

Supporting Information for Publication

Synthesis of 2,3,5,6-Tetrasubstituted Tetrahydropyrans via (3,5)-Oxonium-Ene Reaction

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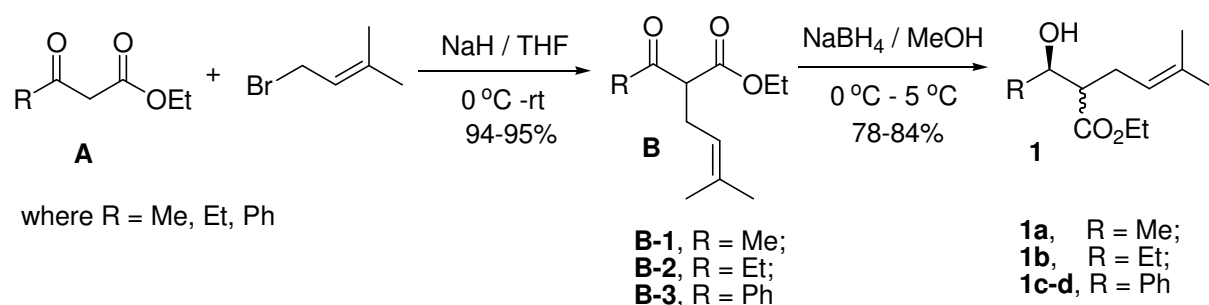
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General Information: All reagents are commercially obtained. $\text{BF}_3 \cdot \text{Et}_2\text{O}$ was distilled over CaH_2 prior to use. ^1H NMR spectra were recorded in CDCl_3 on 400 MHz NMR spectrometer using TMS as internal standard. The ^{13}C and ^{19}F NMR spectra were recorded at 100 MHz and 376 MHz, respectively. For ^{13}C and ^{19}F NMR CDCl_3 and C_6F_6 were used as internal standard. IR spectra were recorded on FT-IR spectrometer. Melting points were measured in open capillary tubes and are uncorrected.

Preparation of ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate

1: The ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate **1** was synthesized starting from β -keto ester **A** and 1-bromo-3-methylbut-2-ene as shown in Scheme 1.¹ Thus the reaction of β -keto ester **A** with 1-bromo-3-methylbut-2-ene in presence



Scheme 1: Synthesis of ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate

of sodium hydride in THF afforded α -substituted β -keto ester **B**, which after reduction with sodium borohydride in methanol gives alcohol **1** in 78-84% yields.

The methyl and ethyl substituted β -keto esters **B-1** and **B-2** afforded two inseparable diastereomers² **1a,b** whereas phenyl substituted β -ketoester **B-3** gave two separable *anti*- and *syn*-diastereomers **1c** and **1d**, respectively. The structures of all compounds are determined

from IR, ^1H , ^{13}C NMR and mass spectroscopy. The stereochemistry of compounds **1c** and **1d** are determined from coupling constants values.^{2b,3}

References:

1. M. Peña-López, M. M. Martinez, L. A. Sarandeses and J. S. Sestelo, *J. Org. Chem.* 2010, **75**, 5337-5339.
2. (a) V. A. Kramer and H. Pfander, *Helv. Chim. Acta* 1982, **65**, 293-301; (b) P. Galatsis, S. D. Millan, P. Nechala and G. Ferguson, *J. Org. Chem.* 1994, **59**, 6643-6651.
3. E. Marcantoni, S. Alessandrini, M. Malavolta, G. Bartoli, M. C. Bellucci and L. Sambri, *J. Org. Chem.* 1999, **64**, 1986-1992.

General procedure for the synthesis of α -alkyl- β -keto esters (B-1-3): To a suspension of sodium hydride (15.69 mmol, 1 equiv.) in THF (15 mL) at 0 °C was added β -keto ester (15.69 mmol, 1 equiv.) dropwise via syringe. After 20 min, a solution of 3,3-dimethylallyl bromide (17.26 mmol, 1.1 equiv.) in THF (5 mL) was added and the mixture was left at room temperature overnight. The solvent was removed under reduced pressure, and the residue was dissolved in Et₂O (10 mL) and washed with brine (25 ml). The organic layer was dried and filtered, and the filtrate was concentrated under reduced pressure. The residue was purified by column chromatography over silica gel to afford the title compounds as colourless oil.

Synthesis of Ethyl 2-acetyl-5-methylhex-4-enoate (B-1): To a suspension of sodium hydride (0.378 g, 15.69 mmol) in THF (15 mL) at 0 °C was added ethyl acetoacetate (2 mL, 15.69 mmol) dropwise via syringe. After 20 min, a solution of 3,3-dimethylallyl bromide (2 mL, 17.26 mmol) in THF (5 mL) was added and the mixture was left at room temperature overnight. The solvent was removed under reduced pressure, and the residue was dissolved in Et₂O (10 mL) and washed with brine (25 mL). The organic layer was dried and filtered, and

chromatography over silica gel (10% EtOAc/hexane) to afford ethyl 2-acetyl-5-methylhex-4-enoate **B-1** (2.97 g, 95%) as a colourless oil; ^1H NMR (400 MHz, CDCl_3): δ 1.27 (t, $J = 7.2$ Hz, 3 H), 1.63 (s, 3 H), 1.68 (s, 3 H), 2.22 (s, 3 H), 2.54 (t, $J = 7.2$ Hz, 2 H), 3.43 (t, $J = 7.6$ Hz, 1 H), 4.19 (q, $J = 7.2$ Hz, 2 H), 5.03 (dt, $J = 7.6$ and 1.6 Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.1, 17.8, 25.8, 27.0, 29.1, 59.8, 61.3, 119.9, 134.7, 169.7, 203.2; IR (KBr, Neat): 2981, 2930, 1739, 1718, 1205, 1150 cm^{-1} . HRMS (APCI) cald. for $\text{C}_{11}\text{H}_{18}\text{O}_3$ ($\text{M}+\text{H}$) $^+$ requires 199.1334; found 199.1337. APCI-MS: m/z (relative intensity): 199.2 ($(\text{M}+\text{H})^+$, 43%), 181.1 (100), 169.1 (55), 143.1 (17), 124.1 (43), 107.1 (73).

Ethyl 5-methyl-2-propionylhex-4-enoate (B-2): Colourless oil (3.13 g, 94%); ^1H NMR (400 MHz, CDCl_3): δ 1.06 (t, $J = 7.2$ Hz, 3 H), 1.26 (t, $J = 7.2$, 3 H), 1.62 (s, 3 H), 1.67 (s, 3 H), 2.47-2.60 (m, 4 H), 3.45 (t, $J = 7.2$ Hz, 1 H), 4.17 (q, $J = 7.2$ Hz, 2 H), 5.02 (dt, $J = 7.6$ and 1.6 Hz, 1 H); ^{13}C NMR (100 MHz, CDCl_3): δ 7.8, 14.2, 17.9, 25.9, 27.2, 35.6, 58.9, 61.4, 120.1, 134.8, 169.9, 206.0; IR (KBr, Neat): 2980, 2937, 1741, 1716, 1198, 1156 cm^{-1} . HRMS (APCI) cald. for $\text{C}_{12}\text{H}_{20}\text{O}_3$ (M^+) requires 212.1412; found 212.1408. APCI-MS: m/z (relative intensity): 212.2 ($(\text{M}+\text{H})^+$, 100%), 194.7 (65), 168.7 (27), 123.8 (22).

Ethyl 2-benzoyl-5-methylhex-4-enoate (B-3): Colourless oil (3.88 g, 95%); ^1H NMR (400 MHz, CDCl_3): δ 1.67 (t, $J = 7.2$ Hz, 3 H), 1.62 (s, 3 H), 1.65 (s, 3 H), 2.61-2.78 (m, 2 H), 4.14 (q, $J = 7.2$ Hz, 2 H), 4.30 (t, $J = 7.2$ Hz, 1 H), 5.11 (dt, $J = 7.2$ and 1.6 Hz, 1 H), 7.44-7.50 (m, 2 H), 7.55-7.61 (m, 1 H), 7.97-8.00 (m, 2 H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.1, 17.9, 25.8, 27.8, 54.6, 61.4, 120.3, 128.7, 128.8, 133.5, 134.7, 136.4, 169.9, 195.2; IR (KBr, Neat): 2979, 2929, 1737, 1688, 1448, 1379, 1241, 1153 cm^{-1} . HRMS (APCI) cald. for $\text{C}_{16}\text{H}_{20}\text{O}_3$ ($\text{M}+\text{H}$) $^+$ requires 261.1490; found 261.1494. APCI-MS: m/z (relative intensity): 261.2 ($(\text{M}+\text{H})^+$, 57%), 215.1 (21), 193.1 (23), 169.1 (12), 124.1 (100).

General procedure for the synthesis of Ethyl 2-(1-hydroxyalkyl/hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1a-d): To a solution of α -alkyl- β -keto esters (14.98 mmol, 1 equiv.) in dry MeOH (15 mL) at 0 °C, was added sodium borohydride (39.25 mmol, 2.62 equiv.) in small portions. The reaction mixture was stirred in between 0 °C to 5 °C for 1.5 h. The progress of the reaction was monitored by TLC with ethyl acetate and hexane as eluents. After completion of the reaction, the product was extracted with ethyl acetate (30 mL) and then washed with water (15 mL) and brine (15 mL). The organic layer was dried (Na₂SO₄) and evaporated to leave the crude product, which was purified by column chromatography over silica gel to give the title compounds.

Synthesis of Ethyl 2-(1-hydroxyethyl)-5-methylhex-4-enoate (1a): To a solution of ethyl 2-acetyl-5-methylhex-4-enoate (2.97 g, 14.98 mmol) in dry MeOH (15mL) at 0 °C, was added sodium borohydride (1.485 g, 39.25 mmol) in small portions. The reaction mixture was stirred in between 0 °C to 5 °C for 1.5 h. The progress of the reaction was monitored by TLC with ethyl acetate and hexane (3:22) as eluents. After completion of the reaction, the product was extracted with ethyl acetate (30 mL) and then washed with water (15 mL) and brine (15 mL). The organic layer was dried (Na₂SO₄) and evaporated to leave the crude products, which were purified by column chromatography over silica gel to give an inseparable mixture of two diastereomers **1a** (2.40 g, 80% overall yield) as a colourless oil; ¹H NMR (400 MHz, CDCl₃): δ 1.19-1.29 (m, 6 H), 1.62 (s, 3 H), 1.69 (s, 3 H), 2.29-2.47 (m, 3 H), 2.51 (brs 0.5 H), 2.69 (brs, 0.5 H), 3.86-3.96 (m, 0.5 H), 3.98-4.06 (m, 0.5 H), 4.08-4.24 (m, 2 H), 5.04-5.14 (m, 1 H); ¹³C NMR (100 MHz, CDCl₃): δ 14.1(2C), 17.5(2C), 20.5, 21.1, 25.6(2C), 26.7, 27.7, 53.0, 53.1, 60.2, 60.3, 67.8, 67.9, 120.5, 121.1, 133.3, 133.6, 174.7, 175.0; IR (KBr, Neat): 3441, 2972, 2929, 1732, 1640, 1182, 1155 cm⁻¹. HRMS (APCI) calcd. for C₁₁H₂₀O₃ (M+H)⁺ requires 201.1490; found 201.1498. APCI-MS: m/z (relative intensity):

201.2 ((M+H)⁺, 30%), 137.1 (4), 125.1 (11), 124.1 (100), 123.1 (14), 82.0 (24).

Ethyl 2-(1-hydroxypropyl)-5-methylhex-4-enoate (1b): Colourless oil (2.50 g, 78%); ¹H NMR (400 MHz, CDCl₃): δ 0.98 (t, *J* = 7.6 Hz, 3 H), 1.25 (t, *J* = 7.2 Hz, 1.5 H), 1.26 (t, *J* = 7.2 Hz, 1.5 H), 1.45-1.54 (m, 2H), 1.61 (s, 1.5 H), 1.62 (s, 1.5 H), 1.69 (s, 3 H), 2.28-2.50 (m, 3 H), 2.67 (brs, 1 H), 3.56-3.64 (m, 0.5 H), 3.70-3.78 (m, 0.5 H), 4.08-4.22 (m, 2 H), 5.04-5.16 (m, 1 H); ¹³C NMR (100 MHz, CDCl₃): δ 10.2, 10.3, 14.3(2C), 17.8(2C), 25.8, 26.1, 27.4(2C), 28.3, 28.6, 50.7, 51.2, 60.5(2C), 73.3, 73.4, 120.7, 121.3, 133.6, 134.1, 175.3, 175.5; IR (KBr, Neat): 3456, 2968, 2932, 1731, 1642, 1183, 1159 cm⁻¹. HRMS (APCI) calcd. for C₁₂H₂₂O₃ (M+H)⁺ requires 215.1647; found 215.1653. APCI-MS: m/z (relative intensity): 215.2 ((M+H)⁺, 100%), 197.2 (6), 169.1 (8), 151.1 (7), 124.1 (35), 123.1 (14), 82.0 (7).

Synthesis of *anti/syn*-Ethyl 2-(hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1c & 1d):

To a solution of ethyl 2-benzoyl-5-methylhex-4-enoate (3.90 g, 14.98 mmol) in dry MeOH (15 mL) at 0 °C, was added sodium borohydride (1.485 g, 39.25 mmol) in small portions. The reaction mixture was stirred in between 0 °C to 5 °C for 1.5 h. The progress of the reaction was monitored by TLC with ethyl acetate and hexane (7:43) as eluents. After completion of the reaction, the product was extracted with ethyl acetate (30 mL) and then washed with water (15 mL) and brine (15 mL). The organic layer was dried (Na₂SO₄) and evaporated to leave the crude products, which were separated by column chromatography over silica gel to give **1c** (1.73 g, 44%) and **1d** (1.58 g, 40%) as a colourless oil.

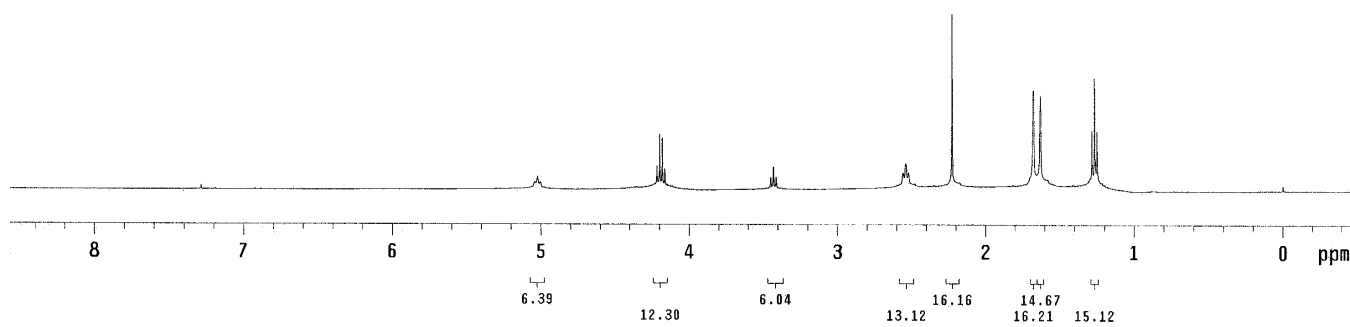
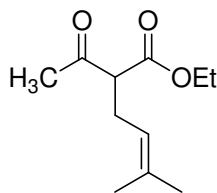
***anti*-Ethyl 2-(hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1c):** Colourless oil (1.73 g, 44%); ¹H NMR (400 MHz, CDCl₃): δ 1.18 (t, *J* = 7.2 Hz, 3 H), 1.50 (s, 3 H), 1.65 (s, 3 H), 2.05 (ddd, *J* = 13.2, 7.6 and 5.6 Hz, 1 H), 2.25 (ddd, *J* = 14.8, 8.0 and 6.8 Hz, 1 H), 2.75 (ddd, *J* = 12.8, 8.8, 5.2 Hz, 1 H), 3.16 (d, *J* = 5.2 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2 H), 4.80 (dd,

$J = 6.4$ and 4.4 Hz, 1H), 5.03 (dt, $J = 6.8$ and 1.2 Hz, 1H), 7.26-7.37 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.3, 17.8, 25.9, 28.4, 53.3, 60.7, 74.8, 120.2, 126.6, 128.0, 128.6, 134.3, 142.2, 175.2; IR (KBr, Neat): 3461, 2978, 2929, 1729, 1452, 1377, 1180, 1037, 766, 702 cm^{-1} . HRMS (APCI) cald. for $\text{C}_{16}\text{H}_{22}\text{O}_3$ (M^+) requires 262.1569; found 262.1573. APCI-MS: m/z (relative intensity): 262.2 (M^+ , 3%), 244.6 (15), 177.7 (15), 176.7 (100), 123.8 (22).

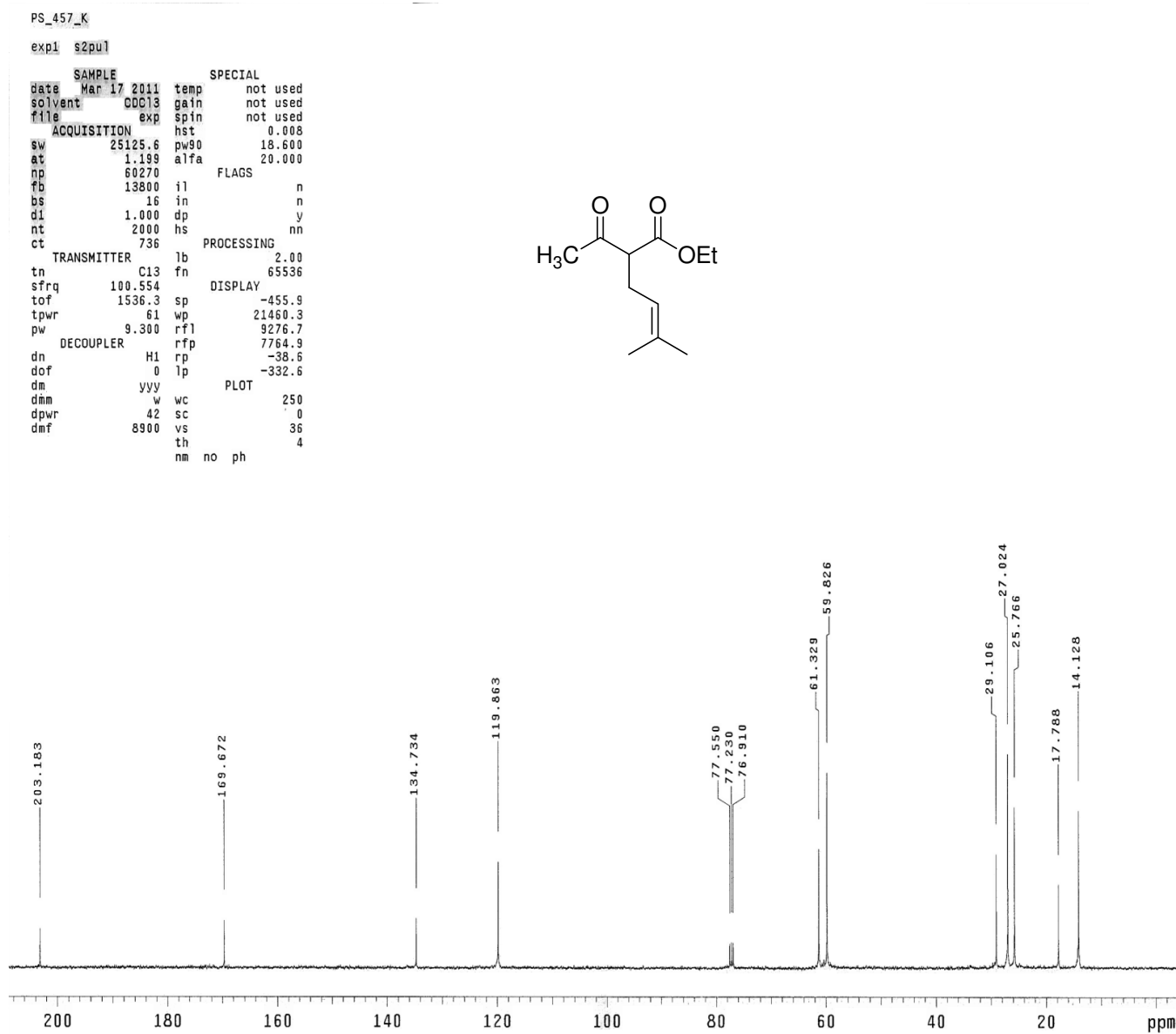
***syn*-Ethyl 2-(hydroxy(phenyl)methyl)-5-methylhex-4-enoate (1d)**: Colourless oil (1.58 g, 40%); ^1H NMR (400 MHz, CDCl_3): δ 1.11 (t, $J = 7.2$ Hz, 3 H), 1.54 (s, 3 H), 1.64 (s, 3 H), 2.29 (ddd, $J = 14.4$, 8.8 and 5.8 Hz, 1 H), 2.46 (ddd, $J = 14.4$, 8.8 and 5.6 Hz, 1 H), 2.72 (ddd, $J = 10.4$, 5.6 and 4.4 Hz, 1 H), 3.03 (brs, 1H), 4.02 (q, $J = 7.2$ Hz, 2 H), 4.95 (d, $J = 5.6$ Hz, 1H), 5.04 (t, $J = 6.8$ Hz, 1H), 7.24-7.39 (m, 5H); ^{13}C NMR (100 MHz, CDCl_3): δ 14.2, 17.8, 25.9, 26.1, 53.3, 60.7, 74.2, 121.1, 126.4, 127.8, 128.4, 134.0, 141.7, 175.0; IR (KBr, Neat): 3460, 2979, 2930, 1728, 1453, 1375, 1180, 1026, 767, 701 cm^{-1} . HRMS (APCI) cald. for $\text{C}_{16}\text{H}_{22}\text{O}_3$ ($\text{M}+\text{H}^+$) requires 263.1647; found 263.1652. APCI-MS: m/z (relative intensity): 263.2 ($(\text{M}+\text{H})^+$, 19%), 192.1 (5), 178.1 (15), 177.1 (100), 171.1 (21), 131.1 (13), 124.1 (75).

¹H NMR spectra of B-1

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¹³C NMR spectra of B-1

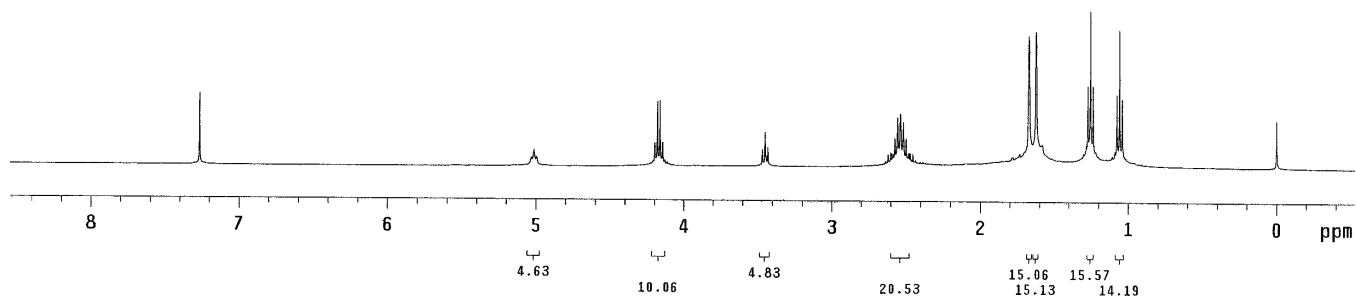
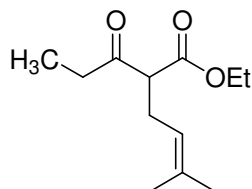


¹H NMR spectra of B-2

PS_449_K

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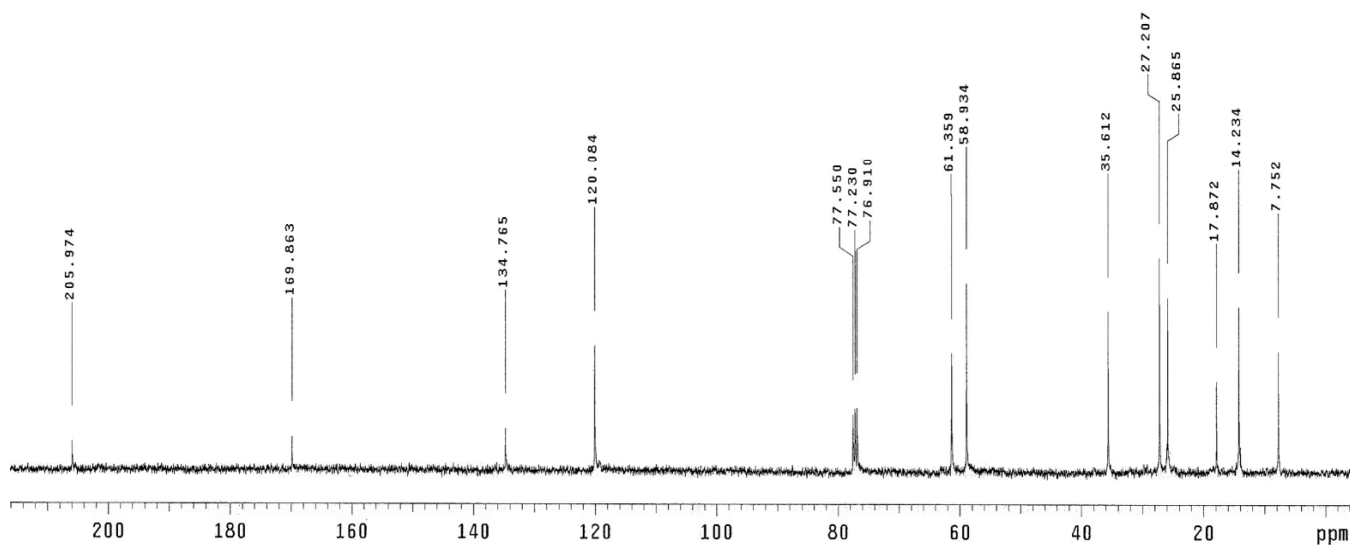
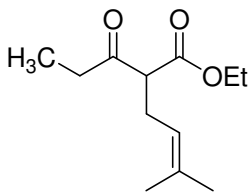


¹³C NMR spectra of B-2

PS_449_Ketone

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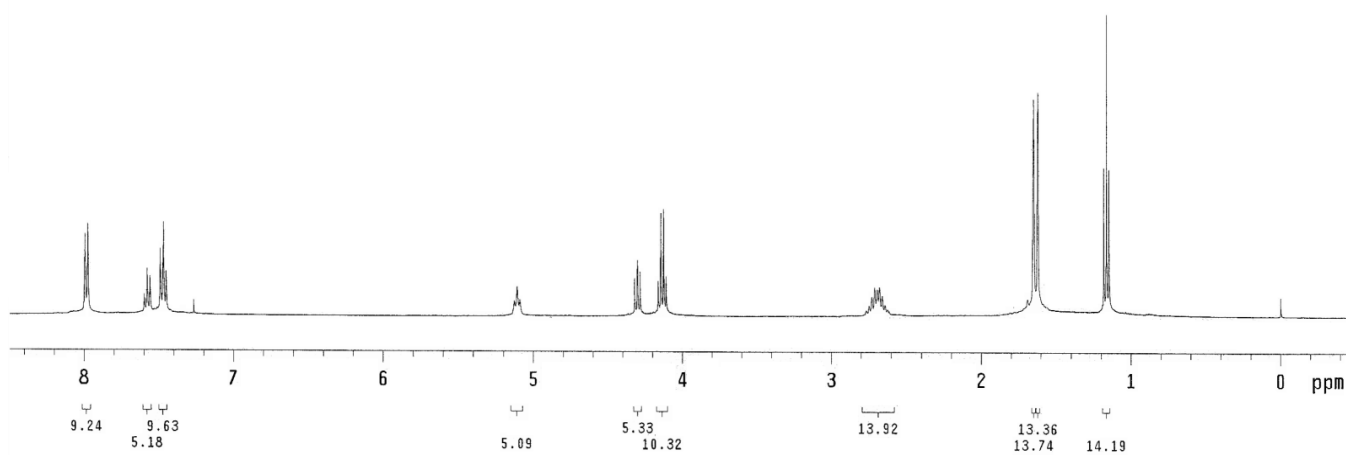
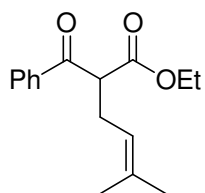


¹H NMR spectra of B-3

PS_447M_Ketone

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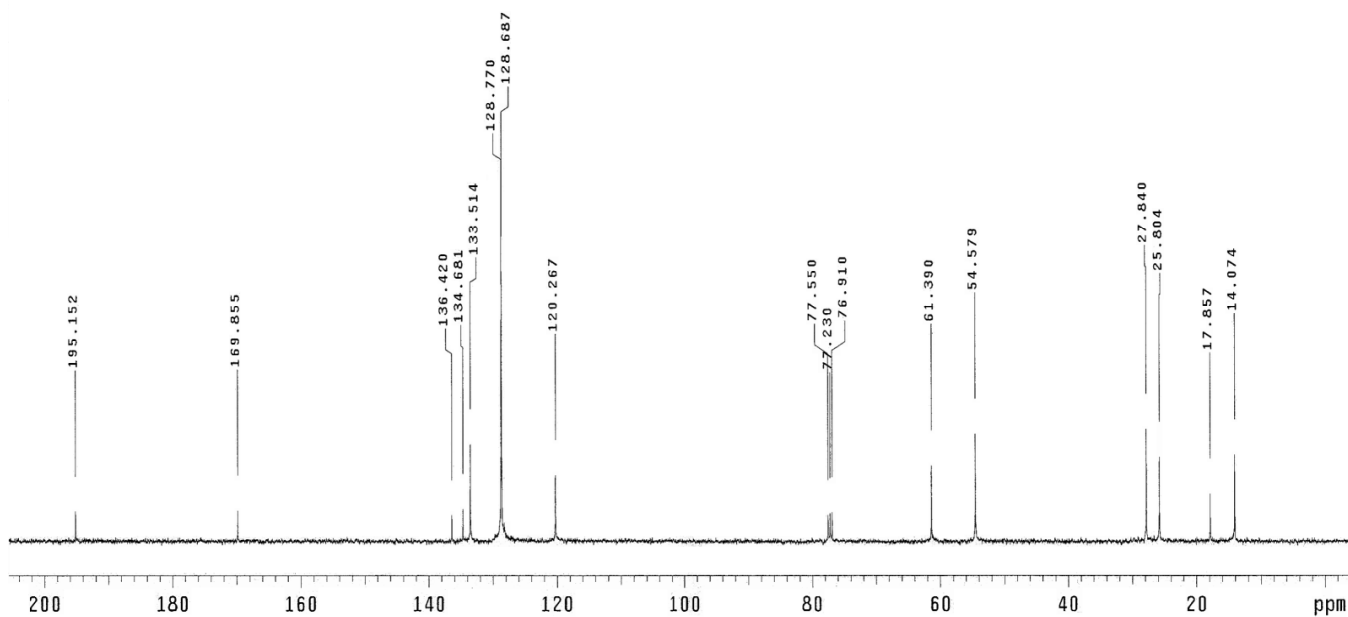
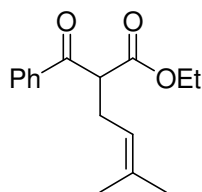
S-12

¹³C NMR spectra of B-3

PS_447M_Ketone

expt s2pu1

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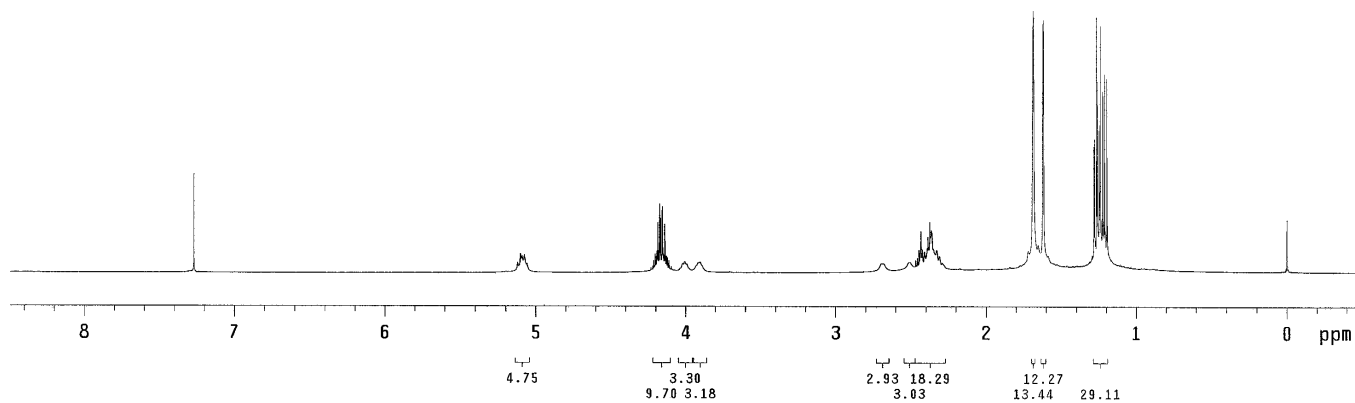
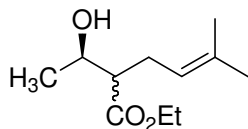


¹H NMR spectra of 1a

PS_457_L

expl std1h

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		nm	cdc ph

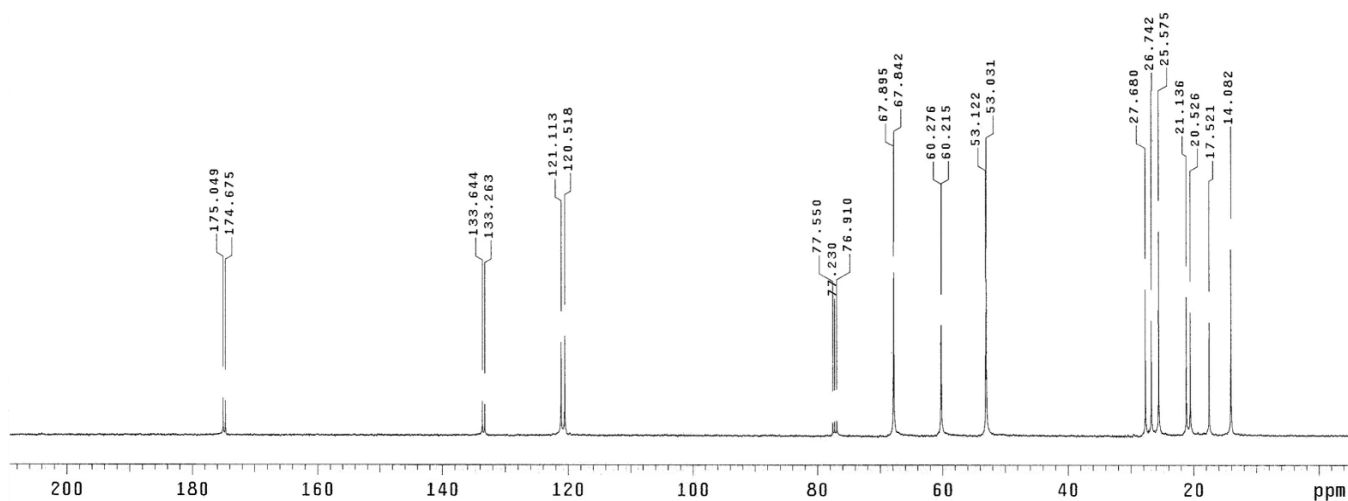
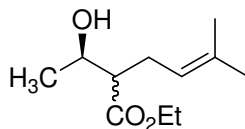


¹³C NMR spectra of 1a

PS_457A1c_L

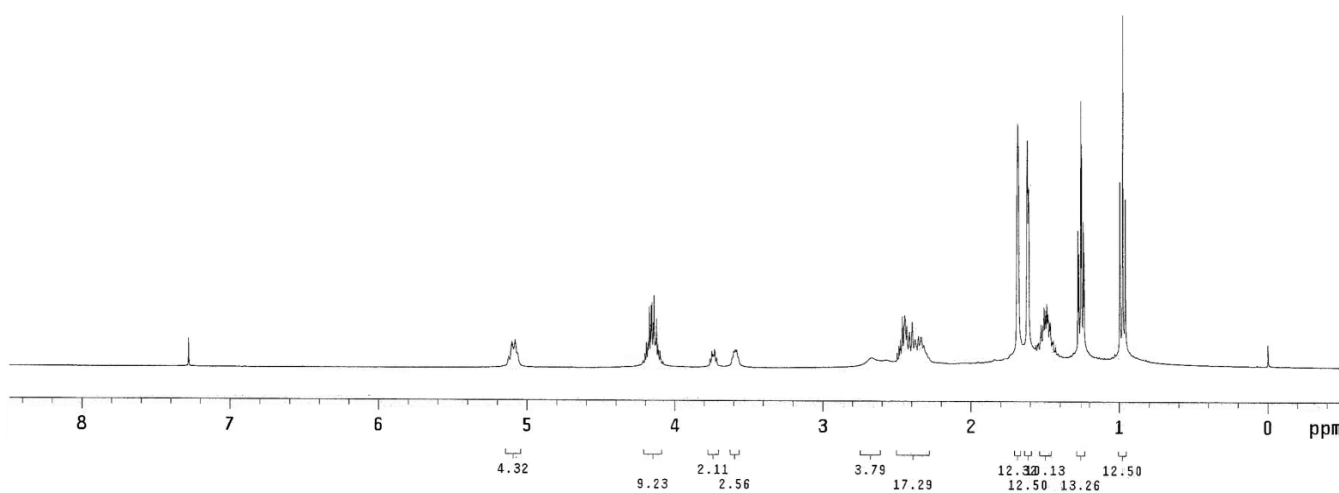
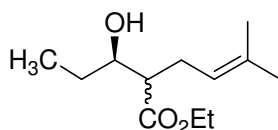
exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 18 2011	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	25125.6	ps0	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	fl	n
bs	16	in	n
d1	1.000	ds	y
nt	2000	hs	nn
ct	848	PROCESSING	
tn	TRANSMITTER	lb	2.00
sfrq	C13	fn	65536
tof	1536.3	sp	-531.1
tpwr	61	wp	21559.3
pw	9.300	rfl	9285.9
DECOUPLER		rfp	7764.9
dn	H1	rp	-42.2
dof	0	lp	-315.5
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	33
	th		1
	nm	no	ph



^1H NMR spectra of 1b

```
PS_449A1c_L
exp1 s2pu1
SAMPLE          SPECIAL
date Feb 28 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_449~ hst 0.008
Alc_L.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8 FLAGS
at 1.998 il n
np 25528 in n
fb not used dp y
bs 8 hs nn
d1 1.000 PROCESSING
nt 100 lb 0.10
ct 100 fn 65536
TRANSMITTER H1 sp DISPLAY
tn H1 sp -215.5
sfrq 399.853 wp 3610.3
tof 362.8 rfl 786.3
tpwr 57 rfp 0
pw 9.850 rp 107.1
DECOUPLER C13 lp -74.2
dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 57
dpwr 50 th 20
dmf 15900 nm cdc ph
```

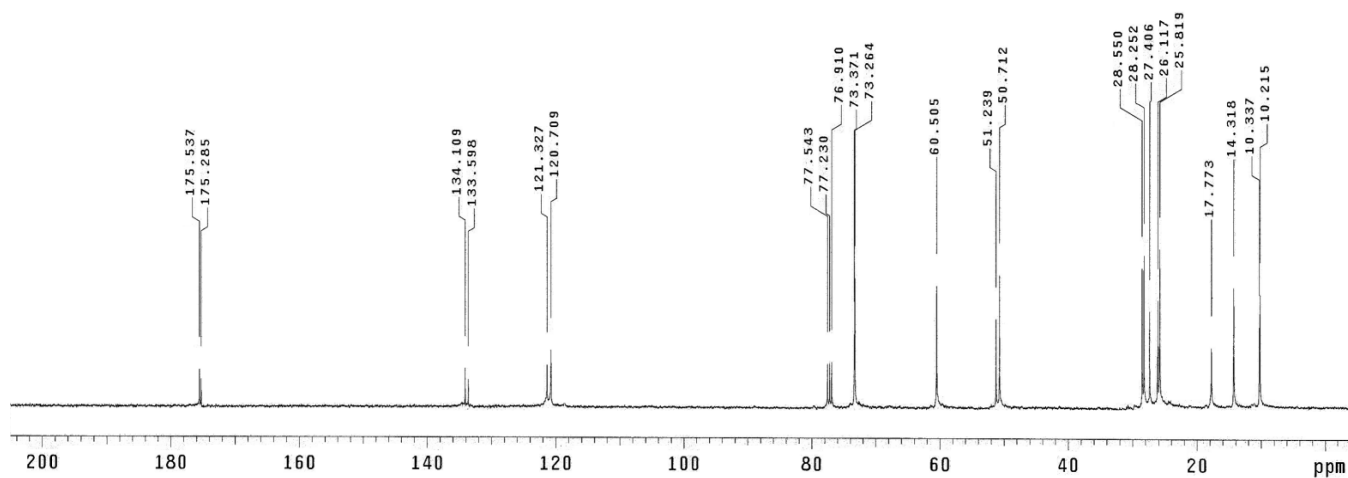
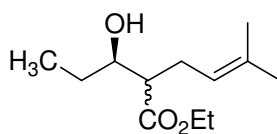


¹³C NMR spectra of 1b

PS_449_L

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 25 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_449	hst		0.008
L_C13.fid	pw90		18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	v
bs	16	hs	nn
dl	1.000	PROCESSING	
nt	4000	lb	2.00
ct	3232	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-422.2
sfrq	100.554	wp	21030.9
tof	1536.3	rfl	9275.9
tpwr	61	rfp	7764.9
pw	9.300	rp	-62.6
DECOUPLER		lp	-285.1
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	25
dpwr	42	th	3
dmf	8900	nm	no
		ph	

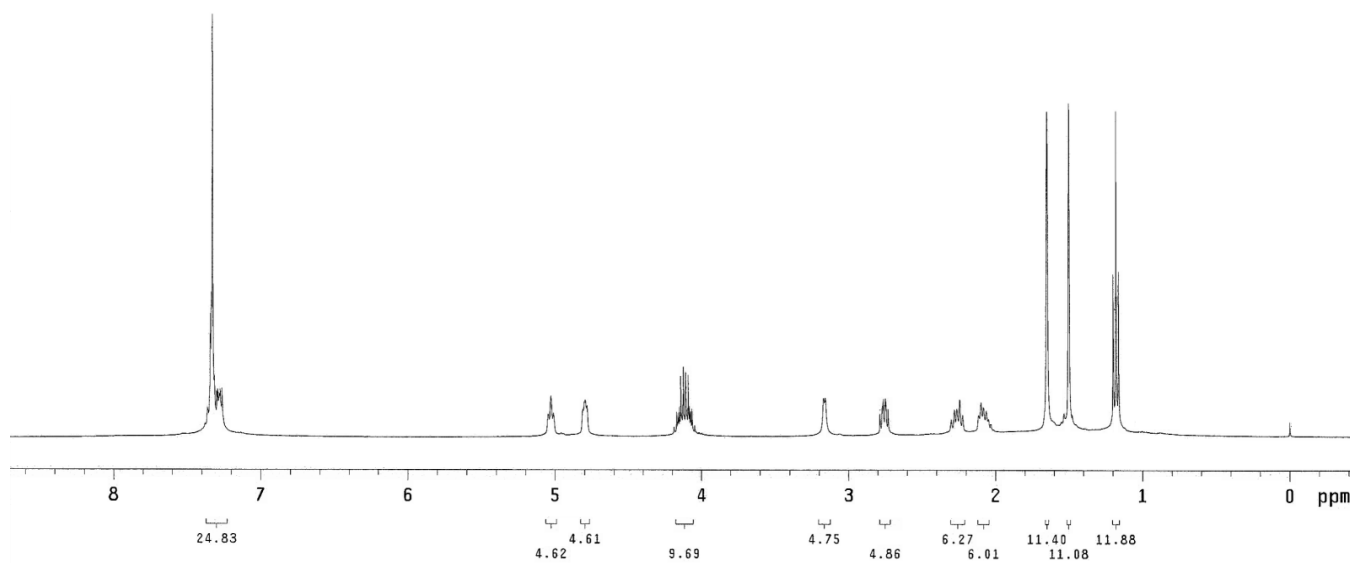
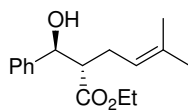


