

Organic Letters

SUPPORTING INFORMATION

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"Synthesis and Reactivity of *N*-Acyl-5-(1-hydroxyalkyl)-2,3-dihydro-4-pyridones"

I. Spectral data for **2a**, **3a-g**, and **4-10** (23 pages).

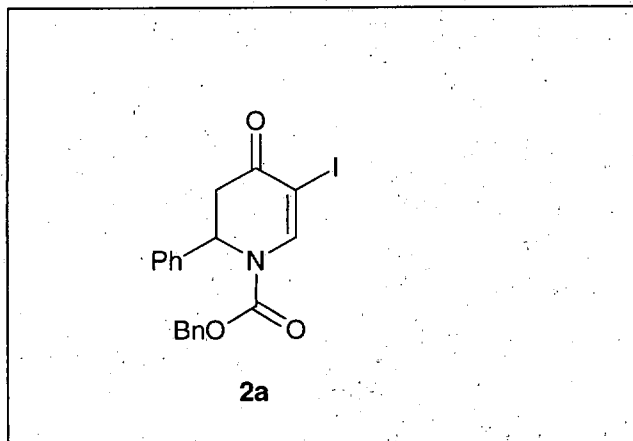
II. ^1H and ^{13}C NMR spectra of **2a**, **3f-g**, **4**, **5** and **6-10** (28 pages).

Total pages of supporting information: (51).

Compound Name: 5-Iodo-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 2a

Notebook Pg(s):

Formula: C₁₉H₁₆NO₃

Molecular Weight: 433.24

Yield(s):

Appearance: yellow solid

Stability: stable away from light

mp/bp: 111-112

[α]_D²³Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 434.0253

Found 434.0256

IR (thin film): cm⁻¹

3032, 2962, 1732, 1675, 1579, 1497, 1454, 1391, 1291, 1248, 1179, 1115, 1020, 951, 913, 737, 697

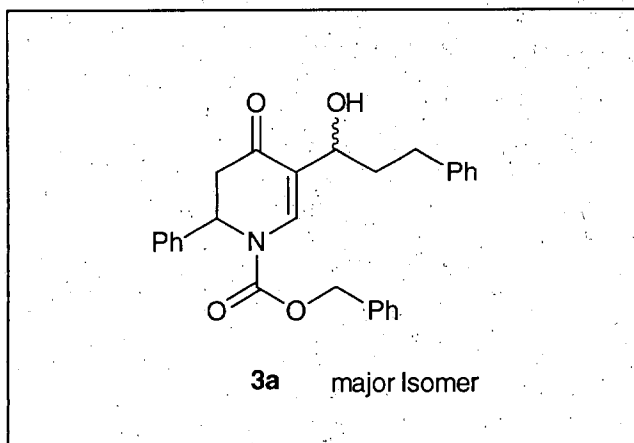
NMR (CDCl₃):¹H-NMR (300 MHz) δ8.51 (s, 1H), 7.37-7.15 (m, 10H), 5.79 (d, 1H, *J*=6.6 Hz), 5.27-5.25 (m, 2H), 3.29-3.10 (m, 2H)¹³C-NMR (75 MHz) δ

185.83, 151.59, 147.52, 137.70, 134.53, 129.02, 128.82, 128.69, 128.39, 128.28, 125.72, 76.00, 69.55, 56.33, 40.57

Literature References:

Compound Name: 5-(1-Hydroxy-3-phenyl-propyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3a**-major
 Notebook Pg(s): 8y, 9y, 11w, 13w, 13y, 14w, 14y, 15w, 15y, 40w



Formula: C₂₈H₂₇NO₄

Molecular Weight: 441.52

Yield(s): 64% d.r. = 1/2.5

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
 Analysis
 (Atlantic Microlabs, Inc.)

	%C	%H	%N	%____
Calculated:	76.17	6.16	3.17	
Found:	76.01	6.24	3.24	

HRMS: Calculated 442.2018

Found 442.2006

IR (thin film): cm⁻¹

3444, 3028, 2920, 1726, 1660, 1621, 1496, 1454, 1386, 1297, 1250, 1118, 1022, 964, 742, 697

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

8.00 (s, 1H), 7.32-7.11 (m, 15H), 5.67 (d, 1H, *J* = 6.9 Hz), 5.28-5.18 (m, 2H), 4.42 (dd, 1H, *J* = 6.3 Hz and 12.6 Hz), 3.06 (dd, 1H, *J* = 7.6 Hz and 16.5 Hz), 2.81-2.52 (m, 3H), 1.98-1.90 (m, 2H)

¹³C-NMR (75 MHz) δ

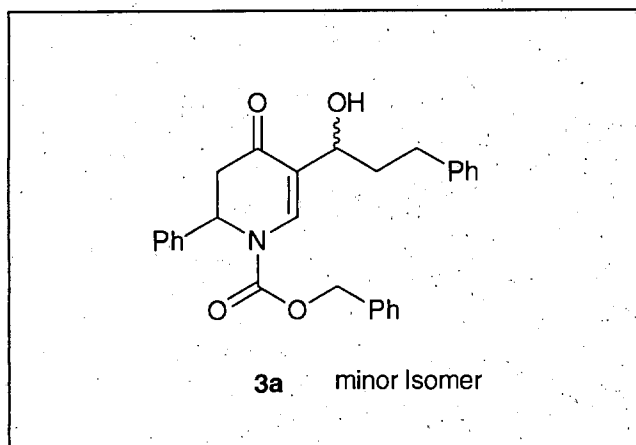
192.20, 152.78, 141.65, 139.39, 138.17, 134.82, 128.88, 128.61, 128.43, 128.30, 128.02, 125.72, 120.54, 69.13, 68.78, 55.64, 41.88, 38.00, 31.92

Literature References:

Compound Name: 5-(1-Hydroxy-3-phenyl-propyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3a**-minor

Notebook Pg(s): 8y, 9y, 11w, 13w, 13y, 14w, 14y, 15w, 15y, 40w



Formula: C₂₈H₂₇NO₄

Molecular Weight: 441.52

Yield(s): 64% d.r. = 1/2.5

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	76.17	6.16	3.17	
Found:	76.46	6.22	3.13	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3439, 3028, 2920, 1726, 1662, 1622, 1496, 1454, 1386, 1298, 1251, 1118, 1024, 961, 750, 698

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

7.90 (s, 1H), 7.34-7.09 (m, 15H), 5.70 (d, 1H, *J* = 6.8 Hz), 5.29-5.19 (m, 2H), 4.26 (t, 1H, *J* = 6.5 Hz), 3.12 (dd, 1H, *J* = 7.4 Hz and 16.8 Hz), 2.83 (d, 1H, *J* = 16.6 Hz), 2.76-2.47 (m, 2H), 2.11-1.84 (m, 2H)

¹³C-NMR (75 MHz) δ

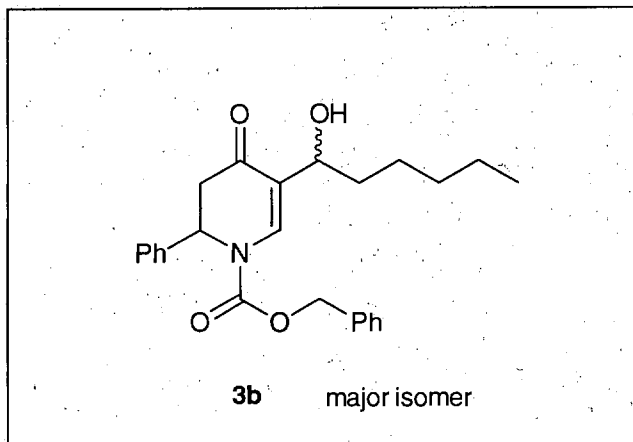
192.63, 152.78, 141.53, 139.42, 137.98, 134.76, 128.89, 128.64, 128.39, 128.30, 128.09, 125.77, 120.38, 70.07, 69.21, 55.42, 41.98, 38.03, 32.10

Literature References:

Compound Name: 5-(1-Hydroxy-hexyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3b**-major

Notebook Pg(s): 16y, 36w, 41y, 55w, 56w



Formula: C₂₅H₂₉NO₄

Molecular Weight: 407.50

Yield(s): 15% - 31% - 52% - 57%

d.r. = 1:1.6

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	73.68	7.17	3.44	
Found:	73.65	7.24	3.48	

HRMS: Calculated 408.2175

Found 408.2170

IR (thin film): cm⁻¹ 3441, 3032, 2930, 2858, 1727, 1662, 1622, 1497, 1454, 1386, 1298, 1247, 1197, 1117, 1022, 962, 763, 697

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

8.01 (s, 1H), 7.33-7.15 (m, 10H), 5.70 (d, 1H, *J* = 7 Hz), 5.29-5.19 (m, 2H), 4.40 (dd, 1H, *J* = 6.3 Hz and 12.6 Hz), 3.14 (dd, 1H, *J* = 7.5 Hz and 16.5 Hz), 2.80 (d, 1H, *J* = 16.5 Hz), 2.52 (d, 1H, *J* = 5.7 Hz), 1.66-1.59 (m, 2H), 1.25 (s, 6H), 0.88-0.84 (m, 3H)

¹³C-NMR (75 MHz) δ

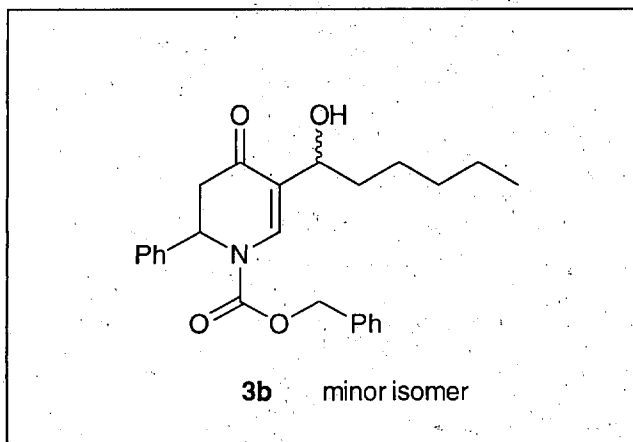
192.05, 152.82, 139.19, 138.26, 134.87, 128.83, 128.60, 128.18, 127.98, 125.76, 121.05, 69.07, 69.00, 55.68, 41.99, 36.58, 31.59, 25.28, 22.55, 13.99

Literature References:

Compound Name: 5-(1-Hydroxy-hexyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3b**-minor

Notebook Pg(s): 16y, 36w, 41y, 55w, 56w



Formula: C₂₅H₂₉NO₄

Molecular Weight: 407.50

Yield(s): 15% - 31% - 52% - 57%

d.r. = 1/1.6

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³:

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	73.68	7.17	3.44	
Found:	73.40	7.35	3.37	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3444, 3092, 3060, 3028, 2953, 2920, 2857, 1723, 1659, 1622, 1494, 1451, 1387, 1301, 1248, 1200, 1114, 1018, 959, 756, 698

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

7.91 (s, 1H), 7.34-7.15 (m, 10H), 5.71 (d, 1H, *J* = 6.9 Hz), 5.25 (dd, 2H, *J* = 12 Hz and 20.1 Hz), 4.21 (dd, 1H, *J* = 6.9 Hz and 13.7 Hz), 3.15 (dd, 1H, *J* = 7.4 Hz and 16.6 Hz), 2.90-2.80 (m, 2H), 1.71-1.56 (m, 2H), 1.24-1.23 (m, 6H), 0.87-0.83 (m, 3H)

¹³C-NMR (75 MHz) δ

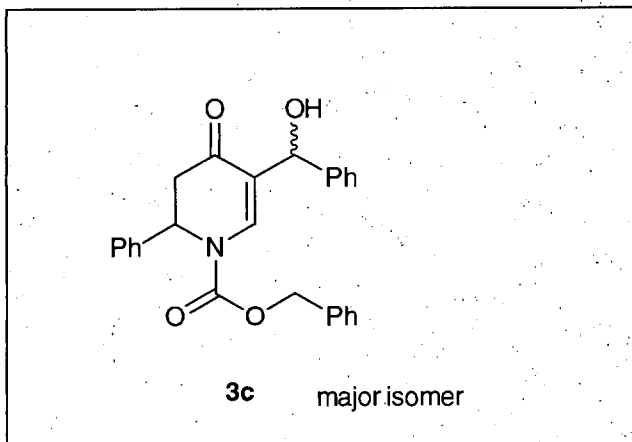
192.38, 152.72, 139.23, 137.98, 134.73, 128.70, 128.51, 128.10, 127.89, 125.71, 120.73, 70.45, 69.02, 55.39, 41.98, 36.47, 31.44, 25.52, 22.43, 13.09

Literature References:

Compound Name: 5-(Hydroxy-phenyl-methyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid
benzyl ester

Compound No: **3c**-major

Notebook Pg(s): 21y, 37w, 37y, 49y



Formula: C₂₆H₂₃NO₄

Molecular Weight: 413.46

Yield(s): 40%, 80%, 53%, 61%

d.r. = 7/1

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³:

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	75.53	5.61	3.39	
Found:	75.28	5.67	3.34	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3442, 3062, 3031, 2962, 2896, 1726, 1665, 1622, 1622, 1496, 1453, 1387, 1317,
1257, 1199, 1119, 1018, 963, 738, 698

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

7.88 (s, 1H), 7.32-7.11 (m, 15H), 5.70 (d, 1H, *J* = 7.2 Hz), 5.62 (d, 1H, *J* = 4.9 Hz), 5.25-5.15 (m, 2H), 3.17 (dd, 1H, *J* = 7.5 Hz and 16.7 Hz), 3.09 (d, 1H, *J* = 4.9 Hz), 2.82 (d, 1H, *J* = 16 Hz)

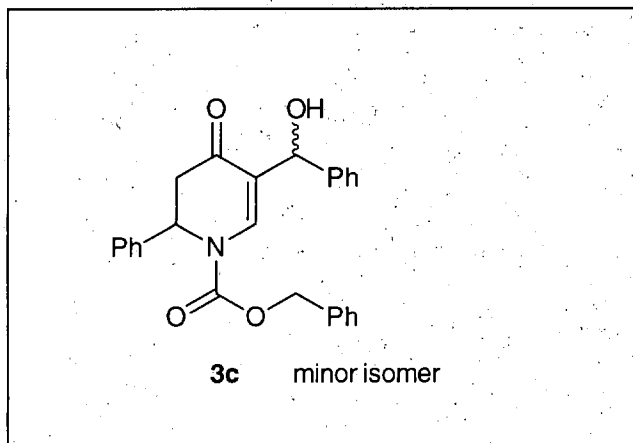
¹³C-NMR (75 MHz) δ

191.49, 152.51, 141.84, 140.22, 138.04, 134.64, 128.59, 128.39, 128.09, 127.82,
127.24, 126.12, 125.70, 121.12, 69.73, 68.81, 55.55, 41.65

Literature References:

Compound Name: 5-(Hydroxy-phenyl-methyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3c**-minor
 Notebook Pg(s): 21y, 37w, 37y, 49y



Formula: C₂₆H₂₃NO₄

Molecular Weight: 413.46

Yield(s): 40%, 80% (7:1), 53%, 61%

d.r. = 7/1

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
 Analysis
 (Atlantic Microlabs, Inc.)

