Supplementary Material (ESI) for Journal of Materials Chemisty

Supporting information

Novel organosoluble filiform zirconium phosphonates with a layered mesoporous backbone

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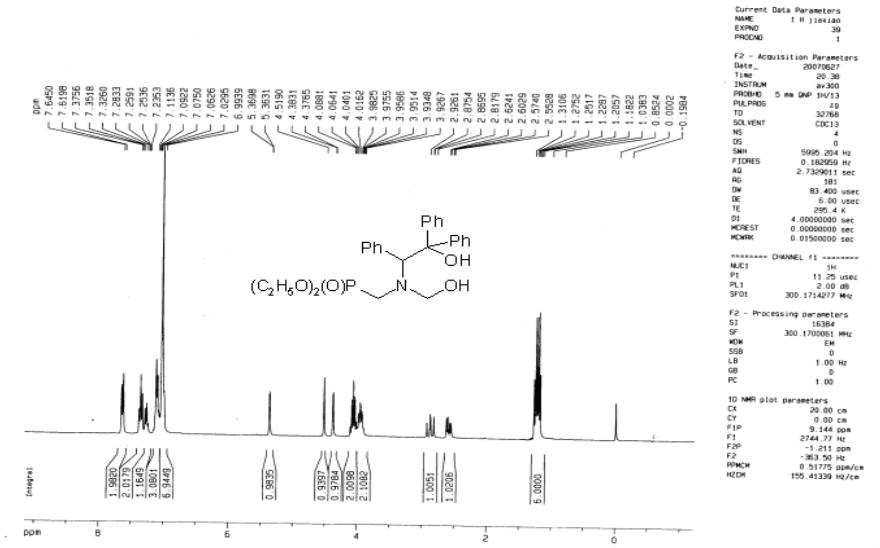
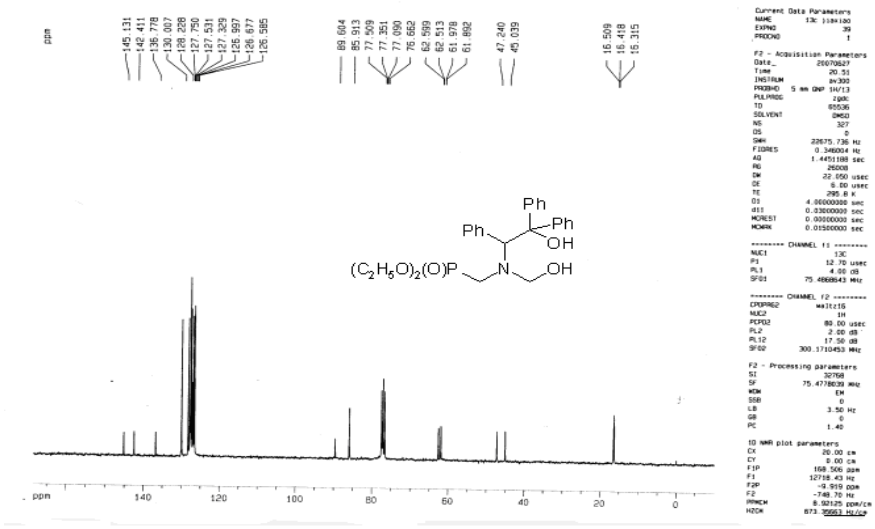
1. The NMR spectra of the compounds in the manuscript





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PROCNO 1

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NS 327
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RG 25008
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DE 6.00 usec
TE 295.6 K
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MCREST 0.00000000 sec
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PL1 4.00 dB
SF01 75.4868543 MHz

-------- CHANNEL f2 --------
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NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.50 dB
SF02 300.1710453 MHz

F2 - Processing parameters
SI 32768
SF 75.4778039 MHz
WDW EM
SSB 0
LB 3.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
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CY 8.00 cm
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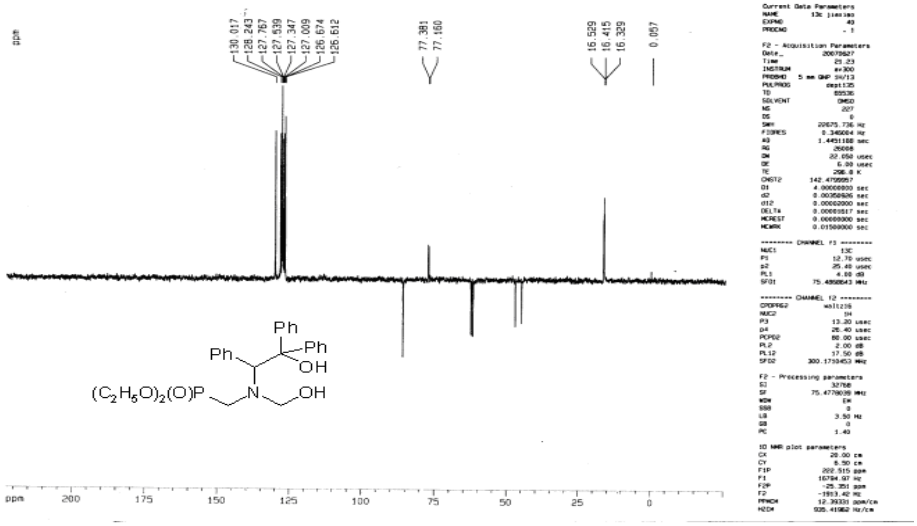
F2 -748.70 Hz
PPMCM 8.92125 ppm/cm
HZCM 673.35663 Hz/cm



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EXPNO 40
PROCNO 1

F2 - Acquisition Parameters
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PROBHD 5 mm DNP 1H/13
PULPROG dept135
TD 65536
SOLVENT DMSO
NS 207
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 25008
DW 22.050 usec
DE 6.00 usec
TE 296.8 K
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d2 0.00356936 sec
d12 0.00002000 sec
DELTA 0.00001817 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

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NUC1 13C
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p2 25.40 usec
PL1 4.00 dB
SF01 75.4868543 MHz

-------- CHANNEL f2 --------
CPDPRG2 waltz16
NUC2 1H
P3 13.20 usec
p4 26.40 usec
PCP02 80.00 usec
PL2 2.00 dB
PL12 17.50 dB
SF02 300.1710453 MHz

F2 - Processing parameters
SI 32768
SF 75.4778039 MHz
WDW EM
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LB 3.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
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CY 8.50 cm
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F2P -25.351 ppm
F2 -1913.42 Hz
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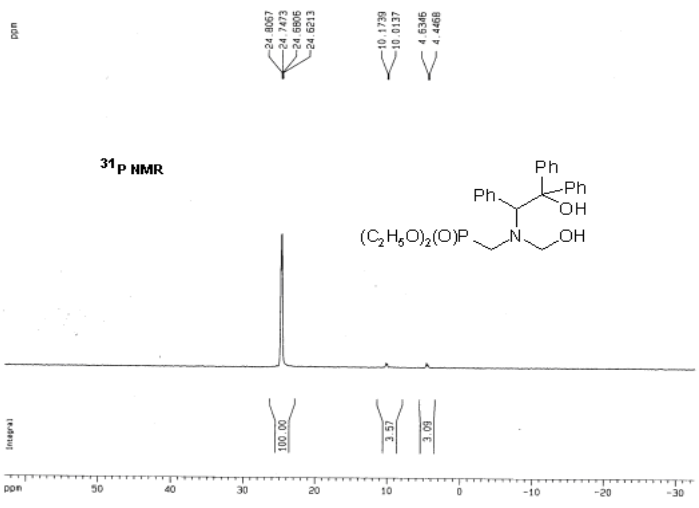
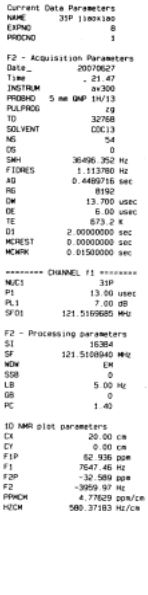
HZCM 935.41062 Hz/cm





