

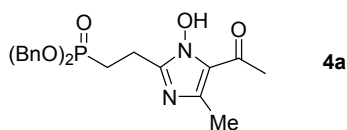
Fosmidomycin Analogues with *N*-Hydroxyimidazole and *N*-Hydroxyimidazolone as Chelating Unit

Camille Midrier,^a Sonia Montel,^a Ralf Braun,^b Klaus Haaf,^b Lothar Willms,^b Arie Van der Lee,^c Jean-Noël Volle,^a Jean-Luc Pirat^{*a} and David Virieux^{*a}

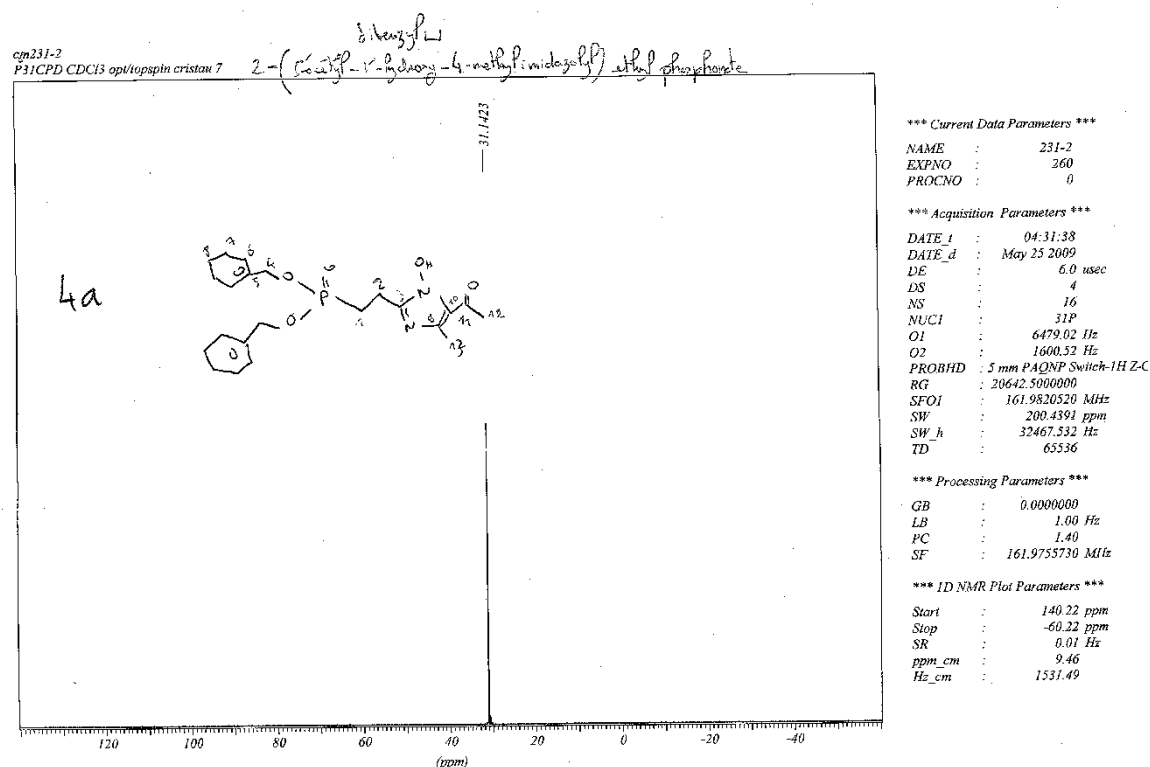
Fosmidomycin has been reported to have many biological activities as antibacterial, antimalarial, along with herbicidal agent. Its unique mode of action involves the inhibition of a key step of the non mevalonate pathway by blockade of a crucial enzyme, the 1-deoxy-D-xylulose 5-phosphate reductoisomerase (DXR), whose expression is present in bacteria, plasmodium parasite and higher plants, but not in mammals. Herein we report the development of fosmidomycin and of FR-900098 constrained analogues belonging to an unusual heterocyclic based complexing subunit involving *N*-hydroxyimidazoles and cyclic *N*-hydroxyureas.

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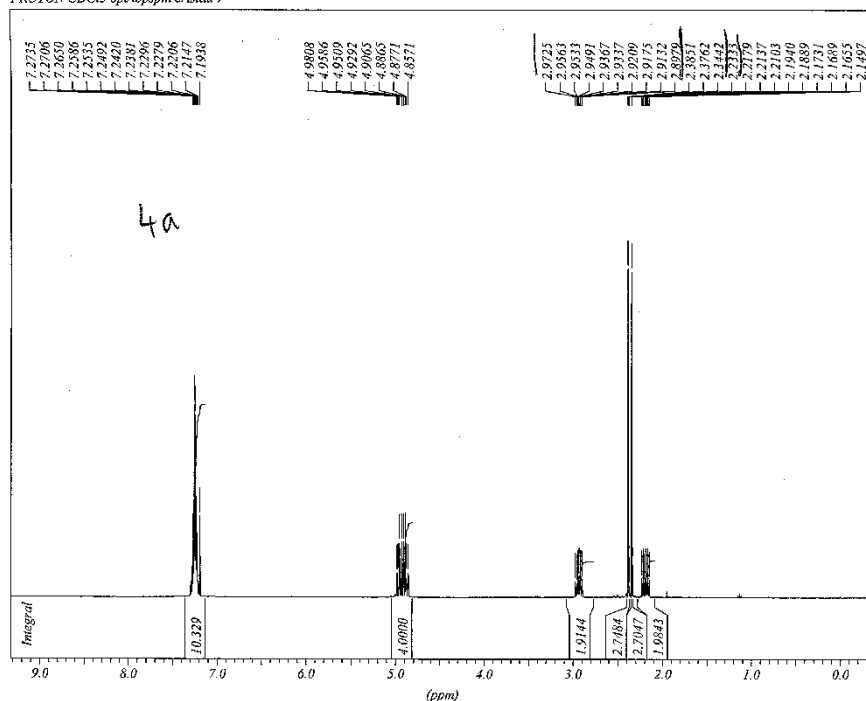
Dibenzyl (2-(5-acetyl-1-hydroxy-1H-imidazol-2-yl)ethyl)phosphonate (4a)



¹H NMR (400.13 MHz, CDCl₃) δ 2.14–2.22 (m, 2H), 2.34 (s, 3H), 2.38 (s, 3H), 2.89–2.97 (m, 2H), 4.88 (dd, ²J_{HH} = 11.8 Hz, ³J_{HP} = 8.0 Hz, 2H), 4.95 (dd, ²J_{HH} = 11.8 Hz, ³J_{HP} = 8.8 Hz, 2H), 7.19–7.27 (m, 10H). ¹³C NMR (100.61 MHz, CDCl₃) δ 16.60 (s), 18.53 (d, ²J_{CP} = 3.6 Hz), 22.97 (d, ¹J_{CP} = 141.3 Hz), 28.19 (s), 67.41 (d, ²J_{CP} = 6.1 Hz), 122.27 (s), 127.87 (s), 128.47 (s), 128.61 (s), 136.04 (d, ³J_{CP} = 6.6 Hz), 141.66 (d, ³J_{CP} = 15.3 Hz), 190.88 (s). ³¹P NMR (161.97 MHz, CDCl₃) δ 31.14 (s). HRMS (ESI) *m/z* [M+H]⁺ calcd for C₂₂H₂₆N₂O₅P 429.1579, found 429.1577.



cm231-2
PROTON CDC13 opt/topspin cristau 7



*** Current Data Parameters ***

NAME : 231-2
EXPNO : 261
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 04:35:00
DATE_d : May 25 2009
DE : 6.0 usec
DS : 2
NS : 16
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 90.5000000
SFO1 : 400.1324008 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 65536

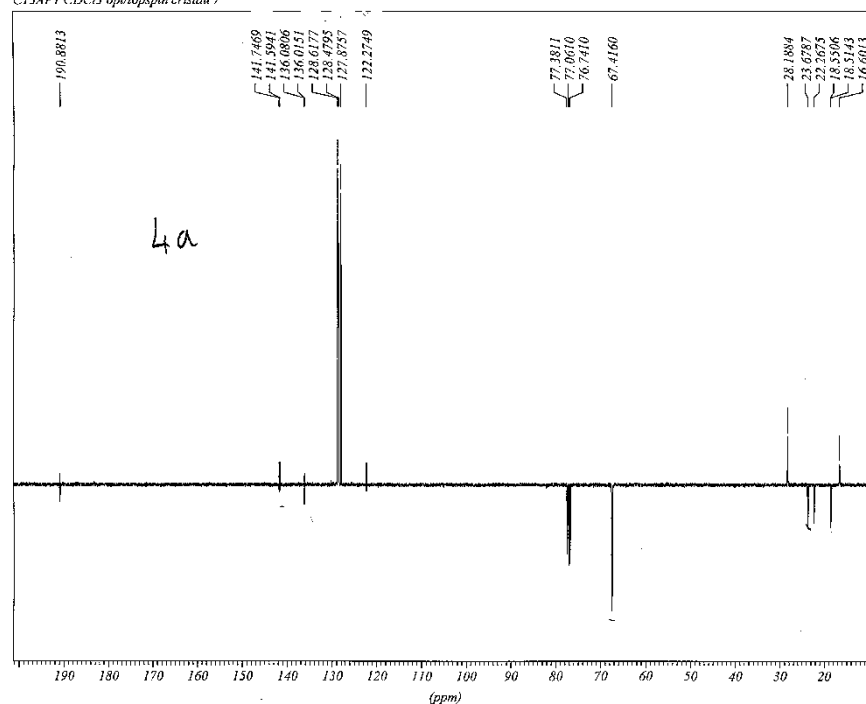
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1300366 MHz

*** 1D NMR Plot Parameters ***

Start : 9.32 ppm
Stop : -0.38 ppm
SR : 36.60 Hz
ppm_cm : 0.46
Hz_cm : 183.11

cm231-2
13APT CDC13 opt/topspin cristau 7



*** Current Data Parameters ***

NAME : 231-2
EXPNO : 262
PROCNO : 0

*** Acquisition Parameters ***

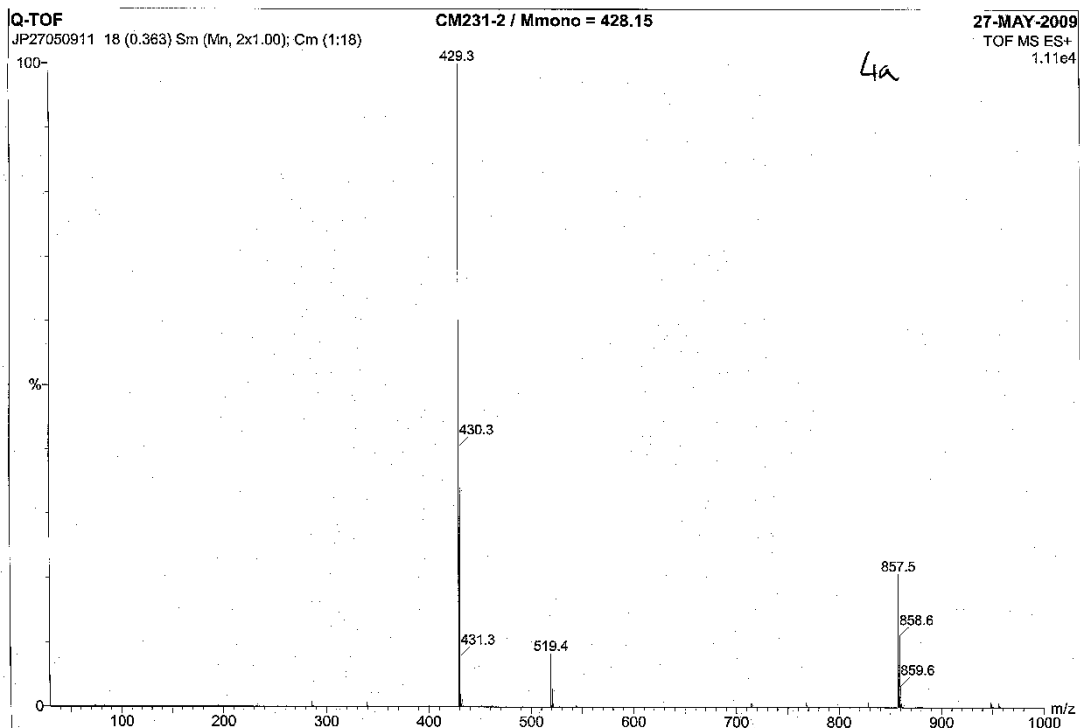
DATE_1 : 21:07:02
DATE_d : May 25 2009
DE : 6.0 usec
DS : 4
NS : 1024
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 23170.5000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Start : 201.20 ppm
Stop : 8.29 ppm
SR : 0.00 Hz
ppm_cm : 9.10
Hz_cm : 915.52



Elemental Composition Report

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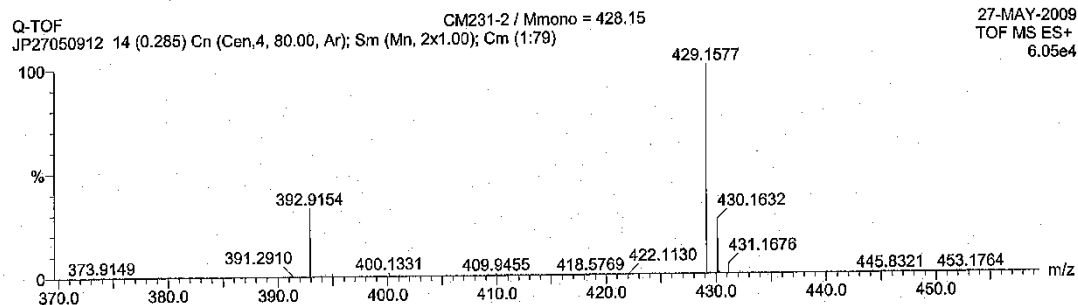
Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

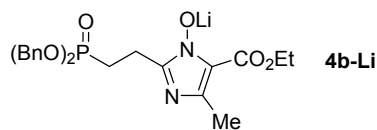
Monoisotopic Mass, Odd and Even Electron Ions

123 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)



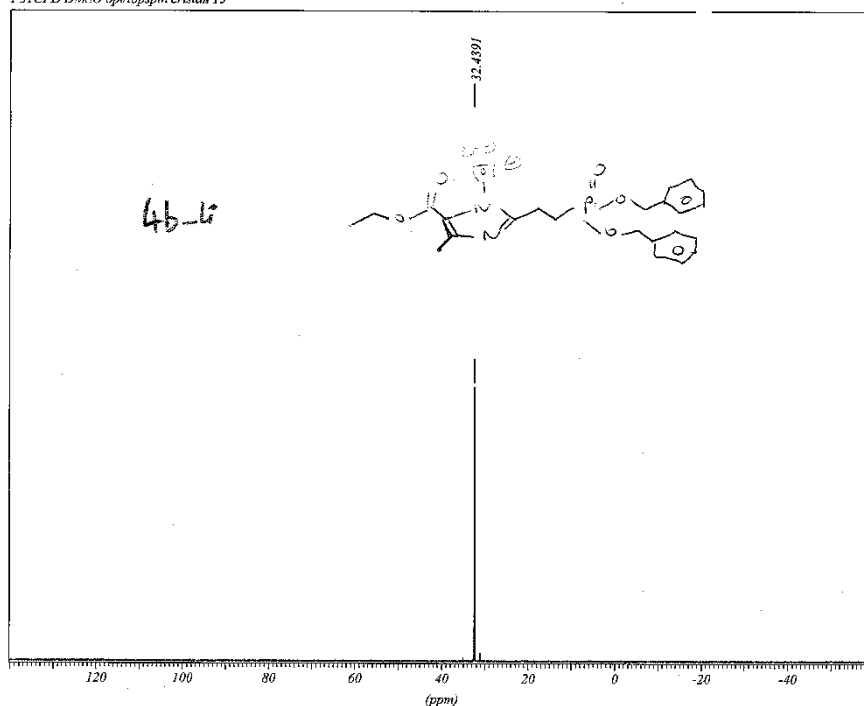
Minimum:				-5.0					
Maximum:		3.0	5.0	50.0					
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula			
429.1577	429.1579	-0.2	-0.5	11.5	1	C22	H26	N2	O5 P
	429.1566	1.1	2.6	12.0	2	C20	H24	N5	O4 P
	429.1606	-2.9	-6.8	16.0	3	C25	H24	N3	O2 P

Lithium 2-(2-(bis(benzyloxy)phosphoryl)ethyl)-5-(ethoxycarbonyl)-1H-imidazol-1-olate (4b-Li)



^1H NMR (400.13 MHz, DMSO- d_6) δ 1.26 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 2.18–2.30 (m, 2H), 2.21 (s, 3H), 2.75–2.81 (m, 2H), 4.19 (q, $^3J_{\text{HH}} = 7.0$ Hz, 2H), 4.98 (dd, $^2J_{\text{HH}} = 12.1$ Hz, $^3J_{\text{HP}} = 7.4$ Hz, 2H), 5.03 (dd, $^2J_{\text{HH}} = 12.1$ Hz, $^3J_{\text{HP}} = 8.3$ Hz, 2H), 7.19–7.27 (m, 10H). ^{13}C NMR (100.61 MHz, DMSO- d_6) δ 14.26 (s), 16.87 (s), 18.76 (d, $^2J_{\text{CP}} = 2.9$ Hz), 21.95 (d, $^1J_{\text{CP}} = 136.8$ Hz), 59.26 (s), 66.25 (d, $^2J_{\text{CP}} = 5.6$ Hz), 115.42 (s), 127.60 (s), 128.04 (s), 128.40 (s), 136.71 (d, $^3J_{\text{CP}} = 6.6$ Hz), 138.98 (s), 141.18 (d, $^3J_{\text{CP}} = 19.0$ Hz), 163.13 (s). ^{31}P NMR (161.97 MHz, DMSO- d_6) δ 32.44 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{23}\text{H}_{27}\text{LiN}_2\text{O}_6\text{P}$ 465.1767, found 465.1778.

cm252-3 (Li)
P31CPD DMSO opt/topspin cristau 13



*** Current Data Parameters ***

NAME : 252-3
EXPNO : 251
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 03:47:55
DATE_d : Feb 19 2009
DE : 6.0 ussec
DS : 4
NS : 16
NUC1 : 31P
O1 : 6479.02 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 20642.500000
SF01 : 161.9820520 MHz
SW : 200.4391 ppm
SW_h : 32467.532 Hz
TD : 65536

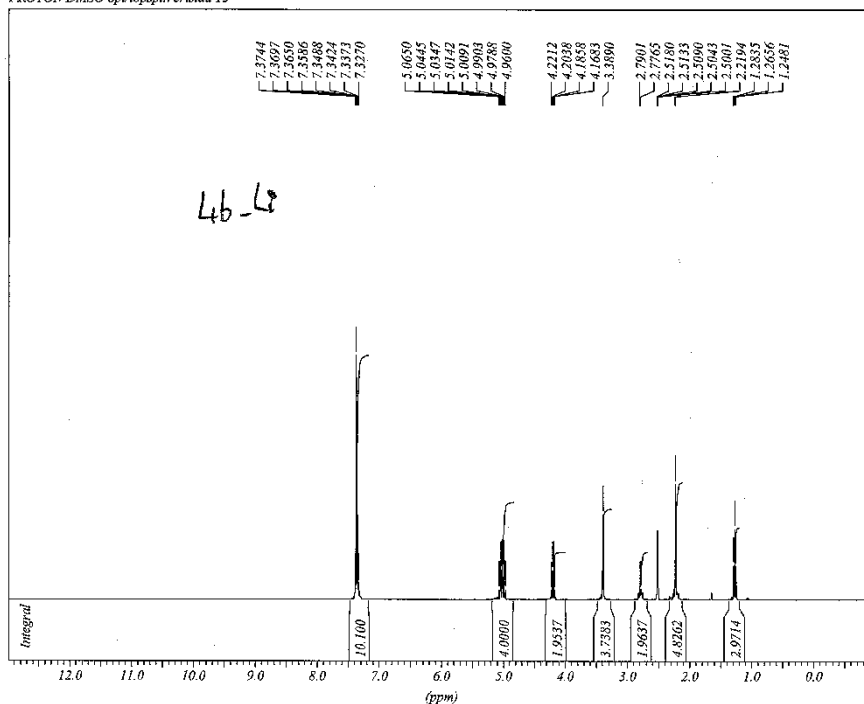
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 161.9755730 MHz

*** 1D NMR Plot Parameters ***

Start : 140.22 ppm
Stop : -60.22 ppm
SR : 0.01 Hz
ppm_cm : 9.46
Hz_cm : 1531.49

cm252-3 (Li)
PROTON DMSO opt/topspin cristau 13



*** Current Data Parameters ***

NAME : 252-3
EXPNO : 232
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 03:51:23
DATE_d : Feb 19 2009
DE : 6.0 usec
DS : 2
NS : 16
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 114.000000
SFO1 : 400.1324008 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 65536

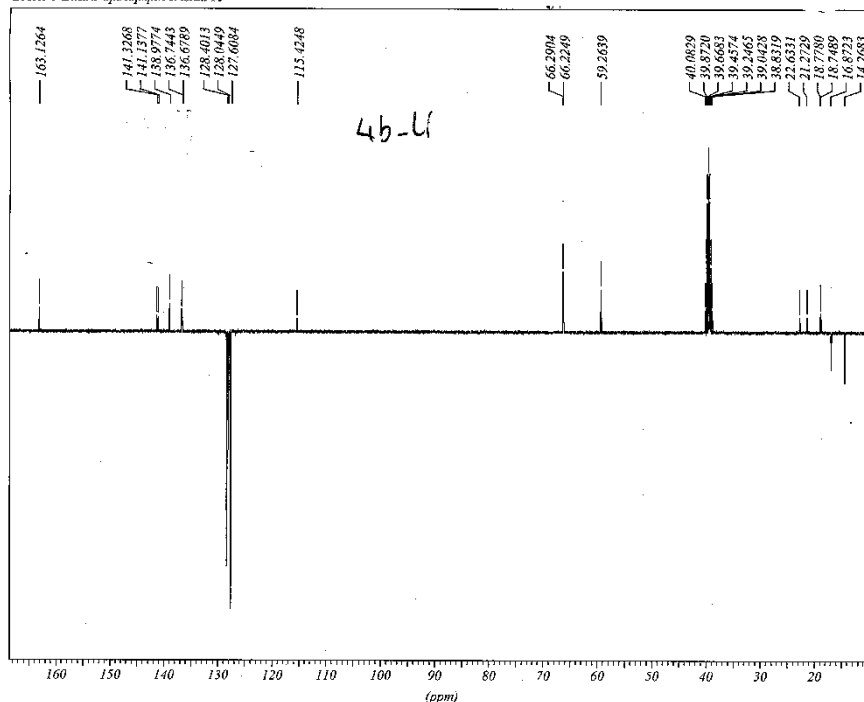
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1300000 MHz

*** 1D NMR Plot Parameters ***

Start : 12.99 ppm
Stop : -0.99 ppm
SR : 0.00 Hz
ppm_cm : 0.66
Hz_cm : 263.81

cm252-3 (Li)
C13APT DMSO opt/topspin cristau 13



*** Current Data Parameters ***

NAME : 252-3
EXPNO : 233
PROCNO : 0

*** Acquisition Parameters ***

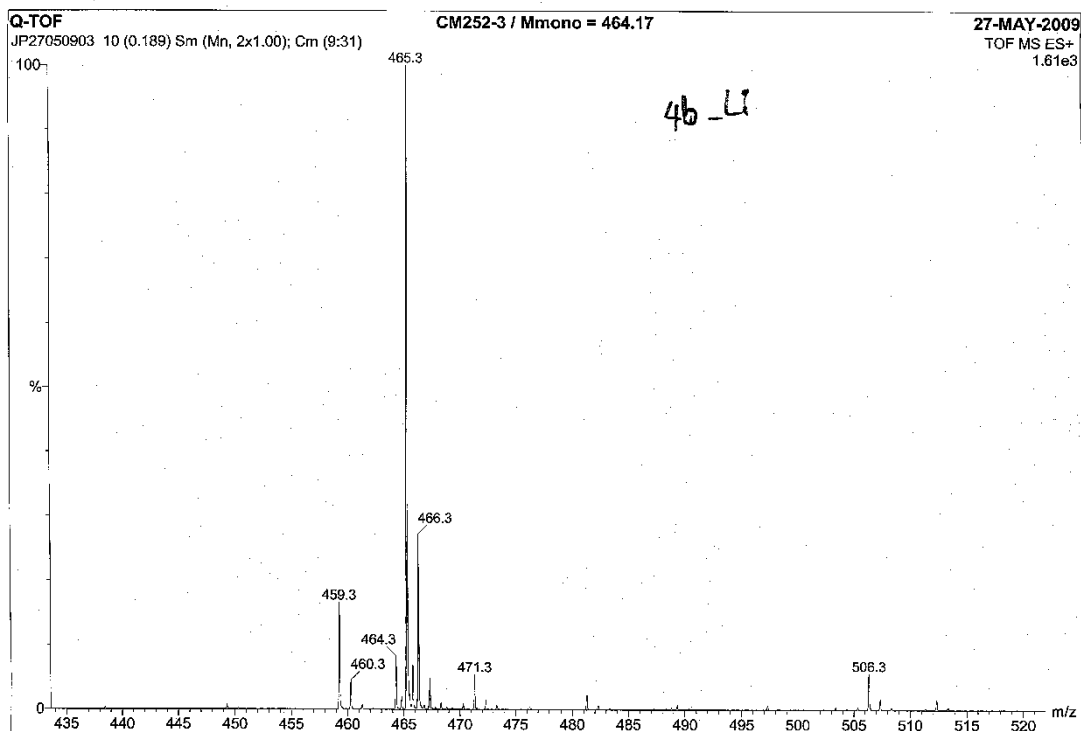
DATE_1 : 20:26:16
DATE_d : Feb 19 2009
DE : 6.0 usec
DS : 4
NS : 1024
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 16384.000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6128193 MHz

*** 1D NMR Plot Parameters ***

Start : 168.60 ppm
Stop : 8.73 ppm
SR : 50.31 Hz
ppm_cm : 7.34
Hz_cm : 758.73



Elemental Composition Report

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Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

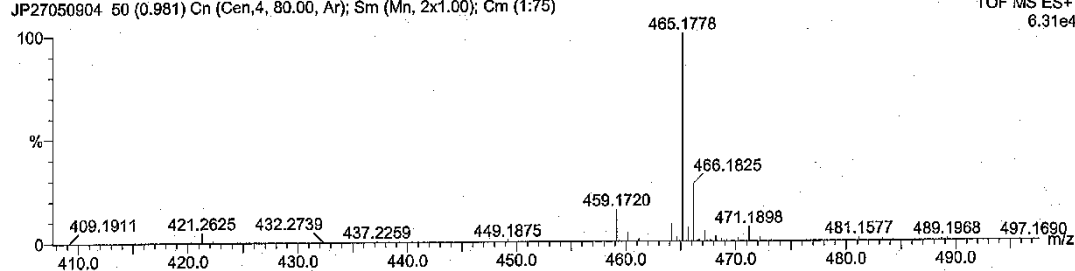
Monoisotopic Mass, Odd and Even Electron Ions

144 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

Q-TOF
JP27050904 50 (0.981) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (1:75)

CM252-3 / Mmono = 464.17

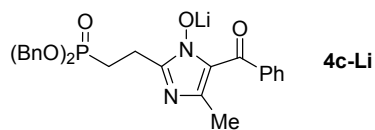
27-MAY-2009
TOF MS ES+
6.31e4



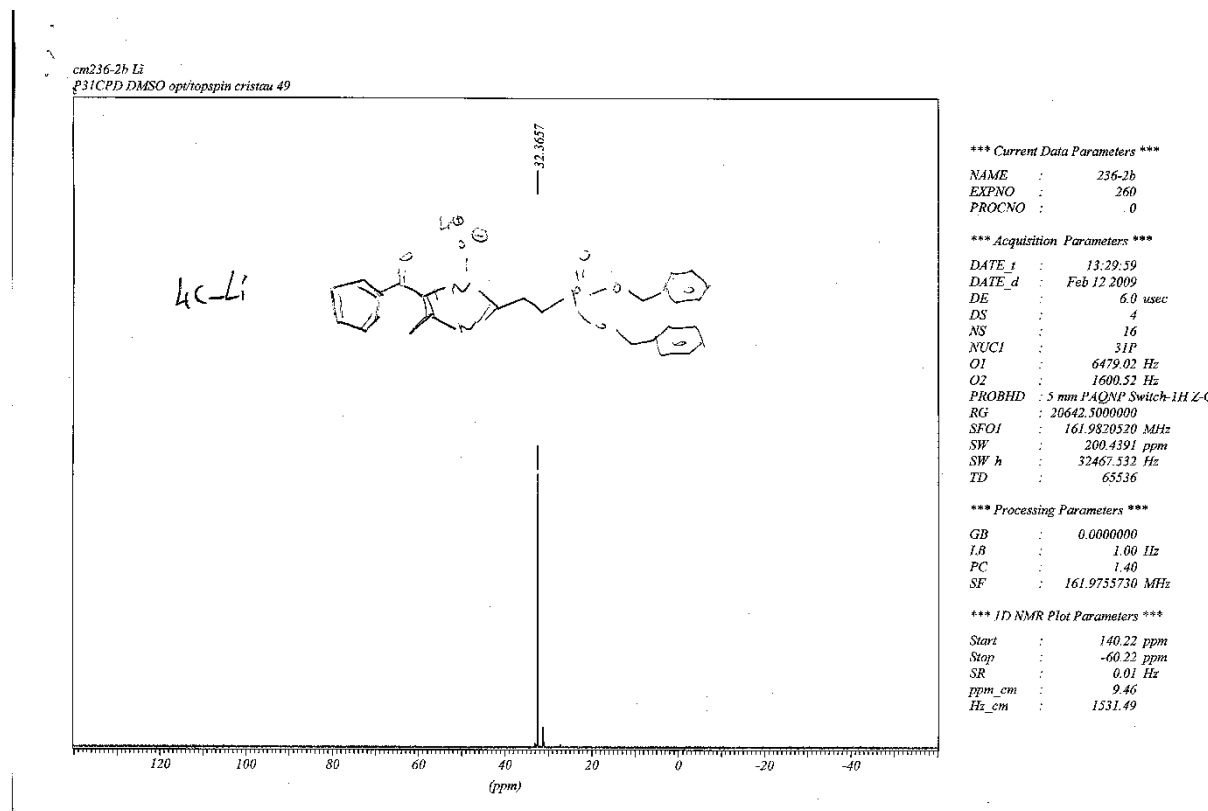
Minimum: -5.0
Maximum: 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
465.1778	465.1767	1.1	2.4	11.5	1	C23 H27 Li N2 O6 P 8
	465.1753	2.5	5.3	12.0	2	C21 H25 Li N5 O5 P

