Supporting Information

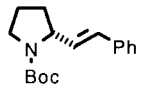
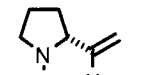
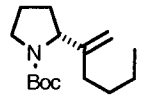
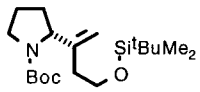
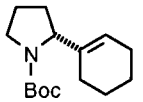
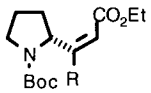
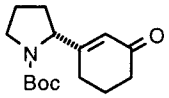
Reactivity and Enantioselectivity in the Reactions of Scalemic Stereogenic α-(*N*-carbamoyl)alkylcuprates

R. Karl Dieter*, Gabriel Oba, Kishan R. Chandupatla, Chris M. Topping, Kai Lu and Rhett T. Watson

Howard L. Hunter Laboratory, Department of Chemistry, Clemson University, Clemson, South Carolina 29634-0973.

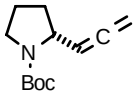
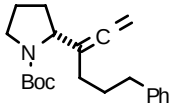
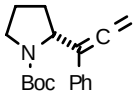
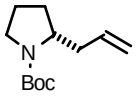
Expanded Tables:

Table 1. Reactions of scalemic α-(*N*-carbamoyl)alkylcuprates, prepared from scalemic α-lithio *N*-Boc pyrrolidine, with a variety of electrophiles.

entry	E^+	Cu(I) equiv[a]	temperature Cu °C (min)[b]	solvent[c]	temperature Rxn, °C (h)[d]	Product[e]	cpd No.	% yield[f]	% er[g]
1	**1**	0.5	-78 to -60 (60)	A	-60 to 25 (12)		**14**	85-90	50:50
2	**1**	0.5	-78 to -60 (60)	B	-60 to 25 (12)		**14**	85-98	90:10-93:7
3	**1**	1.0	-78 to -60 (105)	B	-60 to 25 (12)		**14**	70	89:11
4	**1**	0.5	-78 to -40 (120)	B	-60 to 25 (12)		**14**	86	73:27
5	**1**	0.5[h]	-78 (45) 25 (15)	C	-60 to 25 (12)		**14**	70	51:49-55:45
6	**2a**	0.5	-78 (60)	B	-78 to 25 (12)		**15a**	69	85:15
7	**2a**	0.5	-78 (60)	B 1:1[i]	-78 to 25 (12)		**15a**	66	88:12
8	**2a**	0.5	-78 (60)	B 2:1[i]	-78 to 25 (12)		**15a**	68	72.9:27.1
9	**2a**	0.5	-78 (60)	B 4:1[i]	-78 to 25 (12)		**15a**	59	71.5:28.5
10	**2a**	1.0	-78 (60)	B 1:1.3[i]	-78 to 25 (12)		**15a**	60	72.5:27.5
11	**2b**	1.0	-78 (60)	A	-78 to 25 (12)	**a** X = H	**15b**	77	50:50
12	**2b**	0.5	-78 (60)	B	-78 to 25 (12)		**15b**	96	65.5:34.5
13	**2b**	0.5	-78 (60)	B	-60 (10)	**b** X = $SiMe_3$	**15b**	90	90:10
14	**2c**	1.0	-78 (60)	A	-78 to 25 (12)		**16**	76	-
15	**2c**	1.0	-78 (60)	B 1:1.3	-78 to 25 (12		**16**	70-83	97:3-92:7
16	**2c**	1.0[j]	-78 (60)	B 1:1.3	-78 to 25 (12		**16**	63-82	86.5:13.5-82:18
17	**2c**	0.5	-78 (60)	B	-78 to 25 (12)		**16**	66	95.4:4.6
18	**2d**	0.5	-78 (60)	B	-78 to 25 (12)		**17**	79	94:6
19	**2d**	0.5	-78 (60)	B	-78 to 25 (3-4)		**17**	84[i]	94:6
20	**3a**	0.5	-78 (60)	B 1:1.3[i]	-78 to 25 (12)		**18**	58	90.7:9.3
21	**4a**	0.5	-78 to -50 (60)	B	-50 to 25 (4)		**19a**	63	93:7
22	**4a**	0.5	-78 (60)	B	-78 to 25 (12)		**19a**	89	95:5-96:4
23	**4a**	0.5[i]	-78 (60)	B	-78 to 25 (12)		**19a**	84	93.5:6.5
24	**4a**	0.5[k]	-78 (60)	B	-78 to 25 (12)		**19a**	53	93.5:6.5
25	**4a**	0.5[l]	-78 (60)	B	-78 to 25 (12)		**19a**	80	90:10
26	**4a**	1.0[l]	-78 (60)	B	-78 to 25 (12)	**a** R = H	**19a**	88	86.5:13.5
27	**4b**	0.5	-78 (60)	B	-78 to –35 (3)	**b** R = *n*-Bu	**19b**	53[m]	91:9
28	**6a**	1.0	-78 (60)	B	-78 to 25 (12)		**20**	80	50:50
29	**6a**	0.5	-78 (60)	B	-78 to 25 (12)		**20**	79	65:35
30	**6a**	0.5	-78 (60)	B	-40 (8)		**20**	79	80:20

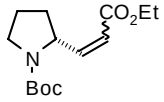
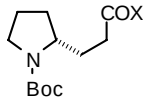
[a] CuCN·2LiCl was employed and reactions run on a 1.0 mmole (**12**) scale unless otherwise noted. [b] Temperature and time at which cuprate formation was achieved. [c] A = THF. B = THF/Et_2O solvent ratio (1:1, v/v, unless otherwise noted) arose by deprotonation of carbamate in Et_2O followed by addition of a THF solution of CuCN·2LiCl. C = Et_2O. [d] Temperature and time at which the cuprate/electrophile reaction was allowed to proceed. [e] Transmetallation and cuprate reactions assumed to proceed with retention of configuration[1-3] from scalemic *N*-Boc-α-lithiopyrrolidine.[4] Confirmed for **14**. [f] Based on products purified and isolated by flash column chromatography. [g] Enantioselectivity (% er) was measured by chiral stationary phase HPLC on a CHIRALCEL™ OD column [cellulose tris(3,5-dimethylphenylcarbamate) on silica gel. [h] Solid CuCN was used. [i] 2.0 mmol scale. [j] CuCl·2LiCl was employed. [k] 6.0 mmol scale. [l] 10 mmol scale. [m] TMSCl (5.0 equiv) was employed.

Table 3. Reactions of scalemic α-(*N*-carbamoyl)alkylcuprates, prepared from scalemic α-lithio *N*-Boc pyrrolidine, with propargyl and allylic substrates.

entry	E^+ (LG)	Cu(I) equiv[a]	temperature Cu °C (min)[b]	solvent[c]	temperature Rxn, °C (h)[d]	Product[e]	cpd. No.	% yield[f]	% er[g]
1	**8a** Br	1.0	-78 (60)	A	-78 to 25 (12)		**23**	74	50:50
2	**8a** Br	0.5	-78 (60)	B	-78 to 25 (12)		**23**	48	75:25
3	**8a** Br	0.5	-78 (60)	B	-78 to 25 (12)		**23**	71	66.7:33.3
4	**8b** OMs	1.0	-78 (60)	B	-78 to 25 (12)		**23**	76	58.4:41.6
5	**8b** OMs	0.5	-78 (60)	B	-78 to 25 (12)		**23**	58	74.4:25.6
6	**8c** OMs	1.0	-78 (60)	A	-78 to 25 (12)		**24**	86	50:50
7	**8c** OMs	1.0	-78 (60)	B	-50 (10)		**24**	79	50:50
8	**8c** OMs	0.5	-78 (60)	B	-78 (5)		**24**	57	90:10
9	**8d** OMs	1.0	-78 to -50 (30)	B	-78 to 25 (12)		**25**	57	65:35
10	**8d** OMs	1.0	-78 to -50 (60)	B	-78 (5)		**25**	40-56	68:32-77:23
11	**8d** OMs	0.5	-78 (60)	B	-78 to 25 (12)		**25**	33	69:31
12	**9** Br	1.0	-78 (60)	A	-78 to 25 (12)		**26**	70	-
13	**9** Br	0.5	-78 (60)	B	-78 to 25 (12)		**26**	100	89:11

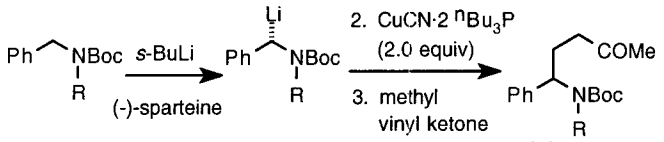
[a] CuCN·2LiCl was employed unless otherwise noted. [b] Temperature and time at which cuprate formation was achieved. [c] A = THF. B = THF/Et_2O solvent ratio (1:1, v/v) arose by deprotonation of carbamate in Et_2O followed by addition of a THF solution of CuCN·2LiCl. [d] Temperature and time at which the cuprate/electrophile reaction was allowed to proceed. [e] Transmetallation and cuprate reactions assumed to proceed with retention of configuration[1-3] from scalemic *N*-Boc-α-lithiopyrrolidine.[4] [f] Based on products purified and isolated by flash column chromatography. [g] Enantioselectivity (% er) was measured by chiral stationary phase HPLC on a CHIRALCEL™ OD column [cellulose tris(3,5-dimethylphenylcarbamate) on silica gel.

Table 4. Reactions of scalemic α-(*N*-carbamoyl)alkylcuprates, prepared from scalemic α-lithio *N*-Boc pyrrolidine with α,β-unsaturated carbonyl compounds.

entry	E^+	Cu(I) equiv[a]	temperature Cu °C (min)[b]	solvent[c]	temperature Rxn, °C (h)[d]	Product[e]	cpd No.	% yield[f]	% er[g]
1	**10**	1.0	-78 (60)	B	-78 to 35 (3)		**27**	56[h]	72:28[h]
2	**11a**	1.0	-78 (60)	A	-78 (30), 25 (12)	 **a** X = Me **b** X = OCH_2Ph **c** X = OMe	**28a**	75	-
3	**11a**	1.0	-78 (60)	B	-78 (30), 25 (12)		**28a**	54	97.6:2.4
4	**11a**	1.0 [i]	-78 (60)	B	-78 (30), 25 (12)		**28a**	55	98:2
5	**11a**	0.5	-78 (60)	B	-78 (30), 25 (12)		**28a**	53	97:3
6	**11a**	0.5	-78 (60)	B	-78 (30), 25 (12)		**28a**	91	90:10
7	**11b**	1.0	-78 (60)	B	-78 to 25 (12)		**28b**	95	50:50
8	**11b**	1.0	-78 (60)	B	-50 (10)		**28b**	39	50:50
9	**11b**	0.5	-78 (60)	B	-78 to 25 (12)		**28b**	60	50:50
10	**11c**	1.0	-78 (60)	A	-55 to 25 (12)		**28c**	60	-
11	**11c**	1.0	-78 (60)	B	-55 to 25 (12)		**28c**	72	50:50
12	**11c**	0.5	-78 (60)	B	-55 to 25 (12)		**28c**	88	60:40[j]

[a] CuCN·2LiCl was employed unless otherwise noted. [b] Temperature and time at which cuprate formation was achieved. [c] A = THF. B = THF/Et_2O, solvent ratio (1:1, v/v) arose by deprotonation of carbamate in Et_2O followed by addition of a THF solution of CuCN·2LiCl. [d] Temperature and time at which the cuprate/electrophile reaction was allowed to proceed. TMSCl (5.0 equiv) was employed as an additive. [e] Transmetallation and cuprate reactions assumed to proceed with retention of configuration[1-3] from scalemic *N*-Boc-α-lithiopyrrolidine.[4] [f] Based on products purified and isolated by chromatography. [g] Enantioselectivity (% er) was measured by chiral stationary phase HPLC on a CHIRALCEL™ OD column [cellulose tris(3,5-dimethylphenylcarbamate) on silica gel. [h] *E*:*Z* = 59:41, 72:28 er for each diastereomer. [I] $^nBu_3P$ (2.0 equiv) was added to solubilize CuCN. [j] HMPA/TMSCl (2.0 equiv) was added along with the methyl acrylate.

Table 5. Reactions of scalemic α-(*N*-carbamoyl)alkylcuprates, prepared from scalemic α-lithio *N*-Boc-*N*-ethylbenzylamine (30b) with methyl vinyl ketone.



29a R = Me **30** **31**
b R = Et

entry	temperature *s*-BuLi°C (h)[a]	solvent	temperature Rxn, °C (h)[b]	% yield[c,d]	% er[e,f] (*R*):(*S*)[g]
1	-78 (2)	Et_2O	-60 (4) to 25 (12)	57	50:50
2	-78 (3)	Et_2O	-78 to 25 (12)	53	50:50
3	-78 (2)	Et_2O	-70 (0.5), 25 (12)[h]	54	30.5:69.5
4	-78 (2)	Et_2O	-60 (0.5), 25 (12)[h]	55	28.9:79.1
5	-60 (1)	Et_2O	-60 (0.5), 25 (12)	53	32:68
6	-60 (3)	Et_2O	-60 (0.5), 25 (12)	48	35.7:66.3
7	-78 (3)	Et_2O	-78 (0.5), 25 (12)	51	28.3:71.7
8	-78 (5)	Et_2O	-78 (0.5), 25 (12	59	31:69
9	-78 (2)	THF	-60 (4) to 25 (12)	63	53.3:46.7
10	-78 (2), -60 (1)	THF	-60 (0.5), 25 (12)	79	59.5:40.5
11	-78 (2.5)[i]	THF	-78 to 25 (12)	50	53.5:46.5

[a] Temperature and time at which deprotonation was effected. Cuprate formation was achieved at -78 °C for 30 min. $R_2CuLi{\cdot}LiCN$ was employed unless noted. [b] Temperature and time at which the cuprate/electrophile reaction was allowed to proceed. TMSCl (2.5 equiv) was employed as an additive. [c] Based on products purified and isolated by chromatography. [d] Transmetallation and cuprate reactions assumed to proceed with retention of configuration[1-3] from the scalemic lithium reagent (*S*)-**30** in Et_2O.[5a-c] [e] Enantioselectivity (% er) was measured by chiral stationary phase HPLC on a CHIRALCEL™ OD column [cellulose tris(3,5-dimethylphenylcarbamate) on silica gel. [f] Major isomer reported in order of elution time (i.e., first in THF and last in Et_2O. [g] Configuration assigned in accord with the assumptions and assignments reported for **29a**.[5a-c] [h] These reactions were performed side-by side at the same time. [i] $CuCN{\cdot}2LiCl$ (1.0 equiv) was employed to afford RCuCNLi.

Experimental

General. NMR spectra were recorded as $CDCl_3$ solutions on a Bruker AC-300 or JEOL 500 MHz instrument. The ^1H NMR chemical shifts are reported as δ values in parts per million (ppm) relative to tetramethylsilane (TMS) or $CHCl_3$ (δ = 7.26 ppm) as internal standard. The ^{13}C NMR chemical shifts are reported as δ values in parts per million (ppm) downfield from TMS and referenced with respect to the $CDCl_3$ signal (triplet, centerline δ = 77.0 ppm). Infrared spectra (IR) were recorded on a Nicolet Magna 550 FT-IR spectrometer as neat samples (liquid films on NaCl plates) unless otherwise noted. Gas chromatography/mass spectrometry measurements were performed on a Hewlett-Packard 5890A GC coupled to a 5970B mass selective detector at 70 eV. Analytical thin-layer chromatography (TLC) was performed on Scientific Adsorbents Inc. silica gel plates, 200 μ mesh with F-254 indicator. Visualization was accomplished by UV light (254 nm), 4% ethanol solution of phosphomolybdic acid, 5% *p*-anisaldehyde, or 5% H_2SO_4-90% EtOH solution. Flash column chromatography was performed with 200-400 μ mesh silica gel.

THF and Et_2O were dried over sodium/benzophenone ketyl and LiCl was flame dried under vacuum in a round bottom flask before use. CuCN was used directly from the bottle as purchased from ACROS. *sec*-Butyllithium was either obtained from FMC corporation or prepared[6] from *sec*-butyl chloride and FMC's Stabi-Li-ze lithium powder. Enantiomeric ratios were determined on a Chiral Technologies, Inc. CHIRALCEL OD [cellulose tris(3,5-dimethylphenylcarbamate) on a 10 μm silica-gel substrate] chiral stationary phase HPLC column (250 x 4.0 mm, L x D).

The benzyl carbamate derived from the methyl ester of proline displays a barrier to rotation [$\Delta G^{\ddagger}$ = 17.1-17.4 Kcal/mole] that is relatively insensitive to solvent.[7] A $\Delta G^{\ddagger}$ = 17.4 Kcal/mole corresponds to a coalescence temperature of 87.3 °C while a value of $\Delta G^{\ddagger}$ = 16.7 corresponds to 61.4 °C for coalescence.[8] Compounds containing the *N*-Boc carbamate functionality generally display absorptions in the ^1H and ^{13}C NMR spectra attributable to rotomers arising from restricted rotation about the C-N bond. Additional absorptions due to rotomers are indicated within parentheses.

