Supporting Information

A highly efficient, rapid one-pot synthesis of some new heteroarylpyrano[2,3-*c*]pyrazoles in ionic liquid under microwave-irradiation

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1.1 General methods

All solvents and reagents were used as supplied from commercial sources. The recorded melting points are uncorrected. IR spectra were recorded in KBr on Shimadzu FT-IR 8401 spectrometer and are reported in wave numbers (cm^{-1}). All microwave heating experiments were performed in a closed system, on Catalyst Scientific Microwave Oven model CATA-R (2.45 GHz, 140 to 700 Watts) from Catalyst^{TM} Systems, Pune, India. ^{1}H NMR and ^{13}C NMR spectra were recorded on a Bruker Avance 400 spectrometer operating at 400 MHz for ^{1}H NMR and 100 MHz for ^{13}C NMR in $CDCl_3$ as both solvent and reference. Chemical shifts are expressed in parts per million (ppm, δ) and referenced to the residual protic solvent. Coupling constants are reported in Hertz (Hz). Splitting patterns are designated as s, for singlet; d, for doublet; t, for triplet; q, for quartet; br, for broad; m, for multiplet; comp, for complex multiplet. Degree of substitution (C, CH, CH_2, and CH_3) was determined by the DEPT-135 method. The ESI mass spectra were taken on Shimadzu LCMS-2010 spectrometer. Elemental analyses (% C, H, N) were carried out by Perkin-Elmer 2400 series-II elemental analyzer (Perkin-Elmer, USA). TLC was performed on Merck 60 F254 precoated silica plates, spots were detected under UV lamp

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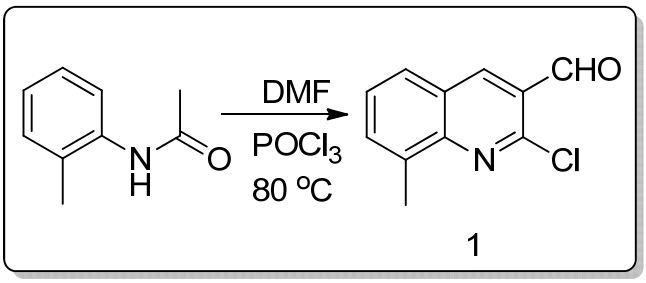
(254 nm, 366 nm), or by dipping into a permanganate solution (prepared by dissolving 3 g $KMnO_4$, 20 g K_2CO_3 , and 5 mL 5% NaOH in 300 mL H_2O), or an anisaldehyde solution (prepared by dissolving 3% *p*-methoxybenzaldehyde and 1% H_2SO_4 in MeOH) or 2,4-dinitro phenyl hydrazine solution (prepared by dissolving 12 g 2,4-DNP and 6 mL Conc. H_2SO_4 in a mixture of 8 mL water and 20 mL EtOH) followed by heating.

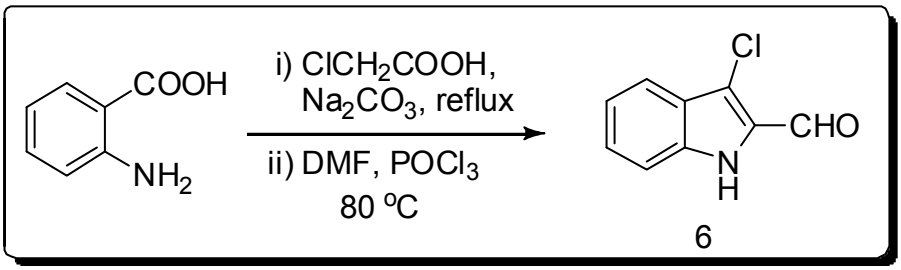
1.2 General Procedure for Synthesis of Aldehydes (1, 6)

1.2.1 Synthesis of Quinoline-3-carboxaldehyde (1):



$POCl_3$ (98.28 mmol) was added drop wise to DMF (34.65 mmol) while maintaining the temperature at 0–5 ˚C. The mixture was allowed to stir for about 5 min. Acetanilide **1a** (10.37 mmol) was then added and the resulting solution heated for 8 h at 75–80 ˚C. The reaction mixture was cooled to room temperature and then poured into crushed ice with stirring. A pale yellow precipitate appeared immediately and was filtered and washed with water and then dried. The crude compound was recrystallized from ethyl acetate.

1.2.2 3-chloro-1H-indole-2-carbaldehyde (6):



a) Phenylglycine-*o*-carboxylic acid:

In a 750 ml round bottomed flask fitted with a reflux condenser place 14 gm of anthranilic acid, 10 gm of chloroacetic acid, 20 gm of anhydrous sodium carbonate and 200 ml of water. Reflux the mixture for 3 h, then pour into a beaker, cool, render slightly acidic with concentrated hydrochloric acid, and allow to stand overnight. Filter off the crude acid and wash it with water. Recrystallize from hot water and dry at 100 °C.

b) **3-chloro-1H-indole-2-carbaldehyde:**

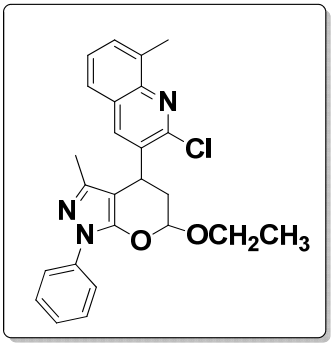
The Vilsmeier reagent was prepared by the drop wise addition of $POCl_3$ (30 mmol) to cooled DMF (5 mL) under constant stirring. The Phenylglycine-*o*-carboxylic acid (5 mmol) was dissolved in 5 mL of DMF and added drop wise to the Vilsmeier reagent. The reaction mixture was gradually allowed to attain rt, stirred for further 30 min, refluxed on awater bath maintained at 60-80 °C for 4-6 h. After the completion of the reaction, the reaction mixture was cool and neutralized with crushed ice. Filter off the crude product and recrystallize from ethyl acetate.

1.3 General Procedure for Synthesis of fused pyrano[2,3-*c*]pyrazoles (3a-c, 4a-c, 5a-c, 7a-c, 8a-c, 9a-c)

Equimolar amounts (5 mmol) of pyrazole **2a-c** and the appropriate heteroaryl aldehyde **1** or **6** with an excess of dienophile (7 mmol) taken in 2 mL ionic liquid TEAA was subjected to microwave irradiation at 420 w to complete the reaction as monitored by TLC. When poured the reaction mass, after cooling to room temperature, into ice, it gave quantitative desired products **3-5**, and **7-9**, as purified by column chromatography using a mixture containing ethyl acetate and n-hexane in a ratio of 30:70 as an eluent. Mixture of ionic liquid and water left after product isolation was then heated to evaporate water to recover the TEAA. All the newly synthesized compounds were characterized based on their elemental, mass, NMR and IR spectroscopy.

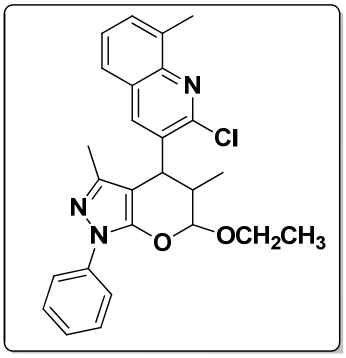
1.4 Analytical data for compounds

4-(2-chloro-8-methylquinolin-3-yl)-6-ethoxy-3-methyl-1-phenyl-1,4,5,6-tetrahydro-pyrano[2,3-*c*]pyrazole (3a):



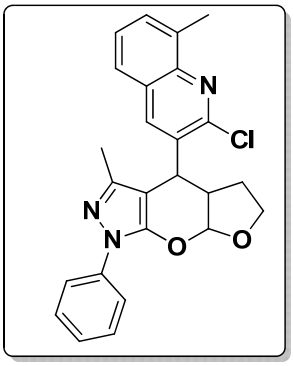
Isolated Yield (1.9 g, 90 %) as colorless crystals, mp 112-114 °C; ν_{max}/cm^{-1} = 2973, 2920, 1607, 1523, 1384, 1167, 1066, 769; δ_H (400 MHz, $CDCl_3$) 0.93 (t, 3H, J = 6.8 Hz & J = 6.4 Hz, $OCH_2\underline{CH_3}$), 1.96 (s, 3H, C(3)CH_3), 2.39-2.53 (m, 2H, C(5)H), 2.81 (s, 3H, C(Quinoline)CH_3),3.47-3.87 (m, 2H, $O\underline{CH_2}CH_3$), 4.58 (d, 1H, J = 3.6 Hz, C(4)H), 5.51 (m, 1H, C(6)H), 7.39-7.93 (m, 9H, C(Ar)H) ppm; δ_C (100 MHz; $CDCl_3$) 13.15, 14.85, 17.82, 30.43, 33.25, 64.77, 96.87, 101.91, 119.99, 125.30, 125.52, 126.71, 127.44, 129.08, 129.98, 134.08, 136.21, 138.82, 138.97, 145.93, 146.94, 149.27, 149.63 ppm; Anal. Calcd for $C_{25}H_{24}ClN_3O_2$: C, 69.20; H, 5.57; N, 9.68. Found: C,69.25; H, 5.55; N, 9.70.

4-(2-chloro-8-methylquinolin-3-yl)-6-ethoxy-3,5-dimethyl-1-phenyl-1,4,5,6-tetrahydro pyrano[2,3-*c*]pyrazole (4a):



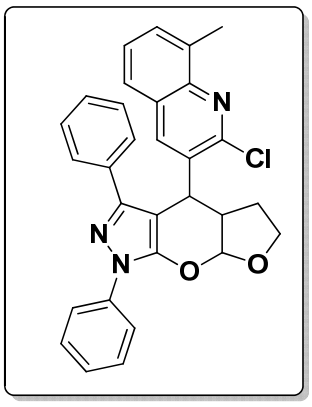
Isolated Yield (1.96 g, 90 %) as colorless crystals, mp 174-176 ˚C; ν_{max}/cm^{-1} = 2958, 2915, 2833, 1653, 1601, 1522, 1367, 982, 745; δ_H (400 MHz, $CDCl_3$) 1.15 (d, 3H, *J* = 6.4 Hz, C(5)CH_3), 1.25 (t, 3H, *J* = 6.4 Hz & *J* = 6.8 Hz, $OCH_2\underline{CH_3}$), 1.78 (s, 3H, C(3)CH_3), 2.30 (m, 1H, C(5)H), 2.83 (s, 3H, C(Quinoline)CH_3), 3.65-4.04 (m, 2H, $O\underline{CH_2}CH_3$), 4.45 (d, 1H, *J* = 4.2 Hz, C(4)H), 5.22 (d, 1H, *J* = 4.2 Hz, C(6)H), 7.24-7.94 (m, 9H, C(Ar)H) ppm; δ_C (100 MHz; $CDCl_3$) 13.09, 13.32, 14.95, 17.79, 65.65, 98.07, 103.97, 119.94, 125.23, 125.42, 126.98, 127.53, 129.04, 130.35, 136.51, 138.80, 146.03, 146.66, 148.87 ppm; m/z(ESI) 447.8 (M^+); Anal. Calcd for $C_{26}H_{26}ClN_3O_2$: C, 69.71; H, 5.85; N, 9.38. Found: C,69.65; H, 5.80; N, 9.32.

4-(2-chloro-8-methylquinolin-3-yl)-3-methyl-1-phenyl-1,4,4a,5,6,7a-hexahydro furo[3',2':5,6]pyrano[2,3-*c*]pyrazole (5a):



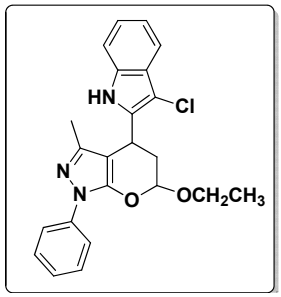
Isolated Yield (1.8 g, 88 %) as colorless crystals, mp 164-166 ˚C; ν_{max}/cm^{-1} = 3038, 2974, 2903, 1610, 1519, 1384, 1072, 753; δ_H (400 MHz, $CDCl_3$) 2.05 (s, 3H, C(3)CH_3), 2.08 (m, 1H, C(4a)H), 2.44-2.72 (m, 2H, C(5)H), 2.81 (s, 3H, C(Quinoline)CH_3), 4.12-4.36 (m, 2H, C(6)H), 4.60 (d, 1H, *J* = 3.8 Hz, C(4)H), 5.58 (d, 1H, *J* = 3.2 Hz, C(7a)H), 7.25-7.94 (m, 9H, C(Ar)H) ppm; δ_C (100 MHz; $CDCl_3$) 12.85, 17.82, 27.48, 33.49, 45.83, 68.74, 102.11, 120.03, 125.45, 125.58, 127.19, 127.44, 129.10, 130.50, 135.04, 136.42, 137.08, 138.69, 146.05, 147.09, 149.30, 149.44 ppm; m/z(ESI) 431.3 (M^+); Anal. Calcd for $C_{25}H_{22}ClN_3O_2$: C, 69.52; H, 5.13; N, 9.73. Found: C, 69.48; H, 5.15; N, 9.70.

4-(2-chloro-8-methylquinolin-3-yl)-1,3-diphenyl-1,4,4a,5,6,7a-hexahydro-furo[3',2':5,6] pyrano[2,3-*c*]pyrazole (5c):



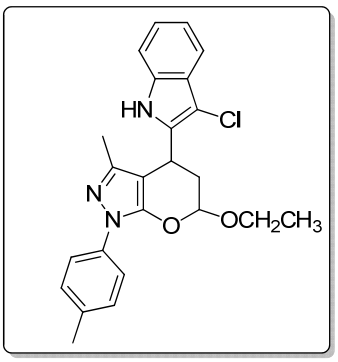
Isolated Yield (2.0 g, 86 %) as colorless crystals, mp 204-206 ˚C; ν_{max}/cm^{-1} = 2970, 2912, 1601, 1509, 1383, 1104, 754; δ_H (400 MHz, $CDCl_3$) 2.04-2.47 (m, 2H, C(5)H), 2.78 (m, 1H, C(4a)H), 2.83 (s, 3H, C(Quinoline)CH_3), 4.13-4.36 (m, 2H, C(6)H), 4.99 (d, 1H, *J* = 4.2 Hz, C(4)H), 5.67 (d, 1H, *J* = 3.6 Hz, C(7a)H), 7.22-8.09 (m, 14H, C(Ar)H) ppm; δ_C (100 MHz; $CDCl_3$) 17.78, 27.34, 34.83, 45.53, 68.86, 91.95, 102.25, 120.54, 125.57, 126.05, 126.16, 127.19, 127.45, 128.06, 128.65, 129.14, 130.60, 132.97, 135.20, 136.39, 137.54, 138.74, 146.17, 147.93, 148.97, 150.17 ppm; Anal. Calcd for $C_{30}H_{24}ClN_3O_2$: C, 72.94; H, 4.90; N, 8.51. Found: C, 72.85; H, 5.10; N, 8.46.

4-(3-chloro-1*H*-indol-2-yl)-6-ethoxy-3-methyl-1-phenyl-1,4,5,6-tetrahydropyrano[2,3-*c*]pyrazole (7a):



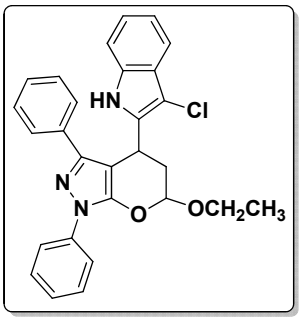
Isolated Yield (1.86 g, 90 %) as colorless crystals, mp= 186-188 °C(d); ν_{max}/cm^{-1} = 2923, 2854, 1622, 1545, 1480, 1370, 1152, 1031, 978, 737; δ_H (400 MHz, $CDCl_3$) 1.15 (t, 3H, *J* = 7.2 Hz & 6.8 Hz, $OCH_2\underline{CH_3}$), 1.36 (m, 1H, 5a-H), 1.79 (s, 3H, 3-CH_3), 2.09 (m, 1H, 5b-H), 3.53-3.77 (m, 2H, $O\underline{CH_2}CH_3$), 4.49 (d-d, 1H, *J* = 6 Hz & 10.4 Hz, 4-H), 5.17 (m, 1H, ,6-H), 7.18-7.73 (m, 9H, Ar-H), 9.97 (s, 1H, NH) ppm. Anal. Calcd for $C_{23}H_{22}ClN_3O_2$: C, 67.73; H, 5.44; N, 10.30. Found: C, 67.90; H, 5.39; N, 10.34.

4-(3-chloro-1*H*-indol-2-yl)-6-ethoxy-3-methyl-1-(*p*-tolyl)-1,4,5,6-tetrahydropyrano[2,3-*c*]pyrazole (7b):



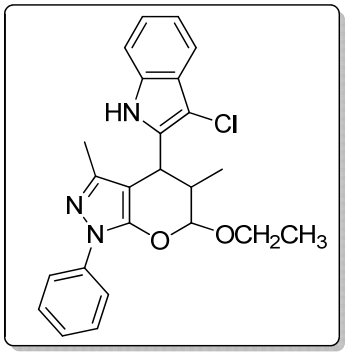
Isolated Yield (2.0 g, 85 %) as colourless crystals, mp 170-172 °C (d); ν_{max}/cm^{-1} = 2923, 2854, 1622, 1545, 1480, 1370, 1152, 1031, 978, 737; δ_H (400 MHz, $CDCl_3$) 1.16 (t, 3H, *J* = 7.2 Hz & *J* = 7.2 Hz, $OCH_2\underline{CH_3}$), 1.30 (1H, C(5a)H), 1.80 (s, 3H, C(3)CH_3), 2.13 (m, 1H, C(5b)H), 2.38 (s, 3H, C(Ph)CH_3), 3.51-3.80 (m, 2H, $O\underline{CH_2}CH_3$), 4.50 (d-d, 1H, *J* = 5.6 & 10 Hz, C(4)H), 5.17 (m, 1H, , C(6)H), 7.18-7.73 (m, 8H, C(Ar)H), 9.97 (s, 1H, NH) ppm; δ_C (100 MHz; $CDCl_3$) 12.80, 14.95, 20.90, 22.65, 24.26, 33.58, 65.03, 96.22, 100.81, 111.34, 117.62, 119.92, 120.11, 120.27, 122.42, 126.02, 129.54, 134.56, 135.17, 136.07, 146.85, 148.55 ppm; Anal. Calcd for $C_{24}H_{24}ClN_3O_2$: C, 68.32; H, 5.73; N, 9.96. Found: C, 68.28; H, 5.72; N, 10.01.

4-(3-chloro-1*H*-indol-2-yl)-6-ethoxy-1,3-diphenyl-1,4,5,6-tetrahydropyrano[2,3-*c*]pyrazole (7c):



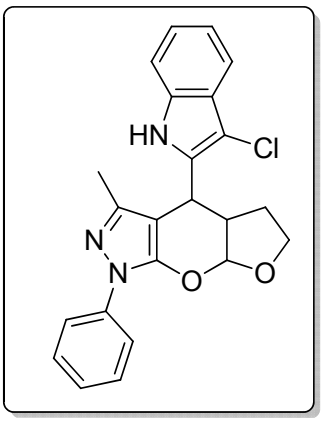
Isolated Yield (2.1 g, 82 %) as colourless crystals, mp 176-178 °C(d); ν_{max}/cm^{-1} = 2977, 2927, 1592, 1573, 1511, 1454, 1384, 1110, 1041, 979, 752; δ_H (400 MHz, $CDCl_3$) 1.27 (t, 3H, *J* = 7.2 Hz & *J* = 6.8 Hz, OCH_2CH_3), 2.14-2.47 (m, 2H, C(5)H), 3.67-4.04 (m, 2H, OCH2CH3), 4.87 (t, 1H, *J* = 6.4 Hz & *J* = 6.8 Hz, C(4)H), 5.41 (m, 1H, 6-H), 7.08-7.99 (m, 14H, C(Ar)H), 8.08 (s, 1H, NH) ppm; ^{13}C δ_C (100 MHz; $CDCl_3$) 15.06, 26.44, 34.31, 65.54, 94.42, 101.26, 103.42, 111.20, 117.55, 120.32, 120.65, 122.44, 126.13, 126.24, 126.46, 127.92, 128.17, 129.10, 132.36, 133.90, 134.71, 138.57, 148.38, 150.06 ppm; m/z(ESI) 469.8 (M^+); Anal. Calcd for $C_{28}H_{24}ClN_3O_2$: C, 71.56; H, 5.15; N, 8.94. Found: C, 71.44; H, 5.08; N, 9.01.

4-(3-chloro-1*H*-indol-2-yl)-6-ethoxy-3,5-dimethyl-1-phenyl-1,4,5,6-tetrahydropyrano [2,3-*c*]pyrazole (8a):



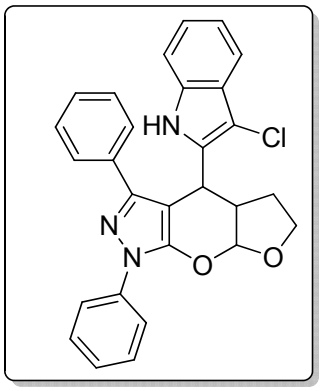
Isolated Yield(2.0 g 87 %) as colourless crystals, mp 190-192 ˚C(d); ν_{max}/cm^{-1} =3010, 2976, 1609, 1514, 1383, 1072, 747; δ_H (400 MHz, $CDCl_3$) 0.86 (d, 3H, J = 7.2 Hz, C(5)CH_3), 1.16 (t, 3H, J = 6.8 Hz, $OCH_2\underline{CH_3}$), 1.35-1.50 (m, 1H, C(5)H), 1.63 (s, 3H, C(3)CH_3), 3.55-4.14 (m, 2H, $O\underline{CH_2}CH_3$), 4.108 (d, 1H, J = 7.0 Hz, C(4)H), 4.91 (d, 1H, J = 2 Hz, C(6)H), 7.18-7.92 (m, 9H, C(Ar)H), 10.27 (s, 1H, NH) ppm; ^{13}C δ_C (100 MHz; $CDCl_3$) 12.51, 13.29, 30.54, 30.63, 38.17, 65.27, 97.66, 104.42, 104.47, 105.12, 111.44, 117.86, 119.70, 119.90, 122.36, 125.24, 125.73, 128.99, 133.49, 134.89, 138.50, 147.33, 148.52 ppm; Anal. Calcd for $C_{24}H_{24}ClN_3O_2$: C, 68.32; H, 5.73; N, 9.96. Found: C, 68.28; H, 5.78; N, 9.92.

4-(3-chloro-1*H*-indol-2-yl)-3-methyl-1-phenyl-1,4,4a,5,6,7a-hexahydrofuro [3',2':5,6]pyrano[2,3-*c*]pyrazole (9a):



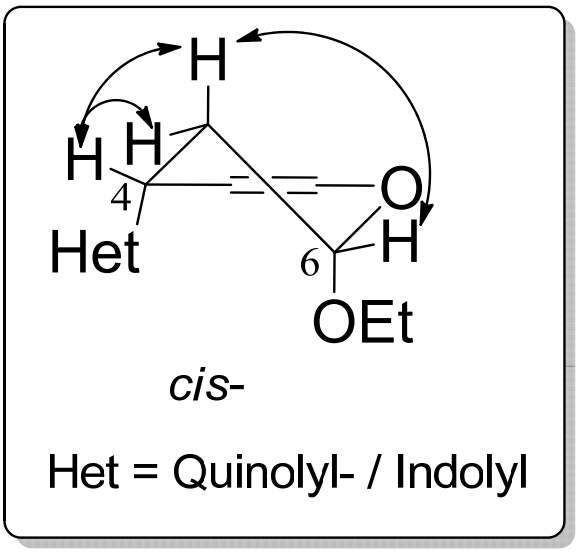
Isolated Yield (2.0 g, 89 %) as colourless crystals, mp 178-180 ˚C(d); ν_{max}/cm^{-1} =3010, 2976, 1609, 1514, 1383, 1072, 747; δ_H (400 MHz, $CDCl_3$) 1.90-1.96 (m, 1H, C(4a)H), 2.01 (s, 3H, C(3)CH_3), 3.35-2.73 (m, 2H, C(5)H), 4.05-4.26 (m, 2H, C(6)H), 4.49 (d, 1H, J =4.8 Hz, C(4)H), 5.31 (d, 1H, J = 3.6 Hz, C(7a)H), 7.21-7.86 (m, 9H, C(Ar)H), 9.29 (br, 1H, N-H) ppm; δ_C (100 MHz; $CDCl_3$) 12.65, 27.27, 28.05, 45.38, 68.52, 102.68, 111.58, 117.69, 118.61, 119.60, 119.67, 120.57, 122.69, 125.66, 126.53, 129.15, 133.96, 135.59, 138.46, 147.54, 149.33 ppm; m/z(ESI) 405.9 (M^+); Anal. Calcd for $C_{23}H_{20}ClN_3O_2$: C, 68.06; H, 4.97; N, 10.35. Found: C, 68.10; H, 5.01; N, 10.32.

