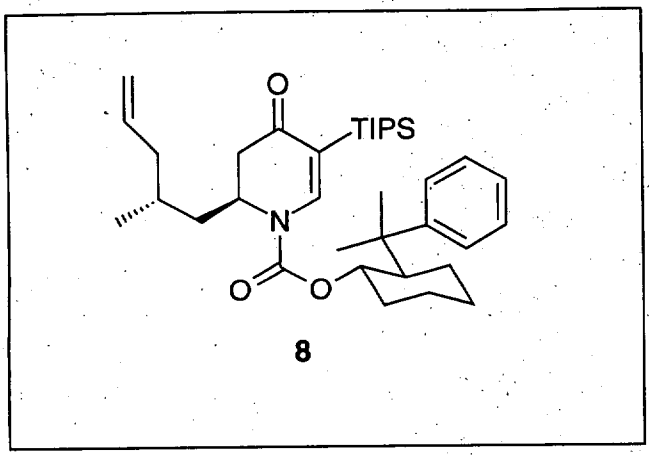
Compound Name: **(+)-(*S*)-[(*R*)-2-Methyl-4-pentenyl]-1-[(1*S*, 2*R*)-2-((1-methyl-1-phenyl) ethyl) cyclohexyl-oxycarbonyl]-5-(triisopropylsilyl)-2,3-dihydro-1*H*-pyridin-4-one**

Chemist: Clint Brooks

Formula: $C_{36}H_{57}NO_3Si$

Molecular Weight: 579.9417



8

Yields(s): 80%

Appearance: white solid

Stability: air stable

mp/bp: 116-118 °C

$[\alpha]^{23}_D$ +58.3 (*c* 0.95, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs)

	%C	%H	%N	%___
Calculated:	74.56	9.91	2.42	
Found:	74.47	9.88	2.45	

IR (thin film): 2925,2850, 1695, 1642, 1563, 1450, 1380, 1315, 1290, 1280, 1110, 1005, 910, 872, 690 cm^{-1}.

NMR ($CDCl_3$):

^1H (300 MHz):δ 7.71 (s, 1H), 7.09-7.34 (m, 5H), 5.55-5.75 (m, 1H), 4.84-5.01 (m, 3H), 2.80-2.95 (m, 1H), 2.36-2.43 (m, 1H), 2.21-2.27 (m, 1H), 0.75-2.10 (m, 44H).

^{13}C (75 MHz):δ 196.9, 152.54, 147.2, 136.2, 128.0, 125.0, 116.4, 110.28, 78.1, 50.7, 49.8, 41.1, 40.3, 39.3, 36.8, 33.2, 30.8, 28.7, 26.7, 25.9, 24.6, 21.46, 20.4, 18.8, 18.7, 11.0.

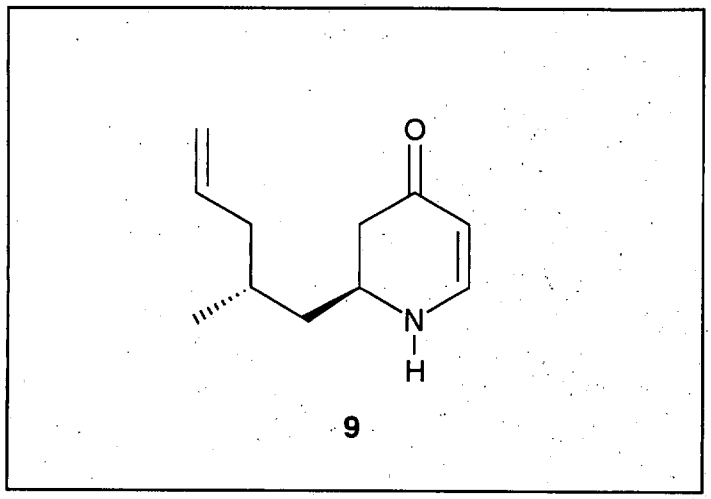
Literature References: none

Compound Name: **(-)-(*S*)-2-[(*R*)-2-Methyl-4-pentenyl]-2,3-dihydro-4-pyridone**

Chemist: Clint Brooks

Formula: $C_{11}H_{17}NO$

Molecular Weight: 179.2627



9

Yields(s): 90%

Appearance: pale yellow oil

Stability: air sensitive

mp/bp:

$[\alpha]^{23}_D$ - 292.7 (*c* 1.32, $CHCl_3$)

HRMS: Calculated: 179.13100 Found: 179.131014

IR (thin film): 3249, 3027, 2956, 2915, 1618, 1572, 1531, 1223 cm^{-1}.

NMR ($CDCl_3$):

^1H (300 MHz) :δ 7.16 (t, *J* = 6.9 Hz, 1 H), 5.75 (m, 1 H), 5.16 (bs, 1 H), 5.08-4.95 (m, 3 H), 3.82-3.70 (m, 1 H), 2.50-2.26 (m, 2 H), 2.14-1.90 (m, 2 H), 1.84-1.54 (m, 2 H) 1.40-1.24 (m, 1 H), 0.95 (d, *J* = 6.5 Hz, 3 H).

^{13}C (75 MHz):δ 193.2, 150.9, 136.4, 116.9, 99.5, 51.2, 43.0, 41.5, 41.0, 29.3, 19.5.

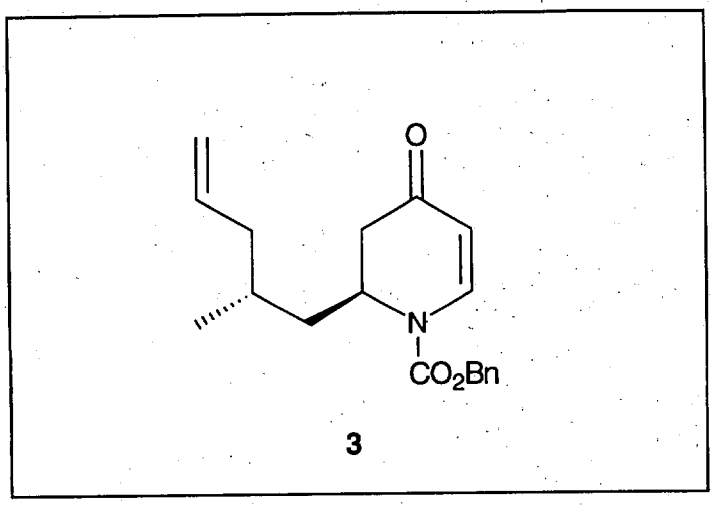
Literature References: none

Compound Name: **(-)-(*S*)-1[(Benzyloxy)carbonyl]-2-[(*R*)-2-Methyl-4-pentenyl]-2,3-dihydro-4-pyridone**

Chemist: Clint Brooks

Formula: $C_{19}H_{23}NO_3$

Molecular Weight: 313.40



3

Yields(s): 98%

Appearance: pale yellow oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ - 84.1 (*c* 1.45, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs)

	%C	%H	%N	%___
Calculated:	72.82	7.40	4.47	
Found:	72.76	7.42	4.53	

IR (thin film): 3060, 2953, 2920, 2878, 1723, 1665, 1600, 1451, 1419, 1381, 1323, 1259, 1189, 1109 cm^{-1}.

NMR ($CDCl_3$):

^1H (300 MHz) :δ 7.75 (d, *J* = 5.6 Hz, 1 H), 7.43-7.35 (m, 5 H), 5.76-5.56 (m, 1 H), 5.38-5.20 (m, 3 H), 5.20-4.90 (m, 2 H), 4.80-4.62 (m, 1 H), 2.82 (dd, *J* = 16.5 and 6.3 Hz, 1 H), 2.41 (d, *J* = 16.5 Hz, 1 H), 2.12-1.60 (m, 3 H), 1.56-1.32 (m, 2 H), 0.89 (bd, 3 H).

