Supporting Information

Cyclization and Ring-Expansion Processes Involving Samarium Diiodide Promoted Reductive Formation and Subsequent Oxidative Ring-Opening of Cyclopropanol Derivatives

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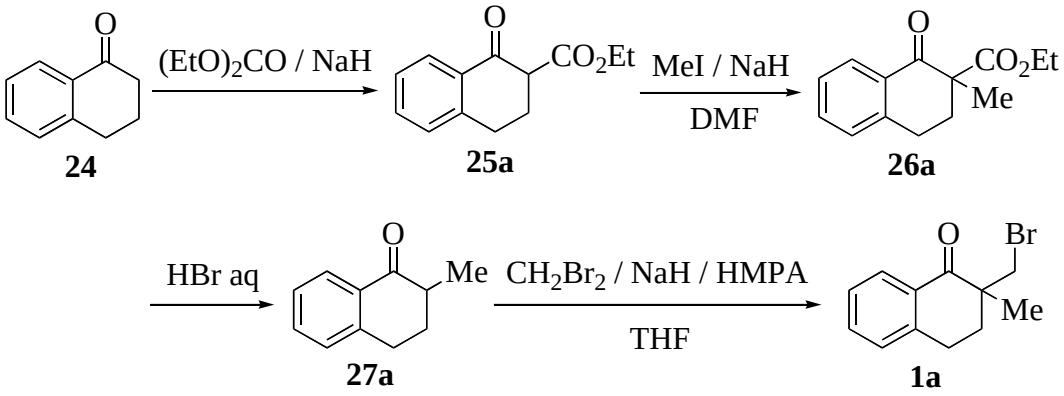
General Procedure. NMR spectra were recorded in $CDCl_3$ with Me_4Si as an internal standard at 200 MHz and 270 MHz for ^{1}H-NMR, and 50 MHz and 68 MHz for ^{13}C-NMR. Column chromatography was performed with silica gel (Wakogel C-200). Preparative TLC was performed on 20 cm x 20 cm plates coated with silica gel (Wakogel B-5F). Anhydrous DMF was purchased and used without distillation. MeCN was distilled over P_2O_5 and subsequently distilled with K_2CO_3. THF was distilled over sodium-benzophenone under N_2. Other reagents and solvents were purchased and used without further purification. Substrates (**1d**,[1] **2a**,[2] **2b**,[3] **2c**,[2] **2d**,[2] **3**,[4] and **4**[4]) and products (**6**,[3] **7a**,[2] **7c**,[2] **7d**,[2] **8a**,[2] **10**,[1] **11**,[5] and **23**[6]) are known compounds. Spectral data of **1a**, **1b**, **1c**, **1e**, **2e**, **3**,[4] **4**,[4] **5**, **7e**, **9a**, **11**,[5] **12**, **19a**, **22**, and **23**[6] are presented in the manuscript. Intensities of molecular ion peaks of low resolution mass spectrometry for **1b**, **1c**, **2e** and **22** were so weak that high resolution mass spectra of these compounds could not be measured.

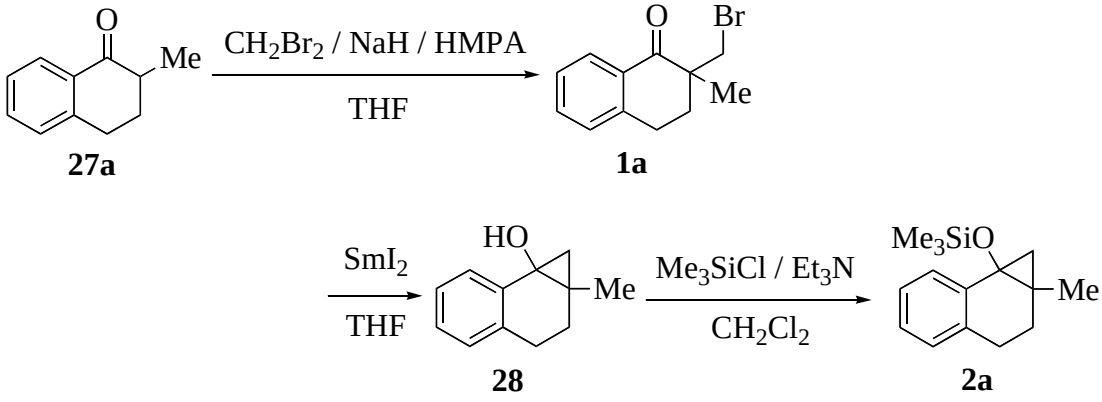
Preparation of substrates. Compound **1a** was prepared from 2-methyl-1-tetralone **27a**, that is obtained through several steps starting from 1-tetralone **24** (Scheme 1). Compounds **1b**, **1c**, **1d** and **1e** were similarly prepared. Procedure for the preparation of **1a** is described in the manuscript.

Scheme 1

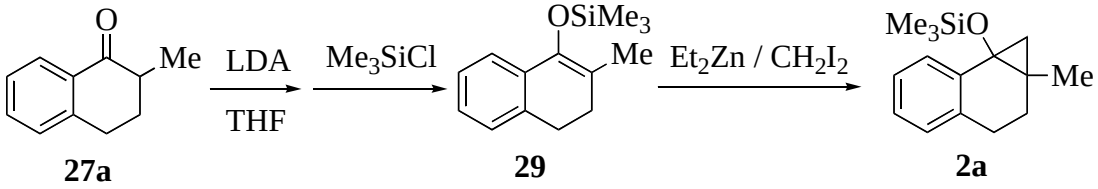


Compound **2a** was prepared from 2-methyl-1-tetralone **27a** either by method A (Scheme 2) or method B (Scheme 3), respectively. Compounds **2b**, **2c**, **2d** and **2e** were similarly prepared by method A. While Procedure of method A is reported in the manuscript, procedure of method B is described below.

Scheme 2 (method A)

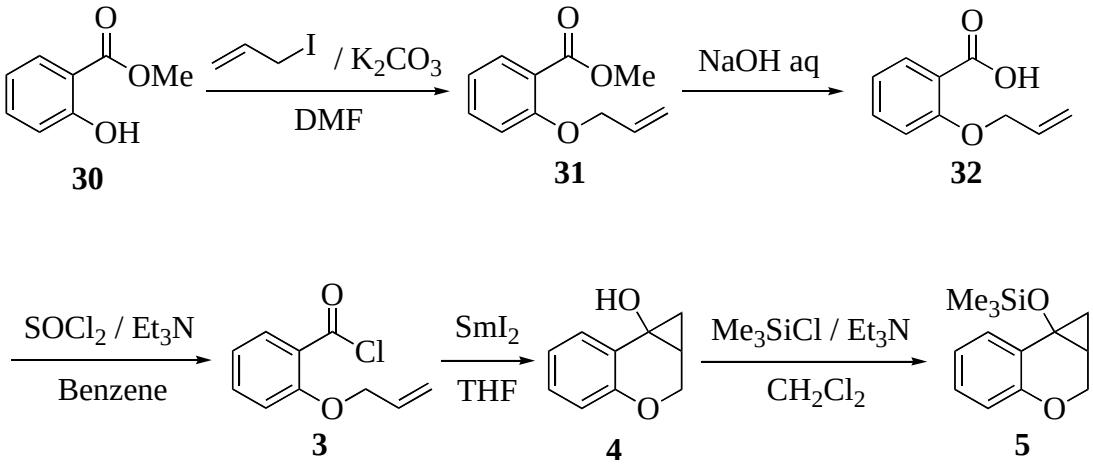


Scheme 3 (method B)



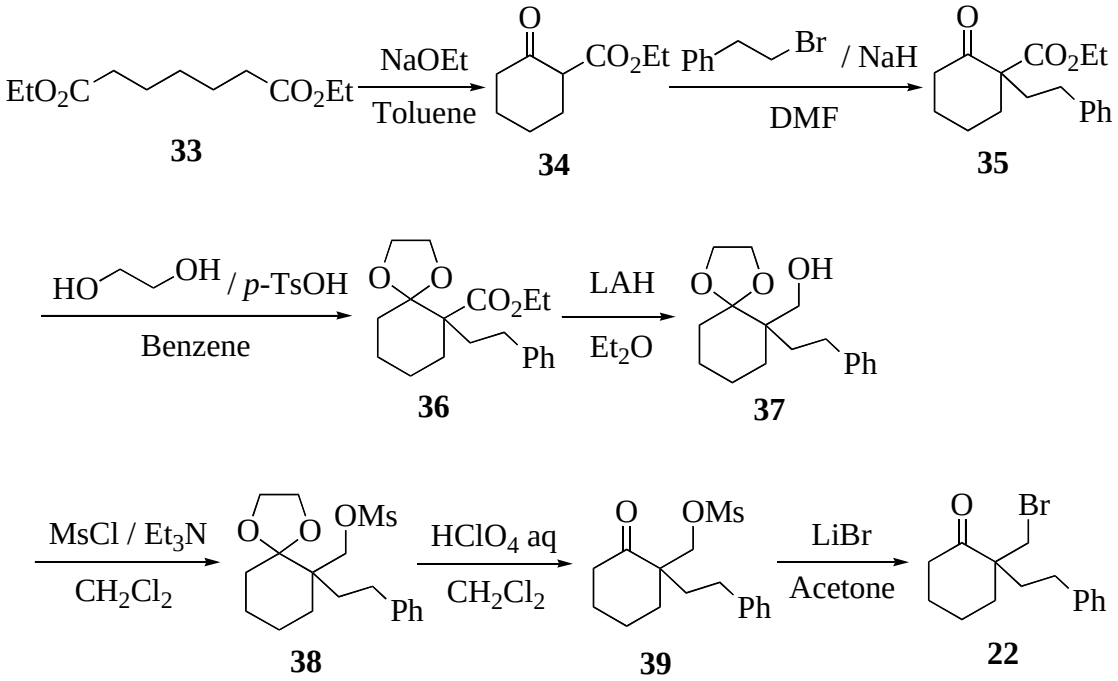
Compound **4** and **5** were prepared through several steps starting from methyl salicylate **30** (Scheme 4). Preparation procedures of **4** and **5** are reported in the manuscript.

Scheme 4



Compound **22** was prepared through several steps starting from diethyl heptanedioate **33** (Scheme 5). Preparation procedure of **22** is reported in the manuscript.

Scheme 5



Substrates (**25a-c**,[7,8] **25e**,[7] **26a-c**,[3,7] **26e**,[7] **27a**,[9] **27b**,[10] **29**,[11] **31**,[12] and **34**[13]) are known compounds. ^{1}H-NMR data of **25a**,[7] **26a**,[7] **26d**, **27a**,[9] **27c-e**, **29**,[11] **31**,[12] **32**, **34**,[13] **35**, **36**, **37**, **38**, and **39** are presented below.

Ethyl 1-tetralone-2-carboxylate (25a).[7] 1-Tetralone **24** (9.3 mL, 70 mmol) was added to the suspension of NaH (3.05 g, 84 mmol) in diethyl carbonate (53.4 mL). The mixture was refluxed under N_2 at 140℃ for 2h. Then, it was extracted with Et_2O after addition of 2N aqueous HCl. The extract was treated with brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated, and diethyl carbonate was removed by distillation under reduced pressure. The residue was distilled

under reduced pressure to give Ethyl 1-tetralone-2-carboxylate **25a** (2.20 g, 56 mmol, 80%). Data for **25a**: yellow oil; ^{1}H-NMR (270 MHz) δ 1.29 (m, 3H), 2.30-2.56 (m, 2H), 2.58 (t, *J* = 8.1 Hz, 2H, enol form), 2.82 (t, *J* = 8.1 Hz, 2H, enol form), 3.04 (m, 2H), 3.60 (dd, *J* = 8.1, 4.1 Hz, 1H), 4.29 (m, 2H), 7.16 (m, 1H, enol form), 7.22-7.38 (m, 2H), 7.51 (m, 1H), 7.80 (m, 1H, enol form), 8.06 (m, 1H), 12.48 (s, 1H, enol form).

Ethyl 2-methyl-1-tetralone-2-carboxylate (26a).[7] Ethyl 1-tetralone-2-carboxylate **25a** (2.00 g, 9.16 mmol) in DMF (4.0 mL) was added to the suspension of NaH (475.4 mg, 13 mmol) in DMF (5.0 mL). The mixture was stirred under N_2 at room temperture. After 1h, methyl iodide (2.0 mL, 32 mmol) was added, and the mixture was stirred for 2h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $Na_2S_2O_3$, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated to give Ethyl 2-methyl-1-tetralone-2-carboxylate **26a** (2.15 g, 9.16 mmol, 100%). Data for **26a**: yellow oil; ^{1}H-NMR (270 MHz) δ 1.17 (t, *J* = 8.1 Hz, 3H), 1.50 (s, 3H), 2.04 (m, 1H), 2.60 (m, 1H), 2.87-3.13 (m, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 7.18-7.35 (m, 2H), 7.48 (m, 1H), 8.05 (m, 1H). Data for **26d**: pale yellow oil; ^{1}H-NMR (270 MHz) δ 1.18 (t, *J* = 7.3 Hz, 3H), 1.92-2.26 (m, 5H), 2.59 (m, 1H), 2.87-2.99 (m, 1H), 3.03-3.16 (m, 1H), 4.15 (q, *J* = 7.7 Hz, 2H), 4.93-5.11 (m, 2H), 5.82 (m, 1H), 7.18-7.37 (m, 2H), 7.48 (m, 1H), 8.04 (m, 1H).

2-Methyl-1-tetralone (27a).[9] Ethyl 2-methyl-1-tetralone-2-carboxylate **26a** (3.26 g, 14.9 mmol) and 48% aqueous HBr (19.0 mL, 168 mmol) was refluxed at 120℃ for 2h. The reaction was quenched by water, and extracted with Et_2O. The combined organic extracts were washed with water, and brine. The ether solution was dried over $MgSO_4$, and the filtrate was concentrated to give 2-Methyl-1-tetralone **27a** (1.88 g, 11.8 mmol, 79%). Data for **27a**: yellow oil; ^{1}H-NMR (270 MHz) δ 1.27 (d, *J* = 8.1 Hz, 3H), 1.81-1.97 (m, 1H), 2.14-2.26 (m, 1H), 2.52-2.67 (m, 1H), 2.90-3.11 (m, 2H), 7.18-7.33 (m, 2H), 7.46 (m, 1H), 8.03 (m, 1H). Data for **27c**: brown oil; ^{1}H-NMR (270 MHz) δ 1.23 (d, *J* = 6.8 Hz, 3H), 1.52-1.80 (m, 2H), 1.84-2.15 (m, 2H), 2.85-3.09 (m, 3H), 7.16-7.31 (m, 2H), 7.38 (m, 1H), 7.67 (m, 1H). Data for **27d**: colorless oil; ^{1}H-NMR (270 MHz) δ 1.49-1.63 (m, 1H), 1.80-1.98 (m, 1H), 2.02-2.35 (m, 4H), 2.45-2.58 (m, 1H), 3.00 (t, *J* = 7.0 Hz, 2H), 4.93-5.15 (m, 2H), 5.84 (m, 1H), 7.18-7.37 (m, 2H), 7.46 (m, 1H), 8.02 (m, 1H). Data for **27e**: yellow oil; ^{1}H-NMR (200 MHz) δ 1.48 (d, *J* = 7.0 Hz, 3H), 2.39-2.73 (m, 2H), 2.98-3.23 (m, 2H), 3.54 (q, J = 7.1 Hz, 1H), 7.17-7.29 (m, 4H).

2-Methyl-1-trimethylsilyloxy-3,4-dihydronaphthalene (29).[11] *n*-BuLi (1.6 M in hexane, 5.9 mL, 9.4 mmol) was added to the diisopropylamine (1.47 mL, 10.5 mmol) in THF (22.5 mL) at 0℃. The mixture was stirred under N_2 at 0℃ for 1h, then cooled at -78℃. 2-Methyl-1-tetralone **27a** (1.26 g, 7.9 mmol) in THF (7.5 mL) was added, and the mixture was stirred at 0℃ for 30 min. The mixture was cooled at -78℃, Me_3SiCl (4.0 mL, 31.3 mmol) was added, and stirred under N_2 at room temperture. After 2h, Et_3N and phosphate buffer was added, and extracted with Et_2O. The extract was treated with water, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated to give 2-Methyl-1-trimethylsilyloxy-3,4-dihydronaphthalene **29** (1.76 g, 7.6 mmol, 96%). Data for **29**: blown oil; ^{1}H-NMR (270 MHz) δ 0.21 (s, 9H), 1.81 (s, 3H), 2.26 (t, *J* = 8.1 Hz, 2H), 2.73 (t, *J* = 8.1 Hz, 2H), 7.06-7.21 (m, 3H), 7.30 (m, 1H).

6-Methyl-1-trimethylsilyloxy-2,3-benzobicyclo[4.1.0]hepta-2-ene (2a) (method B). The flask was flame-dried for 10 min, then 2-Methyl-1-trimethylsilyloxy-3,4-dihydronaphthalene **29** (1.76 g, 7.6 mmol), Et_2O (7.0 mL), Et_2Zn (1.0 M in hexane, 17.2 mL, 17.2 mmol), and CH_2I_2 (1.39 mL, 17.2 mmol) was added, and the mixture was stirred under N_2 at room temperture. After 26h, saturated aqueous NH_4Cl was added, and extracted with Et_2O. The extract was treated with water, brine, and dried over anhydrous $MgSO_4$. The residue obtained after concentration was subjected to column chromatography on silica gel (CH_2Cl_2 / *n*-Hexane = 1 / 5) to give 6-Methyl-1-trimethylsilyloxy-2,3-benzobicyclo[4.1.0]hepta-2-ene **2a** (841.9 mg, 3.4 mmol, 45%).

Methyl 2-allyloxybenzoate (31).[12] Methyl salicylate **30** (4.56 g, 30.0 mmol) in DMF (12.0 mL) and allyl iodide (3.0 mL, 33 mmol) was added to the suspension of K_2CO_3 (8.33 g, 60.0 mmol) in DMF (18.0 mL). The mixture was stirred under N_2 at room temperture for 70h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $Na_2S_2O_3$, saturated aqueous $NaHCO_3$, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated to give Methyl 2-allyloxybenzoate **31** (5.67 g, 29.5 mmol, 98%). Data for **31**: yellow oil; ^{1}H-NMR (270 MHz) δ 3.89 (s, 3H), 4.63 (m, 2H), 5.30 (m, 1H), 5.52 (m, 1H), 6.08 (m, 1H), 6.97 (m, 2H), 7.44 (m, 1H), 7.80 (m, 1H).

2-Allyloxybenzoic acid (32). Methyl 2-allyloxybenzoate **31** (3.00 g, 15.6 mmol) and 20% aqueous NaOH (25.7 mL, 156 mmol) was refluxed at 120℃ for 3h. Then, it was extracted with Et_2O after addition of concentrated aqueous HCl. The combined organic extracts were washed with water, and brine, and dried over $MgSO_4$. The filtrate was concentrated and recrystallized from EtOH to give 2-Allyloxybenzoic acid **32** (1.67 g, 9.4 mmol, 60%). Data for **32**: white solid, m.p. 45.2-45.8℃; ^{1}H-NMR (270 MHz) δ 4.80 (d, *J* = 8.1 Hz, 2H), 5.47 (m, 2H), 6.12 (m, 1H), 7.03 (m, 1H), 7.14 (m, 1H), 7.54 (m, 1H), 8.19 (m, 1H).

Ethyl 2-oxocyclohexane-1-carboxylate (34).[13] Diethyl heptanedioate **33** (5.5 mL, 25 mmol) was added to the suspension of NaOEt (2.55 g, 37.5 mmol) in toluene (20 mL). The mixture was refluxed under N_2 at 120℃ for 14h. Then, it was extracted with Et_2O after addition of 2N aqueous HCl. The extract was treated with saturated aqueous $NaHCO_3$, water, and brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated, and distilled under reduced pressure to give Ethyl 2-oxocyclohexane-1-carboxylate **34** (1.62 g, 9.54 mmol, 38%). Data for **34**: colorless oil; ^{1}H-NMR (270 MHz) δ 1.16-1.50 (m, 3H), 1.54-2.64 (m, 8H), 3.38 (m, 1H), 4.09-4.32 (m, 2H), 12.24 (s, 1H, enol form).

Ethyl 2-phenethylcyclohexanone-2-carboxylate (35). Ethyl 2-oxocyclohexane-1-carboxylate **34** (775.6 mg, 4.56 mmol) in DMF (3.0 mL) was added to the suspension of NaH (218.9 mg, 5.47 mmol) in DMF (2.0 mL). The mixture was stirred under N_2 at room temperture. After 1h, 2-phenethyl bromide (2.46 mL, 18.2 mmol) was added, and the mixture was stirred for 71h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $Na_2S_2O_3$, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated, and 2-phenethyl bromide was removed by distillation under reduced pressure. The residue was subjected to column chromatography on silica gel (EtOAc / *n*-Hexane = 1 / 5) to give Ethyl 2-phenethylcyclohexanone-2-carboxylate **35** (704.8 mg, 2.57 mmol, 56%). Data for **35**: pale yellow

oil; ^{1}H-NMR (270 MHz) δ 1.29 (t, *J* = 6.8 Hz, 3H), 1.47-1.93 (m, 6H), 1.97-2.08 (m, 1H), 2.17 (td, *J* = 10.8, 2.7 Hz, 1H), 2.40-2.69 (m, 4H), 4.22 (q, *J* = 7.2 Hz, 2H), 7.14-7.33 (m, 5H).

Ethyl 6-phenethyl-1,4-dioxaspiro[4.5]decane-6-carboxylate (36). Ethyl 2-phenethylcyclohexanone-2-carboxylate **35** (899.1 mg, 3.28 mmol) in Benzene (6.0 mL) and 1,2-ethanediol (0.55 mL, 9.84 mmol) was added to *p*-TsOH･H_2O (31.2 mg, 0.16 mmol) in Benzene (1.0 mL). The mixture was refluxed under N_2 at 90℃ for 19h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $NaHCO_3$, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated to give Ethyl 6-phenethyl-1,4-dioxaspiro[4.5]decane-6-carboxylate **36** (1.13 g, 3.28 mmol, 100%). Data for **36**: pale yellow oil; ^{1}H-NMR (270 MHz) δ 1.31 (t, *J* = 8.1 Hz, 3H), 1.48-1.70 (m, 6H), 1.80-1.93 (m, 2H), 2.06-2.17 (m, 1H), 2.29-2.61 (m, 3H), 3.92 (m, 4H), 4.21 (m, 2H), 7.12-7.32 (m, 5H).

6-Hydroxymethyl-6-phenethyl-1,4-dioxaspiro[4.5]decane (37). $LiAlH_4$ (373.4 mg, 9.84 mmol) was added to Ethyl 6-phenethyl-1,4-dioxaspiro[4.5]decane-6-carboxylate **36** (1.13 g, 3.28 mmol) in Et_2O (45.0 mL). The mixture was stirred under N_2 at room temperture for 0.5h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $NaHCO_3$, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated to give 6-Hydroxymethyl-6-phenethyl-1,4-dioxaspiro[4.5]decane **37** (827.2 mg, 2.99 mmol, 91%). Data for **37**: colorless oil; ^{1}H-NMR (270 MHz) δ 1.40-1.81 (m, 9H), 1.96 (td, *J* = 12.8, 2.7 Hz, 3H), 2.45-2.72 (m, 2H), 3.51 (d, *J* = 8.8 Hz, 1H), 3.92 (m, 1H), 3.96 (m, 4H), 7.12-7.36 (m, 5H).

6-Mesyloxymethyl-6-phenethyl-1,4-dioxaspiro[4.5]decane (38). Et_3N (1.38 mL, 9.84 mmol) and MsCl (0.61 mL, 7.87 mmol) was added to 6-Hydroxymethyl-6-phenethyl-1,4-dioxaspiro[4.5]decane **36** (827.2 mg, 2.99mmol) in CH_2Cl_2 (10.0 mL). The mixture was stirred at room temperture for 5h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $NaHCO_3$, brine, and dried over anhydrous $MgSO_4$. The residue obtained after concentration was subjected to column chromatography on silica gel (CH_2Cl_2) to give 6-Mesyloxymethyl-6-phenethyl-1,4-dioxaspiro[4.5]decane **38** (1.08 g, 2.99 mmol, 100%). Data for **38**: pale yellow oil; ^{1}H-NMR (270 MHz) δ 1.45-1.85 (m, 9H), 1.96-2.10 (m, 1H), 2.56-2.81 (m, 2H), 3.03 (s, 3H), 3.95 (m, 4H), 4.26 (d, *J* = 8.8 Hz, 1H), 4.45 (d, *J* = 8.8 Hz, 1H) 7.14-7.35 (m, 5H).

2-Mesyloxymethyl-2-phenethyl-1-cyclohexanone (39). 60% aqueous $HClO_4$ (1.0 mL) and water (1.0 mL) was added to 6-Mesyloxymethyl-6-phenethyl-1,4-dioxaspiro[4.5]decane **38** (1.08 g, 2.99 mmol) in CH_2Cl_2 (5.0 mL). The mixture was stirred at room temperture for 15h. Then, it was extracted with Et_2O after addition of water. The extract was treated with water, saturated aqueous $NaHCO_3$, brine, and dried over anhydrous $MgSO_4$. The filtrate was concentrated to give 2-Mesyloxymethyl-2-phenethyl-1-cyclohexanone **39** (914.9 mg, 2.95 mmol, 97%). Data for **39**: yellow oil; ^{1}H-NMR (270 MHz) δ 1.65-2.18 (m, 8H), 2.25-2.70 (m, 4H), 3.06 (s, 3H), 4.35 (dd, *J* = 16.9, 8.8 Hz, 2H), 7.11-7.33 (m, 5H).

Reference

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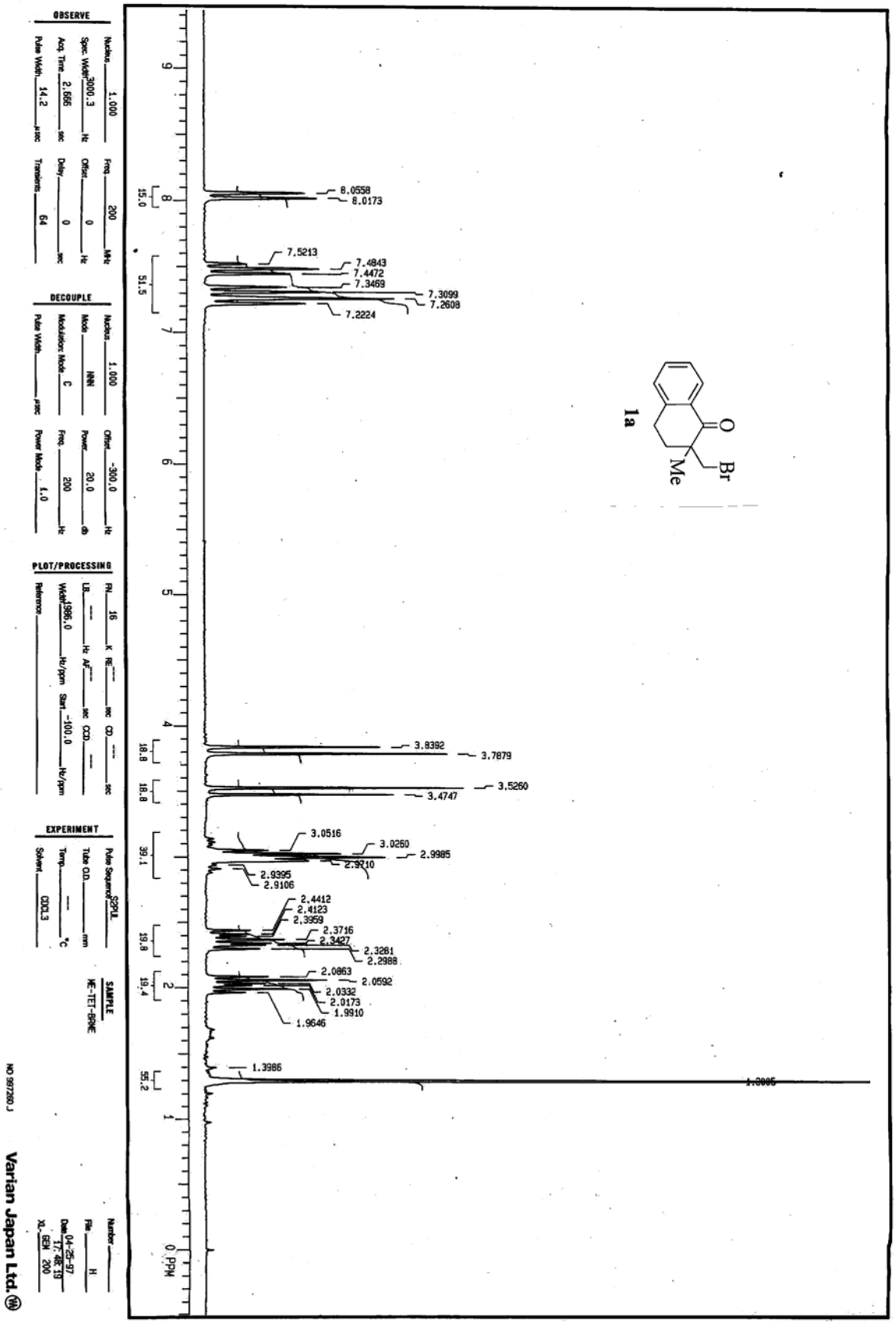
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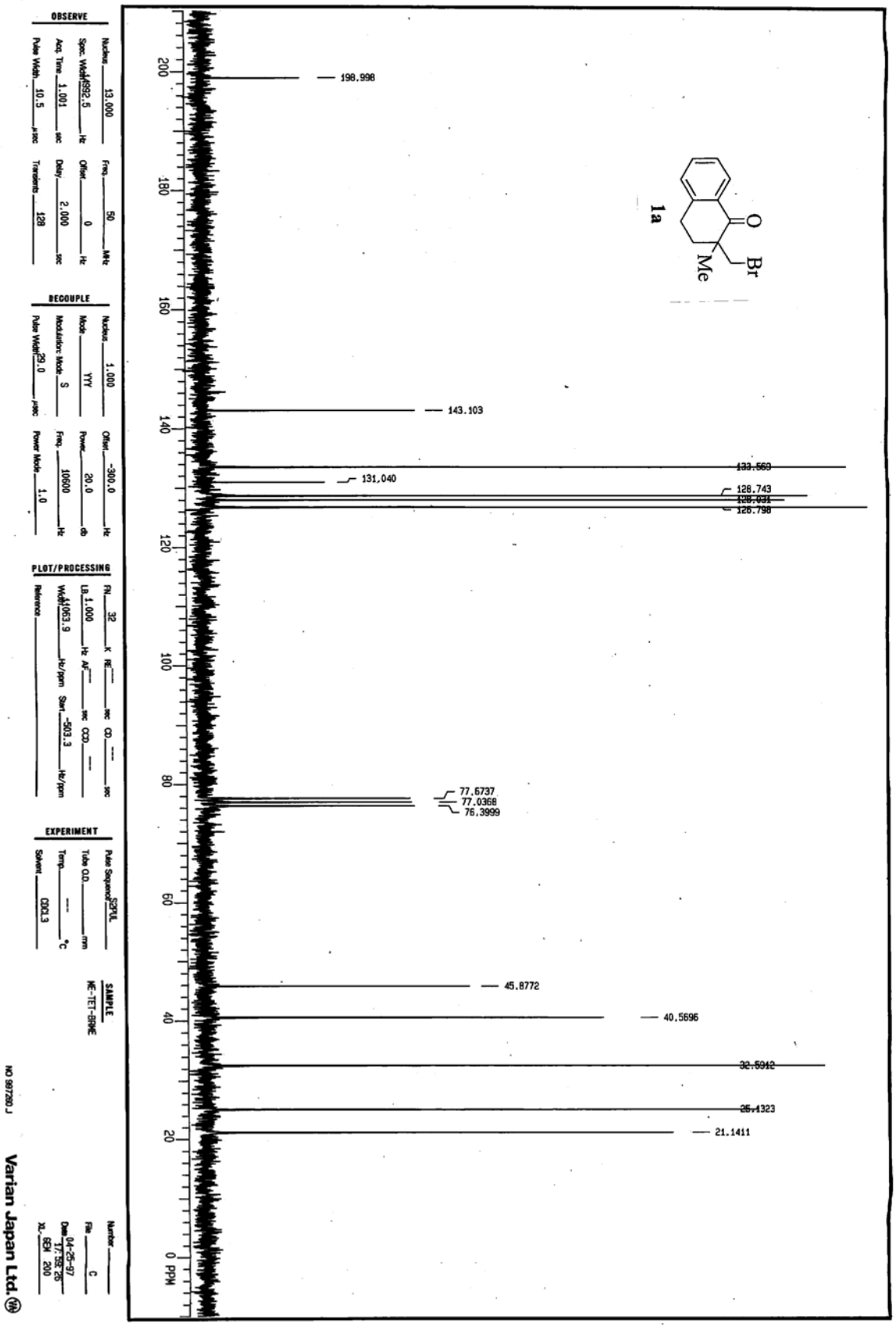
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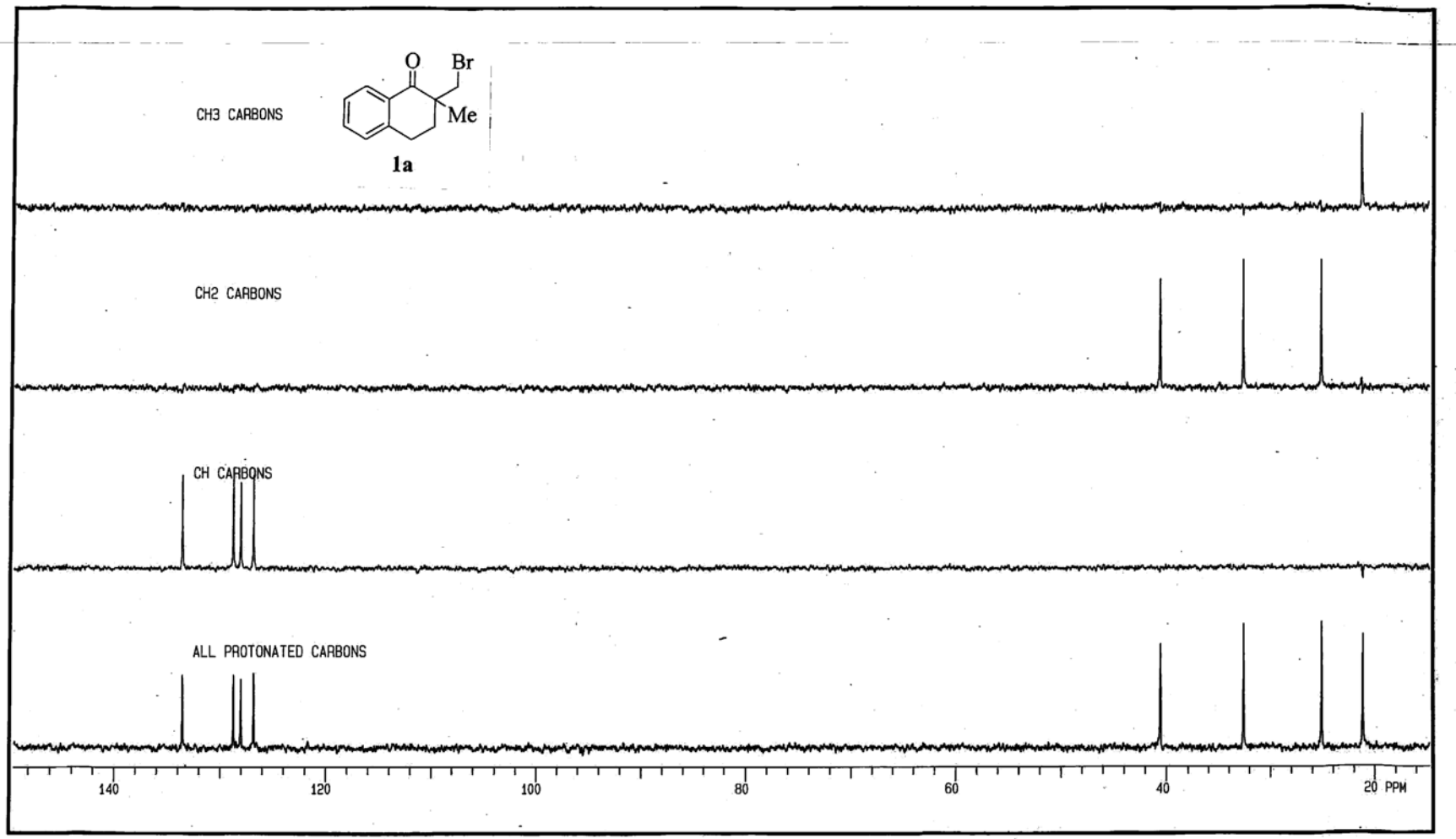
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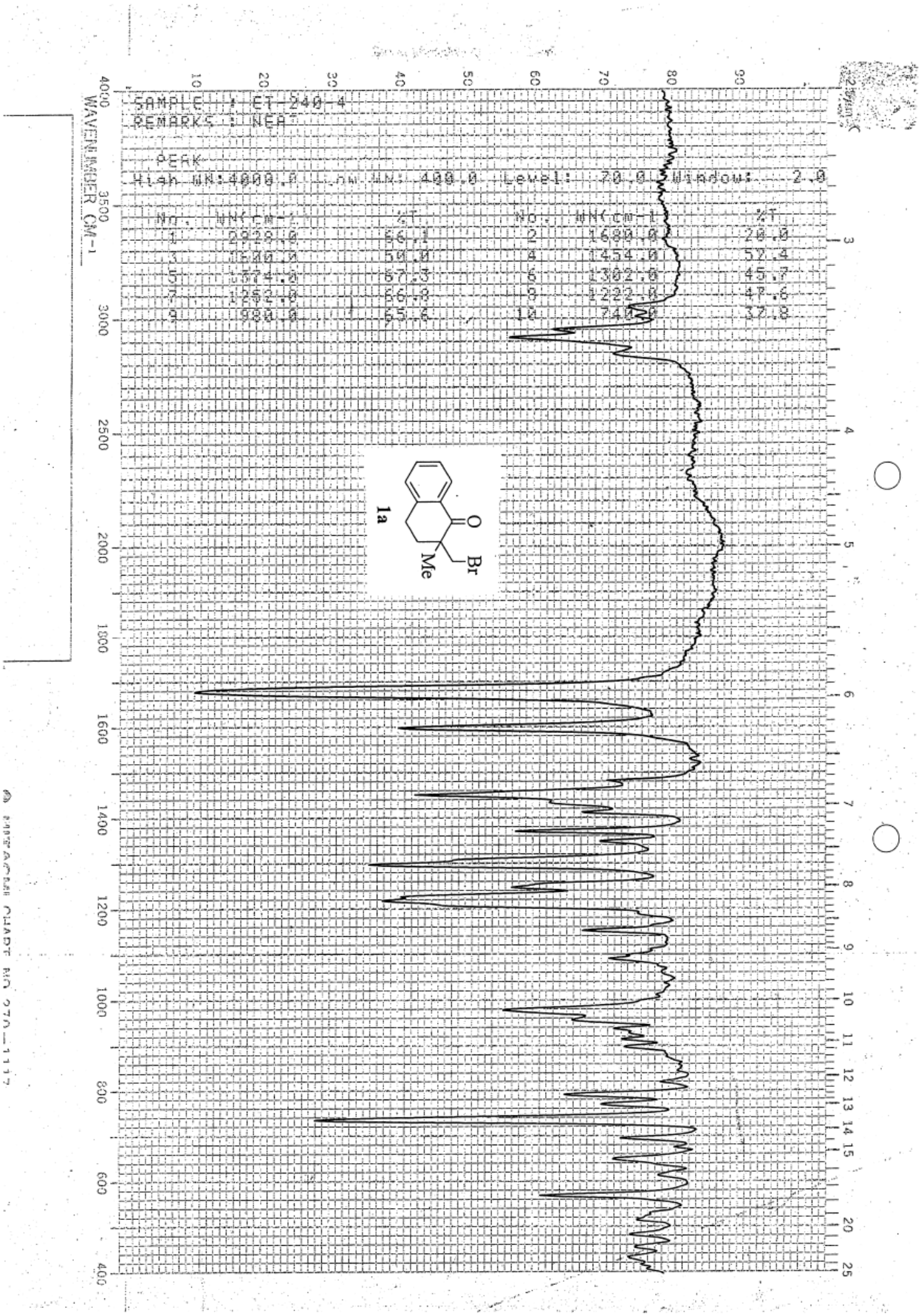
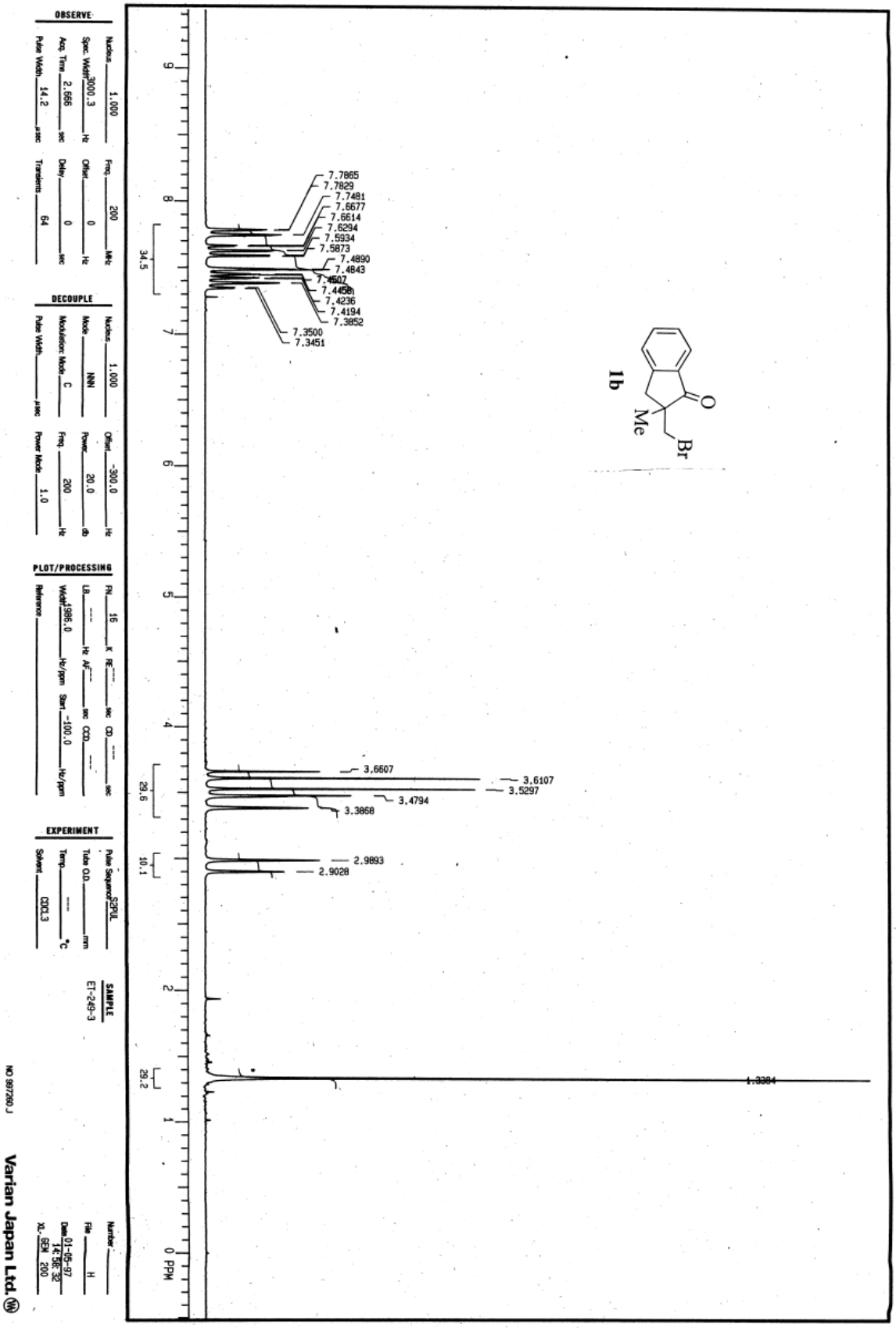
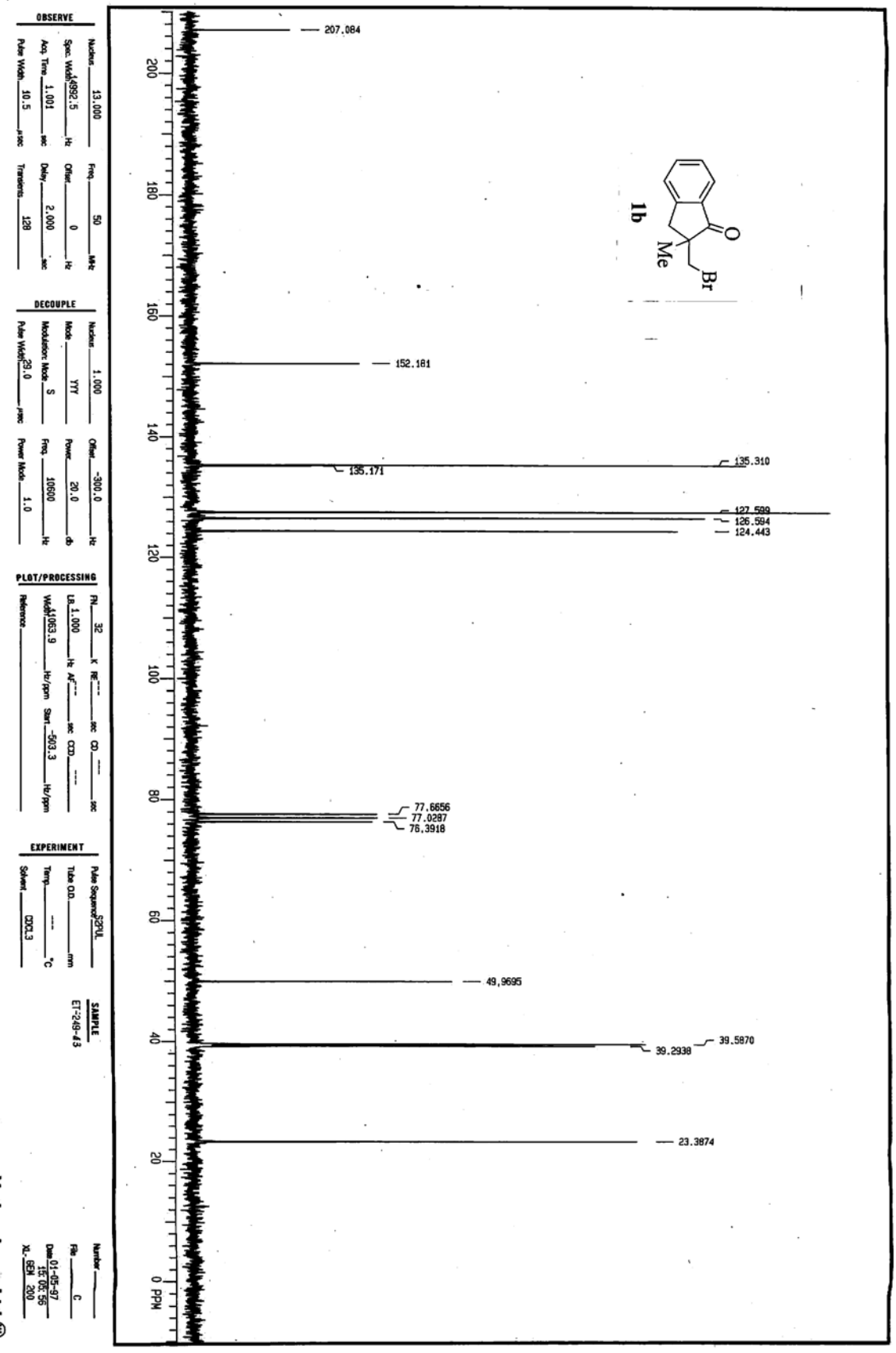
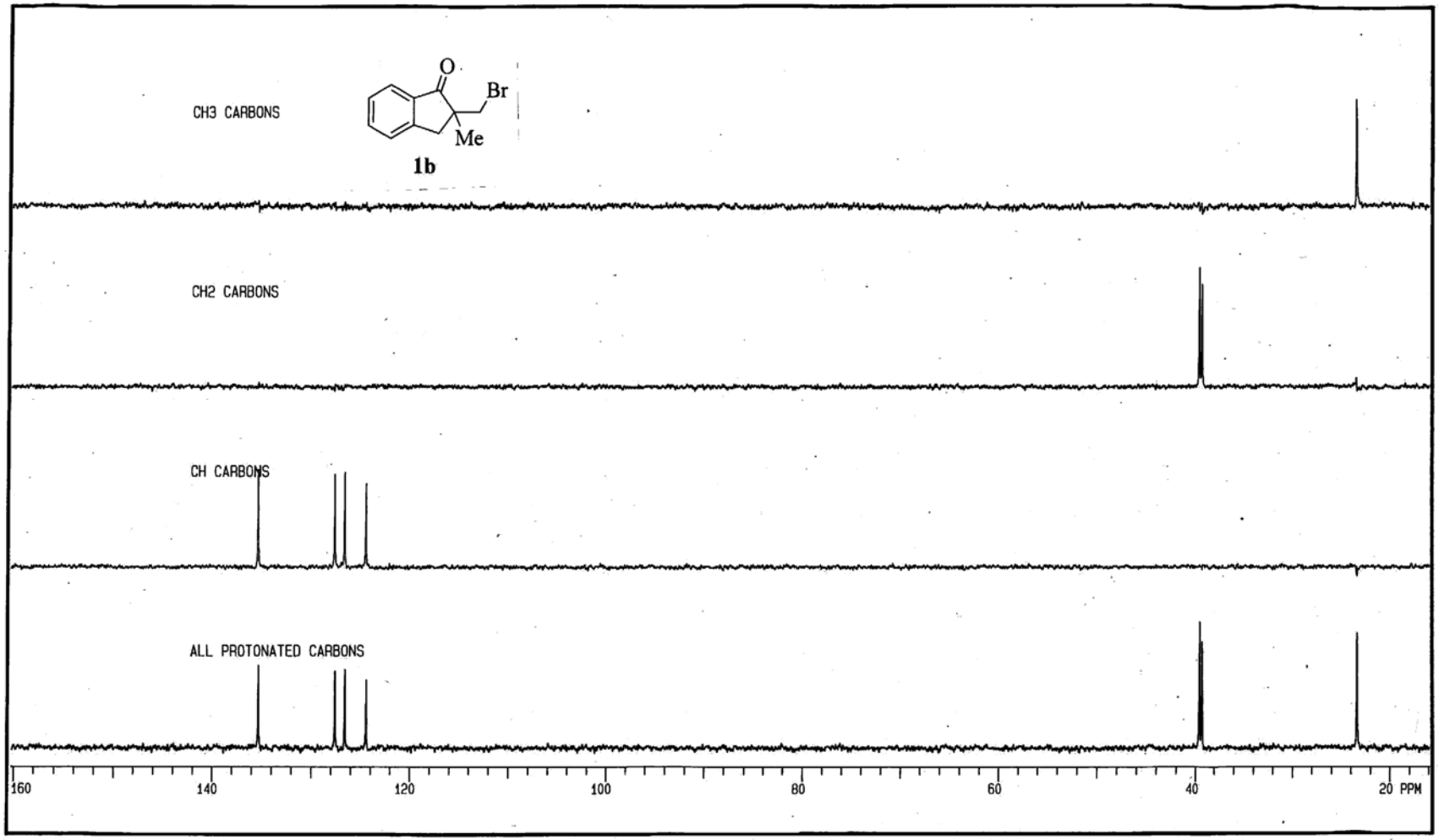
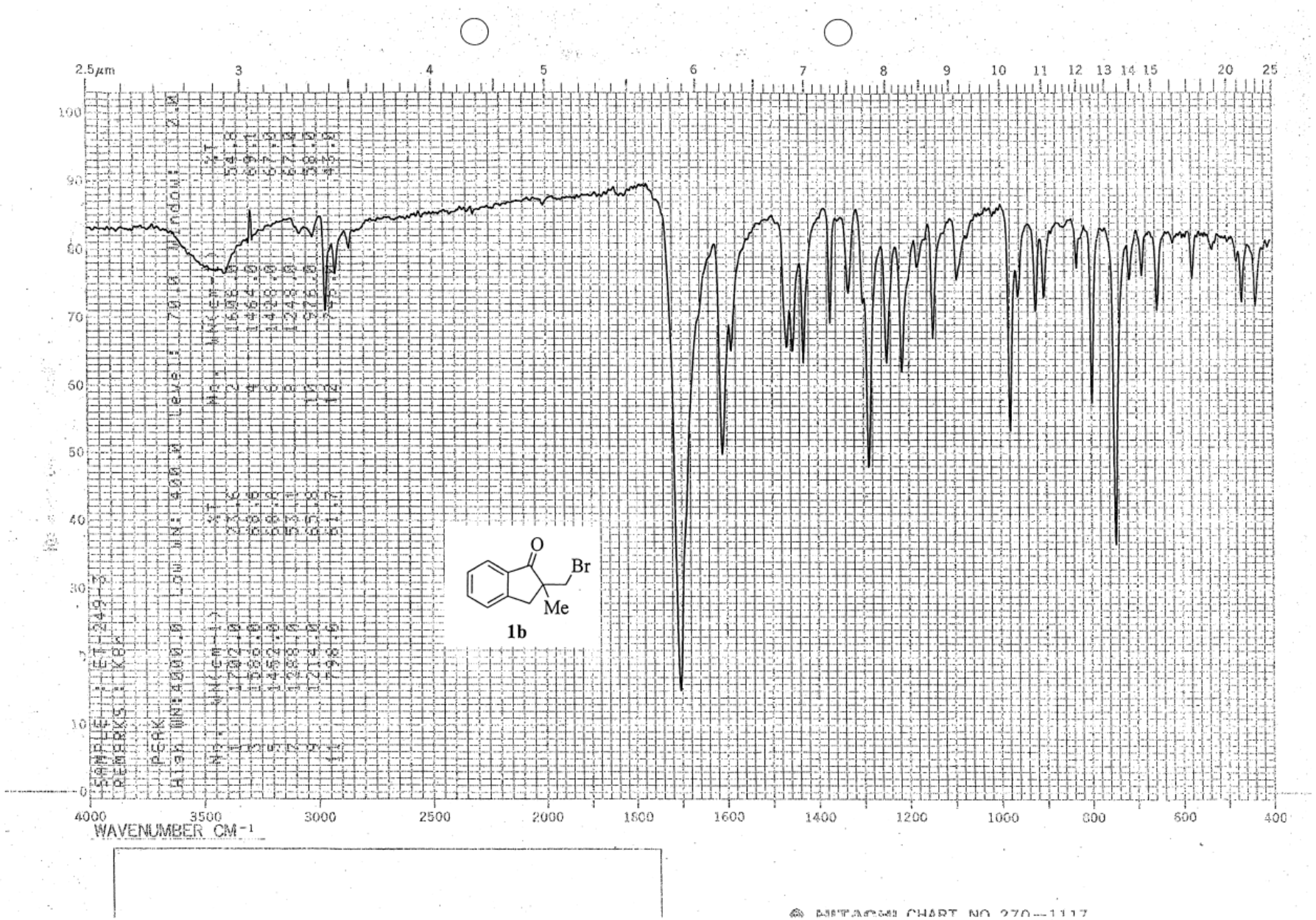
NO 997260 J
Varian Japan Ltd.

