Supplementary Material

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

Abdoul Razak Halidou Dougourikoye^{a,b}, Rachida Babouri^a, Jean-Noël Volle^a, Amadou Tidjani Ilagouma^b, Jean-Luc Pirat^a, and David Virieux^a*

^a ICGM, Univ. Montpellier, ENSCM, CNRS, Montpellier, France; ^b Département de Chimie, Faculté des Sciences, Université Abdou Moumouni, B.P. 10662, Niamey, Niger.

E-mail : <u>david.virieux@enscm.fr</u>

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AR018

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



240 220 200 180 160 120 100 80 60 40 f1 (ppm) 20 0 -20 -40 -60 -80 -100 -130 140



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





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







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



AR070

 7.14

 7.14

 7.116

 7.116

 7.116

 7.116

 7.117

 7.116

 7.117

 7.117

 7.118

 7.1113

 7.1113

 7.1123



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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 formuta(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 Y-JP 17030703 4 (0.175) Cm (4-7:8)

SYNAPT G2-S Y-JP17030703	#UEB205 4 (0.175) Cm (4-				AR		07-Mar-2017 1: TOF MS ES+ 1.54e+007				
100 231.02	245.03 250.16	259.05	273.07277.08	287.08	300.11 305.0	¹⁹ 317.1	333	339.05	355.11	371.08_374.19	386.19
230	240 250	260	270 280	290	300	310 3	20 330	340 3	350 360	370 380	390
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 333.1229	0.0 0.3	0.0 0.9	-0.5 9.5	1436.9 1441.2	0.014	98.58 1.42	C11 H27 C14 H18	07 P2 N6 02 P		

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





240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 fl (ppm)



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 Y-JP17030701 4 (0.175) Cm (3x4-9:11)

SYNAPT G2-S4 Y-JP 17030701	UEB205 4 (0.175) Cm (3:4-	9:11)	20 11		AF	8070		07 1: TOF 3	07-Mar-2017 1: TOF MS ES+ 3.86e+007		
100 261.0 260.0	270.0	275.0	7 <u>287.08</u> 90.0	3 <u>289.10</u> 2 290.0	3 91.08 30	01.10 	10 312.08 310.0	3 317.13 319.11 328.06 331.11 336.08 34 320.0 330.0 340.0	1.09 m/z		
Minimum: Maximum:		1.0	1.0	-1.5 50.0							
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula			
301.0969	301.0970 301.0967	-0.1 0.2	-0.3 0.7	0.5 10.5	2207.2 2218.0	0.000 10.826	100.00 0.00	C10 H23 O6 P2 C13 H14 N6 O P			

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



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

64.20
74.18
-4.16
74.14
74.14
74.12
74.12 AR082 $\begin{array}{c} 22,244\\ 22,244\\ 22,244\\ 22,233\\ 22,233\\ 22,233\\ 22,233\\ 22,233\\ 22,233\\ 22,233\\ 23,23,233\\ 23,23,233\\ 23,23,232\\ 23,23,232\\ 23,23,232\\ 23,23,23\\ 23,23,$ - 1679.10 - 1672.02 - 1664.08 - 1656.92 - 1649.82 $\begin{array}{c} 922.34 \\ \hline 974.91 \\ 967.53 \\ 967.53 \\ 967.53 \\ 958.75 \\ - 951.30 \\ - 951.30 \\ 935.48 \\ 935.21 \\$ Control 2008
 - 534.45 - 527.39 - 520.30 4.25 2.50 2.45 2.40 2.35 2.30 2.25 f1 (ppm) 4.20 4.15 f1 (ppm) 4.10 1.55 1.50 1,45 1.40 1.35 f1 (ppm) 1.30 1.25 1.10 --3.04 √ 12.001 Ч 7.97 12.0 11.5 11.0 10.5 10.0 8.5 7.5 7.0 6.5 6.0 f1 (ppm) 5.0 4.5 3.5 3.0 2.5 1.5 0.5 9.5 9.0 8.0 5.5 4.0 2.0 1.0 0.0 $<_{62.18}^{62.24}$ 23.23 30.88 29.52 16.15 16.09 16.07 16.07 9.91 AR082 -- 6263.39 -- 6256.59 1009.34 1003.11 996.87 1624.98 1623.22 1618.75 1616.94 51 A 16.0 f1 (ppm) 10.2 9.8 _____f1 (ppm) 32 30 ____f1 (ppm)_ 28 16.4

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 fl (ppm)





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 lons 839 formuta(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 Y-JP 17030702 4 (0.175) Cm (3:4-9:11)

