

Supporting Information

Total Synthesis of (±)-Scopadulin

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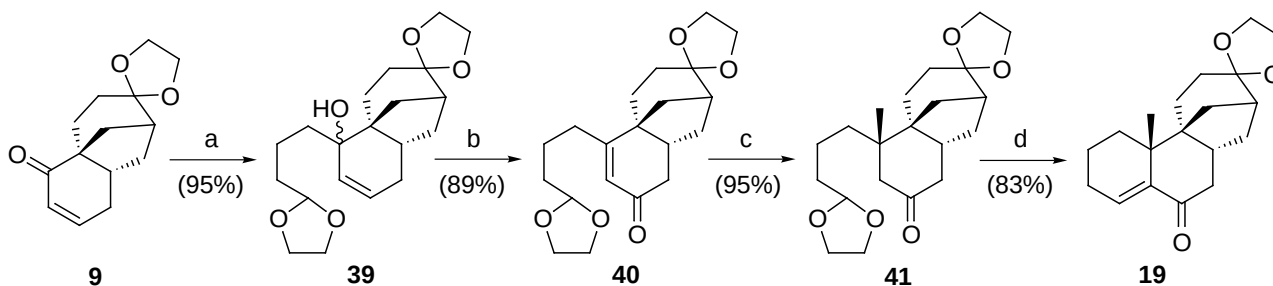
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Preparation of the Enone (19)

As shown in the Scheme S1, the enone **19** was prepared by the similar procedures described in our published literature.¹ However, in some cases better yields of the respective products were observed. Our previous reported data for **19** and the intermediates were precise (recorded by either 200 or 250 MHz) and incomplete (the allylic alcohol **39** was not characterized and none of the compound included ¹³C NMR data). Herein, we present the updated and/or augmented data for the enone **19** and its intermediates (compounds **39**, **40**, and **41**).

1. Tanaka, T.; Okuda, O.; Murakami, K.; Yoshino, H.; Mikamiyama, H.; Kanda, A; Kim, S.-W.; Iwata, C. *Chem. Pharm. Bull.* **1995**, 43, 1407.

Scheme S1



(±)-(1*R*,2*R*,6*R*,8*S*)-2-[3-(1,3-Dioxolan-2-yl)propyl]-2-hydroxytricyclo[6.3.1.0^{1,6}]dodec-3-en-9-one 9,9-Ethylene Acetal and Its (1*S,2*R**,6*S**,8*R**)-Isomer (39).** Colorless oil; IR (KBr) cm⁻¹: 3515, 1645, 1133. ¹H-NMR (CDCl₃, 500 MHz) δ: 1.24-1.96 (m, 16H), 2.01-2.07 (m, 1H), 2.13-2.29 (m, 2H), 3.82-3.87 (m, 4H), 3.88-4.01 (m, 4H), 4.86 (t, *J* = 4.5 Hz, 1H), 5.45 (d, *J* = 10.0 Hz, 1H), 5.73 (ddd, *J* = 10.0, 5.0, 2.0 Hz, 1H). ¹³C-NMR (CDCl₃, 75 MHz) δ: 18.6, 25.6, 28.3, 30.5 (2C), 34.5, 34.8, 37.8, 39.7, 43.0, 47.3, 63.9, 64.5, 64.8 (2C), 77.2, 104.6, 111.8, 128.7, 132.9. MS (EI) *m/z* (%): 350 (M⁺, 5.3), 235 (100), 99 (100). HRMS (EI) Calcd for C₂₀H₃₀O₅: 350.2093. Found: 350.2100.

(±)-(1*R*,6*S*,8*S*)-2-[3-(1,3-Dioxolan-2-yl)propyl]tricyclo[6.3.1.0^{1,6}]dodec-2-ene-4,9-dione 9,9-Ethylene Acetal (40). Colorless oil; IR (KBr) cm⁻¹: 1662, 1602, 1114. ¹H-NMR (CDCl₃, 500

MHz) δ : 1.48-1.72 (m, 9H), 1.81-1.93 (m, 2H), 1.96-2.03 (m, 1H), 2.12 (d, J = 12.0 Hz, 1H), 2.27-2.41 (m, 4H), 2.58-2.68 (m, 1H), 3.82-3.93 (m, 4H), 3.93-4.02 (m, 4H), 4.87 (t, J = 4.5 Hz, 1H), 5.70 (s, 1H). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 20.9, 29.7, 30.6, 31.7, 32.5, 33.3, 36.9, 39.7, 42.7, 44.7, 46.8, 64.0, 64.7, 64.9 (2C), 104.1, 110.8, 124.6, 172.9, 201.0. MS (EI) m/z (%): 348 (M^+ , 4.1), 99 (29.7), 73 (100). HRMS (EI) Calcd for $\text{C}_{20}\text{H}_{28}\text{O}_5$: 348.1936. Found: 348.1943.

($\pm$)-(1*R*,2*R*,6*S*,8*S*)-2-[3-(1,3-Dioxolan-2-yl)propyl]-2-methyltricyclo[6.3.1.0^{1,6}]dodecane-4,9-dione 9,9-Ethylene Acetal (41). Colorless oil; IR (KBr) cm^{-1} : 1706, 1114. ^1H -NMR (CDCl_3 , 500 MHz) δ : 0.96 (s, 3H), 1.25-1.68 (m, 10H), 1.80-1.97 (m, 4H), 2.11 (d, J = 15.5 Hz, 1H), 2.21 (t, J = 6.5 Hz, 1H), 2.33 (d, J = 13.5 Hz, 1H), 2.34-2.40 (m, 1H), 2.35 (d, J = 15.5 Hz, 1H), 2.43 (d, J = 13.5 Hz, 1H), 3.80-4.02 (m, 8H), 4.85 (t, J = 5.0 Hz, 1H). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 18.8, 20.1, 26.0, 29.7, 29.9, 34.7, 35.4, 39.7, 39.9, 40.2, 42.9, 43.6, 46.2, 51.2, 64.0, 64.6, 64.9 (2C), 104.3, 111.3, 211.3. HRMS (EI) Calcd for $\text{C}_{21}\text{H}_{32}\text{O}_5$ (M^+): 364.2247. Found: 364.2237.

($\pm$)-(1*R*,2*S*,10*S*,12*S*)-2-Methyltetracyclo[10.3.1.0^{1,10}.0^{2,7}]hexadec-6-ene-8,13-dione 13,13-Ethylene Acetal (19). Colorless solid; mp 180-181 °C (*n*-hexane- CH_2Cl_2). IR (KBr) cm^{-1} : 1681, 1604, 1111. ^1H -NMR (CDCl_3 , 500 MHz) δ : 1.09 (s, 3H), 1.36-1.40 (m, 2H), 1.50-1.54 (m, 1H), 1.58-1.62 (m, 2H), 1.67-1.86 (m, 6H), 1.96-2.03 (m, 1H), 2.12-2.33 (m, 4H), 2.58-2.65 (m, 1H), 2.67 (d, J = 4.5 Hz, 1H), 3.83-4.02 (m, 4H), 6.72 (dd, J = 4.5, 3.0 Hz, 1H). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 18.1, 22.5, 25.6, 26.7, 29.8, 30.4, 30.6, 34.3, 36.1, 39.6, 41.7, 43.6, 45.8, 64.0, 64.6, 111.4, 138.0, 143.2, 201.5. MS (EI) m/z (%): 302 (M^+) *Anal.* Calcd for $\text{C}_{19}\text{H}_{26}\text{NO}_3$: C, 75.46; H, 8.67. Found: C, 75.35; H, 8.88.

Characterization of (29), (30), (32), and (33).

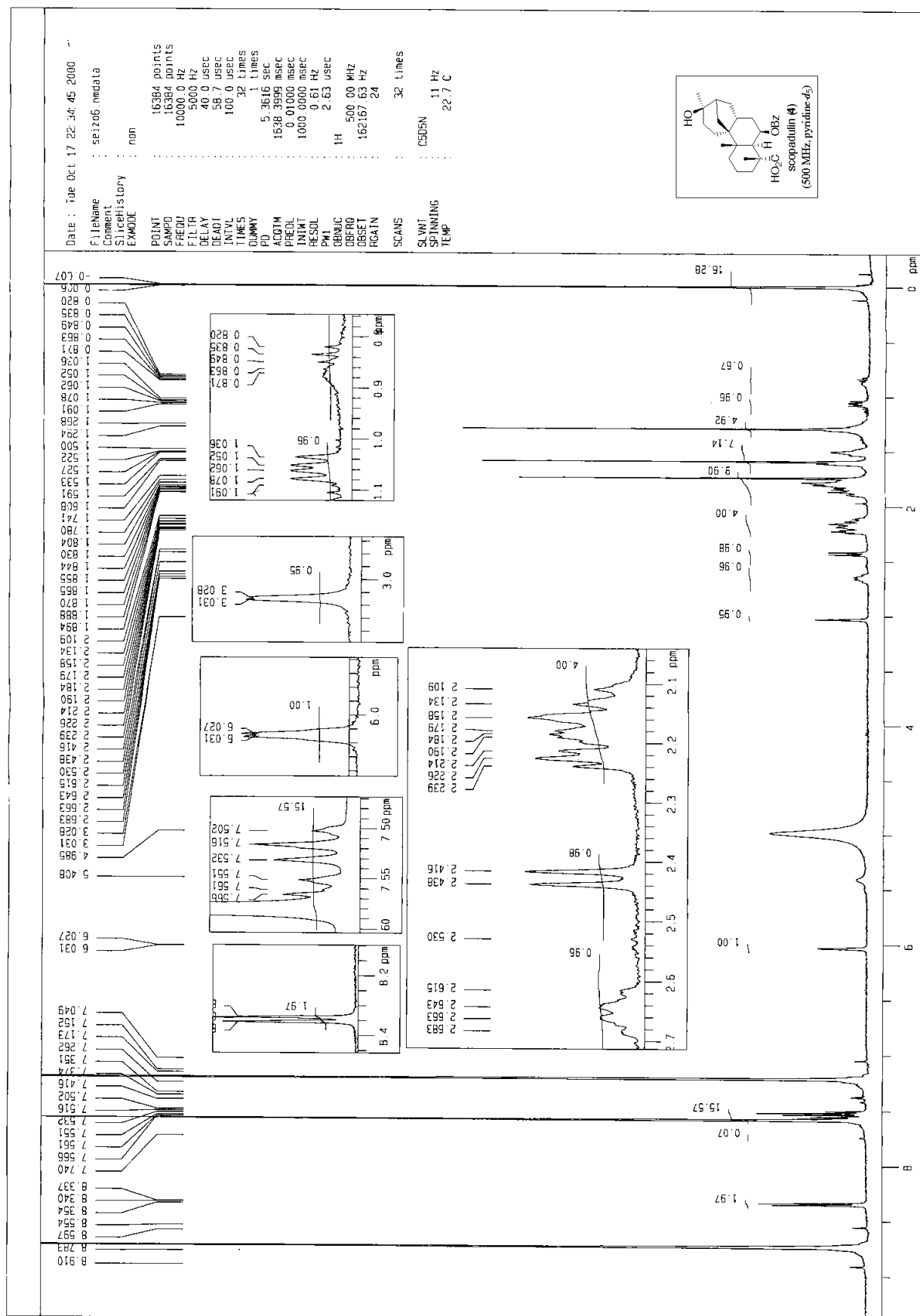
($\pm$)-(1*R*,2*R*,6*S*,7*S*,8*S*,10*S*,12*S*)-6-[(Benzyloxy)methyl]-2,6-dimethyl-13-[(2-phenylacetyl)oxy]-tetracyclo[10.3.1.0^{1,10}.0^{2,7}]hexadec-13-en-8-yl Benzoate (29). Colorless oil; IR (KBr) cm^{-1} : 1716 (br), 1601, 1273. ^1H -NMR (CDCl_3 , 500 MHz) δ : 0.97 (s, 3H), 1.22-1.42 (m, 8H), 1.58 (s, 3H), 1.68-1.90 (m, 3H), 1.98-2.14 (m, 1H), 2.17 (d, J = 2.5 Hz, 1H), 2.28-2.33 (m, 1H), 2.42 (m, 1H), 2.64-2.75 (m, 2H), 2.93 (d, J = 9.0 Hz, 1H), 3.40 (d, J = 9.0 Hz, 1H), 4.49 (d, J = 12.0 Hz, 1H), 4.58 (d, J = 12.0 Hz, 1H), 5.20 (br s, 1H), 5.65 (d, J = 2.5 Hz, 1H), 7.27-7.36 (m, 5H), 7.42-7.48 (m, 4H), 7.57-7.64 (m, 2H), 8.05-8.09 (m, 4H). MS (FAB) m/z (%): 627 (MNa^+ , 8.5), 105 (100). HRMS (FAB) Calcd for $\text{C}_{40}\text{H}_{44}\text{NaO}_5$ (MNa^+): 627.3086. Found: 627.3086.

($\pm$)-(1*R*,2*R*,6*S*,7*S*,8*S*,10*S*,12*S*)-6-[(Benzyloxy)methyl]-2,6-dimethyl-6-[(2-phenylacetyl)oxy]-methyl-13-[(2-phenylacetyl)oxy]tetracyclo[10.3.1.0^{1,10}.0^{2,7}]hexadec-13-en-8-yl Benzoate (30). Colorless oil; IR (KBr) cm^{-1} : 1724 (br), 1601, 1271. ^1H -NMR (CDCl_3 , 500 MHz) δ : 1.15 (s, 3H), 1.21-1.64 (m, 8H), 1.63 (s, 3H), 1.80-2.00 (m, 2H), 2.08 (d, J = 2.0 Hz, 1H), 2.09-2.16 (m, 1H), 2.30-2.34 (m, 2H), 2.47 (m, 1H), 2.62-2.75 (m, 2H), 4.06 (d, J = 11.5 Hz, 1H), 4.19 (d, J = 11.5 Hz, 1H), 5.20 (br s, 1H), 5.73 (br s, 1H), 7.43-7.49 (m, 6H), 7.56-7.59 (m, 3H), 8.02-8.08 (m, 6H). MS (FAB) m/z (%): 641 (MNa^+ , 22), 176 (100). HRMS (FAB) Calcd for $\text{C}_{40}\text{H}_{42}\text{NaO}_6$ (MNa^+):

641.2879. Found: 641.2875.

(±)-(1*R*,2*R*,6*S*,7*S*,8*S*,10*S*,12*S*)-6-[(Benzyloxy)methyl]-2,6-dimethyl-8-hydroxytetracyclo[10.3.1.0^{1,10}.0^{2,7}]hexadecan-13-one (32). Colorless oil: IR (KBr) cm^{-1} : 3506, 1713, 1603. ^1H -NMR (CDCl_3 , 500 MHz) δ : 1.16-1.28 (m, 4H), 1.23 (s, 3H), 1.34 (s, 3H), 1.43-1.63 (m, 4H), 1.67-1.78 (m, 4H), 1.89-1.98 (m, 3H), 2.09-2.22 (m, 2H), 2.46-2.53 (m, 1H), 2.65-2.73 (m, 2H), 2.99 (d, $J = 9.0$ Hz, 1H), 3.35 (d, $J = 9.0$ Hz, 1H), 4.25 (br s, 1H), 4.45 (d, $J = 12.0$ Hz, 1H), 4.53 (d, $J = 12.0$ Hz, 1H), 7.27-7.36 (m, 5H). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 18.6, 18.8, 21.2, 25.3, 29.7, 31.9, 34.4, 35.4, 35.9, 36.2, 36.5, 38.5, 38.8, 40.8, 44.4, 49.2, 68.8, 73.2, 80.5, 127.4 (2C), 127.5, 128.3 (2C), 138.7, 215.3. MS (FAB) m/z (%): 397 (MH^+ , 19), 91 (100). HRMS (FAB) Calcd for $\text{C}_{26}\text{H}_{37}\text{O}_3$ (MH^+): 397.2743. Found: 397.2742.