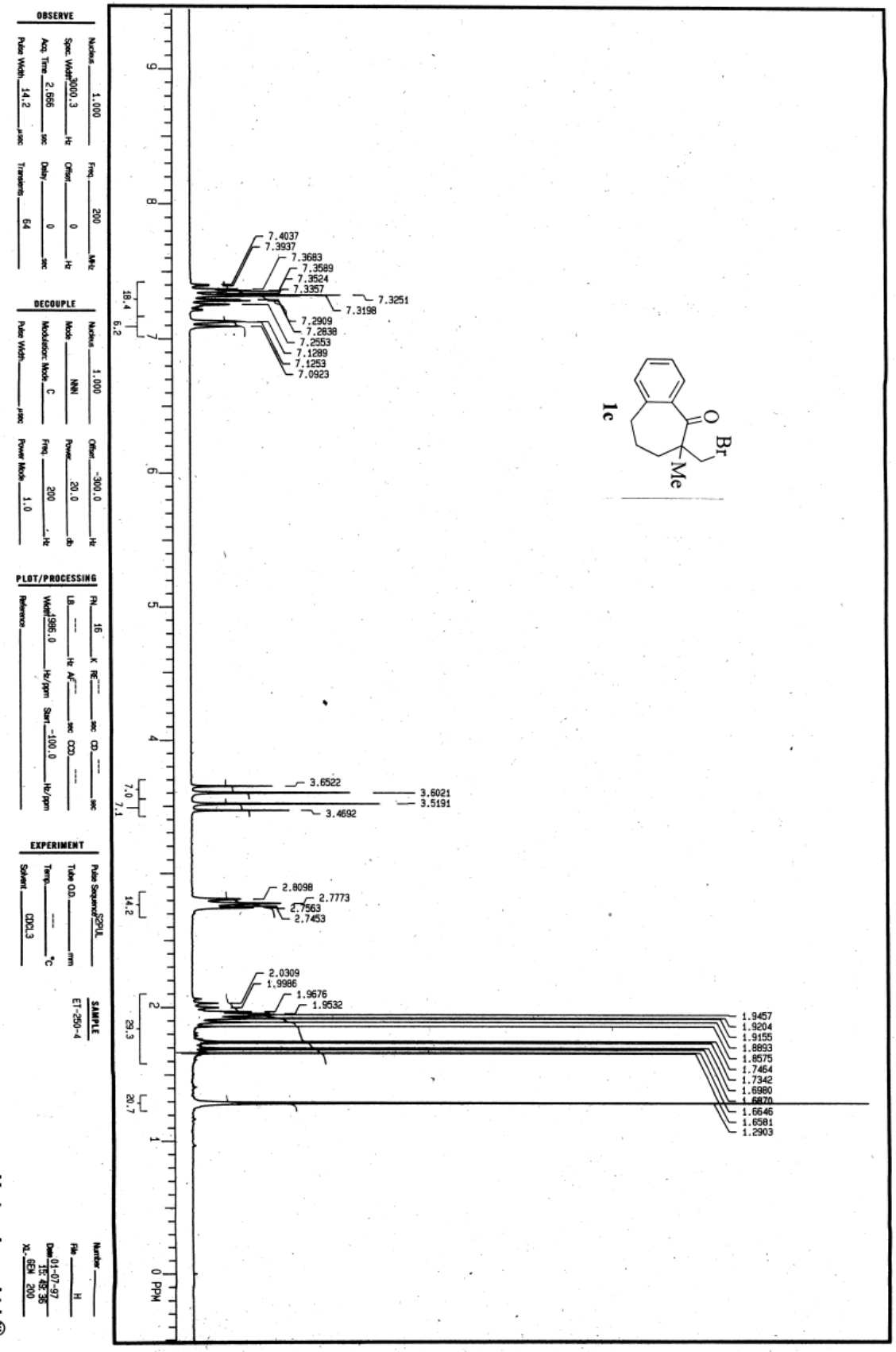

SAMPLE : ET-240-4
REMARKS : NEAT
PEAK
High WN:4000.0 Low WN: 400.0 Level: 20.0 Window: 2.0
No. WN(cm-1) %T
1 2929.0 56.1
2 1680.0 20.0
3 1600.0 50.0
4 1454.0 52.4
5 1374.0 47.5
6 1302.0 45.7
7 1262.0 46.8
8 1222.0 47.6
9 980.0 65.6
10 740.0 37.8
WAVENUMBER CM-1
1a
O
Br
Me

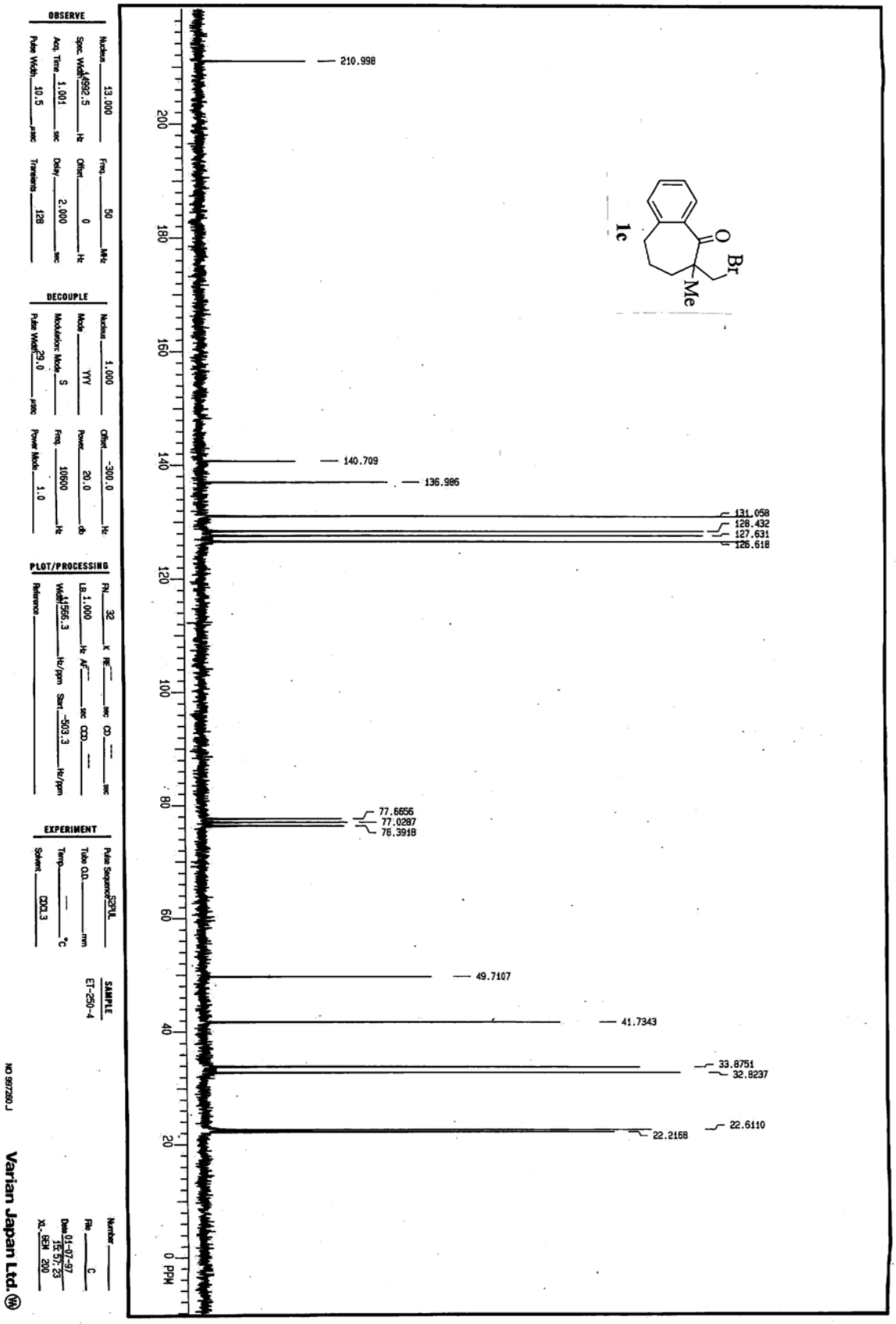

7.7865
7.7829
7.7481
7.6677
7.6614
7.6294
7.5934
7.5873
7.4890
7.4843
7.4507
7.4456
7.4236
7.4194
7.3852
7.3500
7.3451
3.6607
3.6107
3.5297
3.4794
3.3868
2.9893
2.9028
1.3384
O
Me
Br
1b
PPM

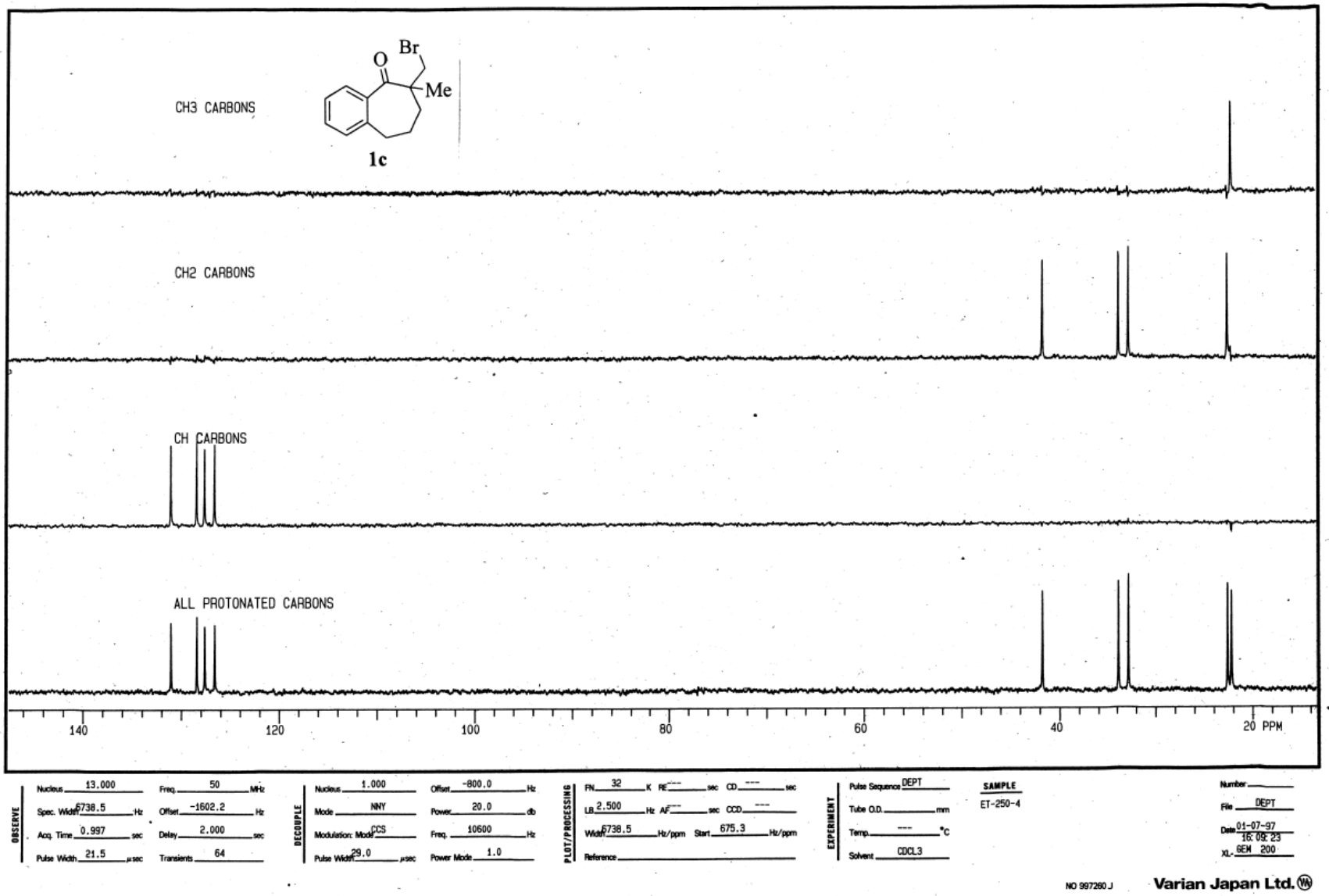

207.084
152.181
135.310
135.171
127.599
126.594
124.443
77.6656
77.0287
76.3918
49.9695
39.5870
39.2938
23.3874
0 PPM
1b
Me
Br
O
OBSERVE
DECOUPLE
PLOT/PROCESSING
EXPERIMENT
SAMPLE
ET-249-43
Varian Japan Ltd.

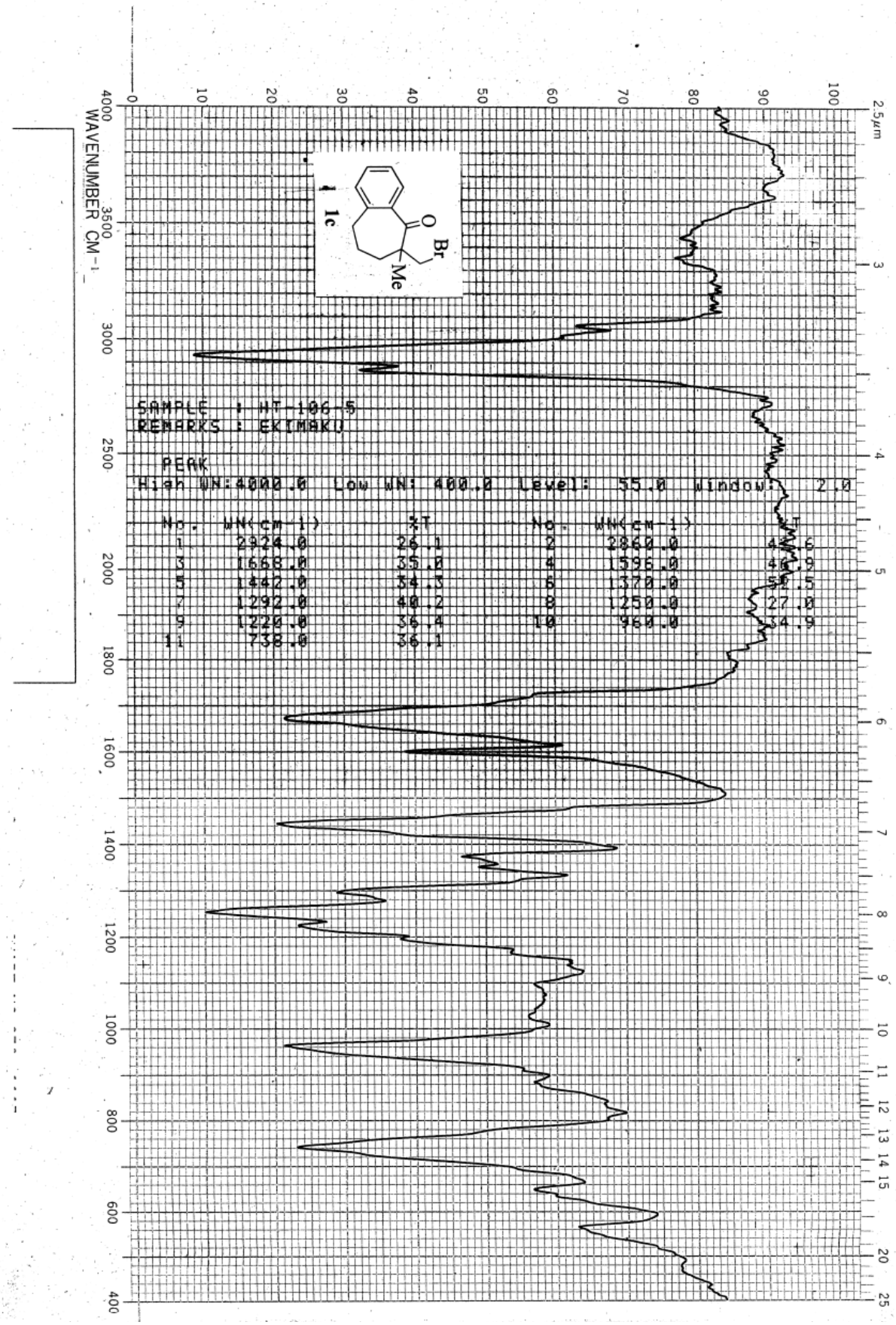

CH3 CARBONS
O
Br
Me
1b
CH2 CARBONS
CH CARBONS
ALL PROTONATED CARBONS
160
140
120
100
80
60
40
20 PPM
OBSERVE
Nucleus 13.000
Freq 50 MHz
Spec. Width 7256.9 Hz
Offset -1220.7 Hz
Acq. Time 1.004 sec
Delay 2.000 sec
Pulse Width 21.5 µsec
Transients 64
DECOUPLE
Nucleus 1.000
Offset -800.0 Hz
Mode NNY
Power 20.0 db
Modulation Mode CCS
Freq. 10600 Hz
Pulse Width 29.0 µsec
Power Mode 1.0
PLOT/PROCESSING
FN 32 K RE --- sec CD --- sec
LB 2.500 Hz AF --- sec CCD ---
Width 7256.9 Hz/ppm Start 798.5 Hz/ppm
Reference
EXPERIMENT
Pulse Sequence DEPT
Tube O.D. mm
Temp. --- °C
Solvent CDCL3
SAMPLE
ET-249-#3
Number
File DEPT
Date 01-05-97
15:30:57
XL- GEN 200
NO 997260 J
Varian Japan Ltd.

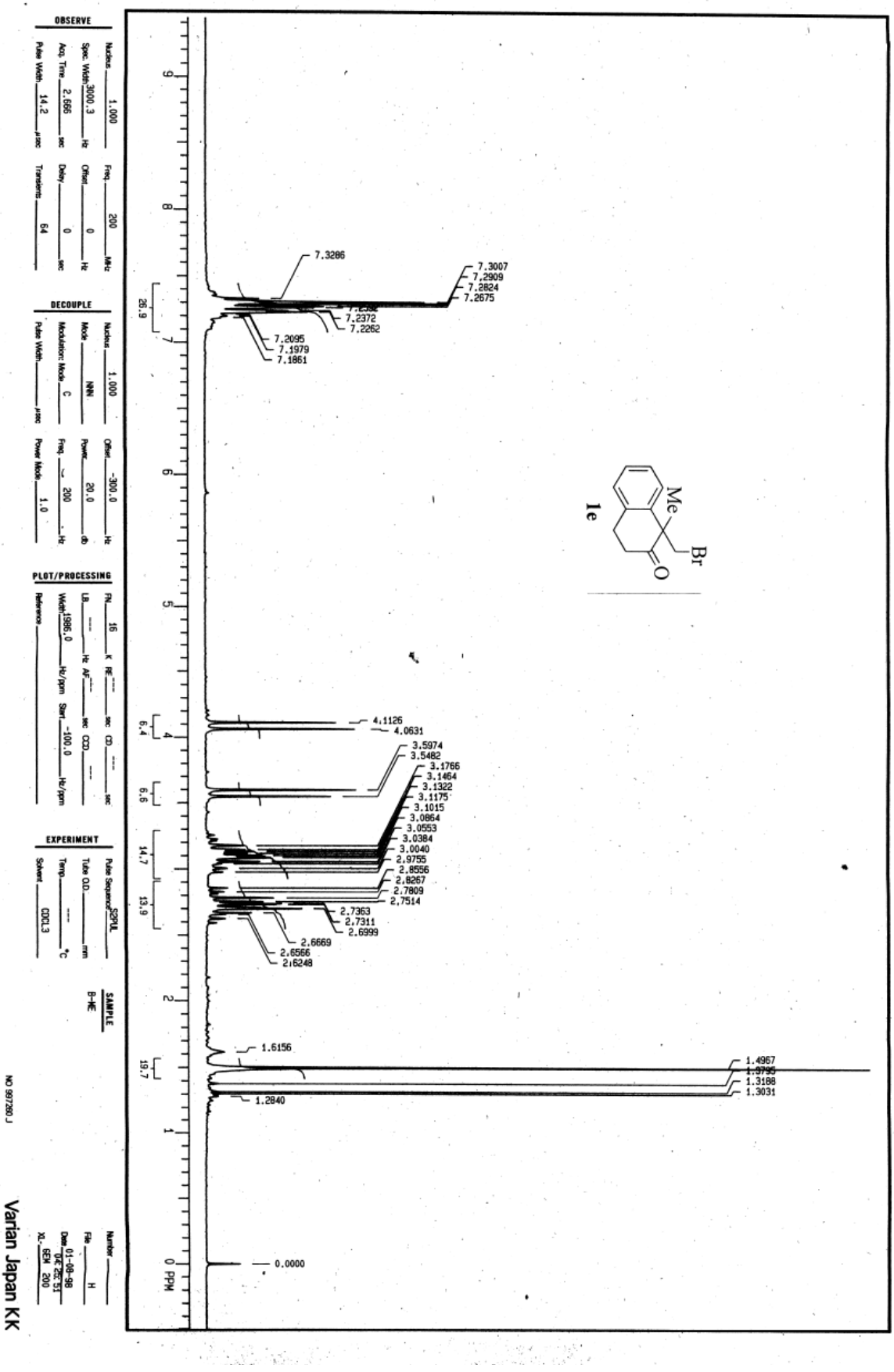

2.5μm
WAVENUMBER CM⁻¹
Br
O
Me
1b

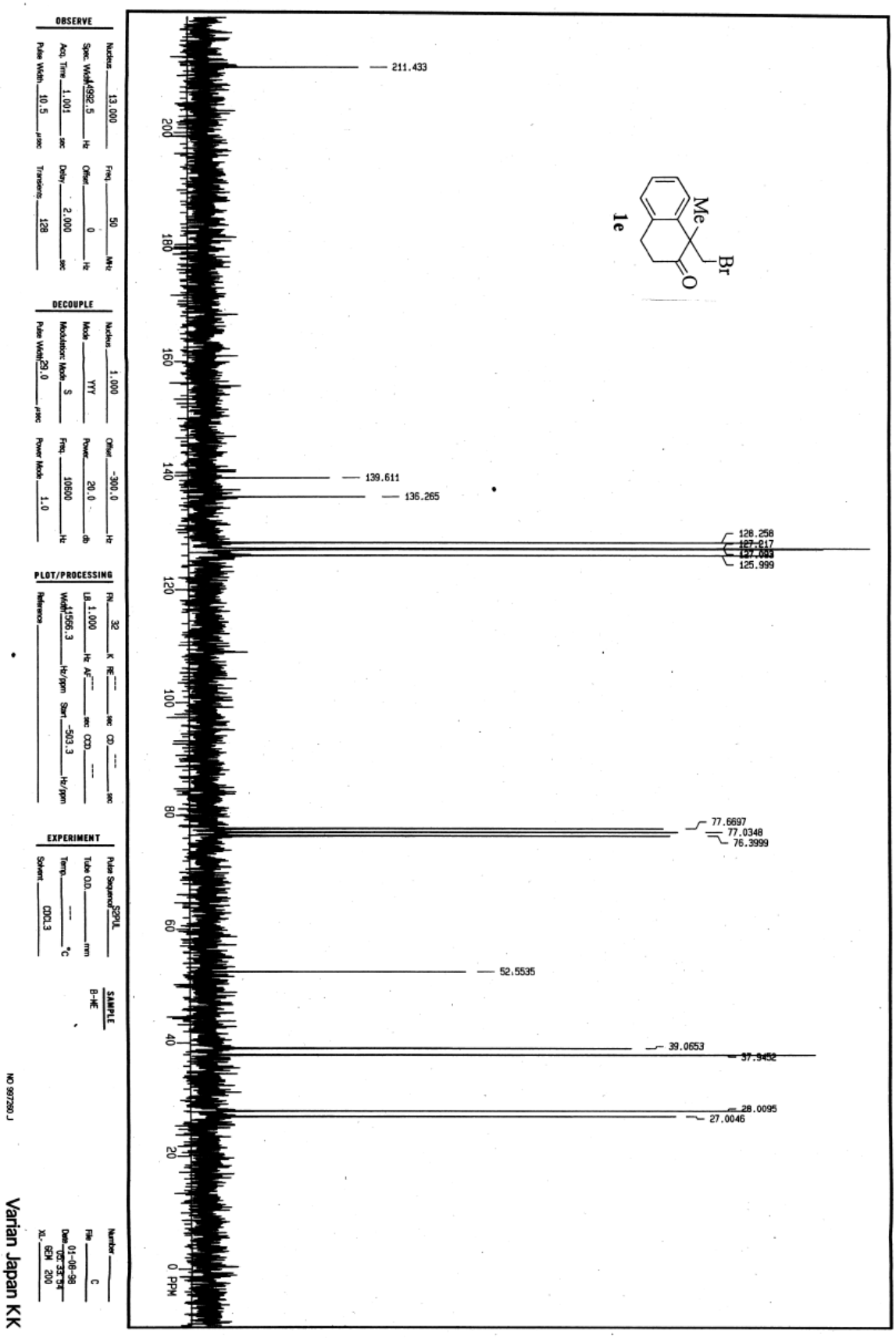

7.4037
7.3937
7.3683
7.3589
7.3524
7.3357
7.3251
7.3198
7.2909
7.2838
7.2553
7.1289
7.1253
7.0923
3.6522
3.6021
3.5191
3.4692
2.8098
2.7773
2.7563
2.7453
2.0309
1.9986
1.9676
1.9532
1.9457
1.9204
1.9155
1.8893
1.8575
1.7464
1.7342
1.6980
1.6646
1.6581
1.2903
O
Br
Me
1c
PPM

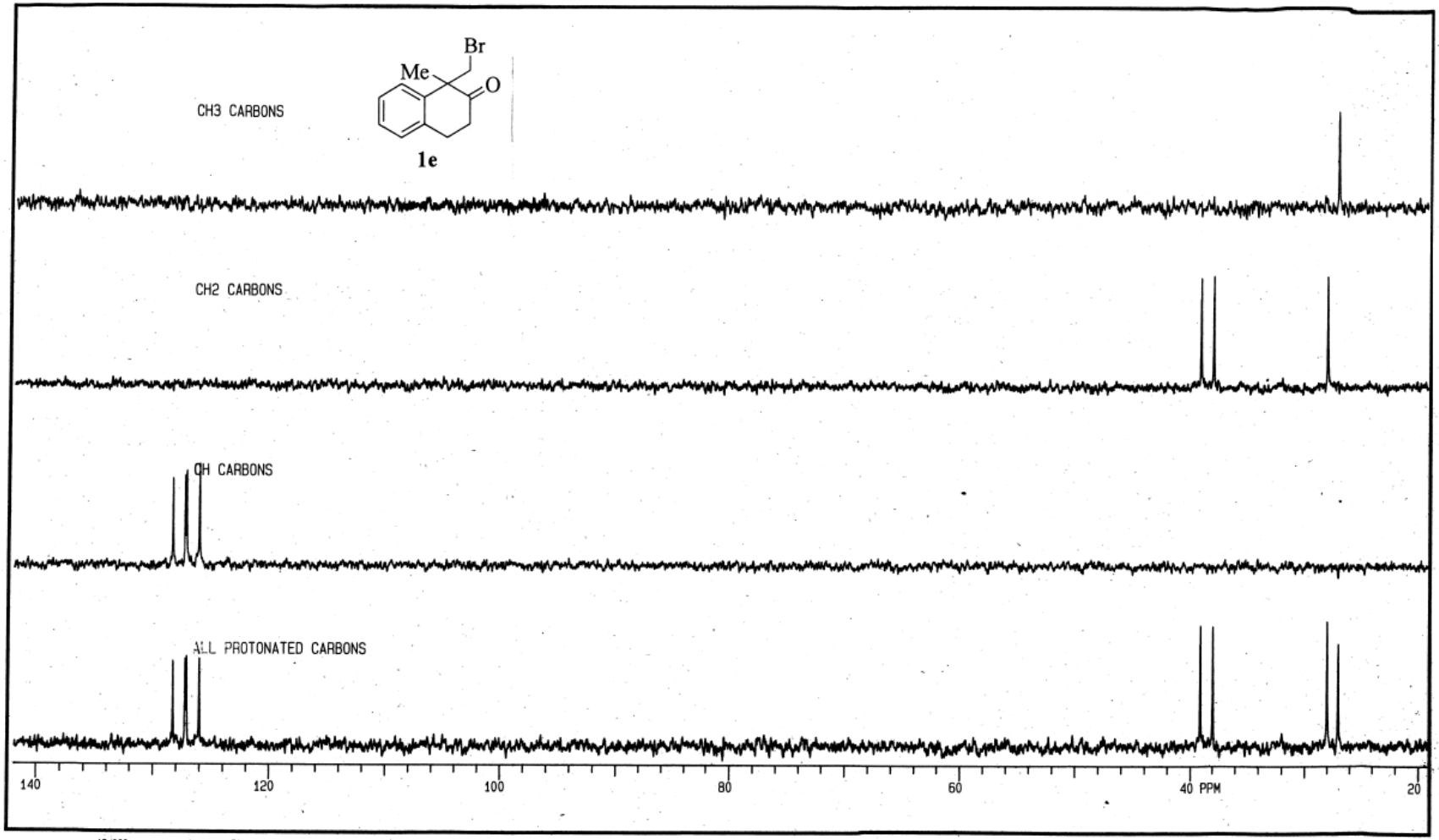

210.998
140.709
136.986
131.058
128.432
127.631
126.618
77.6656
77.0287
76.3918
49.7107
41.7343
33.8751
32.8237
22.6110
22.2168
200
180
160
140
120
100
80
60
40
20
0 PPM
1c
O
Br
Me
OBSERVE
DECOUPLE
PLOT/PROCESSING
EXPERIMENT
SAMPLE
ET-250-4
Varian Japan Ltd.

