## Amphiphilic Allylation of Arylidene–1,3–oxazol–5(4*H*)–one Using Bis–π–allylpalladium Complexes: An Approach to Synthesis of Cyclohexyl and Cyclohexenyl α–Amino Acids

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Page S43: <sup>1</sup>H and <sup>13</sup>C NMR spectrum of compound **17** 

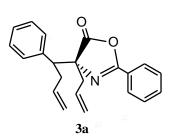
Page S45: <sup>1</sup>H and <sup>13</sup>C NMR spectrum of compound **18** 

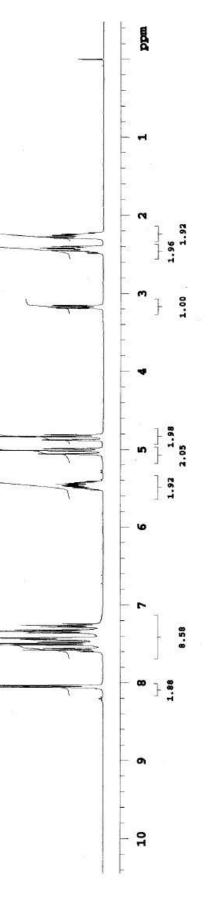
Page S47: <sup>1</sup>H and <sup>13</sup>C NMR spectrum of compound **19** 

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# Allyl-2-phenyl-4-(1-phenyl-but-3-enyl)-4H-oxazol-5-one (3a)

<sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





H1, 399.7630265 MHz

idth 6410.3 Hz

repetitions

Pulse Sequence: s2pul

Tile: XD

odol3 VTIME 1 Varian40

008-9400 perator: Solvent:

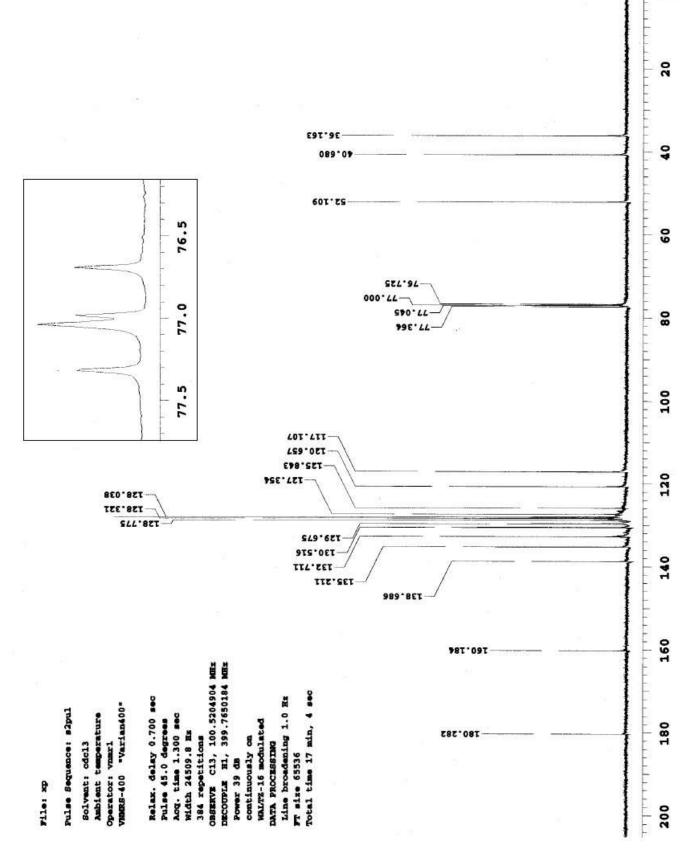
Relax. delay 1.500 time 3.500 se 45.0 degre

Pulles 50

Total time 0 min, 40 sec ine broadening 0.2 Hz

T size 65536

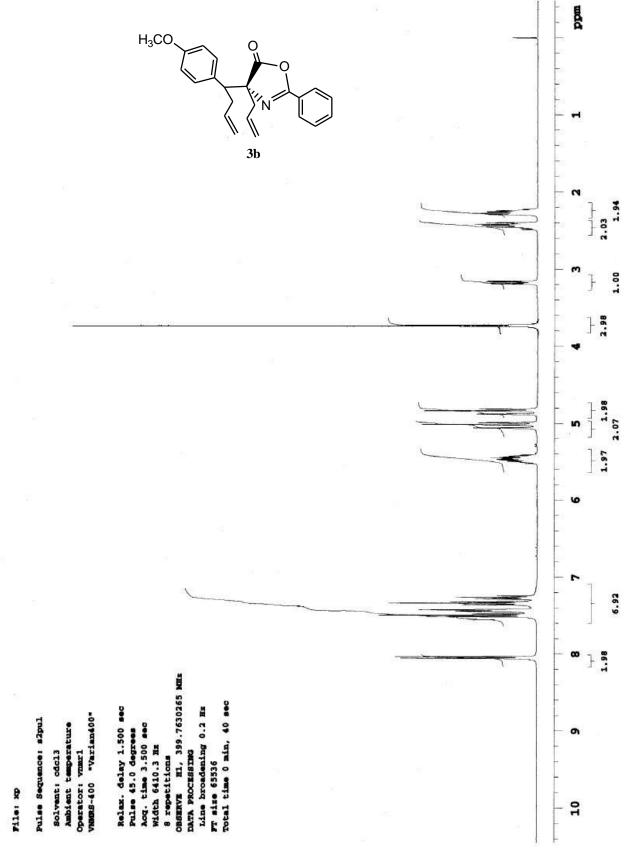
# <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

