Cyclic Alkenenitriles: Chemoselective Oxonitrile Cyclizations

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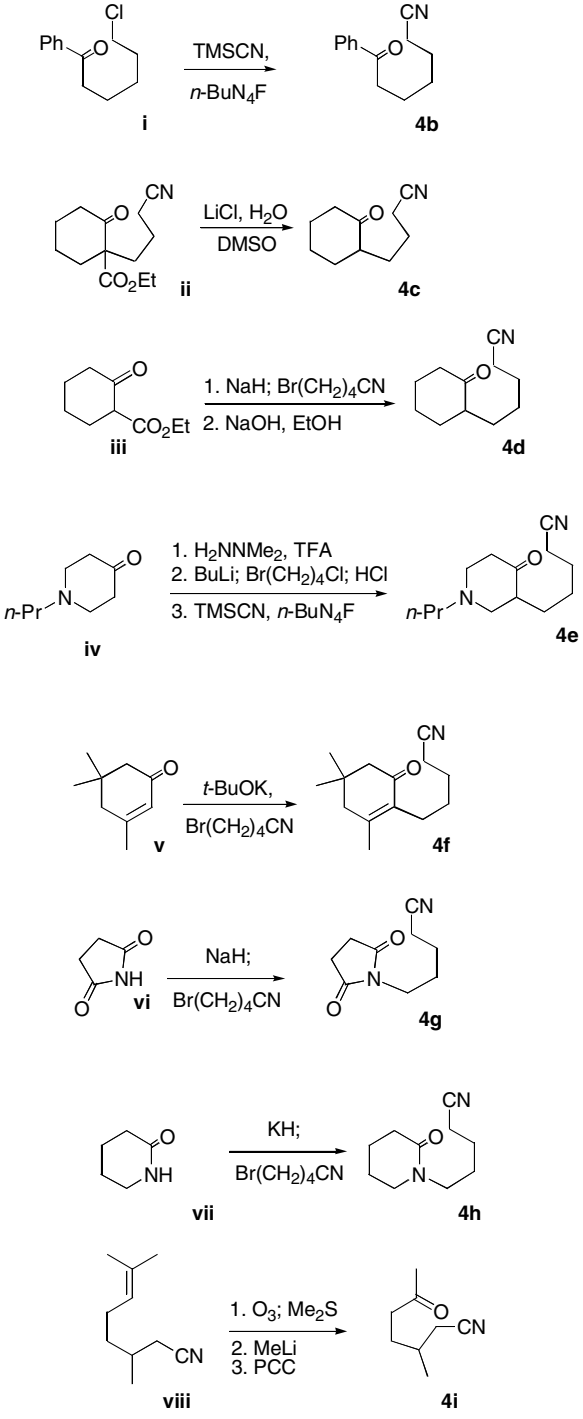
The cyclization precursors **4b – h** were prepared as outlined in the following scheme:



Preparation of Cyclization Precursors

7-Oxo-7-phenylheptanenitrile (4b): A THF solution of *n*-Bu_4NF (0.72 mmol)**,** and neat trimethylsilyl cyanide[1] (0.72 mmol), were sequentially added to an acetonitrile solution of 6-chloro-1-phenylhexan-1-one[2] (100.7 mg, 0.48 mmol). After 16 h the reaction was diluted with water and CH_2Cl_2, the organic phase was separated, washed with water (3x), and then dried (Na_2SO_4). The resulting solution was concentrated under reduced pressure and then purified by radial chromatography (1:99 EtOAc/hexanes, 1 mm plate) to afford 65.2 mg (68%) of **4b** as a white solid that was spectrally identical to material previously synthesized.[3]

4-(2-Oxocyclohexyl)butanenitrile (4c): A DMSO (5 mL) solution of ethyl 1-(3-cyanopropyl)-2-oxocyclohexanecarboxylate (**ii**)[4] (1.00 g, 2.88 mmol), H_2O (62mL, 3.5 mmol), and LiCl (147.0 mg, 3.5 mmol) was refluxed for 6h. After cooling aqueous, saturated NaCl was added and the mixture extracted with EtOAc. The aqueous phase was extracted with EtOAc (3x), and the organic extracts were combined. The combined extracts were dried (Na_2SO_4) and concentrated to afford 450 mg (65%) of pure **4c**[5] as an oil: IR (film) 2245, 1710 cm^{-1}; ^1H NMR 1.25-2.60 (m, 15H); ^{13}C NMR 14.0, 16.7, 22.4, 23.4, 25.6, 27.4, 33.7, 36.0, 40.9, 60.4, 61.2, 119.3, 171.7, 208.0.

5-(2-Oxocyclohexyl)pentanenitrile (4d): Neat ethyl 2-oxocyclohexanecarboxylate (400.0 mg, 2.35 mmol) was added drop wise to a 0 C, stirred THF (10 mL) slurry of NaH (67.8 mg, 2.83 mmol). Neat bromovaleronitrile (494.2 mg, 3.05 mmol) and solid NaI (70.0 mg, 0.467 mmol) were added and then the reaction was heated at reflux. After 24 h the reaction was allowed to cool, saturated, aqueous NH_4Cl was added, the organic phase separated, and the aqueous phase

was extracted with EtOAc. The combined organic extracts were dried (Na_2SO_4), concentrated under reduced pressure, and then purified by radial chromatography (3:10 EtOAc/hexanes, 4 mm plate) to afford 439.7 mg (74%) of ethyl 1-(4-cyanobutyl)-2-oxocyclohexanecarboxylate as an oil: IR (film) 2242, 1712 cm^{-1}; ^1H NMR 1.28 (t, *J*=7.1 Hz, 3H), 1.31-2.07 (m, 10H), 2.34 (t, *J*=7 Hz, 2H); 2.42-2.54 (m, 4H), 4.22 (q, *J*=7.1 Hz, 2H); ^{13}C NMR 14.0, 16.7, 22.4, 23.4, 25.6, 27.4, 33.7, 36.1, 60.4, 61.2, 119.3, 171.7, 207.6. Aqueous NaOH (2M, 7 mL) was added **t**o an ethanolic solution (7 mL) of ethyl 1-(4-cyanobutyl)-2-oxocyclohexanecarboxylate (741.2 mg, 2.95 mmol) and after 60 h saturated, aqueous NaCl was added followed by 1M HCl until a pH of 2. The organic phase was separated, the aqueous phase was extracted with EtOAc (3x), and the organic extracts were combined. The combined extracts were dried (Na_2SO_4), concentrated under reduced pressure, and then purified by radial chromatography (1:5 EtOAc/hexanes, 4 mm plate) to afford 399.6 mg (76%) of **4d**[6] as an oil: IR (film) 2245, 1709 cm^{-1}; ^1H NMR 1.15-1.48 (m, 3H), 1.43 (pent, *J* = 8 Hz, 2H), 1.61-1.88 (m, 6H), 1.93-2.13 (m, 2H), 2.21-2.40 (m, 2H), 2.34 (t, *J* = 7 Hz, 2H); ^{13}C NMR 17.0, 25.0, 25.5, 26.3, 28.0, 26.3, 28.0, 28.5, 34.0, 42.1, 50.4, 119.7, 212.8 MS *m/e* 180 (M + H).

5-(4-Oxo-1-propylpiperidin-3-yl)pentanenitrile (4e): A benzene solution (50 mL) of 1-propyl-4-piperidone (3.67 g, 26.0 mmol), N,N-dimethlyhydrazine (2.37 mL, 31.2 mmol), and trifluoroacetic acid (0.5 mL) was heated at reflux using a Dean Stark apparatus. After 5 h the mixture was cooled to room temperature, and then diluted with ether and water. The organic phase was separated, washed with brine, dried (Na_2SO_4), concentrated under reduced pressure, and distilled to afford 4.29 g (90%) of 1-propylpiperidin-4-one dimethylhydrazone as an oil: IR (film) 1642; ^1H NMR 0.87 (t, *J* = 7 Hz, 3H), 1.48 (sext, *J* = 7 Hz, 2H), 2.26-2.36 (m, 4H), 2.40

(s, 6H), 2.45-2.49 (m, 2H), 2.54 (t, J = 6 Hz, 2H), 2.62 (t, J = 5.8 Hz, 2H); ^{13}C NMR 11.7, 20.1, 28.3, 34.8, 47.2, 52.9, 53.8, 59.8, 166.8; MS *m/e* 184 (M + 1). A hexane solution of *n*-BuLi (9.26 mmol) was added to a cold (0^{o}C) THF solution of 1-propylpiperidin-4-one dimethylhydrazone (1.54 g, 8.42 mmol). After 1h, a THF solution of 1-bromo-4-chlorobutane (1.59 g, 9.26 mmol) was added followed, after 16 h, by hydrolysis with 2 M HCl (23.2 mL, 46.3 mmol) for 1 h. The organic phase was then separated and the aqueous phase extracted with CH_2Cl_2. The combined organic extracts were then sequentially washed with water and brine, and then dried (Na_2SO_4). The dry solution was concentrated under reduced pressure and the crude product was purified by radial chromatography (1:9 EtOAc/hexane) to provide 1.15 g (59%) of 3-(4-Chlorobutyl)-1-propylpiperidin-4-one as an oil: IR (film) 1714 cm^{-1}; ^{1}H NMR 0.94 (t, J = 7 Hz, 3H), 1.17-1.29 (m, 2H), 1.44 (pent, J = 8 Hz, 2H), 1.55 (sext, J = 7 Hz, 2H), 1.67-1.88 (m, 2H), 2.15 (t, J = 11 Hz, 1H), 2.34-2.63 (m, 6H), 3.05-3.10 (m, 2H), 3.53 (t, J = 6.6 Hz, 2H); ^{13}C NMR 11.8, 20.6, 24.4, 26.5, 32.6, 41.0, 44.7, 49.5, 53.8, 59.1, 59.3, 210.6; MS *m/e* 232 (M + 1). 3-(4-Chlorobutyl)-1-propylpiperidin-4-one was converted to the corresponding nitrile as described for **4b** with 3-(4-chlorobutyl)-1-propylpiperidin-4-one (412.8 mg, 1.78 mmol) to afford after purification (1:5 EtOAc/hexanes, 4 mm plate) 254.0 mg (64%) of **4e** as an oil: IR (film) 2246, 1716 cm^{-1}; ^{1}H NMR 0.93 (t, J = 7 Hz, 3H), 1.17-1.29 (m, 2H), 1.34-1.88 (m, 6H), 2.14 (t, J = 10.9 Hz, 2H), 2.20-2.63 (m, 7H), 3.03-3.10 (m, 1H), 3.07 (dd, J = 11, 5 Hz, 1H); ^{13}C NMR 11.8, 17.0, 20.6, 25.5, 26.3, 26.5, 41.0, 49.4, 53.8, 59.2, 59.3, 119.5, 210.5; MS *m/e* 223 (M + 1).

5-(2,4,4-Trimethyl-6-oxocyclohex-1-en-1-yl)pentanenitrile (4f): Solid *t*-BuOK (115.3 mg, 1.03 mmol) was added to a rt, THF solution (10 mL) of isophorone (95.0 mg, 0.688 mmol)

followed, after 30 min, by the slow addition of a THF solution (5 mL) of iodovaleronitrile (171.8 mg, 0.822 mmol). After 12 h, saturated, aqueous NH_4Cl was added, the organic phase was separated, and the aqueous phase was extracted with CH_2Cl_2. The combined organic extracts were dried (Na_2SO_4), concentrated under reduced pressure, and then purified by radial chromatography (1:5 EtOAc/hexanes, 2mm plate) to afford 49.0 mg (77% based on recovered starting material) of **4f** as an oil: IR (film) 2245, 1661, 1632 cm^{-1}; ^1H NMR 0.99 (s, 6H), 1.40-1.51 (m, 2H), 1.64 (pent, *J* = 7 Hz, 2H), 1.92 (s, 3H), 2.22 (s, 4H), 2.31 (t, *J* = 7 Hz, 2H), 2.36 (t, *J* = 7 Hz, 2H); ^{13}C NMR 16.8, 21.2, 23.8, 25.2, 27.9, 28.1, 32.6, 46.9, 51.2, 119.7, 133.6, 153.2, 198.8; MS *m/e* 220 (M + H).

5-(2,5-Dioxopyrrolidin-1-yl)pentanenitrile (4g): Solid NaH (57.6 mg of a 60% dispersion in mineral oil, 1.44 mmol) was added to a rt, THF solution (10 mL) of succinimide (119.6 mg, 1.21 mmol) and, after 1 h, a THF solution (5 mL) of iodovaleronitrile (302.0 mg, 1.45 mmol) was slowly added. After 16 h saturated, aqueous $NaHCO_3$ was added and the aqueous phase extracted with CH_2Cl_2. The combined organic extracts were dried (Na_2SO_4), concentrated under reduced pressure, and purified by radial chromatography (1:9 EtOAc/Hexanes, 2 mm plate) to afford 111.8 mg of **4g** (51%) as an oil: IR (film) 2246, 1774, 1698 cm^{-1}; ^1H NMR 1.60-1.80 (m, 4H), 2.39 (t, *J* = 7 Hz, 2H), 2.71 (s, 4H), 3.54 (t, *J* = 7 Hz, 2H); ^{13}C NMR 16.6 22.7 26.7 28.1 37.5 119.1 177.1 MS *m/e* 181 (M + H).

5-(2-Oxopiperidin-1-yl)pentanenitrile (4h): A THF solution (20 mL) of valerolactam (214.2 mg, 2.16 mmol) was slowly added to a THF washed slurry of KH (181.8 mg, 4.53 mmol). After 3 h, a THF solution (10 mL) of iodovaleronitrile (538.5 mg, 2.57 mmol) was added and after a

further 16 h saturated, aqueous $NaHCO_3$ (50 mL) was added. The organic phase was separated and then the aqueous phase was extracted with CH_2Cl_2. The combined organic extracts were dried (Na_2SO_4), concentrated under reduced pressure, and then purified by radial chromatography (1:5 EtOAc/hexanes, 4 mm plate) to afford 229.3 mg (59%) of **4h** as an oil: IR (film) 2246, 1666 cm^{-1}; ^1H NMR 1.57-1.83 (m, 8H), 2.32-2.36 (m, 2H), 2.40 (t, *J* = 7 Hz, 2H), 3.22-3.26 (m, 2H), 3.38 (t, *J* = 7 Hz, 2H); ^{13}C NMR 16.7, 21.3, 22.5, 23.2, 25.8, 32.2, 45.5, 47.7, 119.5, 169.8; MS *m/e* 181 (M + H).

3-Methyl-6-oxoheptanenitrile (4i): Ozonolysis of a dichloromethane solution (60 mL) of **vii**[7] (1.47 g, 9.72 mmol), terminating the ozonolysis immediately upon observing the distinctive blue color of ozone was followed by addition of neat dimethylsulfide (2 mL) at -78 °C. The resultant mixture was allowed to warm to room temperature, stirred for 20 h, and then the solvent was removed under reduced pressure. Chromatography of the crude product (4 mm silica gel plate, 2:8 EtOAc:hexanes) and concentration of the appropriate fractions under reduced pressure gave 892 mg (73%) of 3-methyl-6-oxo-hexanenitrile as an oil: IR (film) 2246, 1726 cm^{-1}; ^1H NMR 1.09 (d, *J* = 6.6 Hz, 3H), 1.58-1.90 (m, 3H), 2.32 (br t, *J* = 6 Hz, 2H), 2.51 (br t, *J* = 7 Hz, 2H), 9.78 (br t, *J* = 1 Hz, 1 H); ^{13}C NMR 19.1, 24.3, 27.8, 29.9, 41.1, 118.6, 201.8; GC/MS m/e 126 (MH). An ethereal solution of methyllithium (6.82 mL, 1.5 M) was added to a stirred THF solution (35 mL) of 3-methyl-6-oxo-hexanenitrile (853 mg, 6.82 mmol). After 4 h aqueous, saturated ammonium chloride was added. The crude reaction mixture was extracted with EtOAc, washed with brine, dried over magnesium sulfate, and evaporated under reduced pressure. Radial chromatography of the crude product (4 mm silica gel plate, 3:7 EtOAc:hexanes) gave 189 mg (22%) of the starting aldehyde and 552 mg (57%) of 3-methyl-6-hydroxyheptanenitrile as an oil: IR (film) 3452, 2965, 2243 cm^{-1}; 1H NMR : 1.02-1.45 (m, 9H), 1.81-1.93 (m, 2 H), 2.09-2.35 (m, 2H), 3.74 (br sextet, *J* = 6 Hz, 1H); ^{13}C NMR : 19.4, 23.7, 24.5, 30.6, 32.0, 36.4, 68.0,

118.7; GC/MS m/e 142 (MH). Pyridinium chlorochromate (1.04 g, 4.8 mmol) was added to a stirred, dichloromethane solution (23 mL) of 3-methyl-6-hydroxyheptane nitrile (454 mg, 3.2 mmol). After 12 h diethylether (5 mL) was added, the heterogenous mixture was sonicated (2 min) and was then passed through a plug of Florisil (elution with diethylether). Removal of the solvent under reduced pressure followed by radial chromatography (4 mm silica gel plate, 2:8 EtOAc:hexanes) afforded 370 mg (83%) of **4i** as a colorless oil. IR (film): 2970, 2245, 1715 cm^{-1}; ^1H NMR 1.07 (d, J = 6.7 Hz, 3H), 1.57-1.63 (m, 1H), 1.73 (ddd, J = 20, 14, 7 Hz, 1H), 1.87 (ddd, J = 20, 14, 7 Hz, 1H) 2.16 (s, 3H), 2.25 (br dd, J = 17, 7 Hz, 1H), 2.33 (dd, J = 17, 6 Hz, 1H), 2.48 (br t, J = 7 Hz, 2H); ^{13}C NMR 19.2, 24.5, 29.5, 29.9, 30.4, 40.7, 118.4, 207.6; GC/MS: m/e 140 (MH).

General counter-intuitive cyclization procedure

Solid *t*-BuOK (5 equiv) was added to a refluxing, THF solution of the ketonitrile (1 equiv, 0.01-0.05 M). After 5 h the solution was cooled, saturated, aqueous, NH_4Cl was added, the organic phase was separated, and the aqueous phase was extracted with CH_2Cl_2 (3x). The combined organic extracts were dried (Na_2SO_4), concentrated under reduced pressure, and then purified by radial chromatography (EtOAc/Hexanes).

5-Isopropenyl-2-methylcyclopent-1-ene-1-carbonitrile (7a): The general procedure was employed with **4a**[8] (108.5 mg, 0.657 mmol), except that the solution was stirred at room temperature rather than at reflux, followed, after 5 h, by addition of aqueous hydrochloric acid (2%) to afford, after chromatography (1:9 EtOAc/hexanes, 1 mm plate), 57.9 mg (60%) of **7a** as an oil: IR (film) 3078, 2211, 1728, 1638 cm^{-1}; ^1H NMR 1.69 (s, 3H), 1.72-1.84 (m, 1H), 2.03 (s, 3H), 2.10-2.26 (m, 1H), 2.43-2.51 (m, 2H), 3.49-3.56 (m, 1H), 4.82 (d, J = 2.7 Hz, 2H); ^{13}C

NMR 16.7, 19.3, 29.0, 37.5, 54.4, 112.0 (resolves to 113.1 and 113.6 in CS_2), 116.5, 144.8, 161.8; MS *m/e* 148 (M + H). Repetition of the reaction at reflux generates a 1:1 mixture of **7a** and the corresponding amide 5-isopropenyl-2-methylcyclopent-1-ene-1-carboxamide: IR (film) 3377, 3289, 3181, 3082, 1634 cm^{-1}; ^1H NMR 1.74 (s, 3H), 2.05 (s, 3H), 2.46-2.55 (m, 5H), 2.76 (dd, *J* = 13, 8 Hz, 1H), 2.88 (pent, *J* = 8 Hz, 1H), 4.73-4.74 (m, 2H); ^{13}C NMR 20.5, 28.1, 35.7, 39.0, 42.7, 105.2, 109.5, 147.1, 162.2; MS *m/e* 166 (M + H).

2-Phenylcyclohex-1-ene-1-carbonitrile (7b): The general procedure was employed with **4b** (20.3 mg**,** 0.101 mmol), using 1eq of *t*-BuOK (11.0 mg, 0.100 mmol) to provide, after chromatography (1:19 EtOAc/hexanes, 1 mm plate), 12.3 mg (66%) of **7b** as an oil: IR (Film) 3057, 2207 cm^{-1}; ^1H NMR 1.76-1.81 (m, 4H), 2.40–2.49 (m, 4H), 7.38 (s, 5H); ^{13}C NMR 21.3, 21.8, 28.3, 31.3, 107.9, 119.7, 127.1, 128.5, 128.8, 140.1, 155.4; MS *m/e* 184 (M + H).

2,4,5,6,7,7a-Hexahydro-1H-indene-3-carbonitrile (7c): The general procedure was employed with **4c** (82.0 mg, 0.496 mmol) to afford, after chromatography (1:9 EtOAc/hexanes, 1 mm plate), 57.4 mg (79%) of **7c** as an oil: IR (film); 2212, 1644 cm^{-1}; ^1H NMR 1.02 (qd, *J* = 12, 3 Hz, 1H), 1.15-1.67 (m, 3H), 1.73-2.02 (m, 4H), 2.11-2.22 (m, 1H), 2.49-2.54 (m, 3H), 2.79 (d, *J* = 12.9 Hz, 1H); ^{13}C NMR 25.0, 26.2, 28.3, 29.6, 32.6, 34.9, 46.6, 104.1, 116.8, 166.0.

2,3,4,4a,5,6,7,8-Octahydronaphthalene-1-carbonitrile (7d): The general procedure was employed with **4d** (56.3 mg, 0.315 mmol) to afford, after chromatography (1:9 EtOAc/hexanes, 1 mm plate), 33.8 mg (67%) of **7d:** IR (film); 2930, 2856, 2207, 1634 cm^{-1}; ^1H NMR 1.11-2.03

(m, 12H), 2.18-2.23 (m, 2H), 2.91-2.96 (m, 1H); ^{13}C NMR 28.6, 31.0, 35.1, 35.6, 38.0, 42.2, 43.2, 46.6, 112.4, 127.4, 167.8. MS *m/e* 161 (M). Prematurely terminating the reaction affords intermediate alcohol **6d** as a crystalline solid (mp 82-83 °C), tentatively assigned the *trans*-decalin stereochemistry: IR (film); 3546, 2236 cm^{-1}; ^{1}H NMR 1.16-2.06 (m, 16H), 2.37 (dd, *J*=12, 4.5 Hz, 1H); ^{13}C NMR 21.3, 24.8, 25.7, 25.9, 27.3, 28.3, 37.6, 41.3, 43.7, 69.9, 120.5.

2-Propyl-1,2,3,4,6,7,8,8a-octahydroisoquinoline-5-carbonitrile (7e): The general procedure was employed with **4e** (83.2 mg, 0.374 mmol), with the modification that the solution was heated at reflux for 3h, rather than 5 h, followed by addition of $NaHCO_3$ rather than NH_4Cl, to afford,, after chromatography (1:5 EtOAc/hexanes, 1 mm plate), 46.9 mg (61%) of **7e** as an oil: IR (Film) 2209, 1639 cm^{-1}; ^{1}H NMR 0.90 (t, *J* = 7 Hz, 3H), 1.13-1.22 (m, 1H), 1.51 (sext, *J* = 7 Hz, 2H), 1.65 (t, *J* = 11 Hz, 2H), 1.72-1.83 (m, 2H), 1.90-2.03 (m, 1H), 2.15-2.42 (m, 6H), 2.99 (d, *J* = 6.6 Hz, 1H), 3.02-3.06 (m, 2H); ^{13}C NMR 11.8, 20.1, 20.5, 26.4, 27.4, 32.9, 37.3, 53.7, 59.9, 60.4, 105.0, 118.6, 156.3; MS *m/e* 205 (M + H).