	%C	%H	%N	%_____
Calculated:	75.53	5.61	3.39	
Found:	75.61	5.64	3.21	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3448, 1724, 1659, 1626, 1496, 1452, 1387, 1316, 1258, 1119, 1019, 961, 738, 698

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

7.78 (s, 1H), 7.33-7.17 (m, 15H), 5.71 (d, 1H, J = 7.2 Hz), 5.55 (d, 1H, J = 5.4 Hz), 5.19 (dd, 2H, J = 12.3 Hz, 16.3 Hz), 3.37 (d, 1H, J = 5.6 Hz), 3.17 (dd, 1H, J = 7.5 Hz and 16.7 Hz), 2.84 (d, 1H, J = 16.7 Hz)

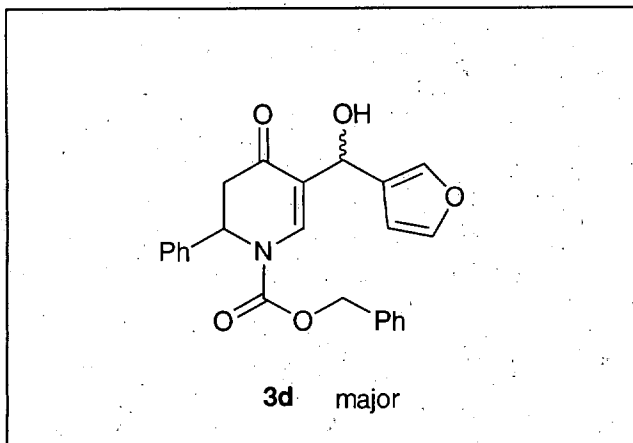
¹³C-NMR (75 MHz) δ

192.43, 152.67, 141.39, 140.68, 138.16, 134.76, 128.94, 128.64, 128.36, 128.17, 128.01, 127.60, 126.32, 126.00, 121.06, 71.40, 59.15, 55.83, 41.94

Literature References:

Compound Name: 5-(Furan-3-yl-hydroxy-methyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3d**-major
 Notebook Pg(s): 43w, 47w



Formula: C₂₄H₂₁NO₅

Molecular Weight: 403.43

Yield(s): 40%, 68% d.r. = 1/2.6

Appearance: yellow oil

Stability: (

mp/bp:

[α]_D²³

Elemental
 Analysis
 (Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	71.45	5.25	3.47	
Found:	71.39	5.33	3.43	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3436, 3064, 3033, 1727, 1665, 1623, 1498, 1453, 1387, 1326, 1295, 1253, 1216, 1158, 1120, 1023, 974, 874, 763, 698

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

8.02 (s, 1H), 7.32-7.12 (m, 12H), 6.26 (s, 1H), 5.71 (d, 1H, *J* = 6.9 Hz), 5.56 (d, 1H, *J* = 3.5 Hz), 5.22 (dd, 2H, *J* = 12.1 Hz and 15.9 Hz), 3.19-3.11 (m, 2H), 2.84 (d, 1H, *J* = 15.4 Hz)

¹³C-NMR (75 MHz) δ

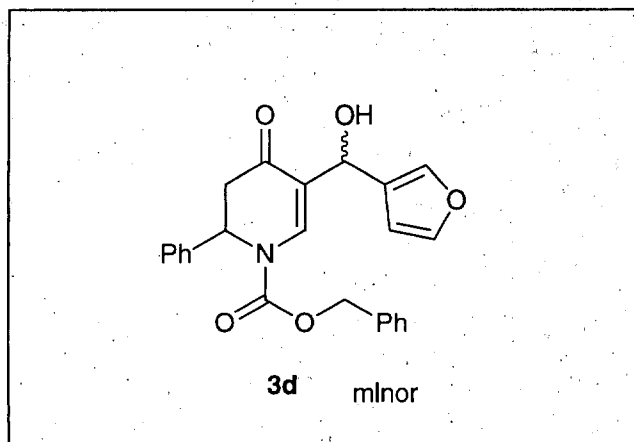
191.56, 152.58, 143.00, 140.20, 139.51, 137.96, 134.66, 128.71, 128.45, 127.95, 127.04, 125.65, 120.38, 108.82, 68.97, 63.29, 55.56, 41.64

Literature References:

Compound Name: 5-(Furan-3-yl-hydroxy-methyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine
-1-carboxylic acid benzyl ester

Compound No: **3d**-minor

Notebook Pg(s): 43w, 47w



Formula: C₂₄H₂₁NO₅

Molecular Weight: 403.43

Yield(s): 40%, 68% d.r. = 1/2.6

Appearance: yellow oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	71.45	5.25	3.47	
Found:	71.09	5.55	3.36	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3433, 3027, 2902, 1726, 1666, 1622, 1498, 1451, 1386, 1321, 1294, 1249, 1214,
1157, 1120, 1023, 958, 874, 762, 697

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

7.94 (s, 1H), 7.33-7.16 (m, 12H), 6.27 (s, 1H), 5.73 (d, 1H, J = 7 Hz), 5.43 (d, 1H, J =
6.5 Hz), 5.22 (dd, 2H, J = 12.2 Hz and 17 Hz), 3.40 (d, 1H, J = 6.6 Hz), 3.17 (dd, 1H, J =
7.5 Hz and 17 Hz), 2.87 (d, 1H, J = 16.6 Hz)

¹³C-NMR (75 MHz) δ

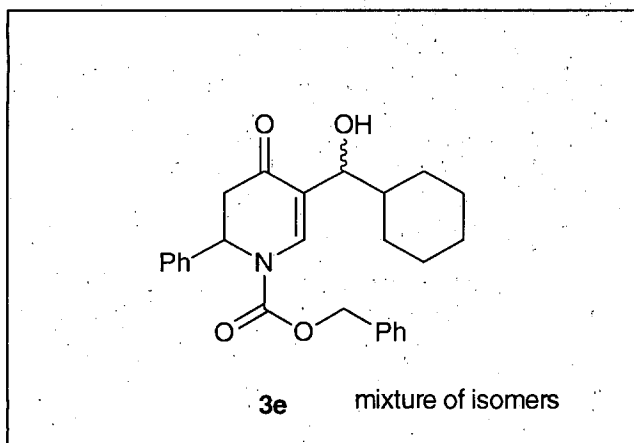
192.43, 152.69, 143.19, 140.40, 139.66, 137.95, 134.72, 128.95, 128.65, 128.19,
128.12, 126.82, 125.87, 120.08, 109.02, 69.24, 65.33, 55.73, 41.92

Literature References:

Compound Name: 5-(Cyclohexyl-hydroxy-methyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **3e**

Notebook Pg(s): 43y book A



Formula: C₂₆H₂₉NO₄

Molecular Weight: 419.51

Yield(s): 64%

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	74.44	6.97	3.34	
Found:	74.21	7.03	3.28	

LRMS: Calculated

Found

IR (thin film): cm⁻¹

3446, 3032, 2925, 2851, 1725, 1661, 1618, 1497, 1450, 1387, 1247, 1197, 1118, 1017, 969, 738, 697

NMR (CDCl₃): 300MHz

¹H-NMR (300 MHz) δ

7.96 (s, 1H), 7.29-7.14 (m, 10H), 5.69 (d, 1H, *J* = 6.18 Hz), 5.22 (dd, 2H, *J* = 12.3 Hz and 16.1 Hz), 4.13 (t, 1H, *J* = 6.1 Hz), 3.08 (dd, 1H, *J* = 7.3 Hz and 16.5 Hz), 2.81-2.74 (m, 2H), 1.75-0.78 (m, 11H)

¹³C-NMR (75 MHz) δ

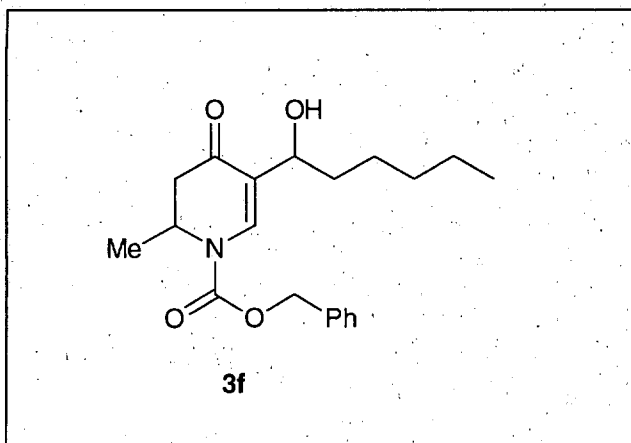
191.76, 152.69, 139.61, 138.02, 134.79, 128.58, 128.43, 128.01, 127.77, 125.70, 120.03, 73.16, 68.87, 55.39, 41.70, 29.35, 28.07, 26.20, 25.90, 25.73

Literature References:

Compound Name: 5-(1-Hydroxy-hexyl)-2-methyl-4-oxo-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 3f

Notebook Pg(s): 56y-59w

Formula: C₂₀H₂₅NO₄

Molecular Weight: 351.40

Yield(s): 64% (1:2.6)

Appearance: oil

Stability:

mp/bp:

[α]_D²³Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

LRMS: Calculated 346

Found 346

IR (thin film): cm⁻¹

3435, 2955, 2930, 2858, 1725, 1662, 1619, 1456, 1388, 1309, 1264, 1138, 1107, 1065, 764, 698

NMR (CDCl₃):¹H-NMR (300 MHz) δ

isomer A 7.74 (s, 1H), 7.39 (s, 5H), 5.33-5.23 (m, 2H), 4.38-4.32 (m, 1H), 2.90-2.81 (m, 1H), 2.35-2.29 (m, 1H), 1.70-1.61 (m, 2H)

isomer B 7.70 (s, 1H), 7.39 (s, 5H), 5.33-5.23 (m, 2H), 4.31-4.24 (m, 1H), 2.90-2.81 (m, 1H), 2.35-2.29 (m, 1H), 1.70-1.61 (m, 2H)

¹³C-NMR (75 MHz) δ

isomer A 193.30, 152.49, 138.13, 135.06, 128.75, 128.38, 119.63, 70.49, 69.72, 68.99, 49.07, 42.07, 36.32, 31.62, 25.62, 22.57, 16.83, 14.00

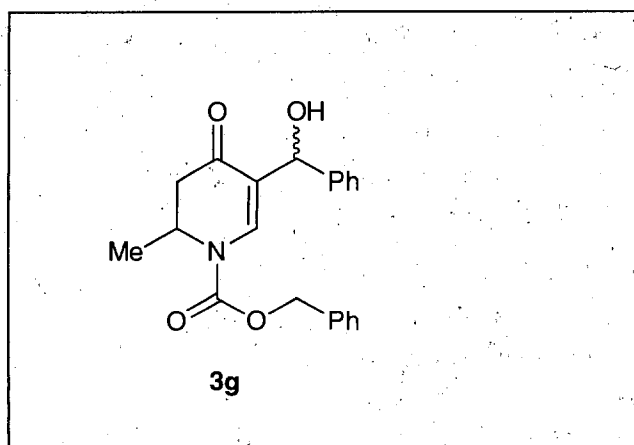
isomer B 193.30, 152.49, 138.13, 135.06, 128.75, 128.38, 119.63, 70.49, 69.72, 68.99, 48.99, 42.25, 36.54, 31.62, 25.76, 22.57, 16.77, 14.00

Literature References:

Compound Name: 5-(Hydroxy-phenyl-methyl)-2-methyl-4-oxo-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 3g

Notebook Pg(s): 57y-58y



Formula: C₂₁H₂₁NO₄

Molecular Weight: 351.40

Yield(s): 57%, 46% (d.r. = 1/3.5 and 1/2.6)

Appearance: oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 351.1417

Found 351.1483

IR (thin film): cm⁻¹

3438, 3063, 3032, 2978, 1724, 1661, 1620, 1496, 1454, 1386, 1318, 1265, 1193, 1136, 1102, 1065, 1026, 989, 946, 738, 699

