

# Environmentally Benign Diastereoselective Synthesis of Granatane and Tropane Aldol Derivatives

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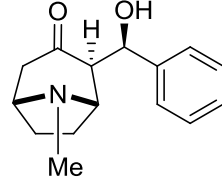
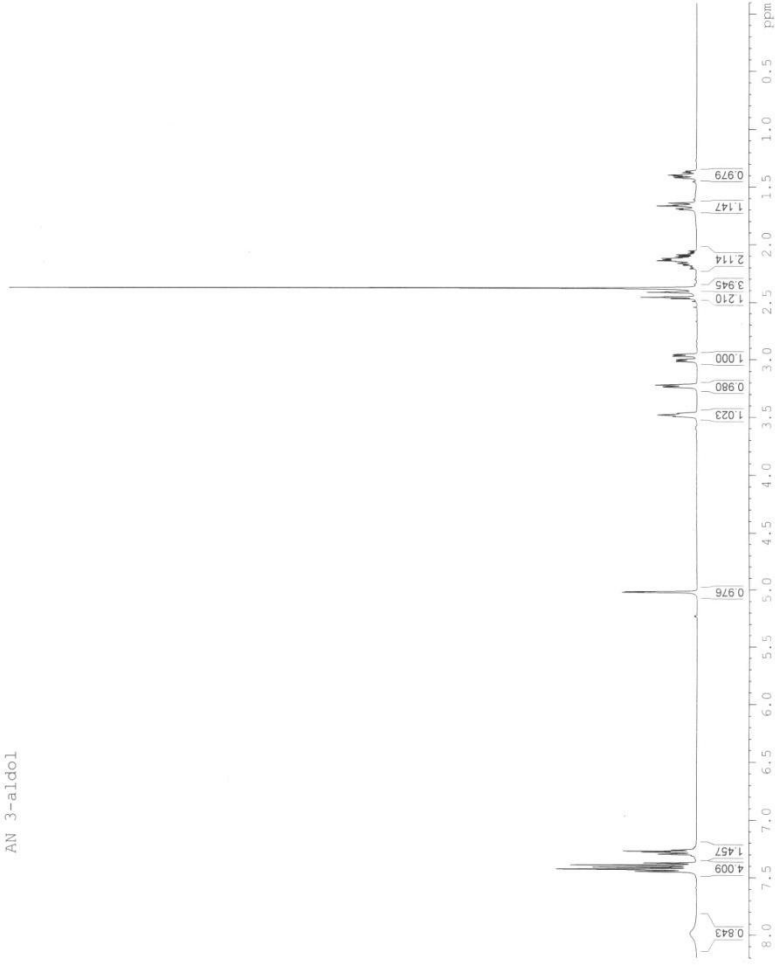
## Supporting information

### Table of Contents

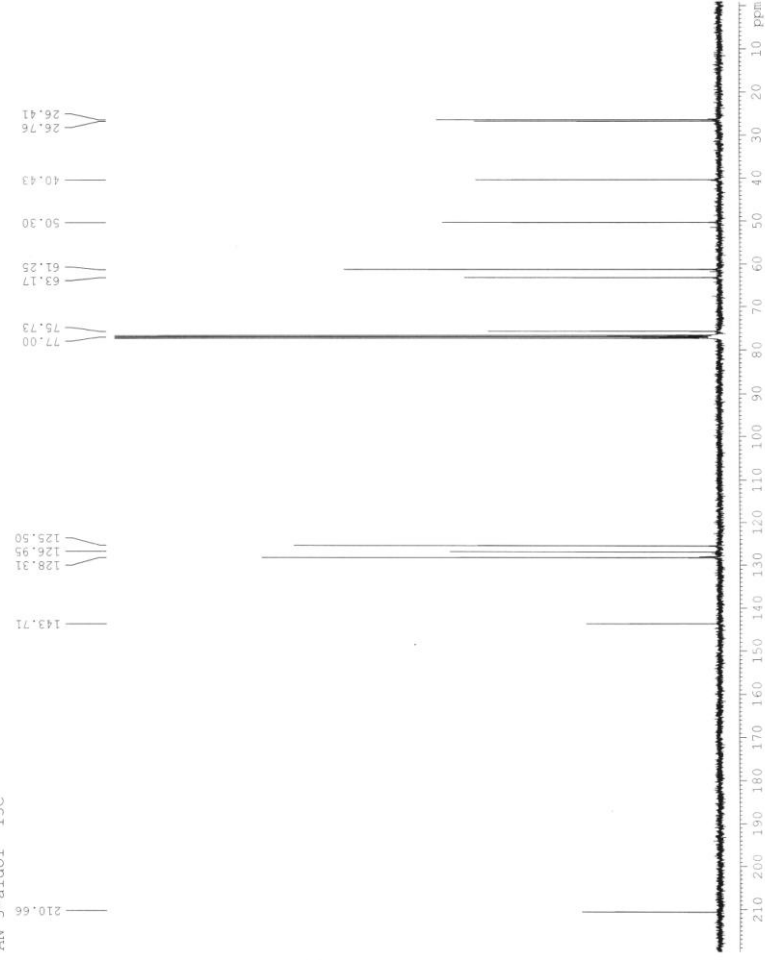
1. Preparation of granatanone.....	S2
2. <sup>1</sup> H NMR and <sup>13</sup> C NMR spectra of tropinone aldols <b>3</b> .....	S3
3. <sup>1</sup> H NMR and <sup>13</sup> C NMR spectra of granatanone aldols <b>4</b> .....	S17
4. <sup>1</sup> H NMR and <sup>13</sup> C NMR spectra of <i>N</i> -alkyl nortropinone and <i>N</i> -alkyl norgranatanone aldols.....	S27
5. <sup>1</sup> H NMR and <sup>13</sup> C NMR spectrum of bisaldol <b>5e</b> and tropinone and granatanone enones <b>7c, 8c, 9c</b> .....	S35
6. Calculations of selected E-factors.....	S39

**Granatanone** (**5**, pseudopelletierine) was synthesized by an adapted literature procedure.<sup>1</sup> Acetonedicarboxylic acid (1.461 g, 10 mmol) was added to a cooled (0 °C) solution of methylamine hydrochloride (0.810 g, 12 mmol) and glutaraldehyde (25% solution; 3.8 mL, 10 mmol) in water (10 mL). The mixture was adjusted to pH = 4 with 10% aqueous AcONa and stirred overnight at rt. Then the mixture was acidified to pH = 2 with 10% aqueous HCl and washed with diethyl ether (2 × 10 mL – wash discarded). The aqueous layer was basified with saturated NaHCO<sub>3</sub> to pH = 8 and extracted with DCM (5 × 10 mL). The combined extracts were dried over Na<sub>2</sub>SO<sub>4</sub>, filtered, and concentrated under vacuum. The crude product was sublimed (Kugelrohr, 80 °C, 1.1 Torr) to give granatanone (1.241 g, 81%) as a white solid. <sup>1</sup>H-NMR: 3.35-3.28 (m, 2H), 2.75 (dd, *J* = 16.7 Hz, 6.6 Hz, 2H), 2.62 (s, 3H), 2.24 (d, *J* = 16.8 Hz, 2H), 2.02-1.89 (m, 2H), 1.58-1.47 (m, 4H); <sup>13</sup>C-NMR: 210.5, 55.5, 41.7, 41.0, 29.4, 15.8.

AN 3-aldol

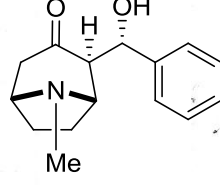
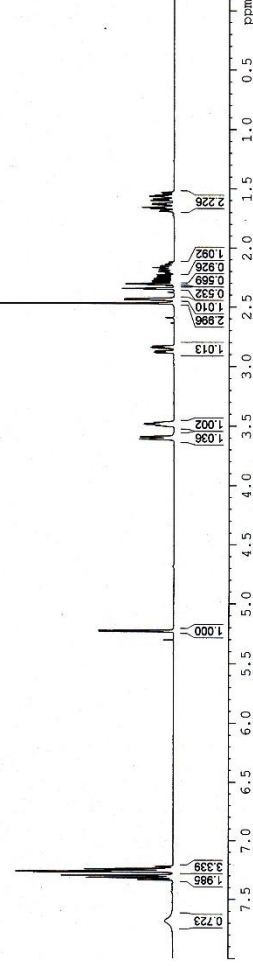


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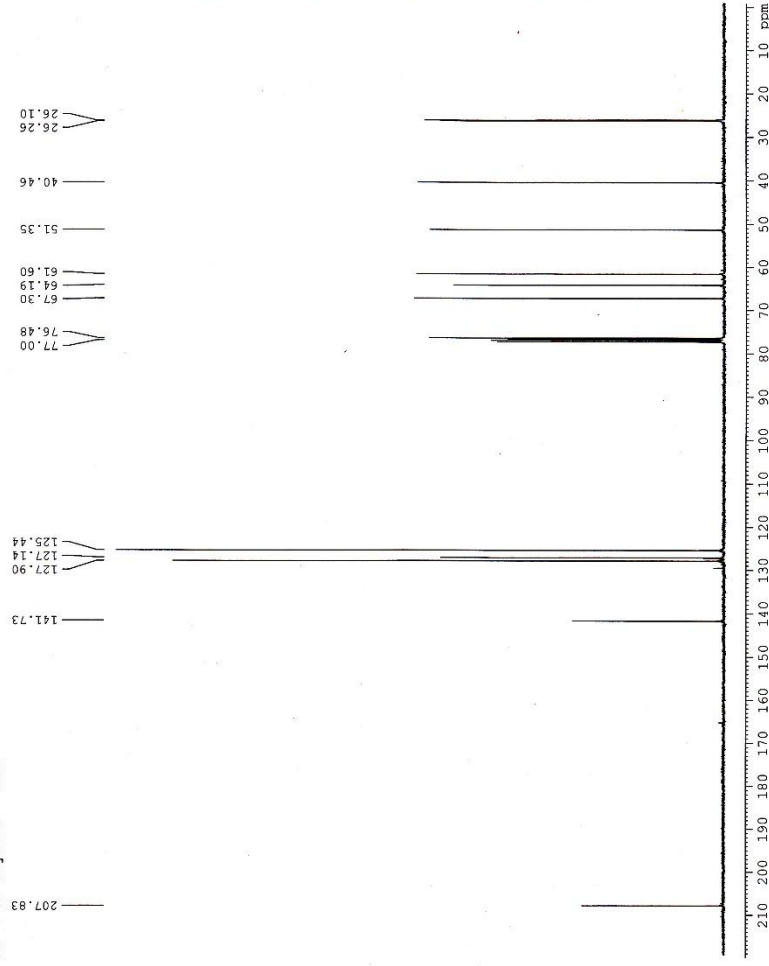
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AN 429C I kryst.

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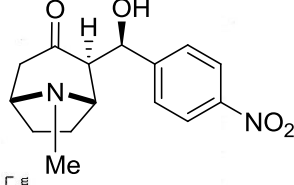
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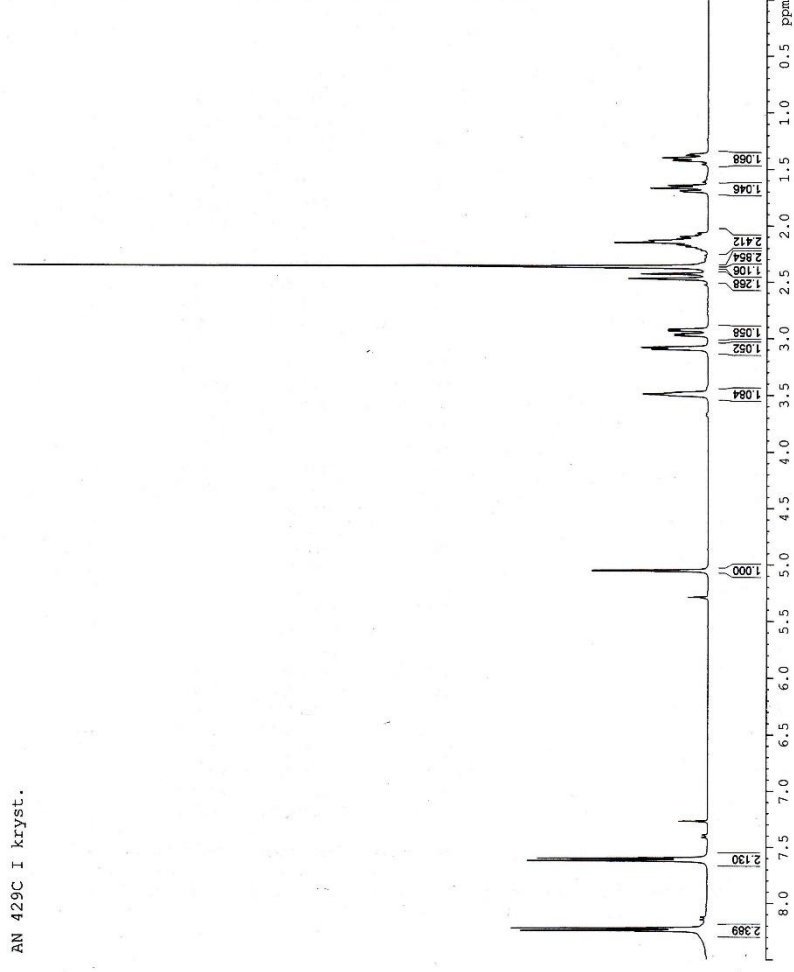
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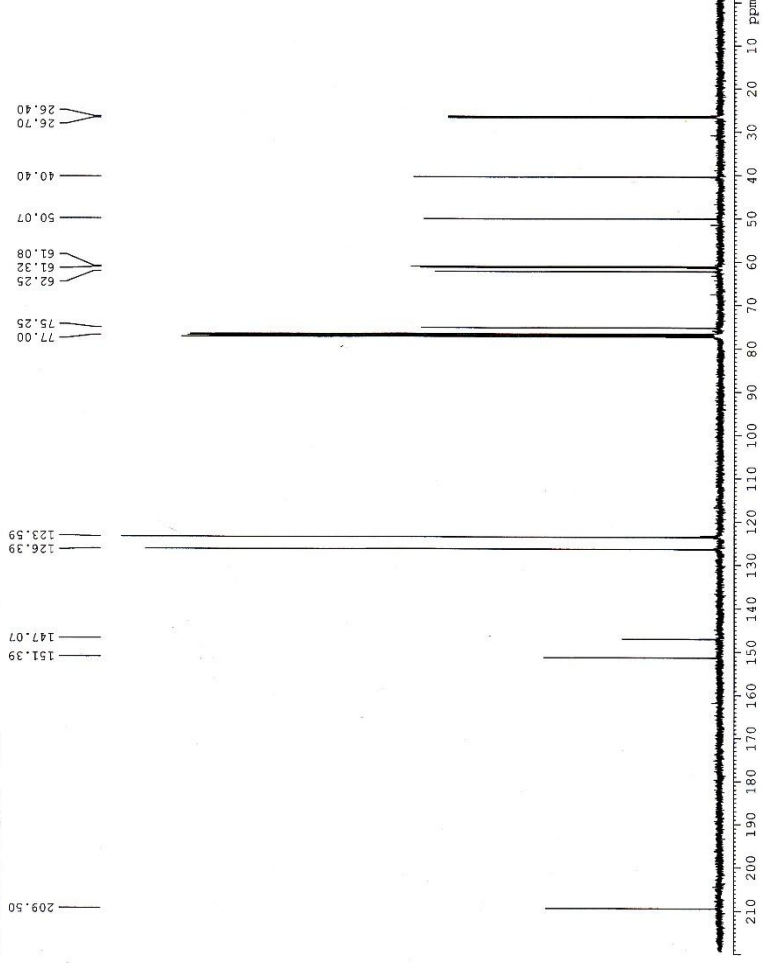
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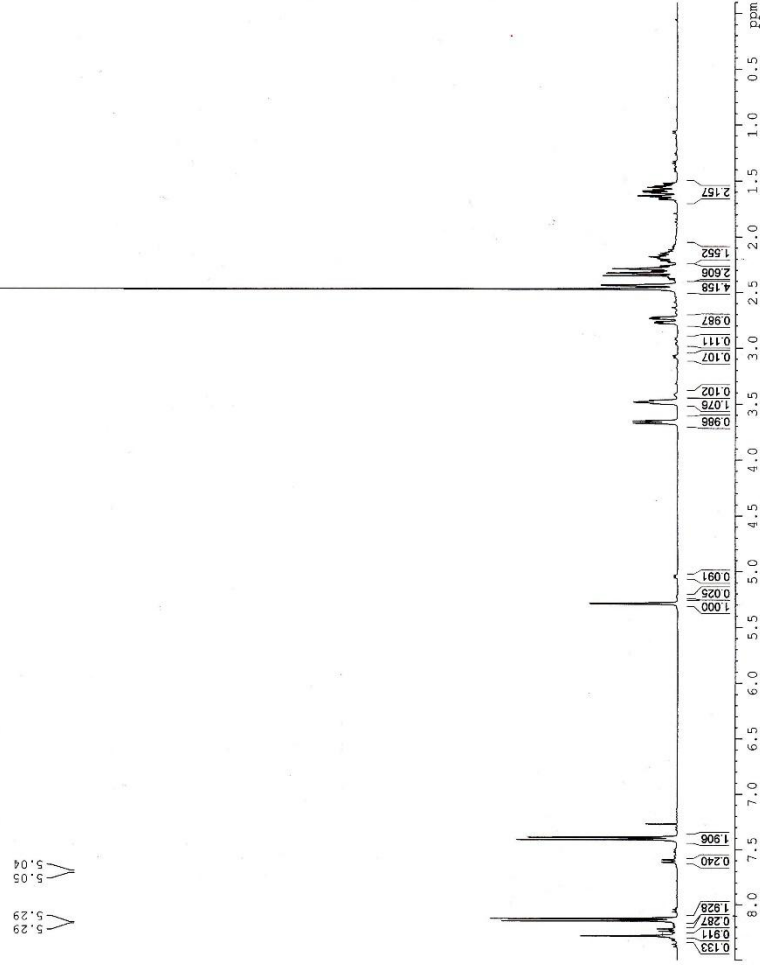
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AN 419C I kryst. 13C