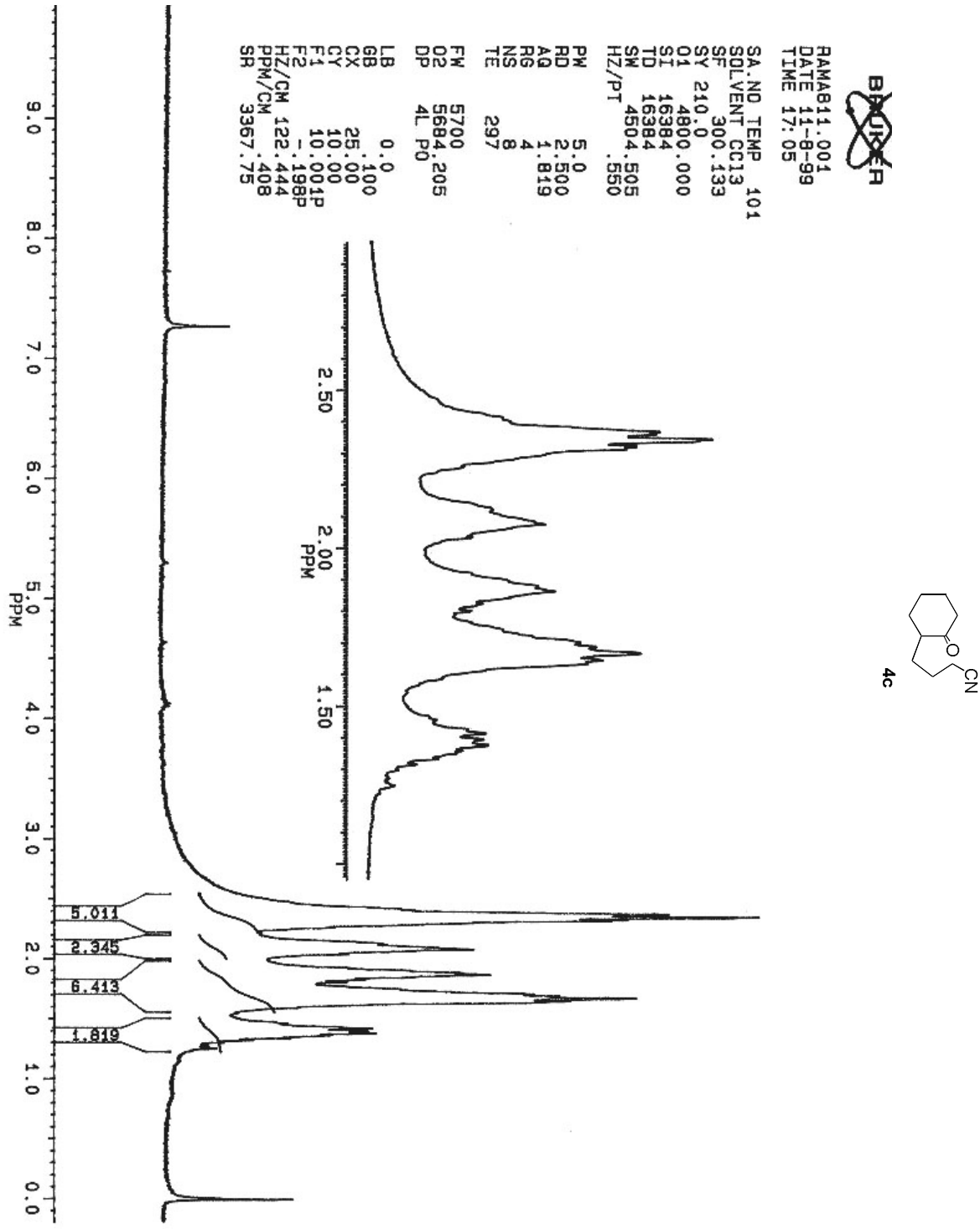
5,7,7-Trimethyl-2,3,4,6,7,8-hexahydronaphthalene-1-carbonitrile (7f): The general procedure was employed using potassium *iso*-butoxide, prepared by adding neat *iso*-butanol (35.9 mg, 0.484 mmol) to a rt, THF washed suspension (5 mL) of KH (16.4 mg, 0.409 mmol). After 15 minutes a THF solution (1 mL) of **4f** (17.6 mg, 0.0857 mmol) was slowly added, followed by refluxing, to afford, after chromatography (1:9 EtOAc/hexanes, 1 mm plate), 9.8 mg (61%) of **7f** as an oil**:** IR (film) 2199, 1624, 1590 cm^{-1}; ^1H NMR 0.92 (s, 6H), 1.68 (pent, J = 6.0 Hz, 2H), 1.75 (s, 3H), 2.01 (s, 2H), 2.29-2.34 (m, 4H), s, 2H), ^{13}C NMR 20.1, 21.9, 25.2, 27.8, 27.9, 30.6, 43.4, 47.2, 103.4, 120.4, 125.5, 137.0, 149.6; MS *m/e* 202 (M + 1).

3-Oxo-1,2,3,5,6,7-hexahydroindolizine-8-carbonitrile (7g): The general procedure was employed with **4g** (53.4 mg, 0.298 mmol) and modified by adding 10% aqueous HCl, rather than NH_4Cl. After 30 min, solid K_2CO_3 was added until the solution was neutral and then saturated, aqueous $NaHCO_3$ was added to afford, after chromatography (1:5 EtOAc/hexanes, 1 mm plate), 29.8 mg (62%) of **7g** as an oil exhibiting IR and 1H NMR spectra identical to that previously reported[9]: ^{13}C NMR 19.6, 23.1, 23.3, 28.1, 38.8, 80.9, 118.8, 154.2, 174.8.

3,4,6,7,8,9-Hexahydro-2H-quinolizine-1-carbonitrile (7h): The general procedure was employed with **4h** (122.6 mg, 0.680 mmol) to provide, after chromatography (1:5 EtOAc/hexanes, 1 mm plate), 66.4 mg (60%) of **7h** that was spectrally identical to material previously synthesized.[10] Alternative cyclization with KH: A THF solution (100 mL) of **7h** (899.1 mg, 5.00 mmol) was slowly added to a THF-washed slurry of KH (600.1 mg, 14.98 mmol) followed by heating the resulting mixture to reflux. After 5 h, the solution was cooled, saturated, aqueous, NH_4Cl was added, the organic phase was separated, and the aqueous phase was extracted with CH_2Cl_2. The combined organic extracts were dried (Na_2SO_4), concentrated

under reduced pressure, and then purified by radial chromatography (1:5 EtOAc/hexanes, 4 mm plate) to afford 511.1 mg (63%) of **8h** spectrally identical to material previously synthesized.[10]

(2, 5-Dimethyl-1-cyclopentenyl)-1-carboxamide (11i): Potassium *tert*-butoxide (1.0 M in *t*-butanol , 0.55 mmol) was added to a stirred, room temperature, *t*-BuOH solution (8 mL) of **4i** (76 mg, 0.55 mmol). After 8 h aqueous saturated ammonium chloride was added. The reaction mixture was extracted with EtOAc, the extracts were washed with brine, dried over $MgSO_4$, and then the solvent was removed under reduced pressure. Radial chromatography of the crude product (1 mm silica gel plate, 4:6 EtOAc:hexanes) afforded 41 mg (54%) of **11j** as crystals upon concentration of the appropriate fractions under reduced pressure. M.P: 65-69 °C; IR (film): 3354, 3160, 2931, 1629, 1500 cm^{-1}; ^1H NMR 1.08 (d, J = 6.7 Hz, 3H), 2.04 (s, 3H), 2.07-2.25 (m, 2H), 2.33 (br ddd, J = 21, 14, 7 Hz, 1H), 2.61 (dd, J = 16, 8 Hz, 1H), 2.77 (dd, J = 12.9, 8 Hz, 1H); ^{13}C NMR 21.1, 28.1, 30.1, 38.9, 42.7, 105.5, 162.6, 195.7; GC/MS: m/e 140 (MH).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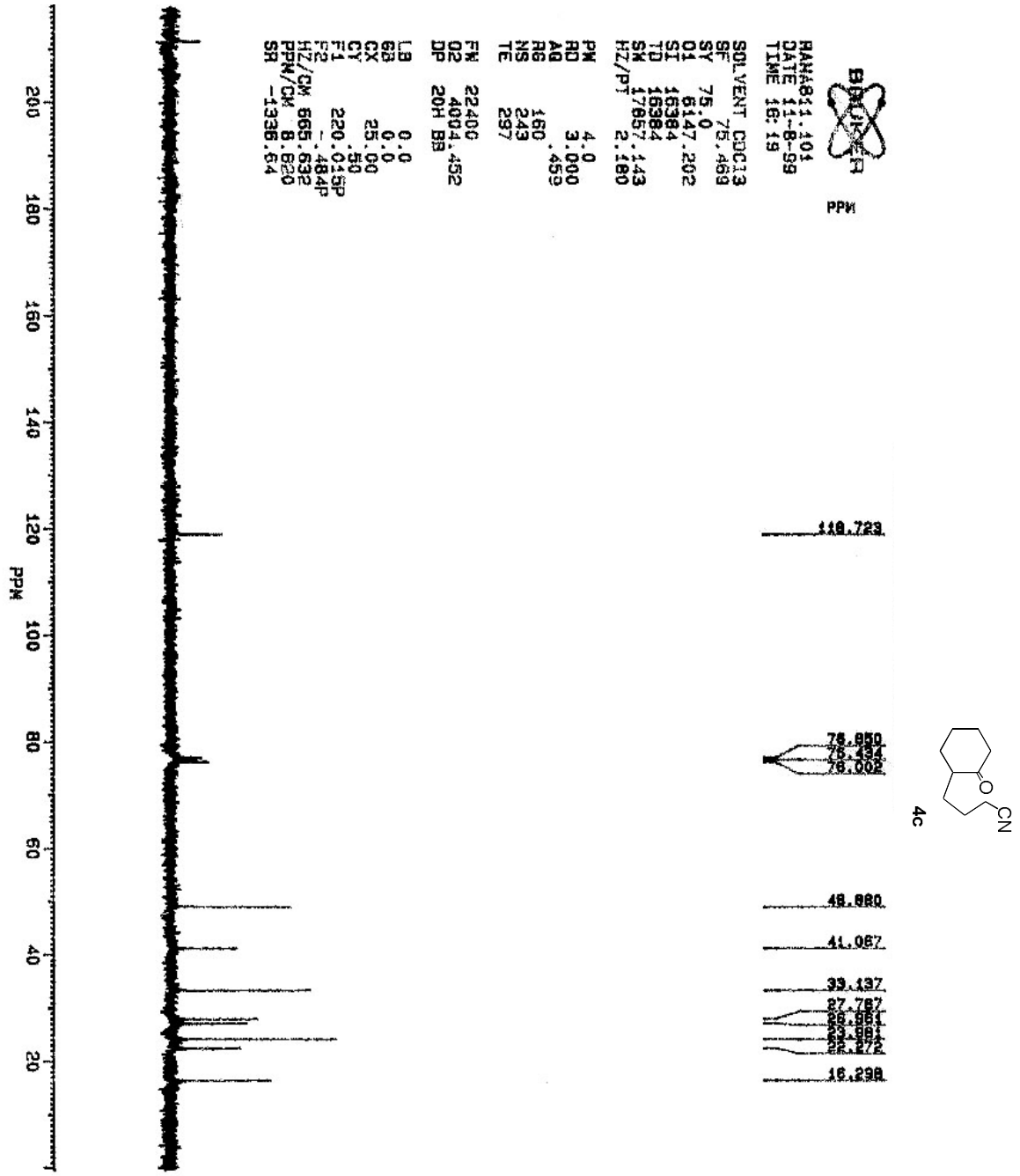
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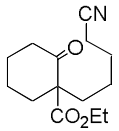
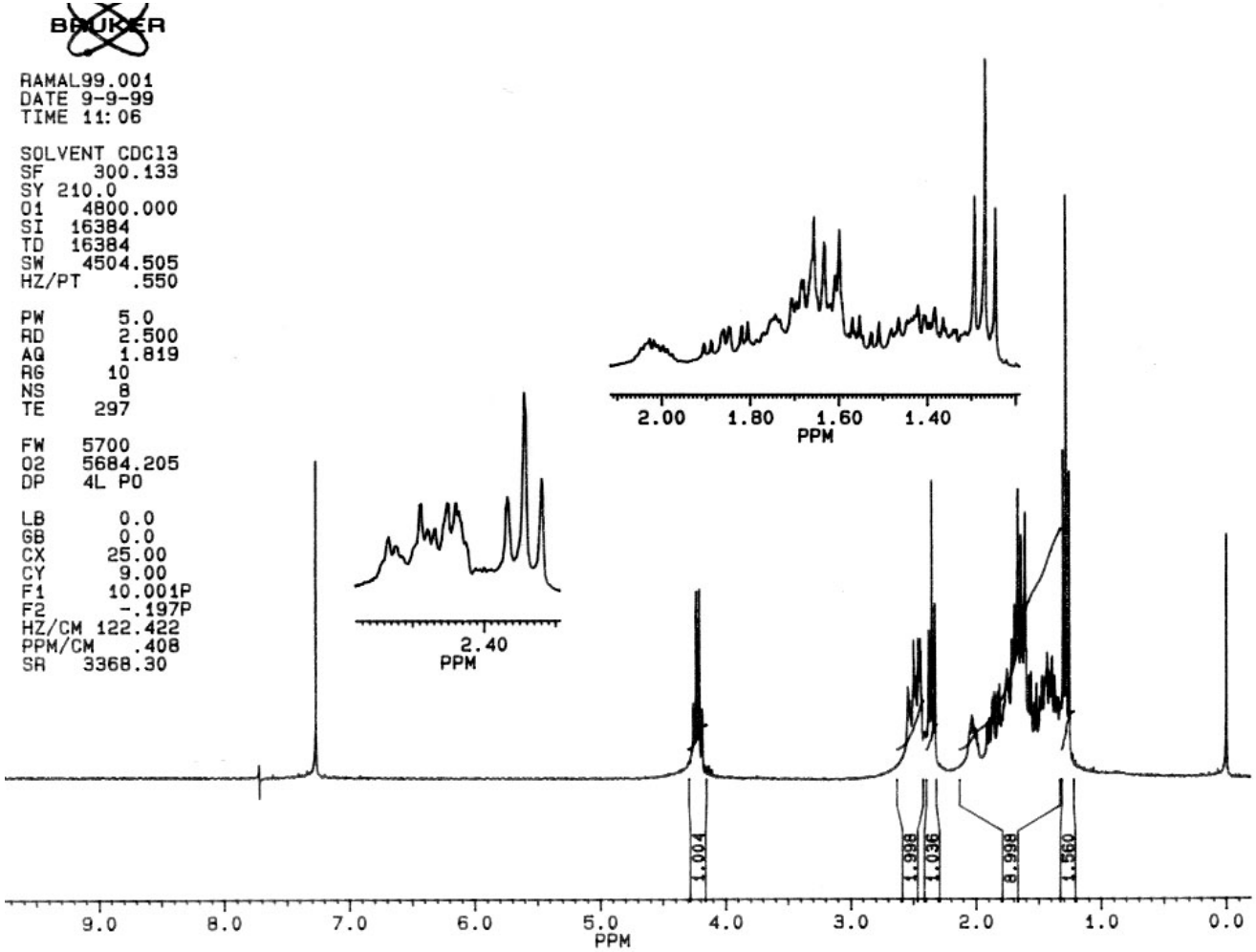
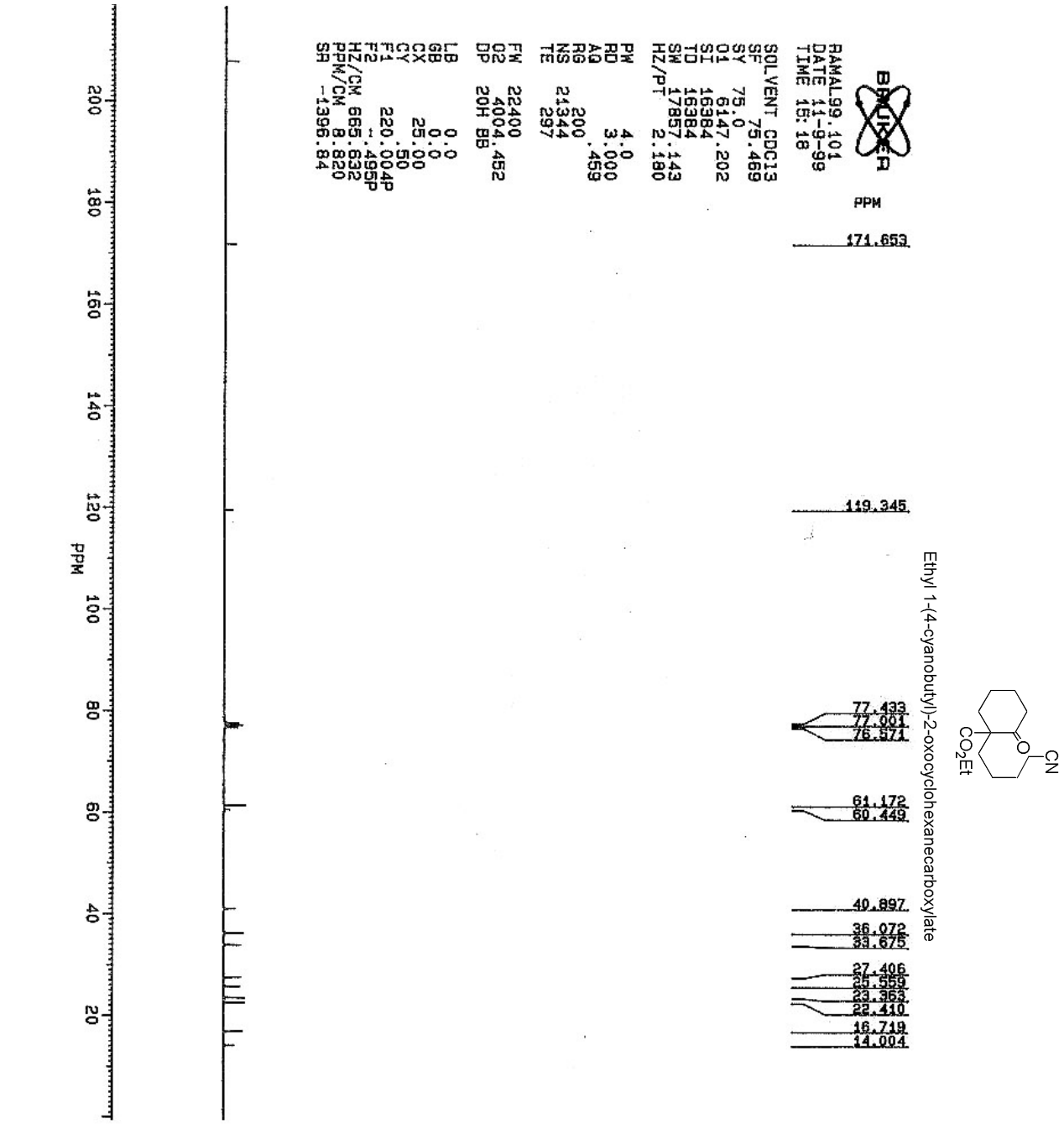
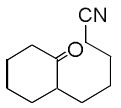


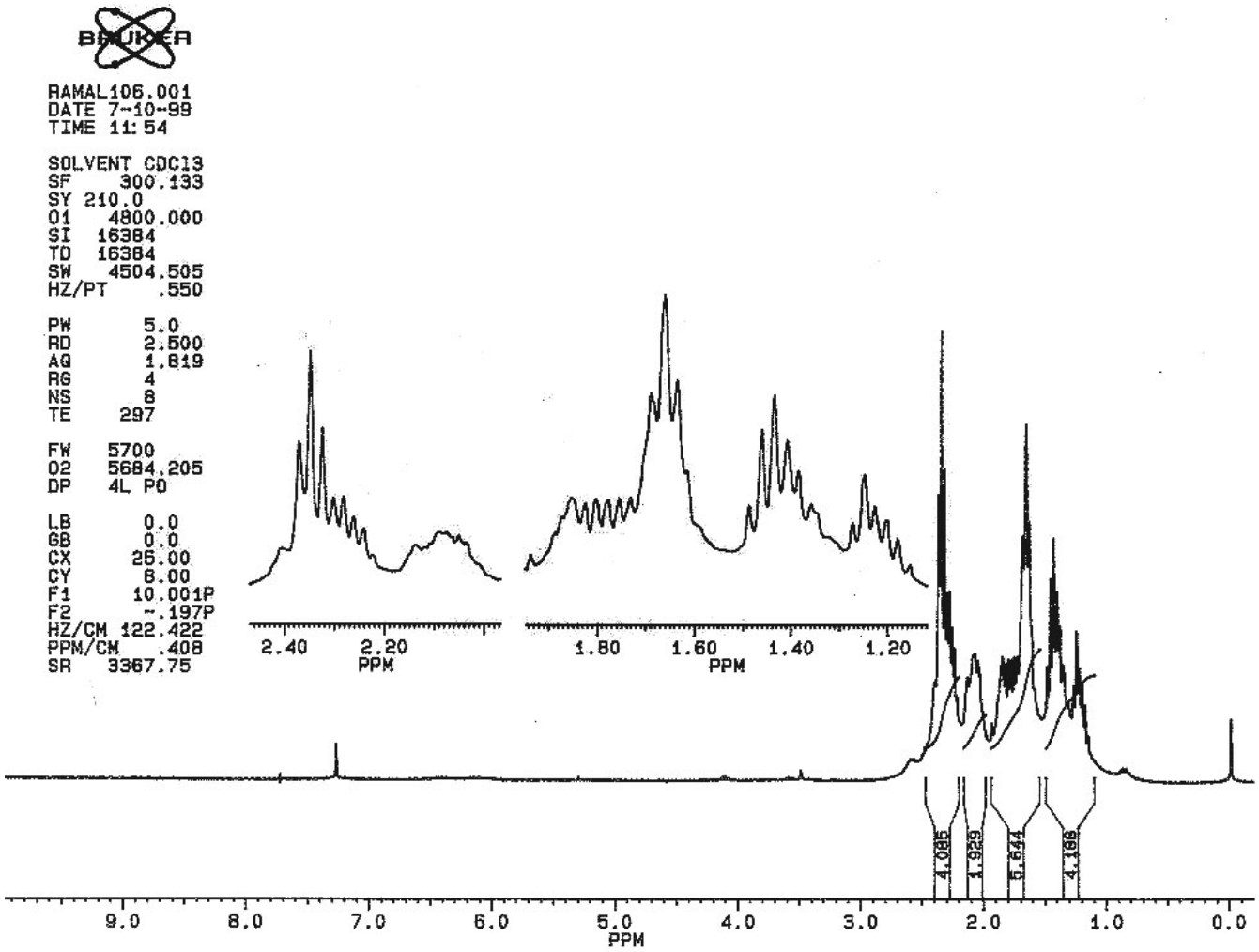
Ethyl 1-(4-cyanobutyl)-2-oxocyclohexanecarboxylate