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

major isomer: 7.64 (s, 1H), 7.36-7.23 (m, 10H), 5.59 (d, 1H, *J* = 4.7 Hz), 5.25-5.16 (m, 2H), 4.65 (t, 1H, *J* = 6.5 Hz), 3.50 (d, 1H, *J* = 4.9 Hz), 2.81 (dd, 1H, *J* = 6.8 and 16.5 Hz), 2.28 (d, 1H, *J* = 16.5 Hz), 1.17 (d, 3H, *J* = 6.8 Hz)

minor isomer: 7.51-7.26 (m, 11H), 5.59 (d, 1H, *J* = 4.7 Hz), 5.21 (s, 2H), 4.68 (t, 1H, *J* = 6.4 Hz), 3.48 (d, 1H, *J* = 3.5 Hz), 5.70 (dd, 1H, *J* = 6.9 and 16.5 Hz), 2.34 (d, 1H, *J* = 16.5 Hz), 1.25 (d, 3H, *J* = 6.7 Hz)

¹³C-NMR (75 MHz) δ

major isomer: 192.81, 152.19, 141.90, 139.33, 134.84, 128.57, 128.26, 128.01, 127.39, 126.14, 119.74, 70.39, 68.78, 49.02, 41.83, 16.82

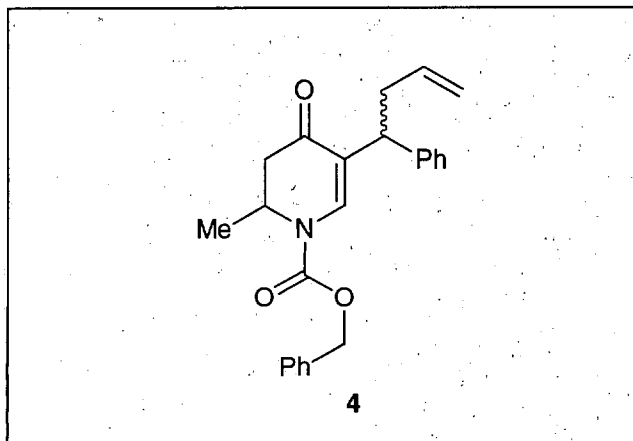
minor isomer: 193.28, 152.26, 141.36, 139.55, 134.87, 128.68, 128.32, 128.02, 127.59, 126.30, 119.94, 71.03, 68.90, 49.15, 41.89, 16.90

Literature References:

Compound Name: 2-Methyl-4-oxo-5-(1-phenyl-but-3-enyl)-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 4

Notebook Pg(s): 61w



Formula: C₂₄H₂₅NO₃

Molecular Weight: 375.46

Yield(s): 100% mix of diastereomers

d.r. = 1/1.8

Appearance: oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 376.1913

Found 376.1924

IR (thin film): cm⁻¹ 3063, 3028, 2975, 2920, 1721, 1665, 1618, 1496, 1452, 1387, 1325, 1268, 1183, 1135, 1106, 1066, 914, 738, 699

NMR (CDCl₃):

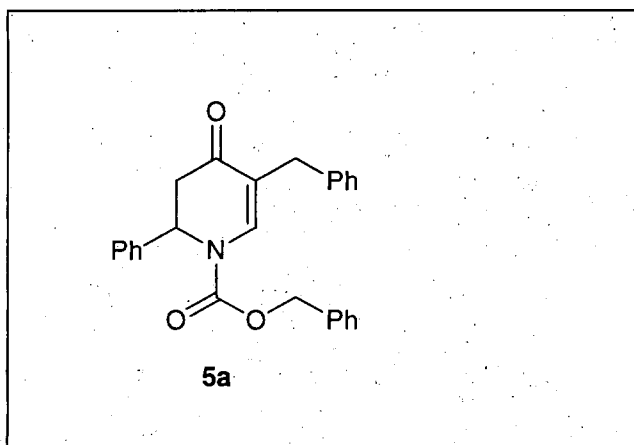
¹H-NMR (300 MHz) δ
 isomer A : 7.55 (s, 1H), 7.38-7.16 (m, 10H), 5.75-5.60 (m, 1H), 5.30-5.21 (m, 2H), 5.02-4.92 (m, 2H), 4.66 (t, 1H, J = 5.7 Hz), 4.08 (t, 1H, J = 7.8 Hz), 2.77 (dd, 1H, J = 6.6 Hz and 16 Hz), 2.64-2.58 (m, 2H), 2.31 (d, 1H, J = 16.2 Hz), 1.22 (d, 3H, J = 6.8 Hz)
 isomer B : 7.62 (s, 1H), 7.38-7.16 (m, 10H), 5.75-5.60 (m, 1H), 5.30-5.21 (m, 2H), 5.02-4.92 (m, 2H), 4.66 (t, 1H, J = 5.7 Hz), 3.97 (t, 1H, J = 7.9 Hz), 2.85-2.78 (m, 1H), 2.64-2.58 (m, 2H), 2.29 (d, 1H, J = 16.2 Hz), 1.14 (d, 1H, J = 6.7 Hz)
¹³C-NMR (75 MHz) δ
 isomer A : 191.65, 152.53, 142.86, 138.41, 136.64, 135.19, 128.67, 128.34, 128.05, 127.84, 126.31, 121.05, 116.34, 68.70, 49.10, 42.11, 38.69, 16.81
 isomer B : 191.74, 152.53, 143.36, 138.11, 136.55, 135.19, 128.59, 128.21, 128.05, 127.74, 126.20, 121.06, 116.41, 68.78, 49.10, 41.49, 38.08, 16.95

Literature References:

Compound Name: 5-Benzyl-4-oxo-2-phenyl-3,4-dihydro-2*H*-pyridine-1-carboxylic acid
benzyl ester

Compound No: 5a

Notebook Pg(s): 41w



Formula: C₂₆H₂₃NO₃

Molecular Weight: 397.47

Yield(s): 77%

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%____
Calculated:				
Found:				

HRMS: Calculated 398.1756

Found 398.1749

IR (thin film): cm⁻¹

3062, 3029, 1722, 1669, 1625, 1496, 1453, 1406, 1386, 1318, 1253, 1199, 1119,
1023, 963, 737, 697

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

7.73 (s, 1H), 7.30-7.08 (m, 15H), 5.70 (d, 1H, *J* = 6.5 Hz), 5.22-5.13 (m, 2H),
3.50 (dd, 2H, *J* = 15.3 Hz and 25 Hz), 3.12 (dd, 1H, *J* = 7.5 Hz and 16.6 Hz), 2.83
(d, 1H, *J* = 16.5 Hz)

¹³C-NMR (75 MHz) δ

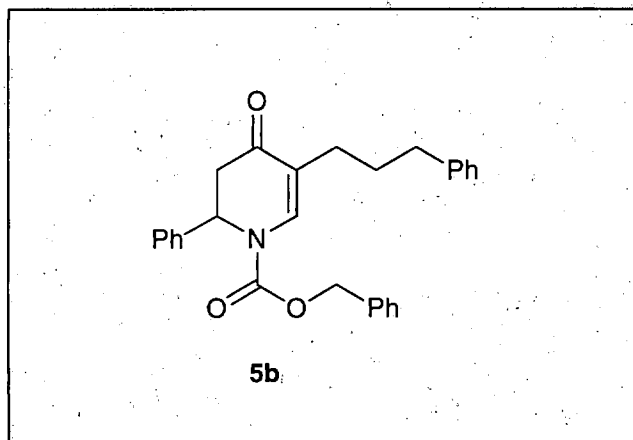
191.15, 152.68, 139.80, 139.42, 138.35, 134.91, 128.69, 128.57, 128.53, 128.45,
128.30, 127.93, 127.84, 126.04, 125.90, 121.22, 119.27, 68.77, 55.88, 41.75, 32.85

Literature References:

Compound Name: 4-Oxo-2-phenyl-5-(3-phenyl-propyl)-3,4-dihydro-2*H*-pyridine-1-carboxylic acid benzyl ester

Compound No: **5b**

Notebook Pg(s): 40y



Formula: C₂₈H₂₇NO₃

Molecular Weight: 425.52

Yield(s): 72%

Appearance: clear oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 426.2069

Found 426.2090

IR (thin film): cm⁻¹

3062, 3028, 2933, 1721, 1667, 1625, 1496, 1453, 1405, 1385, 1304, 1251, 1197, 1117, 1019, 960, 748, 697

NMR (CDCl₃): 300 MHz

¹H-NMR (300 MHz) δ

7.77 (s, 1H), 7.29-7.07 (m, 15H), 5.68 (d, 1H, *J* = 5.5 Hz), 5.25-5.16 (m, 2H), 3.05 (dd, 1H, *J* = 7.4 Hz and 16.5 Hz), 2.80 (d, 1H, *J* = 16.3 Hz), 2.51 (t, 2H, *J* = 7.7 Hz), 2.30-2.08 (m, 2H), 1.81-1.60 (m, 2H)

¹³C-NMR (75 MHz) δ

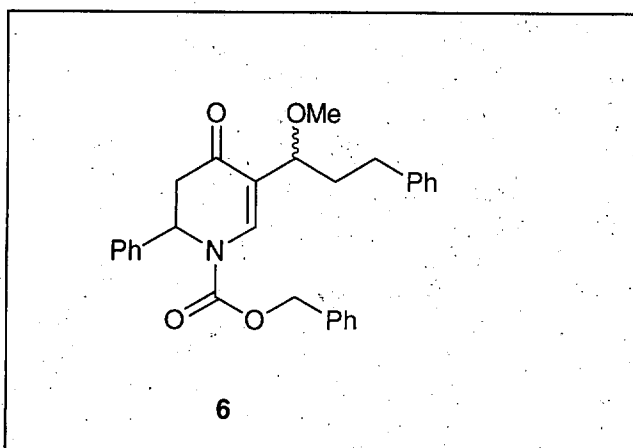
191.52, 152.70, 141.89, 138.59, 138.35, 134.94, 128.61, 128.45, 128.23, 128.07, 127.71, 125.72, 125.51, 119.24, 68.69, 55.62, 41.64, 35.18, 30.47, 26.88

Literature References:

Compound Name: 5-(1-Methoxy-3-phenyl-propyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 6

Notebook Pg(s): 53y



Formula: C₂₉H₂₉NO₄

Molecular Weight: 455.54

Yield(s): 100%

Appearance: oil

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 456.2175

Found 456.2153

IR (thin film): cm⁻¹

3062, 3028, 2930, 2821, 1726, 1665, 1623, 1496, 1453, 1407, 1387, 1295, 1251, 1198, 1105, 1021, 963, 750, 698

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

isomer A : 8.04 (s, 1H), 7.32-7.05 (m, 15H), 5.71 (d, 1H, *J* = 7.11 Hz), 5.67-5.21 (m, 2H), 4.20-4.15 (m, 1H), 3.26 (s, 3H), 3.24-3.11 (m, 1H), 2.89-2.46 (m, 3H), 1.92-1.77 (m, 2H)

isomer B : 8.01 (s, 1H), 7.32-7.05 (m, 15H), 5.76 (d, 1H, *J* = 6.8 Hz), 5.67-5.21 (m, 2H), 4.20-4.15 (m, 1H), 3.16 (s, 3H), 3.04 (dd, 1H, *J* = 7.31 Hz and 16.4 Hz), 2.89-2.46 (m, 3H), 1.92-1.77 (m, 2H)

¹³C-NMR (75 MHz) δ

isomer A : 190.97, 152.83, 141.92, 139.75, 138.06, 134.95, 128.81, 128.61, 128.38, 128.20, 127.94, 125.70, 119.23, 75.58, 69.07, 56.88, 41.65, 37.81, 31.39

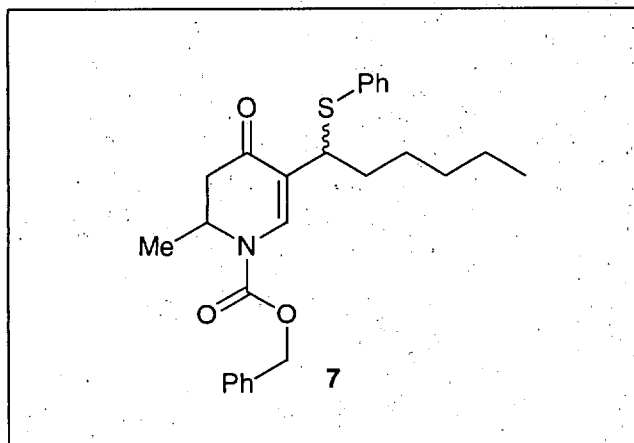
isomer B : 190.89, 152.83, 141.85, 139.55, 138.18, 134.95, 128.81, 128.61, 128.38, 128.20, 127.94, 125.70, 119.23, 75.58, 69.07, 55.87, 42.09, 37.00, 31.92

Literature References:

Compound Name: 2-Methyl-4-oxo-5-(1-phenylsulfanyl-hexyl)-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 7

Notebook Pg(s): 61y



Formula: C₂₆H₃₁NO₃S

Molecular Weight: 437.60

Yield(s): 73% d.r. = 1/2.2

Appearance: oil

Stability:

mp/bp:

[α]_D²³:

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%S
Calculated:				
Found:				

HRMS: Calculated 437.2025

Found 437.2011

IR (thin film): cm⁻¹ 3063, 3034, 2956, 2929, 2857, 1726, 1667, 1619, 1456, 1405, 1384, 1311, 1266, 1187, 1135, 1107, 1066, 741, 696

NMR (CDCl₃):

¹H-NMR (300 MHz) δ isomer A : 7.78 (s, 1H), 7.39-7.14 (m, 10H), 5.30-5.19 (m, 2H), 4.66-4.59 (m, 1H), 4.38 (dd, 1H, J = 7.4 Hz and 14.6 Hz), 2.68 (dd, 1H, J = 6.6 Hz and 16.3 Hz), 2.30 (d, 1H, J = 16.2 Hz), 1.75-1.68 (m, 2H), 1.36-1.26 (m, 6H), 1.17 (d, 1H, J = 6.8 Hz), 0.85-0.83 (m, 3H)

isomer B : 7.73 (s, 1H), 7.39-7.14 (m, 10H), 5.30-5.19 (m, 2H), 4.66-4.59 (m, 1H), 4.38 (dd, 1H, J = 7.4 Hz and 14.6 Hz), 2.79 (dd, 1H, J = 6.7 Hz and 16.5 Hz), 2.30 (d, 1H, J = 16.2 Hz), 1.75-1.68 (m, 2H), 1.36-1.26 (m, 6H), 0.99 (d, 1H, J = 6.8 Hz), 0.85-0.83 (m, 3H)