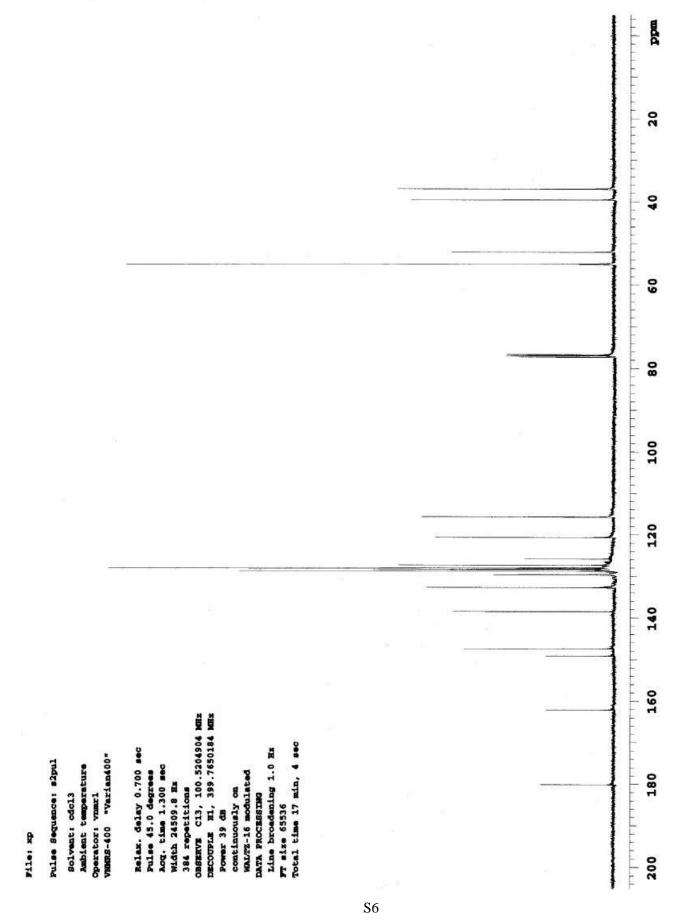


mdd

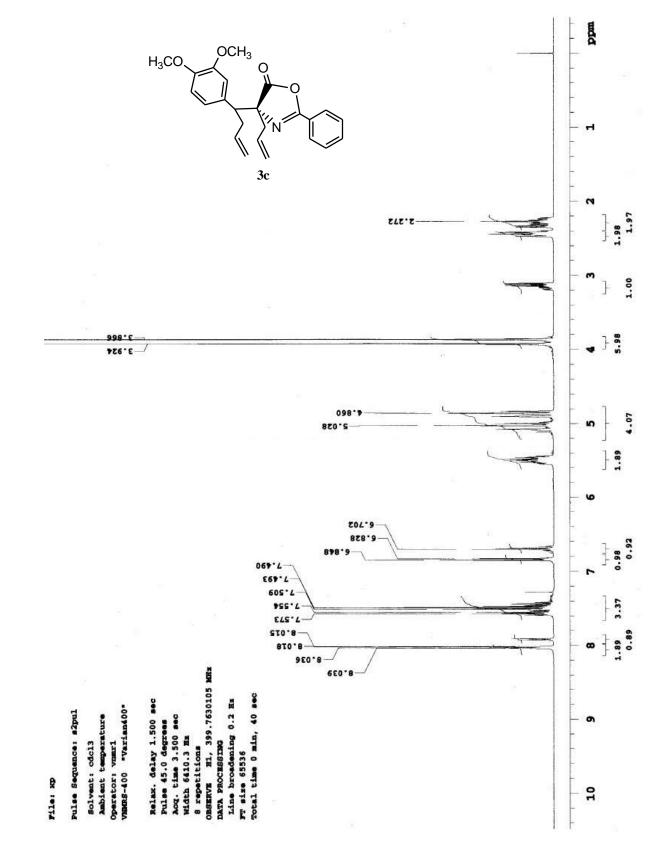
**S**4

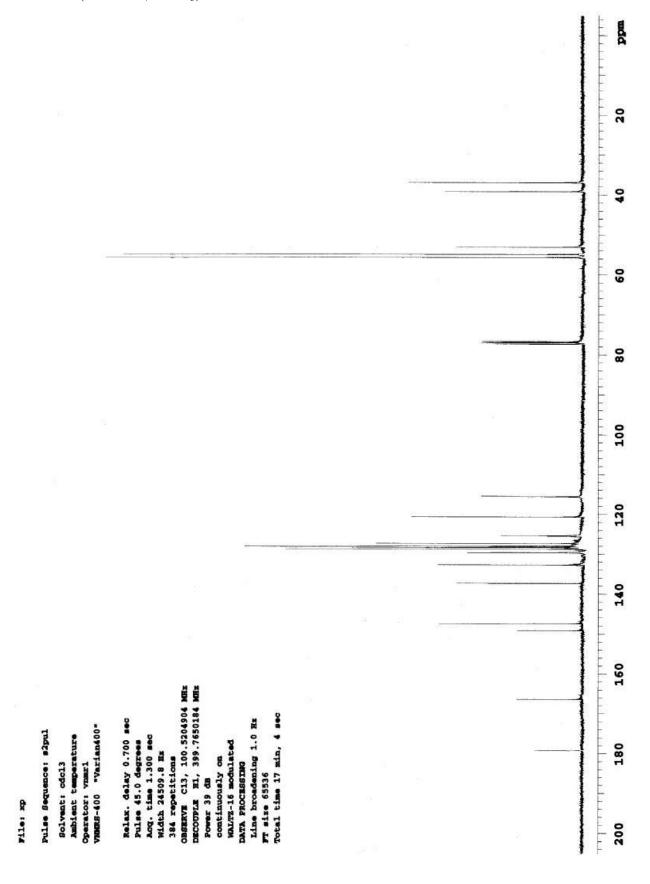
#### 4-Allyl-4-[1-(4-methoxy-phenyl)-but-3-enyl]-2-phenyl-4H-oxazol-5-one (3b)



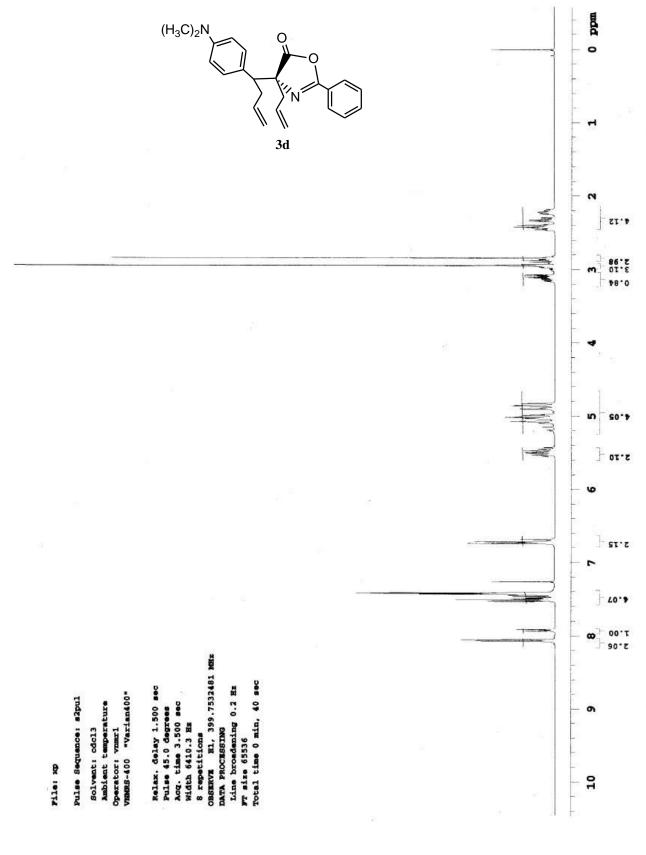


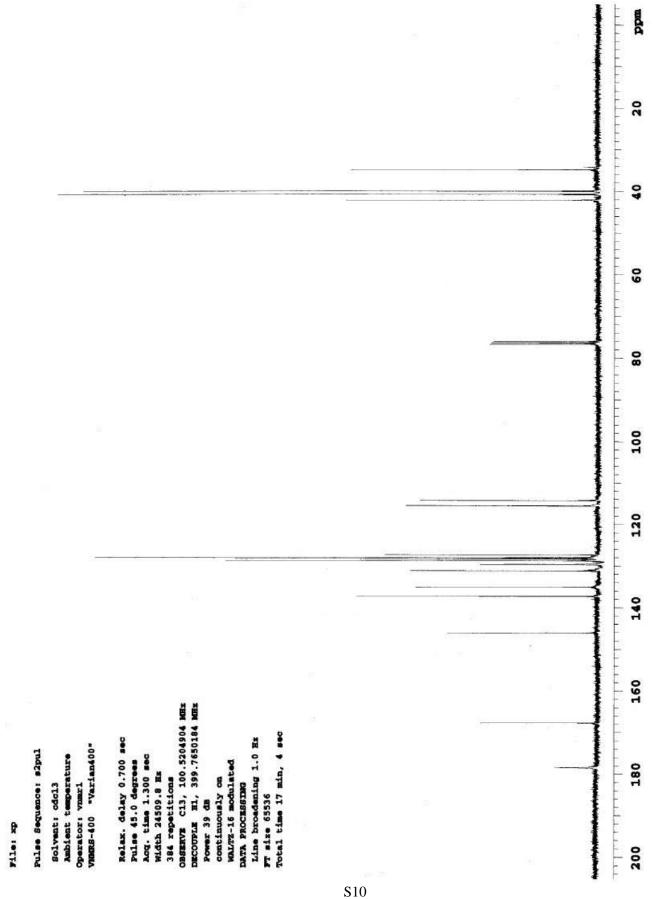
#### 4-Allyl-4-[1-(3,4-dimethoxy-phenyl)-but-3-enyl]-2-phenyl-4H-oxazol-5-one (3c)



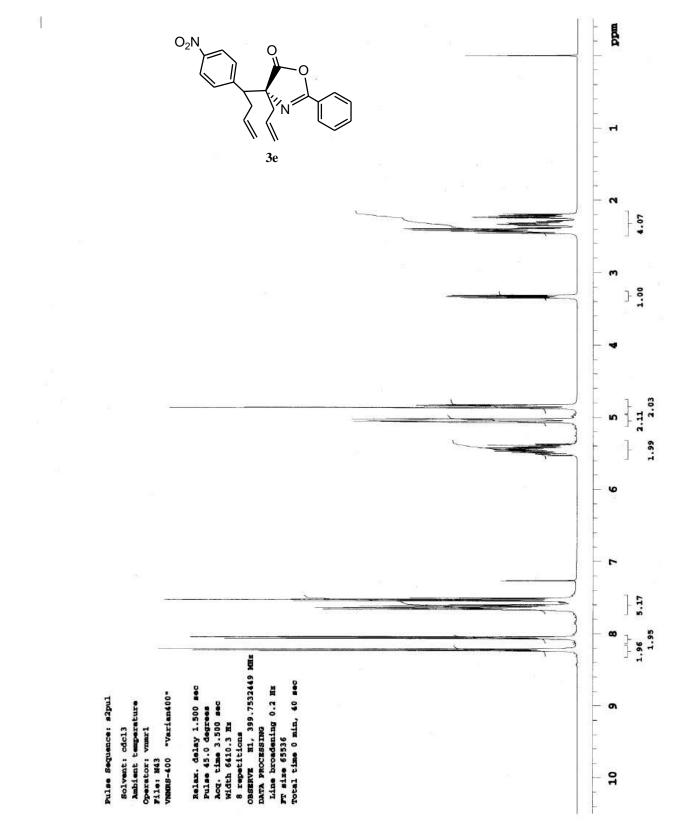


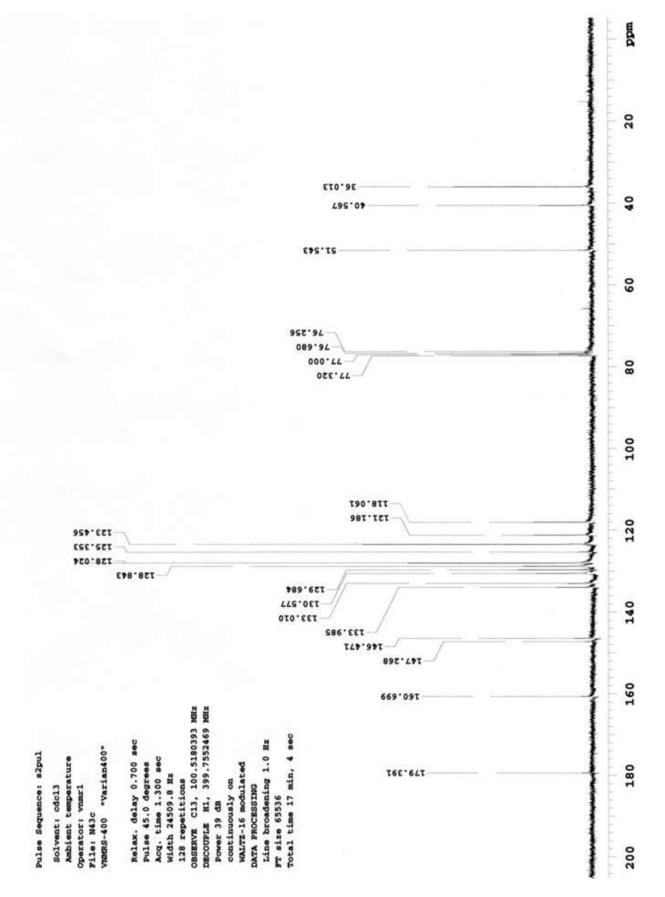
### 4-Allyl-4-[1-(4-dimethylamino-phenyl)-but-3-enyl]-2-phenyl-4H-oxazol-5-one (3d)





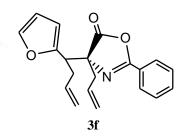
#### 4-Allyl-4-[1-(4-nitro-phenyl)-but-3-enyl]-2-phenyl-4H-oxazol-5-one (3e)





