# Polyisobutylene-Anchored *N*-Heterocyclic Carbene Ligands

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## Supporting Information

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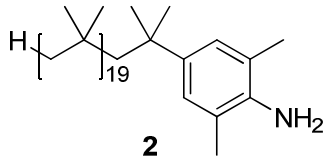
**General Experimental:** The $^{1}$H-NMR spectra were recorded on Inova 500 MHz and 300 MHz spectrometers operating at 499.95 MHz and 299.916 MHz respectively. $^{13}$C-NMR spectra were recorded on an Inova 500 MHz spectrometer operating at 125.719 MHz. Chemical shifts are reported in parts per million (δ) relative to residual proton resonances in deuterated chloroform ($CDCl_3$). Coupling constants (*J* values) are reported in hertz (Hz), and spin multiplicities are indicated by the following symbols: s (singlet), d (doublet), dd (doublet of doublet), t (triplet), q (quartet), b (broad), and m (multiplet). UV-Vis spectra were obtained using an Agilent 8453 UV-Visible spectrometer. ICP-MS data were obtained using a Perkin Elmer DRC II instrument.

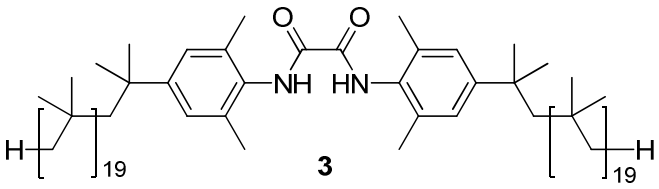
All reactions were carried out under an inert atmosphere unless otherwise noted. Acetonitrile, heptane, dimethylformamide, ethanol, dichloromethane, tetrahydrofuran, and toluene were purchased from EMD and used as received. Polyisobutylene was a gift from BASF. All other chemicals were purchased from Sigma-Aldrich and used as received.

**Synthesis of PIB Supported NHC, Grubbs-Hoveyda 2nd Generation Catalyst, and Ag(I) Complexes**




**2**

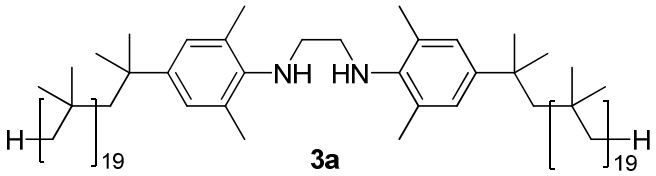
**2,6-Dimethyl-4-(polyisobutyl)aniline (2):** A mixture of 12.4 g (102 mmol) of 2,6-dimethylaniline, 10.15 g (10.15 mmol) of polyisobutylene (Glissopal 1000), and 4.4 g (33 mmol) of aluminum trichloride was stirred for 3 d at 200 °C in a pressure vessel. After 3 d, the deep purple solution reaction was cooled to approximately 100 °C and added to 200 mL of hexane. The solution so formed was washed with 150 mL of dimethylformamide three times and then with 150 mL of 90% ethanol/water three times. After drying over sodium sulfate, the solvent was removed under reduced pressure and the product was purified by column chromatography (eluting first with hexane and then with dichloromethane). Solvent removal afforded the product as a light yellow viscous oil. The yield was 65% (7.39 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 140H), 1.8 (s, 2H), 2.19 (s, 6H), 3.45 (s, 2H), and 6.92 (s, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 18.26, multiple peaks between 30-40 and 58-60, 121.28, 126.30, 140.06, and 140.27.




**3**

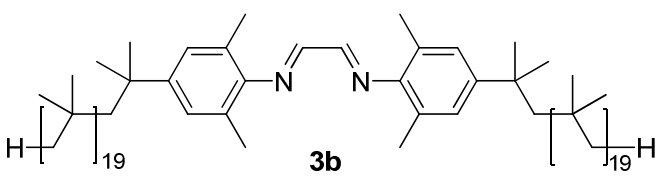
***N,N'*-Bis(2,6-dimethyl-4-(polyisobutyl)phenyl)oxalamide (3):** A mixture of 6 g (5.35 mmol) of 2,6-dimethyl-4-(polyisobutyl)aniline and 0.64 g (6.3 mmol) of triethylamine in 30 mL of dichloromethane was cooled to 0 °C. A solution of oxalyl chloride (0.4 g, 3.15 mmol) in 5 mL of dichloromethane was added slowly to the reaction. The reaction was then stirred overnight. The solvent was removed under reduced pressure and added to 150 mL of hexanes. The hexanes

solution was washed with 100 mL of 90% ethanol/water three times. The hexane phase was dried over sodium sulfate and shaken with 6 g of acid Amberlyst XN-1010 for 4 h, and then the resin was removed by filtration. The solvent was removed under reduced pressure to yield a light yellow viscous oil. The yield was 88% (5.40 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 280H), 1.82 (s, 2H), 2.28 (s, 12H), 7.11 (s, 2H), and 8.82 (s, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 18.26, multiple peaks between 30-40 and 58-60, 126.20, 129.30, 134.05, 150.15, and 158.25.



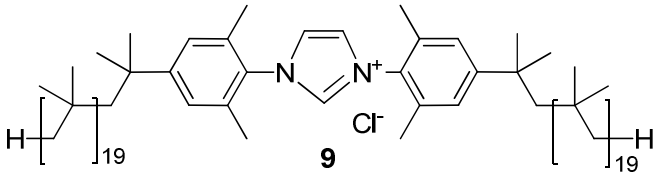

**3a**

***N*,*N*'-Bis(2,6-dimethyl-4-(polyisobutyl)phenyl)ethane-1,2-diamine (3a):** A 6.34 g (2.76 mmol) sample of *N*,*N*'-bis(2,6-dimethyl-4-(polyisobutyl)phenyl)oxalamide was dissolved in 30 mL of toluene, then 1.83 mL (18.67 mmol) of $BH_3$-$SMe_2$ was added to the solution. The solution turned from yellow to almost colorless. The reaction was heated at 90 $^{o}$C overnight. The solvent was removed under reduced pressure and purified by column chromatography (10:1 mixture of hexane and dichloromethane). Solvent removal afforded a light yellow viscous oil. The yield was 61% (3.82 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 280H), 1.77 (s, 2H), 2.30 (s, 12H), 3.17 (s, 4H), and 6.97 (s, 4H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 18.26, multiple peaks between 30-40 and 58-60, 49.30, 126.96, 128.92, 143.23, and 144.18.



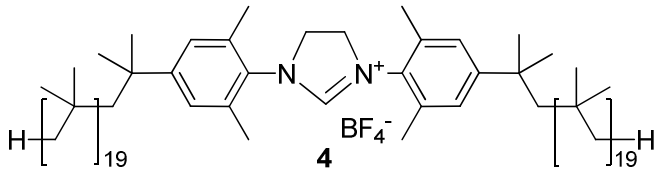

**3b**

***N,N′*-(Ethane-1,2-diylidene)bis(2,6-dimethyl-4-(polyisobutyl)aniline) (3b):** A mixture of 3.85 g (3.43 mmol) of 2,6-dimethyl-4-(polyisobutyl)aniline, and a catalytic amount of formic acid in 13 mL of hexane was prepared. A solution of 0.25 g (1.72 mmol) of glyoxal (40% in water) in 4 mL of isopropanol was then added to this solution. The reaction mixture initially turned cloudy for roughly 5 m and then became a clear yellow solution. The reaction was allowed to stir overnight. The solution was dried with sodium sulfate and the solvent was removed under reduced pressure to yield a dark yellow/brownish viscous oil. The yield was 94% (3.65 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 280H), 1.77 (s, 2H), 2.20 (s, 12H), 7.06 (s, 4H), and 8.12 (s, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 18.90, multiple peaks between 30-40 and 58-60, 126.06, 126.46, 128.50, 147.10, and 163.68.



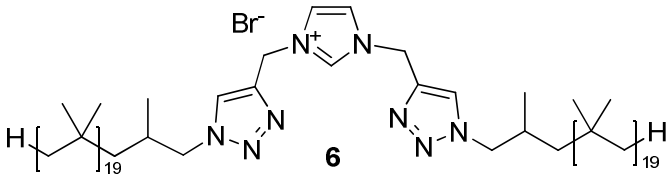

**9**

**1,3-Bis(2,6-dimethyl-4-(polyisobutyl)phenyl)imidazolium chloride (9):** A 0.55 g (0.24 mmol) sample of *N,N′*-(ethane-1,2-diylidene)bis(2,6-dimethyl-4-(polyisobutyl)aniline) was dissolved in 2 mL of THF and 23 mg (0.24 mmol) of chloromethyl ethyl ether was added and heated to 40 $^{0}$C overnight. The solvent was removed under reduced pressure and the residue was purified by column chromatography (dichloromethane). Solvent removal afforded a light brown viscous oil. The yield was 65% (0.36 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 280H), 1.82 (s, 2H), 2.20 (s, 12H), 7.19 (s, 4H), 7.79 (s, 2H), and 10.21 (s, 1H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 18.90, multiple peaks between 30-40 and 58-60, 125.36, 127.44, 130.51, 133.57, 139.20, and 154.63.




4

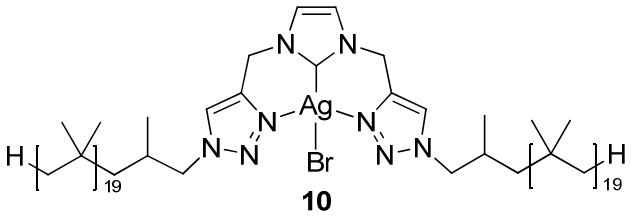
**1,3-Bis(2,6-dimethyl-4-(polyisobutyl)phenyl)-4,5-dihydroimidazolium tetrafluoroborate (4):** A 3.62 g (1.6 mmol) sample of *N*,*N'*-bis(2,6-dimethyl-4-(polyisobutyl)phenyl)ethane-1,2-diamine was dissolved in 10 mL of triethyl orthoformate and followed by the addition of 230 mg (2.19 mmol) of ammonium tetrafluoroborate and heated to 110 $^{o}$C overnight. The solvent was removed under reduced pressure and purified by column chromatography (9:1 dichloromethane:methanol) to yield a dark yellow viscous oil. The yield was 79% (2.99 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 280H), 1.85 (s, 2H), 2.42 (s, 12H), 4.65 (s, 4H), 7.17 (s, 4H), and 7.65 (s, 1H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 18.36, multiple peaks between 30-40 and 58-60, 52.37, 127.60, 129.84, 134.54, 154.11, and 158.25.




6

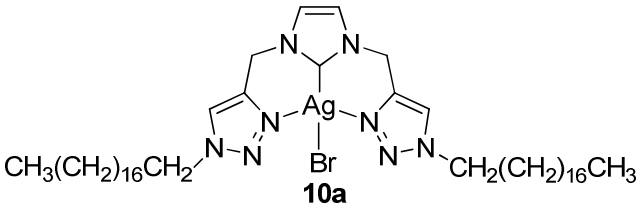
**1,3-Bis-((1-polyisobutyl-1*H*-1,2,3-triazol-4-yl)methyl)imidazolium bromide (6):** A 0.606 g (2.69 mmol) sample of 1,3-di-(prop-2-ynyl)imidazolium bromide, 17 mg (0.17 mmol) of CuCl, and 6.17 g (5.92 mmol) of azide-terminated polyisobutylene[1] were dissolved in 105 mL of dichloromethane and 20 mL of methanol. The solution was stirred at room temperature for 24 h. Then, 1.19 g of EDTA and 20 mL of water was added and the resulting biphasic mixture was stirred at room temperature for 24 h. The dichloromethane layer was separated, the solvent was removed under reduced pressure, and the residue was dissolved in 100 mL of hexane. The hexane solution of the product was then washed with methanol until the methanol layer became

colorless. The hexane was removed under reduced pressure and the crude product was purified by column chromatography (eluting first with dichloromethane and then with a 9:1 mixture of dichloromethane and methanol). The final product was isolated as an orange viscous oil. The yield was 82% (5.12 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.6-1.7 (m, 280H), 2.13 (m, 2H), 4.031 (dd, *J* = 6, 13.5 Hz, 2H), 4.22 (dd, *J* = 7.4, 13.5 Hz, 2H), 5.61 (s, 4H), 7.44 (s, 2H), 8.21 (s, 2H), and 10.85 (b, 1H) $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 20.55, multiple peaks between 28-39 and 57-60, 123.07, 125.59, 136.64, and 139.64.



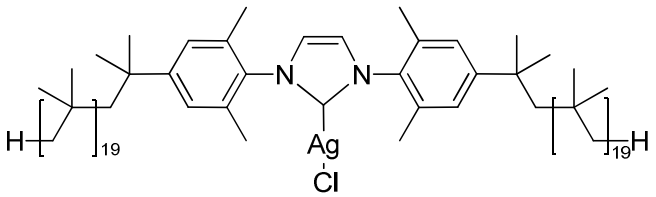

**10**

**1,3-Bis-((1-polyisobutyl-1*H*-1,2,3-triazol-4-yl)methyl)imidazol-2-ylidene-silver(I) bromide (10):** A 0.32 g (0.138 mmol) sample of 1,3-bis-((1-polyisobutyl-1*H*-1,2,3-triazol-4-yl)methyl)imidazolium bromide and 17.4 mg (0.075 mmol) of $Ag_2O$ were dissolved in 3 mL of dichloromethane. The suspension was stirred at 49 $^{o}$C for 24 h. At this point, the suspension was filtered through celite to remove excess silver oxide and the silver oxide was washed by dichloromethane. The dichloromethane solution of the product was then dried with sodium sulfate and solvents were removed under reduced pressure to yield an orange viscous oil. The yield was 93 % (0.313 g). $^{1}$H-NMR (300 MHz, $CDCl_3$), δ: 0.6-1.8 (m, 280H), 2.10 (m, 2H), 4.02 (dd, *J* = 6, 13.5 Hz, 2H), 4.21 (dd, *J* = 7.4, 13.5 Hz, 2H), 5.30 (s, 4H), 7.22 (s, 2H), and 7.68 (s, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 20.68, multiple peaks between 28-39 and 57-60, 121.55, 123.90, 142.14, and 181.16.