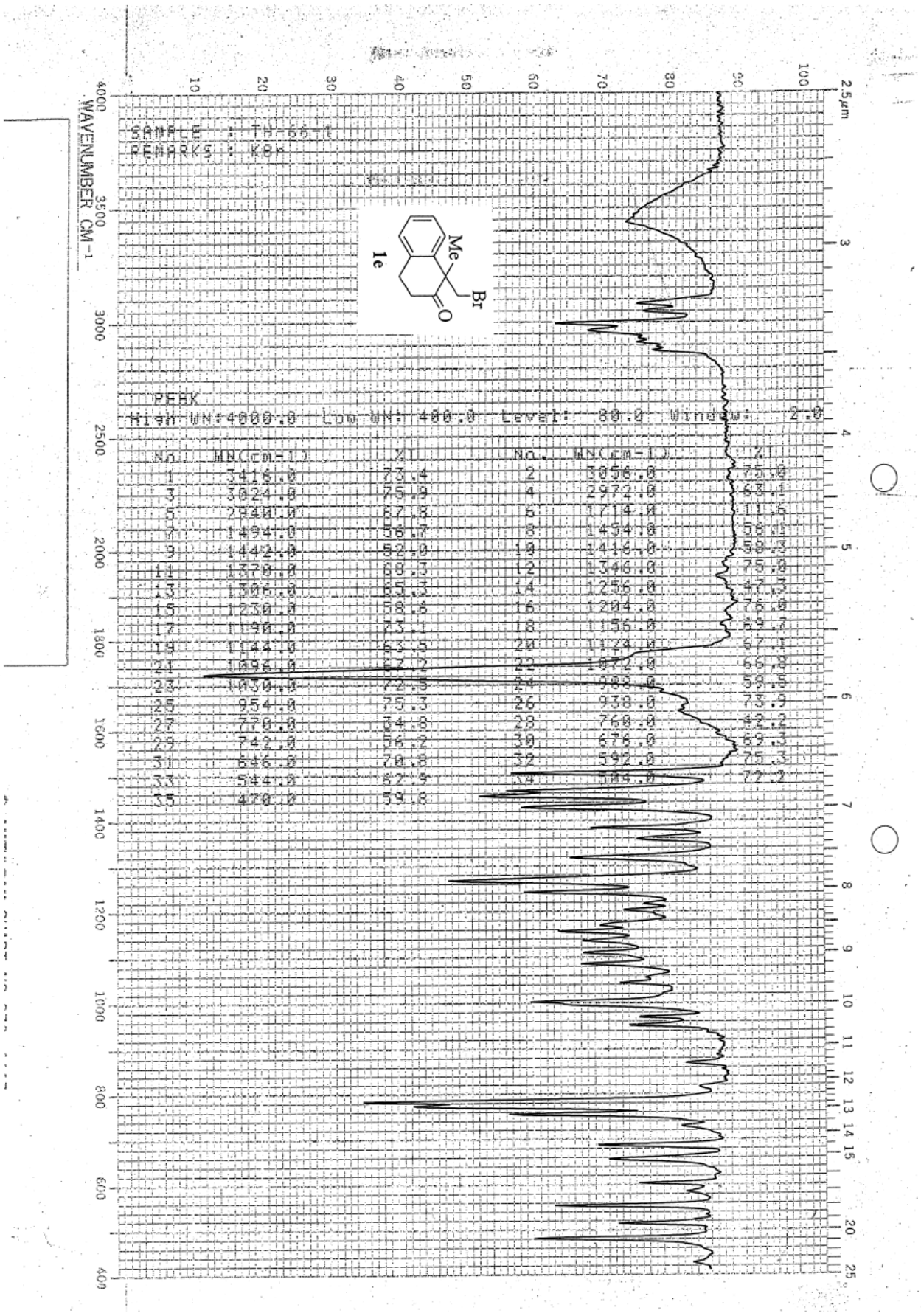

CH3 CARBONS
Br
O
Me
1c
CH2 CARBONS
CH CARBONS
ALL PROTONATED CARBONS
140
120
100
80
60
40
20 PPM
OBSERVE
Nucleus 13.000
Freq. 50 MHz
Spec. Width 6738.5 Hz
Offset -1602.2 Hz
Acq. Time 0.997 sec
Delay 2.000 sec
Pulse Width 21.5 µsec
Transients 64
DECOUPLE
Nucleus 1.000
Offset -800.0 Hz
Mode NNY
Power 20.0 db
Modulation: Mode CCS
Freq. 10600 Hz
Pulse Width 29.0 µsec
Power Mode 1.0
PLOT/PROCESSING
FN 32 K RE sec CD sec
LB 2.500 Hz AF sec CCD
Width 6738.5 Hz/ppm Start 675.3 Hz/ppm
Reference
EXPERIMENT
Pulse Sequence DEPT
Tube O.D. mm
Temp. °C
Solvent CDCL3
SAMPLE
ET-250-4
Number
File DEPT
Date 01-07-97
16:09:23
XL- GEM 200
NO 997260 J
Varian Japan Ltd.

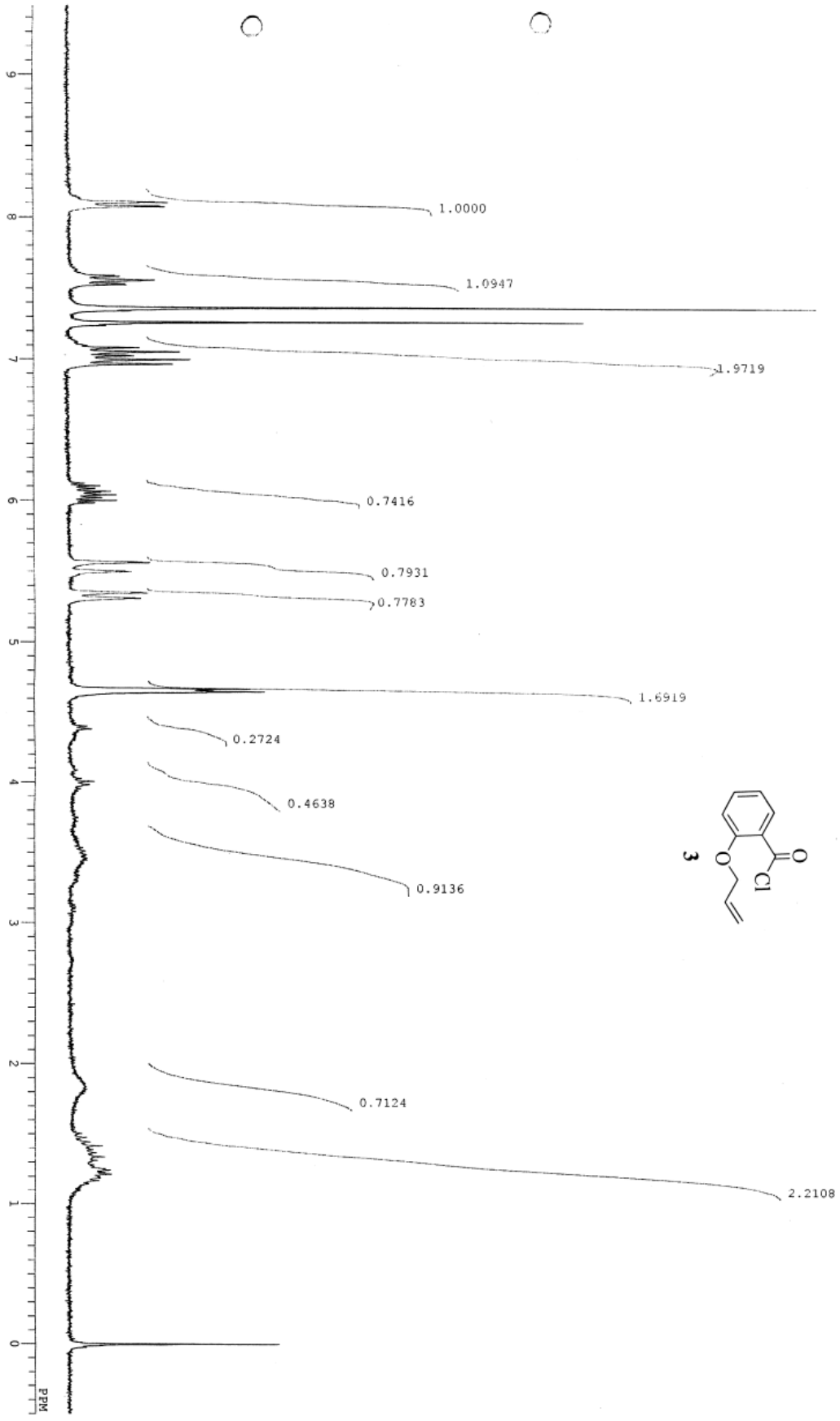

1c
O
Br
Me
SAMPLE : HT-106-5
REMARKS : EKIMAKI
PEAK
High WN:4000.0 Low WN: 400.0 Level: 55.0 Window: 2.0
No. WN(cm-1) %T
1 2924.0 26.1
2 2860.0 45.6
3 1668.0 35.0
4 1596.0 45.9
5 1442.0 54.5
6 1370.0 55.5
7 1292.0 40.2
8 1250.0 27.0
9 1220.0 36.4
10 960.0 34.9
11 738.0 36.1
WAVENUMBER CM-1
2.5μm

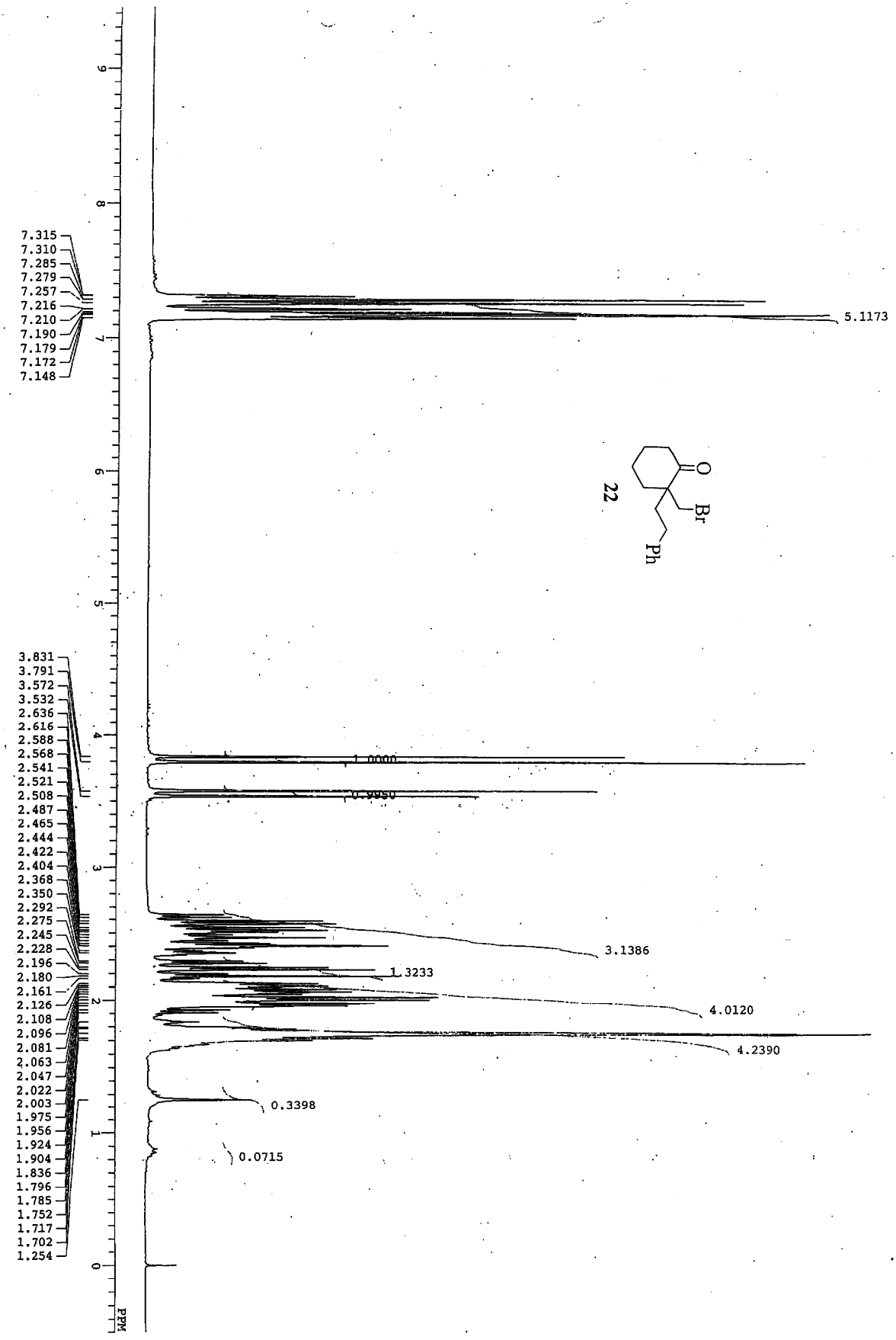

OBSERVE
Nucleus 1.000
Spec. Width 3000.3 Hz
Freq. 200 MHz
Acq. Time 2.666 sec
Offset 0 Hz
Pulse Width 14.2 μsec
Delay 0 sec
Transients 64
DECOUPLE
Nucleus 1.000
Mode NNN
Offset -300.0 Hz
Modulation Mode C
Power 20.0 db
Pulse Width μsec
Freq. 200 Hz
Power Mode 1.0
PLOT/PROCESSING
FN 16
LB
Width 1986.0 Hz/ppm
Start -100.0 Hz/ppm
EXPERIMENT
Pulse Sequence S2PUL
Tube O.D. mm
Temp °C
Solvent CDCl3
SAMPLE
B-ME
Number
File H
Date 01-09-98
Time 04:25:51
XL GEM 200
NO 997280.J
Varian Japan KK
7.3286
7.3007
7.2909
7.2824
7.2675
7.2372
7.2262
7.2095
7.1979
7.1861
4.1126
4.0631
3.5974
3.5482
3.1766
3.1464
3.1322
3.1175
3.1015
3.0864
3.0553
3.0384
3.0040
2.9755
2.8556
2.8267
2.7809
2.7514
2.7363
2.7311
2.6999
2.6669
2.6566
2.6248
1.6156
1.4967
1.3188
1.3031
1.2840
0.0000
PPM
Me
Br
O
1e

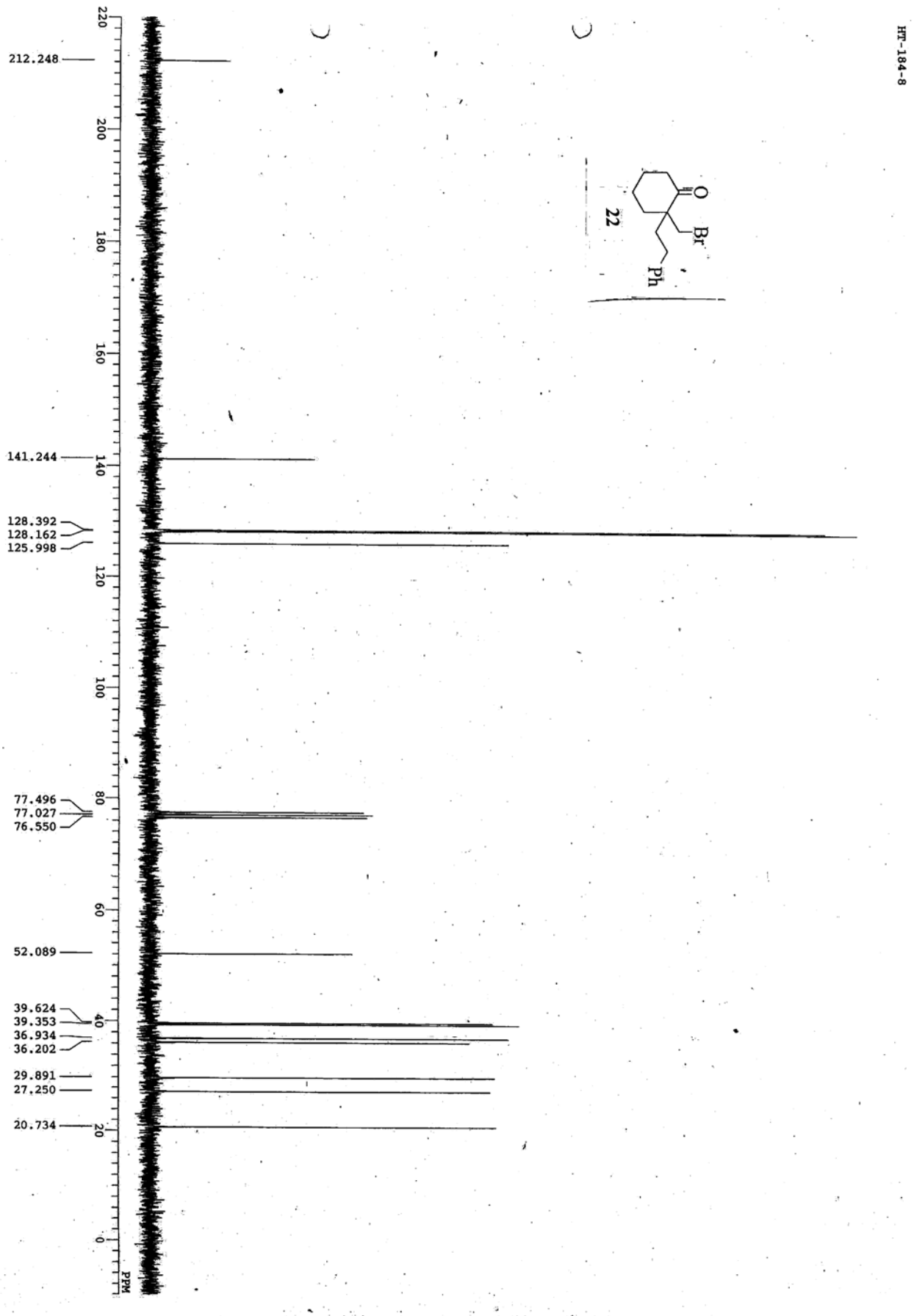

211.433
139.611
136.265
128.258
127.817
127.003
125.999
77.6697
77.0348
76.3999
52.5535
39.0653
37.9452
28.0095
27.0046
0 PPM
Me
Br
O
1e
OBSERVE
Nucleus 13.000
Spec. Width 4992.5 Hz
Acq. Time 1.001 sec
Pulse Width 10.5 μsec
Freq. 50 MHz
Offset 0 Hz
Delay 2.000 sec
Transients 128
DECOUPLE
Nucleus 1.000
Mode YYY
Modulation Mode S
Pulse Width 29.0 μsec
Offset -300.0 Hz
Power 20.0 db
Freq. 10600 Hz
Power Mode 1.0
PLOT/PROCESSING
FN 32
LB 1.000 Hz
Width 1566.3 Hz/ppm
RE ---- K
AF ---- sec
Start -503.3 Hz/ppm
CD ---- sec
COD ---
EXPERIMENT
Pulse Sequence S2PUL
Tube O.D. mm
Temp. ---- °C
Solvent CDCl3
SAMPLE
B-ME
Number
File C
Date 01-06-98
Time 13:34:04
XL 600 200
NO 997280.J
Varian Japan KK

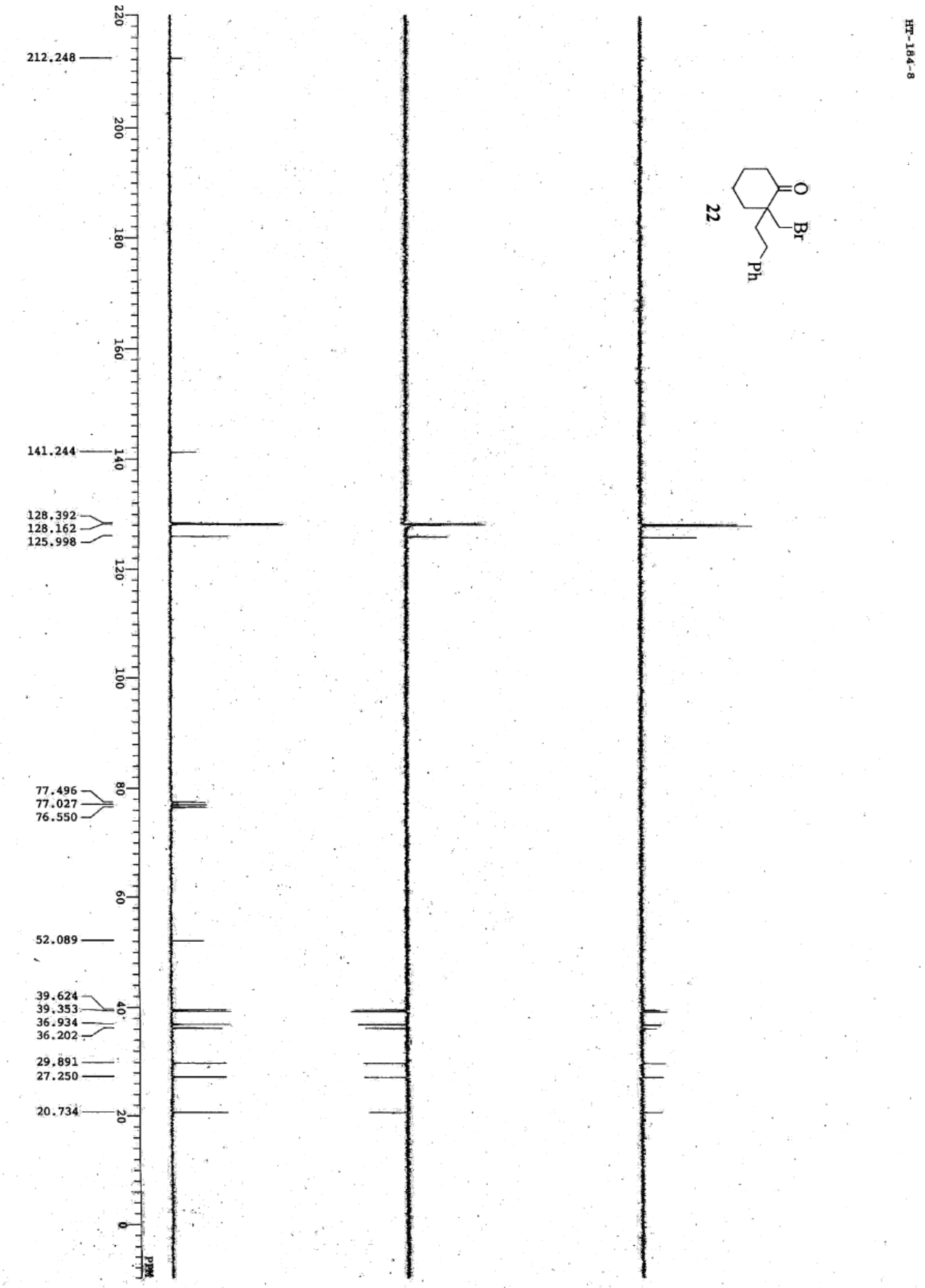

Br
Me
O
1e
CH3 CARBONS
CH2 CARBONS
CH CARBONS
ALL PROTONATED CARBONS
140
120
100
80
60
40 PPM
20
OBSERVE
Nucleus 13.000
Freq. 50 MHz
Spec. Width 6169.0 Hz
Offset -1602.2 Hz
Acq. Time 0.996 sec
Delay 2.000 sec
Pulse Width 21.5 µsec
Transients 64
DECOUPLE
Nucleus 1.000
Offset -800.0 Hz
Mode NNY
Power 20.0 db
Modulation: Mode CCS
Freq. 10600 Hz
Pulse Width 29.0 µsec
Power Mode 1.0
PLOT/PROCESSING
FN 32 K RE --- sec CD --- sec
LB 2.500 Hz AF --- sec CCD ---
Width 6169.0 Hz/ppm Start 962.8 Hz/ppm
Reference
EXPERIMENT
Pulse Sequence DEPT
Tube O.D. mm
Temp. --- °C
Solvent CDCL3
SAMPLE
B-ME
Number
File DEPT
Date 01-08-98
05:47:46
XL- GEM 200
NO 997260.J
Varian Japan KK

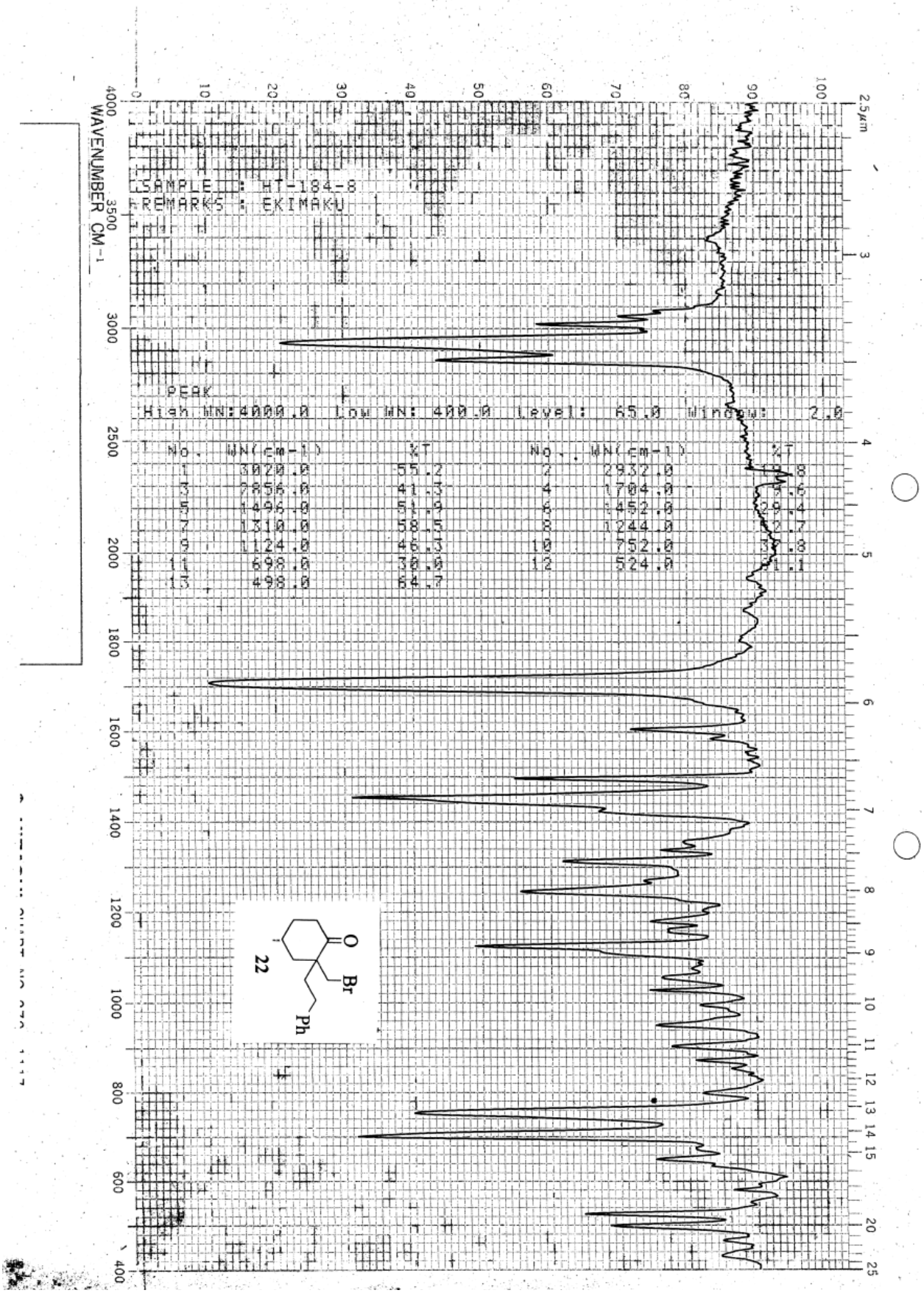

SAMPLE : TH-66-1
REMARKS : KBr
Me
Br
O
1e
PEAK
High WN:4000.0 Low WN: 400.0 Level: 80.0 Window: 2.0
No. WN(cm-1) %T
1 3416.0 73.4
2 3056.0 75.0
3 3024.0 75.9
4 2972.0 63.1
5 2940.0 67.8
6 1714.0 11.6
7 1494.0 56.7
8 1454.0 56.1
9 1442.0 52.0
10 1416.0 58.3
11 1370.0 68.7
12 1346.0 75.0
13 1306.0 55.3
14 1256.0 47.3
15 1230.0 58.6
16 1204.0 76.0
17 1190.0 73.1
18 1156.0 69.7
19 1144.0 63.5
20 1124.0 67.1
21 1096.0 67.2
22 1072.0 66.8
23 1030.0 72.3
24 988.0 59.5
25 954.0 75.3
26 938.0 73.9
27 770.0 34.8
28 760.0 42.2
29 742.0 56.2
30 676.0 69.3
31 646.0 70.8
32 592.0 75.3
33 544.0 62.9
34 [illegible] 72.2
35 470.0 59.8
WAVENUMBER CM-1
2.5μm

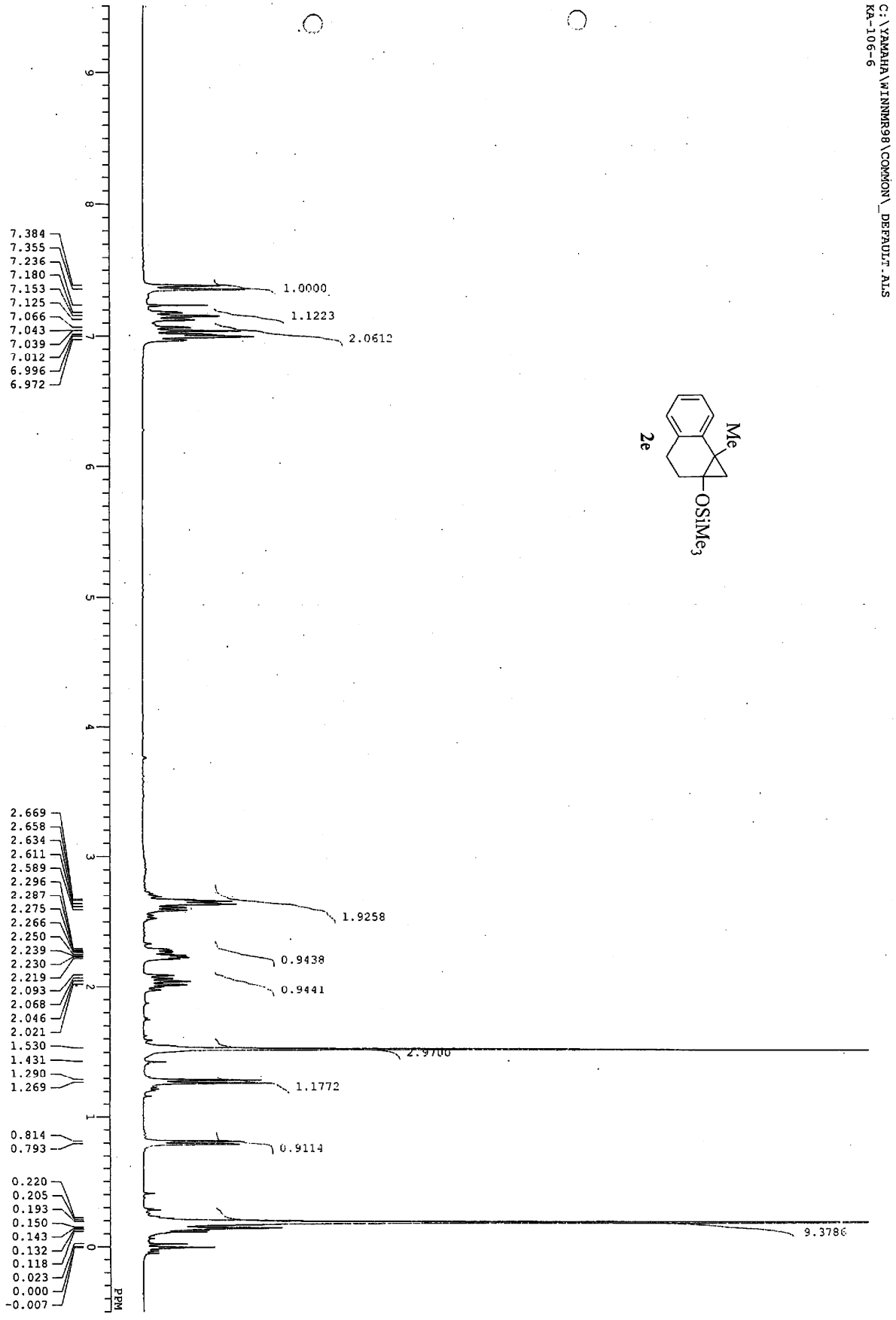

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-335-1
1.0000
1.0947
1.9719
0.7416
0.7931
0.7783
1.6919
0.2724
0.4638
0.9136
0.7124
2.2108
O
Cl
O
3
PPM

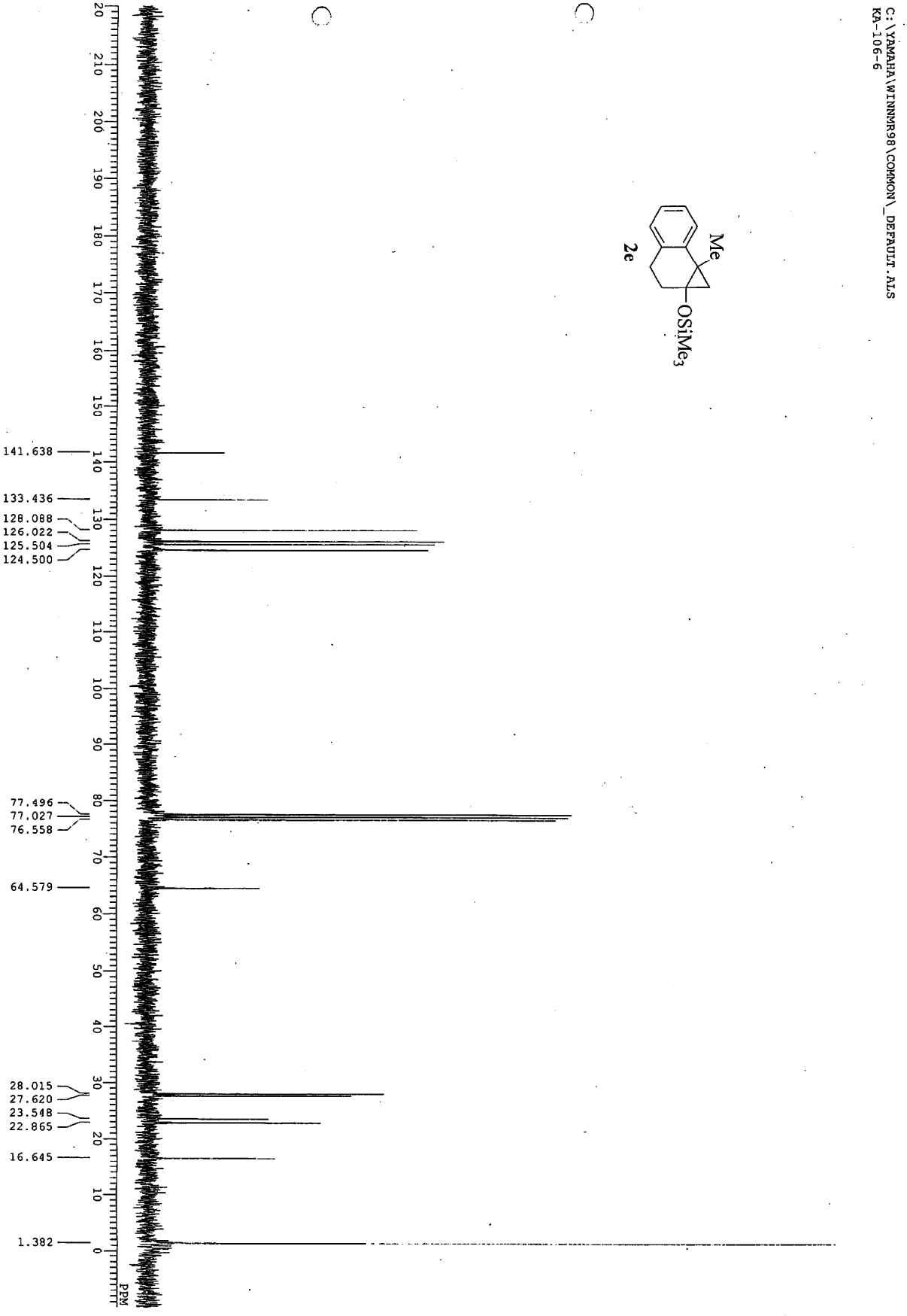

HT-184-8
7.315
7.310
7.285
7.279
7.257
7.216
7.210
7.190
7.179
7.172
7.148
3.831
3.791
3.572
3.532
2.636
2.616
2.588
2.568
2.541
2.521
2.508
2.487
2.465
2.444
2.422
2.404
2.368
2.350
2.292
2.275
2.245
2.228
2.196
2.180
2.161
2.126
2.108
2.096
2.081
2.063
2.047
2.022
2.003
1.975
1.956
1.924
1.904
1.836
1.796
1.785
1.752
1.717
1.702
1.254
5.1173
1.0000
0.9950
3.1386
1.3233
4.0120
4.2390
0.3398
0.0715
O
Br
Ph
22
PPM