## ABO 64/1 kryšt.



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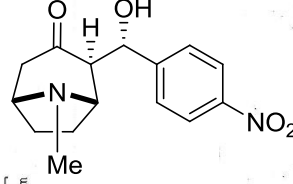
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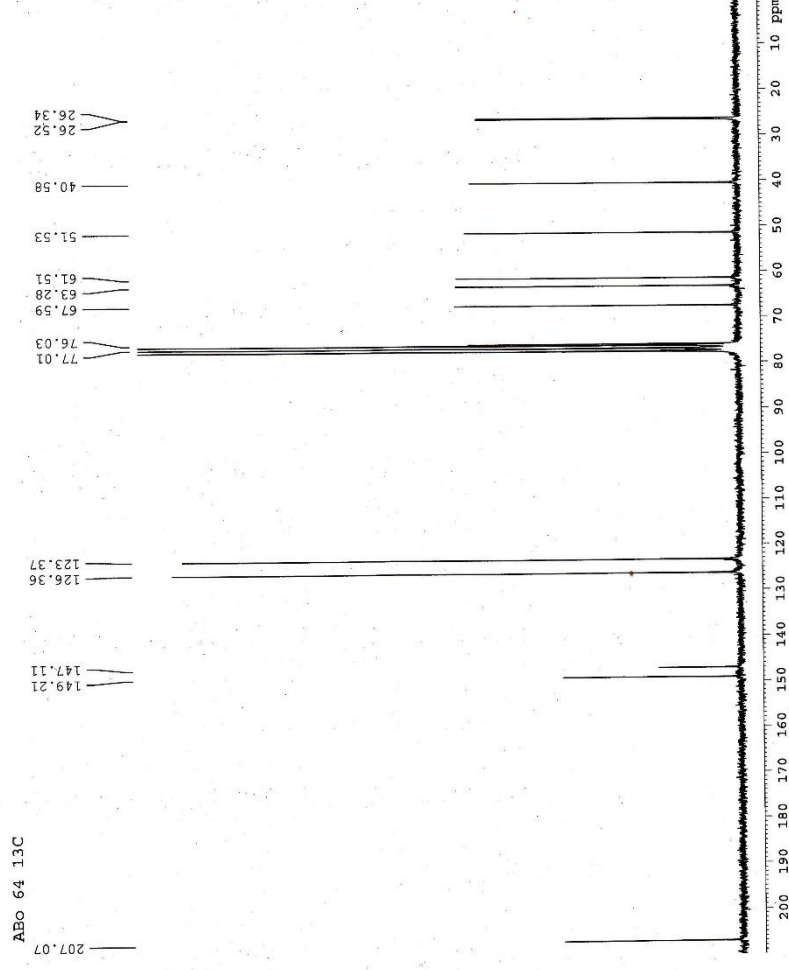
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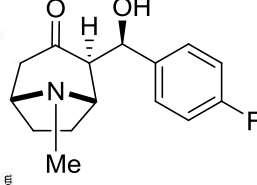
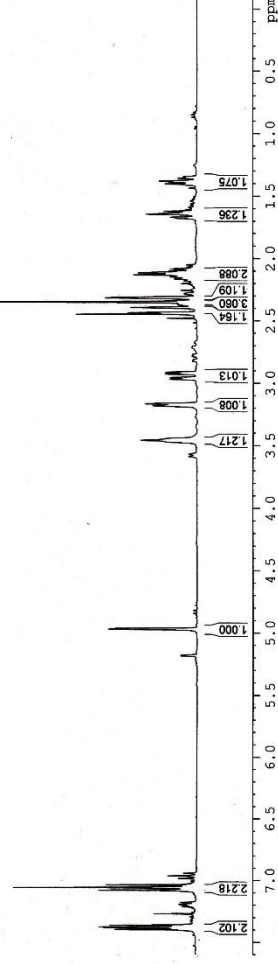
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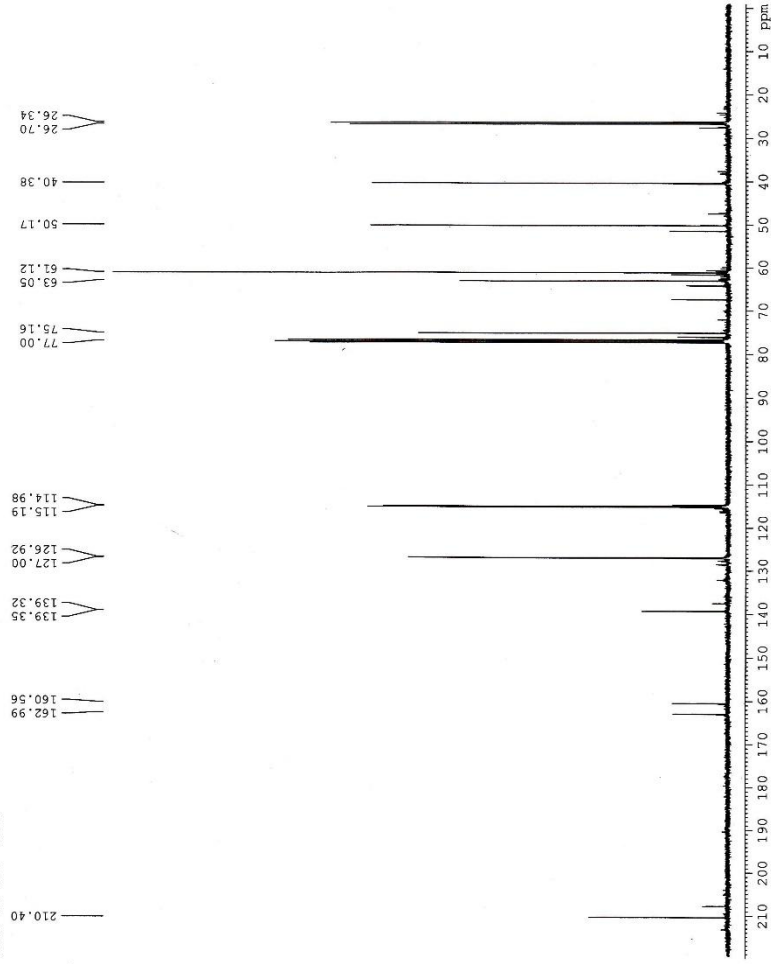
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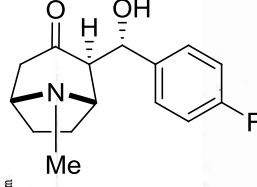
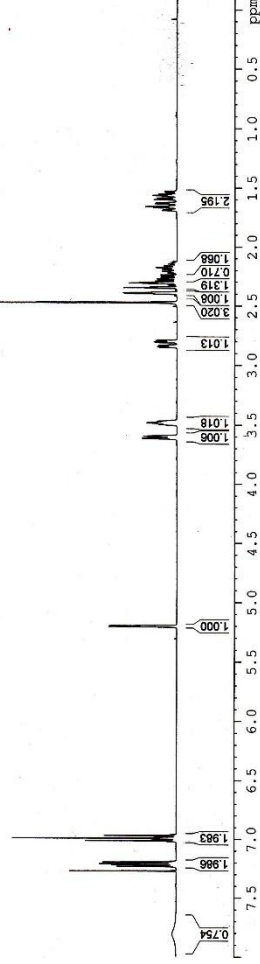
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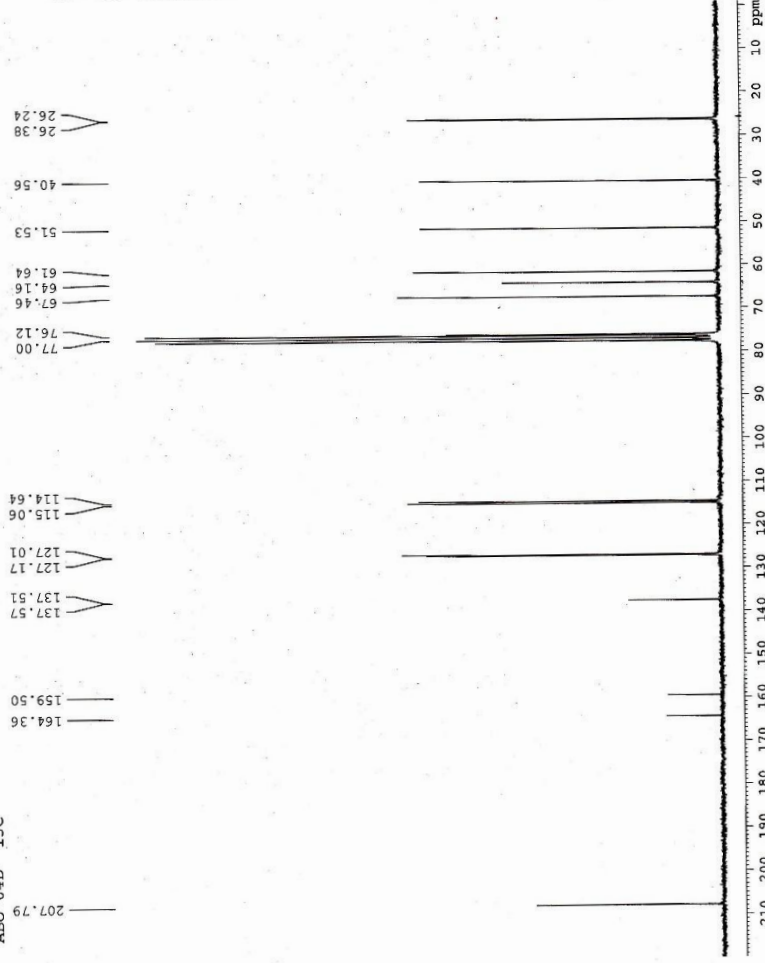
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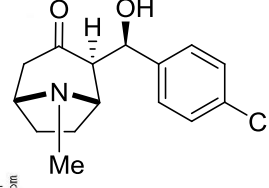
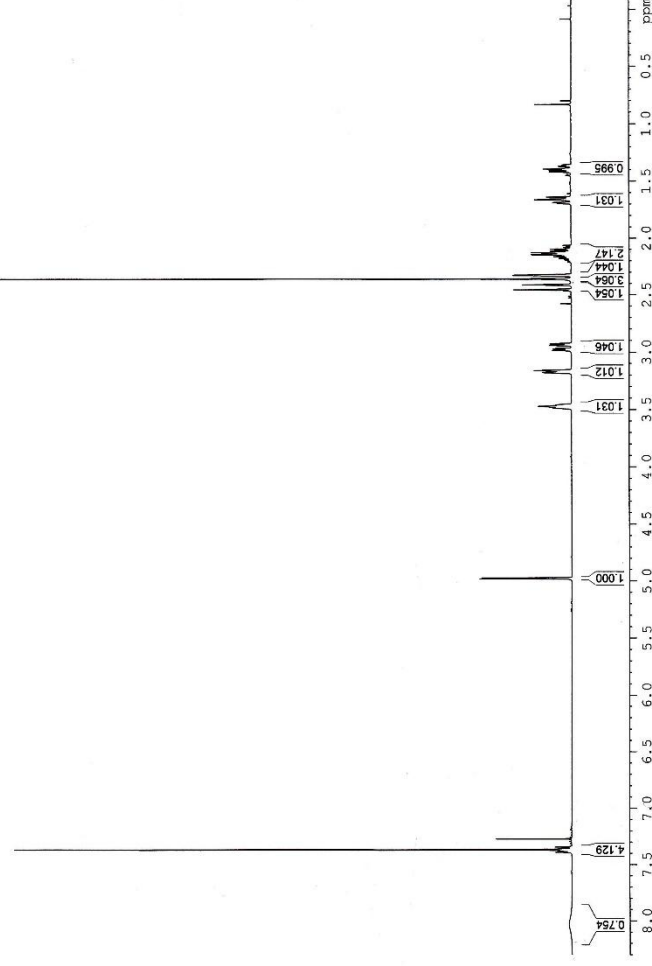




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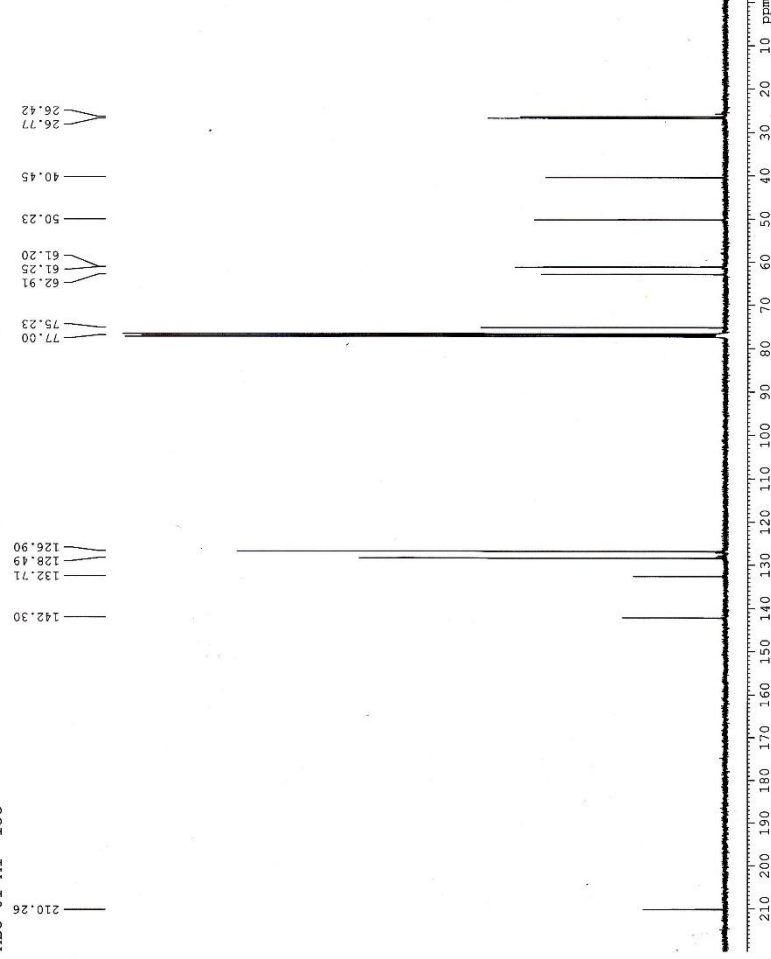
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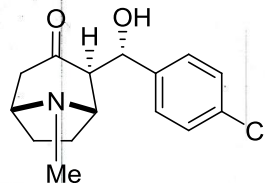
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*exo,anti*-2-((4-chlorophenyl)(hydroxy)methyl)-8-methyl-8-azabicyclo[3.2.1]octan-3-one (*exo,anti*-3d)

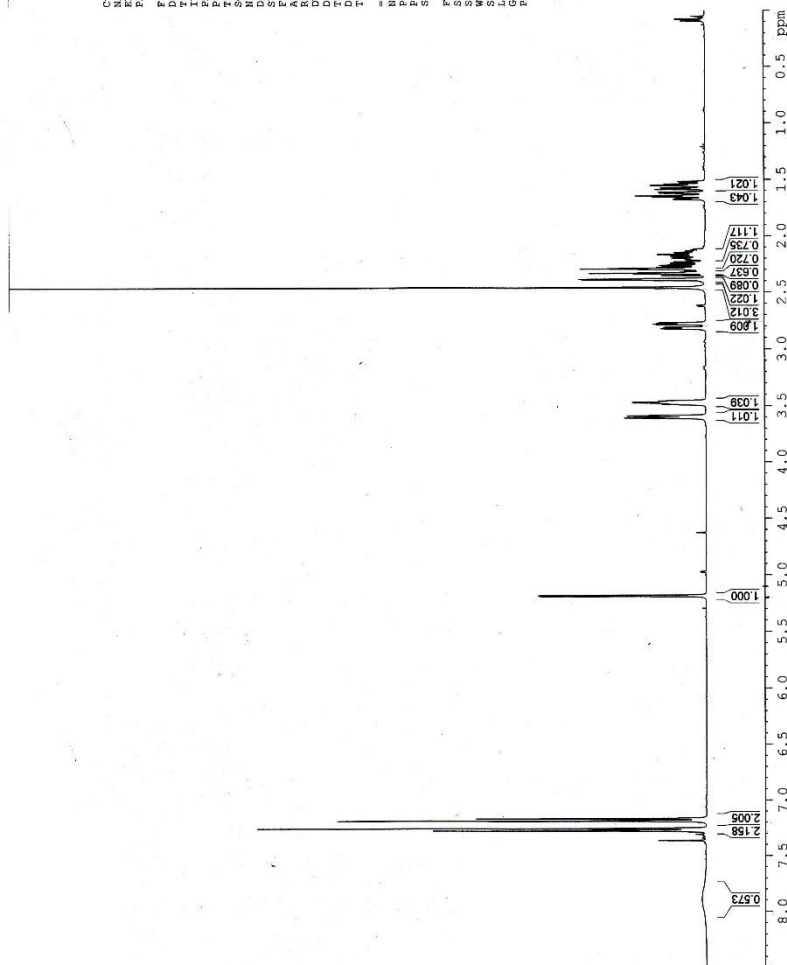


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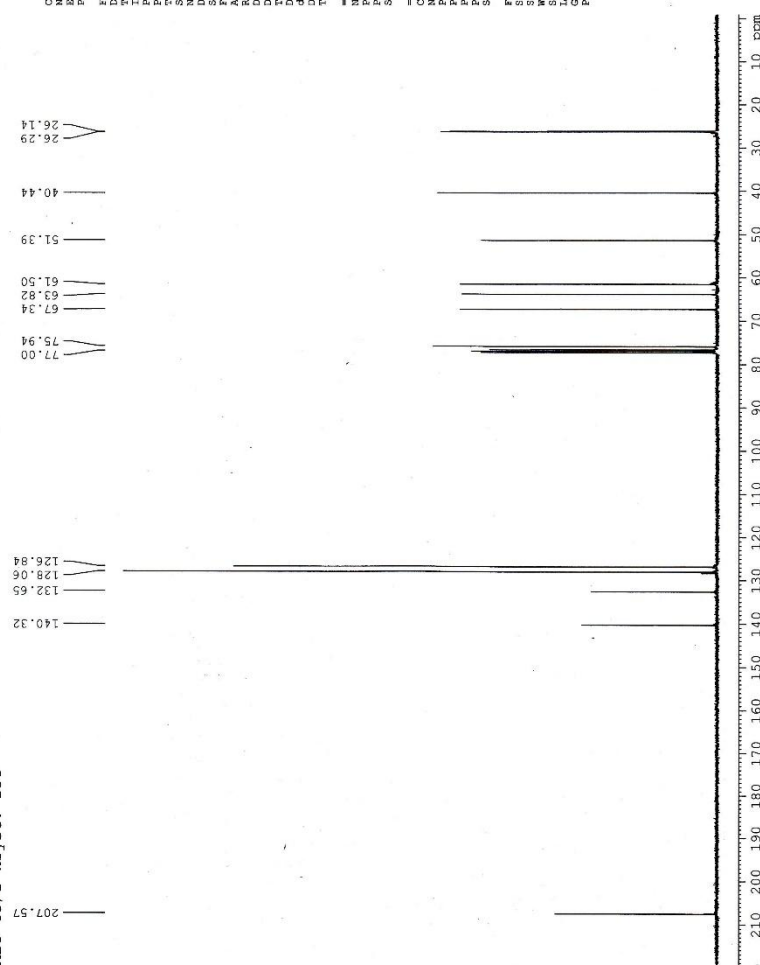
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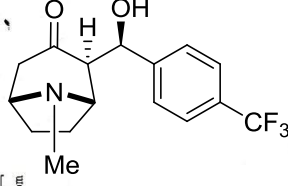
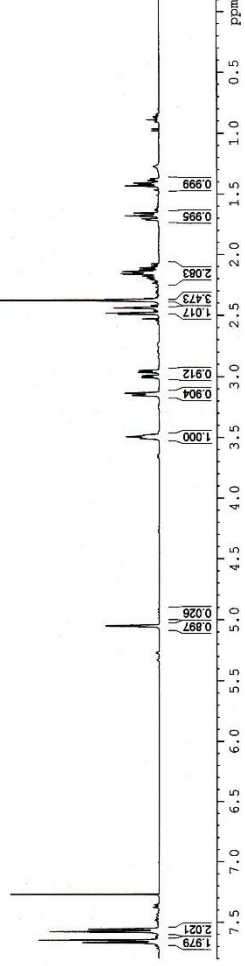


Also 63/I kryst. 13C



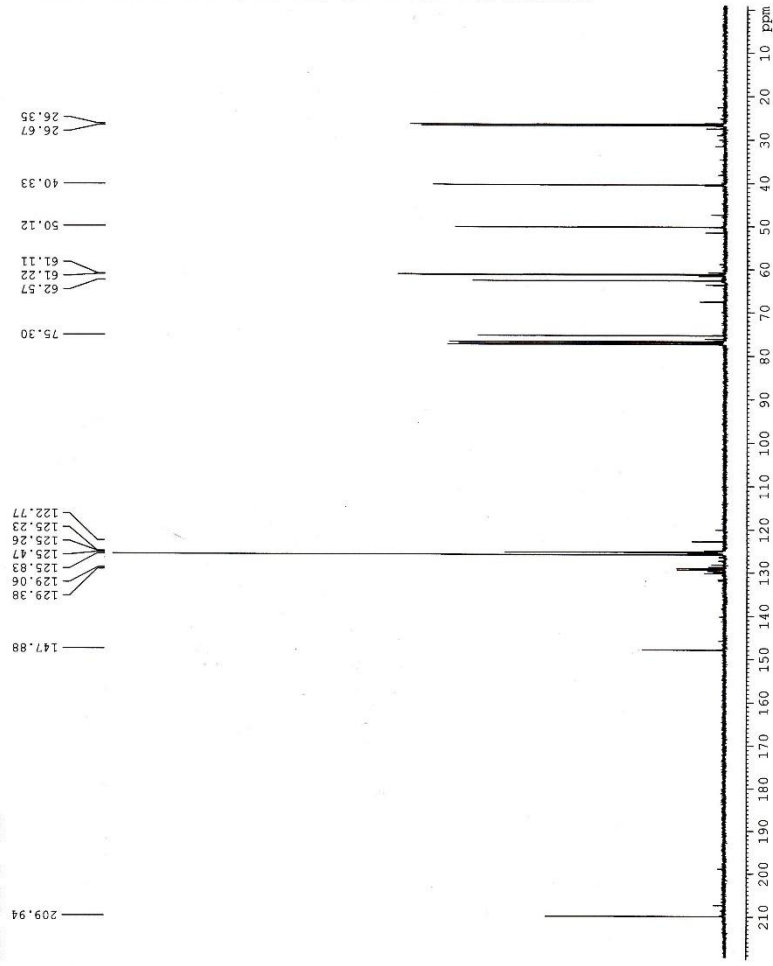
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GB 0  
PC 1.00



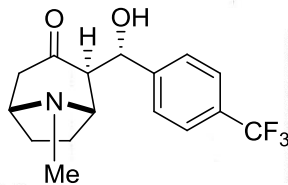
ABO 01C-3 13C

Current Data Parameters  
NAME ABO 01C-3  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20101111  
Time 11.31  
INSTRUM spect  
PULPROG zgpg30  
TD 65536  
SOLVENT CHL3  
DS 4  
SWH 24032.461 Hz  
FIDRES 0.225693 Hz  
AQ 1.3631988 sec  
RG 322  
DE 60.322 usec  
TE 289.5 K  
TDO 1.0000000 sec  
DELTA 1.8995998 sec  
CHANNEL f1  
NUC1 13C  
P1 11.00 usec  
SFO1 100.6278593 MHz  
SI - Processing parameters  
SF 100.6178593 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40