**10a**

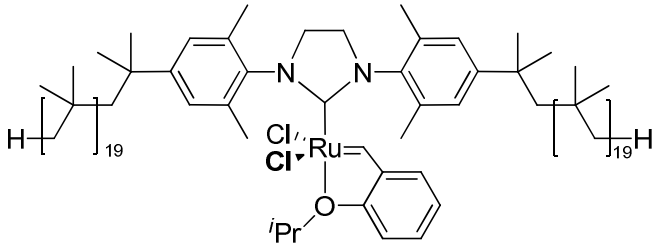
**1,3-Bis-((1-octadecyl-1*H*-1,2,3-triazol-4-yl)methyl)imidazol-2-ylidene-silver(I) bromide (10a):** 158.4 mg (0.194 mmol) of 1,3-bis-((1-octadecyl-1*H*-1,2,3-triazol-4-yl)ethyl)imidazolium bromide and 33.8 mg (0.146 mmol) of $Ag_2O$ was added to 10 mL of dichloromethane. The resulting suspension was stirred at 49 $^oC$ for 24 h. This suspensionwas then filtered through celite to remove excess silver oxide and the solids were washed by dichloromethane. The dichloromethane solution was dried with sodium sulfate and the solvent was removed under reduced pressure to yield an orange viscous oil. The yield was 94 % (0.1688 g). $^1$H-NMR (300 MHz, $CDCl_3$), δ: 0.85 (t, *J* = 6.6 Hz, 2H), 1.11- 1.35 (m, 60H), 1.866 (m, 4H), 4.299 (t, *J* = 7.5 Hz, 4H), 5.365 (s, 4H), 7.216 (s, 2H), 7.794 (s, 2H). $^{13}$C-NMR ($CDCl_3$), δ: 14.10, 22.66, 26.48, multiple peaks between 28-31, 31.89, 46.69, 50.55, 121.58, 123.40, 142.43, 181.67. HRMS (ESI): Calc. for $[M - Br]^+$ ($^{107}$Ag isotope): 841.5713. Found: 841.6153.




**11**

**1,3-Bis (2,6-dimethyl-4-(polyisobutyl)phenyl)imidazol-2-ylidene)silver(I) chloride (11):** A 0.4268 g (0.185 mmol) sample of 1,3-bis(2,6-dimethyl-4-(polyisobutyl)phenyl)imidazolium chloride and 30.8 mg (0.133 mmol) of $Ag_2O$ were added to 4 mL of dichloromethane. The reaction was refluxed for 16 h. After the reaction was cooled to room temperature, the suspension was filtered through celite to remove excess $Ag_2O$. In some cases, the suspension

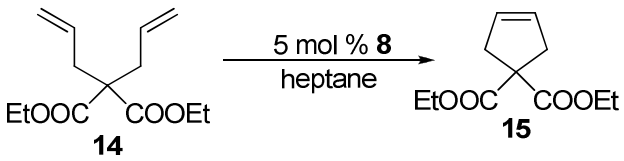
was centrifuged at 5 $^{o}$C to separate the finer silver salt, if this filtration was not successful. The solvent was removed under reduced pressure to yield the product as an orange viscous oil. The yield was quantitative (0.4667 g). $^{1}$H-NMR (300 MHz, $CDCl_3$), δ: 0.7-1.9 (m, 280H), 2.09 (s, 12H), 7.09 (s, 2H), and 7.15 (s, 4H). $^{13}$C NMR (125 MHz, $CDCl_3$), δ: 18.36, multiple peaks between 30-40 and 57-61, 122.68, 126.71, 133.76, 134.92, 152.39, and 182.875 (dd, $J(^{13}C\text{-}^{107}Ag)$ = 236.6 Hz, $J(^{13}C\text{-}^{107}Ag)$ = 271.6 Hz).



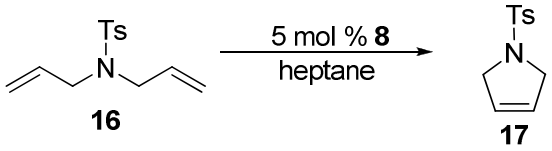

**8**

**PIB supported Grubbs-Hoveyda 2$^{nd}$ generation catalyst (8):** A mixture of 1.14 g (0.48 mmol) of 1,3-bis(2,6-dimethyl-4-(polyisobutyl)phenyl)-4,5-dihydro-imidazolium tetrafluoroborate, 0.15 g (0.75 mmol) of KHMDS, 0.05 g (0.5 mmol) of CuCl and 0.36 g (0.57 mmol) of 1$^{st}$ generation Hoveyda-Grubbs catalyst was prepared was dissolved in 5 mL of toluene. The solution was heated to 100 $^{o}$C for 3 h. The solvent was removed under reduced pressure and the crude product was purified by column chromatography (dichloromethane). After solvent removal, the product was isolated as a dark green viscous oil. The yield was 60% (0.75 g). $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 0.8-1.6 (m, 280H), 1.87 (s, 2H), 2.41 (b, 6H), 2.62 (b, 6H) 4.15 (s, 4H), 4.90 (m, 1H) 6.8 (m, 2H), 6.98 (m, 1H), 7.22 (b, 4H), 7.47 (m, 1H), and 16.67 (s, 1H). $^{13}$C NMR (125 MHz, $CDCl_3$), δ: 21.62, multiple peaks between 30-40 and 58-60, 113.10, 122.42, 123.09, 126.53, 127.01, 129.69, 137.30, 139.21, 145.41, 145.44, 152.16, 152.43, 152.45, 211.19, and 297.23 (m). $\lambda_{max}$ = 340 nm and 580 nm in heptane.

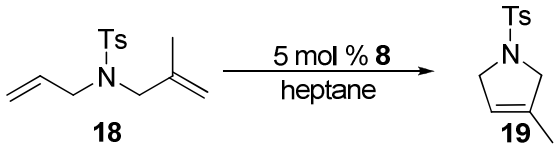
**Procedures for Ring Closing Metathesis Reaction**



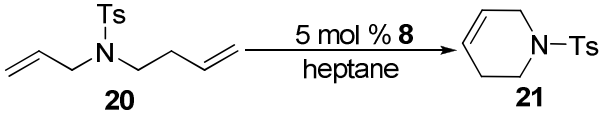

A mixture of 120 mg (0.5 mmol) of substrate **14** and 65 mg (0.025 mmol) of catalyst **8** were dissolved in 5 mL of heptane. After 1 h, the reaction was complete. At this point, 3 mL of acetonitrile was added to the reaction mixture. After vigorous stirring, the mixture was allowed to settle and the two phases were separated. The acetonitrile layer containing the product was dried under reduced pressure to yield compound **15**[2]. $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 1.24 (t, *J* = 7.33 Hz, 6H), 3.0 (s, 4H), 4.19 (q, *J* = 7.33 Hz, 4H), and 5.60 (m, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 172.49, 128.05, 61.76, 59.07, 41.08, and 14.27.




A mixture of 125 mg (0.5 mmol) of substrate **16** and 65 mg (0.025 mmol) of catalyst **8** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h during which time the substrate **17**[2] precipitated from solution. The catalyst solution was separated from the precipitated product by filtration for reuse in a subsequent reaction cycle. The isolated solid product was dried under reduced pressure. $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 2.44 (s, 3H), 4.13 (s, 4H), 5.66 (s, 2H), 7.33 (d, *J* = 8.3 Hz, 2H), and 7.73 (d, *J* = 8.3 Hz, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 143.68, 134.53, 130.0, 127.67, 125.70, 55.08, and 21.78. mp = 123.2-126.5$^{o}$C.

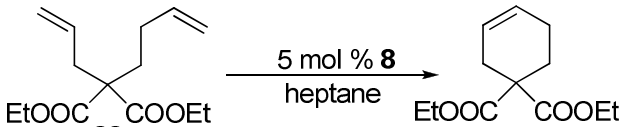


A mixture of 132 mg (0.5 mmol) of substrate **18** and 65 mg (0.025 mmol) of catalyst **8** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h during which time the substrate **19**[2] precipitated from solution. The catalyst solution was separated from the precipitated product by filtration for reuse in a subsequent reaction cycle. The isolated solid product was dried under reduced pressure. $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 1.66 (s, 3H), 2.43 (s, 3H), 3.97 (m, 2H), 4.07 (m, 2H), 5.25 (m, 1H), 7.32 (d, $J$ = 8.2 Hz, 2H), and 7.72 (d, $J$ = 8.2 Hz, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 143.58, 135.31, 129.97, 127.70, 119.32, 117.84, 57.92, 55.37, 21.78, and 14.32. mp = 100.8-101.8°C.



**20** **21**

A mixture of 132 mg (0.5 mmol) of substrate **20** and 65 mg (0.025 mmol) of catalyst **8** were dissolved in 5 mL of heptane. The reaction was stirred for 1 h during which time the substrate **21**[3] precipitated from solution. The catalyst solution was separated from the precipitated product by filtration for reuse in a subsequent reaction cycle. The isolated solid product was dried under reduced pressure. $^{1}$H-NMR (500 MHz, $CDCl_3$), 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, $J$ = 8.3 Hz, 2H), and 7.70 (d, $J$ = 8.3 Hz, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 143.73, 133.62, 129.86, 127.95, 125.31, 123.0, 45.02, 42.88, 25.51, and 21.77. mp = 99.7-102.2°C.



**22** **23**

A mixture of 127 mg (0.5 mmol) of substrate **22** and 65 mg (0.025 mmol) of catalyst **8** were dissolved in 5 mL of heptane. After 1h, the reaction was complete. At this point, 3 mL of acetonitrile was added to the reaction mixture. After vigorous stirring, the mixture was allowed

to settle and the two phases were separated. The acetonitrile layer containing the product was dried under reduced pressure to yield compound **23**[4]. $^{1}$H-NMR (500 MHz, $CDCl_3$), δ: 1.25 (m, 6H), 2.12 (m, 4H), 2.56 (s, 2H), 4.19 (m, 4H), and 5.68 (s, 2H). $^{13}$C-NMR (125 MHz, $CDCl_3$), δ: 171.84, 126.31, 124.24, , 61.49, 53.16, 30.64, 27.57, 22.54, and 14.27.

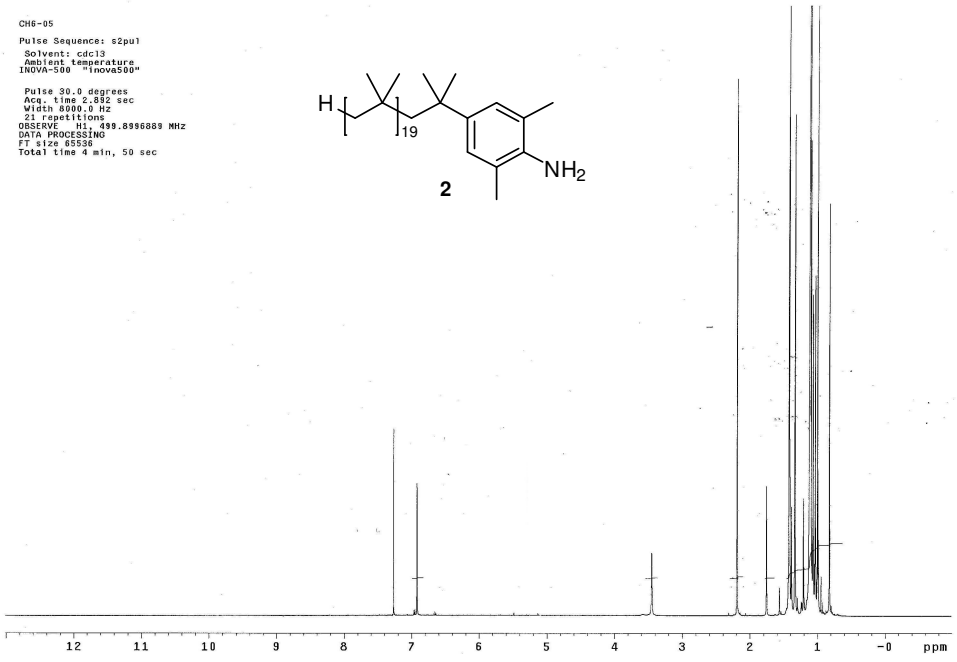
**Phase Selectivity Studies Procedure**

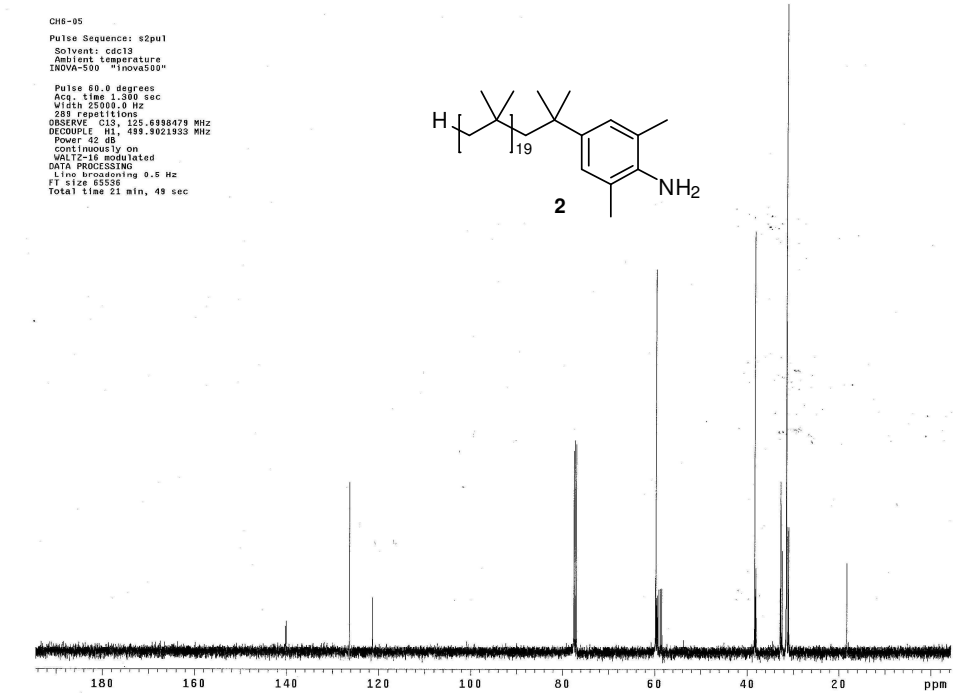
The sample that was to be analyzed (0.12 mg) was dissolved in 12.0 mL of heptane. Then 2 mL of this heptane solution was added to 2 mL of polar solvent (acetonitrile, ethylene glycol diacetate, di(ethylene glycol) monomethyl ether, or heptane-saturated DMF). The mixture was sealed and heated to 120 $^{o}$C to generate a homogeneous solution (in the case of the heptane-acetonitrile system only partial miscibility occurred). The solution was cooled to room temperature and centrifuged for 1 h at 5 $^{o}$C to produce a biphasic solution. Part of each phase was then analyzed by UV-Vis spectroscopy. Another portion of each phase was used as a sample for metal analysis.

