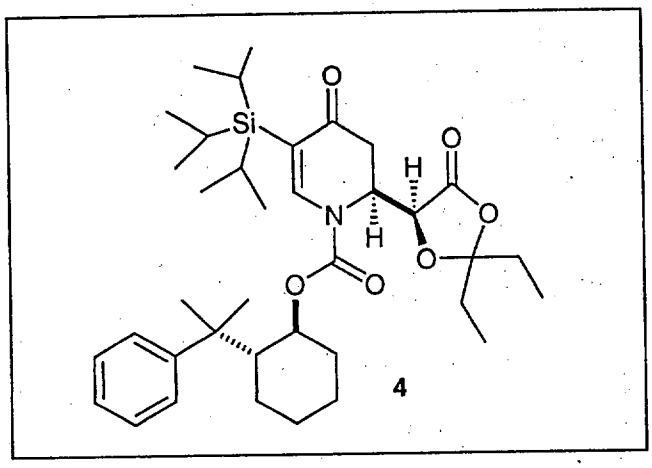
Compound: (2*S*)-2-((5*S*)-2,2-Diethyl-1,3-dioxolan-4-one)-1-[(((1*R*,2*S*)-2-(1-methyl-1-phenylethyl)-cyclohexyl)-oxy)carbonyl]-5-(triisopropylsilyl)-2,3-dihydro-4-pyridone

Compound No: **4**

Notebook Pg(s): I-039Y



4

Formula: $C_{37}H_{57}NO_6Si$

Molecular Weight: 639.9467

Yields(s): 85%

Appearance: white solid

Stability: stable

mp/bp: 141-142 °C

$[\alpha]^{23}_D$ + 17.2 ° (c 0.5, $CHCl_3$)

Elemental Analysis (Atlantic microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	69.44	8.98	2.19	
Found:	69.37	8.92	2.20	

HRMS: Calculated Found

IR (thin film): 2948, 1796, 1718, 1662, 1588, 1236 cm^{-1}

NMR ($CDCl_3$)

^{1}H-NMR (300 MHz) δ 0.80-1.39 (m, 38H), 1.59-2.08 (m, 8H), 2.24 (m, 1H), 2.47 (dd, 1H, *J*=16.4 and 7.2 Hz), 3.35 (m, 1H), 4.15 (m, 1H), 4.95 (m, 1H), 7.12 (m, 1H), 7.30 (m, 4H), 7.76 (s, 1H)

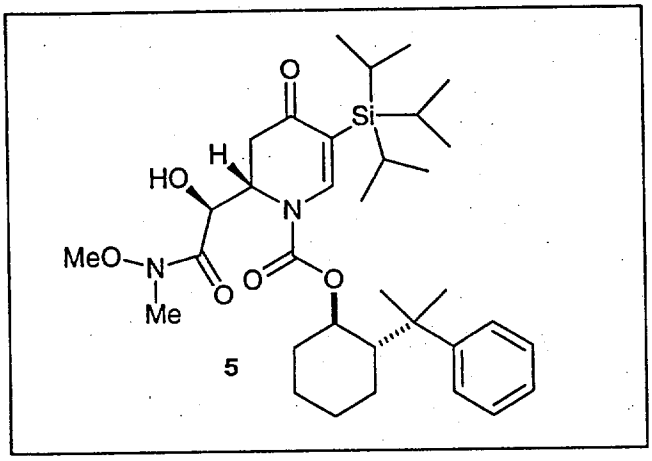
^{13}C-NMR (75 MHz) δ 7.7, 11.4, 19.0, 21.6, 24.9, 25.9, 26.9, 29.6, 30.0, 31.3, 33.6, 37.2, 39.6, 51.3, 52.7, 75.1, 78.5, 110.8, 115.6, 125.3, 128.1, 128.3, 147.8, 151.7, 152.4, 169.9, 194.9

Literature References:

Compound: 2-(2*S*-Hydroxy-*N*-methoxy-*N*-methylacetamide)-1-[(((1*R*,2*S*)-2-(1-methyl-1-phenylethyl)-cyclohexyl)-oxy)carbonyl]-5-(triisopropylsilyl)-2,3-dihydro-4-pyridone

Compound No: **5**

Notebook Pg(s): I-091W



5

Formula: $C_{34}H_{54}N_2O_6Si$

Molecular Weight: 614.897

Yields(s): 97%

Appearance: white solid

Stability: stable

mp/bp: 174-175 °C (ethyl acetate/hexane)

$[\alpha]^{23}_D$ + 18.39 ° (c 0.44, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	66.41	8.85	4.56	
Found:	66.30	8.85	4.58	

HRMS: Calculated Found

IR (thin film): 3439, 2936, 2859, 1714, 1661, 1578, 1385, 1327, 1254 cm^{-1}:

NMR ($CDCl_3$)

^{1}H-NMR (300 MHz) δ 1.02-1.38 (m, 33H), 1.67-2.20 (m, 6H), 3.06 (br s, 1H), 3.24 (s, 3H), 3.68 (s, 3H), 4.33-4.62 (br m, 1H), 4.88 (m, 1H), 7.13 (m, 1H), 7.28 (m, 4H), 7.76 (br s, 1H)

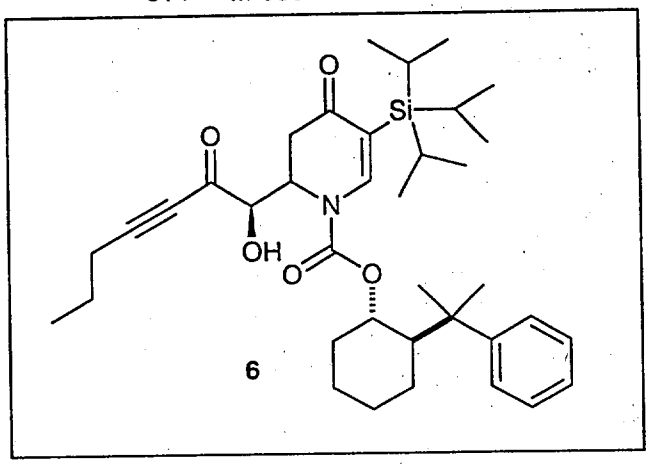
^{13}C-NMR (75 MHz) δ 11.5, 19.2, 22.5, 24.9, 26.0, 27.3, 30.8, 33.9, 36.7, 40.0, 51.3, 54.0, 55.1, 61.7, 69.7, 78.4, 110.6, 125.1, 125.2, 125.6, 128.1, 148.1, 148.6, 152.1, 195.9

Literature References:

Compound Name: (2*R*)-2-((1*R*)-Hydroxy-1-hept-3-yn-2-one)-1-[(((1*S*,2*R*)-2-(1-methyl-1-phenylethyl)-cyclohexyl)-oxy)carbonyl]-5-(triisopropylsilyl)-2,3-dihydro-4-pyridone

Compound No: 6

Notebook Pg(s): III-036W



6

Formula: $C_{37}H_{55}NO_5Si$

Molecular Weight: 621.9315

Yields(s): 96%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -36.57 ° (c 0.99, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 622.3928 $[M + H]^+$ Found: 622.3927 $[M + H]^+$

IR (thin film): 3469, 2935, 2849, 2208, 1719, 1666, 1580, 1463, 1383, 1324, 1255 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 1.02-1.38 (m, 33H), 1.68 (m, 5H), 2.09 (m, 4H), 2.41 (m, 3H), 3.03 (m, 1H), 3.13 (m, 1H), 4.12 (m, 1H), 4.91 (m, 1H), 7.13 (m, 1H), 7.28 (m, 4H), 7.76 (s, 1H)

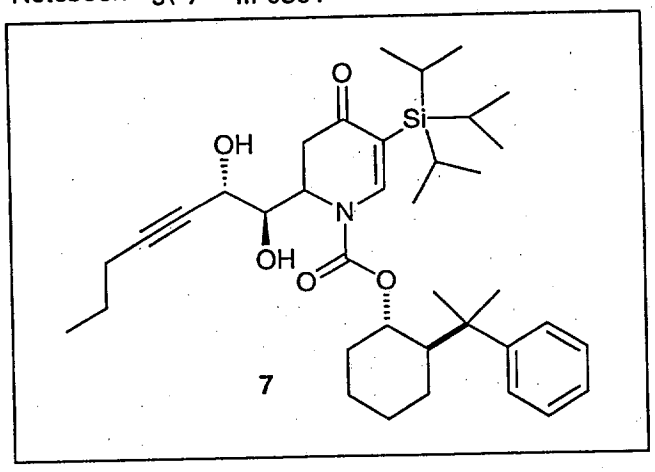
^{13}C-NMR (75 MHz) δ 11.4, 13.8, 19.0, 21.3, 21.6, 24.9, 26.0, 27.0, 31.3, 33.4, 37.4, 39.7, 51.3, 54.5, 78.7, 81.6, 101.2, 111.3, 125.3, 125.6, 128.3, 147.9, 151.8, 152.3, 186.3, 195.6

Literature References:

Compound Name: (2*R*)-2-((1*R*,2*S*)-Dihydroxy-hept-3-yne)-1-[(((1*S*,2*R*)-2-(1-methyl-1-phenylethyl)-cyclohexyl)-oxy)carbonyl]-5-(triisopropylsilyl)-2,3-dihyrdo-4-pyridone

Compound No: 7

Notebook Pg(s): III-036Y



7

Formula: $C_{37}H_{57}NO_5Si$

Molecular Weight: 623.9473

Yields(s): 96%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +49.45 ° (c 0.28, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 624.4084 $[M + H]^+$ Found: 624.4077 $[M + H]^+$

IR (thin film): 3422, 2931,2868, 2241, 1718, 1650, 1577, 1462, 1389, 1326, 1248, 1013 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.97-1.40 (m, 33H), 1.49-1.89 (m, 7H), 1.92-2.52 (m, 7H), 2.87 (m, 1H), 3.49 (m, 1H), 4.14 (m, 1H), 4.89 (m, 1H), 7.15 (m, 1H), 7.30 (m, 4H), 7.74 (s, 1H)

^{13}C-NMR (75 MHz) δ 11.4, 13.8, 19.1, 21.1, 22.2, 24.9, 26.0, 27.2, 29.9, 31.0, 33.7, 37.1, 39.7, 51.2, 52.8, 64.8, 75.1, 78.9, 89.5, 111.3, 125.5, 128.4, 148.2, 152.2, 153.3, 196.8

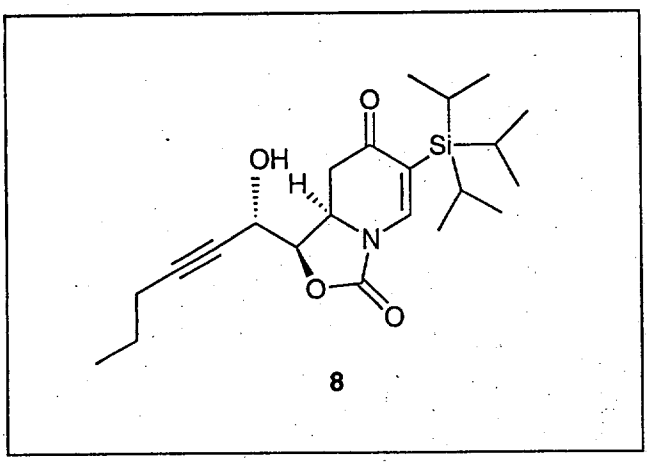
Literature References:

Compound: (1*R*,1'*S*,9*R*)-1-(1'-hydroxyhex-2-ynyl)-1,2,8,8a-tetrahydro-6-triisopropylsilyl-2-oxaindolizine-3,7-dione

Compound No: **8**

Notebook Pg(s): III-040W

Formula: $C_{22}H_{35}NO_4Si$



8

Molecular Weight: 405.6091

Yields(s): 88%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -284.64 ° (c 0.45, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 406.2414 $[M + H]^+$ Found: 406.2421 $[M + H]^+$

IR (neat): 3414, 2938, 2865, 1778, 1659, 1568, 1399, 1266 cm^{-1}

NMR ($CDCl_3$)

^{1}H-NMR (300 MHz) δ 0.98 (t, 3H, *J*=7.3 Hz), 1.06 (m, 18H), 1.33 (m, 3H), 1.52 (m, 2H), 2.19 (t, 2H, *J*=7.1 Hz), 2.53 (d, 1H, *J*=6.8 Hz), 2.71 (dd, 1H, *J*=15.4 and 4.0 Hz), 3.29 (t, 1H, *J*=15.4 Hz), 4.63 (m, 2H), 4.77 (d, 1H, *J*=8.3 Hz), 7.57 (s, 1H)

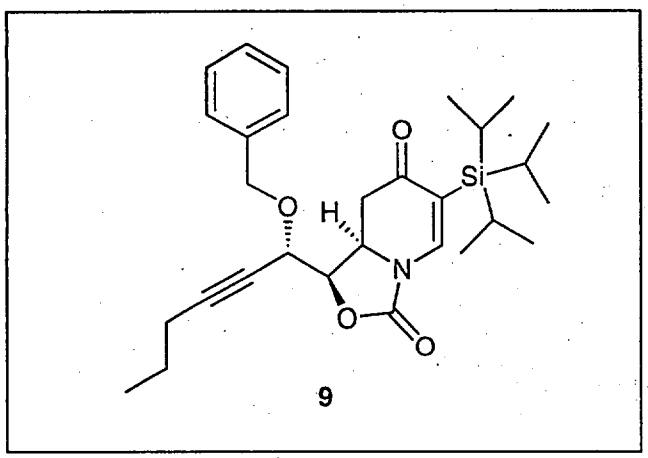
^{13}C-NMR (75 MHz) δ 11.2, 13.8, 18.9, 20.9, 22.0, 37.2, 53.6, 62.7, 75.4, 78.2, 91.1, 112.9, 143.7, 152.1, 195.7

Literature References:

Compound Name: (1*R*,1'*S*,9*R*)-1-((1'-phenylmethoxy)hex-2-ynyl)-1,2,8,8a-tetrahydro-6-triisopropylsilyl-2-oxaindolizine-3,7-dione

Compound No: 9

Notebook Pg(s): III-043W



9

Formula: $C_{29}H_{41}NO_4Si$

Molecular Weight: 495.7335

Yields(s): 80%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -118.10 ° (c 0.21, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 496.2883 $[M + H]^+$ Found: 496.2860 $[M + H]^+$

IR (thin film): 2941, 2864, 2241, 1782, 1659, 1569, 1397, 1265, 1210, 1064 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 1.01 (m, 21H), 1.29 9m, 3H), 1.56 (m, 2H), 2.22 (t, 2H, *J*=7.0 Hz), 2.72 (dd, 1H, *J*=15.4 and 4.2 Hz), 3.32 (t, 1H, *J*=15.4 Hz), 4.55 (m, 3H), 4.74 (dd, 1H, *J*=8.0 and 2.4 Hz), 4.83 (d, 1H, *J*=11.6 Hz), 7.28 (m, 5H), 7.53 (s, 1H)