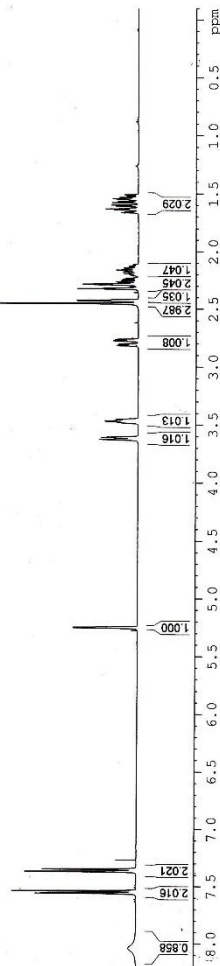


ABO 61/1

***exo,anti*-2-(hydroxy(4-(trifluoromethyl)phenyl)methyl)-8-methyl-8-azabicyclo[3.2.1]-octan-3-one (*exo,anti*-3e)**

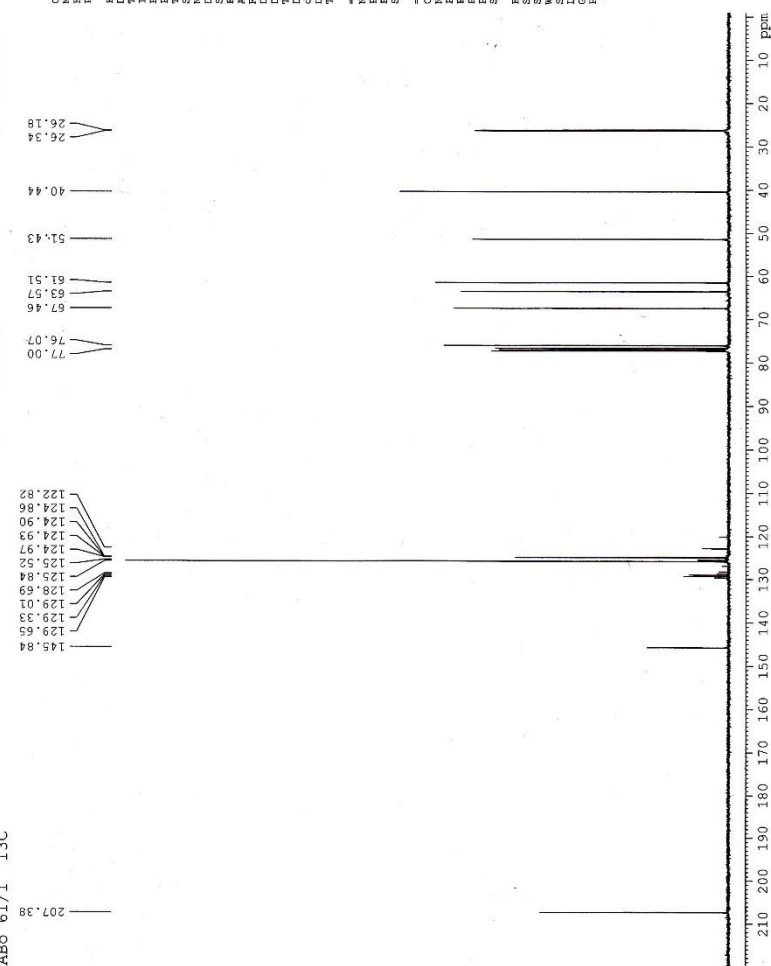


Current Data Parameters  
NAME ABO 61-1  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20131211  
Time 11:52  
INSTRUM spect  
PROBHD 5 mm RABBO 400  
PULPROG zgpg30  
TD 65536  
FIDRES 0.000000  
AQ 8.22685 Hz  
RG 3.9846387 sec  
DE 60.804 usec  
TE 288.7 K  
TDO 1.0000000 sec  
===== CHANNEL f1 =====  
NUC1 1H  
P1 11.15 usec  
PL1 0.00 dB  
SFO1 400.1524711 MHz  
F2 - Processing parameters  
SI 400.1500000 MHz  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB -0.20 Hz  
GB 0.2  
FC 1.00

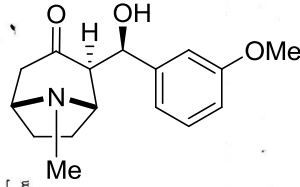
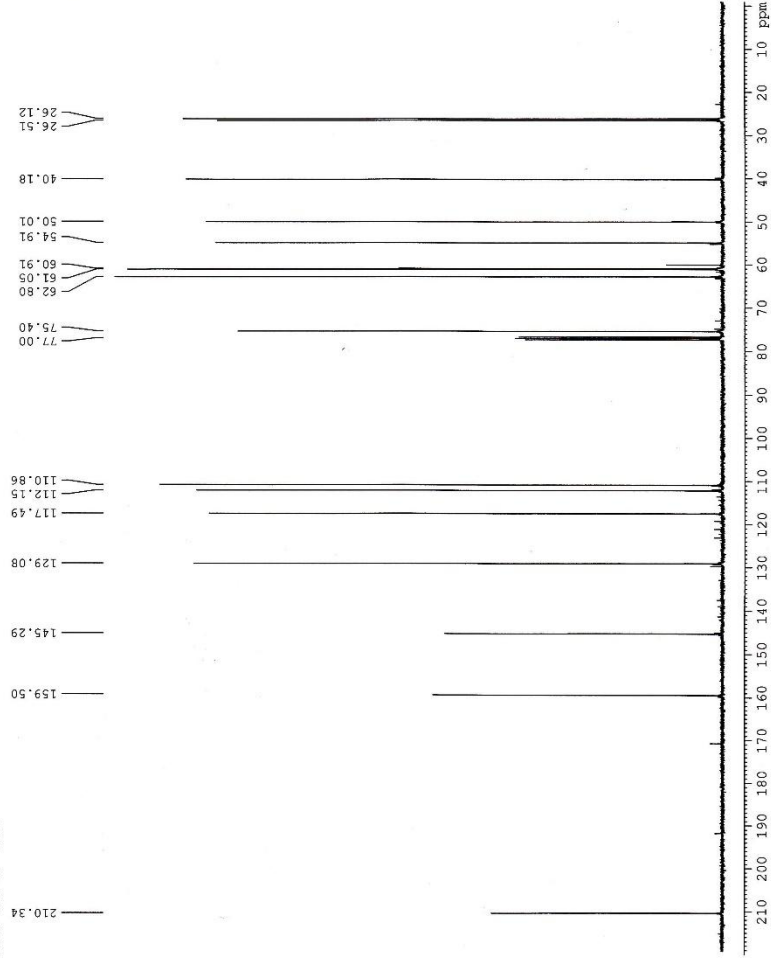


ABO 61/1 13C

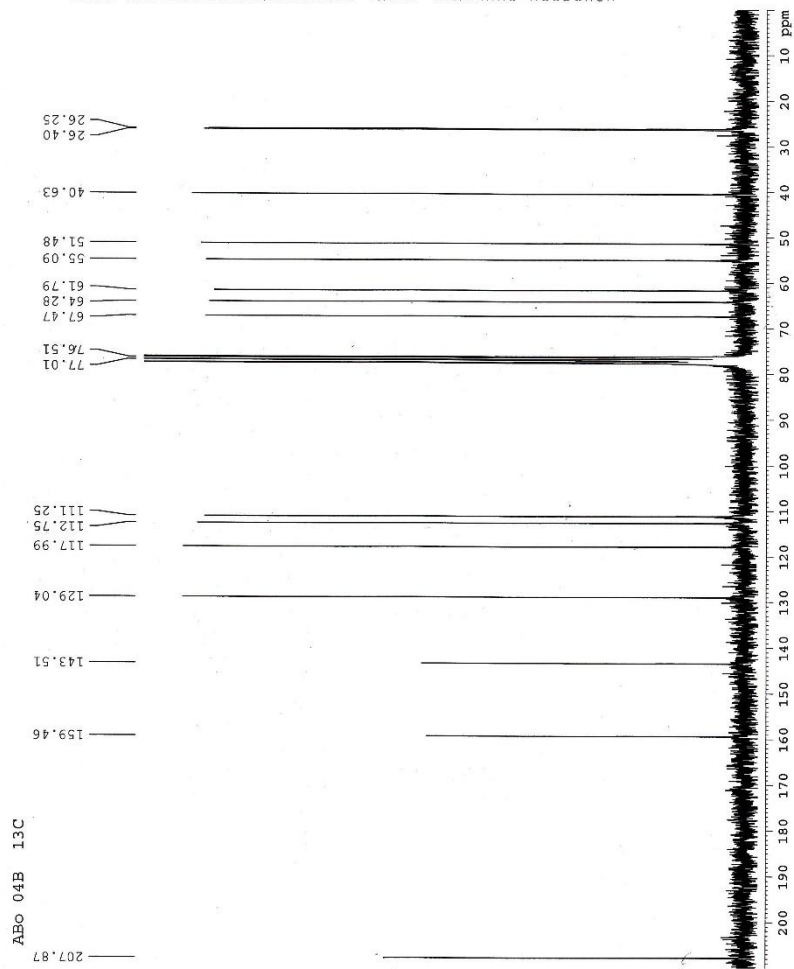
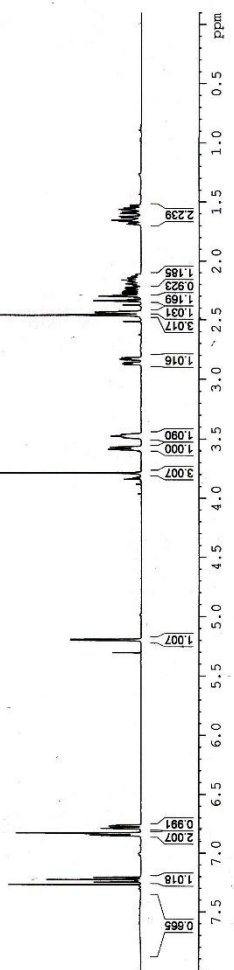
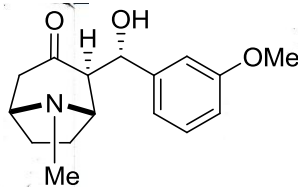
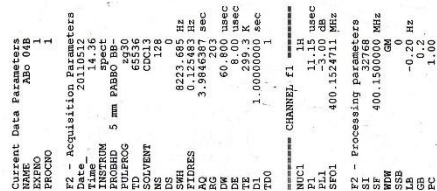
Current Data Parameters  
NAME ABO 61-1  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20131211  
Time 11:55  
INSTRUM spect  
PROBHD 5 mm RABBO 400  
PULPROG zgpg30  
TD 65536  
FIDRES 0.000000  
AQ 8.22685 Hz  
RG 3.9846387 sec  
DE 60.804 usec  
TE 288.7 K  
TDO 1.0000000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 11.00 usec  
PL1 -1.00 dB  
SFO1 100.625751 MHz  
F2 - Processing parameters  
SI 100.625751 MHz  
SF 100.625751 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
FC 1.40



Current Data Parameters		F2 - Acquisition Parameters		CHANNEL #1	
NAME	ABO 02B	Time	201557	PUL1	11.15 used
EXPNO	1	Time2	201557	SP1	-3.00 dB
PROCNO	1	INSTRUM	Spect	SP01	400.152411 MHz
		PROBHD	5 mm PABBO 1H		
		TD	4536	F2 - Processing parameters	
		SOLVENT	CDCl3	F1	31.2266
		DSH	0	WDW	400.150000 MHz
		SWH	8223.85 Hz	GB	0
		RG	3.5846757 sec	PC	-0.02 Hz
		RC	21.5	GB	1.00
		DE	60.000 used		
		TE	298.8 K		
		D1	1.0000000 sec		
		D10	1		

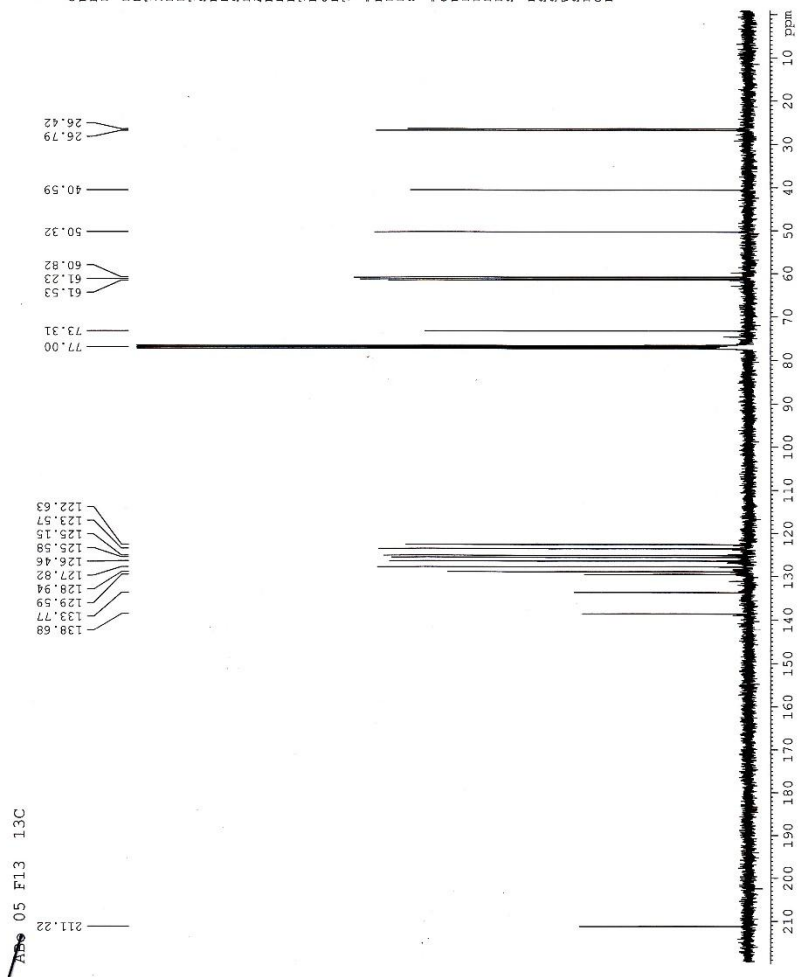
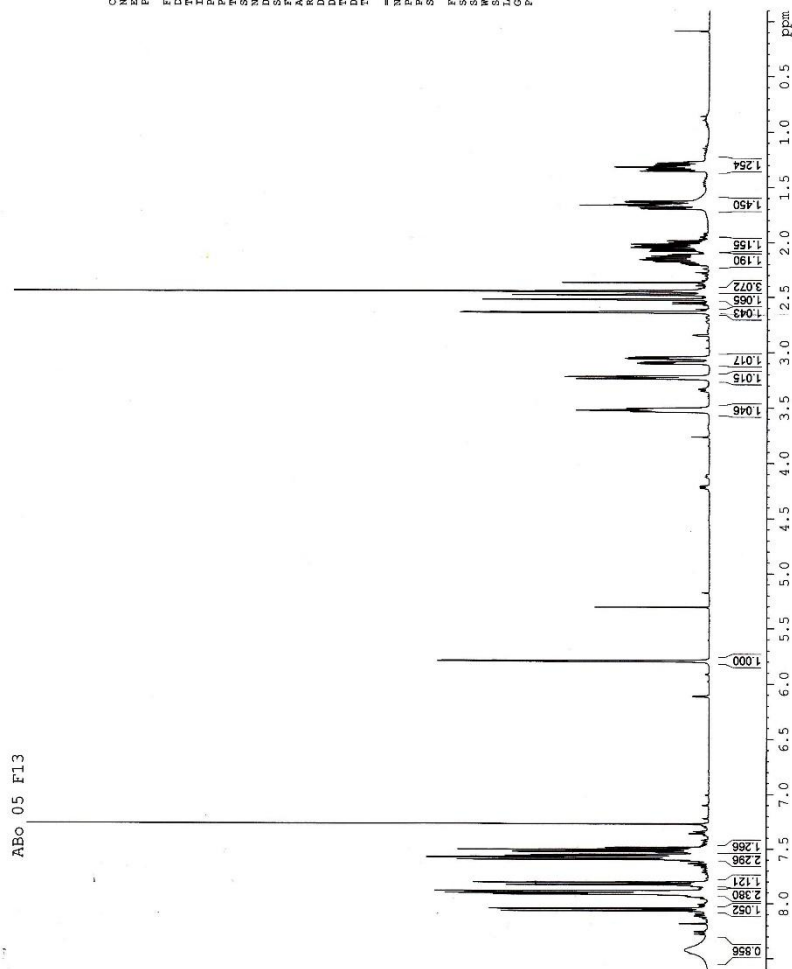
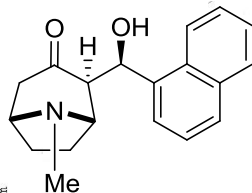
[illegible]

ABO 04 B





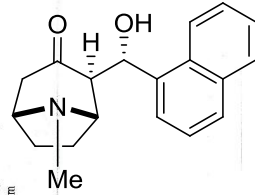
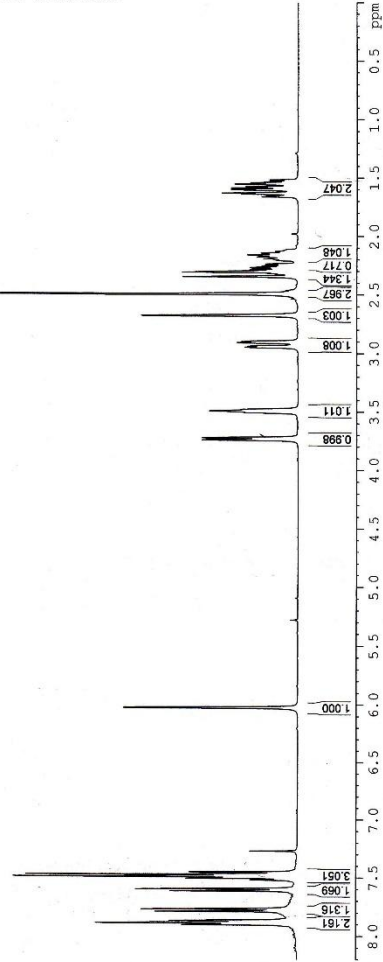
*exo,syn*-2-(hydroxy(naphthalen-2-yl)methyl)-8-methyl-8-azabicyclo[3.2.1]octan-3-one (*exo,syn*-3g)



ABO 62 (1 kryst.)

Current Data Parameters  
 EXNO ABO2(kryst.) 1  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20120127  
 Time\_ 10:55  
 INSTRUM spect  
 PROBD 5 mm PABO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 60  
 DS 4  
 SWH 8223.685 Hz  
 FIDRES 0.125483 Hz  
 RG 3.946000 sec  
 DW 60.800 usec  
 DE 19.000 usec  
 TE 298.1 K  
 D1 1.00000000 sec  
 TDO 1

===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.15 usec  
 PL1 -3.00 dB  
 SFO1 400.1524711 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 400.150000 MHz  
 WDW GM  
 SSF 0  
 GB 0.2 Hz  
 PC 1.00



ABO 62 (1 kryst.) CL3



Current Data Parameters  
 EXNO ABO2(kryst.) 2  
 PROCNO 1  
 F2 - Acquisition Parameters  
 Date\_ 20120127  
 Time\_ 11:14  
 INSTRUM spect  
 PROBD 5 mm PABO BB-  
 PULPROG zgpg30  
 TD 65536  
 SOLVENT CDCl3  
 NS 767  
 DS 4  
 SWH 24038.461 Hz  
 FIDRES 0.36098 Hz  
 RG 1.350000 sec  
 DW 20.800 usec  
 DE 19.000 usec  
 TE 298.2 K  
 D1 1.00000000 sec  
 D11 0.10000000 sec  
 DELTA 1.8959998 sec  
 TDO 1

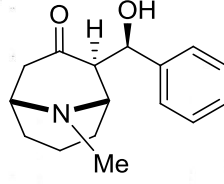
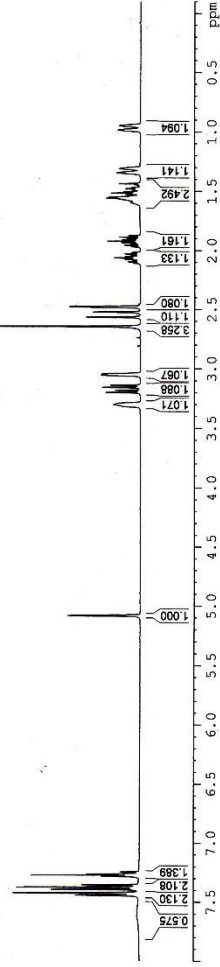
===== CHANNEL f1 =====  
 NUC1 13C  
 P1 11.15 usec  
 PL1 -3.00 dB  
 SFO1 100.6278593 MHz  
 F2 - Processing parameters  
 SI 32768  
 SF 100.6178596 MHz  
 WDW GM  
 SSF 0  
 GB 0.2 Hz  
 PC 1.40

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm



KS 309 egzo, syn kryst.

