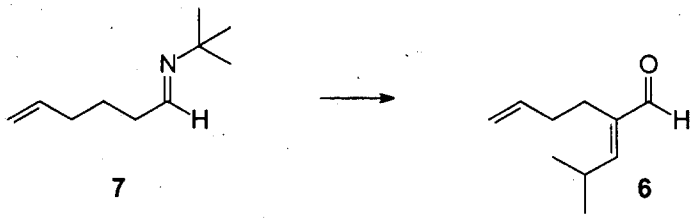
Experimental

General: ^{1}H NMR and ^{13}C NMR spectra were recorded on either a General Electric QE-300 spectrometer operating at either 300 MHz (^{1}H) or 75 MHz (^{13}C) or on a Varian Unity Inova 400 WB operating at either 400 MHz (^{1}H) or 101 MHz (^{13}C). Chemical shifts are reported in δ units and are referenced to the solvent, i.e., 7.26/77.0 for $CDCl_3$, 7.15/128.0 for benzene-d_6, 3.58/67.4 for THF-d_8 and 5.32/53.8 for methylene chloride-d_2. Multiplicities are indicated as: br (broadened), s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sept (septet) or m (multiplet). Coupling constants (J) are reported in Hertz (Hz). Infrared spectra were recorded on a Perkin-Elmer IR 1430 spectrometer. Electron impact mass spectra were performed on a VG-70SE mass spectrometer. Thin-layer chromatography (TLC) was performed on Sigma-Aldrich TLC plates, 250 μm, particle size 5 to 17 μm, pore size 60 Å. Flash column chromatography was performed on ICN silica gel, 32-63, 60 Å. Tetrahydrofuran was distilled from sodium-benzophenone ketyl. Triethylamine, diisopropylamine, morpholine, and benzylamine were distilled from calcium hydride and stored over potassium hydroxide. 1,4-Dioxane was distilled from calcium hydride and stored over activated 4Å molecular sieves. Methylene chloride and trimethylsilyl chloride were distilled from calcium hydride immediately before use. Propionic acid and isobutyraldehyde were distilled before use. Other reagents were used as received. All moisture sensitive reactions were performed under a static nitrogen or argon atmosphere in oven-dried or flame-dried glassware.

8 **7**

To 5-hexenal **8** (1.371 g, 13.97 mmol) at 0°C was added *tert*-butylamine (1.60 mL, 1.11 g, 15.2 mmol). The reaction mixture was warmed to room temperature, stirred for 21 h, and potassium hydroxide pellets were added. Purification by distillation on a Kugelrohr (oven temperature 125°C) under reduced pressure (water aspirator, ca. 25

mmHg) gave the imine **7** (2.011 g, 94%) as a colorless liquid: IR (neat) 2980, 2880, 1675, 1650, 1460, 1370, 1220, 995, 915 cm^{-1}; ^1H NMR (300 MHz, C_6D_6) δ 7.40 (t, J = 4.4 Hz, 1H), 5.72 (ddt, J = 16.9, 10.4, 6.6 Hz, 1H), 5.02-4.93 (m, 2H), 2.11 (td, J = 7.4, 4.4 Hz, 2H), 1.96 (q br, J = 7.2 Hz, 2H), 1.52 (quint, J = 7.4 Hz, 2H), 1.14 (s, 9H); ^{13}C NMR (75 MHz, C_6D_6) δ 156.7, 138.7, 114.9, 56.5, 35.9, 33.7, 29.9, 25.6.



7 **6**

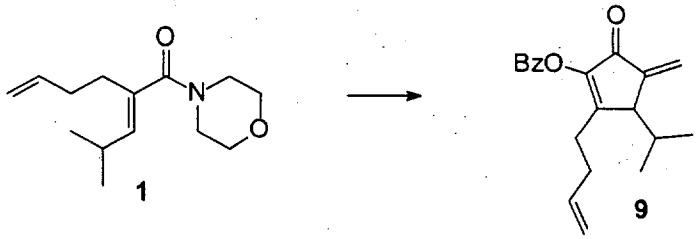
To a solution of diisopropylamine (2.90 mL, 2.09 g, 20.7 mmol) in THF (55 mL) at –78°C was added *n*-BuLi (7.50 mL, 20.9 mmol, 2.78 M in hexanes). After 30 min, a solution of the imine **7** (2.899 g, 18.91 mmol) in THF (20 mL) was added via cannula. The solution was warmed from –78°C to –20°C over 3 h, cooled to –78°C, and trimethylsilyl chloride (2.70 mL, 2.31 g, 21.5 mmol) was added. The reaction mixture was warmed from –78°C to 10°C over 14 h, concentrated, and diluted with petroleum ether, saturated $NaHCO_3$, and water. The aqueous phase was extracted with petroleum ether (3 x) and the combined organic extracts were washed with brine (1 x), dried over K_2CO_3, and concentrated to give the crude α-TMS imine. The crude α-TMS imine was dissolved in THF (20 mL), cooled to –78°C, and added via cannula to a solution of LDA generated from diisopropylamine (2.90 mL, 2.09 g, 20.7 mmol) and *n*-BuLi (7.50 mL, 20.9 mmol, 2.78 M in hexanes) in THF (60 mL) at –78°C. The solution was warmed from –78°C to 0°C over 3 h, cooled to –78°C, and isobutyraldehyde (1.90 mL, 1.51 g, 20.9 mmol) was added. The reaction mixture was warmed from –78°C to 10°C over 5 h, concentrated, and diluted with ether and water. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with water (1 x), brine (1 x), dried over $MgSO_4$, and concentrated to give the crude α,β-unsaturated imine. The crude α,β-unsaturated imine was dissolved in THF (15 mL) and water (15 mL) and oxalic acid dihydrate (5.388 g, 42.74 mmol) were added. The reaction mixture was stirred at room

temperature for 12 h and diluted with ether and water. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with brine (1 x) and dried over $MgSO_4$. Purification by flash cloumn chromatography on silica gel (1.25% to 2.5% to 5% EtOAc in hexanes) gave the aldehyde **6** (2.058 g, 71%) as a colorless oil and a single isomer by ^1H NMR: R_f = 0.40 (5% EtOAc in hexanes); IR (neat) 2980, 2945, 2885, 2830, 1690, 1645, 915 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 9.33 (s, 1H), 6.25 (d, J = 10.3 Hz, 1H), 5.75 (ddt, J = 16.9, 10.3, 6.6 Hz, 1H), 5.01-4.92 (m, 2H), 2.81 (d sept, J = 10.0, 6.6 Hz, 1H), 2.32 (t, J = 7.7 Hz, 2H), 2.10 (q br, J = 7.4 Hz, 2H), 1.07 (d, J = 6.6 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 195.2, 161.6, 140.4, 137.7, 115.0, 33.1, 28.2, 23.8, 22.1.

6 **1**

To a solution of the aldehyde **6** (1.061 g, 6.969 mmol) in 2-methyl-2-butene (30.0 mL, 19.9 g, 283 mmol) and *tert*-butyl alcohol (84 mL) at 0°C was added a solution of $NaClO_2$ (4.905 g, 54.23 mmol) and KH_2PO_4 (7.418 g, 54.51 mmol) in water (48 mL) at 0°C. The reaction mixture was warmed to room temperature, stirred for 7 h, and diluted with EtOAc and water. The aqueous phase was extracted with EtOAc (3 x) and the combined organic extracts were washed with brine (1 x), dried over $MgSO_4$, and concentrated to give the crude acid. To a solution of the crude acid and carbon tetrabromide (2.593 g, 7.818 mmol) in CH_2Cl_2 (14 mL) was added at 0°C, triethylamine (1.10 mL, 799 mg, 7.89 mmol), morpholine (690 μL, 689 mg, 7.19 mmol), and a solution of triphenylphosphine (2.189 g, 8.346 mmol) in CH_2Cl_2 (4 mL). After 30 min, the reaction mixture was partially concentrated under a stream of argon to a volume of approximately 15 mL and loaded directly onto a silica gel flash column. Purification by chromatography (10% to 20% to 30% to 50% EtOAc in hexanes) gave the amide **1** (1.449 g, 88%) as a colorless oil: R_f = 0.26 (30% EtOAc in hexanes); IR (neat) 2975, 2940, 2870, 1640, 1430, 1280, 1260, 1120, 1030, 920 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$)

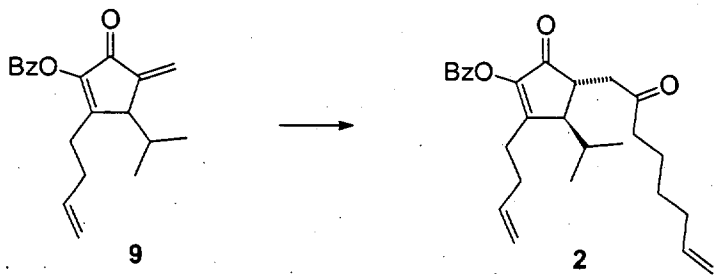
δ 5.73 (ddt, J = 16.9, 10.3, 6.6 Hz, 1H), 5.23 (d, J = 9.5 Hz, 1H), 5.00-4.89 (m, 2H), 3.62-3.40 (m, 8H), 2.56 (d sept, J = 9.8, 6.4 Hz, 1H), 2.36 (t, J = 7.6 Hz, 2H), 2.08 (q br, J = 7.3 Hz, 2H), 0.93 (d, J = 6.8 Hz, 6H); ^{13}C NMR (75 MHz, $CDCl_3$): 171.5, 139.0, 137.7, 131.6, 114.9, 66.7, 44.9 (br), 32.4, 27.9, 26.9, 22.5; mass spectrum m/z 196 (39), 194 (31), 151 (33), 109 (44), 81 (100), 69 (44), 67 (62); exact mass calcd for $C_{14}H_{23}NO_2$ 237.1729, found 237.1733.



1 9

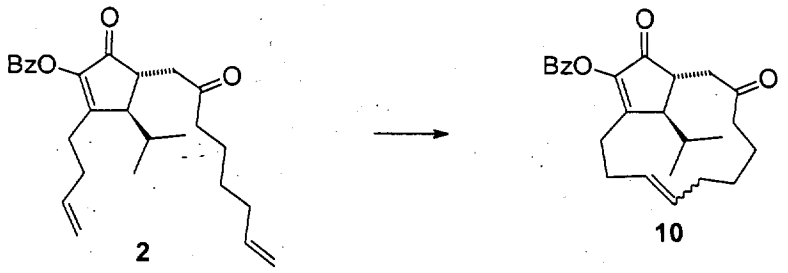
To a solution of methyloxymethyl allenyl ether (170 μL, 157 mg, 1.57 mmol) in THF (4 mL) at –78°C was added *n*-BuLi (630 μL, 1.52 mmol, 2.41 M in hexanes). After 30 min, a solution of the amide **1** (302 mg, 1.27 mmol) in THF (9 mL) at –78°C was added. After 30 min, the reaction mixture was quenched with AcOH (610 μL, 1.52 mmol, 2.49 M in THF), warmed to room temperature, and diluted with ether and water. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with brine (1 x), dried over $MgSO_4$, and concentrated to give the crude cyclopentenone. To a solution of the crude cyclopentenone in CH_2Cl_2 (10 mL) at 0°C was added triethylamine (210 μL, 152 mg, 1.51 mmol) and benzoyl chloride (170 μL, 206 mg, 1.46 mmol). After 30 min, the reaction mixture was diluted with ether and water. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with brine (1 x) and dried over $MgSO_4$. Purification by flash column chromatography on silica gel (2.5% to 5% EtOAc in hexanes) gave the protected cyclopentenone **9** (192 mg, 49%) as a colorless oil: R_f = 0.20 (10% EtOAc in hexanes); IR (neat) 2980, 1750, 1715, 1670, 1635, 1455, 1260, 1180, 1090, 1070, 1025, 710 cm^{-1}; ^{1}H NMR (300 MHz, C_6D_6) δ 8.14 (dm, J = 7.8 Hz, 2H), 7.07 (tt, J = 7.4, 1.3 Hz, 1H), 6.97 (tm, J = 7.6 Hz, 2H), 6.18 (s, 1H), 5.58 (m, 1H), 5.01 (s, 1H), 4.95-4.88 (m, 2H), 3.00 (s br, 1H), 2.45 (m, 1H), 2.15-1.95 (m, 3H), 1.78 (sept d, J = 6.9, 2.9 Hz, 1H), 0.84

(d, J = 6.8 Hz, 3H), 0.67 (d, J = 7.1 Hz, 3H); ^{13}C NMR (75 MHz, C_6D_6) δ 186.8, 163.4, 158.8, 149.1, 142.0, 137.1, 133.5, 130.5, 129.3, 128.7, 117.1, 115.7, 47.8, 30.8, 29.3, 26.5, 21.6, 16.3; mass spectrum m/z 122 (7), 106 (8), 105 (100), 77 (30), 76 (14); exact mass calcd for $C_{20}H_{22}O_3$ 310.1569, found 310.1593.



