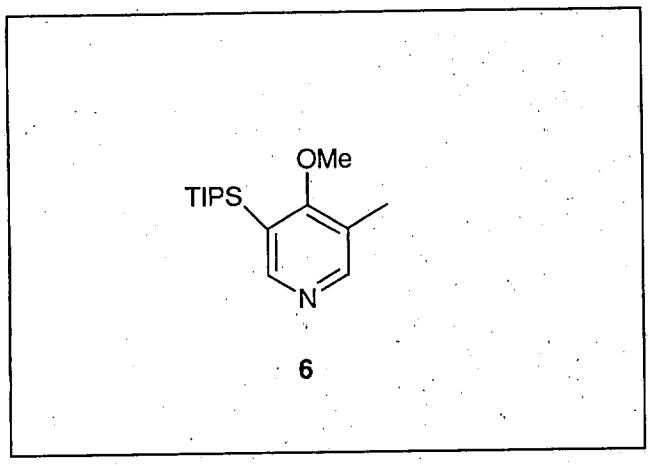
Compound Name: **4-Methoxy-5-methyl-3-triisopropylsilylpyridine**

Compound No: **6**

Notebook Pg(s): SH-III-51y



6

Formula: $C_{16}H_{29}NOSi$

Molecular Weight: 279.4931

Yields(s): 66%

Appearance: white crystalline solid

Stability: stable

mp/bp: 83-84 °C

$[\alpha]^{23}_D$

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	68.76	10.46	5.01	
Found:	68.74	10.50	4.94	

HRMS: Calculated Found

IR (thin film): cm^{-1}

2950, 2861, 1555, 1460, 1393, 1281, 1006, 882, 669

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

8.35 (d, 2 H, J = 12.4 Hz), 3.86 (s, 3 H), 2.33 (s, 3 H), 1.45 (septet, 3 H J = 7.5 Hz), 1.10 (d, 18 H, J = 7.5 Hz)

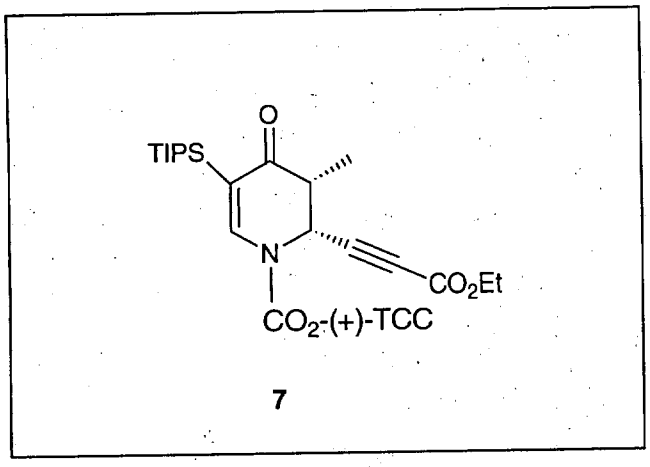
^{13}C-NMR (75 MHz) δ

171.5, 155.6, 154.5, 123.6, 121.6, 60.0, 19.0, 15.7, 11.9.

Literature References:

Compound Name: **(2*R*, 3*R*)-1-[(1*S*, 2*R*)-*trans*-2-(α-Cumyl)cyclohexyloxycarbonyl]-2-(ethyl propiolate)-3-methyl-5-triisopropylsilyl-2,3-dihydro-4-pyridine**

Compound No: **7**
Notebook Pg(s): SH-III-52-w



7

Formula: $C_{36}H_{54}NO_5Si$

Molecular Weight: 608.3768

Yields(s): 70%

Appearance: white solid

Stability: stable

mp/bp: 94-96 °C

$[\alpha]^{23}_D$ -30.2 (*c* 0.41, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	71.01	8.94	2.30	
Found:	71.06	8.76	2.28	

HRMS: Calculated Found

IR (thin film): cm^{-1}

2931, 2868, 1718, 1665, 1574, 1381, 1312, 1243

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

7.63 (s, 1 H), 7.35-7.26 (m, 4 H), 7.15-7.08 (t, 1 H, *J* = 6.5 Hz), 4.98-4.85 (m, 1 H), 4.12 (q, 2 H, *J* = 6.7 Hz), 3.43 (d, 1 H, *J* = 5.5 Hz), 2.43-2.21 (m, 2 H), 2.10 (d, 1 H, *J* = 10.0 Hz), 1.92-1.90 (m, 1 H), 1.82-1.70 (m, 2 H), 1.39-1.18 (m, 15 H), 1.05-.90 (m, 23 H)

^{13}C-NMR (75 MHz) δ

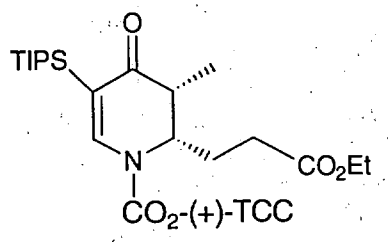
196.7, 153.0, 151.4, 146.1, 127.9, 125.2, 125.0, 111.4, 81.1, 78.6, 72.1, 71.1, 62.0, 51.1, 49.4, 41.7, 41.5, 39.4, 33.3, 31.3, 26.7, 26.3, 25.8, 24.7, 22.9, 20.7, 18.8, 18.7, 14.1, 13.9, 11.1, 10.7