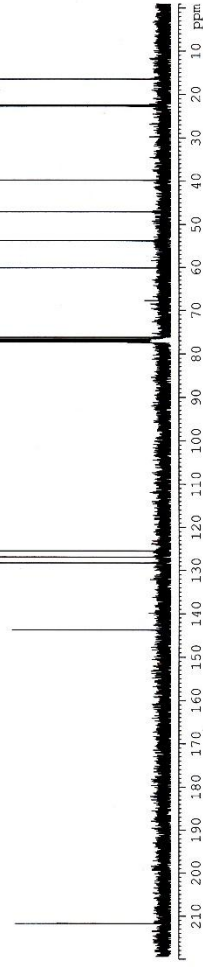
Current Data Parameters  
NAME KS 309egzo syn kryst  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20070726  
Time 16:22  
INSTRUM spect  
PROBHD 5 mm PABBO-BS  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 2  
SWH 8223.680 Hz  
FIDRES 0.125483 Hz  
AQ 0.00000000 sec  
RG 3.9846314 sec  
BW 60.800 usec  
WDW EM  
SSB 0  
TE 298.2 K  
D1 1.00000000 sec  
D0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 22.30 usec  
PL1 -3.00 dB  
SFO1 400.1524711 MHz  
F2 - Processing parameters  
SF 400.1500000 MHz  
WDW GM  
SSB 0  
TE -0.20 Hz  
GB 0.02  
PC 1.00



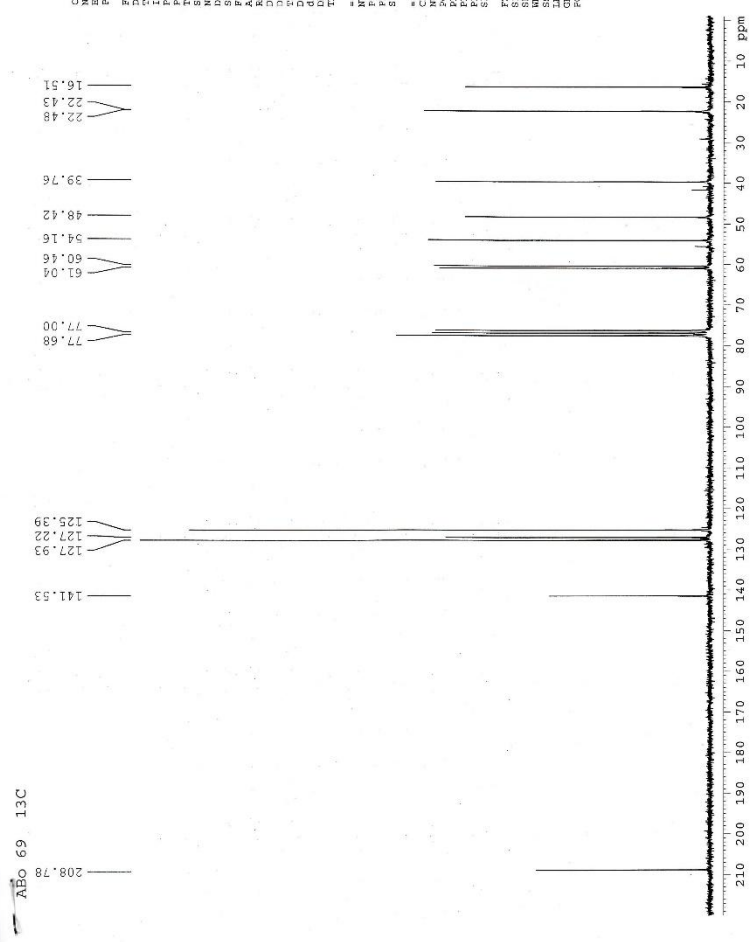
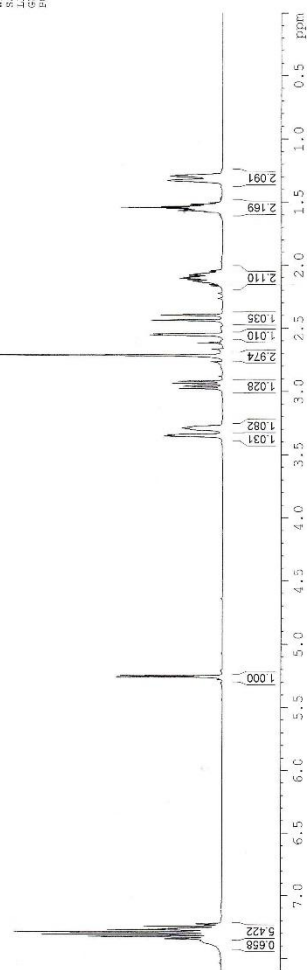
KS 309 egzo, syn kryst. 13C



Current Data Parameters  
NAME KS 309egzo syn kryst  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20070726  
Time 17:02  
INSTRUM spect  
PROBHD 5 mm PABBO-BS  
PULPROG zg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 2  
SWH 8223.680 Hz  
FIDRES 0.125483 Hz  
AQ 0.00000000 sec  
RG 3.9846314 sec  
BW 60.800 usec  
WDW EM  
SSB 0  
TE -0.20 Hz  
D1 1.00000000 sec  
D0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 22.30 usec  
PL1 -3.00 dB  
SFO1 400.1524711 MHz  
F2 - Processing parameters  
SF 400.1500000 MHz  
WDW GM  
SSB 0  
TE -0.20 Hz  
GB 0.02  
PC 1.00



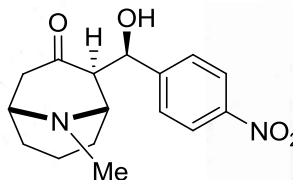
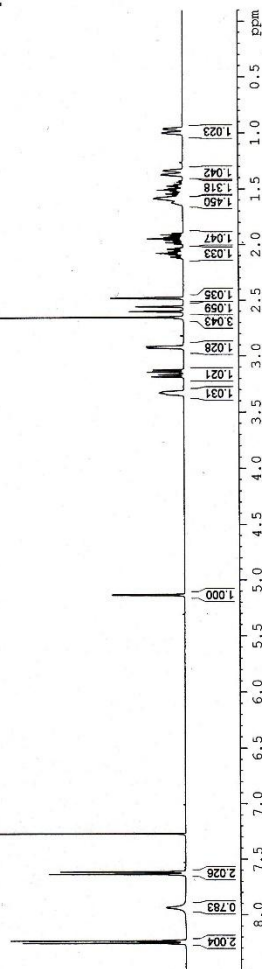
The chemical structure shows a bicyclic system consisting of a six-membered ring fused to a five-membered ring. The nitrogen atom is part of the five-membered ring and has a methyl group attached. The six-membered ring contains a carbonyl group at position 6 and a hydroxyl group at position 7. A phenyl group is attached to the carbon at position 8. Stereochemistry is indicated with wedges and dashes.

CN1CCC(=O)[C@H](O)[C@@H](Cc2ccccc2)CC1

AN aldol

Current Data Parameters  
NAME AN aldol  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20011117  
Time 10.48  
INSTRUM spect  
PROBHD 5 mm PABBO  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 270  
SWH 823.685 Hz  
FIDRES 0.358 Hz  
AQ 3.946387 sec  
RG 287  
GB 60.00 usec  
DE 8.00 usec  
TE 298.7 K  
D1 1.0000000 sec  
TDO 1

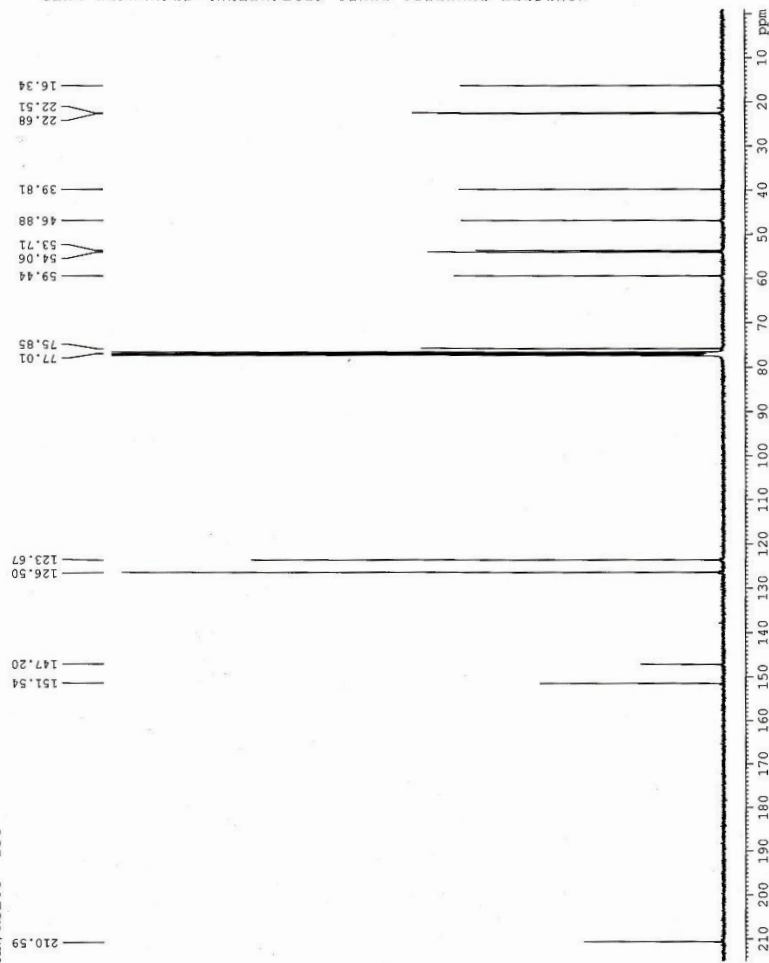
===== CHANNEL f1 =====  
NUC1 1H  
P1 11.15 usec  
PL1 0.00 dB  
SFO1 400.1524711 MHz  
F2 - Processing parameters  
SI 32768  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB 0.20 Hz  
GB 0.2  
PC 1.00



AN/KS208 13C

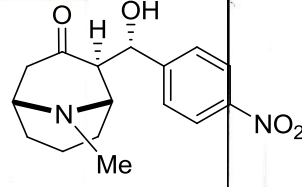
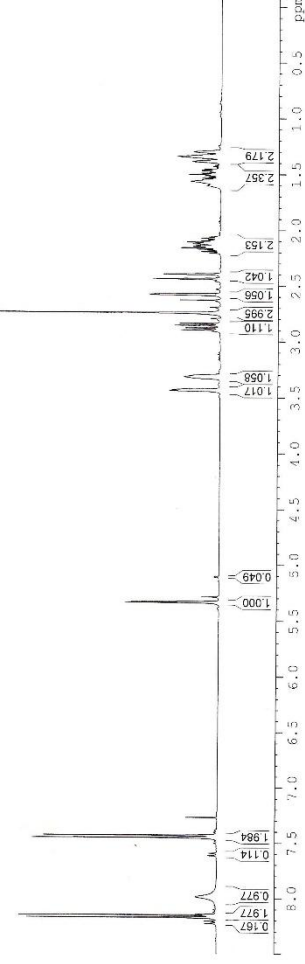
Current Data Parameters  
NAME AN-KS208  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20011117  
Time 17.49  
INSTRUM spect  
PROBHD 5 mm PABBO  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 270  
SWH 27132.21 Hz  
FIDRES 0.41461 Hz  
AQ 1.2058124 sec  
RG 64  
GB 60.00 usec  
DE 6.00 usec  
TE 299.7 K  
D1 0.0100000 sec  
D11 0.0100000 sec  
DETA 1.8599998 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 9.30 usec  
PL1 -1.00 dB  
SFO1 100.628660 MHz  
===== CHANNEL f2 =====  
NAME waitzr1n  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20011117  
Time 17.49  
INSTRUM spect  
PROBHD 5 mm PABBO  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 270  
SWH 27132.21 Hz  
FIDRES 0.41461 Hz  
AQ 1.2058124 sec  
RG 64  
GB 60.00 usec  
DE 6.00 usec  
TE 299.7 K  
D1 0.0100000 sec  
D11 0.0100000 sec  
DETA 1.8599998 sec  
TDO 1



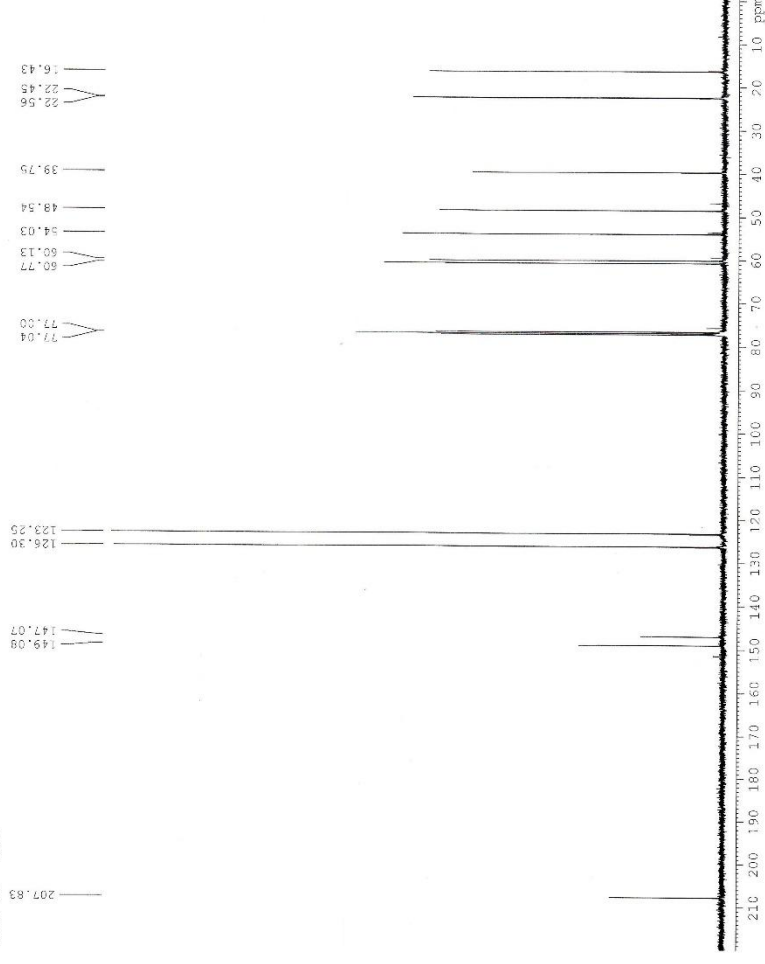
ABO 72

Current Data Parameters  
Date\_ 20120220  
Time\_ 11:15  
INSTRUM spect  
PROBHD 5 mm PABO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 32  
SWH 8223.685 Hz  
FIDRES 0.115483 Hz  
RG 32768.57 sec  
AQ 60.800 usec  
F2 - Processing Parameters  
SI 32768  
SF 400.136000 MHz  
WDW EM  
SSB 0  
GB 0.2 Hz  
PC 1.00

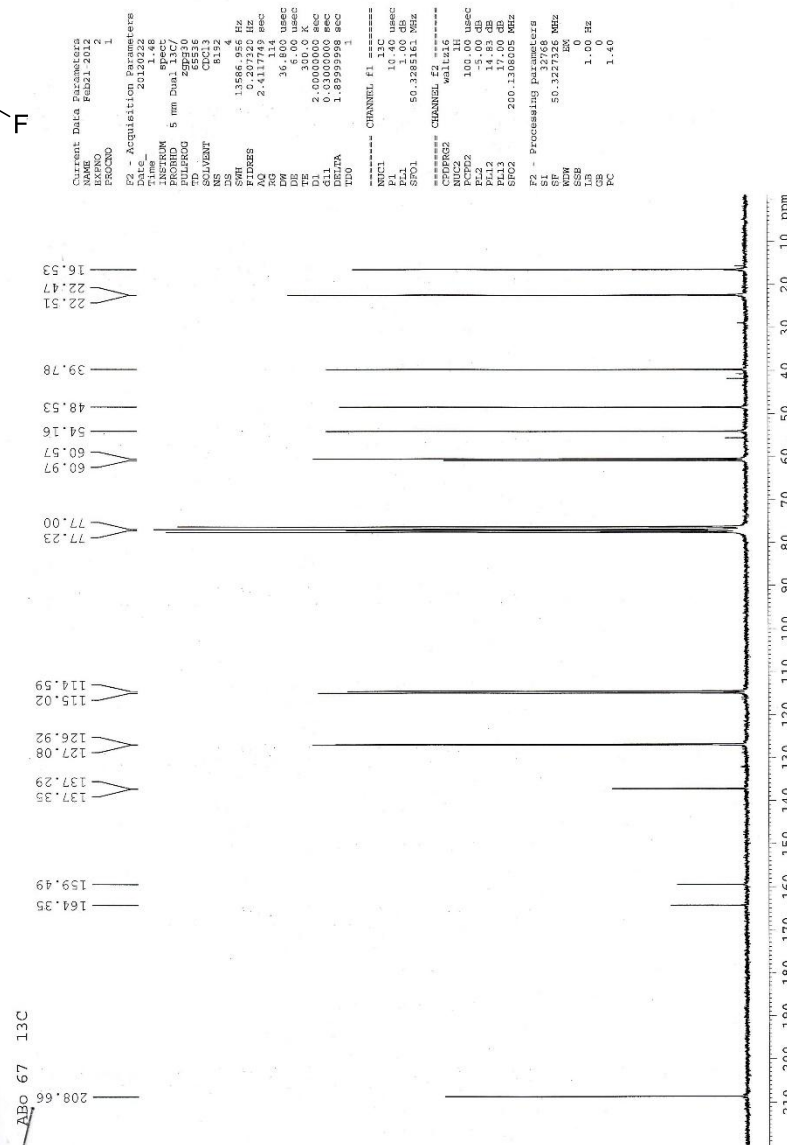
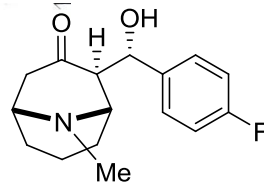
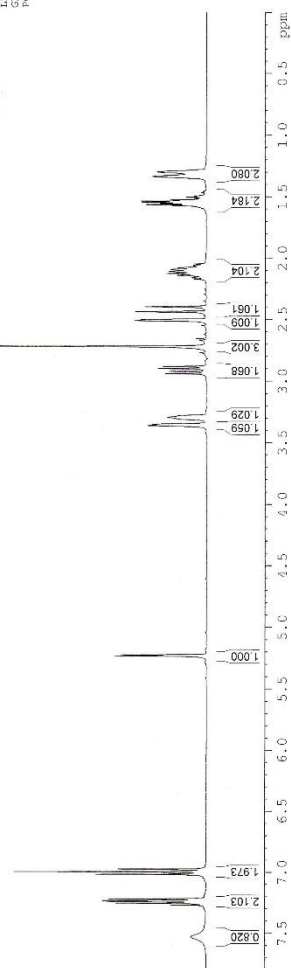
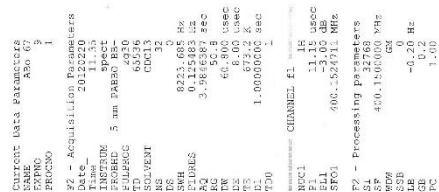


ABO 72 13C

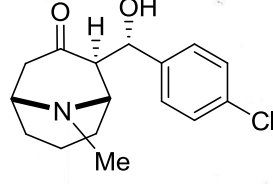
Current Data Parameters  
Date\_ 20120220  
Time\_ 11:15  
INSTRUM spect  
PROBHD 5 mm PABO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 32  
SWH 27173.912 Hz  
FIDRES 0.145621 Hz  
RG 32768.57 sec  
AQ 60.800 usec  
F2 - Processing Parameters  
SI 32768  
SF 400.136000 MHz  
WDW EM  
SSB 0  
GB 0.2 Hz  
PC 1.00



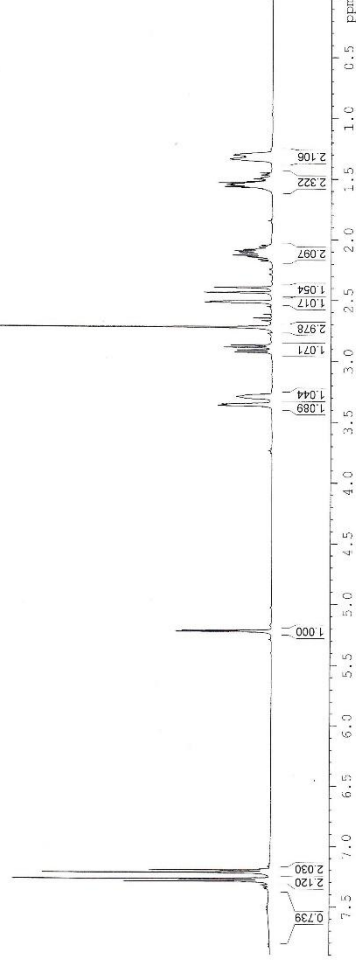
## ABO 67



***exo,anti*-2-((4-chlorophenyl)(hydroxy)methyl)-9-methyl-9-azabicyclo[3.3.1]nonan-3-one (*exo,anti*-4d)**

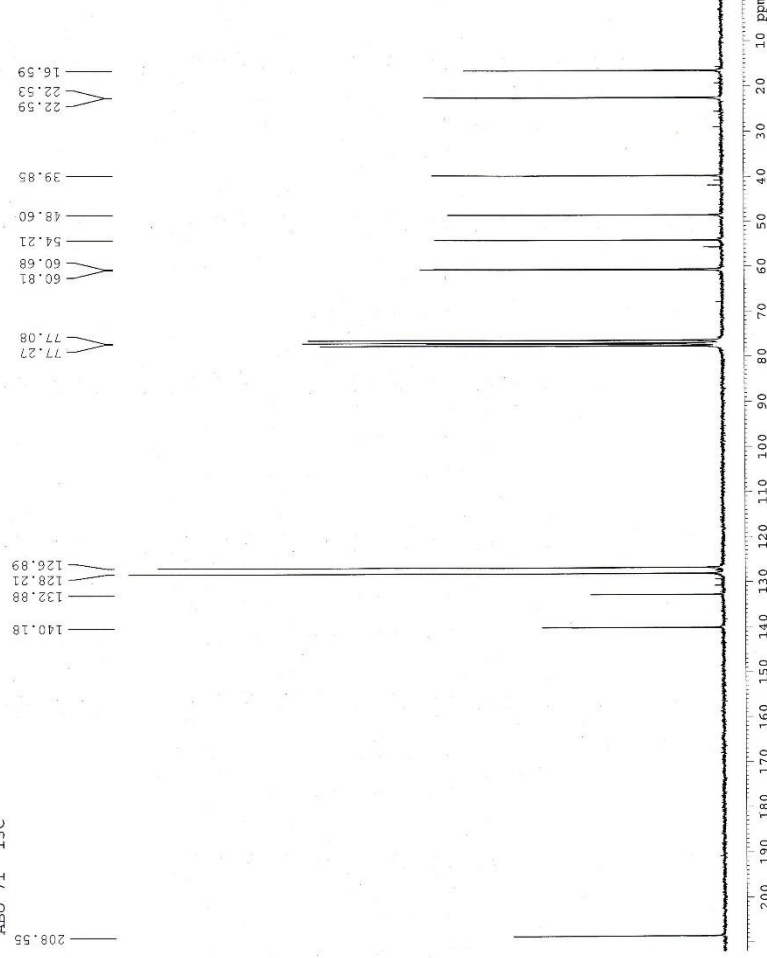


Current Data Parameters  
NAME AB071  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20100220  
Time\_ 11:15  
INSTRUM spect  
PROBHD 5 mm DABO-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 2  
DS 4  
SWH 8223.685 Hz  
FIDRES 0.125491 Hz  
RG 3.984657 um  
AQ 2.411745 sec  
RG 36.334 usec  
DE 6.00 usec  
TE 300.0 K  
D1 0.23000000 sec  
d11 1.89999996 sec  
DELTA 1.89999996 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 11.15 usec  
PL1 -3.00 dB  
SFO1 400.154711 MHz  
F2 - Processing parameters  
SI 32768  
SF 400.156000 MHz  
WDW EM  
SSB 0  
GB 0  
PC 1.00