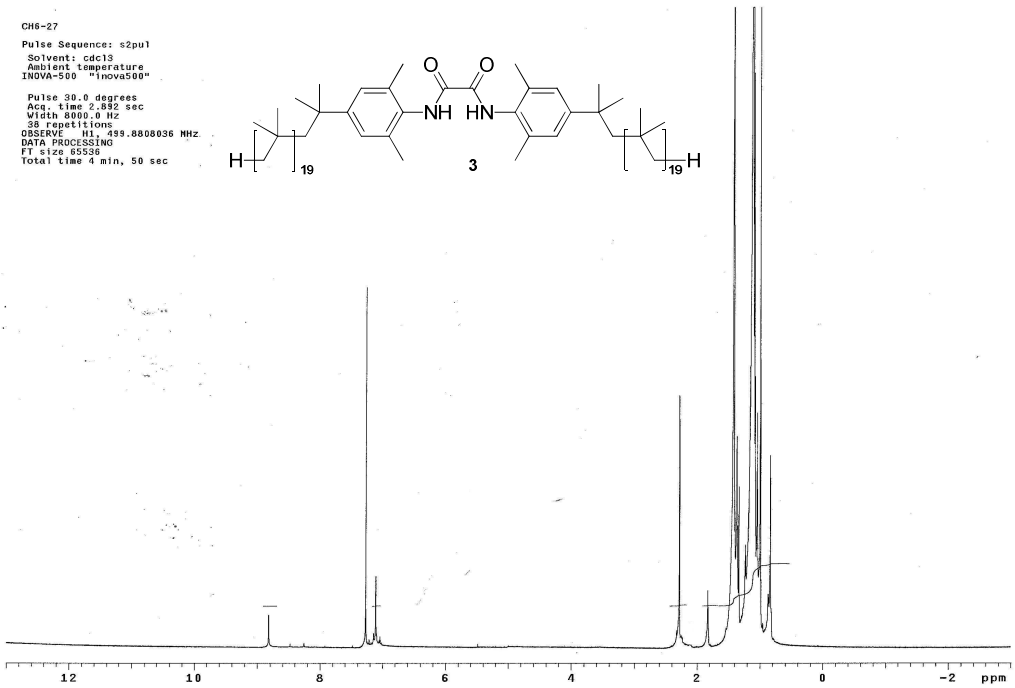
**ICP-MS Digestion Procedure**

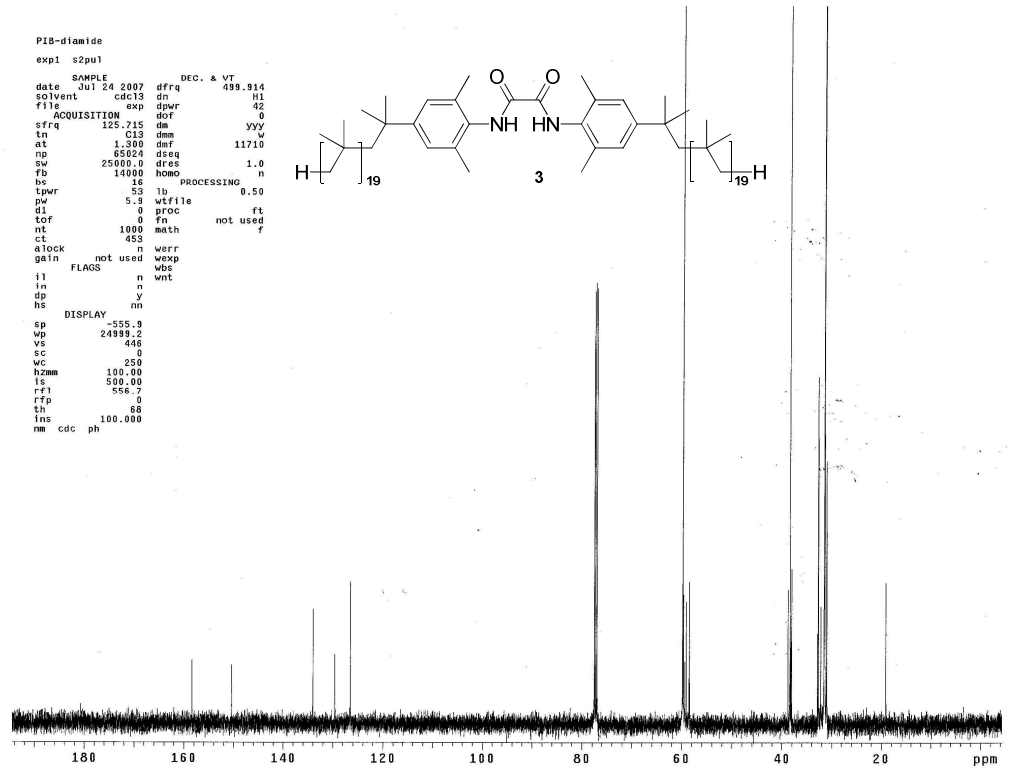
The sample that was to be analyzed (3-25000 μg) and 4 g of concentrated nitric acid were added to a glass vial. The mixture was heated at 120 $^{o}$C for 3 days. At this point, 4 g of concentrated sulfuric acid was added at room temperature and further heating at 120 $^{o}$C was continued for 2 more days or until all the sample had dissolved. The solution was then allowed to stand at room temperature. At this point, the concentrated acidic solution was diluted with 1% nitric acid solution and the diluted sample solution was analyzed by ICP-MS.

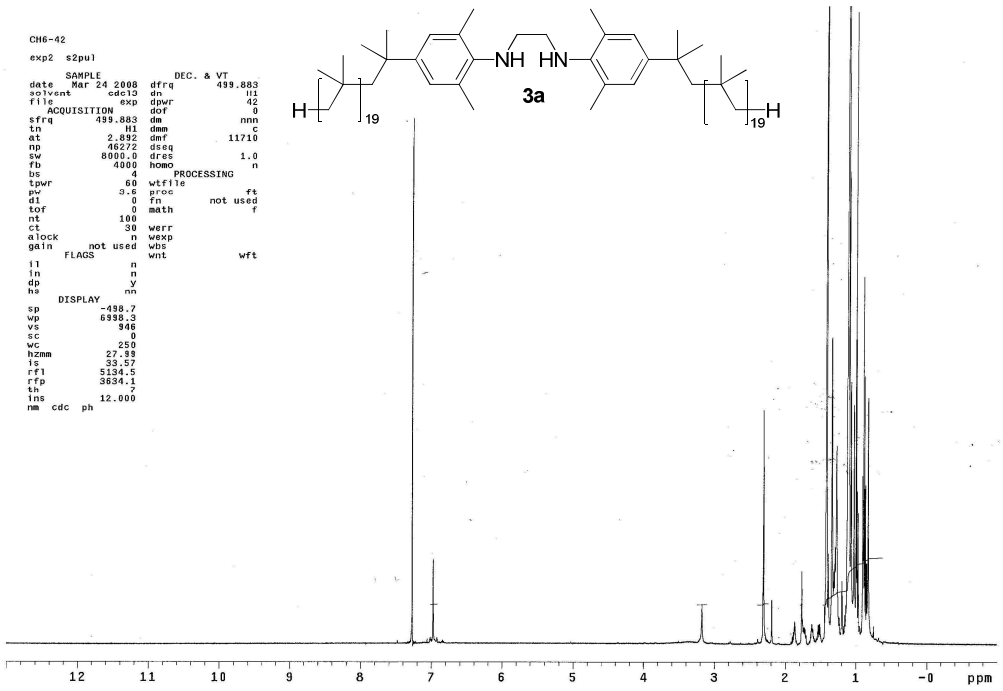
**Copies of NMR spectra of intermediates and products**.

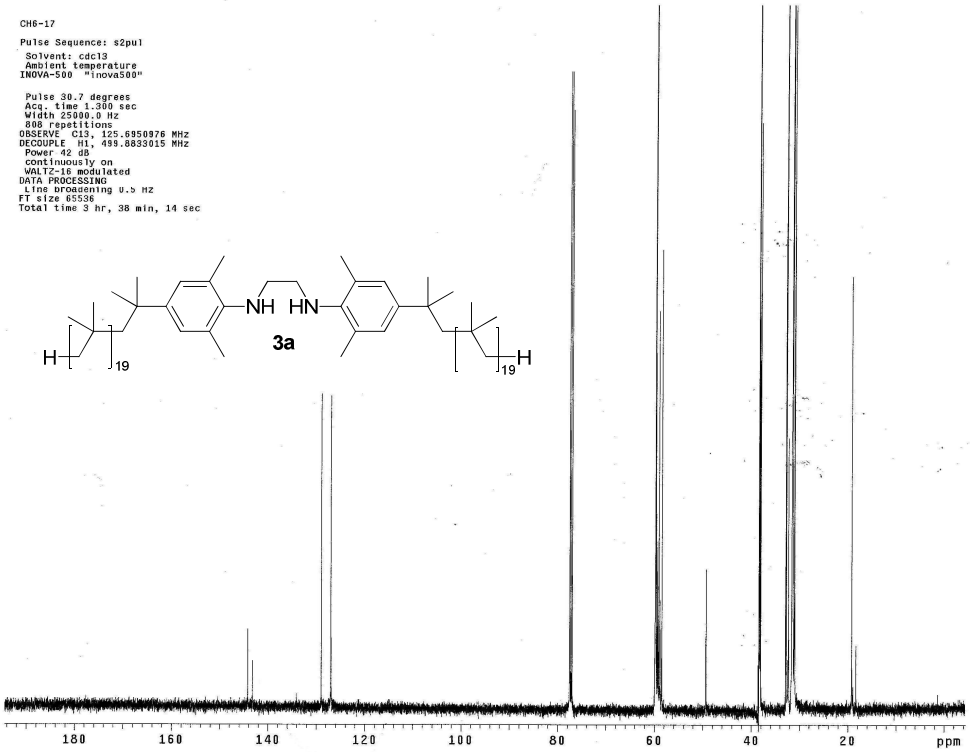


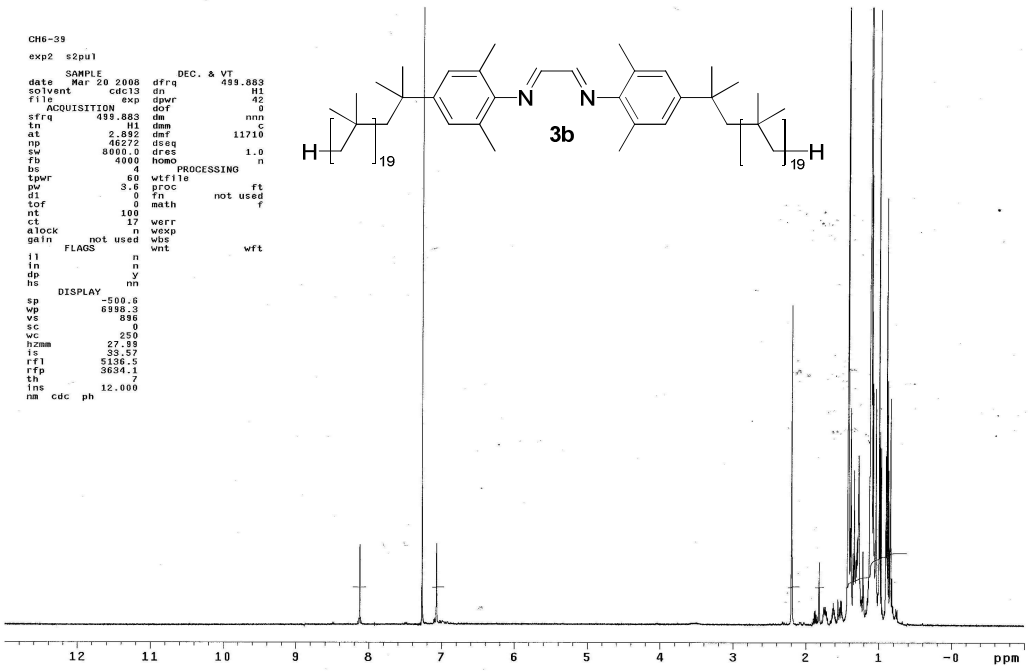



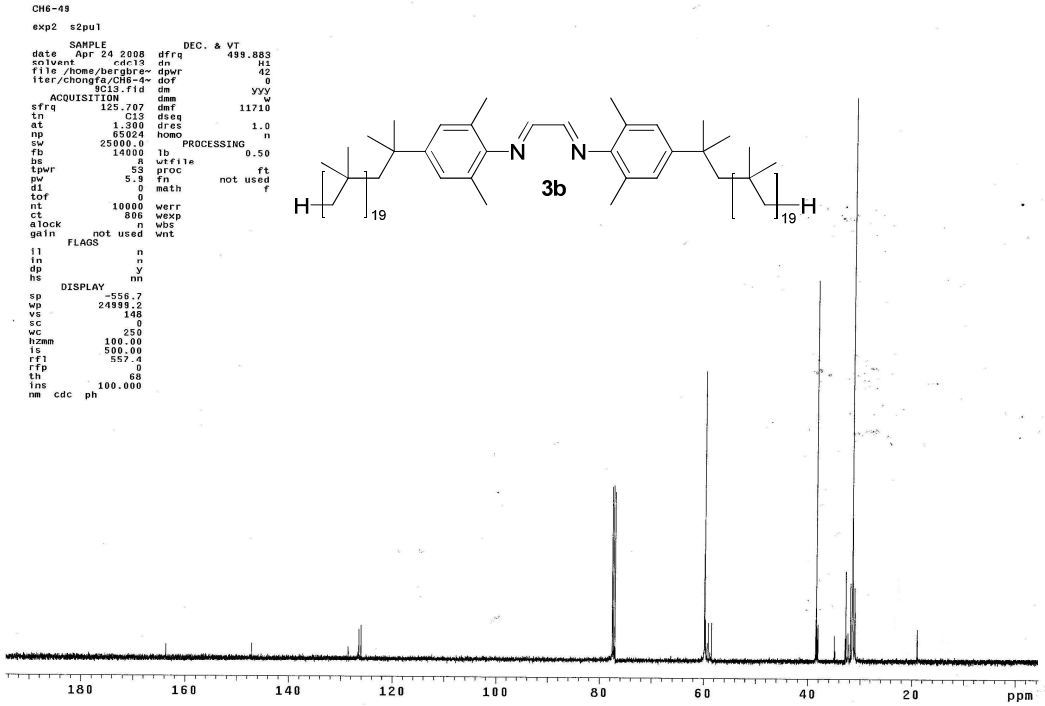

CH6-27
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
INOVA-500 "inova500"
Pulse 30.0 degrees
Acq. time 2.892 sec
Width 8000.0 Hz
38 repetitions
OBSERVE H1, 499.8808036 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 50 sec
O
O
NH HN
H
19
3
19
H
12
10
8
6
4
2
0
-2
ppm