#### 4-Allyl-4-(1-(furan-2-yl)but-3-enyl)-2-phenyloxazol-5(4H)-one (3f)

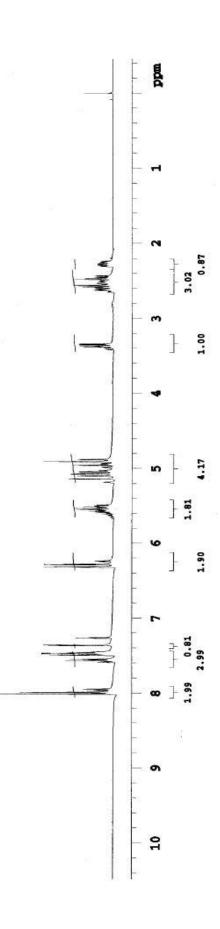
### <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





Pulse Sequence: s2pul

Solvent: odc13 Ambient temperature perator: vnmr1 NMRS-400 "Varian400 Relax. delay 1.500 sec Pulse 45.0 degrees Acg. time 3.500 sec Width 6410.3 Hz 8 repetitions DBSERVE H1, 399.7630181 MHz DATA FROCESSING DATA FROCESSING Line broedening 0.2 Hz FT size 6536 Fotal time 0 min, 40 sec



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



Tile: XD

S14

ndd

20

\$

80

100

120

140

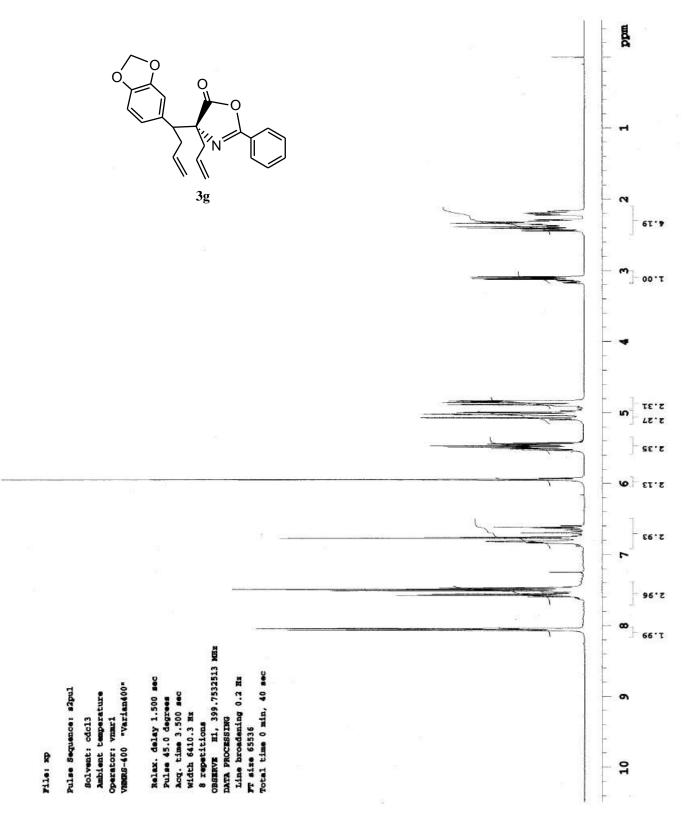
160

180

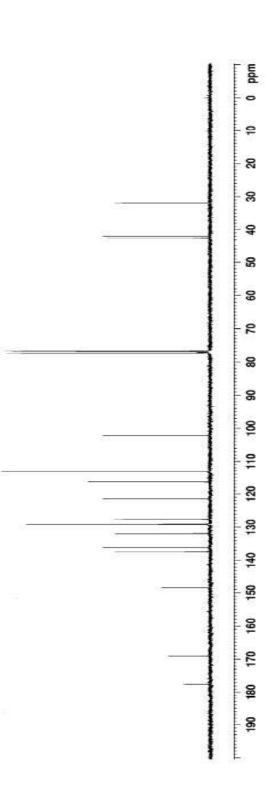
200

- 3

#### 4-Allyl-4-(1-benzo[1,3]dioxol-5-yl-but-3-enyl)-2-phenyl-4H-oxazol-5-one (3g)

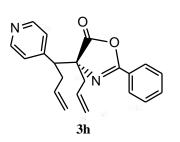






#### 4-Allyl-2-phenyl-4-(1-(pyridin-4-yl)but-3-enyl)oxazol-5(4H)-one (3h)

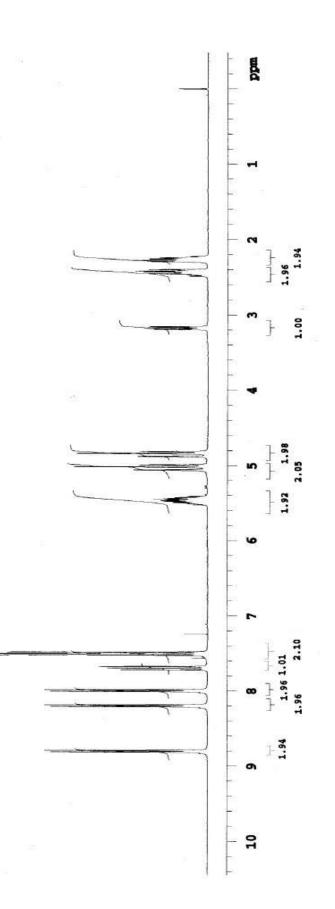
### <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



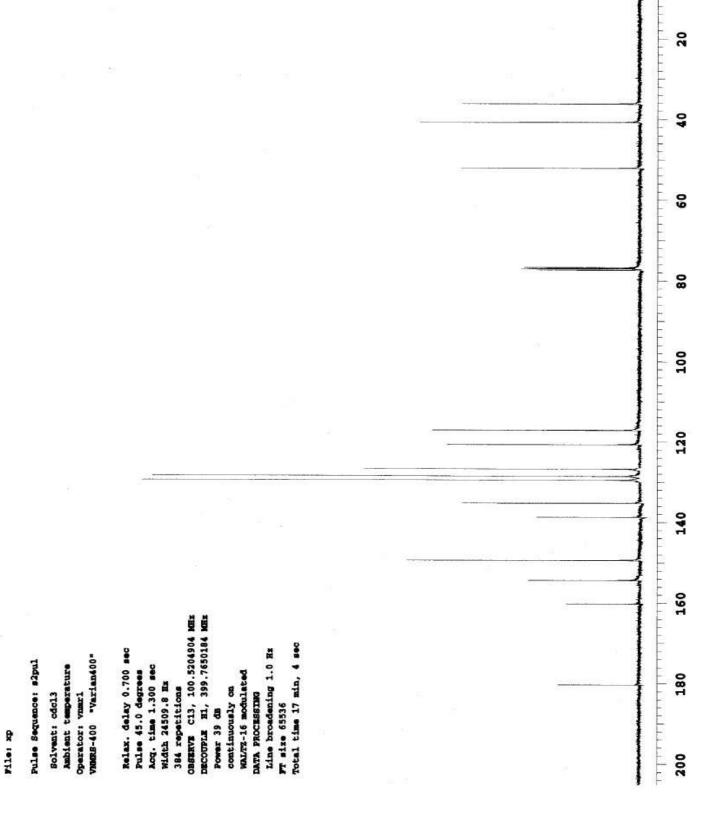


Pulse Sequence: s2pul

Solvent: odc13 Ambient temperature perator: vnmr1 WMR5-400 "Varian400 Relax. delay 1.500 sed Pulse 45.0 degrees Acq. time 3.500 sec Width 6410.3 Rm 8 repetitions 086KNVE H1, 399.7630265 MHz 087A PROCESSING Line broadening 0.2 Hz Line broadening 0.2 Hz Drai time 0 min, 40 sec