4-(3-chloro-1*H*-indol-2-yl)-1,3-diphenyl-1,4,4a,5,6,7a-hexahydrofuro [3',2':5,6]pyrano[2,3-*c*]pyrazole (9c):



Isolated Yield (2.3 g, 90 %) as colourless crystals, mp 210-212 ˚C(d); ν_{max}/cm^{-1} =2999, 2945, 1619, 1583, 1541, 1444, 1353, 1121, 1001, 980, 756; δ_H (400 MHz, $CDCl_3$) 1.97-2.05 (m, 1H, C(4a)H), 2.34-2.96 (m, 2H, C(5)H), 4.12-4.33 (m, 2H, C(6)H), 4.85 (d, 1H, J =0.8 Hz, C(4)H), 5.79 (d, 1H, J = 3.6 Hz, C(7a)H), 7.21-8.05 (m, 14H, C(Ar)H), 8.02 (br, 1H, N-H) ppm; δ_C (100 MHz; $CDCl_3$) 26.99, 29.51, 29.59, 45.20, 68.74, 79.08, 90.14, 102.45, 103.09, 111.61, 120.47, 120.62, 120.78, 122.93, 126.18, 126.62, 128.23, 128.77, 129.14, 132.66, 133.85, 135.58, 138.54, 148.35, 149.98 ppm; Anal. Calcd for $C_{28}H_{22}ClN_3O_2$: C, 71.87; H, 4.74; N, 8.98. Found: C, 71.75; H, 4.82; N, 8.90.

1.5 Characteristicnoe's of 7a



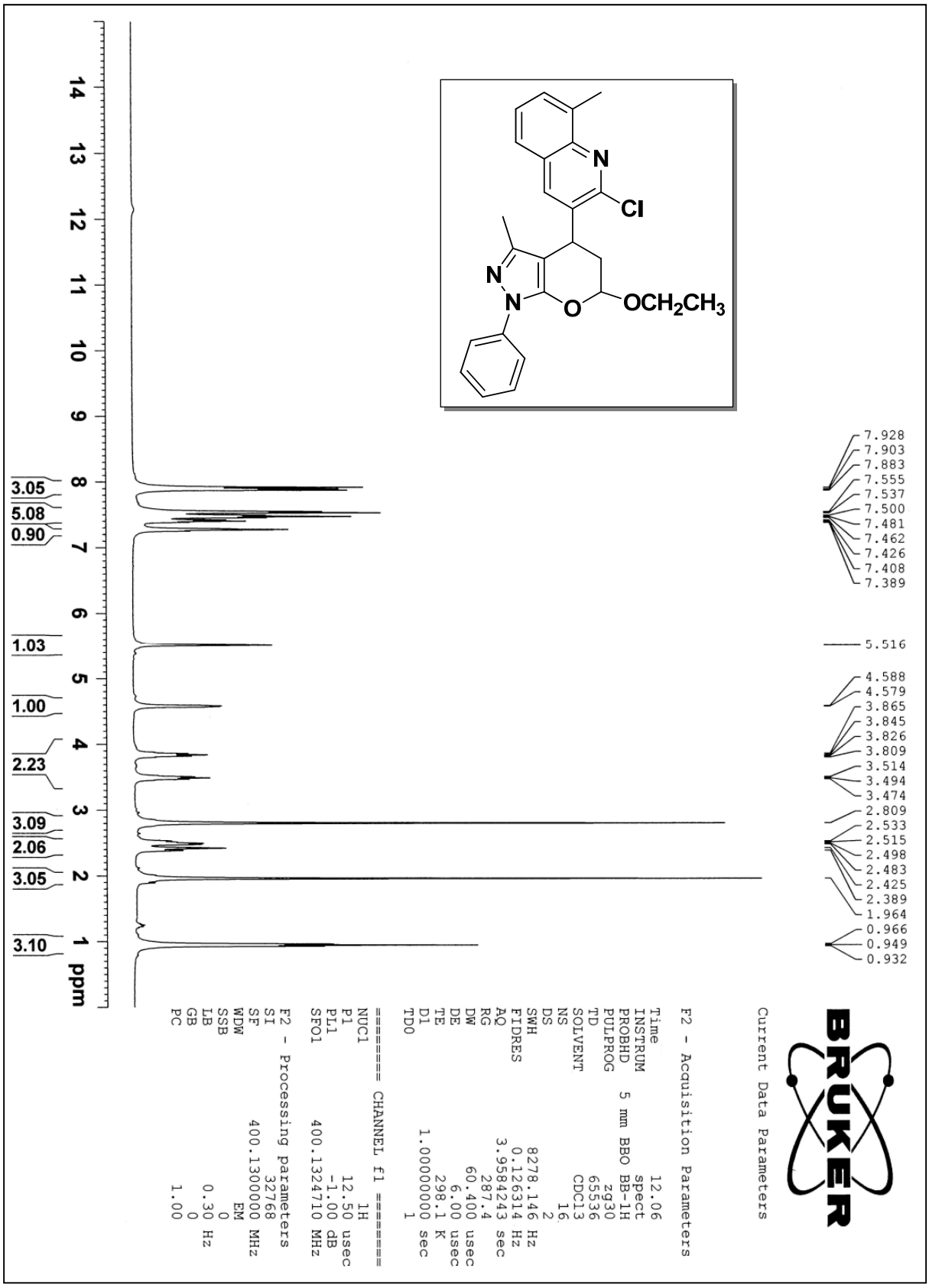
1.6 Signal of H-4 and H-6/H-7a in ^{1}H NMR

Table 4: Signals of H-4 and H-6/H-7a in the ^{1}H NMR spectra of *cis* **3-5** & **7-9**

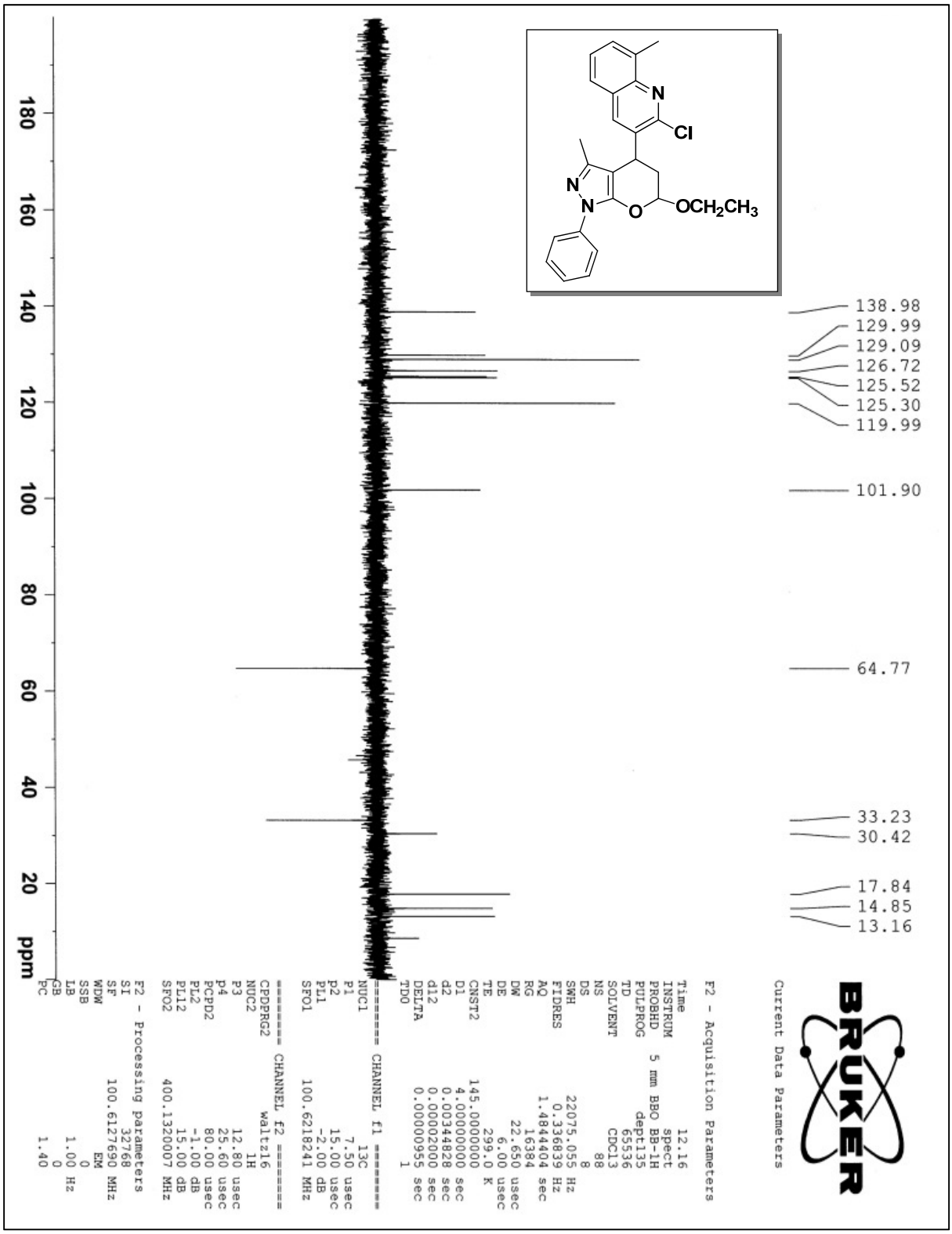
Cis	δ (H-4) (ppm) $J_{5(ax.),4}/J_{5(eq.),4}$ (Hz)	δ (H-6) (ppm) $J_{5(ax.),6}/J_{5(eq.),6}$ (Hz)	Cis	δ (H-4) (ppm) $J_{4a(eq.),4}$ (Hz)	δ (H-7a) (ppm) $J_{4a(eq.),7a}$ (Hz)
Cis-3a	4.58	5.51	Cis-5a	4.60	5.58
	3.6/3.6	-		3.8	3.2
Cis-4a	4.45	5.22	Cis-5c	4.99	5.67
	4.2	4.2		4.2	3.6
Cis-7a	4.49	5.17	Cis-9a	4.49	5.31
	10.4/6.0	-		4.8	3.6
Cis-7b	4.50	5.17	Cis-9c	4.85	5.79
	10.0/5.6	-		0.8	3.6
Cis-7c	4.87	5.41			
	6.8/6.4	-			
Cis-8a	4.10	4.91			
	7.0	2			