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NAME 31P jiaxiao
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
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Time 21.47
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PROBHD 5 mm DNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 54
DS 0
SWH 36496.352 Hz
FIDRES 1.113780 Hz
AQ 0.4489716 sec
RG 8192
DW 13.700 usec
DE 6.00 usec
TE 273.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

-------- CHANNEL f1 --------
NUC1 31P
P1 13.00 usec
PL1 7.00 dB
SF01 121.5169685 MHz

F2 - Processing parameters
SI 16384
SF 121.5108940 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 8.00 cm
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F1 7647.46 Hz
F2P -32.589 ppm
F2 -3959.97 Hz
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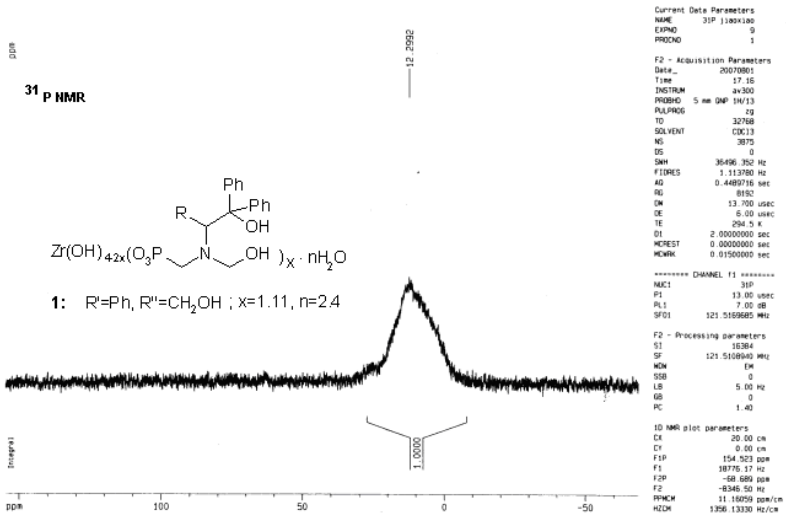
HZCM 580.37183 Hz/cm



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NAME 31P jiaxiao
EXPNO 9
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070801
Time 17.16
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 3875
DS 0
SWH 36496.352 Hz
FIDRES 1.113780 Hz
AQ 0.4489716 sec
RG 8192
DW 13.700 usec
DE 6.00 usec
TE 294.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 31P
P1 13.00 usec
PL1 7.00 dB
SFO1 121.5169685 MHz

F2 - Processing parameters
SI 16384
SF 121.5108940 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 154.523 ppm
F1 18775.17 Hz
F2P -68.689 ppm
F2 -8346.50 Hz
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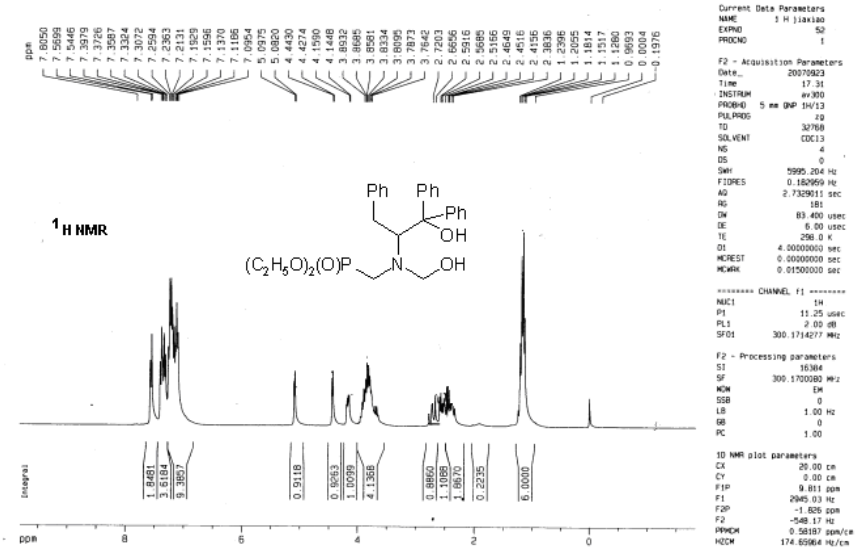
HZCM 1356.13330 Hz/cm



Current Data Parameters
NAME 1 H jiaxiao
EXPNO 52
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070923
Time 17.31
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 181
DW 83.400 usec
DE 6.00 usec
TE 298.0 K
D1 4.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 2.00 dB
SFO1 300.1714277 MHz

F2 - Processing parameters
SI 16384
SF 300.1700080 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
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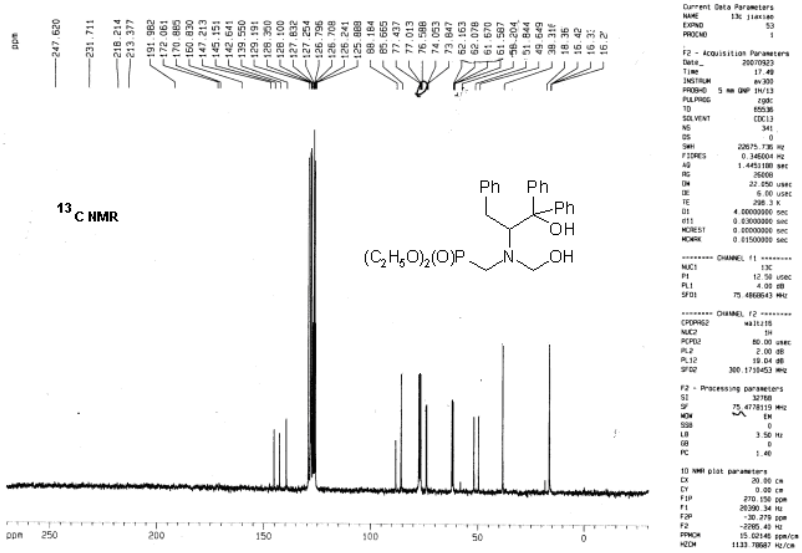
F1 2945.03 Hz
F2P -1.826 ppm
F2 -548.17 Hz
PPMCM 0.58187 ppm/cm
HZCM 174.65964 Hz/cm