^{13}C (75 MHz):δ 193.2, 152.8, 141.6, 136.3, 135.1, 129.0, 128.9, 128.8, 116.7, 107.5, 69.3, 51.7, 40.6, 40.5, 37.4, 29.4, 20.3.

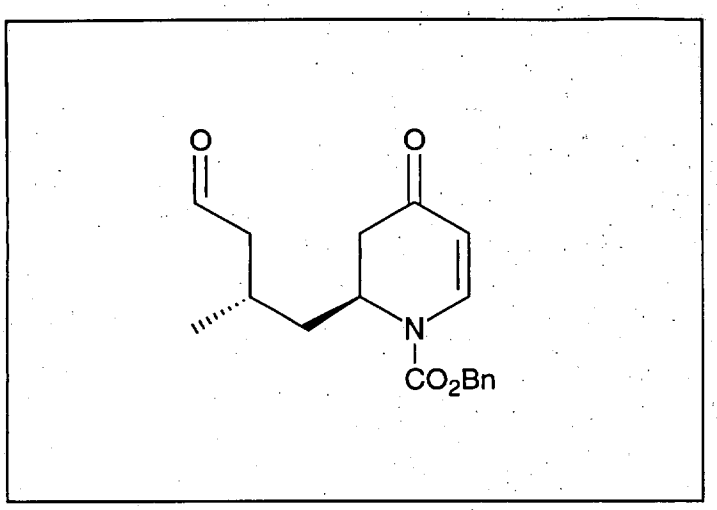
Literature References: none

Compound Name: **(-)-(*S*)-1-[(Benzyloxy)carbonyl]-2-[(*S*)-2-methyl-4-oxobutyl]-2,3-dihydro-4-pyridone**

Chemist: Clint Brooks

Formula: $C_{18}H_{21}NO_4$

Molecular Weight: 315.3687



Yields(s): 84%

Appearance: pale yellow oil

Stability: air sensitive

mp/bp:

$[\alpha]^{23}_D$ - 74.8 (*c* 1.09, $CHCl_3$)

HRMS: Calculated: 315.14704 Found: 315.146970

IR (thin film): 3477, 3081, 2953, 2718, 1723, 1665, 1606, 1456, 1606, 1456, 1387, 1194, 1093, 762 cm^{-1}.

NMR ($CDCl_3$):

^1H (300 MHz) :δ 9.64 (br s, 1 H), 7.77 (br d, *J* = 6.7 Hz, 1 H), 7.40 (s, 5 H), 5.18-5.39 (m, 3 H), 4.64 (br s, 1 H), 2.84 (dd, *J* = 16.6 and 6.5 Hz, 1 H), 2.41 (d, *J* = 16.6, 1 H), 1.40-2.35 (m, 5 H), 0.96 (br d, *J* = 6.4 Hz, 3 H).

^{13}C (75 MHz):δ 201.1, 192.2, 152.0, 141.0, 134.6, 128.3, 106.7, 68.6, 50.9, 49.9, 39.9, 37.4, 24.0, 20.1

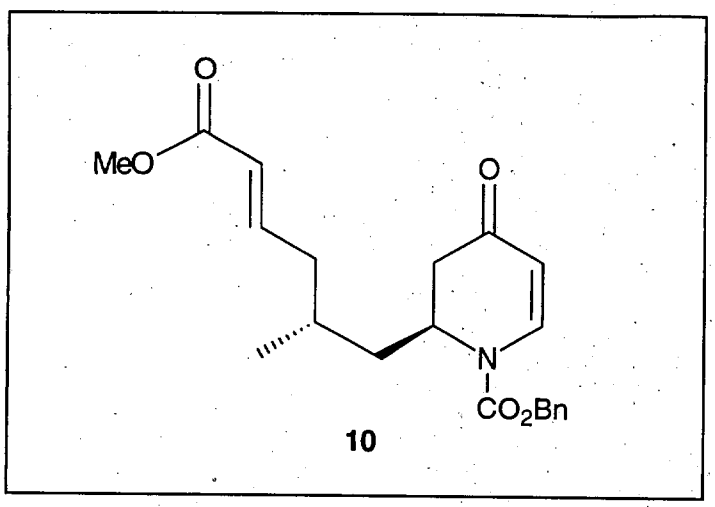
Literature References: none

Compound Name: **(+)-(*S*)-1-[(Benzyloxy)carbonyl]-2-[(*R*)-5-(methoxycarbonyl)-2-methyl-4-pentenyl]-2,3-dihydro-4-pyridone**

Chemist: Clint Brooks

Formula: $C_{21}H_{25}NO_5$

Molecular Weight: 371.43



10

Yields(s): 89%

Appearance: pale oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +61.3 (*c* 2.67, $CHCl_3$)

HRMS: Calculated: 371.17327 Found: 371.1733

IR (thin film): 3063, 2954, 2918, 1723, 1669, 1603, 1428, 1386, 1331, 1271, 1199, 1120, 759, 704 cm^{-1}.

NMR ($CDCl_3$):

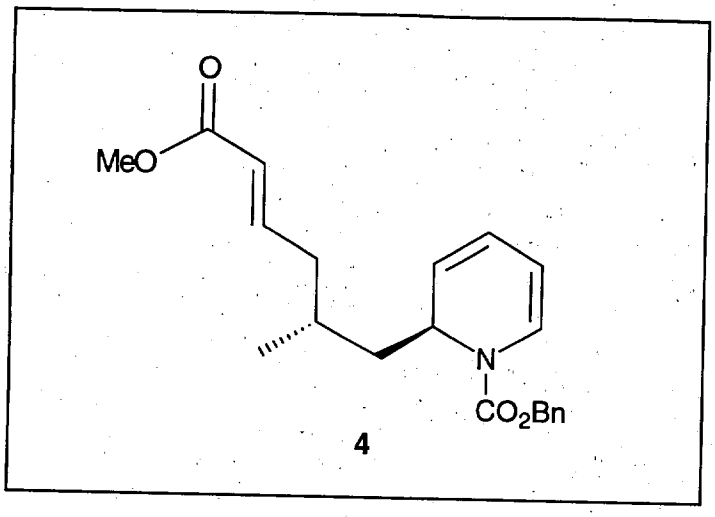
^1H (300 MHz) :δ 7.75 (brd, 1 H), 7.39 (s, 5 H), 6.93-6.77 (m, 1 H), 5.79 (d, *J* = 15.0 Hz, 1 H), 5.40-5.21 (m, 3 H), 4.70 (brs, 1 H), 3.73 (s, 3 H), 2.84 (dd, *J* = 18.0 and 6.0 Hz, 1 H), 2.39 (d, *J* = 18.0 Hz, 1 H), 2.30-1.35 (m, 5 H), 0.92 (brd, 3 H).

^{13}C (75 MHz):δ 192.7, 166.7, 152.5, 146.8, 141.4, 135.0, 128.9, 128.8, 122.8, 107.4, 69.2, 51.4, 40.5, 38.9, 37.6, 29.2, 20.1.

Literature References: none

Compound Name: **(-)-(*S*)-1-[(Benzyloxy)carbonyl]-2-[(*R*)-5-(methoxycarbonyl)-2-methyl-4-pentenyl]-1,2-dihydropyridine**

Chemist: Clint Brooks



4

Formula: $C_{21}H_{25}NO_4$

Molecular Weight: 355.43

Yields(s): 98%

Appearance: clear oil

Stability:

mp/bp:

$[\alpha]^{23}_D$ -499.9 (*c* 2.75, $CHCl_3$)

HRMS: Calculated: 355.17836 Found: 355,1783

IR (thin film): 3039, 2952, 2920, 1711, 1652, 1576, 1393, 1328, 1269, 1113, 983, 719 cm^{-1}.