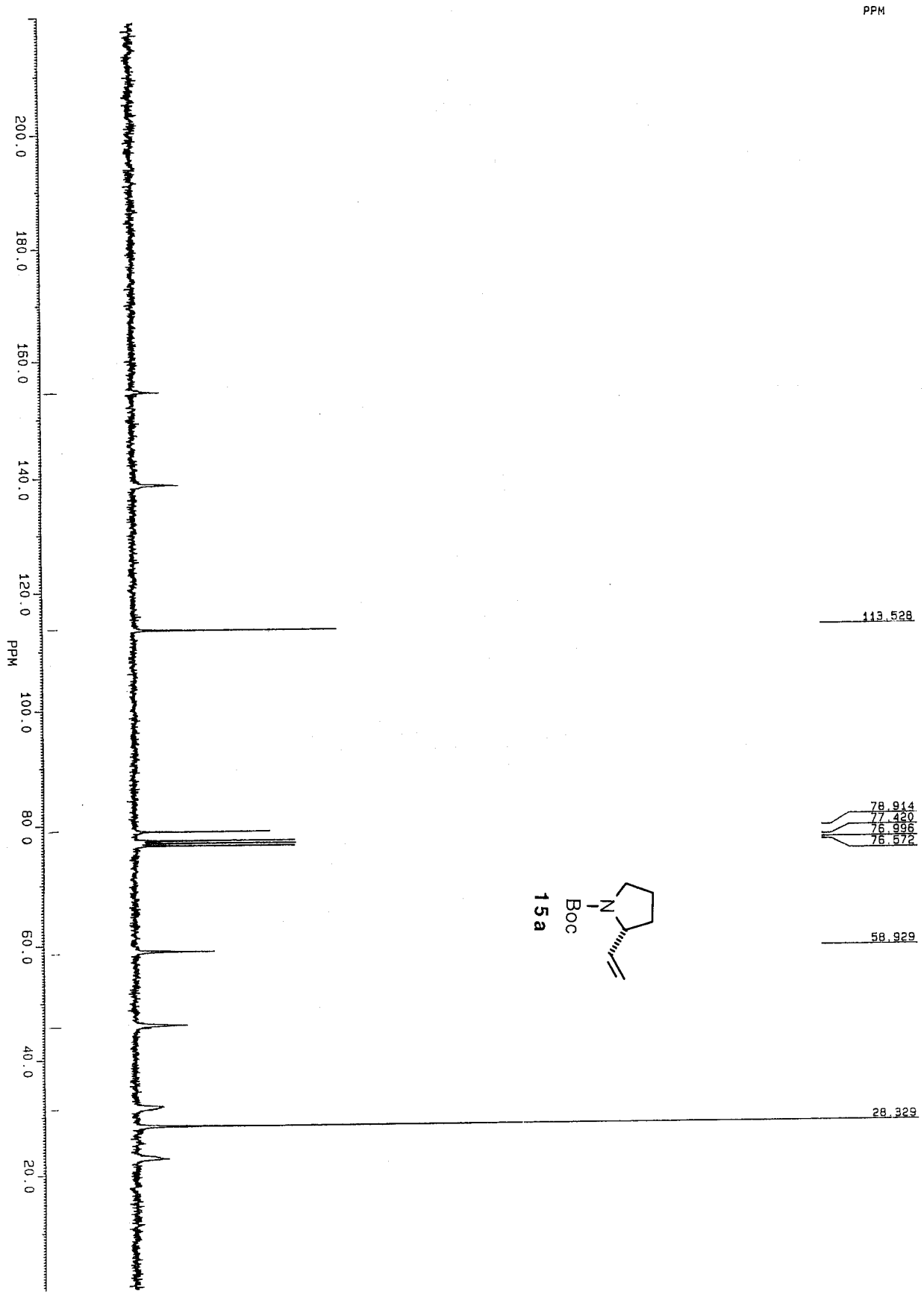
Materials: Hydroboration[9a] or hydroalumination[9b] of commercially available phenyl acetylene followed by iodination of the carbon-metal bond gave (*E*)-1-iodo-2-phenylethene (**1**). Vinyl bromide (**2a**) and 1-bromo-1-trimethylsilylethene (**2b**) were commercially available (Aldrich) while Markovnikov addition of in situ generated HI[10] to the appropriate commercially available 1-alkynes afforded 2-iodo-1-alkenes **2c** and **2d** (after silylation with $^tBuMe_2SiCl$) in good yields. Addition of HI (LiI/AcOH)[11] to commercially available ethyl propiolate (**10**) or to ethyl 2-heptynonate (for preparation of the methyl ester see reference 23b) stereoselectively gave (*Z*)-β-iodoenoates **4a-b**, respectively. Vinyl iodide **6a**,[12] triflates **3b**,[13] **7a**[13] and **7b**[14] and nonaflate **3c**[15] were prepared by established methods. Triflate **3b** could be converted into 1-iodocyclohexene **3a**.[16] Triflate **6b** was prepared by a literature procedures.[17]

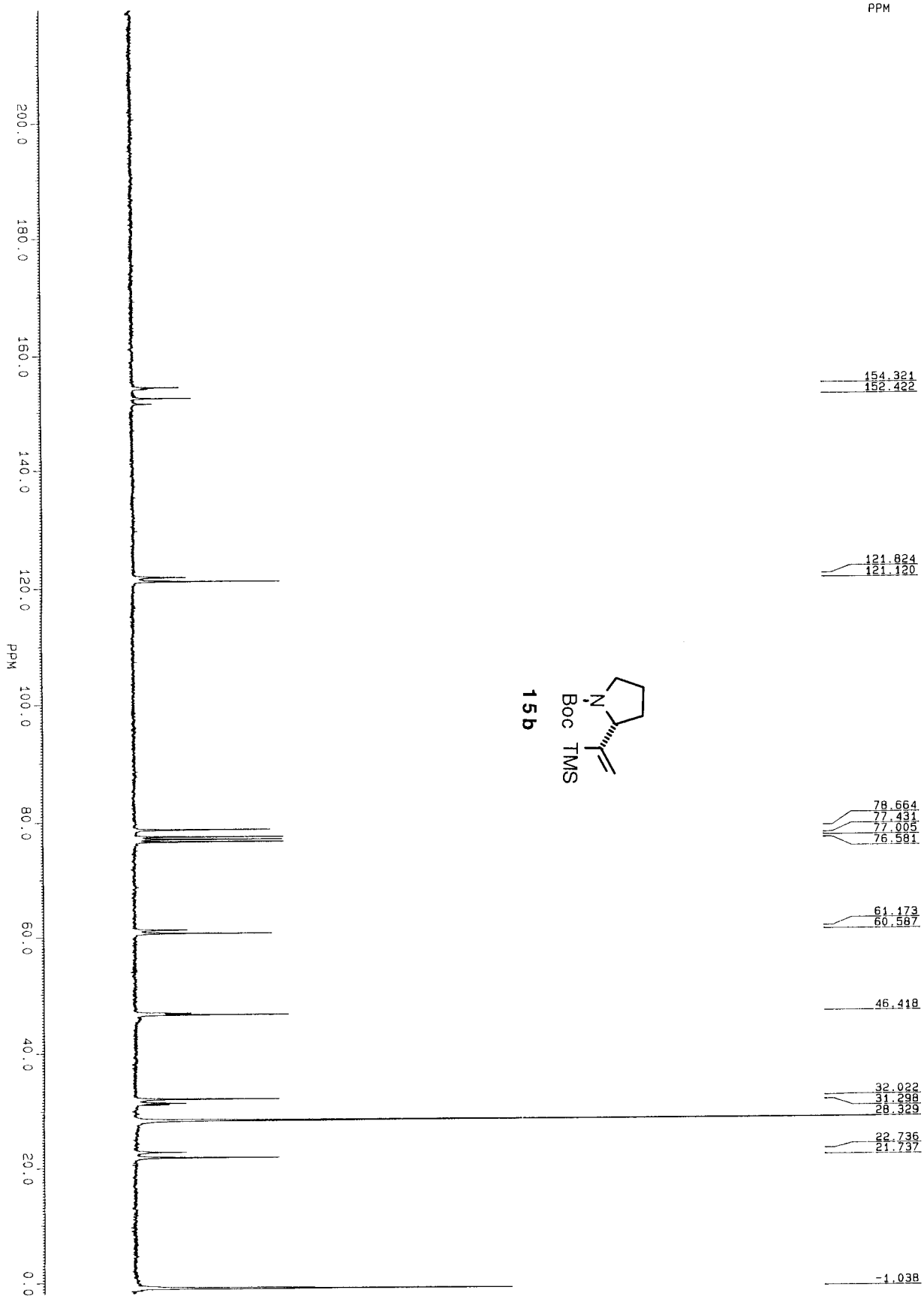
Propargyl mesylate **8b** was prepared from propargyl alcohol[18a] while propargyl mesylates **8c-d** were prepared by lithiation of the commercially available 1-alkynes followed by hydroxymethylation with paraformaldehyde[19] and mesylation.[18] Propargyl bromide **8a**, allyl bromide **9**, ethyl priopiolate **10**, methyl vinyl ketone **11a** and methyl acrylate **11c** are commercially available. Data reduction for compounds **14**,[21,22a] **15a**,[22a] **19a-b**,[20] **26**,[22b] **27**,[21] **28a**[23a] and **28c**[23b] has been reported.

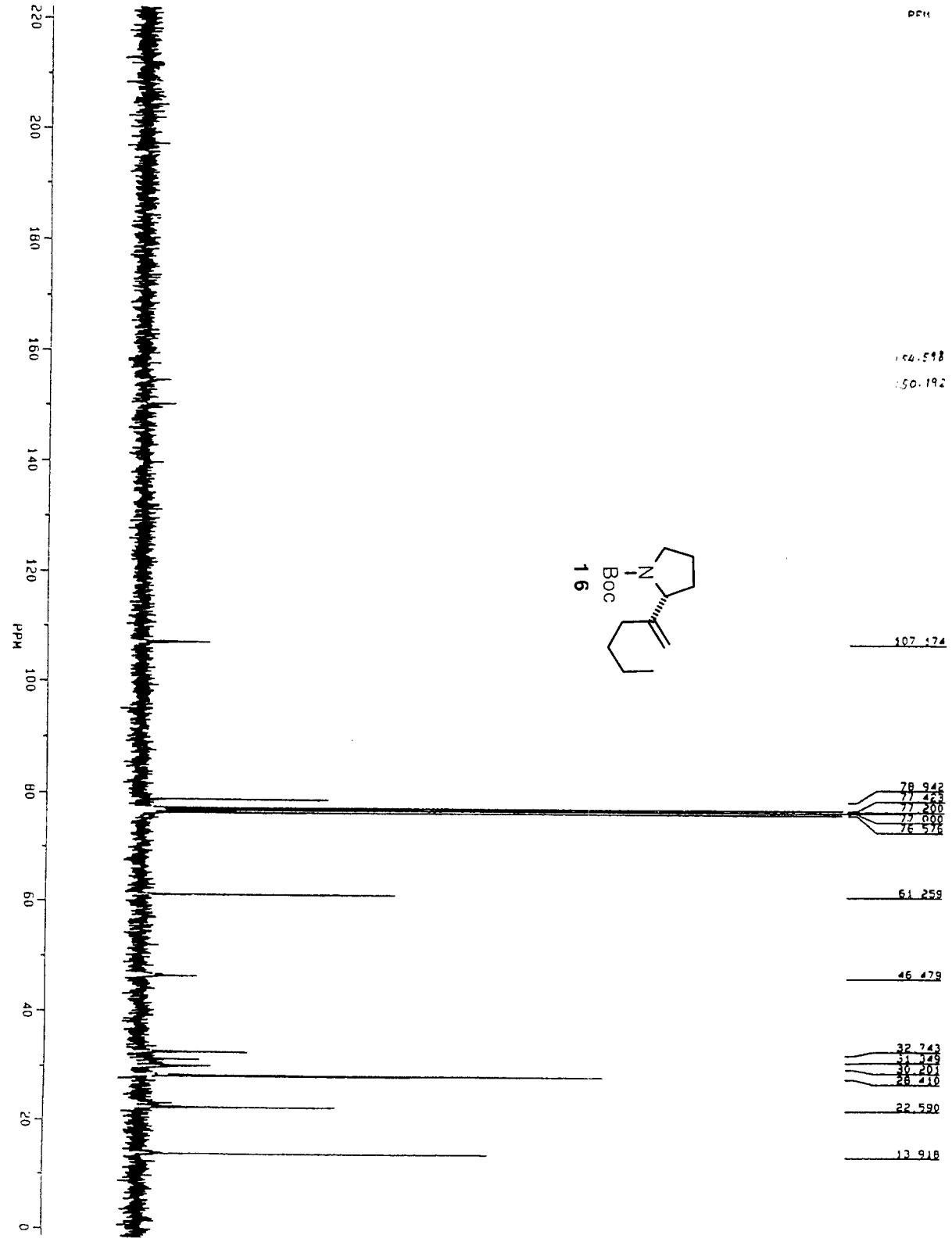
References

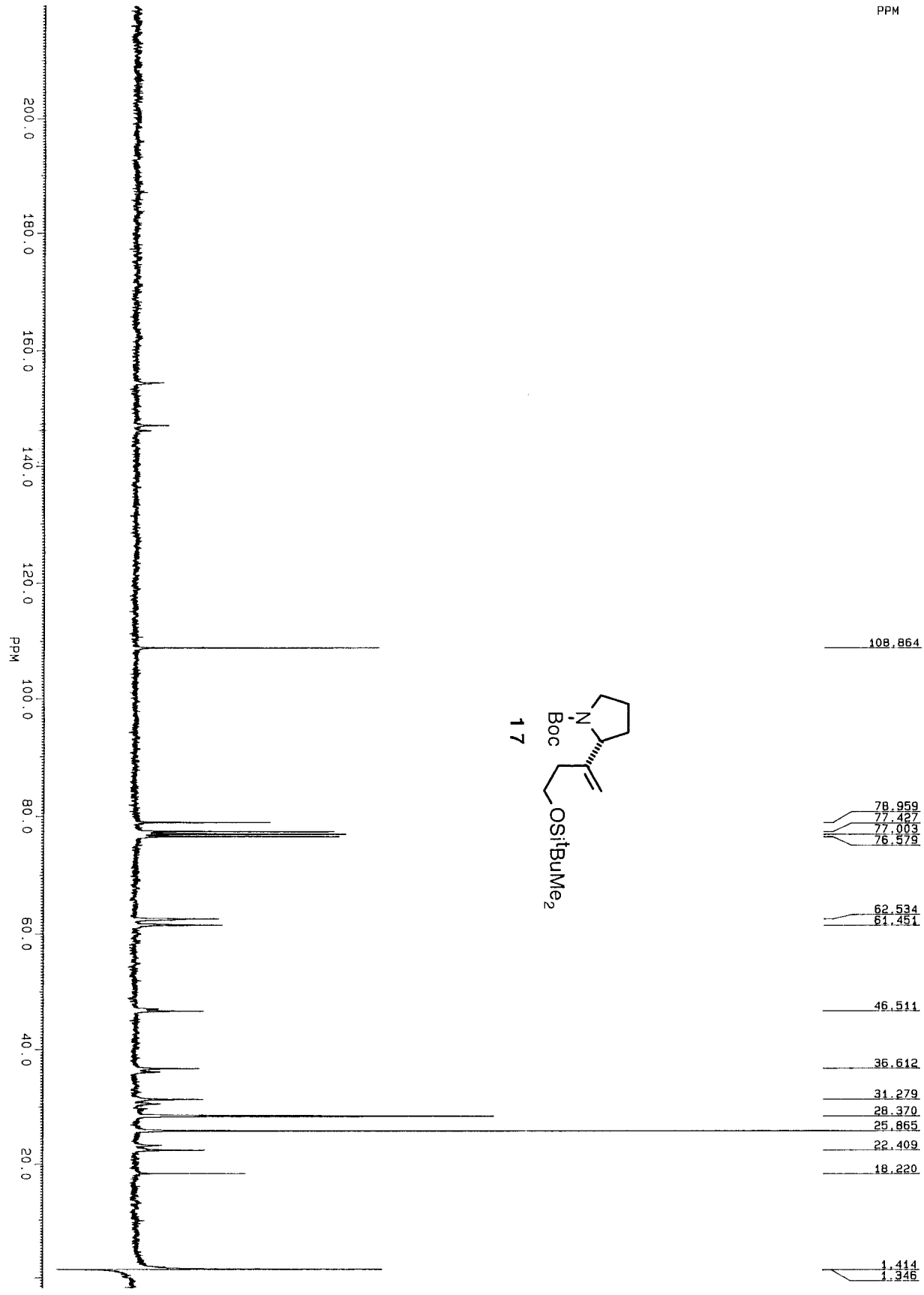
1. Hutchinson, D. K.; Fuchs, P. L. *J. Am. Chem. Soc.* **1987**, *109*, 4930-4939.
2. (a) Linderman, R. J.; Griedel, B. D. *J. Org. Chem.* **1990**, *55*, 5428-5430. (b) Linderman, R. J.; Griedel, B. D. *J. Org. Chem.* **1991**, *56*, 5491-5493. (c) Tomooka, K.; Shimizu, H.; Nakai, T. *J. Organomet. Chem.* **2001**, *624*, 364-366. (d) Rychnovsky, S. D.; Buckmelter, A. J.; Dahanukar, V. H.; Skalitzky, D. J. *J. Org. Chem.* **1999**, *64* , 6849-6860.

3. Dieter, R. K.; Topping, C. M.; Chandupatla, K. R.; Lu, K *J. Am. Chem. Soc.* **2001**, *123*, 5132-5133.
4. (a) Kerrick, S. T.; Beak, P. *J. Am. Chem. Soc.* **1991**, *113*, 9708-9710. (b) Beak, P.; Kerrick, S. T.; Wu, S.; Chu, J. *J. Am. Chem. Soc.* **1994**, *116*, 3231-3239.
5. (a)Schlosser, M.; Limat, D. *J. Am. Chem. Soc.* **1995**, *117*, 12342-12343. (b) Voyer, N.; Roby, J. *Tetrahedron Lett.* **1995**, *36*, 6627-6630. (c) Barberis, C.; Voyer, N.; Roby, J.; Chénard, S.; Tremblay, M.; Labrie, P. *Tetrahedron* **2001**, *57*, 2965-2972.
6. Dieter, R. Karl; Topping, C. M.; Nice, L. E. *J. Org. Chem.* **2001**, *66*, 2302.
7. Cox, C.; Lectka, T. *J. Org. Chem.* **1998**, *63*, 2426.
8. Grunwald, E.; Jumper, C. F.; Meiboom, S. *J. Am. Chem. Soc.* **1963**, *85*, 522.
9. (a) Brown, H. C.; Hamaoka, T.; Ravindran, N. *J. Am. Chem. Soc.* **1973**, *95*, 5786-5788. (b) Zweifel, G.; Whitney, C. C. *J. Am. Chem. Soc.* **1967**, *89*, 2753-2754.
10 Kamiya, N.; Chikami, Y.; Ishii, Y. *Synlett* **1990**, 675-676.
11. (a) Ma, S.; Lu, X.; Li, Z. *J. Org. Chem.* **1992**, *57*, 709-713. (b) Dieter, R. K.; Lu, K. *J. Org. Chem.* **2002**, *67*, 847-855.
12. Piers, E.; Grierson, J. R.; Lau, C. K.; Nagakura, I. *Can. J. Chem.* **1982**, *60*, 210-223.
13. (a) Mori, K.; Matsushima, Y. *Synthesis*, **1995**, 845-850. (b) Martinez, A. G.; Herrera, A.; Martinez, R.; Teso, E.; Garcia, A.; Osio, J.; Pargada, L.; Unanue, R. *J. Heterocyclic Chem.* **1988**, *25*, 1237-1241.
14. Summerville, R. H.; Senkler, C. A.; Schleyer, P. V. R.; Dueber, T. E.; Stang, P. J. *J. Am. Chem. Soc.* **1974**, *96*, 1100-1110.
15. (a) Lyapkalo, I. M.; Webel, M.; Reissig, H.-U. *Synlett* **2001**, 1293-1295. (b) Bellina, F.; Ciucci, D.; Rossi, R.; Vergamini, P. *Tetrahedron* **1999**, *55*, 2103-2112.
16. (a) Su, Z.; Paquette, L. A.; *J. Org. Chem.* **1995**, *60*, 764-766. (b) Martinez, A. G.; Alvavez, R. M.; Fraile, A. G. *Synthesis* **1986**, 222-224.
17. (a) Kim, H.-O.; Ogbu, C. O.; Nelson, S.; Kahn, M. *Synlett.* **1998**, 1059-1060. (b) Martines, G.; Alvarez, R. M.; Casado, M. M.; Subramanian, L. R.; Hanack, M. *Tetrahedron* **1987**, *43*, 275-279.
18. (a) Jackson, R. W.; Perlmutter, P.; Smallridge, A. J. *Aust. J. Chem.* **1988**, *41*, 1201-1208. (b) Claesson, A.; Sahlberg, C. *Tetrahedron* **1982**, *38*, 363-368.
19. (a) Brandsma, L.; "Preparative Acetylenic Chemistry", 1st Ed., Elsevier, Amsterdam, **1971**, p. 69. (b) Zwierzak, A.; Tomassy, B. *Synthetic. Commun.* **1996**, *26*, 3593-3600.
20. Dieter, R. K.; Lu, K. *J. Org. Chem.* **2002**, *67*, 847-855.
21. Dieter, R. K.; Topping, C. M.; Chandupatla, K. R.; Lu, K *J. Am. Chem. Soc.* **2001**, *123*, 5132-5133.
22. (a) Serino, C.; Stehle, N.; Park, Y. S.; Florio, S.; Beak, P. J. *J. Org. Chem.* **1999**, *64*, 1160-1165. (b) Park, S. O.; Kang, H. J.; Ko, S.; Park, S.; Chang, S. *Tetrahedron: Asymmetry* **2001**, *12*, 2621-2624.
23. (a) Dieter, R. K.; Alexander, C. W.; Nice, L. E. *Tetrahedron* **2000**, *56*, 2767-2778. (b) Dieter, R. K.; Lu, K.; Velu, S. E. *J. Org. Chem.* **2000**, *65*, 8715-8724.