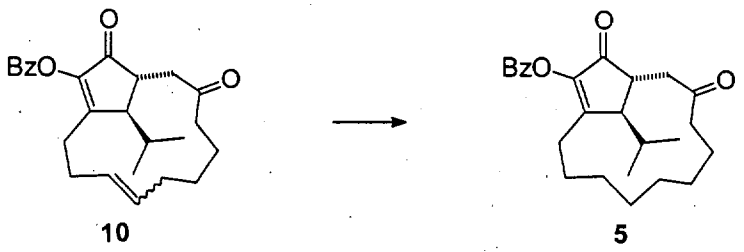
9 **2**

To a mixture of the cyclopentenone **9** (150 mg, 0.483 mmol) and 6-heptenal (120 mg, 1.07 mmol) was added 1.0 mL of a solution of 3-benzyl-5-(hydroxyethyl)-4-methylthiazolium chloride (42 mg, 0.16 mmol) and triethylamine (100 μL, 72.6 mg, 0.717 mmol) in 1,4-dioxane (2.9 mL). The reaction mixture was heated to 70°C in a sealed tube. After 18 h, the reaction mixture was diluted with Et_2O and water. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with brine (1 x) and dried over $MgSO_4$. Purification by flash column chromatography on silica gel (2.5% to 5% to 10% to 20% EtOAc in hexanes) gave the *trans*-diene **2** (123 mg, 60%) as a colorless oil: R_f = 0.16 (10% EtOAc in hexanes); IR (neat) 2975, 2945, 1750, 1725, 1665, 1265, 1100, 1070, 715 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.14 (dm, J = 7.1 Hz, 2H), 7.62 (tt, J = 7.4, 1.2 Hz, 1H), 7.48 (t, J = 7.6 Hz, 2H), 5.87-5.72 (m, 2H), 5.09-4.92 (m, 4H), 2.80-2.73 (m, 2H), 2.69-2.57 (m, 3H), 2.43 (t, J = 7.3 Hz, 2H), 2.38-2.18 (m, 4H), 2.05 (q br, J = 7.2 Hz, 2H), 1.59 (quint br, J = 7.6 Hz, 2H), 1.43-1.33 (m, 2H), 1.06 (d, J = 6.8 Hz, 3H), 0.85 (d, J = 6.8 Hz, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 208.0, 200.8, 164.2, 163.3, 145.6, 138.4, 136.8, 133.6, 130.3, 128.6, 128.5, 115.8, 114.6, 49.7, 44.3, 43.0, 40.7, 33.5, 30.6, 28.44, 28.41, 26.7, 23.2, 21.1, 16.3; mass spectrum m/z 190 (20), 106 (15), 105 (100), 77 (45); exact mass calcd for $C_{27}H_{34}O_4$ 422.2457, found 422.2467.



2 10

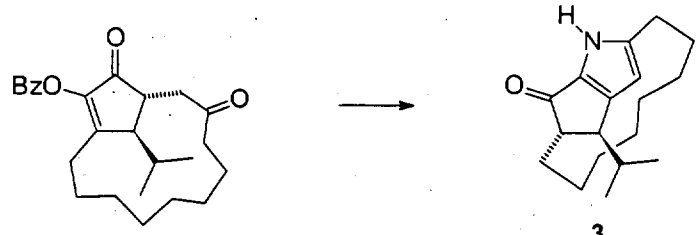
To a solution of Grubbs' catalyst (62 mg, 0.075 mmol) in CH_2Cl_2 (460 mL) was added a solution of the *trans*-diene **2** (103 mg, 0.244 mmol) in CH_2Cl_2 (20 mL). The solution was degassed for 15 min, heated to 40°C for 30 h, partially concentrated, and filtered through silica gel (eluted with 30% EtOAc in hexanes). Purification by flash column chromatography on silica gel (5% to 10% to 20% to 30% EtOAc in hexanes) gave the *E*, *Z* mixture **10** (87 mg, 90%) as a low melting solid.



10 5

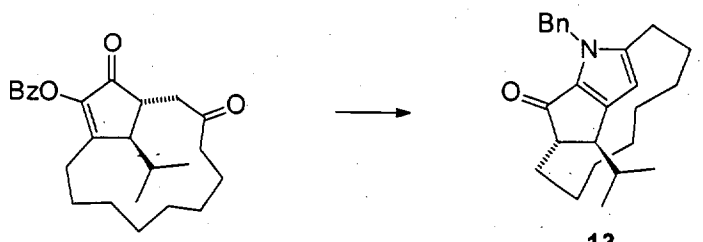
To the *E*, *Z* mixture **10** (84 mg, 0.21 mmol) was added 10% palladium on carbon (35 mg) and THF (10 mL). The nitrogen atmosphere was replaced by hydrogen from a double ballon, stirred at room temperature for 4 h, filtered through cotton, and concentrated. Purification by flash column chromatography on silica gel (5% to 10% to 20% EtOAc in hexanes) gave the macrocycle **5** (78 mg, 92%) as a white solid: mp 94-96°C; R_f = 0.17 (10% EtOAc in hexanes); IR (neat) 2940, 2880, 1745, 1725, 1660, 1455, 1260, 1215, 1100, 1065, 1025, 710 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 8.15 (dm, J = 7.4 Hz, 2H), 7.62 (tm, J = 7.4 Hz, 1H), 7.48 (tm, J = 7.8 Hz, 2H), 3.08 (dd, J = 14.9, 6.8 Hz, 1H), 2.70-2.61 (m, 3H), 2.53 (ddd, J = 14.5, 10.9, 3.4 Hz, 1H), 2.44 (ddd, J = 15.5, 7.8, 3.8 Hz, 1H), 2.37 (ddd, J = 15.5, 8.7, 3.8 Hz, 1H), 2.24 (ddd, J = 14.5, 6.4, 3.0 Hz, 1H), 2.18 (sept d, J = 7.0, 3.2 Hz, 1H), 1.75-1.16 (m, 12H), 1.13 (d, J = 7.0 Hz, 3H), 0.82 (d, J = 7.0 Hz, 3H); ^{13}C NMR (101 MHz, $CDCl_3$) δ 209.7, 201.2, 165.5, 163.5, 146.5, 133.7,

130.4, 128.53, 128.51, 48.0, 44.4, 42.7, 41.9, 29.1, 26.3, 26.14, 26.12, 25.5, 25.4, 24.2, 23.1, 21.1, 16.2; mass spectrum m/z 396 (M^+, 3), 291 (5), 106 (8), 105 (100), 77 (24); exact mass calcd for $C_{25}H_{32}O_4$ 396.2301, found 396.2282.



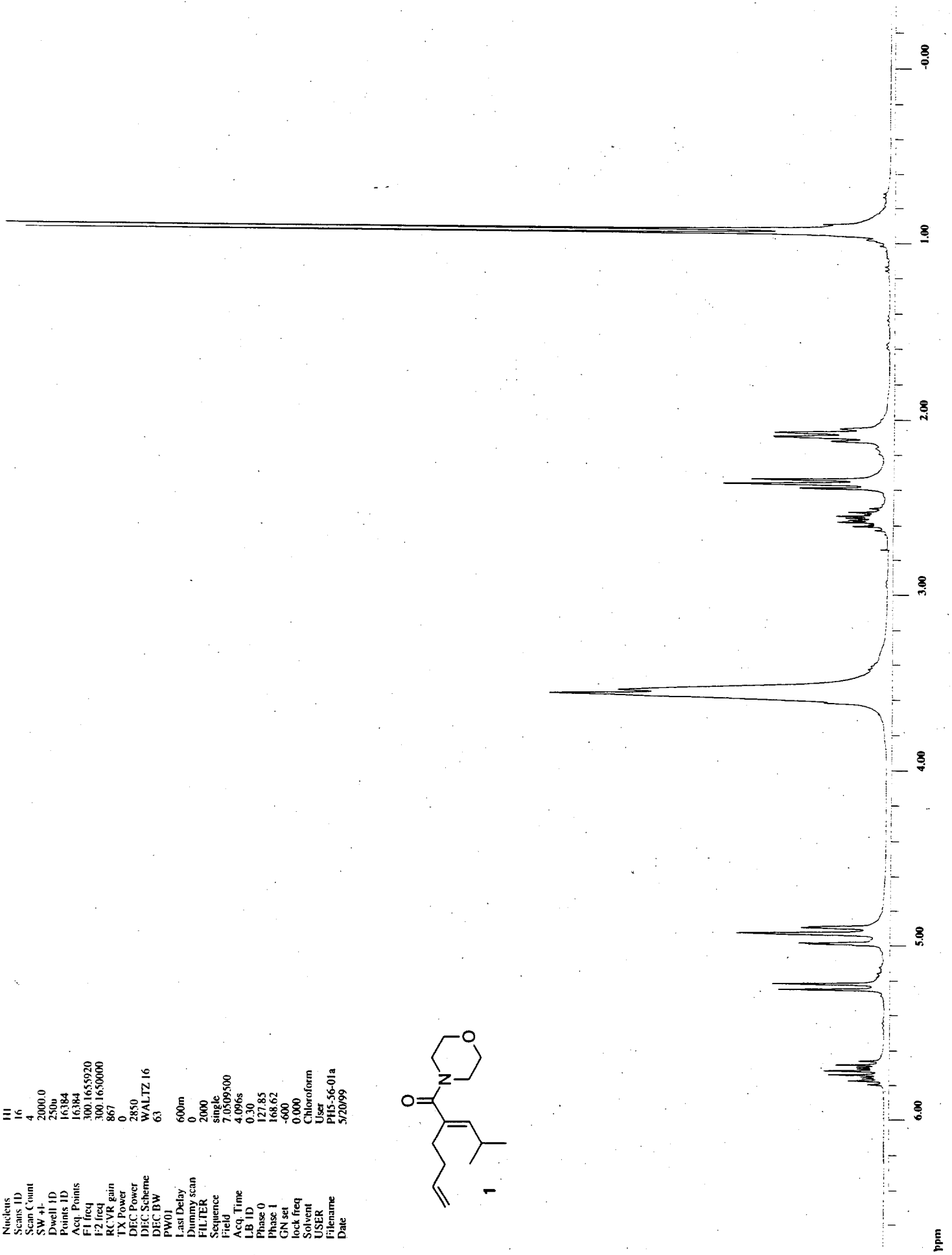
5 **3**

A degassed solution of the diketone **5** (31.7 mg, 0.0799 mmol) and ammonium carbonate (270 mg, 2.81 mmol) in propionic acid (2.2 mL) was heated to 140°C for 10 h. After 10 h, the reaction mixture was diluted with Et_2O and saturated $NaHCO_3$. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with brine (1 x) and dried over $MgSO_4$. Purification by flash column chromatography on silica gel (5% to 10% to 20% EtOAc in hexanes) gave the ketopyrrole **3** (11.4 mg, 52%) as a white solid: R_f = 0.34 (20% EtOAc in hexanes); IR (neat) 3180, 2940, 1675, 1475, 1380, 1340, 1295, 1275, 830, 740, 640 cm^{-1}; ^1H NMR (300 MHz, CD_2Cl_2) δ 11.36 (s br, 1H), 5.99 (d, J = 1.5 Hz, 1H), 2.86 (dt, J = 13.7, 4.9 Hz, 1H), 2.74 (t, J = 4.3 Hz, 1H), 2.65 (ddd, J = 13.4, 10.8, 5.9 Hz, 1H), 2.63 (d, J = 6.6 Hz, 1H), 1.90-1.70 (m, 4H), 1.39-0.75 (m, 9H), 1.00 (d, J = 6.8 Hz, 3H), 0.88 (d, J = 6.6 Hz, 3H), 0.47-0.35 (m, 2H); ^1H NMR (300 MHz, $CDCl_3$) δ 11.36 (s br, 1H), 5.97 (d, J = 1.5 Hz, 1H), 2.86 (dt, J = 12.7, 4.8 Hz, 1H), 2.79 (t br, J = 4.2 Hz, 1H), 2.69 (ddd, J = 13.7, 10.5, 5.6 Hz, 1H), 2.62 (d, J = 6.6 Hz, 1H), 1.98-1.72 (m, 4H), 1.37-0.76 (m, 9H), 1.00 (d, J = 6.6 Hz, 3H), 0.88 (d, J = 6.6 Hz, 3H), 0.47-0.34 (m, 2H); ^{13}C NMR (75 MHz, THF-d_8) δ 192.5, 154.9, 147.7, 135.6, 107.1, 59.6, 49.1, 34.2, 32.7, 29.3, 29.2, 28.7, 28.5, 28.2, 26.6, 26.0, 21.9, 20.3; ^{13}C NMR (75 MHz, $CDCl_3$) δ 194.0, 156.9, 149.9, 133.6, 106.9, 58.8, 48.2, 33.1, 32.0, 28.3, 28.2, 28.1, 27.44, 27.35, 25.6, 24.9, 21.5, 20.0; mass spectrum m/z 273 (M^+, 14), 258 (15), 205 (39), 145 (11), 115 (11), 105 (15), 91 (24), 81 (10), 79 (17), 78 (100); exact mass calcd for $C_{18}H_{27}NO$ 273.2093, found 273.2086.