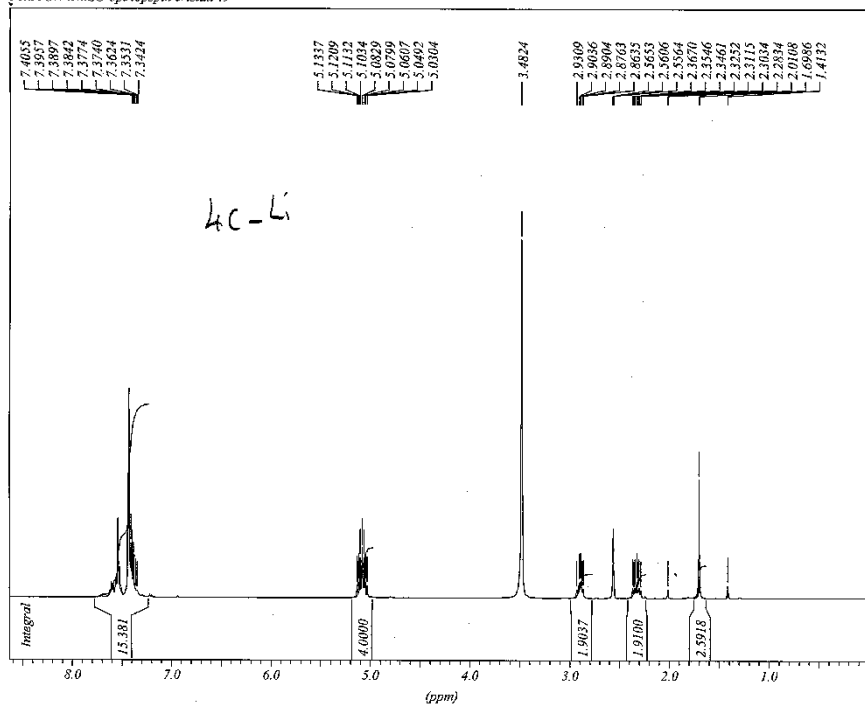
Lithium 5-benzoyl-2-(2-(bis(benzyloxy)phosphoryl)ethyl)-1H-imidazol-1-olate (4c-Li)



^1H NMR (400.13 MHz, DMSO- d_6) δ (ppm) 1.69 (s, 3H), 2.28–2.36 (m, 2H), 2.86–2.93 (m, 2H), 5.06 (dd, $^2J_{\text{HH}} = 12.1$ Hz, $^3J_{\text{HP}} = 7.5$ Hz, 2H), 5.1 (dd, $^2J_{\text{HH}} = 12.1$ Hz, $^3J_{\text{HP}} = 8.2$ Hz, 2H), 7.34–7.62 (m, 15H). ^{13}C NMR (100.61 MHz, DMSO- d_6) δ 17.42 (s), 18.74 (d, $^2J_{\text{CP}} = 2.9$ Hz), 22.68 (d, $^1J_{\text{CP}} = 138.3$ Hz), 66.32 (d, $^2J_{\text{CP}} = 5.8$ Hz), 125.27 (s), 127.63 (s), 128.04 (s), 128.07 (s), 128.24 (s), 128.41 (s), 136.69 (d, $^3J_{\text{CP}} = 6.6$ Hz), 140.45 (s), 141.41 (s), 142.72 (d, $^3J_{\text{CP}} = 18.3$ Hz). ^{31}P NMR (161.97 MHz, DMSO- d_6) δ 32.37 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{27}\text{H}_{27}\text{LiN}_2\text{O}_5\text{P}$ 497.1818, found 497.1817.



cm236-2b Li
PROTON DMSO opt/topspin cristau 49



*** Current Data Parameters ***

NAME : 236-2b
EXPNO : 261
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 13:33:17
DATE_d : Feb 12 2009
DE : 6.0 usec
DS : 2
NS : 16
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 71.8000031
SFO1 : 400.1324008 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 63536

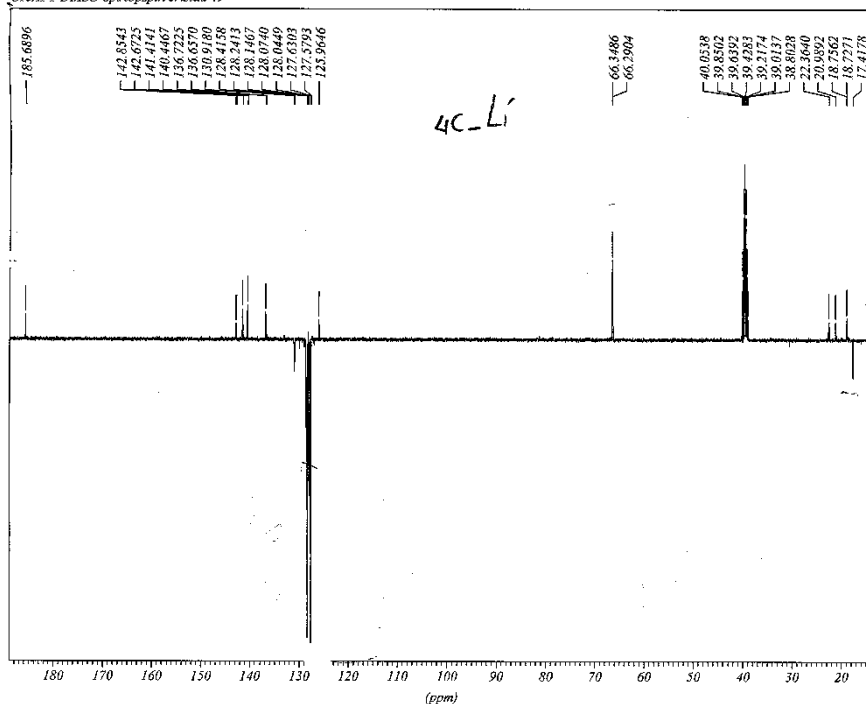
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1299793 MHz

*** 1D NMR Plot Parameters ***

Start : 8.63 ppm
Stop : -0.07 ppm
SR : -20.65 Hz
ppm_cm : 0.41
Hz_cm : 164.14

cm236-2b Li
C13APT DMSO opt/topspin cristau 49



*** Current Data Parameters ***

NAME : 236-2b
EXPNO : 262
PROCNO : 0

*** Acquisition Parameters ***

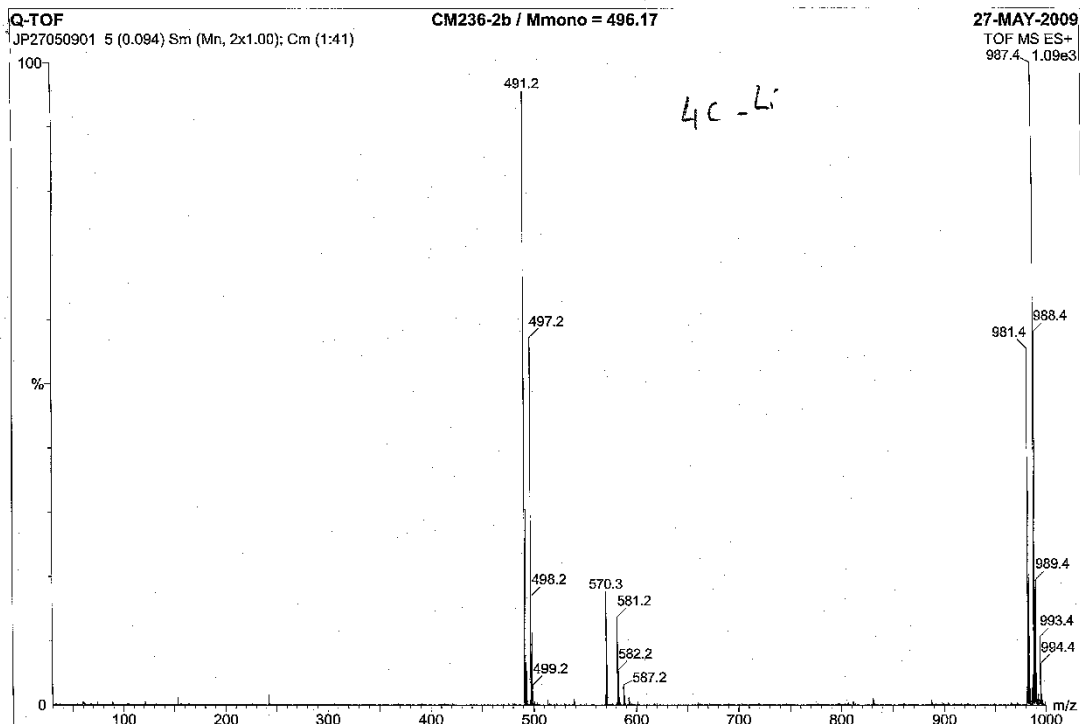
DATE_t : 14:33:42
DATE_d : Feb 12 2009
DE : 6.0 usec
DS : 4
NS : 1024
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 16384.0000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 63536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.60 Hz
PC : 1.40
SF : 100.6128193 MHz

*** 1D NMR Plot Parameters ***

Start : 188.98 ppm
Stop : 13.08 ppm
SR : 50.31 Hz
ppm_cm : 8.30
Hz_cm : 834.81



Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

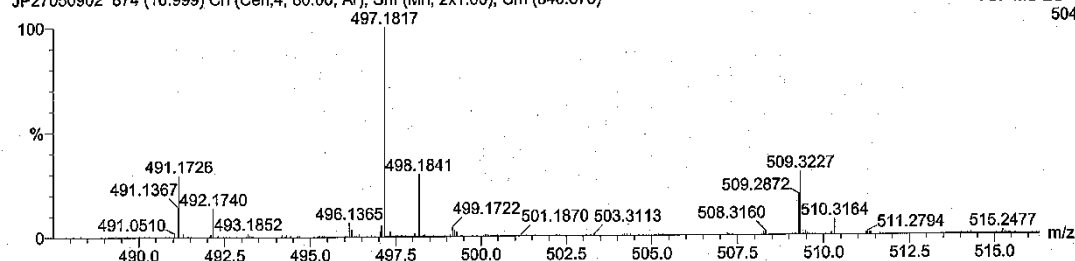
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

125 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Q-TOF
JP27050902 874 (16.999) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (840:875)

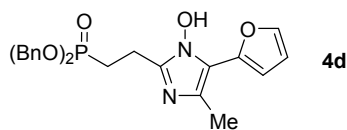
27-MAY-2009
TOF MS ES+
504



Minimum: -5.0
Maximum: 50.0

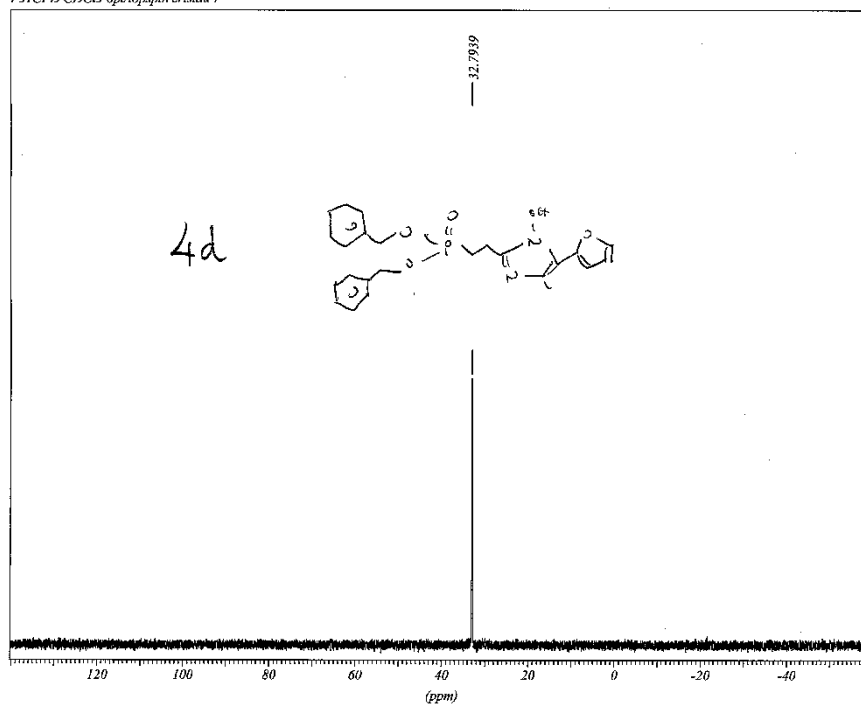
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
497.1817	497.1804	1.3	2.6	16.0	1	C25 H25 N5 O4 P Li
	497.1818	-0.1	-0.1	15.5	2	C27 H27 N2 O5 P Li
	497.1844	-2.7	-5.5	20.0	3	C30 H25 N3 O2 P Li

Dibenzyl (2-(5-(furan-2-carbonyl)-1-hydroxy-1H-imidazol-2-yl)ethyl)phosphonate (4d)



^1H NMR (400.13 MHz, CDCl_3) δ 2.11–2.19 (m, 2H), 2.30 (s, 3H), 2.89–2.98 (m, 2H), 4.91 (dd, $^2J_{\text{HH}} = 11.9$ Hz, $^3J_{\text{HP}} = 8.0$ Hz, 2H), 4.96 (dd, $^2J_{\text{HH}} = 11.9$ Hz, $^3J_{\text{HP}} = 9.3$ Hz, 2H), 6.37 (dd, $^3J_{\text{HH}} = 3.3$ Hz, $^3J_{\text{HH}} = 1.76$ Hz, 1H), 6.72 (d, $^3J_{\text{HH}} = 3.3$ Hz, 1H), 7.19–7.27 (m, 11H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 13.13 (s), 17.66 (s), 23.29 (d, $^1J_{\text{CP}} = 140.5$ Hz), 67.73 (d, $^2J_{\text{CP}} = 6.6$ Hz), 107.13 (s), 110.99 (s), 119.29 (s), 127.93 (s), 128.61 (s), 128.66 (s), 135.76 (d, $^3J_{\text{CP}} = 5.8$ Hz), 141.13 (s), 144.18 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 32.79 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{24}\text{H}_{26}\text{N}_2\text{O}_5\text{P}$ 453.1579, found: 453.1582.

cm297-f1
P31CPD CDCl3 op/loppspin cristau 7



*** Current Data Parameters ***

NAME : 297-f1
EXPNO : 260
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 08:28:57
DATE_d : Apr 08 2009
DE : 6.0 usec
DS : 4
NS : 16
NUC1 : ^{31}P
O1 : 6479.02 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 20642.5000000
SFO1 : 161.9820520 MHz
SW : 200.4391 ppm
SW_h : 32467.532 Hz
TD : 65536

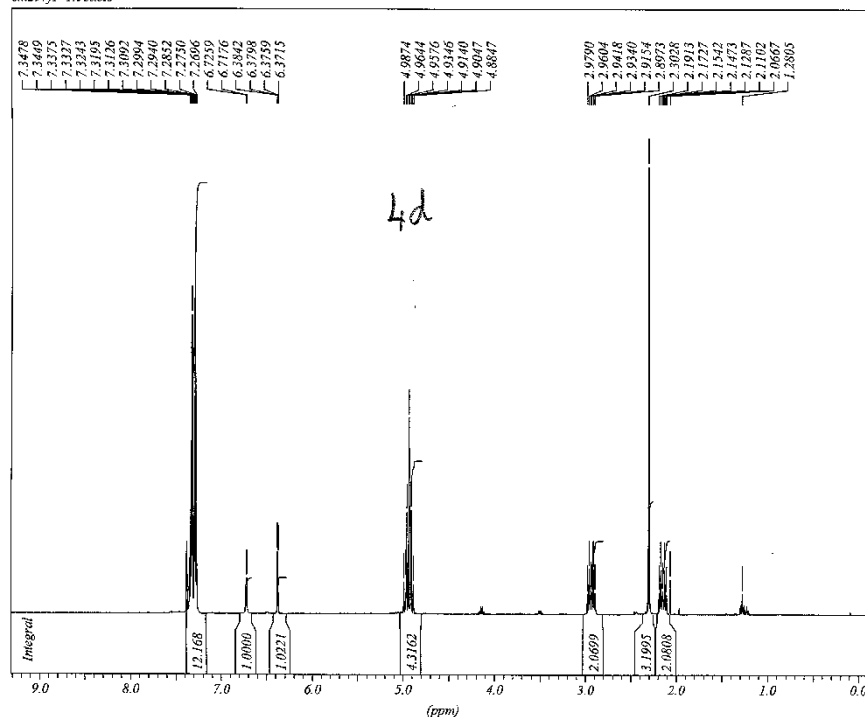
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 161.9755730 MHz

*** 1D NMR Plot Parameters ***

Start : 140.22 ppm
Stop : -60.22 ppm
SR : 0.01 Hz
ppm_cm : 9.46
Hz_cm : 1331.49

cm297f1 1h/cdel13



*** Current Data Parameters ***

NAME : 297-f1
EXPNO : 401
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 09:22:43
DATE_d : Apr 08 2009
DE : 6.0 usec
DS : 1
NS : 16
NUC1 : 1H
O1 : 2470.97 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 101.599985
SFO1 : 400.1324710 MHz
SW : 16.0203 ppm
SW_h : 6410.256 Hz
TD : 65536

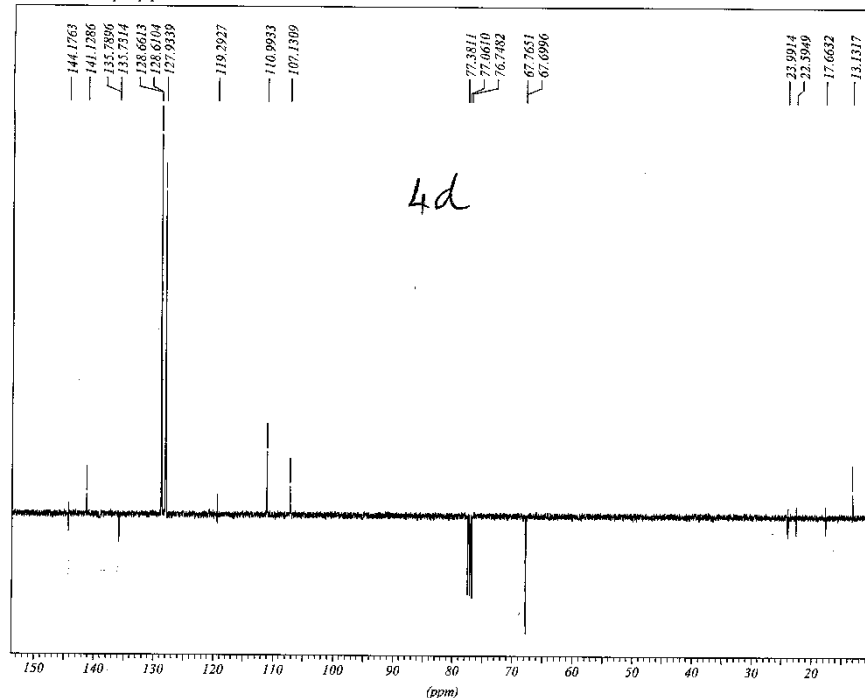
*** Processing Parameters ***

GB : 0.000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1306000 MHz

*** 1D NMR Plot Parameters ***

Start : 9.31 ppm
Stop : -0.18 ppm
SR : -0.00 Hz
ppm_cm : 0.45
Hz_cm : 179.22

cm297-f1
C13APT CDCl3 ap/10p/spin cristau 7



*** Current Data Parameters ***

NAME : 298-1
EXPNO : 262
PROCNO : 0

*** Acquisition Parameters ***

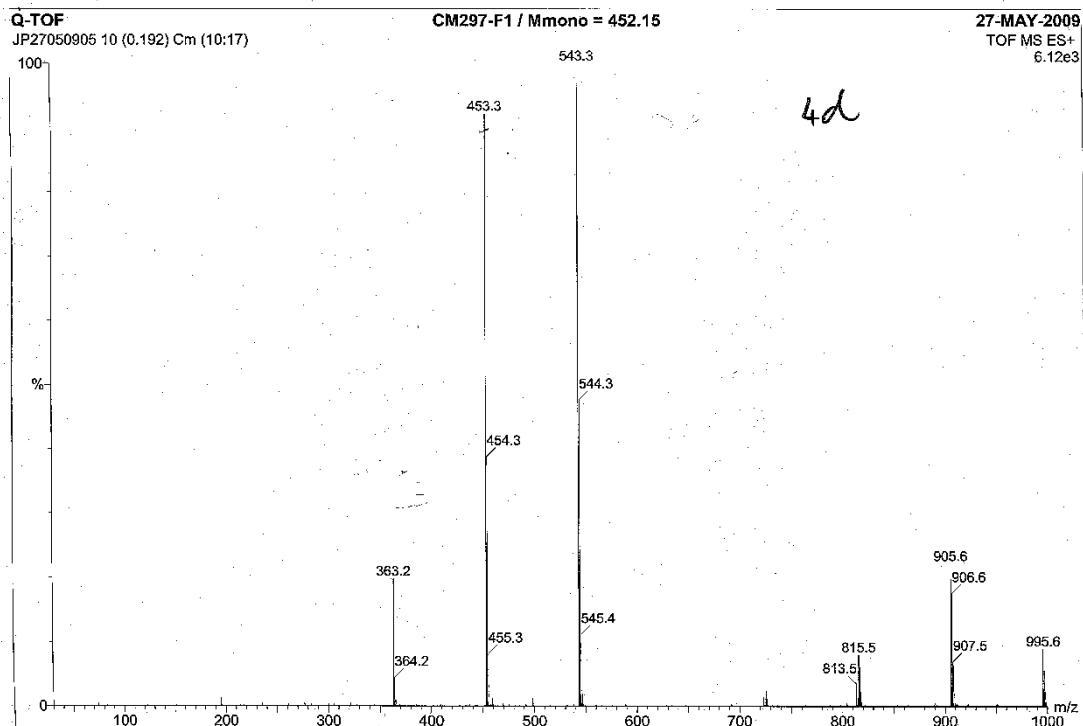
DATE_1 : 22:14:20
DATE_d : Apr 09 2009
DE : 6.0 usec
DS : 4
NS : 256
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 16384.000000
SFO1 : 100.6228298 MHz
SW : 238.5238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Start : 153.76 ppm
Stop : 9.41 ppm
SR : 0.60 Hz
ppm_cm : 6.81
Hz_cm : 685.06



Elemental Composition Report

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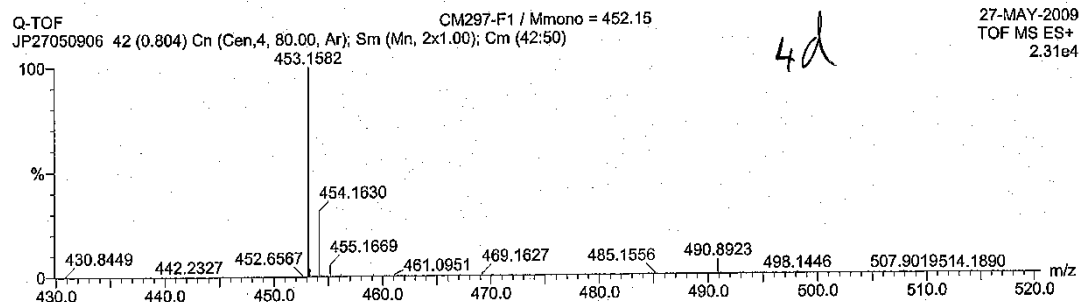
Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

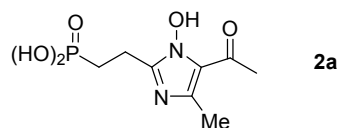
125 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)



Minimum: -5.0
Maximum: 3.0 5.0 50.0

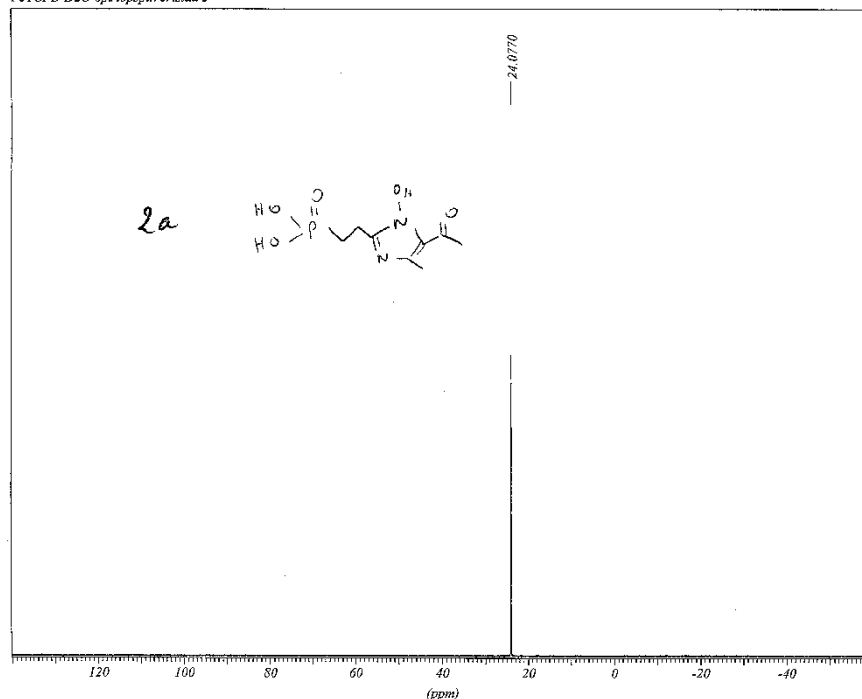
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
453.1582	453.1606	-2.4	-5.3	18.0	1	C27 H24 N3 O2 P
	453.1579	0.3	0.6	13.5	2	C24 H26 N2 O5 P
	453.1566	1.6	3.5	14.0	3	C22 H24 N5 O4 P

(2-(5-Acetyl-1-hydroxy-1H-imidazol-2-yl)ethyl)phosphonic acid (2a)



^1H NMR (400.13 MHz, D_2O) δ 2.00–2.09 (m, 2H), 2.41 (s, 3H), 2.48 (s, 3H), 3.07–3.14 (m, 2H). ^{13}C NMR (100.61 MHz, D_2O) δ 11.52 (s), 17.67 (d, $^2J_{\text{CP}} = 3.6$ Hz), 23.17 (d, $^1J_{\text{CP}} = 137.5$ Hz), 29.58 (s), 125.20 (s), 134.54 (s), 142.96 (d, $^3J_{\text{CP}} = 14.6$ Hz), 190.80 (s). ^{31}P NMR (161.97 MHz, D_2O) δ 24.08 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_8\text{H}_{14}\text{N}_2\text{O}_5\text{P}$ 249.0640, found 249.0637.

cm305-1
P31CPD D2O opt/topspin cristau 3



*** Current Data Parameters ***

NAME : 305-1
EXPNO : 254
PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 04:20:09
DATE_d : May 25 2009
DE : 6.0 usec
DS : 4
NS : 16
NUC1 : ^{31}P
O1 : 6479.02 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Swuch-1H Z-C
RG : 20642.5000000
SFO1 : 161.9820520 MHz
SW : 200.4391 ppm
SW_h : 32467.532 Hz
TD : 65536

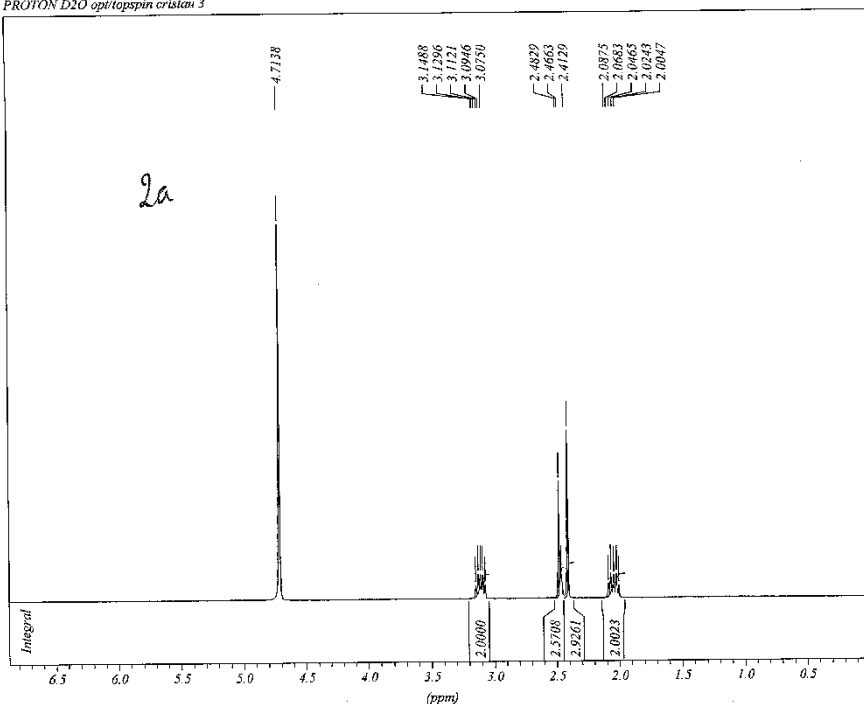
*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 161.9755730 MHz