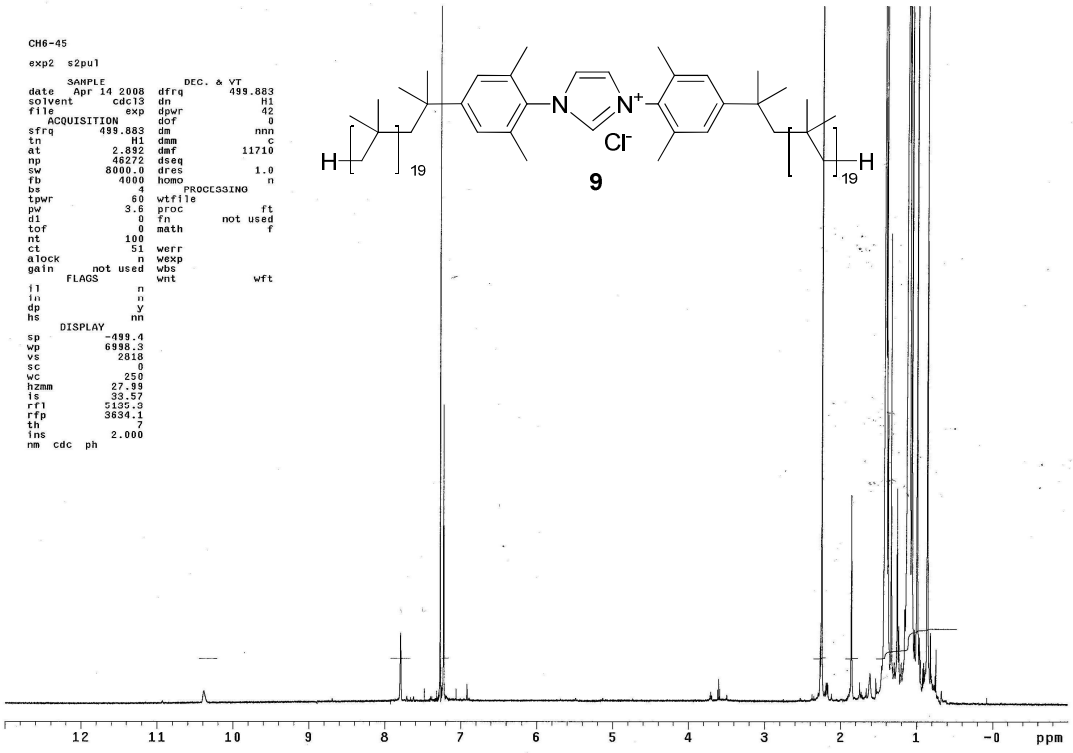

PIB-diamide
exp1 s2pul
SAMPLE
date Jul 24 2007
solvent cdcl3
file exp
ACQUISITION
sfrq 125.715
tn C13
at 1.300
np 65024
sw 25000.0
fb 14000
bs 16
tpwr 53
pw 5.9
d1 0
tof 0
nt 1000
ct 453
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -555.9
wp 24999.2
vs 446
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 556.7
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.914
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
O
O
NH HN
H
19
3
19
H
180
160
140
120
100
80
60
40
20
ppm

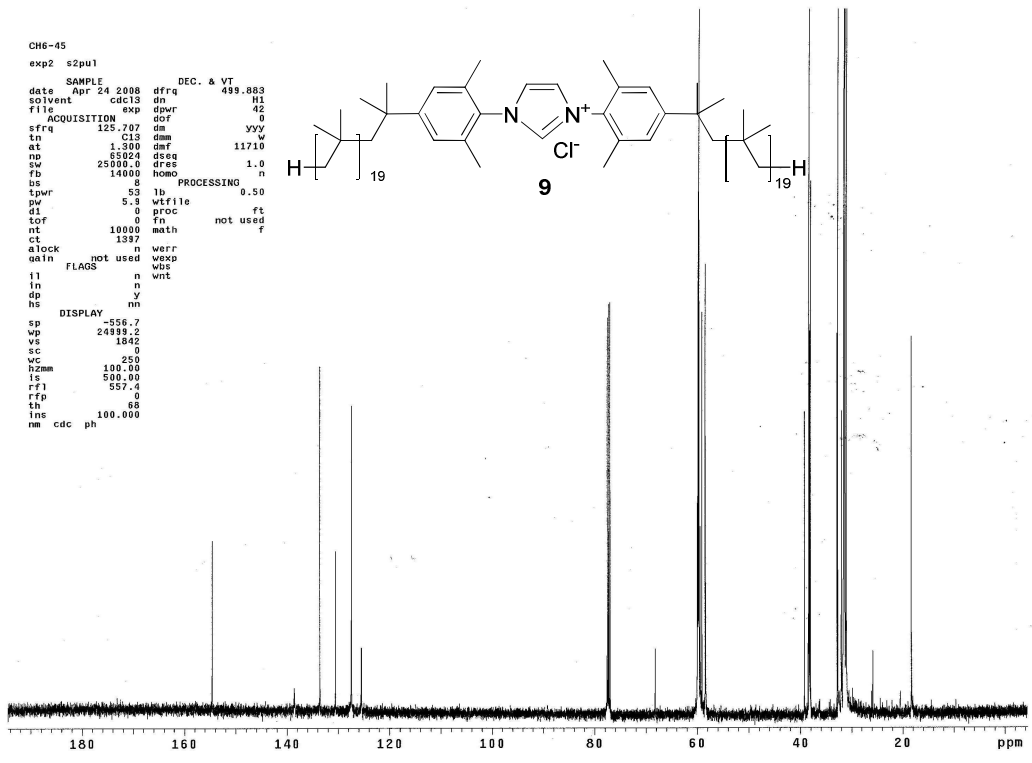

CH6-42
exp2 s2pul
SAMPLE
date Mar 24 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 499.883
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.6
d1 0
tof 0
nt 100
ct 30
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -498.7
wp 6938.3
vs 946
sc 0
wc 250
hzmm 27.98
is 33.57
rfl 5134.5
rfp 3634.1
th 7
ins 12.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
NH HN
3a
H
19
19
H
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

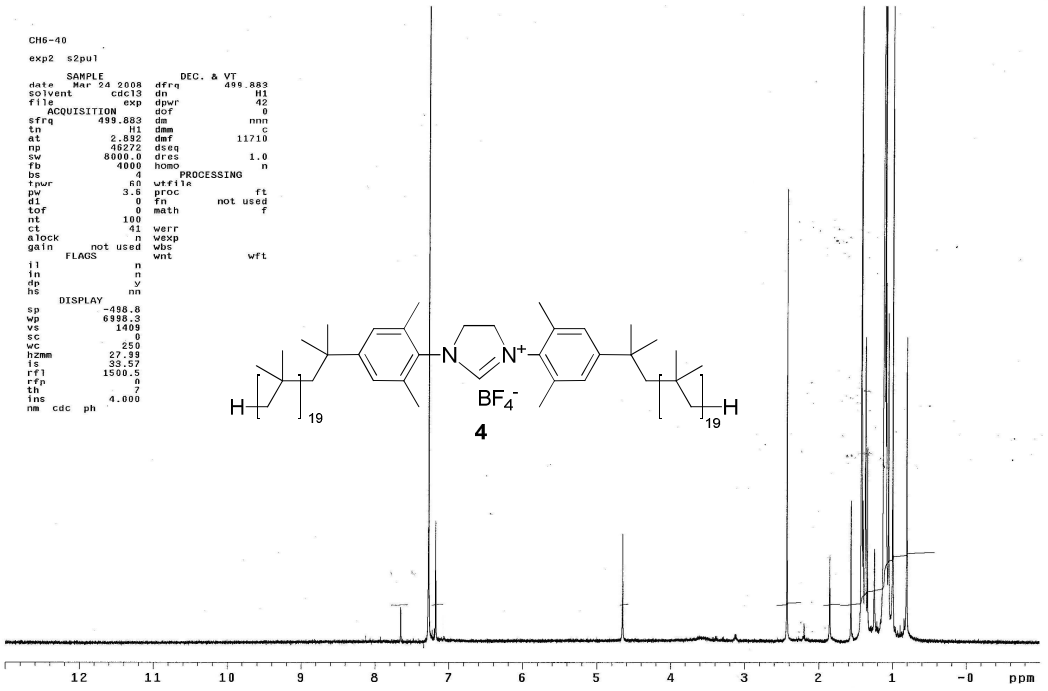

CH6-17
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
INOVA-500 "inova500"
Pulse 30.7 degrees
Acq. time 1.300 sec
Width 25000.0 Hz
808 repetitions
OBSERVE C13, 125.6950976 MHz
DECOUPLE H1, 499.8833015 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 3 hr, 38 min, 14 sec
NH HN
3a
H
19
19
H
180
160
140
120
100
80
60
40
20
ppm

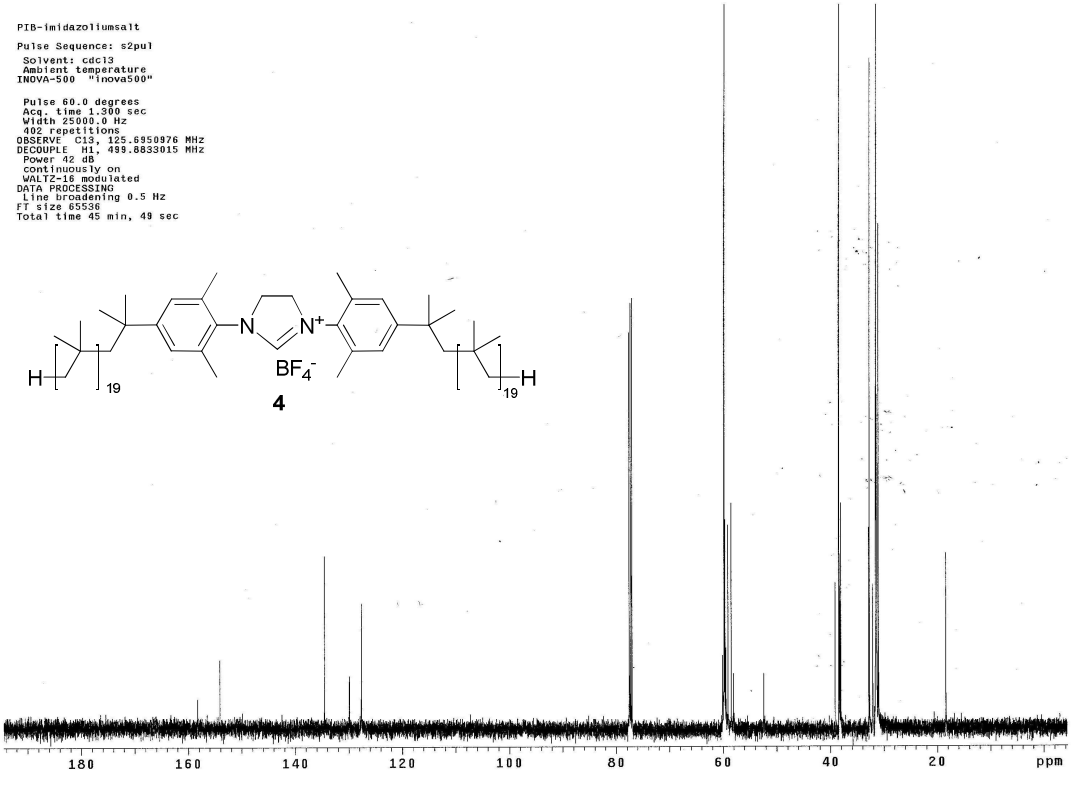

CH6-39
exp2 s2pul
SAMPLE
date Mar 20 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 499.883
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.6
d1 0
tof 0
nt 100
ct 17
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -500.6
wp 6998.3
vs 896
sc 0
wc 250
hzmm 27.88
is 39.57
rfl 5136.5
rfp 3634.1
th 7
ins 12.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
3b
H
19
N
N
19
H
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

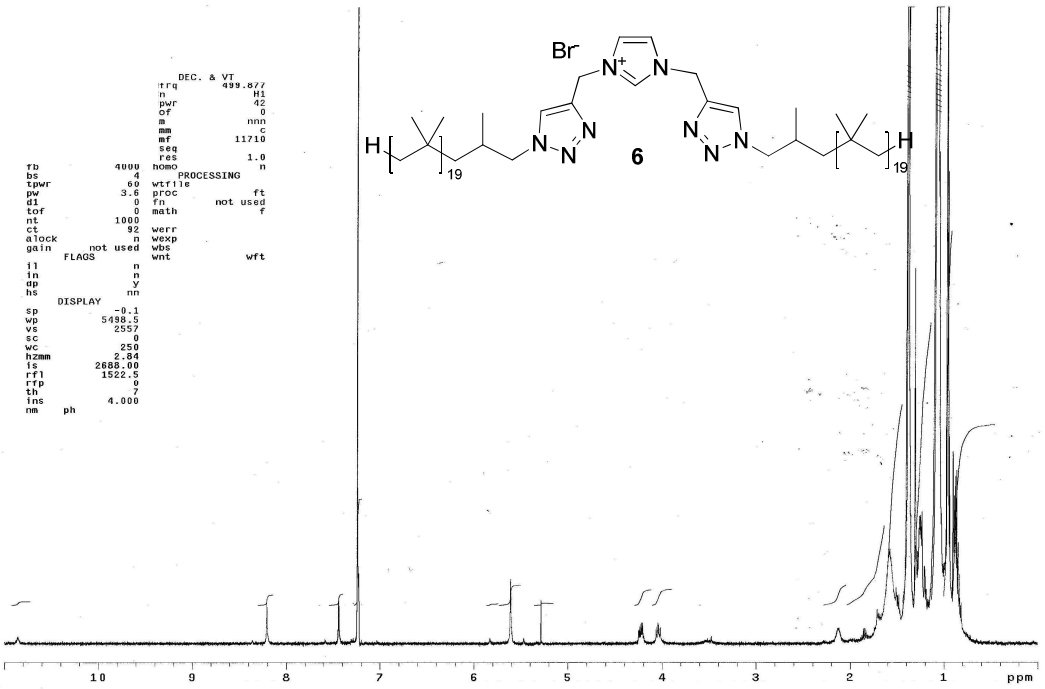

CH6-49
exp2 s2pul
SAMPLE
date Apr 24 2008
solvent cdcl3
file /home/bergbre~
ter/chongfa/CH6-4~
9C13.fid
ACQUISITION
sfrq 125.707
tn C13
at 1.300
np 65024
sw 25000.0
fb 14000
bs 8
tpwr 53
pw 5.9
d1 0
tof 0
nt 10000
ct 806
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -556.7
wp 24999.2
vs 148
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 557.4
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
3b
H
19
N
N
19
H
180
160
140
120
100
80
60
40
20
ppm