Current Data Parameters
NAME 13c jiaxiao
EXPNO 53
PROCNO 1

F2 - Acquisition Parameters
Date_ 20070923
Time 17.49
INSTRUM av300
PROBHD 5 mm QNP 1H/13
PULPROG zgpg
TD 65536
SOLVENT CDCl3
NS 341
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451188 sec
RG 36000
DW 22.050 usec
DE 6.00 usec
TE 298.3 K
D1 4.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 4.00 dB
SFO1 75.4868643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 19.04 dB
SFO2 300.1710453 MHz

F2 - Processing parameters
SI 32768
SF 75.4778119 MHz
WDW EM
SSB 0
LB 3.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
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F2P -30.275 ppm
F2 -2285.40 Hz
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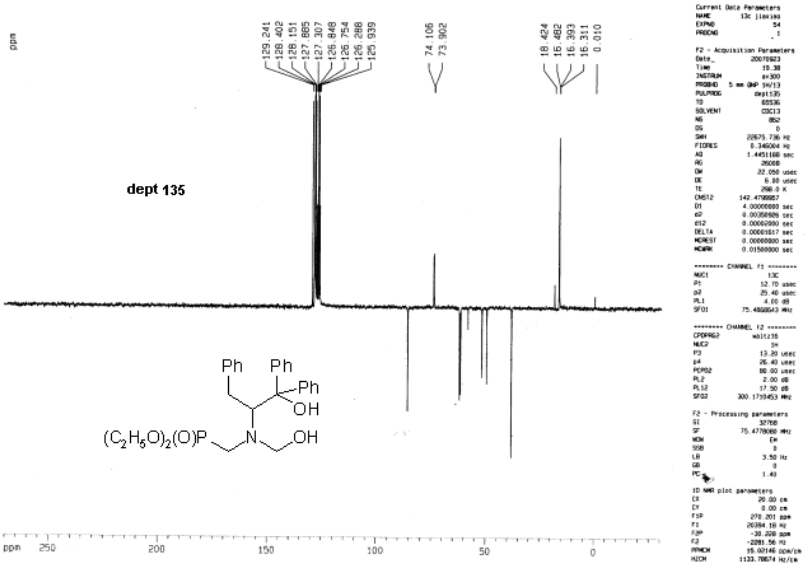
HZCM 1133.78687 Hz/cm



Current Data Parameters
NAME 13c jiaoxiao
EXPNO 54
PROCNO 1

F2 - Acquisition Parameters
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PULPROG dept135
TD 65536
SOLVENT CDCl3
NS 852
DS 0
SWH 20575.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451168 sec
RG 26008
DW 22.050 usec
DE 6.00 usec
TE 298.0 K
CNST2 142.4799857
D1 4.00000000 sec
d2 0.00350926 sec
d12 0.00002000 sec
DELTA 0.00001617 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.70 usec
p2 25.40 usec
PL1 4.00 dB
SFO1 75.4689643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
P3 13.80 usec
p4 26.40 usec
PCPD2 80.00 usec
PL2 2.00 dB
PL12 17.50 dB
SFO2 300.1730453 MHz

F2 - Processing parameters
SI 32768
SF 75.4778686 MHz
WDW EM
SSB 0
LB 3.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
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F2P -30.208 ppm
F2 -2281.56 Hz
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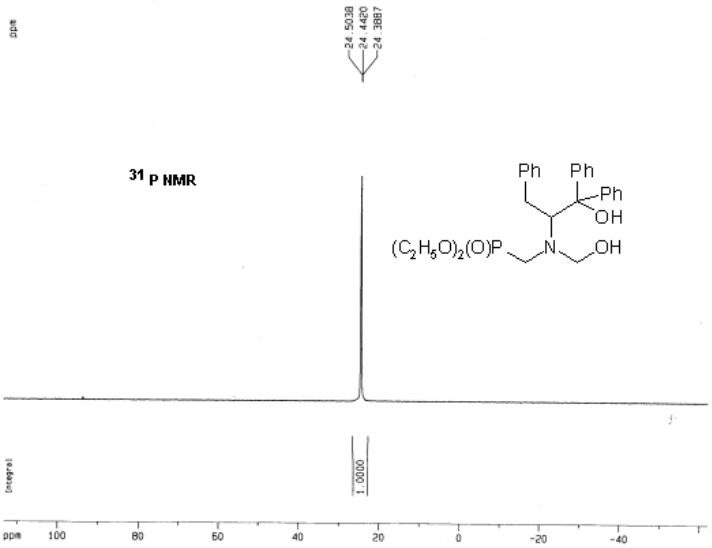

Current Data Parameters
NAME 31P jiaoxiao
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
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Time 17.37
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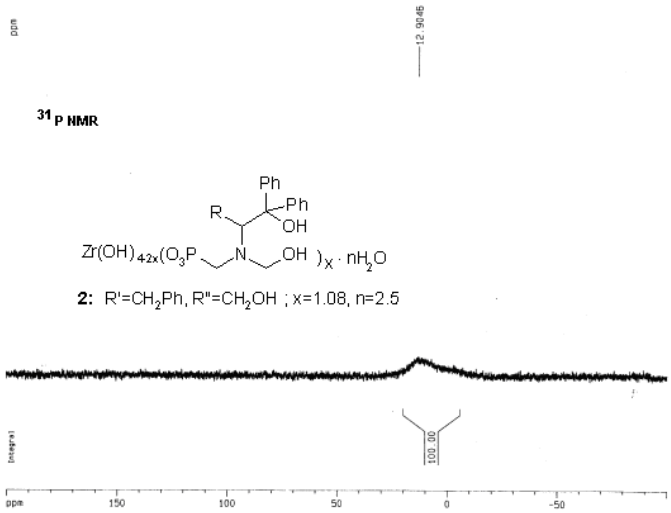
PROBHD 5 mm DNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 132
DS 0
SWH 36496.352 Hz
FIDRES 1.113760 Hz
AQ 0.4489716 sec
RG 8192
DW 13.700 usec
DE 6.00 usec
TE 297.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 31P
P1 13.00 usec
PL1 7.00 dB
SFO1 121.5169685 MHz

F2 - Processing parameters
SI 16384
SF 121.5108940 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 113.389 ppm
F1 13778.03 Hz
F2P -62.099 ppm
F2 -7545.68 Hz
PPMCM 8.77440 ppm/cm
HZCM 1066.16555 Hz/cm



Current Data Parameters
NAME 31P jiaoxiao
EXPNO 13
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080411
Time 2.46
INSTRUM av300
PROBHD 5 mm DNP 1H/13
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 6144
DS 0
SWH 36496.352 Hz
FIDRES 1.113760 Hz
AQ 0.4489716 sec
RG 8192
DW 13.700 usec
DE 6.00 usec
TE 298.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 31P
P1 13.00 usec
PL1 7.00 dB
SFO1 121.5169685 MHz