NMR ($CDCl_3$):

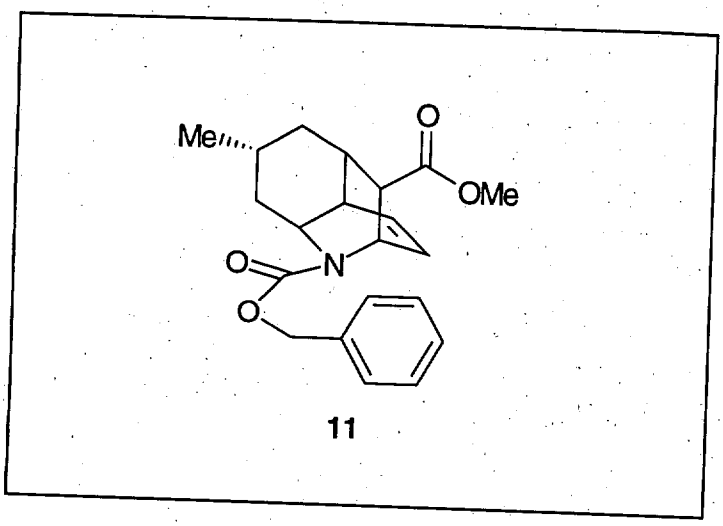
^1H (300 MHz) :δ 7.36 (br s, 5 H), 6.97-6.79 (m, 1 H), 6.76 and 6.66 (pair of d, due to rotamers, *J* = 7.7 Hz, 1 H), 5.98-5.70 (m, 2 H), 5.65-5.50 (m, 1 H), 5.36 and 5.27 (pair of t, due to rotamers, *J* = 6.7 and 6.4 Hz, 1H), 5.21 (s, 2 H), 4.96-4.70 (pair of m, due to rotamers, 1 H), 3.72 (s, 3 H), 2.35-1.30 (m, 5 H), 0.98 and 0.81 (pair of d, *J* = 6.3 Hz, 3 H).

^{13}C (75 MHz):δ 167.0,166.9, 154.2,153.6, 136.3, 128.7, 128.4, 128.2, 125.4, 124.5, 123.5, 122.8 122.5, 122.0,121.4, 106.7, 106.6, 68.1, 68.0 , 51.5, 50.4, 50.1, 40.9, 40.4, 39.7, 39.3, 28.4, 28.0, 20.3.

Literature References: none

Compound Name: **(+)-(1*S*, 3*R*, 4a*R*, 5*S*, 6*S*, 8a*S*)-3-Methyl-1, 2, 3, 4, 4a, 5, 6, 8a-octahydro-1, 6-epiazano-naphthalene-5,9-dicarboxylic acid 9-benzyl ester 5-methyl ester**

Chemist: Clint Brooks



11

Formula: $C_{21}H_{25}NO_4$

Molecular Weight: 355.434

Yields(s): 86%

Appearance: white solid

Stability: stable

mp: 72-73 °C

$[\alpha]^{23}_D$ +114.8 (*c* 1.12, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs)

	%C	%H	%N	%___
Calculated:	70.96	7.09	3.94	
Found:	70.75	7.05	3.86	

IR (thin film): 3051, 2952, 2909, 1734, 1696, 1400, 1284, 1276, 1115, 738, 701 cm^{-1}.

NMR ($CDCl_3$):

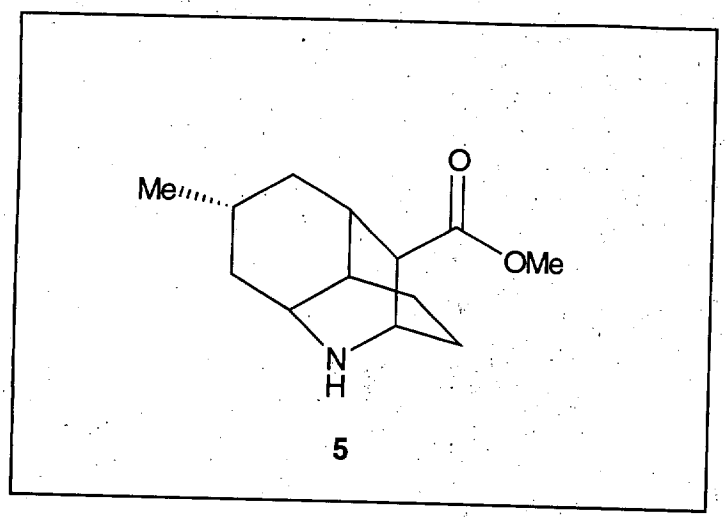
^1H (300 MHz) :δ 7.42-7.28 (m, 5 H), 6.48-6.28 (m, 2 H), 5.30-5.00 (m, 3 H), 3.63 (s, 3 H), 3.34 (br s, 3 H), 2.80 (appar. t, *J* = 3.7 Hz, 1 H), 2.45-2.15 (m, 2 H), 1.85-1.63 (m, 2 H), 1.35-1.10 (m, 1 H), 0.91 and 0.81 (pair of d, due to rotomers, *J* = 6.1 Hz, 3 H).

^{13}C (75 MHz):δ 173.5, 156.4 and 155.4 (due to rotamers), 137.1 and 137.0 (due to rotamers), 136.2, 131.5 and 130.9 (due to rotamers), 128.7, 128.1, 128.0 and 127.9 (due to rotamers), 67.1 and 67.0 (due to rotamers), 52.2, 52.0 and 51.8 (due to rotamers), 49.2 and 49.0 (due to rotamers), 48.0, 39.2 and 38.8 (due to rotamers), 37.9 and 37.5 (due to rotamers), 36.3, 32.5 and 32.2 (due to rotamers), 21.8 and 21.4 (due to rotamers).

Literature References: none

Compound Name: **(+)-(1*S*, 3*R*, 4a*R*, 5*S*, 6*S*, 8a*S*)-3-Methyl-decahydro-1, 6-epiazano-naphthalene-5-carboxylic acid methyl ester**

Chemist: Clint Brooks



5

Formula: $C_{13}H_{21}NO_2$

Molecular Weight: 247.340

Yields(s): 99%

Appearance: white solid

Stability:

mp: 90-91 °C

$[\alpha]^{23}_D$ +41.2 (*c* 0.165, $CHCl_3$)

Elemental Analysis (Atlantic Microlabs)

	%C	%H	%N	%___
Calculated:	69.92	9.48	6.27	
Found:	69.73	9.46	6.23	

IR (thin film): 3344, 2916, 2868, 1734, 1197, 1177, 1049 cm^{-1}.

NMR ($CDCl_3$):

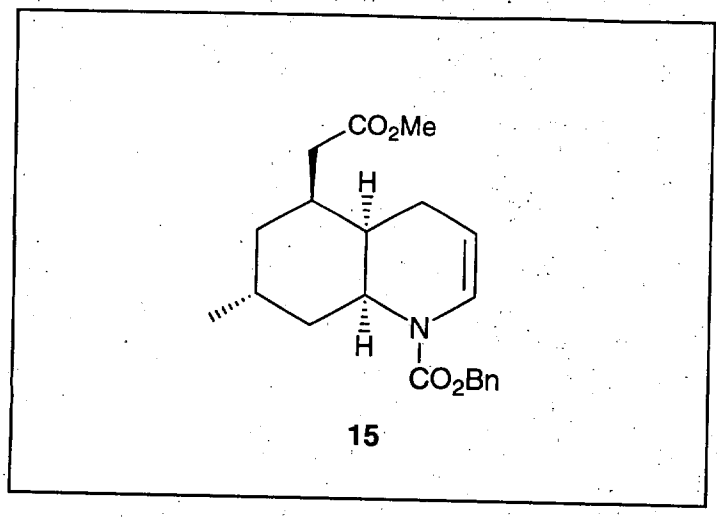
^1H (300 MHz) :δ 3.67 (s, 3 H), 3.22 (s, 1 H), 3.14 (s, 1 H), 2.66 (s, 1 H), 2.45 (s, 1 H), 1.90 (m, 1 H), 1.74-1.47 (m, 5 H), 1.34-1.22 (m, 2 H), 1.18-0.94 (m, 2 H), 0.94-0.84 (m, 4 H).

