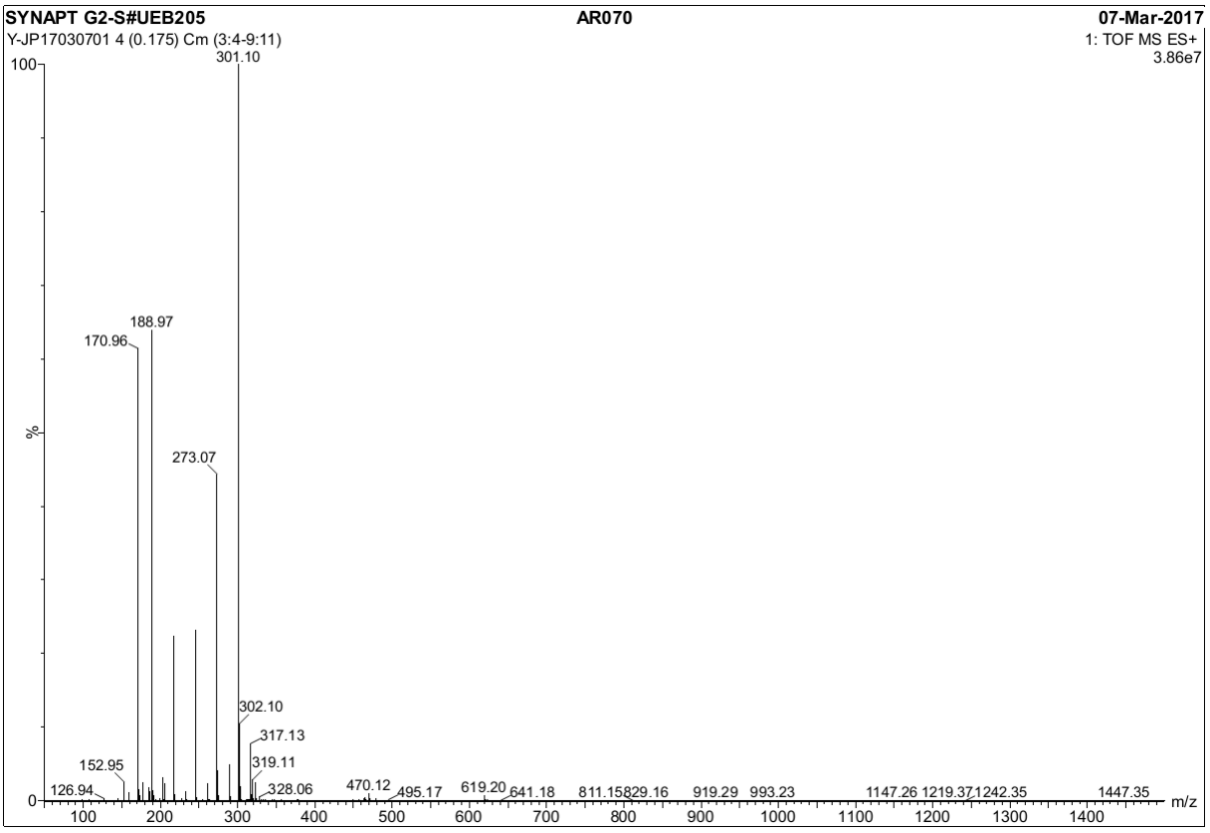
1608.09
f1 (ppm)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07-Mar-2017
Y-JP17030701 4 (0.175) Cm (3:4-9:11)
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188.97
170.96
273.07
302.10
317.13
319.11
152.95
126.94
328.06
470.12
495.17
619.20
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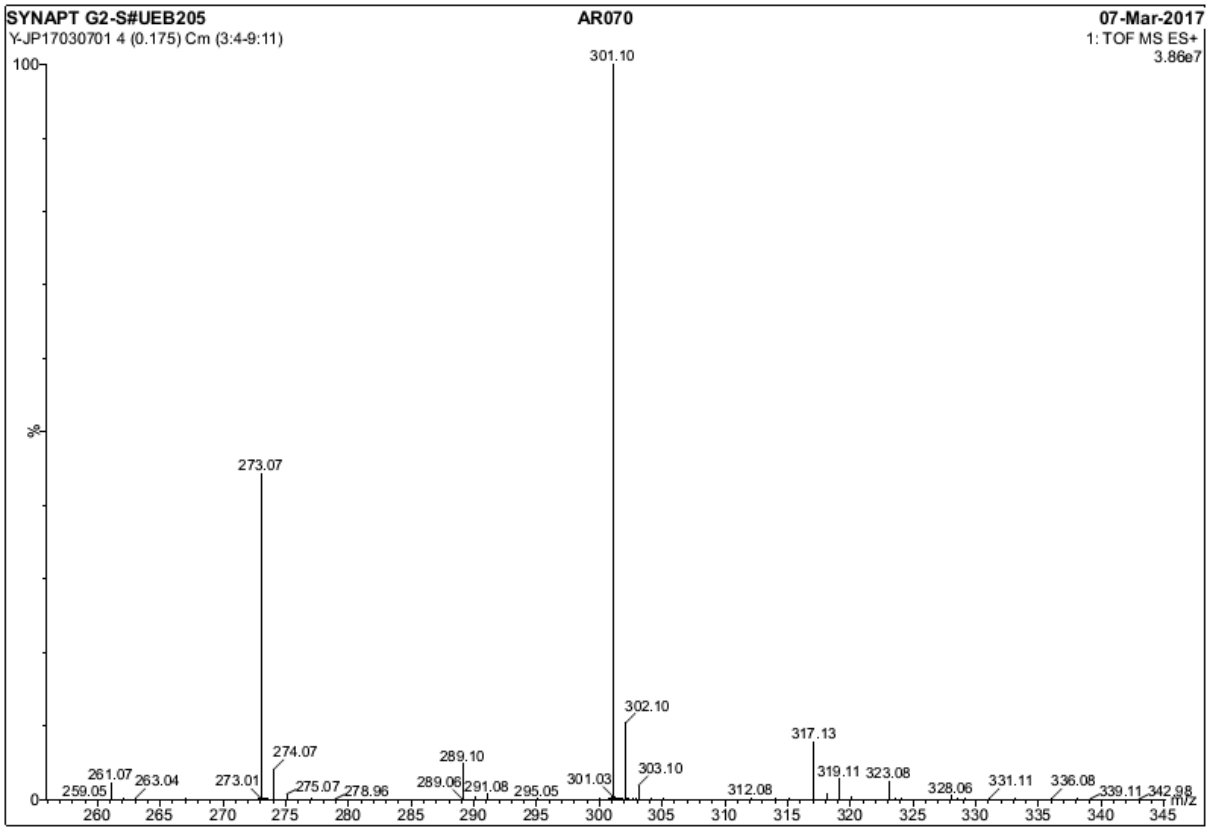
993.23
1147.26
1219.37
1242.35
1447.35
%
m/z


SYNAPT G2-S#UEB205
AR070
07-Mar-2017
Y-JP17030701 4 (0.175) Cm (3:4-9:11)
1: TOF MS ES+
3.86e7
301.10
273.07
302.10
317.13
274.07
289.10
261.07
263.04
259.05
273.01
275.07
278.96
289.06
291.08
295.05
301.03
303.10
312.08
319.11
323.08
328.06
331.11
336.08
339.11
342.98
%
m/z

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-S#UEB205 AR070 07-Mar-2017
Y-JP17030701 4 (0.175) Cm (3:4-9:11) 1: TOF MS ES+
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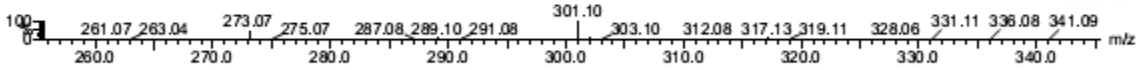

Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
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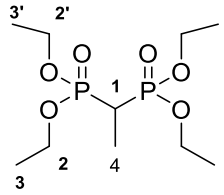
	301.0967	0.2	0.7	10.5	2218.0	10.826	0.00	C13 H14 N6 O P

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

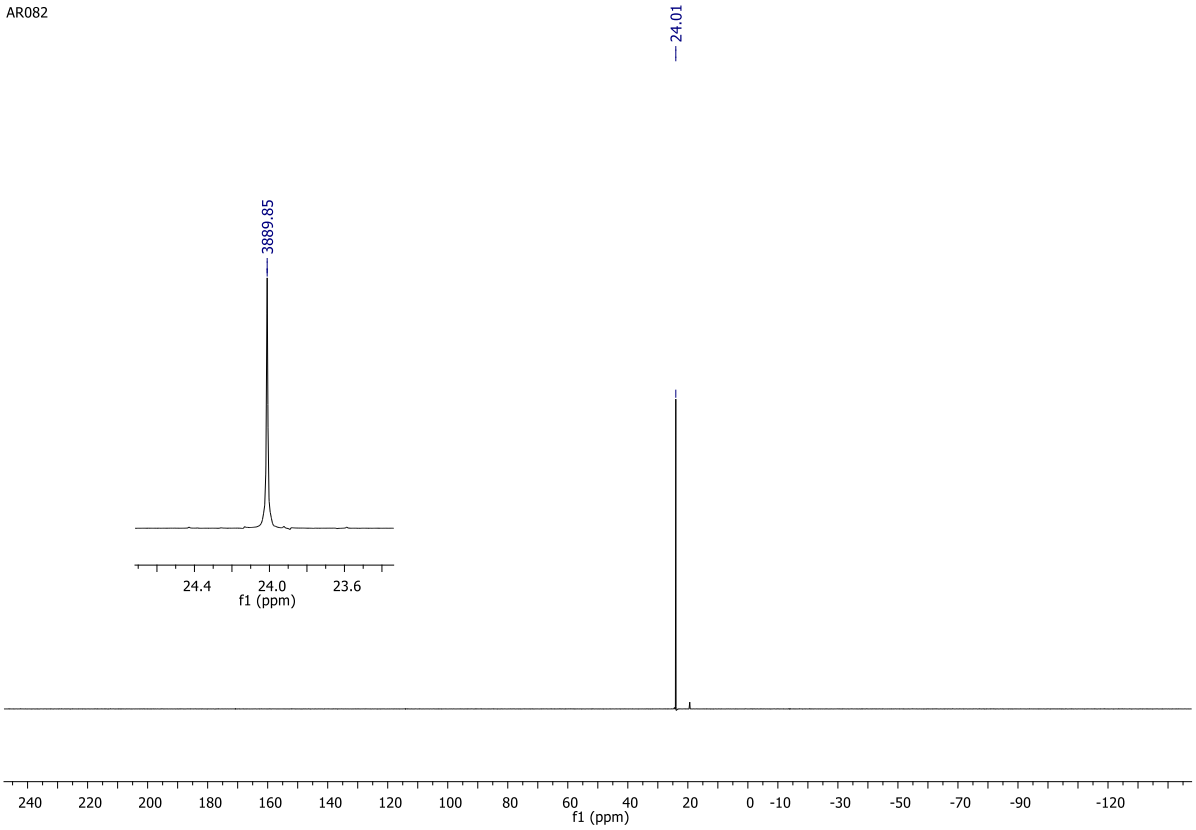


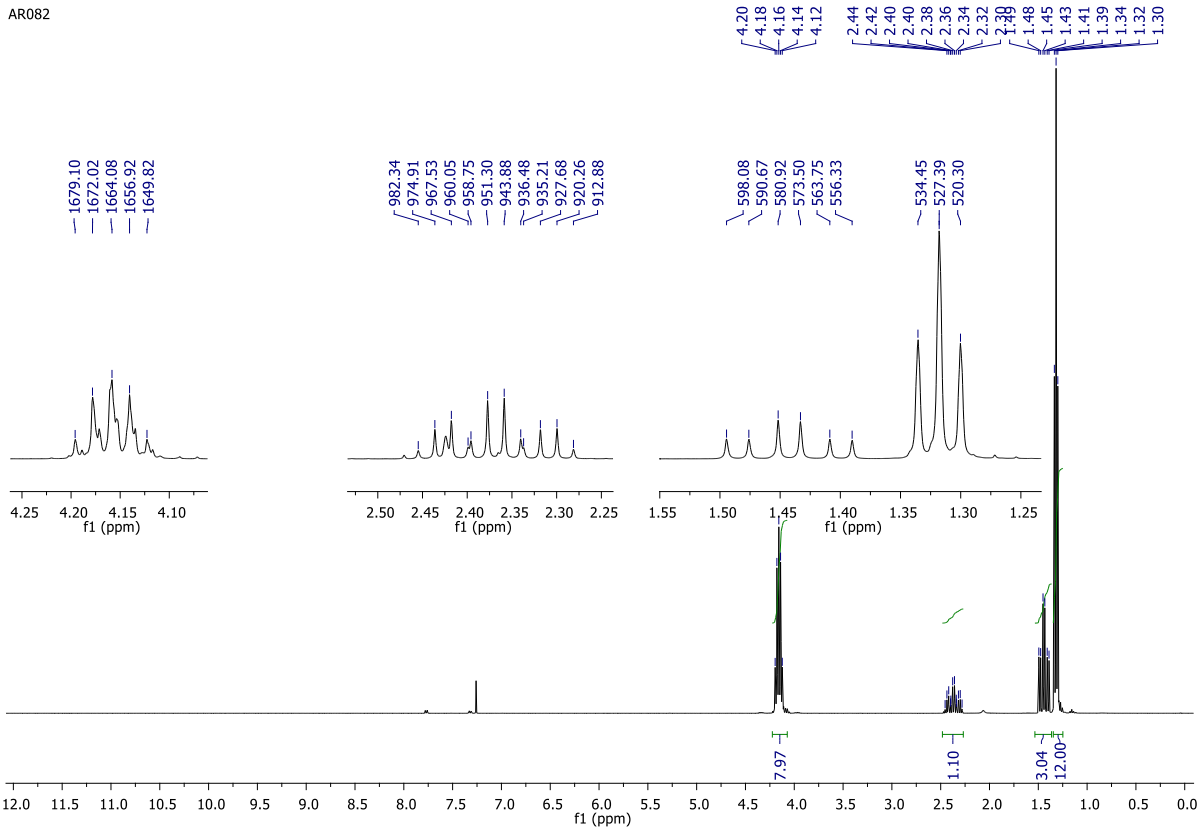
tetraethyl ethane-1,1-diylbis(phosphonate)

$C_{10}H_{24}O_6P_2$

302,24 g/mol

Colorless oil

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1.41
1.39
1.34
1.32
1.30
1679.10
1672.02
1664.08
1656.92
1649.82
982.34
974.91
967.53
960.05
958.75
951.30
943.88
936.48
935.21
927.68
920.26
912.88
598.08
590.67
580.92
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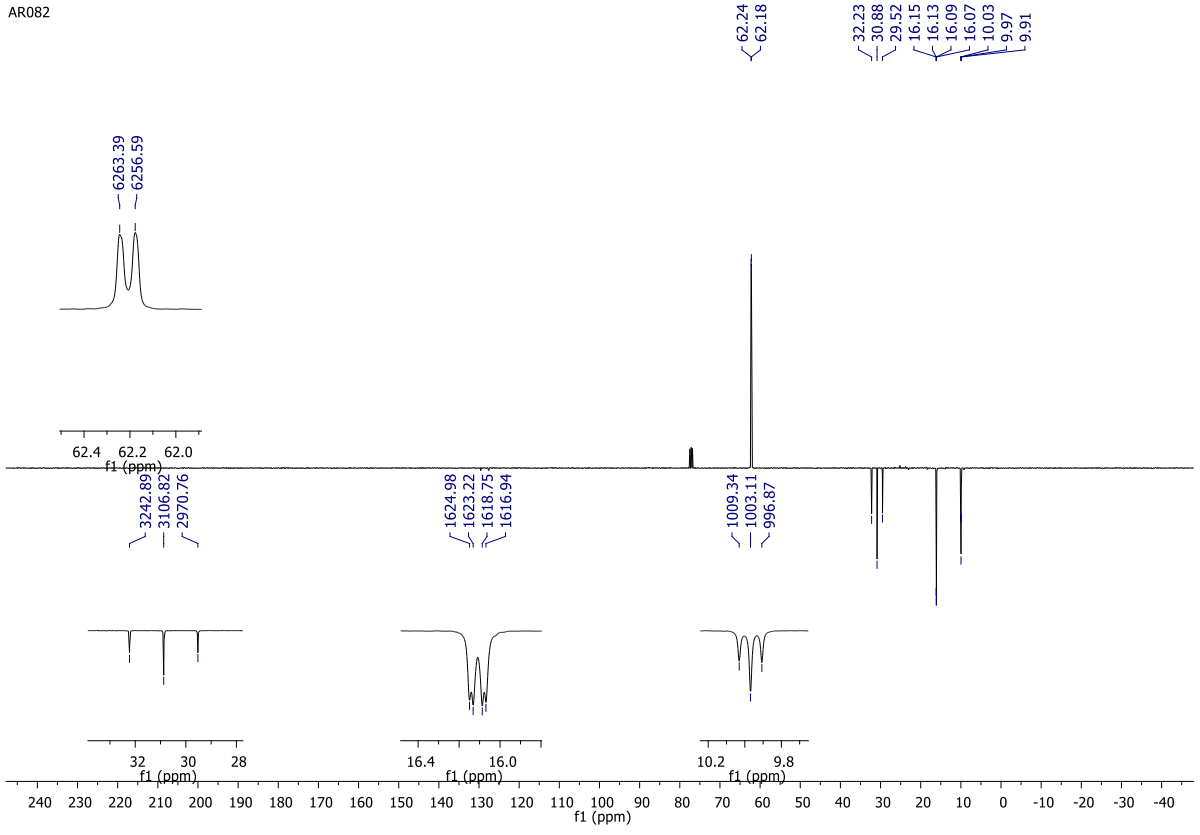
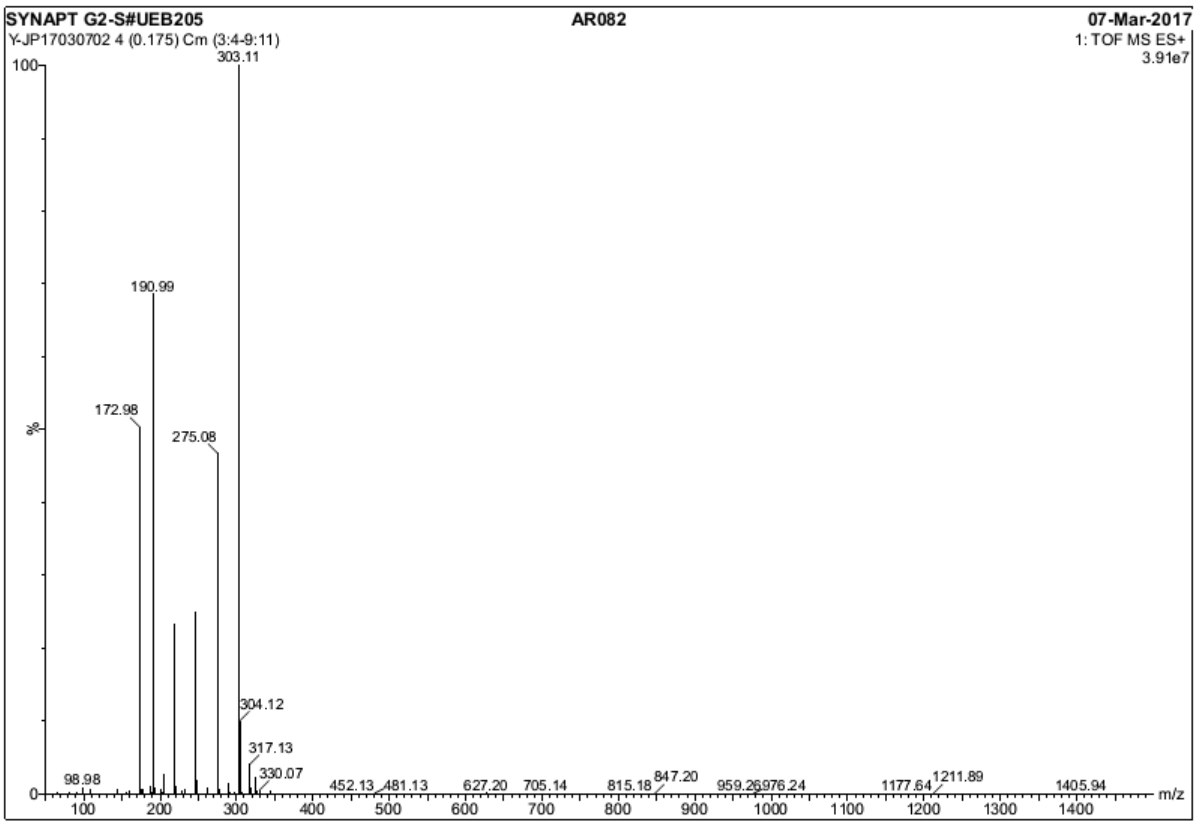
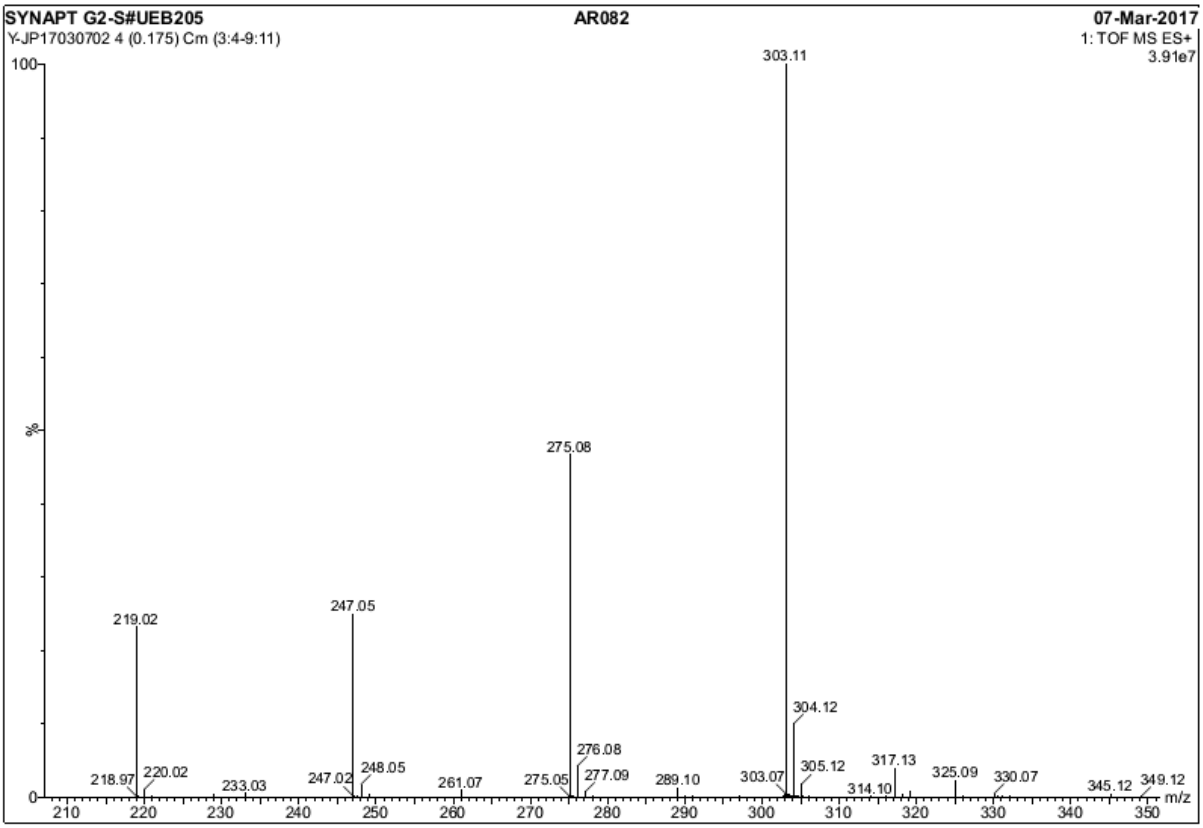
1.10
3.04
12.00
f1 (ppm)


AR082
62.24
62.18
32.23
30.88
29.52
16.15
16.13
16.09
16.07
10.03
9.97
9.91
6263.39
6256.59
3242.89
3106.82
2970.76
1624.98
1623.22
1618.75
1616.94
1009.34
1003.11
996.87
f1 (ppm)


SYNAPT G2-S#UEB205
AR082
07-Mar-2017
Y-JP17030702 4 (0.175) Cm (3:4-9:11)
1: TOF MS ES+
3.91e7
303.11
190.99
172.98
275.08
304.12
317.13
330.07
98.98
452.13
481.13
627.20
705.14
815.18
847.20
959.26
976.24
1177.64
1211.89
1405.94
%
m/z


SYNAPT G2-S#UEB205
AR082
07-Mar-2017
Y-JP17030702 4 (0.175) Cm (3:4-9:11)
1: TOF MS ES+
3.91e7
303.11
275.08
247.05
219.02
304.12
276.08
317.13
325.09
330.07
218.97
220.02
233.03
247.02
248.05
261.07
275.05
277.09
289.10
303.07
305.12
314.10
345.12
349.12
%
m/z

Elemental Composition Report

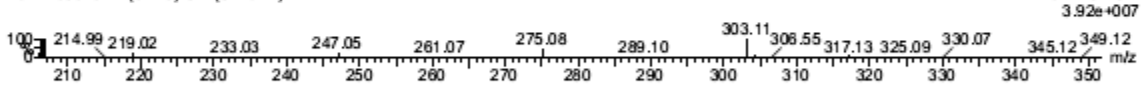
Page 1

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

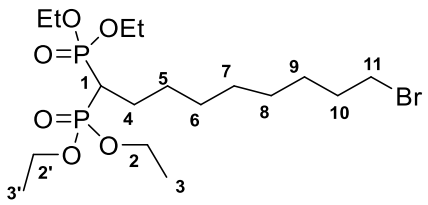
Monoisotopic Mass, Even Electron Ions
839 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 1-2

SYNAPT G2-S#UEB205 AR082 07-Mar-2017
Y-JP17030702 4 (0.175) Cm (3:4-9:11) 1: TOF MS ES+
3.92e+007



Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
Minimum:				-1.5				
Maximum:		1.0	1.0	50.0				
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, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl (9-bromononane-1,1-diyl)bis(phosphonate) (2c)

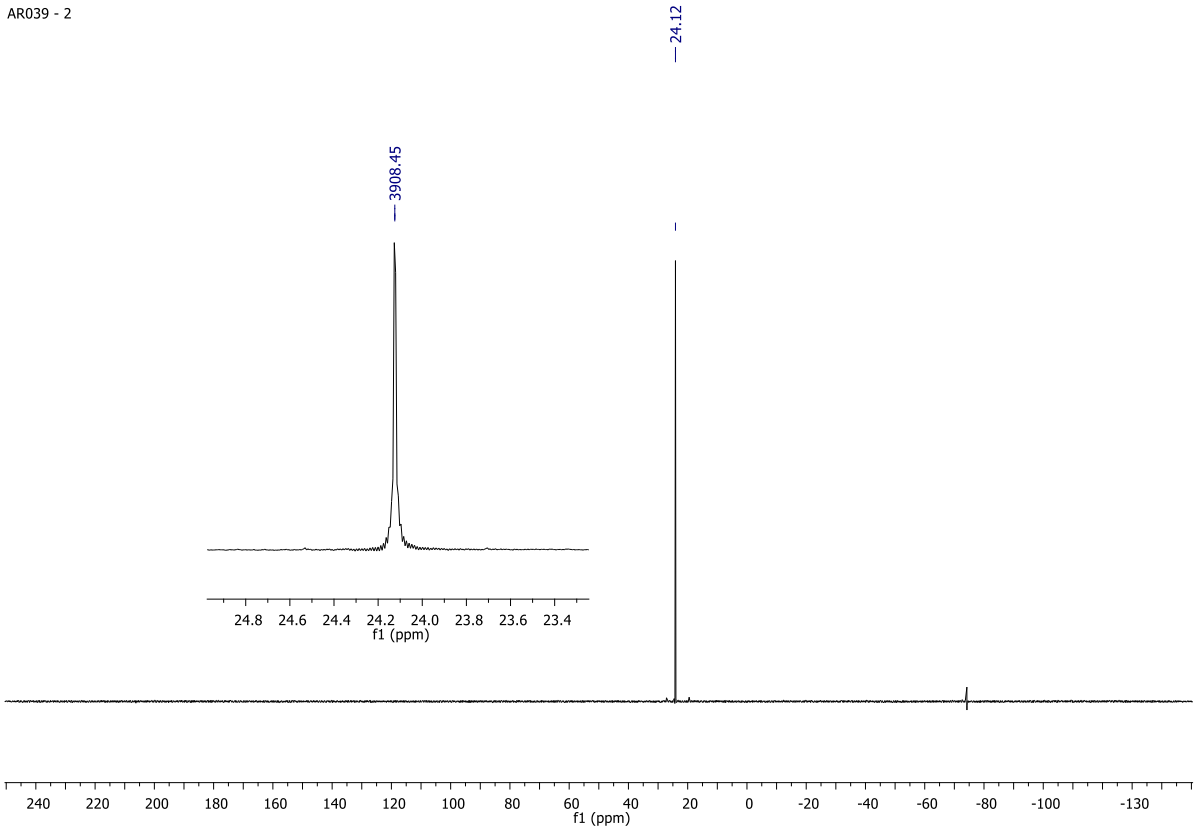


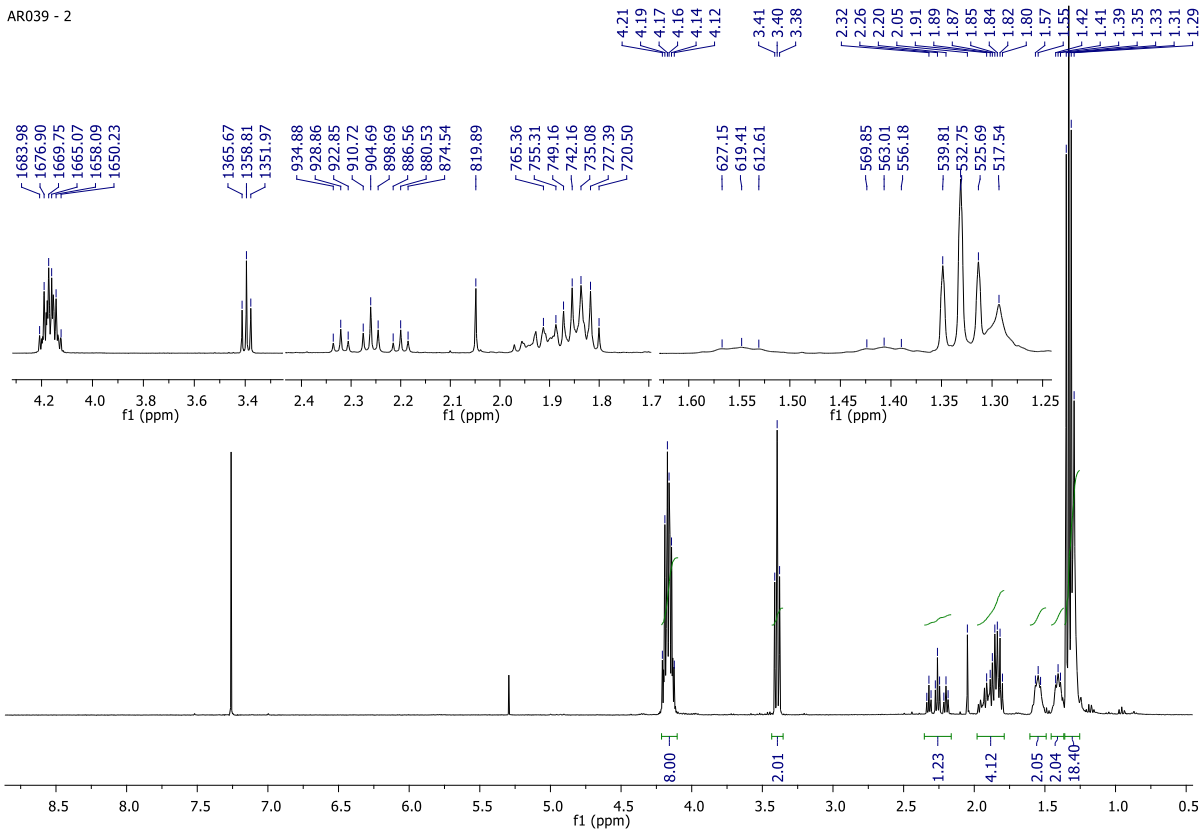
tetraethyl(9-bromononane-1,1-diyl)bis(phosphonate)