F2 - Processing parameters
SI 16384
SF 121.5108940 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
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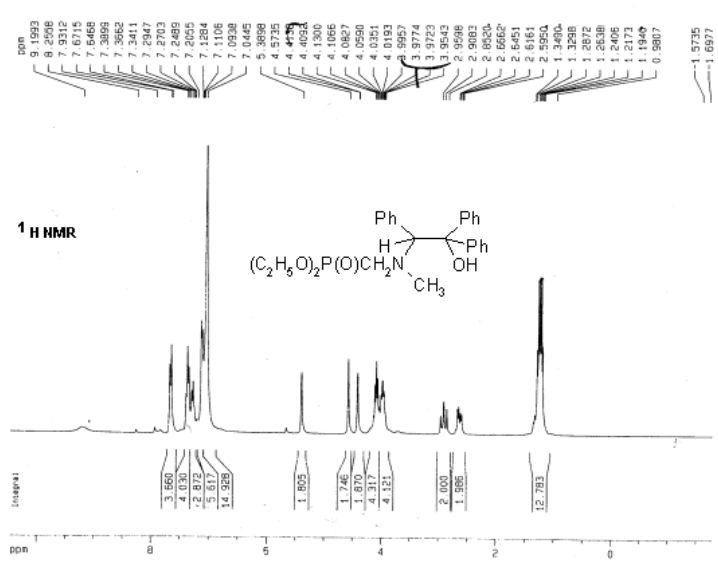
F1 24322.73 Hz
F2P -100.185 ppm
F2 -12173.62 Hz
PPMCM 15.01773 ppm/cm
HZCM 1824.81750 Hz/cm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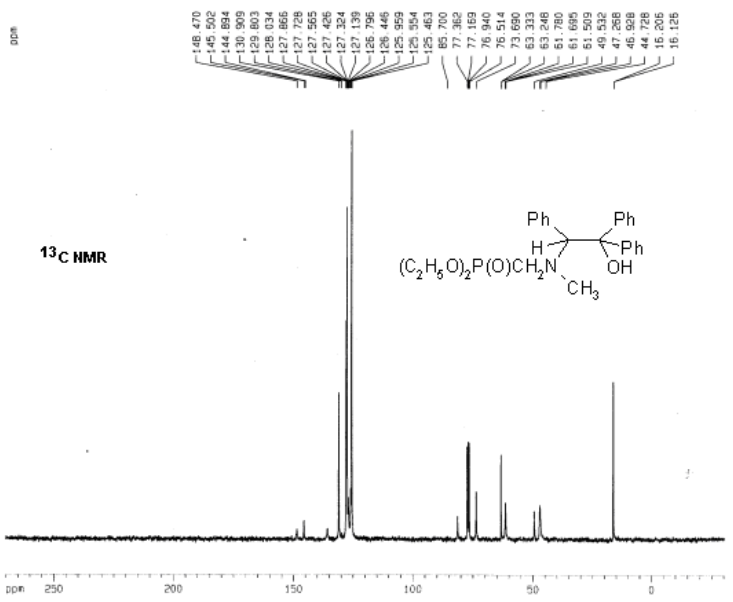
Ph
H
Ph
OH
(C2H5O)2P(O)CH2N
CH3


13C NMR
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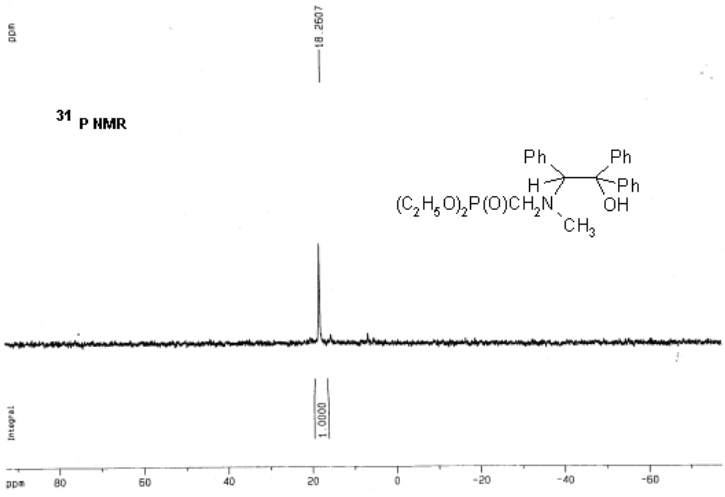
Ph
H
Ph
OH
(C2H5O)2P(O)CH2N
CH3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Ph
H
Ph
OH
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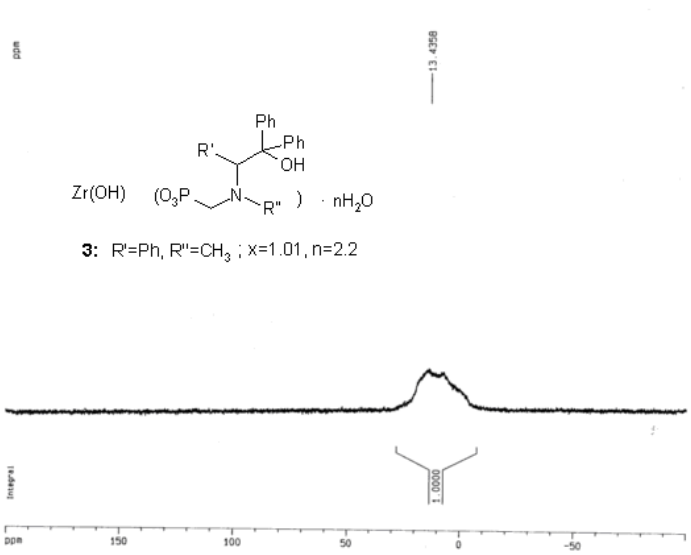
CH3
1.0000



3: R'=Ph, R''=CH_3 ; x=1.01, n=2.2

Current Data Parameters
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EXPNO 4
PROCNO 1

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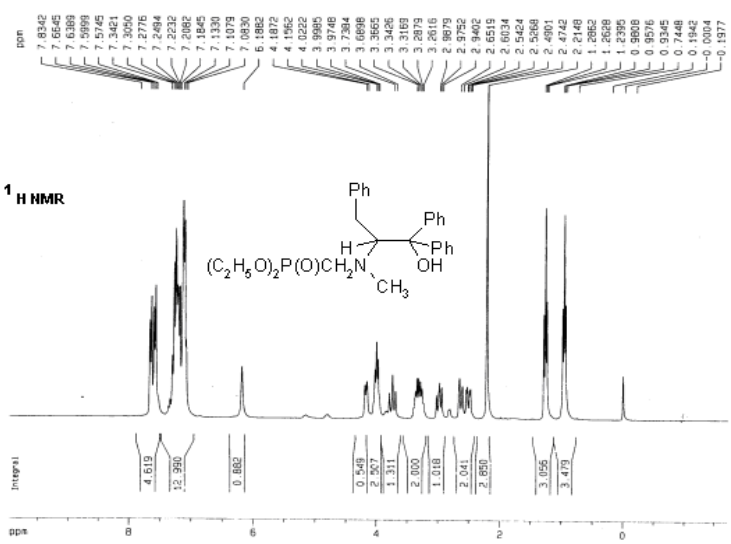
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 685
DS 0
SWH 36496.352 Hz
FIDRES 1.113780 Hz
AQ 0.4489716 sec
RG 1625.5
DW 13.700 usec
DE 6.00 usec
TE 294.8 K
D1 5.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 31P
P1 13.00 usec
PL1 7.00 dB
SFO1 121.5169685 MHz