1.7 ^{1}H NMR, ^{13}C NMR and Mass Spectral Data

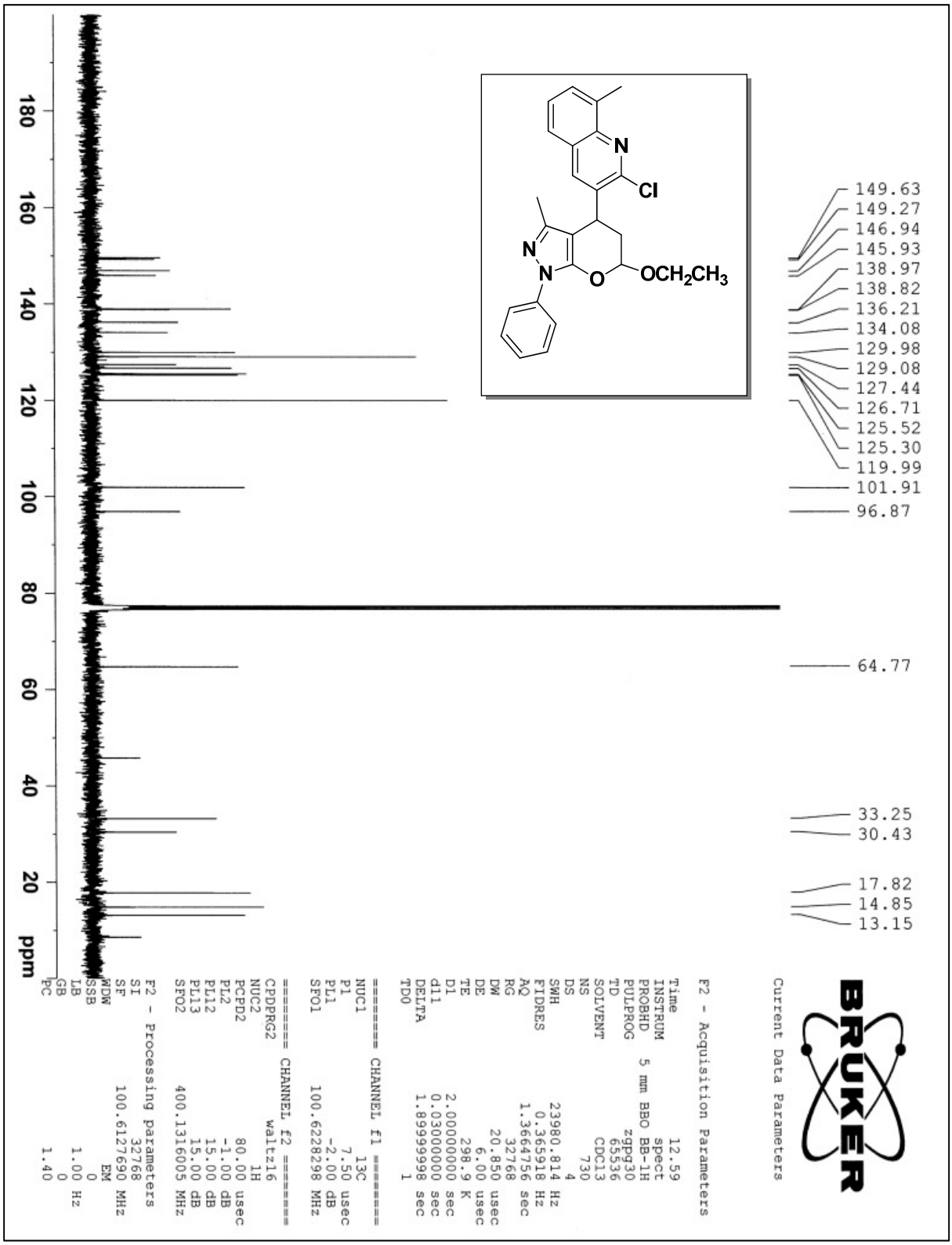
^{1}H-NMR/**3a**/$CDCl_3$



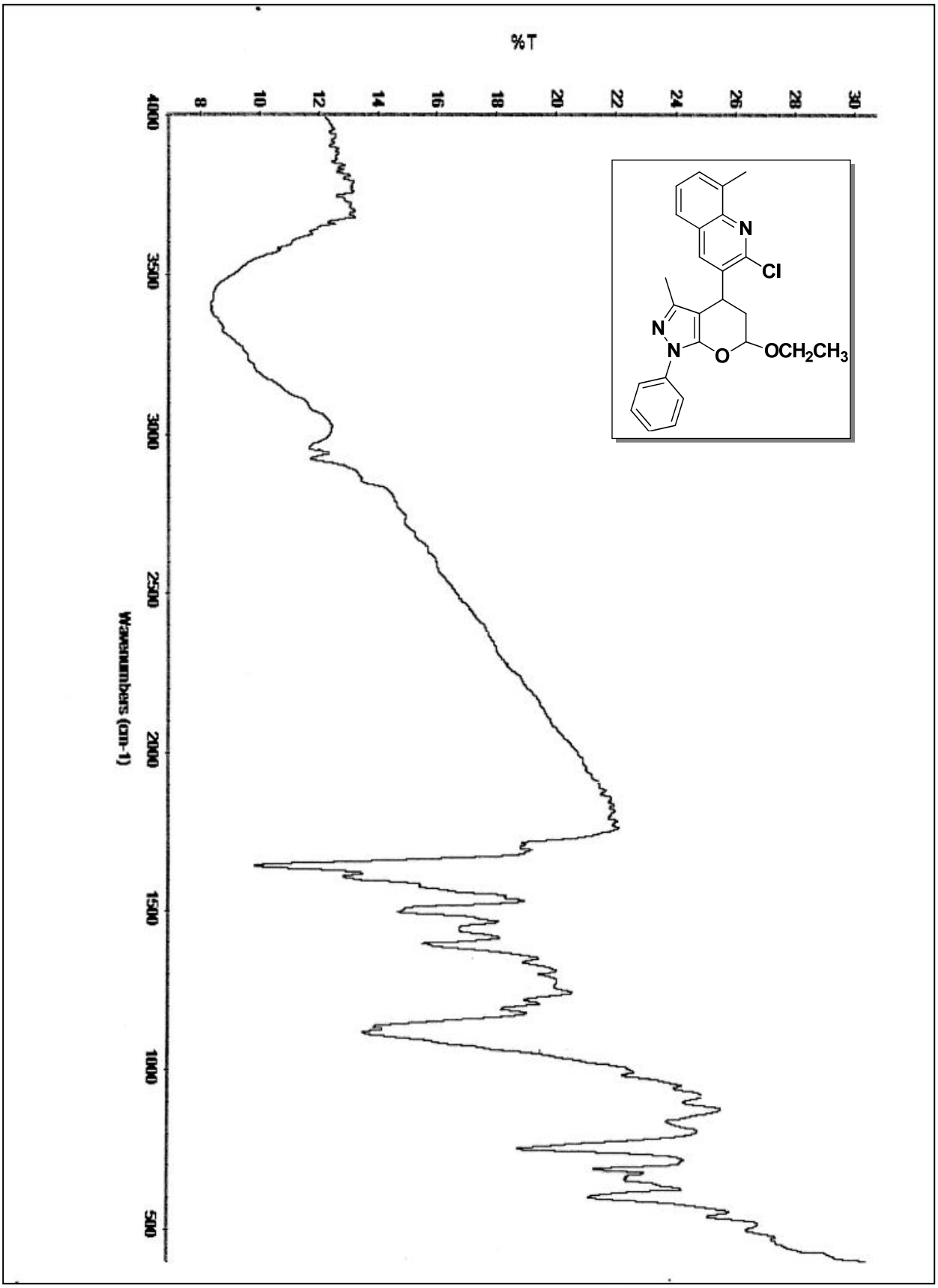
^{13}C-NMR (DEPT-135)/**3a**/ $CDCl_3$



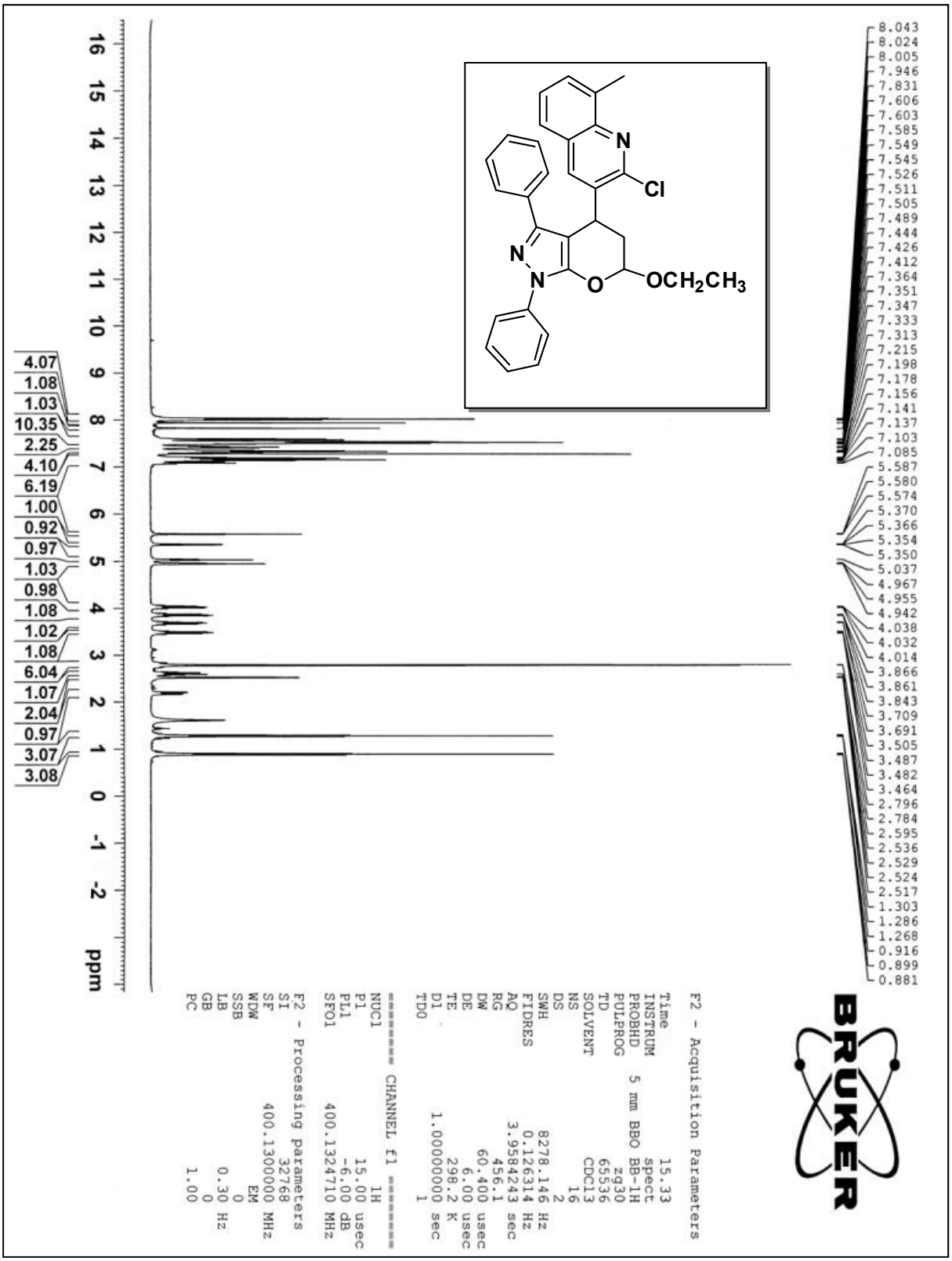
^{13}C-NMR/**3a**/ $CDCl_3$



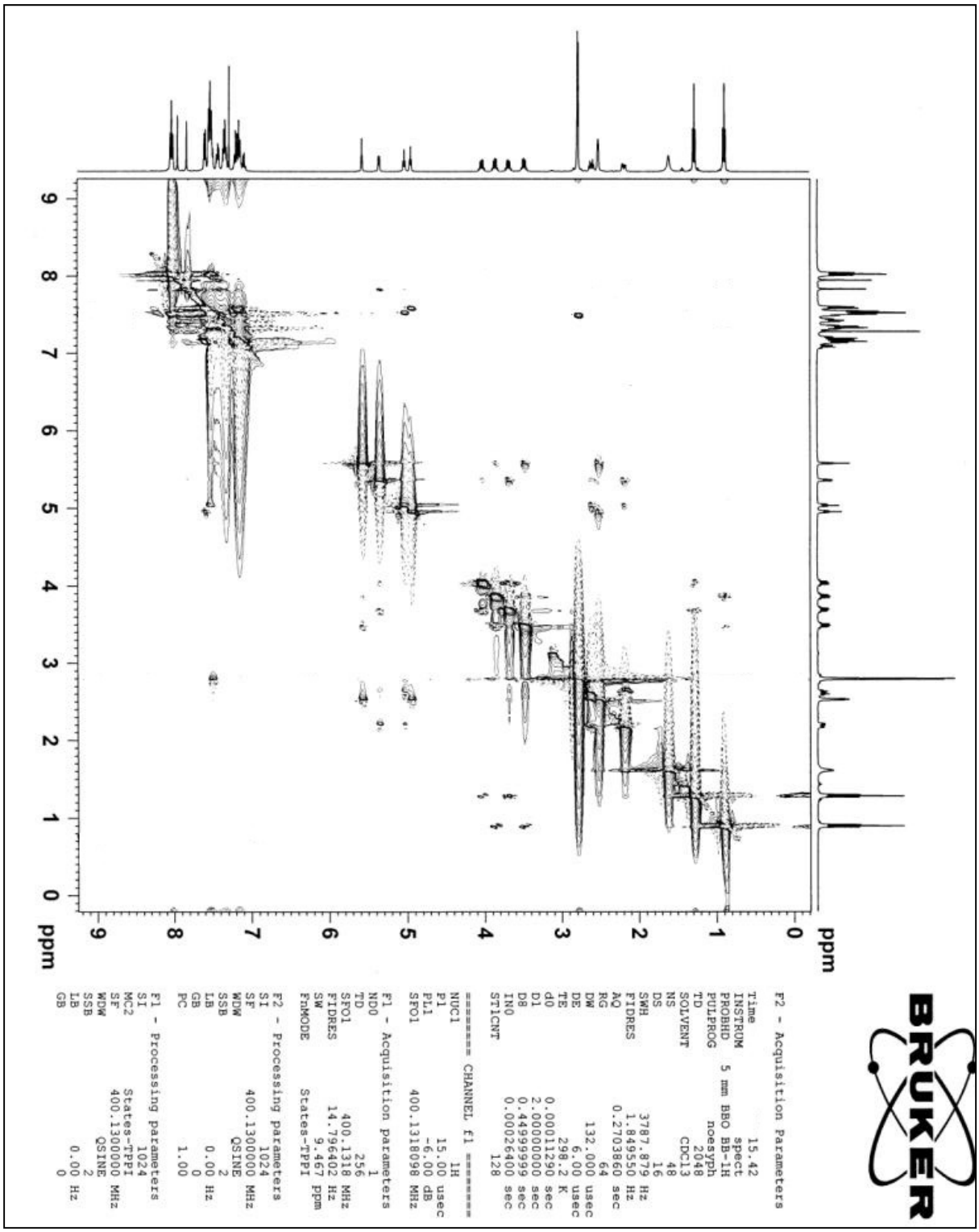
IR/**3a**/KBr



^{1}H-NMR/**3c**/$CDCl_3$



2D NOE for 3c


ppm
ppm
BRUKER
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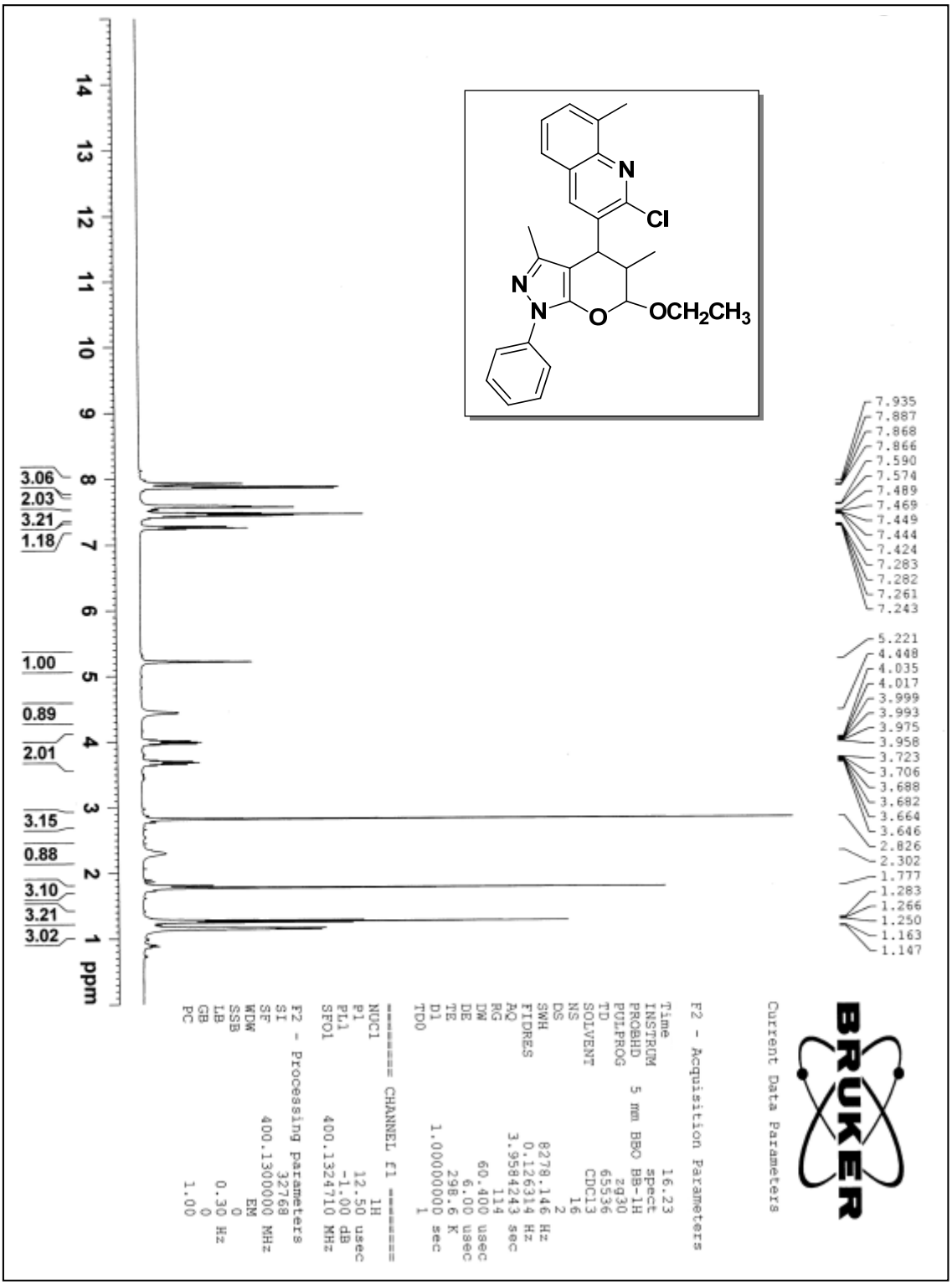
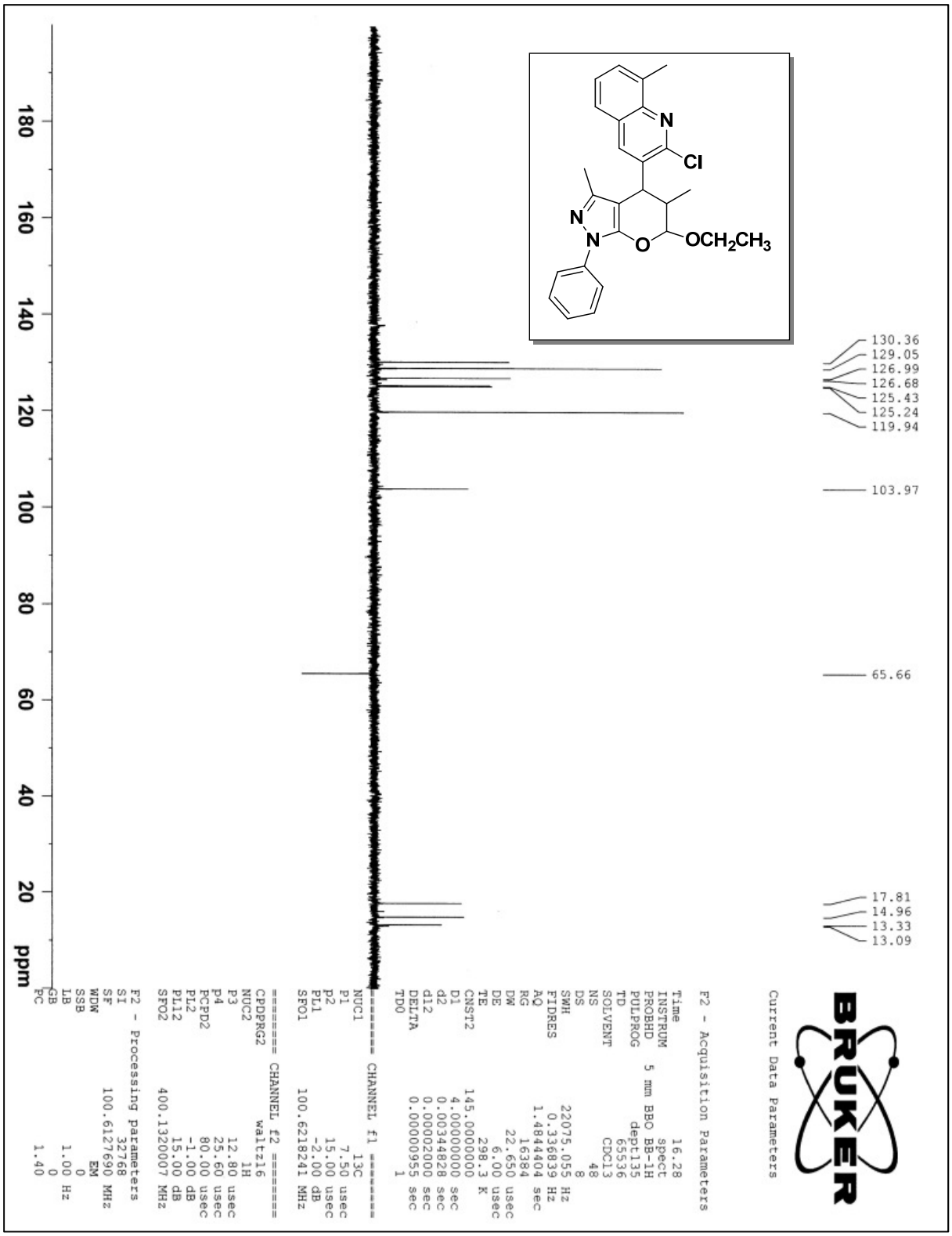
Time 15.42
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG noesyph
TD 2048
SOLVENT CDCl3
NS 48
DS 16
SWH 3787.879 Hz
FIDRES 1.849550 Hz
AQ 0.2703860 sec
RG 64
DW 132.000 usec
DE 6.00 usec
TE 298.2 K
d0 0.00011290 sec
D1 2.00000000 sec
D8 0.44999999 sec
IN0 0.00026400 sec
STSCNT 128
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -6.00 dB
SFO1 400.1318098 MHz
F1 - Acquisition parameters
NDO 1
TD 256
SFO1 400.1318 MHz
FIDRES 14.796402 Hz
SW 9.467 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 1024
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