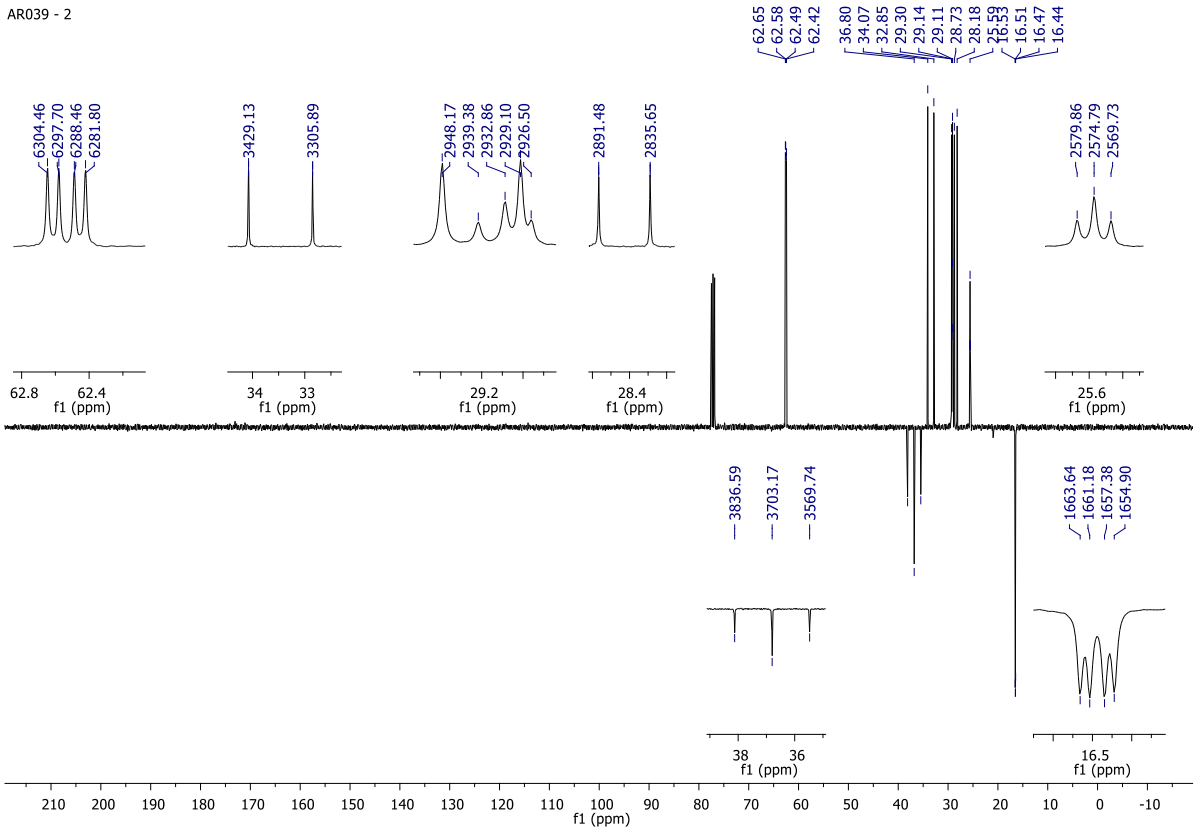
$C_{17}H_{37}O_6BrP_2$

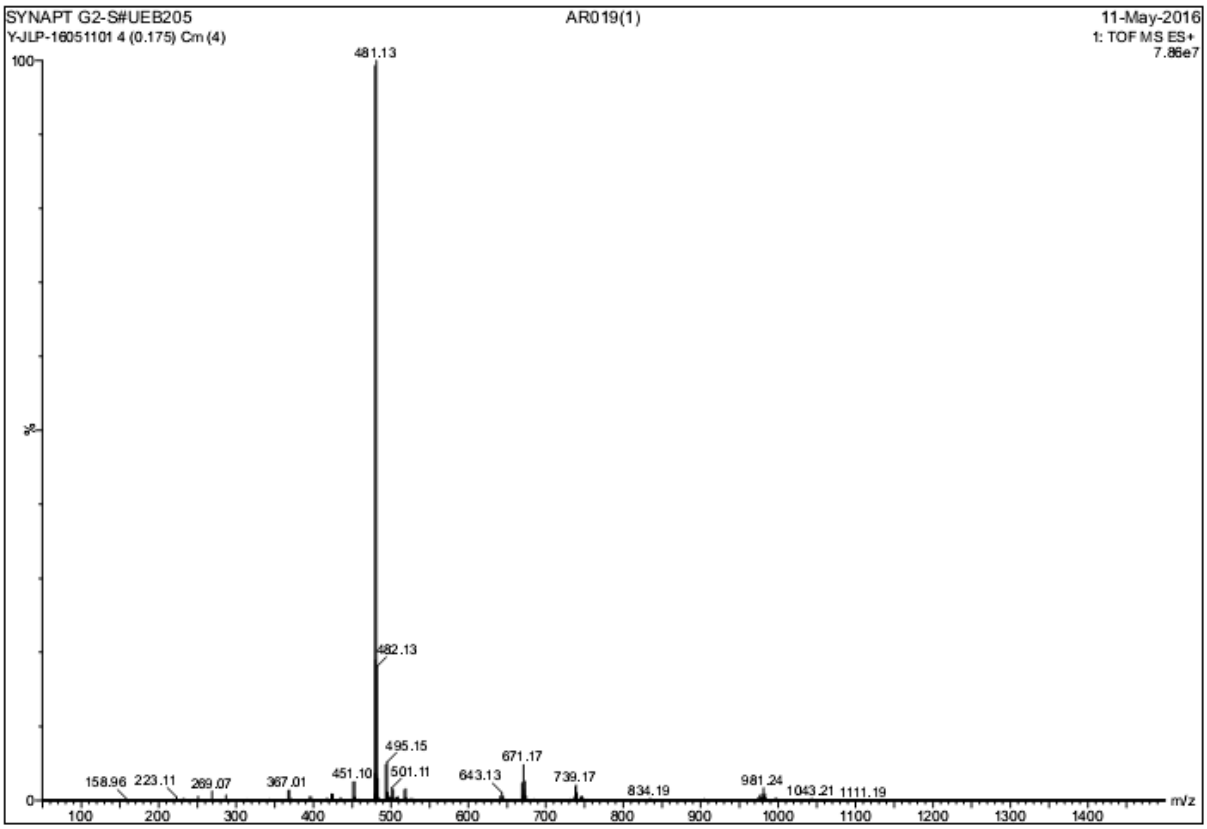
479,33 g/mol

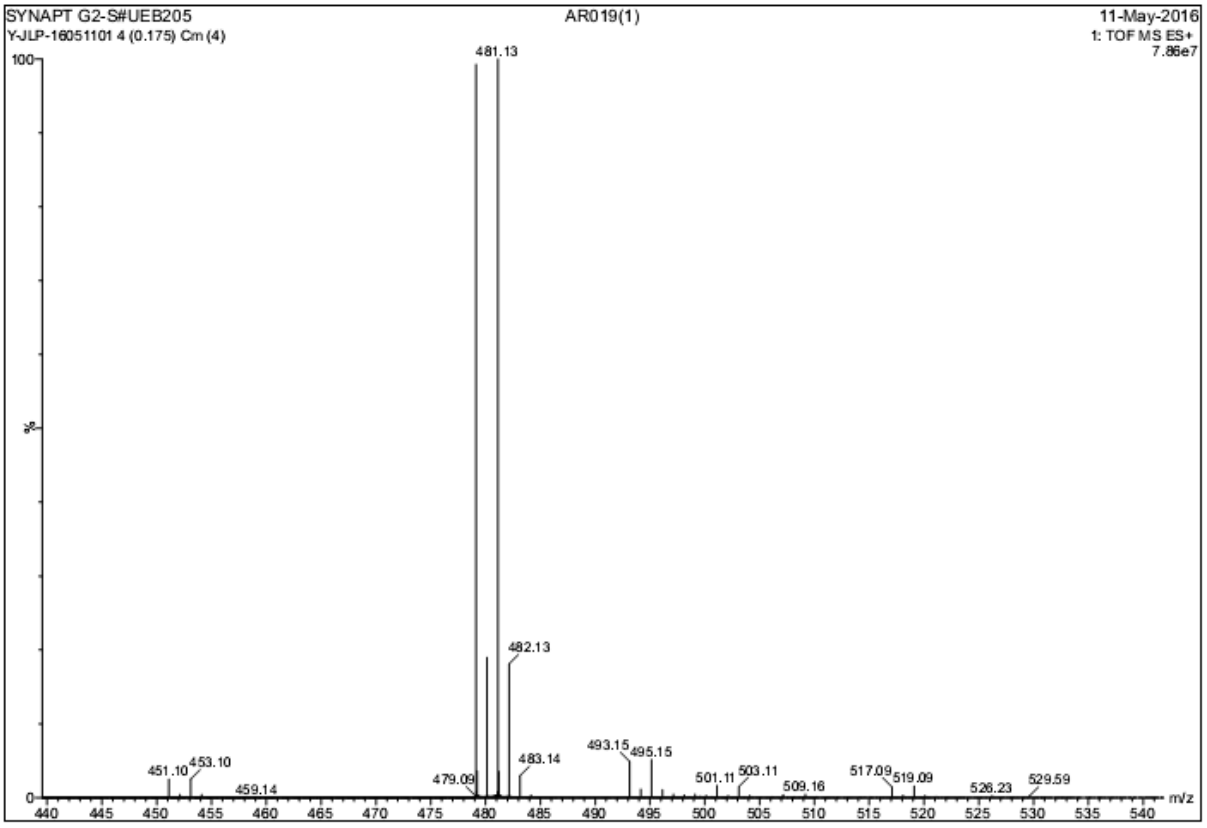
Colorless oil




AR039 - 2
4.21 4.19 4.17 4.16 4.14 4.12 3.41 3.40 3.38 2.32 2.26 2.20 2.05 1.91 1.89 1.87 1.85 1.84 1.82 1.80 1.57 1.55 1.42 1.41 1.39 1.35 1.33 1.31 1.29
1683.98 1676.90 1669.75 1665.07 1658.09 1650.23 1365.67 1358.81 1351.97 934.88 928.86 922.85 910.72 904.69 898.69 886.56 880.53 874.54 819.89 765.36 755.31 749.16 742.16 735.08 727.39 720.50 627.15 619.41 612.61 569.85 563.01 556.18 539.81 532.75 525.69 517.54
f1 (ppm)
8.00 2.01 1.23 4.12 2.05 2.04 18.40


AR039 - 2
62.65 62.58 62.49 62.42 36.80 34.07 32.85 29.30 29.14 29.11 28.73 28.18 25.59 16.51 16.47 16.44
6304.46 6297.70 6288.46 6281.80 3429.13 3305.89 2948.17 2939.38 2932.86 2929.10 2926.50 2891.48 2835.65 2579.86 2574.79 2569.73
f1 (ppm)
3836.59 3703.17 3569.74 1663.64 1661.18 1657.38 1654.90


SYNAPT G2-S#UEB205
AR019(1)
11-May-2016
Y-JLP-16051101 4 (0.175) Cm (4)
1: TOF MS ES+
7.86e7
481.13
482.13
495.15
671.17
158.96
223.11
269.07
367.01
451.10
501.11
643.13
739.17
834.19
981.24
1043.21
1111.19
m/z

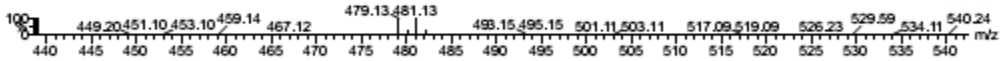

SYNAPT G2-S#UEB205
AR019(1)
11-May-2016
Y-JLP-16051101 4 (0.175) Cm (4)
1: TOF MS ES+
7.86e7
481.13
482.13
451.10
453.10
459.14
479.09
483.14
493.15
495.15
501.11
503.11
509.16
517.09
519.09
526.23
529.59
m/z

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

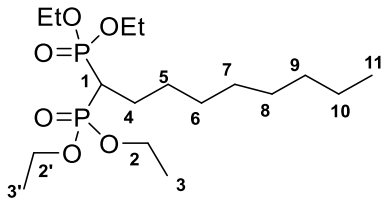
Monoisotopic Mass, Even Electron Ions
2560 formula(e) evaluated with 2 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 0-10 O: 0-10 P: 1-2 Br: 1-1

SYNAPT G2-S#UEB205 AR019(1) 11-May-2016
Y-JLP-16051101 4 (0.175) Cm (4) 1: TOF MS ES+
7.86e+007

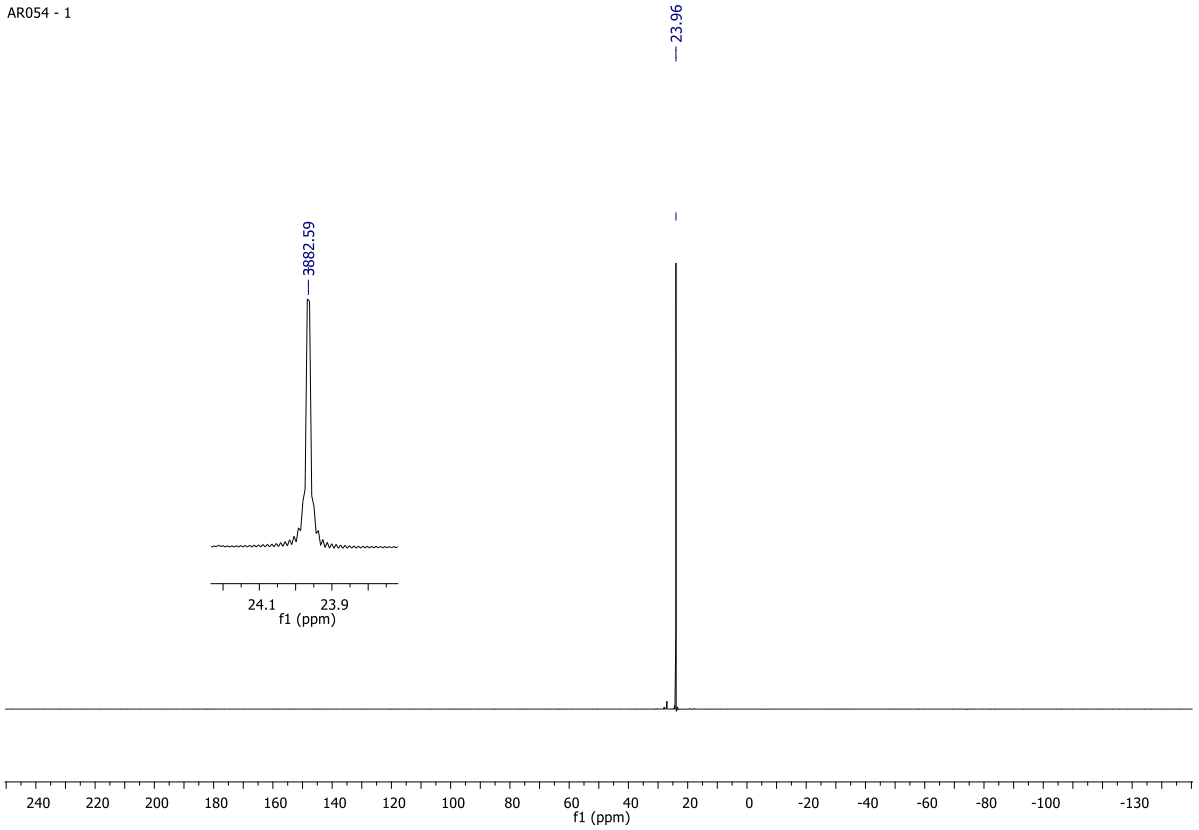


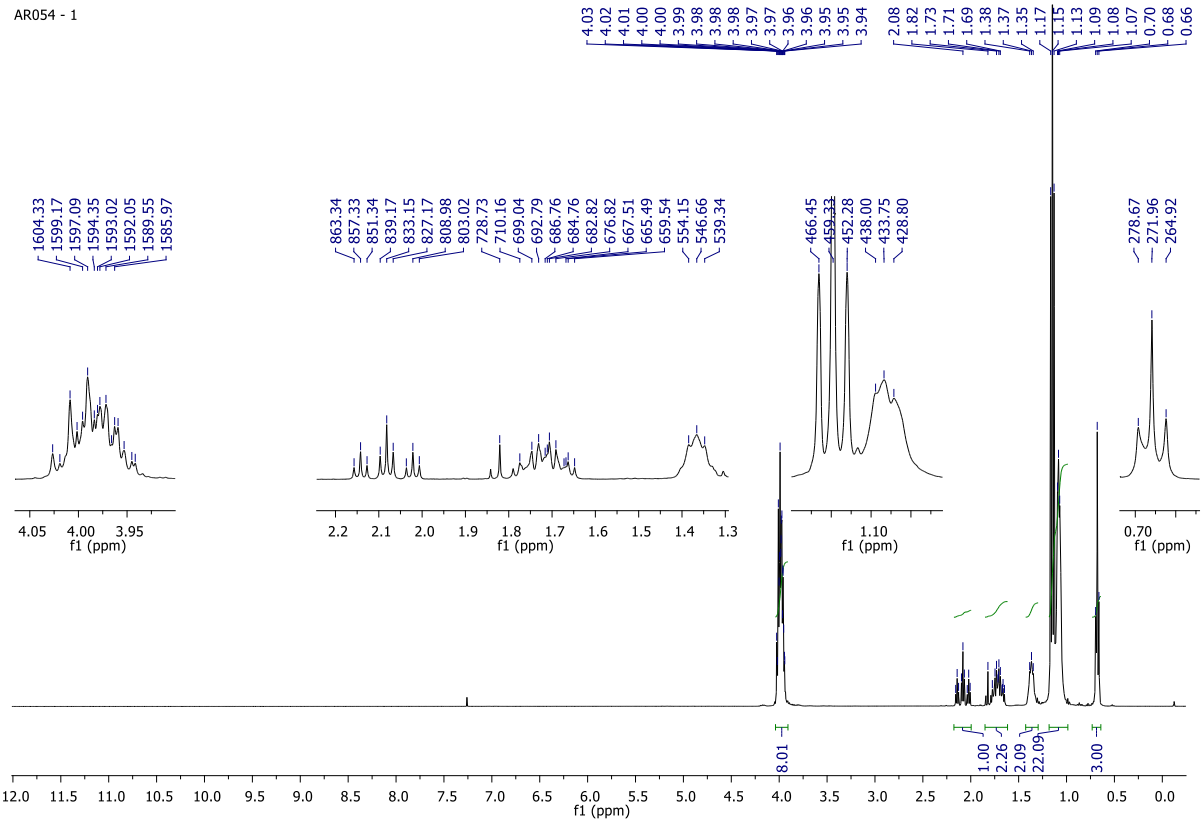
Minimum:				-50.0				
Maximum:		1.1	2.0	100.0				
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
479.1328	479.1327	0.1	0.2	-0.5	1653.9	0.000	100.00	C17 H38 O6 P2 Br
	479.1324	0.4	0.8	9.5	1669.5	15.690	0.00	C20 H29 N6 O P Br

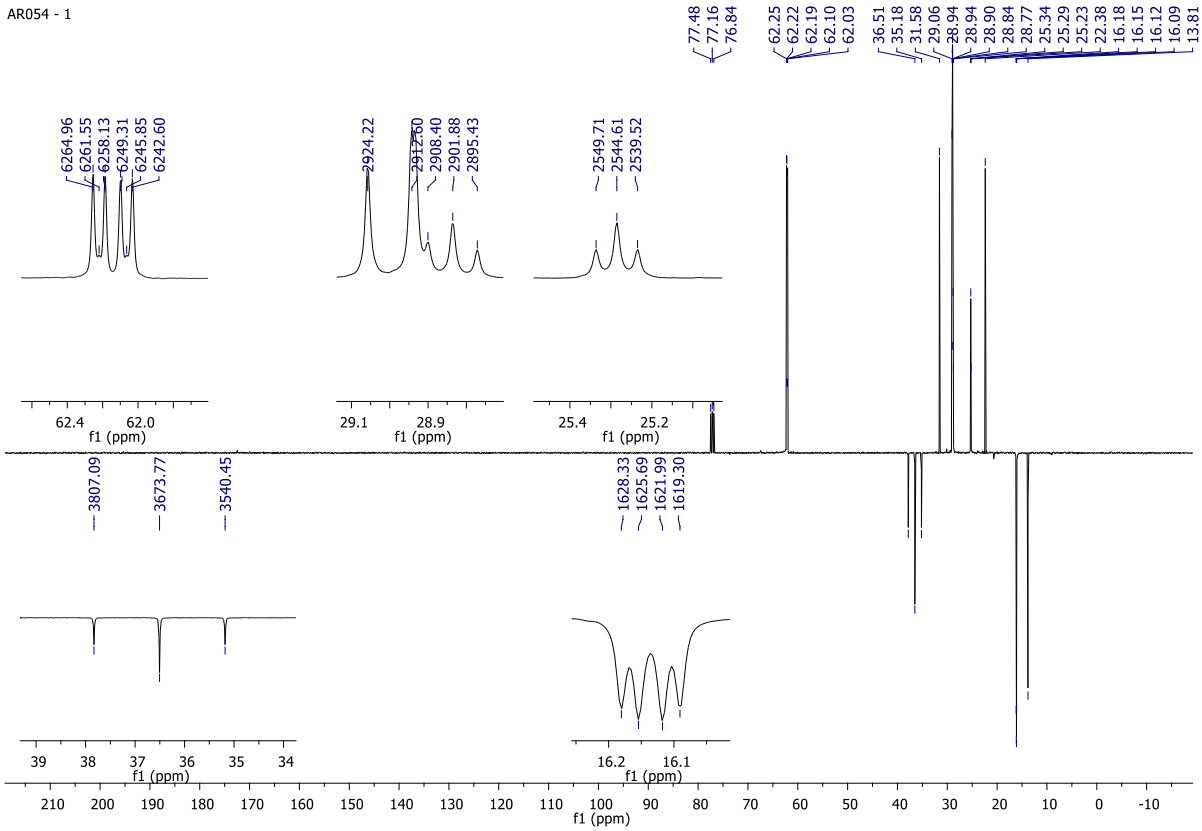
S8: ^{31}P, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl nonane-1,1-diylbis(phosphonate) (2d)

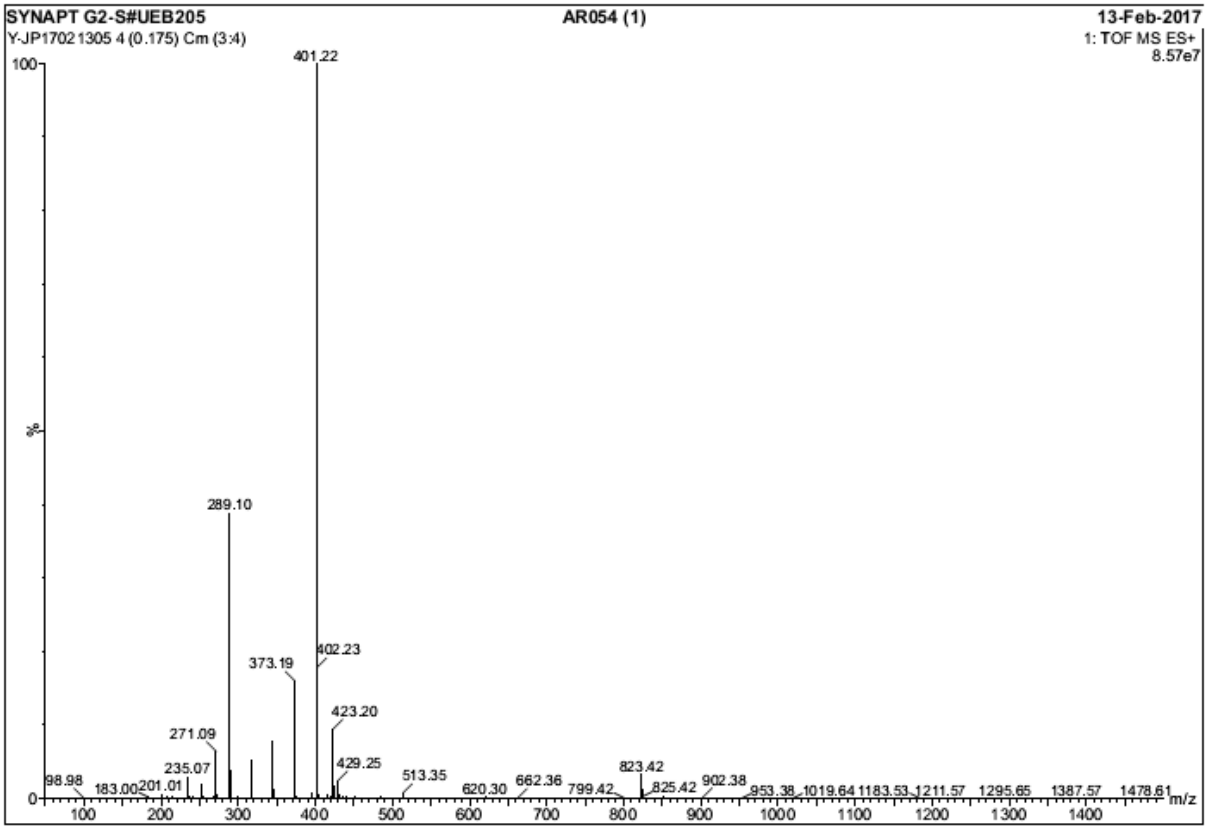


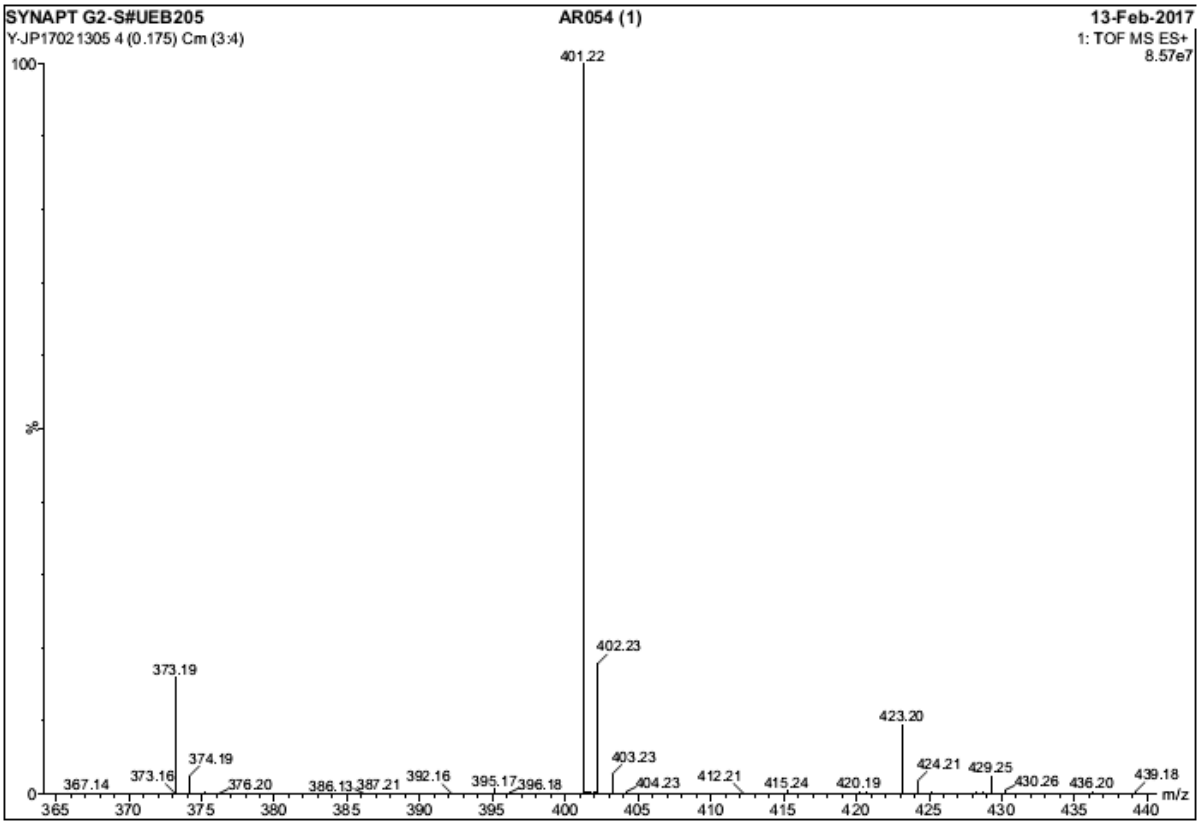
tetraethyl nonane-1,1-diylbis(phosphonate)
$C_{17}H_{38}O_6P_2$
400,43 g/mol
Colorless oil




AR054 - 1
4.03 4.02 4.01 4.00 4.00 3.99 3.98 3.98 3.98 3.97 3.97 3.96 3.96 3.95 3.95 3.94
2.08 1.82 1.73 1.71 1.69 1.38 1.37 1.35 1.17 1.15 1.13 1.09 1.08 1.07 0.70 0.68 0.66
1604.33 1599.17 1597.09 1594.35 1593.02 1592.05 1589.55 1585.97
863.34 857.33 851.34 839.17 833.15 827.17 808.98 803.02 728.73 710.16 699.04 692.79 686.76 684.76 682.82 676.82 667.51 665.49 659.54 554.15 546.66 539.34
466.45 459.33 452.28 438.00 433.75 428.80
278.67 271.96 264.92
8.01 1.00 2.26 2.09 22.09 3.00
f1 (ppm)


AR054 - 1
77.48 77.16 76.84
62.25 62.22 62.19 62.10 62.03
36.51 35.18 31.58 29.06 28.94 28.94 28.90 28.84 28.77 25.34 25.29 25.23 22.38 16.18 16.15 16.12 16.09 13.81
6264.96 6261.55 6258.13 6249.31 6245.85 6242.60
2924.22 2912.60 2908.40 2901.88 2895.43
2549.71 2544.61 2539.52
3807.09 3673.77 3540.45
1628.33 1625.69 1621.99 1619.30
f1 (ppm)


SYNAPT G2-S#UEB205
AR054 (1)
13-Feb-2017
Y-JP1702 1305 4 (0.175) Cm (3:4)
1: TOF MS ES+
8.57e7
100
%
0
401.22
289.10
373.19
402.23
423.20
271.09
235.07
429.25
98.98
183.00
201.01
513.35
620.30
662.36
799.42
823.42
825.42
902.38
953.38
1019.64
1183.53
1211.57
1295.65
1387.57
1478.61
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
m/z

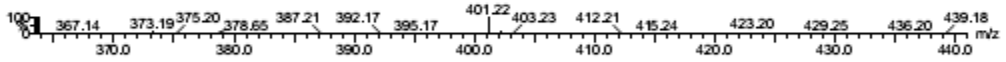

SYNAPT G2-S#UEB205
AR054 (1)
13-Feb-2017
Y-JP1702 1305 4 (0.175) Cm (3:4)
1: TOF MS ES+
8.57e7
100
%
0
401.22
402.23
373.19
423.20
374.19
403.23
373.16
424.21
429.25
367.14
376.20
386.13
387.21
392.16
395.17
396.18
404.23
412.21
415.24
420.19
430.26
436.20
439.18
365
370
375
380
385
390
395
400
405
410
415
420
425
430
435
440
m/z

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

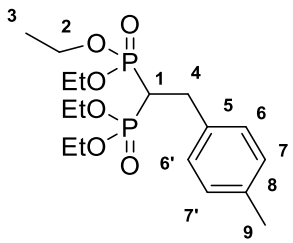
Monoisotopic Mass, Even Electron Ions
841 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 2-2

SYNAPT G2-S#UEB205 AR054 (1) 13-Feb-2017
Y-JP17021305 4 (0.175) Cm (3:4) 1: TOF MS ES+
8.57e+007



Minimum:				-1.5				
Maximum:		10.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	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, ^{1}H and ^{13}C NMR spectra and mass analysis of tetraethyl (2-(p-tolyl)ethane-1,1-diyl)bis(phosphonate) (2e)

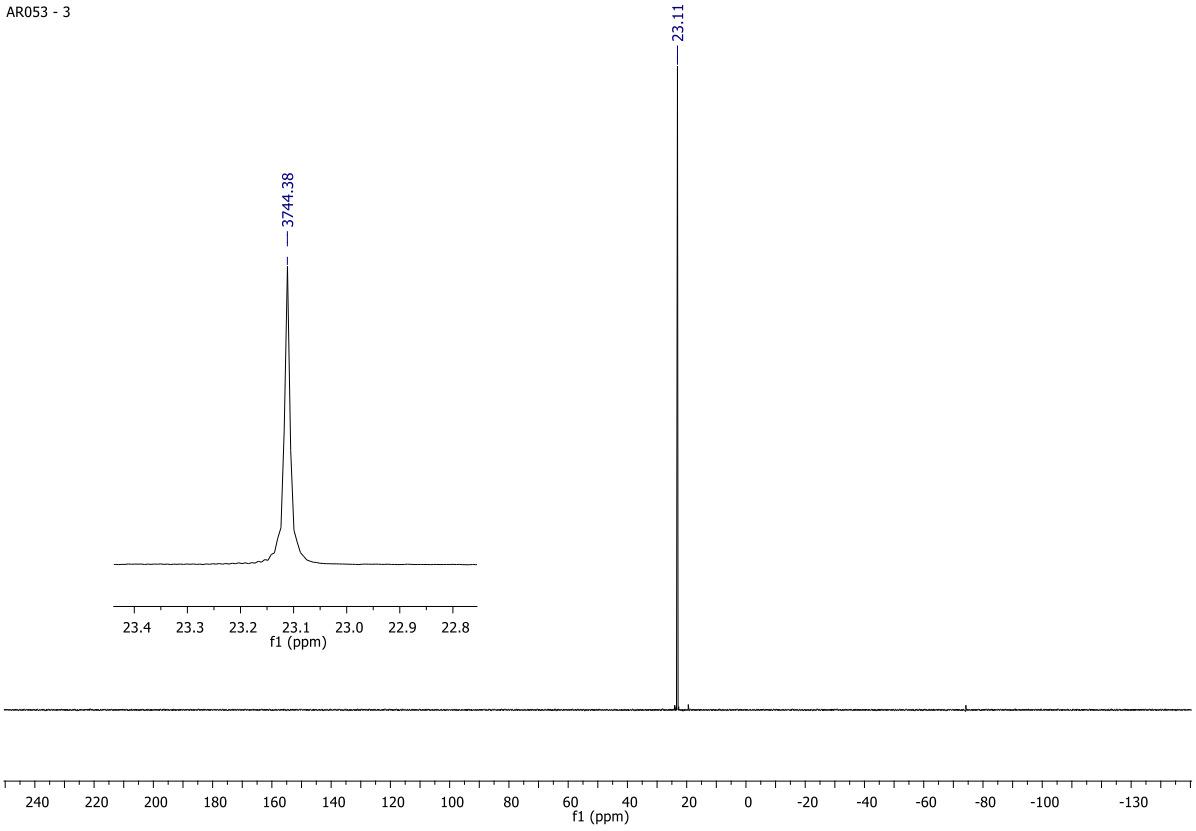


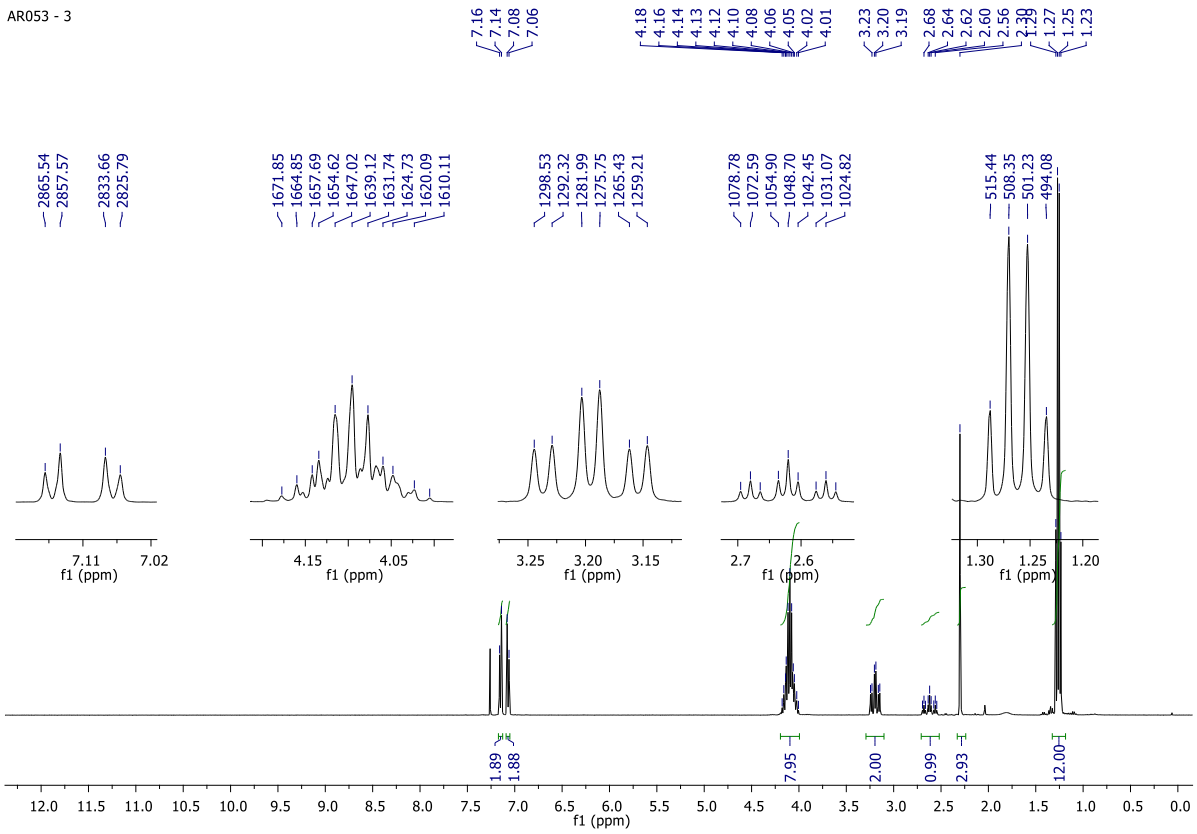
tetraethyl (2-(*p*-tolyl)ethane-1,1-diyl)bis(phosphonate)