¹H NMR spectra of 1c

PS_447_L

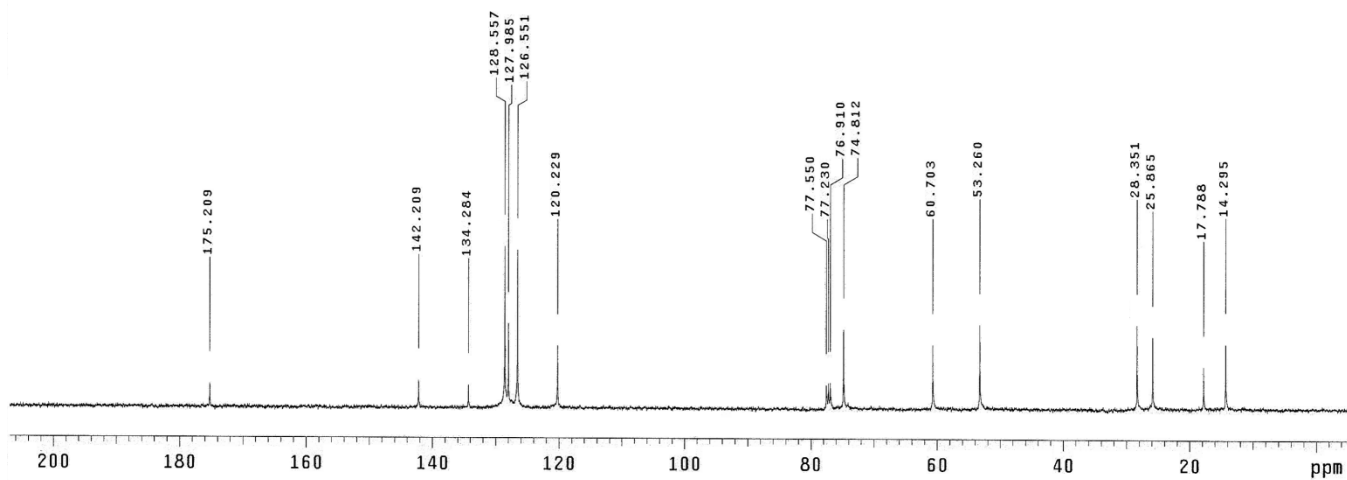
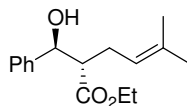
expl s2pu1

SAMPLE		SPECIAL	
date	Feb 19 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_447~	hst	0.008
	_L.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-180.6
sfrq	399.853	wp	3660.9
tof	362.8	rfl	793.5
tpwr	57	rffp	0
pw	9.850	rp	114.9
		lp	-93.0
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	68
dpwr	50	th	20
dmf	15900	nm	cdc ph



¹³C NMR spectra of 1c

```
PS_447_L
exp1 s2pu1
SAMPLE
date Feb 19 2011 temp not used
solvent CDC13 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_447~ hst 0.008
L_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6
at 1.199 f1 FLAGS n
np 68270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 2000 lb 2.00
ct 0 fn 65536
TRANSMITTER C13 DISPLAY -521.9
tn 100.554 wp 21328.5
sfrq 1536.3 rfl 9276.7
tpwr 61 rfp 7764.9
pw 9.300 rp -63.7
DECOUPLER lp -312.0
dn H1 PLOT
dof 0 wc 250
dm yvy sc 0
dmm w vs 26
dpwr 42 th
dmf 8900 nm no ph 2
```

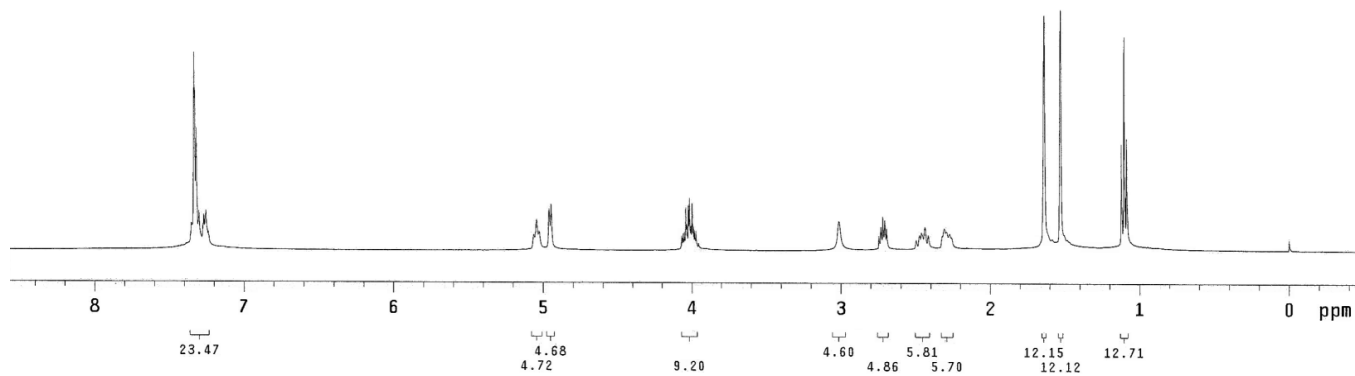
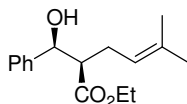


¹H NMR spectra of 1d

PS_447_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 18 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_447~		hst	0.008
	Mi.fid	pw90	19.700
	alfa		20.000
ACQUISITION		FLAGS	
sw	6309.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-183.3
sfrq	399.853	wp	3610.3
tof	362.8	rfl	796.2
tpwr	57	rfp	0
pw	9.850	rp	110.6
	DECOUPLER	lp	-84.9
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	39
dpwr	50	th	20
dmf	15900	nm	cdc ph

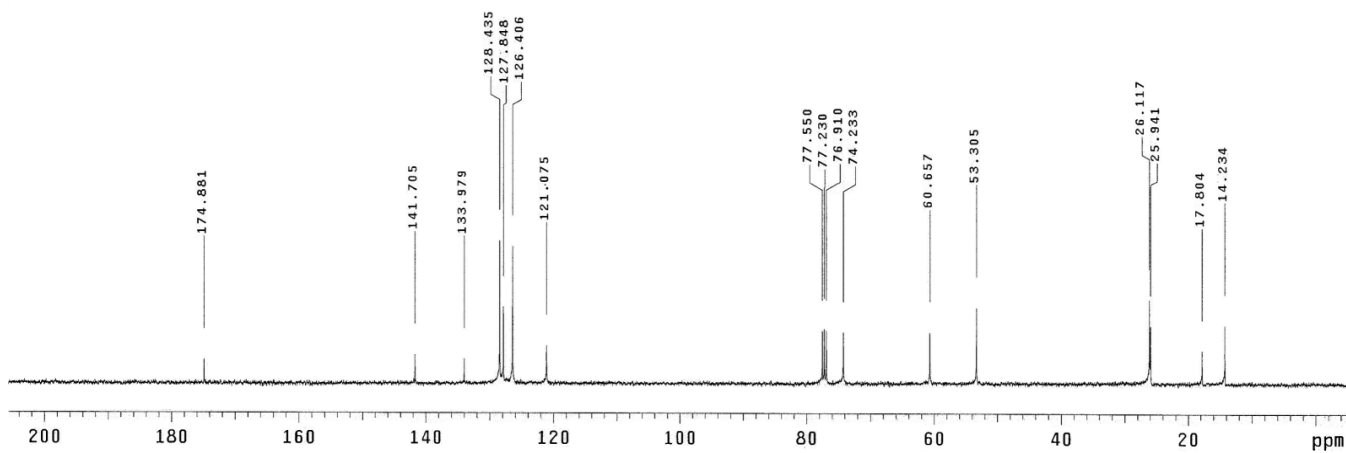
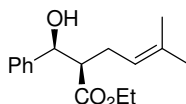


¹³C NMR spectra of 1d

PS_447_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 18 2011	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_447~	hst	0.008
	M_C13.fid	pw90	18.600
		alfa	20.000
ACQUISITION		FLAGS	
sw	25125.6		
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
dl	1.000	PROCESSING	
nt	8000	lb	2.00
ct	1504	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-551.8
sfrq	100.624	wp	21229.5
tof	1536.3	rfl	9272.9
tpwr	61	rfp	7764.9
pw	9.300	rp	-26.3
	DECOUPLER	rp	-358.9
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	23
dpwr	42	th	2
dmf	8900	nm	no ph

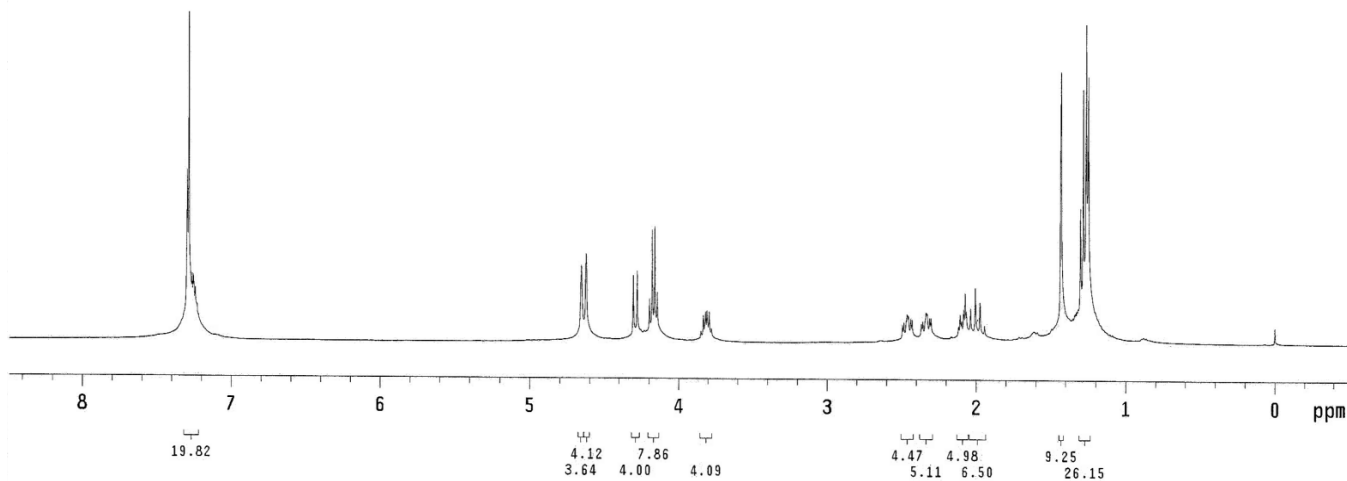
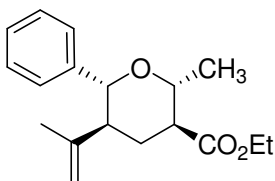


¹H NMR spectra of 3a

PS_392_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 26 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_392~		hst	0.008
	U.fid	pw90	19.700
	alfa	alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8	il	n
at	1.998	in	n
np	25528	dp	y
fb	not used	hs	nn
bs	4	PROCESSING	
d1	1.000	lb	0.10
nt	32	fn	65536
ct	32	DISPLAY	
TRANSMITTER		tn	H1 sp -208.5
tn	H1	sfrq	399.853 wp 3602.0
sfrq	399.853	tof	362.8 rfl 796.2
tof	362.8	tpwr	57 rfp 0
tpwr	57	pw	9.850 rp 106.1
pw	9.850	DECOUPLER	lp -85.7
DECOUPLER		PLOT	
dn	C13	dof	0 wc 250
dof	0	dm	nnn sc 0
dm	nnn	dmm	c vs 48
dmm	c	dpwr	50 th 39
dpwr	50	dmf	15900 nm cdc ph
dmf	15900		

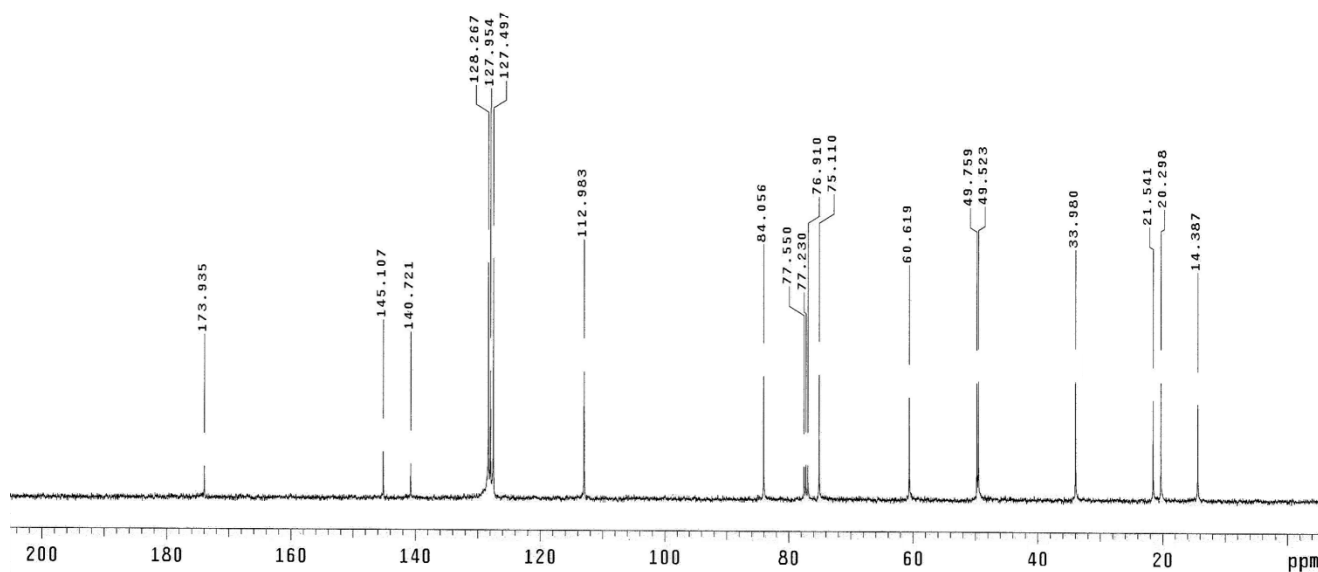
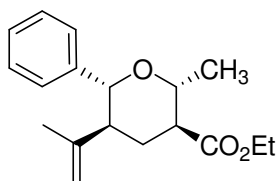


¹³C NMR spectra of 3a

PS_392_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Nov 26 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_392~	hst	0.008
U_C13.fid		pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	3000	lb	2.00
ct	528	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-620.8
sfrq	100.554	wp	21262.5
tof	1536.3	rfl	9276.7
tpwr	61	rff	7764.9
pw	9.300	rp	-52.1
DECOUPLER		lp	-305.8
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	36
dpwr	42	th	3
dmf	8900	nm	no ph

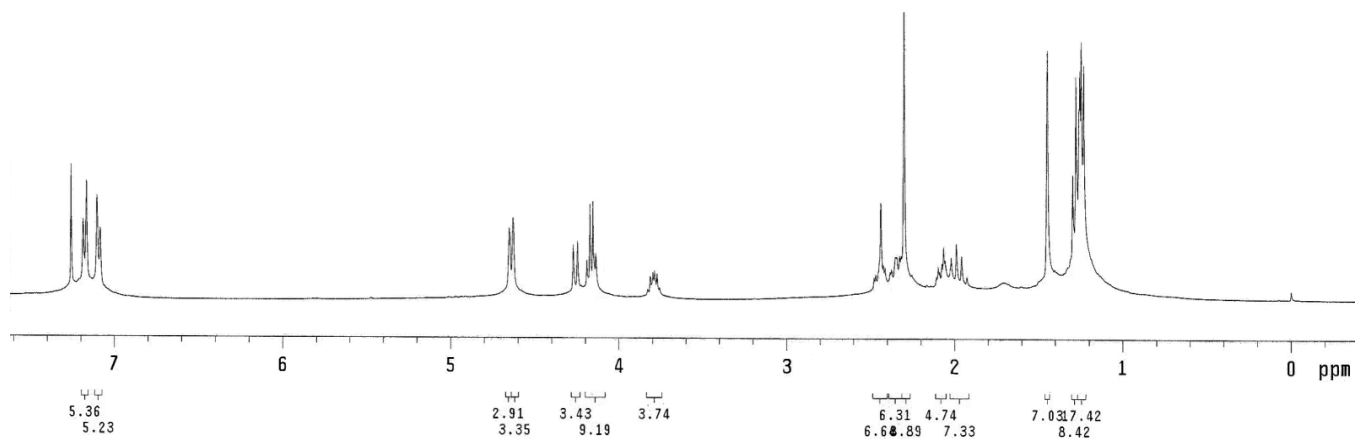
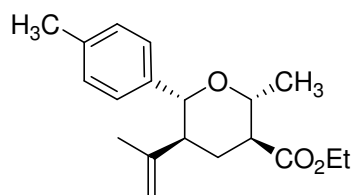


¹H NMR spectra of 3b

PS_343

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 2 2010	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	spin	not used
guest123/AKS_PS_34~		hst	0.008
3_U.fid		pw90	19.700
ACQUISITION		alfa	20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-157.4
sfrq	399.853	wp	3207.5
tof	362.8	rfl	795.4
tpwr	57	rfp	0
pw	9.850	rp	118.1
DECOUPLER		lp	-67.4
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	43
dpwr	50	th	39
dmf	15900	nm	cdc ph

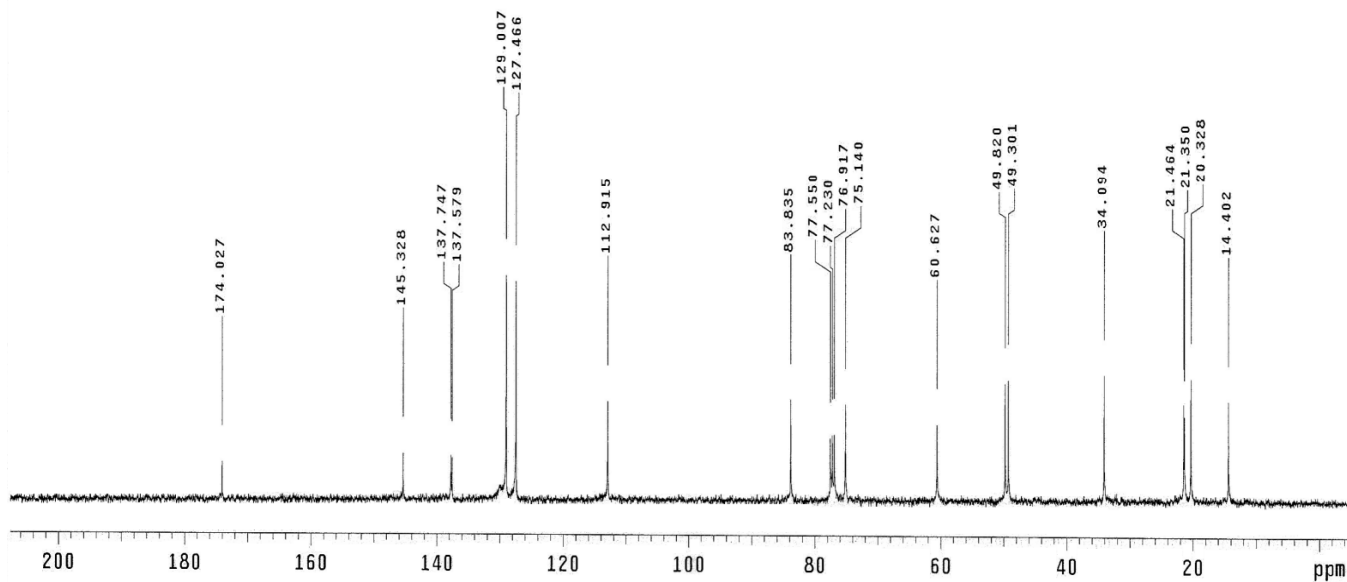
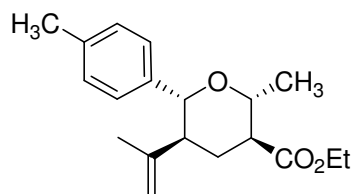


¹³C NMR spectra of 3b

PS_343_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 3 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
guest123/AKS_PS_34		hst	0.008
3U_C13.fid		pw90	18.600
ACQUISITION		alfa	20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	4000	lb	2.00
ct	1632	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-518.0
sfrq	100.554	wp	21394.4
tof	1536.3	rfl	9272.9
tpwr	61	rfp	7764.9
pw	9.300	rp	-52.1
DECOUPLER		lp	-305.8
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	31
dpwr	42	th	4
dmt	8900	nm	no ph

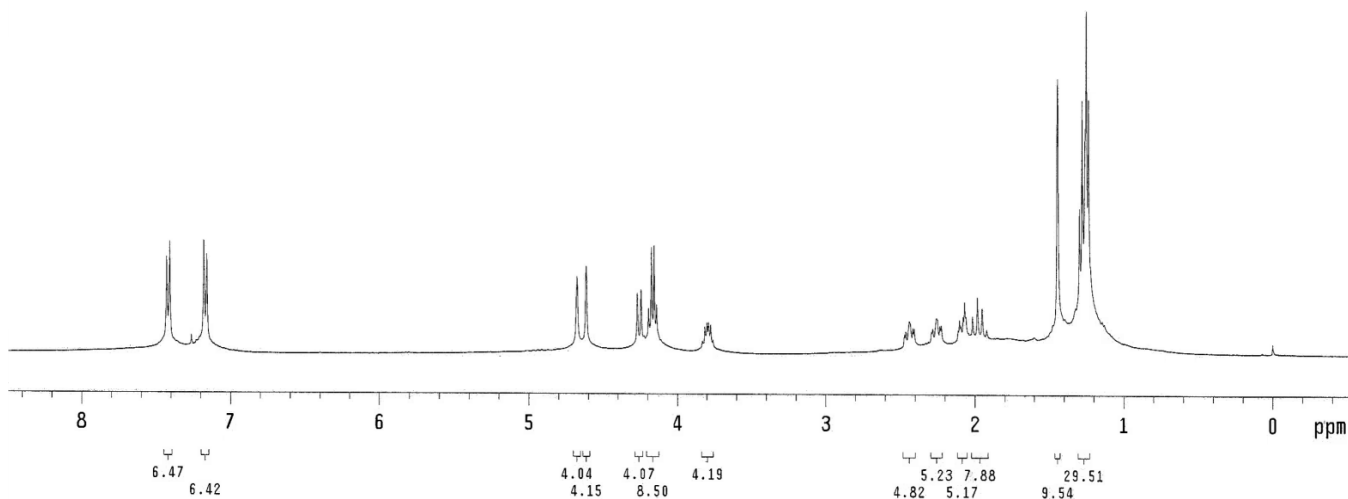
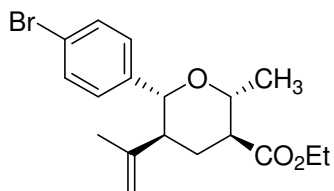


¹H NMR spectra of 3c

PS_341_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 1 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
guest123/AKS_PS_34~		hst	0.008
1_U.fid		pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-214.5
sfrq	399.853	wp	3610.3
tof	362.8	rfl	793.7
tpwr	57	rfp	0
pw	9.850	rp	120.1
		lp	-69.1
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	48
dpwr	50	th	30
dmf	15900	nm	cdc ph

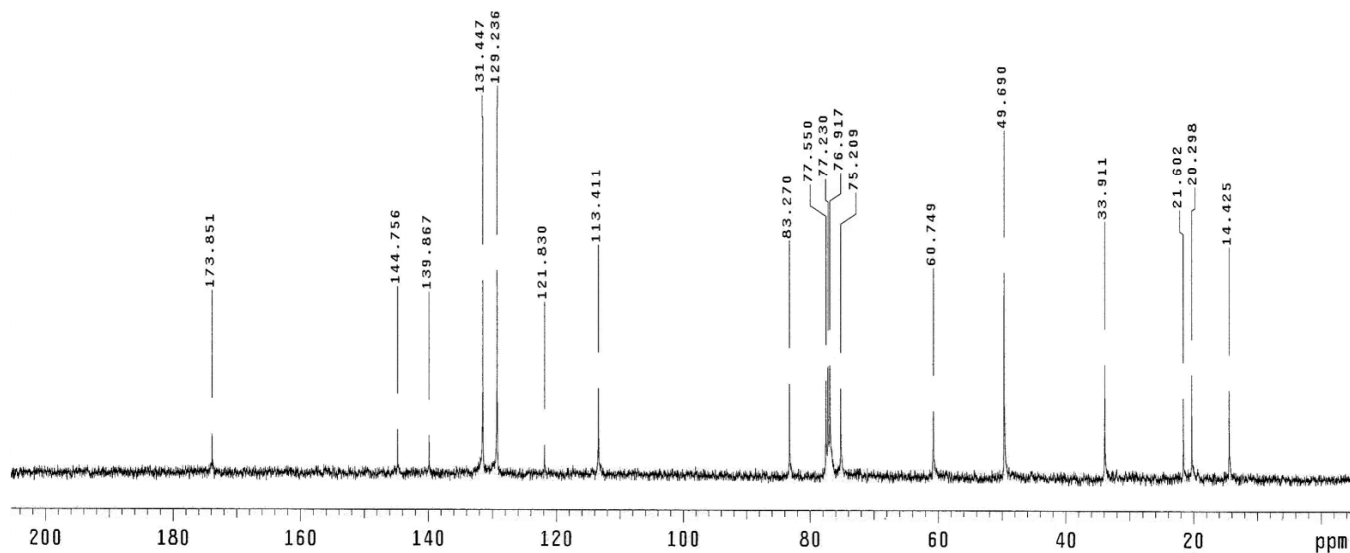
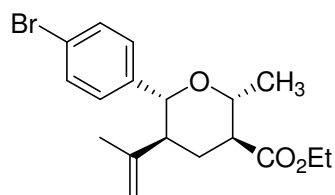


^{13}C NMR spectra of 3c

PS_341_U

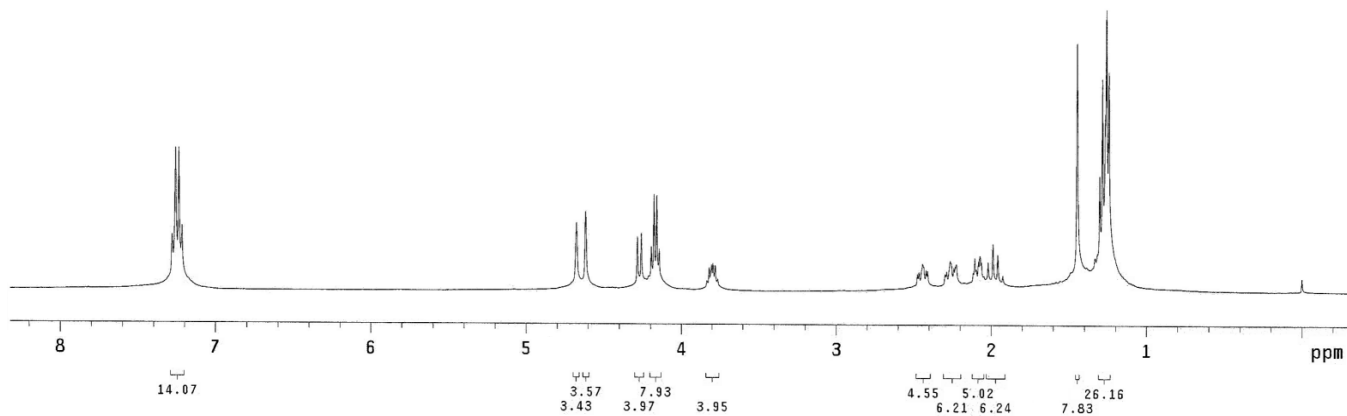
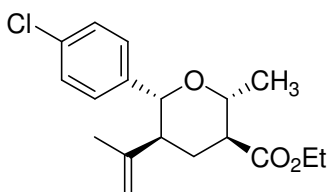
exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 1 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
guest123/AKS_PS_34~		hst	0.008
1M_C13.fid		pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	4000	lb	2.00
ct	3024	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-514.2
sfrq	100.554	wp	21163.6
tof	1536.3	rf1	9269.0
tpwr	61	rfp	7764.9
pw	9.300	rp	-67.1
DECOUPLER		lp	-288.0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	29
dpwr	42	th	4
dmf	8900	nm	no ph



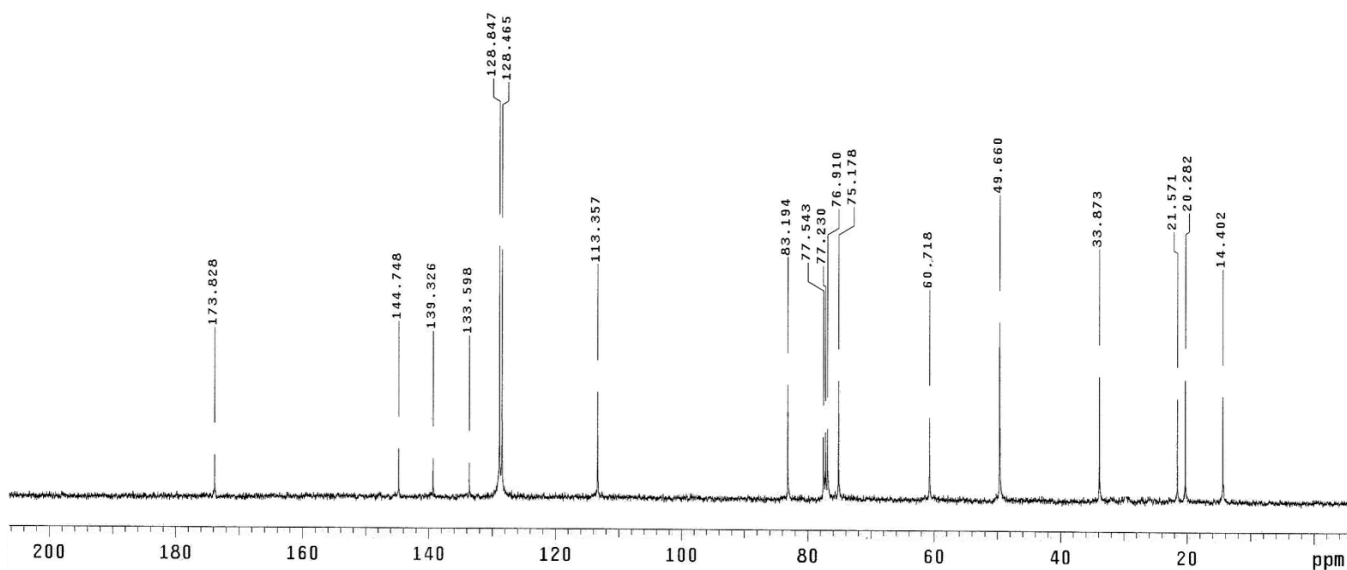
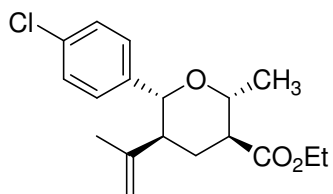
¹H NMR spectra of 3d

```
PS_395_U
exp1 s2pu1
SAMPLE
date Nov 29 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_395~ hst 0.008
U.fid pw90 19.700
alfa 20.000
ACQUISITION
sw 6389.8 FLAGS
at 1.998 il n
np 25528 in n
fb not used dp y
bs 4 hs nn
d1 1.000 PROCESSING
nt 32 lb 0.10
ct 32 fn 65536
TRANSMITTER
tn H1 sp -122.7
sfrq 399.853 wp 3450.8
tof 362.8 rfl 794.3
tpwr 57 rfp 0
pw 9.850 lp 113.5
DECOUPLER C13 PLOT
dn 0 wc 250
dm nnn sc 0
dmm c vs 46
dpwr 50 th 37
dmf 15900 nm cdc ph
```



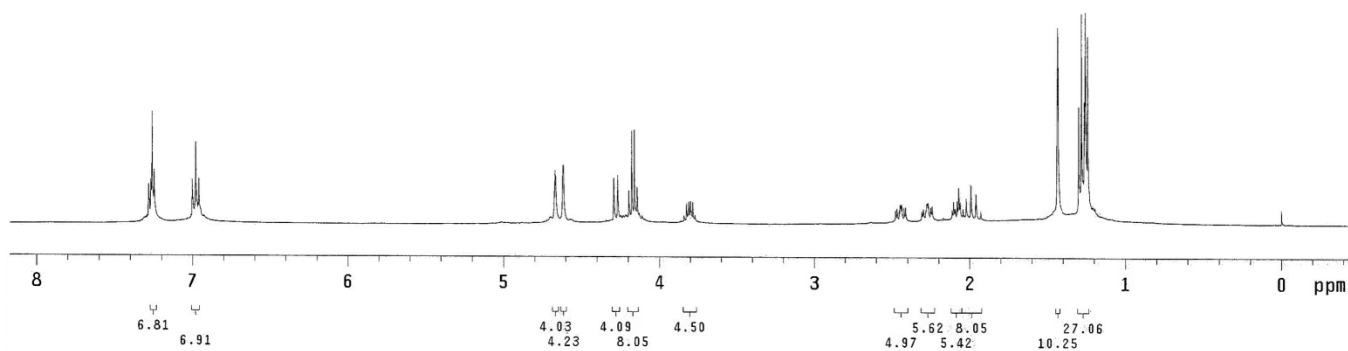
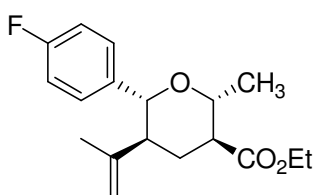
¹³C NMR spectra of 3d

```
PS_395_U
exp1 s2pu1
SAMPLE
date Nov 29 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_395~ hst 0.008
U_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6
at 1.199 fl
np 60270 in
fb 13800 dp
bs 16 hs
d1 1.000
nt 4000 lb PROCESSING
ct 1520 fn 65536
TRANSMITTER C13 sp DISPLAY
tn C13 sp -584.0
sfrq 100.554 wp 21328.5
tof 1536.3 rfl 9272.9
tpwr 61 rfp 7764.9
pw 9.300 rp -47.2
DECOUPLER lp -326.0
dn H1
dof 0 wc PLOT
dm yyy sc 250
dmm w vs 39
dpwr 42 th
dmf 8900 nm no ph 5
```



^1H NMR spectra of 3e

```
PS_396_U
exp1 s2pu1
SAMPLE
date Dec 1 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_396~ hst 0.008
_U.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8
at 1.998 il
np 25528 in
fb not used dp
bs 4 hs
d1 1.000
nt 32 lb
ct 32 fn
TRANSMITTER DISPLAY
tn H1 sp -182.9
sfrq 399.853 wp 3450.8
tof 362.8 rfl 795.8
tpwr 57 rfp 0
pw 9.850 rp 109.5
DECOUPLER lp -107.9
dn C13
dof 0 wc
dm nnn sc
dmm c vs
dpwr 50 th
dmf 15900 nm cdc ph
```

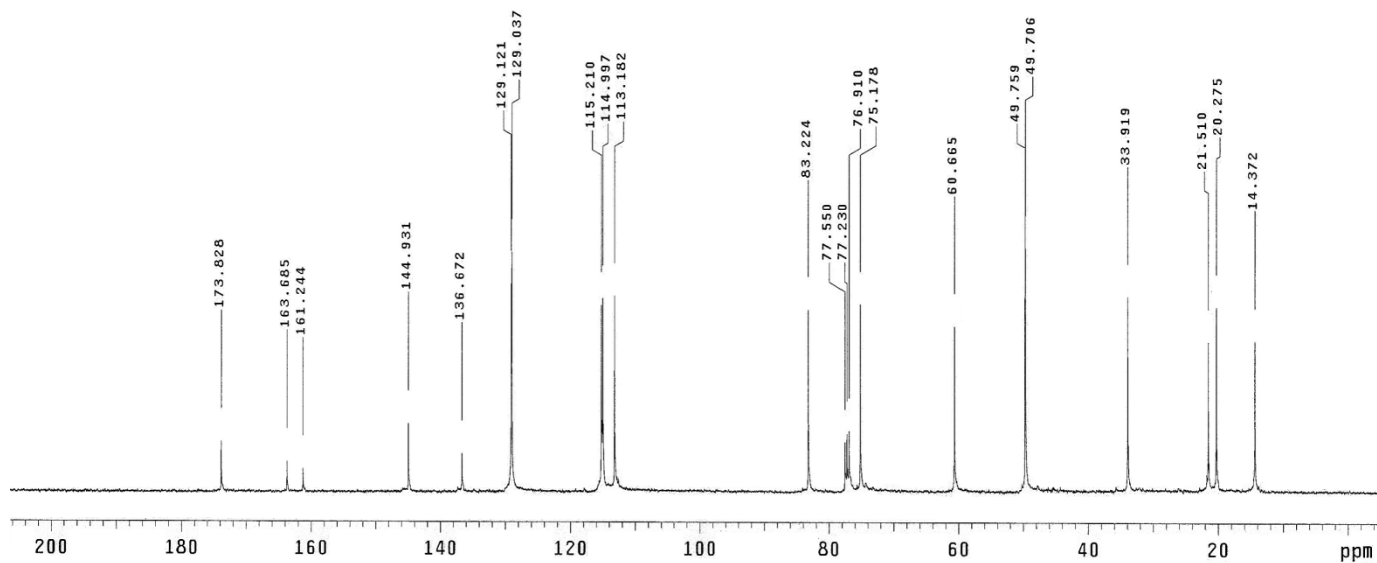
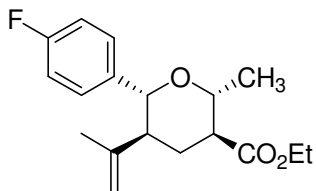


¹³C NMR spectra of 3e

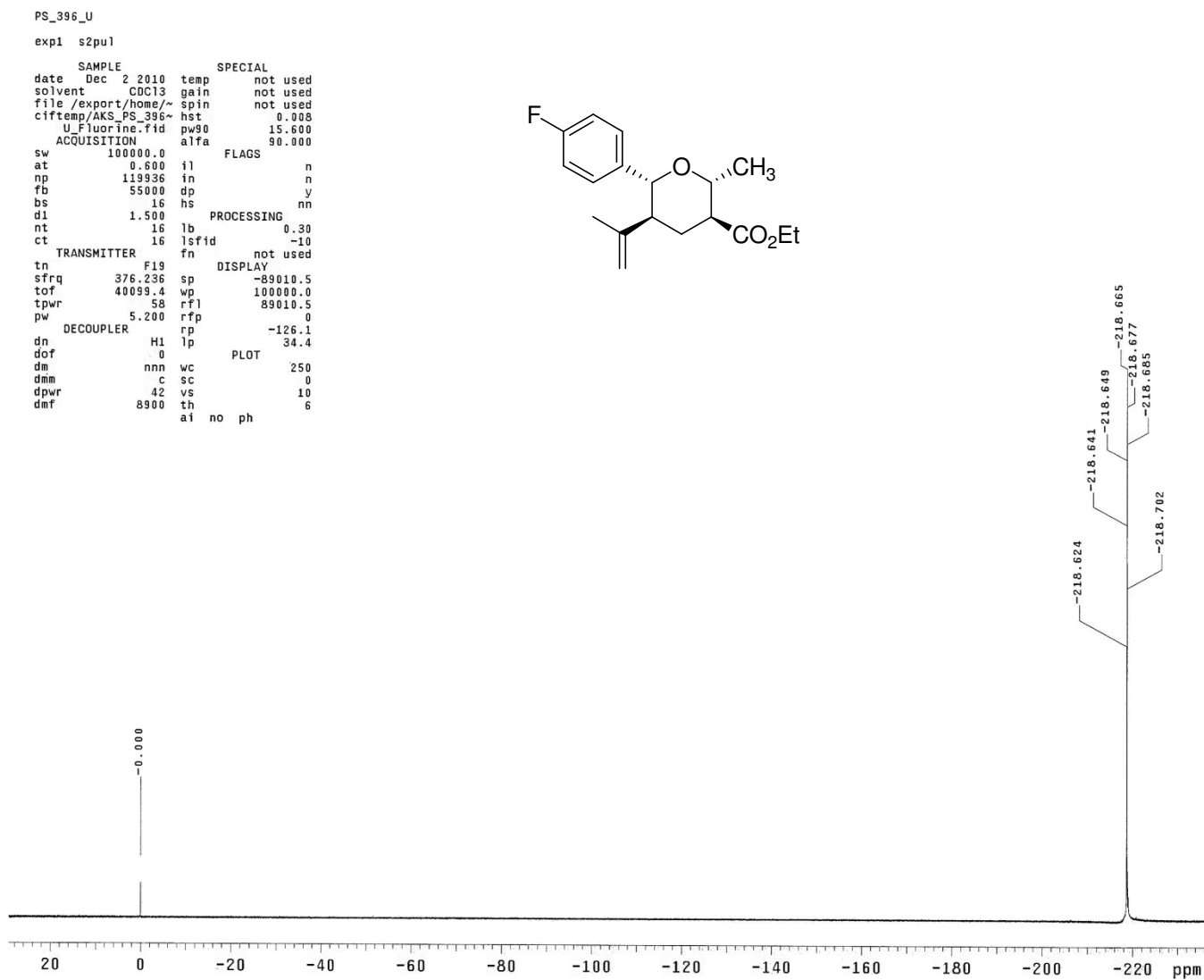
PS_396_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Dec 1 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_396~	hst	0.008
U	C13:fid	pw90	18.600
		alfa	20.000
ACQUISITION			
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	10000	lb	2.00
ct	10000	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-485.8
sfrq	100.554	wp	21229.5
tof	1536.3	rfl	9273.6
tpwr	61	rfp	7764.9
pw	9.300	rp	-43.2
		lp	-343.0
DECOUPLER		PLOT	
dn	H1	wc	250
dof	0	sc	0
dm	yvy	vs	37
dmm	w	th	3
dpwr	42	nm	no
dmf	8900		ph

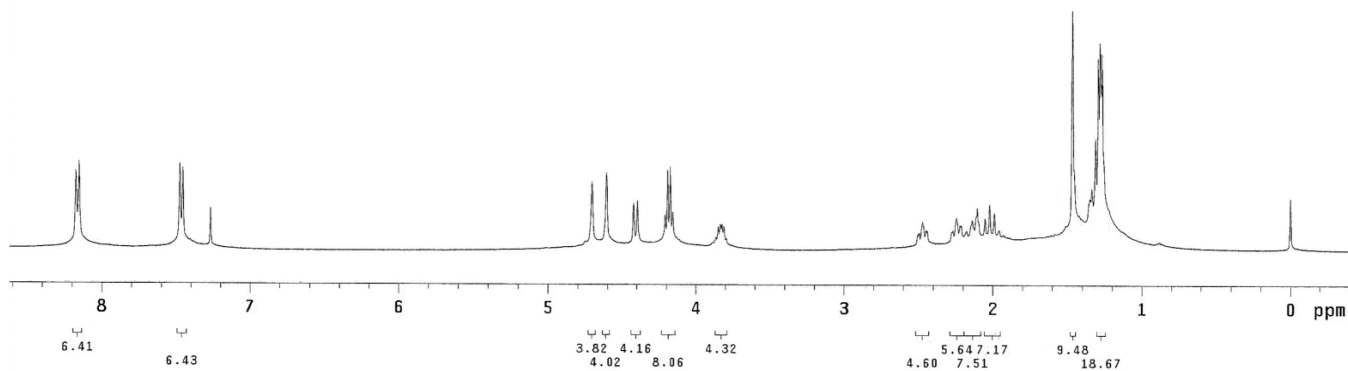
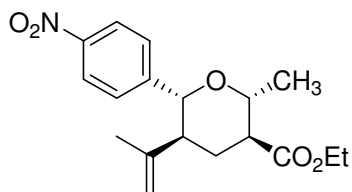


¹⁹F NMR spectra of 3e



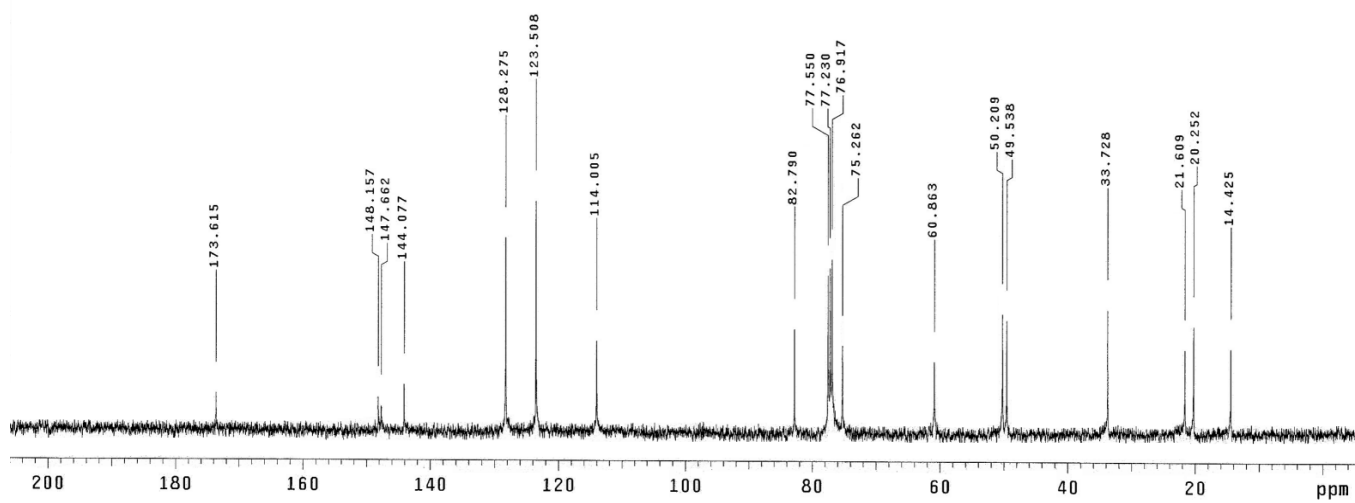
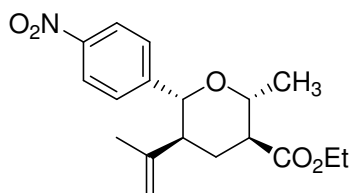
¹H NMR spectra of 3f

```
PS_391_U
exp1 std1h
SAMPLE          SPECIAL
date Nov 16 2010 temp not used
solvent CDC13 gain not used
file /export/home/ spin not used
ciftemp/AKS_PS_391~ hst 0.008
                _U.fid pw90 19.700
ACQUISITION    alfa 20.000
sw 6006.0
at 1.995 il FLAGS n
np 23984 in n
fb not used dp y
bs 16 hs nn
d1 1.000
nt 100 fn PROCESSING not used
ct 100 DISPLAY
tn TRANSMITTER H1 sp -167.2
sfrq 399.853 rf1 wp 3614.3
tof 0 rfp 0
tpwr 57 rp 110.1
pw 7.000 lp -77.9
DECOUPLER C13 wc PLOT 250
dof 0 sc 0
dm nnn vs 44
dmm c th 65
dpwr 50 nm cdc ph
dmf 15900
```



