Heptane-Soluble Ring-Closing Metathesis Catalysts

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Supporting Information

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General Experimental: The ^1H NMR spectra were recorded on an Inova 500MHz spectrometer operating at 499.95 MHz. ^{13}C NMR spectra were recorded on an Inova 500MHz spectrometer operating at 125.719 MHz. Chemical shifts were reported in parts per million (δ) relative to residual proton resonances in the deuterated chloroform (CDCl_3). Coupling constants (J values) were reported in hertz (Hz), and spin multiplicities are indicated by the following symbols: s (singlet), d (doublet), t (triplet), q (quartet), and m (multiplet). UV-Vis spectra were obtained using a Varian Cary 100 spectrometer. IR spectra were obtained using a Bruker Tensor 27 FT-IR. ICP-MS were obtained using a Perkin Elmer DRC II instrument.

All reactions were carried out under an inert atmosphere unless otherwise noted. Acetonitrile, heptane, dimethylformamide, ethanol, dichloromethane, and toluene were purchased from EMD and used as received. All chemicals were purchased from Sigma-Aldrich and used as received. Products were isolated simply by evaporation of solvent without chromatography. The product spectra contain traces of alkane solvents, residual polyisobutylene, or water.

Synthesis of PIB Supported Grubbs-Hoveyda^{2nd} Generation Catalyst

4-(polyisobutyl)phenol: A mixture of 16.75 g (178 mmol) of phenol, 8.9 g (8.9 mmol) of polyisobutylene (Glissopal[®] 1000), and 1.05 g (10.7 mmol) of concentrated sulfuric acid in 200 mL of dichloromethane was stirred for 3 d at room temperature. The solvent was removed under reduced pressure and then 250 mL of hexanes was added to the viscous residue. The hexanes solution was washed first with 150 mL of dimethyl formamide three times and then with 150 mL of 90% ethanol/water three times. The hexane phase was dried over sodium sulfate. The solvent was removed under reduced pressure to yield a light yellow viscous residue. The yield was 70%. ¹H NMR (500 MHz, CDCl₃), δ : 0.8-1.8 (m, 138H), 1.8 (s, 2H), 6.75 (d, J = 8.79 Hz, 2H), and 7.23 (d, J = 8.79 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃), δ : 153.19, 142.99, 127.52, 114.75, multiple peaks between 58 – 60, 38-38.5, and 30.75 – 33.

2-hydroxy-5-(polyisobutyl)benzaldehyde: A mixture of 3.42 g (3.13 mmol) of 4-(polyisobutyl)phenol and 0.58 mL (5 mmol) of 2,6-lutidine in 40 mL of toluene was stirred under room temperature for 30 min. A solution of SnCl₄ (0.15 mL, 1.25 mmol) in 10 mL of toluene was added slowly to the reaction. The reaction was then stirred under room temperature for 1 h at which point 0.56 g (18.78 mmol) of paraformaldehyde was added to reaction. The reaction was heated for 12 h at 100 °C. After the reaction mixture cooled to room temperature, it was acidified to pH 2.0 with 2M HCl. The organic layer was separated, the solvent was removed under reduced pressure, and then 250 mL of hexanes was added to the viscous residue. The hexanes solution was washed

first with 150 mL of dimethyl formamide three times and then with 150 mL of 90% ethanol/water three times. The hexane phase was dried over sodium sulfate. The solvent was removed under reduced pressure to yield a light yellow viscous residue. The yield was 76%. ^1H NMR (500 MHz, CDCl_3), δ : 0.8-1.8 (m, 155H), 1.8 (s, 2H), 6.94 (d, $J = 8.54$ Hz, 1H), 7.48 (m, 1H), 7.57 (dd, $J = 2.44, 8.54$ Hz, 1H), 9.9 (s, 1H), and 10.88 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3), δ : 197.07, 159.65, 142.40, 135.74, 130.73, 120.21, 117.24, multiple peaks between 58 – 60, 38-38.5, and 30.75 – 33.

