

SUPPORTING INFORMATION

Synthesis of 2-Alkylidene Cyclopentanones via Palladium-Catalyzed Cross-Coupling of 1-(1-Alkynyl)cyclobutanols and Aryl or Vinylic Halides

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Synthesis of the 1-(1-Alkynyl)cyclobutanols

Representative Synthesis of a 1-(1-Alkynyl)cyclobutanol: 1-(Phenylethynyl)cyclobutanol (1). A three neck flask with thermometer inlet was charged with diisopropylamine (1.212 g, 12 mmol) in THF (5 ml). The flask was cooled to -78°C and *n*-BuLi (5.7 ml, 12 mmol) was added dropwise under an argon atmosphere. The reaction mixture was allowed to warm to room temperature for 20 min and then cooled to -78°C and phenylacetylene (1.224 g, 12 mmol) in THF (2 ml) was added. The flask was allowed to warm to 0°C for 20 min and then stirred for 1 h. The flask was again cooled to -78°C and cyclobutanone (0.700 g, 10 mmol) in THF (4 ml) was added. The flask was allowed to warm to room temperature for 30 min and further stirred for 2 h, quenched with water and extracted with ether (3 x 25 ml) and the combined extracts were washed with saturated aqueous NH_4Cl , water, and brine, and then dried (Na_2SO_4) and filtered. The solvent was removed under reduced pressure and the resulting oil was purified by flash column chromatography using 60:40 hexanes/diethyl ether to afford 1.566 g (91%) of the desired compound as a white solid: mp 42°C ; ^1H NMR (CDCl_3) δ 1.82-1.93 (m, 2H), 2.10 (br s, 1H), 2.23-2.40 (m, 2H), 2.50-2.58 (m, 2H), 7.26-7.34 (m, 3H), 7.42-7.46 (m, 2H); ^{13}C NMR

(CDCl₃) δ 12.3, 38.6, 68.2, 83.4, 92.6, 122.7, 128.2, 131.7 (one carbon missing as a result of overlap).

1-(1-Propynyl)cyclobutanol (7). A three neck flask with thermometer inlet was charged with anhydrous cerium(III) chloride (2.46 g, 10 mmol) in THF (20 ml). The flask was cooled to -78 °C and 1-propynylmagnesium bromide (20 ml, 10 mmol) was added dropwise under an argon atmosphere. After the mixture had been stirred for 1 h at -78 °C, cyclobutanone (0.56 g, 8 mmol) in THF (6 ml) was added. The resulting reaction mixture was allowed to warm to room temperature for 30 min and further stirred for 2 h, quenched with water and extracted with ether (3 x 25 ml) and the combined extracts were washed with saturated aqueous NH₄Cl, water, and brine, and then dried (Na₂SO₄) and filtered. The solvent was removed under reduced pressure and the resulting oil was purified by flash column chromatography using 70:30 hexanes/diethyl ether to afford 0.6457 g (73%) of the desired compound as a colorless oil: ¹H NMR (CDCl₃) δ 1.71-1.82 (m, 2H), 1.86 (s, 3H), 2.15-2.26 (m, 3H), 2.32-2.41 (m, 2H); ¹³C NMR (CDCl₃) δ 3.6, 12.7, 38.6, 68.0, 79.4, 83.0.

3-Phenyl-1-(phenylethynyl)cyclobutanol (11). This cyclobutanol was prepared by the same method used to prepare cyclobutanol **1**, but 3-phenylcyclobutanone (1.46 g, 10 mmol) was employed. Removal of the solvent afforded 2.1 g (85%) of the cyclobutanol **11** as a white solid: mp 78 °C; ¹H NMR (CDCl₃) δ 2.51-2.59 (m, 2H), 2.88 (br s, 1H), 3.00-3.07 (m, 2H), 3.39-3.51 (m, 1H), 7.26-7.39 (m, 8H), 7.53-7.56 (m, 2H); ¹³C NMR (CDCl₃) δ 30.5, 46.3, 64.4, 83.6, 92.5, 122.6, 126.2, 126.7, 128.3, 128.4, 128.4, 131.7, 144.0.

6-(Phenylethynyl)bicyclo[3.2.0]hept-2-en-6-ol (13). This cyclobutanol was prepared by the same method used to prepare cyclobutanol **1**, but bicyclo[3.2.0]hept-2-en-6-one (1.0 g, 10 mmol) was employed. Removal of the solvent afforded 1.89 g (90%) of the cyclobutanol **13** as a light yellow solid: mp 51 °C; ¹H NMR (CDCl₃) δ 2.06-2.12 (dddd, J = 1.2, 0.9, 1.2, 0.9 Hz, 1H), 2.31-2.53 (m, 2H), 2.81-2.99 (m, 2H), 3.20 (s, 1H), 3.42 (t, J = 7.5 Hz, 1H), 5.83-5.89

(m, 2H), 7.29-7.33 (m, 3H), 7.42-7.48 (m, 2H); ^{13}C NMR (CDCl_3) δ 32.5, 39.3, 45.2, 48.8, 67.6, 83.6, 93.4, 122.7, 128.2, 131.5, 132.5, 134.4.

1-Methyl-7-(phenylethynyl)bicyclo[4.2.0]octan-7-ol (15). This cyclobutanol was prepared by the same method used to prepare cyclobutanol **1**, but 1-methylbicyclo[4.2.0]octan-7-one (1.38 g, 10 mmol) was employed. Removal of the solvent afforded 2.15 g (90%) of the cyclobutanol **15** as a colorless liquid: ^1H NMR (CDCl_3) δ 1.24-1.67 (m, 9H), 1.74-1.86 (m, 2H), 2.03 (br s, 1H), 2.18-2.31 (m, 2H), 2.38 (t, $J = 6.0$ Hz, 1H), 7.28-7.31 (m, 3H), 7.40-7.43 (m, 2H); ^{13}C NMR (CDCl_3) δ 20.8, 21.2, 22.3, 28.8, 31.4, 35.7, 48.9, 50.4, 67.7, 83.3, 94.2, 123.2, 128.2, 128.3, 131.6.

General Procedure for the Palladium-Catalyzed Cross-Coupling of 1-(1-Alkynyl)cyclobutanols and Organic Halides: DMF (5 mL), $\text{Pd}(\text{OAc})_2$ (11 mg, 0.05 mmol), PPh_3 (26 mg, 0.1 mmol), the organic iodide (1.0 mmol), $i\text{-Pr}_2\text{NEt}$ (130 mg, 1.0 mmol), $n\text{-Bu}_4\text{NCl}$ (277 mg, 1.0 mmol) and the 1-(1-alkynyl)cyclobutanol (0.5 mmol) were placed in a 4 dram vial. The vial was flushed with argon and heated in an oil bath at 80 °C for the indicated period of time. The reaction was monitored by TLC to establish completion. The reaction mixture was cooled, diluted with 30 ml of ether, washed with 40 ml of saturated NaCl, dried (Na_2SO_4), and filtered. The solvent was evaporated under reduced pressure and the product was isolated by chromatography on a silica gel column.

Compounds Prepared

2-(Diphenylmethylene)cyclopentanone (2). The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 87 mg (70%) of the indicated compound as a yellow solid: mp 105 °C; ^1H NMR (CDCl_3) δ 1.93 (q, $J = 7.5$ Hz, 2H), 2.38 (t, $J = 7.8$ Hz, 2H), 2.82 (t, $J = 7.0$ Hz, 2H), 7.10-7.14 (m, 2H), 7.17-7.20 (m, 2H), 7.30-7.35 (m, 6H); ^{13}C NMR (CDCl_3) δ 20.5, 32.9, 39.8, 127.8, 127.9, 128.3, 129.4, 129.6, 134.3, 140.1, 141.8, 148.2, 206.5 (one carbon

missing as a result of overlap); IR (CHCl₃, cm⁻¹) 1706; MS *m/z* (rel intensity) 248 (50, M⁺), 247 (100), 191 (30). Anal. Calcd for C₁₈H₁₆O: C, 87.06; H, 6.49.

Found: C, 86.90; H, 6.80.

***E*-2-[(2-Methoxyphenyl)phenylmethylene]cyclopentanone (3).** The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 103 mg (74%) of the indicated compound as a yellow solid: mp 110 °C; ¹H NMR (CDCl₃) δ 1.85-1.95 (m, 2H), 2.40 (t, *J* = 7.8 Hz, 2H), 2.54 (t, *J* = 7.2 Hz, 2H), 3.74 (s, 3H), 6.91-6.96 (m, 2H), 7.04-7.07 (m, 1H), 7.21-7.33 (m, 6H); ¹³C NMR (CDCl₃) δ 19.6, 31.8, 40.3, 55.4, 111.3, 120.4, 127.3, 127.5, 128.9, 129.2, 129.8, 131.2, 135.0, 139.6, 145.2, 156.0, 205.9; IR (CHCl₃, cm⁻¹) 1712; MS *m/z* (rel intensity) 278 (50, M⁺), 247 (100). Anal. Calcd for C₁₉H₁₈O₂: C, 81.99; H, 6.52. Found: C, 81.47; H, 6.75.

***E*-2-[(2-Nitrophenyl)phenylmethylene]cyclopentanone (4).** The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 103 mg (70%) of the indicated compound as a yellow viscous oil: ¹H NMR (CDCl₃) δ 1.88-1.97 (m, 2H), 2.49 (tt, *J* = 6.9, 6.7 Hz, 4H), 7.24-7.29 (m, 5H), 7.39 (dt, *J* = 1.5, 1.2 Hz, 1H), 7.49 (dq, *J* = 1.5, 0.9, 1.5 Hz, 1H), 7.66 (ddd, *J* = 1.2, 1.2, 1.5 Hz, 1H), 8.00 (tt, *J* = 0.7, 0.6 Hz, 1H); ¹³C NMR (CDCl₃) δ 19.5, 31.8, 40.1, 124.8, 127.5, 128.4, 128.8, 129.5, 130.7, 133.4, 134.6, 136.5, 136.9, 144.4, 147.5, 205.0; IR (CHCl₃, cm⁻¹) 1716; HRMS calcd for C₁₈H₁₄NO₃: 292.09737. Found: 292.09678.

***E*-2-[(*2E*)-1-Phenylhept-2-enylidene]cyclopentanone (5).** The reaction mixture was chromatographed using 70:30 hexanes/ether to afford 50 mg (40%) of the indicated compound as a yellow oil: ¹H NMR (CDCl₃) δ 0.88 (t, *J* = 6.9 Hz, 3H), 1.27-1.37 (m, 4H), 1.95-2.03 (m, 2H), 2.17 (q, *J* = 6.6 Hz, 2H), 2.29 (t, *J* = 7.8 Hz, 2H), 2.88 (t, *J* = 7.2 Hz, 2H), 5.55-5.65 (m, 1H), 6.47 (d, *J* = 15.3 Hz, 1H), 7.02-7.05 (m, 2H), 7.31-7.39 (m, 3H); ¹³C NMR (CDCl₃) δ 13.8, 19.4, 22.2, 29.2, 30.9, 33.3, 40.2, 126.9, 127.6, 128.6, 130.8, 131.1, 138.0, 144.9, 206.0; IR (CHCl₃, cm⁻¹) 1708; HRMS calcd for C₁₈H₂₂O: 254.16707. Found: 254.16701.

Z-[Phenyl(4-phenylcyclohex-1-enyl)methylene]cyclopentanone (6a).

The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 60 mg (36%) of the indicated compound as a yellow viscous oil: ^1H NMR (CDCl_3) δ 1.83-2.13 (m, 5H), 2.25-2.48 (m, 5H), 2.71 (t, $J = 6.9$ Hz, 2H), 2.90-3.00 (m, 1H), 5.61-5.62 (m, 1H), 7.19-7.41 (m, 10H); ^{13}C NMR (CDCl_3) δ 20.7, 29.1, 30.0, 32.2, 33.8, 39.5, 39.7, 125.7, 125.9, 126.9, 128.0, 128.3, 128.7, 133.3, 138.1, 140.4, 145.0, 147.2, 150.7, 207.2. This compound was too unstable for further characterization.

E-[Phenyl(4-phenylcyclohex-1-enyl)methylene]cyclopentanone (6b).

The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 57.4 mg (35%) of the indicated compound as a yellow viscous oil: ^1H NMR (CDCl_3) δ 1.75-1.85 (m, 1H), 1.93-2.11 (m, 5H), 2.33-2.43 (m, 3H), 2.50-2.56 (m, 1H), 2.79-2.90 (m, 3H), 5.95 (s, 1H), 7.19-7.41 (m, 10H); ^{13}C NMR (CDCl_3) δ 20.4, 27.9, 29.8, 32.3, 33.5, 39.5, 39.9, 126.1, 126.7, 127.5, 127.7, 128.3, 128.4, 128.6, 131.9, 138.7, 139.0, 146.4, 150.6, 206.5. This compound was too unstable for further characterization.

E-2-(1-Phenylethylidene)cyclopentanone (8). The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 56 mg (60%) of the indicated compound as a yellow oil: ^1H NMR (CDCl_3) δ 1.74-1.83 (m, 2H), 2.37 (t, $J = 7.5$ Hz, 2H), 2.52-2.60 (m, 5H), 7.22-7.39 (m, 5H); ^{13}C NMR (CDCl_3) δ 19.8, 20.3, 31.7, 40.5, 127.2, 127.8, 128.1, 132.6, 143.5, 147.4, 208.8; IR (CHCl_3 , cm^{-1}) 1703; HRMS calcd for $\text{C}_{13}\text{H}_{14}\text{O}$: 186.10447. Found: 186.10391.

E-2-[(2-Methoxyphenyl)ethylidene]cyclopentanone (9). The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 63 mg (63%) of the indicated compound as a yellow oil: ^1H NMR (CDCl_3) δ 1.73-1.83 (m, 2H), 2.34-2.45 (m, 7H), 3.79 (s, 3H), 6.89-7.04 (m, 3H), 7.24-7.29 (m, 1H); ^{13}C NMR (CDCl_3) δ 19.8, 19.9, 30.8, 40.7, 55.4, 111.0, 120.5, 128.1, 128.8, 132.4, 133.3, 145.8, 155.4, 208.7; IR (CHCl_3 , cm^{-1}) 1701; HRMS calcd for $\text{C}_{14}\text{H}_{16}\text{O}_2$: 216.11503. Found: 216.11451.

***E*-2-[(2*E*)-1-Methylhept-2-enylidene]cyclopentanone (10).** The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 34 mg (35%) of the indicated compound as a yellow oil: $^1\text{H NMR}$ (CDCl_3) δ 0.91 (t, $J = 4.5$ Hz, 3H), 1.29-1.45 (m, 4H), 1.85-1.92 (m, 2H), 2.18-2.36 (m, 7H), 2.72 (t, $J = 6.3$ Hz, 2H), 6.21-6.25 (m, 2H); $^{13}\text{C NMR}$ (CDCl_3) δ 13.1, 13.9, 19.4, 22.3, 29.3, 31.3, 33.4, 40.8, 131.0, 131.1, 139.9, 142.7, 208.9; IR (CHCl_3 , cm^{-1}) 1692, 1585; HRMS calcd for $\text{C}_{13}\text{H}_{20}\text{O}$: 192.15142. Found: 192.15127.