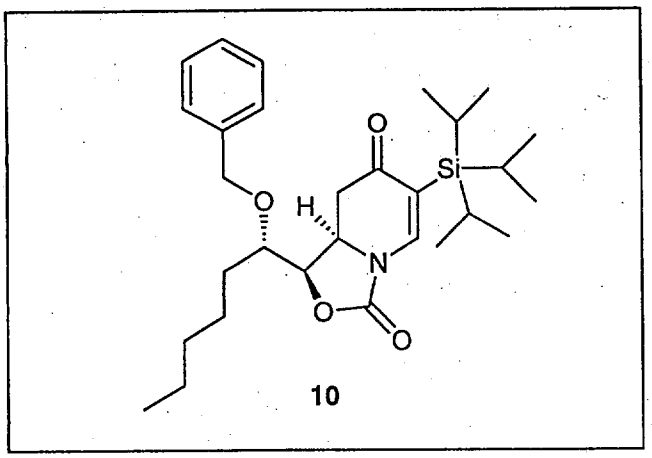
^{13}C-NMR (75 MHz) δ 11.2, 13.8, 18.9, 20.9, 27.0, 38.0, 53.8, 69.0, 71.3, 73.4, 77.4, 91.9, 112.1, 127.8, 128.0, 128.7, 137.0, 143.7, 152.1, 196.1

Literature References:

Compound Name: (1*R*,1'*S*,9*R*)-1-((1'-Phenylmethoxy)hexane)-1,2,8,8a-tetrahydro-6-triisopropylsilyl-2-oxaindolizine-3,7-dione

Compound No: **10**

Notebook Pg(s): III-043Y



10

Formula: $C_{29}H_{45}NO_4Si$

Molecular Weight: 499.7651

Yields(s): 97%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -160.0 ° (c 0.15, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 500.3196 [M + H] Found: 500.3173 [M + H]

IR (thin film): 2942, 2860, 1772, 1659, 1570, 1464, 1397, 1266 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.92 (t, 3H, *J*=6.6 Hz), 1.04 (m, 18H), 1.34 (m, 9H), 1.74 (m, 2H), 2.65 (dd, 1H, *J*=15.0 and 4.2 Hz), 2.97 (t, 1H, *J*=15.0 Hz), 4.49 (d, 1H, *J*=10.5 Hz), 4.60 (m, 4H), 7.31 (m, 5H), 7.55 (s, 1H)

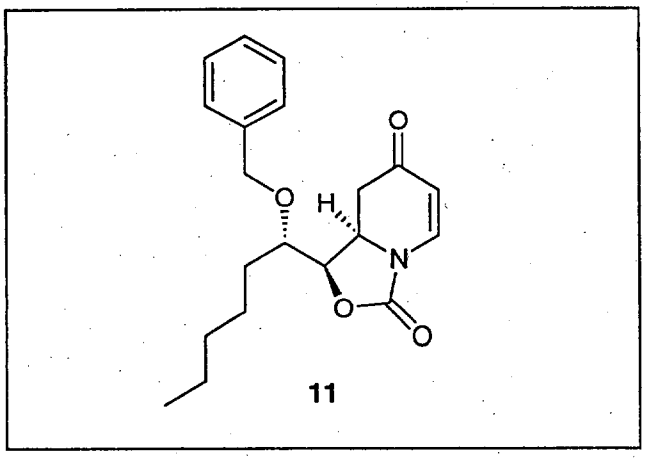
^{13}C-NMR (75 MHz) δ11.3, 14.4, 19.1, 22.9, 24.5, 29.9, 32.2, 28.4, 54.5, 72.6, 78.0, 112.8, 128.1, 128.4, 128.9, 137.6, 144.2, 152.3, 196.3

Literature References:

Compound Name: (1*R*,1'*S*,9*R*)-1-((1'-phenylmethoxy)hexane)-1,2,8,8a-tetrahydro-2-oxaindolizine-3,7-dione

Compound No: **11**

Notebook Pg(s): II-076Y



11

Formula: $C_{20}H_{25}NO_4$

Molecular Weight: 343.4221

Yields(s): 77%

Appearance: white solid

Stability: stable

mp/bp: 68-69 ° C (ethyl acetate/hexane)

$[\alpha]^{23}_D$ -265.88 (c 0.085, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

	%C	%H	%N	%___
Calculated:	69.95	7.34	4.08	
Found:	69.67	7.44	3.97	

HRMS: Calculated Found

IR (thin film): 2933, 2858, 1773, 1666, 1596, 1438, 1359, 1262, 1097 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.91 (t, 3H, *J*=6.4 Hz), 1.35 (m, 6H), 1.73 (m, 2H), 2.69 (dd, 1H, *J*=15.9 and 4.2 Hz), 2.97 (t, 1H, *J*=15.9 Hz), 3.83 (q, 1H, *J*=5.2 Hz), 4.47 (d, 1H, *J*=11.0 Hz), 4.64 (m, 3H), 5.47 (d, 1H, *J*=7.9 Hz), 7.34 (m, 5H), 7.60 (d, 1H, *J*=7.9 Hz)

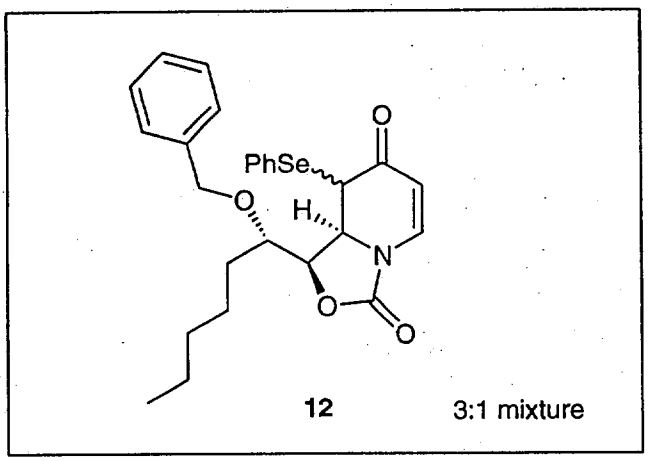
^{13}C-NMR (75 MHz) δ 14.2, 22.7, 24.3, 29.7, 32.1, 37.7, 54.7, 72.3, 77.0, 108.7, 128.1, 128.3, 128.8, 137.4, 138.8, 152.1, 192.3

Literature References:

Compound Name: (1*R*,1'*S*,8*S*,9*R*)-1-((1'-Phenylmethoxy)hexane-1,2,8-tetrahydro-8-phenylselenyl-2-oxaindolizine-3,7-dione

Compound No: **12**

Notebook Pg(s): III-046Y



12 3:1 mixture

Formula: $C_{26}H_{29}NO_4Se$

Molecular Weight: 498.4797

Yields(s): 73%

Appearance: clear oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -206.16 (c 0.095, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 500.1342 $[M + H]^+$ Found: 500.1341 $[M + H]^+$

IR (thin film): 2931, 1783, 1666, 1601, 1437, 1325, 1249, 1090 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.86 (t, 3H, *J*=6.9 Hz), 1.17-1.61 (m, 7H), 1.77 (m, 1H), 3.89 (m, 1H), 4.08 (d, 1H, *J*=15.0 Hz), 4.43 (dd, 1H, *J*=15.0 and 7.3 Hz), 4.55 (d, 1H, *J*=11.4 Hz), 4.60 (d, 1H, *J*=11.4 Hz), 4.91 (dd, 1H, *J*=7.3 and 2.1 Hz), 5.47 (d, 1H, *J*=7.9 Hz), 7.33 (m, 8 H), 7.48 (d, 1H, *J*=7.9 Hz), 7.56 (d, 2H, *J*=7.5 Hz)

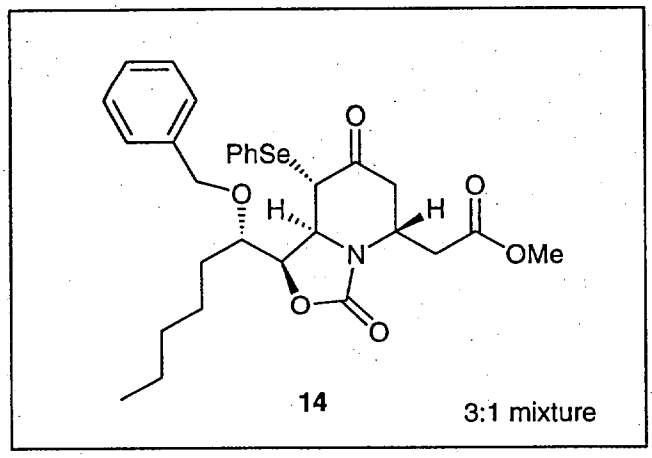
^{13}C-NMR (75 MHz) δ 14.1, 22.7, 25.3, 30.6, 31.8, 44.3, 58.0, 72.7, 78.2, 79.0, 107.2, 125.6, 128.2, 128.7, 129.5, 129.6, 135.8, 136.4, 137.5, 138.3, 152.2, 188.5

Literature References:

Compound Name: (1*R*,1'*S*,5*R*,8*S*,9*S*)-1-((1'-phenylmethoxy)hexane)-8-phenylselenyl-1,2,5,6,6a, 8-hexahydro-2-oxaindolizinel-3,5-dione acetic acid methyl ester

Compound No: **14**

Notebook Pg(s): III-047W



14 3:1 mixture

Formula: $C_{29}H_{35}NO_6Se$

Molecular Weight: 572.5589

Yields(s): 100%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +66.67 (c 0.075, $CHCl_3$)

NMR for major diastereomer

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 574.1710 $[M + H]^+$ Found: 574.1721 $[M + H]^+$

IR (thin film): 2935, 1761, 1732, 1402, 1213, 1074, 1019 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.89 (t, 3H, *J*=6.5 Hz), 1.29 (m, 3H), 1.44 (m, 3H), 1.87 (m, 2H), 2.44 (m, 2H), 2.56 (m, 2H), 3.65 (s, 3H), 3.96 (m, 1H), 4.10 (d, 1H, *J*=9.6 Hz), 4.35 (m, 1H), 4.60 (m, 4H), 7.29 (m, 8H), 7.53 (d, 2H, *J*=7.5 Hz)

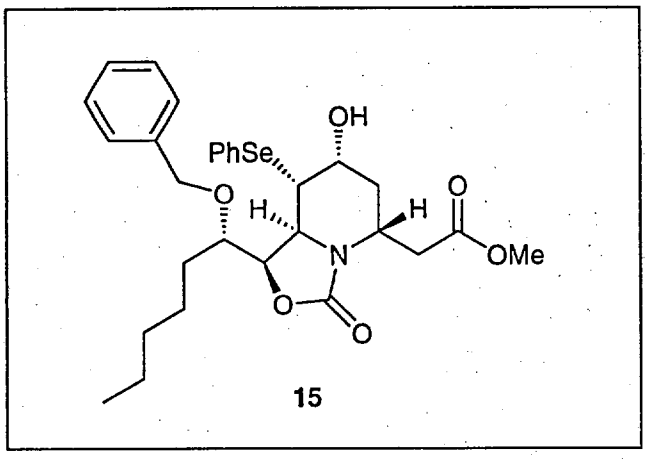
^{13}C-NMR (75 MHz) δ 14.2, 22.8, 24.2, 30.1, 32.0, 37.9, 41.3, 45.3, 47.8, 52.1, 58.1, 71.8, 77.4, 128.0, 128.2, 128.6, 129.6, 129.8, 135.1, 136.7, 137.9, 156.0, 170.4, 202.4

Literature References:

Compound Name: (1*R*,1'*S*,5*R*,7*R*,8*S*,9*S*)-7-Hydroxy-1-((1'-phenylmethoxy)hexane)-8-phenylselenyl-1,2,5,6,6a,7,8-hexahydro-2-oxaindolizine-3,5-dione acetic acid methyl ester

Compound No: **15**

Notebook Pg(s): III-049Wa



15

Formula: $C_{29}H_{37}NO_6Se$

Molecular Weight: 574.5747

Yields(s): 65% single diastereomer

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -56.52 (c 0.115, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 576.1867 $[M + H]^+$ Found: 576.1876 $[M + H]^+$

IR (thin film): 3434, 2936, 2858, 1751, 1736, 1405, 1331, 1253, 1051 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.83 (t, 3H, *J*=7.0 Hz), 1.21 (m, 5H), 1.51 (m, 2H), 1.72 (m, 1H), 1.83 (m, 1H), 2.22 (dd, 1H, *J*=14.6 and 2.4 Hz), 2.89 (m, 3H), 3.18 (d, 1H, *J*=12.0 Hz), 3.67 (s, 3H), 3.90 (m, 1H), 4.09 (m, 1H), 4.17 (dd, 1H, *J*=12.0 and 8.0 Hz), 4.35 (m, 1H), 4.55 (d, 1H, *J*=11.5 Hz), 4.67 (d, 1H, *J*=11.5 Hz), 4.79 (dd, 1H, *J*=8.0 and 3.5 Hz), 7.33 (m, 8H), 7.45 (d, 2H, *J*=7.4 Hz)

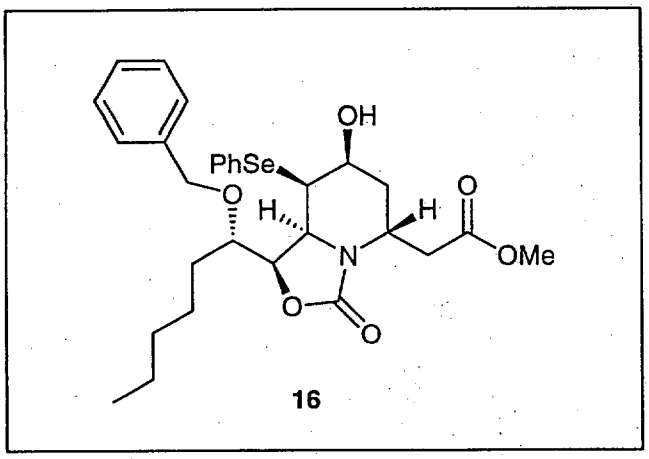
^{13}C-NMR (75 MHz) δ 14.2, 22.9, 24.6, 29.6, 32.0, 32.2, 37.3, 45.2, 47.5, 51.8, 52.0, 65.9, 70.8, 125.9, 128.0, 128.2, 128.6, 129.2, 130.0, 135.1, 138.3, 156.5, 171.9

Literature References:

Compound Name: (1*R*,1'*S*,5*R*,7*S*,8*R*,9*S*)-7-Hydroxy-1-((1'-phenylmethoxy)hexane)-8-phenylselenyl-1,2,5,6,6a,7,8-hexahydro-2-oxaindolizine-3,5-dione acetic acid methyl ester

Compound No: **16**

Notebook Pg(s): III-049Wb



16

Formula: $C_{29}H_{37}NO_6Se$

Molecular Weight: 574.5747

Yields(s): 20% single diastereomer

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ -31.54 (c 0.13, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 567.1867 $[M + H]^+$ Found: 567.1873 $[M + H]^+$

IR (thin film): 3430, 2938, 2860, 1755, 1732, 1400, 1338, 1254 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.92 (t, 3H, *J*=6.9 Hz), 1.32 (m, 6H), 1.77 (m, 3H), 1.91 (dd, 1H, *J*=14.0 and 4.3 Hz), 2.62 (d, 2H, *J*=8.1 Hz), 2.67 (s, 1H), 3.05 (d, 1H, *J*=10.8 Hz), 3.64 (m, 1H), 3.73 (s, 3H), 3.91 (m, 1H), 4.08 (d, 1H, *J*=10.8 Hz), 4.12 (m, 1H), 4.29 (dt, 1H, *J*=10.2 and 3.6 Hz), 4.50 (dd, 1H, *J*=10.2 and 7.2 Hz), 4.61 (m, 1H), 7.01 (m, 2H), 7.33 (m, 6H), 7.64 (m, 2H)

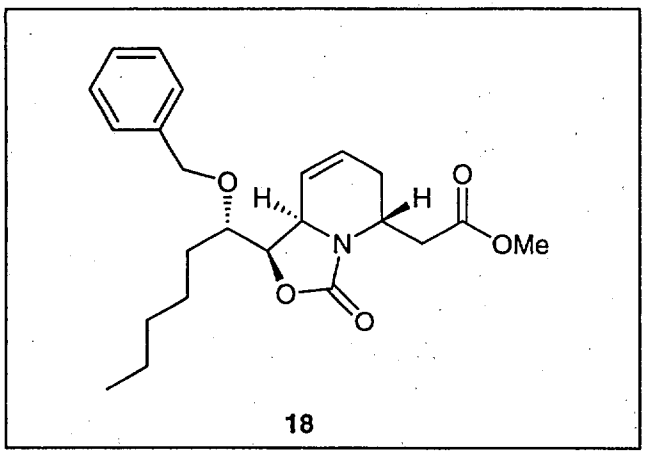
^{13}C-NMR (75 MHz) δ 14.2, 22.6, 22.7, 29.8, 32.3, 35.3, 38.1, 45.9, 52.2, 56.8, 57.5, 66.3, 69.7, 75.1, 76.4, 127.3, 127.8, 128.1, 128.5, 129.9, 138.1, 155.9, 170.4

Literature References:

Compound Name: (1*R*,1'*S*,5*R*,9*R*)-3-Oxo-1-((1'-phenylmethoxy)hexane)-1,5,6,8a-tetrahydro-oxazolo[3,4-*a*]pyridin-5-yl acetic acid methyl ester

Compound No: **18**

Notebook Pg(s): III-052W



18

Formula: $C_{23}H_{31}NO_5$

Molecular Weight: 401.5019

Yields(s): 95%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +27.29 (c 1.40, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 402.2280 [M + H] Found: 402.2286 [M + H]

IR (thin film): 2931, 2860, 1760, 1736, 1413, 1372, 1219, 1072 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.89 (t, 3H, *J*=6.3 Hz), 1.32 (m, 3H), 1.45 (m, 2H), 1.78 (m, 2H), 1.93 (dd, 1H, *J*=18.3 and 3.6 Hz), 2.59 (m, 4H), 3.64 (m, 1H), 3.70 (s, 3H), 4.38 (d, 1H, *J*=11.0 Hz), 4.46 (m, 1H), 4.54 (m, 2H), 4.63 (d, 1H, *J*=11.0 Hz), 5.74 (m, 1H), 5.84 (m, 1H), 7.33 (m, 5H)

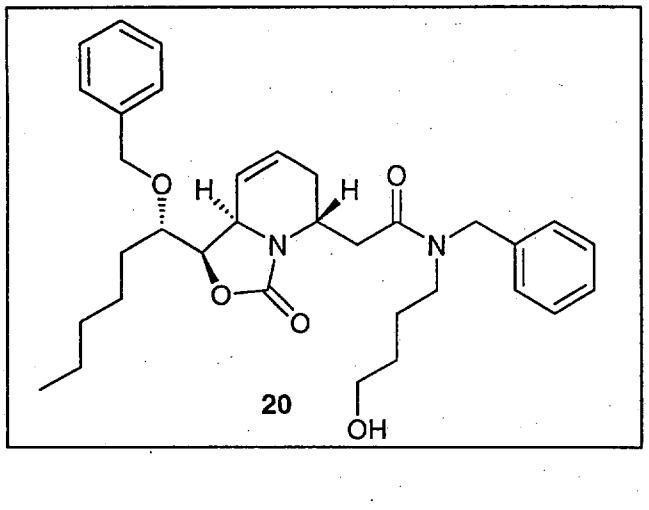
^{13}C-NMR (75 MHz) δ 14.2, 22.8, 23.0, 27.3, 29.1, 32.3, 37.1, 45.8, 52.2, 52.9, 70.6, 76.6, 123.2, 126.8, 127.9, 128.1, 128.7, 138.0, 157.2, 171.1

Literature References:

Compound Name: (1*R*,1'*S*,5*R*,9*R*)-*N*-Benzyl-2-[3-oxo-1-((1'-phenylmethoxy)hexane)-1,5,6,8a-tetrahydro-oxazolo[3,4-*a*]pyridin-5-yl]-*N*-4-hydroxy-butyl) acetamide

Compound No:

Notebook Pg(s): **20**



20

Formula: $C_{33}H_{44}N_2O_5$

Molecular Weight: 548.7216

Yields(s): 82%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +26.82 (c 1.70, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 549.3328 $[M + H]^+$ Found: 549.3345 $[M + H]^+$

IR (thin film): 3448, 2930, 2967, 1749, 1637, 1452, 1417, 1376, 1223, 1070 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.88 (m, 3H), 1.29-1.75 (m, 15 H), 2.05 (m, 1H), 2.6 (m, 3H), 3.25 (m, 1H), 3.62 (m, 3H), 4.38 (m, 2H), 4.57 (m, 4H), 5.78 (m, 2H), 7.30 (m, 10)

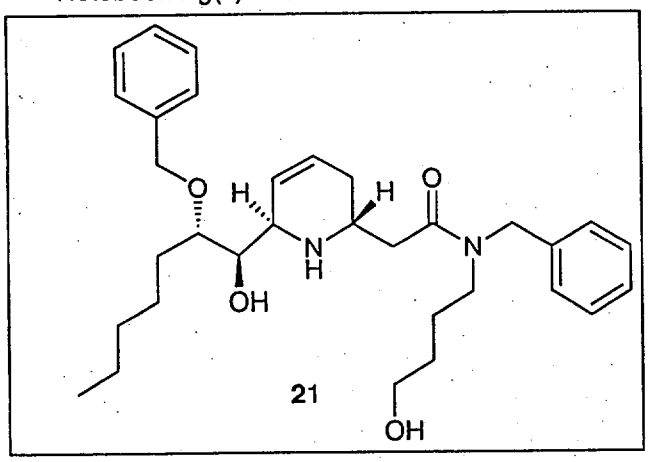
^{13}C-NMR (75 MHz) δ 14.2, 22.8 and 23.0 (due to rotamers), 24.1, 25.3, 27.4 and 27.6 (due to rotamers), 29.0, 29.7 and 29.8 (due to rotamers), 32.3, 36.0 and 36.2 (due to rotamers), 45.8 and 46.0 (due to rotamers), 47.3, 48.4, 51.4, 53.0 and 53.1 (due to rotamers), 62.2 and 62.6 (due to rotamers), 70.6, 76.6, 123.0, 126.4, 126.6, 127.0, 127.6, 127.9, 128.0, 128.1, 128.7, 128.8, 129.2, 136.7, 137.7 and 138.0 (due to rotamers), 157.0, 169.9 and 170.4 (due to rotamers)

Literature References:

Compound Name: (1'*R*,2*R*,2'*S*,6*R*)-*N*-Benzyl-2-[6-(2'-phenylmethoxy-1'-hydroxy-heptyl)-1,2,3,6-tetrahydro-pyridin-2-yl]-*N*-(4-hydroxy-butyl)-acetamide

Compound No: **21**

Notebook Pg(s): JTK III-098Y



21

Formula: $C_{32}H_{46}N_2O_4$

Molecular Weight: 522.727

Yields(s): 88%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +36.78 (c 1.15, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 523.3536 $[M + H]^+$ Found: 523.3560 $[M + H]^+$

IR (thin film): 3401, 2920, 2856, 1627, 1450, 1359, 733, 698 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.88 (m, 3H), 1.26-1.72 (m, 13H), 1.92-2.80 (m, 4H), 3.21 (t, 1H, J = 7.7 Hz), 3.42 (m, 1H), 3.61 (m, 3H), 3.84 (m, 5H), 4.56 (m, 4H), 5.78 (m, 2H), 7.25 (m, 10H)

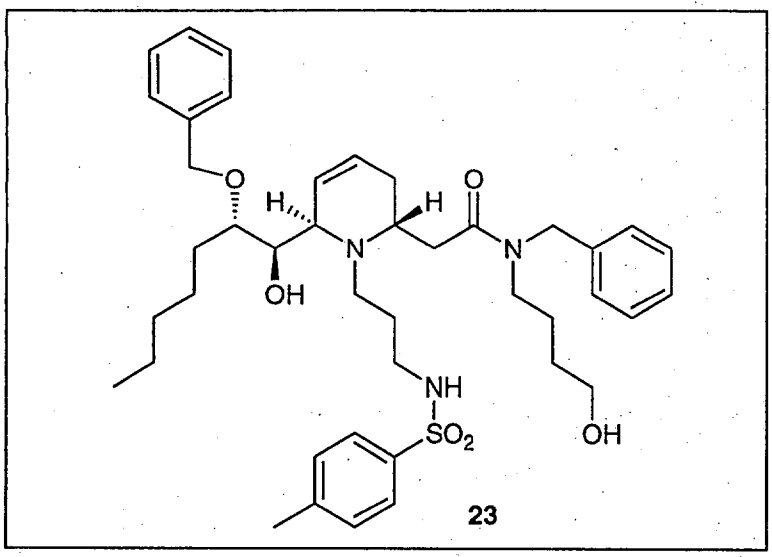
^{13}C-NMR (75 MHz) δ 14.3, 22.9, 24.3 and 24.7 (due to rotamers), 25.2, 29.4, 29.9 and 30.5 (due to rotamers), 32.4, 36.6 and 36.8 (due to rotamers), 46.0, 46.9 and 47.1 (due to rotamers), 48.5, 51.2, 52.1 and 52.3 (due to rotamers), 62.2 and 62.5 (due to rotamers), 71.7 and 71.9 (due to rotamers), 74.5, 79.7, 125.9, 126.4, 126.7, 127.9, 128.1, 128.6, 128.8, 129.2, 136.8 and 137.9 (due to rotamers), 138.3, 171.8 and 172.4 (due to rotamers)

Literature References:

Compound Name: (1'*R*,2*R*,2'*S*,6*R*)-*N*-Benzyl-2-{6-(2'-phenylmethoxy-1'-hydroxy-heptyl)-1-[3-(toluene-4-sulfonylamino)-propyl]-1,2,3,6-tetrahydro-pyridin-2-yl}-*N*-(4-hydroxy-butyl)-acetamide

Compound No: **23**

Notebook Pg(s): JTK IV-018Y



23

Formula: $C_{42}H_{59}N_3O_6S$

Molecular Weight: 734.0115

Yields(s): 84%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +41.50 (c 0.40, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 734.4203 $[M + H]^+$ Found: 734.4237 $[M + H]^+$

IR (thin film): 3436, 3271, 2931, 2860, 1619, 1454, 1325, 1155, 1090 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.89 (m, 3H), 1.30-2.08 (m, 17H), 2.43 (m, 8H), 2.95 (m, 2H), 3.00-3.65 (m, 6H), 3.79 (m, 2H), 4.54 (m, 4H), 5.61 (m, 1H), 5.91 (m, 1H), 6.05 (m, 1H), 7.29 (m, 12H), 7.34 (m, 2H)

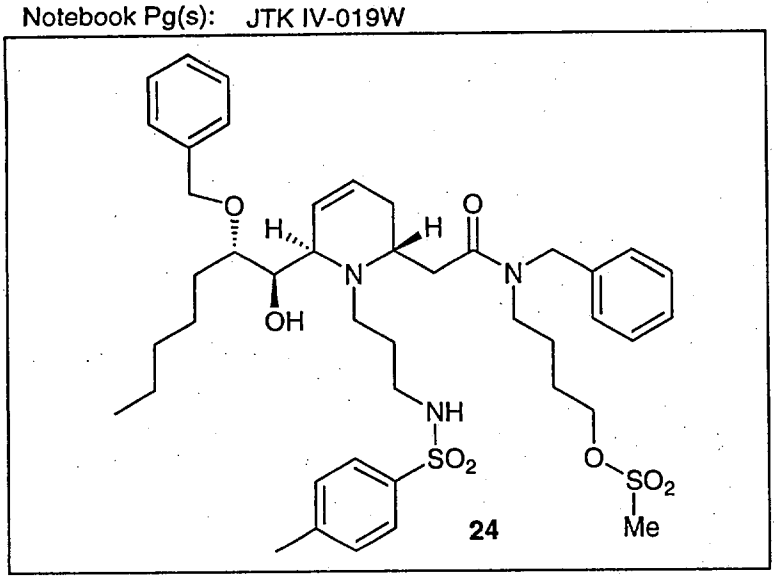
^{13}C-NMR (75 MHz) δ 14.3, 21.7, 22.9, 24.2 and 24.5 (due to rotamers), 25.3, 27.2 and 27.4 (due to rotamers), 29.6 and 30.2 (due to rotamers), 29.8, 32.4, 32.2 and 33.3 (due to rotamers), 41.3 and 41.5 (due to rotamers), 45.0 and 45.1 (due to rotamers), 46.4, 47.4, 48.7, 51.4, 58.8 and 58.9 (due to rotamers), 62.2 and 62.4 (due to rotamers), 71.5, 73.0 and 73.6 (due to rotamers), 79.8 and 80.1 (due to rotamers), 124.8 and 124.9 (due to rotamers), 126.4, 127.3, 127.5, 127.8, 127.9, 128.1, 128.6, 129.2, 129.8, 136.9 and 137.2 (due to rotamers), 137.4 and 137.9 (due to rotamers), 138.7, 143.2 and 143.3 (due to rotamers), 171.9 and 172.8 (due to rotamers)