Literature References:

Compound Name: **(2*S*, 3*R*)-1-[(1*S*, 2*R*)-*trans*-2-(α-Cumyl)cyclohexyloxycarbonyl]-2-(ethyl propionate)-3-methyl-5-triisopropylsilyl-2,3-dihydro-4-pyridine**

Compound No: **8**

Notebook Pg(s): SH-III-52y



8

Formula: $C_{36}H_{58}NO_5Si$

Molecular Weight: 612.4084

Yields(s): 100%

Appearance: clear oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +74.5 (*c* 7.28, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:				
Found:				

HRMS: Calculated 612.4084 Found 612.4102

IR (thin film): cm^{-1}

2942, 2848, 1734, 1713, 1665, 1574, 1462, 1381, 1317, 1280, 1248

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

7.73 (s, 1 H), 7.34-7.25 (m, 4 H), 7.13 (t, *J* = 6.2 Hz, 1 H), 4.95 (br s, 1 H), 4.10-4.09 (m, 2 H), 3.19 (d, *J* = 2.7 Hz, 1 H), 2.5 (br s, 1 H), 2.35-1.60 (m, 9 H), 1.39-1.20 (m, 9 H), 1.08-1.01 (m, 22 H)

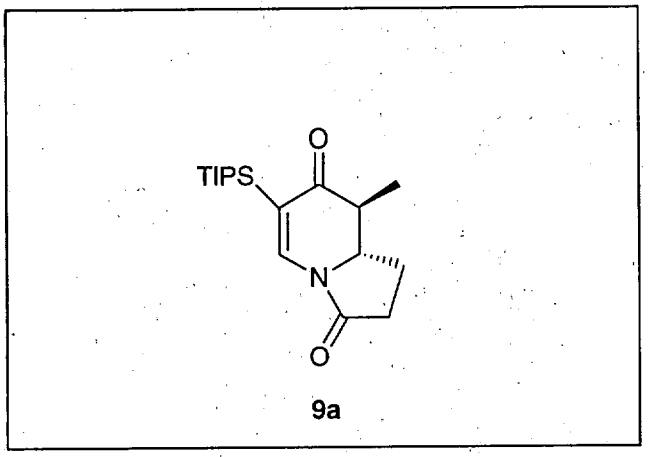
^{13}C-NMR (75 MHz) δ

199.6, 172.5, 152.5, 152.3, 146.9, 128.0, 125.5, 125.3, 110.9, 78.5, 60.6, 56.0, 51.0, 43.0, 39.7, 33.6, 31.1, 30.3, 27.1, 25.9, 24.9, 22.6, 22.1, 19.0, 18.9, 14.3, 11.2, 10.9

Literature References:

Compound Name: **(8*S*, 9*S*)-8-Methyl-6-triisopropylsilyl-8,9-dihydroindolizidine-3,7-dione**

Compound No. **9a**
Notebook Pg(s): SH-III-53w



9a

Formula: $C_{18}H_{31}NO_2Si$

Molecular Weight: 321.53

Yields(s): 80%

Appearance: white solid

Stability: stable

mp/bp: 80-82 °C

$[\alpha]^{23}_D$ -252.0 (*c* 0.60, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	67.24	9.72	4.35	
Found:	67.09	9.68	4.35	

HRMS: Calculated Found

IR (thin film): cm^{-1}

2935, 2869, 1725, 1658, 1565, 1460, 1378, 1301, 1262, 1130, 883

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

7.74 (s, 1 H), 3.80-3.70 (m, 1 H), 2.62-2.56 (m, 2 H), 2.46-2.34 (m, 2 H), 1.83 (pentet, *J* = 11.0 Hz, 1H), 1.40-1.27 (m, 2 H), 1.14 (d, *J* = 6.6 Hz, 2 H), 1.08-1.02 (m, 18 H)

^{13}C-NMR (75 MHz) δ

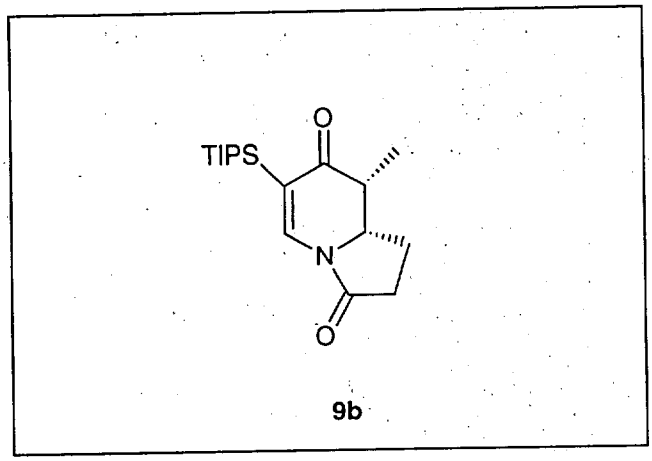
199.7, 171.7, 142.6, 112.6, 60.5, 46.3, 31.2, 25.6, 19.0, 18.9, 11.2, 9.8

Literature References:

Compound Name: **(8*R*, 9*S*)-8-Methyl-6-triisopropylsilyl-5,6-dihydroindolizidine-3,7-dione**