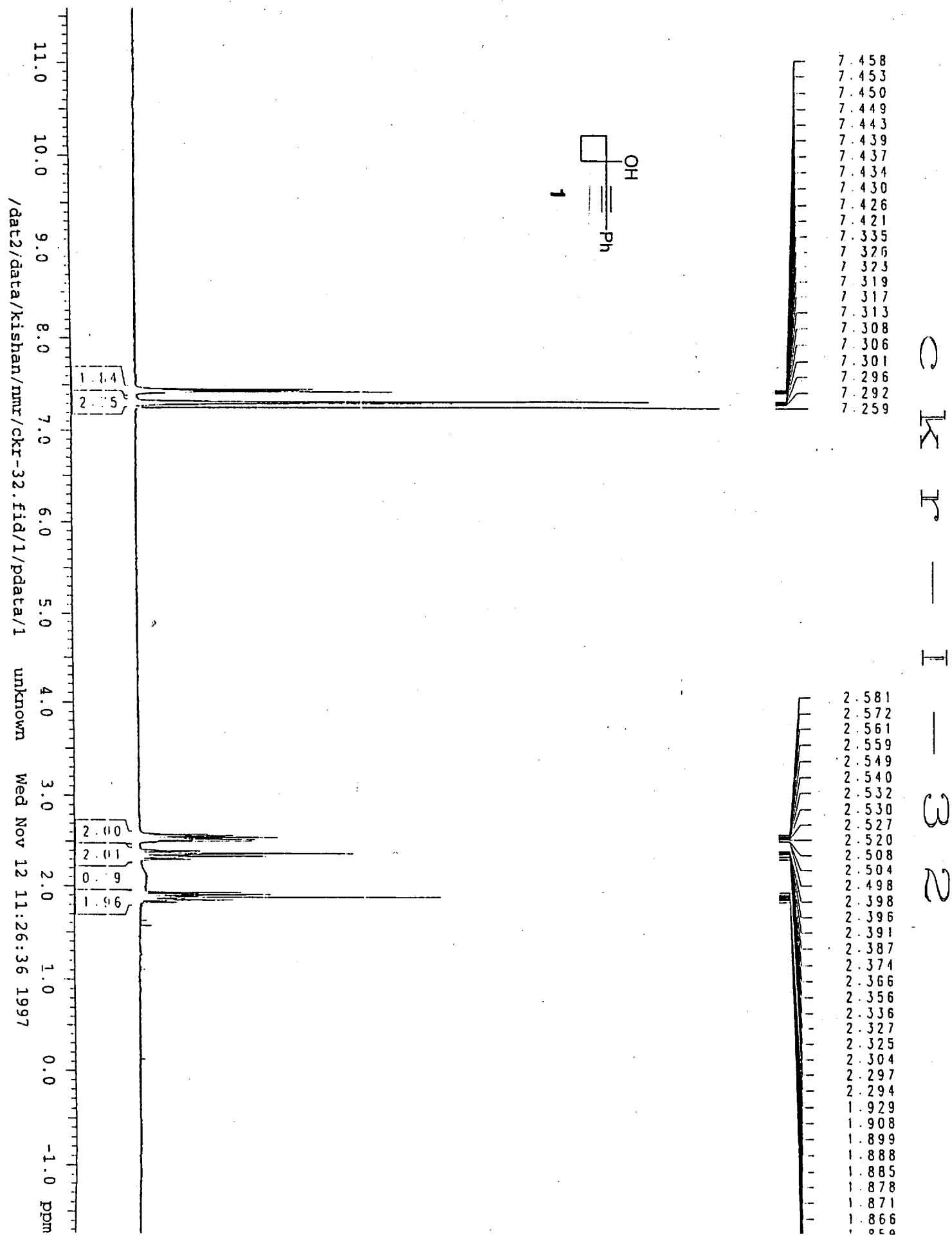
***E*-Ethyl 4-[(2-oxo-4-phenylcyclopentylidene)phenylmethyl]benzoate (12).** The reaction mixture was chromatographed using 80:20 hexanes/ether to afford 126 mg (64%) of the indicated compound as a yellow oil: $^1\text{H NMR}$ (CDCl_3) δ 1.40 (t, $J = 7.0$ Hz, 3H), 2.65 (q, $J = 10.8$ Hz, 1H), 2.83-3.02 (m, 2H), 3.19 (q, $J = 6.9$ Hz, 1H), 3.40-3.52 (m, 1H), 4.41 (q, $J = 7.0$ Hz, 2H), 7.17-7.39 (m, 12H), 8.05 (d, $J = 8.4$ Hz, 2H); $^{13}\text{C NMR}$ (CDCl_3) δ 14.2, 39.2, 40.9, 46.8, 61.0, 126.6, 126.7, 127.9, 128.2, 128.6, 129.2, 129.3, 129.4, 130.2, 134.8, 139.1, 142.8, 145.9, 147.6, 165.9, 204.3 (two carbons missing as a result of overlap); IR (CHCl_3 , cm^{-1}) 1710; HRMS calcd for $\text{C}_{27}\text{H}_{24}\text{O}_3$: 396.1726. Found: 396.1720.

***E*-2-[(2-Methoxyphenyl)phenylmethylene]bicyclo[3.3.0]oct-6-en-3-one (14).** The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 82 mg (52%) of the indicated compound as a yellow oil: $^1\text{H NMR}$ (CDCl_3) δ 2.23-2.31 (m, 3H), 2.69 (q, $J = 9.9$ Hz, 1H), 3.25-3.33 (m, 1H), 3.56 (q, $J = 8.7$ Hz, 1H), 3.72 (s, 3H), 5.62-5.69 (m, 2H), 6.89-6.98 (m, 2H), 7.22-7.31 (m, 7H); $^{13}\text{C NMR}$ (CDCl_3) δ 42.4, 44.1, 45.0, 55.4, 111.4, 120.5, 127.3, 127.5, 128.7, 129.1, 130.7, 133.9, 140.0, 206.4 (six carbons missing as a result of overlap); IR (CHCl_3 , cm^{-1}) 1706; HRMS calcd for $\text{C}_{22}\text{H}_{20}\text{O}_2$: 316.14633. Found: 316.14571.

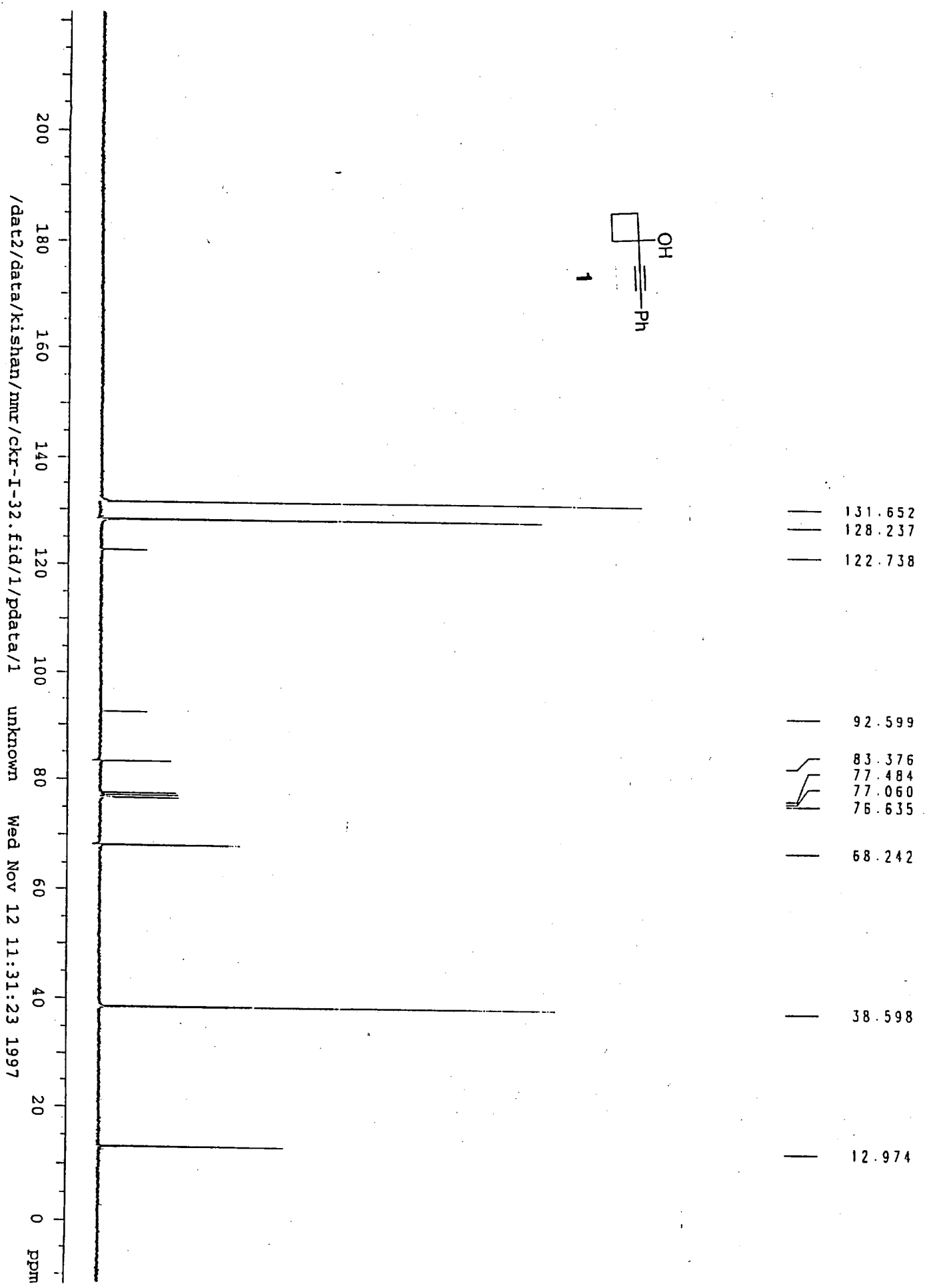
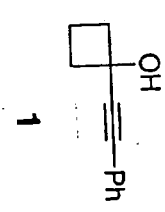
7-Diphenylmethylene-1-methylbicyclo[4.3.0]nonan-8-one (16). The reaction mixture was chromatographed using 60:40 hexanes/ether to afford 94 mg (60%) of the indicated compound as a yellow solid: mp 157-158 °C; $^1\text{H NMR}$ (CDCl_3) δ 1.07-1.47 (m, 7H), 1.59-1.72 (m, 4H), 1.93 (dd, $J = 1.5, 1.2$ Hz, 1H), 2.50 (q, $J = 5.4$ Hz, 1H), 2.68 (d, $J = 18.0$ Hz, 1H), 7.16-7.36 (m, 10H); $^{13}\text{C NMR}$ (CDCl_3) δ 21.7, 24.5, 29.2, 30.2, 34.8, 35.3, 47.2, 49.1, 127.6, 127.7, 128.1,

128.4, 128.9, 140.3, 140.7, 141.9, 148.3, 206.4 (one carbon missing as a result of overlap); IR (CHCl₃, cm⁻¹) 1710; HRMS calcd for C₂₃H₂₃O (M-1): 315.17489. Found: 315.17551.

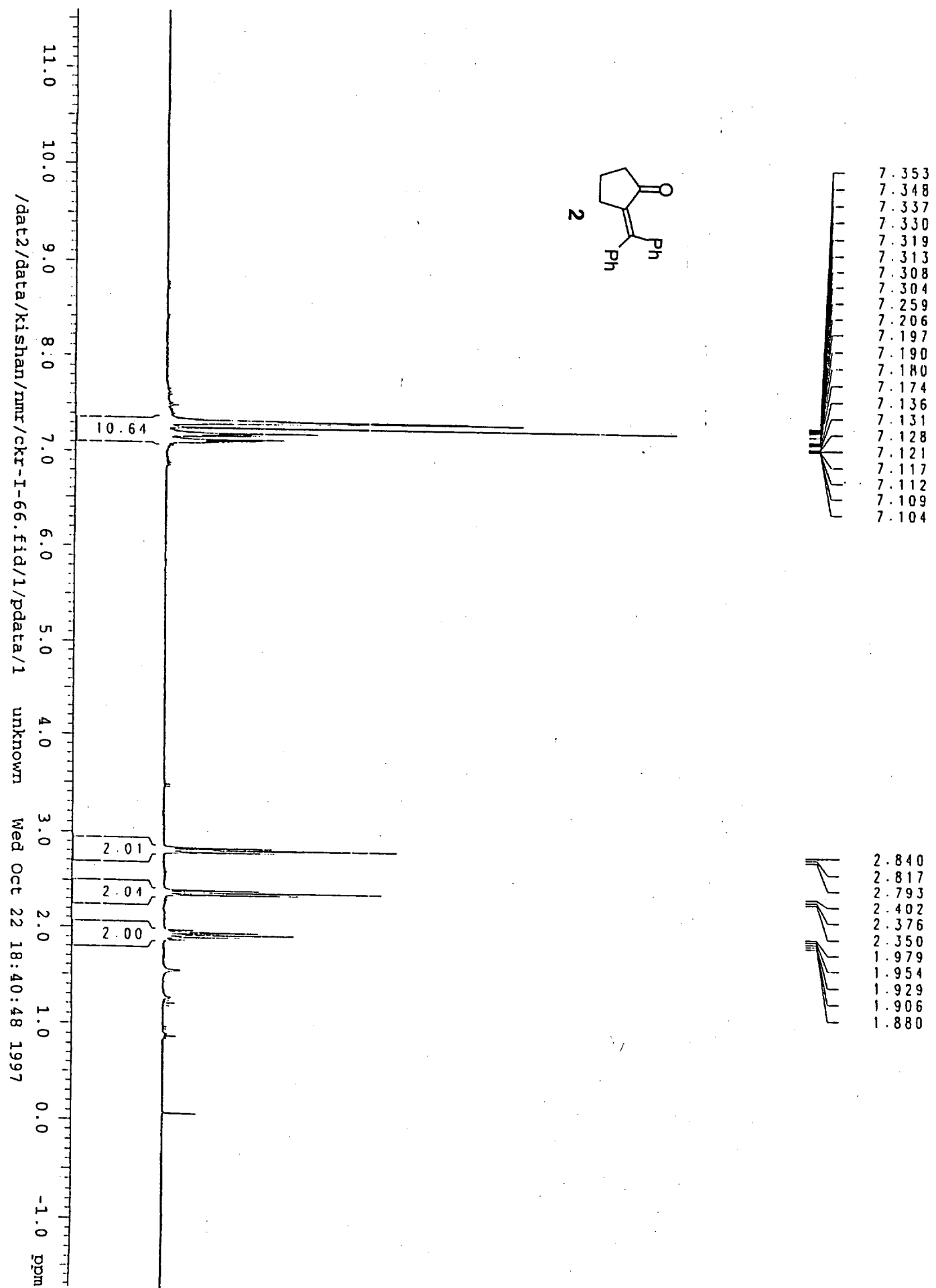
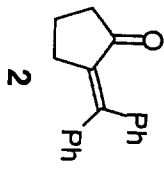
The ¹H and ¹³C NMR spectra for all starting cyclobutanols and all cyclopentanones in Table 1 follow (39 pages).

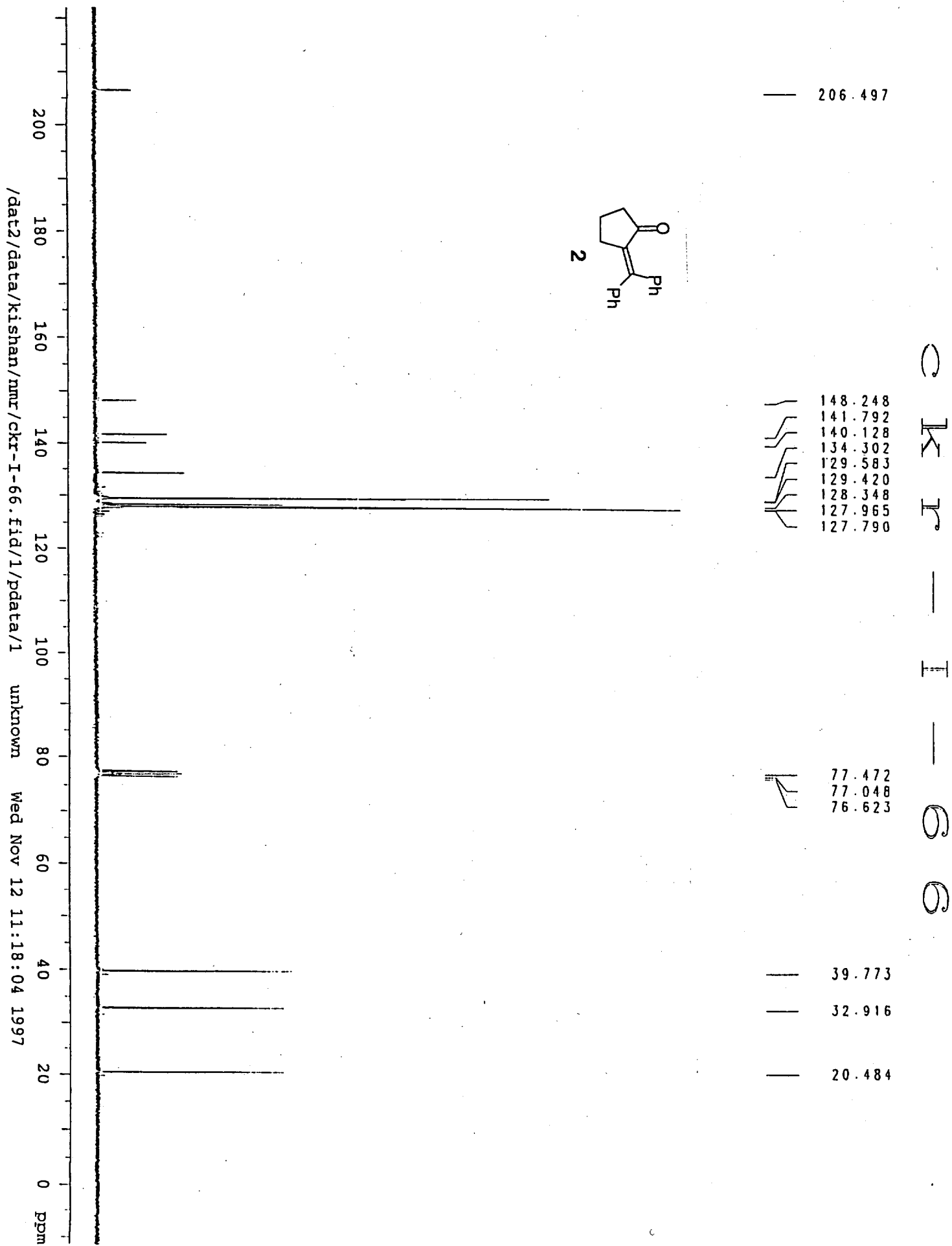


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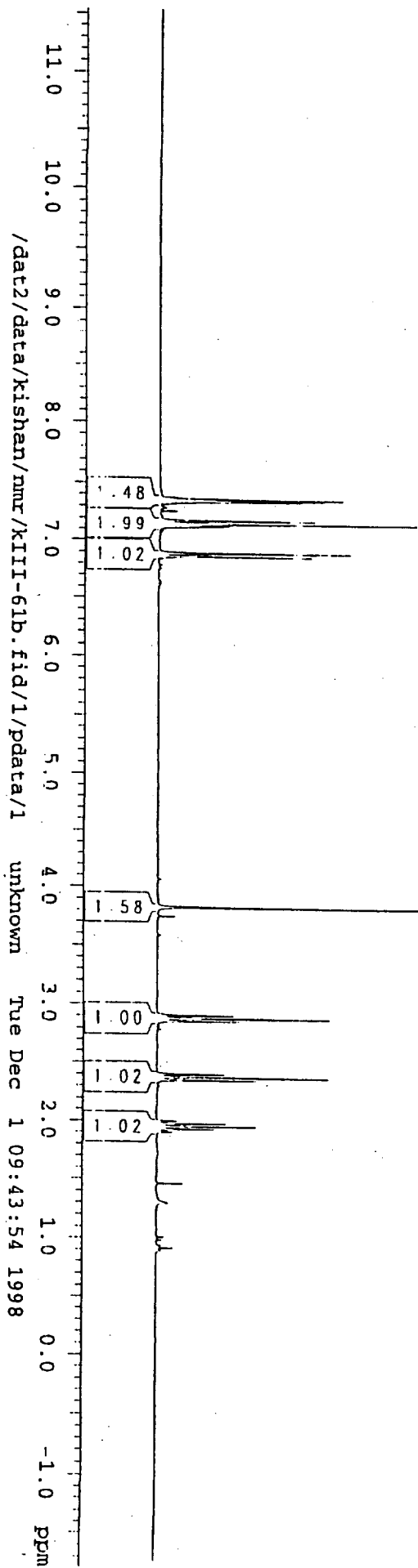
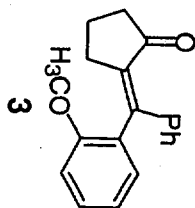