2-isopropoxy-5-(polyisobutyl)benzaldehyde: A mixture of 2.5 g (2.25 mmol) of 2-hydroxy-5-(polyisobutyl)benzaldehyde and 0.425 g (2.5 mmol) of isopropyl iodide in 10 mL of DMF and 10 mL of

heptane was heated to 80 °C over night, then cooled to room temperature. At this point, 50 mL of hexanes was added to the solution. The hexanes solution was washed first with 30 mL of dimethyl formamide three times and then with 30 mL of 90% ethanol/water three times. The hexanes phase was dried over sodium sulfate. The solvent was removed under reduced pressure to yield the product as a light yellow viscous residue in 76% yield. ^1H NMR (500 MHz, CDCl_3), δ : 0.8-1.8 (m, 168H), 1.8 (s, 2H), 4.66 (m, 1H), 6.92 (d, $J = 9.04$ Hz, 1H), 7.53 (dd, $J = 2.69, 8.79$ Hz, 1H), 7.82 (d, $J = 2.69$ Hz, 1H), and 10.49 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3), δ : 190.68, 158.75, 142.92, 134.06, 125.63, 125.21, 113.83, 71.34 multiple peaks between 58 – 60, 38-38.5, and 30.75 – 33.

1-isopropoxy-4-(polyisobutyl)-2-vinylbenzene: Methyltriphenylphosphonium iodide (3.21 g, 7.96 mmol) was added to 30 mL of

THF. Then 5 mL of 1.6M BuLi was added slowly to the solution. After 2 h stirring the solution turned bright yellow. The solution was cooled to -78 °C and a solution of 4.6 g (3.98 mmol) of 2-isopropoxy-5-(polyisobutyl)benzaldehyde ether in THF was added slowly to the mixture. The reaction was stirred overnight. The solvent was removed under reduced pressure and then 100 mL of hexanes was added to the viscous residue. The hexanes solution was washed first with 75 mL of dimethyl formamide three times and then with 75 mL of 90% ethanol/water three times. The hexanes phase was dried over sodium sulfate. The solvent was removed under reduced pressure to yield the product as a light yellow viscous residue in 80% yield. ¹H NMR (500 MHz, CDCl₃), δ: 0.8-1.8 (m, 168 H), 1.8 (s, 2 H), 4.51 (m, 1H), 5.73 (dd, *J* = 1.71, 11.23 Hz, 1H), 5.73 (dd, *J* = 1.46, 17.82 Hz, 1H), 6.81 (d, *J* = 8.54 Hz, 1H), 7.07 (dd, *J* = 10.98, 17.82 Hz, 1H), 7.19 (dd, *J* = 2.45, 8.55 Hz, 1H), and 7.46 (d, *J* = 2.44 Hz 1H). ¹³C NMR (125 MHz, CDCl₃), δ: 153.13, 142.54, 132.96, 127.11, 126.71, 124.62, 113.98, 113.67, 71.11, multiple peaks between 58 – 60, 38-38.5, and 30.75 – 33.

PIB supported Grubbs-Hoveyda 2nd generation catalyst: A

mixture of 2 g (1.72 mmol) of 1-isopropoxy-4-(polyisobutyl)-2-vinylbenzene, 0.12 g (1.21 mmol) of CuCl, and 0.73 g (0.86 mmol) of the 2nd generation Grubbs catalyst in

dichloromethane was stirred at 40 °C overnight. The solution turned from red to green as the reaction progressed. The solvent was removed under reduced pressure and the residue was purified by column chromatography (2:1 hexane:dichloromethane) to yield a dark green viscous solution. The solvent was removed under reduced pressure resulted in dark green viscous residue of product (87% yield). ¹H NMR (500 MHz, CDCl₃), δ: 0.8-1.8 (m, 156H), 1.8 (s, 2H),