Compound No: **9b**

Notebook Pg(s): SH-III-53w



9b

Formula: $C_{18}H_{31}NO_2Si$

Molecular Weight: 321.53

Yields(s): 80%

Appearance: clear oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -188.0 (*c* 1.15, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	67.24	9.72	4.35	
Found:	67.47	9.76	4.07	

HRMS: Calculated Found

IR (thin film): cm^{-1}

2932, 2868, 1723, 1654, 1558, 1456, 1381, 1264

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

7.72 (s, 1 H), 4.31-4.24 (m, 1 H), 2.64-2.59 (m, 2 H), 2.55-2.47 (m, 1 H), 2.13-2.00 (m, 2 H), 1.38-1.29 (m, 3 H), 1.09-1.03 (m, 21 H)

^{13}C-NMR (75 MHz) δ

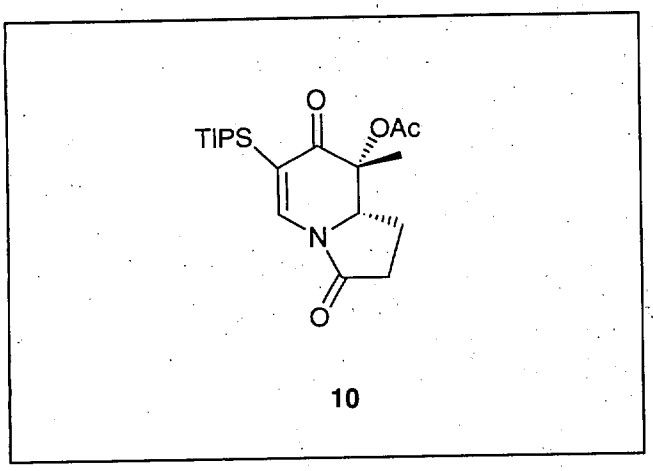
202.1, 172.2, 142.6, 111.1, 58.0, 43.9, 31.0, 20.0, 19.1, 19.0, 11.4, 11.2, 10.5

Literature References:

Compound Name:
(8*R*, 9*S*)-8-Acetoxy-8-methyl-6-triisopropylsilyl-8,9-dihydroindolizidine-3,7-dione

Compound No: **10**

Notebook Pg(s): SH-III-42w



10

Formula: $C_{20}H_{33}NO_4Si$

Molecular Weight: 379.574

Yields(s): 54%

Appearance: white solid

Stability: stable

mp/bp: 119-120 °C

$[\alpha]^{23}_D$ -37.4 (*c* 0.27, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	63.28	8.76	3.69	
Found:	63.28	8.88	3.55	

HRMS: Calculated Found

IR (thin film): cm^{-1}

2989, 2942, 1725, 1685, 1448, 1426, 1370, 1267

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

7.62 (s, 1 H), 4.47 (ddd, *J* = 2.1, 6.8, 13.3 Hz, 1 H), 3.42 (m, 1 H), 3.02 (td, 1 H, *J* = 4.6, 12.3 Hz, 1 H), 2.65-2.22 (m, 6 H), 1.85 (m, 1 H), 1.10 (d, *J* = 6.5 Hz, 3 H)

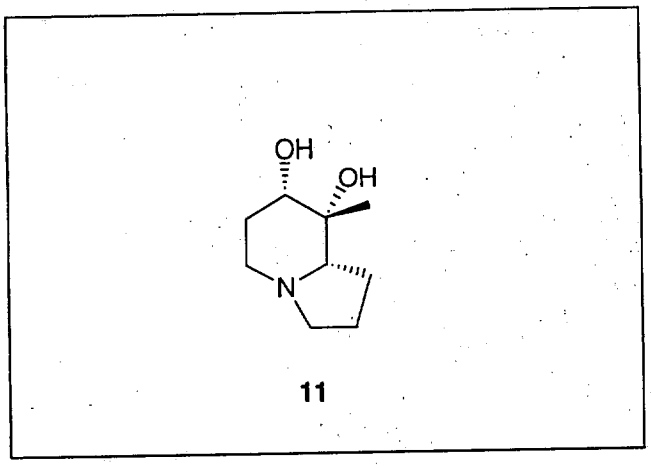
^{13}C-NMR (75 MHz) δ

194.5, 171.4, 169.0, 141.6, 110.5, 76.4, 63.4, 30.9, 21.4, 18.9, 18.7, 18.3, 14.9, 11.2

Literature References:

Compound Name: **(8*S*, 9*R*)-8-Methyl-octahydroindolizine-7,8-diol**

Compound No: **11**
Notebook Pg(s): SH-III-54y



11

Formula: $C_9H_{17}NO_2$

Molecular Weight: 171.1259

Yields(s): 85%

Appearance: white solid

Stability: stable

mp/bp: 128-129 °C

$[\alpha]^{23}_D$ +40.0 (*c* 0.15, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:				
Found:				

HRMS: Calculated 171.1259 Found 171.1264

IR (thin film): cm^{-1}

3479, 2944, 1654, 1364, 1077

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

3.21 (m, 1 H), 2.99 (m, 2 H), 2.72 (s, 1 H), 2.17 (m, 1 H), 2.08-1.50 (m, 8 H), 1.18 (s, 3 H)