*** 1D NMR Plot Parameters ***

Start : 140.22 ppm
Stop : -60.22 ppm
SR : 0.01 Hz
ppm_cm : 9.46
Hz_cm : 1531.49

cm305-1
 • PROTON D2O opt/topspin cristau 3



*** Current Data Parameters ***

NAME : 305-1
 EXPNO : 255
 PROCNO : 0

*** Acquisition Parameters ***

DATE_t : 04:24:27
 DATE_d : May 25 2009
 DE : 6.0 usec
 DS : 2
 NS : 16
 NUC1 : 1H
 O1 : 2400.78 Hz
 O2 : 2470.97 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-C
 RG : 90.5000000
 SFO1 : 400.1324008 MHz
 SW : 13.9775 ppm
 SW_h : 5592.841 Hz
 TD : 65536

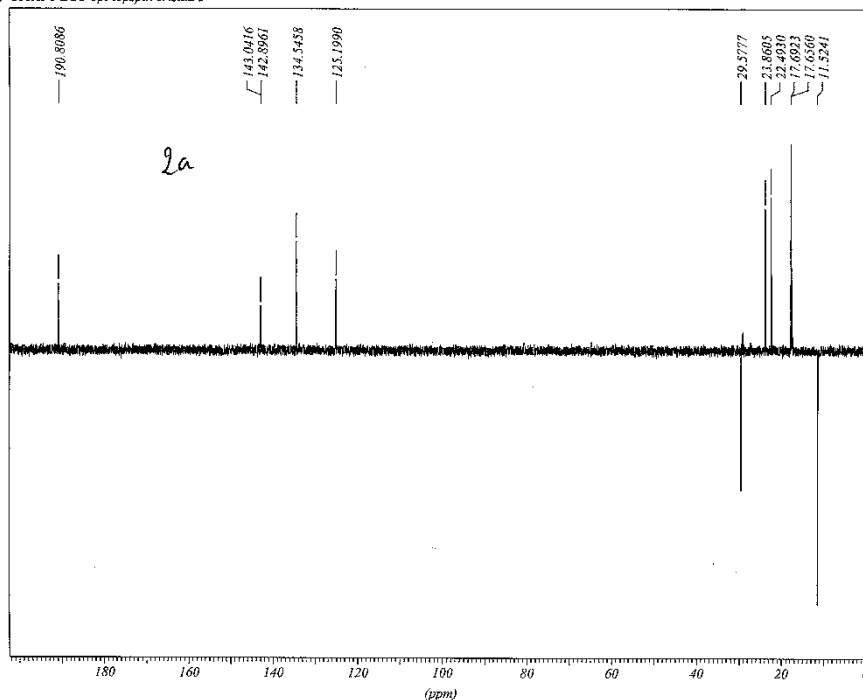
*** Processing Parameters ***

GB : 0.0000000
 LB : 0.10 Hz
 PC : 1.00
 SF : 400.1300029 MHz

*** 1D NMR Plot Parameters ***

Start : 6.88 ppm
 Stop : -0.03 ppm
 SR : 2.95 Hz
 ppm_cm : 0.33
 Hz_cm : 130.30

cm305-1
 • C13APT D2O opt/topspin cristau 3



*** Current Data Parameters ***

NAME : 305-1
 EXPNO : 256
 PROCNO : 0

*** Acquisition Parameters ***

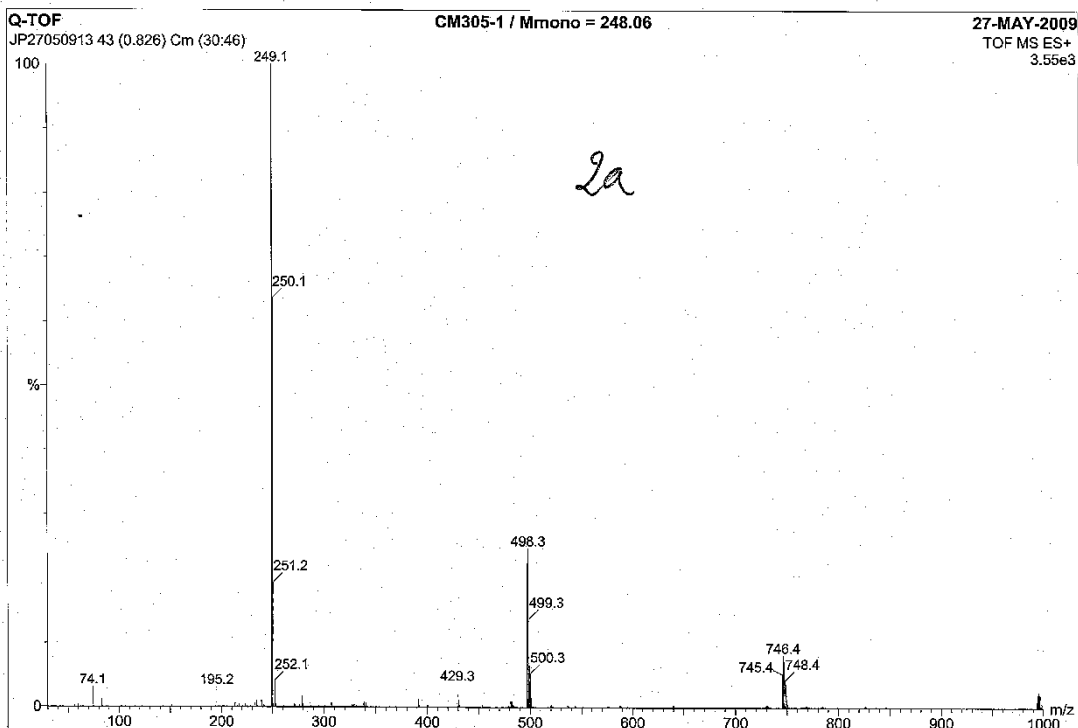
DATE_t : 16:48:21
 DATE_d : May 25 2009
 DE : 6.0 usec
 DS : 4
 NS : 1024
 NUC1 : 13C
 O1 : 10060.80 Hz
 O2 : 1600.52 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-C
 RG : 26008.0000000
 SFO1 : 100.6228298 MHz
 SW : 238.3238 ppm
 SW_h : 23980.815 Hz
 TD : 65536

*** Processing Parameters ***

GB : 0.0000000
 LB : 1.00 Hz
 PC : 1.40
 SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Start : 202.44 ppm
 Stop : -1.62 ppm
 SR : 0.00 Hz
 ppm_cm : 9.63
 Hz_cm : 968.44



Elemental Composition Report

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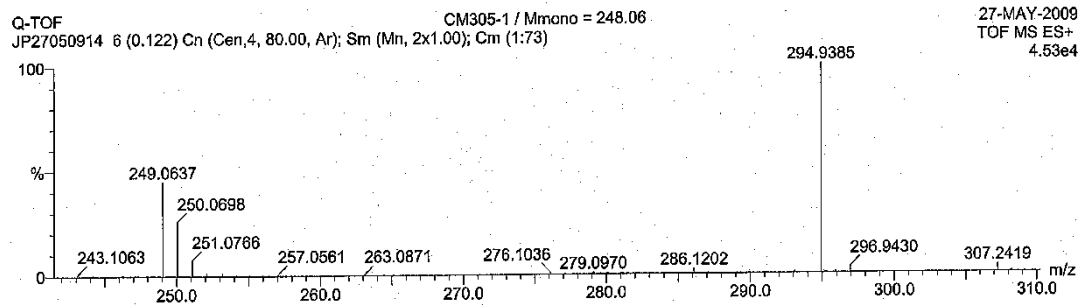
Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

77 formula(e) evaluated with 2 results within limits (up to 50 closest results for each mass)

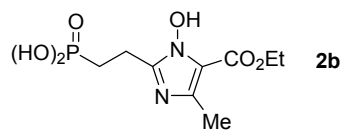


Minimum:
Maximum:

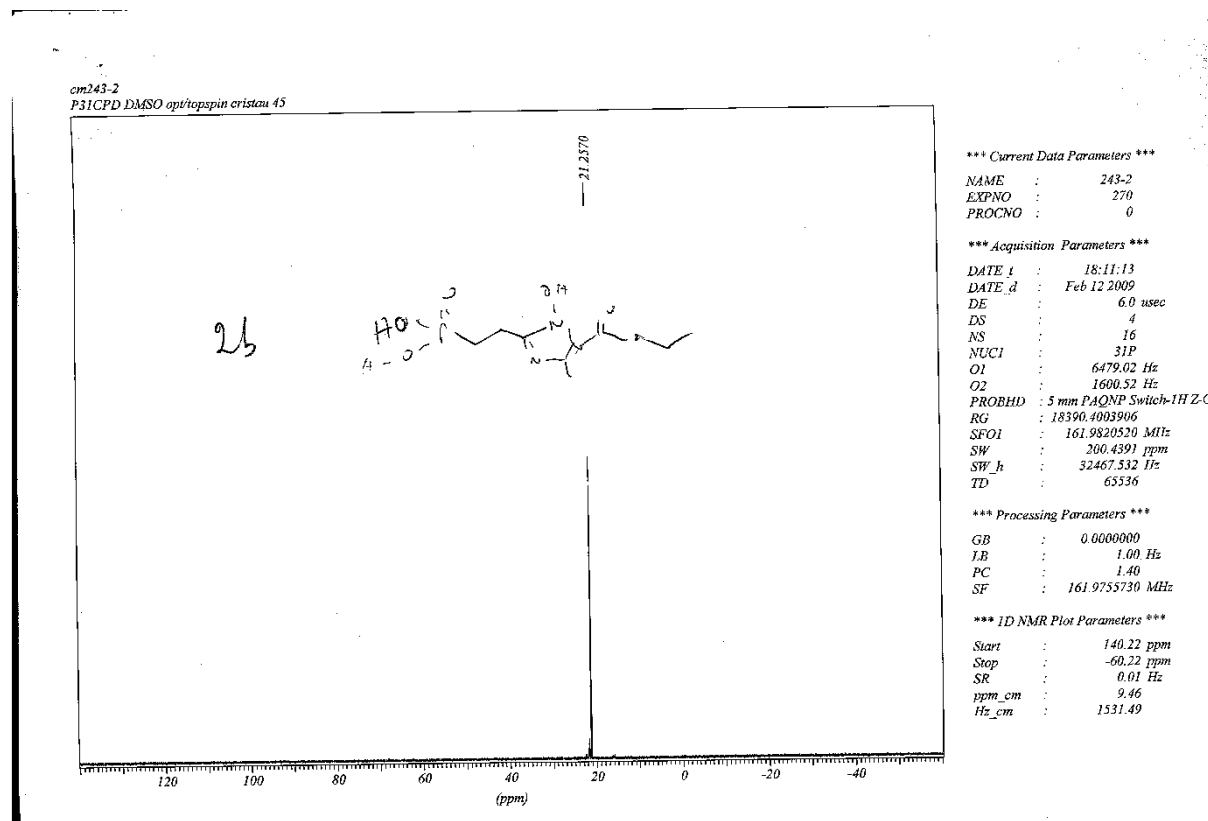
3.0 5.0 -5.0
50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
249.0637	249.0640	-0.3	-1.3	3.5	1	C8 H14 N2 O5 P
	249.0627	1.0	4.0	4.0	2	C6 H12 N5 O4 P

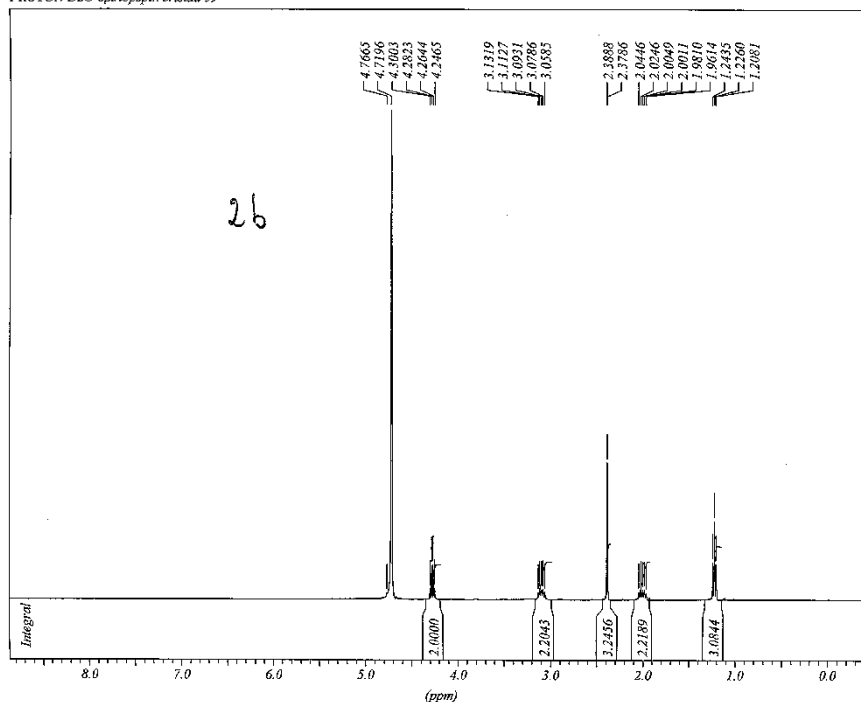
(2-(5-(Ethoxycarbonyl)-1-hydroxy-1H-imidazol-2-yl)ethyl)phosphonic acid (2b)



^1H NMR (400.13 MHz, D_2O): δ 1.23 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.96–2.04 (m, 2H), 2.38 (s, 3H), 3.05–3.14 (m, 2H), 4.27 (q, $^3J_{\text{HH}} = 7.0$ Hz, 2H). ^{13}C NMR (100.61 MHz, $\text{DMSO}-d_6$): δ 8.13 (s), 10.51 (s), 15.09 (d, $^2J_{\text{CP}} = 3.7$ Hz), 20.06 (d, $^1J_{\text{CP}} = 136.8$ Hz), 59.96 (s), 115.32 (s), 132.00 (s), 140.96 (d, $^3J_{\text{CP}} = 13.9$ Hz), 156.08 (s). ^{31}P NMR (161.97 MHz, D_2O) δ 25.67 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_9\text{H}_{16}\text{N}_2\text{O}_6\text{P}$ 279.0746, found: 279.0739.



cm243-2
PROTON D2O opt/topspin cristau 59



*** Current Data Parameters ***

NAME : 243-2
EXPNO : 260
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 06:40:28
DATE_d : Feb 13 2009
DE : 6.0 usec
DS : 2
NS : 32
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 80.5999985
SFO1 : 400.1324008 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 65536

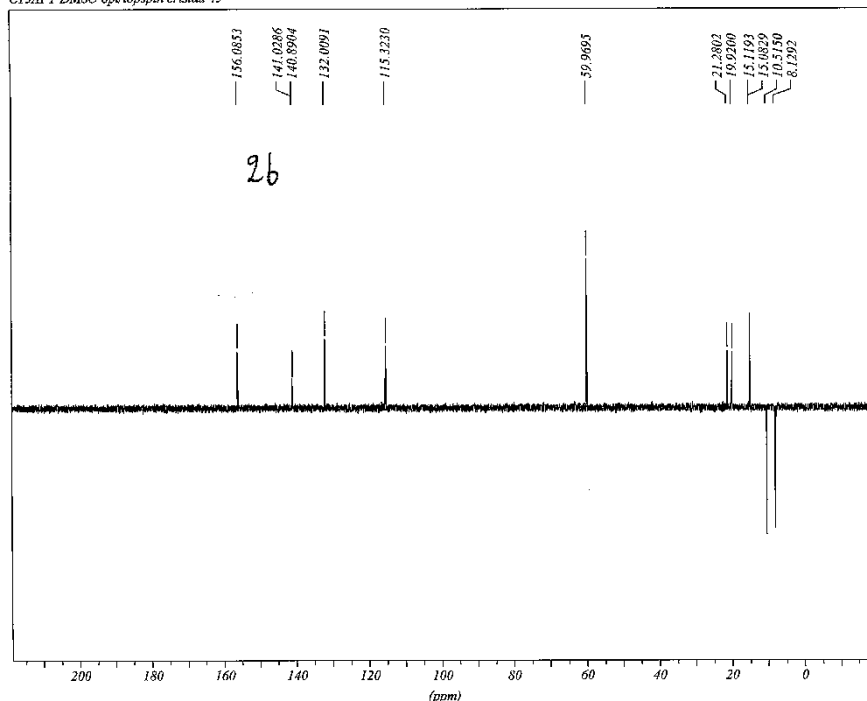
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1299972 MHz

*** 1D NMR Plot Parameters ***

Start : 8.87 ppm
Stop : -0.50 ppm
SR : -2.77 Hz
ppm_cm : 0.44
Hz_cm : 176.71

cm243-2
C13APT DMSO opt/topspin cristau 45



*** Current Data Parameters ***

NAME : 243-2
EXPNO : 272
PROCNO : 0

*** Acquisition Parameters ***

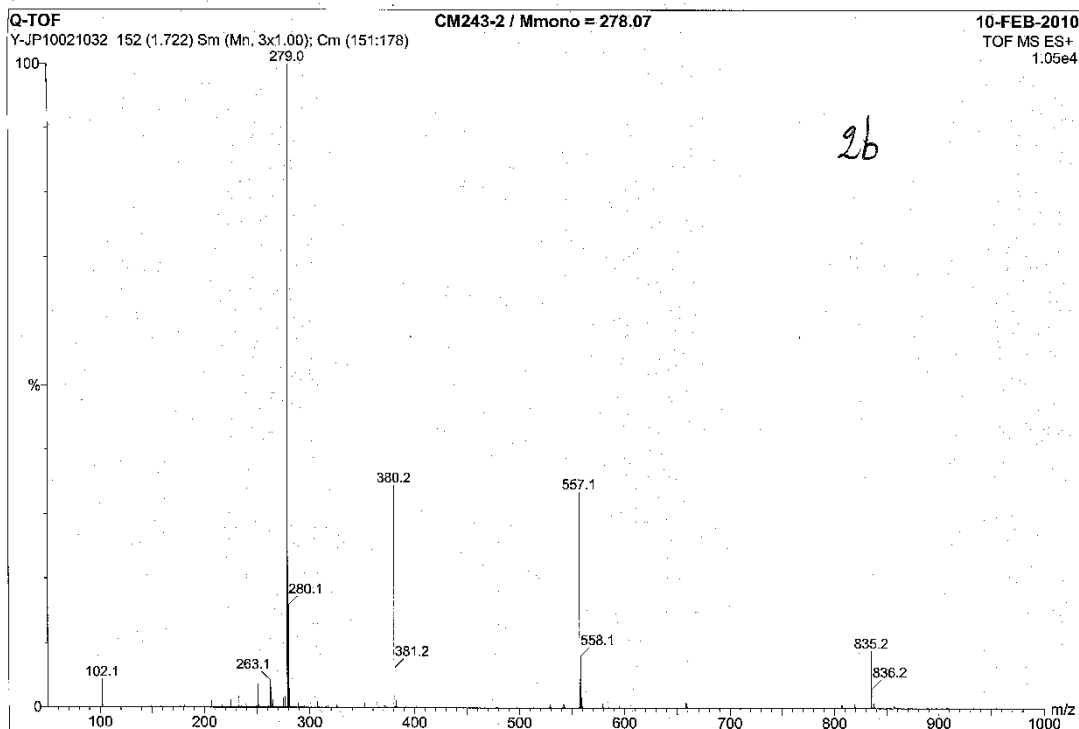
DATE_1 : 19:17:31
DATE_d : Feb 12 2009
DE : 6.0 usec
DS : 4
NS : 1024
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 26008.0000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6128193 MHz

*** 1D NMR Plot Parameters ***

Start : 218.67 ppm
Stop : -19.68 ppm
SR : 50.31 Hz
ppm_cm : 11.24
Hz_cm : 1131.17



Elemental Composition Report

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Single Mass Analysis

Tolerance = 3.0 mDa / DBE: min = -10.0, max = 100.0

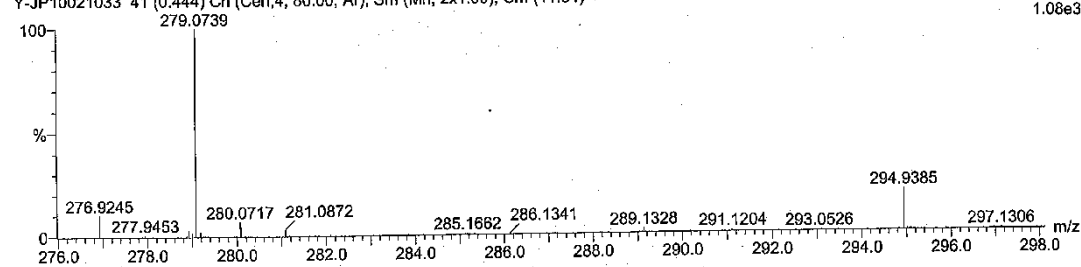
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

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

Q-TOF
Y-JP10021033 41 (0.444) Cn (Cen,4, 80.00; Ar); Sm (Mn, 2x1.00); Cm (41:54)
CM243-2 / Mmono = 278.07

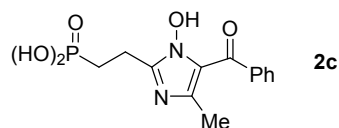
10-FEB-2010
TOF MS ES+
1.08e3



Minimum: -10.0
Maximum: 100.0

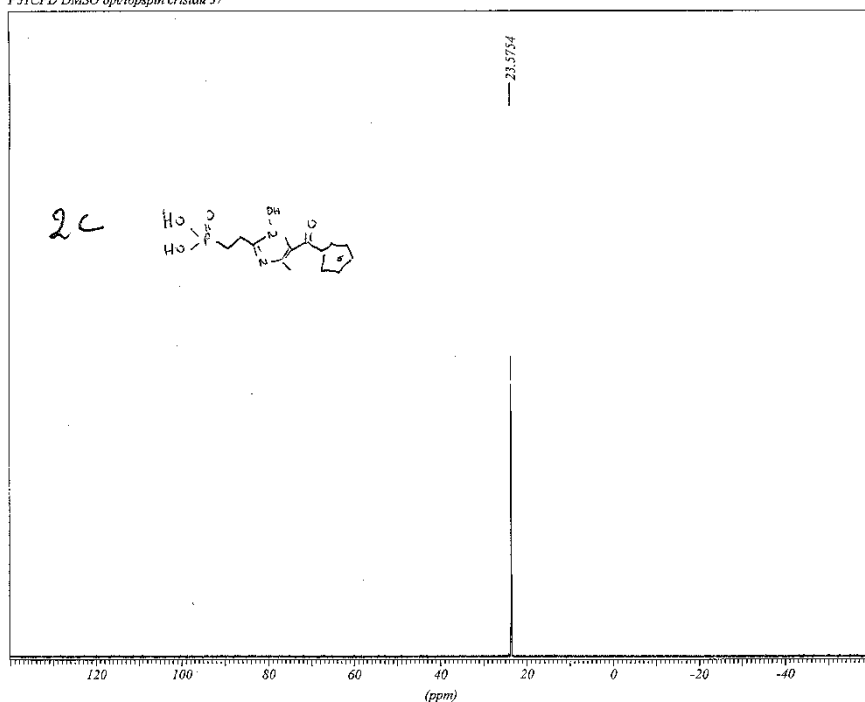
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
279.0739	279.0719	2.0	7.1	-1.0	1	C6 H18 N O9 P
	279.0733	0.6	2.3	4.0	2	C7 H14 N5 O5 P
	279.0746	-0.7	-2.5	3.5	3	C9 H16 N2 O6 P

(2-(5-Benzoyl-1-hydroxy-1H-imidazol-2-yl)ethyl)phosphonic acid (2c)



^1H NMR (400.13 MHz, DMSO- d_6) δ 1.98–2.06 (m, 2H), 2.13 (s, 3H), 2.96–3.03 (m, 2H), 7.56–7.66 (m, 2H), 7.68–7.76 (m, 1H), 7.76–7.82 (m, 2H). ^{13}C NMR (100.61 MHz, DMSO- d_6) δ 14.00 (s), 18.75 (s), 24.78 (d, $^1J_{\text{CP}} = 135.4$ Hz), 125.11 (s), 128.46 (s), 129.03 (s), 132.90 (s), 137.82 (s), 144.51 (d, $^3J_{\text{CP}} = 15.4$ Hz), 184.73 (s). ^{31}P NMR (161.97 MHz, DMSO- d_6) δ 23.58 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_5\text{P}$ 311.0797, found: 311.0797.

cm315-solid
*P31CPD DMSO opt/topspin cristau 37



*** Current Data Parameters ***

NAME : 315-so-1
EXPNO : 251
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 22:45:07
DATE_d : May 14 2009
DE : 6.0 usec
DS : 4
NS : 32
NUC1 : ^{31}P
O1 : 6479.02 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 20642.500000
SFO1 : 161.9820520 MHz
SW : 200.4391 ppm
SW_h : 32467.532 Hz
TD : 65536

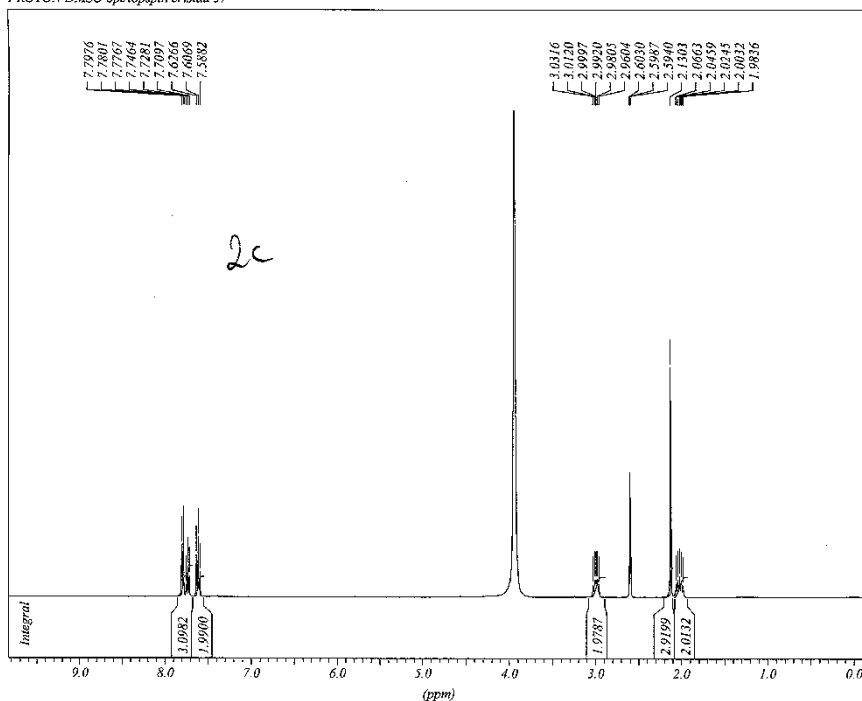
*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
PC : 1.40
SF : 161.9755730 MHz

*** 1D NMR Plot Parameters ***

Start : 140.22 ppm
Stop : -60.22 ppm
SR : 0.01 Hz
ppm_cm : 9.46
Hz_cm : 1531.49

cm315-solide
-PROTON DMSO opt/topspin cristau 37



*** Current Data Parameters ***

NAME : 315-so-1
EXPNO : 252
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 22:50:35
DATE_d : May 14 2009
DE : 6.0 usec
DS : 2
NS : 32
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 80.599983
SFO1 : 400.1324008 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 65536

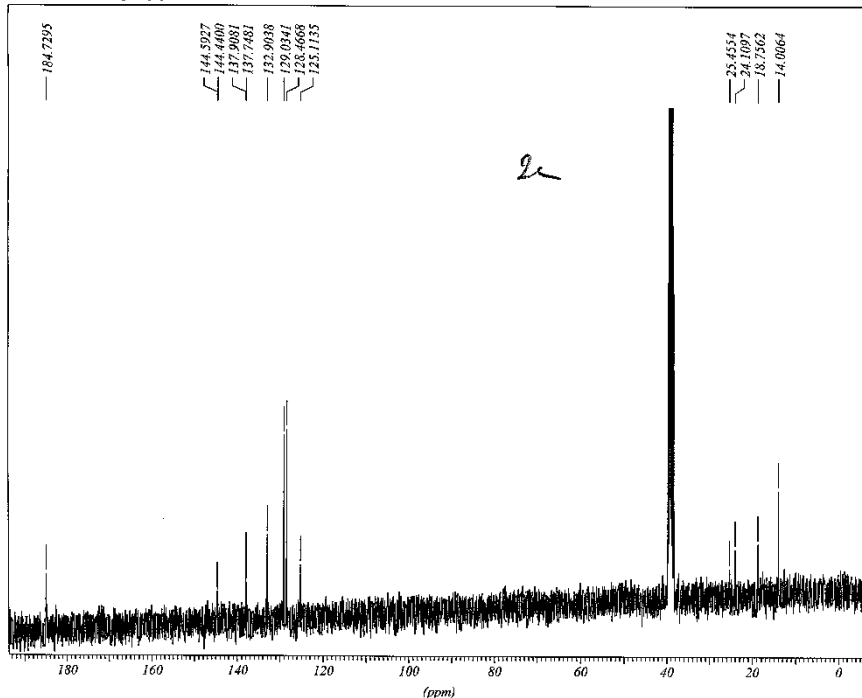
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1299648 MHz