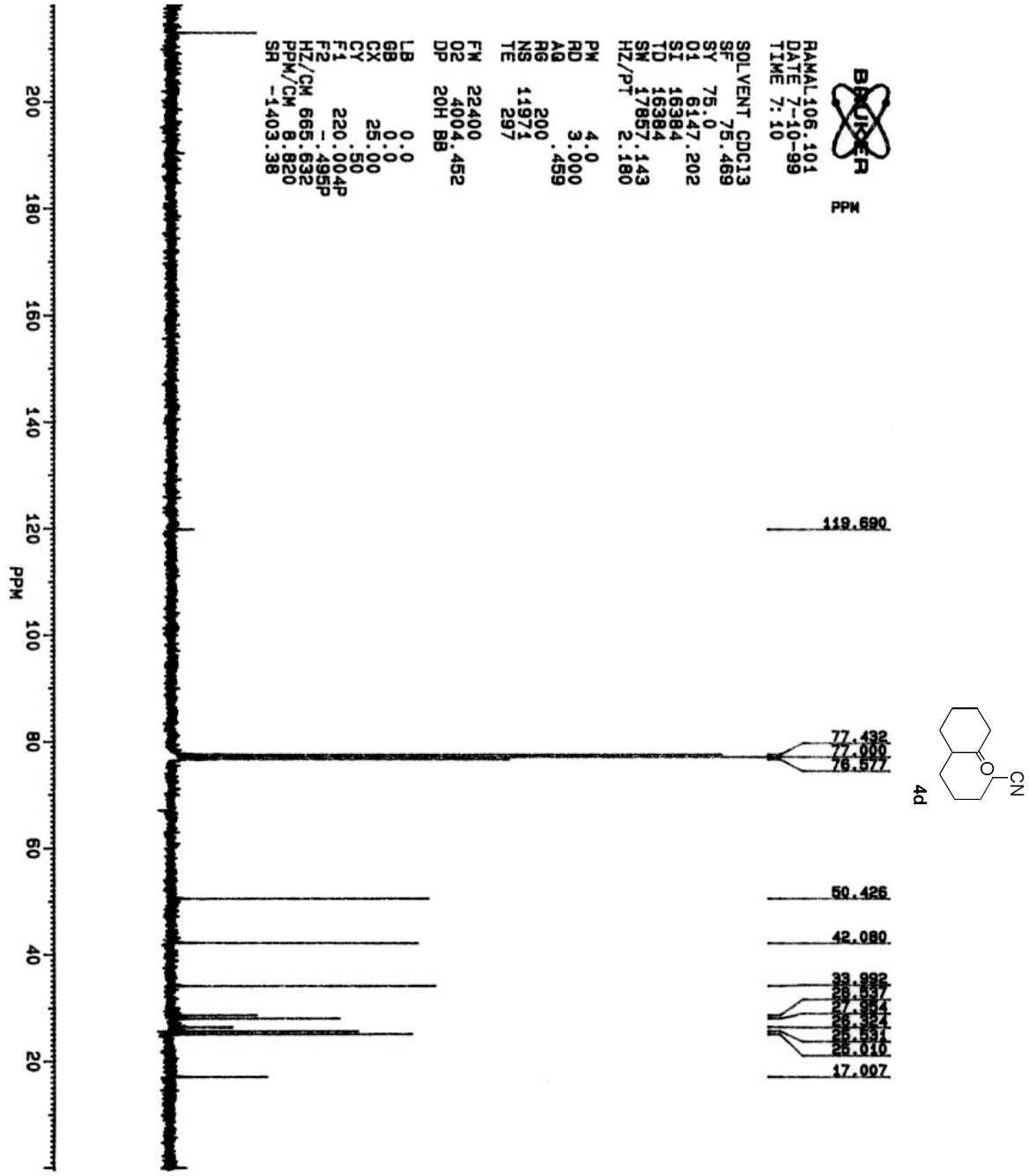


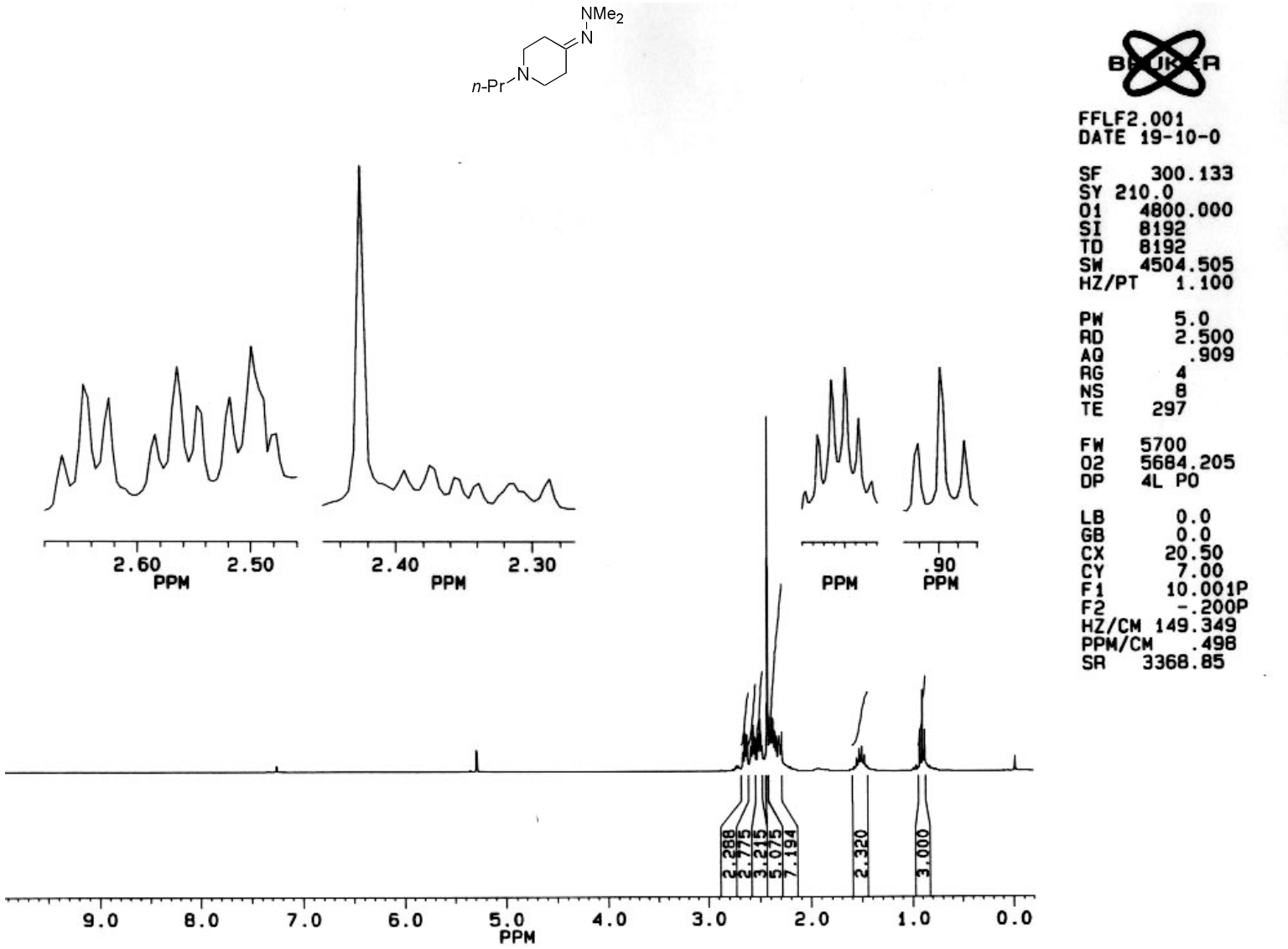

RAMAL99.101
DATE 11-9-99
TIME 15:18
SOLVENT CDCl3
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 21344
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CX 25.00
CY .50
F1 220.004P
F2 -.495P
HZ/CM 665.632
PPM/CM 8.820
SR -1396.84
PPM
171.653
119.345
77.433
77.001
76.571
61.172
60.449
40.897
36.072
33.675
27.406
25.959
23.363
22.410
16.719
14.004
CN
O
CO2Et
Ethyl 1-(4-cyanobutyl)-2-oxocyclohexanecarboxylate
200
180
160
140
120
100
80
60
40
20
PPM


CN
O

4d

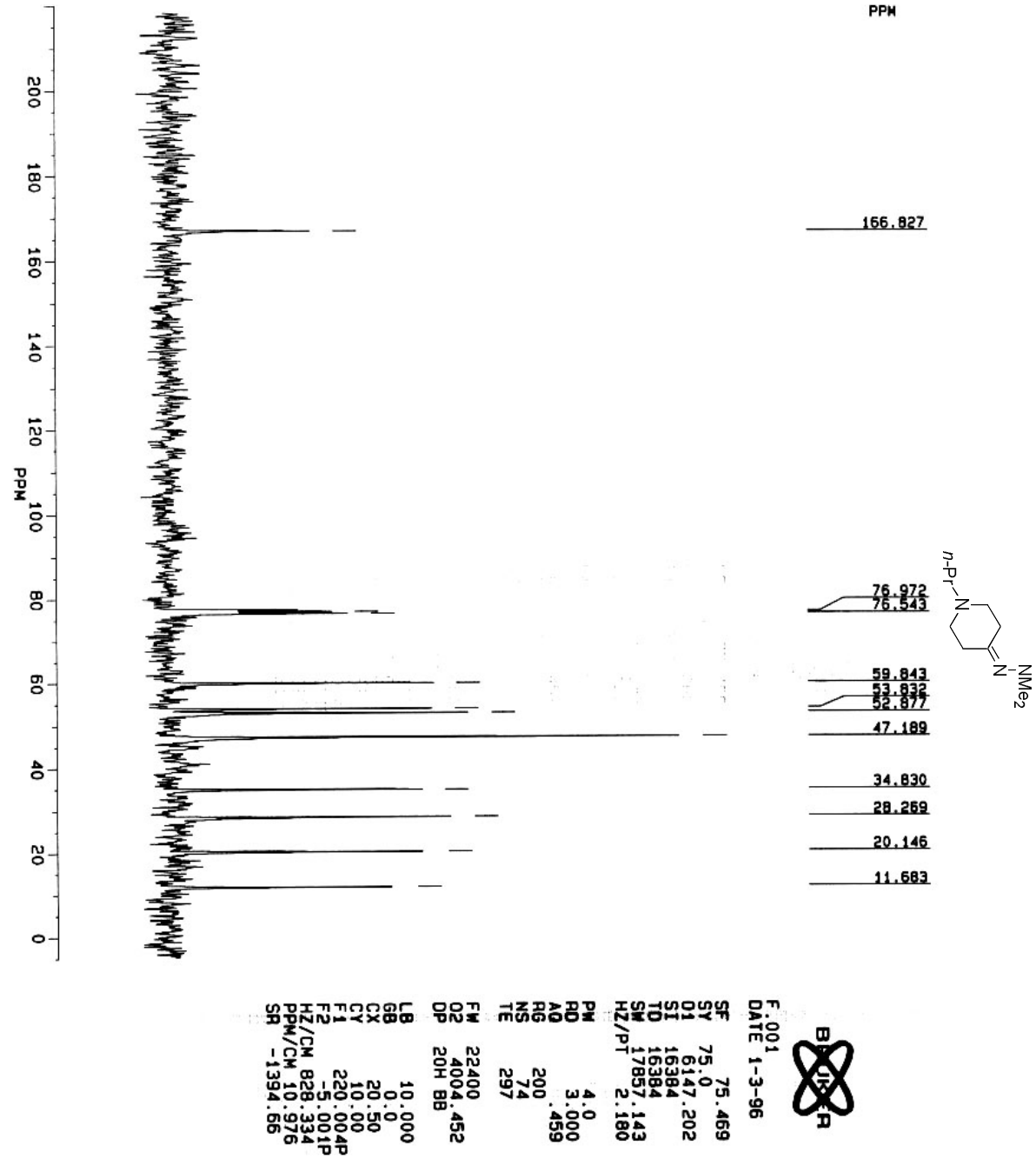

BRUKER
RAMAL106.001
DATE 7-10-99
TIME 11:54
SOLVENT CDCl3
SF 300.133
SY 210.0
O1 4800.000
SI 16384
TD 16384
SW 4504.505
HZ/PT .550
PW 5.0
RD 2.500
AQ 1.819
RG 4
NS 8
TE 297
FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 25.00
CY 6.00
F1 10.001P
F2 -.197P
HZ/CM 122.422
PPM/CM .408
SR 3367.75
2.40
2.20
PPM
1.80
1.60
1.40
1.20
PPM
4.085
1.929
5.644
4.188
9.0
8.0
7.0
6.0
5.0
PPM
4.0
3.0
2.0
1.0
0.0

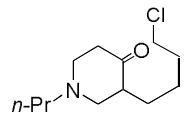

RAMAL106.101
DATE 7-10-99
TIME 7:10
PPM
SOLVENT CDC13
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 11971
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CX 25.00
CY 50
F1 220.0004P
F2 -.495P
HZ/CM 665.632
PPM/CM 8.820
SR -1403.38
119.690
77.432
77.000
76.577
50.426
42.080
33.992
28.537
27.954
26.524
25.591
25.010
17.007
PPM
200
180
160
140
120
100
80
60
40
20
O
CN
4d

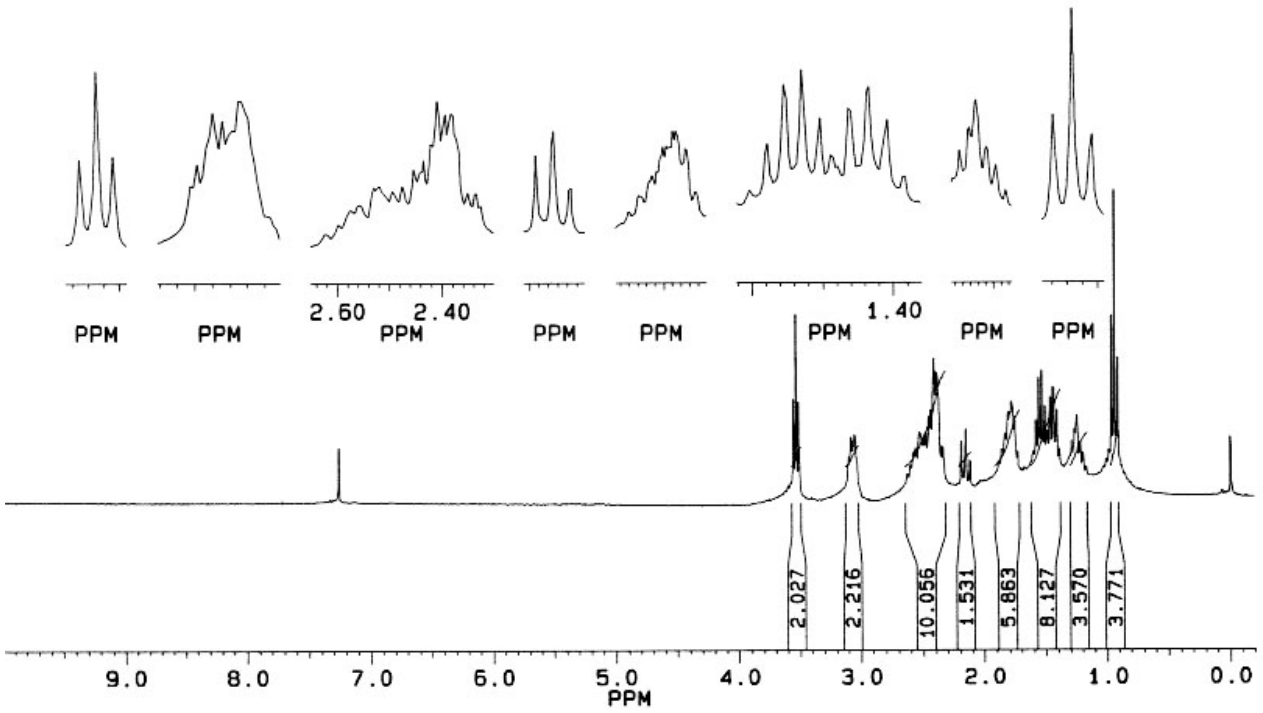


FFLF2.001
DATE 19-10-0

SF	300.133
SY	210.0
O1	4800.000
SI	8192
TD	8192
SW	4504.505
HZ/PT	1.100
PW	5.0
RD	2.500
AQ	.909
RG	4
NS	8
TE	297
FW	5700
O2	5684.205
DP	4L PO
LB	0.0
GB	0.0
CX	20.50
CY	7.00
F1	10.001P
F2	-.200P
HZ/CM	149.349
PPM/CM	.498
SR	3368.85


PPM
166.827
76.972
76.543
59.843
53.832
52.877
47.189
34.830
28.269
20.146
11.683
n-Pr
NMe2
PPM
F.001
DATE 1-3-96
SF 75.469
SY 75.0
D1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 74
TE 297
FW 22400
O2 4004.452
OP 20H BB
LB 10.000
GB 0.0
CX 20.50
CY 10.00
F1 220.004P
F2 -5.001P
HZ/CM 828.334
PPM/CM 10.976
SR -1394.66



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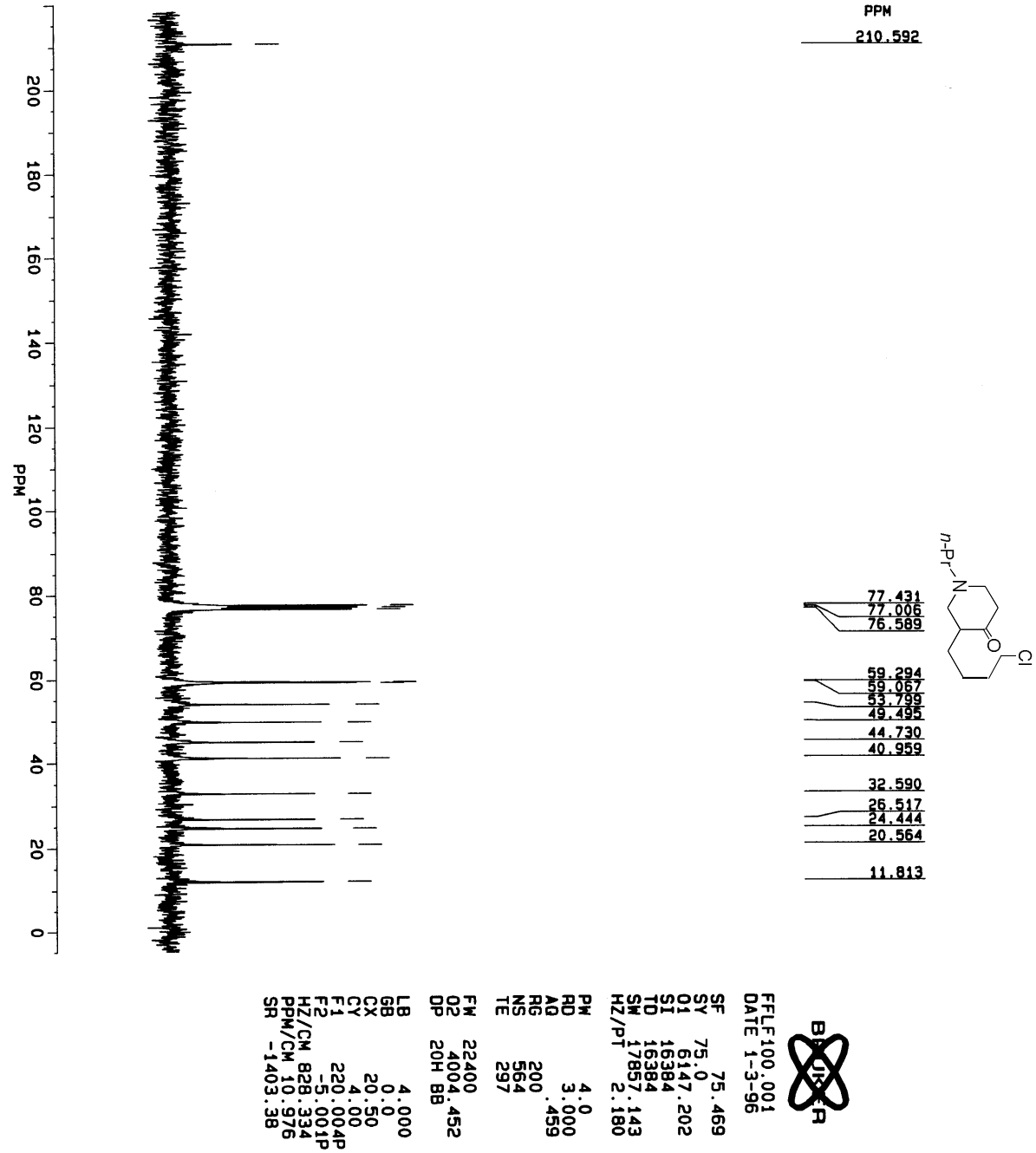
FFLF33.001
DATE 24-10-0

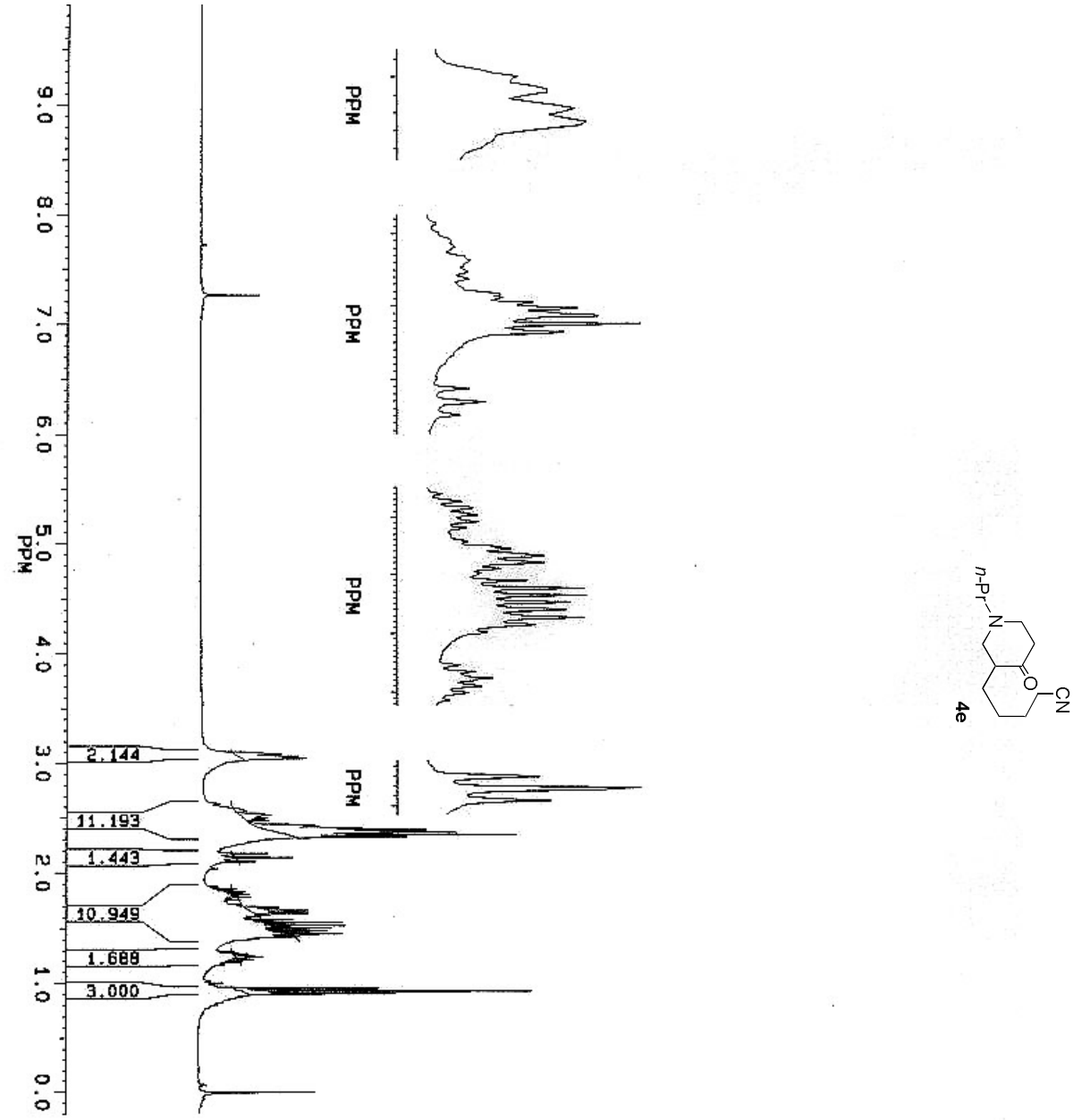
SF     300.133
SY 210.0
O1    4800.000
SI    8192
TD    8192
SW    4504.505
HZ/PT    1.100

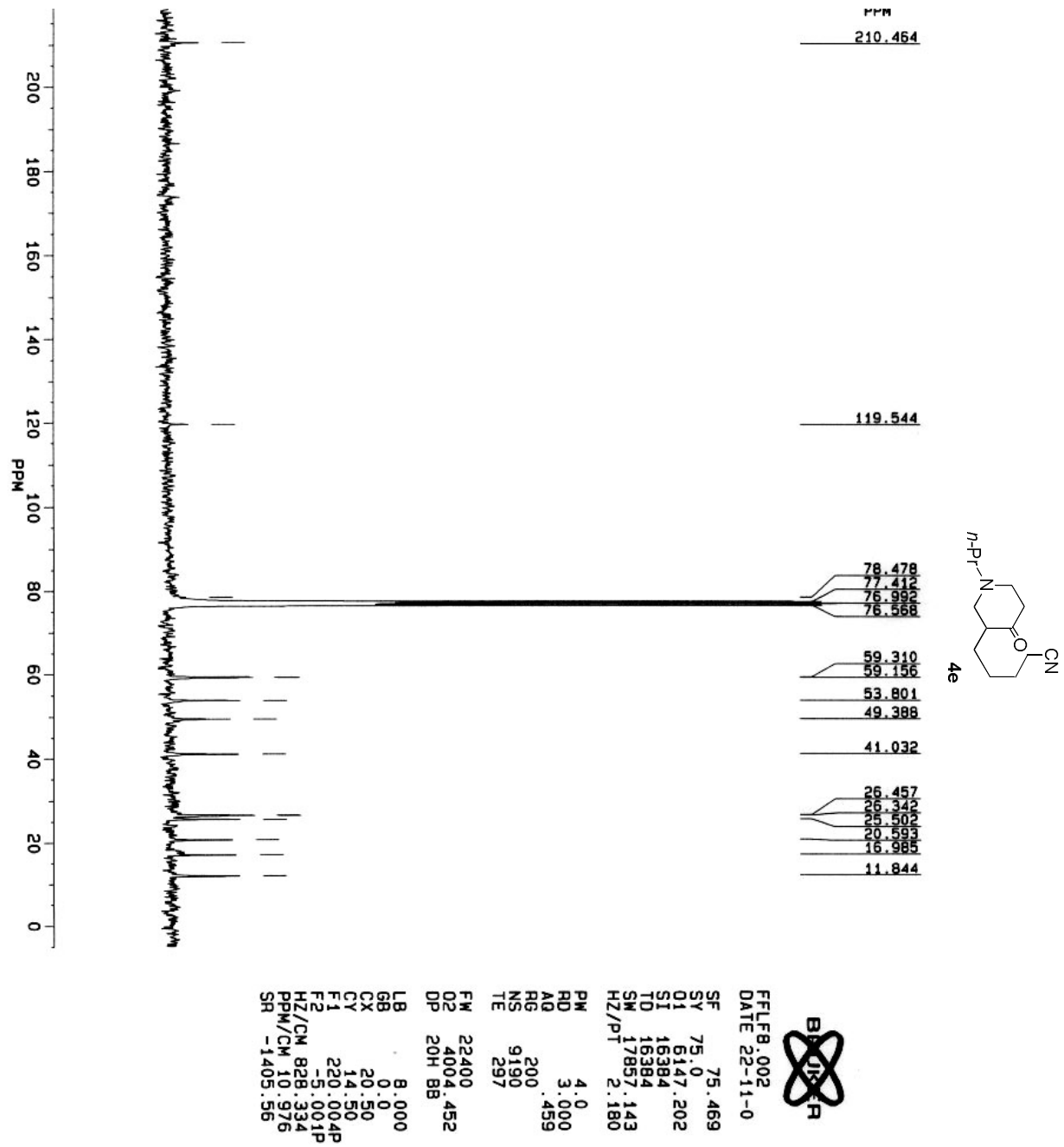
PW       5.0
RD       2.500
AQ        .909
RG       4
NS       8
TE     297