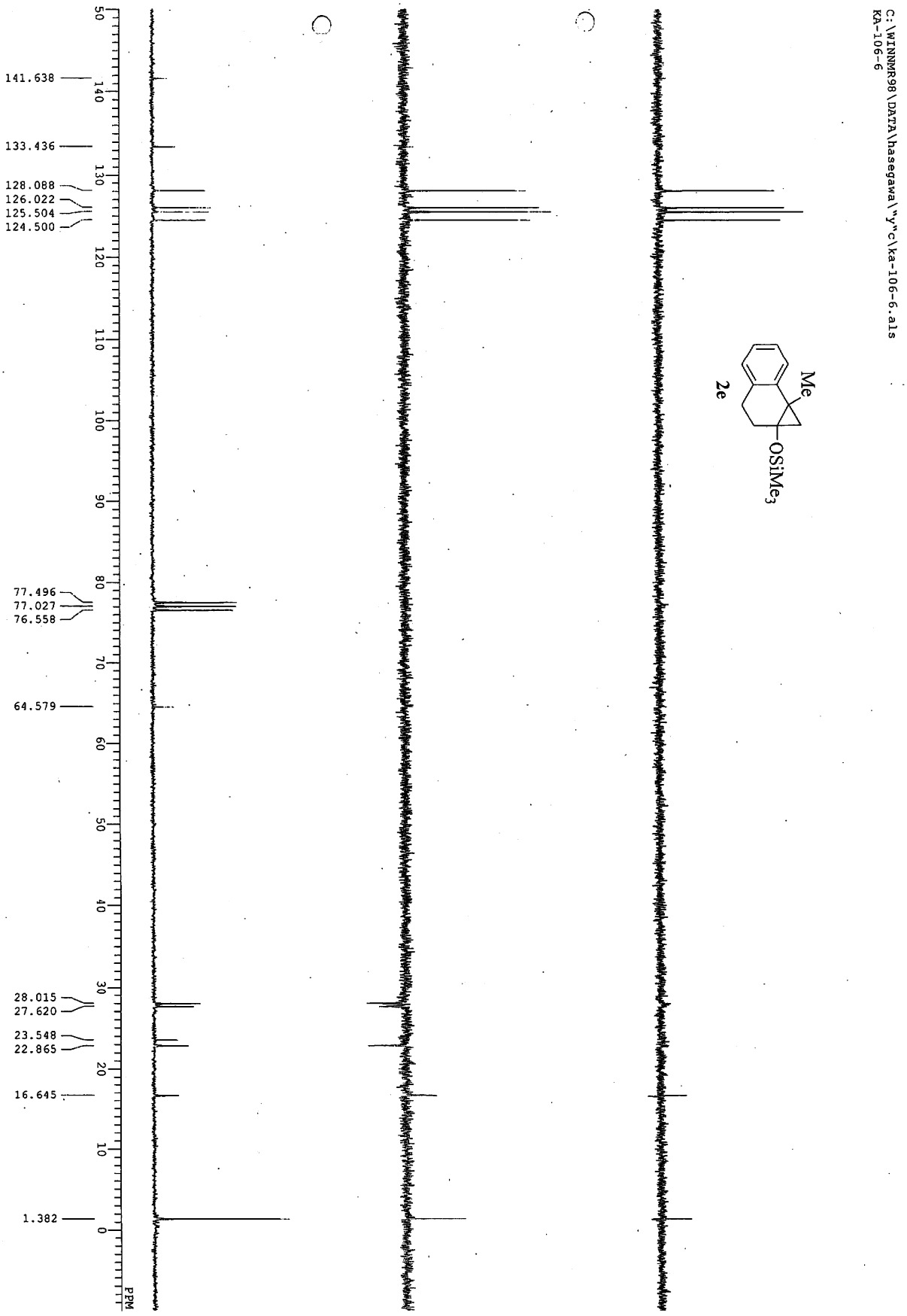

HT-184-8
O
Br
Ph
22
212.248
141.244
128.392
128.162
125.998
77.496
77.027
76.550
52.089
39.624
39.353
36.934
36.202
29.891
27.250
20.734
PPM

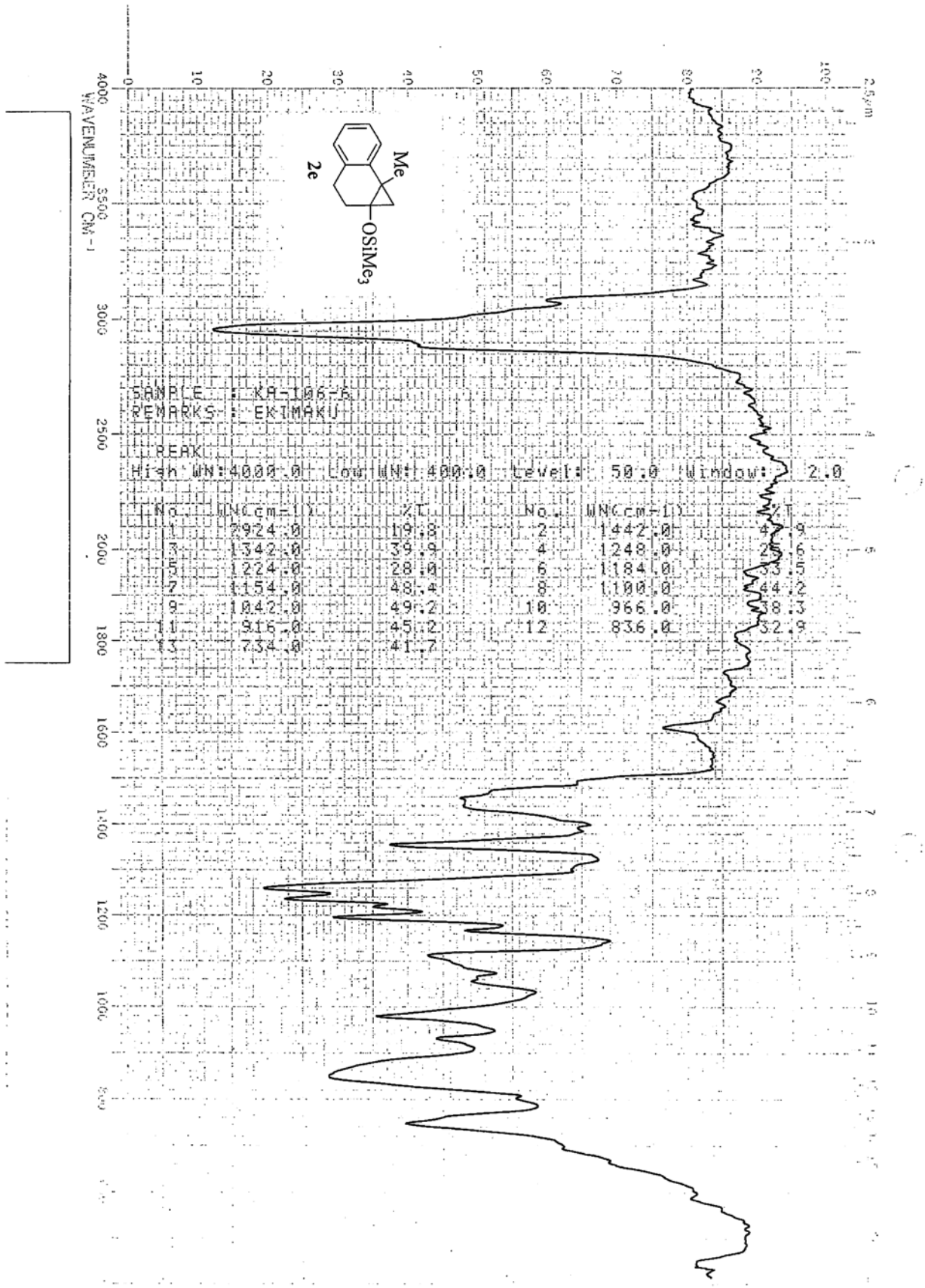

HT-184-8
O
Br
Ph
22
212.248
141.244
128.392
128.162
125.998
77.496
77.027
76.550
52.089
39.624
39.353
36.934
36.202
29.891
27.250
20.734
PPM

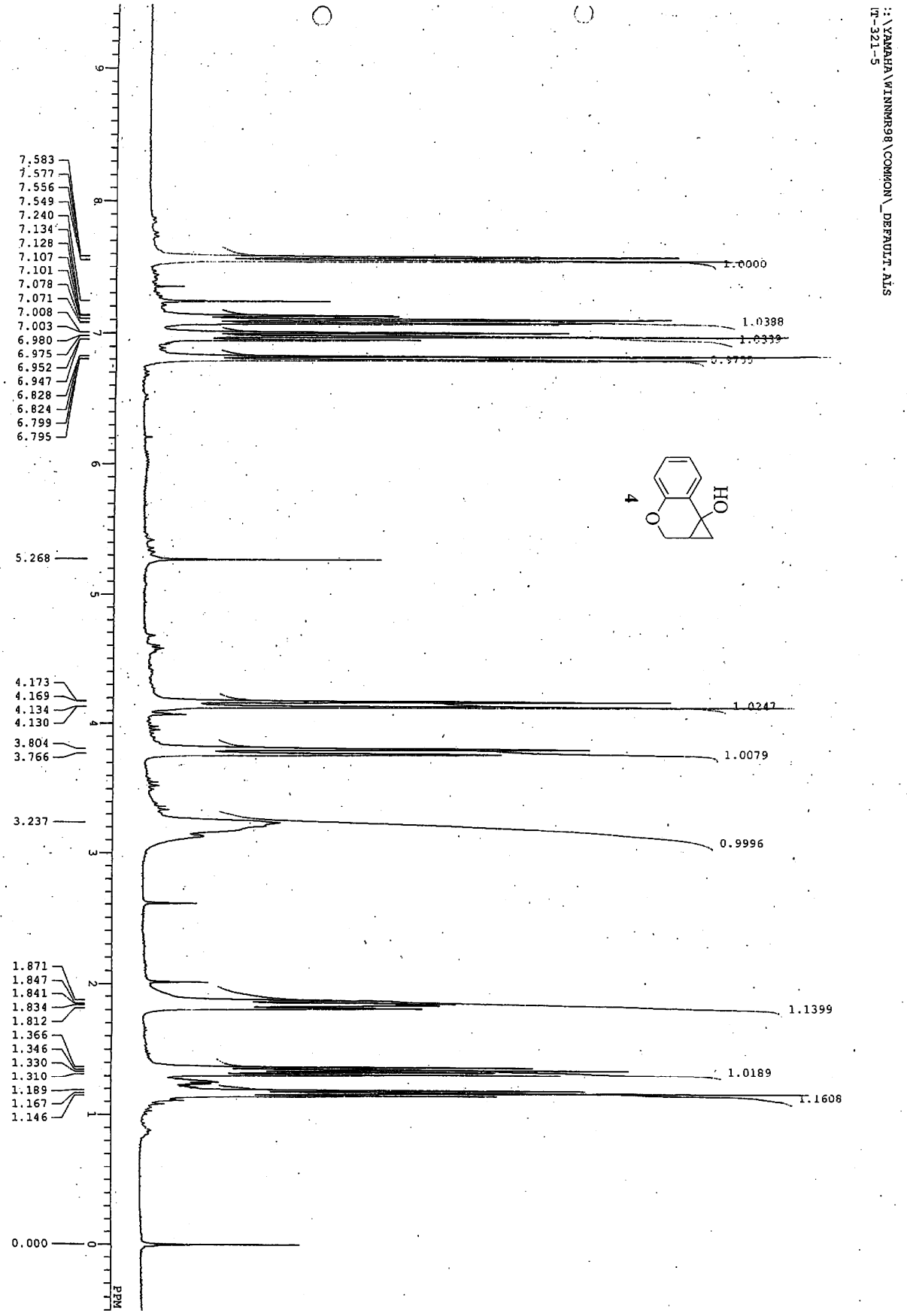

SAMPLE : HT-184-8
REMARKS : EKIMAKU
PEAK
High WN: 4000.0 Low WN: 400.0 Level: 65.0 Window: 2.0
No. WN(cm-1) %T
1 3020.0 55.2
2 2932.0
3 2856.0 41.2
4 1784.0
5 1496.0 54.9
6 1452.0
7 1310.0 58.5
8 1244.0
9 1124.0 46.2
10 752.0
11 690.0 38.7
12 524.0
13 498.0 36
WAVENUMBER CM-1
4000
3500
3000
2500
2000
1800
1600
1400
1200
1000
800
600
400
2.5μm
O
Br
Ph
22

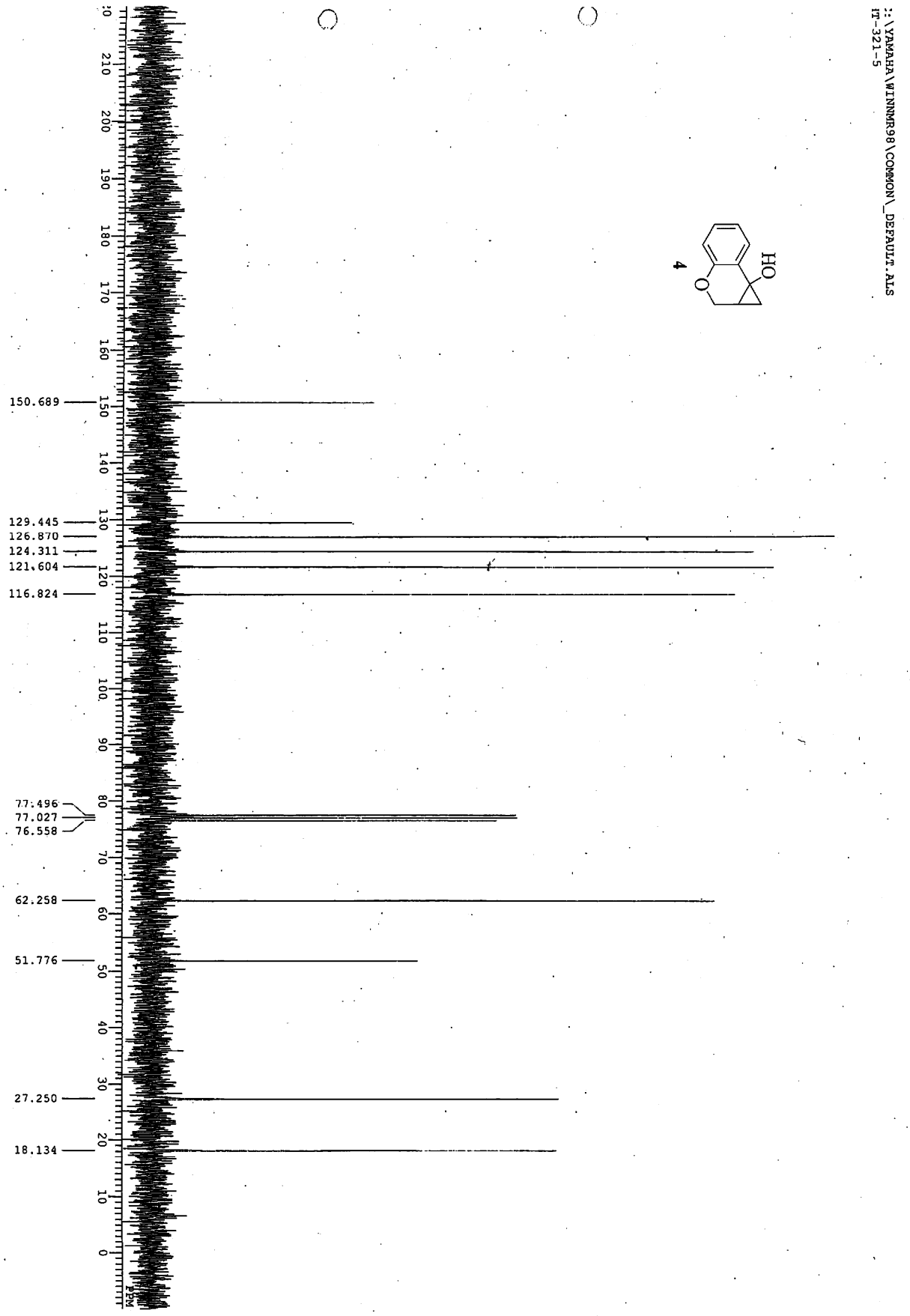

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
KA-106-6
7.384
7.355
7.236
7.180
7.153
7.125
7.066
7.043
7.039
7.012
6.996
6.972
2.669
2.658
2.634
2.611
2.589
2.296
2.287
2.275
2.266
2.250
2.239
2.230
2.219
2.093
2.068
2.046
2.021
1.530
1.431
1.290
1.269
0.814
0.793
0.220
0.205
0.193
0.150
0.143
0.132
0.118
0.023
0.000
-0.007
1.0000
1.1223
2.0612
1.9258
0.9438
0.9441
2.9700
1.1772
0.9114
9.3786
Me
OSiMe3
2e
PPM


C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
KA-106-6
Me
OSiMe3
2e
141.638
133.436
128.088
126.022
125.504
124.500
77.496
77.027
76.558
64.579
28.015
27.620
23.548
22.865
16.645
1.382
PPM


C:\WINNMR98\DATA\hasegawa\"y"c\ka-106-6.als
KA-106-6
141.638
133.436
128.088
126.022
125.504
124.500
77.496
77.027
76.558
64.579
28.015
27.620
23.548
22.865
16.645
1.382
PPM
Me
OSiMe3
2e


Me
OSiMe3
2e
SAMPLE : KA-106-A
REMARKS-: EKIMAKU
REAK
High WN:4000.0 Low WN: 400.0 Level: 50.0 Window: 2.0
No. WNCcm-1) %T
1 2924.0 19.8
2 1442.0 47.9
3 1342.0 39.9
4 1248.0 29.6
5 1224.0 28.0
6 1184.0 33.5
7 1154.0 48.4
8 1100.0 44.2
9 1042.0 49.2
10 966.0 38.3
11 916.0 45.2
12 836.0 32.9
13 734.0 41.7
WAVENUMBER CM-1


C:\YAMAHA\WINNMR98\COMMON_DEFAULT.AIS
T-321-5
7.583
7.577
7.556
7.549
7.240
7.134
7.128
7.107
7.101
7.078
7.071
7.008
7.003
6.980
6.975
6.952
6.947
6.828
6.824
6.799
6.795
5.268
4.173
4.169
4.134
4.130
3.804
3.766
3.237
1.871
1.847
1.841
1.834
1.812
1.366
1.346
1.330
1.310
1.189
1.167
1.146
0.000
PPM
1.0000
1.0388
1.0247
1.0079
0.9996
1.1399
1.0189
1.1608
4
OH
O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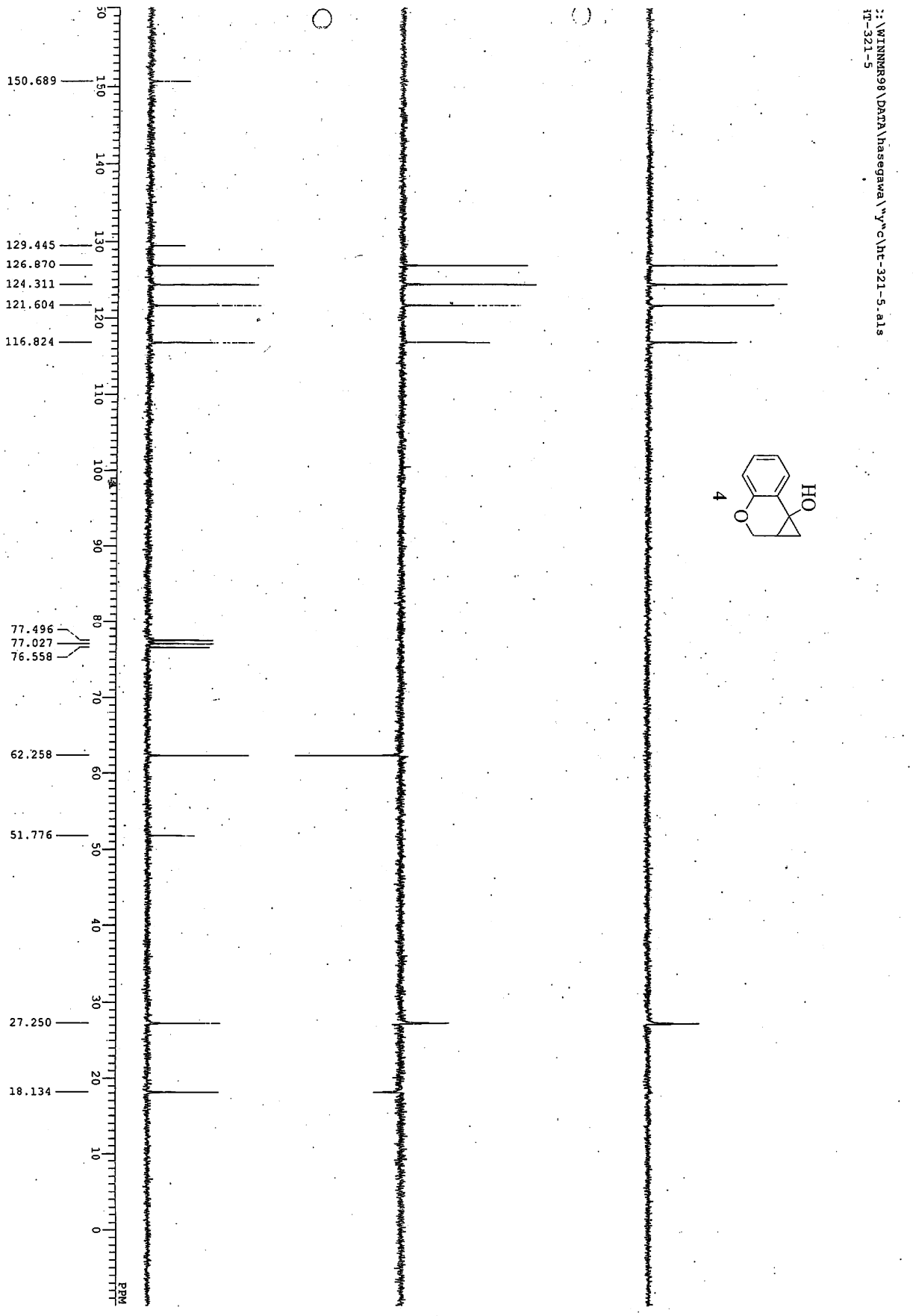
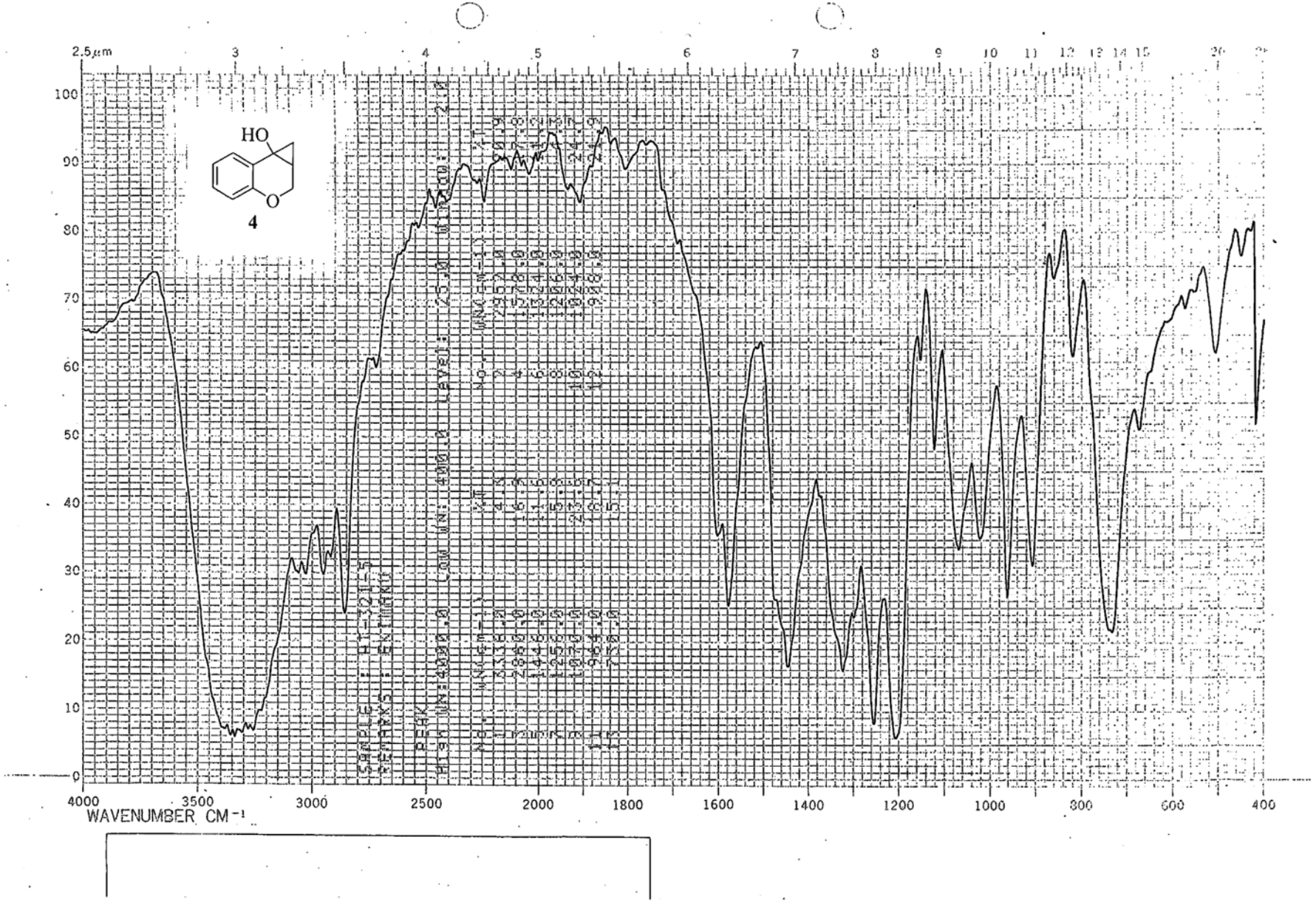
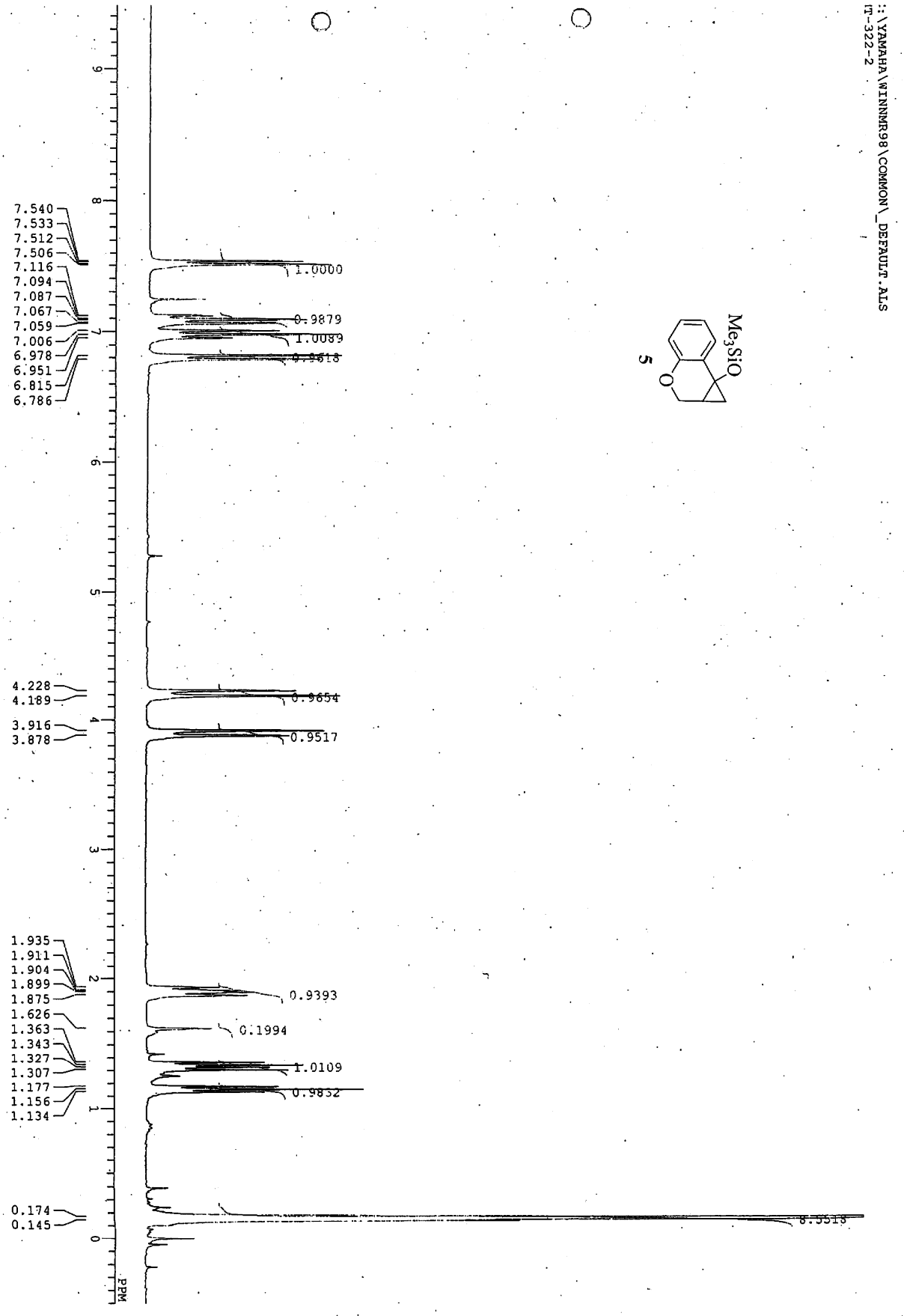
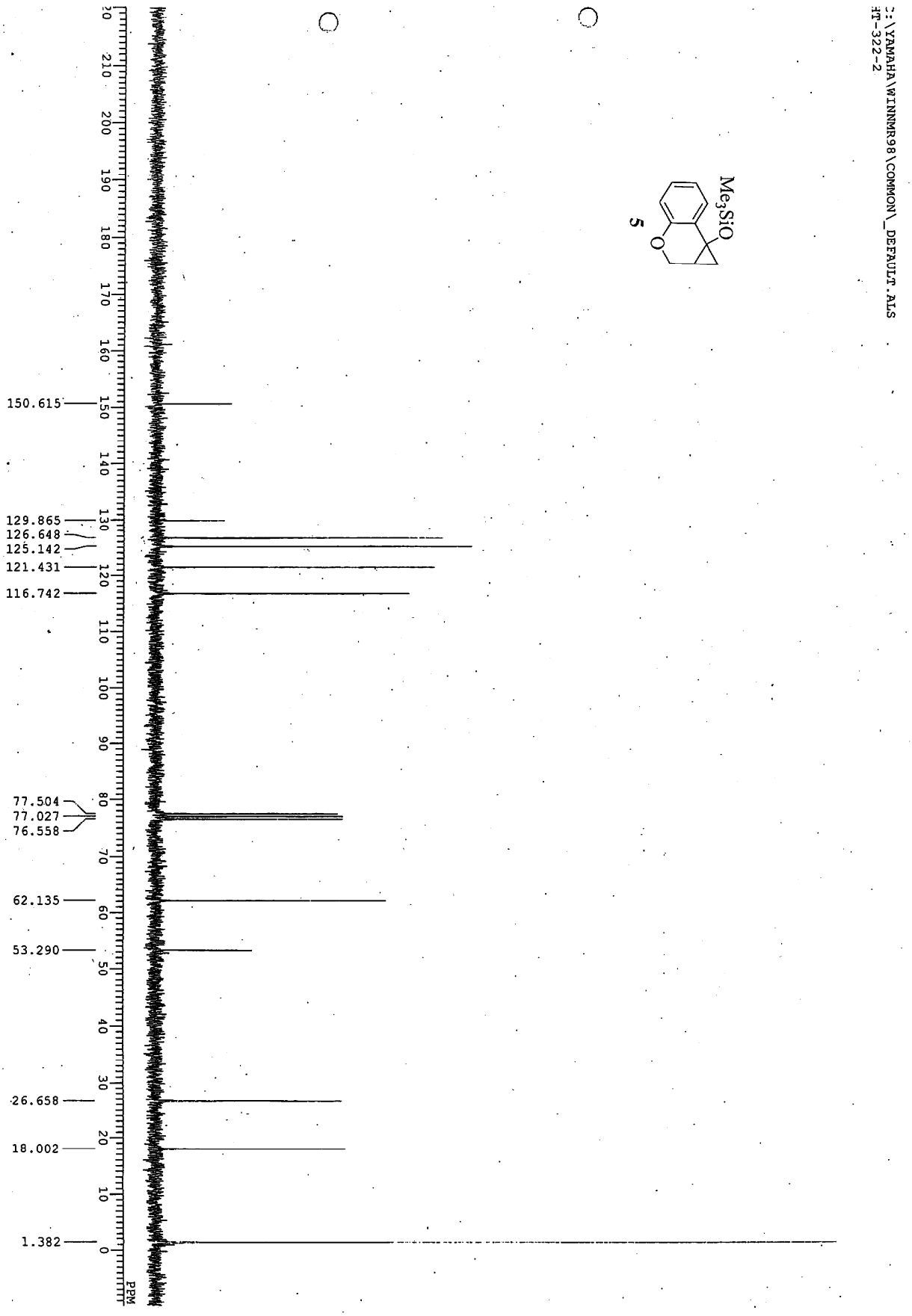
IT-321-5
HO
O
4
150.689
129.445
126.870
124.311
121.604
116.824
77.496
77.027
76.558
62.258
51.776
27.250
18.134
PPM


::\WINNMR98\DATA\hasegawa\"y"c\ht-321-5.als
HT-321-5
150.689
129.445
126.870
124.311
121.604
116.824
77.496
77.027
76.558
62.258
51.776
27.250
18.134
PPM
HO
O
4