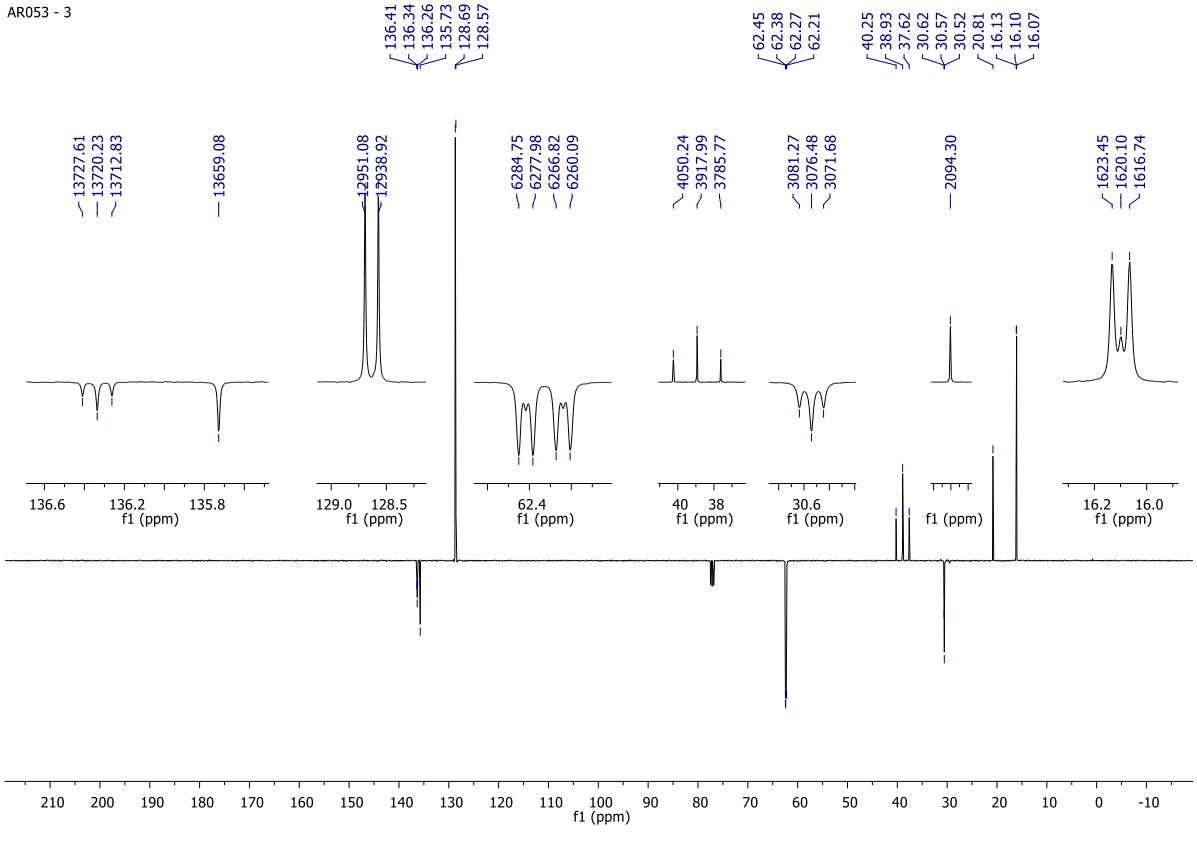
$C_{17}H_{30}O_6P_2$

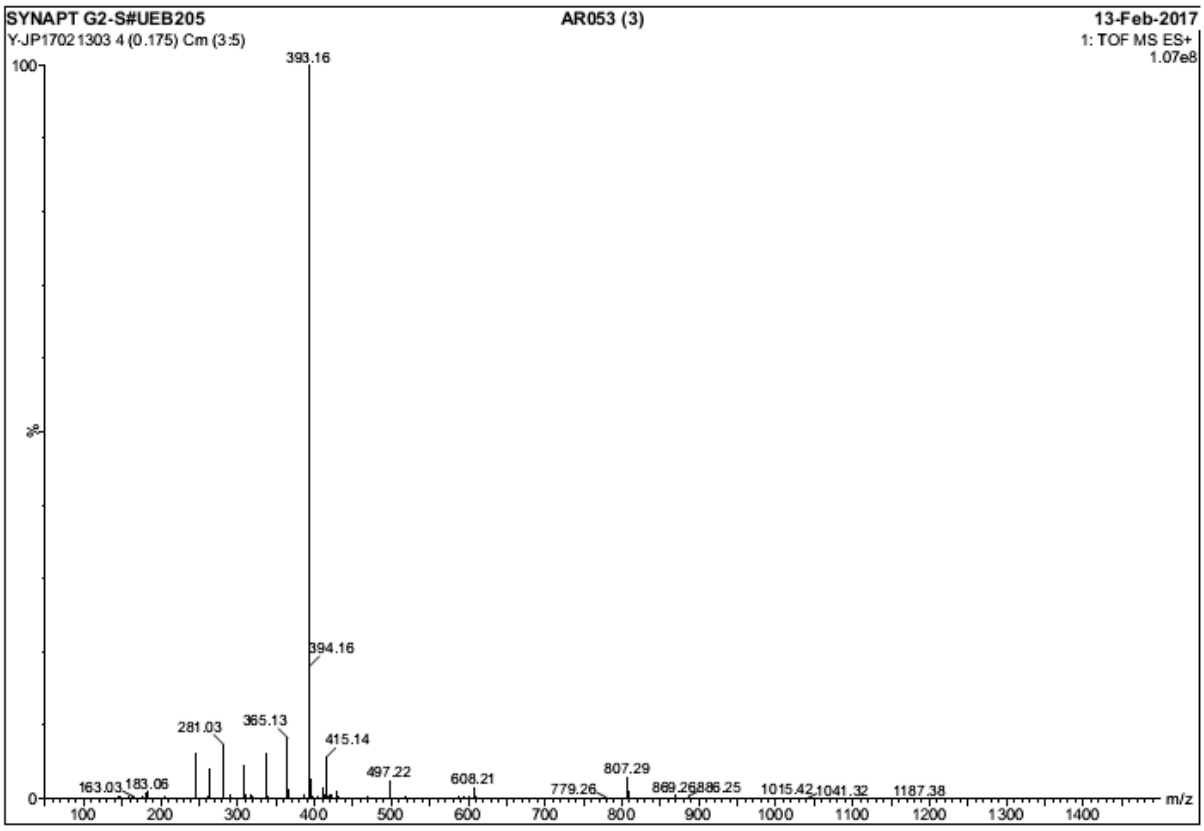
392,37 g/mol

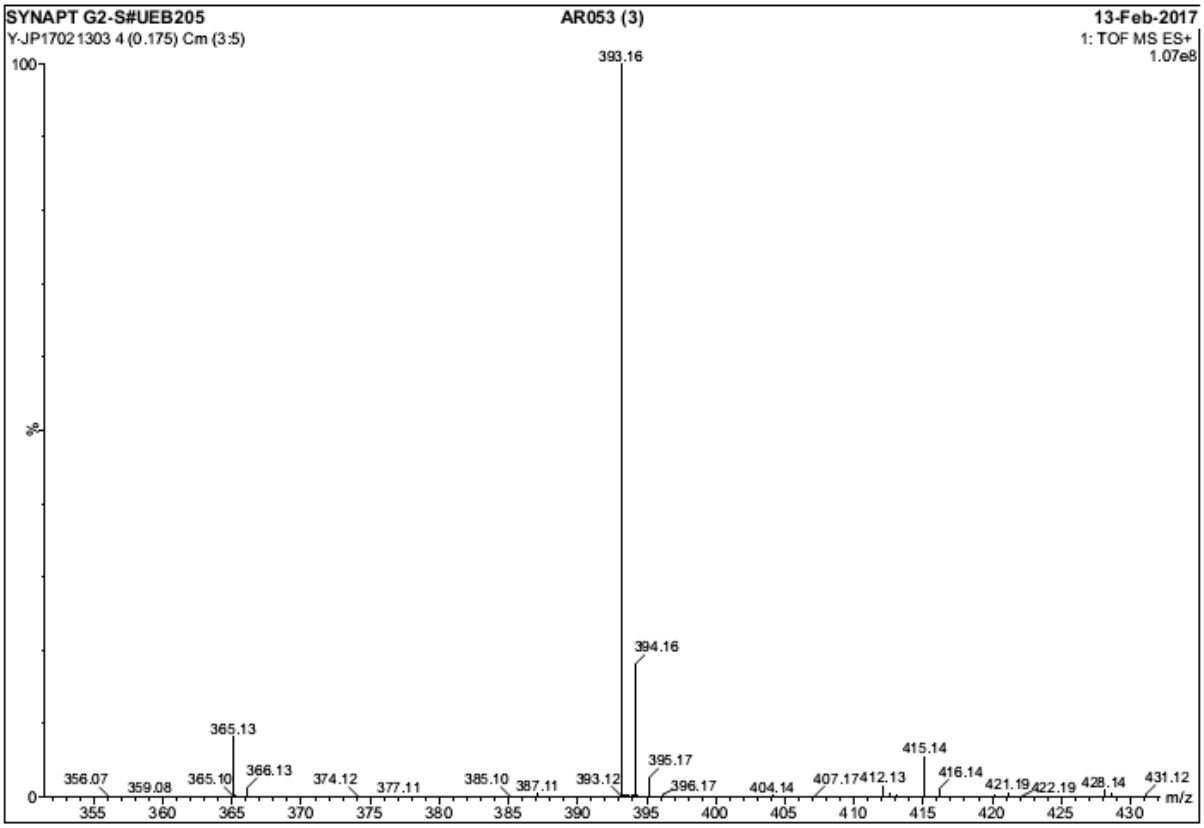
Yellow oil




AR053 - 3
7.16
7.14
7.08
7.06
4.18
4.16
4.14
4.13
4.12
4.10
4.08
4.06
4.05
4.02
4.01
3.23
3.20
3.19
2.68
2.64
2.62
2.60
2.56
1.27
1.25
1.23
2865.54
2857.57
2833.66
2825.79
1671.85
1664.85
1657.69
1654.62
1647.02
1639.12
1631.74
1624.73
1620.09
1610.11
1298.53
1292.32
1281.99
1275.75
1265.43
1259.21
1078.78
1072.59
1054.90
1048.70
1042.45
1031.07
1024.82
515.44
508.35
501.23
494.08
1.89
1.88
7.95
2.00
0.99
2.93
12.00
f1 (ppm)


AR053 - 3
136.41
136.34
136.26
135.73
128.69
128.57
62.45
62.38
62.27
62.21
40.25
38.93
37.62
30.62
30.57
30.52
20.81
16.13
16.10
16.07
13727.61
13720.23
13712.83
13659.08
12951.08
12938.92
6284.75
6277.98
6266.82
6260.09
4050.24
3917.99
3785.77
3081.27
3076.48
3071.68
2094.30
1623.45
1620.10
1616.74
f1 (ppm)


SYNAPT G2-S#UEB205
AR053 (3)
13-Feb-2017
Y-JP1702 1303 4 (0.175) Cm (3:5)
1: TOF MS ES+
1.07e8
393.16
394.16
281.03
365.13
415.14
497.22
608.21
807.29
163.03
183.06
779.26
869.26
886.25
1015.42
1041.32
1187.38
%
m/z

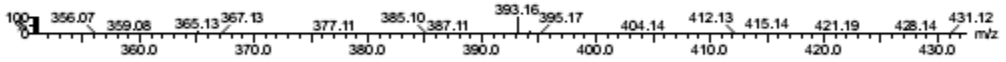

SYNAPT G2-S#UEB205
AR053 (3)
13-Feb-2017
Y-JP1702 1303 4 (0.175) Cm (3:5)
1: TOF MS ES+
1.07e8
393.16
394.16
365.13
395.17
415.14
356.07
359.08
365.10
366.13
374.12
377.11
385.10
387.11
393.12
396.17
404.14
407.17
412.13
416.14
421.19
422.19
428.14
431.12
%
m/z

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

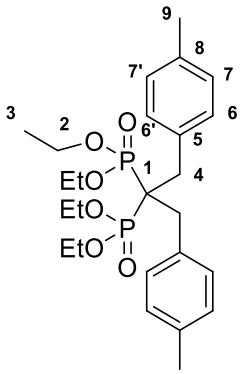
Monoisotopic Mass, Even Electron Ions
794 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 2-2

SYNAPT G2-S#UEB205 AR053 (3) 13-Feb-2017
Y-JP17021303 4 (0.175) Cm (3:5) 1: TOF MS ES+
1.07e+008



Minimum:				-1.5				
Maximum:		10.0	3.0	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, ^{1}H and ^{13}C NMR spectra and mass analysis of tetraethyl (1,3-di-p-tolylpropane-2,2-diyl)bis(phosphonate) (3e)

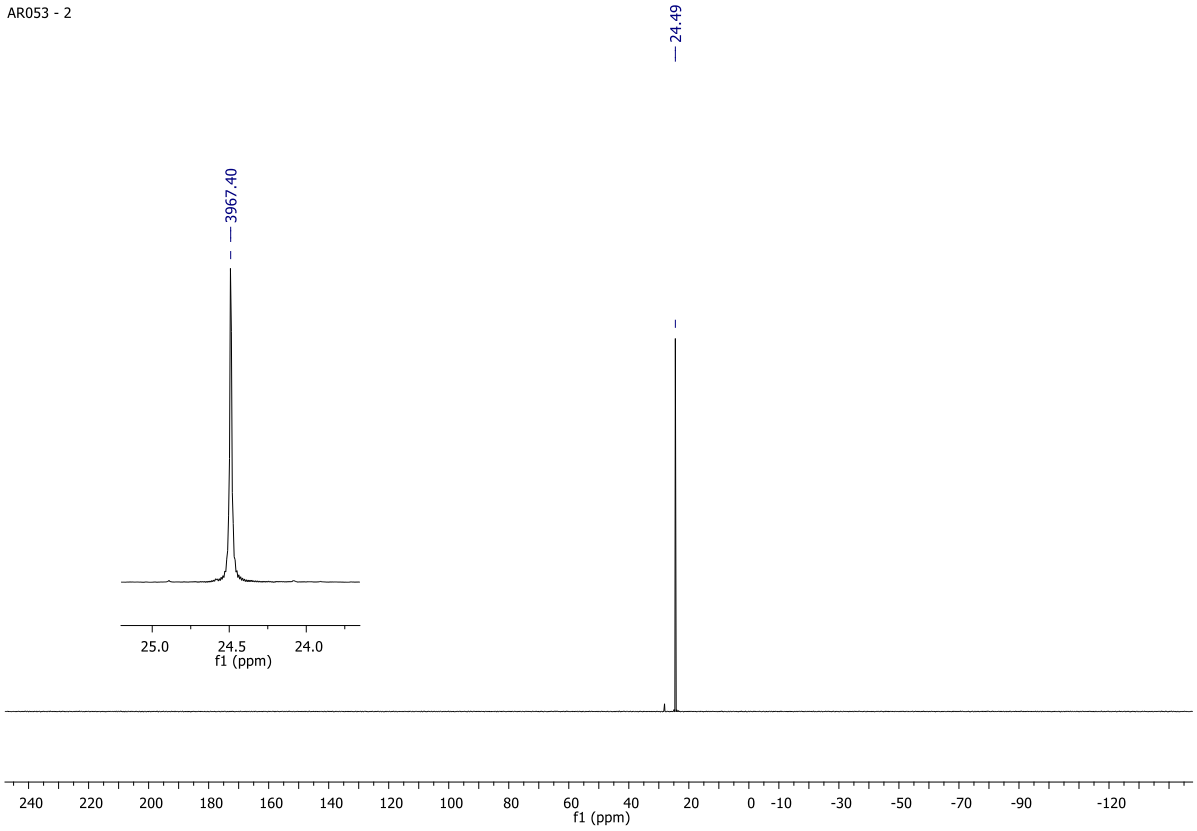


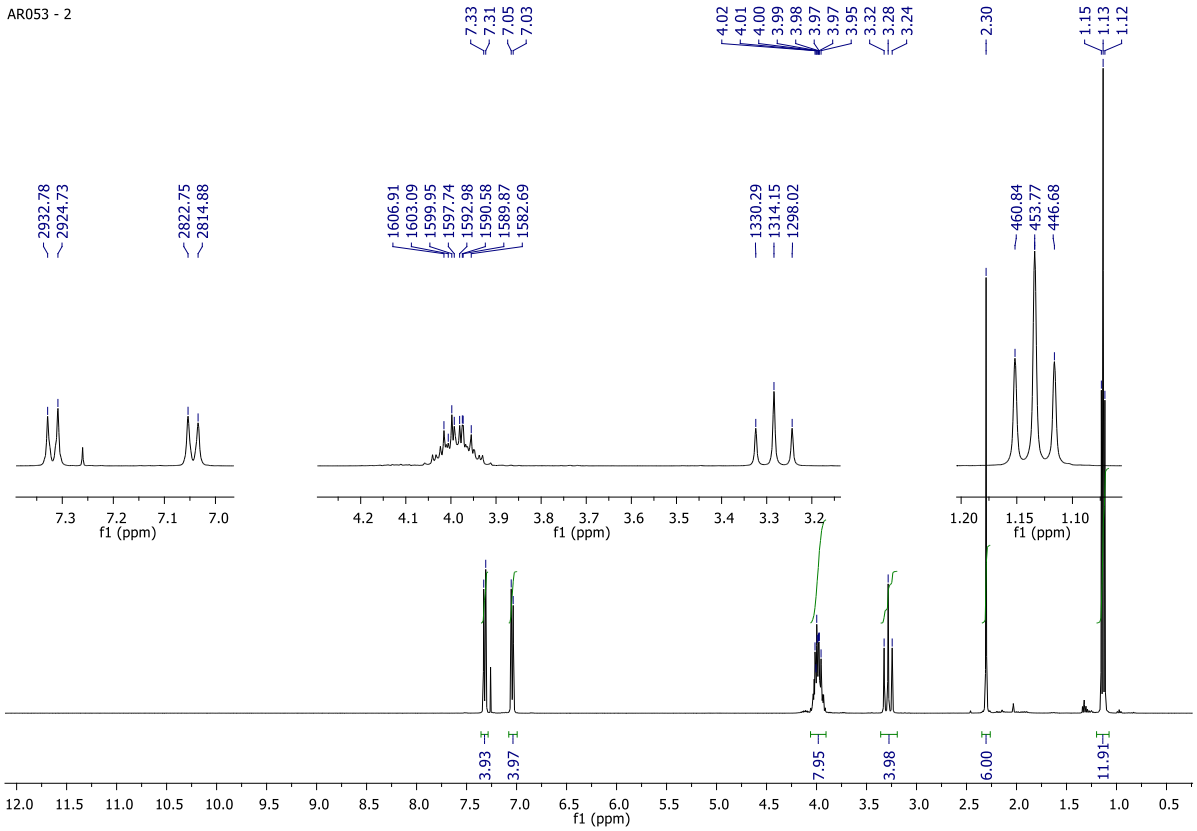
tetraethyl (1,3-di-*p*-tolylpropane-2,2-diyl)bis(phosphonate)

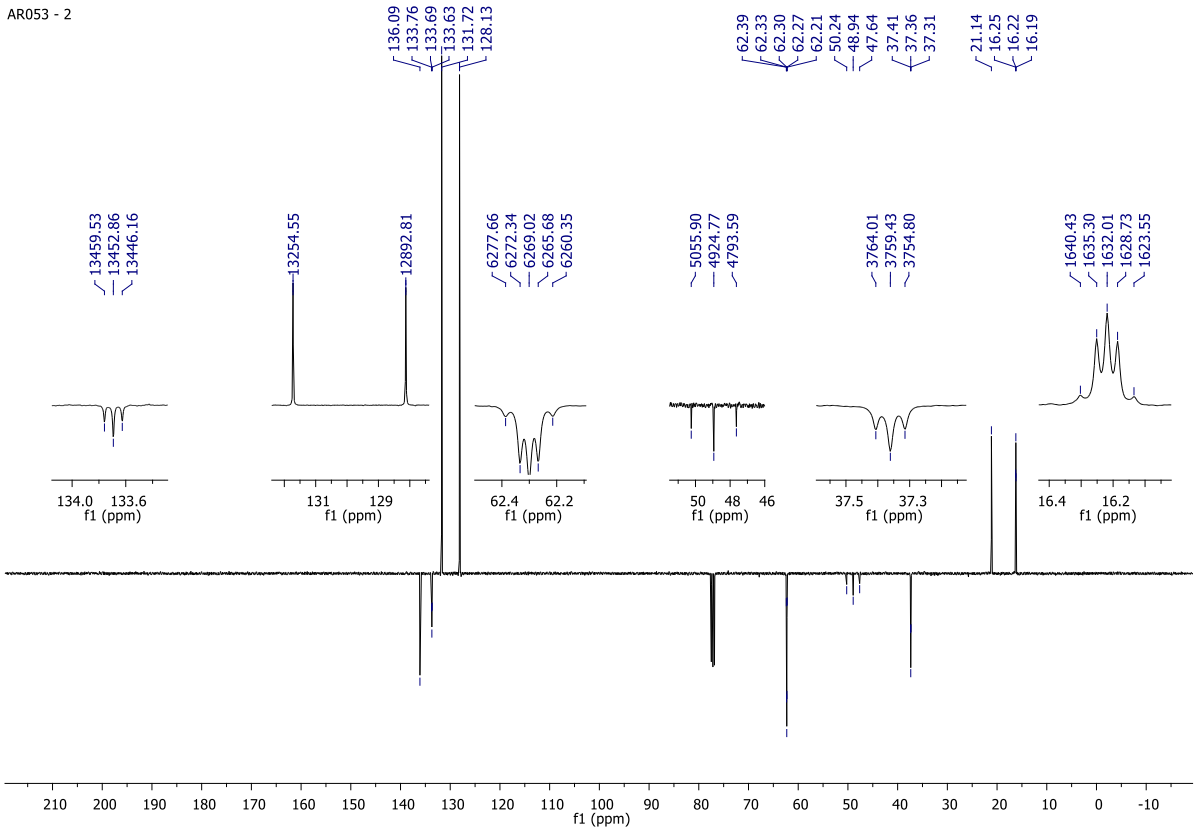
$C_{25}H_{38}O_6P_2$

492,52 g/mol

Yellow oil




AR053 - 2
7.33
7.31
7.05
7.03
4.02
4.01
4.00
3.99
3.98
3.97
3.97
3.95
3.32
3.28
3.24
2.30
1.15
1.13
1.12
2932.78
2924.73
2822.75
2814.88
1606.91
1603.09
1599.95
1597.74
1592.98
1590.58
1589.87
1582.69
1330.29
1314.15
1298.02
460.84
453.77
446.68
f1 (ppm)
3.93
3.97
7.95
3.98
6.00
11.91


AR053 - 2
136.09
133.76
133.69
133.63
131.72
128.13
62.39
62.33
62.30
62.27
62.21
50.24
48.94
47.64
37.41
37.36
37.31
21.14
16.25
16.22
16.19
13459.53
13452.86
13446.16
13254.55
12892.81
6277.66
6272.34
6269.02
6265.68
6260.35
5055.90
4924.77
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3754.80
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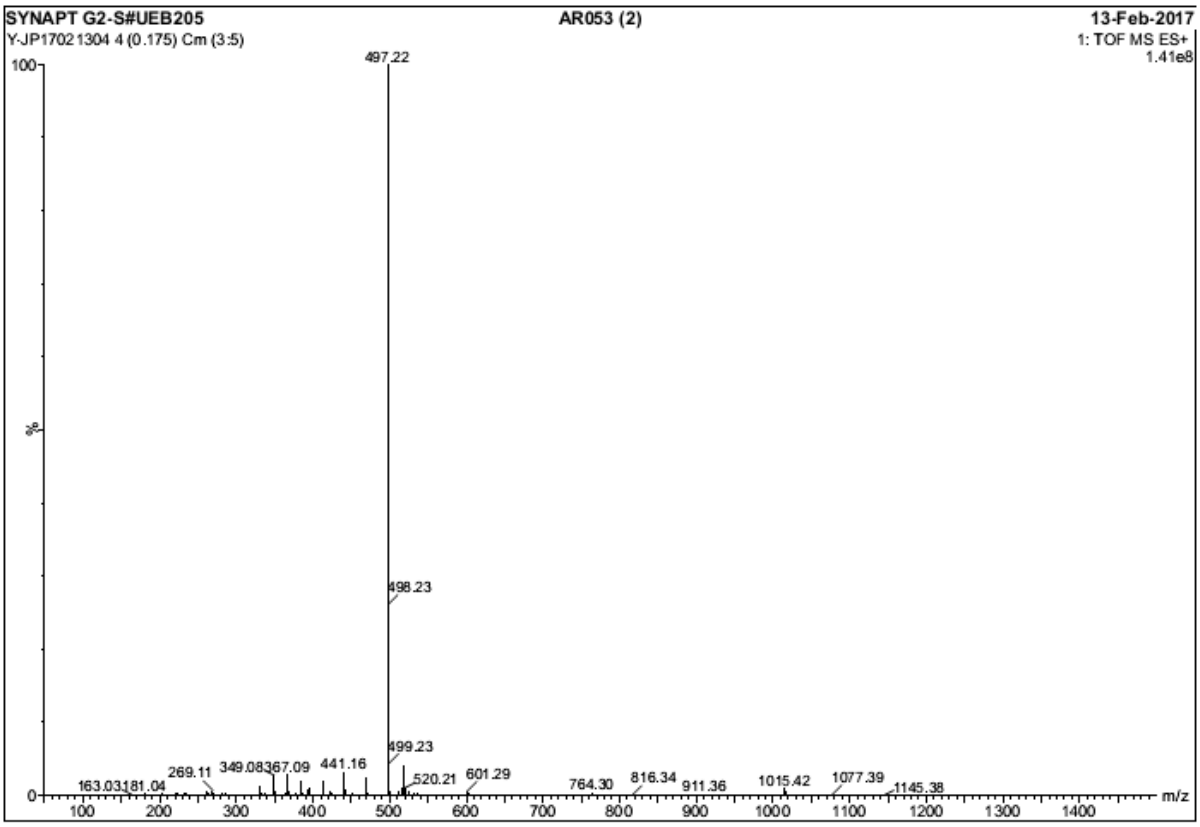
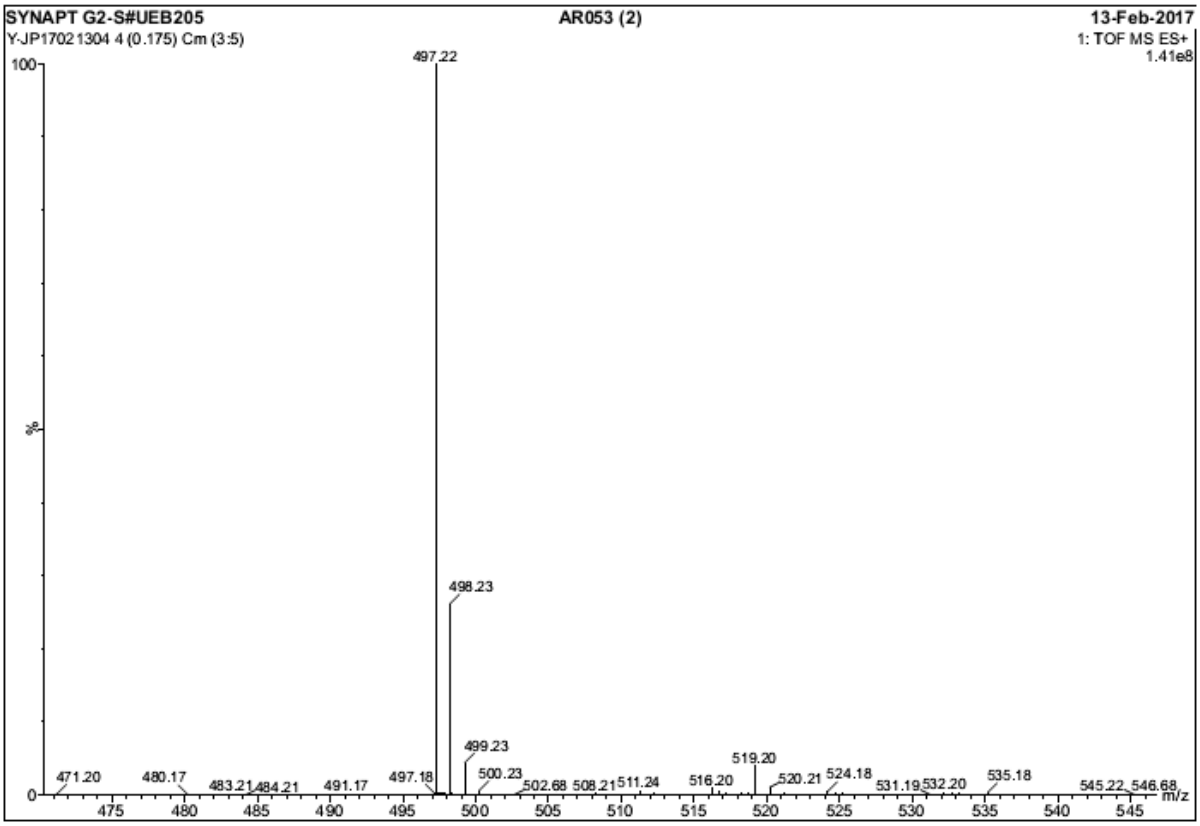
1635.30
1632.01
1628.73
1623.55
f1 (ppm)


SYNAPT G2-S#UEB205
AR053 (2)
13-Feb-2017
Y-JP17021304 4 (0.175) Cm (3:5)
1: TOF MS ES+
1.41e8
497.22
498.23
499.23
163.03
181.04
269.11
349.08
367.09
441.16
520.21
601.29
764.30
816.34
911.36
1015.42
1077.39
1145.38
%
m/z

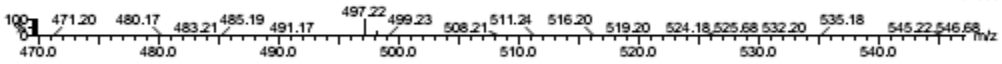

SYNAPT G2-S#UEB205
AR053 (2)
13-Feb-2017
Y-JP17021304 4 (0.175) Cm (3:5)
1: TOF MS ES+
1.41e8
497.22
498.23
499.23
471.20
480.17
483.21
484.21
491.17
497.18
500.23
502.68
508.21
511.24
516.20
519.20
520.21
524.18
531.19
532.20
535.18
545.22
546.68
%
m/z

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

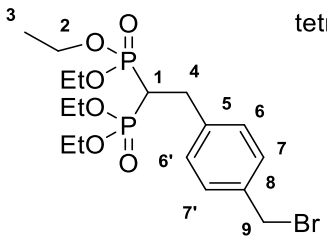
Monoisotopic Mass, Even Electron Ions
1410 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 0-100 H: 0-100 N: 0-20 O: 0-20 P: 2-2

SYNAPT G2-S#UEB205 AR053 (2) 13-Feb-2017
Y-JP17021304 4 (0.175) Cm (3:5) 1: TOF MS ES+
1.41e+008



Minimum:				-1.5				
Maximum:		10.0	1.0	50.0				
Mass	Calc. Mass	mDa	PPM	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, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl (1,3-bis(4-(bromomethyl)phenyl)propane-2,2-diyl)bis(phosphonate) (2f)

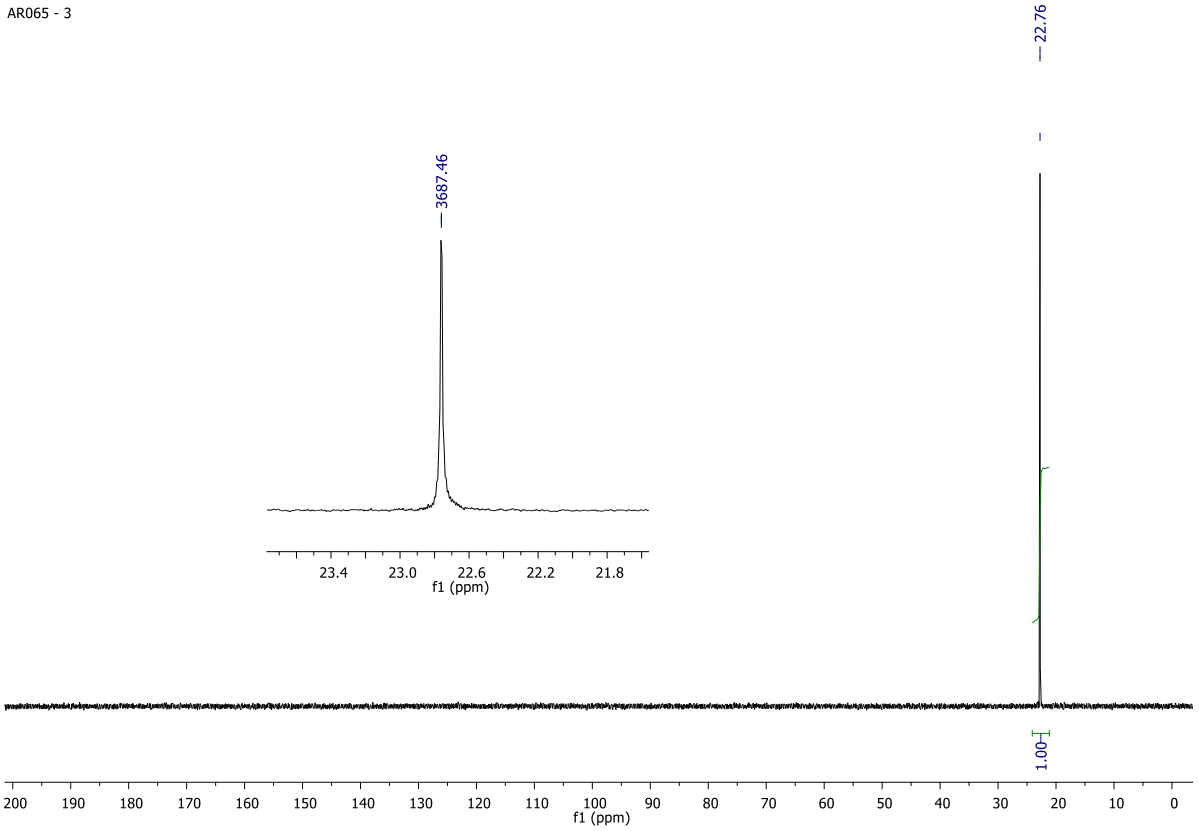


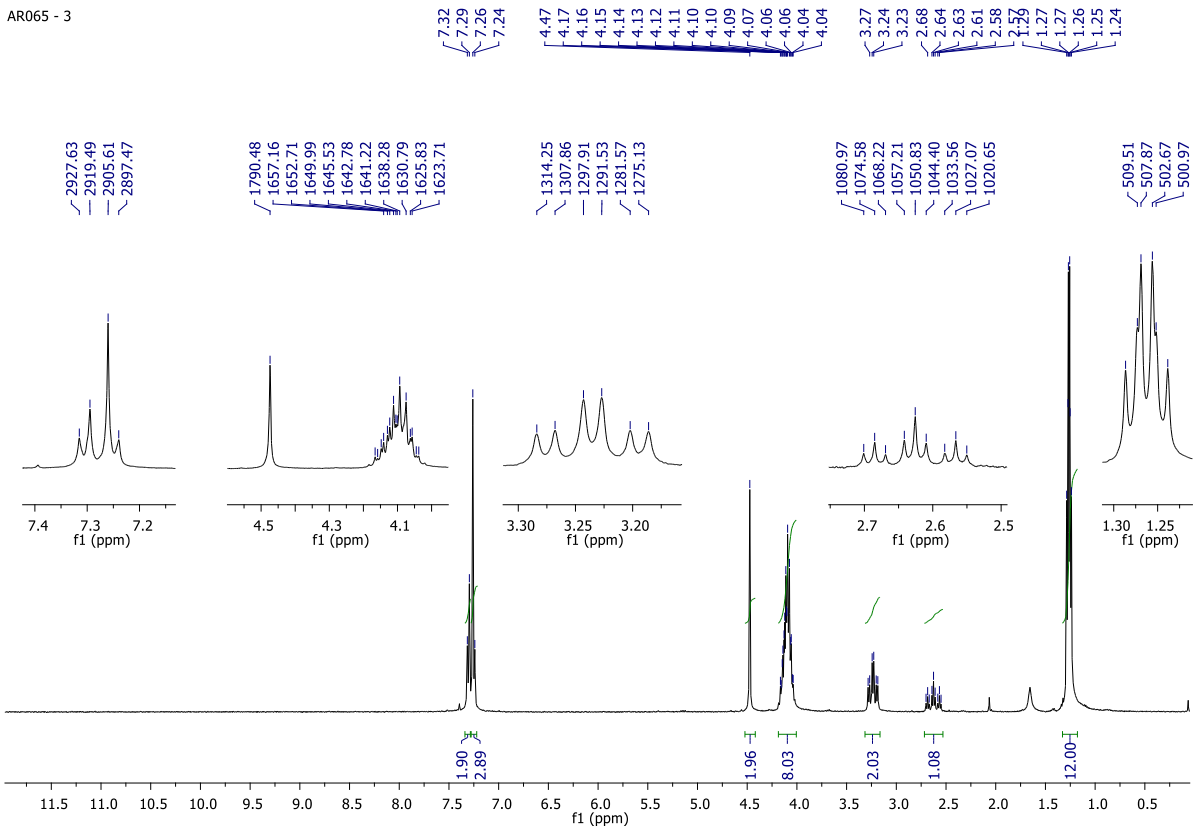
tetraethyl (2-(4-(bromomethyl)phenyl)ethane-1,1-diyl)bis(phosphonate)