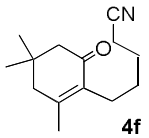
FW    5700
O2    5684.205
DP    4L PO

LB       0.0
GB       0.0
CX      20.50
CY       5.00
F1      10.001P
F2       -.200P
HZ/CM 149.349
PPM/CM    .498
SR    3367.75
```

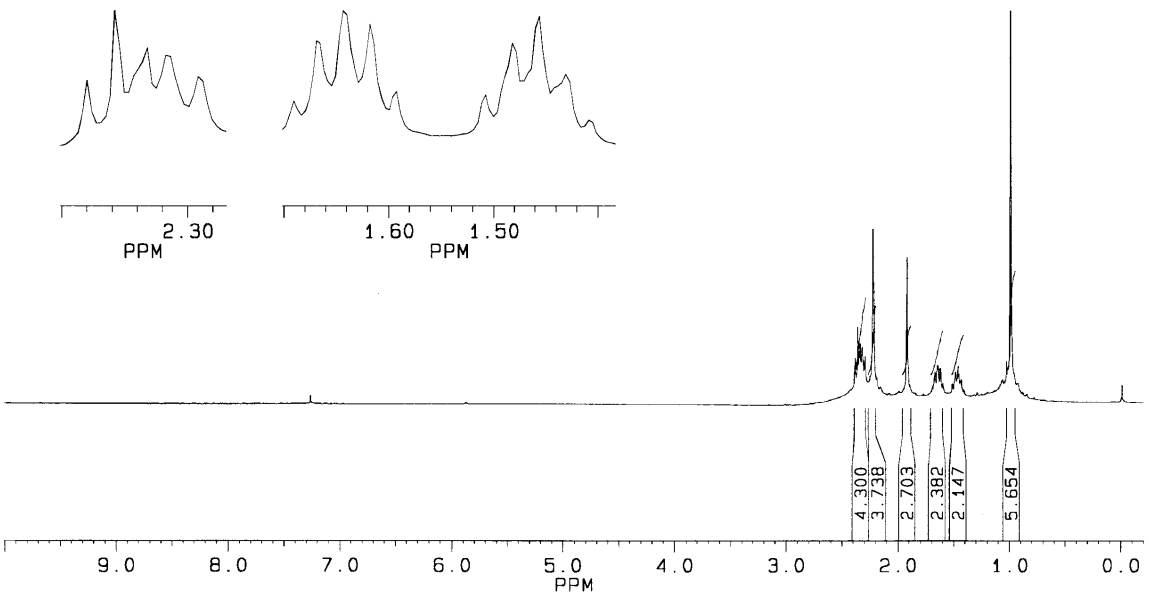

PPM
210.592
77.431
77.006
76.589
59.294
59.067
53.799
49.495
44.730
40.959
32.590
26.517
24.444
20.564
11.813
n-Pr
N
O
Cl
PPM
0
20
40
60
80
100
120
140
160
180
200
FFLF100.001
DATE 1-3-96
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 564
TE 297
FW 22400
O2 4004.452
OP 20H BB
LB 4.000
GB 0.0
CX 20.50
CY 4.00
F1 220.0004P
F2 -5.0001P
HZ/CM 828.334
PPM/CM 10.976
SR -1403.38


n-Pr
N
O
CN
4e
2.144
11.193
1.443
10.949
1.688
3.000
PPM
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
FFLFB.001
DATE 21-11-0
SF 300.133
SY 210.0
O1 4800.000
SI 8192
TD 8192
SW 4504.505
HZ/PT 1.100
PW 5.0
RD 2.500
AQ .909
RG 4
NS 8
TE 297
FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 20.50
CY 6.00
F1 10.0001P
F2 -.200P
HZ/CM 149.349
PPM/CM .498
SR 3367.75


PPM
210.464
119.544
78.478
77.412
76.992
76.566
59.310
59.156
53.801
49.388
41.032
26.457
26.342
25.502
20.593
16.985
11.844
n-Pr
N
O
CN
4e
PPM
0
20
40
60
80
100
120
140
160
180
200
FFLF8.002
DATE 22-11-0
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 9190
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 8.000
GB 0.0
CX 20.50
CY 14.50
F1 220.004P
F2 -5.001P
HZ/CM 828.334
PPM/CM 10.976
SR -1405.56



4f

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FFLF17.001
DATE 2-2-1

SF     300.133
SY 210.0
O1    4800.000
SI    8192
TD    8192
SW    4504.505
HZ/PT    1.100

PW       5.0
RD       2.500
AQ        .909
RG       2
NS       8
TE     297

FW    5700
O2    5684.205
DP    4L PO

LB       0.0
GB       0.0
CX      20.50
CY       7.00
F1      10.001P
F2       -.200P
HZ/CM 149.349
PPM/CM    .498
SR    3367.75
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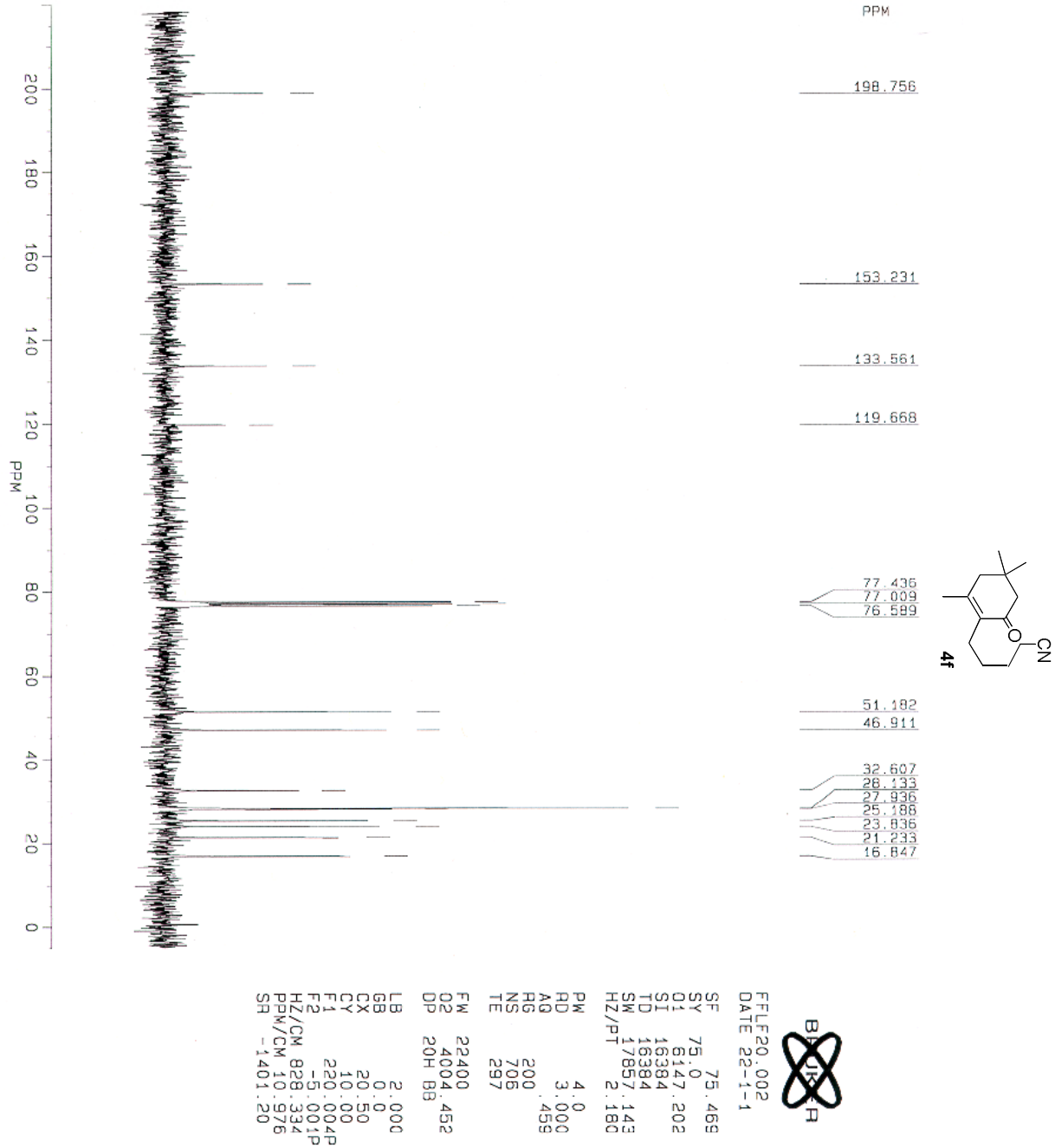
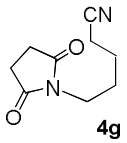
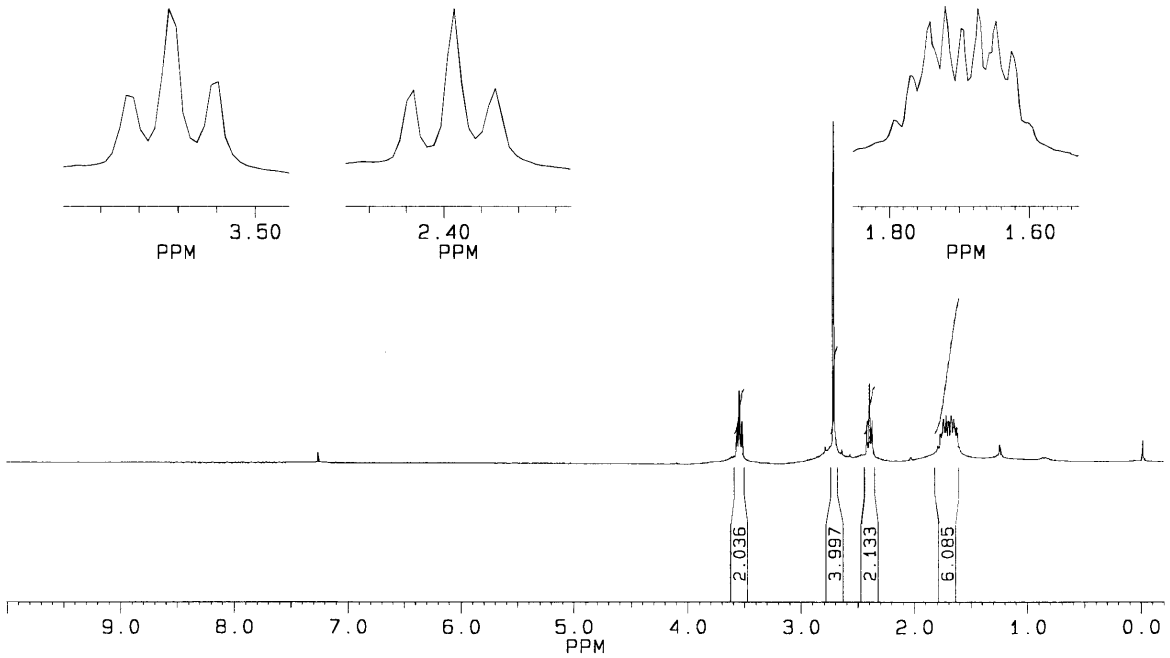

PPM
198.756
153.231
133.561
119.668
77.436
77.009
76.589
51.182
46.911
32.607
28.133
27.936
25.186
23.836
21.233
16.847
4f
CN
O
PPM
200
180
160
140
120
100
80
60
40
20
0
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 706
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 2.000
GB 0.0
CX 20.50
CY 10.000
F1 220.004P
F2 -5.001P
HZ/CM 828.334
PPM/CM 10.976
SR -1401.20
FFLF20.002
DATE 22-1-1
BRUKER



4g

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FFLF16.001
DATE 1-2-1

SF     300.133
SY 210.0
O1    4800.000
SI    8192
TD    8192
SW    4504.505
HZ/PT    1.100

PW       5.0
RD       2.500
AQ        .909
RG       4
NS       8
TE     297

FW    5700
O2    5684.205
DP    4L PO

LB       0.0
GB       0.0
CX      20.50
CY       6.00
F1      10.001P
F2       -.200P
HZ/CM 149.349
PPM/CM    .498
SR    3367.75