HO
O
4
2.5μm
WAVENUMBER CM⁻¹
4000
3500
3000
2500
2000
1800
1600
1400
1200
1000
800
600
400


C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
IT-322-2
Me3SiO
5
O
7.540
7.533
7.512
7.506
7.116
7.094
7.087
7.067
7.059
7.006
6.978
6.951
6.815
6.786
4.228
4.189
3.916
3.878
1.935
1.911
1.904
1.899
1.875
1.626
1.363
1.343
1.327
1.307
1.177
1.156
1.134
0.174
0.145
1.0000
0.9879
1.0089
0.9654
0.9517
0.9393
0.1994
1.0109
0.9852
6.5515
ppm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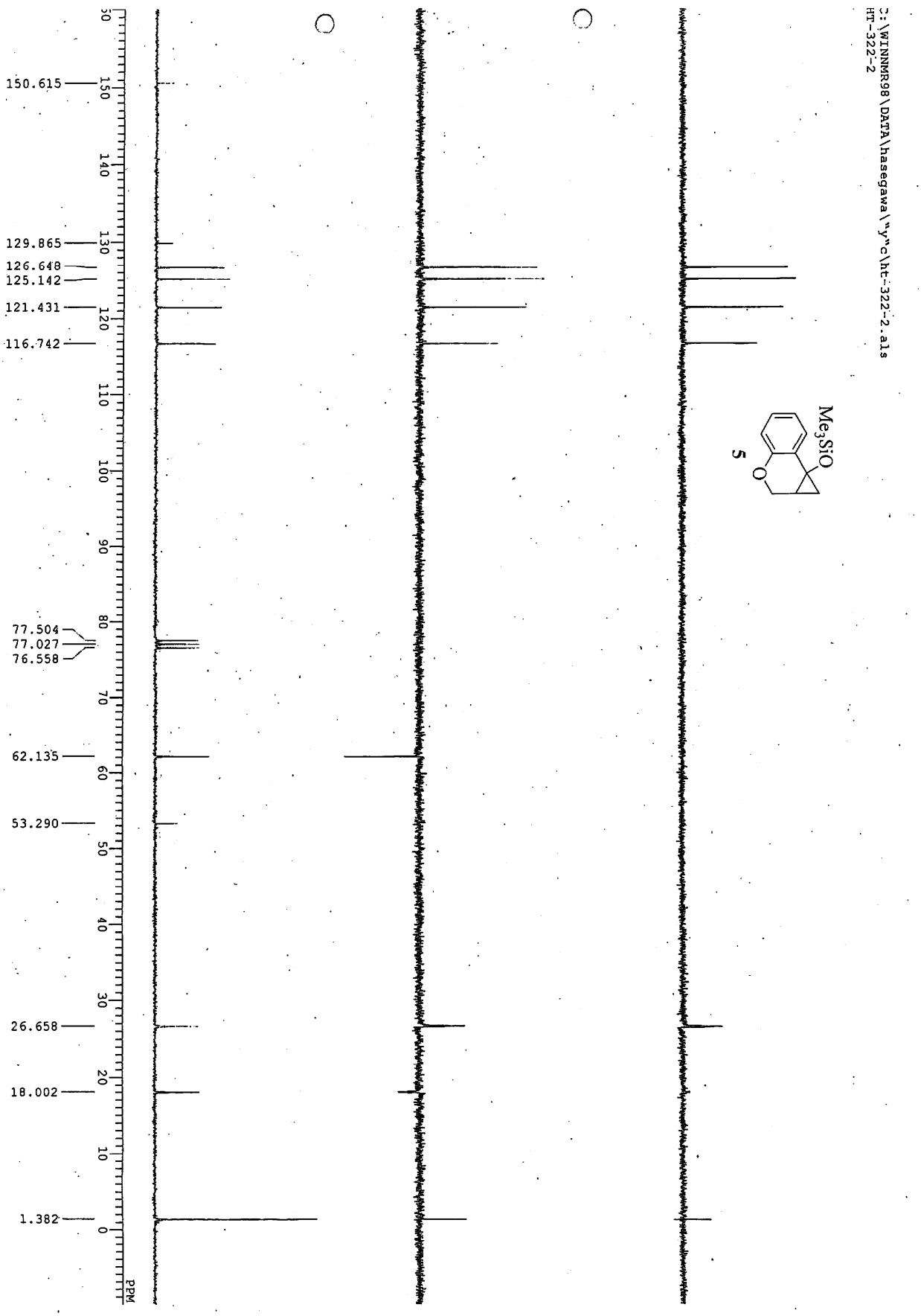
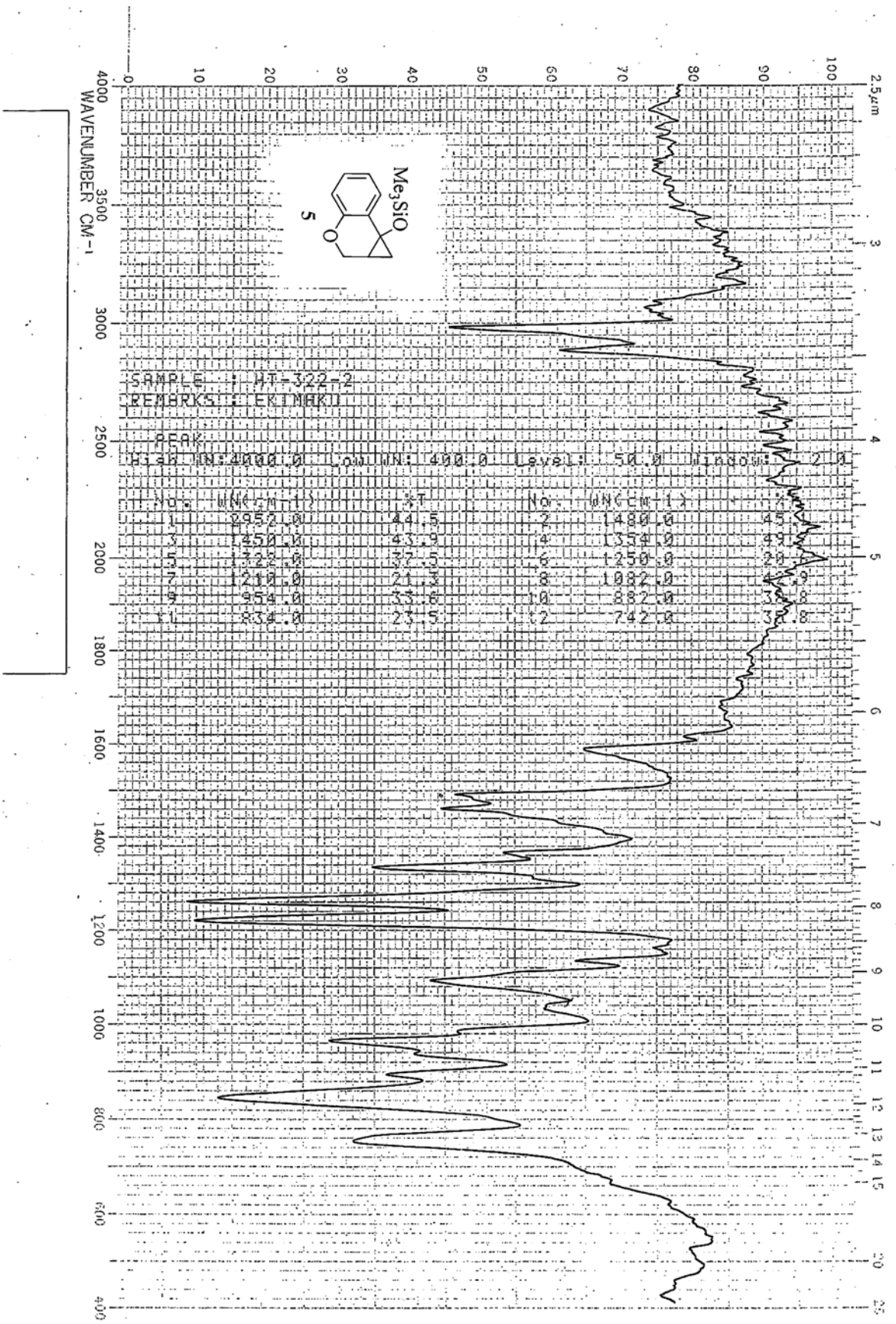
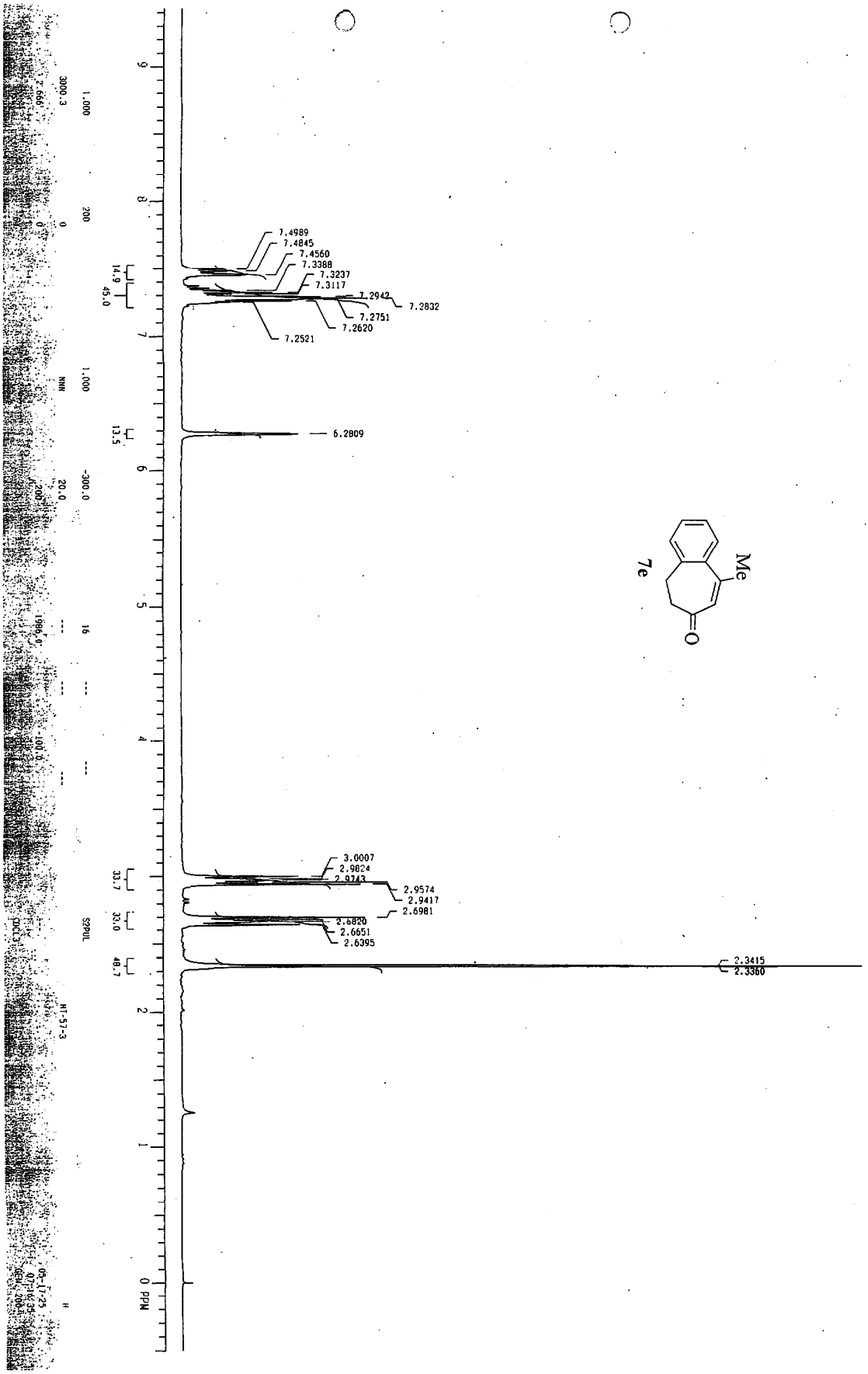
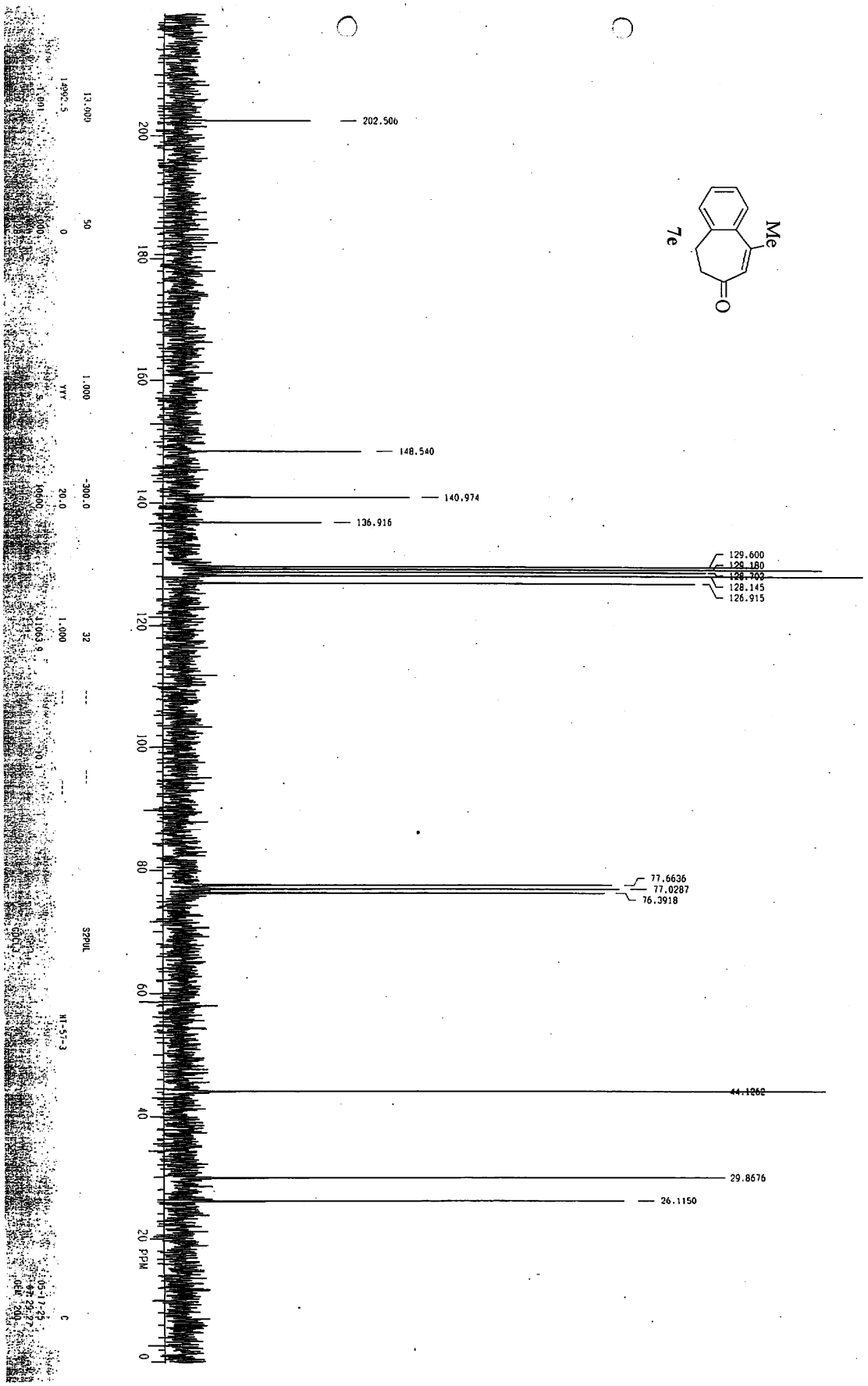
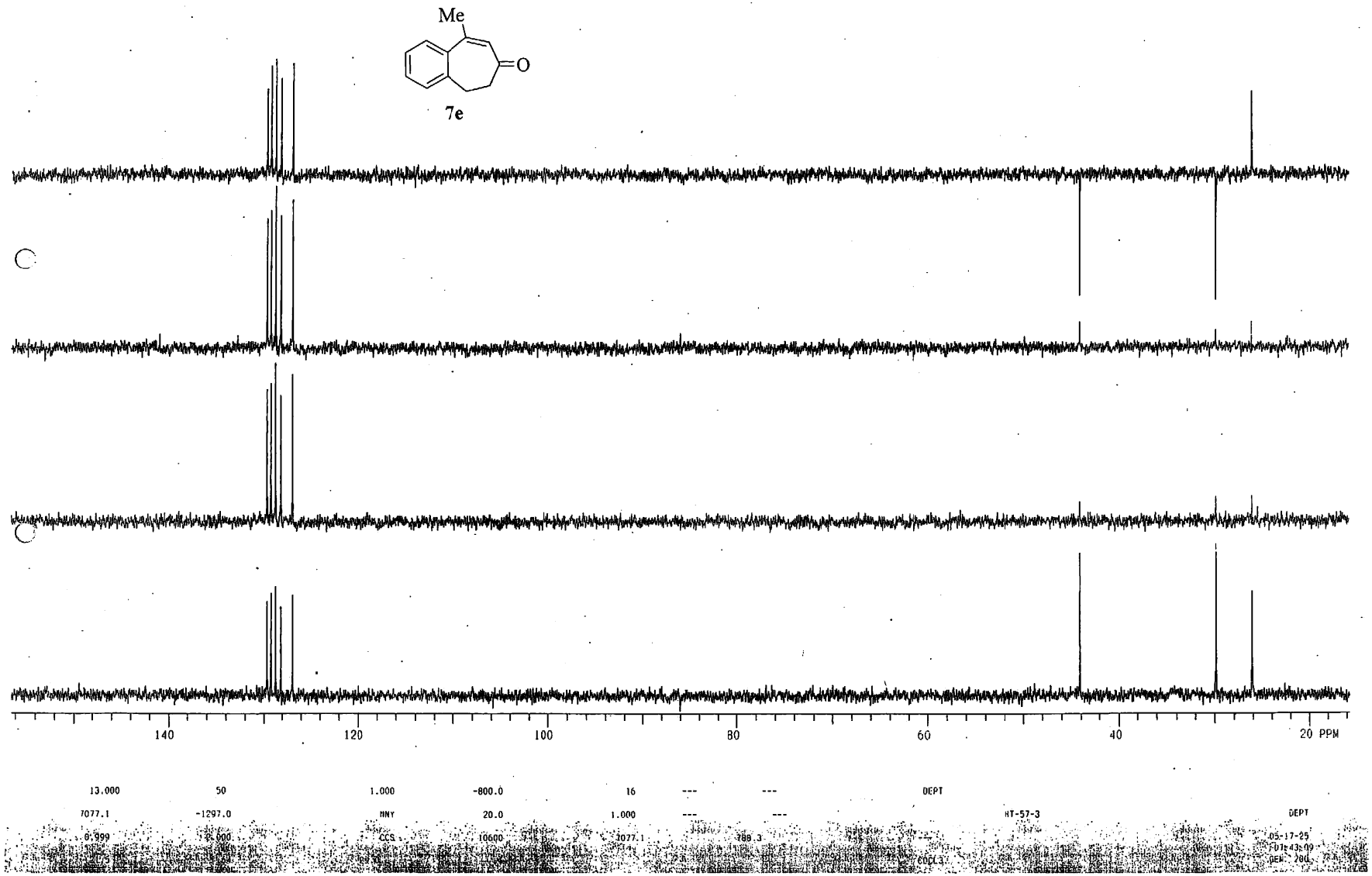
HT-322-2
Me3SiO
O
5
150.615
129.865
126.648
125.142
121.431
116.742
77.504
77.027
76.558
62.135
53.290
26.658
18.002
1.382
PPM

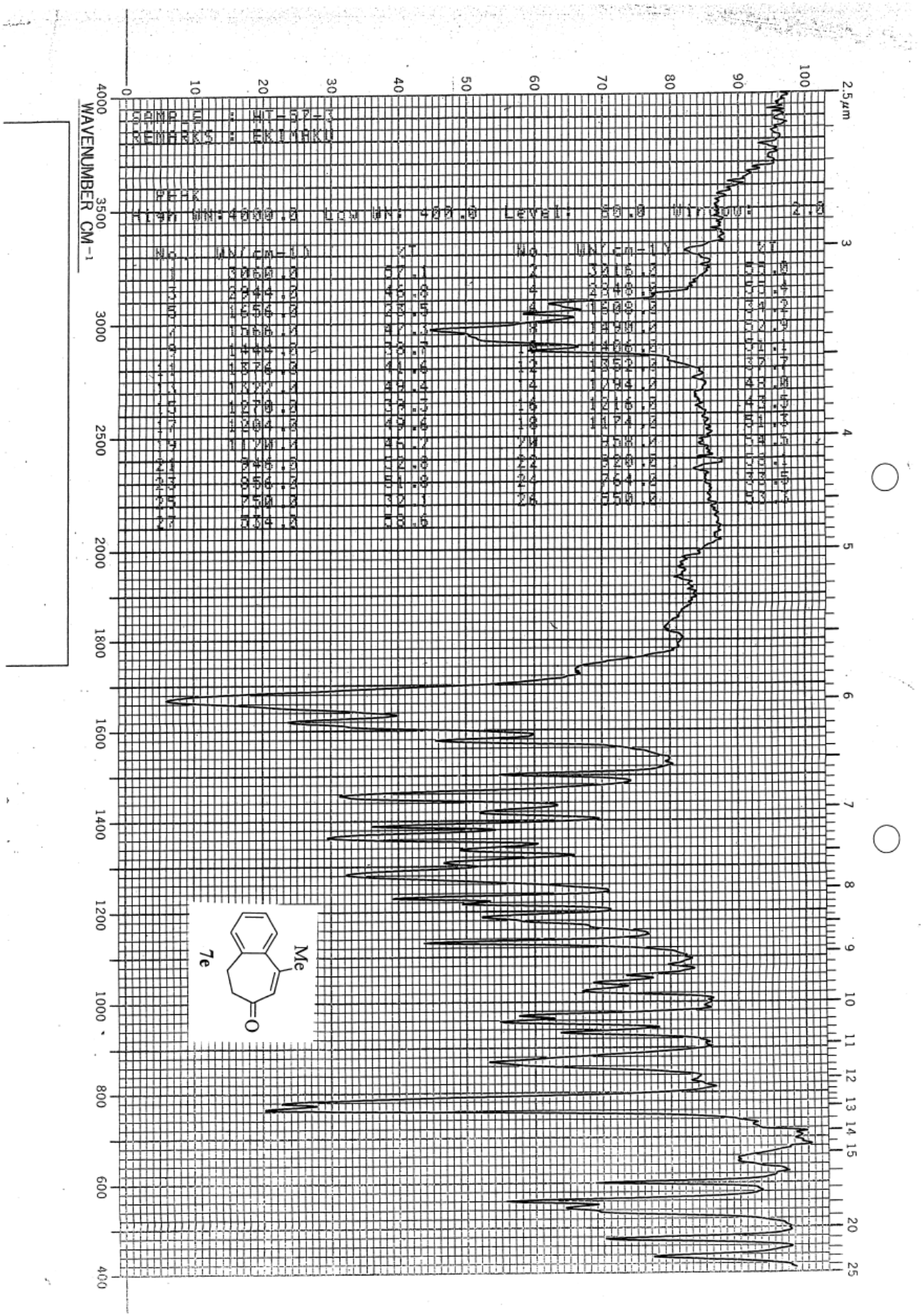

C:\WINNMR98\DATA\hasegawa\"y"c\ht-322-2.als
HT-322-2
Me3SiO
5
O
150.615
129.865
126.648
125.142
121.431
116.742
77.504
77.027
76.558
62.135
53.290
26.658
18.002
1.382
PPM

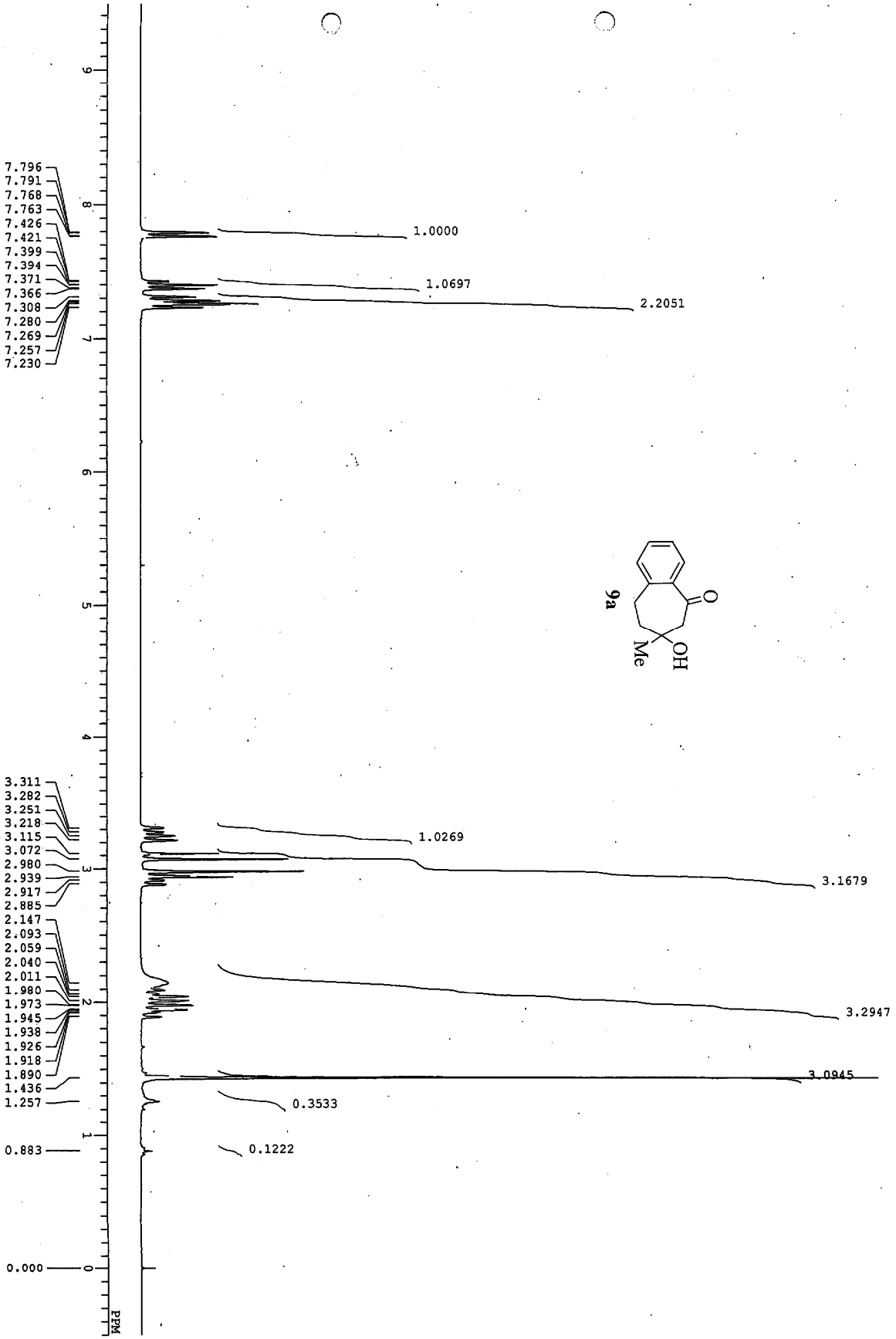

Me3SiO
5
O
SAMPLE : HT-322-2
REMARKS : EKIMAKU
PEAK
High WN:4000.0 Low WN: 400.0 Level: 50.0 Window: 2.0
No. WN(cm-1) %T No. WN(cm-1) %T
1 2952.0 44.5 2 1480.0 45.2
3 1450.0 43.9 4 1354.0 49.2
5 1725.0 72.5 6 1250.0 20
7 1210.0 21.3 8 1082.0 4.9
9 954.0 33.6 10 882.0 38.8
11 834.0 23.5 12 742.0 32.8
WAVENUMBER CM-1
4000
3500
3000
2500
2000
1800
1600
1400
1200
1000
800
600
400
2.5μm

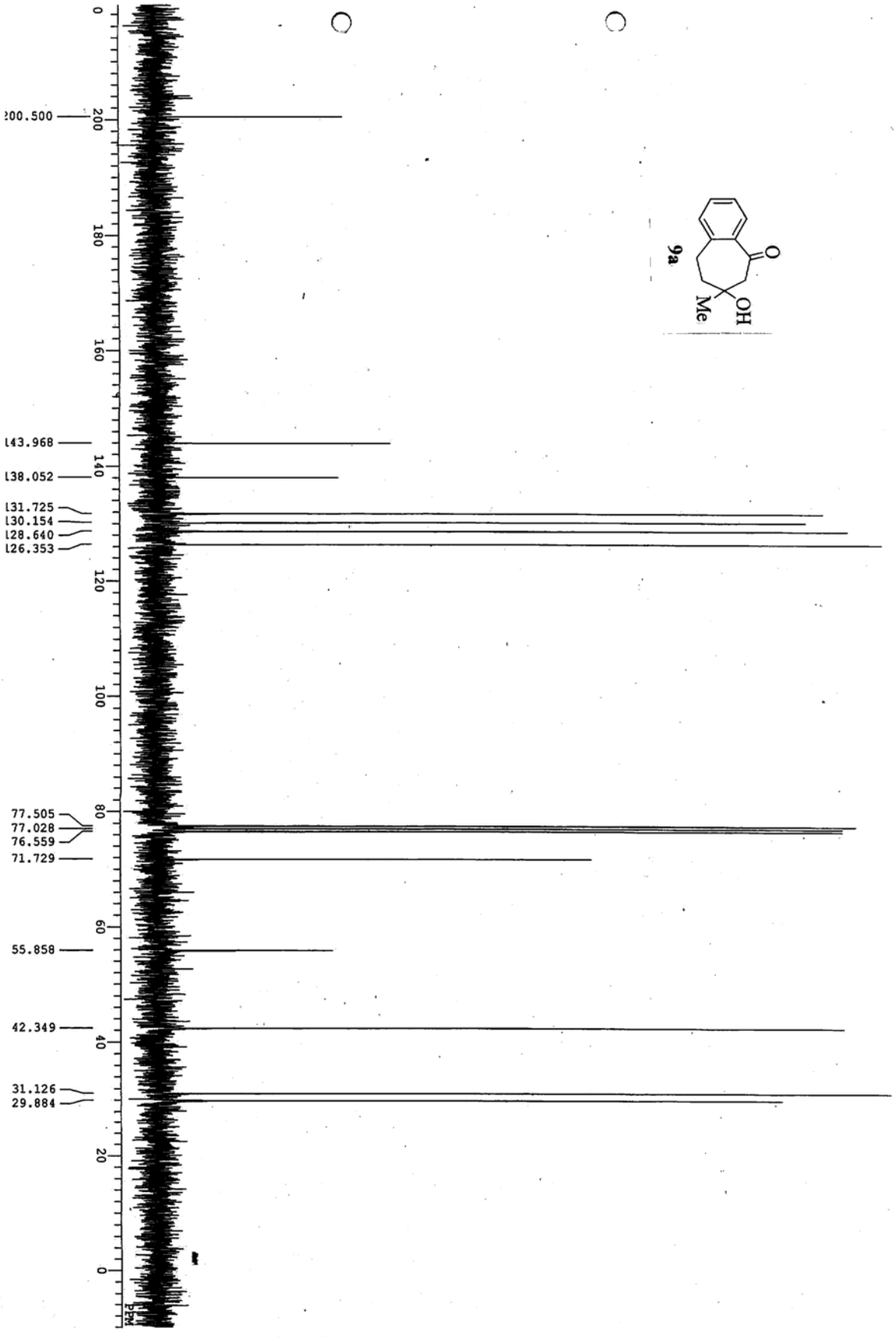

7.4989
7.4845
7.4560
7.3388
7.3237
7.3117
7.2942
7.2832
7.2751
7.2620
7.2521
6.2809
3.0007
2.9824
2.9743
2.9574
2.9417
2.6981
2.6820
2.6651
2.6395
2.3415
2.3360
Me
O
7e
PPM

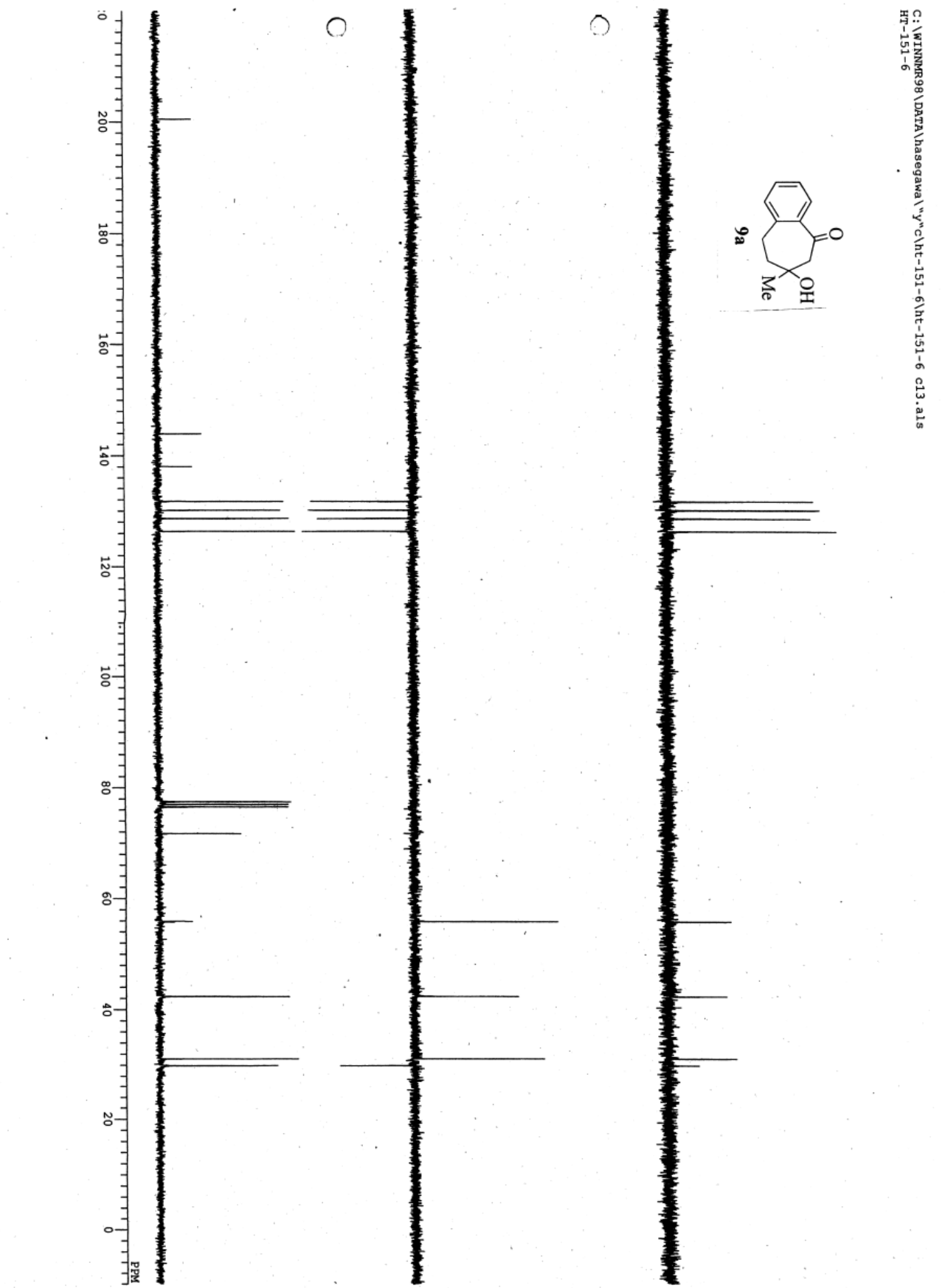

202.506
148.540
140.974
136.916
129.600
129.180
128.703
128.145
126.915
77.6636
77.0287
76.3918
44.1262
29.8676
26.1150
Me
7e
O
200
180
160
140
120
100
80
60
40
20
0
PPM

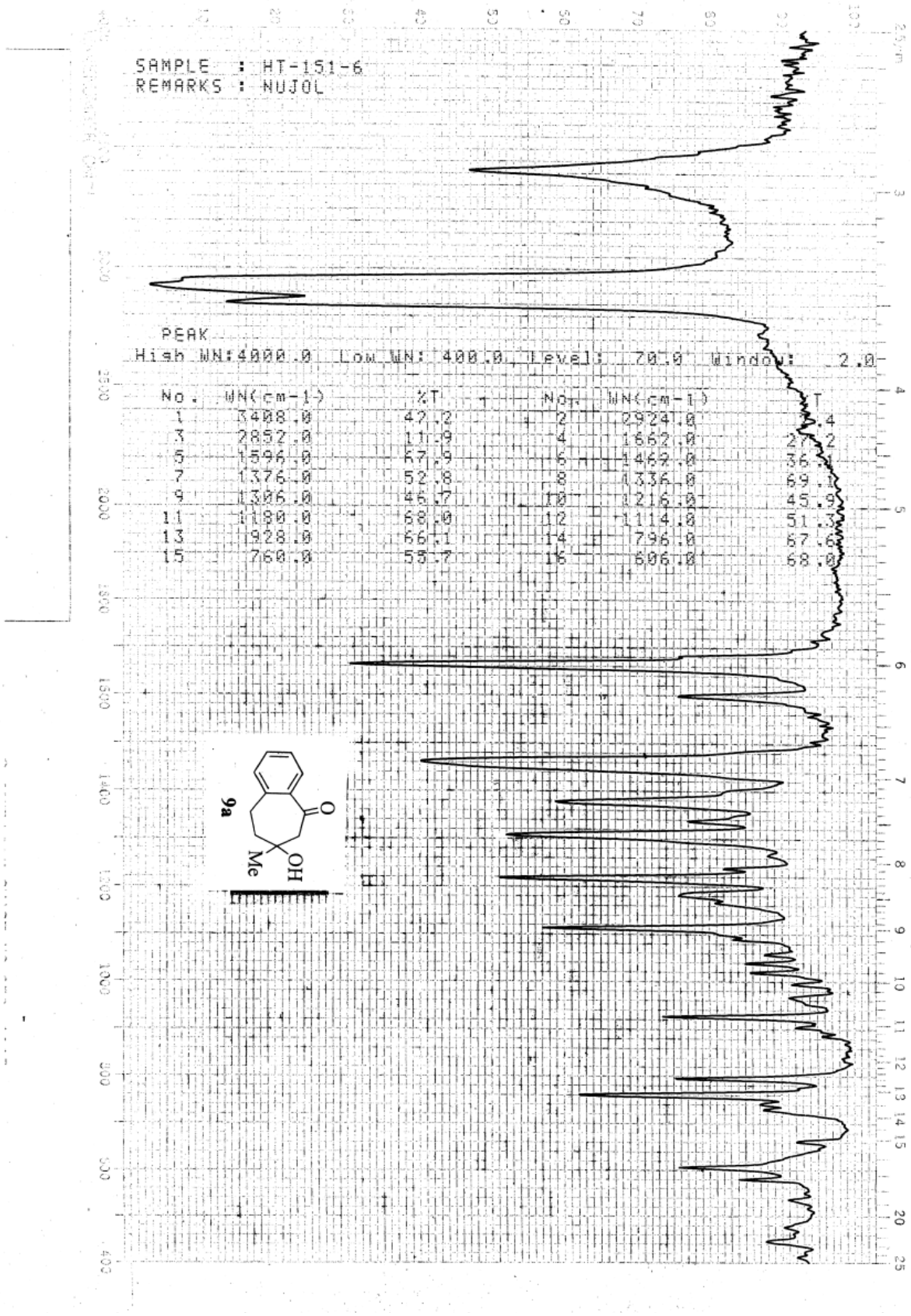

Me
O
7e
140
120
100
80
60
40
20 PPM
13.000
50
1.000
-800.0
16
DEPT
7077.1
-1297.0
WNY
20.0
1.000
HT-57-3
DEPT
0.999
2.000
CCS
10600
7077.1
788.3
CDCL3
05-17-25

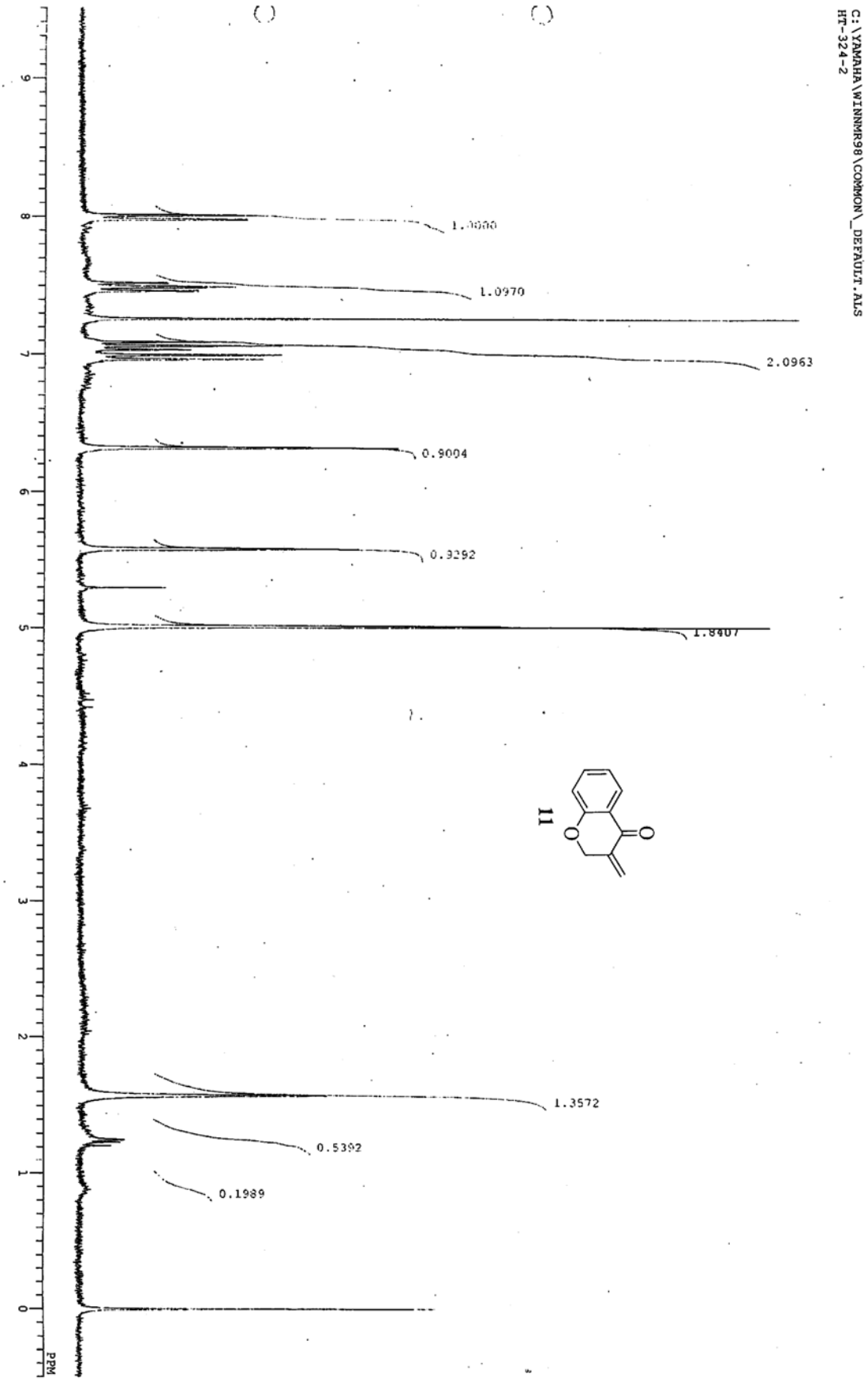

WAVENUMBER CM-1
Me
O
7e

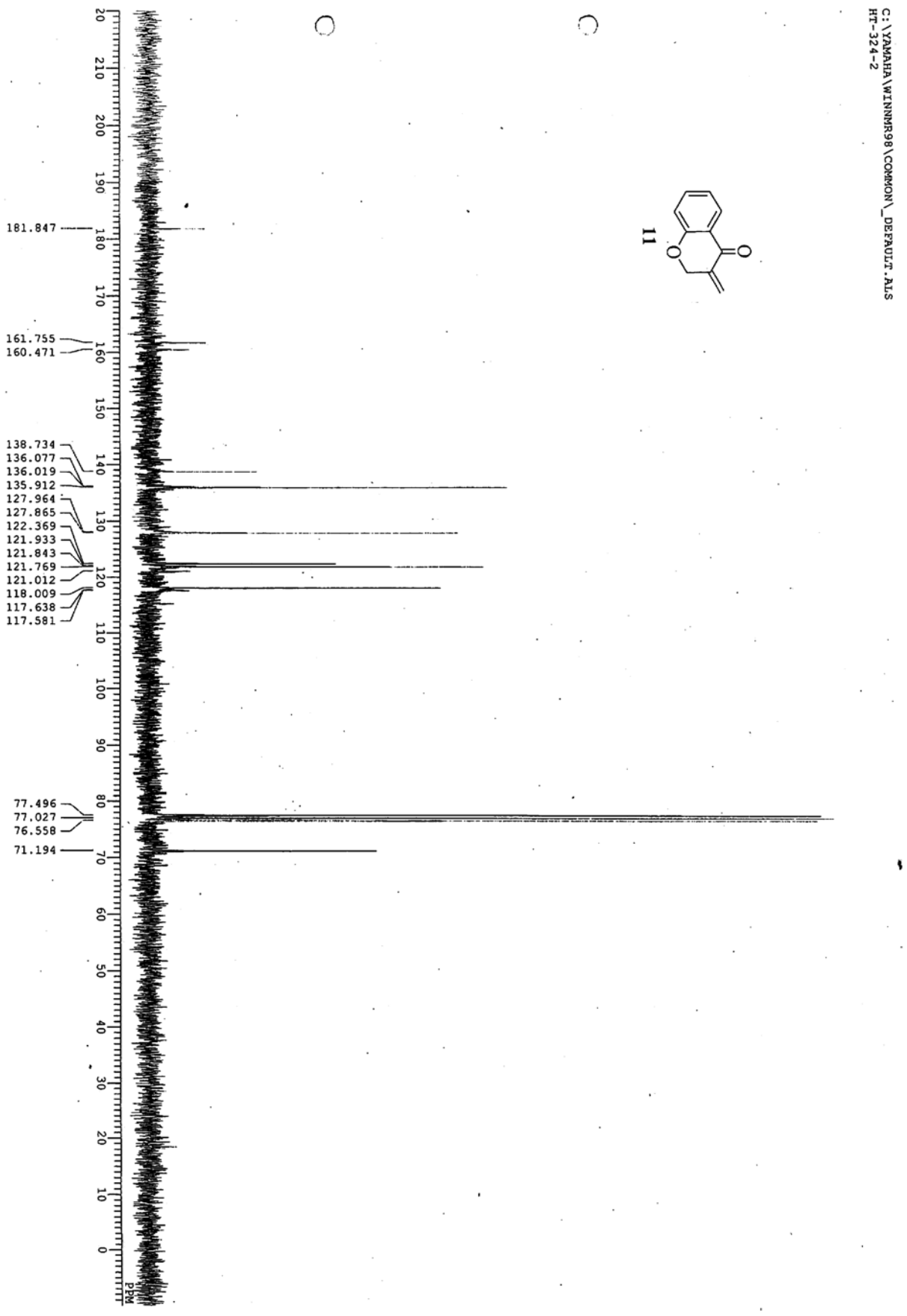

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-151-6
7.796
7.791
7.768
7.763
7.426
7.421
7.399
7.394
7.371
7.366
7.308
7.280
7.269
7.257
7.230
3.311
3.282
3.251
3.218
3.115
3.072
2.980
2.939
2.917
2.885
2.147
2.093
2.059
2.040
2.011
1.980
1.973
1.945
1.938
1.926
1.918
1.890
1.436
1.257
0.883
0.000
1.0000
1.0697
2.2051
1.0269
3.1679
3.2947
3.0945
0.3533
0.1222
9a
O
OH
Me
ppm