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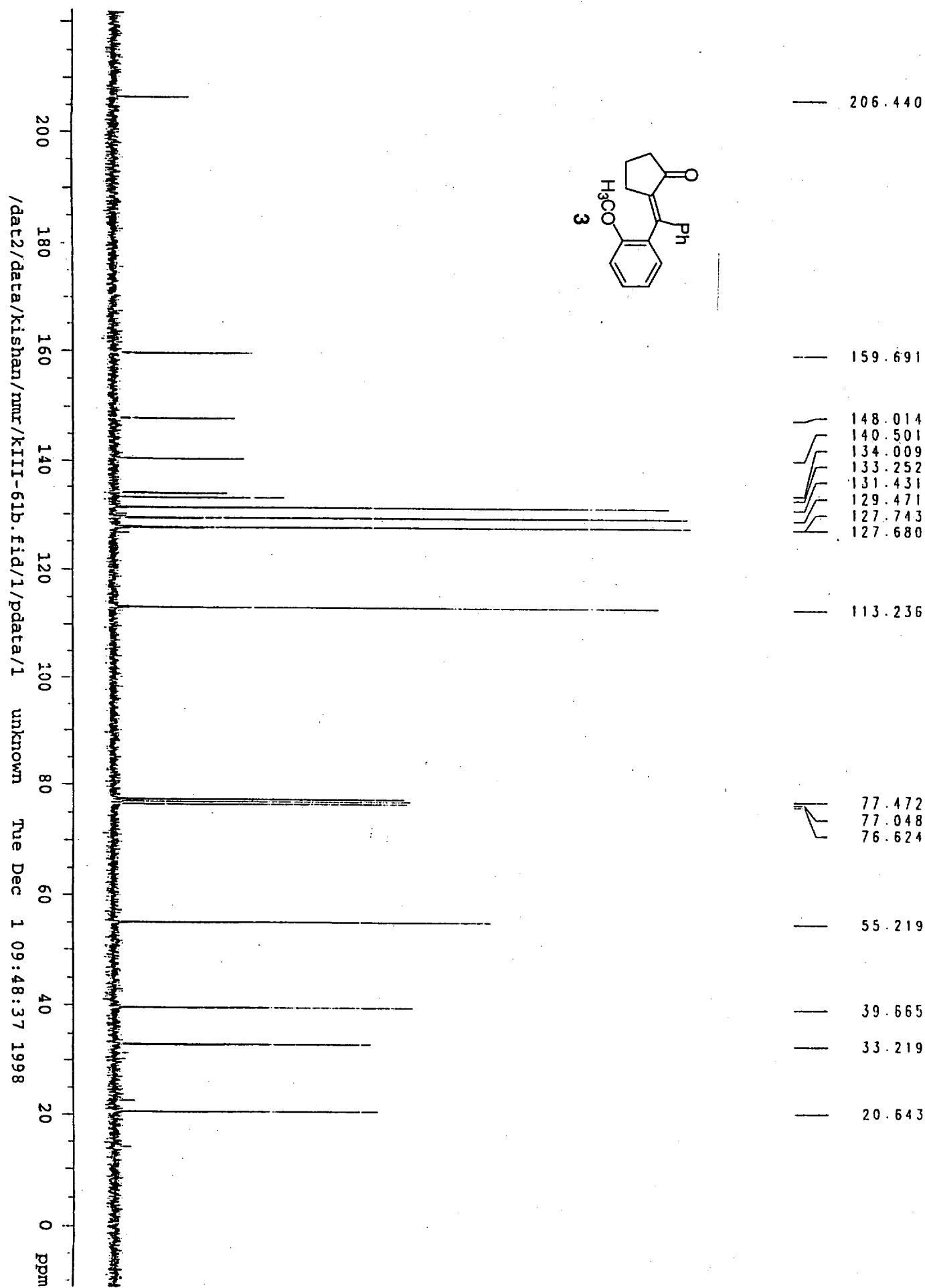
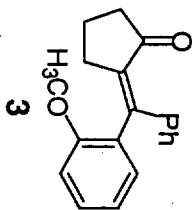


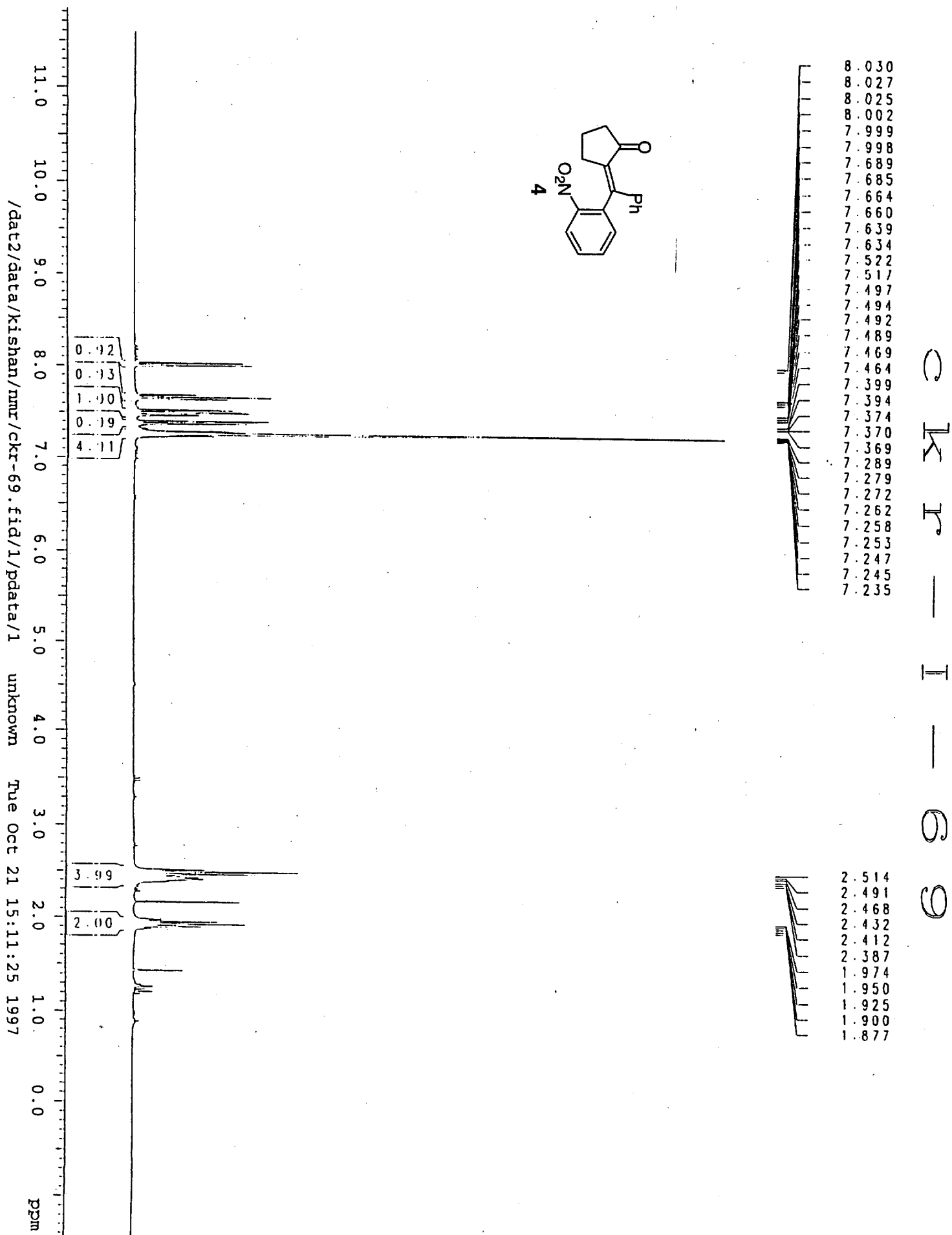
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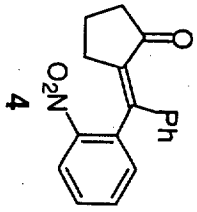
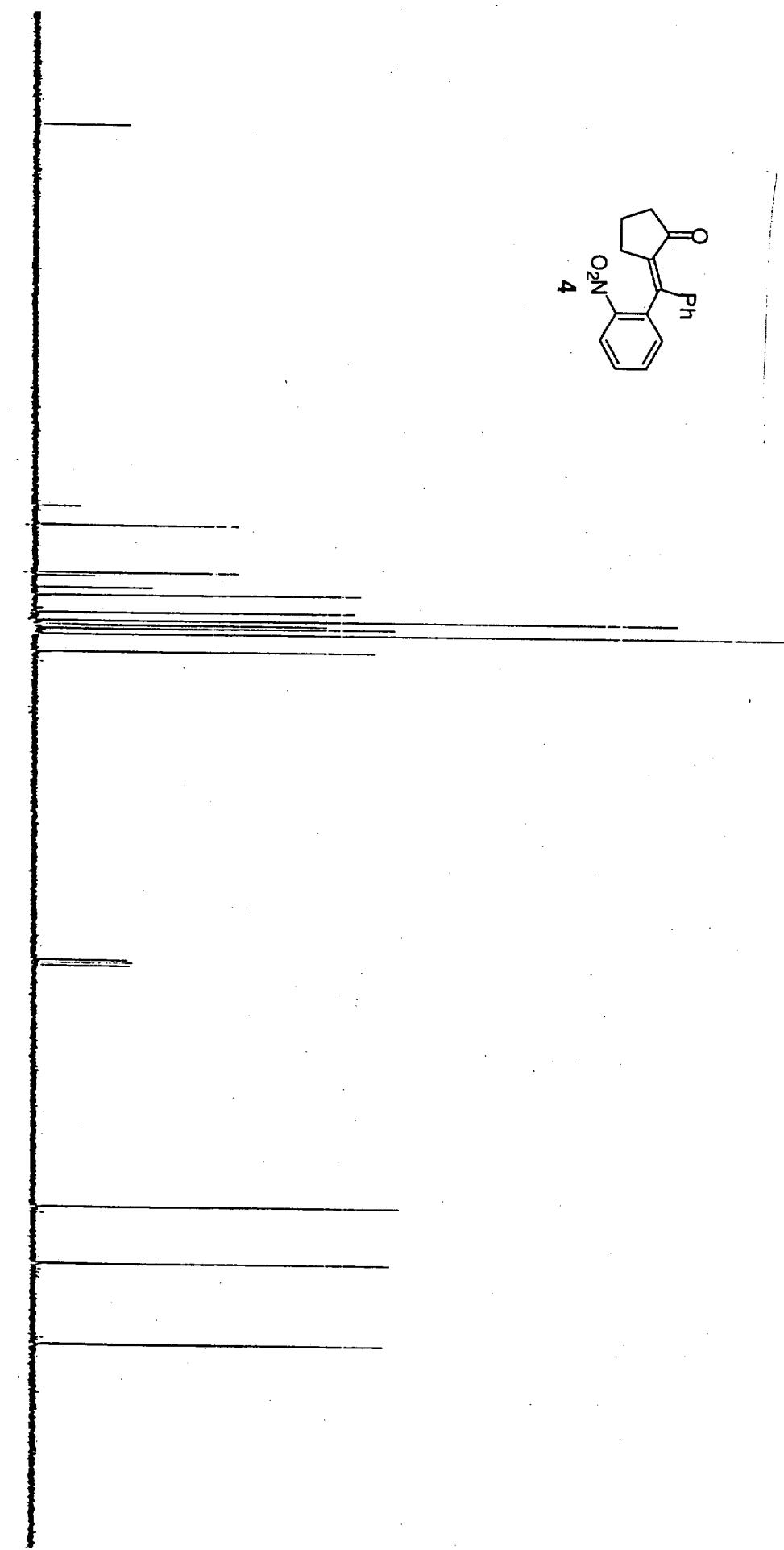
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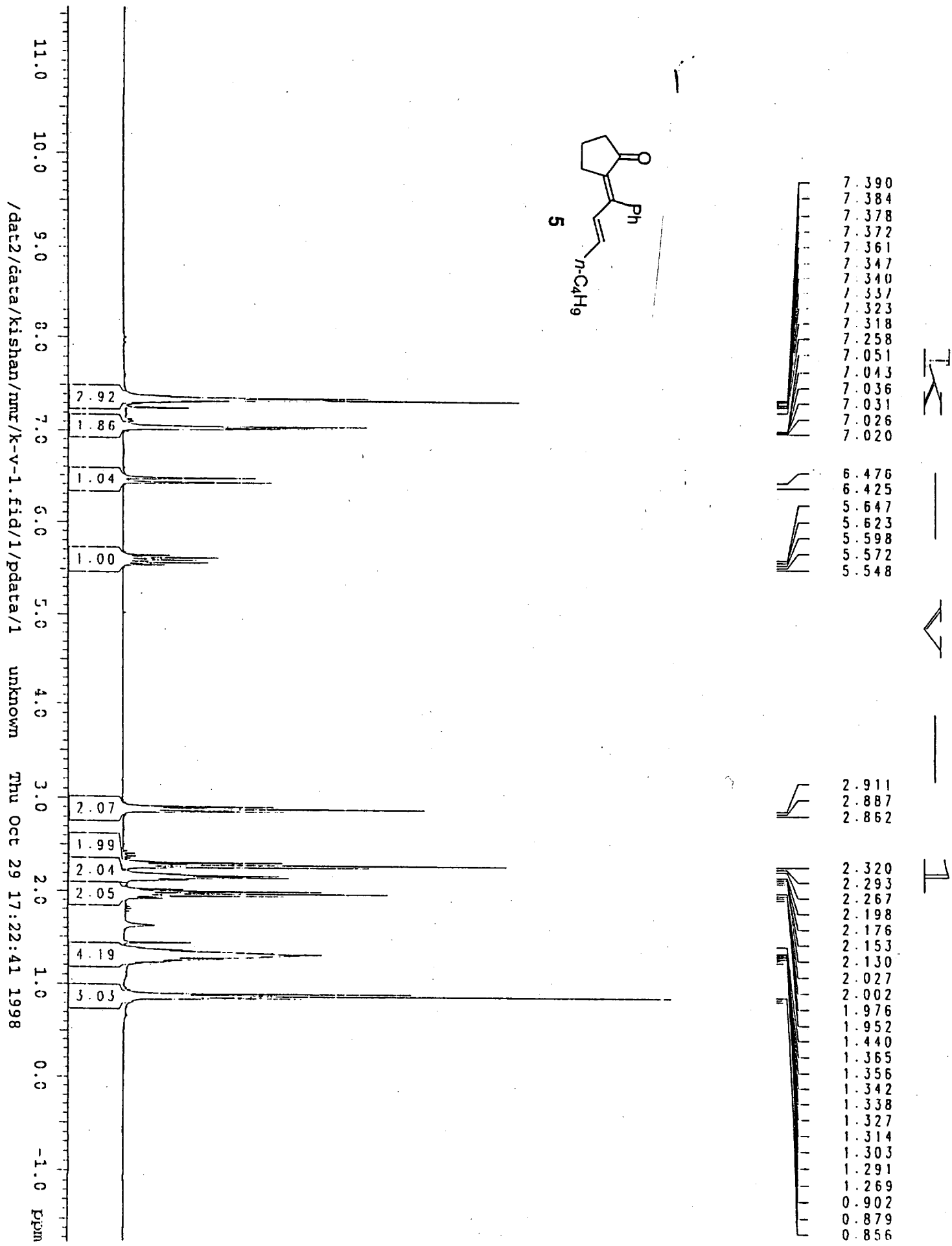
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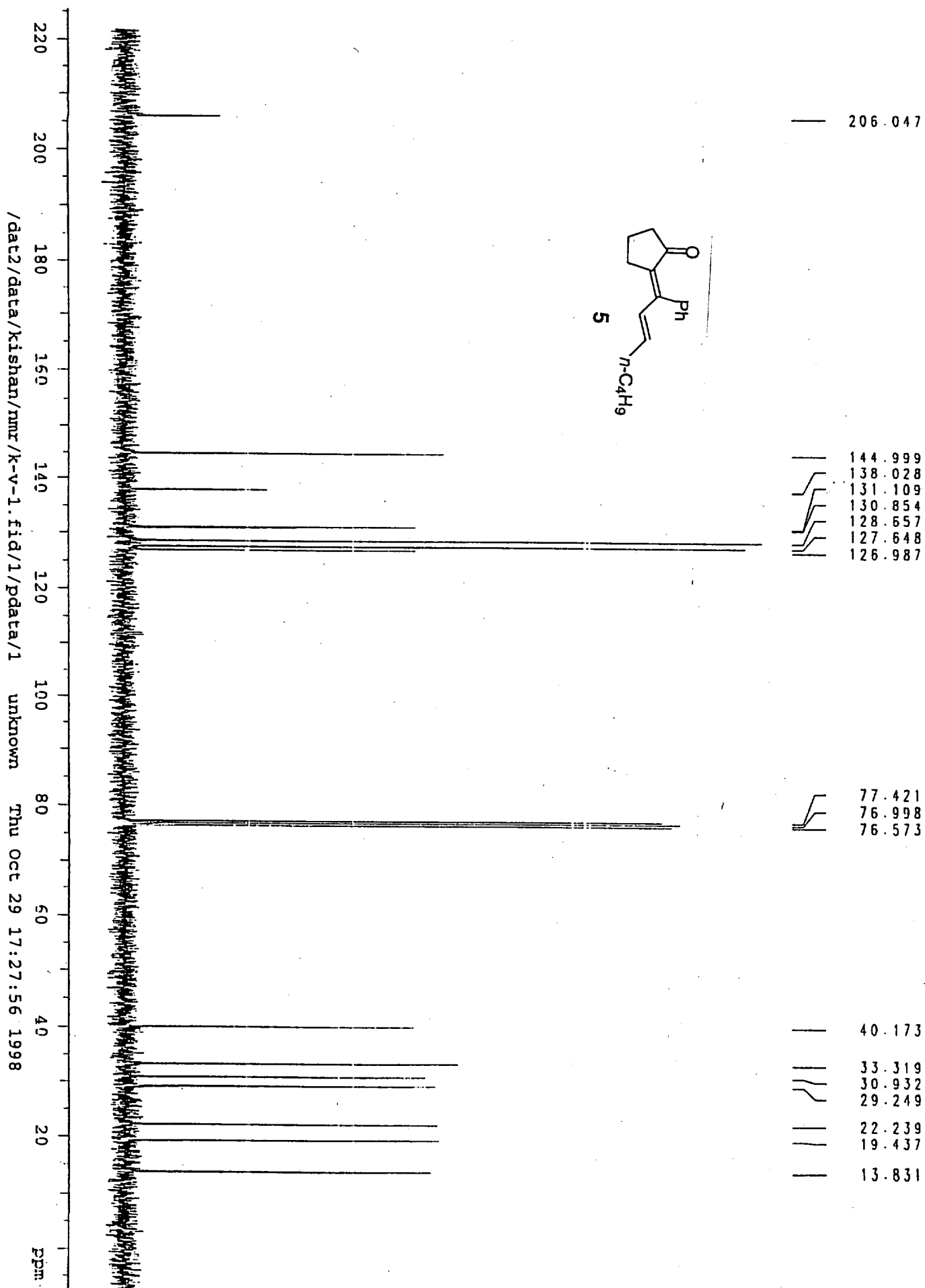
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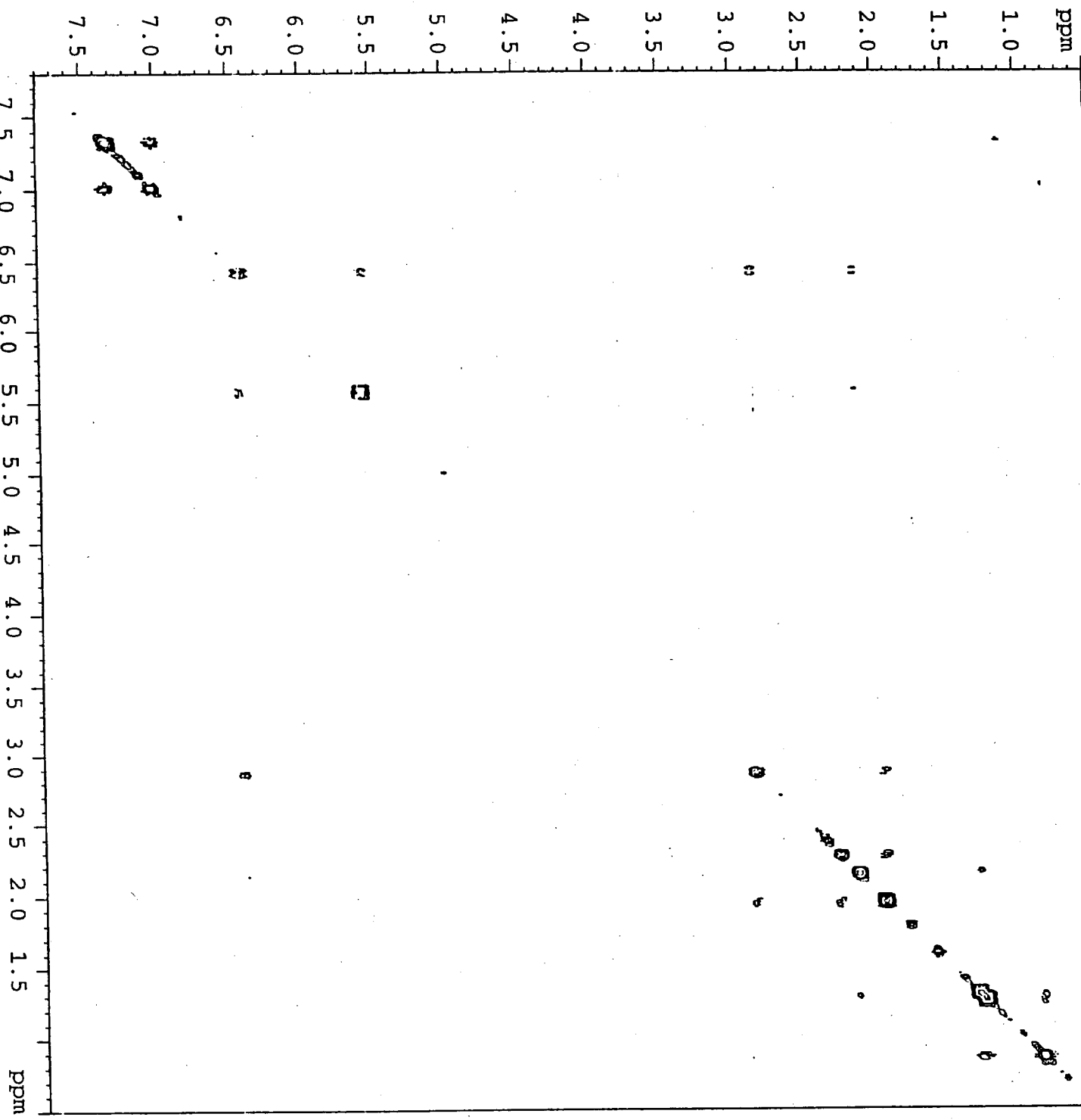
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CKR-I-69