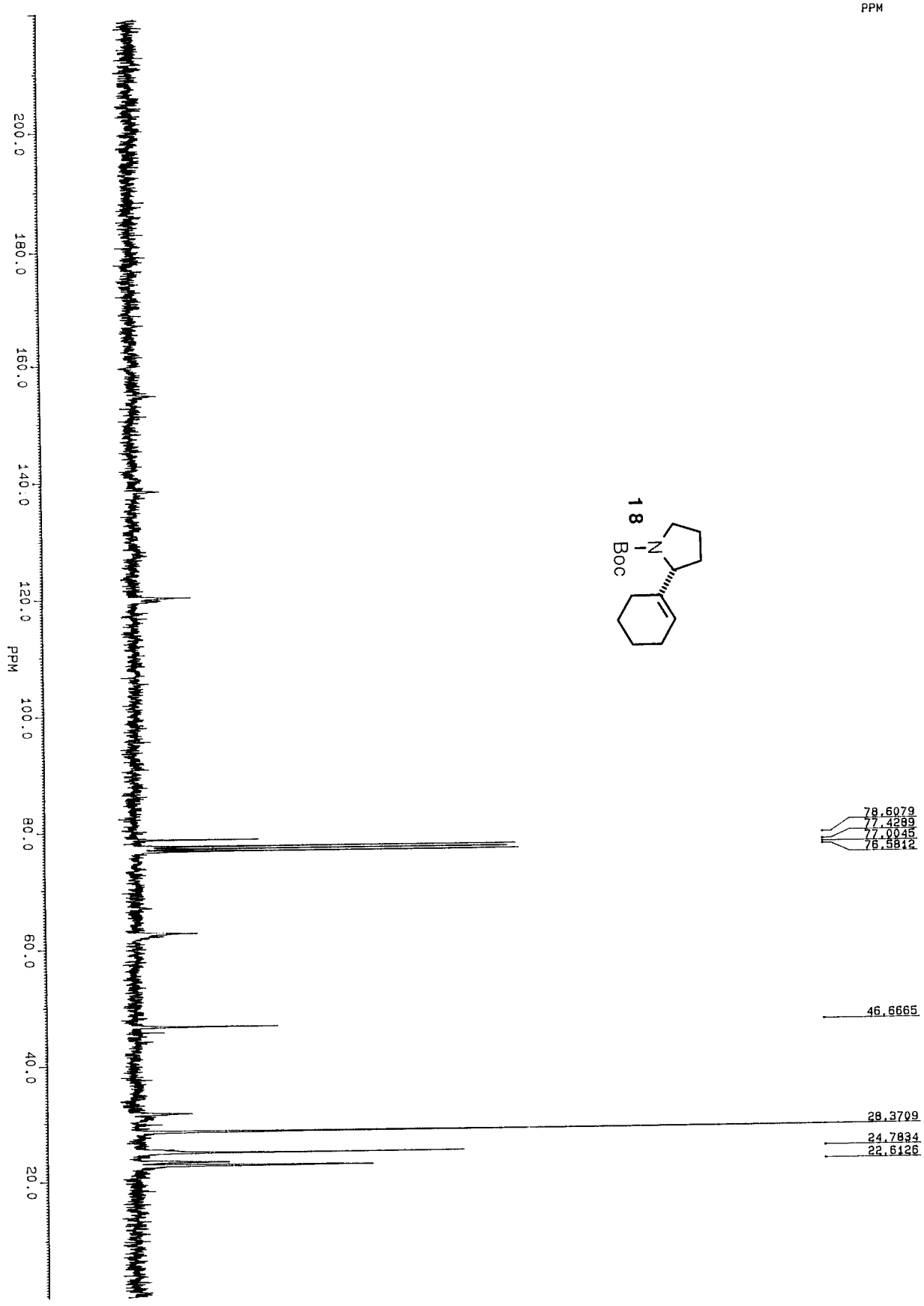

PPM
200.0
180.0
160.0
140.0
120.0
PPM
100.0
80.0
60.0
40.0
20.0
113.528
78.914
77.420
76.996
76.572
58.929
28.329
N
Boc
15a


PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
PPM
154.321
152.422
121.824
121.120
78.664
77.431
77.005
76.581
61.173
60.587
46.418
32.022
31.298
28.329
22.736
21.737
-1.038
N
Boc
TMS
15b

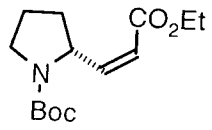

PPM
154.598
150.192
107.174
78.942
77.463
77.200
77.000
76.576
61.259
46.479
32.743
31.349
30.201
28.410
22.590
13.918
16
Boc
N
PPM
220
200
180
160
140
120
100
80
60
40
20
0


PPM
200.0
180.0
160.0
140.0
120.0
PPM
100.0
80.0
60.0
40.0
20.0
108.864
78.959
77.427
77.003
76.579
62.534
61.451
46.511
36.612
31.279
28.370
25.865
22.409
18.220
1.414
1.346
N
Boc
OSitBuMe2
17

CKR-193


PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
18
N
Boc
78.6079
77.4289
77.0045
76.5812
46.6665
28.3709
24.7834
22.6126

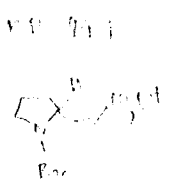
kishan1.5



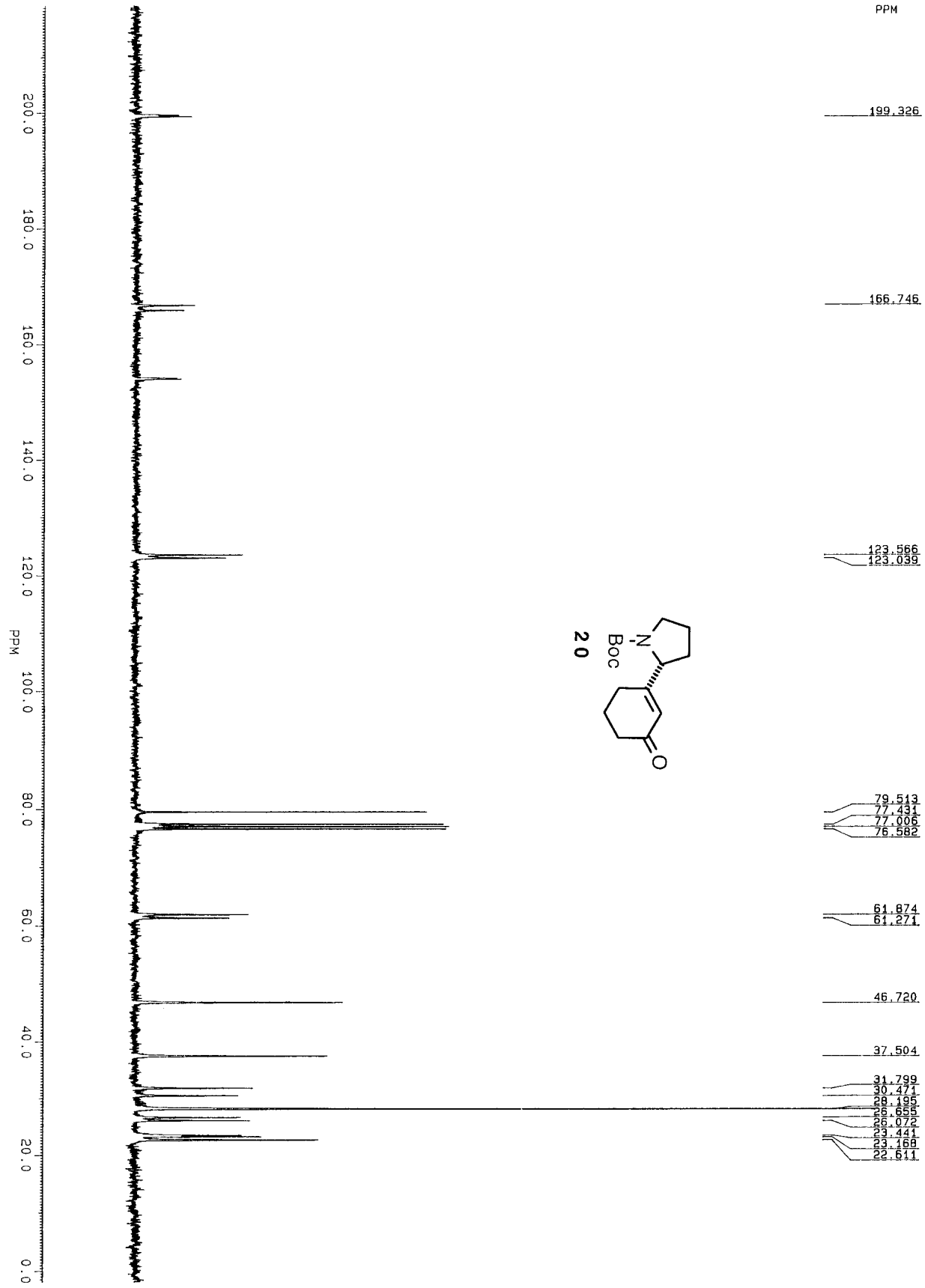
19a

X : parts per Million : 13C

```
---- ACQUISITION PARAMETERS ----
File Name        = kishan1.5
Author           = Clemson NMR
Sample ID        = 1
Content          = single pulse decoupled
Creation Date    = 11-DEC-2001 19:00:58

Revision Date    = 11-DEC-2001 19:01:52
Spec Site        = Eclipse+ 500

Spec Type        = DELTA_NMR
Data Format      = 1D COMPLEX
Dimensions       = X
Dim Title        = 13C
Dim Size         = 32768
Dim Units        = [ppm]
X_domain         = 13C
X_offset         = 100[ppm]

X_freq           = 125.77787547[MHz]
X_sweep          = 31.44654088[kHz]
X_points         = 32768
X_resolution     = 0.95970156[Hz]
X_prescans       = 4
Scans            = 267
Recvr_gain       = 27
Solvent          = CHLOROFORM-D
Spin_get         = 14[Hz]
Temp_get         = 22.2[dC]
Experiment       = single_pulse_dec
Relaxation_delay= 2[s]

---- PROCESSING PARAMETERS ----
dc_balance
sexp : 1.5[Hz]
fft : 1
machinephase
ppm
dc_correct
```

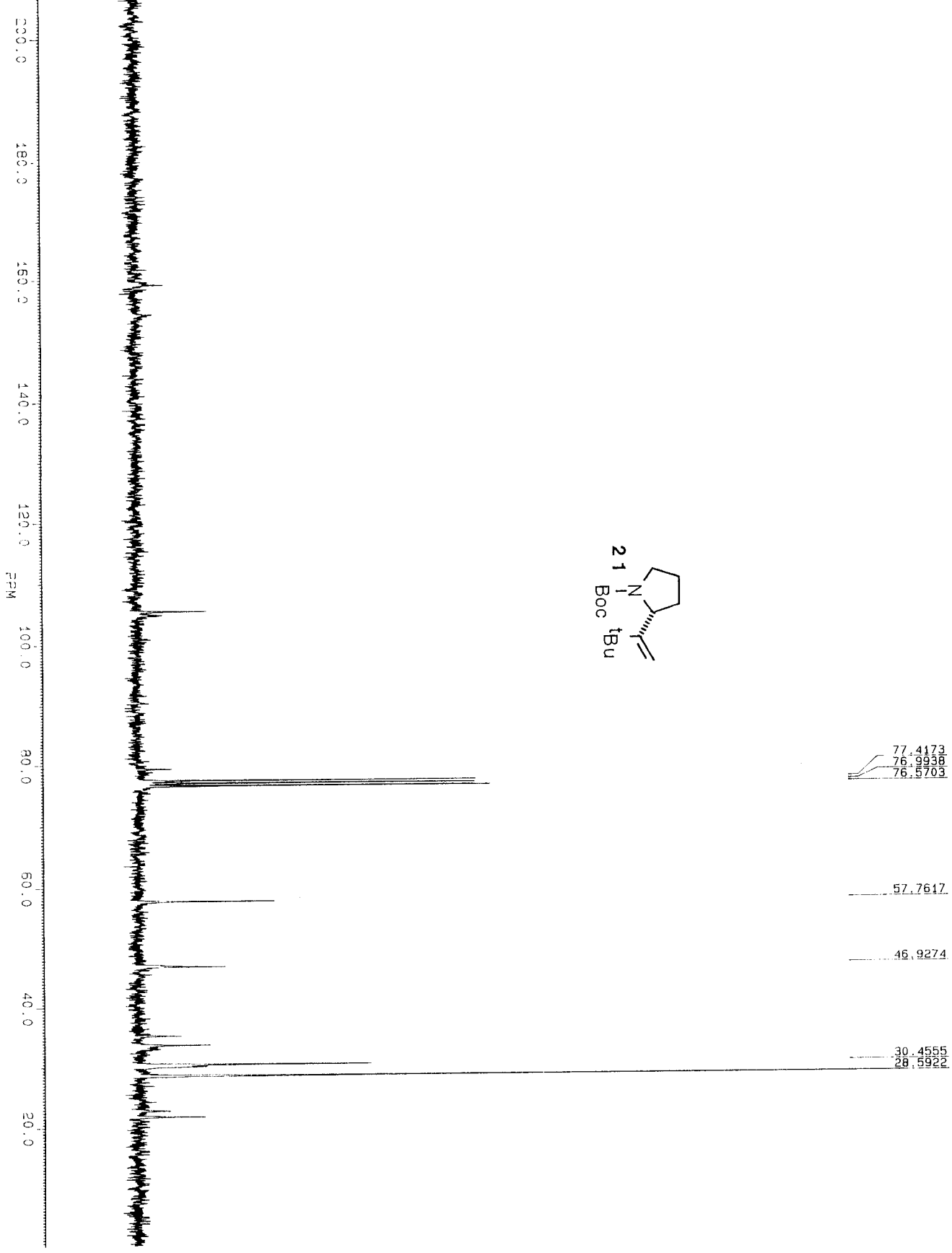


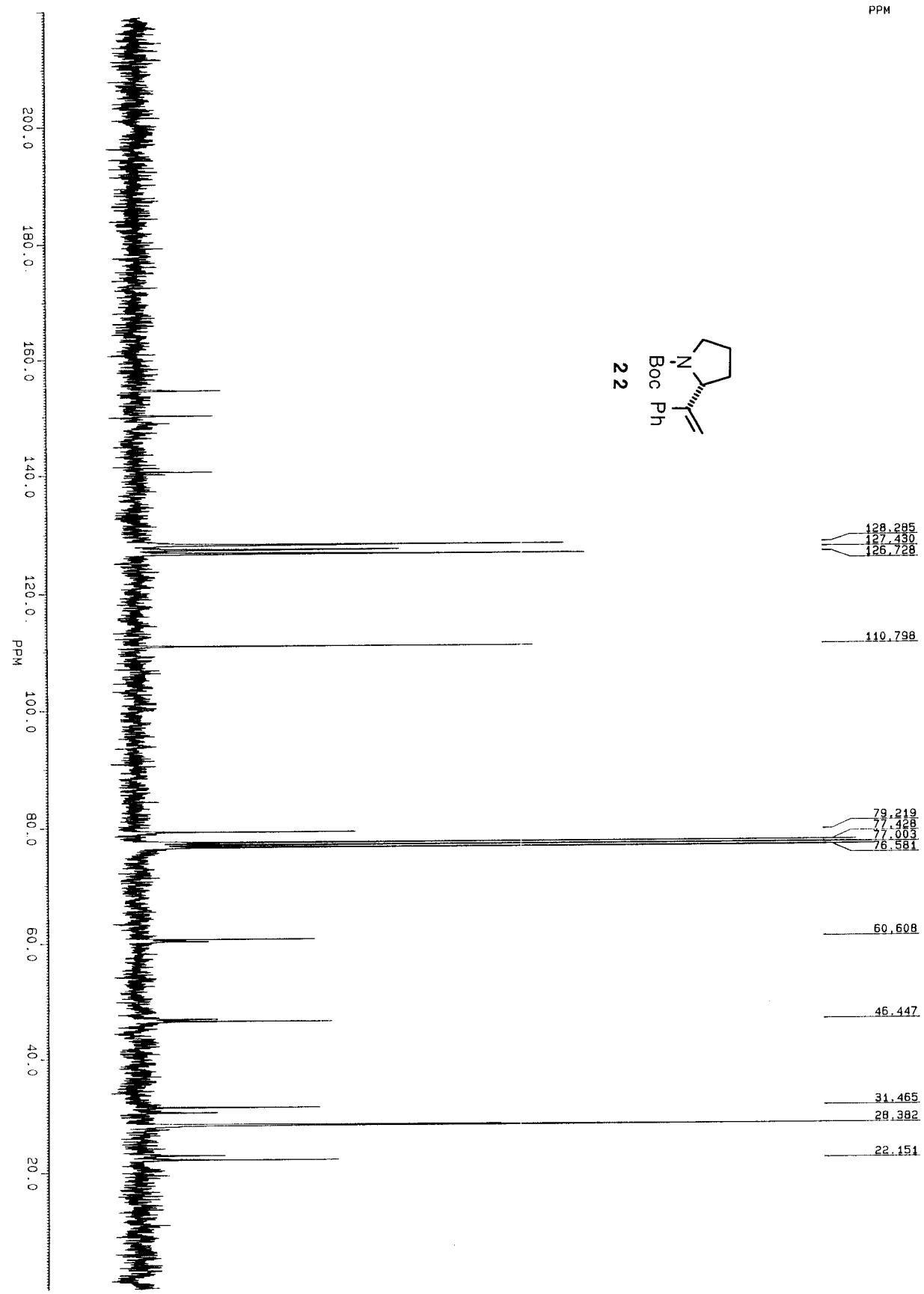
JEOL

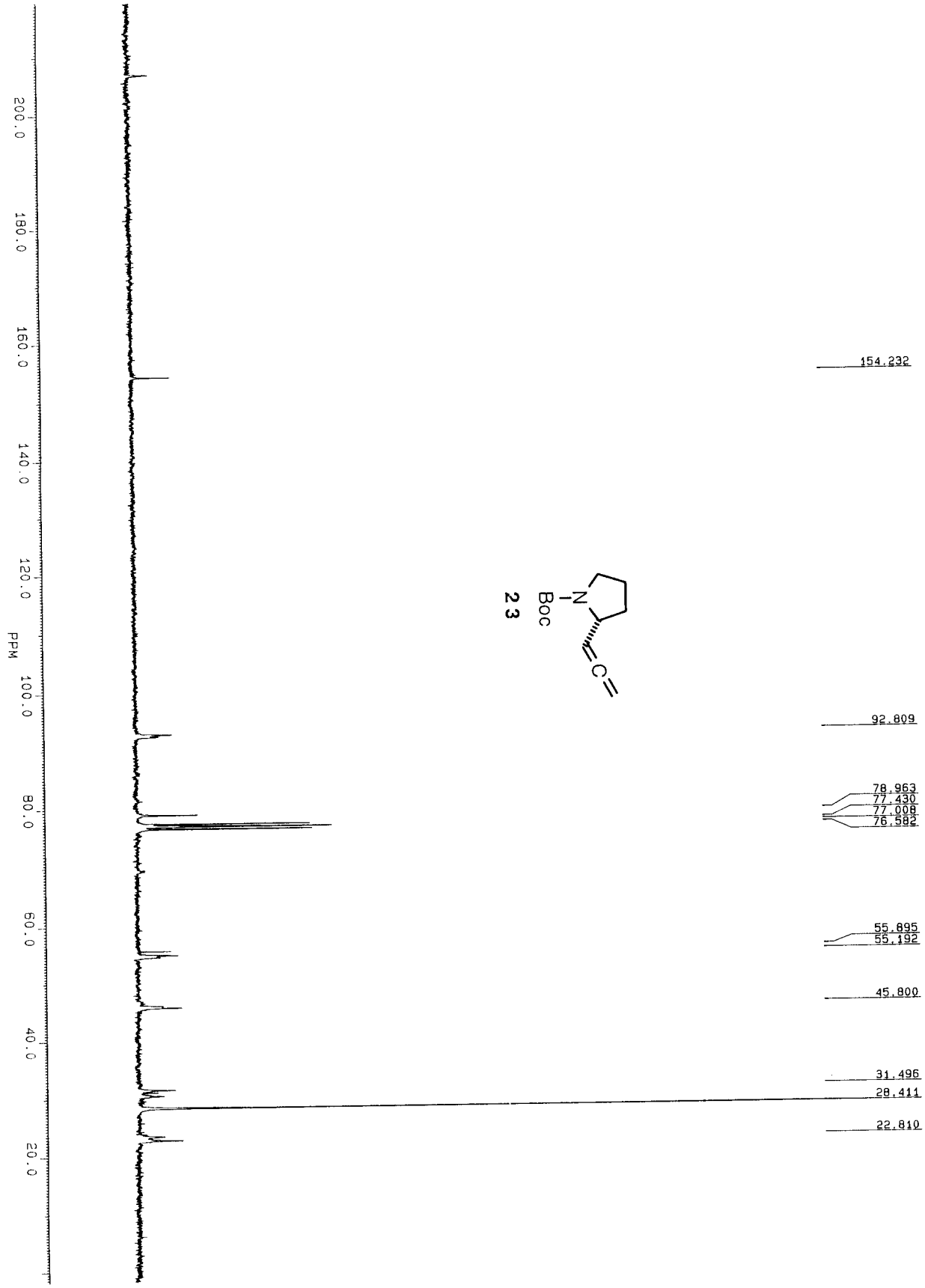

PPM
199.326
166.746
123.566
123.039
79.513
77.431
77.006
76.582
61.874
61.271
46.720
37.504
31.799
30.471
28.195
26.655
26.072
23.441
23.168
22.611
PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
N
Boc
O