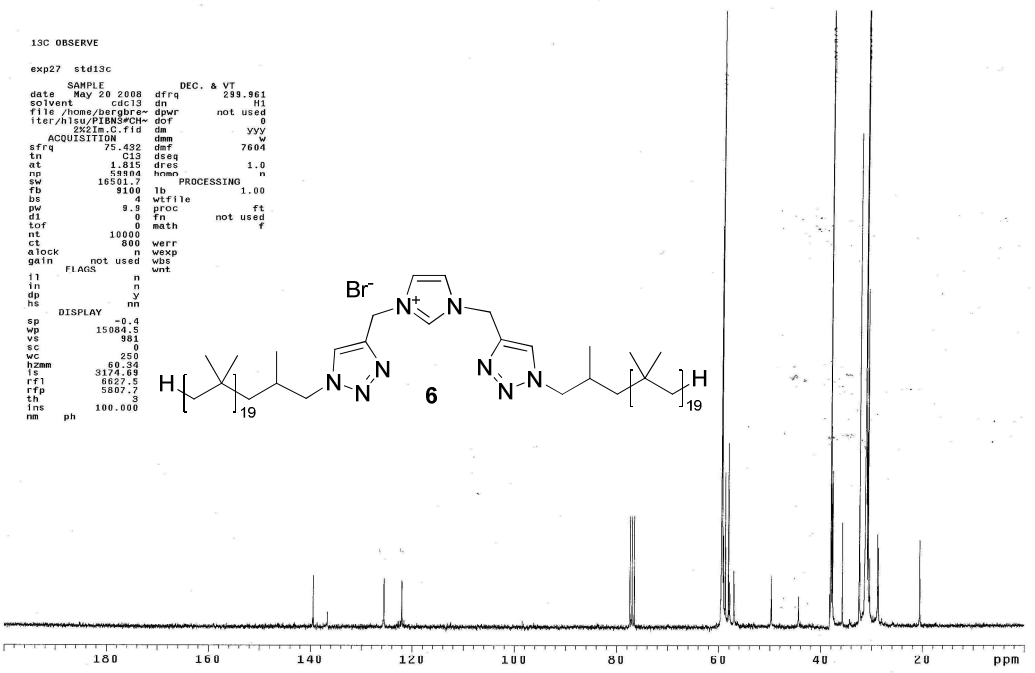

CH6-45
exp2 s2pul
SAMPLE
date Apr 14 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 499.883
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.6
d1 0
tof 0
nt 100
ct 51
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -499.4
wp 6998.3
vs 2818
sc 0
wc 250
hzmm 27.99
is 33.57
rfl 5135.3
rfp 3634.1
th 7
ins 2.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
H
19
N
N+
Cl-
9
19
H
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

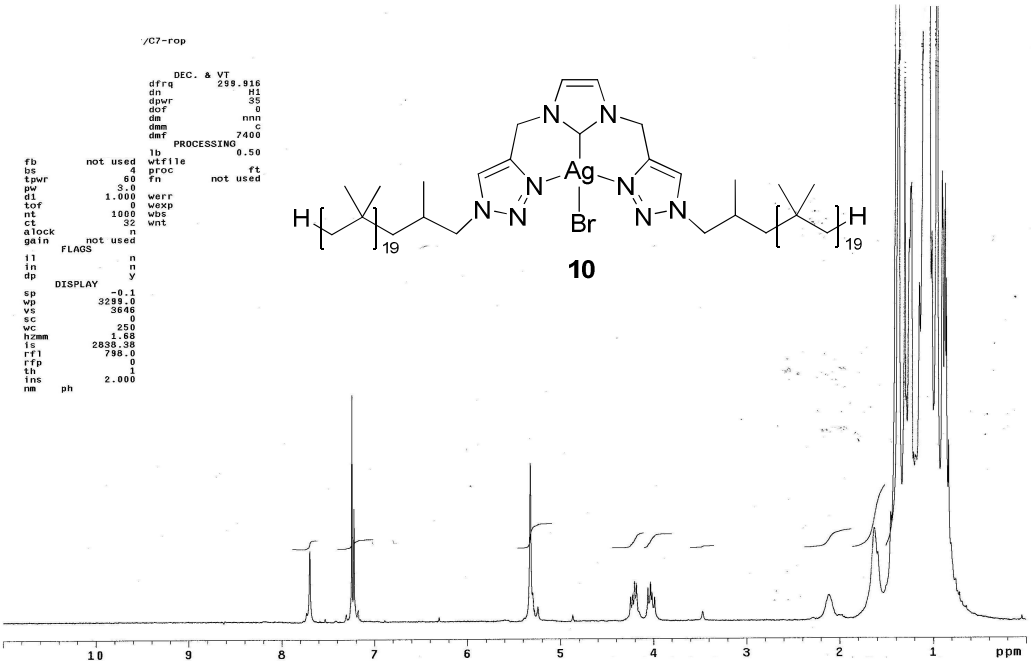

CH6-45
exp2 s2pul
SAMPLE
date Apr 24 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 125.707
tn C13
at 1.300
np 65024
sw 25000.0
fb 14000
bs 8
tpwr 53
pw 5.8
d1 0
tof 0
nt 10000
ct 1397
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -556.7
wp 24999.2
vs 1842
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 557.4
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
H
19
N
N+
Cl-
9
19
H
180
160
140
120
100
80
60
40
20
ppm

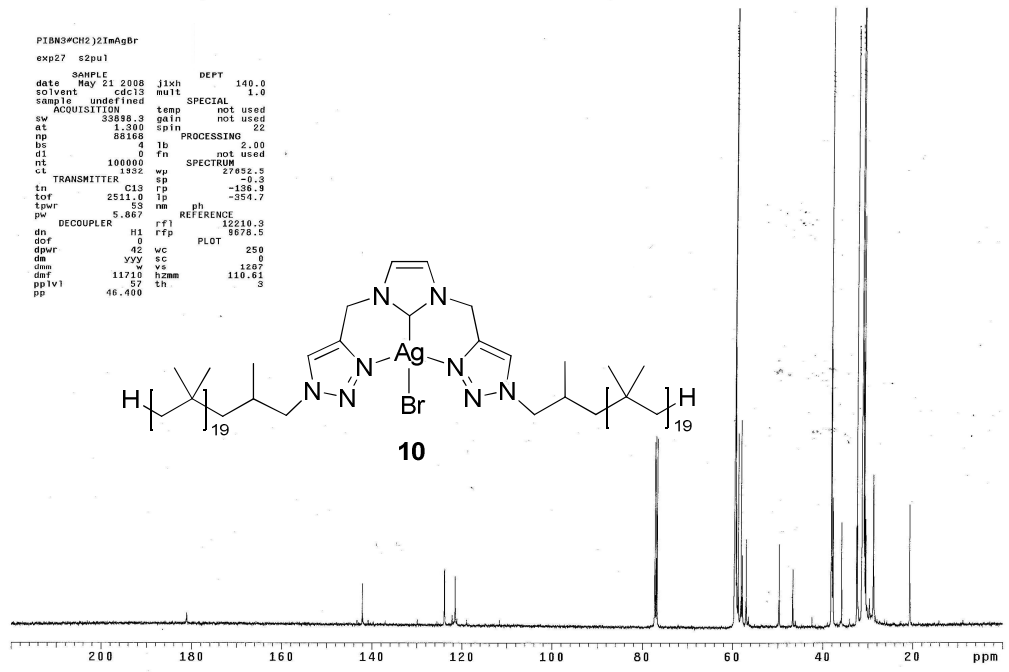

CH6-40
exp2 s2pul
SAMPLE
date Mar 24 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 499.883
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.6
dl 0
tof 0
nt 100
ct 41
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -498.8
wp 6998.3
vs 1409
sc 0
wc 250
hzmm 27.99
is 33.57
rfl 1500.5
rfp 0
th 7
ins 4.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
N
N+
BF4-
H
19
H
19
4
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

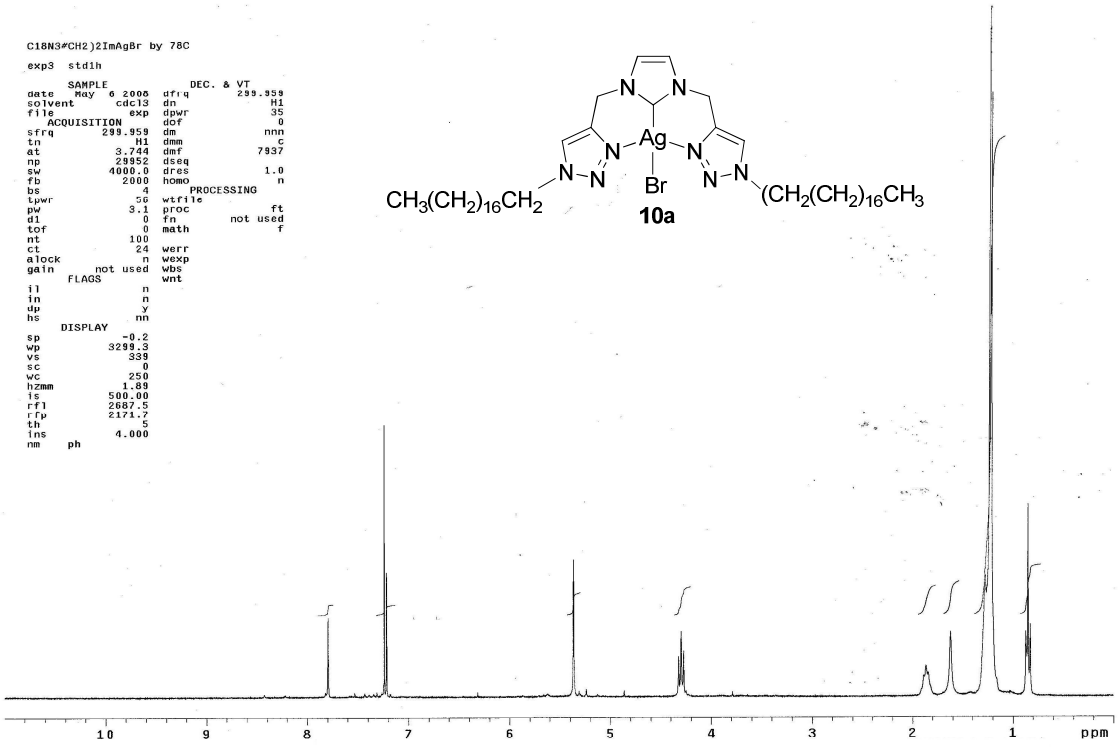

PIB-imidazoliumsalt
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
INOVA-500 "inova500"
Pulse 60.0 degrees
Acq. time 1.300 sec
Width 25000.0 Hz
402 repetitions
OBSERVE C13, 125.6950976 MHz
DECOUPLE H1, 499.8833015 MHz
Power 42 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 45 min, 49 sec
N
N+
BF4-
H
19
H
19
4
180
160
140
120
100
80
60
40
20
ppm

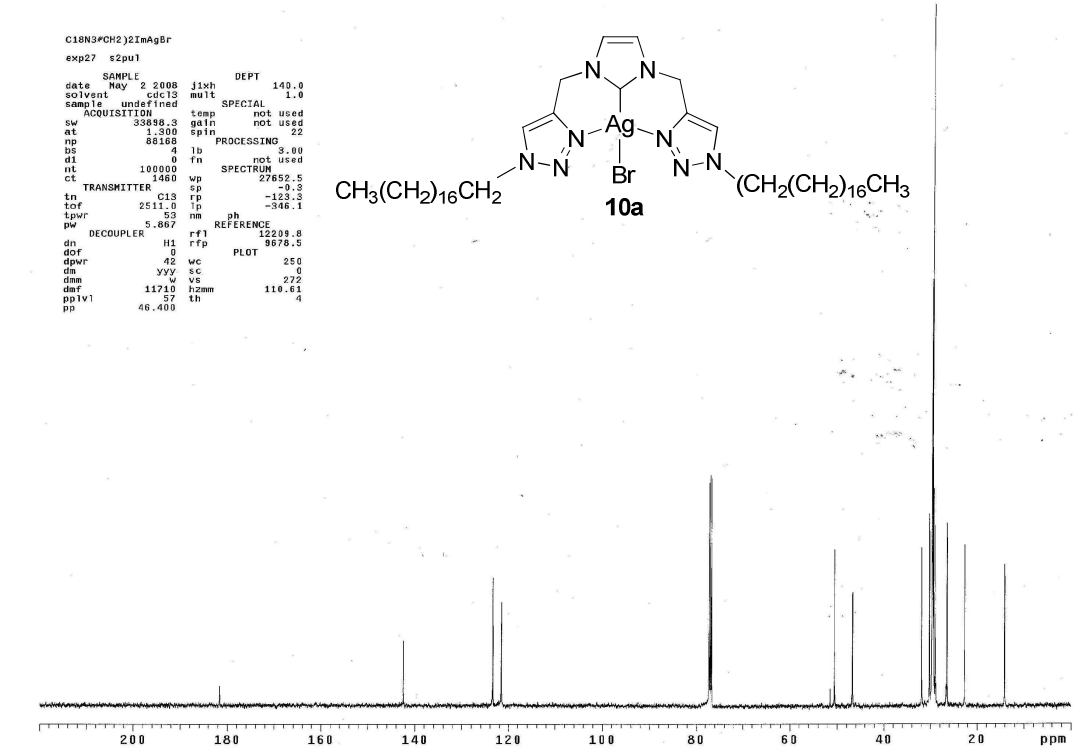

DEC. & VT
sfrq 499.877
n H1
pwr 42
of 0
m nnn
mm c
mf 11710
seq
res 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
tb 4000
bs 4
tpwr 60
pw 3.6
d1 0
tof 0
nt 1000
ct 92
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -0.1
wp 5498.5
vs 2557
sc 0
wc 250
hzmm 2.84
is 2688.00
rfl 1522.5
rfp 0
th 7
ins 4.000
nm ph
Br-
6
19
19
10
9
8
7
6
5
4
3
2
1
ppm