NOESY



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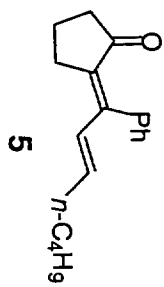
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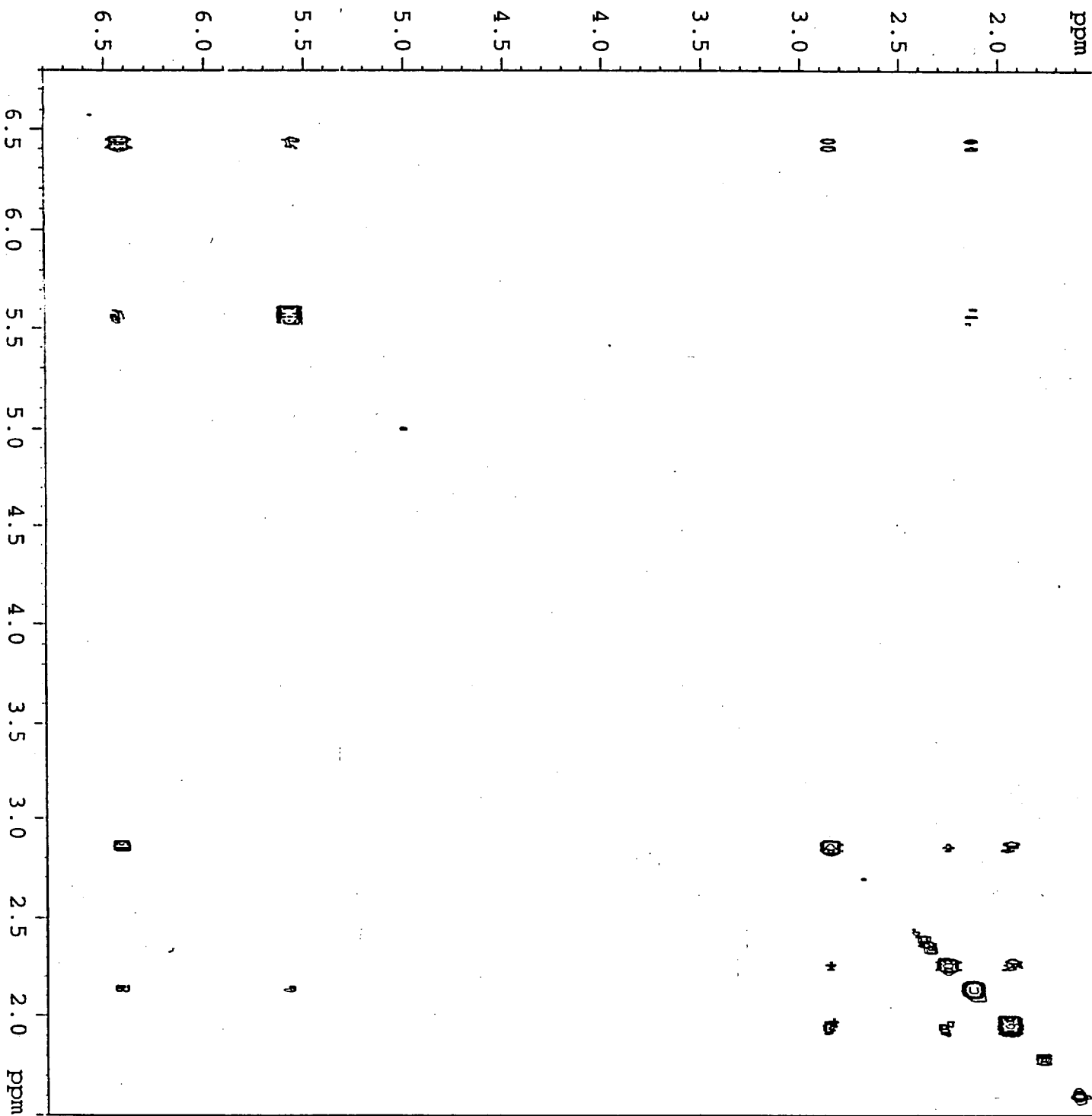
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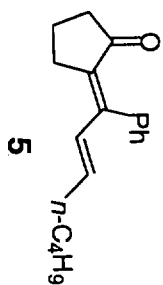
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 PROCNO 1

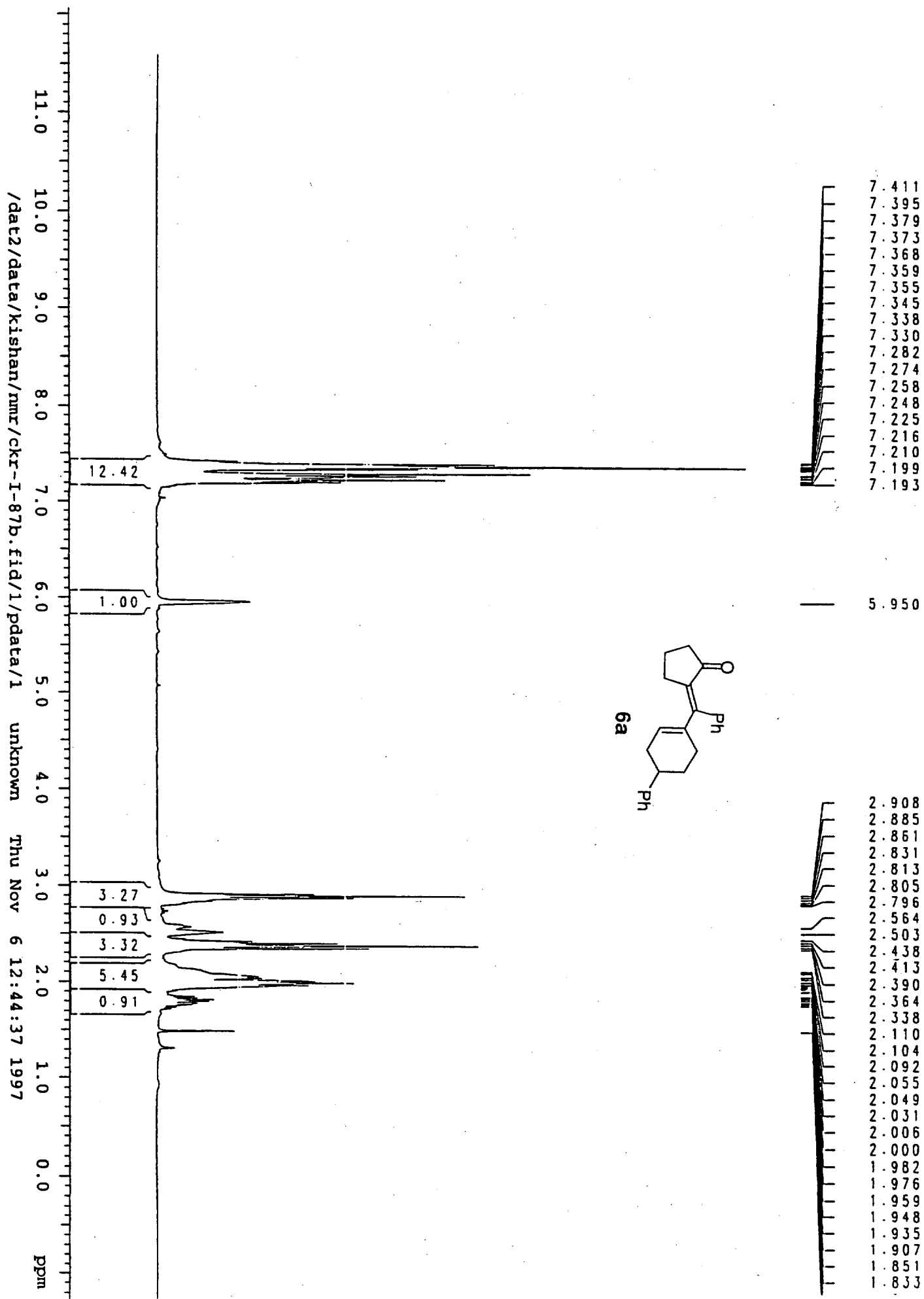
F2 - Acquisition Parameters
 Date_ 981101
 Time 21.01
 INSTRUM spect
 PROBRD 5 mm TXI 31P
 PULPROG noesyat
 TD 2048
 SOLVENT CDCl3
 NS 8
 DS 4
 SWH 3306.878 Hz
 FIDRES 1.61687 Hz
 AQ 0.3097076 sec
 RG 40.3
 INJ 151.200 usec
 DE 4.50 usec
 TE 300.0 K

F1 - Acquisition Parameters
 MD0 1
 TD 512
 SF01 400.1316 MHz
 FIDRES 6.458747 Hz
 SFO 8.264 Ppm

F2 - Processing Parameters
 SI 1024
 SF 400.1300180 MHz
 KDW OSINE
 SSB 2
 LB 0.00 Hz
 GB 6
 PC 1.00

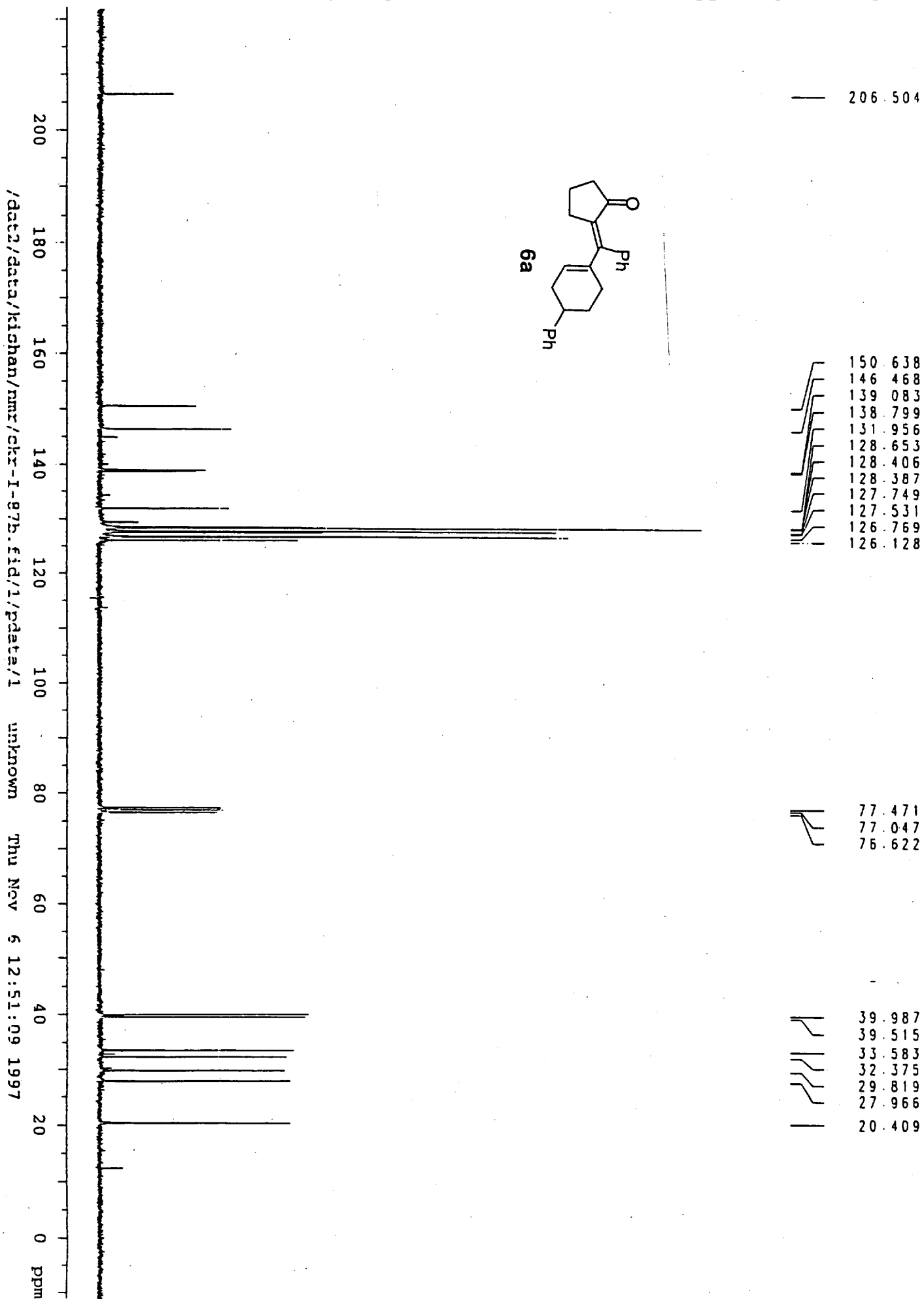
F1 - Processing Parameters
 SI 1024
 WC2 States-TPI
 SF 400.1300180 MHz
 KDW OSINE
 SSB 2
 LB 0.00 Hz
 GB 0