Literature References:

Compound Name: (1'*R*,2*R*,2'*S*,6*R*)-Methanesulfonic acid 4-[benzyl-({6-(2'-phenylmethoxy-1'-hydroxy-heptyl)-1-[3-(toluene-4-sulfonylamino)-propyl]-1,2,3,6-tetrahydro-pyridin-2-yl}-acetyl)-amino]-butyl ester

Compound No: **24**

Notebook Pg(s): JTK IV-019W



24

Formula: $C_{43}H_{61}N_3O_8S_2$

Molecular Weight: 812.0919

Yields(s): 95%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +59.00 (c 0.20, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 812.3978 $[M + H]^+$ Found: 812.3981 $[M + H]^+$

IR (thin film): 3283, 2928, 2869, 1625, 1453, 1352, 1329, 1160, 1093, 937, 815 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.89 (m, 3H), 1.30 (m, 4H), 1.48 (m, 2H), 1.70 (m, 8H), 2.01 (m, 3H), 2.43 (m, 7H), 3.05 (m, 5H), 3.27 (m, 2H), 3.48 (m, 3H), 3.81 (m, 2H), 4.23 (m, 2H), 4.57 (m, 4H), 5.76 (m, 2H), 7.28 (m, 12H), 7.75 (m, 2H)

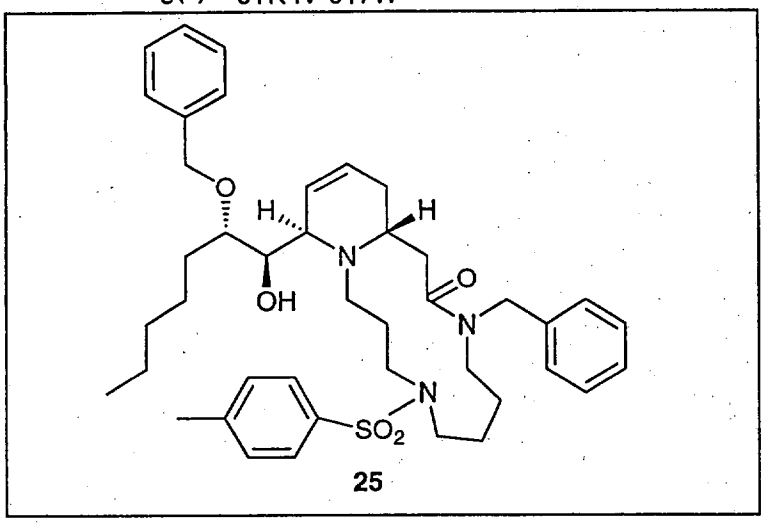
^{13}C-NMR (75 MHz) δ 14.3, 21.5, 22.7, 23.6 and 24.3 (due to rotamers), 24.6 and 24.8 (due to rotamers), 26.5 and 26.6 (due to rotamers), 27.2 and 27.5 (due to rotamers), 28.3, 29.8 and 30.1 (due to rotamers), 32.0 and 32.3 (due to rotamers), 37.3, 41.4 and 41.6 (due to rotamers), 44.5 and 45.2 (due to rotamers), 45.8 and 46.6 (due to rotamers), 48.6, 50.5, 50.9 and 51.2 (due to rotamers), 56.6 and 58.7 (due to rotamers), 69.4 and 69.8 (due to rotamers), 71.3 and 71.4 (due to rotamers), 72.7 and 73.5 (due to rotamers), 79.5 and 79.9 (due to rotamers), 125.0, 126.4, 127.3, 127.6, 127.8, 127.9, 128.2, 128.5, 128.8, 129.2, 129.8, 136.8 and 137.1 (due to rotamers), 137.4 and 137.9 (due to rotamers), 138.7 and 138.8 (due to rotamers), 143.2, 171.9 and 172.8 (due to rotamers)

Literature References:

Compound Name: (1'*R*,4*R*,2'*S*,15a*R*)-13-Benzyl-4-(2'-phenylmethoxy-1'-hydroxy-heptyl)-8-(toluene-4-sulfonyl)-1, 4,6,7,8,9,10,11,12,13,15,15a-dodecahydro-5*H*-4a,8,13-triaza-benzocyclotridecen-14-one

Compound No: **25**

Notebook Pg(s): JTK IV-017W



25

Formula: $C_{42}H_{57}N_3O_5S$

Molecular Weight: 715.9963

Yields(s): 70%

Appearance: colorless oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +24.89 (c 0.225, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated: 716.4097 $[M + H]^+$ Found: 716.4111 $[M + H]^+$

IR (thin film): 3394, 2922, 2860, 1635, 1451, 1338, 1158, 1092 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (300 MHz) δ 0.87 (m, 3H), 1.27 (m, 6H), 1.60 (m, 7H), 1.70-2.17 (m, 5H), 2.43 (s, 3H), 2.55 (m,1H), 2.74 (m, 1H), 2.85-3.18 (m, 4H), 3.53 (m,3 H), 3.76 (m, 1H), 4.00 (m, 3H), 4.60 (m, 3H), 5.25 (d, 1H, J = 14.7 Hz), 5.77 (m, 1H), 5.96 (m, 1H), 7.29 (m, 12H), 7.66 (m, 2H)

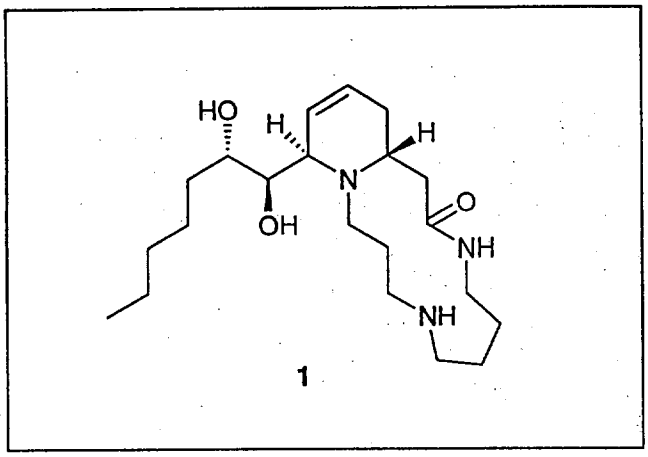
^{13}C-NMR (75 MHz) δ 14.3 and 14.4 (due to rotamers), 21.7, 22.9, 23.9 and 24.4 (due to rotamers), 24.9 and 25.2 (due to rotamers), 25.8, 26.6, 28.3 and 28.9 (due to rotamers), 29.6 and 29.9 (due to rotamers), 31.7 and 32.3 (due to rotamers), 34.6 and 35.4 (due to rotamers), 43.0 and 43.4 (due to rotamers), 46.9 and 47.0 (due to rotamers), 49.4, 50.8 and 51.0 (due to rotamers), 51.6 and 52.2 (due to rotamers), 60.1, 71.3 and 71.5 (due to rotamers), 74.4, 79.9, 126.3 and 126.5 (due to rotamers), 127.3 and 127.5 (due to rotamers), 127.7 and 127.8 (due to rotamers), 127.9 and 128.0 (due to rotamers), 128.2, 128.4, 128.7, 129.1, 129.9, 135.3, 138.0, 139.3, 143.6, and 172.5

Literature References:

Compound Name: (+)-Cannabisativine

Compound No: **1**

Notebook Pg(s): JTK IV-023W



1

Formula: $C_{21}H_{39}N_3O_3$

Molecular Weight: 381.5583

Yields(s): 76%

Appearance: colorless solid

Stability: stable

mp/bp: 165-166 °C (Lit. 167-168 °C)

$[\alpha]^{23}_D$ +51.76 ° (c 0.425, $CHCl_3$)
lit. (+55.1 ° (c 0.53, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs, Inc.)

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

HRMS: Calculated Found

IR (thin film): 3300, 2995, 1635, 1540, 1260 cm^{-1}

NMR ($CDCl_3$):

^{1}H-NMR (500 MHz) δ 0.88 (t, 3H, *J* = 6.4 Hz), 1.30 (s, 6H), 1.42 (m, 2H), 1.50-1.65 (m, 4H), 1.76-1.96 (,. 6H), 2.13 (dd, 1H, *J* = 13.6 and 2.0 Hz), 2.36 (s, 1H), 2.46 (t, 2H, *J* = 12.5 Hz), 2.63 (t, 2H, *J* = 11.2 Hz), 2.73-2.82 (m, 2H), 2.91 (t, 1H, *J* = 12.3 Hz), 3.05 (t, 1H, *J* = 9.0 Hz), 3.17 (s, 1H), 3.49 (t, 2H, *J* = 8.0 Hz), 3.56 (t, 1H, *J* = 7.0 Hz), 4.88 (br s, 1H), 5.84 (dt, 1H, *J* = 10.0 and 1.0 Hz), 5.94 (dd, 1H, *J* = 10.0 and 4.5 Hz), 9.66 (br s, 1H)

^{13}C-NMR (75 MHz) δ 14.4, 23.0, 25.4, 26.4, 27.6, 28.1, 29.9, 32.4, 40.0, 40.1, 42.1, 47.7, 50.8, 52.5, 61.5, 75.0, 75.8, 125.7, 127.4, 171.3

Literature References: Lotter, H. L.; Abraham, D. J.; Turner, C. E.; Knapp, J. E.; Schiff, P. L.; Slatkin, D. J. *Tetrahedron Lett.* **1975**, 2815.

500 MHz ^{1}H NMR for (+)-Cannabisativine

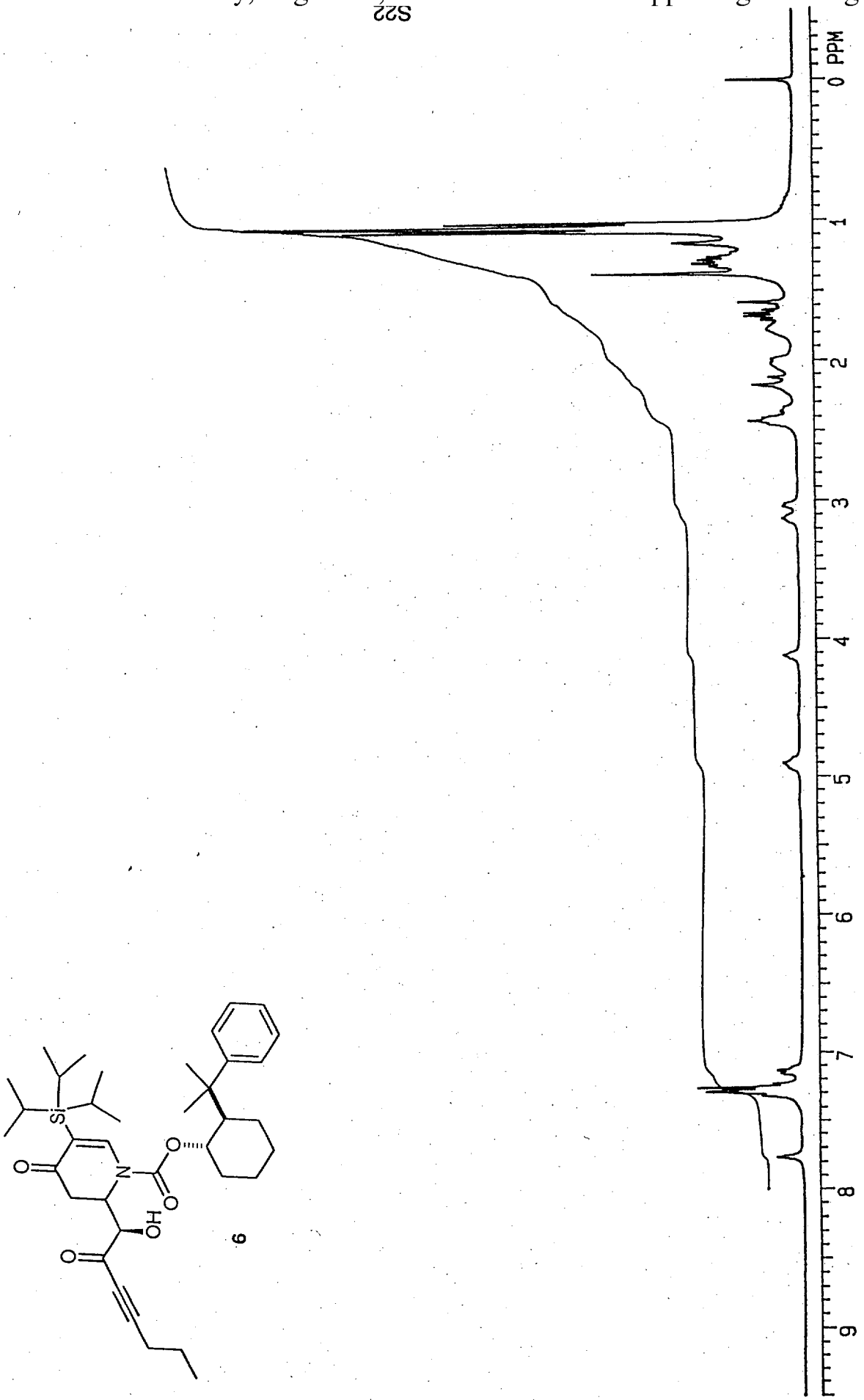
Literature*	Our Synthetic 1
0.88 (t, 3H, J = 6.0 Hz)	0.88 (t, 3H, J = 6.4 Hz)
1.30 (s, 6H)	1.30 (s, 6H)
1.42 (m, 2H)	1.42 (m, 2H)
1.50-1.65 (m, 4H)	1.50-1.65 (m, 4H)
1.75-2.00 (m, 6H)	1.76-1.96 (m, 6H)
2.19 (dd, 1H, J = 13.0 and 3.0 Hz)	2.13 (dd, 1H, J = 13.6 and 2.0Hz)
2.36 (s, 1H)	2.36 (s, 1H)
2.46 (t, 2H, J = 11.0 Hz)	2.46 (t, 2H, J = 12.5 Hz)
2.64 (t, 2H, J = 10.0 Hz)	2.63 (t, 2H, J = 11.2 Hz)
2.75-2.85 (m, 2H)	2.73-2.82 (m, 2H)
2.90 (t, 1H, J = 11.0 Hz)	2.91 (t, 1H, J = 12.3 Hz)
3.03 (t, 1H, J = 8.0 Hz)	3.05 (t, 1H, J = 9.0 Hz)
3.19 (s, 1H)	3.17 (s, 1H)
3.51 (t, 2H, J = 7.0 Hz)	3.49 (t, 2H, J = 8.0 Hz)
3.58 (t, 1H, J = 6.5 Hz)	3.56 (t, 1H, J = 7.0 Hz)
4.93 (br s, 1H)	4.88 (br s, 1H)
5.85 (dt, 1H, J = 6.0 and 1.0 Hz)	5.84 (dt, 1H, J = 10.0 and 1.0 Hz)
5.93 (dd, 1H, J = 6.0 and 3.0 Hz)	5.94 (dd, 1H, J = 10.0 and 4.5 Hz)
9.66 (br s, 1H)	9.66 (br s, 1H)

* Wasserman, H. H.; Leadbetter, M. R. *Tetrahedron Lett.* **1985**, *26*, 2241. The 500 MHz NMR data was kindly provided by Professor Wasserman.