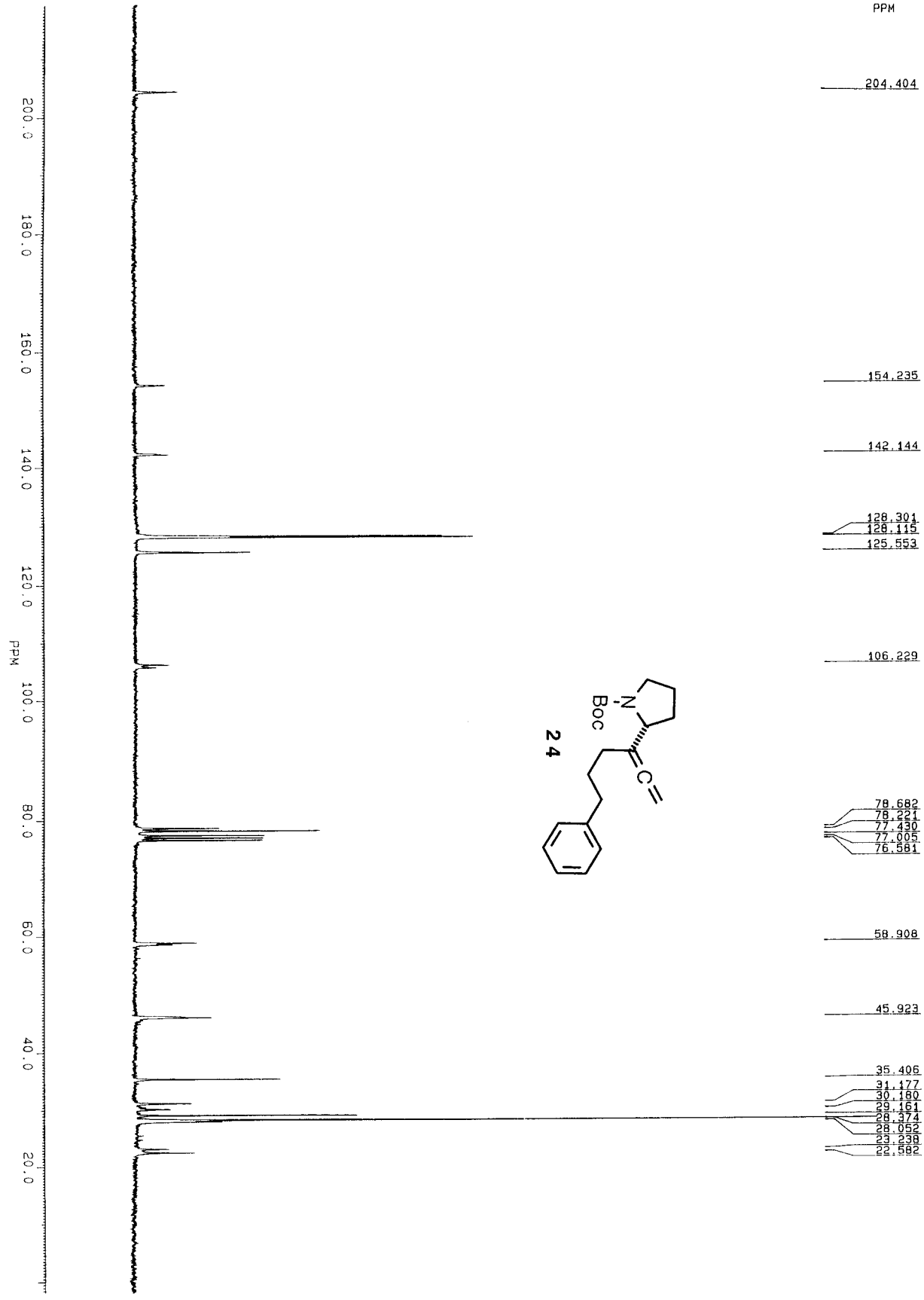
20

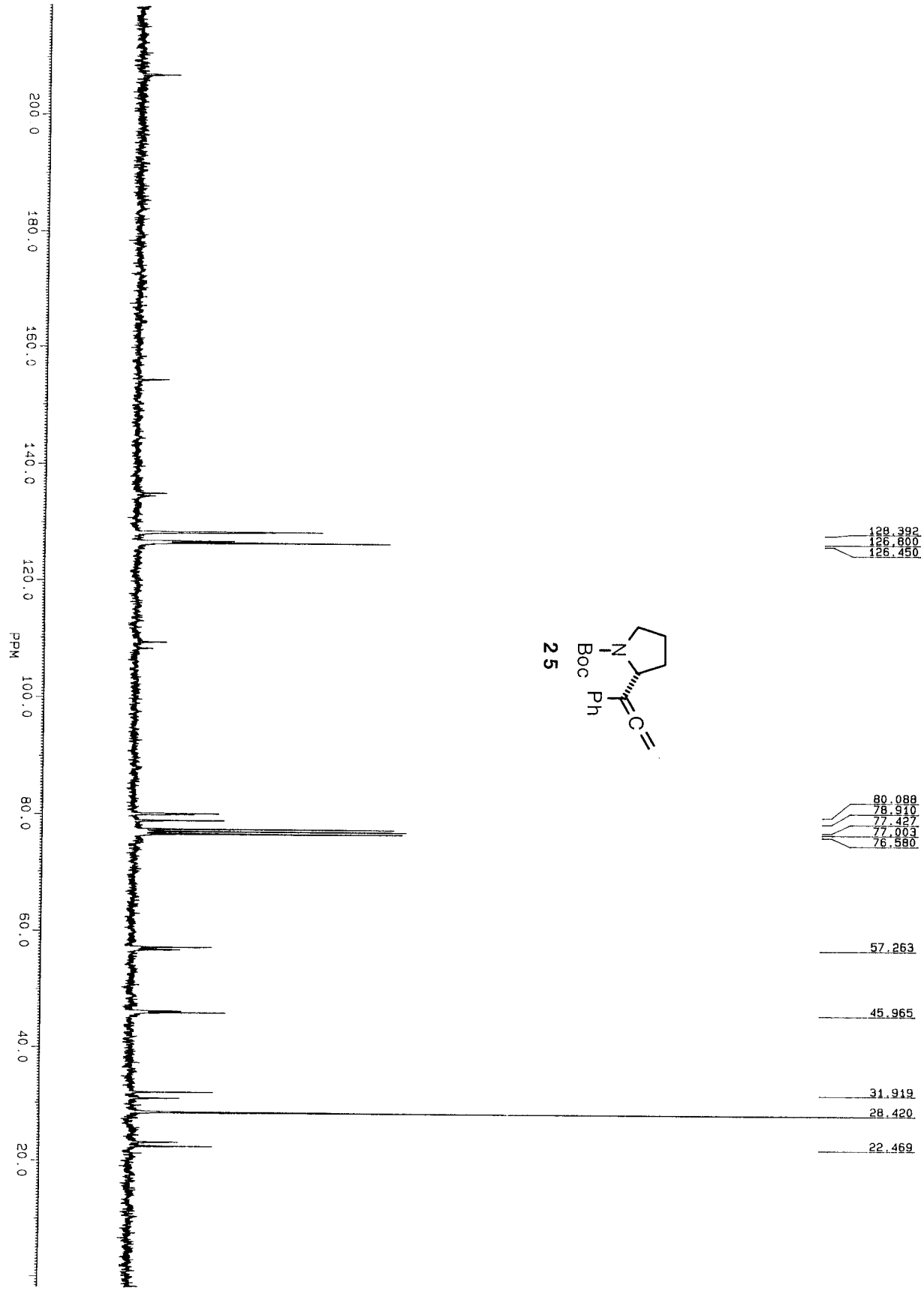
CKR-151

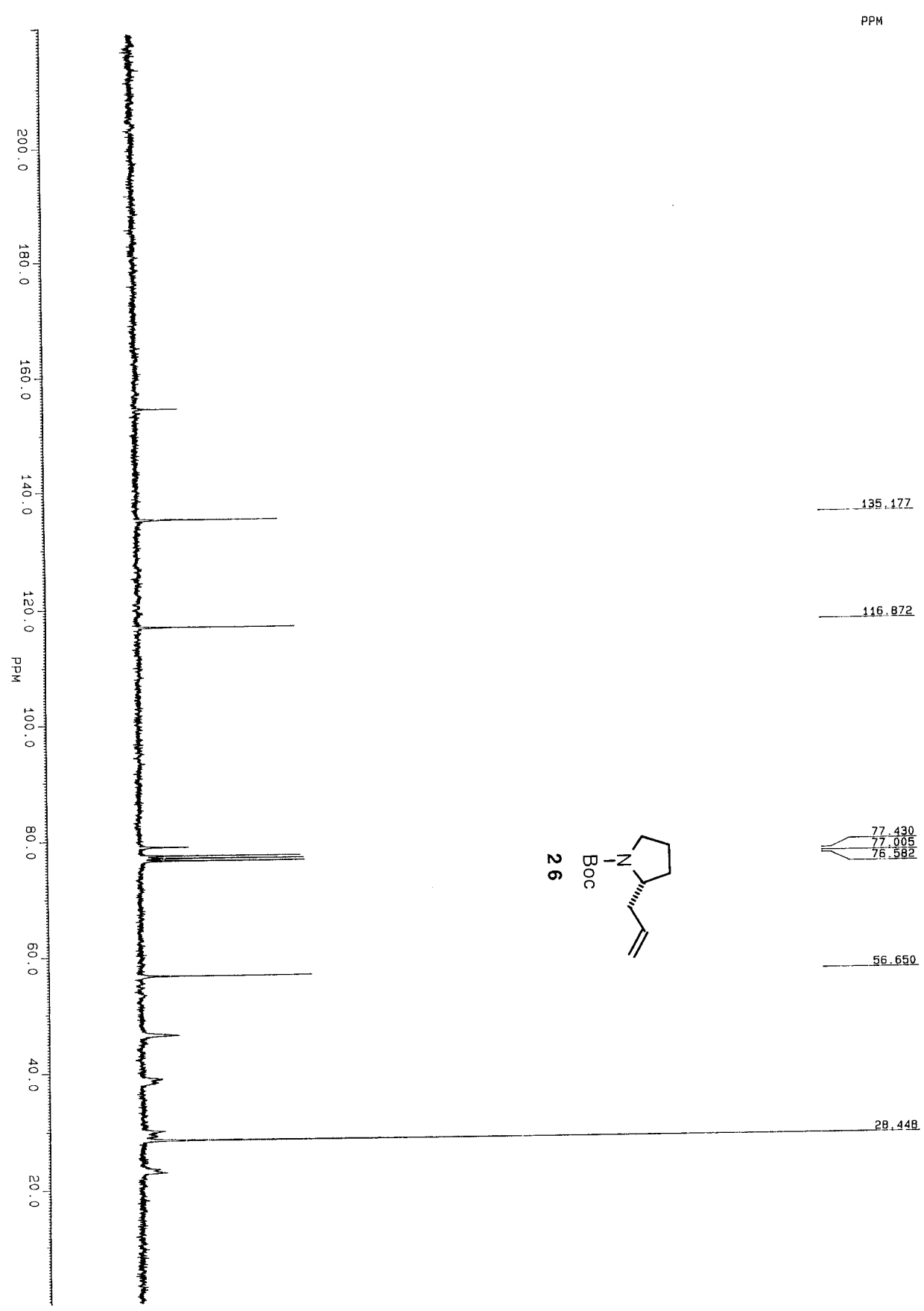

77.4173
76.9938
76.5703
57.7617
46.9274
30.4555
28.5922
FPM
220.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
2 1
N
Boc
tBu

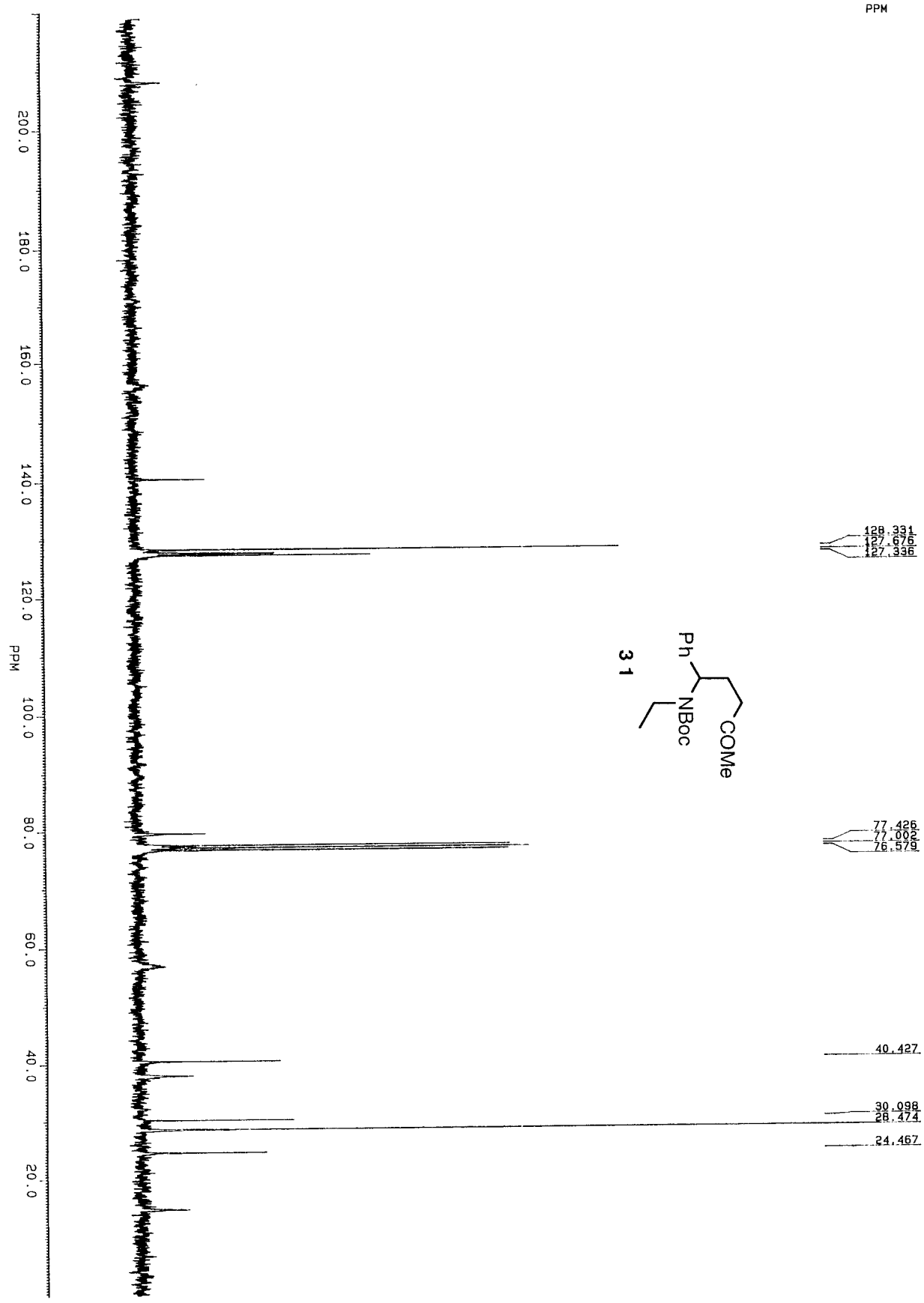

PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
N
Boc Ph
22
128.285
127.430
126.728
110.798
79.219
77.428
77.003
76.581
60.608
46.447
31.465
28.382
22.151

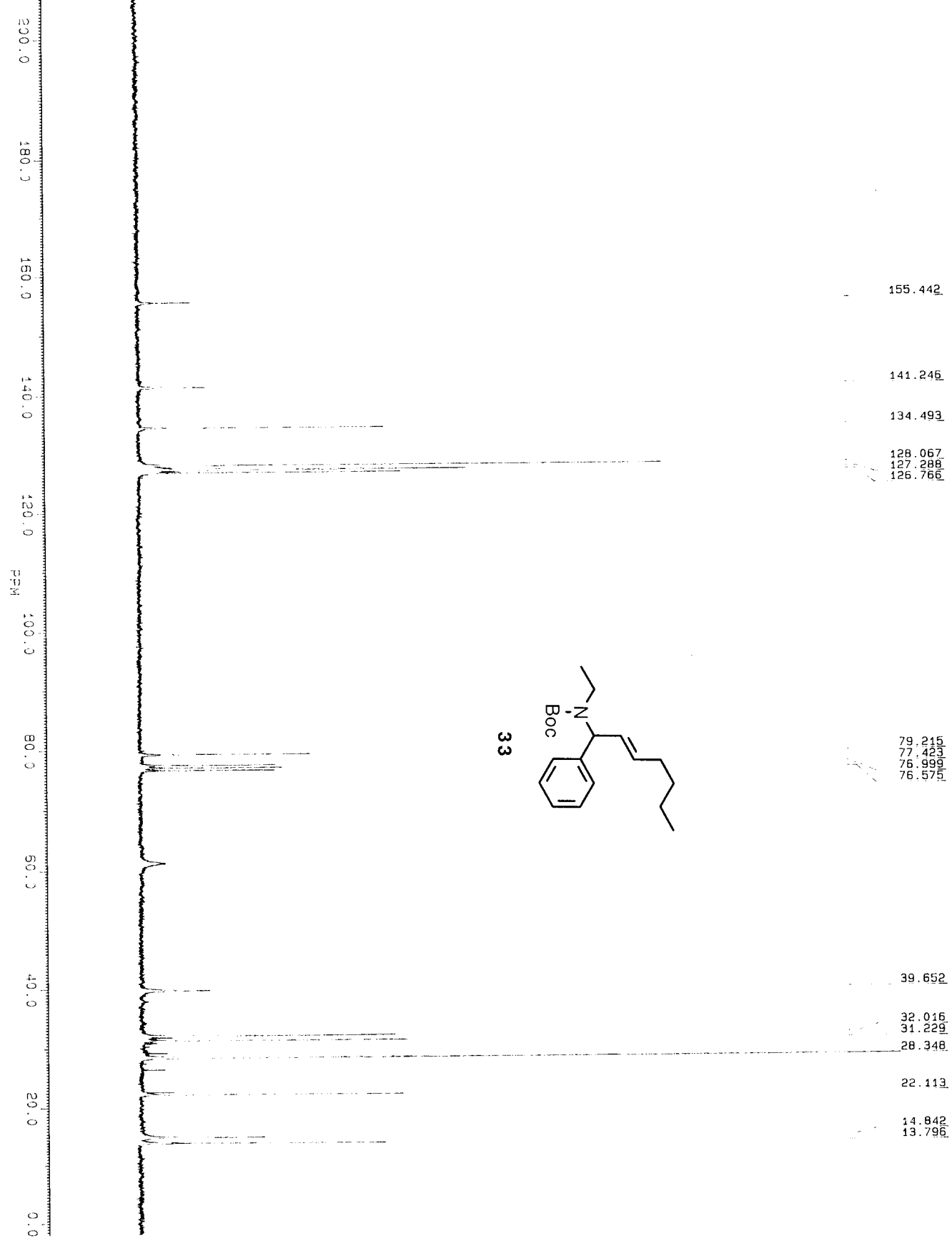

200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
154.232
92.809
78.963
77.430
77.008
76.582
55.895
55.192
45.800
31.496
28.411
22.810
N
Boc
23