*** 1D NMR Plot Parameters ***

Start : 9.83 ppm
Stop : -0.20 ppm
SR : -35.20 Hz
ppm_cm : 0.47
Hz_cm : 189.29

cm315-solide
C13CPD32 DMSO opt/topspin cristau 37



*** Current Data Parameters ***

NAME : 315-so-1
EXPNO : 254
PROCNO : 0

*** Acquisition Parameters ***

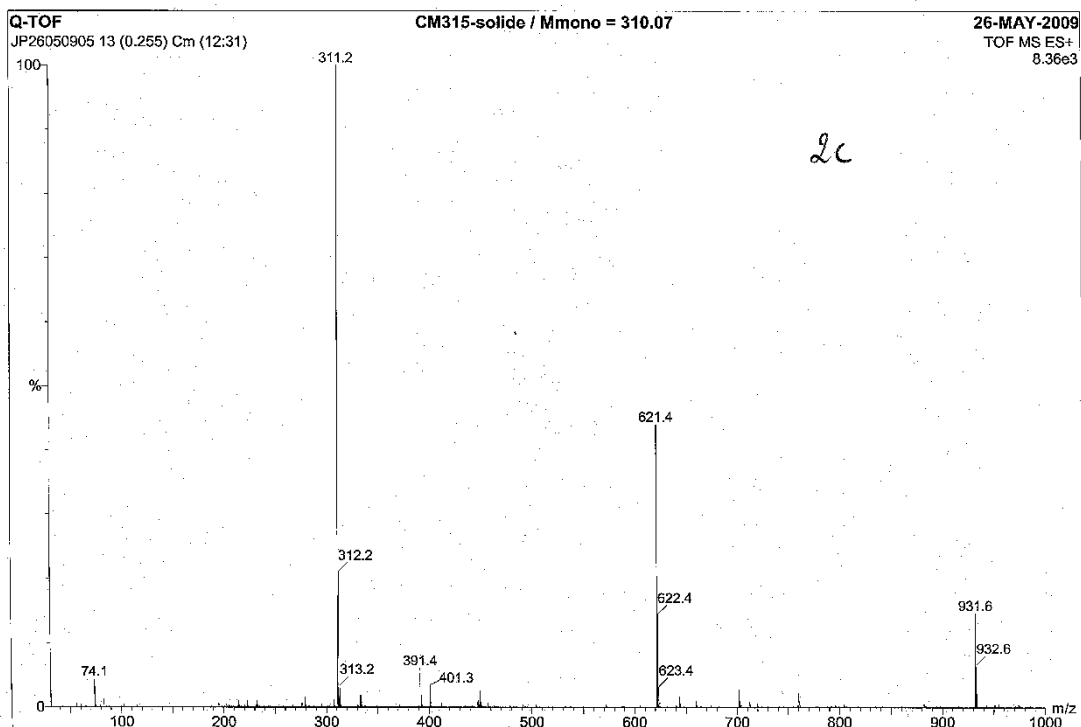
DATE_1 : 06:32:06
DATE_d : May 15 2009
DE : 6.0 usec
DS : 2
NS : 128
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 1448.1999512
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6128193 MHz

*** 1D NMR Plot Parameters ***

Start : 193.68 ppm
Stop : -7.70 ppm
SR : 50.31 Hz
ppm_cm : 9.50
Hz_cm : 955.70



Elemental Composition Report

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Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

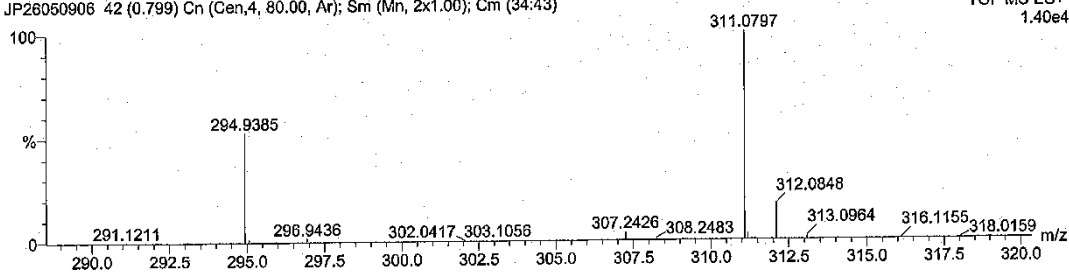
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

98 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Q-TOF
JP26050906 42 (0.799) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (34:43)

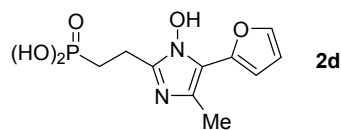
26-MAY-2009
TOF MS ES+
1.40e4



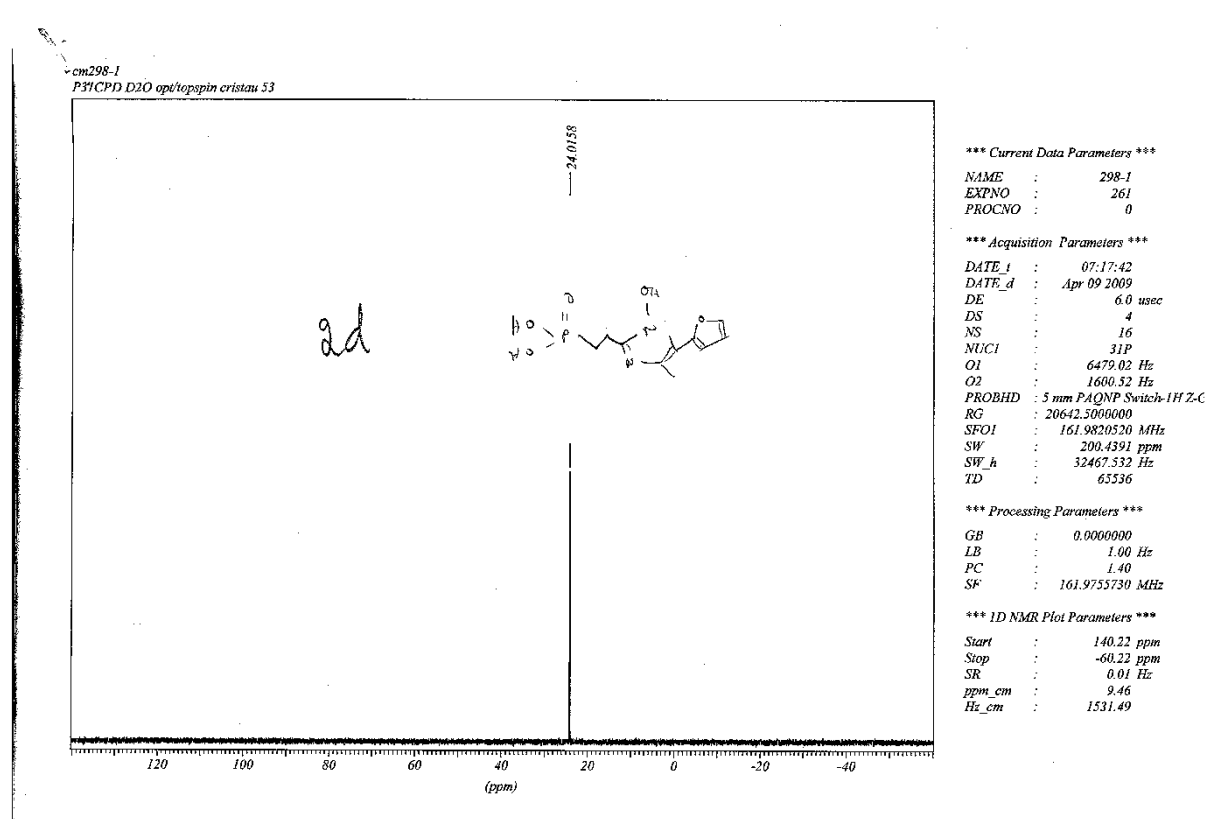
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula				
311.0797	311.0797	0.0	0.0	7.5	1	C13	H16	N2	O5	P
	311.0824	-2.7	-8.6	12.0	2	C16	H14	N3	O2	P
	311.0783	1.4	4.4	8.0	3	C11	H14	N5	O4	P

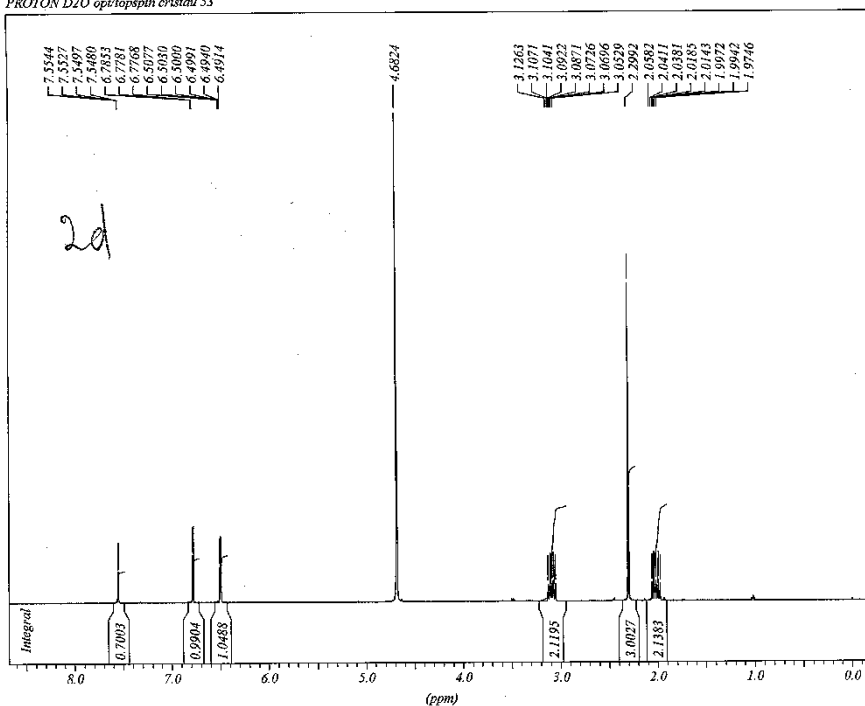
(2-(5-(Furan-2-carbonyl)-1-hydroxy-1H-imidazol-2-yl)ethyl)phosphonic acid (2d)



^1H NMR (400.13 MHz, D_2O) δ 1.97–2.06 (m, 2H), 2.30 (s, 3H), 3.05–3.12 (m, 2H), 6.49 (dd, $^3J_{\text{HH}} = 3.1$ Hz, $^3J_{\text{HH}} = 1.0$ Hz, 1H), 6.78 (d, $^3J_{\text{HH}} = 3.1$ Hz, 1H), 7.55 (d, $^3J_{\text{HH}} = 1.0$ Hz, 1H). ^{13}C NMR (100.61 MHz, D_2O) δ 9.57 (s), 17.71 (d, $^2J_{\text{CP}} = 3.7$ Hz), 23.61 (d, $^1J_{\text{CP}} = 136.1$ Hz), 111.04 (s), 111.39 (s), 119.29 (s), 123.32 (s), 141.23 (d, $^3J_{\text{CP}} = 14.6$ Hz), 144.10 (s), 145.38 (s). ^{31}P NMR (161.97 MHz, D_2O) δ 24.01 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_5\text{P}$ 273.0640, found 273.0640.



cm298-1
PROTON D2O op/topspin cristau 53



*** Current Data Parameters ***

NAME : 298-1
EXPNO : 260
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 07:14:36
DATE_d : Apr 09 2009
DE : 6.0 usec
DS : 2
NS : 16
NUC1 : 1H
O1 : 2470.97 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 322.500000
SFO1 : 400.1324710 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 65536

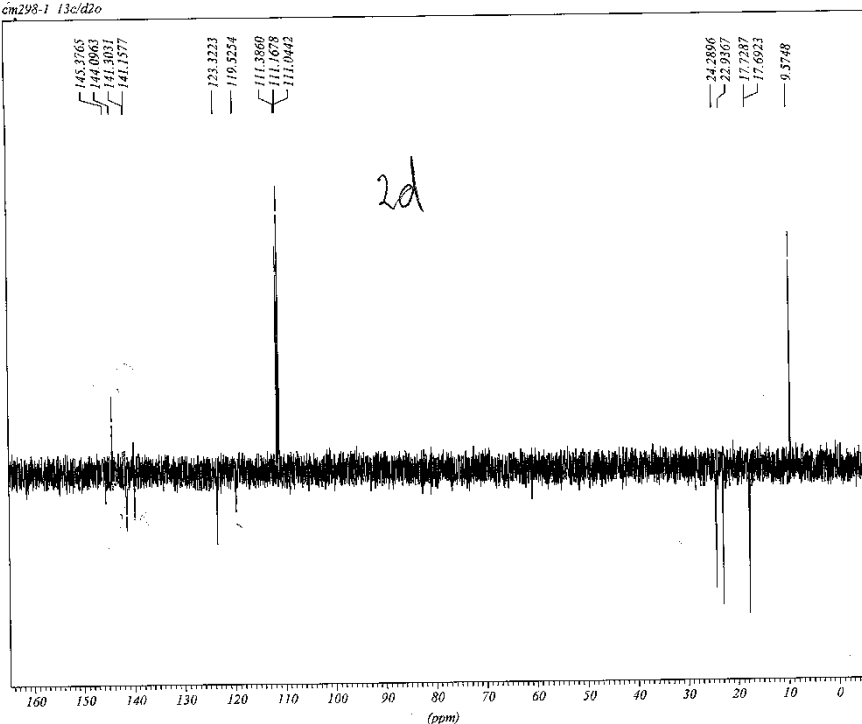
*** Processing Parameters ***

GB : 0.0000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1300104 MHz

*** 1D NMR Plot Parameters ***

Start : 8.68 ppm
Stop : -0.22 ppm
SR : 10.43 Hz
ppm_cm : 0.42
Hz_cm : 168.03

cm298-1 13c/d2o



*** Current Data Parameters ***

NAME : 298-1
EXPNO : 401
PROCNO : 0

*** Acquisition Parameters ***

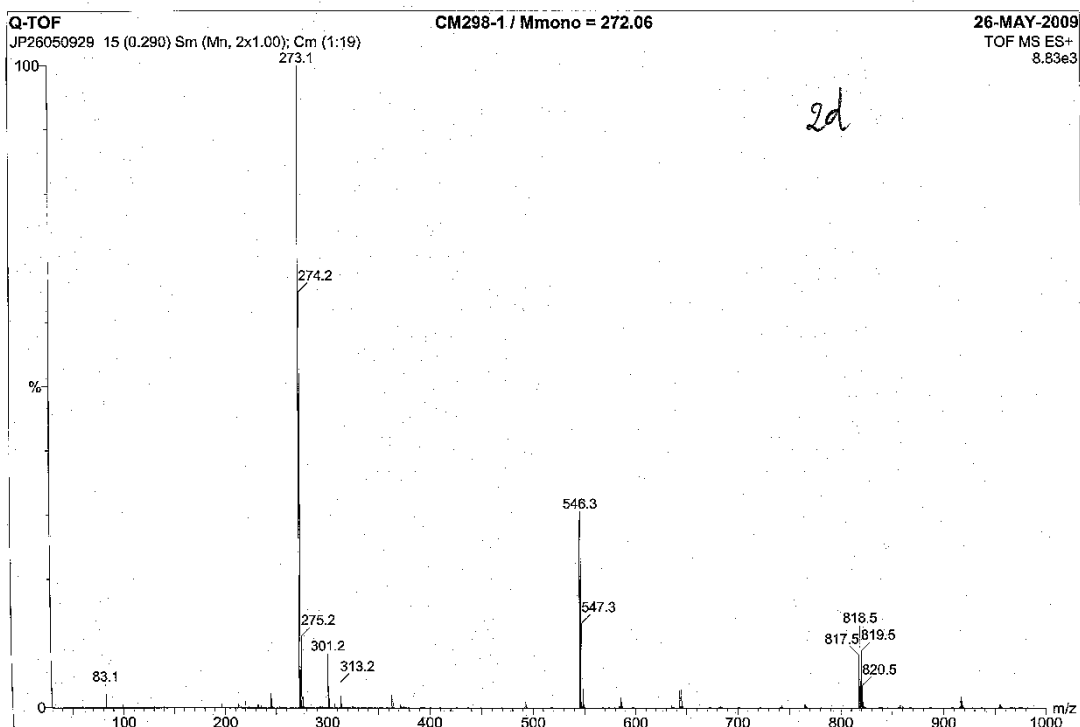
DATE_1 : 22:55:49
DATE_d : May 24 2009
DE : 6.0 usec
DS : 1
NS : 1024
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.52 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 32768.000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.0000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Start : 164.85 ppm
Stop : -6.78 ppm
SR : 0.00 Hz
ppm_cm : 8.10
Hz_cm : 814.55



Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

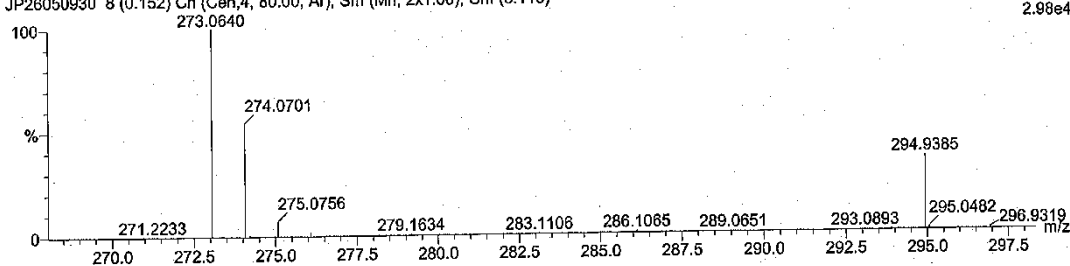
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

83 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Q-TOF
JP26050930 8 (0.152) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (8:116)
CM298-1 / Mmono = 272.06

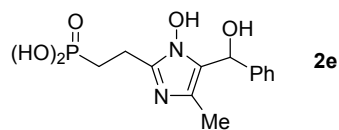
26-MAY-2009
TOF MS ES+
2.98e4



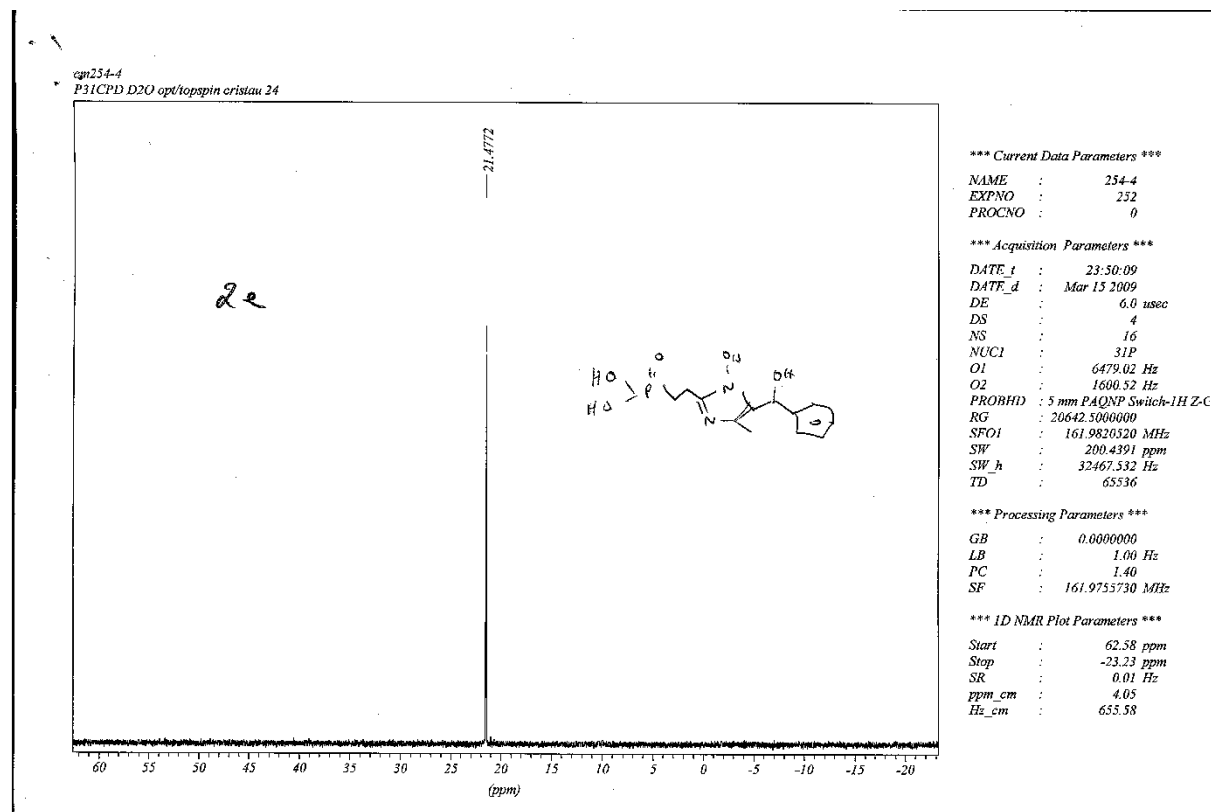
Minimum: -5.0
Maximum: 3.0 5.0 50.0

Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
273.0640	273.0667	-2.7	-9.9	10.0	1	C13 H12 N3 O2 P
	273.0640	0.0	-0.1	5.5	2	C10 H14 N2 O5 P
	273.0627	1.3	4.8	6.0	3	C8 H12 N5 O4 P

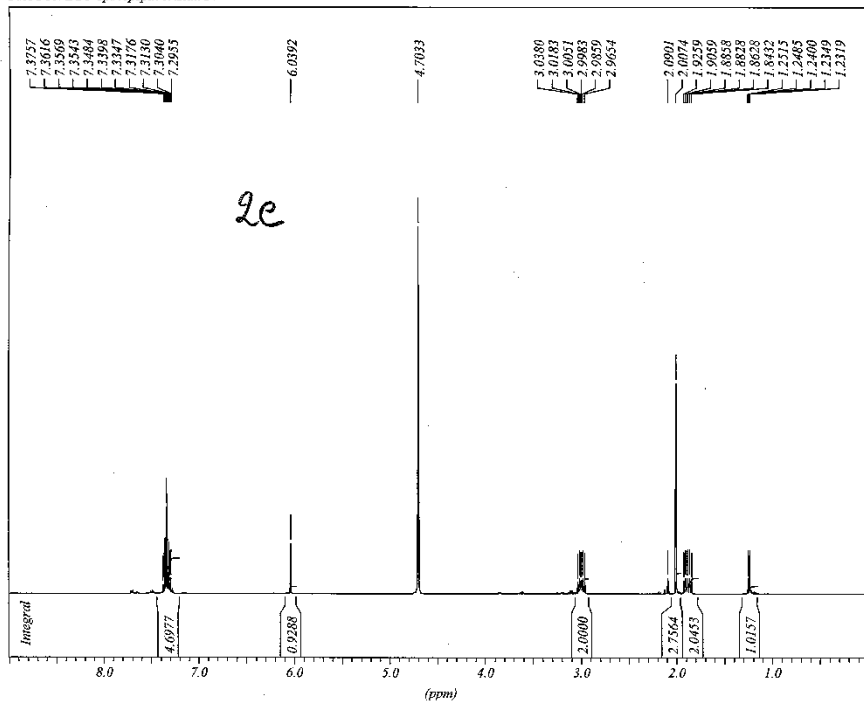
(2-(1-Hydroxy-5-(hydroxy(phenyl)methyl)-1H-imidazol-2-yl)ethyl)phosphonic acid (2e)



^1H NMR (400.13 MHz, D_2O) δ 1.21–1.27 (m, 1H), 1.84–1.92 (m, 2H), 2.01 (s, 3H), 2.96–3.03 (m, 2H), 6.04 (s, 1H), 7.29–7.38 (m, 5H). ^{13}C NMR (100.61 MHz, D_2O) δ 9.05 (s), 17.94 (d, $^2J_{\text{CP}} = 3.6$ Hz), 24.38 (d, $^1J_{\text{CP}} = 134.6$ Hz), 65.11 (s), 123.52 (s), 126.02 (s), 127.50 (s), 128.23 (s), 128.78 (s), 139.37 (s), 140.71 (d, $^3J_{\text{CP}} = 14.6$ Hz). ^{31}P NMR (161.97 MHz, D_2O) δ 21.47 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{13}\text{H}_{18}\text{N}_2\text{O}_5\text{P}$ 313.0953, found 313.0955.



cpm254-4
PROTON D2O opt/topspin cristau 24



*** Current Data Parameters ***

NAME : 254-4
EXPNO : 251
PROCNO : 0

*** Acquisition Parameters ***

DATE_1 : 23:46:41
DATE_d : Mar 15 2009
DE : 6.0 usec
DS : 2
NS : 16
NUC1 : 1H
O1 : 2400.78 Hz
O2 : 2470.97 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 256.000000
SFO1 : 400.1324008 MHz
SW : 13.9775 ppm
SW_h : 5592.841 Hz
TD : 65536

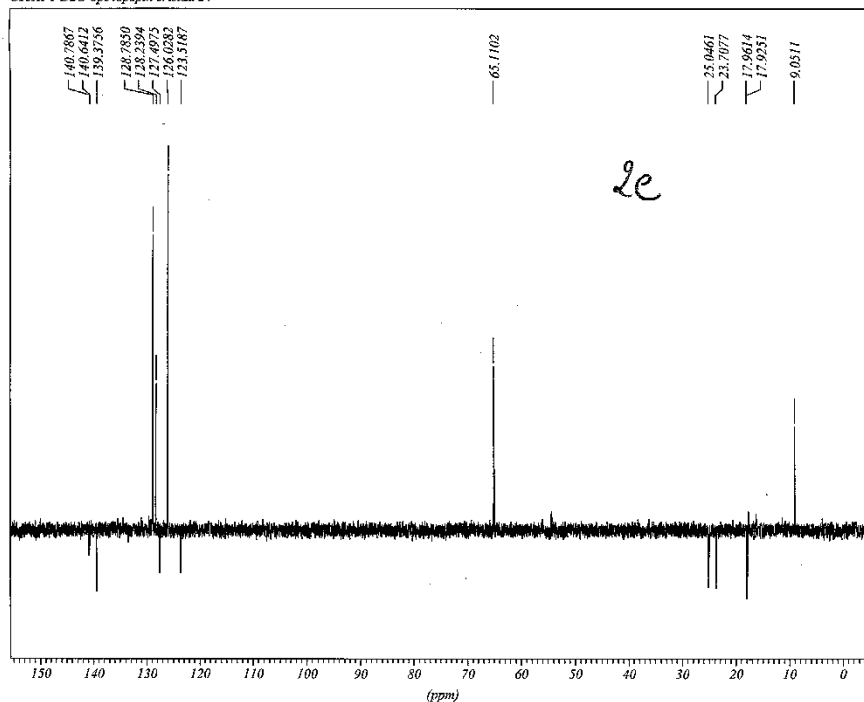
*** Processing Parameters ***

GB : 0.000000
LB : 0.10 Hz
PC : 1.00
SF : 400.1300000 MHz

*** 1D NMR Plot Parameters ***

Start : 9.00 ppm
Stop : -0.07 ppm
SR : 0.00 Hz
ppm_cm : 0.43
Hz_cm : 171.23

cpm254-4
C13APT D2O opt/topspin cristau 24



*** Current Data Parameters ***

NAME : 254-4
EXPNO : 253
PROCNO : 0

*** Acquisition Parameters ***

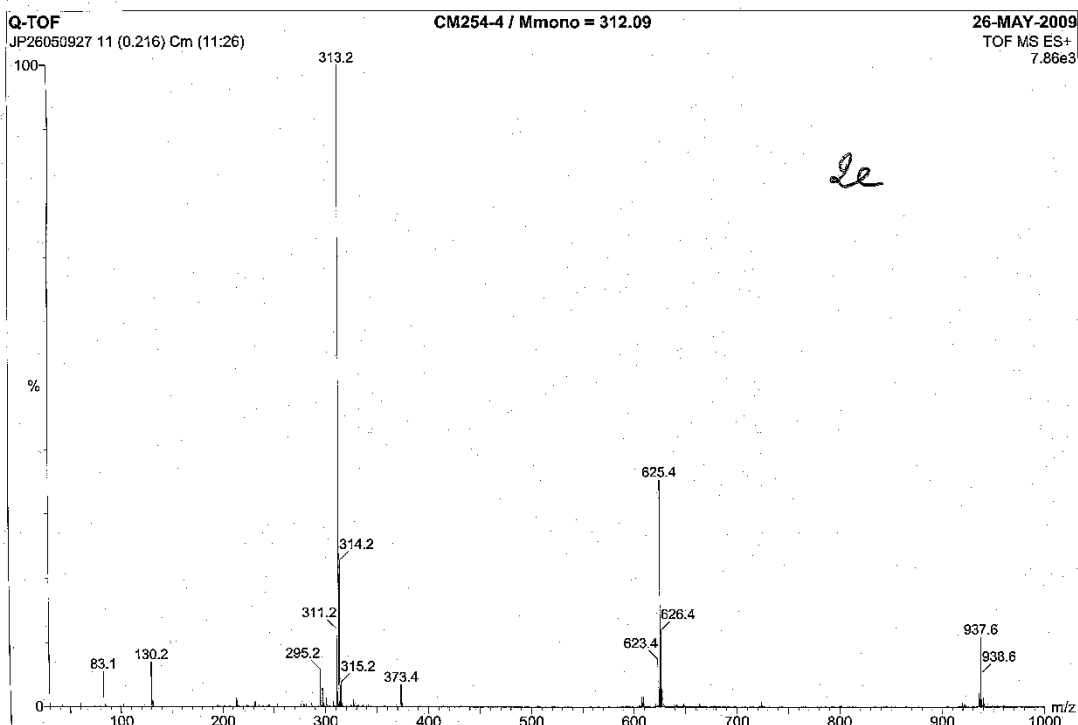
DATE_1 : 12:14:21
DATE_d : Mar 16 2009
DE : 6.0 usec
DS : 4
NS : 1024
NUC1 : 13C
O1 : 10060.80 Hz
O2 : 1600.32 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-C
RG : 26008.000000
SFO1 : 100.6228298 MHz
SW : 238.3238 ppm
SW_h : 23980.815 Hz
TD : 65536