CKR-I-87b

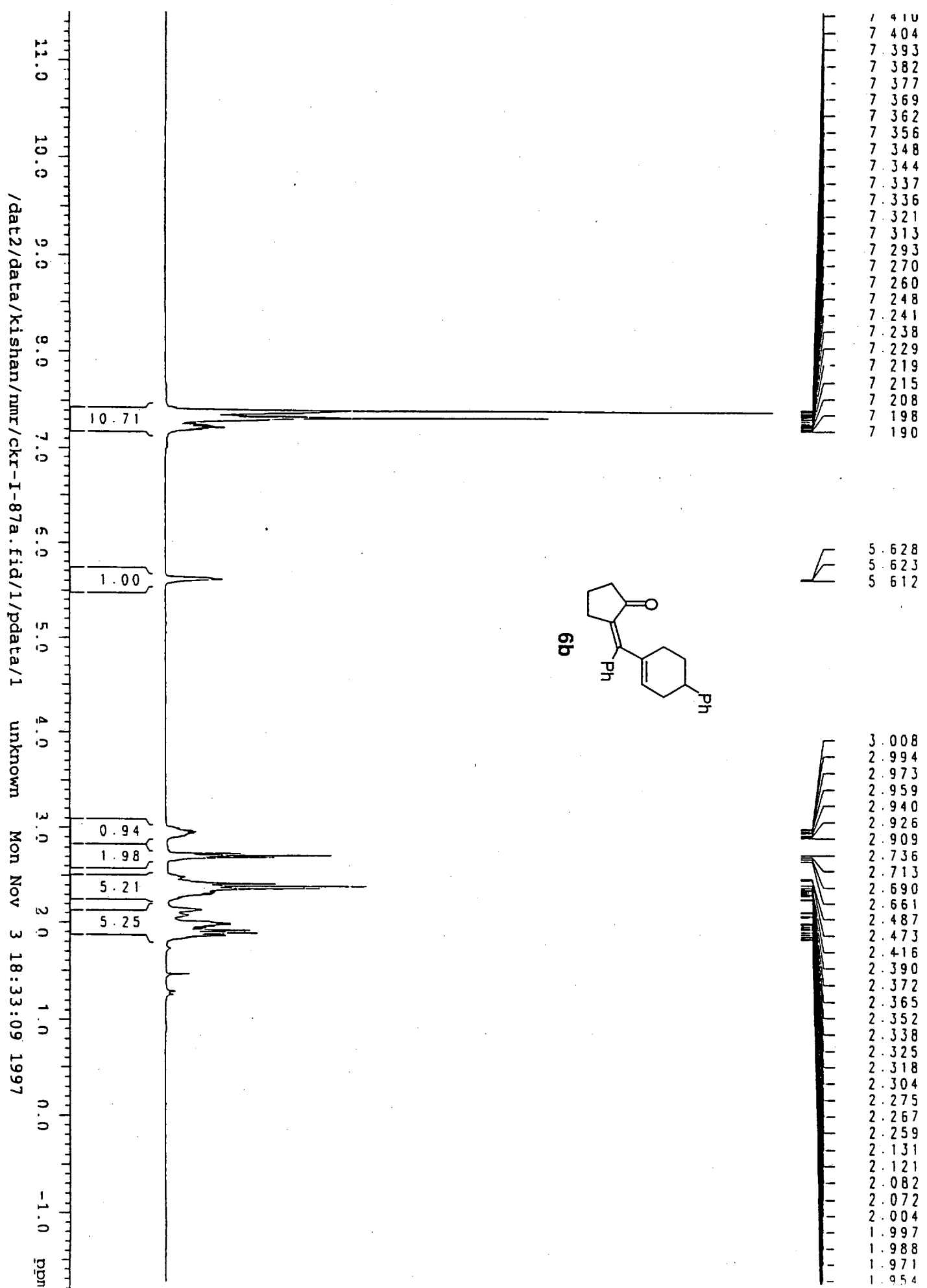
/dat2/data/kishan/nmr/ckr-I-87b.fid/1/pdata/1 unknown Thu Nov 6 12:44:37 1997



C K R - I - 8 7 b

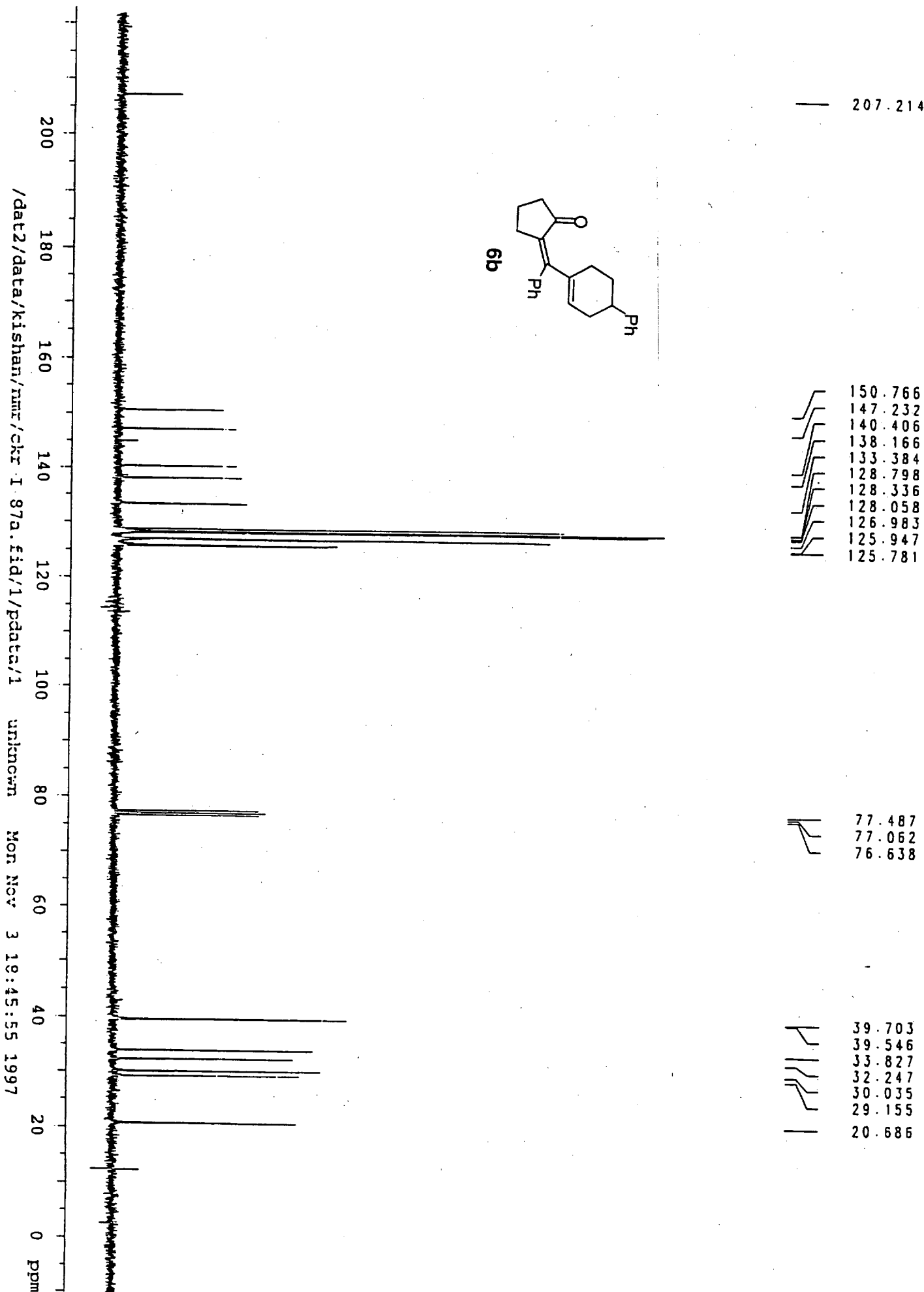
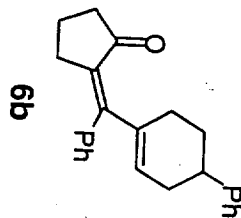
/data/data/kishan/nmr/ckr-I-87b.fid/1/pdata/1 unknown Thu Nov 6 12:51:09 1997

C K R - I - 8 7 a

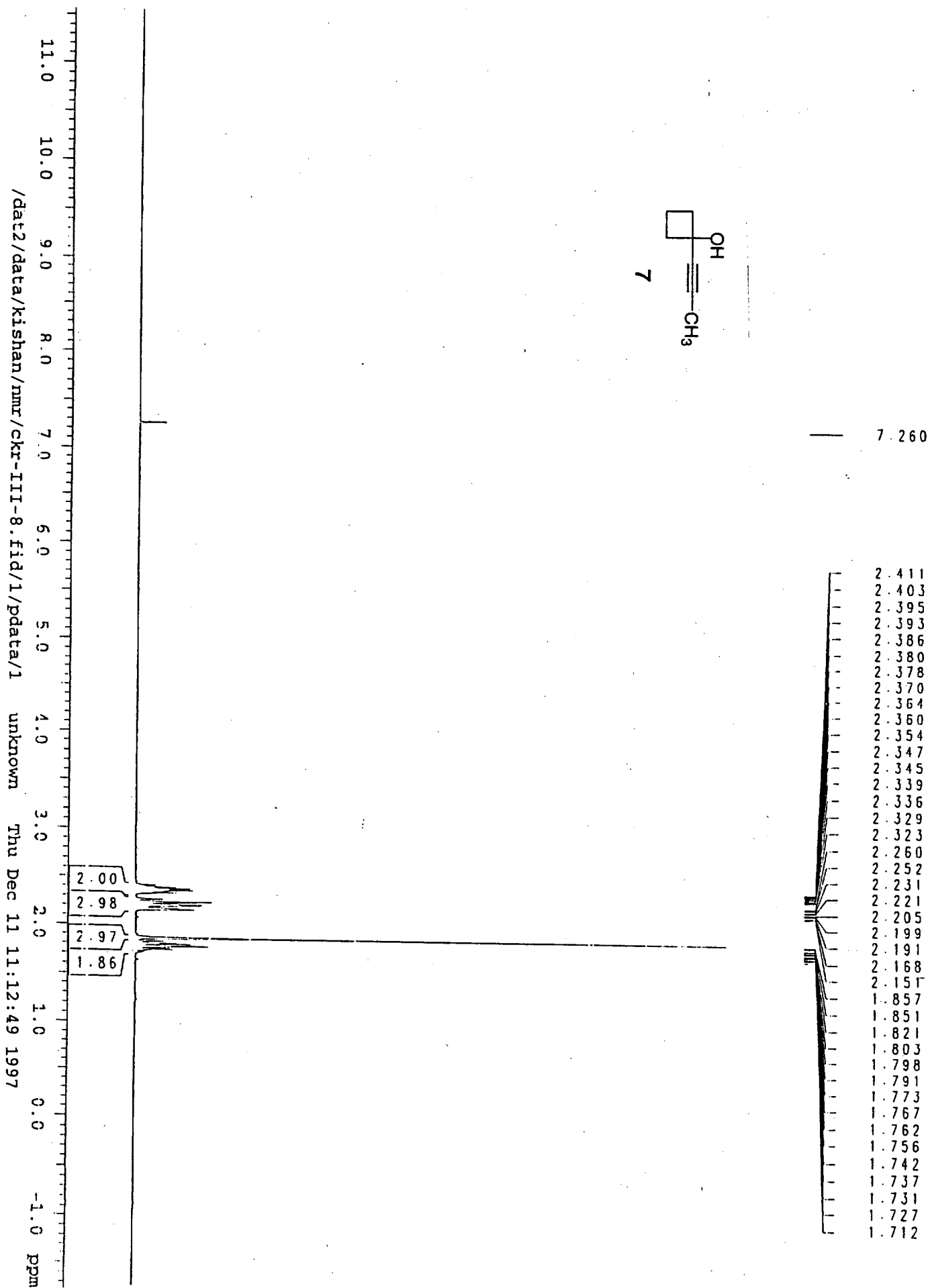
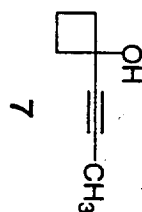


/dat2/data/kishan/nmr/ckr-I-87a.fid/1/pdata/1 unknown Mon Nov 3 18:33:09 1997

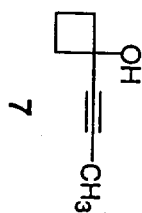
CKR - I - 87a



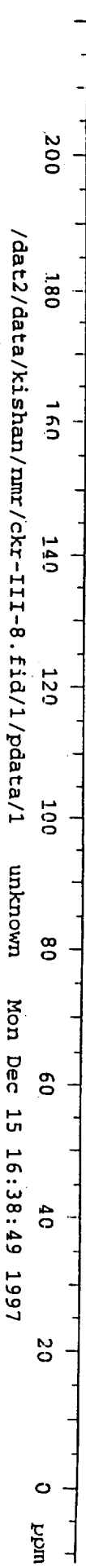
CKR - I I I - 8



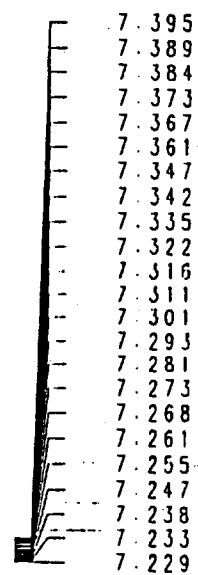
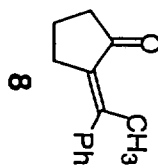
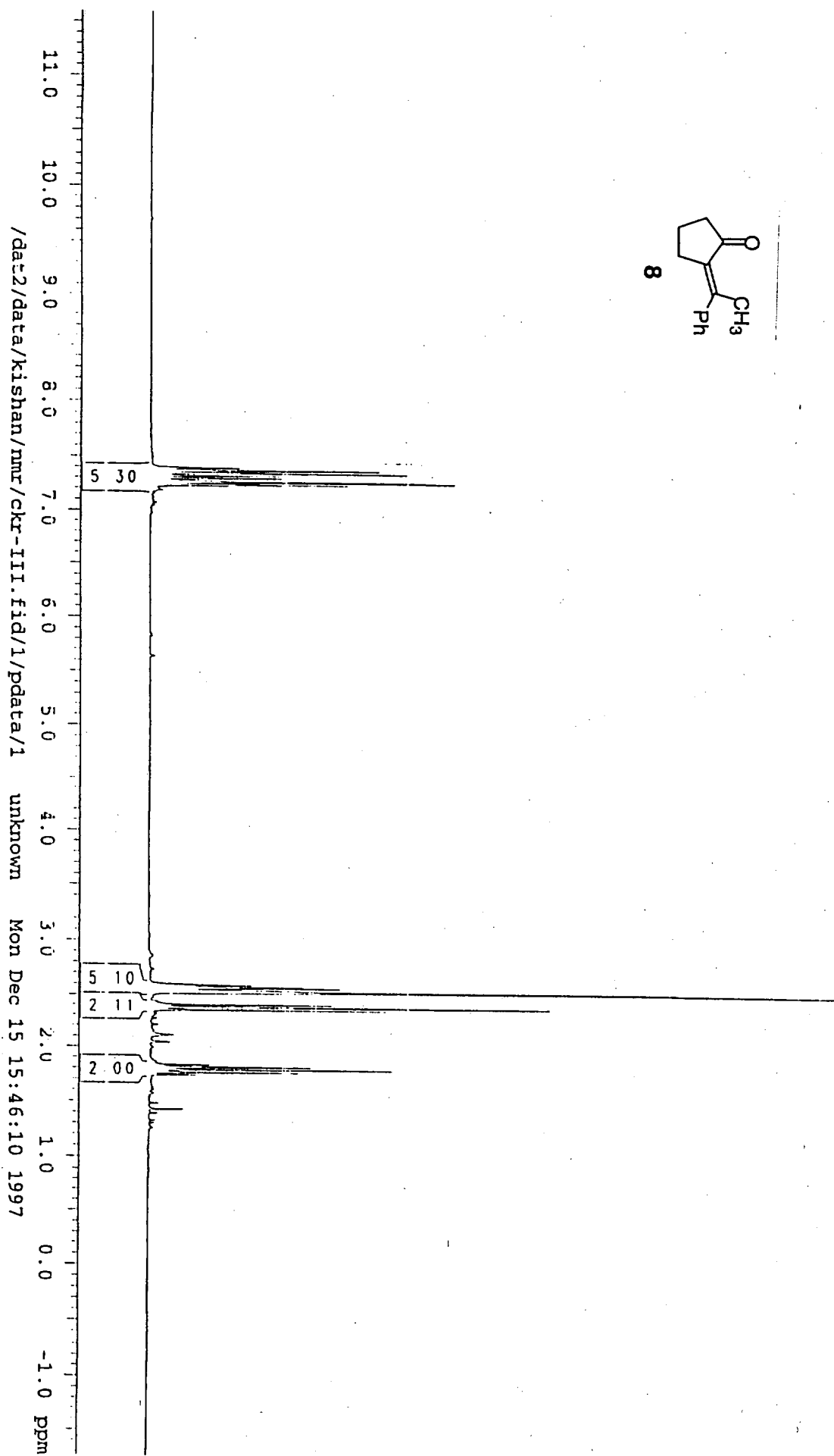
CKR-III-8