(±)-(1*R*,2*R*,6*S*,7*S*,8*S*,10*S*,12*S*)-6-[(Benzyloxy)methyl]-2,6-dimethyl-6-[(2-phenylacetyl)oxy]-methyl-8-[(2-phenylacetyl)oxy]tetracyclo[10.3.1.0^{1,10}.0^{2,7}]hexadecan-13-one (33). Coarse powder: mp 219-220 °C (*n*-hexane/EtOAc). IR (KBr) cm^{-1} : 1716 (br), 1601. ^1H -NMR (CDCl_3 , 500 MHz) δ : 1.16 (s, 3H), 1.22-1.82 (m, 7H), 1.59 (s, 3H), 1.90-2.15 (m, 8H), 2.23-2.31 (m, 1H), 2.45-2.52 (m, 1H), 2.61-2.71 (m, 2H), 4.04 (d, $J = 11.0$ Hz, 1H), 4.21 (d, $J = 11.0$ Hz, 1H), 5.72 (d, $J = 2.0$ Hz, 1H), 7.44-7.50 (m, 4H), 7.57-7.61 (m, 2H), 8.01 (d, $J = 8.5$ Hz, 2H), 8.06 (d, $J = 7.5$ Hz, 2H). ^{13}C -NMR (CDCl_3 , 125 MHz) δ : 18.3, 19.4, 20.2, 24.3, 29.7, 31.3, 32.4, 34.3, 35.7, 36.0, 36.4, 37.9, 38.6, 41.1, 43.5, 48.7, 71.1, 73.0, 128.5 (2C), 128.6 (2C), 129.5 (2C), 129.6 (3C), 130.3, 130.6, 133.1, 165.8, 166.2, 214.3. MS (FAB) m/z (%): 537 (MNa^+ , 22), 176 (100). HRMS (FAB) Calcd for $\text{C}_{33}\text{H}_{38}\text{NaO}_5$ (MNa^+): 537.2617. Found: 537.2615.

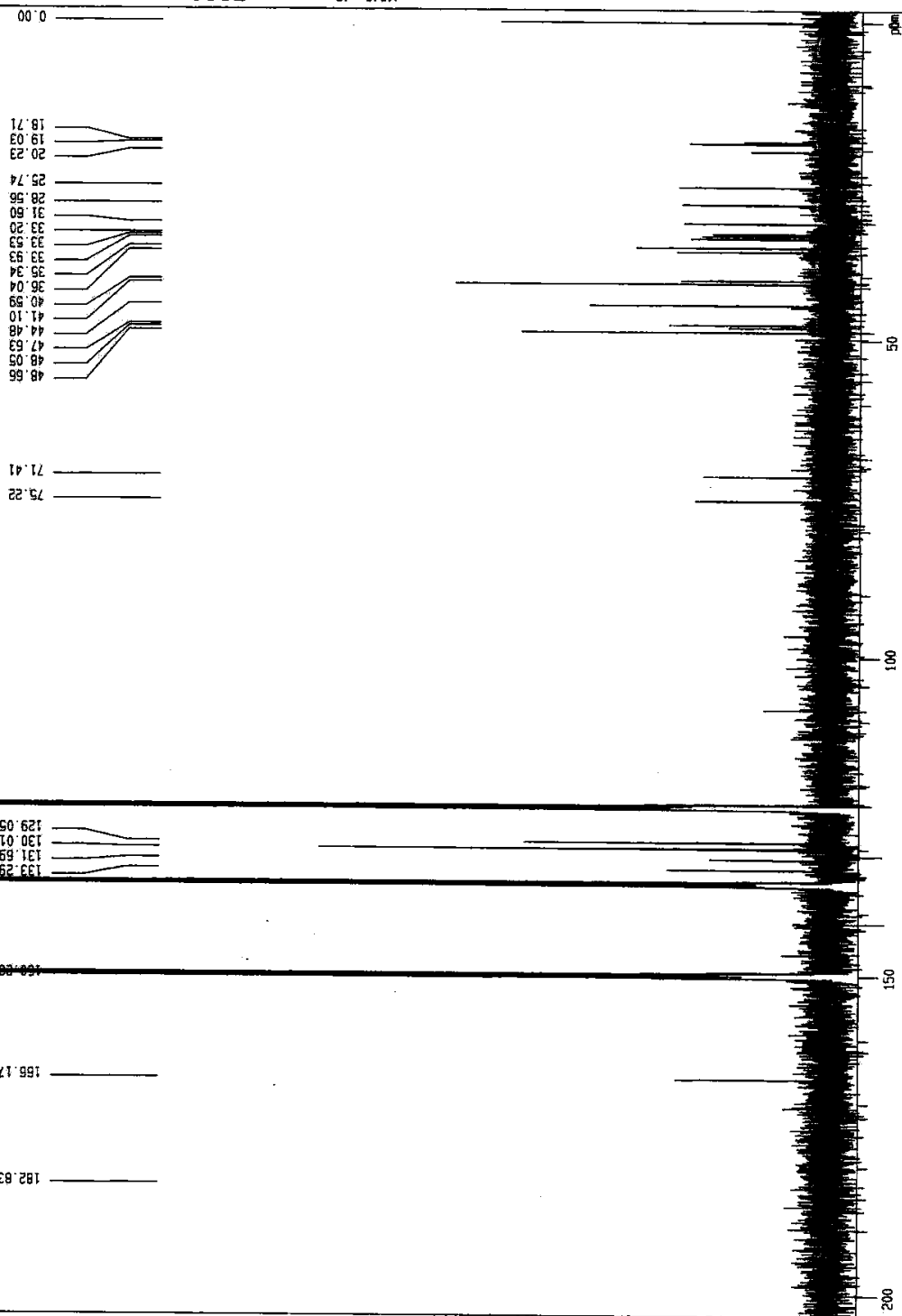
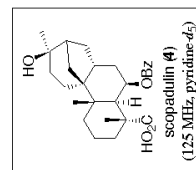


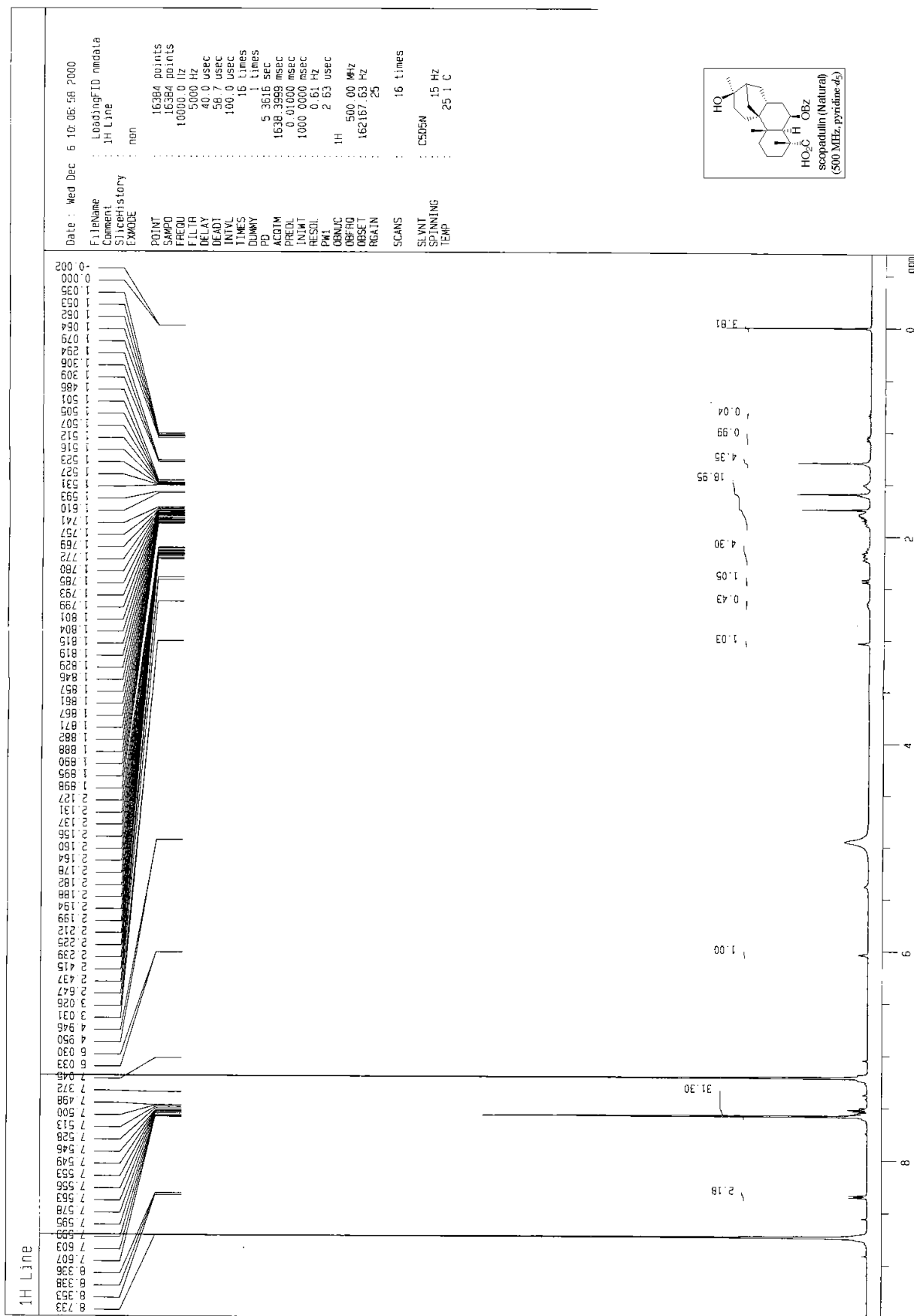
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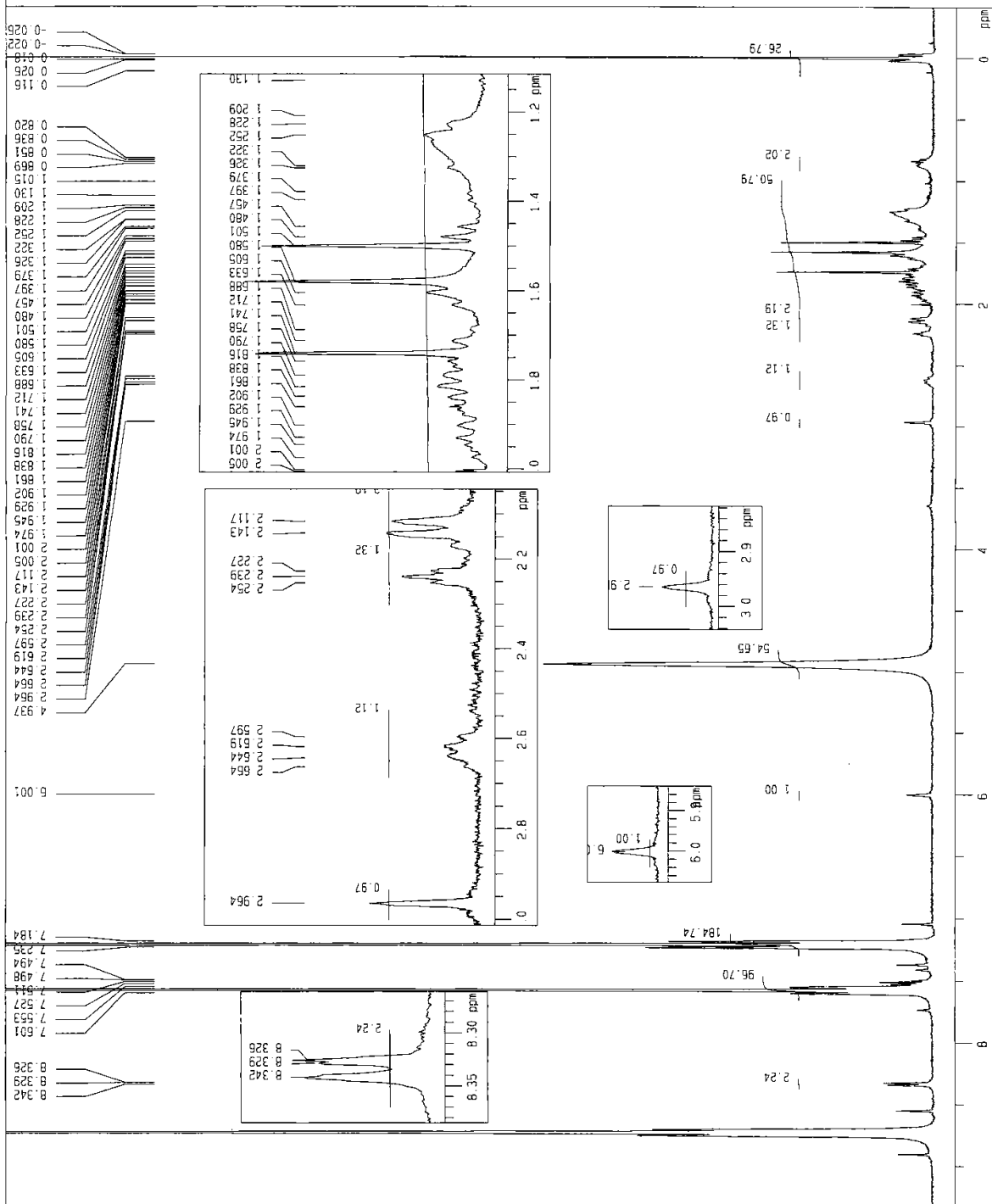
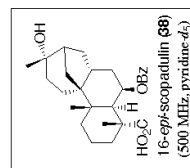
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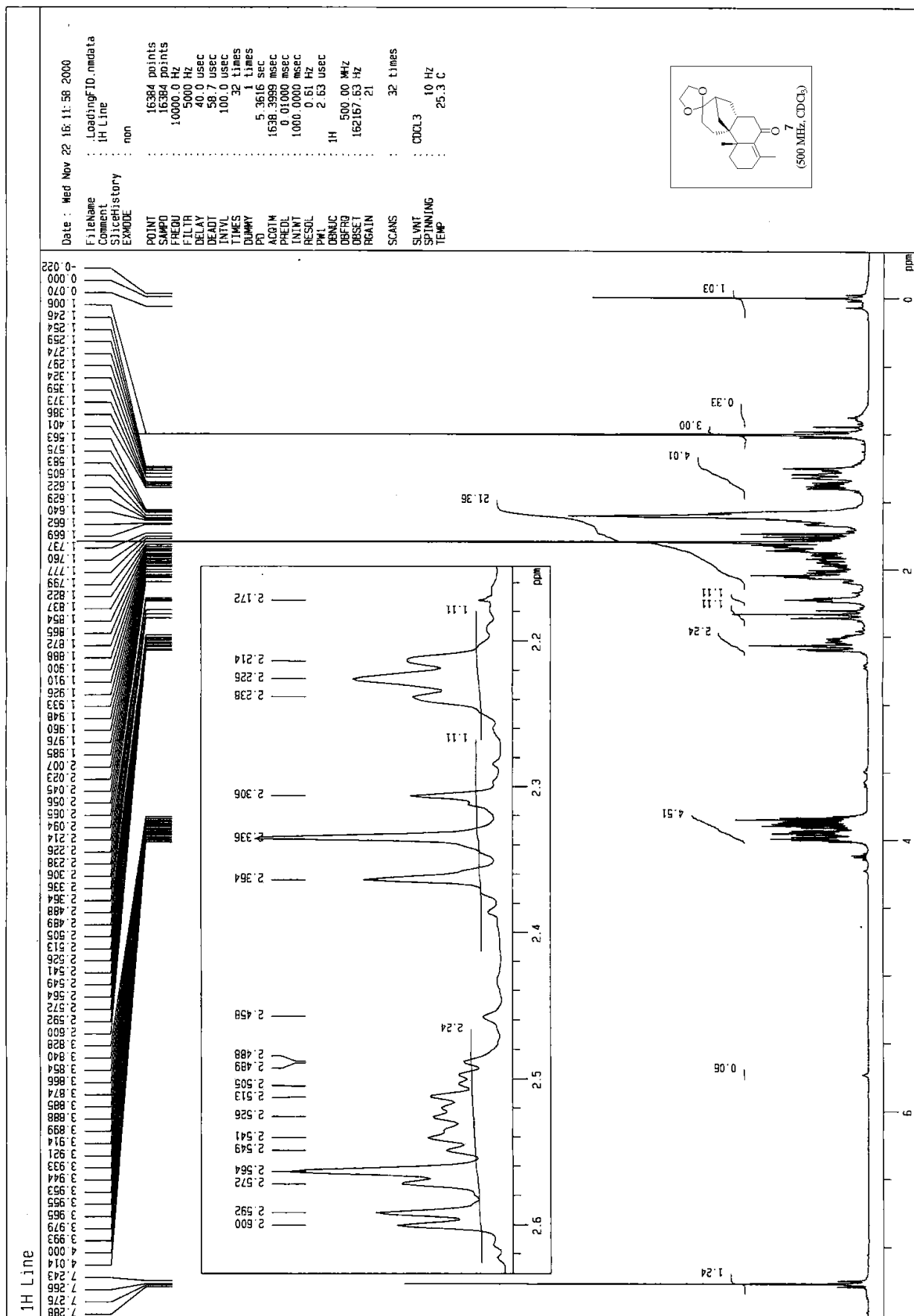