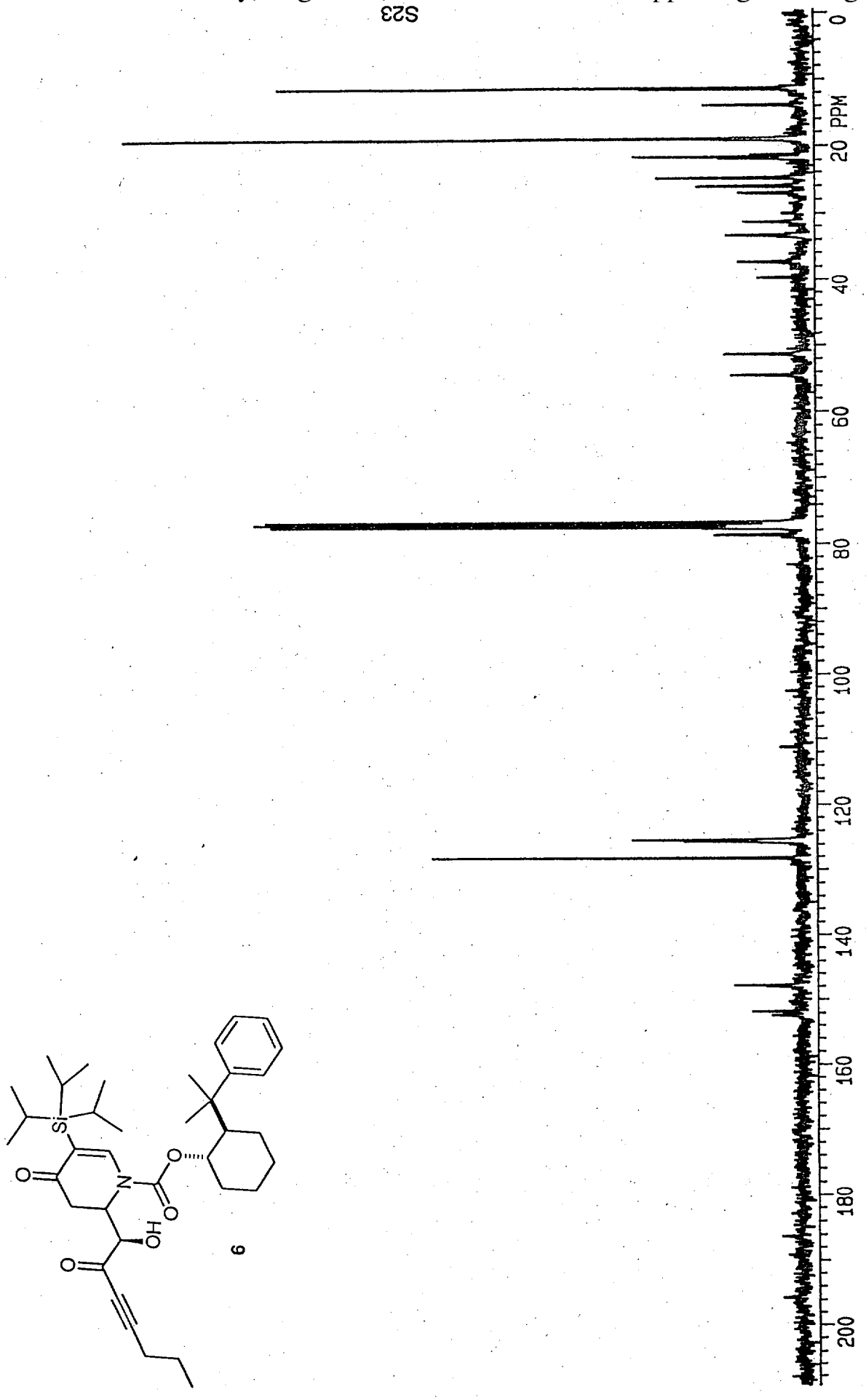
^{13}C NMR for (+)-Cannabisativine

Literature*	Our Synthetic 1
14.4	14.4
23.0	23.0
24.4	25.4
25.6	26.4
25.8	27.6
26.1	28.1
27.3	29.9
29.9	32.4
32.3	32.9
33.6	40.0
38.5	40.1
39.9	42.1
43.6	47.7
48.6	50.8
49.3	52.5
51.9	61.5
59.5	75.0
74.1	75.8
123.4	125.7
128.2	127.4
170.1	171.3

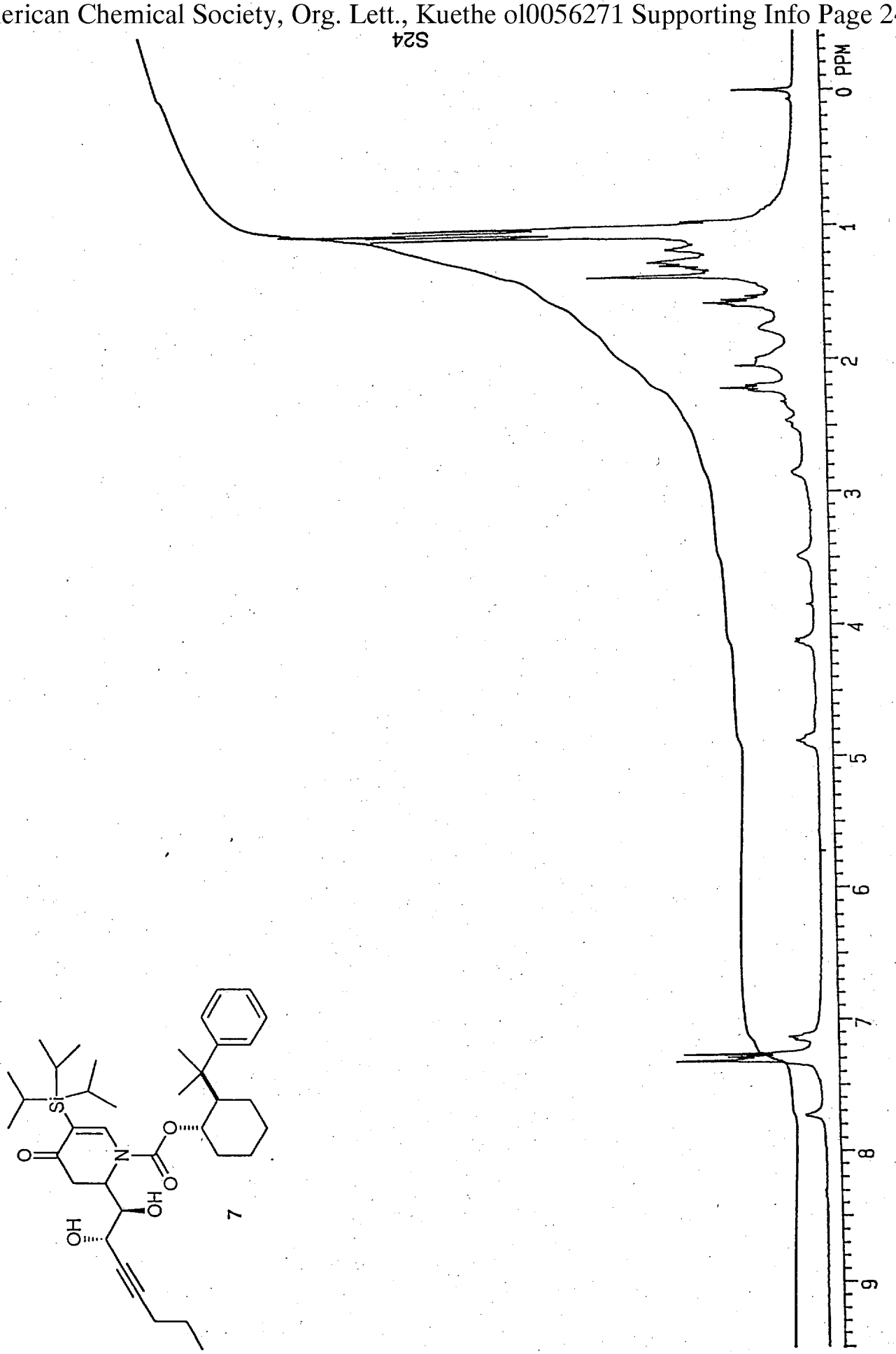
* Ogawa, M.; Kuriya, N.; Natsume, M. *Tetrahedron Lett.* **1984**, *25*, 969.