F2 - Processing parameters
SI 16384
SF 121.5108940 MHz
WDW EM
SSB 0
LB 5.00 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 200.169 ppm
F1 24322.73 Hz
F2P -100.185 ppm
F2 -12173.62 Hz
PPMCM 15.01773 ppm/cm
HZCM 1824.81750 Hz/cm



^{1}H NMR

NAME 1 H jiaxiao
EXPNO 58
PROCNO 1

F2 - Acquisition Parameters
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Time 20.22
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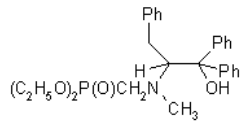
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 4
DS 0
SWH 5995.204 Hz
FIDRES 0.182959 Hz
AQ 2.7329011 sec
RG 25.4
DW 83.400 usec
DE 6.00 usec
TE 295.0 K
D1 4.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

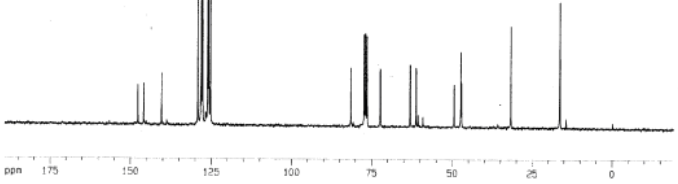
======== CHANNEL f1 ========
NUC1 1H
P1 11.25 usec
PL1 2.00 dB
SFO1 300.1714076 MHz

F2 - Processing parameters
SI 16384
SF 300.1700094 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.00

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 9.954 ppm
F1 2988.03 Hz
F2P -1.721 ppm
F2 -516.55 Hz
PPMCM 0.58377 ppm/cm
HZCM 175.22929 Hz/cm

^{13}C NMR





NAME 13c jiaxiao
EXPNO 62
PROCNO 1

F2 - Acquisition Parameters
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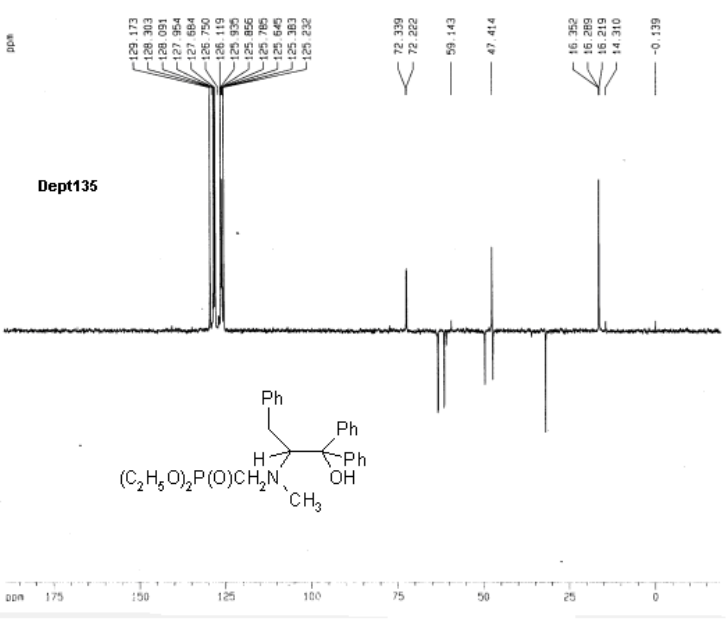
INSTRUM av300
PROBHD 5 mm GNP 1H/13
PULPROG zgdc
TD 65536
SOLVENT CDCl3
NS 520
DS 0
SWH 22675.736 Hz
FIDRES 0.346004 Hz
AQ 1.4451888 sec
RG 26000
DW 22.050 usec
DE 6.00 usec
TE 294.9 K
D1 4.00000000 sec
D11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 12.50 usec
PL1 4.00 dB
SFO1 75.4868643 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 2.00 dB
PL12 19.04 dB
SFO2 300.1703453 MHz

F2 - Processing parameters
SI 32768
SF 75.4778209 MHz
WDW EM
SSB 0
LB 3.50 Hz
GB 0
PC 1.40