¹³C NMR spectra of 3f

```
PS_340_U
exp1 s2pu1
SAMPLE
date Aug 27 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
guest123/AKS_PS_34~ hst 0.008
0U_C13.F1d pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 f1 n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 4000 lb 2.00
ct 4000 fn 65536
TRANSMITTER C13 sp DISPLAY -513.4
sfrq 100.554 wp 21229.5
tof 1536.3 rfl 9268.3
tpwr 61 rfp 7764.9
pw 9.300 rp -18.6
DECOUPLER lp -390.3
dn H1 PLOT
dof 0 wc 250
dm yyy sc 0
dmm w vs 37
dpwr 42 th 3
dmf 8900 nm no ph
```

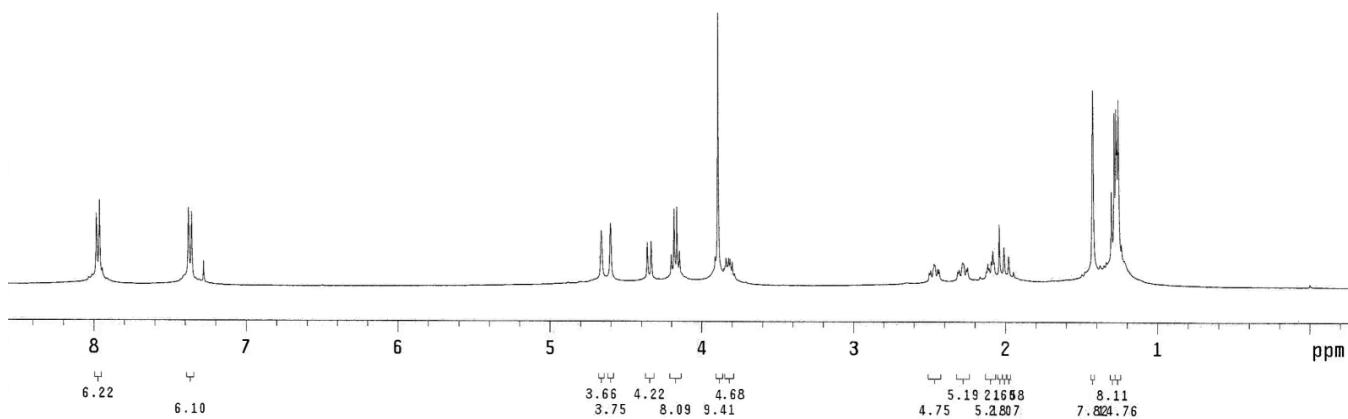
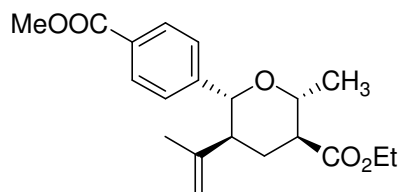


¹H NMR spectra of 3g

PS_338_U

exp1 s2pu1

```
SAMPLE          SPECIAL
date Aug 6 2010 temp      not used
solvent CDC13 gain      not used
file /export/home/~ spin  not used
guest123/AKS_PS_33~ hst   0.008
8_U.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8
at 1.998 il n
np 25528 in n
fb not used dp y
bs 4 hs nn
dl 1.000
nt 32 lb 0.10
ct 32 fn 65536
TRANSMITTER H1 sp DISPLAY -108.0
tn 399.853 wp 3531.4
sfrq 362.8 rfl 786.9
tof 57 rfp 0
tpwr 9.850 rp 125.8
pw DECOUPLER lp -81.9
dn C13
dof 0 wc PLOT 250
dm nnn sc 0
dmm c vs 42
dpwr 50 th 50
dmf 15900 nm cdc ph
```

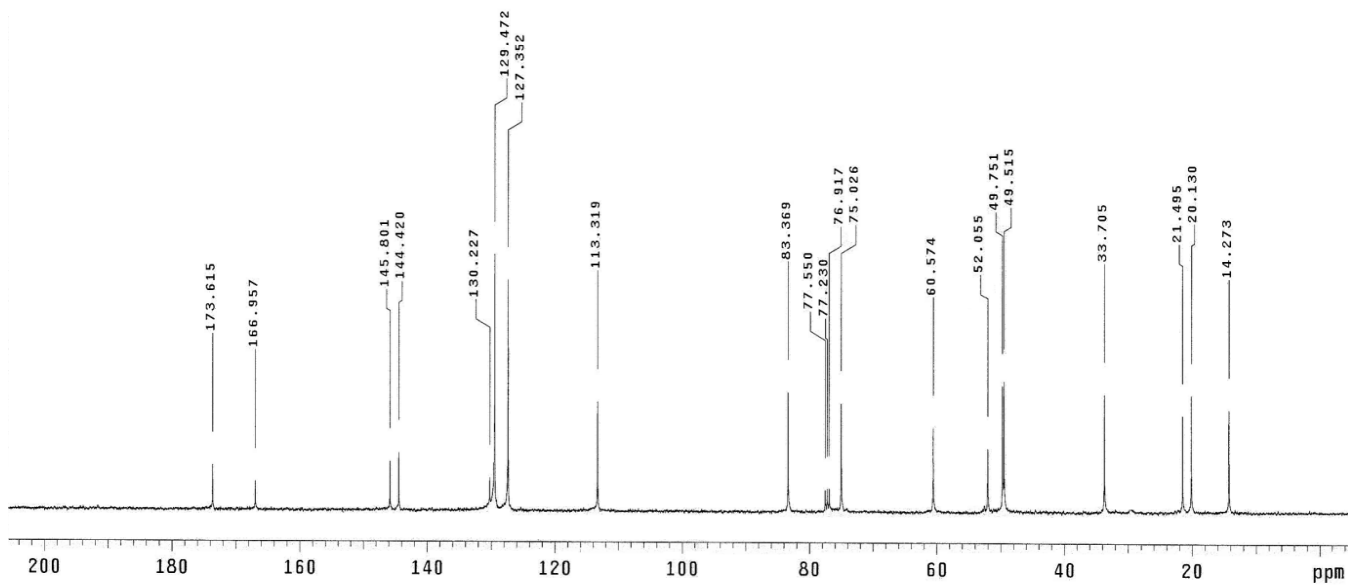
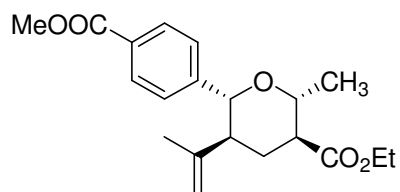


¹³C NMR spectra of 3g

PS_338_U

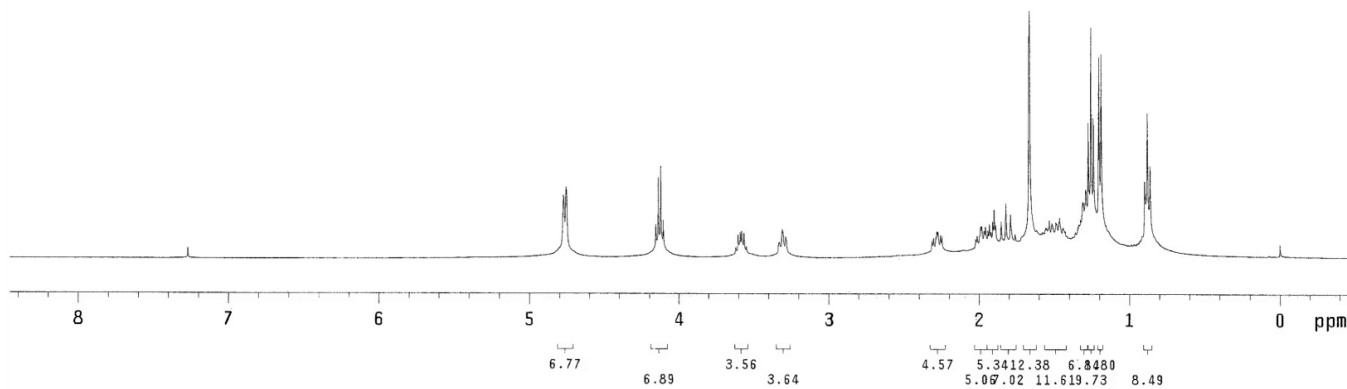
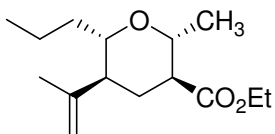
exp1 s2pu1

SAMPLE		SPECIAL	
date	Aug 6 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
guest123/AKS_PS_33~		hst	0.008
8U C13.Fid		pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	3000	lb	2.00
ct	992	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-492.7
sfrq	100.554	wp	21163.6
tof	1536.3	rfl	9280.5
tpwr	61	rfp	7764.9
pw	9.300	rp	-45.6
DECOUPLER		lp	-314.3
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	39
dpwr	42	th	3
dmf	8900	nm	no ph

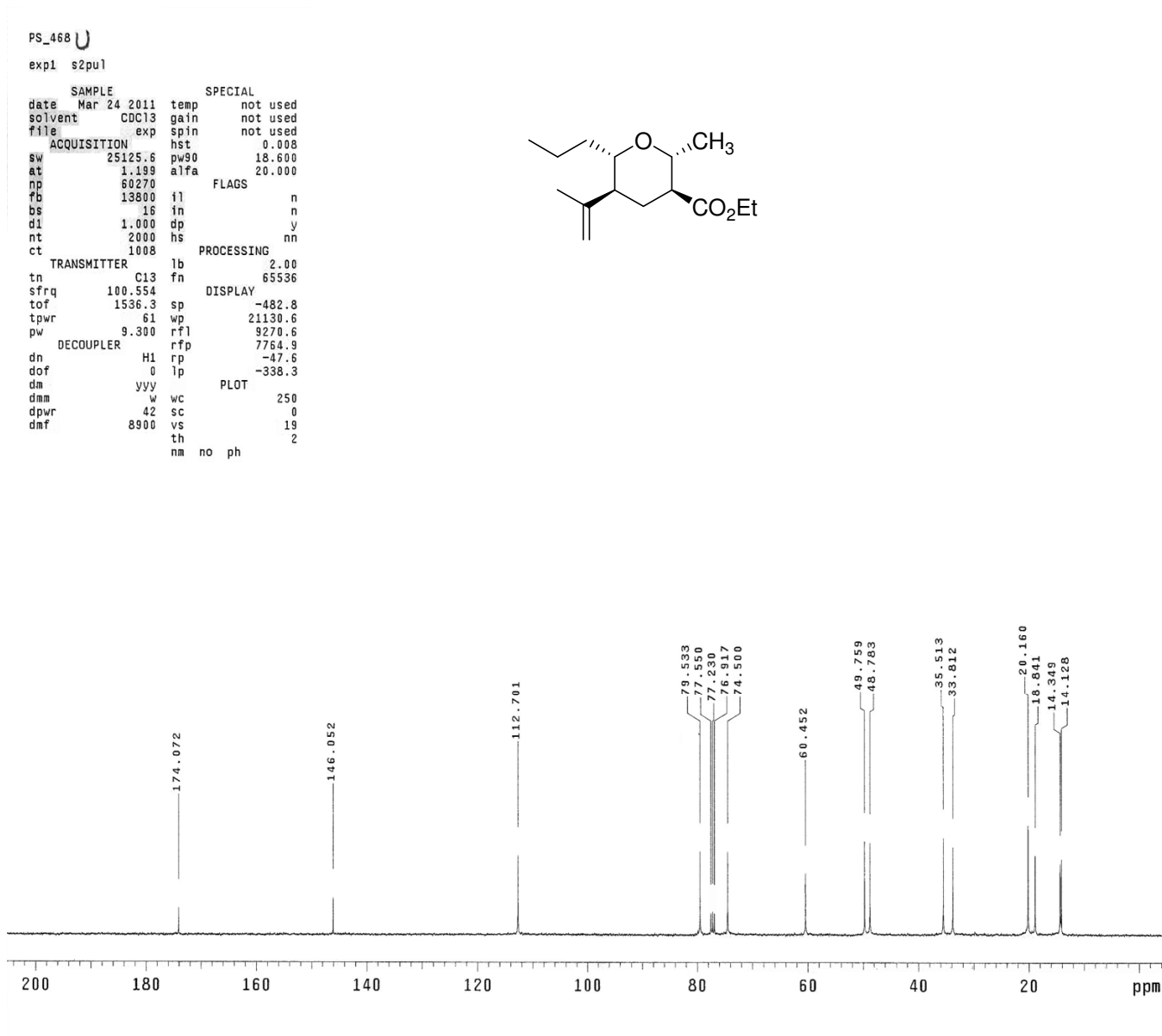


¹H NMR spectra of 3h

```
PS_468_U
exp1 s2pul
SAMPLE
date Mar 23 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION hst 0.008
sw 6389.8 pw90 19.700
at 1.998 alfa 20.000
np 25528
fb not used il n
bs 8 in n
d1 1.000 dp y
nt 100 hs nn
ct 100
PROCESSING
tn H1 lb 0.10
sfrq 399.853 fn 65536
tof 362.8 sp -194.2
tpwr 57 wp 3576.8
pw 9.850 rfl 790.4
DECOUPLER rfp 0
dn C13 rp 105.1
dof 0 lp -55.9
dm nnn
dmm c wc 250
dpwr 50 sc 0
dmf 15900 vs 40
nm cdc ph th 20
```



¹³C NMR spectra of 3h

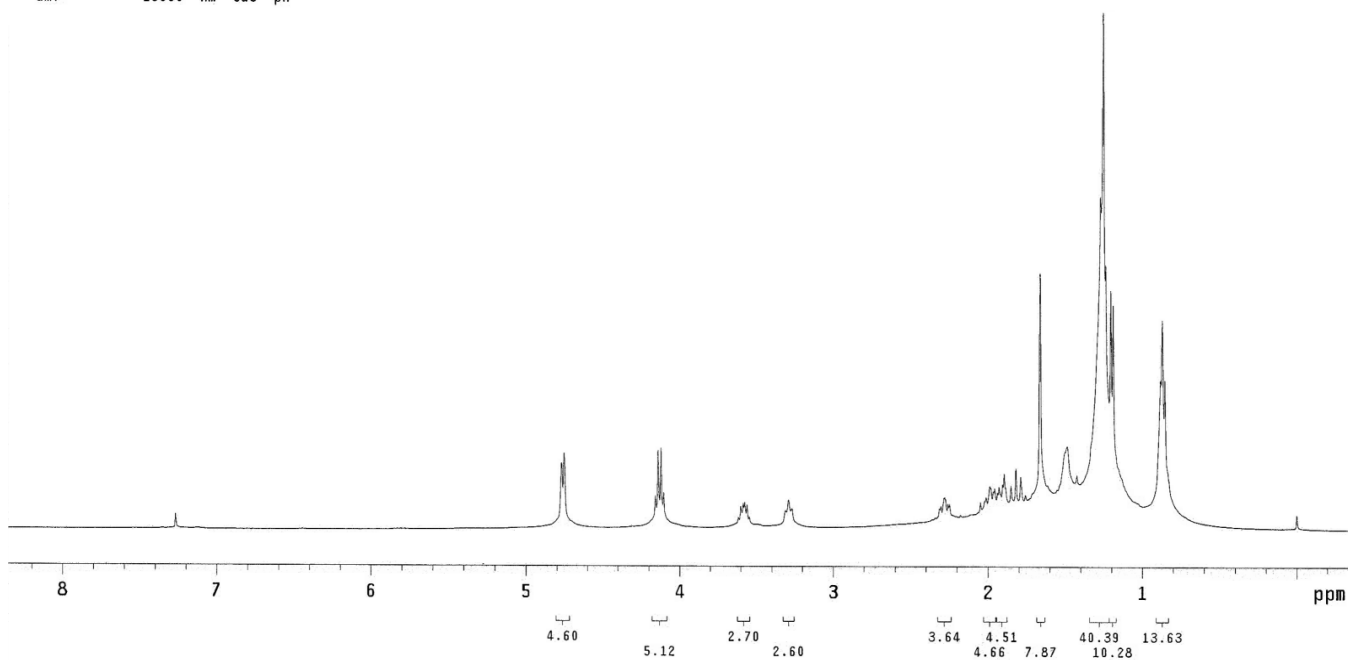
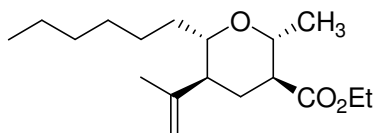


¹H NMR spectra of 3i

PS_415_U

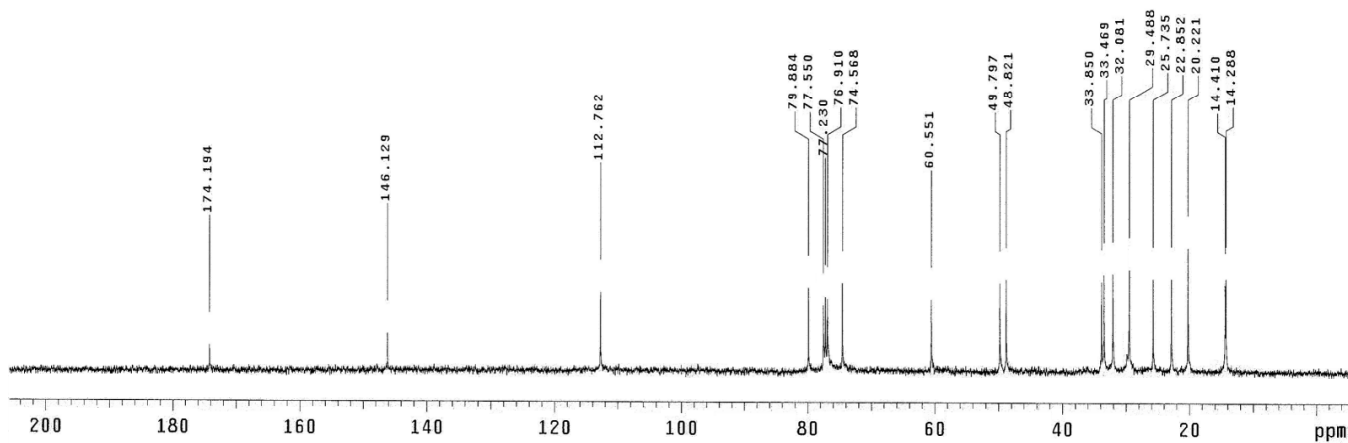
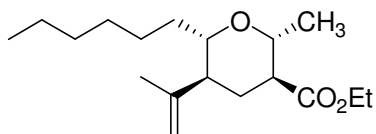
exp1 s2pul

date	Dec 27 2010	temp	not used	SPECIAL
solvent	CDC13	gain	not used	
file	/export/home/~	spin	not used	
ciftemp	/AKS_PS_415~	hst	0.008	
	_U1.fid	pw90	19.700	
ACQUISITION	alfa		20.000	
sw	6389.8	FLAGS		
at	1.998	il	n	
np	25528	in	n	
fb	not used	dp	y	
bs	4	hs	nn	
di	1.000	PROCESSING		
nt	32	lb	0.10	
ct	32	fn	65536	
TRANSMITTER	H1	sp	-144.3	DISPLAY
sfrq	399.853	wp	3484.6	
tof	362.8	rfl	790.8	
tpwr	57	rfp	0	
pw	9.850	rp	110.1	
DECOUPLER	C13	lp	-90.9	
dn	0	wc	250	PLOT
dof	nnn	sc	0	
dmm	c	vs	83	
dpwr	50	th	20	
dmf	15900	nm	cdc	ph



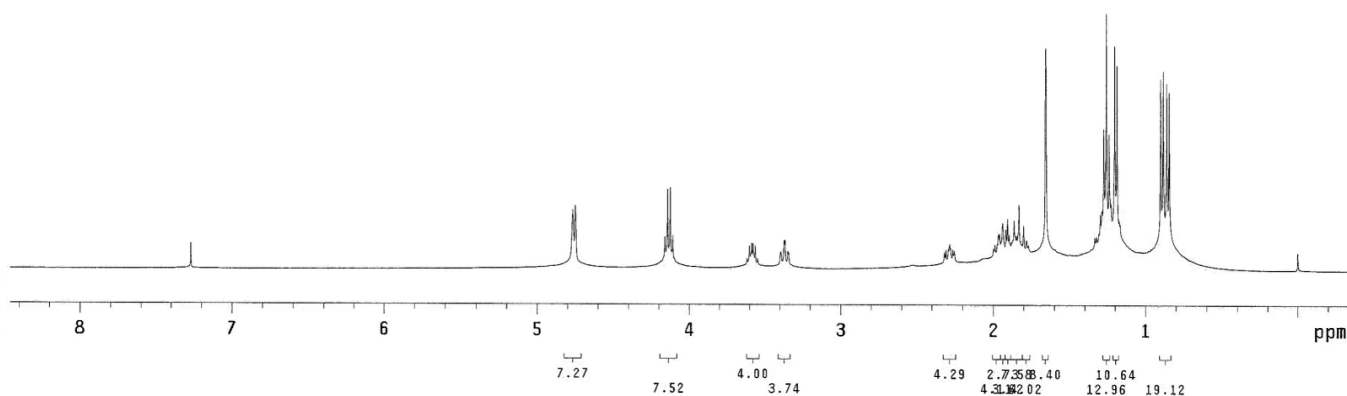
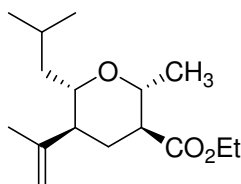
¹³C NMR spectra of 3i

```
PS_417_U
exp1 s2pu1
SAMPLE
date Dec 29 2010 temp not used
solvent CDC13 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_417~ hst 0.008
U_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6
at 1.199 il
np 60270 in
fb 13800 dp
bs 16 hs
d1 1.000 PROCESSING
nt 3000 lb 2.00
ct 2672 fn 65536
TRANSMITTER C13 sp
tn -579.4
sfrq 100.554 wp 21262.5
tof 1536.3 rfj
tpwr 61 rfp 7764.9
pw 9.300 rp -47.5
DECOUPLER lp -334.5
dn H1 PLOT
dof 0 wc 250
dm vvy sc 0
dmm w vs 19
dpwr 42 th 2
dmf 8900 nm no ph
```



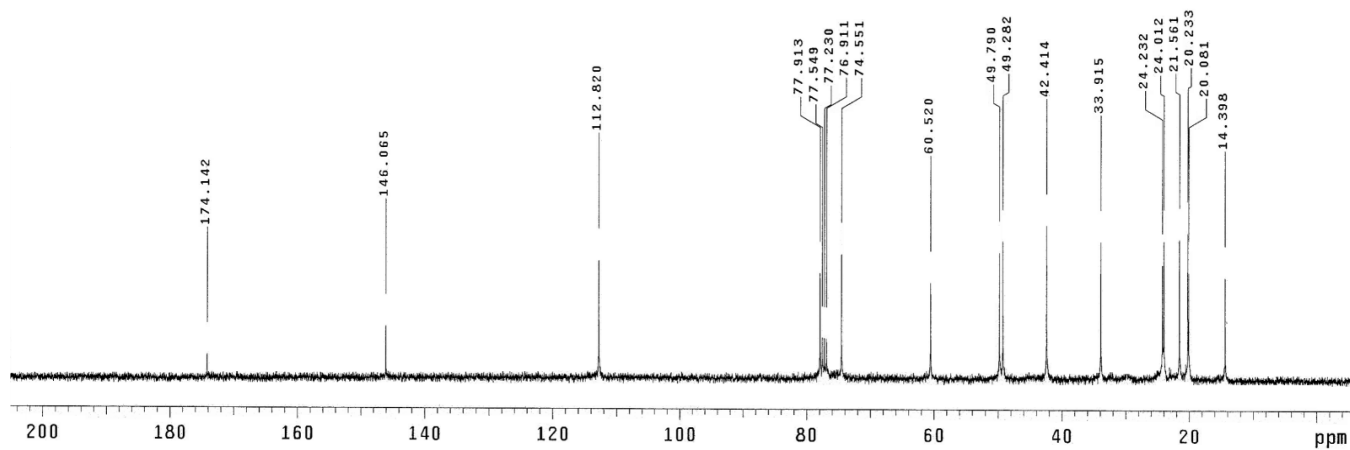
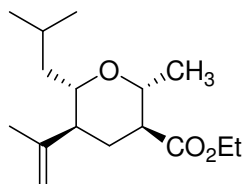
^1H NMR spectra of 3j

```
PS_413_U
exp1 s2pu1
SAMPLE
date Dec 24 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_413~ hst 0.008
U.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8 FLAGS
at 1.998 il n
np 25528 in n
fb not used dp y
bs 4 hs nn
d1 1.000 PROCESSING
nt 32 lb 0.10
ct 32 fn 65536
TRANSMITTER H1 sp DISPLAY
tn 399.853 wp -144.3
sfrq 362.8 rfl 3526.5
tof 57 rfp 790.8
tpwr 0
pw 9.850 rp 123.4
DECOUPLER C13 lp -62.6
dn dof 0 wc PLOT 250
dm dmm c vs 42
dpwr 50 th 20
dmf 15900 nm cdc ph
```



¹³C NMR spectra of 3j

```
PS_413_U
exp1 std13c
SAMPLE
date Dec 24 2010 temp not used
solvent CDC13 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_413~ hst 0.008
U_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25000.0
at 1.199 il n
np 59968 in n
fb 13800 dp y
bs 16 hs nn
d1 0
nt 3000 lb 1.00
ct 2544 fn not used
TRANSMITTER
tn C13 sp -578.1
sfrq 100.552 wp 21189.0
tof 0 rfl 10741.0
tpwr 61 rfp 7764.9
pw 8.667 rp -31.3
DECOUPLER lp -354.1
dn H1
dof 0 wc 250
dm yy sc 0
dmm w vs 24
dpwr 42 th 3
dmf 8900 nm no ph
```

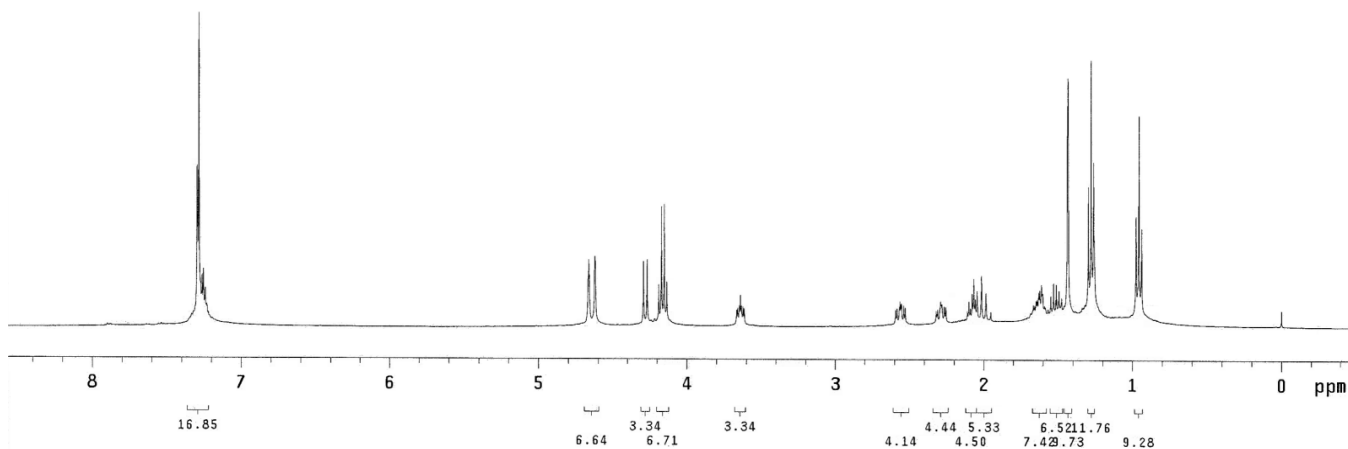
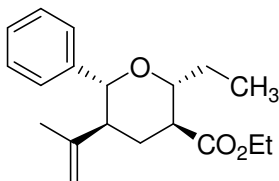


¹H NMR spectra of 3k

PS_456_U

expl s2pu1

SAMPLE		SPECIAL	
date	Feb 28 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_456~	hst	0.008
	_U.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
dl	1.000	PROCESSING	
nt	100	ib	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-192.9
sfrq	399.853	wp	3618.7
tof	362.8	rfl	797.4
tpwr	57	rfp	0
pw	9.850	rp	116.8
		lp	-103.9
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nn	sc	0
dmm	c	vs	52
dpwr	50	th	8
dmf	15900	nm	cdc ph



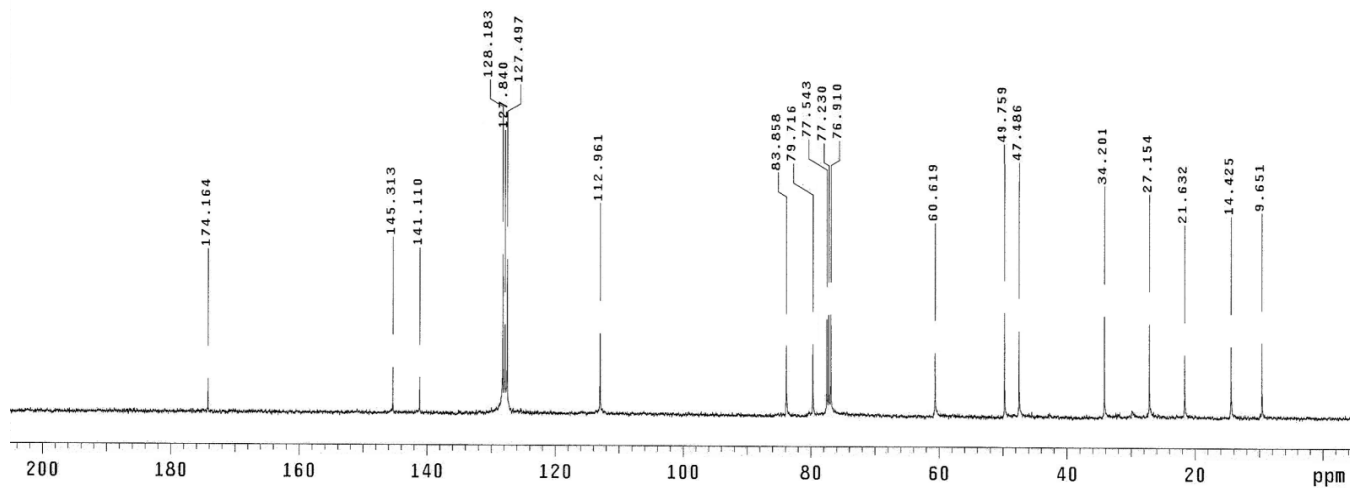
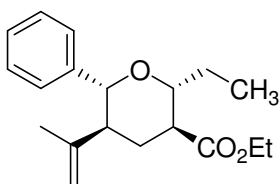
¹³C NMR spectra of 3k

```
PS_456_U
expl s2pu1

SAMPLE          SPECIAL
date Mar 1 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_456~ hst 0.008
U C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
di 1.000 PROCESSING
nt 8000 lb 2.00
ct 8000 fn 65536

TRANSMITTER C13 sp DISPLAY
tn C13 sp -449.0
sfrq 100.554 wp 21096.9
tof 1536.3 rfp 9269.8
tpwr 61 rfp 7764.9
pw 9.300 rp -89.1
DECOUPLER lp -290.8

dn H1 PLOT
dof 0 wc 250
dm yyy sc 0
dmm w vs 25
dpwr 42 th
dmf 8900 nm no ph 4
```

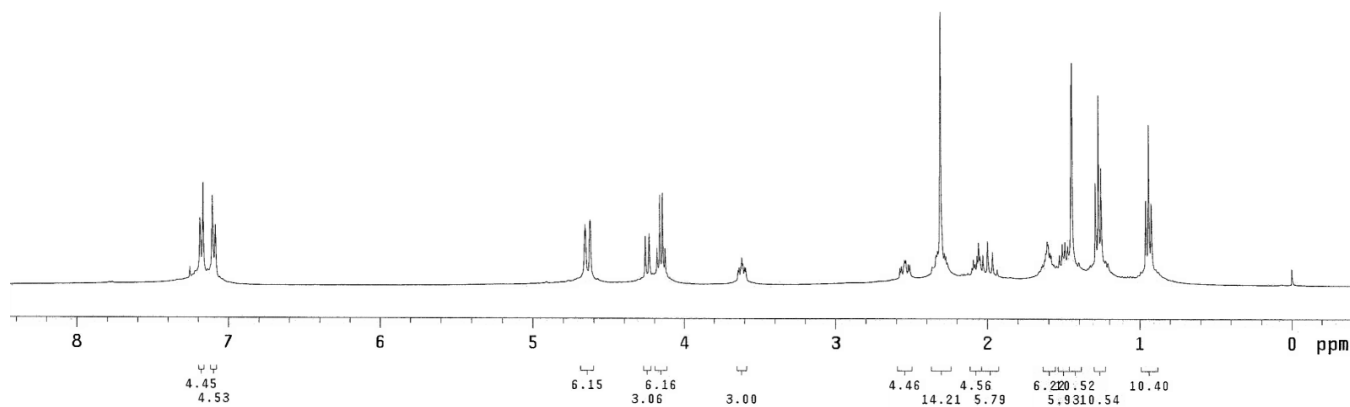
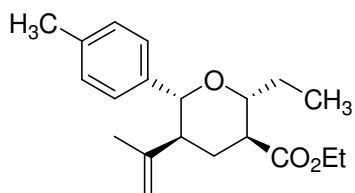


¹H NMR spectra of 3l

PS_464_U

expl s2pu1

```
SAMPLE SPECIAL
date Mar 14 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION hst 0.008
sw 6389.8 pw90 19.700
at 1.998 alfa 20.000
np 25528
fb not used il n
bs 8 in n
d1 1.000 dp y
nt 100 hs nn
ct 100
TRANSMITTER lb 0.10
tn H1 fn 65536
sfrq 399.853
tof 362.8 sp -166.7
tpwr 57 wp 3543.3
pw 9.850 rfl 796.4
DECOUPLER rfp 0
dn C13 rp 115.3
dof 0 lp -71.1
dm nnn
dmm c wc 250
dpwr 50 sc 0
dmf 15900 vs 46
nm cdc ph 15
```

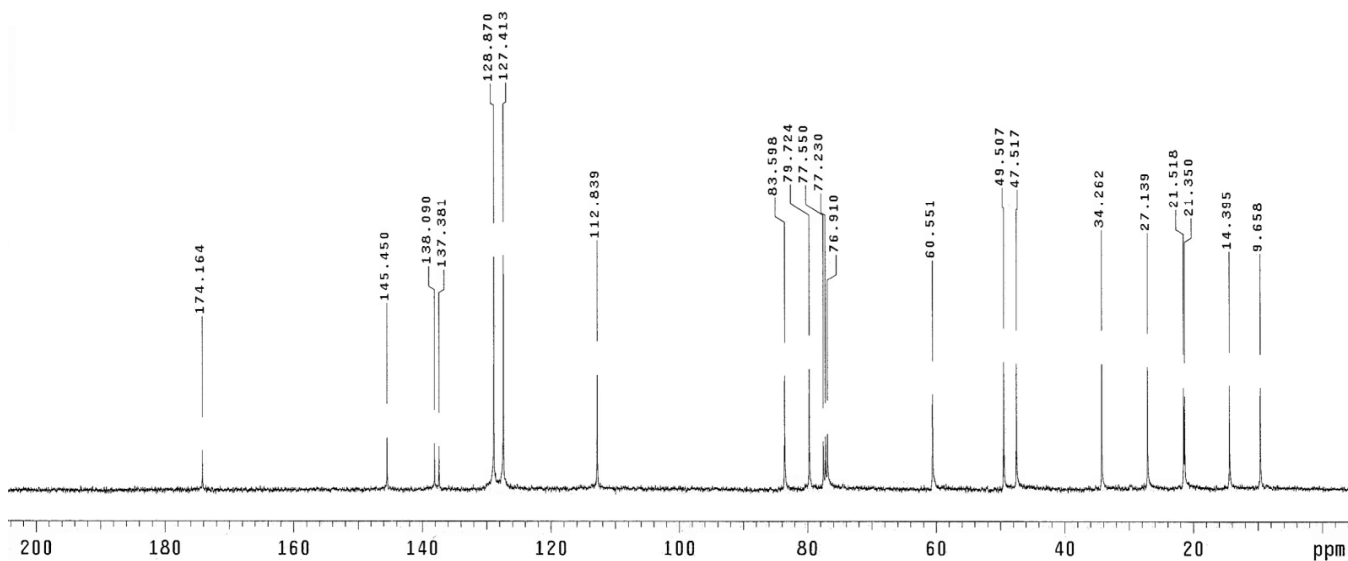
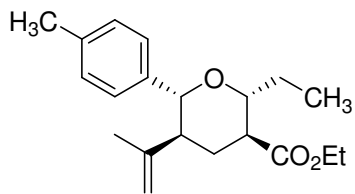


¹³C NMR spectra of 3l

PS_464_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 14 2011	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	fl	n
bs	16	in	n
d1	1.000	dp	y
nt	4000	hs	nn
ct	3024	PROCESSING	
TRANSMITTER	lb		2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-452.9
tpwr	61	wp	21030.9
pw	9.300	rfl	9273.6
DECOUPLER		rfp	7764.9
dn	H1	rp	-71.2
dof	0	lp	-301.8
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	35
		th	4
		nm	no ph

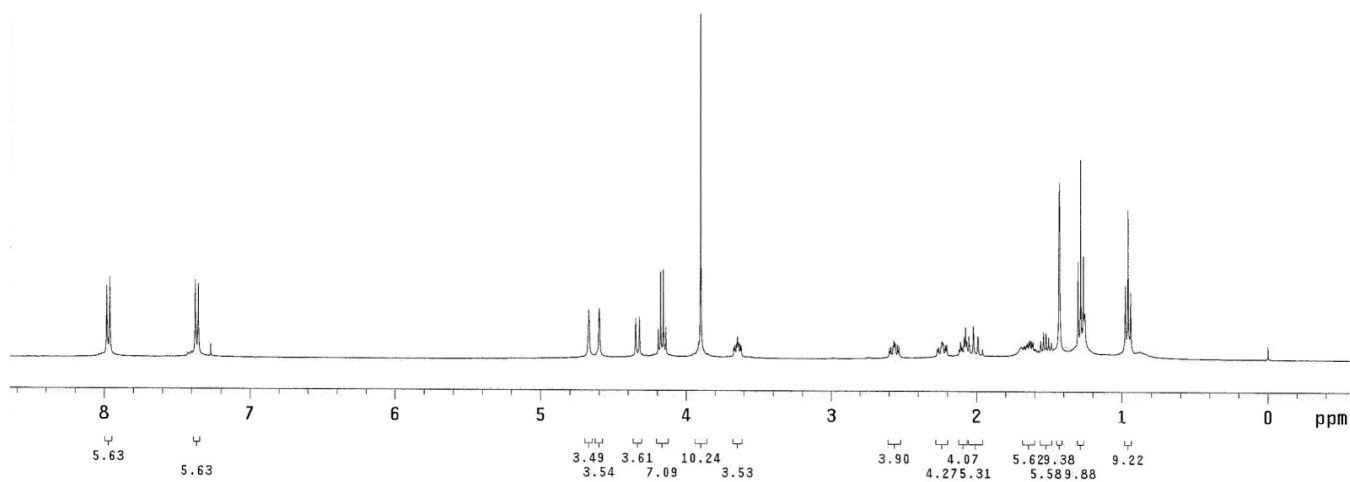
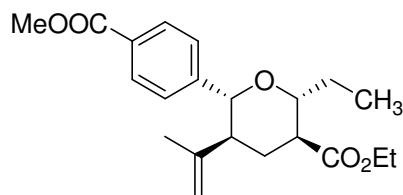


¹H NMR spectra of 3m

PS_458_U

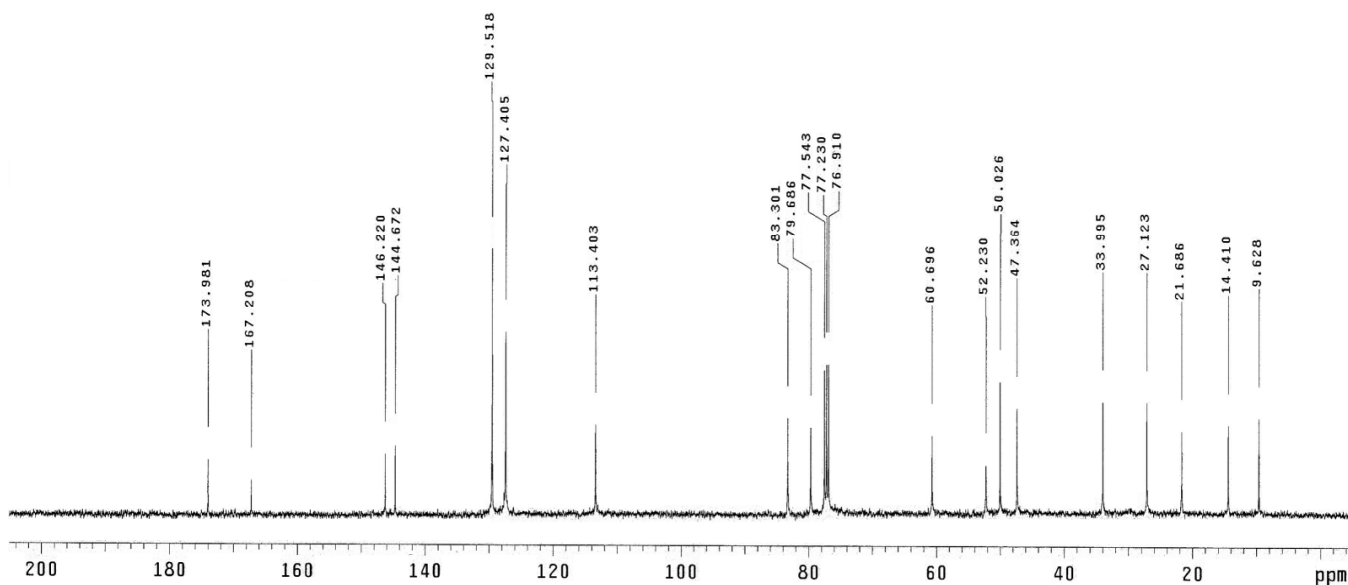
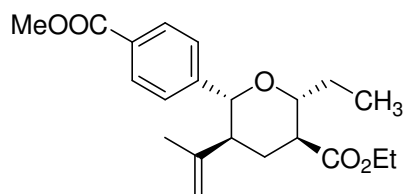
exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 2 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_458~		hst	0.008
U.fid		pw90	19.700
ACQUISITION	alfa		20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-236.5
sfrq	399.853	wp	3694.4
tof	362.8	rfl	790.6
tpwr	57	rffp	0
pw	9.850	rp	111.7
DECOUPLER	l1p		-95.9
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	57
dpwr	50	th	62
dmf	15900	nm	cdc ph



¹³C NMR spectra of 3m

```
PS_458_U
exp1 s2pu1
SAMPLE
date Mar 2 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_458~ hst 0.008
U_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 5000 lb 2.00
ct 4640 fn 65536
TRANSMITTER C13 sp -515.7
sfrq 100.554 wp 21130.6
tof 1536.3 rfl 9270.6
tpwr 61 rfp 7764.9
pw 9.300 rp -56.4
DECOUPLER H1 lp -332.0
dn
dof 0 wc 250
dm yyv sc 0
dmm w vs 41
dpwr 42 th 4
dmf 8900 nm no ph
```

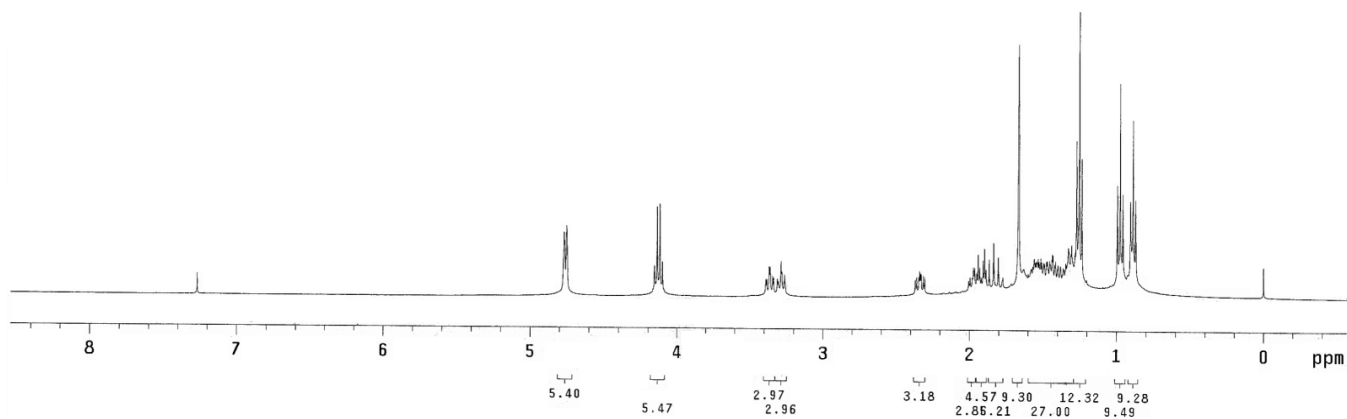
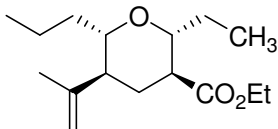