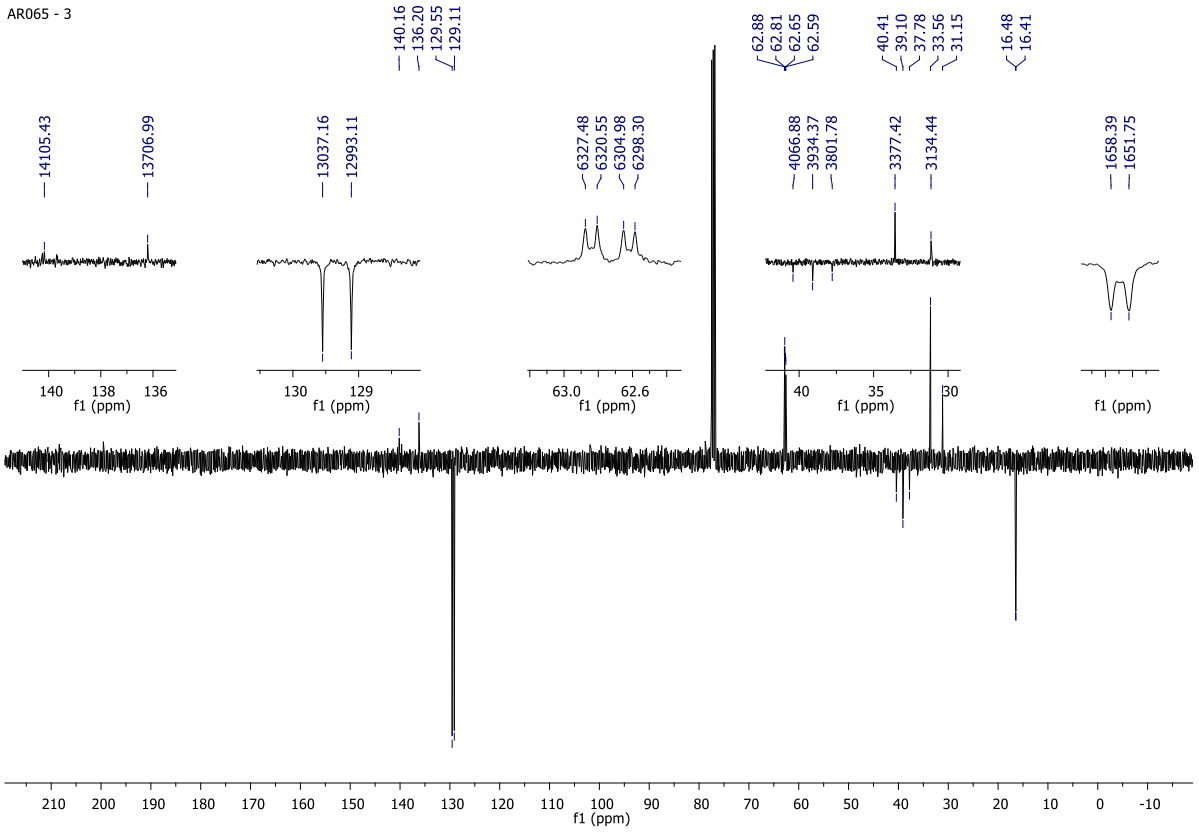
$C_{17}H_{29}O_6BrP_2$

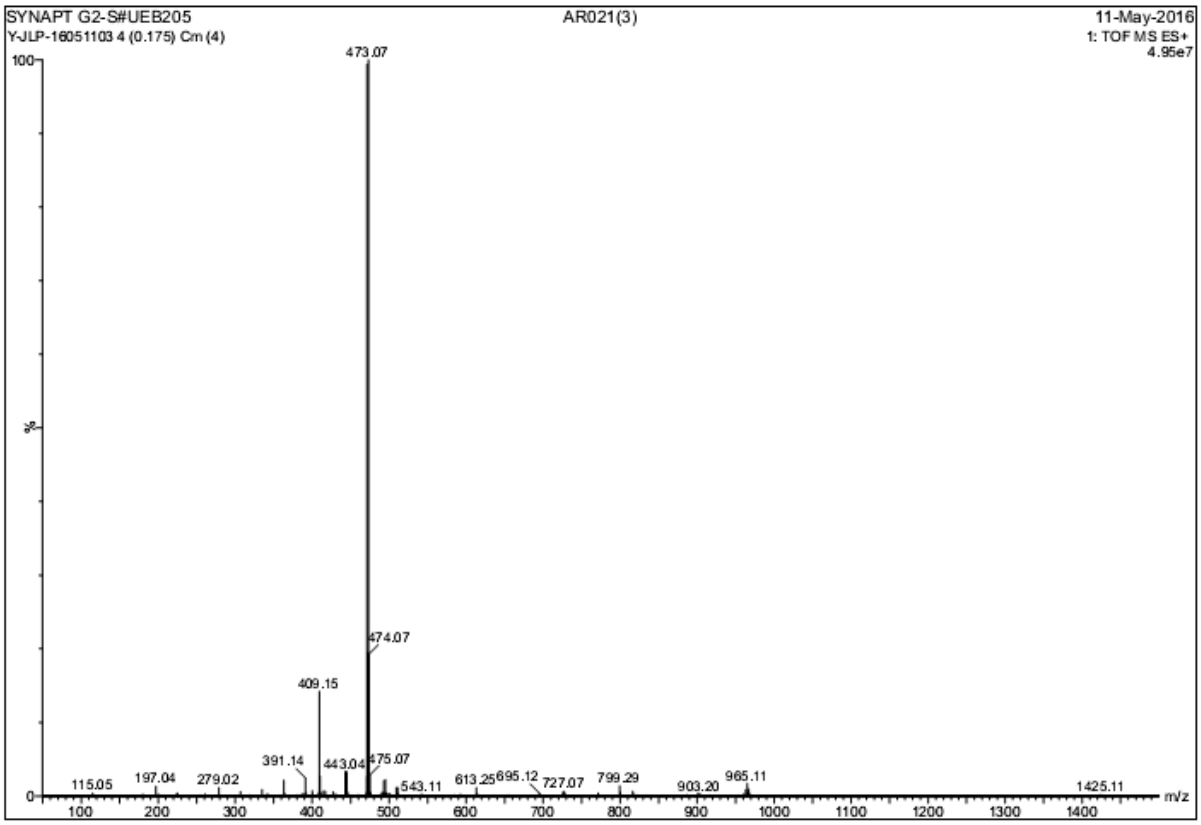
471,26 g/mol

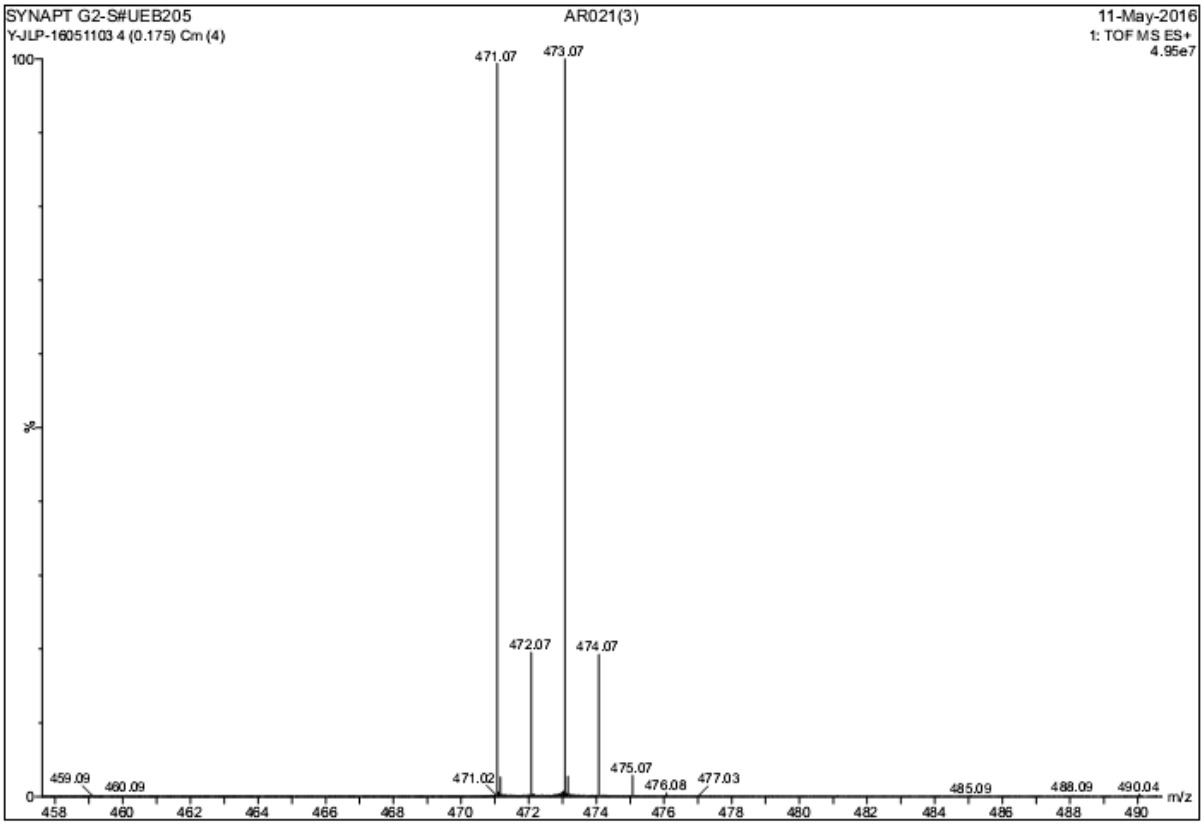
Yellow oil




AR065 - 3
7.32 7.29 7.26 7.24
4.47 4.17 4.16 4.15 4.14 4.13 4.12 4.11 4.10 4.10 4.09 4.07 4.06 4.06 4.04 4.04
3.27 3.24 3.23
2.68 2.64 2.63 2.61 2.58 2.57 2.55 1.27 1.27 1.26 1.25 1.24
2927.63 2919.49 2905.61 2897.47
1790.48 1657.16 1652.71 1649.99 1645.53 1642.78 1641.22 1638.28 1630.79 1625.83 1623.71
1314.25 1307.86 1297.91 1291.53 1281.57 1275.13
1080.97 1074.58 1068.22 1057.21 1050.83 1044.40 1033.56 1027.07 1020.65
509.51 507.87 502.67 500.97
1.90 2.89 1.96 8.03 2.03 1.08 12.00
f1 (ppm)


AR065 - 3
140.16 136.20 129.55 129.11
62.88 62.81 62.65 62.59
40.41 39.10 37.78 33.56 31.15
16.48 16.41
14105.43 13706.99
13037.16 12993.11
6327.48 6320.55 6304.98 6298.30
4066.88 3934.37 3801.78
3377.42 3134.44
1658.39 1651.75
f1 (ppm)


SYNAPT G2-S#UEB205
AR021(3)
11-May-2016
Y-JLP-16051103 4 (0.175) Cm (4)
1: TOF MS ES+
4.95e7
100
%
0
473.07
474.07
409.15
391.14
443.04
475.07
115.05
197.04
279.02
543.11
613.25
695.12
727.07
799.29
903.20
965.11
1425.11
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400
m/z

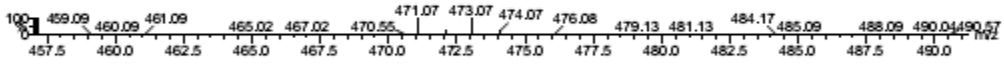

SYNAPT G2-S#UEB205
AR021(3)
11-May-2016
Y-JLP-16051103 4 (0.175) Cm (4)
1: TOF MS ES+
4.95e7
100
%
0
471.07
473.07
472.07
474.07
475.07
459.09
460.09
471.02
476.08
477.03
485.09
488.09
490.04
458
460
462
464
466
468
470
472
474
476
478
480
482
484
486
488
490
m/z

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

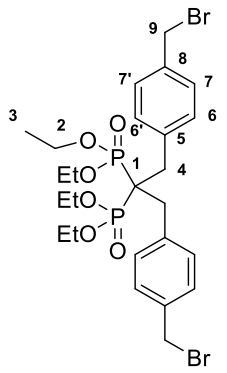
Monoisotopic Mass, Even Electron Ions
4436 formula(e) evaluated with 6 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 0-10 O: 0-10 P: 1-2 Br: 1-2

SYNAPT G2-S#UEB205 AR021(3) 11-May-2016
Y-JLP-16051103 4 (0.175) Cm (4) 1: TOF MS ES+
4.95e+007



Minimum:				-50.0				
Maximum:		1.1	2.0	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

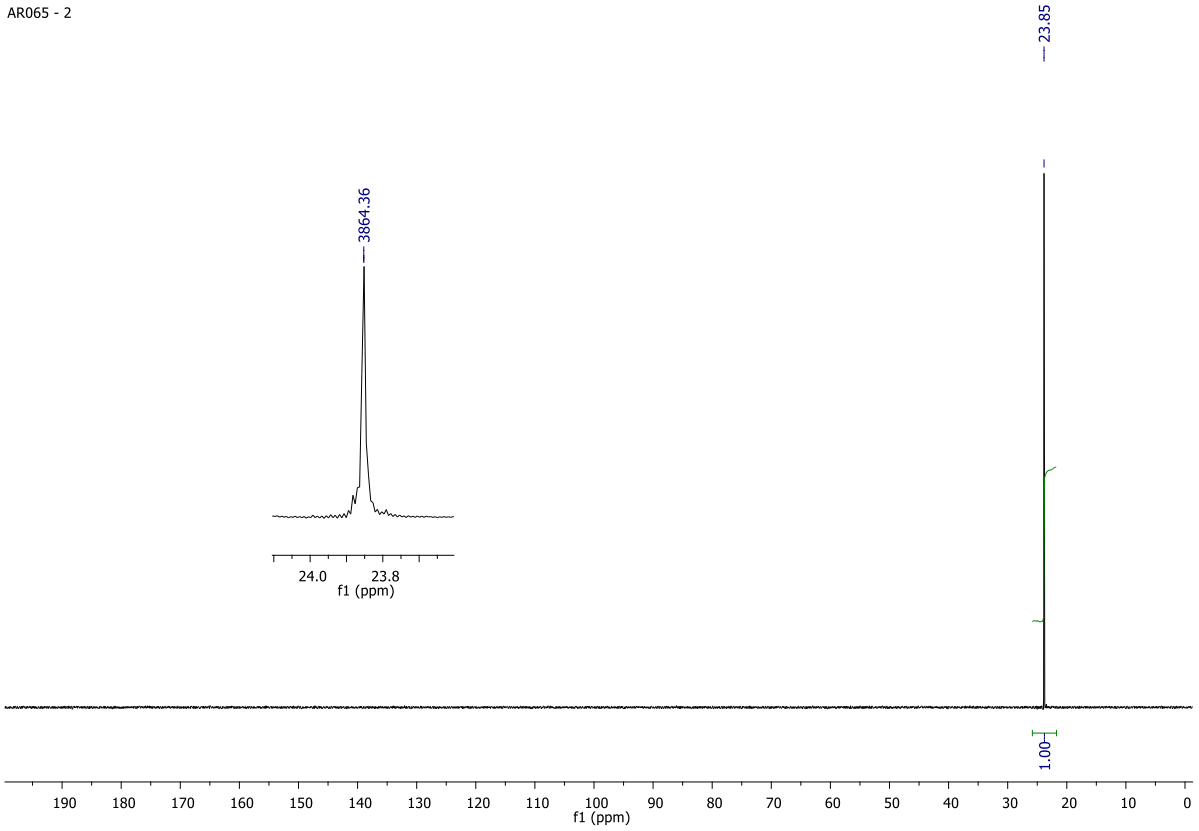
S12: ^{31}P, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl (1,3-bis(4-(bromomethyl)phenyl)propane-2,2-diyl)bis(phosphonate) (3f)

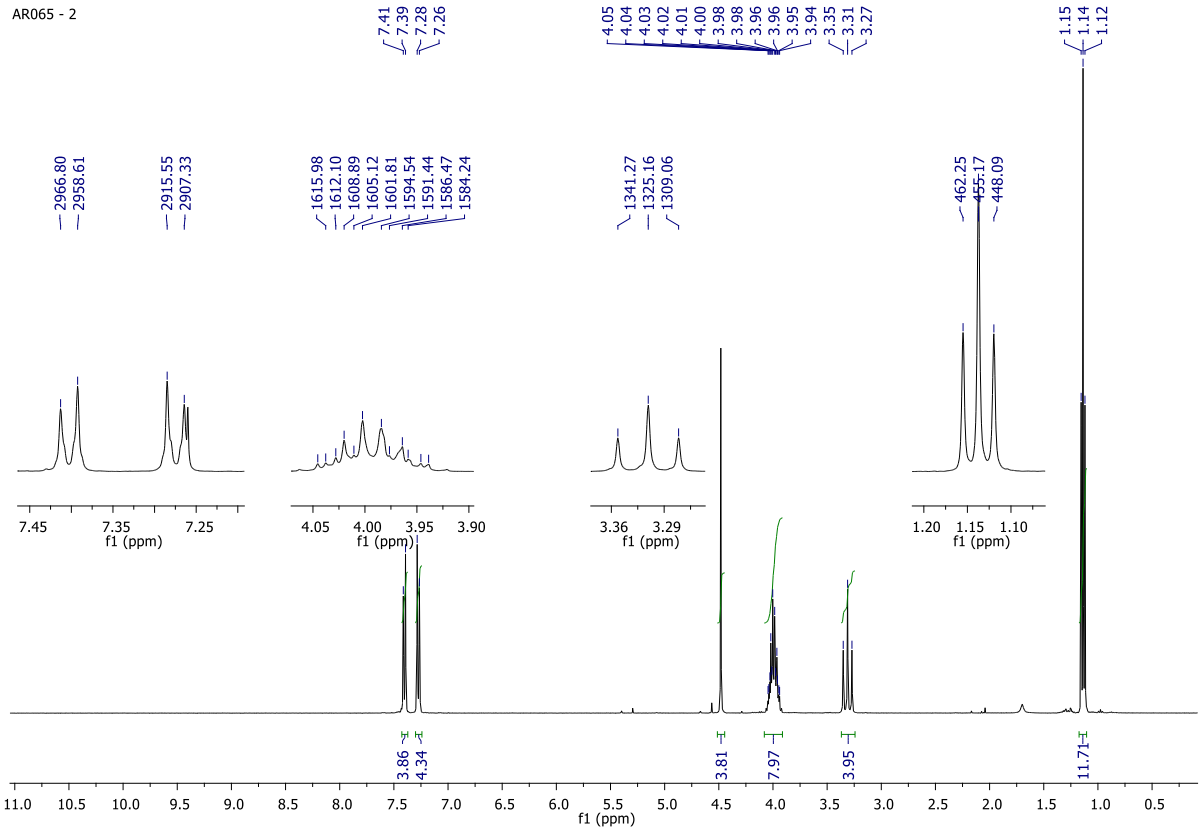


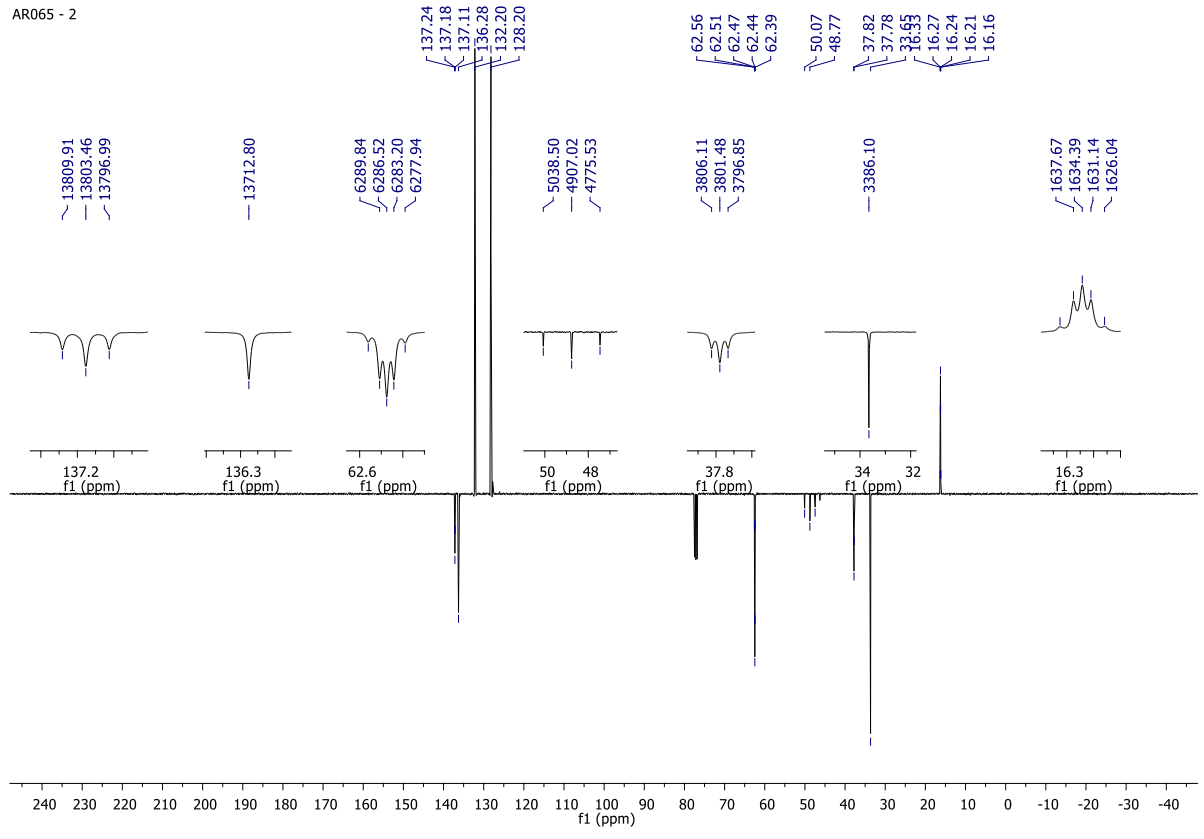
tetraethyl (1,3-bis(4-(bromomethyl)phenyl)propane-2,2-diyl)bis(phosphonate)
$C_{25}H_{36}O_6Br_2P_2$

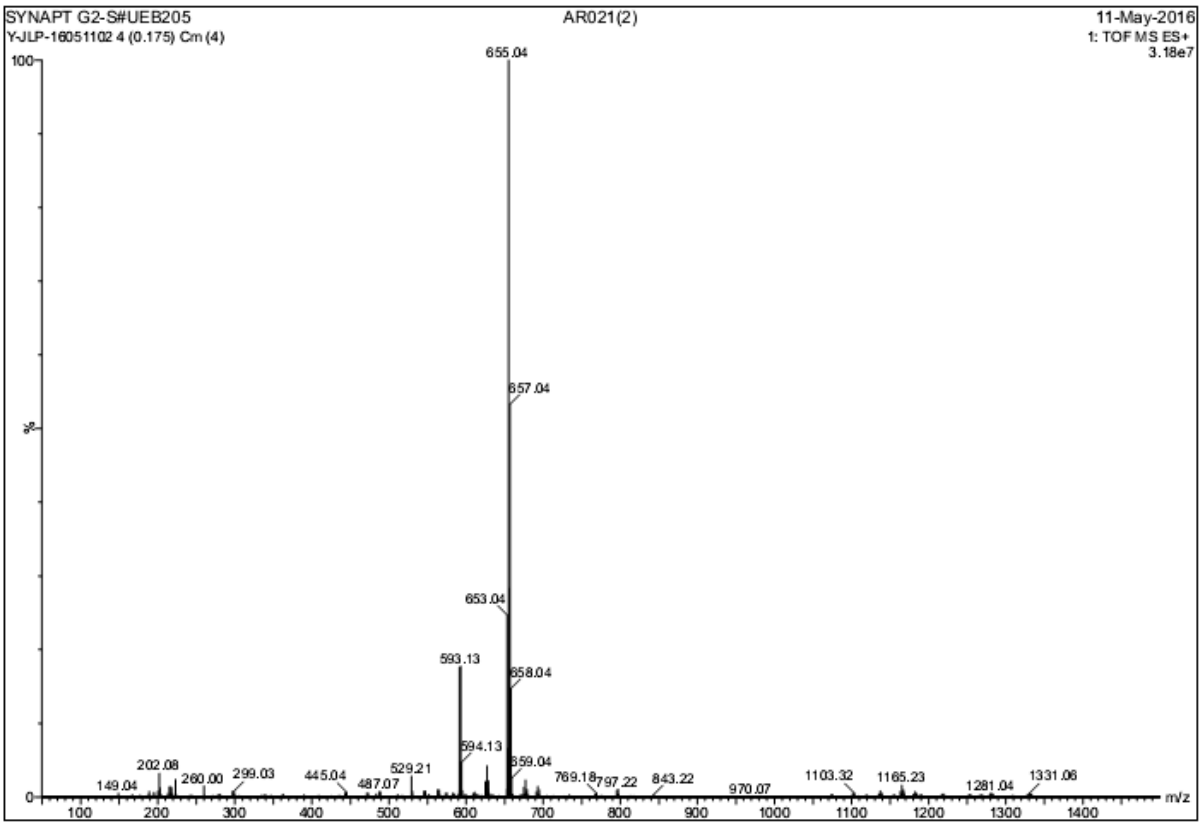
654,31 g/mol

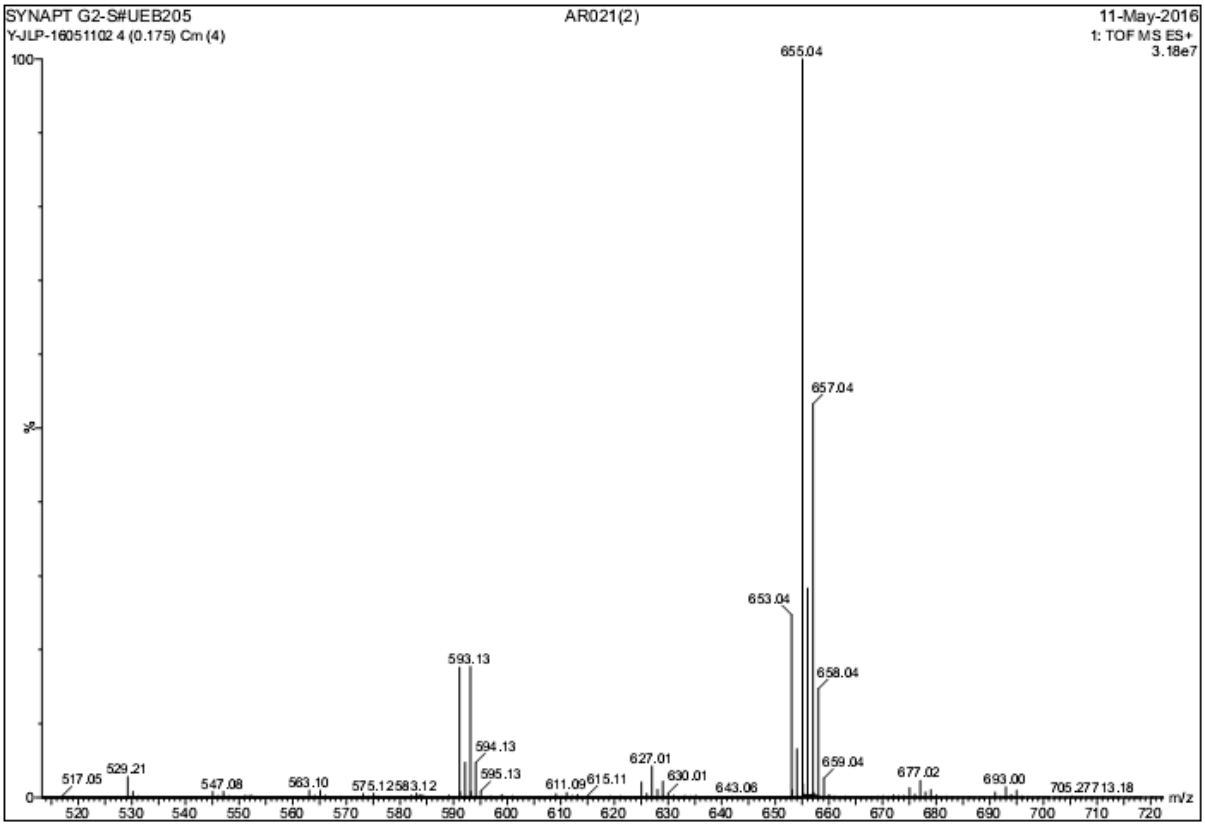
Yellow oil




AR065 - 2
7.41
7.39
7.28
7.26
4.05
4.04
4.03
4.02
4.01
4.00
3.98
3.98
3.96
3.96
3.95
3.94
3.35
3.31
3.27
1.15
1.14
1.12
2966.80
2958.61
2915.55
2907.33
1615.98
1612.10
1608.89
1605.12
1601.81
1594.54
1591.44
1586.47
1584.24
1341.27
1325.16
1309.06
462.25
455.17
448.09
f1 (ppm)
3.86
4.34
3.81
7.97
3.95
11.71


AR065 - 2
137.24
137.18
137.11
136.28
132.20
128.20
62.56
62.51
62.47
62.44
62.39
50.07
48.77
37.82
37.78
33.65
33.33
16.27
16.24
16.21
16.16
13809.91
13803.46
13796.99
13712.80
6289.84
6286.52
6283.20
6277.94
5038.50
4907.02
4775.53
3806.11
3801.48
3796.85
3386.10
1637.67
1634.39
1631.14
1626.04
f1 (ppm)


SYNAPT G2-S#UEB205
AR021(2)
11-May-2016
Y-JLP-16051102 4 (0.175) Cm (4)
1: TOF MS ES+
3.18e7
655.04
657.04
653.04
593.13
658.04
594.13
659.04
202.08
260.00
299.03
445.04
529.21
487.07
149.04
769.18
797.22
843.22
970.07
1103.32
1165.23
1281.04
1331.06
%
m/z

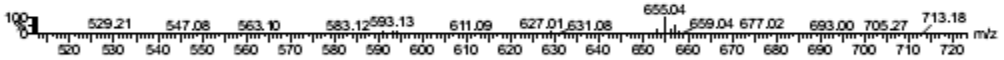

SYNAPT G2-S#UEB205
AR021(2)
11-May-2016
Y-JLP-16051102 4 (0.175) Cm (4)
1: TOF MS ES+
3.18e7
655.04
657.04
653.04
593.13
658.04
594.13
659.04
627.01
529.21
517.05
547.08
563.10
575.12
583.12
595.13
611.09
615.11
630.01
643.06
677.02
693.00
705.27
713.18
%
m/z

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

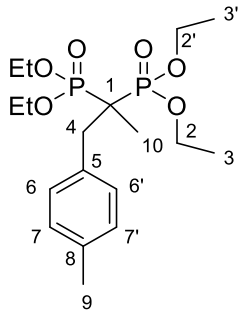
Monoisotopic Mass, Even Electron Ions
2857 formula(e) evaluated with 3 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 0-100 H: 0-150 N: 0-10 O: 0-10 P: 1-2 Br: 2-2

SYNAPT G2-S#UEB205 AR021(2) 11-May-2016
Y-JLP-160511024 (0.175) Cm (4) 1: TOF MS ES+
3.18e+007



Minimum:				-50.0				
Maximum:		1.1	2.0	100.0				
Mass	Calc. Mass	mDa	PPM	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, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl (1-(p-tolyl)propane-2,2-diyl)bis(phosphonate) (6)