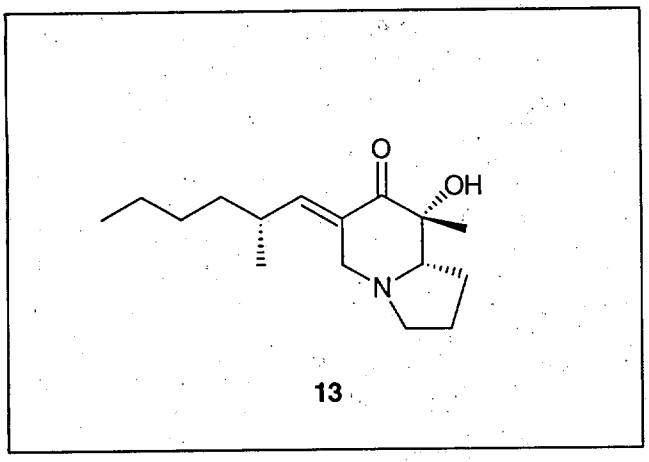
^{13}C-NMR (75 MHz) δ

74.4, 71.2, 70.7, 54.7, 50.1, 31.3, 23.7, 22.3, 20.4

Literature References:

Compound Name:
(8*R*, 8$_\alpha$*S*)-8-Hydroxy-8-methyl-6-((*Z*)-2(*R*)-methyl-hexylidene)octahydroindolizin-7-one

Compound No: **13**
Notebook Pg(s): SH-III-61y



13

Formula: $C_{16}H_{27}NO_2$

Molecular Weight: 265.2042

Yields(s): 51%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -6.3 (*c* 0.50, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:				
Found:				

HRMS: Calculated Found

IR (thin film): cm^{-1}

3421, 2957, 2871, 1700, 1122.

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

6.52 (dd, *J* = 1.4, 10.2 Hz, 1 H), 4.02 (d, *J* = 13.9 Hz, 1 H), 3.67 (br s, 1 H), 3.22 (m, 1 H), 2.97 (dd, *J* = 2.6, 14.0 Hz, 1 H), 2.46-2.28 (m, 3 H), 2.00-1.80 (m, 2 H), 1.40-1.11 (m, 3 H), 1.26 (s, 3 H), 1.02 (d, *J* = 6.6 Hz, 3 H), 0.87 (t, *J* = 6.8 Hz, 3 H)

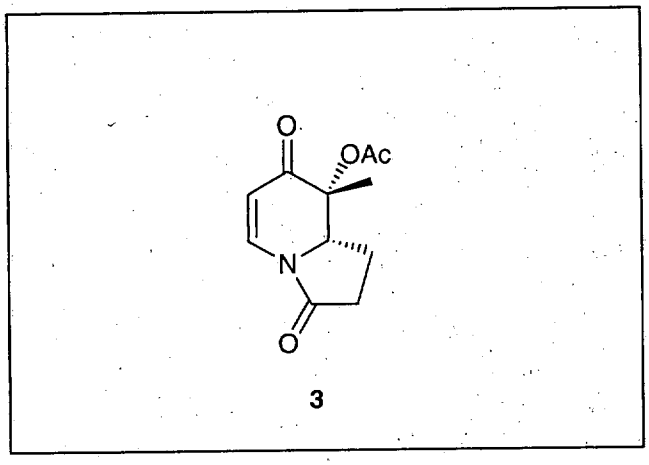
^{13}C-NMR (75 MHz) δ

197.2, 148.0, 129.6, 77.4, 73.1, 69.2, 55.3, 52.1, 36.3, 32.9, 29.8, 23.6, 22.8, 20.0, 17.8, 14.0

Literature References:

Compound Name: **(8*R*, 9*S*)-8-Acetoxy-8-methyl-8,9-dihydroindolizidine-3,7-dione**

Compound No: **3**
Notebook Pg(s): SH-III-45y



3

Formula: $C_{11}H_{13}NO_4$

Molecular Weight: 223.0845

Yields(s): 93%

Appearance: white solid

Stability: stable

mp/bp: 102-104 °C

$[\alpha]^{23}_D$ +27.3 (*c* 0.30, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:				
Found:				

HRMS: Calculated 223.0845 Found 223.0838

IR (thin film): cm^{-1}

2921, 1725, 1685, 1598, 1416, 1325, 1269, 1243, 1198, 1096

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

7.64 (d, *J* = 7.9 Hz, 1 H), 5.44 (d, *J* = 8.1 Hz, 1 H), 3.93 (dd, *J* =7.1, 9.7 Hz, 1 H), 2.66-2.58 (m, 2 H), 2.40-2.15 (m, 2 H), 2.01 (s, 3 H), 1.53 (s, 3 H)

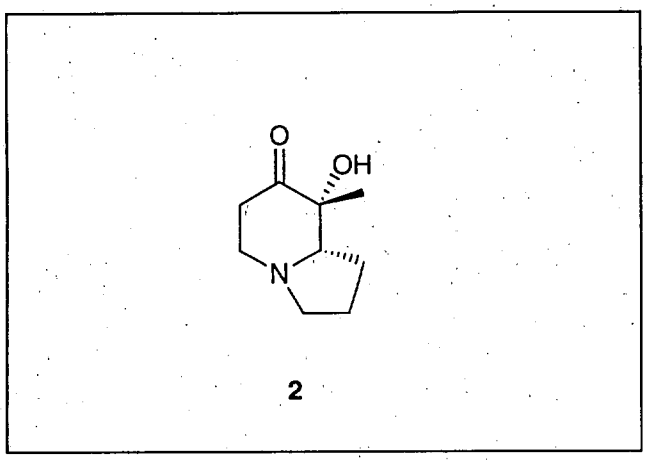
^{13}C-NMR (75 MHz) δ

190.6, 171.7, 169.0, 136.0, 106.2, 77.5, 76.2, 63.6, 30.8, 21.4, 18.3, 14.9, 0.2

Literature References:

Compound Name: **(8*R*, 8$_\alpha$*S*)-8-Hydroxyl-8-methyl-7-octahydroindolizidone)**

Compound No: **2**
Notebook Pg(s): SH-III-56w



2

Formula: $C_9H_{16}NO_2$

Molecular Weight: 170.1181

Yields(s): 75%

Appearance: colorless oil

Stability: not stable

mp/bp:

$[\alpha]^{23}_D$ -45.0 (*c* 0.50, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:				
Found:				

HRMS: Calculated Found

IR (thin film): cm^{-1}

3411, 2934, 1720, 1645, 1374, 1100

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

3.78 (br s, 1 H), 3.25 (dd, *J* = 7.5, 9.6 Hz, 1 H), 3.15 (t, *J* = 7.5 Hz, 1 H), 3.04 (ddd, *J* = 7.5, 12.5, 14.5 Hz, 1 H), 2.35 (ddd, *J* = 3.5, 9.6, 12.5 Hz, 1 H), 2.31 (m, 1 H), 2.23 (dd, *J* = 2.7, 14.5 Hz, 1 H), 2.16 (t, *J* = 8.1 Hz, 1 H), 1.92-1.72 (m, 4 H), 1.16 (s, 3 H)

^{13}C-NMR (75 MHz) δ

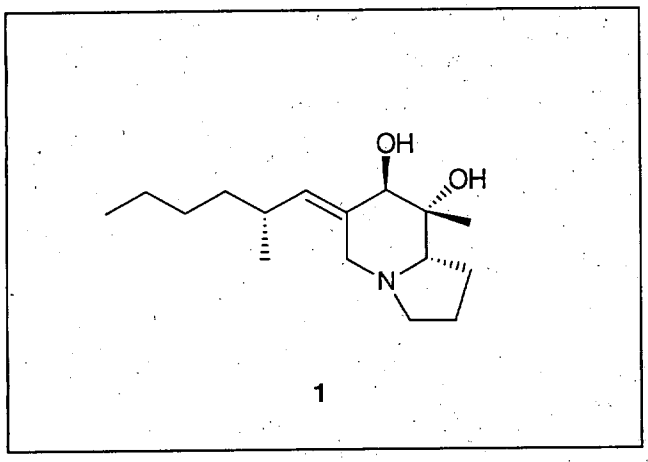
209.5, 75.6, 72.5, 54.2, 50.3, 36.5, 23.7, 23.0, 17.0

Literature References:

Compound Name: **(+)-Allopumjliotoxin 267(A)**

Compound No: **1**

Notebook Pg(s): SH-III-62w



1

Formula: $C_{16}H_{29}NO_2$

Molecular Weight: 267.2198

Yields(s): 95%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +30.0 (*c* 0.50, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:				
Found:				

HRMS: Calculated Found

IR (thin film): cm^{-1}

3420, 2956, 1558, 1456

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ

5.34 (d, *J* = 9.5 Hz, 1 H), 3.73 (s, 1 H), 3.61 (d, *J* = 11.7 Hz, 1 H), 3.05 (m, 1 H), 2.72 (d, *J* = 12.8 Hz, 1 H) 2.50-2.47 (m, 1 H), 2.40-2.36 (m, 1 H), 2.30-2.24 (m, 1 H), 2.0 (br s, 1 H). 1.77-1.60 (m, 2 H), 1.42-1.12 (m, 3 H), 1.22 (s, 3 H), 0.98 (d, *J* = 6.5 Hz, 3 H), 0.87 (t, *J* = 7.2 Hz, 3 H)

^{13}C-NMR (75 MHz) δ

139.0, 133.5, 81.1, 70.5, 65.5, 54.5, 49.1, 37.3, 32.2, 29.9, 23.0, 22.8, 21.4, 20.8, 14.3

Literature References:

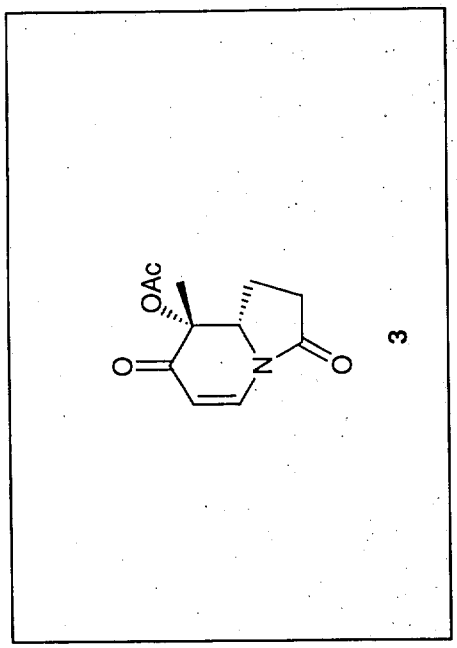
Comparing our (+)-Allopumiliotoxin 267A NMR Data with Overman's (+)-Allopumiliotoxin NMR Data