¹H NMR spectra of 3n

PS_463_U

exp1 s2pu1

date	Mar 12 2011	temp	not used	
solvent	CDCl3	gain	not used	
file	exp	spin	not used	
ACQUISITION		hst	0.008	
sw	6389.8	pw90	19.700	
at	1.998	alfa	20.000	
np	25528	FLAGS		
fb	not used	il	n	
bs	8	in	n	
d1	1.000	dp	y	
nt	100	hs	nn	
ct	100	PROCESSING		
tn	H1		lb	0.10
sfrq	399.853	fn	65536	
tof	362.8	DISPLAY		
tpwr	57	sp	-238.1	
pw	9.850	wp	3652.5	
DECOUPLER		rfl	792.1	
dn	C13	rfp	0	
dof	0	rp	111.9	
dm	nnn	lp	-86.1	
dmm	c	PLOT		
dpwr	50	wc	250	
dmf	15900	sc	0	
		vs	48	
		th	20	
		nm	cdc ph	

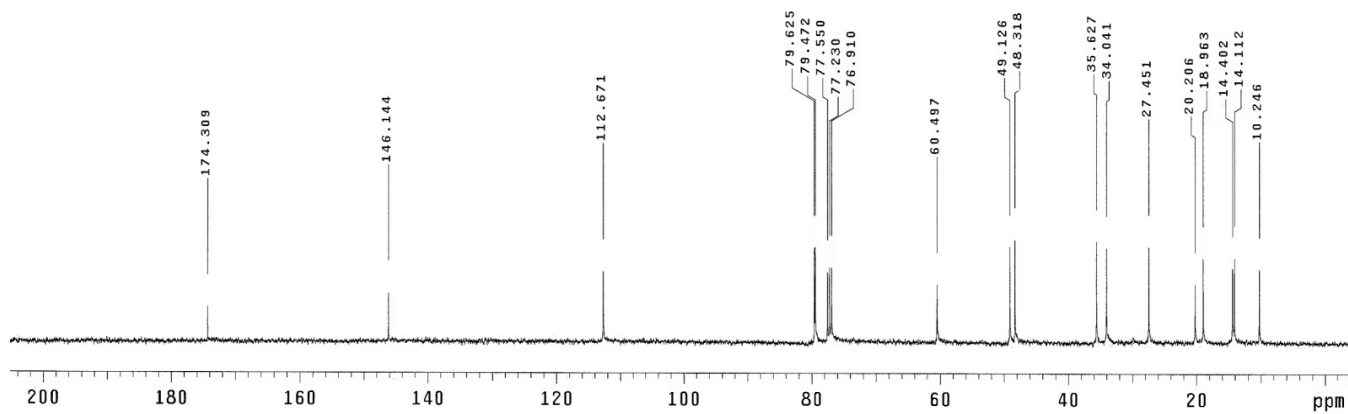
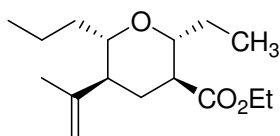


¹³C NMR spectra of 3n

PS_463_U

expl s2pu1

SAMPLE		SPECIAL	
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solvent	CDCl3	gain	not used
file		exp	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	5000	hs	nn
ct	2272	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-414.5
tpwr	61	wp	21063.9
pw	9.300	rfl	9268.3
DECOUPLER		rfp	7764.9
dn	H1	rp	-62.8
dof	0	lp	-346.1
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	16
		th	5
		nm	no ph

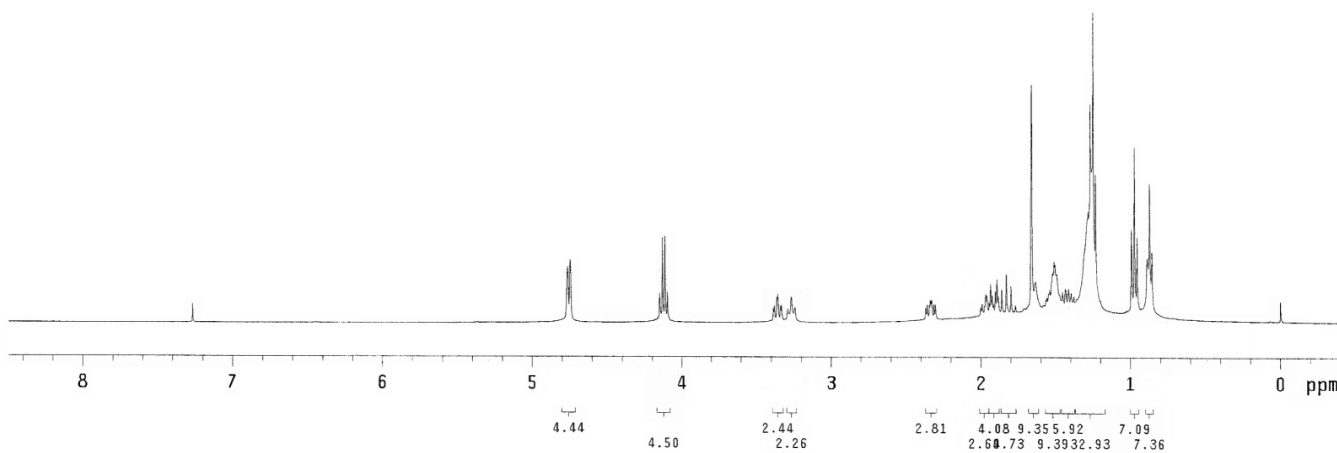
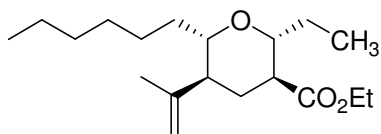


¹H NMR spectra of 3o

PS_469_U

expl s2pu1

SAMPLE		SPECIAL	
date	Mar 27 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION	exp	hst	0.008
sw	6389.8	pw90	19.700
at	1.998	alfa	20.000
np	25528	FLAGS	
fb	not used	il	n
bs	8	in	n
d1	1.000	dp	y
nt	100	hs	nn
ct	100	PROCESSING	
TRANSMITTER	H1	lb	0.10
tn		fn	65536
sfrq	399.853	DISPLAY	
tof	362.8	sp	-171.0
tpwr	57	wp	3568.4
pw	9.850	rfl	792.3
DECOUPLER	C13	rffp	0
dn	0	rp	112.2
dof	0	lp	-82.4
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	50	sc	0
dmf	15900	vs	49
		th	16
		nm	cdc ph

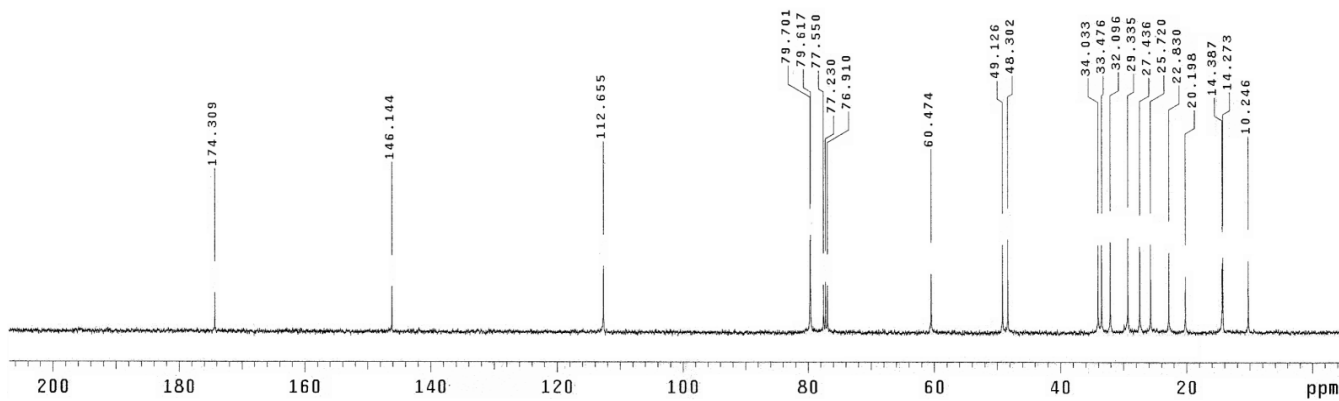
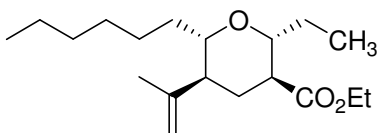


¹³C NMR spectra of 30

PS_469_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 27 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	4000	hs	nn
ct	880	PROCESSING	
tn		lb	2.00
sfrq	100.654	fn	65536
tof	1536.3	DISPLAY	
tpwr	61	sp	-515.0
pw	9.300	wp	21328.5
DECOUPLER		rfl	9269.8
		rfp	7764.9
dn	H1	rp	-55.3
dof	0	lp	-314.1
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	16
		th	
		nm	no ph 2

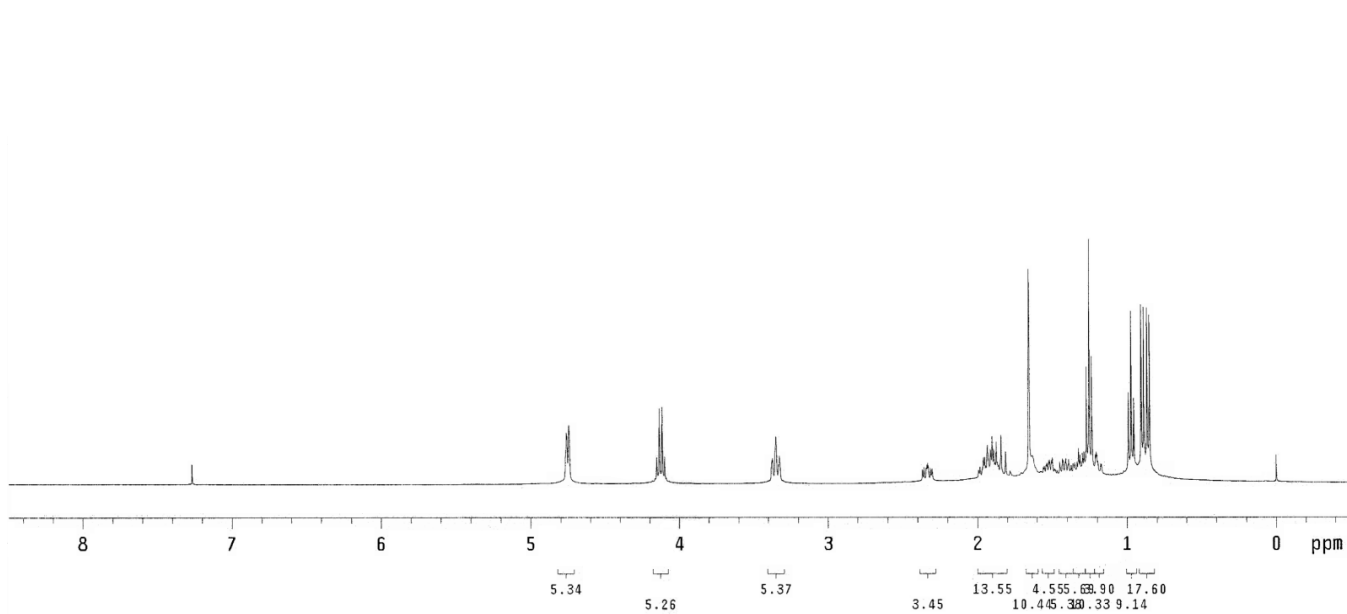
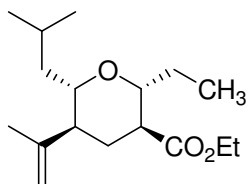


¹H NMR spectra of 3p

PS_467_U

exp1 s2pu1

```
SAMPLE          SPECIAL
date Mar 22 2011 temp not used
solvent CDC13 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_467~ hst 0.008
                U.fid pw90 19.700
ACQUISITION     alfa 20.000
sw 6389.8        FLAGS
at 1.998         il n
np 25528         in n
fb not used     dp y
bs 8             hs nn
d1 1.000         PROCESSING
nt 100           lb 0.10
ct 100           fn 65536
TRANSMITTER      DISPLAY
tn H1            sp -196.0
sfrq 399.853     wp 3593.6
tof 362.8        rfl 792.1
tpwr 57          rfp 0
pw 9.850         rp 109.1
DECOUPLER        lp -84.0
dn C13           PLOT
dof 0            wc 250
dm nnn          sc 0
dmm c           vs 38
dpwr 50         th 20
dmf 15900       nm cdc ph
```

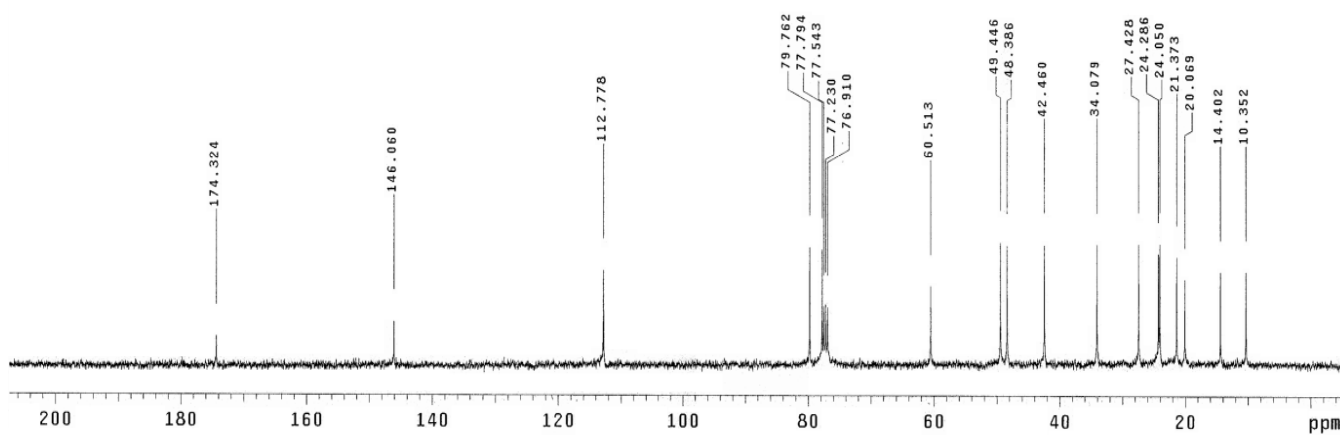
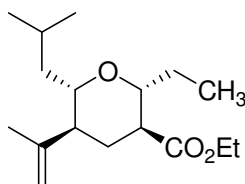


¹³C NMR spectra of 3p

PS_467_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 23 2011	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	8000	hs	nn
ct	1552	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq		DISPLAY	
tof	100.554	sp	-579.4
tpwr	1536.3	wp	21427.4
pw	61	rf1	9268.3
DECOUPLER		rfl	7764.9
dn	H1	rp	-49.4
dof	0	lp	-298.6
dm	yy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	20
		th	3
		nm	no ph

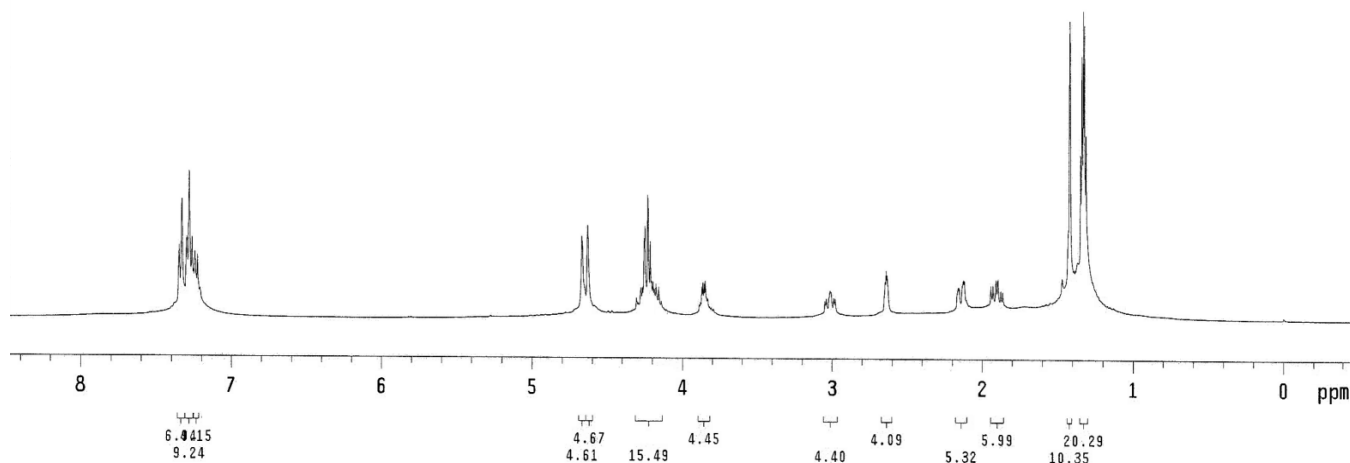
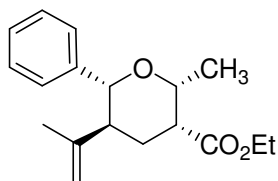


¹H NMR spectra of 4a

PS_425_M

expl s2pu1

SAMPLE		SPECIAL	
date	Jan 7 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_425~	hst	0.008
	_M.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
di	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-193.8
sfrq	399.853	wp	3585.2
tof	362.8	rfl	798.4
tpwr	57	rfp	0
pw	9.850	rp	118.5
		lp	-98.5
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	43
dpwr	50	th	31
dmf	15900	nm	cdc ph

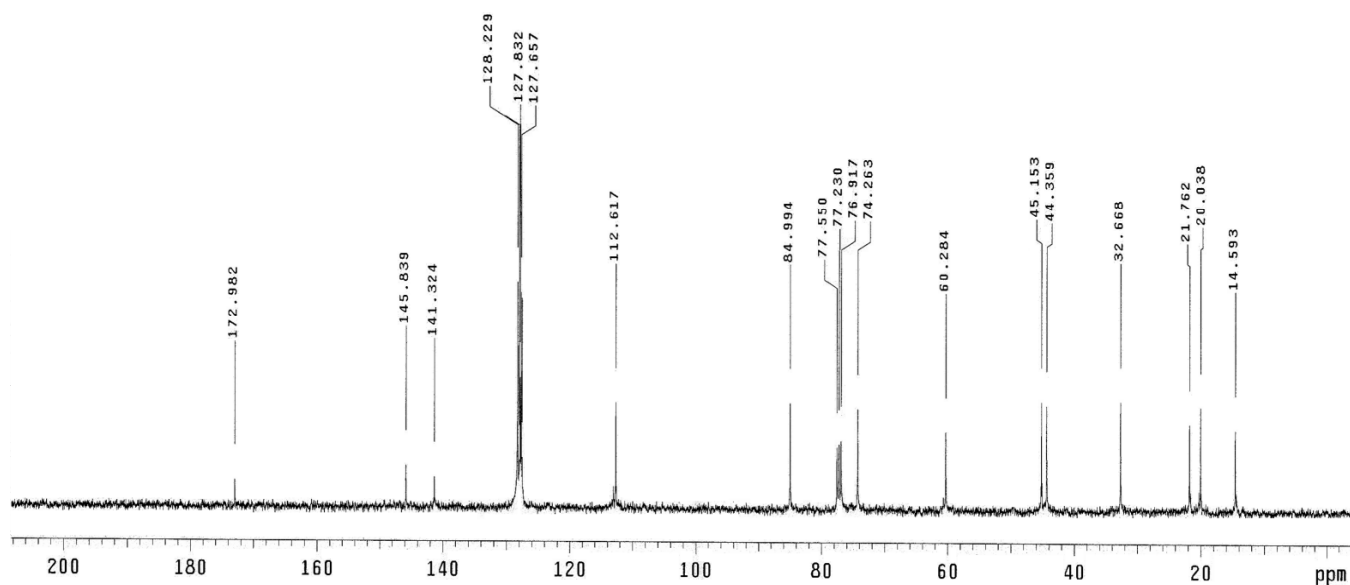
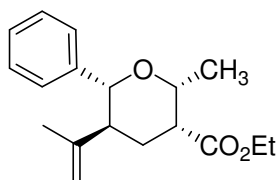


¹³C NMR spectra of 4a

PS_425_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 7 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_425~	hst	0.008
	M_C13_fid	pw90	18.600
		alfa	20.000
ACQUISITION		FLAGS	
sw	25125.6		
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	4000	lb	2.00
ct	1152	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-420.7
sfrq	100.554	wp	21361.4
tof	1536.3	rfl	9274.4
tpwr	61	rffp	7764.9
pw	9.300	rp	-48.1
		lp	-335.6
DECOUPLER		PLOT	
dn	H1		
dof	0	wc	250
dm	vyv	sc	0
dmm	w	vs	33
dpwr	42	th	3
dmf	8900	nm	no ph

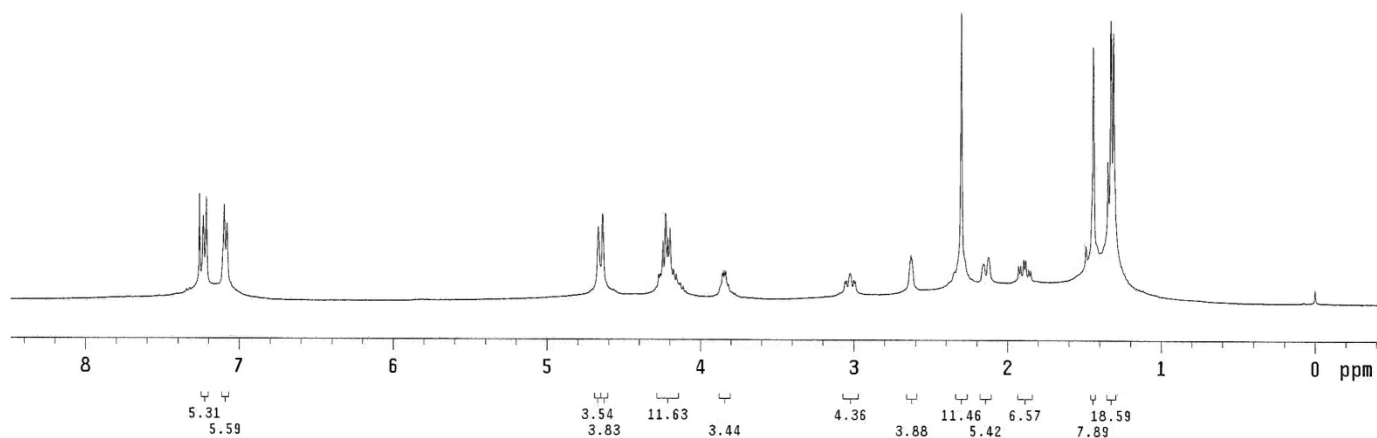
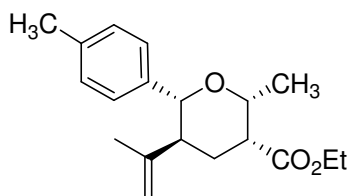


¹H NMR spectra of 4b

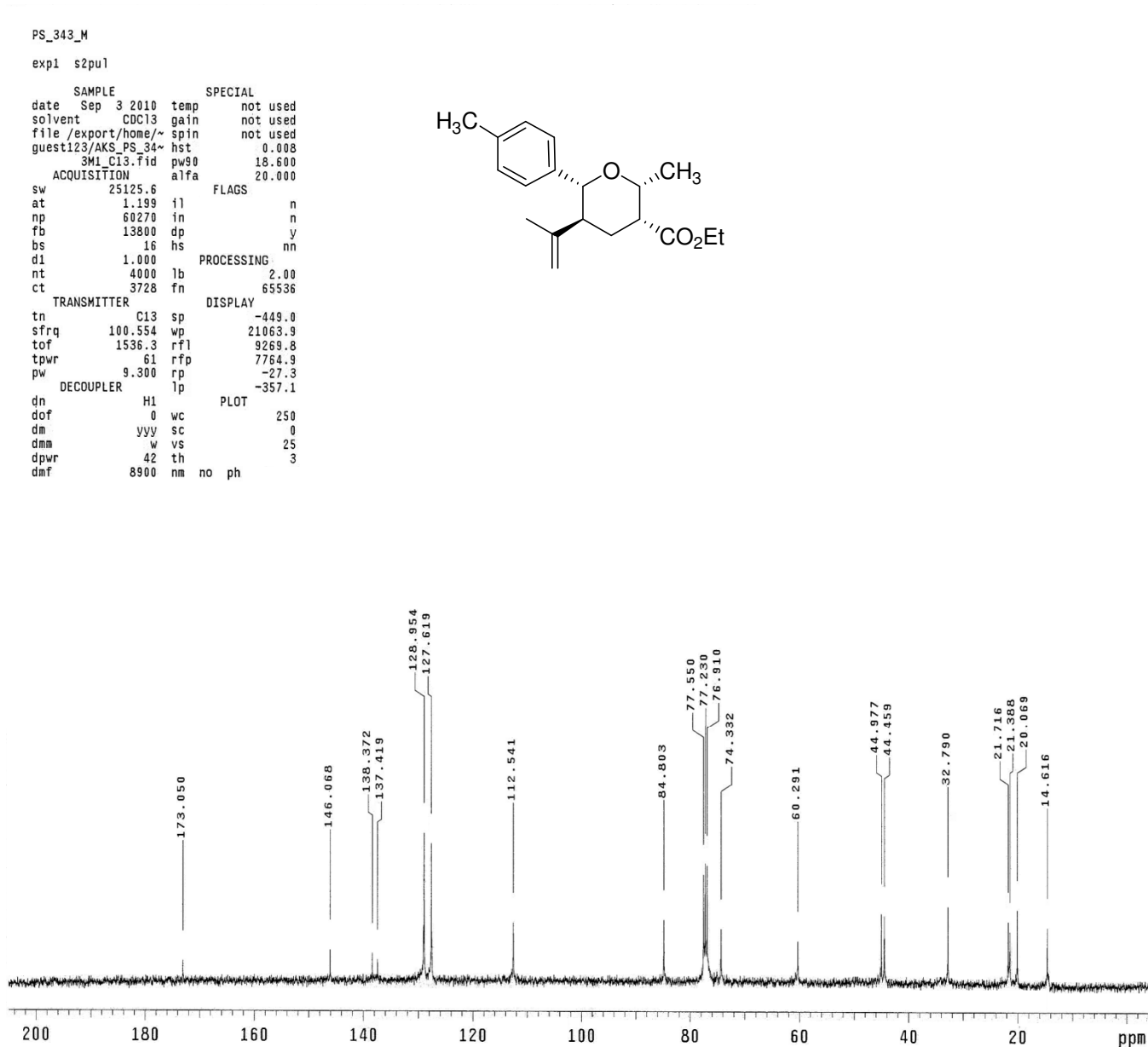
PS_343_μM

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 2 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
guest123/	AKS_PS_34~	hst	0.008
	3_M.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-166.3
sfrq	399.853	wp	3560.0
tof	362.8	rfl	796.0
tpwr	57	rfp	0
pw	9.850	rp	133.1
		lp	-95.1
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	44
dpwr	50	th	37
dmf	15900	nm	cdc ph



¹³C NMR spectra of 4b

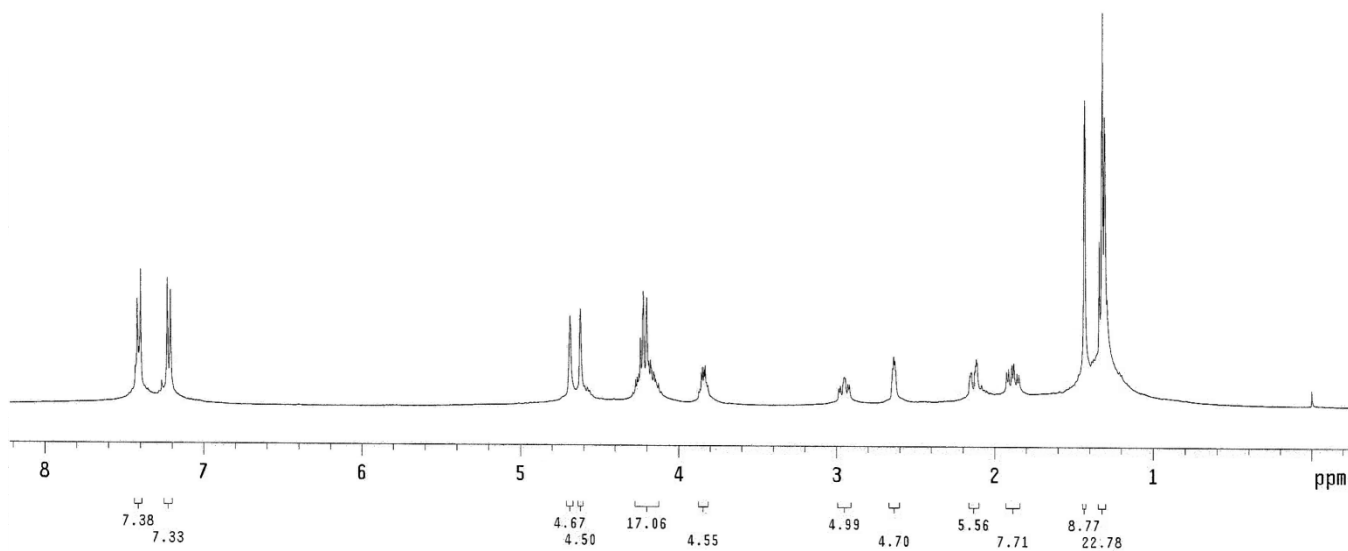
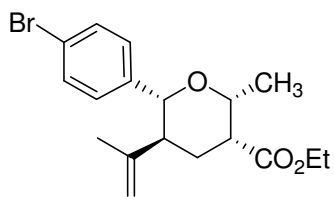


^1H NMR spectra of 4c

PS_341M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 18 2011	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_341~	hst	0.008
	M.fid	pw90	19.700
	alfa	alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8	il	n
at	1.998	in	n
np	25528	dp	y
fb	not used	hs	nn
bs	8		
d1	1.000	PROCESSING	
nt	100	fb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-104.5
sfrq	399.853	wp	3392.1
tof	362.8	rfl	792.9
tpwr	57	rffp	0
pw	9.850	rp	99.8
	lpc	lpc	-80.9
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	57
dmm	c	th	20
dpwr	50	cdc	ph
dmf	15900		

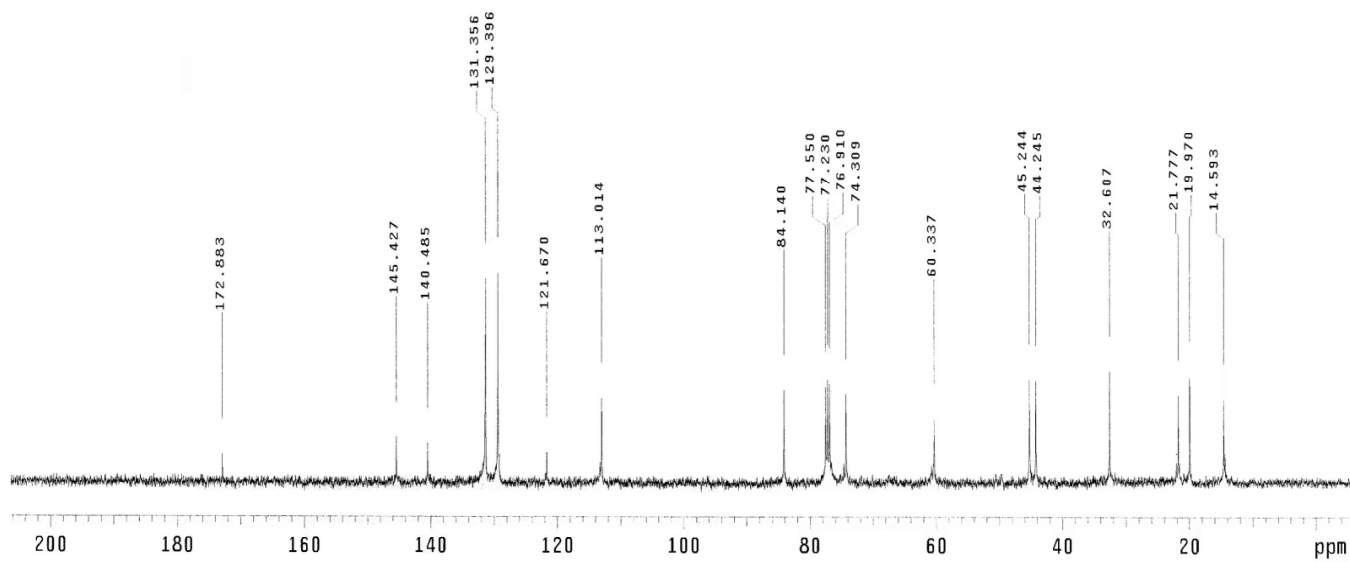
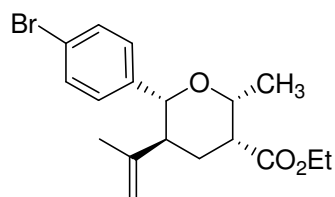


¹³C NMR spectra of 4c

PS_341_M

expl s2pu1

SAMPLE		SPECIAL	
date	Sep 2 2010	temp	not used
solvent	CDC13	gain	not used
file		exp	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	4000	hs	nn
ct	2736	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-586.3
tpwr	61	wp	21323.9
pw	9.300	rf1	9270.6
DECOUPLER		rfp	7764.9
dn	H1	rp	-40.7
dof	0	lp	-332.0
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	30
		th	3
		nm	no ph

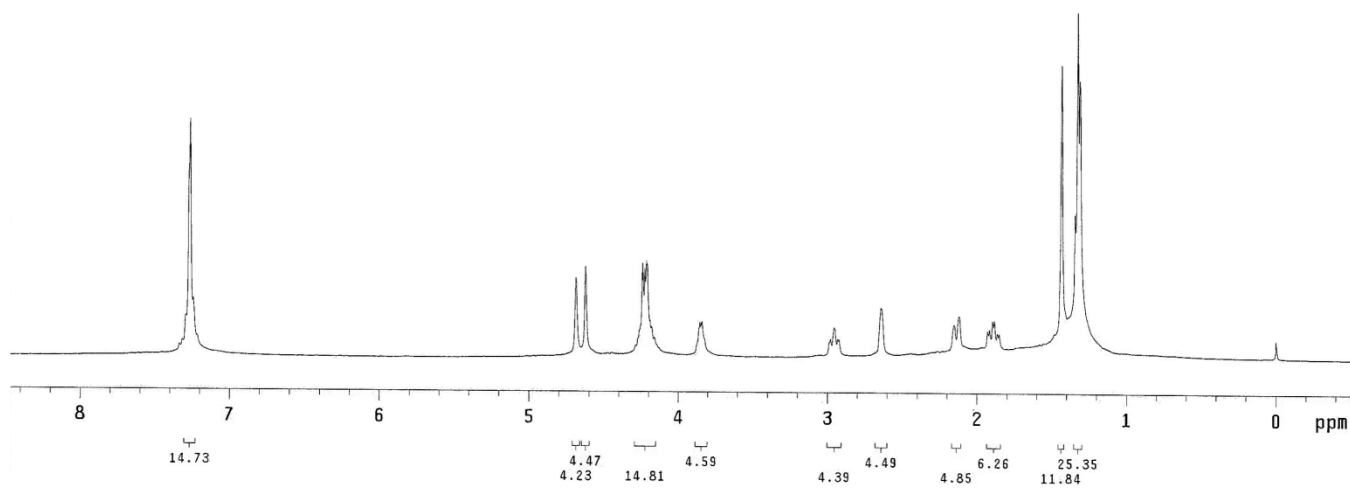
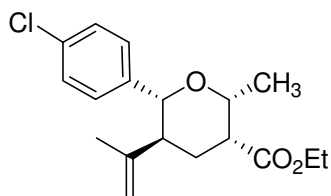


¹H NMR spectra of 4d

PS_395_M

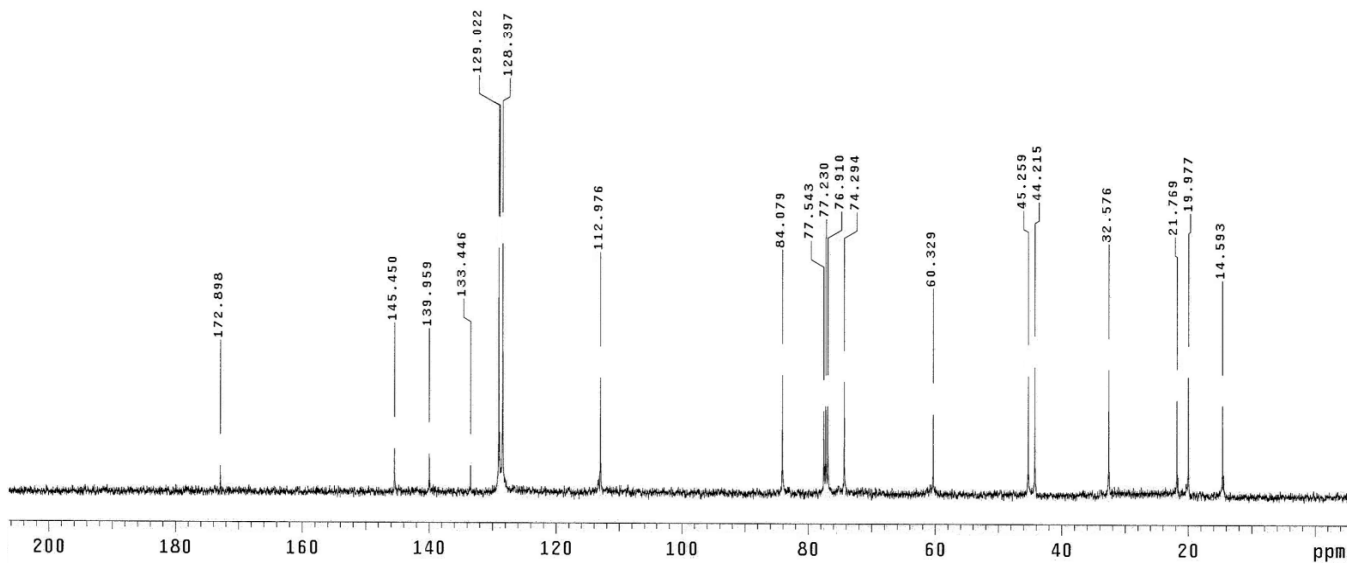
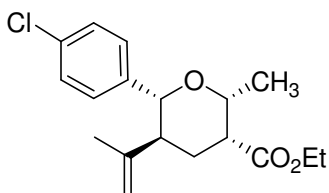
exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 4 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/	AKS_PS_395~	hst	0.008
	M1.fid	pw90	19.700
ACQUISITION	alfa		20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER	H1	DISPLAY	
tn		sp	-208.5
sfrq	399.853	wp	3593.6
tof	362.8	rfl	796.2
tpwr	57	rfp	0
pw	9.850	rp	113.0
DECOUPLER	lp		-103.1
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	56
dpwr	50	th	36
dmf	15900	nm	cdc ph



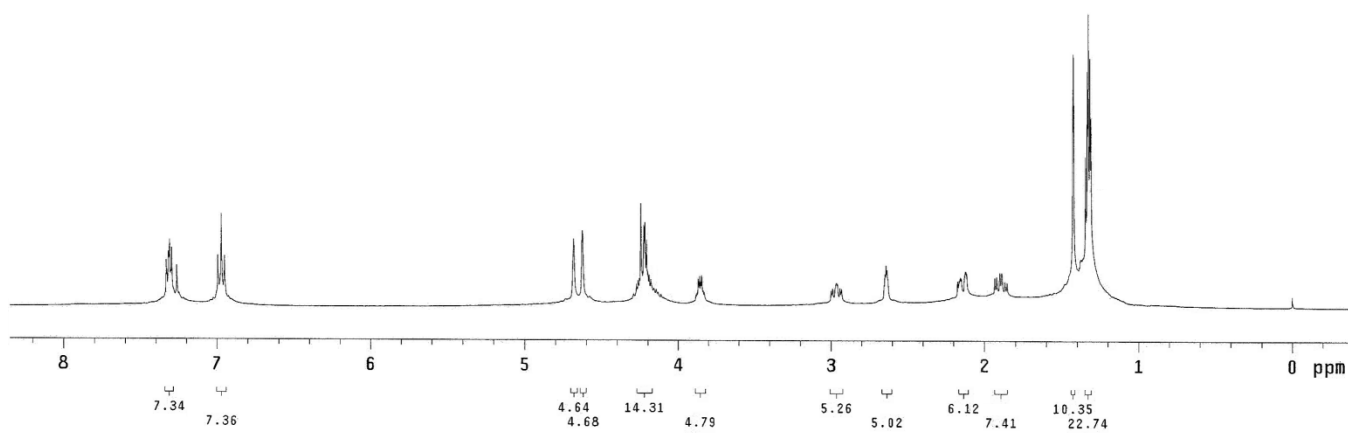
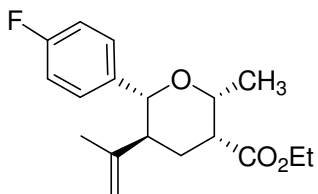
¹³C NMR spectra of 4d

```
PS_395_M
exp1 s2pu1
SAMPLE
date Nov 30 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_395~ hst 0.008
M_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 4000 lb 2.00
ct 1600 fn 65536
TRANSMITTER DISPLAY
tn C13 sp -582.4
sfrq 100.554 wp 21328.5
tof 1536.3 rfl 9271.3
tpwr 61 rfp 7764.9
pw 9.300 rp -28.0
DECOUPLER lp -367.4
dn H1 PLOT
dof 0 wc 250
dm yyv sc 0
dmm w vs 40
dpwr 42 th
dmf 8900 nm no ph 4
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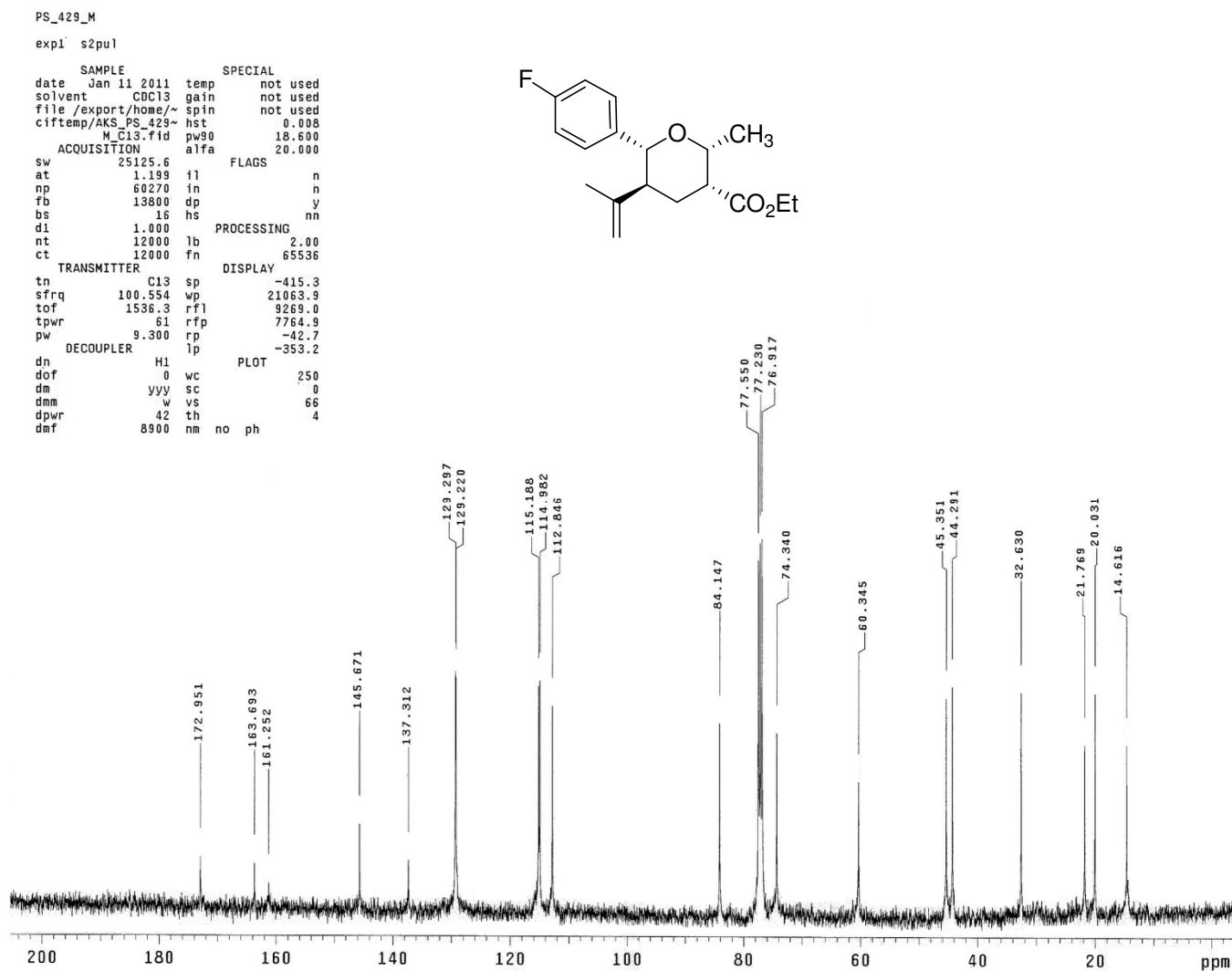


¹H NMR spectra of 4e