*** Processing Parameters ***

GB : 0.000000
LB : 1.00 Hz
PC : 1.40
SF : 100.6127690 MHz

*** 1D NMR Plot Parameters ***

Start : 155.55 ppm
Stop : -6.17 ppm
SR : 0.00 Hz
ppm_cm : 7.63
Hz_cm : 767.50



Elemental Composition Report

Page 1

Single Mass Analysis (displaying only valid results)

Tolerance = 3.0 mDa / DBE: min = -5.0, max = 50.0

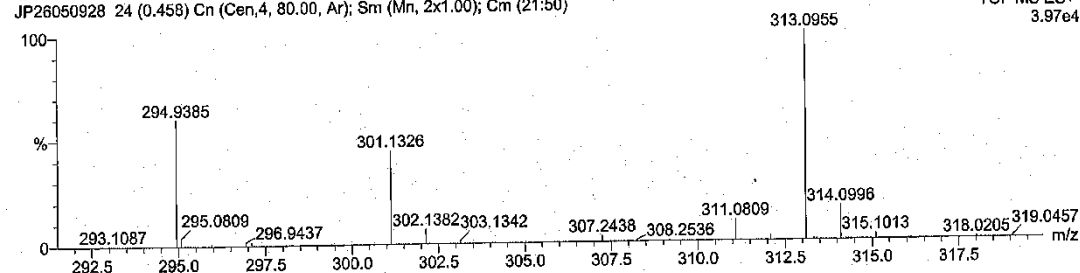
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions

96 formula(e) evaluated with 3 results within limits (up to 50 closest results for each mass)

Q-TOF
JP26050928 24 (0.458) Cn (Cen,4, 80.00, Ar); Sm (Mn, 2x1.00); Cm (21:50)

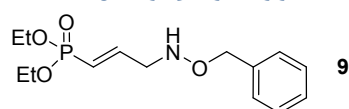
26-MAY-2009
TOF MS ES+
3.97e4



Minimum: -5.0
Maximum: 50.0

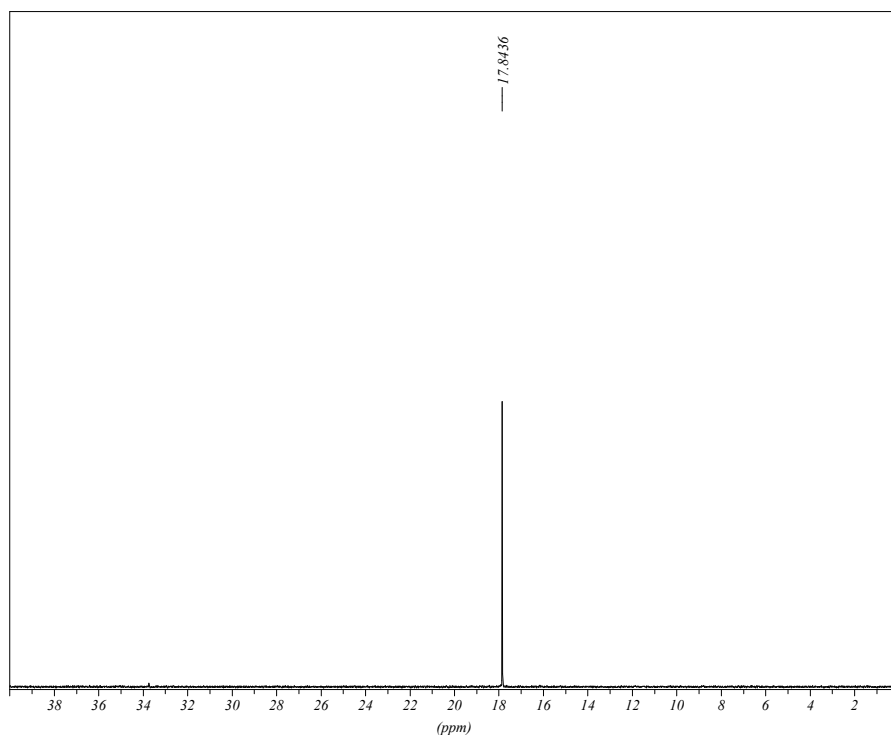
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
313.0955	313.0953	0.2	0.5	6.5	1	C13 H18 N2 O5 P X
	313.0940	1.5	4.8	7.0	2	C11 H16 N5 O4 P
	313.0980	-2.5	-8.0	11.0	3	C16 H16 N3 O2 P

Diethyl (E)-3-((benzyloxy)amino)prop-1-en-1-yl)phosphonate (9)



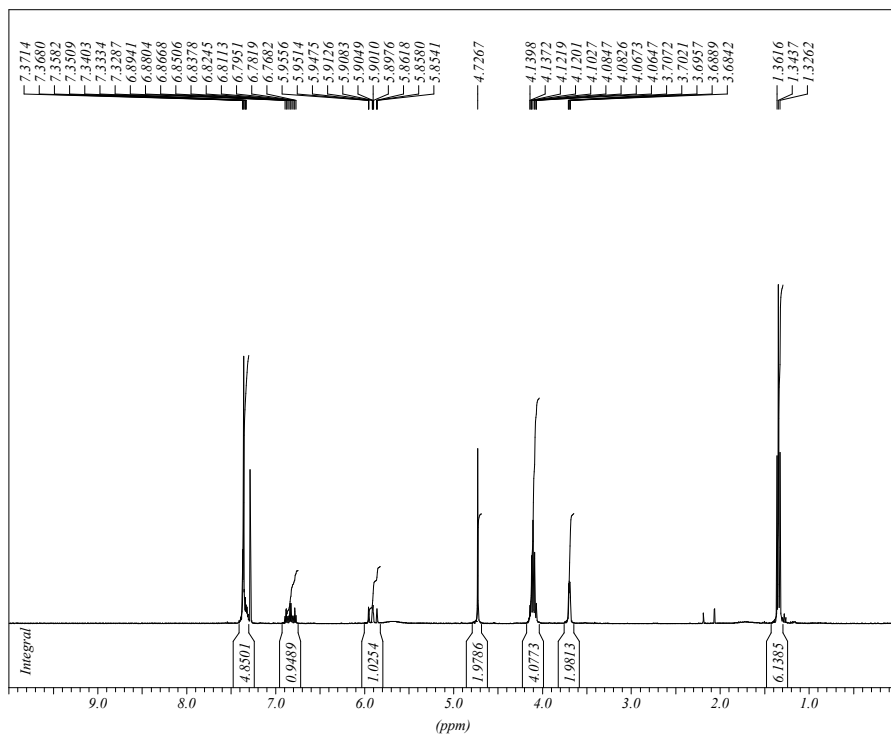
^1H NMR (400.13 MHz, CDCl_3) δ 1.34 (t, $^3J_{\text{HH}} = 7.1$ Hz, 6H), 3.68–3.71 (m, 2H), 4.06–4.14 (m, 4H), 4.73 (s, 2H), 5.85–5.96 (ddt, $^2J_{\text{PH}} = 20.3$ Hz, $^3J_{\text{HH}} = 17.2$ Hz, $^4J_{\text{HH}} = 3.2$ Hz, 1H), 6.77–6.89 (ddt, $^3J_{\text{PH}} = 27.8$ Hz, $^3J_{\text{HH}} = 17.4$ Hz, $^3J_{\text{HH}} = 5.5$ Hz, 1H), 7.33–7.37 (m, 5H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.36 (d, $^3J_{\text{CP}} = 6.4$ Hz), 54.21 (d, $^3J_{\text{CP}} = 23.2$ Hz), 61.77 (d, $^2J_{\text{CP}} = 5.6$ Hz), 77.33 (s), 119.11 (d, $^1J_{\text{CP}} = 187.8$ Hz), 127.96 (s), 128.42 (s), 137.53 (s), 148.55 (d, $^2J_{\text{CP}} = 4.9$ Hz). ^{31}P NMR (161.97 MHz, CDCl_3) δ 17.84 (s).

BG047-P1 P31CPD CDCl_3 opt/topspin am2n1 52



*** Current Data Parameters ***

NAME : 532
 EXPNO : 32
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 21:00:31
 DATE_d : Jun 28 2011
 EXP : PS done
 NS : 16
 O1 : 6479.02 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 20642.500000
 SFO1 : 161.9820520 MHz
 SOLVENT : CDCl_3
 SW : 200.4391 ppm
 TD : 65536
 TE : 673.2 K
 *** Processing Parameters ***
 GB : 0.000000
 LB : 0.50 Hz
 SF : 161.9755730 MHz
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 40.01 ppm
 Stop : 0.01 ppm
 SR : 0.01 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 530
EXPNO : 30\
PROCNO : 1

*** Acquisition Parameters ***

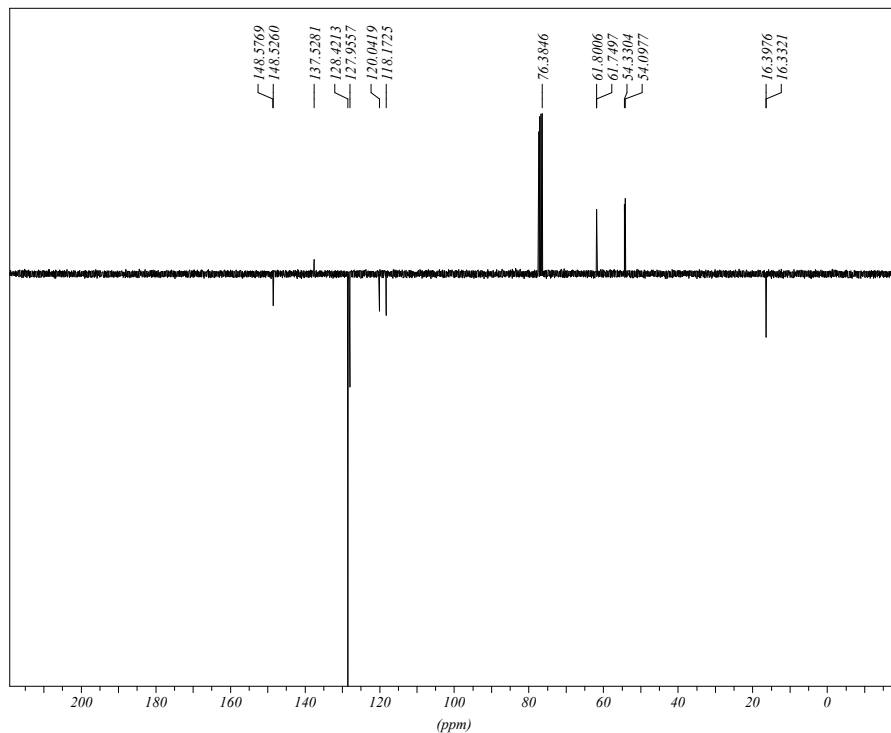
DATE_t : 20:54:14
DATE_d : Jun 28 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 143.6999969
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 13.9775 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 533
EXPNO : 33\
PROCNO : 1

*** Acquisition Parameters ***

DATE_t : 22:00:58
DATE_d : Jun 28 2011
EXP : C13APT
NS : 1026
OI : 10060.80 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 16384.0000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm
TD : 65536
TE : 673.2 K

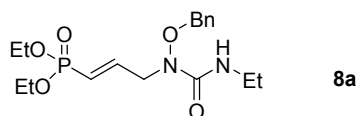
*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

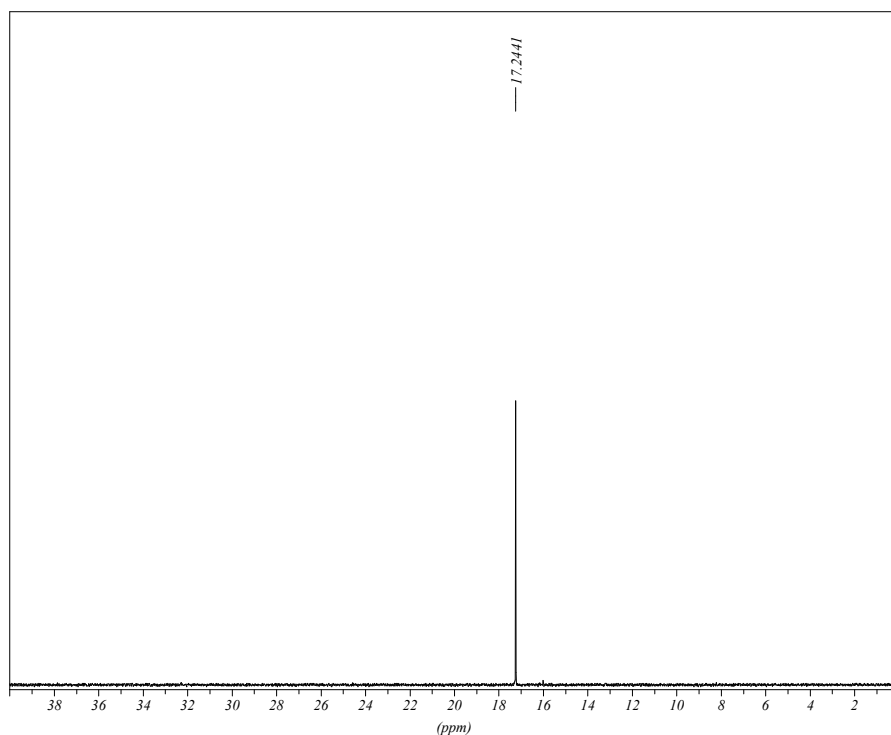
Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

(E)-Diethyl (3-(1-(benzyloxy)-3-ethylureido)prop-1-en-1-yl)phosphonate (8a)



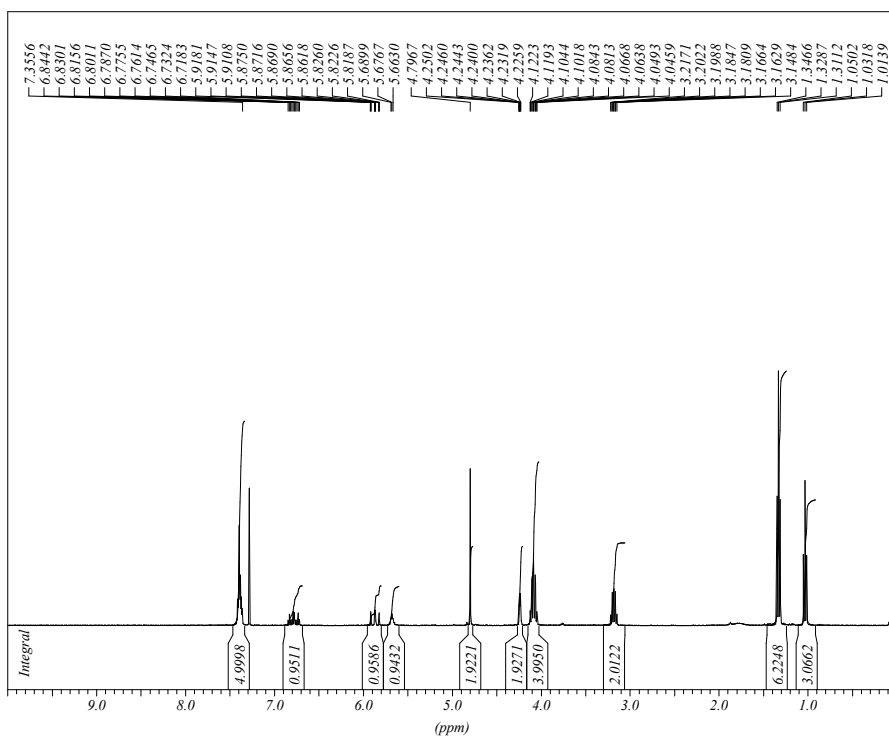
^1H NMR (400.13 MHz, CDCl_3) δ 1.03 (t, $^3J_{\text{HH}} = 7.3$ Hz, 6H), 1.33 (t, $^3J_{\text{HH}} = 7.2$ Hz, 3H), 3.15–3.22 (m, 2H), 4.05–4.12 (m, 4H), 4.23–4.25 (m, 2H), 4.80 (s, 2H), 5.68 (t, $^3J_{\text{HH}} = 5.5$ Hz, 1H), 5.87 (ddt, $^2J_{\text{PH}} = 19.6$ Hz, $^3J_{\text{HH}} = 17.2$ Hz, $^4J_{\text{HH}} = 1.4$ Hz, 1H), 6.72–6.84 (m, 1H), 7.36–7.41 (m, 5H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 15.07 (s), 16.33 (d, $^3J_{\text{CP}} = 6.4$ Hz), 34.94 (s), 54.22 (d, $^3J_{\text{CP}} = 24.5$ Hz), 61.80 (d, $^2J_{\text{CP}} = 5.6$ Hz), 77.41 (s), 120.21 (d, $^1J_{\text{CP}} = 186.9$ Hz), 128.79 (s), 129.01 (s), 129.31 (s), 135.14 (s), 146.89 (d, $^2J_{\text{CP}} = 5.0$ Hz), 159.62 (s). ^{31}P NMR (161.97 MHz, CDCl_3): δ 17.24 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{28}\text{N}_2\text{O}_5\text{P}$ 371.1740, found: 371.1736.

SM272.2-B1 P31CPD CDCl_3 opt/topspin am2n1 1



*** Current Data Parameters ***

NAME : 506
EXPNO : 6
PROCNO : 1
*** Acquisition Parameters ***
DATE_1 : 06:59:19
DATE_d : Aug 23 2011
EXP : PS done
NS : 16
OI : 6479.02 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 20642.5000000
SFO1 : 161.9820520 MHz
SOLVENT : CDCl_3
SW : 200.4391 ppm
TD : 65536
TE : 673.2 K
*** Processing Parameters ***
GB : 0.0000000
LB : 0.50 Hz
SF : 161.9755730 MHz
TI :
*** 1D NMR Plot Parameters ***
Start : 40.01 ppm
Stop : 0.01 ppm
SR : 0.01 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 520
EXPNO : 20\
PROCNO : 1

*** Acquisition Parameters ***

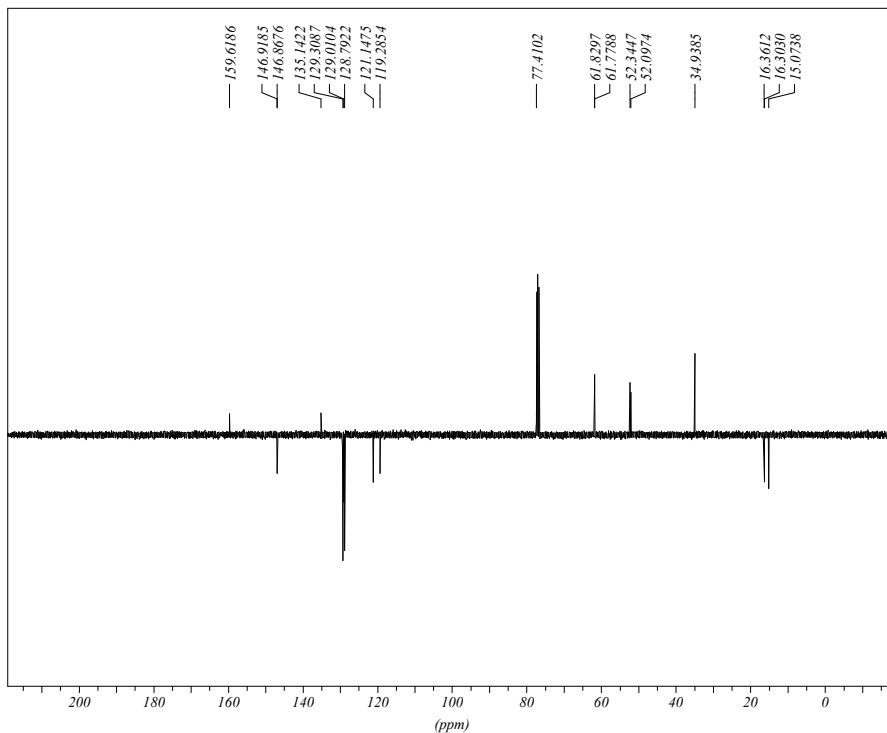
DATE_1 : 08:28:57
DATE_d : Aug 24 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 128.000000
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 13.9775 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 522
EXPNO : 22\
PROCNO : 1

*** Acquisition Parameters ***

DATE_1 : 14:14:07
DATE_d : Aug 24 2011
EXP : C13APT
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 23170.500000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm
TD : 65536
TE : 673.2 K

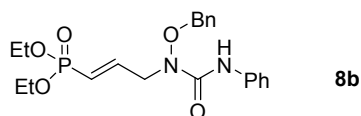
*** Processing Parameters ***

GB : 0.000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

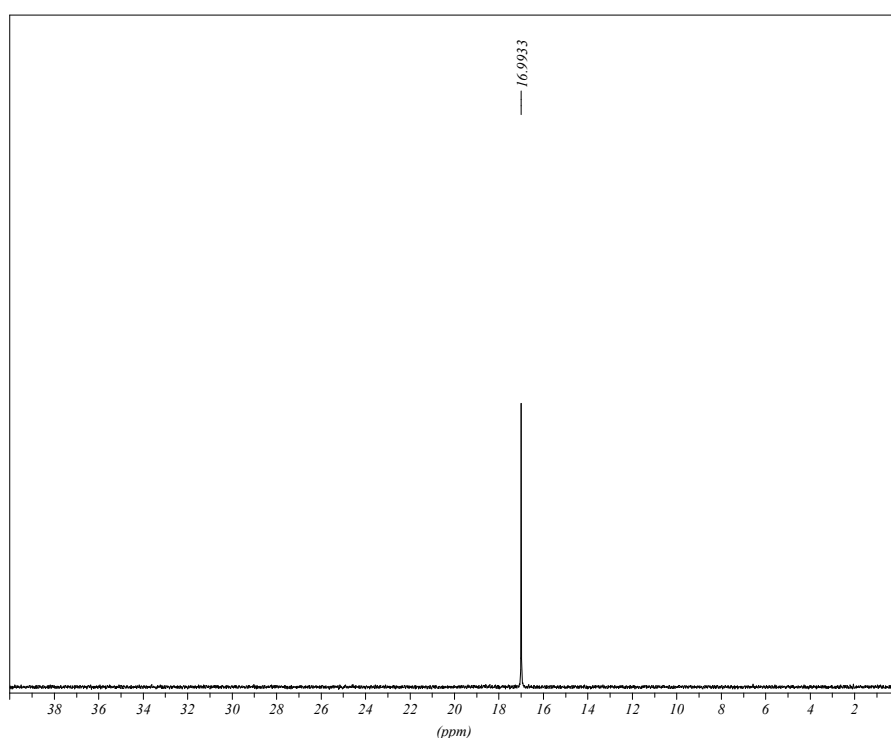
Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

(E)-Diethyl (3-(1-(benzyloxy)-3-phenylureido)prop-1-en-1-yl)phosphonate (8b)

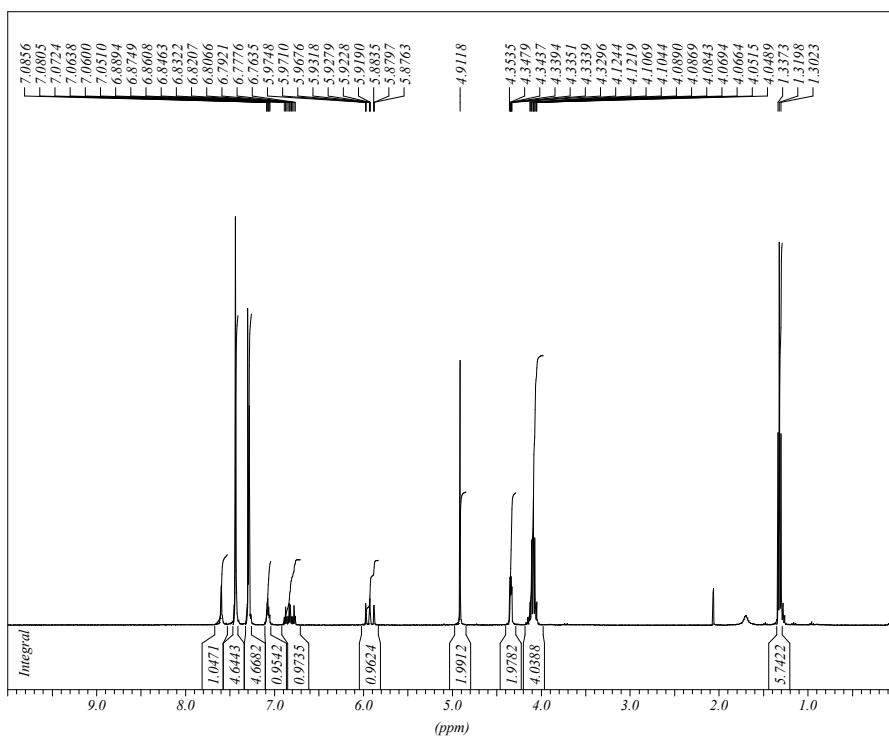


^1H NMR (400.13 MHz, CDCl_3) δ 1.32 (t, $^3J_{\text{HH}} = 7.0$ Hz, 6H), 4.05–4.12 (m, 4H), 4.33–4.35 (m, 2H), 4.91 (s, 2H), 5.88–5.97 (m, 1H), 6.76–6.89 (m, 1H), 7.26–7.31 (m, 5H), 7.41–7.47 (m, 5H), 7.60 (s, 1H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.33 (d, $^3J_{\text{CP}} = 6.6$ Hz), 51.72 (d, $^3J_{\text{CP}} = 24.9$ Hz), 61.87 (d, $^2J_{\text{CP}} = 5.9$ Hz), 77.95 (s), 119.31 (s), 120.70 (d, $^1J_{\text{CP}} = 186.6$ Hz), 123.74 (s), 128.93 (s), 129.02 (s), 129.37 (s), 129.44 (s), 134.71 (s), 137.53 (s), 146.22 (d, $^2J_{\text{CP}} = 5.1$ Hz), 156.68 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 16.99 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{28}\text{N}_2\text{O}_5\text{P}$ 419.1736, found 419.1736.