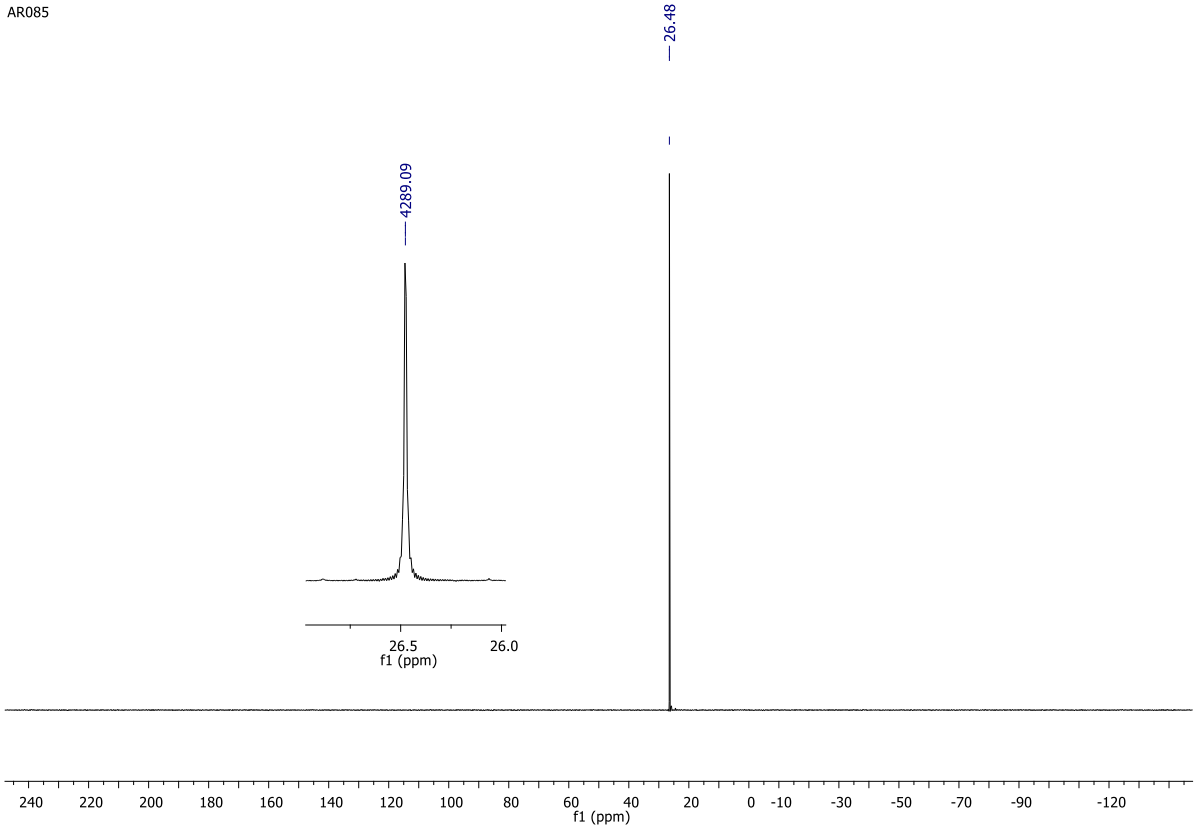


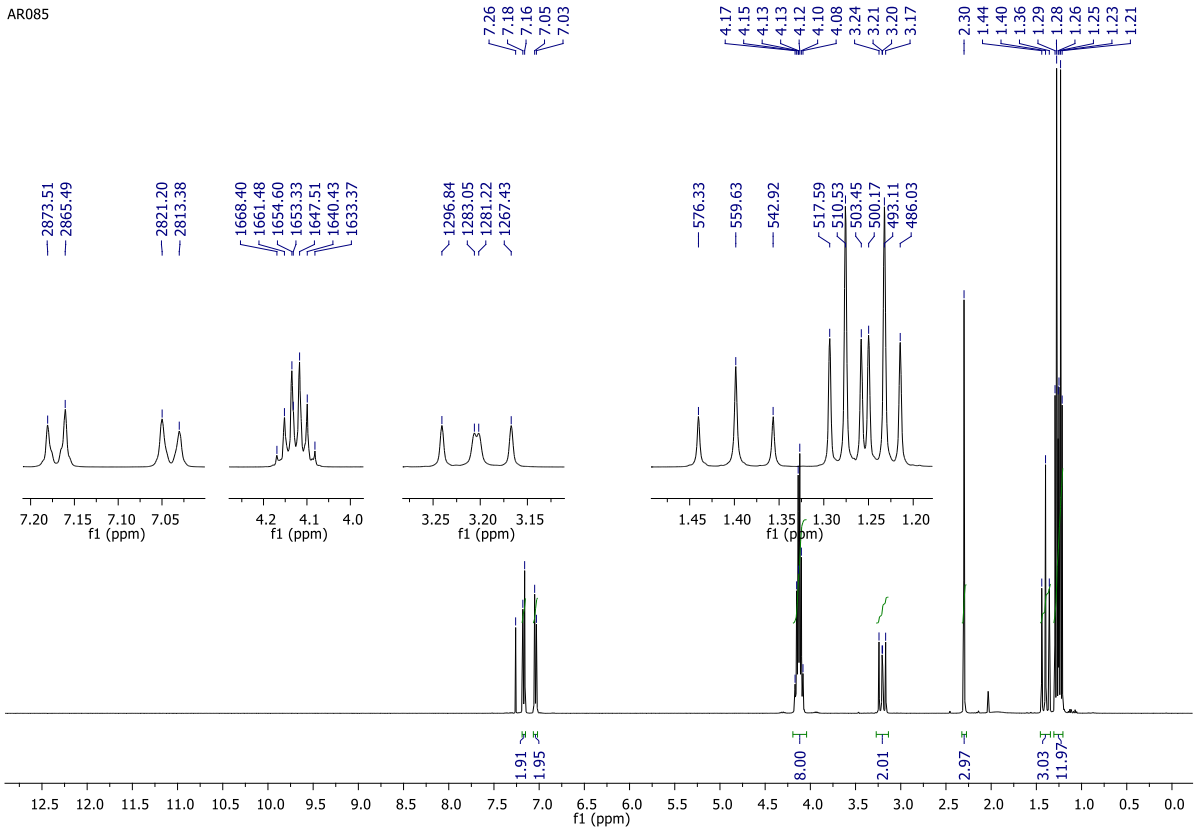
tetraethyl (1-(*p*-tolyl)propane-2,2-diyl)bis(phosphonate)

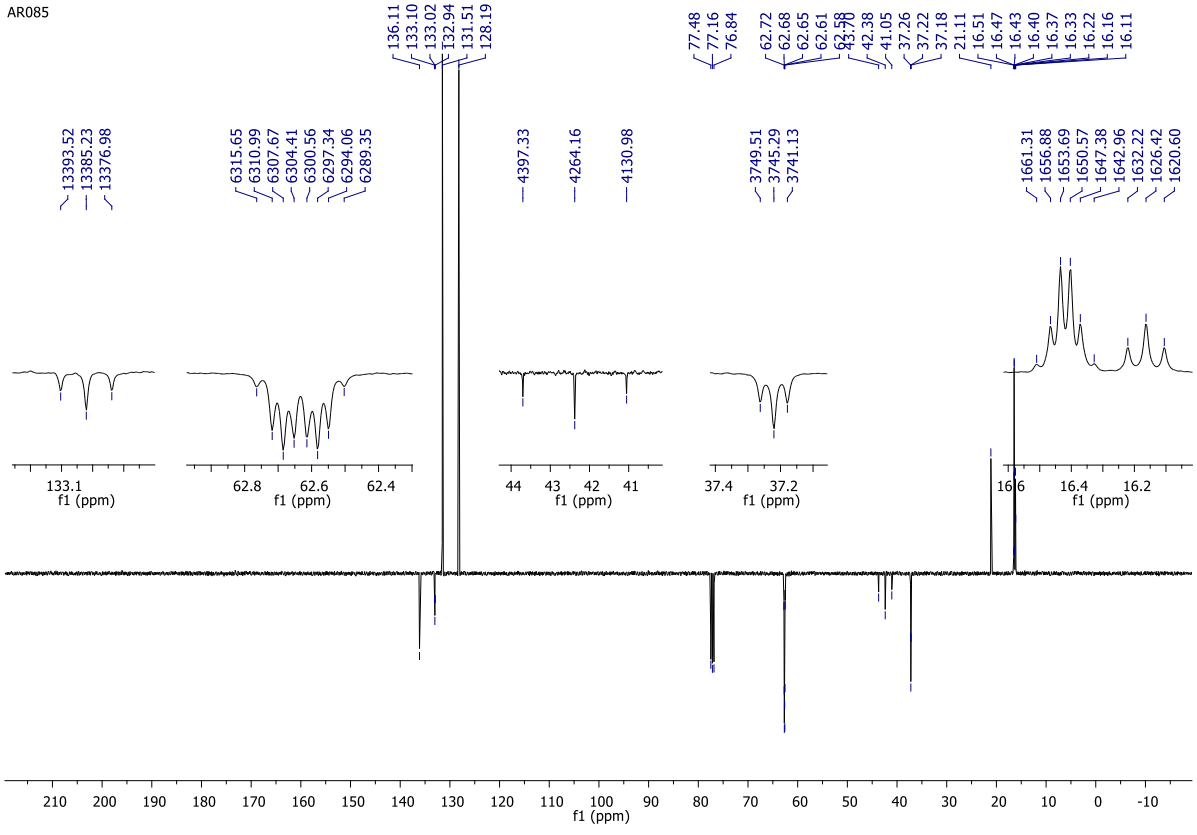
$C_{18}H_{32}O_6P_2$

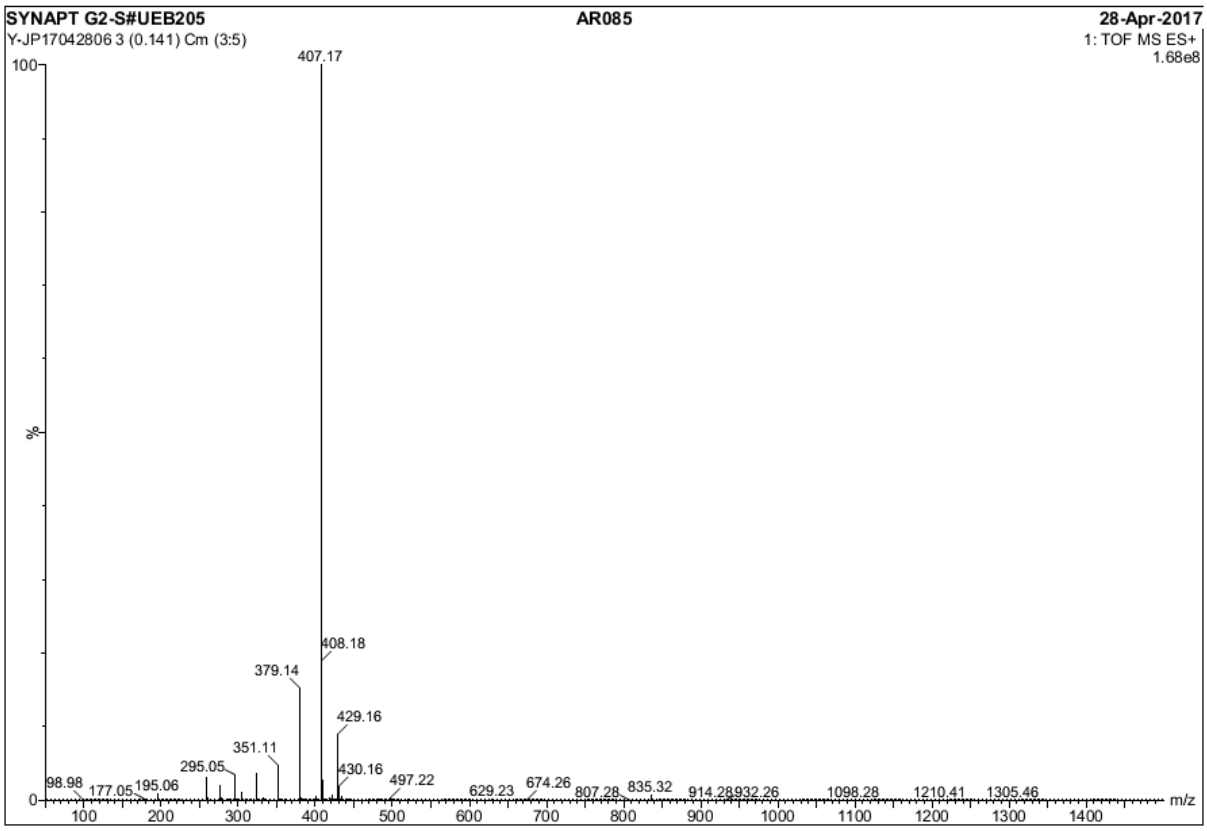
406,40 g/mol

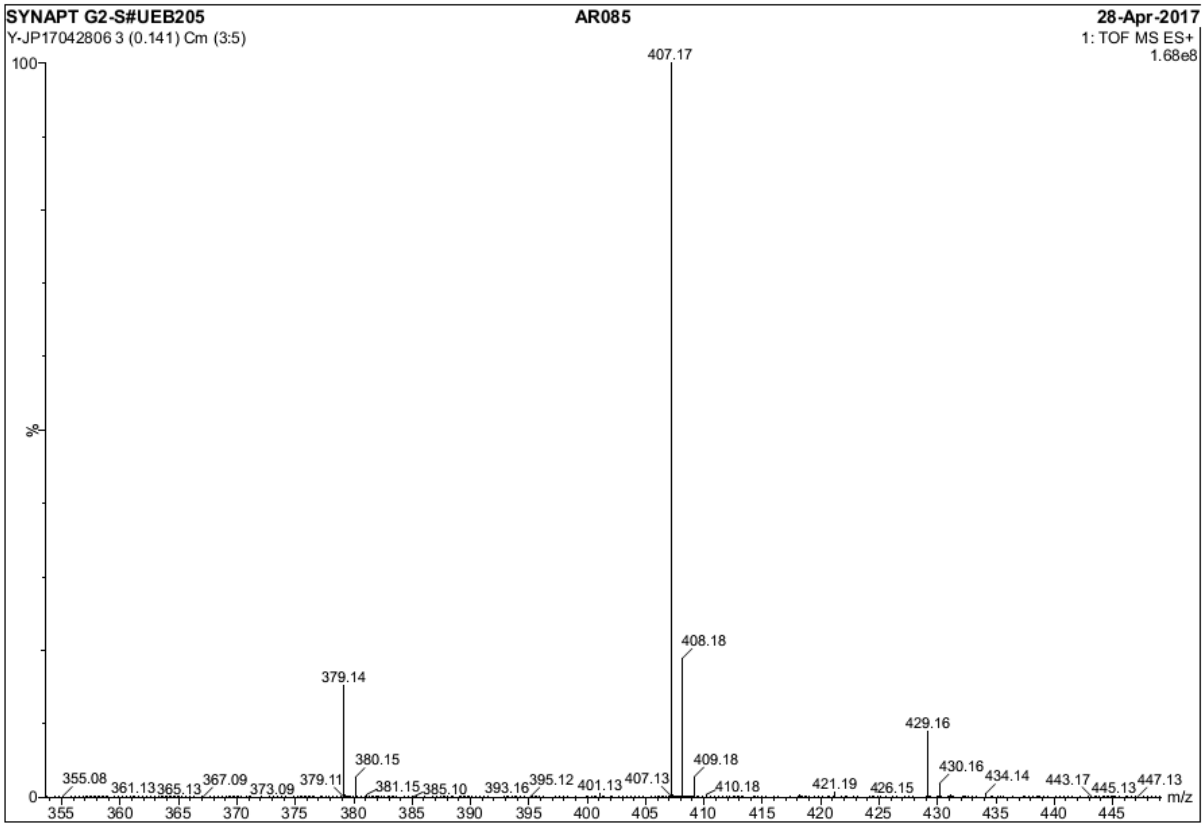
Yellow oil




AR085
7.26
7.18
7.16
7.05
7.03
4.17
4.15
4.13
4.13
4.12
4.10
4.08
3.24
3.21
3.20
3.17
2.30
1.44
1.40
1.36
1.29
1.28
1.26
1.25
1.23
1.21
2873.51
2865.49
2821.20
2813.38
1668.40
1661.48
1654.60
1653.33
1647.51
1640.43
1633.37
1296.84
1283.05
1281.22
1267.43
576.33
559.63
542.92
517.59
510.53
503.45
500.17
493.11
486.03
f1 (ppm)
1.91
1.95
8.00
2.01
2.97
3.03
11.97


AR085
136.11
133.10
133.02
132.94
131.51
128.19
77.48
77.16
76.84
62.72
62.68
62.65
62.61
42.38
41.05
37.26
37.22
37.18
21.11
16.51
16.47
16.43
16.40
16.37
16.33
16.22
16.16
16.11
13393.52
13385.23
13376.98
6315.65
6310.99
6307.67
6304.41
6300.56
6297.34
6294.06
6289.35
4397.33
4264.16
4130.98
3749.51
3745.29
3741.13
1661.31
1656.88
1653.69
1650.57
1647.38
1642.96
1632.22
1626.42
1620.60
f1 (ppm)


SYNAPT G2-S#UEB205
AR085
28-Apr-2017
Y-JP17042806 3 (0.141) Cm (3:5)
1: TOF MS ES+
1.68e8
407.17
408.18
379.14
429.16
351.11
295.05
430.16
98.98
177.05
195.06
497.22
629.23
674.26
807.28
835.32
914.28
932.26
1098.28
1210.41
1305.46
%
m/z


SYNAPT G2-S#UEB205
AR085
28-Apr-2017
Y-JP17042806 3 (0.141) Cm (3:5)
1: TOF MS ES+
1.68e8
407.17
408.18
379.14
429.16
380.15
409.18
355.08
361.13
365.13
367.09
373.09
379.11
381.15
385.10
393.16
395.12
401.13
407.13
410.18
421.19
426.15
430.16
434.14
443.17
445.13
447.13
%
m/z

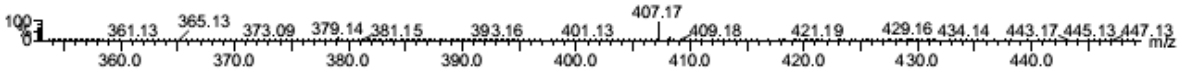
Elemental Composition Report

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

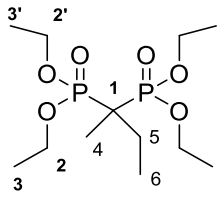
Monoisotopic Mass, Even Electron Ions
3015 formula(e) evaluated with 8 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 1-100 H: 0-100 N: 0-20 O: 0-20 P: 0-2

SYNAPT G2-S#UEB205 AR085 28-Apr-2017
Y-JP17042806 3 (0.141) Cm (3:5) 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, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl butane-2,2-diylbis(phosphonate) (7)

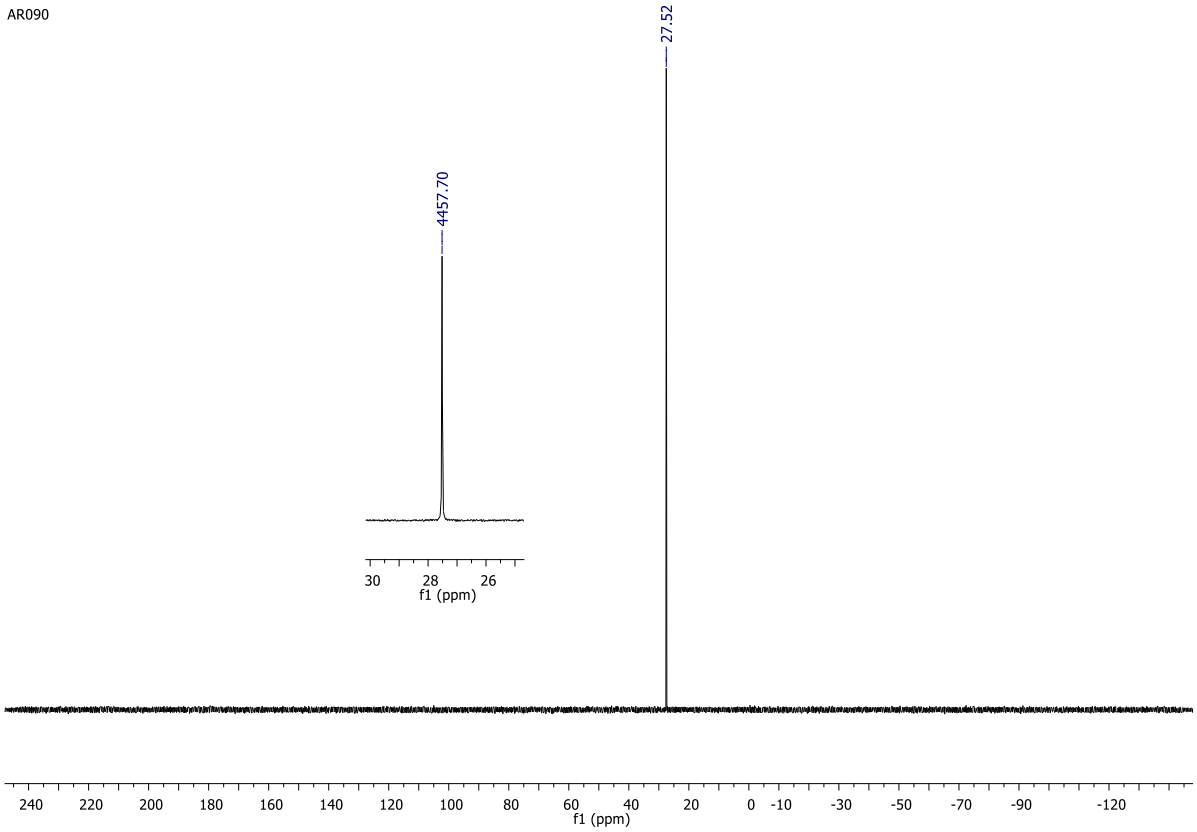


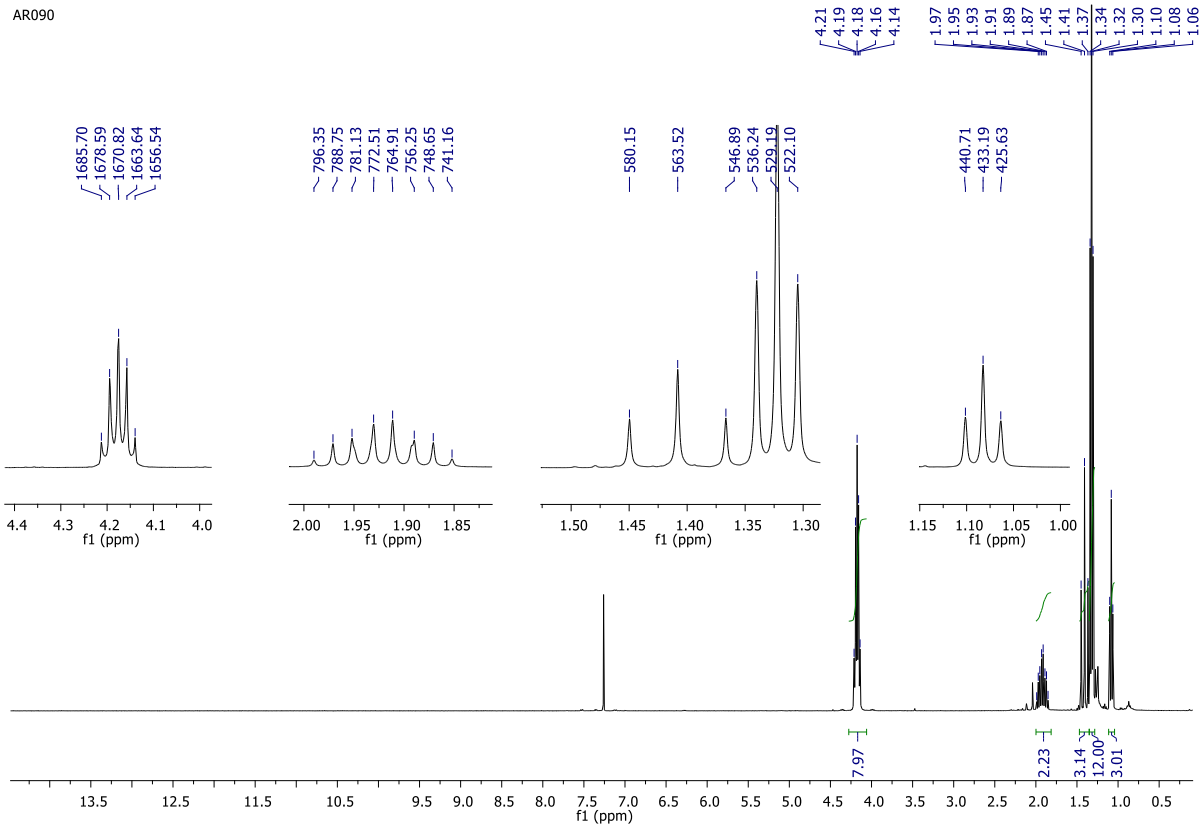
tetraethyl butane-2,2-diylbis(phosphonate)

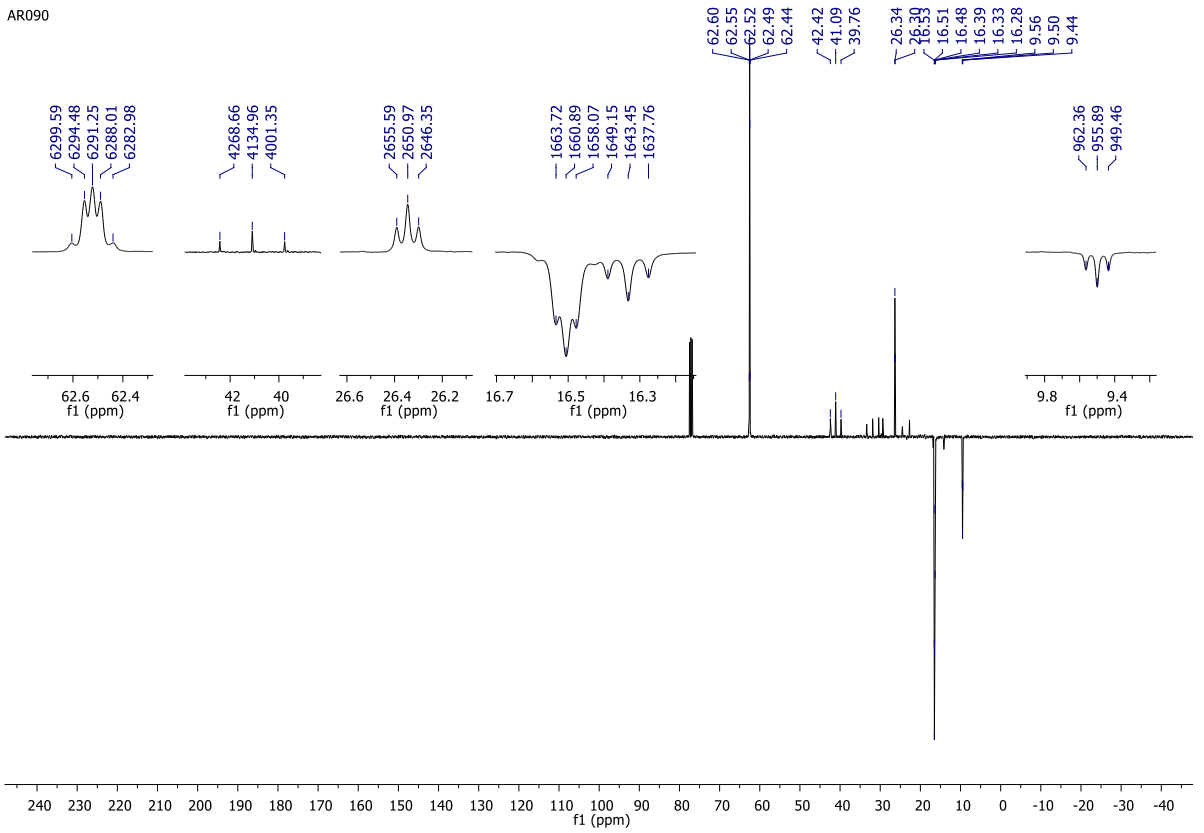
$C_{12}H_{28}O_6P_2$

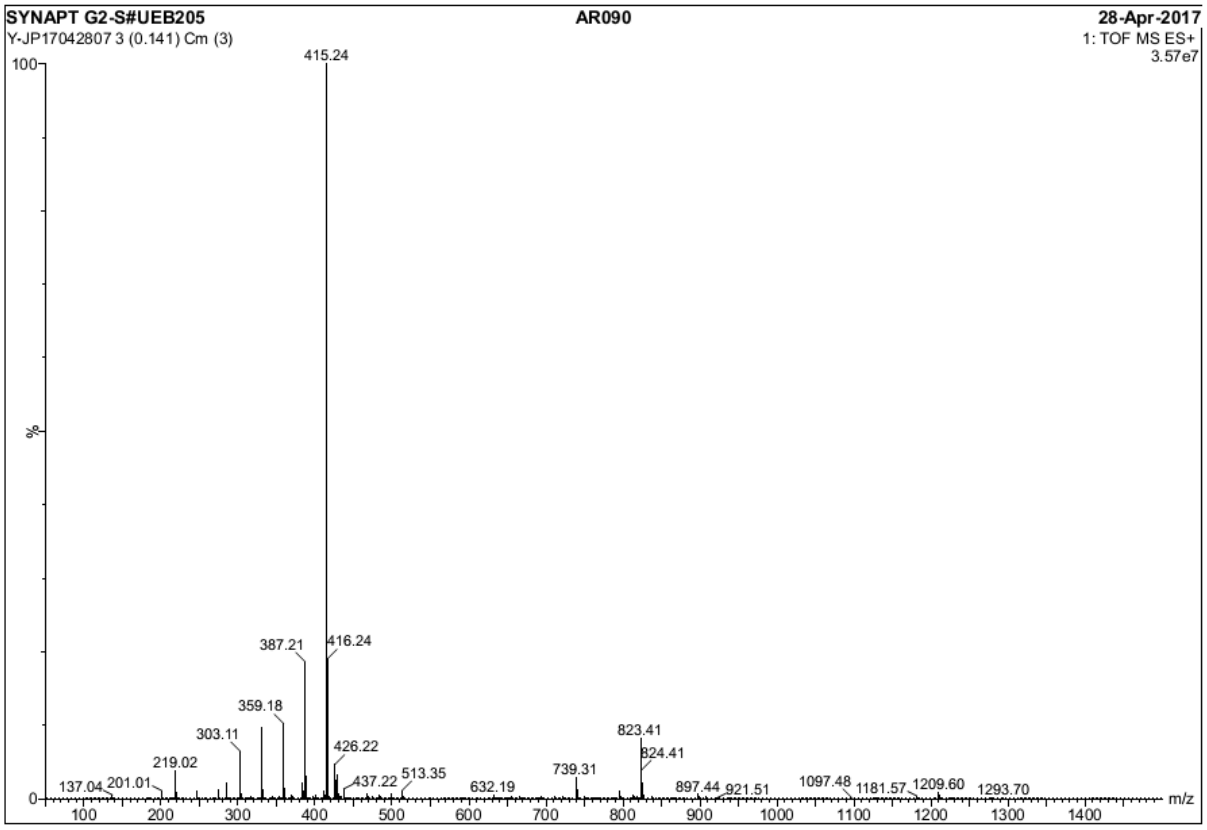
330,30 g/mol

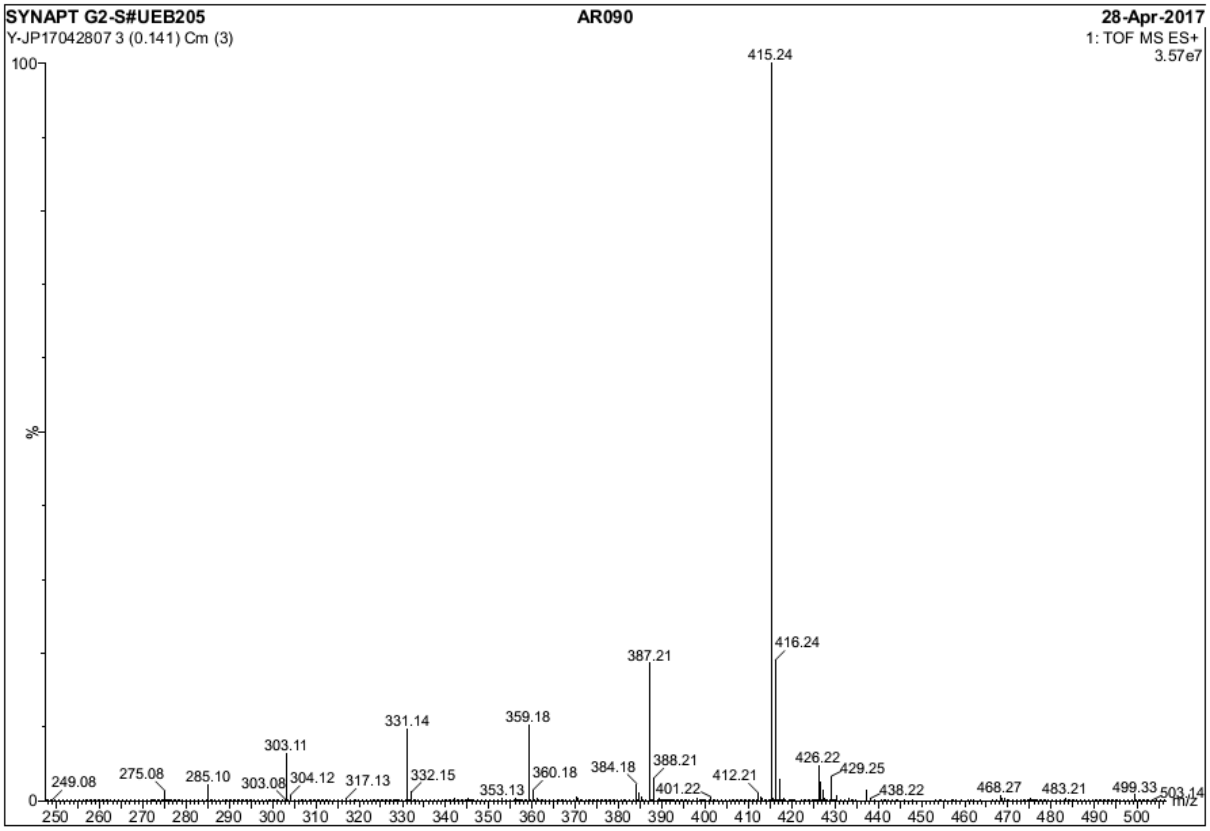
Colorless oil




AR090
4.21
4.19
4.18
4.16
4.14
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
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
f1 (ppm)
7.97
2.23
3.14
12.00
3.01


AR090
62.60
62.55
62.52
62.49
62.44
42.42
41.09
39.76
26.34
26.30
16.53
16.51
16.48
16.39
16.33
16.28
9.56
9.50
9.44
6299.59
6294.48
6291.25
6288.01
6282.98
4268.66
4134.96
4001.35
2655.59
2650.97
2646.35
1663.72
1660.89
1658.07
1649.15
1643.45
1637.76
962.36
955.89
949.46
f1 (ppm)


SYNAPT G2-S#UEB205
AR090
28-Apr-2017
Y-JP17042807 3 (0.141) Cm (3)
1: TOF MS ES+
3.57e7
100
%
0
415.24
387.21
416.24
359.18
303.11
823.41
824.41
426.22
219.02
739.31
137.04
201.01
437.22
513.35
632.19
897.44
921.51
1097.48
1181.57
1209.60
1293.70
m/z
100
200
300
400
500
600
700
800
900
1000
1100
1200
1300
1400