```
PS_4529_M
exp1 s2pu1
SAMPLE
date Jan 11 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_429~ hst 0.008
M.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8 FLAGS
at 1.998 il n
np 25528 in n
fb not used dp y
bs 8 hs nn
d1 1.000 PROCESSING
nt 200 lb 0.10
ct 200 fn 65536
TRANSMITTER H1 sp DISPLAY
tn 399.853 wp -154.6
sfrq 362.8 rfl 3493.0
tpwr 57 rfp 792.7
pw 9.850 rp 0
DECOUPLER lp 119.3
dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 49
dpwr 50 th 39
dmf 15900 nm cdc ph
```



¹³C NMR spectra of 4e

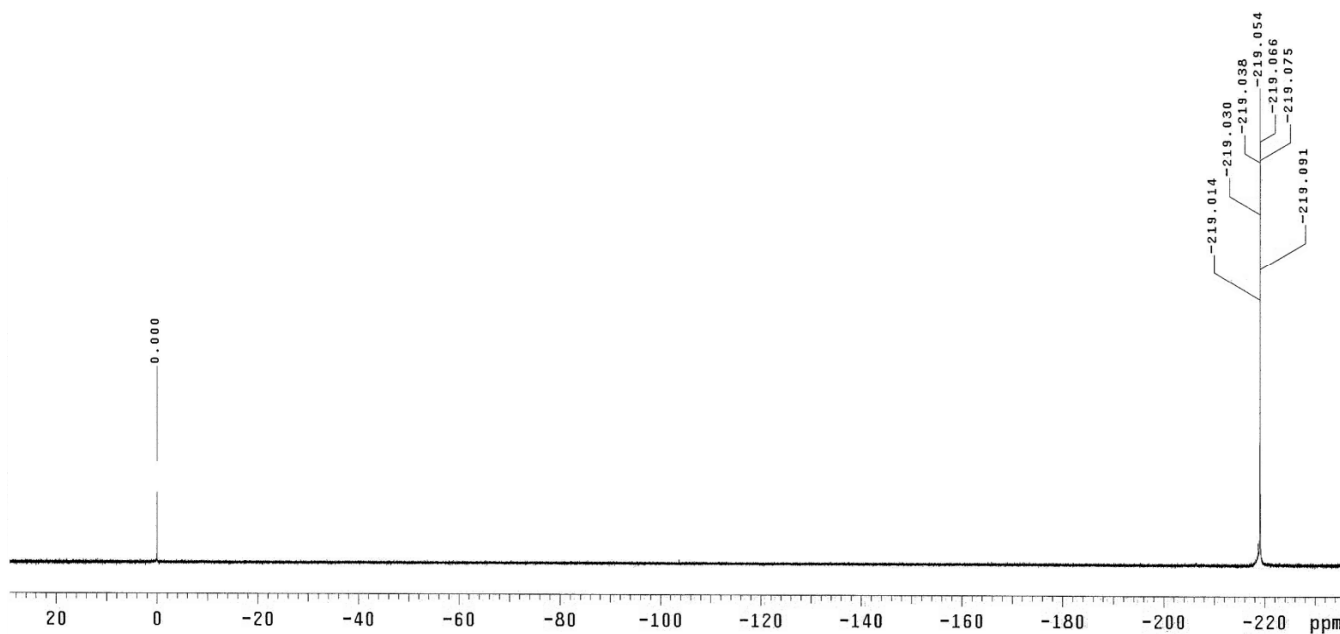
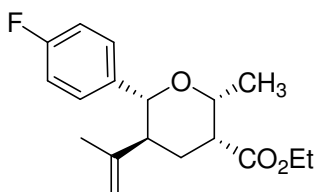


¹⁹F NMR spectra of 4e

PS_429_M

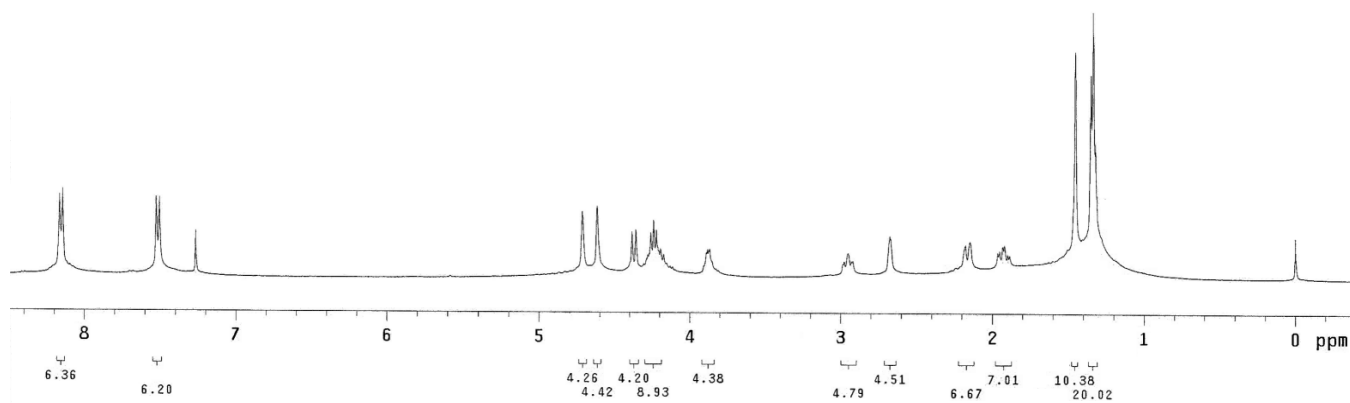
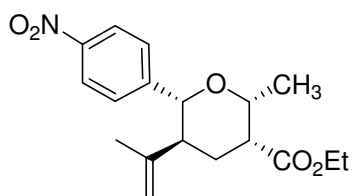
exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 11 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_429~	hst	0.008
M_Fluorine.fid		pw90	15.600
ACQUISITION	alfa		90.000
sw	100000.0	FLAGS	
at	0.600	il	n
np	119936	in	n
fb	55000	dp	y
bs	16	hs	nn
d1	1.500	PROCESSING	
nt	64	lb	0.30
ct	64	lsfid	-10
TRANSMITTER		fn	not used
tn	F19	DISPLAY	
sfrq	376.236	sp	-89016.6
tof	40099.4	wp	100000.0
tpwr	58	rfl	89016.6
pw	5.200	rfp	0
DECOUPLER		rp	-124.8
dn	H1	lp	22.3
dof	0	PLOT	
dm	nm	wc	250
dmm	c	sc	0
dpwr	42	vs	29
dmf	8900	th	10
	ai	no	ph



^1H NMR spectra of 4f

```
PS_391_M
expl std1h
SAMPLE
date Nov 16 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_391~ hst 0.008
_M.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6006.0 FLAGS
at 1.995 il n
np 23964 in n
fb not used dp y
bs 16 hs nn
dl 1.000
nt 200 fn PROCESSING not used
ct 128 DISPLAY
TRANSMITTER sp -156.2
tn H1 wp 3551.3
sfrq 399.853 rfl 963.8
tof 0 rfp 0
tpwr 57 rp 116.5
pw 7.000 lp -88.5
DECOUPLER C13 wc PLOT 250
dof 0 sc 0
dm nnn vs 46
dmm c th 30
dpwr 50 nm cdc ph
dmf 15900
```

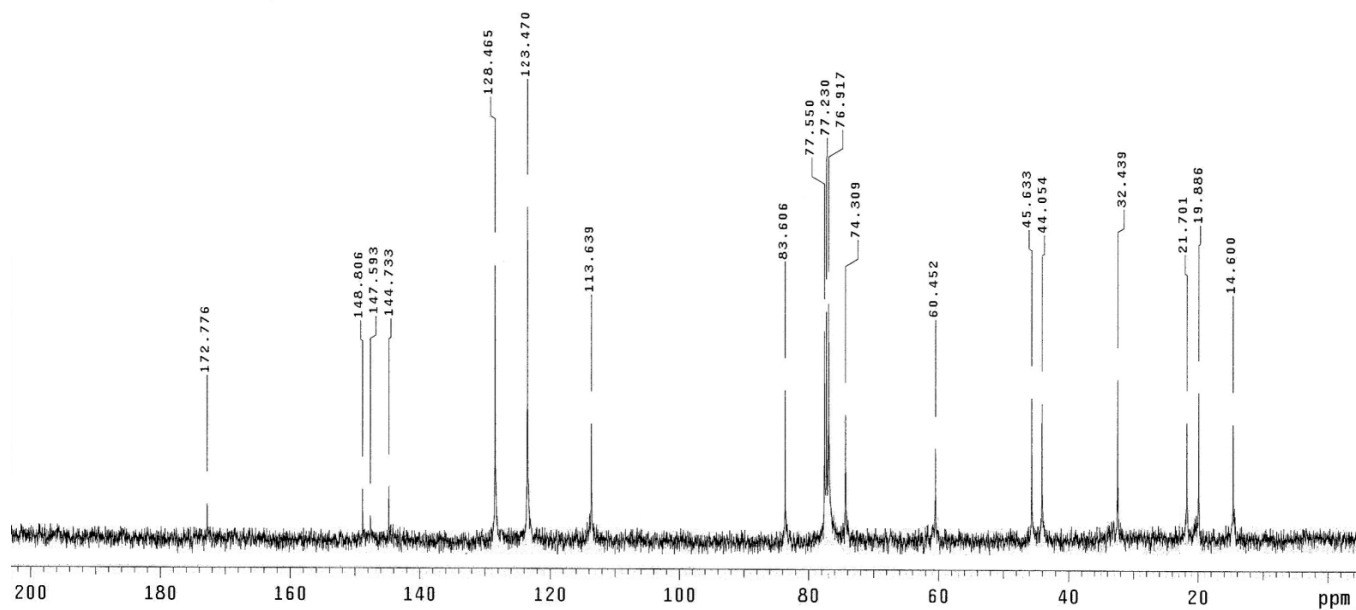
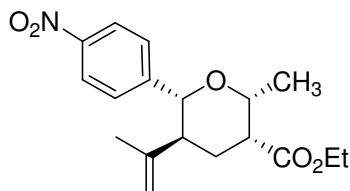


¹³C NMR spectra of 4f

PS_340_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Aug 26 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
guest123/AKS_PS_34~		hst	0.008
0M_C13.fid		pw90	18.600
ACQUISITION		alfa	20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	5000	lb	2.00
ct	3616	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-448.3
sfrq	100.554	wp	20866.1
tof	1536.3	rfl	9269.0
tpwr	61	rfp	7764.9
pw	9.300	rp	-31.3
DECOUPLER		lp	-373.0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	51
dpwr	42	th	3
dmf	8900	nm	no ph

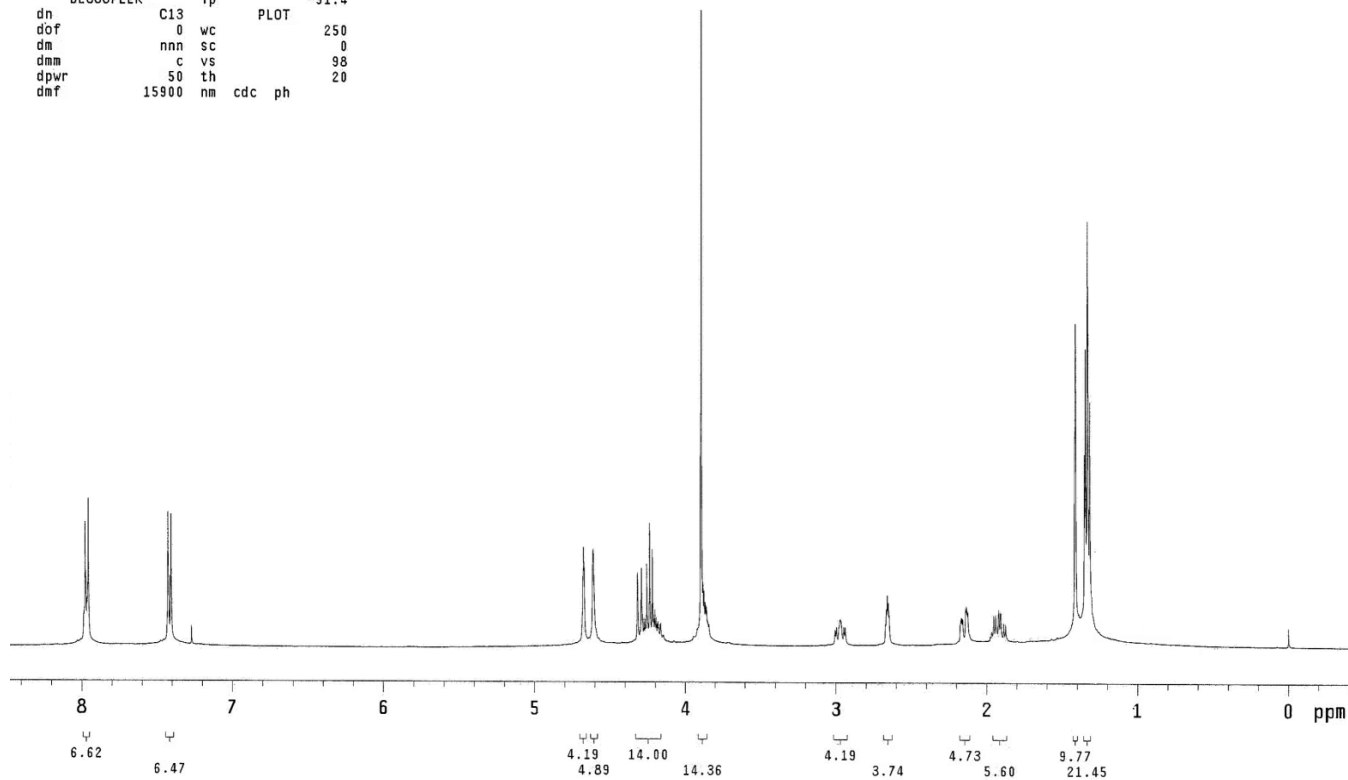
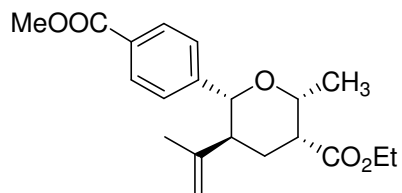


¹H NMR spectra of 4g

PS_338M

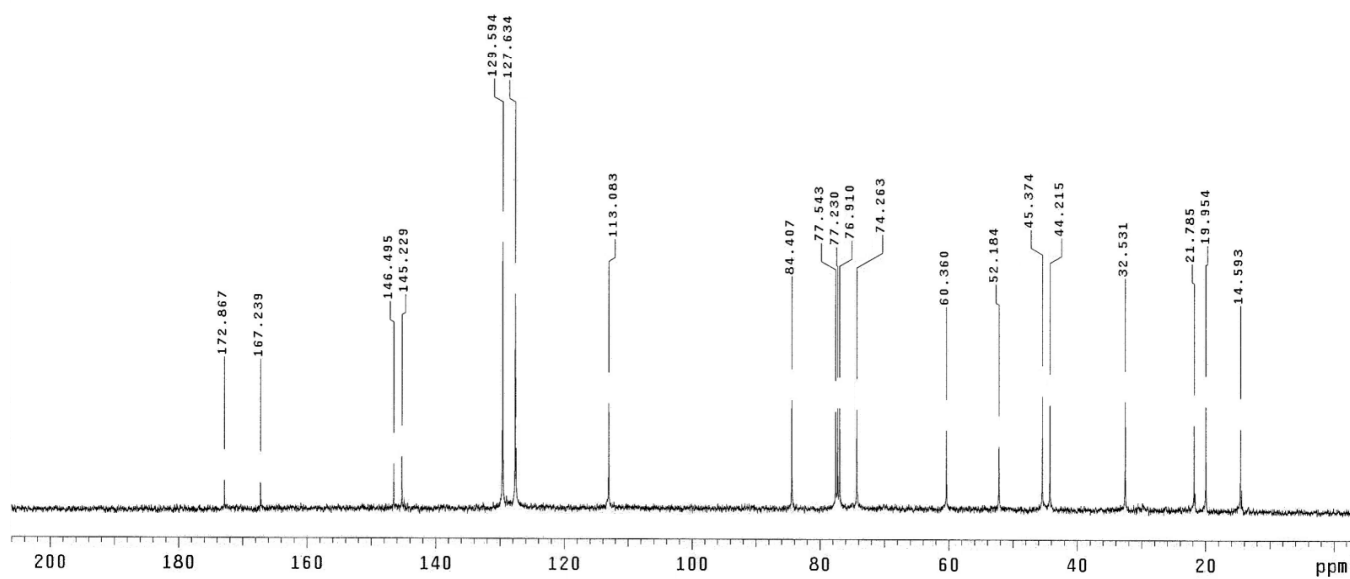
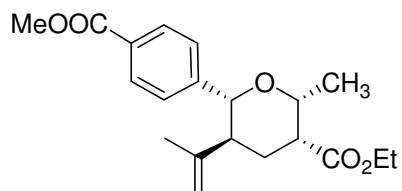
expl s2pu1

SAMPLE		SPECIAL	
date	Feb 3 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_338-	hst		0.008
	M.fid	pw90	19.700
	alfa		20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	88	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-169.1
sfrq	399.853	wp	3560.0
tof	362.8	rfl	790.4
tpwr	57	rfp	0
pw	9.850	rp	107.7
		lp	-91.4
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	98
dpwr	50	th	20
dmf	15900	nm	cdc ph



¹³C NMR spectra of 4g

```
PS_338M
exp1 s2pu1
SAMPLE
date Feb 6 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_338- hst 0.008
M1_C13.fid pw90 18.600
ACQUISITION a17a 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 5000 lb 2.00
ct 2048 fn 65536
TRANSMITTER C13 sp -317.9
sfrq 100.554 wp 21030.9
tof 1536.3 rfl 9271.3
tpwr 61 rfp 7764.9
pw 9.300 rp -29.5
DECOUPLER H1 lp -367.6
dn H1 PLOT
dof 0 wc 250
dm yyy sc 0
dmm w vs 43
dpwr 42 th 4
dmf 8900 nm no ph
```

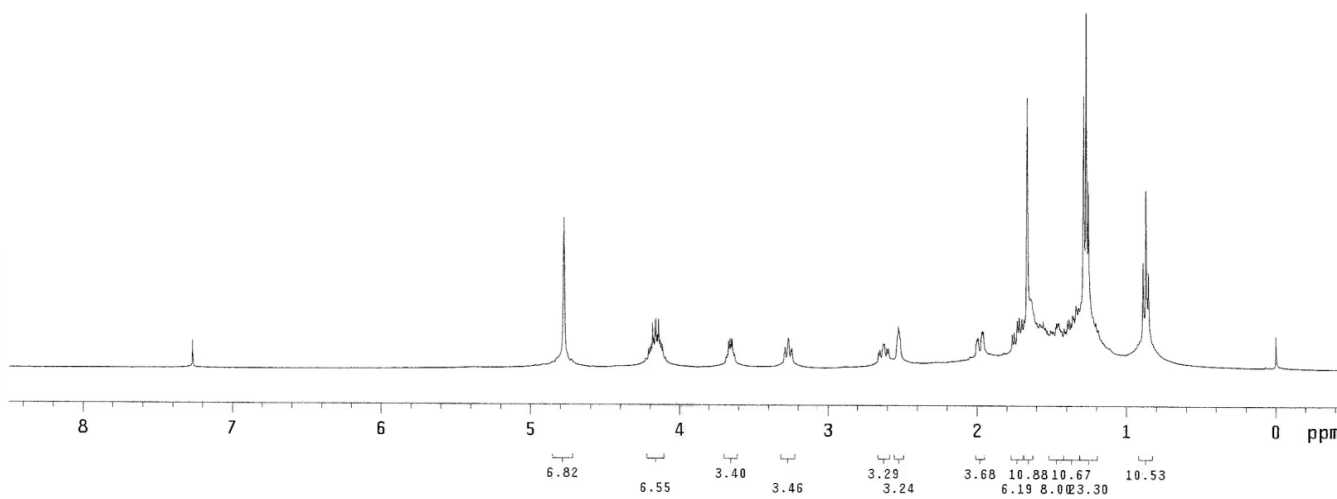
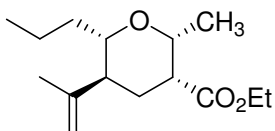


¹H NMR spectra of 4h

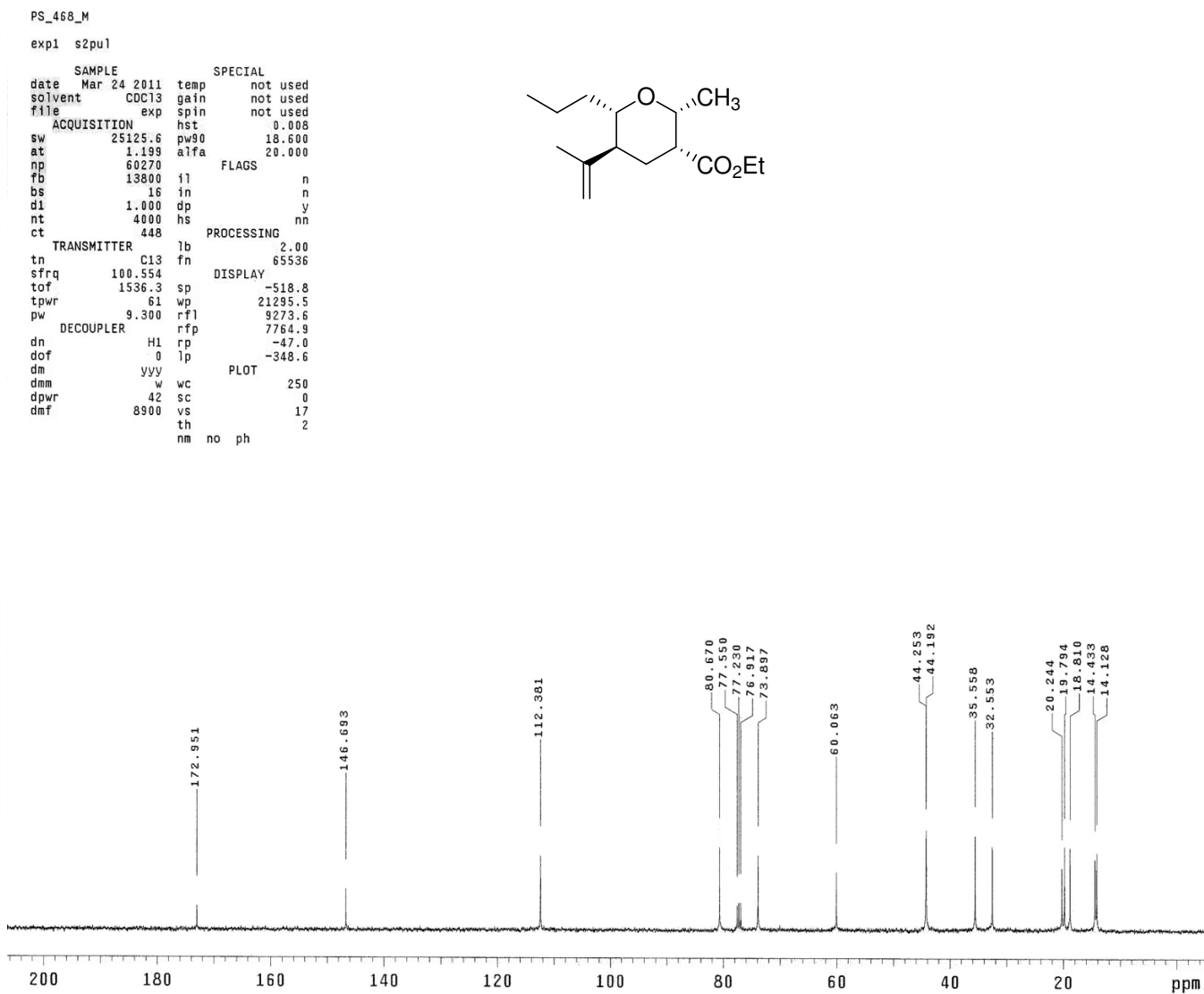
PS_468_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 23 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		exp	0.008
sw	6389.8	pw90	19.700
at	1.998	alfa	20.000
np	25528	FLAGS	
fb	not used	il	n
bs	8	in	n
d1	1.000	dp	y
nt	100	hs	nn
ct	100	PROCESSING	
tn	TRANSMITTER	lb	0.10
sfrq	H1	fn	65536
tof	399.853	DISPLAY	
tpwr	362.8	sp	-188.0
pw	57	wp	3585.2
dn	9.850	rfl	792.5
DECOUPLER		rfp	0
dof	C13	rp	103.4
dm	0	lp	-53.0
dmm	nnn	PLOT	
dpr	c	wc	250
dmf	50	sc	0
	15900	vs	59
		th	20
		nm	cdc ph



¹³C NMR spectra of 4h

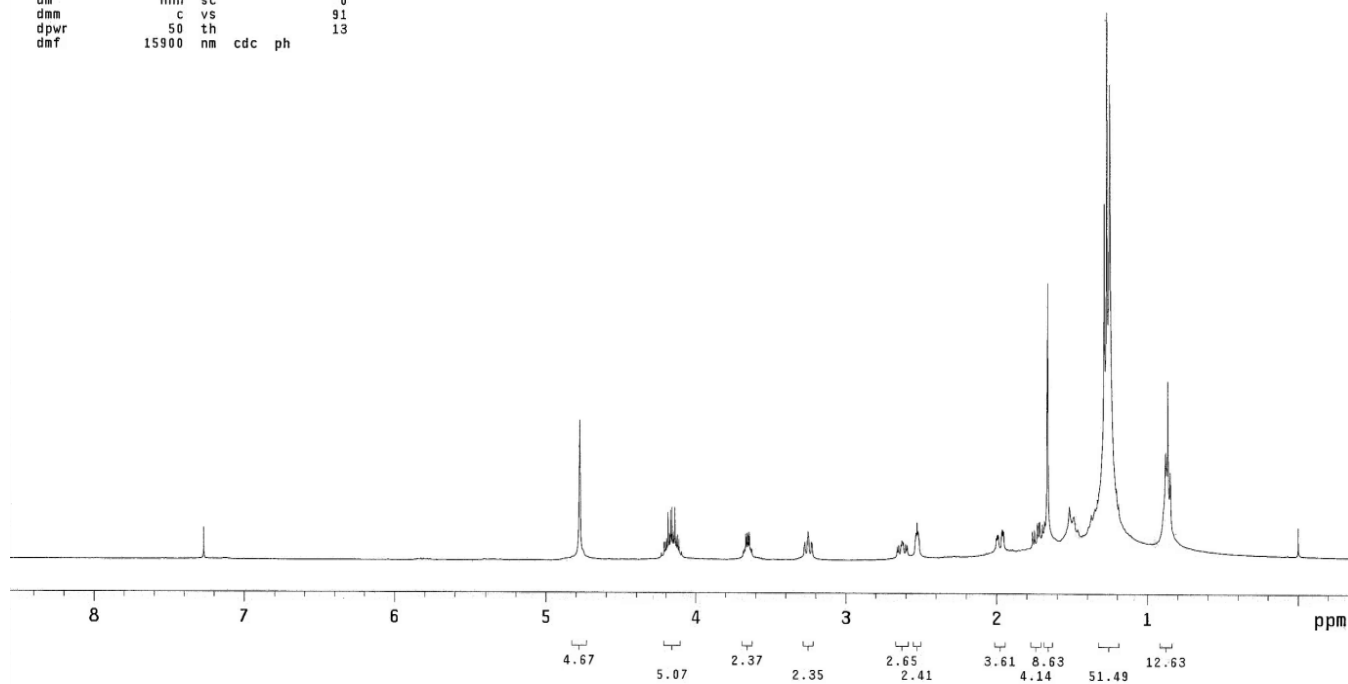
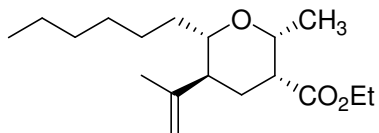


¹H NMR spectra of 4i

PS_417_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 3 2011	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_417~	hst	0.008
	_M2.fid	pw90	19.700
	alfa		20.000
ACQUISITION		FLAGS	
sw	6389.8	il	n
at	1.998	in	n
np	25528	dp	y
fb	not used	hs	nn
bs	8	PROCESSING	
d1	1.000	lb	0.10
nt	100	fn	65536
ct	80	DISPLAY	
TRANSMITTER		H1	sp
trf	399.853	wp	-145.3
stf	362.8	rfl	3568.4
tpwr	57	rff	791.7
pw	9.850	rp	0
		rp	98.2
DECOUPLER		lp	-78.3
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	91
dpwr	50	th	13
dmf	15900	nm	cdc ph

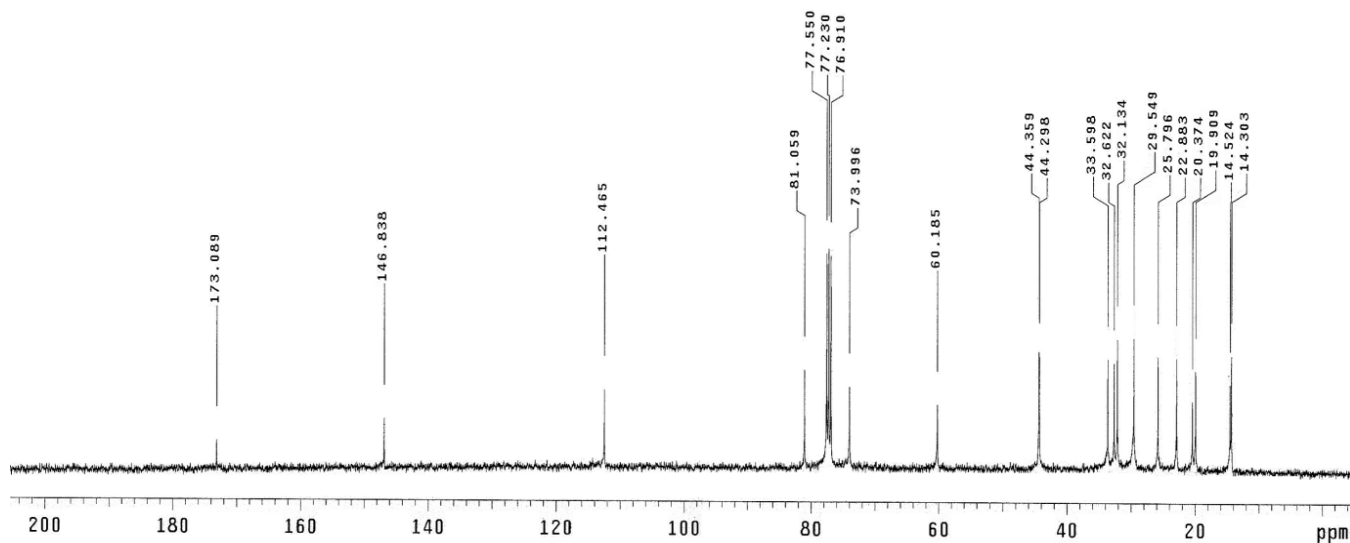
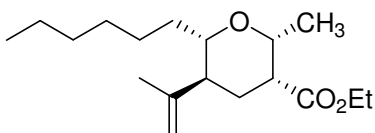


¹³C NMR spectra of 4i

PS_417_M

expl s2pul

SAMPLE		SPECIAL	
date	Jan 5 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_417~	hst	0.008
_M_C13.fid		pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	12000	lb	2.00
ct	12000	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-515.0
sfrq	100.554	wp	21163.6
tof	1536.3	rfl	9269.8
tpwr	61	rfp	7764.9
pw	9.300	rp	-42.2
DECOUPLER		lp	-329.2
dn	H1	PLOT	
dof	0	wc	250
dm	vyv	sc	0
dmm	w	vs	33
dpwr	42	th	3
dmf	8900	nm	no ph

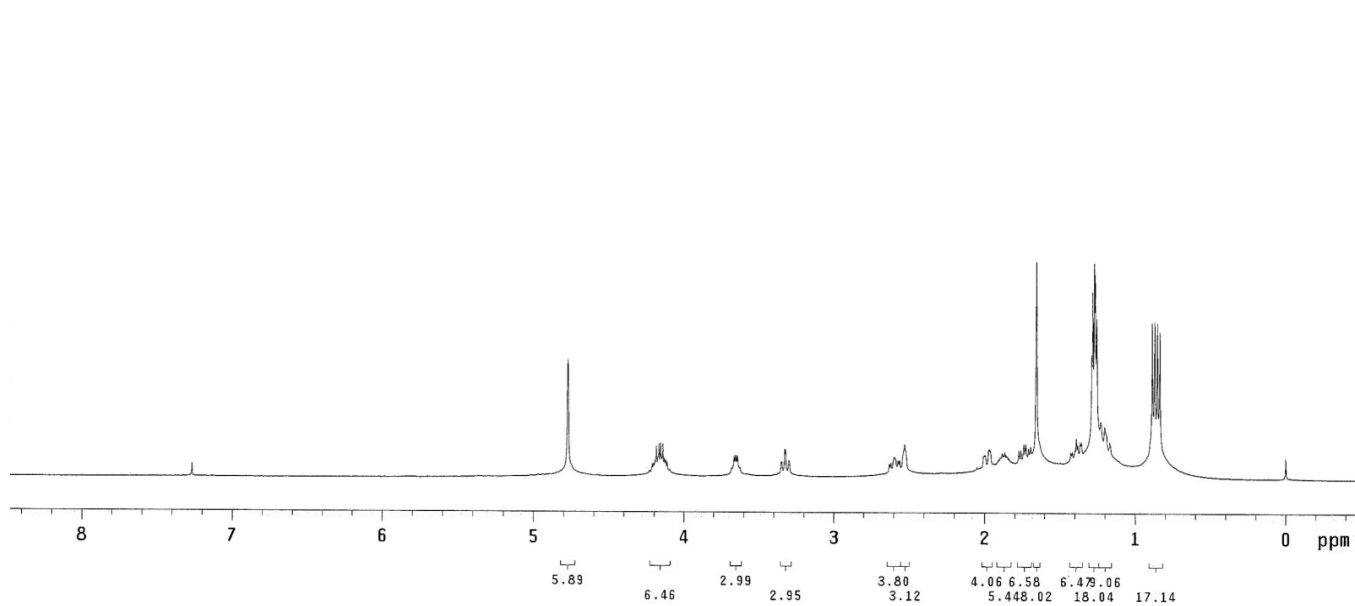
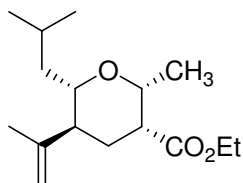


¹H NMR spectra of 4j

PS_413_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Dec 25 2010	temp	not used
solvent	CDCl3	gain	not used
file /export/home/~		spin	not used
ciftemp/AKS_PS_413~		hst	0.008
_M1.fid		pw90	19.700
ACQUISITION	alfa		20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	12	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-187.8
sfrq	399.853	wp	3576.8
tof	362.8	rfl	792.3
tpwr	57	rfl	0
pw	9.850	rp	118.4
DECOUPLER		lp	-77.4
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	36
dpwr	50	th	20
dmf	15900	nm	cdc ph

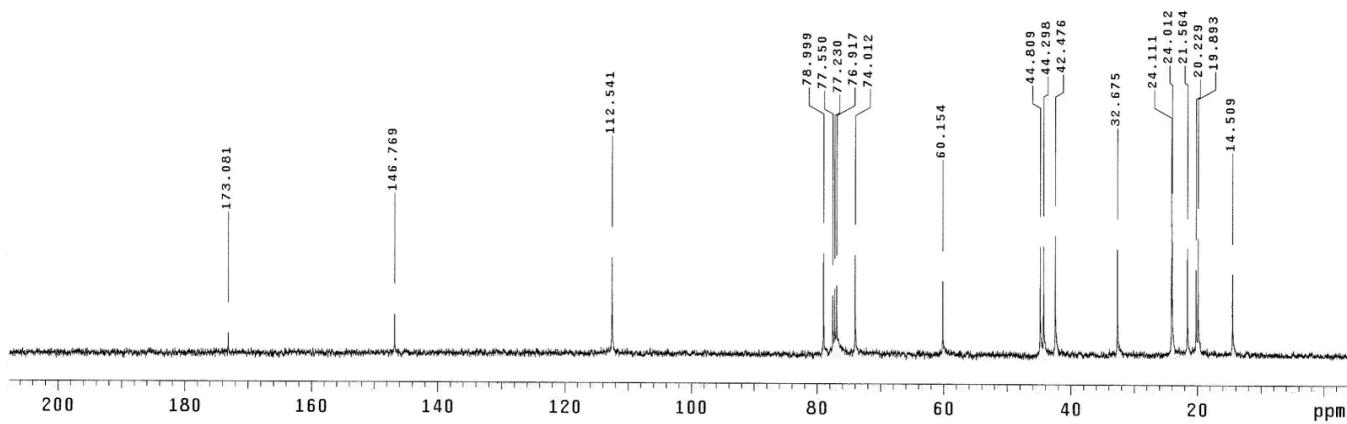
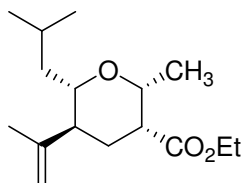


¹³C NMR spectra of 4j

PS_413_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Dec 25 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_413	hst	0.008
	M_C13.fid	pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	4000	lb	2.00
ct	2288	fn	65536
TRANSMITTER	C13	DISPLAY	
tn		sp	-447.5
sfrq	100.554	wp	21328.5
tof	1536.3	rfl	9268.3
tpwr	61	rfp	7764.9
pw	9.300	rp	-57.5
DECOUPLER	H1	lp	-304.2
dn	0	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	22
dpwr	42	th	3
dmf	8900	nm	no ph

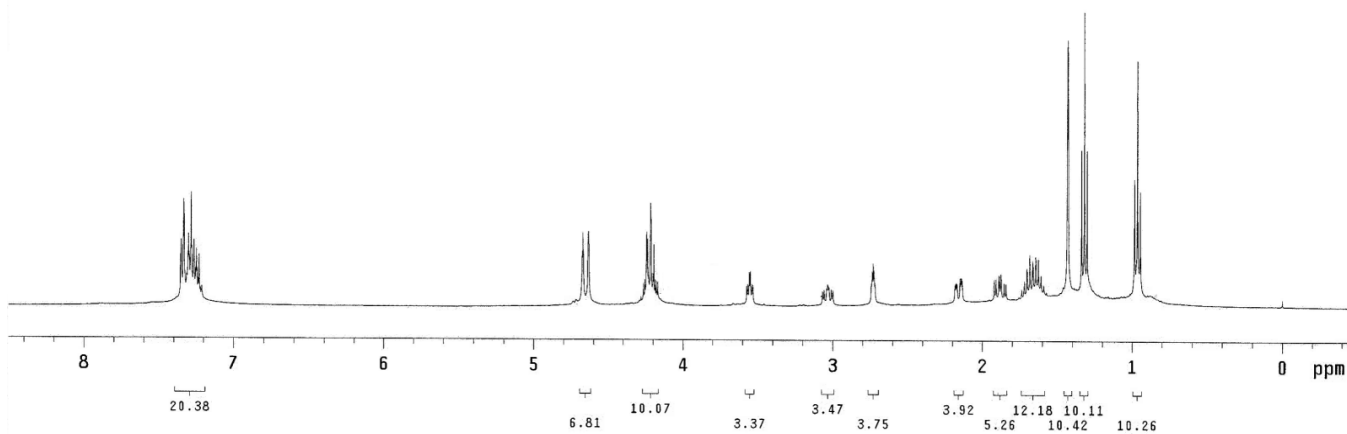
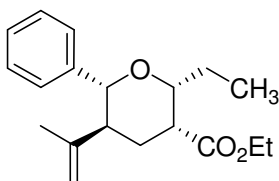


¹H NMR spectra of 4k

PS_456_L

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 27 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_456~	hst	0.008
	~.L.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-184.9
sfrq	399.853	wp	3585.2
tof	362.8	rfl	797.8
tpwr	57	rff	0
pw	9.850	rp	112.6
		lp	-93.9
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	48
dpwr	50	th	20
dmf	15900	nm	cdc ph

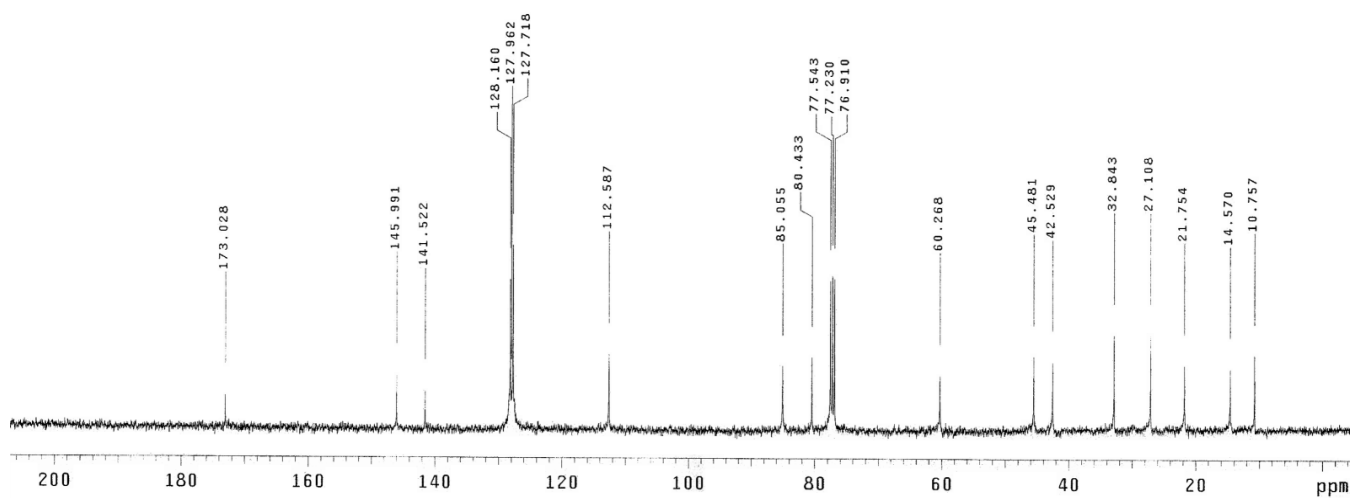
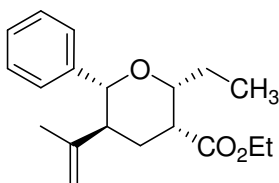


¹³C NMR spectra of 4k

PS_456_L

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 27 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		exp	not used
sw	25125.6	hst	0.008
at	1.199	pw90	18.600
np	60270	alfa	20.000
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	8000	hs	nn
ct	4000	hs	nn
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-516.5
tpwr	81	wp	21328.5
pw	9.300	rfl	9271.3
DECOUPLER		rfp	7764.9
dn	H1	rp	-30.4
dof	0	lp	-376.7
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	30
		th	4
		nm	no ph

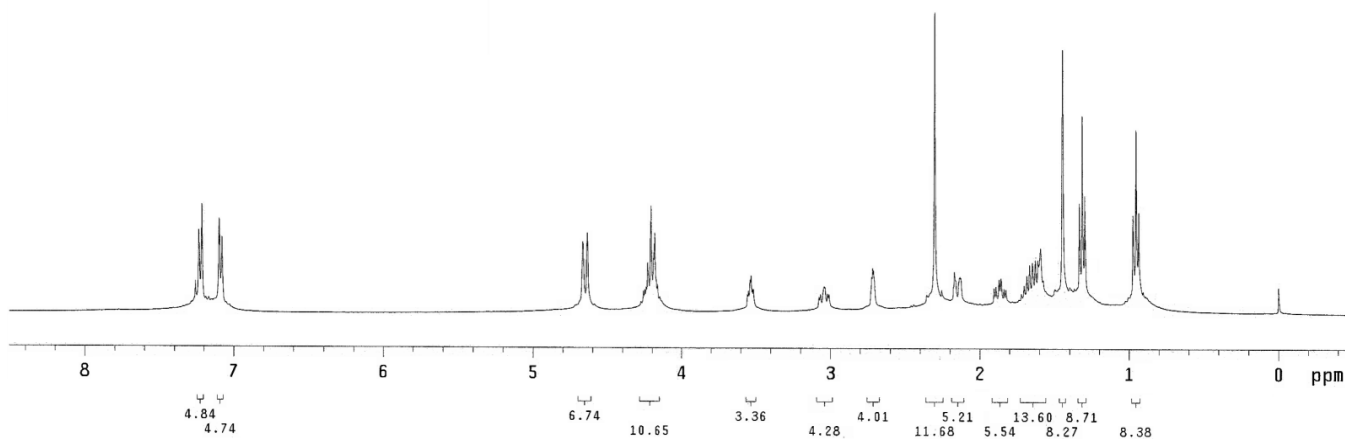
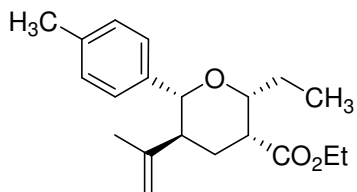


¹H NMR spectra of 4l

PS_464_M

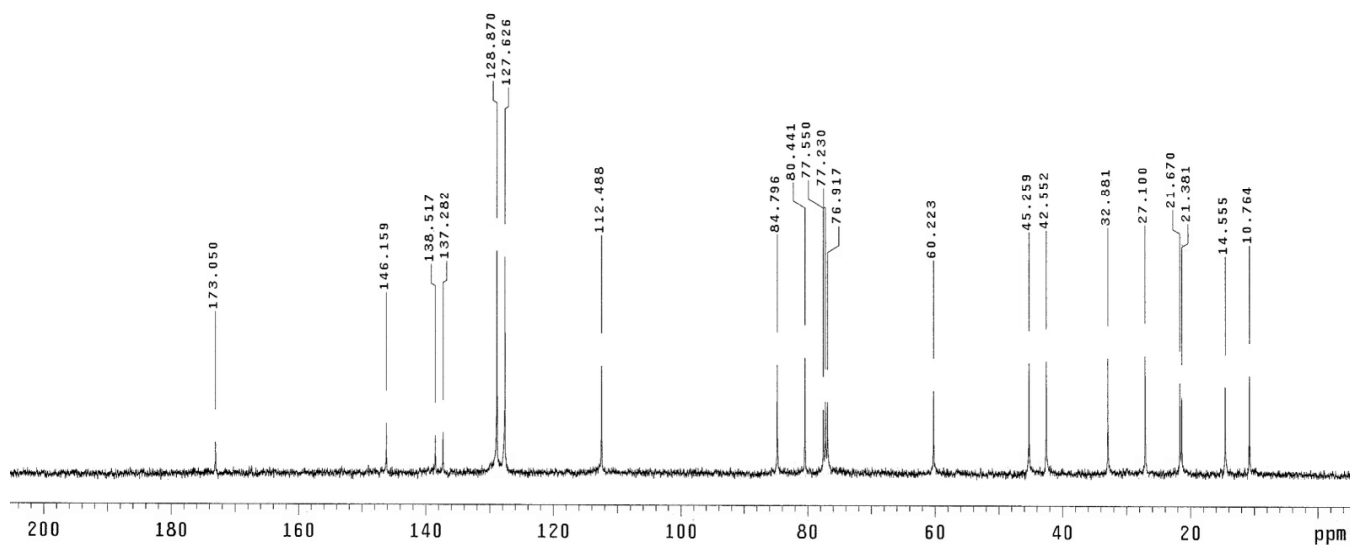
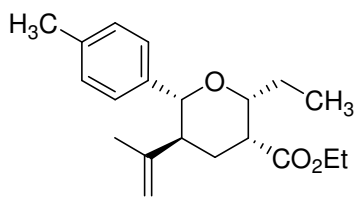
exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 14 2011	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	6389.8	pw90	19.700
at	1.998	alfa	20.000
np	25528	FLAGS	
fb	not used	il	n
bs	8	in	n
d1	1.000	dp	y
nt	100	hs	nn
ct	100	PROCESSING	
tn	TRANSMITTER	lb	0.10
sfrq	H1	fn	65536
tof	399.853	DISPLAY	
tpwr	362.8	sp	-191.1
pw	57	wp	3593.6
dn	9.850	rfl	795.6
dof	0	rfd	0
dmm	C13	rp	114.9
dpwr	50	lp	-75.0
dmf	15900	PLOT	
		wc	250
		sc	0
		vs	50
		th	14
		nm	cdc ph

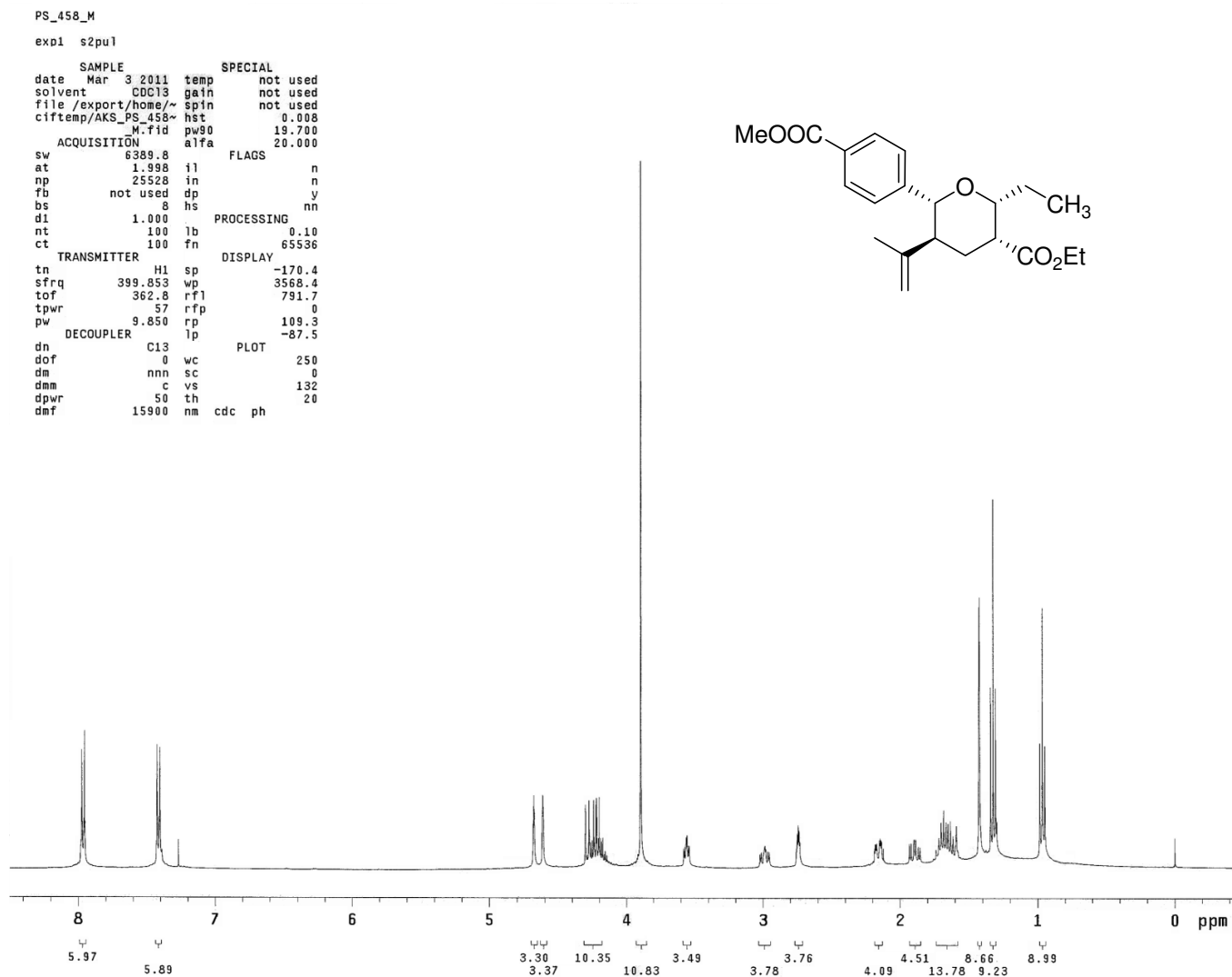


¹³C NMR spectra of 4l