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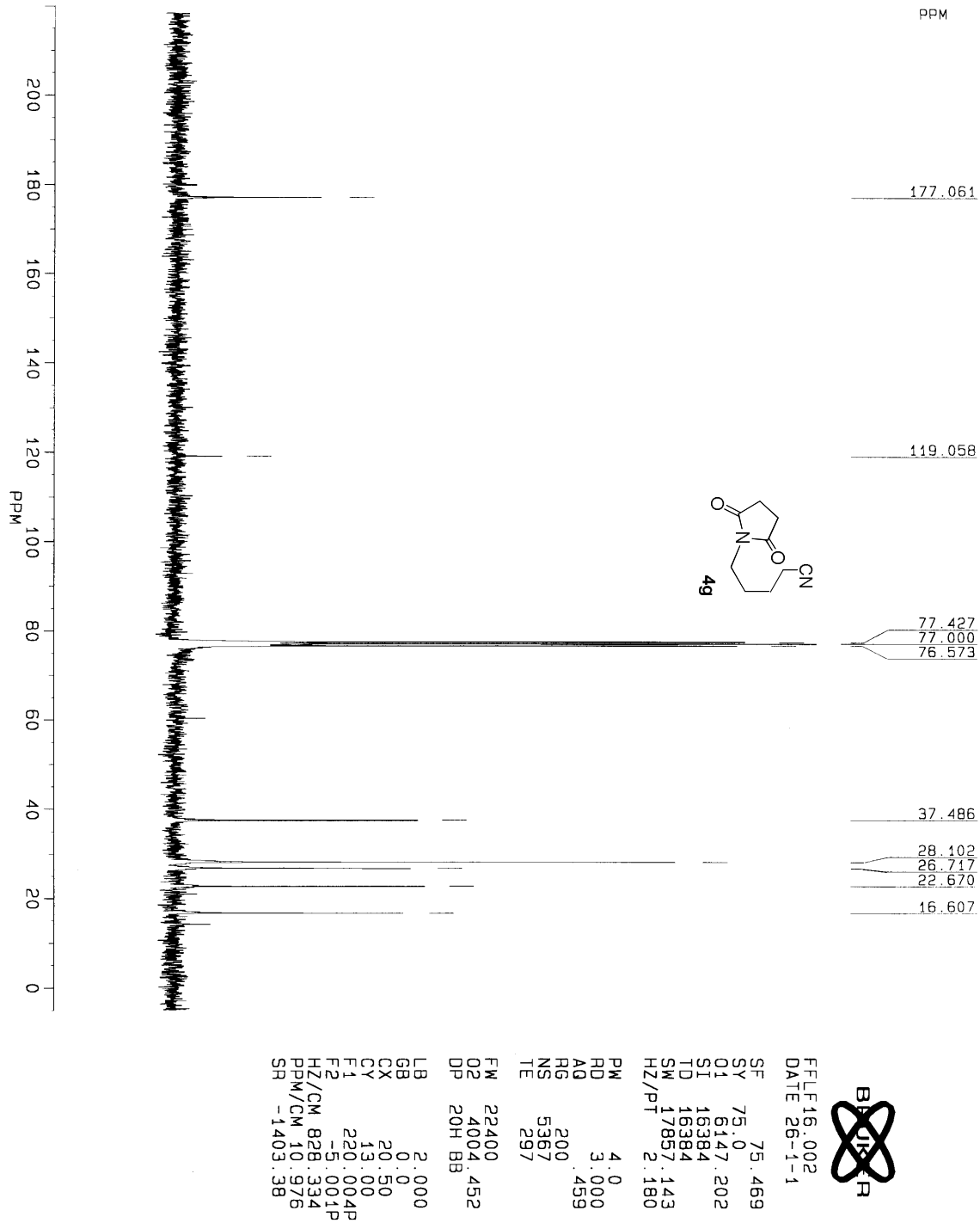
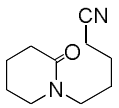
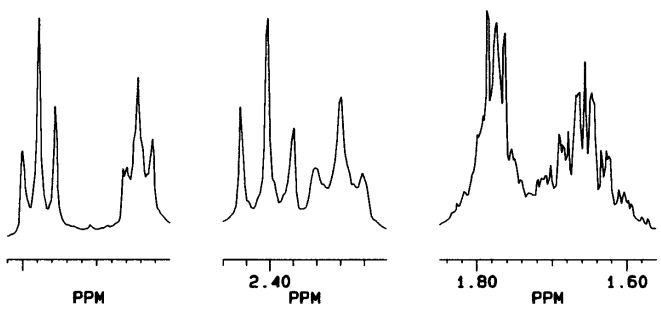
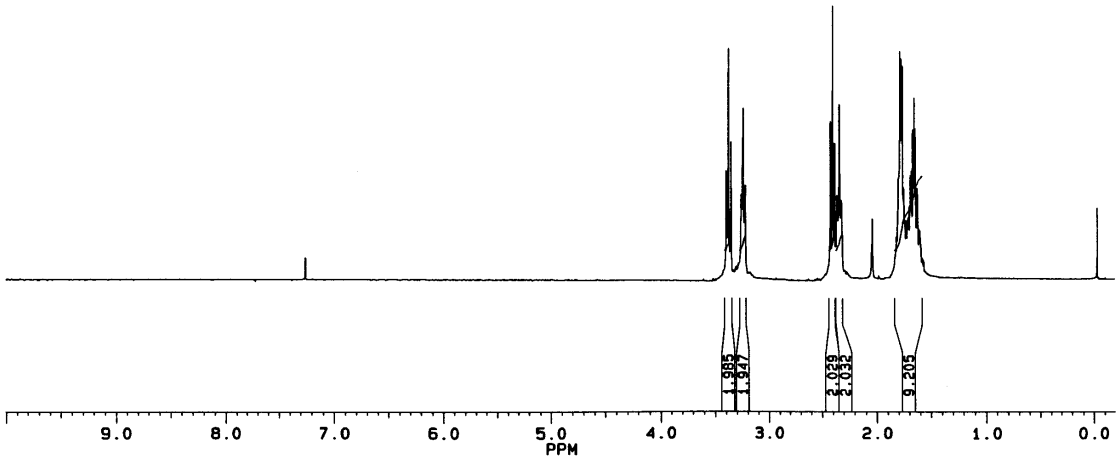

PPM
177.061
119.058
77.427
77.000
76.573
37.486
28.102
26.717
22.670
16.607
4g
O
N
O
CN
200
180
160
140
120
100
80
60
40
20
0
PPM
FFLF16.002
DATE 26-1-1
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 5367
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 2.000
GB 0.0
CX 20.50
CY 13.00
F1 220.004P
F2 -5.001P
HZ/CM 828.334
PPM/CM 10.976
SR -1403.38



4h





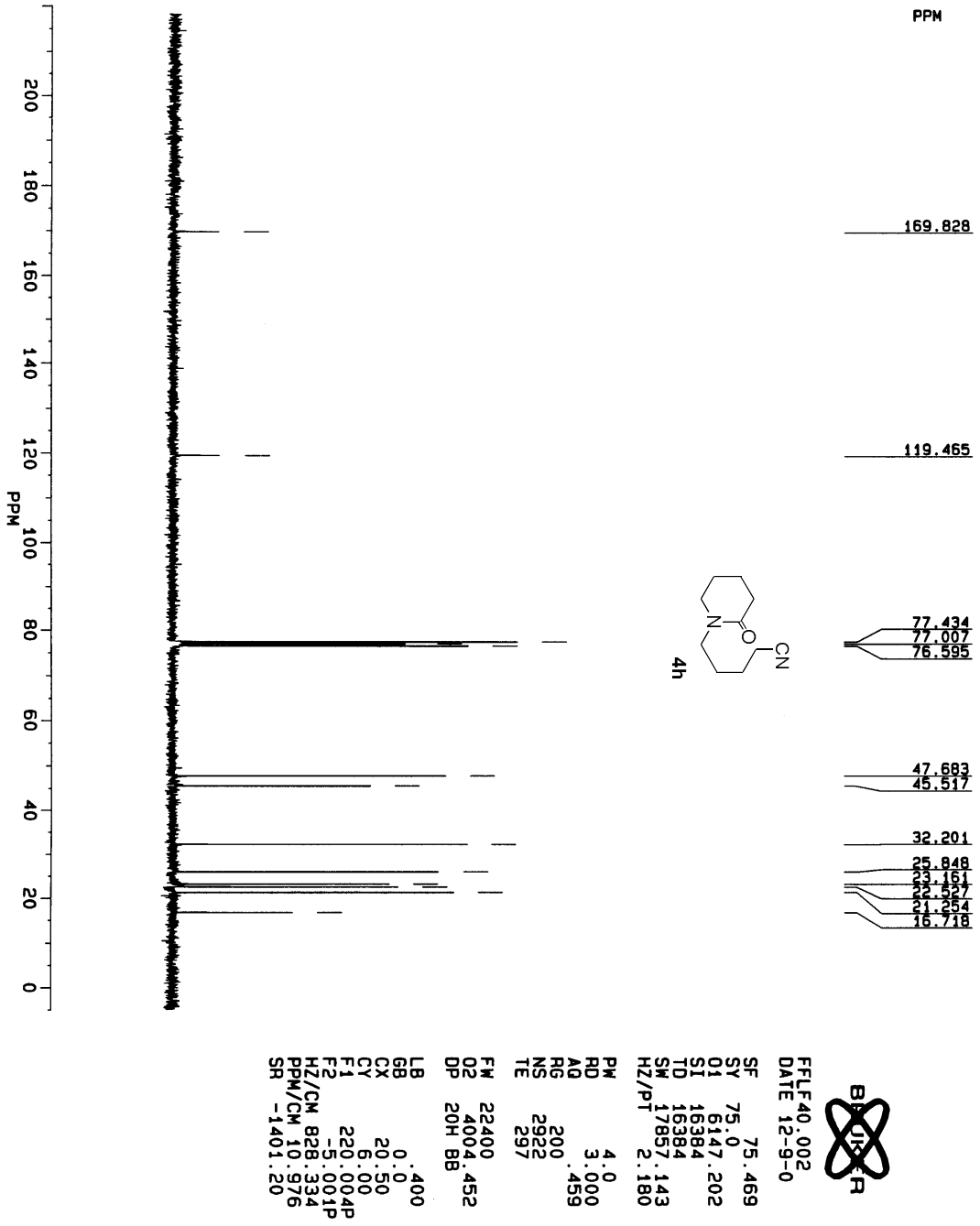
FFLF40.001
DATE 12-9-0

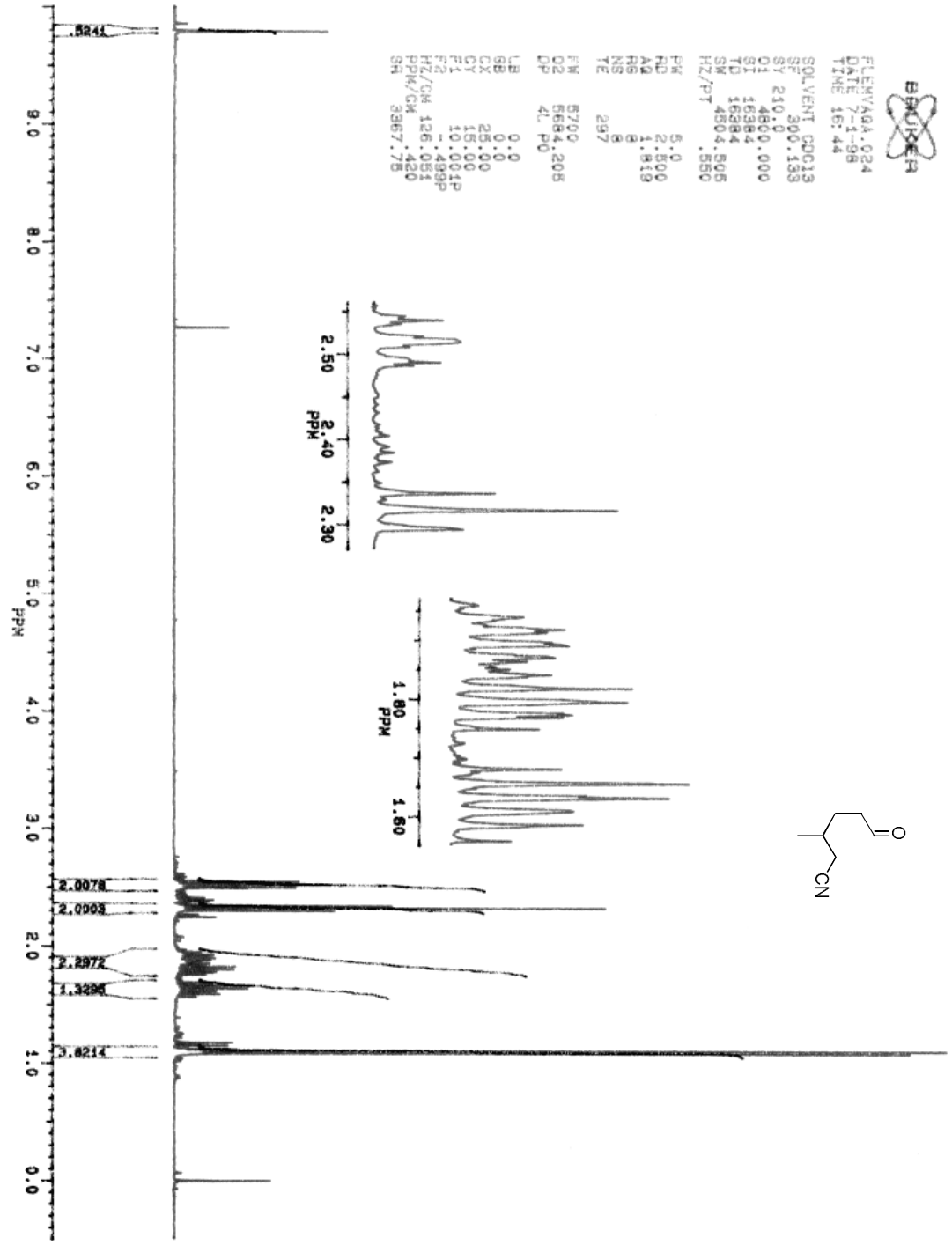
SF 300.133
SY 210.0
O1 4800.000
SI 16384
TD 16384
SW 4504.505
HZ/PT .550

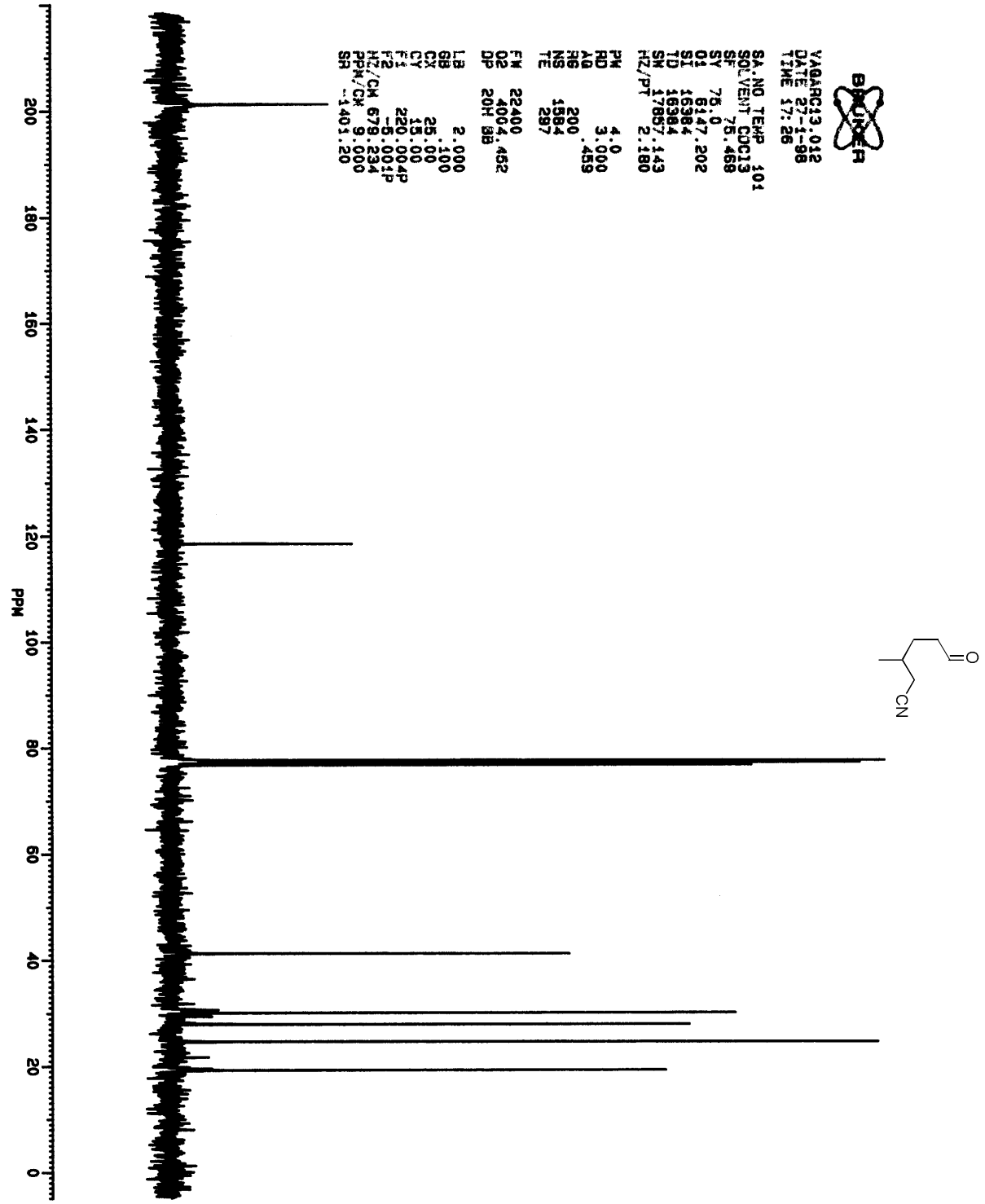
PW 5.0
RD 2.500
AQ 1.819
RG 2
NS 8
TE 297

FW 5700
O2 5684.205
DP 4L PO

LB 0.0
GB .100
CX 20.50
CY 5.00
F1 10.001P
F2 -.198P
HZ/CM 149.322
PPM/CM .498
SR 3367.20


PPM
169.828
119.465
77.434
77.007
76.595
47.683
45.517
32.201
25.848
23.161
22.527
21.254
16.718
4h
N
O
CN
FFLF40.002
DATE 12-9-0
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 2922
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB .400
GB 0.0
CX 20.50
CY 6.00
F1 220.004P
F2 -5.001P
HZ/CM 828.334
PPM/CM 10.976
SR -1401.20


.5241
2.0078
2.0003
2.2972
1.3295
3.0214
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
PPM
2.50
2.40
2.30
PPM
1.80
1.60
PPM
O
CN


BRUKER
YAGARC13.012
DATE 27-1-98
TIME 17:26
SA,NO TEMP 101
SOLVENT CDCl3
SF 75.468
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 1584
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 2.000
GB .100
CX 25.00
CY 15.00
F1 220.0004P
F2 -5.0001P
HZ/CM 678.234
PPM/CM 9.000
SR -1401.20
PPM
0
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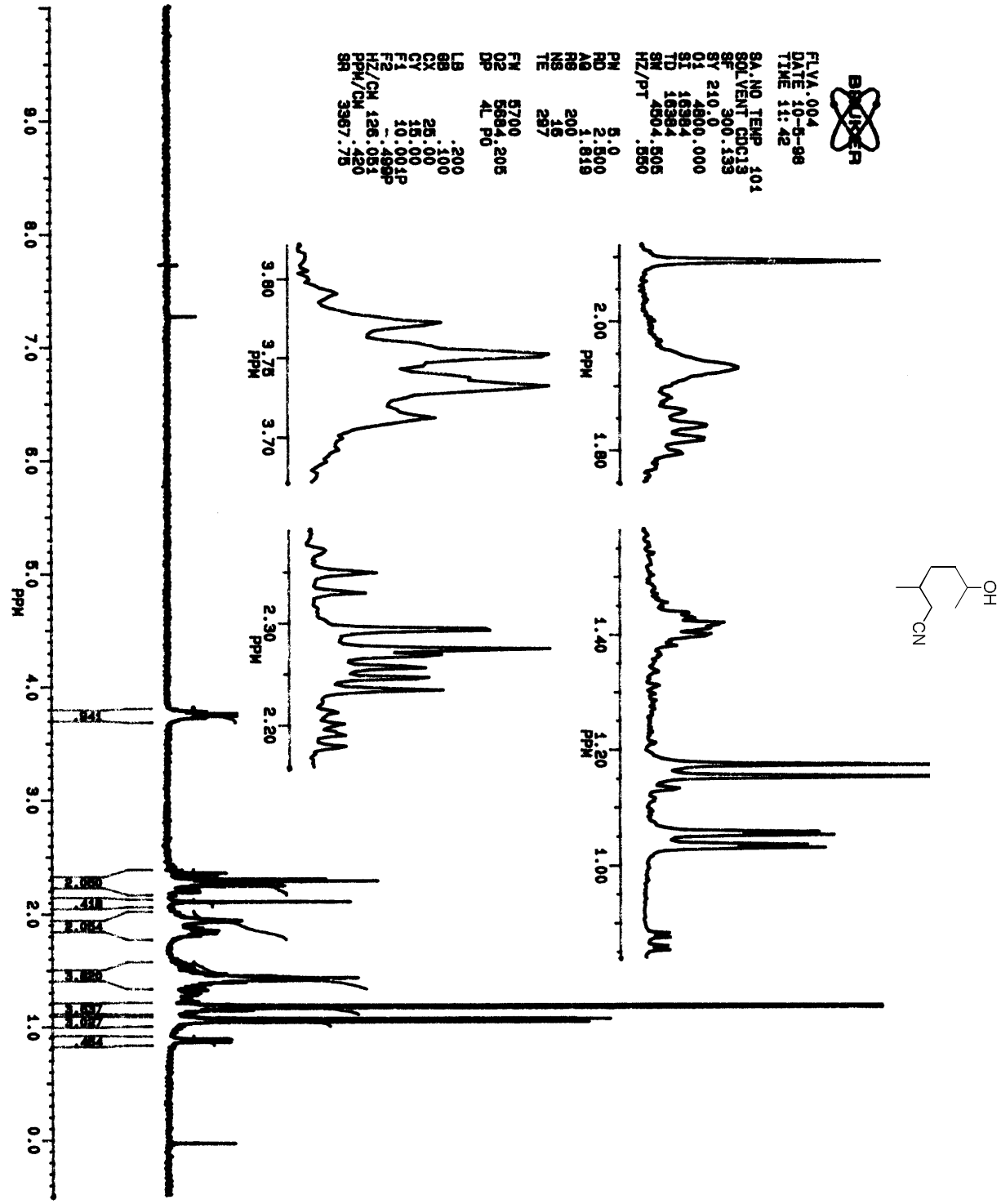
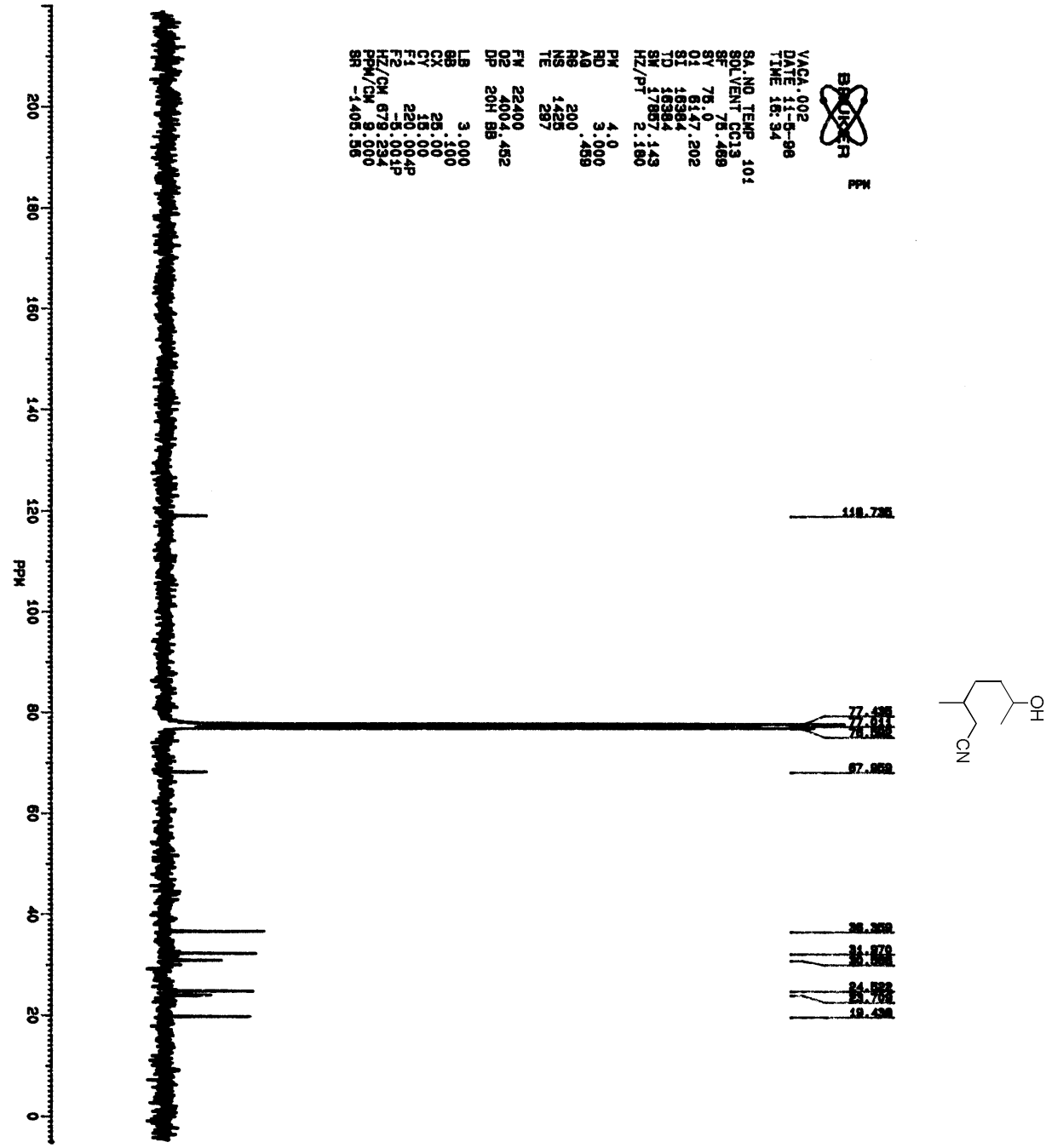
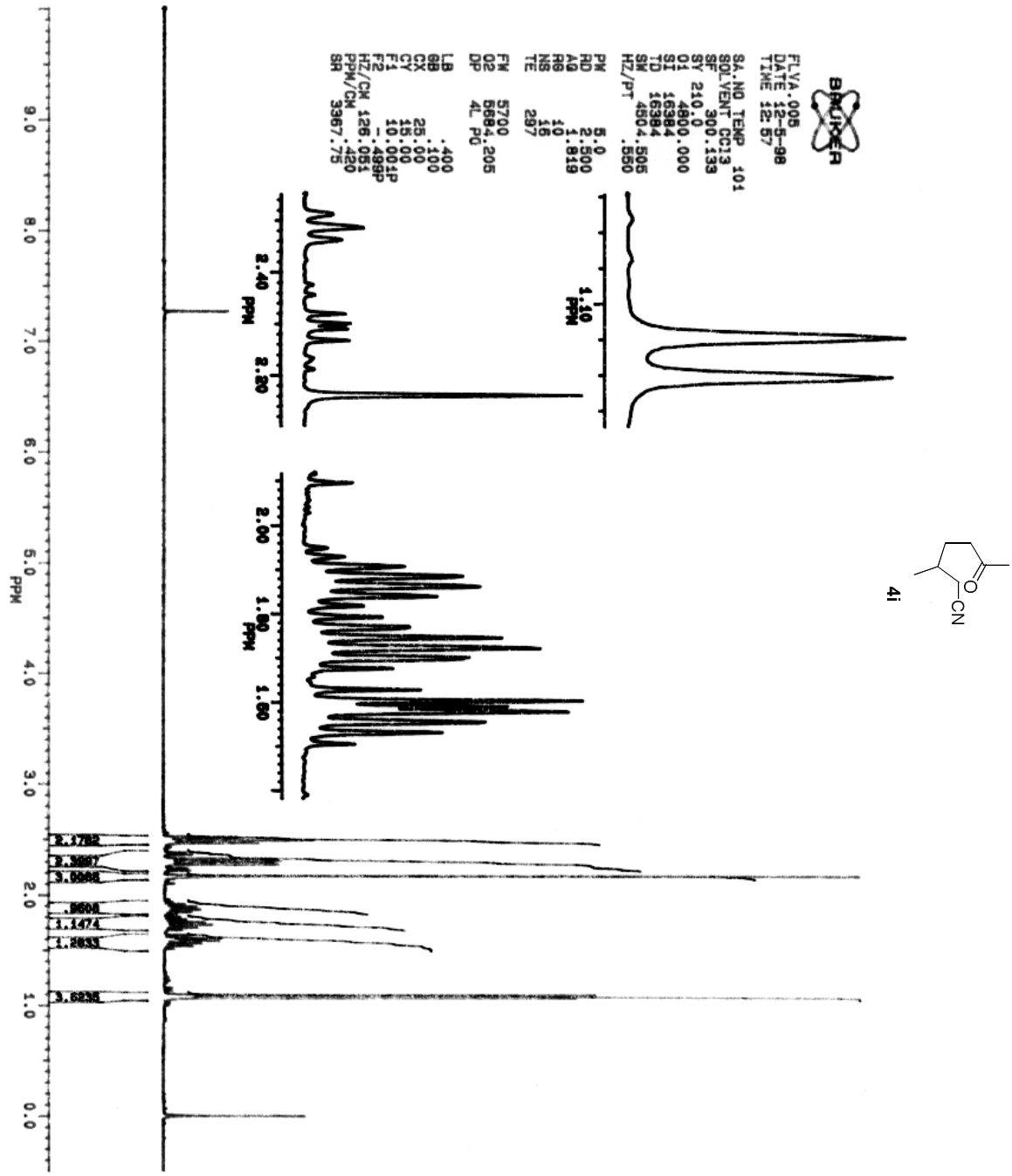
60
80
100
120
140
160
180
200
O
CN


OH
CN


VACA.002
DATE 11-5-98
TIME 16:34
SA.NO TEMP 101
SOLVENT CDCl3
SF 75.469
SW 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
RG 200
NS 1425
TE 297
FW 22400
DP 20H BB
LB 3.000
GB .100
GX 25.00
GY 15.00
F1 220.0004P
F2 -5.0014P
HZ/CM 679.234
PPM/CM 9.000
SR -1405.56
PPM
118.736
77.436
77.011
76.586
67.859
38.359
31.870
30.986
24.822
24.706
19.436
PPM
OH
CN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AQ 1.819
RG 10
NS 16
TE 297
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O2 5684.205
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LB .400
GB .100
CX 25.00
CY 15.00
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F2 -.499P
HZ/CM 126.051
PPM/CM .420
SR 3367.75
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1.10 PPM
2.00 1.90 PPM 1.80
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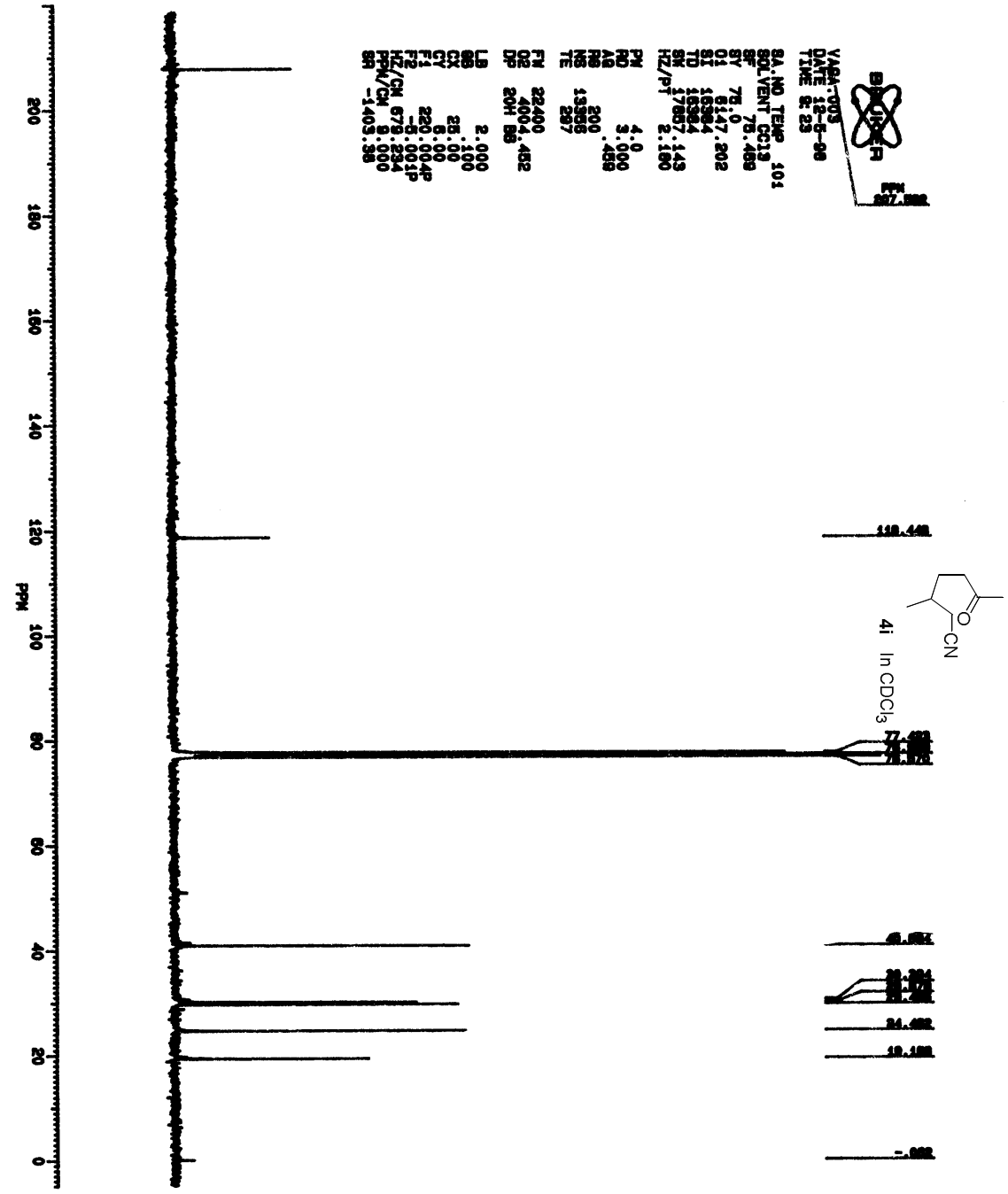
O
CN

4i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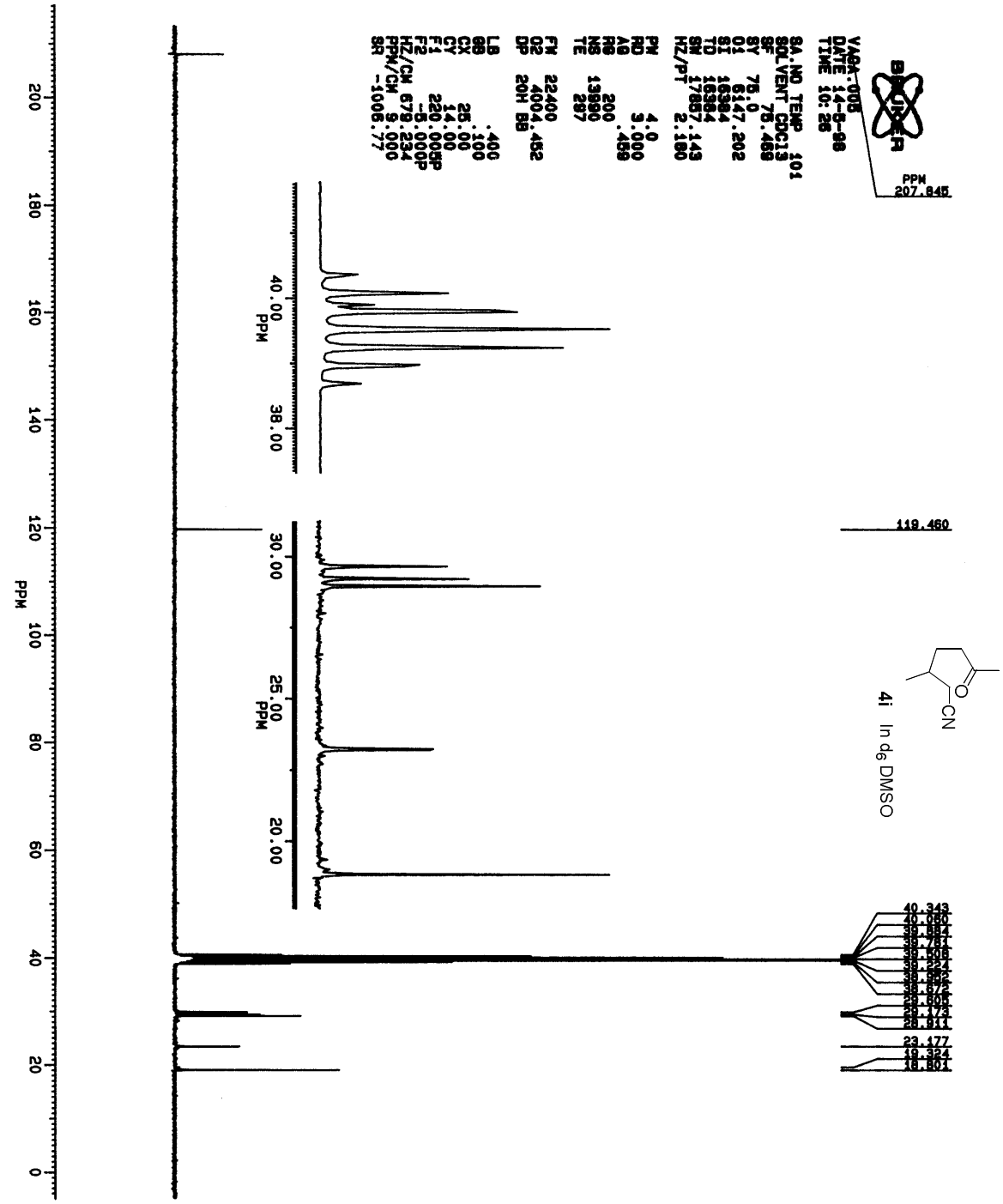