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

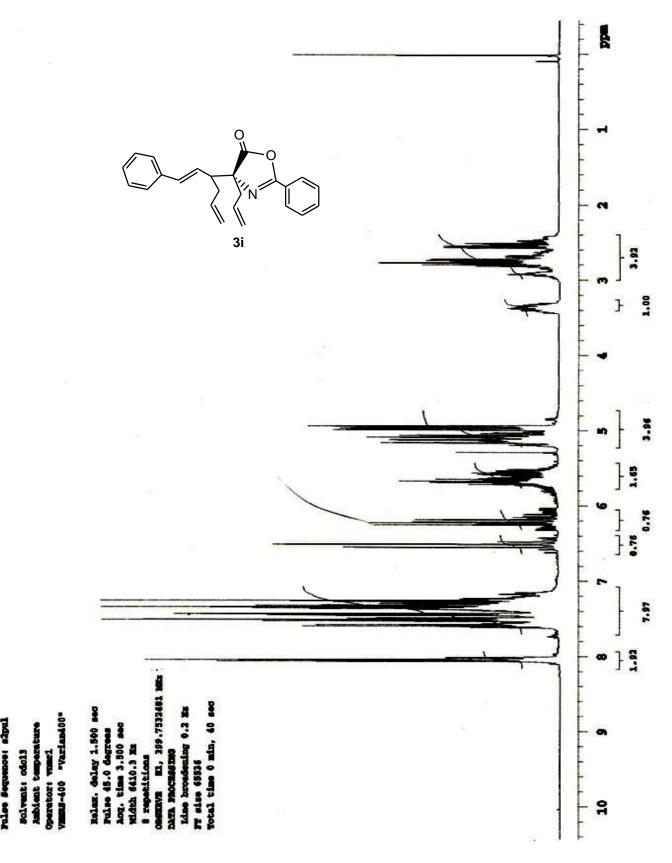


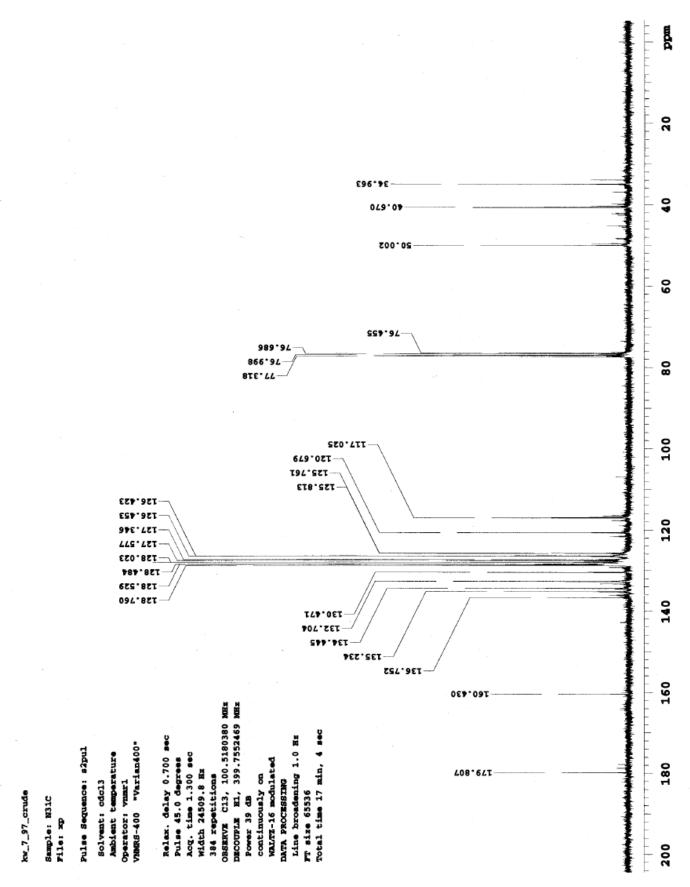
und d

#### (E/Z)-4-allyl-2-phenyl-4-(1-phenylhexa-1,5-dien-3-yl)oxazol-5(4H)-one (3i)

# <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

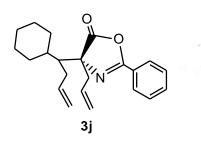
OR INTLL





### 4-Allyl-4-(1-cyclohexylbut-3-enyl)-2-phenyloxazol-5(4H)-one (3j)

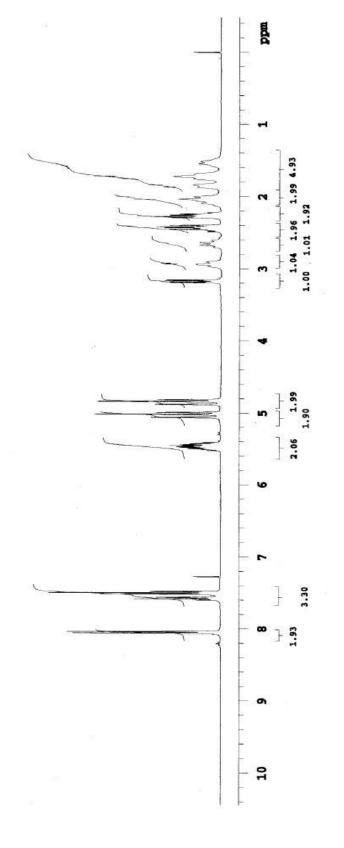
# <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



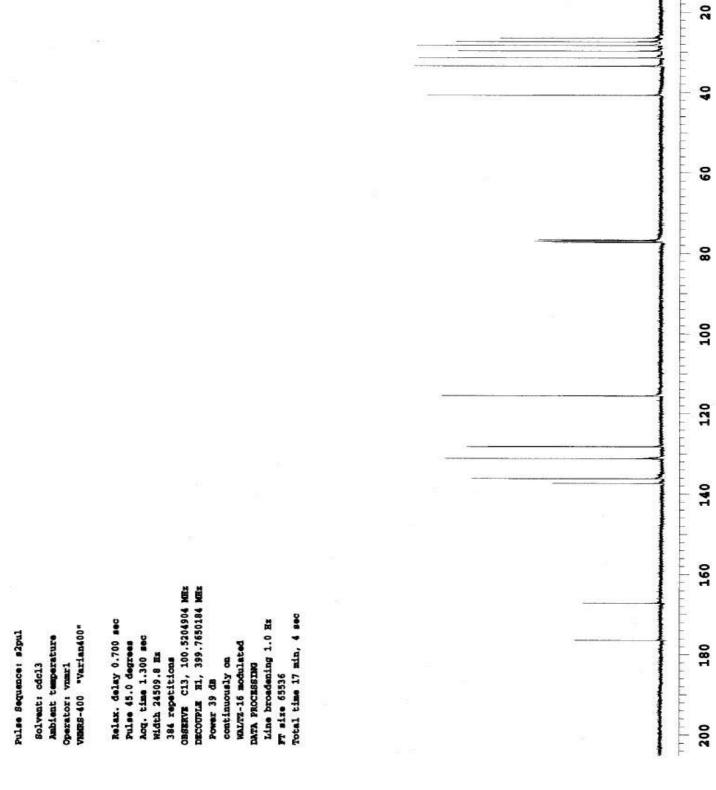


Pulse Sequence: s2pul

Solvent: cdc13 Ambient temperature perator: vnar1 Neuts-400 "Varian400 Relax. delay 1.500 sec Pulse 45.0 degrees Acq. time 3.500 sec Width 6410.3 Hr 8 repetitions DBEENVE H1, 399.7630265 NHr ANTA PROCESSING ALTA PROCESSING Line broadening 0.2 Hr T size 65536 Notal time 0 min, 40 sec



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

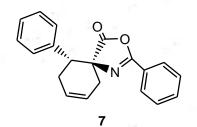


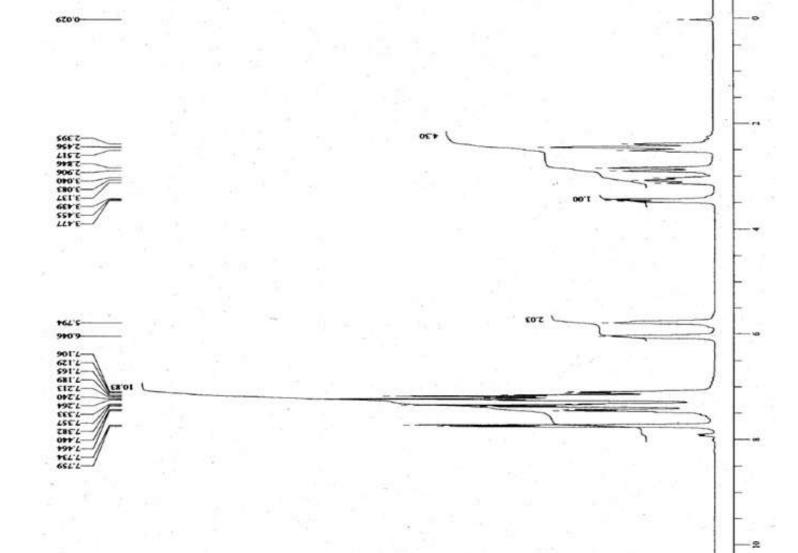
Inda

#### 2,10-Diphenyl-3-oxa-1-aza-spiro[4.5]deca-1,7-dien-4-one (7)

# <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)

DFILE Welt-search(1): 1-125-0-L000A664, MAR OBNUC IR ECOADD NON OFF 300 A/61 CORSET 130 00 Hz POBYT 130 00 Hz





<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



Pulse Sequence: s2pul

Solvent: odc13 Ambient temperature Operator: vnmr1 VNNNE-400 "Varian400" Relax. delay 0.700 sec Pulse 45.0 degrees Acq. time 1.300 sec Width 24509.8 Ex 384 repetitions 384 repetitions 384 repetitions 384 repetitions 384 repetitions 384 repetitions 399.7650184 MEx Power 39 dB continuously on Walry-16 modulated Arra PROCESSING Line broadening 1.0 Ex I aire 6536 Fotal time 17 min, 4 sec