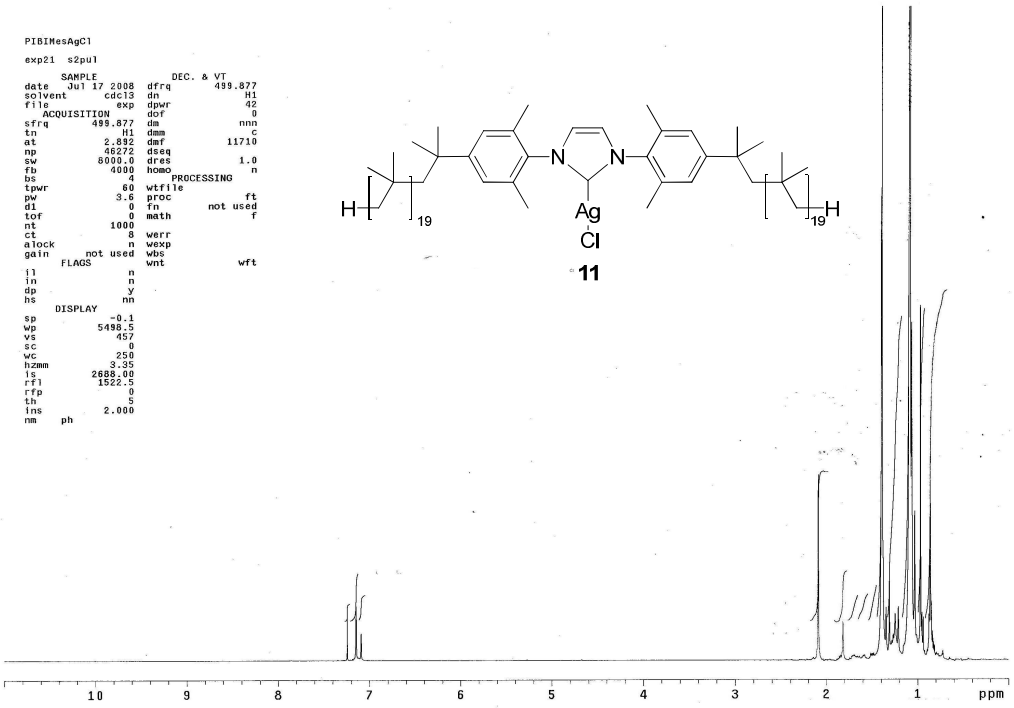

13C OBSERVE
exp27 std13c
SAMPLE
date May 20 2008
solvent cdcl3
file /home/bergbre~
iter/hlsu/PIBNSMCH~
2X2Im.C.fid
ACQUISITION
sfrq 75.432
tn C13
at 1.815
np 59904
sw 16501.7
fb 9100
bs 4
pw 9.9
d1 0
tof 0
nt 10000
ct 800
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -0.4
wp 15084.5
vs 981
sc 0
wc 250
hzmm 60.34
is 3174.69
rfl 6627.5
rfp 5807.7
th 3
ins 100.000
nm ph
DEC. & VT
dfrq 299.961
dn H1
dpwr not used
dof 0
dm yyy
dmm w
dmf 7604
dseq
dres 1.0
homo n
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Br-
6
19
19
180
160
140
120
100
80
60
40
20
ppm

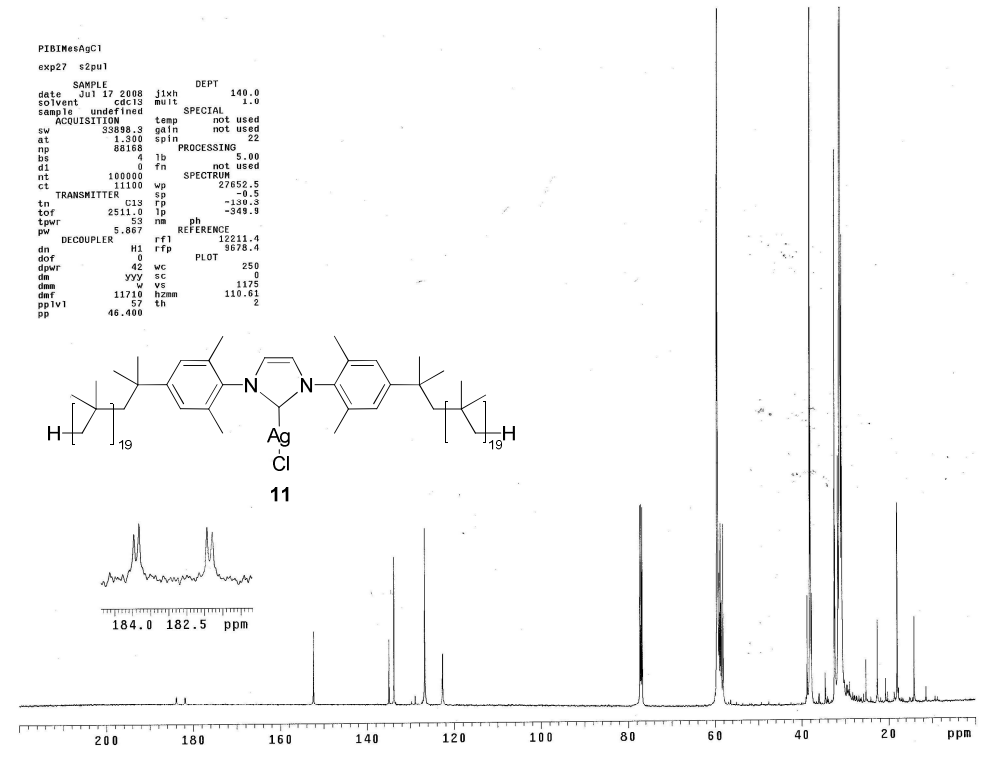

/C7-rop
DEC. & VT
dfrq 299.916
dn H1
dpwr 35
dof 0
dm nnn
dmm c
dmf 7400
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
fb not used
bs 4
tpwr 60
pw 3.0
d1 1.000
tof 0
nt 1000
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.1
wp 3298.0
vs 3646
sc 0
wc 250
hzmm 1.68
is 2838.38
rfl 798.0
rfp 0
th 1
ins 2.000
nm ph
Ag
Br
H
19
10
ppm

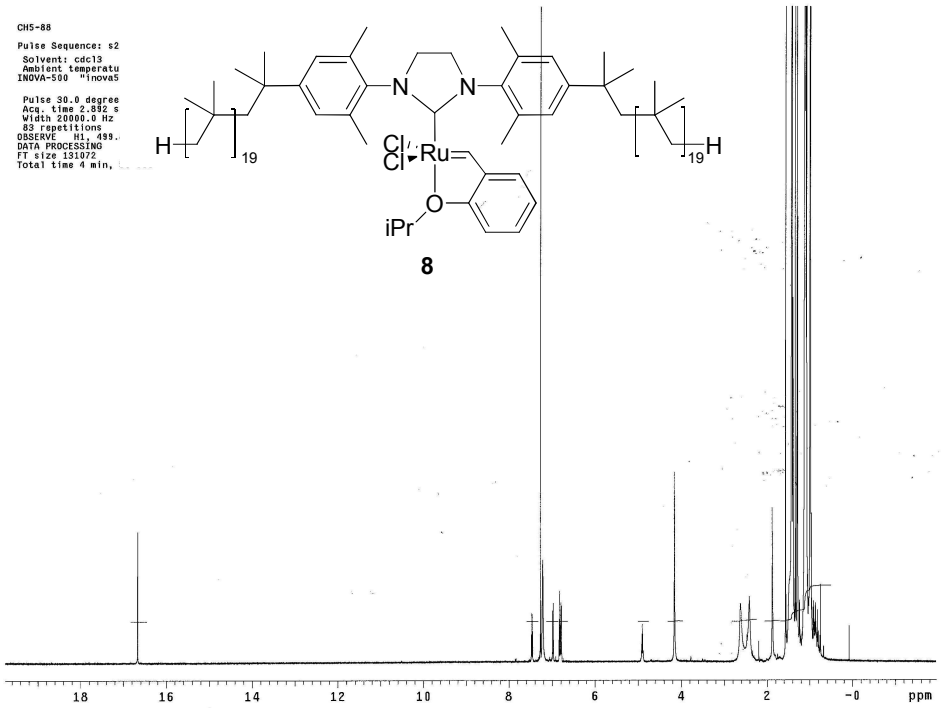

PIBN3#CH2)2ImAgBr
exp27 s2pul
SAMPLE
date May 21 2008
solvent cdcl3
sample undefined
ACQUISITION
sw 33898.3
at 1.300
np 88168
bs 4
d1 0
nt 100000
ct 1932
TRANSMITTER
tn C13
tof 2511.0
tpwr 53
pw 5.867
DECOUPLER
dn H1
dof 0
dpwr 42
dm yyy
dmm w
dmf 11710
pplvl 57
pp 46.400
DEPT
jlxh 140.0
mult 1.0
SPECIAL
temp not used
gain not used
spin 22
PROCESSING
lb 2.00
fn not used
SPECTRUM
wp 27852.5
sp -0.3
rp -136.9
lp -354.7
nm ph
REFERENCE
rfl 12210.3
rfp 8678.5
PLOT
wc 250
sc 0
vs 1267
hzmm 110.61
th 3
Ag
Br
H
19
10
ppm


C18N3#CH2)2ImAgBr by 78C
exp3 std1h
SAMPLE
date May 6 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 299.959
tn H1
at 3.744
np 29952
sw 4000.0
fb 2000
bs 4
tpwr 56
pw 3.1
d1 0
tof 0
nt 100
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -0.2
wp 3299.3
vs 339
sc 0
wc 250
hzmm 1.89
is 500.00
rfl 2687.5
rfp 2171.7
th 5
ins 4.000
nm ph
DEC. & VT
dfrq 299.959
dn H1
dpwr 35
dof 0
dm nnn
dmm c
dmf 7937
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
CH3(CH2)16CH2
(CH2(CH2)16CH3
10a
10
9
8
7
6
5
4
3
2
1
ppm

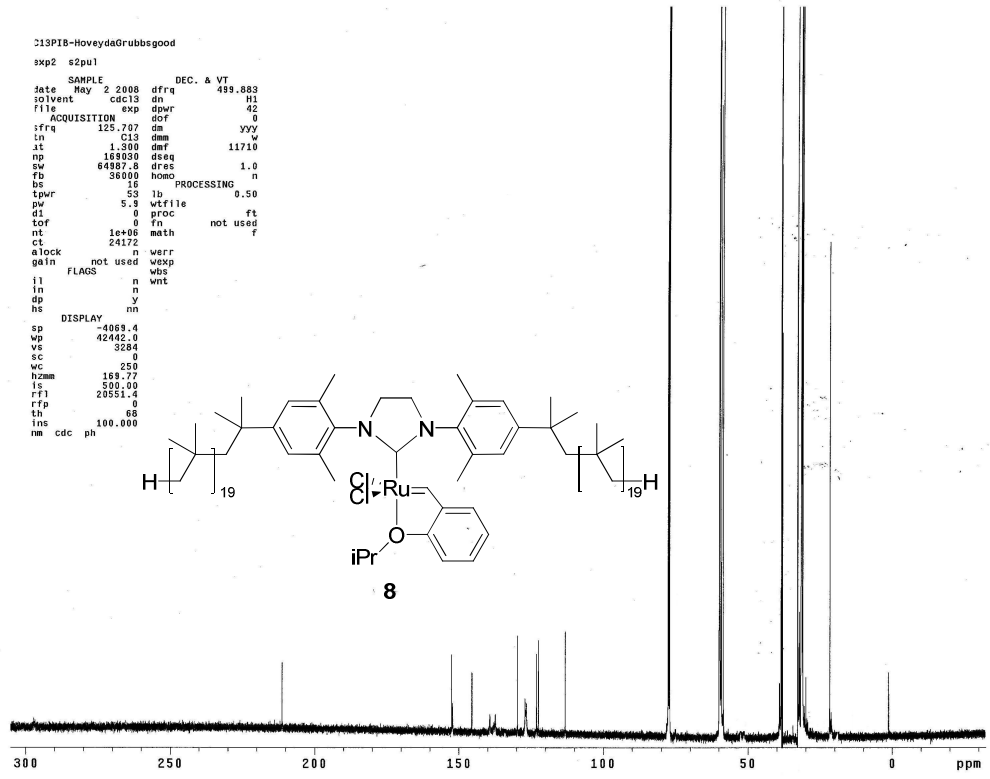

C18N3#CH2)2ImAgBr
exp27 s2pul
SAMPLE
date May 2 2008
solvent cdcl3
sample undefined
ACQUISITION
sw 33898.3
at 1.300
np 88168
bs 4
d1 0
nt 100000
ct 1460
TRANSMITTER
tn C13
tof 2511.0
tpwr 53
pw 5.867
DECOUPLER
dn H1
dof 0
dpwr 42
dm yyy
dmm w
dmf 11710
pplvl 57
pp 46.400
DEPT
jlwh 140.0
mult 1.0
SPECIAL
temp not used
gain not used
spin 22
PROCESSING
lb 3.00
fn not used
SPECTRUM
wp 27652.5
sp -0.3
rp -123.3
lp -346.1
nm ph
REFERENCE
rfl 12209.8
rfp 9678.5
PLOT
wc 250
sc 0
vs 272
hzmm 110.61
th 4
CH3(CH2)16CH2
(CH2(CH2)16CH3
10a
200
180
160
140
120
100
80
60
40
20
ppm


PIBIMesAgCl
exp21 s2pul
SAMPLE
date Jul 17 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 499.877
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.6
d1 0
tof 0
nt 1000
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -0.1
wp 5498.5
vs 457
sc 0
wc 250
hzmm 3.35
is 2688.00
rfl 1522.5
rfp 0
th 5
ins 2.000
nm ph
DEC. & VT
dfrq 499.877
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
H
19
N
N
Ag
Cl
19
H
11
10
9
8
7
6
5
4
3
2
1
ppm


PIBIMesAgCl
exp27 s2pul
SAMPLE
date Jul 17 2008
solvent cdcl3
sample undefined
ACQUISITION
sw 33898.3
at 1.300
np 88168
bs 4
d1 0
nt 100000
ct 11100
TRANSMITTER
tn C13
tof 2511.0
tpwr 53
pw 5.867
DECOUPLER
dn H1
dof 0
dpwr 42
dm yyy
dmm w
dmf 11710
pplvl 57
pp 46.400
DEPT
jlxh 140.0
mult 1.0
SPECIAL
temp not used
gain not used
spin 22
PROCESSING
lb 5.00
fn not used
SPECTRUM
wp 27652.5
sp -0.5
rp -130.3
lp -349.9
nm ph
REFERENCE
rfl 12211.4
rfp 9678.4
PLOT
wc 250
sc 0
vs 1175
hzmm 110.61
th 2
H
19
N
N
Ag
Cl
19
H
11
184.0 182.5 ppm
200
180
160
140
120
100
80
60
40
20
ppm