2.42-2.49 (m, 18H), 4.20 (s, 4 H), 4.87 (m, 1H), 6.71 (d, $J = 8.7$ Hz, 1H), 6.87 (d, $J = 1.83$ Hz, 1H), 7.09 (s, 4H), 7.48 (dd, $J = 2.06, 9.15$ Hz, 1H), and 16.32 (s, 1H). ^{13}C NMR (125 MHz, CDCl_3), δ : 298.35 (m), 212.65, 150.40, 145.13, 145.11, 138.97, 129.58, 129.57, 129.55, 127.55, 120.59, 112.25, 74.81, multiple peaks between 58 – 60, 38-38.5, and 30.75 – 33. $\lambda_{\text{max}} = 591$ nm. IR (KBr, br: broad, s: strong, m: medium, w: weak): 2954 (br), 2870 (br), 1729 (w), 1612 (w), 1592 (w), 1488 (s), 1455 (m), 1391 (s), 1367 (s), 1297 (m), 1267 (s), 1232 (s), 1139 (m), 1108 (m), 1037 (m), 927 (m), 854 (m), 815 (m), 739 (S), 704 (m), 649 (m), 621 (w), 580 (m)

Procedures and Product Characterization Data for Ring Closing Metathesis Reactions

A mixture of 120 mg (0.5 mmol) substrate **8** and 40 mg (0.025 mmol) of catalyst **7** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h. Once the reaction was completed, 3 mL of acetonitrile was added to the reaction mixture and biphasic mixture was stirred vigorously. After a gravity separation, the acetonitrile layer was removed. The solvent was removed from this phase under reduced pressure to yield compound **9**¹. ¹H NMR (500 MHz, CDCl₃), δ : 1.24 (t, J = 7.33 Hz, 6H), 3.0 (s, 4H), 4.19 (q, J = 7.32 Hz, 4H), and 5.60 (m, 2H). ¹³C NMR (125 MHz, CDCl₃), δ : 172.49, 128.05, 61.76, 59.07, 41.08, and 14.27.

A mixture of 127 mg (0.5 mmol) substrate **16** and 40 mg (0.025 mmol) of catalyst **7** were dissolved in 5 mL of heptane. The reaction was stirred for 1 hour. Once the reaction was completed, 3 mL of acetonitrile was added to the reaction mixture and stirred vigorously. After both phase of the solvent is separated, the acetonitrile layer was taken out. The heptane layer was recycled in the subsequent reactions. The solvent was removed under reduced pressure to yield compound **17**². ¹H NMR (500 MHz, CDCl₃), δ : 1.25 (m, 6H), 2.12 (m, 4H), 2.56 (s, 2H), 4.19 (m, 4H), and 5.68 (m, 2H). ¹³C NMR (125 MHz, CDCl₃), δ : 171.84, 126.31, 124.24, , 61.49, 53.16, 30.64, 27.57, 22.54, and 14.27.

In a second procedure, a RCM metathesis reaction was carried out where the product self-separated from the heptane solution of the catalyst. In this reaction, a mixture of 125 mg (0.5 mmol) of the substrate **10**¹ and 40 mg (0.025 mmol) of catalyst **7** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h. As the reaction proceeded, the product **11**¹ precipitated out of solution; therefore it was separated from the reusable heptane solution of the catalyst and dried under reduced pressure. The heptane layer was directly recycled in subsequent reactions. ¹H NMR (500 MHz, CDCl₃), δ : 2.44 (s, 3H), 4.13 (s, 4H), 5.66 (s, 2H), 7.33 (d, J = 8.1 Hz, 2H), and 7.73 (d, J = 8.3 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃), δ : 143.68, 134.53, 130.0, 127.67, 125.70, 55.08, and 21.78. M.P. range = 123.2 – 126.5°C

A second example of a self-separating product involved the diene **12**. A mixture of 125 mg (0.5 mmol) of the substrate **12**¹ and 40 mg (0.025 mmol) of catalyst **7** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h. As the reaction proceeded, product **13**¹ precipitated out of solution. It was separated from the catalyst solution by filtration and dried under reduced pressure. The heptane layer was directly recycled in subsequent reactions. ¹H NMR (500 MHz, CDCl₃), δ : 1.66 (s, 3H), 2.43 (s, 3H), 3.97 (m, 2H), 4.07 (m, 2H), 5.25 (m, 1H), 7.32 (d, J = 8.1 Hz, 2H), and 7.72 (d, J = 8.2 Hz, 2H). ¹³C NMR (125 MHz, CDCl₃), δ : 143.58, 135.31, 129.97, 127.70, 119.32, 117.84, 57.92, 55.37, 21.78, and 14.32. M.P. range = 100.8 – 101.8°C.