BG049-P1 P31CPD CDCl_3 opt/topspin am2n1 59



```
*** Current Data Parameters ***
NAME       : 516
EXPNO      : 16\
PROCNO     : 1
*** Acquisition Parameters ***
DATE_1     : 08:39:13
DATE_2     : Aug 24 2011
EXP        : PS done
NS         : 16
OI         : 6479.02 Hz
PROBHD     : 5 mm PABBO BB/19F-1H/D Z-G
RG         : 20642.5000000
SFO1       : 161.9820520 MHz
SOLVENT    :  $\text{CDCl}_3$ 
SW         : 200.4391 ppm
TD         : 65536
TE         : 673.2 K
*** Processing Parameters ***
GB         : 0.0000000
LB         : 0.50 Hz
SF         : 161.9755730 MHz
TI         :
*** 1D NMR Plot Parameters ***
Start      : 40.01 ppm
Stop       : 0.01 ppm
SR         : 0.01 Hz
SOLVENT    : ?
```



*** Current Data Parameters ***

NAME : 515
 EXPNO : 15
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 08:20:47
 DATE_d : Aug 24 2011
 EXP : PROTON
 NS : 16
 O1 : 2400.78 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 114.000000
 SFO1 : 400.1324008 MHz

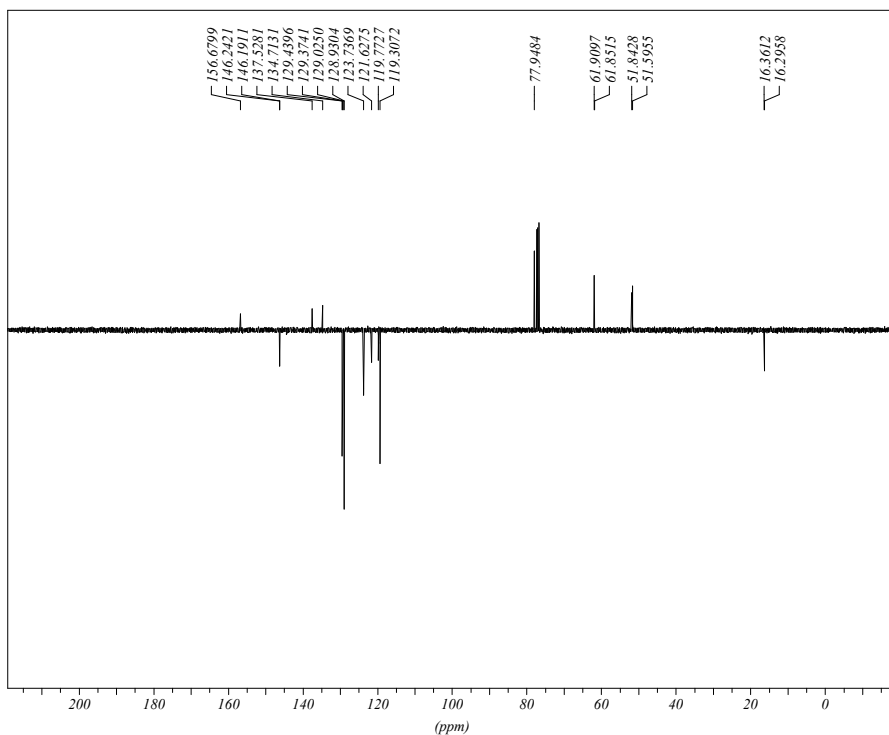
SOLVENT : CDCl3
 SW : 13.9775 ppm
 TD : 65536
 TE : 673.2 K

*** Processing Parameters ***

GB : 0.000000
 LB : 0.50 Hz
 SF : 400.1300000 MHz
 TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
 Stop : 0.00 ppm
 SR : 0.00 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 517
 EXPNO : 17
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 13:09:11
 DATE_d : Aug 24 2011
 EXP : C13APT
 NS : 1024
 O1 : 10060.80 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 23170.500000
 SFO1 : 100.6228298 MHz

SOLVENT : CDCl3
 SW : 238.3238 ppm
 TD : 65536
 TE : 673.2 K

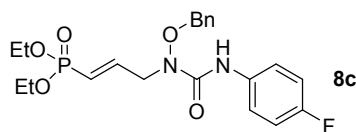
*** Processing Parameters ***

GB : 0.000000
 LB : 0.50 Hz
 SF : 100.6127690 MHz
 TI :

*** 1D NMR Plot Parameters ***

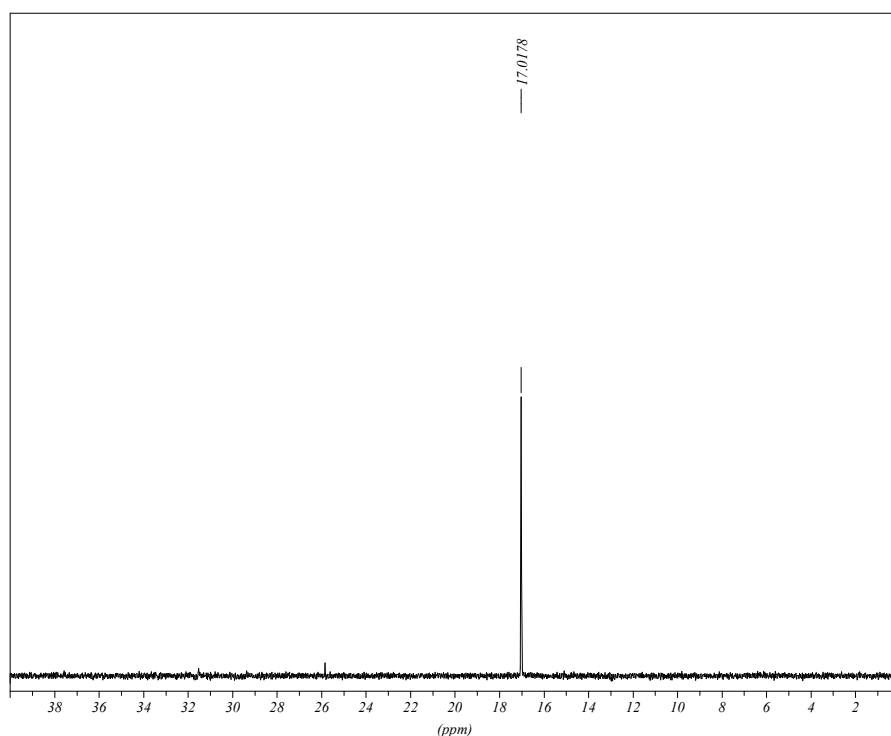
Start : 219.17 ppm
 Stop : -19.18 ppm
 SR : 0.00 Hz
 SOLVENT : ?

(E)-Diethyl (3-(1-(benzyloxy)-3-(4-fluorophenyl)ureido)prop-1-en-1-yl)phosphonate (8c)

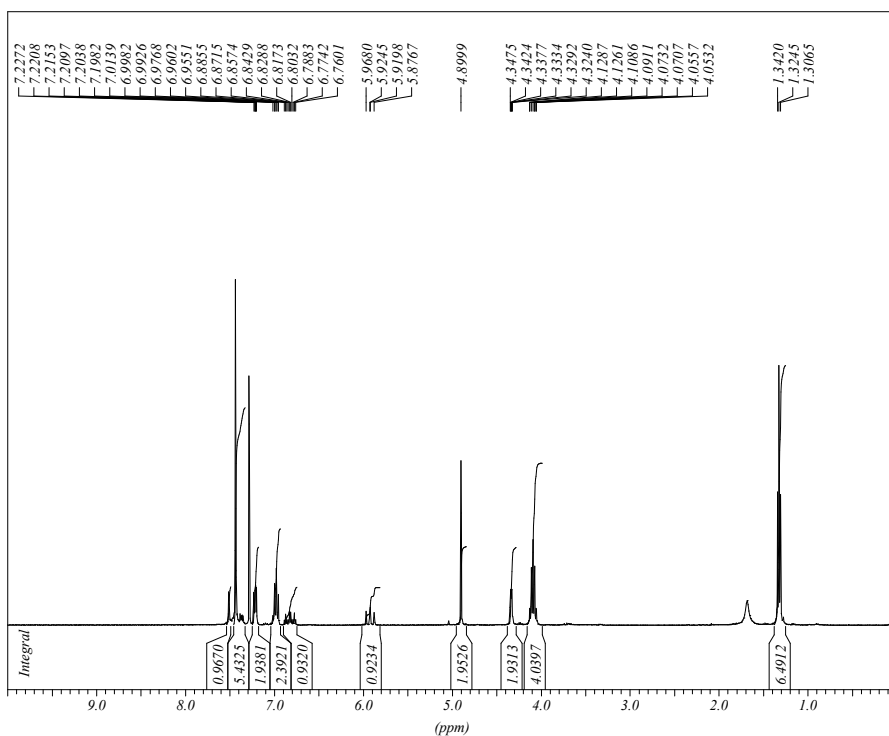


^1H NMR (400.13 MHz, CDCl_3) δ 1.32 (t, $^3J_{\text{HH}} = 7.0$ Hz, 6H), 4.05–4.13 (m, 4H), 4.32–4.35 (m, 2H), 4.90 (s, 2H), 5.88–5.97 (m, 1H), 6.76–6.88 (ddt, m, 1H), 6.95–7.01 (m, 2H), 7.20–7.23 (m, 2H), 7.35–7.44 (m, 5H), 7.51 (s, 1H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.32 (d, $^3J_{\text{CP}} = 5.8$ Hz), 51.67 (d, $^3J_{\text{CP}} = 24.9$ Hz), 61.96 (d, $^2J_{\text{CP}} = 5.9$ Hz), 77.88 (s), 115.51 (d, $^2J_{\text{CF}} = 22.7$ Hz), 120.54 (d, $^1J_{\text{CP}} = 187.4$ Hz), 121.18 (d, $^3J_{\text{CF}} = 7.3$ Hz), 129.03 (s), 129.40 (s), 129.47 (s), 133.46 (d, $^4J_{\text{CF}} = 2.2$ Hz), 134.71 (s), 146.27 (d, $^2J_{\text{CP}} = 5.1$ Hz), 156.82 (s), 159.15 (d, $^1J_{\text{CF}} = 243.0$ Hz). ^{31}P NMR (161.97 MHz, CDCl_3) δ 17.02 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{27}\text{FN}_2\text{O}_5\text{P}$ 437.1642, found 437.1642.

BG050-P1 P31CPD CDCl_3 opt/topspin am2n1 12



*** Current Data Parameters ***
 NAME : 502
 EXPNO : 2\
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 01:24:46
 DATE_d : Aug 31 2011
 EXP : PS done
 NS : 16
 O1 : 6479.02 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 20642.5000000
 SFO1 : 161.9820520 MHz
 SOLVENT : CDCl_3
 SW : 200.4391 ppm
 TD : 65536
 TE : 673.2 K
 *** Processing Parameters ***
 GB : 0.0000000
 LB : 0.50 Hz
 SF : 161.9755730 MHz
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 40.01 ppm
 Stop : 0.01 ppm
 SR : 0.01 Hz
 SOLVENT : ?



*** Current Data Parameters ***

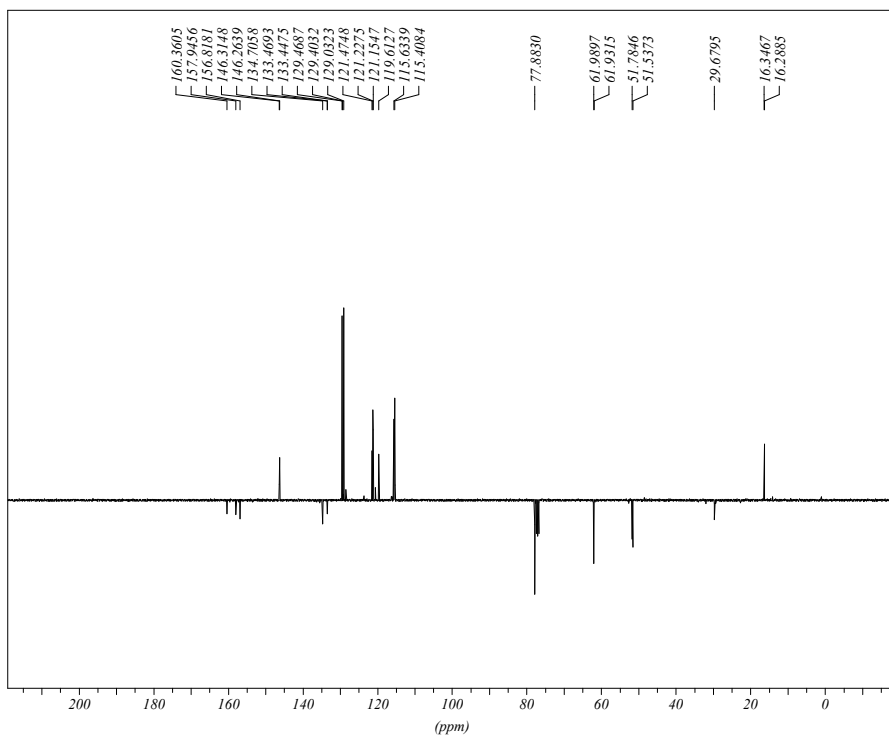
NAME : 501
EXPNO : 1\
PROCNO : 1
*** Acquisition Parameters ***
DATE_t : 01:22:05
DATE_d : Aug 31 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 143.6999969
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 13.9775 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 504
EXPNO : 4\
PROCNO : 1
*** Acquisition Parameters ***
DATE_t : 13:38:05
DATE_d : Sep 06 2012
EXP : C13APT
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD
RG : 16384.0000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm
TD : 65536
TE : 299.2 K

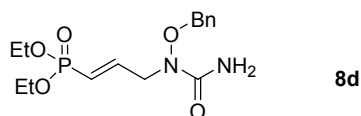
*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

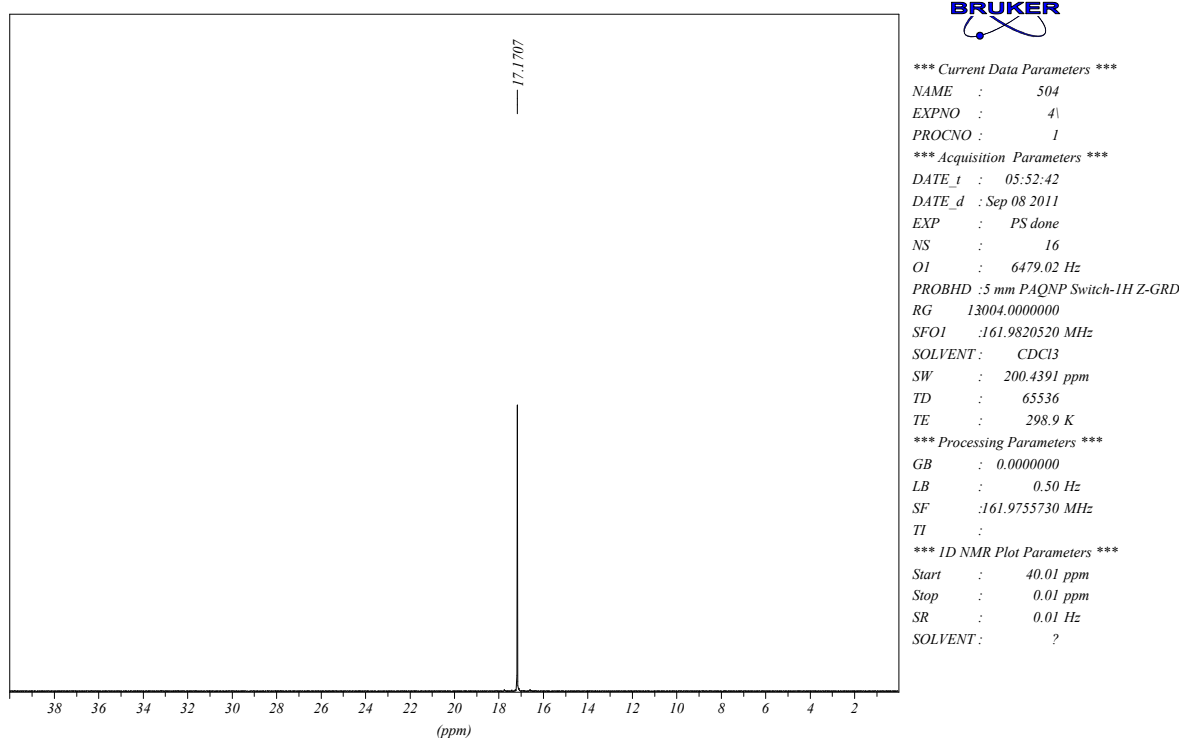
Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

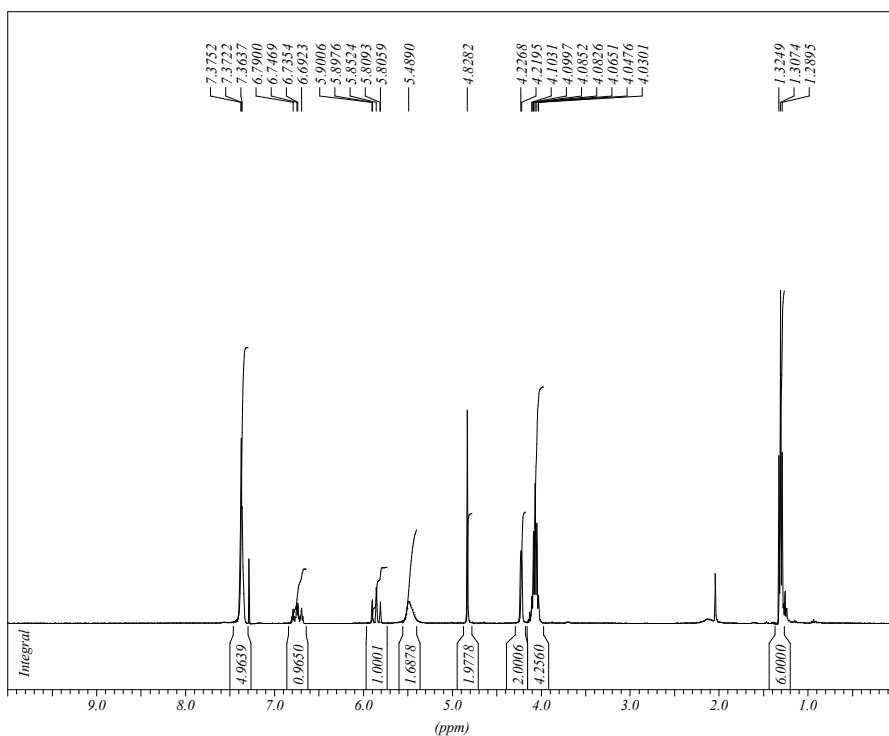
(E)-Diethyl (3-(1-(benzyloxy)-3-ureido)prop-1-en-1-yl)phosphonate (8d)



^1H NMR (400.13 MHz, CDCl_3) δ 1.31 (t, $^3J_{\text{HH}} = 7.0$ Hz, 6H), 4.03–4.10 (m, 4H), 4.22–4.23 (m, 2H), 4.83 (s, 2H), 5.49 (bs, 2H), 5.81–5.90 (m, 1H), 6.68–6.80 (m, 1H), 7.36–7.28 (m, 5H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.35 (d, $^3J_{\text{CP}} = 6.6$ Hz), 51.21 (d, $^3J_{\text{CP}} = 24.9$ Hz), 61.85 (d, $^2J_{\text{CP}} = 5.9$ Hz), 77.42 (s), 120.25 (d, $^1J_{\text{CP}} = 187.3$ Hz), 128.82 (s), 129.10 (s), 129.25 (s), 134.73 (s), 146.31 (d, $^2J_{\text{CP}} = 5.1$ Hz), 160.21 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 17.17 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{15}\text{H}_{24}\text{N}_2\text{O}_5\text{P}$ 343.1417, found 343.1423.

SM272.2-P1 P31CPD CDCl_3 opt/topspin am2n1 8





*** Current Data Parameters ***

NAME : 503
EXPNO : 3
PROCNO : 1

*** Acquisition Parameters ***

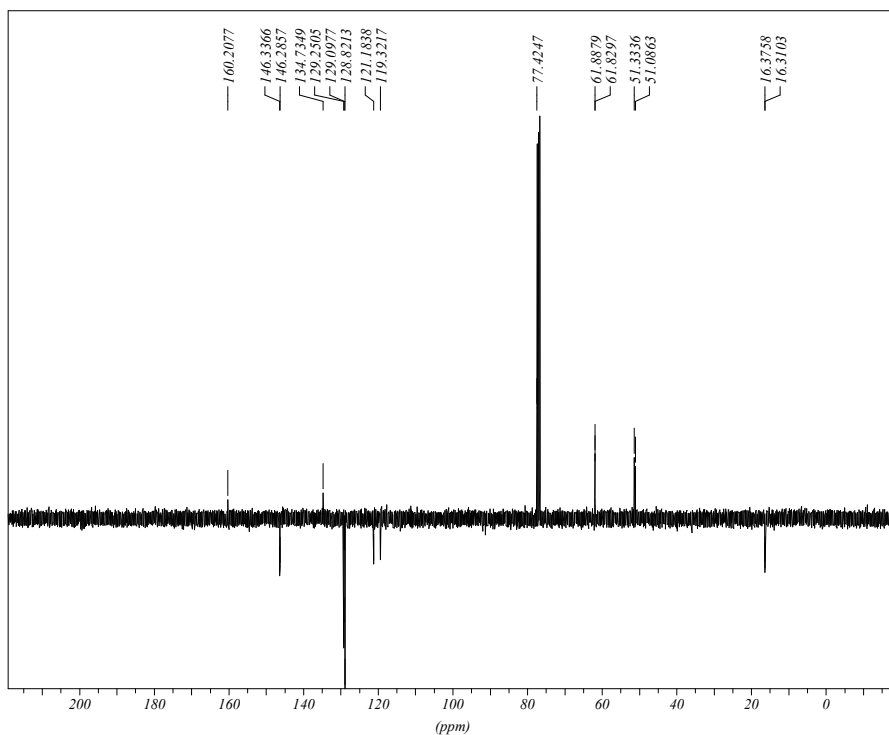
DATE_t : 05:49:50
DATE_d : Sep 08 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD
RG : 80.5999985
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 13.9775 ppm
TD : 65536
TE : 298.8 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 527
EXPNO : 27
PROCNO : 1

*** Acquisition Parameters ***

DATE_t : 12:10:14
DATE_d : Aug 26 2011
EXP : C13APT
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 16384.0000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm
TD : 65536
TE : 673.2 K

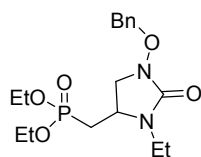
*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

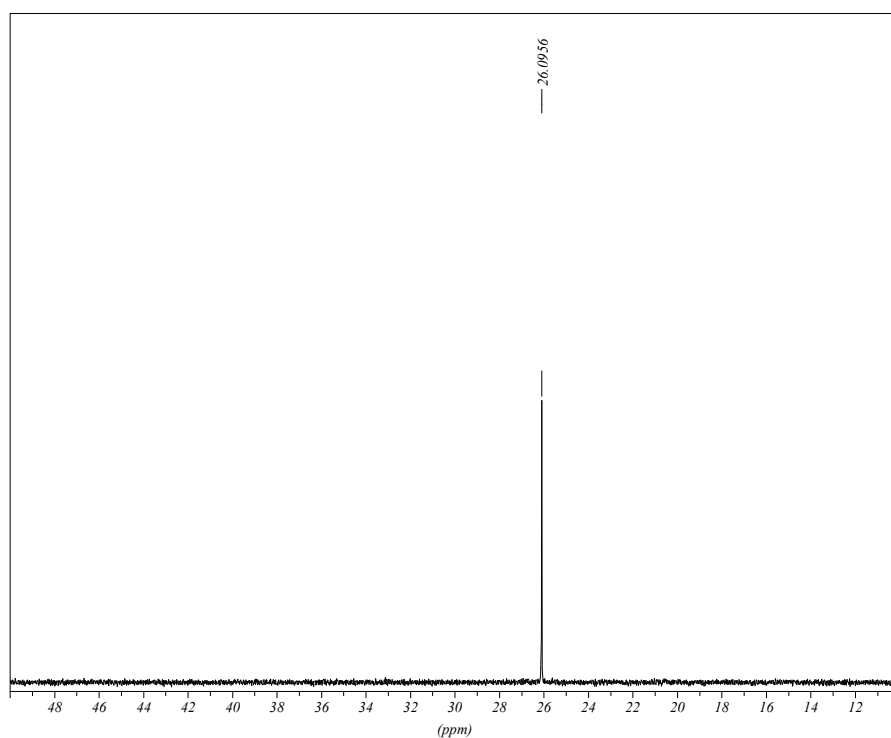
(±)-Diethyl ((1-(benzyloxy)-3-ethyl-2-oxoimidazolidin-4-yl)methyl)phosphonate (7a)



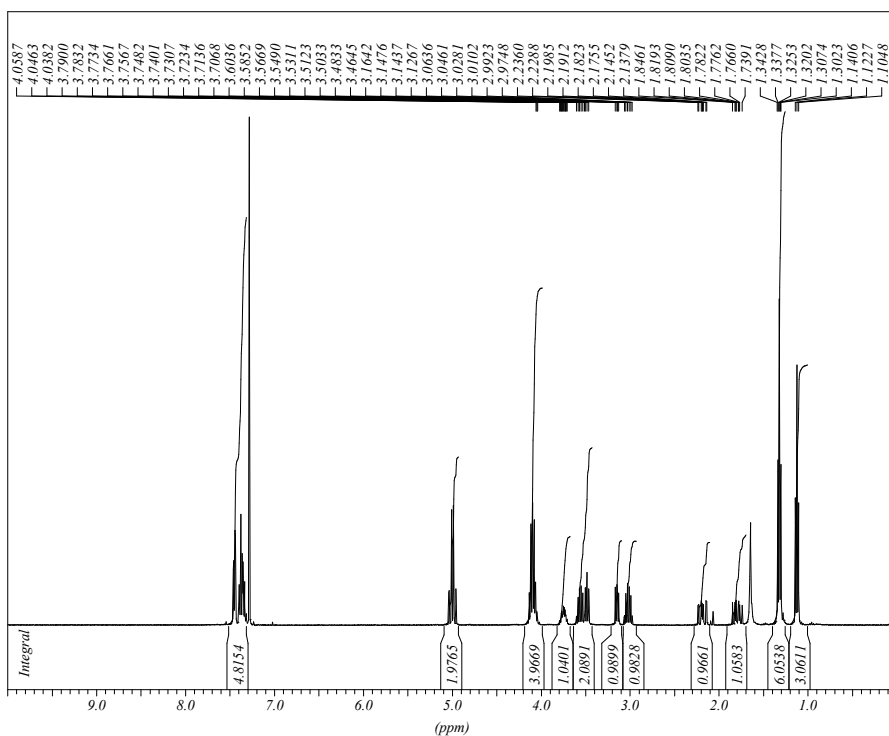
7a

^1H NMR (400.13 MHz, CDCl_3) δ 1.12 (t, $^3J_{\text{HH}} = 7.2$ Hz, 3H), 1.32 (t, $^3J_{\text{HH}} = 7.1$ Hz, 3H), 1.33 (t, $^3J_{\text{HH}} = 7.1$ Hz, 3H), 1.79 (ddd, $^2J_{\text{HP}} = 17.1$ Hz, $^2J_{\text{HH}} = 14.8$ Hz, $^3J_{\text{HH}} = 10.7$ Hz, 1H), 2.19 (ddd, $^2J_{\text{HP}} = 21.5$ Hz, $^2J_{\text{HH}} = 15.0$ Hz, $^3J_{\text{HH}} = 2.9$ Hz, 1H), 3.02 (dq, $^2J_{\text{HH}} = 14.2$ Hz, $^3J_{\text{HH}} = 7.0$ Hz, 1H), 3.14 (dd, $^2J_{\text{HH}} = 8.2$ Hz, $^3J_{\text{HH}} = 6.6$ Hz, 1H), 3.46–3.50 (m, 1H), 3.56 (dq, $^2J_{\text{HH}} = 14.7$ Hz, $^3J_{\text{HH}} = 7.3$ Hz, 1H), 3.71–3.78 (m, 1H), 4.04–4.14 (m, 4H), 4.97 (d, $^2J_{\text{HH}} = 11.4$ Hz, 1H), 5.02 (d, $^2J_{\text{HH}} = 11.4$ Hz, 1H), 7.33–7.46 (m, 5H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 12.55 (s), 16.38 (d, $^3J_{\text{CP}} = 5.9$ Hz), 16.44 (d, $^3J_{\text{CP}} = 5.9$ Hz), 28.66 (d, $^1J_{\text{CP}} = 138.8$ Hz), 35.81 (s), 46.73 (s), 53.57 (d, $^3J_{\text{CP}} = 1.4$ Hz), 61.95 (d, $^2J_{\text{CP}} = 6.6$ Hz), 62.06 (d, $^2J_{\text{CP}} = 6.6$ Hz), 77.88 (s), 128.23 (s), 128.36 (s), 129.08 (s), 136.72 (s), 162.04 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 26.10 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{17}\text{H}_{28}\text{N}_2\text{O}_5\text{P}$ 371.1736, found 371.1736.