SYNAPT G2-S# Y-JP17030702	UEB205 4 (0.175) Cm (3	3:4-9:11)		AR082						07-Mar-2017 1: TOF MS ES+ 3.92e+007		
100 214.99 2	19.02 2	33.03	247.05	261.07	275.0	8 28	9.10	03.11 306.55	317.13 325	.09 330.07	345.12 ^{349.12}	
210	220 230) 240	250	260	270	280	290 30	0 310	320	330	340 350	
Minimum: Maximum:		1.0	1.0	-1.5 50.0								
Mass	Calc. Mas	s mDa	PPM	DBE	i-FIT	Norm	Conf (%)	Formula				
303.1127	303.1126 303.1123	0.1 0.4	0.3	-0.5 9.5	1995.2 2003.4	0.000 8.238	99.97 0.03	C10 H25 C13 H16	06 P2 N6 0 P			













Elemental Composition Report

Page 1

Single Mass Analysis Tolerance = 2.0 PPM / DBE: min = -50.0, max = 100.0 Bement prediction: Off Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron lons 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 Y-JLP-1605110	#UEB205)1 4 (0.175) Cm (4)			AR	11-May-2016 1: TOF MS ES+ 7.85e+007			
¹⁰ 8 449 440 44	9.20.451.10.453.10 5 450 455	459.14 460 465	7.12 470	479.13.481	.13 493 485 49	.15,495.15	501.11.50 500 50	3.11 517.09.519.09 5 510 515 520	526.23 529.59 534.11 540.24 525 530 535 540
Minimum: Maximum:		1.1	2.0	-50.0 100.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula	
479.1328	479.1327 479.1324	0.1	0.2 0.8	-0.5 9.5	1653.9 1669.5	0.000	100.00	C17 H38 O6 P2 H C20 H29 N6 O P	Br Br

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



60 40 f1 (ppm) 240 . 220 200 180 . 160 . 140 120 100 . 80 20 0 -20 -40 . -60 . -80 -100 -130







Page 1

Single Mass Analysis Tolerance = 1.0 PPM / DBE: min = -1.5, max = 50.0 Bement prediction: Off Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron lons 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

C: 0-100 H SYNAPT G2-S: Y-JP17021305	: 0-100 N: 0- #UEB205 4 (0.175) Cm (354)	20 0:0	-20 P:	2-2	ARO	54 (1)			1:	13-Feb-2017 TOF MS ES+ 8.57e+007
198 3 367.14	373.19 ^{375.20} 370.0	378.65 ³ 380.0	87.21 39	2.17 <u>395</u> 0.0	401.2	²² 403.23	412.21 4	423.20 420.0	429.25 436 430.0	5.20 439.18 5.20 / m/z 440.0
Minimum: Maximum:		10.0	1.0	-1.5 50.0						
Mass	Calc. Mass	mDa	PFM	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: ³¹P, ¹H and ¹³C NMR spectra and mass analysis of tetraethyl (2-(p-tolyl)ethane-1,1-diyl)bis(phosphonate) (2e)



. 240 . 220 200 180 100 . 80 60 40 f1 (ppm) 20 0 -20 -40 -60 . -80 -100 -130 160 140 120

-4.18 -4.14 -4.14 -4.14 -4.12 -4.05 -4.05 -4.05 -4.05 AR053 - 3 Z 7.16 Z 7.14 Z 7.08 Z 7.06 -3.23-3.23-3.29-2.68-2.64-2.6→ 1671.85 → 1664.85 → 1664.85 1657.69 1657.69 1654.62 1631.74 1631.74 1631.74 1631.74 1631.74 1631.74 1620.09 1620.09 1298.53
 1292.32
 1292.32
 1281.99
 1275.75
 1265.43
 1265.23 ✓ 1078.78 ~ 1072.59 ✓ 1054.90 ~ 1048.70 ~ 1042.45 1024.82 - 2833.66 - 2825.79 -- 515.44 -- 508.35 -- 501.23 -- 494.08 -- 2865.54 -- 2857.57 11 ML IVI 2.7 2.6 f1 (ppm) 4.15 4.05 f1 (ppm) 3.20 3.15 f1 (ppm) 7.11 f1 (ppm) 3.25 1.30 1.25 f1 (ppm 1.20 7.02 ÷ Ч 2.93 4 폰 Ŧ 12.00H 1.89 95 2.00 <u> 66.C</u> 12.0 11.5 11.0 10.5 10.0 8.5 8.0 7.5 7.0 6.5 6.0 f1 (ppm) 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 9.5 9.0 1.0 0.5 0.0 - 136.41 - 136.34 - 136.26 - 135.73 - 135.73 - 128.69 AR053 - 3 -62.45 -62.38 -62.27 -62.21 40.25 38.93 38.93 37.62 30.62 30.57 30.57 30.57 30.52 16.13 16.13 1 ~ 13727.61 - 13720.23 ~ 13712.83 -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 12 W γγ ΜŅ 1111 16.2 16.0 f1 (ppm) 136.2 135.8 f1 (ppm) 129.0 128.5 f1 (ppm) 40 38 f1 (ppm) 136.6 62.4 f1 (ppm) 30.6 f1 (ppm) f1 (ppm)