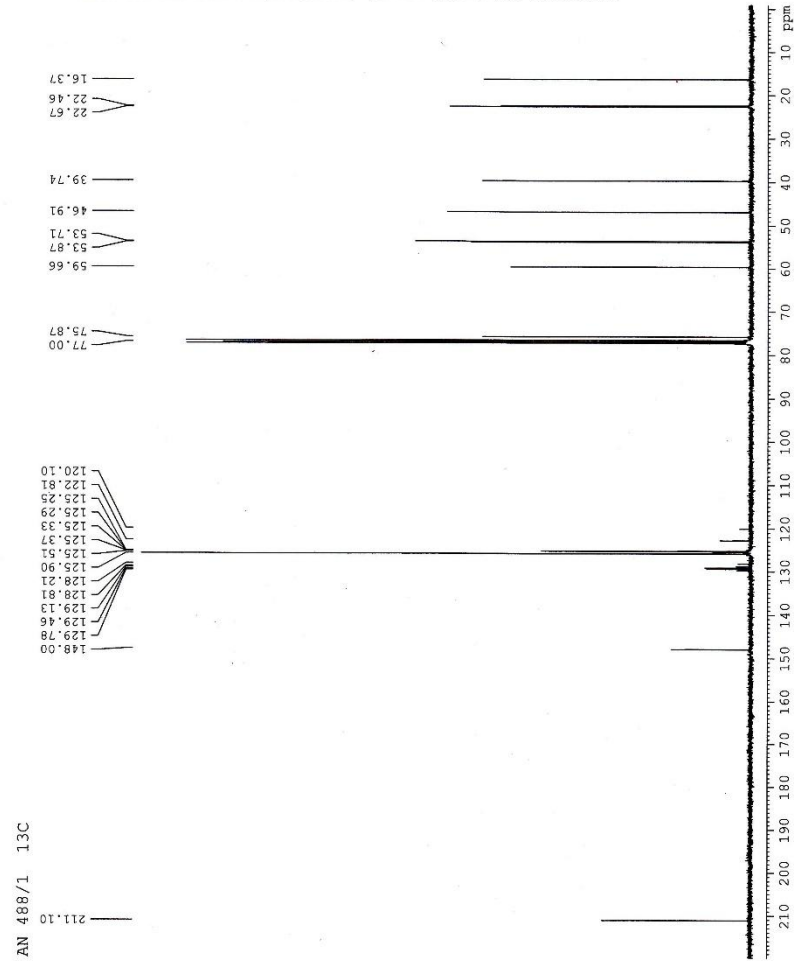
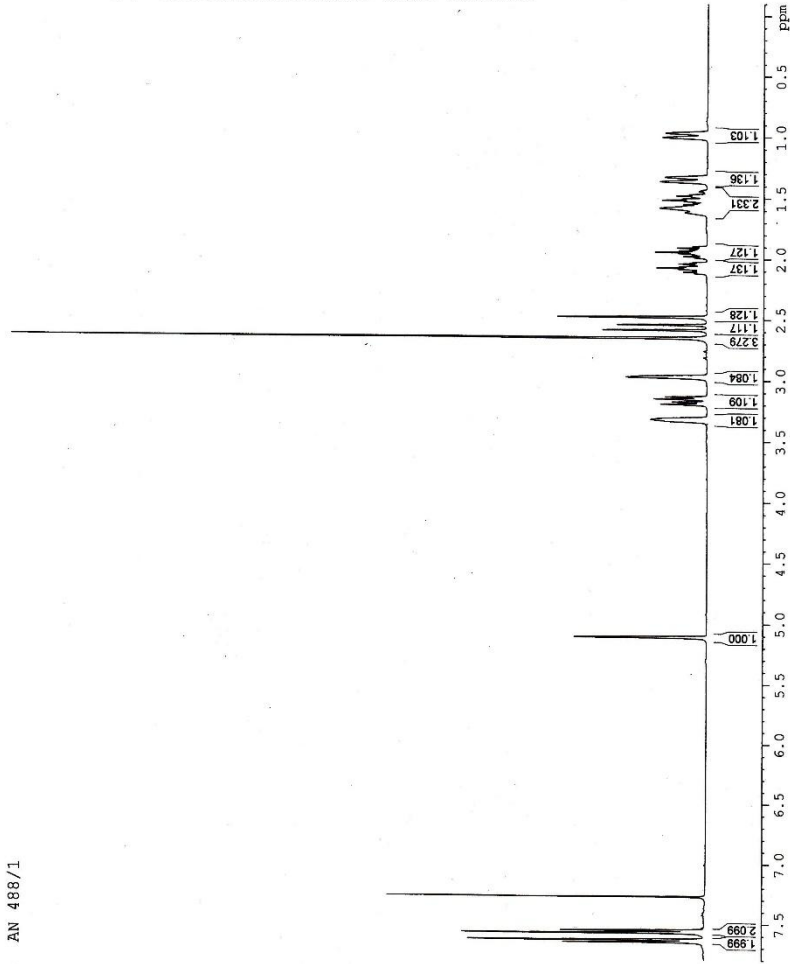
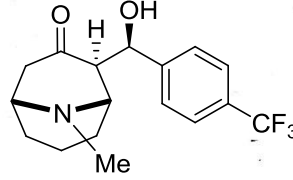


ABO 71 13C

Current Data Parameters  
NAME AB071  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20100220  
Time\_ 11:15  
INSTRUM spect  
PROBHD 5 mm DABO-1H  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 2  
DS 4  
SWH 8223.685 Hz  
FIDRES 0.125491 Hz  
RG 3.984657 um  
AQ 2.411745 sec  
RG 36.334 usec  
DE 6.00 usec  
TE 300.0 K  
D1 0.23000000 sec  
d11 1.89999996 sec  
DELTA 1.89999996 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 11.15 usec  
PL1 -3.00 dB  
SFO1 400.154711 MHz  
F2 - Processing parameters  
SI 32768  
SF 400.156000 MHz  
WDW EM  
SSB 0  
GB 0  
PC 1.00



*exo,syn*-2-(hydroxy(4-(trifluoromethyl)phenyl)methyl)-9-methyl-9-azabicyclo[3.3.1]-nonan-3-one (*exo,syn*-4e)





AN 489a

***exo,anti*-2-(hydroxy(4-(trifluoromethyl)phenyl)methyl)-9-methyl-9-azabicyclo[3.3.1]-nonan-3-one (*exo,anti*-4e)**

Current Data Parameters  
NAME AN 489a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 201214  
Time 13:57  
INSTRUM spect  
PROBHD 5 mm PABBO BBO  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 4  
SWH 823.683 Hz  
FIDRES 0.344 Hz  
AQ 3.9846387 sec  
RG 60  
DE 144  
TE 300.2 K  
D1 2.0000000 sec  
DELTA 1.8999998 sec  
TD0 1

Channel f1  
NUC1 13C  
P1 15.00 usec  
PL1 0.00 dB  
SFO1 100.6284660 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1500000 MHz  
WDW GN  
SSB 0  
LB -0.20 Hz  
GB 0.2  
PC 1.00

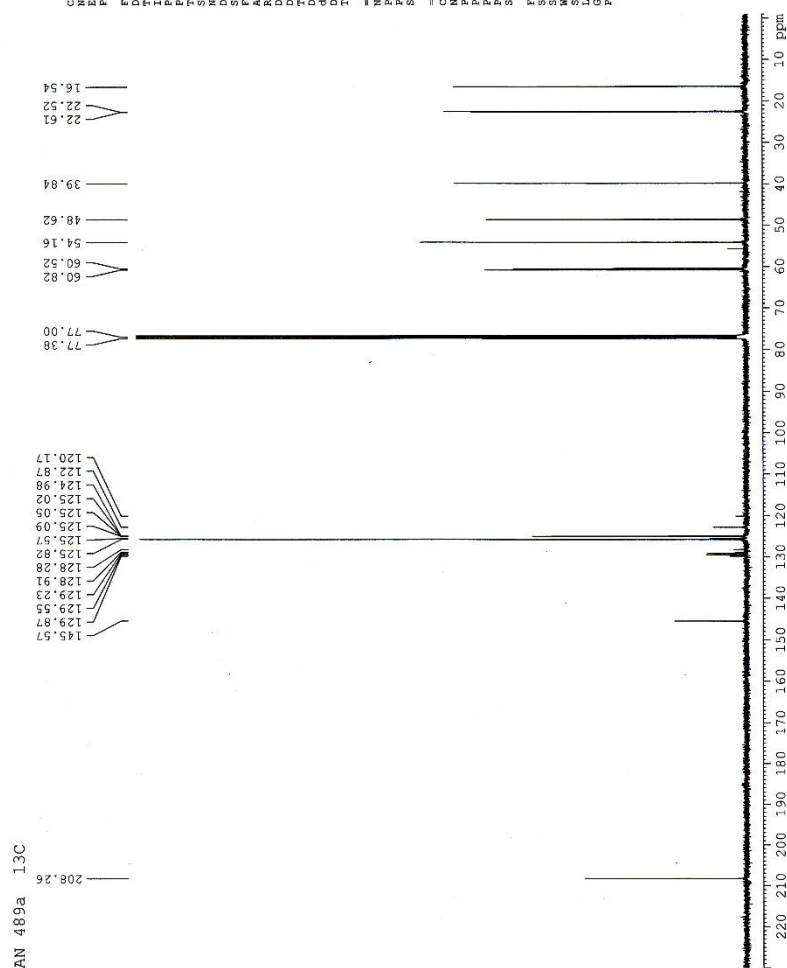
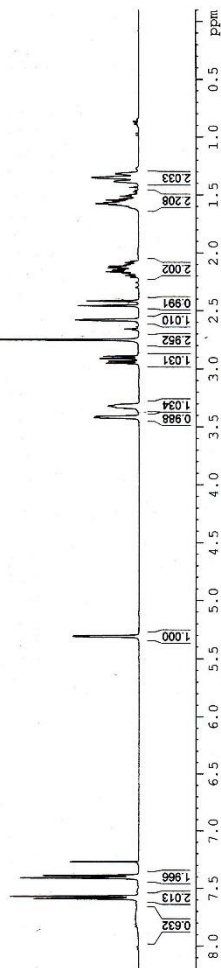
Chemical structure: CC12CCCC3(C1)C(=O)N(C2)C[C@H](C3)C[C@@H](O)Cc4ccc(C(F)(F)F)cc4

Current Data Parameters  
NAME AN 489a  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 201214  
Time 13:57  
INSTRUM spect  
PROBHD 5 mm PABBO BBO  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 4  
SWH 823.683 Hz  
FIDRES 0.344 Hz  
AQ 3.9846387 sec  
RG 60  
DE 144  
TE 300.2 K  
D1 2.0000000 sec  
DELTA 1.8999998 sec  
TD0 1

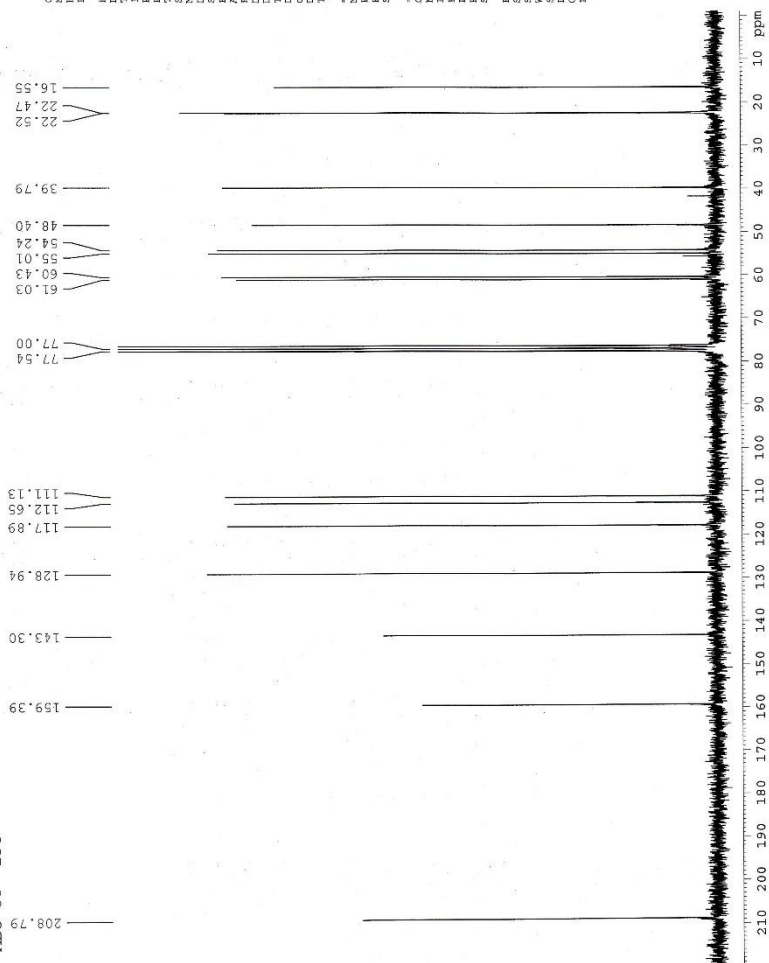
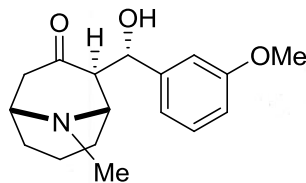
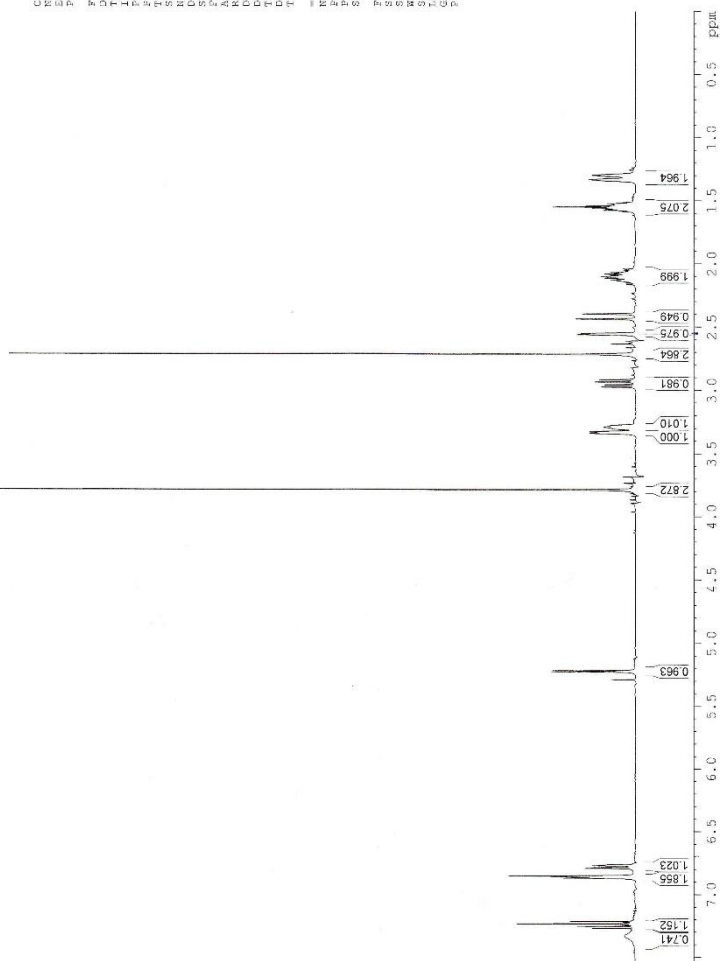
Channel f1  
NUC1 13C  
P1 15.00 usec  
PL1 0.00 dB  
SFO1 100.6284660 MHz

F2 - Processing parameters  
SI 32768  
SF 400.1500000 MHz  
WDW GN  
SSB 0  
LB -0.20 Hz  
GB 0.2  
PC 1.00





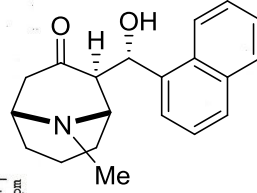
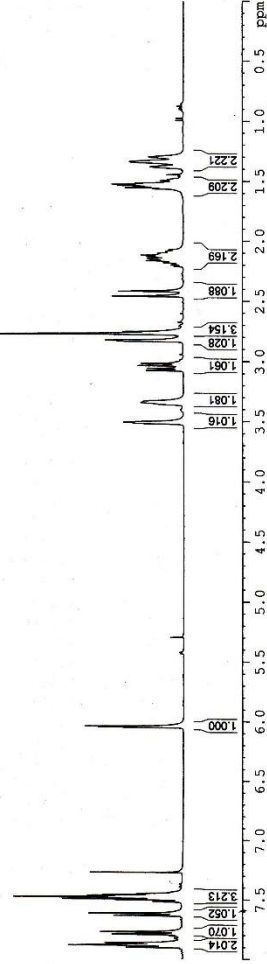
## ABO 66



PZ r03 cz. 1H

Current Data Parameters  
 Name: PZ r03  
 EXNO: 2  
 PROCNO: 1  
 Date: 20090416  
 Time: 5.19  
 INSTRUM: spect  
 PROBHD: 5 mm PABBO B3  
 PULPROG: zgpg30  
 SOLVENT: CDCl3  
 NS: 64  
 DS: 4  
 SWH: 8223.685 Hz  
 FIDRES: 0.12463 Hz  
 AQ: 3.984634 sec  
 RG: 327.68  
 DW: 60.800 usec  
 DE: 25.00 usec  
 TE: 297.8 K  
 D1: 1.0000000 sec  
 TD0: 1

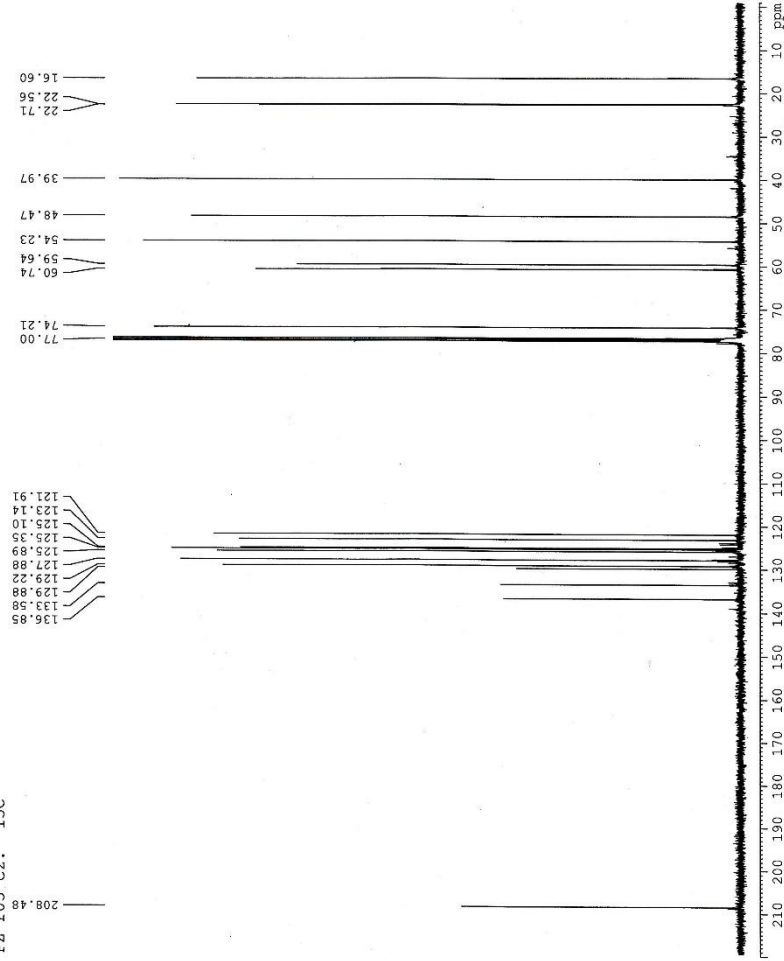
===== CHANNEL f1 =====  
 NUC1: 1H  
 P1: 11.00 usec  
 PL1: -3.00 dB  
 SF01: 400.1524711 MHz  
 F2 - Processing Parameters  
 SI: 327.68  
 SF02: 400.1500000 MHz  
 WDW: EM  
 SS: 0  
 GB: -0.22 Hz  
 PC: 1.00