A mixture of 125 mg (0.5 mmol) the substrate **14**¹ and 40 mg (0.025 mmol) of catalyst **7** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h. As the reaction proceeded, the product **15**³ precipitated out of solution. After 1 h, the product was separated from the heptane solution of the catalysts by filtration. The heptane catalyst solution was directly recycled in subsequent reactions. The product was dried and characterized. ¹H NMR (500 MHz, CDCl₃), δ : 2.24 (m, 2H), 2.45 (s, 3H), 3.19 (t, $J = 5.61$ Hz, 2H), 3.59 (m, 2H), 5.63 (m, 1H), 5.77 (m, 1H), 7.34 (d, $J = 8.06$ Hz, 2H), and 7.70 (d, $J = 8.3$ Hz, 2H). ¹³C NMR (125 MHz, CDCl₃), δ : 143.73, 133.62, 129.86, 127.95, 125.31, 123.0, 45.02, 42.88, 25.51, and 21.77. M.P. range = 99.7 – 102.2°C.

Ring-opening metathesis polymerization. The catalyst (20mg, 0.012 mmol) was dissolved in 2 mL of THF. It was then added drop wise to a THF solution (5 mL) of the monomer (**18**: 60.9 mg, 0.37 mmol, 30 equiv., **19**: 60.5 mg, 0.37 mmol, 30 equiv.) and the reaction was stirred at room temperature. The color of the solution turned from green to light gray in less than 2 min. The solution was stirred for an additional 20 min, then ethyl vinyl ether (0.4mL, 600 equiv.) was added and stirring continued for 15 min. The polymerization solution

was then poured into methanol (100mL) while stirring to give a light greenish solid that was collected and dried under vacuum

Ruthenium Analysis Procedure. The sample that was to be analyzed (20-30 mg) was added to a 20 mL vial along with 4 g of concentrated nitric acid were added. The mixture was heated to 120 °C until everything dissolved. At this point 4 g of concentrated sulfuric acid was added to the solution. The solution was then allowed to stand at room temperature for 24 h. At this point, the concentrated acidic aqueous solution was transferred to a 50 mL plastic bottle and diluted to 50 mL with 1% nitric acid solution. A portion (0.2063 g) of the solution was further diluted to 50 mL with an additional portion of 1% nitric acid. Then, the diluted sample solution was analyzed by ICP-MS.

Copies of NMR and UV spectra of products and intermediates.

CH3-80
Pulse Sequence: s2pu1
Solvent: cdcl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 1.495 sec
Width 4500.5 Hz
16 repetitions
OBSERVE H1, 299.9141913 MHz
DATA PROCESSING
FT size 32768
Total time 5 min, 35 sec

CH3-80C
Pulse Sequence: s2pu1
Solvent: cdcl3
Ambient temperature
INNOVA-500 "innova500"
Pulse 60.0 degrees
Acq. time 1.300 sec
Width 25000.0 Hz
100 repetitions
OBSERVE C13, 125.7175387 MHz
DECOUPLE H1, 499.8725488 MHz
Power 42 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 38 min, 14 sec

CH3-81
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient Temperature
File: CH3-81
INNOVA-300 "Inova300"
Pulse 30.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
8 repetitions
OBSERVE H1, 299.9579345 MHz
DATA PROCESSING
F1 size 32768
Total time 6 min, 15 sec