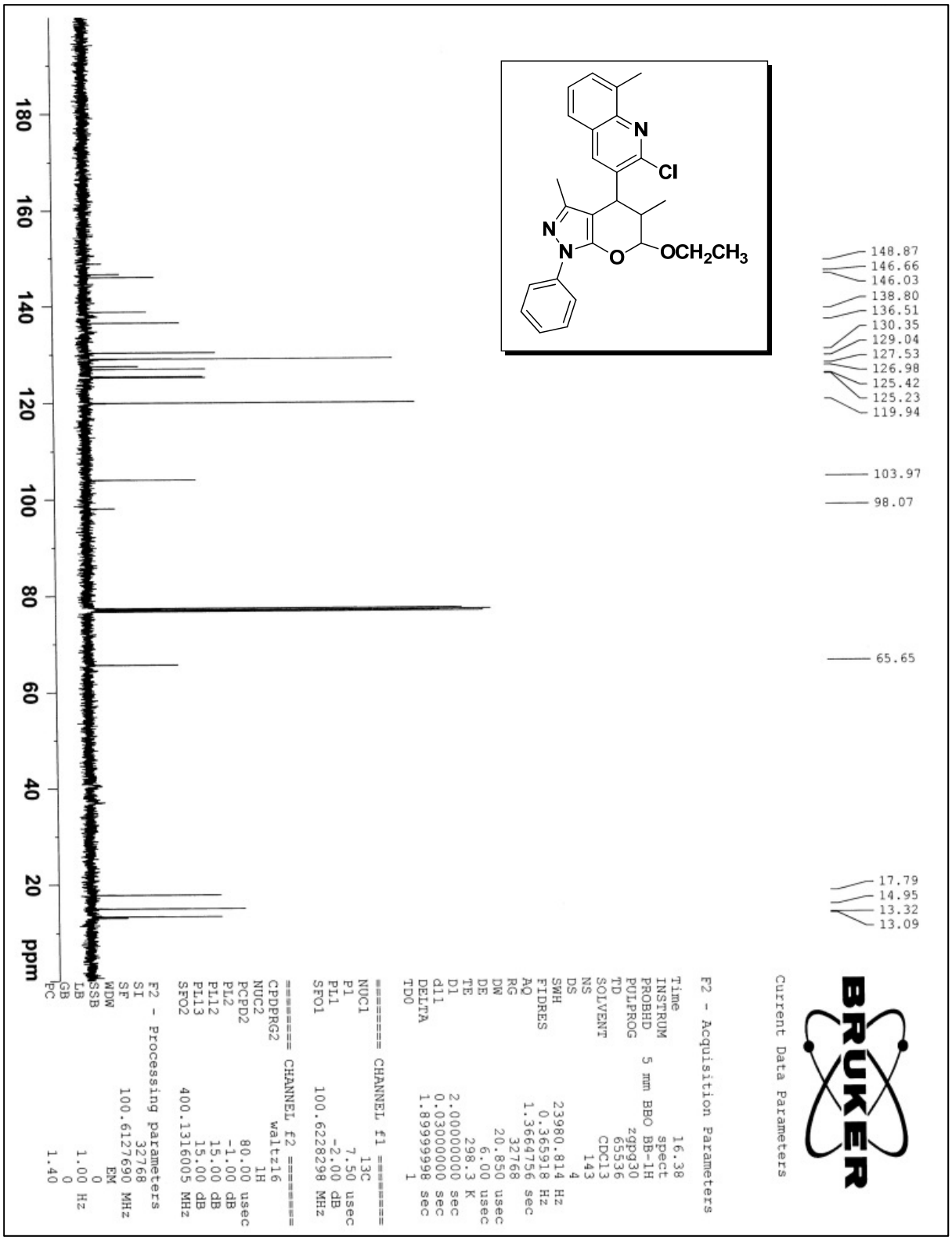
^{1}H-NMR/**4a**/$CDCl_3$



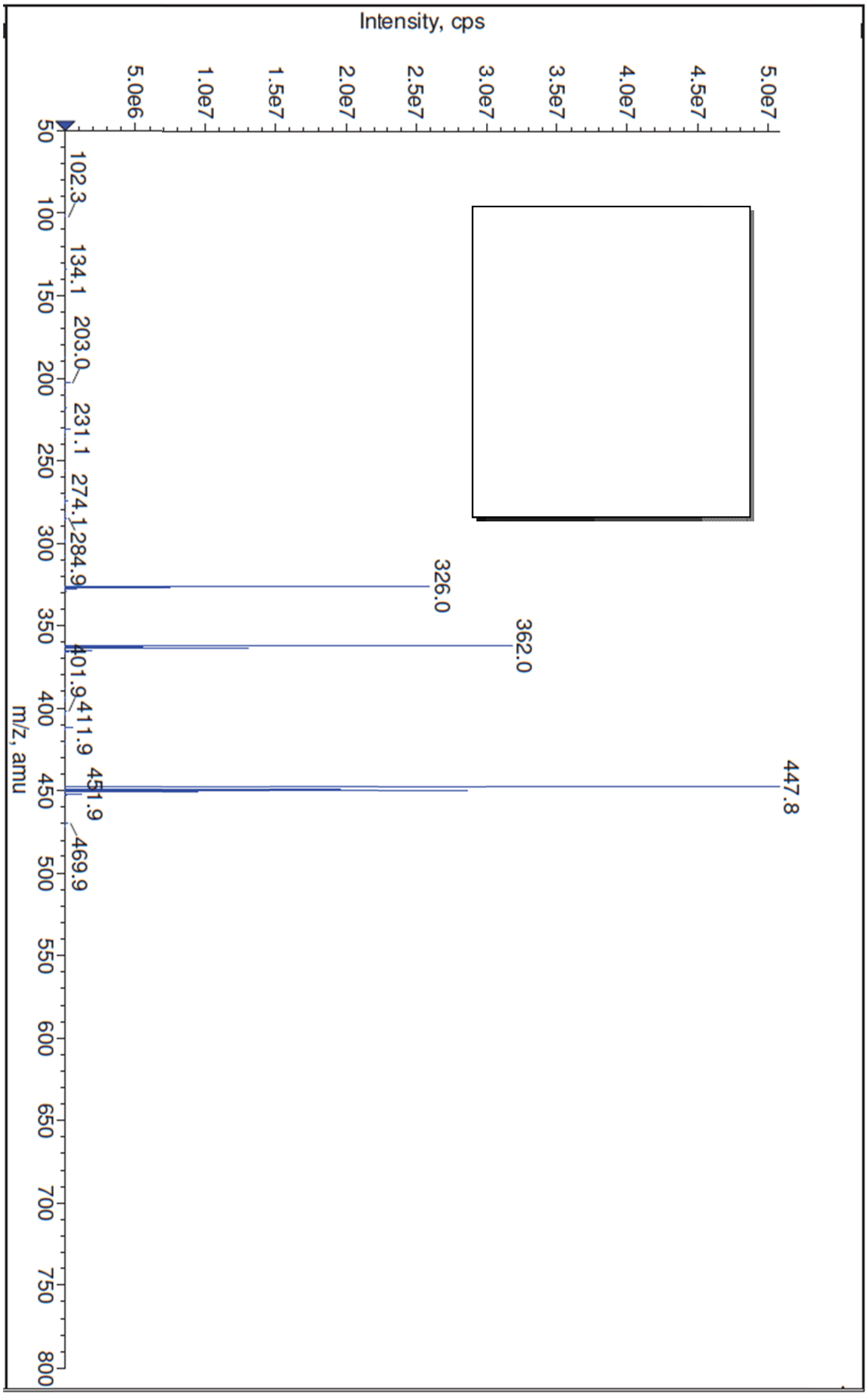
^{13}C-NMR (DEPT-135)/**4a**/ $CDCl_3$



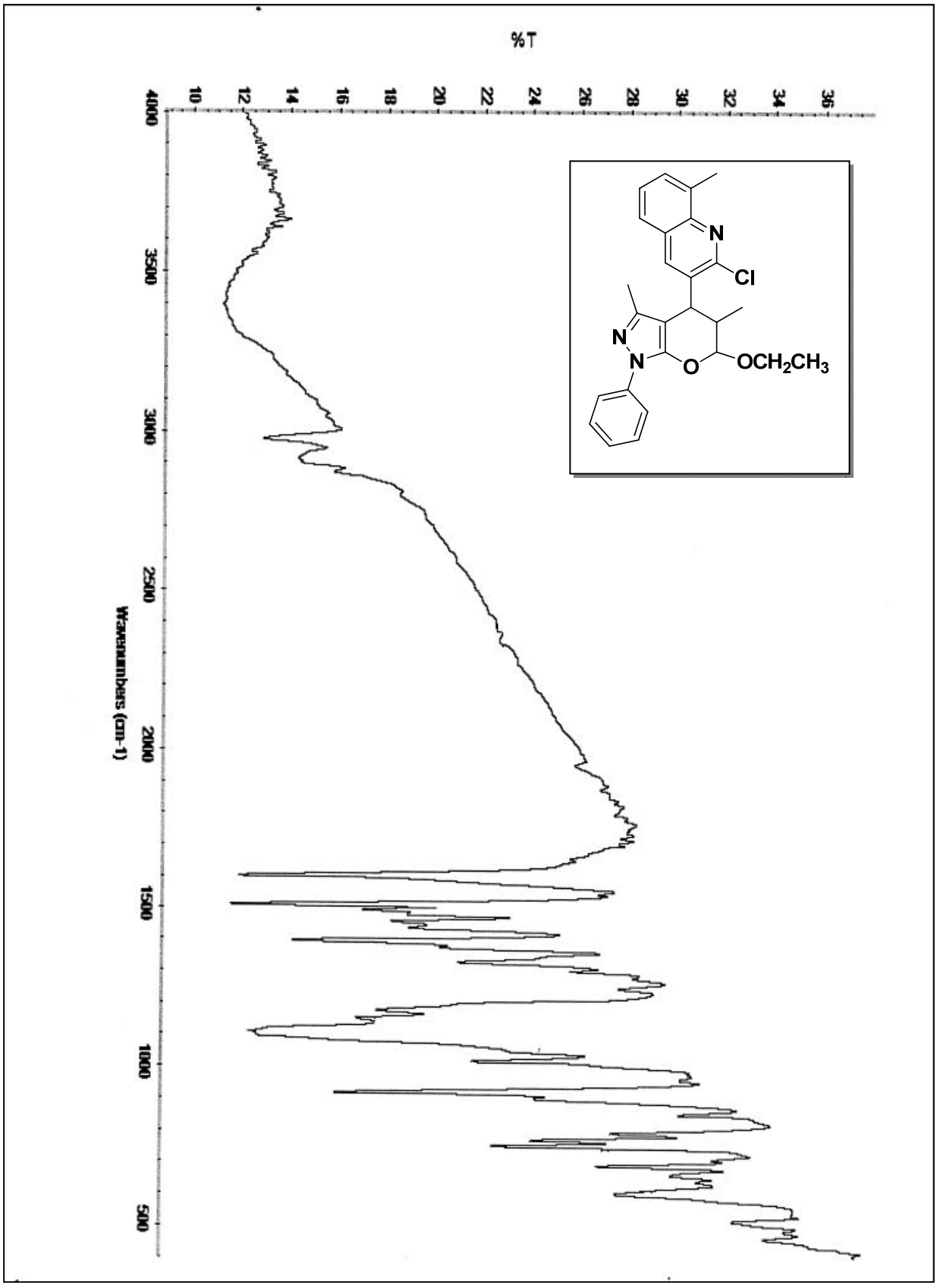
^{13}C-NMR/**4a**/ $CDCl_3$



MS/**4a**



IR/**4a**/KBr


%T
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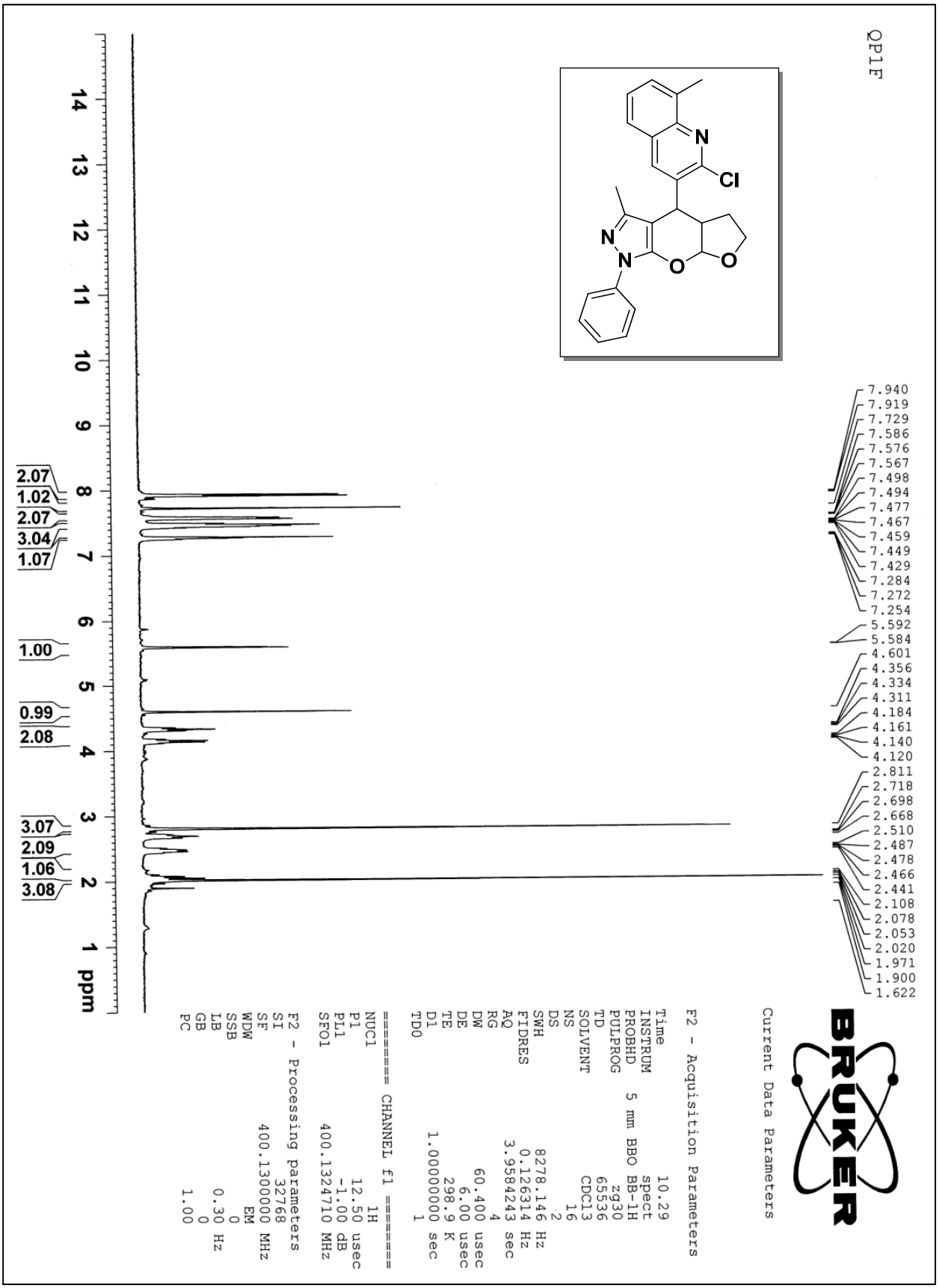
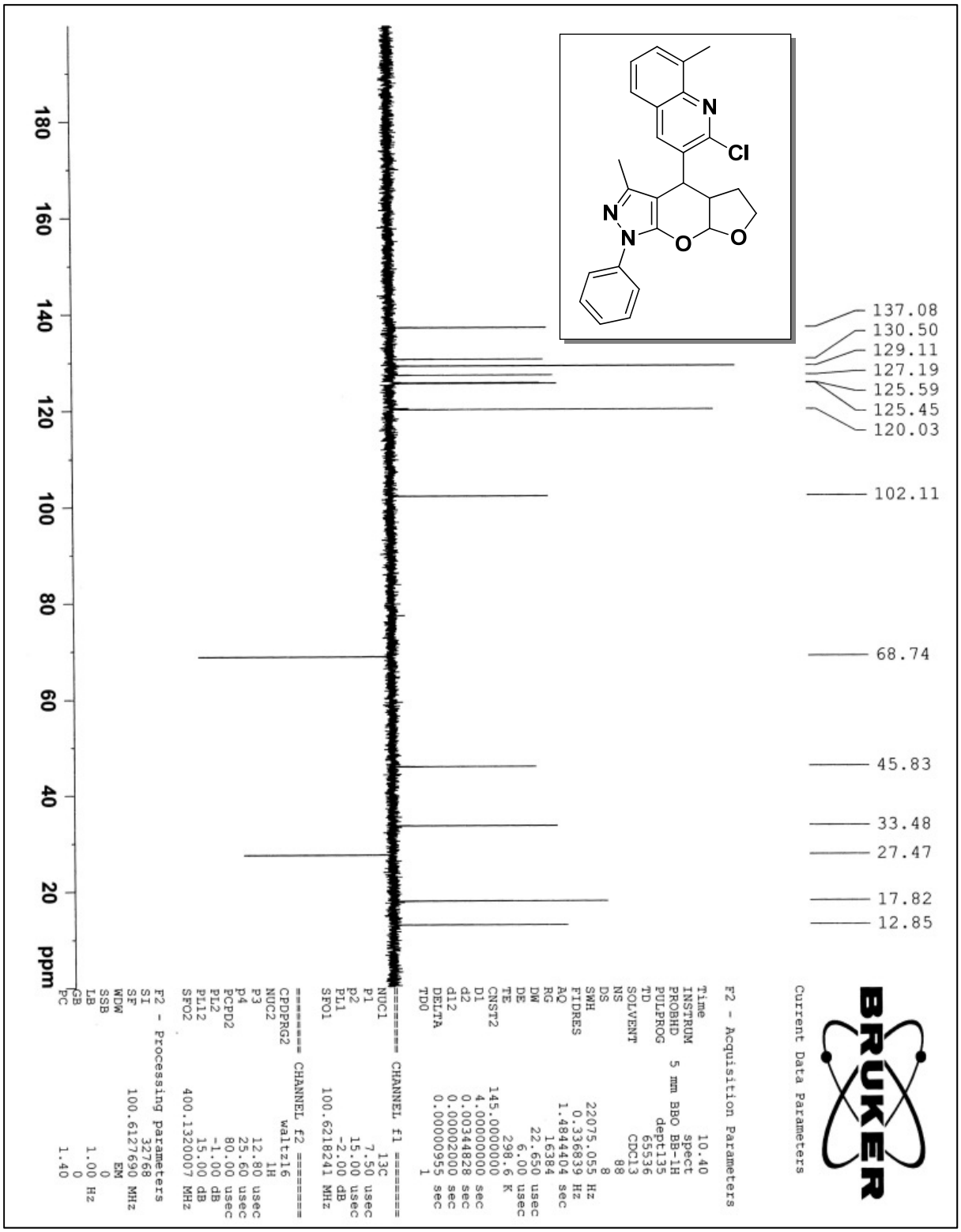
36
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
N
Cl
N
N
O
OCH2CH3

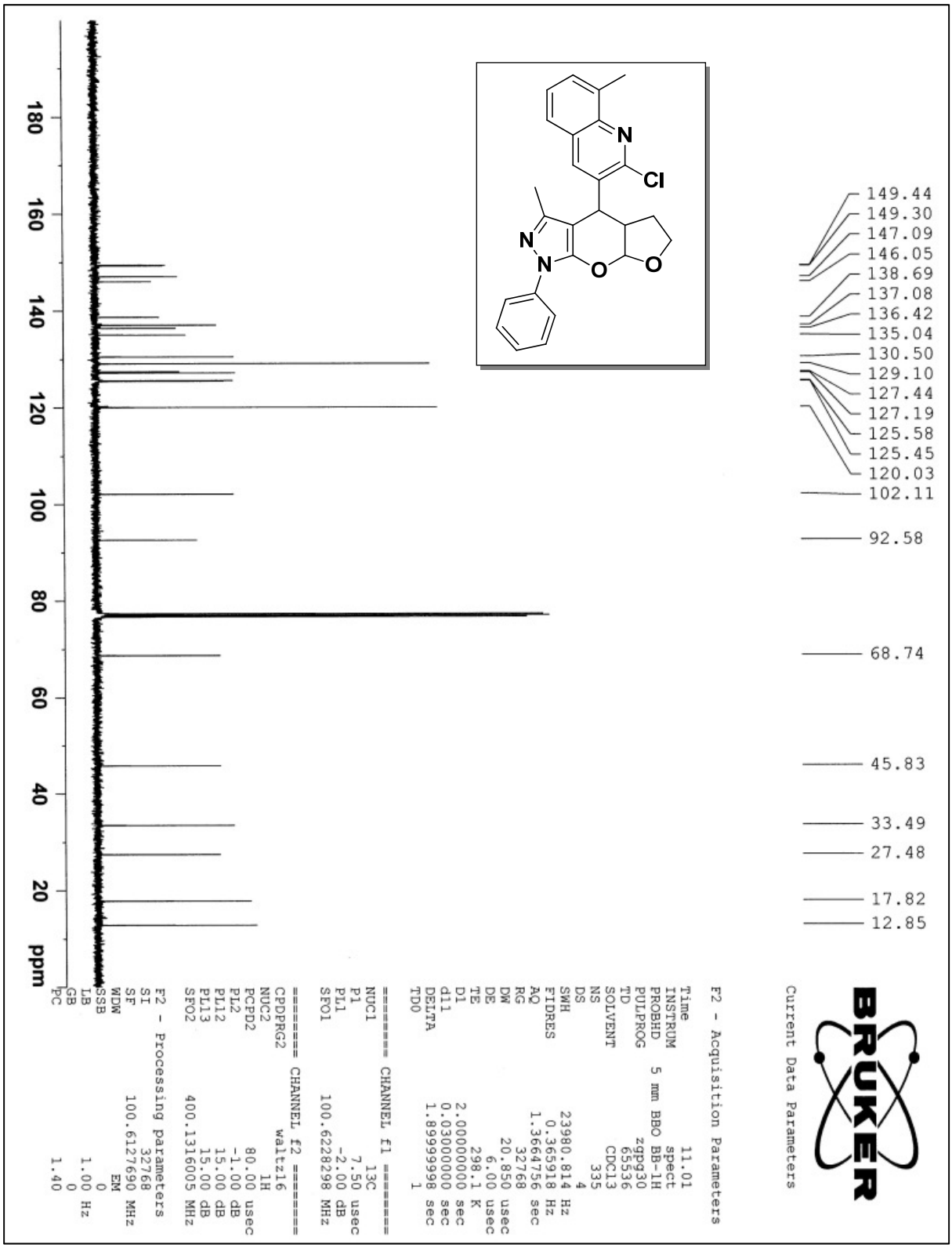
^{1}H-NMR/**5a**/$CDCl_3$



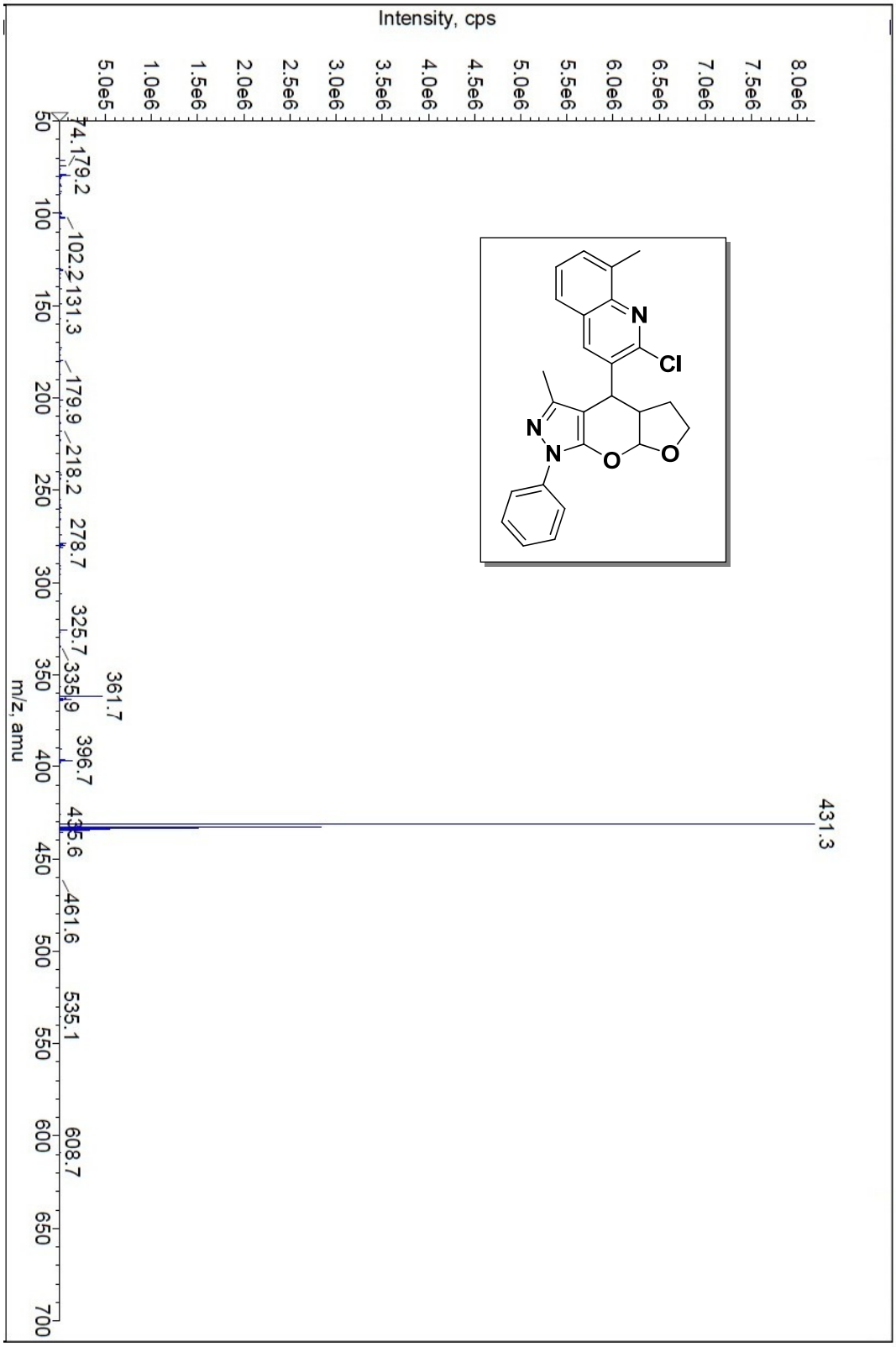
^{13}C-NMR (DEPT-135)/ **5a**/ $CDCl_3$



^{13}C-NMR/**5a**/ $CDCl_3$



MS/**5a**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3.5e6
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4.5e6
5.0e6
5.5e6
6.0e6
6.5e6
7.0e6
7.5e6
8.0e6
74.1
79.2
102.2
131.3
179.9
218.2
278.7
325.7
335.9
361.7
396.7
431.3
435.6
461.6
535.1
608.7
m/z, amu
50
100
150
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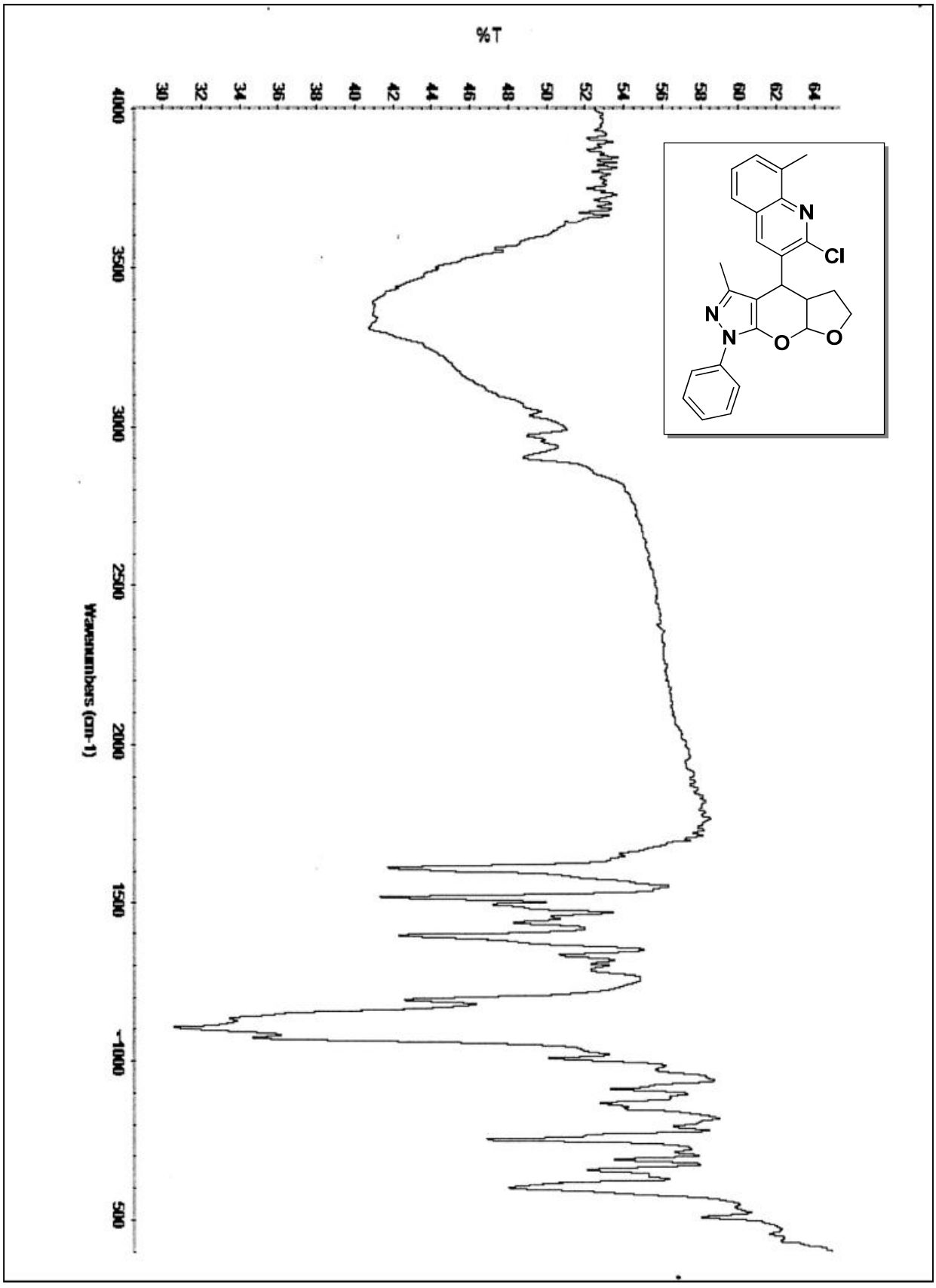
400
450
500
550
600
650
700
N
Cl
N
N
O
O

IR/**5a**/KBr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56
58
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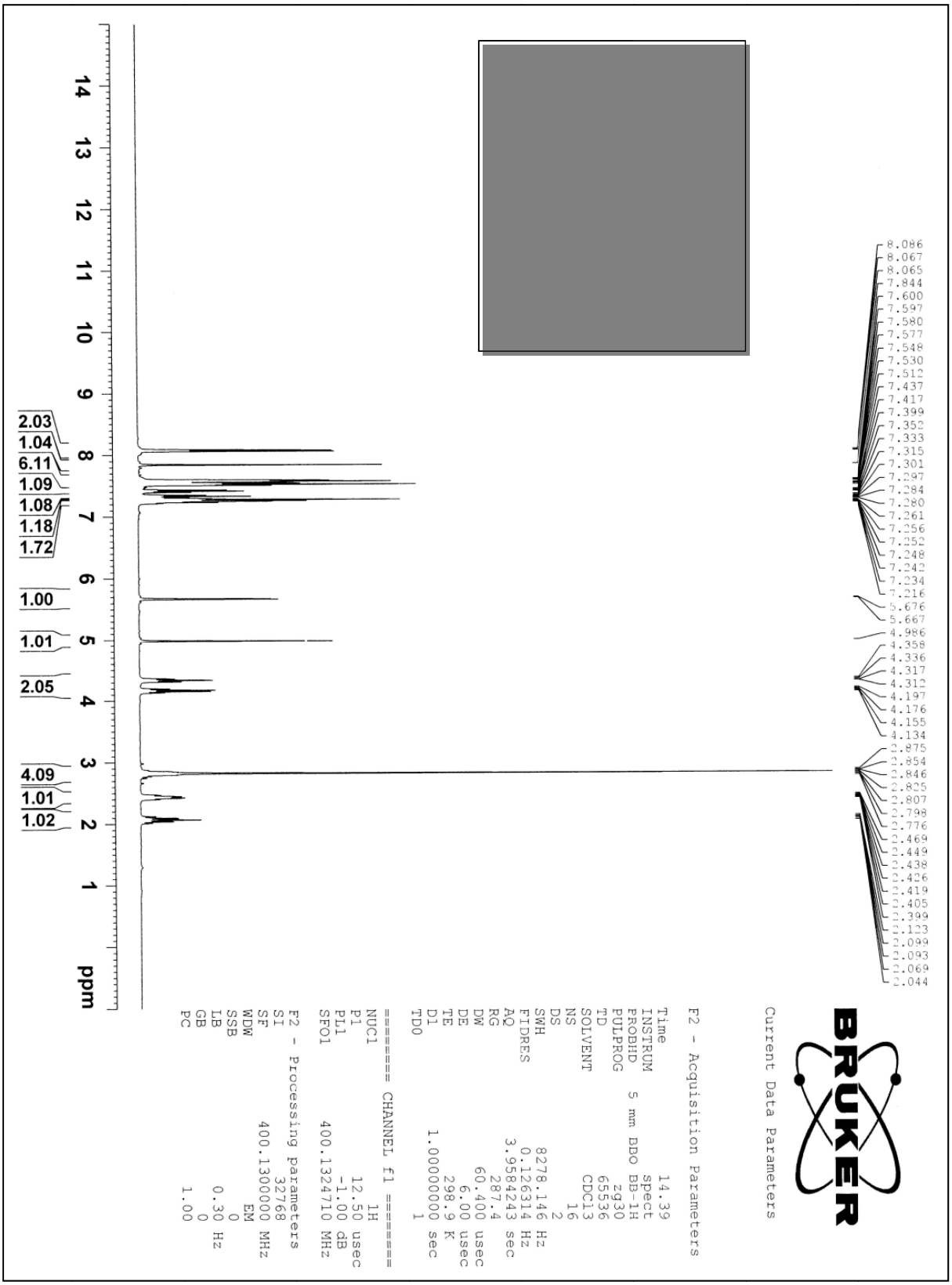
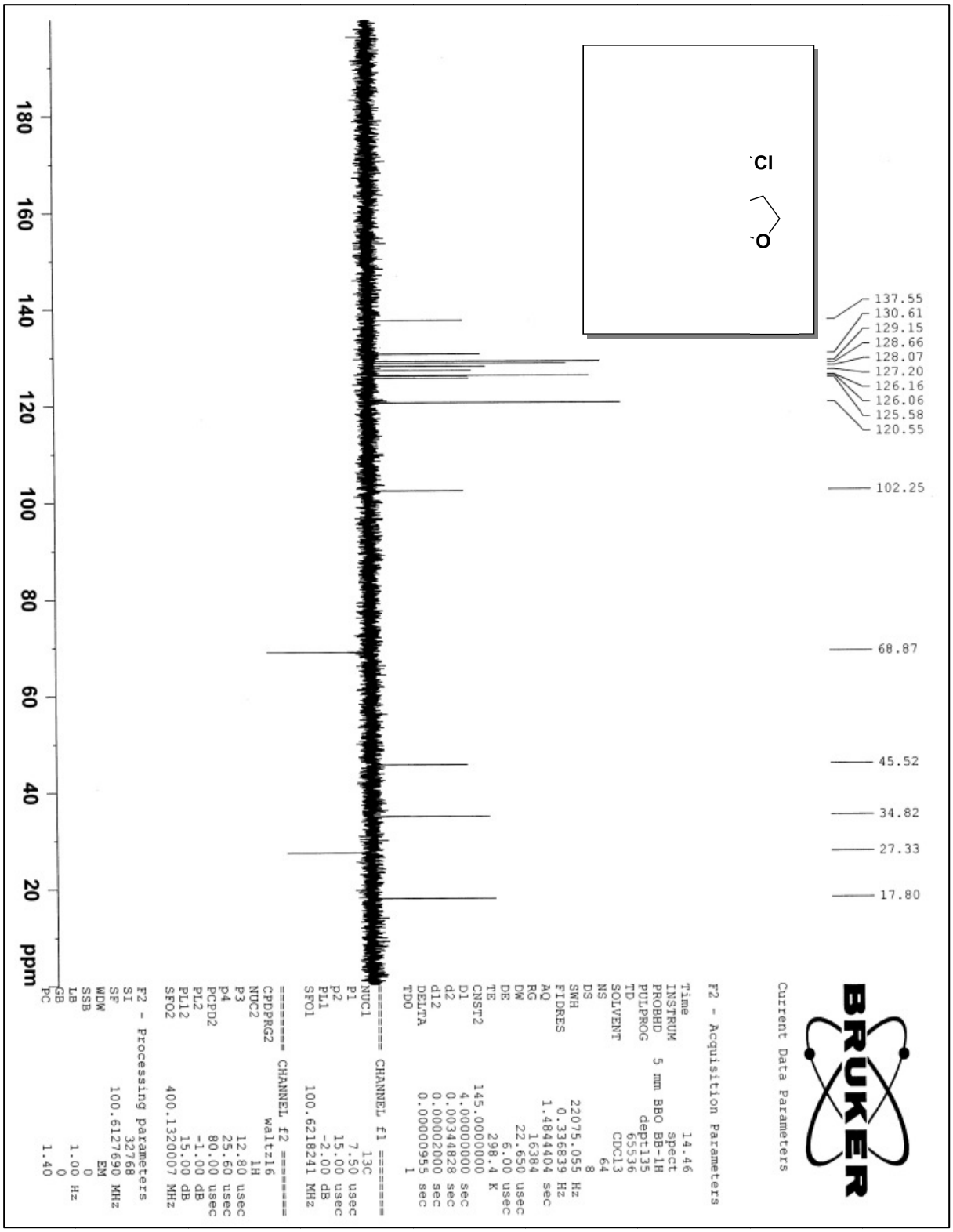
62
64
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
N
Cl
N
N
O
O