9

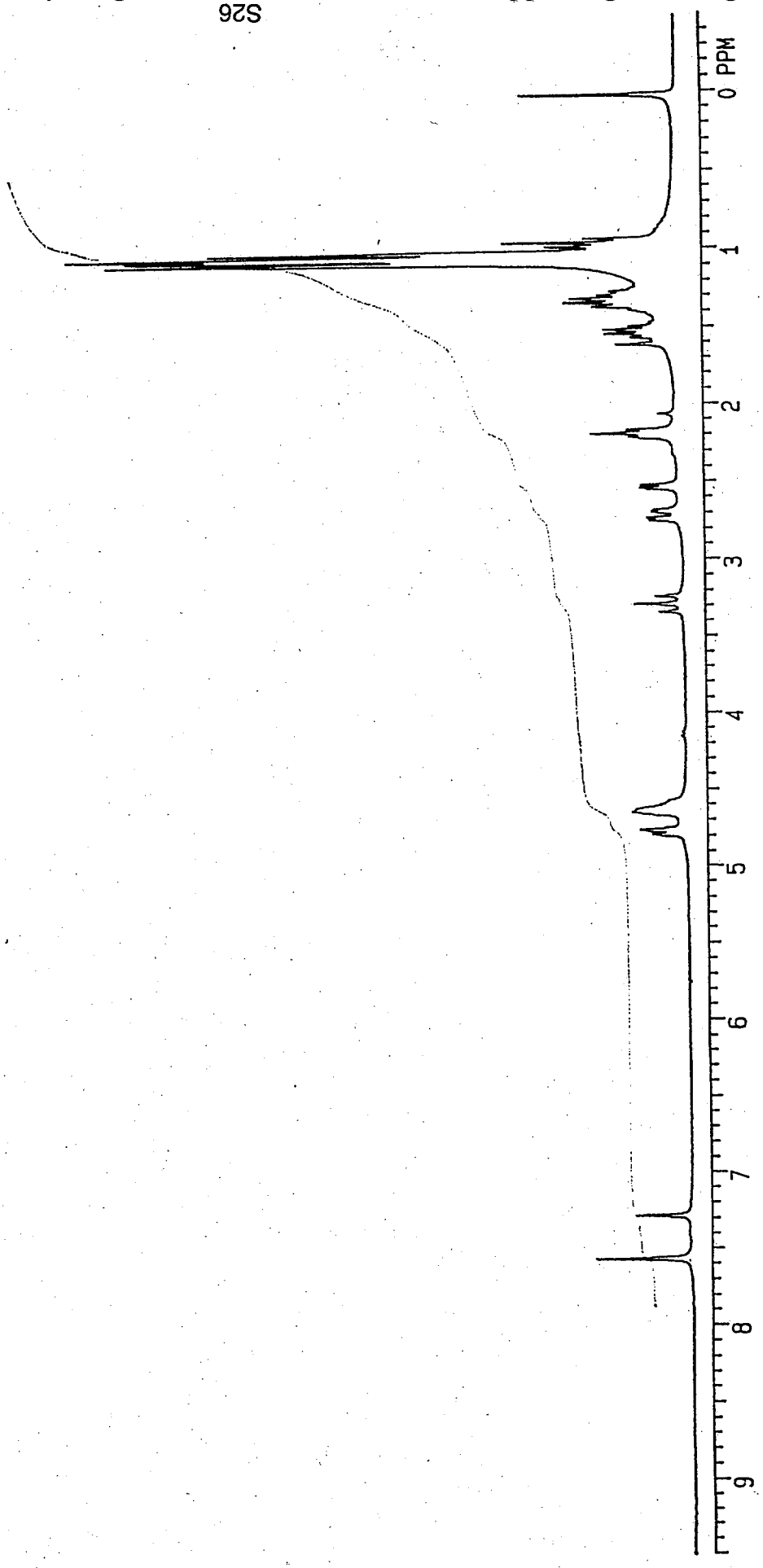


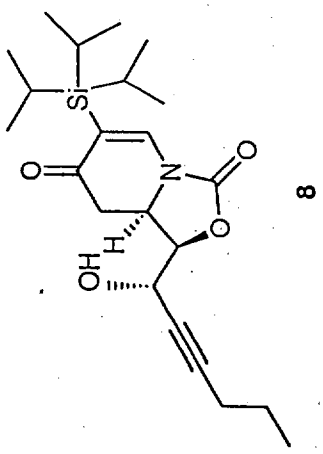
6



7

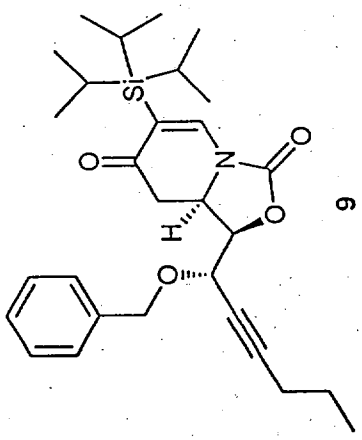
7





8

8



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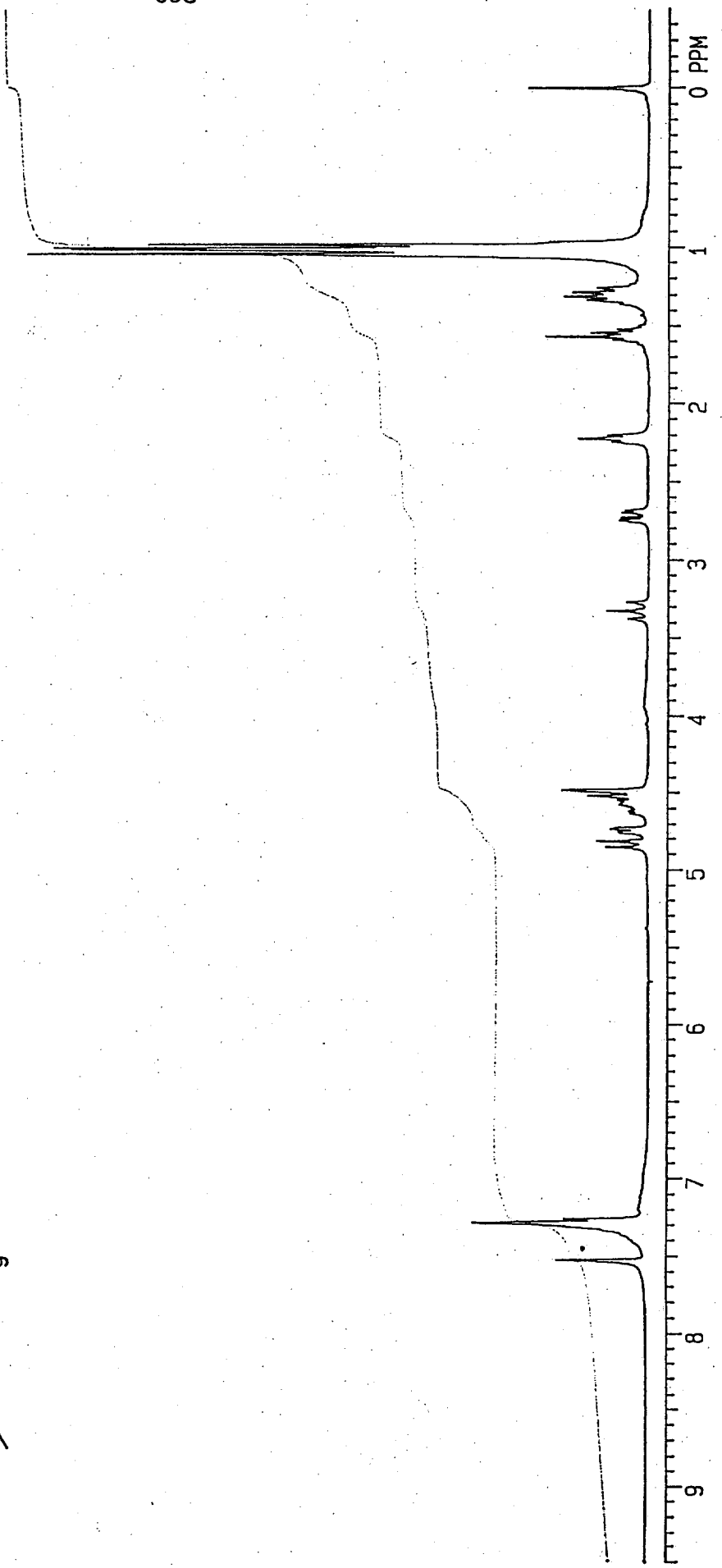

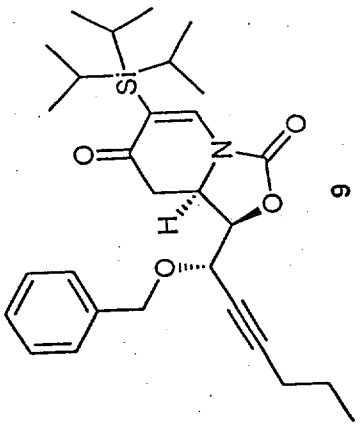

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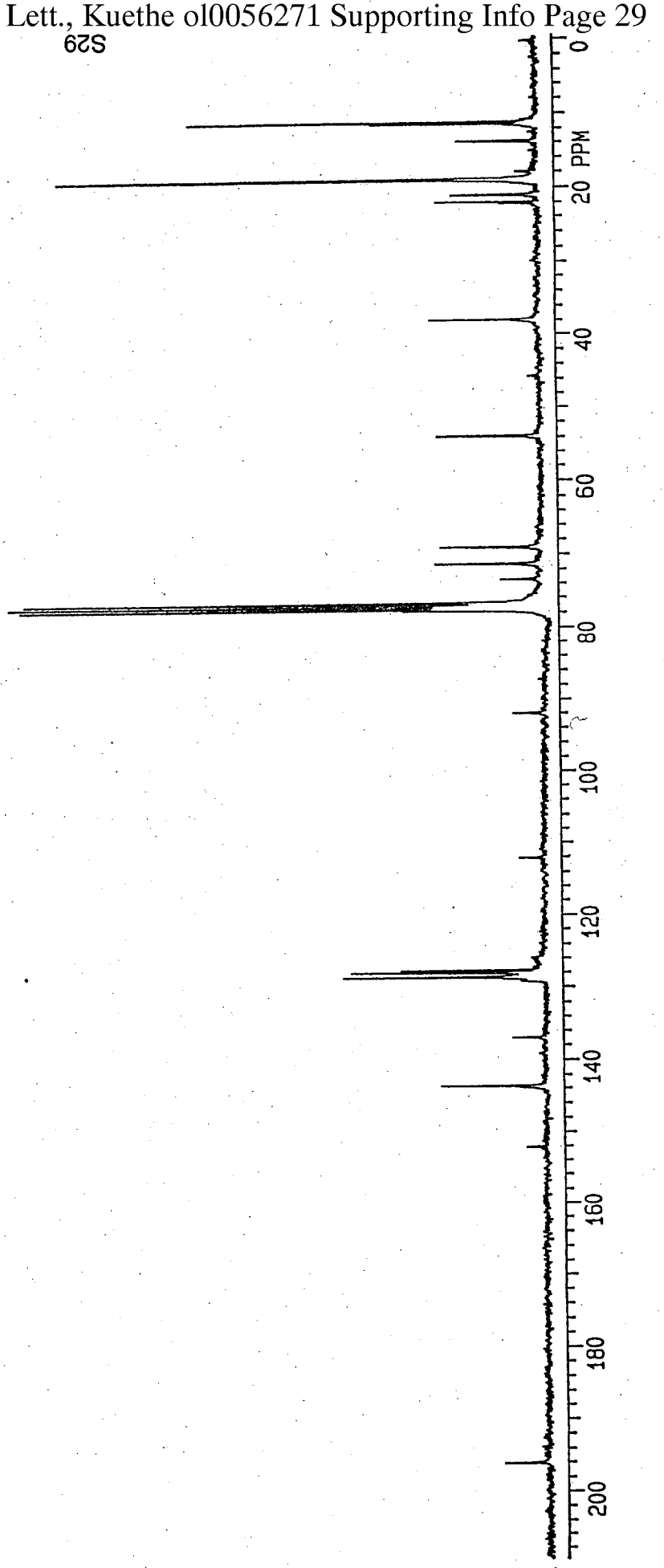

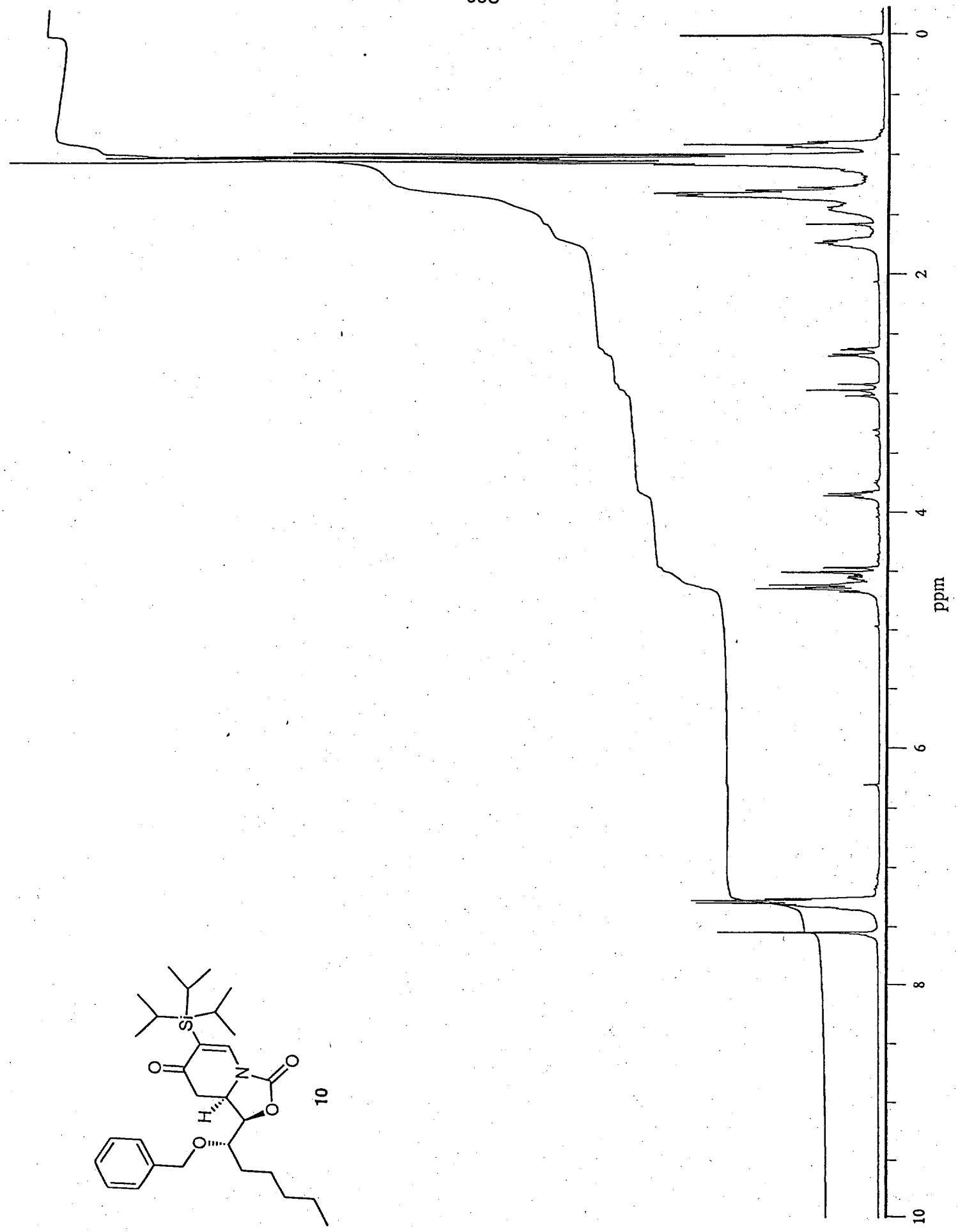

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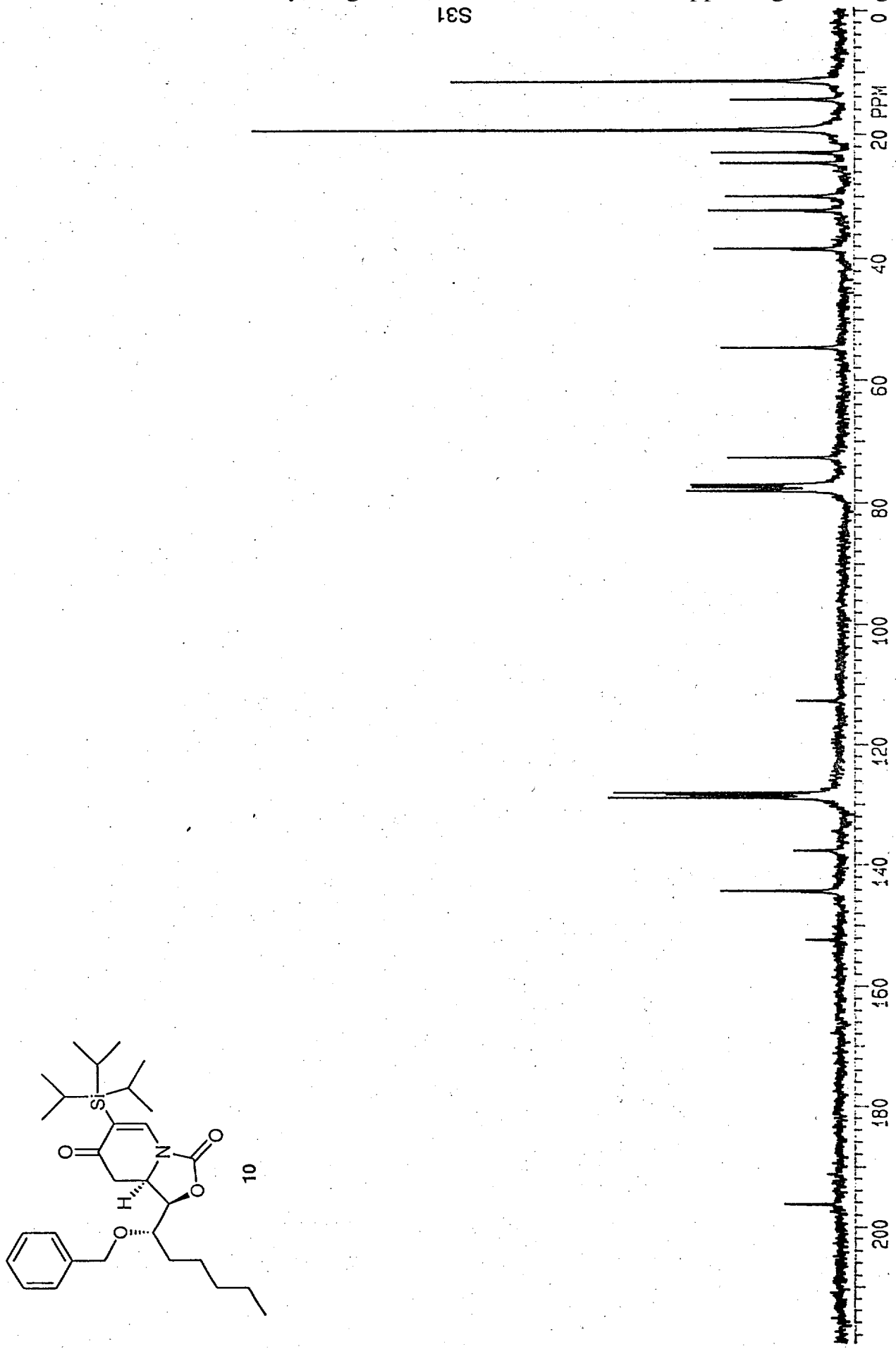
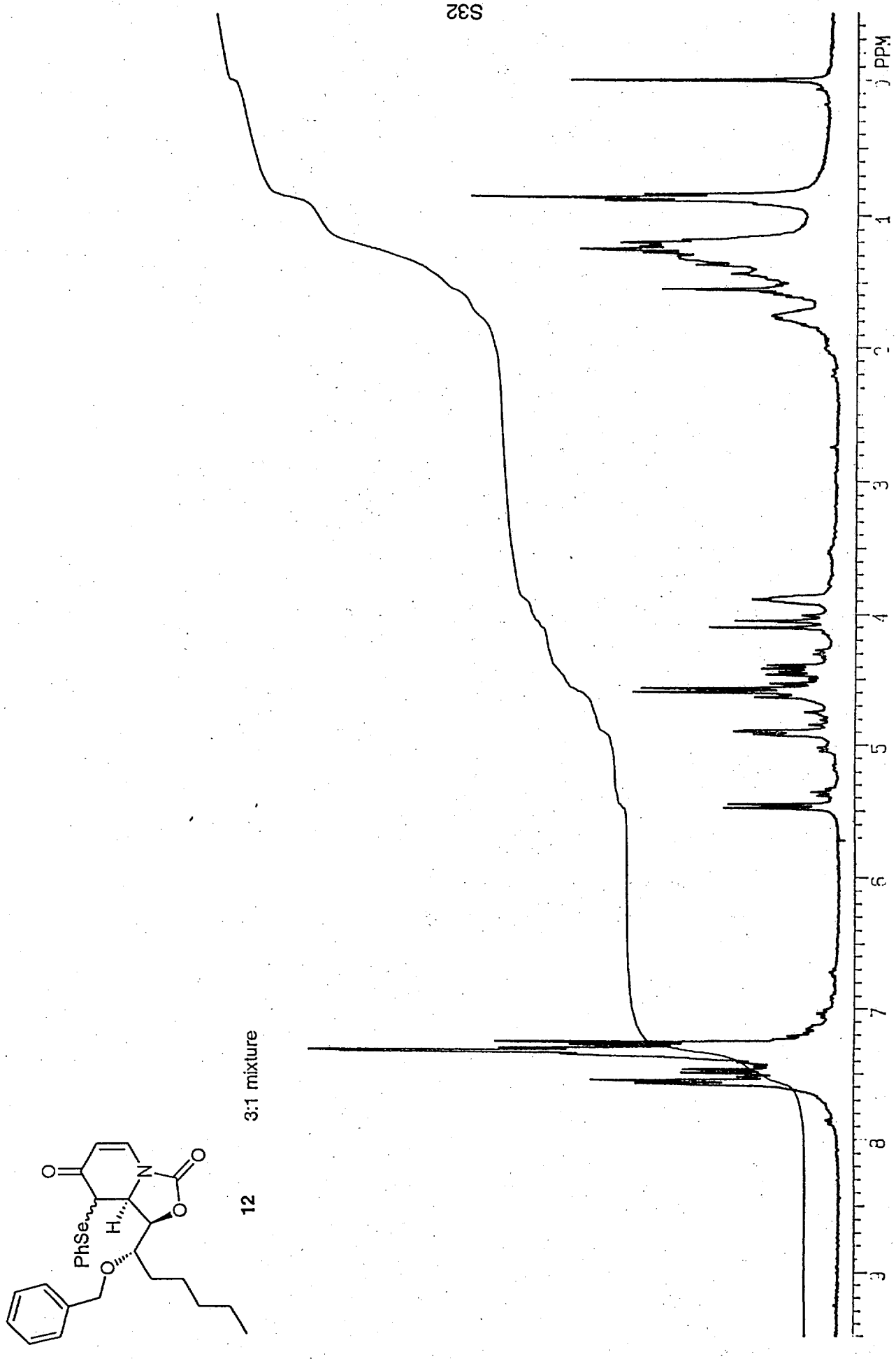
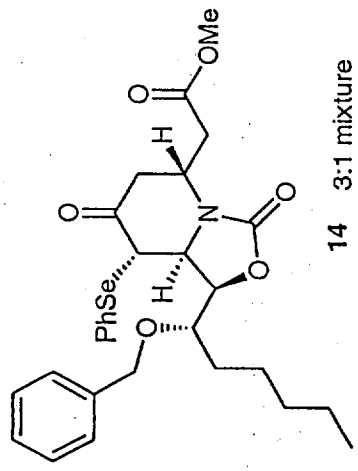