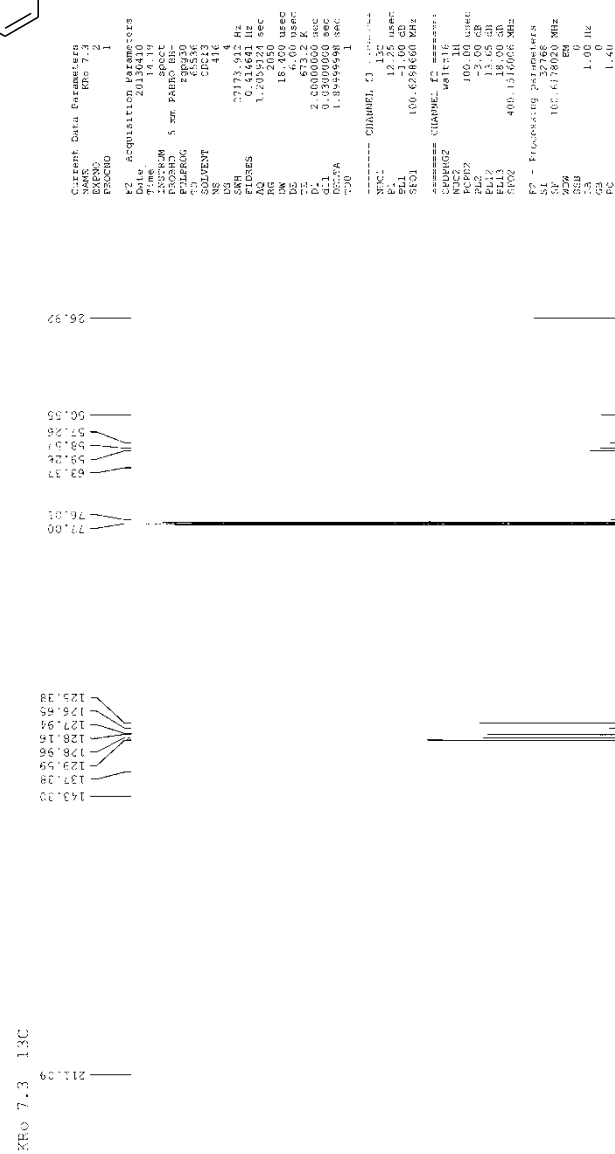
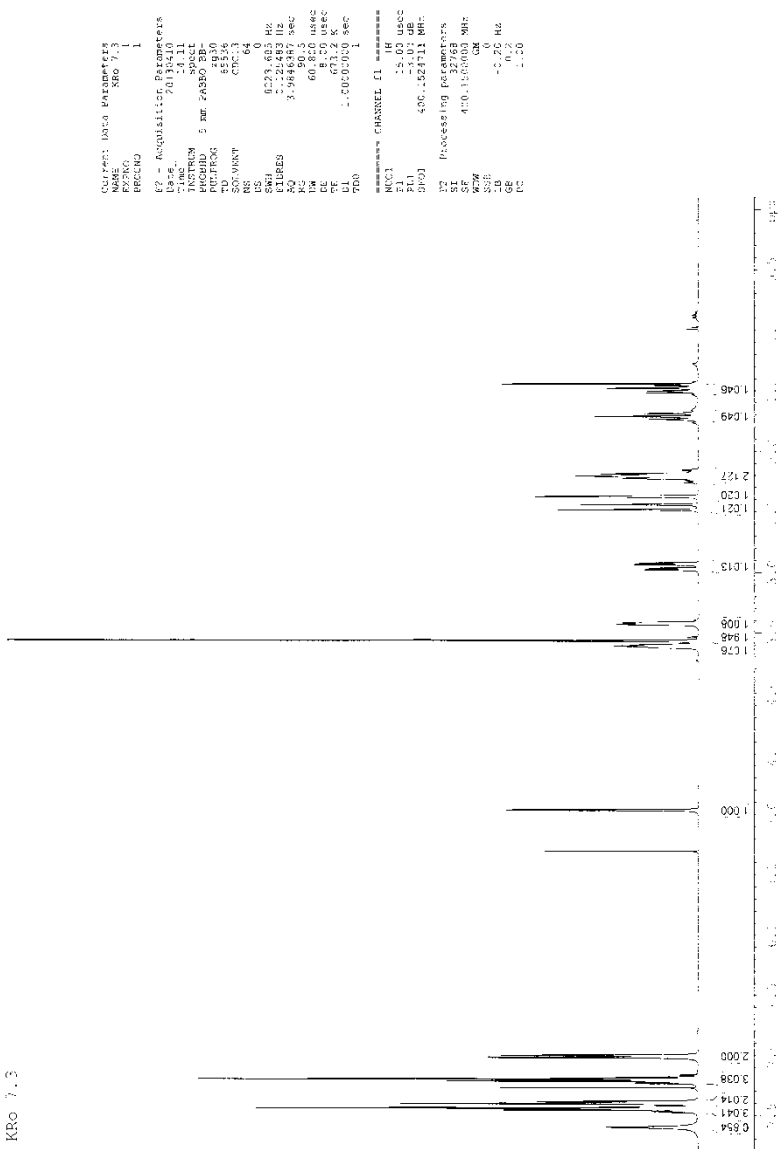
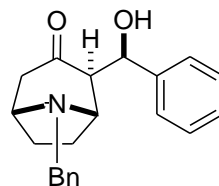
PZ r03 cz. 13C

Current Data Parameters  
 Name: PZ r03  
 EXNO: 2  
 PROCNO: 1  
 Date: 20090416  
 Time: 5.19  
 INSTRUM: spect  
 PROBHD: 5 mm PABBO B3  
 PULPROG: zgpg30  
 SOLVENT: CDCl3  
 NS: 256  
 DS: 4  
 SWH: 24038.461 Hz  
 FIDRES: 0.12463 Hz  
 AQ: 3.984634 sec  
 RG: 327.68  
 DW: 60.800 usec  
 DE: 25.00 usec  
 TE: 297.8 K  
 D1: 1.0000000 sec  
 D11: 0.0000000 sec  
 DELTA: 1.8999998 sec  
 TD0: 1

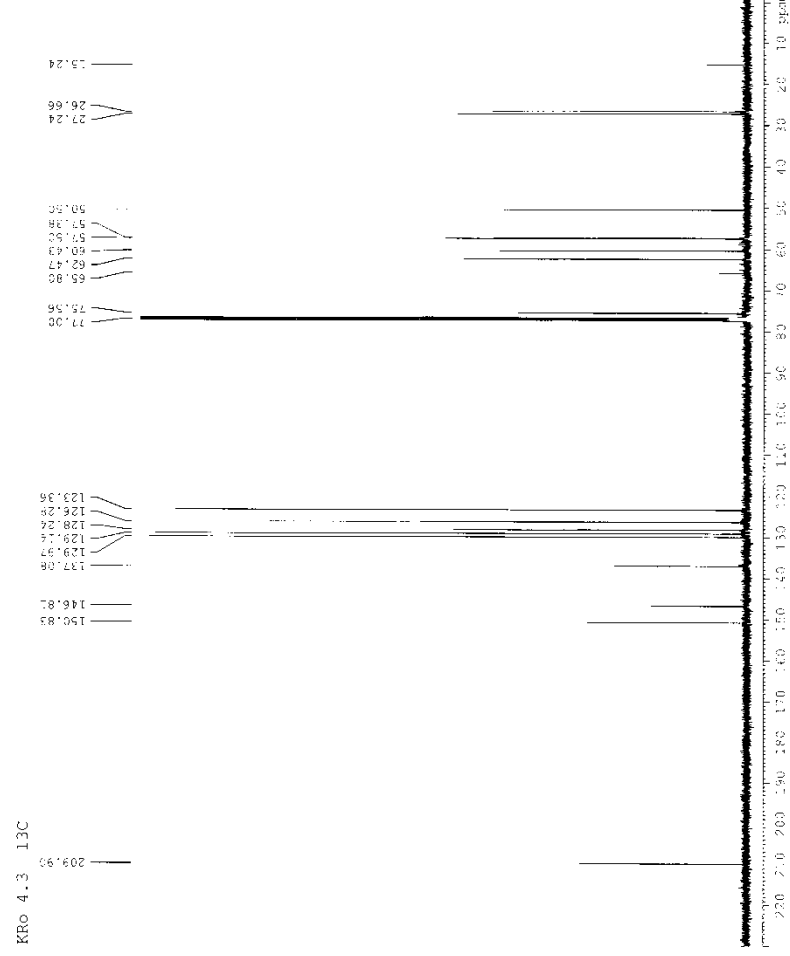
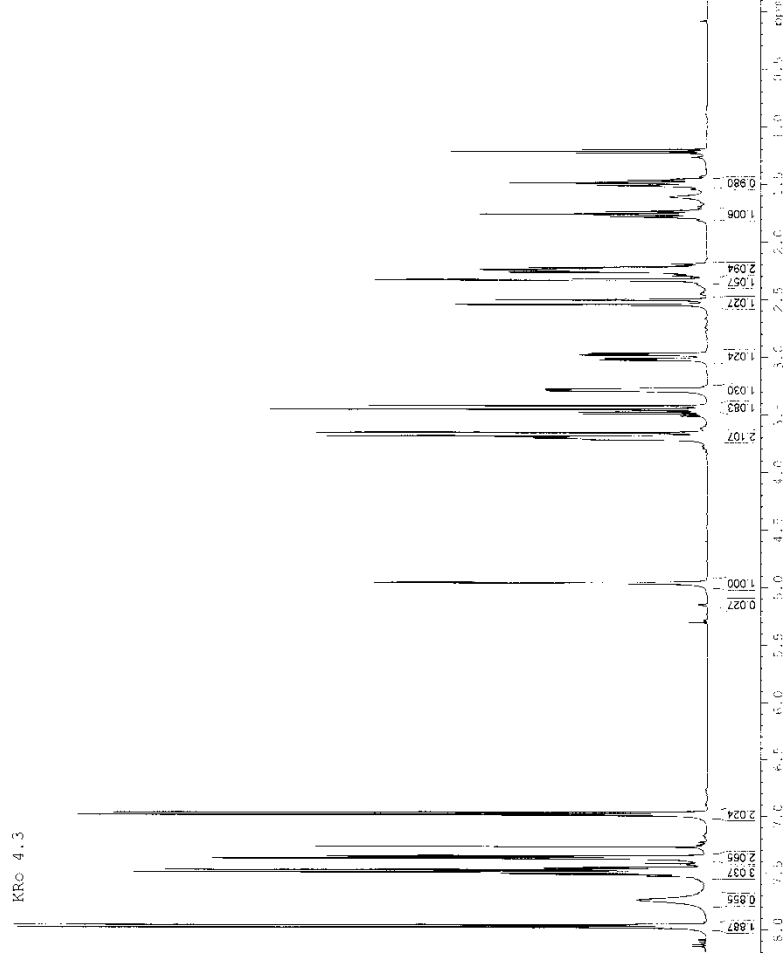
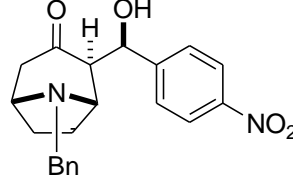
===== CHANNEL f1 =====  
 NUC1: 13C  
 P1: 13.00 usec  
 PL1: -1.00 dB  
 SF01: 100.6278593 MHz  
 F2 - Processing Parameters  
 SI: 327.68  
 SF02: 100.6178033 MHz  
 WDW: EM  
 SS: 0  
 GB: 1.00 Hz  
 PC: 0.10



## KRO 7.3

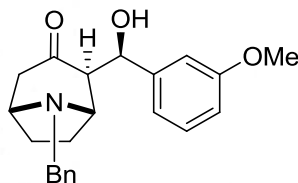


***exo,syn*-8-benzyl-2-(hydroxy(4-nitrophenyl)methyl)-8-aza-bicyclo[3.2.1]octan-3-one (*exo,syn*-14b)**

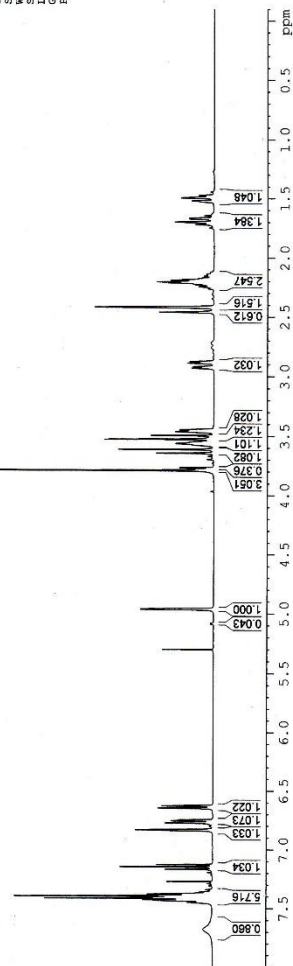


KRo 3a.2

***exo,syn*-8-benzyl-2-(hydroxy(3-methoxyphenyl)methyl-8-azabicyclo[3.2.1]octan-3-one (*exo,syn*-14f)**

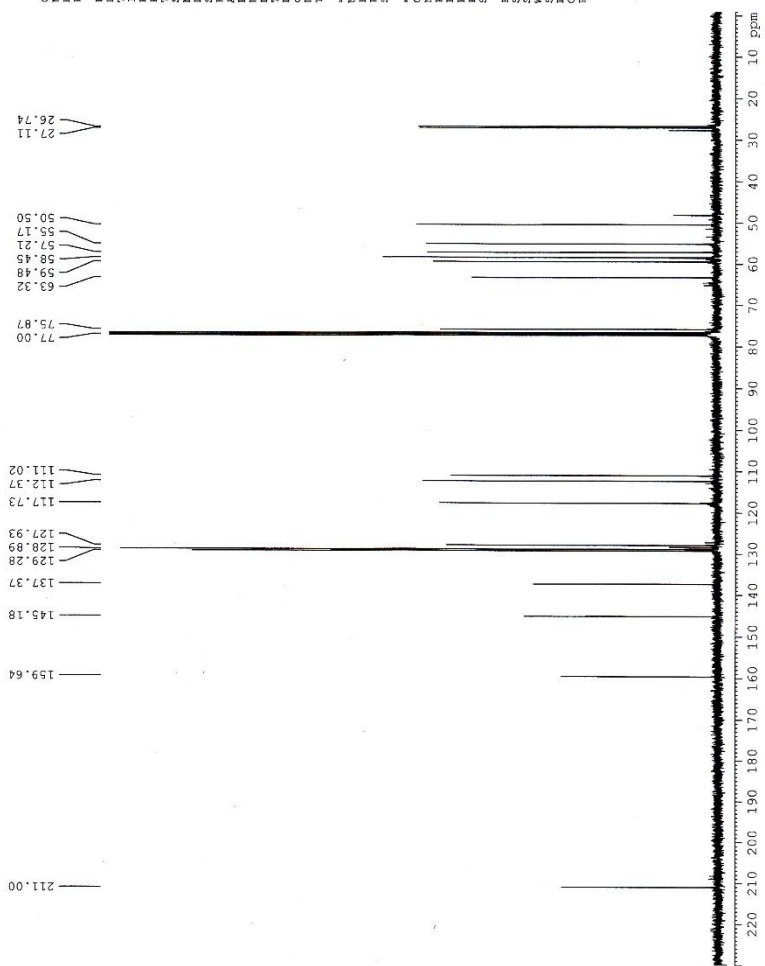


Current Data Parameters  
NAME KRo 3a.2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130207  
Time 15.00  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 0  
AQ 0.025483 Hz  
FIDRES 3.9846387 sec  
RG 32768  
DE 60.800 usec  
TE 300.2 K  
TD0 1.0000000 sec  
===== CHANNEL f1 =====  
NUC1 1H  
P1 22.30 usec  
SFO1 400.1524711 MHz  
F2 - Processing Parameters  
SI 32768  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB -0.20 Hz  
GB 0  
PC 1.00

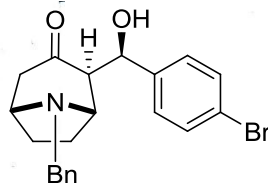


KRo 3a.2 13C

Current Data Parameters  
NAME KRo 3a.2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130207  
Time 15.00  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 32  
DS 4  
AQ 0.025483 Hz  
FIDRES 3.9846387 sec  
RG 32768  
DE 60.800 usec  
TE 300.2 K  
TD0 1.0000000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 12.00 usec  
SFO1 100.6288650 MHz  
F2 - Processing Parameters  
SI 32768  
SF 100.6178012 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



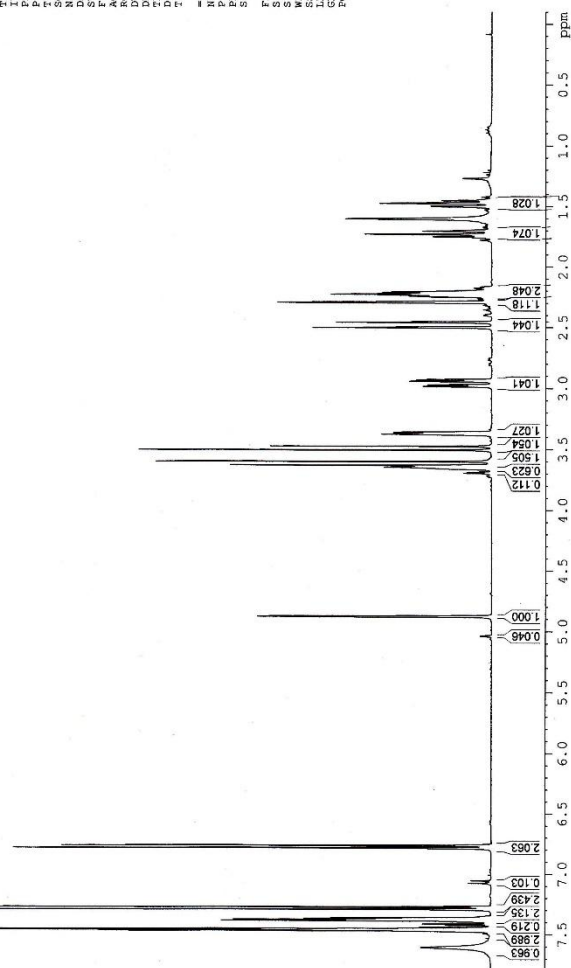
*exo,syn*-8-benzyl-2-((4-bromophenyl)(hydroxy)methyl)-8-azabicyclo[3.2.1]octan-3-one (*exo,syn*-14i)



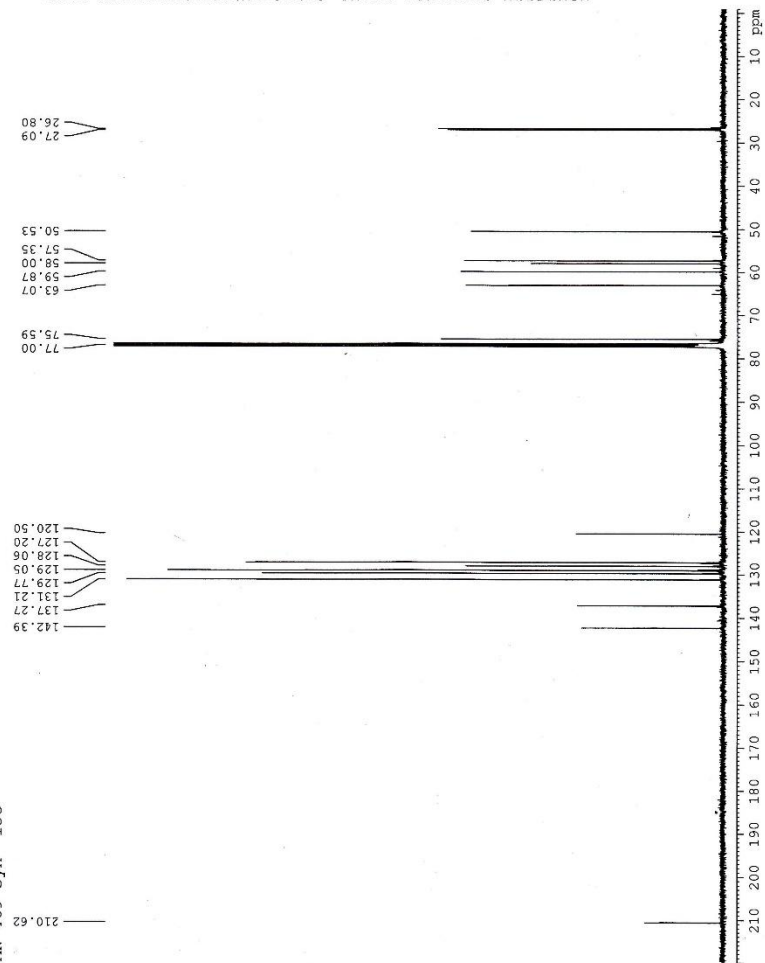
Current Data Parameters  
NAME AN 469syn  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20150721  
Time 17:21  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
FIDRES 0.15483 Hz  
AQ 3.984637 sec  
RG 60.800 usec  
DE 8.00 usec  
TE 300.2 K  
D1 1.0000000 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 1H  
P1 22.30 usec  
PL1 0.00 dB  
SFO1 400.1524711 MHz  
F2 - Processing parameters  
SI 32768  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB -0.50 Hz  
GB 0  
PC 1.00

Current Data Parameters  
NAME AN 469syn  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20150721  
Time 17:21  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
FIDRES 0.15483 Hz  
AQ 3.984637 sec  
RG 60.800 usec  
DE 8.00 usec  
TE 300.2 K  
D1 1.0000000 sec  
D11 0.0000000 sec  
DELTA 1.8999998 sec  
TD0 1  
===== CHANNEL f1 =====  
NUC1 13C  
P1 12.30 usec  
PL1 0.00 dB  
SFO1 100.6288660 MHz  
F2 - Processing parameters  
SI 32768  
SF 100.6177582 MHz  
WDW EM  
SSB 0  
LB -0.50 Hz  
GB 0  
PC 1.40