S24

mdd

30

3

09

80

100

120

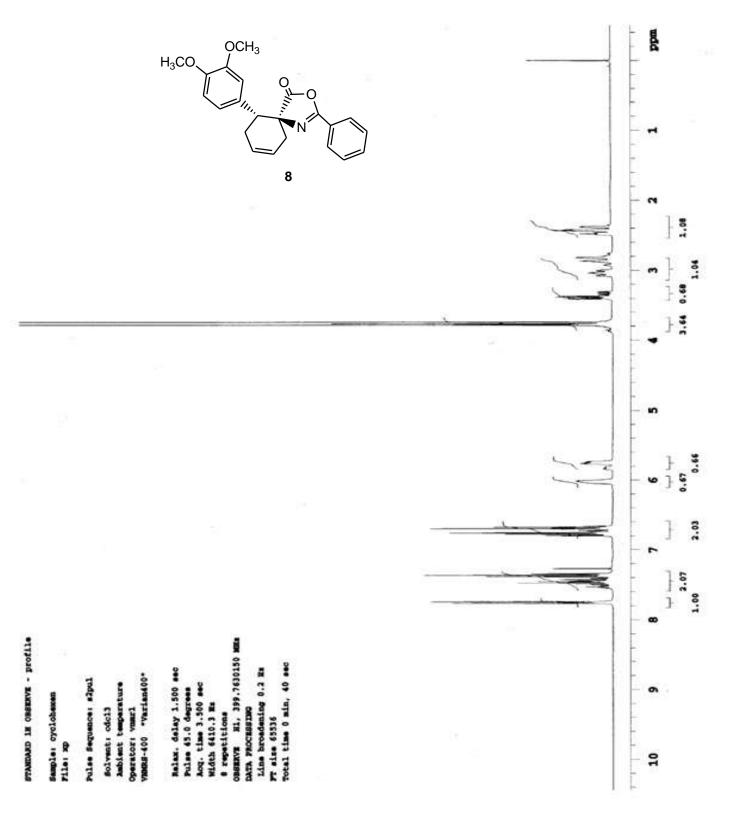
140

160

180

200

### 10-(3,4-Dimethoxy-phenyl)-2-phenyl-3-oxa-1-aza-spiro[4.5]deca-1,7-dien-4-one (8)



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



Pulse Sequence: s2pul

Tile: XD

20

\$

60

80

100

120

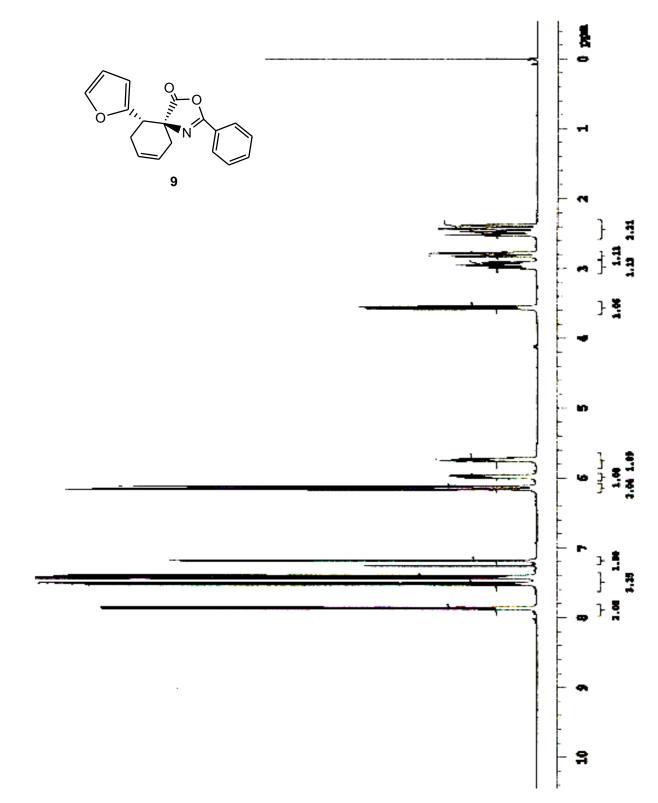
140

160

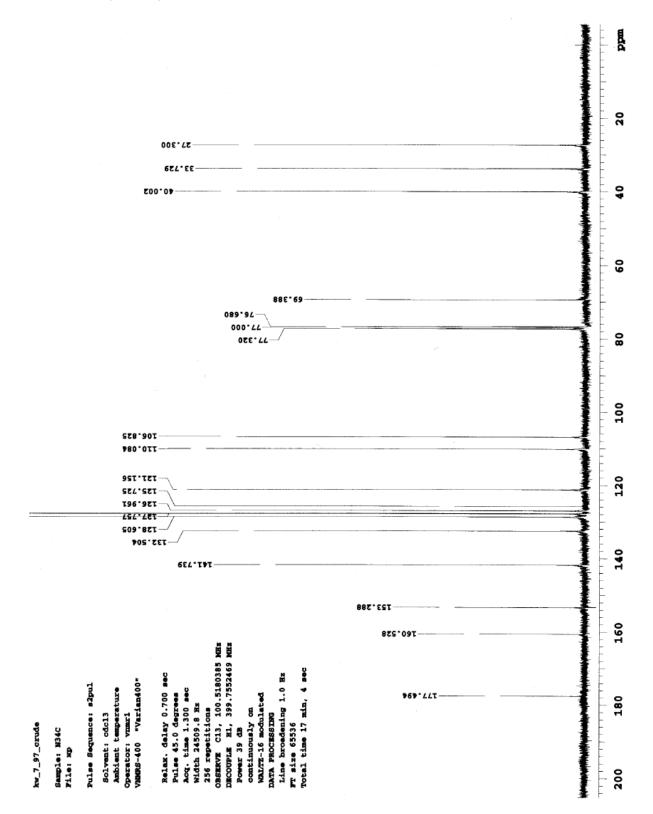
180

200

#### 10-Furan-2-yl-2-phenyl-3-oxa-1-aza-spiro[4.5]deca-1,7-dien-4-one (9)

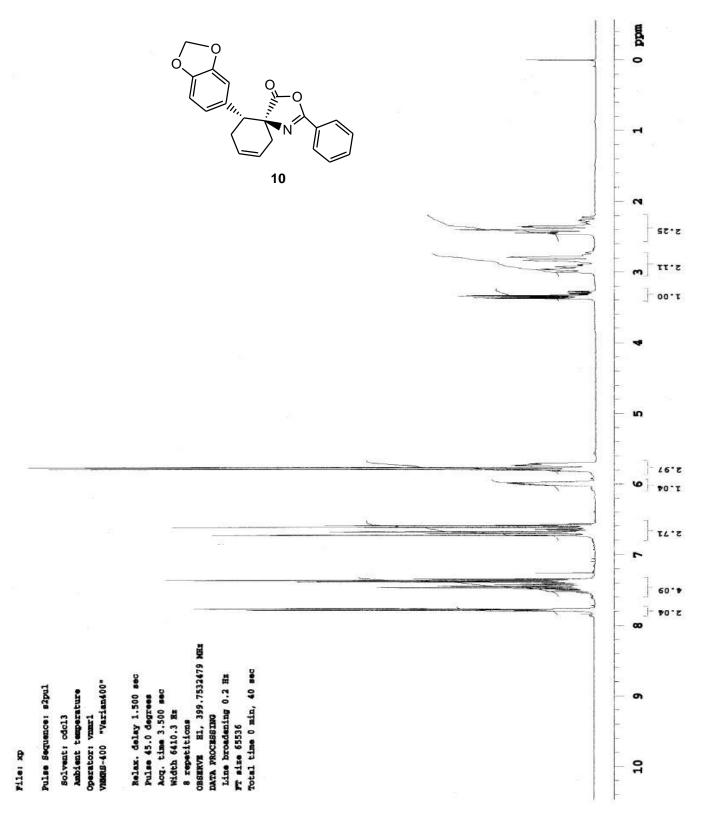


# <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



S28

#### 10-Benzo[1,3]dioxol-5-yl-2-phenyl-3-oxa-1-aza-spiro[4.5]deca-1,7-dien-4-one (10)



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

		100
		120
		140
	2 1994 65	160
Pulse Sequence: s2pul	Solvent: cdc13 Ambient temperature Operator: vnmr1 File: N43c vNNES-400 "Varian400" Relax. delay 0.700 sec Pulse 45.0 degrees Acq. time 1.300 sec Nidth 24509.8 Hz 128 repetitions OSCENYZ C13, 100.5180333 MHz DOSENYZ C13, 100.5180333 MHz DOSENYZ C13, 100.5180333 MHz DOSENYZ C13, 100.5180333 MHz DOSENYZ F1, 399.7552469 MHz Power 39 dB Continuously on NALTZ-16 modulated DATA PROCESSING DATA PROCESSING I.0 Hz Taize 65536 Total time 17 min, 4 sec	180
Pulse Seq	solvent: cdcl3 Ambient temperal Operator: vnmr1 File: N43c VNMRS-400 "Varia Pulse 45.0 degr Acq. time 1.300 Width 24509.8 H 128 repetitions 085ENT Cl3, 100 DECOUPLE M1, 39 DECOUPLE M1, 30 DECOUPLE M1, 30 DE	200