5 13

A solution of the diketone **5** (29 mg, 0.073 mmol) and benzylamine (500 μL, 491 mg, 4.58 mmol) in propionic acid (5 mL) was heated to 200°C for 10 d. After 10 d, the reaction mixture was diluted with Et_2O, 1 M HCl, and water. The aqueous phase was extracted with ether (3 x) and the combined organic extracts were washed with saturated $NaHCO_3$ (2 x), brine (1 x), and dried over $MgSO_4$. Purification by flash column chromatography on silica gel (1.25% to 2.5% to 5% to 10% to 20% to 30% to 50% EtOAc in hexanes) gave the *N*-benzylpyrrole **13** (9.1 mg, 34%) as a low melting solid: R_f = 0.36 (10% EtOAc in hexanes); IR (neat) 2920, 2855, 1685, 1670, 1500, 1475, 1460, 1395, 1255, 800, 725, 700 cm^{-1}; ^1H NMR (400 MHz, CD_2Cl_2) δ 7.31 (tm, J = 7.2 Hz, 2H), 7.25 (tm, J = 7.2 Hz, 1H), 7.19 (dm, J = 7.8 Hz, 2H), 6.00 (s, 1H), 5.67 (d, J = 15.6 Hz, 1H), 4.98 (d, J = 15.6 Hz, 1H), 2.71-2.61 (m, 3H), 2.58 (ddd, J = 14.4, 7.2, 6.0 Hz, 1H), 1.95 (m, 1H), 1.81-1.67 (m, 3H), 1.50 (m, 1H), 1.24 (m, 1H), 1.15-0.84 (m, 6H), 0.99 (d, J = 6.8 Hz, 3H), 0.87 (d, J = 6.8 Hz, 3H), 0.76-0.67 (m, 2H), 0.57 (m, 1H); ^{13}C NMR (101 MHz, CD_2Cl_2) δ 193.7, 154.1, 147.0, 138.7, 135.1, 129.0, 127.8, 127.2, 109.4, 59.7, 48.6, 47.9, 33.5, 32.1, 28.4, 28.0, 27.7, 27.4, 25.7, 25.4, 25.3, 21.5, 19.9; mass spectrum m/z 363 (M^+, 19), 321 (13), 320 (21), 106 (13), 92 (9), 91 (100); exact mass calcd for $C_{25}H_{33}NO$ 363.2562, found 363.2574.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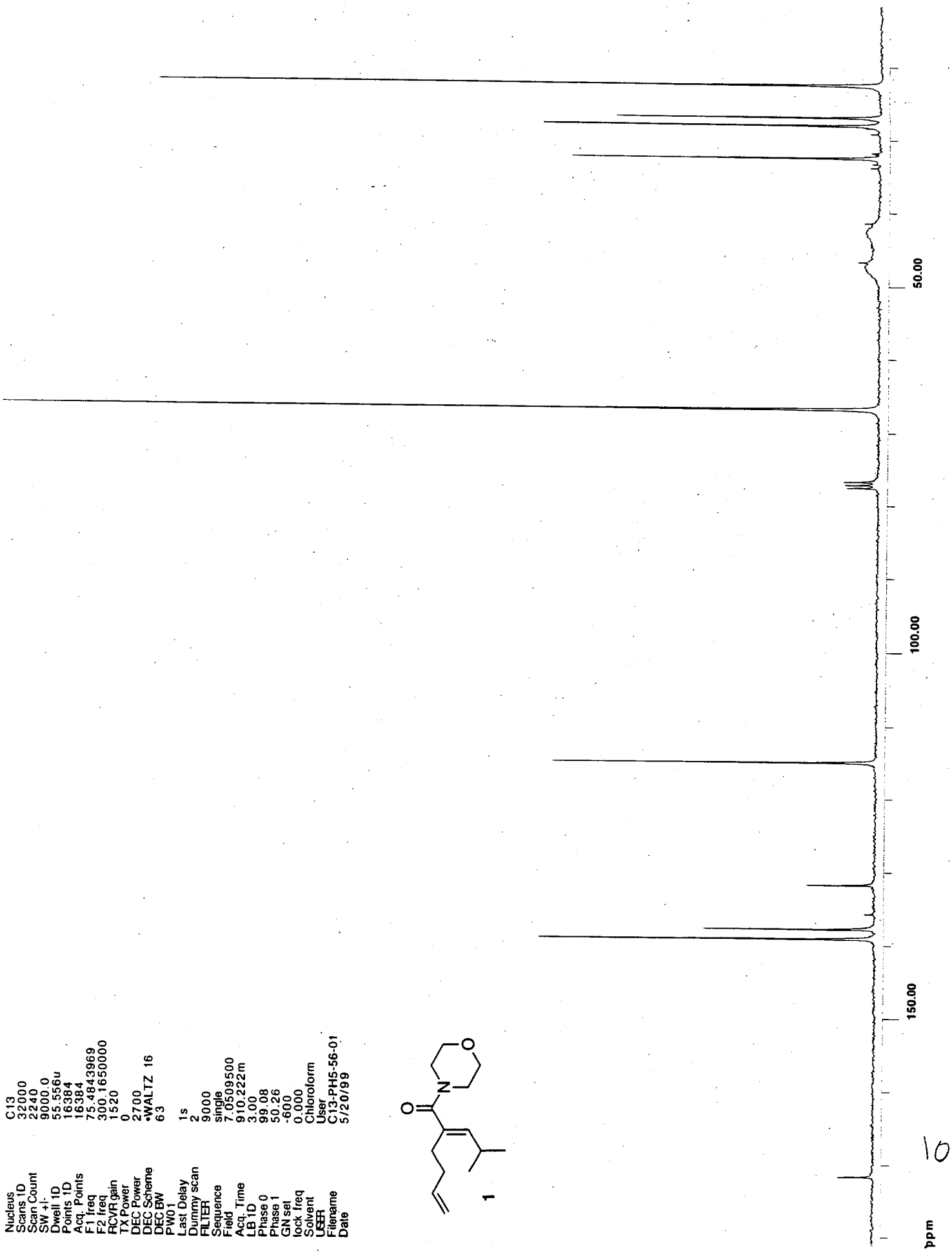
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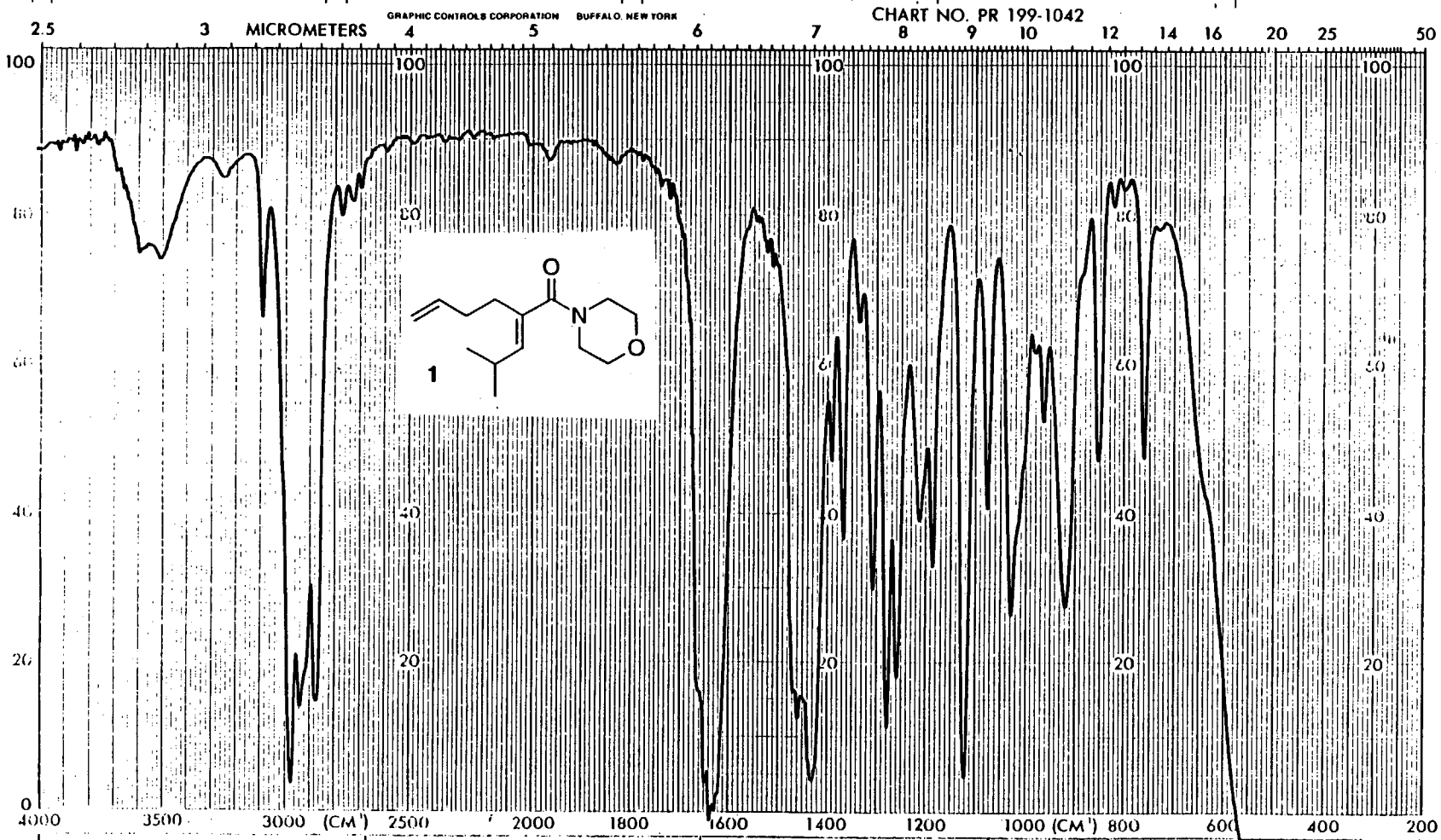

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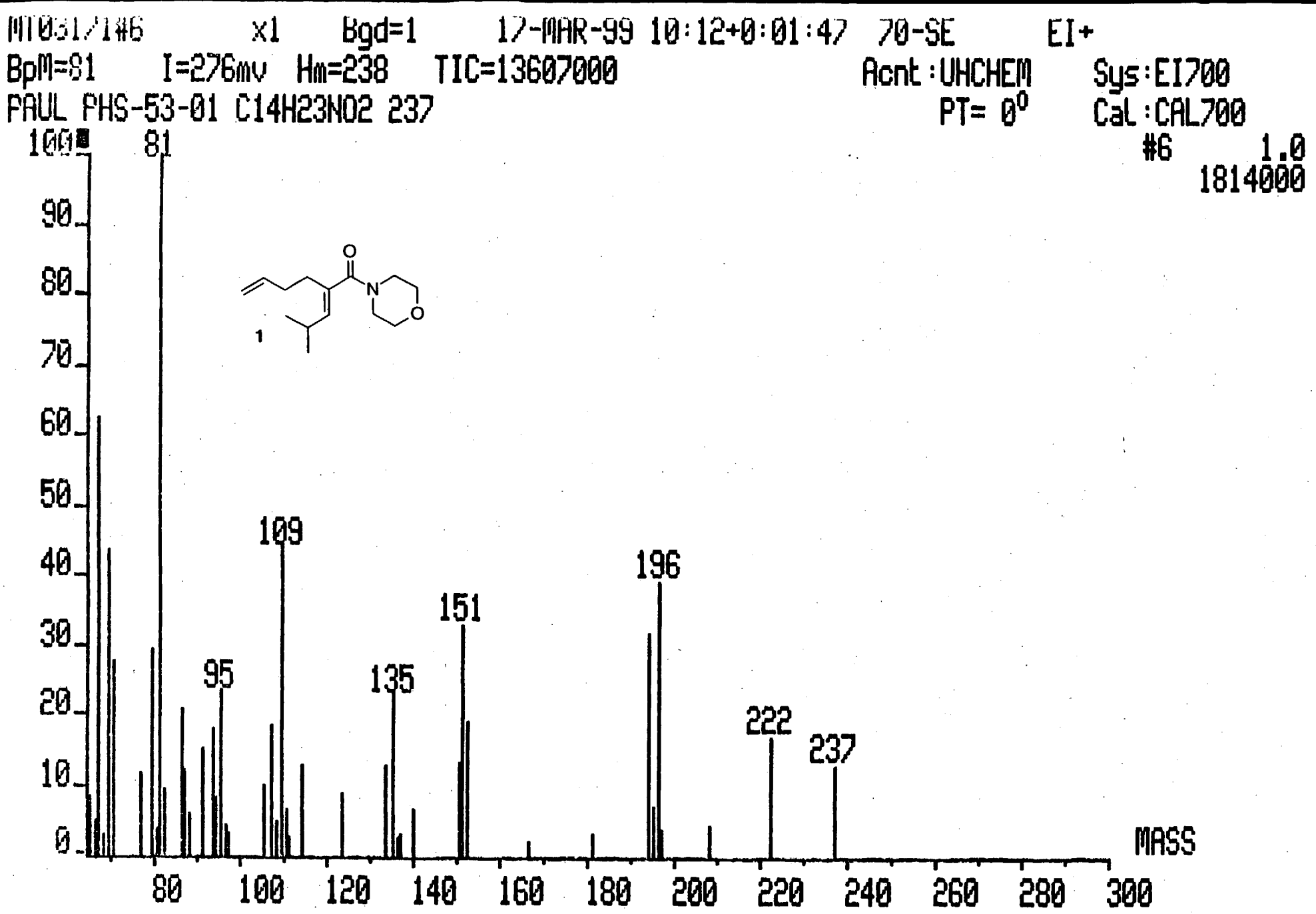