¹³C-NMR (75 MHz) δ

isomer A : 191.12, 152.20, 139.27, 135.13, 131.73, 128.67, 128.23, 126.71, 118.00, 68.83, 49.12, 43.41, 41.71, 35.57, 31.38, 27.14, 22.43, 16.76, 13.94

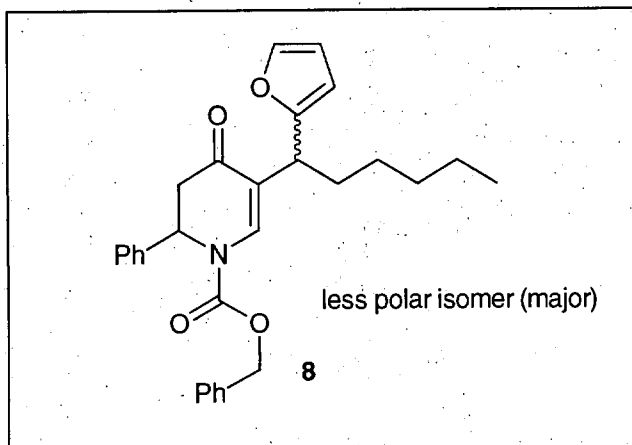
isomer B : 191.02, 152.20, 139.55, 135.13, 131.61, 128.67, 128.23, 126.71, 118.00, 68.83, 49.12, 43.04, 41.88, 35.01, 31.38, 27.14, 22.43, 16.56, 13.94

Literature References:

Compound Name:

Compound No: 8-major

Notebook Pg(s): 70y, 71y

Formula: C₂₉H₃₁NO₄

Molecular Weight: 457.56

Yield(s): 80% d.r. = 1/3.2

Appearance: colorless oil

Stability: stable

mp/bp:

[α]_D²³Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	76.12	6.83	3.06	
Found:				

HRMS: Calculated : 458.2331

Found : 458.2324

IR (thin film): cm⁻¹

2929, 2859, 1725, 1667, 1622, 1499, 1453, 1386, 1298, 1252, 1196, 1118, 1020, 960, 736, 696

NMR (CDCl₃):¹H-NMR (300 MHz) δ

7.83 (s, 1H), 7.33-7.19 (m, 11H), 6.28 (s, 1H), 6.05 (d, 1H, J = 2.9 Hz), 5.71 (d, 1H, J = 6.2 Hz), 5.26-5.17 (m, 2H), 4.02 (t, 1H, J = 7.6 Hz), 3.14 (dd, 1H, J = 7.3 and 16.4 Hz), 2.85 (d, 1H, J = 16.5 Hz), 1.80-1.60 (m, 2H), 1.26-1.11 (m, 6H), 0.84-0.80 (m, 3H)

¹³C-NMR (75 MHz) δ

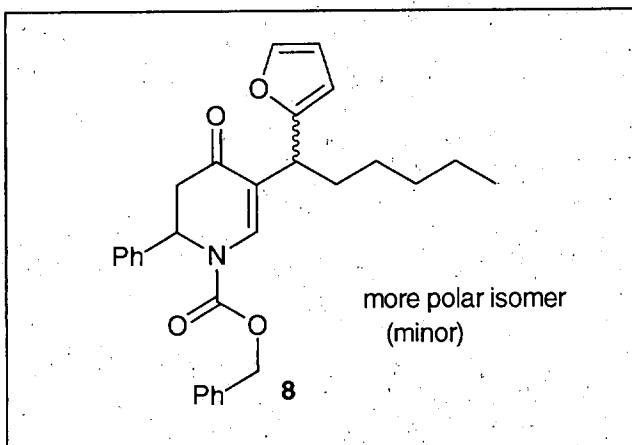
190.45, 156.36, 152.84, 141.21, 139.25, 138.33, 135.00, 128.73, 128.54, 128.47, 127.86, 125.86, 121.01, 110.00, 105.66, 68.79, 55.64, 41.75, 35.00, 34.06, 31.54, 27.08, 22.46, 14.00

Literature References:

Compound Name:

Compound No: **8**-minor

Notebook Pg(s): 70y, 71y

Formula: C₂₉H₃₁NO₄

Molecular Weight: 457.56

Yield(s): 80% d.r. = 1/3.2

Appearance: colorless oil

Stability: stable

mp/bp:

[α]_D²³Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:	76.12	6.83	3.06	
Found:				

HRMS: Calculated : 458.2331

Found : 458.2315

IR (thin film): cm⁻¹

3032, 2927, 2858, 1725, 1669, 1623, 1499, 1453, 1386, 1302, 1252, 1197, 1118, 1020, 961, 736, 697

NMR (CDCl₃):¹H-NMR (300 MHz) δ

7.88 (s, 1H), 7.33-7.09 (m, 11H), 6.23 (d, 1H, J = 2 Hz), 5.96 (d, 1H, J = 3 Hz), 5.67 (d, 1H, J = 7 Hz), 5.21 (s, 2H), 3.97 (t, 1H, J = 7.7 Hz), 3.16 (dd, 1H, J = 7.5 and 16.4 Hz), 2.80 (d, 1H, J = 16.4 Hz), 1.87-1.67 (m, 2H), 1.24 (s, 6H), 0.89-0.82 (m, 3H)

¹³C-NMR (75 MHz) δ

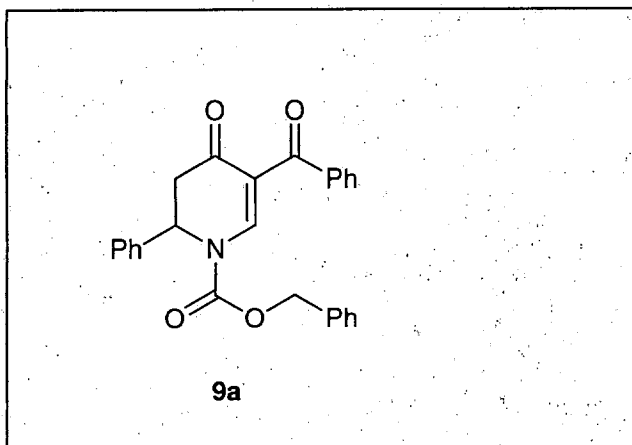
190.51, 156.65, 152.84, 140.98, 139.69, 138.46, 135.00, 128.73, 128.60, 128.54, 128.02, 127.89, 125.92, 120.46, 110.02, 105.40, 68.86, 55.97, 42.10, 35.61, 32.41, 31.57, 27.24, 22.46, 13.99

Literature References:

Compound Name: 5-Benzoyl-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid
benzyl ester

Compound No: 9a

Notebook Pg(s): 44y, 52y



Formula: C₂₆H₂₁NO₄

Molecular Weight: 411.45

Yield(s): 62%

Appearance:

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 412.1549

Found 412.1555

IR (thin film): cm⁻¹

3063, 3033, 2961, 2916, 1740, 1676, 1654, 1598, 1580, 1497, 1449, 1384, 1365,
1312, 1264, 1240, 1190, 1176, 1158, 1116, 1057, 1026, 972, 762, 730, 698

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

8.67 (s, 1H), 7.60-7.22 (m, 15H), 5.80 (d, 1H, *J* = 7.2 Hz), 5.28 (dd, 2H, *J* =
11.9 Hz and 18.9 Hz), 3.26 (dd, 1H, *J* = 7.5 Hz and 16.1 Hz), 2.87 (dd, 1H, *J* =
1.4 Hz and 16.1 Hz)

¹³C-NMR (75 MHz) δ

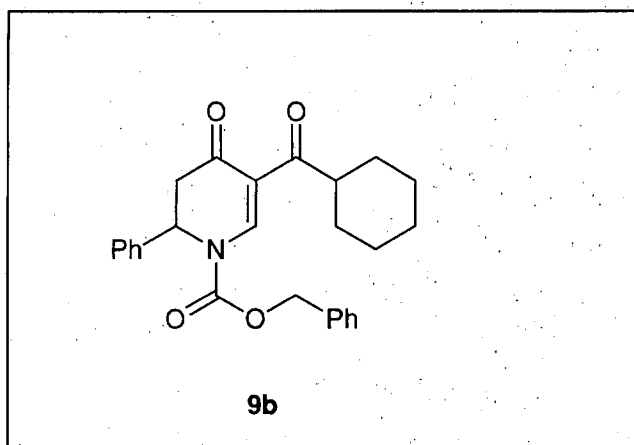
191.97, 188.16, 152.18, 148.10, 138.09, 137.59, 134.21, 132.62, 129.22, 129.15,
128.93, 128.72, 128.44, 128.04, 125.66, 118.92, 69.99, 56.70, 42.38

Literature References:

Compound Name: 5-Cyclohexanecarbonyl-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid
benzyl ester

Compound No: **9b**

Notebook Pg(s): 52w



Formula: C₂₆H₂₇NO₄

Molecular Weight: 417.50

Yield(s): 50%

Appearance:

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%____
Calculated:				
Found:				

HRMS: Calculated 418.2018

Found 418.2006

IR (thin film): cm⁻¹

3064, 2929, 2852, 1743, 1674, 1561, 1497, 1450, 1402, 1381, 1358, 1335, 1297, 1237, 1216, 1167, 1139, 1113, 1084, 1068, 1028, 989, 964, 763, 697

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

8.93 (s, 1H), 7.36-7.11 (m, 10H), 5.70 (d, 1H, *J* = 7.4 Hz), 5.27 (dd, 2H, *J* = 12 Hz and 21.8 Hz), 3.38-3.31 (m, 1H), 3.18 (dd, 1H, *J* = 7.7 Hz and 15.9 Hz), 2.77 (d, 1H, *J* = 15.9 Hz), 1.82-1.15 (m, 10H)

¹³C-NMR (75 MHz) δ

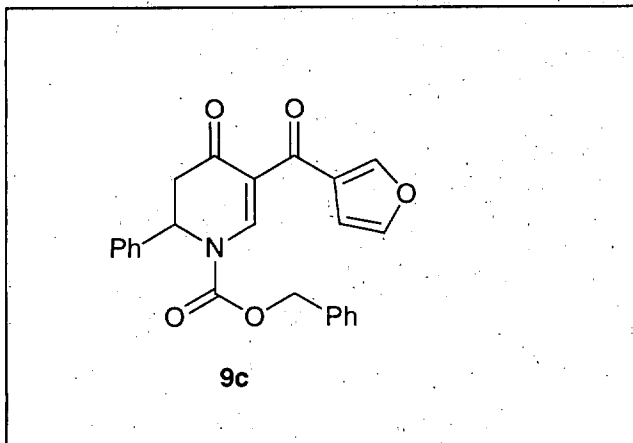
200.98, 188.83, 152.20, 149.87, 137.70, 134.20, 129.08, 128.94, 128.73, 128.51, 128.38, 125.55, 116.57, 70.04, 56.95, 48.33, 42.95, 28.66, 26.07, 25.79

Literature References:

Compound Name: 5-(Furan-3-carbonyl)-4-oxo-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: **9c**

Notebook Pg(s): 45w, 51y



Formula: C₂₄H₁₉NO₅

Molecular Weight: 401.41

Yield(s): 50%

Appearance:

Stability:

mp/bp:

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%O
Calculated:				
Found:				

HRMS: Calculated 402.1341

Found 402.1365

IR (thin film): cm⁻¹

3131, 3064, 3033, 2962, 1740, 1678, 1641, 1561, 1498, 1454, 1389, 1321, 1286, 1240, 1153, 1116, 1061, 1026, 963, 944, 872, 823, 755, 698

NMR (CDCl₃):

¹H-NMR (300 MHz) δ

8.72 (s, 1H), 7.83 (s, 1H), 7.37-7.18 (m, 1H), 6.71 (s, 1H), 5.77 (d, 1H, *J* = 7.2 Hz), 5.28 (dd, 2H, *J* = 12 Hz and 19.3 Hz), 3.24 (dd, 1H, *J* = 7.5 Hz and 16.1 Hz), 2.87 (d, 1H, *J* = 16 Hz)

¹³C-NMR (75 MHz) δ

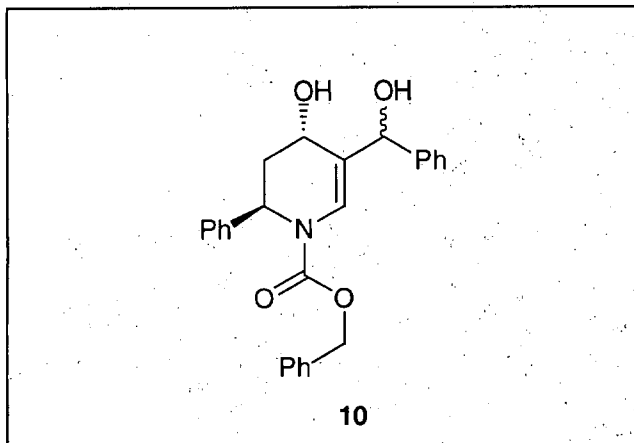
188.08, 183.65, 152.15, 149.12, 148.13, 143.16, 137.53, 134.18, 129.14, 128.95, 128.73, 128.47, 127.32, 125.57, 119.32, 109.87, 70.03, 56.59, 42.51

Literature References:

Compound Name: 4-Hydroxy-5-(hydroxy-phenyl-methyl)-2-phenyl-3,4-dihydro-2H-pyridine-1-carboxylic acid benzyl ester

Compound No: 10

Notebook Pg(s): 62y



Formula: C₂₆H₂₅NO₄

Molecular Weight: 415.48

Yield(s): 77%

Appearance: white solid

Stability:

mp/bp: 93-95°C

[α]_D²³

Elemental
Analysis
(Atlantic Microlabs, Inc.)