PIB-formylpheno1

exp1 s2pul
SAMPLE DEC. & VT
date Mar 23 2007 dfrq 499.932
solvent cdcl3 da H1
file exp dprv 02
ACQUISITION dof 0
frrq 125.719 da ypy
tn C13 dam w
at 1.300 def 11710
np 78020 dseq
sw 29995.3 dres 1.0
fs 17000 hosc n
bs 16 temp 15.0
tpwr 53 PROCESSING
pw 5.9 lb 0.50
dl 0 wfile
tof 0 proc ft
nt 10000 fa not used
ct 132 math f
alock n
gain not used werr
flags n wexp
il n wbt
in n wnt
dp y
hs nm
DISPLAY
sp -3054.0
wp 29995.8
vs 207
sc 0
wc 250
h2am 0.17
is 500.00
rfl 3054.4
rfp 0
th 60
ins 100.000
nm cdc ph

CH3-89
Pulse Sequence: s2pul
Solvent: cdc13
Ambient temperature
INOVA-300 "Inova300"
Pulse 30.0 degrees
Acq. time 3.744 sec
Width 4000.0 Hz
9 repetitions
OBSERVE H1: 299.9579261 MHz
DATA PROCESSING
FT size 32768
Total time 6 min, 15 sec

PIB-formylphenylether
expt s2pul
SAMPLE
date Mar 23 2007 dfrq DEC. & VT 499.932
solvent cdc13 dn H1
r1s cdc13 dn H2
ACQUISITION
sfrq 125.719 dm vvy
tn C13 dm w
at 1.300 dmf 11710
np 78020 dseq
sw 29995.3 dres 1.0
fb 17000 hmo
bs 16 temp 15.0
tpwr 53 PROCESSING
pw 5.8 lb 0.50
d1 0 wfile
tof 0 proc ft
nt 10000 fa not used f
ct 420 math
a lock n
gain not used verr
FLAGS n wexp
in n wnt
dp y
hs nm
DISPLAY
sp -3054.0
vp 29995.6
vs 545
sc 0
wc 250
hzmm 119.95
is 500.00
rfi 3054.4
rfp 0
th 60
inc 100.000
nm cdc ph

CH4-38
Pulse Sequence: s2pu1
Solvent: cdc13
Ambient temperature
INDVA-500 "Inova500"
Pulse 30.0 degrees
Acq. time 2.852 sec
Width 8000.0 Hz
16 Repetitions
OBSERVE H1, 499.9518085 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 50 sec

PI8-isopropoxystyrene
exp1 s2pu1
SAMPLE DEC. & VT
date Mar 23 2007 dfr 499.932
solvent cdc13 dn H1
file exp dpwr 42
ACQUISITION exp dpwr 0
sfreq 125.718 dm vvv
tn C13 dnm v
at 1.300 def 11710
np 78020 dseq
pw 29985.3 dres 1.0
fb 17000 homo n
bs 16 temp 15.0
tpwr 5.5 lb PROCESSING 0.50
dl 0 wtfille
tof 0 proc ft
nt 10000 fn not used f
ct 244 math
alock n
gain not used werr
it FLAGS n wexp
in n wnt
dp v
hs nn
DISPLAY
sp -3054.0
wp 29985.8
vs 304
sc 0
wc 250
hzmm 119.99
ls 500.00
rfi 3054.4
rfp 0
th 66
ins 100.000
nm cdc ph

CH4-13
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
INOVA-500 "inova500"
Pulse 30.0 degrees
Acq. time 2.852 sec
Width 25000.0 Hz
23 Repetitions
OBSERVE H1, 499.9518085 MHz
DATA PROCESSING
FT Size 282144
Total time 4 min, 50 sec

P18-Hoveyda-Grubbs

exp1 s2pul
SAMPLE
date May 16 2007 dfrq DEC. & VT 499.932
solvent cdcl3 dn H1
file exp dpr 42
ACQUISITION
sfrq 125.719 da yvy
tn C13 dm v
at 1.300 def 11710
np 156096 dreq
sw 60015.0 dres 1.0
fu 33600 hmc
bs 64 PROCESSING n
tpwr 53 lb 0.50
pw 5.3 wfile
d1 0 proc ft
tof 0 fa not used f
nt 40000 math
ct 3852
a lock n werr
gain not used wexp
FLACS n wnt
ti n
in n
dp y
hs nm
DISPLAY
sp -2921.8
wp 42884.6
vs 706
vc 0
wc 250
hzmm 170.66
rs 500.00
rfl 18063.8
rff 0
th 68
lms 100.000
nm cdc ph