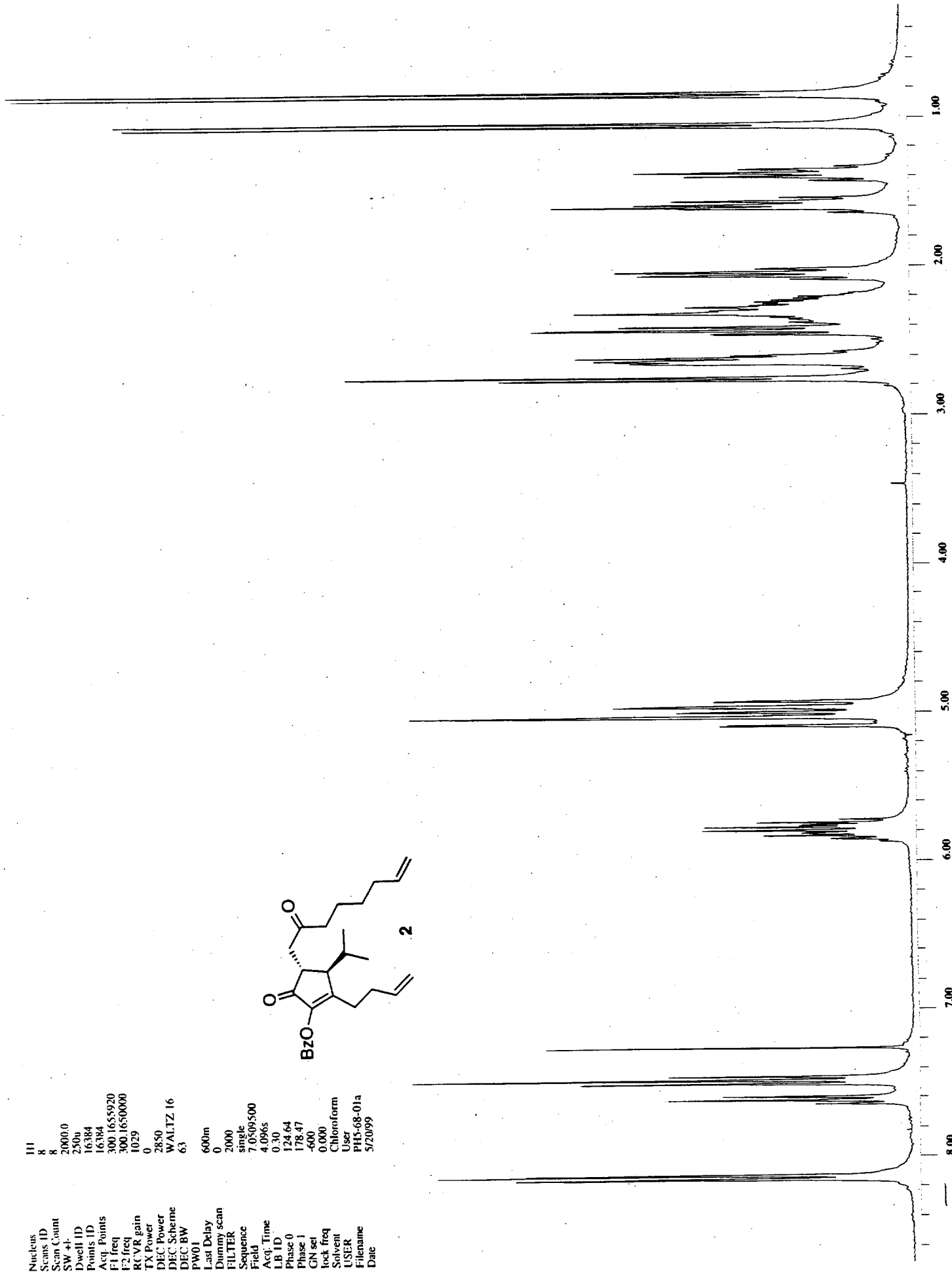

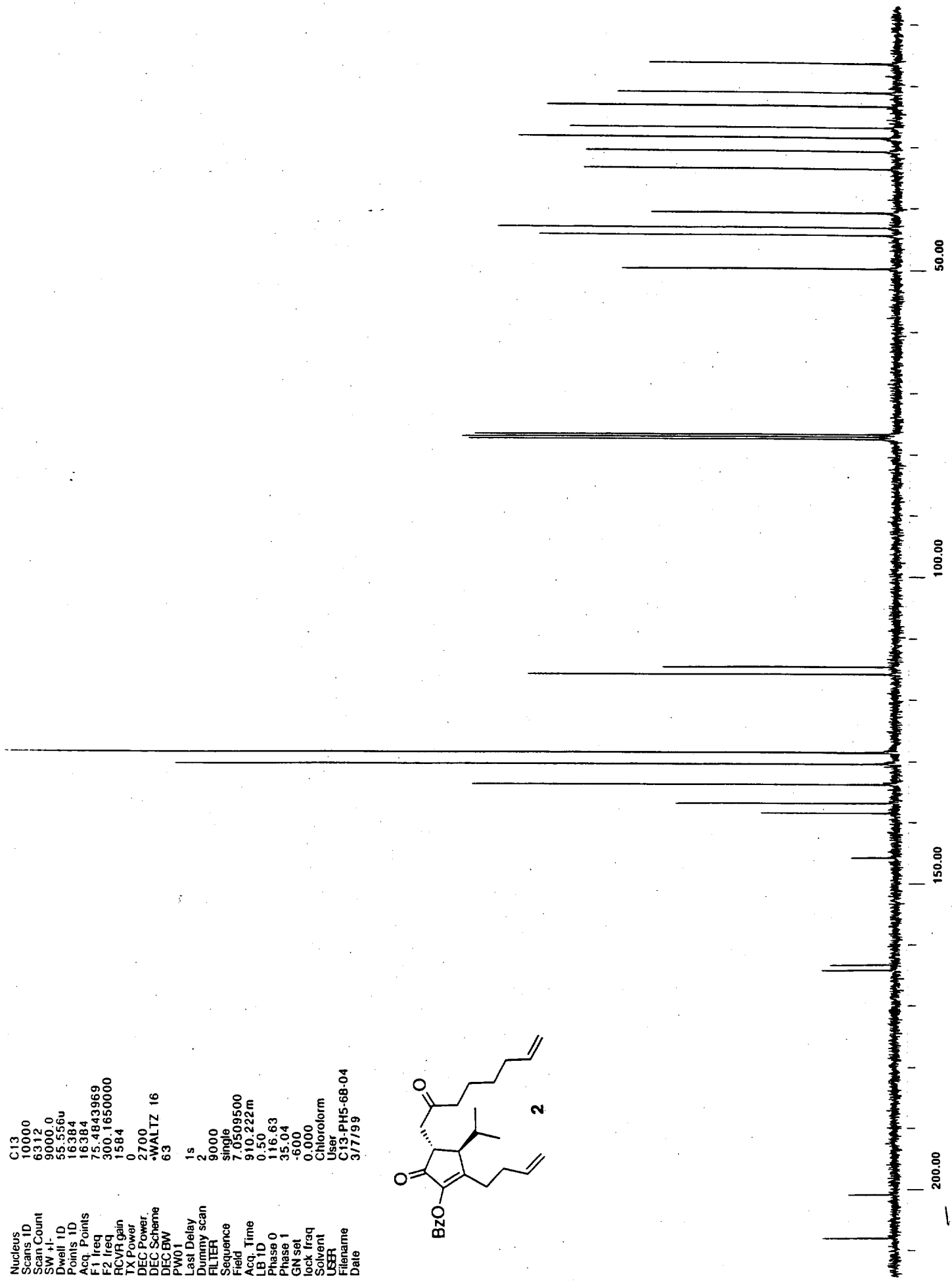

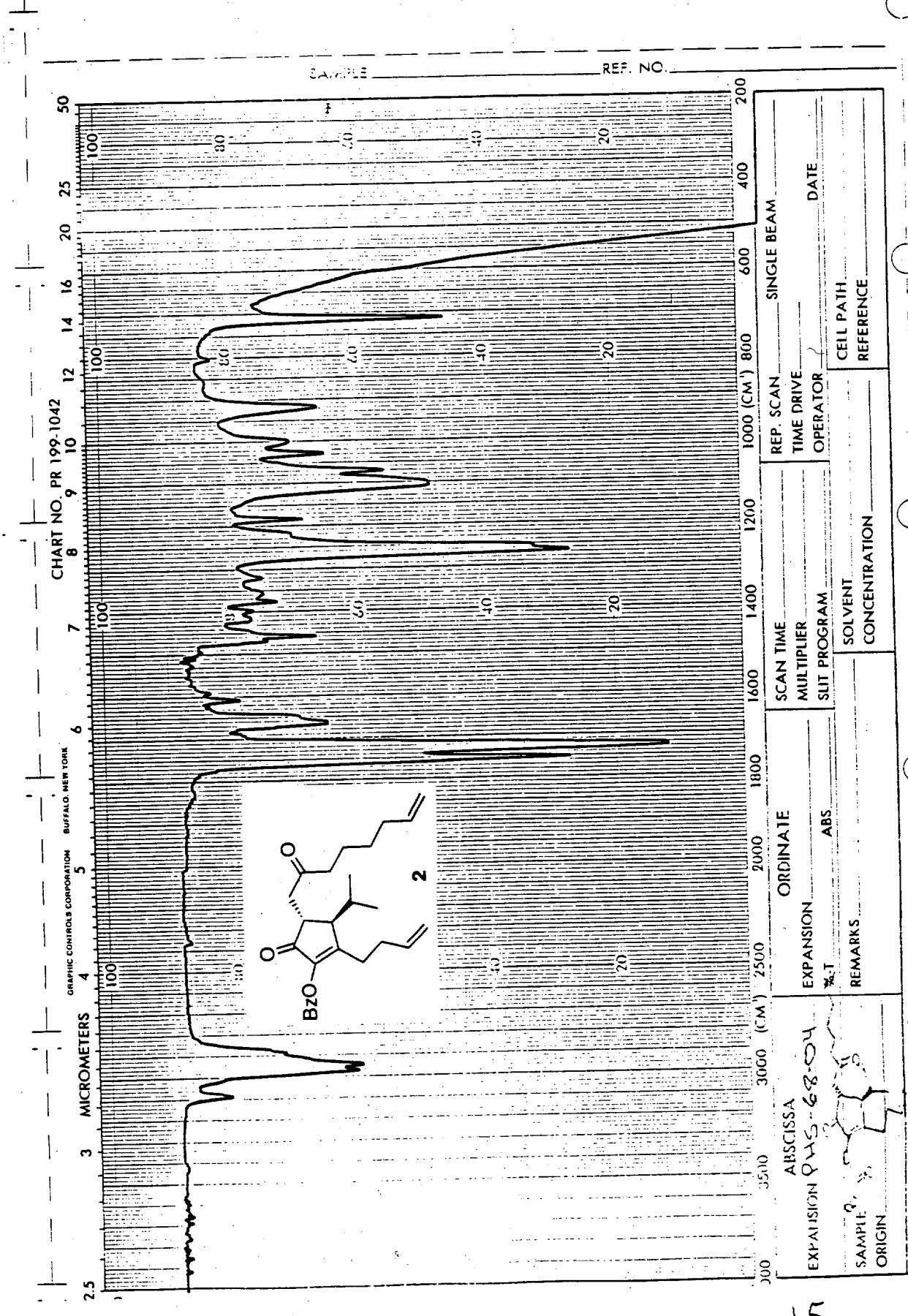