	%C	%H	%N	%__
Calculated:				
Found:				

HRMS: Calculated 415.1784

Found 415.1794

IR (thin film): cm⁻¹ 3424 (broad), 2957, 1708, 1666, 1496, 1452, 1404, 1338, 1241, 1157, 1119, 1068, 1010, 984, 746, 698

NMR (CDCl₃):

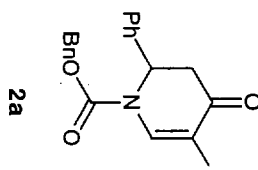
¹H-NMR (300 MHz) δ 7.33-7.02 (m, 16H), 5.44 (d, 1H, *J* = 1.9 Hz), 5.27 (s, 1H), 5.03 (dd, 2H, *J* = 12.6 Hz and 15.8 Hz), 3.94-3.87 (m, 1H), 3.18 (s, 1H), 2.73 (d, 1H, *J* = 5.2 Hz), 2.27-2.22 (m, 1H), 2.13-2.01 (m, 1H)

¹³C-NMR (75 MHz) δ 153.33, 141.68, 140.90, 135.73, 128.53, 128.39, 127.96, 127.71, 127.49, 127.11, 126.71, 124.95, 123.75, 122.16, 73.25, 67.70, 62.92, 54.76, 37.52

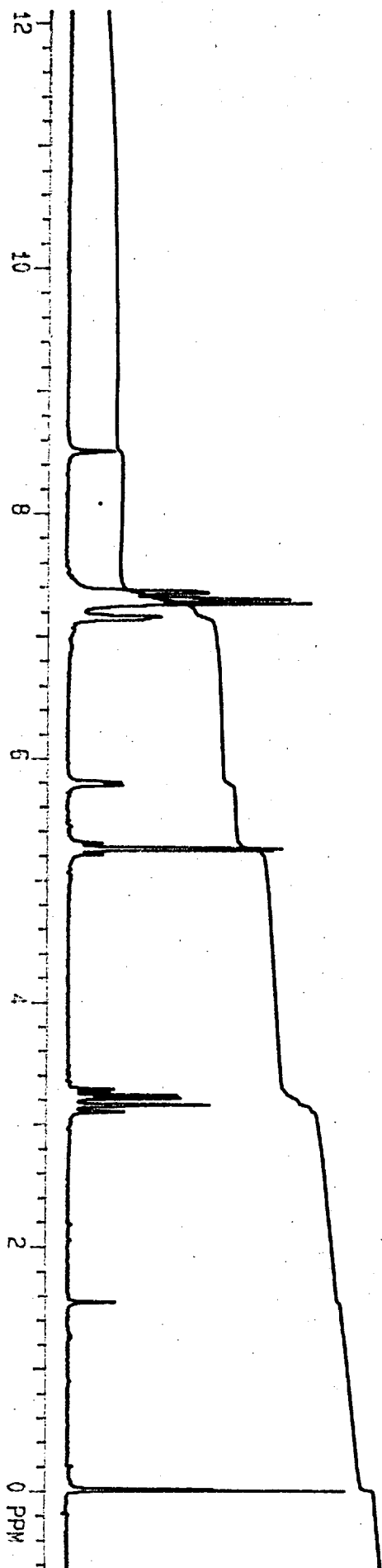
Literature References:

"Synthesis and Reactivity of N-Acyl-5-(1-hydroxyalkyl)-2,3-dihydro-4-pyridones"

D. L. Comins*, Anne-Cecile Hiebel and Shenlin Huang
Dept. of Chemistry, NCSU, Raleigh, NC

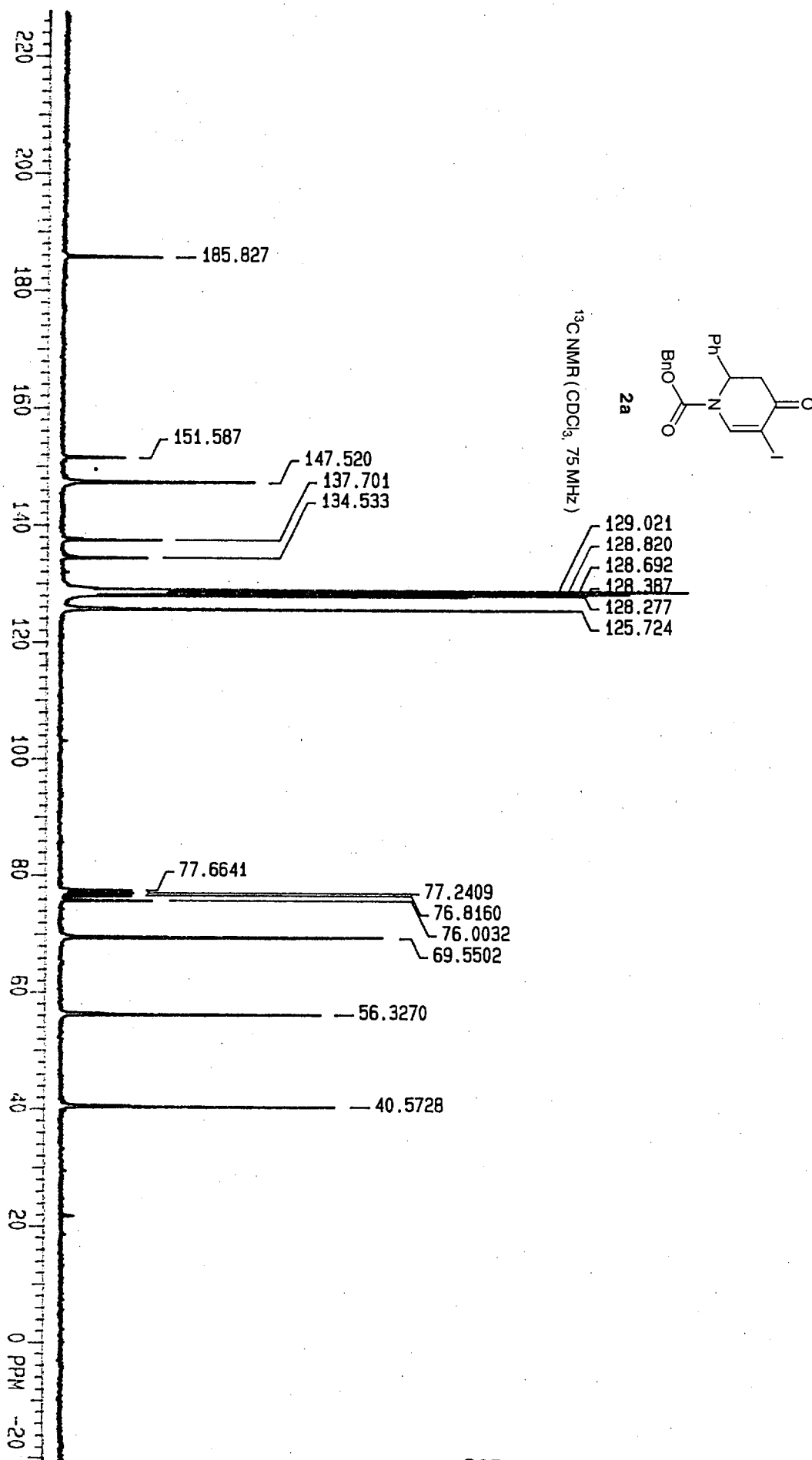


^1H NMR (CDCl_3 , 300 MHz)



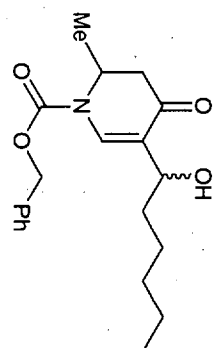
"Synthesis and Reactivity of N-Acyl-5-(1-hydroxyalkyl)-2,3-dihydro-4-pyridones"

D. L. Comins*, Anne-Cecile Hiebel and Shenlin Huang
Dept. of Chemistry, NCSU, Raleigh, NC

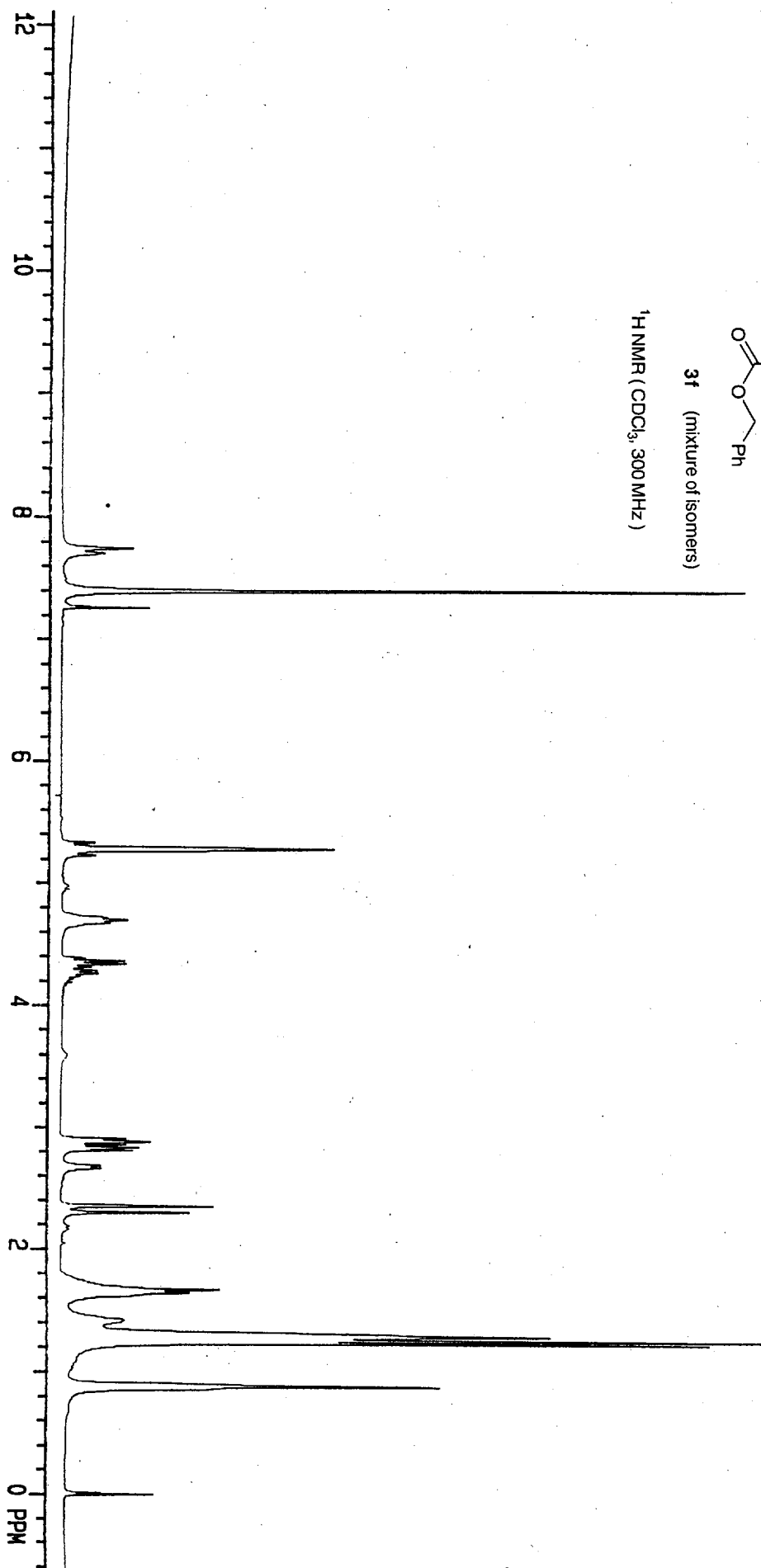


"Synthesis and Reactivity of N-Acyl-5-(1-hydroxyalkyl)-2,3-dihydro-4-pyridones"

D. L. Comins*, Anne-Cecile Hiebel and Shenlin Huang
Dept. of Chemistry, NCSU, Raleigh, NC

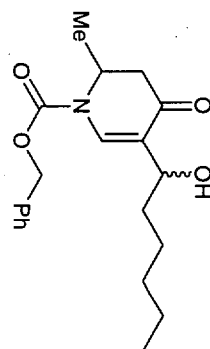


3f (mixture of isomers)
¹H NMR (CDCl₃, 300 MHz)