SYNAPT G2-S#UEB205
AR090
28-Apr-2017
Y-JP17042807 3 (0.141) Cm (3)
1: TOF MS ES+
3.57e7
100
%
0
415.24
416.24
387.21
331.14
359.18
303.11
426.22
429.25
388.21
384.18
360.18
412.21
249.08
275.08
285.10
303.08
304.12
317.13
332.15
353.13
401.22
438.22
468.27
483.21
499.33
503.14
m/z
250
260
270
280
290
300
310
320
330
340
350
360
370
380
390
400
410
420
430
440
450
460
470
480
490
500

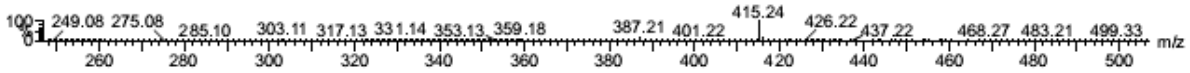
Elemental Composition Report

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

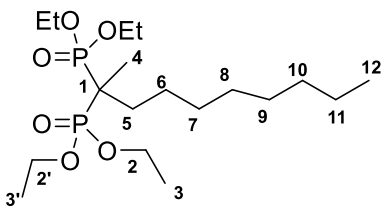
Monoisotopic Mass, Even Electron Ions
1781 formula(e) evaluated with 4 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 1-100 H: 0-100 N: 0-20 O: 0-20 P: 0-2

SYNAPT G2-S#UEB205 AR090 28-Apr-2017
Y-JP17042807 3 (0.141) Cm (3) 1: TOF MS ES+
3.57e+007



Minimum:				-1.5				
Maximum:		1.0	3.0	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, ^{1}H, and ^{13}C NMR spectra and mass analysis of tetraethyl decane-2,2-diylbis(phosphonate) (8)

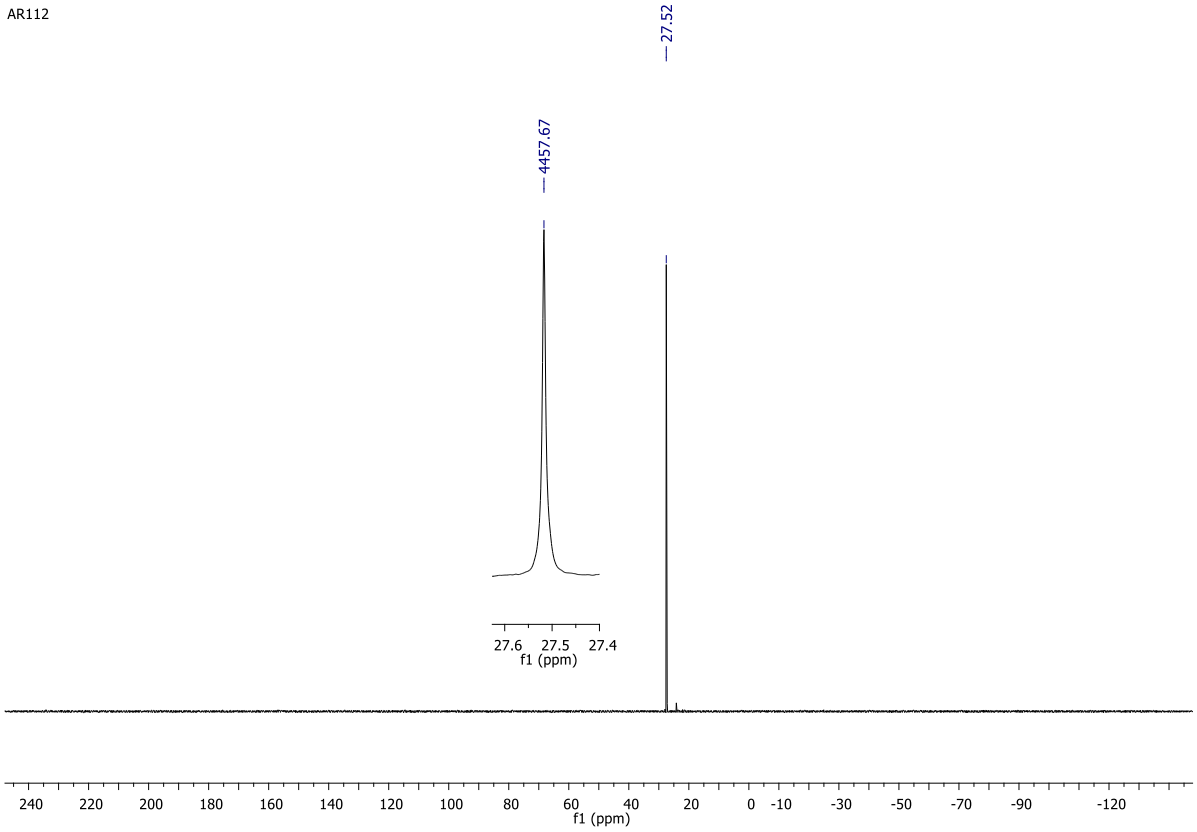


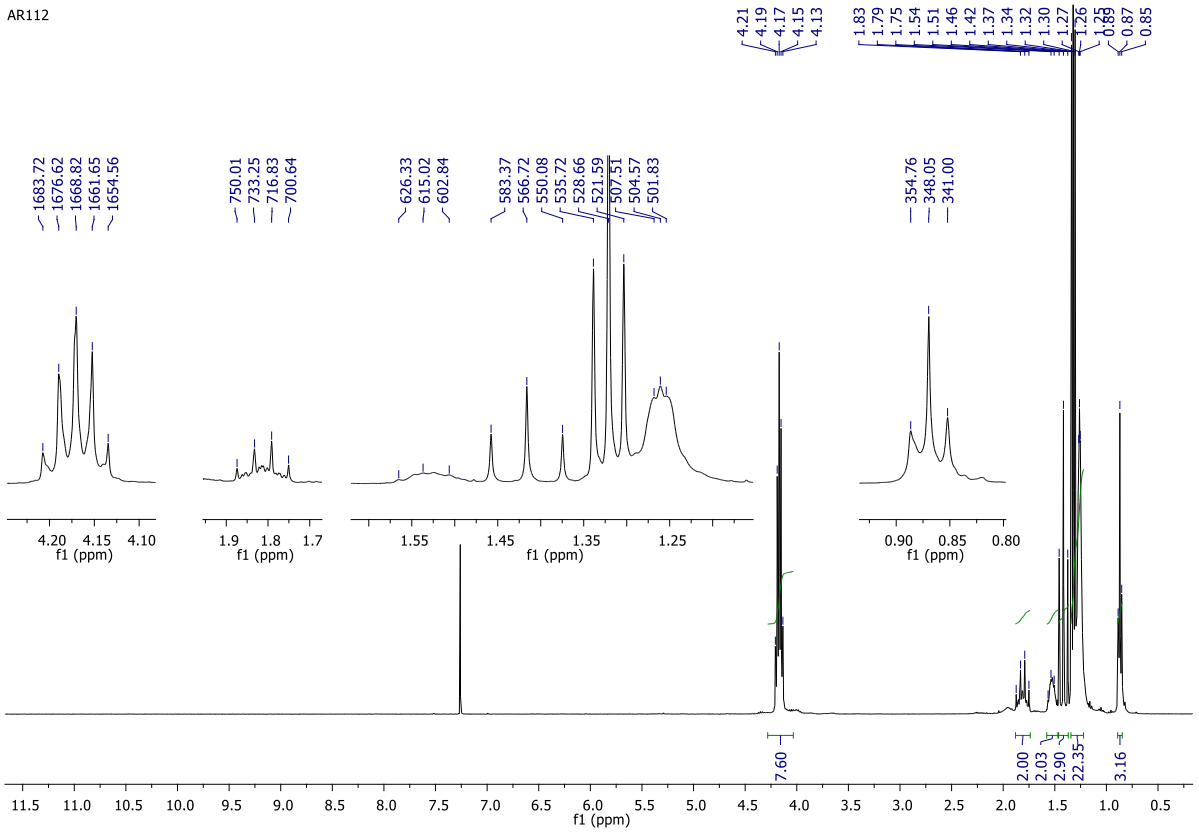
tetraethyl decane-2,2-diylbis(phosphonate)

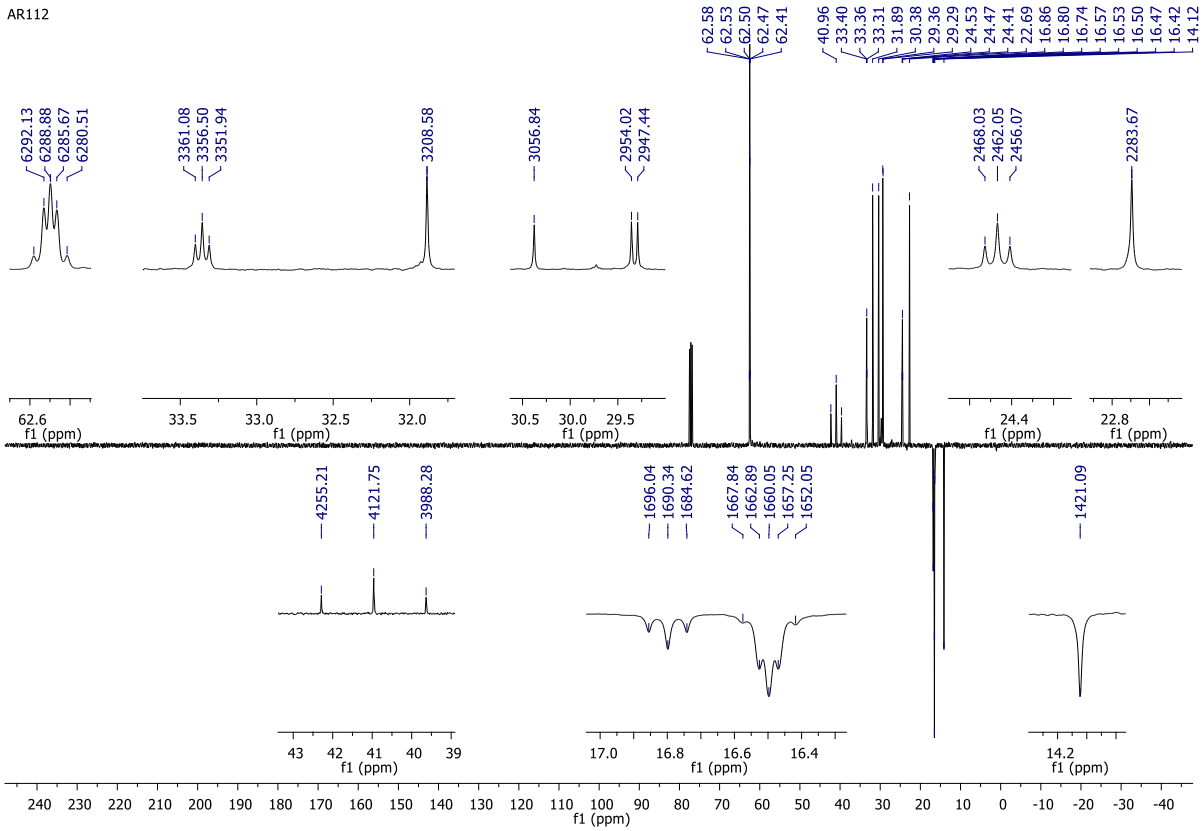
$C_{18}H_{40}O_6P_2$

414,46 g/mol

Yellow oil




AR112
4.21
4.19
4.17
4.15
4.13
1.83
1.79
1.75
1.54
1.51
1.46
1.42
1.37
1.34
1.32
1.30
1.27
1.26
1.25
0.89
0.87
0.85
1683.72
1676.62
1668.82
1661.65
1654.56
750.01
733.25
716.83
700.64
626.33
615.02
602.84
583.37
566.72
550.08
535.72
528.66
521.59
507.51
504.57
501.83
354.76
348.05
341.00
f1 (ppm)
7.60
2.00
2.03
2.90
22.35
3.16


AR112
62.58
62.53
62.50
62.47
62.41
40.96
33.40
33.36
33.31
31.89
30.38
29.36
29.29
24.53
24.47
24.41
22.69
16.86
16.80
16.74
16.57
16.53
16.50
16.47
16.42
14.12
6292.13
6288.88
6285.67
6280.51
3361.08
3356.50
3351.94
3208.58
3056.84
2954.02
2947.44
2468.03
2462.05
2456.07
2283.67
f1 (ppm)
4255.21
4121.75
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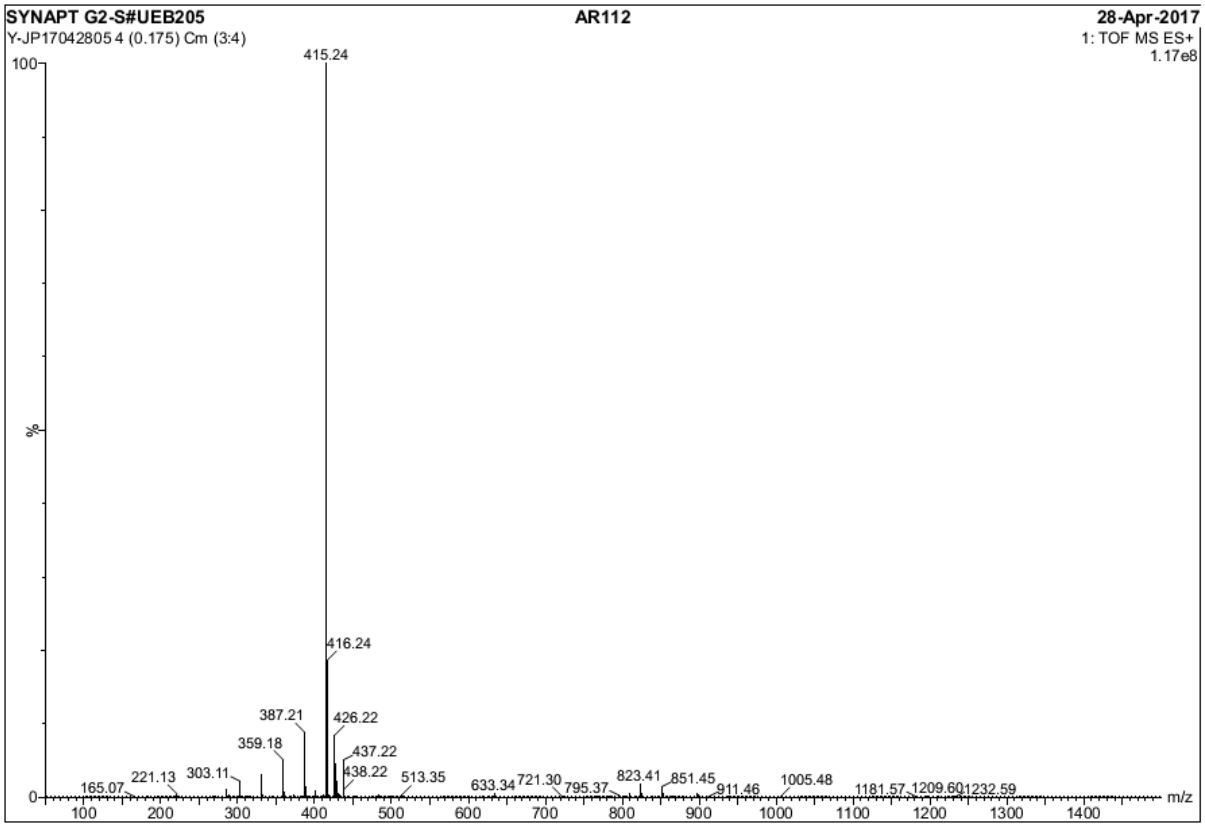
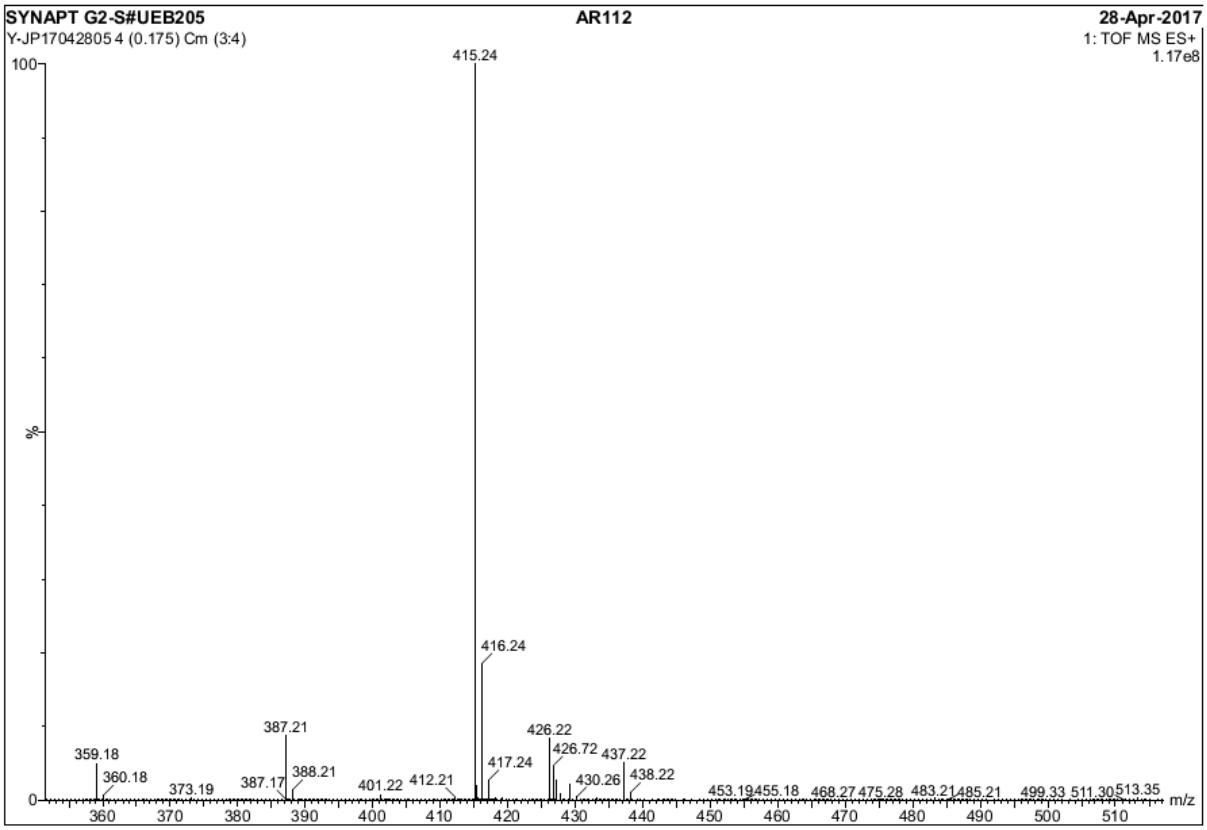
1690.34
1684.62
1667.84
1662.89
1660.05
1657.25
1652.05
1421.09


SYNAPT G2-S#UEB205
AR112
28-Apr-2017
Y-JP17042805 4 (0.175) Cm (3:4)
1: TOF MS ES+
1.17e8
415.24
416.24
387.21
426.22
359.18
437.22
438.22
513.35
165.07
221.13
303.11
633.34
721.30
795.37
823.41
851.45
911.46
1005.48
1181.57
1209.60
1232.59
%
m/z


SYNAPT G2-S#UEB205
AR112
28-Apr-2017
Y-JP17042805 4 (0.175) Cm (3:4)
1: TOF MS ES+
1.17e8
415.24
416.24
387.21
426.22
426.72
437.22
359.18
360.18
388.21
417.24
438.22
373.19
387.17
401.22
412.21
430.26
453.19
455.18
468.27
475.28
483.21
485.21
499.33
511.30
513.35
%
m/z

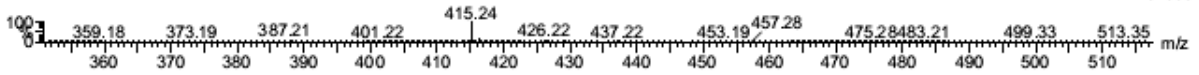
Elemental Composition Report

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

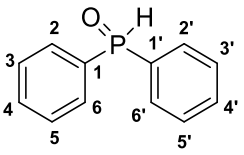
Monoisotopic Mass, Even Electron Ions
3153 formula(e) evaluated with 5 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 1-100 H: 0-100 N: 0-20 O: 0-20 P: 0-2

SYNAPT G2-S#UEB205 AR112 28-Apr-2017
Y-JP17042805 4 (0.175) Cm (3:4) 1: TOF MS ES+
1.17e+008



Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula
Minimum:				-1.5				
Maximum:		1.0	3.0	50.0				
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, ^{1}H and ^{13}C NMR spectra of diphenylphosphine oxide (10)

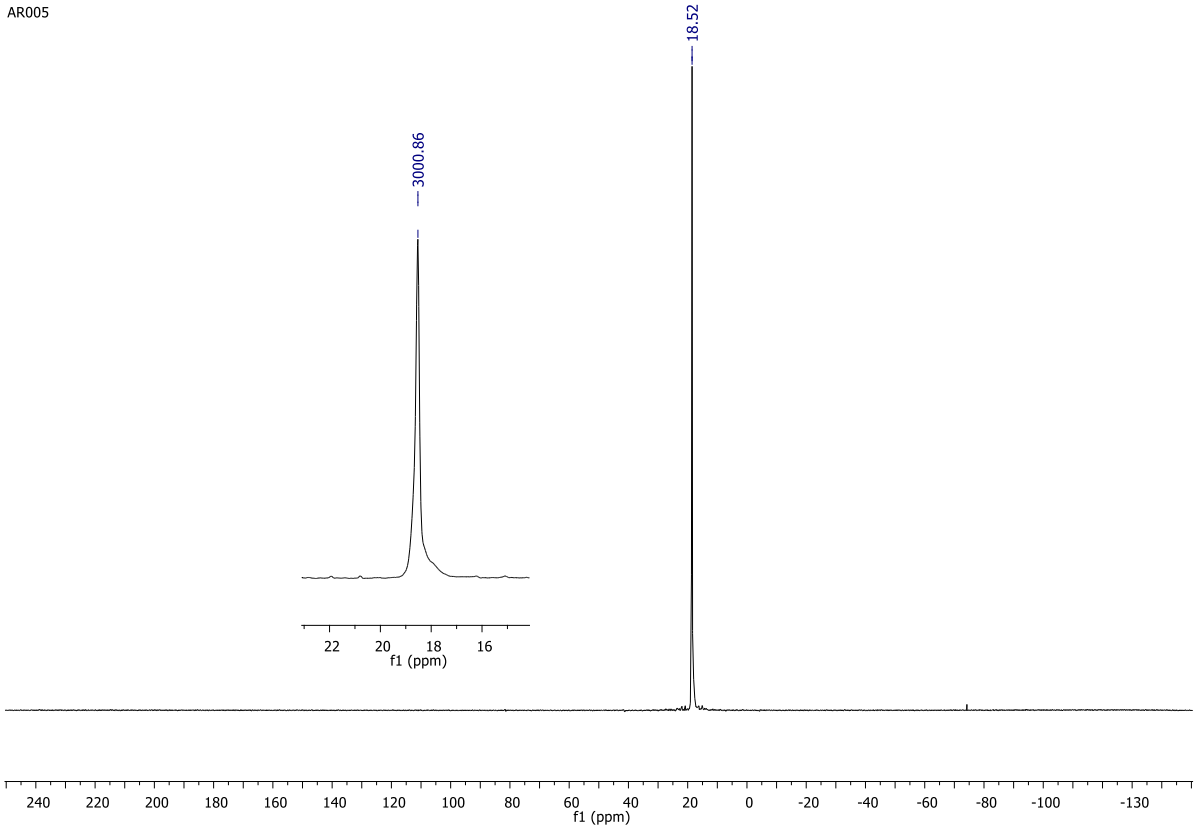


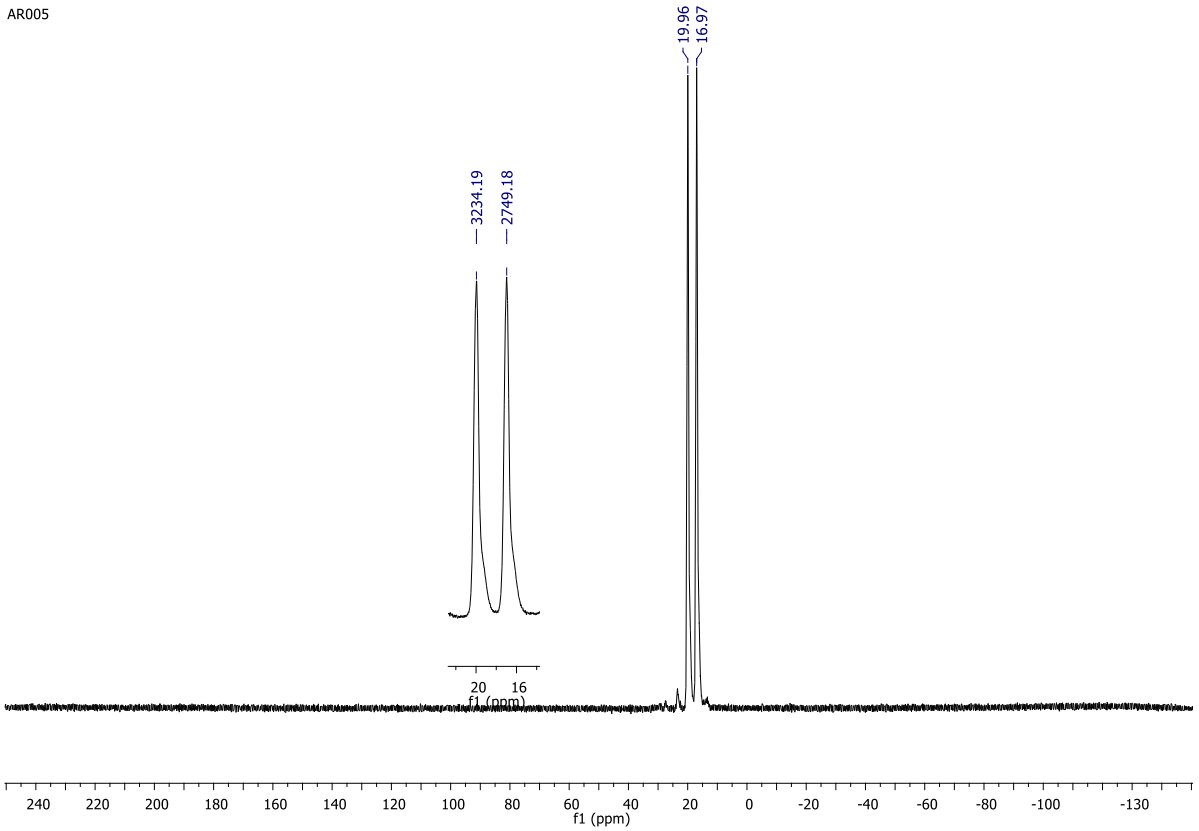
Diphenylphosphine oxide

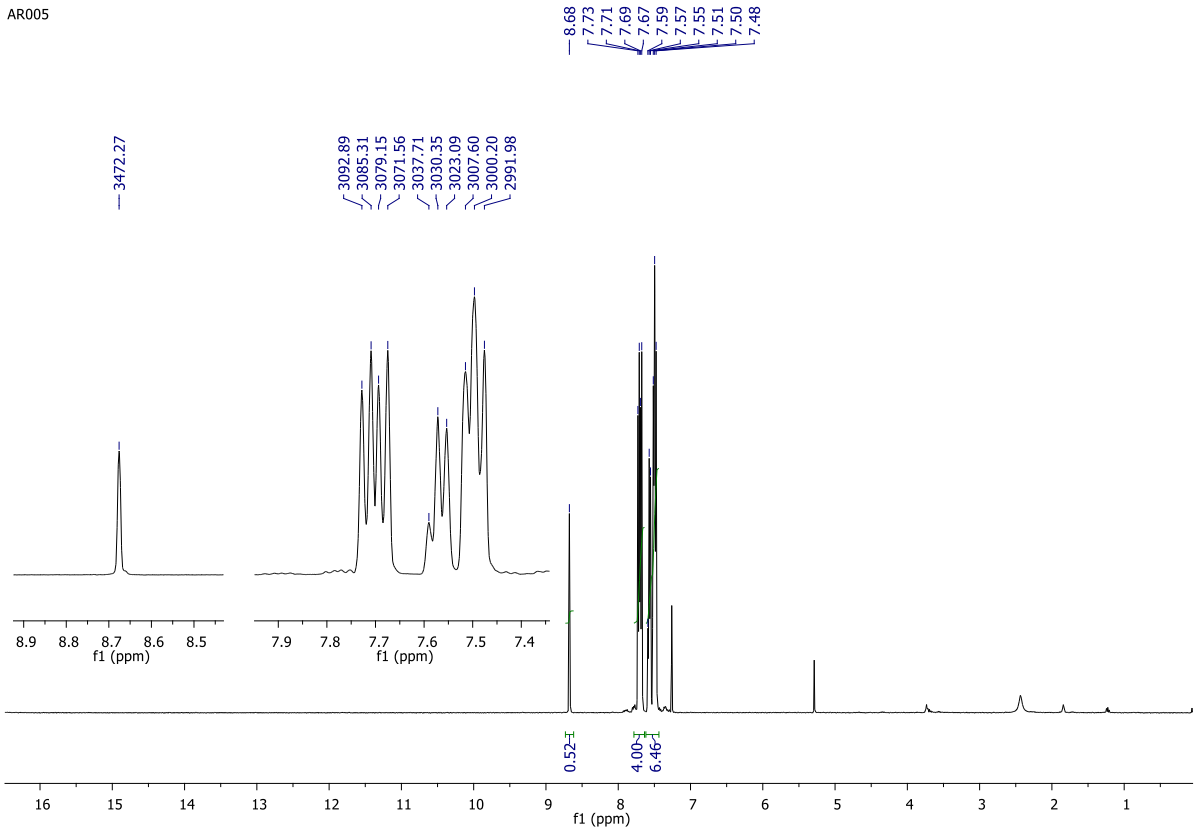
$C_{12}H_{11}OP$

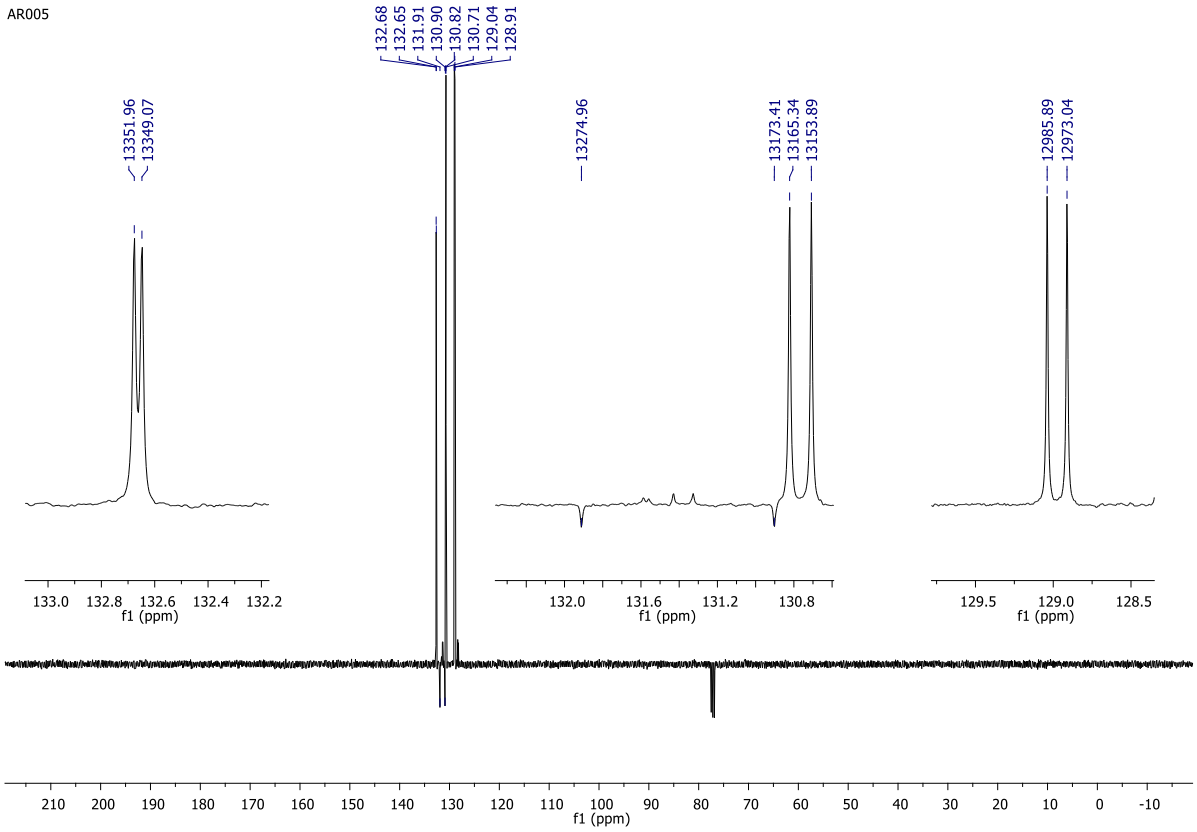
202,19 g/mol

White solid

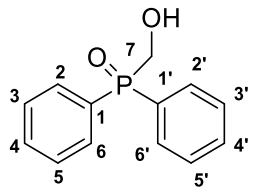



AR005
19.96
16.97
3234.19
2749.18
20
16
f1 (ppm)
240
220
200
180
160
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
f1 (ppm)

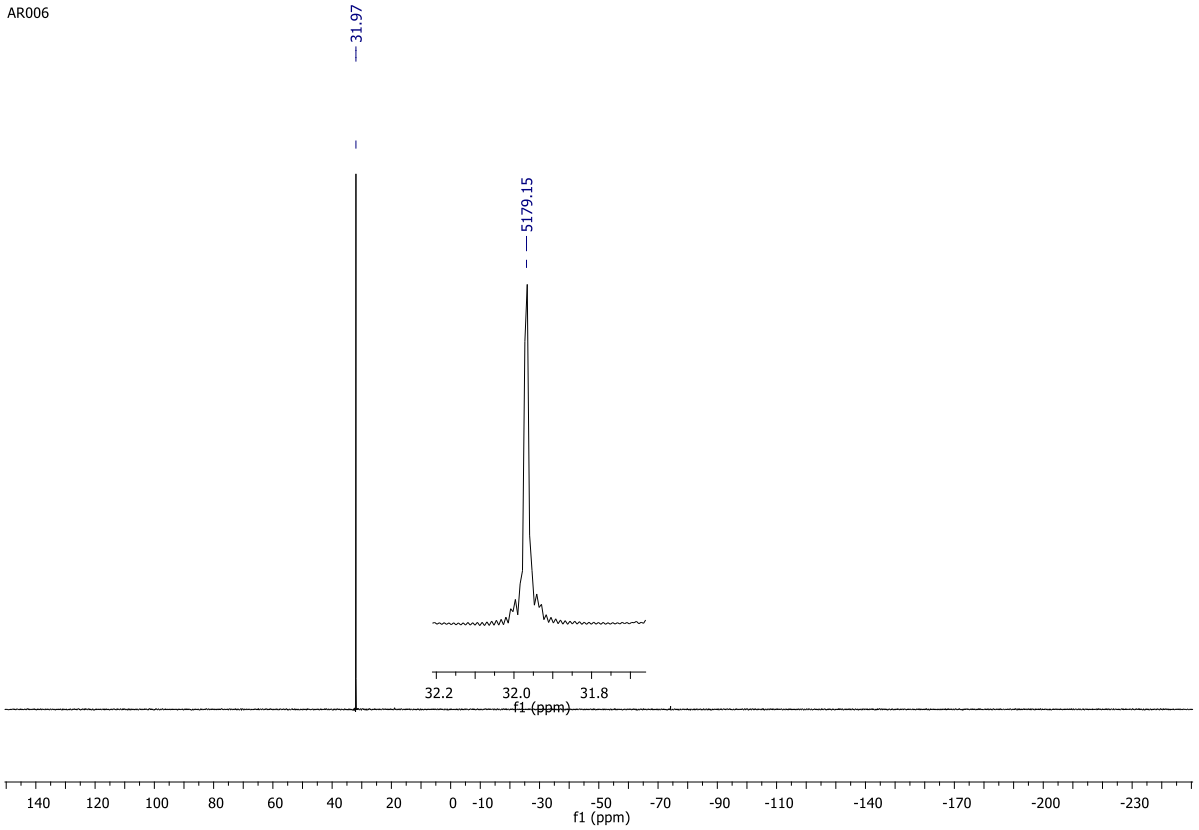

AR005
8.68
7.73
7.71
7.69
7.67
7.59
7.57
7.55
7.51
7.50
7.48
3472.27
3092.89
3085.31
3079.15
3071.56
3037.71
3030.35
3023.09
3007.60
3000.20
2991.98
8.9
8.8
8.7
8.6
8.5
f1 (ppm)
7.9
7.8
7.7
7.6
7.5
7.4
f1 (ppm)
0.52
4.00
6.46
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
f1 (ppm)