CH5-88
Pulse Sequence: s2
Solvent: cdcl3
Ambient temperatu
INOVA-500 "inova5
Pulse 30.0 degree
Acq. time 2.882 s
Width 20000.0 Hz
83 repetitions
OBSERVE H1, 499.
DATA PROCESSING
FT size 131072
Total time 4 min,
H
19
N
N
Cl
Cl
Ru
O
iPr
19
H
18
16
14
12
10
8
6
4
2
-0
ppm

**8**


C13PIB-HoveydaGrubbsgood
exp2 s2pul
SAMPLE
date May 2 2008
solvent cdcl3
file exp
ACQUISITION
sfrq 125.707
tn C13
at 1.300
np 168030
sw 64987.8
fb 36000
bs 16
tpwr 53
pw 5.9
d1 0
tof 0
nt 1e+06
ct 24172
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -4069.4
wp 42442.0
vs 3284
sc 0
wc 250
hzmm 169.77
is 500.00
rfl 20551.4
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.883
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
H
19
N
N
Cl
Cl
Ru
O
iPr
19
H
300
250
200
150
100
50
0
ppm

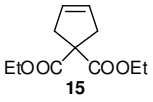
**8**

CH4-77second

Pulse Sequence: s2pul

Solvent: cdcl3
Ambient temperature
INOVA-500 "inova500"

Pulse 30.0 degrees
Acq. time 2.892 sec
Width 8000.0 Hz
12 repetitions
OBSERVE H1, 499.9420431 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 50 sec



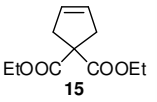

**15**



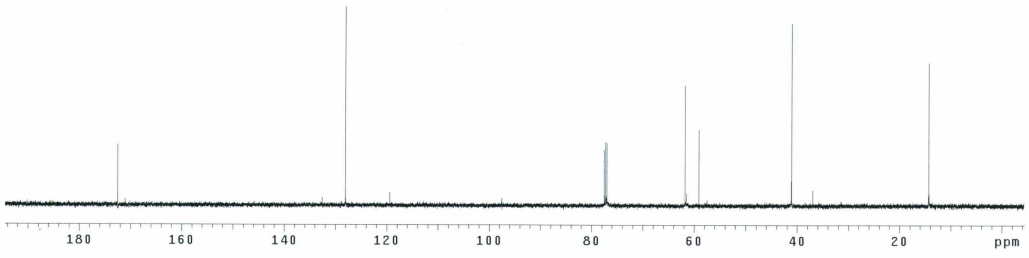

RCM of diallylethylmalonate

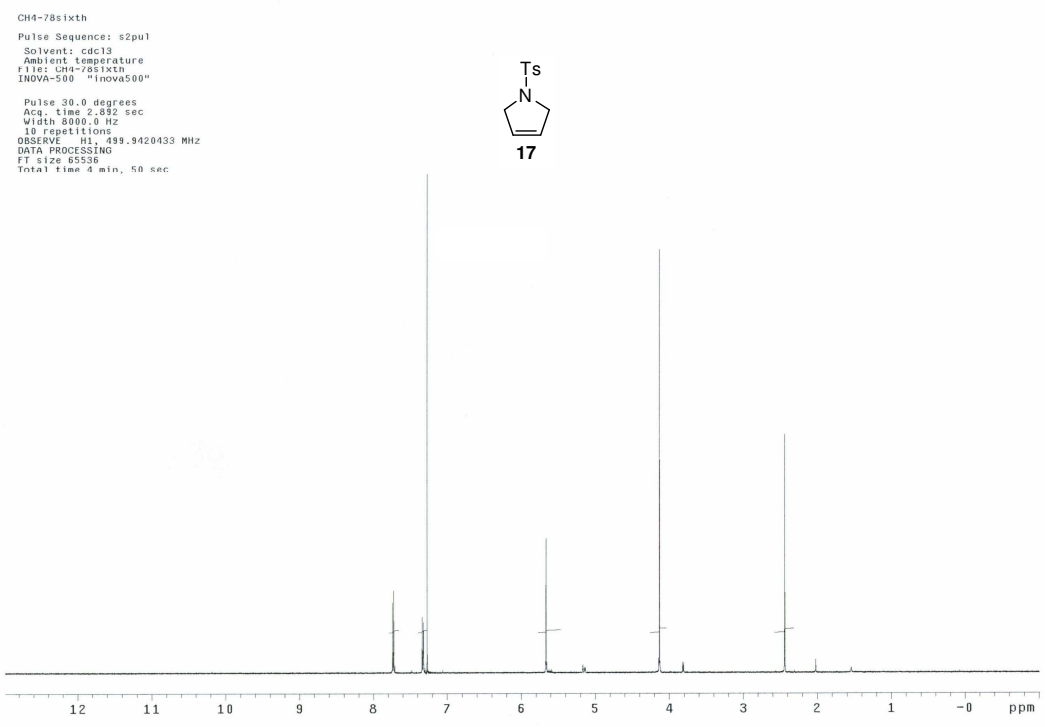
exp1 s2pul

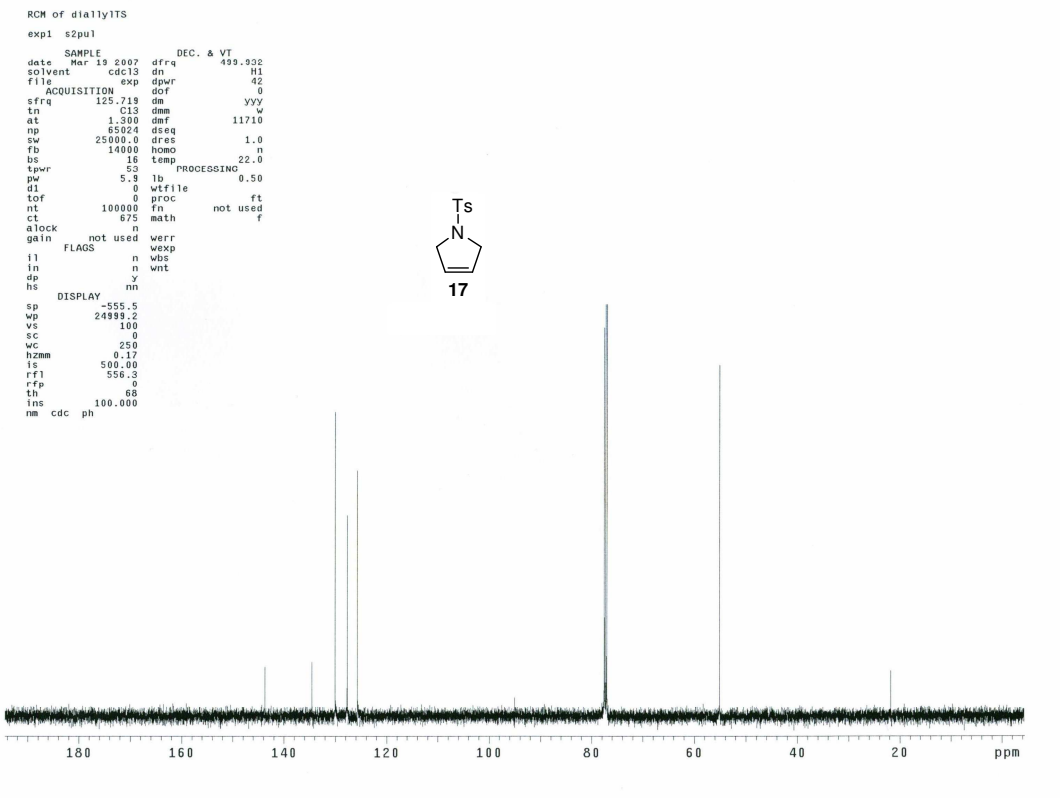
| SAMPLE | | DEC. & VT | |
|---|---|---|---|
| date | Mar 19 2007 | dfrq | 499.932 |
| solvent | cdcl3 | dn | H1 |
| file | exp | dpwr | 42 |
| ACQUISITION | | dof | 0 |
| sfrq | 125.719 | dm | yyy |
| tn | C13 | dmm | w |
| at | 1.300 | dmf | 11710 |
| np | 65024 | dseq | |
| sw | 25000.0 | dres | 1.0 |
| fb | 14000 | homo | n |
| bs | 16 | temp | 22.0 |
| tpwr | 53 | PROCESSING | |
| pw | 5.9 | lb | 0.50 |
| d1 | 0 | wtfile | |
| tof | 0 | proc | ft |
| nt | 10000 | fn | not used |
| ct | 181 | math | f |
| alock | n | | |
| gain | not used | werr | |
| FLAGS | | wexp | |
| il | n | wbs | |
| in | n | wnt | |
| dp | y | | |
| hs | nn | | |
| DISPLAY | | | |
| sp | -555.5 | | |
| wp | 24999.2 | | |
| vs | 50 | | |
| sc | 0 | | |
| wc | 250 | | |
| hzmm | 0.17 | | |
| is | 500.00 | | |
| rfl | 556.3 | | |
| rfp | 0 | | |
| th | 68 | | |
| ins | 100.000 | | |
| nm cdc ph | | | |