CH4-77second
Pulse Sequence: s2pul
Solvent: cdc13
Ambient temperature
INNOVA-500 "Innova500"
Pulse 30.0 degrees
Acq. time 2.892 sec
Width 8000.0 Hz
12 repetitions
OBSERVE W1, 499.9420431 MHz
DATA PROCESSING
F1 size 85538
Total time 4 min, 50 sec

9

RCM of diallylethylmalonate

expl s2pul

SAMPLE DEC. & VT
date Mar 19 2007 dfrq 499.932
solvent cdc13 dn H1
file exp dpwr 42
ACQUISITION dof 0
sfrq 125.719 ds VVV
tn C13 dm 11710
at 1.300 dnf
np 85024 dseq
pw 25000.0 dres 1.0
fb 14000 homo n
DS 16 temp 22.0
tsr SC PROCESSING
pw 5.9 lb 0.50
d1 0 wfile
tof 0 proc ft
nt 10000 fa not used
ct 181 math f
alock n
gain not used werr
FLAGS n wexp
il n wos
ln n wnt
np v
hs m
DISPLAY
sp -555.5
wp 24999.2
vs 50
sc 0
wc 250
hzmm 0.17
ls 500.00
rfi 556.3
rfp n
th 68
ins 100.000
nm cdc ph

9

CH4-7851xth
Pulse Sequence: s2pul
Solvent: cdc13
Ambient Temperature
File: CH4-7851xth
INOVA-500 "Inova500"
Pulse 30.0 degrees
Acq. time 2.852 sec
Width 8000.0 Hz
10 repetitions
OBSERVE H1, 499.9420433 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 50 sec

RCM of diallylTs

expt s2pul

SAMPLE DEC. & VT
date Mar 13 2007 dfrq 499.932
solvent cdc13 dn H1
file exp dpar 42
ACQUISITION dof 0
sfrq 125.719 dm YVY
tn C13 dnm w
at 1.300 dmf 11710
np 65024 dseq
sw 25000.0 dret 1.0
fb 14000 homo n
bs 16 temp 22.0
tpwr 53 PROCESING 0.50
pw 5.5 lb
d1 0 wfile
tof 0 proc ft
nt 100000 fn not used
ct 675 math f
alock n
gain not used werr
FLAGS n wbs
tn n wnt
dp y
hs mn
DISPLAY
sp -555.5
vp 24999.2
vs 100
sc 0
wc 250
hzmm 0.37
ls 500.00
rfl 556.3
rfp 0
th 80
lms 100.000
na cdc ph

CH5-19-4
Pulse Sequence: s2pul
Solvent: cdc13
Ambient Temperature
INNOVA-500 "Inova500"
Pulse 30.0 degrees
Acq. time 2.892 sec
Width 8000.0 Hz
50 repetitions
OBSERVE H1, 499.9296643 MHz
DATA PROCESSING
FT size 85536
Total time 48 min, 20 sec

CH5-19C13
expl s2pul
SAMPLE
date APT 4 2007 dfrq DEC. & VT 499.932
solvent cdc13 da H1
file /home/berghre dper 42
iter/chongfa/CH5-1- dof 0
ACQUISITION
sfrq 125.719 def 11710
in C13 dseq
at 1.300 dres 1.0
np 81022 homo n
sw 34895.6 PROCESSING
fb 18000 lb 0.50
bs 16 wfile
tpwr 53 proc ft
pw 5.8 fa not used f
d1 0 math
tof 0
nt 10000 werr
ct 405 wexp
a1ock n wds
gain not used wnt
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -5559.6
wp 34895.1
vs 100
vc 0
wc 250
hzmm 139.88
is 500.00
rfj 5554.1
rfd 0
th 68
ins cdc ph 100.000

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