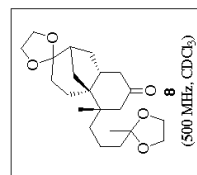
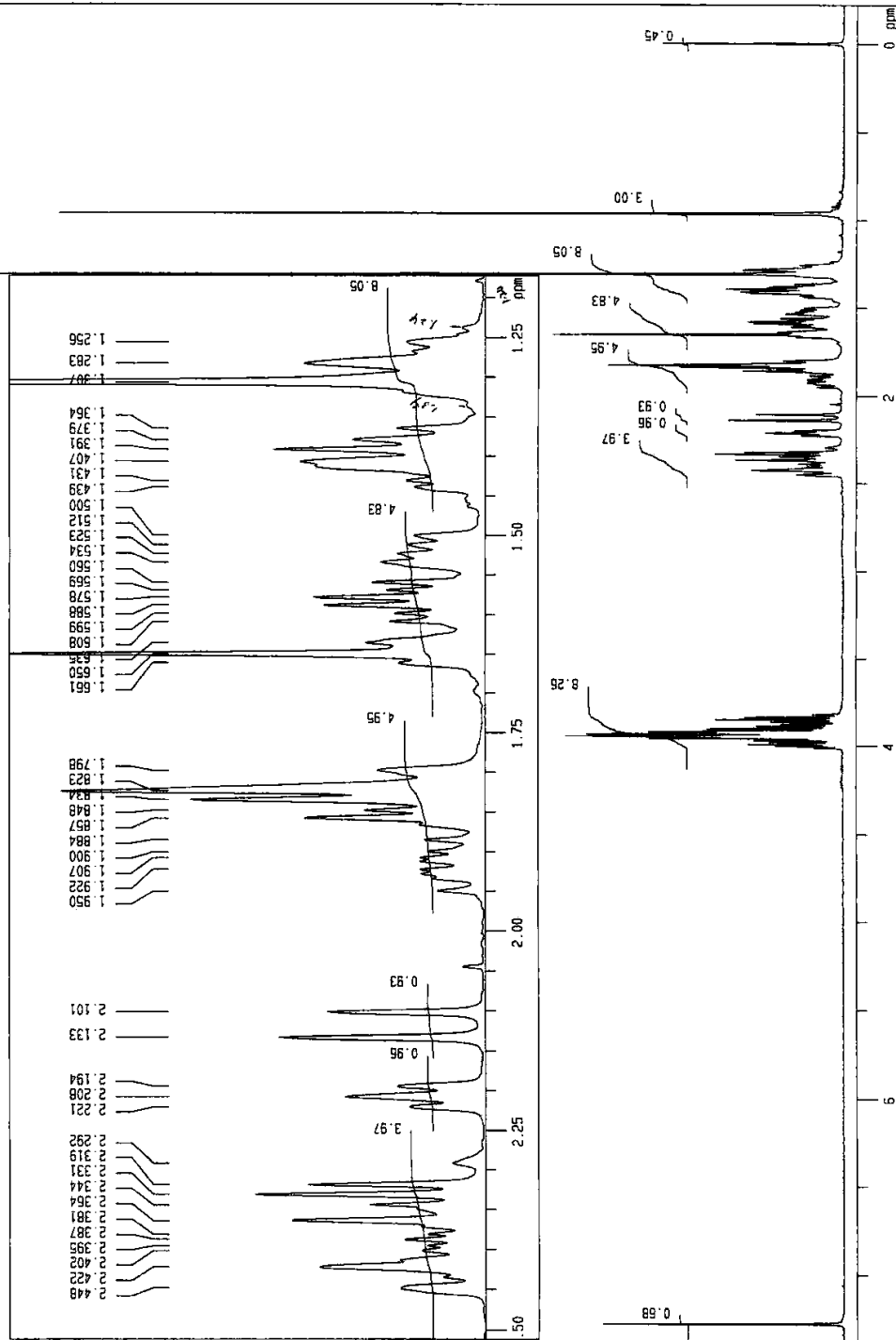
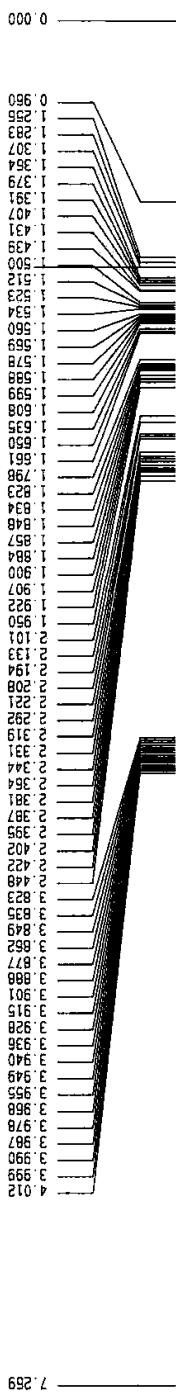
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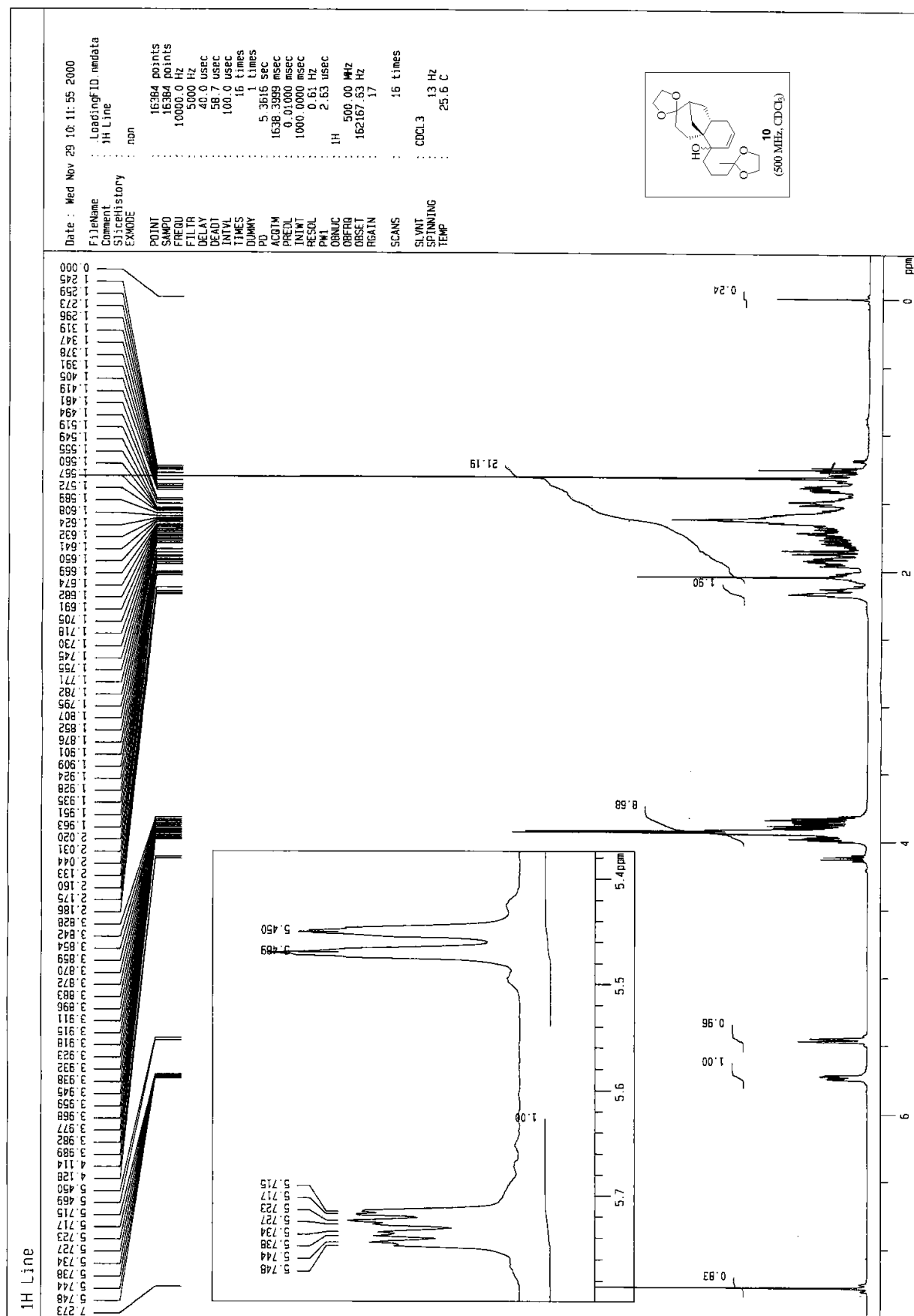