^{13}C (75 MHz):δ 174.6, 52.0, 50.7, 48.9, 46.5, 41.0, 39.7, 32.6, 32.2, 24.2, 23.0, 22.1, 21.6.

Literature References: none

Compound Name: **(+)-(4a*S*, 5*S*, 7*R*, 8a*S*)-5-Methoxycarbonylmethyl-7-methyl-4a, 5, 6, 7, 8 8a-hexahydro-4*H*-quinoline-1-carboxylic acid benzyl ester**

Chemist: Clint Brooks



15

Formula: $C_{21}H_{27}NO_4$

Molecular Weight: 357.45364

Yields(s): 51%

Appearance: clear oil

Stability: stable

mp/bp:

$[\alpha]^{25}_D$ +39.0 (*c* 0.59, $CHCl_3$)

HRMS: Calculated: 357.1940 Found: 357.1945

IR (thin film): 2952, 2922, 1732, 1701, 1660, 1417, 1391, 1334, 1251 cm^{-1}.

NMR ($CDCl_3$):

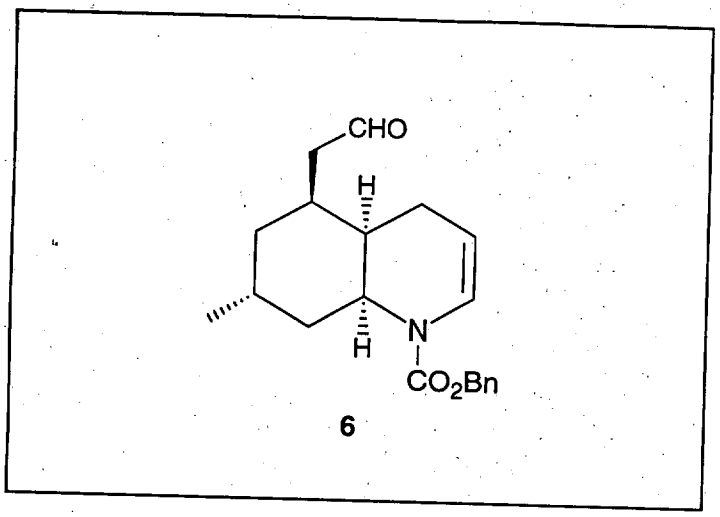
^1H (300 MHz) :δ 7.36 (m, 5 H), 6.82 and 6.73 (pair of d, due to rotamers, *J* = 8.2 Hz, 1 H), 5.18 (m, 2 H), 4.89 and 4.78 (pair of appar. t, due to rotamers, *J* = 6.3, 1 H) 4.42 and 4.33 (pair of d, due to rotamers, *J* = 12.4 Hz, 1 H), 3.68 (s, 3 H), 2.41 (m, 1 H), 2.32-1.75 (m, 5 H),1.70-1.34 (m, 3 H), 1.30-0.79 (m, 5 H).

^{13}C (75 MHz):d 173.1, 153.3 and 152.8 (due to rotamers), 136.6, 128.7, 128.3, 128.2 and 128.0 (due to rotamers), 124.0 and 123.5 (due to rotamers), 104.3, 67.6, 51.8, 49.5 and 49.1 (due to rotamers), 38.5, 34.9 and 34.8 (due to rotamers), 31.9, 31.6, 30.5 and 29.7 (due to rotamers), 27.6, 18.7, 17.2.

Literature References: none

Compound Name: **(+)-(4a*S*, 5*S*, 7*R*, 8a*S*)-5-Methoxycarbonylmethyl-7-methyl-4a, 5, 6, 7, 8, 8a-hexahydro-4*H*-quinoline-1-carboxylic acid benzyl ester**

Chemist: Clint Brooks



6

Formula: $C_{20}H_{25}NO_3$

Molecular Weight: 327.43

Yields(s): 75%

Appearance: clear oil

Stability: stable

mp/bp:

$[\alpha]^{23}_D$ +87.5 (*c* 0.41, $CHCl_3$)

HRMS: Calculated: 327.1834 Found: 327.1850

IR (thin film): 3383, 2955, 2913, 2861, 1702, 1695, 1658, 1418, 1392, 1340, 1251, 1120, 1100m cm^{-1}.

NMR ($CDCl_3$):

^1H (300 MHz) :δ 9.75 (s, 1 H), 7.36 (m, 5 H), 6.83 and 6.73 (pair of d, due to rotamers, J = 7.8 Hz, 1 H), 5.18 (m, 2 H), 4.88 and 4.78 (pair of t due to rotamers, J = Hz, 1 H), 4.44 and 4.35 (pair of d, due to rotamers, J = 9.6 Hz, 1 H), 2.55 (m, 1 H), 2.46-2.21 (m, 2 H), 2.20-1.96 (m, 3 H), 1.86-1.70 (m, 1 H), 1.70-1.34 (m, 3 H), 1.30-1.0 (m, 4 H).

^{13}C (75 MHz):δ 201.9 and 201.7 (due to rotamers), 153.3 and 152.7 (due to rotamers), 136.6 and 136.5 (due to rotamers), 128.7, 128.3, 128.1, 128.0, 124.1, 123.6, 104.0 and 104.0 (due to rotamers), 67.6, 49.4 and 49.1 (due to rotamers), 35.2 and 35.1 (due to rotamers), 31.9, 30.4, 29.1, 27.7, 18.8 and 18.6 (due to rotamers), 17.5 and 17.4 (due to rotamers).

Literature References: none

Compound Name: **(+)-(1*R*, 4*S*, 6*R*, 8*S*, 9*S*)-11-Hydroxy-6-methyl-3-aza-tricyclo[6.2.2.0^{4,9}] dodecane-3-carboxylic acid benzyl ester**

Chemist Name: Clint Brooks

Formula: $C_{20}H_{27}NO_3$

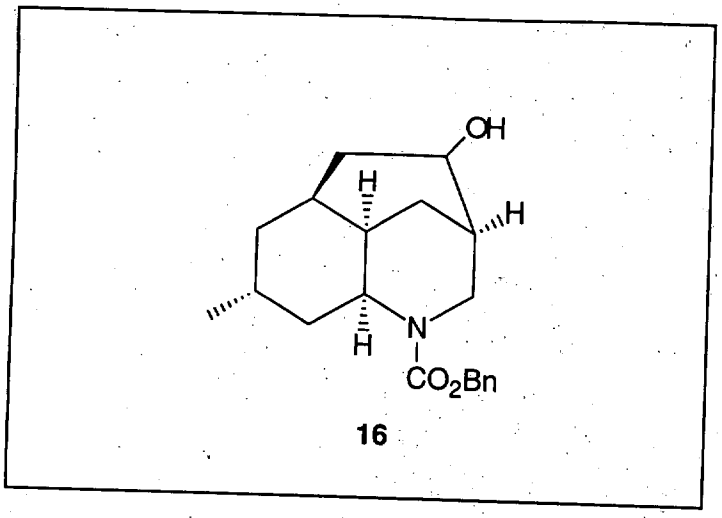
Molecular Weight: 329.443

Yields(s): 61%

Appearance: clear oil

Stability:

mp/bp:



16

HRMS: Calculated: 329.1991 Found: 329.1998

IR (thin film): 3439, 2947, 2906, 2856, 1699, 1679, 1453, 1398, 1297, 1267, 1071 cm^{-1}.