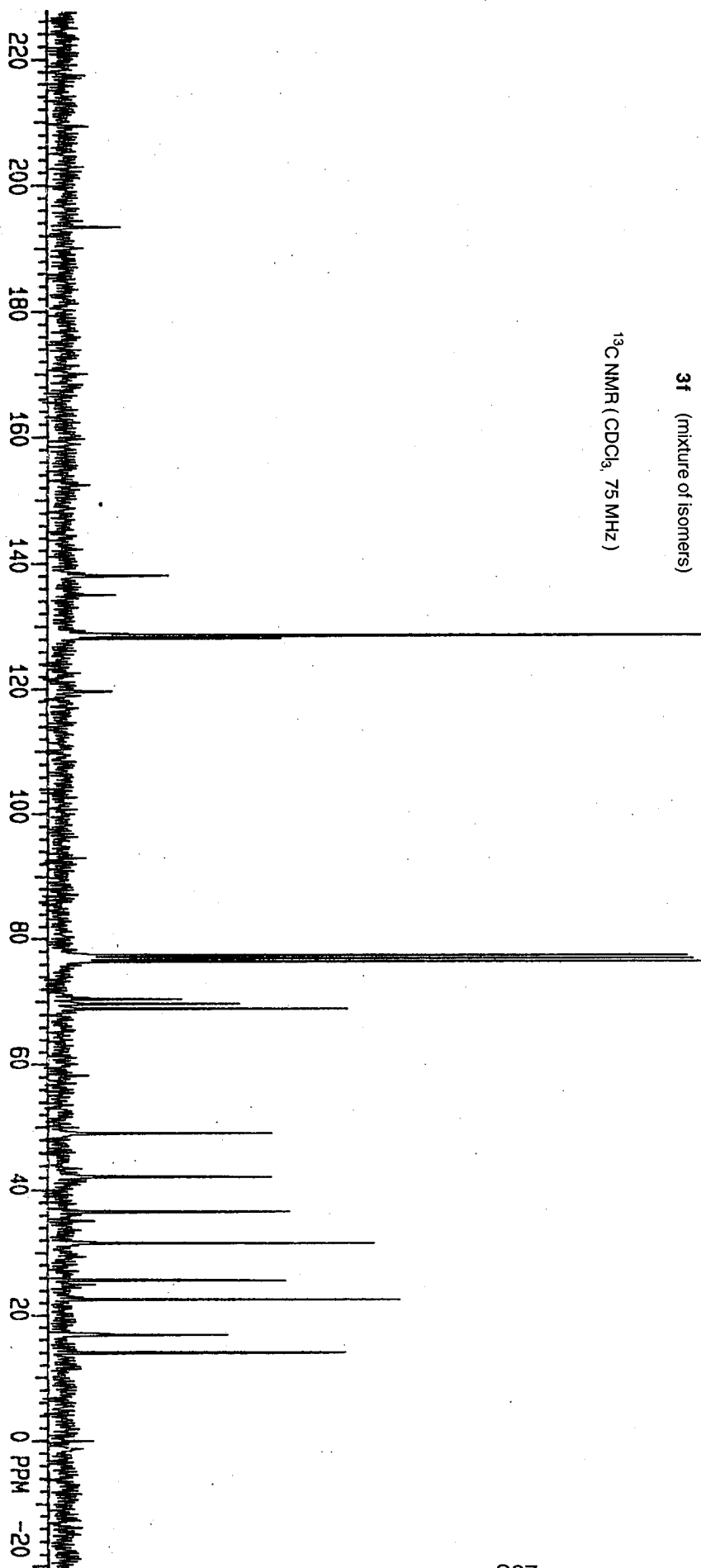
"Synthesis and Reactivity of N-Acyl-5-(1-hydroxyalkyl)-2,3-dihydro-4-pyridones"

D. L. Comins*, Anne-Cecile Hiebel and Shenlin Huang
Dept. of Chemistry, NCSU, Raleigh, NC



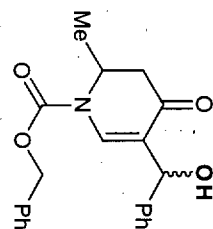
3f (mixture of isomers)

¹³C NMR (CDCl₃, 75 MHz)



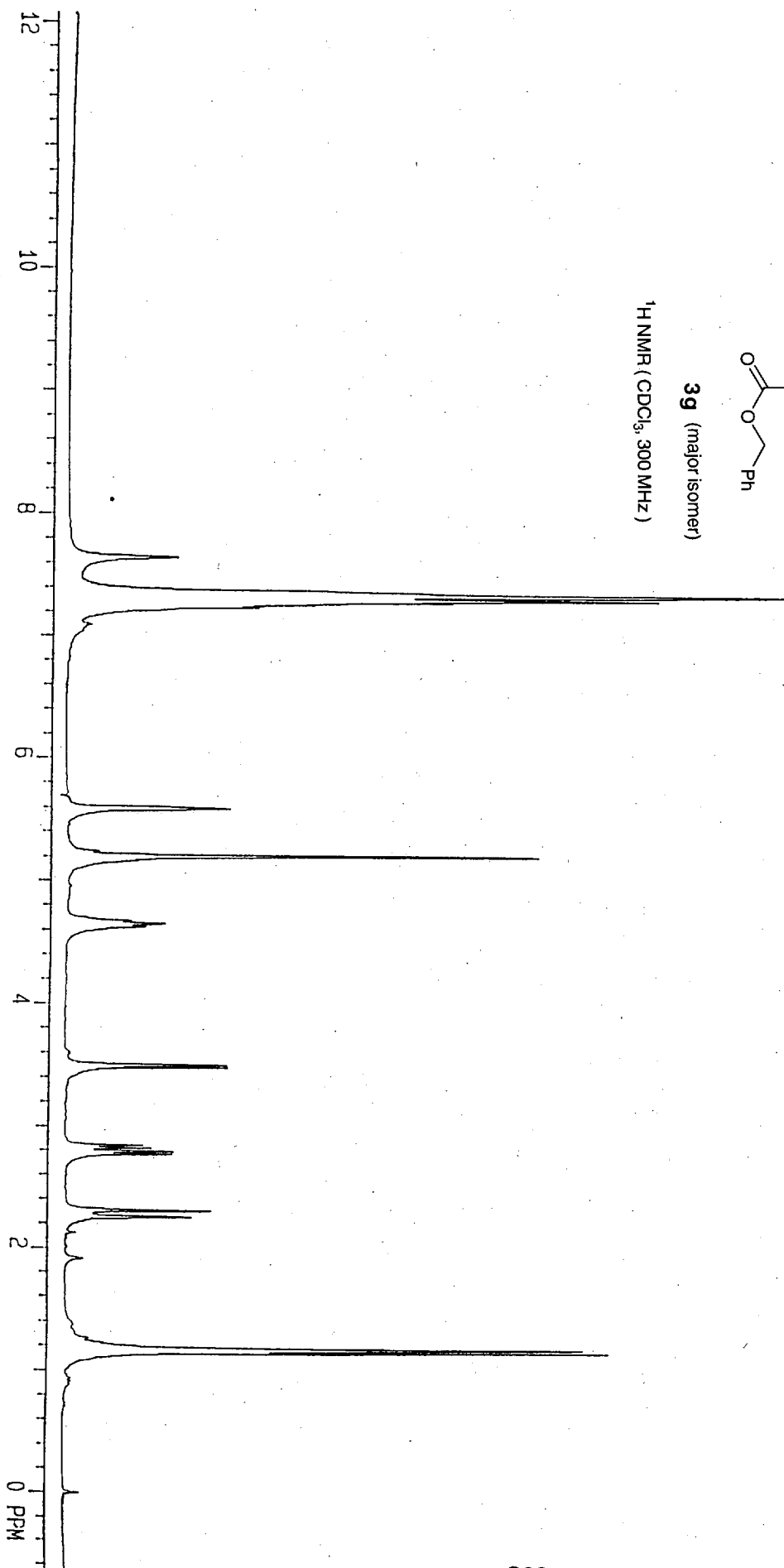
"Synthesis and Reactivity of N-Acyl-5-(1-hydroxyalkyl)-2,3-dihydro-4-pyridones"

D. L. Comins*, Anne-Cecile Hiebel and Shenlin Huang
Dept. of Chemistry, NCSU, Raleigh, NC



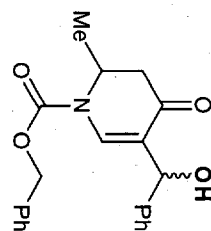
3g (major isomer)

¹H NMR (CDCl₃, 300 MHz)



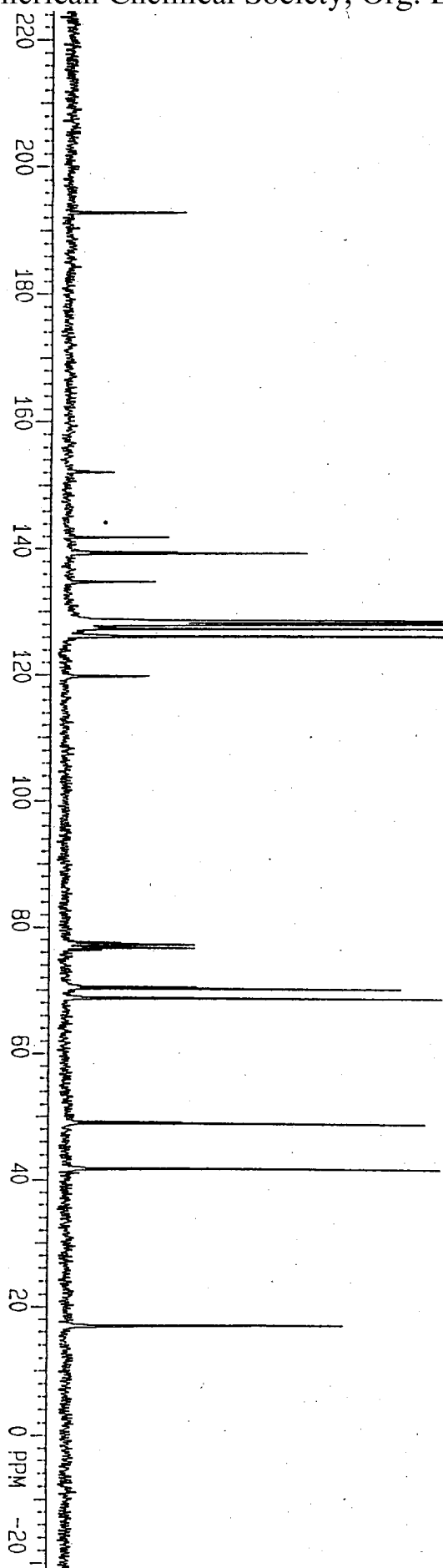
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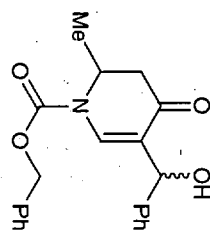
3g (major isomer)

^{13}C NMR (CDCl_3 , 75 MHz)



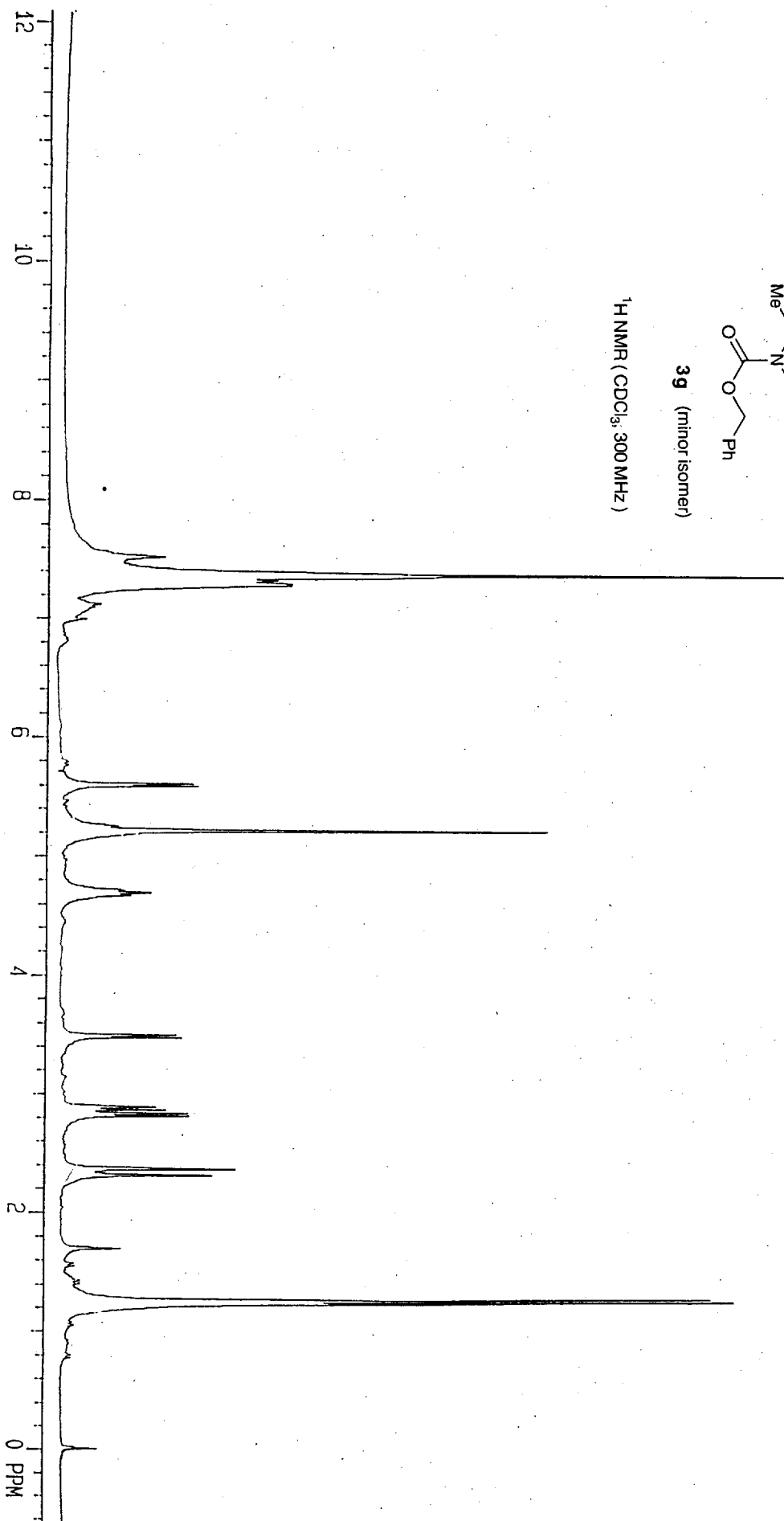
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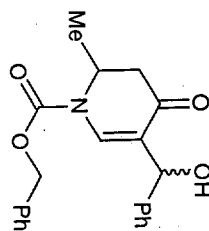
3g (minor isomer)

¹H NMR (CDCl₃, 300 MHz)