PPM
204.404
154.235
142.144
128.301
128.115
125.553
106.229
78.682
78.221
77.430
77.005
76.581
58.908
45.923
35.406
31.177
30.180
29.161
28.374
28.052
23.238
22.582
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
N
Boc
24

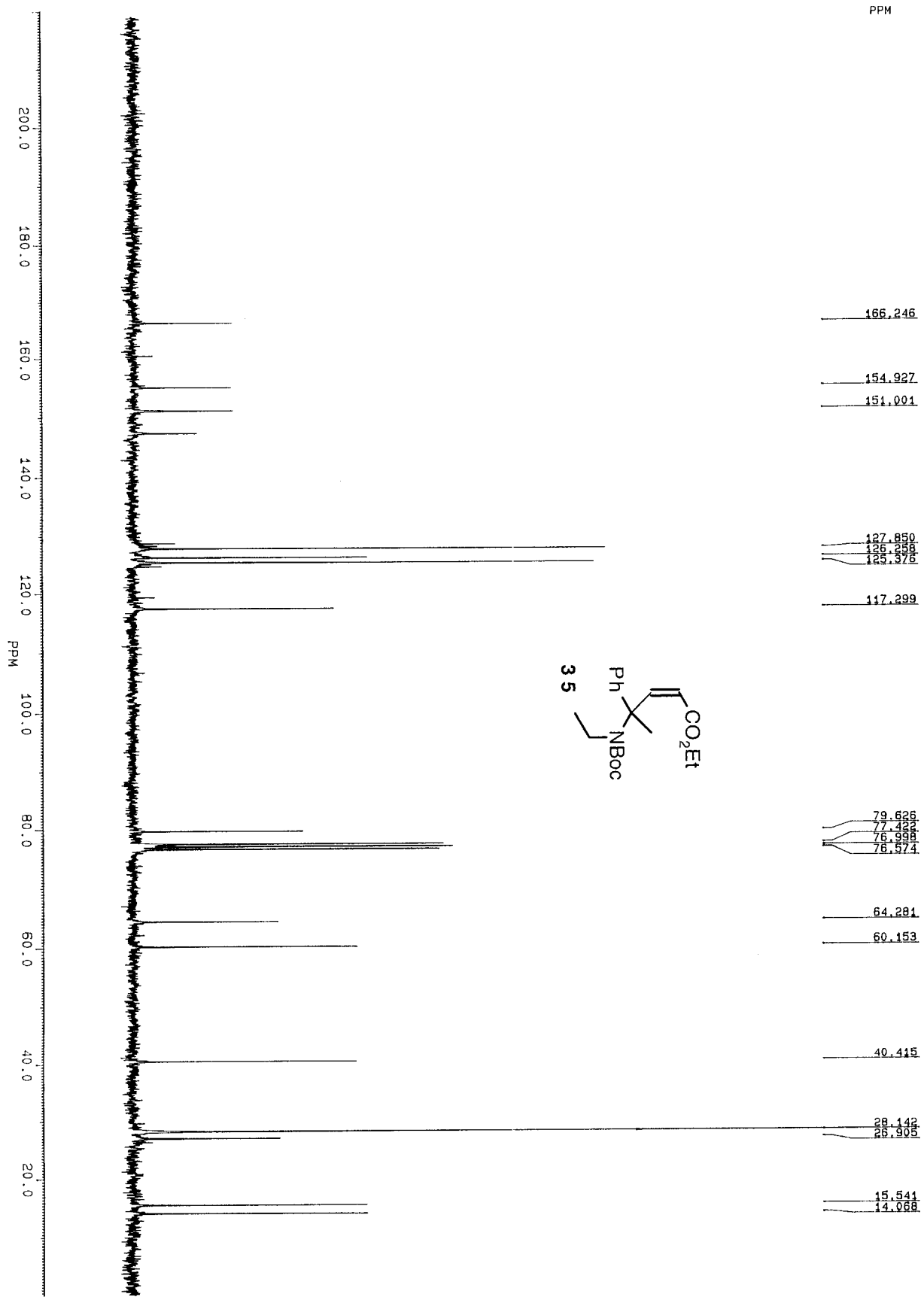

200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
128.392
126.600
126.450
80.088
78.910
77.427
77.003
76.580
57.263
45.965
31.919
28.420
22.469
N
Boc
Ph
C
25

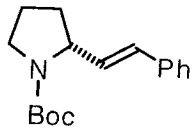

PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
135.177
116.872
77.430
77.005
76.582
56.650
28.448
N
Boc
26


PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
PPM
128.331
127.676
127.336
77.426
77.002
76.579
40.427
30.098
28.474
24.467
31
Ph
NBoc
COMe


155.442
141.246
134.493
128.067
127.288
126.766
79.215
77.423
76.999
76.575
39.652
32.016
31.229
28.348
22.113
14.842
13.796
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
0.0
PPM
N
Boc

33

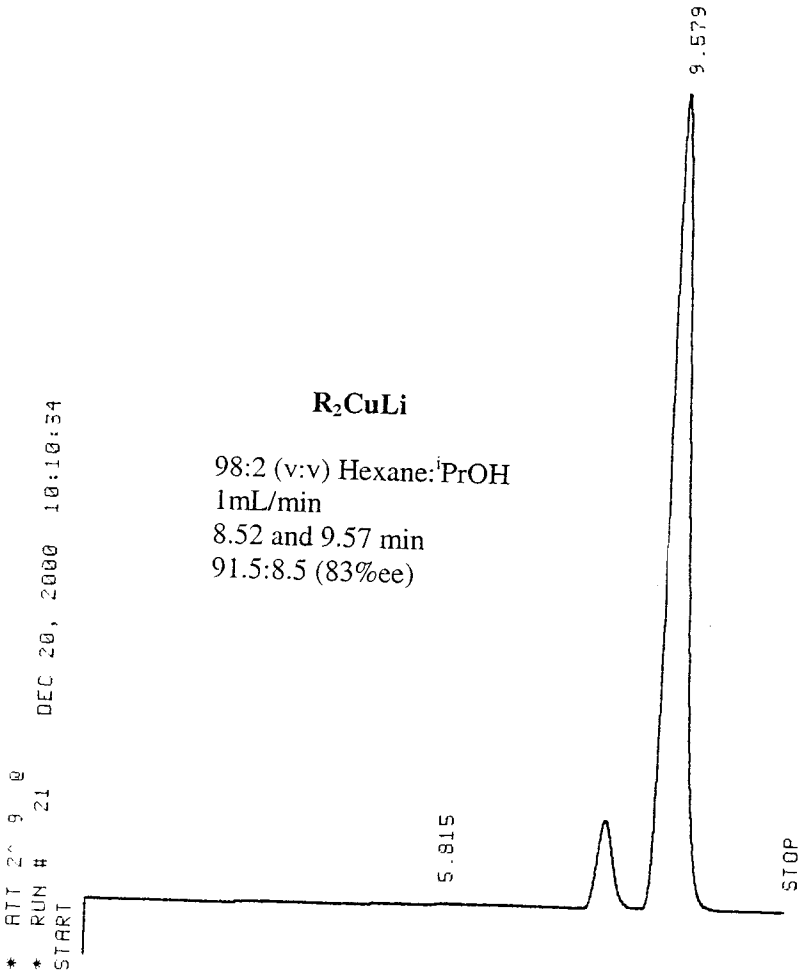

PPM
200.0
180.0
160.0
140.0
120.0
100.0
80.0
60.0
40.0
20.0
166.246
154.927
151.001
127.850
126.256
125.376
117.299
79.626
77.422
76.998
76.574
64.281
60.153
40.415
28.142
26.905
15.541
14.068
3 5
Ph
NBoc
CO2Et



14

R_2CuLi

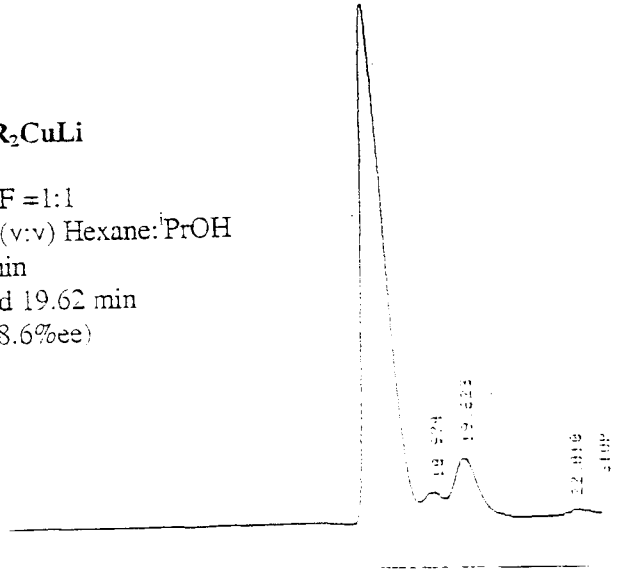
98:2 (v:v) Hexane:^{i}PrOH
1mL/min
8.52 and 9.57 min
91.5:8.5 (83%ee)

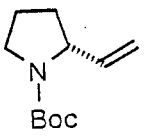



```
* ATT 2^ 9 0
* * RUN #   21     DEC 20, 2000  10:10:34
START

RUN#    21          DEC 20, 2000  10:10:34

AREA%
        RT       AREA TYPE  WIDTH       AREA%
     5.815     161888   BB    .203     .18347
     8.527    7458115   BV    .301    8.45242
     9.579   80616512   VB    .361   91.36413

TOTAL AREA=8.8237E+07
MUL FACTOR=1.0000E+00
```

R_2CuLi

Et_2O:THF =1:1
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
17.65 and 19.62 min
38:12 (88.6%ee)

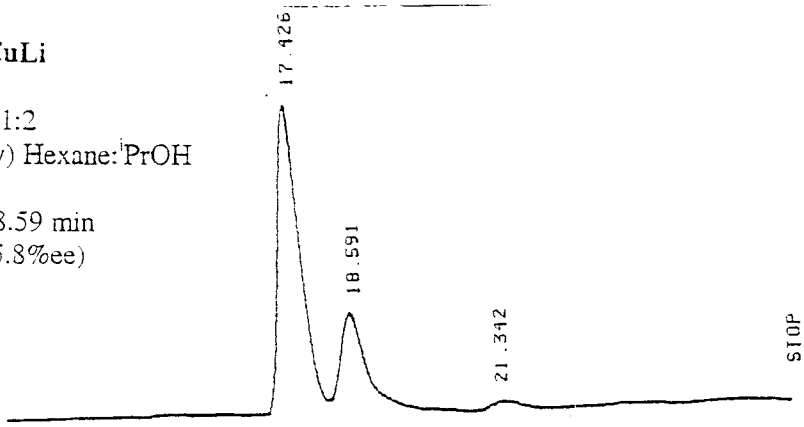




15a

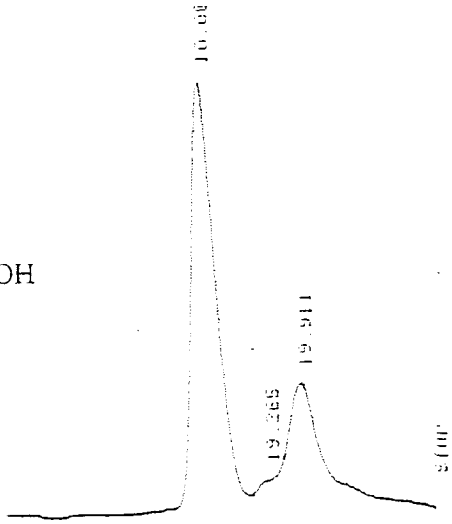
R_2CuLi

Et_2O:THF =1:2
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
17.43 and 18.59 min
72.9:27.1(45.8%ee)



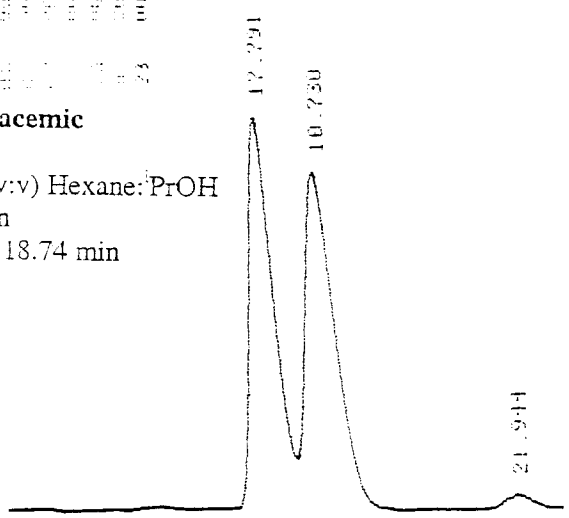
R_2CuLi

Et_2O:THF =1:4
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
18.00 and 19.91 min
71.5:28.5 (43%ee)



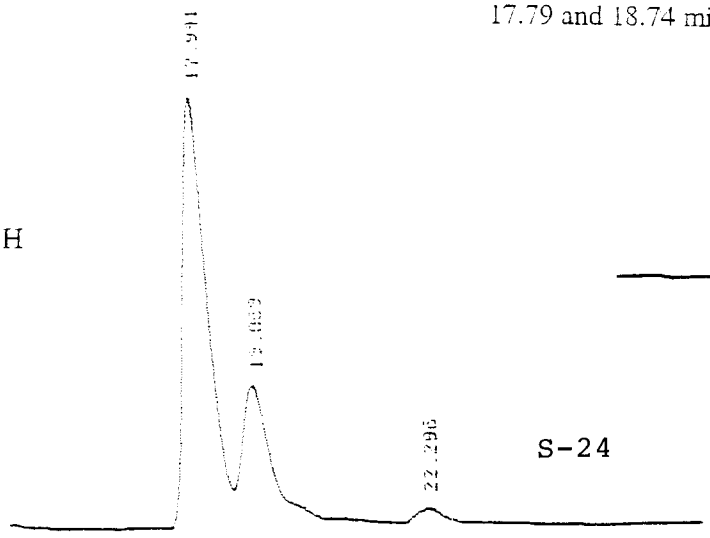
Racemic

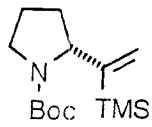
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
17.79 and 18.74 min



RCuCNLi

Et_2O:THF =1.3:1
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
17.94 and 19.07 min
72.5:27.5(45.%ee)





15b

Racemic

99.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
9.37 and 10.39 min

RUN# 249 MAY 9, 2001 21:29:44

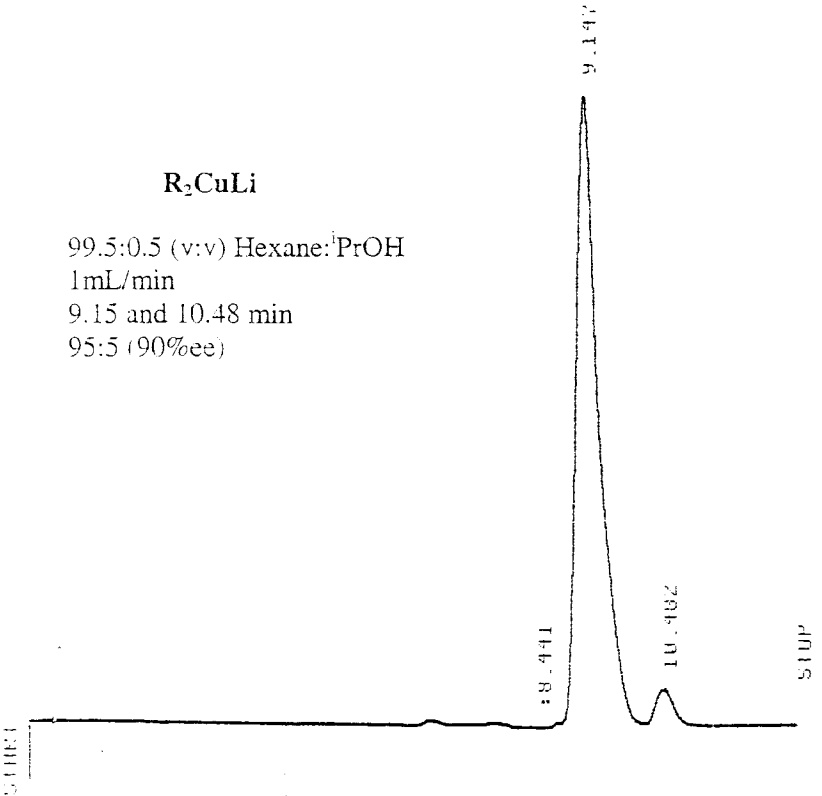
	PT	AREA TYPE	WIDTH	AREA%
	3.320	BV	.196	.42992
	3.875	VV	.156	1.61381
	4.143	VV	.195	1.38125
	9.370	BV	.323	43.96613
	10.390	VB	.399	52.70968

AREA%

TOTAL AREA=1.37972E+07
MUL FACTOR=1.00000E+00

R_2CuLi

99.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
9.15 and 10.48 min
95:5 (90%ee)