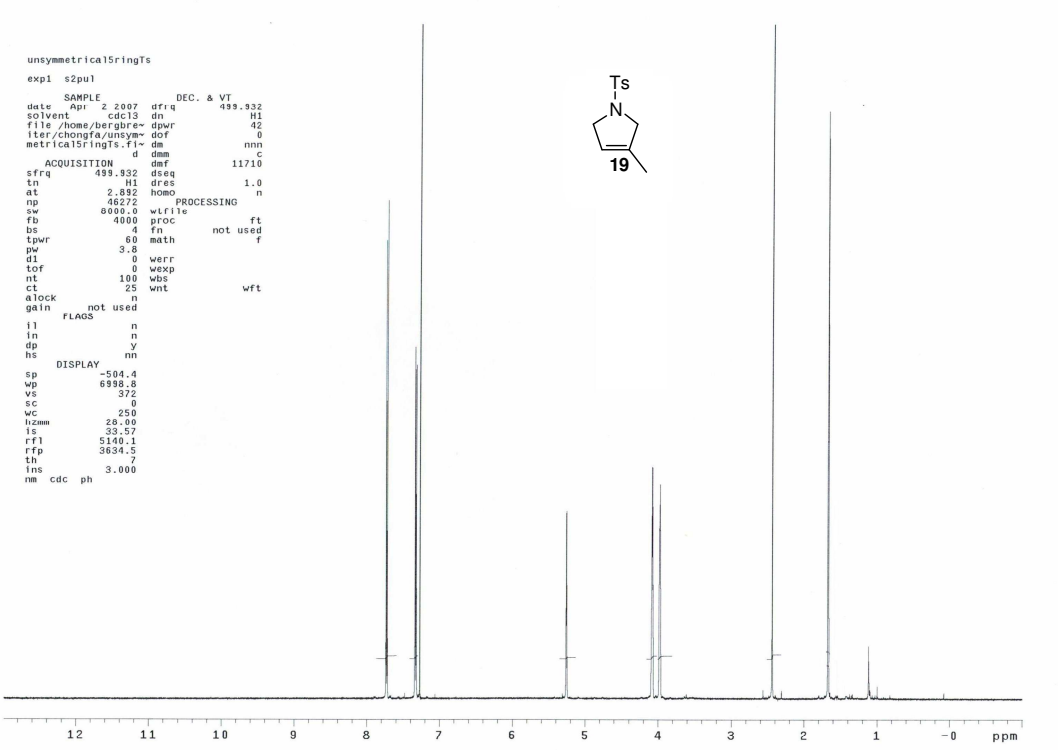


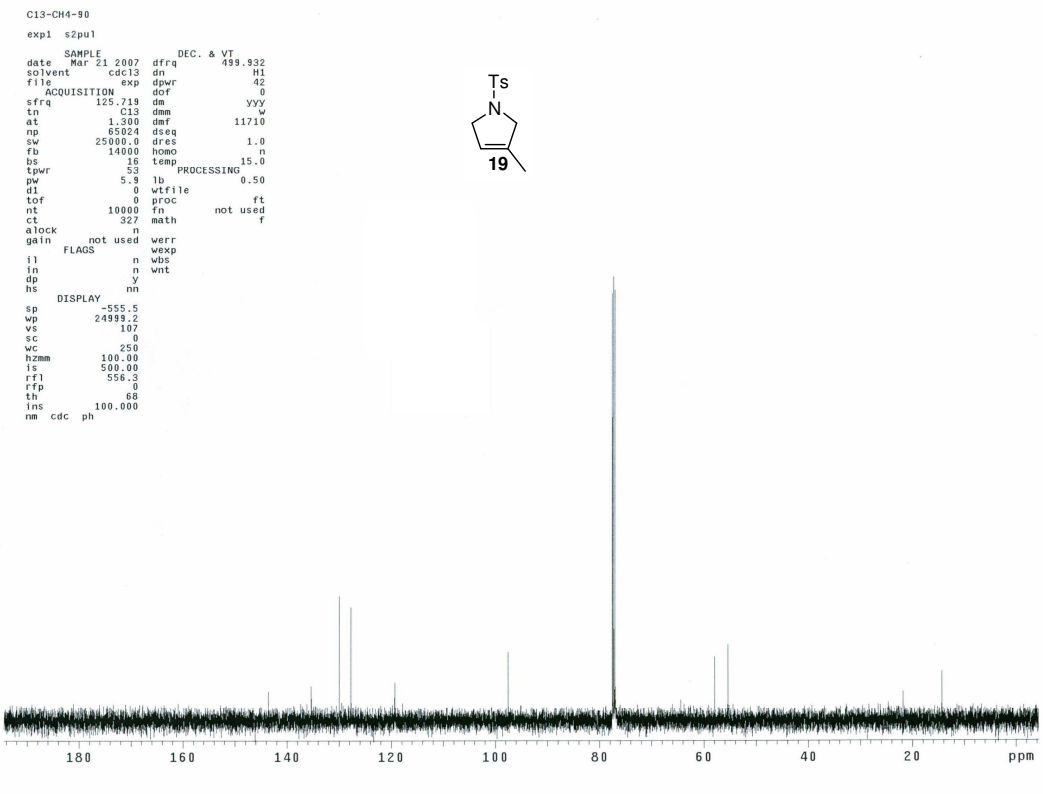

**15**

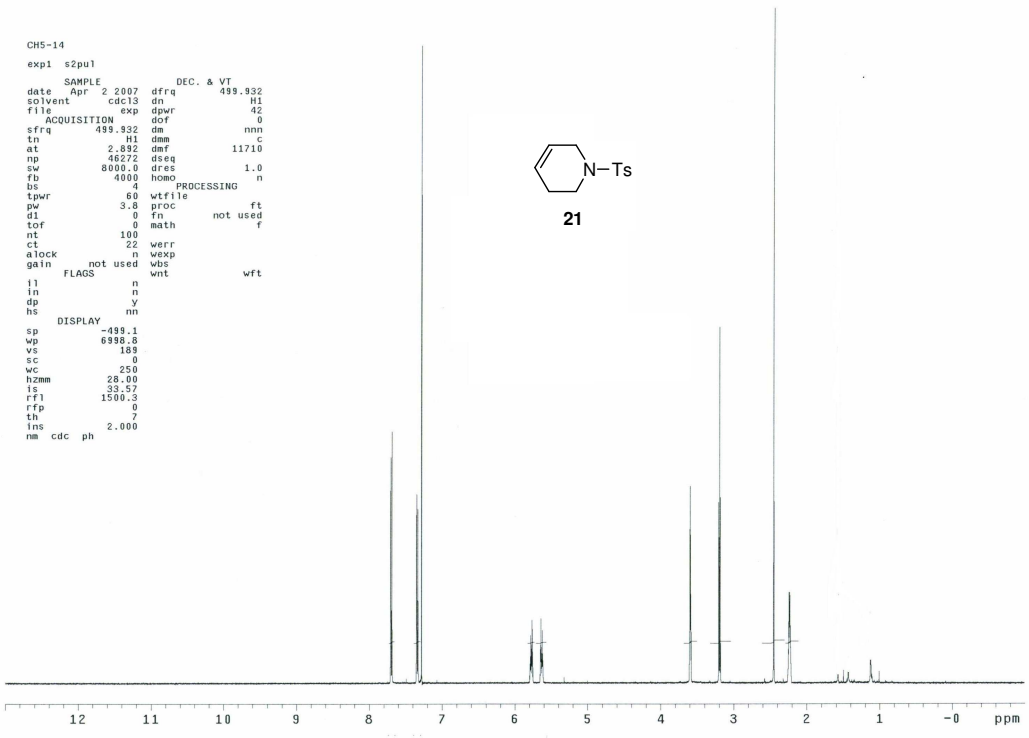


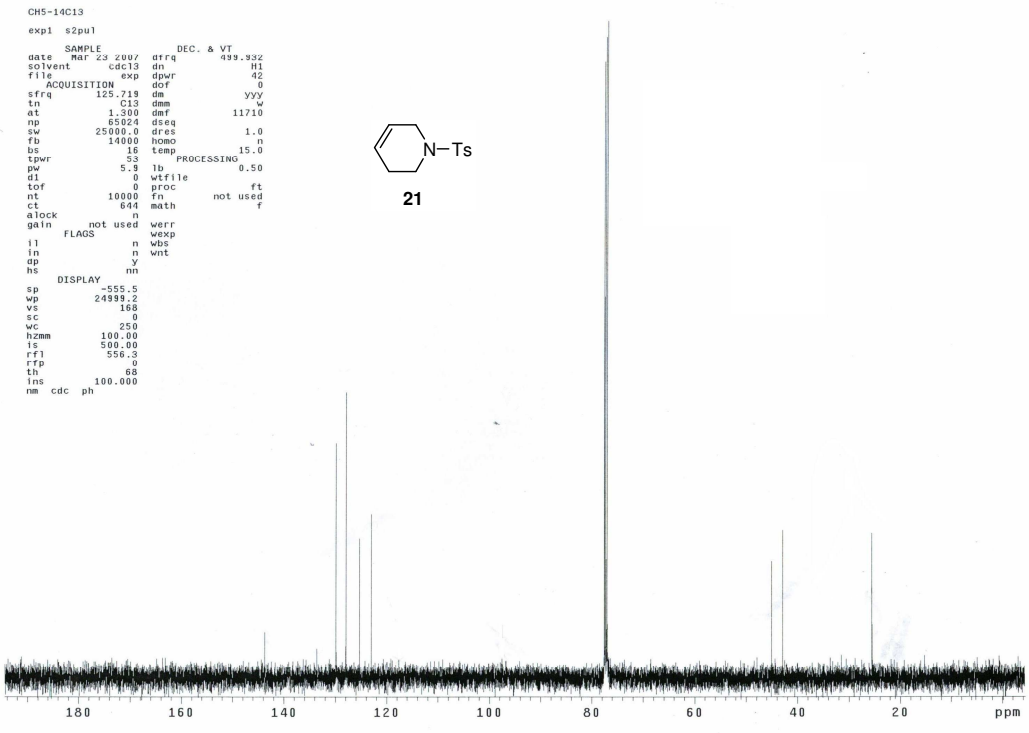

CH4-78sixth
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: CH4-78sixth
INOVA-500 "inova500"
Pulse 30.0 degrees
Acq. time 2.892 sec
Width 8000.0 Hz
10 repetitions
OBSERVE H1, 499.9420433 MHz
DATA PROCESSING
FT size 65536
Total time 4 min, 50 sec
Ts
N
17
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm

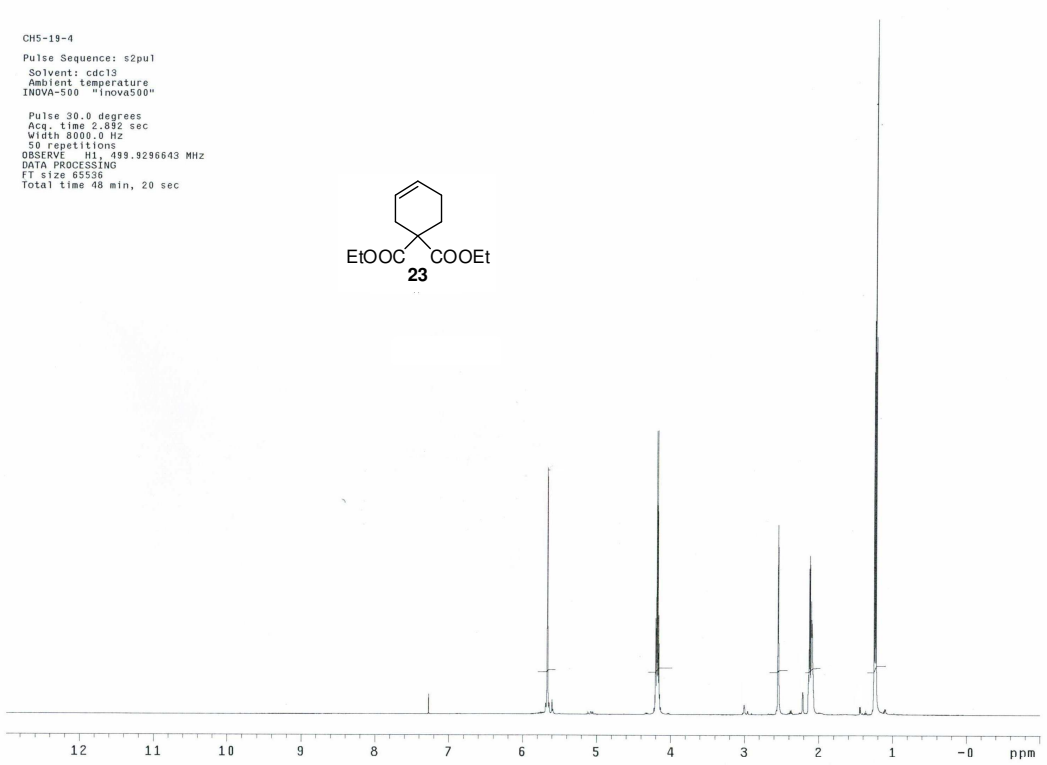

RCM of diallyITS
exp1 s2pul
SAMPLE
date Mar 19 2007
solvent cdcl3
file exp
ACQUISITION
sfrq 125.719
tn C13
at 1.300
np 65024
sw 25000.0
fb 14000
bs 16
tpwr 53
pw 5.9
d1 0
tof 0
nt 100000
ct 675
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -555.5
wp 24999.2
vs 100
sc 0
wc 250
hzmm 0.17
is 500.00
rfl 556.3
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.932
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
temp 22.0
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Ts
N
17
180
160
140
120
100
80
60
40
20
ppm

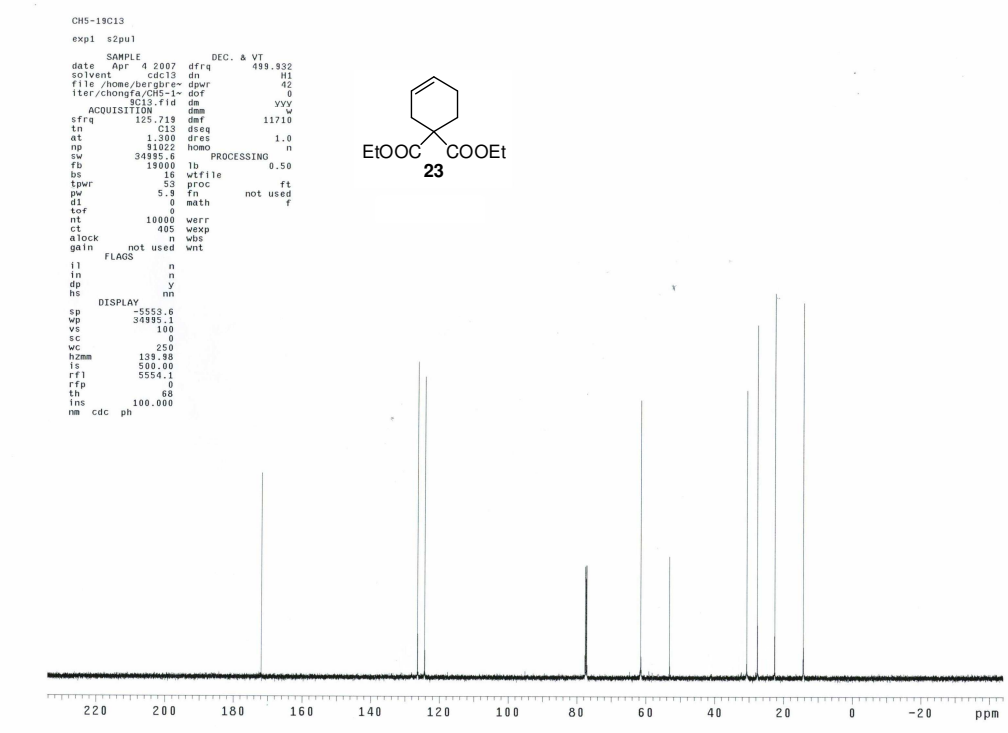

unsymmetrical5ringTs
exp1 s2pul
SAMPLE
date Apr 2 2007
solvent cdcl3
file /home/bergbre~
iter/chongfa/unsym~
metrical5ringTs.fi~
d
ACQUISITION
sfrq 499.932
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.8
d1 0
tof 0
nt 100
ct 25
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -504.4
wp 6998.8
vs 372
sc 0
wc 250
hzmm 28.00
is 33.57
rfl 5140.1
rfp 3634.5
th 7
ins 3.000
nm cdc ph
DEC. & VT
dfrq 499.932
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt wft
Ts
N
19
12
11
10
9
8
7
6
5
4
3
2
1
-0
ppm


C13-CH4-90
exp1 s2pul
SAMPLE
date Mar 21 2007
solvent cdcl3
file exp
ACQUISITION
sfrq 125.719
tn C13
at 1.300
np 65024
sw 25000.0
fb 14000
bs 16
tpwr 53
pw 5.9
d1 0
tof 0
nt 10000
ct 327
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -555.5
wp 24999.2
vs 107
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 556.3
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.932
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
temp 15.0
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
Ts
N
19
180
160
140
120
100
80
60
40
20
ppm


CH5-14
exp1 s2pul
SAMPLE
date Apr 2 2007
solvent cdcl3
file exp
ACQUISITION
sfrq 499.932
tn H1
at 2.892
np 46272
sw 8000.0
fb 4000
bs 4
tpwr 60
pw 3.8
d1 0
tof 0
nt 100
ct 22
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -499.1
wp 6998.8
vs 189
sc 0
wc 250
hzmm 28.00
is 33.57
rfl 1500.3
rfp 0
th 7
ins 2.000
nm cdc ph
DEC. & VT
dfrq 499.932
dn H1
dpwr 42
dof 0
dm nnn
dmm c
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn not used
math f
wexp
wbs
wnt wft
N–Ts
21
12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm


CH5-14C13
exp1 s2pul
SAMPLE
date Mar 23 2007
solvent cdcl3
file exp
ACQUISITION
sfrq 125.719
tn C13
at 1.300
np 65024
sw 25000.0
fb 14000
bs 16
tpwr 53
pw 5.9
d1 0
tof 0
nt 10000
ct 644
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -555.5
wp 24999.2
vs 168
sc 0
wc 250
hzmm 100.00
is 500.00
rfl 556.3
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.932
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
temp 15.0
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
N–Ts
21
180 160 140 120 100 80 60 40 20 ppm


CH5-19-4
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
INOVA-500 "inova500"
Pulse 30.0 degrees
Acq. time 2.892 sec
Width 8000.0 Hz
50 repetitions
OBSERVE H1, 499.9286643 MHz
DATA PROCESSING
FT size 65536
Total time 48 min, 20 sec
EtOOC COOEt
23
12 11 10 9 8 7 6 5 4 3 2 1 -0 ppm


CH5-19C13
exp1 s2pul
SAMPLE
date Apr 4 2007
solvent cdcl3
file /home/bergbre~
iter/chongfa/CH5-1~
9C13.fid
ACQUISITION
sfrq 125.719
tn C13
at 1.300
np 91022
sw 34995.6
fb 19000
bs 16
tpwr 53
pw 5.9
dl 0
tof 0
nt 10000
ct 405
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -5553.6
wp 34995.1
vs 100
sc 0
wc 250
hzmm 139.98
is 500.00
rfl 5554.1
rfp 0
th 68
ins 100.000
nm cdc ph
DEC. & VT
dfrq 499.932
dn H1
dpwr 42
dof 0
dm yyy
dmm w
dmf 11710
dseq
dres 1.0
homo n
PROCESSING
lb 0.50
wtfile
proc ft
fn not used
math f
werr
wexp
wbs
wnt
EtOOC COOEt
23
220 200 180 160 140 120 100 80 60 40 20 0 -20 ppm

## References


(1) Li, J.; Sung, S.; Tian, J.; Bergbreiter, D. E. *Tetrahedron* **2005**, *61*, 12081–12092.
(2) Yao, Q.; Zhang Y. *J. Am. Chem. Soc.* **2004**, *126*, 74-75.
(3) Audic, N.; Clavier, H.; Mauduit, M.; Guillemin, J.-C. *J. Am. Chem. Soc.* **2003**, *125*, 9248-9249.
(4) Romero, P. E.; Piers, W. E.; McDonald, R. *Angew. Chem., Int. Ed.* **2004**, *43*, 6161-6165.