^{1}H NMR our data (300 MHz)	Overman's data (500 MHz)
5.34 (d, J = 9.5 Hz, 1 H)	5.33 (dd, J = 1.5, 9.5 Hz, 1 H)
3.73 (s, 1 H)	3.71 (s, 1 H)
3.61 (d, J = 11.7 Hz, 1 H)	3.60 (d, J = 12.5 Hz, 1 H)
3.05 (m, 1 H)	3.10 (m, 1 H)
2.72 (d, J = 12.8 Hz, 1 H)	2.71 (d, J = 12.0 Hz, 1 H)
2.50-2.47 (m, 1 H)	2.50-2.45 (m, 1 H)
2.40-2.36 (m, 1 H)	2.41-2.37 (m, 1 H)
2.30-2.24 (m, 1 H)	2.40-2.20 (m, 1 H)
2.0 (br s, OH)	2.91 (br s, OH)
1.77-1.60 (m, 2 H)	1.80-1.60 (m, 2 H)
1.42-1.12 (m, 3 H)	1.40-1.10 (m, 3 H)
1.22 (s, 3 H)	1.21 (s, 3 H)
0.98 (d, J = 6.5 Hz, 3 H)	0.97 (d, J = 6.5 Hz, 3 H)
0.87 (t, J = 7.2 Hz, 3 H)	0.87 (t, J = 7.2 Hz, 3 H)

^{13}C NMR our data (75 MHz)	Overman's data (125 MHz)
14.3	14.1
20.8	20.6
21.4	21.2
22.8	22.7
23.0	22.8
29.9	29.7
32.2	32.0
37.3	37.1
49.1	48.9
54.5	54.3
65.5	65.2
70.5	70.3
81.1	80.9
133.5	133.4
139.0	138.7

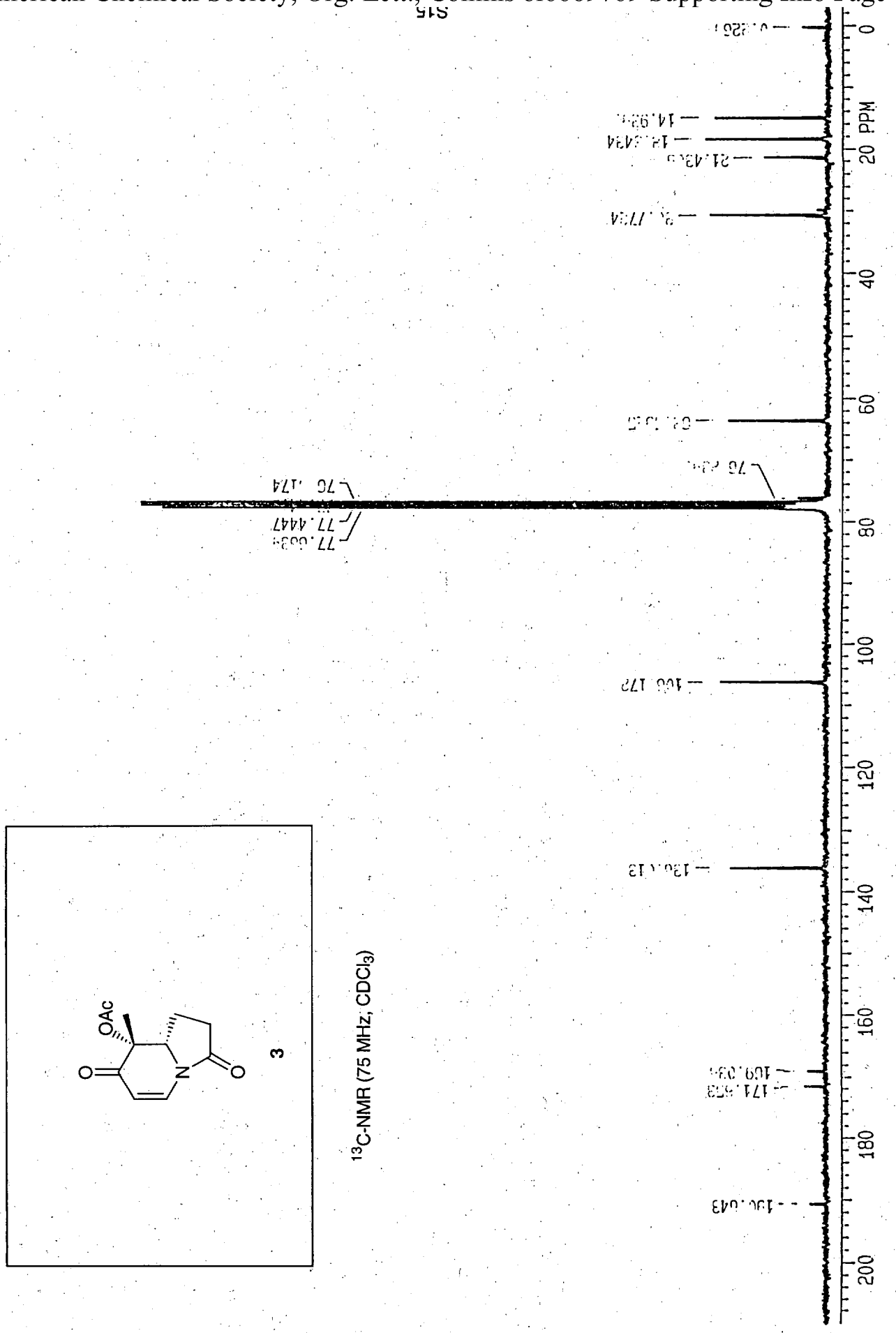
our $[\alpha]^{23}_{D}$ +30.0 (c 0.50, $CHCl_3$)