mdd

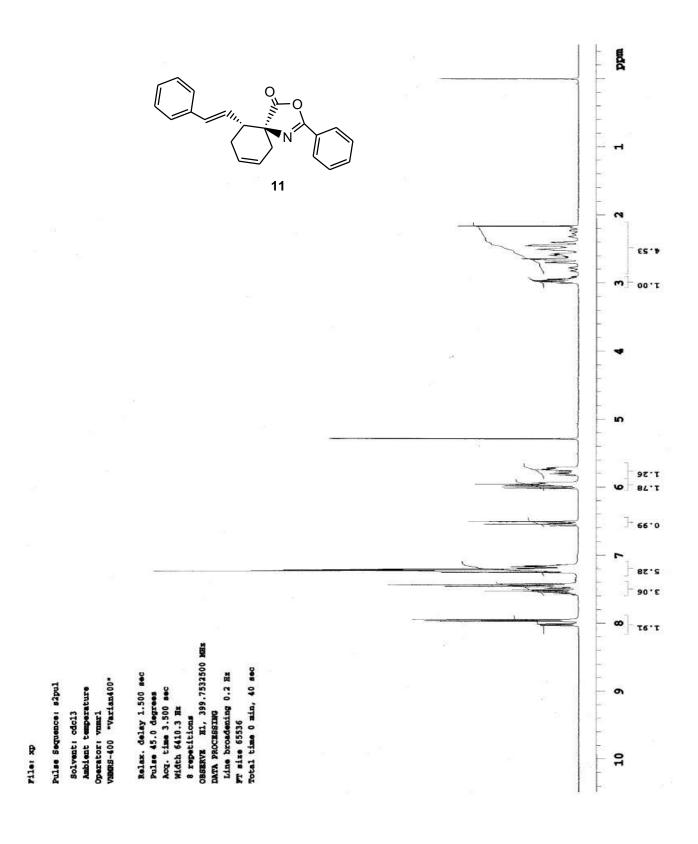
20

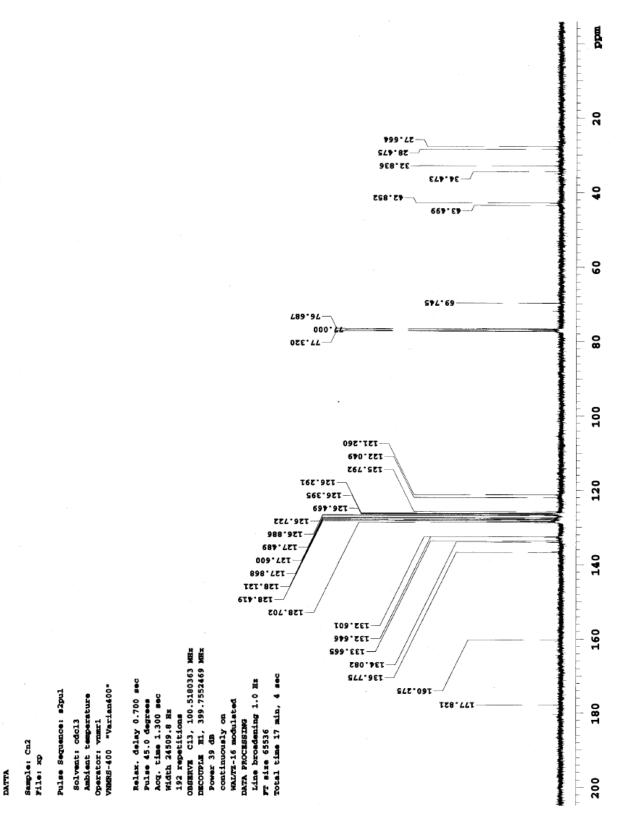
40

60

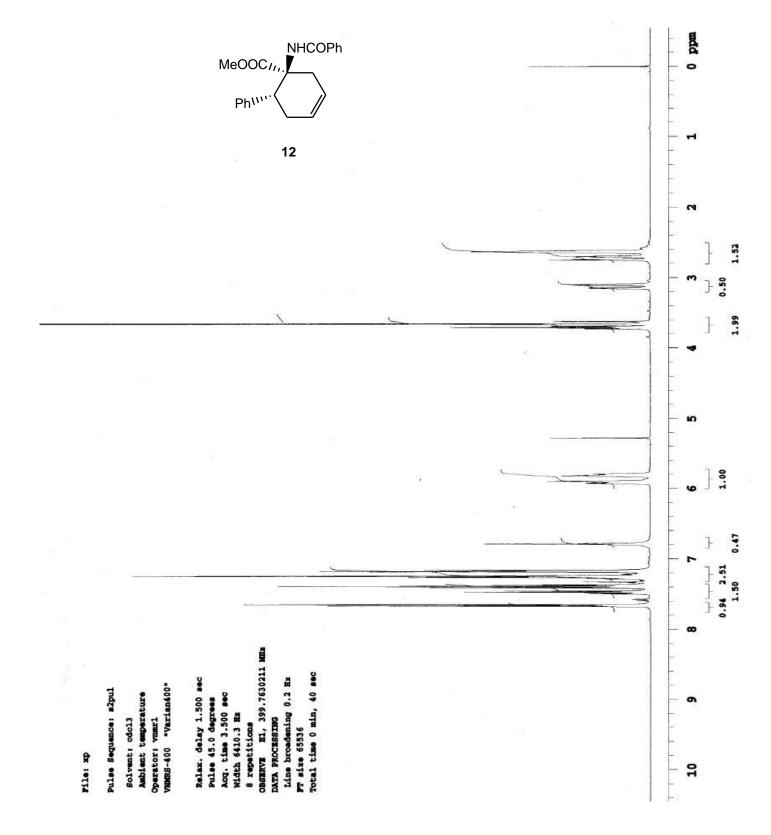
80

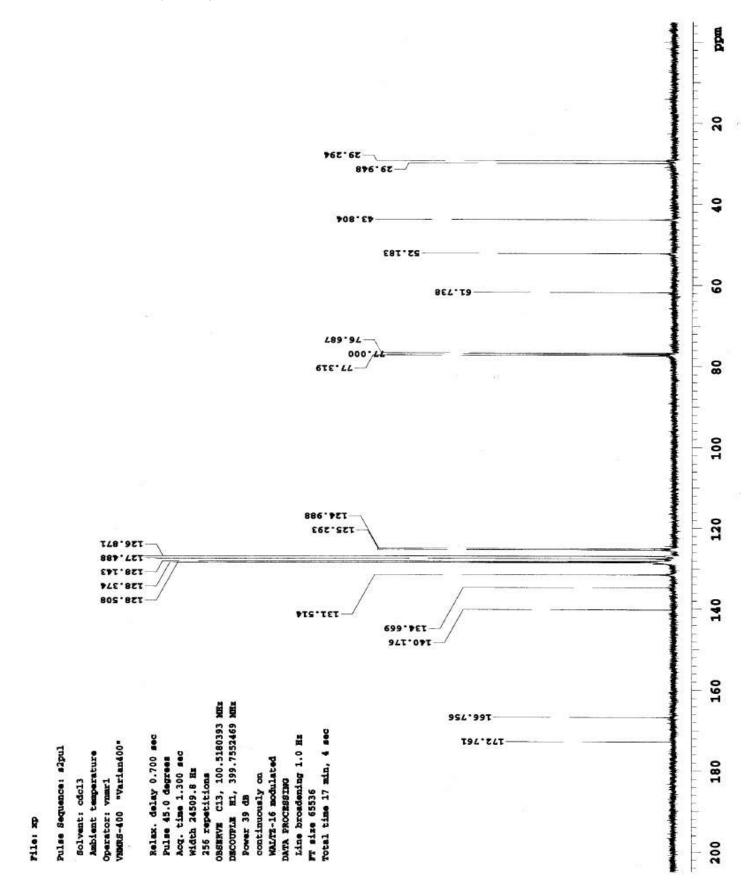
#### 2-Phenyl-10-styryl-3-oxa-1-aza-spiro[4.5]deca-1,7-dien-4-one (11)



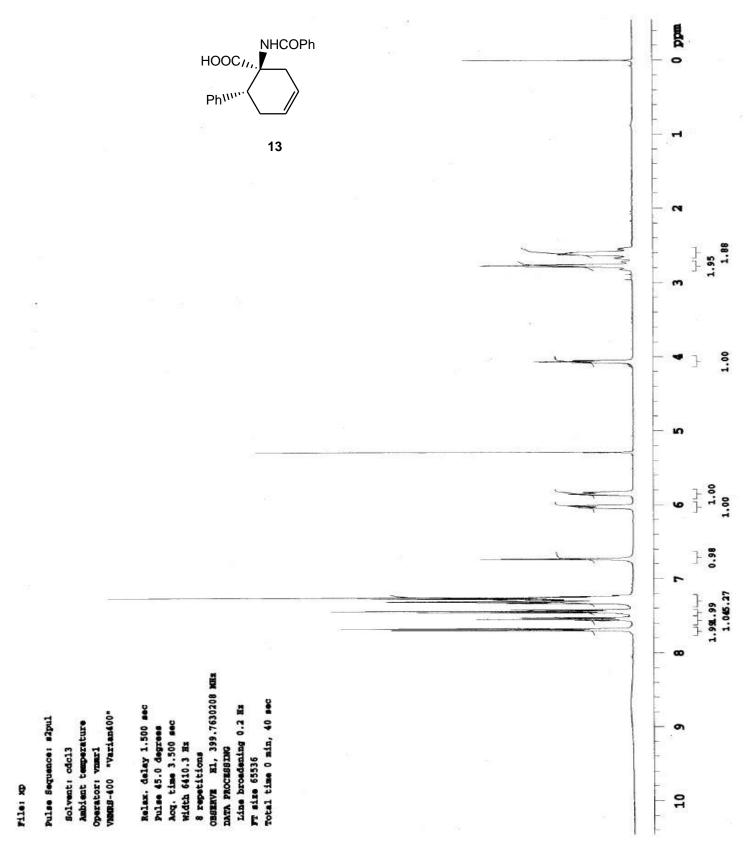


#### Methyl(1-Benzoylamino-6-phenyl-cyclohex-3-enecarboxylate) (12)





#### 1-Benzoylamino-6-phenyl-cyclohex-3-enecarboxylic acid (13)



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



Pulse Sequence: s2pul

Tile: M

Fulse 45.0 degrees Acq. time 1.300 sec Width 24509.8 Hz 384 repetitions 384 repetitions DBERNYE C13, 100.5204904 MHz DBECOUPLE H1, 399.7550184 MHz Power 39 dB Fower 30 dB Fow

S36

mdd

20

3

80

80

100

120

140

160

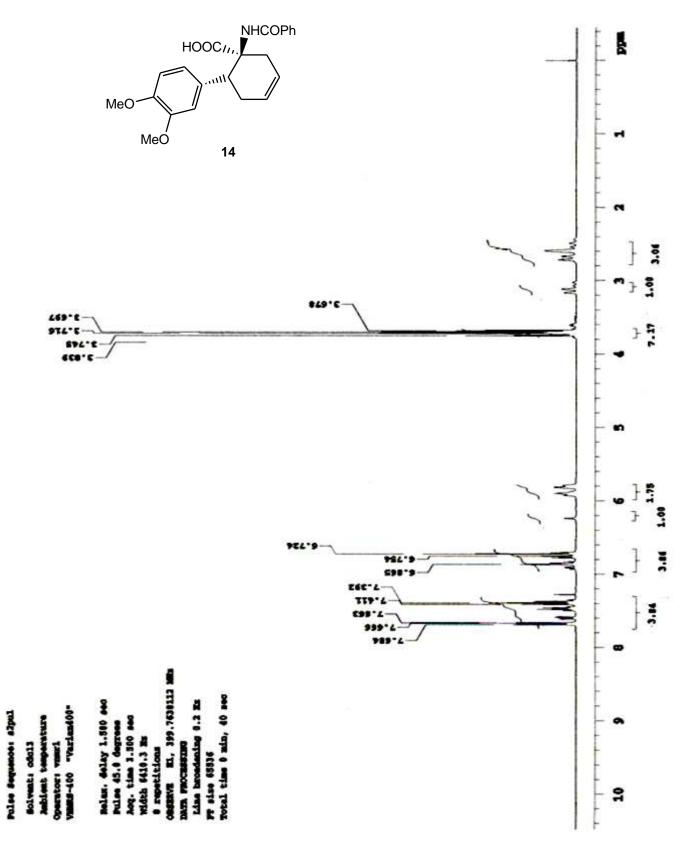
180

200

#### 1-Benzoylamino-6-(3,4-dimethoxy-phenyl)-cyclohex-3-enecarboxylic acid (14)

## <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

iller ag



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



Solvent: cdcl3 Ambient temperature Derator: vnmr1 NNURS-400 "Varian400" Relax. delay 0.700 mec Pulse 45.0 degrees Acg. time 1.300 mec Width 24509.8 Hz 384 repetitions OBSERVE C13, 100.5204904 MHz DECOUPLE H1, 399.7650184 MHz Power 39 dB continuously on NAL/FZ-16 modulated DATA PROCESSING Line broadening 1.0 Hz FT size 65536 Total time 17 min, 4 sec