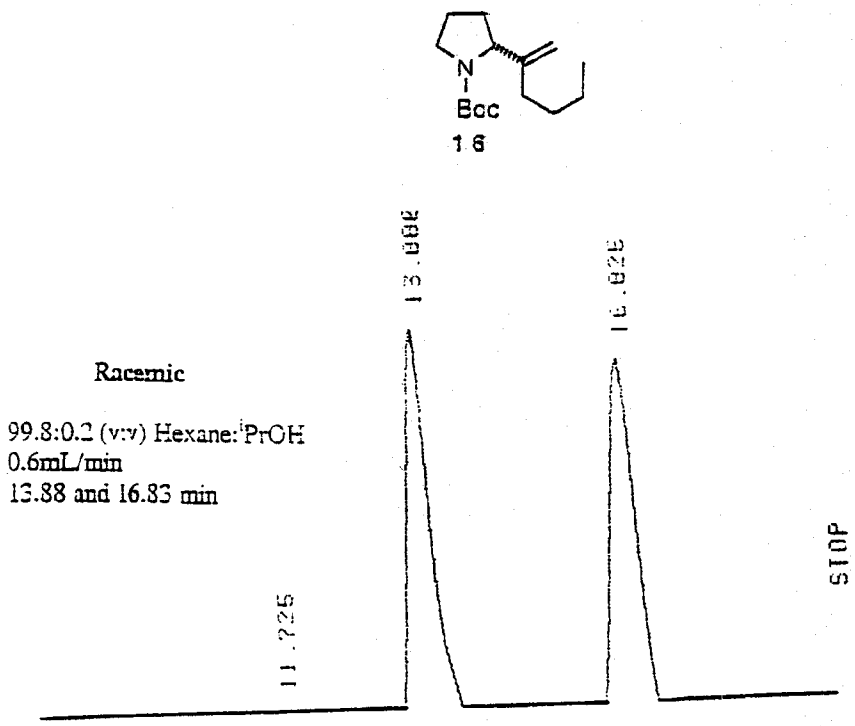


RUN# 98 JAN 6, 1981 18:15:16

AREA%

	RT	AREA TYPE	WIDTH	AREA%
	9.147	18531698 BV	.422	95.05109
	10.482	955510 VV	.374	4.94900

TOTAL AREA=1.95087E+07
MUL FACTOR=1.00000E+00



Racemic

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
13.88 and 16.83 min

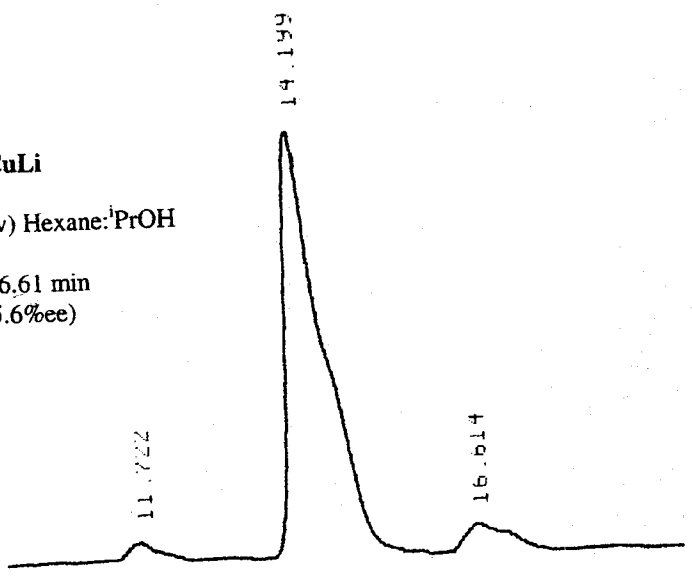
RUN# 52 JAN 2, 1901 10:26:45

AREA%	RT	AREA	TYPE	WIDTH	AREA%
	5.346	82648	BP	.207	.37938
	11.726	22272	PB	.069	.12534
	13.880	10977720	BB	.426	50.46221
	16.825	10671128	BB	.452	49.04309

TOTAL AREA=2.17698+07
MUL FACTOR=1.0000E+00

R_2CuLi

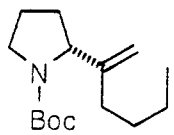
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
14.20 and 16.61 min
97.8:2.2 (95.6%ee)



RUN# 799 JAN 28, 1901 18:37:07

AREA%	RT	AREA	TYPE	WIDTH	AREA%
	.521	31211	PB	.292	.18005
	5.218	414888	VV	.439	2.39336
	11.722	533960	BP	.496	3.08025
	14.199	16317672	PV	.620	94.13158
	16.614	37230	PB	.152	2.21477

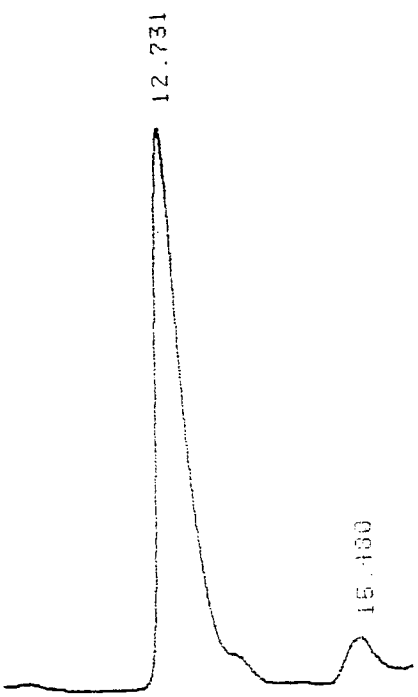
TOTAL AREA=1.7335E+07
MUL FACTOR=1.0000E+00



16

R_2CuLi

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
12.73 and 15.48 min
92.1:7.9 (84.2%ee)

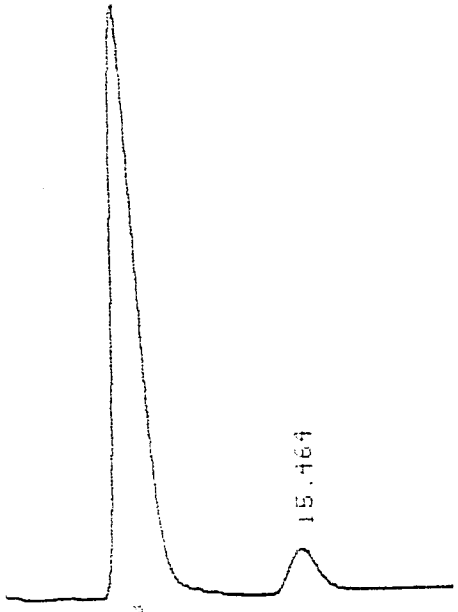


RUN# 175 JAN 6, 1901 11:04:50

	RT	AREA	TYPE	WIDTH	AREA%
	5.181	100312	VV	.214	.52153
	5.680	122803	VV	.188	.63846
	5.934	257850	VP	.233	1.34057
	12.731	16824224	BP	.495	87.46986
	15.480	1442126	BV	.520	7.49767
	16.236	487000	VV	.414	2.53193

RCuCNLi

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
12.95 and 15.46 min
92:8 (84%ee)



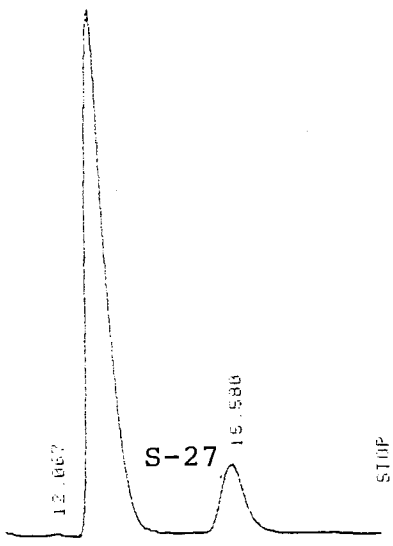
RUN# 151 JAN 5, 1901 20:02:20

	RT	AREA	TYPE	WIDTH	AREA%
	5.046	44289	VV	.131	.24543
	5.292	114463	VV	.219	.63430
	5.667	200347	VV	.252	1.11023
	10.776	649594	VV	.374	3.59974
	12.947	15732472	BV	.432	87.18186
	15.464	1304420	BV	.458	7.22847

TOTAL AREA=1.80046E+07
MUL FACTOR=1.00000E+00

RCuCNLi (used CuCl)

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
12.93 and 15.58 min
86.5:13.5 (73%ee)



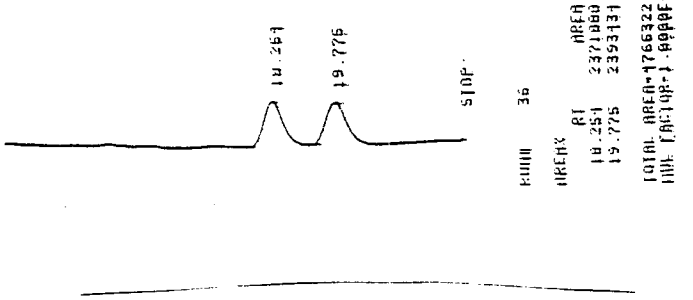
RUN# 155 JAN 5, 1901 20:44:58

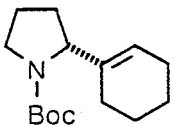
	RT	AREA	TYPE	WIDTH	AREA%
	10.775	706125	VV	.382	3.35251
	12.667	82325	BV	.226	.39130
	12.936	19555286	PV	.456	83.39535
	15.580	3025094	VV	.522	12.90061

TOTAL AREA=2.34196E+07
MUL FACTOR=1.00000E+00

Racemic

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
18.25 and 19.78 min

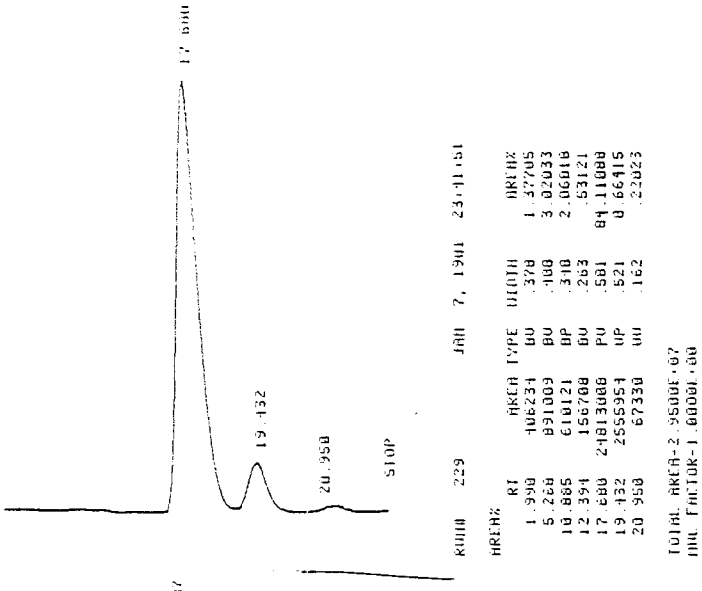




18

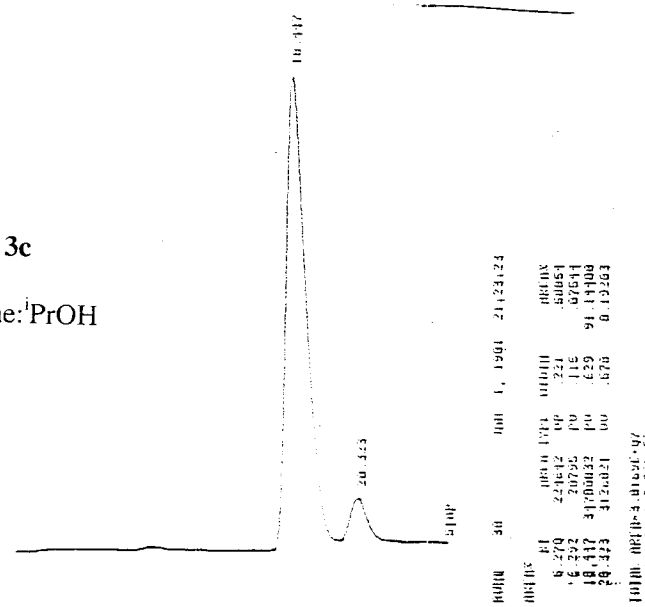
R_2CuLi from 3a

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
17.68 and 19.43 min
90.7:9.3(81.4%ee)



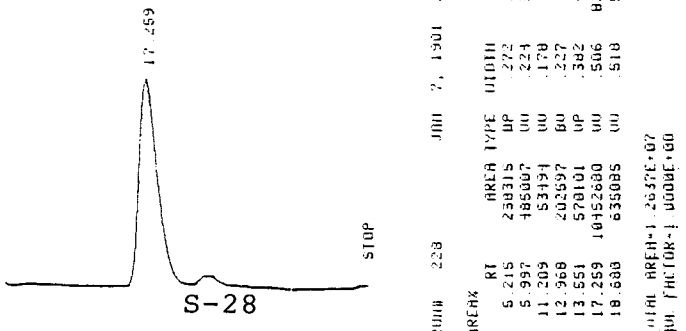
R_2CuLi from -ONf 3c

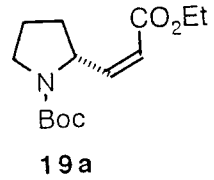
99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
18.45 and 20.32 min
92:8(84%ee)



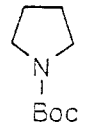
R_2CuLi from 3b

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
17.26 and 18.68 min
94.3:5.7(88.6%ee)





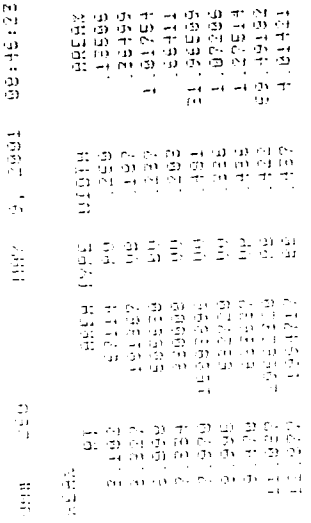
19a

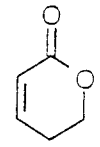


12

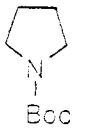
R_2CuLi

99.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
11.04 and 12.88 min
93.7:6.3 (87%ee)

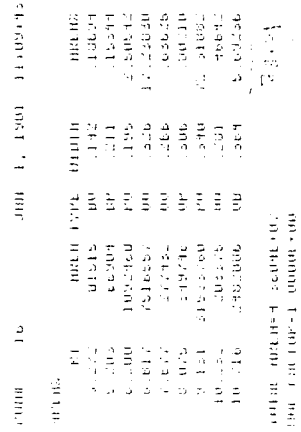


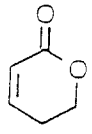
R_2CuLi with  for 0.5h

95.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
9.13 and 10.72 min
92.7:7.3 (85.4%ee)



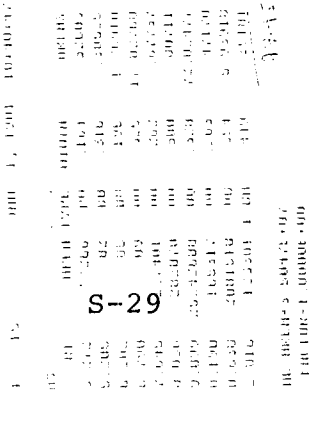
12

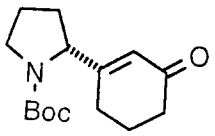


R_2CuLi with  for 1h

99.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
9.09 and 10.64 min
92.5:7.5 (85%)

12

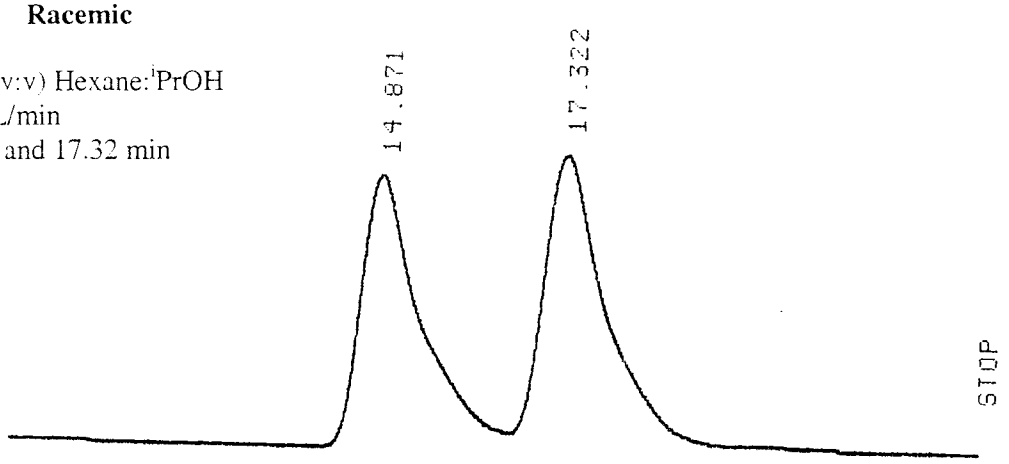




20

Racemic

98:2 (v:v) Hexane:^{i}PrOH
1.3mL/min
14.87 and 17.32 min



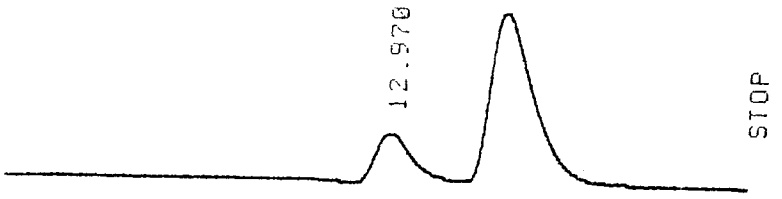
RUN# 26 DEC 20, 2000 15:39:18

AREA%

	RT	AREA	TYPE	WIDTH	AREA%
	14.871	6728378	BV	.903	46.94435
	17.322	7604288	VB	.945	53.05566

R_2CuLi

98:2 (v:v) Hexane:^{i}PrOH
1.3mL/min
12.97 and 14.49 min
80:20 (60%ee)

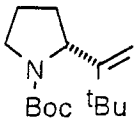


RUN# 66 FEB 5, 2001 17:19:54

AREA%

	RT	AREA	TYPE	WIDTH	AREA%
	2.400	238480	PV	.506	.70972
	2.940	454245	VV	.111	1.35183
	6.391	94400	BV	.318	.28093
	6.889	218594	VV	.332	.65053
	12.970	6609440	PV	.570	19.66966
	14.490	25987056	VB	.671	77.33734

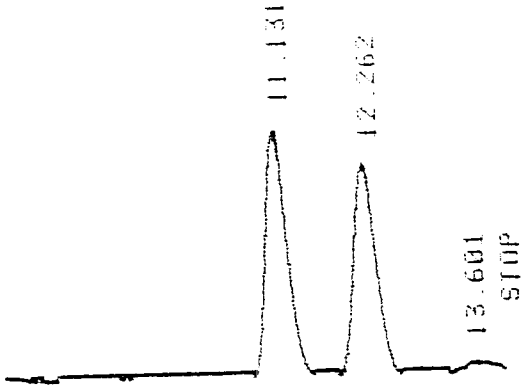
TOTAL AREA=3.3602E+07
MUL FACTOR=1.0000E+00



21

Racemic

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
11.13 and 12.26 min



JAN 7, 1901 20:23:49

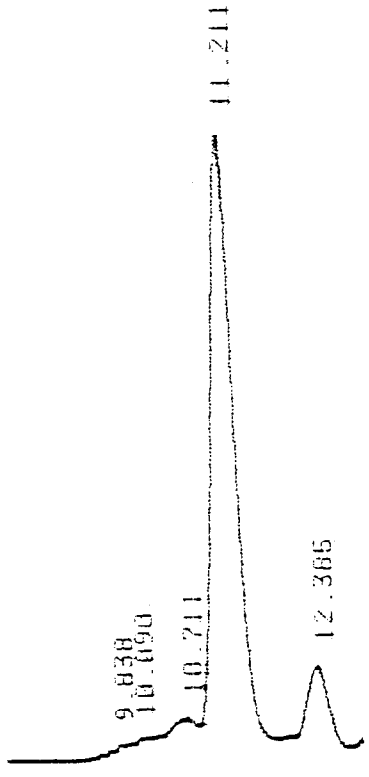
RUN# 223

AREA%

RT	AREA	TYPE	WIDTH	AREA%
5.965	901847	BV	.230	9.09283
11.131	4004878	VB	.302	43.93162
12.262	3796906	BV	.331	41.65114
12.732	259638	VP	.623	2.84811
13.601	152816	PO	.245	1.67632

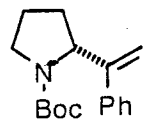
R_2CuLi

99.8:0.2 (v:v) Hexane:^{i}PrOH
0.6mL/min
11.21 and 12.39 min
89.5:10.5 (79%ee)