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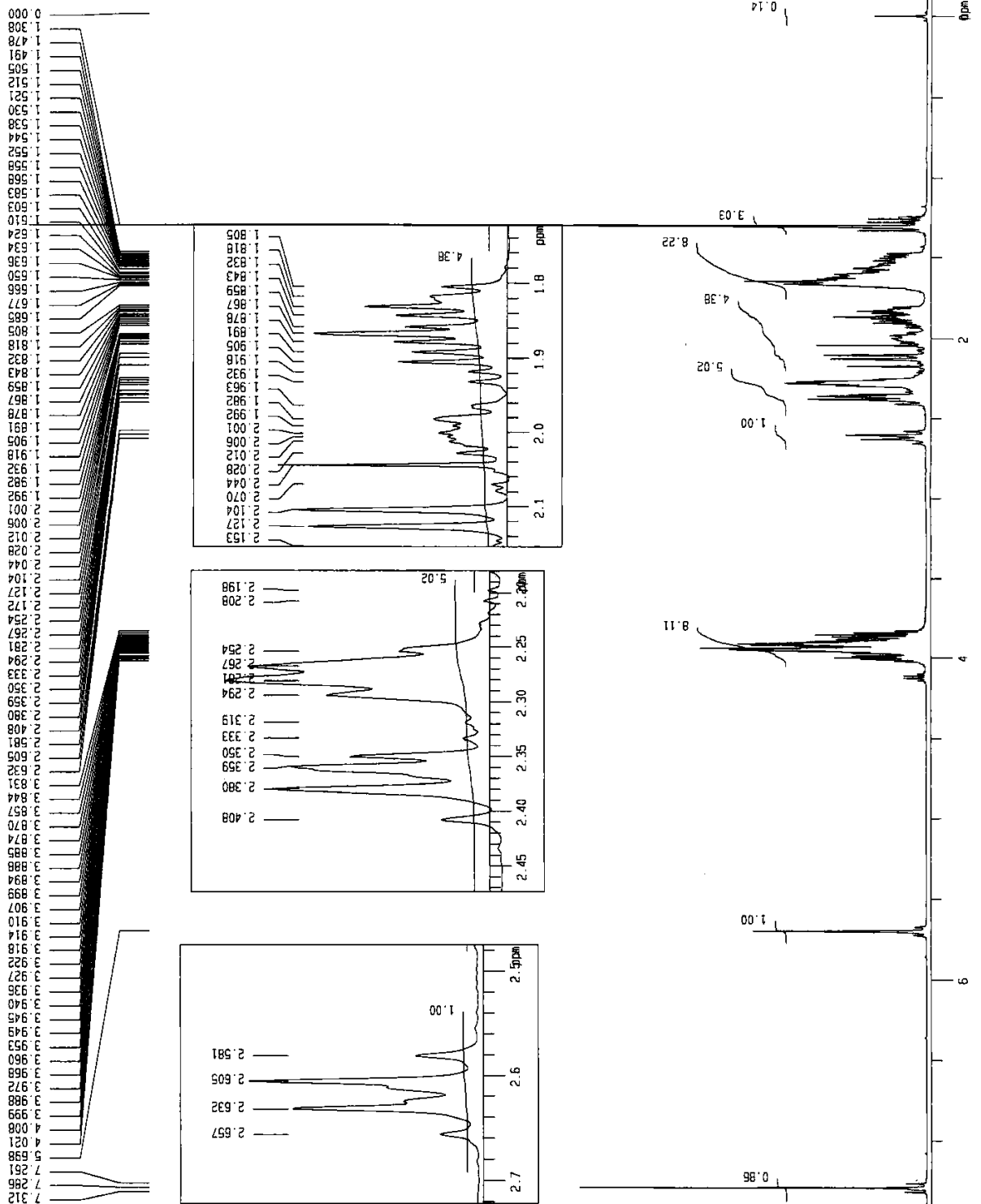
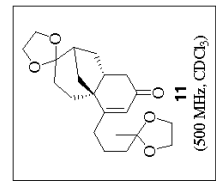


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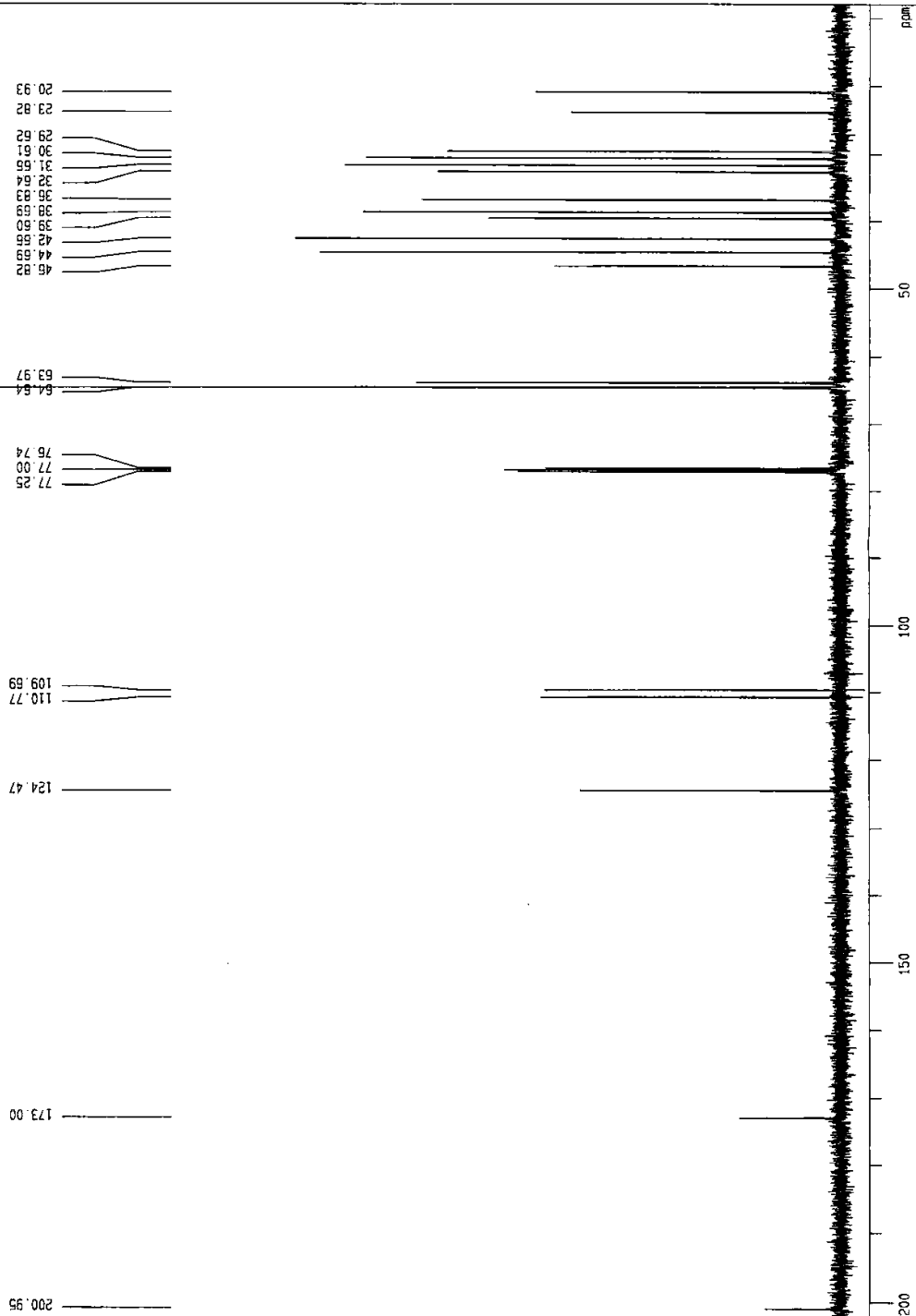
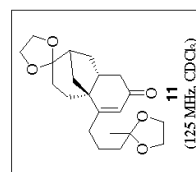


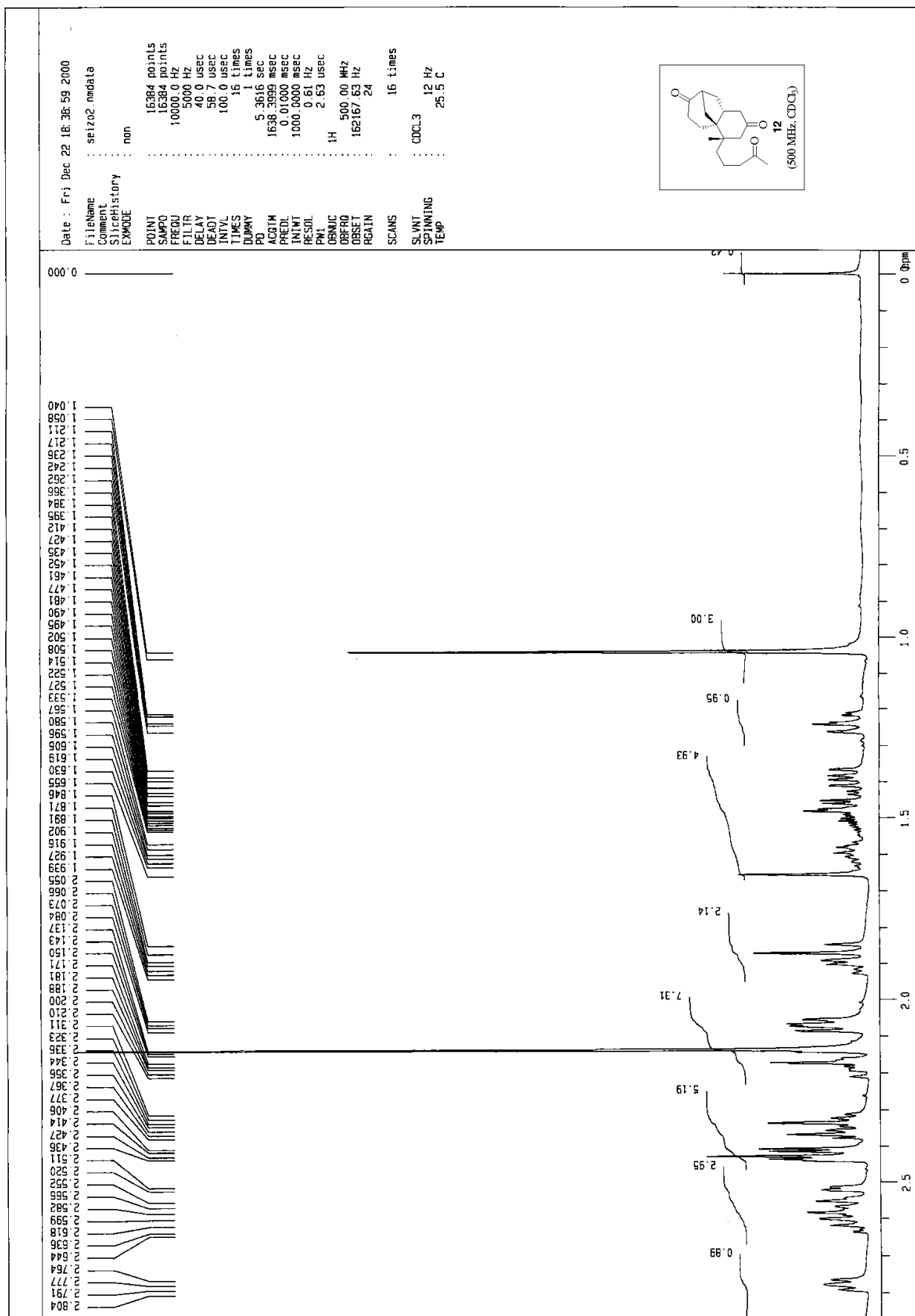
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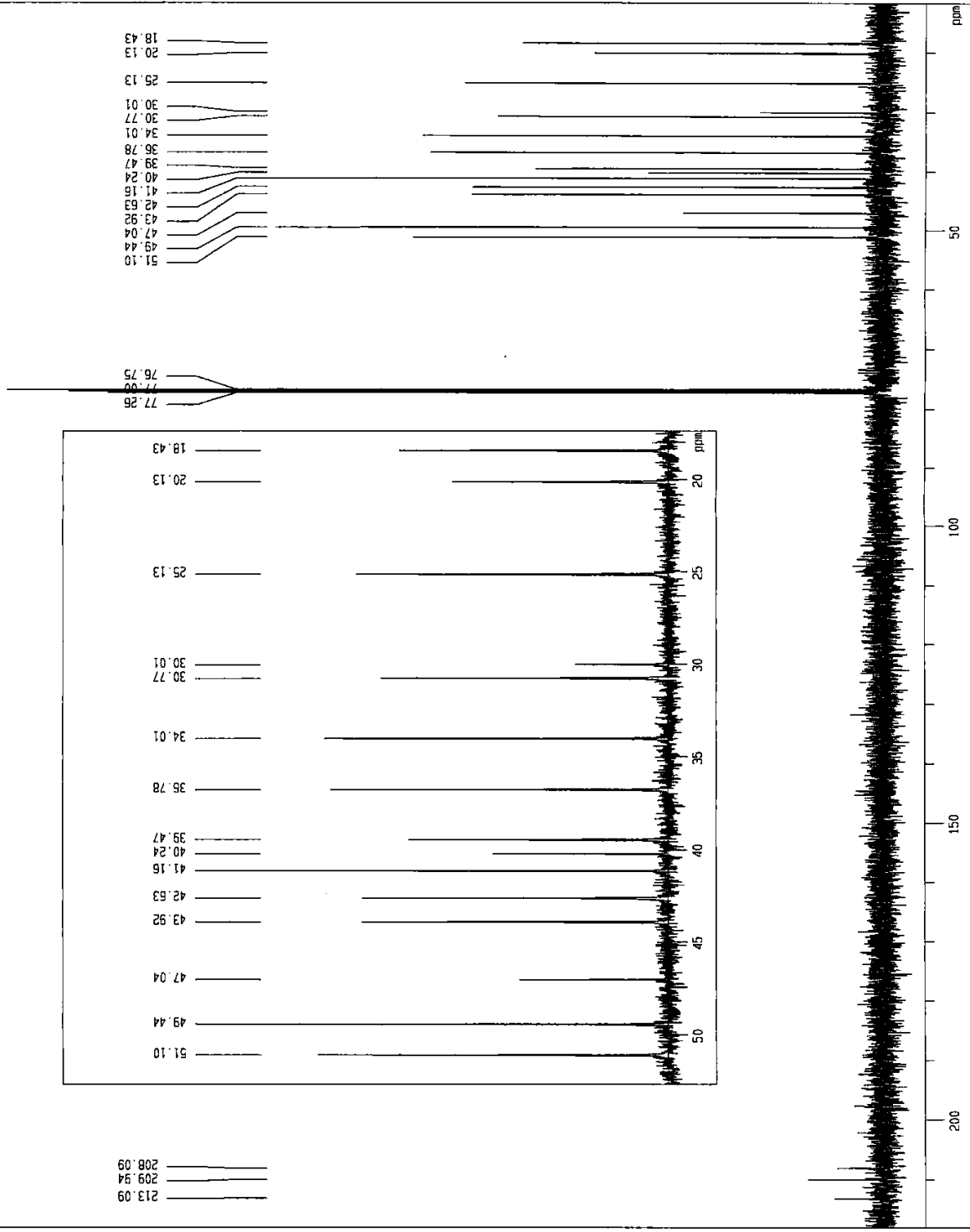
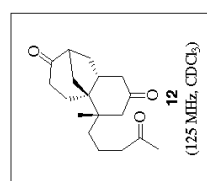
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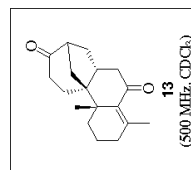
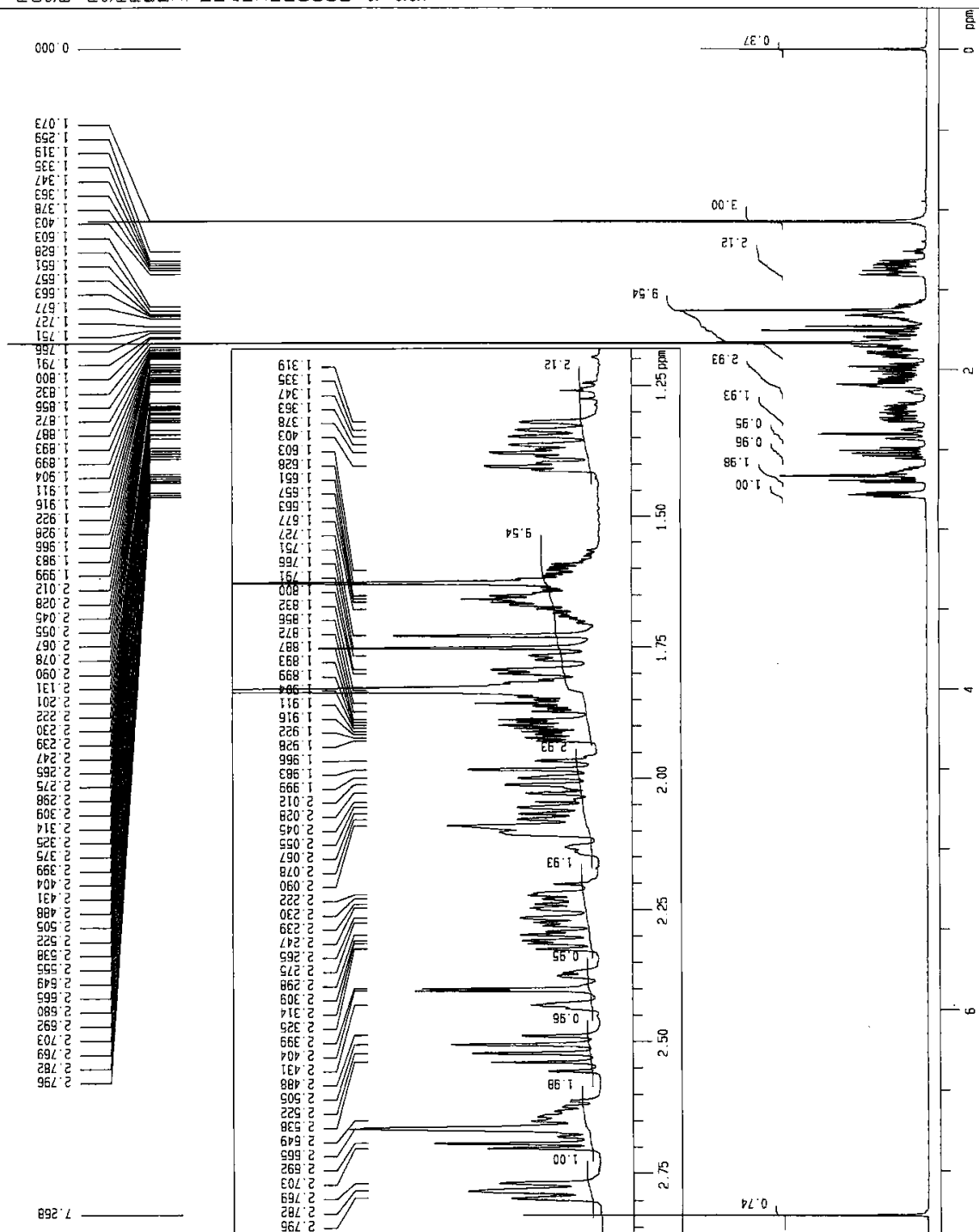