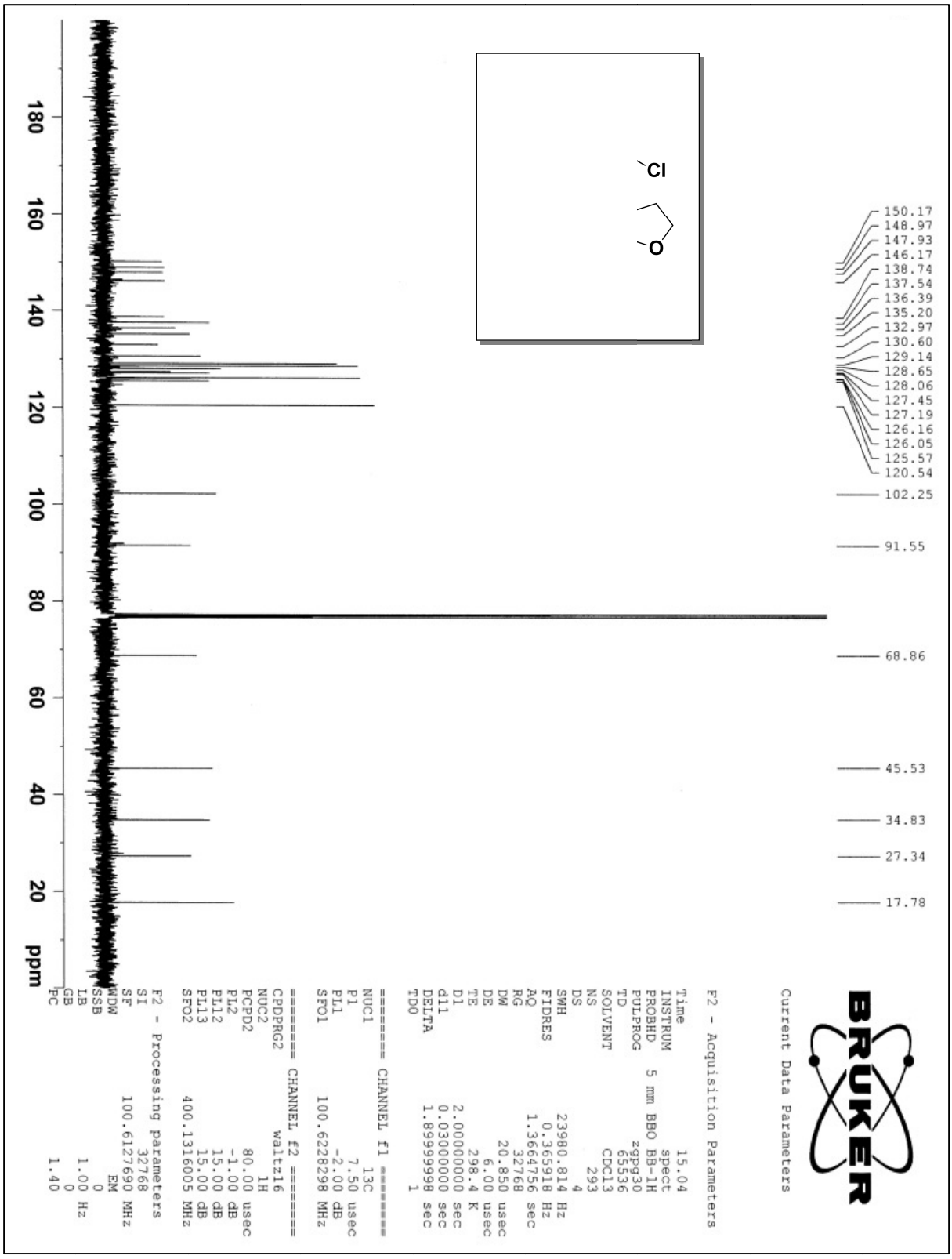
^{1}H-NMR/**5c**/$CDCl_3$



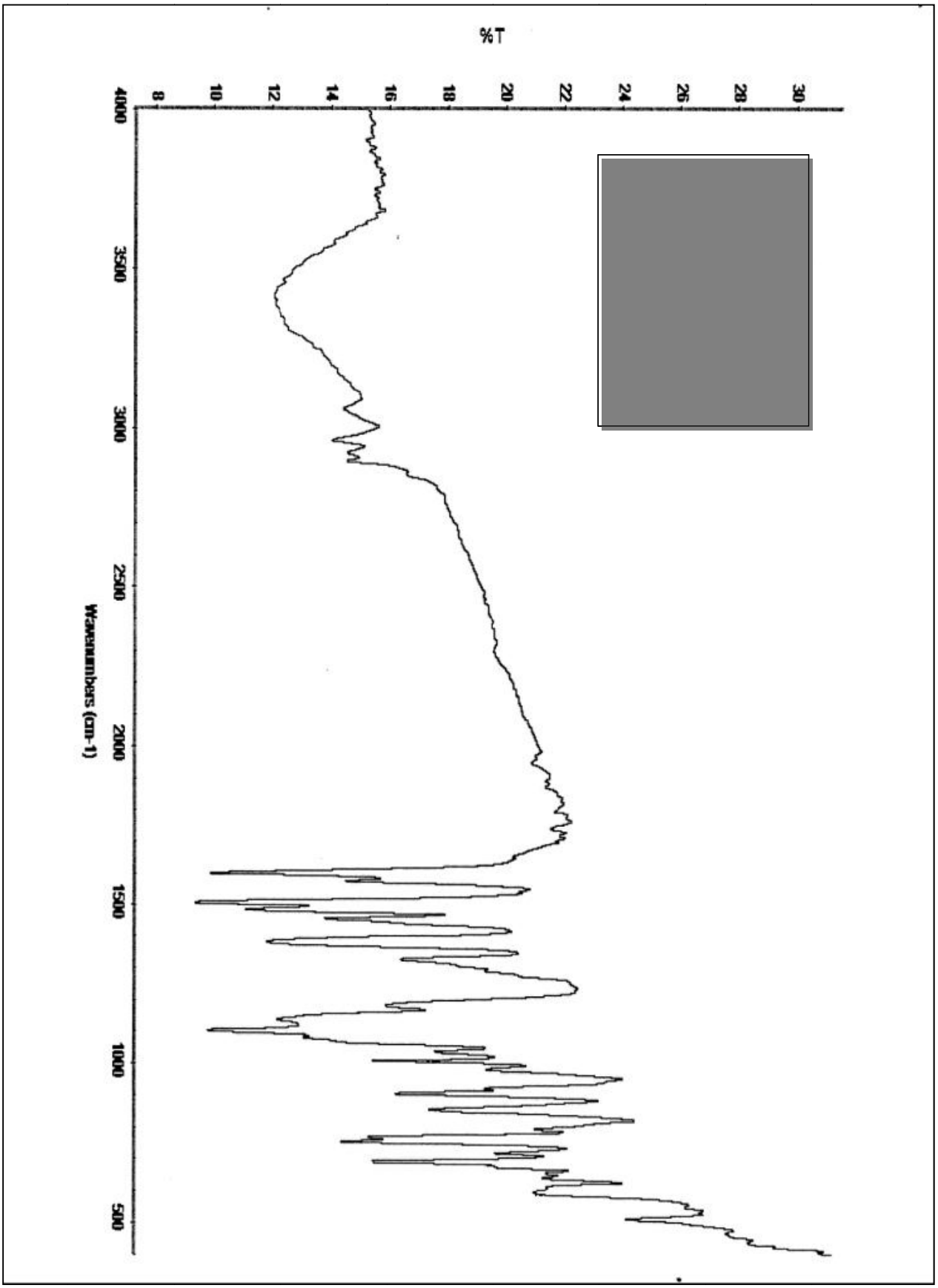
^{13}C-NMR (DEPT-135)/**5c**/ $CDCl_3$



^{13}C-NMR/**5c**/ $CDCl_3$



IR/**5c**/ KBr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2500
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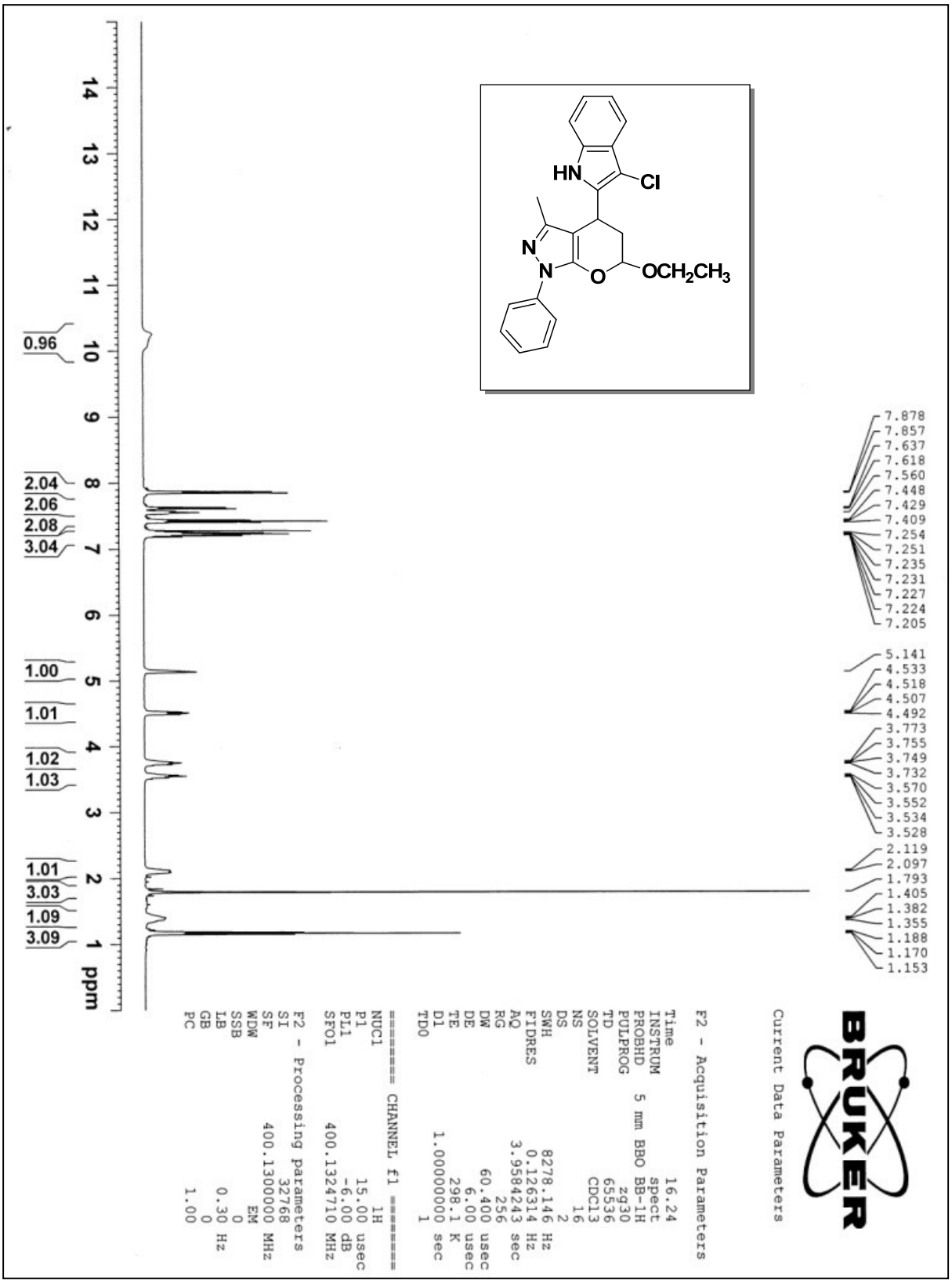
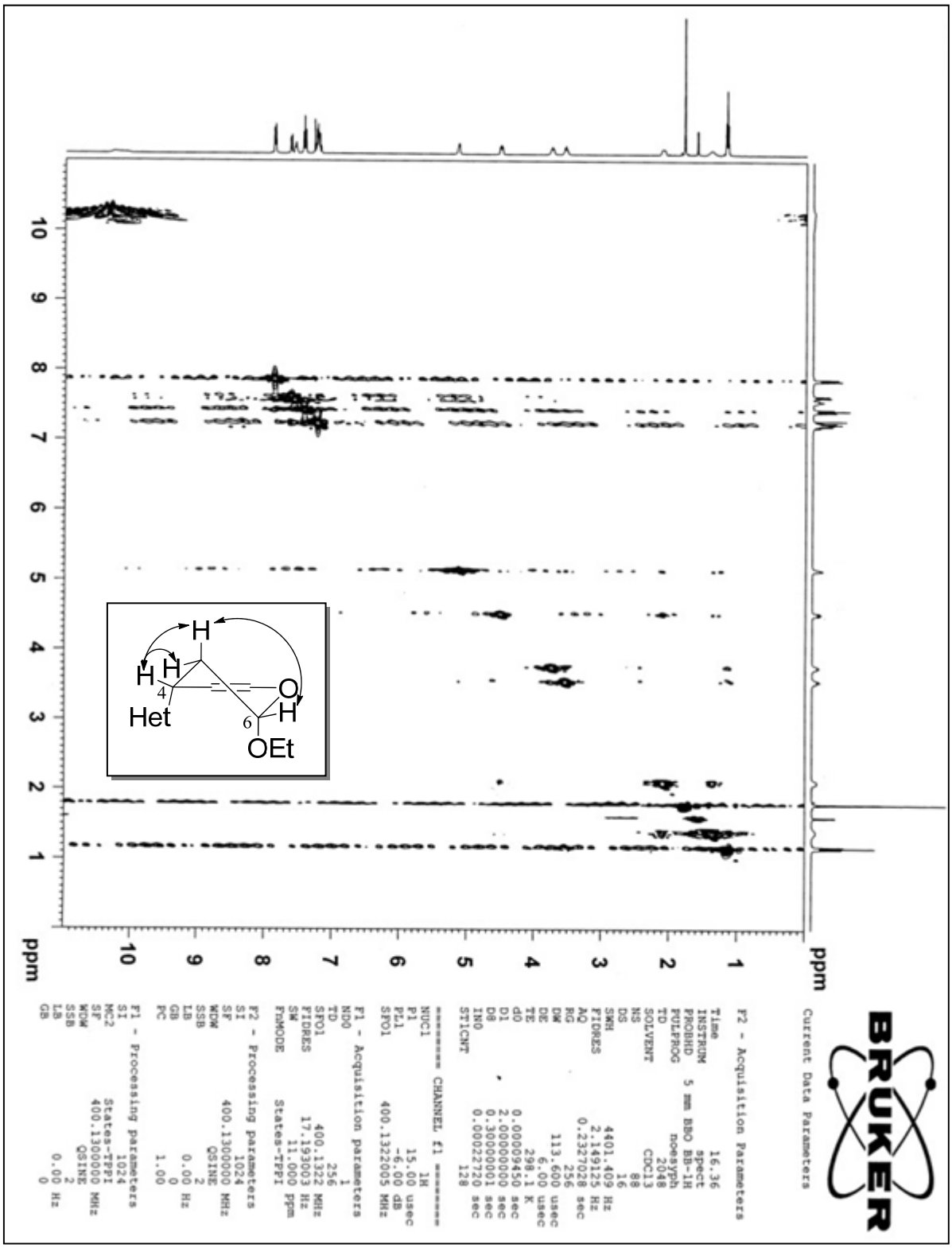
1000
500
Wavenumbers (cm-1)

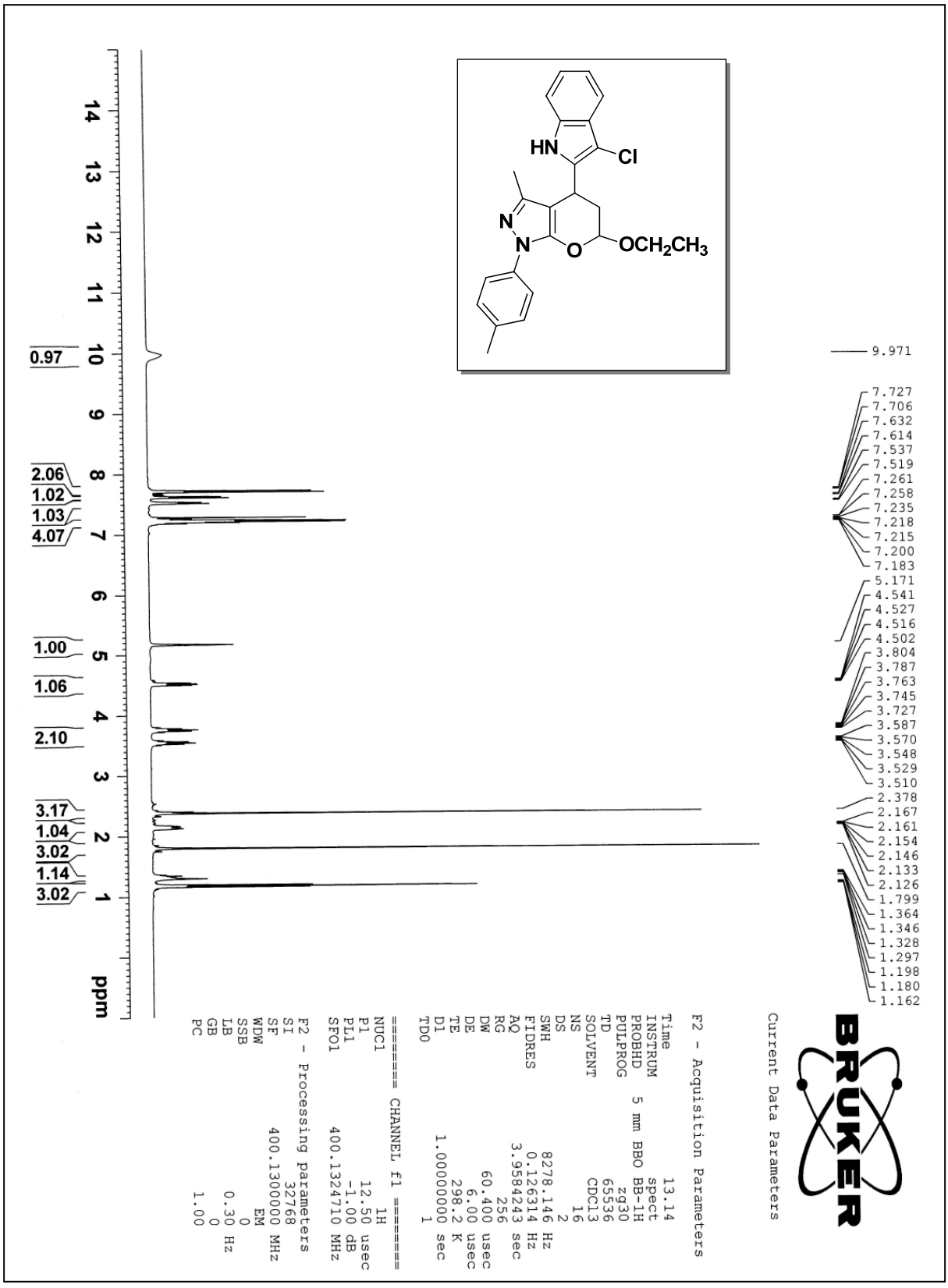
^{1}H-NMR/**7a**/$CDCl_3$



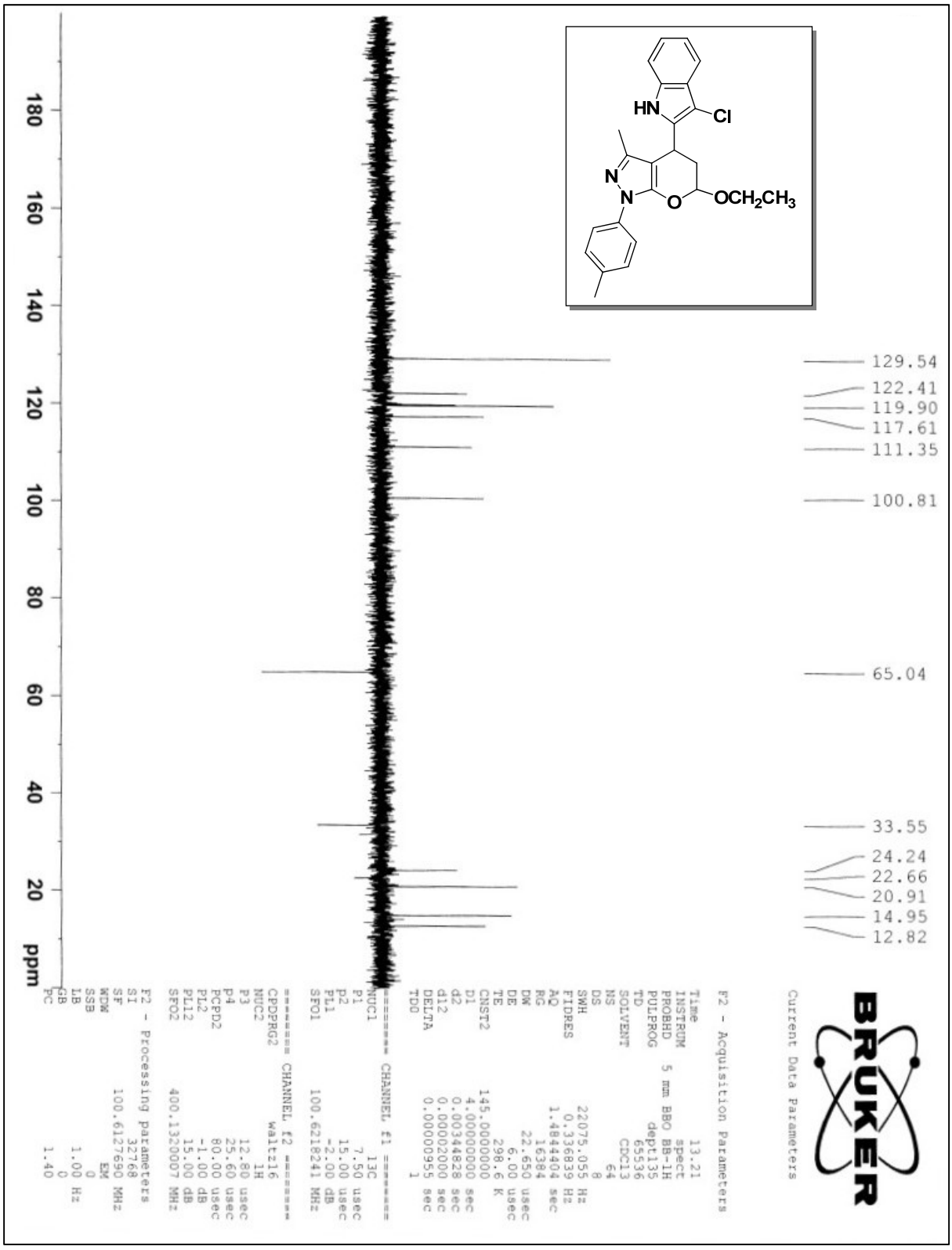
2D NOE for 7a


H
H H
4
Het
O
H
6
OEt
ppm
ppm
BRUKER
Current Data Parameters
F2 - Acquisition Parameters
Time 16.36
INSTRUM spect
PROBHD 5 mm BBO BB-1H
PULPROG noesyph
TD 2048
SOLVENT CDCl3
NS 80
DS 16
SWH 4401.409 Hz
FIDRES 2.149125 Hz
AQ 0.2327028 sec
RG 256
DW 113.600 usec
DE 6.00 usec
TE 298.1 K
d0 0.00009450 sec
D1 2.00000000 sec
D8 0.30000001 sec
IN0 0.00022720 sec
STICNT 128
======== CHANNEL f1 ========
NUC1 1H
P1 15.00 usec
PL1 -6.00 dB
SFO1 400.1322005 MHz
F1 - Acquisition parameters
NDO 1
TD 256
SFO1 400.1322 MHz
FIDRES 17.193003 Hz
SW 11.000 ppm
FnMODE States-TPPI
F2 - Processing parameters
SI 1024
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 1024
MC2 States-TPPI
SF 400.1300000 MHz
WDW QSINE
SSB 2
LB 0.00 Hz
GB 0