SM273-P1 P31CPD CDCl_3 opt/topspin am2n1 60



*** Current Data Parameters ***
 NAME : 504
 EXPNO : 4
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 01:46:42
 DATE_d : Aug 26 2011
 EXP : PS done
 NS : 16
 OI : 6479.02 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 20642.500000
 SFO1 : 161.9820520 MHz
 SOLVENT : CDCl_3
 SW : 200.4391 ppm
 TD : 65536
 TE : 673.2 K
 *** Processing Parameters ***
 GB : 0.000000
 LB : 0.50 Hz
 SF : 161.9755730 MHz
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 50.00 ppm
 Stop : 10.00 ppm
 SR : 0.01 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 503
EXPNO : 31
PROCNO : 1

*** Acquisition Parameters ***

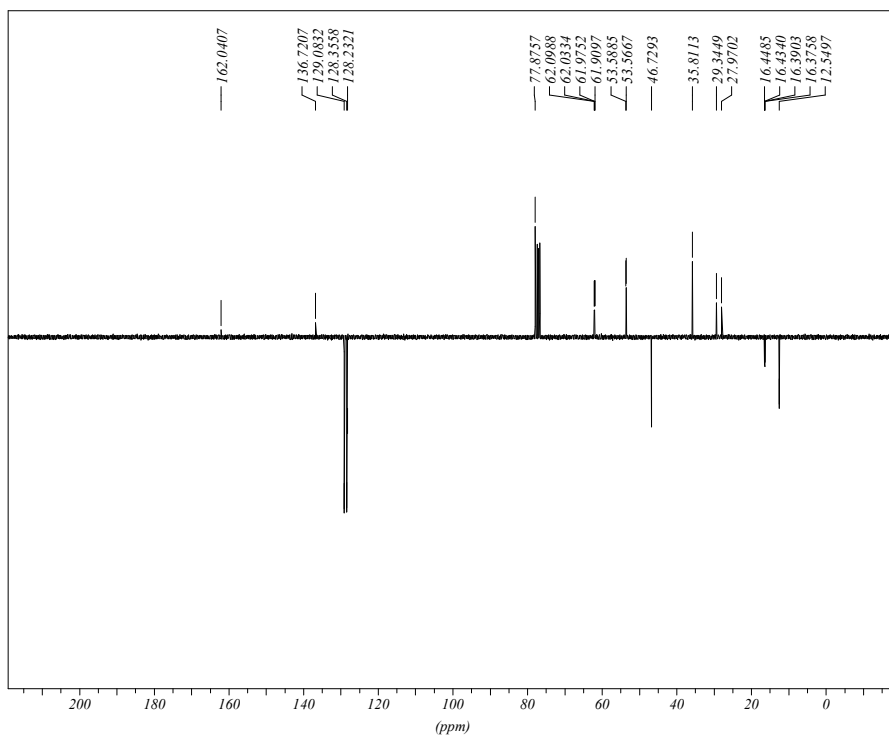
DATE_1 : 01:27:56
DATE_d : Aug 26 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 143.6999969
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 13.9775 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 530
EXPNO : 301
PROCNO : 1

*** Acquisition Parameters ***

DATE_1 : 14:04:01
DATE_d : Jun 25 2011
EXP : C13APT
NS : 1026
OI : 10060.80 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 23170.5000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm
TD : 65536
TE : 673.2 K

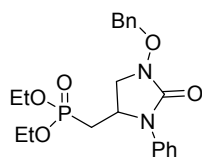
*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

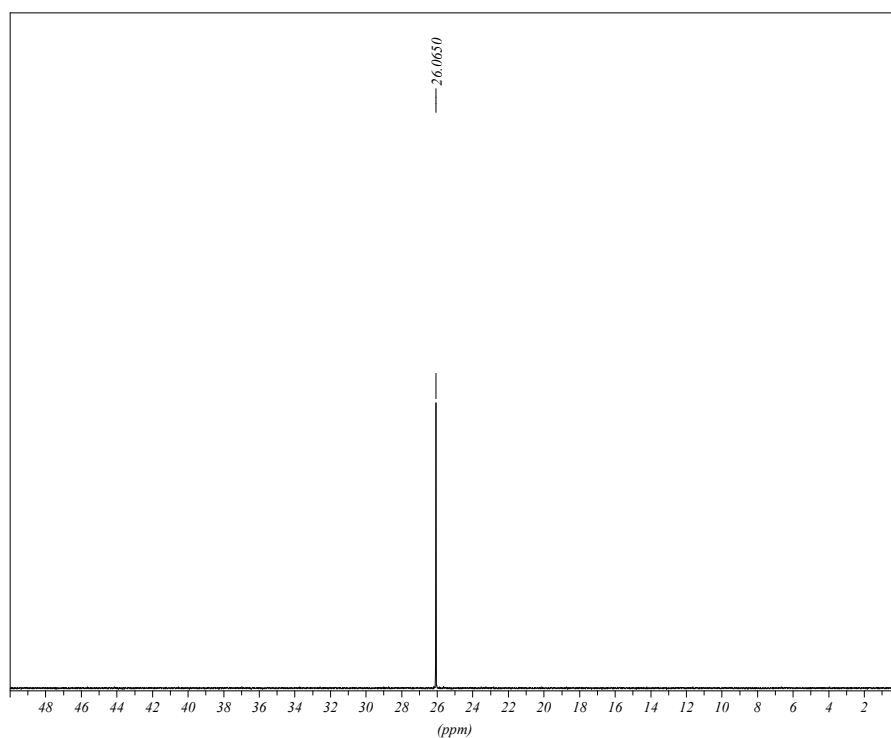
(±)-Diethyl ((1-(benzyloxy)-3-phenyl-2-oxoimidazolidin-4-yl)methyl)phosphonate (7b)



7b

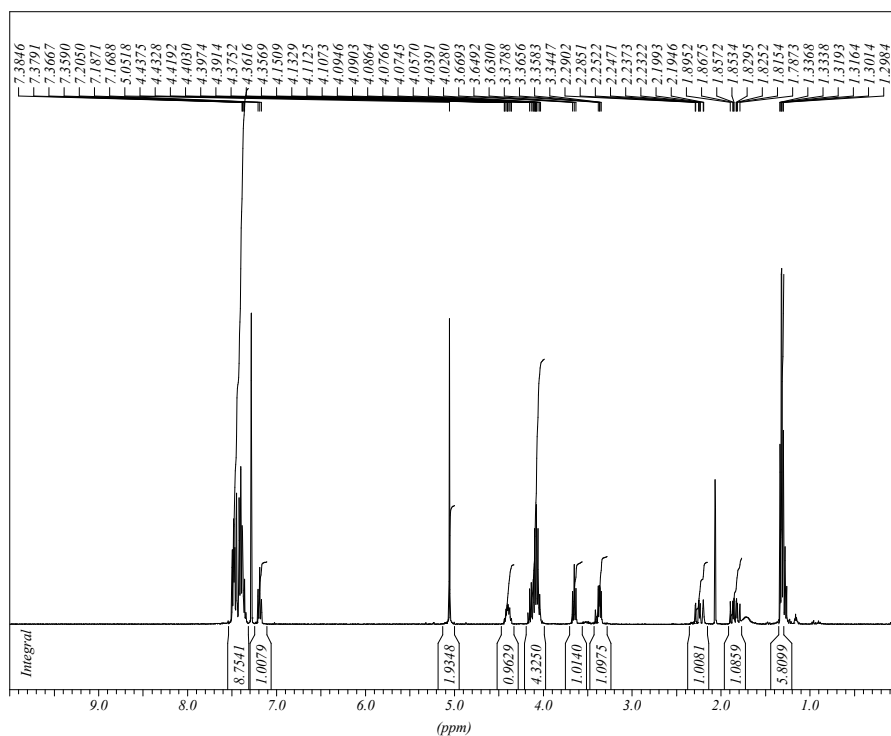
^1H NMR (400.13 MHz, CDCl_3) δ 1.31 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.32 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.85 (ddd, $^2J_{\text{HP}} = 16.7$ Hz, $^2J_{\text{HH}} = 15.2$ Hz, $^3J_{\text{HH}} = 11.1$ Hz, 1H), 2.25 (ddd, $^2J_{\text{HP}} = 21.1$ Hz, $^2J_{\text{HH}} = 15.2$ Hz, $^3J_{\text{HH}} = 2.0$ Hz, 1H), 3.36 (dd, $^2J_{\text{HH}} = 8.2$ Hz, $^3J_{\text{HH}} = 5.3$ Hz, 1H), 3.63–3.67 (m, 1H), 4.03–4.15 (m, 4H), 4.36–4.44 (m, 1H), 5.05 (s, 2H), 7.17–7.20 (m, 5H), 7.36–7.50 (m, 5H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.39 (d, $^3J_{\text{CP}} = 5.9$ Hz), 28.48 (d, $^1J_{\text{CP}} = 138.3$ Hz), 48.05 (d, $^2J_{\text{CP}} = 1.5$ Hz), 52.62 (s), 61.01 (d, $^2J_{\text{CP}} = 6.6$ Hz), 62.06 (d, $^2J_{\text{CP}} = 6.6$ Hz), 77.98 (s), 121.33 (s), 125.02 (s), 128.41 (s), 128.43 (s), 129.22 (s), 129.26 (s), 136.41 (s), 136.73 (s), 159.84 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 26.06 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{28}\text{N}_2\text{O}_5\text{P}$ 419.1724, found 419.1736.

BG051-P1 P31CPD CDCl_3 opt/topspin am2n1 59



*** Current Data Parameters ***

NAME : 539
EXPNO : 39
PROCNO : 1
*** Acquisition Parameters ***
DATE_t : 07:12:10
DATE_d : Jul 05 2011
EXP : PS done
NS : 16
OI : 6479.02 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 20642.500000
SFO1 : 161.9820520 MHz
SOLVENT : CDCl_3
SW : 200.4391 ppm
TD : 65536
TE : 673.2 K
*** Processing Parameters ***
GB : 0.000000
LB : 0.50 Hz
SF : 161.9755730 MHz
TI :
*** 1D NMR Plot Parameters ***
Start : 50.00 ppm
Stop : 0.01 ppm
SR : 0.01 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 537
 EXPNO : 37
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 05:52:06
 DATE_d : Jul 05 2011
 EXP : PROTON
 NS : 16
 O1 : 2400.78 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 287.3999939
 SFO1 : 400.1324008 MHz

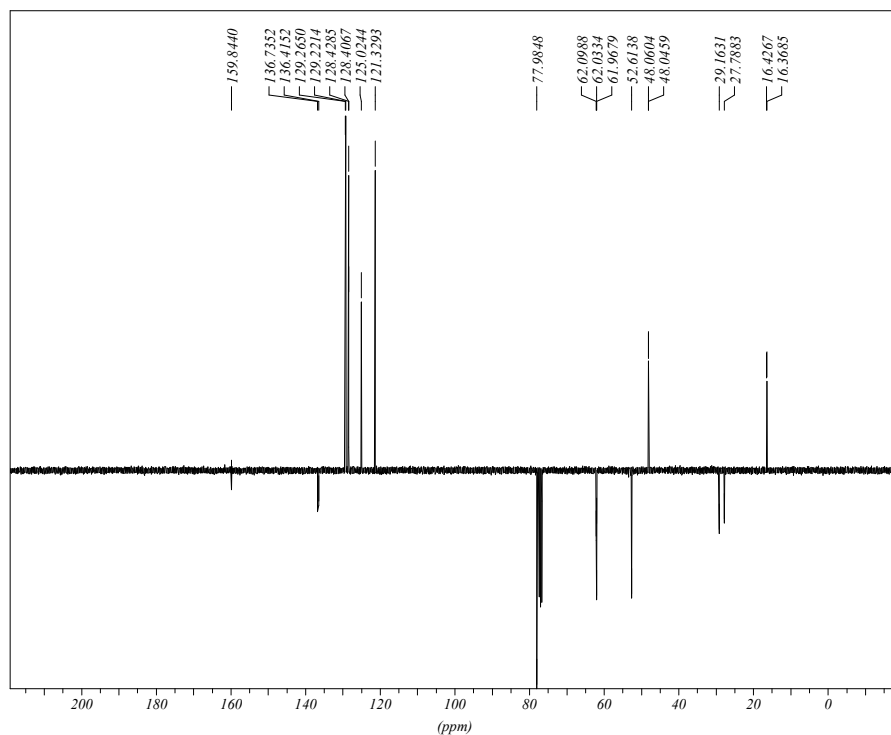
SOLVENT : CDCl3
 SW : 13.9775 ppm
 TD : 65536
 TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
 LB : 0.50 Hz
 SF : 400.1300000 MHz
 TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
 Stop : 0.00 ppm
 SR : 0.00 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 503
 EXPNO : 3
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 11:04:55
 DATE_d : Aug 23 2011
 EXP : C13APT
 NS : 1024
 O1 : 10060.80 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 16384.0000000
 SFO1 : 100.6228298 MHz

SOLVENT : CDCl3
 SW : 238.3238 ppm
 TD : 65536
 TE : 673.2 K

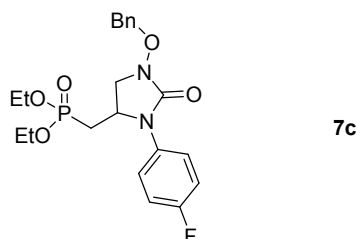
*** Processing Parameters ***

GB : 0.0000000
 LB : 0.50 Hz
 SF : 100.6127690 MHz
 TI :

*** 1D NMR Plot Parameters ***

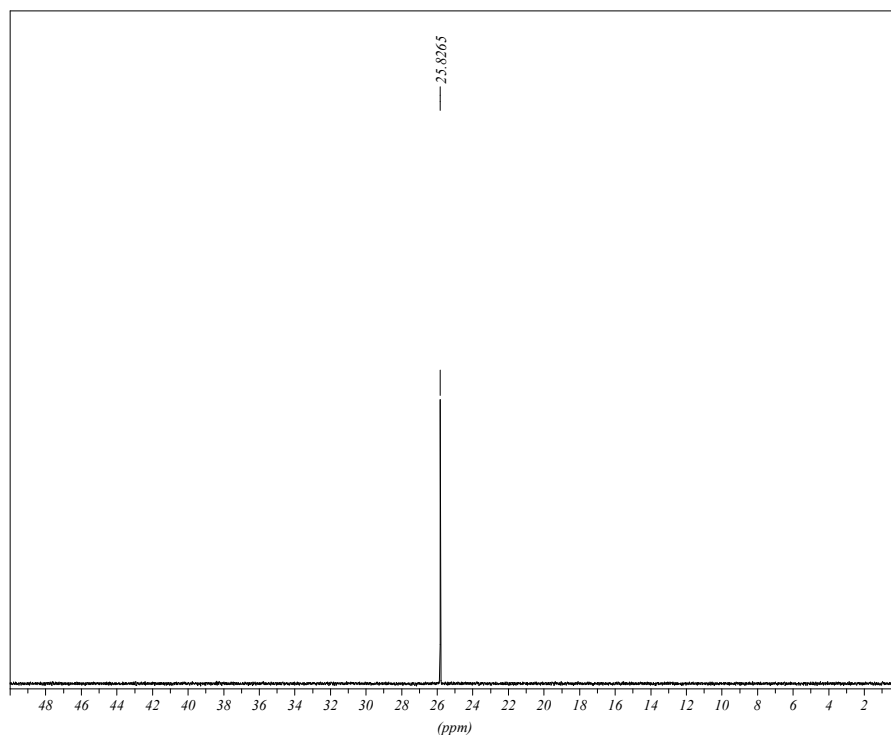
Start : 219.17 ppm
 Stop : -19.18 ppm
 SR : 0.00 Hz
 SOLVENT : ?

(±)-Diethyl ((1-(benzyloxy)-3-(4-fluorophenyl)-2-oxoimidazolidin-4-yl)methyl)phosphonate (7c)



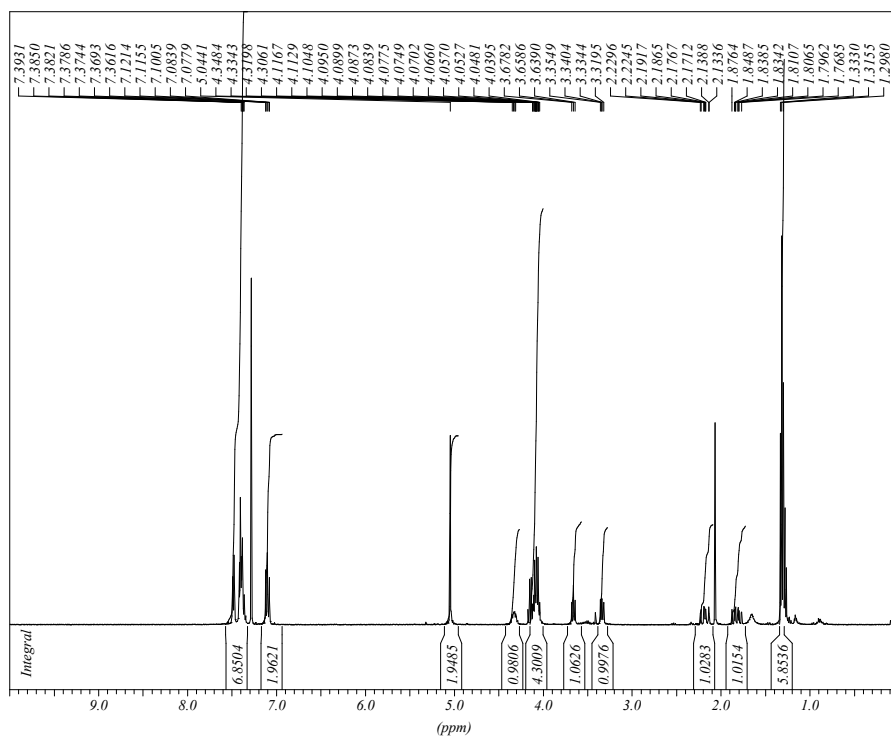
^1H NMR (400.13 MHz, CDCl_3) δ 1.31 (t, $^3J_{\text{HH}} = 7.0$ Hz, 6H), 1.82 (ddd, $^2J_{\text{HP}} = 16.9$ Hz, $^2J_{\text{HH}} = 15.2$ Hz, $^3J_{\text{HH}} = 11.1$ Hz, 1H), 2.18 (ddd, $^2J_{\text{HP}} = 17.2$ Hz, $^2J_{\text{HH}} = 15.2$ Hz, $^3J_{\text{HH}} = 2.1$ Hz, 1H), 3.34 (dd, $^2J_{\text{HH}} = 8.2$ Hz, $^3J_{\text{HH}} = 5.8$ Hz, 1H), 3.64–3.68 (m, 1H), 4.04–4.12 (m, 4H), 4.29–4.36 (m, 1H), 5.04 (s, 2H), 7.08–7.12 (m, 2H), 7.36–7.49 (m, 7H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.40 (d, $^3J_{\text{CP}} = 6.0$ Hz), 28.54 (d, $^1J_{\text{CP}} = 138.9$ Hz), 48.43 (d, $^2J_{\text{CP}} = 1.5$ Hz), 52.72 (s), 62.02 (d, $^2J_{\text{CP}} = 6.6$ Hz), 62.12 (d, $^2J_{\text{CP}} = 6.6$ Hz), 78.03 (s), 116.11 (d, $^2J_{\text{CF}} = 22.7$), 123.53 (d, $^3J_{\text{CF}} = 8.0$ Hz), 128.45 (s), 129.24 (s), 132.68 (s), 136.35 (s), 159.94 (s), 160.05 (d, $^1J_{\text{CF}} = 245.9$ Hz). ^{31}P NMR (161.97 MHz, CDCl_3) δ 25.83 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{21}\text{H}_{27}\text{FN}_2\text{O}_5\text{P}$ 437.1649, found 419.1642.

BG052-P1 P31CPD CDCl_3 opt/topspin am2n1 53



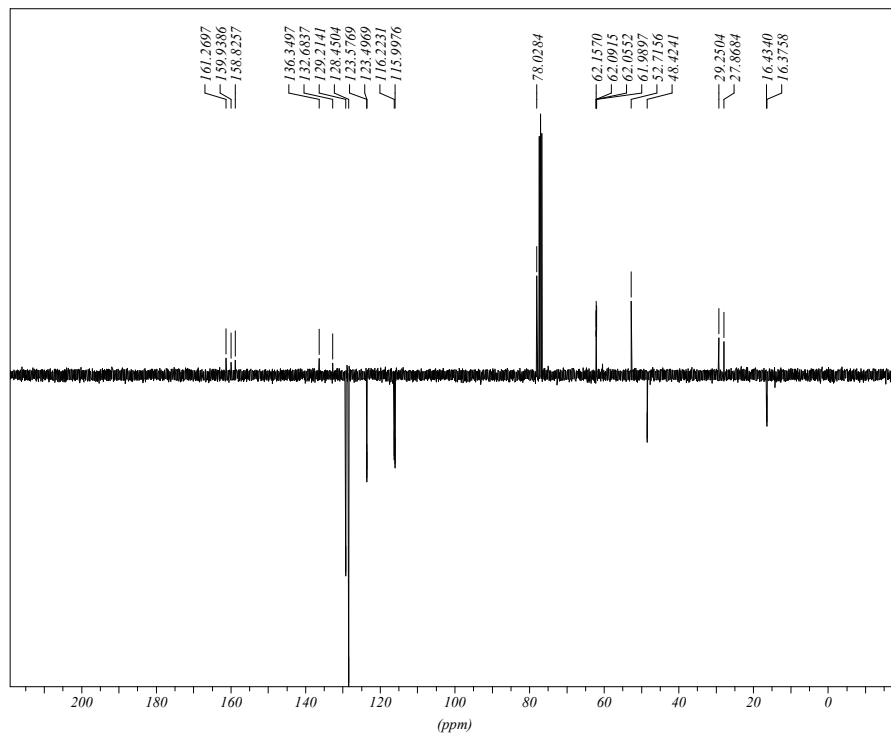
*** Current Data Parameters ***

NAME : 531
EXPNO : 31\
PROCNO : 1
*** Acquisition Parameters ***
DATE_1 : 18:06:37
DATE_d : Jul 05 2011
EXP : PS done
NS : 16
OI : 6479.02 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 20642.5000000
SFO1 : 161.9820520 MHz
SOLVENT : CDCl_3
SW : 200.4391 ppm
TD : 65536
TE : 673.2 K
*** Processing Parameters ***
GB : 0.0000000
LB : 0.50 Hz
SF : 161.9755730 MHz
TI :
*** 1D NMR Plot Parameters ***
Start : 50.00 ppm
Stop : 0.01 ppm
SR : 0.01 Hz
SOLVENT : ?



*** Current Data Parameters ***

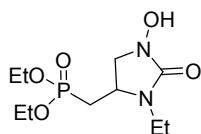
NAME : 530
 EXPNO : 30
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 18:03:36
 DATE_d : Jul 05 2011
 EXP : PROTON
 NS : 16
 O1 : 2400.78 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 181.000000
 SFO1 : 400.1324008 MHz
 SOLVENT : CDCl3
 SW : 13.9775 ppm
 TD : 65536
 TE : 673.2 K
 *** Processing Parameters ***
 GB : 0.000000
 LB : 0.50 Hz
 SF : 400.1300000 MHz
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 10.00 ppm
 Stop : 0.00 ppm
 SR : 0.00 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 533
 EXPNO : 33
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 19:10:24
 DATE_d : Jul 05 2011
 EXP : C13APT
 NS : 1026
 O1 : 10060.80 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 16384.000000
 SFO1 : 100.6228298 MHz
 SOLVENT : CDCl3
 SW : 238.3238 ppm
 TD : 65536
 TE : 673.2 K
 *** Processing Parameters ***
 GB : 0.000000
 LB : 0.50 Hz
 SF : 100.6127690 MHz
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 219.17 ppm
 Stop : -19.18 ppm
 SR : 0.00 Hz
 SOLVENT : ?

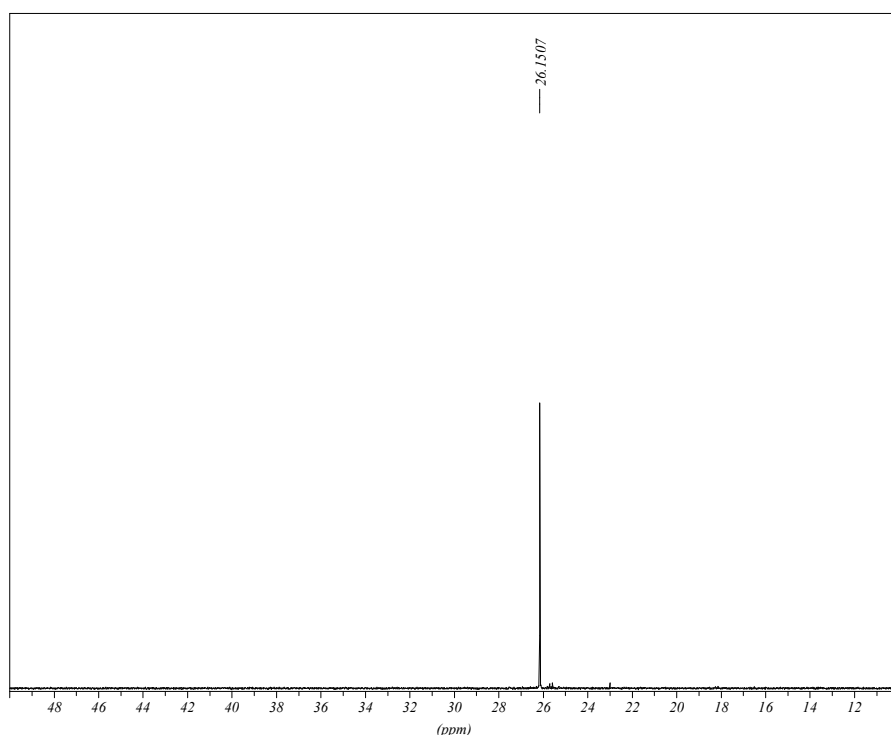
(±)-Diethyl ((1-hydroxy-3-ethyl-2-oxoimidazolidin-4-yl)methyl)phosphonate (**13a**)



13a

^1H NMR (400.13 MHz, CDCl_3) δ 1.12 (t, $^3J_{\text{HH}} = 7.1$ Hz, 3H), 1.36 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.90 (ddd, $^2J_{\text{HP}} = 17.4$ Hz, $^2J_{\text{HH}} = 15.0$ Hz, $^3J_{\text{HH}} = 10.7$ Hz, 1H), 2.25 (ddd, $^2J_{\text{HP}} = 21.3$ Hz, $^2J_{\text{HH}} = 14.8$ Hz, $^3J_{\text{HH}} = 2.7$ Hz, 1H), 3.05 (dq, $^2J_{\text{HH}} = 14.2$ Hz, $^3J_{\text{HH}} = 7.1$ Hz, 1H), 3.34 (dd, $^2J_{\text{HH}} = 8.2$ Hz, $^3J_{\text{HH}} = 6.6$ Hz, 1H), 3.49–3.58 (m, 1H), 3.73–3.77 (m, 1H), 3.82–3.88 (m, 1H), 4.11–4.16 (m, 4H), 8.46 (bs, 1H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 12.59 (s), 16.44 (d, $^3J_{\text{CP}} = 5.9$ Hz), 16.46 (d, $^3J_{\text{CP}} = 5.9$ Hz), 28.70 (d, $^1J_{\text{CP}} = 139.1$ Hz), 36.12 (s), 47.00 (s), 54.28 (d, $^4J_{\text{CP}} = 1.5$ Hz), 62.10 (d, $^2J_{\text{CP}} = 6.6$ Hz), 62.24 (d, $^2J_{\text{CP}} = 6.6$ Hz), 163.96 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 26.15 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{22}\text{N}_2\text{O}_5\text{P}$ 281.1266, found 281.1257.

SM403-P1 P31CPD CDCl_3 opt/topspin am2n1 1



*** Current Data Parameters ***

NAME : 506
EXPNO : 61
PROCNO : 1

*** Acquisition Parameters ***

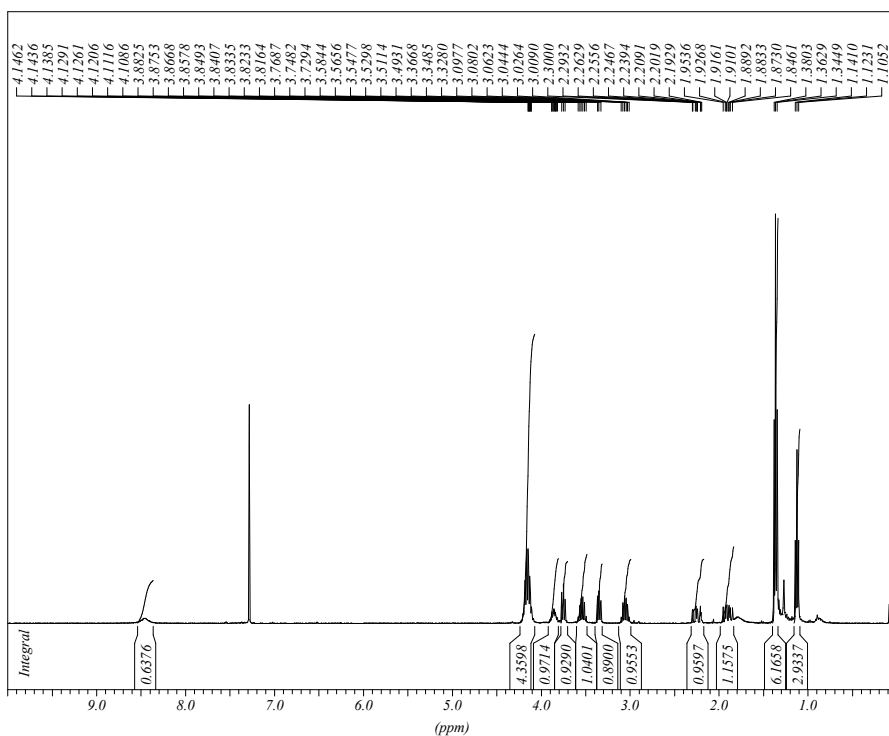
DATE_t : 05:42:20
DATE_d : Mar 26 2012
EXP : PS done
NS : 16
OI : 6479.02 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 20642.5000000
SFO1 : 161.9820520 MHz
SOLVENT : CDCl_3
SW : 200.4391 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 161.9755730 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 50.00 ppm
Stop : 10.00 ppm
SR : 0.01 Hz
SOLVENT : ?



*** Current Data Parameters ***

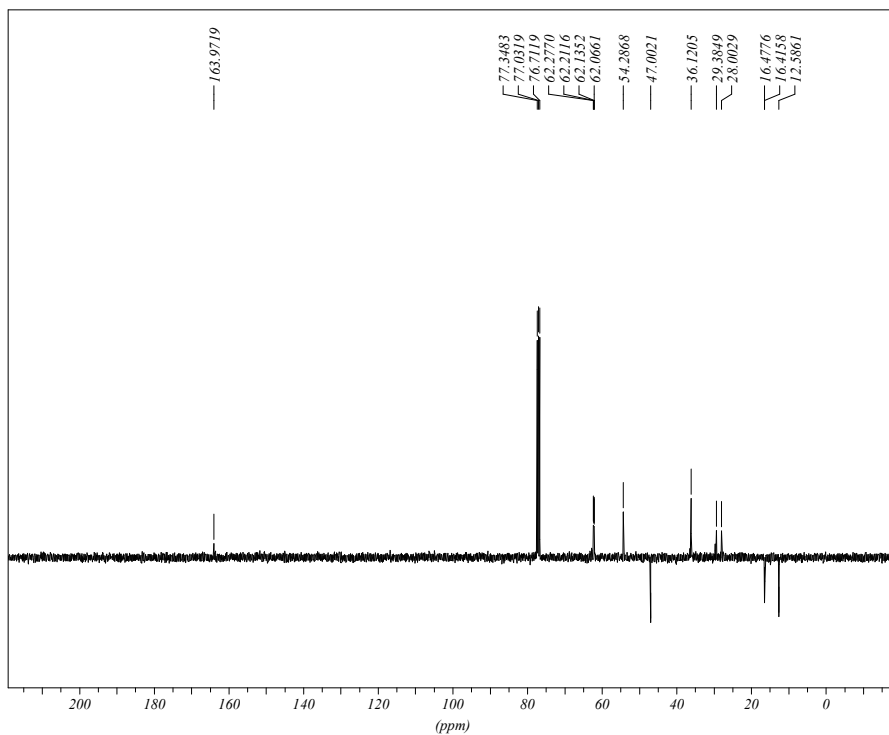
NAME : 505
 EXPNO : 51
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 05:39:07
 DATE_d : Mar 26 2012
 EXP : PROTON
 NS : 16
 O1 : 2400.78 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 322.5000000
 SFO1 : 400.1324008 MHz
 SOLVENT : CDCl3
 SW : 13.9775 ppm
 TD : 65536
 TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
 LB : 0.50 Hz
 SF : 400.1300000 MHz
 TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
 Stop : 0.00 ppm
 SR : 0.00 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : p1
 EXPNO : 509
 PROCNO : 0
 *** Acquisition Parameters ***
 DATE_t : 09:15:28
 DATE_d : Apr 14 2012
 EXP : C13APT
 NS : 1024
 O1 : 10060.80 Hz
 PROBHD : 5 mm PAQNP Switch-1H Z-GRD
 RG : 23170.5000000
 SFO1 : 100.6228298 MHz
 SOLVENT : CDCl3
 SW : 238.3238 ppm
 TD : 65536
 TE : 296.3 K

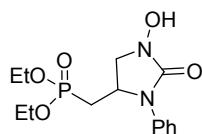
*** Processing Parameters ***

GB : 0.0000000
 LB : 2.00 Hz
 SF : 100.6127690 MHz
 TI :

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
 Stop : -19.18 ppm
 SR : 0.00 Hz
 SOLVENT : ?