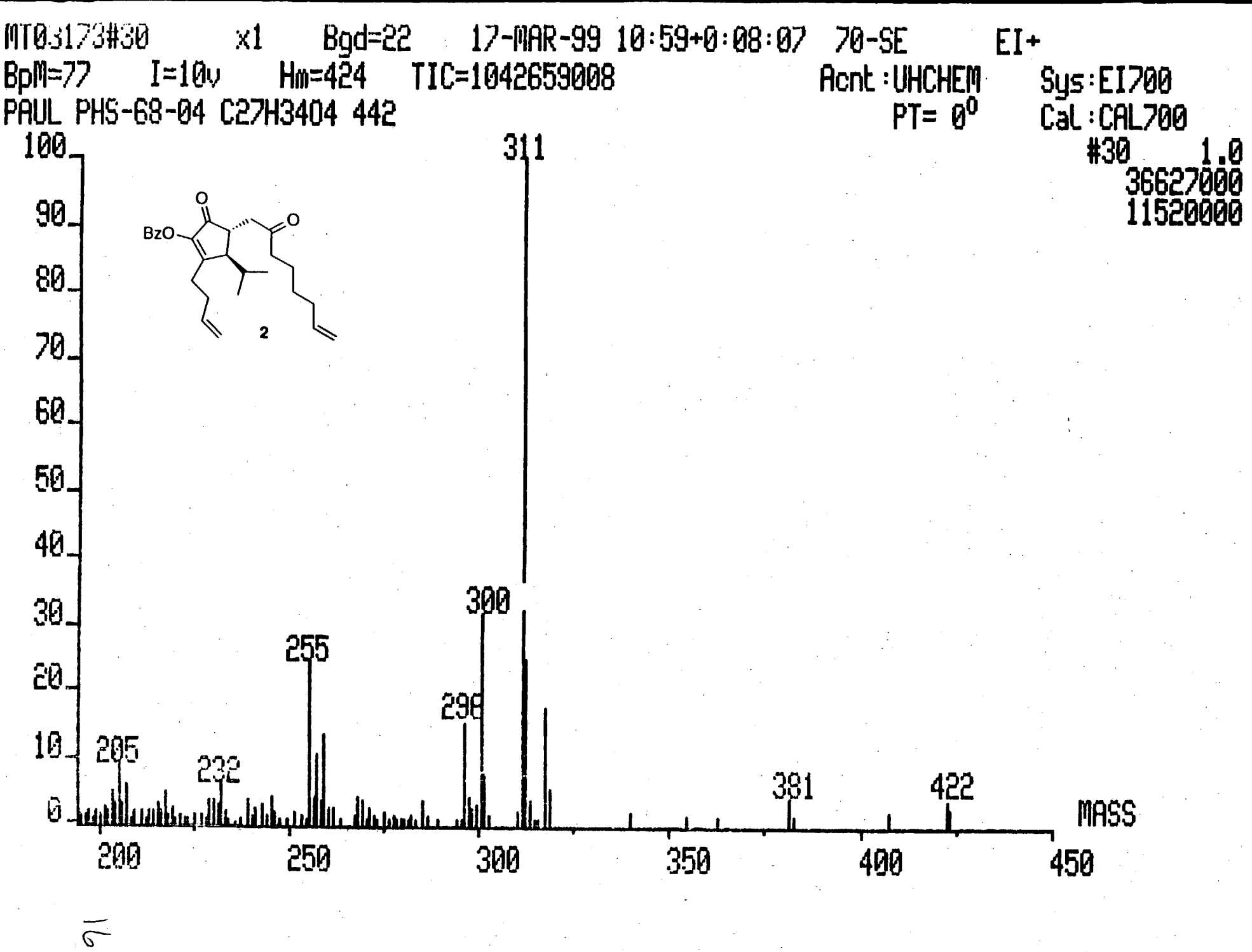

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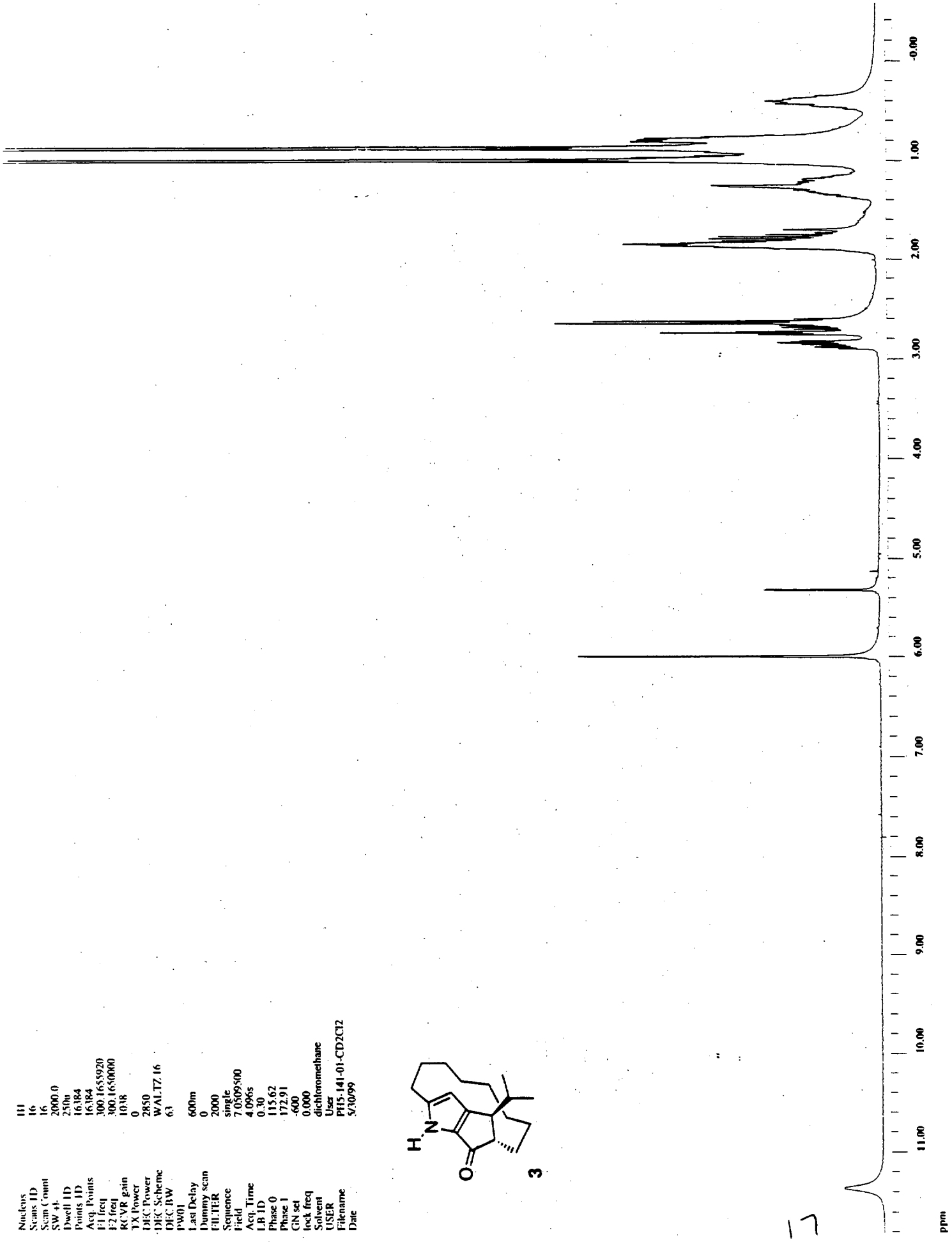