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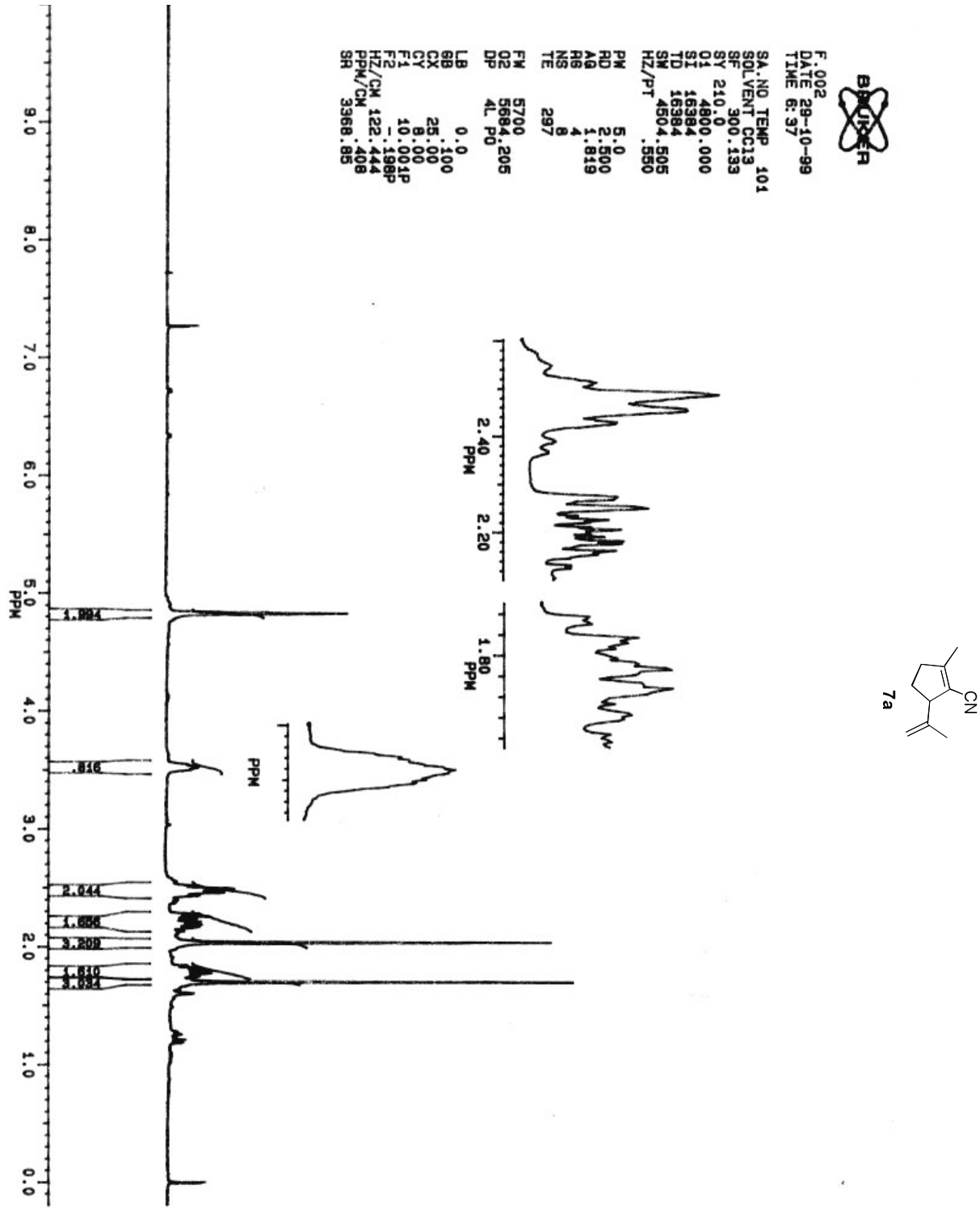
PPM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HZ/PT .550
PW 5.0
RD 2.500
AQ 1.819
RG 4
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TE 297
FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB .100
CX 25.00
CY 8.00
F1 10.0001P
F2 -.198P
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PPM/CM .408
SR 3368.85
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PPM
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1.80
PPM
PPM
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7.0
6.0
5.0
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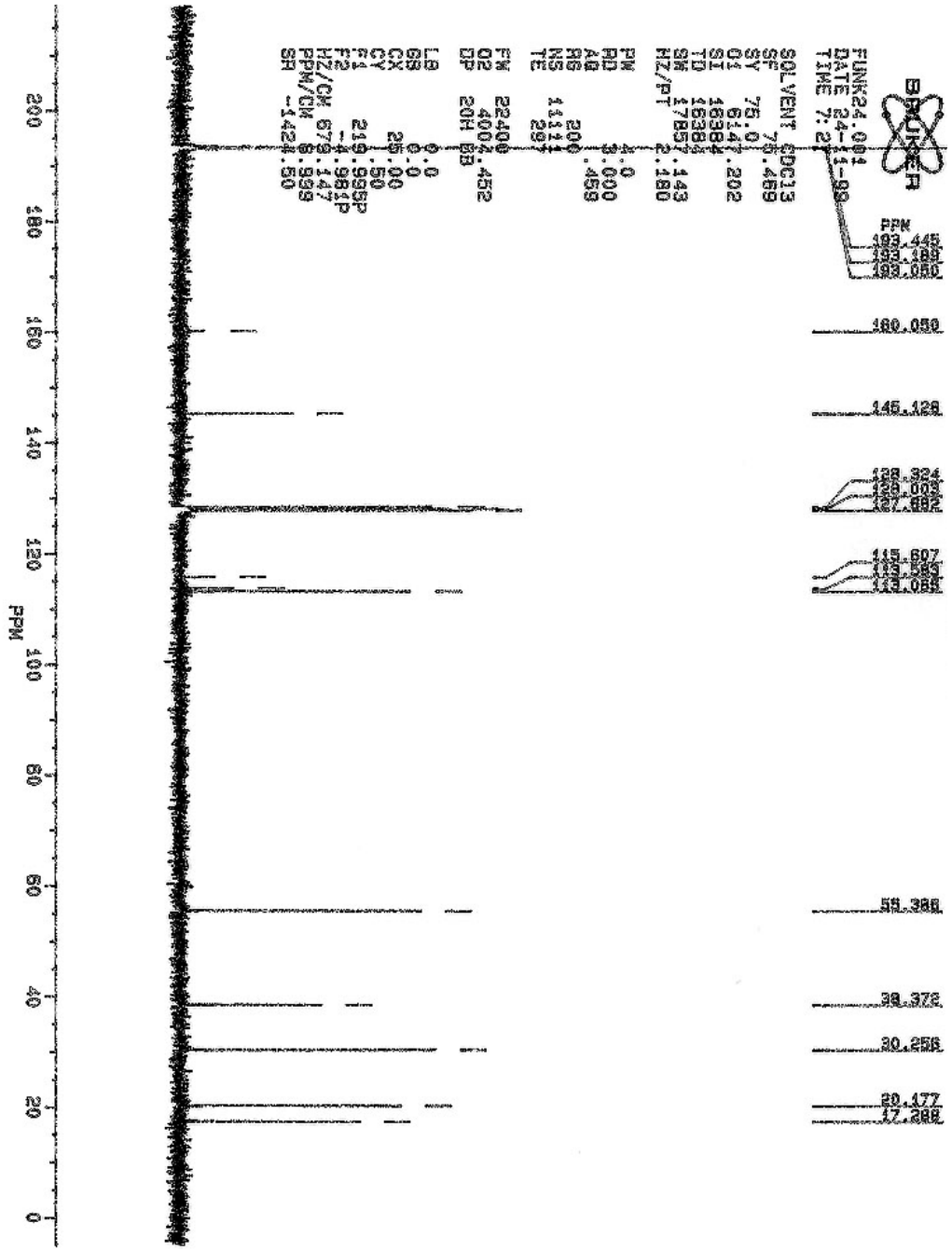
7a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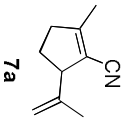
17.288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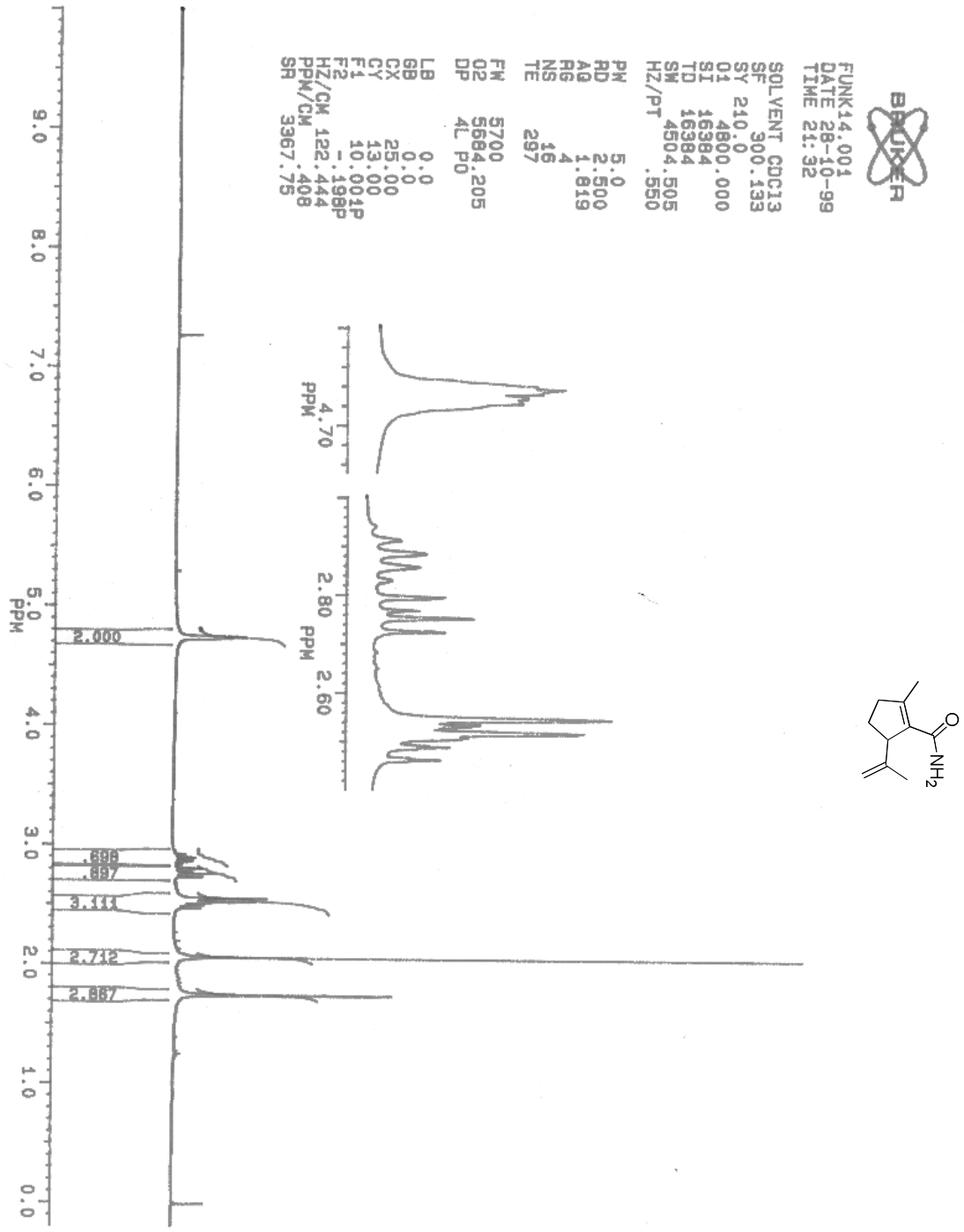
7a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HZ/PT .550
PW 5.0
RD 2.500
AQ 1.819
RG 4
NS 16
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FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 25.00
CY 13.00
F1 10.001P
F2 -.198P
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PPM/CM .408
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2.60
PPM
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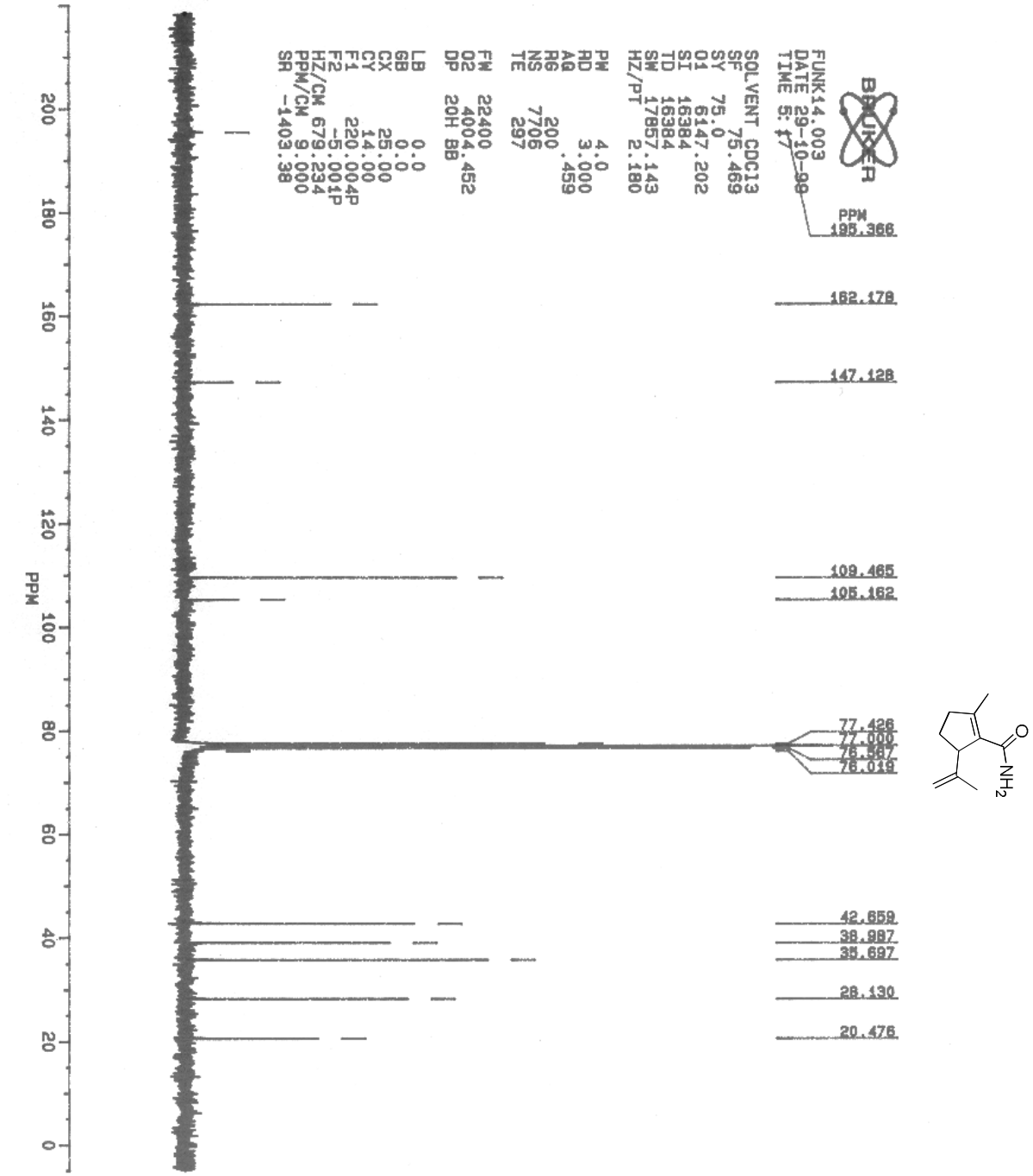
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FUNK14.003
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O1 6147.202
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HZ/PT 2.180
PW 4.0
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AQ .459
RG 200
NS 7706
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CX 25.00
CY 14.00
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SR -1403.38
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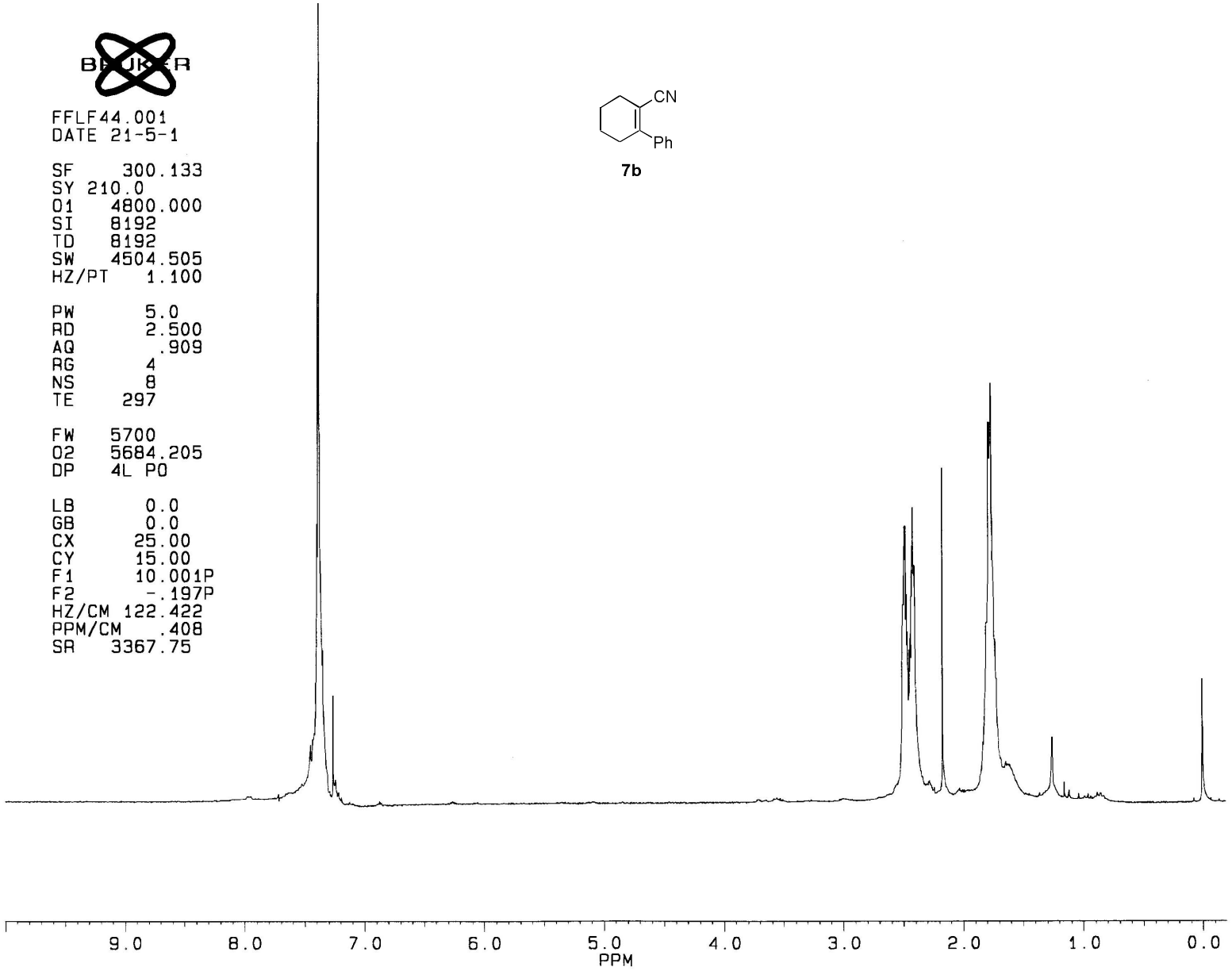
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HZ/PT 1.100
PW 5.0
RD 2.500
AQ .909
RG 4
NS 8
TE 297
FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 25.00
CY 15.00
F1 10.001P
F2 -.197P
HZ/CM 122.422
PPM/CM .408
SR 3367.75
CN
Ph
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7.0
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5.0
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3.0
2.0
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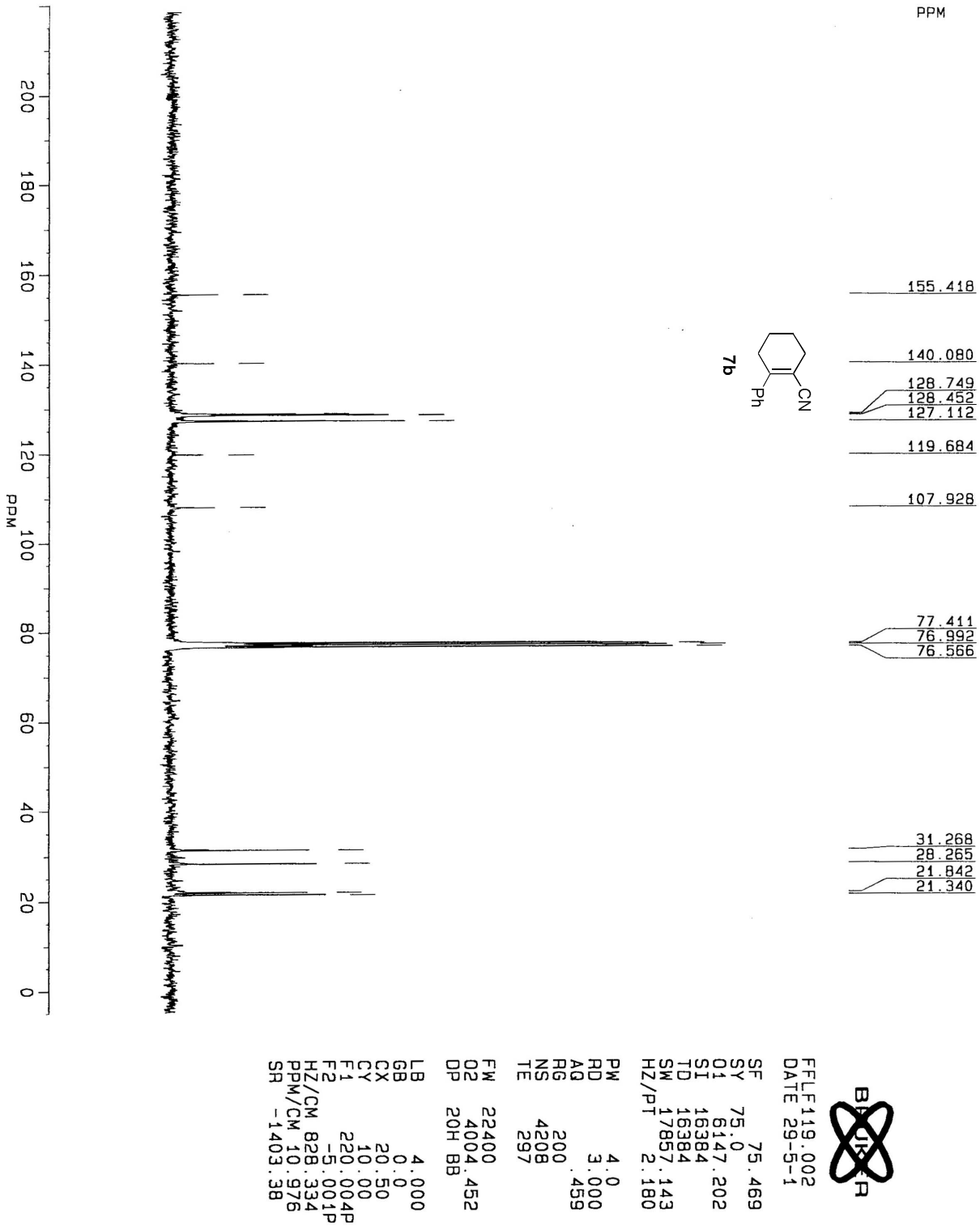
PPM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7b
Ph
CN
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20
40
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80
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120
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160
180
200
PPM
FFLF119.002
DATE 29-5-1
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SY 75.0
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HZ/PT 2.180