1D NMR plot parameters
CX 20.00 cm
CY 0.00 cm
F1P 189.127 ppm
F1 14274.91 Hz
F2P -19.052 ppm
F2 -1438.00 Hz
PPMCM 10.40896 ppm/cm
HZCM 785.64539 Hz/cm




```
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EXPNO                  63
PROCNO                  1

F2 - Acquisition Parameters
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Time               21.28
INSTRUM            av300
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PULPROG           dept135
TD                 65536
SOLVENT             CDCl3
NS                    323
DS                      0
SWH            22675.736 Hz
FIDRES          0.346004 Hz
AQ              1.4451388 sec
RG                  28050
DW                 22.050 usec
DE                  6.00 usec
TE                294.4 K
CNST2          142.4799957
D1              4.00000000 sec
d2              0.00356905 sec
d12             0.00002000 sec
DELTA           0.00001592 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.50 usec
p2                  25.00 usec
PL1                 4.00 dB
SFO1           75.4688643 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
P3                  11.25 usec
p4                  22.50 usec
PCPD2               80.00 usec
PL2                 2.00 dB
PL12                19.04 dB
SFO2          300.1718453 MHz

F2 - Processing parameters
SI                     32768
SF             75.4778091 MHz
WDW                       EM
SSB                        0
LB                      3.50 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                      6.80 cm
F1P                   189.127 ppm
F1                  14274.91 Hz
F2P                  -19.852 ppm
F2                  -1498.88 Hz
PPMCM              10.40896 ppm/cm
HZCM              785.84538 Hz/cm
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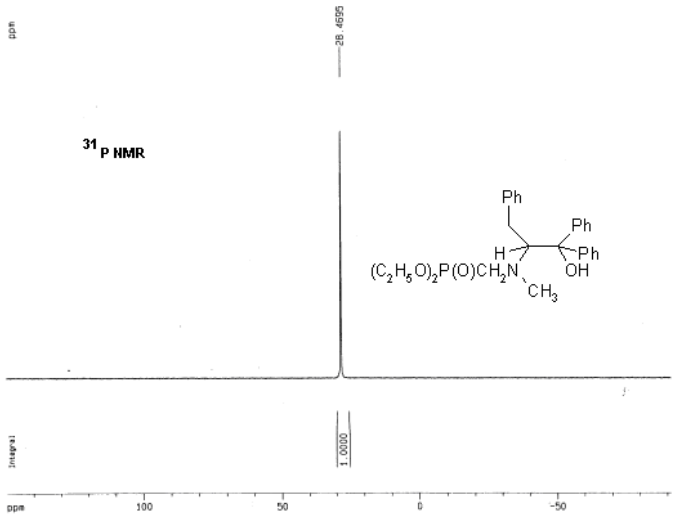


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Current Data Parameters