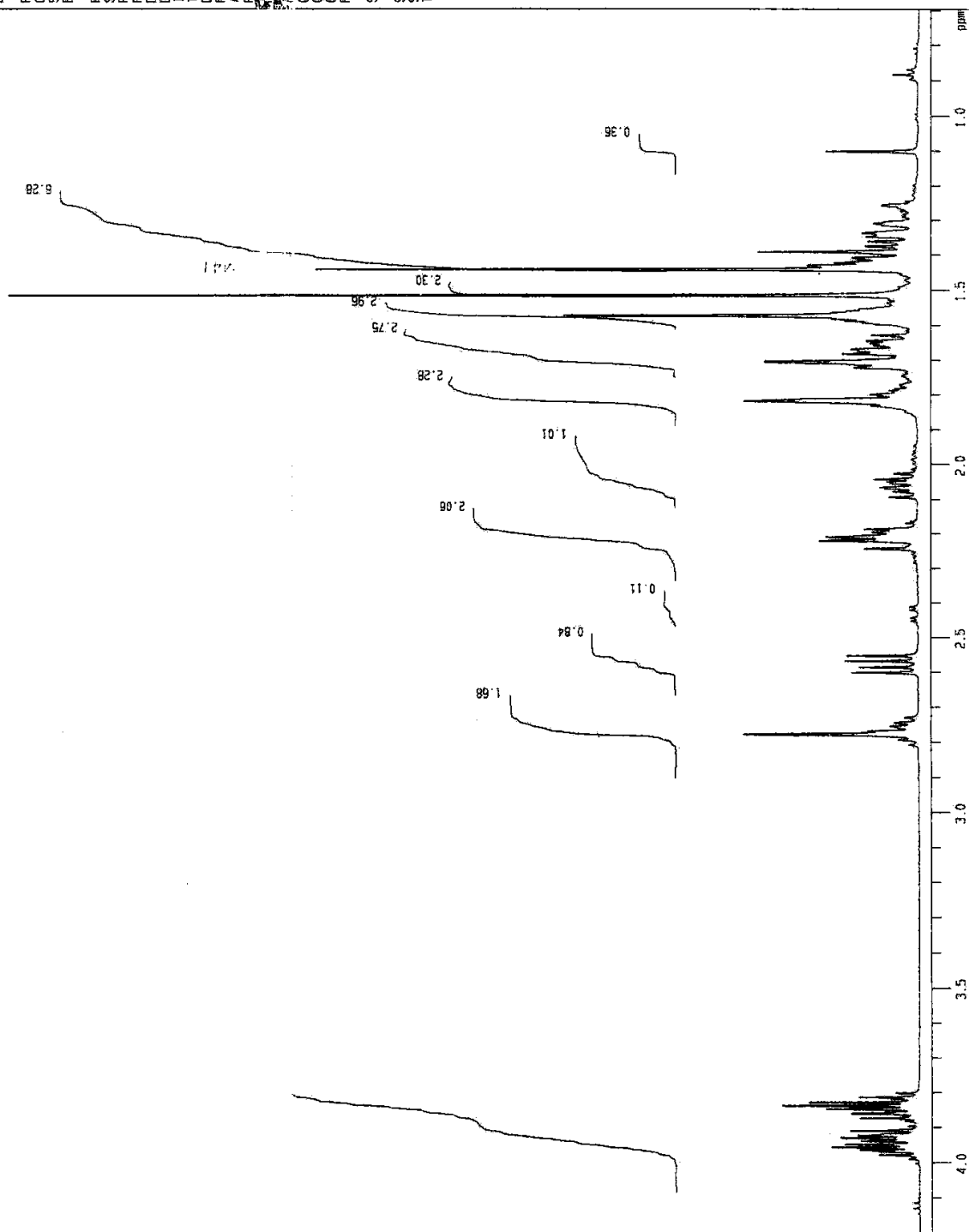
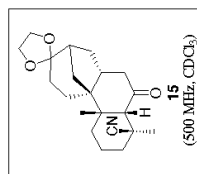
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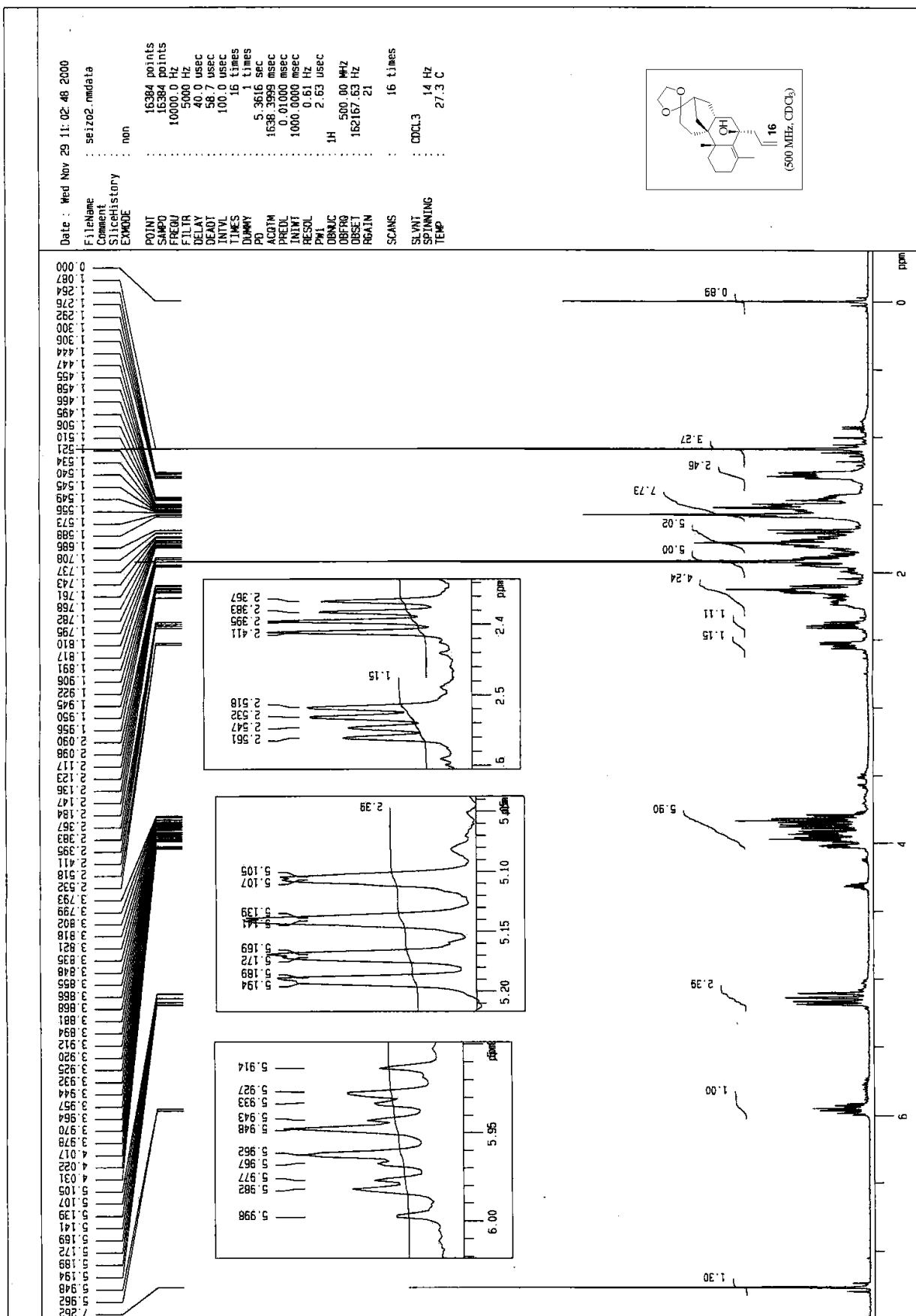
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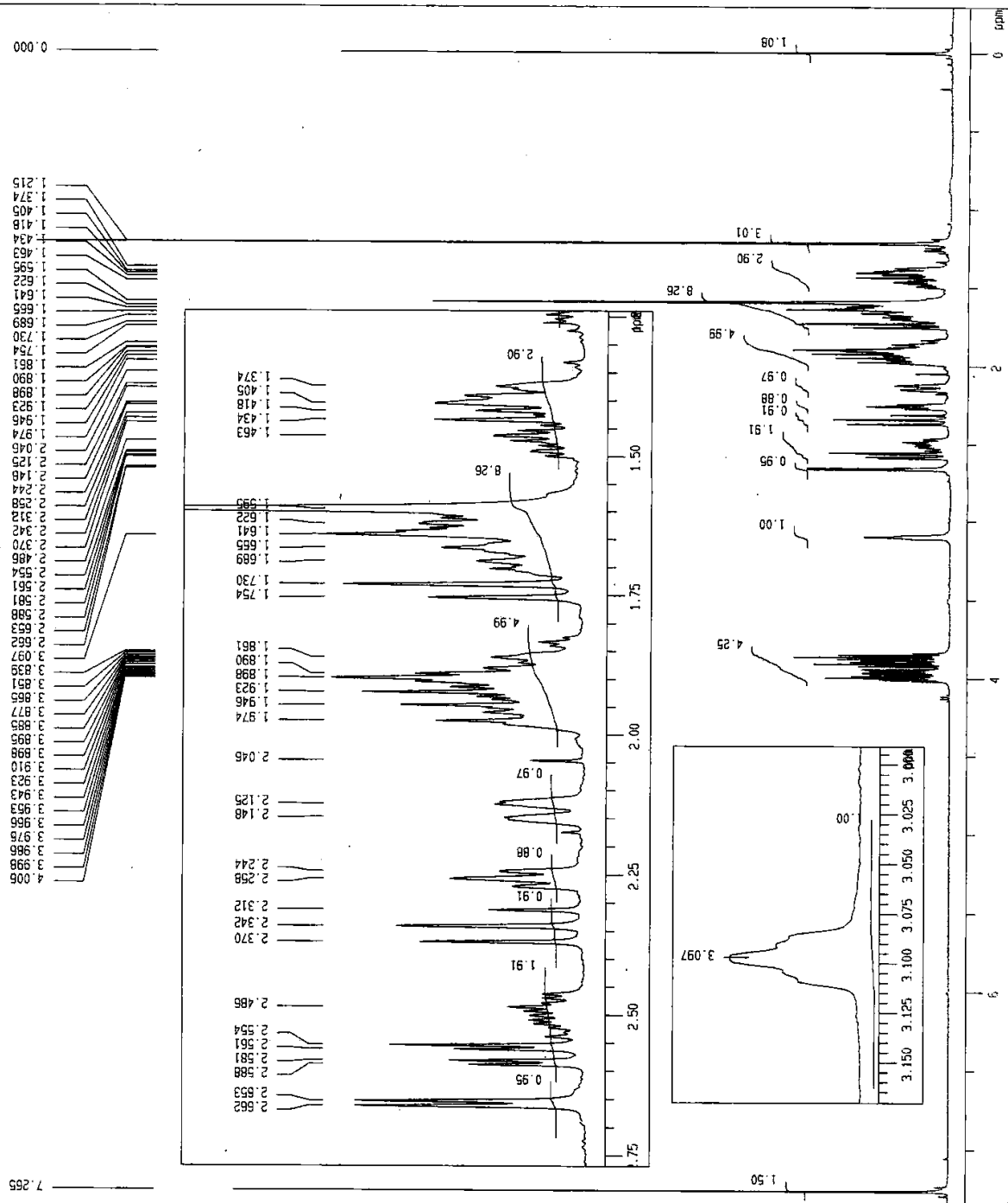
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[illegible]