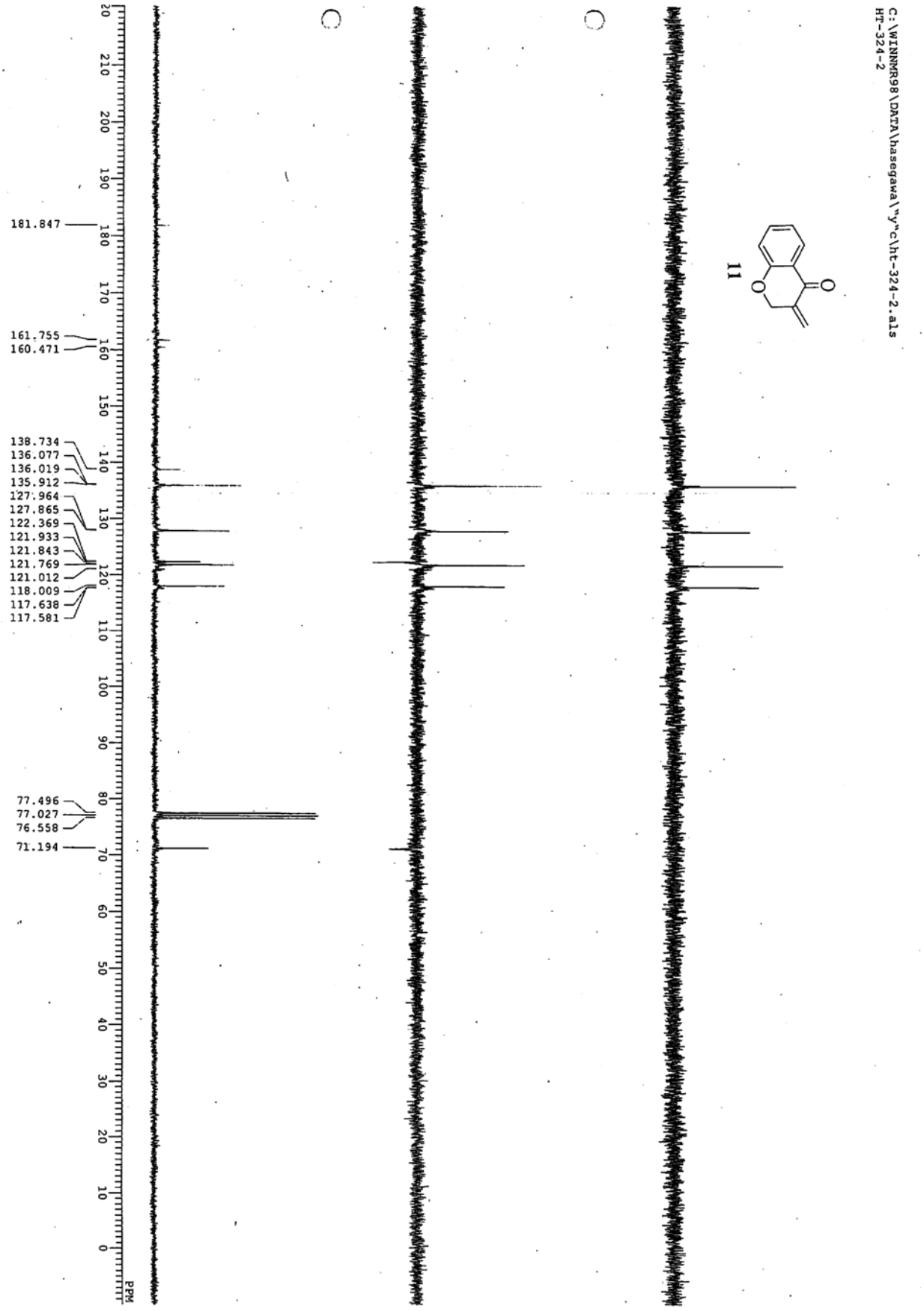

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-151-6
9a
O
OH
Me
200.500
143.968
138.052
131.725
130.154
128.640
126.353
77.505
77.028
76.559
71.729
55.858
42.349
31.126
29.884
0
20
40
60
80
100
120
140
160
180
200
0
PPM

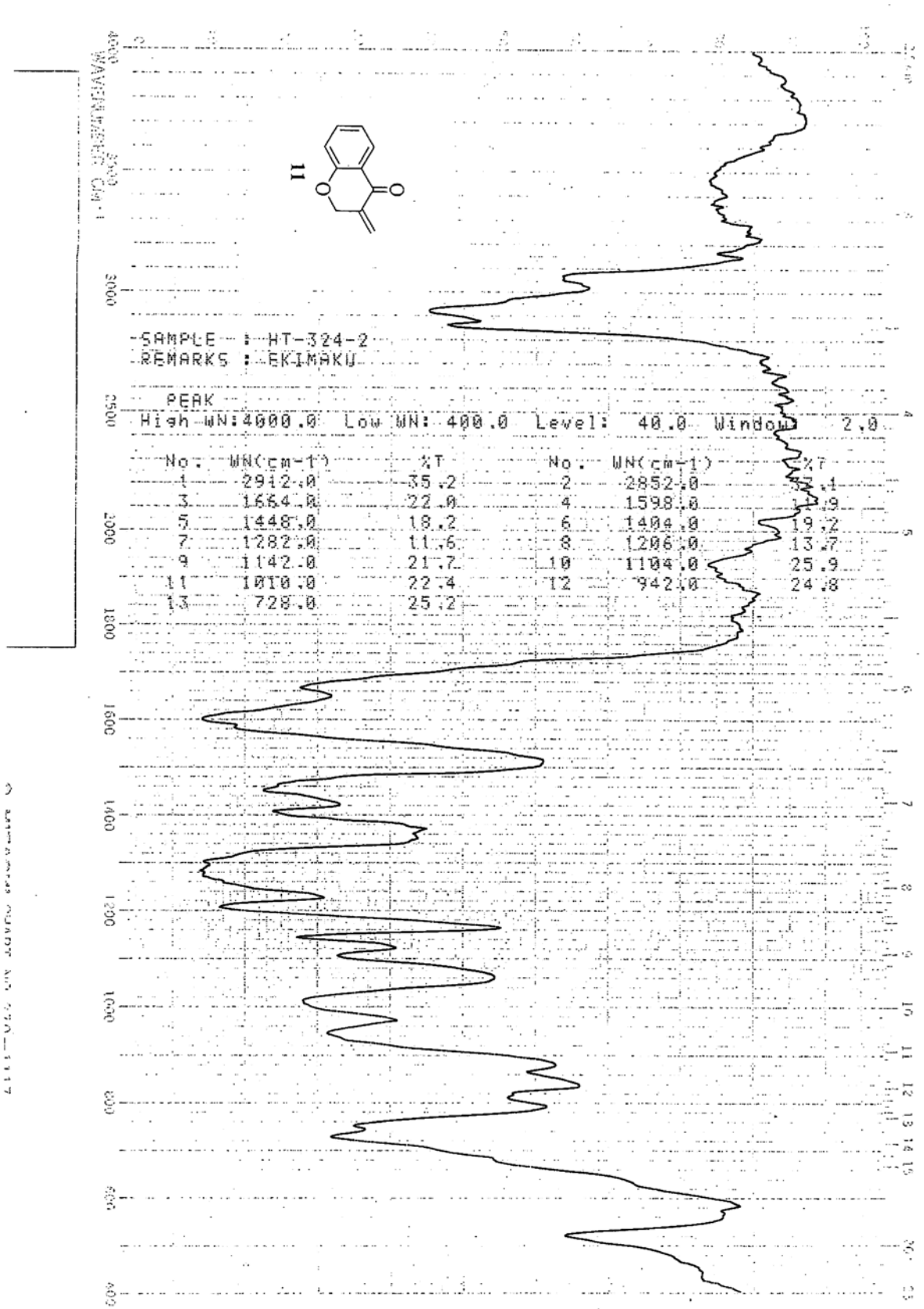

C:\WINNMR98\DATA\hasegawa\"y"c\ht-151-6\ht-151-6 c13.als
HT-151-6
O
OH
Me
9a
200
180
160
140
120
100
80
60
40
20
0
PPM

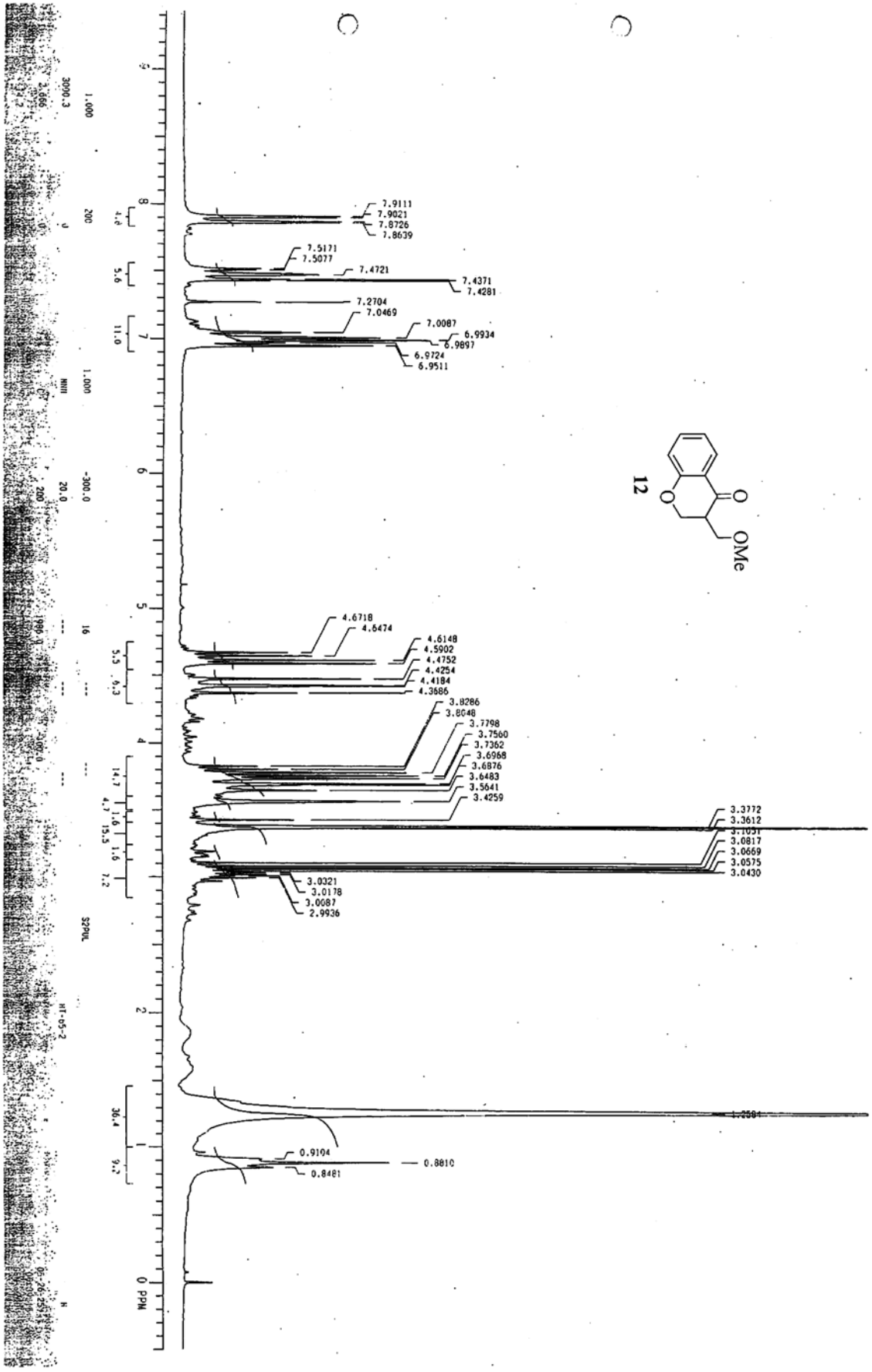

SAMPLE : HT-151-6
REMARKS : NUJOL
PEAK
High WN:4000.0 Low WN: 400.0 Level: 70.0 Windo: 2.0
No. WN(cm-1) %T
1 3408.0 42.2
3 2852.0 11.9
5 1596.0 67.9
7 1376.0 52.8
9 1306.0 46.7
11 1180.0 68.0
13 928.0 66.1
15 760.0 55.7
No. WN(cm-1) T
2 2924.0 .4
4 1662.0 27.2
6 1462.0 36.1
8 1336.0 69.
10 1216.0 45.9
12 1114.0 51.3
14 796.0 67.6
16 606.0 68.0
9a
O
OH
Me

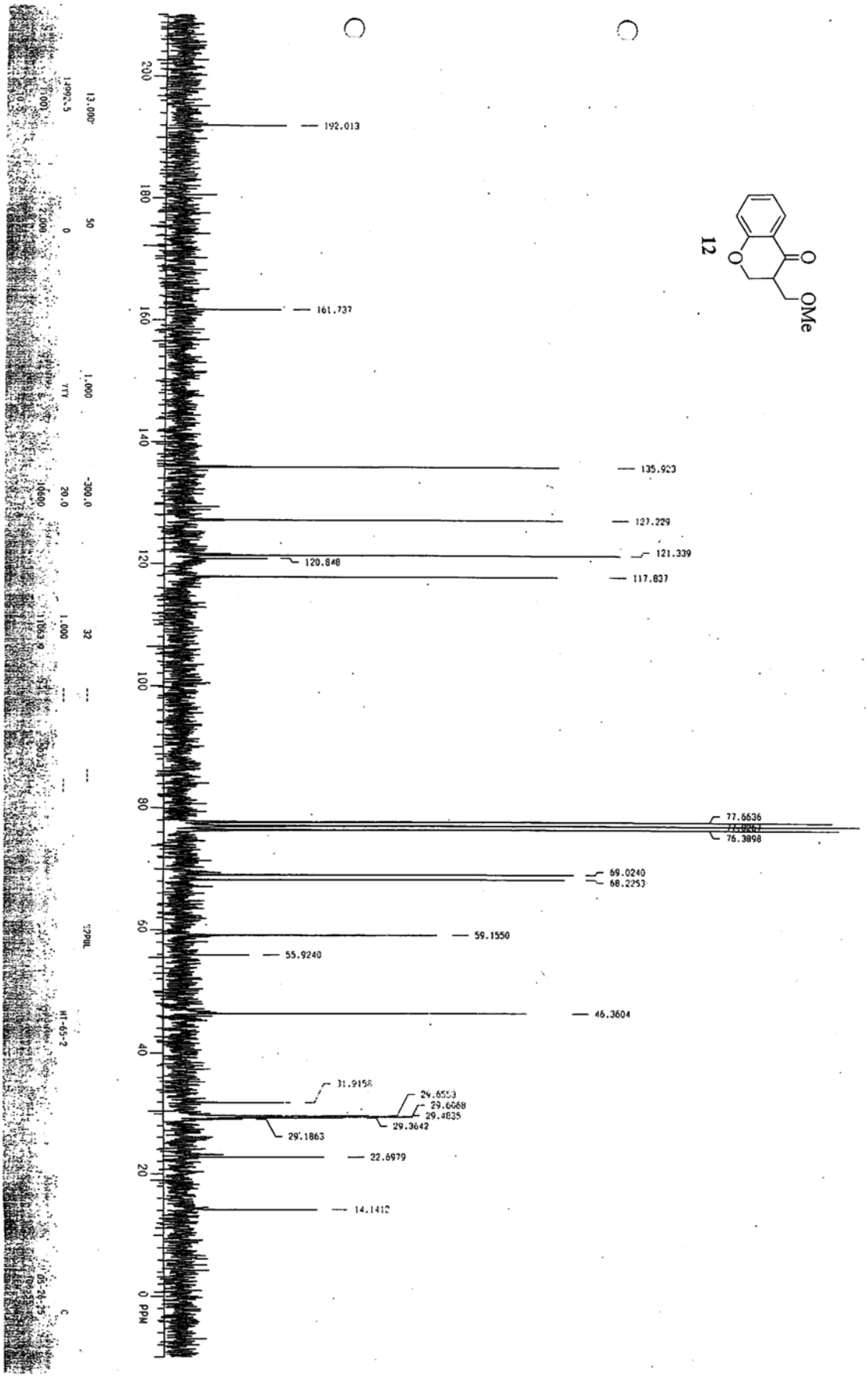

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-324-2
1.0000
1.0970
2.0963
0.9004
0.9292
1.8407
1.3572
0.5392
0.1989
PPM
11

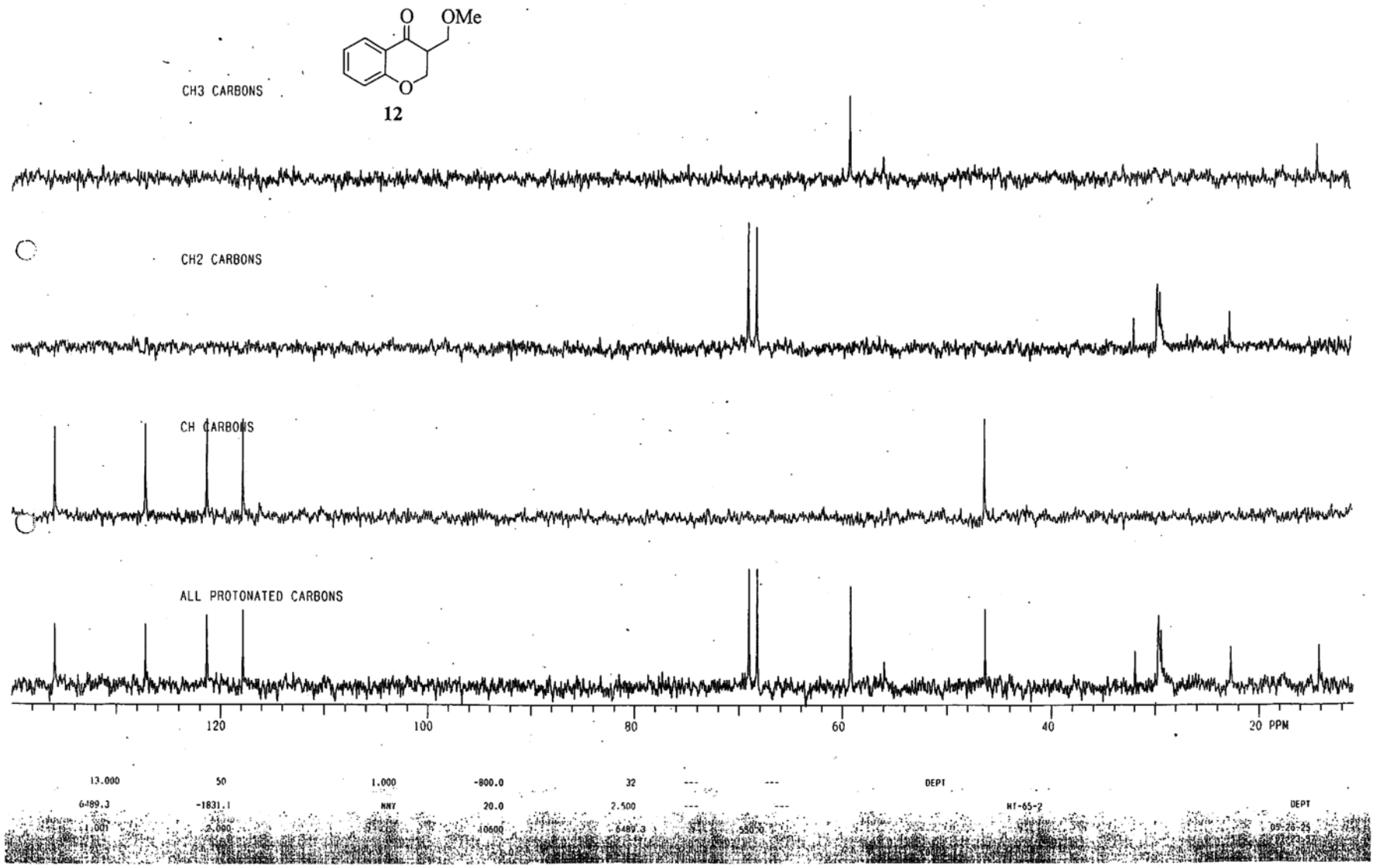

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-324-2
11
181.847
161.755
160.471
138.734
136.077
136.019
135.912
127.964
127.865
122.369
121.933
121.843
121.769
121.012
118.009
117.638
117.581
77.496
77.027
76.558
71.194
PPM

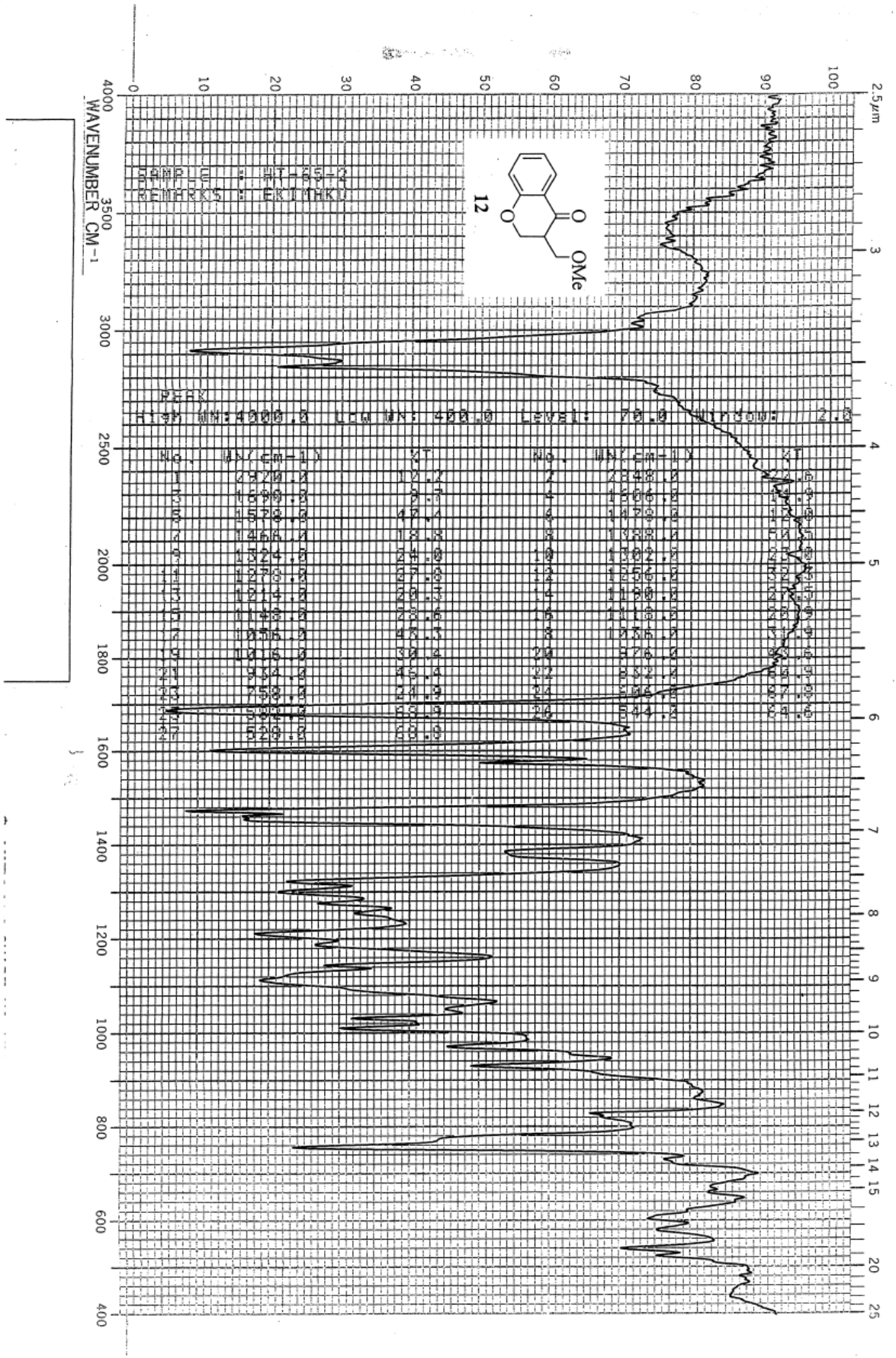

C:\WINNMR98\DATA\hasegawa\"y"c\ht-324-2.als
HT-324-2
11
181.847
161.755
160.471
138.734
136.077
136.019
135.912
127.964
127.865
122.369
121.933
121.843
121.769
121.012
118.009
117.638
117.581
77.496
77.027
76.558
71.194
PPM


11
SAMPLE : HT-324-2
REMARKS : EKIMAKU
PEAK
High WN:4000.0 Low WN: 400.0 Level: 40.0 Window 2.0
No. WN(cm-1) %T No. WN(cm-1) %T
1 2912.0 35.2 2 2852.0 32.1
3 1664.0 22.0 4 1598.0 17.9
5 1448.0 18.2 6 1404.0 19.2
7 1282.0 11.6 8 1206.0 13.7
9 1142.0 21.7 10 1104.0 25.9
11 1010.0 22.4 12 942.0 24.8
13 728.0 25.2

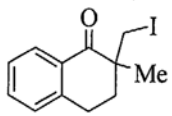

12
OMe
7.9111
7.9021
7.8726
7.8639
7.5171
7.5077
7.4721
7.4371
7.4281
7.2704
7.0469
7.0087
6.9934
6.9897
6.9724
6.9511
4.6718
4.6474
4.6148
4.5902
4.4752
4.4254
4.4184
4.3686
3.8286
3.8048
3.7798
3.7560
3.7362
3.6968
3.6876
3.6483
3.5641
3.4259
3.3772
3.3612
3.0817
3.0669
3.0575
3.0430
3.0321
3.0178
3.0087
2.9936
0.9104
0.8810
0.8481
PPM


192.013
161.737
135.923
127.229
121.339
120.848
117.837
77.6636
76.3898
69.0240
68.2253
59.1550
55.9240
46.3604
31.9158
29.6553
29.6088
29.4835
29.3642
29.1863
22.6979
14.1412
200
180
160
140
120
100
80
60
40
20
0 PPM
12
O
O
OMe

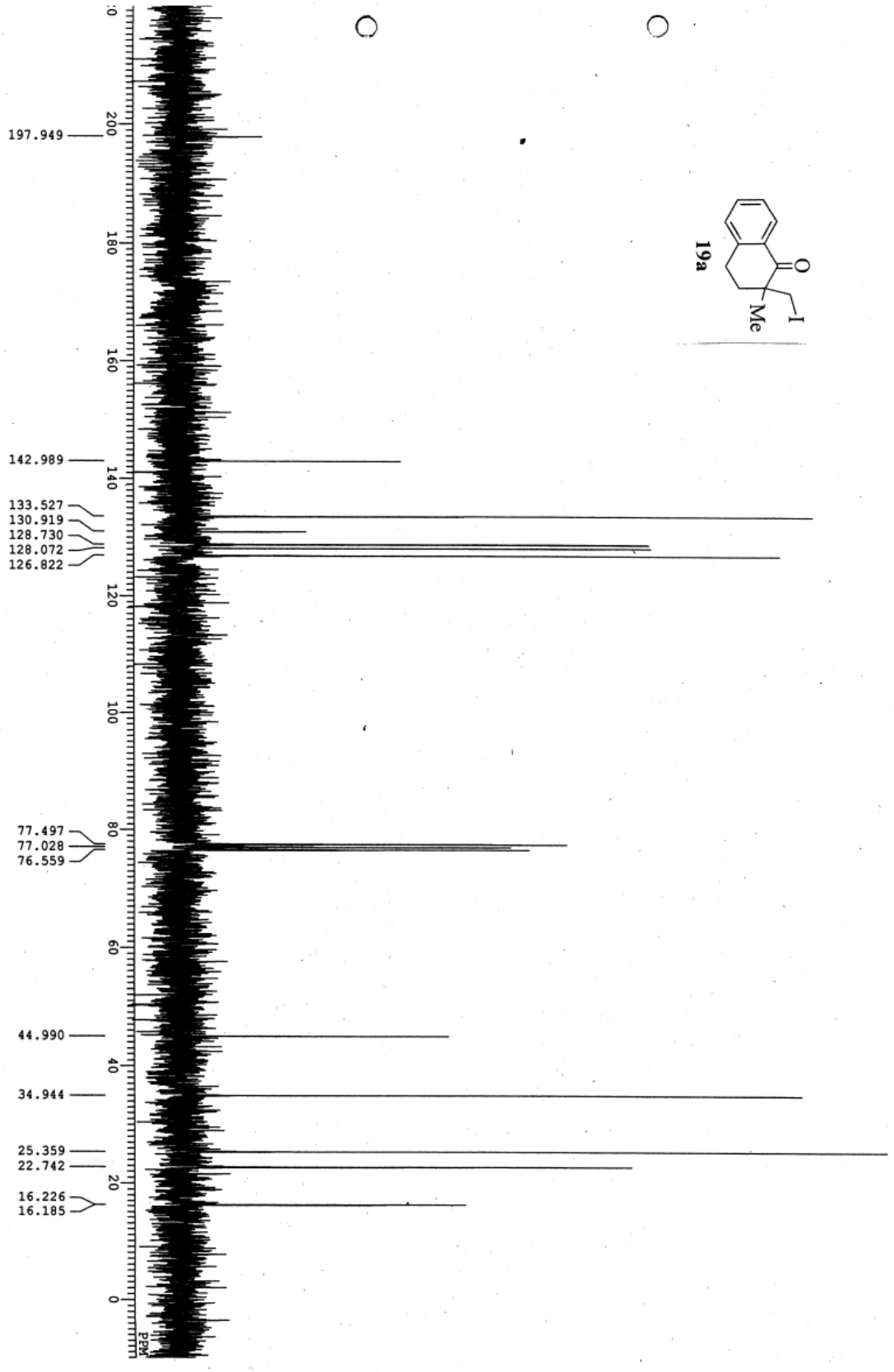

O
OMe
O
12
CH3 CARBONS
CH2 CARBONS
CH CARBONS
ALL PROTONATED CARBONS
120
100
80
60
40
20 PPM
13.000
50
1.000
-800.0
32
DEPT
6489.3
-1831.1
NNY
20.0
2.500
HT-65-2
DEPT
1.001
2.000
10600
6489.3
550.0
09-26-25

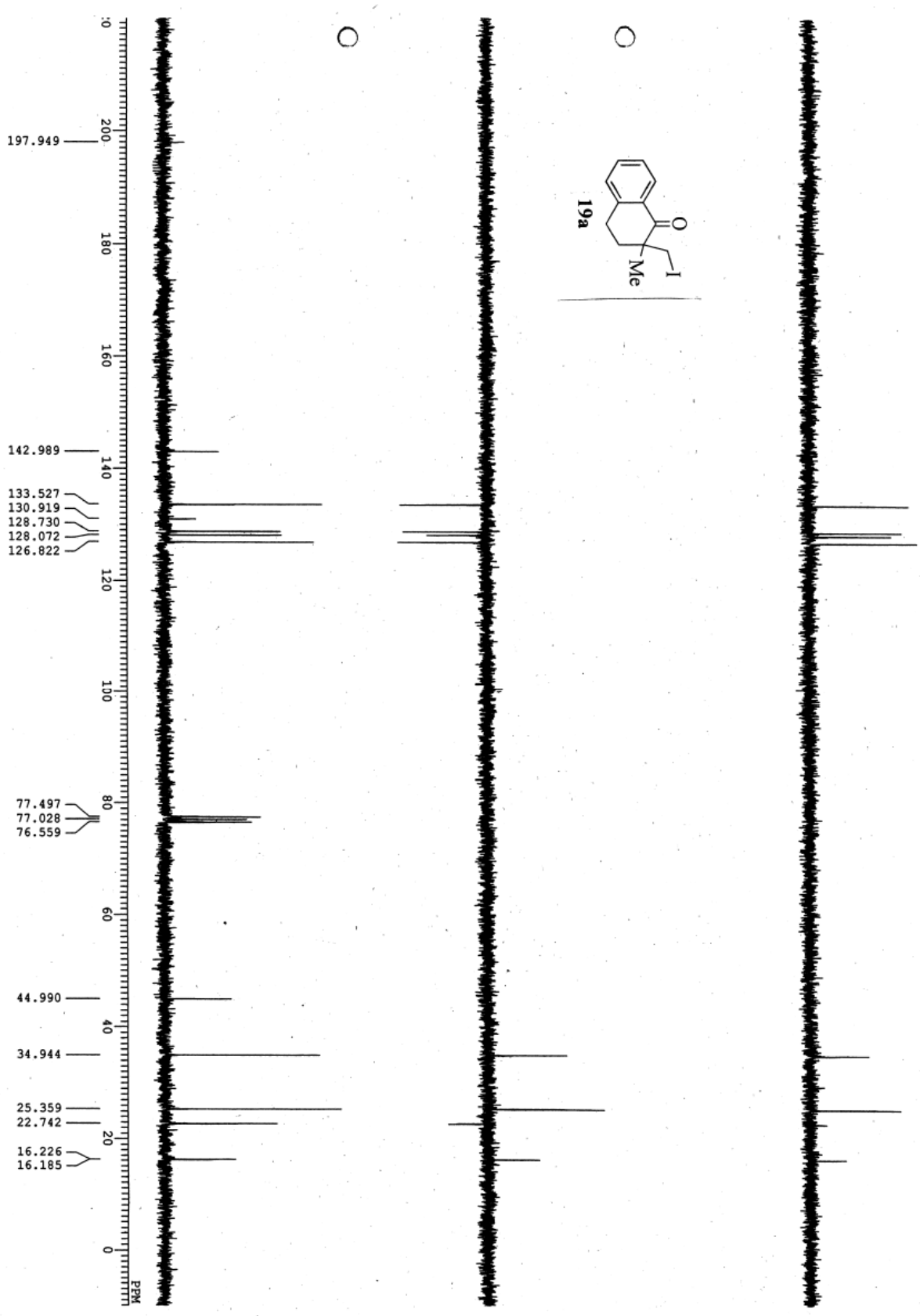

12
OMe
O
O
WAVENUMBER CM-1
2.5μm

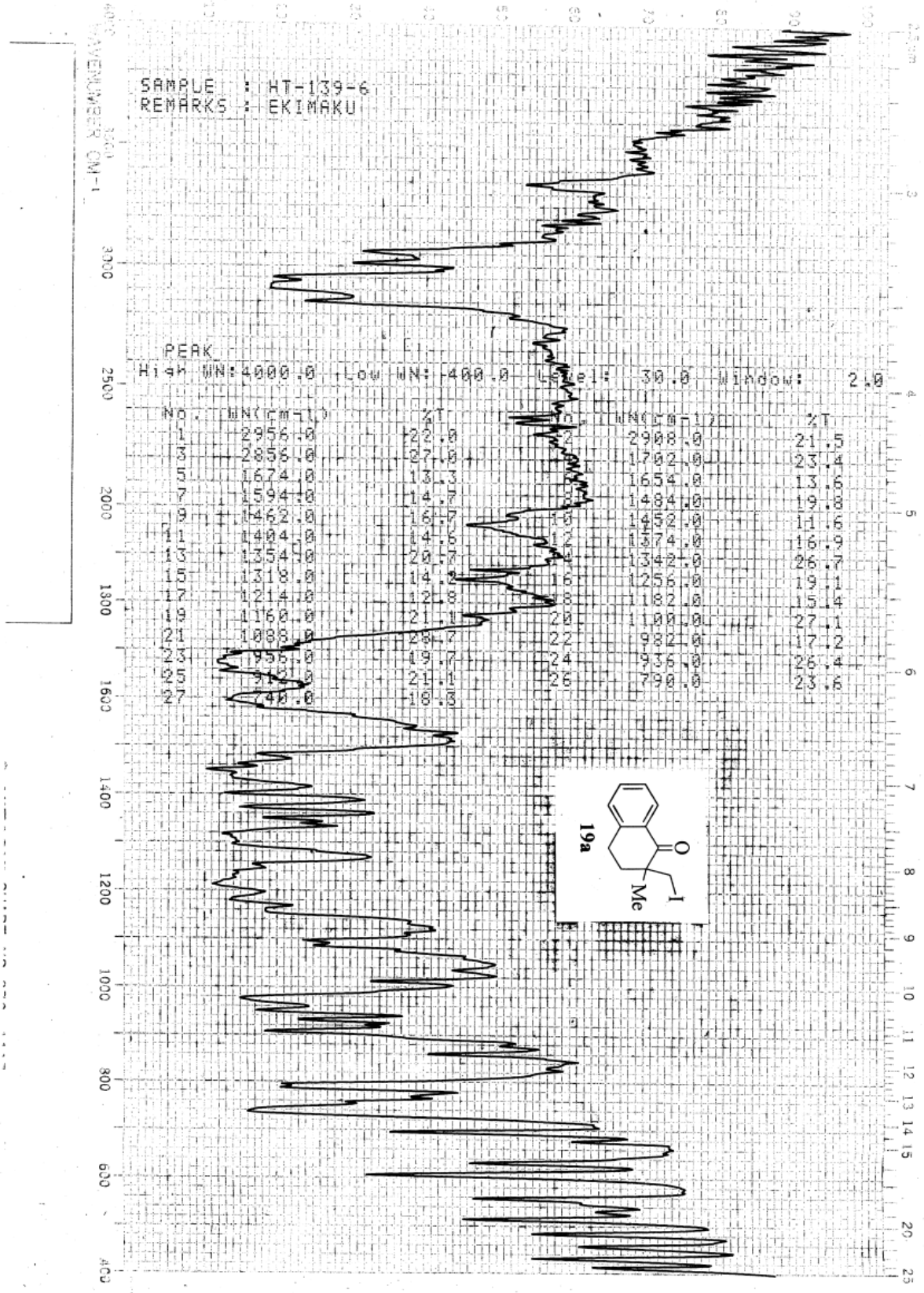
C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
ET-139-6

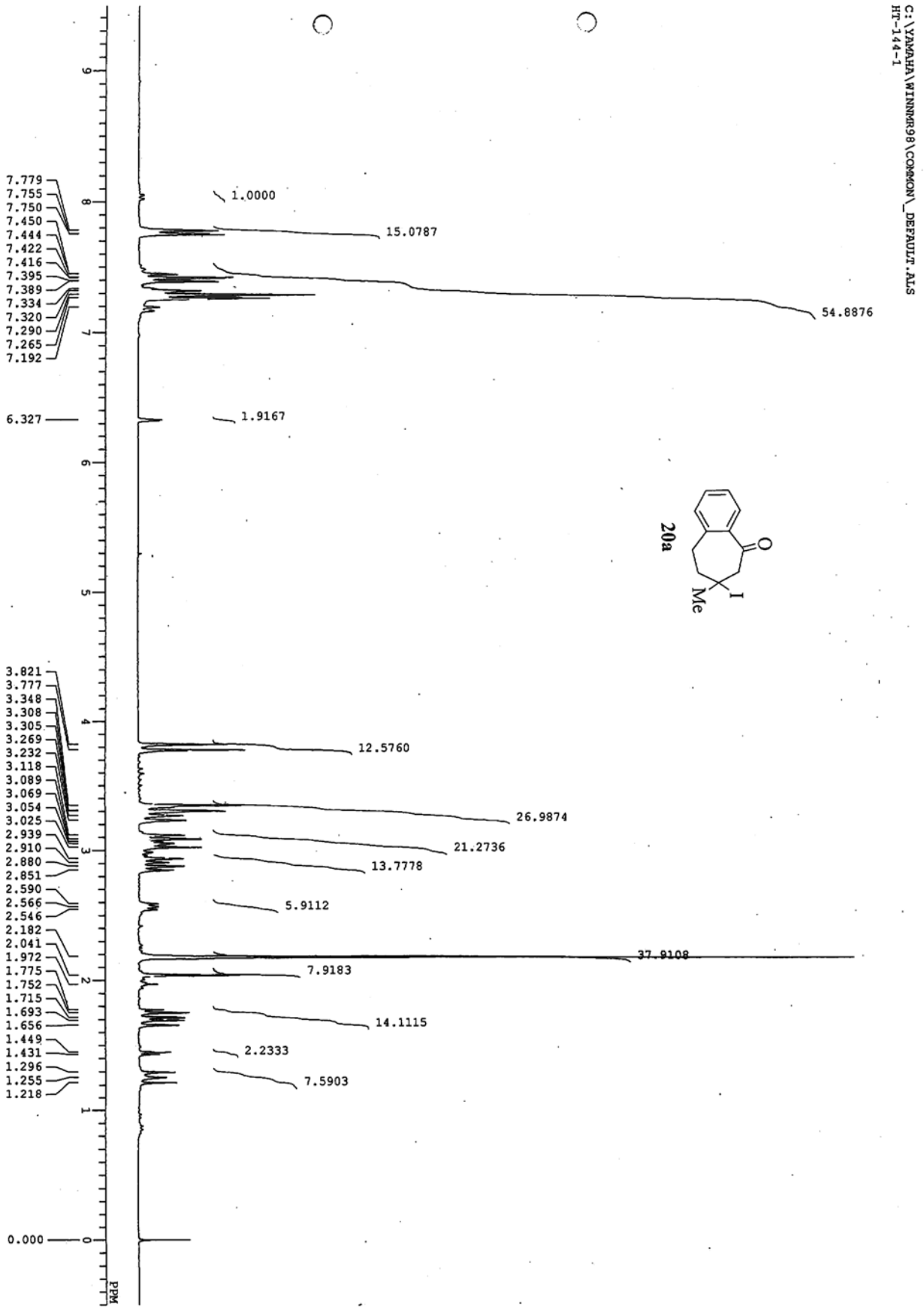


19a


C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-139-6
19a
O
I
Me
197.949
142.989
133.527
130.919
128.730
128.072
126.822
77.497
77.028
76.559
44.990
34.944
25.359
22.742
16.226
16.185
PPM


C:\WINNMR98\DATA\hasegawa\"y"c\ht-139-6\ht-139-6 c13.als
HT-139-6
19a
O
I
Me
197.949
142.989
133.527
130.919
128.730
128.072
126.822
77.497
77.028
76.559
44.990
34.944
25.359
22.742
16.226
16.185
PPM