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RG 200
NS 4208
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 4.000
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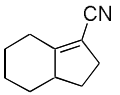
PPM/CM 10.976
SR -1403.38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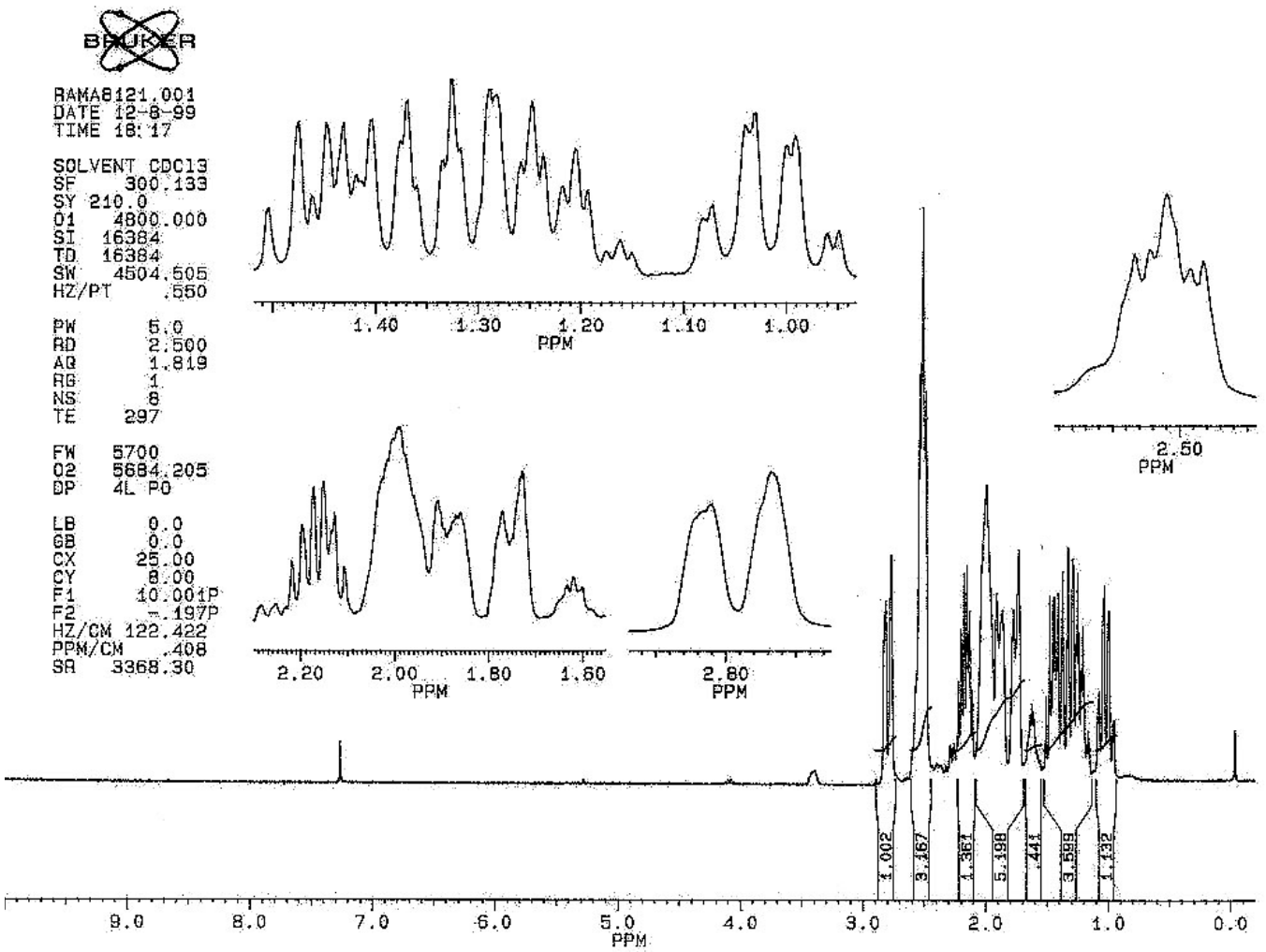
BRUKER
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DATE 12-8-99
TIME 18:17
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SI 16384
TD 16384
SW 4504.505
HZ/PT .550
PW 5.0
RD 2.500
AQ 1.819
RG 1
NS 8
TE 297
FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 25.00
CY 8.00
F1 10.001P
F2 -.197P
HZ/CM 122.422
PPM/CM .408
SR 3368.30
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PPM
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PPM
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PPM
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PPM
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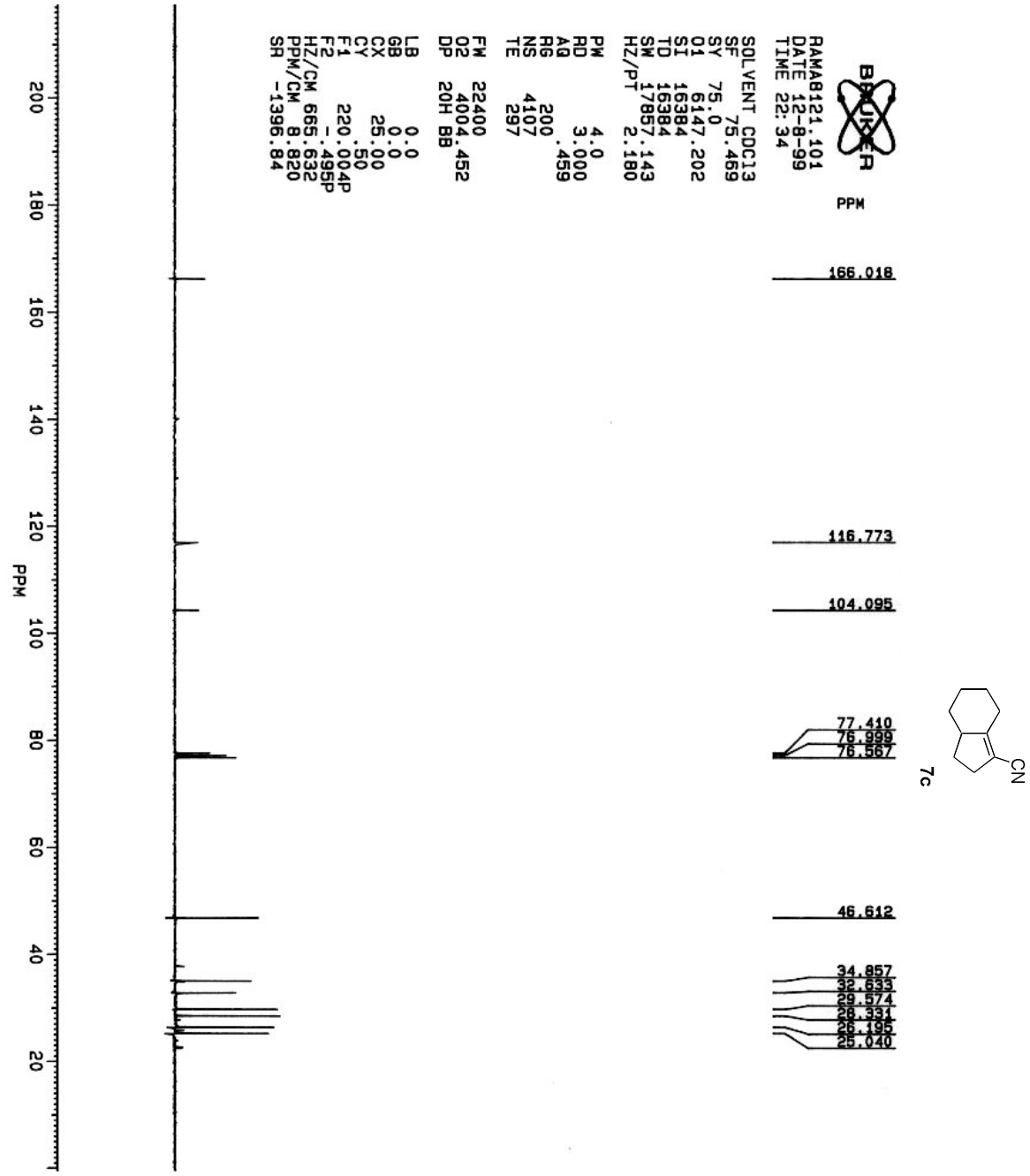
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TD 16384
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HZ/PT 2.180
PW 4.0
RD 3.000
AQ .459
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NS 4107
TE 297
FW 22400
O2 4004.452
DP 20H BB
LB 0.0
GB 0.0
CX 25.00
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F2 -.495P
HZ/CM 665.632
PPM/CM 8.820
SR -1396.84
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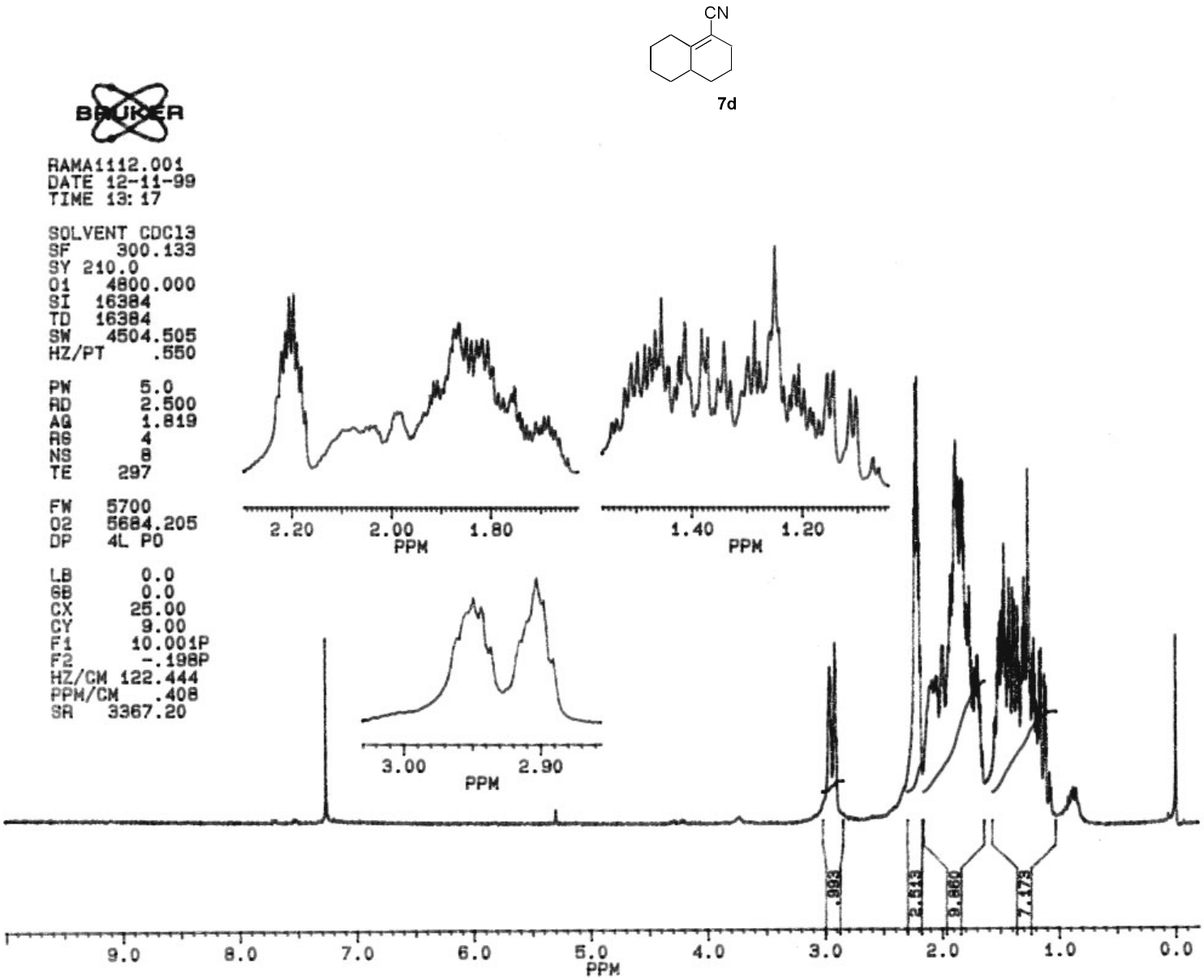
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BRUKER
RAMA1112.001
DATE 12-11-99
TIME 13:17
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SI 16384
TD 16384
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HZ/PT .550
PW 5.0
RD 2.500
AQ 1.819
RG 4
NS 8
TE 297
FW 5700
O2 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 25.00
CY 9.00
F1 10.001P
F2 -.198P
HZ/CM 122.444
PPM/CM .408
SR 3367.20
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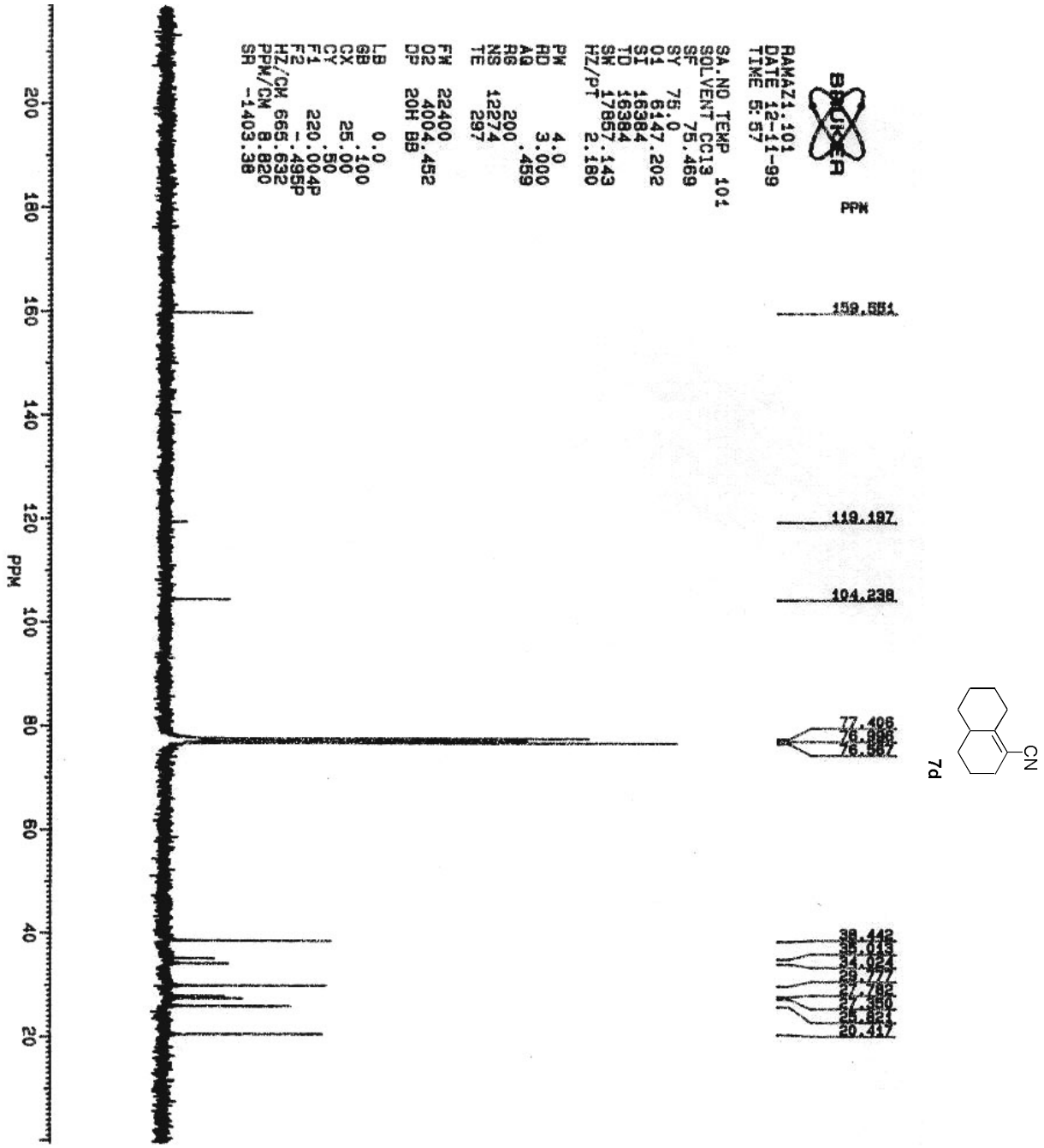
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DP 20H BB
LB 0.0
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PPM/CM 8.820
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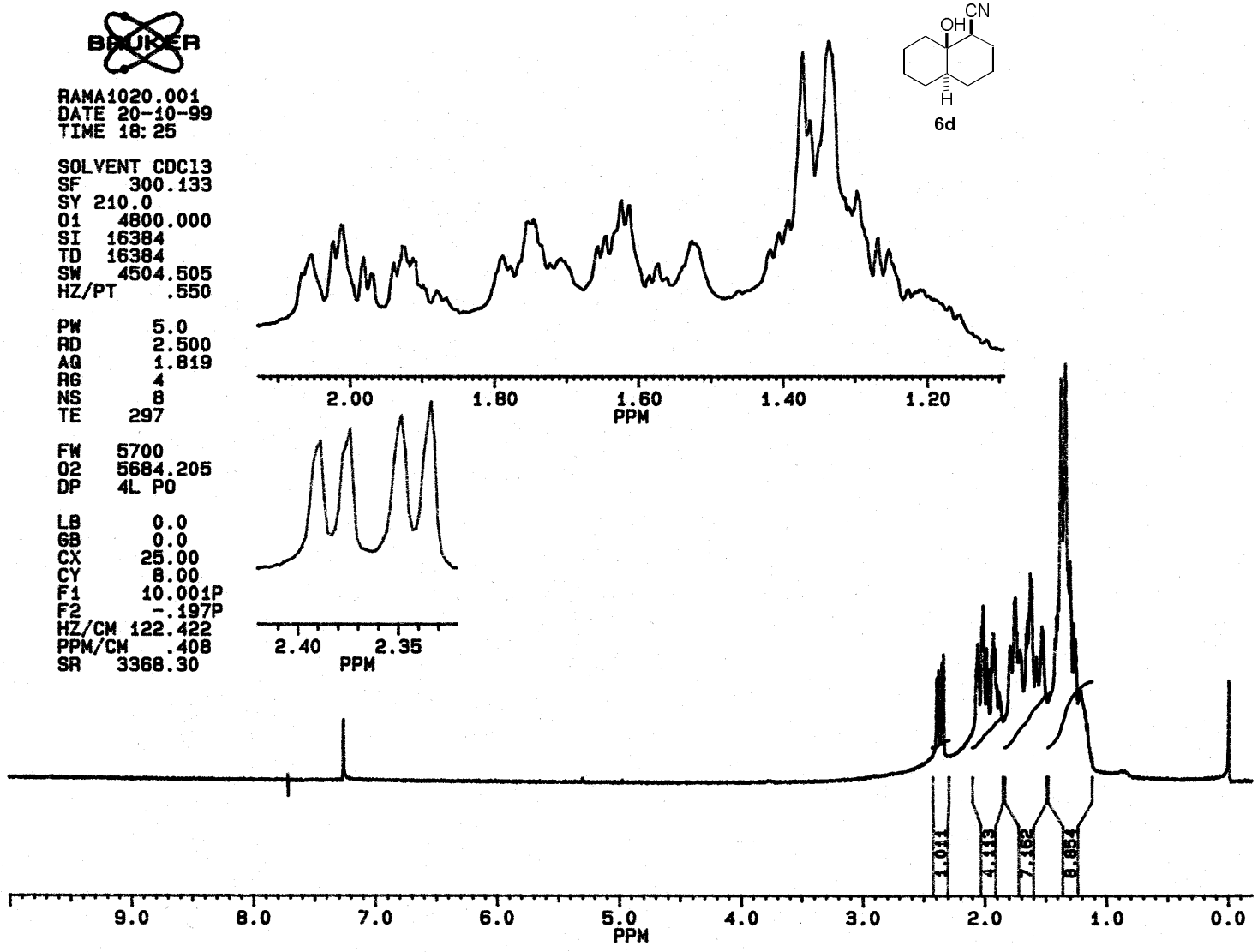
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AQ 1.819
RG 4
NS 8
TE 297
FW 5700
02 5684.205
DP 4L PO
LB 0.0
GB 0.0
CX 25.00
CY 8.00
F1 10.001P
F2 -.197P
HZ/CM 122.422
PPM/CM .408
SR 3368.30
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OH
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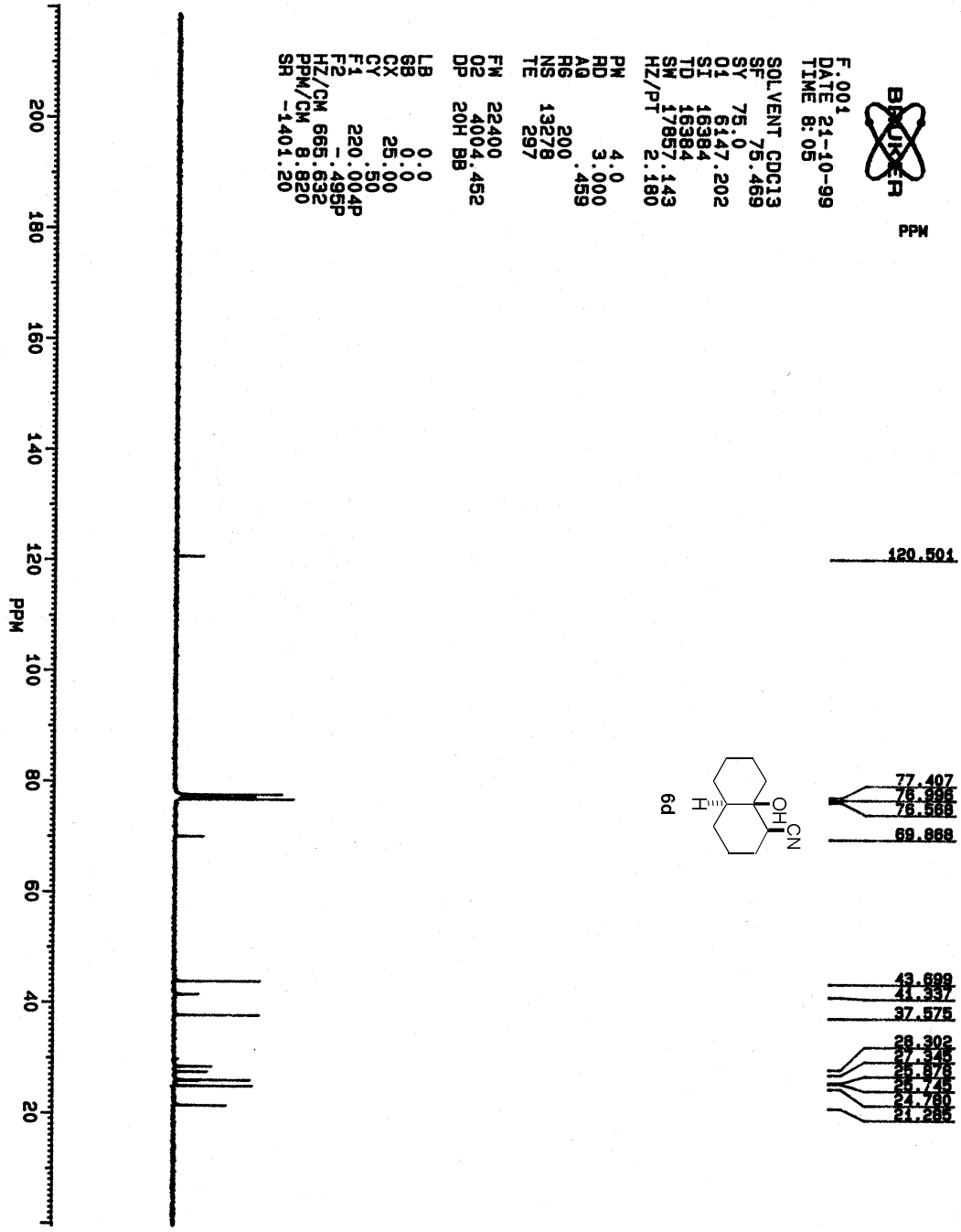
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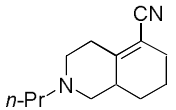
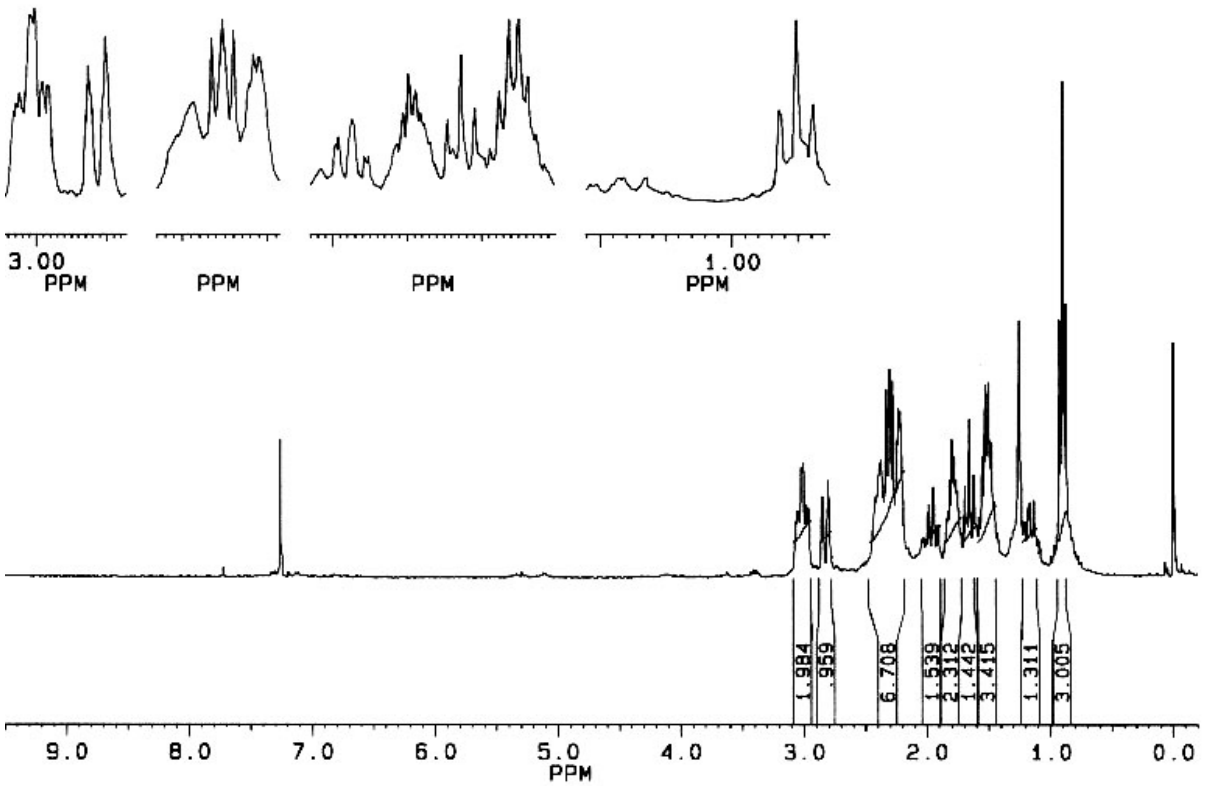
H
PPM



7e

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AQ        .909
RG       8
NS       8
TE     297

FW    5700
O2    5684.205
DP    4L PO