12 3:1 mixture

12

3:1 mixture



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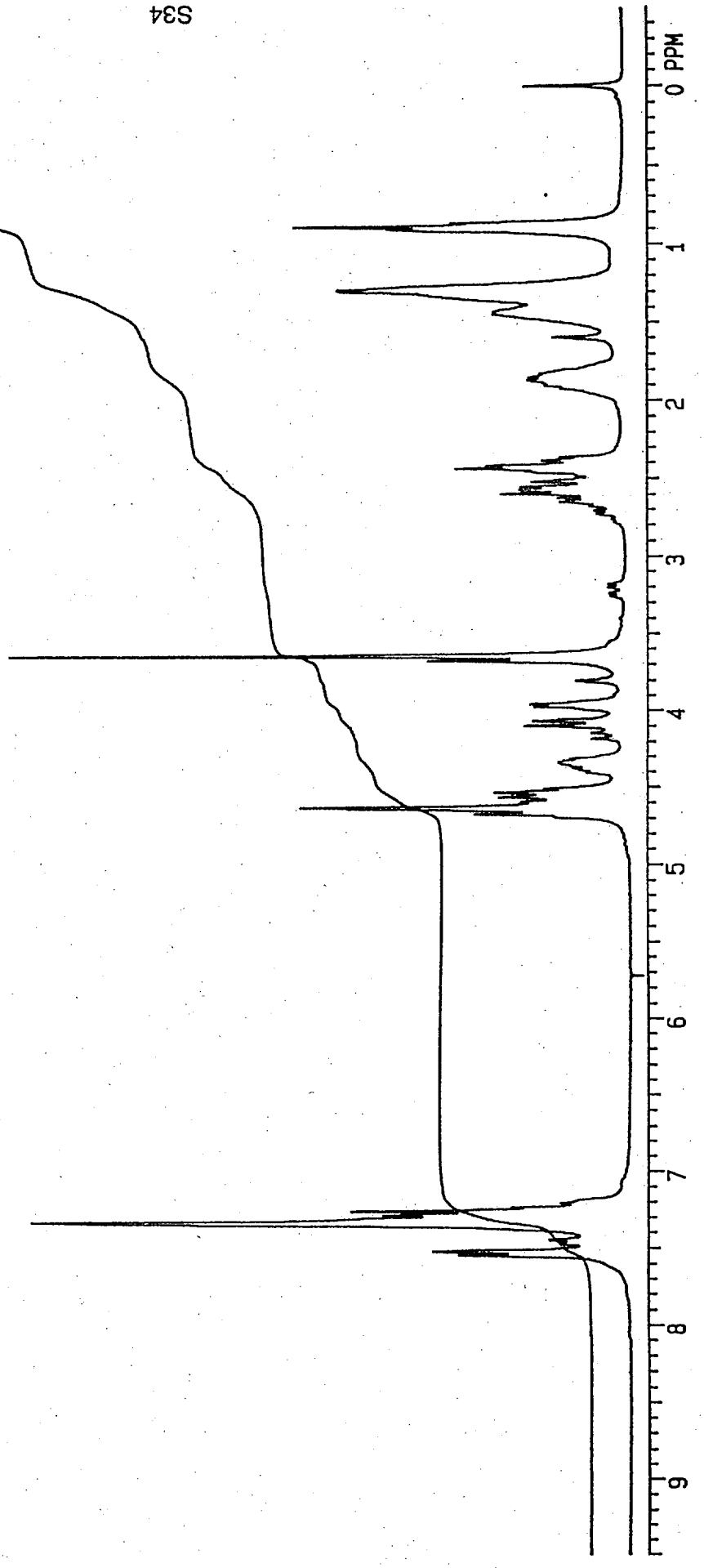

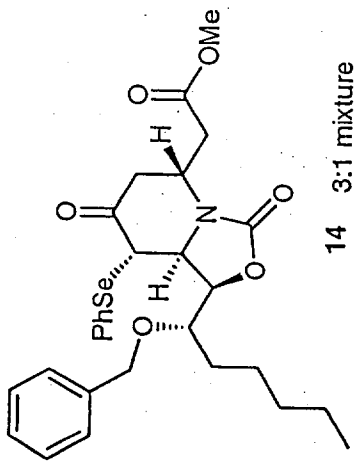

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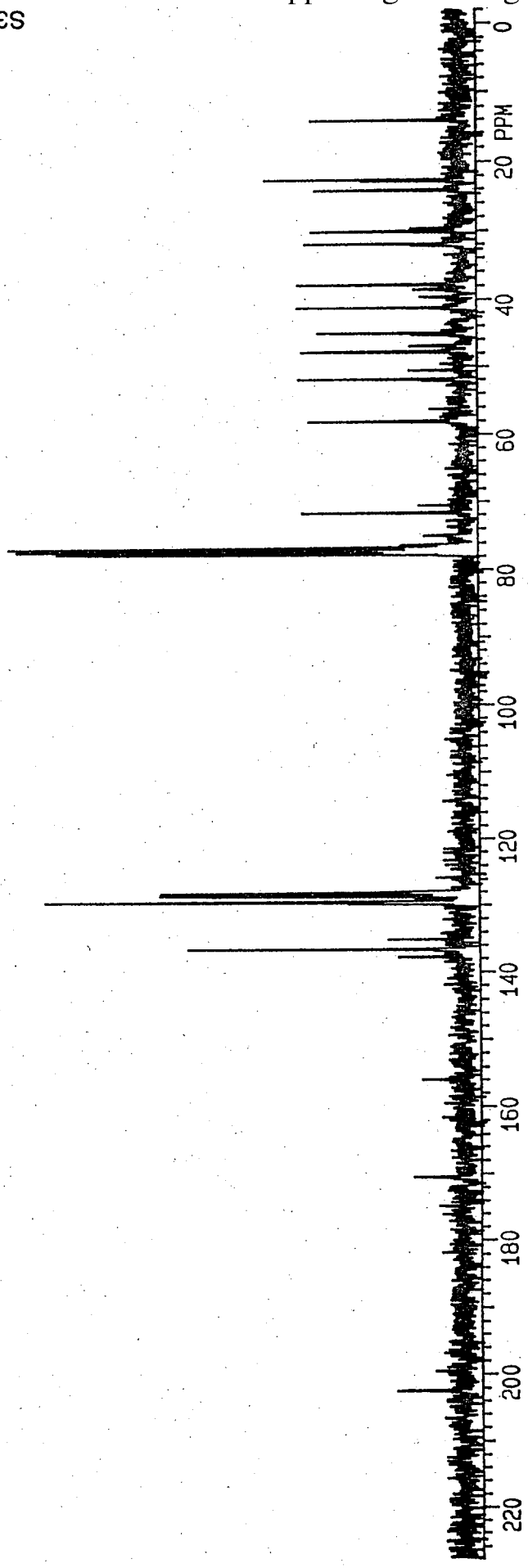

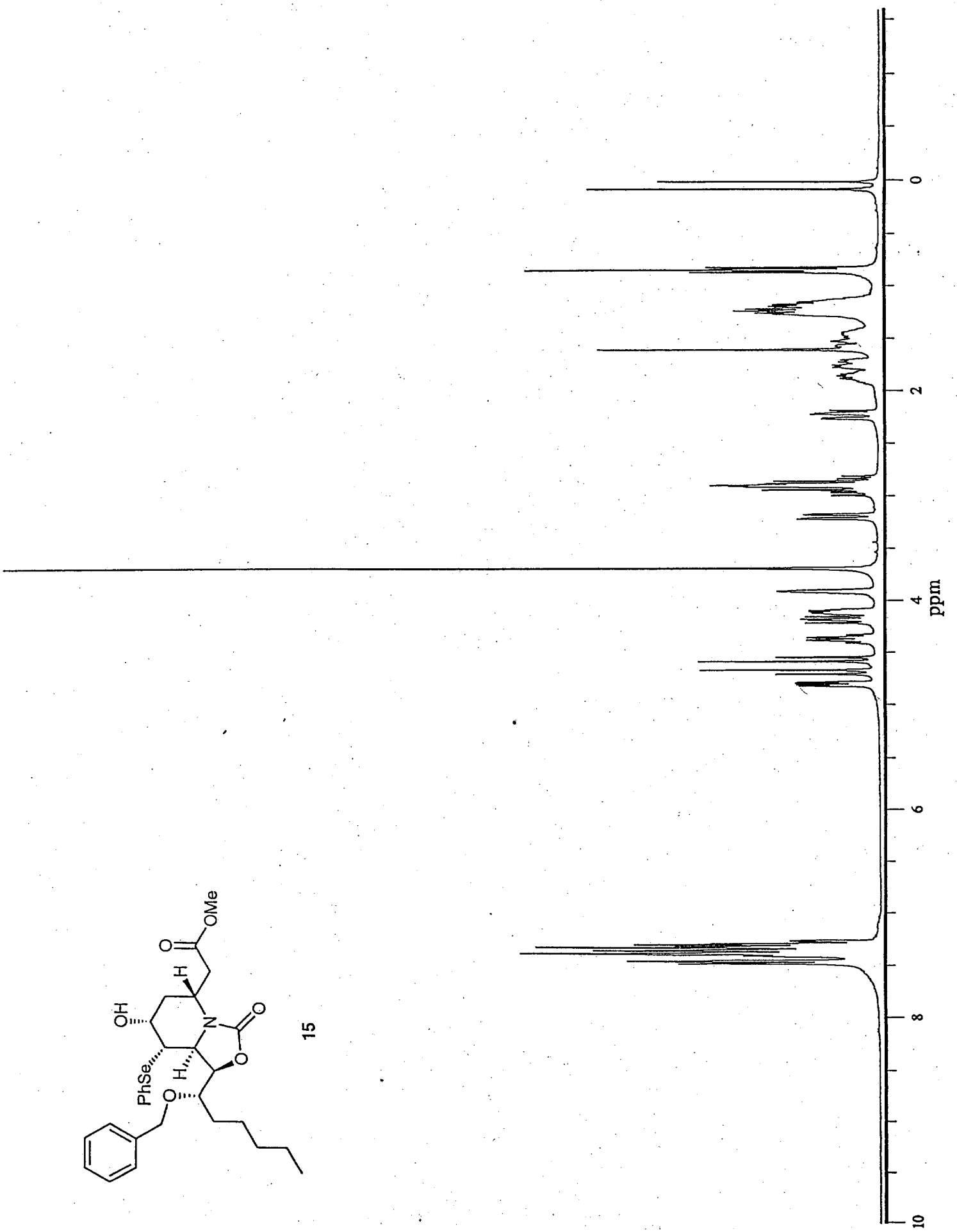

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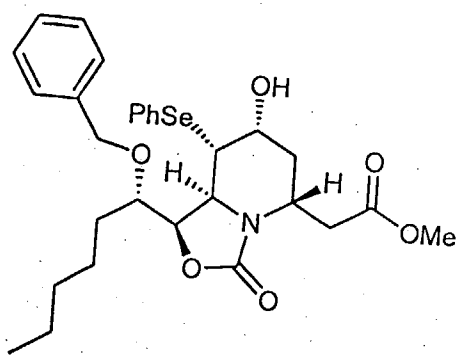