```
PS_464_M
expl s2pu1
SAMPLE          SPECIAL
date Mar 14 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION hst 0.008
sw 25125.6 pw90 18.600
at 1.199 alfa 20.000
np 60270
fb 13800 fl n
bs 16 in n
dl 1.000 dp y
nt 4000 hs nn
ct 2544
TRANSMITTER lb 2.00
tn C13 fn 65536
sfrq 100.554
tof 1536.3
tpwr 61 wp -551.0
pw 9.300 rfl 9272.9
DECOUPLER rfp 7764.9
dn H1 rp -64.5
dof 0 lp -297.0
dm yyy
dmm w wc 250
dpwr 42 sc 0
dmf 8900 vs 34
th 3
nm no ph
```

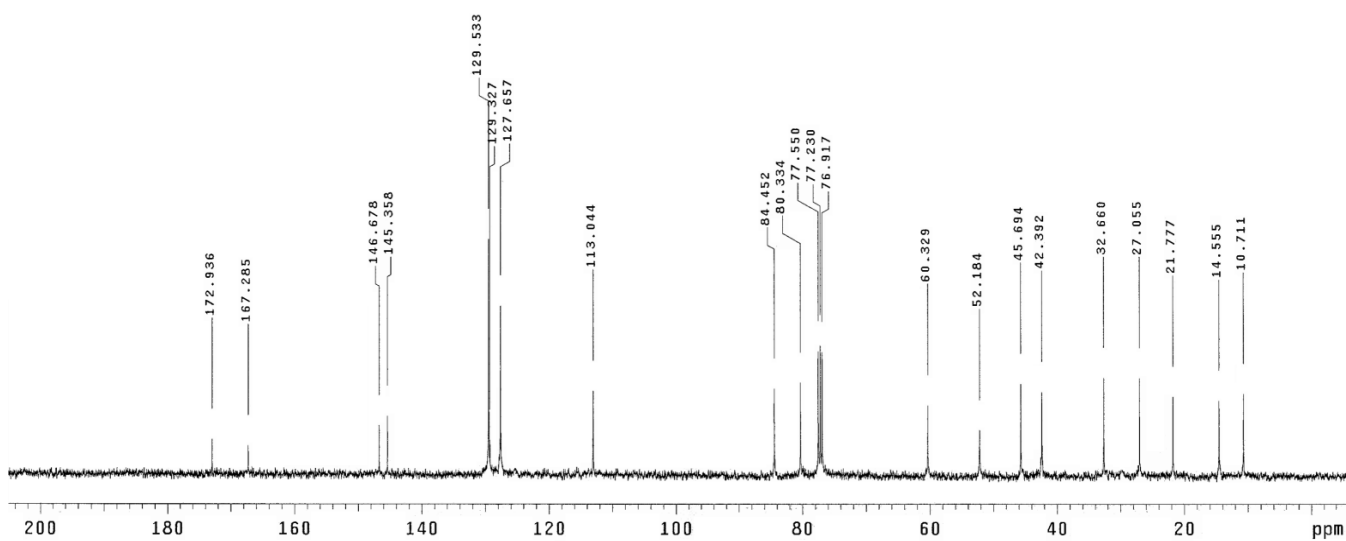
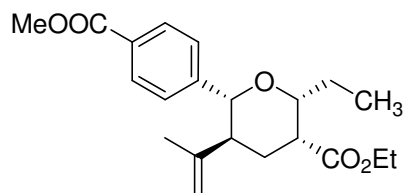


¹H NMR spectra of 4m



¹³C NMR spectra of 4m

```
PS_458_M
exp1 s2pu1
SAMPLE
date Mar 4 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION
sw 25125.6 hst 0.008
at 1.199 pw90 18.600
np 60270 alfa 20.000
fb 13800 FLAGS n
bs 16 in n
dl 1.000 dp y
nt 5000 hs nn
ct 3056 PROCESSING
tn TRANSMITTER lb 2.00
C13 fn 65536
sfrq 100.554 DISPLAY
tof 1536.3 sp -615.4
tpwr 61 wp 21229.5
pw 9.300 rfl 9271.3
DECOUPLER rfp 7764.9
dn H1 rp -51.1
dof 0 lp -334.6
dm vvyv PLOT
dmm w wc 250
dpwr 42 sc 0
daf 8900 vs 39
th 4
nm no ph
```

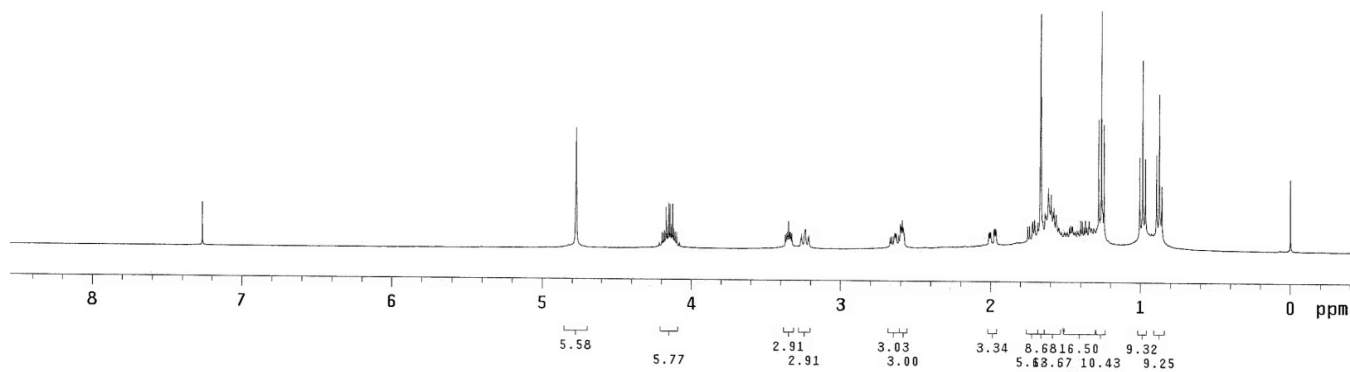
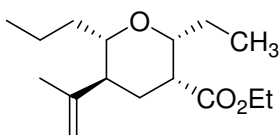


¹H NMR spectra of 4n

PS_463_L

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 12 2011	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	6389.8	pw90	19.700
at	1.998	alfa	20.000
np	25528	FLAGS	
fb	not used	il	n
bs	8	in	n
d1	1.000	dp	y
nt	200	hs	nn
ct	192	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	65536
sfrq	399.853	DISPLAY	
tof	362.8	sp	-172.2
tpwr	57	wp	3593.6
pw	9.850	rfl	793.5
DECOUPLER		rffp	0
dn	C13	rp	117.4
dof	0	lp	-94.6
dm	nnn	PLOT	
dmm	c	wc	250
dpwr	50	sc	0
dmf	15900	vs	40
		th	33
		nm	cdc ph

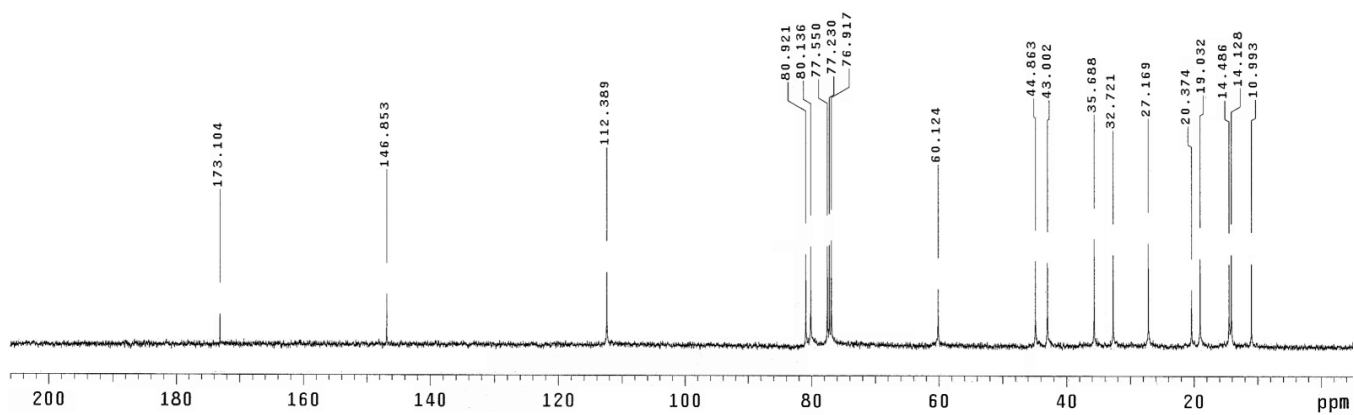
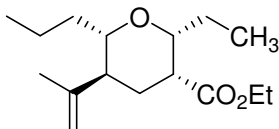


¹³C NMR spectra of 4n

PS_463_L

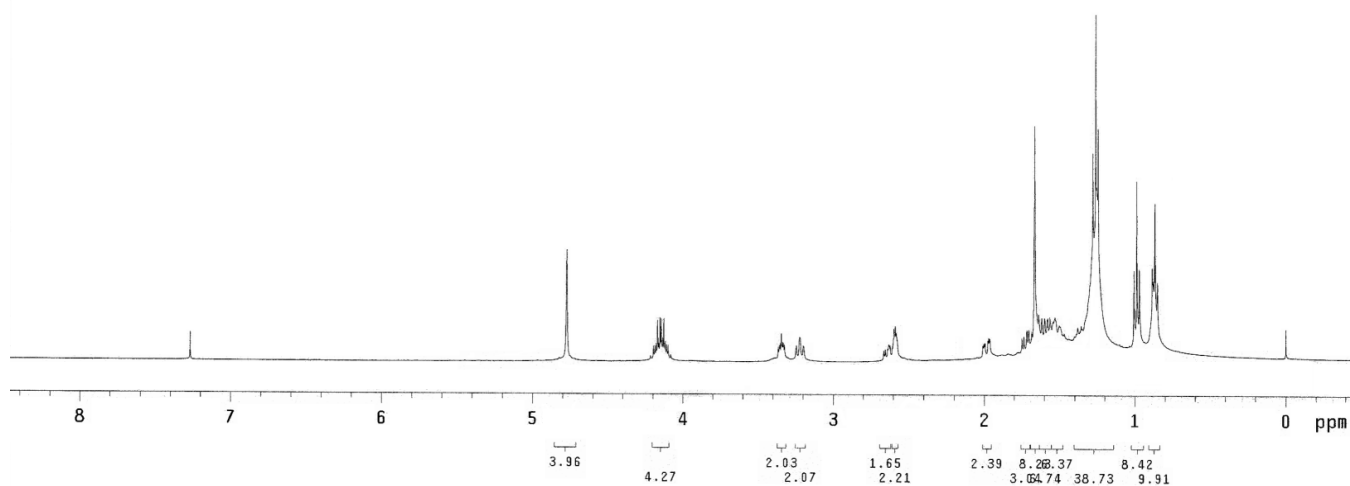
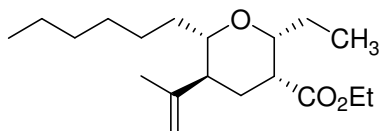
exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 12 2011	temp	not used
solvent	CDC13	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	5000	hs	nn
ct	3216	PROCESSING	
TRANSMITTER		lb	2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-547.2
tpwr	61	wp	21262.5
pw	9.300	rfl	9269.0
DECOUPLER		rffp	7784.3
dn	H1	rp	-54.9
dof	0	lp	-339.8
dm	VVY	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	17
		th	3
		nm	no ph



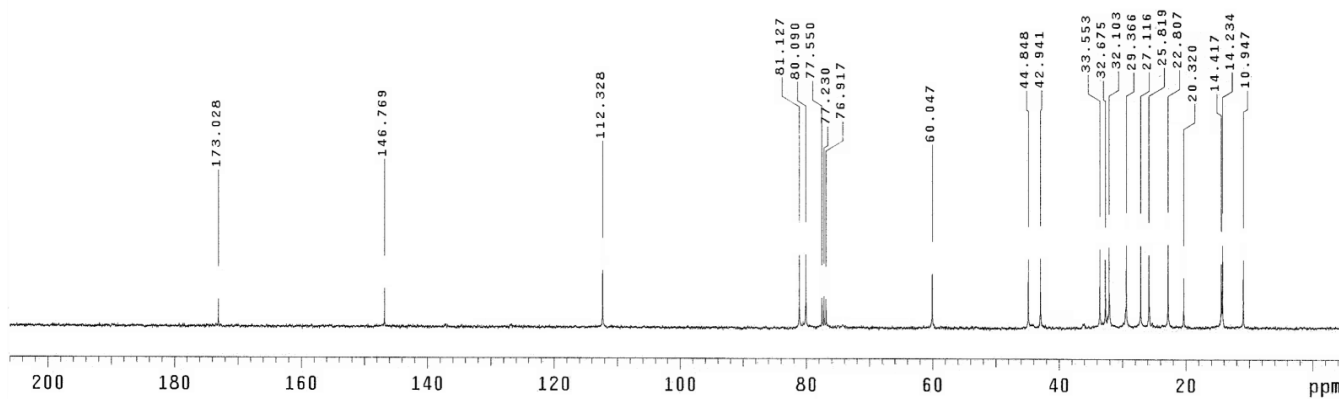
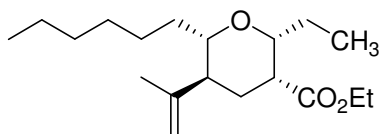
¹H NMR spectra of 4o

```
PS_469_M
exp1 s2pu1
SAMPLE
date Mar 27 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION hst 0.008
sw 6389.8 pw90 19.700
at 1.998 alfa 20.000
np 25528
fb not used il n
bs 8 in n
dl 1.000 dp y
nt 100 hs nn
ct 100
TRANSMITTER lb 0.10
tn H1 fn 65536
sfrq 399.853
tof 362.8 sp DISPLAY -180.4
tpwr 57 wp 3568.4
pw 9.850 rfl 793.3
DECOUPLER rfp 0
dn C13 rp 97.7
dof 0 lp -58.8
dm nnn PLOT
dmm c wc 250
dpwr 50 sc 0
dmf 15900 vs 57
nm cdc ph th 20
```



¹³C NMR spectra of 4o

```
PS_469_M
expl s2pu1
SAMPLE
date Mar 27 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION
sw 25125.6 hst 0.008
at 1.199 pwa 18.600
np 60270 alfa 20.000
fb 13800 il n
bs 16 fn n
d1 1.000 dp y
nt 4000 hs nn
ct 672
TRANSMITTER
tn C13 lb 2.00
sfrq 100.554 fn 65536
tof 1536.3 sp DISPLAY -551.0
tpwr 61 wp 21262.5
pw 9.300 rfl 9272.9
DECOUPLER rfp 7764.9
dn H1 rp -51.7
dof 0 lp -331.1
dm vvy PLOT
dmm w wc 250
dpwr 42 sc 0
dmf 8900 vs 13
th 4
nm no ph
```

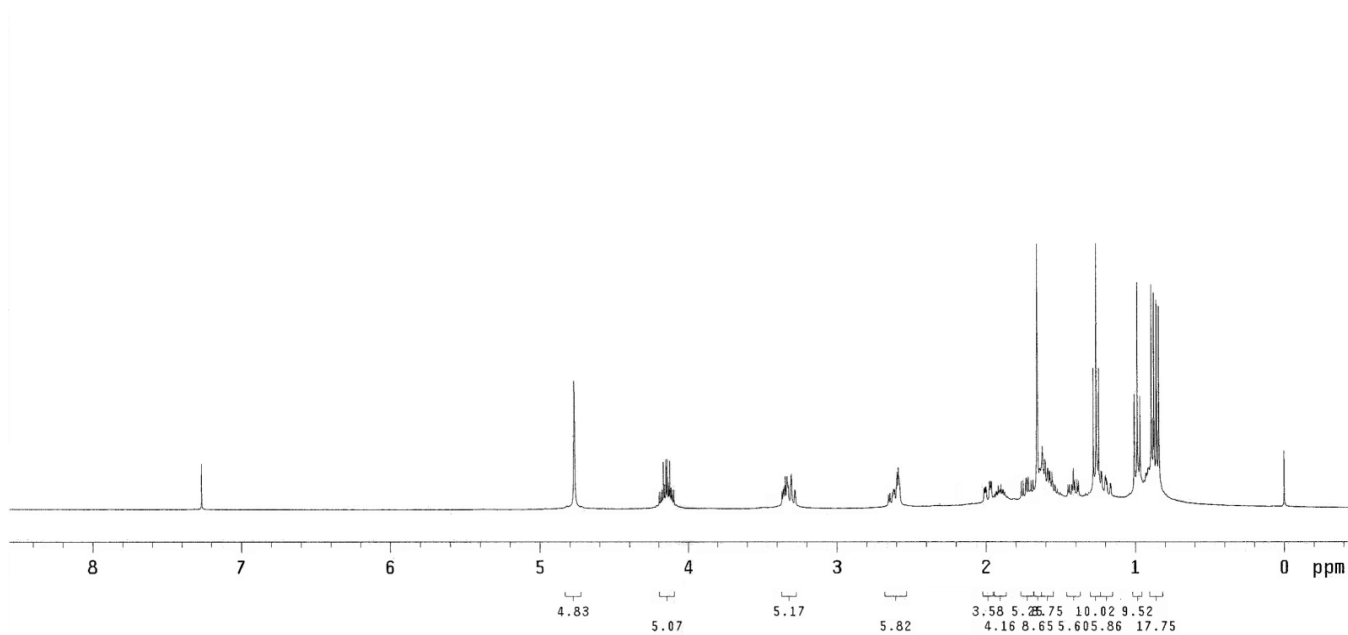
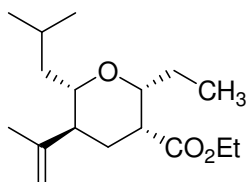


¹H NMR spectra of 4p

PS_467_M

expl s2pu1

```
SAMPLE          SPECIAL
date Mar 22 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_467~ hst 0.008
_M.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8 FLAGS
at 1.998 il n
np 25528 in n
fb not used dp y
bs 8 hs nn
d1 1.000 PROCESSING
nt 100 lb 0.10
ct 100 fn 65536
TRANSMITTER H1 sp DISPLAY
tn -179.4
sfrq 399.853 wp 3602.0
tof 362.8 rfl 792.3
tpwr 57 rfp 0
pw 9.850 rp 106.8
DECOUPLER lp -85.7
dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 43
dpwr 50 th 27
dmf 15900 nm cdc ph
```

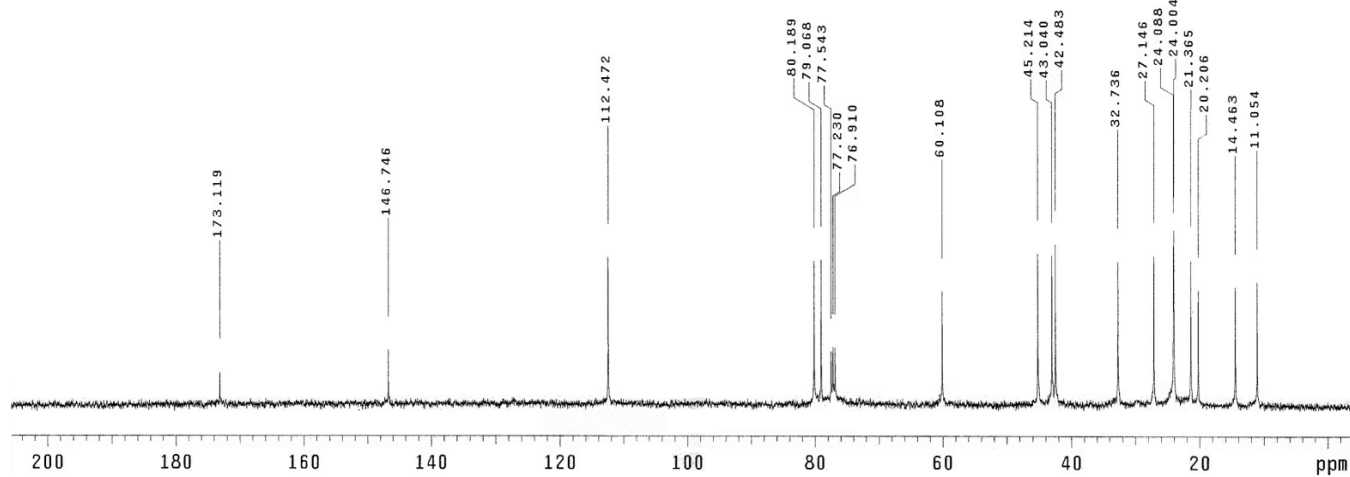
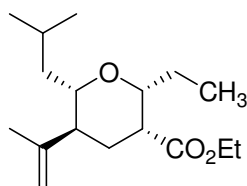


¹³C NMR spectra of 4p

PS_467_M

expl s2pu1

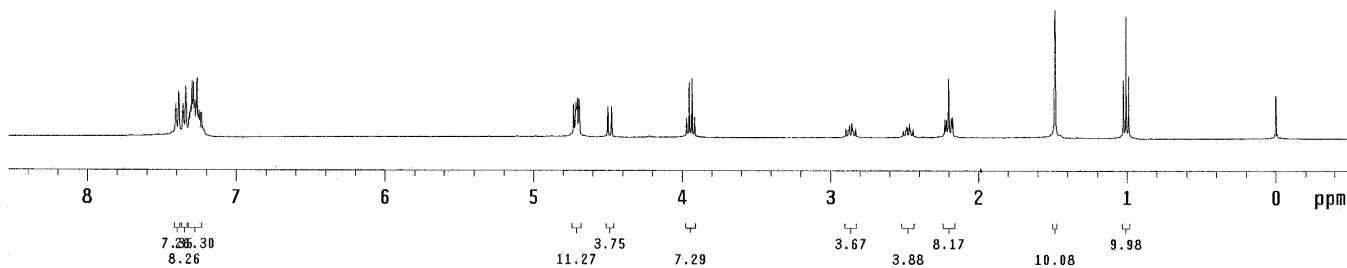
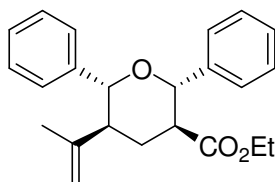
SAMPLE		SPECIAL	
date	Mar 22 2011	temp	not used
solvent	CDC13	gain	not used
file		spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	5000	hs	nn
ct	2528	PROCESSING	
TRANSMITTER	lb		2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-416.1
tpwr	61	wp	21096.9
pw	9.300	rfl	9269.8
DECOUPLER	rfl	rfl	7764.9
dn	H1	rp	-46.5
dof	0	lp	-338.2
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	27
		th	4
		nm	no ph



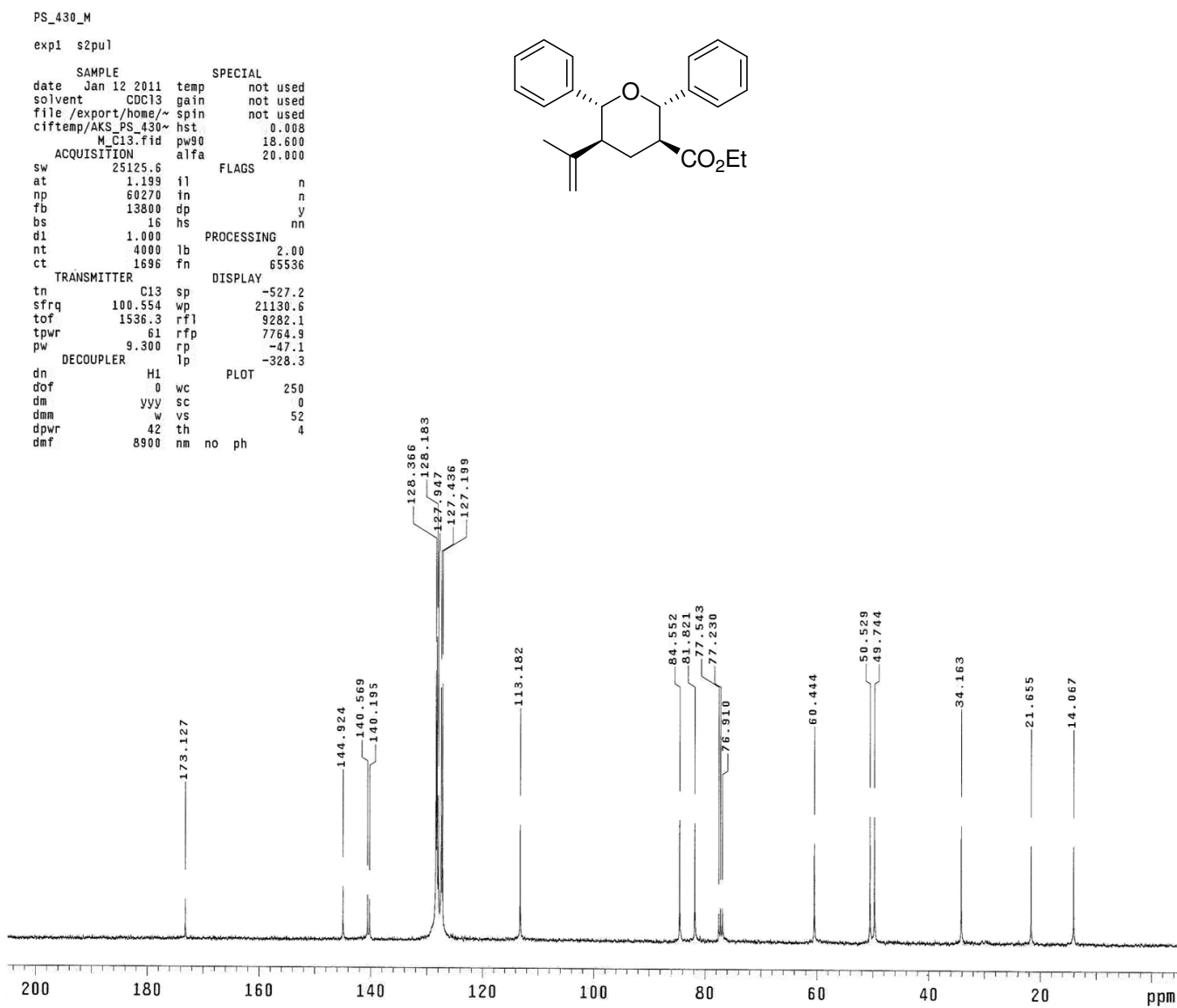
¹H NMR spectra of 11a

expl s2pu1

SAMPLE		SPECIAL	
date	Sep 7 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_430~	hst	0.008
	M_Pdt.fid	pw90	19.700
ACQUISITION		alfa	20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	200	fn	not used
ct	164	DISPLAY	
TRANSMITTER		sp	-207.1
tn	H1	wp	3618.7
sfrq	399.853	rfl	794.5
tof	362.8	rfp	0
tpwr	57	rp	135.4
pw	9.850	lp	-84.7
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	61
dmm	c	th	8
dpwr	50	nm	ph
dmf	15900		



^{13}C NMR spectra of 11a

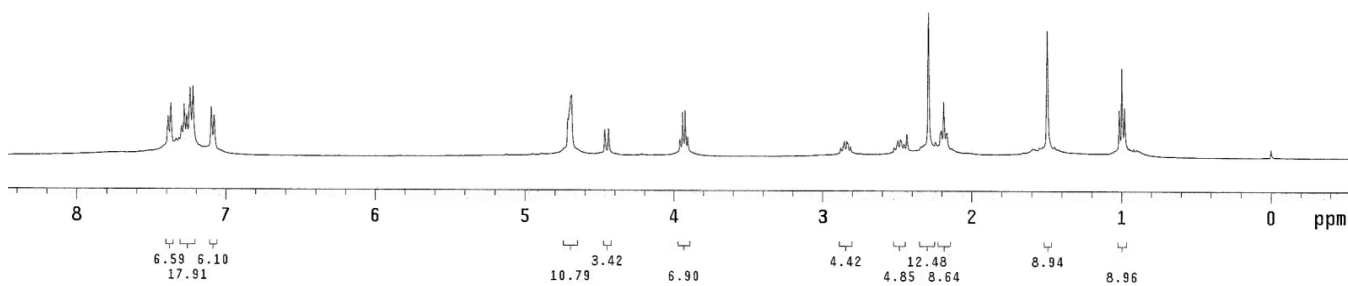
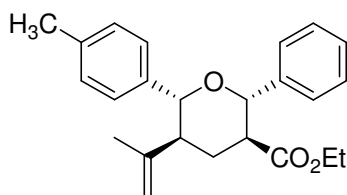


¹H NMR spectra of 11b

PS_431_M

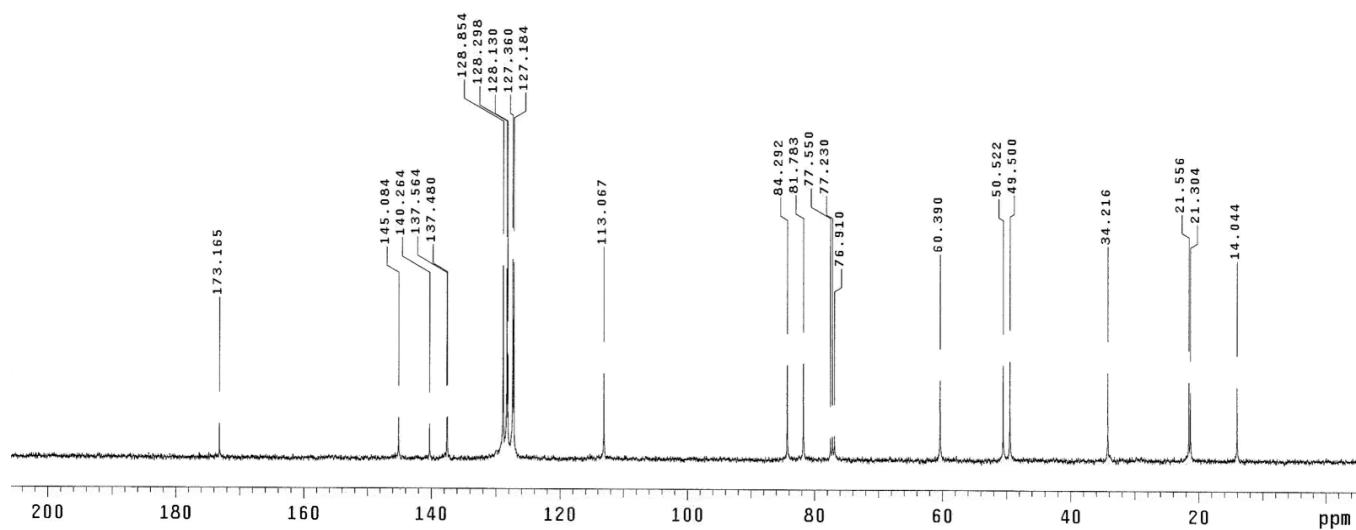
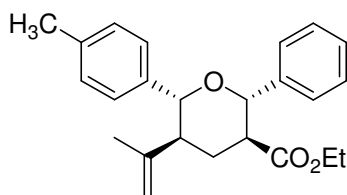
exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 12 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_431~	hst	0.008
	M.fid	pw90	19.700
	alfa		20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	fl	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-219.4
sfrq	399.853	wp	3602.0
tof	362.8	rfl	788.6
tpwr	57	rfp	0
pw	9.850	rp	109.2
	lpc		-82.3
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	23
dpwr	50	th	24
dmf	15900	nm	cdc ph



^{13}C NMR spectra of 11b

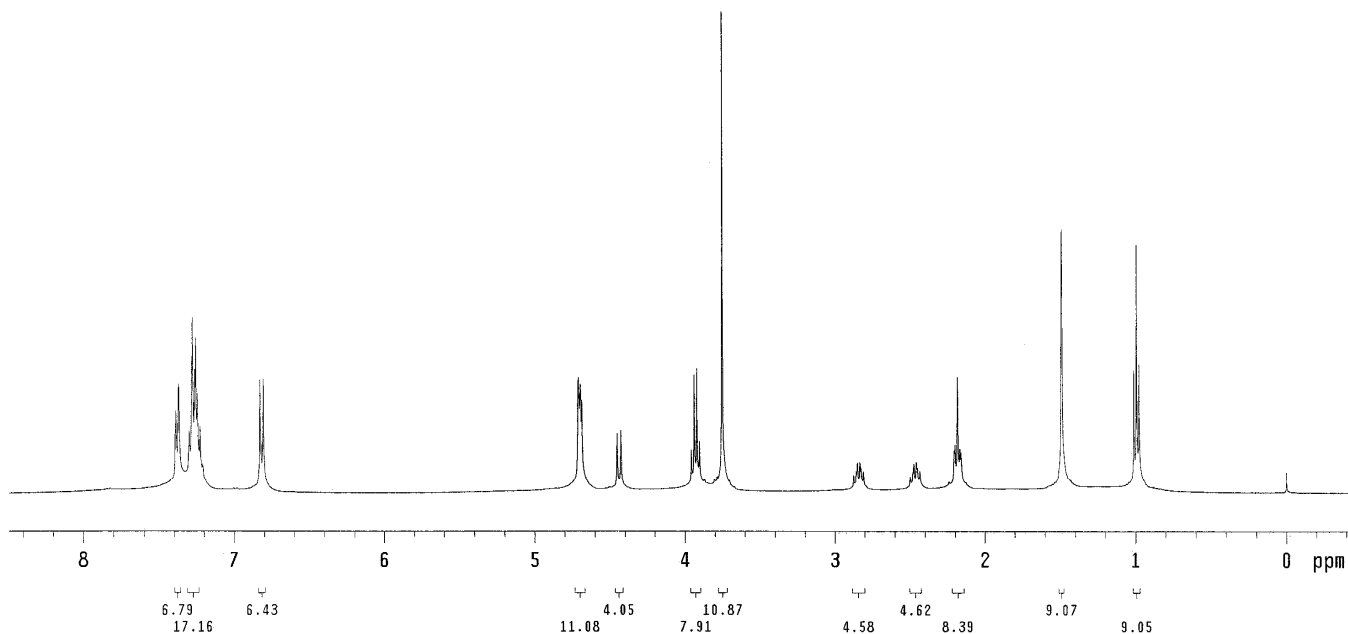
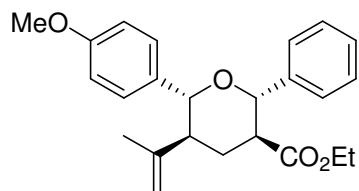
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PS_431_M
exp1 s2pu1
SAMPLE
date Jan 12 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_431~ hst 0.008
M_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 2000 lb 2.00
ct 512 fn 65536
TRANSMITTER DISPLAY
tn C13 sp -495.8
sfrq 100.554 wp 21196.6
tof 1536.3 rfl 9283.6
tpwr 61 rfp 7764.9
pw 9.300 rp -48.7
DECOUPLER lp -324.1
dn H1 PLOT
dof 0 wc 250
dm vyy sc 0
dmm w vs 31
dpwr 42 th 3
dmf 8900 nm no ph
```



¹H NMR spectra of 11c

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jun 9 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_494~		hst	0.008
M_Pdt.fid		pw90	19.700
ACQUISITION	alfa		20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	4	hs	nn
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER	H1	DISPLAY	
tn		sp	-171.8
sfrq	399.853	wp	3568.4
tof	362.8	rfl	801.5
tpwr	57	rffp	0
pw	9.850	rp	119.8
DECOUPLER	1p		-91.4
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	69
dpwr	50	th	42
dmf	15900	nm	cdc ph

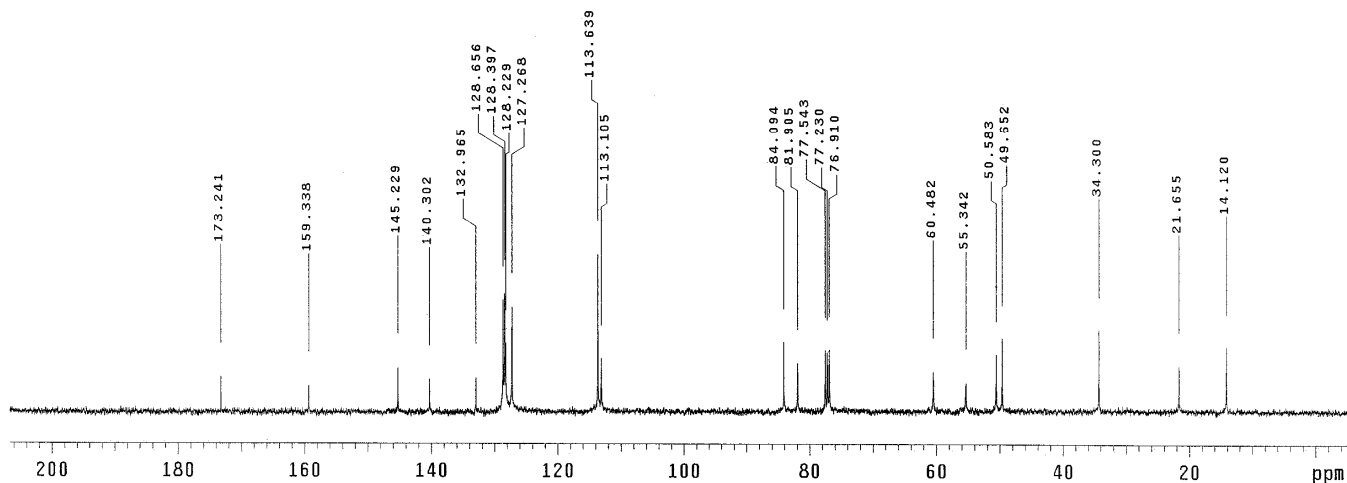
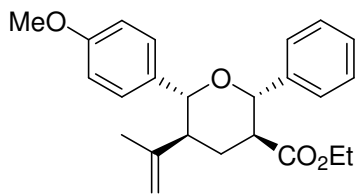