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







Elemental Composition Report

Page 1

Single Mass Analysis Tolerance = 3.0 PPM / DBE: min = -1.5, max = 50.0 Bement prediction: Off Number of isotope peaks used for i-FIT = 3

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

SYNAPT G2:\$#UEB205 AR053 (3) Y-JP17021303 4 (0.175) Cm (355)											
100 1 356.07	359.08 365.13 360.0	367.13 370.0	377.11	385.1(80.0	387.11	393.16 ₃₉₅	.17 404 400.0	4.14 412.13 41 410.0	421.19	428.14 431.12 428.14 / m/z 430.0	
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 393.1609	0.2	0.5 -2.8	3.5 8.5	2237.7 2244.1	0.002 6.393	99.83 0.17	C17 H31 O6 F C18 H27 N4 C	2 2 P2		

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











Page 1

ARKIVOC 2020, vi, S1-S73

Single Mass Analysis Tolerance = 1.0 PPM / DBE: min = -1.5, max = 50.0 Bernent prediction: Off Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron lons 1410 formula(e) evaluated with 2 results within limits (up to 50 best isotopic matches for each mass) Bements Used:

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

C: 0-100 H SYNAPT G2-S Y-JP17021304	4 (0.175) Cm (3:5	-20 O:0-)	20 P. 2-2	AR	053 (2)			13-Feb-20 1: TOF MS E3 1.41e+0)17 5+
100 471.20 0 470.0	480.17 483.2 480.0	485.19 490	497.22	499.23 508.21. 500.0	511.24 516.20 510.0	519.20 520.0	524.18,525.68 532.20 530.0	535.18 545.22.546.68 540.0	n∕z
Minimum: Maximum:		10.0	-1. 1.0 50.	5					
Mass	Calc. Mass	mDa 1	PPM DBE	1-FIT	Norm Con	nf(%) Fo	umula		
S11: ³¹P, ¹H, and ¹³C NMR spectra and mass analysis of tetraethyl (1,3-bis(4-(bromomethyl)phenyl)propane-2,2-diyl)bis(phosphonate) (2f)



AR065 - 3







Page 1

Single Mass Analysis Tolerance = 2.0 PPM / DBE: min = -50.0, max = 100.0 Bement prediction: Off Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron lons 4436 formuta(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 Y-JLP-1605110	AR021(3)						11-May-2016 1: TOF MS ES+ 4.95e+007		
100 459.09 457.5	460.09 ^{461.09} 460.0 462.5	465.02 465.0	467.02 467.5	471 470.55 470.0	472.5	474.07 4	76.08 479 477.5	. <u>13 481.13 ^{484.17}485.09</u> 480.0 482.5 485.0	488.09 490.04490.57 487.5 490.0
Minimum: Maximum: Mass	Calc. Mass	1.1 mDa	2.0 PFM	-50.0 100.0 DBE	i-FIT	Norm	Conf(%)	Formula	
471.0704	471.0701 471.0698 471.0698 471.0712 471.0708 471.0708 471.0695	0.3 0.6 0.6 -0.8 -0.4 0.9	0.6 1.3 1.3 -1.7 -0.8 1.9	3.5 13.5 -16.5 -11.5 -1.5 -6.5	1608.0 1617.1 1623.5 1624.8 1626.0 1626.3	0.000 9.086 15.463 16.714 17.976 18.290	99.99 0.01 0.00 0.00 0.00 0.00 0.00	C17 H30 O6 P2 Br C20 H21 N6 O P Br C4 H43 O10 P2 Br2 C5 H39 N4 O6 P2 Br2 C8 H30 N10 O P Br2 C7 H34 N6 O5 P Br2	

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







240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 f1 (ppm)





Elemental Composition Report

Page 1

Single Mass Analysis Tolerance = 2.0 PPM / DBE: min = -50.0, max = 100.0 Bement prediction: Off Number of isotope peaks used for i-FfT = 3

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

SYNAPT G2-S#UEB205 Y-JLP-16051102 4 (0.175) Cm (4) 11-May-2016 1: TOF MS ES+ 3.18e+007 AR021(2) 655.04 655.04 655.04 659.04 677.02 693.00 705.27 520 530 540 550 560 570 580 590 600 610 620 630 640 650 660 670 680 690 700 710 720 Minimum: -50.0 Maximum: 1.1 2.0 100.0 PPM DBE i-FIT Norm Conf(%) Formula Mass Calc. Mass mDa 0.1 0.2 7.5 0.4 0.6 17.5 -1.3 -2.0 12.5 653.0433 653.0432 1666.3 0.432 64.91 C25 H37 O6 P2 Br2 653.0429 653.0446 1667.5 1.563 20.96 C28 H28 N6 O P Br2 1667.9 1.957 14.13 C26 H33 N4 O2 P2 Br2