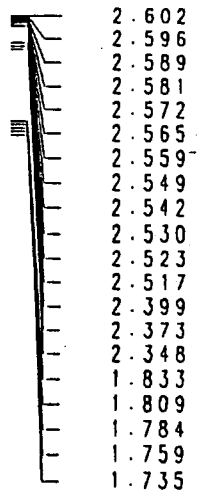
	82.958
	79.348
	77.473
	77.049
	76.625
	67.978
	38.552
	12.749
	3.551



/data2/data/kishan/nmr/ckr-III-8.fid/1/pdata/1 unknown Mon Dec 15 16:38:49 1997



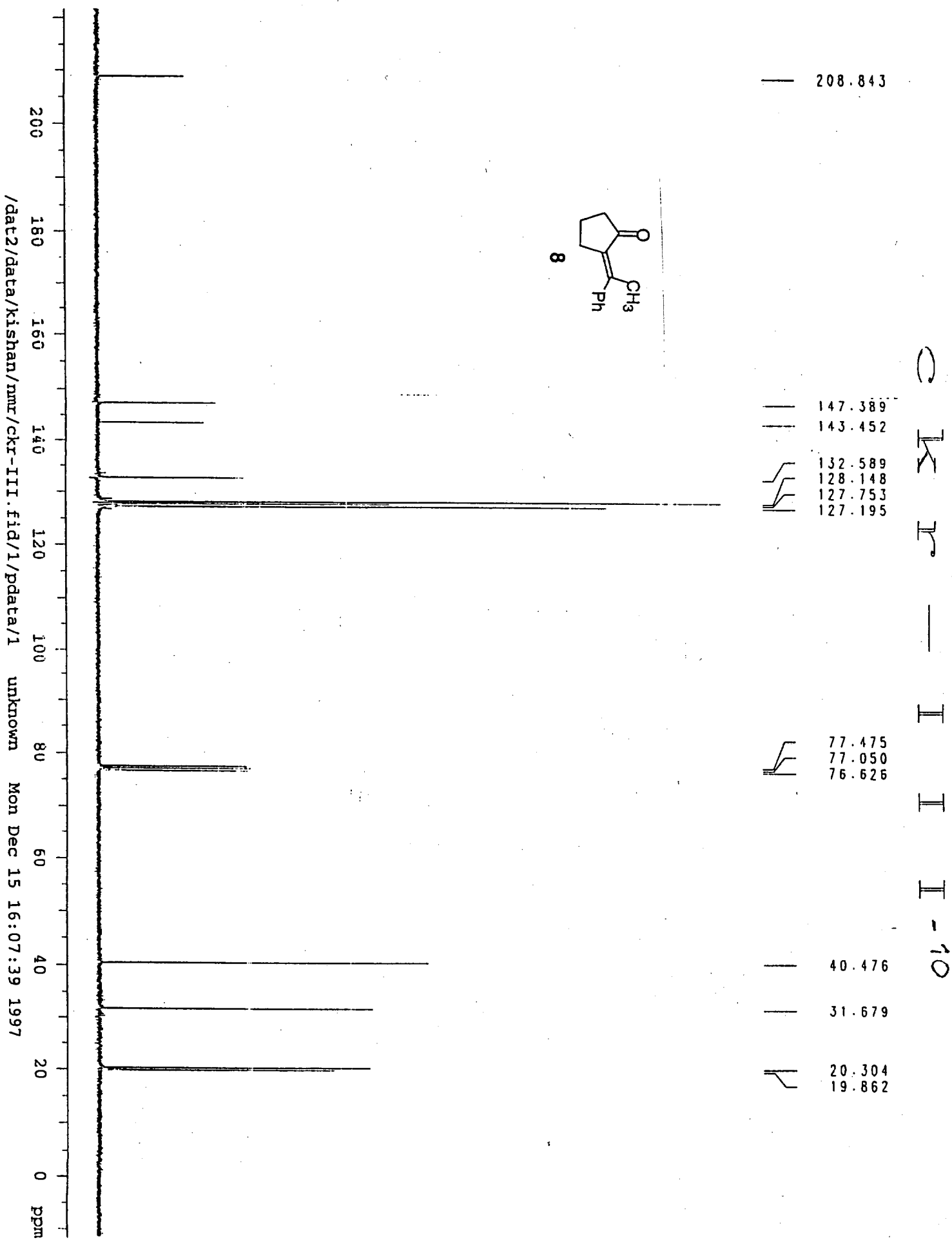
7.395
7.389
7.384
7.373
7.367
7.361
7.347
7.342
7.335
7.322
7.316
7.311
7.301
7.293
7.281
7.273
7.268
7.261
7.255
7.247
7.238
7.233
7.229

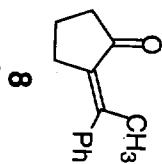
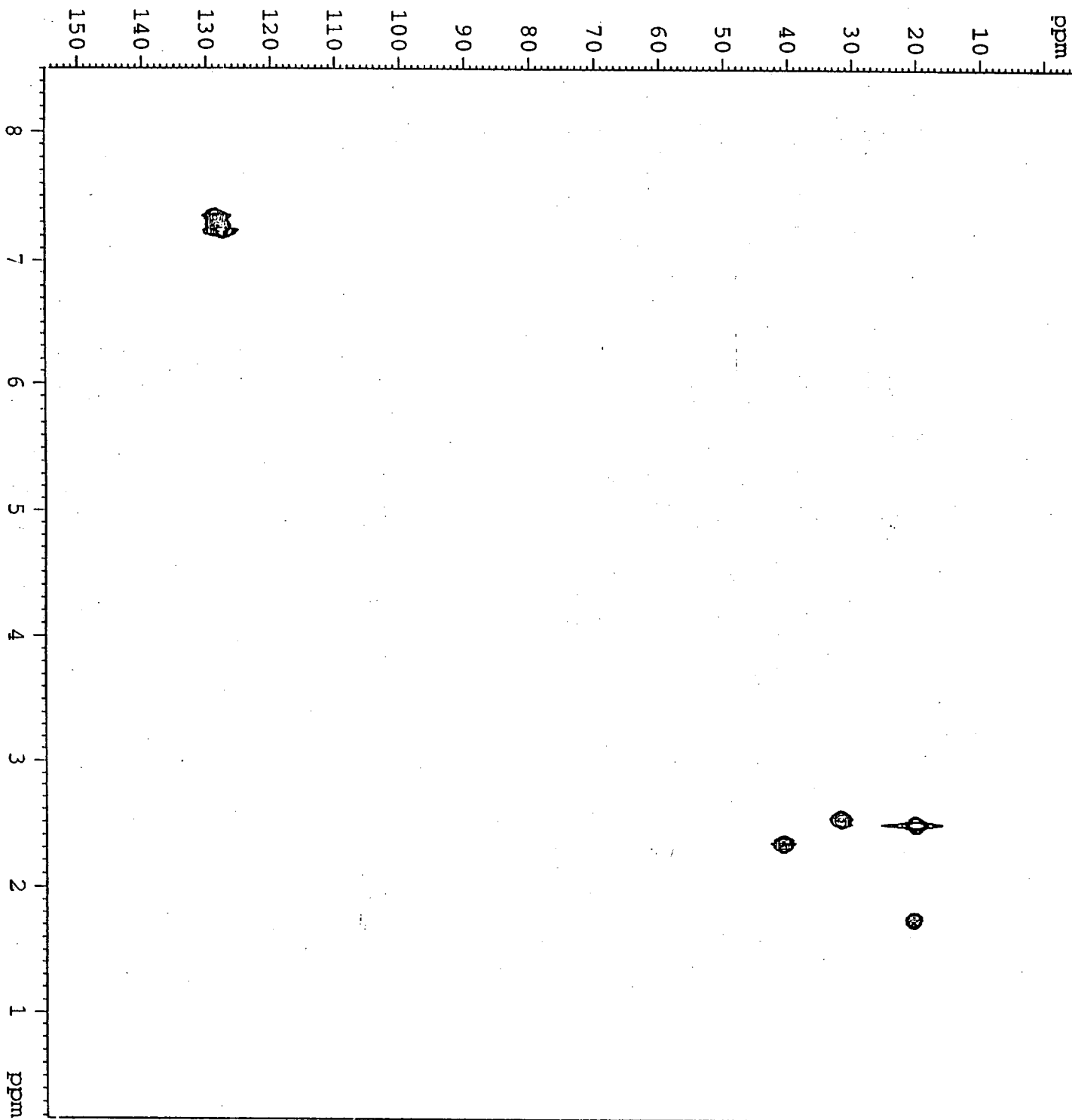


2.602
2.596
2.589
2.581
2.572
2.565
2.559
2.549
2.542
2.530
2.523
2.517
2.399
2.373
2.348
1.833
1.809
1.784
1.759
1.735

CKR - I I I - 10

/dat2/data/Kishan/nmr/ckr-III.fid/1/pdata/1 unknown Mon Dec 15 15:46:10 1997





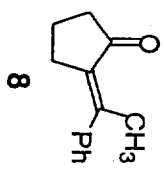
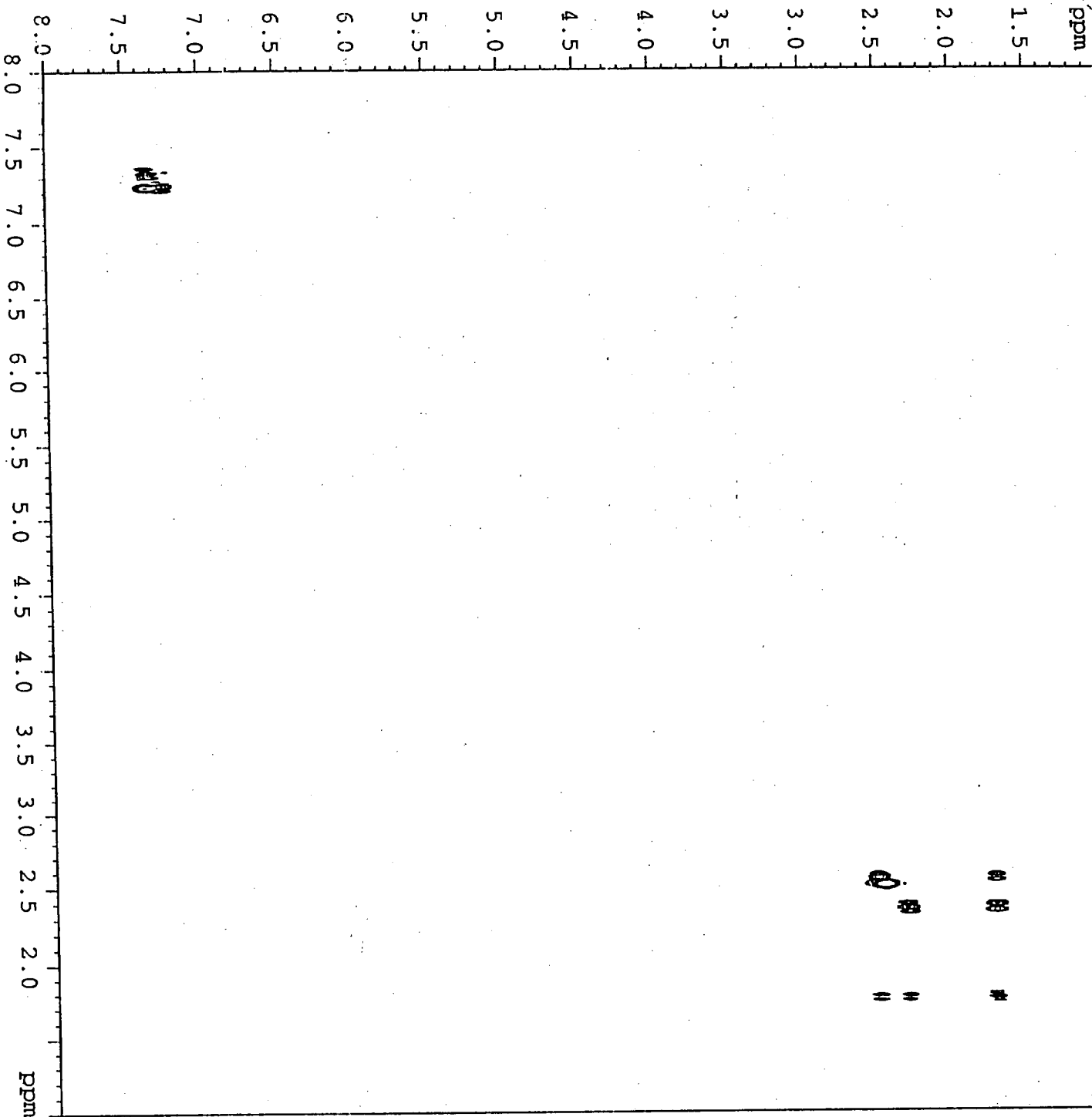
Current Data Parameters
 NAME Id.8000
 EXPNO 5
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980206
 Time 15.33
 INSTRUM spect
 PROBHD 5 mm TXI 31P
 PULPROG Inv4gs
 TD 1024
 SOLVENT CDCl3
 NS 1
 DS 8
 SWH 3333.333 Hz
 FIDRES 3.255208 Hz
 AQ 0.1536500 sec
 RG 9195.2
 DW 150.000 usec
 DE 4.50 usec
 TE 300.0 K

F1 - Acquisition Parameters
 NDO 2
 TD 128
 SFO1 100.6203 MHz
 FIDRES 125.754524 Hz
 SW 159.974 ppm

F2 - Processing Parameters
 SI 1024
 SF 400.1300180 MHz
 WDM QSIINE
 SSB 2
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing Parameters
 SI 1024
 MC2 OF
 SF 100.6127290 MHz
 WDM QSIINE
 CSD 2
 LB 0.00 Hz
 GB 0