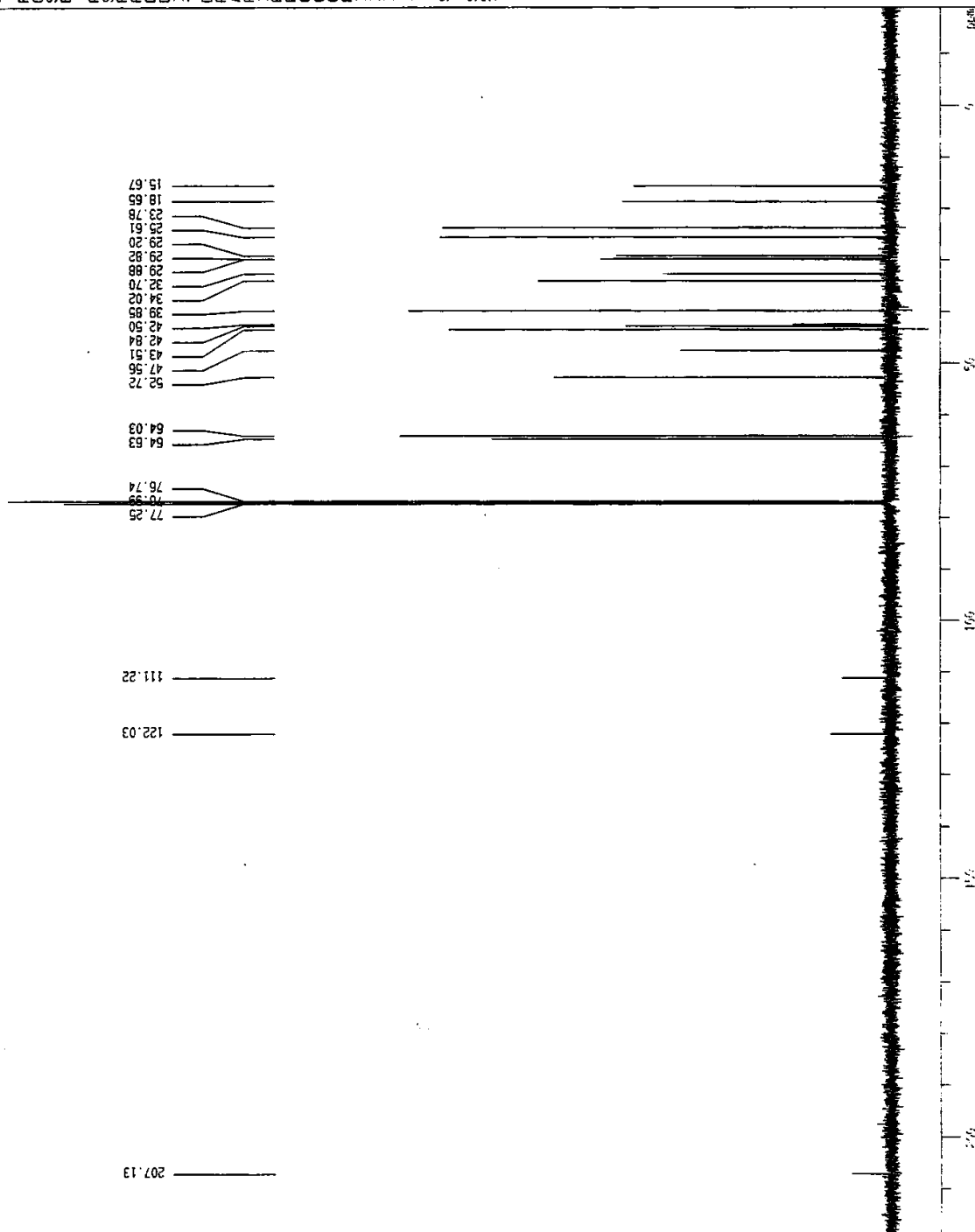
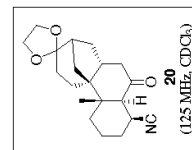
1H Line



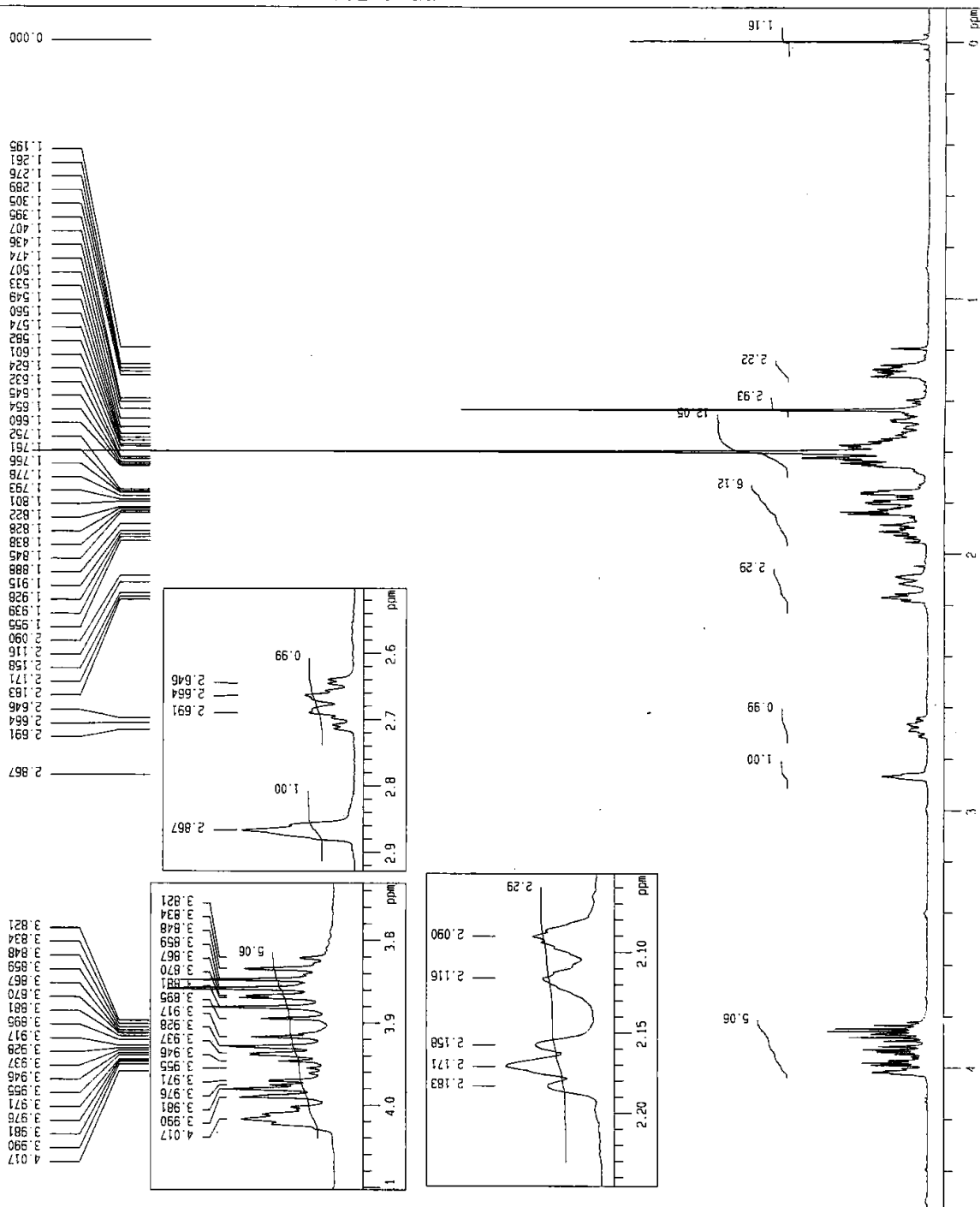
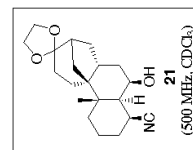
Date : Fri Jul 28 19:09:52 2000
 Filename : LoadingID.mdata
 Comment : 1H Line
 SliceHistory : non
 EXMODE :
 POINT : 16384 points
 SAMPO : 16384 points
 FREQU : 10000.0 Hz
 FILTR : 5000 Hz
 DELAY : 40.0 usec
 DEADT : 58.7 usec
 INTVL : 100.0 usec
 TIMES : 32 times
 DUMMY : 1 times
 PD : 5.3616 sec
 ACQTM : 1638.3999 msec
 PREDL : 0.01000 msec
 ININT : 1000.0000 msec
 RESOL : 0.61 Hz
 PM1 : 2.63 usec
 ORBIC : 500.00 MHz
 ORFIC : 162167.63 Hz
 OFFSET : 22
 REATN :
 SCANS : 32 times
 SLVNT : CDCL3
 SPINNT : 11 Hz
 TEMPR : 23.8 C

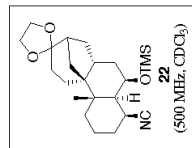
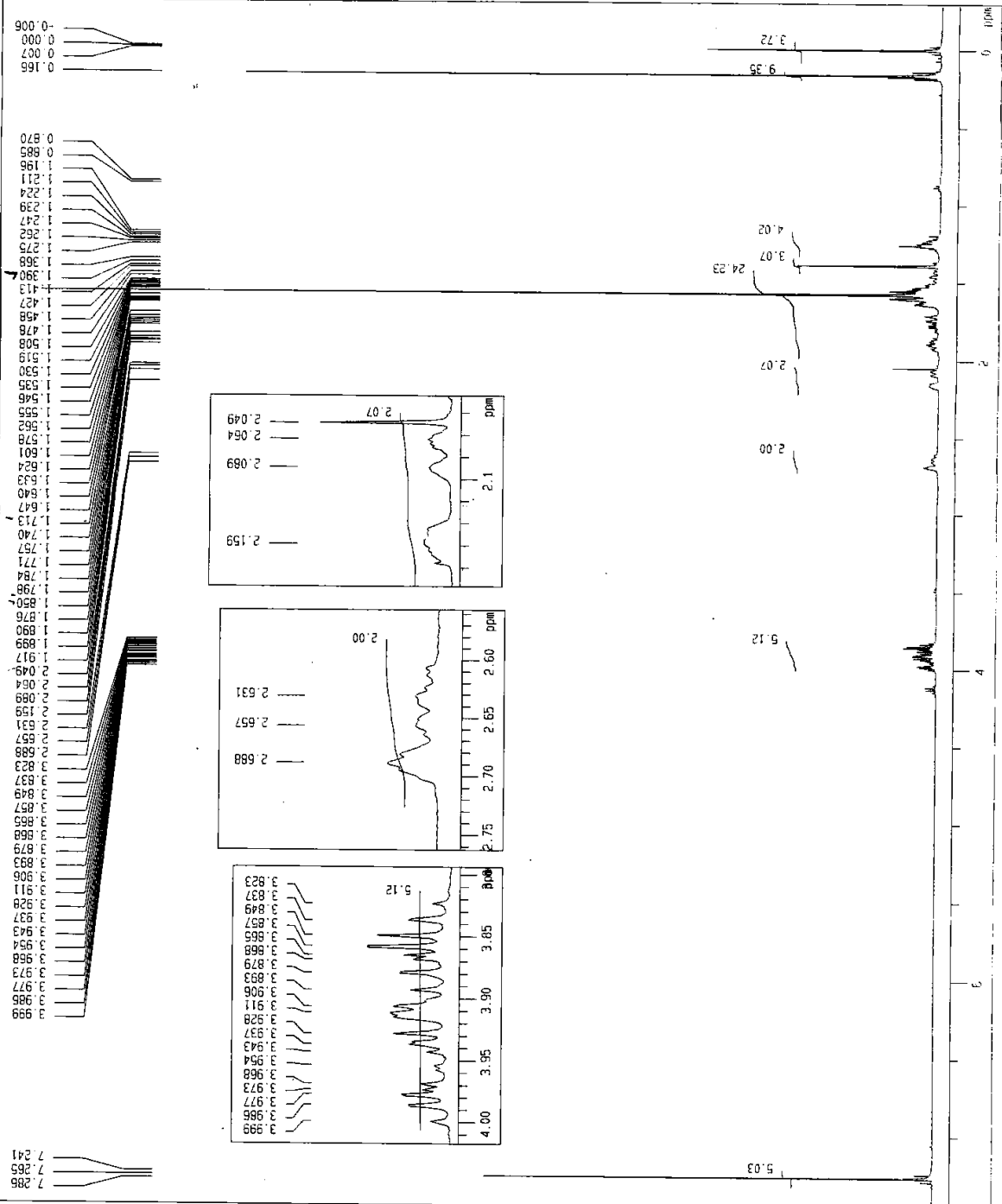
1H-Line