SAMPLE : HT-139-6
REMARKS : EKIMAKU
PEAK
High WN: 4000.0 Low WN: 400.0 Level: 30.0 Window: 2.0
NO. WN(cm-1) %T
1 2956.0 22.0
3 2856.0 27.0
5 1674.0 13.3
7 1594.0 14.7
9 1462.0 16.7
11 1404.0 14.6
13 1354.0 20.7
15 1318.0 14.9
17 1214.0 12.8
19 1160.0 21.1
21 1088.0 26.2
23 956.0 19.7
25 912.0 21.1
27 740.0 18.3
WN(cm-1) %T
2908.0 21.5
1702.0 23.4
1654.0 13.6
1484.0 19.8
1452.0 11.6
1374.0 16.9
1342.0 26.7
1256.0 19.1
1182.0 15.4
1100.0 27.1
982.0 17.2
936.0 26.4
790.0 23.6
19a
O
Me
I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7.395
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7.320
7.290
7.265
7.192
6.327
3.821
3.777
3.348
3.308
3.305
3.269
3.232
3.118
3.089
3.069
3.054
3.025
2.939
2.910
2.880
2.851
2.590
2.566
2.546
2.182
2.041
1.972
1.775
1.752
1.715
1.693
1.656
1.449
1.431
1.296
1.255
1.218
0.000
1.0000
15.0787
54.8876
1.9167
12.5760
26.9874
21.2736
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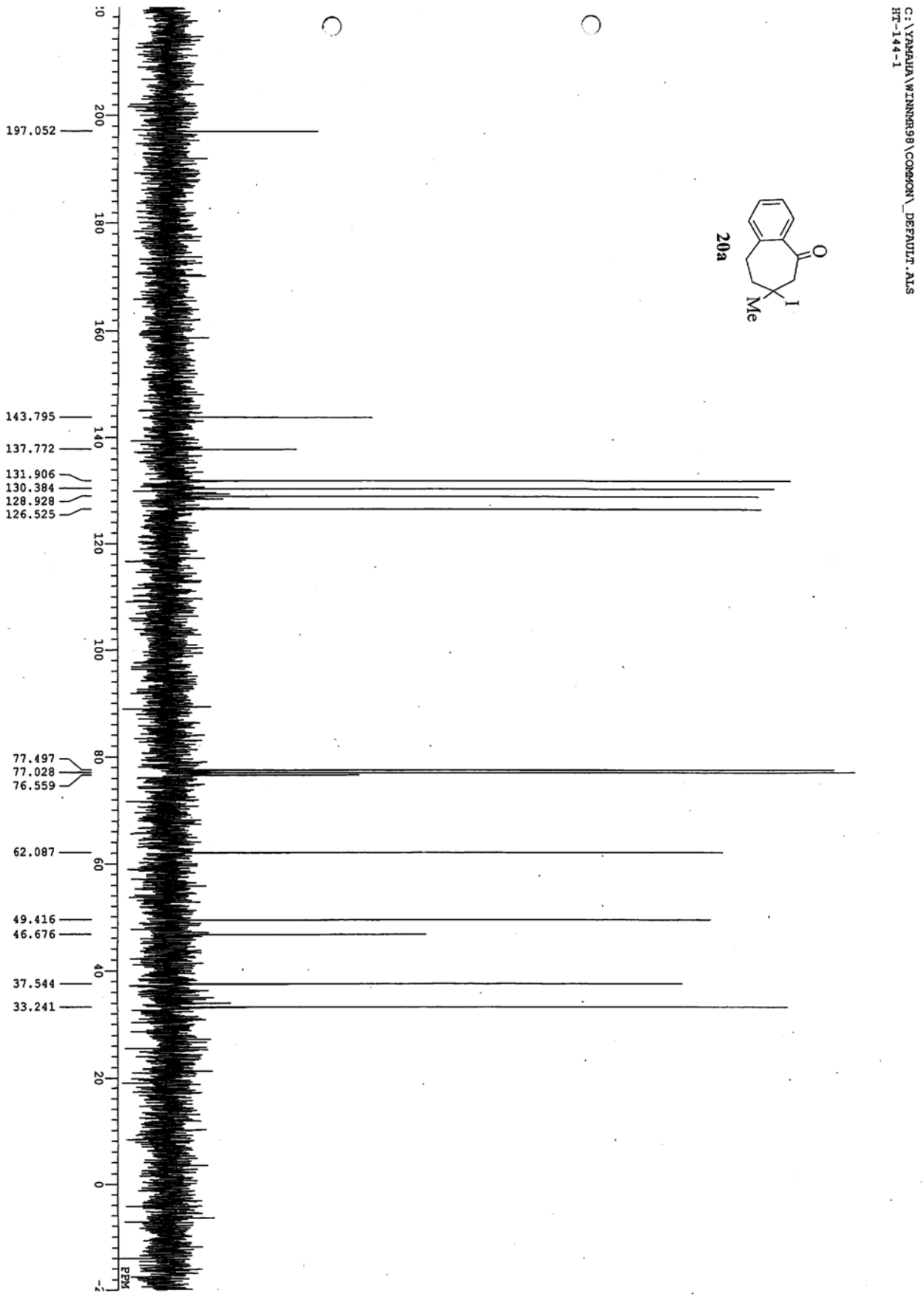
37.9108
7.9183
14.1115
2.2333
7.5903
20a
O
I
Me
PPM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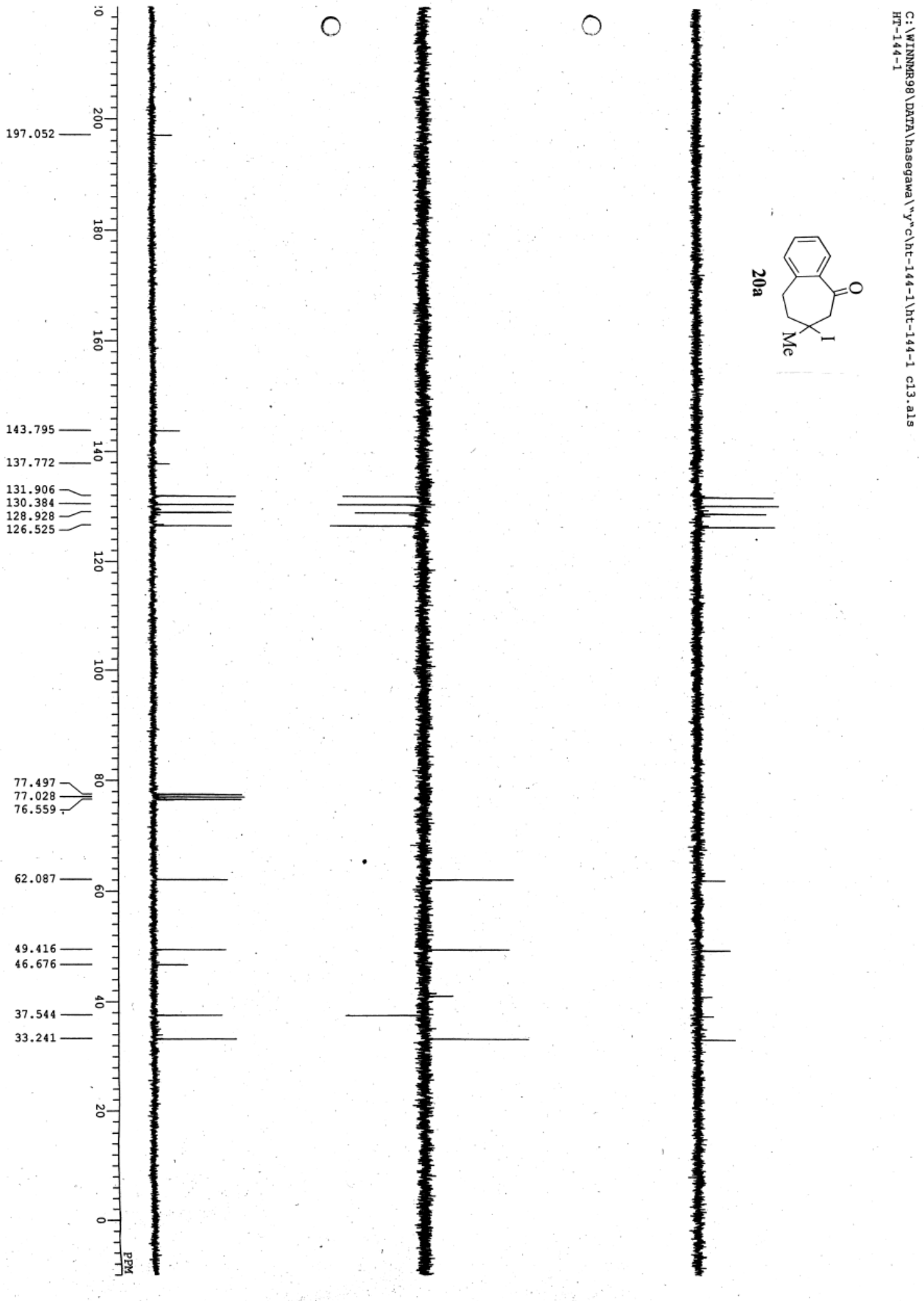
137.772
131.906
130.384
128.928
126.525
77.497
77.028
76.559
62.087
49.416
46.676
37.544
33.241
PPM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143.795
137.772
131.906
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126.525
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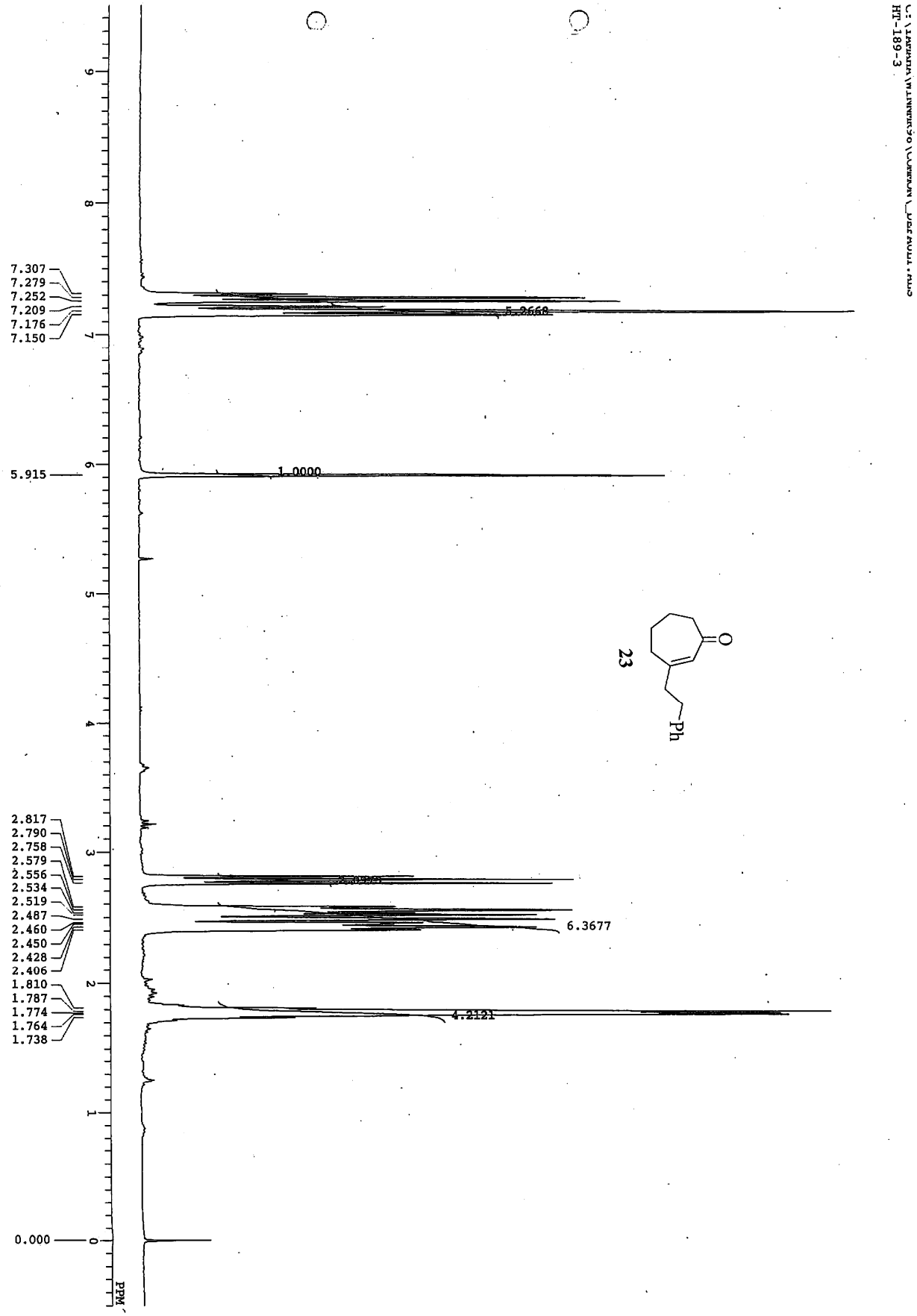
49.416
46.676
37.544
33.241
PPM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7.279
7.252
7.209
7.176
7.150
5.915
2.817
2.790
2.758
2.579
2.556
2.534
2.519
2.487
2.460
2.450
2.428
2.406
1.810
1.787
1.774
1.764
1.738
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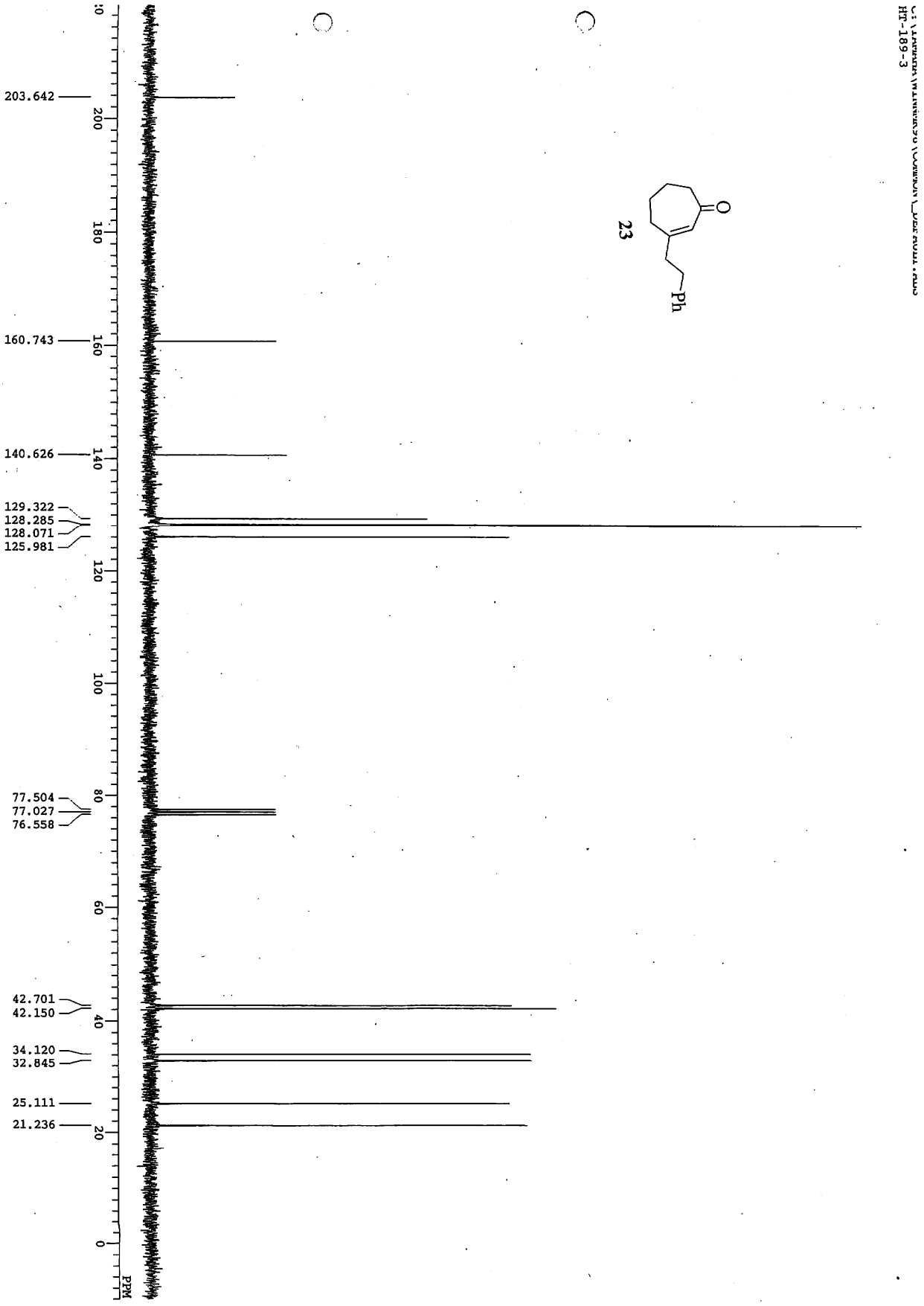
5.2668
1.0000
6.3677
4.2121
PPM
23
Ph
O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Ph
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160.743
140.626
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128.285
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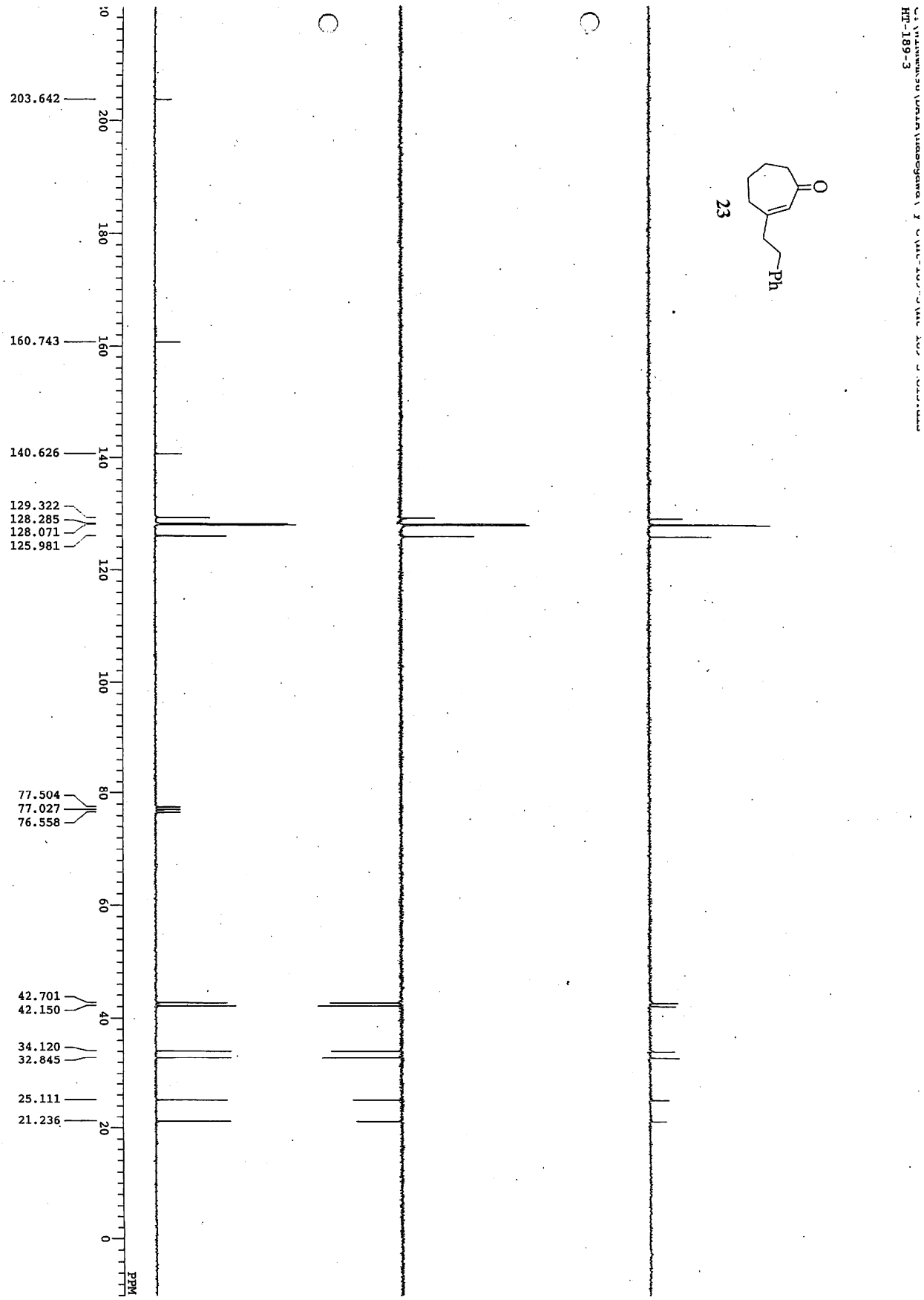
42.701
42.150
34.120
32.845
25.111
21.236
PPM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Ph
O
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128.285
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42.150
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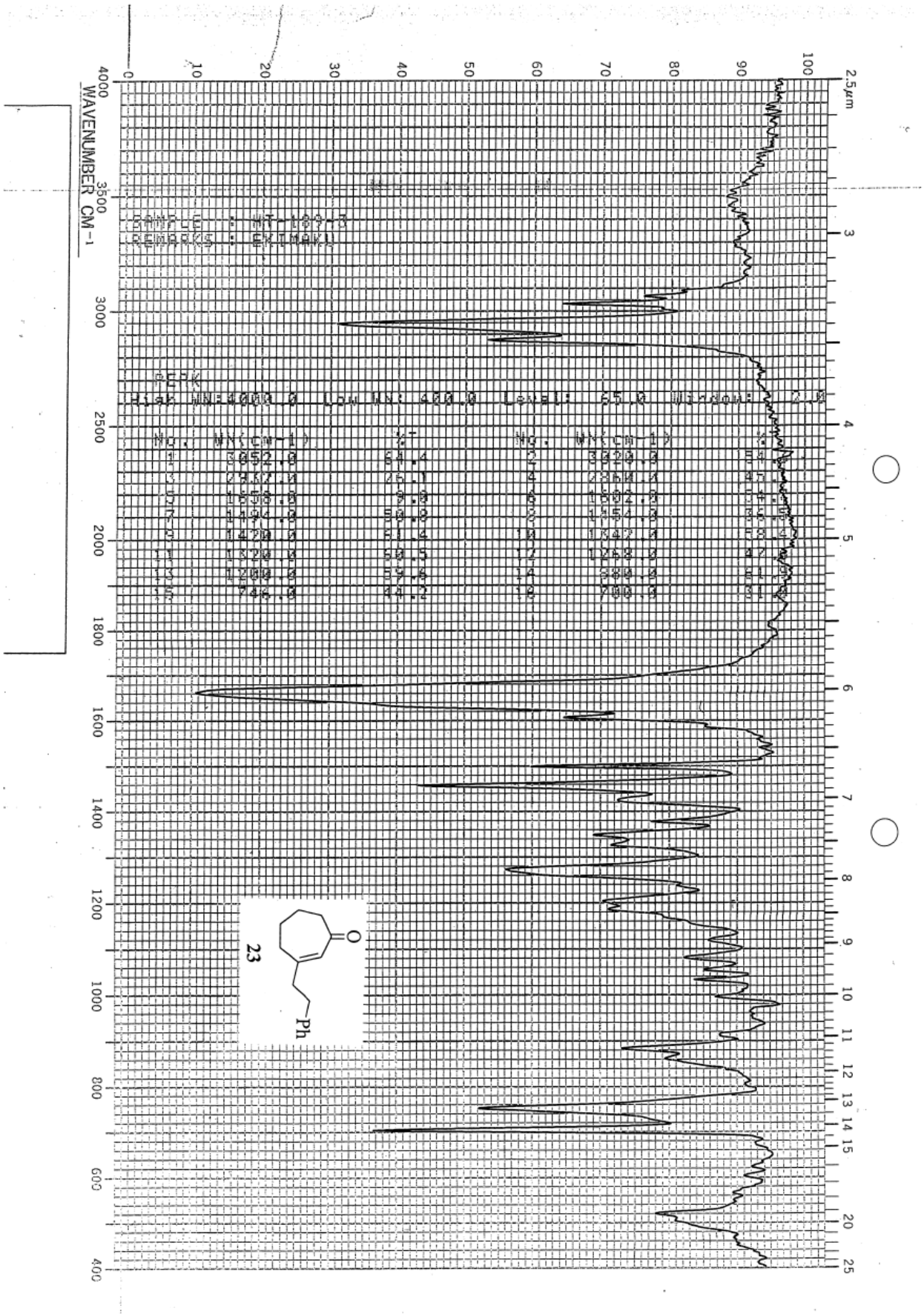
32.845
25.111
21.236
PPM


WAVENUMBER CM-1
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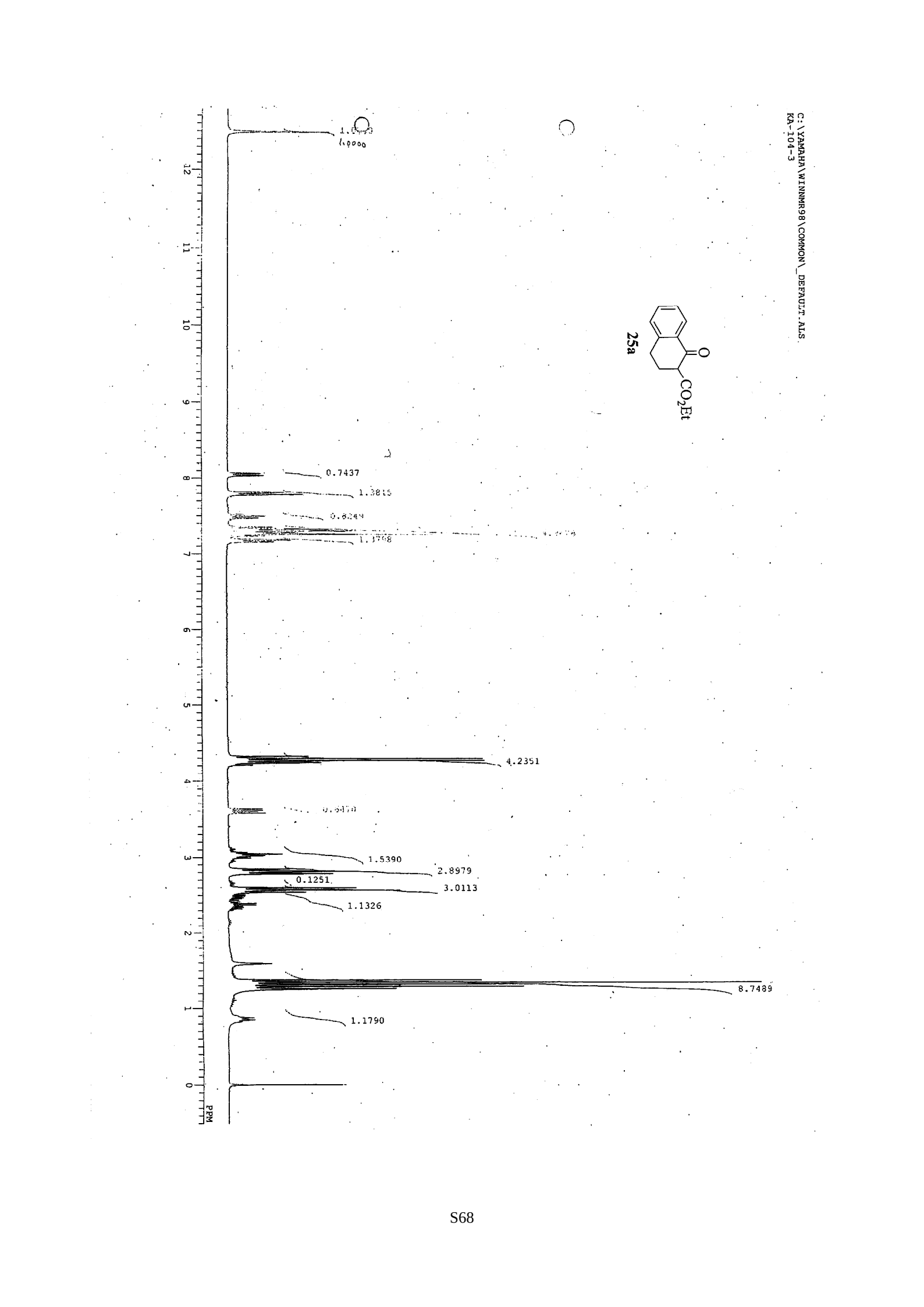
1600
1400
1200
1000
800
600
400
2.5μm
O
Ph
23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O
CO2Et
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0.8249
1.1708
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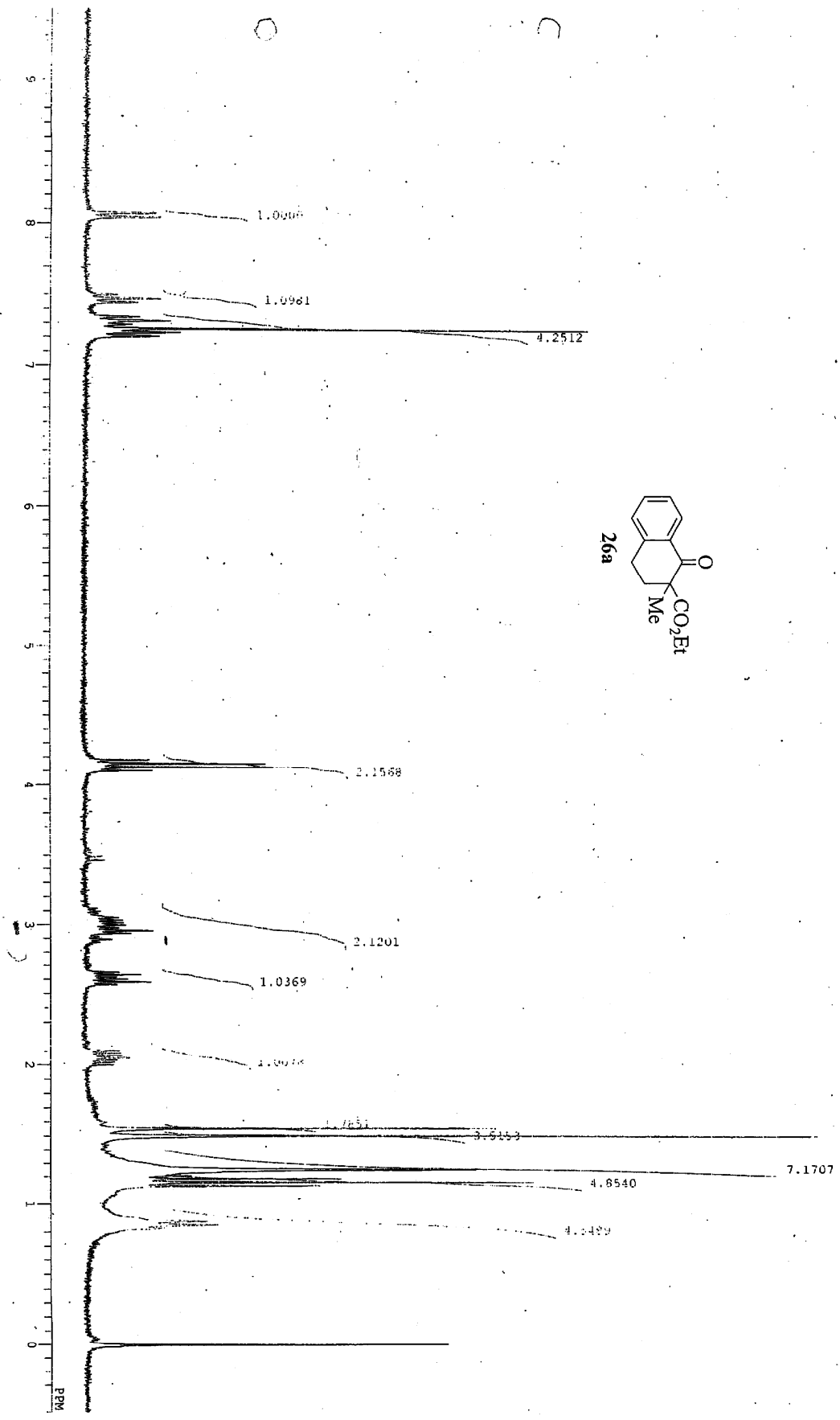
1.5390
2.8979
0.1251
3.0113
1.1326
8.7489
1.1790
PPM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HT-273-1
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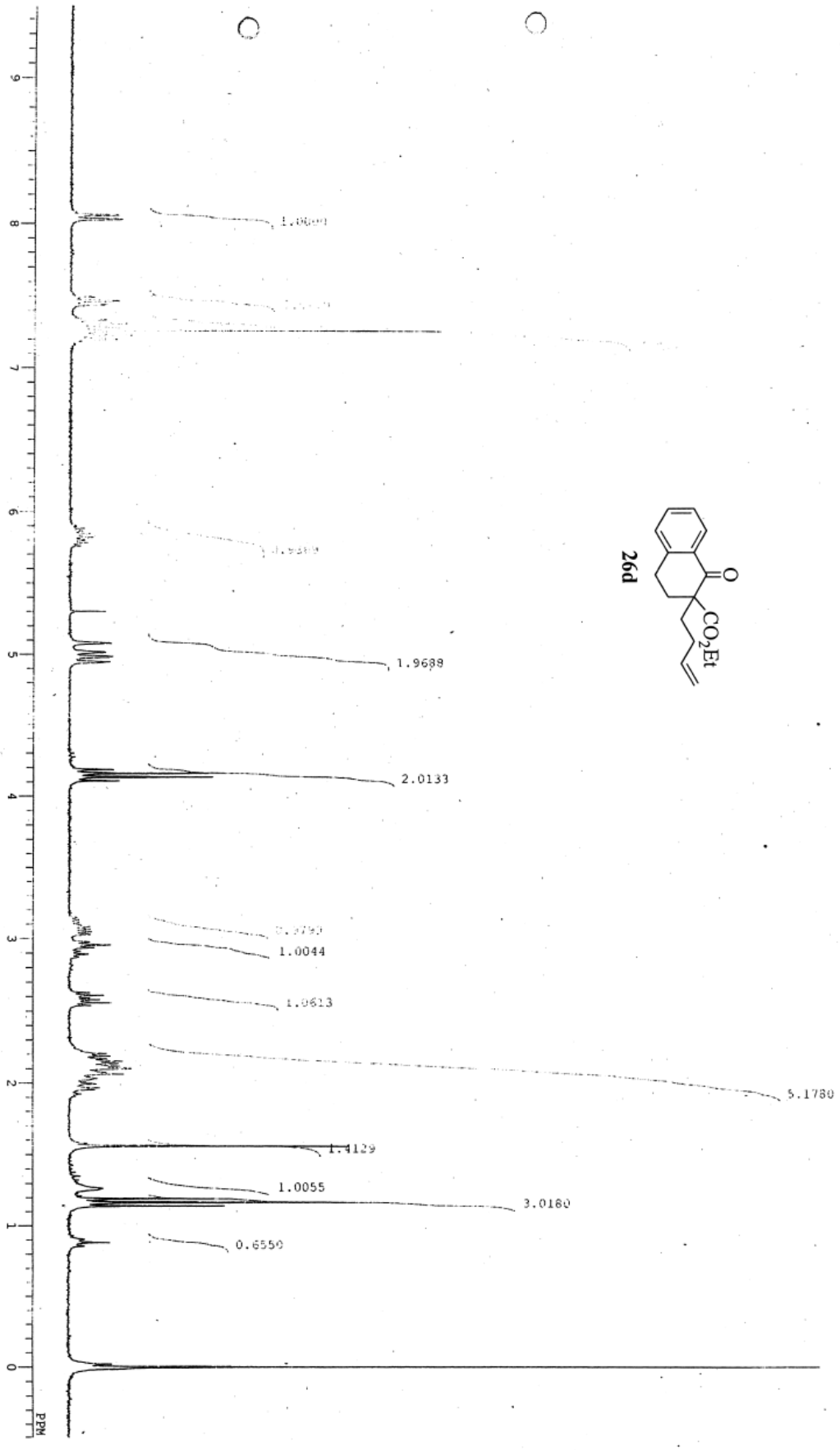
2.1201
1.0369
7.1707
4.8540
PPM
26a
O
CO2Et
Me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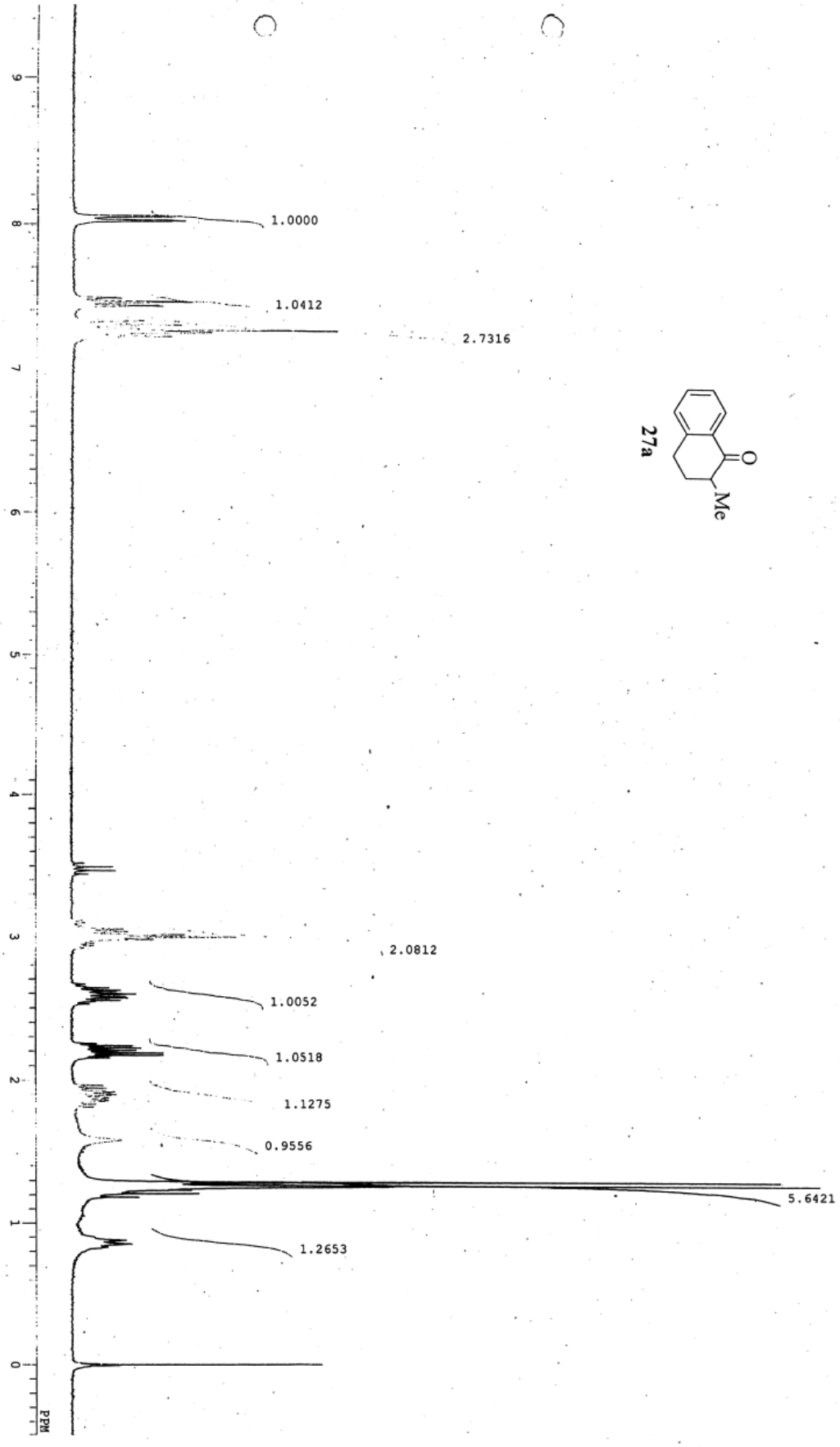
1.0613
5.1780
1.4129
1.0055
3.0180
0.6550
PPM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HT-274-2
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2.7316
2.0812
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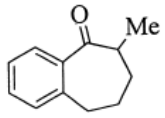
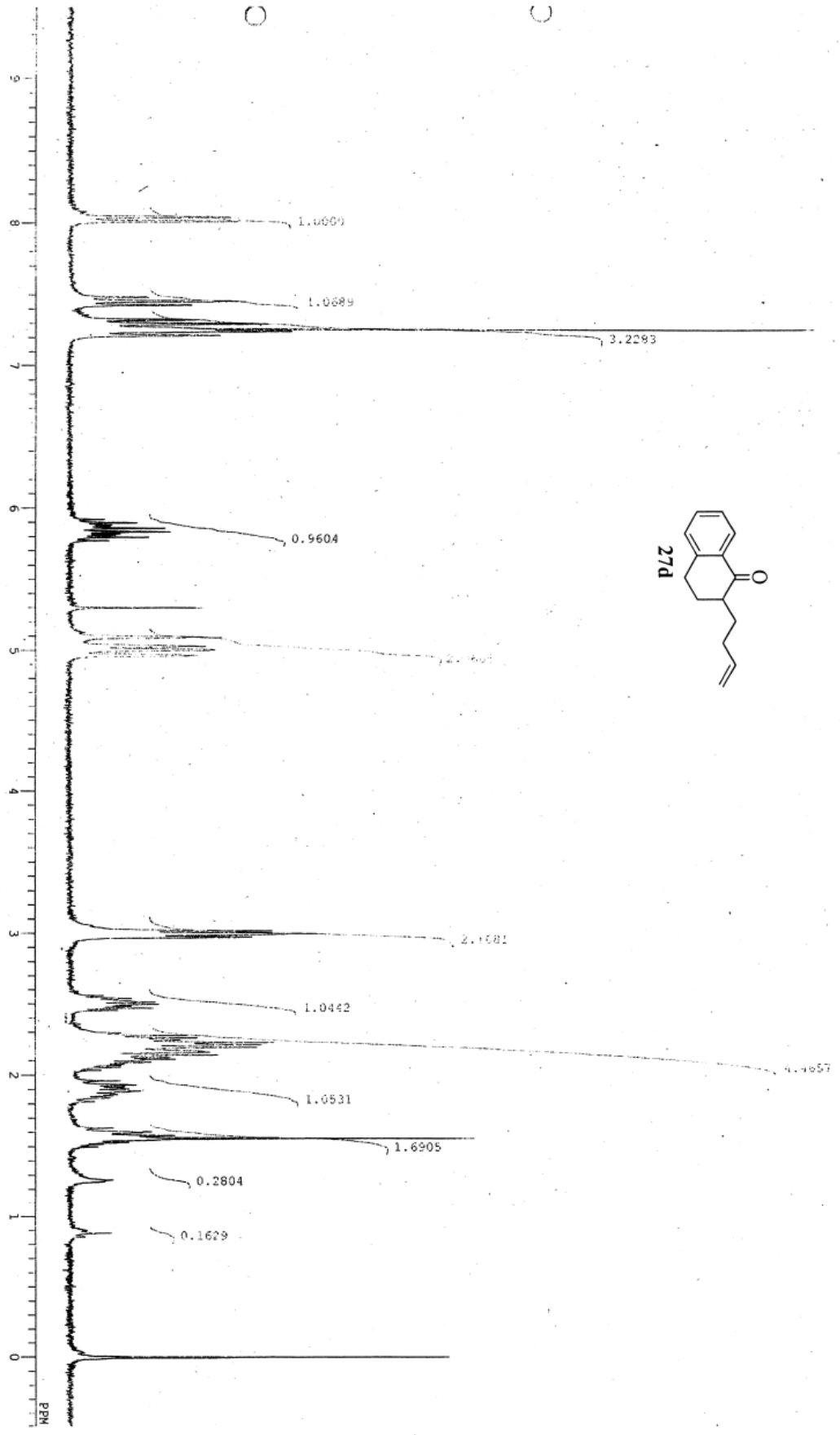
1.0518
1.1275
0.9556
5.6421
1.2653
PPM
27a
O
Me