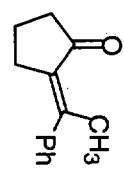
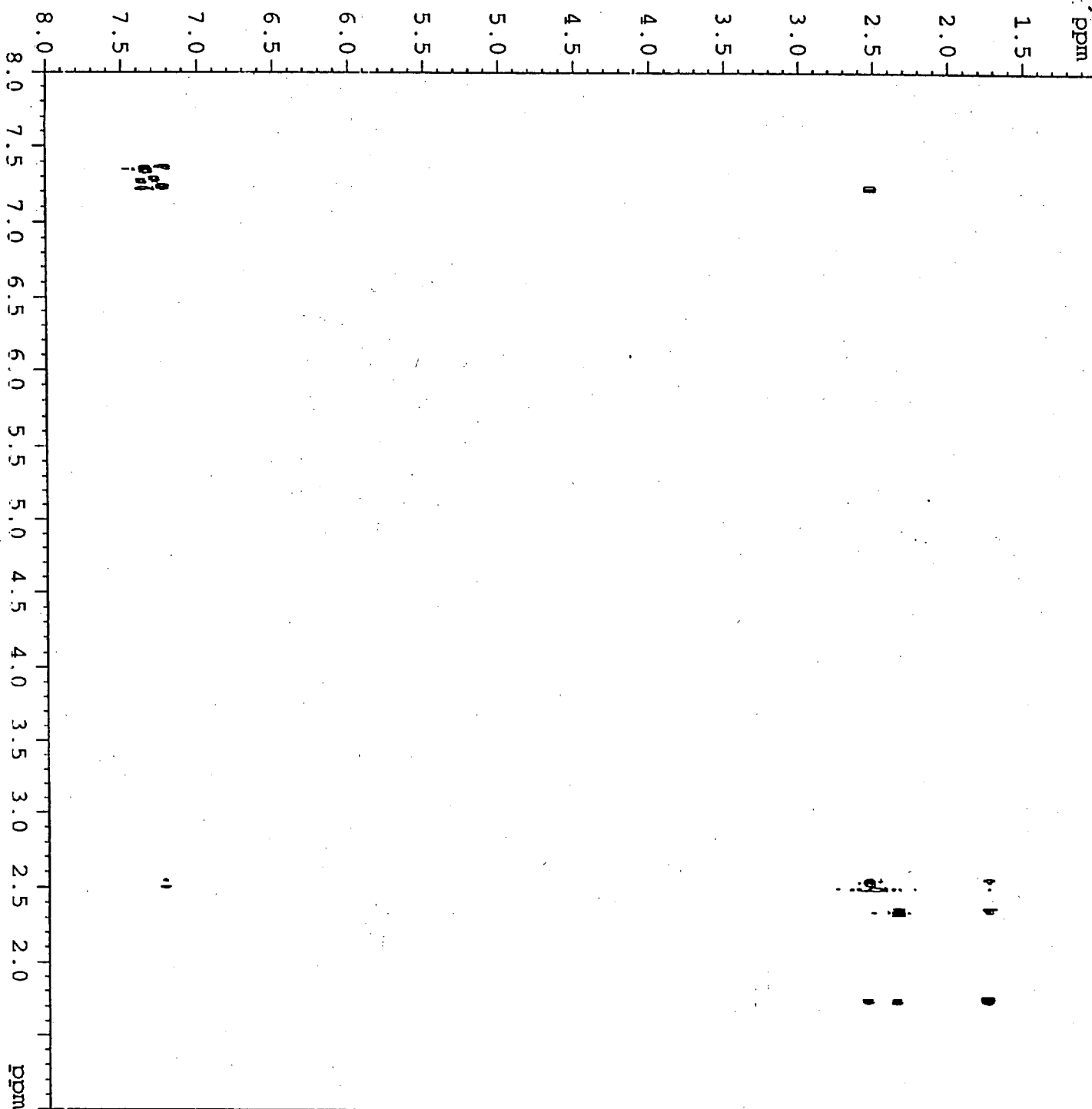
Current Data Parameters
 NAME Id.8000
 EXENO 2
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980206
 Time 15.04
 INSTRUM spect
 PROBHD 5 mm TXI 31P
 PULPROG cosyg
 TD 1024
 SOLVENT CDC13
 NS 1
 DS 16
 SWH 3333.333 Hz
 FIDRES 3.255208 Hz
 AQ 0.1536500 sec
 RG 64
 DW 150.000 usec
 DE 4.50 usec
 TE 300.0 K

F1 - Acquisition parameters
 MD0 1
 TD 128
 SF01 400.1318 MHz
 FIDRES 26.041666 Hz
 SW 8.331 Ppm

F2 - Processing parameters
 SI 1024
 SF 400.1300180 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing parameters
 SI 512
 MC2 OF
 SF 400.1300180 MHz
 WDM SINE
 SSB 0
 LB 0.00 Hz
 GB 0



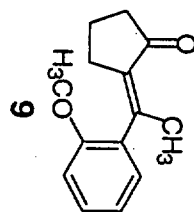
Current Data Parameters
 NAME 1d.8000
 EXPRN 10
 PROCNO 1

F2 - Acquisition Parameters
 Date_ 980206
 Time 15.41
 INSTRUM spect
 PROBRD 5 mm ¹H 31P
 PULPROG noesyzt
 TD 2048
 SOLVENT CDCl3
 NS 8
 DS 4
 SMH 3333.333 Hz
 FIDRES 1.627604 Hz
 AQ 0.3072500 sec
 RG 20.2
 DM 150.000 usec
 DE 4.50 usec
 TE 300.0 K

F1 - Acquisition Parameters
 RD0 1
 TD 340
 SFO1 400.1318 MHz
 FIDRES 9.803922 Hz
 SW 8.331 Ppm

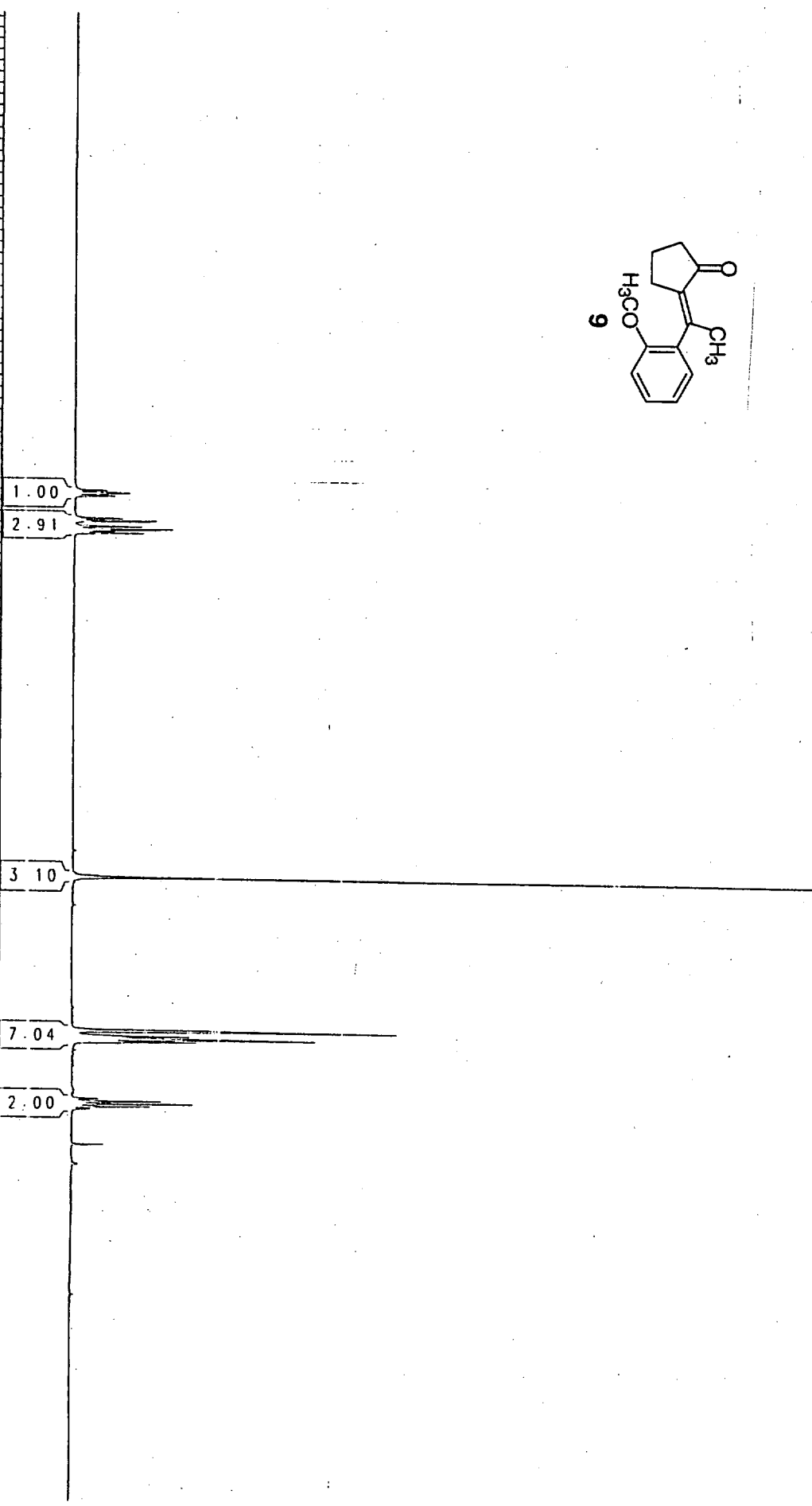
F2 - Processing Parameters
 SI 1024
 SF 400.1300180 MHz
 WDW QSI
 SSB 2
 LB 0.00 Hz
 GB 0
 PC 1.00

F1 - Processing Parameters
 SI 1024
 MC2 States-TPPI
 SF 400.1300180 MHz
 WDW QSI
 SSB 2
 LB 0.00 Hz
 GB 0



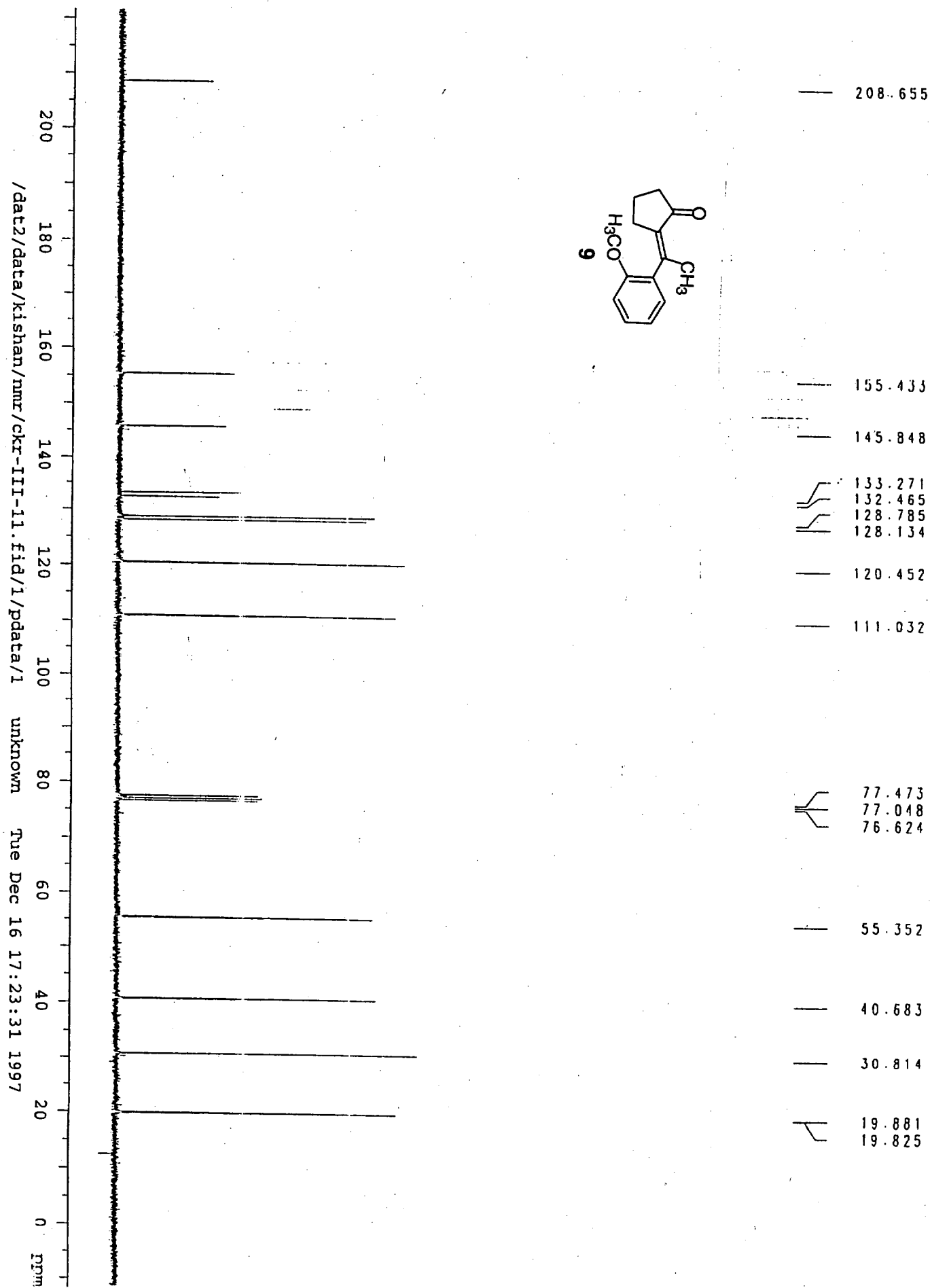
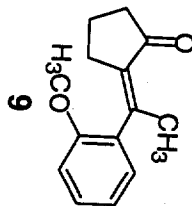
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
-1.0
ppm

/dat2/data/kishan/nmr/ckr-III-11.fid/1/pdata/1
unknown
Tue Dec 16 14:53:10 1997



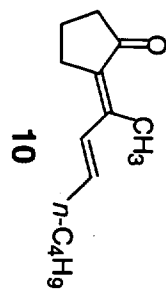
7.295
7.289
7.271
7.268
7.264
7.262
7.259
7.243
7.237
7.040
7.034
7.016
7.010
6.964
6.961
6.940
6.936
6.932
6.926
6.915
6.911
6.897
3.792
2.447
2.440
2.434
2.390
2.380
2.373
2.365
2.349
2.339
1.825
1.801
1.776
1.751
1.726

C K R - I I I - 1 1



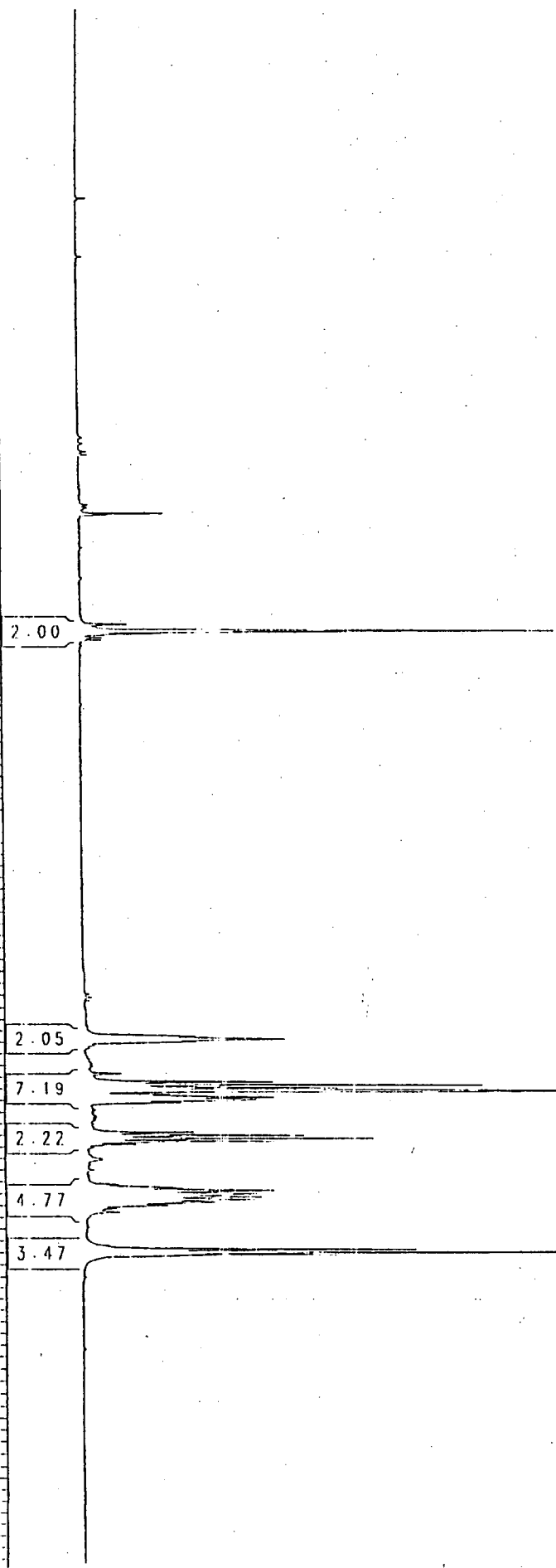
C K R — I I I — I I

/dat2/data/kishan/nmr/ckr-III-11.fid/1/pdata/1 unknown Tue Dec 16 17:23:31 1997

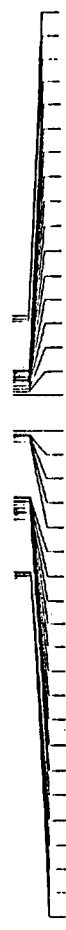


11.0 10.0 9.0 8.0 7.0 6.0 5.0 4.0 3.0 2.0 1.0 0.0 -1.0 ppm

/dat2/data/kishan/nmr/ckr-III-14.fid/1/pdata/1 unknown Wed Dec 24 09:45:45 1997



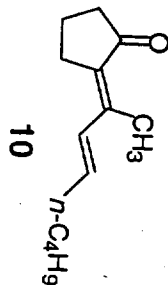
6.249
6.237
6.226
6.216



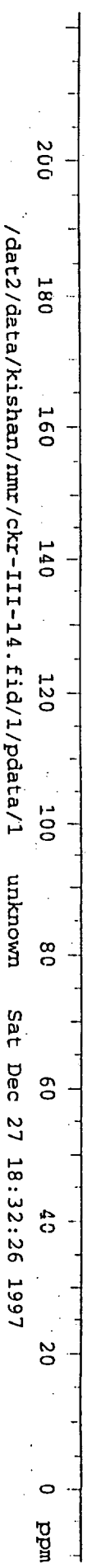
2.751
2.727
2.709
2.363
2.353
2.336
2.327
2.310
2.300
2.289
2.284
2.279
2.267
2.244
2.222
2.203
2.180
1.923
1.898
1.888
1.873
1.848
1.447
1.438
1.421
1.412
1.394
1.370
1.358
1.346
1.334
1.323
1.296
0.931
0.921
0.906
0.897
0.884