NMR ($CDCl_3$):

^{1}H (300 MHz) :δ 7.36 (m, 5 H), 5.23-5.12 (d, *J* = 12.5 Hz, 1 H), 5.08-4.98 (d, *J* = 12.5, 1 H), 4.27 (d, *J* = 13.6 Hz, 1 H), 3.84-3.76 (m, 1 H), 3.49 (s, 1 H), 3.15-3.0 (m, 2 H), 2.16-0.77 (m, 15 H).

^{13}C (75 MHz):δ 155.2, 128.7, 128.1, 72.6, 67.0, 56.2, 45.1, 40.4, 40.1, 35.8, 34.8, 34.7, 34.6, 31.7, 29.9, 22.4, 21.5, 19.9.

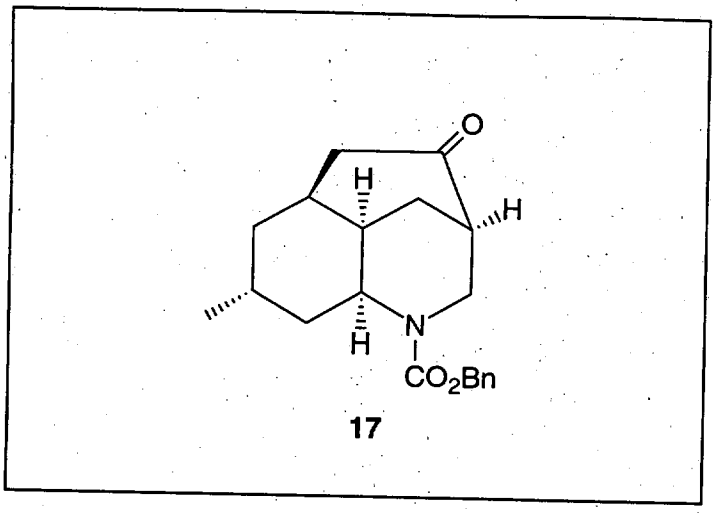
Literature References: none

Compound Name: **(+)-(1*R*, 4*S*, 6*R*, 8*S*, 9*S*)-6-Methyl-11-oxo-3-aza-tricyclo[6.2.2.0^{4,9}]dodecane-3-carboxylic acid benzyl ester**

Chemist Name: Clint Brooks

Formula: $C_{20}H_{25}NO_3$

Molecular Weight: 327.42715



17

Yields(s): 99%

Appearance: clear oil

Stability:

mp/bp:

$[\alpha]^{23}_D$ +124.1 (*c* 0.245, $CHCl_3$)

HRMS: Calculated: 327.1834 Found: 327.1838

IR (thin film): 2939, 2907, 2855, 1706, 1448, 1295, 1211, 1168, 1074 cm^{-1}.

NMR ($CDCl_3$):

^1H (300 MHz) :δ 7.33 (m, 5 H), 5.08 (ABq, 2 H), 4.16 (d, *J* = 13.5 Hz, 1 H), 3.58 (d, *J* = 2.8 Hz, 1 H) 3.28 (dd, *J* = 14.2 and 2.3 Hz, 1 H), 3.11 (dd, *J* = 13.5 and 3.8 Hz, 1 H), 2.70 (dd, *J* = 15.5 and 12.9 Hz, 1 H), 2.52 (m, 1 H), 2.36-2.15 (m, 2 H), 1.90-1.75 (m, 4 H), 1.60-1.24 (m, 2 H), 1.12-0.98 (m, 1 H), 0.87 (d, *J* = 6.5 Hz, 3 H).

^{13}C (75 MHz):δ 213.1, 157.0, 136.4, 128.6, 128.1, 128.0, 67.2, 56.4, 48.4, 44.8 (2 carbons), 39.7, 39.5, 37.0, 35.2, 33.2, 21.9, 21.0.

Literature References: none

Compound Name: **(+)-luciduline**

Chemist Named: Clint Brooks

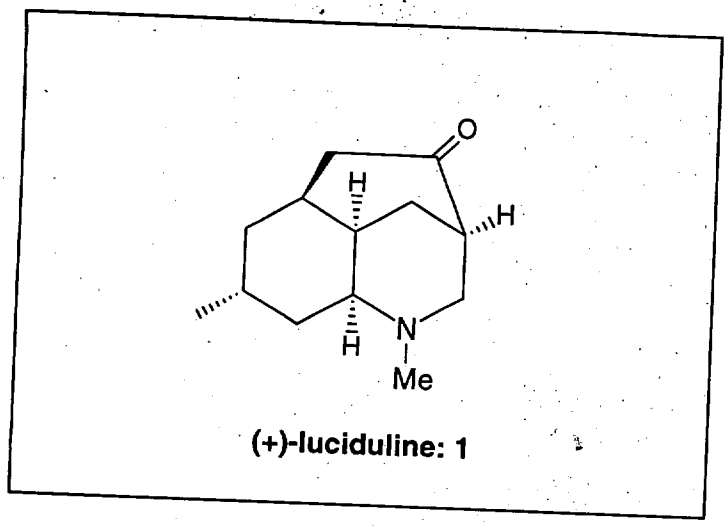
Formula: $C_{13}H_{21}NO$

Molecular Weight: 207.318

Yields(s): 95%

Appearance: clear oil

Stability: moderate



(+)-luciduline: 1

synthetic: $[\alpha]^{23}_D$ +85.3 (*c* 0.15, MeOH)

literature: $[\alpha]^{22}_D$ +87.0 (*c* 2.05, MeOH)

NMR ($CDCl_3$):

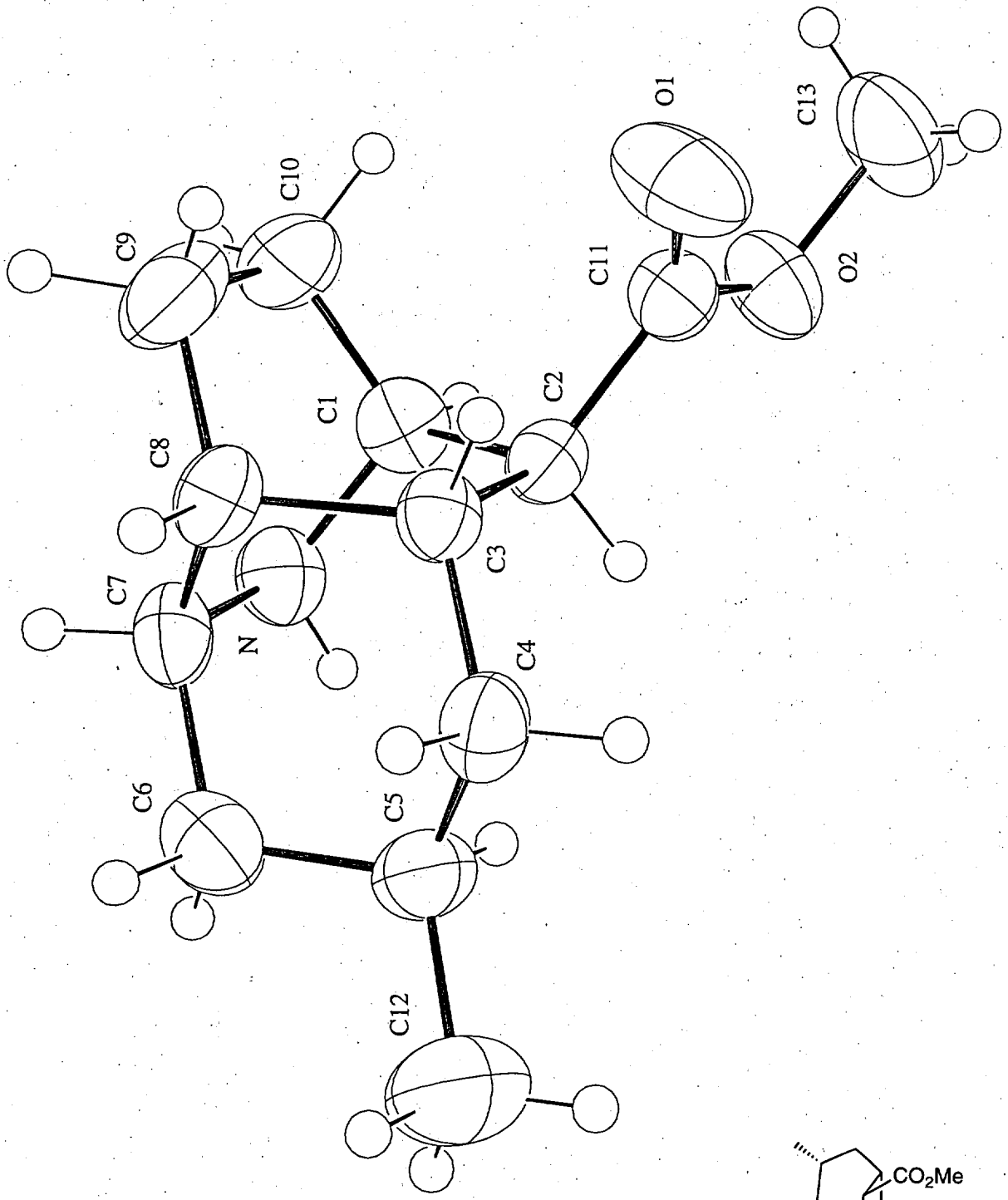
synthetic:

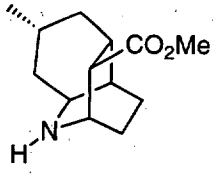
^1H (300 MHz) :δ 3.3-2.7 (2 H), 2.14 (s, 3 H), 2.6-0.8 (m, 13 H), 0.89 (d, J = 6.5 Hz, 3 H).

literature:

^1H (300 MHz): δ 3.3-2.7 (2 H), 2.14 (s, 3 H), 2.6-0.8 (13 H), 0.89 (d, J = 6.5 Hz, 3 H).

Literature References: Oppolzer, W; Petrzilka, M. *Helv. Chim. Acta* **1978**, *61*, 2755.





5

xperimental

ata Collection. The sample (x98046) was submitted by Clint Brooks of omins research group. The sample was mounted on the end of a glass iber using 5 minute epoxy, and transferred to the diffractometer. ll X-ray measurements were made at ambient temperature on an Nonius AD4 diffractometer. The unit cell dimensions were determined by a ymmetry constrained fit of 25 well centered reflections and thier Friedel airs with theta 33 deg < 2(theta) < 36 deg. For a 1.5 to 25.0 degree theta) shell, a unique octant of data was collected. After that data ollection was completed, a 25.0 - 28.0 deg (theta) hemisphere of data as collected. However, in addition to the weak data at high angle, it as also noticed that the crystal underwent moderate radiation damage, and he resulting Rmerge for the high angle shell was 0.17. As a consequence, nly the 1.5 to 25.0 degree data was used in the structure refinement. hree standard reflections were measured every 4800 seconds of X-ray xposure time. The intensities of these standard check reflections ecreased by about 5% over the course of the data collection. Scaling the ata was accomplished using a 5 point smoothed curved routine fit to he intensity check reflections. The intensity data was corrected for orentz and polarization effects. The data were not absorption corrected.

tructure Solution and Refinement. The data were reduced using routines rom the NRCVAX set of programs. The structure was solved using SIR92. ll non-hydrogen atoms were refined anisotropically. Non-methyl hydrogen tom were placed at idealized positions and were allowed to refine sotropically. The methyl hydrogens were refined as a rigid group with he C-H bond distance constrainted to be 0.96 A and the {O,C}-C-H angle ere constrained to be 109.47 deg. The group origin and orientation ere allowed to refine. The isotropic displacement parameters were onstrained to be 0.01 A^2 larger than and ride on the parent carbon atom. his model refined smoothly using the NRCVAX LSTSQ routine to give R inal factors of R = 0.068 and Rw = 0.070. The calculated structure actors were fit to the data using full matrix least-squares based on squared. The calculated structure factors included corrections for nomalous dispersion from the usual tabulation (International Tables or X-ray Crystallography, Vol. IV, (1974), Table 2.3.1, Kynoch Press, irmingham, England). A secondary extinction correction was included in he final refinements. Additional information and relevant literature eferences can be found in the REPORT.OUT file and the reference section f the Facility's Web page.

X98046: COMINS/BROOKS ROOM TEMP

Space Group and Cell Dimensions Orthorhombic P 212121
a 8.8115(5) b 11.7040(9) c 12.1902(12)
Volume 1257.17(17)A**3

Empirical formula : C13 H21 N O2

Cell dimensions were obtained from 24 reflections with 2Theta angle in the range 33.00 - 36.00 degrees.

Crystal dimensions : .44 X .38 X .34 mm

FW = 223.31 Z = 4 F(000) = 488.28

Dcalc 1.180Mg.m-3, mu .08mm-1, lambda .71073A, 2Theta(max) 50.0

The intensity data were collected on a Nonius diffractometer, using the omega scan mode.
The h,k,l ranges used during structure solution and refinement are :--
Hmin,max -10 10; Kmin,max -13 13; Lmin,max -13 14

No. of reflections measured	1331
No. of unique reflections	1305
No. of reflections with Inet > ***sigma(Inet)	1305

Merging R-value on intensities .109
No correction was made for absorption

The last least squares cycle was calculated with
37 atoms, 206 parameters and 1305 out of 1305 reflections.
Weights based on counting-statistics were used.
The weight modifier K in KFo**2 is .000500

The residuals are as follows :--
For significant reflections, RF .068, Rw .070 GoF 2.95
For all reflections, RF .068, Rw .070.
where RF = Sum(Fo-Fc)/Sum(Fo),
Rw = Sqrt[Sum(w(Fo-Fc)**2)/Sum(wFo**2)] and
GoF = Sqrt[Sum(w(Fo-Fc)**2)/(No. of reflns - No. of params.)]
The maximum shift/sigma ratio was .002.

In the last D-map, the deepest hole was -.150e/A**3,
and the highest peak .190e/A**3.

Secondary ext. coeff. .6990microns sigma .0582

The following references are relevant to the NRCVAX System.

1. Full System Reference :
Gabe, E.J., Le Page, Y., Charland, J.-P., Lee, F.L. and White, P.S. (1989) J. Appl. Cryst., 22, 384-387.

2. Scattering Factors from Int. Tab. Vol. 4 :
International Tables for X-ray Crystallography, Vol. IV, (1974) Kynoch Press, Birmingham, England.

The following references may also be relevant.