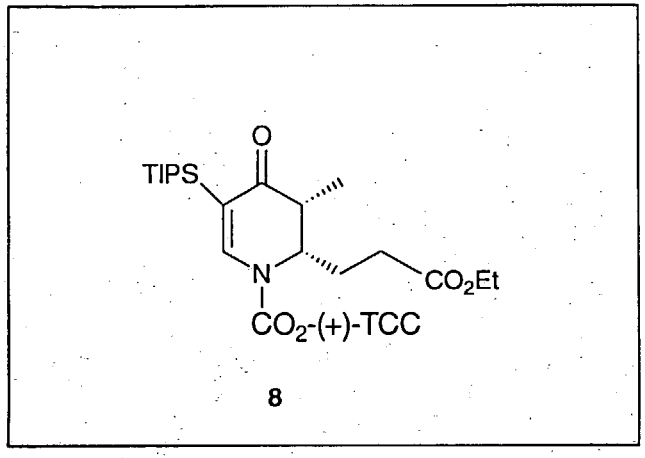
literature $[\alpha]^{23}_{D}$ +31 (c 0.22, MeOH)



3

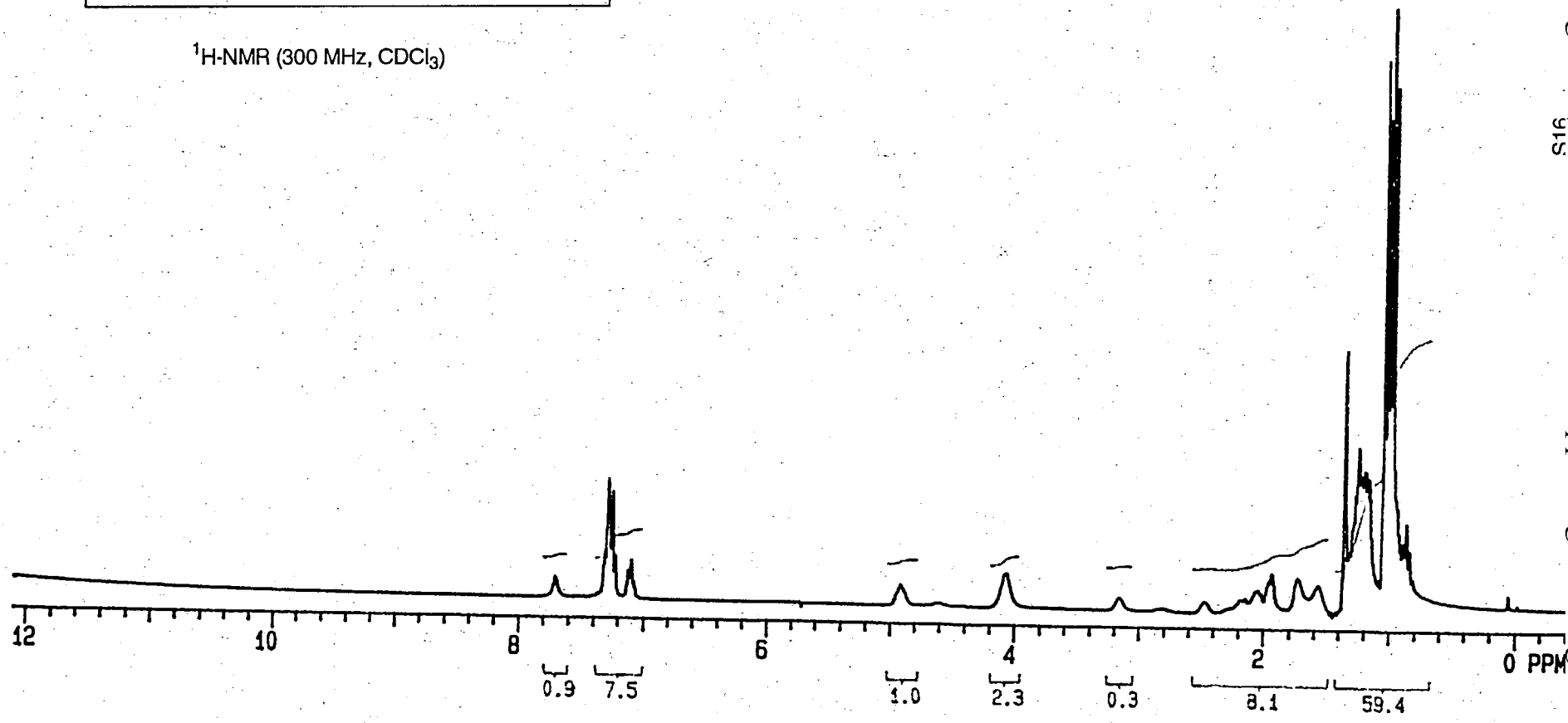
^{1}H-NMR (300 MHz, $CDCl_3$)