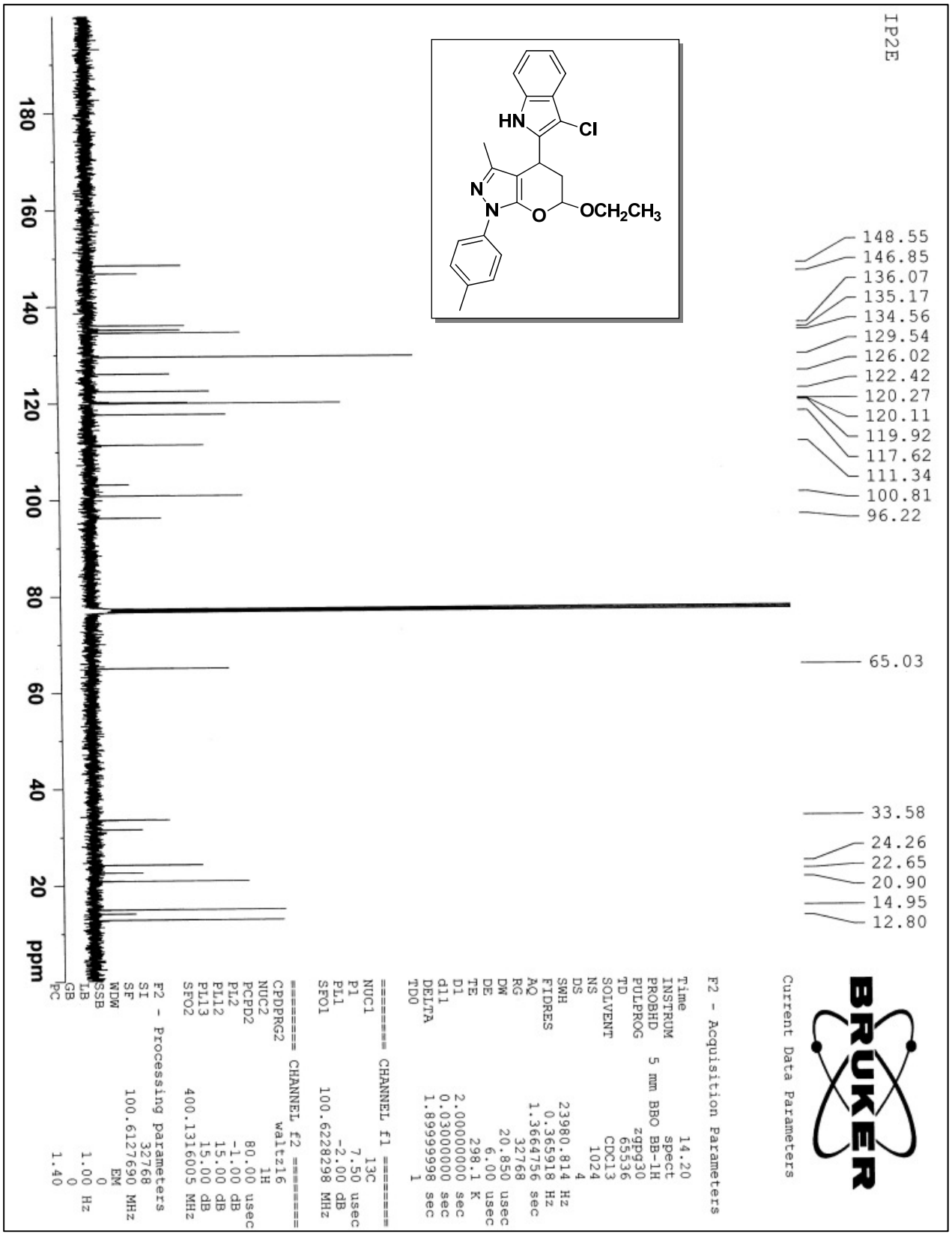
^{1}H-NMR/**7b**/$CDCl_3$



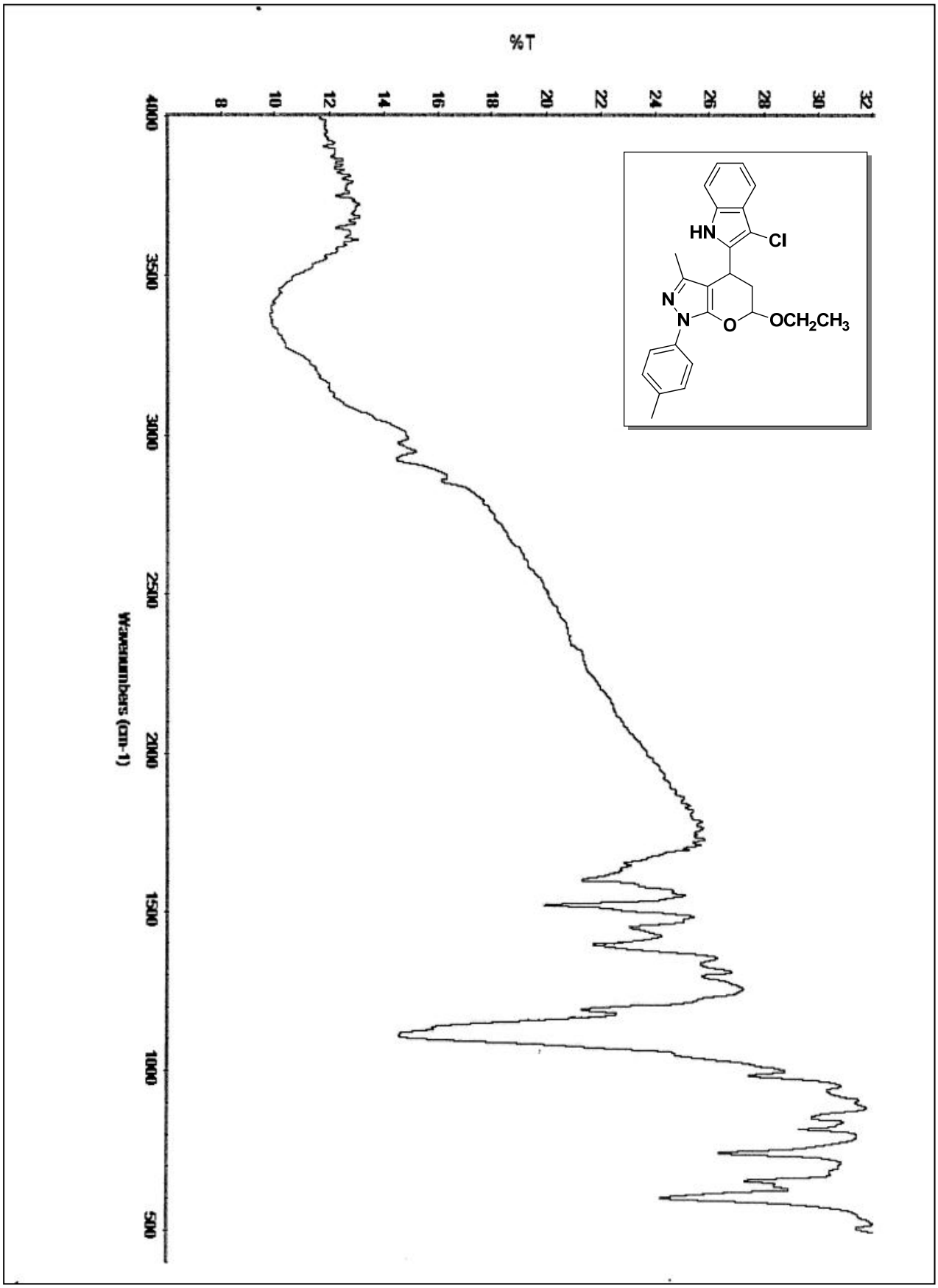
^{13}C-NMR (DEPT-135)/ **7b**/ $CDCl_3$



^{13}C-NMR/**7b**/ $CDCl_3$



IR/**7b**/ KBr


%T
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4000
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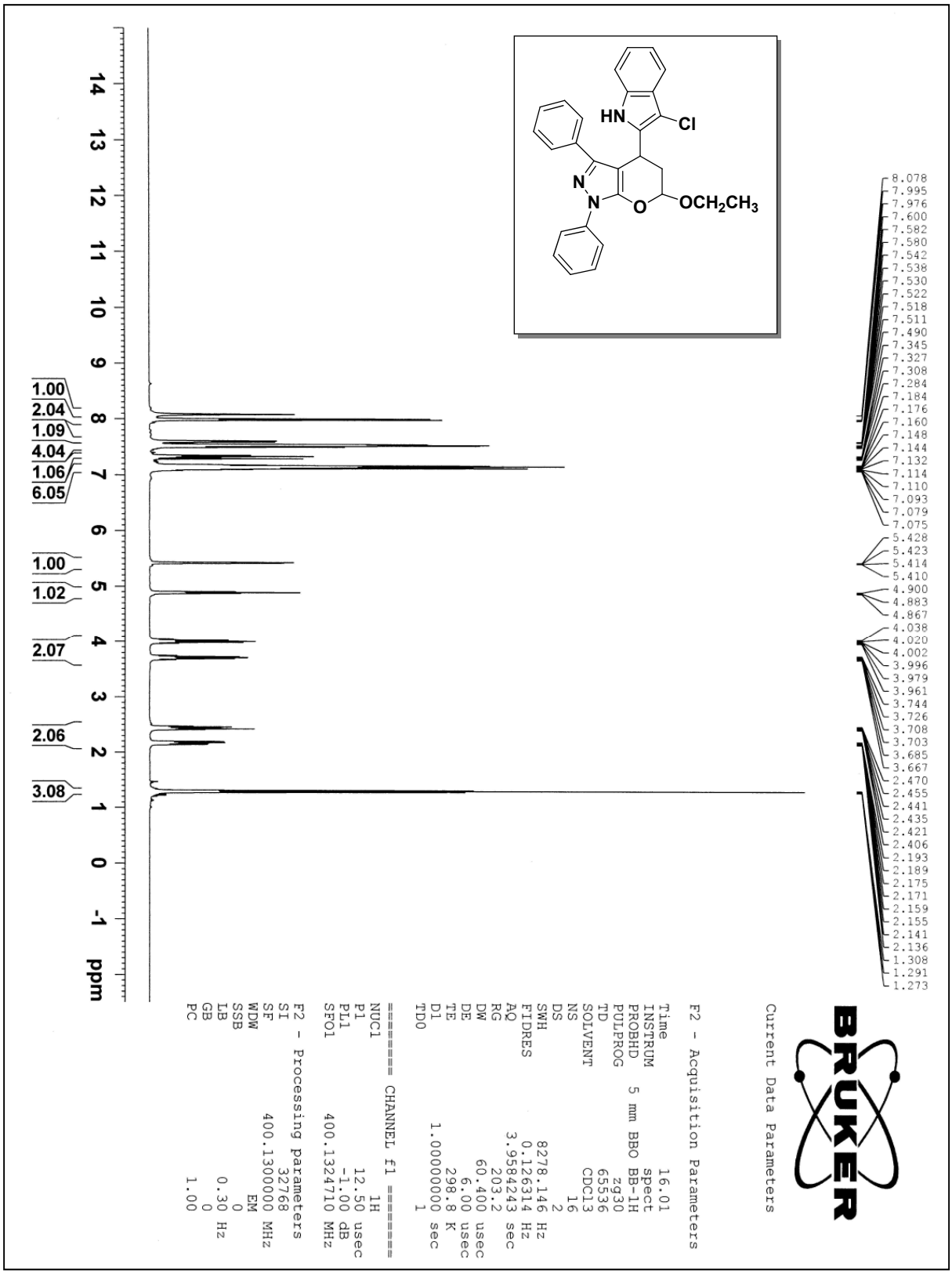
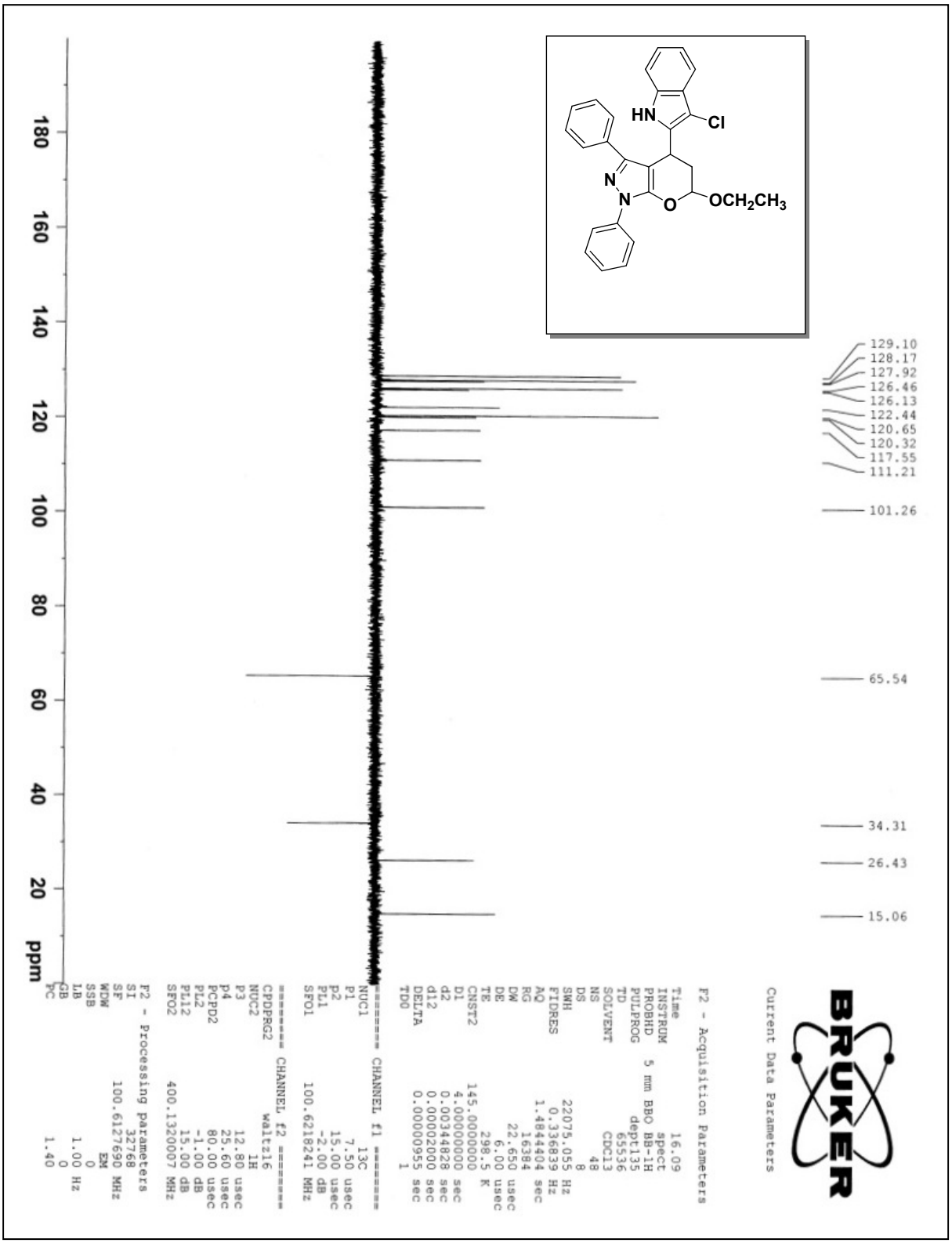
2500
2000
1500
1000
500
Wavenumbers (cm-1)
HN
Cl
N
N
O
OCH2CH3