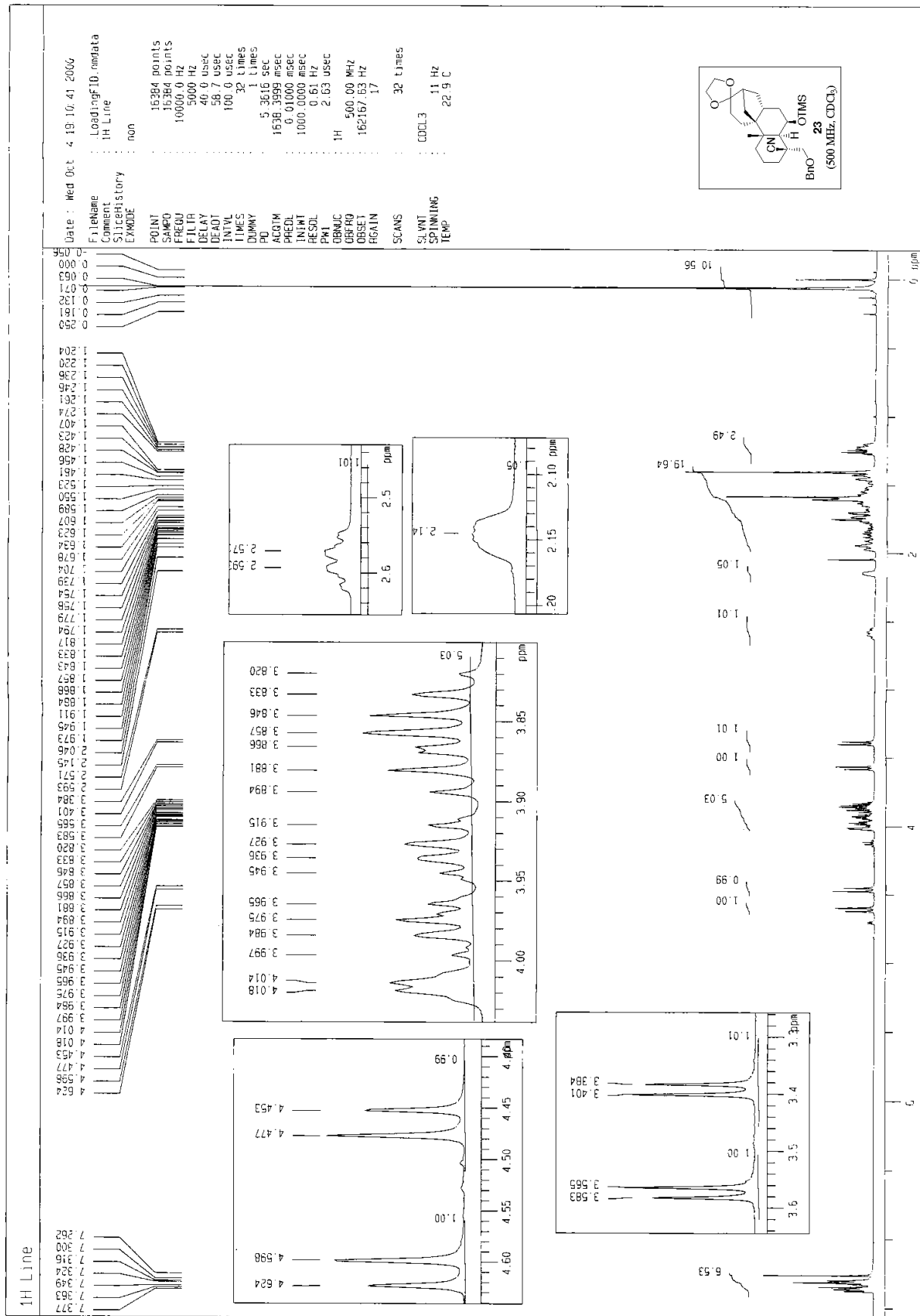
Date : Fri Nov 12 21:08:42 1999
 File Name : LoadingFID.rawdata
 Comment : 1H Line
 SliceHistory : bcm
 EXMODE :
 POINT : 16384 points
 SAMP0 : 16384 points
 FREQ0 : 30030.0 Hz
 FILTR : 15000 Hz
 DELAY : 13.3 usec
 DEAT0 : 17.5 usec
 INTVL : 33.3 usec
 TIMES : 1200 times
 DUMMY : 1 times
 P0 : 2.4544 sec
 ACQTH : 545.5872 msec
 PREDL : 0.01000 msec
 INTMT : 1000.0000 msec
 RESOL : 1.83 Hz
 PM1 : 5.10 usec
 13C : 125.65 MHz
 127958.00 Hz
 1H : 500.00 MHz
 162160.00 Hz
 162160.00 Hz
 46.0 usec
 0
 SCANS : 1200 times
 SLVNT : CDCL3
 SPINNING : 13 Hz
 TEMP : 27.7 C



Date	Fri Aug 4 17:43:53 2000
Filename	.LoadingID.rmdata
Comment	1H Line
Spectral history	
EXMODE	non
POINT	16384 points
SAMP	16384 points
FREQ	10000.0 Hz
FLTR	5000 Hz
DELT	40.0 usec
DEAD	58.7 usec
INVTWL	100.0 usec
TIMES	32 times
DUMMY	1 times
PULPROG	zgpg30 sec
ACQTM	1638.3999 msec
PREDL	0.01000 msec
INVTM	1000.0000 msec
PREOL	0.61 Hz
PW1	2.63 usec
GBOINC	
ORFPG	500.00 MHz
ORBSFT	162167.63 Hz
RGAIN	22
SCANS	32 times
SVMT	CDC13
SPRINTING	
TEMP	14 Hz 22.9 C

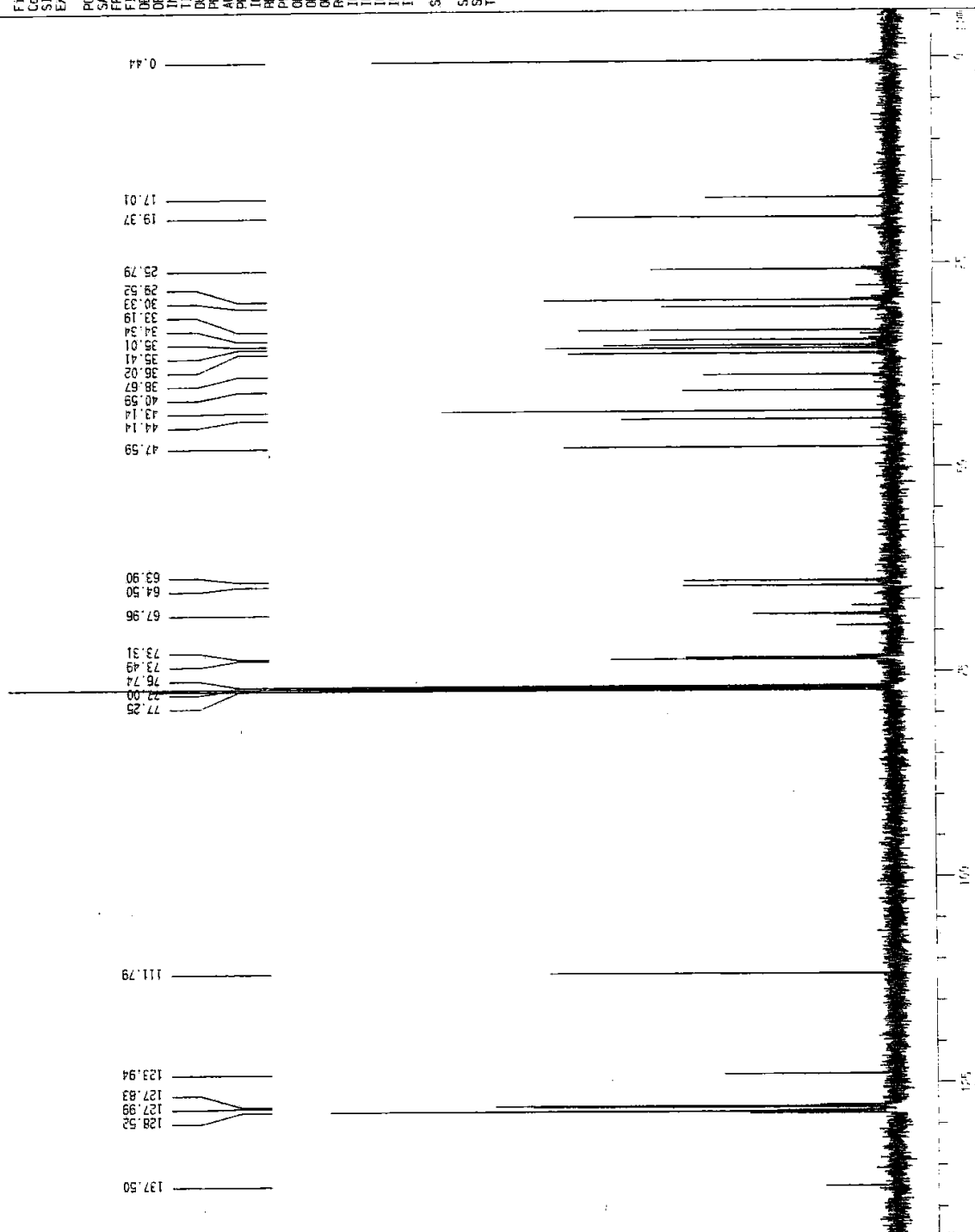
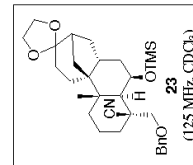