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-240-1



27c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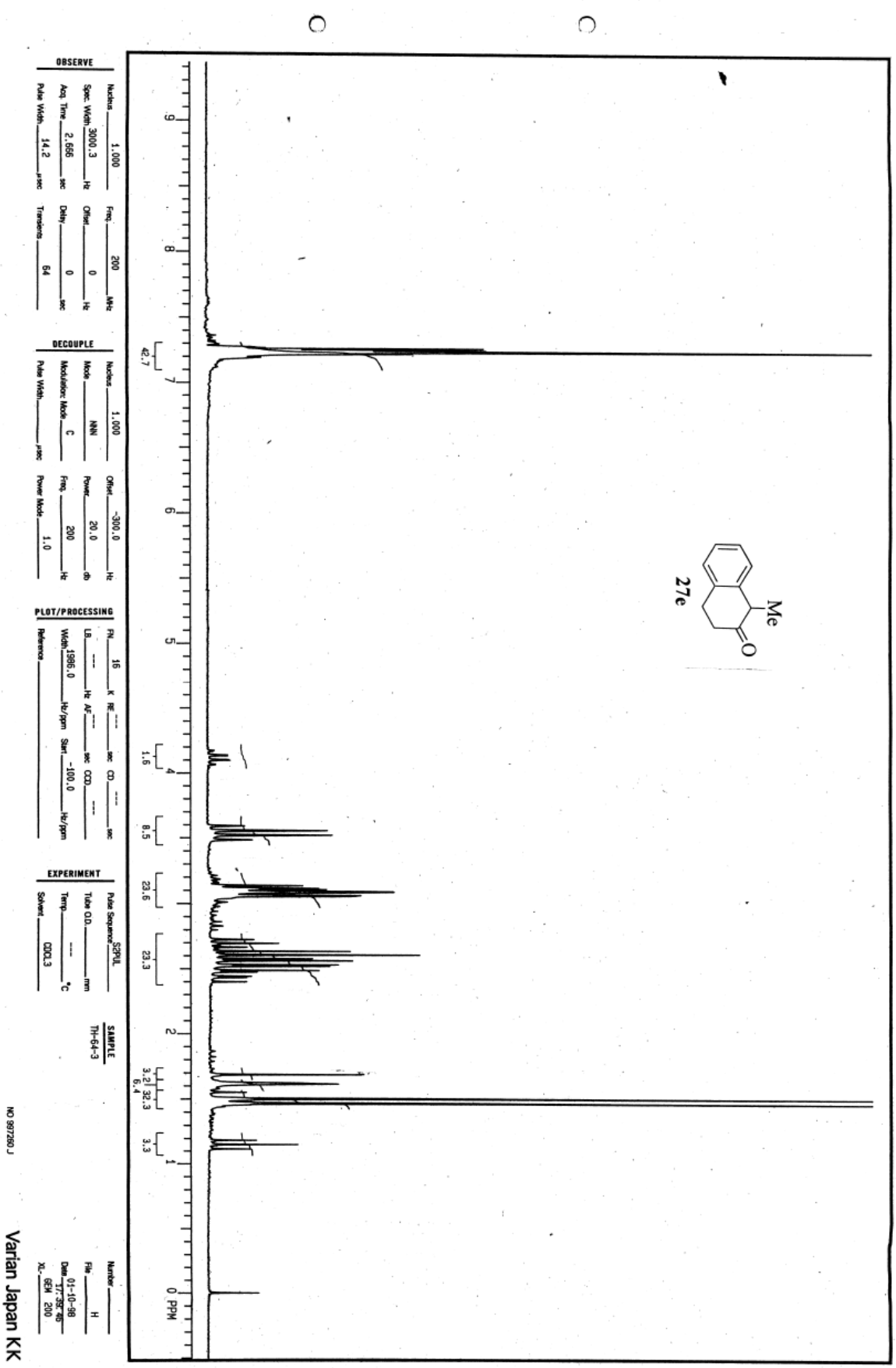
1.0442
4.4857
1.0531
1.6905
0.2804
0.1629
PPM
27d


Me
O
27e
OBSERVE
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Pulse Width 14.2 μsec Transients 64
DECOUPLE
Nucleus 1.000
Mode NNN Offset -300.0 Hz
Modulation Mode C Power 20.0 db
Pulse Width μsec Freq 200 Hz
Power Mode 1.0
PLOT/PROCESSING
FN 16 RE sec CD sec
LB Hz AF sec COD
Width 1986.0 Hz/ppm Start -100.0 Hz/ppm
Reference
EXPERIMENT
Pulse Sequence S2PUL
Tube O.D. mm
Temp °c
Solvent CDCL3
SAMPLE
TH-64-3
Number
File H
Date 01-10-98
17:35:40
XL- GEM 200
NO 997280.J
Varian Japan KK
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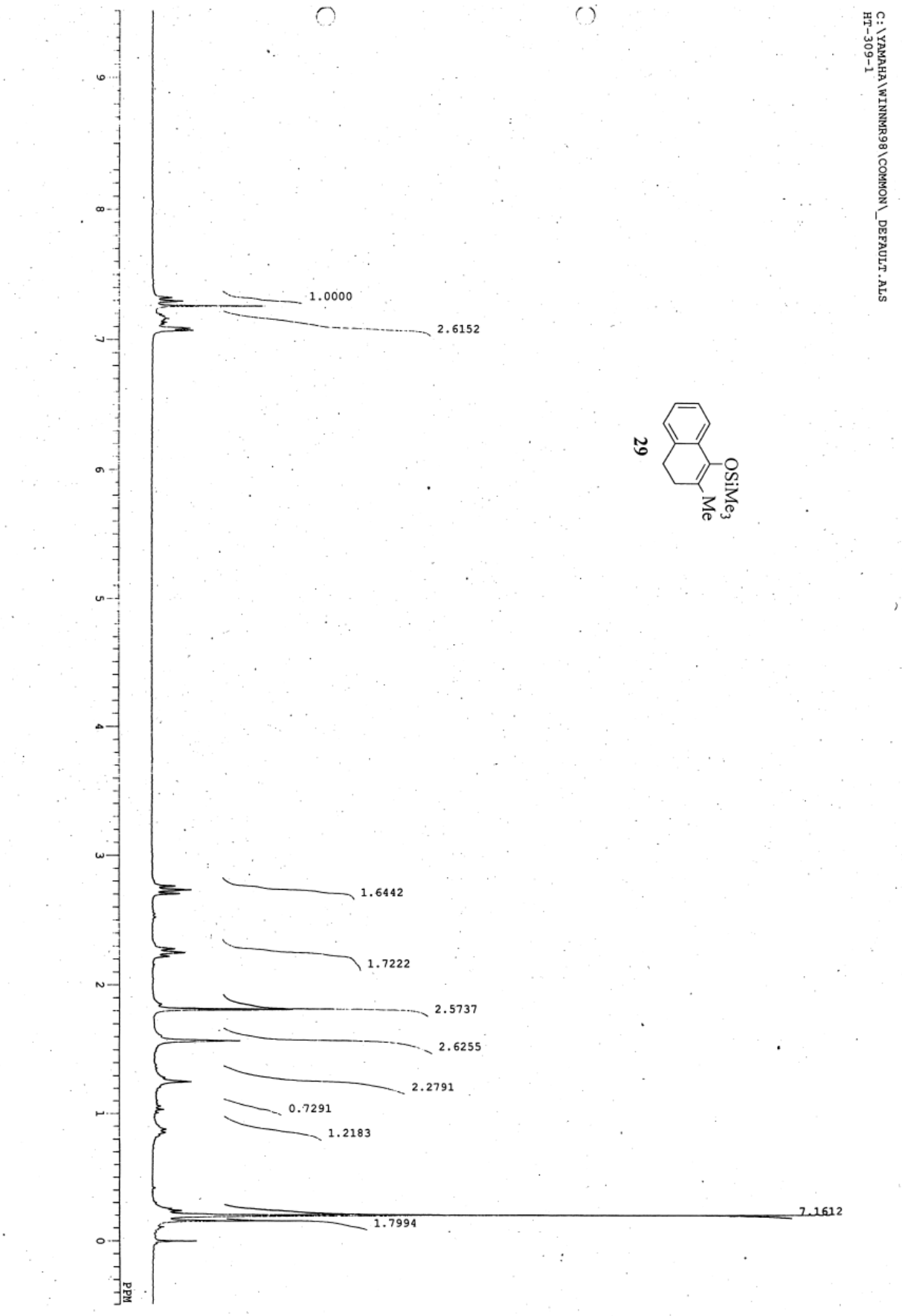
1.6
8.5
23.6
23.3
3.2
6.4
32.3
3.3
PPM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OSiMe3
Me
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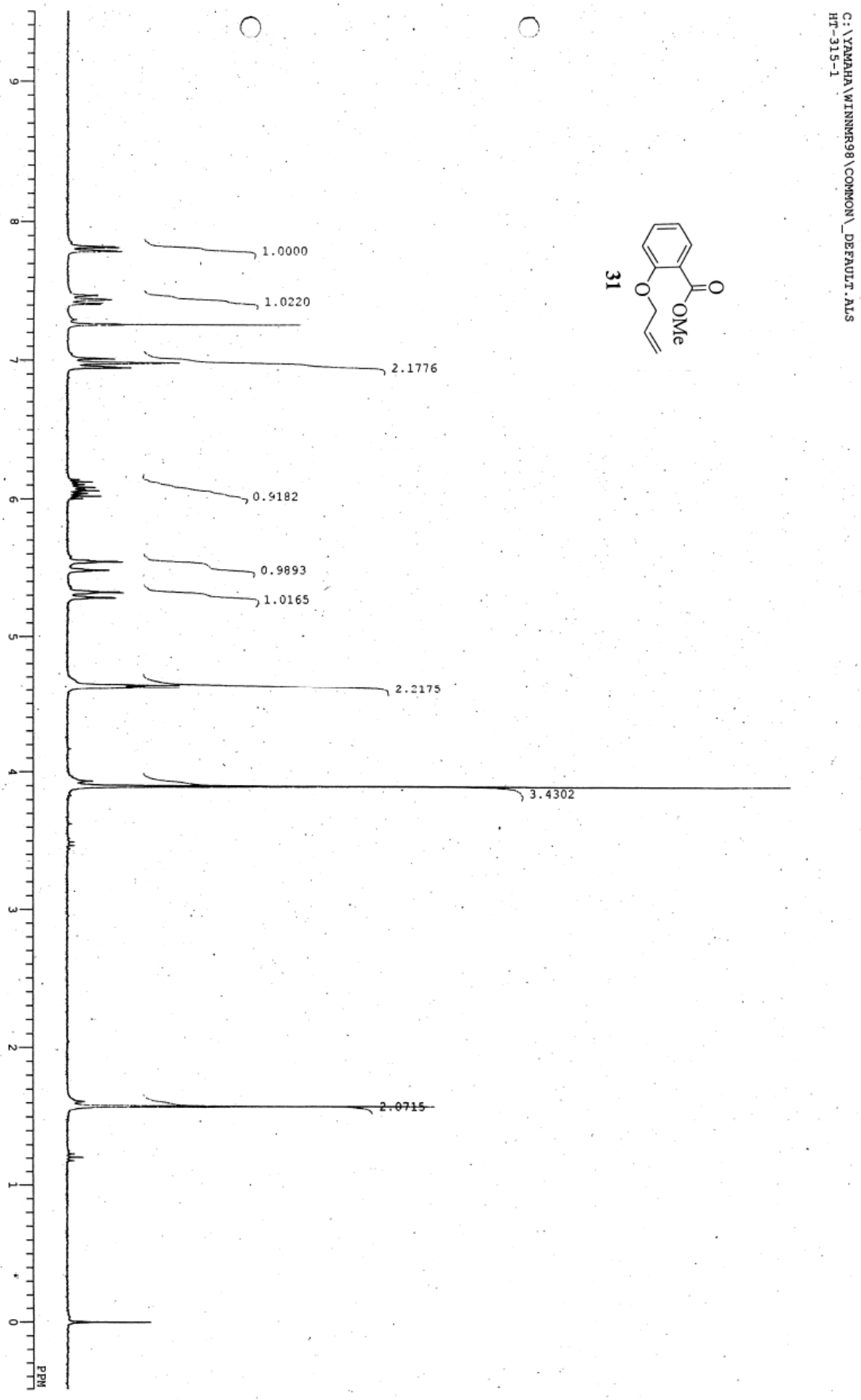
PPM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HT-315-1
31
OMe
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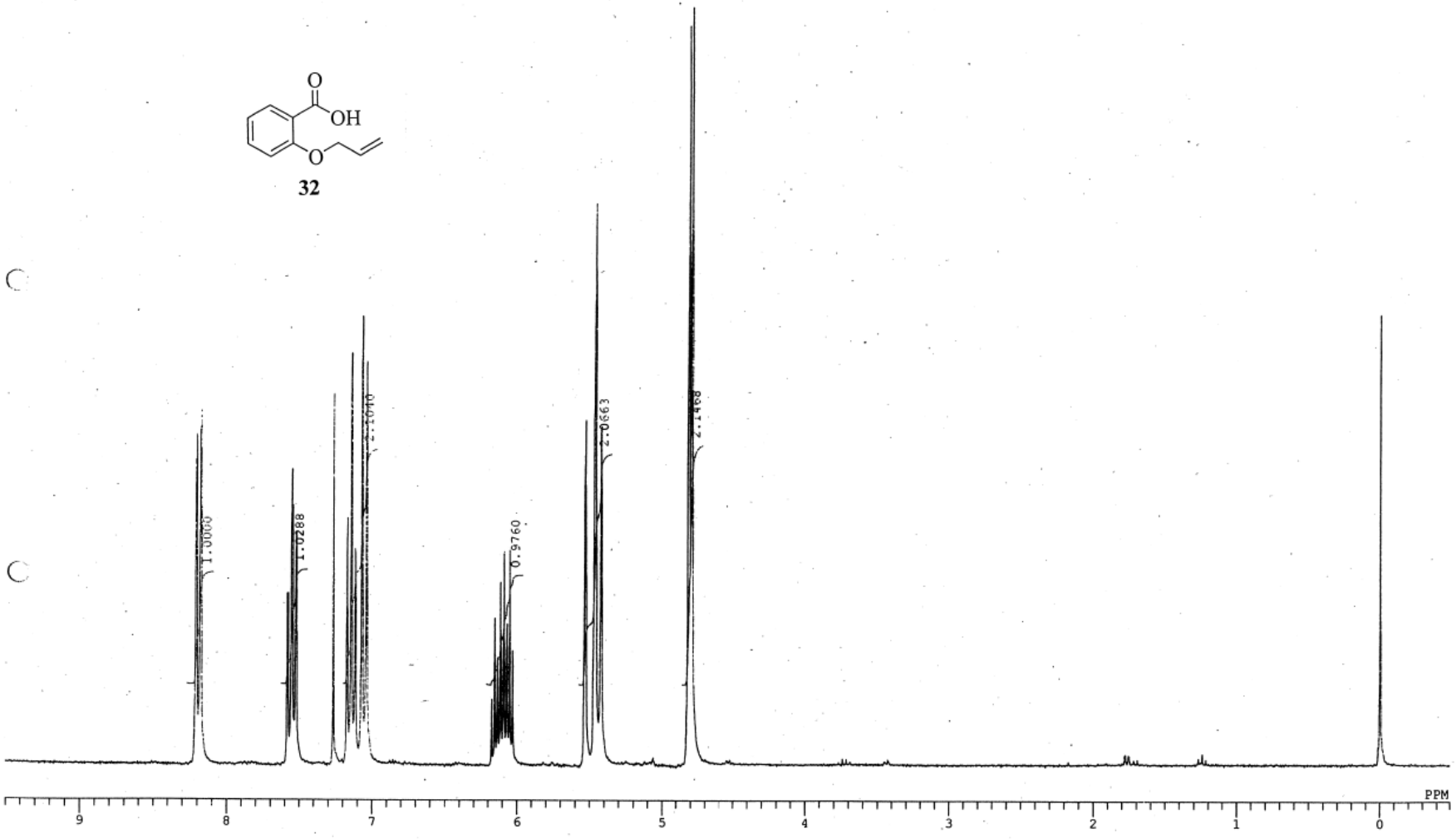
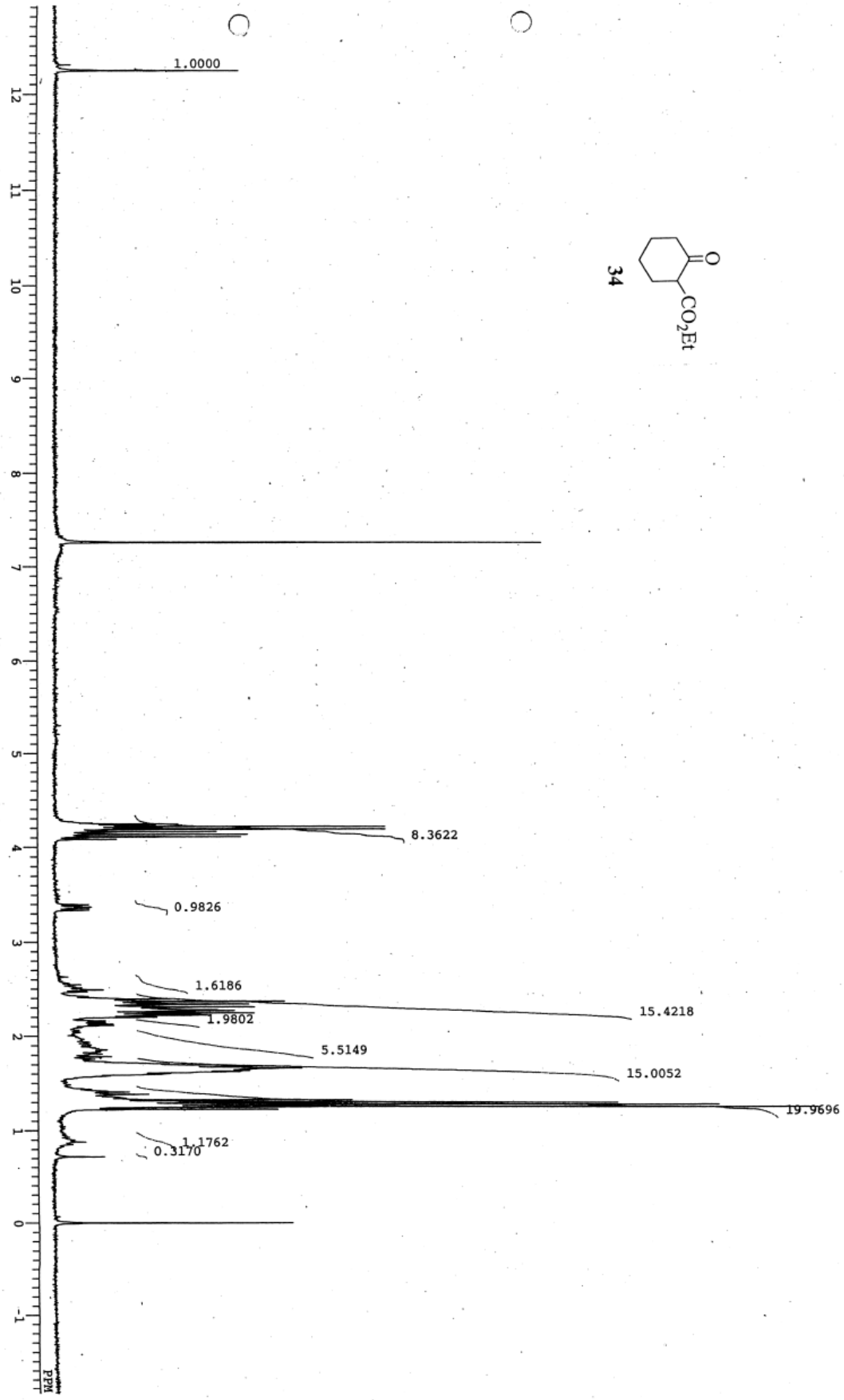
2.2175
3.4302
2.0715
PPM

C:\YAMAHA\WINNMR98\COMMON_DEFAULT.ALS
HT-329-2



32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CO2Et
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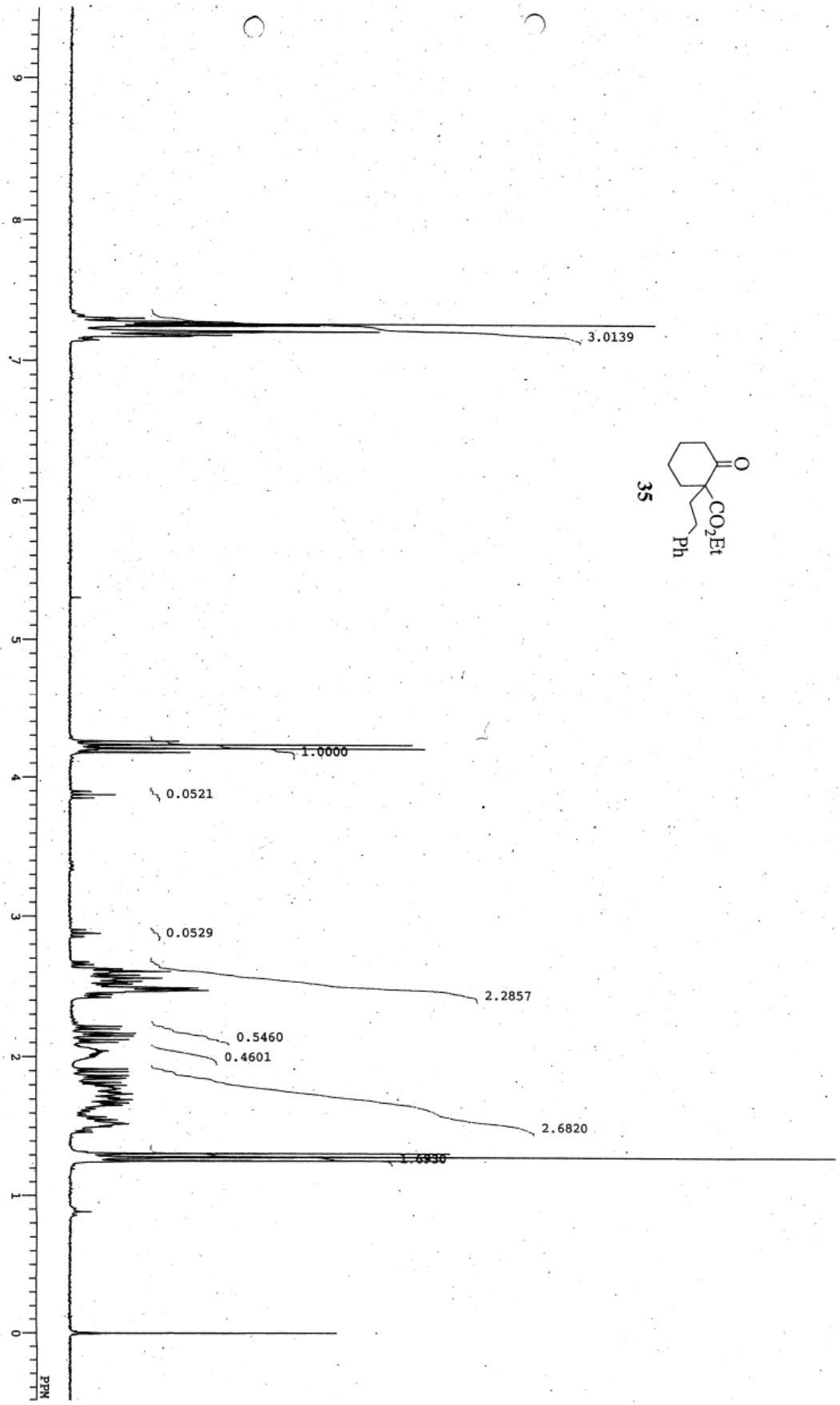
1.9802
15.4218
5.5149
15.0052
19.9696
1.1762
0.3170
PPM


HT-170-4
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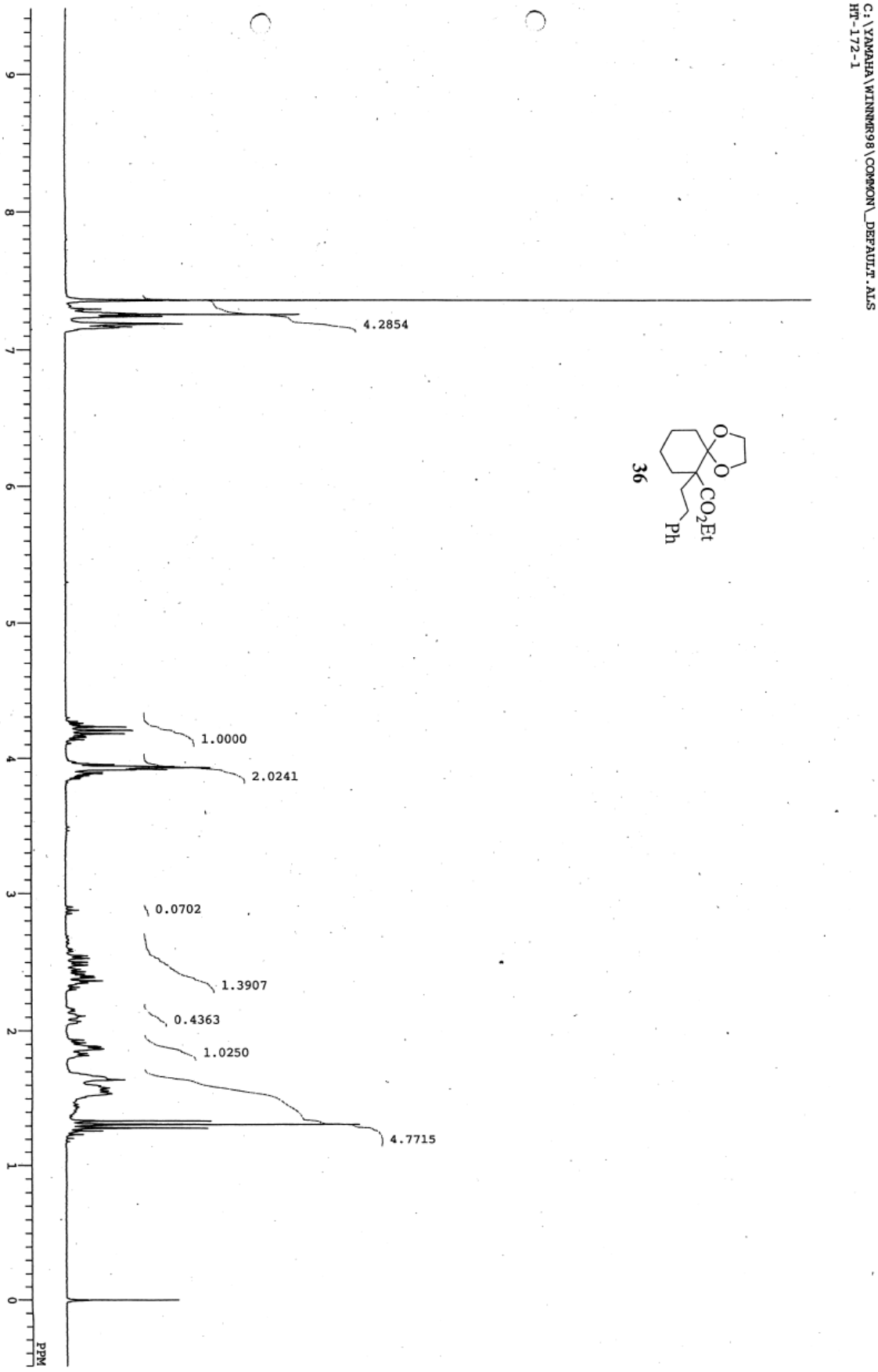
CO2Et
Ph
35
PPM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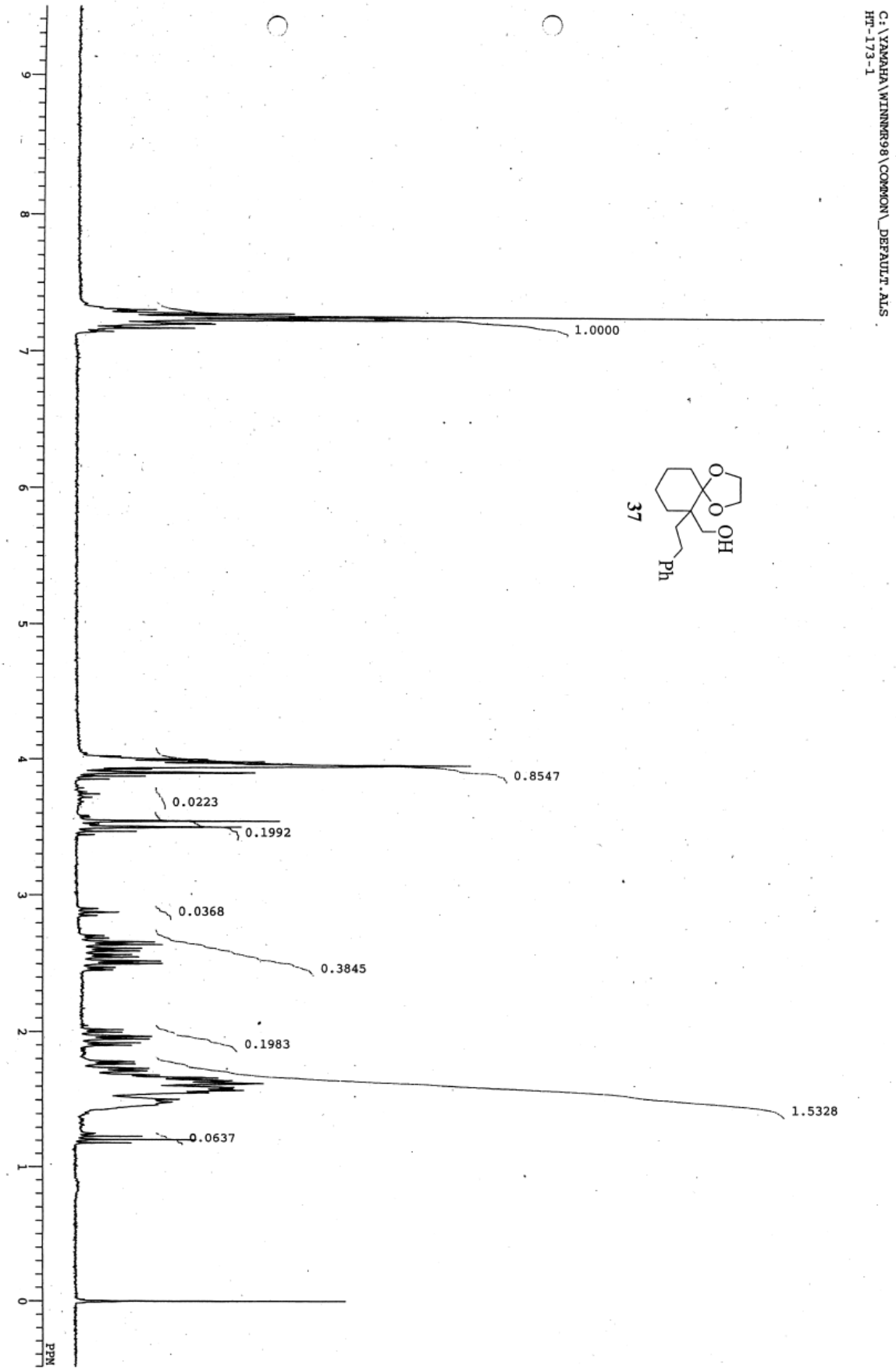
PPM
36
O
O
CO2Et
Ph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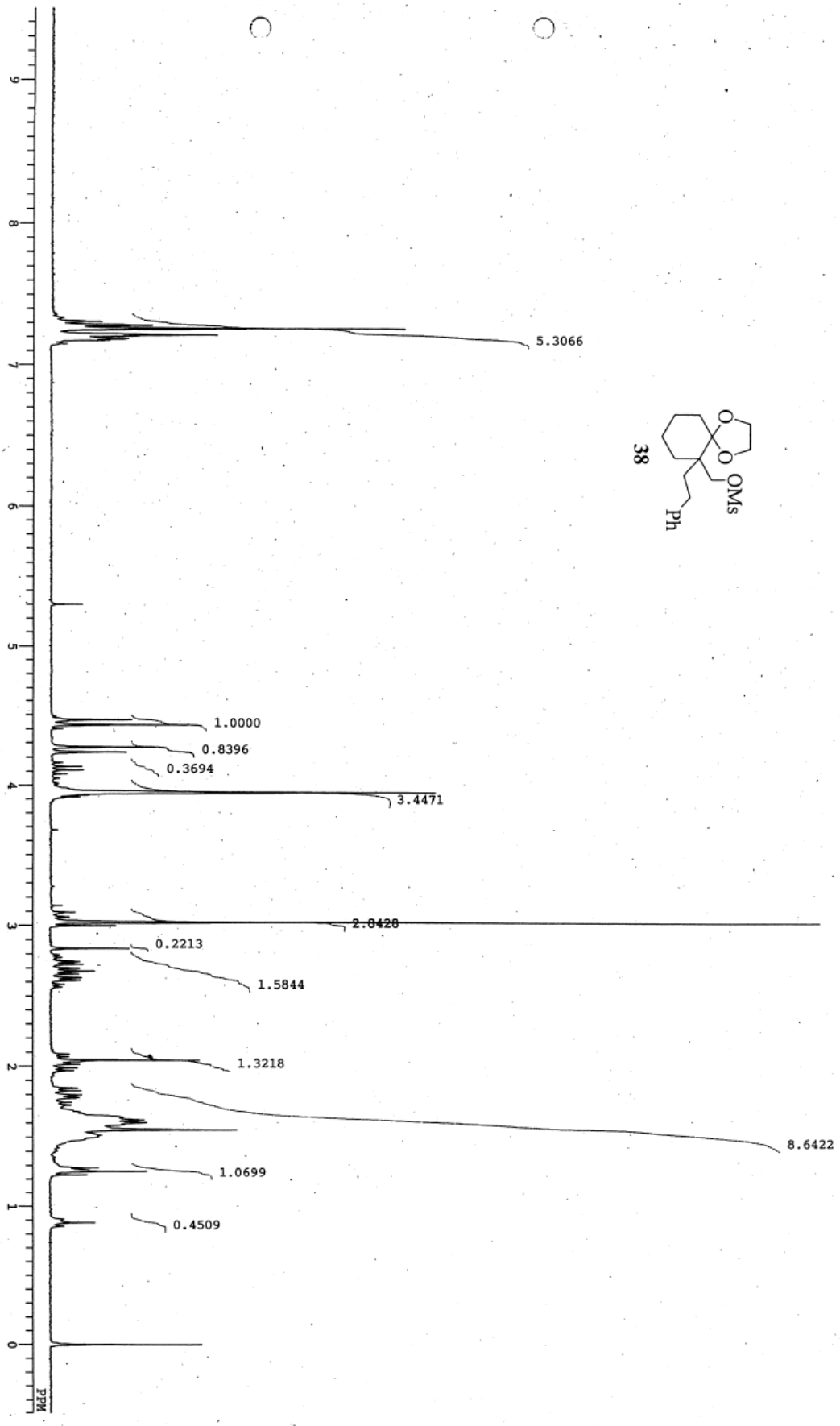
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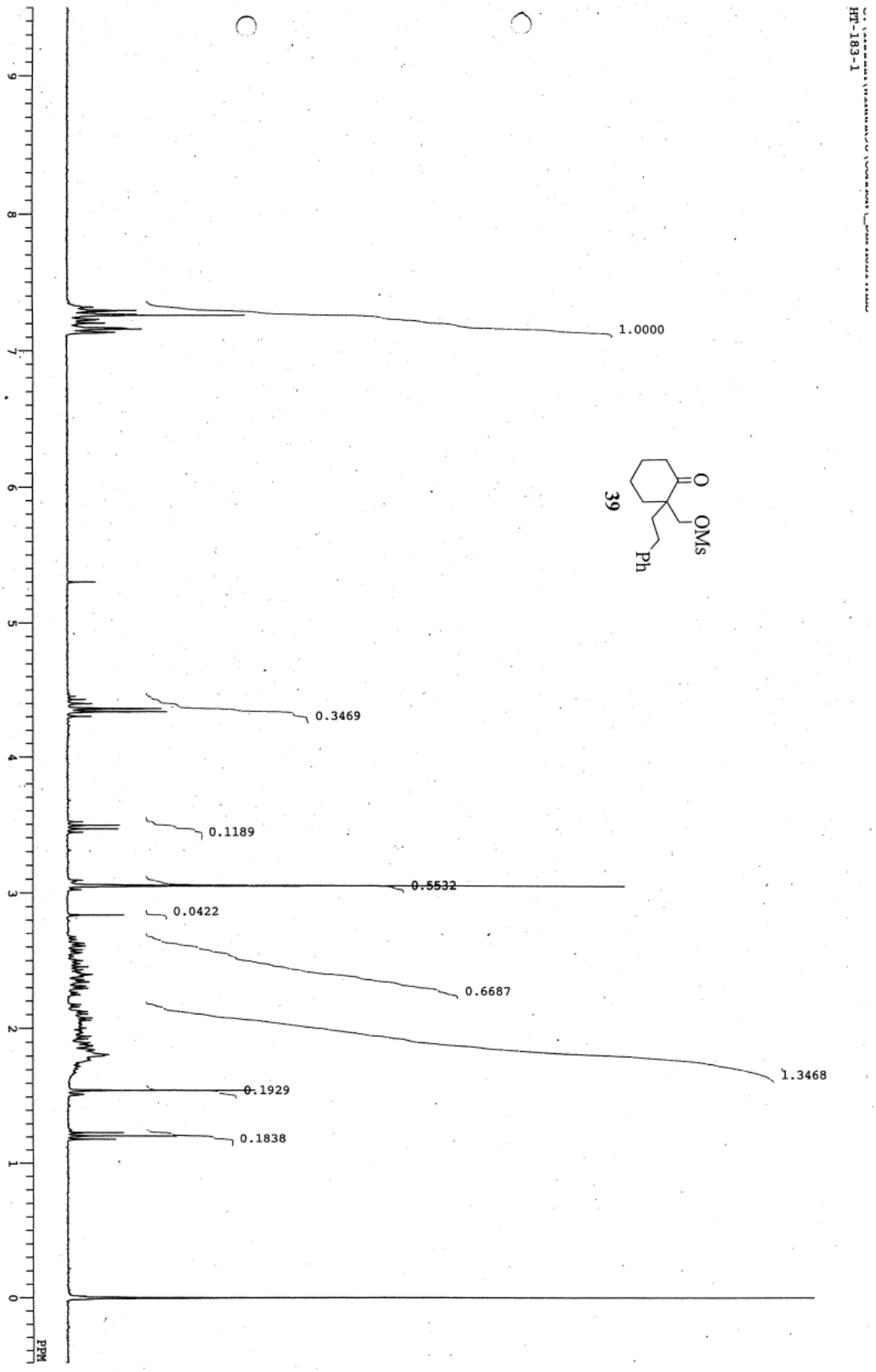
PPM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O
OMs
Ph
39
PPM