AN 469 syn



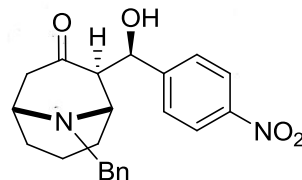
AN 469 syn 13C



KRo 10a.3

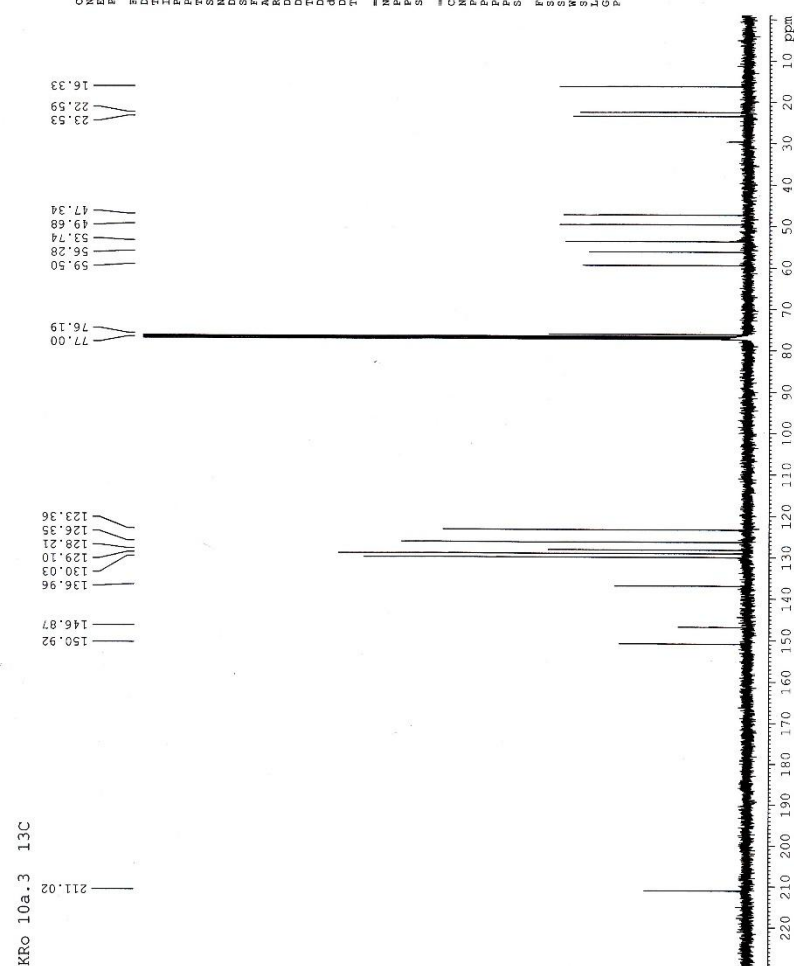
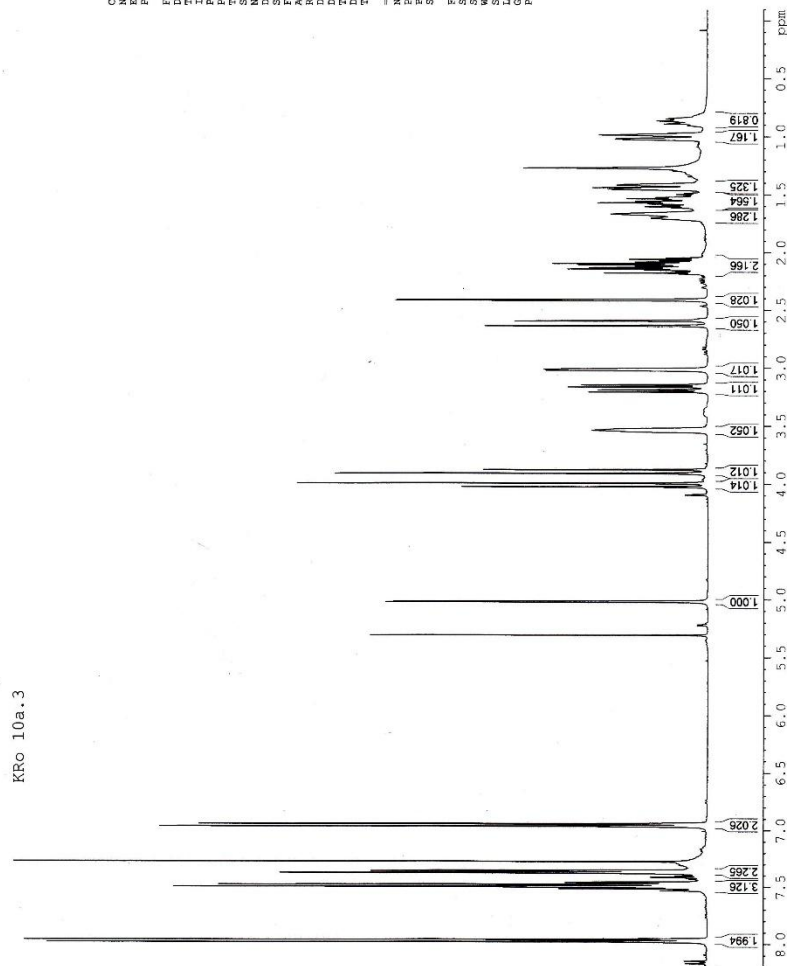
Current Data Parameters  
NAME KRo 10a.3  
EXPRO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130215  
Time 11:10  
INSTRUM spect  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 0  
SWH 823.0 Hz  
FIDRES 0.22485 Hz  
AQ 3.9846387 sec  
RG 60.87 usec  
DE 6.00 usec  
TE 673.2 K  
TDO 1.0000000 sec

===== CHANNEL f1 1H =====  
NUC1 1H  
P1 11.15 usec  
PL 0.00 dB  
SFO1 400.1524111 MHz  
F2 - Processing Parameters  
SI 32768  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB -0.20 Hz  
GB 0.2  
PC 1.00

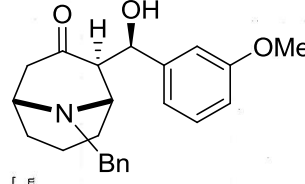


Current Data Parameters  
NAME KRo 10a.3  
EXPRO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130215  
Time 11:10  
INSTRUM spect  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 128  
DS 0  
SWH 823.0 Hz  
FIDRES 0.22485 Hz  
AQ 3.9846387 sec  
RG 60.87 usec  
DE 6.00 usec  
TE 673.2 K  
TDO 1.0000000 sec

===== CHANNEL f1 13C =====  
NUC1 13C  
P1 12.25 usec  
PL 0.00 dB  
SFO1 100.6284660 MHz  
F2 - Processing Parameters  
SI 32768  
SF 100.6177988 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0.2  
PC 0.20

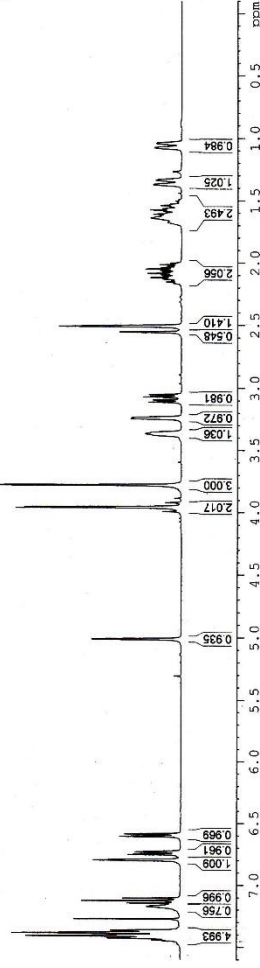


*exo,syn*-9-benzyl-2-(hydroxy(3-methoxyphenyl)methyl)-9-azabicyclo[3.3.1]nonan-3-one (*exo,syn*-15f)



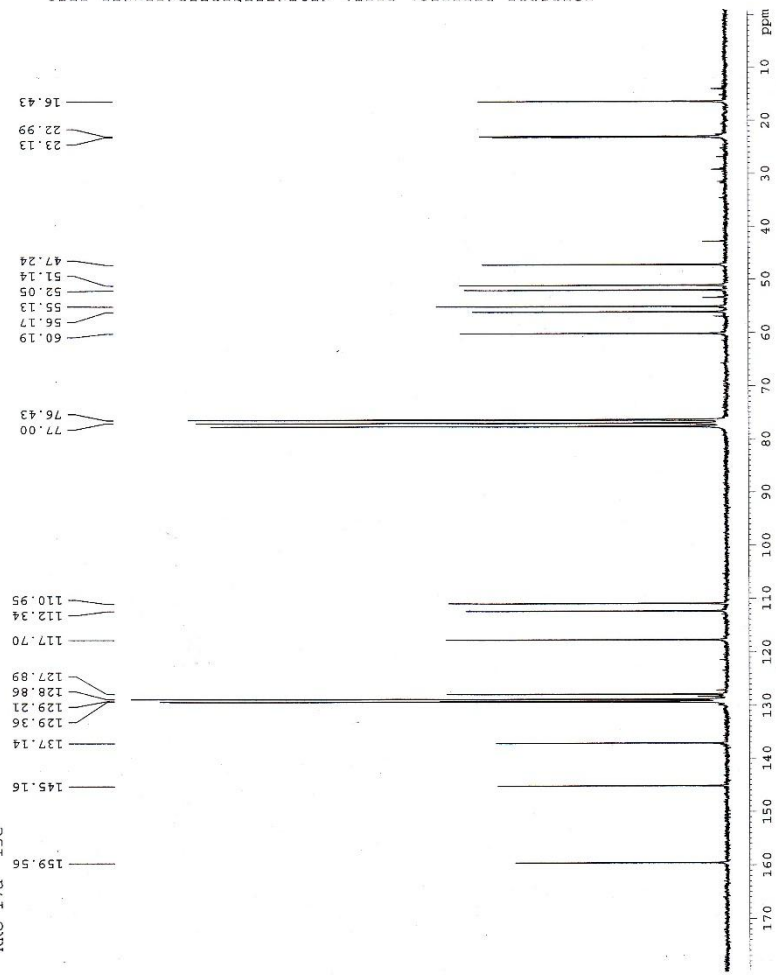
KRo 17a.3

Current Data Parameters  
NAME XRO 17a  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 2010109  
Time 21:11  
INSTRUM spect  
PROBHD 5 mm PABBO H-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 120  
DS 10  
SWH 8223.685 Hz  
AQ 0.0246187 sec  
RG 3.3846397 sec  
RG 181  
DW 60.000 usec  
DE 8.000 usec  
TE 300.5 K  
D1 1.00000000 sec  
D10 1.00000000 sec  
===== CHANNEL f1 =====  
NUC1 13C  
P1 22.30 usec  
PL1 0.00 dB  
PL1 400.157411 MHz  
SFO1 400.157411 MHz  
F2 - Processing parameters  
WDW EM  
SSB 0  
LB 0.2 Hz  
GB 0.2  
PC 1.00



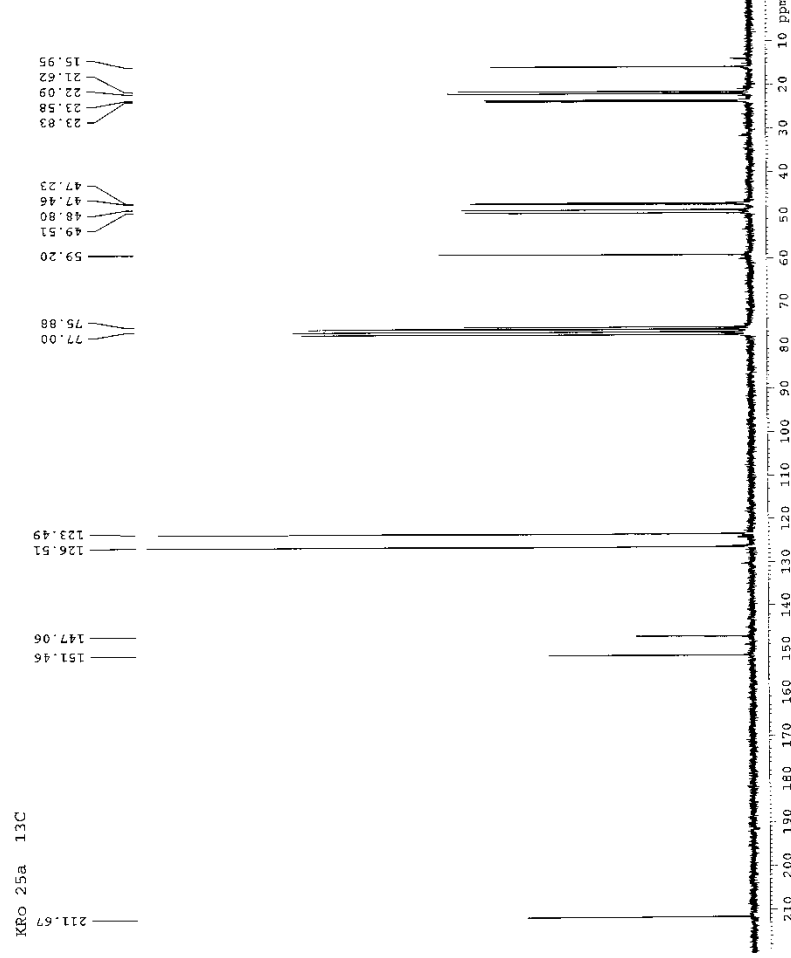
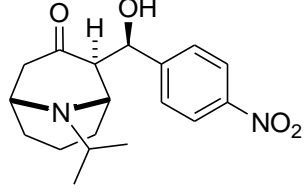
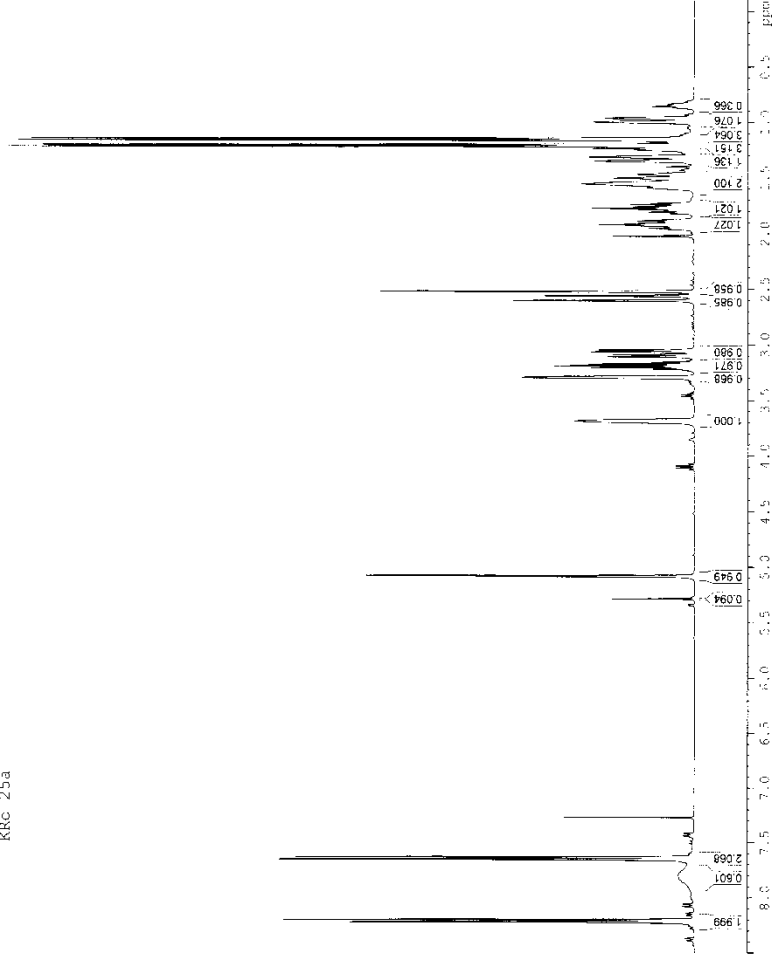
KRo 17a 13C

Current Data Parameters  
NAME XRO 17a  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 2010109  
Time 21:11  
INSTRUM spect  
PROBHD 5 mm Dual 13C/  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 6400  
DS 13513.514 Hz  
SWH 8223.685 Hz  
AQ 0.0246187 sec  
RG 3.3846397 sec  
RG 181  
DW 60.000 usec  
DE 8.000 usec  
TE 300.5 K  
D1 1.00000000 sec  
D10 1.00000000 sec  
D10 1.00000000 sec  
===== CHANNEL f1 13C =====  
NUC1 13C  
P1 22.30 usec  
PL1 0.00 dB  
PL1 400.157411 MHz  
SFO1 400.157411 MHz  
===== CHANNEL f2 1H =====  
NUC2 1H  
P2 10.00 usec  
PL2 0.00 dB  
PL2 400.157411 MHz  
SFO2 400.157411 MHz  
F2 - Processing parameters  
WDW EM  
SSB 0  
LB 0.2 Hz  
GB 0.2  
PC 1.00

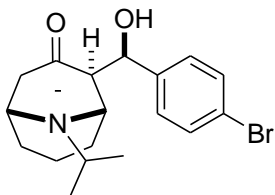




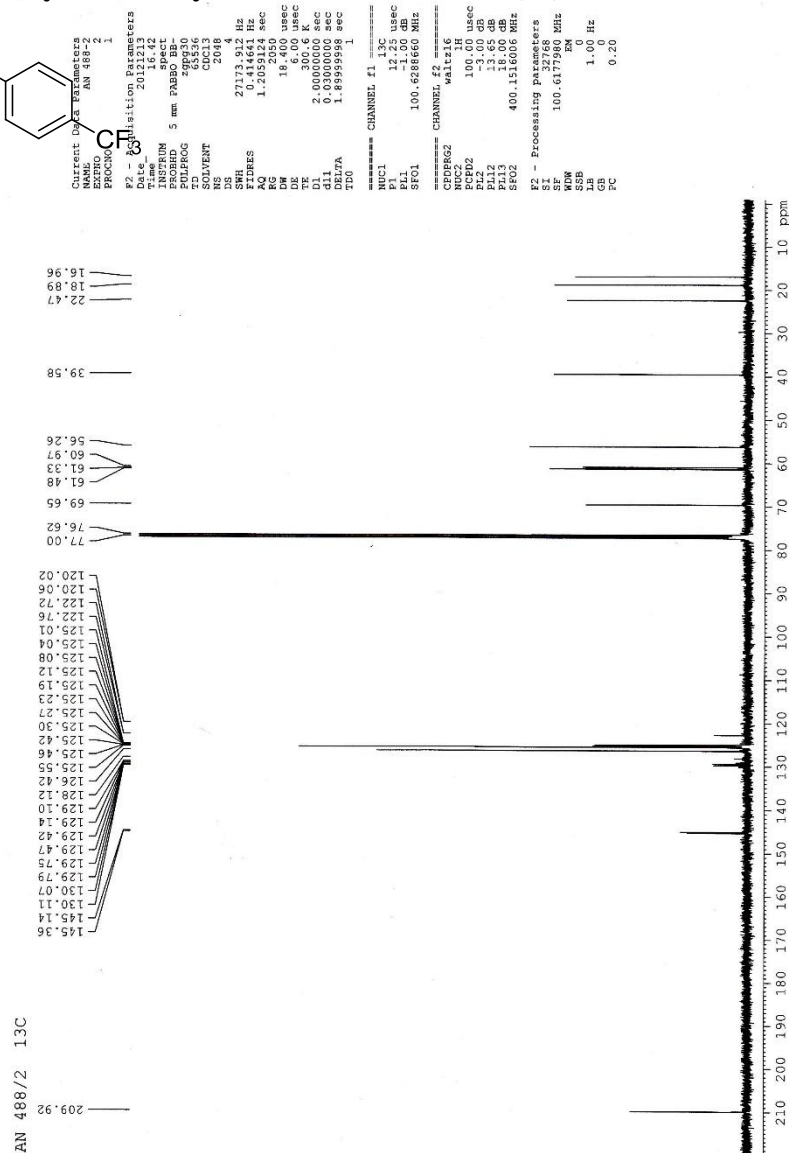
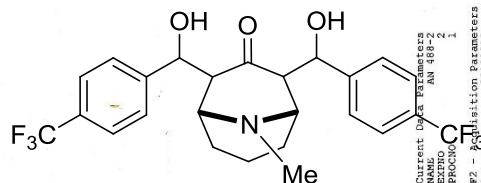
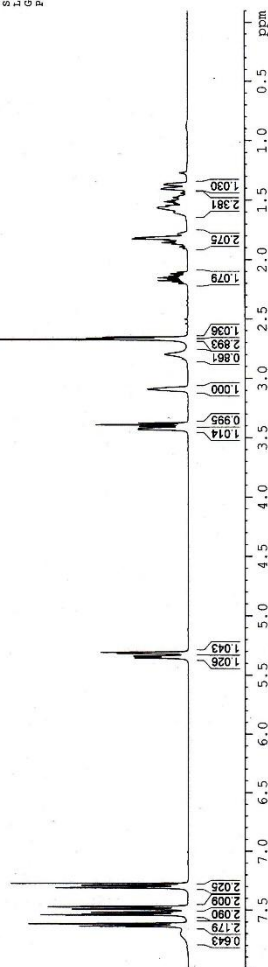
## KRC 25a



## KRO 26



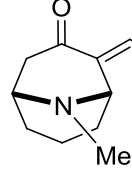
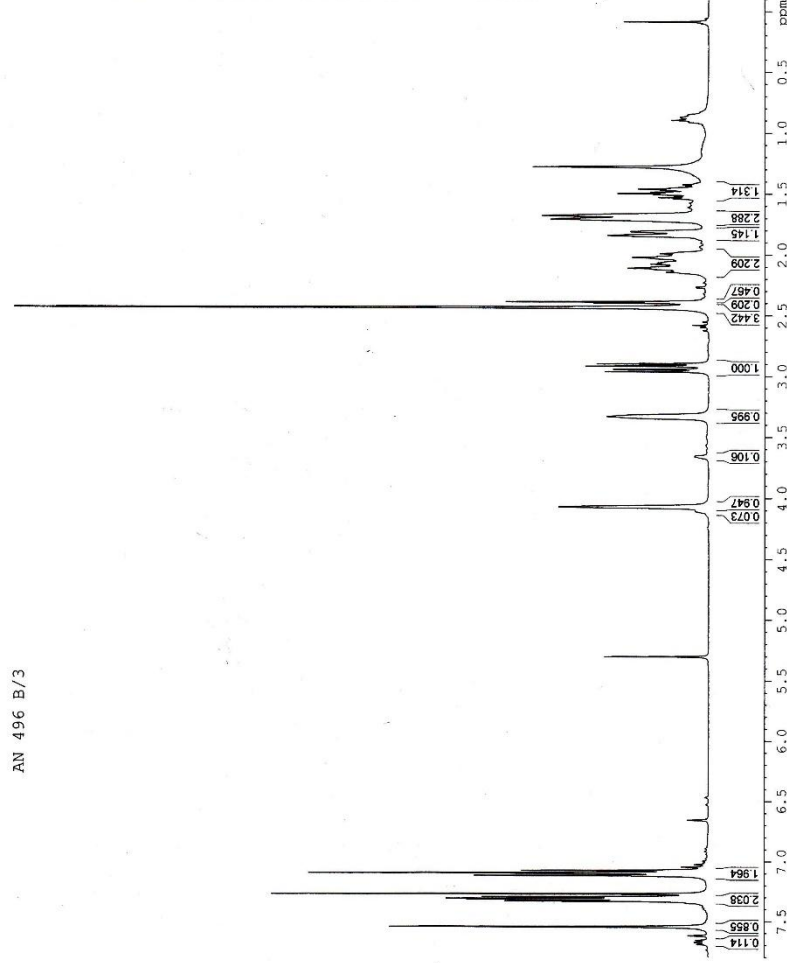
## AN 488/2