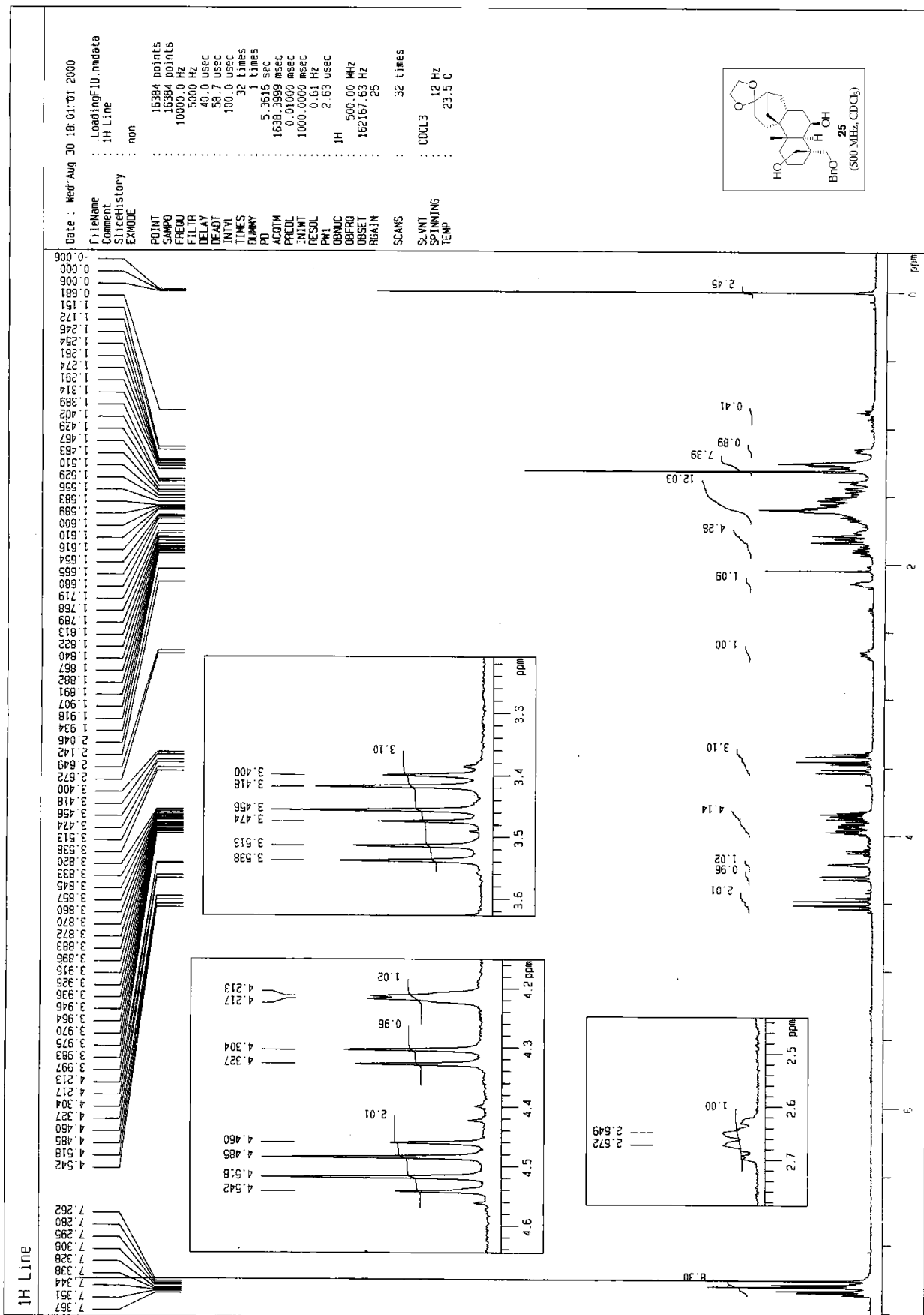


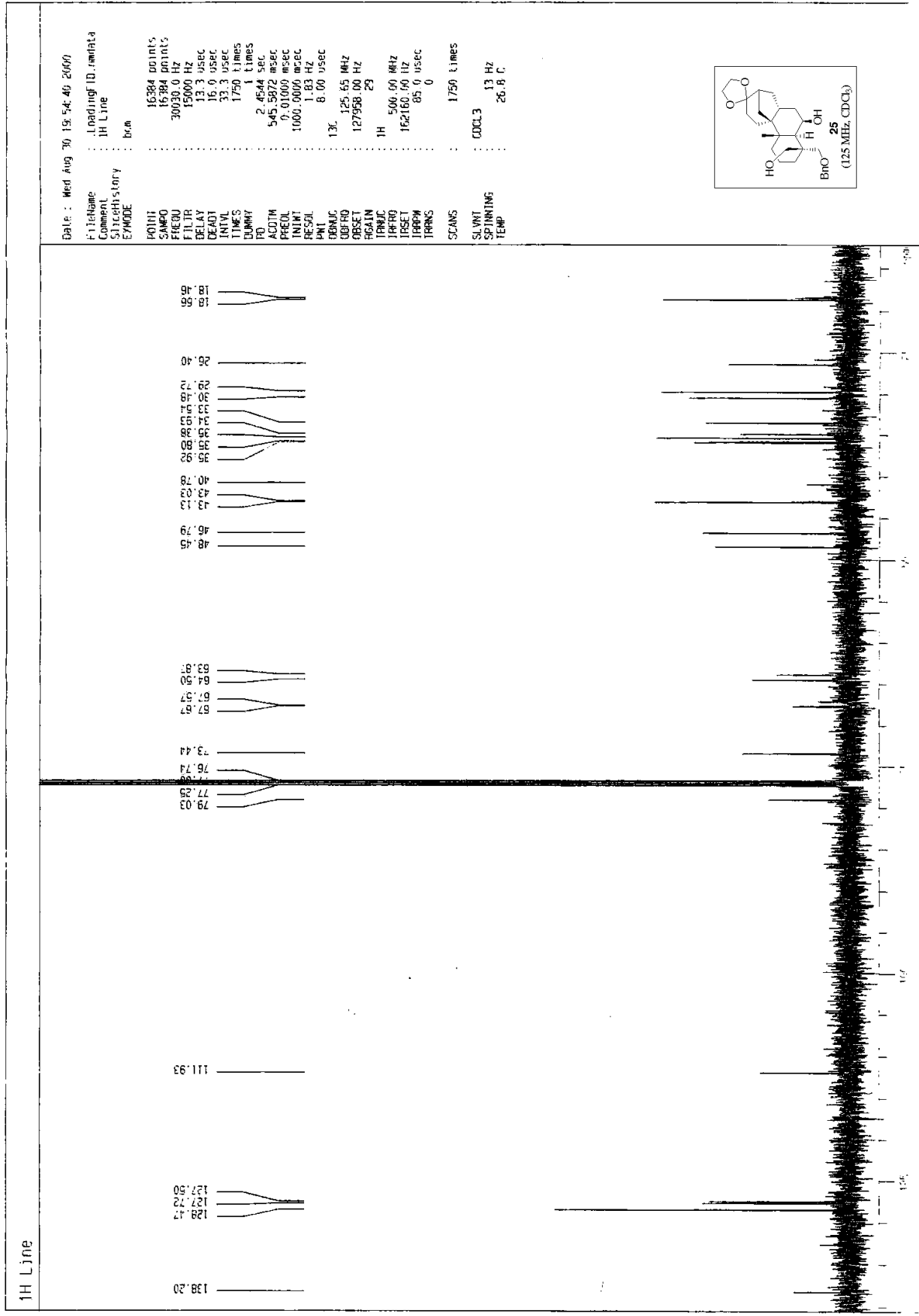


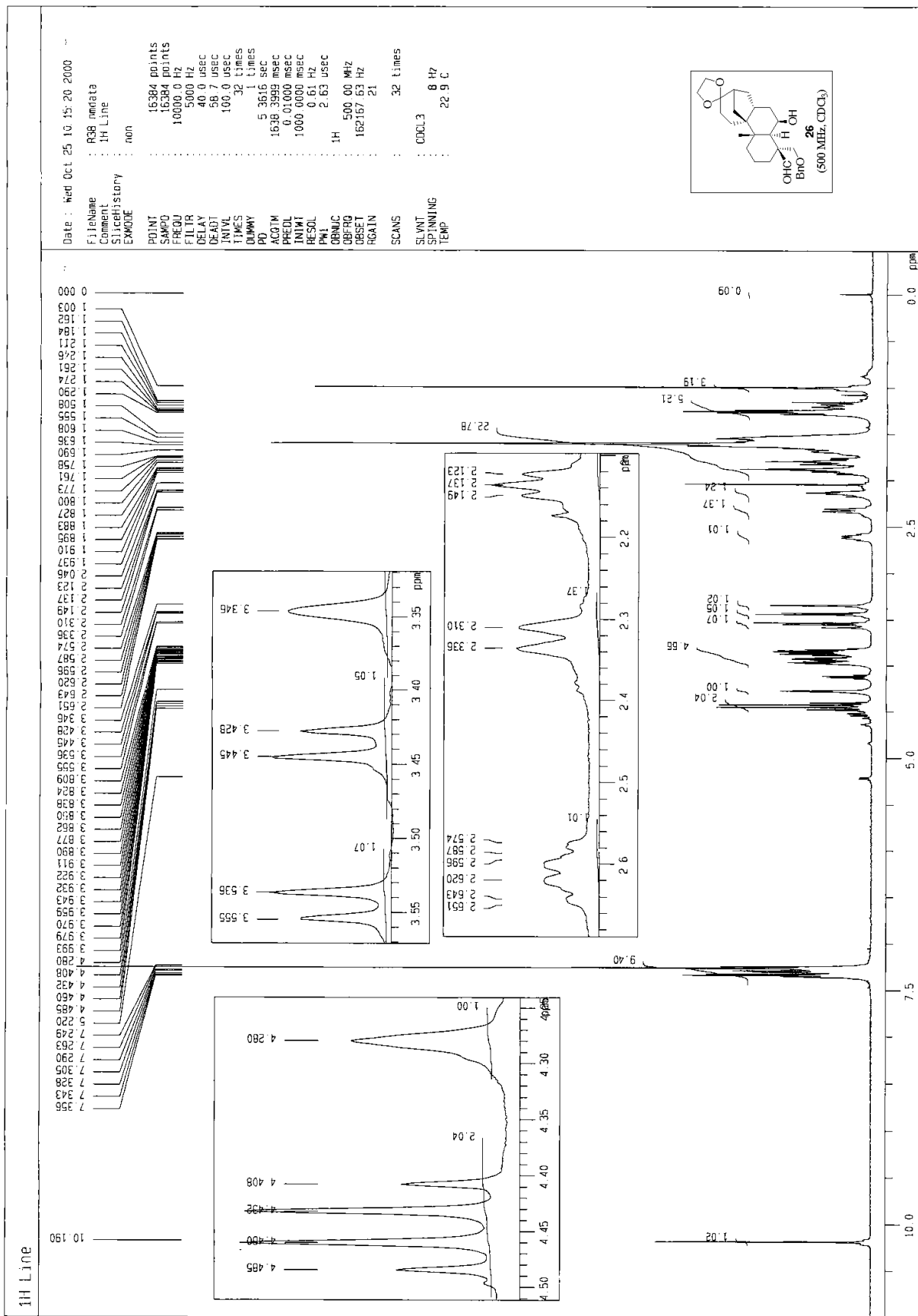
1H Line

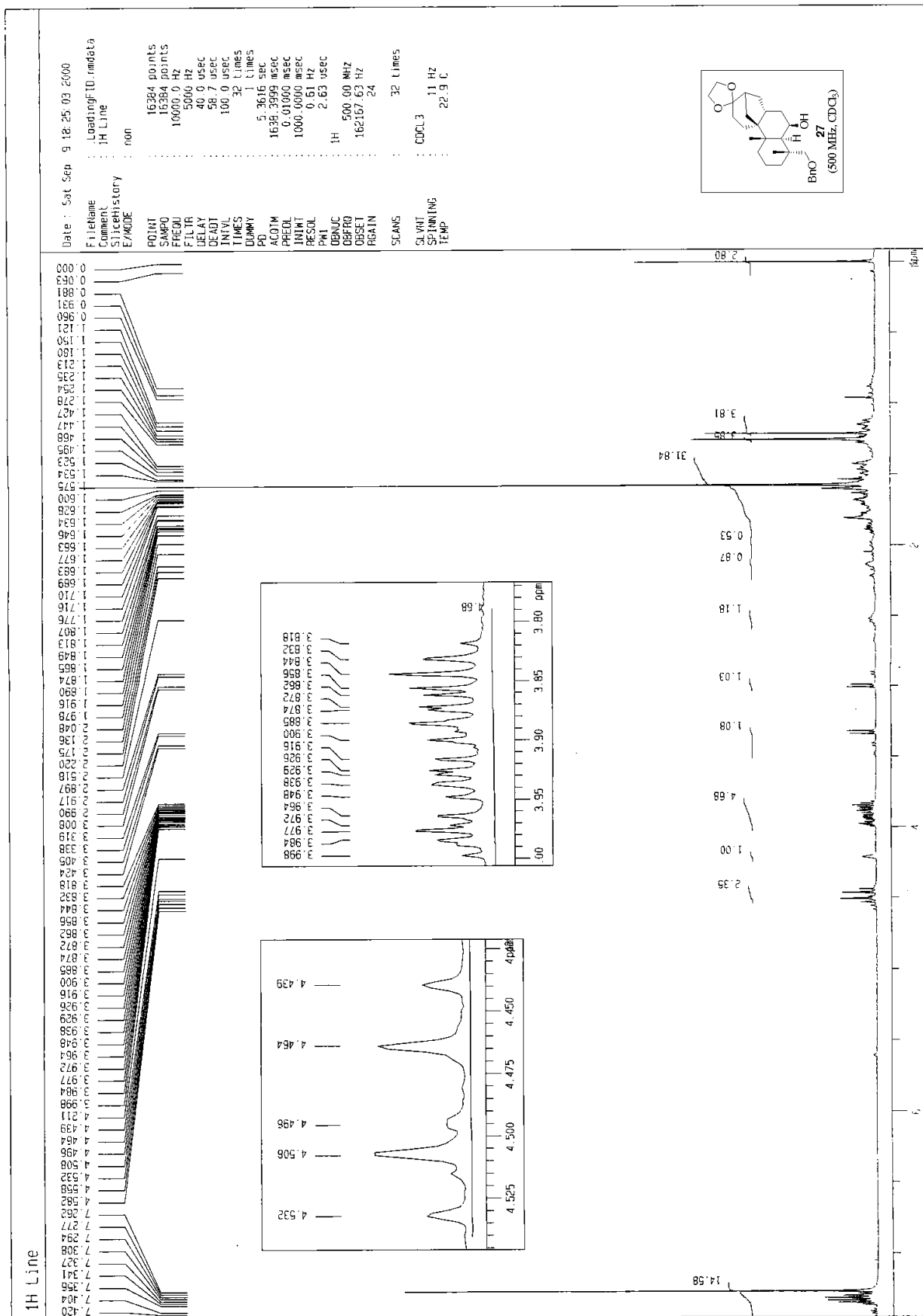
Date : Mon Sep 4 13:17:10 2000
 File Name : LoadingFID.nmdata
 Comment : 1H Line
 Slice History :
 E2MODE : tcm
 POINT : 16384 points
 SAMPLE : 16384 points
 FREQ : 30030.0 Hz
 FIDR : 15000 Hz
 DELAY : 13.3 usec
 DEAT : 16.0 usec
 INVT : 33.3 usec
 TIMES : 1500 times
 DUMMY : 1 times
 PD : 2.4544 sec
 ACQIM : 545.5872 msec
 PRECL : 0.01000 msec
 INVT : 1000.0000 msec
 RESOL : 1.83 Hz
 PWT : 8.00 usec
 PC1 : 13C
 CPDNC : 125.65 MHz
 CPDPO : 127958.00 Hz
 CPDSE : 28
 CPDIN : 1H
 CPDPO : 500.00 MHz
 CPDSE : 162160.00 Hz
 CPDIN : 65.0 usec
 CPDIN : 0
 SCANS : 1500 times
 SUNIT : CDOL3
 SPINNING : 10 Hz
 TEMP : 27.2 C





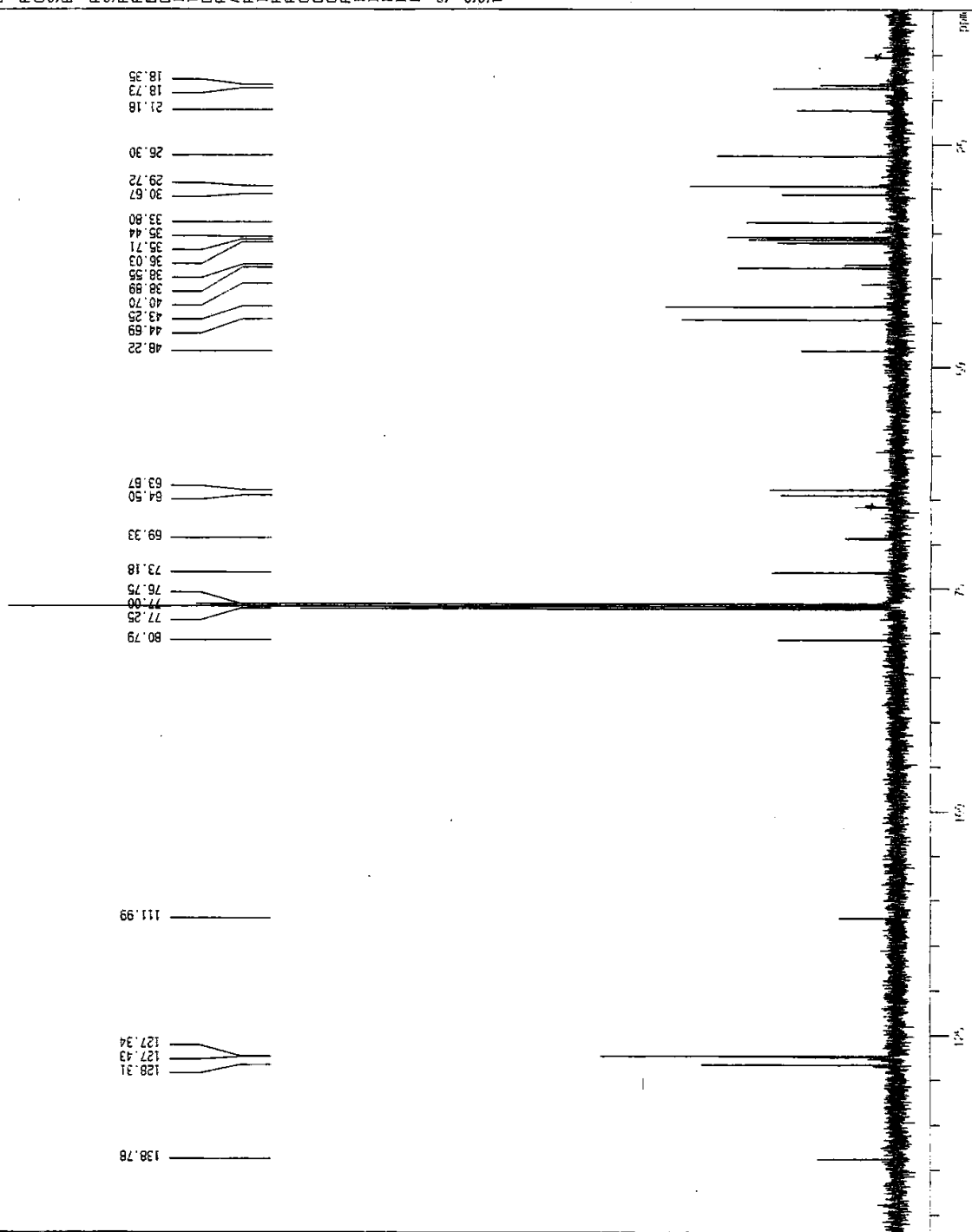
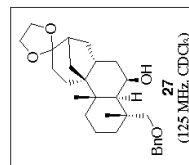






1H Line

Date : Wed Sep 6 19:53:28 2000
 FileName : LoadingFID.mdata
 Comment : 1H Line
 SliceHistory :
 E:MODE : bcm
 POINT : 16384 points
 SAMPL : 16384 points
 FREQ : 300.00 MHz
 F1F2 : 15000.0 Hz
 DELAY : 12.3 usec
 DEADT : 12.3 usec
 T1RHO : 33.3 usec
 T1RHO : 1300 times
 DUMPT : 2.4644 sec
 ACQTM : 545.5872 msec
 PRECL : 0.01000 msec
 TNUIT : 1000.0000 msec
 RESOL : 1.83 Hz
 P1 : 8.00 usec
 13C : 125.65 MHz
 OBSFQ : 127956.00 Hz
 RGAIN : 28
 1H : 500.00 MHz
 PREPQ : 162160.00 Hz
 INPRW : 85.0 usec
 INPRN : 0
 SCANS : 1500 times
 SLWT :
 SPINNING : 11 Hz
 TEMP : 27.0 C



1H Line