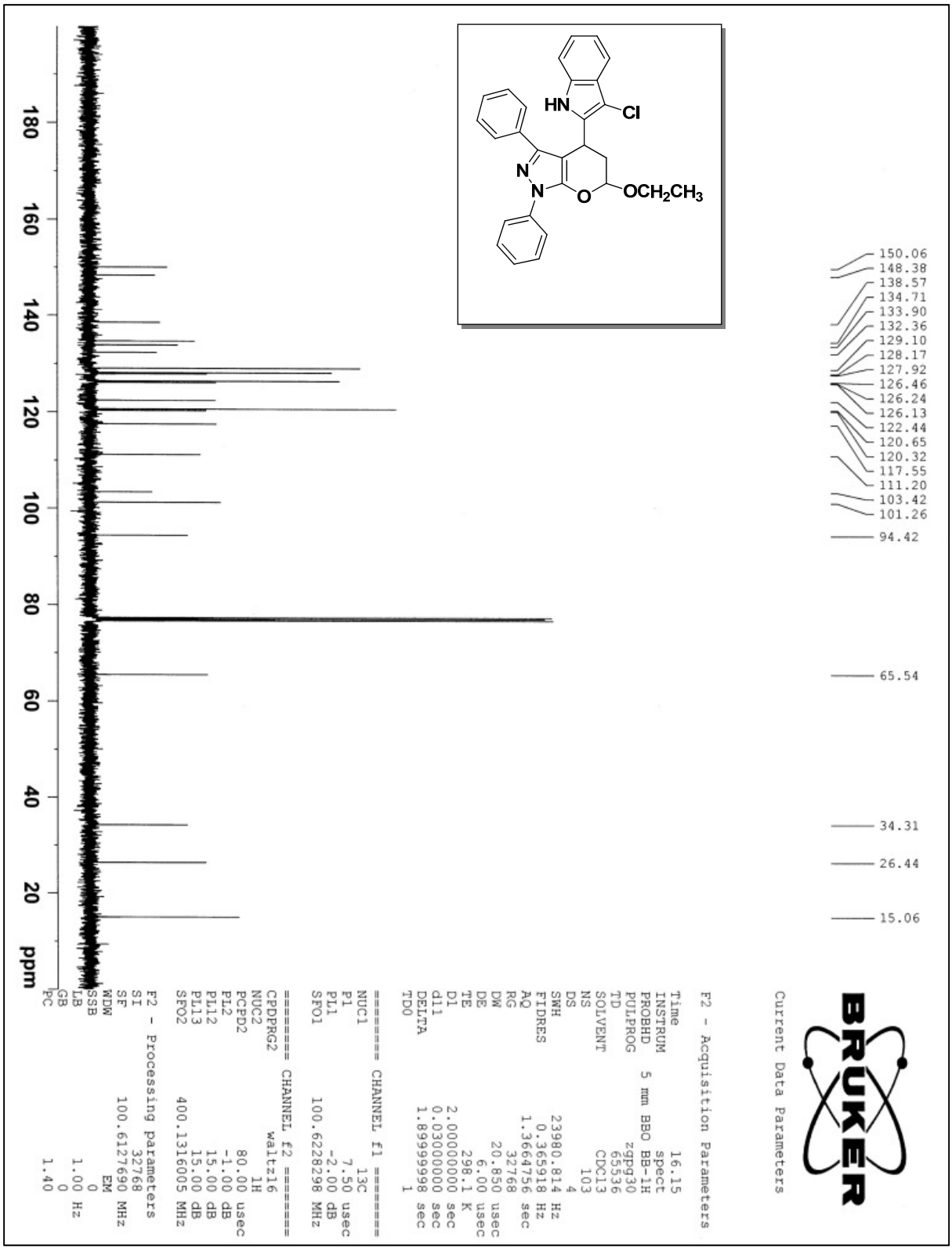
^{1}H-NMR/**7c**/$CDCl_3$



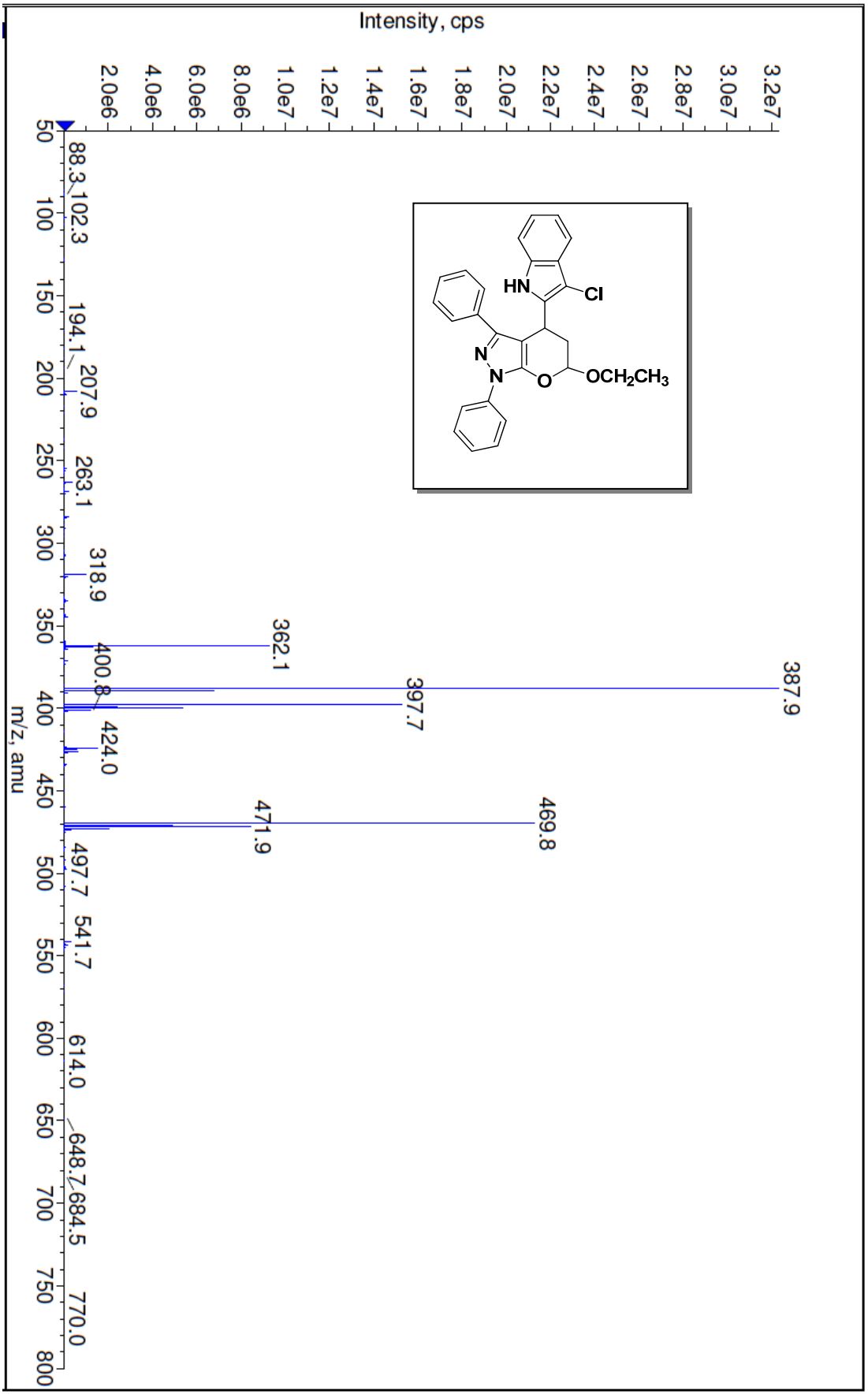
^{13}C-NMR (DEPT-135)/ **7c**/ $CDCl_3$



^{13}C-NMR/**7c**/ $CDCl_3$



MS/**7c**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8.0e6
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1.4e7
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3.0e7
3.2e7
m/z, amu
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100
150
200
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550
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700
750
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102.3
194.1
207.9
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318.9
362.1
400.8
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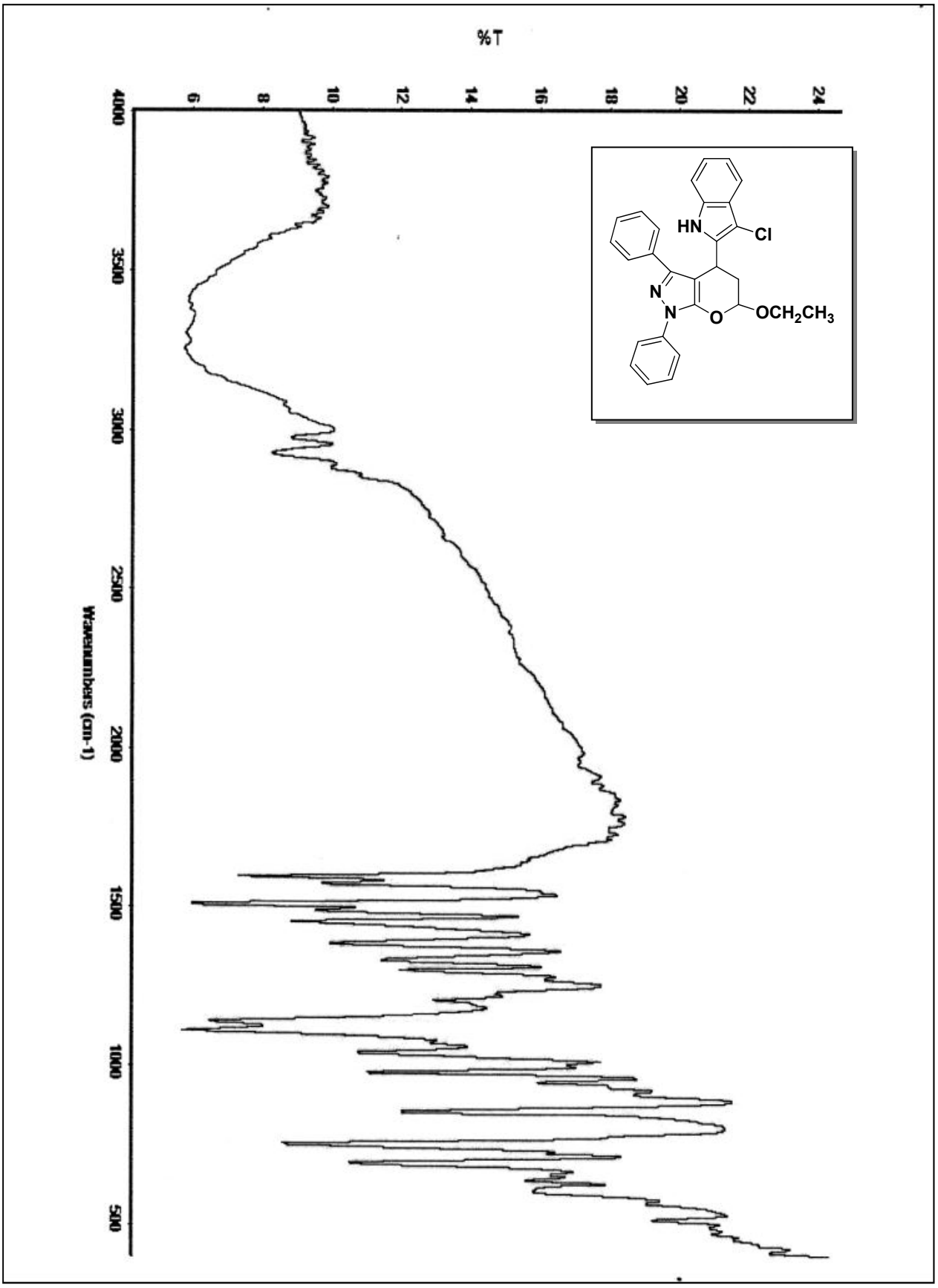
387.9
424.0
471.9
469.8
497.7
541.7
614.0
648.7
684.5
770.0
HN
Cl
N
N
O
OCH2CH3

IR/**7c**/ KBr


%T
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4000
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3000
2500
2000
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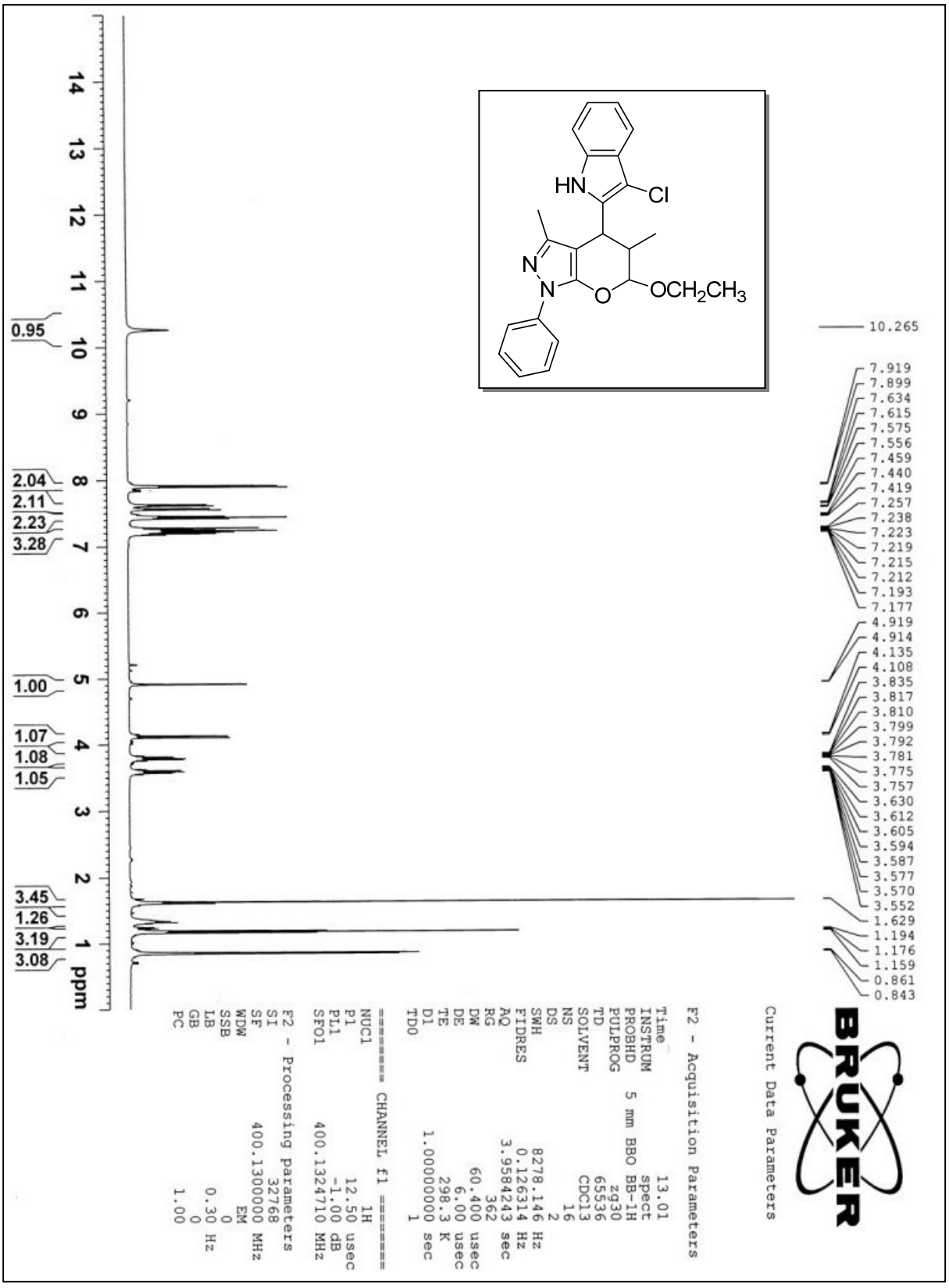
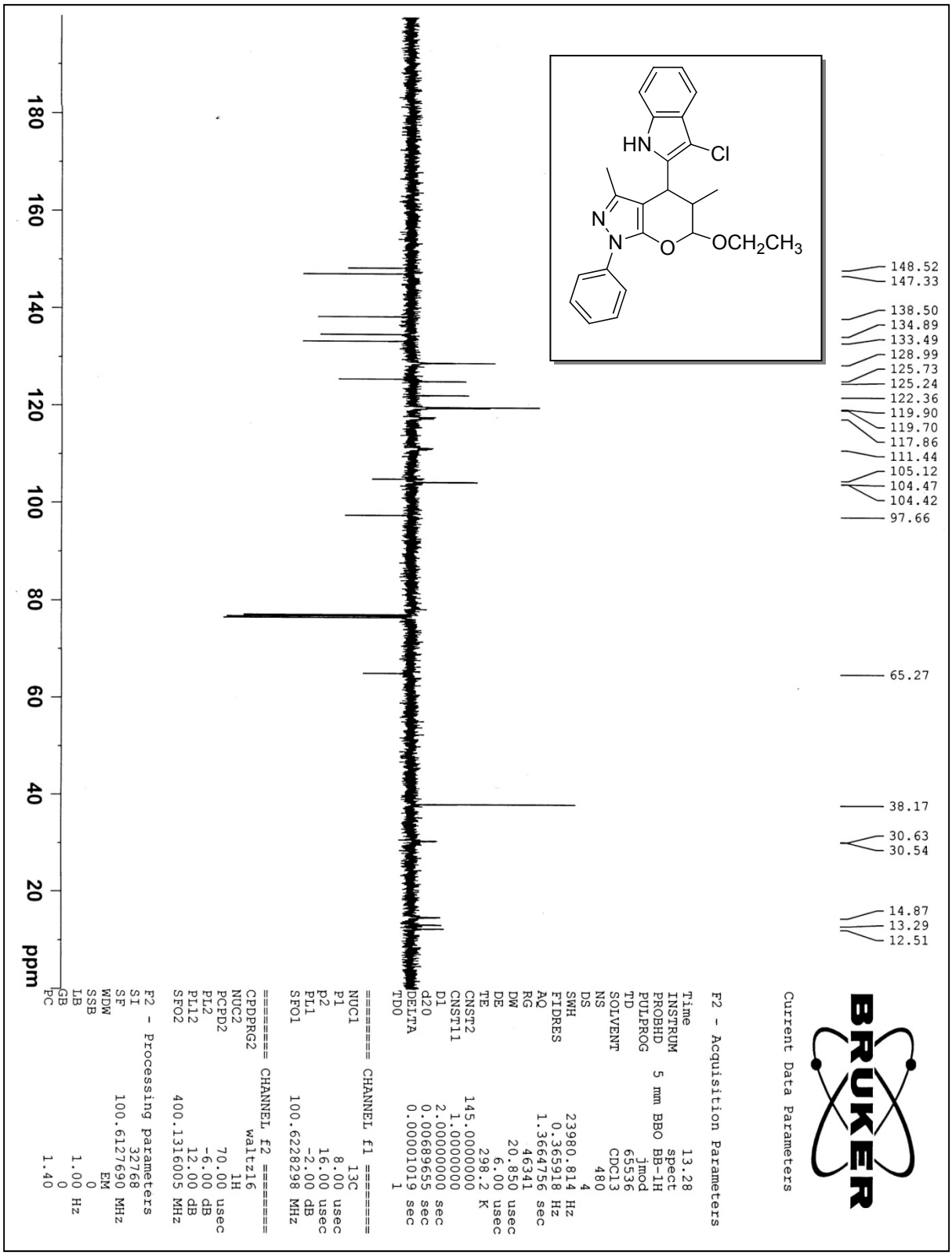
1000
500
Wavenumbers (cm-1)
HN
Cl
N
N
O
OCH2CH3

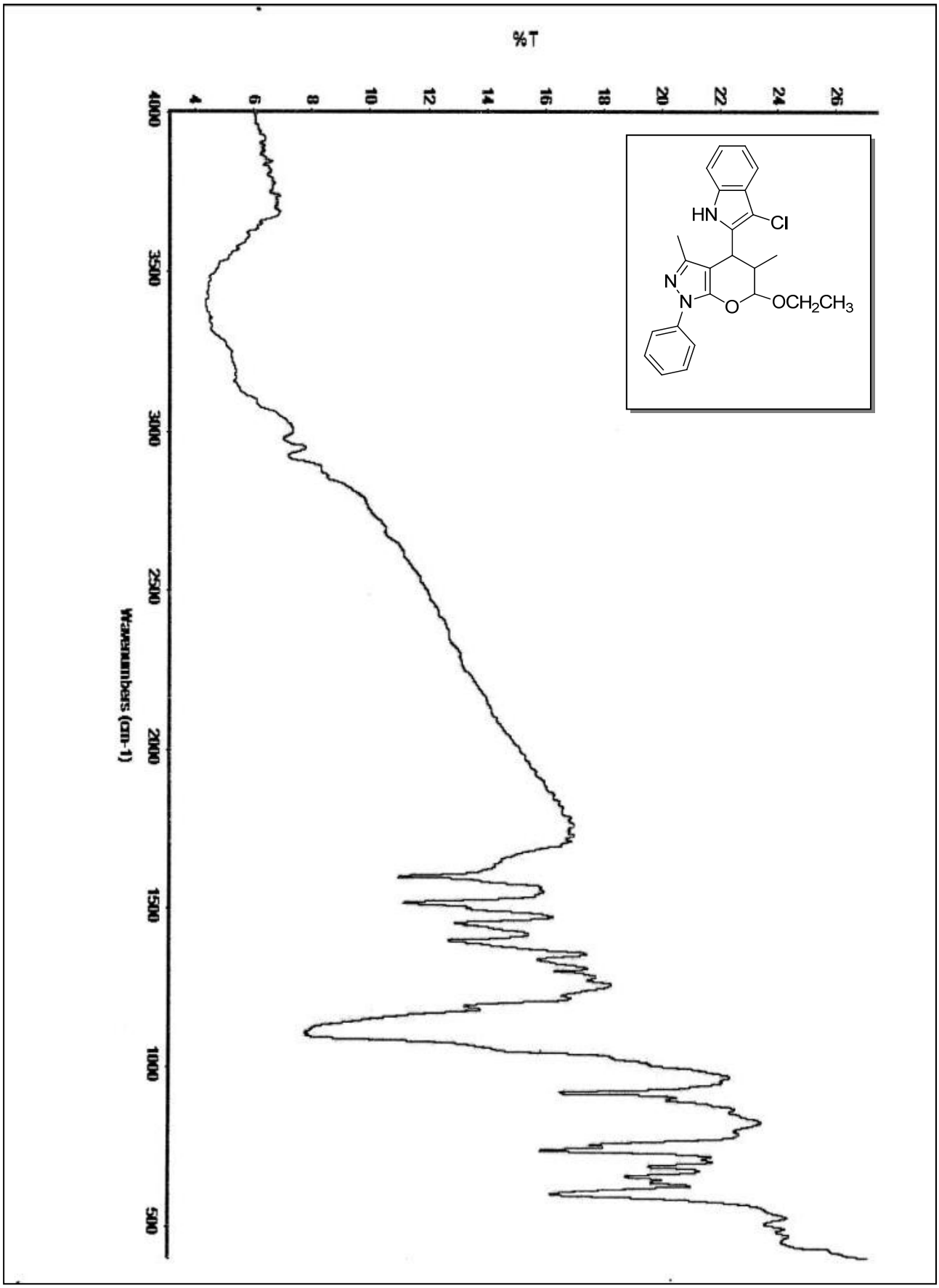
^{1}H-NMR/**8a**/$CDCl_3$



^{13}C-NMR (APT)/ **8a**/ $CDCl_3$



IR/**8a**/KBr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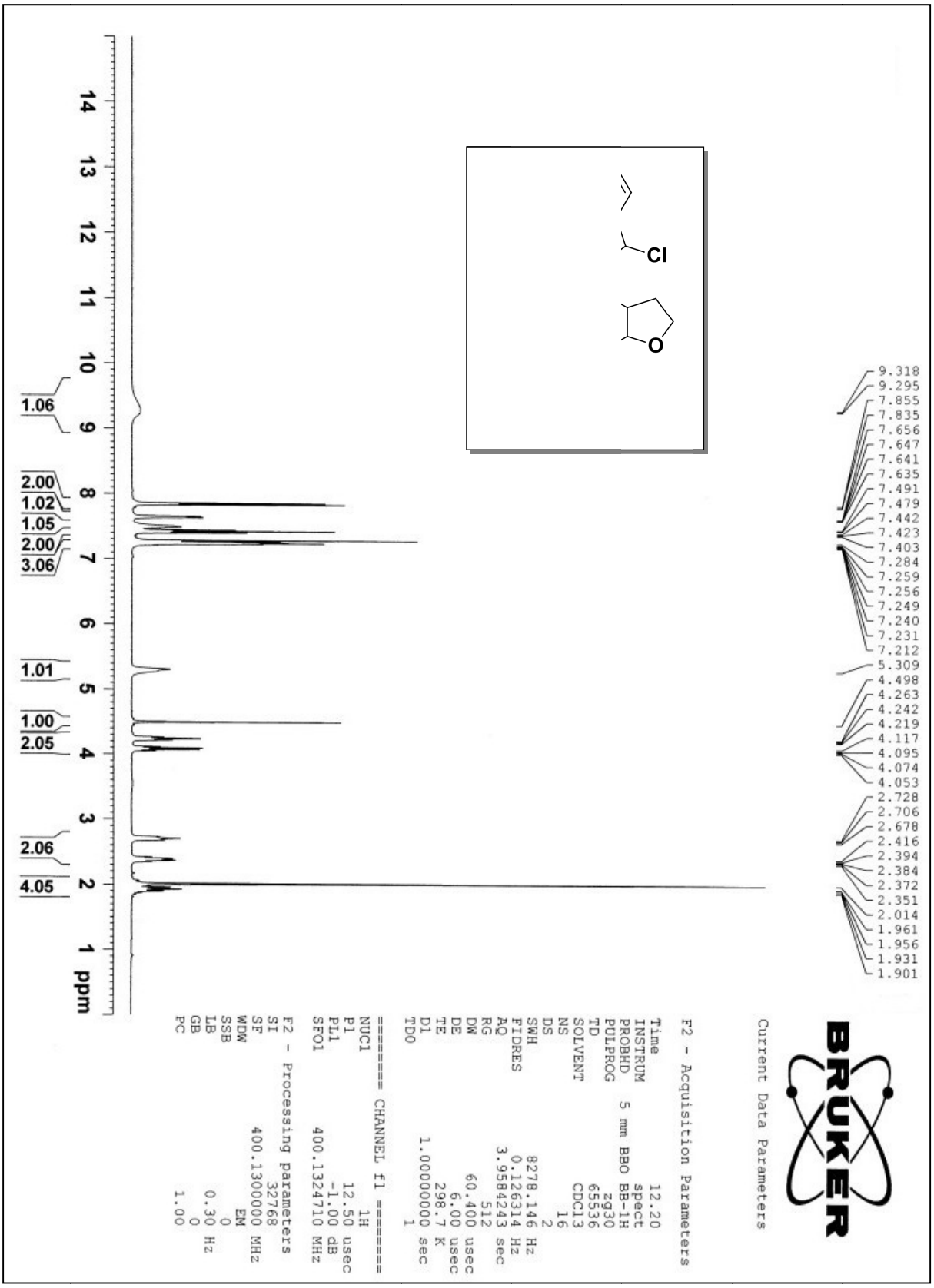
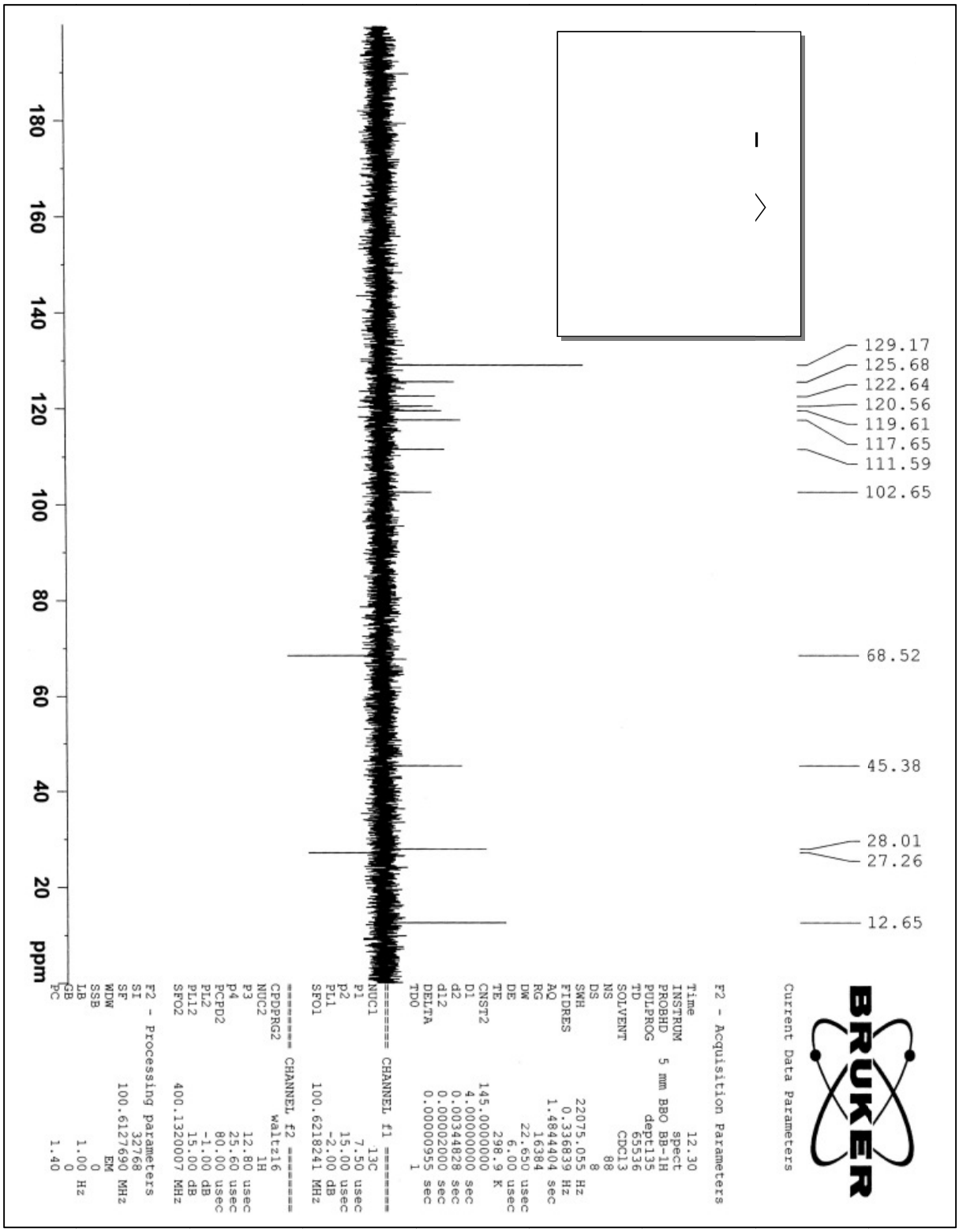
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
HN
Cl
N
N
O
OCH2CH3