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GB       0.0
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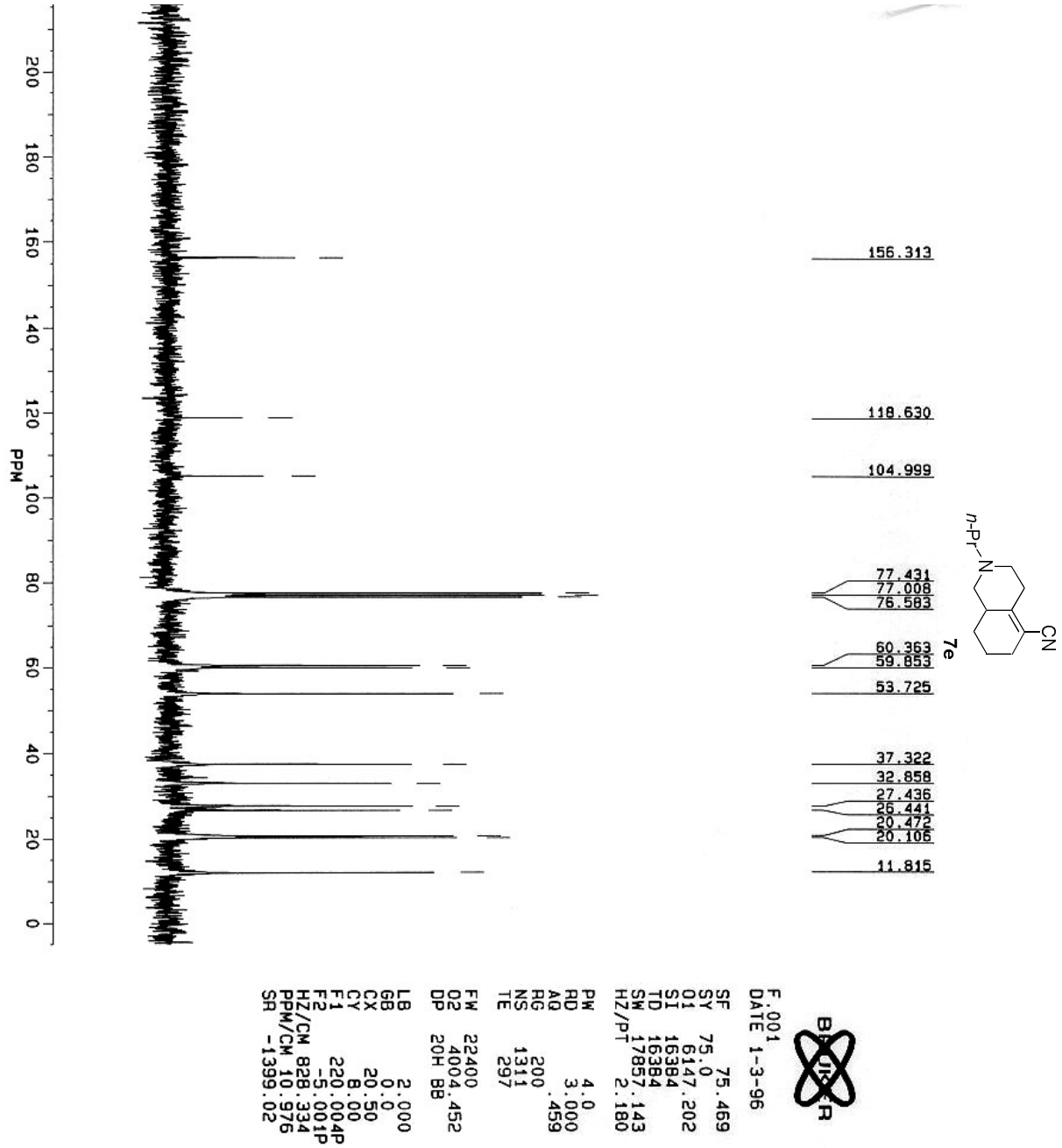
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n-Pr
N
CN
7e
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DATE 1-3-96
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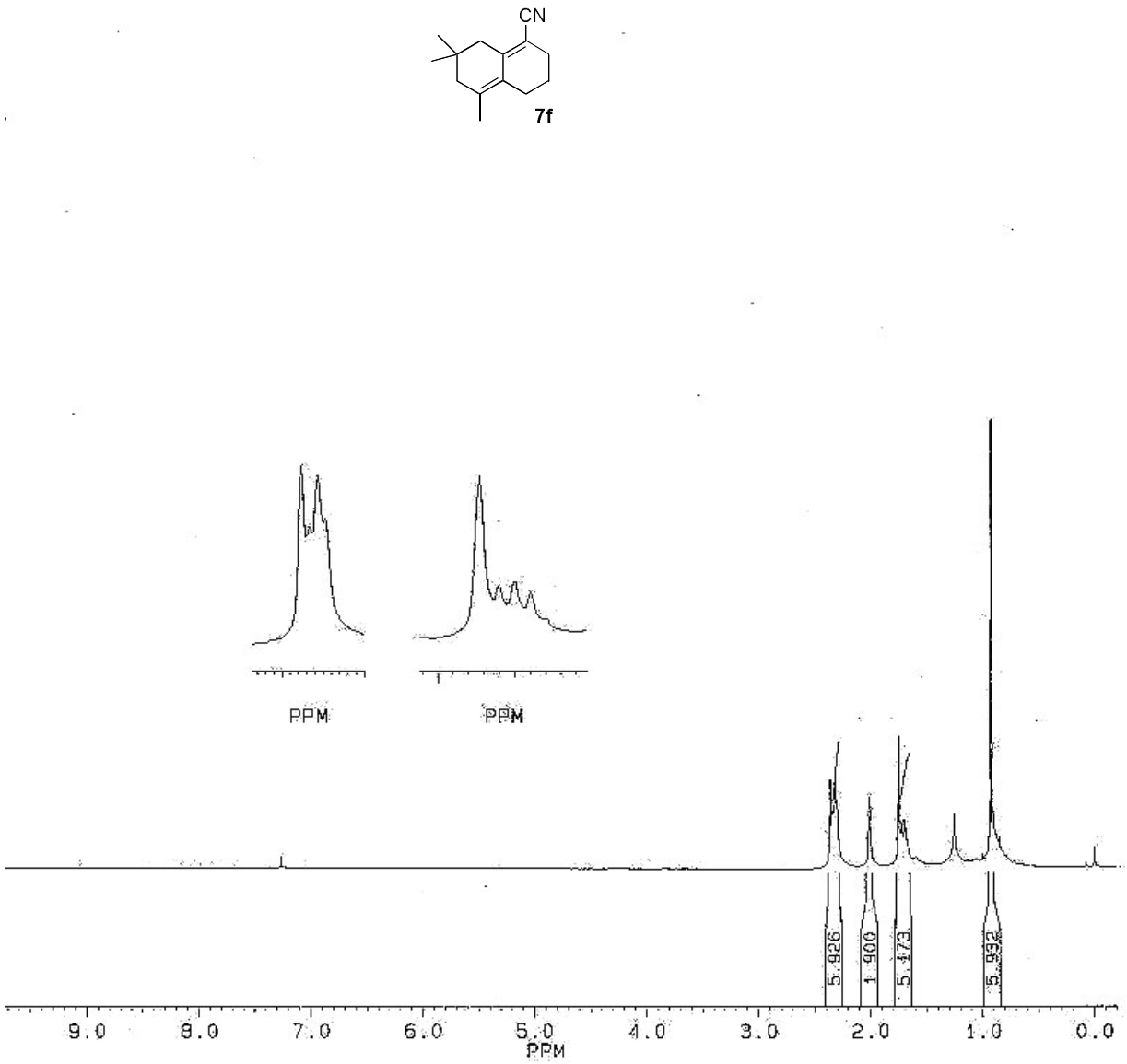

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SI 8192
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HZ/PT 1.100

PW 5.0
RD 2.500
AQ .909
RG 4
NS 32
TE 297

FW 5700
02 5684.205
DP 4L P0

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CY 8.00
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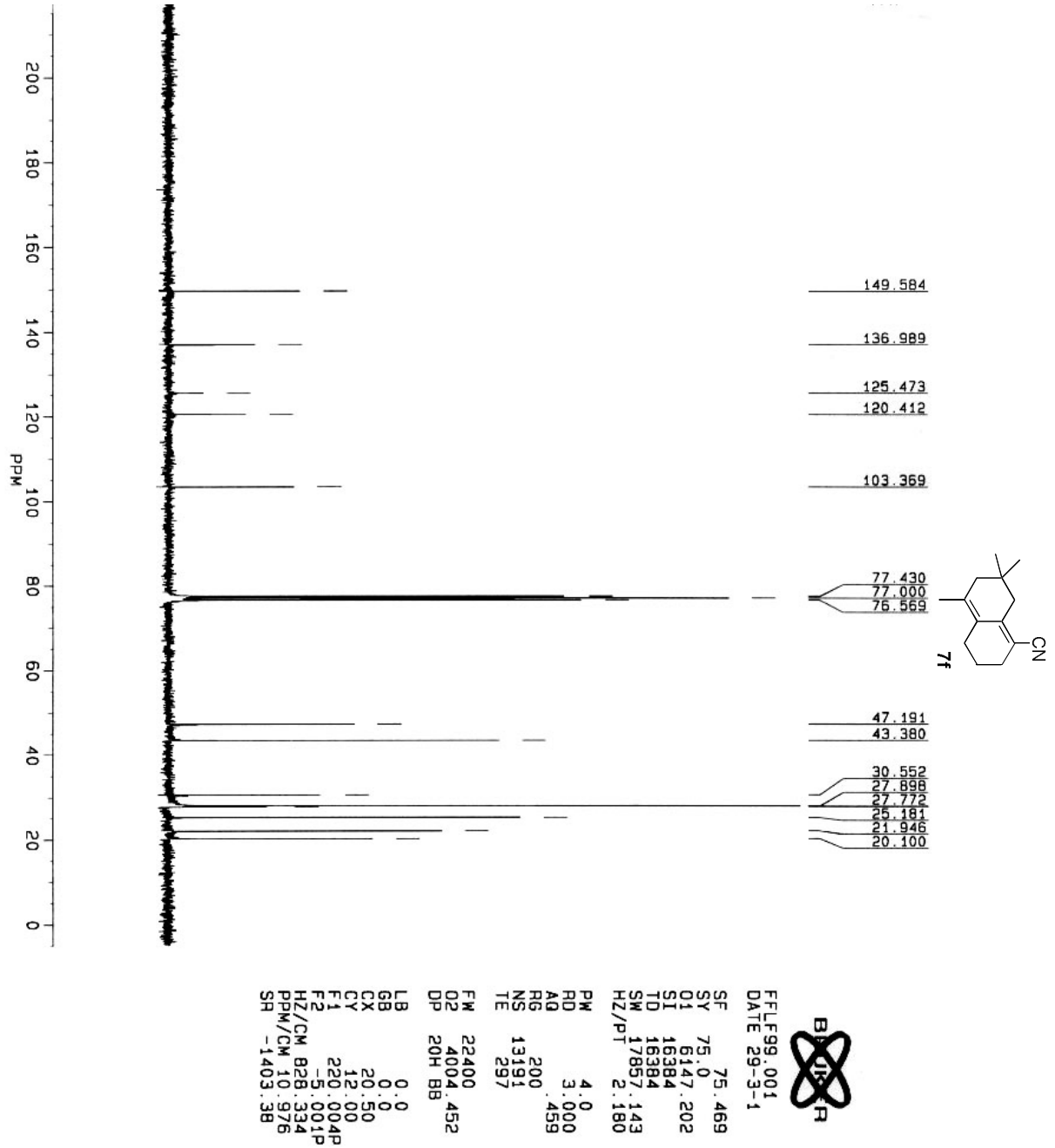
PPM/CM .498
SR 3367.75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CN
7f
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DATE 29-3-1
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TD 16384
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HZ/PT 2.180
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AQ .459
RG 200
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O2 4004.452
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LB 0.0
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F2 -5.001P
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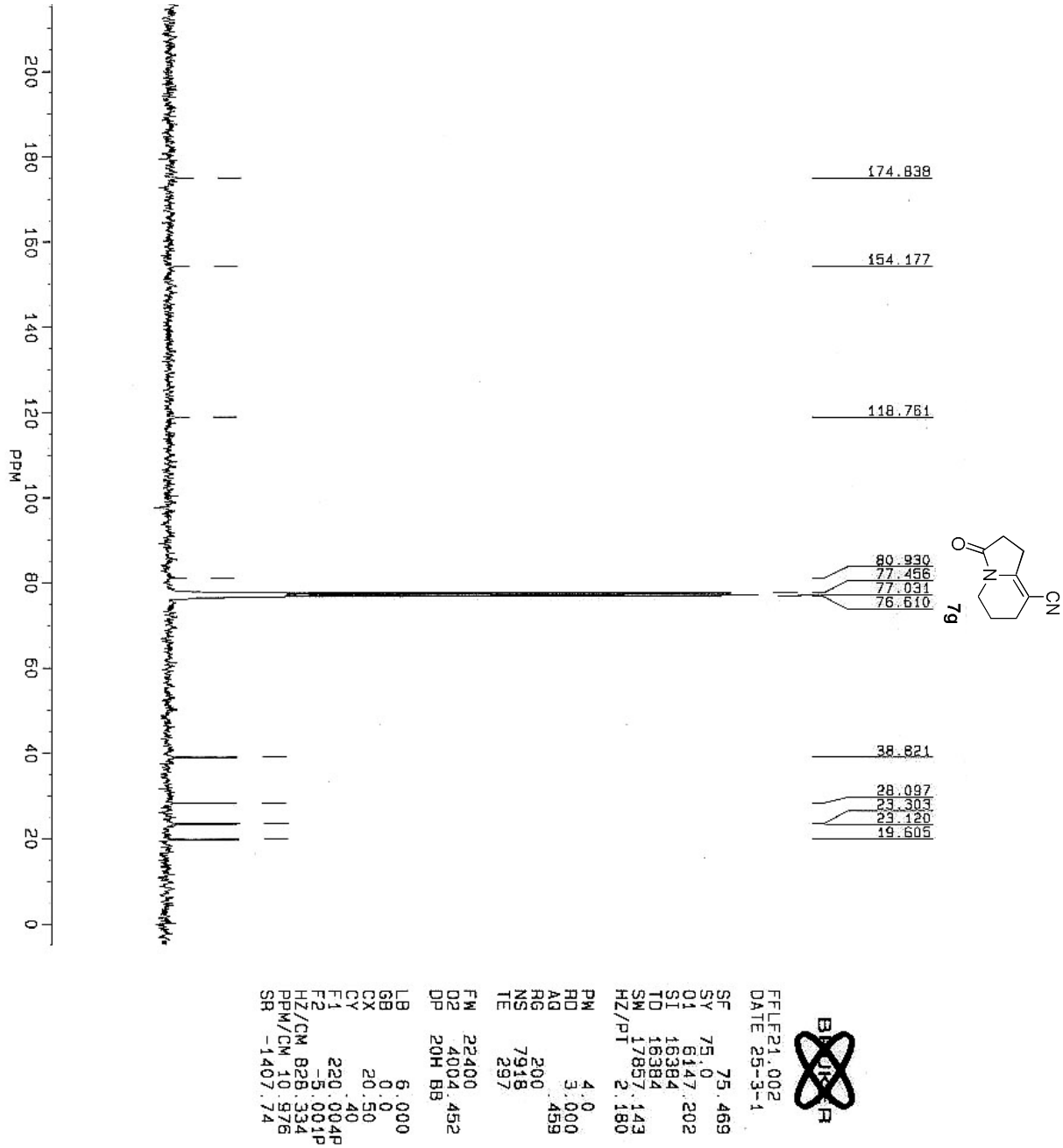
PPM/CM 10.976
SR -1403.38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CN
79
FFLE21.002
DATE 25-3-1
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TD 16384
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HZ/PT 2.180
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RD 3.000
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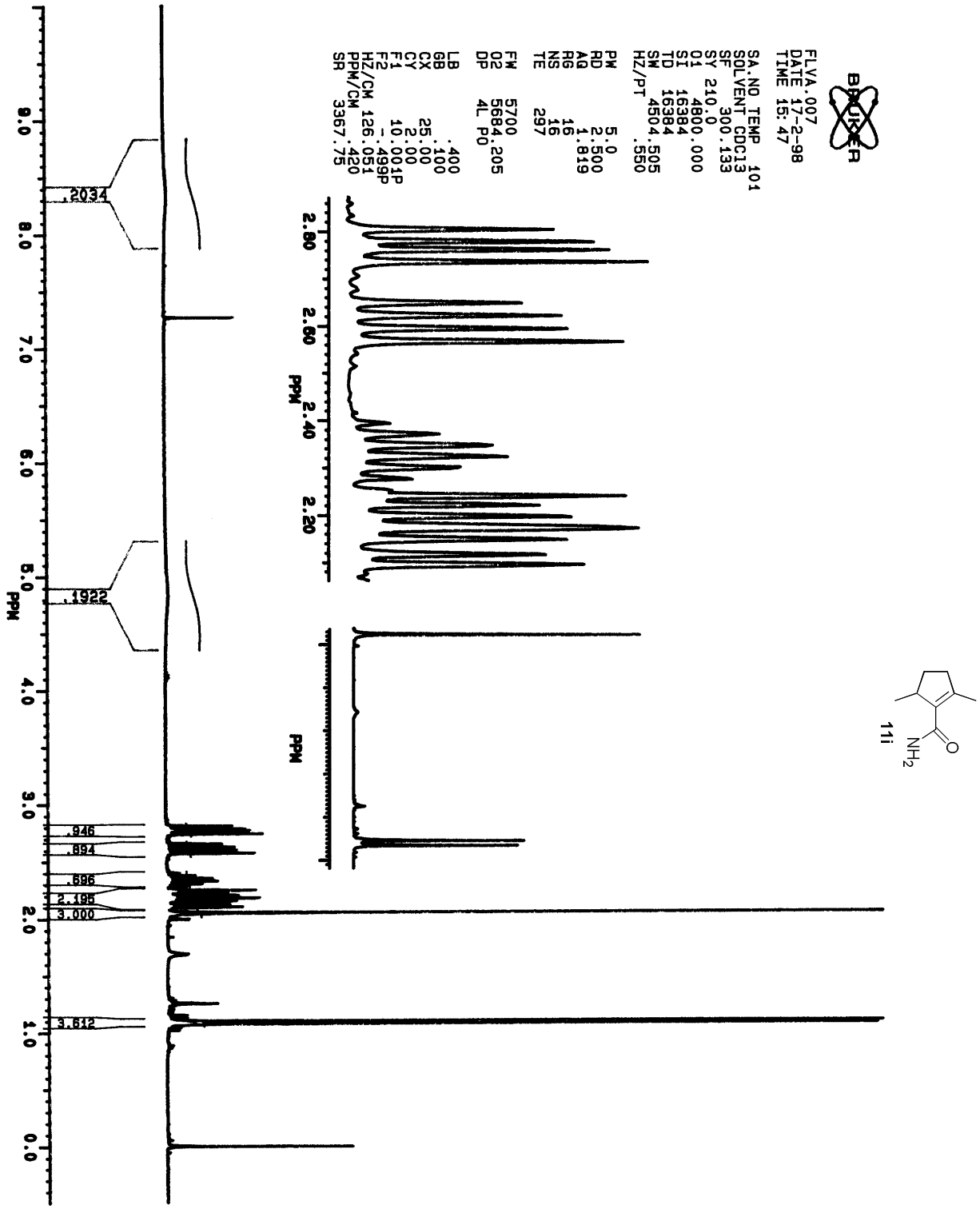
BRUKER
FLVA.007
DATE 17-2-98
TIME 15:47
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NS 16
TE 297
FW 5700
O2 5684.205
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CX 25.00
CY 2.000
F1 10.001P
F2 -.499P
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PPM/CM .420
SR 3367.75
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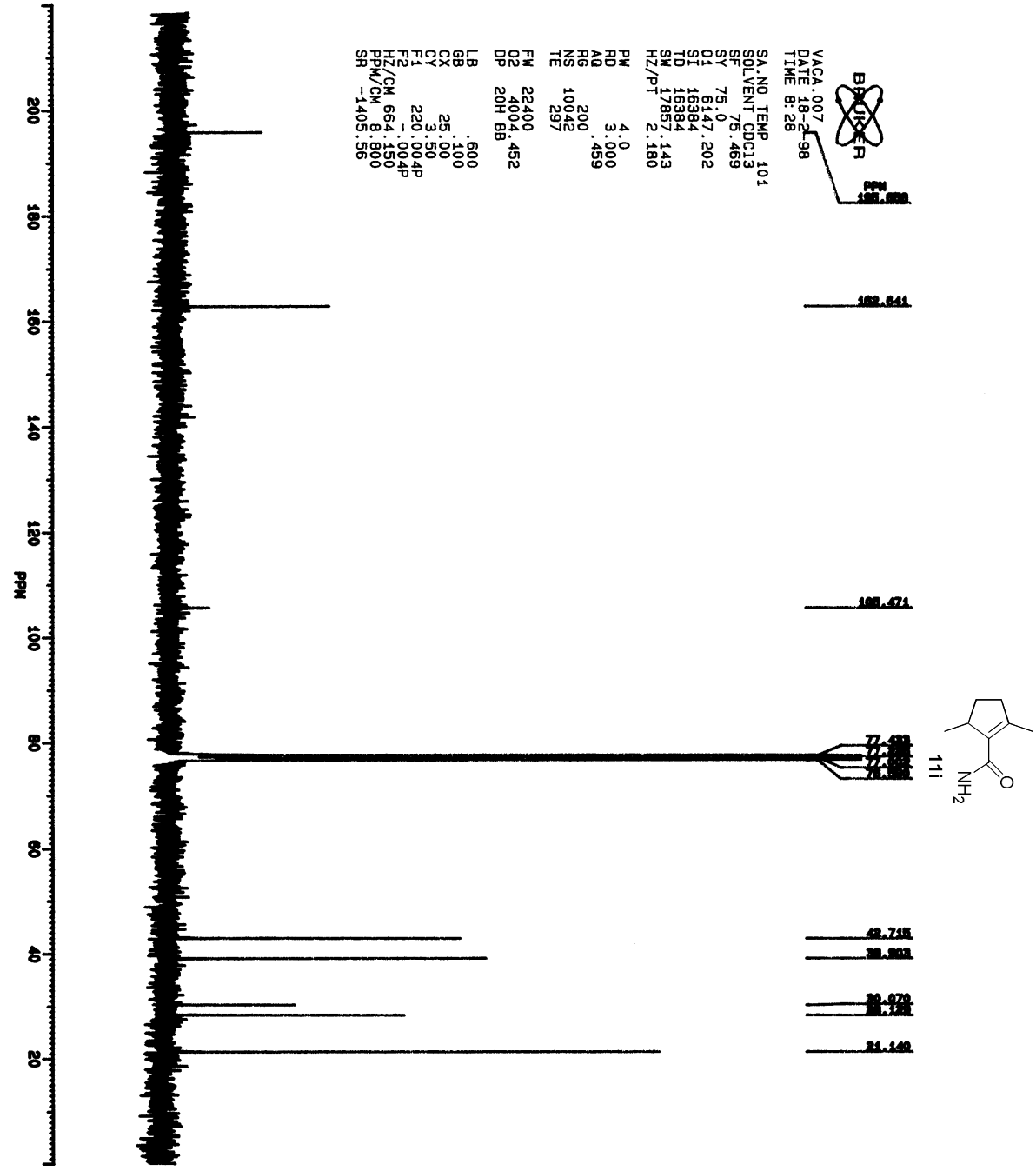
2.195
3.000
3.612
PPM
NH2
O
11i


BRUKER
PPM
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11i
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O
YACA.007
DATE 18-2-98
TIME 8:28
SA.NO TEMP 101
SOLVENT CDCl3
SF 75.469
SY 75.0
O1 6147.202
SI 16384
TD 16384
SW 17857.143
HZ/PT 2.180
PW 4.0
RD 3.000
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RG 200
NS 10042
TE 297
FW 22400
O2 4004.452
DP 20H BB
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GB .100
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CY 3.50
F1 220.0004P
F2 -.0004P
HZ/CM 664.150
PPM/CM 8.800
SR -1405.56
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180
160
140
120
100
80
60
40
20
PPM

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