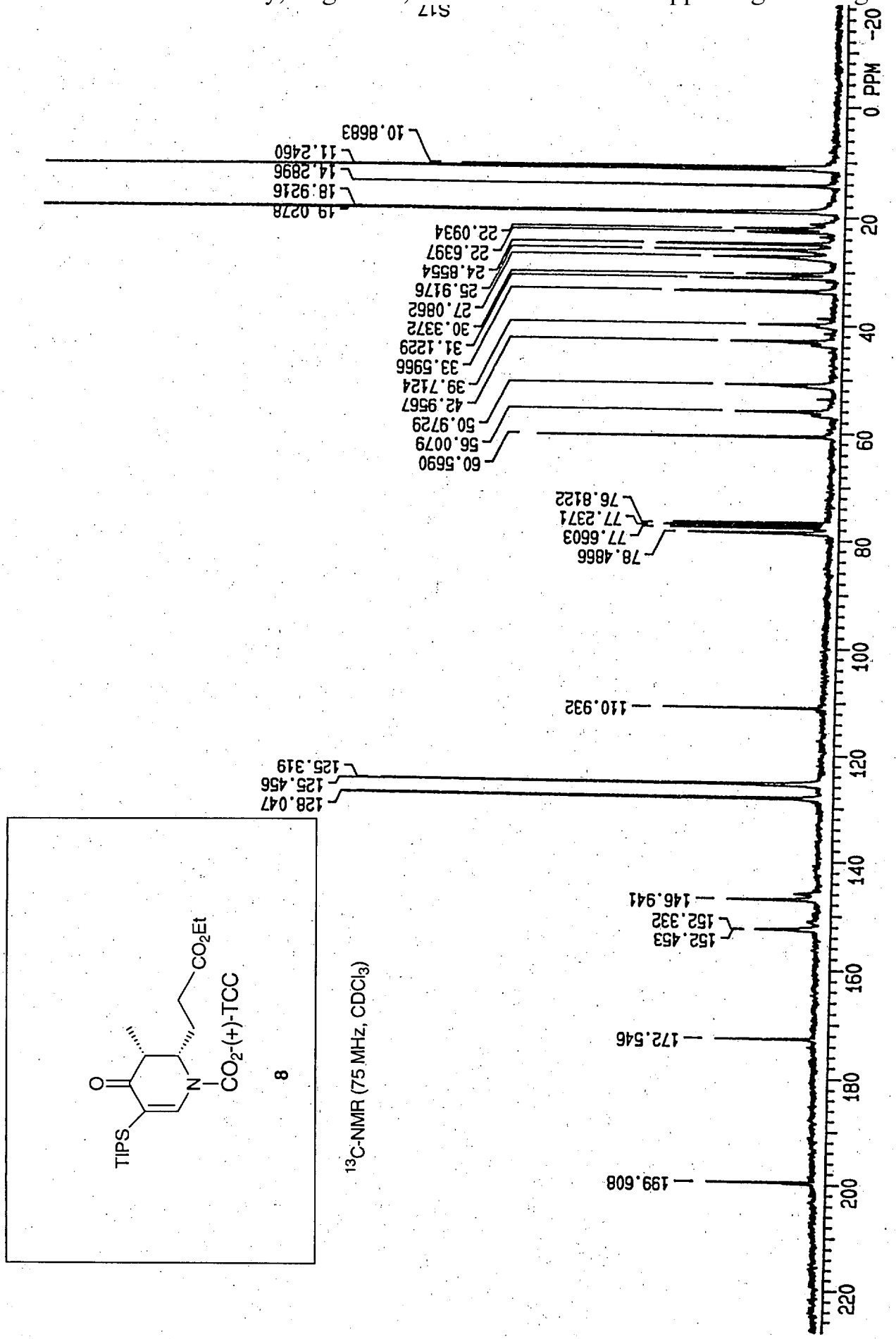


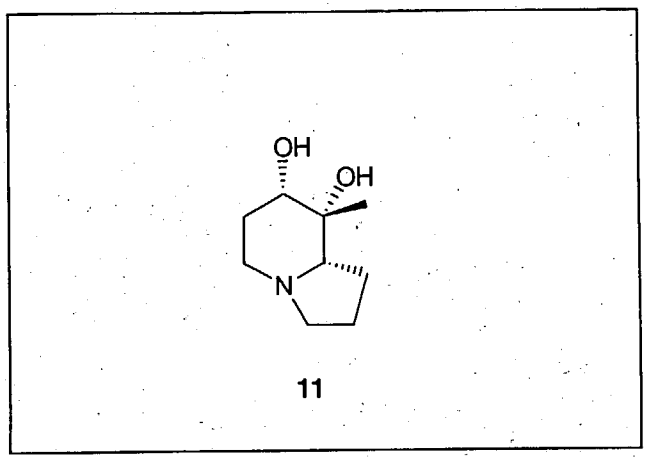


8

^{1}H-NMR (300 MHz, $CDCl_3$)







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^{1}H-NMR (300 MHz, $CDCl_3$)