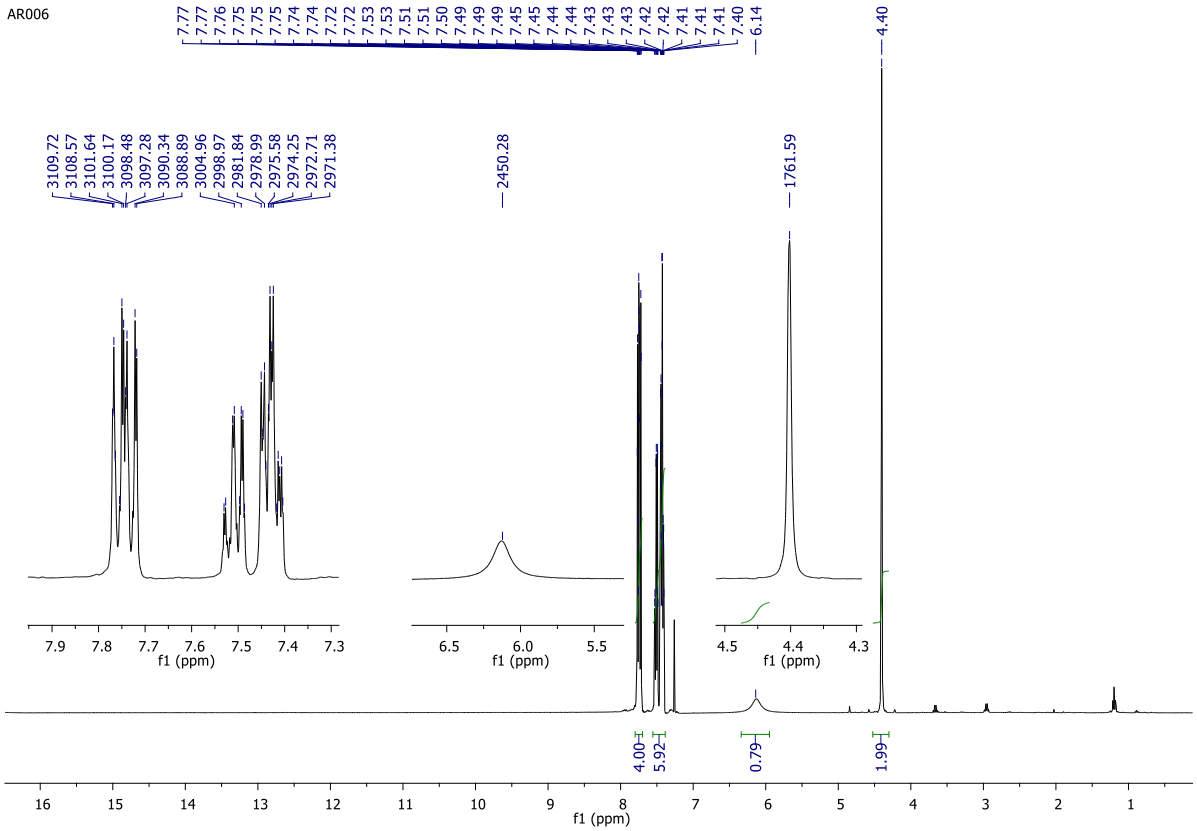

AR005
132.68
132.65
131.91
130.90
130.82
130.71
129.04
128.91
13351.96
13349.07
13274.96
13173.41
13165.34
13153.89
12985.89
12973.04
133.0 132.8 132.6 132.4 132.2
f1 (ppm)
132.0 131.6 131.2 130.8
f1 (ppm)
129.5 129.0 128.5
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

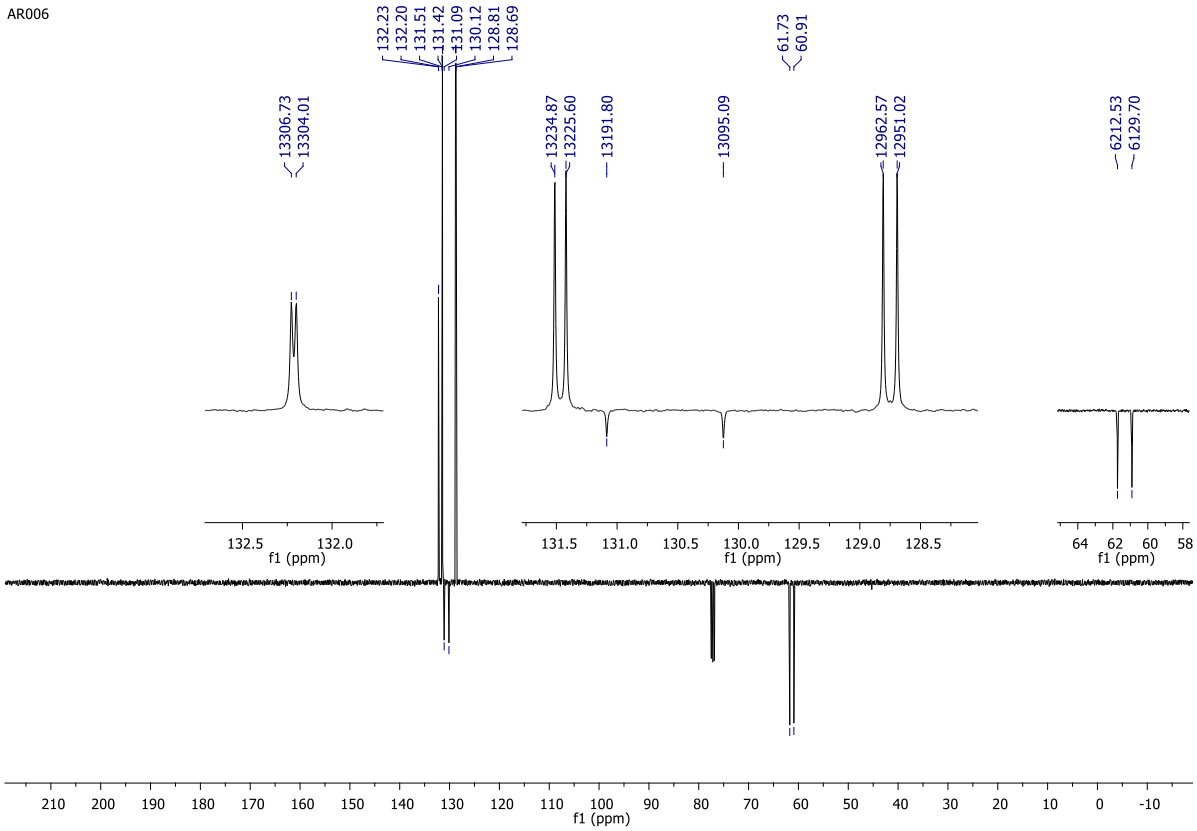
S17: ^{31}P, ^{1}H and ^{13}C NMR spectra of diphenylhydroxymethylphosphine oxide (11)



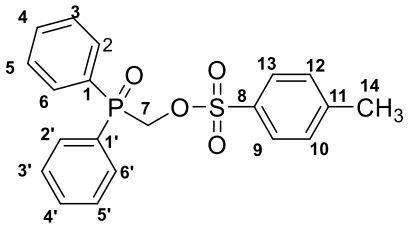
(hydroxymethyl)diphenylphosphine oxide
$C_{13}H_{13}O_2P$
232,22 g/mol
White solid



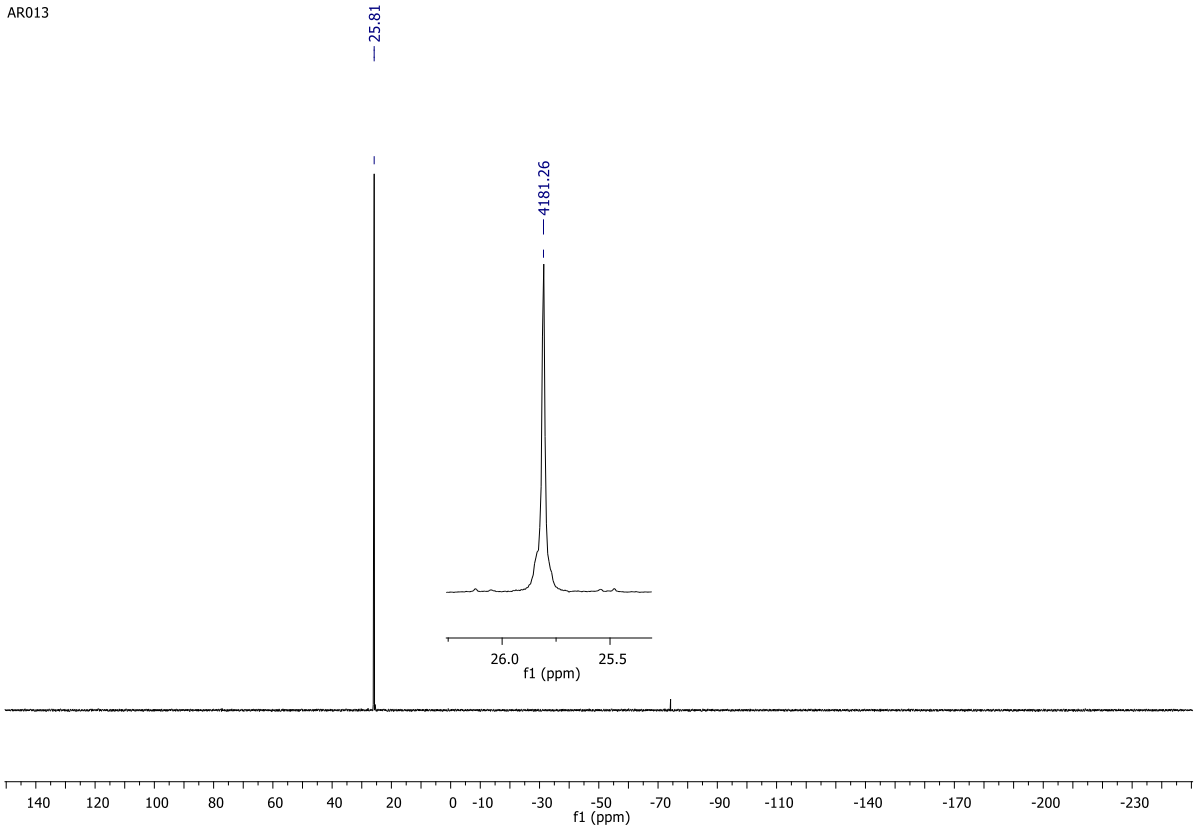

AR006
7.77 7.77 7.76 7.75 7.75 7.75 7.74 7.74 7.72 7.53 7.53 7.51 7.51 7.50 7.49 7.49 7.49 7.45 7.45 7.44 7.44 7.43 7.43 7.43 7.42 7.42 7.41 7.41 7.41 7.40 6.14
4.40
3109.72 3108.57 3101.64 3100.17 3098.48 3097.28 3090.34 3088.89 3004.96 2998.97 2981.84 2978.99 2975.58 2974.25 2972.71 2971.38
2450.28
1761.59
f1 (ppm)
4.00 5.92 0.79 1.99

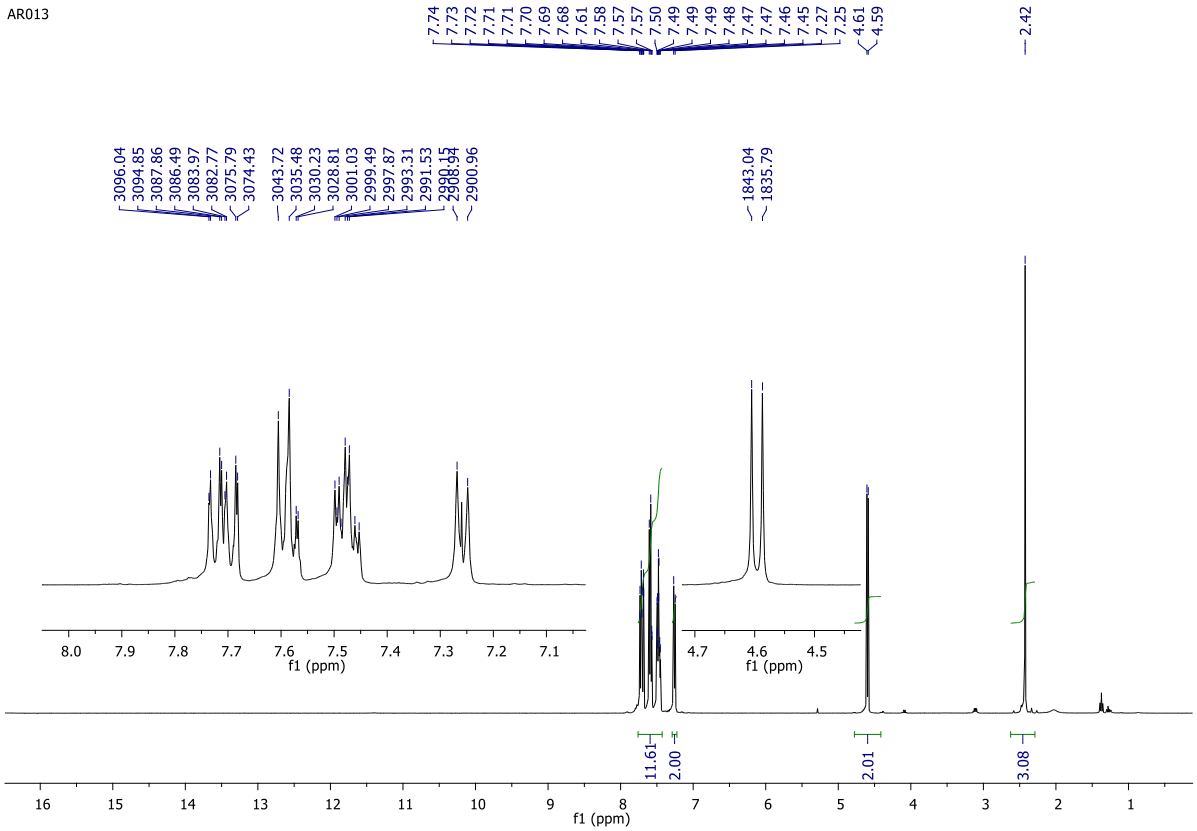

AR006
132.23 132.20 131.51 131.42 131.09 130.12 128.81 128.69
61.73 60.91
13306.73 13304.01
13234.87 13225.60
13191.80
13095.09
12962.57 12951.02
6212.53 6129.70
f1 (ppm)

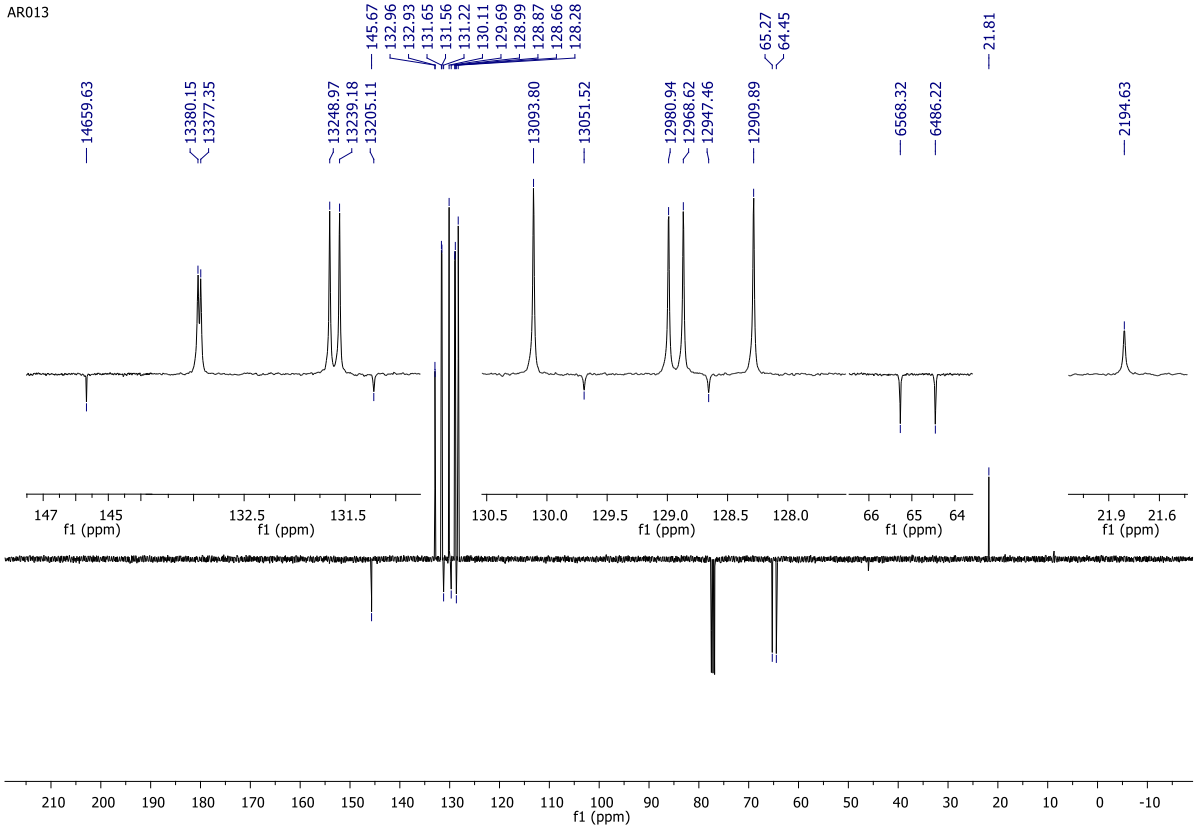
S18: ^{31}P, ^{1}H and ^{13}C NMR spectra of (diphenylphosphoryl)methyl 4-methylbenzenesulfonate (12)



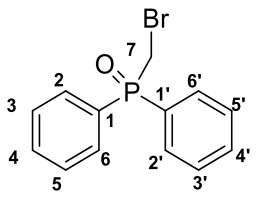
(diphenylphosphoryl)methyl 4-methylbenzenesulfonate
$C_{20}H_{19}O_4SP$
386,40 g/mol
White solid




AR013
7.74
7.73
7.72
7.71
7.71
7.70
7.69
7.68
7.61
7.58
7.57
7.57
7.50
7.49
7.49
7.49
7.48
7.47
7.47
7.46
7.45
7.27
7.25
4.61
4.59
2.42
3096.04
3094.85
3087.86
3086.49
3083.97
3082.77
3075.79
3074.43
3043.72
3035.48
3030.23
3028.81
3001.03
2999.49
2997.87
2993.31
2991.53
2900.96
1843.04
1835.79
f1 (ppm)
11.61
2.00
2.01
3.08


AR013
145.67
132.96
132.93
131.65
131.56
131.22
130.11
129.69
128.99
128.87
128.66
128.28
65.27
64.45
21.81
14659.63
13380.15
13377.35
13248.97
13239.18
13205.11
13093.80
13051.52
12980.94
12968.62
12947.46
12909.89
6568.32
6486.22
2194.63
f1 (ppm)

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

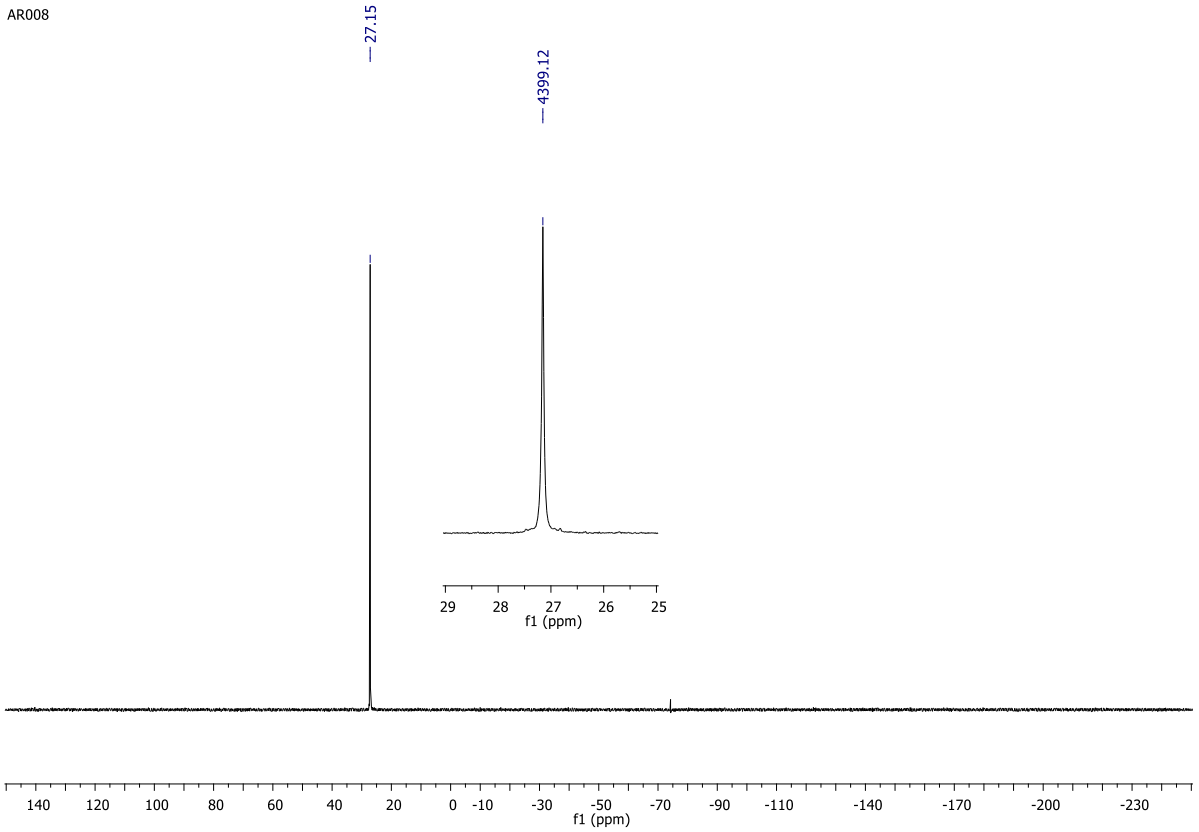


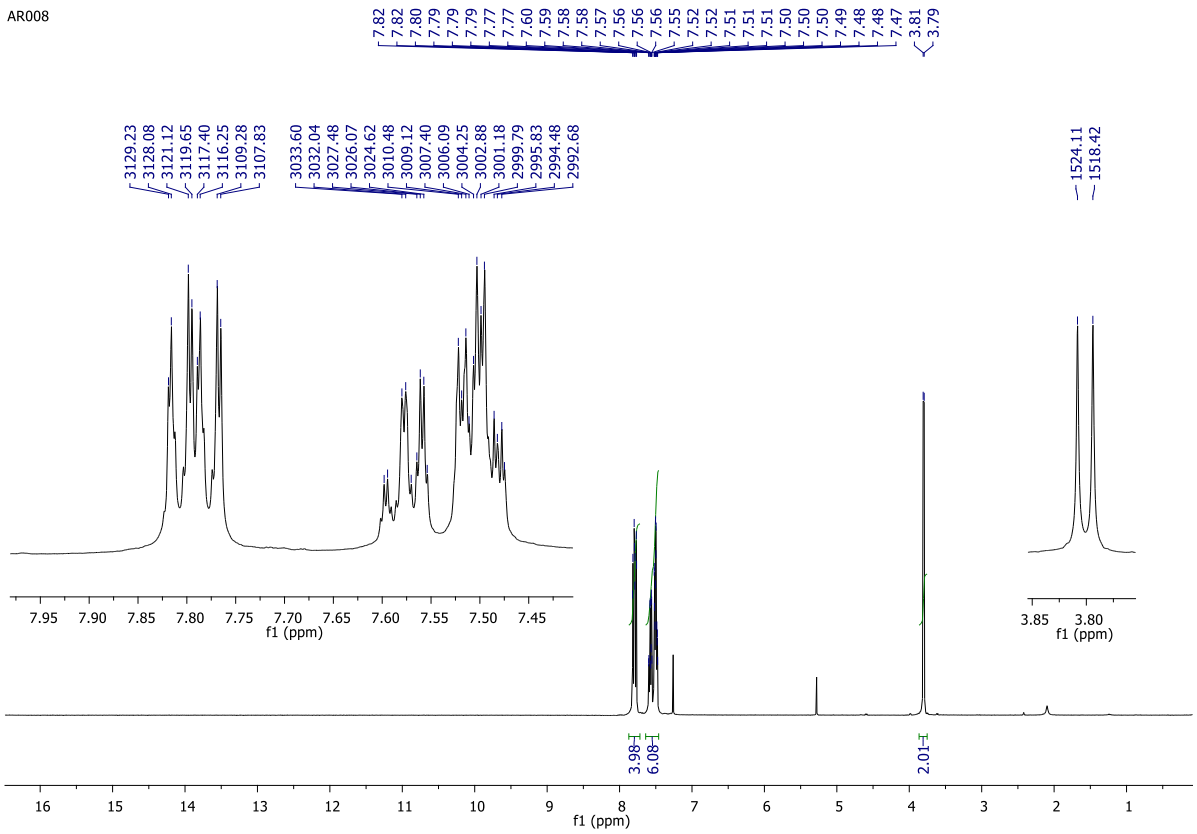
(bromomethyl)diphenylphosphine oxide

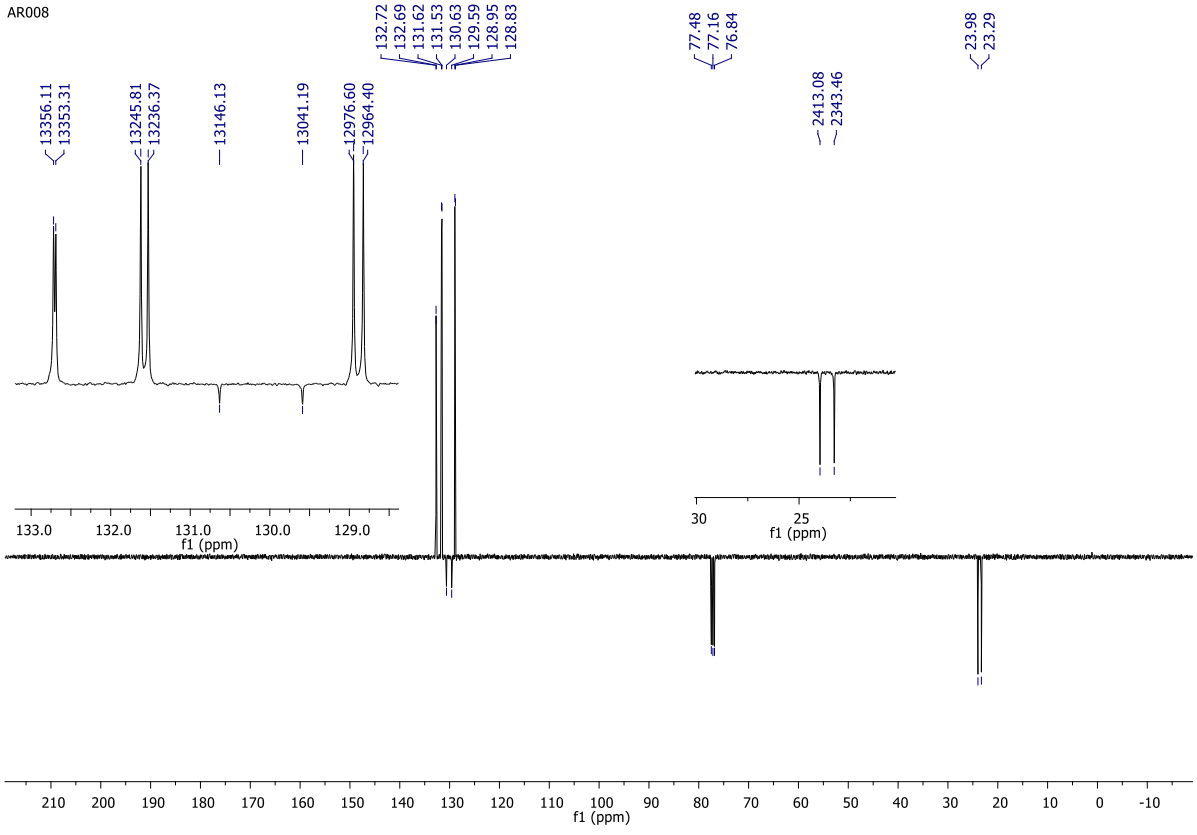
$C_{13}H_{12}OBrP$

295,12 g/mol

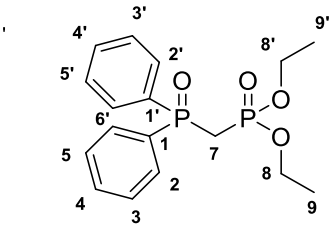
White solid




AR008
7.82 7.82 7.80 7.79 7.79 7.79 7.77 7.77 7.60 7.59 7.58 7.58 7.57 7.56 7.56 7.56 7.55 7.52 7.52 7.51 7.51 7.51 7.50 7.50 7.50 7.49 7.48 7.48 7.47 3.81 3.79
3129.23 3128.08 3121.12 3119.65 3117.40 3116.25 3109.28 3107.83
3033.60 3032.04 3027.48 3026.07 3024.62 3010.48 3009.12 3007.40 3006.09 3004.25 3002.88 3001.18 2999.79 2995.83 2994.48 2992.68
1524.11 1518.42
7.95 7.90 7.85 7.80 7.75 7.70 7.65 7.60 7.55 7.50 7.45
f1 (ppm)
3.85 3.80
f1 (ppm)
3.98 6.08 2.01
16 15 14 13 12 11 10 9 8 7 6 5 4 3 2 1
f1 (ppm)


AR008
132.72 132.69 131.62 131.53 130.63 129.59 128.95 128.83
77.48 77.16 76.84
23.98 23.29
13356.11 13353.31 13245.81 13236.37 13146.13 13041.19 12976.60 12964.40
2413.08 2343.46
133.0 132.0 131.0 130.0 129.0
f1 (ppm)
30 25
f1 (ppm)
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

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

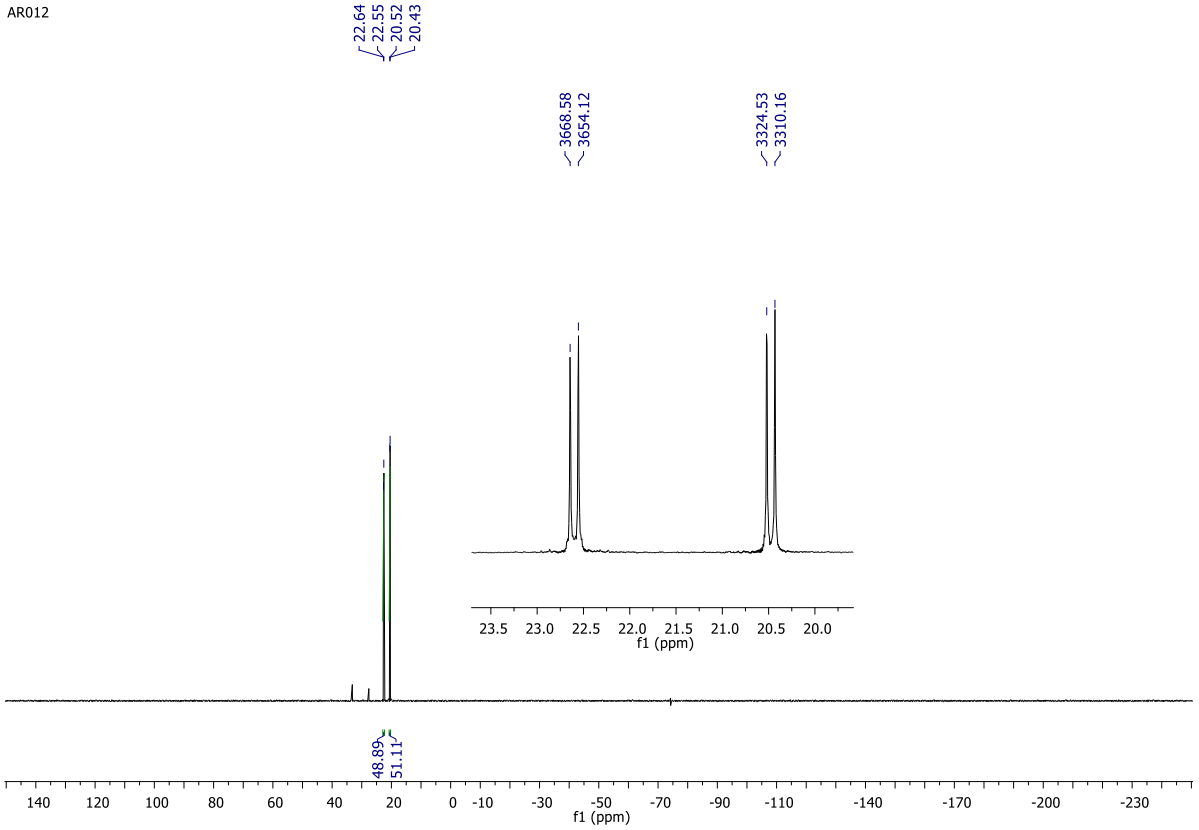


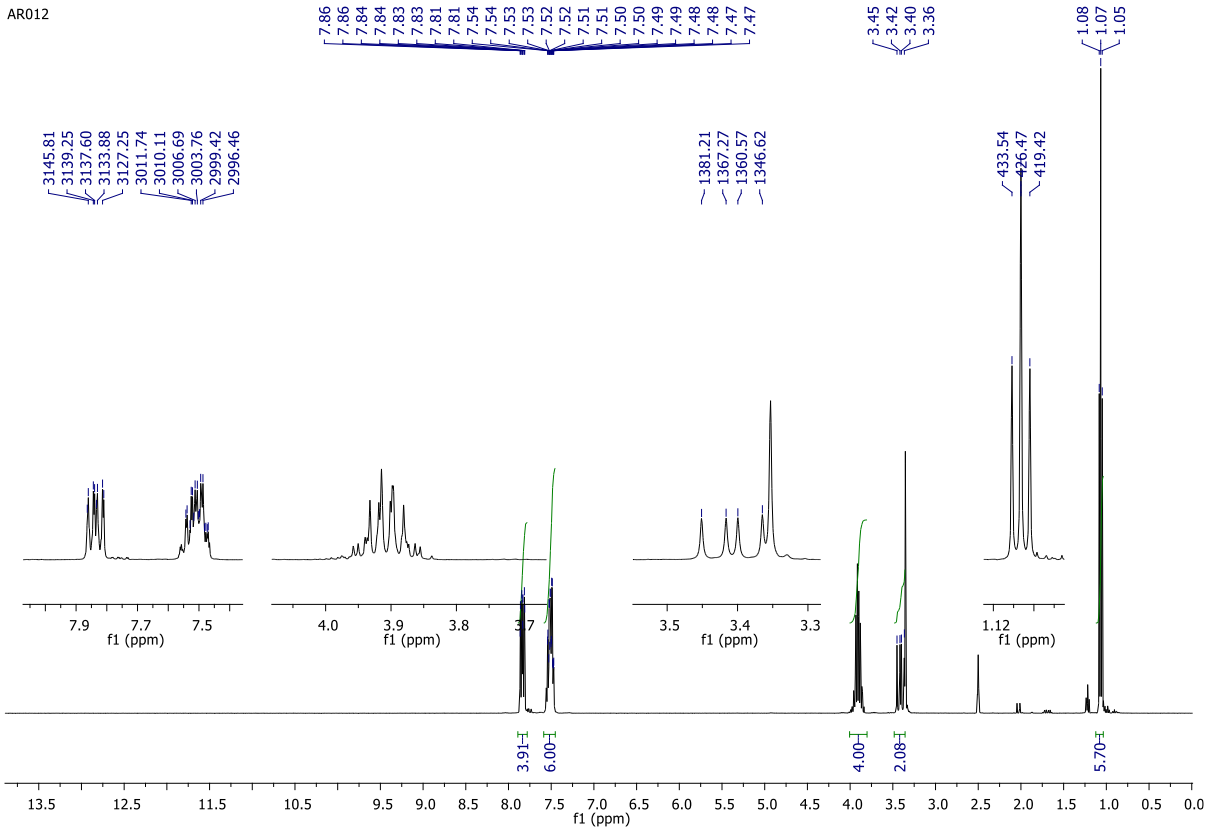
diethyl ((diphenylphosphoryl)methyl)phosphonate