S38

Indd

20

40

09

80

100

120

140

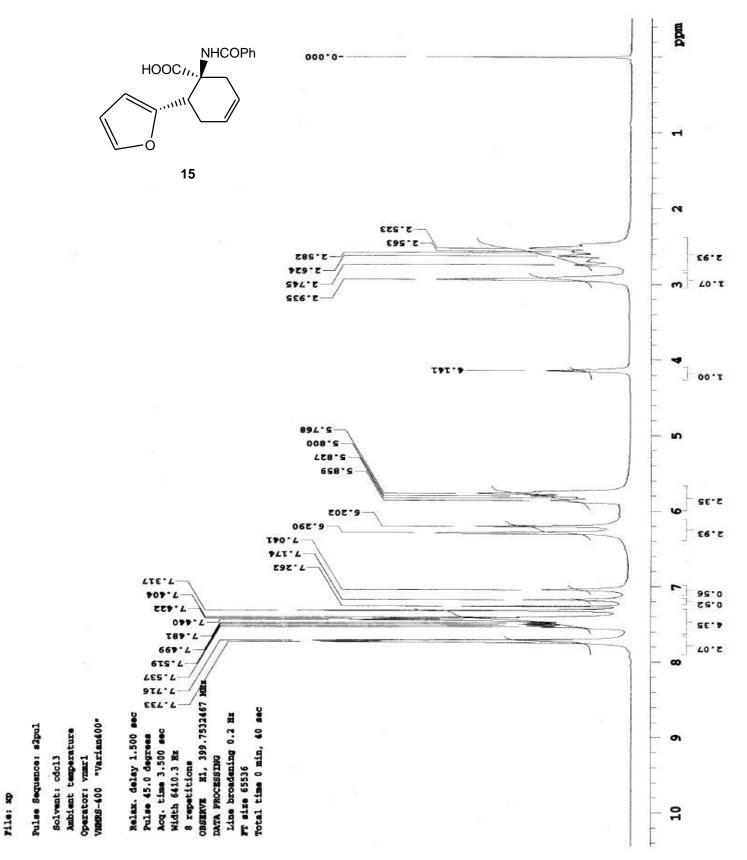
160

180

50

### 6-Benzo[1,3]dioxol-5-yl-1-benzoylamino-cyclohex-3-enecarboxylic acid (15)

## <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



Solvent: cdcl3 Ambient temperature Derator: vnmr1 VNDRS-400 "Varian400" Relax. delay 0.700 sec Pulse 45.0 degrees Acq. time 1.300 sec Width 24509.8 Ex 38 repetitions OBSERVE C13, 100.5204904 MHz DECOUPLE E1, 399.7650184 MHz Prever 39 dB continuously on WALTZ-16 modulated DATA PROCESSING Line broadening 1.0 Ex FT size 65536 Total time 17 min, 4 sec

S40

Idd

8

3

00

8

100

120

140

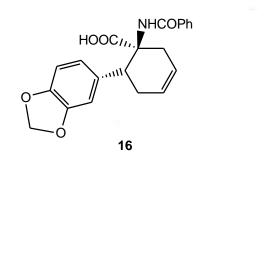
160

180

200

### 1-Benzoylamino-6-furan-2-yl-cyclohex-3-enecarboxylic acid (16)

### <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



BSERVE M1, 399.7532481 MHz

DATA PROCESSING

Relax. delay 1.500 sec

oq. time 3.500 sec

idth 6410.3 Hz

repetitions

ulse 45.0 degrees

"Varian400"

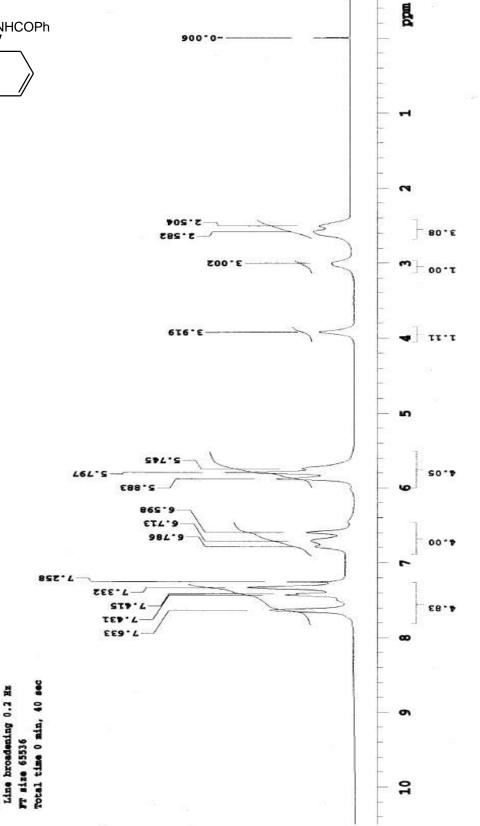
Ambient t Derator: Nong-400

temperature

Solvent: cdcl3

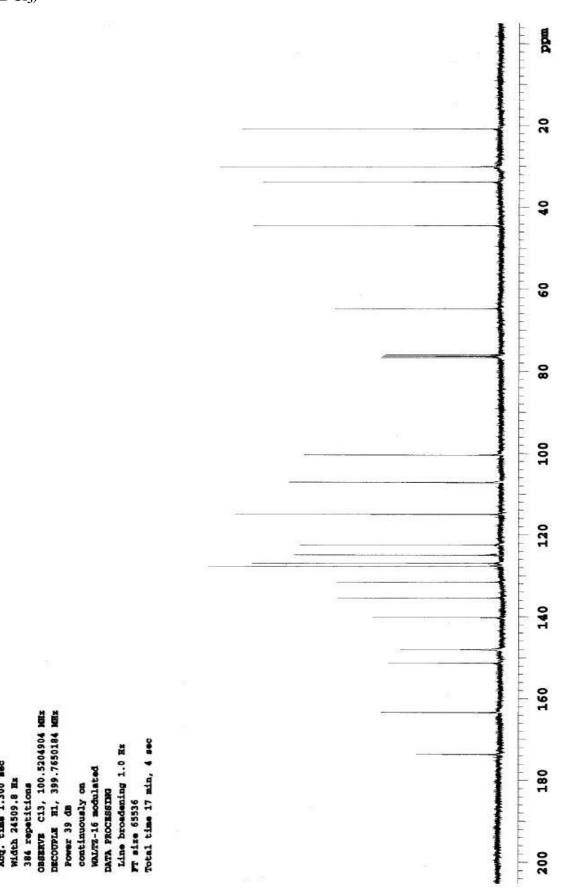
Pulse Sequence: s2pul

File: xp



S41

<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



File: xp

Pulse Sequence: s2pul

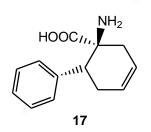
Ambient temperatury

Solvent: cdcl3

Relax. delay 0.700 sec MORE-400 "Varian400" Acq. time 1.300 sec Pulse 45.0 degrees Operator: vnmr1

#### 1-Amino-6-phenyl-cyclohex-3-enecarboxylic acid (17)

# <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)





Pulse Sequence: s2pul

mbient temperatur

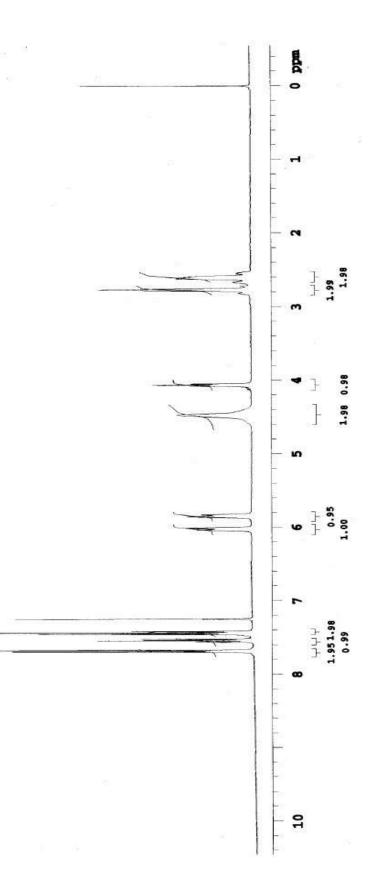
THE OWNER OF

BEALOFI

Jolvent: odol3

NBCRS-400 "Varian400" Relax. delay 1.500 sec Pulse 45.0 degrees Acq. time 3.500 sec Width 6410.3 Hz Width 6410.3 Hz Midth 6410.3 Hz Midth 500 sec Midth 500 sec Midth 200 sec Act a broadening 0.2 Hz Line broadening 0.2 Hz fotal time 0 min, 40 sec





<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)