3. ORTEP Plotting :
Johnson, C.K., (1976) ORTEP - A Fortran Thermal Ellipsoid Plot

Program, Technical Report ORNL-5138, Oak Ridge

4. Pluto Plotting :
S. Motherwell, University Chemical Laboratory, Cambridge, 1978

5. Missing Symmetry Treatment by MISSYM :
Le Page, Y., (1988) J. Appl. Cryst., 21, 983-984.

6. Grouping of Equivalent Reflections in DATRD2 :
Le Page, Y. and Gabe, E.J., (1979) J. Appl. Cryst., 12, 464-466.

7. Extinction Treatment :
Larson, A.C., (1970) p.293, Crystallographic Computing, Munksgaard, Copenhagen.

X98046: COMINS/BROOKS ROOM TEMP 21-Sep-1998

Table . Atomic Parameters x,y,z and Biso.
E.S.Ds. refer to the last digit printed.

	x	y	z	Biso
O1	.4645(3)	.2493(2)	.3853 (2)	6.35(14)
O2	.3403(2)	.4148(2)	.38798(18)	4.89(11)
N	.5721(3)	.4854(3)	.0855 (2)	3.92(13)
C1	.4737(4)	.4279(3)	.1649 (3)	3.72(15)
C2	.5562(4)	.4088(3)	.2763 (2)	3.10(13)
C3	.7062(4)	.3425(3)	.2539 (2)	3.10(13)
C4	.8469(4)	.4104(3)	.2858 (3)	3.88(17)
C5	.8653(3)	.5211(3)	.2219 (3)	4.16(17)
C6	.8554(4)	.4975(4)	.0971 (3)	4.51(18)
C7	.7173(4)	.4250(3)	.0671 (2)	3.89(16)
C8	.7121(4)	.3138(3)	.1302 (3)	3.51(15)
C9	.5736(6)	.2439(4)	.0965 (4)	5.3 (2)
C10	.4293(5)	.3134(4)	.1164 (3)	4.95(20)
C11	.4527(4)	.3464(3)	.3555 (2)	3.69(15)
C12	1.0121(3)	.5823(4)	.2502 (4)	7.4 (2)
C13	.2294(3)	.3641(3)	.4601 (3)	6.5 (2)
H1n	.591 (3)	.559 (3)	.104 (2)	3.3 (7)
H1	.387 (3)	.470 (2)	.177 (2)	2.3 (6)
H2	.575 (3)	.4831(20)	.3091 (17)	1.3 (5)
H3	.701 (3)	.273 (2)	.2970 (18)	2.6 (6)
H4a	.939 (4)	.353 (2)	.270 (3)	5.4 (8)
H4b	.842 (3)	.429 (3)	.362 (2)	4.5 (8)
H5	.780 (3)	.569 (3)	.238 (3)	4.8 (8)
H6a	.846 (4)	.575 (3)	.058 (3)	6.8 (10)
H6b	.951 (4)	.448 (3)	.077 (3)	6.5 (9)
H7	.721 (3)	.412 (2)	-.010 (2)	4.0 (7)
H8	.808 (3)	.269 (2)	.121 (2)	3.7 (7)
H9a	.583 (4)	.212 (3)	.019 (3)	6.6 (10)
H9b	.572 (4)	.180 (3)	.134 (3)	6.0 (11)
H10a	.354 (4)	.276 (3)	.168 (3)	6.6 (10)
H10b	.374 (3)	.327 (3)	.050 (2)	4.7 (8)
H12a	1.0146(13)	.6551(9)	.2140 (16)	7.8
H12b	1.0968(3)	.5370(11)	.2264 (18)	7.8
H12c	1.0178(13)	.5934(18)	.3281 (4)	7.8
H13a	.1545(14)	.4202(7)	.4793 (15)	7.1
H13b	.2788(7)	.3372(18)	.5254 (8)	7.1
H13c	.1812(18)	.3011(12)	.4236 (8)	7.1

Biso is the Mean of the Principal Axes of the Thermal Ellipsoid

Table of u(i,j) or U values *100.
E.S.Ds. refer to the last digit printed

	u11(U)	u22	u33	u12	u13	u23
O1	7.92(18)	6.07(16)	10.2 (2)	.39(16)	2.51(18)	2.57(17)
O2	5.54(14)	7.04(16)	6.00(15)	.83(15)	1.92(13)	.54(14)
N	5.67(18)	5.05(18)	4.19(16)	.68(18)	-.08(15)	.57(15)
C1	3.87(20)	5.4 (2)	4.90(20)	.7 (2)	-.47(17)	.39(18)
C2	4.29(17)	3.68(17)	3.79(17)	-.10(18)	-.22(15)	-.35(16)
C3	4.53(18)	3.79(18)	3.46(17)	.28(19)	-.08(16)	.19(14)
C4	4.8 (2)	6.0 (2)	4.0 (2)	.4 (2)	-.30(16)	-.7 (2)
C5	4.4 (2)	5.1 (2)	6.3 (2)	-.7 (2)	.15(18)	-1.0 (2)
C6	5.4 (2)	5.8 (3)	5.9 (2)	.1 (2)	1.22(20)	.3 (2)
C7	6.1 (2)	5.4 (2)	3.20(19)	.3 (2)	.50(17)	-.11(17)
C8	5.1 (2)	3.89(19)	4.36(19)	.60(18)	-.10(18)	-.89(16)
C9	9.0 (3)	5.0 (2)	6.2 (2)	-.9 (3)	-1.0 (3)	-1.6 (2)
C10	6.0 (2)	7.5 (3)	5.3 (2)	-1.6 (2)	-1.5 (2)	-.8 (2)
C11	4.61(20)	5.01(20)	4.41(19)	-.2 (2)	-.16(18)	-.05(17)
C12	7.0 (3)	10.0 (3)	11.1 (3)	-2.6 (3)	-.1 (2)	-2.0 (3)
C13	6.0 (2)	12.0 (4)	6.8 (2)	-.2 (3)	2.4 (2)	.4 (3)
H1n	4.2 (9)					
H1	2.9 (8)					
H2	1.6 (6)					
H3	3.3 (7)					
H4a	6.9 (10)					
H4b	5.7 (10)					
H5	6.1 (10)					
H6a	8.7 (13)					
H6b	8.3 (12)					
H7	5.1 (9)					
H8	4.7 (9)					
H9a	8.4 (12)					
H9b	7.6 (14)					
H10a	8.4 (13)					
H10b	6.0 (10)					
H12a	9.8					
H12b	9.8					
H12c	9.8					
H13a	9.0					
H13b	9.0					
H13c	9.0					

Anisotropic Temperature Factors are of the form
Temp=-2*Pi*Pi*(h*h*u11*astar*astar+---+2*h*k*u12*astar*bstar+---)

DISANG -- The NRCVAX Distance and Angle Program

The Space Group is P 21 21 21 NonCentrosymmetric
The Equivalent Positions are:

1)	x	y	z	2)	1/2+x	1/2-y	-z
3)	-x	1/2+y	1/2-z	4)	1/2-x	-y	1/2+z

The Lattice is Primitive. There are no Centring Vectors

X98046: COMINS/BROOKS ROOM TEMP 21-Sep-1998
The following Atoms are from the CD File
* Indicates that there are Symmetry Equivalents of an atom.

Name	x	y	z
O1	.46449(31)	.24927(21)	.38529(21)
O2	.34033(23)	.41480(21)	.38798(18)
N	.57212(33)	.48542(27)	.08554(21)
C1	.47374(36)	.42789(31)	.16487(26)
C2	.55624(36)	.40877(27)	.27627(23)
C3	.70625(35)	.34251(26)	.25393(24)
C4	.84692(38)	.41039(33)	.28580(28)
C5	.86534(34)	.52112(31)	.22188(29)
C6	.85536(42)	.49754(36)	.09712(30)
C7	.71729(40)	.42501(32)	.06710(24)
C8	.71206(39)	.31377(27)	.13021(25)
C9	.57359(55)	.24388(37)	.09649(35)
C10	.42935(48)	.31340(36)	.11640(31)
C11	.45268(39)	.34638(29)	.35549(24)
C12	1.01212(32)	.58227(35)	.25022(38)
C13	.22944(33)	.36410(29)	.46008(25)
H1n	.59081(308)	.55884(257)	.10396(230)
H1	.38740(293)	.46966(222)	.17657(200)
H2	.57503(253)	.48310(197)	.30910(174)
H3	.70073(292)	.27300(213)	.29696(184)
H4a	.93884(356)	.35255(247)	.26983(261)
H4b	.84188(310)	.42883(266)	.36163(238)
H5	.77959(345)	.56915(262)	.23806(252)
H6a	.84562(397)	.57547(316)	.05753(259)
H6b	.95127(383)	.44756(285)	.07703(263)
H7	.72119(316)	.41218(232)	-.01028(230)
H8	.80847(316)	.26943(237)	.12119(223)
H9a	.58268(368)	.21173(275)	.01925(295)
H9b	.57205(428)	.18024(303)	.13382(267)
H10a	.35388(382)	.27557(298)	.16795(258)
H10b	.37355(320)	.32729(269)	.04959(234)
H12a	1.01459(133)	.65510(91)	.21404(155)
H12b	1.09681(31)	.53702(108)	.22640(175)
H12c	1.01780(133)	.59336(175)	.32814(44)
H13a	.15449(140)	.42017(67)	.47927(148)
H13b	.27879(66)	.33725(175)	.52539(84)
H13c	.18123(183)	.30113(123)	.42359(80)