JAN 7, 1901 21:19:30

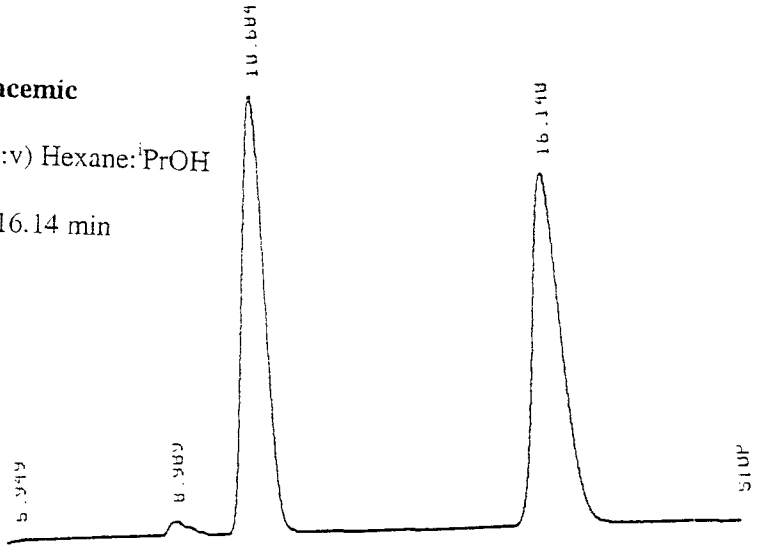
AREA	TYPE	WIDTH	AREA%
378734	BV	.231	2.656622
1943393	VB	.280	1.366836
11731	BV	.078	.082227
25554	VB	.196	.179908
305619	BV	.292	2.143444
10931615	VB	.365	76.666806
1279609	BV	.304	8.97444
216347	VB	.275	1.517353
221902	VP	.370	5.06300
192987	PO	.206	1.35290



22

Racemic

99.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
10.68 and 16.14 min



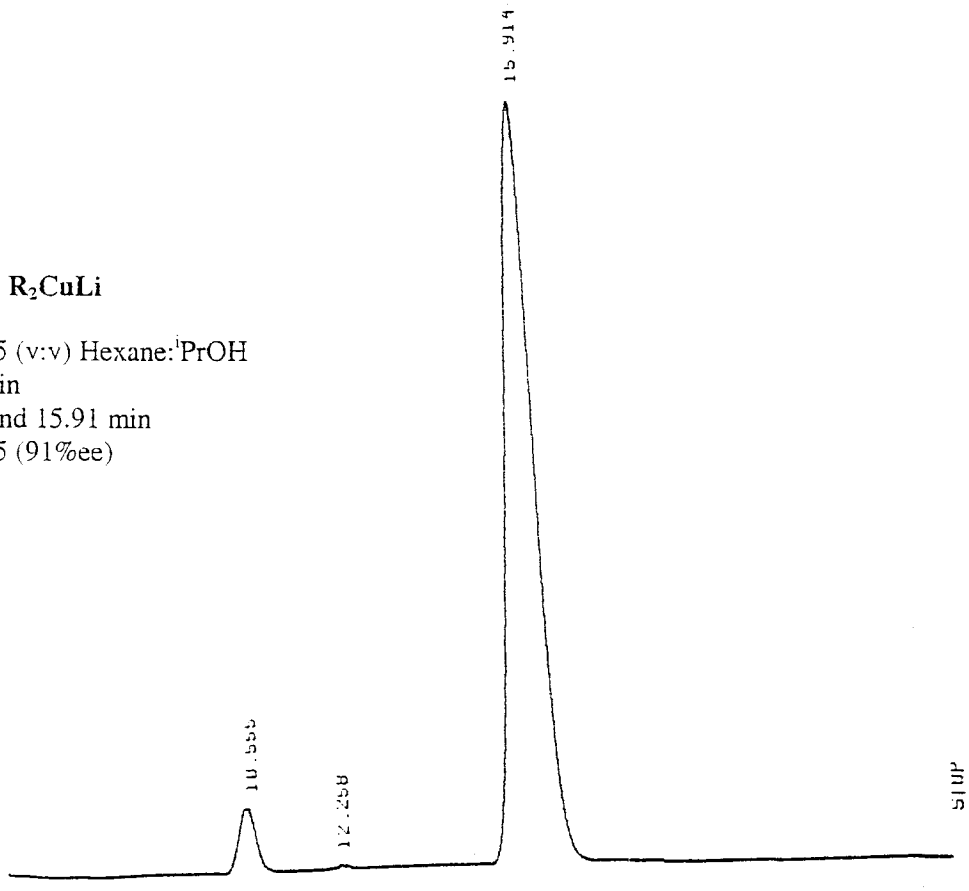
RUN# 28 JAN 1, 1901 08:41:56

HRLH%				
	RT	HRLH TYPE	WIDTH	HRLH%
	5.949	BV	.127	.09565
	8.989	VB	.111	.02698
	10.684	BV	.245	.10798
	16.140	VB	.215	.19582
	16.914	BV	.401	1.59919
	10.684	BV	.485	98.99982
	16.140	VB	.608	99.33597

TOTAL HRLH=6.80725E+07
MUL FACTOR=1.00000E+00

R_2CuLi

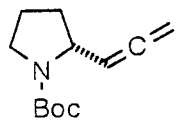
99.5:0.5 (v:v) Hexane:^{i}PrOH
1mL/min
10.56 and 15.91 min
95.5:4.5 (91%ee)



RUN# 29 JAN 1, 1901 07:06:23

HRLH%				
	RT	HRLH TYPE	WIDTH	HRLH%
	5.965	BV	.097	.02109
	9.488	VB	.285	.32688
	10.555	BV	.588	.90700
	12.258	VB	.584	4.41655
	15.914	BV	.549	.26064
	16.140	VB	.614	95.66882

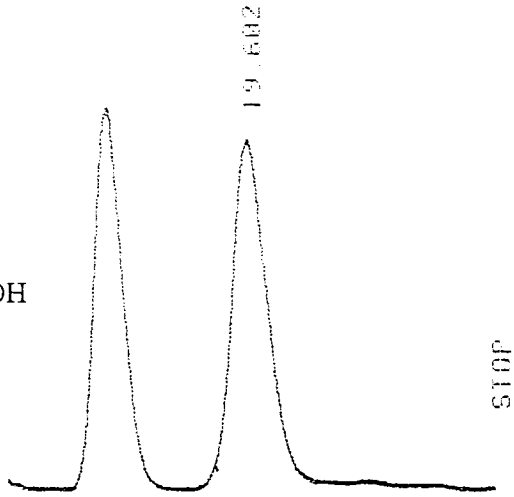
TOTAL HRLH=7.91205E+07
MUL FACTOR=1.00000E+00



23

Racemic

99.5:0.5 (v:v) Hexane:^{i}PrOH
0.5mL/min
17.74 and 19.60 min



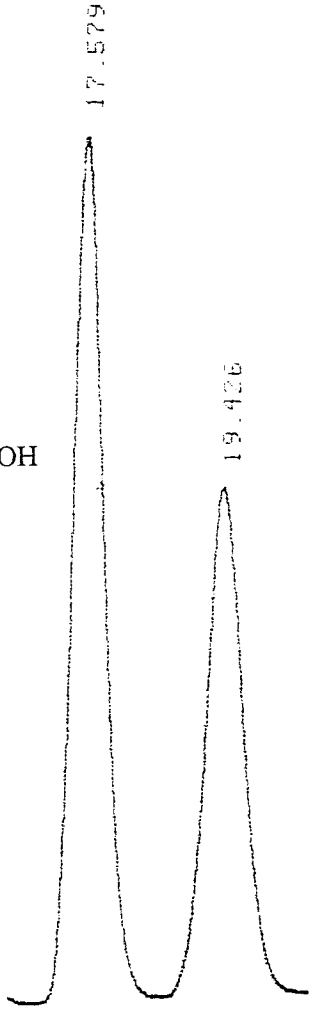
RUN# 243 JAN 8, 1901 11:56:54

AREA%

	RT	AREA	TYPE	WIDTH	AREA%
	6.196	216920	BV	.275	1.09100
	7.858	11252	VB	.065	.05660
	16.138	374107	BV	.407	1.88172
	17.744	9220506	VP	.442	46.41829
	19.602	10050400	VP	.528	50.55234

R_2CuLi from 8a

99.5:0.5 (v:v) Hexane:^{i}PrOH
0.5mL/min
17.58 and 19.43 min
58.4:41.6 (16.8%ee)

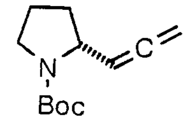


RUN# 242 JAN 8, 1901 10:44:04

AREA%

	RT	AREA	TYPE	WIDTH	AREA%
	5.839	35657	BV	.119	.07872
	6.145	901084	VV	.297	1.98926
	11.201	718068	BV	.314	1.58523
	12.155	758515	BP	.320	1.67452
	13.684	1618640	BP	.330	3.57336
	16.212	1044274	PV	.409	2.30537
	17.579	23008384	BV	.480	50.79395
	19.426	16399800	VV	.577	36.20467
	20.883	813079	VV	.382	1.79498

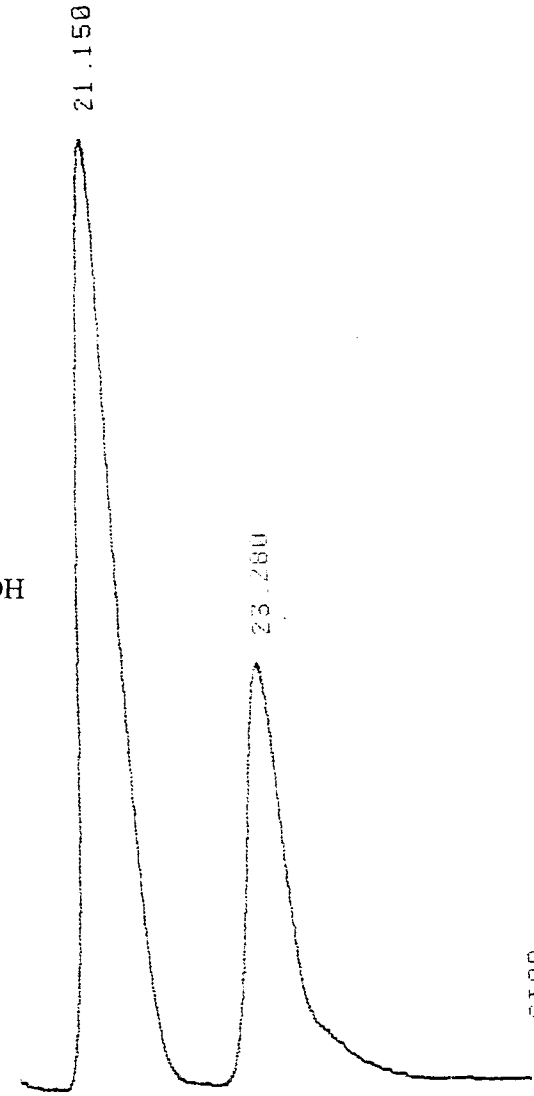
TOTAL AREA=4.5298E+07
MUL FACTOR=1.0000E+00



23

R_2CuLi from 8a

99.5:0.5 (v:v) Hexane:[i]PrOH
0.5mL/min
21.15 and 23.28 min
66.7:33.3 (33.4%ee)



RUN# 299 JAN 10, 1901 03:15:28

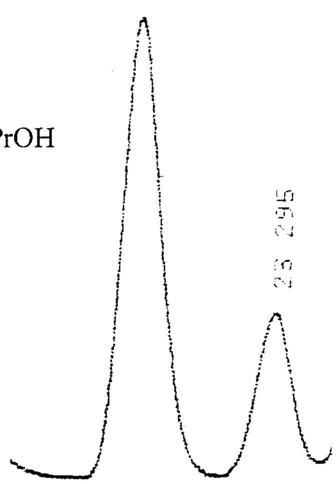
AREA%

RT	AREA	TYPE	WIDTH	AREA%
8.089	2674494	BV	.390	5.47509
9.192	370340	BV	.348	.75814
13.623	16845	BV	.065	.03448
13.897	271015	VV	.269	.55481
15.030	39360	BV	.115	.08058
19.567	1887536	BV	.441	3.86407
21.150	29035232	PV	.560	59.43949
23.280	14553576	VV	.633	29.79337

TOTAL AREA=4.8848E+07
MUL FACTOR=1.0000E+00

R_2CuLi from 8b

99.5:0.5 (v:v) Hexane:[i]PrOH
0.5mL/min
21.47 and 23.29 min
74.4:25.6 (48.6%ee)

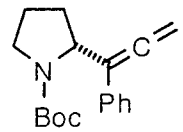


RUN# 296 JAN 10, 1901 01:15:13

AREA%

RT	AREA	TYPE	WIDTH	AREA%
8.070	464760	PV	.391	1.95868
9.078	180808	VV	.231	.76200
11.294	55755	VV	.173	.23497
13.708	27385	BV	.073	.11541
13.905	158126	VV	.252	.66640
19.341	26567	BV	.071	.11196
19.639	330457	VV	.281	1.39267
21.470	14915736	BV	.588	62.86072
23.295	5200640	BV	.572	21.91752
24.371	2368005	VP	.682	9.97969

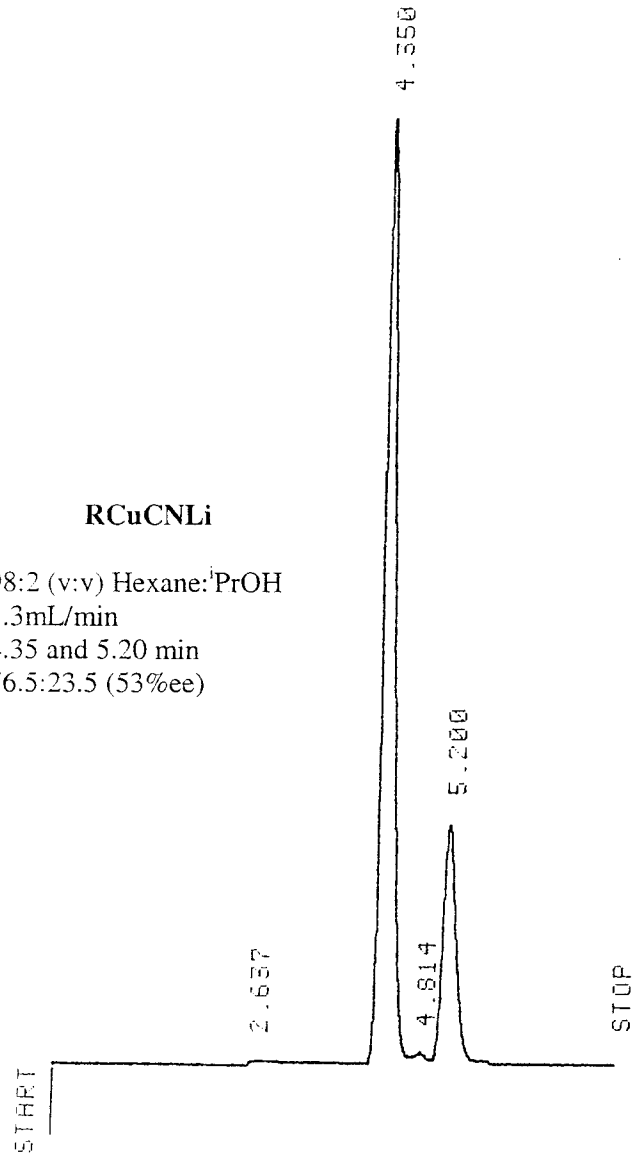
TOTAL AREA=2.3728E+07
MUL FACTOR=1.0000E+00



25

RCuCNLi

98:2 (v:v) Hexane:^{i}PrOH
1.3mL/min
4.35 and 5.20 min
76.5:23.5 (53%ee)

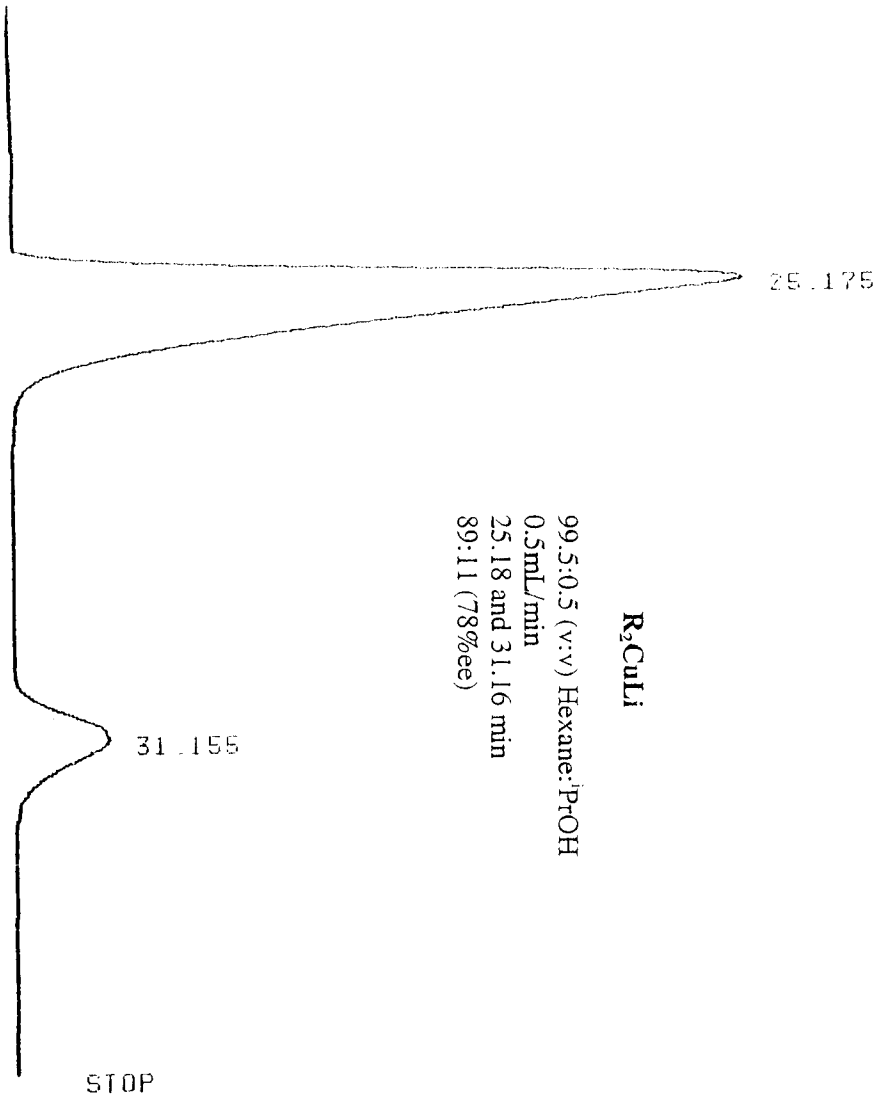


RUN# 137 FEB 27, 2001 05:49:46

AREA%

RT	AREA	TYPE	WIDTH	AREA%
2.637	67321	BV	.109	.14921
4.350	34288960	VV	.168	75.99965
4.814	398808	VV	.180	.88394
5.200	10362176	VB	.199	22.96722

TOTAL AREA=4.5117E+07
MUL FACTOR=1.0000E+00



R_2CuLi

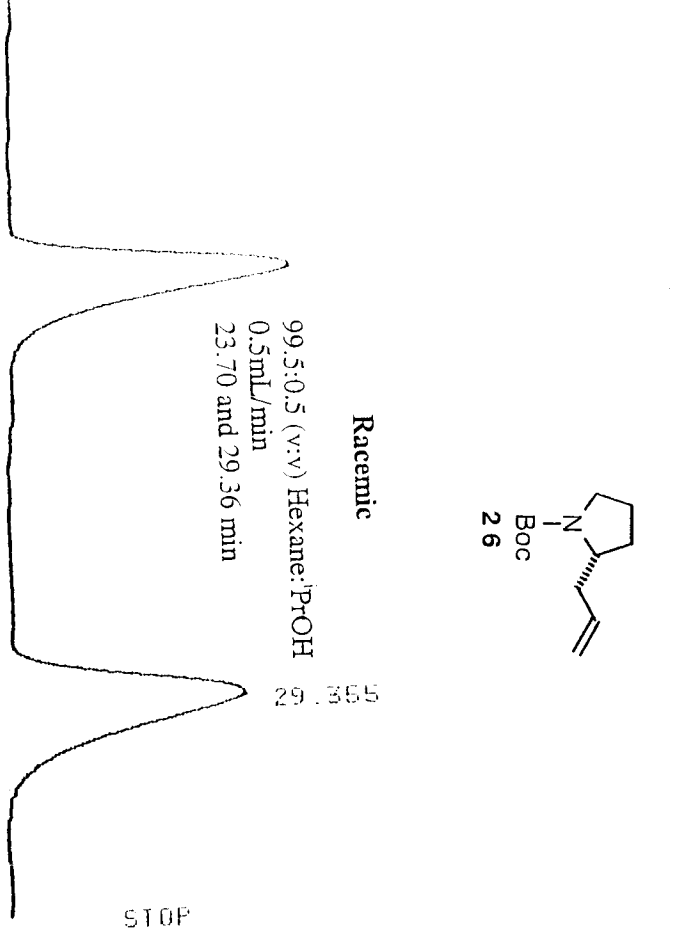
99.5:0.5 (v:v) Hexane:^{i}PrOH
0.5mL/min
25.18 and 31.16 min
89:11 (78%ee)