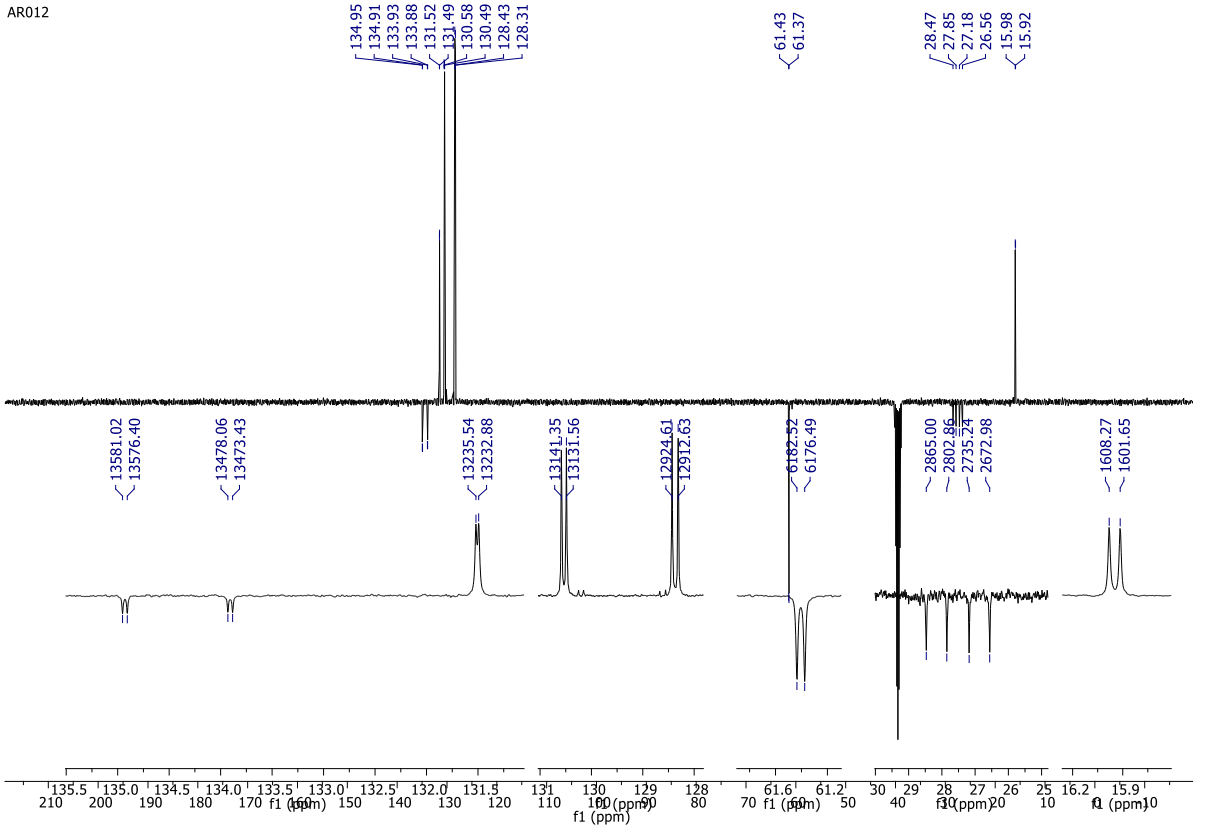
$C_{17}H_{22}O_4P_2$

352,31 g/mol

White solid

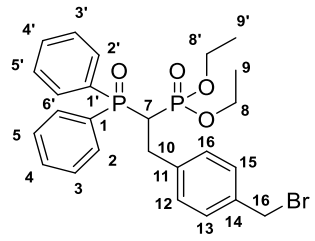



AR012
7.86 7.86 7.84 7.84 7.83 7.83 7.81 7.81 7.54 7.54 7.53 7.53 7.52 7.52 7.51 7.51 7.50 7.50 7.49 7.49 7.48 7.48 7.47 7.47
3.45 3.42 3.40 3.36
1.08 1.07 1.05
3145.81 3139.25 3137.60 3133.88 3127.25 3011.74 3010.11 3006.69 3003.76 2999.42 2996.46
1381.21 1367.27 1360.57 1346.62
433.54 426.47 419.42
3.91 6.00 4.00 2.08 5.70
f1 (ppm)

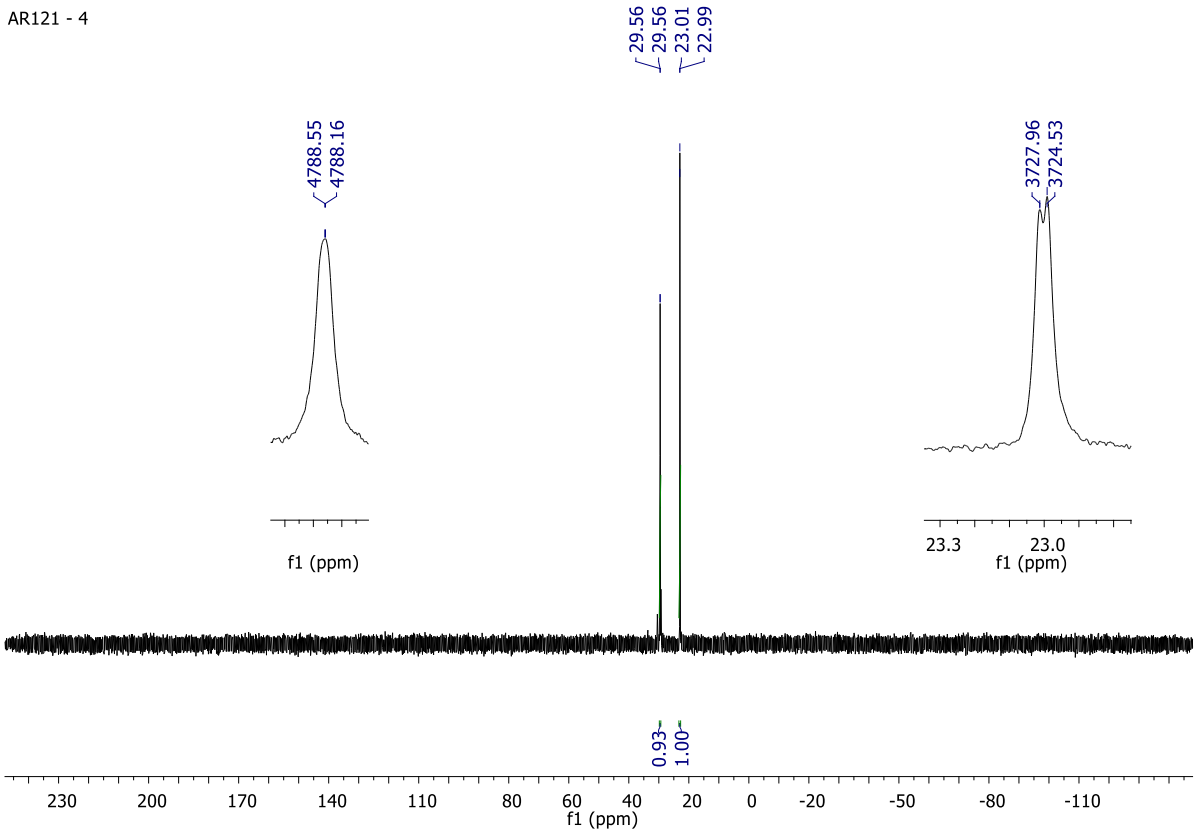

AR012
134.95 134.91 133.93 133.88 131.52 131.49 130.58 130.49 128.43 128.31
61.43 61.37
28.47 27.85 27.18 26.56 15.98 15.92
13581.02 13576.40 13478.06 13473.43 13235.54 13232.88 13141.35 13131.56 12924.61 12912.63 6182.52 6176.49 2865.00 2802.86 2735.24 2672.98 1608.27 1601.65
f1 (ppm)

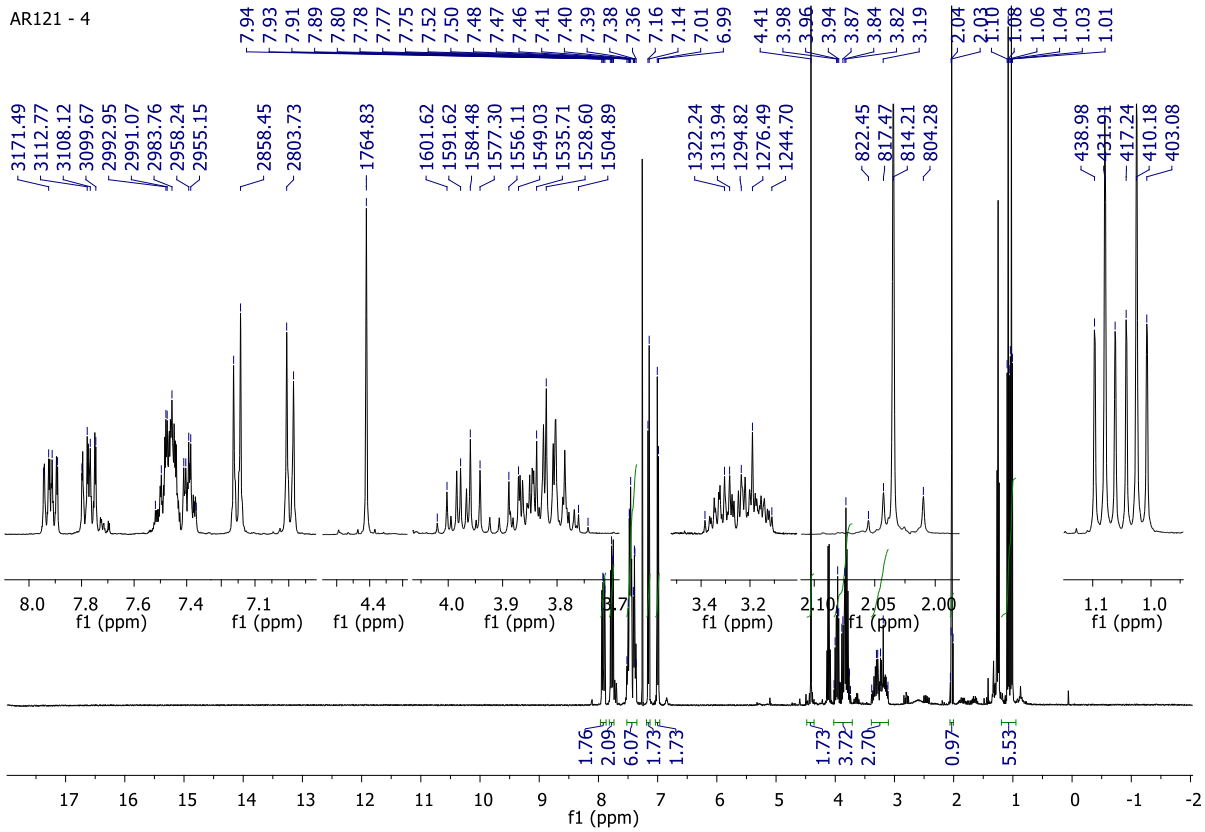
S21: ^{31}P, ^{1}H and ^{13}C NMR spectra of diethyl (2-(4-(bromomethyl)phenyl)-1-(diphenylphosphoryl)ethyl)phosphonate (15a)

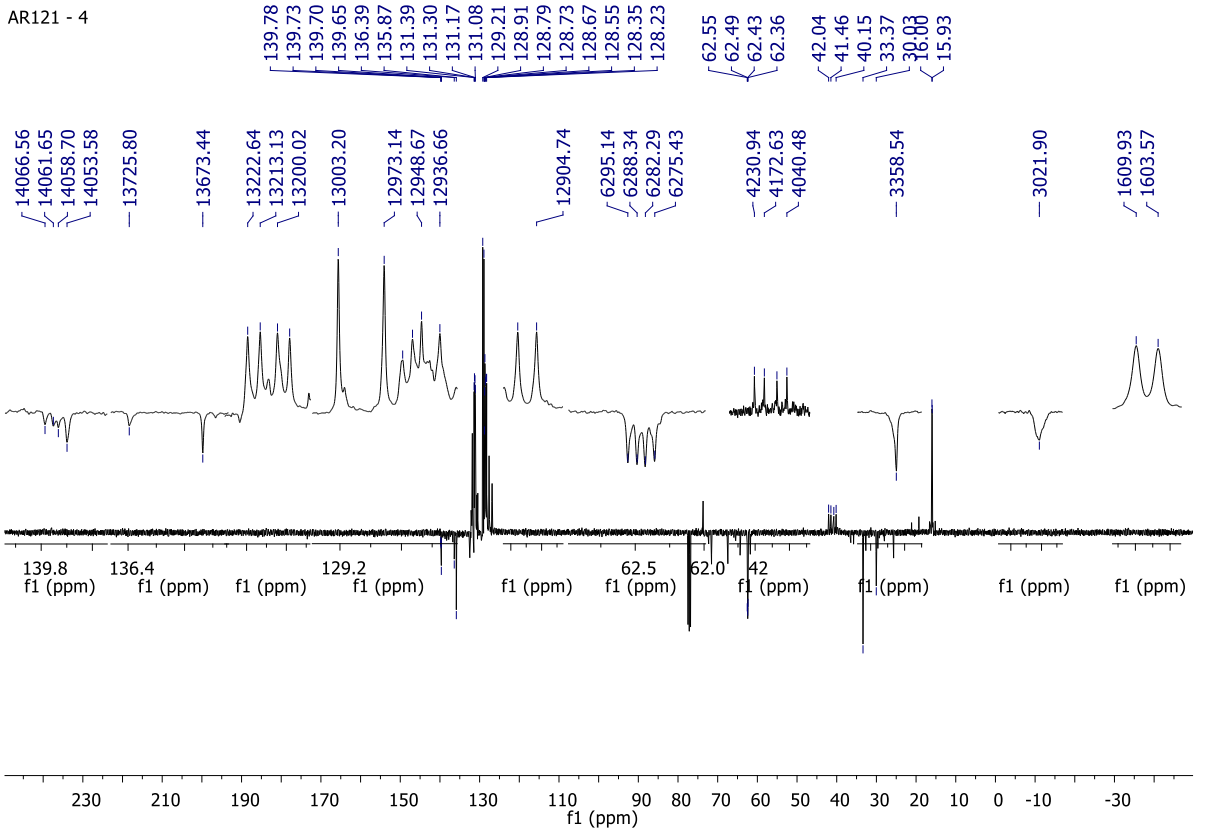
diethyl (2-(4-(bromomethyl)phenyl)-1-(diphenylphosphoryl)ethyl)phosphonate



$C_{25}H_{29}O_4BrP_2$
535,35 g/mol
White solid

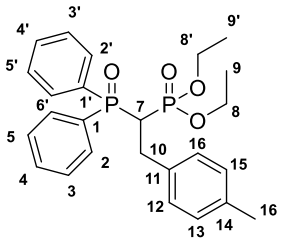



AR121 - 4
7.94 7.93 7.91 7.89 7.80 7.78 7.77 7.75 7.52 7.50 7.48 7.47 7.46 7.41 7.40 7.39 7.38 7.36 7.16 7.14 7.01 6.99
4.41 3.98 3.96 3.94 3.87 3.84 3.82 3.19
2.04 2.03 2.01 1.08 1.06 1.04 1.03 1.01
3171.49 3112.77 3108.12 3099.67 2992.95 2991.07 2983.76 2958.24 2955.15
2858.45 2803.73
1764.83
1601.62 1591.62 1584.48 1577.30 1556.11 1549.03 1535.71 1528.60 1504.89
1322.24 1313.94 1294.82 1276.49 1244.70
822.45 817.47 814.21 804.28
438.98 431.91 417.24 410.18 403.08
f1 (ppm)
1.76 2.09 6.07 1.73 1.73 1.73 3.72 2.70 0.97 5.53

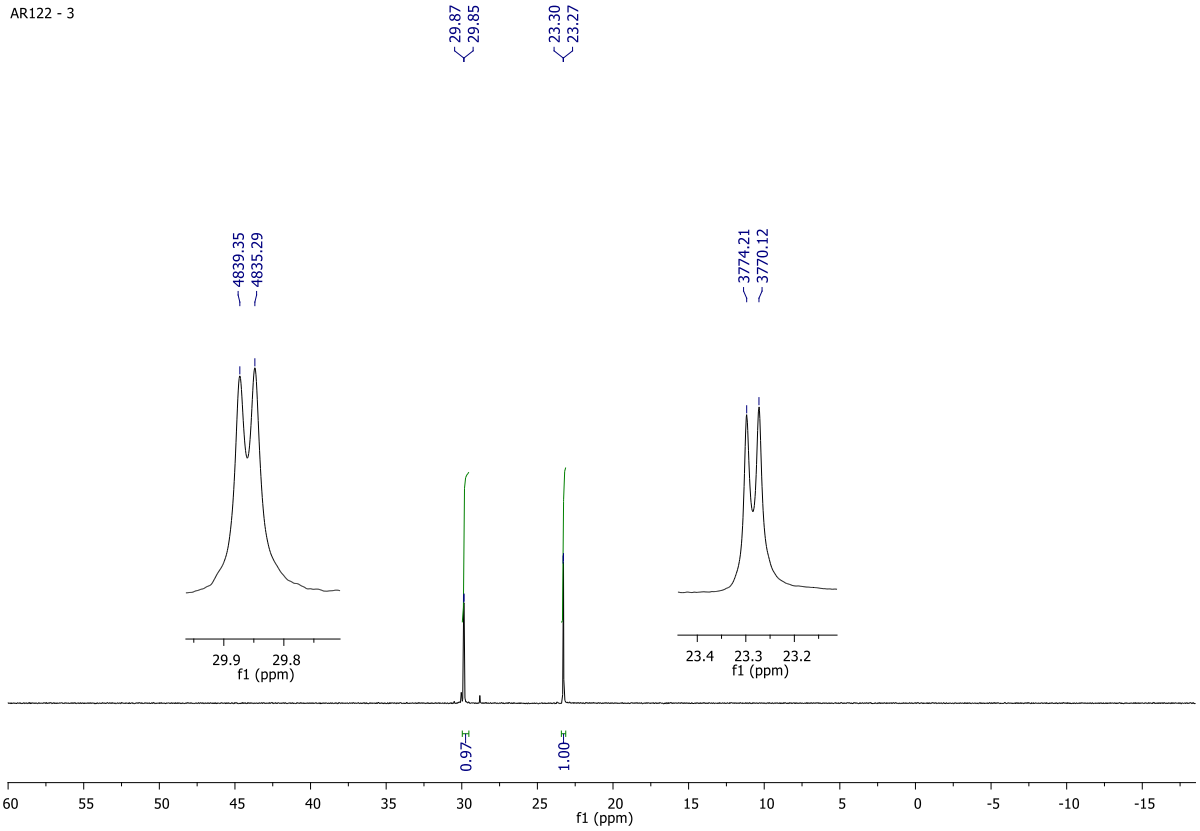

AR121 - 4
139.78 139.73 139.70 139.65 136.39 135.87 131.39 131.30 131.17 131.08 129.21 128.91 128.79 128.73 128.67 128.55 128.35 128.23
62.55 62.49 62.43 62.36
42.04 41.46 40.15 33.37 30.03 16.00 15.93
14066.56 14061.65 14058.70 14053.58
13725.80
13673.44
13222.64 13213.13 13200.02
13003.20
12973.14 12948.67 12936.66
12904.74
6295.14 6288.34 6282.29 6275.43
4230.94 4172.63 4040.48
3358.54
3021.90
1609.93 1603.57
f1 (ppm)

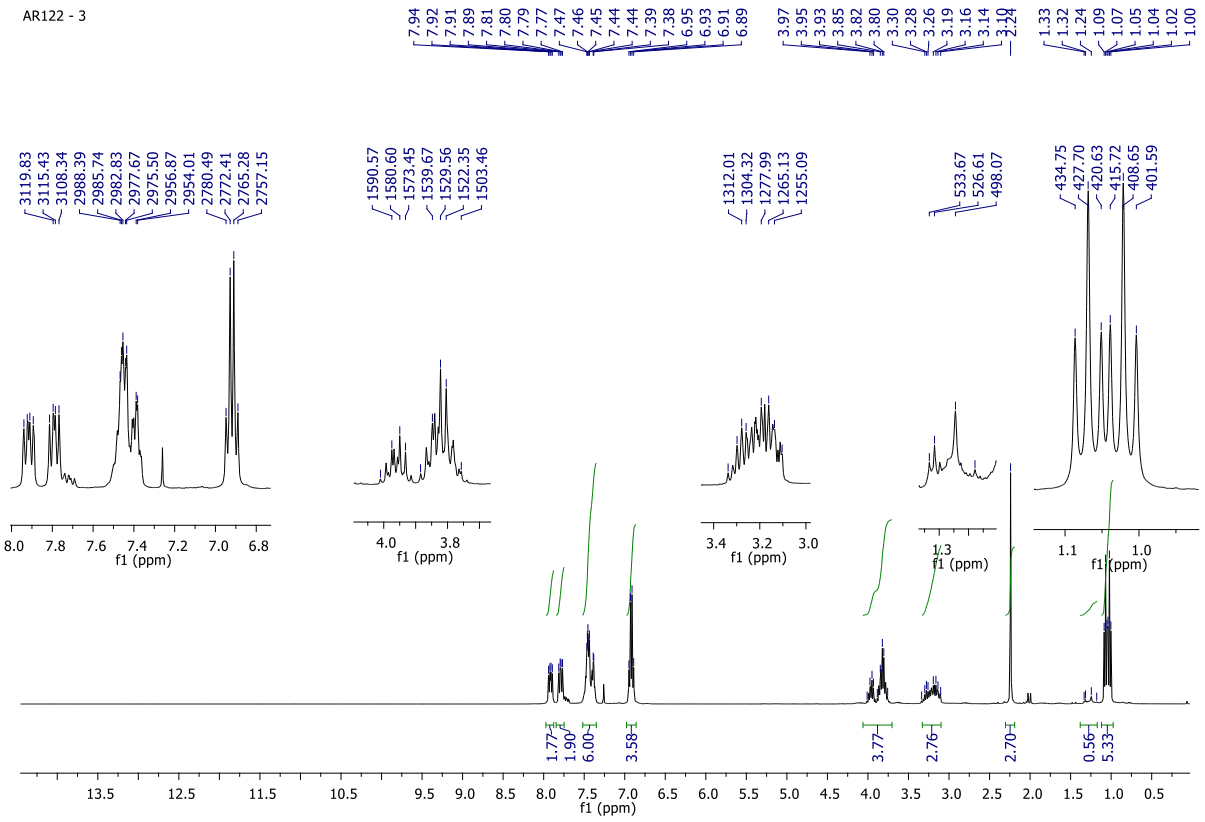
S22: ^{31}P, ^{1}H, and ^{13}C NMR spectra and mass analysis of diethyl (1-(diphenylphosphoryl)-2-(p-tolyl)ethyl)phosphonate (15b)

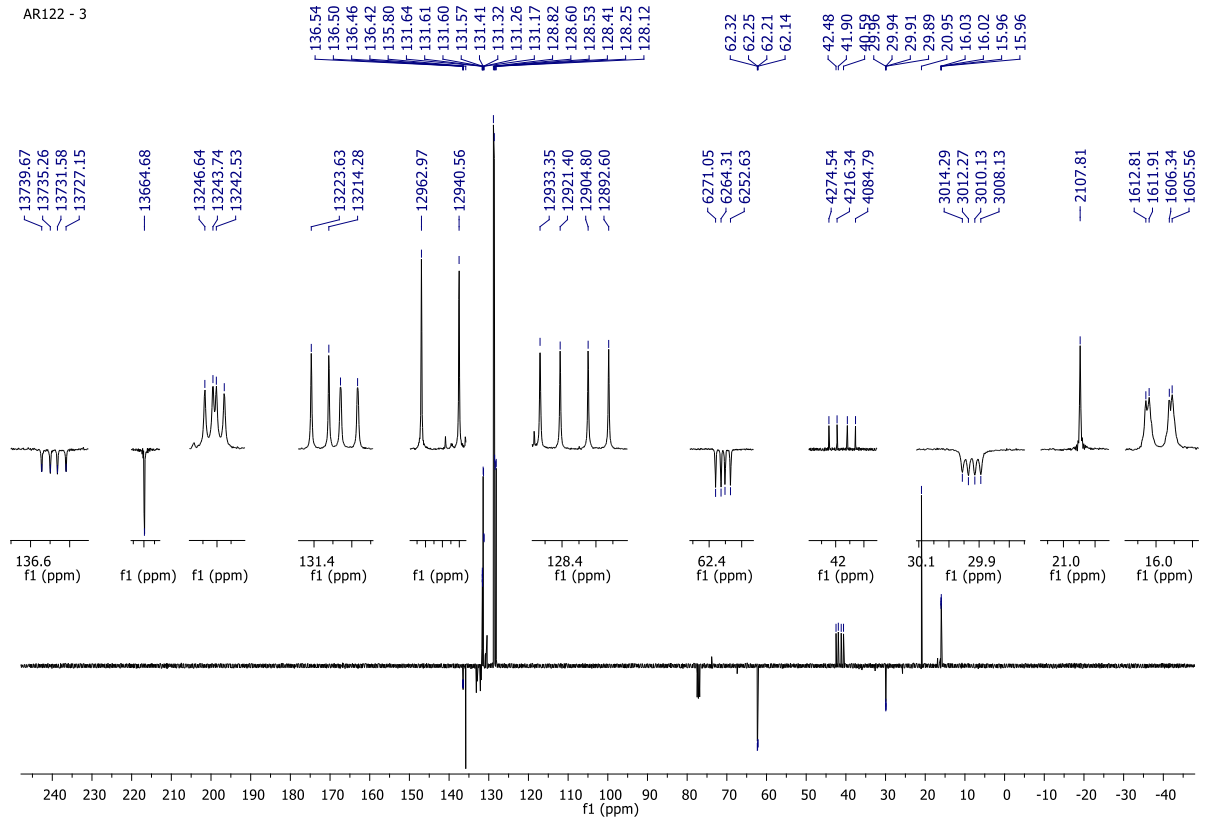
diethyl (1-(diphenylphosphoryl)-2-(p-tolyl)ethyl)phosphonate

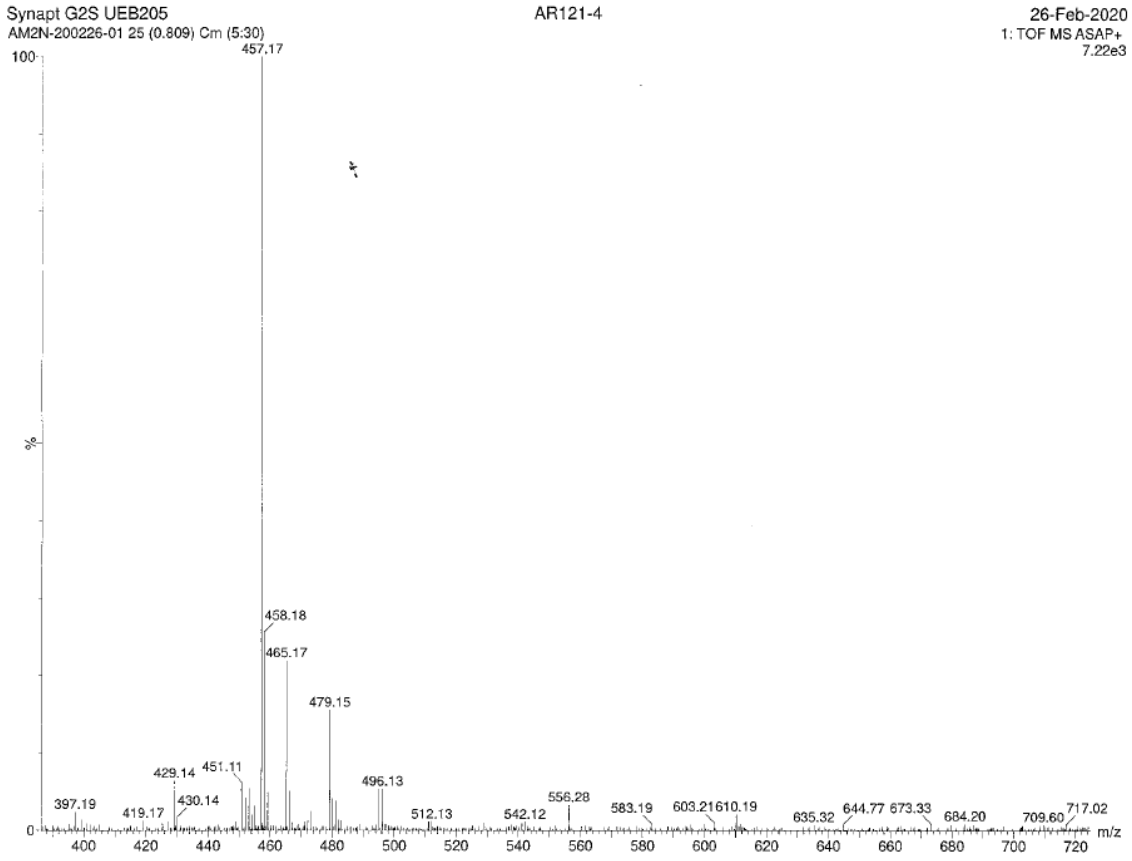


$C_{25}H_{30}O_4P_2$
456,46 g/mol
White solid









High Resolution Mass Result

Analysis Info

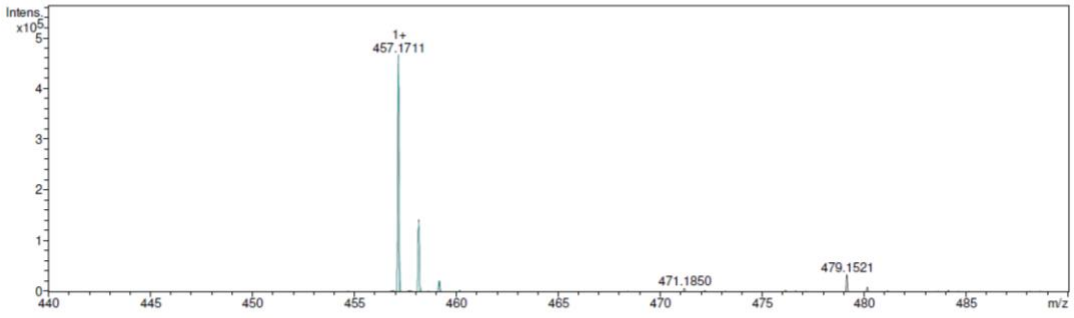
Sample Name **AR122-3**

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
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	2	C25H31O4P2	35.58	457.1692	1.9	4.2	11.5	even	ok

Bruker Compass DataAnalysis 4.1 printed: 2/24/2020 3:31:49 PM Page 1 of 1

Mass Result

Analysis Info

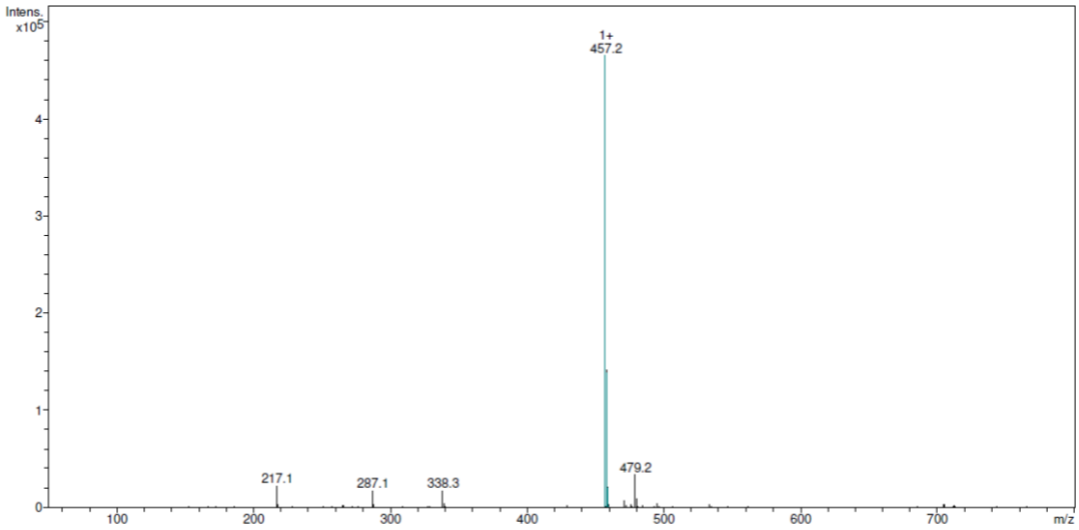
Sample Name **AR122-3**

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



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