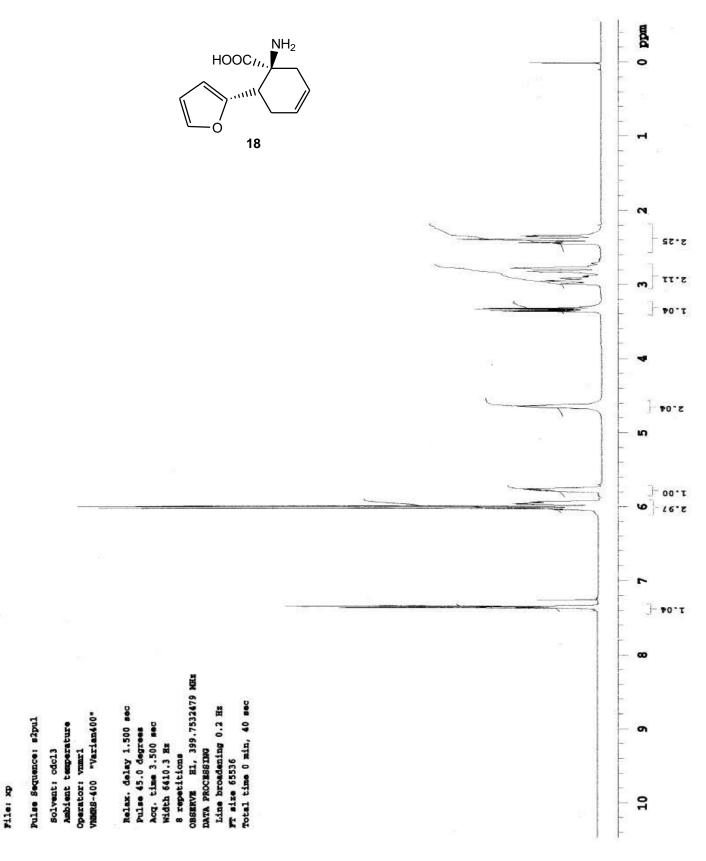


Tile: XD

\$

### 1-Amino-6-furan-2-yl-cyclohex-3-enecarboxylic acid (18)

# <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)

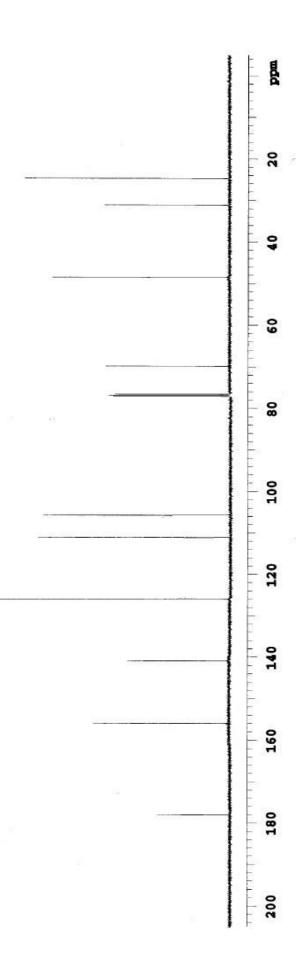


<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



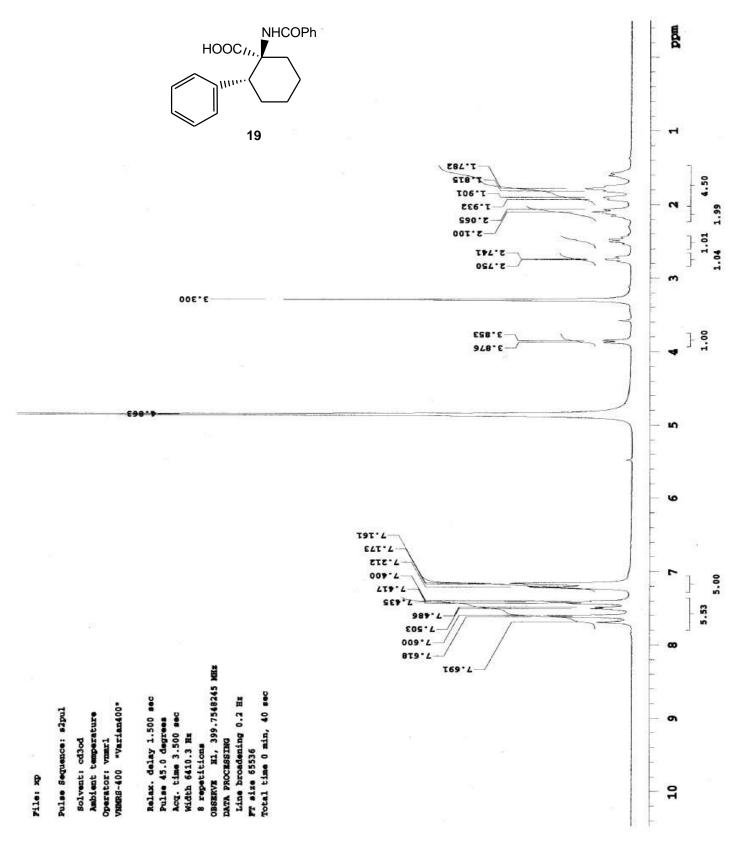
Pulse Sequence: s2pul

Solvent: cdcl3 Ambient temperature Dperator: vnmr1 NBDRS-400 "Varian400" Relax. delay 0.700 sec Fulse 45.0 degrees Acq. time 1.300 sec Width 24509.8 Hz 256 repetitions 256 repetitions DBCOUFLE H1, 399.7552469 MHz Power 39 dB continuously on WALTZ-16 modulated DATA PROCESING Line broadening 1.0 Hz FT size 65536 Fotal time 17 min, 4 sec

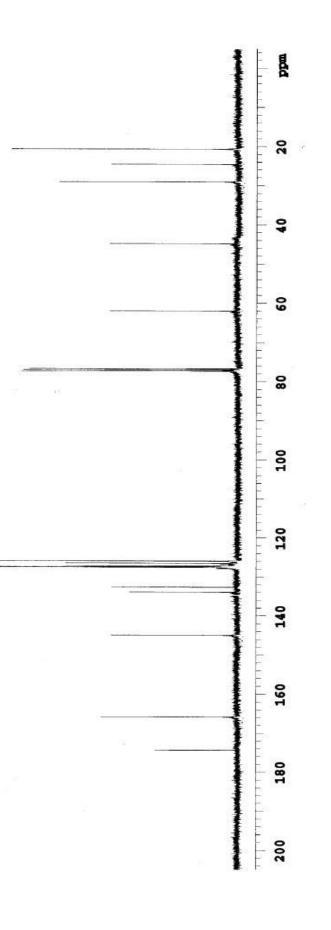


#### 1-benzamido-2-phenylcyclohexanecarboxylic acid (19)

### <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



<sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>)



S48

Total time 17 min, 4 sec

Line broadening 1.0 Hz

**TT size 65536** 

WALTZ-16 modulated

DATA PROCESSING

continuously on

POWER 39 dB

OBSERVE C13, 100.5180393 MEE DECOUPLE H1, 399.7552469 MEE

Relax. delay 0.700 sec

Pulse 45.0 degrees Acq. time 1.300 sec

width 24509.8 Hz

256 repetitions

VBORS-400 "Varian400"

Derator: vnmr1

Ambient temperature

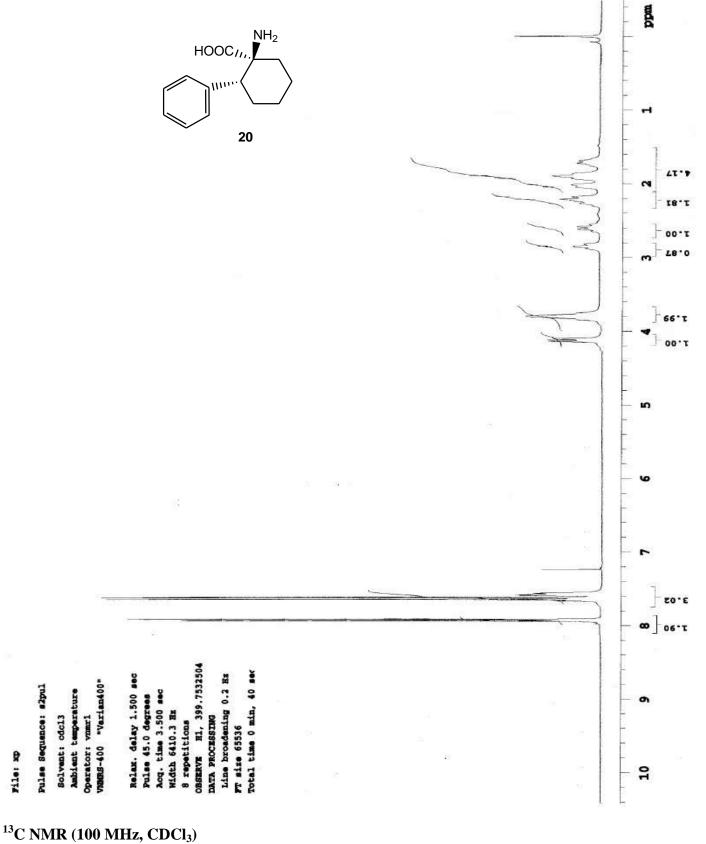
Solvent: cdcl3

Pulse Sequence: s2pul

File: XD

#### 1-Amino-2-phenyl-cyclohexanecarboxylic acid (20)

### <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>)



S49

Tile: XD

Pulse Sequence: s2pul

Solvent: cdc13 Ambient temperature Operator: vnmr1 VNNR5-400 "Varian400" Relax. delay 0.700 sec Pulse 45.0 degrees Acq. time 1.300 sec Width 24509.8 Hz 256 repetitions OBSERVE C13, 100.5180393 MEz DECOUPLE H1, 399.752469 MEz DECOUPLE H1, 399.752469 MEz Power 39 dB continuously on WALT2-16 modulated DATA PROCESSIMO Line broadening 1.0 Hz FT size 65536 Total time 17 min, 4 sec