¹³C NMR spectra of 11c

PS_494_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jun 9 2011	temp	not used
solvent	CDCl3	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	fl	n
bs	16	in	n
d1	1.000	dp	y
nt	4000	hs	nn
ct	1552	PROCESSING	
tn	C13	lb	2.00
sfrq	100.554	fn	65536
tof	1536.3	sp	-551.8
tpwr	61	wp	21328.5
pw	9.300	rfl	9273.6
DECOUPLER		rffp	7764.9
dn	H1	rp	-47.4
dof	0	lp	-349.1
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	24
		th	3
		nm	no ph

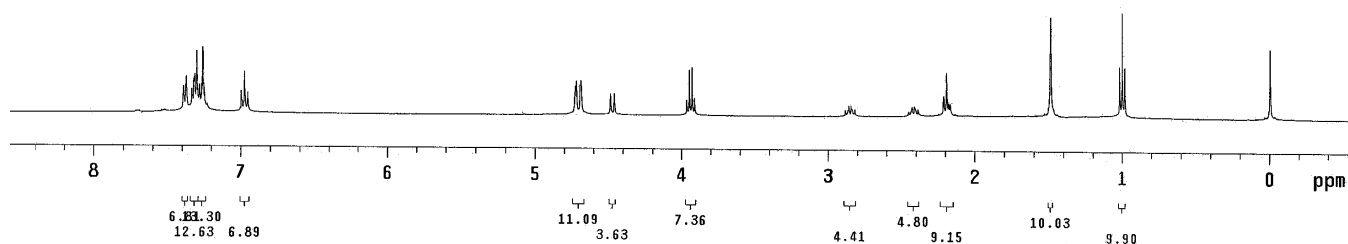
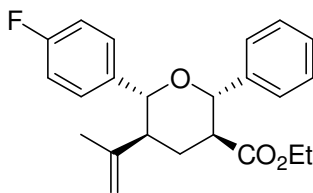


¹H NMR spectra of 11d

PS_435_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Sep 12 2011	temp	not used
solvent	CDCl3	gain	not used
file /export/home/~		spin	not used
ciftemp/AKS_PS_435~		hst	0.008
M1_Pdt.fid		pw90	19.700
ACQUISITION		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8	il	n
at	1.998	in	n
np	25528	dp	y
fb	not used	hs	nn
bs	8	PROCESSING	
d1	1.000	lb	0.10
nt	300	fn	65536
ct	256	DISPLAY	
TRANSMITTER		tn	H1 sp -224.5
sfrq	399.853	wp	3652.5
tof	362.8	rfl	795.2
tpwr	57	rfp	0
pw	9.850	rp	119.0
DECOUPLER		lp	-88.5
dn	C13	PLOT	
dof	0	wc	250
dm	^nn	sc	0
dmm	c	vs	49
dpwt	50	th	11
dmf	15900	nm	cdc ph

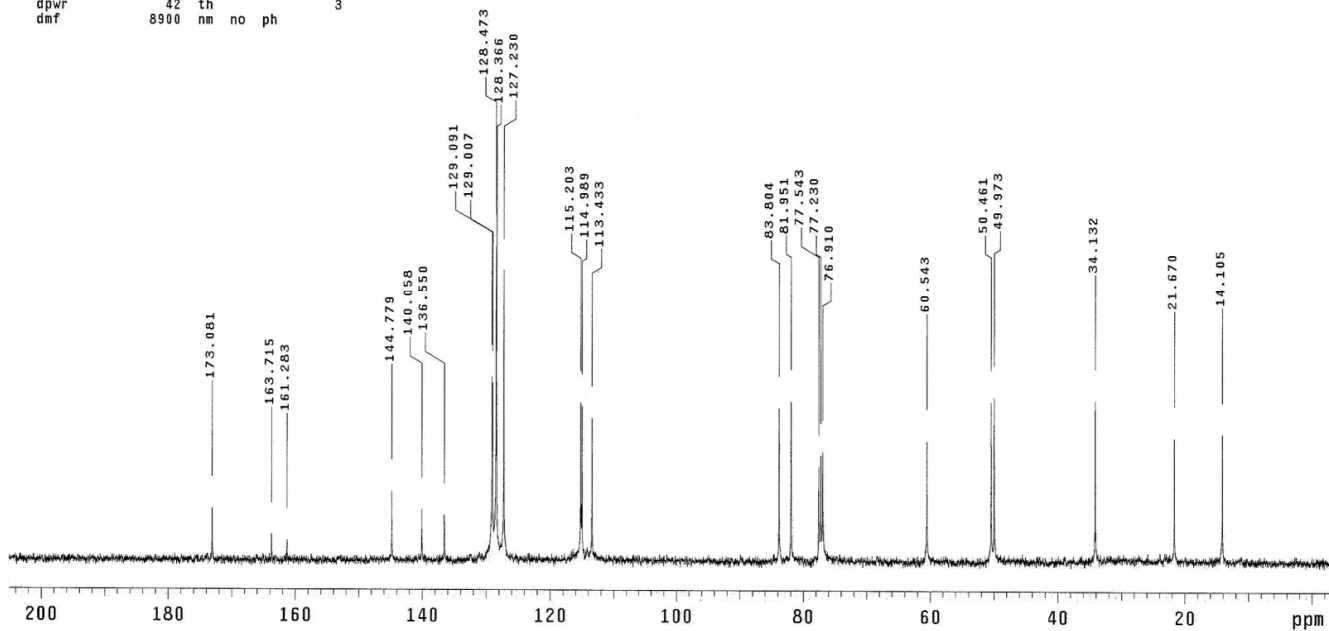
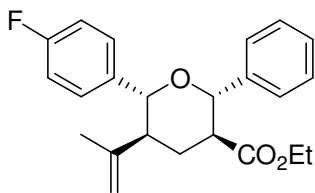


¹³C NMR spectra of 11d

PS_435_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 24 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_435	hst	0.008	
M1_C13.fid	pw90	18.600	
ACQUISITION	alfa	20.000	
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	5000	lb	2.00
ct	3776	fn	65536
TRANSMITTER	C13	DISPLAY	
tn	-285.7		
sfrq	100.554	wp	20889.0
tof	1536.3	rfl	9272.1
tpwr	61	rfd	7764.9
pw	9.300	rp	-48.7
DECOUPLER	lp		-332.1
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	49
dpwr	42	th	3
dmf	8900	nm	no ph

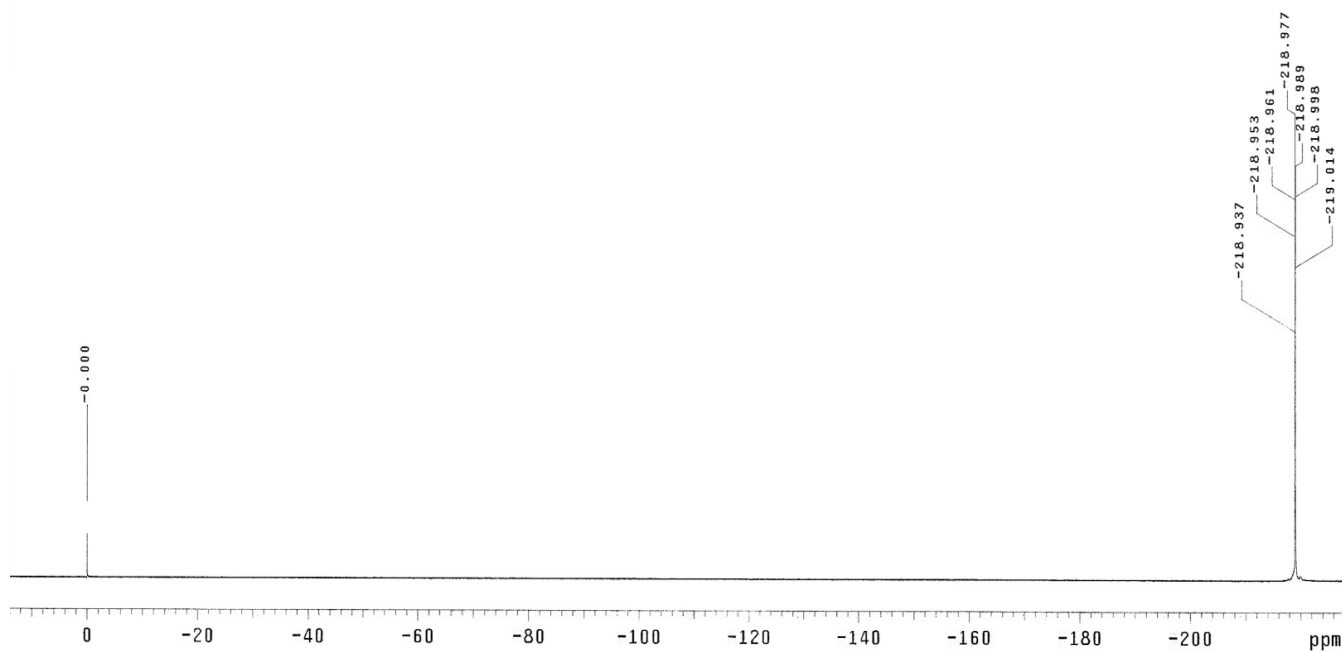
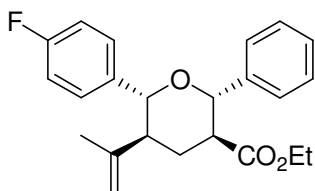


¹⁹F NMR spectra of 11d

PS_453M_Fluprine

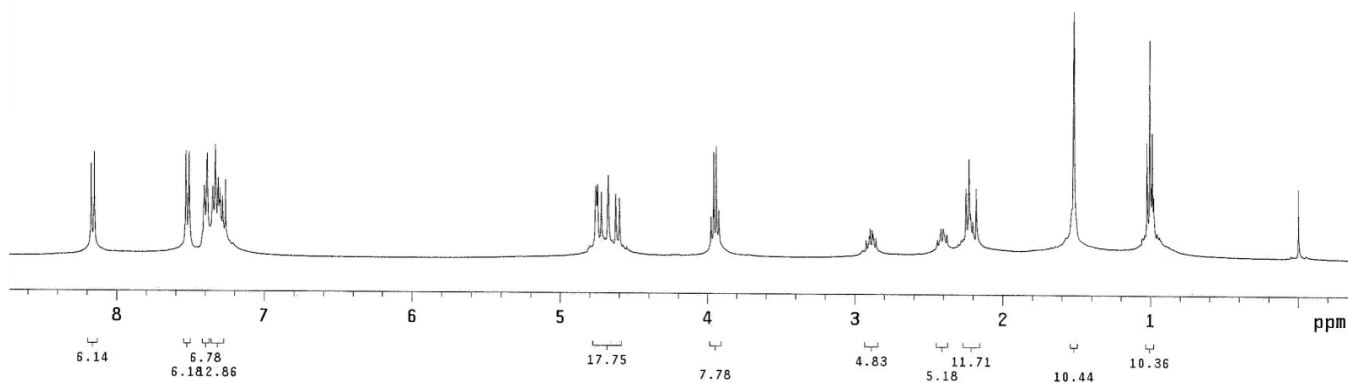
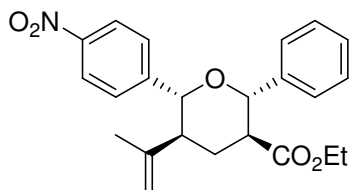
exp1 s2pu1

SAMPLE		SPECIAL	
date	Apr 4 2011	temp	not used
solvent	CDC13	gain	not used
file		sp1n	not used
ACQUISITION		hst	0.008
sw	100000.0	pw90	15.600
at	0.600	alfa	90.000
np	119936	FLAGS	
fb	55000	il	n
bs	16	in	n
dl	1.500	dp	y
nt	64	hs	nn
ct	64	PROCESSING	
TRANSMITTER		lb	0.30
tn	F19	lsfid	-10
sfrq	376.236	fn	not used
tof	40099.4	DISPLAY	
tpwr	58	sp	-85986.1
pw	5.200	wp	91326.8
DECOUPLER		rf1	89007.4
dn	H1	rfl	0
dof	0	rp	-119.6
dm	nnn	lp	-9.0
dmm	c	PLOT	
dpwr	42	wc	250
dmf	8900	sc	0
		vs	11
		th	5
		ai	no ph



¹H NMR spectra of 11e

```
PS_450_M
expl s2pu1
SAMPLE
date Feb 20 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_450~ hst 0.008
_M.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8 FLAGS
at 1.998 f1 n
np 25528 in n
fb not used dp y
bs 8 hs nn
d1 1.000 PROCESSING
nt 200 lb 0.10
ct 0 fn 65536
TRANSMITTER H1 sp DISPLAY
tn -146.6
sfrq 399.853 wp 3635.7
tof 382.8 rfl 793.1
tpwr 57 rfp 0
pw 9.850 rp 109.0
DECOUPLER lp -81.3
dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 42
dpwr 50 th 20
dmf 15900 nm cdc ph
```

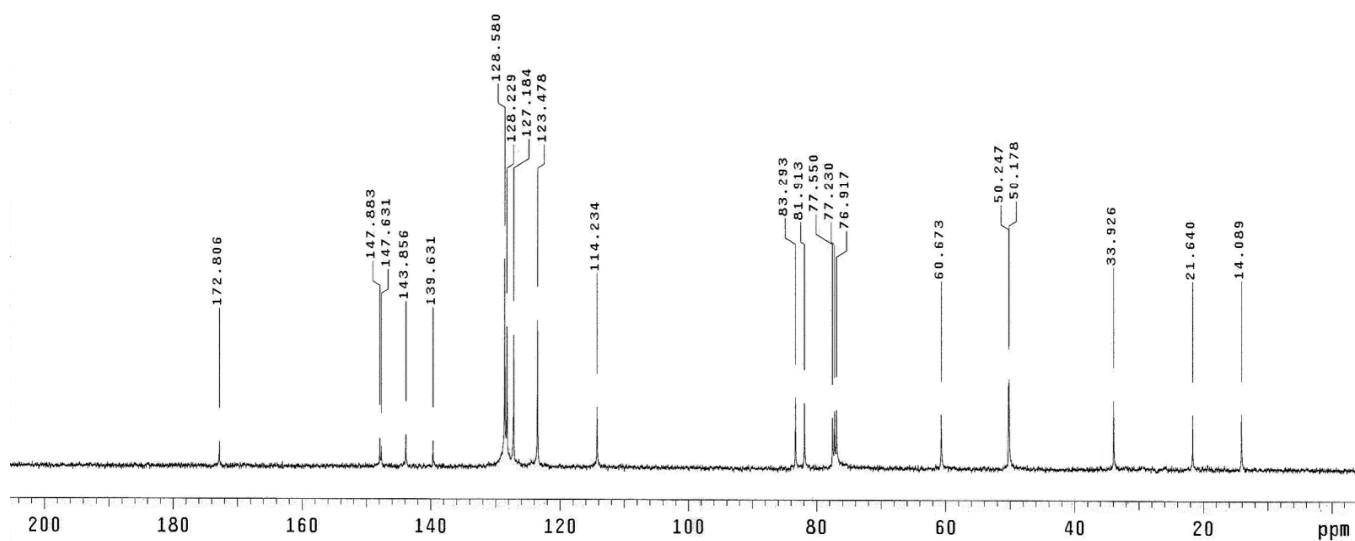
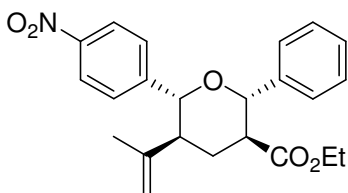


¹³C NMR spectra of 11e

PS_450_M

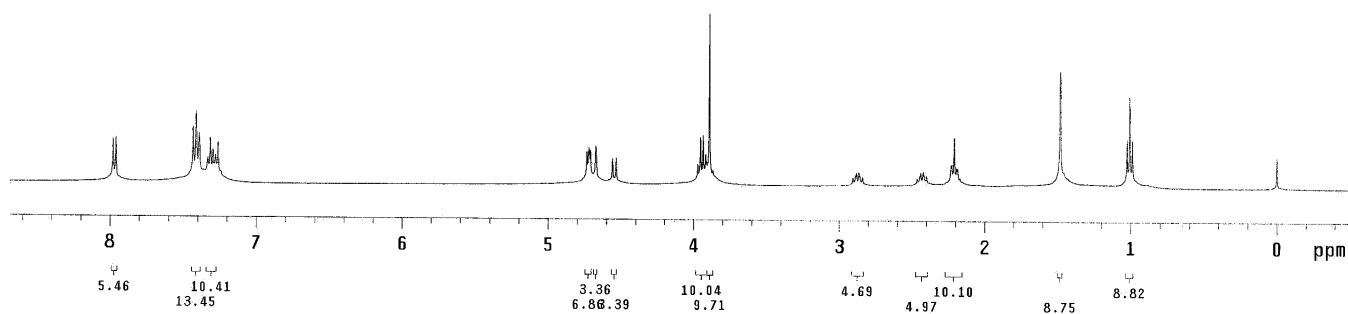
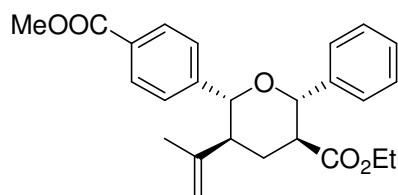
exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 20 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_450~	hst	0.008
	M_C13.fid	pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	5000	lb	2.00
ct	0	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-384.6
sfrq	100.554	wp	21030.9
tof	1536.3	rfl	9271.3
tpwr	61	rpf	7764.9
pw	9.300	rp	-57.6
DECOUPLER		lp	-312.0
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	31
dpwr	42	th	2
dmf	8900	nm	no ph



¹H NMR spectra of 11f

```
PS_418_U
exp1 s2pu1
SAMPLE
date Sep 8 2011 temp not used
solvent CDCl3 gain not used
file exp spin not used
ACQUISITION hst 0.008
sw 6389.8 pw90 19.700
at 1.998 alfa 20.000
np 25528
fb not used il FLAGS n
bs 8 in n
dl 1.000 dp y
nt 200 hs nn
ct 144 PROCESSING
TRANSMITTER lb 0.10
tn H1 fn 65536
sfrq 399.853 DISPLAY
tof 362.8 sp -206.9
tpwr 57 wp 3686.0
pw 9.850 rfi 794.7
DECOUPLER rfp 0
dn C13 rp 124.3
dof 0 lp -82.2
dm nnn PLOT
dmm c wc 250
dpwr 50 sc 0
dmf 15900 vs 28
nm th 20
cdc ph
```

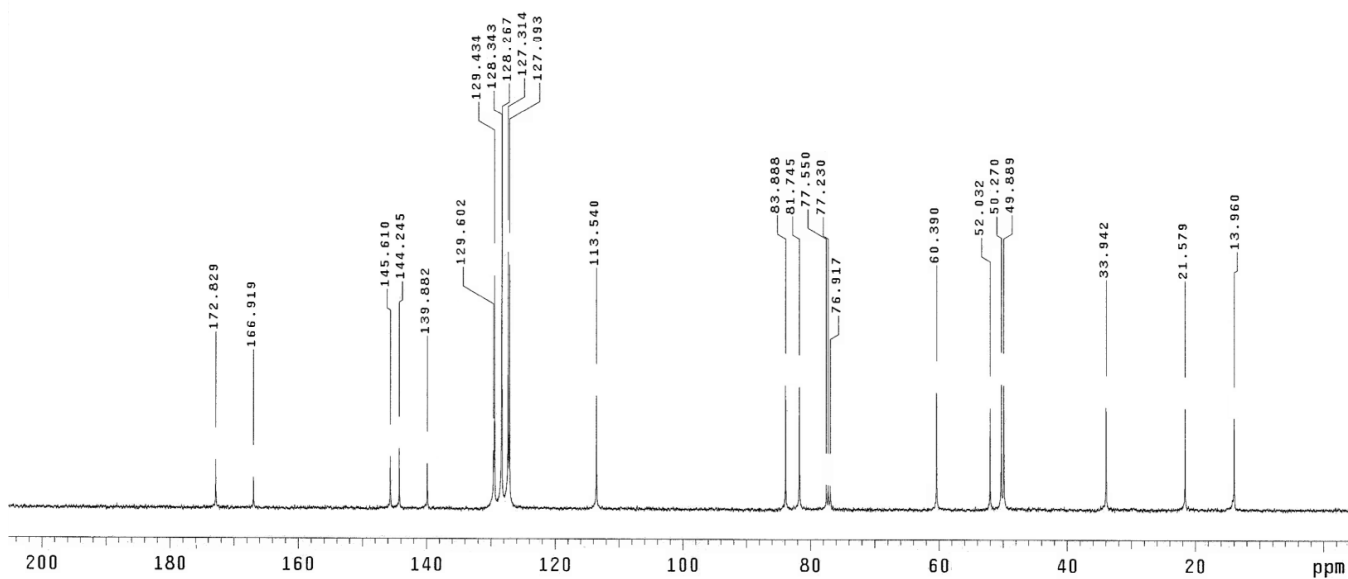
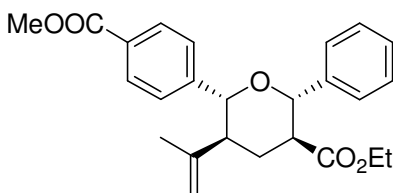


¹³C NMR spectra of 11f

PS_418_U

exp1 s2pu1

SAMPLE		SPECIAL	
date	Mar 20 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION		hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	4000	hs	nn
ct	576	PROCESSING	
tn		lb	2.00
sfrq	100.625	fn	65536
tof	1536.3	sp	-430.6
tpwr	61	wp	21063.9
pw	9.300	rfl	9284.4
DECOUPLER		rff	7764.9
dn	H1	rp	-38.2
dof	0	lp	-336.3
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	40
		th	3
		nm	no ph

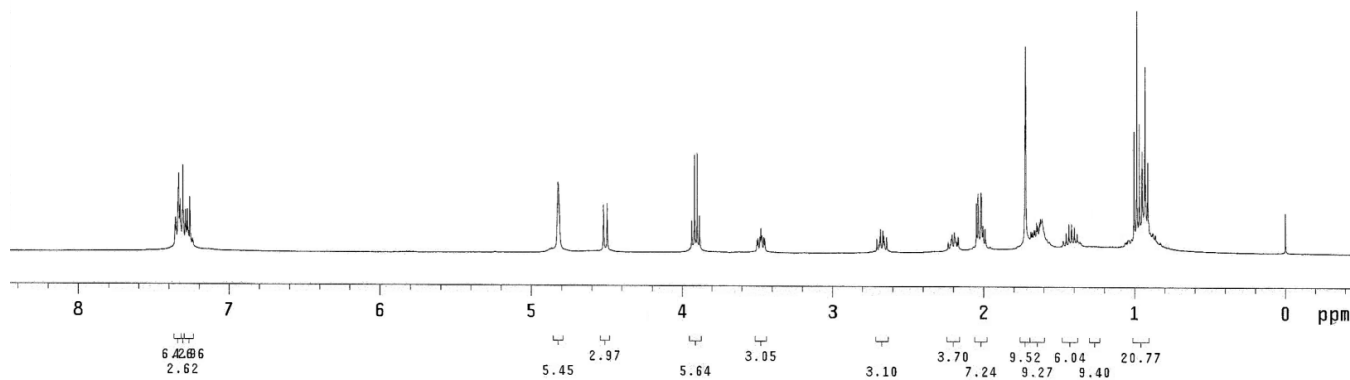
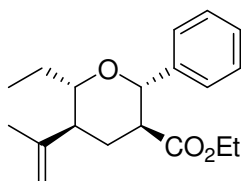


¹H NMR spectra of 11g

PS_424_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 5 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_424		hst	0.008
	_M.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-188.6
sfrq	399.853	wp	3568.4
tof	362.8	rfl	793.1
tpwr	57	rff	0
pw	9.850	rp	103.0
		lp	-90.3
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	39
dpwr	50	th	20
dmf	15900	nm	cdc ph



¹³C NMR spectra of 11g

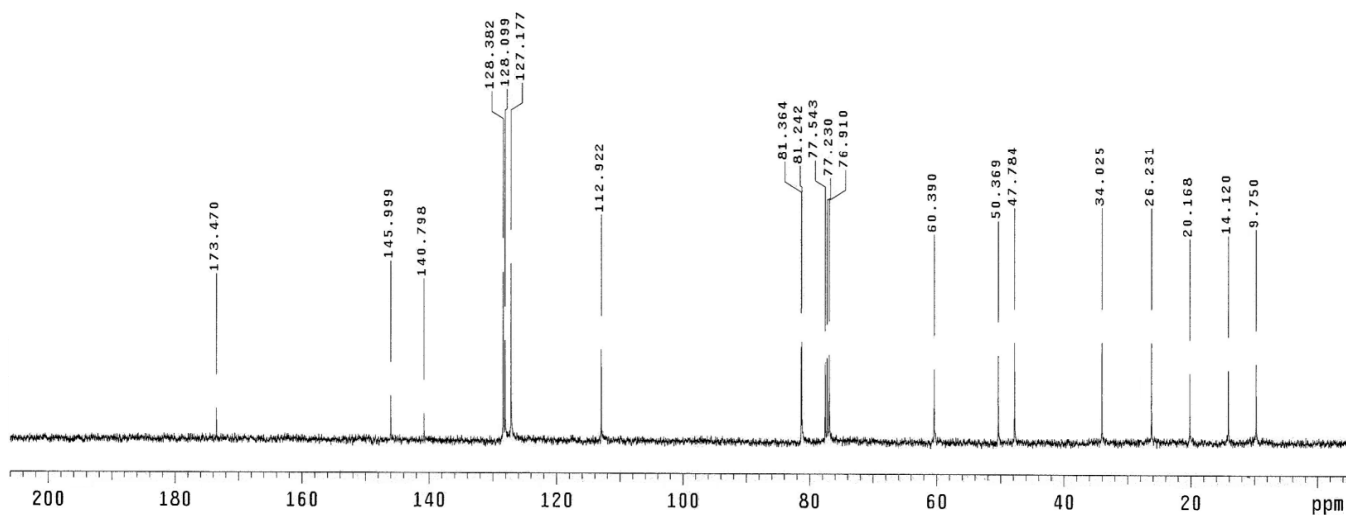
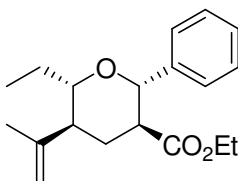
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PS_424_M
exp1 s2pu1

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solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_424~ hst 0.008
M_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6
at 1.199 i1
np 60270 in
fb 13800 dp
bs 16 hs
d1 1.000
nt 4000 lb
ct 1120 fn

SPECIAL
FLAGS
PROCESSING
DISPLAY
TRANSMITTER
DISPLAY
DECOUPLER
PLOT

dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 8900

no ph
wc 250
sc 0
vs 26
th 3
```

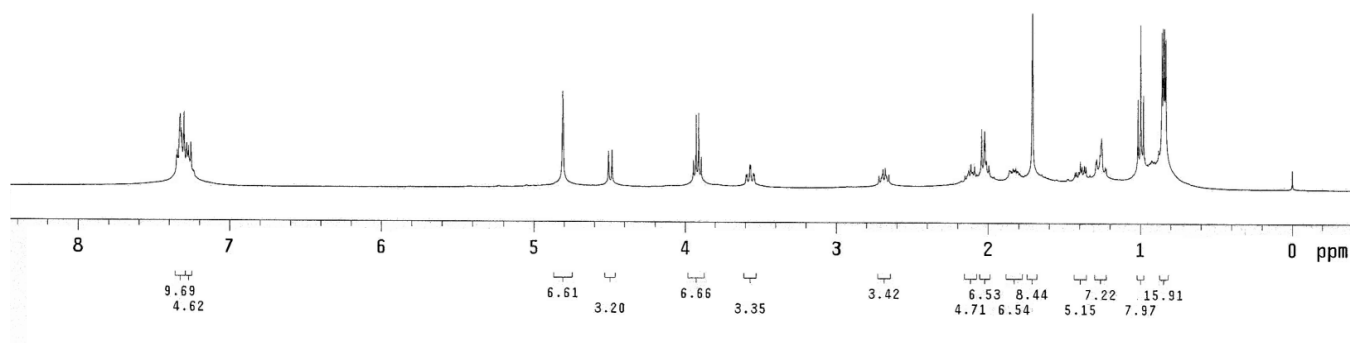
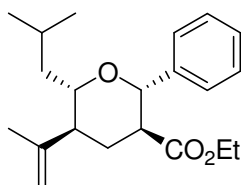


¹H NMR spectra of 11h

PS_409_M

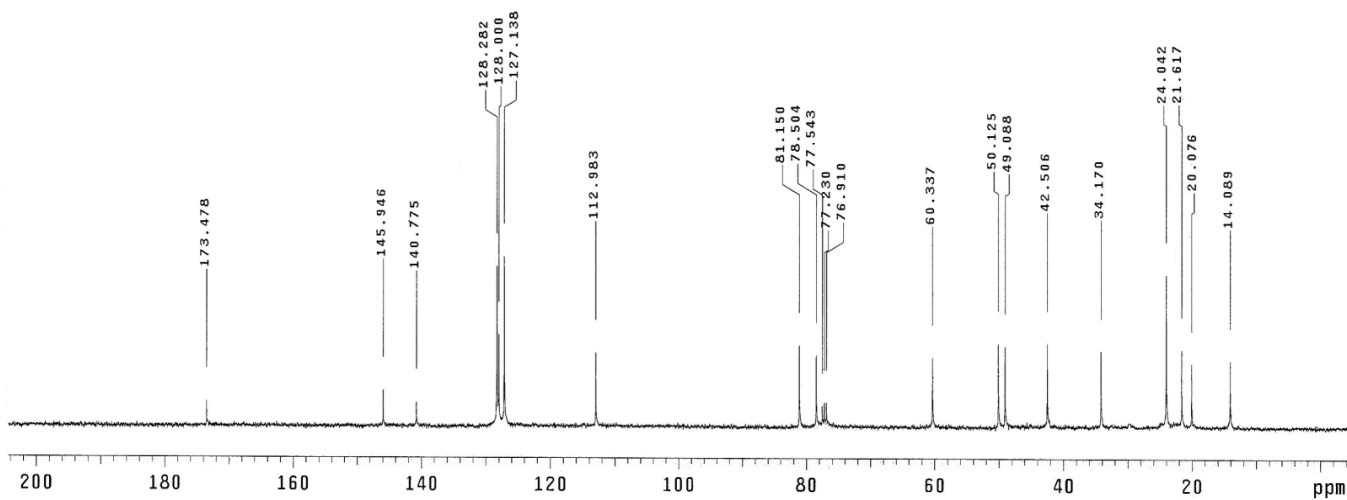
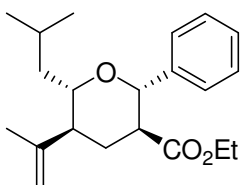
exp1 s2pu1

SAMPLE		SPECIAL	
date	Dec 22 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_409~	hst	0.008
	M1.fid	pw90	19.700
ACQUISITION		alfa	20.000
FLAGS			
sw	6389.8		
at	1.998	fl	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-166.3
sfrq	399.853	wp	3543.3
tof	362.8	rf	796.0
tpwr	57	rfp	0
pw	9.850	rp	124.9
DECOUPLER		lp	-87.5
PLOT			
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	28
dpwr	50	th	42
dmf	15900	nm	cdc ph



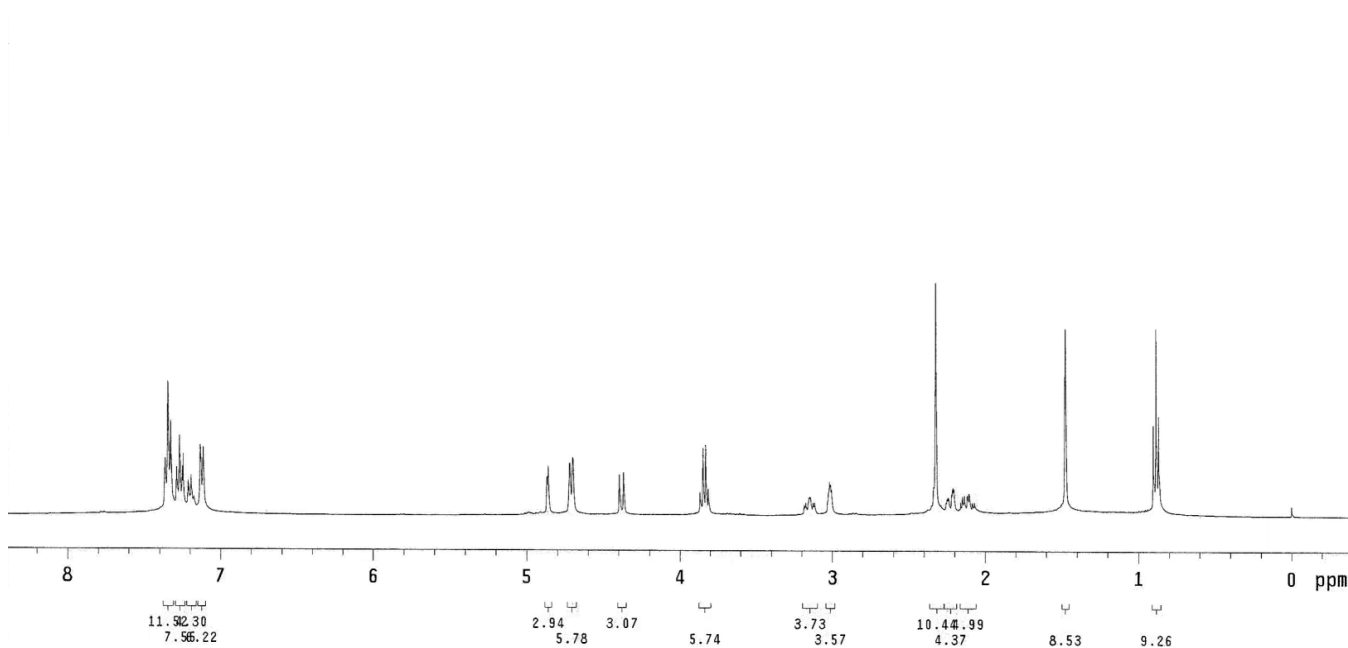
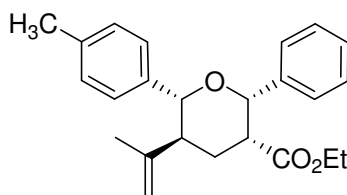
¹³C NMR spectra of 11h

```
PS_409_M
exp1 s2pu1
SAMPLE
date Dec 21 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_409~ hst 0.008
M_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 i1 n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
dl 1.000 PROCESSING
nt 2000 lb 2.00
ct 560 fn 65536
TRANSMITTER C13 sp DISPLAY
tn 100.554 wp -487.4
sfrq 1536.3 rfl 21030.9
tof 61 rfp 9275.2
tpwr 9.300 rp 7764.9
pw 61 rfp -47.4
DECOUPLER H1 lp -341.6
dn 0 wc PLOT
dof 0 wc 250
dm vvy sc 0
dmm w vs 26
dpwr 42 th 3
dmf 8900 nm no ph
```



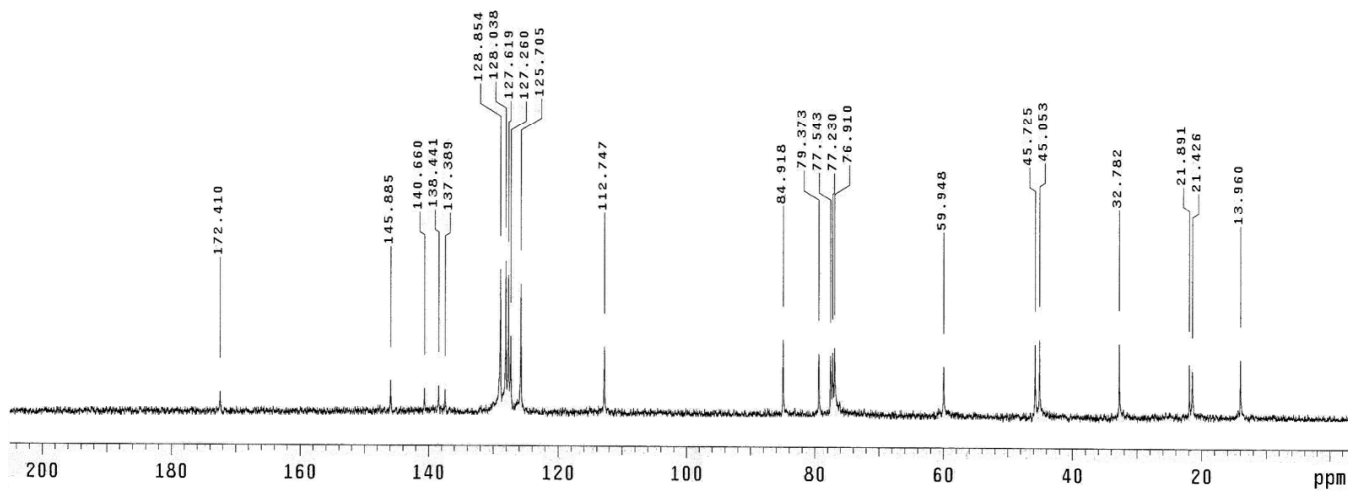
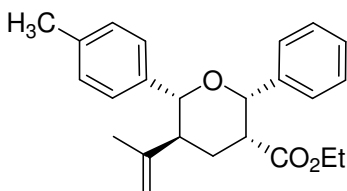
¹H NMR spectra of 12a

```
PS_421_M
expl s2pu1
SAMPLE
date Jan 3 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_421~ hst 0.008
_M.fid pw90 19.700
ACQUISITION alfa 20.000
sw 6389.8
at 1.998 i1
np 25528 in
fb not used dp
bs 8 hs
d1 1.000 PROCESSING
nt 100 ib 0.10
ct 100 fn 65536
TRANSMITTER H1 sp
tn H1 sp -162.8
sfrq 399.853 wp 3518.1
tof 362.8 rfl 800.9
tpwr 57 rfp 0
pw 9.850 rp 109.8
DECOUPLER C13 lp -94.8
dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 36
dpwr 50 th 20
dmf 15900 nm cdc ph
```



¹³C NMR spectra of 12a