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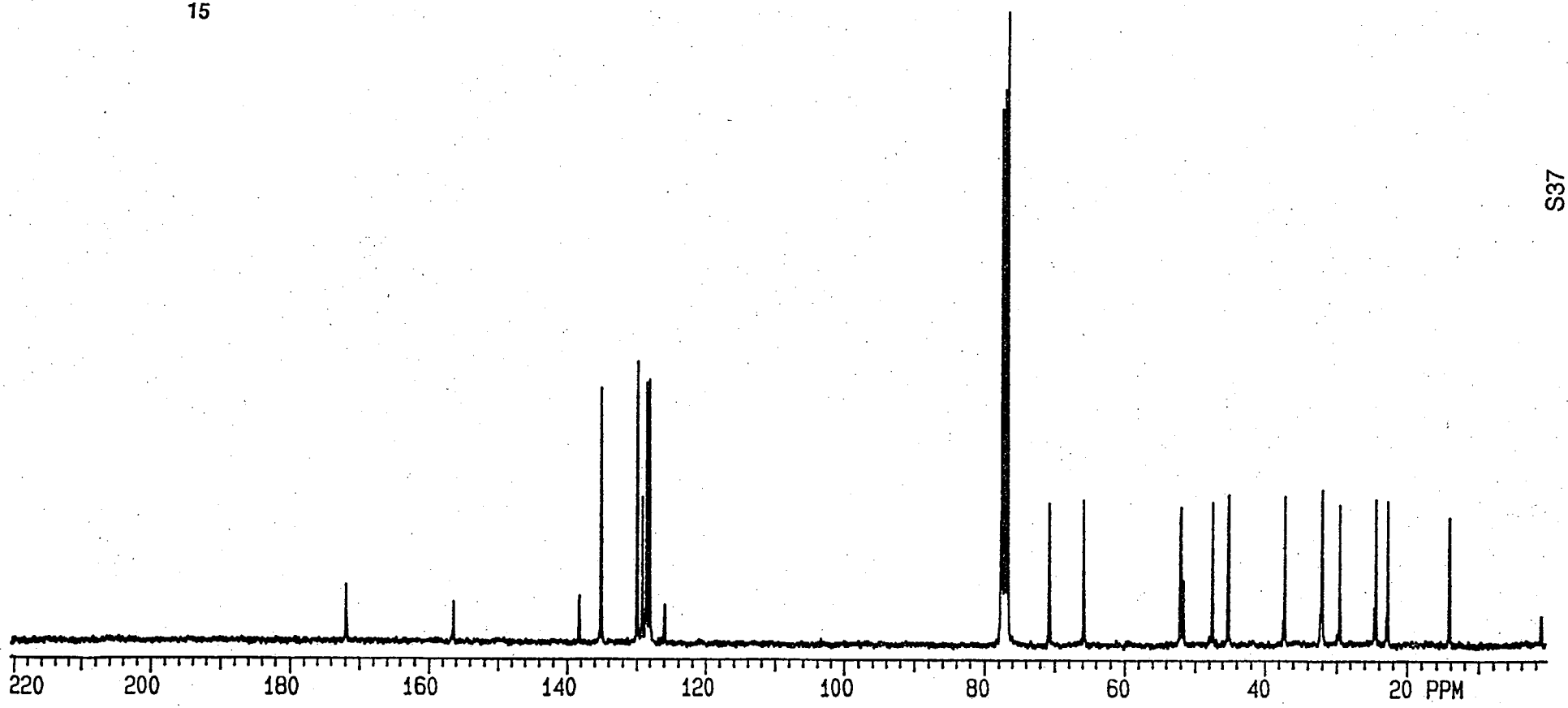

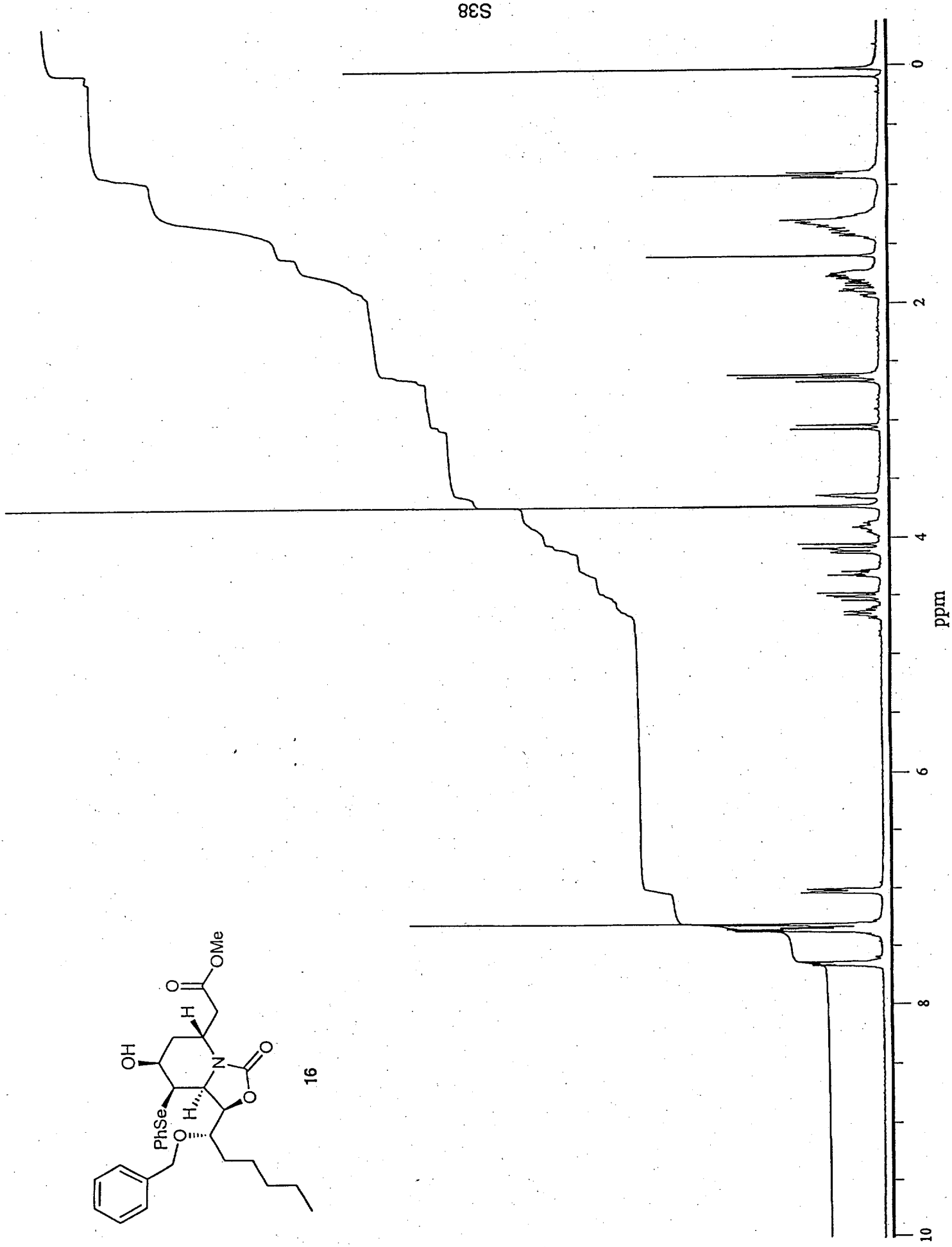

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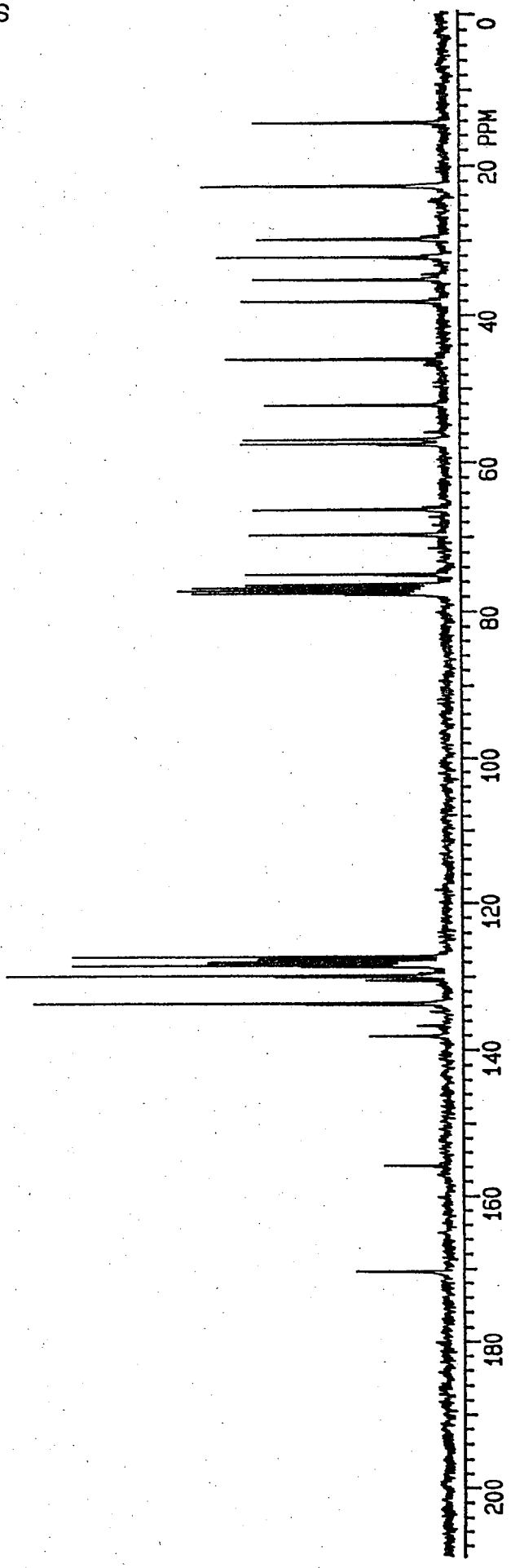

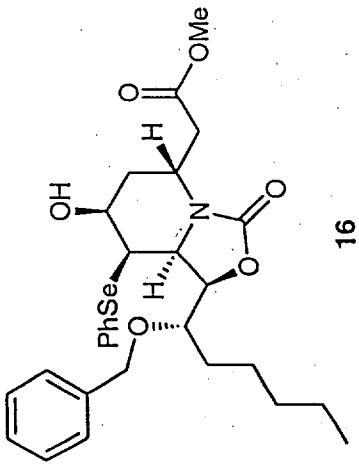

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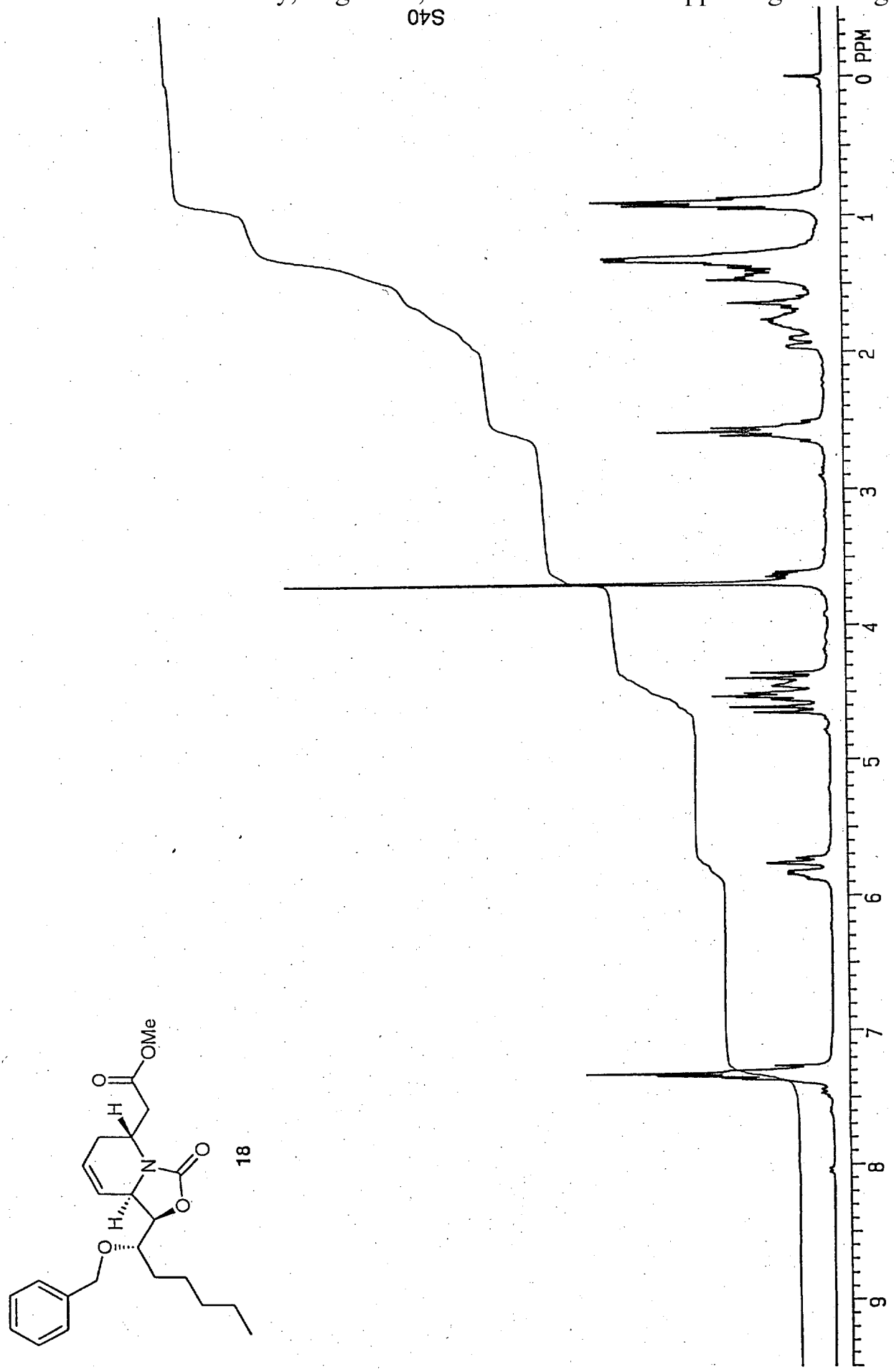

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