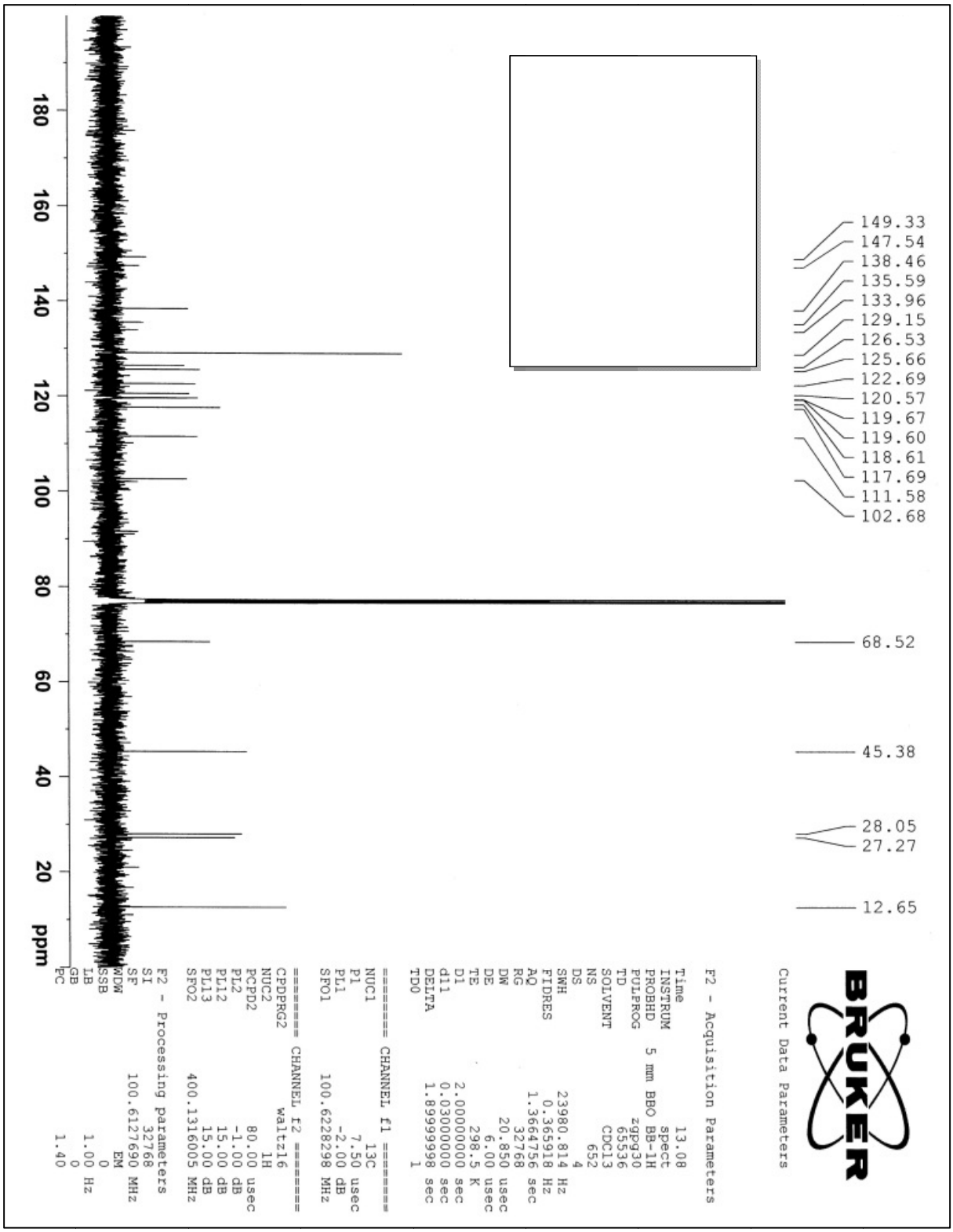
^{1}H-NMR/**9a**/$CDCl_3$



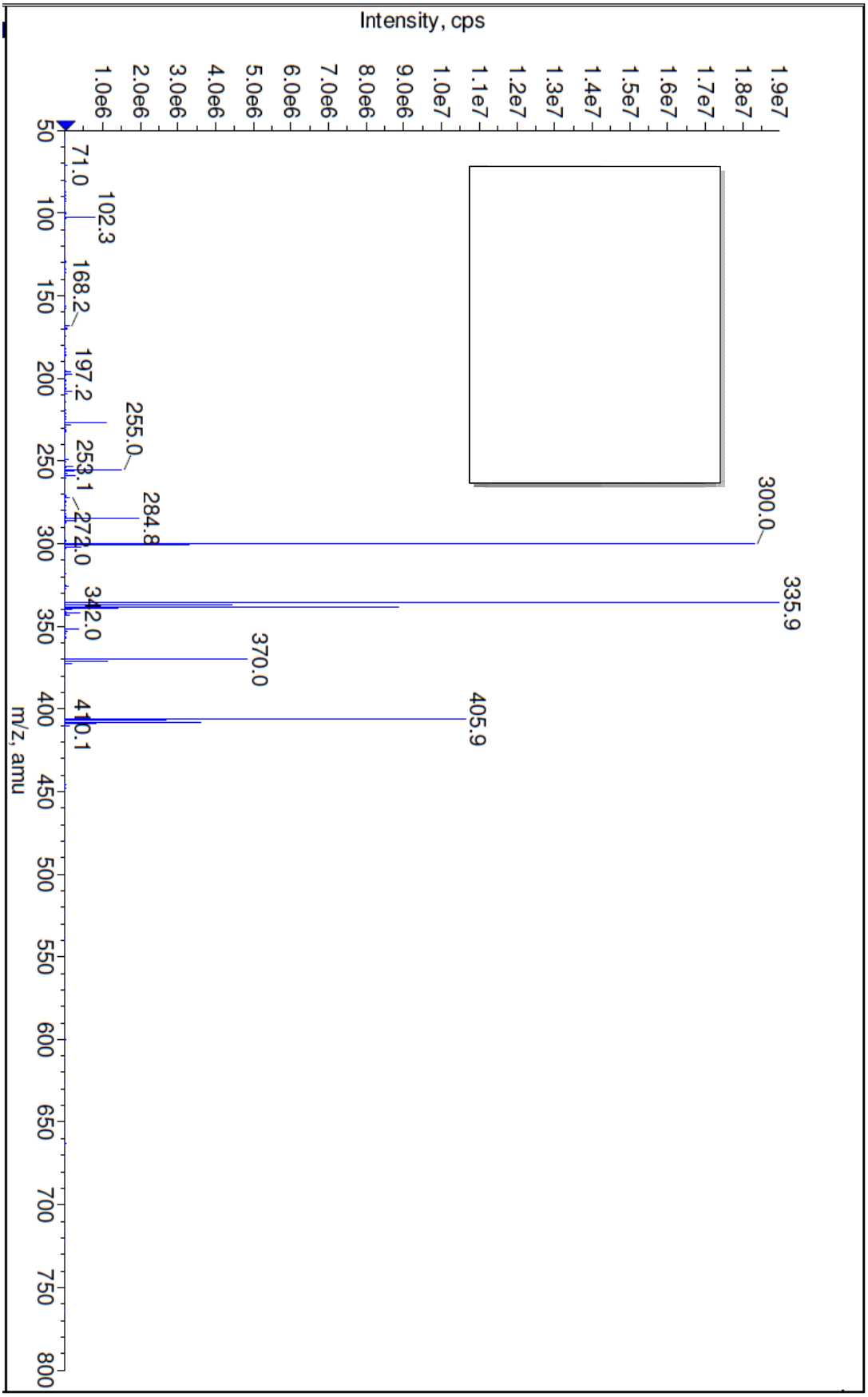
^{13}C-NMR (DEPT-135)/ **9a**/ $CDCl_3$



^{13}C-NMR/**9a**/ $CDCl_3$



MS/**9a**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1.6e7
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1.8e7
1.9e7
m/z, amu
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450
500
550
600
650
700
750
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71.0
102.3
168.2
197.2
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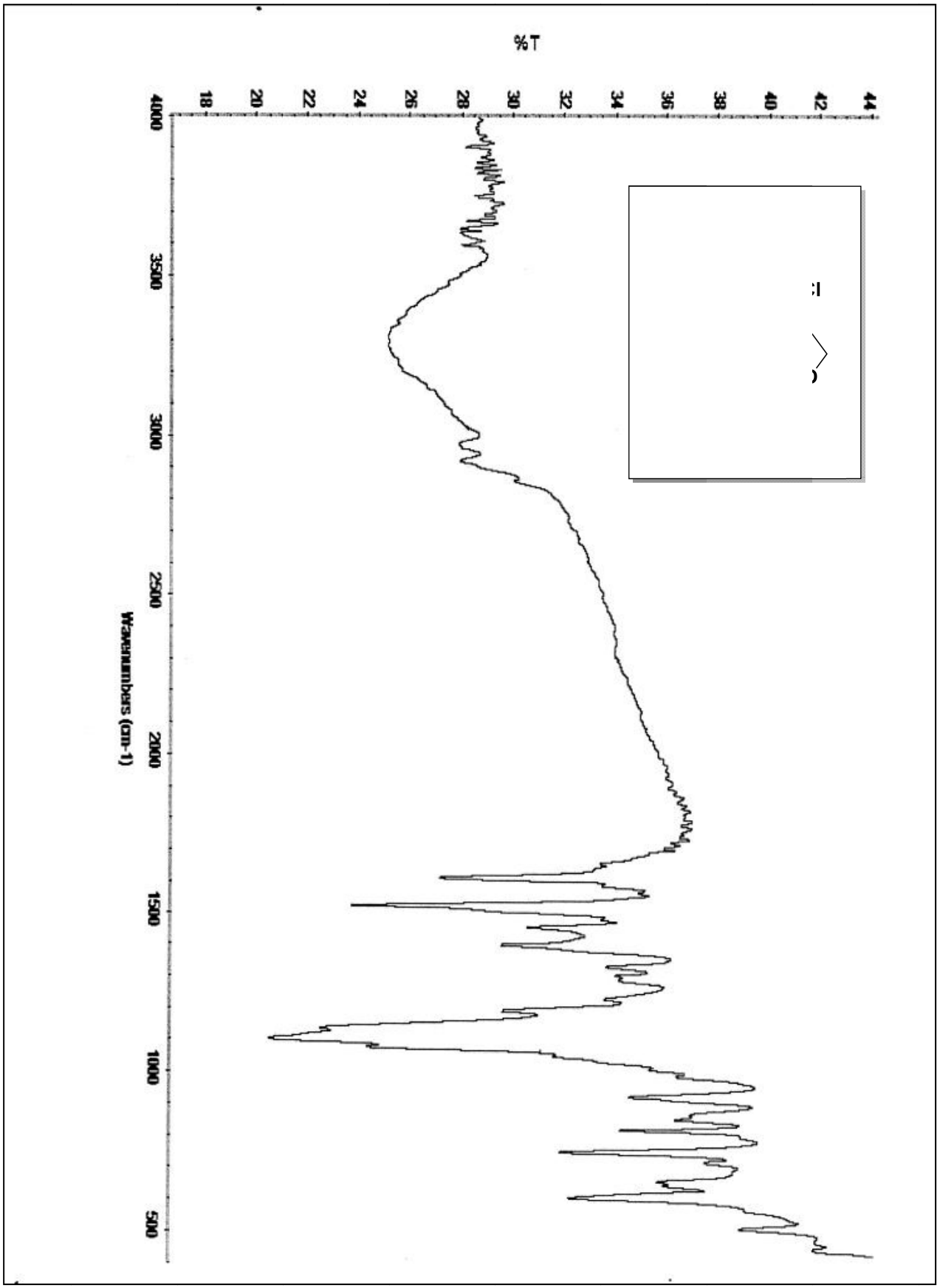
272.0
300.0
335.9
342.0
370.0
410.1
405.9

IR/**9a**/ KBr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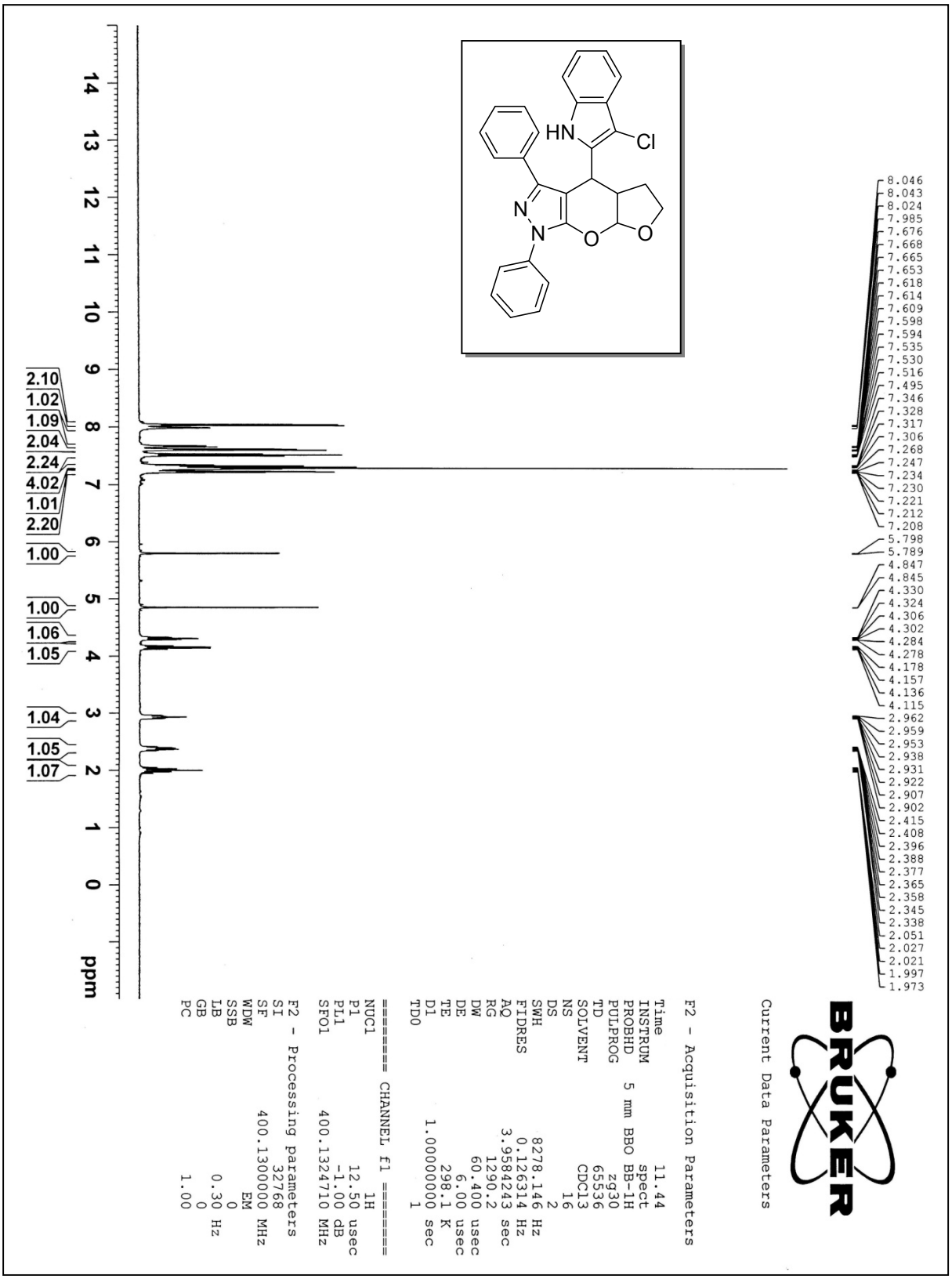
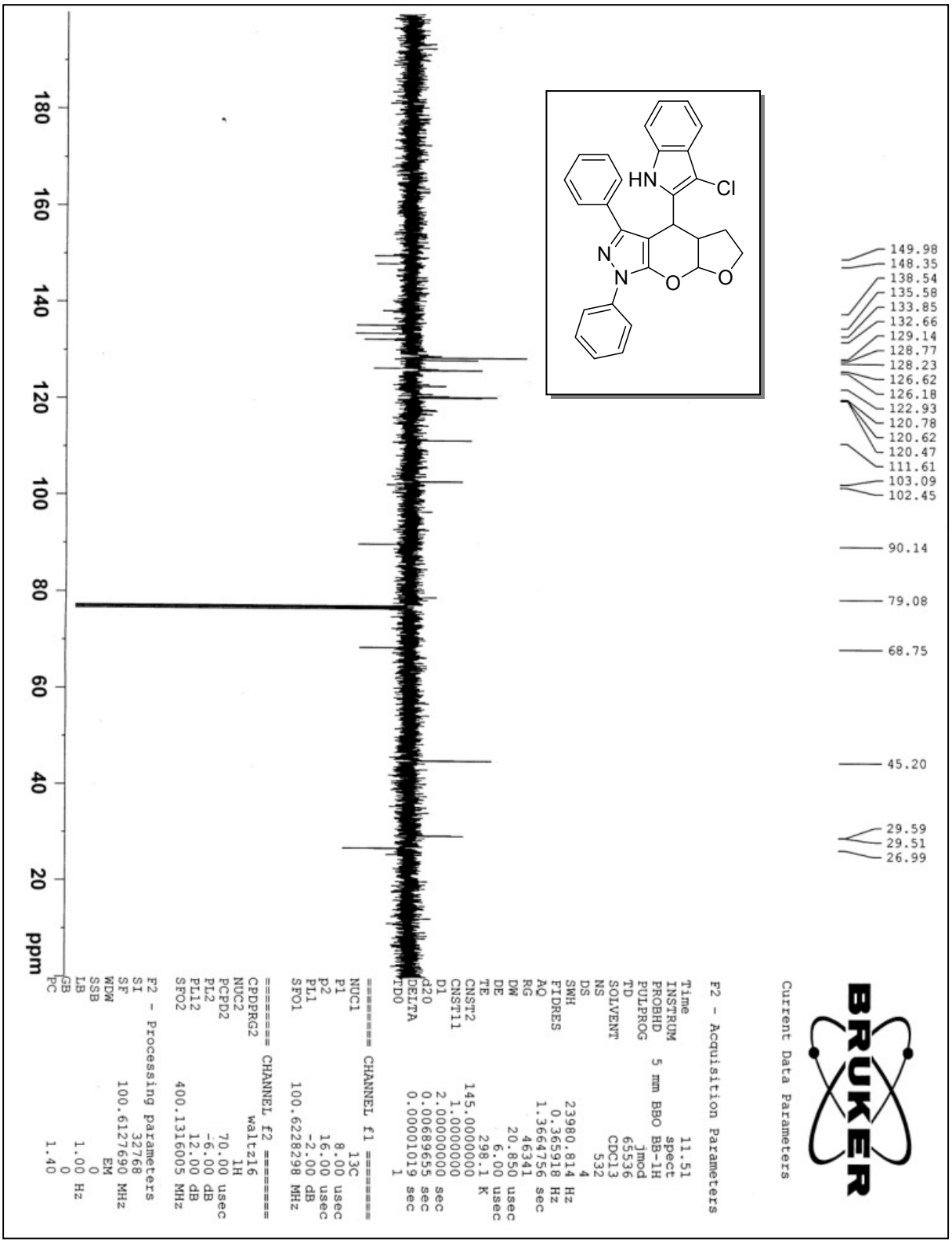
1000
500
Wavenumbers (cm-1)

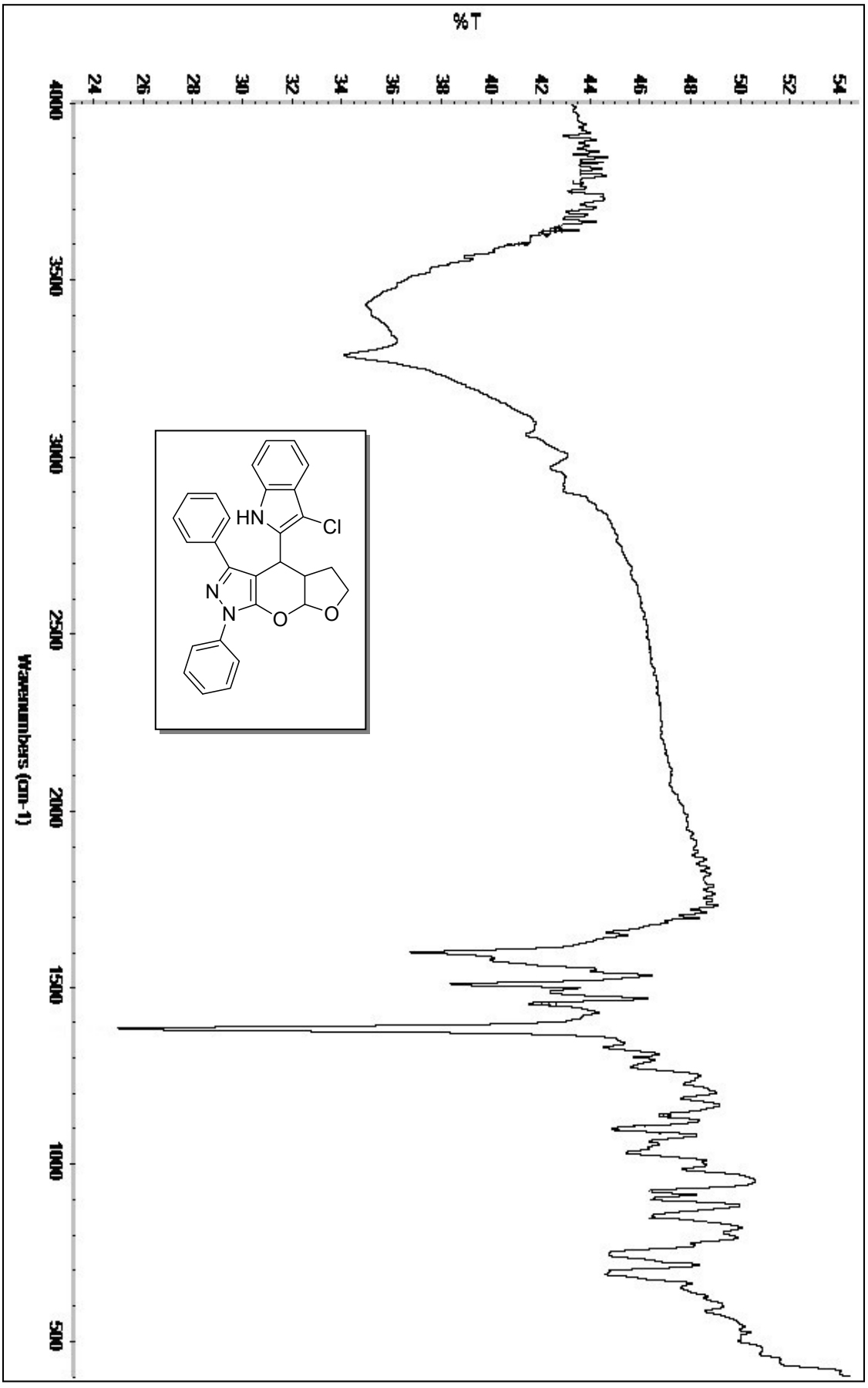
^{1}H-NMR/**9c**/$CDCl_3$



^{13}C-NMR (APT)/**9c**/ $CDCl_3$



IR/**9c**/ KBr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28
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32
34
36
38
40
42
44
46
48
50
52
54
4000
3500
3000
2500
2000
1500
1000
500
Wavenumbers (cm-1)
HN
Cl
N
N
O
O