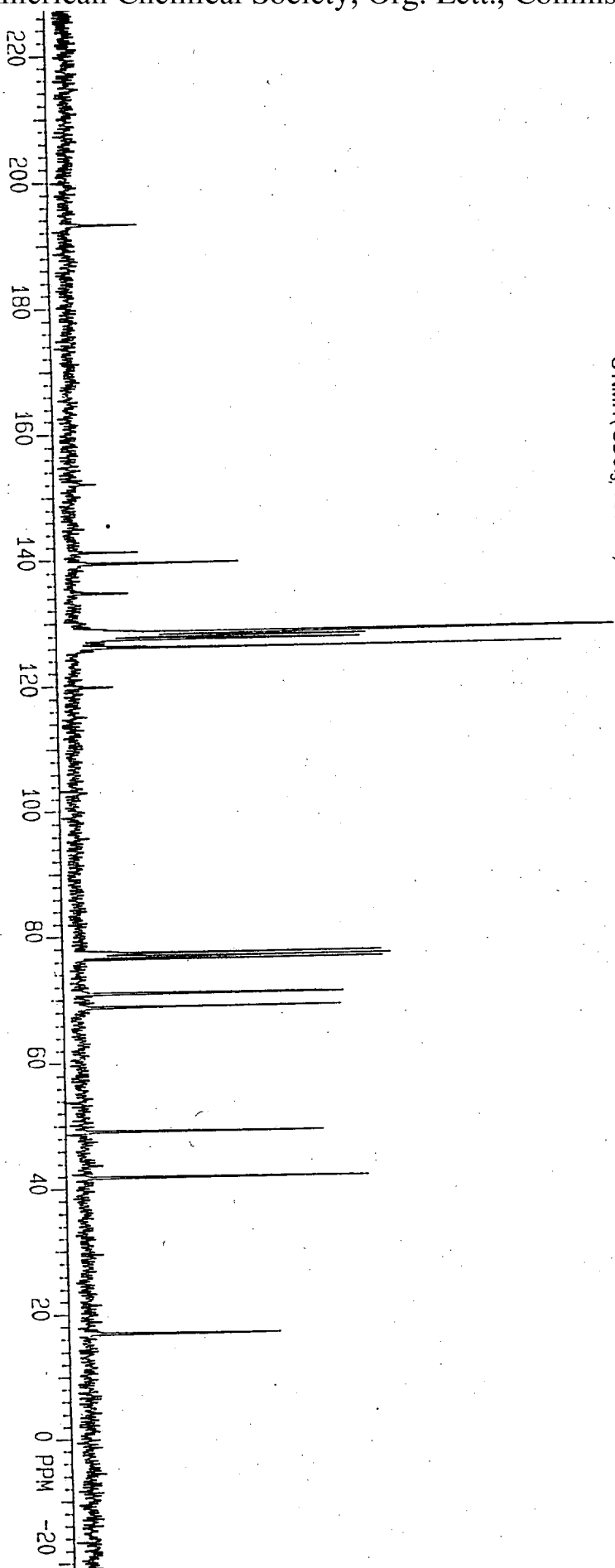
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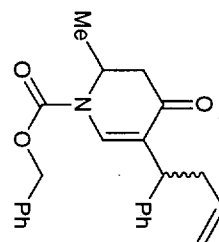
3g (minor isomer)

¹³C NMR (CDCl₃, 75 MHz)



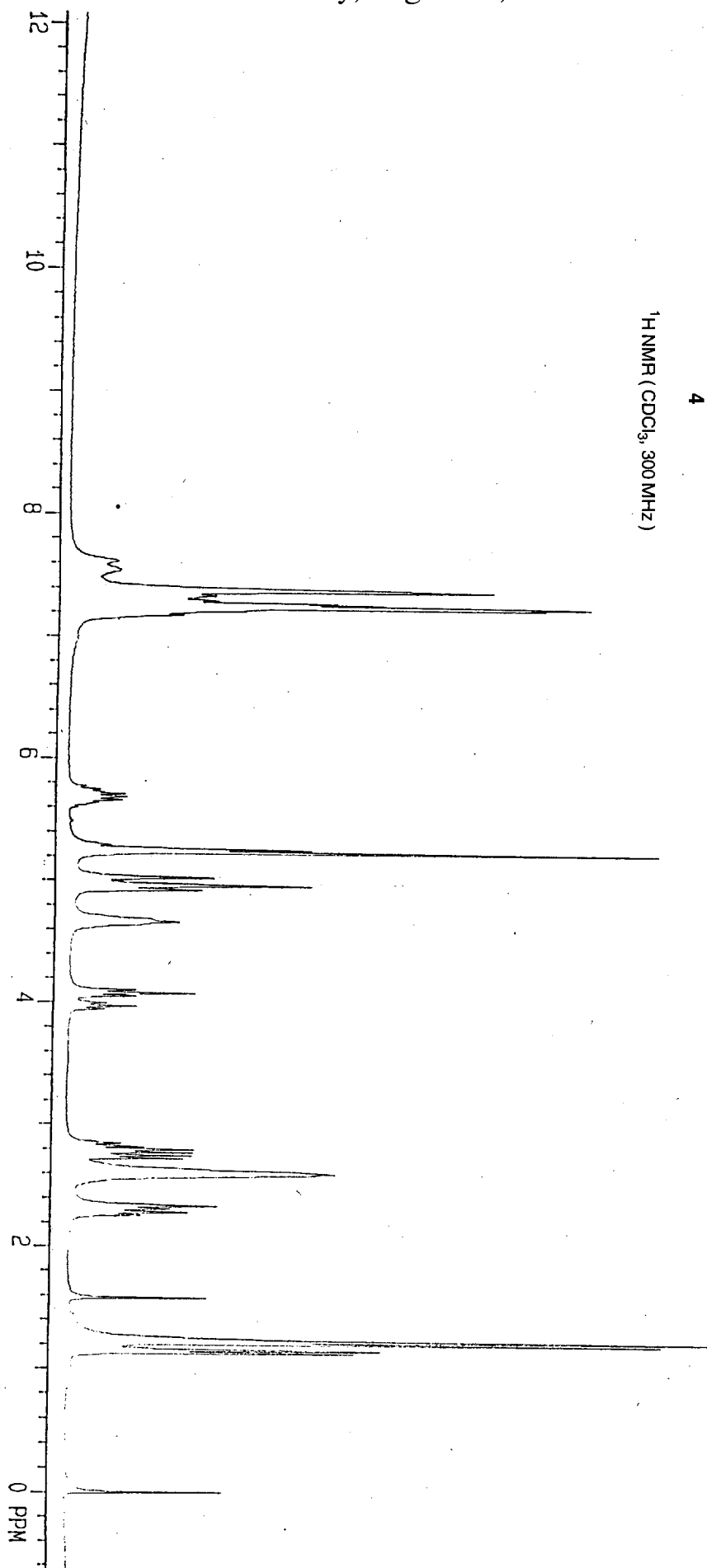
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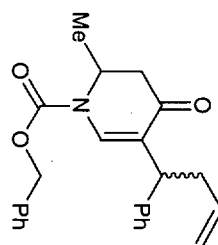
4

¹H NMR (CDCl₃, 300 MHz)



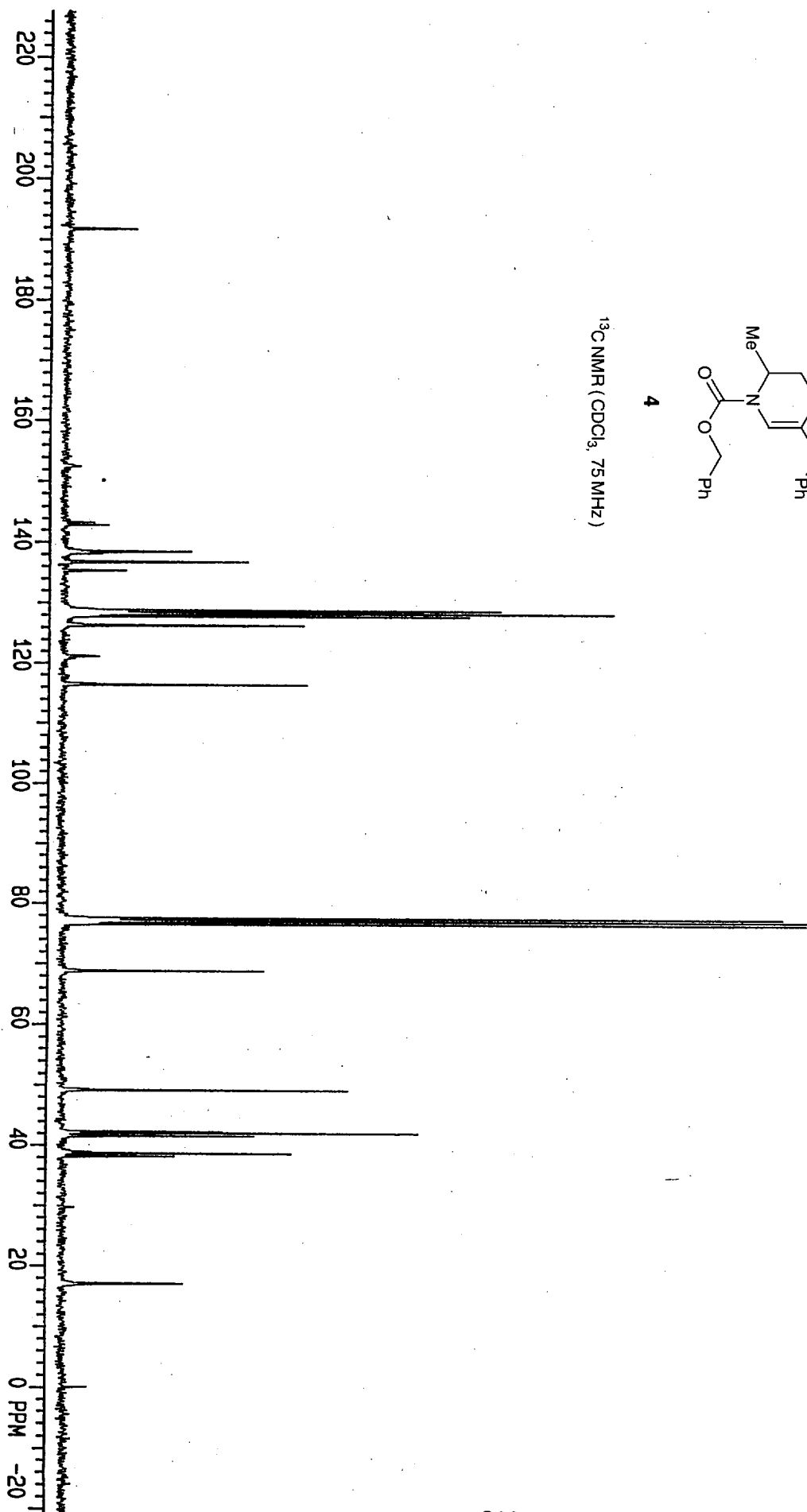
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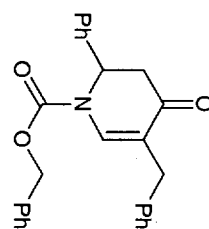
4

¹³C NMR (CDCl₃, 75 MHz)



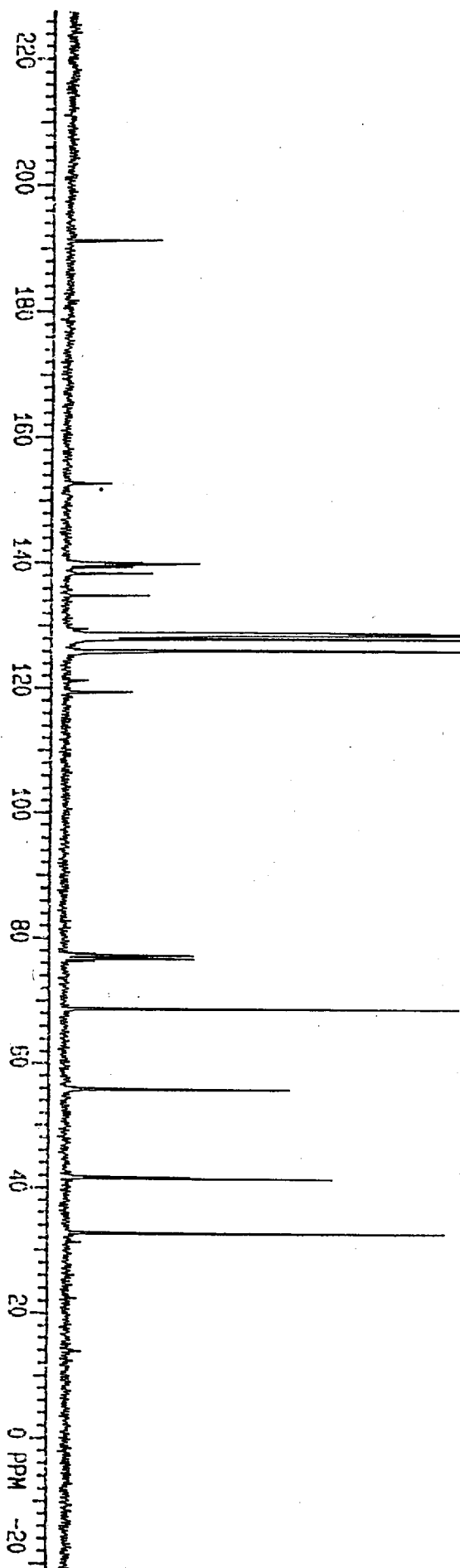
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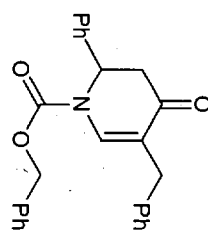
5a

^{13}C NMR (CDCl_3 , 75 MHz)