RUN# 90 JAN 3, 1901 23:56:33

AREA%

RT	AREA	TYPE	WIDTH	AREA%
1.090	7808	BV	.073	.02184
6.802	129963	BV	.278	.36349
8.400	139904	BP	.243	.39129
25.175	31616880	VV	.781	88.42848
31.155	3859646	VV	.750	10.79494

TOTAL AREA=3.5754E+07
MUL FACTOR=1.0000E+00



Racemic

99.5:0.5 (v:v) Hexane:^{i}PrOH
0.5mL/min
23.70 and 29.36 min

26

RUN# 88 JAN 3, 1901 21:12:49

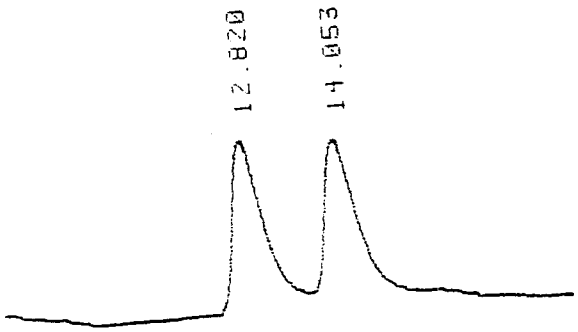
AREA%

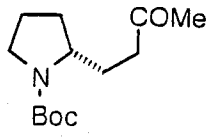
RT	AREA	TYPE	WIDTH	AREA%
.602	3380008	BV	.344	12.44897
1.619	2566558	VB	.311	9.45293
6.615	274537	BV	.297	1.01115
8.297	169227	BP	.250	.62328
23.704	10374888	BP	.673	38.21194
29.355	10385696	BV	.800	38.25174

TOTAL AREA=2.7151E+07
MUL FACTOR=1.0000E+00

Racemic

97:3 (v:v) Hexane:^{i}PrOH
0.5mL/min
12.82 and 14.05 min

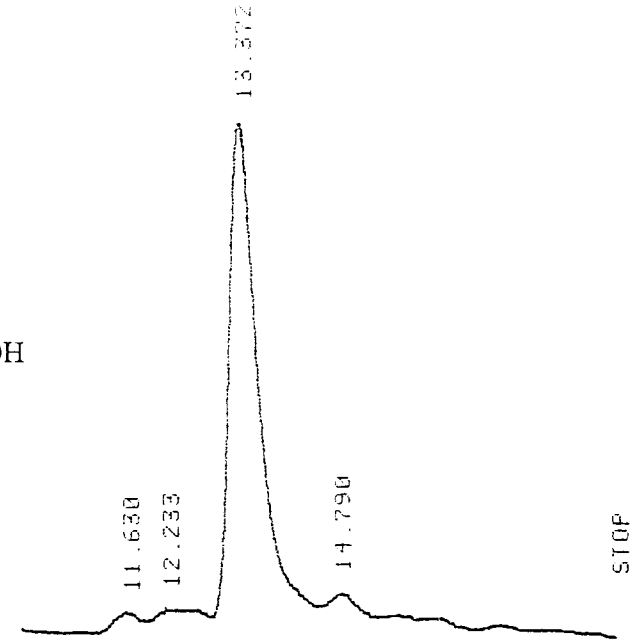




28a

R_2CuLi

97:3 (v:v) Hexane:^{i}PrOH
0.5mL/min
13.37 and 14.79 min
97:3 (94%ee)



RUN# 203 JAN 7, 1901 08:38:45

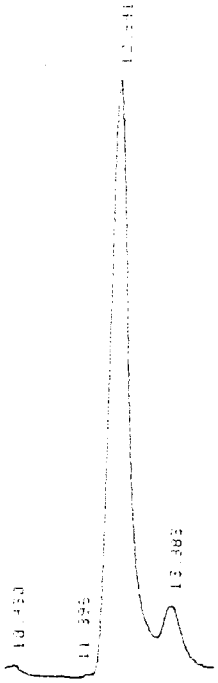
AREA%

	RT	AREA	TYPE	WIDTH	AREA%
1	5.760	3598656	BV	.373	12.79540
2	6.575	7727923	VV	.279	27.47743
3	8.469	333261	BV	.247	1.18494
4	8.982	1569762	VP	.486	5.58145
5	11.630	292164	PP	.332	1.03882
6	12.233	78855	PV	.216	.28038
7	13.372	14099608	PV	.472	50.13259
8	14.790	424396	BP	.376	1.50898

TOTAL AREA=2.8125E+07
MUL FACTOR=1.0000E+00

R_2CuLi

97:3 (v:v) Hexane:^{i}PrOH
0.5mL/min
12.64 and 13.88 min
90:10 (80%ee)



RUN# 362 JAN 15, 1901 21:02:45

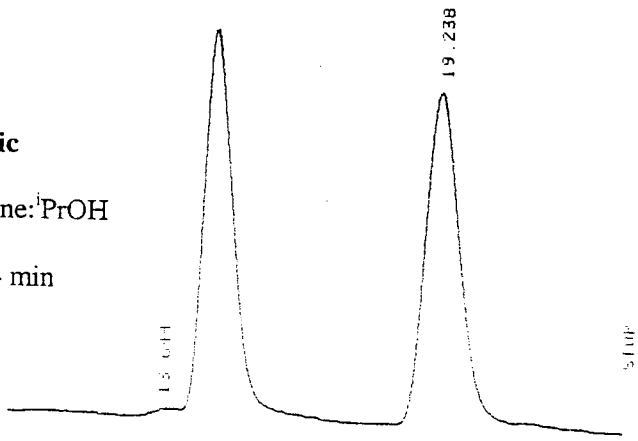
AREA%

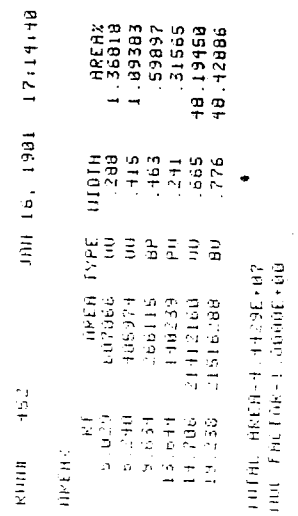
	RT	AREA	TYPE	WIDTH	AREA%
1	5.710	267993	BV	.115	.66527
2	6.035	4102408	VP	.353	10.18009
3	8.357	38605	PV	.157	.09582
4	7.614	3156575	BV	.374	7.83916
5	9.102	1115969	BV	.336	2.77114
6	9.936	317805	VV	.328	.78925
7	10.430	325159	VP	.360	.80751
8	11.895	1422	BB	.018	.00353
9	12.092	23564	BV	.090	.05852
10	12.641	26627568	VV	.485	66.12600
11	13.883	3047734	VV	.522	7.56887
12	15.150	590520	BP	.418	1.46652
13	16.851	651534	VV	.462	1.61805

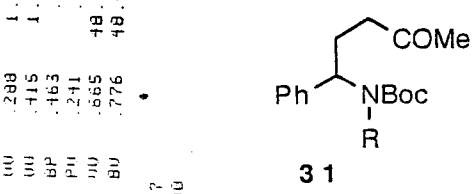
TOTAL AREA=4.0267E+07
MUL FACTOR=1.0000E+00

Racemic

98:2 (v:v) Hexane:^{i}PrOH
0.6mL/min
14.71 and 19.24 min



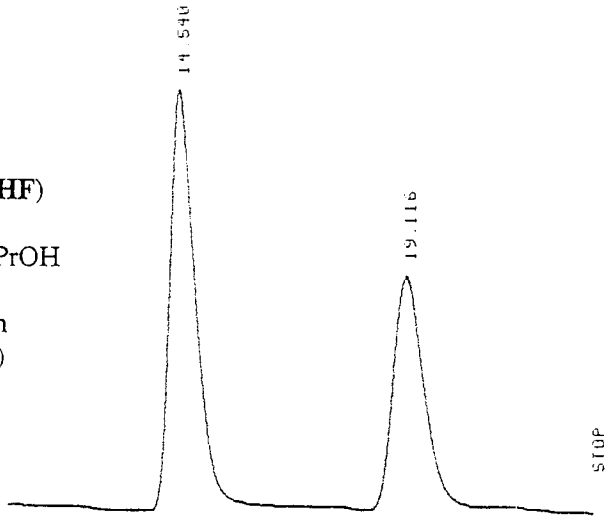


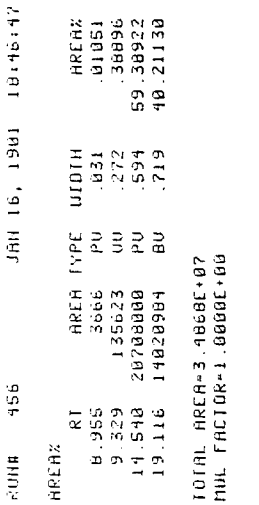


31

R_2CuLi (THF)

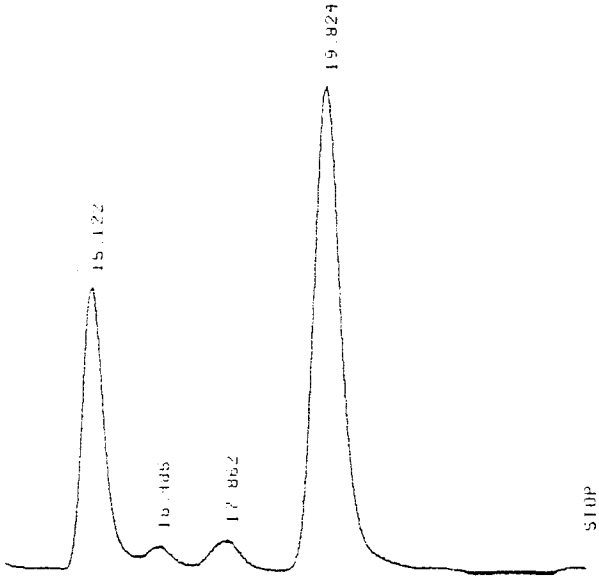
98:2 (v:v) Hexane:^{i}PrOH
0.6mL/min
14.54 and 19.12 min
59.6:40.4 (19.2%ee)

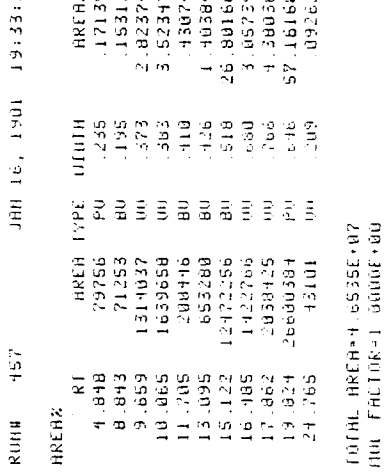


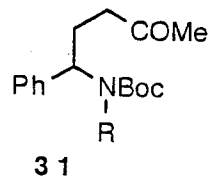


R_2CuLi (Et_2O)

97:3 (v:v) Hexane:^{i}PrOH
0.5mL/min
15.12 and 19.82 min
68.1:31.9 (36.2%ee)



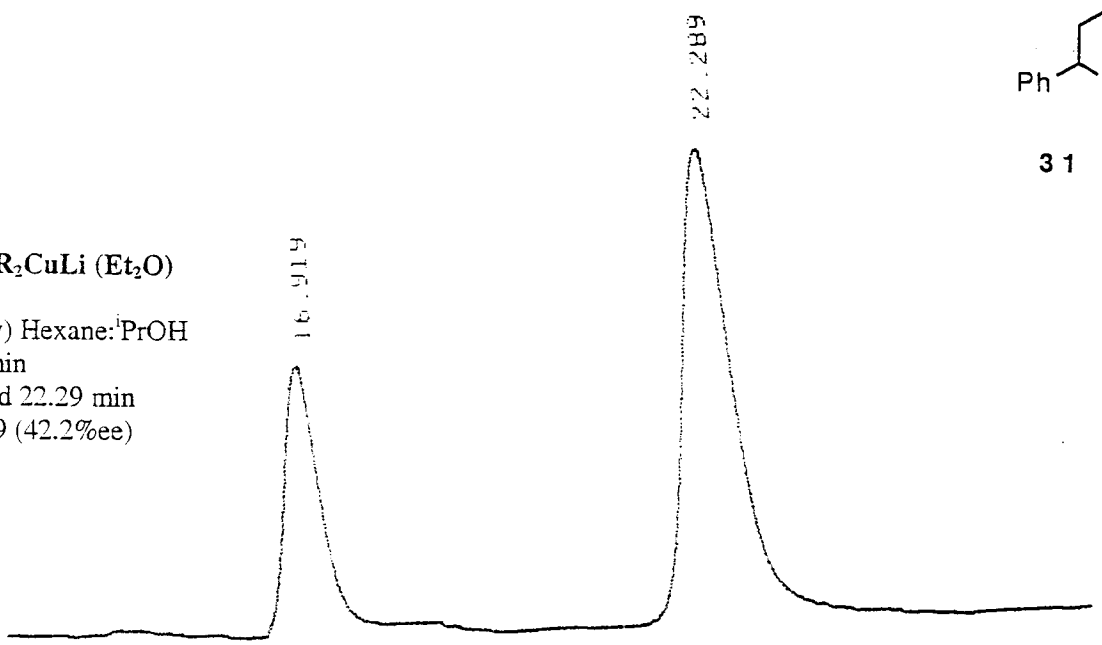




31

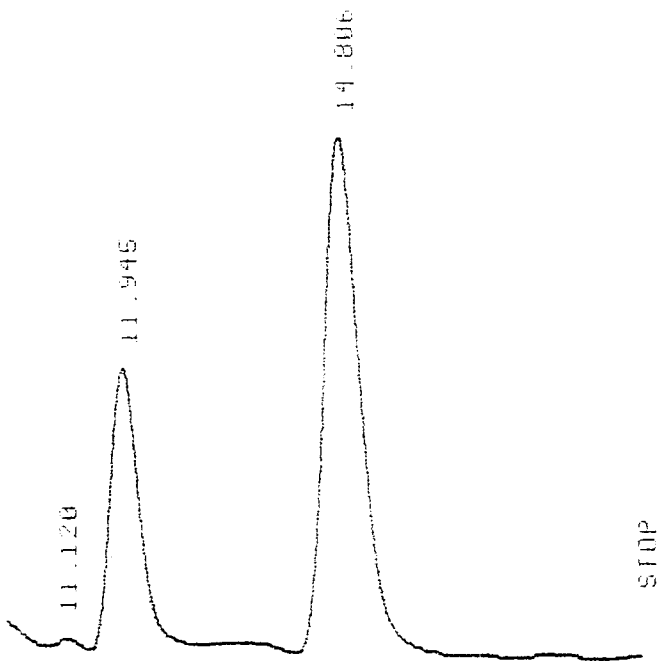
R_2CuLi (Et_2O)

98:2 (v:v) Hexane:^{i}PrOH
0.6mL/min
16.92 and 22.29 min
71.1:28.9 (42.2%ee)



R_2CuLi (Et_2O)

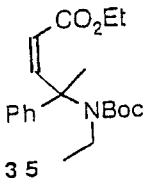
97:3 (v:v) Hexane:^{i}PrOH
0.6mL/min
11.95 and 14.81 min
69.5:30.5 (39%ee)



RUN# 460 JAN 16, 1901 22:11:54

AREA%	RT	AREA	TYPE	WIDTH	AREA%
	4.745	39935	PU	.124	.10950
	5.195	11149360	VV	.577	30.57240
	6.210	3045062	VP	.479	8.34980
	9.369	5566	PU	.036	.01526
	9.749	5112	VV	.036	.01402
	9.870	20321	VV	.095	.05572
	11.120	147877	VP	.[illegible]	.[illegible]
	11.945	6728090	PU	.432	18.44894
	14.806	15327384	VV	.540	42.02886

NB: Retention time is not the same because different solvent ratio's used



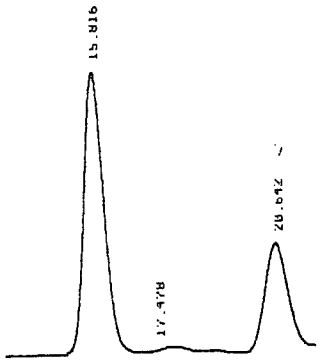
35

from RCuCNLi

(-)-sparteine (THF)

38 % ee

99.5:0.5 (v:v) Hexane:^{i}PrOH
0.6mL/min

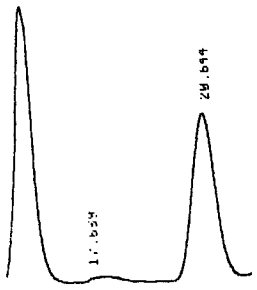


AREA%

RT	AREA	TYPE	WIDTH	AREA%
5.073	117516	BV	.228	.66450
15.816	8374016	PV	.271	47.35096
17.478	10946	BV	.088	.06189
20.642	7849130	BV	.690	44.38296
22.359	1333394	PV	.292	7.53969

(-)-sparteine (Et_2O)

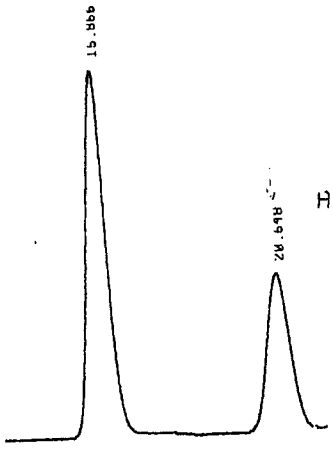
16 % ee



15.884	17522496	VV	.570	50.52717
17.639	6862	PV	.052	.01979
20.644	12904800	BV	.706	37.21178
22.518	3272214	VV	.732	9.43563

TMEDA (Et_2O)

32 % ee

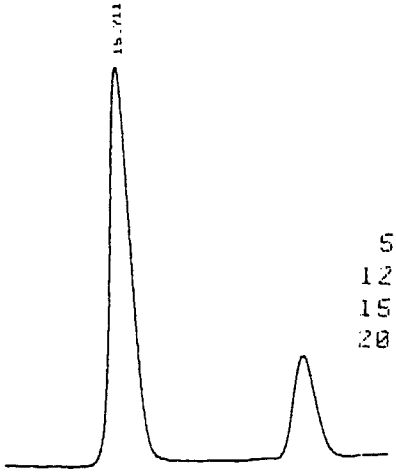


AREA%

RT	AREA	TYPE	WIDTH	AREA%
9.065	57141	PV	.194	.14759
10.569	13793	PV	.106	.03563
15.866	23490672	BV	.585	60.67374
20.648	12410224	VV	.711	32.05419

TMEDA (THF)

55 % ee



RT	AREA	TYPE	WIDTH	AREA%
5.005	20494	PB	.118	.05736
12.148	61188	VV	.195	.17126
15.711	27705472	PV	.636	77.54688
20.541	7940237	VV	.712	22.22451