```
PS_421_M
exp1 s2pu1
SAMPLE
date Jan 2 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_421~ hst 0.008
M_C13.fid pw90 18.600
ACQUISITION alfa 20.000
sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 3000 lb 2.00
ct 3000 fn 65536
TRANSMITTER DISPLAY
tn C13 sp -353.2
sfrq 100.554 wp 20965.0
tof 1536.3 rfl 9273.6
tpwr 61 rfp 7764.9
pw 9.300 rp -64.5
DECOUPLER lp -286.4
dn H1 PLOT
dbf 0 wc 250
dm yvy sc 0
dmm w vs 23
dpwr 42 th 2
dmf 8900 nm no ph
```

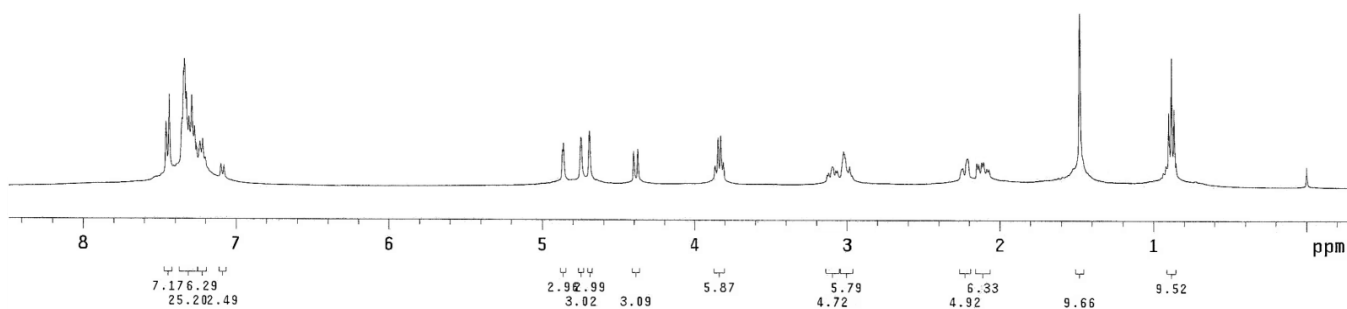
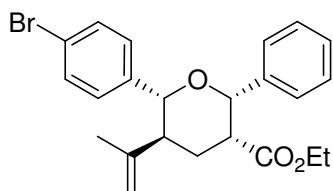


¹H NMR spectra of 12b

PS_432_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 14 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_432~	hst	0.008
	.M.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	f1	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-116.8
sfrq	399.853	wp	3509.7
tof	362.8	rfl	796.8
tpwr	57	rfp	0
pw	9.850	rp	109.7
		lp	-99.5
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	29
dpwr	50	th	18
dmf	15900	nm	cdc ph

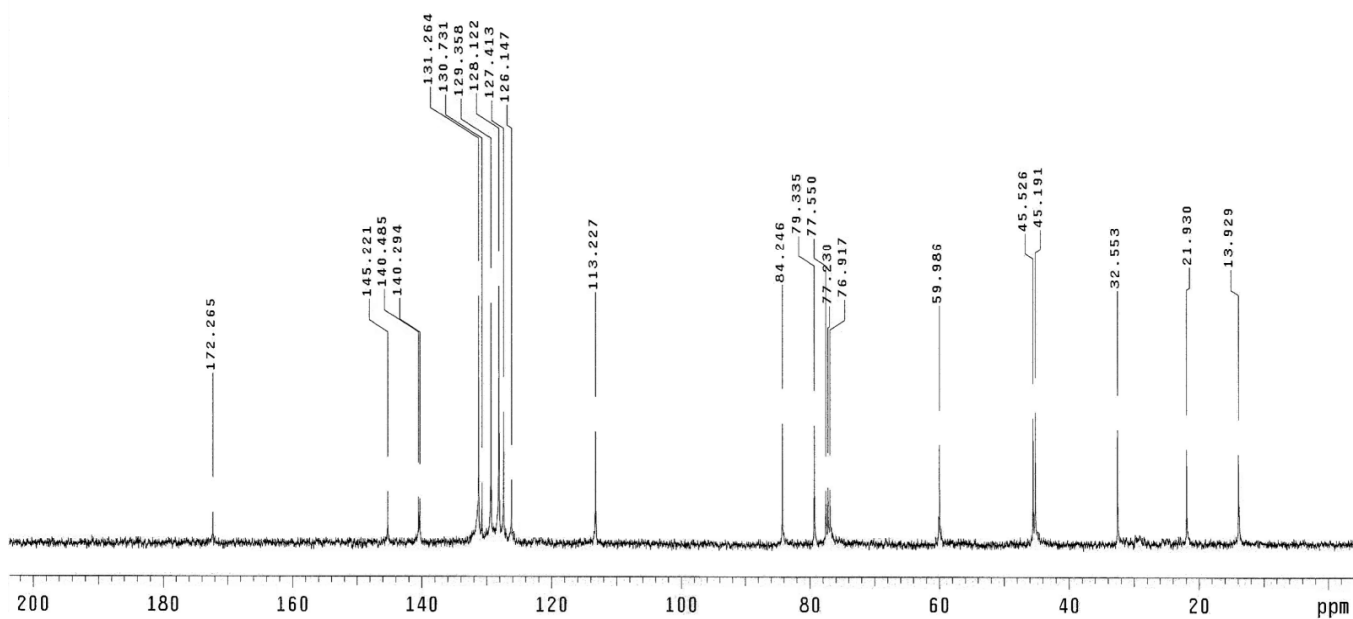
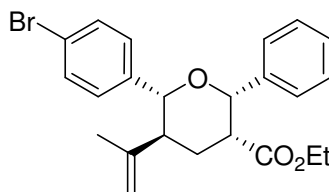


¹³C NMR spectra of 12b

PS_432_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jan 14 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_432-		hst	0.008
M_C13.fid		pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	4000	lb	2.00
ct	1168	fn	65536
TRANSMITTER	C13	DISPLAY	
tn		sp	-423.0
sfrq	100.554	wp	20899.0
tof	1536.3	rfl	9276.7
tpwr	61	rfp	7764.9
pw	9.300	rp	-56.2
DECOUPLER	H1	lp	-292.1
dn		PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	37
dpwr	42	th	4
dmf	8900	nm	no ph

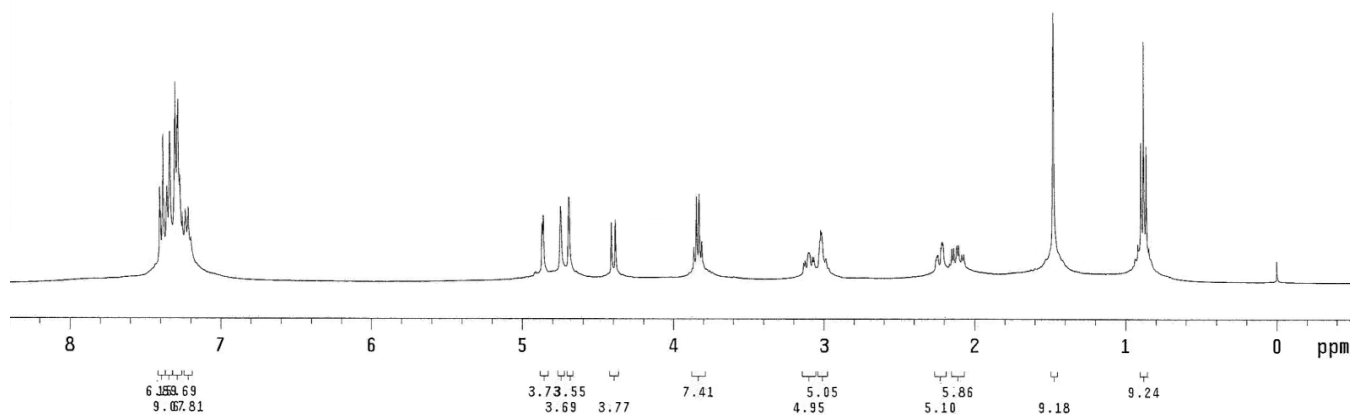
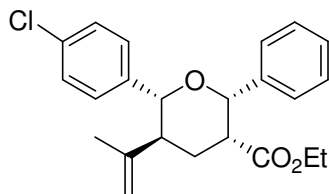


¹H NMR spectra of 12c

PS_434_M

expl s2pu1

SAMPLE		SPECIAL	
date	Jan 15 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_434~	hst		0.008
	M.fid	pw90	19.700
ACQUISITION	alfa		20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-209.6
sfrq	399.853	wp	3568.4
tof	362.8	rfl	797.4
tpwr	57	rfp	0
pw	9.850	rp	115.2
		lp	-109.8
DECOUPLER		PLOT	
dn	C13		
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	42
dpwr	50	th	20
dmf	15900	nm	cdc ph

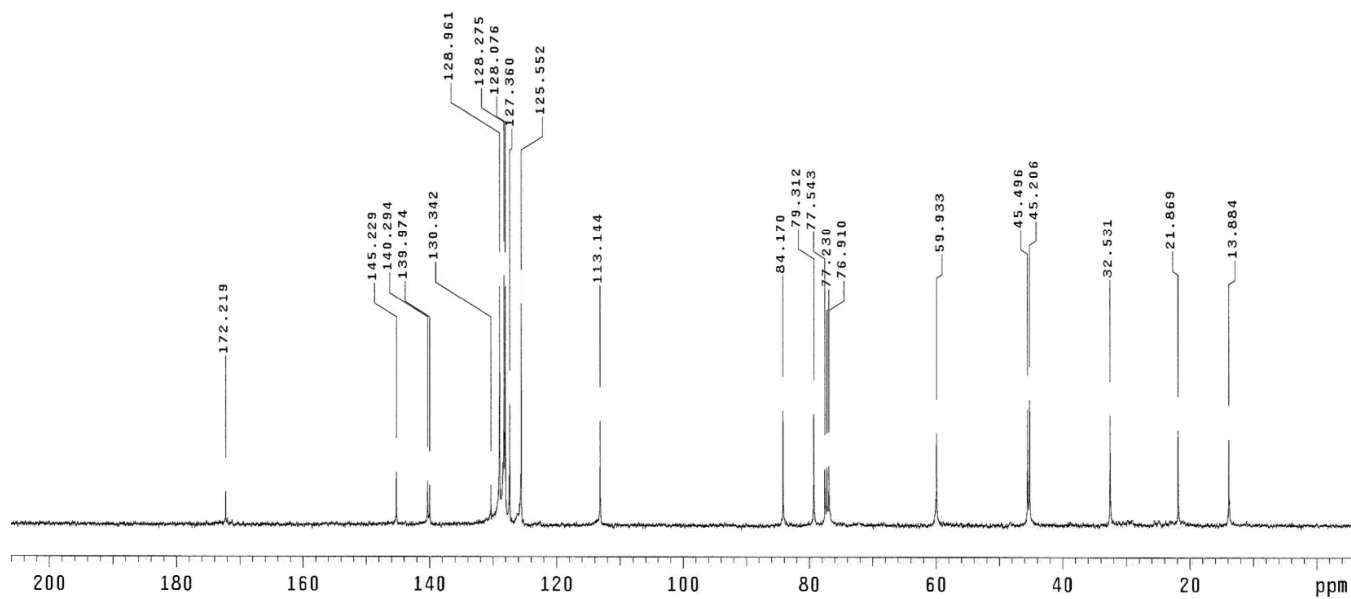
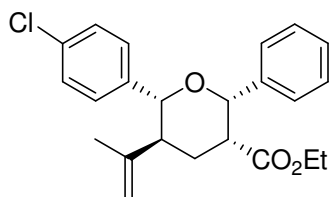


¹³C NMR spectra of 12c

PS_434_M

expl s2pu1

SAMPLE		SPECIAL	
date	Jan 15 2011	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_434~	hst	0.008
	M_C13.fid	pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	8000	lb	2.00
ct	2592	fn	65536
TRANSMITTER		DISPLAY	
tn	C13	sp	-590.1
sfrq	100.554	wp	21328.5
tof	1536.3	rfl	9279.0
tpwr	61	rfp	7764.9
pw	9.300	rp	-50.9
DECOUPLER		lp	-302.4
dn	H1	PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	37
dpwr	42	th	4
dmf	8900	nm	no
		ph	

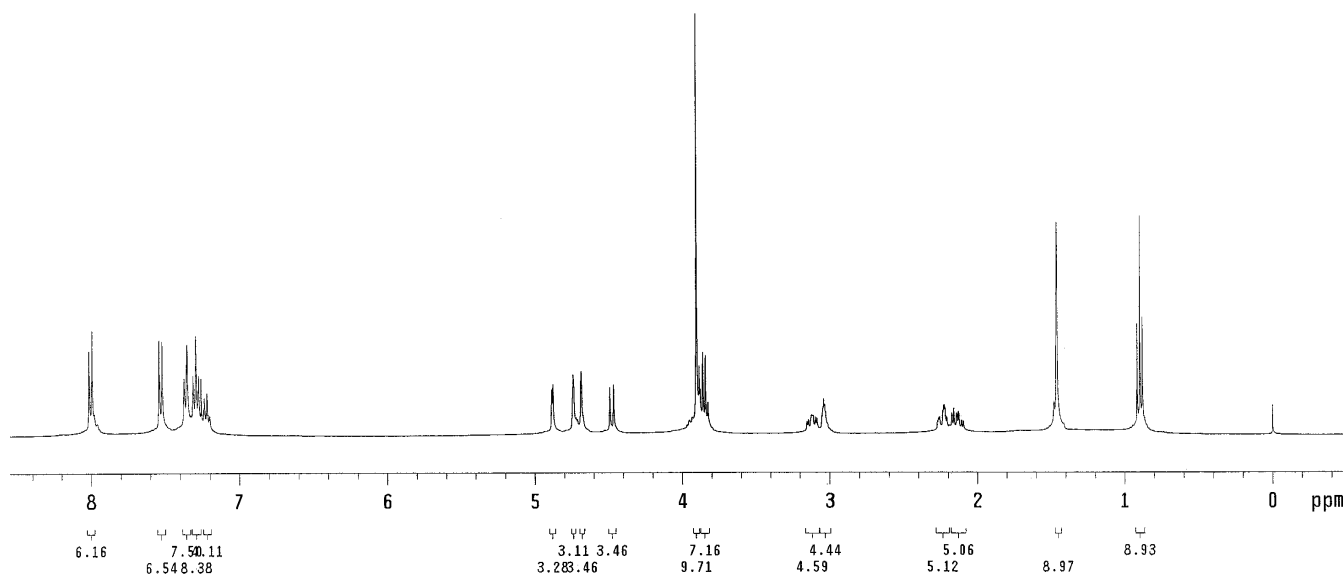
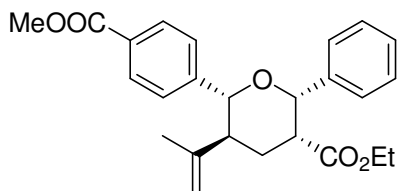


¹H NMR spectra of 12d

PS_493_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jun 6 2011	temp	not used
solvent	CDC13	gain	not used
file		exp	spin
ACQUISITION		hst	0.008
sw	6389.8	pw90	19.700
at	1.998	alfa	20.000
np	25528	FLAGS	
fb	not used	il	n
bs	8	in	n
d1	1.000	dp	y
nt	100	hs	nn
ct	100	PROCESSING	
TRANSMITTER		lb	0.10
tn	H1	fn	65536
sfrq		DISPLAY	
tof	399.853	sp	-208.5
tpwr	362.8	wp	3635.7
pw	57	rfl	796.2
DECOUPLER		rfp	0
dn	C13	rp	122.7
dof	0	lp	-95.2
dm		PLOT	
dmm	nnn	wc	250
dpwr	50	sc	0
dmf	15900	vs	62
		th	14
		nm	cdc ph

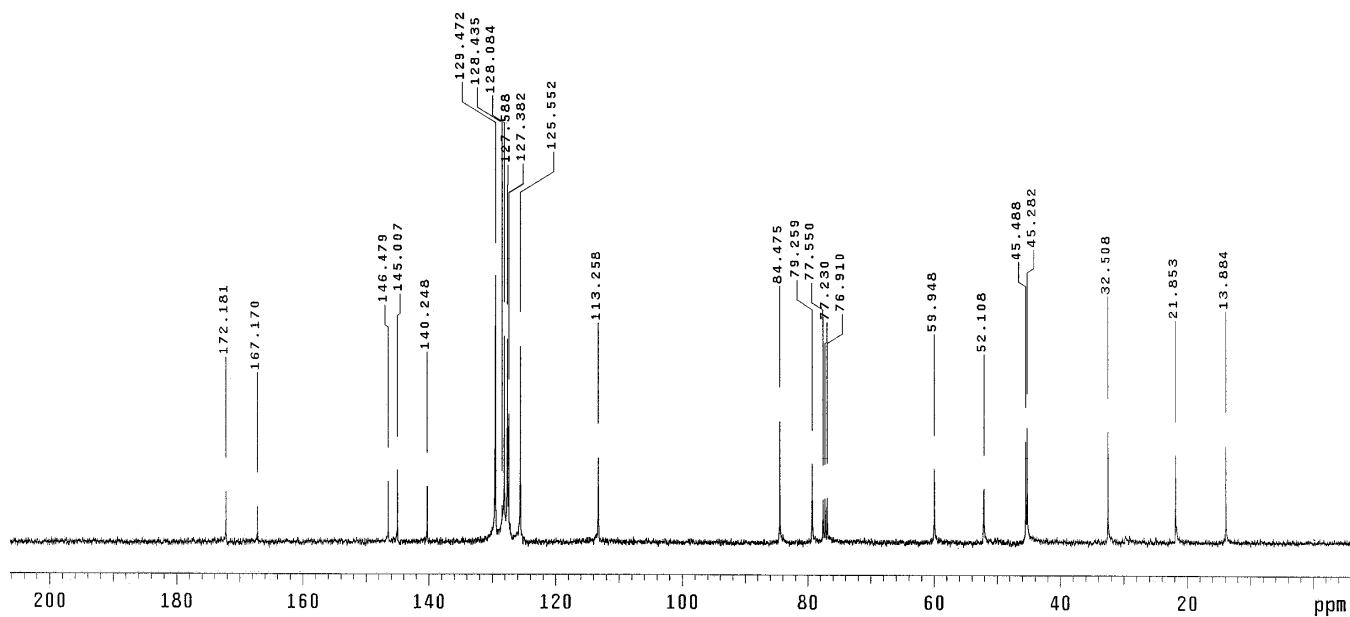
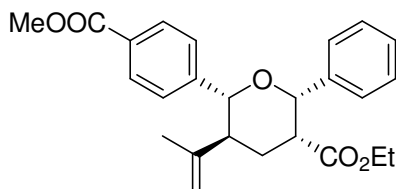


¹³C NMR spectra of 12d

PS_493_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Jun 7 2011	temp	not used
solvent	CDCl3	gain	not used
file		spin	not used
ACQUISITION	exp	hst	0.008
sw	25125.6	pw90	18.600
at	1.199	alfa	20.000
np	60270	FLAGS	
fb	13800	il	n
bs	16	in	n
d1	1.000	dp	y
nt	4000	hs	nn
ct	864	PROCESSING	
TRANSMITTER	lb	lb	2.00
tn	C13	fn	65536
sfrq	100.554	DISPLAY	
tof	1536.3	sp	-623.1
tpwr	61	wp	21361.4
pw	9.300	rfl	9279.0
DECOUPLER	rfl	rfl	7764.9
dn	H1	rp	-71.4
dof	0	lp	-277.0
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	42	sc	0
dmf	8900	vs	39
	th	th	4
	nm	no	ph



¹H NMR spectra of 12e

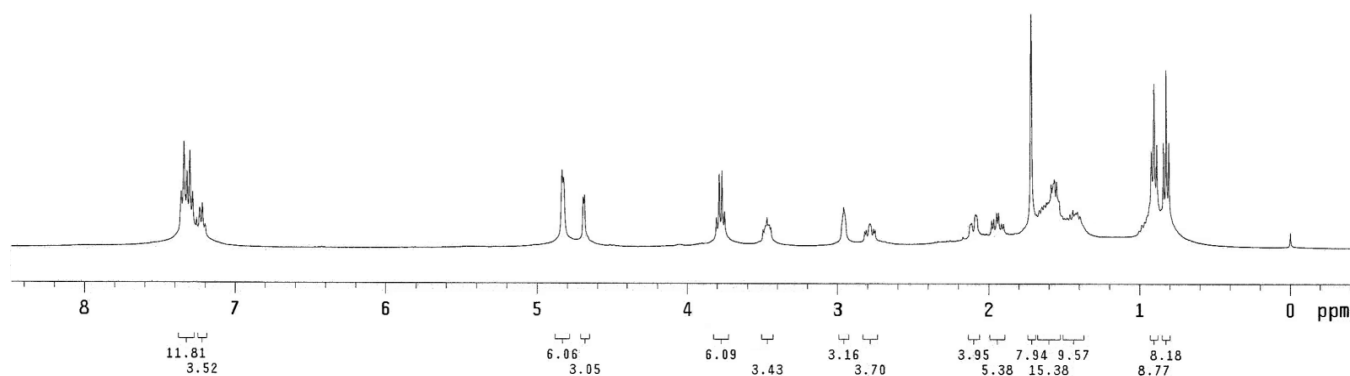
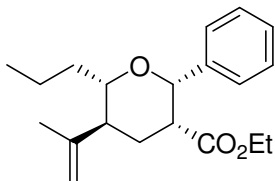
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PS_411_M
exp1 s2pu1

SAMPLE
date Dec 22 2010 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_411~ hst 0.008
_M.fid pw90 19.700
ACQUISITION alfa 20.000

sw 6389.8
at 1.998 il
np 25528 in
fb not used dp
bs 8 hs
d1 1.000 PROCESSING
nt 100 lb 0.10
ct 100 fn 65536

TRANSMITTER H1 sp
tn H1 sp -175.1
sfrq 399.853 wp 3568.4
tof 362.8 rfl 796.4
tpwr 57 rfp 0
pw 9.850 rp 119.8
DECOUPLER lp -83.9

dn C13 PLOT
dof 0 wc 250
dm nnn sc 0
dmm c vs 37
dpwr 50 th
dmf 15900 nm cdc ph
```

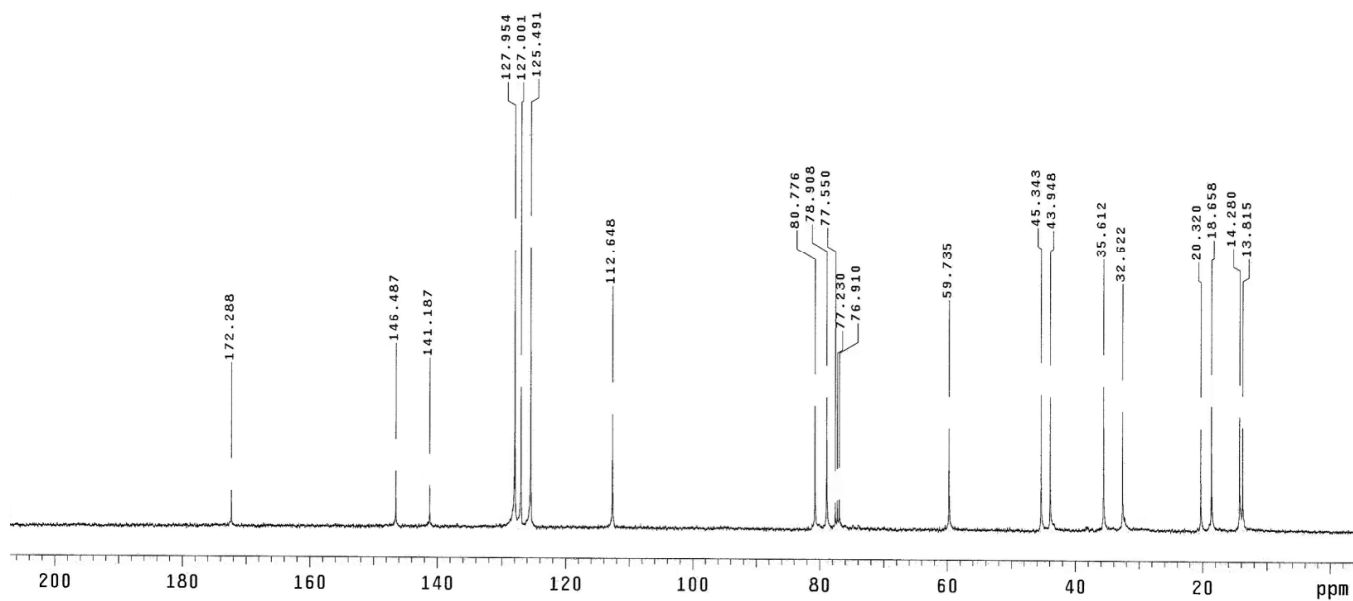
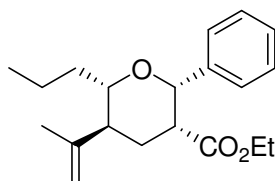


¹³C NMR spectra of 12e

PS_411_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Dec 22 2010	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_411~	hst	0.008	
M_C13.fid	pw90	18.600	
ACQUISITION	alfa	20.000	
sw	25125.6	FLAGS	
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	3000	lb	2.00
ct	992	fn	65536
TRANSMITTER	C13	DISPLAY	
tn		sp	-390.7
sfrq	100.554	wp	21229.5
tof	1536.3	rfl	9277.5
tpwr	61	rffp	7764.9
pw	9.300	rp	-51.8
DECOUPLER	H1	lp	-300.3
dn		PLOT	
dof	0	wc	250
dm	YVY	sc	0
dmm	w	vs	44
dpwr	42	th	4
dmf	8900	nm	no ph

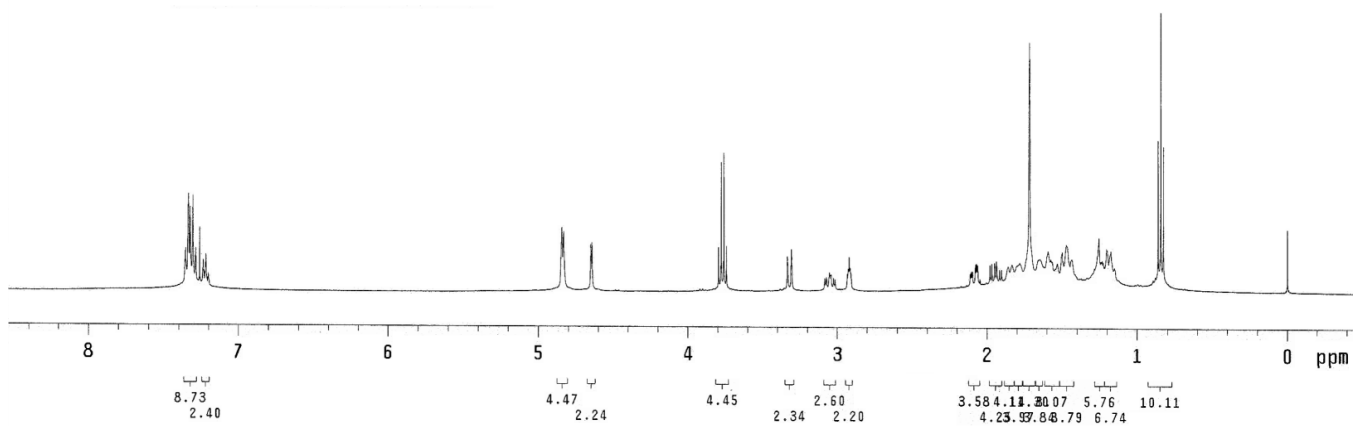
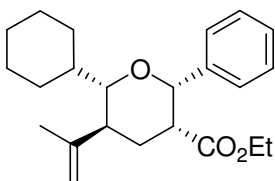


¹H NMR spectra of 12f

PS_452_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 23 2011	temp	not used
solvent	CDC13	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_452~	hst	0.008
	~.M.fid	pw90	19.700
		alfa	20.000
ACQUISITION		FLAGS	
sw	6389.8		
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	100	lb	0.10
ct	100	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-181.4
sfrq	399.853	wp	3593.6
tof	362.8	rfl	794.3
tpwr	57	rfp	0
pw	9.850	rp	114.1
		lp	-91.7
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	43
dmm	c	th	20
dpwr	50	cdc	ph
dmf	15900		

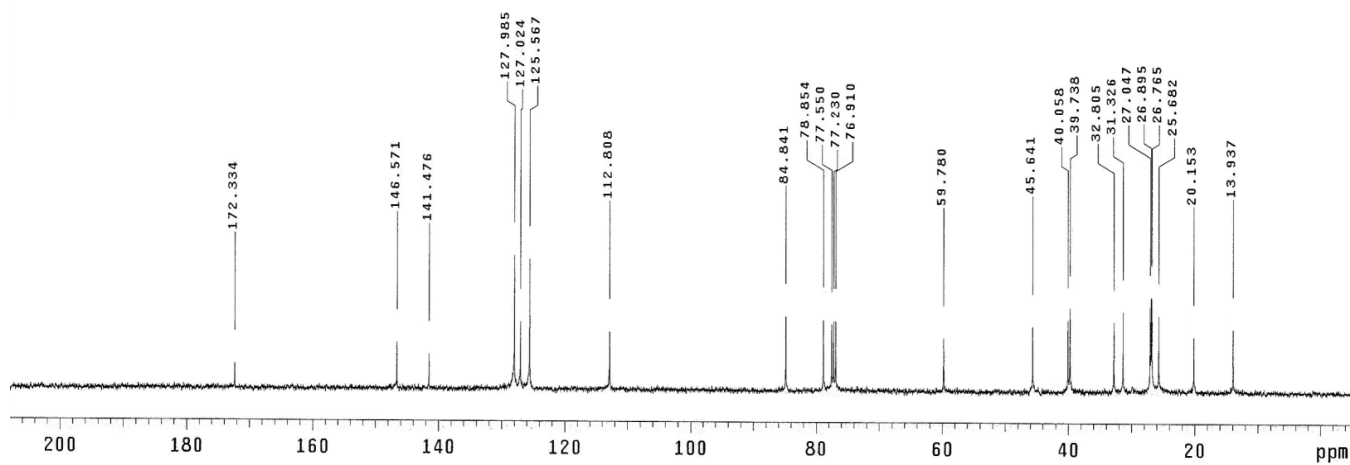
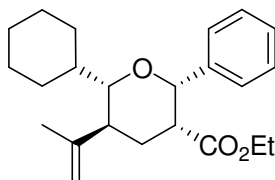


¹³C NMR spectra of 12f

PS_452_M

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 23 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_452~	hst	0.008
M_C13.fid		pw90	18.600
ACQUISITION	alfa		20.000
sw	25125.6	FLAGS	
at	1.199	l1	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	5000	lb	2.00
ct	2080	fn	65536
TRANSMITTER	C13	DISPLAY	
tn		sp	-549.5
sfrq	100.554	wp	21460.3
tof	1536.3	rfl	9271.3
tpwr	61	rfp	7764.9
pw	9.300	rp	-61.1
DECOUPLER	H1	lp	-302.7
dn		PLOT	
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	21
dpwr	42	th	3
dmf	8900	nm	no ph

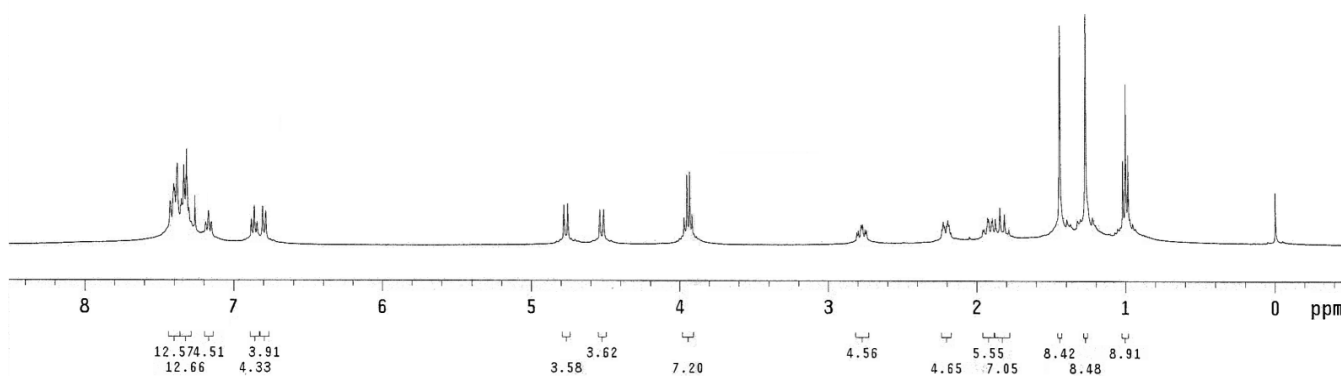
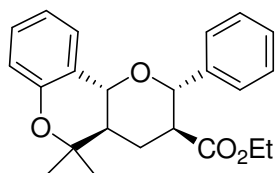


¹H NMR spectra of 13

PS_451_L

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 19 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_451~	hst	0.008
	L.fid	pw90	19.700
ACQUISITION	alfa		20.000
sw	6389.8	FLAGS	
at	1.998	il	n
np	25528	in	n
fb	not used	dp	y
bs	8	hs	nn
d1	1.000	PROCESSING	
nt	200	lb	0.10
ct	0	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-199.5
sfrq	399.853	wp	3602.0
tof	362.8	rfl	795.6
tpwr	57	rfp	0
pw	9.850	rp	106.5
DECOUPLER		lp	-67.6
dn	C13	PLOT	
dof	0	wc	250
dm	nnn	sc	0
dmm	c	vs	36
dpwr	50	th	25
dmf	15900	nm	cdc ph

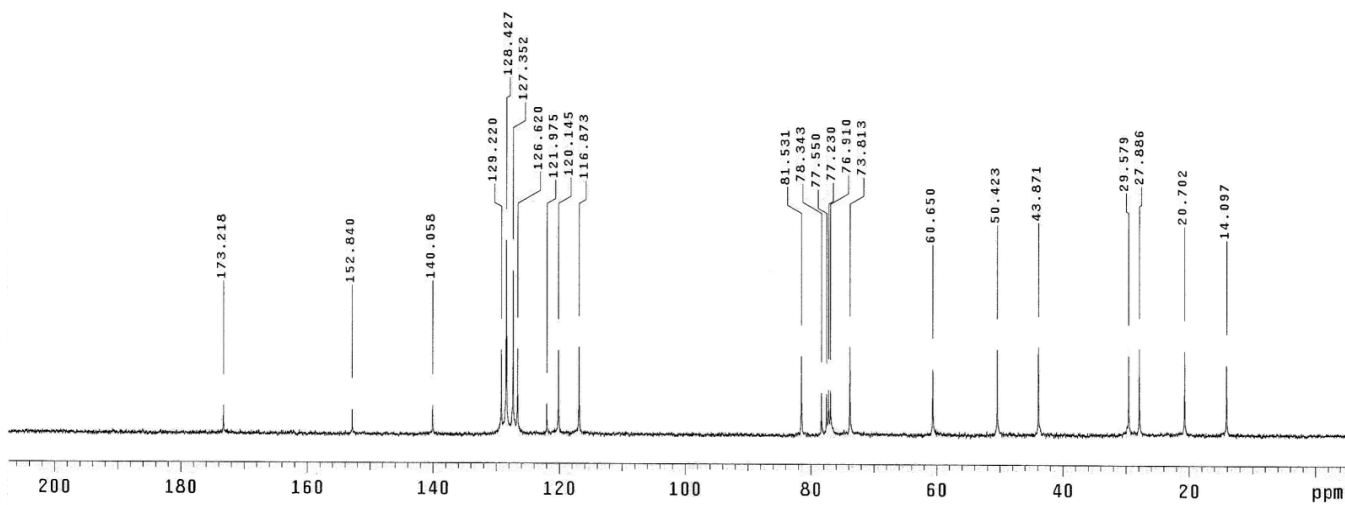
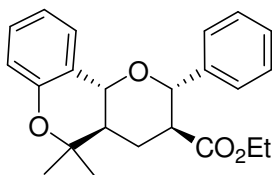


¹³C NMR spectra of 13

PS_451_L

exp1 s2pu1

SAMPLE		SPECIAL	
date	Feb 19 2011	temp	not used
solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp/AKS_PS_451~	L_C13.fid	hst	0.008
ACQUISITION	alfa	pw90	18.600
sw	25125.6	alpha	20.000
at	1.199	il	n
np	60270	in	n
fb	13800	dp	y
bs	16	hs	nn
d1	1.000	PROCESSING	
nt	5000	lb	2.00
ct	0	fn	65536
tn	TRANSMITTER	C13	sp
sfrq	100.554	wp	-554.1
tof	1536.3	rfl	21394.4
tpwr	61	rfp	9275.9
pw	9.300	rp	7764.9
DECOUPLER	H1	lp	-49.0
dn	0	PLOT	-333.6
dof	0	wc	250
dm	yyy	sc	0
dmm	w	vs	31
dpr	42	th	3
dmi	8900	nm	no ph

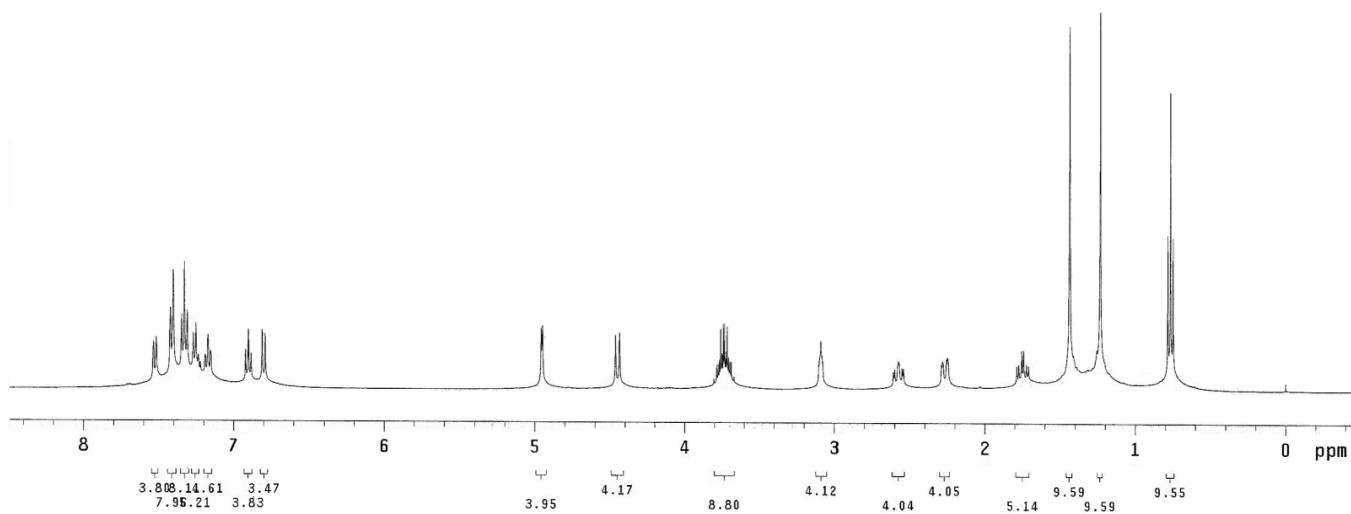
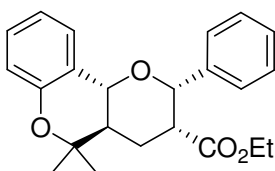


¹H NMR spectra of 14

PS_433_L

exp1 s2pu1

SAMPLE		SPECIAL	
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solvent	CDCl3	gain	not used
file	/export/home/~	spin	not used
ciftemp	AKS_PS_433	hst	0.008
	l1.fid	pw90	19.700
		alpha	20.000
ACQUISITION		FLAGS	
sw	6389.8	il	n
at	1.998	in	n
np	25528	dp	y
fb	not used	hs	nn
bs	4		
d1	1.000	PROCESSING	
nt	32	lb	0.10
ct	32	fn	65536
TRANSMITTER		DISPLAY	
tn	H1	sp	-180.8
sfrq	399.853	wp	3576.8
tof	362.8	rfl	810.4
tpwr	57	rfl	0
pw	9.850	rp	129.6
		lp	-97.3
DECOUPLER		PLOT	
dn	C13	wc	250
dof	0	sc	0
dm	nnn	vs	62
dmm	c	th	20
dpwr	50	cdc	ph
dmf	15900		



¹³C NMR spectra of 14

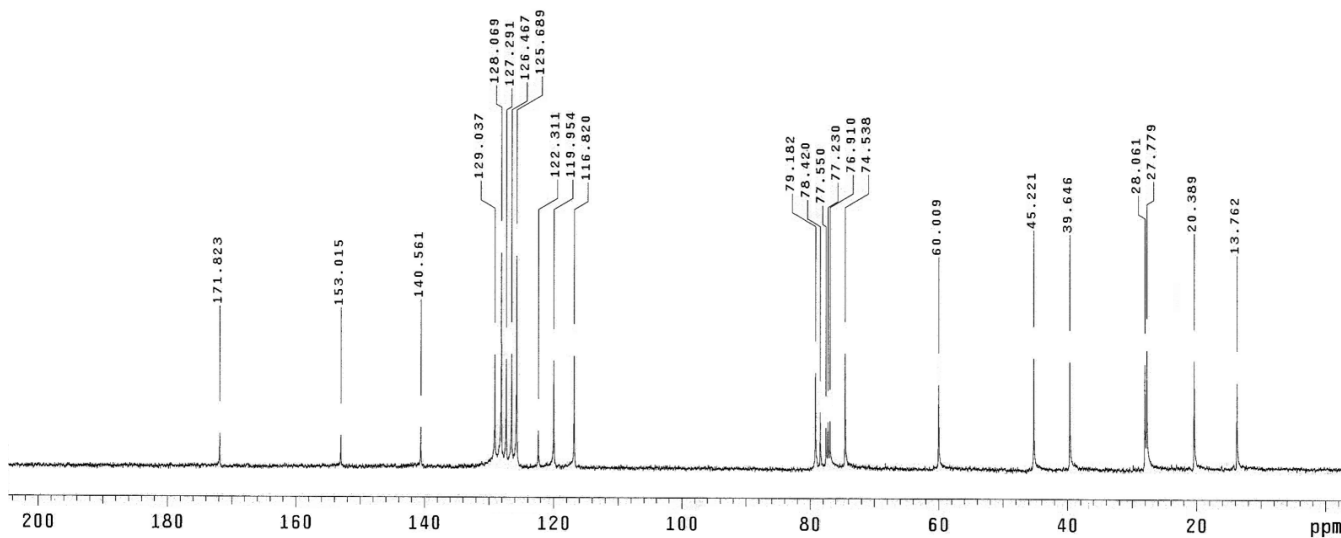
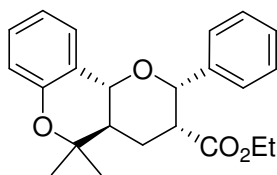
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PS_433_L1
exp1 s2pu1

SAMPLE
date Feb 21 2011 temp not used
solvent CDCl3 gain not used
file /export/home/~ spin not used
ciftemp/AKS_PS_433~ hst 0.008
Li_D13.fid pw90 18.600
ACQUISITION alfa 20.000

sw 25125.6 FLAGS
at 1.199 il n
np 60270 in n
fb 13800 dp y
bs 16 hs nn
d1 1.000 PROCESSING
nt 3000 lb 2.00
ct 3000 fn 65536

TRANSMITTER DISPLAY
tn C13 sp -358.5
sfrq 100.554 wp 20932.0
tof 1536.3 rfi 9279.0
tpwr 61 rfp 7764.9
pw 9.300 rp -77.9
DECOUPLER lp -269.1

dn H1 PLOT
dof 0 wc 250
dm yyv sc 0
dmm w vs 34
dpwr 42 th 4
dmf 8900 nm no ph
```



The crystal parameters of compound **4g**

	4g-CCDC 819363
Formula	C ₂₀ H ₂₆ O ₅
Formula weight	346.41
<i>T</i> /K	296(2)
Crystal system	Orthorhombic
Space group	P n a 21
<i>a</i> /Å	19.8370(10)
<i>b</i> /Å	15.2026(8)
<i>c</i> /Å	6.3744(4)
<i>α</i> /°	90.00
<i>β</i> /°	90.00
<i>γ</i> /°	90.00
<i>V</i> /Å ³	1922.35(18)
<i>Z</i>	4
Abs. Coeff./mm ⁻¹	0.085
Abs. Correction	multi-scan
GOF on <i>F</i> ²	0.943
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0677 <i>wR</i> 2 = 0.1644
<i>R</i> indices [all data]	<i>R</i> 1 = 0.0856 <i>wR</i> 2 = 0.1735

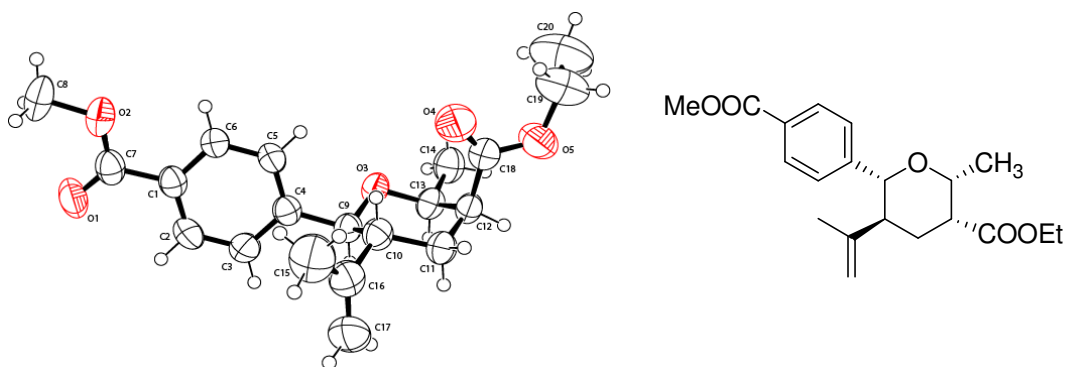


Figure 1: ORTEP diagram of **4g**

The crystal parameters of compound **11f**

	11f-CCDC 819365
Formula	C ₂₅ H ₂₈ O ₅
Formula weight	408.47
<i>T</i> /K	296(2)
Crystal system	Monoclinic
Space group	P 21/c
<i>a</i> /Å	12.0752(3)
<i>b</i> /Å	15.3970(4)
<i>c</i> /Å	12.4647(3)
α /°	90.00
β /°	104.3910(10)
γ /°	90.00
<i>V</i> /Å ³	2244.74(10)
<i>Z</i>	4
Abs. Coeff./mm ⁻¹	0.083
Abs. Correction	multi-scan
GOF on <i>F</i> ²	1.070
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0615 <i>wR</i> 2 = 0.1806
<i>R</i> indices [all data]	<i>R</i> 1 = 0.0759 <i>wR</i> 2 = 0.2203

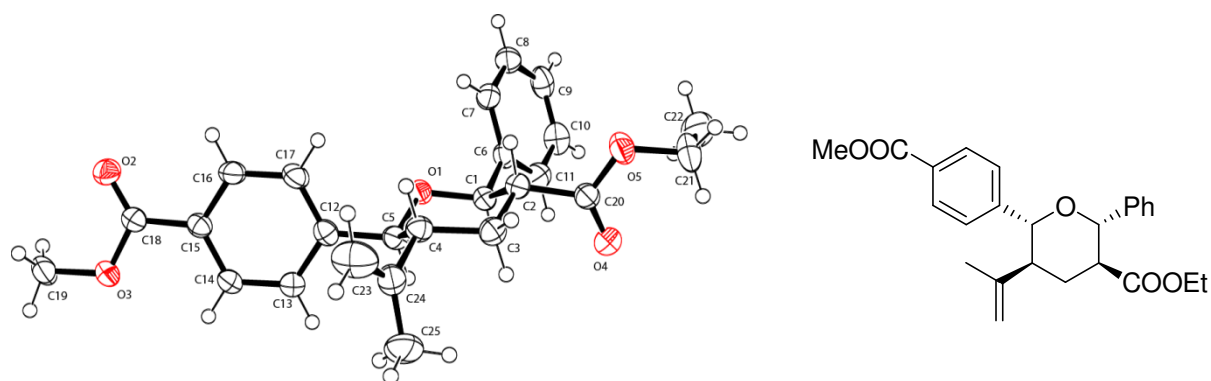


Figure 2: ORTEP diagram of **11f**

The crystal parameters of compound 13

	13-CCDC 819364
Formula	C ₂₃ H ₂₆ O ₄
Formula weight	366.44
<i>T</i> /K	296(2)
Crystal system	Monoclinic
Space group	P 21/n
<i>a</i> /Å	8.9913(7)
<i>b</i> /Å	19.6654(14)
<i>c</i> /Å	11.4452(9)
α /°	90.00
β /°	103.239(4)
γ /°	90.00
<i>V</i> /Å ³	1969.9(3)
<i>Z</i>	4
Abs. Coeff./mm ⁻¹	0.084
Abs. Correction	Multi-scan
GOF on <i>F</i> ²	1.064
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0667 <i>wR</i> 2 = 0.1815
<i>R</i> indices [all data]	<i>R</i> 1 = 0.0923 <i>wR</i> 2 = 0.1973

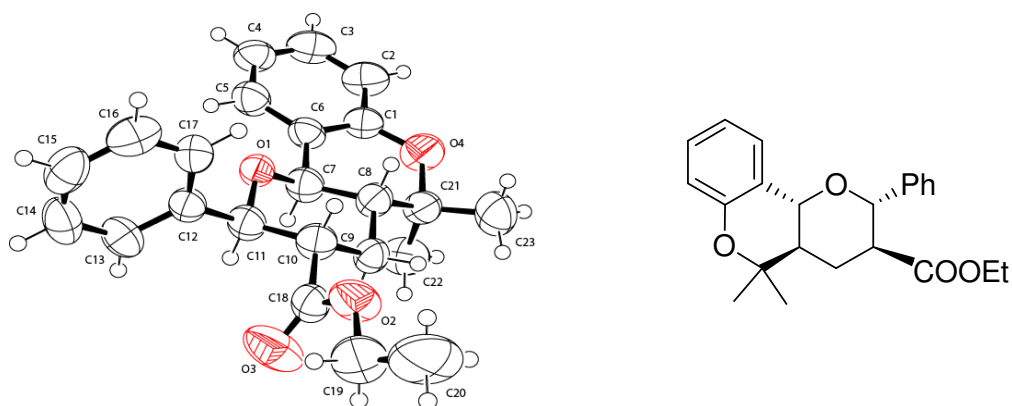


Figure 3: ORTEP diagram of 13

The crystal parameters of compound 14

	14-CCDC 819366
Formula	C ₂₃ H ₂₆ O ₄
Formula weight	366.44
<i>T</i> /K	296(2)
Crystal system	Monoclinic
Space group	P 21/c
<i>a</i> /Å	12.5282(8)
<i>b</i> /Å	8.7884(6)
<i>c</i> /Å	18.2013(12)
α /°	90.00
β /°	94.582(2)
γ /°	90.00
<i>V</i> /Å ³	1997.6(2)
<i>Z</i>	4
Abs. Coeff./mm ⁻¹	0.082
Abs. Correction	Multi-scan
GOF on <i>F</i> ²	1.373
Final <i>R</i> indices [<i>I</i> > 2σ(<i>I</i>)]	<i>R</i> 1 = 0.0613 <i>wR</i> 2 = 0.1695
<i>R</i> indices [all data]	<i>R</i> 1 = 0.0967 <i>wR</i> 2 = 0.2108

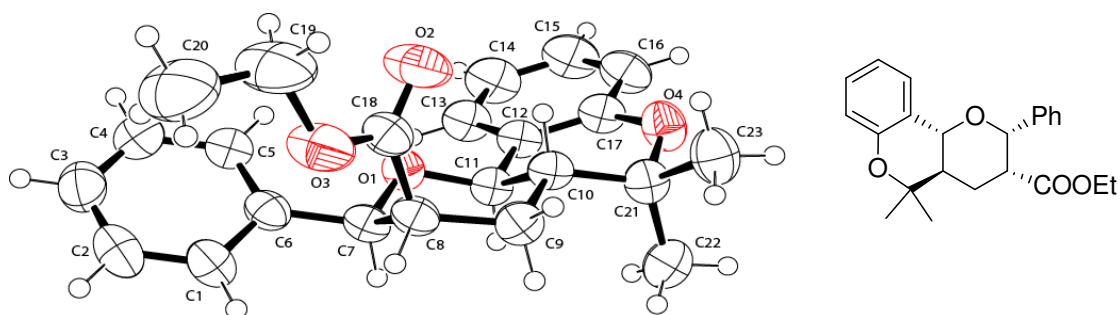


Figure 4: ORTEP diagram of 14