S13: ³¹P, ¹H, and ¹³C NMR spectra and mass analysis of tetraethyl (1-(p-tolyl)propane-2,2diyl)bis(phosphonate) (6)



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







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 AR085 Y-JP17042806 3 (0.141) Cm (3:5)

SYNAPT G2- Y-JP1704280	S#UEB205 6 3 (0.141) Cm (3:	5)		0-2	AR085					28-Apr-2017 1: TOF MS ES+ 1 68+008		
¹⁰⁰ 3 .	361.13 365.13 360.0 370	<u>373.09</u> .0	379.14 <u>.381.1</u> 380.0	5 39 390.0	3.16 40 400	407 1.13 .0	.17 	421.19 420.0	429.16 434.14	443.17.445.13.447.13 440.0		
Minimum: Maximum:		1.0	3.0	-1.5 50.0								
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula				
407.1747	407.1752 407.1749 407.1751 407.1739 407.1738 407.1738 407.1754 407.1754	-0.5 -0.2 -0.4 0.8 0.9 0.9 -0.7 0.6	-1.2 -0.5 -1.0 2.0 2.2 2.2 -1.7 1.5	3.5 13.5 5.5 9.5 0.5 11.5 6.5 1.5	2314.7 2320.3 2321.5 2322.1 2325.0 2327.6 2328.4 2328.9	0.005 5.606 6.836 7.427 10.306 12.962 13.738 14.207	99.46 0.37 0.11 0.06 0.00 0.00 0.00 0.00	C18 H33 O6 C21 H24 N6 C11 H23 N1 C15 H25 N1 C10 H27 N6 C8 H15 N20 C6 H20 N18 C5 H24 N14	P2 0 P 0 07 0 P2 011 0 02 P 06 P			

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



AR090



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



240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 f1 (ppm)





Elemental Composition Report

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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 Y-JP17042807 3 (0.141) Cm (3)

SYNAPT G2-S Y-JP17042807	0.2	Ał	AR090				28-Apr-2017 1: TOF MS ES+ 3.57e+007				
100 1 249.08 0 1 1 1 1 260 260	275.08 285.10	303.11 31 300	17.13 331.1 320	14 353 13 340	359.18 360	387.21 380	415 401.22 400	.24 426.22 437 420 440	468.27 460	483.21	499.33 500 m/z
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 331.1436 331.1438 331.1447	0.2 0.5 0.3 -0.6	0.6 1.5 0.9 -1.8	-0.5 9.5 1.5 13.5	1968.1 1975.3 1975.6 1979.6	0.001 7.164 7.472 11.423	99.86 0.08 0.06 0.00	C12 H29 O6 C15 H20 N6 C5 H19 N10 C21 H19 N2	P2 O P O7 O2		

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



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



240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 f1 (ppm)





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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 AR112 Y-JP17042805 4 (0.175) Cm (3:4)

SYNAPT G2-S Y-JP17042805			AF		28-Apr-2017 1: TOF MS ES+ 1 17e+008				
100 0 360	8 <u>373.19</u> 370 380	387.21 390	401.22 400	415.24 410 4	426.22 20 430	437.22 440	453.19 ⁴ 450 4	57.28 475.28483.21 499.33 460 470 480 490 500	513.35 510 m/z
Minimum: Maximum:		1.0	3.0	-1.5 50.0					
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Norm	Conf(%)	Formula	
415.2374	415.2378 415.2377 415.2375 415.2365 415.2380	-0.4 -0.3 -0.1 0.9 -0.6	-1.0 -0.7 -0.2 2.2 -1.4	-0.5 1.5 9.5 5.5 2.5	2068.3 2073.5 2073.7 2075.0 2080.9	0.012 5.180 5.366 6.712 12.590	98.85 0.56 0.47 0.12 0.00	C18 H41 O6 P2 C11 H31 N10 O7 C21 H32 N6 O P C15 H33 N10 P2 C6 H28 N18 O2 P	

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





60 40 f1 (ppm) 240 220 200 180 160 140 120 100 80 20 0 -20 -40 -60 -80 -100 -130







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)





. 140 . 120 100 80 60 40 20 0 -10 -30 -50 f1 (ppm) . -70 -90 -110 -140 -170 -200 -230



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



AR013



140 120 100 80 60 40 20 0 -10 -30 -50 f1 (ppm) -70 -90 -110 -140 -170 -200 -230 AR013



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



. 140 . 120 100 80 60 40 20 0 -10 -30 -50 f1 (ppm) . -70 -90 -110 -140 -170 -200 -230 AR008

28.2 28.2 28.2 28.2 28.2 28.2 28.2 29.2 29.2 20.2



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





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





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



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