NAME          31P jiaxiao
EXPNO                  16
PROCNO                  1

F2 - Acquisition Parameters
Date_           20071116
Time               21.52
INSTRUM            av300
PROBHD  5 mm QNP 1H/13
PULPROG                zg
TD                 32768
SOLVENT             CDCl3
NS                     68
DS                      0
SWH            36496.352 Hz
FIDRES          1.113780 Hz
AQ              0.4489716 sec
RG                   8192
DW                 13.700 usec
DE                  6.00 usec
TE                294.6 K
D1              2.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  31P
P1                  13.00 usec
PL1                 7.00 dB
SFO1          121.5169685 MHz

F2 - Processing parameters
SI                     16384
SF            121.5108940 MHz
WDW                       EM
SSB                        0
LB                      5.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                   149.863 ppm
F1                  18210.04 Hz
F2P                  -91.308 ppm
F2                  -11094.92 Hz
PPMCM              12.05857 ppm/cm
HZCM             1465.24756 Hz/cm
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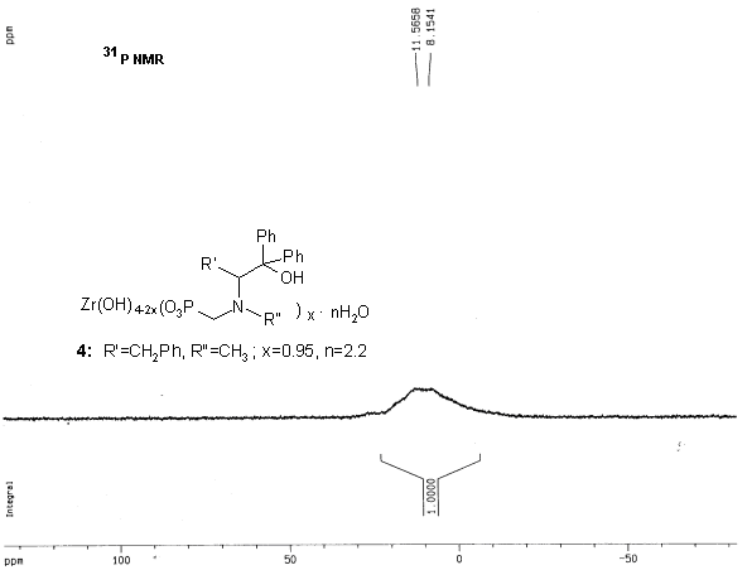


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EXPNO                  7
PROCNO                 1

F2 - Acquisition Parameters
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TD                 32768
SOLVENT              D2O
NS                  2418
DS                     0
SWH            36496.352 Hz
FIDRES          1.113780 Hz
AQ              0.4489716 sec
RG                 1625.5
DW                 13.700 usec
DE                  6.00 usec
TE                297.1 K
D1              5.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  31P
P1                  13.00 usec
PL1                 7.00 dB
SFO1          121.5169685 MHz

F2 - Processing parameters