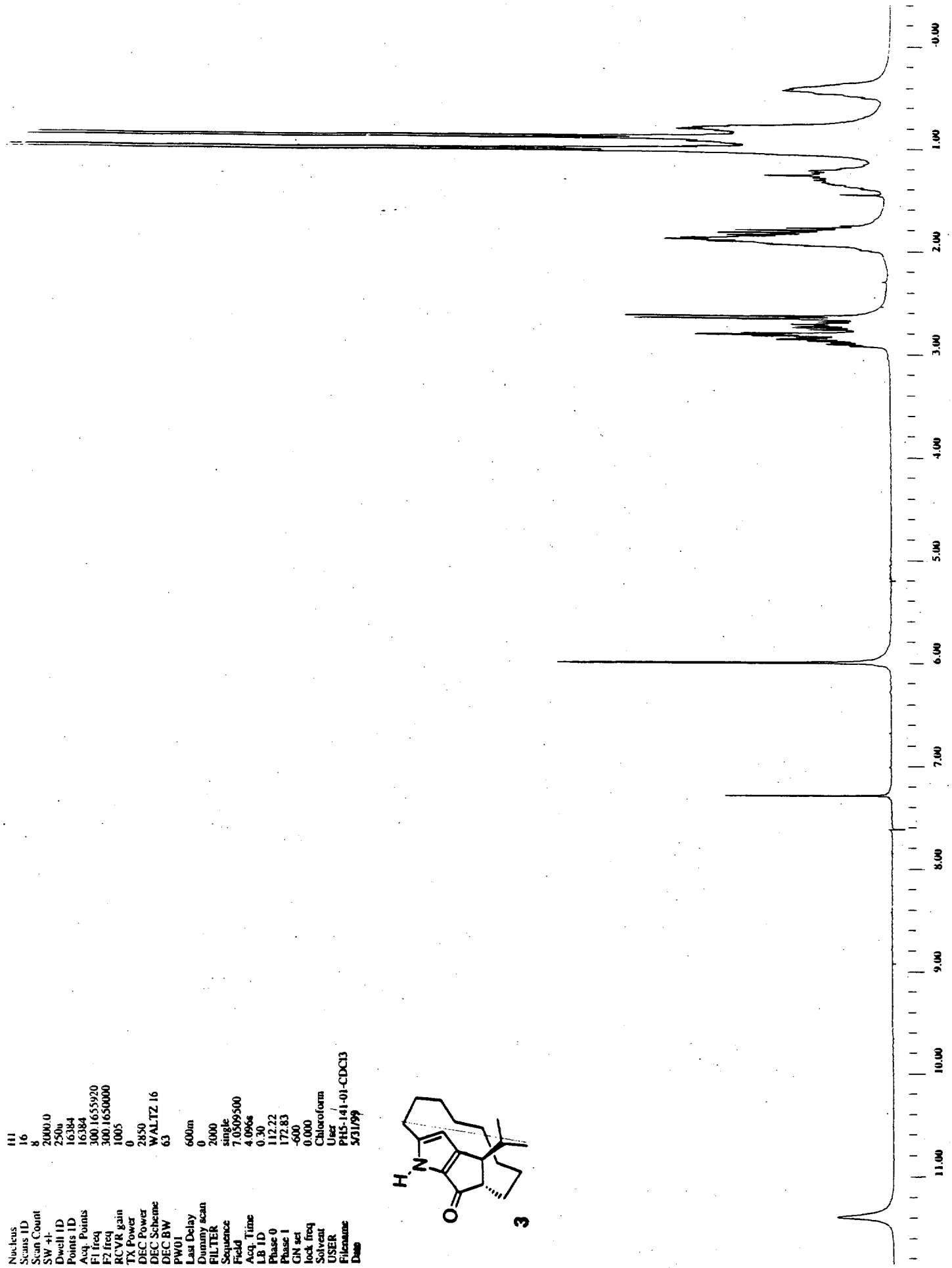

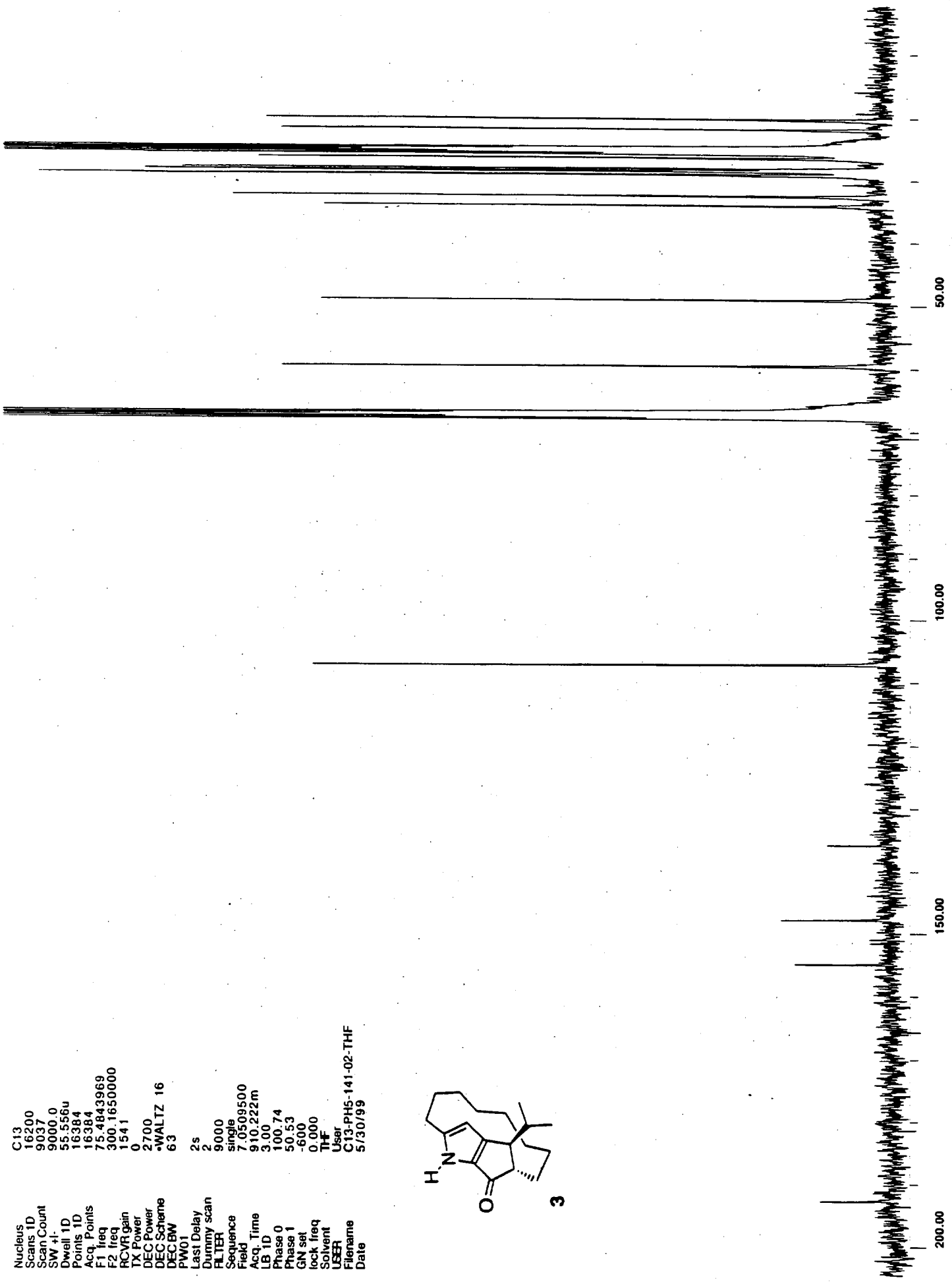
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F2 freq 300.1650000
RCVR gain 1541
TX Power 0
DEC Power 2700
DEC Scheme WALTZ 16
DEC BW 63
PW01
Last Delay 2s
Dummy scan 2
FILTER 9000
Sequence single
Field 7.0509500
Acq. Time 910.222m
LB 1D 3.00
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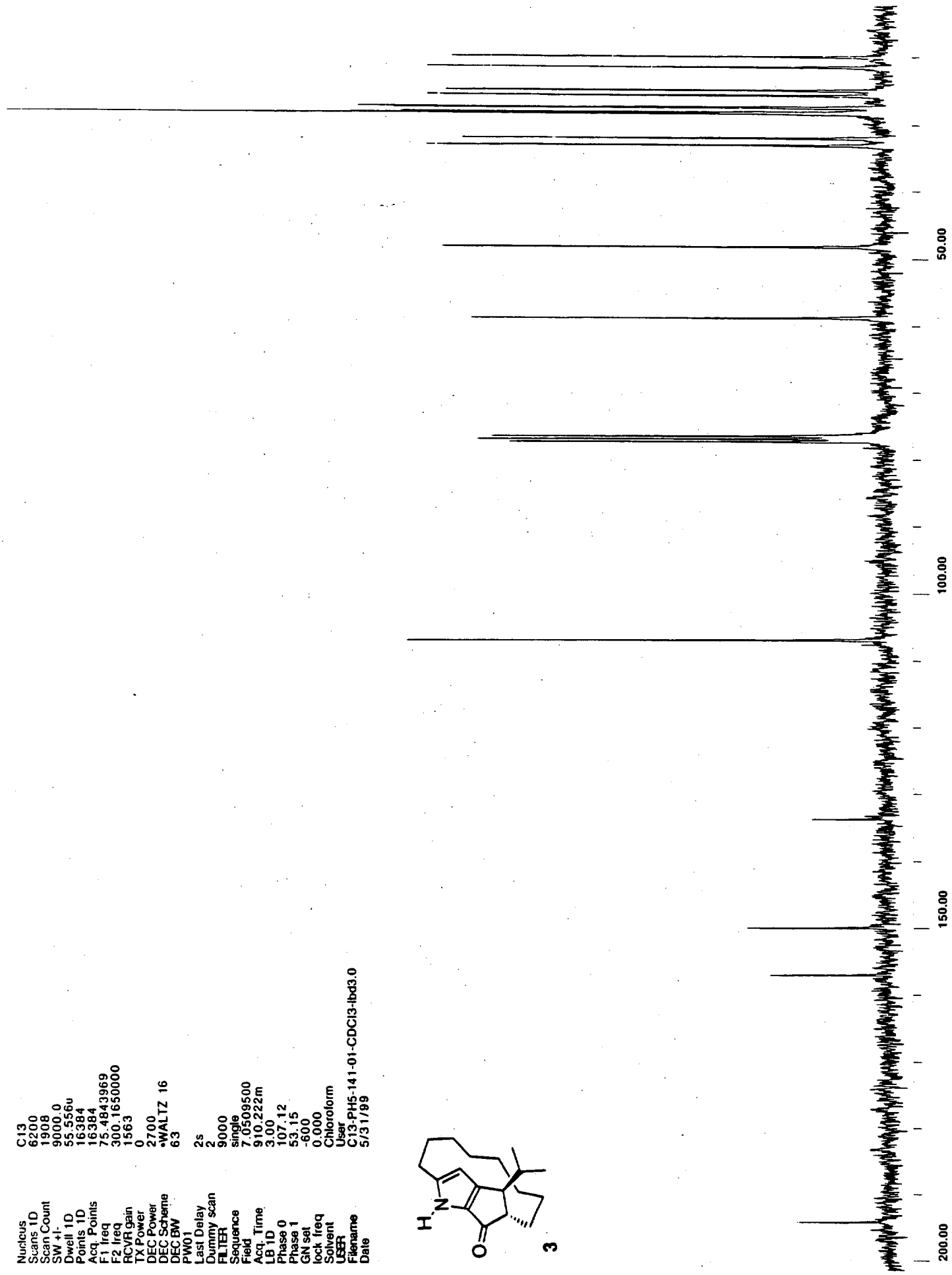