X98046: COMINS/BROOKS ROOM TEMP 21-Sep-1998

O1-C11	1.198(4)	C5-H5	.96(3)
O2-C11	1.333(4)	C6-C7	1.528(5)
O2-C13	1.442(4)	C6-H6a	1.04(4)
N-C1	1.463(4)	C6-H6b	1.06(4)
N-C7	1.479(5)	C7-C8	1.513(5)
N-H1n	.90(3)	C7-H7	.96(3)
C1-C2	1.556(4)	C8-C9	1.525(5)
C1-C10	1.516(5)	C8-H8	1.00(3)
C1-H1	.92(3)	C9-C10	1.528(7)
C2-C3	1.556(4)	C9-H9a	1.02(4)
C2-C11	1.516(5)	C9-H9b	.87(4)
C2-H2	.97(2)	C10-H10a	1.02(3)
C3-C4	1.523(5)	C10-H10b	.97(3)
C3-C8	1.546(4)	C12-H12a	.960(14)
C3-H3	.97(3)	C12-H12b	.960(10)
C4-C5	1.521(5)	C12-H12c	.960(8)
C4-H4a	1.07(3)	C13-H13a	.960(11)
C4-H4b	.95(3)	C13-H13b	.960(12)
C5-C6	1.548(5)	C13-H13c	.960(14)
C5-C12	1.518(5)	H9a-H9b	1.45(5)

C11-O2-C13	115.9(3)	N-C7-C8	107.9(3)
C1-N-C7	113.1(3)	N-C7-H7	104.8(17)
C1-N-H1n	112.4(18)	C6-C7-C8	112.4(3)
C7-N-H1n	109.6(17)	C6-C7-H7	107.2(17)
N-C1-C2	111.5(3)	C8-C7-H7	111.6(17)
N-C1-C10	107.6(3)	C3-C8-C7	108.0(3)
N-C1-H1	110.5(16)	C3-C8-C9	110.7(3)
C2-C1-C10	109.5(3)	C3-C8-H8	104.4(16)
C2-C1-H1	109.2(15)	C7-C8-C9	110.4(3)
C10-C1-H1	108.6(16)	C7-C8-H8	111.4(16)
C1-C2-C3	108.4(2)	C9-C8-H8	111.8(16)
C1-C2-C11	110.1(3)	C8-C9-C10	109.7(3)
C1-C2-H2	108.1(13)	C8-C9-H9a	112.6(19)
C3-C2-C11	112.5(3)	C8-C9-H9b	109(2)
C3-C2-H2	111.9(13)	C10-C9-H9a	114.2(18)
C11-C2-H2	105.8(13)	C10-C9-H9b	111(2)
C2-C3-C4	112.8(3)	H9a-C9-H9b	99(3)
C2-C3-C8	107.9(2)	C1-C10-C9	108.5(3)
C2-C3-H3	106.3(15)	C1-C10-H10a	108.2(19)
C4-C3-C8	109.6(3)	C1-C10-H10b	108.2(19)
C4-C3-H3	109.9(15)	C9-C10-H10a	114.2(19)
C8-C3-H3	110.3(13)	C9-C10-H10b	112.3(17)
C3-C4-C5	113.6(3)	H10a-C10-H10b	105(2)
C3-C4-H4a	103.8(16)	O1-C11-O2	123.0(3)
C3-C4-H4b	109.2(17)	O1-C11-C2	126.7(3)
C5-C4-H4a	111.3(16)	O2-C11-C2	110.3(3)
C5-C4-H4b	108.0(19)	C5-C12-H12a	109.5(8)
H4a-C4-H4b	110(2)	C5-C12-H12b	109.5(8)
C4-C5-C6	110.2(3)	C5-C12-H12c	109.5(9)
C4-C5-C12	112.1(3)	H12a-C12-H12b	109.5(13)
C4-C5-H5	108.0(18)	H12a-C12-H12c	109.5(17)
C6-C5-C12	110.9(3)	H12b-C12-H12c	109.5(15)
C6-C5-H5	105.1(19)	O2-C13-H13a	109.5(8)
C12-C5-H5	110.3(17)	O2-C13-H13b	109.5(6)
C5-C6-C7	112.3(3)	O2-C13-H13c	109.5(8)

C5-C6-H6a	107.8(18)	H13a-C13-H13b	109.5(14)
C5-C6-H6b	106.3(18)	H13a-C13-H13c	109.5(12)
C7-C6-H6a	108.2(20)	H13b-C13-H13c	109.5(14)
C7-C6-H6b	105.9(17)	C9-H9a-H9b	36.5(18)
H6a-C6-H6b	116(3)	C9-H9b-H9a	43(2)
N-C7-C6	112.8(3)		

Torsion angles X98046: COMINS/BROOKS ROOM TEMP

C7	N	C1	C2	55.5(4)
C1	N	C7	C6	-119.5(6)
N	C1	C2	C3	-55.1(4)
C10	C1	C2	C3	63.8(5)
N	C1	C10	C9	60.2(5)
C1	C2	C3	C4	117.6(6)
C11	C2	C3	C4	-120.4(6)
C1	C2	C11	O1	108.3(6)
C3	C2	C11	O1	-12.8(3)
C2	C3	C4	C5	-61.8(4)
C2	C3	C8	C7	63.1(4)
C4	C3	C8	C7	-60.0(4)
C3	C4	C5	C6	-52.1(4)
C5	C6	C7	N	67.1(5)
C7	C6	C5	C4	49.3(4)
N	C7	C8	C3	-65.4(4)
C6	C7	C8	C3	59.5(4)
C3	C8	C9	C10	61.7(5)
C8	C9	C10	C1	-.9(3)
C2	C11	O2	C13	177.4(6)
C7	N	C1	C10	-64.5(5)
C1	N	C7	C8	5.2(3)
N	C1	C2	C11	-178.6(7)
C10	C1	C2	C11	-59.6(5)
C2	C1	C10	C9	-61.1(5)
C1	C2	C3	C8	-3.5(3)
C11	C2	C3	C8	118.5(6)
C1	C2	C11	O2	-70.6(5)
C3	C2	C11	O2	168.4(7)
C8	C3	C4	C5	58.4(4)
C2	C3	C8	C9	-57.9(5)
C4	C3	C8	C9	179.0(7)
C3	C4	C5	C12	-176.1(7)
C5	C6	C7	C8	-55.2(4)
C7	C6	C5	C12	173.9(8)
N	C7	C8	C9	55.7(5)
C6	C7	C8	C9	-179.3(7)
C7	C8	C9	C10	-57.9(5)
O1	C11	O2	C13	-1.5(3)

Table . Distances(A) to the least-squares planes.

Plane no. 1

Equation of the plane : 4.859(9)X + 3.563(19)Y + 9.468(10)Z = 6.791(4)

Distances(A) to the plane from the atoms in the plane.

O1	.002(4)	O2	.015(3)
C2	-.016(4)	C11	.009(4)
C13	-.022(4)		

Chi squared for this plane 67.705