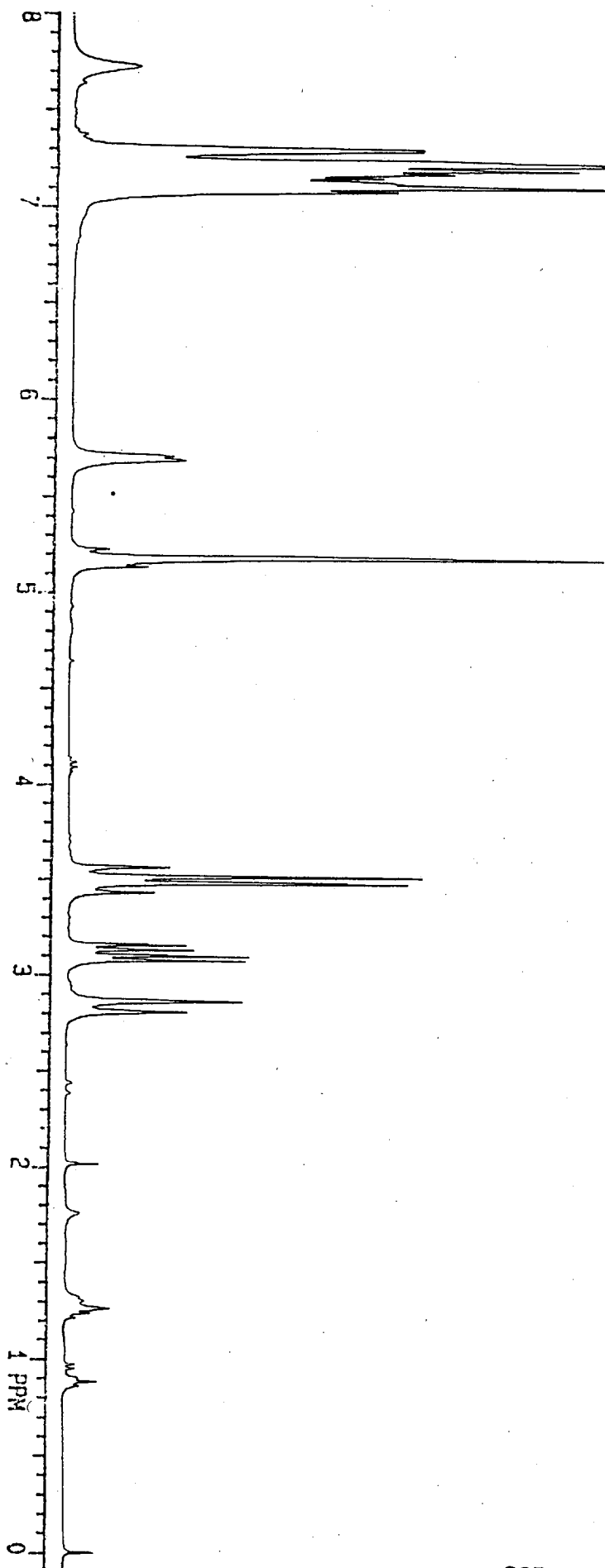
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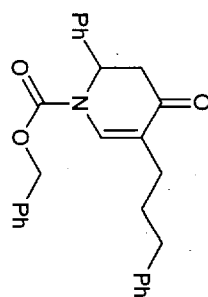


5a

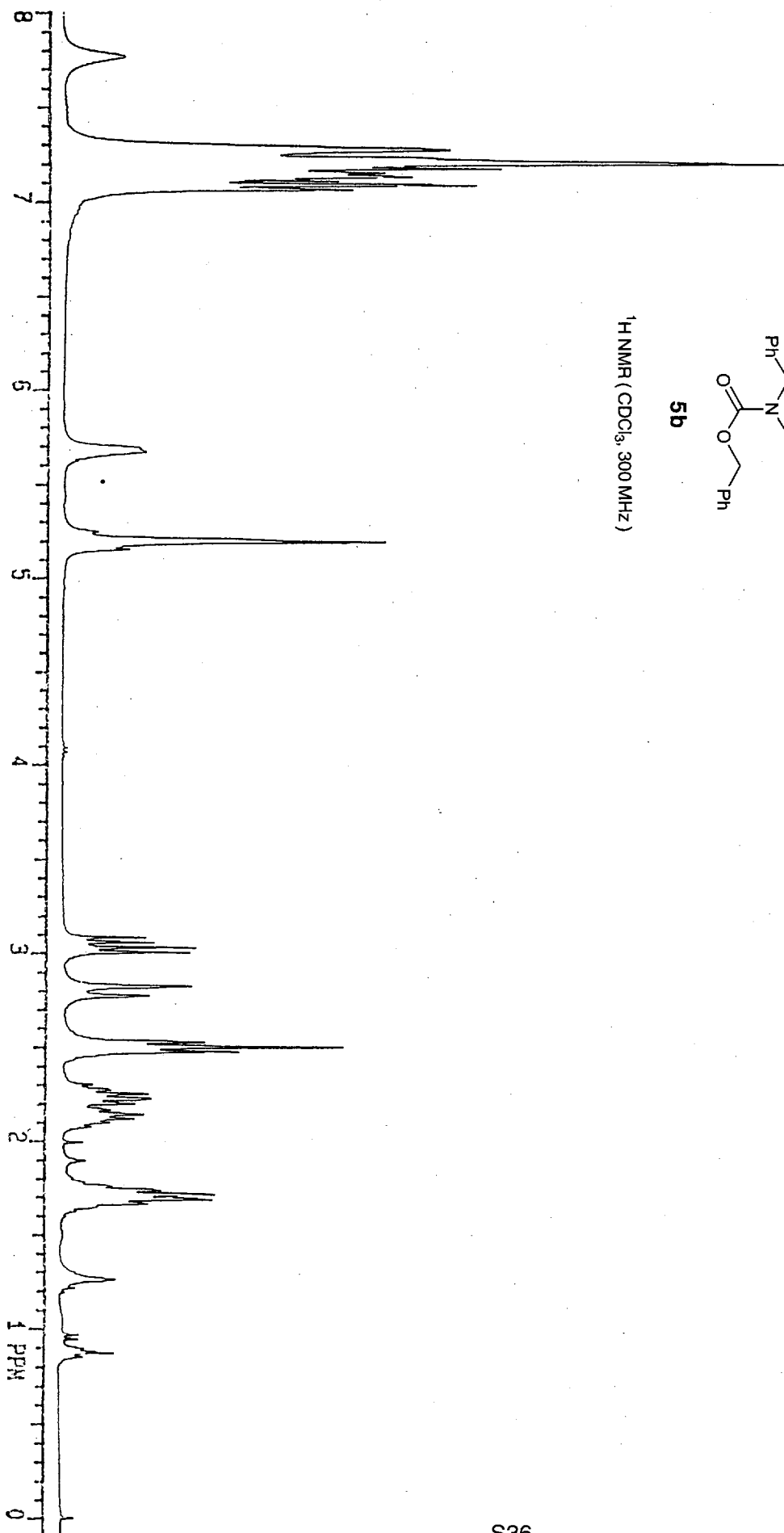
¹H NMR (CDCl₃, 300 MHz)



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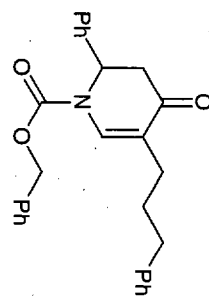


55

¹H NMR (CDCl₃, 300 MHz)

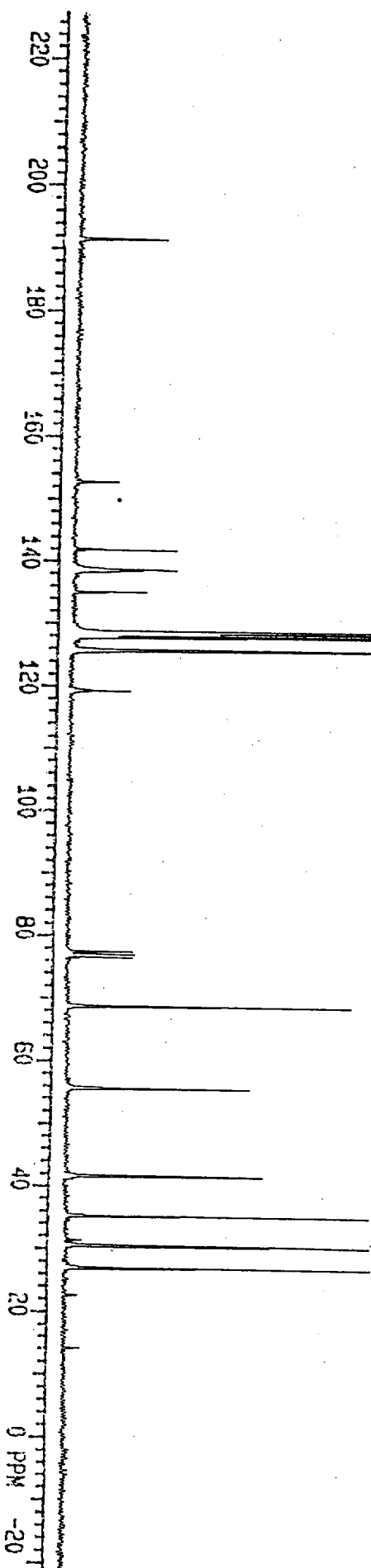
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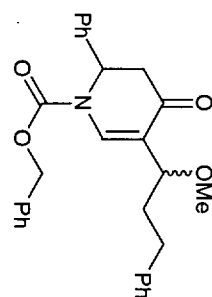
5b

^{13}C NMR (CDCl_3 , 75 MHz)



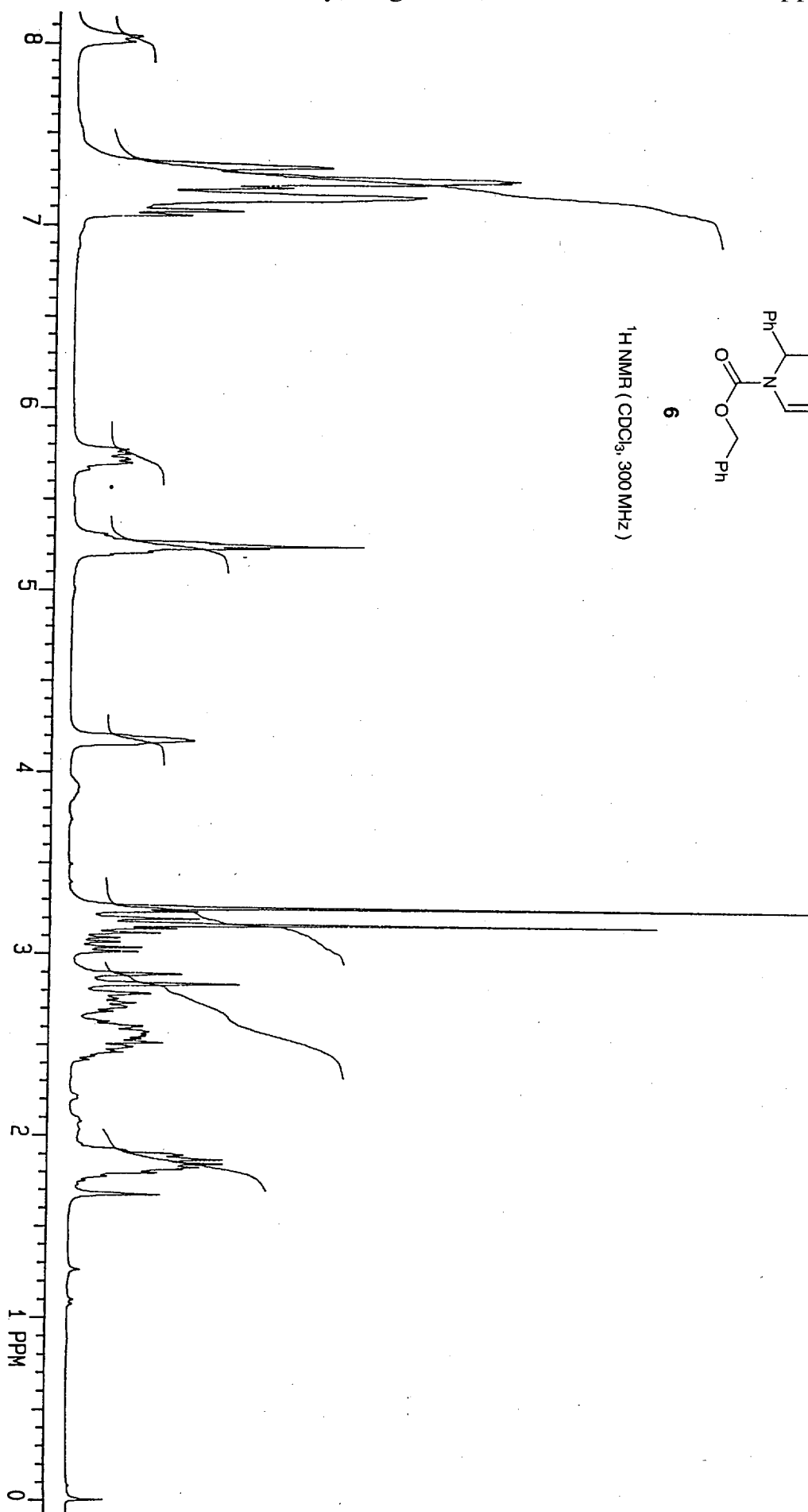
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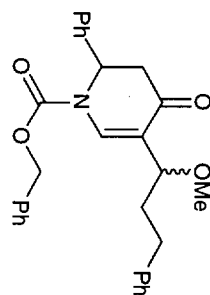
6

¹H NMR (CDCl₃, 300 MHz)



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6

^{13}C NMR (CDCl_3 , 75 MHz)