AN 496 B/3

Current Data Parameters  
NAME AN 496-B3  
EXPNO 1  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130221  
Time\_ 12.11  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 120  
SWH 8223.685 Hz  
FIDRES 0.41461 Hz  
AQ 3.9446387 sec  
RG 60.128 usec  
DE 8.000 usec  
TE 313.0 K  
D1 1.00000000 sec  
D2 1.00000000 sec  
TDO 1

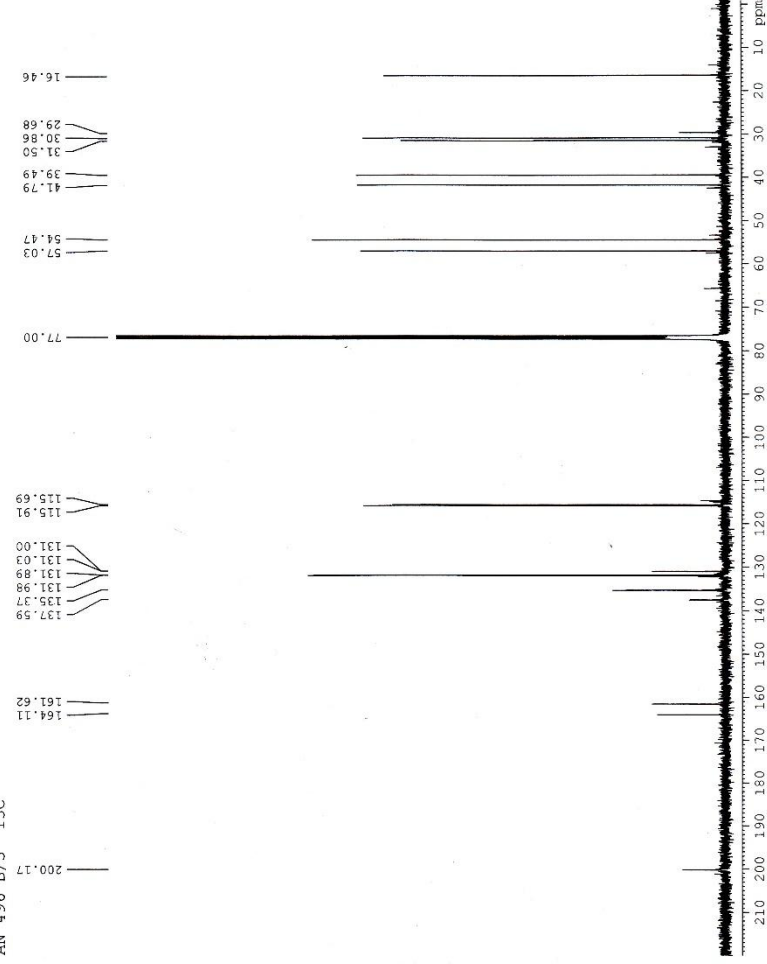
===== CHANNEL f1 =====  
NUC1 13C  
P1 22.30 usec  
PL1 -1.00 dB  
SFO1 400.1524711 MHz  
F2 - Processing parameters  
SI 32768  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB 0.2  
GB 0.2  
PC 1.00



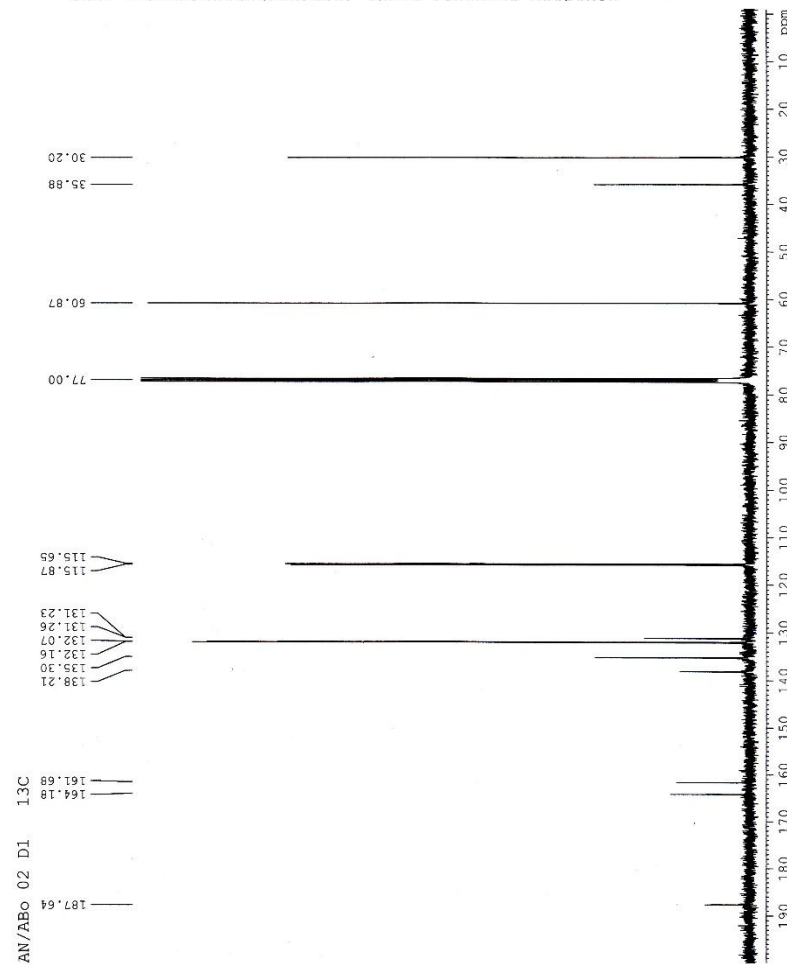
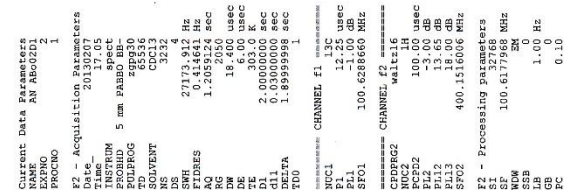
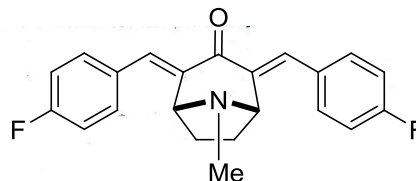
Current Data Parameters  
NAME AN 496-B3  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130227  
Time\_ 12.11  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 10240  
SWH 27173.912 Hz  
FIDRES 0.41461 Hz  
AQ 1.255228 sec  
RG 60.128 usec  
DE 8.000 usec  
TE 313.0 K  
D1 2.00000000 sec  
D2 1.00000000 sec  
DELTA 1.8995998 sec  
TDO 1

===== CHANNEL f1 =====  
NUC1 13C  
P1 22.30 usec  
PL1 -1.00 dB  
SFO1 100.6286660 MHz  
===== CHANNEL f2 =====  
NAME AN 496-B3  
EXPNO 2  
PROCNO 1  
F2 - Acquisition Parameters  
Date\_ 20130227  
Time\_ 12.11  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
DS 10240  
SWH 27173.912 Hz  
FIDRES 0.41461 Hz  
AQ 1.255228 sec  
RG 60.128 usec  
DE 8.000 usec  
TE 313.0 K  
D1 2.00000000 sec  
D2 1.00000000 sec  
DELTA 1.8995998 sec  
TDO 1

AN 496 B/3 13C



## AN/ABO 02 D1



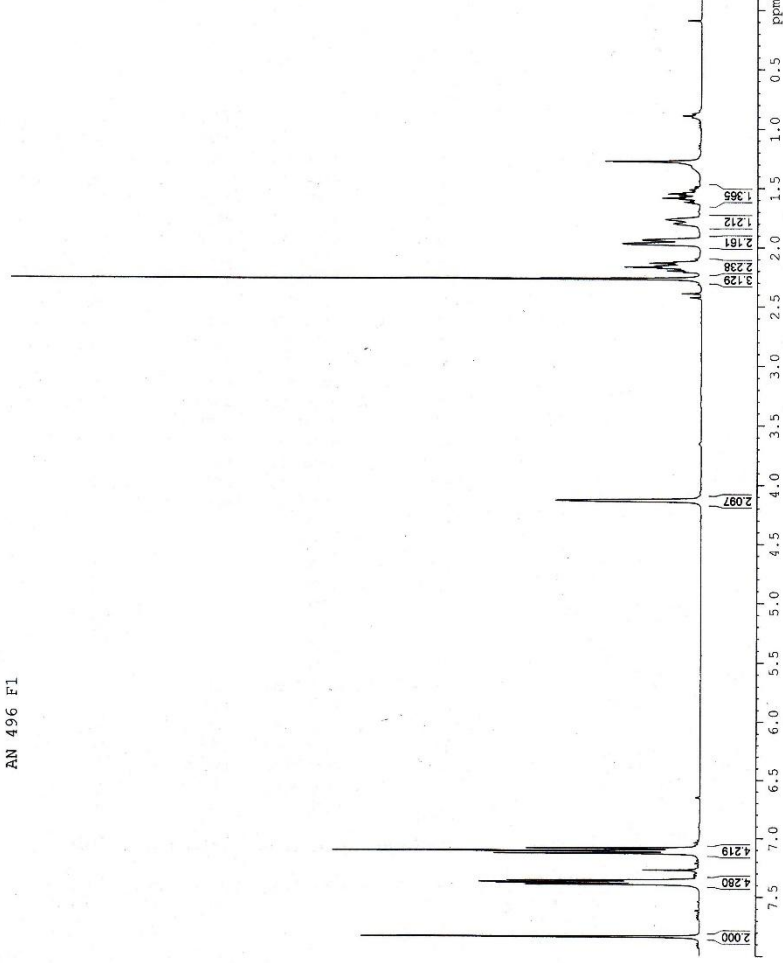
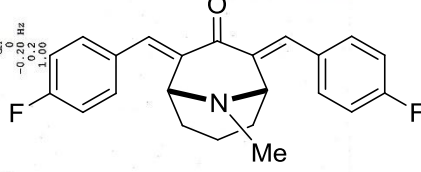
AN 496 F1

Current Data Parameters  
NAME AN 456F1  
EXPNO 1  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20130222  
Time 17:45  
INSTRUM spect  
PROBHD 5 mm PABBO BB  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 64  
DS 0  
SWH 8753.455 Hz  
FIDRES 0.121485 Hz  
AQ 3.9846387 sec  
RG 60.800 usec  
DE 8.00 usec  
TE 313.2 K  
DELTA 1.0000000 sec  
TD0 1.0000000 sec

===== CHANNEL f1 =====  
NUC1 1H  
P1 11.15 usec  
PL1 0.00 dB  
SFO1 400.152711 MHz

F2 - Processing Parameters  
SI 32768  
SF 400.1500000 MHz  
WDW EM  
SSB 0  
LB 0.2 Hz  
GB 0.2 Hz  
PC 1.00



AN 496 F1 13C

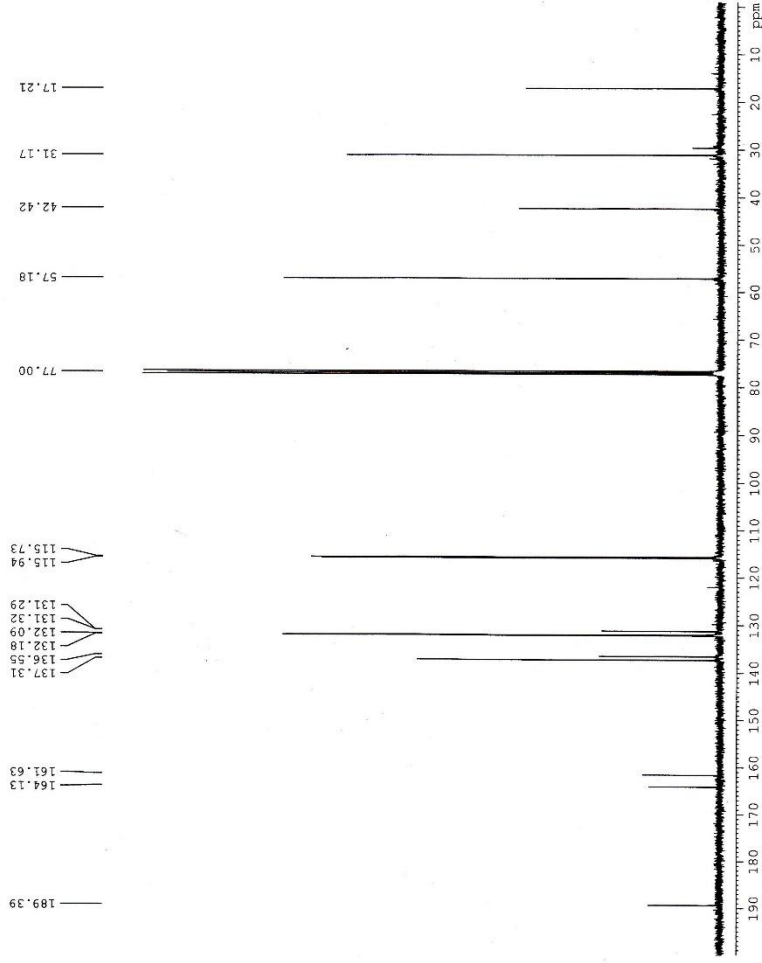
Current Data Parameters  
NAME AN 456F2  
EXPNO 2  
PROCNO 1

F2 - Acquisition Parameters  
Date\_ 20130222  
Time 17:45  
INSTRUM spect  
PROBHD 5 mm PABBO BB  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 64  
DS 4  
SWH 27173.912 Hz  
FIDRES 0.121485 Hz  
AQ 1.2055124 sec  
RG 2050  
DE 18.00 usec  
TE 673.2 K  
DELTA 2.0000000 sec  
TD0 1.0000000 sec

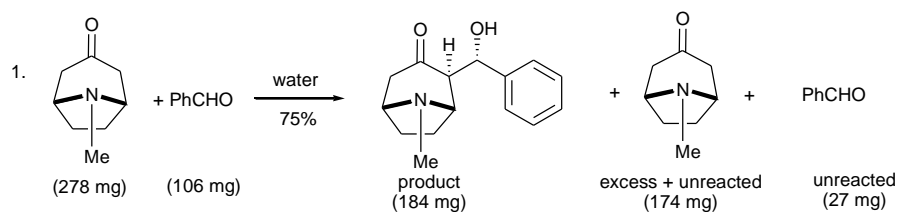
===== CHANNEL f1 =====  
NUC1 13C  
P1 12.15 usec  
PL1 -1.00 dB  
SFO1 100.6288660 MHz

===== CHANNEL f2 =====  
NAME AN 456F2  
F2 - Acquisition Parameters  
Date\_ 20130222  
Time 17:45  
INSTRUM spect  
PROBHD 5 mm PABBO BB  
PULPROG zgpg30  
TD 65536  
SOLVENT CDCl3  
NS 64  
DS 4  
SWH 27173.912 Hz  
FIDRES 0.121485 Hz  
AQ 1.2055124 sec  
RG 2050  
DE 18.00 usec  
TE 673.2 K  
DELTA 2.0000000 sec  
TD0 1.0000000 sec

===== CHANNEL f2 =====  
NAME AN 456F2  
F2 - Processing Parameters  
SI 32768  
SF 100.6178008 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0.2 Hz  
PC 1.40



## E-factor



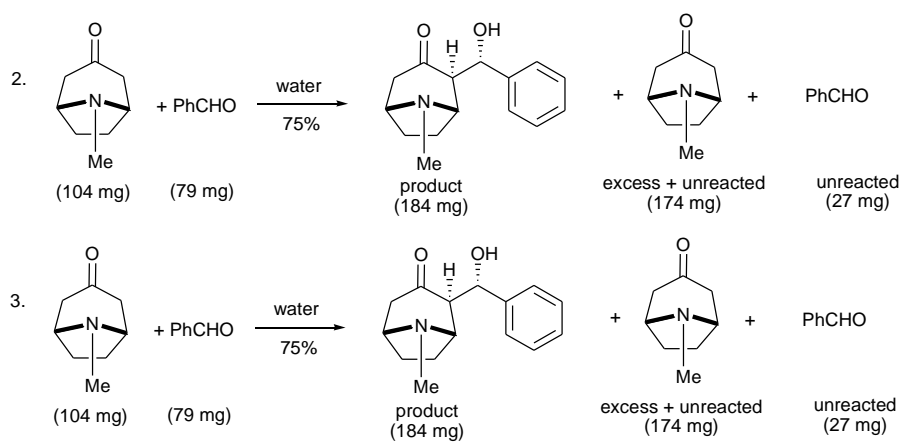
### FOR ONE RUN OF THE REACTION

Total amount of reactants: 278 mg + 106 mg = 384 mg

Amount of final product:  $1 \bullet 184 = 184$  mg, assuming yield = 75% for one run

Amount of waste: (384 mg – 184 mg) = 200 mg

**E-Factor** = Amount of waste / Amount of product = 200 mg / 184 mg = **1.09**



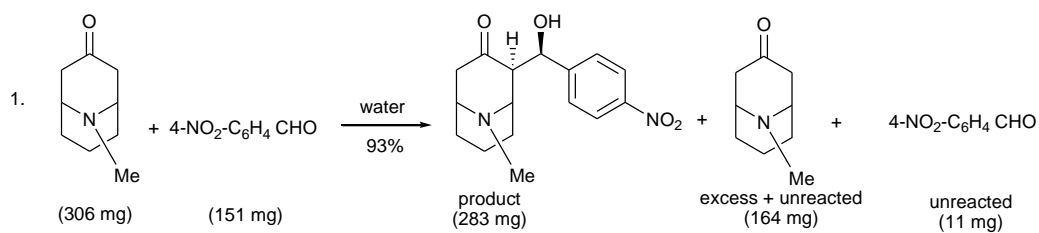
### FOR THREE RUNS OF THE REACTION

Total amount of reactants: 278 mg + 106 mg + 2 • 104 mg + 2 • 79 mg = 750 mg

Amount of final product:  $3 \bullet 184 = 552$  mg, assuming yield = 75% in each cycle

Amount of waste: (750 mg – 552 mg) = 198 mg

**E-Factor** = Amount of waste / Amount of product = 198 mg / 552 mg = **0.36**



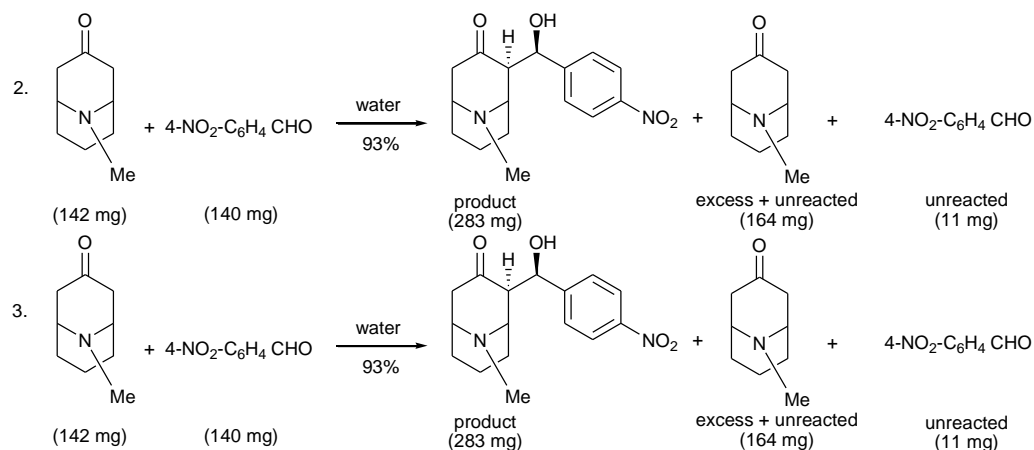
#### FOR ONE RUN OF THE REACTION

Total amount of reactants: 306 mg + 151 mg = 457 mg

Amount of final product:  $1 \cdot 283 = 283$  mg, assuming yield = 93% for one run

Amount of waste: (457 mg – 283 mg) = 174 mg

**E-Factor** = Amount of waste / Amount of product = 174 mg / 283 mg = **0.61**



#### FOR THREE RUNS OF THE REACTION

Total amount of reactants: 306 mg + 151 mg + 2 • 142 mg + 2 • 140 mg = 1021 mg

Amount of final product:  $3 \cdot 283 = 849$  mg, assuming yield = 93% in each cycle

Amount of waste: (1021 mg – 849 mg) = 172 mg

**E-Factor** = Amount of waste / Amount of product = 172 mg / 849 mg = **0.20**