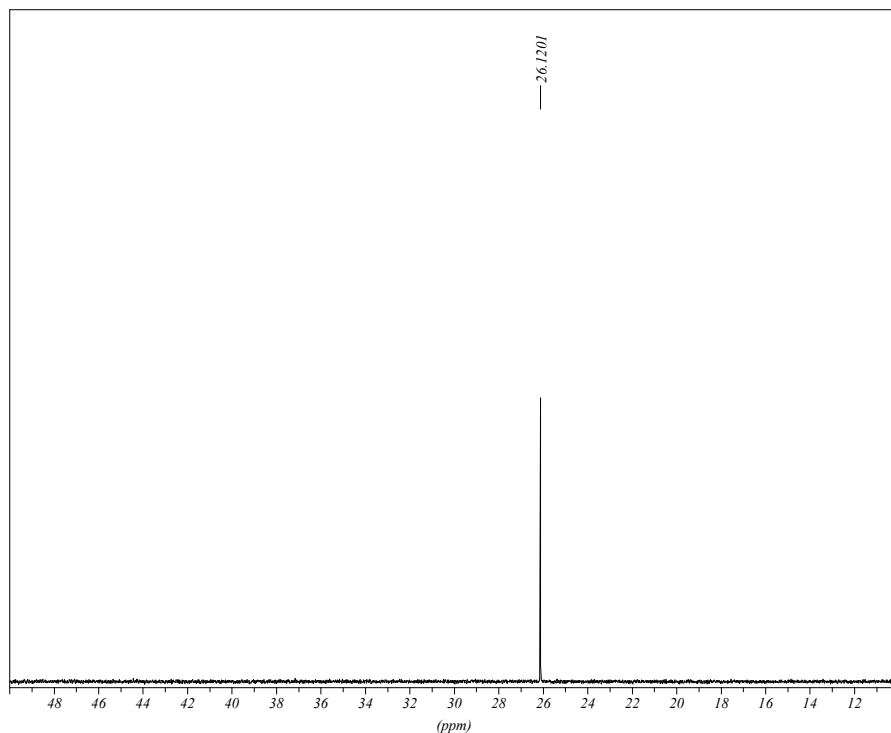
(±)-Diethyl ((1-hydroxy-3-phenyl)-2-oxoimidazolidin-4-yl)methylphosphonate (**13b**)



13b

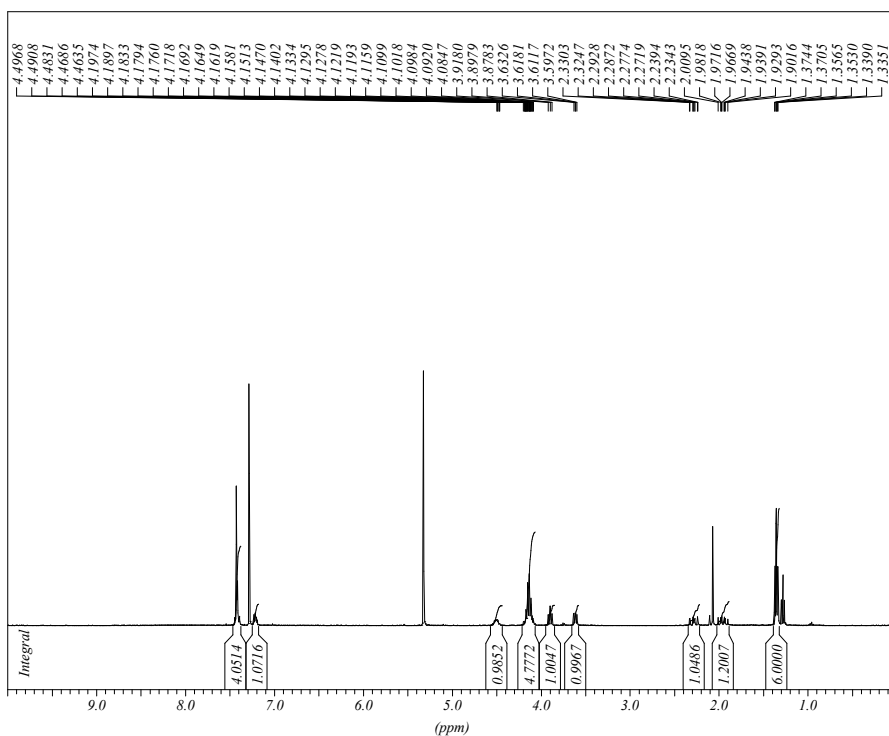
^1H NMR (400.13 MHz, CDCl_3) δ 1.35 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.36 (t, $^3J_{\text{HH}} = 7.1$ Hz, 3H), 1.95 (ddd, $^2J_{\text{HP}} = 17.1$ Hz, $^2J_{\text{HH}} = 15.2$ Hz, $^3J_{\text{HH}} = 11.1$ Hz, 1H), 2.28 (ddd, $^2J_{\text{HP}} = 21.2$ Hz, $^2J_{\text{HH}} = 15.0$ Hz, $^3J_{\text{HH}} = 2.2$ Hz, 1H), 3.61 (dd, $^2J_{\text{HH}} = 8.4$ Hz, $^3J_{\text{HH}} = 5.8$ Hz, 1H), 3.88–3.92 (m, 1H), 4.08–4.20 (m, 4H), 4.46–4.55 (m, 1H), 7.19–7.24 (m, 1H), 7.39–7.44 (m, 4H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.40 (d, $^3J_{\text{CP}} = 6.6$ Hz), 28.52 (d, $^1J_{\text{CP}} = 139.0$ Hz), 48.34 (s), 53.44 (s), 62.24 (d, $^2J_{\text{CP}} = 6.6$ Hz), 62.25 (d, $^2J_{\text{CP}} = 6.6$ Hz), 121.90 (s), 125.31 (s), 129.30 (s), 136.60 (s), 161.93 (s). ^{31}P NMR (161.97 MHz, CDCl_3) δ 26.12 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{22}\text{N}_2\text{O}_5\text{P}$ 329.1272, found 329.1266.

SM312.2-P1 P31CPD CDCl_3 opt/topspin am2n1 1



*** Current Data Parameters ***

NAME : 511
EXPNO : 111
PROCNO : 1
*** Acquisition Parameters ***
DATE_t : 08:47:57
DATE_d : Oct 20 2011
EXP : PS done
NS : 16
OI : 6479.02 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 20642.5000000
SFO1 : 161.9820520 MHz
SOLVENT : CDCl_3
SW : 200.4391 ppm
TD : 65536
TE : 673.2 K
*** Processing Parameters ***
GB : 0.0000000
LB : 0.50 Hz
SF : 161.9755730 MHz
TI :
*** 1D NMR Plot Parameters ***
Start : 50.00 ppm
Stop : 10.00 ppm
SR : 0.01 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 510
EXPNO : 10\
PROCNO : 1

*** Acquisition Parameters ***

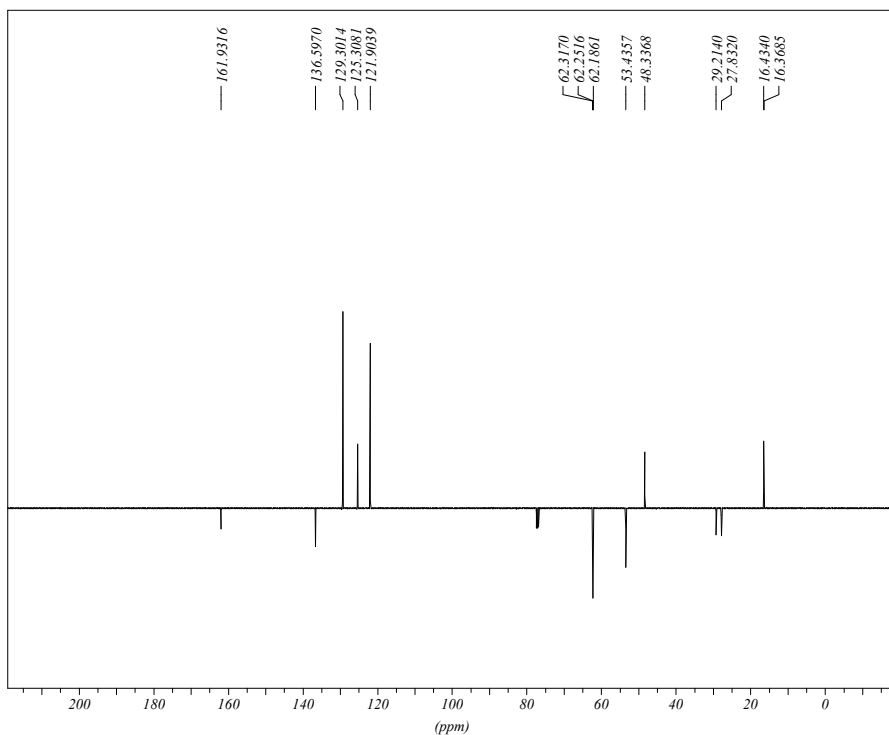
DATE_1 : 08:44:55
DATE_d : Oct 20 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 181.0000000
SFO1 : 400.1324008 MHz
SOLVENT : CDCl3
SW : 13.9775 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 502
EXPNO : 2\
PROCNO : 1

*** Acquisition Parameters ***

DATE_1 : 12:32:50
DATE_d : Sep 06 2012
EXP : C13APT
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PAQNP Switch-1H Z-GRD
RG : 16384.0000000
SFO1 : 100.6228298 MHz
SOLVENT : CDCl3
SW : 238.3238 ppm
TD : 65536
TE : 299.2 K

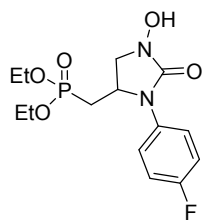
*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?

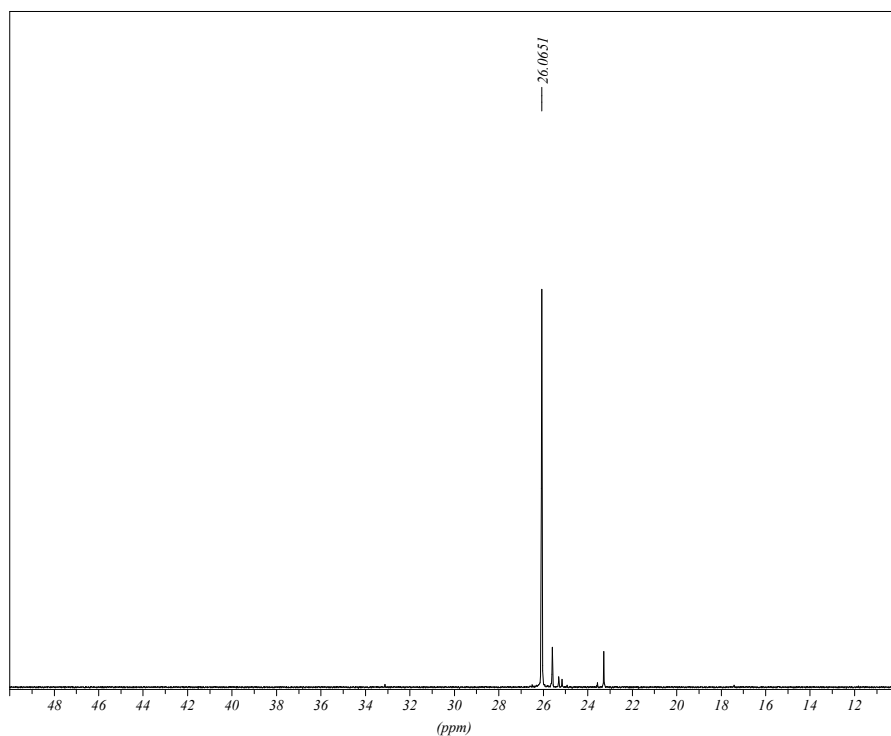
(±)-Diethyl ((1-hydroxy-3-(4-fluorophenyl)-2-oxoimidazolidin-4-yl)methyl)phosphonate (**13c**)



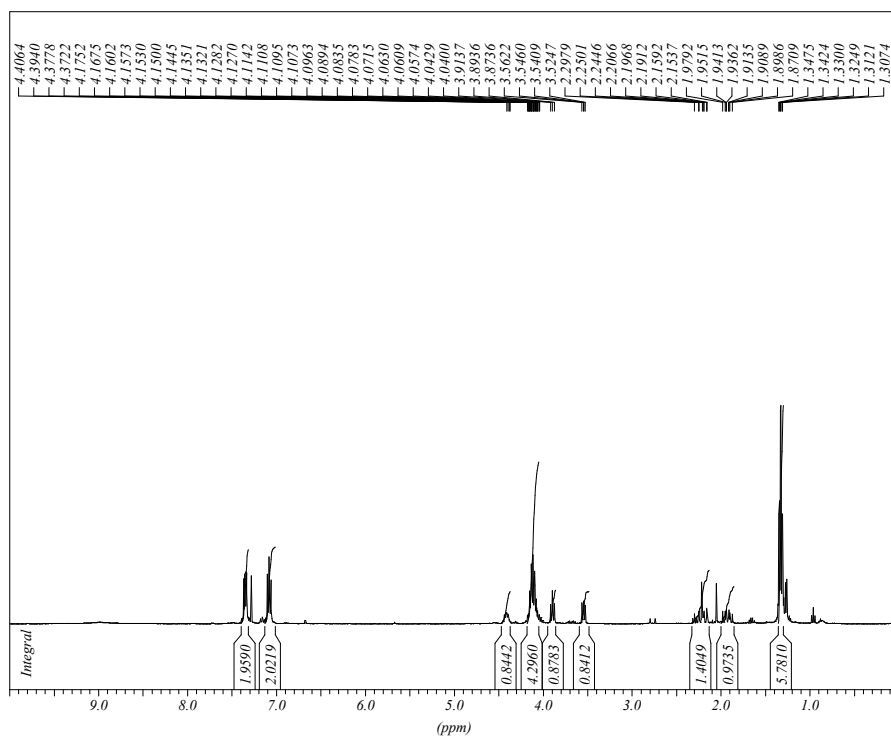
13c

^1H NMR (400.13 MHz, CDCl_3) δ 1.32 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.33 (t, $^3J_{\text{HH}} = 7.0$ Hz, 3H), 1.92 (ddd, $^2J_{\text{HP}} = 17.2$ Hz, $^2J_{\text{HH}} = 15.2$ Hz, $^3J_{\text{HH}} = 11.1$ Hz, 1H), 2.21 (ddd, $^2J_{\text{HP}} = 17.4$ Hz, $^2J_{\text{HH}} = 15.0$ Hz, $^3J_{\text{HH}} = 2.2$ Hz, 1H), 3.54 (dd, $^2J_{\text{HH}} = 8.5$ Hz, $^3J_{\text{HH}} = 6.5$ Hz, 1H), 3.87–3.91 (m, 1H), 4.04–4.17 (m, 4H), 4.37–4.46 (m, 1H), 7.06–7.10 (m, 2H), 7.32–7.37 (m, 2H). ^{13}C NMR (100.61 MHz, CDCl_3) δ 16.40 (d, $^3J_{\text{CP}} = 5.8$ Hz), 28.60 (d, $^1J_{\text{CP}} = 139.1$ Hz), 48.77 (s), 53.47 (s), 62.27 (d, $^2J_{\text{CP}} = 6.6$ Hz), 62.36 (d, $^2J_{\text{CP}} = 6.6$ Hz), 116.18 (d, $^2J_{\text{HF}} = 22.7$), 124.18 (d, $^3J_{\text{HF}} = 8.8$ Hz), 132.51 (d, $^4J_{\text{HF}} = 2.9$ Hz), 161.98 (s), 160.25 (d, $^1J_{\text{CF}} = 245.2$ Hz). ^{31}P NMR (161.97 MHz, CDCl_3) δ 26.06 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{14}\text{H}_{21}\text{FN}_2\text{O}_5\text{P}$ 347.1180, found 347.1172.

SM338.3-P2 P31CPD CDCl_3 opt/topspin am2n1 60



*** Current Data Parameters ***
 NAME : 503
 EXPNO : 3
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 12:57:07
 DATE_d : Jan 25 2012
 EXP : PS done
 NS : 16
 O1 : 6479.02 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 11585.2001953
 SFO1 : 161.9820520 MHz
 SOLVENT : CDCl_3
 SW : 200.4391 ppm
 TD : 65536
 TE : 673.2 K
 *** Processing Parameters ***
 GB : 0.000000
 LB : 0.50 Hz
 SF : 161.9755730 MHz
 TI :
 *** 1D NMR Plot Parameters ***
 Start : 50.00 ppm
 Stop : 10.00 ppm
 SR : 0.01 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 502
 EXPNO : 2\
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 12:54:02
 DATE_d : Jan 25 2012
 EXP : PROTON
 NS : 16
 O1 : 2400.78 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 57.0000000
 SFO1 : 400.1324008 MHz

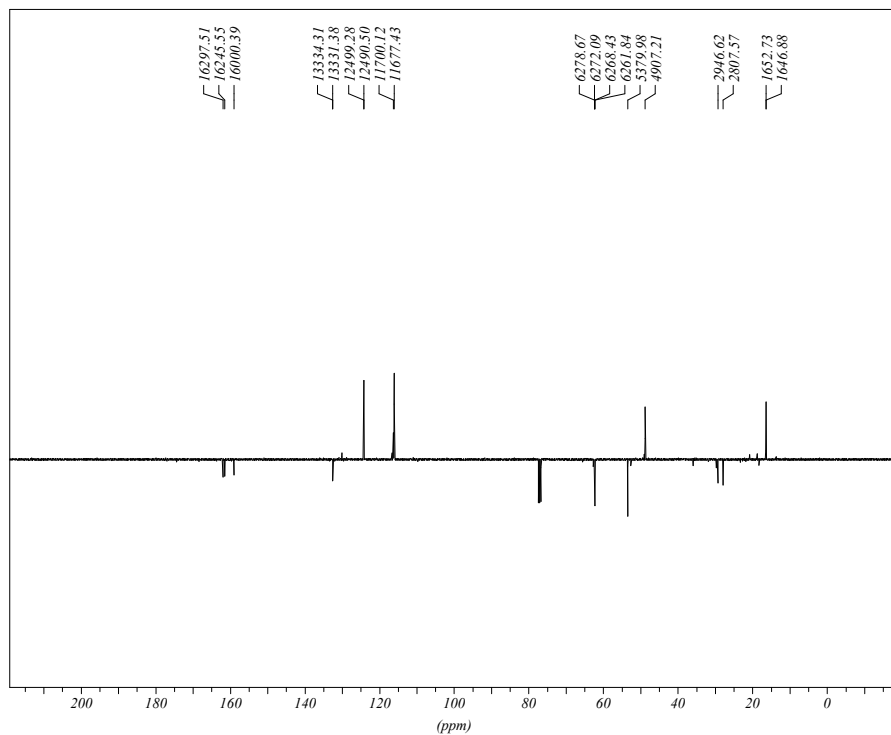
SOLVENT : CDCl3
 SW : 13.9775 ppm
 TD : 65536
 TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
 LB : 0.50 Hz
 SF : 400.1300000 MHz
 TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
 Stop : 0.00 ppm
 SR : 0.00 Hz
 SOLVENT : ?



*** Current Data Parameters ***

NAME : 501
 EXPNO : 1\
 PROCNO : 1
 *** Acquisition Parameters ***
 DATE_t : 12:50:42
 DATE_d : Jan 25 2012
 EXP : C13APT
 NS : 1024
 O1 : 10060.80 Hz
 PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
 RG : 16384.0000000
 SFO1 : 100.6228298 MHz

SOLVENT : CDCl3
 SW : 238.3238 ppm
 TD : 65536
 TE : 673.2 K

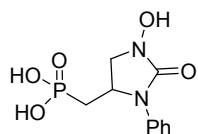
*** Processing Parameters ***

GB : 0.0000000
 LB : 0.50 Hz
 SF : 100.6127690 MHz
 TI :

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
 Stop : -19.18 ppm
 SR : 0.00 Hz
 SOLVENT : ?

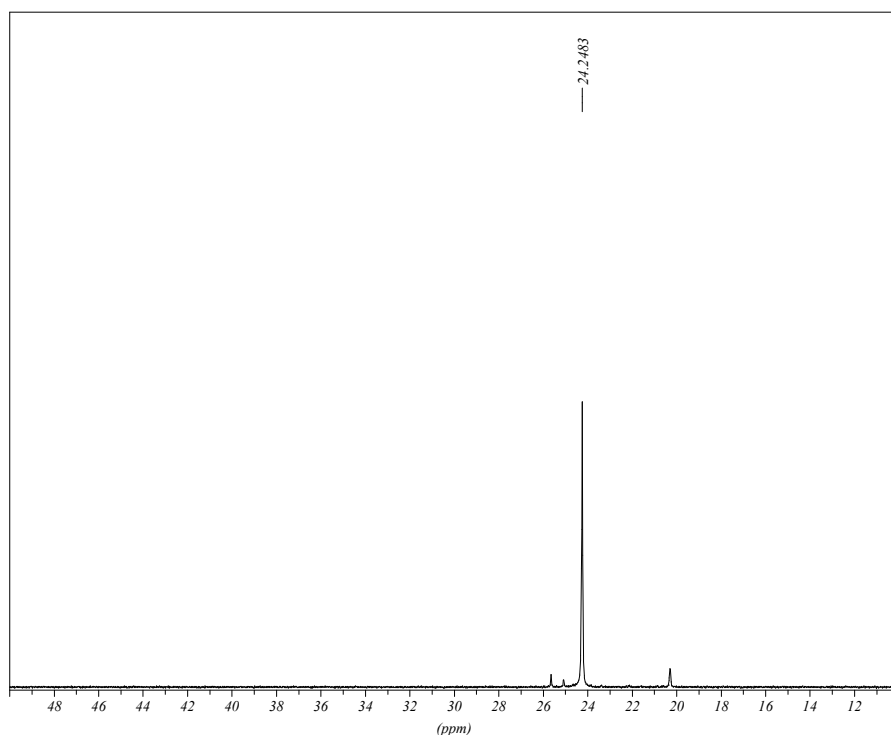
(±)-(1-hydroxy-3-phenyl-2-oxoimidazolidin-4-yl)methylphosphonic acid (**3b**)



3b

^1H NMR (400.13 MHz, D_2O) δ 1.69–1.91 (m, 2H), 3.26–3.30 (m, 1H), 3.64–3.68 (m, 1H), 4.15–4.24 (m, 1H), 7.04–7.09 (m, 3H), 7.18–7.22 (m, 2H). ^{13}C NMR (100.61 MHz, D_2O) δ 28.93 (d, $^1J_{\text{CP}} = 133.9$ Hz), 49.63 (d, $^2J_{\text{CP}} = 1.5$ Hz), 53.52 (s), 124.64 (s), 127.07 (s), 129.48 (s), 135.36 (s), 162.64 (s). ^{31}P NMR (161.97 MHz, D_2O) δ 24.25 (s). HRMS (ESI) m/z $[\text{M}+\text{H}]^+$ calcd for $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_5\text{P}$ 273.0639, found: 273.0640.

SM328-B4 P31CPD D2O opt/topspin am2n1 9



*** Current Data Parameters ***

NAME : 504

EXPNO : 4\

PROCNO : 1

*** Acquisition Parameters ***

DATE_t : 07:15:45

DATE_d : Nov 23 2011

EXP : PS done

NS : 16

O1 : 6479.02 Hz

PROBHD : 5 mm PABBO BB/19F-1H/D Z-G

RG : 9195.2001953

SFO1 : 161.9820520 MHz

SOLVENT : D2O

SW : 200.4391 ppm

TD : 65536

TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000

LB : 0.50 Hz

SF : 161.9755730 MHz

TI :

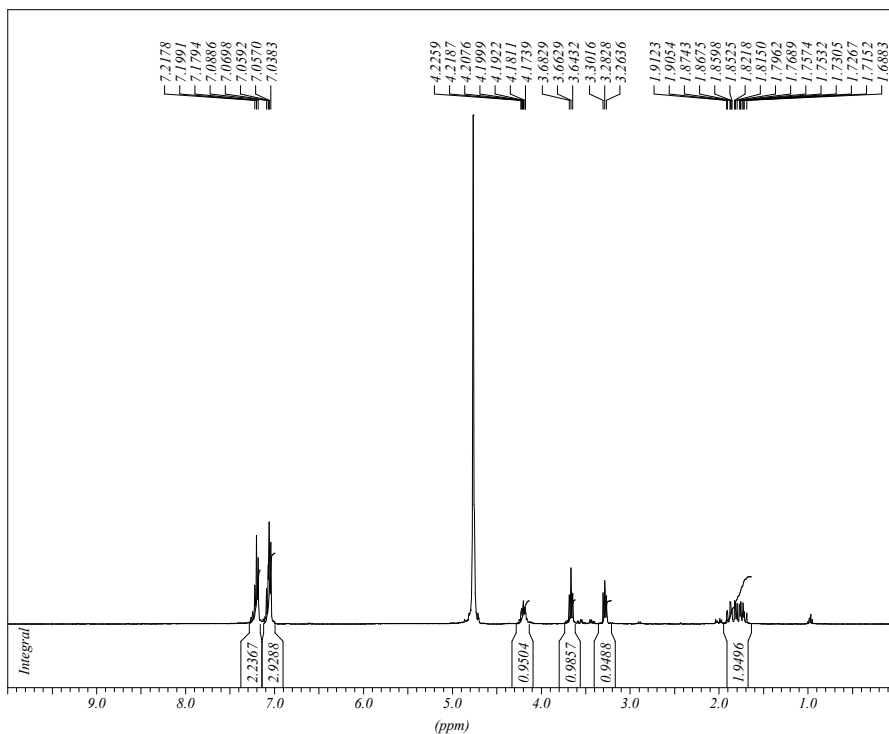
*** 1D NMR Plot Parameters ***

Start : 50.00 ppm

Stop : 10.00 ppm

SR : 0.01 Hz

SOLVENT : ?



*** Current Data Parameters ***

NAME : 503
EXPNO : 3
PROCNO : 1

*** Acquisition Parameters ***

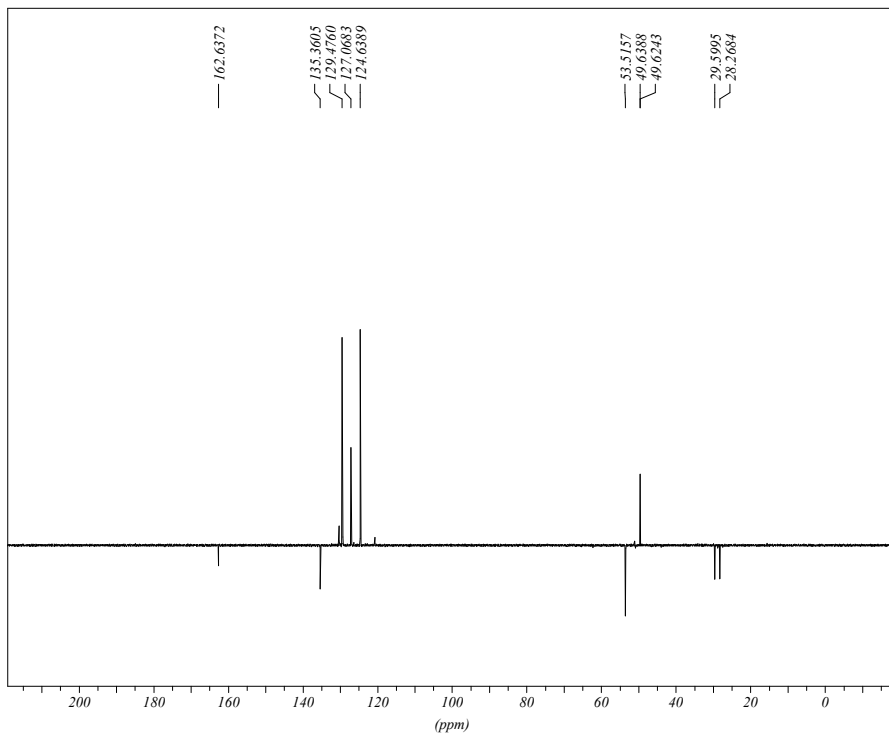
DATE_1 : 07:12:37
DATE_d : Nov 23 2011
EXP : PROTON
NS : 16
OI : 2400.78 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 57.0000000
SFO1 : 400.1324008 MHz
SOLVENT : D2O
SW : 13.9775 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 400.1300000 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 10.00 ppm
Stop : 0.00 ppm
SR : 0.00 Hz
SOLVENT : ?



*** Current Data Parameters ***

NAME : 511
EXPNO : 11
PROCNO : 1

*** Acquisition Parameters ***

DATE_1 : 14:44:07
DATE_d : Nov 23 2011
EXP : C13APT
NS : 1024
OI : 10060.80 Hz
PROBHD : 5 mm PABBO BB/19F-1H/D Z-G
RG : 26008.0000000
SFO1 : 100.6228298 MHz
SOLVENT : D2O
SW : 238.3238 ppm
TD : 65536
TE : 673.2 K

*** Processing Parameters ***

GB : 0.0000000
LB : 0.50 Hz
SF : 100.6127690 MHz
TI :

*** 1D NMR Plot Parameters ***

Start : 219.17 ppm
Stop : -19.18 ppm
SR : 0.00 Hz
SOLVENT : ?