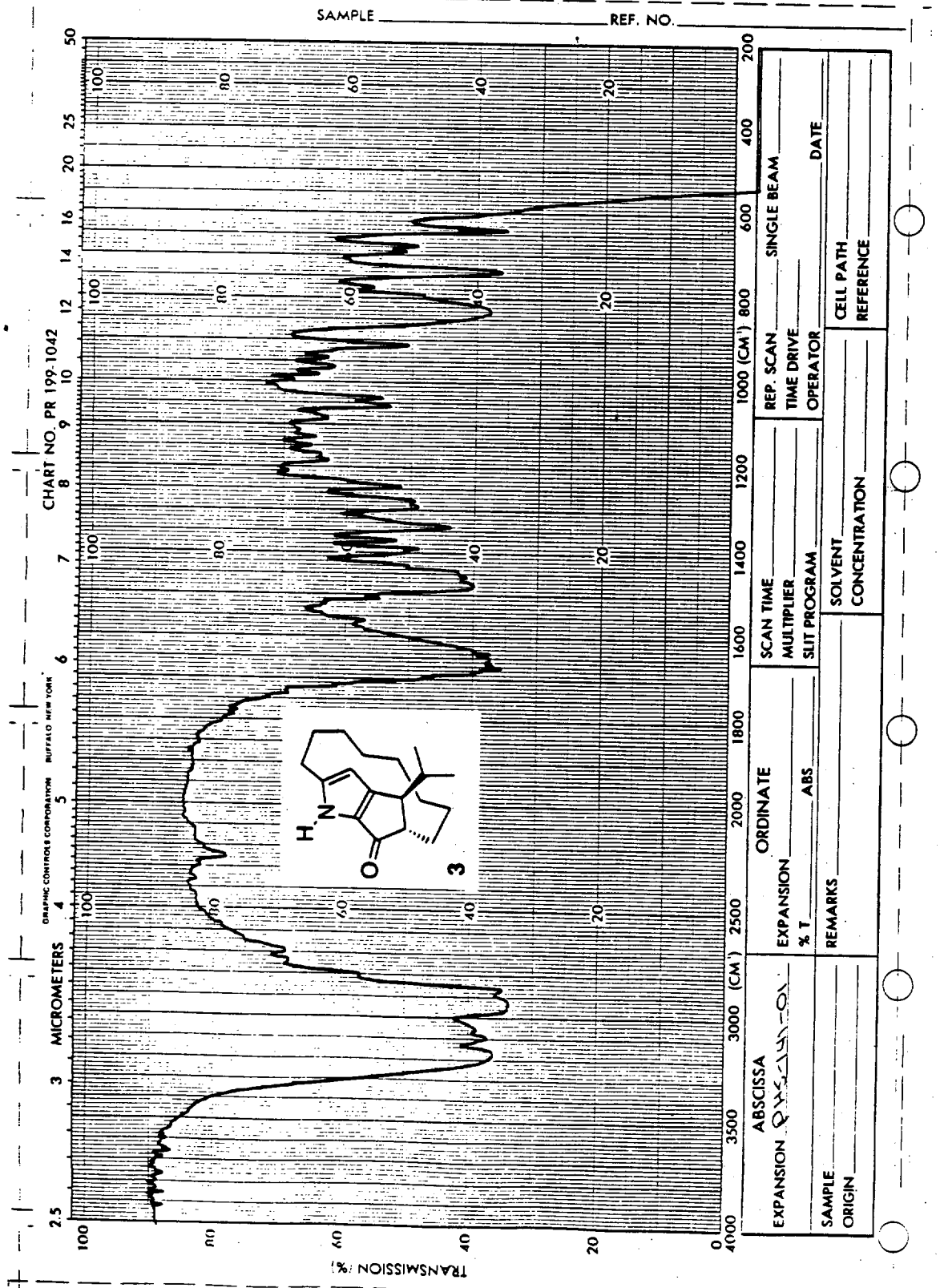

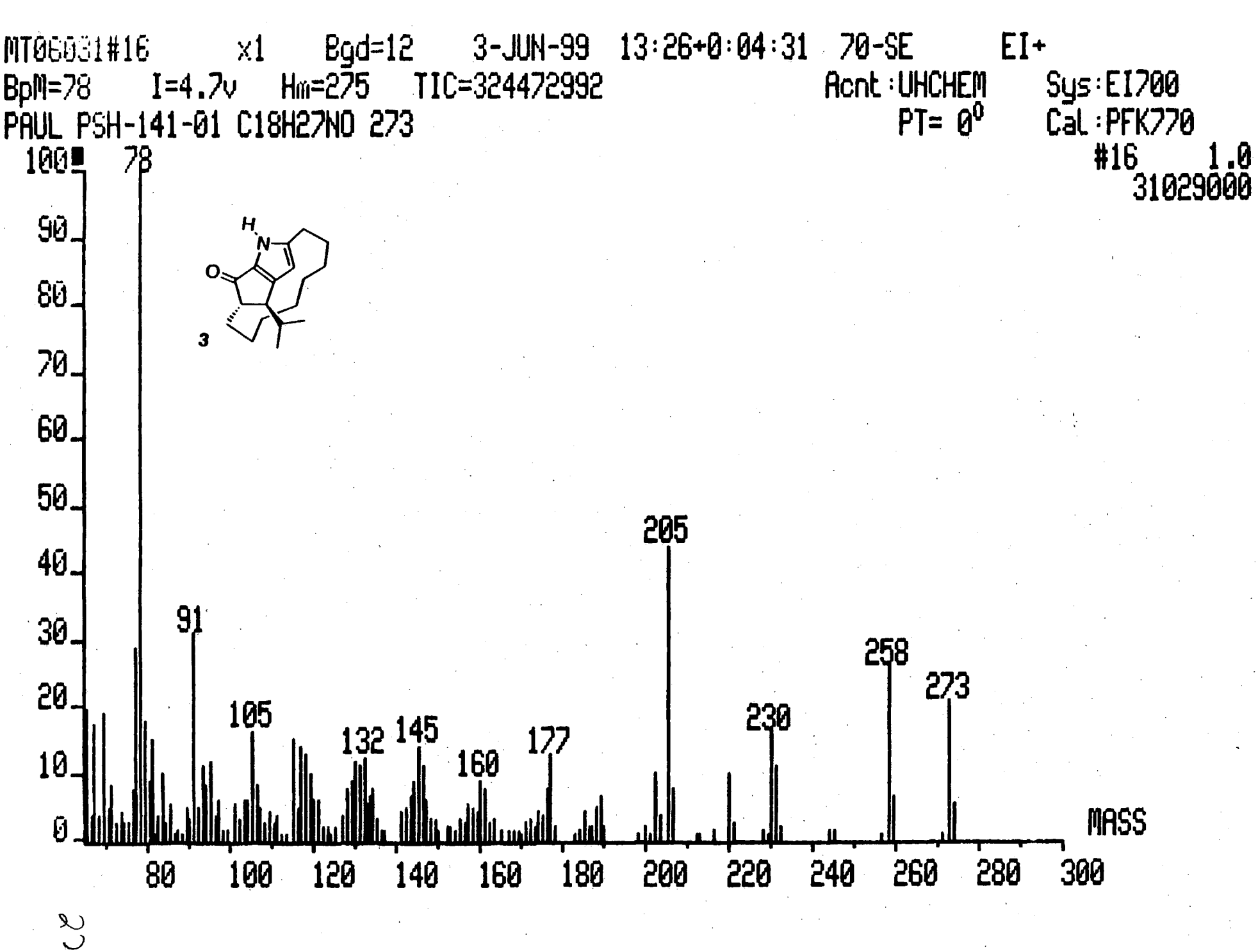

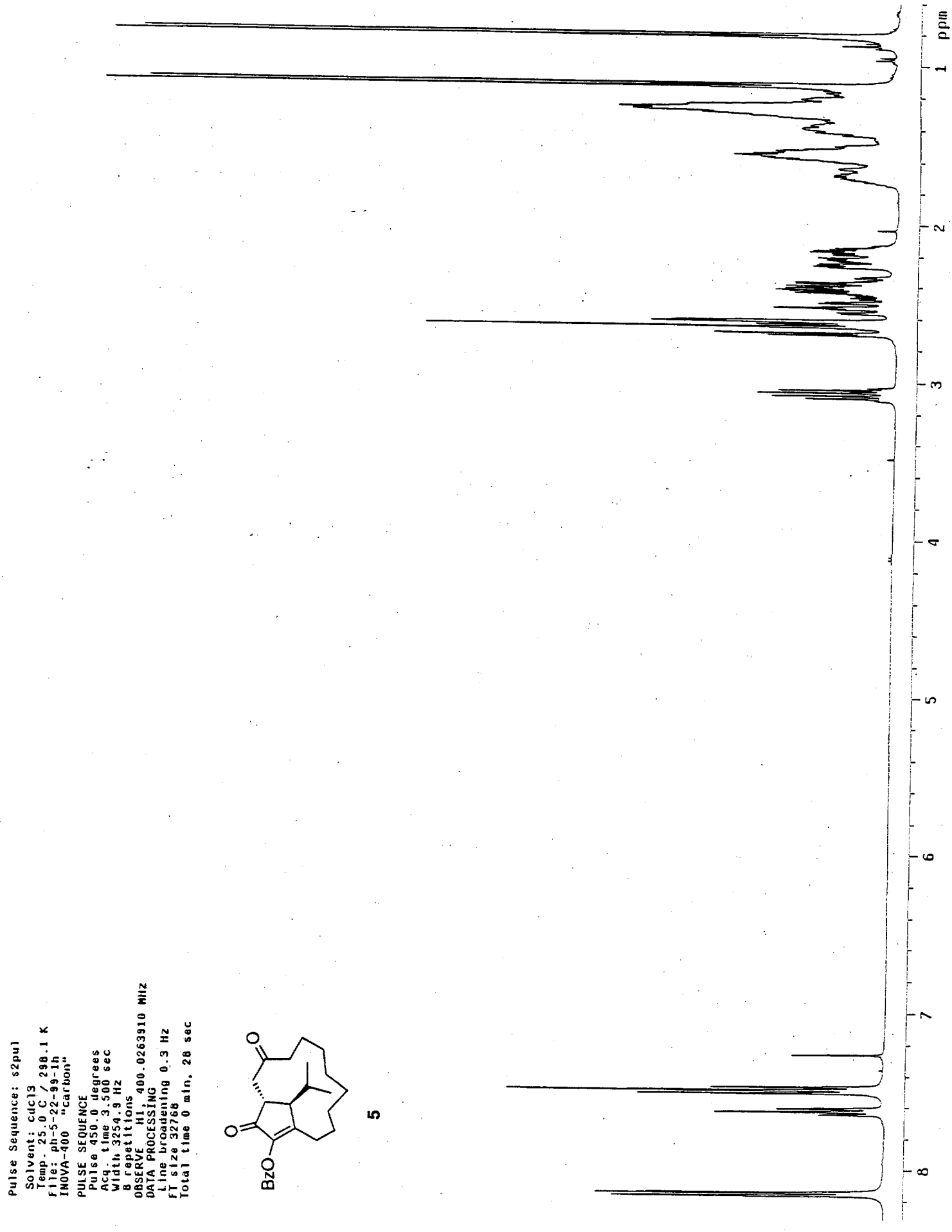

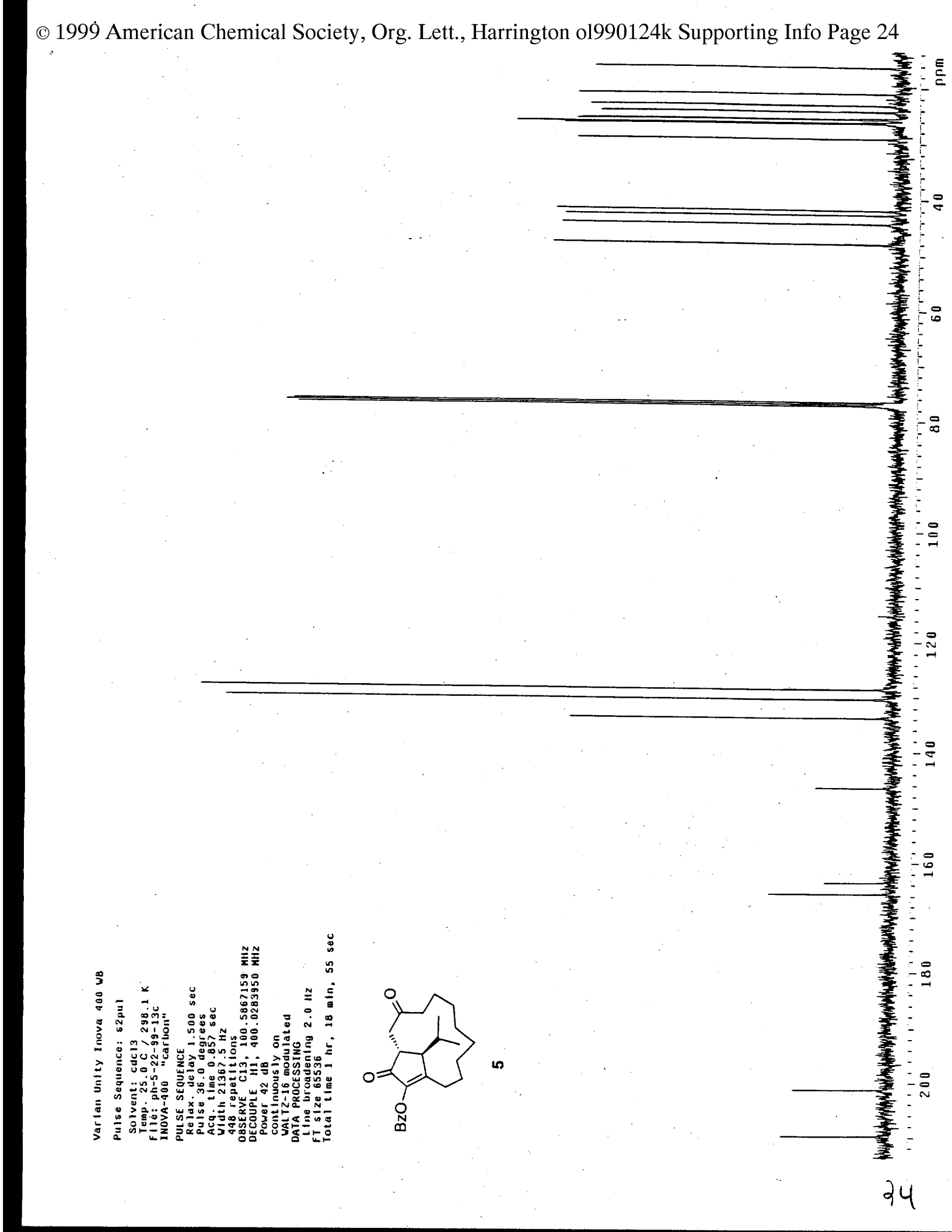

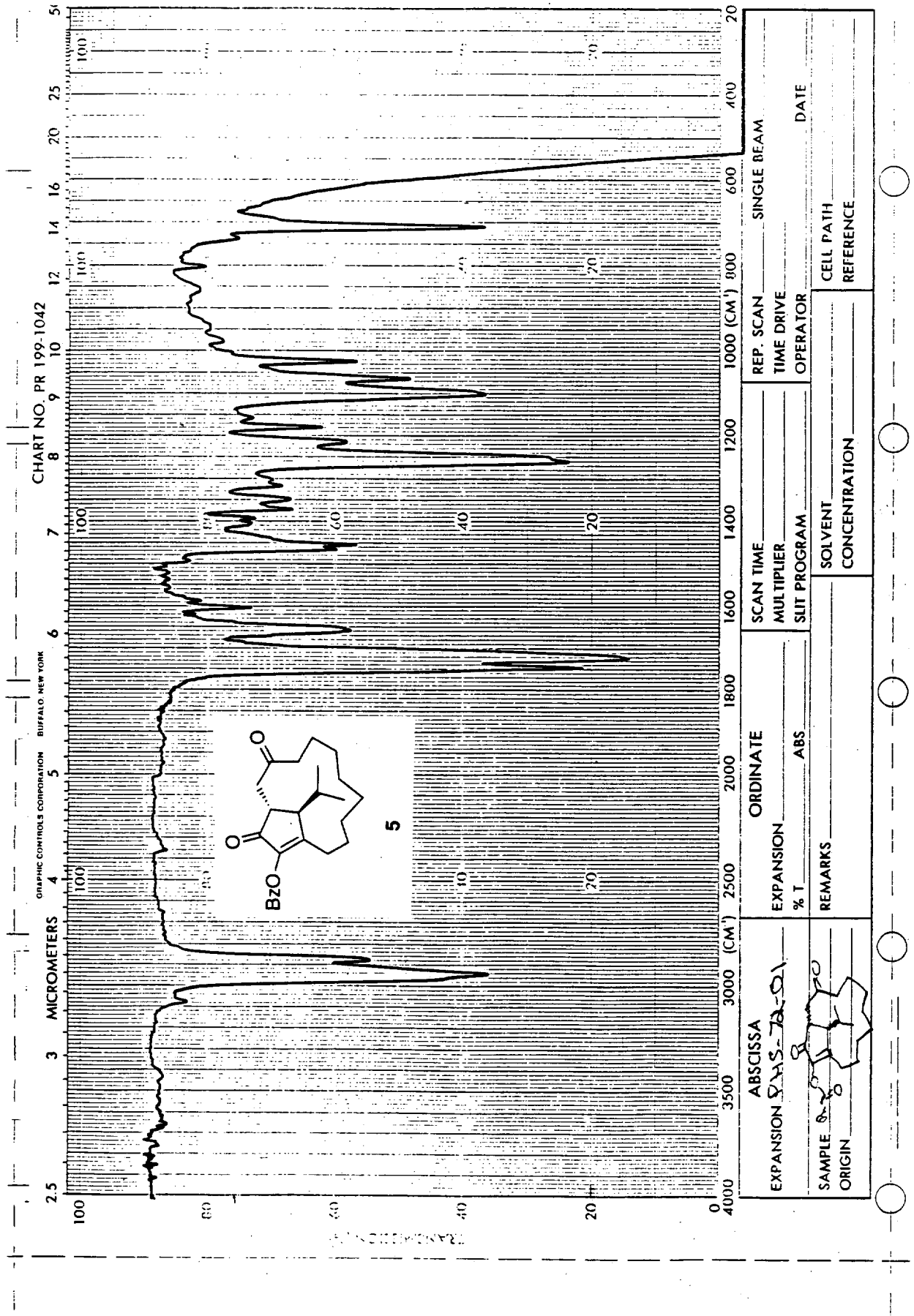

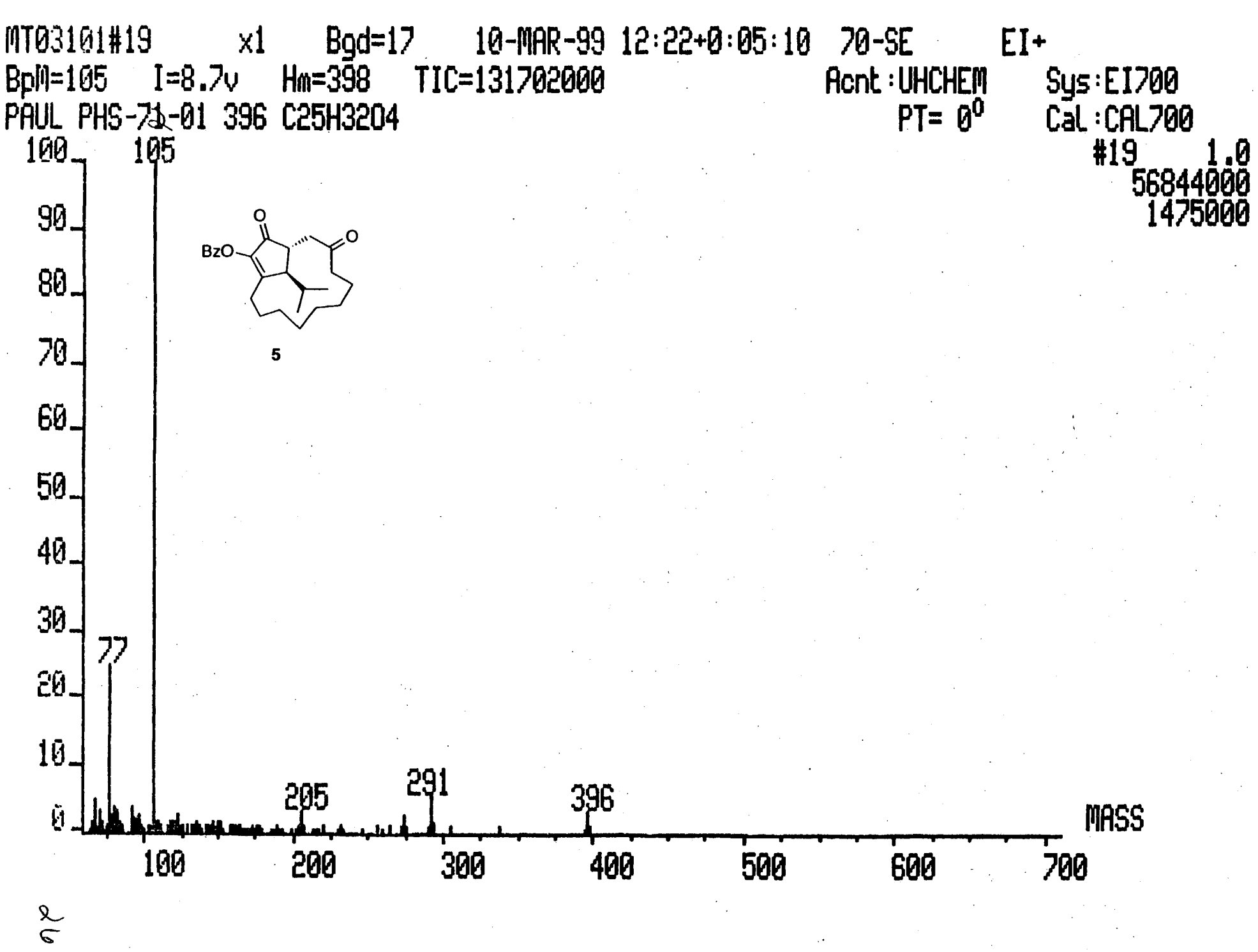

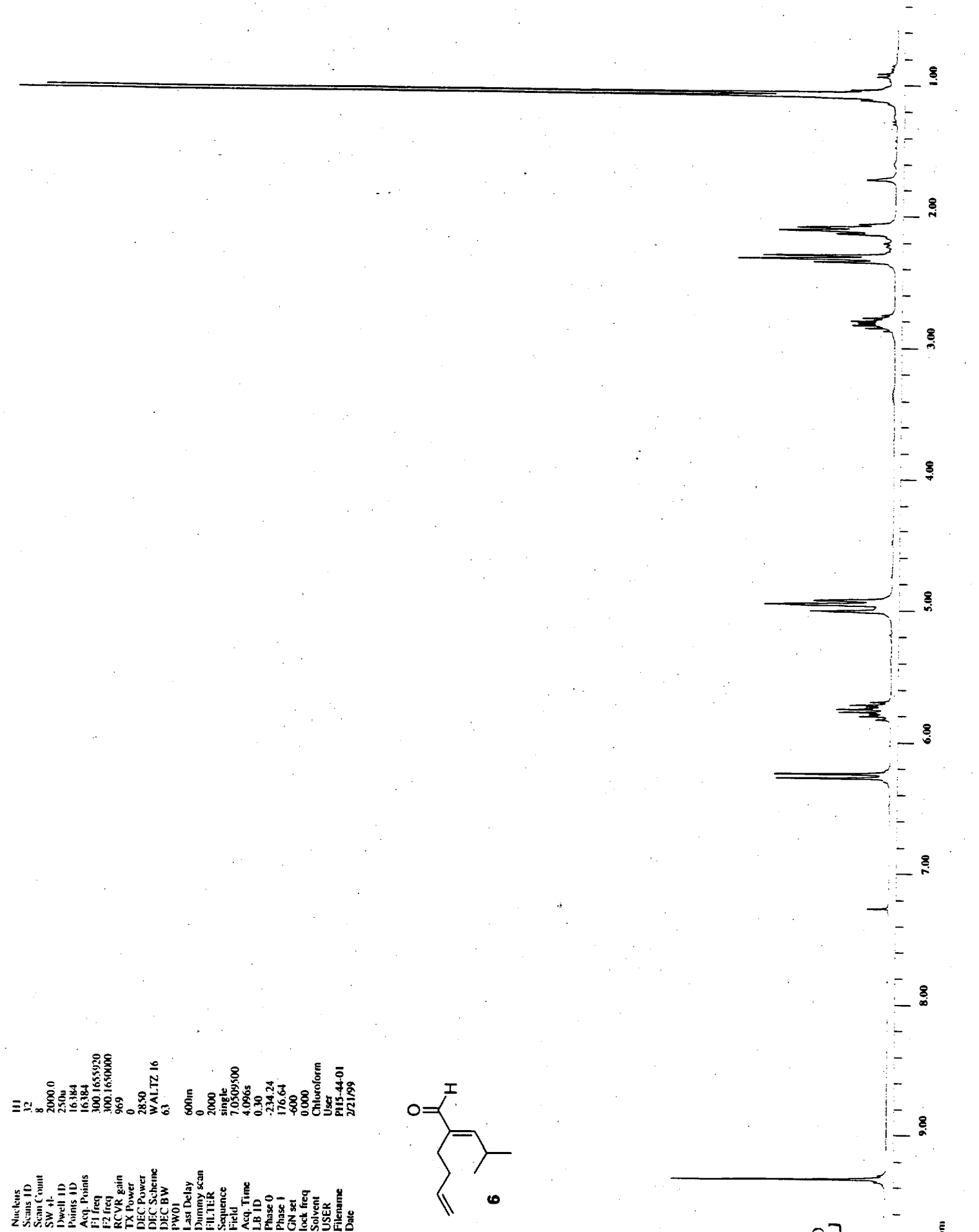

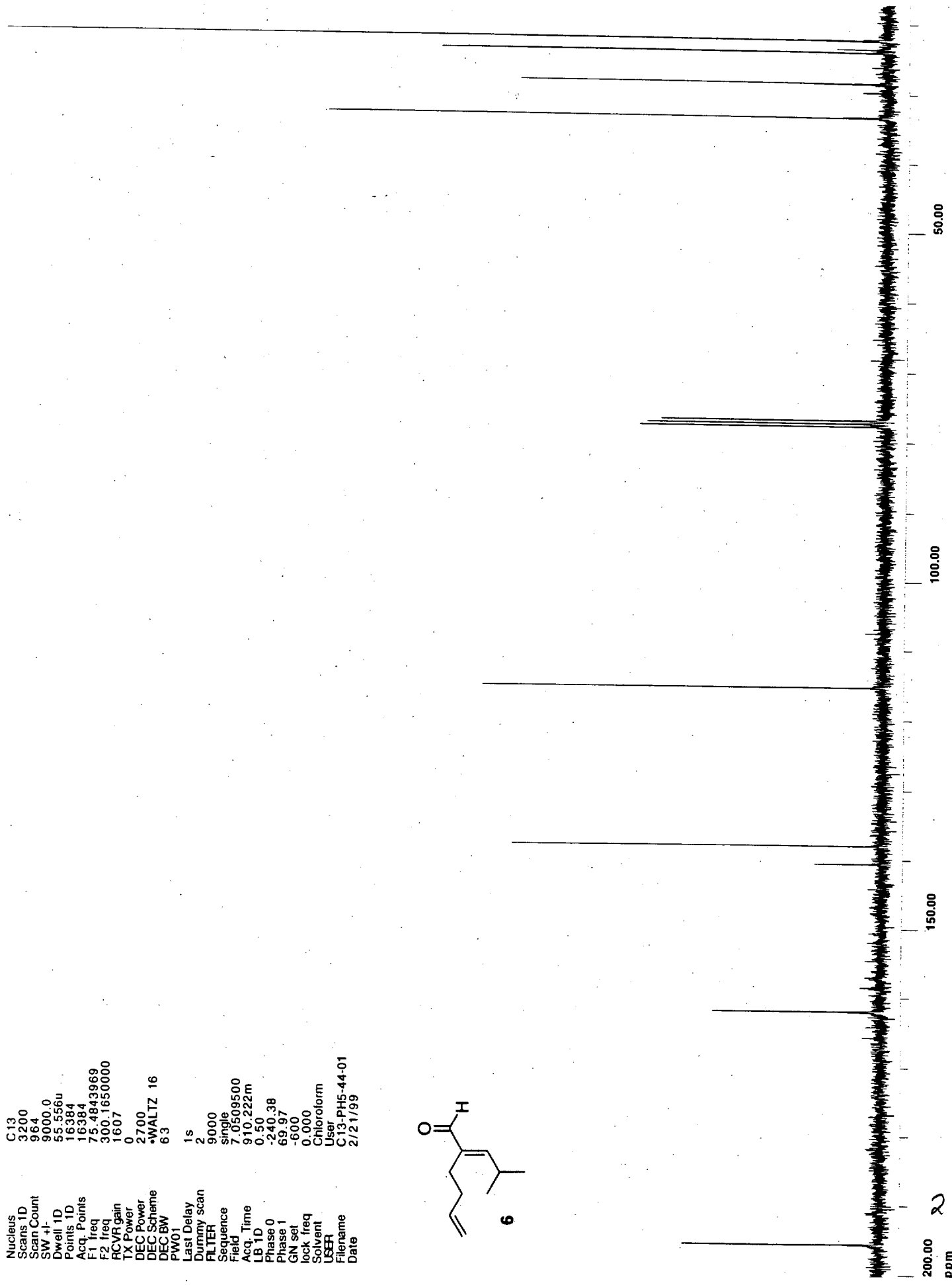
Nucleus C13
Scans 1D 3200
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Acq. Points 16384
F1 freq 75.4843969
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RCVR gain 1607
TX Power 0
DEC Power 2700
DEC Scheme •WALTZ 16
DEC BW 63
PW01
Last Delay 1s
Dummy scan 2
FILTER 9000
Sequence single
Field 7.0509500
Acq. Time 910.222m
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Phase 0 -240.38
Phase 1 69.97
GN set -600
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USER User
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100.00
50.00
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