SI                     16384
SF            121.5108940 MHz
WDW                       EM
SSB                        0
LB                      5.00 Hz
GB                         0
PC                      1.40

1D NMR plot parameters
CX                     20.00 cm
CY                      0.00 cm
F1P                   134.574 ppm
F1                  16352.26 Hz
F2P                   -82.431 ppm
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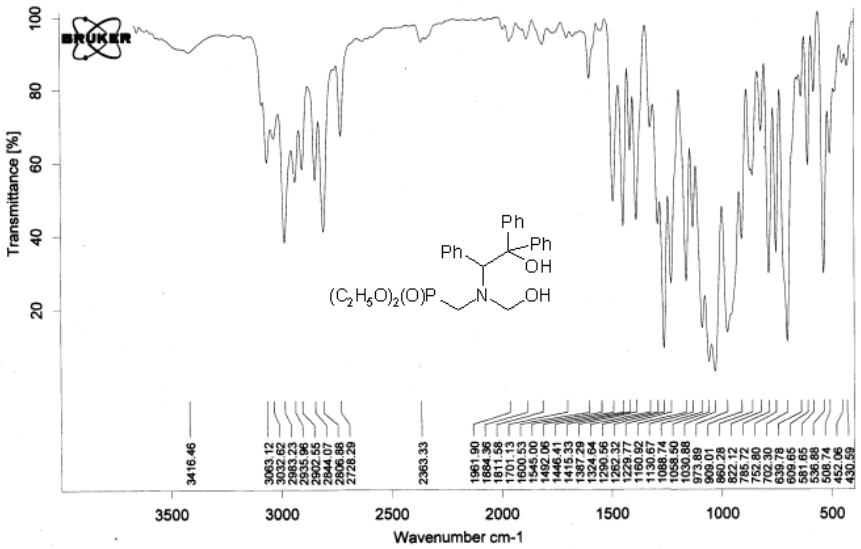
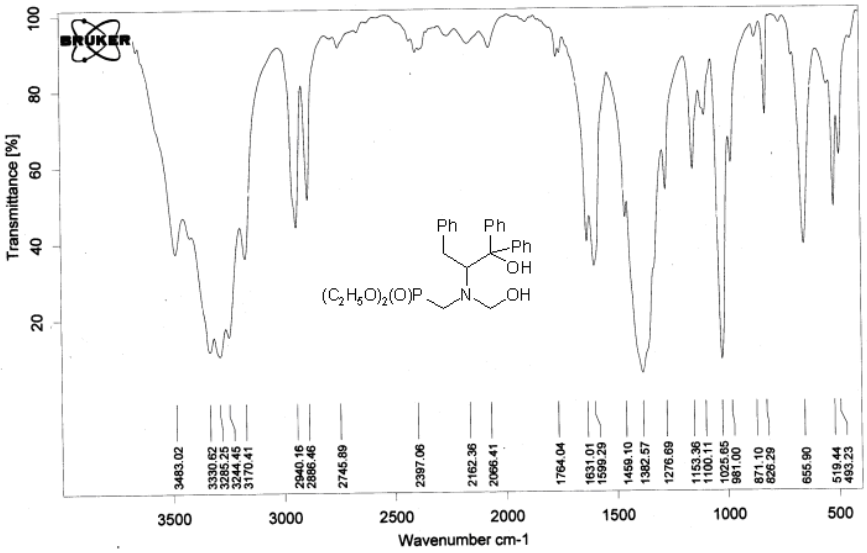
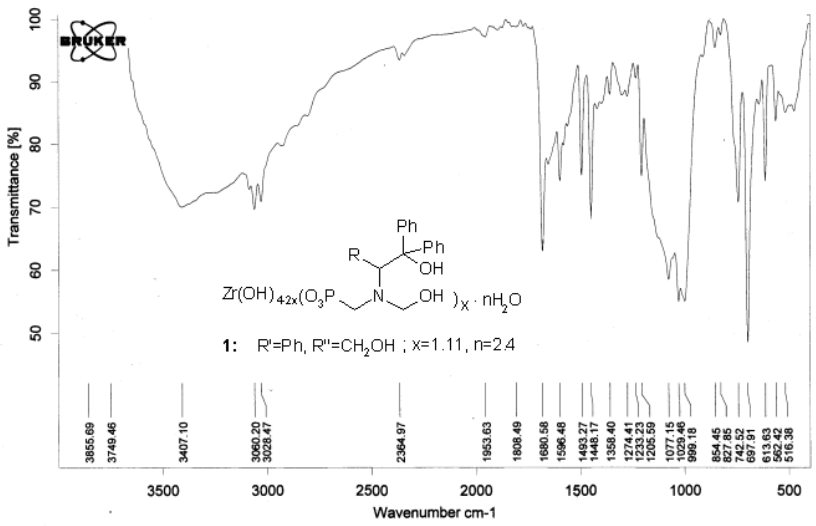
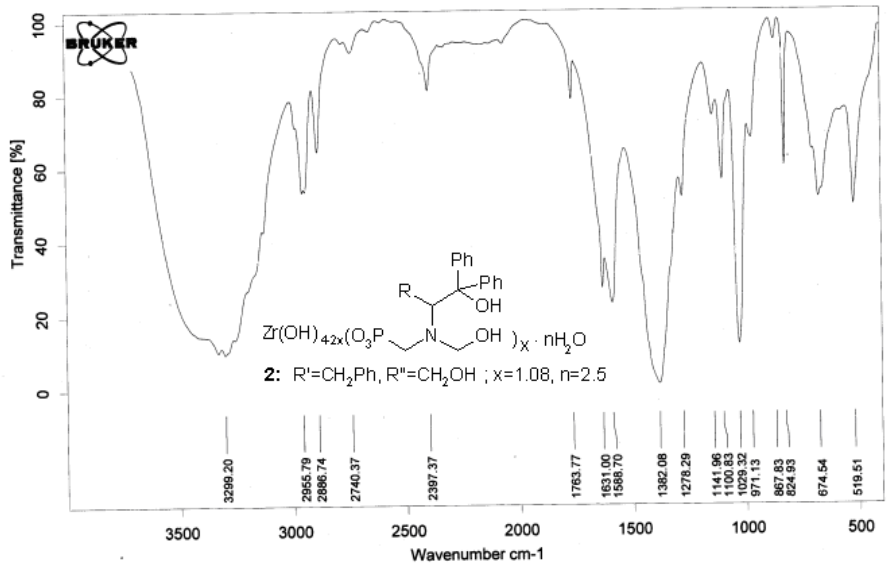
2. The IR spectra of the compounds in the manuscript





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2740.37
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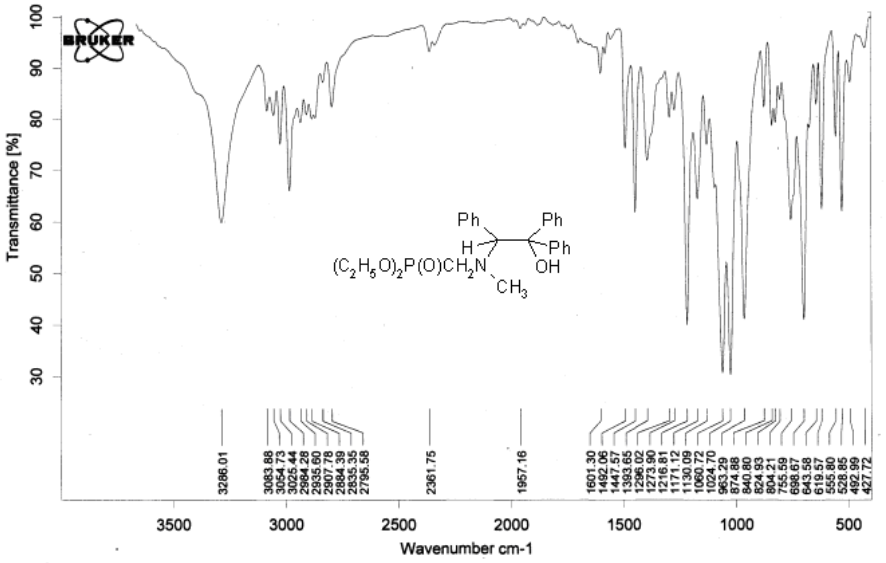
1631.00
1588.70
1382.08
1278.29
1141.96
1100.83
1029.32
971.13
867.83
824.93
674.54
519.51
2: R'=CH2Ph, R''=CH2OH ; x=1.08, n=2.5


Transmittance [%]
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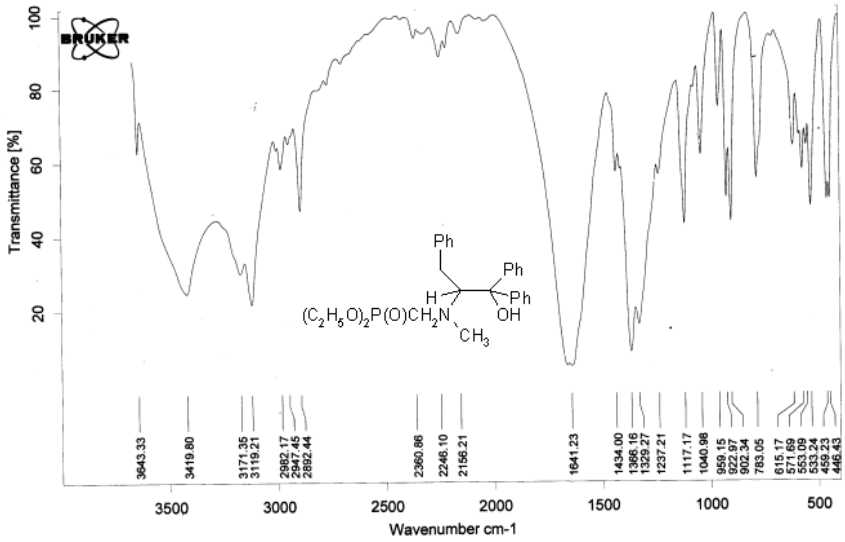
1957.16


Transmittance [%]
Wavenumber cm-1
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3119.21
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2892.44
2360.86
2246.10
2156.21
1641.23
1434.00
1366.16
1329.27
1237.21
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533.24
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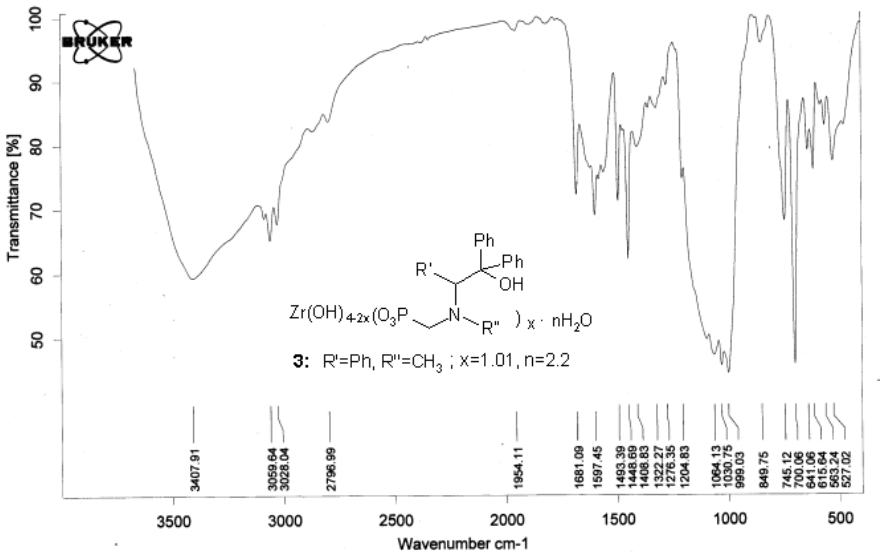
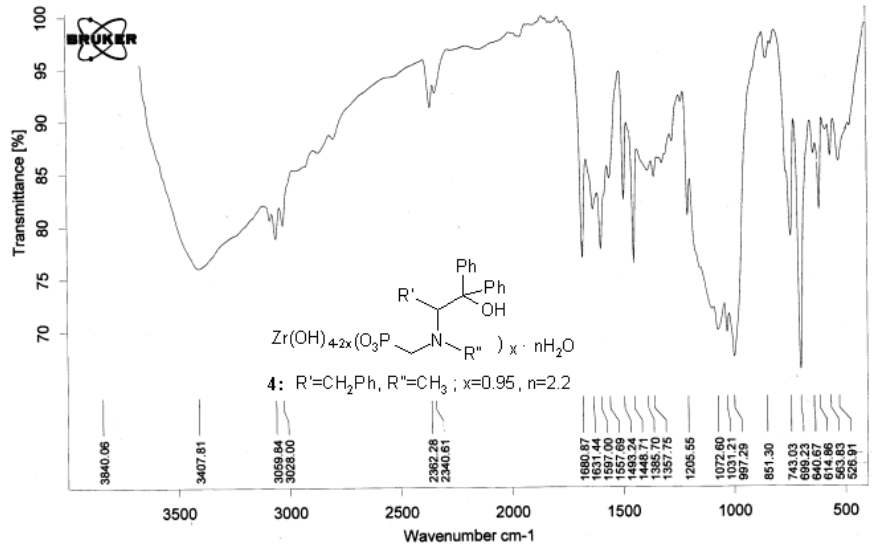
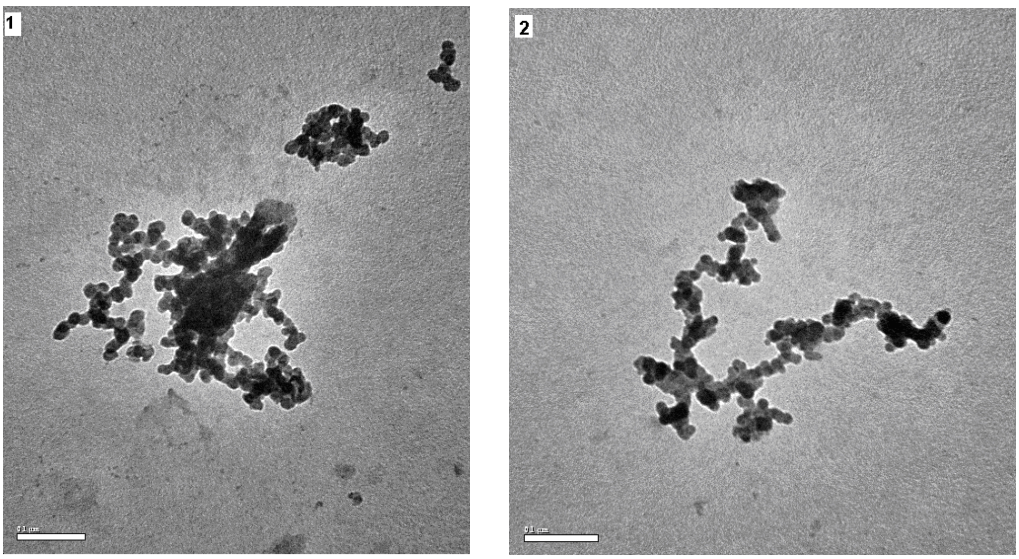
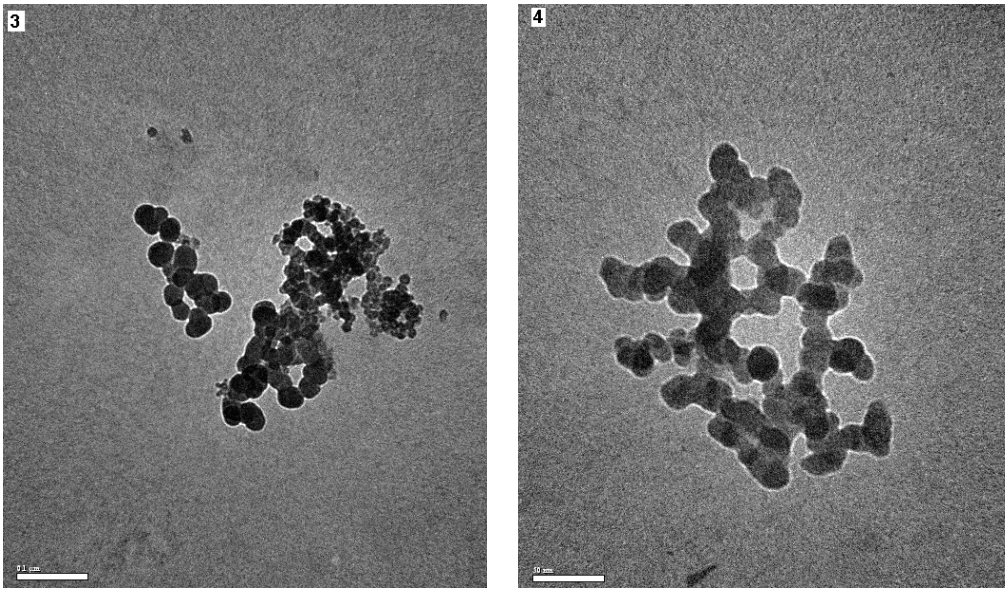




3. The TEM images of the organosoluble zirconium phosphonates

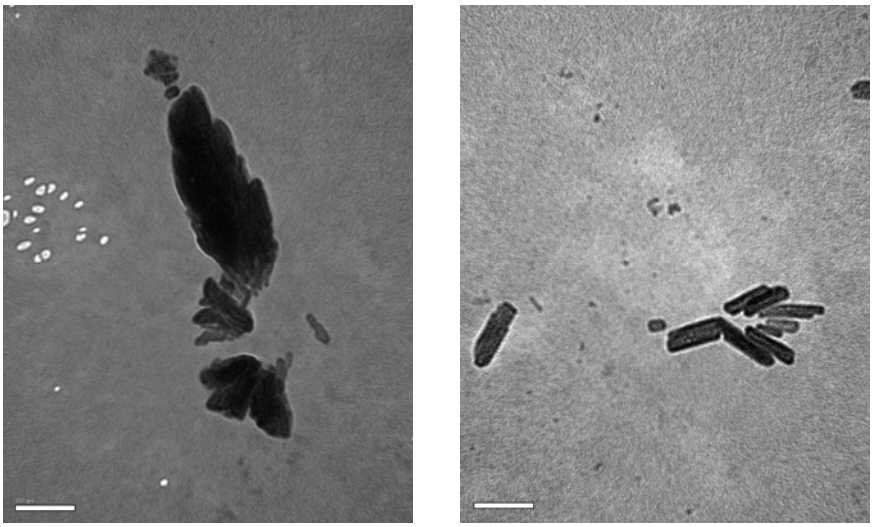


TEM image of zirconium phosphonates **1**and **2**



TEM image of zirconium phosphonate **3** and **4**

4. The TEM images of the insoluble zirconium phosphonate



TEM image of the insoluble zirconium phosphonate **2**