CKR - I I I - 1 4

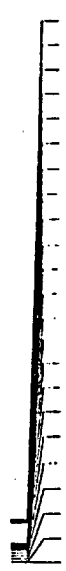
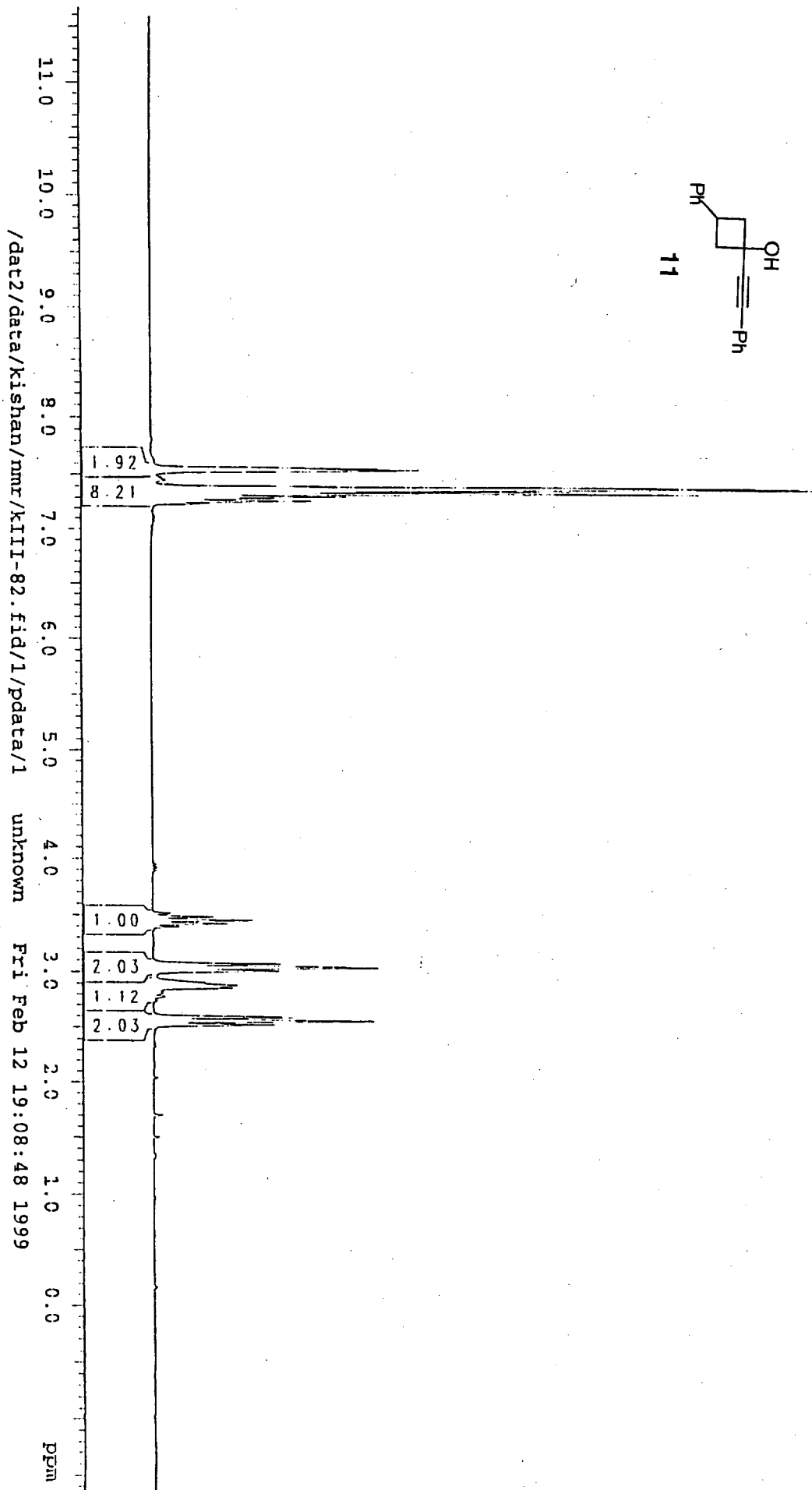
CKR-III-14



—	208.978
	142.708
	139.987
∨	131.040
∨	131.001
∨	77.470
∨	77.047
∨	76.623
—	40.832
∨	33.386
∨	31.300
∨	29.279
∨	22.311
∨	19.446
∨	13.919
∨	13.166



/dat2/data/kishan/nmr/ckr-III-14.fid/1/pdata/1 unknown Sat Dec 27 18:32:26 1997



- 7.561
- 7.555
- 7.549
- 7.546
- 7.541
- 7.537
- 7.535
- 7.529
- 7.398
- 7.386
- 7.381
- 7.378
- 7.373
- 7.368
- 7.365
- 7.359
- 7.350
- 7.348
- 7.329
- 7.305
- 7.287
- 7.264
- 7.258

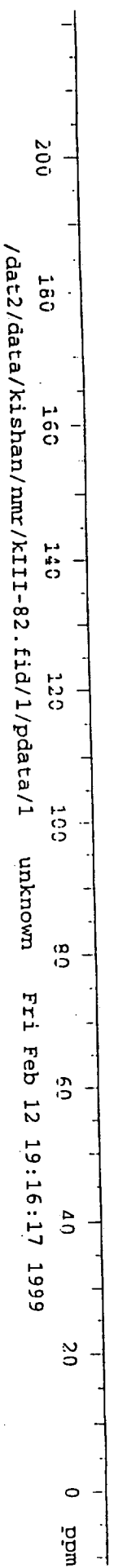
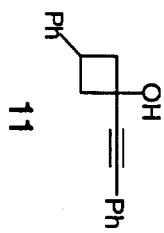


- 3.514
- 3.482
- 3.454
- 3.422
- 3.393
- 3.070
- 3.063
- 3.055
- 3.040
- 3.034
- 3.032
- 3.028
- 3.015
- 3.008
- 3.005
- 3.000
- 2.884
- 2.855
- 2.594
- 2.587
- 2.580
- 2.558
- 2.556
- 2.530
- 2.523
- 2.514

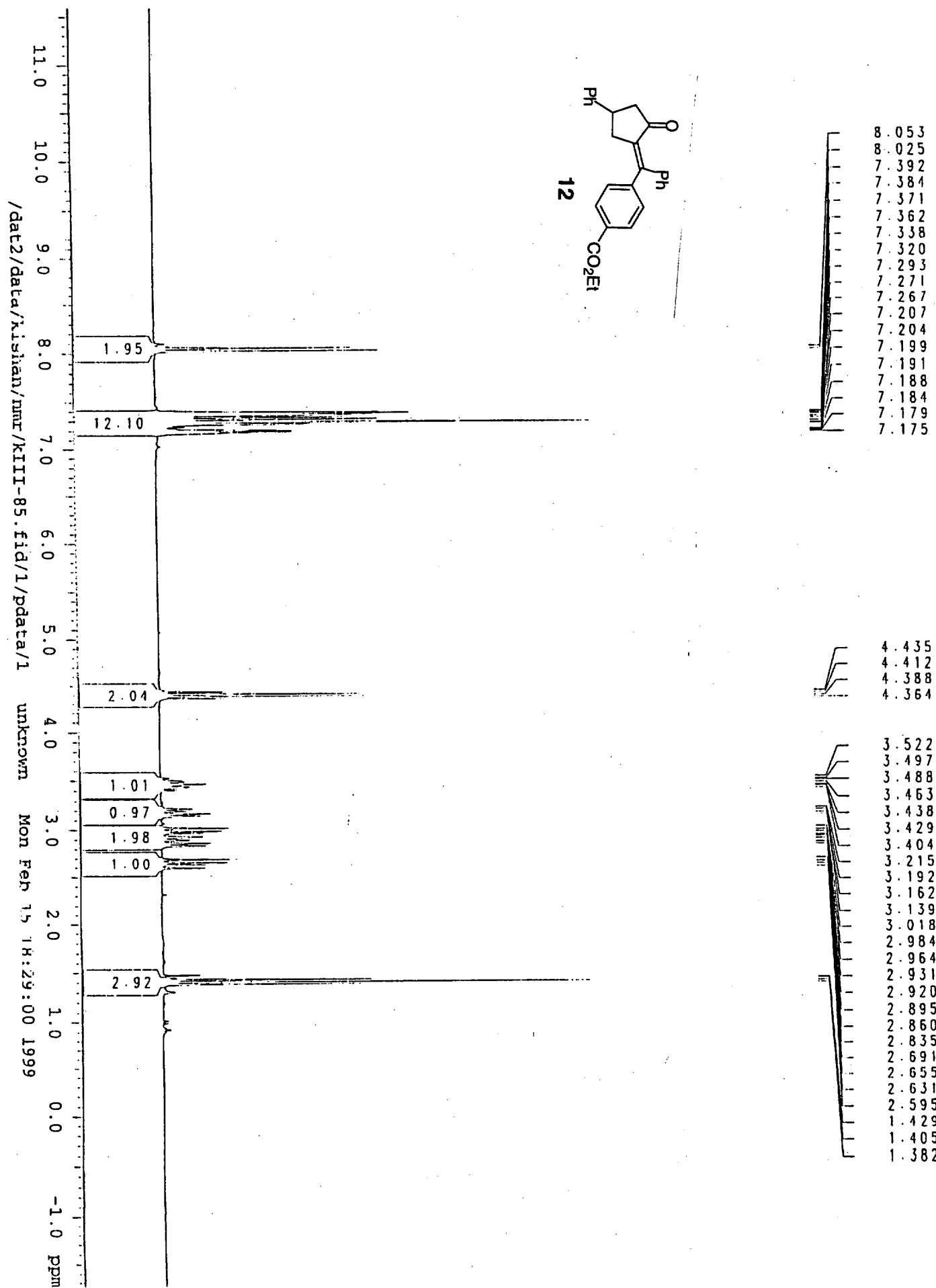
CKR - I I I - 82

/dat2/data/kishan/nmr/kiII-82.fid/1/pdata/1 unknown Fri Feb 12 19:08:48 1999

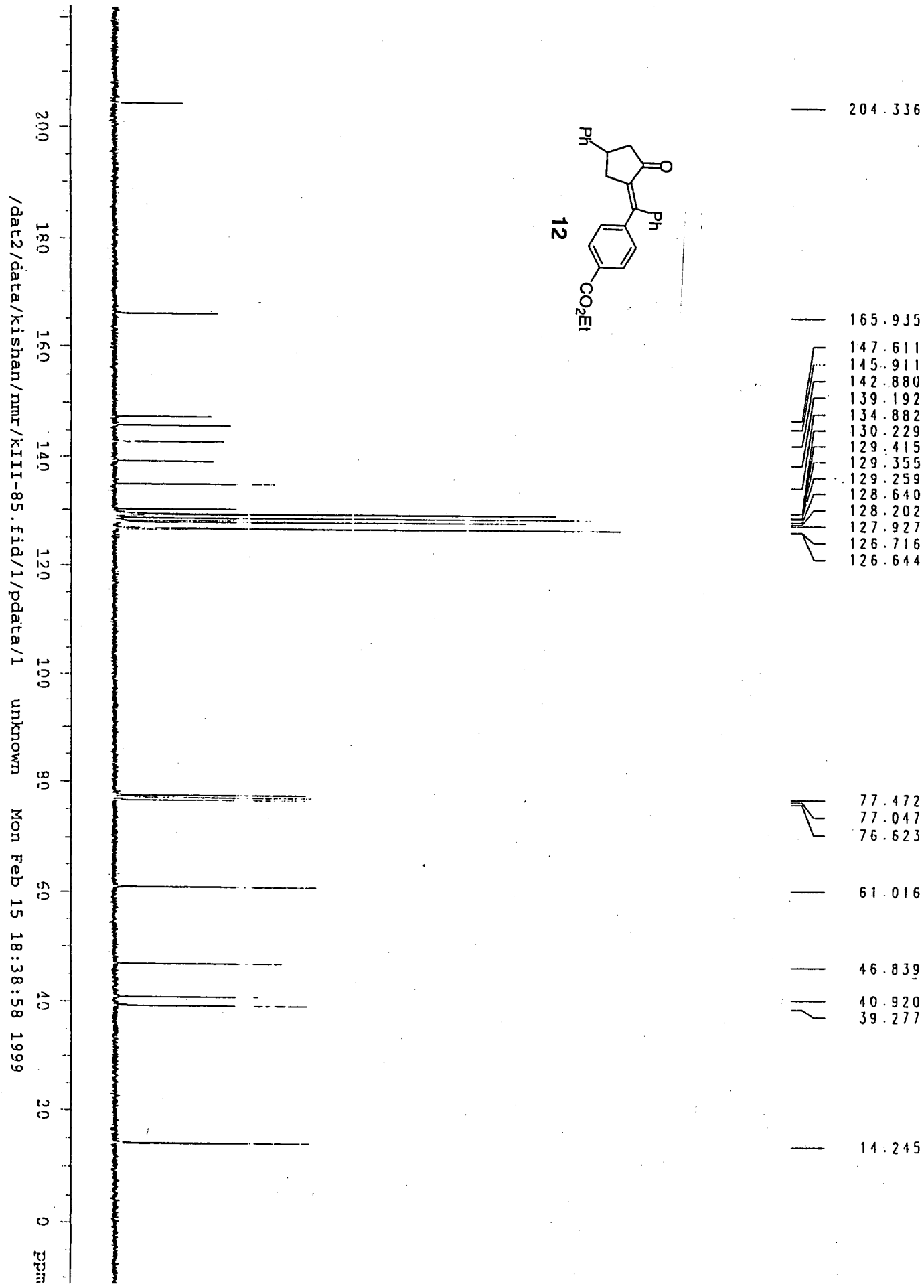
CKR-111-82



—	144.019
—	131.754
—	128.427
—	128.381
—	128.310
—	126.651
—	126.213
—	122.559
—	92.478
—	83.553
—	77.471
—	77.047
—	76.623
—	64.374
—	46.278
—	30.539

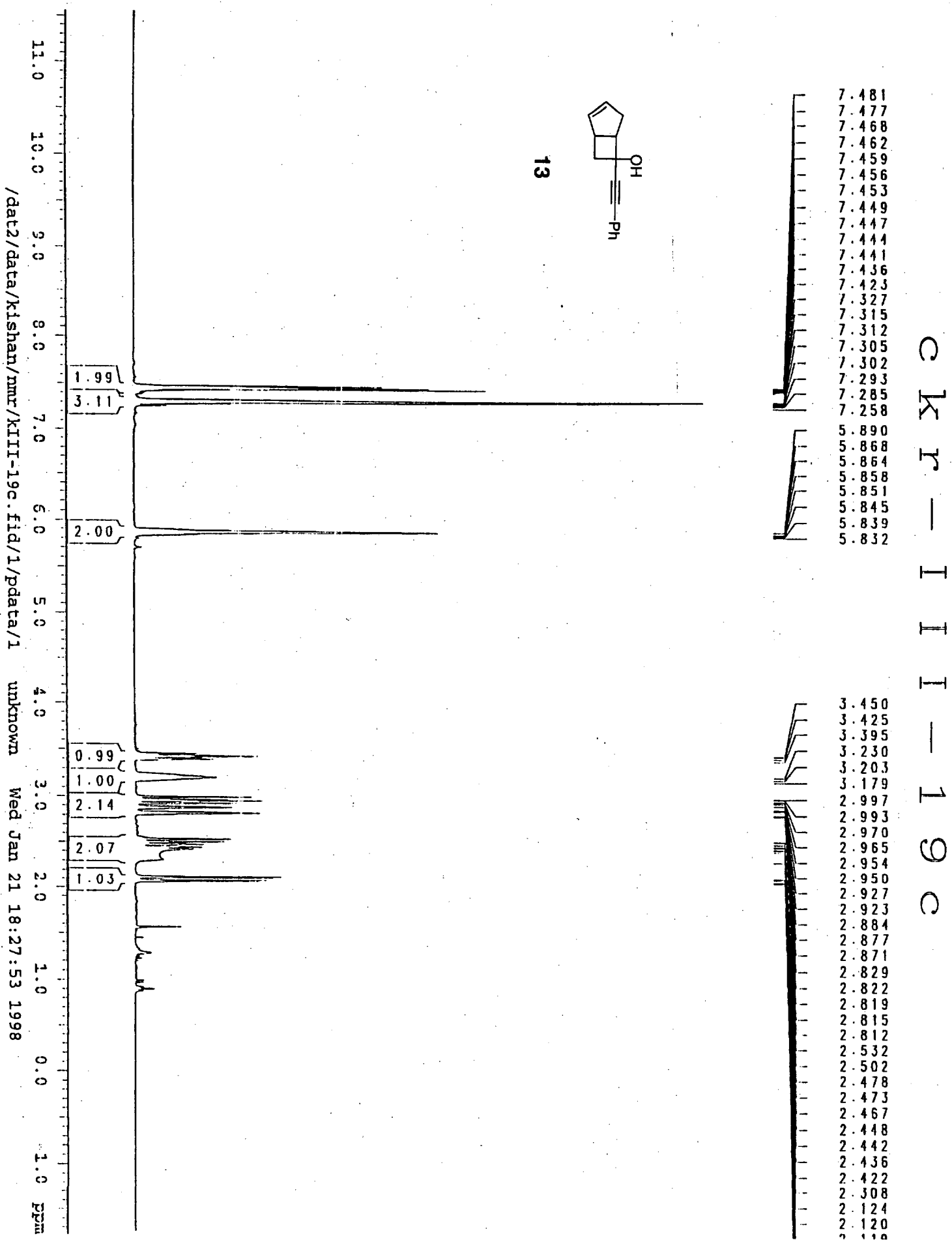


CKR - I I I - 85

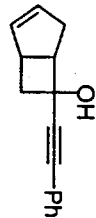


C K r - I I I - 8 5

/dat2/data/kishan/nmr/k111-85.fid/1/pdata/1 unknown Mon Feb 15 18:38:58 1999



C K R - I I I - 1 9 C



13

134.443
132.524
131.542
128.172
122.715
93.382
83.562
77.490
77.064
76.637
67.567
48.808
45.214
39.287
32.491

